

7 with *concentrated ammonia R*. Dilute to 1000.0 mL with *distilled water R* (solution C). Mix equal volumes of solution A, B, and C and adjust to pH 7.5 with *concentrated ammonia R*.

4.2. VOLUMETRIC ANALYSIS

04/2010:40201

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. As_2O_3 . (M_r 197.8). 2000100. [1327-53-3].
Sublime *arsenious trioxide R* in a suitable apparatus.
Storage: over *anhydrous silica gel R*.

Benzoic acid. $\text{C}_7\text{H}_6\text{O}_2$. (M_r 122.1). 2000200. [65-85-0].
Sublime *benzoic acid R* in a suitable apparatus.

Potassium bromate. KBrO_3 . (M_r 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. $\text{C}_8\text{H}_5\text{KO}_4$. (M_r 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. Na_2CO_3 . (M_r 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 270-300 °C, stirring from time to time.

Sodium chloride. NaCl . (M_r 58.44). 2000600. [7647-14-5].
To 1 volume of the *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.

Sulfanilic acid. $\text{C}_6\text{H}_7\text{NO}_3\text{S}$. (M_r 173.2). 2000700. [121-57-3].
Recrystallise *sulfanilic acid R* from boiling *water R*. Filter and dry to constant mass at 100-105 °C.

Zinc. Zn . (M_r 65.4). 2000800. [7440-66-6].
Content: minimum 99.9 per cent.

04/2010:40202

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 L 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 sulfuric 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 thiosulfate*, 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* add, with cooling, 30 mL of sulfuric acid R and dilute to 1000.0 mL with *water R*.

0.1 M Ammonium and cerium sulfate. 3000300.

Dissolve 65.0 g of *ammonium and cerium sulfate R* in a mixture of 500 mL of *water R* and 30 mL of sulfuric acid R. Allow to cool and dilute to 1000.0 mL with *water R*.

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

0.01 M Ammonium and cerium sulfate. 3000400.

To 100.0 mL of 0.1 M *ammonium and cerium sulfate* add, with cooling, 30 mL of sulfuric 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.

Standardisation. To 20.0 mL of 0.1 M *silver nitrate* add 25 mL of *water R*, 2 mL of *dilute nitric acid R* and 2 mL of *ferric ammonium sulfate solution R2*. Titrate with the ammonium thiocyanate solution until a reddish-yellow colour is obtained.

0.1 M Barium chloride. 3000600.

Dissolve 24.4 g of *barium chloride R* in *water R* and dilute to 1000.0 mL with the same solvent.

Standardisation. To 10.0 mL of the barium chloride solution add 60 mL of *water R*, 3 mL of *concentrated ammonia R* and 0.5-1 mg of *phthalein purple R*. Titrate with 0.1 M *sodium edetate*. When the solution begins to decolorise, add 50 mL of *ethanol (96 per cent) R* and continue the titration until the blue-violet colour disappears.

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 *ethanol (96 per cent) R* and dilute to 250 mL with *water R*. Dilute to 1000.0 mL with *buffer solution pH 3.7 R*.

Standardisation. To 5.0 mL of 0.05 M *sulfuric acid* add 5 mL of *water R*, 50 mL of *buffer solution pH 3.7 R* and 0.5 mL of *alizarin s solution R*. Titrate with the barium perchlorate solution until an orange-red colour appears. Standardise immediately before use.

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 100-105 °C, and dilute to 1000.0 mL with the same solvent.

Standardisation. Calculate the molarity of the solution from the content of $C_{27}H_{42}ClNO_2$ in the dried benzethonium chloride determined as follows. Dissolve 0.350 g of the dried substance in 30 mL of *anhydrous acetic acid R* and add 6 mL of *mercuric acetate solution R*. Titrate with 0.1 M *perchloric acid*, using 0.05 mL of *crystal violet solution R* as indicator. Carry out a blank titration.

1 mL of 0.1 M *perchloric acid* is equivalent to 44.81 mg of $C_{27}H_{42}ClNO_2$.

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*.

Standardisation. To 25.0 mL of the bismuth nitrate solution, add 50 mL of *water R* and titrate with 0.01 M *sodium edetate* using 0.05 mL of a 1 g/L solution of *xylene orange R* as indicator.

0.0167 M Bromide-bromate. 3001000.

Dissolve 2.7835 g of *potassium bromate RV* and 13 g of *potassium bromide R* in *water R* and dilute to 1000.0 mL with the same solvent.

0.1 M Cerium sulfate. 3001100.

Dissolve 40.4 g of *cerium sulfate R* in a mixture of 500 mL of *water R* and 50 mL of *sulfuric acid R*. Allow to cool and dilute to 1000.0 mL with *water R*.

Standardisation. To 20.0 mL of the cerium sulfate solution, add 1.6 g of *potassium iodide R*, 100 mL of *water R* and 40 mL of *dilute sulfuric acid R*. Titrate immediately with 0.1 M *sodium thiosulfate* using 0.8 mL of *starch solution R* as indicator.

0.02 M Copper sulfate. 3001200.

Dissolve 5.0 g of *copper sulfate R* in *water R* and dilute to 1000.0 mL with the same solvent.

Standardisation. To 20.0 mL of the copper sulfate solution add 2 g of *sodium acetate R* and 0.1 mL of *pyridylazonaphthol solution R*. Titrate with 0.02 M *sodium edetate* until the colour changes from violet-blue to bright green. Titrate slowly towards the end of the titration.

0.1 M Ferric ammonium sulfate. 3001300.

Dissolve 50.0 g of *ferric ammonium sulfate R* in a mixture of 6 mL of *sulfuric acid R* and 300 mL of *water R* and dilute to 1000.0 mL with *water R*.

Standardisation. To 25.0 mL of the ferric ammonium sulfate solution, add 3 mL of *hydrochloric acid R* and 2 g of *potassium iodide R*. Allow to stand for 10 min. Titrate with 0.1 M *sodium thiosulfate*, using 1 mL of *starch solution R* as indicator.

1 mL of 0.1 M *sodium thiosulfate* is equivalent to 48.22 mg of $FeNH_4(SO_4)_2 \cdot 12H_2O$.

0.1 M Ferrous sulfate. 3001400.

Dissolve 27.80 g of *ferrous sulfate R* in 500 mL of *dilute sulfuric acid R* and dilute to 1000.0 mL with *water R*.

Standardisation. To 25.0 mL of the ferrous sulfate solution add 3 mL of *phosphoric acid R* and titrate immediately with 0.02 M *potassium permanganate*. Standardise immediately before use.

1 M Hydrochloric acid. 3001800.

Dilute 103.0 g of *hydrochloric 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 hydrochloric acid until the solution just becomes yellowish-red. Boil for 2 min. The solution reverts to yellow. Cool and continue the titration until a yellowish-red colour is obtained.

1 mL of 1 M *hydrochloric acid* is equivalent to 53.00 mg of Na_2CO_3 .

0.1 M Hydrochloric acid. 3002100.

Dilute 100.0 mL of 1 M *hydrochloric acid* to 1000.0 mL with *water R*.

Standardisation. Carry out the titration described for 1 M *hydrochloric acid* using 0.100 g of *sodium carbonate RV* dissolved in 20 mL of *water R*.

1 mL of 0.1 M *hydrochloric acid* is equivalent to 5.30 mg of Na_2CO_3 .

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 thiosulfate*, 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 thiosulfate*, 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 *xylene orange tritrate R* and about 2 g of *hexamethylenetetramine R*. Titrate with 0.1 M *zinc sulfate* until the colour changes from yellow to violet-pink.

1 mL of 0.1 M *sodium edetate* is equivalent to 43.30 mg of $La(NO_3)_3 \cdot 6H_2O$.

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*. Stir to effect solution and titrate with the lithium 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 lithium methoxide solution. Standardise immediately before use.

1 mL of 0.1 M *lithium methoxide* is equivalent to 12.21 mg of $C_7H_6O_2$.

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 Na_2CO_3 .

0.1 M Perchloric acid. 3003900.

Place 8.5 mL of *perchloric acid R* in a volumetric flask containing about 900 mL of *glacial acetic acid R* and mix. Add 30 mL of *acetic anhydride R*, dilute to 1000.0 mL with *glacial acetic acid R*, mix and allow to stand for 24 h. Determine the water content (2.5.12) without addition of methanol and, if necessary, adjust the water content to 0.1–0.2 per cent by adding either *acetic anhydride R* or *water R*. Allow to stand for 24 h.

Standardisation. Dissolve 0.350 g of *potassium hydrogen phthalate 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:

$$V_c = V [1 + (t_1 - t_2) 0.0011]$$

t_1 = temperature during standardisation,

t_2 = temperature during the assay,

V_c = corrected volume,

V = observed volume.

1 mL of 0.1 M *perchloric acid* is equivalent to 20.42 mg of $C_8H_5KO_4$.

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.

Dissolve 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.

Dissolve 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.

Dissolve 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 thiosulfate*, using 3 mL of *starch solution R* as indicator, until the colour changes from blue to light green.

0.1 M Potassium hydrogen phthalate. 3004700.

In a conical flask containing about 800 mL of *anhydrous acetic acid R*, dissolve 20.42 g of *potassium hydrogen phthalate RV*. Heat on a water-bath until completely dissolved, protected from humidity. Cool to 20 °C and dilute to 1000.0 mL with *anhydrous acetic acid R*.

1 M Potassium hydroxide. 3009100.

Dissolve 60 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 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 alcoholic 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 alcoholic 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 sulfuric acid R*. Titrate with 0.1 M *sodium thiosulfate*, 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* 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 sulfuric acid R*. Titrate with *0.1 M sodium thiosulfate*, 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 RV* 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 0.1 M to 500.0 mL with *water R*.

0.1 M Sodium arsenite. 3005800.

Dissolve *arsenious trioxide RV* equivalent to 4.946 g of As_2O_3 in a mixture of 20 mL of *strong sodium hydroxide solution R* and 20 mL of *water R*, dilute to 400 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 RV* in 4 mL of *hydrochloric acid R1* 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 RV* in 4 mL of *hydrochloric acid R1* 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 dilute to 200 mL with *water R*. Add about 50 mg 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 400-600 g/L 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 RV* in a mixture of 5 mL of *0.01 M hydrochloric acid* and 50 mL of *ethanol (96 per cent) 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 $\text{C}_7\text{H}_6\text{O}_2$.

0.1 M Sodium hydroxide, ethanolic. 3007000.

To 250 mL of *anhydrous ethanol R* add 3.3 g of *strong sodium hydroxide solution R*.

Standardisation. Dissolve 0.100 g of *benzoic acid RV* in 2 mL of *water R* and 10 mL of *ethanol (96 per cent) R*. Titrate with the ethanolic sodium hydroxide solution, using 0.2 mL of *thymolphthalein solution R* as indicator. Standardise immediately before use.

1 mL of *0.1 M ethanolic sodium hydroxide* is equivalent to 12.21 mg of $\text{C}_7\text{H}_6\text{O}_2$.

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/L 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 RV*. 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 $\text{C}_7\text{H}_6\text{O}_2$.

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 *sulfanilic acid RV* 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 $\text{C}_6\text{H}_7\text{NO}_3\text{S}$.

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 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 thiosulfate. 3007300.

Dissolve 25 g of *sodium thiosulfate 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*, add 40 mL of *water R*, 10 mL of *potassium iodide solution R* and 5 mL of *hydrochloric acid R1*. Titrate with the sodium thiosulfate solution, using 1 mL of *starch solution R*, added towards the end of the titration, as indicator.

0.5 M Sulfuric acid. 3007800.

Dissolve 28 mL of *sulfuric acid R* in *water R* and dilute to 1000.0 mL with the same solvent.

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 sulfuric 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 *sulfuric acid* is equivalent to 53.00 mg of Na_2CO_3 .

0.05 M Sulfuric acid. 3008000.

Dilute 100.0 mL of 0.5 M *sulfuric acid* to 1000.0 mL with *water R*.

Standardisation. Carry out the titration described for 0.5 M *sulfuric acid*, using 0.100 g of *sodium carbonate RV*, dissolved in 20 mL of *water R*.

1 mL of 0.05 M *sulfuric 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, each of 50 mL, of *toluene R*. Add the washings to the filtrate and dilute to 1000.0 mL with *toluene 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/L 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 RV*. 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 $\text{C}_7\text{H}_6\text{O}_2$.

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 sulfate. 3008600.

Dissolve 29 g of *zinc sulfate R* in *water R* and dilute to 1000.0 mL with the same solvent.

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