⟨670⟩ AUXILIARY PACKAGING COMPONENTS

Auxiliary packaging components are articles that are used to support or enhance container–closure systems. These articles include, but are not limited to, pharmaceutical coil and desiccants for containers. The components covered in this chapter must meet the applicable requirements provided and the additional applicable requirements provided in other specified chapters.

PHARMACEUTICAL COIL

Pharmaceutical coil is used as a filling material in multiple-unit containers for solid oral dosage forms to prevent breakage of tablets or capsules during shipment. The filling material should be discarded once the bottle is opened.

- **SOLUTIONS**

Iodinated zinc chloride solution: Dissolve 20 g of zinc chloride and 6.5 g of potassium iodide in 10.5 mL of *Purified Water*. Add 0.5 g of iodine, and shake for 15 min. Filter if necessary. Protect from light.

Zinc chloride–formic acid solution: Dissolve 20 g of zinc chloride in 80 g of an 850 g/L solution of anhydrous formic acid.

1% DuPont Fiber Identification Stain No. 4[1] **solution:** Dissolve 3.8 g of powdered stain in 378.5 mL of deionized water.

- **COTTON PHARMACEUTICAL COIL**

Purified cotton is the hair of the seed of cultivated varieties of *Gossypium hirsutum* Linné, or of other species of *Gossypium* (Fam. Malvaceae). It is deprived of fatty matter and bleached, and does not contain more than traces of leaf residue, pericarp, seed coat, or other impurities. Cotton pharmaceutical coil is used in bottles of solid oral dosage forms to prevent breakage.

Identification

A. When examined under a microscope, each fiber is seen to consist of a single cell, up to about 4 cm long and 40 µm wide, in the form of a flattened tube with thick and rounded walls that are often twisted.

B. When treated with *Iodinated zinc chloride solution*, the fibers become violet.

C. To 0.1 g of fibers add 10 mL of *Zinc chloride–formic acid solution*, heat to 40°, and allow to stand for 2 h, shaking occasionally: the fibers do not dissolve.

D. Weigh about 5 g of fibers, wet with water, and squeeze out the excess. Add fibers to 100 mL of a boiling solution of a *1% DuPont Fiber Identification Stain No. 4 solution*, and gently boil for at least 1 min. Remove the fibers, rinse well in cold water, and squeeze out the excess moisture: the fibers become green.

Acidity or alkalinity: Immerse about 10 g of fibers in 100 mL of recently boiled and cooled *Purified Water*, and allow to macerate for 2 h. Decant 25-mL portions of the water, with the aid of a glass rod, into each of two dishes. To one portion add 3 drops of phenolphthalein TS, and to the other portion add 1 drop of methyl orange TS. Neither portion appears pink when viewed against a white background.

Fluorescence: Examine a layer about 5 mm in thickness under UV light at 365 nm. It displays only a slight brownish-violet fluorescence and a few yellow particles. It shows no intense blue fluorescence, apart from that which may be shown by a few isolated fibers.

Residual hydrogen peroxide concentration: Place 1 g of fibers in a beaker containing 30 mL of *Purified Water*, and stir for 3 min with a stirring rod. Pour contents into another clean container (do not squeeze sample), or alternatively, remove the fibers from the solution with clean tweezers. Remove a peroxide analytical test strip[2] from its container, and immerse the test end into the sample liquid for 2 s. Shake to remove the excess liquid, immediately insert the test strip into a suitable reflectometry instrument, and record the reading in mg/kg (ppm), and calculate residual hydrogen peroxide concentration in ppm.

For an alternate method, place 20 g in a beaker, add 400 mL of *Purified Water*, stir, add 20 mL of 20% sulfuric acid, and stir the contents. Titrate with 0.100 N potassium permanganate solution to a faint pink color that remains for 30 s. Record the amount of titer, and calculate the concentration in ppm.

NMT 50 ppm is found using either method.

Loss on Drying ⟨731⟩

Analysis: Dry 5 g of fibers in an oven at 105° to constant weight.

Acceptance criteria: NMT 8.0%

Residue on Ignition ⟨281⟩

Analysis: Place 5 g of fibers in a porcelain or platinum dish, and moisten with 2 N sulfuric acid. Gently heat the cotton until it is charred, then ignite more strongly until the carbon is completely consumed.

Acceptance criteria: NMT 0.20%

Water-soluble substances: Place 10 g of fibers in a beaker containing 1000 mL of *Purified Water*, and boil gently for 30 min, adding water as required to maintain the volume. Pour the water through a funnel into another vessel, and press out the excess water from the cotton with a glass rod. Wash the cotton in the funnel with two 250-mL portions of boiling water, pressing the cotton after each washing. Filter the combined extract and washings, and wash the filter thoroughly with hot water. Evaporate the combined extract and washings to a small volume, transfer to a tared porcelain or platinum dish, evaporate to dryness, and dry the residue at 105° to constant weight. The residue weighs NMT 0.35%.

Fatty matter: Pack 10 g of fibers in a Soxhlet extractor provided with a tared receiver, and extract with ethyl ether for 4 h at a rate such that the ether siphons over NLT 4 times per h. The ethyl ether solution in the flask shows no trace of blue, green, or brownish color. Evaporate the extract to dryness, and dry at 105° for 1 h. The weight of the residue does not exceed 0.7%.

[1] DuPont Fiber Identification Stain No. 4 is available from Pylam Products Co., 2175 East Cedar Street, Tempe, AZ 85281; www.pylamdyes.com.

[2] A suitable analysis system consisting of Reflectoquant® peroxide test strips and a RQflex® reflectometry instrument may be obtained from EMD Chemicals Inc., 480 S. Democrat Road, Gibbstown, NJ 08027; www.emdchemicals.com.

Dyes: Pack about 10 g of fibers in a narrow percolator, and extract slowly with alcohol until the percolate measures 50 mL. When observed downward through a column 20 cm in depth, the percolate may show a yellowish color, but not a blue or a green tint.

Other foreign matter: Pinches contain no oil stains or metallic particles by visual inspection.

• **RAYON PHARMACEUTICAL COIL**

Rayon pharmaceutical coil is a fibrous form of bleached, regenerated cellulose, to be used as a filler in bottles of solid oral dosage forms to prevent breakage. It consists exclusively of rayon fibers except for a few isolated foreign fibers that may be present. [NOTE—Rayon pharmaceutical coil has been found to be a potential source of dissolution problems for gelatin capsules or gelatin-coated tablets resulting from gelatin cross-linking.]

Identification

A. When treated with *Iodinated zinc chloride solution*, the fibers become violet.

B. Add 10 mL of *Zinc chloride–formic acid solution* to 0.1 g of fibers, heat to 40°, and allow to stand for 2 h, shaking occasionally: the fibers dissolve completely, except for mat rayon fibers where titanium particles remain.

C. Weigh about 5 g of fibers, wet with water, and squeeze out the excess. Add fibers to 100 mL of a boiling solution of a *1% DuPont Fiber Identification Stain No. 4 solution*, and gently boil for at least 1 min. Remove the fibers, rinse well in cold water, and squeeze out the excess moisture: the fibers become blue–green.

Acidity or alkalinity, Fluorescence, Fatty matter, Dyes, and **Other foreign matter:** Proceed as directed under *Cotton Pharmaceutical Coil*, except use rayon pharmaceutical coil. Sample weight for fatty matter is 5 g and weight of residue does not exceed 0.5%.

Loss on Drying ⟨731⟩

Analysis: Dry 5 g of fibers in an oven at 105° to constant weight.

Acceptance criteria: NMT 11.0%

Residue on Ignition ⟨281⟩**:** NMT 1.50%, determined on a 5-g test specimen

Acid-insoluble ash: To the residue obtained in the test for *Residue on Ignition*, add 25 mL of 3 N hydrochloric acid, and boil for 5 min. Collect the insoluble matter on a tared filtering crucible, wash with hot water, ignite, and weigh: the residue weighs NMT 1.25%.

Water-soluble substances: Proceed as directed under *Cotton Pharmaceutical Coil*, except to use rayon pharmaceutical coil. The residue weighs NMT 1.0%.

• **POLYESTER PHARMACEUTICAL COIL**

Polyester pharmaceutical coil is a white odorless material, to be used as a filler in bottles of solid oral dosage forms to prevent breakage.

Identification

A. Proceed as directed under *Infrared spectroscopy* in the *Test Methods* section. Determine the IR spectrum from 4000 to 650 cm^{-1} (2.5 to 15 µm). The spectrum obtained from the specimen exhibits major absorption bands only at the same wavelengths as the spectrum of USP Polyethylene Terephthalate RS.

B. Weigh about 5 g of fibers, wet with water, and squeeze out excess. Add fibers to 100 mL of a boiling solution of a *1% DuPont Fiber Identification Stain No. 4 solution*, and gently boil for at least 1 min. Remove the fibers, rinse well in cold water, and squeeze out the excess moisture: the fibers become pale orange.

Acidity or alkalinity: Proceed as directed under *Cotton Pharmaceutical Coil*, except to use polyester pharmaceutical coil.

Loss on Drying ⟨731⟩

Analysis: Dry 5 g of fibers in an oven at 105° to constant weight.

Acceptance criteria: NMT 1.0%

Residue on Ignition ⟨281⟩**:** NMT 0.5%, determined on a 5-g test specimen

Finish on fibers: The finish on fibers used for processing should comply with FDA food contact regulations.

• **TEST METHODS**

Infrared spectroscopy[3]

Apparatus: FTIR or a double-beam spectrophotometer capable of scanning from 4000 to 650 cm^{-1} (2.5–15 µm).

Change to read:

Specimen preparation: The ATR ▲*Spectroscopic Identification Tests* ⟨197⟩*, Infrared Spectroscopy: 197A*▲ (CN 1-May-2020) technique can be used as alternative methods where the Reference Standard spectra is similarly obtained.

Method 1 (potassium bromide disc)**:** Use scissors to cut polyester fibers (1–3 mg) into short lengths (less than 1 mm long), mix with 200 mg of powdered potassium bromide, and grind in a ball mill for 1–2 min. Transfer to potassium bromide-disc die, and form a disc.

Method 2 (melt film)**:** Produce film by pressing polyester fibers between TFE-fluorocarbon sheets and place between heated plates.

DESICCANTS

Desiccants are used to remove moisture from air in containers in order to protect drug products, particularly solid oral dosage forms. They are supplied in a number of different packaging materials including cotton, Kraft paper, rayon and polyester cloth bags, perforated plastic, polymer films, polymer housings or Tyvek®. The most common types of commercial desiccants are bentonite, calcium chloride, calcium oxide, molecular sieves, and silica gel. Other desiccants are subject to appropriate testing to ensure suitability for the intended application.

Where desiccants are incorporated directly into the wall or cap of packaging containers or bound by a carrier material, use unincorporated desiccant in the test methods. For desiccants that are loaded with a predetermined moisture content, perform the testing before the moisture has been loaded or after the desiccant has been regenerated.

[3] Additional information on fiber identification methods may be found in "Standard Test Methods for Identification of Fibers in Textiles". Current version of ASTM Method D276, published by ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959; www.astm.org.

• **BENTONITE**

Bentonite clay (also referred to as montmorillonite clay) is a native, colloidal, hydrated aluminum silicate.

Appearance: Grayish-white powder or pellets with a yellowish or pinkish tint

Identification—X-Ray Diffraction ⟨941⟩**:** For *Sample preparation A*, the largest peak corresponds to a *d* value between 15.0 and 17.2 Å. The major peak in the region between 1.48 and 1.54 Å from the pattern of *Sample preparation B* is between 1.492 and 1.504 Å.

Identification—precipitation: Formation of a gelatinous white precipitate

Inorganic impurities

Arsenic: NMT 10 mg/kg

Lead: NMT 15 mg/kg

Specific tests

pH ⟨791⟩**:** 4.5–10.5. Disperse 4.0 g in 200 mL of water, mix vigorously to facilitate wetting.

Loss on Drying ⟨731⟩**:** Dry a 5–10 g sample at 110° to a constant weight: it loses NMT 3.0%.

[NOTE—Conduct assay immediately after opening the original container.]

Moisture adsorption capacity

NLT 13% at 40% ± 5% relative humidity (RH) and 25 ± 2°

NLT 23% at 80% ± 5% RH and 25 ± 2°

Test methods

Identification—X-ray diffraction

Sample preparation A: Add 2 g in small portions to 100 mL of water with intense agitation. Allow to stand for 12 h to ensure complete hydration. Place 2 mL of the mixture so obtained on a suitable glass slide, and allow to air-dry at room temperature to produce an oriented film. Place the slide in a vacuum desiccator over a free surface of ethylene glycol. Evacuate the desiccator, and close the stopcock so that ethylene glycol saturates the desiccator chamber. Allow the slide to stand for 12 h.

Sample preparation B: Prepare a random powder specimen of the sample.

Analysis: *Sample preparation A* and *Sample preparation B*. Record the X-ray diffraction pattern of the samples, and determine the *d* values.

Identification—precipitation

Sample: 5 g

Analysis: Add 1 g of potassium nitrate and 3 g of anhydrous sodium carbonate to the *Sample* contained in a metal crucible, heat until the mixture has melted, and allow it to cool. Add 20 mL of boiling water to the residue, mix, filter, and wash the residue with 50 mL of water. Add 1 mL of hydrochloric acid and 5 mL of water to the residue, and filter. Add 1 mL of 10 N sodium hydroxide to the filtrate, filter, and add 3 mL of 2 M ammonium chloride.

Arsenic

Sample solution: Transfer 8.0 g of dried sample into a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), mix, and cover with a watch glass. Boil gently, with occasional stirring, for 15 min without allowing excessive foaming. Pass the hot supernatant liquid through a rapid-flow filter paper into a 200-mL volumetric flask, and wash the filter with four 25-mL portions of hot dilute hydrochloric acid (1 in 25), collecting the washings in the volumetric flask. Cool the combined filtrates to room temperature, add dilute hydrochloric acid (1 in 25) to volume and mix.

Arsenic trioxide stock solution: Accurately weigh 132.0 mg of arsenic trioxide, previously dried at 105° for 1 h, and dissolve in 5 mL of sodium hydroxide solution (1 in 5) in a 1000-mL volumetric flask. Neutralize the solution with 2 N sulfuric acid, add an additional 10 mL of 2 N sulfuric acid, and bring to volume with recently boiled and cooled water and mix.

Standard arsenic solution: Dilute the *Arsenic trioxide stock solution* to obtain solutions of suitable concentrations, adaptable to the linear or working range of the instrument. Keep in an all-glass container, and use within 3 days.

Analysis: Proceed according to *Elemental Impurities—Procedures* ⟨233⟩.

Lead

Sample solution: Transfer 3.75 g of sample into a 250-mL beaker containing 100 mL of dilute hydrochloric acid (1 in 25), stir, and cover with a watch glass. Boil for 15 min, then cool to room temperature, and pass through a rapid-flow filter paper into a 400-mL beaker. Wash the filter with four 25-mL portions of hot water, collecting the washings in the 400-mL beaker. Concentrate the combined extracts by gentle boiling to approximately 20 mL. If a precipitate forms, add 2–3 drops of nitric acid, heat to boiling, and cool to room temperature. Pass the concentrated extracts through a rapid-flow filter paper into a 50-mL volumetric flask. Transfer the remaining contents of the 400-mL beaker through the filter paper and into the flask with water. Dilute with water to volume.

Lead nitrate stock solution: Dissolve 159.8 mg of lead nitrate in 100 mL of water to which has been added 1 mL of nitric acid, then dilute with water to 1000 mL. Prepare and store this solution in glass containers free from soluble lead salts.

Standard lead solution: On the day of use, dilute the *Lead nitrate stock solution* to obtain solutions of suitable concentrations, adaptable to the linear or working range of the instrument.

Analysis: Proceed according to ⟨233⟩.

Moisture adsorption capacity

Equipment: Temperature-humidity chambers capable of controlled humidity at 40% ± 5% RH and 80% ± 5% RH at 25 ± 2° or a desiccator containing water-saturated salts that provide %RH at the required level plus an oven capable of maintaining 25 ± 2°.

Method: 5–10 g. Remove the sample from the packaging material. Where absorbents are incorporated directly into the wall or cap of packaging containers, use unincorporated desiccant. Add the sample to the humidity chamber or desiccator and measure the weight gain over time until this reaches equilibrium when two successive consecutive weighings do not differ by more than 3 mg/g of substance taken, the second weighing following an additional 3

± 1 h of storage at the required temperature and humidity conditions. Calculate the adsorption capacity as percentage weight gained over the initial sample weight. Where two absorbents are packaged in combination, the moisture adsorption capacity specification must be calculated in proportion of the mix. For example, a mixture of 60% molecular sieve and 40% silica gel, the moisture adsorption capacity would be NLT 16.6% (9.0% + 7.6%) when the test condition is 40% ± 5% RH and 25 ± 2° and the moisture adsorption capacities taken at their minimum specification.

Calculation

Molecular sieve: 60% by weight × 15% moisture adsorption capacity = NLT 9.0% moisture adsorption capacity

Silica gel: 40% by weight × 19% moisture adsorption capacity = NLT 7.6% moisture adsorption capacity

- **CALCIUM CHLORIDE, ANHYDROUS**

Identification—calcium: Passes tests

Identification—chloride: Passes test

Assay: NLT 93.0% and NMT 100.5% of calcium chloride ($CaCl_2$)

Inorganic impurities

Arsenic: NMT 3 ppm

Fluoride: NMT 0.004%

Lead: NMT 5 ppm

Magnesium and alkali salts: NMT 25 mg of residue (NMT 5.0%)

Specific tests

pH ⟨791⟩: 4.5–11.0 (1:20 aqueous solution)

Moisture adsorption capacity: NLT 28% at 80% ± 5% RH and 25 ± 2°

Test methods

Identification—calcium

Sample solution—100 mg/mL: Insoluble oxalate salts are formed when solutions of calcium salts are treated in the following manner. Using 2 drops of methyl red TS as the indicator, neutralize a 1:20 solution of a calcium salt with 6 N ammonia, then add 2.7 N hydrochloric acid, dropwise, until the solution is acid. A white precipitate of calcium oxalate forms upon the addition of ammonium oxalate TS. This precipitate is insoluble in acetic acid but dissolves in hydrochloric acid.

Identification—chloride

Sample solution—100 mg/mL: Solutions of chlorides yield with silver nitrate TS a white, curdy precipitate that is insoluble in nitric acid but soluble in a slight excess of 6 N ammonia.

Assay

Sample: 1.5 g

Analysis: Transfer the *Sample* into a 250-mL volumetric flask, dissolve it in a mixture of 100 mL of water and 5 mL of 2.7 N hydrochloric acid, dilute with water to volume, and mix. Transfer 50 mL of this solution into a suitable container and add 50 mL of water. While stirring, preferably with a magnetic stirrer, add about 30 mL of 0.05 M disodium EDTA from a 50-mL buret. Then, add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue indicator. Continue the titration to a blue endpoint. Each mL of 0.05 M disodium EDTA is equivalent to 5.55 mg of calcium chloride ($CaCl_2$).

Arsenic

Sample solution: 1 g in 10 mL

Arsenic trioxide stock solution and **Standard arsenic solution:** Proceed as directed under *Bentonite*.

Analysis: Proceed as directed under *Bentonite*.

Fluoride

Sodium fluoride solution—5 µg/mL: Transfer 2.210 g of sodium fluoride, previously dried at 200° for 4 h and accurately weighed, into a 400-mL plastic beaker, add 200 mL of water, and stir until dissolved. Quantitatively transfer this solution into a 1000-mL volumetric flask with the aid of water, dilute with water to volume, and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 5.0 mL of the stock solution into a 1000-mL volumetric flask, dilute with water to volume, and mix.

Calibration curve: Transfer 1.0, 2.0, 3.0, 5.0, 10.0, and 15.0 mL of the *Sodium fluoride solution* into separate 250-mL plastic beakers. Add 50 mL of water, 5 mL of 1 N hydrochloric acid, 10 mL of 1 M sodium citrate, and 10 mL of 0.2 M disodium EDTA to each beaker and mix. Transfer each solution into separate 100-mL volumetric flasks, dilute with water to volume, and mix. Transfer a 50-mL portion of each solution into separate 125-mL plastic beakers, and measure the potential of each solution with a suitable ion-selective electrode apparatus (such as the Orion Model No. 94–09, with solid-state membrane), using a suitable reference electrode (such as the Orion Model No. 90–01, with single junction). Plot the calibration curve on two-cycle semi-logarithmic paper (such as K & E No. 465130) or with the use of a suitable graphing calculator or spreadsheet program, with µg of F per 100 mL solution on the logarithmic scale.

Analysis: Transfer 1.00 g of sample into a 150-mL glass beaker, add 10 mL of water, and, while stirring continuously, slowly add 20 mL of 1 N hydrochloric acid to dissolve the sample. Boil rapidly for 1 min, then transfer into a 250-mL plastic beaker, and cool rapidly in ice water. Add 15 mL of 1 M sodium citrate and 10 mL of 0.2 M disodium EDTA, and mix. Adjust the pH to 5.5 ± 0.1 with 1 N hydrochloric acid or 1 N sodium hydroxide, if necessary. Transfer into a 100-mL volumetric flask, dilute with water to volume, and mix. Transfer a 50-mL portion of this solution into a 125-mL plastic beaker, and measure the potential of the solution with the apparatus described under *Calibration curve*. Determine the fluoride content, in µg, of the sample from the *Calibration curve*. Determine the percentage of fluoride in the sample taken:

$$\text{Result} = (C/WS) \times F \times 100$$

C = content of fluoride (µg)
WS = sample weight (g)
F = factor converting µg to g, 0.000001

Lead
Sample solution: 1 g in 20 mL
Lead nitrate stock solution and **Standard lead solution:** Proceed as directed under *Bentonite*.
Analysis: Proceed as directed under *Bentonite*.
Magnesium and alkali salts
Sample: 1 g
Analysis: Dissolve the *Sample* in 50 mL of water, add 500 mg of ammonium chloride, mix, and boil for 1 min. Rapidly add 40 mL of oxalic acid TS and stir vigorously until precipitation is well established. Immediately add 2 drops of methyl red TS. Then add 6 N ammonium hydroxide, dropwise, until the mixture is just alkaline, and cool. Transfer the mixture to a 100-mL cylinder, dilute with water to 100 mL, and let it stand for 4 h or overnight. Decant the clear, supernatant liquid through a dry filter paper, and transfer 50 mL of the clear filtrate to a platinum dish. Add 0.5 mL of sulfuric acid to the dish and evaporate the mixture on a steam bath to a small volume. Carefully evaporate the remaining liquid to dryness over a free flame and continue heating until the ammonium salts have been completely decomposed and volatilized. Finally, ignite the residue to constant weight.
Moisture adsorption capacity: Proceed as directed under *Bentonite*.

• **CALCIUM OXIDE**
Identification—calcium: Passes tests
Assay: NLT 95.0% and NMT 100.5% of calcium oxide (CaO), on the ignited basis
Inorganic impurities
Acid-insoluble substances: NMT 1%
Arsenic: NMT 3 ppm
Fluoride: NMT 0.015%
Lead: NMT 2 mg/kg
Magnesium and alkali salts: NMT 3.6%
Specific tests
Loss on ignition: NMT 10.0%
Moisture adsorption capacity: NLT 28% at 80% ± 5% RH and 25 ± 2°
Test methods
Identification—calcium
Sample solution: Shake 1 g of sample with 20 mL of water and add glacial acetic acid until the sample is dissolved.
Analysis: Proceed as directed under *Calcium Chloride, Anhydrous*.
Assay
Sample: 1 g of sample ignited to a constant weight (see *Loss on ignition* below)
Analysis: Dissolve the *Sample* in 20 mL of 2.7 N hydrochloric acid. Cool the solution, dilute with water to 500.0 mL, and mix. Pipet 50.0 mL of this solution into a suitable container, and add 50 mL of water. While stirring, preferably with a magnetic stirrer, add about 30 mL of 0.05 M disodium EDTA from a 50-mL buret. Then, add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue indicator. Continue the titration with disodium EDTA to a blue endpoint. Each mL of 0.05 M disodium EDTA is equivalent to 2.804 mg of calcium oxide (CaO).
Arsenic
Sample solution: 1 g in 15 mL of 2.7 N hydrochloric acid
Arsenic trioxide stock solution and **Standard arsenic solution:** Proceed as directed under *Bentonite*.
Analysis: Proceed as directed under *Bentonite*.
Fluoride
Sample: 1.0 g
Analysis: Proceed as directed under *Calcium Chloride, Anhydrous*.
Lead
Sample solution: 1 g in 15 mL of 2.7 N hydrochloric acid
Lead nitrate stock solution and **Standard lead solution:** Proceed as directed under *Bentonite*.
Analysis: Proceed as directed under *Bentonite*.
Acid-insoluble substances
Sample solution: Shake 5 g of sample, and then mix it with 100 mL of water and sufficient hydrochloric acid, added dropwise, to dissolve it.
Analysis: Boil the *Sample solution*, cool, add hydrochloric acid, if necessary, to make the solution distinctly acid, and pass through a tared glass filter crucible. Wash the residue with water until free of chlorides, dry at 105° for 1 h, cool, and weigh.
Magnesium and alkali salts
Sample: 500 mg
Analysis: Dissolve the *Sample* in 30 mL of water and 15 mL of 2.7 N hydrochloric acid. Heat the solution, boil for 1 min, and rapidly add 40 mL of oxalic acid TS, and stir vigorously. Add 2 drops of methyl red TS, and neutralize the solution with 6 N ammonium hydroxide to precipitate the calcium completely. Heat the mixture on a steam bath for 1 h and allow it to cool. Dilute the mixture with water to 100 mL, mix well, and filter. Add 0.5 mL of sulfuric acid to 50 mL of the filtrate. Then evaporate to dryness and ignite to constant weight in a tared platinum crucible at 800 ± 25°.
Loss on ignition
Sample: 1 g
Analysis: Ignite the *Sample* to constant weight in a tared platinum crucible at 1100 ± 50°.

Moisture adsorption capacity: Proceed as directed under *Bentonite*.

- **MOLECULAR SIEVES**

Molecular sieves are synthetic porous crystalline alkali-metal aluminosilicates in a beaded form with a tightly controlled pore size. The most commonly used molecular sieves for desiccants are described as types 3A, 4A, 5A, and 13X with a pore size range of approximately 3–10 Å.

Identification A: The drop of water becomes turbid.

Identification B: Passes tests

Inorganic impurities

Lead: NMT 5 ppm

Specific tests

pH ⟨791⟩: 6.5–12 (200 mg/mL in carbon dioxide-free water)

Loss on Drying ⟨731⟩: Dry a 5–10 g sample at 575 ± 25° to a constant weight: it loses NMT 4.5%.

[NOTE—Conduct assay immediately after opening the original container.]

Moisture adsorption capacity

NLT 15.0% weight at 40% ± 5% RH and 25 ± 2°

NLT 16.5% weight at 80% ± 5% RH and 25 ± 2°

Test methods

Identification A

Sample: 500 mg

Analysis: Mix the *Sample* with 2.5 g of anhydrous potassium carbonate, and heat the mixture in a platinum or nickel crucible until it melts completely. Cool, add 5 mL of water, and allow to stand for 3 min. Heat the bottom of the crucible gently, detach the melt, and transfer it into a beaker with the aid of about 50 mL of water. Gradually add hydrochloric acid until no effervescence is observed, add 10 mL more of the acid, and evaporate to dryness on a steam bath. Cool, add 20 mL of water, boil, and pass through ash-free filter paper. An insoluble residue of silica remains. [NOTE—Retain the filtrate for *Identification B*.] Transfer the gelatinous residue to a platinum dish, and cautiously add 5 mL of hydrofluoric acid. [**CAUTION—**Handle hydrofluoric acid in a fume hood with appropriate precautions.] The precipitate dissolves. (If it does not dissolve, repeat the treatment with hydrofluoric acid.) Heat the solution and introduce a glass stirring rod with a drop of water on the tip into the resulting vapors.

Identification B—aluminum

Sample: Use 2 portions of the filtrate obtained in *Identification A*.

Analysis: Solution of a first portion of the filtrate obtained in *Identification A* yields a white, gelatinous precipitate with 6 N ammonia that is insoluble in an excess of this reagent. Solution of a second portion of the filtrate obtained in *Identification A* yields a white precipitate with a 1 N sodium hydroxide that is dissolved in an excess of this reagent.

Lead

Sample solution: Transfer 10.0 g of sample into a 250-mL beaker, add 50 mL of 0.5 N hydrochloric acid, cover with a watch glass, and heat slowly to boiling. Boil gently for 15 min, cool, and let the undissolved material settle. Decant the supernatant liquid through Whatman No. 4, or equivalent, filter paper into a 100-mL volumetric flask, retaining as much as possible of the insoluble material in the beaker. Wash the slurry and beaker with three 10-mL portions of hot water, decanting each washing through the filter into the flask. Finally, wash the filter paper with 15 mL of hot water, cool the filtrate to room temperature, dilute with water to volume, and mix.

Lead nitrate stock solution and **Standard lead solution:** Proceed as directed under *Bentonite*.

Analysis: Proceed as directed under *Bentonite*.

Moisture adsorption capacity: Proceed as directed under *Bentonite*.

- **SILICA GEL**

Silica gel is silicon dioxide ($SiO_2 \cdot H_2O$) that has been manufactured by the addition of sodium silicate solution to a mineral acid to produce a gelatinous precipitate that is washed, then dehydrated to produce colorless silica gel in a bead, granular, or micronized form in a range of mesh sizes.

Appearance: A white or translucent bead or granule

Identification A: A deep yellow color is produced.

Identification B: A green-blue spot develops.

Assay: NLT 94.0% of silicon dioxide (SiO_2) on the ignited basis

Inorganic impurities

Lead: NMT 5 ppm

Soluble ionizable salts (as Na_2SO_4): The conductance produced by the sample is NMT that produced by the control solution (equivalent to NMT 5.0%).

Specific tests

pH ⟨791⟩: 4–8 in a slurry (1 in 20)

Loss on Drying ⟨731⟩: Dry a 5–10-g sample at 145° for 3 h: it loses NMT 3.0% of its weight.

[NOTE—Conduct assay immediately after opening the original container.]

Moisture adsorption capacity

NLT 19% wt at 40% ± 5% RH and 25 ± 2°

NLT 27% wt at 80% ± 5% RH and 25 ± 2°

Test methods

Identification A

Sample: 5 mg

Analysis: Place into a platinum crucible, mix with 200 mg of anhydrous potassium carbonate, and ignite over a burner at a red heat for about 10 min. Cool, dissolve the melt in 2 mL of freshly distilled water, warming if necessary, and slowly add 2 mL of ammonium molybdate TS.

Identification B—aluminum

Sample: Solution remaining from *Identification A*

Analysis: Place 1 drop of the *Sample* from *Identification A* on a filter paper, and evaporate the solvent. Add 1 drop of a saturated solution of *o*-tolidine in glacial acetic acid, and place the paper over ammonium hydroxide.

[NOTE—Avoid contact with *o*-tolidine when performing this test, and conduct the test in a well-ventilated hood.]

Assay

Sample: 1 g, previously dried

Analysis: Transfer the *Sample* into a tared platinum crucible, ignite at 1000 ± 25° to constant weight, cool in a desiccator, and weigh to obtain the ignited sample weight (*W1*). Moisten the residue with a few drops of alcohol, add 3 drops of sulfuric acid, then add enough hydrofluoric acid to cover the wetted sample.

[**CAUTION**—Handle hydrofluoric acid in a well-ventilated fume hood with appropriate precautions.]

Evaporate to dryness on a hot plate, using medium heat (95°–105°), then add a few mL of hydrofluoric acid enough to cover the *Sample*, swirl the dish carefully to wash down the sides, and again evaporate to dryness taking care that the *Sample* does not spatter as dryness is approached. Heat the crucible to a red heat using a Meker burner, a propane torch, or in a muffle furnace. Ignite the residue at 1000 ± 25° for 30 min, cool in a desiccator, and weigh to obtain the final weight (*W2*). If a residue remains, repeat the *Analysis* beginning with the addition of hydrofluoric acid until a constant weight is obtained. The difference between the ignited sample weight and the final weight (*W1* – *W2*) represents the weight, in g, of silicon dioxide (SiO_2) in the initially ignited sample. Express the result as a percentage of the initially ignited basis.

Lead

Sample solution: Transfer 5.0 g of sample into a 250-mL beaker, add 50 mL of 0.5 N hydrochloric acid, cover with a watch glass, and slowly heat to boiling. Boil gently for 15 min, cool, and let the undissolved material settle. Decant the supernatant liquid through a Whatman No. 3, or equivalent, filter paper into a 100-mL volumetric flask, retaining as much as possible of the insoluble material in the beaker. Wash the slurry and beaker with three 10-mL portions of hot water, decanting each washing through the filter into the flask. Finally, wash the filter paper with 15 mL of hot water, cool the filtrate to room temperature, dilute with water to volume, and mix.

Lead nitrate stock solution and **Standard lead solution:** Proceed as directed under *Bentonite*.

Analysis: Proceed as directed under *Bentonite*.

Soluble ionizable salts (as Na_2SO_4)

Sample: 5 g, previously dried

Control solution: 1 mg/mL of anhydrous sodium sulfate, made to 250 mL

Analysis: Stir the *Sample* with 150 mL of water for at least 5 min in a high-speed mixer. Filter with the aid of suction, and wash the mixer and filter with 100 mL of water in divided portions, adding the washings to the filtrate. Dilute the filtrate with water to 250 mL. Determine the conductances of the diluted filtrate and of the *Control solution* with a suitable conductance bridge assembly.

Moisture adsorption capacity: Proceed as directed under *Bentonite*.

Reagents—test solutions

Ammonium molybdate TS: Dissolve 6.5 g of finely powdered molybdic acid in a mixture of 14 mL of water and 14.5 mL of ammonium hydroxide. Cool the solution, and add it slowly, with stirring, to a well-cooled mixture of 32 mL of nitric acid and 40 mL of water. Allow to stand for 48 h, and filter through a fine-porosity, sintered-glass crucible. This solution deteriorates upon standing and is unsuitable for use if, upon the addition of 2 mL of dibasic sodium phosphate TS to 5 mL of the solution, an abundant yellow precipitate does not form at once or after slight warming. Store it in the dark. If a precipitate forms during storage, use only the clear supernatant.

Oxalic acid TS: Dissolve 6.3 g of oxalic acid in water to make 100 mL.