
**Fruits, vegetables and derived
products — Determination of sorbic acid
content**

*Fruits, légumes et produits dérivés — Détermination de la teneur en
acide sorbique*

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5519 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 3, *Fruit and vegetable products*.

This second edition cancels and replaces the first edition (ISO 5519:1978), which has been technically revised.

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Introduction

The determination of the sorbic acid content of fruits, vegetables and derived products has been studied in numerous projects during the acid's use as a fungicide, especially in wines. Because of its great volatility (very similar to that of acetic acid), the simplest extraction process is its entrainment by steam. This method has the advantage of producing an almost pure aqueous solution of sorbic acid.

Two techniques for the determination of the quantity of sorbic acid contained in this solution are described in this International Standard, namely:

- Technique A: spectrophotometry in the ultraviolet range, carried out after oxidation of sulfur dioxide, which would interfere. The oxidation occurs spontaneously in a few minutes in air, after the addition of a trace of a copper catalyst.
- The natural essential oils of citrus fruits do not interfere with the determination, provided that they are present in the small quantities normal in juice not enriched with essential oils. When the quantities of essential oils are significant, they may be eliminated beforehand by the same method as that applied in technique B.
- Technique B: colorimetry based on Schmidt's reaction, which requires the elimination of ethanol and essential oils by the evaporation of an aliquot portion of the distillate. This technique, not as rapid as technique A, but giving comparable results, is provided for use when a spectrophotometer allowing measurements in the ultraviolet range is not available.

The interference caused by essential oils of garlic, onion or leek may be eliminated, when using either technique, by the evaporation of an aliquot portion of the distillate.

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Fruits, vegetables and derived products — Determination of sorbic acid content

1 Scope

This International Standard specifies a method for extracting the sorbic acid present in fruits, vegetables and derived products, and two techniques for determining the sorbic acid extracted.

2 Principle

Homogenization of the product, followed by quantitative entrainment, by steam, of the sorbic acid present in a test portion. Determination of this acid in the distillate obtained, either by spectrophotometry in the ultraviolet range (technique A), or by measuring by photolorimetry or by spectrophotometry, the pink colour obtained after oxidation by chromic acid and then treatment with thiobarbituric acid (technique B).

3 Reagents

Use only reagents of recognized analytical quality, and distilled water or water of at least equivalent purity.

3.1 Tartaric acid $[\text{COOH}(\text{CH}(\text{OH}))_2\text{COOH}]$, crystalline.

3.2 Sorbic acid $[\text{CH}_3(\text{CH}:\text{CH})_2\text{COOH}]$, 0,010 g/l standard solution, prepared by one of the following methods (3.2.1 or 3.2.2).

3.2.1 Dissolve 0,100 g of sorbic acid in 10 ml to 12 ml of a 0,1 N sodium hydroxide solution. Transfer quantitatively into a 1 000 ml volumetric flask, and dilute to the mark with water. Introduce 100 ml of the solution obtained into a second 1 000 ml flask, and dilute to the mark with water.

3.2.2 Dissolve 0,134 g of potassium sorbate, $\text{CH}_3(\text{CH}:\text{CH})_2\text{COOK}$, (previously recrystallized and dried to constant mass in a drier at 105 °C or in a desiccator over concentrated sulfuric acid) in water in a 1 000 ml volumetric flask, and dilute to the mark with water. Introduce 100 ml of the solution obtained into a second 1 000 ml flask, and dilute to the mark with water.

3.3 Calcium hydroxide $[\text{Ca}(\text{OH})_2]$ (if necessary), about 0,04 N (1,48 g/l) solution.

3.4 Acetic acid (CH_3COOH) , 0,1 N solution.

3.5 Lactic acid $[\text{CH}_3\text{CH}(\text{OH})\text{COOH}]$, 1 N solution.

3.6 Copper, catalyst solution, for technique A.

In a 1 000 ml volumetric flask, dissolve, in a little water, 0,5 g of sodium hydrogen carbonate (NaHCO_3), and 0,001 g of pure copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$). Dilute to the mark with water.

3.7 Chromic/sulfuric acid solution, for technique B.

Dissolve 0,050 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in approximately 90 ml of water. Transfer quantitatively into a 200 ml volumetric flask. Add 100 ml of a 0,3 N sulfuric acid (H_2SO_4) solution. Dilute to the mark with water.

1 litre of 0,3 N sulfuric acid solution contains 14,7 g of sulfuric acid, i.e. 8,4 ml of sulfuric acid, $\rho_{20} = 1,84 \text{ g/ml}$.

3.8 Thiobarbituric acid solution, for technique B.

Dissolve 0,500 g of thiobarbituric acid ($C_4H_4N_2O_2S$) in 50 ml of water to which has been added 10 ml of a 1 N sodium hydroxide (NaOH) solution. Transfer quantitatively into a 100 ml volumetric flask, and add 11 ml of a 1 N hydrochloric acid (HCl) solution. Dilute to the mark with water.

This solution is not stable and must be used within 5 h following its preparation.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Analytical balance.

4.2 Homogenizer or mortar, as appropriate.

4.3 Boiling water bath.

4.4 Steam distillation apparatus (see Figure A.1), comprising the items listed in 4.4.1 to 4.4.5.

4.4.1 Steam generator flask, of capacity 1 000 ml to 1 500 ml.

4.4.2 Bubbler, consisting of a cylindrical tube 30 mm in diameter and 270 mm in height, the lower part of which is closed and enlarged into a sphere having a diameter of 60 mm. The steam supply tube shall end 10 mm above the bottom of the bubbler. The spherical part in which the product is placed, may be heated either electrically or by a flame. In the latter case, the burnt gases shall be deflected by a metal disc of 150 mm diameter, having a central orifice of approximately 40 mm diameter in which the bottom of the bubbler is engaged. This device avoids the pyrogenation of materials which may be extracted from the product. The auxiliary heating shall be controlled so that the volume of the product placed in the bubbler neither decreases nor increases by more than 5 ml during the distillation.

4.4.3 Fractionating column, through which the vapour containing volatile acid passes. It may consist of

- a cylindrical tube of diameter 20 mm and height 500 mm, containing a corrugated helix of No. 100 stainless steel mesh, the helix having a pitch of 15 mm, or
- a column of diameter 20 mm and height 600 mm, having glass internal points, or
- any other device having the same fractionating efficiency.

The fractionation of the vapour is indispensable to retain hydroxymethylfurfural when it is present. This substance and its hydrolysis products absorb ultraviolet radiations at 256 nm. The fractionating column may be reduced to 200 mm in height or be replaced by a Kjeldahl flask when the product is free of hydroxymethylfurfural.

4.4.4 Condenser, of the West type, of effective length 400 mm, placed vertically to ensure condensation of the vapour and complete cooling of the distillate.

4.4.5 Receiver flasks, of capacities 200 ml or 500 ml:

- liquid products: 200 ml flask with a graduation mark at 200 ml;
- thick or solid products: 500 ml flask.

4.4.6 Checking of efficiency of steam distillation apparatus.

The distillation apparatus shall allow 300 ml of distillate to be collected in 12 min to 15 min, and shall also comply with the following minimal conditions.

- a) In normal distilling conditions, 99,5 % of a known quantity of acetic acid added to the sample shall be found in the distillate, which shall be 200 ml. For this test, use 20 ml of a 0,1 N acetic acid solution (3.4).
- b) In the same distilling conditions, not more than 5 parts per thousand of a known quantity of lactic acid added to the sample shall be found in the distillate, which shall be 200 ml. For this test, use 20 ml of 1 N lactic acid solution (3.5).

4.5 Pipettes, of capacities 10 ml, 20 ml and 25 ml.

4.6 Graduated pipettes, of appropriate capacities.

4.7 Evaporating dish.

4.8 Conical flasks, for technique A, of capacity 50 ml.

4.9 Spectrophotometer, for technique A, allowing measurements at a wavelength of 256 nm (ultraviolet), with silica cells of 10 mm optical path length.

4.10 Volumetric flasks, for technique B, of capacity 25 ml.

4.11 Photocolorimeter, fitted with a green filter, or **spectrophotometer** allowing measurements at a wavelength of 532 nm, for technique B.

5 Sample

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

6 Procedure

6.1 Preparation of test sample

6.1.1 Liquid products (juices, pulpy fluid products, syrups, etc.)

Thoroughly mix the laboratory sample.

6.1.2 Thick products (marmalades, jams, etc.)

Homogenize the laboratory sample after having carefully mixed it.

6.1.3 Solid products (fruits, vegetables)

Cut a part of the laboratory sample into small pieces, and remove seeds, stalks and carpellary cells, if necessary, and carefully homogenize approximately 40 g of the sample.

6.1.4 Frozen or deep-frozen products

After thawing the sample in a closed container and removing, if necessary, seeds, stalks and carpellary cells, mix the product with the liquid formed during the thawing process and proceed as described in 6.1.1, 6.1.2 or 6.1.3, as appropriate.