

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 1: DETERMINATION OF DRY MATTER CONTENT(oven 99 °C)

1. SCOPE AND FIELD OF APPLICATION

This method determines the dry matter 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 dry matter content of condensed milks: dry matter content as determined by the method specified.

3. PRINCIPLE

A known amount of the sample is diluted with water, mixed with sand and dried at a temperature of $99\text{ °C} \pm 1\text{ °C}$. The mass after drying is the mass of dry matter and is calculated as a percentage by mass of the sample.

4. REAGENTS

Quartz sand or sea sand, treated with hydrochloric acid (size of the grains: 0,18 to 0,5 mm, that is passing through a 500 micron sieve and retained by a 180 micron sieve). It should meet the following control test:

Heat about 25 g of sand for two hours in the drying oven (5.3) as described in 6.1. to 6.3. Add 5 ml of water, heat again in the oven for two hours, cool and reweigh. The difference between the two masses should not exceed 0,5 mg.

If necessary treat the sand with a 25 % hydrochloric acid solution for three days, mixing occasionally. Wash with water until the acid reaction disappears or the wash water is chloride free. Dry at 160 °C and re-test as above.

5. APPARATUS

5.1. Analytical balance.

5.2. Metal dishes, preferably of nickel, aluminium or stainless steel. The dishes must have lids which fit very well but which are readily removed. Suitable dimensions are: diameter 60 to 80 mm and depth about 25 mm.

5.3. Atmospheric-pressure drying oven, well ventilated, thermostatically controlled with temperature regulated at $99\text{ °C} \pm 1\text{ °C}$. The temperature should be uniform throughout the oven.

- 5.4. Desiccator, containing freshly activated silica gel with a water content indicator or an equivalent desiccant.
- 5.5. Glass rods, flattened at one end of such a length that they can fit inside the metal dishes (5.2).
- 5.6. Waterbath, boiling.
6. PROCEDURE
- 6.1. Place about 25 g sand (4) and a short glass rod (5.5) in the dish (5.2).
- 6.2. Without covering the dish and contents with the lid, place the dish, contents and lid in the oven (5.3) and heat for two hours.
- 6.3. Replace lid and transfer the dish to the desiccator (5.4). Allow to cool to room temperature and accurately weigh to the nearest 0,1 mg (M0).
- 6.4. Tilt the sand to one side of the dish. Introduce into the clear space about 1,5 g of the sample of sweetened condensed milk and 3,0 g of unsweetened condensed milk. Replace the lid and accurately weigh to the nearest 0,1 mg (M1).
- 6.5. Remove the lid, add 5 ml of water and, with the aid of the glass rod, mix the liquids and then the sand and the liquid portion. Leave the rod in the mixture.
- 6.6. Place the dish on a boiling waterbath (5.6) until the water has evaporated; this usually takes 20 minutes. Stir the mixture from time to time with the rod to keep the mass well aerated so that the mass when dry does not form a cake. Lay the rod inside the dish.
- 6.7. Place the dish and lid in the oven for one and a half hours.
- 6.8. Replace the lid, transfer the dish to the desiccator (5.4), allow to cool to room temperature and accurately weigh to the nearest 0,1 mg.
- 6.9. Replace the dish and lid in the oven, uncover the dish and heat it with its lid for a further hour.
- 6.10. Repeat process 6.8.
- 6.11. Repeat the described processes 6.9 and 6.10 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 M2 g.

7. EXPRESSION OF RESULTS

7.1. Method of calculation

The content of dry matter, calculated as a percentage by mass of the sample, is given by:

$$\frac{M2 - M0}{M1 - M0} \times 100$$

where:

- M0 = mass, in g of the dish, lid and sand after process 6.3;
 M1 = mass, in g of the dish, lid, sand and sample after process 6.4;
 M2 = mass, in g of the dish, lid, sand and dried sample after process 6.11.

7.2. Repeatability

The difference between the 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 0,2 g of dry matter per 100 g of the product.

8. CALCULATION OF TOTAL MILK SOLIDS AND MILK SOLIDS NOT FAT

8.1. The total milk solids content of the sweetened condensed milk is given by:

Total dry matter (obtained by method 1, Annex II) — sucrose (obtained by method 5, Annex II).

8.2. The milk solids not fat content of the sweetened condensed milks is given by:

Total dry matter (obtained by method 1, Annex II) — (sucrose content obtained by method 5, Annex II) and fat content (obtained by method 3, Annex II).

8.3. The milk solids not fat content of unsweetened condensed milks is given by:

Total dry matter (obtained by method 1, Annex II) — fat content (obtained by method 3, Annex II).