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Sweetened condensed milk — Determination of total solids content (Reference method)

Lait concentré sucré — Détermination de la matière sèche (Méthode de référence)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

International Dairy Federation
Diamant Building • Boulevard Auguste Reyers 80 • B-1030 Brussels
Tel. + 32 2 733 98 88
Fax + 32 2 733 04 13
E-mail info@fil-idf.org
Web www.fil-idf.org

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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 6734 | IDF 15 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 6734 | IDF 15 cancels and replaces the first edition (ISO 6734:1989), 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 6734 | IDF 15 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 Joint ISO-IDF Action Team on *Water*, now part of the Standing Committee on *Analytical methods for composition*.

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Sweetened condensed 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 sweetened condensed 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 or steam bath and the remaining water is subsequently evaporated in the presence of sand at a temperature of 102 °C ± 2 °C in a drying oven.

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 Drying oven, ventilated, capable of being maintained thermostatically at 102 °C ± 2 °C throughout the total working space.

4.4 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.

4.5 Boiling water bath or **steam bath**, provided with openings of adjustable size.

4.6 Water bath, capable of being maintained at 30 °C to 40 °C.

4.7 Short glass stirring rods, flattened at one end and of suitable size to fit into the dish (4.4).

4.8 **Quartz sand** or **sea sand**, which passes through a woven wire cloth sieve of nominal size of openings 500 µm, but is retained by a sieve of nominal size of openings 180 µm, and which passes the following suitability test.

4.8.1 Place approximatively 20 g of sand in a dish containing a stirring rod (4.7). Heat the open dish and sand, stirring rod and lid in the oven (4.3) for at least 2 h. Fit the lid, allow the dish to cool in the desiccator (4.2) to the temperature of the balance room and weigh to the nearest 0,1 mg.

4.8.2 Moisten the sand with approximately 5 ml of water, mix the sand and water using the stirring rod and heat the dish and sand, stirring rod and lid in the oven (4.3) for at least 4 h. Fit the lid, allow the dish to cool in the desiccator (4.2) to the temperature of the balance room and weigh again to the nearest 0,1 mg.

The difference between the two weighings shall not exceed 0,5 mg.

If the difference between the weighings exceeds 0,5 mg, the sand can be made suitable for the determination as follows.

Leave the sand immersed in 25 % mass fraction hydrochloric acid ($\rho_{20} \approx 1{,}12$ g/ml) for 3 days. Stir occasionally. Decant the supernatant liquid as far as possible. Then wash the sand with water until the acid reaction has disappeared.

Heat the sand at approximately 160 °C for at least 4 h. Then repeat the test for the suitability of the sand as described above.

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

Open the container and thoroughly mix the milk with a spoon or spatula. Use an up and down rotary movement in such a way that the top layers and the contents of the lower corners are moved and mixed. Take care to incorporate in the sample any milk adhering to the wall and ends of the container.

Transfer the sample as completely as possible to a second container made of glass, provided with an airtight lid, and close this container. Heat the closed container in the water bath (4.6) maintained at 30 °C to 40 °C. Cool to 20 °C to 25 °C. Stir the sample in the container thoroughly. Mix until the whole mass is homogeneous. Close this container.

In the case of a collapsible tube, open it and transfer the contents to a glass container. Cut open the tube and transfer all material adhering to the interior as completely as possible to the container.

7 Procedure

7.1 Preparation of the dish

Heat a dish (4.4), containing approximately 25 g of sand (4.8), with its lid alongside and a stirring rod (4.7) on top of the lid, in the oven (4.3) for at least 1 h.

Place the lid (with the stirring rod on top) on the dish, immediately transfer to the desiccator (4.2), allow to cool to room temperature (at least 45 min), and weigh the dish, with lid and rod, to the nearest 0,1 mg.

7.2 Test portion

Tilt the sand to one side of the prepared dish (7.1), place on the clear space about 2,0 g of the prepared test sample, replace the lid with the stirring rod on top and weigh the dish to the nearest 0,1 mg.

7.3 Determination

7.3.1 Add 5 ml of water to the test portion in the dish and mix with the stirring rod. Thoroughly mix together the diluted test portion and the sand, and spread the mixture evenly over the bottom of the dish. Leave the stirring end of the rod in the mixture with the other end resting on the rim of the dish.

7.3.2 Heat the dish without lid on the boiling water bath or steam bath (4.5), with as much as possible of the bottom of the dish exposed to steam, for approximately 30 min, stirring the mixture frequently in the early stages of drying so that the mixture is well aerated and becomes crumbly.

7.3.3 Remove the dish from the water bath or steam bath, and then lay the stirring rod flat inside the dish and heat the dish, with its lid alongside, in the oven (4.3) for 2 h. Place the lid on the dish and immediately transfer to the desiccator (4.2).

7.3.4 Allow the dish to cool to room temperature (at least 45 min) and weigh to the nearest 0,1 mg.

7.3.5 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 7.3.4 and weigh to the nearest 0,1 mg.

7.3.6 Repeat the operations described in 7.3.5 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 (including sand), lid, and stirring rod (see 7.1);

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

m_2 is the mass, in grams, of the dish, lid, stirring rod, and dried test portion (including sand) (see 7.3.6).

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 0,4 g 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.

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 0,6 g 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.

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 6734 | IDF 15:2010);

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.