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**ISO
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**IDF
20-3**

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Milk — Determination of nitrogen content —

Part 3:
Block-digestion method (Semi-micro rapid routine method)

Lait — Détermination de la teneur en azote —

Partie 3: Méthode de minéralisation en bloc (Méthode de routine semi-micro rapide)

Reference numbers
ISO 8968-3:2004(E)
IDF 20-3:2004(E)

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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.

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 8968-3|IDF 20-3 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.

ISO 8968|IDF 20 consists of the following parts, under the general title *Milk — Determination of nitrogen content*:

- *Part 1: Kjeldahl method*
- *Part 2: Block-digestion method (Macro method)*
- *Part 3: Block-digestion method (Semi-micro rapid routine method)*
- *Part 4: Determination of non-protein-nitrogen content*
- *Part 5: Determination of protein-nitrogen content*

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 and AOAC International 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 IDF National Committees casting a vote.

ISO 8968-3|IDF 20-3 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.

All work was carried out by the Joint ISO/IDF/AOAC Action Team, *Nitrogen compounds,* of the Standing Committee on *Main components of milk,* under the aegis of its project leader Mrs S. Berman (IL).

This edition of ISO 8968-3|IDF 20-3 cancels and replaces IDF 20-3:1993, which has been technically revised.

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— *Part 5: Determination of protein-nitrogen content*

INTERNATIONAL STANDARD

ISO 8968-3:2004(E)
IDF 20-3:2004(E)

Milk — Determination of nitrogen content —

Part 3:
Block-digestion method (Semi-micro rapid routine method)

WARNING — The use of this part of ISO 8968|IDF 20 may involve the use of hazardous materials, operations and equipment. This standard does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of local regulatory limitations prior to use.

1 Scope

This part of ISO 8968|IDF 20 specifies a method for the determination of the nitrogen content of liquid, whole or skimmed milk.

It concerns a semi-micro rapid routine method following the block-digestion principle.

NOTE The method is a more rapid method than that described in ISO 8968-1|IDF 20-1 and ISO 8968-2|IDF 20-2 since the digestion time is reduced by taking a lower mass of test portion and using hydrogen peroxide together with sulfuric acid and a catalyst in the digestion.

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 385:—[1], *Laboratory glassware — Burettes*

3 Terms and definitions

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

3.1
nitrogen content
mass fraction of nitrogen determined by the procedure specified in this part of ISO 8968|IDF 20.

NOTE The nitrogen content is expressed as a percentage by mass.

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[1] To be published. (Revision of ISO 385-1:1984, ISO 385-2:1984 and ISO 385-3:1984)

4 Principle

A test portion is digested by using a block-digestion apparatus with a mixture of concentrated sulfuric acid, hydrogen peroxide and potassium sulfate, together with a catalyst to convert the organic nitrogen present to ammonium sulfate. Excess sodium hydroxide is added to the cooled digest to liberate ammonia.

The liberated ammonia is distilled using a manual, semi-automatic or fully automatic steam distillation unit. In the case of manual or semi-automatic steam distillation, the ammonia is distilled into excess boric acid solution then titrated with hydrochloric acid solution. Where a fully automatic distillation unit is employed, titration of the ammonia is carried out automatically with endpoint detection using a photometric or pH system. The nitrogen content is calculated from the amount of ammonia produced.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water.

5.1 Kjeldahl catalyst tablets

These tablets may be purchased commercially. Tablets comprising 3,5 g of potassium sulfate, 0,105 g of copper(II) sulfate pentahydrate and 0,105 g of titanium dioxide per tablet are suitable.

Other types of tablet may be used provided that

a) they contain a quantity of potassium sulfate such that 7 g of potassium sulfate can be dispensed using an integer number of whole tablets, and

b) they contain no salts of toxic metals such as selenium or mercury.

5.2 Sulfuric acid (H_2SO_4), with mass fraction of at least 95 % to 98 %, nitrogen free [$\rho_{20}(H_2SO_4)$ = 1,84 g/ml approximately].

5.3 Hydrogen peroxide solution (H_2O_2), approximately 30 g of H_2O_2 per 100 ml.

5.4 Antifoaming agent

A silicone preparation is recommended, for example with a mass fraction of 30 % aqueous emulsion.

5.5 Sodium hydroxide solution (NaOH), nitrogen free, containing approximately 40 g of NaOH per 100 ml.

5.6 Boric acid solutions

5.6.1 Boric acid solution, $c(H_3BO_3)$ = 40,0 g/l.

Dissolve 40,0 g of boric acid in 1 litre of hot water in a 1 000 ml one-mark volumetric flask. Allow the flask and its contents to cool to 20 °C. Add 3 ml of indicator solution (5.7.1). Adjust to the mark with water and mix well.

Store the solution, which will be light orange in colour, in a borosilicate glass bottle. Protect the solution from light and sources of ammonia fumes during storage.

5.6.2 Boric acid solution, $c(H_3BO_3)$ = 10,0 g/l, to be used in the photometric endpoint titration.

Dissolve 10,0 g of boric acid in 1 litre of hot water in a 1 000 ml one-mark volumetric flask. Allow the flask and its contents to cool to 20 °C. Add 7 ml of methyl red solution (5.7.2) and 10 ml of bromocresol green solution (5.7.2) and mix.

Dilute to the 1 000 ml mark with water and mix well. Neutralize the boric acid solution with 0,1 mol/l sodium hydroxide until its colour changes to green.

NOTE The addition of 3 ml of 0,1 mol/l NaOH into 1 litre of 1 % boric acid usually gives good adjustments.

5.7 Indicator solutions

5.7.1 Indicator solution to be used in the pH endpoint titration

Dissolve 0,1 g of methyl red in 95 % (volume fraction) ethanol. Dilute to 50 ml with the ethanol. Dissolve 0,5 g of bromocresol green in a small quantity of 95 % (volume fraction) ethanol. Dilute to 250 ml with the ethanol.

Mix amounts of one part of methyl red solution with five parts of bromocresol green solution, or combine and mix both solutions.

5.7.2 Indicator solution to be used in the boric acid solution (5.6.2) for the photometric endpoint titration

a) Dissolve 0,1 g of bromocresol green in 100 ml of 95 % (volume fraction) ethanol.

b) Dissolve 0,1 g of methyl red in 100 ml of 95 % (volume fraction) ethanol.

5.8 Hydrochloric acid standard volumetric solution, c(HCl) = (0,1 ± 0,0005) mol/l.

If the titration (9.2.3) is carried out manually, the use of a more diluted hydrochloric acid standard volumetric solution, c(HCl) = (0,05 ± 0,0005) mol/l, is recommended.

5.9 Ammonium sulfate [$(NH_4)_2SO_4$], minimum assay 99,9 % (mass fraction) on dried material.

Immediately before use, dry the ammonium sulfate at 102 °C ± 2 °C for not less than 2 h. Cool to room temperature in a desiccator.

5.10 Tryptophan ($C_{11}H_{12}N_2O_2$) or lysine hydrochloride ($C_6H_{15}ClN_2O_2$), minimum assay 99 % (mass fraction).

Do not dry the reagents in an oven before use.

5.11 Sucrose, with a nitrogen content of not more than 0,002 % (mass fraction).

Do not dry the sucrose in an oven before use.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Water bath, capable of being maintained at between 38 °C and 40 °C.

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

6.3 Digestion block, aluminium alloy block or equivalent block, fitted with an adjustable temperature control and device for measuring block temperature.

6.4 Digestion tubes, of capacity 250 ml, suitable for use with the digestion block (6.3).

6.5 Exhaust manifold, suitable for use with the digestion tubes (6.4).

6.6 Centrifugal scrubber apparatus, filter pump or aspirator, constructed of acid-resistant material, for use with mains water supply.

6.7 Automatic pipettes (dispensers), of capacity 5 ml and 10 ml.

6.8 Graduated measuring cylinders, of capacity 50 ml.

6.9 Distillation unit, capable of steam distilling, manual or semi-automatic, suited to accept the digestion tubes (6.4) and the conical flasks (6.10).

6.10 Conical flasks, of capacity 250 ml.

6.11 Burette, of capacity 25 ml, with a readability of at least 0,01 ml, complying with the requirements of ISO 385:—, class A. Alternatively, an automatic burette may be used if it fulfils the same requirements.

6.12 Automatic titrator, provided with a pH-meter.

The pH-meter shall be correctly calibrated in the range of pH 4 to 7 following normal laboratory pH-calibration procedures.

7 Sampling

Sampling is not part of the method specified in this part of ISO 8968|IDF 20. A recommended sampling method is given in ISO 707.

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

8 Preparation of test sample

Warm the test sample in the water bath (6.1) to between 38 °C and 40 °C. Cool the sample to room temperature while gently mixing the test sample immediately prior to weighing the test portion (see 9.1).

9 Procedure

9.1 Test portion and pretreatment

NOTE 1 In the normal course of events, the test samples are analysed in batches according to the described procedure.

Place the required number of Kjeldahl catalyst tablets (5.1), containing 7 g of potassium sulfate, in a clean and dry digestion tube (6.4). Weigh, to the nearest 0,1 mg, approximately 2 g of prepared test sample (Clause 8) into the tube.

NOTE 2 For advice on test portion size to apply this method to dairy products other than milk, see Annex B.

Carefully add 10 ml of sulfuric acid (5.2) using an automatic pipette (6.7), followed by three or four drops antifoaming agent (5.4). Gently swirl the tube to mix its contents. Leave the tube and its contents to stand for 5 min. Carefully, add 5 ml of hydrogen peroxide solution (5.3) by using an automatic pipette (6.7) while allowing the solution to run down the inside of the tube. Gently swirl the tube again to mix its contents. Leave to stand for 10 min.

9.2 Determination

9.2.1 Digestion

Transfer the digestion tube and its contents (9.1) to the digestion block (6.3) set at the temperature specified by the manufacturer. Immediately place the exhaust manifold (6.5), which is connected to a centrifugal scrubber or similar device (6.6), in the top of the tube.

Digest the test portion for the period specified by the manufacturer of the block. Take care that charred digest does not foam up the neck of the digestion flask. Digestion is normally completed in 40 min to 60 min when the remaining solution is visually clear. Digest the test portion for a further 15 min after the solution has become clear.

Remove the tube from the block with the exhaust manifold in place and allow cooling for at least 15 min. Once the tubes are sufficiently cool to handle, remove the exhaust manifold and carefully add 50 ml of water to each tube.

NOTE In automated systems, the water addition is done automatically in the distillation unit.

9.2.2 Distillation

Transfer the digestion tube (9.2.1) to the distillation unit (6.9).

Where the distillation unit is fully automated to include titration of the ammonia content of the distillate, follow the manufacturer's instructions for operation of the distillation unit. Where titration of the ammonia content of the distillate is performed manually, the below-mentioned procedure applies.

Place a conical flask (6.10) containing 50 ml of boric acid solution with indicator solution (5.6.1) under the outlet of the condenser so that the delivery tube is below the surface of the excess boric acid solution. Adjust the distillation unit to dispense 40 ml of sodium hydroxide solution (5.5).

Operate the distillation unit in accordance with the manufacturer's instructions and distil off the ammonia liberated by the addition of the sodium hydroxide solution. Collect the distillate in the excess boric acid solution.

NOTE In a semi-automatic distillation unit, the addition of excess sodium hydroxide and the steam distillation are performed automatically.

9.2.3 Titration

Titrate the contents of the conical flask (9.2.2) with hydrochloric acid standard volumetric solution (5.8) using a burette (6.11). The endpoint is reached at the first trace of pink colour in the contents. Read the amount of titrant used by estimating the burette reading at least to the nearest 0,05 ml. An illuminated magnetic stirrer plate may aid visualization of the endpoint.

Alternatively, titrate the contents of the conical flask (9.2.2) with the hydrochloric acid standard volumetric solution (5.8) using a properly calibrated automatic titrator provided with a pH meter (6.12). The pH endpoint of the titration is reached at pH 4,6, being the steepest point in the titration curve (inflection point). Read on the automatic titrator the amount of the titrant used. The endpoint can be also detected by a photometric probe or cell.

NOTE The first trace of pink is observed between pH 4,6 and 4,3 for the indicator system and the boric acid solution specified in this method. In practice the rate of change of pH as a function of added 0,1 mol/l HCl is very fast within this range of pH. It takes about 0,05 ml of 0,1 mol/l HCl to change the pH by 0,3 units in the range of pH from 4,6 to 4,3 in this system.

9.3 Blank test

Carry out a blank test following the procedure described in 9.1 to 9.2.3 by taking 2 ml of water and about 0,20 g of sucrose (5.11) instead of the test portion. Keep a record of blank values. If blank values change, identify the cause.

It is recommended to add a further three or four drops of the antifoaming agent (5.4) to the digested blank prior to distillation, to reduce frothing.

The amount of titrant used in the blank test should always be greater than zero. Blanks within the same laboratory should be consistent over time. The blank should not be pink prior to beginning the titration. Usually in such cases, the conical flasks are not clean or water from the air that may condense on the outside of the condenser apparatus has dripped down into the collection flask to cause the contamination. Typical blank values are equal or below 0,15 ml.

NOTE The purpose of the sucrose in a blank is to act as organic material to consume an amount of sulfuric acid during digestion that is roughly equivalent to a test portion.

The blanks for test samples other than milk should contain a mass of sucrose that will consume approximately the same amount of acid during digestion as an average sample.

9.4 Recovery tests

9.4.1 Check the accuracy of the procedure regularly by means of the following recovery tests, carried out in accordance with 9.1 to 9.2.3.

9.4.2 Check the efficiency of the digestion and distillation procedures by using a test portion of either 0,06 g of lysine hydrochloride or 0,08 g of tryptophan (5.10), together with 0,15 g of sucrose (5.11), each weighed to the nearest 0,1 mg. Determine the nitrogen content according to the procedure described in 9.1 to 9.2.3. The expected nitrogen content is 15,33 % in lysine and 13,72 % in tryptophan, both mass fractions.

NOTE The amount of added sucrose is for the determination of the nitrogen recovery under the conditions of the average amount of residual acid present at the end of digestion when testing a test sample of milk. For samples other than milk, see ISO 8968-1|IDF 20-1:2001, Annex A.

The nitrogen recovery from the lysine hydrochloride or tryptophan shall lie between 98 % and 100,0 %.

9.4.3 Prepare a solution of the ammonium sulfate (5.9) with an exact concentration of 0,05 mol/l. Pipette a 10,0 ml aliquot of the ammonium sulfate solution into a digestion tube. Add 50 ml of water. Determine the nitrogen content of the solution according to the procedure described in 9.2.2 and 9.2.3.

Alternatively, directly weigh 0,06 g of ammonium sulfate (5.9) into a digestion tube. The expected nitrogen content in ammonium sulfate is 21,09 % (mass fraction).

The nitrogen recovery from the ammonium sulfate shall be between 99,0 % and 100,0 %.

9.4.4 Results in either of the recovery tests (9.4.2 and 9.4.3) lower than 98,0 % (in 9.4.2) or 99,0 % (in 9.4.3), or higher than 100,0 %, indicate failures in the procedure and/or inaccurate concentration of the standard volumetric hydrochloric acid solution (5.8).

10 Calculation and expression of results

10.1 Calculation of nitrogen content

10.1.1 Calculate the nitrogen content of the sample w_N, expressed as a percentage by mass, by using the following equation

$$w_N = \frac{1{,}4007(V_s - V_b)M_r}{m}$$

where

V_s is the numerical value of the volume, in millilitres, of the hydrochloric acid standard volumetric solution (5.8) used in the determination (9.2.3), expressed to at least the nearest 0,05 ml;

V_b is the numerical value of the volume, in millilitres, of the hydrochloric acid standard volumetric solution (5.8) used in the blank test (9.3), expressed to at least the nearest 0,05 ml;

M_r is the numerical value of the exact molarity of the hydrochloric acid standard volumetric solution (5.8), expressed to four decimal places;

m is the numerical value of the mass, in grams, of the test portion (9.1), expressed to the nearest 0,1 mg.

10.1.2 Express the obtained results to four decimal places if needed for further calculations. For end results, express those obtained for the nitrogen content to three decimal places and those for the protein content to two decimal places.

The obtained results should not be rounded further until the final use of the test value is made. This is particularly true when the values are going to be used for further calculations. One example is when the individual test values obtained from the analysis of many sample materials are used to calculate method performance statistics for within and between laboratory variation. Another example is when the values are used as a reference for instrument calibration (e.g. infrared milk analyser) where the values from many samples will be used in a simple or multiple regression calculation. In such cases, the obtained results should not be rounded before they are used for further calculations.

10.2 Calculation of crude protein content

10.2.1 Calculate the crude protein content, w_p, expressed as a percentage by mass, using the following equation:

$$w_p = w_N \times 6{,}38$$

where

w_N is the nitrogen content of the sample, expressed as a percentage by mass to four decimal places (10.1);

6,38 is the generally accepted multiplying factor to express the nitrogen content as crude protein content.

10.2.2 Express the obtained results for the crude protein content to three decimal places if needed for further calculations. For end results (see 10.1.2), express these to two decimal places.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A.

The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,0055 % for nitrogen content (0,035 % for crude protein content).

11.3 Reproducibility

The absolute difference between two 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 0,0071 % for nitrogen content (0,045 % for crude protein content).

12 Test report

The test report shall specify:

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

b) the sampling method used, if known;

c) the test method used, with reference to this part of ISO 8968|IDF 20;

d) all operating details not specified in this part of ISO 8968|IDF 20, or regarded as optional, together with details of any incident which may have influenced the result(s);

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

Annex A
(informative)

Results of interlaboratory test

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 Table A.1. The international collaborative study was carried out by Cecalait Laboratory (Poligny, France) and involved 14 laboratories from 6 countries: Israel, Spain, Switzerland, Italy, Sweden and France.

The results of one laboratory were not considered due to low recovery of one of the standards (tryptophan). The test consisted of 6 milk samples in blind duplicates with 2 samples at each nitrogen level: low, medium and high.

Table A.1 — Results of the interlaboratory test

	Samples					
	S1	**S2**	**S3**	**S4**	**S5**	**S6**
No. of participating laboratories after eliminating outliers[a]	13	13	13	13	13	13
Mean value (g N per 100 g milk)	0,5995	0,5712	0,5414	0,4814	0,4219	0,3922
Repeatability standard deviation, s_r	0,0028	0,0008	0,0015	0,0020	0,0021	0,0021
Coefficient of variation of repeatability (%)	0,461	0,133	0,270	0,424	0,505	0,529
Repeatability limit, r (= 2,8 s_r), (g N per 100 g milk)	0,0078	0,0022	0,0041	0,0058	0,0060	0,0059
Reproducibility standard deviation, s_R (g N per 100 g milk)	0,0032	0,0018	0,0018	0,0045	0,0032	0,0023
Coefficient of variation of reproducibility (%)	0,526	0,312	0,334	0,939	0,762	0,581
Reproducibility limit, R (= 2,8 s_R), (g N per 100 g milk)	0,0089	0,0050	0,0051	0,0128	0,0091	0,0064

[a] After Grubbs and Cochran 1 % elimination.

The following data (all expressed in g N per 100 g milk) were obtained.

For 6 samples in the test: s_r = 0,0020 and r = 0,0055
s_R = 0,0030 and R = 0,0082

According to the Cochran test, however, the variance of sample S4 differs statistically from the results of the other samples. Taking that into account while eliminating the results of this sample, the precision data (all expressed in g N per 100 g milk) then become:

For 5 samples in the test: s_r = 0,0020 and r = 0,0055
s_R = 0,0025 and R = 0,0071

Annex B
(informative)

Modified procedure for analysis of other milk products

B.1 General

The procedure described in this part of ISO 8968|IDF 20 has been optimized and its performance evaluated for the analysis of milk. The same procedure may be used, with slight modification, for the determination of the nitrogen content of a range of milk products. However, it should be noted that the procedure and its performance for such uses have not been validated.

B.2 Procedure

Follow the procedure described in 9.1 to 9.4, but adjust the mass of the test portion, taken from an appropriately prepared test sample, so that it contains approximately 0,01 g of nitrogen or 0,06 g of protein (see Table B.1).

NOTE Further information with regard to the modified procedure for other milk products is given in ISO 8968-1|IDF 20-1:2001, Annex A.

Table B.1 — Calculation of mass of test portion to be taken to ensure that 0,01 g of nitrogen is digested

Product	**Approximate protein content** % (mass fraction)	**Approximate nitrogen content** % (mass fraction)	**Approximate mass of test portion,** g
Milk powder (skimmed, whole or fat, filled)	32,0	5,0	0,2
Whey powder	12,5	2,0	0,5
Whey	0,8	0,13	8,0
Cheese	26,0	4,0	0,25
Cream	1,3 to 2,6	0,2 to 0,4	2,5 to 5,0

For samples such as cream, it might be necessary to use 20 ml of sulfuric acid to obtain complete digestion. The volume of sodium hydroxide solution added prior to distillation should be increased accordingly.

Bibliography

[1] ISO 707, *Milk and milk products — Guidance on sampling*[2]

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] ISO 8968-1|IDF 20-1:2001, *Milk — Determination of nitrogen content — Part 1: Kjeldahl method*

[5] ISO 8968-2|IDF 20-2, *Milk — Determination of nitrogen content — Part 2: Block-digestion method (Macro method)*

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[2] Equivalent to IDF 50.

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