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. $C_7H_6O_2$. (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. $C_8H_5KO_4$. (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 R1*. Remove the hydrochloric acid by heating on a water-bath and dry the crystals to constant mass at 300 °C.

Sulfanilic acid. $C_6H_7NO_3S$. (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.