

solution of *lithium carbonate R* and dry in an oven before use. Envelop the substance to be examined in the paper and place it in the sample carrier. Introduce into the flask *water R* or the prescribed solution designed to absorb the combustion products, displace the air with oxygen by means of a tube having its end just above the liquid, moisten the neck of the flask with *water R* and close with its stopper. Ignite the paper strip by suitable means with the usual precautions. Keep the flask firmly closed during the combustion. Shake the flask vigorously to completely dissolve the combustion products. Cool and after about 5 min, unless otherwise prescribed, carefully unstopper the flask. Wash the ground parts and the walls of the flask, as well as the sample carrier, with *water R*. Combine the combustion products and the washings and proceed as prescribed in the monograph.

01/2008:20511

2.5.11. COMPLEXOMETRIC TITRATIONS

ALUMINIUM

Introduce 20.0 mL of the prescribed solution into a 500 mL conical flask, add 25.0 mL of 0.1 M sodium edetate and 10 mL of a mixture of equal volumes of a 155 g/L solution of *ammonium acetate R* and *dilute acetic acid R*. Boil for 2 min, then cool. Add 50 mL of *ethanol R* and 3 mL of a freshly prepared 0.25 g/L solution of *dithizone R* in *ethanol R*. Titrate the excess of sodium edetate with 0.1 M zinc sulfate until the colour changes from greenish-blue to reddish-violet.

1 mL of 0.1 M sodium edetate is equivalent to 2.698 mg of Al.

BISMUTH

Introduce the prescribed solution into a 500 mL conical flask. Dilute to 250 mL with *water R* and then, unless otherwise prescribed, add dropwise, with shaking, *concentrated ammonia R* until the mixture becomes cloudy. Add 0.5 mL of *nitric acid R*. Heat to about 70 °C until the cloudiness disappears completely. Add about 50 mg of *xylenol orange triturate R* and titrate with 0.1 M sodium edetate until the colour changes from pinkish-violet to yellow.

1 mL of 0.1 M sodium edetate is equivalent to 20.90 mg of Bi.

CALCIUM

Introduce the prescribed solution into a 500 mL conical flask, and dilute to 300 mL with *water R*. Add 6.0 mL of *strong sodium hydroxide solution R* and about 15 mg of *calconecarboxylic acid triturate R*. Titrate with 0.1 M sodium edetate until the colour changes from violet to full blue.

1 mL of 0.1 M sodium edetate is equivalent to 4.008 mg of Ca.

MAGNESIUM

Introduce the prescribed solution into a 500 mL conical flask and dilute to 300 mL with *water R*. Add 10 mL of *ammonium chloride buffer solution pH 10.0 R* and about 50 mg of *mordant black 11 triturate R*. Heat to about 40 °C then titrate at this temperature with 0.1 M sodium edetate until the colour changes from violet to full blue.

1 mL of 0.1 M sodium edetate is equivalent to 2.431 mg of Mg.

LEAD

Introduce the prescribed solution into a 500 mL conical flask and dilute to 200 mL with *water R*. Add about 50 mg of *xylenol orange triturate R* and *hexamethylenetetramine R* until the solution becomes violet-pink. Titrate with 0.1 M sodium edetate until the violet-pink colour changes to yellow.

1 mL of 0.1 M sodium edetate is equivalent to 20.72 mg of Pb.

ZINC

Introduce the prescribed solution into a 500 mL conical flask and dilute to 200 mL with *water R*. Add about 50 mg of *xylenol orange triturate R* and *hexamethylenetetramine R* until the solution becomes violet-pink. Add 2 g of *hexamethylenetetramine R* in excess. Titrate with 0.1 M sodium edetate until the violet-pink colour changes to yellow.

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

01/2008:20512
corrected 6.0

2.5.12. WATER: SEMI-MICRO DETERMINATION

The semi-micro determination of water is based upon the quantitative reaction of water with sulfur dioxide and iodine in a suitable anhydrous medium in the presence of a base with sufficient buffering capacity.

Apparatus

The apparatus consists of a titration vessel with:

- 2 identical platinum electrodes;
- tight inlets for introduction of solvent and titrant;
- an inlet for introduction of air via a desiccant;
- a sample inlet fitted with a stopper or, for liquids, a septum.

Inlet systems for introduction of dry nitrogen or for aspiration of solvents may also be fitted.

The titration is carried out according to the instrument supplier's instructions. Care is taken throughout the determination to avoid exposure of reagents and solvents to atmospheric moisture. The end-point is determined using 2 identical indicator electrodes connected to an electrical source that maintains between the electrodes either a constant current or a constant voltage. Where direct titration is used (method A), addition of titrant causes either a decrease in voltage where constant current is maintained or an increase in current where constant voltage is maintained, until the end-point is reached. Instruments with automatic end-point detection are commonly used.

Standardisation. To the titration vessel, add *methanol R*, dried if necessary, or the solvent recommended by the supplier of the titrant. Where applicable for the apparatus used, eliminate residual water from the measurement cell or carry out a pre-titration. Introduce a suitable amount of water in an appropriate form (*water R* or a certified reference material) and carry out the titration, stirring for the necessary time. The water equivalent is not less than 80 per cent of that indicated by the supplier. Standardise the titrant before the first use and at suitable intervals thereafter.

Unless otherwise prescribed, use Method A.

Method A. Introduce into the titration vessel *methanol R*, or the solvent indicated in the monograph or recommended by the supplier of the titrant. Where applicable for the apparatus used, eliminate residual water from the measurement cell or carry out a pre-titration. Introduce the substance to be examined rapidly and carry out the titration, stirring for the necessary extraction time.

Method B. Introduce into the titration vessel *methanol R*, or the solvent indicated in the monograph or recommended by the supplier of the titrant. Where applicable for the apparatus used, eliminate residual water from the measurement cell or carry out a pre-titration. Introduce the substance to be examined rapidly and in a suitable state of division. Add an accurately measured volume of the titrant, sufficient to give an excess of about 1 mL or the prescribed volume. Allow to stand protected from light for 1 min or the prescribed time, with stirring. Titrate the excess of reagent using *methanol R* or the prescribed solvent, containing an accurately known quantity of water.