
Prehrana - Določanje slednih elementov - Določanje svineca, kadmija, cinka, bakra, železa in kroma s spektrometrijo atomne absorpcije (AAS) po sušenju

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

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

Produits alimentaires - Dosage des éléments traces - Détermination du plomb, cadmium, zinc, cuivre, fer et chrome par spectrométrie d'absorption atomique (AAS) après calcination à sec

Ta slovenski standard je istoveten z: EN 14082:2003

ICS:

67.050	Spolšne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products
--------	--	---

SIST EN 14082:2003**en**

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN 14082:2003](#)

<https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003>

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 14082

March 2003

ICS 67.050

English version

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

Produits alimentaires - Dosage des éléments traces - Détermination du plomb, cadmium, zinc, cuivre, fer et chrome par spectrométrie d'absorption atomique (AAS) après calcination à sec

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

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

	page
Foreword.....	3
1 Scope	3
2 Normative references	3
3 Principle	3
4 Reagents	4
5 Apparatus and equipment.....	5
6 Procedure	5
7 Calculation.....	8
8 Precision	9
9 Test report	10
Annex A (informative) Results of the interlaboratory test	11
Bibliography	17

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN 14082:2003](https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003)

<https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003>

Foreword

This document (EN 14082:2003) 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 September 2003, and conflicting national standards shall be withdrawn at the latest by September 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, iron and chromium in foodstuffs by atomic absorption spectrometry (AAS) after dry ashing at 450 °C.

The method is applicable to determination in various types of foodstuffs. 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, fruit, liver and milk.

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 publication referred to applies (including amendments).

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

3 Principle

The samples are dry ashed at 450 °C under a gradual increase in temperature. The ash is dissolved in hydrochloric acid, and the solution obtained evaporated to dryness. The final residue is redissolved in *c* (0,1 mol/l) nitric acid, 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.

EN 14082:2003 (E)

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 37 % mass fraction, having a density of approximately ρ (HCl) = 1 190 mg/ml.

4.2.1 Hydrochloric acid, c (6 mol/l). Dilute 500 ml concentrated hydrochloric acid (4.2) with water to 1 000 ml.

4.3 Nitric acid, not less than 65 % mass fraction, having a density of approximately ρ (HNO₃) = 1 400 mg/ml.

4.3.1 Nitric acid, c (0,1 mol/l). Dilute 7 ml concentrated nitric acid (4.3) with water to 1 000 ml.

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 1000 mg Pb in 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.4.2 Cadmium standard solution. 1 000 mg/l. Dissolve 1 000 mg Cd in 14 ml water + 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.4.3 Chromium standard solution. 1 000 mg/l. Dissolve 3 735 mg K₂CrO₄ in 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.4.4 Zinc standard solution. 1 000 mg/l. Dissolve 1 000 mg Zn in 14 ml water + 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.4.5 Copper standard solution. 1 000 mg/l. Dissolve 1 000 mg Cu in 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.4.6 Iron standard solution. 1 000 mg/l. Dissolve 1 000 mg Fe in 14 ml water + 7 ml nitric acid (4.3) in 1-l-volumetric flask. Dilute to volume with water.

4.5 Calibration solutions

4.5.1 For graphite furnace analysis

Dilute standard solutions 4.4.1 to 4.4.3 with c (0,1 mol/l) nitric acid (4.3.1) to a range of standards that covers the linear range of the element to be determined.

4.5.2 Working standard solutions for flame analysis

Dilute standard solutions 4.4.4 to 4.4.6 with c (0,1 mol/l) nitric acid (4.3.1) to a range of standards that covers the concentration 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 Furnace, programmable with thermostat capable of maintaining (450 ± 25) °C. If a non-programmable furnace is used, a separate pre-ashing device is required (see 5.5 to 5.9).

5.5 Hot plate, with stepwise heating control, up to about 300 °C.

5.6 Lamp, IR 250 W, fixed to a retort stand in a way that allows adjustment of the distance to the plate.

5.7 Ceramic plate, e.g. dessicator plate on a low stand, with a diameter that suits the hot plate.

5.8 Glass cover, e.g. crystallising dish, diameter 185 mm, height 100 mm.

5.9 Wash-bottle containing sulphuric acid for purification of air.

5.10 Platinum or quartz crucibles, 50 ml to 75 ml.

Special attention should be paid to the crucibles. Quartz crucibles shall be stored in a mixture of concentrated nitric acid and water (1+ 9 parts by volume) then rinsed with de-ionised water before use. When necessary, these crucibles have to be boiled with acid as well before use. Platinum crucibles should preferably be heated until red hot and thereafter boiled with acid prior to use.

5.11 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.

6.2 Dry ashing

Weigh a convenient amount of sample (10 g to 20 g in general) into a crucible, to the nearest 10 mg, depending on the kind of sample. Proceed according to type of furnace.

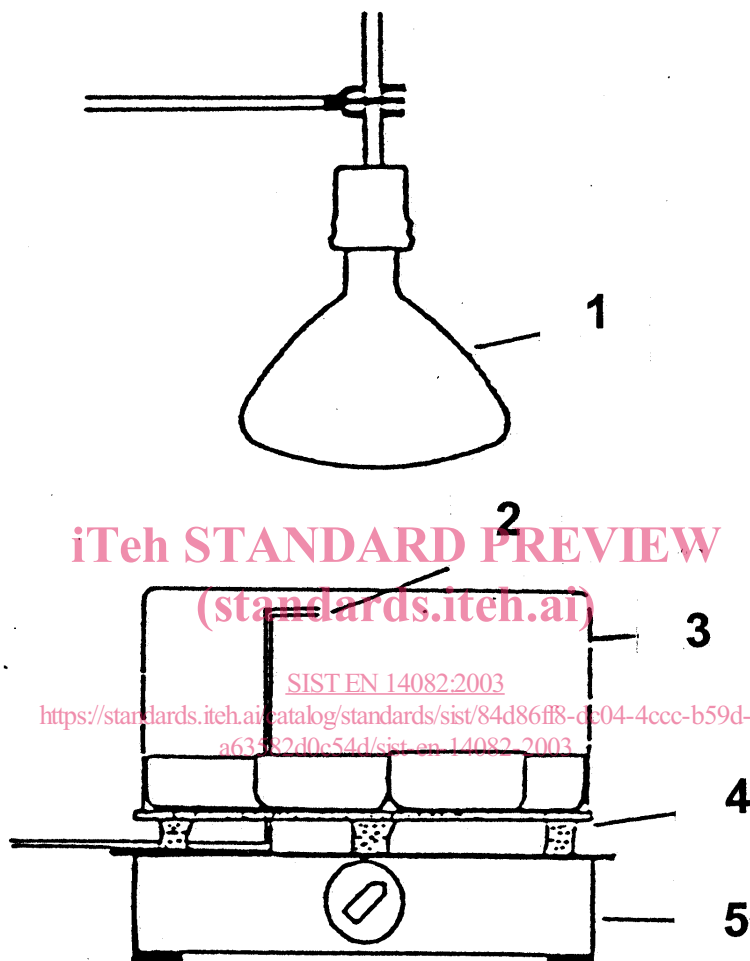
NOTE Calcinations aid can be used, and validated, by the individual laboratory.

6.2.1 Drying and ashing in a programmable furnace: Place the crucible with the test portion in the furnace at an initial temperature not higher than 100 °C. Increase the temperature at a maximum rate of 50 °C/h to 450 °C. Let the sample stand overnight. If there is a risk of heavy boiling make sure that the drying temperature/time is sufficiently low/long. Continue according to 6.2.3.

EN 14082:2003 (E)

6.2.2 Drying and ashing in a non programmable furnace with thermostat and an assembly for drying/pre-ashing (hot plate with ceramic plate and glass cover + IR lamp + wash-bottle with sulphuric acid for purification of air): See Figure 1.

NOTE A programmable furnace is preferable.



Key

- 1 Infrared lamp
- 2 Air
- 3 Crystallising dish
- 4 Ceramic plate
- 5 Hot plate

Figure 1 — Apparatus for pre-ashing of samples

Place the crucible with the test portion covered with the glass cover on the ceramic plate, and let purified air coming through a glass tube sweep over the sample. Put the IR lamp at a distance of 30 cm to 40 cm from the sample and set the hot plate at about 100 °C. Reduce the distance as the drying proceeds until the sample can be assumed to be dry. The lamp should then be right down at the cover.

Pre-ash the sample by increasing the temperature slowly and stepwise with an IR lamp and a hot plate. The final temperature on the ceramic plate should then be about 300 °C. The time required for the pre-ashing varies strongly depending on the type of sample.

Put the crucible in the furnace at 200 °C to 250 °C and slowly raise the temperature to 450 °C at a rate of not more than 50 °C/h. Let the sample stand overnight.

6.2.3 Dissolution of the sample ash

Take the crucible out of the furnace and let it cool. Wet the ash with 1 ml to 3 ml water, and evaporate it on a water-bath or hot plate. Put the crucible back in the furnace at no more than 200 °C and raise the temperature stepwise to 450 °C. Proceed with the ashing at 450 °C for 1 h to 2 h or longer. Repeat this procedure until the sample is completely combusted, i.e. the ash should be white/grey or slightly coloured (the number of repetitions necessary varies strongly depending on the type of sample). Add 5 ml hydrochloric acid (4.2.1) to the crucible, ensuring that all the ash comes into contact with the acid. Evaporate the acid on a water-bath or hot plate. Dissolve the residue in an exact volume (10,0 ml to 30,0 ml) of nitric acid (4.3.1). Swirl the crucible with care so that all the ash comes into contact with the acid. Cover with a watch glass and let the sample stand for 1 h to 2 h. Then stir the solution in the crucible thoroughly with a stirring rod and transfer the contents to a plastic bottle.

CAUTION — Samples with a very high fat and/or sugar content should be ashed with great care. Fatty samples can otherwise easily self-ignite. Sugar rich samples tend to expand during the ashing and can be lost. The pre-ashing procedure according to 6.2.2 is then recommended.

Treat reagent blanks in the same way as samples.

6.3 Atomic absorption spectrometry (AAS)

6.3.1 General

The method to be used - flame or graphite furnace technique - is determined by the concentration of the element to be analysed. Flame AAS should be used whenever possible, since this technique is less sensitive to interference than graphite furnace AAS. Determination of Pb, Cd and Cr in foodstuffs generally require graphite furnace AAS. Zn, Cu and Fe can in most foodstuffs be determined by flame AAS. Examples of wavelengths, gas mixtures/temperature programmes and other instrumental parameters appropriate for each element are found in the manual provided with the instrument. Background correction shall always be used, also when Cr is determined, unless proven to be unnecessary. When necessary, the sample solutions should be diluted with *c* (0,1 mol/l) nitric acid (4.3.1).

[SIST EN 14082:2003](https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003)

[https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-](https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003)

6.3.2 Flame AAS technique, (FAAS) [a63582d0c54d/sist-en-14082-2003](https://standards.iteh.ai/catalog/standards/sist/84d86ff8-dc04-4ccc-b59d-a63582d0c54d/sist-en-14082-2003)

The metal contents of the samples are derived from the calibration curves made up of a minimum of 3 standards. Table 1 exemplifies instrumental parameters for flame AAS.

Table 1 — Instrumental parameters for flame AAS

Metal	Flame	Wavelength, nm
Zn	Air-acetylene, oxidising	213,9
Cu	- " -	324,7
Fe	Nitrous oxide-acetylene, oxidising	248,3

6.3.3 Graphite furnace AAS technique

The method of addition should always be used unless proven to be unnecessary. It is of utmost importance that the measurements are made in the linear range when the method of addition is used. Measurements are preferably made on peak area rather than peak height. Table 2 shows examples of instrumental parameters applicable to a Perkin Elmer/HGA 500 instrument¹. The temperature programme for ashing and atomisation should be optimised for each matrix.

¹ Perkin Elmer/HGA 500 is a trade name of a product supplied by Perkin Elmer Corporation, 761 Main Avenue, Norwalk, CT 06859-0226 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.