2.1 General identification tests

2022-01

Acetylated substances

Place a quantity of the test 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 test 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, allow to stand for 2 minutes 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.

Bromides

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.

NOTE: 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.

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 dichloromethane R, it becomes colourless whereas the dichloromethane layer turns reddish.

Calcium

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.

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 dichloromethane R to give a red solution.

Chlorides

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.

NOTE: 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.

Mix the quantity of the test 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.

Citrates

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.

Dissolve in 5 mL of water R a quantity of the substance to be examined equivalent to about 50 mg of citric acid or use 5 mL of the prescribed solution. Add 0.5 mL of sulfuric acid R and 1 mL of potassium permanganate (~25 g/L) TS. Warm until the colour of the permanganate is discharged. Add 0.5 mL of a 100 g/L solution of sodium nitroprusside R in dilute sulfuric acid (~98 g/L) TS and 4 g of sulfamic acid R. Make alkaline with concentrated ammonia R, added dropwise until all the sulfamic acid has dissolved. Addition of an excess of ammonia (~260 g/L) TS produces a violet colour, turning to violet-blue.

Ferric salts

Dissolve a quantity of the test substance to be examined equivalent to not less than 1 mg of iron (Fe^{3+}) in 1 mL of water R or use 1 mL of the prescribed solution. Add 1 mL of potassium ferrocyanide (~53 g/L) TS. A blue precipitate is formed that does not dissolve on addition of 5 mL of hydrochloric acid (~70 g/L) TS.

Ferrous salts

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.

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

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.

NOTE: For testing iodides of insoluble or sparingly soluble bases. Prepare a 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.

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 dichloromethane R, it becomes colourless whereas the dichloromethane layer turns violet.

Nitrates

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.

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.

Orthophosphates

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.

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

Dissolve 0.1 g of the test substance in 2 mL of water R or use 2 mL of the solution described in the monograph. Add 2 mL of potassium carbonate (150 g/L) TS and heat to boiling. No precipitate is formed. Add 4 mL of potassium pyroantimonate (13 g/L) TS and heat to boiling. Allow to cool in iced water and, if necessary, rub the inside of the test tube with a glass rod. A dense white precipitate is produced.

Dissolve a quantity of the substance to be examined equivalent to about 2 mg of sodium (Na+) in 0.5 mL of water R or use 0.5 mL of the prescribed solution. Add 1.5 mL of methoxyphenylacetic R and cool in ice-water for 30 minutes. A voluminous, white, crystalline precipitate is produced. Place in water at 20 °C and stir for 5 minutes. The precipitate does not disappear. Add 1 mL of ammonia (~100 g/L) TS. The precipitate dissolves completely. Add 1 mL of ammonium carbonate solution R. No precipitate is produced.

Sulfates

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.

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

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.

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.