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Milk, milk products, infant formula and adult nutritionals — Determination of minerals and trace elements — Inductively coupled plasma atomic emission spectrometry (ICP-AES) method

Lait, produits laitiers, formules infantiles et produits nutritionnels pour adultes — Détermination de la teneur en minéraux et en oligo-éléments — Méthode par spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES)

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

International Dairy Federation
Silver Building • Bd Auguste Reyers 70/B
B-1030 Brussels
Phone: +32 2 325 67 40
Fax: +32 2 325 67 41
Email: info@fil-idf.org
Website: www.fil-idf.org

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Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products* and the International Dairy Federation (IDF), in collaboration with AOAC INTERNATIONAL.

It is being published jointly by ISO and IDF and separately by AOAC INTERNATIONAL. The method described in this document is equivalent to the AOAC Official Method 2011.14: *Minerals and Trace Elements in Infant Formula*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

ISO and IDF collaborate closely on all matters of standardization relating to methods of analysis and sampling for milk and milk products. Since 2001, ISO and IDF jointly publish their International Standards using the logos and reference numbers of both organizations.

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It is being published jointly by ISO and IDF and separately by AOAC INTERNATIONAL. The method described in this document is equivalent to the AOAC Official Method 2011.14: *Minerals and Trace Elements in Infant Formula*. All work was carried out by the ISO/IDF Action Team on C17 of the Standing Committee on Analytical Methods for Composition under the aegis of its project leader, Mr H. Cruijsen (NL).

Milk, milk products, infant formula and adult nutritionals — Determination of minerals and trace elements — Inductively coupled plasma atomic emission spectrometry (ICP-AES) method

1 Scope

This document specifies a method for the quantitative determination of calcium (Ca), copper (Cu), iron (Fe), magnesium (Mg), manganese (Mn), phosphorus (P), potassium (K), sodium (Na) and zinc (Zn) using inductively coupled plasma atomic emission spectrometry (ICP-AES). The method is applicable for milk, dried milk, butter, cheese, whey, dried whey, infant formula and adult nutritional formula in the ranges given in Table 1.

Table 1 — Analytical ranges

	Ca	Cu	Fe	Mg	Mn	P	K	Na	Zn
Lower analytical range[a], in mg/100 g	20	0,03	0,5	3	0,01	15	10	10	0,2
Upper analytical range[a], in mg/100 g	1 280	1,2	20	110	1,0	800	2 000	850	18

[a] concentrations apply to

— milk and "ready-to-feed" liquids as-is, using a typical sample size of 4 g per final analytical solution volume of 25 ml and

— reconstituted milk powder, infant formula powders and adult nutritional powders (25 g into 200 g of water), using a typical sample size of mass of the reconstituted slurry per final analytical solution volume of 25 ml.

Ranges for non-reconstituted dairy ingredients (butter, cheese, whey powders, whey protein concentrates) are adjusted proportionally upward from these values based upon the sample size used for the ingredient. For example, if 0,6 g of cheese is digested the ranges will be 4 g/0,6 g = 6,7 × higher.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — Single-volume pipettes*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The organic matter is decomposed by wet digestion using nitric acid in a pressurized microwave assisted wet digestion system or any other appropriate instrumentation for wet digestion and diluted. An internal standard is used. The test and calibration solutions are atomized into plasma of an inductively coupled plasma spectrometer and the emission is measured at appropriate wavelengths using external calibration.

5 Reagents

WARNING — The use of this document can involve hazardous materials, operations and reagents. This document does not propose to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish safety and health practices.

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water, complying with ISO 3696, grade 2, unless otherwise stated**.**

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

5.3 Nitric acid solution (HNO_3), 10 % volume fraction. Add one part of HNO_3 (5.2) to seven parts of water and mix**.**

5.4 Element stock solutions of Ca, P, K (each of mass concentration ρ = 10 000 mg/l) and Cu, Fe, Mg, Mn, Na, Zn (each of ρ = 1 000 mg/l).

Use a suitable element stock solution, preferably a certified one. Both single-element stock solutions and multi-element stock solutions with an adequate specification, stating the acid used (mostly being nitric acid) and the preparation technique, are commercially available. Do not use the element stock solution after the expiry date.

5.5 Standard solutions.

5.5.1 General

Depending on the scope, different multi-element standard solutions can be necessary. In general, when combining multi-element standard solutions, their chemical compatibility and possible hydrolysis of the components shall be taken into account. The examples given below also consider the measuring range of various ICP-AES instruments and the expected concentration of the elements in milk and milk products.

The multi-element standard solutions are considered stable for several months if stored in the dark (observe the expiry date specified by the manufacturer). Other combinations of elements at different concentrations may be used, provided that the element stock solutions (5.4) are diluted with the same acid with a similar concentration that is used for the test solution so as to prepare a range of standards that covers the concentration of the elements to be determined.

Store the standard working solutions in a high-density polyethylene bottle (6.6) so as to avoid any contamination.

5.5.2 Standard working solution of Fe, ρ = 400 mg/l

Pipette 20,0 ml of Fe stock solution (5.4) into a 50 ml volumetric flask. Dilute with water to the 50 ml mark. Transfer the standard working solution to a suitable storage bottle (6.6).

5.5.3 Standard working solution of Cu, Mn and Zn, ρ = 50 mg Cu, 50 mg Mn and 200 mg Zn/l

Pipette 5,00 ml of Cu, 5,00 ml of Mn and 20,0 ml Zn of the concerned element stock solution (5.4) into a 100 ml volumetric flask. Dilute with water to the 100 ml mark. Transfer the standard working solution to a suitable storage bottle (6.6).

5.6 Internal standard solution, for example, yttrium, indium or strontium, ρ = 1 000 mg/l.

5.7 H_2O_2, with a volume fraction of 30 %.

6 Apparatus

Clean all glassware and plasticware, including the microwave digestion vessels, thoroughly with the nitric acid solution (5.3), rinse three times with water (5.1) and allow to dry. Store the clean glassware and plasticware in a dust-free environment to ensure they are free of any contamination when used.

Usual laboratory apparatus, and in particular, the following.

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

6.2 One-mark volumetric flasks, of different nominal capacity, Class A in accordance with ISO 1042.

6.3 One-mark pipettes, of different nominal capacity, complying with the requirements of ISO 648.

6.4 Micropipettes, capable of adjusting to between 0,100 ml and 1,000 ml and to between 1 ml and 5 ml, with plastic pipette tips.

6.5 Dispenser, capable of dispensing between 1 ml and 10 ml.

6.6 High-density polyethylene bottles, for storing the standard and sample solutions.

6.7 Pressurized microwave assisted wet digestion system, with operator selectable output of between 0 W and 1 800 W microwave power, provided with temperature and pressure controllers and an air cooling device, equipped with appropriate vessels of capacity 75 ml, commercially available or equivalent.

6.8 ICP-AES instrument.

The instrument shall be equipped with radial plasma as a minimum requirement. Axial plasma is equally acceptable. Background correction shall also be performed when necessary. Settings of the working conditions [e.g. viewing height, gas flows, radio frequency (RF) or plasma power, sample uptake rate, integration time, number of replicates] shall be optimized according to the manufacturer's instructions.

6.9 Water baths, capable of maintaining a temperature of 20 °C ± 2 °C and 40 °C ± 2 °C**.**

6.10 Appropriate grinding device.

6.11 Sampler tubes.

6.12 Cyclonic spray chamber.

6.13 Concentric nebulizer.

7 Sampling

A representative sample should be sent to the laboratory. It should not be damaged or changed during transport or storage. Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 707 | IDF 50[1].

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

8 Preparation of test sample

8.1 Milk and whey

Place the test sample in the water bath (6.9) 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 (6.9) at 40 °C. Mix gently by inversion only. When the sample is mixed thoroughly, cool it quickly in the water bath (6.9) at 20 °C.

8.2 Dried milk, dried whey and infant formula

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.

8.3 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 a grinding device (6.10). Quickly mix the whole mass and preferably grind the mass again quickly.

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

Analyse the test sample as soon as possible after grinding. Do not examine any ground cheese that shows unwanted mould growth or has started to deteriorate.

9 Procedure

9.1 Test portion

9.1.1 General

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

WARNING — When using a system running under pressure (e.g. pressurized microwave assisted wet decomposition system), pay special attention to avoid any risk of explosion. Consider, in particular, the size of test portion. In a decomposition vessel of about 75 ml, not more of any sample shall be digested corresponding to an amount of dry matter of 1 g.

9.1.2 Milk and whey

Weigh to the nearest 1 mg, 4 g of prepared test sample (8.1) in the microwave vessel (6.7) or digestion tube.

9.1.3 Dried milk, dried whey, infant formula, butter and cheese

Weigh to the nearest 1 mg, 1 g of prepared test sample (8.2) or 0,5 g butter or 0,5 g to 0,8 g cheese (8.3) in the microwave vessel (6.7) for pressurized digestion. Weigh to the nearest 1 mg, 0,4 g to 0,5 g of prepared test sample (8.2) or 0,3 g butter or 0,3 g cheese (8.3) into a digestion tube.

If the repeatability in the samples does not meet the criteria, make a pre-step to reduce variation of homogeneity. Dissolve 25 g powder in 200 g water and then take an aliquot for analysis.

9.1.4 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.

The added quantities may be modified.

9.2 Decomposition of organic matter

9.2.1 Wet digestion

9.2.1.1 General

Either use pressurized microwave assisted wet digestion system (6.7) or any appropriate instrumentation for wet digestion.

9.2.1.2 Pressurized microwave assisted wet digestion system with internal standard

Place the vessels containing the test sample (9.1.2 or 9.1.3) and blank (9.1.4) in a fume cupboard. Add 10 ml of nitric acid (5.2) and 0,125 ml of internal standard solution (5.6). Swirl gently and wait a few min before closing the vessel. Place the vessel into the microwave oven (6.7). Apply the decomposition programme with the pressurized system mentioned in Table 2.

Table 2 — Decomposition programme for pressurized microwave assisted digestion system

Step	Outset power W	Time min	Final temperature °C	Cooling system
1	max. 1 800	20	180 to 200	Low
2	max. 1 800	35	180 to 200	Low
3	0	15	< 50	High

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 analyse.

9.3 Determination

9.3.1 Preparation of the test solution

9.3.1.1 Wet digestion with internal standard

Cool the digested solution (9.2.1.2) to room temperature while reducing to atmospheric pressure.

Add 0,5 ml of H_2O_2 (5.7) to all tubes except the blanks, to neutralize the nitric vapour. Transfer the content of the tube quantitatively in a sampler tube. Dilute to 25 ml with water. Mix thoroughly. If necessary continue with the dilution procedure in 9.3.1.2.