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 20 ppm of iron.

- **LIMIT OF SULFUR DIOXIDE,** *Method IV* ⟨**525**⟩**:** NMT 50 ppm
- **LIMIT OF PROPYLENE GLYCOL**

Internal standard solution: 0.5 mg/mL of 1,3-propanediol in anhydrous pyridine

Standard stock solution: 0.5 mg/mL of USP Propylene Glycol RS in *Internal standard solution*

Standard solution: Transfer 0.1 mL of the *Standard stock solution* to a 2-mL vessel with a screw cap fitted with a septum. Add 0.9 mL of anhydrous pyridine, 0.2 mL of hexamethyldisilazane, and 0.1 mL of trimethylchlorosilane. Close, and mix. Allow to stand for 15 min before injection.

Sample stock solution: Transfer 200 mg of Pregelatinized Hydroxypropyl Potato Starch to a 100-mL volumetric flask. Add 1.0 mL of the *Internal standard solution* and 9.0 mL of anhydrous pyridine. Boil under reflux using a water bath for 20 min. Allow to cool to room temperature.

Sample solution: Transfer 1.0 mL of the *Sample stock solution* to a 2-mL vessel with a screw cap fitted with a septum. Add 0.2 mL of hexamethyldisilazane and 0.1 mL of trimethylchlorosilane. Close, and mix. Allow to stand for 15 min before injection.

Chromatographic system

(See *Chromatography* ⟨621⟩*, System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.32-mm × 30-m fused-silica capillary column; 0.25-µm layer of phase G1

Temperature

Detector: 250°

Injection port: 250°

Column: 70°. [NOTE—The column must be desorbed regularly. Conditions: Program from 70° to 300° at 7°/min, and maintain 10 min at 300°.]

Carrier gas: Helium

Flow rate: 3 mL/min

Injection type: Split ratio of 1:30

Injection size: 1 µL

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for the trimethylsylilated derivative of propylene glycol and the trimethylsylilated derivative of 1,3-propanediol are 1.0 and 1.4, respectively.]

Suitability requirements

Resolution: NLT 2.0 between the peaks due to the trimethylsylilated derivative of propylene glycol and the trimethylsylilated derivative of 1,3-propanediol

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of propylene glycol in the portion of Pregelatinized Hydroxypropyl Potato Starch taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = internal standard ratio (peak response of propylene glycol/peak response of 1,3-propanediol) from the *Sample solution*

R_S = internal standard ratio (peak response of propylene glycol/peak response of 1,3-propanediol) from the *Standard solution*

C_S = concentration of USP Propylene Glycol RS in the *Standard solution* (mg/mL)

C_U = concentration of Pregelatinized Hydroxypropyl Potato Starch in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 0.1%

- **LIMIT OF OXIDIZING SUBSTANCES**

Sample: 4.0 g

Analysis: Transfer the *Sample* to a glass-stoppered 125-mL conical flask, and add 50.0 mL of a mixture of water and methanol (1:1). 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 ppm, calculated as H_2O_2).

SPECIFIC TESTS

- **MICROBIAL ENUMERATION TESTS** ⟨**61**⟩ and **TESTS FOR SPECIFIED MICROORGANISMS** ⟨**62**⟩**:** The total aerobic microbial count does not exceed 10^3 cfu/g, the total combined molds and yeasts count does not exceed 10^2 cfu/g, and it meets the requirements of the test for the absence of *Escherichia coli*.
- **pH** ⟨**791**⟩

Sample solution: Progressively suspend 3.0 g of Pregelatinized Hydroxypropyl Potato Starch in 100.0 mL of carbon dioxide-free water, stirring continuously. Determine the pH when all the solid is wetted.

Acceptance criteria: 4.5–8.0

- **LOSS ON DRYING** ⟨**731**⟩**:** Dry about 1 g at 130° for 90 min: it loses NMT 20.0% of its weight.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers. Store at room temperature.
- **USP REFERENCE STANDARDS** ⟨**11**⟩

USP Propylene Glycol RS

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.

Pregelatinized Modified Starch

DEFINITION

Pregelatinized Modified Starch is Modified Starch that has been chemically or mechanically processed, or both, to rupture all or part of the granules to produce a product that swells in cold water.

IDENTIFICATION

- **A.**
  Sample: 0.6 g
  Analysis: Transfer the *Sample* to a 25-mL glass vial with a plastic cap. Add 9.4 g of water, cap, and shake vigorously to evenly disperse the starch. Add 10 g of 2% (w/w) NaOH solution, cap, and shake vigorously for 1 min to create a smooth mixture. Evaluate within 1 min.
  Acceptance criteria: The final solution is translucent to opaque with a fluid consistency. A yellow tint of the final solution is acceptable.
- **B.** An aqueous dispersion of Pregelatinized Modified Starch is colored orange-red to deep blue by iodine TS.

IMPURITIES

- **RESIDUE ON IGNITION** ⟨**281**⟩
  Sample: 2.0 ± 0.1 g
  Acceptance criteria: NMT 1.5%
- **LIMIT OF SULFUR DIOXIDE**
  Sample solution: Mix 20.0 ± 0.1 g of Pregelatinized Modified Starch with 100 mL of 95% alcohol, and stir for several min to completely wet the starch.
  Analysis: Slowly add 100 mL of water to the *Sample solution*, and stir until a smooth suspension is obtained. Allow the starch mixture to set undisturbed until most of the starch has settled, and filter the aqueous portion through paper (Whatman No. 1 or equivalent). To 100 mL of the clear filtrate add 100 mL of water. Add 3 mL of starch TS, and titrate with 0.01 N iodine VS to the first permanent blue or purple color.
  Acceptance criteria: NMT 1.7 mL of 0.010 N iodine is consumed (NMT 0.005%).

SPECIFIC TESTS

- **pH** ⟨**791**⟩
  Sample: 10.0 ± 0.1 g
  Analysis: Wet the *Sample* with 10 mL of alcohol, then dilute with water to 300 mL to obtain an aqueous dispersion. Stir continuously at a moderate rate for 5 min, and determine the pH to the nearest 0.1 unit.
  Acceptance criteria: 3.0–9.0
- **LOSS ON DRYING** ⟨**731**⟩
  Analysis: Dry at 120° for 4 h.
  Acceptance criteria: NMT 15%
- **MICROBIAL ENUMERATION TESTS** ⟨**61**⟩ and **TESTS FOR SPECIFIED MICROORGANISMS** ⟨**62**⟩**:** The total aerobic microbial count does not exceed 1×10^3 cfu/g, and the total combined molds and yeasts count does not exceed 1×10^2 cfu/g. It meets the requirements of the tests for absence of *Salmonella* species and *Escherichia coli*.
- **IRON** ⟨**241**⟩
  Sample: The residue obtained in the test for *Residue on Ignition* ⟨281⟩
  Analysis: Dissolve the *Sample* in 8 mL of hydrochloric acid with the aid of gentle heating. Dilute with water to 100 mL in a volumetric flask. Dilute 25 mL of this solution with water to 47 ± 1 mL.
  Acceptance criteria: NMT 20 µg/g
- **OXIDIZING SUBSTANCES**
  Sample solution: To 5 g of Pregelatinized Modified Starch add 20 mL of a mixture of methanol and water (1:1).
  Analysis: To the *Sample solution* 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.

Rice Starch

Portions of this monograph that are national *USP* text, and are not part of the harmonized text, are marked with symbols (◆) to specify this fact.

DEFINITION

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

IDENTIFICATION

- **A. PROCEDURE**
  Analysis: 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 presents polyhedral, simple grains 1–10 µm, mostly 4–6 µm in size. These simple grains often gather in ellipsoidal, compound grains 50–100 µm in diameter. The granules have a poorly visible central hilum and there are no concentric striations. Between orthogonally orientated polarising plates or prisms, the starch granules show a distinct black cross intersecting at the hilum.