SLOVENSKI STANDARD
SIST-TS CEN/TS 15506:2007

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Živila - Določevanje elementov v sledovih - Določevanje kositra v sadju in zelenjavi v konzervah z uporabo spektrometrije s plamensko atomsko absorpcijo (AAS)

Foodstuffs - Determination of trace elements - Determination of tin in fruit and vegetables preserved in cans by flame atomic absorption spectrometry (AAS)

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Zinn in Obst- und Gemüsekonserven mit Flammen-Atomabsorptionsspektrometrie (AAS)

Produits alimentaires - Dosage des éléments traces - Détermination de l'étain dans les fruits et légumes en boîtes de conserve par spectrométrie d'absorption atomique flamme (SAA)

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TECHNICAL SPECIFICATION

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English Version

Foodstuffs - Determination of trace elements - Determination of tin in fruit and vegetables preserved in cans by flame atomic absorption spectrometry (AAS)

Produits alimentaires - Dosage des éléments traces - Détermination de l'étain dans les fruits et légumes en boîtes de conserve par spectrométrie d'absorption atomique flamme (SAA)

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Zinn in Obst- und Gemüsekonserven mit Flammen-Atomabsorptionsspektrometrie (AAS)

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The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (CEN/TS 15506:2007) has been prepared by Technical Committee CEN/TC 275 “Food analysis — Horizontal methods”, the secretariat of which is held by DIN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This document specifies a method for the determination of tin in vegetable foods preserved in cans by flame atomic absorption spectrometry (AAS).

This method is applicable to the determination of extractable tin in fruits and vegetables and collaboratively tested in concentrations from 25 mg/kg to 350 mg/kg. It is a method for determination of tin in canned fruit and vegetables contaminated with migrated tin from the can. The method can be applied with the prescribed amount of sample to products with a maximum total dry matter of 30 %. Products with higher total solid contents may be analysed using sample amounts less than 30 % after corresponding dilution with deionised water.

2 Normative references

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

EN 13804, *Foodstuffs – Determination of trace elements – Performance criteria, general considerations and sample preparation*

3 Principle

Canned fruit and vegetables are extracted with hydrochloric acid at 80 °C and the tin content is determined by flame atomic absorption spectrometry.

WARNING — Use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use, thus it is the responsibility of the user to establish appropriate safety and health practices and determine the applicability or regulatory limitations prior to use.

4 Reagents

4.1 General

The concentration of tin in the reagents and water used shall be low enough not to affect the results of determination.

4.2 Hydrochloric acid

Mass fraction w ≥ 30 %, with a mass concentration of approximately ρ(HCl) = 1,15 g/ml.

4.3 **Hydrochloric acid**, substance concentration c = 6 mol/l

Dilute 50 ml of hydrochloric acid (4.2) with water to 100 ml.

4.4 **Tin stock standard solution**, in hydrochloric acid (4.3), mass concentration ρ(Sn) = 1 000 mg/l.

The use of a certified stock solution is advisable.

4.5 Calibration solutions

Prepare an appropriate number of standard solutions with mass concentrations of Sn in the range of 3 mg/l to 200 mg/l. When analysing fruit and vegetables there is normally no need for a concentration higher than ρ(Sn) = 50 mg/l, corresponding to approximately 500 mg Sn per kilogram product. All standard solutions shall contain 100 ml of concentrated hydrochloric acid (4.2) per litre.

5 Apparatus and equipment

5.1 Block thermostat, or other device for rapid heating and temperature control, e.g. water bath.

The required temperature accuracy is ± 3 °C.

5.2 Atomic absorption spectrometer, with nitrous oxide/acetylene flame.

5.3 Hollow cathode lamp, or electrodeless discharge lamp (EDL) for tin.

5.4 Filter paper, resistant to hydrochloric acid.

6 Sampling

To prevent any increase in the tin content between opening of the tin can and analysis, the sample shall be immediately transferred to a glass or plastic container.

7 Procedure

7.1 Sample preparation

Homogenise the sample in accordance with the recommendations in EN 13804.

Weigh 5 g of homogenized sample into a glass tube suitable for the block thermostat (5.1) or directly into a 50 ml volumetric flask. Add 10 ml of hydrochloric acid (4.3). Incubate the sample for 60 min in a block thermostat or in a water bath preheated to 80 °C ± 3 °C. Stir the mixture 3 to 4 times during the heating period.

Transfer the sample quantitatively to a 50 ml volumetric flask and after cooling, make up to volume with water. Filter the sample through filter paper (5.4). The filtrate is ready for measuring by flame atomic absorption spectrometry. Perform the determination preferably within 5 h to 6 h, or alternatively store the extract in a sealed plastic flask.

7.2 Reagent blank

Prepare a reagent blank analogously to the sample by transferring 10 ml hydrochloric acid (4.3) into a glass tube or a volumetric flask and following the same procedure as for the sample.

7.3 Determination of the tin concentration

Ignite the nitrous oxide/acetylene flame (5.2) according to the instrument instructions and adjust the gas flow to give a reducing flame with an approximately 2 cm pink/red band above the burner head. Adjust the wavelength to 235,5 nm. Zero the instrument using a mixture of hydrochloric acid (4.2) and water (1+4; *V/V*).

Determine the tin content in the reagent blank, the standard solutions and the sample solution.

Construct a calibration curve by plotting the mass concentration of tin in the standard solutions in milligram per litre against the peak height. Use the calibration curve to determine the concentration of tin in the sample solution and in the reagent blank.

8 Evaluation and calculation

Calculate the tin content, w, as mass fraction of tin in mg/kg of the sample, using equation (1):

$$w = \frac{a \cdot V}{m} \qquad (1)$$

Where

a is the mass concentration of tin in the sample solution, in milligram per litre;

V is the volume of the sample solution, in millilitre;

m is the initial sample mass, in gram.

If necessary, subtract the tin content of the blank solution from a.

9 Limit of determination

In the collaborative study the limit of determination was in the order of 5 mg to 10 mg tin per kg product. The detection and quantification limits should be estimated according to EN 13804, taking into account the standard deviation (SD) found in the long term evaluation.

10 Repeatability and reproducibility

Repeatability is the absolute difference between two independent single test results (blind duplicate), obtained with 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 the cases exceed the values of r given in Table 1.

Reproducibility is the absolute difference between two single test results obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of the cases exceed the values of R given in Table 1.

Table 1 — Repeatability and reproducibility

Sample	$\bar{x}$ mg/kg	r mg/kg	R mg/kg
Apple sauce no 1	35,0	8,1	9,6
Tomato soup no 1	55,2	6,7	18,4
Apple sauce no 2	107	8,7	12,1
Tomato soup no 2	326	32	76

11 Test report

The test report shall specify at least the following:

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

b) test method used, with reference to this document;

c) results obtained and the units in which they are specified;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document or regarded as optional, together with details of any incidents occurred when performing the method which might have influenced the test result(s).