2.4. LIMIT TESTS

01/2005:20401

2.4.1. AMMONIUM

Unless otherwise prescribed, use method A.

METHOD A

Dissolve the prescribed quantity of the substance to be examined in 14 ml of *water R* in a test-tube, make alkaline if necessary by the addition of *dilute sodium hydroxide solution R* and dilute to 15 ml with *water R*. To the solution 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* with 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 of internal diameter 5 mm. The lower part of the tube is drawn to an internal diameter of 1.0 mm, and 15 mm from its tip is a lateral orifice 2 mm to 3 mm in diameter. When the tube is in position in the stopper, the lateral orifice should be at least 3 mm below the lower surface of the stopper. The upper end of the tube has a perfectly flat, ground surface at right angles to the axis of the tube. A second glass tube of the same internal diameter and 30 mm long, with a similar flat ground surface, is placed in contact with the first, and is held in position by two spiral springs. Into the lower tube insert 50 mg to 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 mg to 60 mg. Between the flat surfaces of the tubes place a disc or a small square of *mercuric bromide paper R* large enough to cover the orifice of the tube (15 mm × 15 mm).

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 two parts of the apparatus immediately and immerse the flask in a bath of water 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*.

After not less than 2 h the stain produced on the mercuric bromide paper in the test is not more intense than that in the standard.

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, any colour in the test solution is not more intense than that in the standard.

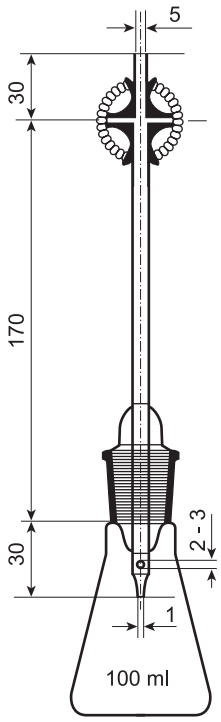

Figure 2.4.2.-1. - *Apparatus for limit test A for arsenic*
Dimensions in millimetres

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2.4.3. CALCIUM

All solutions used for this test should be prepared with distilled water R.

To 0.2 ml of *alcoholic calcium standard solution (100 ppm Ca) R*, add 1 ml of *ammonium oxalate solution R*. After 1 min, add a mixture of 1 ml of *dilute acetic acid R* and 15 ml of a solution containing the prescribed quantity of the substance to be examined and shake. Prepare a standard in the same manner using a mixture of 10 ml of *aqueous calcium standard solution (10 ppm Ca) R*, 1 ml of *dilute acetic acid R* and 5 ml of *distilled water R*.

After 15 min, any opalescence in the test solution is not more intense than that in the standard.