INTERNATIONAL STANDARD 2447

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Fruit and vegetable products — Determination of tin

Produits dérivés des fruits et légumes — Dosage de l'étain

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2447 was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, and circulated to the Member Bodies in July 1971.

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It has been approved by the Member Bodies of the following countries :

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Australia	Hungary	Portugal
Brazil	India	South Africa, Rep. of
Bulgaria	Iran	Spain
Chile	Israel	Thailand
Egypt, Arab Rep. of	Korea, Dem. P. Rep. of	Turkey
Finland	Netherlands	United Kingdom
France	Poland	

This International Standard has also been approved by the Association of Official Analytical Chemists (AOAC).

The Member Body of the following country expressed disapproval of the document on technical grounds :

Czechoslovakia

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Fruit and vegetable products — Determination of tin

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of tin 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;
- 0,6 g of zinc;
- 40 g of phosphorus.

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2 REFERENCE

ISO . . ., *Fruit and vegetable products — Wet method for the destruction of organic matter.* [1)]

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3 PRINCIPLE

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

4 REAGENTS

All reagents used shall be of analytical reagent quality and the water used shall be distilled water or water of at least equivalent purity.

The following reagents are necessary, in addition to those used for the destruction of organic matter and specified in ISO . . . :

4.1 Sulphuric acid, ρ_{20} 1,84 g/ml.

4.2 Sulphuric acid, 2,5 N solution.

4.3 Ascorbic acid, 50 g/l solution.

4.4 Polyvinyl alcohol, 16 g/l solution.

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

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

4.6 Tin, standard volumetric solution, containing 500 µg/ml in a sulphuric acid medium, approximately 6 N.

Dissolve, with heating, 0,5 g of pure tin in a mixture of 50 ml of sulphuric acid (4.1), 5 ml of nitric acid, ρ_{20} 1,42 g/ml, 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 sulphuric acid (4.1) and 100 ml of water. Cool and dilute to 1 000 ml with water.

4.7 Tin, standard volumetric solution containing 10 µg/ml in a sulphuric acid medium, approximately 0,5 N.

Transfer 20 ml of the tin standard volumetric solution (4.6) to a 1 000 ml volumetric flask. Add 10 ml of sulphuric acid (4.1) and dilute to 1 000 ml with water.

4.8 Phenylfluorone reagent (2, 6, 7-trihydroxy-9-phenyl-3-isoxanthone).

Dissolve 0,1 g of phenylfluorone in 10 ml of methanol and 1 ml of concentrated hydrochloric acid, ρ_{20} 1,19 g/ml, in a 500 ml volumetric flask. Dilute to the mark with 95 % (*V*/*V*) ethanol. 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 equipment not otherwise specified, in addition to that used for the destruction of organic matter and specified in ISO . . . , and in particular :

5.1 One-mark volumetric flasks, capacity 50 and 200 ml, complying with class A of ISO/R 1042.

5.2 Pipettes for delivering 1, 2, 3, 4, 5, 10 and 20 ml, complying with class A of ISO/R 648 or ISO/R 835.

1) In preparation.

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 to 530 nm.

5.4 Analytical balance.

6 PROCEDURE

6.1 Preparation of test portion

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

6.2 Destruction of organic matter

Proceed as described in ISO . . . .

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

6.3 Colorimetry

6.3.1 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 and 125 mg/kg, diluting to 20 ml with sulphuric acid solution (4.2);
- 5 ml, if the tin content of the sample is above 125 mg/kg, diluting to 20 ml with sulphuric acid solution (4.2).

6.3.2 Then add in succession :

- 10 ml of buffer solution (4.5);
- 1 ml of ascorbic acid solution (4.3);[1]
- 5 ml of polyvinyl alcohol solution (4.4);
- 5 ml of phenylfluorone reagent (4.8).

Swirl the flask, avoiding foam formation from the polyvinyl 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).

6.3.3 Carry out two determinations on the same prepared sample taken for the destruction of organic matter (see 6.1).

6.4 Preparation of calibration curve

6.4.1 Into a series of six 50 ml volumetric flasks (5.1), each containing 20 ml of sulphuric acid solution (4.2), add the following volumes of standard volumetric tin solution (4.7)

0 ml, equivalent to 0 µg of tin
1 ml, equivalent to 10 µg of tin
2 ml, equivalent to 20 µg of tin
3 ml, equivalent to 30 µg of tin
4 ml, equivalent to 40 µg of tin
5 ml, equivalent to 50 µg of tin

6.4.2 Then proceed as indicated in 6.3.2.

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

7 EXPRESSION OF RESULTS

7.1 Method of calculation and formula

By means of the calibration curve, convert the figure obtained in 6.3.2 into micrograms of tin.

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

$$\frac{m_1}{1\,000} \times \frac{200}{V} \times \frac{10^3}{m_0} = \frac{m_1 \times 200}{V \times m_0}$$

where

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

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

V is the volume, in millilitres, of solution A taken for colorimetric measurement (see 6.3.1).

Take as the result the arithmetic mean of the determinations, if the requirement concerning repeatibility (see 7.2) is satisfied.

7.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 5 % of the mean value.

8 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for complete identification of the sample.

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

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