2.4. LIMIT TESTS

01/2008:20401

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.

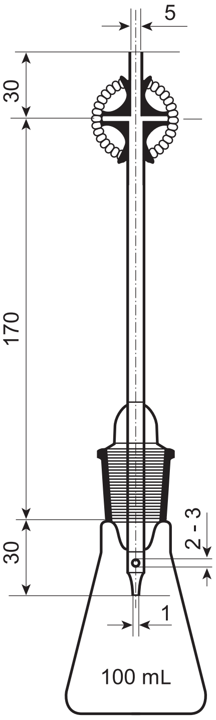

Figure 2.4.2.-1. - *Apparatus for limit test A for arsenic*
Dimensions in millimetres

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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.

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2.4.4. CHLORIDES

To 15 mL of the prescribed solution add 1 mL of *dilute nitric acid R* and pour the mixture as a single addition into a test-tube containing 1 mL of *silver nitrate solution R2*. Prepare a standard in the same manner using 10 mL of *chloride standard solution (5 ppm Cl) R* and 5 mL of *water R*. Examine the tubes laterally against a black background.

After standing for 5 min protected from light, any opalescence in the test solution is not more intense than that in the standard.

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2.4.5. FLUORIDES

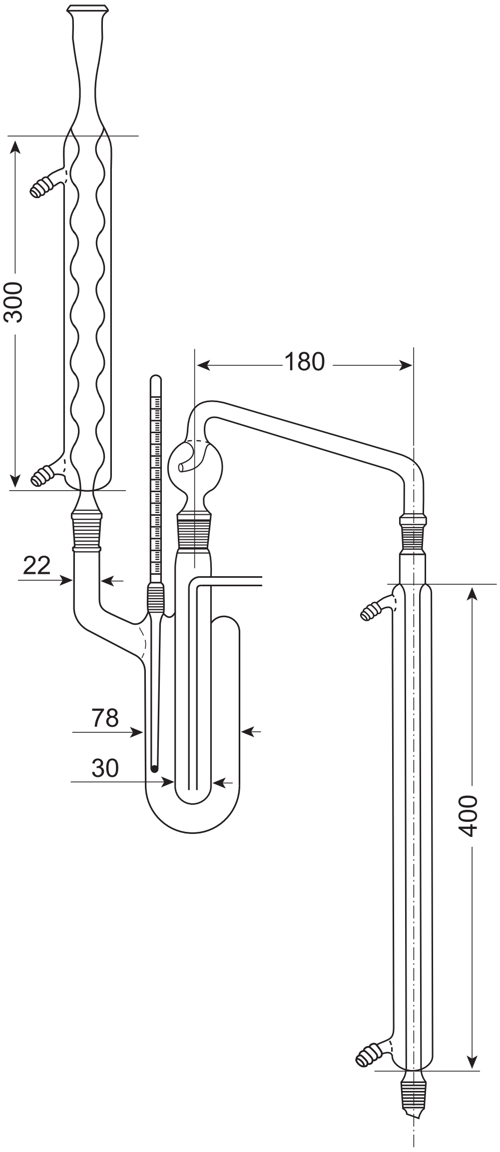

Figure 2.4.5.-1. – *Apparatus for limit test for fluorides*

Dimensions in millimetres

Introduce into the inner tube of the apparatus (see Figure 2.4.5.-1) the prescribed quantity of the substance to be examined, 0.1 g of acid-washed *sand R* and 20 mL of a mixture of equal volumes of *sulfuric acid R* and *water R*. Heat the jacket containing *tetrachloroethane R* maintained at its boiling point (146 °C). Heat the steam generator and distil, collecting the distillate in a 100 mL volumetric flask containing 0.3 mL of *0.1 M sodium hydroxide* and 0.1 mL of *phenolphthalein solution R*. Maintain a constant volume (20 mL) in the tube during distillation and ensure that the distillate remains alkaline, adding *0.1 M sodium hydroxide* if necessary. Dilute the distillate to 100 mL with *water R* (test solution). Prepare a standard in the same manner by distillation, using 5 mL of *fluoride standard solution (10 ppm F) R* instead of the substance to be examined. Into two glass-stoppered cylinders introduce 20 mL of the test solution and 20 mL of the standard and 5 mL of *aminomethylalizarindiacetic acid reagent R*.

After 20 min, any blue colour in the test solution (originally red) is not more intense than that in the standard.

01/2008:20406

2.4.6. MAGNESIUM

To 10 mL of the prescribed solution add 0.1 g of *disodium tetraborate R*. Adjust the solution, if necessary, to pH 8.8 to pH 9.2 using *dilute hydrochloric acid R* or *dilute sodium hydroxide solution R*. Shake with 2 quantities, each of 5 mL, of a 1 g/L solution of *hydroxyquinoline R* in *chloroform R*, for 1 min each time. Allow to stand. Separate and discard the organic layer. To the aqueous solution add 0.4 mL of *butylamine R* and 0.1 mL of *triethanolamine R*. Adjust the solution, if necessary, to pH 10.5 to pH 11.5. Add 4 mL of the solution of hydroxyquinoline in chloroform, shake for 1 min, allow to stand and separate. Use the lower layer for comparison. Prepare a standard in the same manner using a mixture of 1 mL of *magnesium standard solution (10 ppm Mg) R* and 9 mL of *water R*.

Any colour in the solution obtained from the substance to be examined is not more intense than that in the standard.

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2.4.7. MAGNESIUM AND ALKALINE-EARTH METALS

To 200 mL of *water R* add 0.1 g of *hydroxylamine hydrochloride R*, 10 mL of *ammonium chloride buffer solution pH 10.0 R*, 1 mL of *0.1 M zinc sulfate* and about 15 mg of *mordant black 11 triturate R*. Heat to about 40 °C. Titrate with *0.01 M sodium edetate* until the violet colour changes to full blue. To the solution add the prescribed quantity of the substance to be examined dissolved in 100 mL of *water R* or use the prescribed solution. If the colour of the solution changes to violet, titrate with *0.01 M sodium edetate* until the full blue colour is again obtained.

The volume of *0.01 M sodium edetate* used in the second titration does not exceed the prescribed quantity.

07/2010:20408

2.4.8. HEAVY METALS

The methods described below require the use of *thioacetamide reagent R*. As an alternative, *sodium sulfide solution R1* (0.1 mL) is usually suitable. Since tests prescribed in monographs have been developed using *thioacetamide reagent R*, if *sodium sulfide solution R1* is used instead, it is necessary to include also for methods A, B and H a monitor solution, prepared from the quantity of the substance to be examined prescribed for the test, to which has been added the volume of lead standard solution prescribed for preparation of the reference solution. The test is invalid if the monitor solution is not at least as intense as the reference solution.

METHOD A

Test solution. 12 mL of the prescribed aqueous solution of the substance to be examined.

Reference solution (standard). A mixture of 10 mL of *lead standard solution (1 ppm Pb) R* or *lead standard solution (2 ppm Pb) R*, as prescribed, and 2 mL of the prescribed aqueous solution of the substance to be examined.

Blank solution. A mixture of 10 mL of *water R* and 2 mL of the prescribed aqueous solution of the substance to be examined.

To each solution, add 2 mL of *buffer solution pH 3.5 R*. Mix and add to 1.2 mL of *thioacetamide reagent R*. Mix immediately. Examine the solutions after 2 min.

System suitability: the reference solution shows a slight brown colour compared to the blank solution.

Result: any brown colour in the test solution is not more intense than that in the reference solution.