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Milk and milk products — Determination of calcium, sodium, potassium and magnesium contents — Atomic absorption spectrometric method

Lait et produits laitiers — Détermination des teneurs en calcium, sodium, potassium et magnésium — Méthode spectrométrique par absorption atomique

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8070|IDF 119 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This second edition of ISO 8070|IDF 119 cancels and replaces the first edition (ISO 8070:1987), which has been technically revised.

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Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the IDF National Committees casting a vote.

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

ISO 8070|IDF 119 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the Joint ISO-IDF Action Team *Minor compounds*, of the Standing Committee on *Minor components and characterization of physical properties*, under the aegis of its project leaders, Mr L. Noël (FR) and Mr. M. Carl (DE).

This edition of ISO 8070|IDF 119 cancels and replaces IDF 119A:1987 and IDF 154:1992, which have been technically revised.

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Milk and milk products — Determination of calcium, sodium, potassium and magnesium contents — Atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of calcium, sodium, potassium and magnesium contents in milk and milk products.

The method is applicable for milk and whey, buttermilk, yogurt, cream, dried milk, butter, cheese, casein and caseinate.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
calcium, sodium, potassium, magnesium contents
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The respective contents are expressed in milligrams per gram.

4 Principle

The organic matter is decomposed by dry ashing or by wet digestion using nitric acid either in an open microwave-assisted wet digestion system or in a pressurized microwave-assisted wet digestion system or in a pressurized polytetrafluoroethylene (PTFE) decomposition vessel or any appropriate instrumentation in wet digestion. The ash containing calcium, sodium, potassium, and magnesium is dissolved in a nitric acid solution in the case of dry ashing, or the digests diluted in the case of wet digestion. The test and calibration solutions are atomized into an air–acetylene flame of an atomic absorption spectrometer and their absorption is measured at appropriate wavelengths.

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5 Reagents

Use reagents of recognized analytical grade, unless otherwise specified, and water complying with grade 2 in accordance with ISO 3696, unless otherwise stated.

SAFETY PRECAUTIONS — When using acids, operators should protect themselves by wearing glasses and gloves. Manipulation of acids shall be done under an appropriate fume hood.

5.1 Nitric acid (HNO_3), concentrated, with a mass fraction of 65 %.

5.2 Nitric acid (HNO_3) solution, with a volume fraction of 25 %.

Dilute 25 ml of nitric acid (5.1) to 100 ml with water and mix.

5.3 Lanthanum trichloride solution, with $c(LaCl_3.7H_2O)$ = 27 g/l.

Dissolve 27 g of lanthanum trichloride heptahydrate ($LaCl_3.7H_2O$) in an amount of water. Dilute to 1 litre and mix.

5.4 Calcium ion stock solution.

Commercially available or equivalent corresponding to $c(Ca^{2+})$ = 1 g/l.

5.5 Sodium ion stock solution.

Commercially available or equivalent corresponding to $c(Na^{+})$ = 1 g/l.

5.6 Potassium ion stock solution.

Commercially available or equivalent corresponding to $c(K^{+})$ = 1 g/l.

5.7 Magnesium ion stock solution.

Commercially available or equivalent corresponding to $c(Mg^{2+})$ = 1 g/l.

5.8 Standard working solution, containing 100 mg/l calcium, 20 mg/l sodium, 20 mg/l potassium and 10 mg/l magnesium ions.

Pipette 10 ml of calcium ion stock solution (5.4), 2 ml sodium ion stock solution (5.5), 2 ml potassium ion stock solution (5.6) and 1 ml magnesium ion stock solution (5.7) into a 100 ml one-mark volumetric flask (6.3) and mix. Add 5 ml of nitric acid solution (5.2). Dilute to the 100 ml mark with water and mix again.

Store the standard working solution in a PE-HD bottle (6.7) so that any contamination is avoided.

5.9 Light petroleum (petroleum ether), with a boiling range of between 40 °C and 60 °C.

Distil the light petroleum, if necessary, in a contamination free distillation unit.

5.10 Hydrogen peroxide (H_2O_2), with a volume fraction of 30 %.

6 Apparatus

6.1 General

Keep the clean glassware in a nitric acid solution with a mass fraction of about 10 %. Clean all glassware and plastic ware thoroughly with the 10 % nitric acid and keep it in that solution for at least 6 h. Before use, rinse the glassware and plastic ware three times with double-distilled water and allow it all to dry.

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Store the clean glassware and plastic ware in a dust-free environment to ensure that they are free from any contamination when used.

Usual laboratory apparatus, and in particular, the following.

6.2 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

6.3 One-mark volumetric flasks, of nominal capacities 20 ml, 50 ml, 100 ml, 250 ml and 1000 ml, complying with the requirements of ISO 1042.

6.4 One-mark pipettes, of nominal capacities 1 ml, 2 ml, 5 ml and 10 ml, complying with the requirements of ISO 648.

6.5 Micropipette, capable of adjusting to between 1 ml and 5 ml, with plastic pipette tips.

6.6 Graduated measuring cylinder, of capacity 10 ml.

6.7 High-density polyethylene (PE-HD) bottles, capable of storing the standard and sample solutions.

6.8 Silica crucibles, of capacity between 25 ml and 50 ml.

6.9 Programmable furnace oven, capable of attaining a minimum temperature of 550 °C ± 25 °C and of programming with a heating rate of 50 °C/h.

6.10 Open focused microwave-assisted wet digestion system, delivering 200 W microwave power, equipped with appropriate vessels of capacity 50 ml, with an adapted cooling system.

6.11 Pressurized microwave-assisted wet digestion system, with operator selectable output of between 0 W and 1 000 W microwave power, provided with temperature and pressure controllers and an air cooling device, equipped with appropriate vessels of capacity 50 ml, commercially available or equivalent.

6.12 Decomposition vessels, made of stainless steel, with adapted PTFE inner vessels of capacity 23 ml with screw caps (pressurized wet decomposition PTFE vessel), commercially available or equivalent.

6.13 Oven, capable of attaining a temperature of 150 °C (for digestion bombs).

6.14 Flame atomic absorption spectrometer, with an air–acetylene burner, suitable for measuring at different wavelengths: at 422,7 nm for calcium, at 589,6 nm for sodium, at 766,5 nm for potassium and 285,2 nm for magnesium ion content-determination procedures; equipped with hollow cathode lamps of single element type or combined type.

6.15 Water baths, capable of maintaining temperatures of 20 °C ± 2 °C, at 40 °C ± 1 °C, at 45 °C ± 1 °C, and at 65 °C ± 1°C.

6.16 Centrifuge, capable of producing a radial acceleration of 2 500g, with tubes of capacity at least 150 ml.

6.17 Appropriate grinding device.

6.18 Sieve, free of contaminated material, with nominal size of aperture 0,5 mm.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707 | IDF 50.

Store the test sample in such a way that deterioration and change in its composition are prevented.

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8 Preparation of test sample

Avoid any contamination of the test sample.

8.1 Milk and whey

Place the test sample in the water bath (6.15) set at 20 °C and mix carefully. If in the case of milk the fat is not evenly dispersed, warm the test sample slowly in a water bath (6.15) set at 40 °C. Mix gently by inversion only. When the sample is mixed thoroughly then cool quickly again in the water bath (6.15) set at 20 °C.

8.2 Buttermilk

Remove, if necessary, any butter granules. Immediately before weighing (9.1.1.1 or 9.1.2.1), place the test sample in the water bath (6.15) set at 20 °C and mix carefully.

8.3 Yogurt

Place the test sample in the water bath (6.15) set at 20 °C and mix carefully. If the serum separates, stir the sample vigorously immediately before weighing (9.1.1.1 or 9.1.2.1).

8.4 Cream

Place the test sample in the water bath (6.15) set at 20 °C. Mix or stir thoroughly but not so vigorously as to cause frothing or churning. If the cream is very thick, or if the fat is not evenly dispersed, warm the sample slowly in a water bath (6.15) set at 40 °C to facilitate mixing. Cool the test sample quickly in the water bath (6.15) set at 20 °C.

NOTE Reliable results cannot be expected if adequate mixing of the test sample is not achieved or if the test sample shows any evidence of churning or any other signs of abnormality.

8.5 Dried milk

Transfer the test sample into a container of capacity about twice the volume of the sample and provided with an airtight lid. Close the container immediately. Mix the milk powder thoroughly by repeatedly shaking and inverting the container.

8.6 Butter

Because of the possible inhomogeneous distribution of the ions in butter they are determined in the serum.

NOTE The ion contents in the fat fraction, obtained from butter in the way described, are negligible compared with the contents in the serum and can be ignored.

Weigh, to the nearest 100 mg, 100 g of test sample in a dry centrifuge tube (6.16), of which the mass has been determined previously. Place the tube in a water bath (6.15) set at 45 °C. As soon as the butter is melted, centrifuge the tube with a radial acceleration of 2 500g.

Remove as much as possible the clear fat layer from the tube by using a pipette. Add 10 ml light petroleum (5.9) to dilute the remaining fat layer in the tube and remove the mixture again with a pipette. Repeat the addition and removal of light petroleum mixture twice.

Remove the residual light petroleum by warming the tube in a water bath (6.15) set at 65 °C. Cool in the water bath (6.15) set in advance at 20 °C. Dry the outside of the tube with a clean paper tissue. Weigh the tube and its contents to the nearest 100 mg. Mix the contents carefully immediately before weighing the test sample (9.1.1.1 or 9.1.2.1).

NOTE The butter can also be digested directly if dry ashing or pressurized microwave-assisted wet digestion is used, in which the butter is warmed to say 30 °C, homogenized by thorough stirring, and a test portion taken directly.

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8.7 Cheese

Remove the rind, smear or mouldy surface layer of the cheese, in such a way as to provide a test sample representative of the cheese as it is usually consumed. Grind the test sample by means of an appropriate device (6.17). Quickly mix the whole mass and preferably grind the mass again quickly.

If the test sample (e.g. soft cheese) cannot be ground, mix the whole sample thoroughly. Transfer the pretreated sample, or a representative part of it, immediately into a container provided with an airtight lid.

Analyse the test sample without delay, as soon as possible after grinding. Ground cheese showing unwanted mould growth or beginning to deteriorate should not be examined.

8.8 Caseins and caseinates

8.8.1 If most of the test sample is fine enough to pass through the sieve (6.18), it may be used without any grinding. Transfer about 50 g of sieved test sample to a container of capacity about twice the volume of test sample and provided with an airtight lid.

Close the container immediately. Mix the test sample thoroughly by repeatedly shaking and inverting the container.

8.8.2 If most of the test sample is not fine enough to pass through the sieve (6.18), grind about 50 g of test sample until most of it does pass. Transfer all sieved test sample to a container. Continue the procedure as described in 8.8.1.

9 Procedure

9.1 Test portion

NOTE If it is required to check whether the repeatability requirement is met, carry out two single determinations under repeatability conditions.

9.1.1 Test portion for dry ashing

9.1.1.1 Milk, yogurt, cream, whey, butter and buttermilk

Weigh, to the nearest 1 mg, 10 g of prepared test sample (8.1 to 8.4, 8.6) in the silica crucible (6.8).

9.1.1.2 Dried milk, cheese, casein and caseinate

Weigh, to the nearest 1 mg, 1 g of prepared test sample (8.5, 8.7, 8.8) in the silica crucible (6.8).

9.1.2 Test portion for wet digestion

WARNING — When using a system operating under pressure (pressurized PTFE decomposition vessel or pressurized microwave-assisted wet decomposition system), special attention should be paid to avoid any risk of explosion. In particular, the size of test portion has to be especially well considered. In a wet decomposition vessel of about 25 ml, no more of any sample shall be digested corresponding to an amount of dry matter of 200 mg (the total amount of sample shall be no more than 1 g). The oven in which the digestion is carried out shall be placed in a hood.

9.1.2.1 Milk, yoghurt, cream, whey, butter, buttermilk

Weigh, to the nearest 1 mg, 0,5 g to 1 g of prepared test sample (8.1 to 8.4, 8.6) in the microwave vessel (6.10 or 6.11) or in the PTFE vessel (6.12).

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9.1.2.2 Dried milk, casein, caseinate or cheese

Weigh, to the nearest 1 mg, 0,2 g to 0,5 g of prepared test sample (8.5, 8.7, 8.8) in the microwave vessel (6.10 or 6.11) or in the PTFE vessel (6.12).

9.2 Decomposition of organic matter

9.2.1 Dry ashing

Place the silica crucible (9.1.1.1 or 9.1.1.2) into the programmable furnace oven (6.9) set at room temperature. Start the heating program of the oven which includes the following steps: for drying and pre-ashing steps increase the temperature by 50 °C/h up to 550 °C. Maintain the temperature of the oven at 550 °C for 6 h.

If the obtained ash of the test portion still has a grey colour after cooling down, dissolve the ash in 1 ml of nitric acid solution (5.2). Continue the dry ashing procedure by restarting it again at the beginning of 9.2.1.

9.2.2 Wet digestion

9.2.2.1 Microwave assisted digestion

Either use the open focused (9.2.2.1.1) or the pressurized (9.2.2.1.2) microwave-assisted wet digestion system.

9.2.2.1.1 Open microwave-assisted wet digestion system

Apply the decomposition program using the open microwave-assisted wet system (6.10) mentioned in Table 1.

NOTE The parameters such as type and volume of reagent to be added, the microwave power and decomposition time can be modified according to the type and size of test sample to be analysed.

Table 1 — Open microwave-assisted digestion system — Decomposition programme

Step	Add reagent	Volume ml	Power W	Time min
1	H_2O (distilled)	2	—	—
2	HNO_3 (5.1)	7	30	5
3	—	—	80	15
4	H_2O_2 (5.10)	1	60	5

9.2.2.1.2 Pressurized microwave-assisted wet digestion system

Add 3 ml of nitric acid solution (5.2) in the wet microwave vessel (6.11) before closing it. Place the vessel into the microwave oven (6.11). Apply the decomposition program with the pressurized system mentioned in Table 2.

NOTE The parameters such as type and volume of reagent to be added, the microwave power and decomposition time can be modified according to the type and size of test sample to be analysed.

Table 2 — Pressurized microwave-assisted digestion system — Decomposition programme

Step	Outset power W	Time min	Final power W	Cooling system
1	500	10	800	Low
2	800	20	1 000	Low
3	0	20	0	High

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9.2.2.2 Decomposition vessel

Add 3 ml of nitric acid solution (5.2) in the decomposition vessel (6.12) before closing it. Place the vessel in the oven (6.13) set at room temperature. Increase the oven temperature to 150 °C and keep the vessel at 150 °C for at least 3 h.

9.3 Determination

9.3.1 Preparation of the test solution

9.3.1.1 Dry ashing

Dissolve the obtained ash (9.2.1) in 1 ml of nitric acid solution (5.2). Transfer quantitatively the crucible content into a 250 ml one-mark volumetric flask (6.3) by rinsing with water. Dilute to the 250 ml mark with water. Mix thoroughly and continue the dilution procedure in 9.3.1.3.

9.3.1.2 Wet digestion

Cool the digested solution (9.2.2) firstly to room temperature while reducing to atmospheric pressure before transferring it quantitatively into a 50 ml one-mark volumetric flask (6.3). Dilute to the 50 ml mark with water. Mix thoroughly and continue with the dilution procedure (9.3.1.3.).

9.3.1.3 Dilution

According to the type of test sample and the ion measured, dilute (dilution factor, f_1) the test solution (either 9.3.1.1 or 9.3.1.2) by using the micropipette (6.5) in the required one-mark volumetric flasks (6.3). Add a volume fraction of 10 % (one tenth of the measuring flask volume) of the lanthanum trichloride solution (5.3) by using a graduated measuring cylinder (6.6). Dilute to the mark of the volumetric flask used with water.

9.3.2 Blank test

In parallel with the procedure of the test portion, carry out a blank test using the same procedure and the same amount of each reagent being added in the decomposition (9.2) and the determination (9.3) steps of the test portion.

9.3.3 Flame atomic absorption spectrometric measurement

Adjust the flame spectrometer (6.14) and its flame conditions according to the manufacturer's recommendations in order to yield optimum precision and sensitivity. Set the spectrometer at the required wavelength depending on the ion (analyte) to be determined (see 6.14).

9.3.3.1 Calibration

The volumes and corresponding concentrations are only given for guidance. Select both within the linear range of the particular instrument used (at least five concentrations including zero member).

Transfer, by using the micropipette (6.5), each of the volumes 0 ml (zero member), 1,0 ml, 2,0 ml, 3,0 ml, 4,0 ml, and 5,0 ml of standard working solution (5.8) into six 100 ml one-mark volumetric flasks (6.3). Add 10 ml of lanthanum trichloride solution (5.3) to each flask. Dilute to the 100 ml mark with water and mix. The calibration solutions obtained are listed in Table 3.

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Table 3 — Calibration solutions

Flask No	Calcium ion solution mg/l	Sodium ion solution mg/l	Potassium ion solution mg/l	Magnesium ion solution mg/l
1	0	0	0	0
2	1,0	0,2	0,2	0,1
3	2,0	0,4	0,4	0,2
4	3,0	0,6	0,6	0,3
5	4,0	0,8	0,8	0,4
6	5,0	1,0	1,0	0,5

9.3.3.2 Calibration graphs

For each individual ion, subsequently aspirate the zero member solution and the five calibrating solutions three times each. Calculate the mean of the absorbance values. Subtract the mean of the absorbance value of the zero member solution from the means of absorbance values. Plot the resulting net absorbance values against the corresponding ion concentration.

NOTE Depending on the instrument facilities; the subtraction can also be done by auto-zeroing.

9.3.3.3 Measurement of test solution

Measure the absorbance values of the test solution (9.3.1) and the blank test (9.3.2) immediately after the calibration measurements under the same conditions for each ion. Dilute (dilution factor, f_2) the test solution with the zero member solution (see 9.3.3.1) if its signal is above that of the highest standard. Add to each dilution such an amount of lanthanum trichloride solution (5.3) as to obtain a final volume fraction of it in the solution of 10 %. Repeat the measurements. In order to check for any drifting during measurement, measure at least one calibrating solution at the end of series for each ion.

For each test solution, repeat the measurement three times. Calculate the average of the absorbance values. Subtract from the obtained average value the mean absorbance value of the blank. Take the corrected average value thus obtained to read the corresponding content from the calibration graph (9.3.3.2).

10 Calculation and expression of results

10.1 Calculation

Calculate the ion content, w, by using the following equation:

$$w = \frac{c \times V}{m \times 1000} \times f_1 \times f_2$$

where

w is the ion (one of Ca^{2+}, Na^{+}, K^{+}, Mg^{2+}) mass fraction, expressed in milligrams per gram, of the test sample;

c is the ion concentration, in milligrams per litre, in the test solution (9.3.1) read from the calibration graph (9.3.3.2);

V is the volume, in millilitres, of the flask in which the dry ashes (V = 250 ml) or digested solutions (V = 50 ml) are transferred quantitatively (9.3.1);

m is the mass, in grams, of the test sample used in the procedure (9.1.1 or 9.1.2) (for butter, take m as the mass of the butter sample corresponding to the mass of the serum sample used in testing, see 8.6);

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f_1 is the dilution factor of the test solution carried out during the preparation step (9.3.1.3);

f_2 is the dilution factor of the test solution carried out during the measurement step (9.3.3.3).

10.2 Expression of test results

Express the test results to three decimal places.

11 Precision

The values for repeatability and reproducibility limit were derived from the results of an interlaboratory test carried out in accordance with ISO 5725-1 and ISO 5725-2. A full report of the test is published in an IDF Bulletin [6].

The values are expressed for the 95 % probability level and may not be applicable to content ranges and matrices other than those given, in particular for contents near the limit of determination (see example acid casein in Annex A).

NOTE In the aforementioned interlaboratory test, the test samples were taken from the same solution after mineralization, also analysed in parallel by inductively coupled plasma-optical emission spectroscopy (ICP-OES). From this design and its results it can be concluded that:

a) precision data are — with exception of potassium — generally better for ICP-OES compared to atomic absorption spectrometry (AAS) determination;

b) both methods resulted in well comparable mean values, being slightly but not significantly higher for ICP-OES. The AAS method described in this International Standard can therefore also be regarded as accurate;

c) for practical use, both AAS determination and ICP-OES determination can be regarded as equivalent with respect to their results.

11.1 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 be greater than:

for sodium (Na^+): 13 %;
for potassium (K^+): 10 %;
for calcium (Ca^{2+}) 8 %; and
for magnesium (Mg^{2+}) 8 %.

NOTE Each percentage is expressed relative to the arithmetic mean of the results, for sodium, potassium, calcium and magnesium, respectively.

11.2 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than:

for sodium (Na^+): 19 %;
for potassium (K^+): 16 %;
for calcium (Ca^{2+}) 19 %; and
for magnesium (Mg^{2+}) 13 %.

NOTE Each percentage is expressed relative to the arithmetic mean of the results, for sodium, potassium, calcium and magnesium, respectively.

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12 Test report

The test report shall specify:

a) all the information required for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, together with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s);

e) the test result(s) obtained and, if the repeatability has been checked, the final quoted results obtained.

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Annex A
(informative)

Interlaboratory trials

International collaborative tests involving 8 to 13 laboratories were carried out on two different samples of each milk product listed in the tables containing different sodium, potassium, calcium and magnesium ion mass fractions. The test was organized by MUVA, Kempten, Germany.

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Tables A.1 to A.4.

Table A.1 — Interlaboratory study result for sodium

Sample	No. of labs with valid results (outliers)	Mean of valid results g/kg	Standard deviation of repeatability, s_r	Repeatability value, $r = 2{,}8 s_r$	Coefficient of variation of repeatability, CV(r) %	Standard deviation of reproducibility, s_R	Repro-ducibility value, $R = 2{,}8 s_R$	Coefficient of variation of reproducibility, CV(R) %	$CV(R)_H$ [a] %	Horwitz ratio HorRat[b]
Control solution	8(1)	40,2 (mg/l)	0,7	2,0	1,7	0,7	2,0	1,6	9,2	0,2
Ref. material BCR 063	12	4,34	0,17	0,49	4,0	0,30	0,85	7,0	4,5	1,5
Whey protein concentrate	13	1,74	0,10	0,29	6,1	0,16	0,44	9,2	5,2	1,8
Whole milk powder	13	2,99	0,14	0,40	4,8	0,26	0,72	8,6	4,8	1,8
Processed cheese I	10(1)	8,17	0,29	0,82	3,6	0,29	0,82	3,6	4,1	0,9
Whey powder	13	6,30	0,29	0,81	4,6	0,47	1,33	7,5	4,3	1,7
Casein (acid)	8(1)	0,040	0,041	0,11	102	0,046	0,13	114	9,2	12
Processed cheese II	12(1)	6,06	0,25	0,71	4,2	0,43	1,19	7,0	4,3	1,6
Freeze dried cheese	13	16,8	0,5	1,5	3,2	0,7	1,9	4,0	3,7	1,1
Skim milk powder	13	4,05	0,26	0,73	6,4	0,29	0,82	7,2	4,6	1,6

[a] coefficient of variation of reproducibility $[(s_R/x) \times 100]$ calculated from the Horwitz equation, i.e.

$$CV(R)_H = 2^{(1-0{,}5 \lg w)}$$

where w is the mass fraction (i.e. 1 = 100 g/100 g, 0,001 = 1 g/kg)

[b] $CV(R)/CV(R)_H$ (see Horwitz, 1982[4]); the Horwitz ratio gives a comparison of the actual precision measured with the precision predicted by the Horwitz equation for a method measuring at that particular level of analyte.

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