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**Milk and milk products — Determination of
nitrogen content — Routine method using
combustion according to the Dumas
principle**

*Lait et produits laitiers — Détermination de la teneur en azote — Méthode
pratique par combustion selon le principe de Dumas*

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Contents

Page

Foreword.....	iv, vi
1 Scope	1
2 Normative reference	1
3 Term and definition	1
4 Principle.....	1
5 Reagents.....	1
6 Apparatus	2
7 Sampling.....	3
8 Preparation of test sample.....	3
8.1 General.....	3
8.2 Liquid test samples	3
9 Procedure	4
9.1 General.....	4
9.2 Test portion	4
9.3 Control of oxygen demand.....	4
9.4 Calibration	4
9.5 Determination.....	4
9.6 Detection and integration	5
10 Calculation and expression of results.....	5
10.1 Calculation	5
10.2 Expression of results	5
11 Precision.....	6
11.1 Interlaboratory test.....	6
11.2 Repeatability	6
11.3 Reproducibility.....	7
12 Test report	7
Annex A (informative) Flowchart for the basic design of a Dumas apparatus.....	8
Annex B (informative) Schemes of suitable types of Dumas apparatus.....	9
Annex C (informative) Equipment calibration	11
Annex D (informative) Comparison of results using the Dumas method with those obtained by the Kjeldahl method.....	13
Bibliography.....	14

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Foreword

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

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

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14891 | IDF 185 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.

Annexes A, B, C and D are for information only.

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Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO 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 National Committees casting a vote.

International Standard ISO 14891/IDF 185 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, *Main compounds of milk*, under the aegis of its project leader, Prof. H. Frister (DE).

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Milk and milk products — Determination of nitrogen content — Routine method using combustion according to the Dumas principle

1 Scope

This International Standard specifies a routine method for the determination of the total nitrogen content of milk and milk products.

2 Normative reference

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

ISO 8968-1 | IDF 20-1, *Milk — Determination of nitrogen content — Part 1: Kjeldahl method*
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3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1

nitrogen content

mass fraction of the total nitrogen determined by the procedure specified in this International Standard

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

4 Principle

A test portion is heated to destruction in a combustion tube at high temperature (900 °C to 1 200 °C) in an oxygen atmosphere according to the Dumas principle. All interfering components are removed from the resulting gas mixture. The nitrogen compounds in the test portion are converted to molecular nitrogen, followed by quantitative determination using a thermal conductivity detector. The nitrogen content is calculated using a microprocessor.

5 Reagents

Use only reagents of recognized analytical grade, or reagents of equivalent purity as specified by instrument manufacturers.

5.1 Carrier gases: use one of the following.

5.1.1 Carbon dioxide (CO₂), purity 99,9 % (volume fraction) for samples with moderate nitrogen contents; 99,995 % (volume fraction) for samples with low nitrogen contents.

5.1.2 Helium (He), purity 99,996 % (volume fraction).

5.2 Oxygen (O₂), purity 99,995 % (volume fraction).

5.3 Absorbent for SO₂, e.g. lead chromate to eliminate any sulfur compounds from the combustion products.

5.4 Copper oxide platinum catalyst (as filling material for the post-combustion tube).

Platinum catalyst (5 % of Pt on Al₂O₃) is blended with CuO at a ratio of 1 to 7, 8 or 9 parts.

To prevent separation as result of the different bulk densities of the two materials, it is not recommended to prepare the mixture before filling the tube. It is advisable to pour the platinum catalyst and copper oxide simultaneously into the post-combustion tube using a suitable funnel.

5.5 Silver wool.

Silver wool should be desegregated before being inserted in the post-combustion or reduction tube.

5.6 Silica (quartz) or glass wool.

5.7 Copper or tungsten (wire, cuttings or powder), suitable for filling the reduction tube.

To improve the precision of analytical results for low nitrogen contents, the use of copper or tungsten wire is recommended.

5.8 Phosphorus pentoxide (P₂O₅) or **granulated magnesium perchlorate** [Mg(ClO₄)₂], or another suitable support material, to fill the drying tubes.

5.9 Hollow corundum spheres or aluminium oxide pellets, suitable for filling the combustion tube.

5.10 Copper oxide (CuO), suitable as a filling material for the combustion tube.

5.11 Sodium hydroxide (NaOH), on a support material.

5.12 Standard nitrogen compounds, e.g. aspartic acid (C₄H₇NO₄), ethylenediaminetetraacetic acid (C₁₀H₁₆N₂O₈) (see annex C), or other suitable reference reagents with known constant nitrogen content (e.g. urea, tyrosine, phenylalanine); minimum assay should be 99 %.

For calibration purposes (see 9.4), use a standard nitrogen compound to prepare a standard series with a nitrogen content of 4 mg to 100 mg in equal steps of about 5 mg nitrogen. Prepare a second standard series with a nitrogen content of 10 mg to 200 mg in equal steps of about 10 mg nitrogen.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to 0,000 1 g.

6.2 Drying oven, capable of maintaining a temperature of between 105 °C and 110 °C.

6.3 Crucibles, e.g. made of stainless steel or ceramic, or **tin capsules**, suitable for the Dumas apparatus used.

NOTE 1 Several commercial instruments are provided with an automatic sampler.

NOTE 2 Some solid samples (e.g. powders) can be pressed to form pellets.

6.4 Dumas apparatus, with a thermal conductivity detector and a suitable device for signal integration.

Suitable types of Dumas apparatus available on the market operate according to the general scheme as given in Figure A.1, although they may be different in arrangement, both in composition and function.

Flow diagrams of two available instruments are shown as examples in annex B (Figures B.1 and B.2).

NOTE Instruments from the manufacturer Elementar Analysensysteme GmbH and LECO Instruments¹⁾ have been found to be suitable.

To avoid leaks, O-rings used for sealing shall be slightly lubricated prior to installation using a high-vacuum grease.

Experience has shown that it is important to clean all pieces of quartz and glassware carefully and to remove fingerprints from the tubes using a suitable solvent (e.g. acetone) before inserting them in the furnace.

7 Sampling

Sampling is not part of the method specified in this International Standard. 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

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8.1 General

Prepare the test sample in such a way that a homogeneous sample is obtained which is representative of the product.

ISO 14891:2002

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The preparation of the test samples may be carried out as given in subclause 8.1 of ISO 6732:1985.

NOTE In the future, however, the preparation of the test sample could be done automatically.

8.2 Liquid test samples

For liquid samples, such as milk and whey, the following pre-drying procedure is recommended when the instruments make use of metal sample boats or crucibles.

Weigh, to the nearest 0,000 1 g, 0,5 g to 3,5 g (depending on the nitrogen content and the type of instrument) of the prepared test sample into a boat or crucible and dry for 1 h in an oven (6.2) set at between 105 °C and 110 °C. If a rack loaded with up to 50 test samples is placed in the oven (6.2), a longer drying time could be necessary. Check whether the contents of the boats or crucibles are dry before taking them out of the oven.

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

9 Procedure

9.1 General

Since various Dumas instruments are available, different in design and handling, the operator shall carefully follow the instrument manufacturer's instructions for instrument setting up, optimization, calibration and operation. Switch the instrument on and allow its operating conditions to stabilize for about 1 h.

To assure that equivalent results are obtained, reference materials should be regularly used for method and instrument performance testing, as far as such materials are available. A matrix/instrument specific correction factor derived from those measurements may be used, if necessary.

If reference materials are not available, a regular comparison using the Kjeldahl method described in ISO 8968-1 | IDF 20-1 is recommended.

9.2 Test portion

Weigh, to the nearest 0,000 1 g, at least 0,2 g of the test sample (8.1 or 8.2) into a crucible or tin capsule (6.3). For samples with a protein content of less than 1 % (mass fraction), increase the amount of the test portion up to 3,5 g, depending on both the type of the Dumas equipment used and the nature of the test sample.

9.3 Control of oxygen demand

Some types of Dumas equipment require an estimation of the oxygen demand of the test portion. The calculated oxygen demand of some compounds used for calibration is given in annex C, Table C.1. For instruments with a self-optimizing oxygen control, the mass fraction of the residual oxygen content shall be between 2 % and 8 %.

9.4 Calibration

Pure standard nitrogen compounds (5.12) with a known constant nitrogen content are used for long-term instrument calibration. To prepare a calibration graph, the compound and its mass should be chosen in such a way that an absolute mass of nitrogen between 4 mg and 100 mg could be detected. For calibration, use 10 to 20 (or more) samples about equally covering the mass range of nitrogen. The same shall apply to the range between 10 mg to 200 mg of nitrogen. For more than 200 mg of nitrogen the calibration curve is expected to be nonlinear. In this nonlinear section, several straight segments may be used for the calibration. To assure the quality of calibration in this section, sample intakes should differ in steps of 1 mg to 5 mg of nitrogen.

Calibration may also be performed using aqueous standard solutions of nitrogen compounds.

Before starting a series of determinations, check the instrument response by running at least three nitrogen standards of known content. When the response is constant and the values obtained correspond to the long-term calibration as established above, proceed with the determination of the daily calibration factor by analysing at least four standard nitrogen compounds representing a nitrogen mass higher than the test samples to be analysed.

Use this factor for calibration of the actual measurement series.

Full range re-calibration will become necessary if the daily calibration factor deviates from its expected value by more than 10 % relative, or if essential parts of the instrument having a direct influence on calibration (e.g. thermal conductivity detector) have been replaced.

9.5 Determination

Operate the instrument and introduce the test portion according to manufacturer's instructions.

During analysis, the following processes take place in the instrument (see Figures B.1 and B.2):

The test portion is quantitatively combusted under standardized conditions. Volatile decomposition products (mainly N_2 , NO_x , CO_2 , H_2O) are transported by the carrier gas through a ballast column, if available on the instrument employed, or directly. The nitrogen oxides are reduced to molecular nitrogen and the excess of oxygen is bound in the reduction column.

Water is removed by means of an appropriate cooler, magnesium perchlorate or other drying agents. Unless carbon dioxide is used as carrier gas, it is removed by being passed over a suitable absorbent, e.g. sodium hydroxide on a supporting material.

Interfering compounds (e.g. volatile halogen and sulfur compounds) are removed by absorbents or contact materials (e.g. silver wool, sodium hydroxide on a suitable support material, or by all other material proposed by the manufacturers).

The nitrogen in the residual gas mixture consisting of nitrogen and carrier gas is passed through a thermal conductivity detector.

9.6 Detection and integration

A sensitive thermal conductivity cell, provided with automatic zero adjustment between each measurement of individual test portions and optimized for the carrier gas employed, is used for quantitative nitrogen determination. After amplification and A/D conversion of the detector signal, the obtained data are processed by a microprocessor and transferred to a personal computer.

10 Calculation and expression of results

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10.1 Calculation

10.1.1 Nitrogen content

ISO 14891:2002
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Results for the total nitrogen content, w_N , expressed as a percentage by mass, are available from the computer readouts or its printouts.

10.1.2 Crude protein content

The crude protein content, w_p , is obtained by using the following equation:

$$w_p = w_N \cdot R_f$$

where

w_p is the crude protein content, expressed as a percentage by mass;

w_N is the nitrogen content, expressed as a percentage by mass (10.1.1);

R_f is the agreed ratio factor ($R_f = 6,38$) between the protein and nitrogen content.

10.2 Expression of results

Express the results to three decimal places.