INTERNATIONAL STANDARD

ISO 5537

IDF 26

Second edition
2023-07

Dried milk and dried milk products — Determination of moisture content (reference method)

Lait sec et produits à base de lait sec — Détermination du taux d'humidité (méthode de référence)

Reference numbers
ISO 5537:2023(E)
IDF 26:2023(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11

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

Forewords

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 the European Committee for Standardization (CEN) Technical Committee CEN/TC 302, *Milk and milk products - Methods of sampling and analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement). It is being published jointly by ISO and IDF.

This second edition cancels and replaces the first edition (ISO 5537 | IDF 26:2004), which has been technically revised to add precision data for additional matrices.

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.* It is being published jointly by ISO and IDF.

The work was carried out by the IDF/ISO Action Team C54 of the *Standing Committee on Analytical Methods for Composition* under the aegis of its project leaders Dr H. van den Bijgaart (NL) and Mrs F. de Boer (NL).

Dried milk and dried milk products — Determination of moisture content (reference method)

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

1 Scope

This document specifies a method for the determination of the moisture content of all types of dried milk and dried milk products.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

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

3.1
moisture content
mass fraction of substances determined by the procedure specified in this document

Note 1 to entry: The moisture content is expressed as a percentage by mass.

4 Principle

A test portion is dried in a drying oven set at 87 °C for 5 h while dry air is passed through the test portion. The loss of mass of the test portion (which is related to the content of "non-chemically bound" water) is determined.

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

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

5.2 Drying oven, capable of being maintained at 87 °C ± 1 °C throughout the working space, with forced ventilation, thermostatically controlled, with the following equipment (see also Figure A.1).

5.2.1 Metal block, provided with channels of diameter 4,3 mm for holding the columns (5.4) in the drying oven.

5.2.2 Copper tubes, of length 1 500 mm, of internal diameter 2 mm, connected to the metal block in the drying oven.

5.2.3 Constant pressure regulator, provided with restrictors, capable of delivering 33 ml/min ± 2 ml/min of dry air to each column in the drying oven.

5.2.4 Tube, made of polycarbonate, of length 350 mm, of diameter 40 mm, filled with silica gel with hygrometric indicator.

The silica gel should have been dried at 150 °C for more than 12 h before use. Using the dry compressed air (5.11), no colour change of the hygrometric indicator should be noticed.

5.3 Desiccator, containing freshly dried silica gel with hygrometric indicator.

5.4 Columns, made of hard polypropylene (Phenomenex 1213–10211)[1)], of length 90 mm, of internal diameter 20 mm, provided with two polyethene filters (Phenomenex 1212–1023)[1)], narrowed towards one end to fit onto the block (5.2.1).

5.5 Synthetic stoppers, of adequate sizes to fit with the columns (5.4), made of soft polyethylene (Emergo 20273 B198 and 20371 U1)[1)].

5.6 Container, suitable for holding the columns (5.4).

5.7 Container, suitable for holding the synthetic stoppers (5.5).

5.8 Rod, made of polyvinyl chloride (PVC), of length 120 mm, of diameter 18 mm, suitable for placing the polyethylene filters in the column (5.4).

5.9 Tweezers, suitable for removing the polyethylene filters from the column (5.4).

5.10 Calibrated airflow meter, suitable for measuring a flow rate of 33 ml/min ± 2 ml/min.

Electronic airflow meters, as for instance used with gas chromatography, have been found to be suitable. For use, the inlet of the airflow meter is connected to a copper tube, with an internal diameter of 2 mm, that is punctured through the centre of a synthetic stopper (5.5).

5.11 Dry compressed air, minimum pressure of 200 kPa, moisture content of ≤ 0,01 mg/l at atmospheric pressure, free of any organic material.

5.12 Container, e.g. made of glass, provided with an airtight lid.

NOTE The apparatus mentioned in 5.2, 5.4, 5.5, 5.6, 5.7, 5.8 and 5.10 are available commercially (e.g. Funke-Dr.N.Gerber Labortechnik GmbH, Berlin, Germany)[2)].

1) Phenomenex and Emergo are examples of suitable products 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 products named. Equivalent products may be used if they can be shown to lead to the same results.

2) Funke Gerber is the trade name of products supplied by Funke-Dr.N.Gerber Labortechnik GmbH, Ringstrasse 42, 12105, Berlin, Germany. 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 Sampling

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

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

7 Preparation of test sample

Transfer the whole test sample to a dry, hermetically closed container (5.12) of capacity approximately twice the volume of the sample. Mix intensively by rotating and shaking the container.

Use a statistical sampling plan if there is evidence of sample inhomogeneity even after the intensive mixing mentioned above.

8 Procedure

8.1 Preparation of column

8.1.1 Set the constant pressure regulator at approximately 100 kPa. Place the synthetic stopper that is connected with the airflow meter (5.10) sequentially on top of each column. Measure each with the airflow meter (5.10) to give the airflow rate for each channel. Adjust the pressure, if necessary, to obtain an airflow of 33 ml/min ± 2 ml/min per channel.

8.1.2 Remove both synthetic stoppers from the column (5.4). Place the stoppers in the container (5.7) and store at room temperature.

8.1.3 Place the column, provided with filters placed in position as shown in Figure A.1, in the metal block (5.2.1) in the oven (5.2) set at 87 °C for at least 1 h.

8.1.4 Take the column out of the oven and close it with the synthetic stoppers (see 8.1.2). Place the closed column in the container (5.6) with the other prepared columns. Place the container with the columns in the desiccator (5.3). Close the desiccator and allow to cool for 60 min ± 10 min.

8.2 Preparation of test portion

8.2.1 After cooling (see 8.1.4), take one closed column at a time out of the container while leaving the container in the desiccator. Close the desiccator immediately after taking out a column. Weigh the closed column to the nearest 0,001 g, recording the mass to four decimal places.

8.2.2 Remove the synthetic stoppers from the pre-weighed column (see 8.2.1). Using tweezers (5.9), also remove the upper filter from the column. Keep the stoppers and the filter in a dry place in the weighing room. In cases where multiple columns are used, ensure that stoppers do not get mixed up between samples.

8.2.3 Add 5,0 g ± 0,3 g of prepared test sample (see Clause 7) to the column. Using the rod (5.8), place the upper filter back in position in the column. Remove any test sample material above the filter with a clean tissue. Close the column with both synthetic stoppers (see 8.2.2).

8.2.4 Immediately weigh the closed column to the nearest 0,001 g, recording the mass to four decimal places. Open the desiccator, place the closed column back into the container and close it again.

8.2.5 When the analysis involves more than one test sample, prepare all test portions by repeating the procedure from 8.2.1 to 8.2.4 for each separate test portion. Handle only one column at a time.

8.3 Determination

8.3.1 Open the desiccator. Take one closed column with the prepared test portion (see 8.2.4) at a time out of the container. Remove both synthetic stoppers from each column. Place the stoppers in the container (5.7) and store at room temperature.

8.3.2 Place each column and its contents in the metal block (5.2.1) which is placed in the drying oven (5.2). As soon as it is ready, close the oven. Dry the columns and their contents in the oven (5.2) set at 87 °C for 5 h ± 10 min.

8.3.3 After drying, remove each column from the metal block. Replace both synthetic stoppers. Open the desiccator and place the dried columns and their contents back into the container (5.6). Immediately close the desiccator after placing the last column into the container. Allow the whole to cool for 60 min ± 10 min.

8.3.4 After cooling (see 8.3.3), open the desiccator and, in the case of more than one test sample, take one closed column at a time out of the container leaving the container in the desiccator. Close the desiccator immediately after taking out a column. Weigh the closed column to the nearest 0,001 g, recording the mass to four decimal places.

9 Calculation and expression of results

9.1 Calculation

Calculate the mass fraction of moisture in the sample, w, in using Formula (1):

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad (1)$$

where

m_0 is the numerical value of the mass of the column, filters and stoppers (see 8.1.2), in grams;

m_1 is the numerical value of the mass of test portion, the column, filters and stoppers before drying (see 8.2.4), in grams;

m_2 is the numerical value of the mass of the test portion, column, filters and stoppers after drying (see 8.3.4), in grams.

9.2 Expression of test results

Express the test results to two decimal places.

10 Precision

10.1 Interlaboratory test

Details of interlaboratory tests on the precision of the method are given in Annex B. The values derived from this test are not always applicable to concentration ranges and matrices other than those given.