2.4. LIMIT TESTS

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2.4.1. AMMONIUM

Unless otherwise prescribed, use method A.

METHOD A

Introduce the prescribed solution into a test-tube or dissolve the prescribed quantity of the substance to be examined in 14 mL of *water R* in a test-tube. Make the solution alkaline if necessary by the addition of *dilute sodium hydroxide solution R*, dilute to 15 mL with *water R* and add 0.3 mL of *alkaline potassium tetraiodomercurate solution R*. Prepare a standard by mixing 10 mL of *ammonium standard solution (1 ppm NH_4) R*, 5 mL of *water R* and 0.3 mL of *alkaline potassium tetraiodomercurate solution R*. Stopper the test-tubes.

After 5 min, any yellow colour in the test solution is not more intense than that in the standard.

METHOD B

In a 25 mL jar fitted with a cap, place the prescribed quantity of the finely powdered substance to be examined and dissolve or suspend in 1 mL of *water R*. Add 0.30 g of *heavy magnesium oxide R*. Close immediately after placing a piece of *silver manganese paper R* 5 mm square, wetted with a few drops of *water R*, under the polyethylene cap. Swirl, avoiding projections of liquid, and allow to stand at 40 °C for 30 min. If the silver manganese paper shows a grey colour, it is not more intense than that of a standard prepared at the same time and in the same manner using the prescribed volume of *ammonium standard solution (1 ppm NH_4) R*, 1 mL of *water R* and 0.30 g of *heavy magnesium oxide R*.

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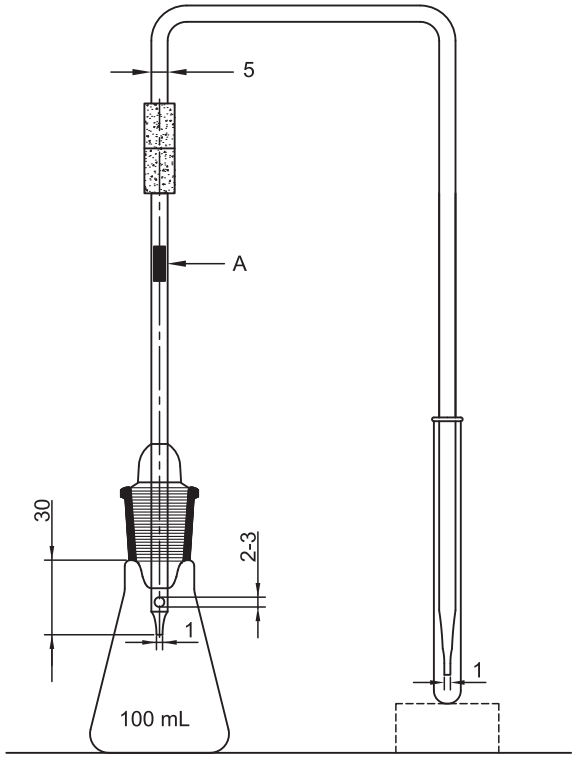
2.4.2. ARSENIC

METHOD A

The apparatus (see Figure 2.4.2.-1) consists of a 100 mL conical flask closed with a ground-glass stopper through which passes a glass tube about 200 mm long and about 5 mm in internal diameter. The lower part of the tube tapers to an internal diameter of 1 mm, and about 20 mm from its tip is a lateral orifice 2-3 mm in diameter. When the tube is in position in the stopper, the lateral orifice is at least 3 mm below the lower surface of the stopper. A second glass tube of the same internal diameter is connected to the first tube. The second tube is bent twice at right angles and the free end of the tube tapers to an internal diameter of 1 mm. This end is immersed in a test-tube containing 3.0 mL of *silver diethyldithiocarbamate solution R*. Other suitable equipment may be used. Into the first tube insert 50-60 mg of *lead acetate cotton R*, loosely packed, or a small plug of cotton and a rolled piece of *lead acetate paper R* weighing 50-60 mg.



A. Lead acetate paper/cotton

Figure 2.4.2.-1. – *Apparatus for the limit test for arsenic (method A)*

Dimensions in millimetres

In the conical flask, dissolve the prescribed quantity of the substance to be examined in 25 mL of *water R*, or in the case of a solution adjust the prescribed volume to 25 mL with *water R*. Add 15 mL of *hydrochloric acid R*, 0.1 mL of *stannous chloride solution R* and 5 mL of *potassium iodide solution R*, allow to stand for 15 min and introduce 5 g of *activated zinc R*. Assemble the 2 parts of the apparatus immediately and immerse the flask in a water-bath at a temperature such that a uniform evolution of gas is maintained. Prepare a standard in the same manner, using 1 mL of *arsenic standard solution (1 ppm As) R*, diluted to 25 mL with *water R*. If foaming occurs, 1 mL of *2-propanol R* may be added to the flask.

After at least 2 h, the colour obtained in the test-tube with the test solution is not more intense than that obtained with the standard.

Suitability test. The colour obtained in the test-tube with the standard is at least as intensely coloured as 3 mL of a mixture of 3.0 mL of yellow primary solution, 0.6 mL of red primary solution and 11.40 mL of a solution of *hydrochloric acid R* (10 g/L HCl) (*2.2.2, Method I*).

METHOD B

Introduce the prescribed quantity of the substance to be examined into a test-tube containing 4 mL of *hydrochloric acid R* and about 5 mg of *potassium iodide R* and add 3 mL of *hypophosphorous reagent R*. Heat the mixture on a water-bath for 15 min, shaking occasionally. Prepare a standard in the same manner, using 0.5 mL of *arsenic standard solution (10 ppm As) R*.

After heating on the water-bath, the colour obtained in the test-tube with the test solution is not more intense than that obtained with the standard.