

BRIEFING

Corn Starch, page 715 of *PF* 30(2) [Mar.–Apr. 2004]; **Potato Starch**, page 718 of *PF* 30(2) [Mar.–Apr. 2004]; **Wheat Starch**, page 723 of *PF* 30(2) [Mar.–Apr. 2004]. It is proposed to revise *Identification* test C to reflect the use of a new test solution, *Iodine and potassium iodide TS 2*, which appears in the section *Reagents, Indicators, and Solutions* in this issue of *PF*.

(EMC: J. Lane) RTS—41144-2

Add the following:**▲ Corn Starch**

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

Packaging and storage — Preserve in well-closed containers.

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.

Change to read:**Identification**—

A: Under a microscope, using not less than 20 \times magnification and using a mixture of glycerin and water (1:1) as a mounting agent, it appears as either angular polyhedral granules of irregular sizes with diameters ranging from about 2 μm to about 23 μm or as rounded or spheroidal granules of irregular sizes with diameters ranging from about 25 μm to about 35 μm . The central hilum consists of a distinct cavity or two- to five-rayed cleft, and there are no concentric striations. Between crossed nicol prisms, the starch granules show a distinct black cross intersecting at the hilum.

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

C: ~~To 10 mL of the mucilage obtained in *Identification* test B, add 0.04 mL of iodine and potassium iodide TS; an orange-red to dark blue color is produced, which disappears on heating.~~

C: To 1 mL of the mucilage obtained in *Identification* test B, add 0.05 mL of ~~iodine solution~~: ■ iodine and potassium iodide TS 2: ■ 1S (*NF*23)

an orange-red to dark blue color is produced, which disappears on heating.

~~Iodine solution~~ — Dissolve 12.7 g of iodine and 20 g of potassium iodide in water, and dilute with water to 1000.0 mL. To 10.0 mL of this solution, add 0.6 g of potassium iodide, and dilute with water to 100.0 mL. Prepare immediately before use.

■ ■ 1S (*NF*23)

Microbial limits (61)— The total aerobic microbial count does not exceed 1000 cfu per g, the total combined molds and yeasts count does not exceed 100 cfu per 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*.

pH (791)— 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. Agitate continuously at a moderate rate for 1 minute. Stop the agitation, and allow to stand for 15 minutes. Determine the pH to the nearest 0.1 unit: the pH, determined potentiometrically, is between 4.0 and 7.0.

Loss on drying (731)— Dry about 1 g, accurately weighed, at 130° for 90 minutes: it loses not more than 15.0% of its weight.

Change to read:

Residue on ignition (281): not more than 0.6%, determined on a 1.0-g test specimen. Ignition temperature $600 \pm 50^{\circ}\text{C}$. ■

■ 1S (NF23)

Limit of iron— 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), 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 (*Test Solution*). Prepare a *Standard Iron Solution* containing the equivalent of 10 µg of iron per mL as directed under Iron (241). Immediately before use, quantitatively dilute an accurately measured volume of this solution with water to obtain a *Diluted Standard Iron Solution* containing the equivalent of 1 µg of iron per mL. Prepare the *Standard Solution* by transferring 10 mL of the *Diluted Standard Iron Solution* to a test tube and proceeding in the same manner as directed for the preparation of the *Test Solution*, beginning with “add 2 mL of citric acid solution (2 in 10).” After 5 minutes, any pink color in the *Test Solution* is not more intense than that in the *Standard Solution*, corresponding to a limit of 10 µg of iron per g.

Limit of oxidizing substances— 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 minutes. 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 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-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. Not more than 1.4 mL of 0.002 N sodium thiosulfate is required (20 µg per g, calculated as H₂O₂).

Limit of sulfur dioxide— Not more than 50 µg per g.

REAGENTS—

Carbon dioxide— Use carbon dioxide, with a flow regulator that will maintain a flow of 100 ± 10 mL per minute.

Bromophenol blue indicator solution— Dissolve 100 mg of bromophenol blue in 100 mL of dilute alcohol (1 in 5), and 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— In this test, the sulfur dioxide is released from the test specimen 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 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 having a jacket length of 200 mm, and a delivery tube connecting the upper end of the reflux condenser to the bottom of a receiving test tube. 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.

PROCEDURE— 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 per minute through the *Apparatus*. Start the condenser coolant flow. Add 10 mL of *Hydrogen peroxide solution* to a receiving test tube. After 15 minutes, without interrupting the flow of carbon dioxide, remove the separatory funnel from the boiling flask, and transfer 25.0 g of test specimen 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. ~~Position the Apparatus in a water bath, and~~ Boil the mixture for 1 hour. 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 minutes, 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, with the color change lasting for at least 20 seconds. Perform a blank determination, and make any necessary correction (see Titrimetry <541>). Calculate the content, in µg per g, of sulfur dioxide in the test specimen taken by the formula:

$$1000(32.03)VN/W,$$

in which 32.03 is the milliequivalent weight of sulfur dioxide; V is the volume, in mL, of titrant consumed; N is the normality of the titrant; and W is the weight, in g, of test specimen taken.

Organic volatile impurities, Method IV <467>: meets the requirements. ▲NF23

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Pharmacopeial Forum : Volume No. 30(4) Page 1457

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