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Sweetened condensed milk — Determination of sucrose content — Polarimetric method

Laits concentrés sucrés — Détermination de la teneur en saccharose — Méthode polarimétrique

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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 2911 was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* and was circulated to the Member Bodies in March 1975.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Poland
Austria	Ghana	Portugal
Belgium	Hungary	Romania
Brazil	India	Spain
Bulgaria	Israel	Turkey
Canada	Malaysia	United Kingdom
Czechoslovakia	Mexico	Yugoslavia
Ethiopa	Netherlands	
France	New Zealand	

No Member Body expressed disapproval of the document.

NOTE — The method specified in this International Standard has been developed on the basis of a standard of the International Dairy Federation.

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Sweetened condensed milk — Determination of sucrose content — Polarimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a polarimetric method for the determination of sucrose in sweetened condensed milk.

The method is applicable to sweetened condensed milk of normal composition prepared from whole, partially skimmed or skimmed milk and sucrose only and containing no altered sucrose.

2 REFERENCES

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

ISO/R 1737, *Evaporated milk and sweetened condensed milk — Determination of fat content (reference method).*

3 DEFINITION

sucrose content of sweetened condensed milk : The content of unaltered sucrose (saccharose), expressed as a percentage by mass, determined using the method specified below.

4 PRINCIPLE

Treatment with ammonia, so as to bring mutarotation of lactose to final equilibrium. Neutralization. Clarification by successive addition of zinc acetate and potassium hexacyanoferrate(II), followed by filtration.

On part of the filtrate, determination of the optical rotation.

On another part of the filtrate, inversion (based on the Clerget principle) by mild acid hydrolysis of the sucrose, leaving lactose and other sugars virtually unaffected. Determination of the optical rotation after inversion.

From the change in optical rotation on inversion, calculation of the sucrose content.

5 REAGENTS

All reagents shall be of analytical quality and the water used shall be distilled or shall be water of at least equal purity.

5.1 Zinc acetate, 2,0 N solution.

Dissolve in water 21,9 g of zinc acetate dihydrate $[Zn(C_2H_3O_2)_2.2H_2O]$ and 3 ml of glacial acetic acid and dilute to 100 ml.

5.2 Potassium hexacyanoferrate(II), 1,0 N solution.

Dissolve in water 10,6 g of potassium hexacyanoferrate(II) trihydrate $[K_4Fe(CN)_6.3H_2O]$ and dilute to 100 ml.

5.3 Hydrochloric acid, 6,35 ± 0,20 N [20 to 22 % (*m/m*)] solution.

5.4 Ammonia solution, 2,0 ± 0,2 N [3,5 % (*m/m*)].

5.5 Acetic acid, 2,0 ± 0,2 N [12 % (*m/m*)] solution, of exactly known normality.

6 APPARATUS

Usual laboratory apparatus, including the following items :

6.1 Analytical balance.

6.2 Glass beaker, 100 ml.

6.3 Volumetric flasks, 200 ml and 50 ml, conforming to class A of ISO 1042.

6.4 Pipette, either 20 ml, conforming to class A of ISO/R 648, or 40 ml, of corresponding accuracy.

6.5 Graduated measuring cylinders, 25 ml.

6.6 Graduated pipettes, 10 ml.

6.7 Filter funnel, diameter 8 to 10 cm, and folded medium grade filter papers, diameter 15 cm.

6.8 Polarimeter tube, exactly 2 dm long.

6.9 Polarimeter or saccharimeter :

6.9.1 Polarimeter using sodium light or mercury green light (mercury vapour lamp with prism or the special Wratten screen No. 77A), capable of being read to an accuracy of at least 0,05 angular degrees.

6.9.2 Saccharimeter with international sugar scale, using white light passing through a filter of 15 mm depth of a 6 % solution of potassium dichromate, or sodium light, capable of being read to an accuracy of at least 0,1 international sugar scale degrees.

6.10 Water baths, capable of being maintained at about 40 °C and at 60 ± 1 °C respectively.

7 SAMPLING

See ISO/R 707.

8 PROCEDURE

8.1 Preparation of the test sample

8.1.1 *Samples of recently manufactured products in which no appreciable separation of components may be expected*

Open the container, transfer all material adhering to the lid into the container and thoroughly mix by an up and down movement of a spoon in such a way that the top layers and the contents of the lower corners are moved and mixed. When the product is in a can, transfer the contents to a jar with a well-fitting lid. When the product is in a collapsible tube, transfer as much as possible of the contents to a jar with a well-fitting lid; then cut open the tube, scrape out all material adhering to the interior and transfer this also to the jar. Mix the contents of the jar as described above.

8.1.2 *Samples of older products and samples in which separation of components may be expected*

Heat in a water bath (6.10) at about 40 °C until the sample has nearly reached this temperature, open the container and proceed as described in 8.1.1. When the product is in a can or tube, transfer the contents to a jar, scrape out all material adhering to the walls (in the case of a collapsible tube, after cutting open the tube) and continue the mixing until the whole mass is homogeneous, reducing the size of any large crystals by crushing them with a glass rod. Close the jar with a well fitting lid. Allow to cool.

8.2 Check test

In order to check the procedure, the reagents and the apparatus, make a check test as described below in duplicate, on a mixture of 100 g of whole milk (or 110 g of skimmed milk) and 18,00 g of pure sucrose. This mixture corresponds to 40,00 g of condensed milk containing 45,0 % of sucrose.

Calculate the sugar content by means of the formulae in 9.1, using in formula (2) for m, F and P respectively the quantity of milk weighed and the fat and protein content of this milk, and in formula (1) for m, the value 40,00.

The mean of the values found shall be within the range 45 ± 0,1 % (*m/m*).

8.3 Determination

8.3.1 Weigh, to the nearest 0,01 g, a test portion of approximately 40 g of the well-mixed test sample into the glass beaker (6.2). Add 50 ml of hot water (80 to 90 °C) and mix well.

8.3.2 Transfer the mixture quantitatively to the 200 ml volumetric flask (6.3), rinsing the beaker with successive quantities of water at 60 °C, until the total volume is between 120 and 150 ml. Mix and cool to 20 ± 2 °C.

8.3.3 Add 5 ml of the ammonia solution (5.4). Mix again and then allow to stand for 15 min (20 ± 2 °C).

8.3.4 Neutralize the ammonia by adding the stoichiometric equivalent quantity of the acetic acid solution (5.5). Mix.

8.3.5 Add, mixing gently by rotating the tilted flask, 12,5 ml of the zinc acetate solution (5.1).

8.3.6 In the same manner as for the addition of the zinc acetate solution, add 12,5 ml of the potassium hexacyanoferrate(II) solution (5.2).

8.3.7 Bring the contents of the flask to 20 °C and dilute to the mark with water at 20 °C.

NOTE – Up to this stage, all additions of water or reagents shall be made in such a manner as to avoid the formation of air bubbles, and, with the same object in view, all mixing shall be done by rotation of the flask rather than by shaking. If air bubbles are found to be present before completion of dilution to 200 ml, they can be removed by temporarily connecting the flask to a vacuum pump, and rotating the flask.

8.3.8 Close the flask with a dry stopper and mix thoroughly by vigorous shaking.

8.3.9 Allow the precipitate to settle for a few minutes and then filter the solution through a dry filter paper, rejecting the first 25 ml of filtrate.

8.4 Direct polarization

Determine the optical rotation of the filtrate (8.3.9) at 20 ± 2 °C.

8.5 Inversion

Pipette 40 ml (two 20 ml portions if a 40 ml pipette is unavailable) of the filtrate (8.3.9) into the 50 ml volumetric flask (6.3). Add 6,0 ml of the hydrochloric acid (5.3).

Place the flask in a water bath (6.10) at 60 ± 1 °C for 15 min, the flask being immersed to the base of the neck. Mix by rotating the flask during the first 5 min, in which time the contents of the flask should have reached the temperature of the water bath. Cool to 20 °C and dilute to the mark with water at 20 °C. Mix and allow to stand for 1 h at this temperature.

8.6 Invert polarization

Determine the optical rotation of the inverted solution at 20 ± 2 °C. (If the temperature of the liquid in the polarization tube differs by more than 0,2° from 20 °C during the measurement, the temperature correction referred to in note 2 to 9.1 shall be applied.)

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formulae

The sucrose content, S, of the sample, expressed as a percentage by mass, is equal to :

$$\frac{A - 1{,}25\,B}{Q} \times \frac{V - \Delta V}{V} \times \frac{V}{L \times m} \qquad \ldots (1)$$

where

m is the mass of the test portion (8.3.1), in grams;

A is the direct polarimeter reading before inversion (8.4);

B is the polarimeter reading after inversion (8.6);

L is the length, in decimetres, of the polarimeter tube;

Q is the inversion division factor (the values of which are given in 9.2);

V is the volume, in millilitres, to which the sample is diluted before filtration (8.3.7);

ΔV is the correction, in millilitres, for the volume of the precipitate formed during the clarification :

$$\Delta V = \frac{m}{100}\,(1{,}08\,F + 1{,}55\,P) \qquad \ldots (2)$$

m being as above;

F being the percentage of fat in the sample (determined in accordance with ISO/R 1737);

P being the percentage of protein (6,38 times the nitrogen content) in the sample.

NOTES

1 When exactly 40,00 g of condensed milk are weighed and a polarimeter with sodium light, angular degrees and a 2 dm polarimeter tube at 20,0 ± 0,1 °C are used, the sucrose content of normal condensed milk [i.e. when C, defined in 9.2, is 9 % (m/m)] can be calculated from the following formula :

$$S = (A - 1{,}25\,B)\,(2{,}833 - 0{,}006\,12\,F - 0{,}008\,78\,P)$$

2 If the invert polarization is measured at a temperature, t, other than 20 ± 0,2 °C, the value of B should be multiplied by

$$1 + 0{,}003\,7\,(t - 20)$$

and this corrected value used in the calculation.

9.2 Values of the inversion division factor Q

The following formulae give accurate values for Q, for various sources of light with corrections, where necessary, for concentration and temperature.

Sodium light and polarimeter with scale in angular degrees :

$$Q = 0{,}882\,5 + 0{,}000\,6\,(C - 9) - 0{,}003\,3\,(t - 20)$$

Mercury green light and polarimeter with scale in angular degrees :

$$Q = 1{,}039\,2 + 0{,}000\,7\,(C - 9) - 0{,}003\,9\,(t - 20)$$

White light with dichromate filter or sodium light and saccharimeter with scale in international sugar scale degrees :

$$Q = 2{,}549 + 0{,}001\,7\,(C - 9) - 0{,}009\,5\,(t - 20)$$

where

C is the percentage of total sugars in the inverted solution according to the polarimetric reading;

t is the temperature, in degrees Celsius, of the inverted solution during the polarimetric reading.

NOTES

1 The percentage of total sugars C in the inverted solution may be calculated from the direct reading and the change on inversion in the usual manner, using the usual values for the specific rotations of sucrose, lactose and invert sugar.

The correction 0,000 6 $(C - 9)$ etc. is only accurate when C is approximately 9; for normal condensed milk, this correction can be neglected, C being very close to 9.

2 Variation in temperature from 20 °C makes little difference in the direct reading, but variation of more than 0,2 °C in the invert reading necessitates a correction. The correction factor given in note 2 to 9.1 is only accurate between 18 and 22 °C.

9.3 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,3 g of sucrose per 100 g of sweetened condensed milk.

10 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.

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