INTERNATIONAL STANDARD

ISO 17240

First edition
2004-10-01

Fruit and vegetable products — Determination of tin content — Method using flame atomic absorption spectrometry

Produits dérivés des fruits et légumes — Détermination de la teneur en étain — Méthode par spectrométrie d'absorption atomique avec flamme

Reference number
ISO 17240:2004(E)

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Published in Switzerland

Foreword

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ISO 17240 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 3, *Fruit and vegetable products*.

Fruit and vegetable products — Determination of tin content — Method using flame atomic absorption spectrometry

1 Scope

This International Standard specifies an atomic absorption spectrometric method for the determination of the tin content of fruit and vegetable products in the concentration range 10 mg/kg to 500 mg/kg. It is a rapid method, especially suitable for routine determinations of tin in canned fruits and vegetables contaminated with tin which has migrated from the can. The method can be applied with the prescribed amount of sample to products with a maximum total dry matter content of 30 %. Products with higher contents of total solids can be analysed using smaller amounts of sample after corresponding dilution with deionized water.

NOTE The method of tin determination in fruit and vegetables products is based on NMKL method No 126/1988 (reference [1] in Annex A).

2 Principle

Fruit and vegetable products are digested in hydrochloric acid at 80 °C and the tin content is determined by flame atomic absorption spectrometry.

3 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and deionized water or water of equivalent purity.

3.1 Hydrochloric acid, concentrated (ρ_{20} = 1,19 g/ml).

3.2 Hydrochloric acid, dilute (c = 6 mol/l).

Dilute 50 ml of hydrochloric acid (3.1) with water to 100 ml.

3.3 Tin, standard solution corresponding to 1,0 mg of tin per millilitre.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Mechanical grinder, the inside and the blades of which are coated with polytetrafluoroethylene (PTFE).

4.2 Block thermostat, or other device for rapid heating and temperature control. The required temperature accuracy is ± 3 °C.

4.3 Atomic absorption spectrometer, provided with a nitrous oxide/acetylene burner (5 cm), suitable for measurements at a wavelength of 235,5 nm.

4.4 Tin lamp, hollow cathode lamp or electrodeless discharge lamp (EDL).

NOTE The detection limit of the method is significantly lower when EDL lamps are used.

4.5 Filter paper, Schleicher and Schull 589/1 (Black band)[1)] or equivalent.

4.6 Analytical balance.

5 Sample

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

6 Procedure

6.1 Preparation of test sample

Thoroughly mix the laboratory sample. If necessary, first remove any seeds, stalks and hard seed-cavity walls, and then grind in the mechanical grinder (4.1).

Frozen or deep-frozen products shall be previously thawed in a closed vessel, and the liquid formed during this process shall be added to the product before mixing.

6.2 Test portion

Weigh, to the nearest 0,001 g, 5 g of the test sample (6.1) into a glass tube suitable for the block thermostat (4.2) or directly into a 50 ml volumetric flask.

6.3 Decomposition

Add 10 ml of dilute hydrochloric acid (3.2). Incubate the sample for 60 min in a block thermostat or in a water bath preheated to 80 °C ± 3 °C. Stir the mixture three or four times during the heating period.

Transfer the sample quantitatively to a 50 ml volumetric flask and, after cooling, dilute to volume with water. Filter the sample through filter paper (4.5). The filtrate is now ready for measurement by atomic absorption spectrometry. Perform the determination preferably within 5 h to 6 h, or alternatively store the extract in a sealed plastic flask.

6.4 Blank test

Carry out a blank test by transferring 10 ml of dilute hydrochloric acid (3.2) into a glass tube or a volumetric flask and use the same decomposition procedure (6.3) as for the test portion (6.2).

6.5 Determination

6.5.1 Preparation of the calibration graph

Dilute the standard tin solution (3.3) adding 10 ml of concentrated hydrochloric acid (3.1) per 100 ml, to obtain an appropriate number of standard solutions in the range 3,0 mg/l to 200 mg/l, when using a hollow cathode lamp, and in the range 1,0 mg/l to 200 mg/l, when using an EDL lamp.

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1) Schleicher and Schull 589/ (Black Band) is an example of a suitable product available commercially. This information is only given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Ignite the nitrous oxide/acethylene flame of the atomic absorption spectrometer (4.3) according to the instrument instructions and adjust the gas flow to give a 2 cm red band above the burner head.

Aspirate each of these solutions, in turn, into the flame of the spectrometer (4.3). Use a mixture of dilute hydrochloric acid with water (1 + 9) as a blank.

Record the corresponding values of absorbance and plot the calibration graph (absorbance against tin concentration in milligrams per litre).

6.5.2 Spectrometric measurement

Set the instrument to the previously established optimum conditions, using the nitrous oxide/acethylene flame and a 235,5 nm resonant wavelength.

Aspirate the test solution (6.3) and the blank solution (6.4) into the flame of the spectrometer (4.3). Record the corresponding absorbances.

7 Calculation

The tin content of the sample, *w*, expressed in milligrams per kilogram of the product, is given by the equation:

$$w = \frac{(\rho - \rho_b) \times 50}{m}$$

where

ρ is the tin concentration of the test solution, expressed in milligrams per litre, read from the calibration graph;

ρ_b is the tin concentration of the blank test solution, expressed in milligrams per litre, read from the calibration graph;

m is the mass, in grams, of the test portion.

8 Precision

8.1 General

The precision of the method has been checked by collaborative studies (13 laboratories) of tin determinations in two samples of tomato soup and two samples of apple sauce (Reference [1] in Annex A).

Statistical parameters are expressed according to ISO 5725-2 (Reference [2] in Annex A).

8.2 Repeatability

8.2.1 Tomato soup

Sample 1: the repeatability coefficient of variation is 4,3 %.

Sample 2: the repeatability coefficient of variation is 2,4 %.

8.2.2 Apple sauce

Sample 1: the repeatability coefficient of variation is 1,078.

Sample 2: the repeatability coefficient of variation is 0,406.

NOTE The coefficient is dependent on the content of tin in the sample, and decreases with increasing tin content.

8.3 Reproducibility

8.3.1 Tomato soup

Sample 1: the reproducibility coefficient of variation is 12,0 %.

Sample 2: the reproducibility coefficient of variation is 7,1 %.

8.3.2 Apple sauce

Sample 1: the reproducibility coefficient of variation is 9,4 %.

Sample 2: the reproducibility coefficient of variation is 4,0 %.

NOTE The coefficient is dependent on the content of tin in the sample, and decreases with increasing tin content.

9 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used;

c) the test method used, with reference to this International Standard;

d) 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);

e) the test result(s) obtained, or, if the repeatability has been checked, the final quoted result obtained.

Bibliography

[1] Nordic Committee on Food Analysis. No 126/1988. *Tin. Determination by atomic absorption in fruits and vegetables*

[2] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ICS 67.080.01

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