

B. GENERAL TESTS

Qualitative Tests

The Qualitative Tests are mainly applied to Identification tests. Unless otherwise specified, the concentration of the sample solution used is about 1%.

Acetate (1) When solutions of acetates are warmed with sulfuric acid, the odor of acetic acid is evolved.

(2) When acetates are heated with ethanol and a small quantity of sulfuric acid, the odor of ethyl acetate is evolved.

(3) When ferric chloride solution (1 : 10) is added to neutral solutions of acetates (1 : 20), a red-brown color is formed, and a red-brown precipitate is formed by boiling. When hydrochloric acid is added, the precipitate dissolves and the color of the solution changes to yellow.

Aluminum Salt (1) When ammonium chloride solution (1 : 10) and ammonia TS are added to solutions of aluminum salts (1 : 20), a white gelatinous precipitate is formed. When excess ammonia TS is then added, the precipitate does not dissolve.

(2) When sodium hydroxide solution (1 : 25) is added to solutions of aluminum salts (1 : 20), a white gelatinous precipitate is formed. When excess sodium hydroxide solution (1 : 25) is then added, the precipitate dissolves.

(3) When ammonia TS is added to solutions of aluminum salts until a precipitate is slightly formed and then 5 drops of alizarin S solution (1 : 1,000) are added, the color of the precipitate changes to red.

Ammonium Salt When excess sodium hydroxide solution (1 : 25) is added to ammonium salts and they are warmed, a gas with the odor of ammonia is evolved. This gas changes red litmus paper moistened with water from red to blue.

Benzoate (1) When diluted hydrochloric acid (1 : 4) is added to solutions of benzoates (1 : 20) to acidify, a crystalline precipitate is formed. The separated precipitate, when washed well with cold water and dried, melts between about 121 and 123 °C.

(2) When ferric chloride solution (1 : 10) is added to neutral solutions of benzoates (1 : 20), a light-yellow-red precipitate is formed. When diluted hydrochloric acid (1 : 4) is then added, the color of the precipitate changes to white.

Bicarbonate (1) When diluted hydrochloric acid (1 : 4) is added to bicarbonates, a gas is evolved and an effervescence occurs. When the evolved gas is passed into calcium hydroxide TS, a white precipitate is immediately formed. (Common with carbonates)

(2) When magnesium sulfate solution (1 : 10) is added to solutions of bicarbonates (1 : 20), no precipitate is formed at ordinary temperature but a white precipitate is formed when they are boiled.

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(3) When phenolphthalein TS is added to solutions of bicarbonates, no or a faint pink color is exhibited. (To discriminate from carbonates)

Bromate (1) When 2 to 3 drops of silver nitrate solution (1 : 50) are added to solutions of bromates (1 : 20) acidified with nitric acid, a white crystalline precipitate is formed and this precipitate dissolves, when heated. When 1 drop of freshly prepared sodium nitrite solution (1 : 10) is then added, a light yellow precipitate is formed.

(2) When 5 to 6 drops of freshly prepared sodium nitrite solution (1 : 10) are added to solutions of bromates (1 : 20) acidified with nitric acid, a yellow to red-brown color is developed.

Calcium Salt (1) When the Flame Coloration Test is performed for calcium salts, the flame develops a yellow-red color.

(2) When ammonium oxalate solution (1 : 30) is added to solutions of calcium salts, a white precipitate is formed. The separated precipitate does not dissolve, when diluted acetic acid (1 : 20) is added, and then when diluted hydrochloric acid (1 : 4) is added, the precipitate dissolves.

Carbonate (1) When diluted hydrochloric acid (1 : 4) is added to carbonates, a gas is evolved and an effervesce occurs. When the evolved gas is passed into calcium hydroxide TS, a white precipitate is immediately formed. (Common with bicarbonate)

(2) When magnesium sulfate solution (1 : 10) is added to solutions of carbonates (1 : 20), a white precipitate is formed. When diluted acetic acid (1 : 20) is then added, the precipitate dissolves.

(3) When phenolphthalein TS is added to solutions of carbonates, an extremely pink color is exhibited. (To discriminate from bicarbonates)

Chloride (1) When sulfuric acid and potassium permanganate are added to solutions of chlorides (1 : 20) and then heated, a gas with the odor of chlorine is evolved. This gas changes the color of potassium iodide - starch paper moistened with water to blue.

(2) When silver nitrate solution (1 : 50) is added to solutions of chlorides, a white precipitate is formed. The separated precipitate does not dissolve in diluted nitric acid (1 : 10), but dissolves in excess ammonia TS.

Chlorite (1) When 5 ml of diluted hydrochloric acid (1 : 4) is added to solutions of chlorites (1 : 20), a yellow gas is evolved and the solution exhibits a yellow color.

(2) When 0.1 ml of potassium permanganate solution (1 : 300) and 1 ml of diluted sulfuric acid (1 : 20) are added to 5 ml of solutions of chlorites (1 : 20), a red-purple color of the solution disappears.

Citrate (1) When 20 ml of pyridine-acetic anhydride mixture (3 : 1) is added to 1 to 2 drops of solutions of citrates (1 : 20), a red-brown color is developed.

(2) When solutions of citrates (1 : 10) are previously neutralized, an equal

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volume of diluted sulfuric is added to the neutralized solutions, then about a two-thirds volume of potassium permanganate solution (1 : 300) is added, the obtained solutions are heated until the color disappears, and bromide TS is added dropwise, a white precipitate is formed.

Cupric Salt (1) When a well polished iron fragment is immersed in solutions of cupric salts acidified with hydrochloric acid and is allowed to stand, a yellow-red metal is deposited on the surface of the fragment.

(2) When a small quantity of ammonia TS is added to solutions of cupric salts, a light blue precipitate is formed. When ammonia TS is then added, the precipitate dissolves and the solution develops a dark blue color.

(3) When freshly prepared potassium ferrocyanide solution (1 : 10) is added to solutions of cupric salts, a red-brown precipitate is formed. The separated precipitate does not dissolve in diluted acetic acid (1 : 20), but it dissolves in ammonia TS and the solution develops a dark blue color.

Ferric Salt (1) When freshly prepared potassium ferrocyanide solution (1 : 10) is added to weakly acidic solutions of ferric salts, a blue precipitate is formed. The precipitate does not dissolve when diluted hydrochloric acid (1 : 4) or diluted nitric acid (1 : 10) is then added.

(2) When sodium hydroxide solution (1 : 25) or ammonia TS is added to solutions of ferric salts, a red-brown gelatinous precipitate is formed. When sodium sulfide TS is then added, the color of the precipitate changes to black. When diluted hydrochloric acid (1 : 4) is added to the separated precipitate, the precipitate dissolves and white turbidity appears.

(3) When ammonium thiocyanate solution (2 : 25) is added to a neutral to weakly acidic solutions of ferric salts, a red color is developed and does not disappear when hydrochloric acid is added.

Ferrous Salt (1) When freshly prepared potassium ferricyanide solution (1 : 10) is added to weakly acidic solutions of ferrous salts, a blue precipitate is formed. When diluted hydrochloric acid (1 : 4) or diluted nitric acid (1 : 10) is then added, the precipitate does not dissolve.

(2) When sodium hydroxide solution (1 : 25) or ammonia TS is added to solutions of ferrous salts, a white gelatinous precipitate is formed. (By shaking, the color of the precipitate quickly changes to grayish green, then gradually changes to red-brown.)

When sodium sulfide TS is then added, the color of the precipitate changes to black. The separated precipitate dissolves, when diluted hydrochloric acid (1 : 4) is added.

Glycerophosphate (1) When ammonium molybdate TS is added to solutions of glycerophosphates, no precipitate is formed unless they are heated, but a yellow precipitate is formed by boiling for a long time.

(2) When glycerophosphates are mixed with an equal volume of powdered

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potassium hydrogen sulfate and then gently heated over a free flame, the pungent odor of acrolein is evolved.

Hypochlorite (1) When 2 ml of hydrochloric acid is added to 5 ml of hypochlorite solutions, a gas is evolved and an effervesce occurs.

(2) When 1 ml of sodium hydroxide solutions (1 : 2,500) and 0.2 ml of potassium iodide TS are added to solutions of hypochlorite, yellow color is developed. When 0.5 ml of starch TS is then added, the solutions exhibit a deep blue color.

(3) When 0.1 ml of potassium permanganate solution (1 : 300) and 0.1 ml of diluted sulfuric acid (1 : 20) are added to 5 ml of solutions of chlorite (1 : 20), the red-purple color of solution does not fade (To discriminate from chlorites)

Lactate When potassium permanganate solution (1 : 50) is added to solutions of lactates (1 : 20) acidified with sulfuric acid and then heated, the odor of acetaldehyde is evolved.

Magnesium Salt When ammonium chloride solution (1 : 10) and ammonium carbonate TS are added to solutions of magnesium salts, no precipitate is formed, and when disodium phosphate solution (1 : 10) is then added, a white crystalline precipitate is formed. When ammonia TS is added, the separated precipitate does not dissolve.

Nitrate (1) When an equal volume of sulfuric acid is added to a solution of nitrate, the mixture is shaken well and cooled, and ferrous sulfate TS is superimposed, a dark brown ring is produced at the junction of the two liquids.

(2) When potassium permanganate solution (1 : 300) is added to solutions of nitrates acidified with sulfuric acid, the red-purple color of solution does not fade. (To discriminate from nitrites)

Nitrite (1) When solutions of nitrites (1 : 20) are acidified with diluted sulfuric acid (1 : 20), a yellow-brown gas with a characteristic odor is evolved. When a small amount of ferrous sulfate crystals is then added, a dark brown color is exhibited.

(2) When 2 to 3 drops of potassium iodide TS are added to solutions of nitrites and then diluted hydrochloric acid (1 : 4) is dropwise added, a yellow-brown color is exhibited, and then a black-purple precipitate is formed. When starch TS is then added, the solutions exhibit a deep blue color.

Peroxide (1) When an equal volume of ethyl acetate and 1 or 2 drops of potassium dichromate solution (3 : 40) are added to solutions of peroxides, and then the solutions are acidified with diluted sulfuric acid (1 : 20), a blue color develops in the water layer. When the mixture is shaken immediately and allowed to stand, the blue color is transferred to the ethyl acetate layer.

(2) When potassium permanganate solution (1 : 300) is dropwise added to solutions of peroxides acidified with sulfuric acid, an effervescence occurs and the color of the solution disappears.

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Phosphate (Orthophosphate) (1) When silver nitrate solution (1 50) is added to neutral solutions of phosphates, a yellow precipitate is formed. When diluted nitric acid (1 10) or ammonia TS is then added, the precipitate dissolves.

(2) When ammonium molybdate TS is added to neutral solutions of phosphates or solutions of phosphates acidified with nitric acid and then the solutions are warmed, a yellow precipitate is formed. When sodium hydroxide solution (1 25) or ammonia TS is then added, the precipitate dissolves.

Potassium Salt (1) When the Flame Coloration Test is performed on potassium salts, the flame develops pale purple color. When the flame is a yellow color, a red-purple color is seen through cobalt glass.

(2) When freshly prepared sodium hydrogen tartrate solution (1 10) is added to neutral solutions of potassium salts (1 20), a white crystalline precipitate is formed. (Rubbing the inside wall of the test tube with a glass rod accelerates the formation of the precipitate.) The collected precipitate dissolves in ammonia TS, sodium hydroxide solution (1 25), or anhydrous sodium carbonate solution (1 8).

Sodium Salt (1) When Flame Coloration Test is performed on sodium salts, the flame develops yellow color.

(2) When solutions of sodium salts (1 20) are neutralized and potassium pyroantimonate TS is added to the neutralized solutions, a white crystalline precipitate is formed. (Rubbing the inside wall of the test tube with a glass rod accelerates the formation of the precipitate.)

Succinate When the pH of succinate solutions (1 20) is adjusted to 6 to 7 and 1 ml of ferric chloride solution (1 10) is added to 5 ml of the succinate solution, a brown precipitate is formed.

Sulfate (1) When barium chloride solution (3 25) is added to solutions of sulfates, a white precipitate is formed. When hydrochloric acid or diluted nitric acid (1 10) is then added, the precipitate does not dissolve.

(2) When lead acetate TS is added to neutral solutions of sulfates, a white precipitate is formed. When ammonium acetate solution (1 10) is then added, the precipitate dissolves.

(3) When an equal volume of diluted hydrochloric acid (1 4) is added to solutions of sulfates, no white turbidity nor odor of sulfur dioxide is produced. (To discriminate from sulfites)

Sulfite and Bisulfite (1) When iodine - potassium iodide TS is dropwise added to solutions of sulfites or bisulfites acidified with acetic acid, the color of the TS disappears.

(2) When an equal volume of diluted hydrochloric acid (1 4) is added to solutions of sulfites or bisulfites (1 20) acidified with acetic acid, the odor of sulfur dioxide is evolved and no turbidity is produced. When 1 drop of sodium sulfide TS is

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then added, white turbidity appears immediately, and then it changes to a yellow precipitate.

Tartrate (1) When solutions of tartrates (1 : 20) are neutralized and silver nitrate solution (1 : 50) is added to them, a white precipitate is formed. The collected precipitate dissolves in nitric acid. Also, the collected precipitate dissolves and silver mirror is gradually formed when ammonia TS is added and the obtained liquid is warmed.

(2) When 2 drops of diluted acetic acid (1 : 4), 1 drop of ferrous sulfate TS, and 2 to 3 drops of hydrogen peroxide TS are added to solutions of tartrates (1 : 20), and then excess sodium hydroxide solution (1 : 25) is added, a red-purple to purple color is developed.

(3) When 2 to 3 drops of resorcinol solution (1 : 50) and 2 to 3 drops of potassium bromide solution (1 : 10) are added to 5 ml of sulfuric acid, and the obtained solution is added to 2 to 3 drops of a solution of a tartrate (1 : 20), then heated for 5 to 10 minutes on a water bath, a dark blue color is developed. Then, when the solution is cooled and poured into an excess amount of water, a red color is developed.

Thiocyanate (1) When excess silver nitrate solution (1 : 10) is added to solutions of thiocyanates, a white precipitate is formed. The collected precipitate does not dissolve in diluted nitric acid (1 : 10) but dissolves in aqueous ammonia.

(2) When ferric chloride solution (1 : 10) is added to solutions of thiocyanates, a red color is developed and does not fade with hydrochloric acid.

Zinc Salt (1) When sodium sulfide TS is added to neutral to alkaline solutions of zinc salts, a whitish precipitate is formed. The collected precipitate does not dissolve when diluted acetic acid (1 : 20) is added, but it dissolves when diluted hydrochloric acid (1 : 4) is further added.

(2) When freshly prepared potassium ferrocyanide solution (1 : 10) is added to solutions of zinc salts, a white precipitate is formed. The collected precipitate does not dissolve in diluted hydrochloric acid (1 : 4), but dissolves in sodium hydroxide solution (1 : 25).