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Hydroxypropyl Starch

(Tentative)

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HYDROXYPROPYL STARCH
(TENTATIVE)

Prepared at the 82nd JECFA (2016) and published in FAO JECFA Monograph 19 (2016), superseding specifications for Hydroxypropyl starch included in the specifications for Modified starches prepared at the 79th JECFA (2014), published in FAO JECFA Monographs 16 (2014). An ADI "not specified" was established at the 26th JECFA (1982).

Information is required on:

- *A suitable method for the determination of propylene chlorohydrin*

SYNONYMS INS No. 1440

DEFINITION Starch is a carbohydrate polymer consisting of alarge number of glucose units linked together primarily by alpha 1-4 glucosidic bonds. The starch polymers come in two forms: linear (amylose)and branchedthrough alpha 1-6 glucosidic bonds (amylopectin), with each glucose unit possessing a maximum of three hydroxyls that can undergo chemical substitution.
Hydroxypropyl starch is a modified starch. It is obtained by etherification of food starch with propylene oxide, in accordance with good manufacturing practice. Hydroxypropylation results in substitution of hydroxyl groups with 2-hydroxypropyl ether.

Hydroxypropyl starch may additionally be subjected to acid, alkali, enzyme, or bleaching treatment in accordance with good manufacturing practice.

C.A.S number 9049-76-7
74315-67-6 (modified amylopectin)

DESCRIPTION White or nearly white powder or granules or (if pregelatinized) flakes, or amorphous powder or coarse particles.

FUNCTIONAL USES Thickener, stabilizer, binder, emulsifier

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in cold water (if not pre-gelatinized); forming typical colloidal solutions with viscous properties in hot water; insoluble in ethanol.

Microscopy Passes test
See description under TESTS

Iodine stain Passes test
See description under TESTS

Copper reduction	Passes test See description under TESTS
Hydroxypropyl ether groups	Passes test See description under TEST
PURITY	
Loss on drying	Cereal starch: not more than 15.0% Potato starch: not more than 21.0% Other starches: not more than 18.0% (120°, 4 h, vacuum not exceeding 100 mm Hg)
Hydroxypropyl groups	Not more than 7.0% on the dried basis See description under TESTS
Propylene chlorohydrin	Not more than 1 mg/kg See description under TESTS
Sulfur dioxide (Vol. 4)	Not more than 50 mg/kg on the dried basis for modified cereal starches Not more than 10 mg/kg on the dried basis for other modified starches
Lead (Vol. 4)	Not more than 2 mg/kg on the dried basis Determine using a method appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under “General Methods, Metallic Impurities”).
Manganese (Vol. 4)	Not more than 50 mg/kg on the dried basis Determine using a method appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under “General Methods, Metallic Impurities”).
Carboxyl groups (Vol. 4)	Not more than 0.1% on the dried basis

TESTS

IDENTIFICATION TESTS	
Microscopy	Modified starches which have not been pre-gelatinized retain their granular structure and can be identified as starches by microscopic observation. Shape, size and sometimes striations are characteristics of the botanical origin. In polarized light under cross nicol prisms the typical polarization cross will be observed
Iodine stain	Add a few drops of 0.1 N potassium tri-iodide to an aqueous suspension of the sample. These starches stain with iodine in the same way as native starches. The colour can range from dark blue to red

Copper reduction

Place about 2.5 g of the sample previously washed with water, in a boiling flask, add 10 ml of dilute hydrochloric acid (3%) and 70 ml of water, mix, reflux for about three hours and cool. Add 0.5 ml of the resulting solution to 5 ml of hot alkaline cupric tartrate TS. A copious red precipitate is produced

Hydroxypropyl ether groups[1]

Ninhydrin reagent
A 3% solution of 1,2,3-triketohydrindene crystals in 4.55% aqueous sodium bisulfite solution.

Procedure
Weigh 100 mg of the sample into a 100-ml volumetric flask and add 12.5 ml of 2 N sulfuric acid. Prepare a sample of unmodified starch of the same source (i.e. corn or potato) in the same manner. Place the flasks in a boiling water bath and heat until the samples are in solution. Cool and dilute the contents to 100 ml with water. Pipet 1 ml of the solutions into 25-ml graduated test tubes with glass stoppers and, with the tubes immersed in cold water, add dropwise 8 ml of concentrated sulfuric acid to each. Mix well and place the tubes in a boiling water bath for exactly 3 min. Immediately transfer the tubes to an ice bath until the solution is chilled. Add 0.6 ml of ninhydrin reagent, carefully allowing the reagent to run down the walls of the test tubes. Immediately shake well, and place the tubes in a 25° water bath for 100 min. Adjust the volume in each tube to 25 ml with concentrated sulfuric acid and mix by inverting the tubes several times. (Do not shake).
A violet colour develops only in the modified sample within 5 min due to the presence of hydroxypropyl groups (starch ether).For all other non-hydroxypropyl treated starches a light pink colour is observed.

PURITY TESTS

Hydroxypropyl groups

Ninhydrin reagent
A 3% solution of 1,2,3-triketohydrindene crystals in 5% aqueous sodium bisulfite solution.

Procedure
Accurately weigh 50 - 100 mg of the sample into a 100-ml volumetric flask and add 25 ml of 1 N sulfuric acid. Prepare a sample of unmodified starch of the same source (i.e. corn or potato) in the same manner. Place the flasks in a boiling water bath and heat until the samples are in solution. Cool and dilute the contents to 100 ml with water. If necessary, dilute the sample further to assure the presence of no more than 4 mg of hydroxypropyl group per 100 ml, and then dilute the blank starch in the same proportion. Pipet 1 ml of the solutions into 25-ml graduated test tubes with glass stoppers and, with the tubes immersed in cold water, add dropwise 8 ml of concentrated sulfuric acid to each. Mix well and place the tubes in a boiling water bath for exactly 3 min. Immediately transfer the tubes to an ice bath until the solution is chilled. Add 0.6 ml of ninhydrin reagent, carefully allowing the reagent to run down the walls of the test tubes. Immediately shake well, and

[1] USP29-NF34: U.S. Pharmacopeial Convention, Hydroxylpropyl corn starch monograph, 2015. Reproduced with permission.

place the tubes in a 25° water bath for 100 min. Adjust the volume in each tube to 25 ml with concentrated sulfuric acid and mix by inverting the tubes several times. (Do not shake). Immediately transfer portions of the solutions to 1-cm cells and after exactly 5 min, measure the absorption (A) at 590 nm, using the starch blank as the reference. Prepare a calibration curve with 1-ml aliquots of standard aqueous solutions, containing 10, 20, 30, 40 and 50 µg of propylene glycol per ml.

Calculation

$$\text{Hydroxypropyl groups (\%)} = \frac{C \times 0.7763 \times 10 \times F}{W}$$

where

C is the amount of propylene glycol in the sample solution read from the calibration curve (µg/ml);
F is the dilution factor (if a further dilution has been necessary); and
W is the weight of sample (mg).

Propylene chlorohydrin *Information required*