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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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Contents

Foreword

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ISO 2911 | IDF 35 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

This edition of ISO 2911 | IDF 35 cancels and replaces ISO 2911:1976, of which it constitutes a minor revision. Only editorial changes have been made.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Group of Experts, *Lactose* (E6), under the aegis of its project leader, Mr E. Langridge (GB).

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Sweetened condensed milk — Determination of sucrose content — Polarimetric method

1 Scope

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 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1737, *Evaporated milk and sweetened condensed milk — Determination of fat content — Gravimetric method (Reference method)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
sucrose content of sweetened condensed milk
content of unaltered sucrose (saccharose) determined using the method specified in this International Standard

NOTE It is expressed as a mass fraction in percent.

4 Principle

A test sample is treated with ammonium hydroxide, so as to bring mutarotation of lactose to final equilibrium. It is neutralized and then clarified by successive additions of zinc acetate and potassium hexacyanoferrate(II), followed by filtration.

The optical rotation is determined on a portion of the filtrate.

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

The sucrose content is calculated from the change in optical rotation on inversion.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

5.1 Zinc acetate solution, 1,0 mol/l.

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

5.2 Potassium hexacyanoferrate(II) solution, 0,25 mol/l.

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

5.3 Dilute hydrochloric acid, c(HCl) = (6,35 $\pm$ 0,20) mol/l [20 % to 22 % (mass fraction)].

5.4 Ammonium hydroxide solution, $c(NH_4OH)$ = (2,0 $\pm$ 0,2) mol/l [3,5 % (mass fraction)].

5.5 Dilute acetic acid, $c(CH_3CO_2H)$ = (2,0 $\pm$ 0,2) mol/l [12 % (mass fraction)], of exactly known concentration.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 0,01 g.

6.2 Glass beaker, of capacity 100 ml.

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

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

6.5 Graduated measuring cylinders, of capacity 25 ml.

6.6 Graduated pipettes, of capacity 10 ml.

6.7 Filter funnel, of diameter 8 cm to 10 cm, and folded medium-grade **filter papers**, of 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 $\pm$ 1) °C respectively.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707[1].

8 Procedure

8.1 Preparation of 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 equations in 9.1, using in Equation (2) for m, F and P respectively the quantity of milk weighed and the fat and protein content of this milk, and in Equation (1) for m, the value 40,00.

The mean of the values found shall be within the range (45 $\pm$ 0,1) % (mass fraction).

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 °C 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 ml and 150 ml. Mix and cool to (20 $\pm$ 2) °C.

8.3.3 Add 5 ml of the ammonium hydroxide solution (5.4). Mix again and then allow to stand for 15 min at (20 $\pm$ 2) °C.

8.3.4 Neutralize the ammonium hydroxide by adding the stoichiometrically equivalent quantity of the dilute acetic acid (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.

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 carried out by rotation of the flask rather than by shaking. If air bubbles are found to be present before completion of the dilution to 200 ml, they may 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 dilute hydrochloric acid (5.3).

Place the flask in a water bath (6.10) set at 60 °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 °C from 20 °C during the measurement, the temperature correction [Equation (4)] given in 9.1 shall be applied.

9 Expression of results

9.1 Method of calculation and equations

The sucrose content, w_S, of the sample, expressed as a percentage by mass, is given by:

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

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:

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

where

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

F is the mass fraction, as a percentage, of fat in the sample (determined in accordance with ISO 1737);

P is the mass fraction, as a percentage, of protein (6,38 times the nitrogen content) in the sample.

NOTE 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 % (mass fraction)] can be calculated from the following equation:

$$w_S = (A - 1{,}25\,B)\,(2{,}833 - 0{,}006\,12\,F - 0{,}008\,78\,P) \qquad (3)$$

If the invert polarization is measured at a temperature, t, other than (20 ± 0,2) °C, the value B shall be multiplied by the correction factor

$$1 + 0{,}003\,7\,(t - 20) \qquad (4)$$

and this corrected value shall be used in the calculation.

9.2 Values of the inversion division factor, Q

The following equations 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:

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

Mercury green light and polarimeter with scale in angular degrees:

$$Q = 1{,}039\,2 + 0{,}000\,7\,(C - 9) - 0{,}003\,9\,(t - 20) \qquad (6)$$

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

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

where

C is the mass fraction, as a 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.

NOTE 1 The mass fraction 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 corrections 0,000 6 (C – 9), etc., in Equations (5), (6) and (7) are only accurate when C is approximately 9; for normal condensed milk, this correction can be neglected, C being very close to 9.

NOTE 2 Variations in temperature from 20 °C make little difference in the direct reading, but variations of more than 0,2 °C in the invert reading necessitate a correction. The correction factor given in Equation (4) in 9.1 is only accurate between 18 °C and 22 °C.

10 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,3 g of sucrose per 100 g of sweetened condensed milk.

11 Test report

The test report shall specify:

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;

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 results;

e) the test results obtained, or, if the repeatability has been checked, the final quoted result obtained.

Bibliography

[1] ISO 707, *Milk and milk products — Guidance on sampling*[1)]

1) Equivalent to IDF 50.

ICS 67.100.10

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