chloride buffer TS and 0.1 mL of eriochrome black TS, and titrate with 0.05 *M* disodium ethylenediaminetetraacetate VS until the solution is deep blue in color. Each mL of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 22.78 mg of $C_{12}H_{22}O_{14}Zn$.

Zinc Oxide

ZnO 81.39
Zinc oxide.
Zinc oxide [*1314-13-2*].

» Zinc Oxide, freshly ignited, contains not less than 99.0 percent and not more than 100.5 percent of ZnO.

Packaging and storage—Preserve in well-closed containers.

Identification—

A: When strongly heated, it assumes a yellow color that disappears on cooling.

B: A solution of it in a slight excess of 3 *N* hydrochloric acid responds to the tests for *Zinc* ⟨191⟩.

Alkalinity—Mix 1.0 g with 10 mL of hot water, add 2 drops of phenolphthalein TS, and filter: if a red color is produced, not more than 0.30 mL of 0.10 *N* hydrochloric acid is required to discharge it.

Loss on ignition ⟨733⟩—Weigh accurately about 2 g, and ignite at 500° to constant weight: it loses not more than 1.0% of its weight.

Carbonate and color of solution—Mix 2.0 g with 10 mL of water, add 30 mL of 2 *N* sulfuric acid, and heat on a steam bath, with constant stirring: no effervescence occurs and the resulting solution is clear and colorless.

Arsenic, *Method I* ⟨211⟩: 6 ppm.

Lead—Add 2 g to 20 mL of water, stir well, add 5 mL of glacial acetic acid, and warm on a steam bath until solution is effected: the addition of 5 drops of potassium chromate TS produces no turbidity or precipitate.

Iron and other heavy metals—Cooled 5-mL portions of the solution obtained in the test for *Carbonate and color of solution* yield white precipitates with potassium ferrocyanide TS and with sodium sulfide TS.

Assay—Dissolve about 1.5 g of freshly ignited Zinc Oxide, accurately weighed, and 2.5 g of ammonium chloride in 50.0 mL of 1 *N* sulfuric acid VS with the aid of gentle heat, if necessary. When solution is complete, add methyl orange TS, and titrate the excess sulfuric acid with 1 *N* sodium hydroxide VS. Each mL of 1 *N* sulfuric acid is equivalent to 40.69 mg of ZnO.

Zinc Oxide Ointment

» Zinc Oxide Ointment contains not less than 18.5 percent and not more than 21.5 percent of ZnO.

It may be prepared as follows:

Zinc Oxide	200 g
Mineral Oil	150 g
White Ointment	650 g
To make	1000 g

Levigate the Zinc Oxide with the Mineral Oil to smooth paste, and then incorporate the White Ointment [see *Ointments and Suppositories* under *Added Substances* (*Ingredients and Processes*) in the *General Notices*].

Packaging and storage—Preserve in well-closed containers, and avoid prolonged exposure to temperatures exceeding 30°.

Identification—The residue obtained in the *Assay* is yellow when hot and white when cool.

Minimum fill ⟨755⟩: meets the requirements.

Calcium, magnesium, and other foreign substances—Heat about 2 g gently until melted, and continue the heating, gradually raising the temperature until the mass is thoroughly charred. Ignite the mass until the residue is uniformly yellow. To the residue add 6 mL of 3 *N* hydrochloric acid: no effervescence occurs. Heat the mixture on a steam bath for 10 to 15 minutes: not more than a trace of insoluble residue remains. Filter the solution, dilute with water to 10 mL, add 6 *N* ammonium hydroxide until the precipitate first formed redissolves, then add 2 mL each of ammonium oxalate TS and dibasic sodium phosphate TS: not more than a slight turbidity is produced in 5 minutes.

Assay—Weigh accurately in a porcelain crucible about 700 mg of Ointment, heat gently until melted, and continue the heating, gradually raising the temperature until the mass is thoroughly charred. Ignite the mass until the residue is uniformly yellow, and cool. Dissolve the residue in 10 mL of 2 *N* sulfuric acid, warming if necessary to effect complete solution, transfer the solution to a beaker, and rinse the crucible with small portions of water until the combined solution and rinsings measure 50 mL. Add 15 mL of ammonia–ammonium chloride buffer TS and 1 mL of eriochrome black TS, and titrate with 0.05 *M* disodium ethylenediaminetetraacetate VS until the solution is blue in color. Each mL of 0.05 *M* disodium ethylenediaminetetraacetate is equivalent to 4.069 mg of ZnO.

Zinc Oxide Paste

» Zinc Oxide Paste contains not less than 24.0 percent and not more than 26.0 percent of ZnO.

It may be prepared as follows:

Zinc Oxide	250 g
Starch	250 g
White Petrolatum	500 g
To make	1000 g

Mix the ingredients.

Packaging and storage—Preserve in well-closed containers, and avoid prolonged exposure to temperatures exceeding 30°.

Identification—The residue obtained in the *Assay* is yellow when hot and white when cool.

Minimum fill ⟨755⟩: meets the requirements.

Assay—Using about 600 mg of Paste, proceed as directed in the *Assay* under *Zinc Oxide Ointment*.

Zinc Oxide and Salicylic Acid Paste

» Zinc Oxide and Salicylic Acid Paste contains not less than 23.5 percent and not more than 25.5 percent of zinc oxide (ZnO), and not less than 1.9 percent and not more than 2.1 percent of salicylic acid ($C_7H_6O_3$).

It may be prepared as follows:

Salicylic Acid, in fine powder	20 g
Zinc Oxide Paste, a sufficient quantity, to make	1000 g

Diiodofluorescein TS—Dissolve 500 mg of diiodofluorescein in a mixture of 75 mL of alcohol and 30 mL of water.

Diluted Lead Subacetate TS—See *Lead Subacetate TS, Diluted.*

***p*-Dimethylaminobenzaldehyde TS**—Dissolve 125 mg of *p*-dimethylaminobenzaldehyde in a cooled mixture of 65 mL of sulfuric acid and 35 mL of water, and add 0.05 mL of ferric chloride TS. Use within 7 days.

Dinitrophenylhydrazine TS—Carefully mix 10 mL of water and 10 mL of sulfuric acid, and cool. To the mixture, contained in a glass-stoppered flask, add 2 g of 2,4-dinitrophenylhydrazine, and shake until dissolved. To the solution add 35 mL of water, mix, cool, and filter.

Diphenylamine TS—Dissolve 1.0 g of diphenylamine in 100 mL of sulfuric acid. The solution should be colorless.

Diphenylcarbazone TS—Dissolve 1 g of crystalline diphenylcarbazone in 75 mL of alcohol, then add alcohol to make 100 mL. Store in a brown bottle.

Dithizone TS—Dissolve 25.6 mg of dithizone in 100 mL of alcohol. Store in a cold place, and use within 2 months.

Edetate Disodium TS—Dissolve 1 g of edetate disodium in 950 mL of water, add 50 mL of alcohol, and mix.

Eosin Y TS (adsorption indicator)—Dissolve 50 mg of eosin Y in 10 mL of water.

Eriochrome Black TS—Dissolve 200 mg of eriochrome black T and 2 g of hydroxylamine hydrochloride in methanol to make 50 mL.

Eriochrome Cyanine TS—Dissolve 750 mg of eriochrome cyanine R in 200 mL of water, add 25 g of sodium chloride, 25 g of ammonium nitrate, and 2 mL of nitric acid, and dilute with water to 1000 mL.

Fehling's Solution—See *Cupric Tartrate TS, Alkaline.*

Ferric Ammonium Sulfate TS—Dissolve 8 g of ferric ammonium sulfate in water to make 100 mL.

Ferric Chloride TS—Dissolve 9 g of ferric chloride in water to make 100 mL.

Ferrous Sulfate TS—Dissolve 8 g of clear crystals of ferrous sulfate in about 100 mL of recently boiled and thoroughly cooled water. Prepare this solution fresh.

Ferrous Sulfate, Acid, TS—Dissolve 7 g of ferrous sulfate crystals in 90 mL of recently boiled and thoroughly cooled water, and add sulfuric acid to make 100 mL. Prepare this solution immediately prior to use.

Folin-Ciocalteu Phenol TS—Into a 1500-mL flask introduce 100 g of sodium tungstate, 25 g of sodium molybdate, 700 mL of water, 50 mL of phosphoric acid, and 100 mL of hydrochloric acid. Reflux the mixture gently for about 10 hours, and add 150 g of lithium sulfate, 50 mL of water, and a few drops of bromine. Boil the mixture, without the condenser, for about 15 minutes, or until the excess bromine is expelled. Cool, dilute with water to 1 liter, and filter: the filtrate has no greenish tint. Before use, dilute 1 part of the filtrate with 1 part of water.

Formaldehyde TS—Use *Formaldehyde Solution* (see in the section, *Reagents*).

Fuchsin-Pyrogallol TS—Dissolve 100 mg of basic fuchsin in 50 mL of water that previously has been boiled for 15 minutes and allowed to cool slightly. Cool, add 2 mL of a saturated solution of sodium bisulfite, mix, and allow to stand for not less than 3 hours. Add 0.9 mL of hydrochloric acid, mix, and allow to stand overnight. Add 100 mg of pyrogallol, shake until solution is effected, and dilute with water to 100 mL. Store in an amber-glass bottle in a refrigerator.

Fuchsin–Sulfurous Acid TS—Dissolve 200 mg of basic fuchsin in 120 mL of hot water, and allow the solution to cool. Add a solution of 2 g of anhydrous sodium sulfite in 20 mL of water, then add 2 mL of hydrochloric acid. Dilute the solution with water to 200 mL, and allow to stand for at least 1 hour. Prepare this solution fresh.

Gastric Fluid, Simulated, TS—Dissolve 2.0 g of sodium chloride and 3.2 g of pepsin in 7.0 mL of hydrochloric acid and sufficient water to make 1000 mL. This test solution has a pH of about 1.2.

Gelatin TS (for the assay of *Corticotropin Injection*)—Dissolve 340 g of acid-treated precursor gelatin (Type A) in water to make 1000 mL. Heat the solution in an autoclave at 115° for 30 minutes after the exhaust line temperature has reached 115°. Cool the solution, and add 10 g of phenol and 1000 mL of water. Store in tight containers in a refrigerator.

Glacial Acetic Acid TS—See *Acetic Acid, Glacial, TS.*

Glucose oxidase–chromogen TS—A solution containing, in each mL, 0.5 µmol of 4-aminoantipyrine, 22.0 µmol of sodium *p*-hydroxybenzoate, not less than 7.0 units of glucose oxidase, and not less than 0.5 units of peroxidase, and buffered to a pH of 7.0 ± 0.1.[80]

Suitability—When used for determining glucose in Inulin, ascertain that no significant color results by reaction with fructose, and that a suitable absorbance-versus-concentration slope is obtained with glucose.

Gold Chloride TS—Dissolve 1 g of gold chloride in 35 mL of water.

Hydrogen Peroxide TS—Use *Hydrogen Peroxide Topical Solution* (USP monograph).

Hydrogen Sulfide TS—A saturated solution of hydrogen sulfide, made by passing H_2S into cold water. Store it in small, dark amber-colored bottles, filled nearly to the top. It is unsuitable unless it possesses a strong odor of H_2S, and unless it produces at once a copious precipitate of sulfur when added to an equal volume of ferric chloride TS. Store in a cold, dark place.

Hydroxylamine Hydrochloride TS—Dissolve 3.5 g of hydroxylamine hydrochloride in 95 mL of 60 percent alcohol, and add 0.5 mL of bromophenol blue solution (1 in 1000) and 0.5 *N* alcoholic potassium hydroxide until a greenish tint develops in the solution. Then add 60 percent alcohol to make 100 mL.

8-Hydroxyquinoline TS—Dissolve 5 g of 8-hydroxyquinoline in alcohol to make 100 mL.

Indigo Carmine TS (*Sodium Indigotindisulfonate TS*)—Dissolve a quantity of sodium indigotindisulfonate, equivalent to 180 mg of $C_{16}H_8N_2O_2(SO_3Na)_2$, in water to make 100 mL. Use within 60 days.

Indophenol-Acetate TS (for the assay of *Corticotropin Injection*)—To 60 mL of standard dichlorophenol-indophenol solution (see in the section *Volumetric Solutions*) add water to make 250 mL. Add to the resulting solution an equal volume of sodium acetate solution freshly prepared by dissolving 13.66 g of anhydrous sodium acetate in water to make 500 mL and adjusting with 0.5 *N* acetic acid to a pH of 7. Store in a refrigerator, and use within 2 weeks.

Intestinal Fluid, Simulated, TS—Dissolve 6.8 g of monobasic potassium phosphate in 250 mL of water, mix, and add 190 mL of 0.2 *N* sodium hydroxide and 400 mL of water. Add 10.0 g of pancreatin, mix, and adjust the resulting solution with 0.2 *N* sodium hydroxide to a pH of 7.5 ± 0.1. Dilute with water to 1000 mL.

Iodine TS—Use *0.1 N Iodine* (see in the section, *Volumetric Solutions*).

Iodine Monochloride TS—Dissolve 10 g of potassium iodide and 6.44 g of potassium iodate in 75 mL of water in a glass-stoppered container. Add 75 mL of hydrochloric acid and 5 mL of chloroform, and adjust to a faint iodine color (in the chloroform) by adding dilute potassium iodide or potassium iodate solution. If much iodine is liberated, use a stronger solution of potassium iodate than 0.01 *M* at first, making the final adjustment with the 0.01 *M* potassium iodate. Store in a dark place, and readjust to a faint iodine color as necessary.

Iodine and Potassium Iodide TS—Dissolve 500 mg of iodine and 1.5 g of potassium iodide in 25 mL of water.

Iodobromide TS—Dissolve 13.615 g of iodine, with the aid of heat, in 825 mL of glacial acetic acid that shows no reduction with dichromate and sulfuric acid. Cool, and titrate 25.0 mL of the solution with 0.1 *N* sodium thiosulfate VS, recording the volume consumed as *B*. Prepare another solution containing 3 mL of bromine in 200 mL of glacial acetic acid. To 5.0 mL of 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.

Weigh accurately 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 indicator, and continue the titration with the edetate disodium solution to a blue endpoint. Calculate the molarity taken by the formula:

$$W/(100.09V),$$

in which W is the weight, in mg, of $CaCO_3$ in the portion of calcium carbonate taken, and V is the volume, in mL, of edetate disodium solution consumed.

Ferric Ammonium Sulfate, Tenth-Normal (0.1 *N*)

$FeNH_4(SO_4)_2 \cdot 12H_2O$, **482.18**
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.

Measure accurately 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, and calculate the normality.

Store in tight containers, protected from light.

Ferrous Ammonium Sulfate, Tenth-Normal (0.1 *N*)

$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, **392.13**
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:

Measure accurately 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. From the volume of 0.1 *N* ceric sulfate consumed, calculate the normality.

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.

Weigh accurately about 1.5 g of primary standard anhydrous sodium carbonate that previously has been heated at a temperature of about 270° for 1 hour. Dissolve it in 100 mL of water, and add 2 drops of methyl red TS. Add the acid slowly from a buret, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration. Heat again to boiling, and titrate further as necessary until the faint pink color is no longer affected by continued boiling. Calculate the normality. Each 52.99 mg of anhydrous sodium carbonate is equivalent to 1 mL of 1 *N* hydrochloric acid.

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.

Weigh accurately about 800 mg of primary standard anhydrous sodium carbonate that previously has been heated at a temperature of about 270° for 1 hour. Proceed as directed under *Hydrochloric Acid, Normal* (1 *N*), beginning with "Dissolve it in 100 mL of water."

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.

Weigh accurately about 150 mg of arsenic trioxide, previously dried at 105° for 1 hour, and dissolve in 20 mL of 1 *N* sodium hydroxide by warming if necessary. Dilute with 40 mL of water, add 2 drops of methyl orange TS, and follow with diluted hydrochloric acid until the yellow color is changed to pink. Then add 2 g of sodium bicarbonate, dilute with 50 mL of water, and add 3 mL of starch TS. Slowly add the iodine solution from a buret until a permanent blue color is produced. Calculate the normality. Each 4.946 mg of arsenic trioxide is equivalent to 1 mL of 0.1 *N* iodine.

Preserve in amber-colored, glass-stoppered bottles.

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

CH_3LiO, **37.98**
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.

Lithium Methoxide, Tenth-Normal (0.1 *N*) in Benzene

CH_3OLi, **37.98**
3.798 g in 1000 mL

Dissolve 0.6 g 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 benzene. 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.

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

CH_3OLi, **37.98**
3.798 g in 1000 mL

Dissolve 0.7 g 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 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.

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. Calculate the molarity. Each 17.22 mg of sulfanilamide is equivalent to 1 mL of 0.1000 *M* sodium nitrite.

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 is equivalent to 955.1 mg of sodium tetraphenylboron. From the weight of sodium tetraphenylboron obtained, calculate the molarity of the sodium tetraphenylboron solution.

NOTE—Prepare this solution fresh.

Sodium Thiosulfate, Tenth-Normal (0.1 *N*)

$Na_2S_2O_3 \cdot 5H_2O$, **248.17**
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.

Weigh accurately about 210 mg of primary standard potassium dichromate, previously pulverized and dried at 120° for 4 hours, 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 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 to the discharge of the blue color. Calculate the normality.

Restandardize the solution frequently.

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

H_2SO_4, **98.07**
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 anhydrous sodium carbonate as described under *Hydrochloric Acid, Half-Normal (0.5 N) in Methanol.*

Sulfuric Acid, Normal (1 *N*)

H_2SO_4, **98.07**
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 sodium carbonate as described under *Hydrochloric Acid, Normal (1 N).*

Tetrabutylammonium Hydroxide, Tenth-Normal (0.1 *N*)

$(C_4H_9)_4NOH$, **259.48**
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 liquid 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.

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. Calculate the molarity.

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. Calculate the molarity.

Titanium Trichloride, Tenth-Normal (0.1 *N*)

$TiCl_3$, **154.24**
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