

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.

01/2008:20404 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 *aminomethylizarindiacetic acid reagent R*. After 20 min, any blue colour in the test solution (originally red) is not more intense than that in the standard.

01/2008:20405

01/2008:20406

2.4.5. FLUORIDES

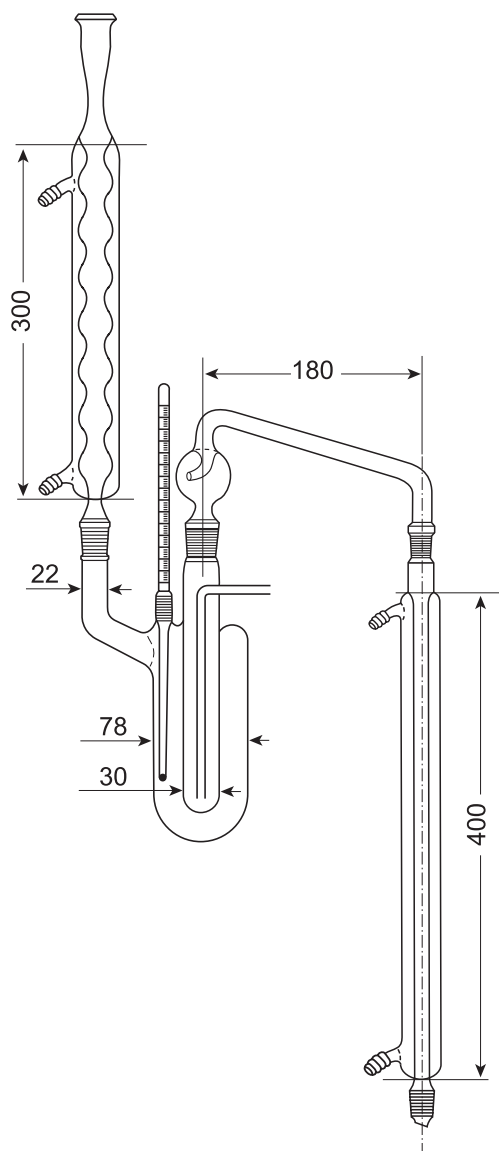


Figure 2.4.5.-1. – Apparatus for limit test for fluorides
Dimensions in millimetres

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 *sulphuric acid R* and *water R*. Heat the jacket containing *tetrachloroethane R* maintained at its boiling point (146 °C). Heat the steam generator and

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/1 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.

01/2008:20407

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 sulphate* 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.

01/2008:20408
corrected 6.0

2.4.8. HEAVY METALS

The methods described below require the use of *thioacetamide reagent R*. As an alternative, *sodium sulphide solution R1* (0.1 ml) is usually suitable. Since tests prescribed in monographs have been developed using *thioacetamide reagent R*, if *sodium sulphide solution R1* is used instead, it is necessary to include also for methods A and B a monitor solution, prepared from the quantity of the substance to be examined prescribed for the test, to