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Foodstuffs – Determination of trace elements – Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion

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English version

Foodstuffs - Determination of trace elements - Determination of lead, cadmium, zinc, copper and iron by atomic absorption spectrometry (AAS) after microwave digestion

Produits alimentaires - Dosage des éléments traces - Dosage du plomb, du cadmium, du zinc, du cuivre et du fer par spectrométrie d'absorption atomique (AAS) après digestion par micro-ondes

Lebensmittel - Bestimmung von Elementspuren - Bestimmung von Blei, Cadmium, Zink, Kupfer und Eisen mit Atomabsorptionsspektrometrie (AAS) nach Mikrowellenaufschluss

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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents

Foreword

This document (EN 14084:2003) 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of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.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, zinc, copper and iron in foodstuffs by atomic absorption spectrometry (AAS) after microwave digestion.

The method is applicable to determination in various types of foodstuffs. The method is not applicable to oils, fats and other extremely fatty products.

The method has been successfully tested in an interlaboratory trial in which 16 laboratories participated [1]. Foodstuffs covered by the validation of the method include composite diets, cereals, fish, beef, milk and fungi.

Specific foodstuffs for which European Standards exist are excluded from the scope of this horizontal European Standard. It is the task of the analyst to review if vertical standards exist.

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 publications referred to applies (including amendments).

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

3 Principle

The samples are digested in closed vessels in a microwave oven in a mixture of nitric acid and hydrogen peroxide. The resulting solution is diluted with water, and the metal contents are determined by flame or graphite furnace atomic absorption spectrometry-procedures.

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 Nitric acid, not less than 65 %, (mass fraction), having a density of approximately ρ (HNO_3) = 1,4 g/ml.

4.2.1 Nitric acid, $c \approx$ 0,1 mol/l:

Dilute 7 ml concentrated nitric acid (4.2) with water to 1 000 ml.

4.2.2 Nitric acid, $c \approx$ 3 mol/l:

Dilute 200 ml of concentrated nitric acid (4.2) with water to 1 000 ml.

4.3 Hydrogen peroxide, 30 % (mass fraction).

4.4 Standard solutions

NOTE The standard solutions for Pb, Cd, Zn, Cu and Fe can be prepared from metals or metal salts. Standard solutions can also be commercially available. It is advisable to use certified standard solutions. The following preparation of standard solutions are given as an example.

4.4.1 Lead standard solution, 1 000 mg/l:

Dissolve 1,000 g Pb in 7 ml nitric acid (4.2) in a 1 000 ml volumetric flask. Dilute to volume with water.

4.4.2 Cadmium standard solution, 1 000 mg/l:

Dissolve 1,000 g Cd in 20 ml of a mixture of 10 ml water and 10 ml nitric acid (4.2) in a 1 000 ml volumetric flask. Dilute to volume with water.

4.4.3 Zinc standard solution, 1 000 mg/l:

Dissolve 1,000 g Zn in 14 ml of water and 7 ml nitric acid (4.2) in a 1 000 ml volumetric flask. Dilute to volume with water.

4.4.4 Copper standard solution, 1 000 mg/l:

Dissolve 1,000 g Cu in 7 ml nitric acid (4.2) in a 1 000 ml volumetric flask. Dilute to volume with water.

4.4.5 Iron standard solution, 1 000 mg/l:

Dissolve 1,000 g Fe in 14 ml of water and 7 ml nitric acid (4.2) in a 1 000 ml volumetric flask. Dilute to volume with water.

4.5 Calibration solutions

Dilute standard solutions 4.4.1 to 4.4.5 with $c \approx$ 0,1 mol/l nitric acid (4.2.1) to a range of standards that covers the linear range of the element to be determined.

5 Apparatus and equipment

5.1 General

All glassware and plastic ware should be carefully cleaned and rinsed according to the procedure in EN 13804.

5.2 Atomic absorption spectrometer, with background correction, supplied with a graphite furnace/autosampler, burners for flame analysis and an appropriate gas supply.

5.3 Element specific lamps, e.g. hollow cathode lamps, for all elements analysed.

5.4 Graphite tubes, pyrolytically coated and with platforms for Pb and Cd.

5.5 Microwave oven, designed for laboratory use (check for delivered power according to the procedure in EN 13804) and digestion vessels with a capacity of typically 100 ml and withstanding a pressure of at least 1,4 MPa.

5.6 Plastic bottles, with leak-proof closures, 100 ml.

6 Procedure

6.1 Pre-treatment

Homogenise the sample in accordance with the recommendations in EN 13804. If necessary, dry the sample in a way that does not affect the element content, e.g. by freeze-drying.

6.2 Pressure digestion

Weigh an amount of sample equivalent to 0,2 g to 0,5 g dry matter into the digestion vessel (5.5) or as recommended by the manufacturer of the microwave digestion oven used. The maximum test portion from a sample having a water content of e.g. 50 % is thus 1 g (= 0,5 g dry matter); for a material containing 95 % water the test portion may be 2 g (< 0,5 g dry matter). Include one reagent blank in every batch.

Add to the digestion vessel typically 5 ml nitric acid (4.2) and 2 ml hydrogen peroxide (4.3) or as recommended by the manufacturer of the microwave oven used. Seal the vessel and place it in its holder in the microwave oven and close the door. Set the oven programme (power against time) as recommended by the manufacturer for the weighed type of sample.

Typically an oven programme includes a stage at low power for a few minutes followed by one or more stages at higher power settings. A gradual increase between the selected stages is recommended in order to prevent sudden pressure peaks to occur inside the pressure vessels. An example of a programme has been shown in Table 1.

Table 1 — Pressure digestion programme
(The parameters are given as an example applicable to a CEM MDS 2000 oven[1])

Step	Power, W	Time, min
1	250	3:00
2	630	5:00
3	500	22:00
4	0	15:00

The programme used should be valid on the condition that the full number of pressure vessels is treated simultaneously. If fewer vessels are digested, the remaining vessels may be treated as blanks. If the oven used includes pressure control in only one pressure vessel, that vessel with the expected highest pressure should be monitored. This is generally the vessel with the highest sample intake calculated as the dry matter.

When digesting unknown samples, observe caution since a too large amount of sample may rupture the safety membrane of the digestion vessel. In particular, samples high in carbon (especially sugar, fat and/or ethanol) may cause sudden pressure peaks during the ashing process. In all cases, the sample intake should be in strict compliance with the manufacturers recommendations.

6.3 Dilution

Remove the digestion vessels from the microwave oven and allow to cool thoroughly before attempting to open them. Open the vessel and rinse down the lid and the walls with water into the container. Make up to a definite volume, at least 25 ml, with water into a plastic bottle (5.6). Treat the blank in the same way.

6.4 AAS

6.4.1 General

The method to be used – flame or graphite furnace technique – is determined by the concentration of the metal to be analysed. Pb and Cd in foodstuffs generally require graphite furnace-AAS. Zn, Cu and Fe can usually be analysed by flame AAS. Examples of wavelength, gas mixture/temperature programmes and other instrumental parameters appropriate for each metal are found in manuals provided with the instrument. Background correction should always be used, unless proven to be unnecessary.

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.

6.4.2 Flame AAS technique

Dilute the sample solution 1 + 1 with $c \approx 0{,}1$ mol/l nitric acid (4.2.1). Dilute standard solutions 1 + 1 with $c \approx 3$ mol/l nitric acid (4.2.2). All further dilutions should be made with $c \approx 0{,}1$ mol/l nitric acid (4.2.1). The high acid concentration of the sample solution after digestion has detrimental effects on both results and the instrument. It is, therefore, important that the solution is diluted as much as possible and that standard and sample solution have the same acid concentration. When this is done Zn and Cu can, as a rule, be determined against a traditional standard curve.

[1] CEM MDS 2000 is a trade name of a product supplied by CEN, P.O. Box 200, Matthews, NC 28106-200 USA. This information is given for the convenience of the users of this European Standard and does not constitute an endorsement by CEN of the product named.