

2.4. LIMIT TESTS

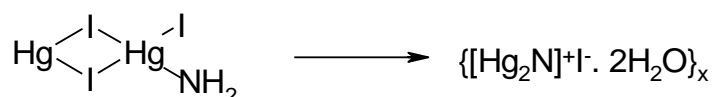
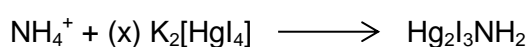
2.4.1. AMMONIUM

METHOD A

Reagents: alkaline potassium tetraiodomercurate solution (Nessler's reagent) in alkaline solution of NaOH

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 NH₄) R* with 5 mL of *water R* and 0.3 mL of *alkaline potassium tetraiodomercurate solution R*.

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



High molecular base, three- dimensional structure
Orange to red- brown colour

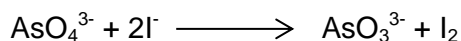
2.4.2. ARSENIC

METHOD B

Reagents: hydrochloric acid, potassium iodide, hypophosphorous reagent

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.



2.4.3. CALCIUM

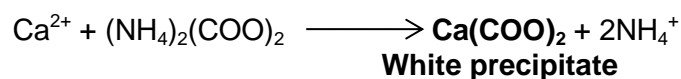
Reagents: ammonium oxalate, dilute acetic acid

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.



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 0.1 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 0.1 M sodium edetate until the full blue colour is again obtained.

The volume of 0.1 M sodium edetate used in the second titration does not exceed the prescribed quantity.

2.4.4. CHLORIDES

Reagents: *dilute nitric acid, silver nitrate*

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.



2.4.8. HEAVY METALS

METHOD A

Reagents: *buffer solution pH 3.5, thioacetamide reagent*

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

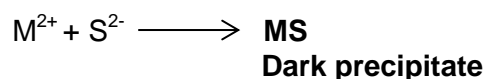
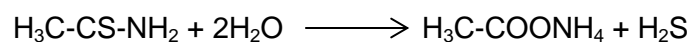
Reference solution (standard). A mixture of 10 mL of *lead standard solution (1 ppm Pb) R* or *lead standard solution (2 ppm Pb) R*, as prescribed, and 2 mL of the prescribed aqueous solution of the substance to be examined.

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.

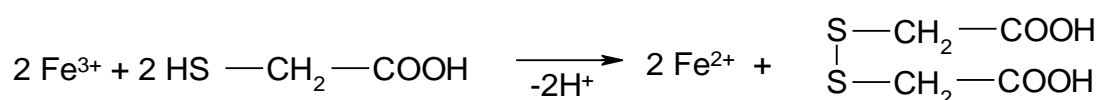
Result: any brown colour in the test solution is not more intense than that in the reference solution.



2.4.9. IRON

Reagents: *thioglycollic acid R, citric acid, ammonia R*

Dissolve the prescribed quantity of the substance to be examined in *water R* and dilute to 10 mL with the same solvent or use 10 mL of the prescribed solution. Add 2 mL of a 200 g/L solution of *citric acid R* and 0.1 mL of *thioglycollic acid R*. Mix, make alkaline with *ammonia R* and dilute to 20 mL with *water R*. Prepare a standard in the same manner, using 10 mL of *iron standard solution (1 ppm Fe) R*. After 5 min, any pink colour in the test solution is not more intense than that in the standard.



complex Fe(II)- thioglycolate
colourless or light yellow

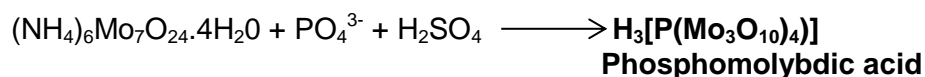
oxidation to Fe (III)
pink or red

2.4.11. PHOSPHATES

Reagents: *sulfomolybdic reagent, stannous chloride*

To 100 mL of the solution prepared and, if necessary, neutralised as prescribed add 4 mL of *sulfomolybdic reagent R3*. Shake and add 0.1 mL of *stannous chloride solution R1*. Prepare a standard in the same manner using 2 mL of *phosphate standard solution (5 ppm PO₄) R* and 98 mL of *water R*. After 10 min, compare the colours using 20 mL of each solution.

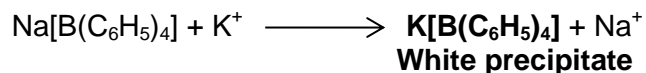
Any colour in the test solution is not more intense than that in the standard.



Reagents: sodium tetrphenylborate

To 10 mL of the prescribed solution add 2 mL of a freshly prepared 10 g/L solution of *sodium tetrphenylborate R*. Prepare a standard in the same manner using a mixture of 5 mL of *potassium standard solution (20 ppm K) R* and 5 mL of *water R*.

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



2.4.13. SULFATES

Reagents: barium chloride, acetic acid

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

Add 3 mL of a 250 g/L solution of *barium chloride R* to 4.5 mL of *sulfate standard solution (10 ppm SO₄) R1*. Shake and allow to stand for 1 min. To 2.5 mL of this suspension, add 15 mL of the solution to be examined and 0.5 mL of *acetic acid R*. Prepare a standard in the same manner using 15 mL of *sulfate standard solution (10 ppm SO₄) R* instead of the solution to be examined.

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

