INTERNATIONAL STANDARD

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Second edition
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Fruit and vegetable products — Determination of tin content

Produits dérivés des fruits et légumes — Détermination de la teneur en étain

Reference number
ISO 2447:1998(E)

Foreword

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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.

International Standard ISO 2447 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 3, *Fruit and vegetable products*.

This second edition cancels and replaces the first edition (ISO 2447:1974), which has been technically revised.

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Fruit and vegetable products — Determination of tin content

1 Scope

This International Standard specifies a method for the determination of the tin content in fruit and vegetable products.

The method is applicable to products which may contain, per kilogram, up to:

— 1,25 g of copper;

— 0,6 g of lead;

— 6 g of zinc;

— 40 g of phosphorus.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5515:1979, *Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Wet method.*

3 Principle

After destruction of the organic matter by means of sulfuric and nitric acids, and conversion of the tin to the stannic form, a complex is formed in a buffered solution of pH 1,0 to pH 1,2 [the iron(III) being masked, if necessary, by reduction with ascorbic acid]. The complex is coloured orange with phenylfluorone and the colour compared with those obtained under the same conditions but starting from standard solutions of pure tin.

4 Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water or water of equivalent purity.

4.1 Sulfuric acid, ρ_{20} = 1,84 g/ml.

4.2 Dilute sulfuric acid, 1,25 mol/l.

4.3 Ascorbic acid, 50 g/l solution.

4.4 Nitric acid, ρ_{20} = 1,42 g/ml.

4.5 Hydrochloric acid, ρ_{20} = 1,19 g/ml.

4.6 Methanol.

4.7 Ethanol, 95 % (*V/V*).

4.8 Poly(vinyl alcohol), 16 g/l solution.

Dissolve 1,6 g of poly(vinyl alcohol) in a little water with gentle warming and agitation. Dilute to 100 ml after cooling.

4.9 Buffer solution, containing 450 g of sodium acetate (CH_3COONa) and 240 ml of acetic acid (CH_3COOH) per litre.

4.10 Tin, standard volumetric solution I, containing 500 µg/ml in a sulfuric acid medium, approximately 3 mol/l.

Dissolve, with heating, 0,5 g of pure tin in a mixture of 50 ml of sulfuric acid (4.1), 5 ml of nitric acid (4.4) and 25 ml of water. After complete solution, oxidize the tin to the stannic form by boiling until white fumes appear.

Cool the solution and pour it into a 1 000 ml volumetric flask containing 116 ml of sulfuric acid (4.1) and 100 ml of water. Cool and dilute to 1 000 ml with water.

NOTE Alternatively, the tin standard solution I (4.10) may be prepared by dilution of a suitable proprietary standard solution.

4.11 Tin, standard volumetric solution II, containing 10 µg/ml in a sulfuric acid medium, approximately 0,25 mol/l.

Transfer 20 ml of the tin standard volumetric solution I (4.10) to a 1 000 ml volumetric flask. Add 10 ml of sulfuric acid (4.1) and dilute to 1 000 ml with water.

4.12 Phenylfluorone reagent (2,6,7-trihydroxy-9-phenyl-3-isoxathone).

Dissolve 0,1 g of phenylfluorone in 10 ml of methanol (4.6) and 1 ml of hydrochloric acid (4.5) in a 500 ml volumetric flask. Dilute to the mark with ethanol (4.7).

The reagent shall be stored in a brown bottle in the dark. It is recommended that it should not be stored for longer than 1 week.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 One-mark volumetric flasks, of capacity 50 ml, 200 ml and 500 ml.

5.2 Pipettes, for delivering 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 10 ml and 20 ml.

5.3 Spectrophotometer or **photocolorimeter**, with green filter, fitted with a cell of 10 mm light path, enabling measurements to be made at wavelengths from 500 nm to 530 nm.

5.4 Analytical balance, capable of weighing to the nearest 0,001 g.

6 Sampling

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

Sampling is not part of the method specified in this International Standard. As there is no specific International Standard dealing with fruit and vegetable products, it is recommended that the parties concerned come to an agreement on the subject.

7 Procedure

7.1 Preparation of test portion

Proceed as described in ISO 5515, preferably weighing a mass of about 10 g to the nearest 0,01 g.

7.2 Destruction of organic matter

Proceed as described in ISO 5515.

Add 5 ml of sulfuric acid (4.1) to the resulting solution. Cool and pour it into a 200 ml volumetric flask (5.1), and dilute to the mark with water (solution A).

7.3 Determination

7.3.1 By means of a pipette (5.2), transfer to a 50 ml volumetric flask (5.1) an appropriate volume of solution A, i.e.

— 20 ml, if the tin content of the sample is below 50 mg/kg;

— 10 ml, if the tin content of the sample is between 50 mg/kg and 125 mg/kg, diluting to 20 ml with dilute sulfuric acid (4.2);

— 5 ml, if the tin content of the sample is above 125 mg/kg, diluting to 20 ml with dilute sulfuric acid (4.2).

7.3.2 Then add in succession:

— 10 ml of buffer solution (4.9);

— 1 ml of ascorbic acid solution (4.3);[1)]

— 5 ml of poly(vinyl alcohol) solution (4.8);

— 5 ml of phenylfluorone reagent (4.12).

Swirl the flask, avoiding foam formation from the poly(vinyl alcohol). Leave to stand for 5 min.

Dilute to the mark with water and leave to stand for 30 min. Then carry out the measurement at a wavelength of 505 nm in the spectrophotometer or photocolorimeter (5.3).

7.3.3 Carry out two determinations on the same prepared sample taken for the destruction of organic matter (solution A) (see 7.2).

7.4 Preparation of calibration curve

7.4.1 Into a series of six 50 ml volumetric flasks (5.1), each containing 20 ml of dilute sulfuric acid (4.2), add the following volumes of standard volumetric tin solution II (4.11):

— 0 ml, equivalent to 0 µg of tin

— 1 ml, equivalent to 10 µg of tin

— 2 ml, equivalent to 20 µg of tin

1) The addition of ascorbic acid is not necessary if the content of iron(III) is equal to or less than 25 mg/kg.

— 3 ml, equivalent to 30 µg of tin

— 4 ml, equivalent to 40 µg of tin

— 5 ml, equivalent to 50 µg of tin

7.4.2 Then proceed as indicated in 7.3.2.

7.4.3 Prepare the calibration curve, showing the difference of optical density as a function of the number of micrograms of tin.

8 Expression of results

By means of the calibration curve, convert the figure obtained in 7.3.2 into micrograms of tin.

The tin content, expressed in milligrams per kilogram of product, is equal to

$$\frac{m_1}{1000} \times \frac{200}{V} \times \frac{1000}{m_0} = \frac{m_1 \times 200}{V \times m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass of tin, in micrograms, read from the calibration curve;

V is the volume, in millilitres, of solution A taken for colorimetric measurement (see 7.3.1).

Take as the result the arithmetic mean of the results of the two determinations (7.3.3), provided that their difference does not exceed 5 % of the arithmetic mean.

9 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 5 % of the arithmetic mean of the two results.

Reject both results if the difference exceeds 5 % of the arithmetic mean and carry out two new single determinations.

10 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, together with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained;

— if the repeatability has been checked, the final quoted result obtained.

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