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Prehrana - Določanje slednih elementov - Določanje cinka s plamensko in grafitno pečnico atomsko absorpcijsko spektrometrijo (FAAS in GFAAS) po tlakni prečiščitvi

Foodstuffs - Determination of trace elements - Determination of zinc by flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS) after pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Zinn mit Flammen- und Graphitofen- Atomabsorptionsspektrometrie (FAAS und GFAAS) nach Druckaufschluss

Produits alimentaires - Dosage de l'étain par spectrométrie d'absorption atomique flamme (SAA) et spectrométrie d'absorption atomique à four graphite (GFAAS) après digestion sous pression

**Ta slovenski standard je istoveten z: EN 15764:2009**

**ICS:**

67.050	Spolšne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products
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**SIST EN 15764:2010****en,fr,de**

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EUROPEAN STANDARD

EN 15764

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EUROPÄISCHE NORM

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ICS 67.050

English Version

## Foodstuffs - Determination of trace elements - Determination of tin by flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS) after pressure digestion

Produits alimentaires - Dosage des éléments traces - Dosage de l'étain par spectrométrie d'absorption atomique flamme (SAAF) et spectrométrie d'absorption atomique à four graphite (SAAF) après digestion sous pression

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Zinn mit der Flammen- und Graphitofen-Atomabsorptionsspektrometrie (FAAS und GFAAS) nach Druckaufschluss

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

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## Foreword

This document (EN 15764:2009) 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of an identical text or by endorsement, at the latest by June 2010, and conflicting national standards shall be withdrawn at the latest by June 2010.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 15764:2009 (E)****1 Scope**

This European Standard specifies a method for the determination of tin in foodstuffs and canned foods by flame and graphite furnace atomic absorption spectrometry (AAS) after pressurized digestion.

The collaborative study included foodstuffs such as carrot puree, tomato puree, pineapple, mixed fruit, white wine, peach powder, tomato powder, powdered beans, powdered fruit yoghurt, fish powder, having mass fractions of tin ranging from 43 mg/kg to 260 mg/kg (Flame-AAS) and from 2,5 mg/kg to 269 mg/kg (Graphite Furnace AAS).

**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. In the resulting digestion solution, tin is quantified by flame AAS (F-AAS) or graphite furnace AAS (GF-AAS) depending on the concentration in the sample solution.

**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.

**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**

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

**4.4 Tin stock solution**

$\rho(\text{Sn}) = 1\,000 \text{ mg/l}$ .

**4.5 Tin standard and calibration solutions****4.5.1 General**

The standard and calibration solutions are prepared from the stock solution by dilution in glass volumetric flasks. For calibration, prepare at least four calibration solutions of different concentrations. The acid concentration shall correspond to the concentration in the measurement solution.

The preparation of the solutions in 4.5.2 and 4.5.3 is given as an example.

#### 4.5.2 Calibration solutions

of  $\rho(\text{Sn}) = 5 \text{ mg/l}$ ,  $10 \text{ mg/l}$ ,  $20 \text{ mg/l}$  and  $30 \text{ mg/l}$  for flame AAS.

Fill four 50 ml volumetric flasks with 10 ml to 20 ml of water, add 5 ml of nitric acid (4.2) and 1 ml of hydrochloric acid (4.3) and mix. Cool the solutions to ambient temperature, and pipette exactly 0,25 ml, 0,50 ml, 1,00 ml and 1,50 ml of tin stock solution (4.4) for the respective calibration solutions of mass concentrations 5 mg/l, 10 mg/l, 20 mg/l and 30 mg/l into the four different 50 ml volumetric flasks. Mix the solutions and dilute to volume with water. These solutions are stable for at least one day.

The calibration solutions described here shall be understood as examples. The concentrations prepared shall be in the linear range of the measuring device. The acid concentration of the calibration solutions shall be matched to the acid concentration in the sample solution.

#### 4.5.3 Calibration solutions

of  $\rho(\text{Sn}) = 0,010 \text{ mg/l}$ ,  $0,020 \text{ mg/l}$ ,  $0,040 \text{ mg/l}$  and  $0,060 \text{ mg/l}$  for GF-AAS.

To prepare standard solution 1 ( $\rho(\text{Sn}) = 50 \text{ mg/l}$ ), 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 the solution to ambient temperature, add exactly 2,5 ml of tin stock solution (4.4) and dilute to volume. This solution is stable for at least one week.

To prepare standard solution 2 ( $\rho(\text{Sn}) = 1,0 \text{ mg/l}$ ), 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 the solution to ambient temperature, add exactly 1,0 ml of tin standard solution 1 by pipette and dilute to volume.

Prepare the calibration solutions for the graphite furnace AAS from standard solution 2 according to the following procedure:

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Fill four 50 ml volumetric flasks with 10 ml to 20 ml of water, add 5 ml of nitric acid (4.2) and 1 ml of hydrochloric acid (4.3) and mix. Cool the solutions to ambient temperature, pipette exactly 0,50 ml, 1,0 ml, 2,0 ml and 3,0 ml of standard solution 2 for the respective calibration solutions of mass concentrations 0,010 mg/l, 0,020 mg/l, 0,040 mg/l and 0,060 mg/l into the four different 50 ml volumetric flasks and dilute to final volume with water. These solutions shall be re-prepared for each day of measurement.

The calibration solutions described here shall be understood as an example. The concentrations prepared shall be in the linear range of the measuring device. The acid concentration of the calibration solutions shall be matched to the amounts of acid used in the digestion.

#### 4.6 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 10 ml of nitric acid (4.2) and 2 ml of hydrochloric acid (4.3) in 100 ml of water.

#### 4.7 Matrix modifiers for graphite furnace AAS

##### 4.7.1 General

Tin compounds shall be stabilized by matrix modifiers for quantification by graphite furnace AAS during the ashing step. Different matrix modifiers are used at different concentrations. To use a suitable modifier, first consider the recommendations of the device manufacturer. Select a suitable modifier; use the sample matrix to be examined to verify the ashing temperature of the graphite furnace program and optimize it in such a way that no tin is lost in the graphite tube during the mineralization step.

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In 4.7.2 to 4.7.4 an example is presented of a modifier for quantifying tin.

**4.7.2 Ammonium dihydrogen phosphate solution**

Mass fraction  $w \approx 10\%$ .

Dissolve 10,0 g of ammonium dihydrogen phosphate ( $\text{NH}_4\text{H}_2\text{PO}_4$ ) in 100 ml of water.

**4.7.3 Magnesium nitrate solution**

Mass concentration  $\rho(\text{Mg}) = 10\text{ g/l}$ .

Dissolve 10,5 g of magnesium nitrate hexahydrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) in 100 ml of water.

NOTE Commercially available solutions can also be used.

**4.7.4 Matrix modifiers for quantification of tin**

$\rho(\text{NH}_4\text{H}_2\text{PO}_4) = 50\ \mu\text{g}/10\ \mu\text{l}$  and  $\rho(\text{Mg}(\text{NO}_3)_2) = 3\ \mu\text{g}/10\ \mu\text{l}$ .

Pipette 2,5 ml of ammonium dihydrogen phosphate solution (4.7.2) and 0,25 ml of magnesium nitrate solution (4.7.3) into a 50 ml volumetric flask; add 1 ml of nitric acid (4.2), and dilute to volume with water.

This solution remains usable for several months when refrigerated.

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**5 Apparatus and equipment****5.1 General**

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

**5.2 Atomic absorption spectrometer**

with nitrous oxide/acetylene burner and atomization system.

**5.3 Atomic absorption spectrometer**

with background correction (Zeemann background correction is recommended), graphite tube furnace and autosampler.

**5.4 Element-selective detection****5.4.1 Element-specific lamp**

for tin (hollow-cathode or electrodeless discharge lamp).

**5.4.2 Continuum radiation source**

with high-resolution monochromatic illuminator as an alternative to 5.4.1. The resolution of the measuring equipment at normal operation shall correspond to at least the half-width value of the emission line of the element specific lamp (usually 1 pm to 3 pm).



## 6 Procedure

### 6.1 Digestion of the sample

Mineralize the sample in 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 the 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** Precisely weigh 0,4 g to 0,5 g of dry sample (residual moisture of less than 20 %) in a 100 ml digestion vessel and mix it with 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 can be used directly or can be diluted for the subsequent quantification of tin.

### 6.2 Flame atomic absorption spectrometry

#### 6.2.1 General

Start the instrument and let it stabilise, then optimise it according to the manufacturer's specifications. Set the wavelength to 286,3 nm or 235,5 nm. The nitrous oxide/acetylene flame needs special attention. In general reducing conditions give the best response. If deposits are formed on the burner they need to be removed before they interfere with the determination. If necessary, use background correction.

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#### 6.2.2 Quantification by flame AAS

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Warm up and stabilize the instrument; then begin measurements.

#### 6.2.3 Calibration and measurement

Use the blank solution (4.6) to zero the instrument. Measure the calibration solutions (4.5.2) and use the extinctions (absorbance) and concentrations to generate a calibration curve. Determine the linear range of the calibration function.

The measurement solution is taken up and measured. Use the calibration curve to convert the determined extinction into concentration units.

For long series of measurements, verify the blank and the calibration function multiple times.

### 6.3 Graphite furnace atomic absorption spectrometry

#### 6.3.1 General

Set and optimize the instrument according to the manufacturer's specifications. Set the wavelength to 286,3 nm. Use the manufacturer's specifications to set the slit width, volumes of samples and modifiers, and the temperatures and times of the drying, ashing and atomizing steps. Adjust these parameters to the sample matrix to be examined, and optimize the ashing and atomizing times and temperatures in particular.

The instrument parameters depend on the type and amount of modifier used, the type of graphite tube heating and the settings that are recommended by the manufacturer and that result from direct optimization.