SLOVENSKI STANDARD
oSIST prEN 13806-1:2023

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Živila - Določevanje elementov v sledovih - 1. del: Določevanje celotnega živega srebra v živilih z atomsko absorpcijsko spektrometrijo (AAS) - Tehnika hladne pare po razklopu pod pritiskom

Foodstuffs - Determination of trace elements - Part 1: Determination of total mercury in foodstuffs by atomic absorption spectrometry (AAS) - cold vapour technique after pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Teil 1: Bestimmung von Quecksilber mit Atomabsorptionsspektrometrie-(AAS-)Kaltdampftechnik nach Druckaufschluss

Produits alimentaires - Dosage des éléments traces - Partie 1 : Dosage du mercure par spectrométrie d'absorption atomique par génération de vapeurs froides après digestion sous pression

Ta slovenski standard je istoveten z: **prEN 13806-1**

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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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English Version

Foodstuffs - Determination of trace elements - Part 1: Determination of mercury by cold-vapour atomic absorption spectrometry (CVAAS) after pressure digestion

Produits alimentaires - Dosage des éléments-traces - Dosage du mercure par spectrométrie d'absorption atomique par génération de vapeurs froides après digestion sous pression

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Quecksilber mit Atomabsorptionsspektrometrie (AAS)-Kaltdampftechnik nach Druckaufschluss

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 275.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 13806-1:2023) has been prepared by Technical Committee CEN/TC 275 “Food analysis – Horizontal methods”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13806:2002.

In comparison with the previous edition, the following technical modifications have been made:

— the document has been split up into three separate parts: EN 13806-1 covering the AAS-cold-vapour technique, EN 13806-2[1] the AFS-cold-vapour technique and EN 13806-3[2] the solid sample AAS technique;

— full technical revision to bring the technical realization up to date with the latest technology;

— Stabilization of the digest solution;

— Update of statistical data by new collaborative study;

— full editorial revision.

This document was developed by the “Element Analysis” working group of the Federal Office of Consumer Protection and Food Safety (BVL) according to the German Food and Feed Act, Paragraph 64.

[1] Under preparation. Stage at the time of publication: pr EN 13806-2:2023.
[2] Under preparation. Stage at the time of publication: pr EN 13806-3:2023.

Introduction

This document has been developed in parallel with EN 13806-2:—[1] [1] and EN 13806-3:—[2] [2]. All three methods were validated in parallel in collaborative studies with the same scope. They are statistically compatible in performances. This allows the users of these documents to employ the most appropriate/available method depending on the purpose of their studies. The statistical parameters of these standards are presented in the respective documents.

1 Scope

This document specifies a method for the determination of total mercury in foodstuffs by cold vapour atomic absorption spectrometry (AAS) after pressure digestion.

This method was tested in a collaborative study carried out in connection with the pressure digestion method EN 13805 on seven different materials with a mercury concentration in the range from 0,005 mg/kg to 5,06 mg/kg and successfully validated in the range from 0,015 mg/kg to 5,06 mg/kg.

The following foodstuffs were analysed:

— Saithe (dried);

— Celery (dried);

— Wheat noodle powder;

— Wild mushrooms (dried);

— Pig liver (dried);

— Cacao powder;

— Tuna fish (dried).

The lower limit of the method's applicability varies depending on the food matrix and the water content of the foodstuff. It is a laboratory-specific value and is defined by the laboratory when calculating the limit of quantification (see 9.2).

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 13804:2013, *Foodstuffs - Determination of elements and their chemical species - General considerations and specific requirements*

EN 13805:2014, *Foodstuffs - Determination of trace elements - Pressure digestion*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

Mercury (Hg) is determined by AAS-cold-vapour technique in the test solution obtained by performing the pressure digestion process described in EN 13805:2014.

The test solution is transferred into the reaction vessel of the mercury analyser to reduce the oxidized forms of mercury to elemental mercury with bivalent tin or sodium borohydride. The elemental mercury is then flushed into the cuvette of the AAS instrument using a carrier gas stream. The absorption at the mercury line of 253,7 nm is used to determine the mercury concentration. In case of very small quantities of mercury in the test solution, it is recommended to use a technique capable to concentrate the stripped mercury on a gold/platinum gauze (amalgam technique) prior to determination.

The total content of mercury is understood as the content measured using this described method. It is indicated in mg/kg or mg/l, depending on the sample type.

WARNING 1 — The use of this method can involve the application of dangerous substances, actions or equipment. Nevertheless, the method description cannot mention all dangers possibly involved in its application. Each operator of the method is responsible for taking the appropriate safety precautions and to respect the corresponding regulations.

5 Reagents

5.1 General

Unless otherwise specified, “solutions” are understood to be aqueous solutions.

The content of mercury in the chemicals and water shall be low enough not to affect the results.

5.2 Hydrochloric acid, ω = 30 % to 37 %.

5.3 Nitric acid, ω = 65 % to 69 %.

5.4 Diluted nitric acid.

Nitric acid (5.3) + water, V_1 + V_2, approx. 1 + 9.

5.5 Reducing agent.

5.5.1 General

Tin(II)chloride or sodium borohydride can be used to reduce the oxidized forms of mercury to elemental mercury (Hg^0). However, the alternate use of both reagents is not recommended. The mass concentrations of the reducing agent solutions can vary depending on the system. Follow the respective information of the instrument manufacturer.

5.5.2 Tin(II) chloride solution, e.g. ϱ = 100 g/l.

Dissolve 50 g of tin(II) chloride, $SnCl_2 \bullet 2\ H_20$, in approx. 100 ml of hydrochloric acid (5.2) in a 500 ml volumetric flask and fill up to the mark with water.

The solution shall be prepared freshly before use.

NOTE 1 The required concentration of tin(II) chloride depends on the analysis system and the concentration of nitric acid in the test solution. Too low concentrations of tin(II) chloride could lead to insufficient recoveries.

NOTE 2 Tin(II) chloride ages quickly, visible in light yellow precipitates of tin(IV) oxides. In case of higher concentrations of tin(II) chloride, these precipitates occur more frequently.

NOTE 3 If necessary, the tin(II) chloride solution can be flushed with argon gas in order to remove any possible traces of mercury.

5.5.3 Sodium borohydride solution, e.g. at a concentration of ϱ = 3 g/l.

Dissolve 1,5 g of sodium borohydride together with 0,5 g of sodium hydroxide pellets in water in a 500 ml volumetric flask and fill up to the mark.

This solution shall be prepared freshly and filtered before use.

WARNING — It is imperative to observe the safety instructions when working with sodium borohydride. In combination with acids, sodium borohydride produces hydrogen. This can lead to an explosive mix of air and hydrogen. A fume hood shall be available.

5.6 Carrier solution

Diluted hydrochloric acid (prepared from 5.2, e.g. ω = 3 %) is used as the carrier solution.

The mass concentration of the carrier solution can vary depending on the instrument. Observe the respective information of the instrument manufacturer.

5.7 Stabilization

The standard, calibration, zero-point and sample digestion solutions are stabilized with hydrochloric acid (5.2). For hydrochloric acid, it is recommended to use a sufficient concentration. In the validation study, the mass fraction of approx. 1 % in the solution was adjusted. An example is provided in Table 1 and Table 2.

Alternatively, other stabilizing reagents may be used (see Annex B).

5.8 Mercury stock solution, with a concentration of ϱ = 1 000 mg/l.

The stock solution is commercially available. It is recommended to use certified stock solutions.

5.9 Mercury standard solution

The standard solutions are prepared from the stock solution (5.8) in several dilution steps.

For this purpose, fill approx. 20 ml of water into a 100 ml volumetric flask, add the necessary amount of stabilizing reagent (5.7) and mix. After the mixture has cooled down to room temperature, the stock (5.7) or standard solution (see Table 1) is added into the flask and filled-up to the mark with water.

The standard solutions are stable for at least one month.

Table 1 — Example for the preparation of Hg standard solutions in 100 ml volumetric flasks

Standard solution [Hg concentration]	**Stabilization**	**Initial solution**	**Fill up to the mark with water to**
Standard solution 1 [10 mg/l]	3 ml hydrochloric acid (5.2)	1 ml Hg stock solution (5.8)	100 ml
Standard solution 2 [0,1 mg/l]	3 ml hydrochloric acid (5.2)	1 ml standard solution 1	100 ml

5.10 Zero-point solution

Fill approx. 20 ml of water into a 100 ml volumetric flask, add the necessary amounts of stabilizing and digestion reagents (e.g. 3 ml of hydrochloric acid (5.2) and 10 ml of nitric acid (5.3)), mix and fill up to the mark with water.

The zero-point solution is used as calibration blank (see Table 2) and might be used for further dilutions of the digestion solutions in case of excessive concentrations in the samples.

5.11 Calibration solutions

The acid concentrations in the calibration solutions and in the zero-point solution shall correspond to the acid concentration in the test solution. Even at higher concentrations, mercury calibration solutions do not remain stable for a long time and therefore shall be prepared freshly every working day. If possible, the concentrations of the calibration solutions are to be selected in such a way that the linear range of the calibration function is not exceeded. It is recommended to use at least three calibration solutions with different concentrations (see examples in Table 2).

For this purpose, fill approx. 20 ml of water into a 100 ml volumetric flask, add the necessary amounts of stabilizing and digestion reagents according to Table 2 and mix. After the mixture has cooled down to room temperature, add standard solution 2 in accordance with Table 2 into the flask and fill up to the mark with water.