British Standard

BRITISH STANDARD

BS EN 14084:2003

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

The European Standard EN 14084:2003 has the status of a British Standard

ICS 67.050

British Standards

National foreword

This British Standard is the official English language version of EN 14084:2003.

The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Horizontal analysis, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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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

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.

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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of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.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.

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[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.

The determination of Fe can be strongly affected by interferences from the matrix. Therefore use either the method of standard addition or matrix-matched standard curve. When experiencing severe interferences, the use of an oxidising nitrous oxide flame may be an alternative. Table 2 contains examples of instrumental parameters for flame technique AAS.

Table 2 — Instrumental parameters for flame AAS

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

6.4.3 Graphite furnace technique

This technique is generally required for determination of Pb and Cd. Use pyrolytically coated tubes with platforms. Since microwave digestion results in fairly large dilutions of the sample the graphite furnace may often be useful in the determination of e.g. Cu as well.

Program the autosampler to deliver a sample volume to the graphite furnace, which gives a background absorbance of not more than about 0,5 absorbance units (peak hight). To enhance the absorbance at very low concentrations, multiple injections may be employed. Table 3 shows examples of instrumental parameters applicable to a Perkin Elmer/HGA 600[2] instrument.

Evaluate new matrices by means of ash/atomization curves in order to optimize parameters of the graphite furnace technique.

NOTE Matrix modifiers can be used, and validated, by the individual laboratory. When determining lead and cadmium see 4.7 in EN 14083:2003.

Table 3 — Instrumental parameters for determination by graphite furnace AAS and with an injection volume of 20 µl.

Metal	**Wavelength** nm	**Parameter**	**Step 1**	**Step 2**	**Step 3**	**Step 4**
Pb	283,3	Temp., °C	130	450	1900	2500
		Ramp, s	10	15	0	2
		Hold, s	30	10	4	2
Cd	228,8	Temp., °C	130	350	1200	2500
		Ramp, s	10	15	0	2
		Hold, s	30	10	4	2

Always use the method of standard addition unless it is proven to be unnecessary.

A simplified version of the method of standard addition is to use a matrix-matched standard curve, which is applicable to samples with the same matrix and sample weight: The sample and standard solutions are mixed and used to make a standard addition curve. This curve is then parallel transferred to the origin and is used as the standard curve for the samples that follows and that have been diluted in the same proportions. The matrix matched standard and the sample solutions will thus have the same matrix concentration. This function is often available in the software of modern AAS instruments.

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[2] Perkin Elmer/HGA 600 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 constitue an endorsement by CEN of the product named.

7 Calculation

7.1 General

Measure the peak area when using GFAAS, for flame AAS use the steady state signal. Construct a standard curve and read the concentration of the metal from the curve. Calculate the content, *w*, as mass fraction of the element to be determined, in milligram per kilogram of sample, using the following equation:

$$w = \frac{(a - b) \cdot V}{m} \tag{1}$$

where

a is the concentration in the sample solution in milligram per litre;

b is the mean concentration in the reagent blank solutions in milligram per litre;

V is the volume of the sample solution in millilitres;

m is the sample mass in gram.

If (a-b) is lower than the sample solution detection limit (see 7.2), substitute (a-b) with the value for the detection limit in the sample solution for calculation of the limit of detection in the sample.

If the sample has been diluted, take into account the dilution factor.

If required recalculate the result to fresh weight if it is based on the dry matter content of the sample. Give the result with an applicable number of significant figures.

7.2 Estimation of the detection and quantification limits

The detection and quantification limits should be estimated for each element according to EN 13804, taking into account the SD found in the long term evaluation.

8 Precision

8.1 General

The method has been tested on dry materials only, but may under certain conditions be used for samples containing water (see 6.2). Details of the results of an interlaboratory test are summarised in annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

8.2 Repeatability

The absolute difference between two independent single test results obtained with the same test method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time will in not more than 5 % of the cases exceed the repeatability limit *r* given in Table 4.

8.3 Reproducibility

The absolute difference between two single test results obtained with the same test method on identical test material in different laboratories by different operators using different equipment will in not more than 5 % of the cases exceed the reproducibility limit *R* given in Table 4.

Table 4 – Mean values, repeatability and reproducibility limits

Metal	Sample	$\bar{x}$ (mg/kg)	***r*** (mg/kg)	***R*** (mg/kg)
Pb	CRM Composite diets E/F[a]	0,39	0,18	0,27
	CRM NIST Bovine muscle no.8414	0,40		0,24
	Minced fish	0,48		0,36
	CRM Mushroom[b]	1,62		0,73
Cd	Liver	0,164	0,070	0,094
	Wheat bran	0,17	0,022	0,063
	CRM Composite diets E/F[a]	0,76	0,14	0,29
	CRM NIST Bovine muscle no.8414	0,012		0,010
	Minced fish	0,21		0,099
	CRM Mushroom[b]	0,48		0,15
Zn	Milk powder	35,3		9,1
	Liver	182	7,9	25
	Wheat bran	73,5	7,1	9,9
	CRM Composite diets E/F[a]	47,8	5,4	7,1
	CRM NIST Bovine muscle no.8414	147		7,0
	Minced fish	4,5		1,2
	CRM Mushroom[b]	56,9		8,4
Cu	Milk powder	0,58		0,44
	Liver	108	9,3	12
	Wheat bran	10,3	2,1	2,9
	CRM Composite diets E/F[a]	63,4	2,7	5,3
	Bovine muscle	2,9		1,1
	Minced fish	0,25		0,20
	CRM Mushroom[b]	37,7		6,0
Fe	Liver	484	75	90
	Wheat bran	124	15	30
	CRM Composite diets E/F[a]	303	34	48
	Bovine muscle	73		14
	Minced fish	7,6		3,9
	CRM Mushroom[b]	104		24

[a] See [2].

[b] See [3].

9 Test report

The test report shall specify at least the following:

a) all information necessary for the identification of the sample;

b) the test method used, with reference to this European Standard;

c) the results obtained and the units in which they are specified;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this European Standard or regarded as optional, together with details of any incidents occurred when performing the method which might have influenced the test result(s).

Annex A
(informative)

Results of the interlaboratory test

The precision of the method was established by NMKL [1], [6] and has been verified in an interlaboratory test evaluated in accordance with AOAC-Guidelines for Collaborative Study Procedures [4]. The results are given in Table A.1.

Table A.1 — Statistical results of the interlaboratory tests

		Sample						
Element	**Parameter**	**Milk powder**[a]	**Liver**[a]	**Wheat bran**[a]	**Composite diet E/F**	**Bovine muscle**	**Fish**	**Mushroom**
Pb	Number of laboratories	12	12	12	12	12	12	12
	Number of outliers	2	1	0	0	2	0	0
	Number of laboratories after elimination of outliers	10	11	12	12	10	12	12
	Mean value $\bar{x}$ (mg/kg)	0,005	0,13	0,16	0,39	0,40	0,48	1,62
	Repeatability – standard deviation s_r (mg/kg)	-	0,05	0,09	0,06	-	-	-
	RSD_r (%)	-	37	57	16	-	-	-
	Repeatability limit r (mg/kg)	-	0,14	0,25	0,18	-	-	-
	Reproducibility – standard deviation s_R (mg/kg)	0,019	0,06	0,09	0,10	0,09	0,13	0,26
	RSD_R (%)	381	42	59	25	22	27	16
	Reproducibility limit R (mg/kg)	0,052	0,15	0,26	0,27	0,24	0,36	0,73
	Horwitz value R	36	22	21	18	18	18	15
	Horrat R index	11	1,9	2,8	1,4	1,2	1,5	1,1

[a] The results indicate that the mean concentration is below the limit of quantification of the method.

Table A.1 *(continued)*

		Sample						
Element	**Parameter**	**Milk powder**[a]	**Liver**	**Wheat bran**	**Composite diet E/F**	**Bovine muscle**	**Fish**	**Mushroom**
Cd	Number of laboratories	13	13	13	13	13	12	13
	Number of outliers	2	0	2	1	1	0	2
	Number of laboratories after elimination of outliers	11	13	11	12	12	12	11
	Mean value $\bar{x}$ (mg/kg)	0,000	0,164	0,171	0,764	0,012	0,211	0,482
	Repeatability – standard deviation s_r (mg/kg)	-	0,025	0,008	0,050	-	-	-
	RSD_r (%)	-	15	4,6	6,5	-	-	-
	Repeatability limit r (mg/kg)	-	0,070	0,022	0,14	-	-	
	Reproducibility – standard deviation s_R (mg/kg)	0,003	0,034	0,022	0,105	0,003	0,035	0,053
	RSD_R (%)	-8 392	20	13	14	28	17	11
	Reproducibility limit R (mg/kg)	0,009	0,094	0,063	0,294	0,010	0,099	0,149
	Horwitz value R	-	21	21	17	31	20	18
	Horrat R index	-	1,0	0,6	0,8	0,9	0,8	0,6

[a] The results indicate that the mean concentration is below the limit of quantification of the method.

Table A.1 *(continued)*

		Sample						
Element	**Parameter**	**Milk powder**	**Liver**	**Wheat bran**	**Composite diet E/F**	**Bovine muscle**	**Fish**	**Mushroom**
Zn	Number of laboratories	14	14	14	14	14	13	14
	Number of outliers	0	2	1	1	3	0	0
	Number of laboratories after elimination of outliers	14	12	13	13	11	13	14
	Mean value $\bar{x}$ (mg/kg)	35,3	182	73,5	47,8	147,3	4,5	56,9
	Repeatability – standard deviation s_r (mg/kg)	-	2,8	2,5	1,9	-	-	-
	RSD_r (%)	-	1,6	3,4	4,0	-	-	-
	Repeatability limit r (mg/kg)	-	7,9	7,1	5,4	-	-	-
	Reproducibility – standard deviation s_R (mg/kg)	3,3	9	3,5	2,5	2,5	0,43	3,0
	RSD_R (%)	9,3	4,8	4,8	5,3	1,7	9,7	5,3
	Reproducibility limit R (mg/kg)	9,1	25	9,9	7,1	7,0	1,2	8,4
	Horwitz value R	9	7	8	9	8	13	9
	Horrat R index	1,0	0,7	0,6	0,6	0,2	0,8	0,6

Table A.1 *(continued)*

		Sample						
Element	**Parameter**	**Milk powder**	**Liver**	**Wheat bran**	**Composite diet E/F**	**Bovine muscle**	**Fish**	**Mushroom**
Cu	Number of laboratories	14	14	14	14	14	13	14
	Number of outliers	3	0	0	2	0	0	0
	Number of laboratories after elimination of outliers	11	14	14	12	14	13	14
	Mean value $\bar{x}$ (mg/kg)	0,58	108	10,3	63,4	2,89	0,254	37,7
	Repeatability – standard deviation s_r (mg/kg)	-	3,3	0,75	1,0	-	-	-
	RSD_r (%)	-	3,1	7,2	1,5	-	-	-
	Repeatability limit r (mg/kg)	-	9,3	2,1	2,7	-	-	-
	Reproducibility – standard deviation s_R (mg/kg)	0,16	4,1	1,06	1,9	0,42	0,071	2,2
	RSD_R (%)	27	3,8	10	3,0	14	28	5,7
	Reproducibility limit R (mg/kg)	0,44	12	2,92	5,3	1,15	0,199	6,0
	Horwitz value R	17	8	11	9	14	20	9
	Horrat R index	1,6	0,5	0,9	0,4	1,0	1,4	0,6

Table A.1 *(continued)*

		Sample						
Element	**Parameter**	**Milk powder**[a]	**Liver**	**Wheat bran**	**Composite diet E/F**	**Bovine muscle**	**Fish**	**Mushroom**
Fe	Number of laboratories	13	13	13	13	13	12	13
	Number of outliers	1	0	0	2	1	0	0
	Number of laboratories after elimination of outliers	12	13	13	11	12	12	13
	Mean value $\bar{x}$ (mg/kg)	3,3	484	124,2	303	73,0	7,6	103,8
	Repeatability – standard deviation s_r (mg/kg)	-	27	5,3	12	-	-	-
	RSD_r (%)	-	5,6	4,2	4,0	-	-	-
	Repeatability limit *r* (mg/kg)	-	75	14,8	34	-	-	-
	Reproducibility – standard deviation s_R (mg/kg)	1,6	32	10,6	17	5,0	1,4	8,5
	RSD_R (%)	47	6,7	8,6	5,7	6,9	18	8,2
	Reproducibility limit *R* (mg/kg)	4,4	90	29,8	48	14,1	3,9	23,7
	Horwitz value *R*	13	6	8	7	8	12	8
	Horrat *R* index	3,5	1,1	1,1	0,8	0,8	1,5	1,0

[a] The results indicate that the mean concentration is below the limit of quantification of the method.

Results of the analysis of certified reference materials in an interlaboratory test are given in Table A.2. The number of laboratories remaining after elimination of outliers is shown under ”*n*”. All results in milligram per kilogram dry matter. The certified values for the composite diets and the mushroom refer to [2] and [3].

Table A.2 — Certified values

Metal	**CRM**	**Found**			**Certified**			**Z-score**[a]
		n	$\bar{x}$	s_R	n	$\bar{x}$	s_R	
Pb	Composite diet E	12	0,287	0,055	10	0,273	0,024	0,8
	Composite diet F	12	0,501	0,127	9	0,439	0,034	1,6
	Mushroom	12	1,62	0,26	6	1,43	0,10	2,2
	Bovine muscle	10	0,40	0,09		0,38	0,24[b]	
Cd	Composite diet E	12	0,580	0,088	13	0,536	0,051	1,5
	Composite diet F	12	0,948	0,119	11	0,877	0,074	1,7
	Mushroom	11	0,482	0,053	7	0,437	0,033	2,2
	Bovine muscle	12	0,012	0,003		0,013	0,011	
Zn	Composite diet E	13	39,6	3,0	8	39,5	4,0	0,1
	Composite diet F	13	56,0	1,9	8	55,8	5,0	0,1
	Mushroom	14	56,9	3,0	8	55,0	2,4	1,6
	Bovine muscle	11	147	2		142	14	
Cu	Composite diet E	12	49,8	1,9	9	46,5	1,8	4,1
	Composite diet F	12	77,0	1,9	8	72,7	1,2	6,2
	Mushroom	14	37,7	2,2	9	34,4	4,5	2,0
	Bovine muscle	14	2,89	0,41		2,84	0,45	
Fe	Composite diet E	11	232	16	9	216	20	1,9
	Composite diet F	11	374	19	9	349	24	2,5
	Mushroom	13	104	8	6	101	10	0,6
	Bovine muscle	12	73,0	5,0		71,2	9,2	

[a] Z-score according to NMKL Procedure No. 9 [5].

[b] 95 % confidence interval.

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