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 3: DETERMINATION OF FAT CONTENT IN CONDENSED MILKS (RÖSE-GOTTLIEB METHOD)

1. SCOPE AND FIELD OF APPLICATION

This method determines the fat content of:

— unsweetened condensed high fat milk,
— unsweetened condensed milk,
— unsweetened condensed partly skimmed milk,
— unsweetened condensed skimmed milk,
— sweetened condensed milk,
— sweetened condensed partly skimmed milk,
— sweetened condensed skimmed milk.

2. DEFINITION

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

3. PRINCIPLE

The fat content is determined by extraction of the fat from an ammoniacal alcoholic solution of the sample with diethyl ether and light petroleum followed by evaporation of the solvents and weighing of the residue and calculation as a percentage by mass of the sample, according to the principle of Rose-Gottlieb.

4. REAGENTS

All reagents should conform to the requirements specified in the blank test (6.1). If necessary, reagents may be redistilled in the presence of about 1 g of butterfat for 100 ml of solvent.

4.1. Ammonia solution, approximately 25 % (m/m) NH_3 (density at 20 ^oC approximately 0.91 g/ml), or a stronger solution of known concentration.

4.2. Ethanol, 96 ± 2 % (v/v) or, if not available, ethanol denatured with methanol, ethyl methyl ketone or light petroleum.

4.3. Diethyl ether, peroxide-free.

Note 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 freshly prepared 10 % potassium iodide solution. Shake and let stand for one minute. No yellow colour should be observed in either layer.

Note 2:

Diethyl ether may be maintained free from peroxides by adding wet zinc foil that has been completely immersed in dilute acidified copper sulphate solution for one minute and subsequently washed with water. Use per litre approximately 8 000 mm^2 zinc foil; cut in strips long enough to reach at least halfway up the container.

4.4. Light petroleum (petroleum ether), with any boiling range between 30 and 60 ^{o}C.

4.5. Mixed solvent, prepared shortly before use by mixing equal volume of diethyl ether (4.3) and light petroleum (4.4) (where the use of mixed solvent is indicated, it may be replaced by either diethyl ether or light petroleum alone).

5. APPARATUS

5.1. Analytical balance.

5.2. Suitable extraction tubes or flasks, provided with ground glass stoppers or other closures un-. affected by the solvents used.

5.3. Flasks, thin-walled and flat-bottomed, 150 to 250 ml capacity.

5.4. Atmospheric pressure drying oven, well ventilated and thermostatically controlled (adjusted to operate at 102 ^{o}C ± 1 ^{o}C.

5.5. Anti-bumping granules, fat-free, non porous, non friable in use, e.g. glass beads or pieces of silicon carbide (the use of this material is optional; see clause 6.2.1.

5.6. Siphon, to fit extraction tubes.

5.7. Centrifuge (optional).

6. PROCEDURE

6.1. Blank test

At the same time as the determination of the fat content of the sample, carry out a blank determination on 10 ml of water using the same type of extraction apparatus, the same reagents in the same amounts and the same procedure as described hereafter, excluding clause 6.2.2. If the blank exceeds 0.5 mg, the reagents should be checked and the impure reagent or reagents should be purified or replaced.

6.2. Determination

6.2.1. Dry a flask (5.3) (together with, if required, some anti-bumping granules (5.5) to promote gentle boiling during the subsequent removal of the solvents) in the oven (5.4) for half to one hour. Allow the flask to cool to the temperature of the balance room and accurately weigh the cooled flask to the nearest 0,1 mg.

6.2.2. Stir the prepared sample and immediately weigh, to the nearest 1 mg, 2 to 2,5 g of the sample if sweetened or 4 to 5 g of the sample if unsweetened directly in, or by difference into, the extraction apparatus (5.2). Add water to 10,5 ml and shake gently with slight warming (40 to 50 ^{o}C) until the product is completely dispersed. The sample must be dispersed completely otherwise the determination should be repeated.

6.2.3. Add 1,5 ml ammonia (25 %) (4.1) or a corresponding volume of a stronger solution, and mix well.

6.2.4. Add 10 ml ethanol (4.2) and mix the liquids gently but thoroughly in the unclosed apparatus.

6.2.5. Add 25 ml diethyl ether (4.3). Cool under running water. Close the apparatus and shake vigorously and invert repeatedly for one minute.

6.2.6. Remove the stopper carefully and add 25 ml light petroleum (4.4) using the first few millilitres to rinse the stopper and inside of the neck of the apparatus, allowing the rinsings to run into the apparatus. Close by replacing the stopper and shake and invert repeatedly for 30 seconds. Do not shake too vigorously if centrifuging is not to be used in 6.2.7.

6.2.7. Allow the apparatus to stand until the upper liquid layer has become clear and has distinctly separated from the lower aqueous layer. Alternatively carry out the separation using a suitable centrifuge (5.7).

Note:

When a centrifuge which is not driven by a three-phase motor, is used, sparks may occur and care must therefore be taken to avoid an explosion or fire from any ether vapours, coming, for example, from a broken tube.

6.2.8. Remove the stopper, rinse it and the inside of the neck of the apparatus with a few millilitres of mixed solvent (4.5) and allow the rinsings to run into the apparatus. Carefully transfer as much as possible of the supernatant layer by decantation or by means of a siphon (5.6) into the pre pared flask (6.2.1).

Note:

If the transfer is not made using a siphon, it may be necessary to add a little water in order to raise the interface between the two layers thus aiding decantation.

6.2.9. Rinse the outside and the inside of the neck of the apparatus or the tip and the lower part of the siphon with a few millilitres of mixed solvent (4.5). Allow the rinsings from the outside of the apparatus to run into the flask and the rinsings from the inside of the neck and from the siphon to run into the extraction apparatus.

6.2.10. Make a second extraction by repeating the procedure of 6.2.5 to 6.2.9 inclusive but using only 15 ml diethyl ether and 15 ml light petroleum.

6.2.11. Make a third extraction by repeating the procedure of 6.2.10 but omit the final rinsing (6.2.9).

Note:

It is not mandatory to carry out this third extraction when analysing skimmed unsweetened condensed milk and skimmed sweetened condensed milk samples.

6.2.12. Carefully evaporate or distil off as much solvent (including the ethanol) as possible. If the flask is of small capacity, it will be necessary to remove some of the solvent as above after each extraction.

6.2.13. When there is no appreciable odour of solvent place the flask on its side in the oven and heat for one hour.

6.2.14. Remove the flask from the oven, allow to cool to the temperature of the balance room and accurately weigh to the nearest 0,1 mg.

6.2.15. Repeat 6.2.13 and 6.2.14 for heating periods of 30 to 60 minutes until the difference in mass of two successive weighings is less than 0.5 mg or until the mass increases. If an increase in mass occurs use the lowest mass obtained in the calculation (7.1). Let the final weight recorded be M1 g.

6.2.16. Add 15 to 25 ml light petroleum in order to confirm that the extracted matter is wholly soluble. Warm gently and swirl the solvent until all the fat is dissolved.

6.2.16.1. If the extracted matter is wholly soluble in the light petroleum, the mass of fat is the difference between the weights determined at stages 6.2.1 and 6.2.15.

6.2.16.2. If any insoluble matter is present, or in case of doubt, completely extract the fat from the flasks 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 one hour in the oven, allow to cool to the temperature of the balance room as before (6.2.1) and weigh to the nearest 0,1 mg. The mass of fat is the difference between the mass obtained at 6.2.15 and this final mass.

7. EXPRESSION OF RESULTS

7.1. Calculation

The mass, in g of fat extracted is:

(M1 — M2) — (B1 — B2)

and the fat content of the sample, expressed as a percentage is:

$$\frac{(M1 - M2)-(B1 - B2)}{S} \times 100$$

where:

M1	=	mass, in g of flask M with fat after stage 6.2.15;
M2	=	mass, in g of flask M after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;
B1	=	mass, in g of flask B of the blank after stage 6.2.15;
B2	=	mass, in g of flask B after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;
S	=	mass, in g of sample used.

7.2. Repeatability

The difference between results of two determinations carried out obtained simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0,05 g fat per 100 g of the product.