2.1 General identification tests

Acetylated substances

Place a quantity of the substance as specified in the monograph in a test-tube (of maximum 18 mm diameter) and treat it with 3 drops of phosphoric acid (~1440 g/l) TS. Close the tube with a stopper through which passes a smaller test-tube filled with water and on the outside of which hangs a drop of lanthanum nitrate (30 g/l) TS. Heat the apparatus in a boiling water-bath for 5 minutes. Transfer the drop of lanthanum nitrate to a white porcelain spot plate and mix with a drop of iodine (0.02 mol/l) VS. Place at the edge of the mixture a drop of ammonia (~100 g/l) TS. A blue colour slowly appears at the interface of the two liquids and persists for a short time.

Amines, primary aromatic

Dissolve a quantity of the substance as specified in the monograph in 2 mL of hydrochloric acid (~70 g/l) TS with the aid of heat, if necessary. Cool in ice, treat it with 4 mL of sodium nitrite (10 g/l) TS and pour the mixture into 2 mL of 2-naphthol TS1 containing 1 g of sodium acetate R. A heavy precipitate, coloured as specified in the monograph, is produced.

Ammonia and volatile aliphatic amines

Dissolve a quantity of the substance as specified in the monograph, place the solution in a test-tube and add 1 g of magnesium oxide R; warm, if specified in the monograph. Alkaline vapours evolve gradually and turn manganese/silver paper R black, the reagent paper being placed in the upper part of the test-tube.

Ammonium

Carry out the test in an apparatus consisting of stoppered test-tubes A and B connected by a bent glass tube to permit a stream of air to pass consecutively through test-tubes A and B.

Place the solution as specified in the monograph and 0.2 g of magnesium oxide into test-tube A, and 1 mL of hydrochloric acid (0.1 mol/l) VS containing 1 drop of methyl red/ethanol TS in test-tube B. Bubble air through the apparatus. Evolved ammonia turns the colour of the solution in test-tube B to yellow. On the addition of 1 mL of sodium cobaltinitrite (100 g/l) TS to this solution a yellowish brown precipitate is formed.

Bismuth

A. Prepare the solution in hydrochloric acid (~250 g/l) TS as specified in the monograph and dilute 10 times with water. A white precipitate is formed, which turns dark brown on the addition of sodium sulfide TS.

B. Treat the solution in nitric acid (~1000 g/l) TS as specified in the monograph with potassium iodide (80 g/l) TS. A black precipitate is formed, which is soluble in an excess of the reagent to give a yellowish brown or orange solution. Dilute this solution with several volumes of water and heat; an orange or copper-coloured precipitate is obtained. The black precipitate that is first formed on the addition of potassium iodide (80 g/l) TS also becomes orange or copper-coloured when heated with water.

Bromides

A. Prepare a solution as specified in the monograph, acidify with nitric acid (~130 g/l) TS and add silver nitrate (40 g/l) TS. A yellowish curdy precipitate is produced, which is partially soluble in ammonia (~260 g/l) TS, but almost insoluble in ammonia (~100 g/l) TS and in nitric acid (~1000 g/l) TS.

B. (For testing bromides or hydrobromides of insoluble or sparingly soluble bases.) Prepare the solution as specified in the monograph, add ammonia (~100 g/l) TS, filter, acidify the filtrate with nitric acid (~130 g/l) TS, and proceed with test A.

C. Prepare the solution as specified in the monograph, acidify with sulfuric acid (~100 g/l) TS, and mix with chlorine TS. A brown solution results; after shaking with chloroform R it becomes colourless, whereas the chloroform layer turns reddish.

Calcium

A. Prepare the solution as specified in the monograph and add to it ammonium oxalate (25 g/l) TS. A white precipitate is formed, which is soluble in hydrochloric acid (~250 g/l) TS but is practically insoluble in acetic acid (~300 g/l) TS.

B. Treat 1 drop of a solution as specified in the monograph with 4 drops of glyoxal bis(2-hydroxyanil) TS, and 1 drop of sodium hydroxide (~80 g/l) TS. A reddish brown precipitate is formed, which dissolves in chloroform R to give a red solution.

Chlorides

A. Prepare a solution as specified in the monograph, acidify with nitric acid (~130 g/l) TS and add silver nitrate (40 g/l) TS. A white curdy precipitate is produced, which is soluble in ammonia (~100 g/l) TS but is practically insoluble in nitric acid (~1000 g/l) TS.

B. (For testing chlorides or hydrochlorides of insoluble or sparingly soluble bases.) Prepare the solution as specified in the
monograph, add ammonia (~100 g/l) TS, filter and acidify the filtrate with nitric acid (~130 g/l) TS and proceed with test A.

C. Mix the quantity of the substance as specified in the monograph with an equal quantity of manganese dioxide R, moisten with sulfuric acid (~1760 g/l) TS and heat gently. The evolved chlorine is recognizable by its greenish colour and produces a blue coloration of moistened starch/iodide paper R. Carry out the reaction preferably under a hood.

**Citrate**

A. Treat at ambient temperature a neutral solution as specified in the monograph with calcium chloride (55 g/l) TS. No precipitate is formed, but on boiling, a white solid is produced, which is soluble in acetic acid (~300 g/l) TS.

B. Boil a solution with mercuric sulfate TS as specified in the monograph and filter if necessary. After the addition of a few drops of potassium permanganate (10 g/l) TS to the filtrate, the colour is discharged and a white precipitate is produced.

**Ferrous salts**

A. Prepare a solution as specified in the monograph and add potassium ferricyanide (10 g/l) TS. A dark-blue precipitate is formed, which is practically insoluble in hydrochloric acid (~70 g/l) TS.

B. Prepare a solution as specified in the monograph, acidify with sulfuric acid (~100 g/l) TS, and treat with o-phenanthroline (1 g/l) TS. An intense red colour is produced, which is discharged by the addition of ceric sulfate (35 g/l) TS.

**Iodides**

A. Prepare a solution as specified in the monograph, acidify with nitric acid (~130 g/l) TS and add silver nitrate (40 g/l) TS. A yellow curdy precipitate is formed, which is practically insoluble in ammonia (~100 g/l) TS and in nitric acid (~1000 g/l) TS.

B. (For testing iodides of insoluble or sparingly soluble bases.) Prepare the solution as specified in the monograph, add ammonia (~100 g/l) TS, filter and acidify the filtrate with nitric acid (~130 g/l) TS and proceed with test A.

C. Prepare a solution as specified in the monograph, acidify with sulfuric acid (~100 g/l) TS and add potassium nitrite (100 g/l) TS. A brown solution results; after shaking with chloroform R, it becomes colourless, whereas the chloroform layer turns violet.

**Nitrate**

A. Prepare a solution as specified in the monograph and treat it with ferrous sulfate (15 g/l) TS. No brown colour appears unless sulfuric acid (~1760 g/l) TS is cautiously added to form a lower layer. A brown colour is then produced at the interface of the two liquids.

B. Add 2 mg of the finely ground test substance to a mixture of 0.1 mL of nitrobenzene R and 0.2 mL of sulfuric acid (~1760 g/l) TS. Allow to stand at room temperature for 5 minutes, cool in ice, and add slowly while mixing 5 mL of water and 3 mL of sodium hydroxide (~400 g/l) TS. Add 5 mL of acetone R, shake and allow to separate. An intense violet colour is produced in the upper phase.

**Orthophosphate**

A. Add drop by drop a quantity of nitric acid (~130 g/l) TS to 5 mL of ammonium molybdate (95 g/l) TS until any precipitate that may appear dissolves. Divide this solution into 2 portions, add to one portion the test solution acidified with nitric acid (~130 g/l) TS as specified in the monograph, and boil both portions. A yellow precipitate is formed with the test solution while the other shows no more than a slight opalescence.

B. Prepare a neutral solution as specified in the monograph and add silver nitrate (40 g/l) TS. A yellow precipitate is produced, which does not darken upon heating the solution to boiling. The precipitate is soluble in ammonia (~100 g/l) TS and in nitric acid (~130 g/l) TS.

**Potassium**

Prepare an alkaline solution as specified in the monograph and treat it with sodium tetraphenylborate (30 g/l) TS. A white precipitate is produced.

**Salicylates**

Treat a neutral solution as specified in the monograph with ferric chloride (25 g/l) TS. An intense reddish violet colour appears, which remains on the addition of a small amount of acetic acid (~300 g/l) TS but disappears on the addition of hydrochloric acid (~70 g/l) TS, with separation of a white crystalline precipitate.

**Sodium**

A. Moisten a quantity of the substance with hydrochloric acid (~250 g/l) TS. An intense yellow colour is produced when the solution is introduced into a nonluminous flame.
NOTE: Perform test B if for technical reasons test A cannot be carried out.

B. Acidify a solution as specified in the monograph with acetic acid (~60 g/l) TS, filter, if necessary, and treat it with uranyl/zinc acetate TS. A yellow crystalline precipitate is produced.

**Sulfates**

A. Prepare a solution as specified in the monograph and add barium chloride (50 g/l) TS. A white precipitate is formed, which is practically insoluble in hydrochloric acid (~250 g/l) TS.

B. To a solution as specified in the monograph, add lead acetate (80 g/l) TS. A white precipitate is formed, which is soluble in ammonium acetate (80 g/l) TS and in sodium hydroxide (~80 g/l) TS, but practically insoluble in hot water.

**Tartrates**

A. Acidify a solution as specified in the monograph with acetic acid (~300 g/l) TS and add 1 drop of ferrous sulfate (15 g/l) TS, a few drops of hydrogen peroxide (~60 g/l) TS, and enough sodium hydroxide (~80 g/l) TS to make the solution alkaline. A purple or violet colour is produced.

B. Mix a few mL of sulfuric acid (~1760 g/l) TS with a few drops of resorcinol (20 g/l) TS and a few drops of potassium bromide (100 g/l) TS and add 2 or 3 drops of a solution as specified in the monograph. Warm the liquid in a water-bath for 5 to 10 minutes. An intense blue colour is produced. Cool the liquid and pour it into water. The solution becomes red.