dilute to 500.0 ml with the same solvent. Adjust to pH 5.0 to 5.5 with a 335 g/l solution of sodium hydroxide R and dilute to 1000.0 ml with distilled water R.

**Total-ionic-strength-adjustment buffer R1. 4008800.**

Solution (a). Dissolve 210 g of citric acid R in 400 ml of distilled water R. Adjust to pH 7.0 (2.2.3) with concentrated ammonia R. Dilute to 1000.0 ml with distilled water R.

Solution (b). Dissolve 132 g of ammonium phosphate R in distilled water R and dilute to 1000.0 ml with the same solvent.

Solution (c). To a suspension of 292 g of (ethylenedinitrilo)tetra-acetic acid R in about 500 ml of distilled water R, add about 200 ml of concentrated ammonia R to dissolve. Adjust the pH to 6 to 7 (2.2.3) with concentrated ammonia R. Dilute to 1000.0 ml with distilled water R.

Mix equal volumes of solution (a), (b), and (c) and adjust to pH 7.5 with concentrated ammonia R.

### 4.2. VOLUMETRIC ANALYSIS

**4.2.1. PRIMARY STANDARDS FOR VOLUMETRIC SOLUTIONS**

Primary standards for volumetric solutions are indicated by the suffix RV. Primary standards of suitable quality may be obtained from commercial sources or prepared by the following methods.

**Arsenious trioxide.** As₂O₃. (Mₙ 197.8). 2000100. [1327-53-3].

Sublime arsenious trioxide R in a suitable apparatus.

**Storage:** over anhydrous silica gel R.

**Benzoic acid.** C₆H₅CO₂H. (Mₙ 122.1). 2000200. [65-85-0].

Sublime benzoic acid R in a suitable apparatus.

**Potassium bromate.** KBrO₃. (Mₙ 167.0). 2000300. [7758-01-2].

Crystallise potassium bromate R from boiling water R. Collect the crystals and dry to constant mass at 180 °C.

**Potassium hydrogen phthalate.** C₈H₅KO₄. (Mₙ 204.2). 2000400. [877-24-7].

Recrystallise potassium hydrogen phthalate R from boiling water R, collect the crystals at a temperature above 35 °C and dry to constant mass at 110 °C.

**Sodium carbonate.** Na₂CO₃. (Mₙ 106.0). 2000500. [497-19-8].

Filter at room temperature a saturated solution of sodium carbonate R. Introduce slowly into the filtrate a stream of carbon dioxide R with constant cooling and stirring. After about 2 h, collect the precipitate on a sintered-glass filter (2.1.2). Wash the filter with iced water R containing carbon dioxide. After drying at 100 °C to 105 °C, heat to constant mass at 270 °C to 300 °C, stirring from time to time.

**Sodium chloride.** NaCl. (Mₙ 58.44). 2000600. [7647-14-5].

To 1 volume of a saturated solution of sodium chloride R add 2 volumes of hydrochloric acid R. Collect the crystals formed and wash with hydrochloric acid R. Remove the hydrochloric acid by heating on a water-bath and dry the crystals to constant mass at 300 °C.

**Sulphanilic acid.** C₆H₇NO₃S. (Mₙ 173.2). 2000700. [121-57-3].

Recrystallise sulphanilic acid R from boiling water R. Filter and dry to constant mass at 100 °C to 105 °C.

**Zinc.** Zn. (Mₙ 65.4). 2000800. [7440-66-4].

Use a quality containing not less than 99.9 per cent of Zn.

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### 4.2.2. VOLUMETRIC SOLUTIONS

Volumetric solutions are prepared according to the usual chemical analytical methods. The accuracy of the apparatus used is verified to ensure that it is appropriate for the intended use.

The concentration of volumetric solutions is indicated in terms of molarity. Molarity expresses, as the number of moles, the amount of substance dissolved in 1 litre of solution. A solution which contains x moles of substance per litre is said to be x M.

Volumetric solutions do not differ from the prescribed strength by more than 10 per cent. The molarity of the volumetric solutions is determined by an appropriate number of titrations. The repeatability does not exceed 0.2 per cent (relative standard deviation).

Volumetric solutions are standardised by the methods described below. When a volumetric solution is to be used in an assay in which the end-point is determined by an electrochemical process (for example, amperometry or potentiometry) the solution is standardised by the same method. The composition of the medium in which a volumetric solution is standardised should be the same as that in which it is to be used.

Solutions more dilute than those described are obtained by dilution with carbon dioxide-free water R of the least-concentrated solution that describes a standardisation. The correction factors of these solutions are the same as those from which the dilutions were prepared.

**0.1 M Acetic acid.** 3008900.

Dilute 6.0 g of glacial acetic acid R to 1000.0 ml with water R.

**Standardisation.** To 25.0 ml of acetic acid add 0.5 ml of phenolphthalein solution R and titrate with 0.1 M sodium hydroxide.

**0.1 M Ammonium and cerium nitrate.** 3000100.

Shake for 2 min a solution containing 56 ml of sulphuric acid R and 54.82 g of ammonium and cerium nitrate R, add five successive quantities, each of 100 ml, of water R, shaking after each addition. Dilute the clear solution to 1000.0 ml with water R. Standardise the solution after 10 days.

**Standardisation.** To 25.0 ml of the ammonium and cerium nitrate solution add 2.0 g of potassium iodide R and 150 ml of water R. Titrate immediately with 0.1 M sodium thiosulphate, using 1 ml of starch solution R as indicator.

**Storage:** protected from light.

**0.01 M Ammonium and cerium nitrate.** 3000200.

To 100.0 ml of 0.1 M ammonium and cerium nitrate R, add with cooling, 30 ml of sulphuric acid R and dilute to 1000.0 ml with water R.

**0.1 M Ammonium and cerium sulphate.** 3000300.

Dissolve 65.0 g of ammonium and cerium sulphate R in a mixture of 500 ml of water R and 30 ml of sulphuric acid R. Allow to cool and dilute to 1000.0 ml with water R.
0.01 M Ammonium and cerium sulphate. 3000400.
To 100.0 ml of 0.1 M ammonium and cerium sulphate add, with cooling, 30 ml of sulphuric acid R and dilute to 1000.0 ml with water R.

0.1 M Ammonium thiocyanate. 3000500.
Dissolve 7.612 g of ammonium thiocyanate R in water R and dilute to 1000.0 ml with the same solvent.

0.05 M Barium perchlorate. 3000700.
Dissolve 15.8 g of barium hydroxide R in a mixture of 7.5 ml of perchloric acid R and 75 ml of water R. Adjust the solution to pH 3 by adding perchloric acid R and filter if necessary. Add 150 ml of alcohol R and dilute to 250 ml with water R. Dilute to 1000.0 ml with buffer solution pH 3.7 R.

0.025 M Barium perchlorate. 3009600.
Dilute 500.0 ml of 0.05 M barium perchlorate to 1000.0 ml with buffer solution pH 3.7 R.

0.004 M Benzethonium chloride. 3000900.
Dissolve in water R 1.792 g of benzethonium chloride R, previously dried to constant mass at 100 °C to 105 °C, and dilute to 1000.0 ml with the same solvent.

0.01 M Bismuth nitrate. 3010000.
Dissolve 4.86 g of bismuth nitrate pentahydrate R in 60 ml of dilute nitric acid R and dilute to 1000.0 ml with water R.

0.0167 M Bromide-bromate. 3001000.
Dissolve 2.7835 g of potassium bromate R and 13 g of potassium bromide R in water R and dilute to 1000.0 ml with the same solvent.

0.1 M Cerium sulphate. 3001100.
Dissolve 40.4 g of cerium sulphate R in a mixture of 500 ml of water R and 50 ml of sulphuric acid R. Allow to cool and dilute to 1000.0 ml with water R.

0.02 M Copper sulphate. 3001200.
Dissolve 5.0 g of copper sulphate R in water R and dilute to 1000.0 ml with the same solvent.

0.1 M Ferric ammonium sulphate. 3001300.
Dissolve 50.0 g of ferric ammonium sulphate R in a mixture of 6 ml of sulphuric acid R and 300 ml of water R and dilute to 1000.0 ml with water R.

0.1 M Hydrochloric acid. 3001800.
Dilute 103.0 g of hydrochloric acid R to 1000.0 ml with water R.

0.1 M Hydrochloric acid, alcoholic. 3008800.
Dilute 9.0 ml of hydrochloric acid R to 1000.0 ml with aldehyde-free alcohol R.
0.5 M Iodine. 3009400.
Dissolve 127 g of iodine R and 200 g of potassium iodide R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. To 2.0 ml of the iodine solution add 1 ml of dilute acetic acid R and 50 ml of water R. Titrate with 0.1 M sodium thiosulphate, using starch solution R as indicator.
Storage: protected from light.

0.05 M Iodine. 3002700.
Dissolve 12.7 g of iodine R and 20 g of potassium iodide R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. To 20.0 ml of the iodine solution add 1 ml of dilute acetic acid R and 30 ml of water R. Titrate with 0.1 M sodium thiosulphate, using starch solution R as indicator.
Storage: protected from light.

0.01 M Iodine. 3002900.
Add 0.3 g of potassium iodide R to 20.0 ml of 0.05 M iodine and dilute to 100.0 ml with water R.

0.1 M Lanthanum nitrate. 3010100.
Dissolve 43.30 g of lanthanum nitrate R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. To 20 ml of the lanthanum nitrate solution, add 15 ml of water R and 25 ml of 0.1 M sodium edetate. Add about 50 mg of xylenol orange triturate R and about 2 g of hexamethylenetetramine R. Titrate with 0.1 M zinc sulphate until the colour changes from yellow to violet-pink.

0.1 M Lead nitrate. 3003100.
Dissolve 33 g of lead nitrate R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. Take 20.0 ml of the lead nitrate solution and carry out the determination of lead by complexometry (2.5.11).

0.05 M Lead nitrate. 3009700.
Dilute 50.0 ml of 0.1 M Lead nitrate to 100.0 ml with water R.

0.1 M Lithium methoxide. 3003300.
Dissolve 0.694 g of lithium R in 150 ml of anhydrous methanol R and dilute to 1000.0 ml with toluene R.
Standardisation. To 10 ml of dimethylformamide R add 0.05 ml of a 3 g/l solution of thymol blue R in methanol R and titrate with the lithium methoxide solution until a pure blue colour is obtained. Immediately add 0.200 g of benzoic acid RV to the solution and titrate with the lithium methoxide solution until the blue colour is again obtained. Protect the solution from atmospheric carbon dioxide throughout the titration. From the volume of titrant used in the second titration ascertain the exact strength of the lithium methoxide solution. Standardise immediately before use.
1 ml of 0.1 M lithium methoxide is equivalent to 12.21 mg of C7H6O2.

0.1 M Magnesium chloride. 3003400.
Dissolve 20.33 g of magnesium chloride R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. Carry out the determination of magnesium by complexometry (2.5.11).

1 M Nitric acid. 3003600.
Dilute 96.6 g of nitric acid R to 1000.0 ml with water R.

Standardisation. Dissolve 1.000 g of sodium carbonate RV in 50 ml of water R, add 0.1 ml of methyl orange solution R and titrate with the nitric acid until the solution just becomes reddish-yellow; boil for 2 min. The solution reverts to yellow. Cool and continue the titration until a reddish-yellow colour is obtained.
1 ml of 1 M nitric acid is equivalent to 53.00 mg of Na2CO3.

0.1 M Perchloric acid. 3003900.
Place 8.5 ml of perchloric acid RV in 50 ml of anhydrous acetic acid R, warming gently if necessary. Allow to cool protected from the air, and titrate with the perchloric acid solution, using 0.05 ml of crystal violet solution R as indicator. Note the temperature of the perchloric acid solution at the time of the titration. If the temperature at which an assay is carried out is different from that at which the 0.1 M perchloric acid has been standardised, the volume used in the assay becomes:

\[ V_c = V_i + (t_i - t_f) \times 0.0011 \]

where \( t_i \) is the temperature during standardisation, \( t_f \) is the temperature during the assay, \( V_i \) is the corrected volume, and \( V \) is the observed volume.
1 ml of 0.1 M perchloric acid is equivalent to 20.42 mg of C8H5KO4.

0.05 M Perchloric acid. 3004000.
Dilute 50.0 ml of 0.1 M perchloric acid to 100.0 ml with anhydrous acetic acid R.

0.02 M Perchloric acid. 3009900.
Dilute 20.0 ml of 0.1 M perchloric acid to 100.0 ml with anhydrous acetic acid R.

0.033 M Potassium bromate. 3004200.
Dilute 5.5670 g of potassium bromate RV in water R and dilute to 1000.0 ml with the same solvent.

0.02 M Potassium bromate. 3004300.
Dilute 3.340 g of potassium bromate RV in water R and dilute to 1000.0 ml with the same solvent.

0.0167 M Potassium bromate. 3004400.
Prepare by diluting 0.033 M Potassium bromate.

0.0083 M Potassium bromate. 3004500.
Prepare by diluting 0.033 M Potassium bromate.

0.0167 M Potassium dichromate. 3004600.
Dilute 4.90 g of potassium dichromate R in water R and dilute to 1000.0 ml with the same solvent.
Standardisation. To 20.0 ml of the potassium dichromate solution add 1 g of potassium iodide R and 7 ml of dilute hydrochloric acid R. Add 250 ml of water R and titrate with 0.1 M sodium thiosulphate, using 3 ml of starch solution R as indicator, until the colour changes from blue to light green.
Dilute 2.0 ml of 0.01 M Potassium hydroxide, alcoholic.

Dilute 20.0 ml of 0.1 M Potassium hydroxide, alcoholic.

0.5 ml of 0.5 M alcoholic potassium hydroxide and dilute to 100.0 ml with anhydrous acetic acid R.

Dissolve 3 g of 0.5 M Potassium hydroxide, alcoholic.

Standardisation. Titrate 20.0 ml of the potassium hydroxide solution with 1 M hydrochloric acid, using 0.5 ml of phenolphthalein solution R as indicator.

0.1 M Potassium hydroxide. 3004800.

Dissolve 6 g of potassium hydroxide R in carbon dioxide-free water R and dilute to 1000.0 ml with the same solvent.

Standardisation. Titrate 20.0 ml of the potassium hydroxide solution with 0.1 M hydrochloric acid, using 0.5 ml of phenolphthalein solution R as indicator.

0.5 M Potassium hydroxide in alcohol (60 per cent V/V). 3004900.

Dissolve 3 g of potassium hydroxide R in aldehyde-free alcohol R (60 per cent V/V) and dilute to 100.0 ml with the same solvent.

Standardisation. Titrate 20.0 ml of the alcholic potassium hydroxide solution (60 per cent V/V) with 0.5 M hydrochloric acid, using 0.5 ml of phenolphthalein solution R as indicator.

0.5 M Potassium hydroxide, alcoholic. 3005000.

Dissolve 3 g of potassium hydroxide R in 5 ml of water R and dilute to 100.0 ml with aldehyde-free alcohol R.

Standardisation. Titrate 20.0 ml of the alcholic potassium hydroxide solution with 0.5 M hydrochloric acid, using 0.5 ml of phenolphthalein solution R as indicator.

0.1 M Potassium hydroxide, alcoholic. 3005100.

Dilute 20.0 ml of 0.5 M alcoholic potassium hydroxide to 100.0 ml with aldehyde-free alcohol R.

0.01 M Potassium hydroxide, alcoholic. 3009000.

Dilute 2.0 ml of 0.5 M alcoholic potassium hydroxide to 100.0 ml with aldehyde-free alcohol R.

0.05 M Potassium iodate. 3005200.

Dissolve 10.70 g of potassium iodate R in water R and dilute to 1000.0 ml with the same solvent.

Standardisation. Dilute 25.0 ml of the potassium iodate solution to 100.0 ml with water R. To 20.0 ml of this solution add 2 g of potassium iodide R and 10 ml of dilute sulphuric acid R. Titrate with 0.1 M sodium thiosulphate, using 1 ml of starch solution R, added towards the end of the titration, as indicator.

0.001 M Potassium iodide. 3009200.

Dilute 10.0 ml of potassium iodide solution R (166 g/l) to 100.0 ml with water R. Dilute 5.0 ml of this solution to 500.0 ml with water R.

0.02 M Potassium permanganate. 3005300.

Dissolve 3.2 g of potassium permanganate R in water R and dilute to 1000.0 ml with the same solvent. Heat the solution for 1 h on a water-bath, allow to cool and filter through a sintered-glass filter (2.1.2).

Standardisation. To 20.0 ml of the potassium permanganate solution, add 2 g of potassium iodide R and 10 ml of dilute sulphuric acid R. Titrate with 0.1 M sodium thiosulphate, using 1 ml of starch solution R, added towards the end of the titration, as indicator. Standardise immediately before use.

Storage: protected from light.

0.1 M Silver nitrate. 3005600.

Dissolve 17.0 g of silver nitrate R in water R and dilute to 1000.0 ml with the same solvent.

Standardisation. Dissolve 0.100 g of sodium chloride R in 30 ml of water R. Titrate with the silver nitrate solution, determining the end-point potentiometrically (2.2.20).

1 ml of 0.1 M silver nitrate is equivalent to 5.844 mg of NaCl.

Storage: protected from light.

0.001 M Silver nitrate. 3009300.

Dilute 5.0 ml of silver nitrate R to 1000.0 ml with water R.

0.1 M Sodium arsenite. 3005800.

Dissolve arsenious trioxide R equivalent to 4.946 g of As₂O₃ in a mixture of 20 ml of strong sodium hydroxide solution R and 20 ml of water R, dilute to 200 ml with water R and add dilute hydrochloric acid R until the solution is neutral to litmus paper R. Dissolve 2 g of sodium hydrogen carbonate R in the solution and dilute to 500.0 ml with water R.

0.1 M Sodium edetate. 3005900.

Dissolve 37.5 g of sodium edetate R in 500 ml of water R, add 100 ml of 1 M sodium hydroxide and dilute to 1000.0 ml with water R.

Standardisation. Dissolve 0.120 g of zinc R in 4 ml of hydrochloric acid R and add 0.1 ml of bromine water R. Drive off the excess of bromine by boiling, add dilute sodium hydroxide solution R until the solution is weakly acid or neutral and carry out the assay of zinc by complexometry (2.5.11).

1 ml of 0.1 M sodium edetate is equivalent to 6.54 mg of Zn.

Storage: in a polyethylene container.

0.02 M Sodium edetate. 3006000.

Dissolve 7.444 g of sodium edetate R in water R and dilute to 1000.0 ml with the same solvent.

Standardisation. Dissolve 0.100 g of zinc R in 4 ml of hydrochloric acid R and add 0.1 ml of bromine water R. Drive off the excess of bromine by boiling. Transfer the solution to a volumetric flask and dilute to 100.0 ml with water R. Transfer 25.0 ml of the solution to a 500 ml conical flask and add 0.1 ml of xylene orange triturate R and hexamethylenetetramine R until the solution becomes violet-pink. Add 2 g of hexamethylenetetramine R in excess. Titrate with the sodium edetate solution until the violet-pink colour changes to yellow.

1 ml of 0.02 M sodium edetate is equivalent to 1.308 mg of Zn.

1 M Sodium hydroxide. 3006300.

Dissolve 42 g of sodium hydroxide R in carbon dioxide-free water R and dilute to 1000.0 ml with the same solvent.

Standardisation. Titrate 20.0 ml of the sodium hydroxide solution with 1 M hydrochloric acid using the indicator prescribed in the assay in which 1 M sodium hydroxide is used.
If sodium hydroxide free from carbonate is prescribed, prepare it as follows. Dissolve sodium hydroxide \( R \) in water \( R \) to give a concentration of 400 g/1 to 600 g/1 and allow to stand. Decant the clear supernatant liquid, taking precautions to avoid the introduction of carbon dioxide, and dilute with carbon dioxide-free water \( R \) to the required molarity. The solution complies with the following test. Titrate 20.0 ml of hydrochloric acid of the same molarity with the solution of sodium hydroxide, using 0.5 ml of phenolphthalein solution \( R \) as indicator. At the end-point add just sufficient of the acid to discharge the pink colour and concentrate the solution to 20 ml by boiling. During boiling add just sufficient acid to discharge the pink colour, which should not reappear after prolonged boiling. The volume of acid used does not exceed 0.1 ml.

**0.1 M Sodium hydroxide. 3006600.**

Dilute 100.0 ml of 1 M sodium hydroxide to 1000.0 ml with carbon dioxide-free water \( R \).

*Standardisation.* Titrate 20.0 ml of the sodium hydroxide solution with 0.1 M hydrochloric acid, using the end-point detection prescribed for the assay in which the 0.1 M sodium hydroxide is used.

*Standardisation (for use in the assay of halide salts of organic bases).* Dissolve 0.100 g of benzoic acid \( R \) in a mixture of 5 ml of 0.01 M hydrochloric acid and 50 ml of alcohol \( R \). Carry out the titration (2.2.20), using the sodium hydroxide solution. Note the volume added between the 2 points of inflexion.

1 ml of 0.1 M sodium hydroxide is equivalent to 12.21 mg of \( C_6H_5O_2 \). 

**0.1 M Sodium hydroxide, ethanolic. 3007000.**

To 250 ml of ethanol \( R \) add 3.3 g of strong sodium hydroxide solution \( R \).

*Standardisation.* Dissolve 0.100 g of benzoic acid \( R \) in 2 ml of water \( R \) and 10 ml of alcohol \( R \). Titrate with the ethanolic sodium hydroxide solution, using 0.2 ml of phenolphthalein solution \( R \) as indicator. Standardise immediately before use.

1 ml of 0.1 M ethanolic sodium hydroxide is equivalent to 12.21 mg of \( C_6H_5O_2 \).

**0.1 M Sodium methoxide. 3007100.**

Cool 175 ml of anhydrous methanol \( R \) in iced water \( R \) and add, in small portions, about 2.5 g of freshly cut sodium \( R \). When the metal has dissolved, dilute to 1000.0 ml with toluene \( R \).

*Standardisation.* To 10 ml of dimethylformamide \( R \) add 0.05 ml of a 3 g/1 solution of thymol blue \( R \) in methanol \( R \), and titrate with the sodium methoxide solution until a pure blue colour is obtained. Immediately add 0.200 g of benzoic acid \( R \). Stir to effect solution and titrate with the sodium methoxide solution until the pure blue colour is again obtained. Protect the solution from atmospheric carbon dioxide throughout the titration. From the volume of titrant used in the second titration ascertain the exact strength of the sodium methoxide solution. Standardise immediately before use.

1 ml of 0.1 M sodium methoxide is equivalent to 12.21 mg of \( C_6H_5O_2 \).

**0.1 M Sodium nitrite. 3007200.**

Dissolve 7.5 g of sodium nitrite \( R \) in water \( R \) and dilute to 1000.0 ml with the same solvent.

*Standardisation.* Dissolve 0.300 g of sulphamic acid \( R \) in 50 ml of dilute hydrochloric acid \( R \) and carry out the determination of primary aromatic amino-nitrogen (2.5.8), using the sodium nitrite solution and determining the end-point electrometrically. Standardise immediately before use.

1 ml of 0.1 M sodium nitrite is equivalent to 17.32 mg of \( C_7H_6NO_2S \). 

**0.1 M Sodium periodate. 3009500.**

Dissolve 21.4 g of sodium periodate \( R \) in about 500 ml of water \( R \) and dilute to 1000.0 ml with the same solvent. 

*Standardisation.* In a stoppered flask, introduce 20.0 ml of the sodium periodate solution and add 5 ml of perchloric acid \( R \). Close the flask and shake. Adjust the solution to pH 6.4 (2.2.3) using a saturated solution of sodium hydrogen carbonate \( R \). Add 10 ml of potassium iodide solution \( R \), close, shake and allow to stand for 2 min. Titrate with 0.025 M sodium arsenite until the yellow colour almost disappears. Add 2 ml of starch solution \( R \) and titrate slowly until the colour is completely discharged.

**0.1 M Sodium thiosulphate. 3007300.**

Dissolve 25 g of sodium thiosulphate \( R \) and 0.2 g of sodium carbonate \( R \) in carbon dioxide-free water \( R \) and dilute to 1000.0 ml with the same solvent.

*Standardisation.* To 10.0 ml of 0.033 M potassium bromate \( R \), add 40 ml of water \( R \), 10 ml of potassium iodide solution \( R \) and 5 ml of hydrochloric acid \( R \). Titrate with the sodium thiosulphate solution, using 1 ml of starch solution \( R \), added towards the end of the titration, as indicator.

**0.5 M Sulphuric acid. 3007800.**

Dilute 28 ml of sulphuric acid \( R \) in water \( R \) and dilute to 1000.0 ml with the same solvent.

*Standardisation.* Dissolve 1.000 g of sodium carbonate \( R \) in 50 ml of water \( R \), add 0.1 ml of methyl orange solution \( R \), and titrate with the sulphuric acid until the solution begins to turn reddish-yellow. Boil for about 2 min. The colour of the solutions reverts to yellow. Cool and titrate again until the reddish-yellow colour reappears.

1 ml of 0.5 M sulphuric acid is equivalent to 53.00 mg of \( Na_2CO_3 \).

**0.05 M Sulphuric acid. 3008000.**

Dilute 100.0 ml of 0.5 M sulphuric acid to 1000.0 ml with water \( R \).

*Standardisation.* Carry out the titration described for 0.5 M sulphuric acid, using 0.100 g of sodium carbonate \( R \), dissolved in 20 ml of water \( R \).

1 ml of 0.05 M sulphuric acid is equivalent to 5.30 mg of \( Na_2CO_3 \).

**0.1 M Tetrabutylammonium hydroxide. 3008300.**

Dissolve 40 g of tetrabutylammonium iodide \( R \) in 90 ml of anhydrous methanol \( R \), add 20 g of finely powdered silver oxide \( R \) and shake vigorously for 1 h. Centrifuge a few millilitres of the mixture and test the supernatant liquid for iodides. If a positive reaction is obtained, add an additional 2 g of silver oxide \( R \) and shake for a further 30 min. Repeat this procedure until the liquid is free from iodides, filter the mixture through a fine sintered-glass filter (2.1.2) and rinse the reaction vessel and filter with three quantities of each of 50 ml of tolune \( R \). Add the washings to the filtrate and dilute to 1000.0 ml with tolune \( R \). Pass dry carbon dioxide-free nitrogen through the solution for 5 min.

*Standardisation.* To 10 ml of dimethylformamide \( R \) add 0.05 ml of a 3 g/1 solution of thymol blue \( R \) in methanol \( R \) and titrate with the tetrabutylammonium hydroxide solution until a pure blue colour is obtained. Immediately add 0.200 g of benzoic acid \( R \). Stir to effect solution, and titrate with the tetrabutylammonium hydroxide solution until the pure
blue colour is again obtained. Protect the solution from atmospheric carbon dioxide throughout the titration. From the volume of titrant used in the second titration ascertain the exact strength of the tetrabutylammonium hydroxide solution. Standardise immediately before use.

1 ml of 0.1 M tetrabutylammonium hydroxide is equivalent to 12.21 mg of C7H6O2.

**0.1 M Tetrabutylammonium hydroxide in 2-propanol.**

3008400.

Prepare as described for 0.1 M tetrabutylammonium hydroxide using 2-propanol R instead of toluene R and standardise as described.

**0.05 M Zinc chloride.** 3008500.

Dissolve 6.82 g of zinc chloride R, weighed with appropriate precautions, in water R. If necessary, add dropwise dilute hydrochloric acid R until the opalescence disappears. Dilute to 1000.0 ml with water R.

*Standardisation.* To 20.0 ml of the zinc chloride solution add 5 ml of dilute acetic acid R and carry out the determination of zinc by complexometry (2.5.11).

**0.1 M Zinc sulphate.** 3008600.

Dissolve 29 g of zinc sulphate R in water R and dilute to 1000.0 ml with the same solvent.

*Standardisation.* To 20.0 ml of the zinc sulphate solution add 5 ml of dilute acetic acid R and carry out the determination of zinc by complexometry (2.5.11).