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Foodstuffs - Determination of trace elements - Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Blei, Cadmium, Chrom und Molybdän mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss
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Produits alimentaires - Dosage des éléments traces - Dosage du plomb, du cadmium, du chrome et molybdène par spectrométrie d'absorption atomique en four graphite après digestion sous pression

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analizne metode za živilske
proizvode

General methods of tests and
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

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English version

Foodstuffs - Determination of trace elements - Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion

Produits alimentaires - Dosage des éléments traces -
Dosage du plomb, du cadmium, du chrome et molybdène
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Lebensmittel - Bestimmung von Elementspuren -
Bestimmung von Blei, Cadmium, Chrom und Molybdän mit
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Druckaufschluss

This European Standard was approved by CEN on 18 December 2002.

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 Management Centre 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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 14083:2003) 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 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of lead, cadmium, chromium and molybdenum in foodstuffs by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion.

Specific foodstuffs for which European Standards exist are excluded from the scope of this horizontal European Standard. The analyst shall review if vertical standards exist.

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2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 13804, *Foodstuffs - Determination of trace elements - Performance criteria, general considerations and sample preparation*.

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

3 Principle

Determination of the elements in the test solution by graphite furnace atomic absorption spectrometry (GFAAS) [1], [2], [4] after pressure digestion according to EN 13805.

WARNING — The use of this standard can 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 safety and health practices and determine the applicability of regulatory limitations prior to use.

4 Reagents

4.1 General

The concentration of the trace elements in the reagents and water, used shall be low enough not to affect the results of the determination.

4.2 Hydrochloric acid, not less than 25 %, (mass fraction), having a density of approximately $\rho(\text{HCl}) = 1,13 \text{ g/ml}$ and suitable for trace element analysis.

4.3 Nitric acid, not less than 65 %, (mass fraction), having a density of approximately $\rho(\text{HNO}_3) = 1,4 \text{ g/ml}$ and suitable for trace element analysis.

4.4 Stock solutions

NOTE The stock solutions for lead, cadmium, chromium and molybdenum can be prepared from metals or metal salts. They can be commercially available also. It is advisable to use certified stock solutions. The preparation of stock solutions are given as an example in the following:

4.4.1 Lead stock solution, with a lead concentration of 1 000 mg/l.

Dissolve 1,598 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in nitric acid 1% (10 ml according to 4.3 diluted with water to 1 000 ml) and make up with nitric acid 1 % to 1 000 ml.

4.4.2 Cadmium stock solution, with a cadmium concentration of 1 000 mg/l.

Dissolve 1,000 g of cadmium metal in a minimum amount of hydrochloric acid 1 % (10 ml according to 4.2 and diluted with water to 1 000 ml) and make up to 1 000 ml with 1 % (parts by volume) hydrochloric acid.

4.4.3 Chromium stock solution, with a chromium concentration of 1 000 mg/l.

Dissolve 3,735 g of potassium chromate (K_2CrO_4) in water and make up with water to 1 000 ml.

4.4.4 Molybdenum stock solution, with a molybdenum concentration of 1 000 mg/l.

Dissolve 1,840 g of ammonium heptamolybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in ammonia solution 1% (10 ml ammonia solution of min 25 %, $\rho(\text{NH}_3) = 0,91 \text{ g/ml}$, diluted with water to 1 000 ml) and make up with ammonia solution 1 % to 1 000 ml.

4.5 Calibration solutions

Dilute the stock solutions to the concentrations needed for calibration; the concentrations shall be chosen so as not to exceed the linear range of the calibration function. It is recommended to use a minimum of 3 calibration solutions with different concentrations. The concentration of acid in the calibration solutions shall be equal to that in the test solution (see EN 13805).

4.6 Zero member compensation solutions, which shall contain water and an amount of acid which is equal to the acid concentration in the test solution.

4.7 Modifier

4.7.1 General

The examples of modifiers listed below may vary considerably both in combination and in concentration. The recommendation of the manufacturer of the apparatus shall be taken as a starting point for subsequent optimisation. Instead of the solutions according to 4.7.2 to 4.7.5, commercial magnesium nitrate and palladium nitrate solutions may be used.

NOTE The analyst should be aware that the specific use of modifiers for a given element should be applied during establishment of the optimum furnace temperature program.

4.7.2 Magnesium nitrate solution

Dissolve 0,25 g of magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) in 100 ml of water.

4.7.3 Palladium/magnesium nitrate solution

Dissolve 0,075 g of palladium in 2 ml of hot nitric acid (4.3), dilute to 25 ml with water, add 0,05 g of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and make up to 50 ml with water.

4.7.4 Ammonium phosphate/magnesium nitrate solution

Dissolve 0,5 g of ammonium dihydrogenphosphate ($\text{H}_2\text{NH}_4\text{PO}_4$) in water, add 0,05 g of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 1 ml nitric acid 4.3 and make up to 50 ml with water.

4.7.5 Palladium/ascorbic acid solution

Solution A: add 500 μl of hydrochloric acid (4.2) to 1 ml of a palladium stock solution containing 10 g of palladium per litre and make up to 10 ml.

Solution B: dissolve 1 g ascorbic acid in 100 ml of water.

Prepare the palladium/ascorbic acid solution by mixing one part by volume of solution A with one part by volume of solution B.

5 Apparatus and equipment

5.1 General

To minimise the contamination, all apparatus which comes into direct contact with the sample and the solutions should be carefully pre-treated according to EN 13804.

5.2 Atomic absorption spectrometer

With background correction (Zeeman correction is recommended), graphite furnace supplied with argon and with a measurement recording system.

5.3 Graphite tubes

Recommendation, pyrolytically coated tubes with or without L'vov platform.

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5.4 Automatic sample injector

5.5 Element-specific lamps (hollow-cathode or electrodeless discharge lamps) for lead, cadmium, chromium and molybdenum.

6 Procedure

6.1 Sample preparation

The test solution obtained by the pressure digestion method according to EN 13805 may be used for the determination without further treatment.

6.2 Atomic absorption spectrometry (graphite furnace AAS)

6.2.1 Spectrometer settings

Before every determination, adjust the instrument as specified in the manufacturer's operating manual. Then determine an optimum test schedule (if possible using a sample matrix), taking into account, in particular, of parameters such as decomposition temperature and time, atomization temperature and time. In addition matrix modification and injected volumes could be checked. Examples of some instrument parameters are given in Table 1:

Table 1 — Instrument parameters
(standards.iteh.ai)

Parameter	Lead	Cadmium	Chromium	Molybdenum
Radiation source	Hollow-cathode lamp			
Wavelength	283,3 nm	228,8 nm	357,9 nm	313,3 nm
Slit	0,7 nm	0,7 nm	0,7 nm	0,7 nm

6.2.2 AAS-determination

6.2.2.1 General

The absorbance is determined for an aliquot of the test solution by atomic absorption. If there is no significant difference between the slope of the calibration function in the case of the standard addition method and the calibration method, the latter can be employed. Interferences can be reduced by using matrix modifiers (as in 4.7) in conjunction with the L'vov platform.

NOTE The data acquisition during the atomise step can be performed by peak height or peak area measurement. When using platform technique, peak area measurement is recommended.

6.2.2.2 Calibration method

Set the zero of the instrument using the zero member compensation solutions according to 4.6. Derive a calibration function for each element by measuring the absorbances of the calibration solutions with different element concentrations. Plot the absorbances obtained against the concentrations in a graph.

NOTE Modern AAS instruments are capable of deriving the calibration curves themselves and display the measurement for the sample directly in concentration units.

Find the linear range of the calibration function for each element and check it regularly. Carry out the calibration within the linear range using the calibration solution, taking account of the concentration of the element in the digestion solution. The determination can now be carried out using the digestion solution, either untreated or, if the concentration is outside the linear range, after suitable dilution. Check the zero member compensation solutions and individual calibration solutions in the course of fairly long series of measurements.

6.2.2.3 Standard addition method

Determine the linear range of the calibration function as in the case of the calibration method (6.2.2.1).

It is important that the measurements are made in the linear range when the method of standard addition is used. A standard addition curve should consist of at least three points of which at least two are standard additions. The concentration of the highest standard should be 3 to 5 times the concentration in the sample solution. The concentration of the lower standard should be half of the highest standard.

Plot a graph of the absorbances obtained in this way against the added concentrations and extrapolate the resulting straight line until it intercepts the concentration axis.

In the case of AAS instruments with automatic sample injection systems, in which the addition takes place directly into the graphite furnace, the determination can be carried out without prior dilution and this substantially eliminates the risk of contamination.

6.3 Analytical quality control

For analytical quality control, blank solutions and reference samples having reliably known contents of the elements to be determined shall be analysed in parallel with all the series of samples analysed according to EN 13804. The reference samples shall be subjected to all the steps in the method, starting from digestion.

7 Calculation

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Calculate the content, w , as mass fraction, of the element to be determined, in milligram per kilogram of the sample, using the following equation:

$$w = \frac{a \cdot V \cdot F}{m} \quad (1)$$

where

a is the concentration of the element in the test solution, in milligram per litre;

V is the volume of the digestion solution after being made up, in millilitres;

F is the dilution factor of the test solution;

m is the initial sample mass, in gram.

If necessary, subtract the element content of the blank solution from a .

8 Limit of quantification

The atomic absorption spectrometer shall be capable to determine the concentrations given in Table 2 at an injection volume of 20 μ l of the measuring solution: