

## (191) IDENTIFICATION TESTS—GENERAL

Under this heading are placed tests that are frequently referred to in the Pharmacopeia for the identification of official articles. Before using any acid or base to modify the pH of the sample solution, make sure that the added substance will not interfere with the results of the test. [NOTE—The tests are not intended to be applicable to mixtures of substances unless so specified.]

**Acetate**—Dissolve about 30 mg of the substance to be examined in 3 mL of water, or use 3 mL of the prescribed solution. Adjust the pH of the solution with sodium hydroxide to slightly alkaline. Add 0.25 mL of lanthanum nitrate TS. If a white precipitate is formed, filter the solution. Add successively 0.1 mL of iodine and potassium iodide TS 3 and 0.1 mL of ammonia TS 2 to the solution. If no blue color is observed, heat carefully to boiling. In the presence of acetates, a dark color develops or a blue precipitate is formed. With neutral solutions of acetates, ferric chloride TS produces a red color that is destroyed by the addition of mineral acids.

**Aluminum**—With 6 N ammonium hydroxide, solutions of aluminum salts yield a gelatinous, white precipitate that is insoluble in an excess of 6 N ammonium hydroxide. 1 N sodium hydroxide or sodium sulfide TS produces the same precipitate, which dissolves in an excess of either of these reagents.

**Ammonium**—Add 0.2 g of magnesium oxide to the solution under test. Pass a current of air through the mixture, and direct the gas that escapes to just beneath the surface of an indicator solution, prepared by mixing 1 mL of 0.1 M hydrochloric acid and 0.05 mL of methyl red TS 2. In the presence of ammonium, the color of the indicator solution is changed to yellow. After directing the gas into the indicator solution for a sufficient period of time, add 1 mL of freshly prepared sodium cobaltinitrite TS to the indicator solution. Upon the addition of the sodium cobaltinitrite TS, a yellow precipitate is formed when ammonium is present.

**Antimony**—With hydrogen sulfide, solutions of antimony (III) compounds, strongly acidified with hydrochloric acid, yield an orange precipitate of antimony sulfide that is insoluble in 6 N ammonium hydroxide, but is soluble in ammonium sulfide TS.

**Barium**—Solutions of barium salts yield a white precipitate with 2 N sulfuric acid. This precipitate is insoluble in hydrochloric acid and in nitric acid. Barium salts impart a yellowish-green color to a nonluminous flame that appears blue when viewed through green glass.

**Benzoate**—In neutral solutions, benzoates yield a salmon-colored precipitate with ferric chloride TS. In moderately concentrated solutions, benzoates yield a precipitate of benzoic acid upon acidification with 2 N sulfuric acid. This precipitate is readily soluble in ethyl ether.

**Bicarbonate**—See *Carbonate*.

**Bismuth**—When dissolved in a slight excess of nitric acid or hydrochloric acid, bismuth salts yield a white precipitate upon dilution with water. This precipitate is colored brown by hydrogen sulfide, and the resulting compound dissolves in a warm mixture of equal parts of nitric acid and water.

**Bisulfite**—See *Sulfite*.

**Borate**—To 1 mL of a borate solution, acidified with hydrochloric acid to litmus, add 3 or 4 drops of iodine TS and 3 or 4 drops of polyvinyl alcohol solution (1 in 50): an intense blue color is produced. When a borate is treated with sulfuric acid, methanol is added, and the mixture is ignited, it burns with a green-bordered flame.

**Bromide**—Solutions of bromides, upon the addition of chlorine TS, dropwise, liberate bromine, which is dissolved by shaking with chloroform, coloring the chloroform red to reddish brown. Silver nitrate TS produces in solutions of bromides a yellowish-white precipitate that is insoluble in nitric acid and is slightly soluble in 6 N ammonium hydroxide.

**Calcium**—Solutions of calcium salts form insoluble oxalates when treated as follows. To a solution of the calcium salt (1 in 20) add 2 drops of methyl red TS, and neutralize with 6 N ammonium hydroxide. Add 3 N hydrochloric acid, dropwise, until the solution is acid to the indicator. Upon the addition of ammonium oxalate TS, a white precipitate is formed. This precipitate is insoluble in 6 N acetic acid but dissolves in hydrochloric acid. Calcium salts moistened with hydrochloric acid impart a transient yellowish-red color to a nonluminous flame.

**Carbonate**—Carbonates and bicarbonates effervesce with acids, evolving a colorless gas that, when passed into calcium hydroxide TS, produces a white precipitate immediately. A cold solution (1 in 20) of a soluble carbonate is colored red by phenolphthalein TS, while a similar solution of a bicarbonate remains unchanged or is only slightly colored.

**Chlorate**—Solutions of chlorates yield no precipitate with silver nitrate TS. The addition of sulfurous acid to this mixture produces a white precipitate that is insoluble in nitric acid, but is soluble in 6 N ammonium hydroxide. Upon ignition, chlorates yield chlorides, recognizable by appropriate tests. When sulfuric acid is added to a dry chlorate, decrepitation occurs, and a greenish yellow-gas is evolved. [*Caution—Use only a small amount of chlorate for this test, and exercise extreme caution in performing it.*]

**Chloride**—With silver nitrate TS, solutions of chlorides yield a white, curdy precipitate that is insoluble in nitric acid but is soluble in a slight excess of 6 N ammonium hydroxide. When testing amine (including alkaloidal) hydrochlorides that do not respond to the above test, add one drop of diluted nitric acid and 0.5 mL of silver nitrate TS to a solution of the substance being examined containing, unless otherwise directed in the monograph, about 2 mg of chloride ion in 2 mL: a white, curdy precipitate is formed. Centrifuge the mixture without delay, and decant the supernatant layer. Wash the precipitate with three

1-mL portions of nitric acid solution (1 in 100), and discard the washings. Add ammonia TS dropwise to this precipitate. It dissolves readily. When a monograph specifies that an article responds to the test for dry chlorides, mix the solid to be tested with an equal weight of manganese dioxide, moisten with sulfuric acid, and gently heat the mixture: chlorine, which is recognizable by the production of a blue color with moistened starch iodide paper, is evolved.

**Citrate**—To 15 mL of pyridine add a few mg of a citrate salt, dissolved or suspended in 1 mL of water, and shake. To this mixture add 5 mL of acetic anhydride, and shake: a light red color is produced.

**Cobalt**—Solutions of cobalt salts (1 in 20) in 3 N hydrochloric acid yield a red precipitate when heated on a steam bath with an equal volume of a hot, freshly prepared solution of 1-nitroso-2-naphthol (1 in 10) in 9 N acetic acid. Solutions of cobalt salts, when saturated with potassium chloride and treated with potassium nitrite and acetic acid, yield a yellow precipitate.

**Copper**—Solutions of cupric compounds, acidified with hydrochloric acid, deposit a red film of metallic copper upon a bright, untarnished surface of metallic iron. An excess of 6 N ammonium hydroxide, added to a solution of a cupric salt, produces first a bluish precipitate and then a deep blue-colored solution. With potassium ferrocyanide TS, solutions of cupric salts yield a reddish-brown precipitate, insoluble in diluted acids.

**Hypophosphite**—When strongly heated, hypophosphites evolve spontaneously flammable phosphine. Hypophosphites in solution yield a white precipitate with mercuric chloride TS. This precipitate becomes gray when an excess of hypophosphite is present. Solutions of hypophosphites, acidified with sulfuric acid, and warmed with cupric sulfate TS yield a red precipitate.

**Iodide**—Solutions of iodides, upon the addition of chlorine TS, dropwise, liberate iodine, which colors the solution yellow to red. When the solution is shaken with chloroform, the latter is colored violet. The iodine thus liberated gives a blue color with starch TS. Silver nitrate TS produces, in solutions of iodides, a yellow, curdy precipitate that is insoluble in nitric acid and in 6 N ammonium hydroxide.

**Iron**—Ferrous and ferric compounds in solution yield a black precipitate with ammonium sulfide TS. This precipitate is dissolved by cold 3 N hydrochloric acid with the evolution of hydrogen sulfide.

**Ferric Salts**—Acid solutions of ferric salts yield a dark blue precipitate with potassium ferrocyanide TS. With an excess of 1 N sodium hydroxide, a reddish-brown precipitate is formed. With ammonium thiocyanate TS, solutions of ferric salts produce a deep red color that is not destroyed by dilute mineral acids.

**Ferrous Salts**—Solutions of ferrous salts yield a dark blue precipitate with potassium ferricyanide TS. This precipitate is insoluble in 3 N hydrochloric acid but is decomposed by 1 N sodium hydroxide. With 1 N sodium hydroxide, solutions of ferrous salts yield a greenish-white precipitate, the color rapidly changing to green and then to brown when shaken.

**Lactate**—When solutions of lactates are acidified with sulfuric acid, potassium permanganate TS is added, and the mixture is heated, acetaldehyde is evolved. This can be detected by allowing the vapor to come into contact with a filter paper that has been moistened with a freshly prepared mixture of equal volumes of 20% aqueous morpholine and sodium nitroferricyanide TS: a blue color is produced.

**Lead**—With 2 N sulfuric acid, solutions of lead salts yield a white precipitate that is insoluble in 3 N hydrochloric or 2 N nitric acid, but is soluble in warm 1 N sodium hydroxide and in ammonium acetate TS. With potassium chromate TS, solutions of lead salts, free or nearly free from mineral acids, yield a yellow precipitate that is insoluble in 6 N acetic acid but is soluble in 1 N sodium hydroxide.

**Lithium**—With sodium carbonate TS, moderately concentrated solutions of lithium salts, made alkaline with sodium hydroxide, yield a white precipitate on boiling. The precipitate is soluble in ammonium chloride TS. Lithium salts moistened with hydrochloric acid impart an intense crimson color to a nonluminous flame. Solutions of lithium salts are not precipitated by 2 N sulfuric acid or soluble sulfates (*distinction from strontium*).

**Magnesium**—Solutions of magnesium salts in the presence of ammonium chloride yield no more than a slightly hazy precipitate when neutralized with ammonium carbonate TS, but on the subsequent addition of dibasic sodium phosphate TS, a white, crystalline precipitate, which is insoluble in 6 N ammonium hydroxide, is formed.

**Manganese**—With ammonium sulfide TS, solutions of manganous salts yield a salmon-colored precipitate that dissolves in acetic acid.

**Mercury**—When applied to bright copper foil, solutions of mercury salts, free from an excess of nitric acid, yield a deposit that upon rubbing, becomes bright and silvery in appearance. With hydrogen sulfide, solutions of mercury compounds yield a black precipitate that is insoluble in ammonium sulfide TS and in boiling 2 N nitric acid.

**Mercuric Salts**—Solutions of mercuric salts yield a yellow precipitate with 1 N sodium hydroxide. They yield also, in neutral solutions with potassium iodide TS, a scarlet precipitate that is very soluble in an excess of the reagent.

**Mercurous Salts**—Mercurous compounds are decomposed by 1 N sodium hydroxide, producing a black color. With hydrochloric acid, solutions of mercurous salts yield a white precipitate that is blackened by 6 N ammonium hydroxide. With potassium iodide TS, a yellow precipitate, that may become green upon standing, is formed.

**Nitrate**—When a solution of a nitrate is mixed with an equal volume of sulfuric acid, the mixture is cooled, and a solution of ferrous sulfate is superimposed, a brown color is produced at the junction of the two liquids. When a nitrate is heated with sulfuric acid and metallic copper, brownish-red fumes are evolved. Nitrates do not decolorize acidified potassium permanganate TS (*distinction from nitrites*).

**Nitrite**—When treated with dilute mineral acids or with 6 N acetic acid, nitrites evolve brownish-red fumes. The solution colors starch-iodide paper blue.

**Oxalate**—Neutral and alkaline solutions of oxalates yield a white precipitate with calcium chloride TS. This precipitate is insoluble in 6 N acetic acid but is dissolved by hydrochloric acid. Hot acidified solutions of oxalates decolorize potassium permanganate TS.

**Permanganate**—Solutions of permanganates acidified with sulfuric acid are decolorized by hydrogen peroxide TS and by sodium bisulfite TS, in the cold, and by oxalic acid TS, in hot solution.

**Peroxide**—Solutions of peroxides slightly acidified with sulfuric acid yield a deep blue color upon the addition of potassium dichromate TS. On shaking the mixture with an equal volume of ethyl ether and allowing the liquids to separate, the blue color is found in the ethyl ether layer.

**Phosphate**—[NOTE—Where the monograph specifies the identification test for *Phosphate*, use the tests for orthophosphates, unless the instructions specify the use of the pyrophosphate tests or indicate that the product is to be ignited before performing the test.] With silver nitrate TS, neutral solutions of orthophosphates yield a yellow precipitate that is soluble in 2 N nitric acid and in 6 N ammonium hydroxide. With ammonium molybdate TS, acidified solutions of orthophosphates yield a yellow precipitate that is soluble in 6 N ammonium hydroxide. This precipitate may be slow to form. With silver nitrate TS, pyrophosphates obtained by ignition yield a white precipitate that is soluble in 2 N nitric acid and in 6 N ammonium hydroxide. With ammonium molybdate TS, a yellow precipitate that is soluble in 6 N ammonium hydroxide is formed.

**Potassium**—Potassium compounds impart a violet color to a nonluminous flame, but the presence of small quantities of sodium masks the color unless the yellow color produced by sodium is screened out by viewing through a blue filter that blocks emission at 589 nm (sodium) but is transparent to emission at 404 nm (potassium). Traditionally, cobalt glass has been used, but other suitable filters are commercially available. In neutral, concentrated or moderately concentrated solutions of potassium salts (depending upon the solubility and the potassium content), sodium bitartrate TS produces a white crystalline precipitate that is soluble in 6 N ammonium hydroxide and in solutions of alkali hydroxides and carbonates. The formation of the precipitate, which is usually slow, is accelerated by stirring or rubbing the inside of the test tube with a glass rod. The addition of a small amount of glacial acetic acid or alcohol also promotes the precipitation.

**Salicylate**—In moderately dilute solutions of salicylates, ferric chloride TS produces a violet color. The addition of acids to moderately concentrated solutions of salicylates produces a white, crystalline precipitate of salicylic acid that melts between 158° and 161°.

**Silver**—With hydrochloric acid, solutions of silver salts yield a white, curdy precipitate that is insoluble in nitric acid, but is readily soluble in 6 N ammonium hydroxide. A solution of a silver salt to which 6 N ammonium hydroxide and a small quantity of formaldehyde TS are added deposits, upon warming, a mirror of metallic silver upon the sides of the container.

**Sodium**—Unless otherwise specified in an individual monograph, prepare a solution to contain 0.1 g of the sodium compound in 2 mL of water. Add 2 mL of 15% potassium carbonate, and heat to boiling. No precipitate is formed. Add 4 mL of potassium pyroantimonate TS, and heat to boiling. Allow to cool in ice water and, if necessary, rub the inside of the test tube with a glass rod. A dense precipitate is formed. Sodium compounds impart an intense yellow color to a nonluminous flame.

**Sulfate**—With barium chloride TS, solutions of sulfates yield a white precipitate that is insoluble in hydrochloric acid and in nitric acid. With lead acetate TS, neutral solutions of sulfates yield a white precipitate that is soluble in ammonium acetate TS. Hydrochloric acid produces no precipitate when added to solutions of sulfates (*distinction from thiosulfates*).

**Sulfite**—When treated with 3 N hydrochloric acid, sulfites and bisulfites yield sulfur dioxide, which blackens filter paper moistened with mercurous nitrate TS.

**Tartrate**—Dissolve a few mg of a tartrate salt in 2 drops of sodium metaperiodate solution (1 in 20). Add a drop of 1 N sulfuric acid, and after 5 minutes add a few drops of sulfurous acid followed by a few drops of fuchsin-sulfurous acid TS: a reddish-pink color is produced within 15 minutes.

**Thiocyanate**—With ferric chloride TS, solutions of thiocyanates yield a red color that is not destroyed by moderately concentrated mineral acids.

**Thiosulfate**—With hydrochloric acid, solutions of thiosulfates yield a white precipitate that soon turns yellow, and sulfur dioxide, which blackens filter paper moistened with mercurous nitrate TS. The addition of ferric chloride TS to solutions of thiosulfates produces a dark violet color that quickly disappears.

**Zinc**—In the presence of sodium acetate, solutions of zinc salts yield a white precipitate with hydrogen sulfide. This precipitate is insoluble in acetic acid, but is dissolved by 3 N hydrochloric acid. Ammonium sulfide TS produces a similar precipitate in neutral and in alkaline solutions. With potassium ferrocyanide TS, zinc salts in solution yield a white precipitate that is insoluble in 3 N hydrochloric acid.