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

Zinc Oxide

ZnO 81.39

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 dis-

appears on cooling.

B: A solution of it in a slight excess of 3 N hydrochloric acid

does not respond 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 0.1 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.

Iron and other heavy metals—Cooled 5-mL portions of the solu-
tion 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, ac-

curately weighed, and 2.5 g of ammonium chloride in 500.0 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 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 Petroleum 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.

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–

\( \text{H}_2\text{O} \)).

It may be prepared as follows:

Salicylic Acid, in fine powder 20 g
Zinc Oxide Paste, a sufficient quantity, to make 1000 g
Diodofluorescein TS—Dissolve 500 mg of diodofluorescein in a mixture of 75 mL of alcohol and 30 mL of water.

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

p-Dimethylanilinoenzaldehyde TS—Dissolve 125 mg of p-dimethylanilinoenzaldehyde in a cooled mixture of 65 mL of sulfuric acid and 35 mL of water, and add 0.05 mL of ferric chloride US. 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 g 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 hydroxymethylhydrochloride 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-aminooantipyrine, 22.0 μmol of sodium p-hydroxybenzoate, not less than 7.0 μL of glucose oxidase, and not less than 0.5 μL of peroxidase, and buffered to a pH of 7.0 ± 0.1.

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 Sulfide TS—A saturated solution of hydrogen sulfide, made by passing H₂S 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 H₂S, and unless it produces 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 bromphenol blue solution (1 in 1000) and 0.5 N alcohold 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 Indigotinidisulfonate TS)—Dissolve a quantity of sodium indigotinidisulfonate, equivalent to 180 mg of C₁₄H₁₀N₂O₂(SO₃Na)₂, in water to make 100 mL. Use within 60 days.

Indophenol-Indigo 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 solution. 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).

Iodide 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 chloriform, and adjust to a faint iodine color (in the chloriform) 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
Solutions / Volumetric Solutions

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)
1, 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 dilute 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.

Lithium Methoxide, Fiftieth-Normal (0.02 N) in Methanol
CH₃OLi, 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 lithiummethoxide.

Lithium Methoxide, Tenth-Normal (0.1 N) in Benzene
CH₃OLi, 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).

Lithium Methoxide, Tenth-Normal (0.1 N) in Chlorobenzene
CH₃OLi, 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).

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.
with a magnetic stirrer, but avoid pulling a vortex of air beneath the surface. Use the indicator specified in the individual mono-
graph, or, if a potentiometric procedure is specified, determine the
drop point electrometrically, using platinum-calomel or plat-
num-platinum electrodes. When the titration is within 1 mL of the
deck, add the titrant in 0.1-mL portions, and allow 1 minute between additions. Calculate the molarity. Each 17.22 mg of sulphanilamide is equivalent to 1 mL of 0.1000 M sodium nitrite.

**Sodium Tetraperhydroborate, Fiftieth-Molar (0.02 M)**

\[ \text{NaB(C}_2\text{H}_4\text{H}_2\text{)}_3 \text{, 342.22} \]

6.845 g in 1000 mL

Dissolve an amount of sodium tetraperhydroborate, equivalent to
6.845 g of NaB(C₂H₄H₂)₃, in water to make 1000 mL, and stan-
dardize the solution as follows.

Pipe 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 tetraperhydroborate solution in the following standardization pro-
cedure. Filter the second mixture through a tared filtering crucible, and wash the precipitate with three 5-mL portions of satu-
rated potassium tetraperhydroborate solution. Dry the precipitate at 105°C for 1 hour. Each g of potassium tetraperhydroborate is equivalent to 955.1 mg of sodium tetraperhydroborate. From the weight of sodium tetraperhydroborate obtained, calculate the mo-
ularity of the sodium tetraperhydroborate solution.

NOTE—Prepare this solution fresh.

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

\[ \text{Na}_2\text{SO}_3 \cdot \text{SH}_2\text{O, 248.17} \]

24.82 g in 1000 mL

Dissolve about 26 g of sodium thiocyanate and 200 mg of sodium carbonate in 1000 mL of freshly boiled and cooled water. Stan-
dardize the solution as follows.

Weigh accurately about 210 mg of primary standard potassium dichromate, previously pulverized and dried at 120°C 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 thiocyanate 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**

\[ \text{H}_2\text{SO}_4, 98.07 \]

24.52 g in 1000 mL

Add slowly, with stirring, 13.9 mL of sulfuric acid to a suffi-
cient 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)**

\[ \text{H}_2\text{SO}_4, 98.07 \]

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°C, and determine the normality by titration against sodium carbonate as described under Hydrochloric Acid, Normal (1 N).

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

\( (\text{C}_4\text{H}_9\text{)}_4\text{NOH}, \text{259.48} \)

25.95 g in 1000 mL

Dissolve 40 g of tetrabutylammonium 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 agita-
tion. 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 rins-
ings 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 diox-
ide-free nitrogen. [NOTE—If necessary to obtain a clear solu-
tion, 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 end point with the tetrabutylammonium hydroxide so-
lution, 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 tetrabutylammoni-
uum hydroxide is equivalent to 12.21 mg of benzoic acid.

**Tetramethylammonium Bromide, Tenth-Molar (0.1 M)**

\( (\text{CH}_3)_4\text{NB}, \text{154.05} \)

15.41 g in 1000 mL

Dissolve 15.41 g of tetrabutylammonium 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 dilute 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)**

\( (\text{CH}_3)_4\text{NCl, 109.60} \)

10.96 g in 1000 mL

Dissolve 10.96 g of tetrabutylammonium 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 dilute nitric acid and 50.0
mL of 0.1 N silver nitrate VS, and mix. Add 5 mL of nitroben-
zeine 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)**

\( \text{TiCl}_3, \text{154.24} \)

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 de-
scribed.

**Apparatus—**Store the titanium trichloride solution in the res-
ervoir 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