PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

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Molar Solutions:

A molar solution contains 1g molecule of the reagent in 1000 ml of the solution. Thus, each litre of a molar solution of sodium nitrite contains 69.0g of $NaNO_2$ and each litre of a molar solution of disodium edetate contains 372.2 g of $C_{10}H_{14}N_2Na_2O_8,2H_2O$. Solutions containing one-tenth of a gram-molecule of thereagent in 1000 ml are designated as 'tenth-molar' or 0.1 M; other molarities are similarly indicated.

Preparation and Standardisation of Volumetric:

It is not always possible nor is it essential, to prepare volumetric solutions of a desired theoretical molarity. A solution of approximately the desired molarity is prepared and standardised by titration against a solution of a primary standard. The molarity factor so obtained is used in all calculations, where such standardised solutions are employed. As the strength of a standard solution may change upon standing, the molarity factor should be redetermined frequently. Volumetric solutions should not differ from the prescribed strength by more than 10% and the molarity should be determined with a precision of 0.2%.

When solutions of a reagent are used in several molarities, the details of the preparation and standardisation are usually given for the most commonly used strength. Stronger or weaker solutions are prepared and standardised using proprionate amounts of the reagent or by making an exact dilution of a stronger solution. Volumetric solutions prepared by dilution should be restandardised either as directed for the stronger solution or by comparison with another volumetric solution having a known ratio to the stronger solution.

The water used in preparing volumetric solutions complies with the requirements of the monograph on Purified Water, unless otherwise specified. When used for the preparation of unstable solutions such as potassium permanganate or sodium thiosulphate, it should be freshly boiled and cooled. When a solution is to be used in an assay in which the end-point is determined by an electrochemical process (eg. Potentiometrically), the solution must be standardised in the same way.

Blank Determinations

Where it is directed that "any necessary correction" be made by a blank determination, the determination should be done using the same quantities of the same reagents treated in the same manner as the solution or mixture containing the portion of the substance being examined but omitting the substance being examined.

Primary Standards

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These are materials which, after drying under the specified conditions, are recommended for use as primary standards in the standardisation of volumetric solutions. The following are recommended for use as primary standards.

Arsenic Trioxide: Sublime arsenic trioxide in an appropriate apparatus and store over silica gel.

Benzoic Acid: Sublime benzoic Acid in an appropriate apparatus and store over silica gel.

Potassium Bromate: Recrystallise potassium bromate from boiling water. Collect the crystals and dry to constant weight at 180°. Store in a tightly-closed container.

Potassium Dichromate: Heat potassium dichromate to 140° to 150° in an oven, cool in a desiccator and powder in a glass mortar.

Potassium Hydrogen Phthalate: Recrystallise potassium hydrogen phthalate from boiling water, collect the crystals at a temperature above 35° and dry to constant weight at 110°. Store in a tightly-closed container.

Potassium Iodate: Recrystallise potassium iodate from boiling water. Collect the crystals and dry to constant weight at 120°. Store in tightly-closed container.

Sodium Carbonate, Anhydrous: Filter at room temperature a saturated solution of sodium carbonate. Introduce slowly into the filtrate a stream of carbon dioxide, with constant cooling and stirring. After about 2 hours, collect the precipitate on a sintered glass filter. Wash the filter with ice-cold water saturated with carbon dioxide. After drying at 100° to 105° heat to constant weight at 270° to 300° stirring from time to time. Store in a tightly closed container.

Sodium Chloride: To 1 volume of a saturated solution of sodium chloride add 2 volumes of hydrochloric acid. Collect the crystals formed and wash with hydrochloric acid. Remove the hydrochloric acid by heating on a water-bath and dry the crystals to constant weight at 300°. Store in a tightly closed container.

Sulphanilic Acid: Recrystallise sulphanilic acid from boiling water. Filter and dry to constant weight at 100° to 105°.

Zinc, Granulated: Wash granulated zinc with dilute hydrochloric acid, followed by water, ethanol (95%) and finally acetone. Dry at 100° for 5 minutes and cool in a disiccator over silica gel.

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Volumetric Solutions

Ammonium Thiocyanate, 0.1M: Dissolve 7.612 g of ammonium thiocyanate in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

Pipette 30.0 ml of 0.1 M silver nitrate into a glass-stoppered flask, dilute with 50 ml of water, add 2 ml of nitric acid and 2 ml of ferric ammonium sulphate solution and titrate with the ammonium thiocyanate solution to the first appearance of a red-brown colour. Each ml of 0.1 M silver nitrate is equivalent 0.007612 g of NH_4SCN.

Barium Chloride, 0.05M: Dissolve 12.2 g of barium chloride in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

To 10.0 ml of the solution add 60 ml of water, 3 ml of strong ammonia solution and 0.5 to 1 mg of metalphthalein as indicator and titrate with 0.05 M disodium edetate. As the solution begins to decolorise, add 50 ml of ethanol (95%) and titrate until the bluish violet colour is discharged. Each ml of 0.05 M disodium edetate is equivalent to 0.012215 g of $BaCl_2,2H_2O$.

Benzethonium Chloride, 0.004M: Dissolve 1.792 g of benzethonium chloride, previously dried to constant weight at 105°, in sufficient water to produce 1000 ml. Standardise the solution from the content of C27H42CINO2 in the dried benzethonium chloride determined in the following manner.
Dissolve 0.35 g of the dried substance in 30 ml of anhydrous. Glacial acetic acid, add 6 ml of mercuric acetate soluton and carry out method B for non-aqueous titration. Appendix 0.05 ml of crystal violet solution as indicator. Perform a blank determination and make any necessary correction. Each ml of 0.1M perchloride acid is equivalent to 0.04481 g of $C_{27}H_{42}CINO_2$.

Bromine, 0.05M: Dissolve 3 g of potassium bromate and 15 g of potassium bromide in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

Pipette 25.0 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. Add 5 ml of potassium iodide solution, again insert the stopper and allow it to stand for 5 minutes in the dark. Titrate the liberated iodine with 0.1M sodium thiosulphate using 3 ml of starch solution, added towards the end of the titration, as indicator. Each ml of 0.1M sodium thiosulphate is equivalent to 0.01598 g of Br2.

Store in dark amber-coloured, glass stoppererd bottles.

Ceric Ammonium Nitrate, 0.1 M; Ammonium Ceric Nitrate, 0.1M: shake a solution containing 56 ml of sulphuric acid and 54.82 g of ceric ammonium nitrate for 2 minutes and carefully add five successive

quantities, each of 100 ml, f water, shaking after each addition. Dilute the clear solution to 1000 ml with water. After 10 days, standardise the solution as described under 0.1M ceric ammonium sulphate. Each ml of 0.1M ceric ammonium nitrate is equivalent to 0.004946 g of As_2O_3.
Store in light-resistant containers.

Cupric Sulphate, 0.02M: Dissolve 5.0 g of cupric sulphate in water and dilute to 1000 ml with water. Standardise the solution in the following manner.

To 20.0 ml add 2 g of sodum acetate and titrate with 0.02M disodium edetate, using 0.1 ml of pyridylazonaphthol solution as indicator, until the colour changes from violet-blue to bright green, adding the titrant slowly towards the end-point. Each ml of 0.02M disodium edetate is equivalent to 0.004994 g of $CuSO_4,5H_2O$.

Dioctyl Sodium Sulphosuccinate, 0.0005M: Dissolve 0.225 g of dicotyl sodium sulphosuccinate in warm water, cool and dilute to 1000 ml with water. Standardise the solution in the following manner.

To 25.0 ml add 25.0 ml of a solution containing 20% w/v of anhydrous sodium sulphate and 2% w/v sodium carbonate, 50 ml of chloroform and 1.5 ml of bromophenol blue solution and mix. Titrate with 0.01M tetrabutylammonium iodide until about 1 ml remains to be added for the end-point. Stopper the flask, shake vigorously for 2 minutes and continue the titration, in increments of 0.5 ml, shaking vigorusly and allowing the flask to stand for abut 10 seconds after each addition. Continue the titration until a blue colour just appears in the chloroform layer. Each ml of 0.01M tetrabutylammonium iodide is equivalent to 0.004446 g of $C_{20}H_{37}NaO_7S$.

Disodium Edetate, 0.1M: Dissolve 37.2 g of disodium edetate in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

Weigh accurately about 0.8 g of granulated zinc, dissolve by gentle warming in 12 ml of dilute hydrochloric acid and 0.1 ml of bromine water. Boil to remove excess bromine, cool and add sufficient water to produce 200.0 ml. Pipette 20.0 ml of the resulting solution into a flask and nearly neutralise with 2M sodium hydroxide. Dilute to about 150 ml with water, add sufficient ammonia buffer pH 10.0 to dissolve the precipitate and add 5 ml in excess. Add 50 mg of mordant black 11 mixture and titrate with the disodium edetate solution until the solution turns green. Each ml of 0.05M disodium edetate is equivalent to 0.000654 g of Zn.

Ferric Ammonium Sulphate, 0.1M: Ammonium Iron (III_ Sulphate, 0.1M: Dissolve 50 g of ferric ammonium sulphate in a mixture of 300 ml of water and 6 ml of sulphuric acid and dilute with sufficient freshly boiled and cooled water to produce 1000 ml. Standardise the solution in the following manner.

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To 25.0 ml add 3 ml of hydrochloric acid and 2 g of potassium iodide, allow to stand for 10 minutes and titrate the liberated iodine with 0.1M sodium thiosulphate using starch solution, added towards the end of the titration, as indicator. Each ml of 0.1M sodium thiosulphate is equivalent to 0.004822 g of $FeNH_4(SO_4)_2,12H_2O$.

Ferrous Ammonium Sulphate, 0.1 M; Ammonium Iron(II) Sulphate, 0.1 M; Dissolve 40 g of ferrous ammonium sulphate in a previously cooled mixture of 40 ml of sulphhuric acid and 200 ml of water, dilute with sufficient freshly boiled and cooled water to produce 1000 ml. Standardise the solution in the following manner. Measure accurately 25.0 ml of the solution into a flask, add 2 drops of 1,10-phenanthroline solution and titrate with 0.1 M ceric ammonium sulphate until the red colour is changed to pale blue. Each ml of 0.1 M ceric ammonium sulphate is equivalent to 0.03921g of Fe(NH4)2(SO4)2, 6H2O.

Hydrochloric Acid, 1M: Dilute 85 ml of hydrochloric acid with water to produce 1000 ml. Standardise the solution in the following manner.
Weigh accurately about 1.5 g of anhydrous sodium carbonate, previously heated at about 270o for 1 hour. Dissolve it in 100 ml of water and add 0.1 ml of methyl red solution. Add the acid slowly from a burette, 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 colour is no longer affected by continued boiling. Each ml of 1M hydrochloric acid is equivalent to 0.05299 g of Na2CO3.

Hydrochloric Acid, 0.05 M Methanolic: Take 40 ml of water in a 1000 ml volumetric flask and slowly add 43 ml of hydrochloric acid. Cool and add methanol to volume. Standardise the solution in the following manner.

Weigh accurately about 800 mg of anhydrous sodium carbonate, previously heated at about 270o for 1 hour, and proceed as directed under 1 M hydrochloric acid.

Iodine, 0.05 M: Dissolve about 14 g of iodine in a solution of 36 g of potassium iodide in 100 ml of water, add three drops of hydrochloric acid and dilute with water to 1000 ml. Standardise the solution in the following manner.
Weigh accurately about 0.15 g of arsenic trioxide, previously dried at 105o for 1 hour, and dissolve in 20 ml of 1M sodium hydroxide by warming, if necessary. Dilute with 40 ml of water, add 0.1 ml of methyl orange solution and add dropwise dilute hydrochloric acid until the yellow colour is changed to pink. Add 2 g of sodium carbonate, dilute with 50 ml of water and add 3 ml of starch solution. Titrate with the iodine solution until a permanent blue colour is produced. Each ml of 0.05 M iodine is equivalent to 0.004946 g of As2O3.
Store in amber-coloured, glass stoppered bottles.

Lead Nitrate, 0.1 M : Dissolve 33.12 g of lead nitrate in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

Pipette 50.0 ml of the solution into a flask, add 50 mg of xylenol orange mixture and sufficient hexamine to produce a violet-pink colour and titrate with 0.1M disodium edetate to a lemon-yellow end point. Each ml of 0.1 M disodium edetate is equivalent to 0.03312 g of $Pb(NO_3)_2$.

Lithium Methoxide, 0.1 M : Dissolve in small portions 0.7 g of freshly cut lithium in 150 ml of anhydrous methanol cooling the flask during the addition of the metal. When reaction is complete add sufficient toluene to produce 1000 ml. If cloudiness or precipitation occurs, add sufficient anhydrous methanol to clarify the solution. Standardise the solution immediately before use in the following manner.
Weigh accurately about 0.25 g of benzoic acid, dissolve in 25 ml of dimethylformamide and carry out Method B for non-aqueous titration, Appendix 3.45, using the lithium methoxide solution as the titrant and quinaldine red solution as the indicator and protecting the solution form atmospheric carbon dioxide throughout the titration. Perform a blank determination and make any necessary correction. Each ml of 0.1 M lithium methoxide is equivalent to 0.01221 g of $C_7H_6O_2$.
Store the solution in a manner suitably protected from carbon dioxide and moisture.

Magnesium Sulphate, 0.05 M : Dissolve 12.5 g of magnesium sulphate in sufficient water to produce 1000 ml. Standardise the solution in the following manner.
Dissolve 15 mg of sodium chloride in 50 ml of water and titrate with the mercuric nitrate solution determining the end point potentiometrically, usin ga platinum or mercury indicator electrode and a mercury-mercurous sulphate refernce electrode. Each ml of 0.02 M mercuric nitrate is equivalent to 0.002338 g of NaCl.

Nitric Acid, 1M: Dilute 63 ml of nitric acid with sufficient water to produce 1000 ml. Standardise the solution in the following manner.
Dissolve 2 g of anhydrous sodium carbonate in 50 ml of water and titrate with the nitric acid solution using methyl orange as indicator until the solution becomes reddish yellow. Boil for 2 minutes, cool and continue the titration until the reddish yellow colour is restored. Each ml of 1M nitric acid is equivalent to 0.0053 g of Na_2CO_3.

Perchloric Acid, 0.1 M: Mix 8.5 ml of perchloric acid with 500 ml of anhydrous glacial acetic acid and 25 ml of acetic anhydride, cool and add anhydrous glacial acetic acid to produce 1000 ml. Allow the prepared solution to stand for 1 day for the excess acetic anhydride to be continued and carry out the determination of water, Appendix 3.24. If the water content exceeds 0.05 % , add more acetic anhydride. If the solution contains no titrable water, add sufficient water to obtain a content of water between 0.02% and 0.05%. Allow the solution to stand for 1 day and again titrate the water content. The solution so obtained should contain between 0.02% and 0.05% of water. Standardise the solution in the following manner.
Weigh accurately about 0.35 g of potassium hydrogen phthalate, previously powdered lightly and dried at 120o for 2 hours and dissolve it in 50 ml of anhydrous glacial acetic acid. Add 0.1 ml of crystal violet solution and titrate with the perchloric acid solution until the violet colour changes to emerald-green. Peform a blank

determination and make any necessary correction. Each ml of 0.1 M perchloric acid is equivalent to 0.02042 g of $C_8H_5KO_4$.
Other strengths of perchloric acid should be prepared by diluting 0.1 M perchloric acid appropriately with anhydrous glacial acetic acid.
In the tests and assays of the Pharmacopoeia, this solution is specified as "0.1 N perchloric acid:. Thus the solution in anhydrous glacial acetic acid is to be used unless the words "in dioxan" are stated.

Potassium Dichromate, 0.0167 M : Weigh 4.9 g of potassium dichromate, previously powdered and dried in a desiccator for 4 hours, and dissolve in sufficient water to produce 1000 ml. Srandardise the solution in the following manner.
To 20.0 ml of the solution add 1 g of potassium iodide and 7 ml of 2M hydrochloric acid. Add 250 ml of water and titrate with 0.1 M sodium thiosulphate, using 3 ml of starch solution, added towards the end point of the titration, as indicator until the colour changes from blue to light green. Each ml of 0.1 M sodium thiosulphate is equivalent to 0.0049 g of $K_2Cr_2O_7$.

Potassium Hydrogen Phthalate, 0.005 M: Dissolve 10.21 g of potassium hydrogen phthalate in about 800 ml of anhydrous glacial acetic acid, heat on a water bath until completely dissolved, protected from humidity, cool to 20^o and add sufficient anhydrous glacial acetic acid to produce 1000 ml.

Potassium Hydroxide, 0.1 M: Dissolve about 6 g of potassium hydroxide in sufficient carbon dioxide free water to produce 1000 ml. Standardise the solution in the following manner.
Titrate 20.0 ml of the solution with 0.1 M hydrochloric acid using 0.5 ml of phenolphthalein solution as indicator. Each ml of 0.1 M hydrochloric acid is equivalent to 0.005611 g of KOH.

Potassium Hydroxide, 0.1 M Ethanolic: Dissolve about 6 g of potassium hydroxide in 5 ml of water and add sufficient aldehyde-free ethanol (95 %) to produce 1000 ml. Allow the solution to stand in a tightly stoppered bottle for 24 hours. Then quickly decant the clear supernatant liquid into a suitable, tightly closed container and standardise the solution in the following manner.
Pipette 20.0 ml of 0.1 M hydrochloric acid into a flask, dilute with 50 ml of water, add 0.1 ml of phenolphthalein solution and titrate with the ethanolic potassium hydroxide solution until a permanent pale pink colour is produced. Each ml of 0.1 M hydrochloric acid is equivalent to 0.00561 g of KOH.
Store in tightly-stoppered, light resistant bottles.

Potassium Hydroxide in ethanol (60%), 0.5 M : Dissolve 30 g of potassium hydroxide in sufficient ethanol (60 %) to produce 1000 ml. Standardise the solution in the following manner.
Pipette 20.0 ml of standardise 0.5 M hydrochloric acid into a flask, add 0.1 ml of phenolphthalein solution and titrate with the ethanolic potassium hydroxide solution until permanent pale-pink colour is produced. Each ml of 0.5 M hydrochloric acid is equivalent to 0.02806 g of KOH.

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Potassium Iodate, 0.05 M: Weigh accurately 10.7 g of potassium iodate, previously dried at 110o to constant weight, in sufficient water to produce 1000 ml. Standardise the solution in the following manner.
Dilute 25.0 ml of the solution to 100 ml with water and to 20.0 ml of this solution add 2 g of potassium iodide and 10 ml of 1 M sulphuric acid. Titrate with 0.1 M sodium thiosulphate using 1 ml of starch solution, added towards the end of the titration, as indicator. Each ml of 0.1 M sodium thiosulphate is equivalent to 0.03566 g of KIO_3.

Potassium Permanganate, 0.02 M : Dissolve 3.2 g of potassium permanganate in 1000 ml of water, heat on a water bath for 1 hour, allow to stand for 2 days and filter through glass wool. Standardise the solution in the following manner.
To 25.0 ml of the solution in a glass-stoppered flask add 2 g of potassium iodide, followed by 10 ml of 1 M sulphuric acid. Titrate the liberated iodine with 0.1 M sodium thiosulphate, using 3 ml of starch solution, added towards the end of the titration, as indicator. Perform a blank determination and make necessary correction. Each ml of 0.1 M sodium thiosulphate is equivalent to 0.003161 g of $KmnO_4$.
Store in light-resistant containers.

Silver Nitrate, 0.1 M: Disslove 17.0 g in sufficient water to produce 1000 m . standardise the solution in the following manner.
Weigh accurately about 0.1 g of sodium chloride, previously dried at 110^o for 2 hours and dissolve in 5 ml of water. Add 5 ml of acetic acid, 50 ml of methanol and 0.15 ml of eosin solution. Stir, preferably with magnetic stirrer, and titrate with the silver nitrate solution. Each ml of 0.1 M silver nitrate is equivalent to 0.005844 g of NaCl.
Store in light-resistant containers.

Sodium Dodecyl Sulphate, 0.001 M : Dissolve 0.2884 g of sodium dodecyl sulphate, calculated with reference to the substance dried at 105^o for 2 hours, in sufficient water to produce 1000 ml. Standardise the solution in the following manner.
To 50.0 ml add 15 ml of chloroform, 10 ml of 1M sulphuric acid and 1 ml of dimethyl yellow-oracet blut solution and titrate with 0.004M benzethonium chloride, shaking vigorously and allowing the layers to separate after each addition, until the chloroform layer acquires a permanent clear green colour. Each ml of 0.004 M benzethonium chloride is equivalent to 0.001154 g of $C_{12}H_{25}NaO_4S$.

Sodium Hydroxide, 1M: Dissolve 42 g of sodium hydroxide in sufficient carbon dioxide-free water to produce 1000 ml. Standardise the solution in the following manner.
Weigh accurately about 5 g of potassium hydrogen phthalate, previously powdered and dried at 120^o for 2 hours, and dissolve in 75 ml of carbon dioxide-free water. Add 0.1 ml of phenolphthalein solution and titrate

with the sodium hydroxide solution until a permanent pink colour is produced. Each ml of 1M sodium hydroxide is equivalent to 0.2042 g of $C_8H_5KO_4$.
Store in bottles with well-fitted suitable stoppers which prevent access to atmospheric carbon dioxide.
Volumetric Solutions of sodium hydroxide must be restandardise frequently. Solutions of lower concentrations are prepared by quantitatively diluting accurately measured volumes of 0.1 M sodium hydroxide with sufficient carbon dioxide-free water to give the desired concentration.

Sodium Hydroxide, 0.1 M Ethanolic: Dissolve 4.2 g of sodium hydroxide in 5 ml of water and add sufficient aldehyde-free ethanol to produce 1000 ml. Allow the solution to stand in a tightly-stoppered bottle for 24 hours. Then quickly decant the clear supernatant liquid into a suitable, tightly closed container. Standardise the solution in the following manner.
Weigh accurately about 0.6 g of benzoic acid, dissolve in a mixture of 30 ml of ethanol (95%) and 6 ml of water and titrate with the ethanolic sodium hydroxide solution, using 0.2 ml of thymolphthalein solution as indicator. Each ml of 0.1 M ethanolic sodium hydroxide is equivalent to 0.01221 of $C_7H_6O_2$.
Store in tightly-stoppered, light-resistant bottles.

Sodium Methoxide, 0.1 M: Cool 150 ml of anhydrous methanol in ice water and add, in small portions, about 2.5 g of freshly cut sodium. When the metal has dissolved, add sufficient toluene, previously dried over sodium wire, to produce 1000 ml. Standardise the solution in the following manner immediately before use.
Weigh accurately about 0.4 g of benzoic acid, dissolve in 80 ml of dimethylformamide, add 0.15 ml of thymolphthalein solution and titrate with sodium methoxide solution to a blue end point. Protect the solution from atmospheric carbon dioxide throughout the titration. Perform a blank determination and make any necessary correction. Each ml of 0.1 M sodium methoxide is equivalent to 0.01221 g of $C_7H_6O_2$.
Store in a container protected from carbon dioxide and moisture.

Sodium Nitrite, 0.1 M : Dissolve 7.5 g of sodium nitrite in sufficient water to produce 1000 ml. Standardise the solution in the following manner.
Dissolve 0.3 g of sulphanilic acid in 50 ml of 2 M hydrochloric acid, add 3 g of potassium bromide, cool in ice and titrate with the sodium nitrite solution determining the end-point potentiometrically. Each ml of 0.1 M sodium nitrite is equivalent to 0.01732 g of $C_6H_7NO_3S$.

Sodium Thiosulphate, 0.1M : Dissolve 25 g of sodium thiosulphate and 0.2 g of sodium carbonate in carbon dioxide-free water and dilute to 1000 ml with the same solvent. Standardise the solution in the following manner.

Dissolve 0.200 g of potassium bromate, weighed accurately, in sufficient water to produce 250.0 ml. To 50.0 ml of this solution add 2 g of potassium iodide and 3 ml of 2M hydrochloric acid and titrate with the sodium

thiosulphate solution using starch solution, added towards the end of the titration, as indicator until the blue colour is discharged. Each ml of 0.1M sodium thiosulphate is equivalent to 0.002784 g of $KbrO_3$.

Restandardise the solution frequently.

Sulphuric Acid, 0.5M: Add slowly, with stirring, 30 ml of sulphuric acid to about 1000 ml of water, allow to cool 25° and standardise against anhydrous sodium carbonate as described under 1M hydrochloric acid. Each ml of 0.5M sulphuric acid is equivalent to 0.05299 g of Na_2CO_3.

Sulphuric Acid, 0.25M Ethanolic: Add slowly, with stirring, 13.9 ml of sulphuric acid to a sufficient quantity of ethanol to produce 1000 ml. Cool and standardise against anhydrous sodium carbonate as described under 0.5M methanolic hydrochloric acid.

Tetrabutylammonium Hydroxide, 0.1M: Dissolve 40 g of tetrabutylammonium iodide in 90 ml of dehydrated methanol in a glass-stoppered flask. Place in an ice-bath, add 20 g of powdered silver oxide, insert the stopper and agitate vigorously for 1 hour. Centrifuge a few ml, and test the supernatant liquid for iodides, Appendix 3.1 and test is positive, add an additional 2 g of silver oxide and continue to stand for 30 minutes with intermittent agitation. When all of the iodide has reacted, filter through fine sintered-glass filter. Rinse the flask and filter with three quantities, each of 50 ml, of anhydrous toluene. Add the washings of the filtrate and dilute to 1000 ml with anhydrous toluene. Flush the solution for 10 minutes with dry, carbon dioxide-free nitrogen. Store in a container protected from carbon dioxide and moisture, and discard after 60 days.

Alternatively, prepare the solution by diluting a suitable volume of commercially available tetrabutylammonium hydroxide solution in methanol with a mixture of four volumes of anhydrous toluene and 1 volume of dehydrated methanol.

Standardise the solution in the following manner immediately before use.

Weigh accurately about 0.4 g of benzoic acid, dissolve in 80 ml of dimethylformamide, add a few drops of a 1% w/v solution of thymol blue in dimethylformamide and titrate with the tetrabutylammonium hydroxide solution to a blue end-point. Protect the solution from atmospheric carbon dioxide throughout the titration. Perform a blank determination and make any necessary correction. Each ml of 0.1M tetrabutylammonium hydroxide is equivalent to 0.01221 g of $C_7H_6O_2$.

Zinc Chloride, 0.1M: Dissolve 6.6 g of granulated zinc, previously washed with 0.1M hydrochloric acid and then with water, in the minimum amount of 2M hydrochloric acid and add sufficient water to produce 1000 ml. Standardise the solution in the following manner.

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To 25.0 ml of the solution add 4 g of ammonium acetate and 25 ml of water. Add 50 mg of xylenol orange mixture and sufficient hexamine, and titrate with 0.1M disodium edetate until the colour changes to yellow. Each ml of 0.1M disodium edetate is equivalent to 0.013630 g of $ZnCl_2$.

Zinc Sulphate, 0.1M: Dissolve 29 g of zinc sulphate in sufficient water to produce 1000 ml. Standardise the solution in the following manner.

To 20.0 ml add 5 ml of 2M acetic acid and carry out the method for the determination of zinc, Appendix 3.25. Each ml of 0.1M disodium edetate is equivalent to 0.02875 g of $ZnSO_4,7H_2O$.