



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), 82<sup>nd</sup> meeting 2016

## **Acetylated Distarch Phosphate**

(Tentative)

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

## ACETYLATED DISTARCH PHOSPHATE (TENTATIVE)

*Prepared at the 82nd JECFA (2016) and published in FAO JECFA Monograph 19 (2016), superseding specifications for Acetylated distarch phosphate included in the specifications for Modified starches prepared at the 79th JECFA (2014), published in FAO JECFA Monographs 17 (2014). An ADI “not specified” was established at the 26th JECFA (1982).*

*Information is required on:*

- *A suitable test for identification of the phosphate groups and of crosslinking.*

### SYNONYMS

INS No. 1414

### 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 distarch phosphate is a modified starch. It is obtained by esterification/cross-linking of food starch with sodium trimetaphosphate or phosphorus oxychloride combined with esterification with acetic anhydride or vinyl acetate in accordance with good manufacturing practice. Acetylation results in substitution of hydroxyl groups with acetyl esters. In cases of cross-linking, where a polyfunctional substituting agent, such as phosphorus oxychloride, connects two chains, the structure can be represented by: Starch-O-R-O-Starch, where R = cross-linking group and Starch refers to the linear and/or branched structure.

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

### C.A.S number

9067-33-8  
68130-14-3  
113894-91-0 (modified amylopectin)

### DESCRIPTION

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

### FUNCTIONAL USES CHARACTERISTICS

Thickener, stabilizer, binder, emulsifier

### 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.

<u>Microscopy</u>	Passes test See description under TESTS
<u>Iodine stain</u>	Passes test See description under TESTS
<u>Copper reduction</u>	Passes test See description under TESTS
<u>Phosphate groups</u>	<i>Information required</i>
<u>Crosslinking</u>	<i>Information required</i>
<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 (Vol. 4)</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>Phosphate (calculated as phosphorus) (Vol. 4)</u>	Not more than 0.14% on the dried basis for potato and wheat starch Not more than 0.04% on the dried basis for other starches
<u>Vinyl acetate</u>	Not more than 0.1 mg/kg See description under TESTS
<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”).
<u>Carboxyl groups (Vol. 4)</u>	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

#### Specific reaction for acetyl groups

##### Principle

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<sup>-1</sup> 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.

### Vinyl acetate

- Headspace Gas Chromatographic method

#### Chromatographic system

Use a gas chromatograph equipped with a 2 m x 2 mm (i.d.) glass column containing Porapak Q, 80-100 mesh (or equivalent) fitted with a flame ionization detector, under the following conditions:

- Carrier gas flow (nitrogen): 20 ml/min
- injection port temperature: 200°
- column temperature: 50
- detector temperature: 200°

Standard preparation: Accurately weigh 150 mg vinyl acetate (reagent grade) into a 100 ml volumetric flask. Dissolve and make up to volume with distilled water. Place 1 ml of this solution in a 10-ml volumetric flask and make up to volume with distilled water. Add 1 ml of this dilute solution to 30 g unmodified starch of the same botanical origin as the test substance in a 100-ml flask with a septum-liner. Seal the flask immediately with the septum-liner. This provides a standard starch preparation with a vinyl acetate content of 5 mg/kg.

#### Procedure

Weigh 30 g of the test substance into a 100-ml flask with a septum-liner. Seal the flask. Place the flask containing the test substance and the flask containing the standard preparation in a constant temperature water bath at 70° for 30 min. Withdraw 2.0 ml from the headspace volume of the flask containing the standard preparation using a gas-tight syringe, inject directly into the injection port of the gas chromatograph and record the peak height of the chromatogram. Similarly inject 2.0 ml of the headspace volume from the flask containing the test substance into the chromatograph. Calculate the content of vinyl acetate in the test substance from a comparison of the peak heights of the two chromatograms.