
Živila - Določevanje elementov v sledovih - 2. del: Določevanje celotnega živega srebra v živilih z atomsko florescenčno spektrometrijo (AFS) - Tehnika hladne pare po razklopu pod pritiskom

Foodstuffs - Determination of trace elements - Part 2: Determination of total mercury in foodstuffs by atomic fluorescence spectrometry (AFS) - Cold vapour technique after pressure digestion

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Produits alimentaires - Dosage des éléments traces - Partie 2 : Dosage du mercure total dans les produits alimentaires par spectrométrie de fluorescence atomique (SFA) - Génération de vapeurs froides après digestion sous pression

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General methods of tests and
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**Foodstuffs - Determination of trace elements - Part 2:
Determination of total mercury in foodstuffs by atomic
fluorescence spectrometry (AFS) - Cold vapour technique
after pressure digestion**

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 foreword

This document (prEN 13806-2:2023) 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 13806-1 covering the AAS-cold-vapour technique, EN 13806-2 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 13806-2:2023](https://standards.iteh.ai/catalog/standards/sist/9003c582-55a1-48cb-b607-cfeb56dde93/osist-pren-13806-2-2023)

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Introduction

This document has been developed in parallel with EN 13806-1:2023¹[3] and EN 13806-3:2023²[4]. 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 Under preparation. Stage at the time of publication: prEN 13806-1:2023.

² Under preparation. Stage at the time of publication: prEN 13806-3:2023.

1 Scope

This document specifies a method for the determination of total mercury in foodstuffs by cold vapour atomic fluorescence spectrometry (AFS) 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 0,006 mg/kg to 5,38 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*

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 AFS-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 samples type.

WARNING — 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 Ascorbic acid solution, $\rho = 100$ g/l.

Dissolve 10 g of ascorbic acid in 100 ml of water.

The solution remains stable for a maximum of one week.

5.6 Reducing agent

5.6.1 Tin(II) chloride solution, e.g. $\rho = 100$ g/l.

Dissolve 50 g of tin(II) chloride, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, 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 measurement 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.

5.7 Carrier solution

Diluted hydrochloric acid (prepared from 5.2, e.g. $\omega = 3\%$) 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.

5.8 Stabilization

5.8.1 General

Potassium bromide/bromate reagent is recommended to stabilize the digestion, calibration, zero-point and test solutions.

The potassium bromide/bromate reagent can be produced from the respective salts. Ampoules with ready-mixed potassium bromide-potassium bromate solution are commercially available and can also be used.

Alternative methods can also be used to avoid the interference resulting from the nitrous gases (see also 8.1) and to stabilize the mercury.

5.8.2 Example for preparation of a stabilizing reagent

5.8.2.1 Potassium bromate solution, $\rho = 5,56$ g/l.

Dissolve 1,39 g of potassium bromate in 250 ml of water. A possible mercury contamination of the potassium bromate can be eliminated by heating in a muffle furnace at $250\text{ °C} \pm 20\text{ °C}$ overnight. The solution remains stable for at least one month.

5.8.2.2 Potassium bromide solution, $\rho = 23,8$ g/l.

Dissolve 5,95 g of potassium bromide in 250 ml of water. A possible mercury contamination can be eliminated by heating the potassium bromide in a muffle furnace at $300\text{ °C} \pm 20\text{ °C}$ overnight. The solution remains stable for at least one month.

5.8.2.3 Stabilizing reagent, potassium bromide/bromate reagent.

Mix equal parts of potassium bromate solution (5.8.2.1) and potassium bromide solution (5.8.2.2). 100 samples can be treated with 200 ml of reagent. The reagent remains stable for several days to weeks. This shall be checked. The solution shall be colourless.

5.9 Mercury stock solution, $\rho = 1\ 000$ mg/l.

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

5.10 Mercury standard solutions

The standard solutions are prepared from the stock solution (5.9) in several dilution steps.

For this purpose, fill approx. 20 ml of water into a 100 ml volumetric flask, add the necessary amount of hydrochloric acid (5.2) for stabilization and mix. After the mixture has cooled down to room temperature, the stock (5.9) or standard solution (see Table 1) is added into the flask and filled up to the mark with water.

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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)	1,0 ml Hg stock solution (5.9)	100 ml
Standard solution 2 [0,1 mg/l]	3 ml hydrochloric acid (5.2)	1,0 ml Standard solution 1	100 ml

5.11 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.

5.12 Calibration solution

The concentrations of the stabilizing reagent and digestion acid in the calibration solutions and zero-point solution (5.11) shall correspond to the concentrations 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.