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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.** Suspend 1 g of it in 50 mL of water, boil for 1 min, 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.

IMPURITIES

Inorganic Impurities

• **Residue on Ignition ⟨281⟩:** NMT 0.6%, determined on a 1.0-g sample

Change to read:

• **Limit of Iron**

Standard iron stock solution: Prepare a solution containing the equivalent of 10 µg/mL of iron, as directed under ▲*Iron ⟨241⟩, Procedures, Procedure 1*▲ (CN 1-Jun-2023).

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 ($K_2S_2O_5$) 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*).

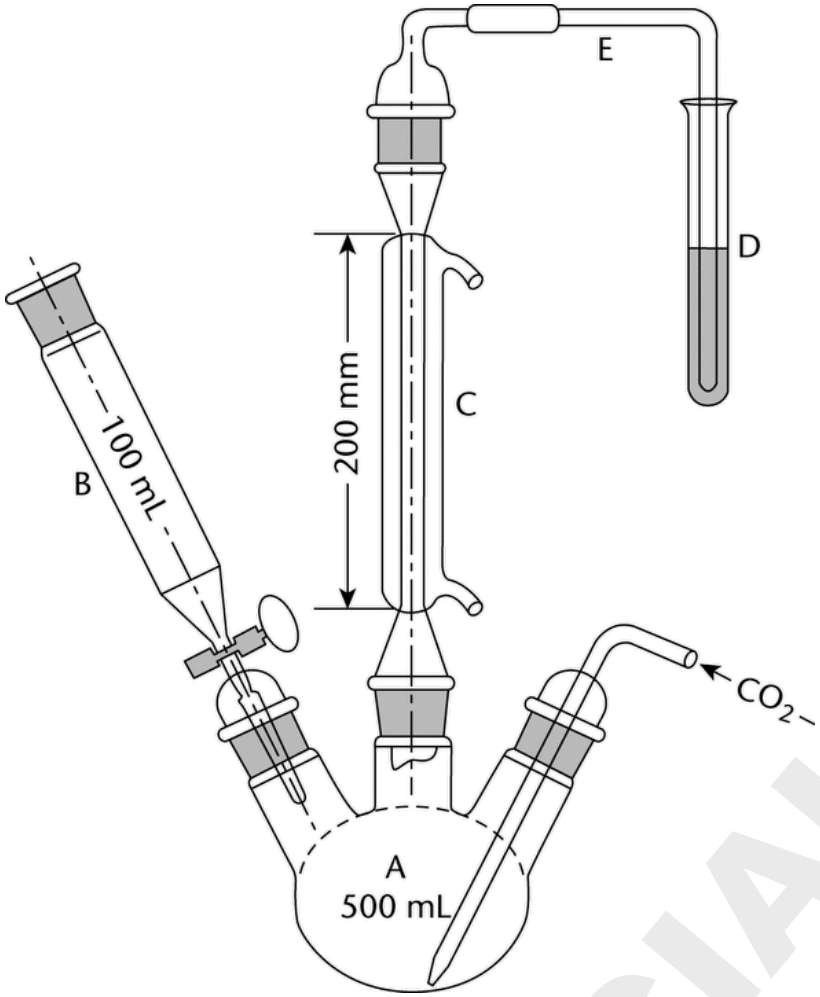

Figure 1

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:

$$\text{Result} = (F \times MW \times V \times N)/V_P$$

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

V_P = 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*:

$$\text{Result} = (F \times MW \times V_I \times N_I)/V_P$$

F = factor for conversion of mg to µg, 1000

MW = milliequivalent weight of sulfur dioxide, 32.03

V_I = the volume of the iodine solution used in the test (mL)

N_I = normality of the iodine solution

V_P = 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:

$$\text{Result} = (F \times MW \times V \times N)/W$$

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 H_2O_2).

- **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.

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

Topic/Question	Contact	Expert Committee
PEA STARCH	Documentary Standards Support	CE2020 Complex Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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