SLOVENSKI STANDARD
SIST EN 15517:2009

01-april-2009

Ģivila - Doloļ evanje elementov v sledovih - Doloļ evanje anorganskega arzena z metodo atomske absorpcijske spektrometrije - hidridna tehnika (HGAAS) po kislinski ekstrakciji

Foodstuffs - Determination of trace elements - Determination of inorganic arsenic in seaweed by hydride generation atomic absorption spectrometry (HGAAS) after acid extraction

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von anorganischem Arsen in Meeresalgen mit Atomabsorptionsspektrometrie-Hydridtechnik (HGAAS) nach Säureextraktion

Produits alimentaires - Dosage des éléments traces - Dosage de l'arsenic inorganique dans les algues marines par spectrométrie d'absorption atomique par génération d'hydrures (SAAGH) après extraction acide

Ta slovenski standard je istoveten z: EN 15517:2008

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

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Contents

Page

Foreword

This document (EN 15517:2008) 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of an identical text or by endorsement, at the latest by September 2008, and conflicting national standards shall be withdrawn at the latest by September 2008.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 a procedure for the determination of hydrochloric acid (gastric acid concentration) extractable inorganic arsenic in seaweed. Collaborative studies have been carried out (Annex A). The method is suitable for the determination of inorganic arsenic not less than 1 mg/kg and below 100 mg/kg on a dry weight basis. The amount of inorganic arsenic is considered to be that part determined by the procedure described in this document.

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*

3 Principle

Arsenic compounds are extracted from the sample by diluted hydrochloric acid (in accordance with EN 71-3 [5]) and the arsenic in the extract is determined by hydride generation AAS. In acidic media inorganic compounds of arsenic(III) and arsenic(V) as well as the monomethylarsonic acid (MMA) and dimethylarsinic (cacodylic) acid (DMA) form a volatile hydride with sodium borohydride. There is no reaction of the stable organic arsenic compounds like arsenosugar arseno-betaine and arseno-choline under these conditions. The gaseous hydride is transferred into a heated measuring cell (cuvette) by means of a carrier gas stream and decomposed. The absorption at 193,7 nm (arsenic line) serves as a measure of arsenic concentration. The hydride signal sensitivity of DMA reaches generally low rates as compared to As(III). The contribution of MMA in the hydride signal can be neglected, since MMA occurs in seaweed only in small amounts. The hydride generation AAS in combination with this hydrochloric acid extraction may be used as nearly selective method of determination for inorganic arsenic.

Generation of arsine from As(III) is much faster and gives greater sensitivity than generation from As(V) and is also less subject to interference. Arsenic(V) shall be reduced to arsenic(III) (pre-reduction) in order to avoid incorrect measurements.

4 Reagents

4.1 General

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

4.2 Hydrochloric acid, mass fraction w = 30 %, mass concentration ρ(HCl) = 1,15 g/ml.

4.3 Hydrochloric acid solution, substance concentration c = (0,07 ± 0,005) mol/l.

4.4 Hydrochloric acid, approximately c = 2 mol/l.

4.5 Sodium borohydride, $w \geq 96$ %

4.6 Sodium hydroxide, $w \geq 98$ %

4.7 Sodium borohydride solution, e.g. substance concentration c = 2 g/l (example for using the flow injection procedure described under 6.2.1 (b)).

Dissolve 2 g of sodium hydroxide pellets in water, add 2 g of sodium borohydride and dilute to 1 000 ml with water.

A fresh solution shall be prepared daily and filtered before use.

The concentration by mass of the sodium borohydride solution may vary with the system and the instructions of the relevant manufacturer shall therefore be observed.

4.8 Diluted hydrochloric acid, e.g. mass fraction $w \approx 3$ % (carrier solution, only for use in the flow injection procedure).

Dilute approximately 90 ml of hydrochloric acid (4.2) to 1 000 ml with water.

The concentration by mass of the carrier solution may vary with the system and the instructions of the relevant manufacturer shall therefore be observed.

4.9 L-Ascorbic acid, $w(C_6H_8O_6) \geq 99,7$ %

4.10 Potassium iodide, w(KI) ≥ 99,5 %

4.11 Potassium iodide/ascorbic acid solution

Dissolve 3 g of potassium iodide and 5 g of ascorbic acid in water and dilute to 100 ml.

Prepare a fresh solution daily.

The concentrations of the potassium iodide and ascorbic acid may vary slightly with the system and the instructions of the relevant manufacturer shall therefore be observed.

4.12 Diarsenic trioxide (As2O3), $w(As_2O_3) \geq 99,5$ %

4.13 Arsenic stock solution, with an arsenic mass concentration of 1000 mg/l.

If commercial stock solutions are not available, proceed as follows: dissolve 1,320 g of diarsenic trioxide

(4.12) in 25 ml of potassium hydroxide solution (ρ = 20 g/100 ml), neutralize with 20 % (mass fraction) sulfuric acid with phenolphthalein as indicator and dilute to 1 000 ml with 1 % (mass fraction) sulfuric acid.

4.14 Arsenic standard solutions

Dilute the arsenic stock solution (4.13) in several steps. The arsenic standard solutions shall contain an adequate amount of hydrochloric acid, e.g. 2 ml of hydrochloric acid (4.2) per 100 ml.

Example of a dilution series:

$$1000\ \text{mg/l} \xrightarrow[5/100]{} 50\ \text{mg/l} \xrightarrow[5/50]{} 5\ \text{mg/l} \xrightarrow[1/50]{} 0{,}1\ \text{mg/l}$$

A standard solution of 5 mg/l arsenic in 0,6 % (mass fraction) hydrochloric acid is stable for at least one week.

4.15 Arsenic calibration solutions

Prepare five calibration solutions in the required calibration range from the standard solution of 0,1 mg/l (4.14), ensuring that the concentrations of the calibration solutions are not outside the linear range of the calibration function and are also in the expected sample content range. The concentration of acid in the calibration solutions shall be equal to that in the sample solution.

Example for the 1 µg/l to 10 µg/l range:

$$0{,}1\ \text{mg/l} \xrightarrow[1/100]{} 1\ \mu\text{g/l}$$

$$\xrightarrow[3/100]{} 3\ \mu\text{g/l}$$

$$\xrightarrow[5/100]{} 5\ \mu\text{g/l}$$

$$\xrightarrow[8/100]{} 8\ \mu\text{g/l}$$

$$\xrightarrow[10/100]{} 10\ \mu\text{g/l}$$

The calibration solutions may also be prepared from the appropriately diluted standard solution in the measurement vessel itself by adding the reagents for the pre-reduction (see 6.1.3).

Prepare fresh calibration solutions daily.

The following procedure is recommended for the preparation of standard and calibration solutions: pour some water into the volumetric flask and add the requisite amount of acid. After cooling to room temperature, add the stock or standard solution using a pipette and dilute to the mark with water.

4.16 Zero member compensation solution, containing water and acid in a concentration equal to that in the sample solution.

5 Apparatus and equipment

5.1 General

To minimise the contamination, all apparatus that come into direct contact with the sample and the solutions shall be carefully pre-treated according to EN 13804.

5.2 Atomic absorption spectrometer, with measurement recording system and accessories for the hydride generation method.

5.3 Element-specific lamp (hollow-cathode or electrodeless discharge lamp) for arsenic.

5.4 Centrifuge

5.5 Syringe filter (unit), pore size 0,45 µm, diameter 25 mm, resistant to hydrochloric acid (4.4). Membranes of polyester or nylon have been proven suitable.

5.6 Indicator paper

5.7 Device for thermostating, at approximately 37 °C.

5.8 Stirrer or shaking machine

6 Procedure

6.1 Sample preparation

6.1.1 General

It is possible that extracted arsenic compounds are decomposed to inorganic arsenic, even when stored in a refrigerator. Therefore, determination by hydride generation AAS should be conducted as soon as possible, latest within one week.

6.1.2 Hydrochloric acid extraction

In imitation of EN 71-3 the well homogenized sample is weighed (minimum weighed portion 0,2 g) into a vessel, which is suitable for the extraction and allows sufficient agitation motion. The ratio of weighed portion to extracting agent (extractant) shall be 1:50 (1 part + 49 parts). Add the appropriate amount of hydrochloric acid solution (4.3) of approximately 37 °C to the sample and mix for 1 min. Transfer one drop of this mixture onto indicator paper. If the pH is more than 1,5, add dropwise hydrochloric acid (4.4) while stirring until the pH value lies between 1,0 and 1,5. Continuously agitate (by stirring or shaking) the suspension at a temperature of approximately 37 °C for 1 h and then allow it to stand for 1 h at approximately 37 °C. Immediately afterwards solids shall be separated from the solution. First centrifuge for 10 min and then filter through a syringe filter (5.5). The extract shall be free of particles. The concentration of arsenic in the solution should be measured by hydride generation AAS as soon as possible. The extraction solution is stored in a suitable vessel in a refrigerator until measurement.

6.1.3 Pre-reduction

Depending on the hydride system used, it may be necessary to use larger or smaller volumes than described below. The ratios specified shall, however, be maintained.

Introduce and thoroughly mix 2 ml of calibration solution (4.15) and 2 ml of hydrochloric acid (4.2) into the measurement vessel of the hydride system. Then add 1 ml of potassium iodide/ascorbic acid solution (4.11) and again mix thoroughly. After leaving for 45 min at room temperature in an open vessel, dilute to 10 ml with water and mix thoroughly to obtain a solution ready for measuring. If the calibration solution is prepared in the measurement vessel itself, use the appropriate quantity of standard solution and dilute to 2 ml with the zero member compensation solution (4.16), then proceed as described above.

Treat the zero member compensation and the sample solutions in the same way. Up to 2 ml hydrochloric acid extract according to 6.1.2 are used for the pre-reduction. If necessary, the dilutions are made with zero member compensation solution (4.16) prior to the pre-reduction. Compensate using less than 2 ml of sample solution by adding the appropriate amount of zero member compensation solution (4.16).