SLOVENSKI STANDARD
SIST EN 17264:2019

01-november-2019

Živila - Določevanje elementov in njihovih spojin - 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)

Ta slovenski standard je istoveten z: **EN 17264:2019**

ICS:

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

SIST EN 17264:2019 **en,fr,de**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 17264

September 2019

ICS 67.050

English Version

Foodstuffs - Determination of 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 mittels Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS)

This European Standard was approved by CEN on 28 July 2019.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. EN 17264:2019 E

Contents

Page

European foreword

This document (EN 17264:2019) 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 March 2020, and conflicting national standards shall be withdrawn at the latest by March 2020.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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 was validated for infant formula, wheat noodle, cheese, liver, beetroot and cocoa powder at 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*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

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 https://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 ρ = 1 000 mg/l.

5.3 Rhodium stock solution, ρ = 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)].

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, ρ = 10 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, ρ = 1 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 ρ = 10 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, ρ = 0,1 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 ρ = 1 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 solution 1), ρ = 10 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 internal standard solution 1 is stable for at least 3 months.

5.4.6 Rhodium standard solution 2 (internal standard solution 2), ρ = 1,0 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 ρ = 10 mg/l (5.4.5) and fill up with water to the mark. This internal standard solution 2 is stable for at least 3 months.

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

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. Note the information on contaminations provided in B.1.

Prepare the calibration solutions from aluminium 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	Volume of aluminium standard solution	Volume of internal standard solution 2 (5.4.6) µl	Mass concentration of aluminium in the calibration solution µg/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).

The ICP-MS shall include 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) [1].

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

6.3 Test tube shaker, optional.

6.4 Analytical balance, capable to weigh to the nearest milligram.

7 Procedure

7.1 Digestion

7.1.1 General

To ensure complete dissolution of aluminium the following shall be respected:

— ensure that the sample is sufficiently homogeneous;

— in case of incomplete digestion or high aluminium contents, it may 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;

— for samples containing silicates, digestion temperatures above 220 °C may be necessary in order to dissolve the aluminium as completely as possible;

— 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 digestion.

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 7.1.2 refer to digestion vessels with volumes from 70 ml to 100 ml and a minimum liquid volume of 5 ml.

All indications in 7.1.2 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 [2], 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.