SLOVENSKI STANDARD
SIST EN 15505:2009

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Živila - Določevanje elementov v sledovih - Določevanje natrija in magnezija z uporabo spektrometrije s plamensko atomsko absorpcijo (AAS) po mikrovalovnem razklopu

Foodstuffs - Determination of trace elements - Determination of sodium and magnesium by flame atomic absorption spectrometry (AAS) after microwave digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Natrium und Magnesium mit Flammen-Atomabsorptionsspektrometrie (AAS) nach Mikrowellenaufschluss

Produits alimentaires - Dosage des éléments traces - Dosage du sodium et du magnésium par spectrométrie d'absorption atomique de flamme après digestion par micro-ondes

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Foodstuffs - Determination of trace elements - Determination of sodium and magnesium by flame atomic absorption spectrometry (AAS) after microwave digestion

Produits alimentaires - Dosage des éléments traces - Dosage du sodium et du magnésium par spectrométrie d'absorption atomique de flamme après digestion par micro-ondes

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Natrium und Magnesium mit Flammen-Atomabsorptionsspektrometrie (AAS) nach Mikrowellenaufschluss

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Contents

Page

Foreword

This document (EN 15505:2008) 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of an identical text or by endorsement, at the latest by September 2008, and conflicting national standards shall be withdrawn at the latest by September 2008.

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1 Scope

This document specifies methods for the determination of sodium and magnesium in foodstuffs by flame atomic absorption spectrometry (AAS) after microwave digestion. Collaborative studies have been carried out (Annex A). The method is suitable for the determination of sodium not less than 1 500 mg/kg and magnesium not less than 250 mg/kg. Data for calcium is included for information (Annex B). The method is not applicable to wheat bran.

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 13804, *Foodstuffs — Determination of trace elements — Performance criteria, general considerations and sample preparation*

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

3 Principle

The samples are digested in closed vessels in a microwave oven in a mixture of nitric acid and hydrogen peroxide. The resulting solution is diluted with water, and the sodium and magnesium contents are determined by flame-AAS using matrix modifiers.

WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

4 Reagents

4.1 General

Use only reagents and water, which shall have an element level low enough not to affect results. The use of certified stock solutions is advisable.

4.2 Nitric acid

4.2.1 Nitric acid, not less than 65 % of approximately mass concentration $\rho(HNO_3)$ = 1,4 g/ml.

In case of insufficient purity, it is necessary to purify the nitric acid in a distillation apparatus as described in EN 13805.

4.2.2 Diluted nitric acid solution 1, mass fraction w = 2,7 %

Dilute 42 ml nitric acid (4.2.1) to 1 000 ml with water.

4.2.3 Diluted nitric acid solution 2, mass fraction w = 0,65 %

Mix nitric acid (4.2.1) and water in a proportion minimum of 1 + 99 parts by volume.

4.3 Hydrochloric acid, mass fraction w = 37 %

4.4 Hydrogen peroxide, mass fraction w = 30 %

4.5 Cesium chloride solution (CsCl), for use in AAS analysis

Dissolve 31,75 g of cesium chloride in water and dilute to 250 ml. The solution will be stable in a refrigerator for at least 6 months.

4.6 Lanthanum(III)oxide (La_2O_3) solution, for use in AAS analysis, 5 % (weight/volume)

Weigh 14,66 g of lanthanum(III)oxide into a 250 ml beaker, moisten with 10 ml of water and add 62,5 ml of HCl (4.3). Transfer to a 250 ml volumetric flask and fill up with water. The solution will be stable in a refrigerator for one month.

4.7 Sodium solution

4.7.1 Sodium stock solution, mass concentration ρ(Na) = 1 000 mg/l

4.7.2 Sodium standard solution, mass concentration ρ(Na) = 10 mg/l

Dilute 1 ml of sodium stock solution (4.7.1) to 100 ml in a volumetric flask with 2,7 % nitric acid (4.2.2). The solution will remain stable for one month at room temperature.

4.8 Magnesium solution

4.8.1 Magnesium stock solution, mass concentration ρ(Mg) = 1 000 mg/l

4.8.2 Magnesium standard solution, mass concentration ρ(Mg) = 10 mg/l

Dilute 1 ml of magnesium stock solution (4.8.1) to 100 ml in a volumetric flask with 0,65 % nitric acid (4.2.3). The solution will remain stable for one month at room temperature.

5 Apparatus and equipment

5.1 General

All glassware and plastic ware should be carefully cleaned and rinsed according to the procedure in EN 13804.

5.2 Laboratory microwave oven

Check the microwave oven for delivered power according to the procedure in EN 13804.

5.3 Atomic absorption spectrometer

5.4 Element specific lamps

For sodium and magnesium element specific lamps with wavelength of 589,0 nm and 285,2 nm respectively will apply.

5.5 Acetylene

Use acetylene of appropriate quality.

5.6 Air

6 Procedure

6.1 Pre-treatment

Homogenise the sample in accordance with the recommendations in EN 13804. If necessary, dry the sample in a way that does not affect the element contents, e.g. by freeze drying.

6.2 Sample preparation

Use the test solution obtained by microwave oven digestion method according to EN 13805 for the determination of sodium and magnesium.

6.3 Dilution

Pipette a suitable volume of the sample solution, add 5 % La-solution (4.6) and dilute this volume with 0,65 % nitric acid (4.2.3) so that the final concentration of Mg is within the linear range of measurement of the elements. In this example, the following ranges are selected for the standard curves; for Mg, 0,05 mg/l to 0,4 mg/l. The lowest point may be lower, if required by the concentrations of the sample solutions. Add 5 % of La-solution (4.6) to a final mass fraction of La of 1 % (e.g. 2 ml 5 % is diluted to 10 ml).

Pipette a suitable volume of the sample solution, add 1 ml of Cs-solution (4.5) and dilute this volume with 2,7 % nitric acid (4.2.2) so that the final concentration of Na is within the range of measurement of the element. In this example, the following range is selected for the standard curve: 0,1 mg/l to 1,0 mg/l. The lowest point may be lower required by concentration of the sample solution.

6.4 Preparation of standard solution

Prepare working standard solutions for Na of 0,1 mg/l, 0,25 mg/l, 0,5 mg/l, 0,75 mg/l and 1,0 mg/l from the Na standard solution (4.7.2). Add 0,5 ml, 1,25 ml, 2,5 ml, 3,75 ml and 5 ml to separate 50 ml volumetric flasks, add 1 ml of Cs-solution (4.5) and dilute to the mark with diluted nitric acid solution 1 (4.2.2). Prepare fresh solution daily.

Prepare working standard solutions for Mg of 0,05 mg/l, 0,1 mg/l, 0,2 mg/l and 0,4 mg/l from the Mg standard solution (4.8.2). Add 0,25 ml, 0,5 ml, 1,0 ml and 2,0 ml to separate 50 ml volumetric flasks, add 10 ml of 5 % La-solution (4.6) and dilute to the mark with diluted nitric acid solution 2 (4.2.3). Prepare fresh solutions daily.

6.5 Settings for the atomic absorption spectrometer

Before every determination, adjust the instrument as specified in the manufacturer's operating manual. Example of instrument parameters for sodium and magnesium may be 589,0 nm and 285,2 nm (wavelength) and 0,2 nm and 0,7 nm (slit), respectively.

7 Calculation

Calculate the element content, w, as mass fraction of sodium and magnesium in milligram per kilogram of the sample, using the following equation:

$$w = \frac{a \times V \times F}{m} \tag{1}$$

where

a is the content of the element in the test solution, in milligram per litre;

V is the volume of the digestion solution after being made up, in millilitres;

F is the dilution factor of the test solution;

m is the initial sample mass, in grams.

If necessary, subtract the concentration in the blank solution from the concentration of the sample solution, before the calculation is made.

8 Precision

8.1 General

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

8.2 Repeatability and reproducibility

Repeatability is the absolute difference between two independent single test results (blind duplicate), obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time. This will in not more than 5 % of the cases exceed the values of r given in Table 1 to Table 2. Reproducibility is the absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment. This will in not more than 5 % of the cases exceed the values of R given in Table 1 to Table 2.