INTERNATIONAL STANDARD

ISO 3727-2

IDF 80-2

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Butter — Determination of moisture, non-fat solids and fat contents —

Part 2: Determination of non-fat solids content (Reference method)

Beurre — Détermination des teneurs en eau, en matière sèche non grasse et en matière grasse —

Partie 2: Détermination de la teneur en matière sèche non grasse (Méthode de référence)

Reference numbers
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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 member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 3727 | IDF 80 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3727-2 | IDF 80-2 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.

This first edition of ISO 3727-2 | IDF 80-2, together with ISO 3727-1 | IDF 80-1 and ISO 3727-3 | IDF 80-3, cancels and replaces ISO 3727:1977, which has been technically revised.

ISO 3727 | IDF 80 consists of the following parts, under the general title *Butter — Determination of moisture, non-fat solids and fat contents*:

— *Part 1: Determination of moisture content (Reference method)*

— *Part 2: Determination of non-fat solids content (Reference method)*

— *Part 3: Calculation of fat content*

Annex A of this part of ISO 3727 | IDF 80 is for information only.

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.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team, *Water*, of the Standing Committee on *Main components of milk*, under the aegis of its project leader, Mr G. J. Beutick (NL).

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Butter — Determination of moisture, non-fat solids and fat contents —

Part 2:
Determination of non-fat solids content (Reference method)

WARNING — The determination involves the use of volatile flammable solvents. When using such solvents, all electrical apparatus employed must comply with legislation relating to possible hazards in using such solvents.

1 Scope

This part of ISO 3727 | IDF 80 specifies the reference method for the determination of the non-fat solids content of butter.

2 Term and definition

For the purposes of this part of ISO 3727 | IDF 80, the following term and definition applies.

2.1
non-fat solids content
mass fraction of substances determined by the procedure specified in this part of ISO 3727 | IDF 80

NOTE The non-fat solids content is expressed as a percentage by mass.

3 Principle

Water from a known mass of butter is evaporated. The fat is extracted with light petroleum and the mass of substances remaining is determined.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified. The reagents shall leave no more than 1 mg of residue when the method is carried out by the method specified.

4.1 Light petroleum, with any boiling range of between 30 °C and 60 °C or, as equivalent, **pentane** [$CH_3(CH_2)_3CH_3$] with a boiling point of 36 °C.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

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

5.2 **Drying oven**, ventilated, thermostatically controlled, capable of maintaining a temperature of 102 °C ± 2 °C throughout the entire working space.

5.3 **Desiccator**, containing a suitable drying agent, for example freshly dried silica gel with hygrometric indicator.

5.4 **Dishes**, made of glazed porcelain or metal, resistant to corrosion under the conditions of the test, with height of between 20 mm and 40 mm, and diameter of between 50 mm and 70 mm, provided with a spout.

5.5 **Filter crucible**, made of sintered glass, pore size index of between 16 µm to 40 µm, with suction flask.

5.6 **Water bath** or **steam bath**, capable of boiling water.

5.7 **Glass stirring rod**.

6 Sampling

Sampling is not part of the method specified in this part of ISO 3727 | IDF 80. 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.

The test sample shall be received in an airtight container closed with a lid to avoid loss of moisture. The capacity of the container shall be such that the test sample occupies one-half to two-thirds of its volume.

Until commencing the preparation of the test sample, store the sample in the airtight container at a temperature of between 2 °C and 14 °C.

7 Preparation of test sample

7.1 Warm the test sample in the unopened airtight container to a temperature not exceeding 35 °C.

If fat separation may be expected (e.g. in low-hard-fraction test samples or through knowledge obtained from laboratory experience), warm such test samples in the unopened airtight container to a more typical homogenization temperature of between 24 °C and 30 °C.

Mix the test sample in the unopened container to a homogeneous state (either by a mechanical shaker or by hand) without getting any rupture of emulsion. Take precautions to avoid loss of moisture.

7.2 Before weighing, open the sample container and stir the test sample with a suitable device such as a spoon or spatula for no longer than 10 s.

8 Procedure

8.1 Preparation of the dish, rod and filter crucible

8.1.1 Dry the dish (5.4) for 1 h in the drying oven (5.2) set at 102 °C, with both the rod (5.7) and the filter crucible (5.5) inside the dish.

8.1.2 Cool the dish, together with the rod and the filter crucible, in the desiccator (5.3) to the temperature of the weighing room. Using the analytical balance (5.1), weigh the dish with the rod and the filter crucible to the nearest 1 mg.

NOTE A cooling period of 45 min is normally sufficient to allow the dish to reach the temperature of the weighing room.

8.1.3 Remove the filter crucible. Weigh the dish with the rod to the nearest 1 mg.

A similar combination of dish, rod and crucible should be used for each test portion if more than one test portion is analysed in the batch.

8.2 Preparation of test portion

8.2.1 Weigh, to the nearest 1 mg, approximately 5 g of the test sample (7.2) into the prepared dish (8.1.3).

8.2.2 Heat the dish with the test portion and the rod (8.2.1) for at least 15 h in the drying oven (5.2) set at 102 °C.

Alternatively, the dish with the test portion may be heated on the water bath (5.6), with as much as possible of the bottom of the dish exposed to the steam of the boiling water, for approximately 30 min. While heating, stir the test portion frequently with the glass rod. Heat the dish and the test portion subsequently for 30 min in the drying oven (5.2) set at 102 °C.

8.2.3 Cool the dish and the test portion to room temperature.

8.3 Determination

8.3.1 Add 15 ml of light petroleum (4.1) to the test portion in the dish (8.2.3) at a temperature of approximately 25 °C. Detach as much as possible of the sediment adhering to the wall or bottom of the dish using the glass rod. Transfer the solvent to the prepared filter crucible (8.1.3) and allow it to filter into the suction flask.

8.3.2 Carry out the procedure given in 8.3.1 four additional times. If no visible traces of fat are left in the dish, transfer quantitatively during the fourth washing as much as possible of the sediment to the filter crucible. If fat traces are left, repeat the procedure given in 8.3.1 again until all traces of fat are completely eliminated.

8.3.3 Wash the sediment in the crucible with 25 ml of prewarmed light petroleum (4.1) at about 25 °C.

8.3.4 Dry the washed dish, together with the glass rod and the filter crucible, for 30 min in the drying oven (5.2) set at 102 °C.

8.3.5 Allow the dish, together with the glass rod and the filter crucible, to cool in the desiccator to room temperature. Weigh the dish, together with the glass rod and the filter crucible, to the nearest 1 mg.

Repeat the drying procedure given in 8.3.4 and the above-mentioned cooling and weighing procedure until the difference in mass between two consecutive weighings of the dish and the rod and filter crucible does not exceed 1 mg or until the mass increases. In the latter case, use the lowest mass for the calculation.

9 Calculation and expression of results

9.1 Calculation

Calculate the non-fat solids content, w_{nf}, using the following equation:

$$w_{nf} = \frac{m_3 - m_0}{m_2 - m_1} \times 100\ \%$$

where

w_{nf} is the non-fat solids content of the sample, expressed as a mass fraction in percent;

m_0 is the mass, in grams, of the prepared dish, rod and filter crucible (8.1.2);

m_1 is the mass, in grams, of the prepared dish and rod (8.1.3);

m_2 is the mass, in grams, of the test portion together with the dish and rod before drying (8.2.1);

m_3 is the mass, in grams, of the residue together with the dish, rod and filter crucible after drying (8.3.5).

9.2 Expression of results

Express the test results to two decimal places.

10 Precision

10.1 Interlaboratory test

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

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

10.2 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 a mass fraction of 0,15 %.

10.3 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 a mass fraction of 0,25 %.

11 Test report

The test report shall specify:

— all information required for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this part of ISO 3727 | IDF 80;

— all operating details not specified in this part of ISO 3727 | IDF 80, or regarded as optional, together with details of any incident which may have influenced the result(s);

— the test result(s) obtained and, if the repeatability has been checked, the final quoted results obtained.

Annex A
(informative)

Results of interlaboratory trial

An international collaborative test involving ten laboratories and six countries was carried out on six samples divided into 12 blind duplicated samples.

Four samples were of unsalted butter and two samples were of salted butter. The test was organized by CNEVA, France, in collaboration with ADAS, United Kingdom. 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.

NOTE IDF 135 provides specific guidance for interlaboratory tests on methods of analysis and milk products. It is based on ISO 5725.

Table A.1 — Precision data

	Unsalted butter				Salted butter	
	A	B	C	D	E	F
No. of participating laboratories after eliminating outliers	8	8	8	8	8	8
Mean value, %	1,29	1,32	1,59	1,55	3,04	3,91
Repeatability standard deviation, s_r, %	0,04	0,05	0,05	0,04	0,03	0,08
Coefficient of variation of repeatability, %	3,09	3,22	2,91	2,37	1,12	2,15
Repeatability limit r (2,8 s_r), %	0,11	0,14	0,13	0,10	0,10	0,24
Reproducibility standard deviation, s_R, %	0,07	0,08	0,05	0,08	0,04	0,14
Coefficient of variation of reproducibility, %	5,32	4,98	3,14	5,42	1,44	3,53
Reproducibility limit R (2,8 s_R), %	0,19	0,21	0,14	0,24	0,12	0,39