01/2008:20403
corrected 8.0

2.4.3. CALCIUM

All solutions used for this test are 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 the prescribed solution or 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.

01/2008:20404

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.

01/2008:20405

2.4.5. FLUORIDES

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.

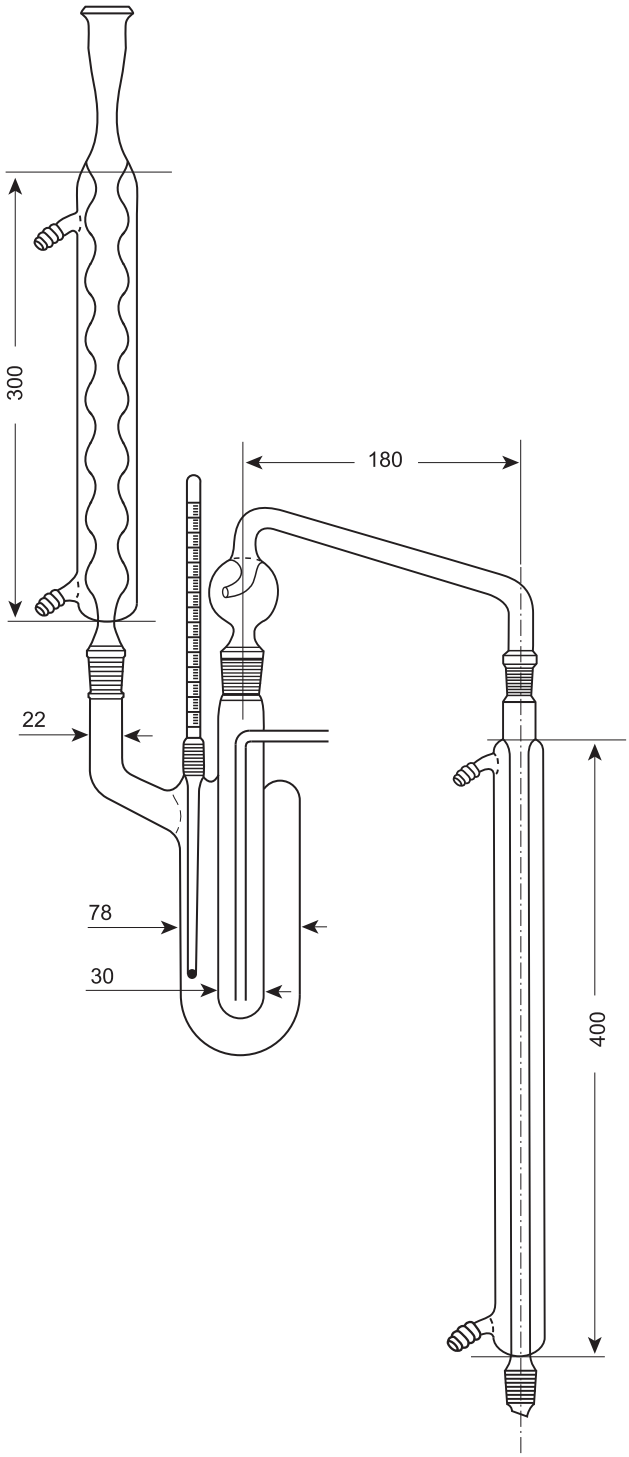

Figure 2.4.5.-1. – *Apparatus for limit test for fluorides*
Dimensions in millimetres

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