

INTERNATIONAL STANDARD

**ISO
6731**

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

iTeh STANDARD PREVIEW

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

[ISO 6731:1989](#)

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INTERNATIONAL

ISO



Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6731 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, in collaboration with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC) and will also be published by these organizations.

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 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 707 : 1985, *Milk and milk products — Methods of sampling*.

3 Definition

For the purposes of this International Standard, the following definition applies.

total solids content: The mass fraction of substances remaining after completion of the heating process specified in this International Standard.

It is expressed as a percentage by mass.

4 Principle

Pre-drying of a test portion on a boiling water-bath and subsequent evaporation of the remaining water in a drying oven at a temperature of $102\text{ °C} \pm 2\text{ °C}$.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Analytical balance.

5.2 Desiccator, provided with an efficient desiccant (for example freshly dried silica gel with a hygrometric indicator).

5.3 Boiling water-bath, provided with openings of adjustable size.

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

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

5.6 Water-baths.

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

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

5.7 Homogenizer (optional; see 7.1).

6 Sampling

See ISO 707.

7 Preparation of the test sample

7.1 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 (5.6.1) 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.

7.2 Cream

Warm the sample slowly to a temperature of 35 °C to 40 °C on a water-bath (5.6.1). 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.

7.3 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 air-tight 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 (5.6.2) 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.

8 Procedure

8.1 Preparation of the dish

Heat a dish (5.5), with its lid alongside, in the oven (5.4) for at least 1 h. Place the lid on the dish and immediately transfer to the desiccator (5.2).

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

8.2 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 (8.1). 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 distilled water or water of at least equivalent purity, tilt the dish to mix and spread the test portion evenly over the bottom of the dish.

8.3 Determination

8.3.1 Place the dish without lid on the vigorously boiling water-bath (5.3) in such a way that the bottom of the dish is maximally exposed to and directly heated by the steam. Leave for 30 min.

8.3.2 Remove the dish from the water-bath and then heat it, with its lid alongside, in the oven (5.4) for 2 h. Place the lid on the dish and immediately transfer to the desiccator (5.2).

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

8.3.4 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 8.3.3 and weigh to the nearest 0,1 mg.

8.3.5 Repeat the operations described in 8.3.4 until the difference in mass between two consecutive weighings does not exceed 1 mg. Record the lowest mass.

9 Expression of results

9.1 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 8.1);

m_1 is the mass, in grams, of the dish, lid and test portion (see 8.2);

m_2 is the mass, in grams, of the dish, lid and dried test portion (see 8.3.5).

Round the value obtained to the nearest 0,01 % (m/m).

9.2 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 STEIGER, G. and MARTENS, R. *Bulletin of the International Dairy Federation*, 1986, No. 207) carried out in accordance with ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

9.2.1 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

9.2.2 Reproducibility

The difference between two single and independent results found by two operators working in different laboratories on

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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

10 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the result.

The test report shall include all the information necessary for the complete identification of the sample.

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