



SLOVENSKI STANDARD SIST EN 15765:2010

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Foodstuffs - Determination of trace elements - Determination of tin by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Zinn mit Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) nach Druckaufschluss

Produits alimentaires - Dosage de l'étain par spectrométrie de masse à plasma induit par haute fréquence (ICP-MS) après digestion sous pression

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67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products
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EUROPEAN STANDARD

EN 15765

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Foodstuffs - Determination of trace elements - Determination of tin by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion

Produits alimentaires - Dosage des éléments traces -
Dosage de l'étain par spectrométrie de masse à plasma
induit par haute fréquence (ICP-MS) après digestion sous
pression

Lebensmittel - Bestimmung von Elementspuren -
Bestimmung von Zinn mit Massenspektrometrie mit
induktiv gekoppeltem Plasma (ICP-MS) nach
Druckaufschluss

This European Standard was approved by CEN on 7 November 2009.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents		Page
Foreword.....		3
1	Scope	4
2	Normative references	4
3	Principle	4
4	Reagents	4
5	Apparatus and equipment	5
6	Procedure	6
7	Evaluations	7
8	Precision	7
9	Test report	8
Annex A (informative) Results of the inter-laboratory test		9
Bibliography		11

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Foreword

This document (EN 15765:2009) has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2010, and conflicting national standards shall be withdrawn at the latest by June 2010.

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EN 15765:2009 (E)**1 Scope**

This European Standard specifies a method for the determination of tin in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressurized digestion. The collaborative study included carrot puree, tomato puree, pineapple, mixed fruit, white wine, peach powder, tomato powder, beans powder, powdered fruit yoghurt and fish powder foodstuffs having a mass fraction of tin ranging from 2,5 mg/kg to 259 mg/kg.

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 13805, *Foodstuffs — Determination of trace elements — Pressure digestion*

3 Principle

The sample is mineralized through pressurized digestion with nitric acid and hydrochloric acid in accordance with EN 13805. The digestion solution obtained thereby is diluted and then atomised and ionised in an inductively coupled argon plasma. After extraction from the plasma by a system of sampler and skimmer cones the positive (charged) tin ions are separated according to their mass charge ratio in a mass spectrometer and quantified in a detector system.

4 Reagents

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4.1 General

The concentration of tin in the reagents and water used shall be low enough not to affect the results of the determination. Solution shall be understood as an aqueous solution unless otherwise specified.

4.2 Nitric acid, mass fraction $w(\text{HNO}_3) \geq 65 \%$, mass concentration $\rho(\text{HNO}_3) \approx 1,4 \text{ g/ml}$.

4.3 Hydrochloric acid, $w(\text{HCl}) \geq 30 \%$, density $\rho(\text{HCl}) \approx 1,15 \text{ g/ml}$.

4.4 Stock solutions

4.4.1 Tin stock solution, mass concentration $\rho(\text{Sn}) = 1\,000 \text{ mg/l}$.

4.4.2 Rhodium stock solution (internal standard), mass concentration $\rho(\text{Rh}) = 1\,000 \text{ mg/l}$.

4.5 Standard solutions

4.5.1 Tin standard solution 1, mass concentration $\rho(\text{Sn}) = 50 \text{ mg/l}$

Fill a 50 ml volumetric flask with 10 ml to 20 ml of water, add 2,5 ml of hydrochloric acid (4.3) and mix. Cool to ambient temperature, and add by means of a pipette exactly 2,5 ml of tin stock solution (4.4.1) and dilute to volume with water. This solution is stable for at least one week.

4.5.2 Tin standard solution 2, mass concentration $\rho(\text{Sn}) = 1,0 \text{ mg/l}$

Fill a 50 ml volumetric flask with 10 ml to 20 ml of water, add 2,5 ml of hydrochloric acid (4.3) and mix. Cool to ambient temperature, and add by means of a pipette exactly 1,0 ml of tin standard solution 1 (4.5.1) and dilute to volume with water. This solution is stable for one week.

4.5.3 Rhodium standard solution, mass concentration $\rho(\text{Rh}) = 10 \text{ mg/l}$

Fill a 50 ml volumetric flask with 10 ml to 20 ml of water, add 2,5 ml of hydrochloric acid (4.3) and mix. Cool to ambient temperature, and add by means of a pipette exactly 0,5 ml of rhodium stock solution (4.4.2) and dilute to volume with water. This solution is stable for at least four weeks.

4.6 Calibration solutions

The following concentrations of the calibration solutions are examples and may be changed according to the sensitivity of measuring instrument and the concentration range to be investigated. Care shall be taken that calibration is carried out within the linear range of the detector system while paying attention to the varying frequency of isotopes. The quantity of internal standard added should be sufficient enough to obtain a stable and reproducible intensity. For calibration at least three calibration solutions of different concentration shall be used. The acid concentration should correspond to the one in the measuring solution.

The preparation of the following solutions is given as an example:

Calibration solutions with mass concentrations of 5 $\mu\text{g/l}$, 10 $\mu\text{g/l}$, 20 $\mu\text{g/l}$ and 40 $\mu\text{g/l}$.

The calibration solutions are prepared from the tin standard solution 2 (4.5.2) according to following procedure:

Fill four 50 ml volumetric flasks with 10 ml to 20 ml of water, add 0,5 ml of nitric acid (4.2) and 0,1 ml of hydrochloric acid (4.3) and mix. Cool to ambient temperature, and pipette exactly the same amount of internal standard, e.g. 0,5 ml of rhodium standard solution (4.5.3), into each of the measuring flasks. Then, for the calibration solutions of mass concentrations of 5 $\mu\text{g/l}$, 10 $\mu\text{g/l}$, 20 $\mu\text{g/l}$ and 40 $\mu\text{g/l}$, pipette exactly 0,25 ml, 0,50 ml, 1,0 ml and 2,0 ml of the standard solution 2 (4.5.2), respectively, into the four separate 50 ml volumetric flasks and make up to volume with water. These solutions shall be freshly prepared on each day of measurement.

The calibration solutions described here shall be understood as examples. The concentrations prepared shall be in the linear range of the ICP-MS detector system. Furthermore, the acid concentration of the calibration solutions shall be matched to the amounts of acid being present in the diluted digestion solution.

4.7 Blank solution (blank solution)

The blank solution contains water, nitric acid and hydrochloric acid in amounts that correspond to the concentrations in the measurement solution, for example 0,5 ml of nitric acid (4.2) and 0,1 ml of hydrochloric acid (4.3) in 50 ml as well as the same amount of internal standard (0,5 ml rhodium standard solution (4.5.3)) as used for the calibration solutions in 50 ml.

5 Apparatus and equipment

5.1 General

All apparatus and equipment that come into direct contact with the sample and solutions shall be pre-cleaned appropriately.

EN 15765:2009 (E)

5.2 ICP-MS instrument with inductively coupled argon plasma as ionisation unit, sample feeding and nebulising system as well as an instrument controlling unit and an evaluation unit.

6 Procedure**6.1 Digestion of the sample**

Mineralize the sample by pressurized digestion in accordance with EN 13805. For the quantification of tin, add 0,5 ml to 1 ml of hydrochloric acid (4.3) to the digestion vessel, which contains nitric acid (4.2) used for digestion, at an amount that corresponds to the amount of nitric acid. Do not add the hydrochloric acid until the spontaneous reaction with nitric acid has subsided. After addition of the hydrochloric acid close the digestion vessel immediately in order to avoid loss of active chlorine. Start pressurized digestion shortly thereafter. The digestion requirements are based on the specifications of the instrument manufacturer, the reactivity of the sample, the maximum pressure stability of the digestion vessel and the attainable temperature.

EXAMPLE 1 Precisely weigh 0,4 g to 0,5 g of dry sample (residual moisture of less than 20 %) into a 100 ml digestion vessel and add 5 ml of nitric acid (4.2). Carefully shake the digestion vessel to prevent clots from forming in the sample. After the spontaneous reaction has subsided, add 1 ml of hydrochloric acid (4.3) and close the digestion vessel as quickly as possible. For samples with a higher degree of moisture content, the weighed quantities can be increased.

The digestion solution that results from the pressurized digestion according to EN 13805 is made up to a defined volume, e.g. 20 ml, by water. This solution is diluted, using water, by a factor of 10 or higher for the subsequent determination of tin. It is important that the amount of internal standard is exactly the same, both in the measurement solutions and in the calibration solutions.

EXAMPLE 2 For preparation of the measurement solution directly in the vessel used for measurement, pipette exactly 1,0 ml digestion solution and 0,5 ml rhodium standard solution (4.5.3) into the measurement vessel and make up with water to 10 ml. This solution should be measured on the day of preparation.

6.2 Inductively coupled plasma mass spectrometry**6.2.1 ICP-MS operating conditions**

Set the instrument according to the manufacturer's specifications and ignite the plasma. Following sufficient warming up and stabilization of the instrument, optimise the settings.

6.2.2 Determination by ICP-MS

Once the instrument is optimised start the measurements. For evaluation tin isotopes of the masses 117 and 118 shall be used. The internal standard rhodium is analysed for the mass 103. Correction with internal standard is implemented by the method settings of the instrument software.

Measure the blank solution (4.7) and calibration solutions (4.6) and use the counting rate (counts/s) and concentrations to generate a calibration curve. Determine the linear range of the calibration function.

The measurement solution is aspirated and measured. Use the calibration curve to convert the determined counting rate into concentration units.

6.3 Quality control

For quality control, analyse a reference material with reliably known content of tin in parallel to each series of measurements. Include all procedural steps starting at the digestion. This also applies to each series of digestions including all procedural steps to prepare and measure blank solutions.

7 Evaluations

7.1 Calculation of the tin content in foodstuffs

Calculate the tin content, w , in milligrams per kilogram of sample or ϕ , in milligrams per litre of sample according to Equation (1):

$$w \text{ or } \phi = \frac{a \times V \times 1\,000 \times F}{E \times 1\,000 \times 1\,000} \quad (1)$$

where

a is the mass concentration of tin of the measurement solution, in micrograms per litre ($\mu\text{g/l}$);

V is the volume of the digestion solution diluted to volume, in millilitres (ml);

E is the initial sample mass, in grams (g), or the initial sample volume, in millilitres (ml);

F is the dilution factor (≥ 10).

7.2 Limit of quantification

The ICP-MS instrument should be able to quantify $1 \mu\text{g/l}$ tin in the diluted digestion solution. When using ICP-MS, the limit of quantification in the digestion solution prepared according to EN 13805 is mainly affected by the matrix content.

Regarding the foodstuff, the limit of quantification depends on the amount of sample used for digestion, on the final volume of digestion solution and on the minimum dilution applied (in this case 10). For trace elements the limit of quantification is conventionally defined as 6σ , where σ is the standard deviation of the field blank signal. Table 1 lists relevant examples.

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Table 1 — Examples of quantification limits in the sample

Weighed quantity of sample g	Final volume ml	Dilution	Mass fraction of tin mg/kg
0,5	20	1:10	0,4
2,0	20	1:10	0,1

8 Precision

8.1 General

Details of an inter-laboratory test are summarised in Annex A. The values derived from this inter-laboratory test may not be applicable to concentration ranges and matrices other than those given in Annex A.

8.2 Repeatability

The absolute difference between two independent single test results obtained with the same test method on identical test material in the same laboratory by the same operator using the same apparatus within a short time interval will exceed the repeatability limit r given in Table 2 in not more than 5 % of the cases.

8.3 Reproducibility

The absolute difference between two single test results obtained with the same test method on identical test material in different laboratories by different operators using different equipment will exceed the reproducibility