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

Part 1:  
**Determination of moisture content**

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

*Partie 1: Détermination de la teneur en eau*



Reference numbers  
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## Foreword

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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 8851-1|IDF 191-1 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 8851-1|IDF 191-1 cancels and replaces the first edition of IDF 137:1986, 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 1: Determination of moisture 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 moisture 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 3727-1|IDF 80-1, *Butter — Determination of moisture, non-fat solids and fat contents — Part 1: Determination of moisture content (Reference method)*

ISO 8851-2|IDF 191-2, *Butter — Determination of moisture, non-fat solids and fat contents (Routine method) — Part 2: Determination of non-fat solids content*

### 3 Terms and definitions

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

#### 3.1

##### **moisture content**

mass fraction of substances determined by the procedure specified in this part of ISO 8851|IDF 191

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

### 4 Principle

A known mass of butter is heated under controlled conditions in an open beaker to evaporate the volatile constituents. The loss of mass is determined.

## 5 Apparatus

Usual laboratory equipment and, in particular, the following.

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

**5.2 Drying oven**, electrically heated, ventilated, thermostatically controlled, capable of being maintained at a temperature of  $102\text{ °C} \pm 2\text{ °C}$  throughout its working space.

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

**5.4 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.

**5.5 Beaker tongs**, to hold the beaker (5.4) by its outer surface only.

**5.6 Stone or metal plate**, flat, to allow rapid cooling of the beaker (5.4).

## 6 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  $5\text{ °C}$  and  $14\text{ °C}$  until commencing the preparation of the test sample.

## 7 Preparation of test sample

**7.1** 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  $24\text{ °C}$  and  $28\text{ °C}$ , and shall normally not exceed  $35\text{ °C}$ .

**7.2** 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.

## 8 Procedure

### 8.1 Preparation of the beaker

**8.1.1** Dry the empty beaker (5.4) (with rod if used) in the drying oven (5.2), set at  $102\text{ °C}$ , for at least 1 h.

**8.1.2** Allow the empty beaker to cool on the stone or metal plate (5.6). Weigh it to the nearest 1 mg.

NOTE Generally a cooling time of 15 min is sufficient.

## 8.2 Determination

**8.2.1** Weigh, to the nearest 1 mg, 5 g to 10 g of test sample (7.2) into the prepared beaker (8.1.2).

If the dried test portion remaining from this determination will also be used for the determination of the non-fat solids content described in ISO 8851-2|IDF 191-2, weigh, to the nearest 1 mg, 9,5 g to 10,5 g of test sample (7.2) into the prepared beaker (8.1.2).

**8.2.2** Heat the beaker and its contents, agitating continuously by swirling the beaker on or over the heating apparatus (5.3), or by stirring the contents with the glass rod. Use beaker tongs (5.5), 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 (5.3), 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 contents of the beaker shall be between 140 °C and 160 °C to ensure all moisture is removed.

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

The determination of the endpoint of the heating process involves a subjective assessment. Therefore, periodically check the obtained results with this method against those obtained with the reference method described in ISO 3727-1|IDF 80-1.

**8.2.3** Allow the beaker and its contents to cool on the stone or metal plate (5.6). Weigh to the nearest 1 mg.

NOTE Generally a cooling time of 15 min is sufficient.

## 9 Calculation and expression of results

### 9.1 Calculation

Calculate the moisture content of the test sample,  $w_m$ , in percent by mass, by using the following equation:

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

where

$m_0$  is the mass, in grams, of the empty beaker (with rod if used) (8.1.2);

$m_1$  is the mass, in grams, of the test portion and beaker (with rod if used) before heating (8.2.1);

$m_2$  is the mass, in grams, of the test portion and beaker (with rod if used) after heating (8.2.3).

### 9.2 Expression of results

Express the test results to one decimal place.

## 10 Precision

### 10.1 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.

### 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 0,31 %.

### 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 0,42 %.

## 11 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 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  $i$ th 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 % <sup>a</sup>	$r$ <sup>b</sup> % <sup>a</sup>	$R$ <sup>c</sup> % <sup>a</sup>	RSD( $r$ ) <sup>d</sup> %	RSD( $R$ ) <sup>e</sup> %
Salted Ammix	[4]	9	15,61	0,39	0,40	0,89	0,91
Low salt Ammix	[4]	9	15,62	0,30	0,35	0,68	0,79
Unsalted Fritz	[4]	9	15,36	0,20	0,29	0,45	0,67
Salted Fritz	[4]	9	15,71	0,12	0,25	0,26	0,57
Salted Fritz	[4]	9	15,75	0,21	0,25	0,47	0,57
Salted Fritz	[4]	9	15,75	0,64	0,65	1,44	1,46
Unsalted Fritz	[4]	9	15,81	0,30	0,45	0,68	1,02
Salted Fritz	[5]	9	15,74	0,11	0,24	0,37	0,84
Unsalted Fritz	[5]	9	15,77	0,18	0,41	0,35	0,80
Salted Fritz	[5]	9	15,66	0,45	1,02	0,60	1,37
Salted Fritz	[5]	9	15,93	0,31	0,70	0,38	0,86
Salted Fritz	[5]	9	15,76	0,21	0,48	0,29	0,67
Salted Ammix	[5]	9	16,04	0,13	0,29	0,23	0,50
Low salt Ammix	[5]	9	15,90	0,38	0,85	0,61	1,38
Salted Ammix	[5]	9	15,78	0,30	0,68	0,50	1,12

- <sup>a</sup> Mass fraction.
- <sup>b</sup> Repeatability limit ( $2,8 s_r$ ).
- <sup>c</sup> Reproducibility limit ( $2,8 s_R$ )
- <sup>d</sup> Relative repeatability standard deviation.
- <sup>e</sup> Relative reproducibility standard deviation.

## Bibliography

- [1] ISO 707, *Milk and milk products — Guidance on sampling*<sup>1)</sup>
- [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

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1) Corresponds to IDF 50.

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