



SLOVENSKI STANDARD
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Živila - Določevanje elementov in njihovih kemijskih oblik - Določevanje aluminija z masno spektrometrijo z induktivno sklopljeno plazmo (ICP-MS)

Foodstuffs - Determination elements and their chemical species - Determination of aluminium by inductively coupled plasma mass spectrometry (ICP-MS)

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Aluminium mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS)

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

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Foodstuffs - Determination elements and their chemical species - Determination of aluminium by inductively coupled plasma mass spectrometry (ICP-MS)

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

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Aluminium mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS)

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European foreword

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This document is currently submitted to the CEN Enquiry.

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1 Scope

This document specifies a method for the determination of aluminium in food by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion. This method is suitable for mass fractions in the range of 1 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-MS 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, with a certified mass concentration $\rho = 1\ 000$ mg/l¹⁾

5.3 Rhodium stock solution, $\rho = 1\ 000$ mg/l as internal standard

The internal standard shall be free from aluminium impurities and shall be present in the sample in negligibly small amounts only.

Rhodium and indium are suitable as internal standards. Other internal standards, especially those having mass/charge ratios (m/z) < 100, shall not be used, since molecule ion interferences can distort the measurement.

NOTE The verification of molecule ion interferences was performed in the collaborative study using a high-resolution ICP mass spectrometer with a resolution of $R = 4\ 000$ ¹⁾.

1) resolution $R = m/\Delta m$.

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 1 $\rho = 10 \text{ mg/l}$

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 μl of the aluminium stock solution (5.2) to the flask, and fill up with water to the mark. This standard solution 1 is stable for at least 3 months.

5.4.3 Aluminium standard solution 2 $\rho = 1 \text{ mg/l}$

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 5 ml of the aluminium standard solution 1 of $\rho = 10 \text{ mg/l}$ (5.4.2) to the flask and fill up with water to the mark. This standard solution 2 is stable for at least 3 months.

5.4.4 Aluminium standard solution 3 $\rho = 0,1 \text{ mg/l}$

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 5 ml of the aluminium standard solution 2 of $\rho = 1 \text{ mg/l}$ (5.4.3) and fill up with water to the mark. This standard solution 3 is stable for at least 3 months.

5.4.5 Rhodium standard solution 1 (internal standard 1) $\rho = 10 \text{ mg/l}$

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 μl of the rhodium stock solution (5.3) to the flask and fill up with water to the mark. This standard solution 1 is stable for at least 3 months.

5.4.6 Rhodium standard solution 2 (internal standard 2) $\rho = 1,0 \text{ mg/l}$

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 5 ml of the rhodium standard solution 1 of $\rho = 10 \text{ mg/l}$ (5.4.5) and fill up with water to the mark. This standard solution 2 is stable for at least 3 months.

5.5 Aluminium calibration solutions

The mass 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. Make sure that the calibration is carried out within the linear range of the detector system. For calibration 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.2 is to be regarded.

The calibration solutions are prepared from standard solution 2 and 3 (5.4.3 and 5.4.4) according to the following scheme in Table 1:

Table 1 — Example of aluminium calibration solutions

Calibration solution no	Quantity of standard solution	Quantity of internal standard 2 (5.4.6)	Mass concentration of aluminium in the calibration solution
		μl	$\mu\text{g/l}$
1	200 μl of standard solution 3 (5.4.4)	200	1
2	100 μl of standard solution 2 (5.4.3)	200	5
3	500 μl of standard solution 2 (5.4.3)	200	25
4	2000 μl of standard solution 2 (5.4.3)	200	100

EXAMPLE Fill 4 ml to 5 ml of water into a 20 ml volumetric flask, add 250 μl of nitric acid (5.1), and mix. After cooling down to room temperature, pipette the standard solution and the internal standard solution 2 according to Table 1 to the flask, fill up to the mark with water and mix.

Alternatively the internal standard 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 10 (in case of a dilution with water).

The calibration solutions shall be freshly prepared each working day.

5.6 Zero-point solution

For the zero-point solution add 1,25 ml of nitric acid (5.1) and if applicable the internal standard 2 (in exactly the same mass concentration as in the calibration solutions, according to the example given in 5.5) to a 100 ml volumetric flask and fill up with water.

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 mass spectrometer (ICP-MS),

with inductively coupled argon plasma, sample supply and nebulising system as well as instrument controlling and data acquisition. In order to avoid interferences on the atomic mass of aluminium, it can be necessary to use a mass spectrometer that is able to delete or minimize interferences (e.g. which is equipped with cell technology or resolution above 300).

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 220 °C can be necessary in order to quantify the aluminium as completely as possible.

Further information 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 [5], 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 initial 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 welled. 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.

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If precipitations or turbidities are visible in the digestion solutions, low findings can occur. In these cases use smaller sample weights 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. Twenty ml. Add the internal standard (5.4.5) (e.g. 100 µl) to an aliquot of the digestion solution, e.g. 1 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 10 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 Mass spectrometry with inductively coupled plasma**7.2.1 ICP-MS 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. Select the instrument settings in such a way that not only a high sensitivity is achieved, but also a low amount of molecule ion interferences (e.g. oxide ratio, twofold charged ions).

For this purpose, measure an optimization solution containing e.g. Mg, Rh, Pb, and Ce ($\rho = 10 \mu\text{g/l}$). Depending on the manufacturer's recommendations, the formation rate of oxides and twofold charged ions should be $< 3 \%$, for example.

When employing an instrument equipped with a collision or reaction cell, which is used e.g. to reduce interferences caused by polyatomic ions, optimize the flow rate of the cell gas/gasses.

When applying different resolutions of the mass spectrometer, adjust the mass windows for each selected resolution in order to make sure that the isotope to be determined is positioned in the centre of the window. Measure at least one internal standard at each resolution level.

Commercially available mass spectrometers often use different types of detectors or detector operating modes in order to cover a larger linear concentration range. In such cases, the instrument shall be able to ensure that the sensitivity transitions of the detectors or operating modes are continuous and without any leaps.

7.2.2 Determination by ICP-MS

Once the instrument is optimized, start the measurements. Aluminium is determined at a mass/charge ratio (m/z) of 27. If the resolution of the mass spectrometer is $R = 300$, the following molecule ions can cause interferences: $^{12}\text{C}^{15}\text{N}$, $^{13}\text{C}^{14}\text{N}$, $\text{H}^{12}\text{C}^{14}\text{N}$, ^{26}MgH .

NOTE Low digestion temperatures usually lead to an incomplete digestion of the foodstuffs. The non-digested organic compounds can then cause carbon-based molecular ion interference.

Rhodium or indium are recommended as internal standards for the determination of aluminium. Other internal standards, especially those having mass/charge ratios (m/z) < 100 , shall not be used, since molecule ion interferences can distort the measurement.