



# SLOVENSKI STANDARD

## oSIST prEN 17265:2018

01-oktober-2018

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**Živila - Določevanje elementov in njihovih kemijskih oblik - Določevanje aluminija z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP-OES)**

Foodstuffs - Determination of elements and their chemical species - Determination of aluminium by inductively coupled plasma optical emission spectrometry (ICP-OES)

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Aluminium mit der optischen Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)

Produits alimentaires - Dosage des éléments et de leurs espèces chimiques - Dosage de l'aluminium par spectrométrie d'émission optique avec plasma à couplage inductif (ICP-OES)

**Ta slovenski standard je istoveten z: prEN 17265**

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**ICS:**

67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products
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**oSIST prEN 17265:2018**

**en,fr,de**



EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**DRAFT**  
**prEN 17265**

July 2018

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ICS 67.050

English Version

## Foodstuffs - Determination of elements and their chemical species - Determination of aluminium by inductively coupled plasma optical emission spectrometry (ICP-OES)

Produits alimentaires - Dosage des éléments et de leurs espèces chimiques - Dosage de l'aluminium par spectrométrie d'émission optique avec plasma à couplage inductif (ICP-OES)

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Aluminium mit der optischen Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 275.

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## European foreword

This document (prEN 17265:2018) has been prepared by Technical Committee CEN/TC 275 “Food Analysis – Horizontal methods”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

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SIST EN 17265:2019

<https://standards.iteh.ai/catalog/standards/sist/5a23b9d0-662e-4eef-901f-95c6da1a4c2d/sist-en-17265-2019>

## prEN 17265:2018 (E)

### 1 Scope

This document describes a method for the determination of aluminium in food by inductively coupled plasma optical emission spectrometry (ICP-OES) after pressure digestion. This method is suitable for mass fraction in the range of 15 mg/kg to 200 mg/kg. At concentrations above 200 mg/kg digestion temperatures higher than 220 °C can be necessary to recover the aluminium as completely as possible.

### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 elements and their chemical species - General considerations and specific requirements*

EN 13805, *Foodstuffs - Determination of trace elements - Pressure digestion*

### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

### 4 Principle

Aluminium is determined quantitatively by ICP-OES after digestion of the sample with nitric acid, with addition of water in case of foods with low water content, according to the pressure digestion process described in EN 13805 but without the use of hydrofluoric acid. The digestion conditions are chosen in such a way that even for samples with aluminium compounds of low solubility (e.g. silicates, oxides) low findings are kept to a minimum.

### 5 Reagents

The mass concentration of aluminium shall be low enough in the reagents and water not to affect the results.

All reagents shall be of analytical grade, i.e. pro analysi, p.a. or similar unless otherwise specified.

Use water conforming to grade 2 of EN ISO 3696.

**5.1 Nitric acid**, mass fraction  $w$  = at least 65 %, density = 1,4 g/ml

**5.2 Aluminium stock solution**, mass concentration  $\rho$  = 1000 mg/l

**5.3 Scandium stock solution**,  $\rho$  = 1000 mg/l as **internal standard**

Scandium is mentioned as an example of an internal standard. Scandium, yttrium and ytterbium are suitable as well. Only a negligible quantity of the internal standard may be present in the sample.

## 5.4 Standard solutions

### 5.4.1 General

When preparing solutions special care shall be taken to avoid contaminations. For aluminium there are various sources of contamination, e.g. volumetric flasks made of glass. Annex B of this document requires special attention.

### 5.4.2 Aluminium standard solution, $\rho = 10 \text{ mg/l}$

Fill 10 ml of water into a 50-ml volumetric flask, add 2 ml of nitric acid (5.1) and mix. After cooling down to room temperature, pipette exactly 500  $\mu\text{l}$  of the aluminium stock solution (5.2) to the flask and fill up with water to the mark. This standard solution is stable for at least 3 months.

### 5.4.3 Rhodium standard solution (internal standard solution), $\rho = 10 \text{ mg/l}$

Fill approximately 10 ml of water into a 50-ml volumetric flask, add 2 ml of nitric acid (5.1) and mix. After cooling down to room temperature, pipette exactly 500  $\mu\text{l}$  of the scandium stock solution (5.3) to the flask and fill up with water to the mark. This standard solution is stable for at least 3 months.

## 5.5 Aluminium calibration solutions

The concentrations of the calibration solutions described are examples and may be changed according to the sensitivity of the measuring instrument and the concentration range to be analysed. Carry out the calibration within the linear range of the detector system. At least 3 calibration solutions of different concentrations should be prepared. Make sure that the acid concentration of the calibration solutions corresponds to the test solution.

In order to avoid contaminations originating from the flask material, do not use volumetric flasks made from borosilicate glass. The information on contaminations provided in B.1 is to be regarded.

Mix all calibration and test solutions as well as the zero-point solution with an equal proportion of internal standard solution (5.4.3).

The calibration solutions are prepared from the standard solution (5.4.2) according to the following scheme in Table 1:

**Table 1 — Example of aluminium calibration solutions**

Calibration solution no	Volume of standard solution (5.4.2)	Volume of internal standard solution (5.4.3)	Mass concentration of aluminium in the calibration solution
	$\mu\text{l}$	$\mu\text{l}$	mg/l
1	100	2000	0,05
2	200	2000	0,10
3	500	2000	0,25
4	1000	2000	0,50
5	2000	2000	1,00

**EXAMPLE** Fill 4 ml to 5 ml of water into a 20-ml volumetric flask, add 1,25 ml of nitric acid (5.1) and mix. After cooling down to room temperature, pipette standard solution and internal standard solution according to Table 1 above to the flask, fill up to the mark with water, and mix.

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Alternatively the internal standard solution can be pumped into the sample flow via a Y-piece during measurement. In this case the internal standard solution is not pipetted into the calibration solutions.

NOTE The acid concentration of the calibration solution in the example is adapted to a digestion with 2,5 ml of nitric acid (5.1), a filling volume of 20 ml, and a dilution factor of 2 (in case of a dilution with water).

The calibration solutions shall be freshly prepared each working day.

**5.6 Zero-point solution**

20 ml of zero-point solution contain water, 1,25 ml of nitric acid (5.1) and 2 000 µl internal standard solution (5.4.3) according to the example given in 5.5.

**6 Apparatus**

All equipment and labware that come into direct contact with the sample and the solutions used shall be carefully pretreated/cleaned according to EN 13804 to minimize the blank value (see Annex B for details). In addition to standard laboratory equipment, use the following:

**6.1 Inductively coupled plasma optical emission spectrometer (ICP-OES)**

Optical emission spectrometer with inductively coupled argon plasma, sample supply and atomization system as well as device control and data acquisition. The instrument settings shall allow the measurement of the emission intensities on both sides of the emission line and enable a background correction on both sides.

**6.2 Digestion vessels**, e.g. of tetrafluoroethylene or quartz with volumes from 70 ml to 100 ml

**6.3 Test tube shaker**, optional

**7 Procedure****7.1 Digestion****7.1.1 General**

To quantify aluminium in food certain basic conditions shall be respected especially during digestion:

- in case of high aluminium contents it could be beneficial to use a test portion as small as possible (at least 200 mg for dry samples) in order to digest aluminium compounds as completely as possible. Ensure that the sample is sufficiently homogeneous;
- in case of samples with low water content, first add water and mix intensively, before adding concentrated nitric acid. Do not use hydrogen peroxide;
- for samples containing silicates digestion temperatures above 200 °C can be necessary in order to quantify the aluminium as completely as possible.

Further instructions regarding test portions and digestion are given in B.2 and B.3.

For pressure digestion according to EN 13805, different vessels may be used depending on the instrument type and manufacturer. The maximum test portion and the minimum liquid volume depend on the pressure stability of the respective vessels. The specifications according to 7.2 refer to digestion vessels with volumes from 70 ml to 100 ml and a minimum liquid volume of 5 ml.

All indications in 7.2 shall be adjusted to the digestion instrument used. For safety reasons the manufacturer's specifications shall be strictly followed.



### 7.1.2 Digestion procedure

Before digestion different amounts of water, depending on the different types of food [7], are added in order to obtain comparable acid concentrations in the final digestion solution. The amount of water to be added depends on the test portion and thus on the content of carbon and water in the food type.

Add just as much water to the test portion that is necessary to suspend the food completely. Then complete the test portion with water to reach 3 g. Water is also added to fat-containing foods, even if a suspension is hardly possible.

**EXAMPLE 1** Example for digestion of powdery food: Add 2,5 ml of water to the dry food, e.g. 0,5 g of flour. Mix the contents of the digestion vessel intensively, e.g. on a test-tube shaker. Repeat shaking every 10 min to 15 min until the suspension is homogeneous. No agglutinated particles shall remain, and the sample material shall be well. Fat-containing samples require more time and repeated shaking. After at least 30 min, add 2,5 ml of nitric acid (5.1) to the sample. Mix thoroughly again to obtain a homogeneous suspension. After the pre-reaction has subsided, close the digestion vessel, and start the digestion.

**EXAMPLE 2** Example for digestion of a sample with high water content, e.g. lettuce: Directly add 2,5 ml of nitric acid (5.1) to 3 g of lettuce and mix thoroughly. It is not necessary to add water. After the pre-reaction has subsided, close the digestion vessel, and start the digestion.

A digestion temperature of at least 200 °C to be kept for at least 20 min is required for a complete digestion in the microwave.

The digestion conditions depend on the manufacturer's specifications, the reactivity of the sample, the maximum pressure stability of the digestion vessel, and the attainable temperature. To monitor the laboratory-specific digestion conditions, it is recommended to carry out measurements with reference materials containing certified contents.

If precipitations or turbidities are visible in the digestion solutions, low findings can occur. In these cases use smaller test portions and higher digestion temperatures up to 300 °C in order to minimize or avoid possible low findings.

**NOTE 2** Turbidities or small amounts of precipitates adhering to the wall or bottom of the vessel are only visible in vessels with high transparency (e.g. aluminium-free quartz vessels). Plastic vessels are often not completely transparent, making precipitates difficult to discern.

Fill up the digestion solution obtained by pressure digestion to a defined volume, e.g. 20 ml. Add the internal standard (5.4.3) (e.g. 1000 µl) to an aliquot of the digestion solution, e.g. 5 ml, and fill up with water to 10 ml. Separate insoluble residues before measurement. If the digestion solution has a different final volume than 20 ml and a different dilution factor than 2 after addition of the internal standard, adapt the acid concentration of the calibration solutions respectively. All test solutions shall have approximately the same acid concentration as the calibration solutions. The digestion solution shall be measured within 2 weeks.

Matrix effects can occur in digestion solutions. It is recommended to dilute the digestion solution by a factor of 10 to reduce matrix effects and avoid contamination of instruments.

In order to determine low aluminium contents reliably, choose a small filling-up volume after digestion. During sample preparation special care should be taken to avoid contaminations. See also general information in Annex B.

## 7.2 Inductively coupled plasma optical emission spectrometry

### 7.2.1 ICP-OES operating conditions

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

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Once the instrument is optimized, start the measurement. For the determination of aluminium a wavelength of 396,15 nm is recommended. It is necessary to measure the emission intensity of the background on both sides of the emission line and to subtract it from the intensity measured at a wavelength of 396,15 nm (background correction on both sides).

Alternatively use the emission lines from Table C.1, which show different sensitivities, but also more interferences than the wavelength recommended.

Measure the zero-point solution (5.6) and the calibration solutions 1 to 5 (5.5, Table 1) and generate a calibration curve from the emission intensities and concentrations. In case of complex matrices it can be of advantage to perform a standard addition.

Aspirate and measure the sample test solution. Determine the counting rate and correct it by the background intensity, then convert it into concentration units with the help of the calibration curve.

Check the stability of the emission intensities of the calibration samples by measuring a control sample at sufficiently frequent intervals (e.g. after 10 samples). If necessary, re-calibrate the system.

In case of samples with high element concentrations, sufficient rinsing is necessary before analysing the next test solution. The rinsing behaviour can be monitored with the help of zero-point solutions (5.6).

When preparing dilutions, make sure that the diluted test solutions have the same concentration of acids as the original test solutions.

**7.2.3 Quality control**

For quality control purposes, analyse control samples with reliably known contents of aluminium (see B.4) in parallel to every series of measurement. Include all steps of the procedure, beginning with the digestion. Prepare and measure blank solutions for every digestion series, also including all steps of the procedure.

**8 Evaluation****8.1 Calculation**

Calculate the mass fraction  $w$  in milligrams per kilogram or the mass concentration,  $\rho$ , in milligrams per litre of sample according to Formula (1):

$$w \text{ or } \rho = \frac{a \cdot V \cdot F}{m \text{ or } v} \quad (1)$$

where

- $a$  is the mass fraction of aluminium in the sample solution in micrograms per litre;
- $V$  is the volume of the sample solution after digestion in millilitres;
- $F$  is the dilution factor of the sample measuring solution;
- $m$  is the mass of the test portion in milligrams or the sample volume in microlitres used for digestion.

It is not recommended to subtract the blank value. The aluminium content of the blank solution should be low enough not to considerably change the test result when subtracted. If this is not the case, determine the origin of the aluminium content in the blank solution, and if necessary, repeat the digestion series.