U.S. Pharmacopeial Convention

Pregelatinized Starch

DEFINITION

Pregelatinized Starch is Starch that has been chemically and/or mechanically processed to rupture all or part of the granules in the presence of water and subsequently dried. Some types of Pregelatinized Starch may be modified to render them compressible and flowable in character.

IDENTIFICATION

- A water slurry of it is colored orange-red to deep blue by iodine TS.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 〈281〉: NMT 0.5%, determined on a 2.0-g test specimen
- Iron 〈241〉: NMT 20 ppm
  Analysis: Dissolve the residue obtained in the test for Residue on Ignition in 8 mL of hydrochloric acid with the aid of gentle heating, and dilute with water to 100 mL. Dilute 25 mL of this solution with water to 47 mL.
- Limit of Sulfur Dioxide
  Sample solution: Mix 20 g with 200 mL of a 1 in 5 solution of anhydrous sodium sulfate, and filter.
  Analysis: To 100 mL of the clear filtrate add 3 mL of starch TS, and titrate with 0.01 N iodine VS to the first permanent blue color.
  Acceptance criteria: NMT 2.7 mL is consumed (80 ppm).

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: It meets the requirements of the tests for absence of Salmonella species and Escherichia coli. The total aerobic microbial count does not exceed 1000 cfu/g; and the total combined molds and yeasts count does not exceed 100 cfu/g.
- pH 〈791〉: 4.5–7.0
  Prepare a slurry by weighing 10.0 ± 0.1 g in 10 mL of alcohol and by diluting with water to 100 mL. Agitate continuously at a moderate rate for 5 min, then cease agitation and immediately potentiometrically determine the pH to the nearest 0.1 unit.

- Loss on Drying 〈731〉: Dry a sample at 120° for 4 h: it loses NMT 14.0% of its weight.
- Oxidizing Substances
  Sample: 5 g
  Analysis: To the Sample add 20 mL of a mixture of equal volumes of methanol and water, then add 1 mL of 6 N acetic acid, and stir until a homogeneous suspension is obtained. Add 0.5 mL of a freshly prepared, saturated solution of potassium iodide, and allow to stand for 5 min.
  Acceptance criteria: No distinct blue, brown, or purple color is observed.

ADDITIONAL REQUIREMENTS

- Packaging and Storage: Preserve in well-closed containers. No storage requirements specified.
- Labeling: Label it to indicate the botanical source from which it was derived.

Corn Starch

Attributes	EP	JP	USP
Definition	+	+	+
Identification A	+	+	+
Identification B	+	+	+
Identification C	+	+	+
pH	+	+	+
Loss on Drying	+	+	+
Residue on Ignition	+	+	+
Limit of Iron	+	+	+
Limit of Oxidizing Substances	+	+	+
Limit of Sulfur Dioxide	+	+	+

Legend: + will adopt and implement; − will not stipulate

Nonharmonized attributes: Characters, Microbial Enumeration Tests and Tests for Specified Microorganisms, Labeling, and Packaging and Storage (USP)

Specific local attributes: Foreign matter (EP)

Reagents and reference materials: Each pharmacopeia will adapt the text to take account of local reference materials and reagent specifications.

DEFINITION

Corn Starch consists of the starch granules separated from the mature grain of corn [Zea mays Linnè (Fam. Gramineae)].

IDENTIFICATION

A. Procedure: Examine under a microscope, using NLT 20× magnification and using a mixture of glycerin and water (1:1) as a mounting agent.

Acceptance criteria: It appears either as angular polyhedral granules of irregular sizes with diameters ranging from 2–23 µm, or as rounded or spheroidal granules of irregular sizes with diameters ranging from 25–35 µm. The central hilum consists of a distinct cavity or two- to five-rayed cleft, and there are no concentric striations. Between orthogonally oriented polarizing plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.

B. Procedure

Sample solution: 20 mg/mL in water

Analysis: Boil for 1 min, and cool.

Acceptance criteria: A thin, cloudy mucilage is formed.

C. Procedure

Sample solution: 1 mL of the mucilage obtained in Identification test B

Analysis: Add 0.05 mL of iodine and potassium iodide TS 2 to the Sample solution.

Acceptance criteria: An orange-red to dark blue color is produced, which disappears upon heating.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 〈281〉: NMT 0.6%, determined on a 1.0-g sample
- Limit of Iron

  Standard iron stock solution A: Equivalent to 10 µg/mL of iron prepared as directed under Iron 〈241〉

  Standard iron stock solution B: 1 µg/mL of iron from Standard iron stock solution A in water

  [Note—Prepare immediately before use.]

  Standard iron solution: Transfer 10 mL of Standard iron stock solution B to a test tube, and add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

  Sample solution: Shake 1.5 g of Corn Starch with 15 mL of 2 N hydrochloric acid, and filter. Transfer 10 mL of the filtrate to a test tube, add 2 mL of citric acid solution (2 in 10), and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

  Acceptance criteria: After 5 min, any pink color in the Sample solution is not more intense than that in the Standard iron solution, corresponding to a limit of 10 ppm of iron.
- Limit of Sulfur Dioxide

  Carbon dioxide: Use carbon dioxide, with a flow regulator that will maintain a flow of 100 ± 10 mL/min.

  Bromophenol blue indicator solution: 0.2 mg/mL of bromophenol blue in dilute alcohol. Filter if necessary.

  Hydrogen peroxide solution: Dilute 30% hydrogen peroxide with water to obtain a 3% solution. Just before use, add 3 drops of Bromophenol blue indicator solution, and

neutralize to a violet-blue endpoint with 0.01 N sodium hydroxide. Do not exceed the endpoint.

Apparatus: Figure 1.

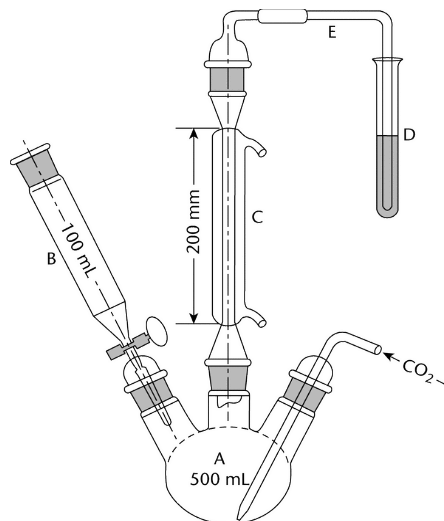

Figure 1

In this test, the sulfur dioxide is released from the sample in a boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to sulfuric acid and titrated with standard alkali. The apparatus consists essentially of a 500-mL three-neck, round-bottom boiling flask, A; separatory funnel, B, having a capacity of 100 mL or greater; a gas inlet tube of sufficient length to permit introduction of the carbon dioxide within 2.5 cm of the bottom of the boiling flask; a reflux condenser, C, having a jacket length of 200 mm, and a delivery tube, E, connecting the upper end of the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of stopcock grease to the sealing surfaces of all of the joints except the joint between the separatory funnel and the boiling flask, and clamp the joints to ensure tightness.

Sample: 25.0 g of Corn Starch

Analysis: Add 150 mL of water to the boiling flask. Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide at a rate of 100 ± 5 mL/min through the Apparatus. Start the condenser coolant flow. Add 10 mL of Hydrogen peroxide solution to a receiving test tube. After 15 min, without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask, and transfer the Sample into the boiling flask with the aid of 100 mL of water. Apply stopcock grease to the outer joint of the separatory funnel, and replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80 mL of 2 N hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 h. Remove the receiving test tube, and transfer its contents to a 200-mL wide-necked, conical flask. Rinse the receiving test tube with a small portion of water, add the rinsing to the 200-mL conical flask, and mix. Heat on a water bath for 15 min, and allow to cool.

Add 0.1 mL of Bromophenol blue indicator solution, and titrate the contents with 0.1 N sodium hydroxide VS until the color changes from yellow to violet-blue. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉).

Calculate the content, in ppm, of sulfur dioxide in the Sample taken:

Result = 1000 (32.03) VN/W

32.03	=	milliequivalent weight of sulfur dioxide
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
W	=	weight of the Sample (g)

Acceptance criteria: NMT 50 ppm

- Limit of Oxidizing Substances
  Sample solution: Transfer 4.0 g to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5–1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS.
  Analysis: Titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.
  Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate is required (20 ppm, calculated as H2O2).

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: The total aerobic microbial count does not exceed 1000 cfu/g; the total combined molds and yeasts count does not exceed 100 cfu/g; and it meets the requirements of the test for the absence of Escherichia coli. Where it is intended for use in preparing Absorbable Dusting Powder, it also meets the requirements of the tests for absence of Staphylococcus aureus and Pseudomonas aeruginosa.
- Loss on Drying 〈731〉: Dry 1 g at 130° for 90 min: it loses NMT 15.0% of its weight.
- pH 〈791〉: 4.0–7.0
  Sample solution: Prepare a slurry by weighing 5.0 g of Corn Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.
  Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, and allow to stand for 15 min. Determine the pH to the nearest 0.1 unit.

ADDITIONAL REQUIREMENTS

- Packaging and Storage: Preserve in well-closed containers. No storage requirements specified.

- Labeling: Where Corn Starch is intended for use in preparing Absorbable Dusting Powder, it is so labeled, and the label states that it must be subjected to further processing during the preparation of Absorbable Dusting Powder.

Pea Starch

DEFINITION

Pea Starch is obtained from the seeds of Pisum sativum L.

IDENTIFICATION

A. Examined under a microscope using a mixture of equal volumes of glycerin and water, it presents a majority of large elliptical granules, 25-45 µm in size, sometimes irregular, or reniform. It also presents a minority of small rounded granules, 5-8 µm in size. Granules can present cracks or irregularities. Sometimes, granules show barely visible concentric striations. Exceptionally, granules show a slit along the main axis. Between orthogonally oriented polarizing plates or prisms, the granules show a distinct black cross.

B. B. Suspend 1 g of it in 50 mL of water, boil for 1 min, and cool: a thin, cloudy mucilage is formed.

C. C. To 1 mL of the mucilage obtained in Identification test B add 0.05 mL of iodine and potassium iodide TS 2: an orange-red to dark blue color is produced, which disappears on heating.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 《281》: NMT 0.6%, determined on a 1.0-g sample
- Limit of Iron

  Standard iron stock solution: Prepare a solution containing the equivalent of 10 µg/mL of iron, as directed under Iron 《241》.

  Diluted standard iron solution: Immediately before use, dilute a measured volume of Standard iron stock solution quantitatively with water to obtain a solution containing the equivalent of 1 µg/mL of iron.

  Standard solution: Transfer 10 mL of the Diluted standard iron solution to a test tube. Add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid, and mix. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, dilute with water to 20 mL, and mix.

  Sample solution: Shake 1.0 g of Pea Starch with 50 mL of 2 N hydrochloric acid, and filter. Transfer 10 mL of the filtrate to a test tube. Add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid, and mix. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, dilute with water to 20 mL, and mix.

Acceptance criteria: After 5 min, any pink color in the Sample solution is not more intense than that in the Standard solution, corresponding to a limit of 50 µg/g of iron.

- Limit of Sulfur Dioxide

  [Note—Perform either Test 1 or Test 2.]

Test 1

Carbon dioxide: Use carbon dioxide with a flow regulator that will maintain a flow of 100 ± 10 mL/min.

Hydrogen peroxide solution: Dilute 30% hydrogen peroxide with water to obtain a 3% solution. Neutralize the solution with 0.01 N sodium hydroxide to a pH of 4.1, determined potentiometrically.

Potassium metabisulfite solution: Transfer 0.87 g of potassium metabisulfite (K2S2O5) and 0.2 g of edetate disodium to a 1000-mL volumetric flask. Dilute with water to volume before mixing. [Note—Edetate disodium is used to protect the sulfite ion from oxidation.]

Apparatus: In this test, the sulfur dioxide is released from the sample in a boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to sulfuric acid and titrated with standard alkali. A suitable apparatus for sulfur dioxide determination is shown in the accompanying diagram (Figure 1).

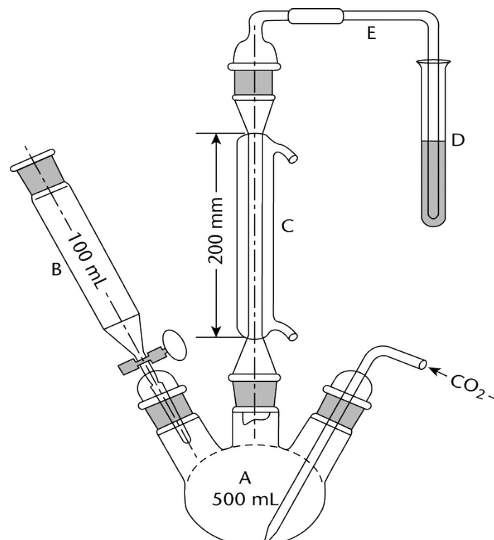

Figure 1

The apparatus consists of a 500-mL three-neck, round-bottom boiling flask, A; a separatory funnel, B, having a capacity of 100 mL or greater; a gas inlet tube of sufficient length to permit introduction of the carbon dioxide within 2.5 cm of the bottom of the boiling flask; a reflux condenser, C, having a jacket length of 200 mm; and a delivery tube, E, connecting the upper end of the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of stopcock grease to the sealing surfaces of all the joints except the joint between the separatory funnel and the boiling flask, and clamp the joints to ensure tightness.

System suitability test

Test A: Using the Potassium metabisulfite solution as the standard, proceed as directed in Analysis, except for replacing the 25.0 g of Pea Starch with 20 mL of the Potassium metabisulfite solution.

Calculate the content, in µg/mL, of sulfur dioxide in the Potassium metabisulfite solution taken:
Result = $(F \times MW \times V \times N)/VP$

F	=	factor for conversion of mg to µg, 1000
MW	=	milliequivalent weight of sulfur dioxide, 32.03
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
VP	=	volume of the Potassium metabisulfite solution taken for the test (mL)

Test B: In a 100-mL conical flask, add 20 mL of 0.02 N iodine solution and 5 mL of 2 N hydrochloric acid. Add 1 mL of starch TS, and titrate with Potassium metabisulfite solution until the first discoloration is observed.
Calculate the content, in µg/mL, of sulfur dioxide in Potassium metabisulfite solution:
Result = $(F \times MW \times VI \times NI)/VP$

F	=	factor for conversion of mg to µg, 1000
MW	=	milliequivalent weight of sulfur dioxide, 32.03
VI	=	the volume of the iodine solution used in the test (mL)
NI	=	normality of the iodine solution
VP	=	volume of the Potassium metabisulfite solution consumed (mL)

The difference between the sulfur dioxide contents obtained from Test A and Test B is NMT 5% of their mean value. Test B shall be performed within 15 min after the completion of Test A. [Note—This time limit avoids potential variation in the sulfur dioxide content of the Potassium bisulfite solution when stored at room temperature.]

Analysis: Add 150 mL of water to the boiling flask (A) (see Figure 1). Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide through the apparatus at a rate of 100 ± 5 mL/min. Start the condenser coolant flow. Place 10 mL of Hydrogen peroxide solution in the receiving test tube (D). After 15 min, without interrupting the flow of carbon dioxide, remove the separatory funnel (B) from the boiling flask, and transfer 25.0 g of Pea Starch to the boiling flask with the aid of 100 mL of water. Apply stopcock grease to the outer joint of the separatory funnel, and replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80 mL of 2 N hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 h. Open the stopcock of the funnel, stop the flow of carbon dioxide, discontinue heating the flask, and turn off the cooling water in the condenser. Remove the receiving test tube, and transfer its contents to a 200-mL wide-necked conical flask. Rinse the receiving test tube with a small portion of water, add the rinsing to the 200-mL conical flask, and mix. Heat on a water bath for 15 min, and allow to cool. Titrate the contents with 0.1 N sodium hydroxide VS until the pH

reaches 4.1, determined potentiometrically. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉).

Calculate the content, in µg/g, of sulfur dioxide in the Pea Starch taken:

Result = (F × MW × V × N)/W

F	=	factor for conversion of mg to µg, 1000
MW	=	milliequivalent weight of sulfur dioxide, 32.03
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
W	=	weight of the Pea Starch taken (g)

Test 2

Determine the content of sulfur dioxide as directed under Sulfur Dioxide 〈525〉, Method I. Use 200 mL of water as a solvent. Then, to 100 mL of the clear filtrate, add 3 mL of starch TS, and titrate with 0.01 N iodine VS to the first permanent blue color.

Acceptance criteria: NMT 50 µg/g of sulfur dioxide

Organic Impurities

- Procedure 1: Limit of Oxidizing Substances
  Sample: 4.0 g of Pea Starch
  Analysis: Transfer the Sample to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5–1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS, and titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch-iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate VS is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.
  Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate VS is required (20 µg/g, calculated as H2O2).
- Procedure 2: Foreign Matter
  Analysis: Examine under a microscope, using a mixture of equal volumes of glycerin and water.
  Acceptance criteria: NMT traces of matter other than starch granules are present. No starch grains of any other origin are present.

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: The total aerobic microbial count does not exceed 1000 cfu/g, the total combined molds and yeasts count does not exceed 100 cfu/g, and it meets the requirements of the test for the absence of Escherichia coli.
- pH 〈791〉

Sample solution: Prepare a slurry by weighing 5.0 g of Pea Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.
Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, allow to stand for 15 min, and shake again. Determine the pH to the nearest 0.1 unit: the pH is determined potentiometrically.
Acceptance criteria: 5.0–8.0

- Loss on Drying 〈731〉: Dry about 1 g at 130° for 90 min: it loses NMT 16.0% of its weight.

ADDITIONAL REQUIREMENTS

- Packaging and Storage: Preserve in well-closed containers. Store at room temperature.

Potato Starch

Attributes	EP	JP	USP
Definition	+	+	+
Identification A	+	+	+
Identification B	+	+	+
Identification C	+	+	+
pH	+	+	+
Loss on Drying	+	+	+
Residue on Ignition	+	+	+
Limit of Iron	+	+	+
Limit of Oxidizing Substances	+	+	+
Limit of Sulfur Dioxide	+	+	+

Legend: + will adopt and implement; − will not stipulate

Nonharmonized attributes: Characters, Microbial Enumeration Tests and Tests for Specified Microorganisms, Labeling, and Packaging and Storage (USP)

Specific local attributes: Foreign matter (EP)

Reagents and reference materials: Each pharmacopeia will adapt the text to take account of local reference materials and reagent specifications.

DEFINITION

Potato Starch is obtained from the tuber of Solanum tuberosum L.

IDENTIFICATION

A. Procedure: Examine under a microscope using a mixture of equal volumes of glycerol and water.
   Acceptance criteria: It presents granules, either irregularly shaped, ovoid or pear-shaped, usually 30–100 µm in size but occasionally exceeding 100 µm, or rounded, 10–35 µm in size. There are occasional compound granules having two to four components. The ovoid and pear-shaped granules have an eccentric hilum and the rounded granules acentric or slightly eccentric hilum. All granules show clearly visible concentric striations. Between orthogonally oriented polarizing plates or prisms, the granules show a distinct black cross intersecting at the hilum.
B. Procedure
   Sample solution: 20 mg/mL in water
   Analysis: Boil for 1 min, and cool.
   Acceptance criteria: A thin, cloudy mucilage is formed.
C. Procedure
   Sample solution: 1 mL of the mucilage obtained in Identification test B
   Analysis: Add 0.05 mL of iodine and potassium iodide TS 2 to the Sample solution.
   Acceptance criteria: An orange-red to dark blue color is produced, which disappears upon heating.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 〈281〉: NMT 0.6%, determined on a 1.0-g sample
- Limit of Iron
  Standard iron stock solution A: Equivalent of 10 µg/mL of iron prepared as directed under Iron 〈241〉
  Standard iron stock solution B: 1 µg/mL of iron from Standard iron stock solution A in water
  [Note—Prepare immediately before use.]
  Standard iron solution: Transfer 10 mL of Standard iron stock solution B to a test tube, and add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

Sample solution: Shake 1.5 g of Potato Starch with 15 mL of 2 N hydrochloric acid, and filter. Transfer 10 mL of the filtrate to a test tube, add 2 mL of citric acid solution (2 in 10), and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

Acceptance criteria: After 5 min, any pink color in the Sample solution is not more intense than that in the Standard iron solution, corresponding to a limit of 10 ppm of iron.

- Limit of Sulfur Dioxide

  Carbon dioxide: Use carbon dioxide, with a flow regulator that will maintain a flow of 100 ± 10 mL/min.

  Bromophenol blue indicator solution: 0.2 mg/mL of bromophenol blue in dilute alcohol. Filter if necessary.

  Hydrogen peroxide solution: Dilute 30% hydrogen peroxide with water to obtain a 3% solution. Just before use, add 3 drops of Bromophenol blue indicator solution, and neutralize to a violet-blue endpoint with 0.01 N sodium hydroxide. Do not exceed the endpoint.

Apparatus: Figure 1.

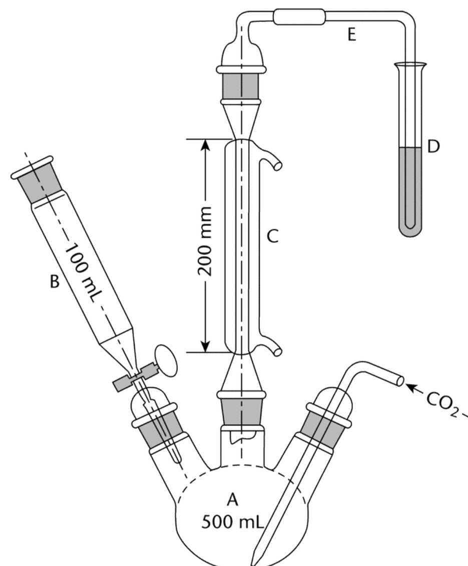

Figure 1

In this test, the sulfur dioxide is released from the sample in a boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to sulfuric acid and titrated with standard alkali. The apparatus consists essentially of a 500-mL three-neck, round-bottom boiling flask, A; a separatory funnel, B, having a capacity of 100 mL or greater; a gas inlet tube of sufficient length to permit introduction of the carbon dioxide within 2.5 cm of the bottom of the boiling flask; a reflux condenser, C, having a jacket length of 200 mm; and a delivery tube, E, connecting the upper end of the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of stopcock

grease to the sealing surfaces of all of the joints except the joint between the separatory funnel and the boiling flask, and clamp the joints to ensure tightness.

Sample: 25.0 g of Potato Starch

Analysis: Add 150 mL of water to the boiling flask. Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide at a rate of 100 ± 5 mL/min through the Apparatus. Start the condenser coolant flow. Add 10 mL of Hydrogen peroxide solution to a receiving test tube. After 15 min, without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask, and transfer the Sample into the boiling flask with the aid of 100 mL of water. Apply stopcock grease to the outer joint of the separatory funnel, and replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80 mL of 2 N hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 h. Remove the receiving test tube, and transfer its contents to a 200-mL wide-necked, conical flask. Rinse the receiving test tube with a small portion of water, add the rinsing to the 200-mL conical flask, and mix. Heat on a water bath for 15 min, and allow to cool.

Add 0.1 mL of Bromophenol blue indicator solution, and titrate the contents with 0.1 N sodium hydroxide VS until the color changes from yellow to violet-blue. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉).

Calculate the content, in ppm, of sulfur dioxide in the Sample taken:

Result = 1000 (32.03) VN/W

32.03	=	milliequivalent weight of sulfur dioxide
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
W	=	weight of the Sample (g)

Acceptance criteria: NMT 50 ppm

- Limit of Oxidizing Substances

  Sample solution: Transfer 4.0 g to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5–1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS.

  Analysis: Titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.

  Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate is required (20 ppm, calculated as H2O2).

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: The total aerobic microbial count does not exceed 1000 cfu/g; the total combined molds and yeasts count does not exceed 100 cfu/g; and it meets the requirements of the test for the absence of Escherichia coli.♦
- Loss on Drying 〈731〉: Dry 1 g at 130° for 90 min: it loses NMT 20.0% of its weight.
- pH 〈791〉: 5.0–8.0
  Sample solution: Prepare a slurry by weighing 5.0 g of Potato Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.
  Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, and allow to stand for 15 min. Determine the pH to the nearest 0.1 unit.

ADDITIONAL REQUIREMENTS

- Packaging and Storage: Preserve in well-closed containers. No storage requirements specified.♦

Rice Starch

Attributes	EP	JP	USP
Definition	+	+	+
Identification A	+	+	+
Identification B	+	+	+
Identification C	+	+	+
pH	+	+	+
Iron	+	+	+
Oxidizing Substances	+	+	+
Sulfur Dioxide	+	+	+
Loss on Drying	+	+	+
Sulfated Ash [Residue on Ignition]	+	+	+

Legend: + will adopt and implement; − will not stipulate.

Nonharmonized attributes: Characters, Microbial Enumeration Tests and Tests for Specified Microorganisms, Packaging and Storage

Specific local attributes: Foreign matter (EP)

Reagents and Reference materials: Each pharmacopeia will adapt the text to take account of local reference materials and reagent specifications.

DEFINITION

Rice Starch is obtained from the caryopsis of Oryza sativa L.

IDENTIFICATION

A. Procedure

Analysis: Examine under a microscope, using NLT 20x magnification and using a mixture of glycerin and water (1:1) as a mounting agent.

Acceptance criteria: It appears either as angular polyhedral granules of irregular sizes with diameters ranging from 2–23 µm, or as rounded or spheroidal granules of irregular sizes with diameters ranging from 25–35 µm. The central hilum consists of a distinct cavity or two- to five-rayed cleft, and there are no concentric striations. Between orthogonally oriented polarizing plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.

B. Procedure

Sample solution: 20 mg/mL in water

Analysis: Boil for 1 min, and cool.

Acceptance criteria: A thin, cloudy mucilage is formed.

C. Procedure

Sample solution: 1 mL of the mucilage obtained in Identification test B

Analysis: Add 0.05 mL of iodine and potassium iodide TS 2 to the Sample solution.

Acceptance criteria: An orange-red to dark blue color is produced, which disappears upon heating.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 〈281〉: NMT 0.6%, determined on a 1.0-g sample
- Limit of Iron

  Standard iron stock solution A: Equivalent to 10 µg/mL of iron prepared as directed under Iron 〈241〉

  Standard iron stock solution B: 1 µg/mL of iron from Standard iron stock solution A in water

  [Note—Prepare immediately before use.]

  Standard iron solution: Transfer 10 mL of Standard iron stock solution B to a test tube, and add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

  Sample solution: Shake 1.5 g of Rice Starch with 15 mL of 2 N hydrochloric acid, and filter. Transfer 10 mL of the filtrate to a test tube, and add 2 mL of citric acid solution (2 in 10) and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.

  Acceptance criteria: After 5 min, any pink color in the Sample solution is not more intense than that in the Standard iron solution, corresponding to a limit of 10 ppm of iron.
- Limit of Sulfur Dioxide

Carbon dioxide: Use carbon dioxide, with a flow regulator that will maintain a flow of 100 ± 10 mL/min.

Bromophenol blue indicator solution: 0.2 mg/mL of bromophenol blue in dilute alcohol. Filter if necessary.

Hydrogen peroxide solution: Dilute 30% hydrogen peroxide with water to obtain a 3% solution. Just before use, add 3 drops of Bromophenol blue indicator solution, and neutralize to a violet-blue endpoint with 0.01 N sodium hydroxide. Do not exceed the endpoint.

Apparatus: Figure 1.

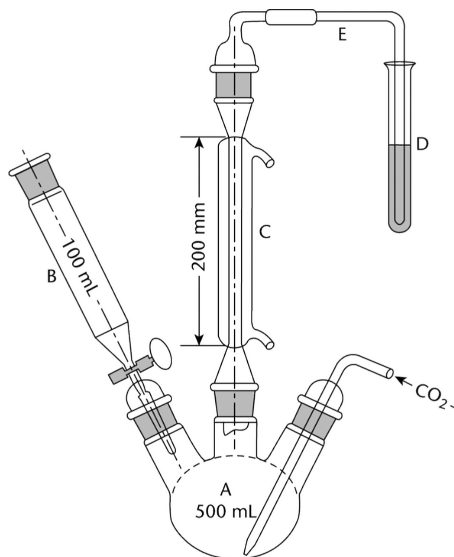

Figure 1

In this test, the sulfur dioxide is released from the sample in a boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to sulfuric acid and titrated with standard alkali. The apparatus consists essentially of a 500-mL three-neck, round-bottom boiling flask, A; a separatory funnel, B, having a capacity of 100 mL or greater; a gas inlet tube of sufficient length to permit introduction of the carbon dioxide within 2.5 cm of the bottom of the boiling flask, a reflux condenser, C, having a jacket length of 200 mm, and a delivery tube, E, connecting the upper end of the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of stopcock grease to the sealing surfaces of all of the joints except the joint between the separatory funnel and the boiling flask, and clamp the joints to ensure tightness.

Sample: 25.0 g of Rice Starch

Analysis: Add 150 mL of water to the boiling flask. Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide at a rate of 100 ± 5 mL/min through the Apparatus. Start the condenser coolant flow. Add 10 mL of Hydrogen peroxide solution to a receiving test tube. After 15 min, without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask, and transfer the Sample into the boiling flask with the aid of 100 mL of water. Apply stopcock grease to the outer joint of the separatory funnel, and replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80 mL of 2 N hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the

separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 h. Remove the receiving test tube, and transfer its contents to a 200-mL wide-necked, conical flask. Rinse the receiving test tube with a small portion of water, add the rinsing to the 200-mL conical flask, and mix. Heat on a water bath for 15 min, and allow to cool.

Add 0.1 mL of Bromophenol blue indicator solution, and titrate the contents with 0.1 N sodium hydroxide VS until the color changes from yellow to violet-blue. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉).

Calculate the content, in ppm, of sulfur dioxide in the Sample taken:

Result = 1000 (32.03) VN/W

32.03	=	milliequivalent weight of sulfur dioxide
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
W	=	weight of the Sample (g)

Acceptance criteria: NMT 50 ppm

- Limit of Oxidizing Substances

  Sample solution: Transfer 4.0 g to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5–1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS.

  Analysis: Titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.

  Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate is required (20 ppm, calculated as H2O2).

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: The total aerobic microbial count does not exceed 1000 cfu/g; the total combined molds and yeasts count does not exceed 100 cfu/g; and it meets the requirements of the test for the absence of Escherichia coli.♦
- Loss on Drying 〈731〉: Dry 1 g at 130° for 90 min: it loses NMT 15.0% of its weight.
- pH 〈791〉: 5.0–8.0

  Sample solution: Prepare a slurry by weighing 5.0 g of Rice Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.

  Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, and allow to stand for 15 min. Determine the pH to the nearest 0.1 unit.

ADDITIONAL REQUIREMENTS

Packaging and Storage: Preserve in well-closed containers. No storage requirements specified.

Tapioca Starch

Tapioca Starch consists of the starch granules separated from the tubers of tapioca (cassava) [Manihot utilissima Pohl (Fam. Euphorbiaceae)].

Packaging and storage—Preserve in well-closed containers. No storage requirements specified.

IDENTIFICATION

A. Examine Tapioca Starch under a microscope, using not less than 20× magnification and using glycerin as the mounting agent: it appears as spherical granules with one truncated side, typically having a 5- to 35-µm diameter and having circular or several-rayed central clefts.

B. Suspend 1 g of Tapioca Starch in 50 mL of water, boil for 1 minute, and cool: a thin, cloudy mucilage is formed.

C. To 1 mL of the mucilage obtained in Identification test B add 0.05 mL of iodine and potassium iodide TS 2: an orange-red to dark blue color is produced, which disappears on heating.

- Microbial enumeration tests 〈61〉 and Tests for specified microorganisms 〈62〉—It meets the requirements of the test for absence of Escherichia coli. The total aerobic microbial count does not exceed 1000 cfu per g, and the total combined molds and yeasts count does not exceed 100 cfu per g.
- pH 〈791〉—Weigh 20.0 ± 0.1 g of Tapioca Starch, transfer to a suitable nonmetallic container, and add 100 mL of water to obtain a slurry. Agitate continuously at a moderate rate for 5 minutes, then stop agitation, and immediately determine the pH: between 4.5 and 7.0.
- Loss on drying 〈731〉—Dry it at 130° for 90 minutes: it loses not more than 16.0% of its weight.
- Residue on ignition 〈281〉: not more than 0.6%, determined on a 1.0-g specimen.
- Iron 〈241〉: 0.002%, the Test Preparation being prepared as follows. Shake 0.75 g of Tapioca Starch with 15 mL of 0.1 N hydrochloric acid, and filter. Use 10 mL of this solution as the Test Preparation.

Limit of oxidizing substances—Transfer 4.0 g of Tapioca Starch to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 minutes. Decant into a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5 to 1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25 to 30 minutes in the dark. Add 1 mL of starch TS, and titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodide color. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉). Each mL of 0.002 N sodium thiosulfate VS is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide. Not more than 1.4 mL of

0.002 N sodium thiosulfate VS is required: not more than 0.002% of oxidizing substances is found.

Limit of sulfur dioxide—Mix 20 g of Tapioca Starch with 200 mL of water until a smooth suspension is obtained, and filter. To 100 mL of the clear filtrate add 3 mL of starch TS, and titrate with 0.01 N iodine solution VS to the first permanent blue color. Not more than 1.7 mL is consumed: not more than 0.005% of sulfur dioxide is found.

Auxiliary Information—Please check for your question in the FAQs before contacting USP.

Wheat Starch

Attributes	EP	JP	USP
Definition	+	+	+
Identification A	+	+	+
Identification B	+	+	+
Identification C	+	+	+
pH	+	+	+
Loss on Drying	+	+	+
Residue on Ignition	+	+	+
Limit of Iron	+	+	+
Limit of Oxidizing Substances	+	+	+
Limit of Sulfur Dioxide	+	+	+
Total Protein	+	+	+

Legend: + will adopt and implement; - will not stipulate.

Nonharmonized attributes: Characters, Microbial Enumeration Tests and Tests for Specified Microorganisms, and Packaging and Storage (USP)

Specific local attributes: Foreign matter (EP)

Reagents and Reference materials: Each pharmacopeia will adapt the text to take account of local reference materials and reagent specifications.

DEFINITION

Wheat starch is obtained from the caryopsis of Triticum aestivum L. (T. vulgare Vill.).

IDENTIFICATION

A. Procedure

Analysis: Examine under a microscope using equal volumes of glycerol and water.

Acceptance criteria: It presents large and small granules, and, very rarely, intermediate sizes. The large granules, usually 10–60 µm in diameter, are discoid or, more rarely,

reniform when seen face-on. The central hilum and striations are invisible or barely visible, and the granules sometimes show cracks on the edges. Seen in profile, the granules are elliptical and fusiform and the hilum appears as a slit along the main axis. The small granules, rounded or polyhedral, are 2–10 µm in diameter. Between orthogonally oriented polarizing plates or prisms, the granules show a distinct black cross intersecting at the hilum.

B. Procedure
Sample solution: 20 mg/mL in water
Analysis: Boil for 1 min, and cool.
Acceptance criteria: A thin, cloudy mucilage is formed.

C. Procedure
Sample solution: 1 mL of the mucilage obtained in Identification test B
Analysis: Add 0.05 mL of iodine and potassium iodide TS 2 to the Sample solution.
Acceptance criteria: An orange-red to dark blue color is produced, which disappears upon heating.

IMPURITIES

Inorganic Impurities

- Residue on Ignition 〈281〉: NMT 0.6%, determined on a 1.0-g sample
- Limit of Iron
  Standard iron stock solution A: Equivalent to 10 µg/mL of iron prepared as directed under Iron 〈241〉
  Standard iron stock solution B: 1 µg/mL of iron from Standard iron stock solution A in water
  [Note—Prepare immediately before use.]
  Standard iron solution: Transfer 10 mL of Standard iron stock solution B to a test tube, and add 2 mL of citric acid solution (2 in 10), and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.
  Sample solution: Shake 1.5 g of Wheat Starch with 15 mL of 2 N hydrochloric acid, and filter. Transfer 10 mL of the filtrate to a test tube, add 2 mL of citric acid solution (2 in 10), and 0.1 mL of thioglycolic acid. Add 10 N ammonium hydroxide until the solution is distinctly alkaline to litmus, and dilute with water to 20 mL.
  Acceptance criteria: After 5 min, any pink color in the Sample solution is not more intense than that in the Standard iron solution, corresponding to a limit of 10 ppm of iron.
- Limit of Sulfur Dioxide
  Carbon dioxide: Use carbon dioxide, with a flow regulator that will maintain a flow of 100 ± 10 mL/min.
  Bromophenol blue indicator solution: 0.2 mg/mL of bromophenol blue in dilute alcohol. Filter if necessary.
  Hydrogen peroxide solution: Dilute 30% hydrogen peroxide with water to obtain a 3% solution. Just before use, add 3 drops of Bromophenol blue indicator solution, and

neutralize to a violet-blue endpoint with 0.01 N sodium hydroxide. Do not exceed the endpoint.

Apparatus: Figure 1.

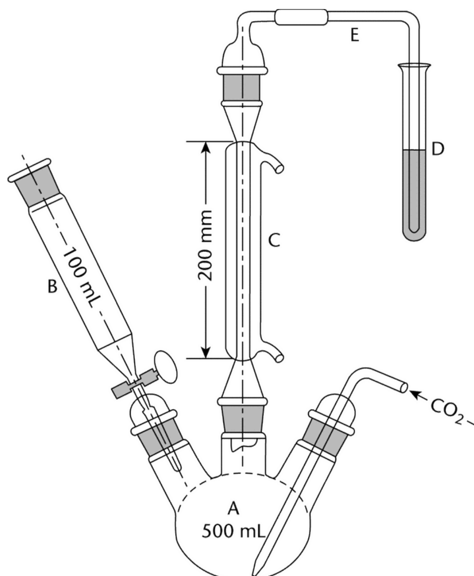

Figure 1

In this test, the sulfur dioxide is released from the sample in a boiling acid medium and is removed by a stream of carbon dioxide. The separated gas is collected in a dilute hydrogen peroxide solution where the sulfur dioxide is oxidized to sulfuric acid and titrated with standard alkali. The apparatus consists essentially of a 500-mL three-neck, round-bottom boiling flask, A; a separatory funnel, B, having a capacity of 100 mL or greater; a gas inlet tube of sufficient length to permit introduction of the carbon dioxide within 2.5 cm of the bottom of the boiling flask; a reflux condenser, C, having a jacket length of 200 mm; and a delivery tube, E, connecting the upper end of the reflux condenser to the bottom of a receiving test tube, D. Apply a thin film of stopcock grease to the sealing surfaces of all of the joints except the joint between the separatory funnel and the boiling flask, and clamp the joints to ensure tightness.

Sample: 25.0 g of Wheat Starch

Analysis: Add 150 mL of water to the boiling flask. Close the stopcock of the separatory funnel, and begin the flow of carbon dioxide at a rate of 100 ± 5 mL/min through the Apparatus. Start the condenser coolant flow. Add 10 mL of Hydrogen peroxide solution to a receiving test tube. After 15 min, without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask, and transfer the Sample into the boiling flask with the aid of 100 mL of water. Apply stopcock grease to the outer joint of the separatory funnel, and replace the separatory funnel in the boiling flask. Close the stopcock of the separatory funnel, and add 80 mL of 2 N hydrochloric acid to the separatory funnel. Open the stopcock of the separatory funnel to permit the hydrochloric acid solution to flow into the boiling flask, guarding against the escape of sulfur dioxide into the separatory funnel by closing the stopcock before the last few mL of hydrochloric acid drain out. Boil the mixture for 1 h. Remove the receiving test tube, and transfer its contents to a 200-mL wide-necked, conical flask. Rinse the receiving test tube with a small portion of water, add the rinsing to the 200-mL conical flask, and mix. Heat on a water bath for 15 min, and allow to cool. Add 0.1 mL of Bromophenol blue indicator solution, and titrate the contents with 0.1 N sodium

hydroxide VS until the color changes from yellow to violet-blue. Perform a blank determination, and make any necessary correction (see Titrimetry 〈541〉).
Calculate the content, in ppm, of sulfur dioxide in the Sample taken:
Result = 1000 (32.03) VN/W

32.03	=	milliequivalent weight of sulfur dioxide
V	=	volume of titrant consumed (mL)
N	=	normality of the titrant
W	=	weight of the Sample (g)

Acceptance criteria: NMT 50 ppm

- Limit of Oxidizing Substances
  Sample solution: Transfer 4.0 g to a glass-stoppered, 125-mL conical flask, and add 50.0 mL of water. Insert the stopper, and swirl for 5 min. Transfer to a glass-stoppered, 50-mL centrifuge tube, and centrifuge to clarify. Transfer 30.0 mL of the clear supernatant to a glass-stoppered, 125-mL conical flask. Add 1 mL of glacial acetic acid and 0.5 g to 1.0 g of potassium iodide. Insert the stopper, swirl, and allow to stand for 25–30 min in the dark. Add 1 mL of starch TS.
  Analysis: Titrate with 0.002 N sodium thiosulfate VS to the disappearance of the starch–iodine color. Perform a blank determination, and make any necessary correction. Each mL of 0.002 N sodium thiosulfate is equivalent to 34 µg of oxidant, calculated as hydrogen peroxide.
  Acceptance criteria: NMT 1.4 mL of 0.002 N sodium thiosulfate is required (20 ppm, calculated as H2O2).

SPECIFIC TESTS

- Microbial Enumeration Tests 〈61〉 and Tests for Specified Microorganisms 〈62〉: The total aerobic microbial count does not exceed 1000 cfu/g; the total combined molds and yeasts count does not exceed 100 cfu/g; and it meets the requirements of the test for the absence of Escherichia coli.
- Loss on Drying 〈731〉: Dry 1 g at 130° for 90 min: it loses NMT 15.0% of its weight.
- pH 〈791〉: 4.5–7.0
  Sample solution: Prepare a slurry by weighing 5.0 g of Wheat Starch, transferring to a suitable nonmetallic container, and adding 25.0 mL of freshly boiled and cooled water.
  Analysis: Agitate continuously at a moderate rate for 1 min. Stop the agitation, and allow to stand for 15 min. Determine the pH to the nearest 0.1 unit.
- Total Protein
  Analysis: Weigh 6.0 g of sample containing 2 mg of nitrogen; transfer to a combustion flask; add 4 g of a powdered mixture consisting of 100 g of potassium sulfate, 5 g of cupric sulfate, and 2.5 g of selenium; and add three glass beads. Wash any adhering particles from the neck into the flask with 5 mL of sulfuric acid, allowing it to run down the sides of the flask, and mix the contents by rotation. Close the mouth of the flask loosely, for example by means of a glass bulb with a short stem, to avoid excessive loss

of sulfuric acid. Heat gradually at first, then increase the temperature until there is vigorous boiling with condensation of sulfuric acid in the neck of the flask; precautions should be taken to prevent the upper part of the flask from becoming overheated. Continue the heating for 30 min, unless otherwise prescribed. Cool, dissolve the solid material by cautiously adding to the mixture 25 mL of water, cool again, and place in a steam distillation apparatus. Add 30 mL of sodium hydroxide solution (42 in 100), and distill immediately by passing steam through the mixture. Collect 40 mL of distillate in 20.0 mL of 0.01 N hydrochloric acid and enough water to cover the tip of the condenser. Toward the end of the distillation, lower the receiver so that the tip of the condenser is above the surface of the acid. Take precautions to prevent any water on the outer surface of the condenser from reaching the contents of the receiver. Titrate the distillate with 0.01 N sodium hydroxide, using methyl purple TS as the indicator (n1 mL of 0.01 N sodium hydroxide).

Repeat the test using 50 mg of glucose in place of the substance to be examined (n2 mL of 0.01 N sodium hydroxide).

Content of nitrogen = [0.01401 (n2 − n1)]/m

m = amount of test substance weighed (g)

Acceptance criteria: NMT 0.3% (corresponding to 0.048% N_2, conversion factor: 6.25)

ADDITIONAL REQUIREMENTS

Packaging and Storage: Preserve in well-closed containers. No storage requirements specified.

Source: http://usp35.infostar.com.cn/