

# INTERNATIONAL STANDARD

# ISO 6734

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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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## Foreword

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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 6734 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.

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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 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 or steam-bath and subsequent evaporation of the remaining water in the presence of sand at a temperature of  $102\text{ °C} \pm 2\text{ °C}$  in a drying oven.

## 5 Apparatus and materials

The water used shall be distilled water or water of at least equivalent purity.

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

**5.4 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.

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

**5.6 Water-bath**, capable of being maintained at  $30\text{ °C}$  to  $40\text{ °C}$ .

**5.7 Short glass stirring rods**, flattened at one end and of suitable size to fit into the dish (5.4).

**5.8 Quartz sand or sea sand**, which passes through a woven wire cloth sieve of nominal aperture size  $500\text{ }\mu\text{m}$ , but is retained by a sieve of nominal aperture size  $180\text{ }\mu\text{m}$ , and which passes the following suitability test.

**5.8.1** Place approximately 20 g of sand in a dish containing a stirring rod (5.7). Heat the open dish and sand, stirring rod and lid in the oven (5.3) for at least 2 h. Fit the lid, allow the dish to cool in the desiccator (5.2) to the temperature of the balance room and weigh to the nearest 0,1 mg.

**5.8.2** 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 (5.3) for at least 4 h. Fit the lid, allow the dish to cool in the desiccator (5.2) 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.

NOTE — If this requirement is not met, the sand can be made suitable for the determination as follows.

Leave the sand immersed in 25 % (*m/m*) hydrochloric acid ( $\rho_{20} \approx 1,12\text{ g/ml}$ ) 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  $160\text{ °C}$  for at least 4 h. Then repeat the test for the suitability of the sand as described above.

## 6 Sampling

See ISO 707.

## 7 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 (5.6) 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.

## 8 Procedure

### 8.1 Preparation of the dish

Heat a dish (5.4), containing approximately 25 g of sand (5.8) with its lid alongside and a stirring rod (5.7) on top of the lid, in the oven (5.3) for at least 1 h.

Place the lid (with the stirring rod on top) on the dish, immediately transfer to the desiccator (5.2), allow to cool to room temperature (at least 45 min), and weigh the dish, with lid and rod, to the nearest 0,1 mg.

### 8.2 Test portion

Tilt the sand to one side of the prepared dish (8.1), 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.

### 8.3 Determination

**8.3.1** 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.

**8.3.2** Heat the dish without lid on the boiling water-bath or steam-bath (5.5), 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.

**8.3.3** 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 (5.3) for 2 h. Place the lid on the dish and immediately transfer to the desiccator (5.2).

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

**8.3.5** 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 8.3.4 and weigh to the nearest 0,1 mg.

**8.3.6** Repeat the operations described in 8.3.5 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 (including sand), lid and stirring rod (see 8.1);

$m_1$  is the mass, in grams, of the dish (including sand), lid, stirring rod and test portion (see 8.2);

$m_2$  is the mass, in grams, of the dish, lid, stirring rod and dried test portion (including sand) (see 8.3.6).

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*, 1988, No. 235) 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 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.

### 9.2.2 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.

### 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 information necessary for the complete identification of the sample.

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