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Starch acetates — Specifications and test methods

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products).*

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Starch consists mainly of amylose and amylopectin. Amylose is a linear molecule of α-D-glucopyranosyl units linked by (1-4)-α-linkages. Amylopectin is a highly branched polymer of α-D-glucopyranosyl units linked by (1-4)-α-linkages and by (1-6)-α- linkages that constitute the branch points. In general, each glucose unit possesses a maximum of three hydroxyls that can undergo chemical substitution. A fourth substitution is also possible at carbon four (4) if that carbon is not involved in a glycosidic bond. Native starches can be chemically modified for improved functionality. The most common sources of native starch used in these modifications are various roots, tubers, cereals and legumes. Modified starches are used in applications requiring special properties that are not attainable by their respective native starches.

Acetylated forms of food starches (including those extracted from hybrid crops such as high-amylose maize) are widely accepted additives that are used in the food industry globally. Starch acetate (INS No. 1420), produced by esterification of food starch with acetic anhydride or vinyl acetate, with the acetyl groups not exceeding more than 2,5 % of the acetylated product.

Starch acetates — Specifications and test methods

1 Scope

This document specifies the physical, chemical and microbiological requirements for and test methods of starch acetates.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1666, *Starch — Determination of moisture content — Oven-drying method*

ISO 3188, *Starches and derived products — Determination of nitrogen content by the Kjeldahl method — Titrimetric method*

ISO 3947, *Starches, native or modified — Determination of total fat content*

ISO 580, *Plastics piping and ducting systems — Injection-moulded thermoplastics fittings — Methods for visually assessing the effects of heating*

ISO 5379, *Starches and derived products — Determination of sulfur dioxide content — Acidimetric method and nephelometric method*

ISO 11212-1, *Starch and derived products — Heavy metals content — Part 1: Determination of arsenic content by atomic absorption spectrometry*

ISO 11212-2, *Starch and derived products — Heavy metals content — Part 2: Determination of mercury content by atomic absorption spectrometry*

ISO 11212-3, *Starch and derived products — Heavy metals content — Part 3: Determination of lead content by atomic absorption spectrometry with electrothermal atomization*

ISO 4832:2006, *Microbiology of food and animal feeding stuffs — Horizontal method for the enumeration of coliforms — Colony-count technique*

ISO 4833-1, *Microbiology of the food chain — Horizontal method for the enumeration of microorganisms — Part 1: Colony count at 30 °C by the pour plate technique*

ISO 4833-2, *Microbiology of the food chain — Horizontal method for the enumeration of microorganisms — Part 2: Colony count at 30 °C by the surface plating technique*

ISO 21527-2, *Microbiology of food and animal feeding stuffs — Horizontal method for the enumeration of yeasts and moulds — Part 2: Colony count technique in products with water activity less than or equal to 0,95*

AOAC official method 2011.14:2011, Calcium, Copper, Iron, Magnesium, Manganese, Potassium, Phosphorus, Sodium, and Zinc in Fortified Food Products. Microwave Digestion and Inductively Coupled Plasma-Optical Emission Spectrometry.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
starch
starch is a carbohydrate polymer consisting of a large number of glucose units linked together primarily by alpha 1-4 glycosidic bonds

Note 1 to entry: The starch polymers come in two forms: linear (amylose) and branched through alpha 1-6 glycosidic bonds (amylopectin), with each glucose unit possessing a maximum of three hydroxyls that can undergo chemical substitution

3.2
native starch
starch extracted from plant cells in its natural state as granules, which has not been subjected to any form of modification resulting in physical and/or chemical change

3.3
starch acetate
modified starch esterified with acetic anhydride or vinyl acetate. It is gluten free and can be used as a stabilizer, thickener, binder, emulsifier during food and cosmetic processing

4 Requirements

4.1 Physical indexes

Physical indexes shall comply with the requirements given in Table 1.

Table 1 — Physical requirements of starch acetates

Item	Description
Appearance	Powder, granule, coarse particles
Colour	White or nearly white
Microscopy	Granular structure typical of the starch source Typical polarization cross
Solubility	Insoluble in cold water, ether, alcohol
Smell and taste	No smell and foreign tastes
Foreign material	Free of any foreign matter

4.2 Chemical indexes

Chemical indexes shall comply with the requirements given in Table 2.

Note Sulfur dioxide method incorporates the ISBT [Manual][3] with modifications.

Table 2 — Chemical indices of starch acetates

Item	Limit/Description	Test Method
Iodine stain	dark blue to red colour	Clause 5.1
pH	3,0 – 9,0	Clause 5.2
TS – test solution; d.w - dry weight; NMT – not more than		

Table 2 *(continued)*

Item	Limit/Description		Test Method
Sulfur dioxide	≤ 50 mg/kg dry weight for modified cereal starches	≤ 10 mg/kg dry weight for other modified starches	Clause 5.3
Moisture content	≤ 14 %		ISO 1666
Copper reduction	Copious red precipitate		Clause 5.4.2
Proportion % for dry weight	Acetyl groups NMT 2,5	Ester groups NMT 0,5	Clause 5.4
TS – test solution; d.w - dry weight; NMT – not more than			

4.3 Contaminant limits

Contaminant limit shall comply with the requirements shall comply with given in Table 3.

Table 3 — Contaminant limits of Starch acetates

Item	Limit (NMT)	Test method
Arsenic (As) mg/kg	1	ISO 11212-1
Lead (Pb) mg/kg	2	ISO 11212-3
Mercury (Hg) mg/kg	0,1	ISO 11212-2
Manganese (Mn) mg/kg	50	AOAC Method
Crude Fat, %	0,15	ISO 3947
Protein, %	1	ISO 3188
TS – test solution; d.w - dry weight; NMT – not more than		

4.4 Microbiological limit

Pathogenic bacterium limit shall comply with the requirements given in Table 4

Table 4 — Pathogenic bacterium limit

Pathogenic bacterium [a]CFU/g	**Limit**	**Test method**
Aerobic Plate Count	≤ 1 000	ISO 4833-1, ISO 4833-2
Yeast and Mold	≤ 1 000	ISO 21527-2
Total Coliform	≤10	ISO 4832
[a] Colony forming unit (CFU)		

5 Test Methods

5.1 Iodine Stain

To an aqueous suspension of the starch acetate sample, add 3-5 drops of 0,1 N potassium triiodide. A colour change from dark blue to red should be observed.

5.2 pH

Add 10 g of starch acetate sample to distilled water (90 ml) and agitate continuously at a moderate rate for 5 minutes. Determine pH with a pH meter electrode that has been calibrated with the appropriate pH standards.

5.3 Sulfur dioxide

5.3.1 Procedure

— Accurately weigh 50 g of starch acetates into a 250 ml Erlenmeyer flask to produce a 20 % (w/v) starch solution; weigh 25 g if the sulfur dioxide level is more than 8 mg/l.

— Add sufficient purified water to bring total volume to 250 ml.

— Mix the starch acetates and water until the solution is homogenous.

— Cool to 10 °C or below.

— Place cold sample on a magnetic stirrer and stir at a rate sufficient to produce a small vortex at the solution surface.

— Add 10 ml of cold 1,5 N sodium hydroxide solution and stir for 15 to 20 seconds. Add 10 ml of cold 2,0 N sulfuric acid solution; titrate immediately with 0,005 N standard iodine solution until a light blue colour persists for one minute.

— Perform a blank titration using 200 mL of purified water and all reagents.

5.3.2 Calculation

$$C\ (\mathrm{mg/L}) = \frac{\left(v_{sample} - v_{blank}\right) \times N\ \mathrm{Iodine} \times 0{,}032^{*} \times 1{,}000{,}000}{m_s\ (\mathrm{g})}$$

$$^{*}\ (\mathrm{mEq})\ \mathrm{Milliequivalent\ Weight\ of\ Sulfur\ Dioxide} = \frac{64{,}07\ \mathrm{g/mole}}{(2 \times 1\ 000)}$$

where

c_{Sul} concentration of sulfur (mg/l)

v_{sample} volume of sample titrant (ml)

v_{blank} volume of blank titrant (ml)

m_s mass of sample (g)

N eq/l

5.4 Acetyl and Ester groups

5.4.1 Qualitative analysis for acetyl groups

5.4.1.1 Procedure of analysis for acetyl groups

— Suspend the sample (10 g) in distilled water (25 mL).

— Add 0,4 M NaOH (20 mL) and shake for 1 hour.

— Filter the resultant mix and collect the filtrate.

— Heat the filtrate in a 110 °C oven to achieve complete evaporation.

— Dissolve the remaining residue, from evaporation, with 3-5 drops of distilled water and transfer to a test tube.

— Add a spatula of calcium hydroxide and apply heat

  — If the sample is an acetylated starch, acetate will be liberated upon saponification of acetylated starch (heating with calcium hydroxide) and converted to acetone.

  — The acetone vapours generated will 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 as a result, of the acetone being stained by o-nitrobenzaldehyde, and the colour is more distinct when the original yellow colour of the reagents is removed with 1 drop of a 1:10 solution of hydrochloric acid.

5.4.2 Copper reduction

5.4.2.1 Qualitative analysis for acetyl groups

— Wash approximately 2,5 g of sample with distilled water and place in a boiling flask.

— Add 3 % hydrochloric acid (10 ml) and distilled water (70 ml).

— Mix**,** reflux for approximately three hours and cool.

— Add 0,5 ml of the resulting solution to 5 ml of hot alkaline cupric tartrate TS.

Note Copious red precipitate is observed after addition of hot alkaline cupric tartrate to a test sample refluxed under acidic condition.

5.4.3 Quantitative analysis for ester groups

The infrared spectrum of a thin film gives a typical absorption band at about 1 720 cm^{-1} which is an indication for ester groups. The limit of detection is approximately 0,5 % acetyl groups in the product.

5.4.3.1 Procedure of analysis for ester groups

— Accurately weigh 5 g of the sample and transfer into a 250 ml conical flask.

— Suspend in distilled water (50 mL), add 5-8 drops of phenolphthalein TS, and titrate with 0,1 M sodium hydroxide to a permanent pink endpoint.

— Add 0,45 M sodium hydroxide (25,0 mL), stopper the flask, and shake vigorously for 30 minutes, preferably with a mechanical shaker.

  NOTE Temperatures should not exceed 30 °C as some starches may gelatinize.

— Remove stopper and wash with a small quantity of distilled water, allowing the water that runs off the stopper to flow into the flask and not spill.

— Use 3-5 mL of distilled water to also wash the sides of the flask.

— 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 the 0,2 M hydrochloric acid required as B.

— Using equation 1, calculate the 5 acetyl groups present.

  Equation 1

$$\text{Acetyl group \%} = \frac{(B-S)\times M\times 0,043\times 100}{W}$$

where

M molarity of hydrochloric acid solution; and

W weight of sample

S volume in mL of 0,2 M hydrochloric acid

B 0,2 M hydrochloric acid required in mL

6 Marking, packaging, transport, and storage requirements

6.1 Marking

At least the following information shall be marked on each package or on a label:

— Name of the product, trade name or brand name, if any;

— Name and address of the producer or packer;

— Net weight;

— Producing country;

— Batch code / Lot identification;

— Date of minimum durability;

— Storage mode and instructions.

6.2 Packaging

Packaging in contact with starch acetates should be fit for food contact, safe, fresh, clean, dry, impervious to air, resistant to movement and not harmful to the health of the product.

6.3 Transport

Transportation of starch acetates consumed in the food industry can be carried out by specially designed, sterile carrier vessel. The products should be protected from exposure to sunlight and rain, and shall not be transported with toxic; corrosive material or material with peculiar smell or that might have bad effect on the quality.

6.4 Storage

The products should be stored in a hygienic and odor-free warehouse. Do not store products together with toxic, corrosive material or material with peculiar smell or that might volatilize. The products should be stored away from wall and ground. Put products in order and leave a channel in the middle.

Bibliography

[1] FAO/WTO Modified Starches Residue Monograph prepared by the meeting of the Joint FAO/WTO Expert Committee on Food Additives (JECFA), 86th Meeting 2018 FAO JECFA

[2] International Starch Institute 2*ISI 20-1e Determination of Sulphur Dioxide in Starch* Test Method and Specification Denmark

[3] International Society of Beverage Technologists (ISBT), High Fructose Syrups 42 & 55 Quality Guidelines and Analytical Procedures, 2020.

[4] Codex General Standard for Food Additives, CODEX 2013