INTERNATIONAL STANDARD

ISO 21424

IDF 243

First edition
2018-11

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

Lait, produits laitiers, formules infantiles et produits nutritionnels pour adultes — Détermination de la teneur en minéraux et en oligo-éléments — Méthode par spectrométrie de masse avec plasma à couplage inductif (ICP-SM)

Reference numbers
ISO 21424:2018(E)
IDF 243:2018(E)

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

Published in Switzerland

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 2015.06: *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.

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. 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.

This document was prepared by the IDF Standing Committee on Analytical Methods for Composition and ISO Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, 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 2015.06: *Minerals and Trace Elements in Infant Formula*. All work was carried out by the ISO/IDF Action Team on C40 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 mass spectrometry (ICP-MS) 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), zinc (Zn), chromium (Cr), molybdenum (Mo) and selenium (Se) using inductively coupled plasma and mass spectrometry (ICP-MS).

The method is applicable for the determination of all 12 elements in infant formula and adult nutritional products. The method is also applicable for milk, milk powder, whey powder, butter and cheese excluding the determination of Cr, because all Cr results were below the quantification limit and reproducibility could not be determined in these matrices[1]. The present method is an extension of ISO 20649 | IDF 235 (AOAC 2011.19[2]) which was validated only for Cr, Mo and Se in infant formula and adult nutritional products.

The ranges given in Table 1 are in scope (see also Table A.1).

Table 1 — Analytical ranges

	Ca	Cu	Fe	Mg	Mn	P	K	Na	Zn	Cr	Se	Mo
Lower analytical range[a], in mg/100 g	3	0,002	0,04	0,7	0,002	3	3	2	0,07	0,002	0,000 6	0,000 2
Upper analytical range[a], in mg/100 g	1 280	1,2	20	110	1,0	800	2 000	850	18	0,16	0,05	0,10

[a] Concentrations apply to

— milk and "ready-to-feed" liquid as-is, using a typical sample size of 1 g per final analytical solution volume of 50 ml, and

— reconstituted milk powder, reconstituted infant formula powder and reconstituted adult nutritional powder (25 g into 200 g of water), using a typical sample size of 1,8 g of the reconstituted slurry per final analytical solution volume of 50 ml.

Ranges for non-reconstituted dairy ingredients (butter, cheese, whey powder, whey protein concentrate) are adjusted proportionally upward from these values based upon the sample size used for the ingredient. For example, if 0,3 g of cheese is digested the ranges will be 1 g/ 0,3 g = 3,3 × higher.

2 Normative references

There are no normative references in this document.

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

Nitric acid, internal standard, and hydrogen peroxide are added to the sample in microwave vessels and the samples are digested at 200 °C using a pre-programmed temperature control digestion. The addition of hydrogen peroxide helps reduce carbon and nitrous oxide levels in the digestate. The presence of carbon in the samples causes signal enhancement of Se. Therefore, to matrix match the samples, carbon in the form of methanol is added to both the standard solutions and the digestate before analysis. An ionization buffer is not necessary because the multielement standards and samples always contain significant amounts of the easily ionized elements. Germanium (Ge, for 11 elements) and tellurium (Te just for Se) are used as internal standards. Analysis is performed by ICP-MS. Polyatomic interferences on the low mass elements are reduced or eliminated by analysing in He collision mode using kinetic energy discrimination (KED). For Se measurements the H_2 gas mode is preferred for increased sensitivity. Quantitation of 12 elements is achieved essentially simultaneously by comparing the analyte/internal standard response ratios in the unknown samples to a standard curve constructed from response ratios of calibration standards.

5 Reagents

WARNING — The use of this document can involve hazardous materials, operations and reagents. This standard 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.

5.1 Methanol, purity ≥ 99,99 %, analytical reagent grade.

5.2 Nitric acid (HNO_3), concentrated, ultrapure reagent grade.

5.3 Nitric acid (HNO_3), concentrated, trace metal grade.

5.4 H_2O_2, with a volume fraction of 30 %, ACS reagent grade.

5.5 Laboratory water, metal-free, organic-free, pyrogen-free, filtered 18 MΩ-cm quality.

5.6 Surfactant, for example, Tergitol®[1] Type 15-S-9, Sigma or equivalent (optional).

5.7 Argon gas, purity ≥ 99,996 %.

5.8 Helium gas, purity ≥ 99,999 9 %.

5.9 Hydrogen gas, purity ≥ 99,999 5 %, for Se analyses (recommended).

5.10 Multi-element standard stock solution.

NIST or NIST-traceable containing Se at mass concentration ρ = 20 µg/l; Cr and Mo at 40 µg/l; Mn and Cu at 0,25 mg/l; Zn at 1 mg/l; Fe at 2,5 mg/l; Mg at 10 mg/l; P at 25 mg/l; Ca and K at 50 mg/l and Na at 25 mg/l in 2 % HNO_3 + trace hydrofluoric acid (HF). This stock standard solution expires on the date given by the manufacturer.

1) Tergitol® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or IDF of the product named.

5.11 Multi-element internal standard stock solution (ISTD stock).

NIST or NIST-traceable containing Ge and Te at 5 mg/l in 2 % HNO_3 + trace HF. This stock standard solution expires on the date given by the manufacturer.

5.12 Tuning and pulse/analogue (P/A) factor tuning stock solutions.

NIST or NIST-traceable containing various elements at concentration levels recommended by the manufacturer. Since this method determines the major elements at relatively high concentrations for an ICP-MS, it is important to understand the solutions needed and the procedure to obtain high quality calibration curves in which the detector is used in both pulse counting and analogue modes. A properly calibrated instrument will deliver the linearity requirements of the method; for example, that calibration residuals are < 4 %, see Clause 9.

5.13 Quality control sample (QCS).

Standard Reference Material (SRM)® 1849a[2)] milk-based hybrid infant/adult nutritional powder with certified values for Ca, Cu, Cr, Fe, Mg, Mn, Mo, P, K, Se, Na, and Zn. Supplied as a unit of 10 packets each containing approximately 10 g of material. This is the recommended control material for this analysis, but other suitable SRMs could be substituted.

6 Preparation of standards and solutions

6.1 Surfactant solution (optional, approximately 5 %)

Add about 700 ml laboratory water to a 1 l plastic bottle containing a polytetrafluoroethylene (PTFE)-coated stirring bar. Place the bottle on a magnetic stirrer and begin stirring at a moderate speed. Slowly add 50 ml surfactant from a graduated cylinder. When the surfactant is dissolved, fill the bottle to approximately 1 000 ml with laboratory water. Transfer to 1 l plastic bottle fitted with a PTFE-constructed dispenser with adjustable volume from 1 ml to 10 ml. This solution is added to the autosampler rinse solution to minimize residue build-up in the spray chamber. It does not otherwise affect the analysis. Expiration: 6 months. Store at room temperature.

6.2 Nitric acid rinse solution for autosampler rinse port, 2 %

Mix 20 ml of concentrated nitric acid (5.2) with 20 ml surfactant solution (6.1) and laboratory water to prepare a total volume of 1 000 ml. Expiration: 3 months. Store at room temperature.

6.3 P/A factor tuning working solution

Dilute and/or combine P/A factor tuning stock solutions (or equivalent) to the manufacturer's recommended dilution level with laboratory water for use at the instrument. Expiration: 6 months. Store at room temperature.

6.4 Calibration blank (Cal Blk) and preparation blank (PB) solution

Add approximately 15 ml laboratory water to a 50 ml volumetric flask. Dispense (using bottle dispenser or pipet) 5 ml nitric acid (5.2) into the same volumetric flask. Pipet (using calibrated digital pipet) 0,5 ml of the ISTD stock and 0,5 ml of methanol (5.1). Dilute to volume with laboratory water. This solution serves as both the calibration blank (Cal Blk) and preparation blank (PB). Use same lots of reagent for samples. Expiration: 2 days. Store at room temperature.

2) SRM 1849a is the trade name of a product supplied by the National Institute of Standards and Technology (NIST). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or IDF of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6.5 Calibration standard solution set

Prepare Cal Blk, Cal Std 1, Cal Std 2, Cal Std 3, and Cal Std 4 standard solutions by pipetting with a volumetric pipet (7.10) 0 ml, 1,00 ml, 5,00 ml, 20,00 ml and 40,00 ml, respectively, of the multi-element standard stock solution into separate 50 ml volumetric flasks or sample tubes. Add 0,5 ml of the ISTD with a volumetric pipet (7.10) or digital pipet calibrated at point-of-use to 0,8 % accuracy), 5 ml (using repipetter or PTFE bottle dispenser) of nitric acid (5.2), and 0,5 ml of methanol (5.1) to each flask. Fill the flasks to volume with laboratory water. Expiration: 2 days. Store at room temperature. The analyte and ISTD concentrations in the calibration standard solutions are shown in Table 2.

Table 2 — Concentrations of standards and ISTD in calibration standard solutions

	Calibration Blank	Calibration Standard 1	Calibration Standard 2	Calibration Standard 3	Calibration Standard 4	ISTD[a]
Na, mg/l	0	0,500	2,50	10,0	20,0	Ge
Mg, mg/l	0	0,200	1,00	4,00	8,00	Ge
P, mg/l	0	0,500	2,50	10,0	20,0	Ge
K, mg/l	0	1,00	5,00	20,0	40,0	Ge
Ca, mg/l	0	1,00	5,00	20,0	40,0	Ge
Cr, µg/l	0	0,800	4,00	16,0	32,0	Ge
Mn, mg/l	0	0,005 00	0,025 0	0,100	0,200	Ge
Fe, mg/l	0	0,050 0	0,250	1,00	2,00	Ge
Cu, mg/l	0	0,005 00	0,025 0	0,100	0,200	Ge
Zn, mg/l	0	0,020 0	0,100	0,400	0,800	Ge
Se, µg/l	0	0,400	2,00	8,00	16,0	Te
Mo, µg/l	0	0,800	4,00	16,0	32,0	Ge

[a] Internal standard (ISTD) added at 50 µg/l.

7 Apparatus

7.1 Inductively coupled plasma mass spectrometer.

It shall contain a quartz spray chamber, quartz torch, Ni/Pt sample cone, Ni/Pt skimmer cone, autosampler and printer. The ICP mass spectrometer shall have collision reaction cells (CRCs). In two separate interlaboratory studies, four different model ICP-MS instruments from three major vendors delivered equivalent performance[1][2].

7.2 Microwave oven.

Commercial microwave designed for laboratory use at 0 °C to 300 °C, with closed vessel system and controlled temperature ramping capability. Use vessels recommended by the manufacturer. In the interlaboratory study, five different microwave designs delivered equivalent performance[1].

CAUTION — Microwave operation involves hot pressurized acid solution. Use appropriate face protection and laboratory clothing.

7.3 Hydrogen generator, recommended for better Se sensitivity.

On-demand supply of > 99,999 % pure hydrogen at > 150 ml/min. Alternatively, a high pressure cylinder (99,999 % purity) may be used.

7.4 Magnetic stir plate and PTFE-coated magnetic stir bars.

7.5 Analytical balance.

7.6 Fume hood.

7.7 Common laboratory glassware/plasticware.

7.8 Repipetter, 50 ml.

7.9 Bottle top dispenser, PTFE, adjustable volume 0,5 ml to 5 ml.

7.10 Volumetric pipets, Class A in accordance with ISO 1042[3], assorted sizes.

7.11 Digital pipets, 1 ml, adjustable, to deliver 500 µl with accuracy tolerance of better than 0,8 % and precision of better than 0,2 % relative standard deviation (RSD).

8 Preparation of test sample

Make a single sample preparation to determine all 12 elements.

NOTE ISO 20649 | IDF 235[4] for Cr, Mo, and Se requires duplicate samples to be taken and their results averaged, but comparable reproducibility was obtained in a second inter-laboratory study with the 12-element method described in this document in which only a single sample was analysed[1].

In sample vessels, weigh test portions to the nearest 0,000 1 g. For liquid products (including milk), the test portion size is 1,0 g. Liquid samples shall be thoroughly shaken (5 min in a mechanical shaker is appropriate), the container opened and the contents dumped into a plastic container into which a magnetic stir bar is placed. While stirring, remove the 1 g sample with a disposable pipet for weighing directly into the tared microwave vessel. For ingredients such as whey powder or whey protein concentrate use a direct mass of 0,3 g.

For powdered products, including whole milk powders, the test portion size is net 0,20 g of a powder sample, which should be taken from a 25 g powder and 200 g warm (60 °C) laboratory water reconstitution (i.e. 1,8 g of the 11,1 % reconstitution).

For butter or processed cheese (take a mould-free portion) use a direct mass of 0,3 g.

After weighing the sample, add 0,5 ml of ISTD stock using a digital pipet, 5 ml of nitric acid (5.2), and 2 ml of 30 % hydrogen peroxide.

The PB/Cal Blk solution prepared with the standards is the correct sample blank for this method. Specifically, do not microwave digest the sample blank, which can subject the blank to contamination[5]. The digital pipet used for the addition of ISTD solution shall be calibrated at point of use to ensure that it delivers a volume of 0,500 ml to a tolerance of better than 0,8 % and precision better than 0,2 % RSD.

Seal the vessels, and place into microwave oven. Execute a heating programme equivalent to that shown in Table 3, suitable for total digestion of the sample.

After digestion, place vessels in a fume hood. Unscrew the cap/venting nut slowly to gradually release the pressure. Then, completely remove the cap.

Slowly add approximately 20 ml of laboratory water to the contents of the vessel and transfer contents to a 50 ml sample vial. Add 0,5 ml methanol (5.1) to the sample vial and dilute to approximately 50 ml with laboratory water. Shake briefly. The transfer or the final volume does not need to be quantitative because internal standards were added prior to digestion. Therefore, the analyte/ISTD ratios will be constant.

Table 3 — Microwave operating parameters

Stage 1 sample digestion		
1	Power	100 % (1 600 W)
2	Ramp to temp., min	20
3	Hold time	20
4	Temp., °C	180
5	Cool down, min	20
Stage 2 sample digestion		
1	Power	100 % (1 600 W)
2	Ramp to temp., min	20
3	Hold time	20
4	Temp., °C	200
5	Cool down, min	20
Total, h		2
NOTE Stages 1 and 2 are operated sequentially, without removing vessels from the oven.		

9 Determination

Using the appropriate tuning solutions, tune the instrument for optimal sensitivity in the kinetic energy discrimination mode and/or reaction mode according to the instrument design. Also, tune the instrument to find the P/A calibration factors that are needed for those calibration curves that will extend above roughly 100 µg/l (depends on instrument type). Table 4 summarizes typical instrument parameters for analysis.

Analyse test solutions using an ICP-MS instrument standardized with the indicated standard solutions given in Table 2. Ge is used as the ISTD for the 11 elements not including Se. Those 11 elements are determined in the He collision mode, employing KED. Te shall be used as the ISTD for Se determinations. Hydrogen mode is recommended for the determination of low levels of Se in infant formula and, depending on the instrument model, it may not be possible to easily switch between helium and hydrogen mode. In such a case, follow the instructions of the instrument manufacturer for changing from helium to hydrogen mode and analyse Se separately from the other elements. Alternatively, verify in separate experiments that the practical limit of quantification (PLOQ) for Se is at or below 10 ng/g in the sample when using an alternate collision/reaction gas. One laboratory successfully completed the reproducibility study using helium, and another with ammonia gas[2].

Typical calibration correlation coefficients are 0,999 5 or better for all analytes, but suitability is determined by calibration residuals as follows. Analyse Cal Std 3, or other suitable quality control solution, as a sample every 10 test portions to monitor for instrument drift and linearity. The result shall be within 4 % of the standard's nominal concentration. For good performance, include a method blank (run as a sample, its measured concentration shall be less than half of the lowest calibration standard) and known reference materials serving as control samples (recovery check within control or certified limits). Duplicate samples as required in the first multi-lab testing (MLT) study[2] are now optional, because they did not generally improve reproducibility[1]. If used, the mean result is reported and appropriate criteria based upon the data would be a relative percent difference within 10 % for Cr, 7 % for Se, and 5 % for all other elements. If any of these quality control checks fails, results should be considered invalid.

The order of analysis should be calibration standards, followed by rinse, blank check, check standard, control sample, sample, sample duplicate (if used), and a repeated check standard.

Table 4 — Typical ICP-MS parameters for Agilent 7700x[a3)]

Radio frequency (RF) power, W	1 600
RF matching, V	1,8
Sample depth, mm	9
Extract 1 lens, V	0
Carrier gas, l/min	0,9
Make-up gas, l/min	0,2
Nebulizer (glass concentric)	MicroMist
Spray chamber temperature, °C	2
Interface cones	Ni
He cell gas flow rate, ml/min	4,5
H_2 cell gas flow rate, ml/min	4,2
Nebulizer pump rate, rps	0,1 (0,5 ml/min)
Peristaltic pump tubing	White/white, 1,02 mm internal diameter (i.d.)
Drain tubing	Blue/yellow, 1,52 mm i.d.

a The isotopes used for analysis are ^{23}Na, ^{24}Mg, ^{31}P, ^{39}K, ^{44}Ca, ^{52}Cr, ^{55}Mn, ^{56}Fe, ^{63}Cu, ^{66}Zn, ^{78}Se, and ^{95}Mo, with ^{72}Ge and ^{130}Te as internal standards.

10 Calculation

Calculate sample concentrations automatically by the software using a non-weighted least-squares linear regression calibration analysis to produce a best-fit line, using Formula (1):

$$y = a \times x + \text{blank} \tag{1}$$

Note that the sample blank is identical to the Cal Blk for this method and is essentially zero because high purity reagents are used.

Calculate the analyte mass fraction in the sample, w, in ng/g, using Formula (2):

$$w = \frac{(y - x)}{a} \times \frac{V_s}{m_s} \tag{2}$$

where

y is the analyte to ISTD intensity ratio, which is the measured count of each analyte's standard solution data point in the calibration curve divided by the counts of the ISTD at the same level;

x is the analyte to ISTD intensity ratio, which is the measured count of the blank standard solution data point in the calibration curve divided by the counts of the ISTD at the same level as the blank standard solution;

a is the slope of the calibration curve, in ml/ng;

V_S is the volume of the sample solution, in ml;

m_S is the sample mass.

3) Agilent 7700x is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or IDF of the product named.

11 Precision data

11.1 General

Details of the interlaboratory test of the precision of the method are summarized in Annex A. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and/or matrices other than those given in Annex A.

11.2 Repeatability

Method results are expected to have a coefficient of variation of repeatability, $C_{V,r}$, % of 5 % or lower as prescribed in the standard method performance requirements (SMPR). See Table A.1 for overall repeatability and Annex A for detailed performance.

11.3 Reproducibility

Method results are expected to have a coefficient of variation of reproducibility, $C_{V,R}$, %, of 10 % or lower as prescribed in the SMPR (≤15 % for Cr, Mo, Se and ≤16 % for Mn and Cu). See Table A.1 for overall reproducibility and Annex A for detailed performance.

12 Test report

The test report shall contain the following data:

a) all information necessary for the identification of the sample (type of sample, origin and designation of the sample);

b) a reference to this document, i.e. ISO 21424 | IDF 243;

c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of test;

f) the test results and the units in which they have been expressed;

g) any operations not specified in the method or regarded as optional, which might have affected the results.

Annex A
(informative)

Precision data

A.1 General

The method in this document is very well characterized. The single laboratory validation (SLV) data are given in Reference [6]. Two independent interlaboratory studies were also carried out, see References [1] and [2], to measure the reproducibility of the method, and a special study focused on the performance of the method at very low levels between the lowest calibration standard and the actual limit of quantification (LOQ) as measured from digested and undigested blanks[5]. Accuracy of the ICP-MS results was confirmed by comparing the mean MLT results from this method to those from ISO 15151 | IDF 229[11], which employed ICP-AES testing on the same sample set[1].

Based upon all these data, Table A.1 summarizes the latest figures of merit for the method in this document. The last row gives the recommended analytical ranges of this method to fully meet the SMPR requirements. The minimum limits for Mn, Cu, and Fe are slightly above the SMPR criteria, which were very aggressive for these analytes (well into the inherent level seen in the kinds of products that apply to this method). Note that the method can be used for analyte concentrations in the sample below the PLOQs, down to the LOQ limits given in Table A.1. This would allow measurement at the Codex Alimentarius minimum limits for Mn and Cr, for example, but the expected repeatability would be above 5 % RSD, and the expected reproducibility would be above 15 % RSD, with nonlinearity of the calibration curves expected to produce a bias of ≥ 10 % in the result.

Table A.1 — Overall performance of the method in interlaboratory testing

	Na	Mg	P	K	Ca	Mn	Fe	Cu	Zn	Cr	Se	Mo
Low standard (µg/l)	500	200	500	1 000	1 000	5,00	50,0	5,00	20,0	0,800	0,400	0,800
PLOQ[a] (µg/l)	50	20	100	100	100	2,5	5,0	0,50	4,0	0,080	0,20	0,080
PLOQ[b] (mg/100 g)	0,25	0,10	0,50	0,50	0,50	0,012	0,025	0,002 5	0,020	0,000 40	0,001 0	0,000 40
PLOQ meets SMPR?[c]	YES	YES	YES	YES	YES	NO	NO	NO	YES	YES	YES	YES
PLOQ meets CODEX?[d]	YES	YES	YES	YES	YES	NO	YES	YES	YES	YES	NO	YES
LOQ[e] (mg/100 g)	0,11	0,004 9	0,13	0,56	0,53	0,000 50	0,007 3	0,000 48	0,056	0,000 73	0,000 31	0,000 48
LOQ meets SMPR?[c]	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES
LOQ meets CODEX?[d]	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES

[a] Based upon work in Reference [5]. The practical limit of quantitation (PLOQ) is the lowest level at which the measured 3-day mean of a standard concentration, run as an unknown against the calibration curve, is within 5 % of its nominal concentration. This is the preferred lower limit if the method, as it limits the amount of calibration bias in the result. However, the sensitivity of ICP-MS typically affords a lower actual LOQ, as measured in the traditional way of measuring blank concentrations over several days;

[b] PLOQ converted to a product concentration level, using the typical dilution factor of 1 g to 50 ml for a ready-to-feed (RTF);

[c] The lower limit of the analytical range as set forth in the Standard Method Performance Requirements given in References [7] and [8];

[d] CODEX STAN 72 -1981 minimum levels for infant formulas and FSMP products;

[e] LOQs from Reference [5] These are the average of digested and undigested blanks, each run on five separate days. Both sets of blanks yielded similar results;

[f] For 18 samples tested at 10 laboratories in the interlaboratory study[1], not counting the adult high fat RTF sample or the samples in which the analyte was at or below the QL (as opposed to < PLOQ);

[g] To meet SMPR requirements. These are extrapolated from the interlaboratory study[1], relying on reproducibility data for usually 1 to 2 samples that are no more than a factor of three in concentration from the indicated lower limit;

[h] Mn and Se meet repeatability and reproducibility criteria at this level but, unlike copper, there is some additional calibration bias that might affect the accuracy of the result below the PLOQ;

[i] Although Cr has an adequate PLOQ and LOQ when measured under SLV conditions, the SMPR criteria for reproducibility (< 15 % RSD) are not consistently met below the SMPR lower limit of 0,002 mg/100 g (20 ng/g). Slight contamination of the samples is a likely possibility. If only a single analysis is performed, testing Cr below 20 ng/g is not recommended.

Table A.1 *(continued)*

	Na	Mg	P	K	Ca	Mn	Fe	Cu	Zn	Cr	Se	Mo
Average % repeatability[f]	1,5	1,5	1,9	1,4	1,8	2,1	2,9	2,3	1,6	3,0	3,3	2,1
Average % Reproducibility[f]	3,6	3,5	4,3	3,6	4,1	3,8	6,1	6,3	4,3	4,9	5,0	3,6
Recommended analytical range[g] (mg/100 g)	2 to 850	0,7 to 110	3 to 800	3 to 2 000	3 to 1 280	0,002[h] to 1,0	0,04 to 20	0,002 to 1,2	0,07 to 18	0,002[i] to 0,16	0,000 6[h] to 0,050	0,000 2 to 0,10

[a] Based upon work in Reference [5]. The practical limit of quantitation (PLOQ) is the lowest level at which the measured 3-day mean of a standard concentration, run as an unknown against the calibration curve, is within 5 % of its nominal concentration. This is the preferred lower limit if the method, as it limits the amount of calibration bias in the result. However, the sensitivity of ICP-MS typically affords a lower actual LOQ, as measured in the traditional way of measuring blank concentrations over several days;

[b] PLOQ converted to a product concentration level, using the typical dilution factor of 1 g to 50 ml for a ready-to-feed (RTF);

[c] The lower limit of the analytical range as set forth in the Standard Method Performance Requirements given in References [7] and [8];

[d] CODEX STAN 72 -1981 minimum levels for infant formulas and FSMP products;

[e] LOQs from Reference [5] These are the average of digested and undigested blanks, each run on five separate days. Both sets of blanks yielded similar results;

[f] For 18 samples tested at 10 laboratories in the interlaboratory study[1], not counting the adult high fat RTF sample or the samples in which the analyte was at or below the QL (as opposed to < PLOQ);

[g] To meet SMPR requirements. These are extrapolated from the interlaboratory study[1], relying on reproducibility data for usually 1 to 2 samples that are no more than a factor of three in concentration from the indicated lower limit;

[h] Mn and Se meet repeatability and reproducibility criteria at this level but, unlike copper, there is some additional calibration bias that might affect the accuracy of the result below the PLOQ;

[i] Although Cr has an adequate PLOQ and LOQ when measured under SLV conditions, the SMPR criteria for reproducibility (< 15 % RSD) are not consistently met below the SMPR lower limit of 0,002 mg/100 g (20 ng/g). Slight contamination of the samples is a likely possibility. If only a single analysis is performed, testing Cr below 20 ng/g is not recommended.

A.2 Precision data for milk, milk products. infant formula and adult nutritionals

The data given in Tables A.2 to A.23 were obtained in an interlaboratory study[1], in accordance with ISO 5725-2[9] and AOAC-IUPAC Harmonized Protocol for collaborative study procedures, to access precision characteristics of a method of analysis[10]. The study was performed based on requirements given in Reference [10]. There is no table for Cr because all samples were below the LOQ.

Table A.2 — Precision data for calcium in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10
Number of outliers (laboratories)	0	0	0	0	0	0
Number of laboratories retained after eliminating outliers	10	10	10	10	10	10
Number of accepted results	20	20	20	20	20	20
Mean value, mg/100 g	19,1[g]	498	670	115	106	689
Repeatability standard deviation s_r, mg/100 g	0,80	7,1	10	1,6	1,6	10
Reproducibility standard deviation s_R, mg/100 g	1,4	26	120	4,8	4,3	40
Coefficient of variation of repeatability, $C_{V,r}$, %	4,2	1,4	1,5	1,4	1,5	1,5
Coefficient of variation of reproducibility, $C_{V,R}$, %	7,4	5,2	18[h]	4,2	4,1	5,8
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	2,2	20	29	4,6	4,5	29
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	4,0	73	336	13	12	112
HorRat value	1,0	1,2	4,2	0,75	0,73	1,4

[a] Butter, [b] Cheese, [c] Whey, [d] Whole milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.), except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.3 — Precision data for copper in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	9	10	9	9	10
Number of outliers (laboratories)	0	3	0	4	4	0
Number of laboratories retained after eliminating outliers	10	6	10	5	5	10
Number of accepted results	20	12	20	10	10	20
Mean value, mg/100 g	0,004 2[g]	0,049 8	0,034 5	0,005 2	0,004 0	0,117 2
Repeatability standard deviation s_r, mg/100 g	0,001 5	0,001 4	0,002 2	0,000 050	0,000 19	0,001 6

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.3 *(continued)*

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Reproducibility standard deviation s_R, mg/100 g	0,004 2	0,003 0	0,009 9	0,000 29	0,000 30	0,004 9
Coefficient of variation of repeatability, $C_{V,r}$, %	36[g,h]	2,9	6,3[h]	0,97	4,7	1,4
Coefficient of variation of reproducibility, $C_{V,R}$, %	99[g,h]	5,9	29[h]	5,5	7,5	4,2
Repeatability limit r [$r = 2,8 \times s_r$], mg/100 g	0,004 2	0,004 0	0,006 1	0,000 14	0,000 52	0,004 59
Reproducibility limit R [$R = 2,8 \times s_R$], mg/100 g	0,012	0,008 3	0,028	0,000 80	0,000 84	0,014
HorRat value	3,9[g]	0,33	1,5	0,22	0,29	0,27

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.4 — Precision data for iron in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	9	10	10	9	9
Number of outliers (laboratories)	2	4	1	2	5	2
Number of laboratories retained after eliminating outliers	8	5	9	8	4	7
Number of accepted results	16	10	18	16	8	14
Mean value, mg/100 g	0,027 9[g]	0,183	0,300	0,017 1[g]	0,027 4	0,656
Repeatability standard deviation s_r, mg/100 g	0,005 2	0,008 3	0,015	0,000 63	0,000 38	0,009 0
Reproducibility standard deviation s_R, mg/100 g	0,011	0,013	0,082	0,006 4	0,001 5	0,017
Coefficient of variation of repeatability, $C_{V,r}$, %	19[g,h]	4,6	5,1[h]	3,7	1,4	1,4
Coefficient of variation of reproducibility, $C_{V,R}$, %	40[g,h]	7,1	27[h]	38[g,h]	5,5	2,6
Repeatability limit r [$r = 2,8 \times s_r$], mg/100 g	0,015	0,023	0,043	0,001 8	0,001 1	0,025
Reproducibility limit R [$R = 2,8 \times s_R$], mg/100 g	0,031	0,036	0,23	0,018	0,004 2	0,048
HorRat value	2,1	0,49	2,0	1,8	0,28	0,22

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.5 — Precision data for potassium in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10
Number of outliers (laboratories)	0	0	0	0	0	0
Number of laboratories retained after eliminating outliers	10	10	10	10	10	10
Number of accepted results	20	20	20	20	20	20
Mean value, mg/100 g	35,7[g]	97,7	2 004	163	135	831
Repeatability standard deviation s_r, mg/100 g	2,1	2,5	60,1	4,0	3,6	19,5
Reproducibility standard deviation s_R, mg/100 g	2,8	3,8	281	8,5	9,1	59
Coefficient of variation of repeatability, $C_{V,r}$, %	5,8[h]	2,6	3,0	2,4	2,7	2,4
Coefficient of variation of reproducibility, $C_{V,R}$, %	7,9	3,9	14[h]	5,2	6,8	7,1
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	5,8	7,0	168	11	10	55
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	7,9	11	786	24	26	165
HorRat value	1,2	0,68	3,9	1,0	1,3	1,7

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.6 — Precision data for magnesium in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	9	8	10	9	10
Number of outliers (laboratories)	1	2	0	0	2	0
Number of laboratories retained after eliminating outliers	9	7	8	10	7	10
Number of accepted results	18	14	16	20	14	20
Mean value, mg/100 g	2,24[g]	20,4	203	10,7	9,44	75,3
Repeatability standard deviation s_r, mg/100 g	0,1	0,4	3,0	0,1	0,1	0,9
Reproducibility standard deviation s_R, mg/100 g	0,14	0,74	13	0,54	0,26	4,4
Coefficient of variation of repeatability, $C_{V,r}$, %	3,4	1,8	1,5	1,0	1,3	1,1
Coefficient of variation of reproducibility, $C_{V,R}$, %	6,2	3,6	6,5	5,0	2,8	5,9
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	0,21	1,0	8,5	0,30	0,34	2,4
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	0,39	2,1	37	1,5	0,7	12
HorRat value	0,62	0,51	1,3	0,63	0,34	1,0

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.7 — Precision data for manganese in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	9	10	8	10	10	9
Number of outliers (laboratories)	2	0	4	1	1	2
Number of laboratories retained after eliminating outliers	7	10	4	9	9	7
Number of accepted results	14	20	8	18	18	14
Mean value, mg/100 g	0,000 828[g]	0,014 3	0,006 64[g]	0,002 28	0,002 18	0,008 03
Repeatability standard deviation s_r, mg/100 g	0,000 37	0,000 44	0,000 48	0,000 082	0,000 043	0,000 35
Reproducibility standard deviation s_R, mg/100 g	0,000 52	0,000 68	0,000 69	0,000 15	0,000 11	0,000 42
Coefficient of variation of repeatability, $C_{V,r}$, %	44,8[g,h]	3,1	7,3[g,h]	3,6	2,0	4,3
Coefficient of variation of reproducibility, $C_{V,R}$, %	63,3[g,h]	4,8	10,4	6,4	5,2	5,2
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	0,001 0	0,001 2	0,001 4	0,000 23	0,000 12	0,001 0
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	0,001 5	0,001 9	0,001 9	0,000 41	0,000 32	0,001 2
HorRat value	1,9	0,22	0,43	0,23	0,18	0,22

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.8 — Precision data for molybdenum in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	8	8	7	8	8	8
Number of outliers (laboratories)	2	0	2	0	0	0
Number of laboratories retained after eliminating outliers	6	8	5	8	8	8
Number of accepted results	12	16	10	16	16	16
Mean value, mg/100 g	0,003 3[g]	0,011 5	0,081 3	0,003 7	0,003 4	0,162
Repeatability standard deviation s_r, mg/100 g	0,000 19	0,000 15	0,001 1	0,000 057	0,000 034	0,002 5
Reproducibility standard deviation s_R, mg/100 g	0,000 19	0,000 40	0,003 0	0,000 081	0,000 11	0,004 3
Coefficient of variation of repeatability, $C_{V,r}$, %	5,7[h]	1,3	1,3	1,5	1,0	1,6

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE 1 These milk and milk products were not studied in ISO 20649 | IDF 235, although there were other collaborative data for molybdenum by the same method.

NOTE 2 Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.8 *(continued)*

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Coefficient of variation of reproducibility, $C_{V,R}$, %	5,7	3,5	3,6	2,2	3,4	2,7
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	0,000 53	0,000 43	0,003 1	0,000 16	0,000 094	0,007 0
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	0,000 53	0,001 1	0,008 3	0,000 23	0,000 32	0,012
HorRat value	0,21	0,16	0,22	0,080	0,13	0,18

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE 1 These milk and milk products were not studied in ISO 20649 | IDF 235, although there were other collaborative data for molybdenum by the same method.

NOTE 2 Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.9 — Precision data for sodium in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	9	10
Number of outliers (laboratories)	0	0	0	0	2	0
Number of laboratories retained after eliminating outliers	10	10	10	10	7	10
Number of accepted results	20	20	20	20	14	20
Mean value, mg/100 g	243	1 014	868	37,5	33,6	265
Repeatability standard deviation s_r, mg/100 g	7,9	17,8	11,5	0,4	0,6	3,4
Reproducibility standard deviation s_R, mg/100 g	13	79	156	1,7	1,1	17
Coefficient of variation of repeatability, $C_{V,r}$, %	3,2	1,8	1,3	0,9	1,8	1,3
Coefficient of variation of reproducibility, $C_{V,R}$, %	5,2	7,8	18[g]	4,6	3,2	6,5
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	22	50	32	1,0	1,7	9,4
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	36	222	437	4,9	3,0	48
HorRat value	1,1	2,0	4,4	0,70	0,48	1,3

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.10 — Precision data for phosphorous in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	8	9	10	10	10
Number of outliers (laboratories)	0	4	2	0	0	0
Number of laboratories retained after eliminating outliers	10	4	7	10	10	10
Number of accepted results	20	8	14	20	20	20
Mean value, mg/100 g	26,1[g]	867	983	95,0	87,5	516
Repeatability standard deviation s_r, mg/100 g	1,2	10	14	1,5	1,7	14
Reproducibility standard deviation s_R, mg/100 g	1,5	50	181	3,7	4,8	30
Coefficient of variation of repeatability, $C_{V,r}$, %	4,5	1,2	1,5	1,6	2,0	2,7
Coefficient of variation of reproducibility, $C_{V,R}$, %	5,9	5,8	18,4[h]	3,9	5,5	5,9
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	3,3	28	40	4	4,8	39
Reproducibility limit R [$R = 2{,}8 \times s_R$], mg/100 g	4,3	141	506	10	14	85
HorRat value	0,85	1,4	4,6	0,69	0,96	1,3

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.11 — Precision data for selenium in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	8	7	7	8	8	8
Number of outliers (laboratories)	0	3	2	0	1	0
Number of laboratories retained after eliminating outliers	8	4	5	8	7	8
Number of accepted results	16	8	10	16	14	16
Mean value, mg/100 g	0,000 7[g]	0,005 5	0,015	0,001 8	0,001 3	0,030 5
Repeatability standard deviation s_r, mg/100 g	0,000 10	0,000 22	0,001 1	0,000 11	0,000 041	0,002 0
Reproducibility standard deviation s_R, mg/100 g	0,000 22	0,000 36	0,001 4	0,000 14	0,000 071	0,002 0
Coefficient of variation of repeatability, $C_{V,r}$, %	14,1[g,h]	4,0	7,0[h]	6,1[h]	3,2	6,4

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE 1 These milk and milk products were not studied in ISO 20649 | IDF 235, although there were other collaborative data for selenium by the same method.

NOTE 2 Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.11 *(continued)*

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Coefficient of variation of reproducibility, $C_{V,R}$, %	31,3[g,h]	6,5	9,6	7,8	5,4	6,4
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	0,000 28	0,000 61	0,002 9	0,000 31	0,000 12	0,005 5
Reproducibility limit R [$R = 2{,}8 \times s_R$], ng/g	0,001	0,001 0	0,004 0	0,000 39	0,000 20	0,005 5
HorRat value	0,92	0,26	0,45	0,27	0,18	0,34

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE 1 These milk and milk products were not studied in ISO 20649 | IDF 235, although there were other collaborative data for selenium by the same method.

NOTE 2 Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.12 — Precision data for zinc in milk and milk products

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	9	10	10	10
Number of outliers (laboratories)	0	0	2	0	0	1
Number of laboratories retained after eliminating outliers	10	10	7	10	10	9
Number of accepted results	20	20	14	20	20	18
Mean value, mg/100 g	0,084 4[g]	2,46	0,182[g]	0,388	0,356	0,871
Repeatability standard deviation s_r, mg/100 g	0,028	0,029	0,006 2	0,004 2	0,004 2	0,014
Reproducibility standard deviation s_R, mg/100 g	0,042	0,105	0,043	0,010	0,017	0,032
Coefficient of variation of repeatability, $C_{V,r}$, %	33[g,h]	1,2	3,4	1,1	1,2	1,7
Coefficient of variation of reproducibility, $C_{V,R}$, %	50[g,h]	4,3	24[g,h]	2,6	4,9	3,7
Repeatability limit r [$r = 2{,}8 \times s_r$], mg/100 g	0,078 0	0,082	0,017	0,012	0,012	0,040
Reproducibility limit R [$R = 2{,}8 \times s_R$], ng/g	0,118	0,29	0,12	0,029	0,048	0,090
HorRat value	3,0	0,43	1,6	0,20	0,37	0,32

[a] Butter, [b] Cheese, [c] Whey, [d] Whole Milk, [e] Milk PWD, [f] WPC, [g] Below (equivalent, as-is) SMPR limit and below LOQ, [h] Failed SMPR reproducibility or repeatability criteria.

NOTE Results are on an as-is basis for all matrices (e.g. mg/100 g of butter, cheese, etc.) except for milk powder, for which results are in mg per 100 g of the reconstituted powder (25 g in 200 g water).

Table A.13 — Precision data for calcium in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	9	10	10
Number of outliers (individual replicates)	1	0	0	1	0	0	0	4	0	0	0	0	0	0	0	2	0	0
Number of accepted results	19	20	20	19	20	20	20	16	20	20	20	20	20	20	20	16	20	20
Mean value, mg/100 g	34,2	53,9	14,4	96,5	7,45	34,2	48,2	43,3	67,9	61,9	45,5	69,2	36,8	57,7	73,8	52,6	29,6	65,5
Repeatability standard deviation s_r, mg/100 g	0,43	4,15	0,39	2,13	0,12	0,40	0,64	0,56	0,99	0,84	0,78	0,76	0,44	1,47	1,26	3,02	0,37	1,14
Reproducibility standard deviation s_R, mg/100 g	1,4	11,4	0,6	4,5	0,4	1,6	1,5	1,7	2,6	1,9	1,9	2,1	1,3	2,7	3,0	3,0	1,2	2,2
Coefficient of variation of repeatability, $C_{V,r}$, %	1,3	7,7	2,7	2,2	1,6	1,2	1,3	1,3	1,5	1,4	1,7	1,1	1,2	2,6	1,7	5,7	1,3	1,7
Coefficient of variation of reproducibility, $C_{V,R}$, %	4,1	21,2	4,4	4,7	5,2	4,8	3,1	4,0	3,8	3,1	4,2	3,0	3,4	4,7	4,0	5,7	4,1	3,4
Repeatability limit r, [$r = 2{,}8 \times s_r$]	1,21	11,62	1,10	5,97	0,33	1,13	1,78	1,58	2,78	2,36	2,18	2,13	1,24	4,12	3,53	8,45	1,04	3,19
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	3,94	31,99	1,76	12,59	1,08	4,61	4,18	4,87	7,15	5,33	5,28	5,81	3,52	7,54	8,31	8,45	3,39	6,27
HorRat value	0,62	3,42	0,58	0,79	0,62	0,72	0,49	0,61	0,63	0,51	0,65	0,5	0,52	0,76	0,68	0,92	0,60	0,57
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder with FOS (fructo-oligosaccharides) and GOS (galacto-oligosaccharides), [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).

Table A.14 — Precision data for copper in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	9	9	9	8	10	10	10	10	10	10	10	10	10	10
Number of outliers (individual replicates)	0	0	1	0	2	2	2	4	0	0	0	0	0	0	0	1	2	1
Number of accepted results	20	20	19	20	16	16	16	12	20	20	20	20	20	20	20	19	18	19
Mean value, mg/100 g	0,069 6	0,233	0,008 7	0,181 4	0,010 1	0,006 6	0,108 2	0,044 3	0,082 1	0,001 4	0,056 4	0,060 9	0,047 1	0,060 0	0,065 8	0,046 5	0,002 5	0,027 8
Repeatability standard deviation s_r, mg/100 g	0,00	0,01	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
Reproducibility standard deviation s_R, mg/100 g	0,00	0,01	0,00	0,01	0,00	0,00	0,01	0,00	0,01	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
Coefficient of variation of repeatability, $C_{V,r}$, %	1,6	2,4	5,4	1,6	2,2	3,9	0,8	1,2	1,7	11	1,1	1,1	2,3	0,9	1,0	1,2	9,2	0,9
Coefficient of variation of reproducibility, $C_{V,R}$, %	4,9	4,6	12	4,1	4,4	7,1	5,2	5,7	6,8	133	7,0	5,1	5,9	4,6	5,2	3,7	16	4,4
Repeatability limit r, [$r = 2{,}8 \times s_r$]	0,003	0,015	0,001	0,008	0,001	0,001	0,002	0,002	0,004	0,000	0,002	0,002	0,003	0,002	0,002	0,002	0,001	0,001
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	0,010	0,030	0,003	0,021	0,001	0,001	0,016	0,007	0,016	0,005	0,011	0,009	0,008	0,008	0,010	0,005	0,001	0,003
HorRat value	0,29	0,33	0,51	0,28	0,19	0,30	0,33	0,32	0,41	4,4	0,40	0,30	0,33	0,27	0,31	0,21	0,56	0,23
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder FOS-GOS, [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).

Table A.15 — Precision data for iron in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	9	10	10	10	10	8	9	8	10	9	10	9	10	10	10	10	9	10
Number of outliers (individual replicates)	2	0	1	0	0	5	2	4	0	3	0	2	2	0	0	0	3	0
Number of accepted results	16	20	19	20	20	11	16	12	20	15	20	16	18	20	20	20	15	30
Mean value, mg/100 g	0,650	2,34	0,384	2,03	0,430	0,046 5	1,21	0,720	1,18	0,028 2	1,02	1,27	0,553	1,27	1,28	1,37	0,017 3	0,826
Repeatability standard deviation s_r, mg/100 g	0,016	0,142	0,039	0,087	0,043	0,001	0,013	0,008	0,015	0,001	0,031	0,012	0,005	0,015	0,010	0,019	0,001	0,006
Reproducibility standard deviation s_R, mg/100 g	0,002	0,168	0,059	0,112	0,053	0,004	0,032	0,046	0,061	0,002	0,055	0,035	0,031	0,054	0,070	0,046	0,004	0,045
Coefficient of variation of repeatability, $C_{V,r}$, %	2,5	6,1	10,2	4,3	10,1	2,9	1,1	1,2	1,3	3,2	3,1	1,0	0,9	1,2	0,8	1,4	8,6	0,8
Coefficient of variation of reproducibility, $C_{V,R}$, %	3,5	7,2	15,3	5,5	12,3	7,6	2,7	6,4	5,2	7,6	5,4	2,8	5,5	4,3	5,4	3,4	26,0	5,4
Repeatability limit r, [$r = 2{,}8 \times s_r$]	0,046	0,397	0,109	0,244	0,121	0,004	0,037	0,024	0,042	0,003	0,088	0,035	0,014	0,041	0,028	0,053	0,004	0,018
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	0,063	0,470	0,164	0,314	0,148	0,010	0,090	0,129	0,170	0,006	0,154	0,099	0,086	0,153	0,195	0,130	0,013	0,125
HorRat value	0,29	0,72	1,17	0,54	0,96	0,42	0,24	0,54	0,47	0,39	0,48	0,25	0,45	0,39	0,50	0,31	1,25	0,46
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder FOS-GOS, [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).

Table A.16 — Precision data for potassium in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10	10	8	10	10	10	10	10	10	10	10	10	10
Number of outliers (individual replicates)	0	0	0	0	0	0	0	4	0	0	0	0	0	0	0	0	0	0
Number of accepted results	20	20	20	20	20	20	20	12	20	20	20	20	20	20	20	20	20	20
Mean value, mg/100 g	44,7	220	178	151	98,9	7,90	70,9	56,4	87,4	86,1	68,1	80,0	49,5	77,8	84,1	80,8	62,2	138,1
Repeatability standard deviation s_r, mg/100 g	0,53	3,1	3,3	1,8	19	0,13	0,5	1,2	1,0	1,3	1,1	1,2	0,89	1,3	1,0	0,87	0,75	1,5
Reproducibility standard deviation s_R, mg/100 g	1,7	11,5	5,1	7,5	4,2	0,5	1,9	1,9	2,4	3,2	3,0	1,7	1,8	2,8	1,2	3,0	2,6	4,2
Coefficient of variation of repeatability, $C_{V,r}$, %	1,2	1,4	1,9	1,2	2,0	1,6	0,7	2,1	1,2	1,5	1,7	1,5	1,8	1,7	1,2	1,1	1,2	1,1
Coefficient of variation of reproducibility, $C_{V,R}$, %	3,8	5,2	2,9	4,9	4,2	6,0	2,8	3,4	2,8	3,7	4,4	2,1	3,6	3,6	2,5	3,7	4,1	3,0
Repeatability limit r, [$r = 2{,}8 \times s_r$]	1,5	8,7	9,3	4,9	5,5	0,4	1,3	3,2	2,9	3,7	3,2	3,4	2,5	3,7	2,8	2,4	2,1	4,3
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	4,8	32,1	14,2	20,9	11,6	1,3	5,5	5,4	6,8	8,8	8,4	4,7	5,0	7,8	5,8	8,3	7,2	11,6
HorRat value	0,60	1,04	0,55	0,93	0,74	0,72	0,46	0,56	0,48	0,63	0,74	0,36	0,58	0,61	0,42	0,63	0,68	0,58
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder FOS-GOS, [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).

Table A.17 — Precision data for magnesium in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
Number of outliers (individual replicates)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Number of accepted results	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20
Mean value, mg/100 g	13,2	38,5	2,06	32,8	1,77	1,96	9,45	5,72	5,40	5,22	4,48	6,72	4,78	5,23	8,25	6,33	3,52	6,17
Repeatability standard deviation s_r, mg/100 g	0,23	0,82	0,06	0,33	0,02	0,04	0,11	0,07	0,09	0,08	0,07	0,08	0,07	0,12	0,09	0,07	0,04	0,07
Reproducibility standard deviation s_R, mg/100 g	0,39	1,75	0,12	1,32	0,08	0,09	0,23	0,25	0,13	0,20	0,21	0,14	0,13	0,14	0,20	0,28	0,15	0,12
Coefficient of variation of repeatability, $C_{V,r}$, %	1,8	2,1	2,8	1,0	1,2	1,8	1,2	1,2	1,6	1,6	1,6	1,2	1,5	2,2	1,1	1,1	1,2	1,1
Coefficient of variation of reproducibility, $C_{V,R}$, %	2,9	4,6	5,8	4,0	4,5	4,6	2,4	4,4	2,5	3,9	4,7	2,0	2,8	2,7	2,4	4,4	4,2	1,9
Repeatability limit r, [$r = 2{,}8 \times s_r$]	0,65	2,31	0,16	0,94	0,06	0,10	0,31	0,19	0,24	0,24	0,20	0,23	0,20	0,32	0,25	0,19	0,12	0,18
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	1,08	4,90	0,33	3,69	0,22	0,25	0,64	0,70	0,37	0,57	0,59	0,38	0,37	0,40	0,56	0,77	0,41	0,33
HorRat value	0,38	0,70	0,57	0,60	0,43	0,45	0,30	0,50	0,28	0,44	0,52	0,24	0,31	0,31	0,30	0,51	0,45	0,22
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder FOS-GOS, [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).

Table A.18 — Precision data for manganese in infant formula and adult nutritionals

Sample	1[a]	2[b]	3[c]	4[d]	5[e]	6[f]	7[g]	8[h]	9[i]	10[j]	11[k]	12[l]	13[m]	14[n]	15[o]	16[p]	17[q]	18[r]
Year of interlaboratory test	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016	2016
Number of laboratories submitting results	10	10	10	10	10	10	10	8	10	10	10	10	10	10	10	10	10	10
Number of outliers (individual replicates)	0	0	0	1	0	1	0	4	0	0	0	0	0	0	0	0	3	2
Number of accepted results	20	20	20	19	20	19	20	12	20	20	20	20	20	20	20	20	17	18
Mean value, mg/100 g	0,158	0,349	0,008 27	0,411	0,006 27	0,003 72	0,165	0,009 19	0,051 7	0,003 81	0,013 2	0,024 1	0,012 5	0,006 12	0,034 9	0,011 8	0,000 857	0,085 8
Repeatability standard deviation s_r, mg/100 g	0,004 9	0,019 5	0,000 4	0,000 6	0,000 2	0,000 1	0,001 5	0,000 1	0,000 7	0,000 1	0,000 2	0,000 4	0,000 3	0,000 1	0,000 4	0,000 2	0,000 1	0,000 6
Reproducibility standard deviation s_R, mg/100 g	0,005 8	0,048 5	0,000 5	0,017 5	0,000 3	0,000 2	0,006 4	0,000 3	0,001 3	0,000 1	0,000 4	0,000 6	0,000 4	0,000 3	0,000 9	0,000 5	0,000 2	0,001 6
Coefficient of variation of repeatability, $C_{V,r}$, %	3,1	5,6	5,3	2,1	2,7	3,0	0,9	1,3	1,4	2,1	1,7	1,9	2,1	2,4	1,2	2,1	10,9	0,7
Coefficient of variation of reproducibility, $C_{V,R}$, %	3,7	13,9	6,2	4,3	4,2	6,0	3,9	3,3	2,5	3,7	2,8	2,4	3,5	5,4	2,5	4,0	19,1	1,8
Repeatability limit r, [$r = 2{,}8 \times s_r$]	0,013 8	0,054 7	0,001 2	0,024 2	0,000 5	0,000 3	0,004 1	0,000 3	0,002 1	0,000 2	0,000 6	0,001 3	0,000 7	0,000 4	0,001 1	0,000 7	0,000 3	0,001 7
Reproducibility limit R, [$R = 2{,}8 \times s_R$]	0,016 1	0,135 8	0,001 4	0,049 0	0,000 7	0,000 6	0,017 8	0,000 8	0,003 6	0,000 4	0,001 0	0,001 6	0,001 2	0,000 9	0,002 4	0,001 3	0,000 5	0,004 4
HorRat value	0,24	1,05	0,26	0,33	0,17	0,23	0,26	0,14	0,14	0,14	0,13	0,12	0,16	0,22	0,13	0,18	0,58	0,11
Repeatability limit SMPR[8]	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Reproducibility limit SMPR[8]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10

[a] Adult powder low fat, [b] Adult RTF high fat, [c] Adult RTF high fat placebo, [d] Adult RTF high protein, [e] Adult RTF high protein placebo, [f] Child milk powder placebo, [g] Child milk powder, [h] Illuma child powder, [i] Infant elemental powder, [j] Infant elemental powder placebo, [k] Infant PH powder milk, [l] Infant PH powder soy, [m] Infant powder FOS-GOS, [n] Infant powder milk, [o] Infant powder soy, [p] Infant RTF milk, [q] Infant RTF placebo, [r] Toddler powder.

NOTE The results are expressed as mg/100 g reconstituted final product ("ready-to-feed" liquids "as is"; reconstituted powders (25 g into 200 g of water).