



Food and Agriculture
Organization of the
United Nations



World Health
Organization

Residue Monograph prepared by the meeting of the Joint FAO/WHO Expert Committee
on Food Additives (JECFA), 82nd meeting 2016

ACETYLATED OXIDIZED STARCH

This monograph was also published in: *Compendium of Food Additive Specifications. Joint
FAO/WHO Expert Committee on Food Additives (JECFA), 82nd meeting 2016. FAO JECFA
Monographs 19*

ACETYLATED OXIDIZED STARCH

Prepared at the 82nd JECFA (2016) and published in FAO JECFA Monograph 19 (2016), superseding specifications for Acetylated oxidized 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 57th JECFA (2001).

SYNONYMS

INS No. 1451

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.

Acetylated oxidized starch is a modified starch. It is obtained by treatment of food starch with sodium hypochlorite followed by esterification with acetic anhydride in accordance with good manufacturing practice. Oxidation involves the deliberate production of carboxyl groups. Acetylation results in substitution of hydroxyl groups with acetyl esters.

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

C.A.S number

68187-08-6

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

<u>Hypochlorite oxidized starch</u>	See description under TESTS Passes test See description under TESTS
<u>Specific reaction for acetyl groups</u>	Passes test See description under TESTS
<u>Ester groups</u>	Passes test See description under TESTS
PURITY	
<u>Loss on drying</u>	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)
<u>Acetyl groups</u>	Not more than 2.5 % on the dried basis See description under TESTS
<u>Carboxyl groups (Vol. 4)</u>	Not more than 1.3 % on the dried basis
<u>Sulfur dioxide (Vol. 4)</u>	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
<u>Lead (Vol. 4)</u>	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”).
<u>Manganese (Vol. 4)</u>	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”).
TESTS	
IDENTIFICATION TESTS	
<u>Microscopy</u>	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
<u>Iodine stain</u>	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

Hypochlorite oxidized starchPrinciple

Because of the carboxyl group content, hypochlorite-oxidized starch has anionic properties. It can be dyed with positively charged dyes such as methylene blue. The test is not suitable for acetylated slightly oxidized potato starch due to the presence of phosphate groups.

Procedure

50 mg of the sample are kept in suspension for 5-10 min in 25 ml of a 1% aqueous dye solution and stirred occasionally. After decantation of the excess solution, the starch is washed with distilled water. Microscopic inspection clearly shows colouring, if the sample is hypochlorite-oxidized starch. By this test hypochlorite-oxidized starch is distinguished from native and acid modified starch of the same botanical origin.

Specific reaction for acetyl groupsPrinciple

Acetate is liberated upon saponification of acetylated starch. After concentration the acetate is converted to acetone by heating with calcium hydroxide. The acetone thus produced stains blue with o-nitrobenzaldehyde.

Procedure

About 10 g of the sample is suspended in 25 ml water to which is added 20 ml of 0.4 M NaOH. After shaking for 1 h the starch is filtered off and the filtrate evaporated in an oven at 110°. The residue is dissolved in a few drops of water and transferred to a test tube. Add calcium hydroxide and heat the tube. If the sample is acetylated starch, acetone vapours are produced. These produce a blue colour on a paper strip soaked in a fresh saturated solution of o-nitrobenzaldehyde in 2 M NaOH. The blue colour is more distinct when the original yellow colour of the reagents is removed with 1 drop of a 1 in 10 solution of hydrochloric acid.

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% acetyl groups in the product.

PURITY TESTS

Acetyl groups

Accurately weigh about 5 g of the sample and transfer into a 250 ml conical flask. Suspend in 50 ml of water, add a few drops of phenolphthalein TS, and titrate with 0.1 M sodium hydroxide to a permanent pink end-point. Add 25.0 ml of 0.45 M sodium hydroxide, stopper the flask, and shake vigorously for 30 min, preferably with a mechanical shaker. (NOTE: the temperature should not exceed 30° as some starches may gelatinize). Remove the stopper, wash the stopper and sides of the flask with a few ml of water, and titrate the excess

alkali with 0.2 M hydrochloric acid to the disappearance of the pink colour. Record the volume, in ml of 0.2 M hydrochloric acid required as S.

Perform a blank titration on 25.0 ml of 0.45 M sodium hydroxide, and record the volume, in ml, of 0.2 M hydrochloric acid required as B.

$$\text{Acetyl groups (\%)} = \frac{(B - S) \times M \times 0.043 \times 100}{W}$$

where

M is the molarity of hydrochloric acid solution; and

W is the weight of sample, In grams.