

## <191> IDENTIFICATION TESTS—GENERAL

### INTRODUCTION

Procedures in this chapter are referenced in monographs for the identification of official articles and their components. Any acids, bases, or other reagents used in these procedures should not interfere with the results. Volumes may be scaled proportionally unless otherwise indicated. All of the tests include approximate amounts, except where specified. Instrumental techniques described in this chapter may be used in lieu of chemical identification tests. Those instrumental techniques are not exhaustive and other techniques, such as nuclear magnetic resonance, ion-selective electrodes, and near-infrared, may be used in lieu of a chemical identification test provided that they are suitable and validated. Unless otherwise specified in the monograph, if a chemical identification test is selected for an ion, then all chemical test procedures listed for the ion shall be met. If an instrumental identification test is selected, then only one instrumental technique is required for the ion(s).

### CHEMICAL IDENTIFICATION TESTS

#### Acetate

- **A.** 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 be slightly alkaline. Add 0.25 mL of lanthanum nitrate test solution (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.
- **B.** With neutral solutions of acetates, ferric chloride TS produces a red color that is destroyed by the addition of mineral acids.

#### Aluminum

- **A.** By using 6 N ammonium hydroxide, solutions of aluminum salts yield a gelatinous, white precipitate that is insoluble in an excess of 6 N ammonium hydroxide.
- **B.** 1 N sodium hydroxide or sodium sulfide TS with solutions of aluminum salts produces a similar gelatinous, white precipitate, which dissolves in an excess of either of the same reagents.

#### Ammonium

- **A.** 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 the indicator solution prepared previously 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

- **A.** 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

- **A.** Solutions of barium salts yield a white precipitate with the addition of 2 N sulfuric acid. This precipitate is insoluble in hydrochloric acid and in nitric acid.

#### Benzoate

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

#### Bicarbonate

- **A.** Bicarbonates effervesce with acids, evolving a colorless gas that, when passed into calcium hydroxide TS, produces a white precipitate immediately.
- **B.** A cold solution (1 in 20) of a soluble bicarbonate or the solution prescribed in the specific monograph remains unchanged or is only slightly colored by phenolphthalein TS.

#### Bismuth

- **A.** 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

- **A.** To 1 mL of a borate solution acidified with hydrochloric acid, 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.

#### Bromide

- **A.** 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.
- **B.** Silver nitrate TS produces, in solutions of bromides, a yellowish-white precipitate that is insoluble in nitric acid and is slightly soluble when 6 N ammonium hydroxide is added.

#### Calcium

- **A.** Solutions of calcium salts form insoluble oxalates when treated as follows. To a solution of the calcium salt (1 in 20) or the solution prescribed in the specific monograph, 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 when 6 N acetic acid is added but dissolves in hydrochloric acid.

**Carbonate**

- A. Carbonates effervesce with acids, evolving a colorless gas that, when passed into calcium hydroxide TS, produces a white precipitate immediately.
- B. A cold solution (1 in 20) of a soluble carbonate or the solution prescribed in the specific monograph is colored red by phenolphthalein TS, whereas a similar solution of a bicarbonate remains unchanged or is only slightly colored.

**Chlorate**

- A. 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.
- B. Upon ignition, chlorates yield chlorides, recognizable by appropriate tests.
- C. 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.]

**Change to read:****Chloride**

- A. 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 previous test, add 1 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. ▲ (USP 1-May-2021)

**Citrate**

- A. To 15 mL of pyridine add a solution or suspension of a few milligrams of a citrate salt in 1 mL of water or the solution prescribed in the specific monograph, and shake. To this mixture add 5 mL of acetic anhydride, and shake: a light red color is produced.

**Cobalt**

- A. Solutions of cobalt salts (1 in 20) in 3 N hydrochloric acid or the solution prescribed in the specific monograph 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.
- B. Solutions of cobalt salts, when saturated with potassium chloride and treated with potassium nitrite and acetic acid, yield a yellow precipitate.

**Copper**

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

**Hypophosphite**

- A. Hypophosphites in solution yield a white precipitate with mercuric chloride TS. This precipitate becomes gray when an excess of hypophosphite is present.
- B. Solutions of hypophosphites, acidified with sulfuric acid and warmed with cupric sulfate TS, yield a red precipitate.

**Iodide**

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

**Iron**

- A. Ferrous and ferric compounds in solution yield a black precipitate with ammonium sulfide TS. This precipitate dissolves in cold 3 N hydrochloric acid and evolves hydrogen sulfide.

**Ferric salts**

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

**Ferrous salts**

- A. 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.
- B. With 1 N sodium hydroxide, solutions of ferrous salts yield a greenish-white precipitate; when shaken, the color changes rapidly to green and then to brown.

**Lactate**

- A. When solutions of lactates are acidified with sulfuric acid, potassium permanganate TS is added; the mixture is heated, and acetaldehyde evolves. Acetaldehyde 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 nitroferrocyanide TS: a blue color is produced.

**Lead**

- A. 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.

- **B.** 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**

- **A.** 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.
- **B.** Solutions of lithium salts are not precipitated by 2 N sulfuric acid or soluble sulfates (distinction from strontium).

#### **Magnesium**

- **A.** Solutions of magnesium salts in the presence of ammonium chloride yield NMT 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**

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

#### **Mercury**

- **A.** 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.
- **B.** 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**

- **A.** Solutions of mercuric salts yield a yellow precipitate with 1 N sodium hydroxide.
- **B.** Solutions of mercuric salts yield, in neutral solutions with potassium iodide TS, a scarlet precipitate that is very soluble in an excess of the reagent.

#### **Mercurous salts**

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

#### **Nitrate**

- **A.** 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.
- **B.** When a nitrate is heated with sulfuric acid and metallic copper, brownish-red fumes evolve.
- **C.** Nitrates do not decolorize acidified potassium permanganate TS (distinction from nitrites).

#### **Nitrite**

- **A.** 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**

- **A.** 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.
- **B.** Hot acidified solutions of oxalates decolorize potassium permanganate TS.

#### **Permanganate**

- **A.** 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 a hot solution.

#### **Peroxide**

- **A.** 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.]

#### **Orthophosphates**

- **A.** 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.
- **B.** 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.

#### **Pyrophosphates**

- **A.** With silver nitrate TS, pyrophosphates obtained by ignition yield a white precipitate that is soluble in 2 N nitric acid and soluble in 6 N ammonium hydroxide.
- **B.** With ammonium molybdate TS, pyrophosphates obtained by ignition yield a yellow precipitate that is soluble in 6 N ammonium hydroxide.

#### **Potassium**

- **A.** 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**

- **A.** In moderately concentrated solutions of salicylates, ferric chloride TS produces a violet color.
- **B.** Addition of acids to moderately concentrated solutions of salicylates produces a white, crystalline precipitate of salicylic acid that melts between 158° and 161°.

**Silver**

- **A.** 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.
- **B.** 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**

- **A.** 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.

**Sulfate**

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

**Sulfite**

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

**Tartrate**

- **A.** Dissolve a few mg of a tartrate salt in 2 drops of sodium metaperiodate solution (1 in 20). Add 1 drop of 1 N sulfuric acid and after 5 min 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 min.

**Thiocyanate**

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

**Thiosulfate**

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

**Zinc**

- **A.** 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.
- **B.** Ammonium sulfide TS produces a similar precipitate in neutral and in alkaline solutions.
- **C.** With potassium ferrocyanide TS, zinc salts in solution yield a white precipitate that is insoluble in 3 N hydrochloric acid.

**INSTRUMENTAL IDENTIFICATION TESTS**

Instrumental techniques described in this section may be used in lieu of procedures described in *Chemical Identification Tests*. Instrumental techniques provide flexibility in the choice of identification tests. All instrumental techniques shall follow method validation procedures for identification tests (see *Validation of Compendial Procedures* (1225), *Validation, Data Elements Required for Validation, Category IV*). *Instrumental Identification Tests* must demonstrate specificity. In addition, other suitable, validated instrumental techniques may be used.

The selection of the appropriate sample preparation depends on the material under test and must be appropriate for the technique being used. The analyst may use any of the following preparation procedures, with the appropriate verification. An electronic library spectrum of the Reference Standard may be used in comparison to the test sample provided adequate specificity is maintained. When using solvents, the solvent must be free of interfering species. Use *USP Reference Standards* where available (see *General Notices, 5.80 USP Reference Standards*).

**• IDENTIFICATION USING X-RAY FLUORESCENCE SPECTROMETRY**

X-ray fluorescence spectrometry (XRF) can typically be used for identification of elements with atomic numbers from magnesium through uranium. The actual range of elements for which the instrument is capable is dependent on the instrument design. Guidance regarding the use of XRF may be found in *X-Ray Fluorescence Spectrometry* (735).

**Sample preparation**

**Powders/solids:** Powders and solids can be used without further manipulation, or material can be made into pellets as described in (735).

**Neat liquids:** Liquids can be used without further manipulation, provided the liquid is a single phase, is compatible with the sample holder, and has sufficiently low volatility.

**Samples in solution:** Samples may be dissolved in an appropriate solvent.

**Procedure**

**Blank:** For *Powders/solids* and *Neat liquids*, prepare a blank sample holder. For *Samples in solution*, use the solvent as a blank.

**Reference standard and Sample:** Prepare each *Reference standard* and *Sample* by using sufficient material as required for the specific manufacturer's instrumentation.

**Analysis:** Analyze the *Blank*, *Reference standard*, and *Sample* according to the manufacturer's suggestions for the specific instrument. If there is any interference, use the *Blank* to make the correction. The spectrum of the *Reference standard* exhibits radiation characteristic of the element(s) under investigation. The energy bands of the *Sample* exhibit radiation characteristic of the element(s) under investigation and compare qualitatively with that of the *Reference standard*.

**• IDENTIFICATION USING ATOMIC SPECTROSCOPY TECHNIQUES: ATOMIC ABSORPTION SPECTROSCOPY, INDUCTIVELY COUPLED PLASMA–OPTICAL EMISSION SPECTROSCOPY, INDUCTIVELY COUPLED PLASMA–MASS SPECTROMETRY**

Atomic absorption (AA) spectroscopy techniques can be used for the identification of many elements. Guidance regarding the use of AA spectroscopy may be found in *Atomic Absorption Spectroscopy* (852). Guidance regarding the use of

inductively coupled plasma–optical emission spectroscopy (ICP–OES) or inductively coupled plasma–mass spectrometry (ICP–MS) may be found in *Plasma Spectrochemistry* (730).

**Sample preparation:** Samples should be dissolved in an appropriate solvent. If dissolving the sample is not possible, digestion may be necessary.

**Procedure**

**Blank:** Prepare an appropriate solution that does not contain the analyte(s) of interest and is compatible with the technique being used.

**Standard solution:** Standards must contain the analyte of interest. All solutions should be matrix-matched where possible.

**Sample solution:** Prepare the sample in the same solvent as the standard.

**Analysis:** Analyze the *Blank*, *Standard solution*, and *Sample solution* according to the manufacturer's suggestions for the specific instrument. When a wavelength-based technique is used, wherever possible select at least two wavelengths characteristic of the analyte(s) of interest. When a mass-based technique is used, wherever possible select at least two isotopes characteristic of the analyte(s) of interest. If two isotopes are unavailable, or if an element is monoisotopic, it may be possible to monitor the oxide of the analyte(s) ( $m + 16$ ), if one is formed. If no oxide is formed, or if multiple isotopes cannot be evaluated because of interferences or because of a limited number of isotopes, then a different technique may be required. If interfering elements are present in the sample solution, wavelengths or masses examined must be selected to unequivocally identify the analyte(s) of interest.

• **IDENTIFICATION USING ION CHROMATOGRAPHY**

Ion chromatography (IC) can be used to identify a number of anions and cations found in drug substances (for additional information, see *Ion Chromatography* (1065)).

**Apparatus:** Analyte detection will typically utilize suppressed conductivity, although other detection methods are possible, depending on the analyte (see (1065)). The ion-exchange column must be capable of separating the analyte from any other ions of the same charge known to be in the sample at a concentration  $\geq 5\%$  of the analyte.

**Procedure**

**Blank:** Use the sample solvent as a blank.

**Standard and Sample solutions:** Dissolve or dilute the sample in water. Other solvents can be used if compatible with the IC column.

**Analysis:** Analyze an equal volume of the *Blank*, Reference Standard, and sample (according to the manufacturer's suggestions for the specific instrument and column dimensions). The counterion is identified if a peak in the sample has the same retention time as the peak in the Reference Standard, and there is not a peak of  $\geq 5\%$  of its size at the same retention time in the *Blank*.

• **IDENTIFICATION BY OTHER LIQUID CHROMATOGRAPHY TECHNIQUES IN CHROMATOGRAPHY (621)**

Some of the ions suitable for IC identification can also be identified by other forms of liquid chromatography. Sample and Reference Standard concentrations, as well as injection volumes, need to be adjusted, depending on the detection technique used. All high-concentration components in the test substance should be analyzed to judge whether there is interference with the analyte of interest.

• **IDENTIFICATION USING RAMAN SPECTROSCOPY**

Raman spectroscopy can be used for identification of counterions. Guidance regarding the use of Raman spectroscopy may be found in *Raman Spectroscopy—Theory and Practice* (1858).

**Apparatus:** Prepare the spectrometer for operation according to the instrument instruction manual and the instrument manufacturer's recommendations. An instrument performance check and the quality of spectra collected should be evaluated at time of use or following manufacturer's instructions.

**Procedure**

**Reference standard and Sample:** All reference material and sample spectra should be collected using identical instrumental parameters. These instrumental parameters may be determined by the analyst based on the nature of the sample and the type of analysis and should be selected based on the quality of spectra needed. Use the appropriate sample container and/or sample holding apparatus, depending on the type of reference material and sample (powder, liquid, paste, film, or other) being analyzed. Transfer the sample or reference material into the appropriate sample container and/or holder, as needed, and acquire the spectrum for each.

**Analysis:** Qualitatively compare the Raman spectra obtained from the reference material and the *Sample*. The *Sample* complies with the identity test if the spectrum exhibits maxima only at the same wavelengths as that of a similar preparation of the corresponding USP Reference Standard, where available.

• **IDENTIFICATION USING MID-INFRARED SPECTROSCOPY**

Mid-infrared spectroscopy techniques can be used for identification of counterions. Guidance regarding the use of mid-infrared spectroscopy may be found in *Mid-Infrared Spectroscopy* (854) and *Spectroscopic Identification Tests* (197). If the monograph contains an identification test by IR (e.g., (197)) and a reference to (191), then mid-infrared cannot be used as an instrumental replacement for the chemical identification tests prescribed in this chapter.

**Procedure**

**Standard and Sample solutions:** Sample preparation may be conducted using any of the procedures described in (854) that are appropriate for the sample of concern. The standard preparation should be conducted in the same way but using the USP Reference Standard of the substance under test, where available.

**Analysis:** Record the spectra of the test specimen and the corresponding Reference Standard over the range from about 3800 to 650  $\text{cm}^{-1}$  (2.6–15  $\mu\text{m}$ ). The IR absorption spectrum of the preparation of the test specimen, previously dried under conditions specified for the corresponding Reference Standard, unless the Reference Standard is to be used without drying, exhibits maxima only at the same wavelengths as that of a similar preparation of the corresponding USP Reference Standard, where available.