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# Starch Sodium Octenylsuccinate

(Tentative)

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# STARCH SODIUM OCTENYLSUCCINATE
(TENTATIVE)

*Prepared at the 82nd JECFA (2016) and published in FAO JECFA Monograph 19 (2016), superseding specifications for Starch sodium octenylsuccinate 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 test for identification of the octenylsuccinate groups*

**SYNONYMS** INS No. 1450

DEFINITION Starch is a carbohydrate polymer consisting of a large number of glucose units linked together primarily by alpha 1-4 glucosidic bonds. The starch polymers come in two forms: linear (amylose) and branched through alpha 1-6 glucosidic bonds (amylopectin), with each glucose unit possessing a maximum of three hydroxyls that can undergo chemical substitution.

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Starch sodium octenylsuccinate is a modified starch. It is obtained by esterification of food starch with octenylsuccinic anhydride, and neutralisation with either sodium hydroxide or sodium carbonate as a pH buffer, in accordance with good manufacturing practice.

Starch sodium octenylsuccinate may additionally be subjected to acid, alkali, enzyme, or bleaching treatment in accordance with good manufacturing practice.

C.A.S number 66829-29-6
52906-93-1
125109-81-1 (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<br>See description under TESTS |
| Copper reduction | Passes test<br>See description under TESTS |
| Ester groups | Passes test<br>See description under TESTS |
| *Octenylsuccinate groups* | *Information required* |

PURITY

| | |
|---|---|
| Loss on drying | Cereal starch: not more than 15.0%<br>Potato starch: not more than 21.0%<br>Other starches: not more than 18.0%<br>(120°, 4 h, vacuum not exceeding 100 mm Hg) |
| Octenylsuccinyl groups | Not more than 3% on the dried basis<br>See description under TEST |
| Residual octenylsuccinic acid | Not more than 0.3% on the dried basis<br>See description under tests |
| Sulfur dioxide (Vol. 4) | Not more than 50 mg/kg on the dried basis for modified cereal starches<br>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<br><br>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<br><br>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

Ester groups — The infrared spectrum of a thin film gives a typical absorption band at about 1720 cm-1 which is an indication for ester groups.
The limit of detection is about 0.5% octylsuccinyl groups in the product.

*Octenylsuccinyl groups* — *Information required*

PURITY TESTS

Octenylsuccinyl groups in starch sodium octenyl succinate

Principle

The sample is equilibrated with mineral acid to convert octenyl succinate salts to the acid form. Cations and excess acid are removed by thorough washing with 90% isopropanol in water. The washed sample is titrated with standard alkali.

Procedure

Weigh accurately about 5.000 g of sample into a 150-ml beaker and wet the sample with a few ml of isopropanol. Add 25 ml of 2.5 M hydrochloric acid in isopropanol, allowing the acid to wash down any sample on the sides of the beaker. Stir the mixture with a magnetic stirrer for 30 min. Using a graduated measuring cylinder, add 100 ml of 90% isopropanol in water and stir the contents for another 10 min. Filter through a Buchner funnel and wash the filter cake with 90% isopropanol in water until the filtrate is negative for chloride (check using 0.1 M silver nitrate). Quantitatively transfer the filter cake into a 600-ml beaker using distilled water and, making sure to rinse the Buchner funnel to wash any starch into the beaker.Bring to about 300-ml using distilled water. Place the beaker on a boiling water bath for 10 min with stirring. Titrate, while hot, with 0.1 M sodium hydroxide using phenolphthalein TS as an indicator. Repeat the titration procedure with native unmodified starch of the same origin as the OSA starch sample, as a blank.

Calculation

$$\text{Octenylsuccinyl groups (\%)} = \frac{21.1 \times M \times [V_{sample} - V_{blank}]}{W}$$

where

$V_{sample}$ is the titration volume of sodium hydroxide for the sample, ml
$V_{blank}$ is the titration volume of sodium hydroxide for the blank, ml
M is the molarity of sodium hydroxide
W is the dry weight of sample, g

Residual octenyl succinic acid in starch Sodium octenyl succinate

Determine by HPLC on the 2-bromoacetophenone-derivatised methanolic extract of the sample.

Extraction and Preparation of Sample Solution

Accurately weigh 500 mg (to nearest 0.1 mg) of the sample in a 25 ml Erlenmeyer flask, add 15 ml of methanol, stopper the flask and shake it on a shaker overnight. Filter the extract using a filter paper, wash the residue, three times with 7 ml portions of methanol and combine the filtrate (about 80% of the OSA residues is extracted by this procedure). Add 1 ml of 0.16 M KOH in methanol to the combined filtrate. Dry the extract using a flash evaporator at 30° and dissolve the residue in 2 ml of methanol. Pipette 0.5 ml of this solution into a reaction vial, add 0.5 ml of derivatisation reagent [2.8 g of 2-p-dibromoacetophenone and 0.28 g of 1,4,7,10,13,16-hexaoxacyclooctadecane (18-Crown-6) in 50 ml $CH_3CN$]. Add 2 ml $CH_3CN$ to the reaction vial, cap the vial and heat at 80° for 30 min. Allow the vial to reach room temperature and analyse by HPLC within 24 h.

HPLC Conditions:

Column: µ-Bondapack C18 or equivalent
Mobile Phase: Methanol and Water with gradient elution: 70% to 80% of methanol in water in 5 min
Flow rate: 1.5 ml/min
Detector: UV at 254 nm
Injection volume: 5 µl

Preparation of Standard Curve

Prepare a 105.14 mg/ml solution of octenylsuccinic acid anhydride (available from Milliken Chemicals) in methanol (Solution A). Using a syringe draw 0.25 ml of Solution A, transfer into a 25-ml volumetric flask and dilute to mark with methanol (Solution B).

Prepare three working standards (Solution C1, C2 and C3) by transferring 0.5, 1 and 2 ml each of Solution B into three 50-ml round bottom flasks, add 1 ml of 0.16 M KOH in methanol to each flask, dry the solution using a flash evaporator at 30° and dissolve the residue in 2.0 ml of methanol. To 0.5 ml each of these solutions in reaction vials, add 0.5 ml each of derivatisation reagent [2.8 g of 2-p-dibromoacetophenone and 0.28 g of 1,4,7,10,13,16-hexaoxacyclooctadecane (18-Crown-6) in 50 ml of $CH_3CN$]. Add 2 ml of $CH_3CN$ to each vial; cap the vials and heat for 30 min at 80°. Allow the vials to reach room temperature and analyze by HPLC immediately. The amount of octenyl succinic acid in each 5-µl injection is as follows:

Solution C1: 0.2375 µg
Solution C2: 0.4750 µg
Solution C3: 0.9500 µg

Construct the standard curve using peak height against the amount of standard in the injection.

Inject 5-µl of prepared sample solution and read the amount of octenyl succinic acid in the injection from the standard curve.

Calculation

$$\% \text{ Residual octenyl succinic acid} = \frac{300 \times V}{W}$$

where

V is the amount of OSA in the injected volume; and
W is the weight of the sample (mg).

NOTE: The formula is corrected to 100% recovery by dividing with 0.80, so that 240/0.80 = 300.