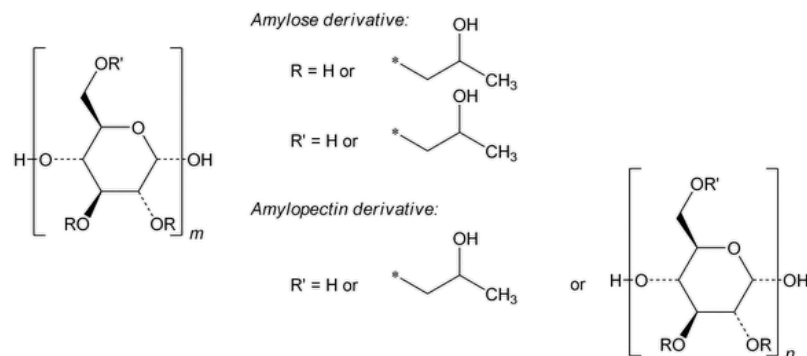


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Hydroxypropyl Pea Starch



For the Amylose derivative, m is about 300–1000.

DEFINITION

Hydroxypropyl Pea Starch is partially substituted 2-hydroxy propylether obtained from pea starch by a chemical modification of etherification with propylene oxide. In addition, this starch may be partially hydrolyzed using acids or enzymes to obtain thinned starch. It contains NLT 2.0% and NMT 7.0% of hydroxypropyl groups, on the dried basis.

IDENTIFICATION

• A. PROCEDURE

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

Acceptance criteria: 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. PROCEDURE

Sample solution: Suspend 1 g of Hydroxypropyl Pea Starch in 50 mL of water, boil for 1 min, and cool.

Acceptance criteria: A translucent or clear mucilage is formed.

• C. PROCEDURE

Analysis: To 1 mL of the *Sample solution* obtained in *Identification* test B add 0.05 mL of iodine and potassium iodide TS 2.

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

• D. PROCEDURE

Ninhydrin solution: Dissolve 3 g of ninhydrin in 100 mL of a 45.5-g/L solution of sodium metabisulfite.

Diluted sulfuric acid: 98 g/L of H_2SO_4

Sample: 100 mg of Hydroxypropyl Pea Starch

Analysis: Transfer the *Sample* to a 100-mL volumetric flask, and add 12.5 mL of *Diluted sulfuric acid*. Place the flask in a water bath, and heat until the *Sample* is dissolved. Cool, and dilute with water to 100 mL. [CAUTION—When sulfuric acid is miscible with water, it produces intense heat.]

Pipet 1 mL of this solution to a glass-stoppered, 25-mL graduated test tube and, with the tube immersed in cold water, add drop-wise 8 mL of sulfuric acid. Mix well, and place the tube in a boiling water bath for exactly 3 min. Immediately transfer the tube to an ice bath until the solution is chilled. Add 0.6 mL of *Ninhydrin solution*, carefully allowing the reagent to run down the walls of the test tube. Immediately shake the tube well, and place it in a water bath at 25° for 100 min. Dilute with sulfuric acid to 25 mL [CAUTION—Use sulfuric acid cautiously.], and mix by inverting the tube several times. Do not shake.

Acceptance criteria: A violet color develops within 5 min due to the presence of hydroxypropyl groups (starch ether).

ASSAY**• PROCEDURE FOR HYDROXYPROPYL GROUPS**

Deuterium chloride solution: Dilute 1 mL of deuterium chloride (38% w/w) with 5 mL of deuterium oxide.

Internal standard solution: Dissolve 50.0 mg of sodium 3-trimethylsilyl-1-propane sulfonate in about 5 g of deuterium oxide, weighed to the nearest 0.1 mg. Store in a sealed bottle.

Sample solution: Disperse 20 g of Hydroxypropyl Pea Starch in 200.0 mL of carbon dioxide-free water at room temperature. Agitate for 15 min, and filter. Repeat the operation two more times. If poor dispersibility or slow filtration is observed, use refrigerated carbon dioxide-free water for the washing operation. Dry the washed starch for NLT 4 h in vacuum at $30 \pm 5^\circ$. Weigh 12.0 mg of this sample in a 5-mm NMR tube. Add 0.75 mL of deuterium oxide and 0.1 mL of *Deuterium chloride solution*. Cap the tube, mix, and place it in a boiling water bath until a clear solution is obtained. [NOTE—This may take 3 min to 1 h.] When a clear solution is obtained, allow to cool to room temperature. Dry the exterior of the tube, and weigh to the nearest 0.1 mg. Add 0.05 mL of *Internal standard solution*, and weigh to the nearest 0.1 mg. Determine the mass of the *Internal standard solution* added. Mix thoroughly.

Nuclear magnetic resonance spectrometry

(See [Nuclear Magnetic Resonance Spectroscopy \(761\), Quantitative Applications](#).)

Apparatus: FT-NMR spectrometer at minimum 300 MHz

Acquisition of ^1H NMR spectra: The following parameters may be used.

Sweep width: 8 ppm (about -1.0 to +7 ppm)

Irradiation frequency offset: None

Time domain: NLT 64 K

Pulse width: 90 degree

Pulse delay: 10 s

Dummy scans: 0

Number of scans: 8

Use the CH_3 signal of the internal standard for shift referencing. Set the shift of the peak of the singlet to 0 ppm. Record the FID signal.

Analysis

Samples: *Internal standard solution* and *Sample solution*

Call the integration sub-routine after phase corrections and baseline correction between -0.5 and +6 ppm.

Measure the peak areas of the doublet from the methyl groups of the hydroxypropyl function at +1.2 ppm (A_2), and of the methyl groups at 0 ppm of the internal standard (A_1) without ^{13}C -satellites.

Measure the signal coming from the 3 protons of the methyl group in the hydroxypropyl function.

Calculate the content of hydroxypropyl groups as a percentage (w/w, dried basis):

$$\text{Result} = (N \times A_2/A_1) \times (C_i \times W_i/W) \times (M_{r2}/M_{r1}) \times [100/(100 - B)] \times 100$$

N = numerical value representing the 3 methyl groups in the internal standard (sodium 3-trimethylsilyl-1-propane sulfonate), 3

A_2 = area of the methyl groups of hydroxypropyl in Hydroxypropyl Pea Starch

A_1 = area of the methyl groups in the internal standard (sodium 3-trimethylsilyl-1-propane sulfonate)

C_i = concentration of the internal standard in the *Internal standard solution* (mg/g)

W_i = weight of the *Internal standard solution* in the NMR tube (g)

W = weight of the washed and dried Hydroxypropyl Pea Starch in the NMR tube (mg)

M_{r1} = molecular weight of the internal standard, 218.32 g/mol

M_{r2} = molar mass of hydroxypropyl group, 59.09 g/mol

B = moisture content of the washed and dried Hydroxypropyl Pea Starch used in the *Sample solution*, as a percentage (w/w)

Acceptance criteria: The content of hydroxypropyl groups is 2.0%–7.0%.

IMPURITIES**INORGANIC IMPURITIES**

• [RESIDUE ON IGNITION \(281\)](#): NMT 0.6%, determined on a 1.0-g test specimen

Change to read: