



# SLOVENSKI STANDARD

## SIST EN 14627:2005

01-september-2005

Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Gesamtarsen und Selen mit Atomabsorptionsspektrometrie-Hydridtechnik (HGAAS) nach Druckaufschluss

Produits alimentaires - Dosage des éléments traces - Dosage de l'arsenic total et du sélénium par spectrométrie par absorption atomique par génération d'hydrures (SAAGH) après digestion sous pression

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**Ta slovenski standard je istoveten z: EN 14627:2005**

### **ICS:**

67.050

Splošne preskusne in  
analizne metode za živilske  
proizvode

General methods of tests and  
analysis for food products

**SIST EN 14627:2005**

**en**

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ICS 67.050

English version

**Foodstuffs - Determination of trace elements - Determination of total arsenic and selenium by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion**

Produits alimentaires - Dosage des éléments traces -  
Dosage de l'arsenic total et du sélénium par spectrométrie  
par absorption atomique par génération d'hydrures  
(SAAGH) après digestion sous pression

Lebensmittel - Bestimmung von Elementspuren -  
Bestimmung von Gesamtarsen und Selen mit  
Atomabsorptionsspektrometrie-Hydridtechnik (HGAAS)  
nach Druckaufschluss

This European Standard was approved by CEN on 15 March 2005.

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.

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.

CEN members are the national standards bodies of 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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## Foreword

This document (EN 14627:2005) 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 October 2005, and conflicting national standards shall be withdrawn at the latest by October 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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## EN 14627:2005 (E)

### 1 Scope

This European Standard specifies two methods: one method for the determination of total arsenic and another method for the determination of selenium in foodstuffs by hydride generation atomic absorption spectrometry (HGAAS) after pressure digestion.

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

For the determination of total arsenic in high concentration in e.g. seafood, the method specified in EN 14332 can be used.

### 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

#### 3.1 General

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Determination of the element to be determined in the test solution by HGAAS after pressure digestion according to EN 13805.

**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 health and safety practices and determine the applicability or regulatory limitations prior to use.**

#### 3.2 Determination of total arsenic

Arsenic ions are converted to arsenic hydride by sodium borohydride in an acid solution. Arsenic hydride is transferred by a gas stream into a heated measurement cell and decomposed. Arsenic is determined at 193,7 nm.

Since arsenic (III) and arsenic (V) show a different sensitivity with the hydride technique, it is necessary to reduce arsenic (V) to arsenic (III) in order to avoid incorrect measurements.

A digestion temperature up to 320 °C may be necessary in order to enable the hydridisation of the arsenic compounds and in order to determine all different arsenic species in the foodstuff.

NOTE This known fact in the case of materials of marine origin was additionally observed for Rice powder and Kidney powder during the inter-laboratory tests for this procedure.

#### 3.3 Determination of selenium

Selenium ions are converted to selenium hydride by sodium borohydride in an acid solution. This selenium hydride is transferred by a gas stream into a heated measurement cell and decomposed. Selenium is determined at 196,0 nm.

Selenium (VI) is not determined by the hydridisation as described here. It is therefore necessary to adjust the digestion conditions to yield only selenium (IV). Digestion temperatures greater than 280 °C can lead to increased formation of selenium (VI).

## 4 Reagents

### 4.1 General

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

Commercially available stock standard solutions should preferably be accompanied by a certificate (see 4.2.8 and 4.3.6).

### 4.2 Reagents for the determination of arsenic

**4.2.1 Hydrochloric acid**, 30 %, (mass concentration),  $\rho(\text{HCl}) = 1,15 \text{ g/ml}$ .

**4.2.2 Nitric acid**, at least 65 %, of approximately  $\rho(\text{HNO}_3) = 1,4 \text{ g/ml}$ .

**4.2.3 Diluted nitric acid**, mix nitric acid (4.2.2) and water in a proportion of 1 + 1 parts by volume.

**4.2.4 Sodium borohydride solution**, e.g. substance concentration  $c = 2 \text{ g/l}$  (if, for example, the flow injection procedure is used, see 6.3.1 (3)).

Dissolve 2 g of sodium hydroxide pellets in water, add 2 g of sodium borohydride and dilute to 1000 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.

**4.2.5 Diluted hydrochloric acid**, e.g. mass fraction  $w = \text{about } 3 \%$  (carrier solution, only for use in the flow injection procedure).

Dilute approximately 90 ml of hydrochloric acid (4.2.1) to 1000 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.

### 4.2.6 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.

### 4.2.7 Hydroxyl ammonium chloride solution

Dissolve 10 g of hydroxyl ammonium chloride in 100 ml of water.

**4.2.8 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 ( $\text{As}_2\text{O}_3$ ) in 25 ml of potassium hydroxide solution ( $c = 20 \text{ g/100 ml}$ ), neutralize with 20 % (mass fraction) sulfuric acid with

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phenolphthalein as indicator and dilute to 1000 ml with 1 % (mass fraction) sulfuric acid.

**4.2.9 Arsenic standard solutions**

Dilute the arsenic stock solution (4.2.8) in several steps. The arsenic standard solutions shall contain an adequate amount of hydrochloric acid, e.g. 2 ml of hydrochloric acid (4.2.1) 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 concentration) hydrochloric acid is stable for at least one week.

**4.2.10 Arsenic calibration solutions**

Prepare five calibration solutions in the required calibration range from the standard solution of 0,1 mg/l (4.2.9), 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:

$$0,1 \text{ mg/l} \xrightarrow{1/100} 1 \text{ µg/l}$$

$$\xrightarrow{3/100} 3 \text{ µg/l}$$

$$\xrightarrow{5/100} 5 \text{ µg/l}$$

$$\xrightarrow{8/100} 8 \text{ µg/l}$$

$$\xrightarrow{10/100} 10 \text{ µg/l}$$

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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 6.1.2).

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.

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

**4.3 Reagents for the determination of selenium**

**4.3.1 Hydrochloric acid**, 25 % (mass concentration),  $\rho(\text{HCl}) = 1,13 \text{ g/ml}$ .

**4.3.2 Nitric acid**, see 4.2.2.



**4.3.3 Diluted nitric acid**, see 4.2.3.

**4.3.4 Sodium borohydride solution**, see 4.2.4.

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

**4.3.5 Diluted hydrochloric acid**, e.g.  $w =$  about 3 % (carrier solution, only for use in the flow injection procedure).

Dilute approximately 110 ml of hydrochloric acid (4.3.1) to 1000 ml with water.

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

**4.3.6 Selenium stock solution**, with a selenium concentration of 1000 mg/l.

If commercial stock solutions are not available, proceed as follows: Dissolve 1,405 g of selenium dioxide ( $\text{SeO}_2$ ) and 2 g of sodium hydroxide in approximately 50 ml water, and dilute to 1000 ml with water.

**4.3.7 Selenium standard solution**

Dilute the stock solution (4.3.6) in several steps. The solutions shall contain an adequate amount of hydrochloric acid, e.g. 2 ml of hydrochloric acid (4.3.1) 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 selenium in 0,5 % (mass fraction) hydrochloric acid is stable for at least one week.

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**4.3.8 Selenium calibration solutions**

Prepare five calibration solutions in the required calibration range from the standard solution of 0,1 mg/l (4.3.7), 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  $\mu\text{g/l}$  to 10  $\mu\text{g/l}$  range:

$$\begin{aligned} 0,1 \text{ mg/l} &\xrightarrow{1/100} 1 \mu\text{g/l} \\ &\xrightarrow{3/100} 3 \mu\text{g/l} \\ &\xrightarrow{5/100} 5 \mu\text{g/l} \\ &\xrightarrow{8/100} 8 \mu\text{g/l} \\ &\xrightarrow{10/100} 10 \mu\text{g/l} \end{aligned}$$

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.