



SLOVENSKI STANDARD

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Živila - Določevanje elementov v sledovih - 3. del: Določevanje celotnega živega srebra v živilih z atomsko absorpcijo neposredno iz živila (elementarna analiza živega srebra)

Foodstuffs - Determination of trace elements - Part 3: Determination of total mercury in foodstuffs with atomic absorption directly from the foodstuff (elemental mercury analysis)

Lebensmittel - Bestimmung von Elementspuren - Teil 3: Bestimmung des Gesamtquecksilbers in Lebensmitteln durch Atomabsorption direkt aus dem Lebensmittel (Elementare Quecksilberanalyse)

Produits alimentaires - Dosage des éléments traces - Partie 3 : Dosage du mercure total dans les produits alimentaires par absorption atomique directe (analyse du mercure élémentaire)

Ta slovenski standard je istoveten z: prEN 13806-3

ICS:

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 3:
Determination of total mercury in foodstuffs with atomic
absorption directly from the foodstuff (elemental mercury
analysis)**

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 13806-3:2023) 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.

The main changes compared to the previous edition are listed below:

- the document has been split up into three separate parts: EN 13806-1¹ covering the AAS-cold-vapour technique, EN 13806-2² the AFS-cold-vapour technique and EN 13806-3 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.

[oSIST prEN 13806-3:2023](https://standards.iteh.ai/catalog/standards/sist/cb5fb57f-8aa5-4129-918d-707c7636928f/osist-pren-13806-3-2023)

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¹ Under preparation. Stage at the time of publication: prEN 13806-1:2023.

² Under preparation. Stage at the time of publication: prEN 13806-2:2023.

Introduction

This document has been developed in parallel with EN 13806-1:—¹[2] and EN 13806-2:—²[3]. 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.

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1 Scope

This document specifies a method for the determination of total mercury (Hg) in foodstuffs using direct atomic absorption spectrometry after thermal decomposition in an oxygen or air flow and concentration by amalgam formation. The method is applicable for solid and liquid samples.

This method was tested in a collaborative study carried out on seven different materials with a mercury concentration in the range from 0,005 mg/kg to 5,20 mg/kg and successfully validated in this range.

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*

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>

prEN 13806-3:2023 (E)**4 Principle**

The samples to be analysed are weighed directly - without pressure digestion - into suitable sample containers and are thermally decomposed in the analyser system in an oxygen atmosphere at approx. 700 °C. The resulting reaction gases are catalytically cleaned together with the mercury vapour, and the mercury is concentrated on a consecutive amalgamator. Subsequently, the mercury is released as atomic vapour by fast heating up of the amalgamator, transferred into a system of measurement cuvettes and quantified by absorption at 253,7 nm.

By the simultaneous use of measurement cuvettes of different lengths, a dynamic measurement range over several orders of magnitude can be achieved.

The total content of mercury is understood as the content measured using this document. It is indicated in mg/kg.

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, $\omega = 30\%$ to 37% , density = approx. 1,15 g/ml.

5.3 Nitric acid, $\omega = 65\%$ to 69% , density = approx. 1,4 g/ml.

5.4 Diluted nitric acid

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

5.5 Stabilization

The standard, calibration and zero-point solutions are stabilized with hydrochloric acid (5.2). It is recommended to adjust the hydrochloric acid concentration to approx. $\omega = 1\%$ in the solutions.

Alternatively, other stabilizing reagents may be used (for details see the instructions of the instrument manufacturer or [2]).

5.6 Mercury stock solution, e.g. $\rho = 1\,000$ mg/l.

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

5.7 Mercury standard solution

The standard solution is prepared from the stock solution (5.6) by means of dilution (e.g. see Table 1).

For this purpose, fill approx. 5 ml of water into a 20 ml volumetric flask, add the necessary amount of stabilizing acid (e.g. 0,6 ml hydrochloric acid (5.2)) and mix. After the mixture has cooled down to room temperature, add the stock solution (5.6) and fill up to the mark with water.

The standard solution is stable for at least one month.

Table 1 — Example for the preparation of Hg standard solution in a 20 ml volumetric flask

Standard solution [Hg concentration]	Stabilization	Initial solution	Fill up to the mark with water to
Standard solution [10 mg/l]	600 µl hydrochloric acid (5.2)	200 µl Hg stock solution (5.6)	20 ml

5.8 Mercury calibration solutions and zero-point solution

To produce the examples of calibration solutions and zero-point solution described below, a 20 ml volumetric flask is filled with approx. 5 ml of water, the necessary amount of stabilizing acid added according to Table 2, and mixed. After the mixture has cooled down to room temperature, pipette the standard solution/calibration solution into the flask and fill up to the mark with water.

Table 2 — Example for the preparation of the Hg calibration solutions and zero-point solution in 20 ml volumetric flasks

Zero-point/ calibration solution [Hg concentration]	Stabilization	Standard solution/ calibration solution	Fill up to the mark with water to
Zero-point solution	600 µl hydrochloric acid (5.2)	-	20 ml
Calibration solution 1 [1 000 µg/l]	600 µl hydrochloric acid (5.2)	2 000 µl standard solution (5.7)	20 ml
Calibration solution 2 [100 µg/l]	600 µl hydrochloric acid (5.2)	2 000 µl calibration solution 1	20 ml
Calibration solution 3 [10 µg/l]	600 µl hydrochloric acid (5.2)	2 000 µl calibration solution 2	20 ml
Calibration solution 4 [1 µg/l]	600 µl hydrochloric acid (5.2)	2 000 µl calibration solution 3	20 ml

The mercury calibration solutions and the zero-point solution are used to set up the calibration of the instrument as described under 8.2.3.2. For this purpose, they shall be prepared fresh.

NOTE Alternatively, appropriate, certified reference materials can be used for calibration.

5.9 Oxygen or air

The manufacturer's instructions shall be taken into account, e.g. for the analytical purity.

6 Apparatus

6.1 General

All equipment and labware that come into direct contact with the sample and the solutions used, shall be carefully pre-treated/cleaned according to EN 13804:2013 to minimize contamination.

For the cleaning of glass and quartz glass equipment the following steps in particular are recommended: rinsing with tap water, treating with a detergent solution, rinsing again with tap water and immersing overnight or longer in diluted nitric acid (5.4). Finally, rinse the equipment with high-purity water and dry them before use. Steam stripping with nitric acid is an effective cleaning method and is used regularly in element trace analysis.

When preparing solutions, special care shall be taken to avoid contaminations. The volumetric flasks used for the preparation of the solutions should be made of glass or quartz glass.

Due to the strong adsorption behaviour of mercury on vessel walls it is recommended to always reuse the same flasks for each concentration of standard and calibration solution. Nevertheless, they shall be cleaned before each use.

6.2 Elemental mercury analyser

Elemental mercury analyser with instrument control unit and sample containers made of suitable materials (e.g. nickel, quartz glass or ceramic). The sample containers usually have a capacity of approx. 500 mg of solid sample or 500 µl, 1 000 µl and 1 500 µl of liquids. The instructions in Annex B shall be observed.

6.3 Analytical balance, readability of at least 0,000 1 g.

For an automated transfer of the sample weights, it is recommended to link the balance to the Hg-analyser's control unit (6.2) via a data interface.

7 Sampling

The sampling procedure is not part of the analytical method described in this document.

Sampling shall be carried out in such a way that there is no loss of or contamination with mercury.

8 Procedure

8.1 Preparations for measurement

8.1.1 Sample carrier cleaning

Mechanically remove any residues adhering to the sample containers. Sample containers made of quartz glass may be pre-cleaned with diluted nitric acid (5.4) according to EN 13804:2013. Sample containers made from nickel are only cleaned of adhering residues mechanically.

Subsequently, rinse the sample carriers with water and decontaminate them in a muffle furnace according to the manufacturers' instructions, e.g. at approx. 600 °C for at least 15 min. Alternatively, the sample carriers, without sample weight, could be decontaminated by measuring them in the mercury analyser by the sample program.

After cleaning, store the sample carriers in a dust-protected place (e.g. in a covered glass petri dish).

Irrespective of the material they are made of, carefully cleaned sample carriers do not show any memory effects.

8.1.2 Sample weight

The sample prepared according to EN 13804:2013 is weighted directly in the pre-cleaned sample carrier. The maximum sample quantity depends on the capacity of the sample carrier, the type of sample material and the expected mercury content (further information under B.2). If the measured absorption value is outside the calibration range, repeat the analysis with a lower sample weight. With small quantities attention shall be paid to the homogeneity of the sample and the lowest possible sample weight of the analytical balance. Solid samples cannot be diluted.

Typical sample weights for sample containers with a volume of approx. 0,5 ml:

Liquid, pasty samples (e.g. milk, meat, fruit)	0,1 g to 0,5 g
Powdery samples (e.g. flour)	0,05 g to 0,15 g
Samples with high fat content (e.g. nuts, margarine)	0,05 g to 0,1 g

When analysing samples with high water content, the sample weight may be increased to 0,5 g to 1 g. For these samples preferably use sample containers with a capacity of 1 ml to 1,5 ml.

NOTE 1 Liquid samples can be applied, for example, to approx. 50 mg of a substrate with low mercury content (e.g. sea sand, alumina, starch or flour with contents < 0,001 ng of mercury absolute).

Liquid samples should also be weighed in, to prevent insufficient inaccuracies due to sample viscosity and dosing inaccuracies due to the sample type.

NOTE 2 Samples with high fat content can be coated with a substrate with low mercury content (e.g. sea sand or alumina with contents < 0,001 ng of mercury absolute), in order to prevent them from squirting in the furnace during the drying process. Alternatively, furnace programmes with slow heating rates can be used (see 8.2.4, Table 5).

8.2 Direct atomic absorption spectrometry using thermal decomposition

8.2.1 General

Follow the manufacturer's instructions with regard to the initial operation, stabilizing time and measurement with the elemental mercury analyser.

8.2.2 Instrument blank value

For solid sample systems the instrument blank value (system blank value originating from measurements of cleaned sample carriers) is determined every working day, and is deducted from all subsequent quantifications.

To determine the instrument blank value, measure cleaned sample carriers (8.1.1) in the elemental mercury analyser. If the absorptions are above a set value, e.g. 0,01, the sample carrier shall be cleaned again. Discard the blank values measured after switching on and stabilizing the instrument, e.g. 1 to 3. Compare the following, outlier-free blank values (e.g. 4 to 6) to the instrument's currently set system blank value, and, in case of significant deviations (absolute difference of the absorption greater than approx. 30 % of the set system blank value), factor them into the system blank value. This procedure is described in detail in the manufacturer's handbook.

The instrument blank value changes depending on the mercury concentrations measured before (memory effect of the system). After measuring samples with high mercury concentrations, carry out a sufficient number of cleaning cycles in order to obtain a representative instrument blank value.