

BS ISO 6731:2010



BSI Standards Publication

Milk, cream and evaporated milk — Determination of total solids content (Reference method)

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National foreword

This British Standard is the UK implementation of ISO 6731:2010. It supersedes BS 1741-2:1990 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/5, Chemical analysis of milk and milk products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Milk, cream and evaporated milk —
Determination of total solids content
(Reference method)**

Lait, crème et lait concentré non sucré — Détermination de la matière sèche (Méthode de référence)



Reference numbers
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IDF 21:2010(E)

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Foreword

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ISO 6731|IDF 21 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This second edition of ISO 6731|IDF 21 cancels and replaces the first edition (ISO 6731:1989), of which it constitutes a minor revision.

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50 % of IDF National Committees casting a vote.

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ISO 6731|IDF 21 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the former Joint ISO-IDF Action Team on *Water*, now part of the Standing Committee on *Analytical methods for composition*.

This edition of ISO 6731|IDF 21 cancels and replaces IDF 21B:1987.

Milk, cream and evaporated milk — Determination of total solids content (Reference method)

1 Scope

This International Standard specifies the reference method for the determination of the total solids content of milk, cream and evaporated milk.

2 Terms and definitions

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

2.1

total solids content

mass fraction of substances remaining after completion of the heating process specified in this International Standard

NOTE Total solids content is expressed as a percentage by mass.

3 Principle

A test portion is predried on a boiling water bath and the remaining water subsequently evaporated in a drying oven at a temperature of $102\text{ °C} \pm 2\text{ °C}$.

4 Apparatus and materials

Unless otherwise stated, use only distilled or demineralized water or water of equivalent purity.

Usual laboratory apparatus and in particular the following.

4.1 Analytical balance.

4.2 Desiccator, provided with an efficient desiccant (e.g. freshly dried silica gel with a hygrometric indicator).

4.3 Boiling water bath, provided with openings of adjustable size.

4.4 Drying oven, ventilated, capable of being maintained thermostatically at $102\text{ °C} \pm 2\text{ °C}$ throughout the total working space.

4.5 Flat-bottom dishes, of height 20 mm to 25 mm, diameter 50 mm to 75 mm, and made of appropriate material (e.g. stainless steel, nickel or aluminium), provided with well-fitting, readily removable lids.

4.6 Water baths.

4.6.1 Water bath, capable of being maintained at 35 °C to 40 °C .

4.6.2 Water bath, capable of being maintained at 40 °C to 60 °C.

4.7 Homogenizer (optional; see 7.1).

5 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707 | IDF 50^[1].

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

6 Preparation of the test sample

6.1 Milk

Bring the sample to a temperature of 20 °C to 25 °C. Mix thoroughly to ensure a homogeneous distribution of the fat throughout the sample. Avoid agitating so vigorously as to cause frothing of the milk or churning of the fat. If it is found difficult to disperse the cream layer, warm slowly to 35 °C to 40 °C on a water bath (4.6.1) with careful mixing and incorporate any cream adhering to the container. Cool the sample quickly to 20 °C to 25 °C.

If desired, a homogenizer may be used to assist the dispersion of the fat.

NOTE Correct results cannot be expected if the sample contains separated liquid fat or separate visible irregularly shaped white particles adhering to the walls of the container.

6.2 Cream

Warm the sample slowly to a temperature of 35 °C to 40 °C on a water bath (4.6.1). Mix or stir the cream thoroughly but not so vigorously as to cause frothing or churning. Cool the sample quickly to 20 °C to 25 °C. In order to reduce the evaporation of water to a minimum during mixing, the container should be uncovered for as short a time as possible.

NOTE Correct results cannot be expected if adequate mixing of the sample is not achieved or if the sample shows any evidence of churning or any other signs of abnormality.

6.3 Evaporated milk

Shake the container thoroughly with frequent inversion. Open this container and pour the milk slowly into another container made of glass or other suitable material, provided with an airtight lid, taking care to incorporate in the sample any fat or other constituents adhering to the walls of the original container. Stir vigorously and close the container.

Heat the closed container in a water bath (4.6.2) at 40 °C to 60 °C. Remove and shake the container vigorously every 15 min. After 2 h, remove the container and cool to 20 °C to 25 °C. Remove the lid and mix thoroughly by stirring the milk with a spoon or spatula.

NOTE If the fat separates, correct results cannot be expected.

7 Procedure

7.1 Preparation of the dish

Heat a dish (4.5), with its lid alongside, in the oven (4.4) for at least 1 h. Place the lid on the dish and immediately transfer to the desiccator (4.2).

Allow to cool to room temperature (at least 30 min) and weigh to the nearest 0,1 mg.

7.2 Test portion

Quickly weigh, to the nearest 0,1 mg, 1 g to 5 g of the prepared test sample (depending on the expected solids content) into the prepared dish (7.1). In the case of milk or cream, tilt the dish to spread the test portion evenly over the bottom of the dish. In the case of evaporated milk, add 3 ml to 5 ml of water, tilt the dish to mix, and spread the test portion evenly over the bottom of the dish.

7.3 Determination

7.3.1 Place the dish without lid on the vigorously boiling water bath (4.3) in such a way that the bottom of the dish is maximally exposed to and directly heated by the steam. Leave for 30 min.

7.3.2 Remove the dish from the water bath and then heat it, with its lid alongside, in the oven (4.4) for 2 h. Place the lid on the dish and immediately transfer to the desiccator (4.2).

7.3.3 Allow the dish to cool to room temperature (at least 30 min) and weigh to the nearest 0,1 mg.

7.3.4 Again heat the dish, with its lid alongside, in the oven but for only 1 h. Place the lid on the dish and immediately transfer to the desiccator. Allow to cool as in 7.3.3 and weigh to the nearest 0,1 mg.

7.3.5 Repeat the operations specified in 7.3.4 until the difference in mass between two consecutive weighings does not exceed 1 mg. Record the lowest mass.

8 Expression of results

8.1 Method of calculation

The total solids content, expressed as a percentage by mass, is equal to

$$\frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the dish and lid (see 7.1);

m_1 is the mass, in grams, of the dish, lid and test portion (see 7.2);

m_2 is the mass, in grams, of the dish, lid and dried test portion (see 7.3.5).

Round the value obtained to the nearest 0,01 % mass fraction.

8.2 Precision

NOTE The values for repeatability and reproducibility are expressed at the 95 % probability level and were derived from the results of an inter-laboratory test (see Reference [3]) carried out in accordance with ISO 5725:1986^[2].

8.2.1 Repeatability

The difference between two single results found on identical test material by one analyst using the same apparatus within a short time interval will exceed the following values of total solids per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method:

— for milk	0,10 g
— for cream	0,20 g
— for evaporated milk	0,30 g

8.2.2 Reproducibility

The difference between two single and independent results found by two operators working in different laboratories on identical test material will exceed the following values of total solids per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method:

— for milk	0,20 g
— for cream	0,35 g
— for evaporated milk	0,50 g

9 Test report

The test report shall contain at least the following information:

- 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 International Standard (ISO 6731|IDF 21:2010);
- d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- e) the test result(s) obtained;
- f) if the repeatability has been checked, the final quoted result obtained.

Bibliography

- [1] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*
- [2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*¹⁾
- [3] STEIGER, G., MARTENS, R. *Bull. Int. Dairy Fed.* 1986, (207)

1) Superseded.

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