International Standard 1737

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Evaporated milk and sweetened condensed milk — Determination of fat content (Reference method)

Lait concentré sucré ou non sucré — Détermination de la teneur en matière grasse (Méthode de référence)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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.

International Standard ISO 1737 was developed by Technical Committee ISO/TC 34, *Agricultural food products*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 1737-1970, which had been approved by the member bodies of the following countries :

Australia	Greece	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Sweden
Chile	Israel	Switzerland
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Peru	USSR
Germany, F. R.	Poland	

No member body had expressed disapproval of the document.

NOTE — The method specified in this International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, USA) on the basis of a preliminary IDF Standard, for the purpose of being included in the FAO/WHO Code of Principles concerning milk and milk products and associated standards.

The text as approved by the above organizations was also published by FAO/WHO (Code of Principles, Standard No. B-7), by the IDF (IDF Standard 13A) and by the AOAC [Official Methods of Analysis, 12th edition (1975) 16.154/16.167].

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Evaporated milk and sweetened condensed milk — Determination of fat content (Reference method)

1 Scope and field of application

This International Standard specifies the reference method for the determination of the fat content of evaporated milk and sweetened condensed milk.

The method is applicable to evaporated whole milk, evaporated skimmed milk, sweetened condensed whole milk and sweetened condensed skimmed milk.

2 Reference

ISO/R 707, *Milk and milk products — Sampling.*

3 Definition

fat content of evaporated milk and of sweetened condensed milk : The substances extracted by the procedure specified in this International Standard and expressed as a percentage by mass.

4 Principle

Extraction of the fat from a test portion in ammoniacal ethanolic solution using diethyl ether and light petroleum, evaporation of the solvents and weighing of the residue. (This is commonly known as the Röse-Gottlieb method.)

5 Reagents

All reagents shall be of recognized analytical quality. If necessary, solvents may be redistilled in the presence of about 1 g of butterfat per 100 ml of solvent. The water used shall be distilled water or water of at least equivalent purity.

5.1 Ammonium hydroxide, approximately 25 % (*m/m*) solution, ϱ_{20} approximately 0,91 g/ml, or a solution of higher, known concentration.

5.2 Ethanol, 94 to 97 % (*V/V*) or, failing this, ethanol denatured with methanol, butan-2-one or light petroleum.

5.3 Diethyl ether, peroxide-free.

NOTES

1 To test for peroxides, add to 10 ml of the ether in a small glass-stoppered cylinder, previously rinsed with the ether, 1 ml of freshly prepared 10 % potassium iodide solution. Shake and allow to stand for 1 min. No yellow colour should be observed in either layer.

2 Diethyl ether may be freed and maintained free from peroxides by adding wet zinc foil that has previously been completely immersed in dilute, acidified copper sulphate solution for 1 min and then washed in water. Approximately 80 cm^2 of zinc foil should be used per litre of ether and it should be cut in strips sufficiently long to reach at least half way up the container.

5.4 Light petroleum (petroleum ether), having any boiling range between 30 and 60 °C.

5.5 Mixed solvent.

Shortly before use, mix equal volumes of the diethyl ether (5.3) and the light petroleum (5.4).

NOTE — Where mixed solvent is specified, the diethyl ether or the light petroleum may be used alone instead.

6 Apparatus

Usual laboratory apparatus not otherwise specified, and the following

6.1 Analytical balance.

6.2 Suitable extraction apparatus, provided with ground glass stoppers, bark corks of good quality, or other closures unaffected by the solvents used.

NOTE — Treat bark corks by extracting successively with diethyl ether and light petroleum, keeping for at least 20 min in water at 60 °C or above, and cooling in the water so that they are saturated when used.

6.3 Thin-walled, flat-bottomed flasks, of capacity 150 to 250 ml.

6.4 Drying oven, well ventilated, capable of being thermostatically controlled at 102 ± 2 °C, or **vacuum drying oven,** capable of being controlled at 70 to 75 °C, at a pressure of less than 66 mbar (50 mmHg).

6.5 Boiling aid, fat-free, non-porous, non-friable in use, for example glass beads or pieces of silicon carbide.

NOTE — The use of a boiling aid is optional (see 8.3).

6.6 Centrifuge, in which the extraction apparatus (6.2) can be subjected to a rotational frequency of 500 to 600 min^{-1}.

NOTES

1 The use of a centrifuge is optional (see 8.5.2).

2 When using a centrifuge not provided with a three-phase motor, sparks may occur and care is therefore necessary to avoid explosion or fire due to the occurrence of solvent vapour following breakage of apparatus.

7 Sampling

Carry out sampling by the appropriate method described in ISO/R 707.

8 Procedure

8.1 Preparation of the test sample

8.1.1 Evaporated milk

Shake and invert the container. Open the container, pour the milk slowly into a second container (provided with an air-tight lid) and mix by repeated transfer, taking care to incorporate in the sample any fat or other constituent adhering to the wall and ends of the first container. Finally transfer the milk as completely as possible to the second container. Close the container.

In the case of a sealed can, warm the unopened can, if necessary, in a water bath at 40 to 60 °C. Remove and shake the can vigorously every 15 min. After 2 h, remove the can and allow it to cool to ambient temperature. Remove the lid and thoroughly mix the contents of the can by stirring with a spoon or spatula (if fat separates, do not test the sample).

8.1.2 Sweetened condensed milk

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 lower layers of the container are moved and mixed. Take care to incorporate in the sample any milk adhering to the wall and ends of the container. Transfer the milk as completely as possible to a second container (provided with an air-tight lid). Close the container.

In the case of a sealed can, warm the unopened can, if necessary, in a water bath at 30 to 40 °C. Open, scrape out all milk adhering to the interior of the can, transfer to a dish sufficiently large to permit thorough stirring, and mix until the whole mass is homogeneous.

In the case of a collapsible tube, open the tube and transfer the contents to a jar. Cut open the tube, scrape out all material adhering to the interior and transfer to the jar.

8.2 Blank test

Simultaneously with the determination, carry out a blank test on 10 ml of water using the same type of extraction apparatus, the same reagents in the same amounts and the same procedure. If the result of the blank test exceeds 0,000 5 g, check the reagents and purify or replace the impure reagent or reagents.

8.3 Preparation of the flask

Dry a flask (6.3) [if desired, with a boiling aid (6.5) to promote gentle boiling during the subsequent removal of the solvents] in the oven (6.4), controlled at 102 ± 2 °C, or in the vacuum drying oven (6.4), controlled at 70 to 75 °C, for 30 to 60 min. Allow the flask to cool to ambient temperature and weigh it to the nearest 0,000 1 g.

8.4 Test portion

Stir the prepared sample and immediately weigh, to the nearest 0,001 g, either in, or by difference into, the extraction apparatus (6.2), about 4 g of evaporated milk or 2 to 2,5 g of sweetened condensed milk. Add water to a volume of 10,5 ml and shake gently with slight warming (40 to 50 °C) until the product is completely dispersed. Cool, for example, in running water.

8.5 Determination

8.5.1 Add to the test portion 1,5 ml of the ammonium hydroxide solution (5.1), or an equivalent volume if a solution of higher concentration is used, and mix well.

Add 10 ml of the ethanol (5.2) and mix the liquids gently but thoroughly in the unclosed apparatus.

8.5.2 Add 25 ml of the diethyl ether (5.3), close the apparatus with a moistened stopper, and shake vigorously and invert repeatedly for 1 min. Cool, if necessary, in running water. Carefully remove the stopper and add 25 ml of the light petroleum (5.4), using the first few millilitres to rinse the stopper and the inside of the neck of the apparatus and allowing the rinsings to run into the apparatus.

Replace the stopper and shake and invert repeatedly for 30 s. Do not shake too vigorously if centrifuging is not to be performed. Allow the apparatus to stand until the upper liquid layer has become clear and is distinctly separated from the aqueous layer. Alternatively, perform the separation by means of the centrifuge (6.6).

8.5.3 Remove the stopper, rinse it and the inside of the neck of the apparatus with a few millilitres of the mixed solvent (5.5), and allow the rinsings to run into the apparatus. Carefully transfer as much as possible of the supernatant layer by decantation (see 10.1) or by means of a siphon tube into the dried flask (see 8.3).

Rinse the outside and the inside of the neck of the apparatus or the tip and the lower part of the siphon tube with a few millilitres of mixed solvent. Allow the rinsings from the outside of the apparatus to run into the flask and the rinsings from the inside of the neck or from the siphon tube to run into the extraction apparatus.

NOTE — If a siphon tube is used, the supernatant liquid may then be transferred, without further shaking, to the flask and the operations of rinsing and transference repeated.

8.5.4 Carry out a second extraction, repeating the procedures described in 8.5.2 and 8.5.3 [including the rinsing(s)] but using only 15 ml of the diethyl ether and 15 ml of the light petroleum.

8.5.5 Carry out a third extraction in the same way, but omit the final rinsing(s) (see 10.2).

8.5.6 Carefully evaporate or distil off as much solvent (including the ethanol) as possible. If the flask is of small capacity, some of the solvent will need to be removed in this manner after each extraction.

When there is no longer any odour of solvent, heat the flask, placed on its side, for 1 h in the oven (6.4), controlled at 102 ± 2 °C, or in the vacuum drying oven (6.4), controlled at 70 to 75 °C. Allow the flask to cool to ambient temperature as before (see 8.3), and weigh to the nearest 0,000 1 g.

Repeat the operations of heating for periods of 30 to 60 min, cooling and weighing until there is no further decrease in mass.

8.5.7 Add 15 to 25 ml of the light petroleum in order to determine whether the extracted matter is wholly soluble. Warm gently and swirl the solvent until all the fat is dissolved.

8.5.7.1 If the extracted matter is wholly soluble in the light petroleum, take the mass of fat as the difference between the final mass of the flask containing the extract and its initial mass (see 8.3).

8.5.7.2 If the extracted matter is not wholly soluble in the light petroleum, or in case of doubt and always in case of dispute, extract the fat completely from the flask by repeated washing with warm light petroleum, allowing the undissolved material to settle before each decantation. Rinse the outside of the neck of the flask three times. Heat the flask, placed on its side, for 1 h in the oven (6.4), controlled at 102 ± 2 °C, or in the vacuum drying oven (6.4), controlled at 70 to 75 °C, allow to cool to ambient temperature as before (see 8.3) and weigh to the nearest 0,000 1 g. Take the mass of fat as the difference between the mass of the flask containing the total extract and the final mass.

8.6 Number of determinations

Carry out two determinations on the same test sample.

9 Expression of results

9.1 Method of calculation and formula

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

$$\frac{(m_1 - m_2) - (m_3 - m_4)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the flask and fat after heating to constant mass;

m_2 is the mass, in grams, of the flask after the first heating (see 8.3) or, in the case of undissolved material, after the final heating;

m_3 is the mass, in grams, of the flask used in the blank test after heating to constant mass;

m_4 is the mass, in grams, of the flask used in the blank test after the first heating (see 8.3) or, in the case of undissolved material, after the final heating.

Take as the result the arithmetic mean of the two determinations (8.6), provided that the requirement for repeatability (see 9.2) is satisfied.

9.2 Repeatability

The difference between the results of the two determinations (8.6), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 0,05 g of fat per 100 g of product.

10 Notes on procedure

10.1 If the transfer is made by decantation, it may be necessary to add a little water to raise the interface between the two layers in order to facilitate the decantation.

10.2 It is not essential to carry out the third extraction (see 8.5.5) in the case of evaporated skimmed milk and sweetened condensed skimmed milk.

11 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for the complete identification of the sample.