First Commission Directive of 13 November 1979 laying down
Community methods of analysis for testing certain partly or wholly
dehydrated preserved milk for human consumption (79/1067/EEC)

ANNEX II

METHODS OF ANALYSIS RELATING TO THE COMPOSITION
OF CERTAIN PARTLY OR WHOLLY DEHYDRATED PRESERVED
MILK PRODUCTS INTENDED FOR HUMAN CONSUMPTION
METHOD 5: DETERMINATION OF SUCROSE CONTENT (POLARIMETERIC METHOD)

1.   SCOPE AND FIELD OF APPLICATION

This method determines the sucrose content of:
—   sweetened condensed milk,
—   sweetened condensed partly skimmed milk,
—   sweetened condensed skimmed milk.

Samples must not contain invert sugar.

2.   DEFINITION

The sucrose content of sweetened condensed milks: the sucrose content as determined by the method specified.

3.   PRINCIPLE

The method is based on the principle of the Clerget inversion, a mild treatment of the sample with acid which produces complete hydrolysis of sucrose but almost none of lactose or other sugars. The sucrose content is obtained from the change in rotating power of the solution.

A clear filtrate of the sample, without mutarotation by lactose, is prepared by treatment of the solution with ammonia followed by neutralization and clearing by the successive addition of zinc acetate and potassium hexacyanoferrate II solutions.

In a portion of the filtrate the sucrose is hydrolyzed in a specified manner.

From the rotation of the filtrate before and after inversion, the sucrose content is calculated using the appropriate formulae.

4.   REAGENTS

4.1.   Zinc acetate solution, 1 M: dissolve 21,9 g crystallized zinc acetate dihydrate $Zn(C_2H.O_2)_2.2H_2O$ and 3 ml glacial acetic acid in water and make up to 100 ml with water.

4.2.   Potassium hexacyanoferrate (II) solution, 0,25 M: dissolve 10,6 g crystallized potassium hexacyanoferrate (II) trihydrate $K_4[Fe(CN)_6]. 3H_2O$ in water and make up to 100 ml with water.

4.3.   Hydrochloric acid solution, $6,35 \pm 0,2$ M (20 to 22 %) or $5,0 \pm 0,2$ M (16 to 18 %).

4.4.   Ammonia solution, $2,0 \pm 0,2$ M (3,5 %).

4.5.   Acetic acid solution, $2,0 \pm 0,2$ M (12 %).

4.6.   Bromothymol blue indicator, 1 % (m/v) solution in ethanol.

5.   APPARATUS

5.1.   Balance, sensitivity 10 mg.

5.2.   Polarimeter tube, 2dm, of exactly calibrated length.

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5.3.   Polarimeter or saccarimeter:

(a)   Polarimeter with sodium light or mercury green light (mercury vapour lamp with prism or the special Wratten Screen No 77 A), to be read with an accuracy of at least 0.05 angular degrees,

(b)   Saccarimeter with international sugar scale, using white light passing through a filter of 15 mm of a 6 % solution of potassium bichromate, or sodium light, to be read with an accuracy of at least $0,1^o$ on the international sugar scale.

5.4.   Water bath, regulated at $60\ ^oC \pm 1\ ^oC$.

6.   PROCEDURE

6.1.   Control determination

In order to standardize the procedure, reagents and apparatus, carry out a control determination in duplicate as described below using a mixture of 100 g of milk and 18 g pure sucrose or a mixture of 110 g of skimmed milk and 18 g pure sucrose, each corresponding to 40 g of condensed milk containing 45 % sucrose. Calculate the sugar content using the formulae under 7, substituting for M, F and P respectively in formula 1 the quantity of milk taken and the fat and protein content of this milk, and in formula 2 for M, the value of 40,0. The mean of the values found shall not differ by more than 0,2 % from 45,0 %.

6.2.   Determination

6.2.1.   Weigh to within 10 mg, approximately 40 g of the well mixed sample into a 100 ml glass beaker. Add 50 ml of hot water (80 to 90 ^oC) and mix well.

6.2.2.   Transfer the mixture quantitatively to a 200 ml measuring flask, rinsing the beaker with successive quantities of water at 60 ^oC, until the total volume is between 120 and 150 ml. Mix and cool to room temperature.

6.2.3.   Add 5 ml of the dilute ammonia solution (4.4). Mix again and then allow to stand for 15 minutes.

6.2.4.   Neutralize the ammonia by adding an equivalent quantity of the diluted solution of acetic acid (4.5). Determine the exact number of ml beforehand by titration of the ammonia solution using bromothymol blue as indicator (4.6). Mix.

6.2.5.   Add, with gently mixing by rotating the tilted flask, 12.5 ml of zinc acetate solution (4.1).

6.2.6.   Add 12.5 ml of potassium hexacyanoferrate (II) solution (4.2) in the same way as for the acetate solution.

6.2.7.   Bring the contents of the flask to 20 ^oC and make up to the 200 ml mark with water at 20 ^oC.

Note:

During any of the stages so far described all additions of water or reagents should have been made in such manner as to avoid the formation of air bubbles, and with the same object in view, all mixing should have been carried out by rotation of the flask rather than by shaking. If air bubbles are found to be present before making up to 200 ml volume, their removal can be assisted by temporarily connecting the flask to a vacuum pump, and rotating the flask.

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

6.2.9.   Allow to stand for a few minutes and then filter through a dry filter paper, rejecting the first 25 ml of filtrate.

6.2.10.   Direct polarization: determine the optical rotation of the filtrate at 20 ^{o}C ± 1 ^{o}C.

6.2.11.   Inversion: pipette 40 ml of the filtrate obtained above into a 50 ml volumetric flask. Add 6,0 ml of 6,35 M hydrochloric acid or 7,5 ml of 5,0 M hydrochloric acid (4.3).

Place the flask in a waterbath of 60 ^{o}C for 15 minutes, ensuring that the entire bulb of the flask has been immersed. Mix by a rotatory movement during the first five minutes, in which time the contents of the flask should have attained the temperature of the bath. Cool to 20 ^{o}C, and make up to volume with water at 20 ^{o}C. Mix and allow to stand for one hour at this temperature.

6.2.12.   *Invert polarization*

Determine the rotation of the inverted solution at 20 ^{o}C ± 0.2 ^{o}C. (However, if temperature T of the liquid in the polarization tube differs by more than 0.2 ^{o}C during the measurement, the temperature correction referred to under 7.2 must be applied.)

7.   EXPRESSION OF RESULTS

7.1.   Method of calculation

Calculate the sucrose content by means of the following formulae:

(1)   v =

$$\frac{M}{100}(1{,}08F + 1{,}55P)$$

(2)   S =

$$\frac{D - 1{,}25I}{Q} \times \frac{V-v}{V} \times \frac{V}{L \times M} \%$$

where:

S	= sucrose content;
M	= mass of the weighed sample in grams;
F	= percentage of fat in the sample;
P	= percentage Of protein (N x 6.38) in the sample;
V	= volume in ml to which the sample is diluted before filtration;
v	= correction in ml for the volume of the precipitate formed during clarification;
D	= direct polarimeter reading (polarization before inversion);
I	= polarimeter reading after inversion;
L	= length in dm of the polarimeter tube;
Q	= inversion factor, the values of which are given below.

Remarks:

(a)   When exactly 40,0 g of condensed milk are weighed and a polarimeter with sodium light, angular degrees and a 2dm polarimeter tube at 20,0 ^{o}C ± 0,1 ^{o}C is used the sucrose content of normal condensed milk (C = 9) can be calculated from the following formula:

S = (D — 1,25 I) x (2,833 — 0,00612 F — 0,00878 P)

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(b)   If the invert polarization is measured at a temperature other than 20 ^{o}C, the figures should be multiplied by:

(1 + 0,0037 (T — 20).

7.2.   Values of the inversion factor Q

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

Sodium light and polarimeter with angular degrees:

Q   = 0,8825 + 0,0006 (C — 9) — 0,0033 (T — 20).

Mercury green light and polarimeter with angular degrees:

Q   = 1,0392 + 0,0007 (C — 9) — 0,0039 (T — 20).

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

Q   = 2,549 + 0,0017 (C — 9) — 0,0095 (T — 20).

In the above formulae:

C   = Percentage of total sugars in the inverted solution as polarized,
T   = Temperature of the inverted solution in the polarimetric reading.

Note 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 0,0006 (C — 9) etc., is only accurate when C is approximately 9; for normal condensed milk, this correction can be neglected, C being close to 9.

Note 2:

Variation in temperature from 20 ^{o}C of 1 ^{o}C makes little difference in the direct reading, but variation of over 0,2 ^{o}C in the invert reading necessitates a correction. The correction - 0,0033 (T — 20) etc., is only accurate between 18 ^{o}C and 22 ^{o}C.

7.3.   Repeatability

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