SLOVENSKI STANDARD
SIST EN 14332:2005

01-januar-2005

Ģivila - Doloļevanje elementov v sledovih - Doloļevanje arzena v morskih sadeģih z metodo atomske absorpcijske spektrometrije z grafitno kiveto po mikrovalovnem razklopu

Foodstuffs - Determination of trace elements - Determination of arsenic in seafood by graphite furnace atomic absorption spectrometry (GFAAS) after microwave digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Arsen in Meeresfrüchten mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Mikrowellenaufschluss

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Produits alimentaires - Dosage des éléments trace - Détermination de l'arsenic dans les aliments d'origine marine par spectrométrie d'absorption atomique a four graphite (GFAAS) apres digestion par micro-ondes

Ta slovenski standard je istoveten z: EN 14332:2004

ICS:

67.120.30 Ribe in ribji proizvodi Fish and fishery products

SIST EN 14332:2005 en

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14332

July 2004

ICS 67.120.30

English version

Foodstuffs - Determination of trace elements - Determination of arsenic in seafood by graphite furnace atomic absorption spectrometry (GFAAS) after microwave digestion

Produits alimentaires - Dosage des éléments trace - Détermination de l'arsenic dans les aliments d'origine marine par spectrométrie d'absorption atomique à four graphite (GFAAS) après digestion par micro-ondes

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This European Standard was approved by CEN on 30 April 2004.

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 Central Secretariat or to any CEN member.

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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 Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 14332:2004) 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 January 2005, and conflicting national standards shall be withdrawn at the latest by January 2005.

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

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1 Scope

This document specifies a method for the determination of arsenic in seafood by graphite furnace atomic absorption spectrometry (GFAAS) after microwave digestion [1], [2]. The collaborative study has included food having an arsenic content ≥ 2 mg/kg dry matter.

Specific foodstuffs for which European Standards exist are excluded from the scope of this horizontal document. It is the task of the analyst to review if vertical documents exist.

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.*

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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 arsenic contents are determined by GFAAS using matrix modifiers.

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

4 Reagents

4.1 General

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

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4.2 Nitric acid, $w \geq 65$ % (mass fraction), having a density of approximately $\rho(HNO_3) = 1,4$ g/ml. In case of insufficient purity it is necessary to purify the acid in a distillation apparatus as described in EN 13805.

4.2.1 Diluted nitric acid solution 1, $w = 6,5$ % (mass fraction), to be prepared by mixing nitric acid (4.2) and water in a proportion minimum of 1 + 9 parts by volume.

4.2.2 Diluted nitric acid solution 2, $w = 0,65$ % (mass fraction), to be prepared by mixing nitric acid (4.2) and water in a proportion minimum of 1 + 99 parts by volume.

4.3 Hydrogen peroxide, w (H_2O_2) = 30 % (mass fraction)

4.4 Arsenic stock solution, with a mass concentration ρ(As) = 1 000 mg/l.

The use of a stock solution accompanied by a certificate is advisable.

4.5 Arsenic calibration solutions

Dilute arsenic stock solution (4.4) with diluted nitric acid (4.2.2) to the required number of solutions, which cover the linear range of the calibration curve of arsenic.

4.6 Matrix modifiers

4.6.1 General

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Instead of solutions 4.6.2 and 4.6.4, commercially available palladium nitrate and magnesium nitrate solutions may be used.

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4.6.2 Palladium stock solution ρ(Pd) = 10 mg/ml

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Dissolve 2,16 g of palladium nitrate, $Pd(NO_3)_2$ in approximately 10 ml of 6,5 % nitric acid (4.2.1) in a 100 ml volumetric flask and dilute to volume with water.

4.6.3 Palladium solution ρ(Pd) = 1,5 mg/ml

Dilute 1,5 ml of palladium stock solution (4.6.2) to 10 ml in a volumetric flask with 0,65 % nitric acid (4.2.2).

4.6.4 Magnesium stock solution ρ(Mg) = 10 mg/ml

Dissolve 10,54 g magnesium nitrate, $Mg(NO_3)_2 \cdot 6H_2O$ in approximately 10 ml of 6,5 % nitric acid (4.2.1) in a 100 ml volumetric flask and dilute to volume with water.

4.6.5 Magnesium solution ρ(Mg) = 1 mg/ml

Dilute 1 ml magnesium stock solution (4.6.4) to 10 ml in a volumetric flask with 0,65 % nitric acid (4.2.2).

4.6.6 Palladium nitrate/magnesium nitrate solution, to be prepared with appropriate volume of palladium solution (4.6.3) and magnesium solution (4.6.5) (1 + 1). 10 µl of the solution gives 7,5 µg Pd and 5,0 µg Mg.

5 Apparatus and equipment

5.1 General

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

5.2 Microwave oven, for laboratory use, checked for delivered power according to the procedure in

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EN 13804.

5.3 Atomic absorption spectrometer (AAS), with background correction, supplied with a graphite furnace consisting of a pyrolytically coated graphite tube with a pyrolytically coated platforms and autosampler.

5.4 Element specific lamp for arsenic

5.5 Argon, the purity not less than 99,998 %

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 arsenic content, e.g. by freeze drying.

6.2 Sample preparation

Use the test solution obtained by a suitable microwave oven digestion method e.g. EN 13805 for the determination of arsenic.

6.3 Determination

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6.3.1 Spectrometer settings

Before every determination, adjust the instrument as specified in the manufacturer's operating manual. Then determine an optimum test schedule (if possible using a sample matrix) taking into account, in particular, of parameters such as temperatures and times for the different steps in the temperature program. Example of instrument parameters for arsenic may be a wavelength of 193,7 nm and slit 0,7 nm.

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6.3.2 GFAAS-determination

6.3.2.1 General

Table 1 shows examples of instrumental parameters of the graphite furnace.

Table 1 — Graphite furnace time/temperature programme

Step (#)	Temperature (°C)	Ramp time (s)	Hold time (s)	Gas flow (ml/min)	Gas Type	Read
1	110	1	20	250	Ar	
2	130	9	30	250	Ar	
3	1 350	12	20	250	Ar	
4	2 150	fast ramp	4	stop		active
5	2 450	1	3	250	Ar	

The absorbance is determined for an aliquot of the test solution by GFAAS. If there is no significant difference between the slope of the calibration curve in the case of the standard addition method and the external calibration method, the latter can be employed. The calibration function shall consist of a minimum of 3 points including 0.

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NOTE 1 The date acquisition during the atomisation step can be performed by peak height or peak area measurement. When using platform technique, peak area measurement is recommended.

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NOTE 2 Too large an injection volume may cause foaming during the drying stage, which may cause poor precision.

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6.3.2.2 Standard addition method

Determine the linear range of the standard calibration function. It is important that the measurements are made in the linear range when the method of standard addition is used. A standard addition curve should consist of at least three points of which at least two are standard additions. The concentration of the highest standard should be 3 to 5 times the concentration in the sample solution. The concentration of the lower standard should be half of the highest standard.

Plot a graph of the absorbances obtained in this way against the added concentrations and extrapolate the resulting straight line until it intercepts the concentration axis.

In the case of AAS instruments with automatic sample injection systems, in which the addition takes place directly into the graphite furnace, the determination can be carried out without prior dilution and the risk of contamination will be significantly reduced.

6.4 Analytical quality control

For analytical quality control, blank solutions and reference samples having reliably known contents of arsenic to be determined shall be analysed in parallel with all the series of samples analysed according to EN 13804. The reference samples and the blank solutions shall be subjected to all the steps in the method, starting from digestion.