
**Animal feeding stuffs — Determination
of the contents of calcium, copper, iron,
magnesium, manganese, potassium,
sodium and zinc — Method using atomic
absorption spectrometry**

*Aliments des animaux — Détermination des teneurs en calcium, cuivre, fer,
magnésium, manganèse, potassium, sodium et zinc — Méthode par
spectrométrie d'absorption atomique*



Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6869 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*.

Annex A of this International Standard is for information only.

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Animal feeding stuffs — Determination of the contents of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc — Method using atomic absorption spectrometry

1 Scope

This International Standard specifies an atomic absorption spectrometric method for the determination of the contents of calcium (Ca), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), potassium (K), sodium (Na) and zinc (Zn) in animal feeding stuffs.

The method is applicable to all animal feeding stuffs.

The limit of determination for the elements concerned is as follows:

K and Na	500 mg/kg;
Ca and Mg	50 mg/kg;
Cu, Fe, Mn and Zn	5 mg/kg.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative documents referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 6498, *Animal feeding stuffs — Preparation of test samples*.

3 Principle

A test portion is dissolved in hydrochloric acid, if necessary after ashing in a muffle furnace at (550 ± 15) °C. Any silica compounds present are removed by precipitation and filtration. The precipitate is dissolved in hydrochloric acid and diluted to the desired volume, then aspirated into the air-acetylene flame of the atomic absorption spectrometer.

The absorbance of each element is measured by comparison with the absorbance of calibration solutions for the same element.

4 Reagents and materials

Use only reagents of recognized analytical grade.

4.1 Water, complying with at least grade 3 in accordance with ISO 3696.

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4.2 Concentrated hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$ ($\rho = 1,19 \text{ g/ml}$).

4.3 Hydrochloric acid, $c(\text{HCl}) = 6 \text{ mol/l}$.

4.4 Dilute hydrochloric acid, $c(\text{HCl}) = 0,6 \text{ mol/l}$.

4.5 Lanthanum nitrate solution.

Dissolve 133 g of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ in 1 litre of water (4.1).

Another lanthanum salt may be used if the lanthanum content of the prepared solution is the same.

4.6 Caesium chloride solution.

Dissolve 100 g of CsCl in 1 litre of water (4.1).

Another caesium salt may be used if the caesium content of the obtained solution is the same.

4.7 Stock solution of Cu, Fe, Mn and Zn.

Mix 100 ml of water (4.1) and 125 ml of concentrated hydrochloric acid (4.2) in a 1 litre volumetric flask.

Weigh out the following:

- 392,9 mg of copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$);
- 702,2 mg of ammonium iron(II) sulfate hexahydrate [$(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$];
- 307,7 mg of manganese sulfate monohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$);
- 439,8 mg of zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$).

Transfer the weighed salts to the volumetric flask and dissolve them in water (4.1). Dilute to the mark with water.

The contents of Cu, Fe, Mn and Zn of this stock solution are each 100 $\mu\text{g/ml}$.

NOTE Ready-prepared commercially available solutions may be used.

4.8 Standard solution of Cu, Fe, Mn and Zn.

Dilute 20,0 ml of the stock solution (4.7) with water (4.1) to 100 ml in a volumetric flask.

The contents of Cu, Fe, Mn and Zn of this solution are each 20 $\mu\text{g/ml}$.

Prepare the solution fresh on the day of use.

4.9 Stock solution of Ca, K, Mg and Na.

Weigh out the following:

- 1,907 g of potassium chloride (KCl);
- 2,028 g of magnesium sulfate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$);
- 2,542 g of sodium chloride (NaCl).

Transfer the weighed salts to a 1 litre volumetric flask.

Add 50 ml of hydrochloric acid (4.3) to a beaker. Weigh into the beaker 2,497 g of calcium carbonate (CaCO₃).

CAUTION: Beware of the development of carbon dioxide.

Boil for 5 min on an electric hot plate (5.4). Cool and transfer the solution to the volumetric flask containing the weighed salts of K, Mg and Na. Dissolve the salts and dilute to the mark with dilute hydrochloric acid (4.4).

The contents of Ca, K and Na of this solution are each 1 mg/ml; the content of Mg of the solution is 200 µg/ml.

NOTE Ready-prepared commercially available solutions may be used.

4.10 Standard solution of Ca, K, Mg and Na.

Dilute 25,0 ml of stock solution (4.9) with dilute hydrochloric acid (4.4) to 250 ml in a volumetric flask.

The contents of Ca, K and Na of this solution are each 100 µg/ml; the content of Mg of the solution is 20 µg/ml.

Prepare the solution fresh in the week of use and store it in a polyethylene bottle.

4.11 Lanthanum/caesium blank solution

Add 5 ml of lanthanum nitrate solution (4.5), 5 ml of caesium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3) to a 100 ml volumetric flask. Dilute to the mark with water (4.1).

5 Apparatus

Rinse all volumetric ware, including the pipettes used for preparing calibration solutions, with dilute hydrochloric acid (4.4) before use.

If dedicated incineration dishes and glassware are used, it is not necessary to boil with hydrochloric acid each time before use.

Usual laboratory apparatus and, in particular, the following.

5.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.2 Incineration dishes of platinum, quartz or porcelain, free from potassium and sodium, with a smooth unetched inner surface, upper internal diameter 4 cm to 6 cm, lower internal diameter 2 cm to 2,5 cm, and a height of about 5 cm. Before use, boil with hydrochloric acid (4.3).

5.3 Glassware of hard borosilicate glass.

Before use, boil with hydrochloric acid (4.3) and rinse with water.

5.4 Electric hot plate or **gas burner**.

5.5 Boiling water bath.

5.6 Electric muffle furnace, capable of being maintained at $(550 \pm 15) ^\circ\text{C}$.

5.7 Atomic absorption spectrometer (see ISO 6956 [6]), suitable for measuring at the wavelengths specified in 8.6.1 and 8.7.1, provided with an air-acetylene flame and a facility for correction for, or measurement of, background absorption.

5.8 Hollow cathode lamps or **electrode-less discharge lamps**, for the determination of Ca, Cu, Fe, K, Mg, Mn, Na or Zn.

5.9 Filter paper which does not release minerals.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497 [5].

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Store the sample so that deterioration and other changes in composition are prevented.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 6498.

8 Procedure

8.1 Detection of presence of organic matter

Heat a spatula with some test sample in a flame.

If the test sample melts without smoke, little organic matter is present.

If the test sample changes in colour and melting does not occur, the test sample contains organic matter.

8.2 Test portion

Depending on the expected content, weigh 1 g to 5 g of the prepared test sample (clause 7), to the nearest 1 mg, into an incineration dish (5.2).

If the test sample contains organic matter (see 8.1), proceed in accordance with 8.3.

If the test sample contains little or no organic matter (see 8.1), proceed in accordance with 8.4.

8.3 Dry ashing

Heat the incineration dish on a hot plate or over a gas burner (5.4) until the test portion has been completely carbonized. Avoid burning the test portion.

Transfer the dish to the muffle furnace (5.6) which has already been at a temperature of 550 °C for 15 min. Ash the sample for 3 h at this temperature.

Allow the sample to cool down then moisten the contents of the dish with 2 ml of water. If many carbon particles are present, dry the dish over the water bath (5.5).

Ash for another 2 h in the muffle furnace set at 550 °C.

Allow to cool down then add 2 ml of water.

8.4 Decomposition

While swirling, add 10 ml of hydrochloric acid (4.3), first dropwise until effervescence (possible development of carbon dioxide) has ceased, then faster. Swirl and heat the contents of the dish until almost dry. While drying, take care to avoid loss by splattering.

Dissolve the residue by heating with 5 ml of hydrochloric acid (4.3) and transfer the solution quantitatively with some 5 ml portions of water to a 50 ml volumetric flask.

Allow to cool, then dilute to the mark with water and mix. Allow the particles to settle and filter (see 5.9) the solution if it is not clear after 4 h.

8.5 Blank solution

Prepare for each measuring series a blank solution by carrying out the procedure according to 8.2, 8.3 and 8.4 without the test sample.

8.6 Determination of copper, iron, manganese and zinc

8.6.1 Measuring conditions

Adjust the atomic absorption spectrometer (5.7) in accordance with the manufacturer's instructions. Optimize the response of the instrument for measurement with the air-acetylene flame. For the determination of Cu, Fe, Mn and Zn set the following wavelengths:

Cu: 324,8 nm;

Fe: 248,3 nm;

Mn: 279,5 nm;

Zn: 213,8 nm.

8.6.2 Preparation of calibration curve

Prepare a series of appropriate calibration solutions by diluting the standard solution (4.8) with dilute hydrochloric acid (4.4).

Measure the absorbance of the hydrochloric acid (4.4). Measure the absorbance of the calibration solutions and subtract the absorbance measured for the hydrochloric acid (4.4).

Draw a calibration curve by plotting the corrected absorbances against the respective contents of Cu, Fe, Mn and Zn.

8.6.3 Measurement of test solution

Measure parallel to the calibration solutions, under identical circumstances, the absorbance of the test solution (8.4) and the blank solution (8.5). Subtract the latter absorbance from the first absorbance.

If necessary, dilute a quantity of the test solution and blank solution with dilute hydrochloric acid (4.4) to obtain an absorbance in the linear part of the calibration curve.

Proceed in accordance with clause 9.

8.7 Determination of calcium, magnesium, potassium and sodium

8.7.1 Measuring conditions

Adjust the atomic absorption spectrometer in accordance with the manufacturer's instructions. Optimize the response of the instrument for measurement with the air-acetylene flame. For the determination of Ca, K, Mg and Na set the following wavelengths:

Ca: 422,6 nm;

K: 766,5 nm;

Mg: 285,2 nm;

Na: 589,6 nm.

8.7.2 Preparation of calibration curve

Dilute the standard solution (4.10) with water (4.1). Add per 100 ml of diluted standard solution 5 ml of lanthanum nitrate solution (4.5), 5 ml of caesium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Choose the dilutions so that appropriate calibration solutions are obtained.

Measure the absorbance of the lanthanum/caesium blank solution (4.11).

Measure the absorbance of the calibration solutions and subtract the absorbance measured for the lanthanum/caesium blank solution (4.11).

Draw a calibration curve by plotting the corrected absorbances against the respective contents of Ca, K, Mg and Na.

8.7.3 Measurement of test solution

Dilute a quantity of the test solution (8.4) and blank solution (8.5) with water (4.1). Add per 100 ml of diluted solution 5 ml of lanthanum nitrate solution (4.5), 5 ml of caesium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3).

Measure parallel to the calibration solutions, under identical circumstances, the absorbance of the diluted test solution and the diluted blank solution. Subtract the latter absorbance from the first absorbance.

If necessary, dilute a quantity of the test solution and blank solution with lanthanum/caesium blank solution (4.11) to obtain an absorbance in the linear part of the calibration curve.

9 Expression of results

Calculate the content of each of the elements calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc starting from the calibration curve and taking into account the mass of the test portion and the dilutions applied.

Round the result in accordance with Table 1 and express the result in milligrams per kilogram or grams per kilogram.

Table 1 — Rounding of the calculated content

Content		Round to
from	to	
5 mg/kg	10 mg/kg	0,1 mg/kg
10 mg/kg	100 mg/kg	1 mg/kg
100 mg/kg	1 g/kg	10 mg/kg
1 g/kg	10 g/kg	100 mg/kg
10 g/kg	100 g/kg	1 g/kg

10 Precision

10.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are given in annex A. The values derived from these tests may not be applicable to concentration ranges and matrices other than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same 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 cases exceed the repeatability limit (r) mentioned in or derived from Table 2 or Table 3.

Table 2 — Repeatability limit (r) and reproducibility limit (R) for premixes

Values in milligrams per kilogram

Element	Element content		r	R
	from	to		
Ca	3 000	300 000	$0,07 \times \bar{w}$	$0,20 \times \bar{w}$
Cu	200	20 000	$0,07 \times \bar{w}$	$0,13 \times \bar{w}$
Fe	500	30 000	$0,06 \times \bar{w}$	$0,21 \times \bar{w}$
K	2 500	30 000	$0,09 \times \bar{w}$	$0,26 \times \bar{w}$
Mg	1 000	100 000	$0,06 \times \bar{w}$	$0,14 \times \bar{w}$
Mn	150	15 000	$0,08 \times \bar{w}$	$0,28 \times \bar{w}$
Na	2 000	250 000	$0,09 \times \bar{w}$	$0,26 \times \bar{w}$
Zn	3 500	15 000	$0,08 \times \bar{w}$	$0,20 \times \bar{w}$

\bar{w} is the mean of the two results, in milligrams per kilogram.

Table 3 — Repeatability limit (r) and reproducibility limit (R) for animal feeds

Values in milligrams per kilogram

Element	Element content		r	R
	from	to		
Ca	5 000	50 000	$0,07 \times \bar{w}$	$0,28 \times \bar{w}$
Cu	10	100	$0,27 \times \bar{w}$	$0,57 \times \bar{w}$
Cu	100	200	$0,09 \times \bar{w}$	$0,16 \times \bar{w}$
Fe	50	1 500	$0,08 \times \bar{w}$	$0,32 \times \bar{w}$
K	5 000	30 000	$0,09 \times \bar{w}$	$0,28 \times \bar{w}$
Mg	1 000	10 000	$0,06 \times \bar{w}$	$0,16 \times \bar{w}$
Mn	15	500	$0,06 \times \bar{w}$	$0,40 \times \bar{w}$
Na	1 000	6 000	$0,15 \times \bar{w}$	$0,23 \times \bar{w}$
Zn	25	500	$0,11 \times \bar{w}$	$0,19 \times \bar{w}$

\bar{w} is the mean of the two results, in milligrams per kilogram.

NOTE In Table 2 and Table 3, the repeatability and reproducibility limits are given in the form of a formula for each element and for the indicated range. The factor in this formula is an average over the investigated samples in the indicated range. In exceptional cases higher values have been obtained for the determination of a specific element in specific samples. These samples have not been taken into account. Most likely cause of these deviations is lack of homogeneity of the samples concerned (see annex A).

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases exceed the reproducibility limit (R) mentioned in or derived from Table 2 or Table 3.

11 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this International Standard;
- all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result obtained, or the two test results obtained if the repeatability has been checked.

Annex A (informative)

Results of interlaboratory tests

Starting in 1983, the precision of the method has been tested by several interlaboratory tests carried out on mixed feeds and premixes in accordance with ISO 5725:1986 [2]. The statistical results of a first series of tests were published in reference [7]. As a consequence of the results of these tests, the method was modified. The precision of the modified method was established by two interlaboratory tests.

In the first test (in the period 1986-1987) 26 laboratories from 7 countries participated. Samples were investigated of a premix, two mixed feeds, barley, wilted silage, milk powder and pigs' faeces. In view of the unsatisfactory results for premixes, it was decided to amend the method for premixes. The precision of the amended method for premixes was established by an additional interlaboratory test.

In this second test (in the period 1987-1988), 13 laboratories participated. Samples were investigated of eight premixes (five of which contained organic matter).

The statistical results of the two interlaboratory tests are summarized in Tables A.1 to A.8.

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Table A.1 — Statistical results for calcium

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVR %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	12	0	24	51 000	1 413	2,9	4 000	5 654	10,9	16 000
P2	12	2	22	292 000	6 007	2,1	17 000	16 254	5,7	46 000
P3	12	2	22	136 000	2 120	1,6	6 000	12 367	9,0	35 000
PO1	12	0	24	156 000	2 120	1,3	6 000	10 247	6,5	29 000
PO2	12	2	22	11 900	389	3,2	1 100	636	5,5	1 800
PO3	12	2	22	3 000	106	3,8	300	247	8,6	700
PO4	12	0	24	7 900	177	2,1	500	530	6,9	1 500
PO5	12	0	24	5 700	141	2,4	400	318	5,9	900
B	21	8	34	592	62	10,4	175	179	30,3	507
WS	21	8	34	4 860	130	2,7	369	604	12,4	1 710
MFH	21	10	32	7 650	195	2,6	552	753	9,8	2 130
MP	21	6	36	13 300	417	3,1	1 180	1 484	11,1	4 200
PF	21	2	40	33 200	565	1,7	1 600	6 855	20,6	19 400
MFL	21	9	33	47 400	1 106	2,3	3 130	3 424	7,2	9 690

- ^a P1 is premix;
P2 is premix;
P3 is premix;
PO1 is premix containing organic matter;
PO2 is premix containing organic matter;
PO3 is premix containing organic matter;
PO4 is premix containing organic matter;
PO5 is premix containing organic matter;
B is barley;
WS is wilted silage;
MFH is mixed feed H;
MP is milkpowder;
PF is pigs' faeces;
MFL is mixed feed L;

- ^b n_1 is the number of laboratories with results;
 n_2 is the number of outliers;
 n_3 is the number of accepted results;
 \bar{w} is the mean calcium content of the sample;
 s_r is the repeatability standard deviation;
CVR is the repeatability coefficient of variation;
 r is the repeatability limit [$r = 2,8 \times s_r$];
 s_R is the reproducibility standard deviation;
CVR is the reproducibility coefficient of variation;
 R is the reproducibility limit [$R = 2,8 \times s_R$].

Table A.2 — Statistical results for copper

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVr %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	13	0	26	900	25	2,7	70	64	7,0	180
P2	13	0	26	1 000	28	2,8	80	67	6,8	190
P3	13	0	26	17 400	212	1,2	600	495	2,9	1 400
PO1	13	0	26	4 540	78	1,7	220	152	3,3	430
PO2	13	0	26	217	6,4	2,9	18	10	4,7	29
PO3	13	2	24	1 050	57	5,4	160	141	13,5	400
PO4	13	0	26	6 660	251	3,8	710	293	4,4	830
PO5	13	0	26	39 100	495	1,2	1 400	1 555	3,1	4 400
B	22	6	38	6	0,4	8,4	1	1,4	21,3	4
WS	22	4	40	14	1,1	7,0	3	2,1	15,9	6
MFH	22	4	40	15	2,1	14,5	6	3,5	23,8	10
MP	22	4	40	2	0,4	19,4	1	1,4	61,7	4
PF	22	10	34	224	4,9	2,2	14	11	5,0	32
MFL	22	4	40	118	5,3	4,6	15	7,4	6,3	21

^{a, b} See Table A.1, except that \bar{w} is the mean copper content.

Table A.3 — Statistical results for iron

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVr %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	12	2	22	7 500	141	1,9	400	565	7,3	1 600
P2	12	2	22	22 600	565	2,5	1 600	1 555	6,9	4 400
P3	12	0	24	31 000	707	2,4	2 000	2 473	8,5	7 000
PO1	12	0	24	4 000	106	3,0	300	318	8,3	900
PO2	12	0	24	500	18	3,7	50	28	6,0	80
PO3	12	0	24	9 600	212	2,2	600	636	6,7	1 800
PO4	12	4	20	18 600	177	0,9	500	1 166	6,2	3 300
PO5	12	0	24	19 000	353	2,6	1 000	2 120	11,1	6 000
B	21	4	38	79	2,8	3,7	8	11	13,9	31
WS	21	8	34	599	10	1,7	29	70	11,6	197
MFH	21	2	40	362	59	16,2	166	85	23,5	241
MP	21	4	38	8	2,5	30,5	7	5,7	68,5	16
PF	21	4	38	3 430	86	2,5	244	491	14,4	1 390
MFL	21	6	36	1 290	40	3,1	113	111	8,6	315

^{a, b} See Table A.1, except that \bar{w} is the mean iron content.

Table A.4 — Statistical results for magnesium

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVR %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	12	0	24	111 000	2 120	1,9	6 000	5 654	5,2	16 000
P2	12	2	22	18 000	106	0,5	300	742	4,2	2 100
P3	12	0	24	2 050	53	2,5	150	152	7,4	430
PO1	12	2	22	5 770	113	2,0	320	145	2,5	410
PO2	12	2	22	1 640	28	1,7	80	49	3,2	140
PO3	12	0	24	1 000	32	3,2	90	67	6,6	190
PO4	12	0	24	1 670	42	2,5	120	110	6,6	310
PO5	12	2	22	1 650	28	1,6	80	71	4,2	200
B	22	10	34	1 200	34	2,8	95	58	4,8	163
WS	22	8	36	2 260	80	3,5	226	128	5,7	363
MFH	22	8	36	1 640	45	2,7	126	120	7,3	340
MP	22	4	40	1 250	94	7,5	265	147	11,8	417
PF	22	8	36	9 980	128	1,3	362	576	5,8	1 630
MFL	22	10	34	3 040	27	0,9	76	143	4,7	405

^{a, b} See Table A.1, except that \bar{w} is the mean magnesium content.

Table A.5 — Statistical results for manganese

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVR %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	13	0	26	2 600	71	3,3	200	318	12,0	900
P2	13	0	26	4 800	106	2,6	300	742	15,6	2 100
P3	13	0	26	10 100	247	2,4	700	883	8,8	2 500
PO1	13	0	26	1 700	71	4,2	200	212	12,6	600
PO2	12	0	24	159	5	3,1	14	18	11,2	50
PO3	13	2	24	13 200	353	2,8	1 000	989	7,5	2 800
PO4	13	4	22	4 870	78	1,6	220	166	3,4	470
PO5	13	0	26	5 600	141	2,3	400	530	9,5	1 500
B	22	6	38	16	0,4	2,4	1	3,2	19,8	9
WS	22	4	40	174	2,5	1,4	7	14	8,2	41
MFH	22	4	40	65	2,1	3,1	6	11	17,1	32
MP	22	8	36	1	0,4	26,6	1	0,7	52,0	2
PF	22	2	42	417	8,1	2,0	23	66	15,8	187
MFL	22	4	40	361	3,5	1,0	10	35	9,8	100

^{a, b} See Table A.1, except that \bar{w} is the mean manganese content.

Table A.6 — Statistical results for potassium

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVr %	r m/kg	s_R mg/kg	CVR %	R mg/kg
P1	11	0	22	700	71	8,3	200	212	33,9	600
P2	11	0	22	570	120	21,4	340	212	37,5	600
P3	11	0	22	1 000	71	7,7	200	247	25,0	700
PO1	11	2	20	3 700	141	3,7	400	459	12,8	1 300
PO2	11	0	22	10 000	247	2,4	700	989	9,9	2 800
PO3	11	0	22	2 400	71	3,4	200	247	9,9	700
PO4	11	2	20	14 900	495	3,3	1 400	777	5,2	2 200
PO5	11	0	22	3 900	141	3,7	400	318	8,1	900
B	17	2	32	4 850	118	2,4	334	746	15,4	2 110
WS	17	4	30	30 100	551	1,8	1 560	2 237	7,4	6 330
MFH	17	2	32	6 330	160	2,5	454	898	14,2	2 540
MP	17	2	32	17 100	583	3,4	1 650	1 336	7,8	3 780
PF	17	2	32	11 200	505	4,5	1 430	936	8,3	2 650
MFL	17	6	28	17 800	746	4,2	2 110	1 155	6,5	3 270

^{a, b} See Table A.1, except that \bar{w} is the mean potassium content.

Table A.7 — Statistical results for sodium

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVr %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	11	0	22	237 000	636	2,7	1 800	12 721	5,4	36 000
P2	11	2	20	5 900	212	3,3	600	777	13,5	2 200
P3	11	0	22	155 000	3 536	2,3	10 000	9 187	5,9	26 000
PO1	11	0	22	33 900	1 025	3,0	2 900	2 155	6,4	6 100
PO2	11	0	22	1 840	78	4,3	220	212	11,5	600
PO3	11	2	20	680	120	17,6	340	120	17,9	340
PO4	11	0	22	660	110	16,7	310	141	21,3	400
PO5	11	0	22	580	134	23,0	380	155	26,8	440
B	15	8	22	117	10	8,7	29	20	17,3	57
WS	16	6	26	3 830	187	4,9	529	258	6,7	729
MFH	15	4	26	1 240	84	6,9	238	170	13,7	480
MP	16	6	26	4 210	142	3,4	402	246	5,8	696
PF	16	2	30	1 890	119	6,3	336	309	16,4	875
MFL	16	2	30	5 650	375	6,6	1 060	406	7,2	1 150

^{a, b} See Table A.1, except that \bar{w} is the mean sodium content.

Table A.8 — Statistical results for zinc

Sample ^a	Parameter ^b									
	n_1	n_2	n_3	\bar{w} mg/kg	s_r mg/kg	CVR %	r mg/kg	s_R mg/kg	CVR %	R mg/kg
P1	13	2	24	5 200	177	3,1	500	353	7,1	1 000
P2	13	2	24	8 700	141	1,7	400	671	7,8	1 900
P3	13	4	22	14 600	318	2,3	900	459	3,2	1 300
PO1	13	0	26	3 500	141	4,0	400	353	10,4	1 000
PO2	13	0	26	260	21	7,6	60	39	15,0	110
PO3	13	0	26	10 900	318	2,9	900	1 237	11,4	3 500
PO4	13	2	24	10 000	247	2,4	700	671	6,6	1 900
PO5	13	2	24	11 900	283	2,3	800	565	4,9	1 600
B	22	8	36	29	1,1	3,4	3	2,8	9,4	8
WS	22	12	32	44	1,1	2,1	3	1,8	4,3	5
MFH	22	6	38	47	3,2	6,5	9	3,9	8,3	11
MP	22	8	36	46	2,1	4,5	6	3,2	7,0	9
PF	22	8	36	569	11	2,0	32	33	5,8	94
MFL	22	6	38	106	3,5	3,4	10	7,1	6,7	20

^{a, b} See Table A.1, except that \bar{w} is the mean zinc content.

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