SLOVENSKI STANDARD
SIST EN 17266:2020

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Živila - Določevanje elementov in njihovih kemijskih oblik - Določevanje organskih živosrebrovih spojin v morski hrani z analizo elementarnega živega srebra

Foodstuffs - Determination elements and their chemical species - Determination of organomercury in seafood by elemental mercury analysis

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Organoquecksilber in Fisch- und Meeresfrüchten mit Feststoffquecksilberbestimmung

Produits alimentaires - Dosage des éléments et de leurs espèces chimiques - Dosage du mercure organique dans les fruits de mer par analyse du mercure élémentaire

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67.120.30	Ribe in ribji proizvodi	Fish and fishery products

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Foodstuffs - Determination elements and their chemical species - Determination of organomercury in seafood by elemental mercury analysis

Produits alimentaires - Dosage des éléments et de leurs espèces chimiques - Dosage du mercure organique dans les produits de la mer par analyse du mercure élémentaire

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Organoquecksilber in Fisch und Meeresfrüchten mittels Elementaranalyse von Quecksilber

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European foreword

This document (EN 17266:2019) 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of an identical text or by endorsement, at the latest by May 2020, and conflicting national standards shall be withdrawn at the latest by May 2020.

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1 Scope

This document specifies a method for the determination of organomercury in seafood by elemental mercury analysis. The method has been successfully validated in an interlaboratory study on oyster tissue, mussel tissue, lobster hepatopancreas, dogfish liver and tuna at levels from 0,01 mg/kg to 5 mg/kg referring to dry weight and expressed as mercury [1], [2].

The limit of quantification is approximately 0,01 mg/kg of organomercury [3], [4].

Organic species of mercury, other than monomethylmercury, are also extracted and thus determined with this method. However, in seafood the contribution from organic species of mercury other than monomethylmercury is negligible.

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 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:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Organomercury in seafood/fishery products is separated from the matrix by double liquid-liquid extraction, first with an organic solvent (toluene) and subsequently with L-cysteine solution and is determined by elemental mercury analyser [3], [4].

Elemental mercury analyser, also known as automated or direct mercury analyser, is a single purpose atomic absorption spectrophotometer for mercury determination. The determination of mercury with an elemental mercury analyser is based on sample drying and subsequent thermal decomposition, including electro thermal atomisation of mercury. A gold amalgamator selectively traps and pre-concentrates the mercury from the flow of decomposition products. Finally, the trapped mercury is thermally released and detected by atomic absorption at 253,7 nm. Organomercury results are expressed in mg/kg as mercury.

Alternative detection techniques can be used, provided that equivalence of method performance is proven.

5 Reagents

The mass concentration of mercury in the reagents and water used shall be low enough not to affect the results. All reagents shall be of analytical grade, i.e. pro analysis, p.a. or similar unless otherwise specified.

Use water conforming to grade 2 of EN ISO 3696.

5.1 Nitric acid, mass fraction, w (HNO_3) = 65 % (m/m), density approximately 1,4 g/ml

5.2 Hydrochloric acid, w (HCl) = 32 % (m/m), density approximately 1,18 g/ml

5.3 Diluted hydrochloric acid solution

Mix equal volumes of hydrochloric acid (5.2) and water.

5.4 Hydrobromic acid, w (HBr) approximately 47 %, density approximately 1,47 g/ml

5.5 Toluene

5.6 L-cysteine monohydrate hydrochloride, e.g. Ph. Eur. or USP grade[1)]

5.7 Sodium sulfate anhydrous

5.8 Sodium acetate anhydrous

5.9 L-cysteine solution, mass concentration ρ = 1 g/100 ml

Weigh 1,0 g of L-cysteine monohydrate hydrochloride (5.6), 12,5 g of sodium sulfate (5.7) and 0,8 g of sodium acetate (5.8) into a 100 ml beaker. Add about 75 ml of water and stir until complete dissolution. Transfer this solution completely to a 100 ml volumetric flask and make up to volume with water. This solution can be stored for 1 day at ambient temperature. Other preparation volumes may be used as long as proportions are kept.

The mass concentration of mercury in the L-cysteine solution should be as low as possible. The purity of this solution should be such that the response for mercury shall be less than half the response of the 1 µg/l mercury standard solution (calibration solution 1, see 5.13).

L-cysteine precipitates on the catalytic tube which should thus be changed or cleaned as appropriate.

5.10 Mercury stock solution, ρ(Hg) = 1 000 mg/l Hg

5.11 Monomethylmercury (MMHg) chloride, minimum purity of 95 %

5.12 Mercury standard solutions

5.12.1 Mercury standard solution 1 (ρ = 10 mg/l Hg)

Pipette 1,0 ml of the commercial mercury stock solution 1 000 mg/l Hg (5.10) in a 100 ml volumetric flask, add 2,0 ml of diluted hydrochloric acid solution (5.3) and make up to volume with water. This solution is stable in a glass container in the refrigerator at approximately 2 °C to 10 °C for 6 months.

5.12.2 Mercury standard solution 2 (ρ = 0,50 mg/l Hg)

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1) Ph. Eur. = European Pharmacopoeia; USP = United States Pharmacopoeia.

Pipette 2,5 ml of mercury standard solution 1 (5.12.1) in a 50 ml volumetric flask, add 1,0 ml of diluted hydrochloric acid solution (5.3) and make up to volume with water. This solution is stable in a glass container in the refrigerator at approximately 2 °C to 10 °C for 2 months.

5.13 Calibration solutions

Due to the highly stable response of elemental mercury analysers, there is no need to recalibrate for each analytical sequence. Calibration is usually stable for at least 1 year. For that reason every instrumental calibration will be maintained for that period, provided that quality controls for each sequence are satisfactory. Nevertheless, if the gold amalgamator or catalyst tube is changed, response could change and a new calibration is necessary. In such a case, analyse 500 µl of each calibration solution (from blank to 100 µg/l Hg). For selection of corresponding calibration solutions for each calibration curve, see 7.4. For acceptance criteria regarding the calibration curve, see 8.3.

Select calibration solutions depending on the cell used, thus on the expected concentration level of the sample. This is comprehensively described in 7.4.

Prepare all calibration solutions freshly for each calibration.

Other volumes of preparation are suitable provided that they maintain the proportions described below. Pipette either mercury standard solution 2 (5.12.2) or calibration solution 7 (see Table 1) in a 50 ml volumetric flask and fill up to the mark with L-cysteine solution (5.9) according to the scheme presented in Table 1.

Use L-cysteine solution (5.9) as blank (level 0) for calibration.

Table 1 — Example of calibration solutions

Calibration solution no	Initial solution	Volume of initial solution ml	Final Hg concentration (ρ) µg/l
0	L-cysteine solution (5.9)	50	0
1	calibration solution no 7	1,0	1,0
2	calibration solution no 7	2,5	2,5
3	standard solution 2 (5.12.2)	0,5	5,0
4	standard solution 2 (5.12.2)	1,0	10
5	standard solution 2 (5.12.2)	1,5	15
6	standard solution 2 (5.12.2)	2,5	25
7	standard solution 2 (5.12.2)	5,0	50
8	standard solution 2 (5.12.2)	7,5	75
9	standard solution 2 (5.12.2)	10,0	100

5.14 Internal quality control solutions

5.14.1 General

As the response of elemental mercury analysers is highly stable, there is no need to recalibrate the instrument for each analytical sequence. However, some control solutions are used to ensure the validity of the latest calibration. Each calibration curve needs to be compared against an external solution to demonstrate absence of error in intermediate calibration solutions preparation.

Quality control solution 2 (QC2) ensures that quantification at low level is still correct. Quality control solution 1 (QC1) demonstrates that the response is stable at high concentrations and that no uncontrolled drifts are detected, since QC1 is placed at the end of the analytical sequence.

As an alternative the analysis of a reference material (RM) may be used.

5.14.2 Quality control solution 1 (QC1), (ρ = 50 µg/l Hg)

Pipette 5,0 ml of mercury standard solution 2 (5.12.2) in a 50 ml volumetric flask and make up to volume with the L-cysteine solution (5.9). Prepare freshly for each analytical sequence.

The following alternative options for the preparation of QC1 are provided as examples:

— QC1 may be prepared using intermediate solutions prepared from brands or batches of the commercial 1 000 mg/l Hg (5.10) stock solutions different than those used for the preparation of the calibration solutions.

— QC1 may be prepared from a standard solution of MMHg (5.11). In that case, pipette 2,7 ml of spiking solution 2 (5.15.2) in a 50 ml volumetric flask and make up to volume with L-cysteine 1 % (w/v) solution (5.9).

5.14.3 Quality control solution 2 (QC2), (ρ = 1 µg/l Hg)

Pipette 1,0 ml of QC1 (5.14.2) in a 50 ml volumetric flask and make up to volume with the L-cysteine solution (5.9). Prepare freshly for each analytical sequence.

5.15 Spiking Solutions

5.15.1 Spiking solution 1 (ρ = 76,7 mg/l, expressed as Hg)

Weigh accurately 24 mg of monomethylmercury chloride (5.11, taking into account the purity for the final concentration), in a 250 ml volumetric flask, add about 4 ml of diluted hydrochloric acid solution (5.3) and 200 ml of water. Shake thoroughly until complete solubilisation and make up to volume with water. This solution is stable in a glass container in a refrigerator (at approximately 2 °C to 10 °C) for 1 year.

5.15.2 Spiking solution 2 (ρ = 0,96 mg/l, expressed as Hg)

Pipette 625 µl of spiking solution 1 (5.15.1) in a 50 ml volumetric flask, add 1 ml of diluted hydrochloric acid solution (5.3) and make up to volume with water. This solution is stable in a glass container in a refrigerator (at approximately 2 °C to 10 °C) for 3 months.