INTERNATIONAL STANDARD

ISO 8851-2

IDF 191-2

First edition
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Butter — Determination of moisture, non-fat solids and fat contents (Routine methods) —

Part 2: Determination of non-fat solids content

Beurre — Détermination des teneurs en eau, en matière sèche non grasse et en matière grasse (Méthodes de routine) —

Partie 2: Détermination de la teneur en matière sèche non grasse

Reference numbers
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Foreword

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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.

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ISO 8851-2 | IDF 191-2 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.

ISO 8851 | IDF 191 consists of the following parts, under the general title *Butter — Determination of moisture, non-fat solids and fat contents (Routine methods)*:

— *Part 1: Determination of moisture content*

— *Part 2: Determination of non-fat solids content*

— *Part 3: Calculation of fat 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 the National Committees casting a vote.

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This first edition of ISO 8851-2 | IDF 191-2 cancels and replaces the first edition of IDF 11A:1986, which has been technically revised.

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

Butter — Determination of moisture, non-fat solids and fat contents (Routine methods) —

Part 2:
Determination of non-fat solids content

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 8851|IDF 191 specifies the routine method for the determination of the non-fat solids content of butter.

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 8851-1|IDF 191-1, *Butter — Determination of moisture, non-fat solids and fat contents (Routine methods) — Part 1: Determination of moisture content*

3 Terms and definitions

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

3.1
non-fat solids content
mass fraction of substances determined by the procedure specified in this part of ISO 8851|IDF 191

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

4 Principle

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

5 Reagents

It is recommended to use reagents of recognized analytical grade. Technical grade reagents may be used only if the results produced therewith are not significantly different from those produced using analytical grade reagents.

5.1 Petroleum ether, with any boiling range of between 40 °C and 80 °C.

The use of petroleum ether (60 to 80) °C BP is preferred over the use of petroleum ether (40 to 60) °C BP for safety reasons.

6 Apparatus

WARNING — As the determination involves the use of volatile flammable solvents, any electrical apparatus used shall comply with legislation relating to the hazards in using such solvents.

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 1 mg.

6.2 Drying oven, electrically heated, ventilated, thermostatically controlled, capable of being maintained at a temperature of 102 °C ± 2 °C throughout its working space.

6.3 Heating apparatus, for example an electric hot plate, Bunsen burner, Teclu-burner or spirit (alcohol) burner.

6.4 Beaker, made of aluminium, stainless steel or glass, with smooth surface, of such dimensions that losses by spattering or frothing are avoided.

If desired, the beaker may be provided with a glass stirring rod. Recommended dimensions are diameter 60 mm to 80 mm, and height 50 mm to 70 mm.

NOTE A 250 ml glass beaker meets the recommended dimensions.

6.5 Beaker tongs, to hold the beaker (6.4) by its outer surface only.

6.6 Stone or **metal plate**, flat, to allow rapid cooling of the beaker (6.4).

6.7 Measuring cylinders, of capacity 100 ml or, alternatively, **dispensing equipment**, to measure the volume of the petroleum ether.

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

6.9 Sloping stand, used for the beakers (6.4).

6.10 Glass rods.

7 Sampling

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

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

The laboratory sample should be received in an airtight container. The capacity of the container shall be such that one-half to two-thirds is filled by the sample. Store the samples in the closed container at a temperature of between 5 °C and 14 °C until commencing the preparation of the test sample.

8 Preparation of test sample

8.1 Warm the test sample in the original unopened container to a temperature at which the sample will be soft enough to facilitate thorough mixing to a homogeneous state (either by a mechanical shaker or by hand) without any rupture of the emulsion. The temperature of mixing should typically be between 24 °C and 28 °C, and shall normally not exceed 35 °C.

8.2 Where applicable, cool the test sample to ambient temperature while mixing until cooling is complete. As soon as possible after cooling, open the sample container and stir briefly for no longer than 10 s with a suitable device (e.g. a spoon or spatula) before weighing.

9 Procedure

9.1 Test portion

If the test portion has already been used to analyse the moisture content according to ISO 8851-1| IDF 191-1, start the procedure to determine the non-fat solids content at step 9.3.3 by the addition of 30 ml of petroleum ether (5.1) to the dried test portion remaining from the moisture analysis. However, if a fresh test portion is taken from the test sample, then start the procedure at step 9.2.

9.2 Preparation of the beaker

9.2.1 Dry the empty beaker (6.4) (with rod if used) in the drying oven (6.2), set at 102 °C, for at least 1 h.

9.2.2 Allow the empty beaker to cool on the stone or metal plate (6.6). Weigh it to the nearest 1 mg.

NOTE Generally a cooling time of 15 min is sufficient.

9.3 Determination

9.3.1 Weigh, to the nearest 1 mg, 9,5 g to 10,5 g of test sample (8.2) into the prepared beaker (9.2.2).

9.3.2 Heat the beaker and its contents, agitating continuously by swirling the beaker on or over the heating apparatus (6.3), or by stirring the contents with the glass rod. Use beaker tongs (6.5), if necessary, to handle the beakers. Control the heating and agitation so that losses by spattering and frothing are avoided. Continue the heating until frothing stops, the foam has broken and the colour of the non-fat solids has become light brown or yellow brown.

If using a hot plate (6.3), it is recommended to use a heating temperature between 120 °C and 160 °C. However, butter manufactured according to the Ammix process may require a lower temperature than 120 °C at the beginning of the heating process to avoid spattering of the test portion. In this case, the final temperature of the hot plate shall be between 140 °C and 160 °C to ensure all moisture is removed.

NOTE Normally, the heating time should not exceed 20 min.

WARNING — From step 9.3.3 to 9.3.12, the procedure should take place in a fume hood or on a vent bench.

9.3.3 Remove the beaker from the heating apparatus. Add 30 ml of petroleum ether (5.1) to its contents.

9.3.4 Heat the beaker and its contents so that the petroleum ether boils, while taking adequate precautions against fire. Then cool on the stone or metal plate (6.6). This heating step will prevent any non-fat solids floating in the petroleum ether.

NOTE When petroleum ether with a boiling point of (60 to 80) °C is used, the appearance of gentle bubbles is sufficient; vigorous boiling is not required.

9.3.5 Add 60 ml of petroleum ether (5.1) to the beaker contents. Mix carefully by rotating the beaker.

9.3.6 Place the beaker in the sloping stand (6.9). Allow the non-fat solids to settle for 5 min or until all the non-fat solids have settled on the bottom of the beaker.

9.3.7 Decant the petroleum ether to waste. Wipe the outside of the beaker, and especially the area from where the solvent is decanted, with a tissue dampened with clean petroleum ether (5.1), to remove fat residues.

Ensure that there is no loss of non-fat solids particles during decanting, as this will cause an underestimation of the non-fat solids content.

9.3.8 Repeat the petroleum ether solvent extraction steps described in 9.3.5 to 9.3.7.

9.3.9 Heat the beaker and its contents gently [e.g. between 70 °C and 80 °C on a hot plate (6.3)] until all solvent and moisture have been removed and the non-fat solids are thoroughly dried. Beware of the non-fat solids crusting and bursting. Heat the beaker gently. The drying period usually takes between 10 min and 15 min.

It is important that the non-fat solids are completely dried at this stage, otherwise it is difficult to break up the lumps (step 9.3.10).

9.3.10 Repeat the petroleum ether extraction by adding 40 ml of petroleum ether (5.1) and breaking up the lumps with a glass rod (6.10). Rinse the glass rod with 20 ml of petroleum ether, adding the rinsings to the beaker. Mix the contents carefully by rotating the beaker. Repeat steps 9.3.6 and 9.3.7.

9.3.11 Heat the beaker and its contents gently [e.g. between 70 °C and 80 °C on a hot plate (6.3)] until the non-fat solids are thoroughly dried. This usually takes 10 min.

9.3.12 Cool the beaker and its contents in a desiccator (6.8), then weigh to the nearest 1 mg.

10 Calculation and expression of results

10.1 Calculation

Calculate the non-fat solids content of the test sample, w_s, in percent by mass, by using the following equation:

$$w_s = \frac{(m_1 - m_0)}{(m_2 - m_0)} \times 100\ \%$$

where

m_0 is the mass, in grams, of the empty beaker (9.2.2), or the mass recorded from the determination in ISO 8851-1 | IDF 191-1 (8.1.2);

m_1 is the mass, in grams, of the beaker and dried non-fat solids (9.3.12);

m_2 is the mass, in grams, of the test portion and beaker (9.3.1), or the mass recorded from the determination in ISO 8851-1 | IDF 191-1 (8.2.1).

10.2 Expression of results

Express the results to one decimal place.

11 Precision

11.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are given in Annex A. The values derived from these interlaboratory tests 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 0,20 %.

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 0,39 %.

12 Test report

The test report shall specify:

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

b) the sampling method used, if known;

c) the test method used, together with reference to this part of ISO 8851 | IDF 191;

d) all operating details not specified in this part of ISO 8851 | IDF 191, or regarded as optional, together with details of any incident that may have influenced the result(s);

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

Annex A
(informative)

Results of interlaboratory trials

The results obtained from two collaborative studies [4], [5] were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2. Additionally, a meta-analysis was performed to calculate pooled precision estimates for repeatability and reproducibility using the following equation [5]:

$$x_p^2 = \frac{\sum v_i x_i^2}{\sum v_i}$$

where

x_p is the pooled estimate for repeatability or reproducibility;

x_i is the ith estimate of repeatability or reproducibility for each study;

v_i is the number of degrees of freedom associated with estimate x_i.

Table A.1 — Results of interlaboratory tests

Sample	Bibliographic reference	Number of labs.	Mean % [a]	r [b] % [a]	R [c] % [a]	RSD(r) [d] %	RSD(R) [e] %
Salted Ammix	[4]	8	2,77	0,16	0,42	2,01	5,43
Low salt Ammix	[4]	8	2,49	0,24	0,44	3,38	6,33
Unsalted Fritz	[4]	8	3,01	0,20	0,52	2,40	6,19
Salted Fritz	[4]	8	1,73	0,27	0,41	5,57	8,43
Salted Fritz	[4]	8	3,06	0,28	0,57	3,24	6,60
Unsalted Fritz	[4]	8	1,24	0,19	0,39	5,43	11,27
Salted Fritz	[4]	8	1,33	0,14	0,22	3,86	6,00
Unsalted Fritz	[4]	8	2,57	0,17	0,42	2,32	5,80
Salted Fritz	[5]	9	2,73	0,17	0,41	2,27	5,35
Unsalted Fritz	[5]	9	1,30	0,19	0,41	5,23	11,15
Salted Fritz	[5]	9	2,60	0,21	0,43	2,88	5,96
Salted Fritz	[5]	9	2,85	0,17	0,24	2,07	3,05
Salted Fritz	[5]	9	3,23	0,11	0,29	1,18	3,25
Salted Ammix	[5]	9	2,64	0,19	0,29	2,54	3,98
Low salt Ammix	[5]	9	2,06	0,24	0,30	4,27	5,19
Salted Ammix	[5]	9	2,60	0,28	0,28	3,85	3,85

[a] Mass fraction.
[b] Repeatability limit (2,8 s_r).
[c] Reproducibility limit (2,8 s_R).
[d] Relative repeatability standard deviation.
[e] Relative reproducibility standard deviation.

Bibliography

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

[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] EVERS, J.M., CRAWFORD, R.A., WIGHTMAN, L.M. and KISSLING, R.C. Routine methods for the determination of solids-not-fat, moisture and fat (by difference) in butter – robustness, bias and precision. *International Dairy Journal*, **11**(3), 2001 pp. 127-136

[5] EVERS, J.M., CRAWFORD, R.A. and KISSLING, R.C. Determination of moisture, solids-not-fat and fat-by-difference in butter using routine methods according to ISO 8851|IDF 191 – An international collaborative study and a meta-analysis. *International Dairy Journal*, **13**(1), 2003, pp. 55-65

1) Corresponds to IDF 50.

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