

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)

- Article 1 Member States shall take all measures necessary to ensure that...
Article 2 Where alternative methods for a single determination are
specified, the...
Article 3 Member States shall bring into force the laws, regulations and...
Article 4 This Directive is addressed to the Member States.
Signature

ANNEX I

SCOPE OF THE FIRST COMMUNITY METHODS OF ANALYSIS FOR CERTAIN
PARTLY OR . WHOLLY DEHYDRATED PRESERVED MILK DIRECTIVE

- I. General provisions
- II. Determination of dry matter in:
- III. Determination of moisture in:
- IV. Determination of fat in:
- V. Determination of sucrose in:
- VI. Determination of lactic acid and lactates in:
- VII. Determination of phosphatase activity in:

ANNEX II

METHODS OF ANALYSIS RELATING TO THE COMPOSITION
OF CERTAIN PARTLY OR WHOLLY DEHYDRATED PRESERVED
MILK PRODUCTS INTENDED FOR HUMAN CONSUMPTION

GENERAL PROVISIONS

- 1. PREPARATION OF THE SAMPLE FOR CHEMICAL ANALYSIS
 - 1.1. Unsweetened condensed high fat milk
 - 1.2. Sweetened condensed milk
 - 1.3. Dried high fat milk or high fat milk powder
- 2. REAGENTS
 - 2.1. Water
 - 2.1.1. Wherever mention is made of water for dissolution, dilution or...
 - 2.1.2. Wherever reference is made to 'dissolution', 'solution' or 'dilution' without...

- 2.2. Chemicals
- 3. EQUIPMENT
 - 3.1. Lists of equipment
 - 3.2. Analytical balance
- 4. EXPRESSION OF RESULTS
 - 4.1. Calculation of percentage
 - 4.2. Number of significant figures
- 5. TEST REPORT

METHOD 19. DETERMINATION OF DRY MATTER CONTENT

- 1. SCOPE AND FIELD OF APPLICATION
- 2. DEFINITION
- 3. PRINCIPLE
- 4. REAGENTS
- 5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Metal dishes, preferably of nickel, aluminium or stainless steel. The...
 - 5.3. Atmospheric-pressure drying oven, well ventilated, thermostatically controlled with temperature regulated...
 - 5.4. Desiccator, containing freshly activated silica gel with a water content...
 - 5.5. Glass rods, flattened at one end of such a length...
 - 5.6. Waterbath, boiling.
- 6. PROCEDURE
 - 6.1. Place about 25 g sand (4) and a short glass...
 - 6.2. Without covering the dish and contents with the lid, place...
 - 6.3. Replace lid and transfer the dish to the desiccator (5.4)...
 - 6.4. Tilt the sand to one side of the dish. Introduce...
 - 6.5. Remove the lid, add 5 ml of water and, with...
 - 6.6. Place the dish on a boiling waterbath (5.6) until the...
 - 6.7. Place the dish and lid in the oven for one...
 - 6.8. Replace the lid, transfer the dish to the desiccator (5.4),...
 - 6.9. Replace the dish and lid in the oven, uncover the...
 - 6.10. Repeat process 6.8.
 - 6.11. Repeat the described processes 6.9 and 6.10 until the difference...
- 7. EXPRESSION OF RESULTS
 - 7.1. Method of calculation
 - 7.2. Repeatability
- 8. CALCULATION OF TOTAL MILK SOLIDS AND MILK SOLIDS NOT FAT...
 - 8.1. The total milk solids content of the sweetened condensed milk...
 - 8.2. The milk solids not fat content of the sweetened condensed...
 - 8.3. The milk solids not fat content of unsweetened condensed milks...

METHOD 20. DETERMINATION OF MOISTURE

- 1. SCOPE AND FIELD OF APPLICATION
- 2. DEFINITION
- 3. PRINCIPLE
- 4. APPARATUS
 - 4.1. Analytical balance.
 - 4.2. Dishes, preferably of nickel, aluminium, stainless steel or glass. The...
 - 4.3. Atmospheric-pressure drying oven, well ventilated, thermostatically controlled with temperature regulation...

- 4.4. Desiccator, containing freshly activated silica gel with a water content...
5. PROCEDURE
 - 5.1. Uncover the dish (4.2) and place it and its lid...
 - 5.2. Place the lid on the dish and transfer the covered...
 - 5.3. Introduce approximately 2 g of dried milk sample into the...
 - 5.4. Uncover the dish and put it with its lid in...
 - 5.5. Replace the lid, transfer the covered dish to the desiccator,...
 - 5.6. Uncover the dish and heat it and its lid for...
 - 5.7. Repeat process 5.5.
 - 5.8. Repeat processes 5.6 and 5.5 until the decrease in mass...
6. EXPRESSION OF RESULTS
 - 6.1. Method of calculation
 - 6.2. Repeatability

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

1. SCOPE AND FIELD OF APPLICATION
2. DEFINITION
3. PRINCIPLE
4. REAGENTS
 - 4.1. Ammonia solution, approximately 25 % (m/m) NH₃ (density at 20 oC approximately...
 - 4.2. Ethanol, 96 ± 2 % (v/v) or, if not available, ethanol...
 - 4.3. Diethyl ether, peroxide-free.
 - 4.4. Light petroleum (petroleum ether), with any boiling range between 30...
 - 4.5. Mixed solvent, prepared shortly before use by mixing equal volume...
5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Suitable extraction tubes or flasks, provided with ground glass stoppers...
 - 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...
 - 5.5. Anti-bumping granules, fat-free, non porous, non friable in use, e.g....
 - 5.6. Siphon, to fit extraction tubes.
 - 5.7. Centrifuge (optional).
6. PROCEDURE
 - 6.1. Blank test
 - 6.2. Determination
 - 6.2.1. Dry a flask (5.3) (together with, if required, some anti-bumping...
 - 6.2.2. Stir the prepared sample and immediately weigh, to the nearest...
 - 6.2.3. Add 1,5 ml ammonia (25 %) (4.1) or a corresponding volume...
 - 6.2.4. Add 10 ml ethanol (4.2) and mix the liquids gently...
 - 6.2.5. Add 25 ml diethyl ether (4.3). Cool under running water....
 - 6.2.6. Remove the stopper carefully and add 25 ml light petroleum...
 - 6.2.7. Allow the apparatus to stand until the upper liquid layer...
 - 6.2.8. Remove the stopper, rinse it and the inside of the...
 - 6.2.9. Rinse the outside and the inside of the neck of...
 - 6.2.10. Make a second extraction by repeating the procedure of 6.2.5...

- 6.2.11. Make a third extraction by repeating the procedure of 6.2.10...
 - 6.2.12. Carefully evaporate or distil off as much solvent (including the...
 - 6.2.13. When there is no appreciable odour of solvent place the...
 - 6.2.14. Remove the flask from the oven, allow to cool to...
 - 6.2.15. Repeat 6.2.13 and 6.2.14 for heating periods of 30 to...
 - 6.2.16. Add 15 to 25 ml light petroleum in order to...
 - 6.2.16.1 If the extracted matter is wholly soluble in the light...
 - 6.2.16.2 If any insoluble matter is present, or in case of...
 - 7. EXPRESSION OF RESULTS
 - 7.1. Calculation
 - 7.2. Repeatability
- METHOD 4: DETERMINATION OF FAT CONTENT IN DRIED MILKS (RÖSE-GOTTLIEB...
- 1. SCOPE AND FIELD AND APPLICATION
 - 2. DEFINITION
 - 3. PRINCIPLE
 - 4. REAGENTS
 - 4.1. Ammonia solution, approximately 25 % (m/m) NH₃ (density at 20 oC approximately...
 - 4.2. Ethanol, 96 ± 2 % (v/v) or, if not available, ethanol...
 - 4.3. Diethyl ether, peroxide-free
 - 4.4. Light petroleum (petroleum ether), with any boiling range between 30...
 - 4.5. Mixed solvent, prepared shortly before use by mixing equal volumes...
 - 5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Suitable extraction tubes or flasks, provided with ground glass stoppers...
 - 5.3. Flasks, thin-walled, flat-bottomed, of 150 to 250 ml capacity.
 - 5.4. Atmospheric pressure drying oven, well ventilated and thermostatically controlled (adjusted...
 - 5.5. Anti-bumping granules, fat-free, non porous, non friable in use, e.g....
 - 5.6. Waterbath, at 60 to 70 oC.
 - 5.7. Siphon to fit extraction tubes.
 - 5.8. Centrifuge (optional).
 - 6. PROCEDURE
 - 6.1. Blank test
 - 6.2. Determination
 - 6.2.1. Dry the flask (5.3) together with, if required, some anti-bumping...
 - 6.2.2. Accurately weigh, to the nearest 1 mg, directly in, or...
 - 6.2.3. Add 1.5 ml ammonia (25 %) (4.1) or a corresponding volume...
 - 6.2.4. Add 10 ml ethanol (4.2) and mix the liquids gently...
 - 6.2.5. Add 25 ml diethyl ether (4.3). Cool in running water...
 - 6.2.6. Remove the stopper carefully and add 25 ml light petroleum...
 - 6.2.7. Allow the apparatus to stand until the upper liquid layer...
 - 6.2.8. Remove the stopper, rinse it and the inside of the...
 - 6.2.9. Rinse the outside and the inside of the neck of...
 - 6.2.10. Make a second extraction by repeating the procedure of 6.2.5...
 - 6.2.11. Make a third extraction by repeating the procedure of 6.2.10...

- 6.2.12. Carefully evaporate or distil off as much solvent (including the...
- 6.2.13. When there is no appreciable odour of solvent, place the...
- 6.2.14. Remove the flask from the oven, allow to cool to...
- 6.2.15. Repeat 6.2.13 and 6.2.14 for heating periods of 30 to...
- 6.2.16. Add 15 to 25 ml light petroleum in order to...
- 6.2.16.1 If the extracted matter is wholly soluble in the light...
- 6.2.16.2 If any insoluble matter is present, or in case of...
- 7. EXPRESSION OF RESULTS
 - 7.1. Calculation
 - 7.2. Repeatability

METHOD 5: DETERMINATION OF SUCROSE CONTENT (POLARIMETERIC METHOD)

- 1. SCOPE AND FIELD OF APPLICATION
- 2. DEFINITION
- 3. PRINCIPLE
- 4. REAGENTS
 - 4.1. Zinc acetate solution, 1 M: dissolve 21,9 g crystallized zinc...
 - 4.2. Potassium hexacyanoferrate (II) solution, 0,25 M: dissolve 10,6 g crystallized...
 - 4.3. Hydrochloric acid solution, $6,35 \pm 0,2$ M (20 to 22 %)...
 - 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.
 - 5.3. Polarimeter or saccharimeter:
 - 5.4. Water bath, regulated at $60 \text{ }^{\circ}\text{C} \pm 1 \text{ }^{\circ}\text{C}$.
- 6. PROCEDURE
 - 6.1. Control determination
 - 6.2. Determination
 - 6.2.1. Weigh to within 10 mg, approximately 40 g of the...
 - 6.2.2. Transfer the mixture quantitatively to a 200 ml measuring flask,...
 - 6.2.3. Add 5 ml of the dilute ammonia solution (4.4). Mix...
 - 6.2.4. Neutralize the ammonia by adding an equivalent quantity of the...
 - 6.2.5. Add, with gently mixing by rotating the tilted flask, 12.5...
 - 6.2.6. Add 12.5 ml of potassium hexacyanoferrate (II) solution (4.2) in...
 - 6.2.7. Bring the contents of the flask to $20 \text{ }^{\circ}\text{C}$ and make...
 - 6.2.8. Close the flask with a dry stopper and mix thoroughly...
 - 6.2.9. Allow to stand for a few minutes and then filter...
 - 6.2.10. Direct polarization: determine the optical rotation of the filtrate at...
 - 6.2.11. Inversion: pipette 40 ml of the filtrate obtained above into...
 - 6.2.12. Invert polarization
- 7. EXPRESSION OF RESULTS
 - 7.1. Method of calculation
 - 7.2. Values of the inversion factor Q
 - 7.3. Repeatability

METHOD 6: DETERMINATION OF LACTIC ACID AND LACTATES CONTENT

1. SCOPE AND FIELD OF APPLICATION
2. DEFINITION
3. PRINCIPLE
4. REAGENTS
 - 4.1. Copper (II) sulphate solution: dissolve 250 g of copper (II)...
 - 4.2. Calcium hydroxide suspension.
 - 4.2.1. Grind 300 g of calcium hydroxide (Ca(OH)₂) in a mortar...
 - 4.2.2. Calcium hydroxide suspension: grind 300 g of calcium hydroxide (Ca(OH)₂)...
 - 4.3. Sulphuric acid — copper (II) sulphate solution: Add to 300...
 - 4.4. p-hydroxydiphenyl (C₆H₅C₆H₄OH) solution: dissolve, by shaking and by heating slightly...
 - 4.5. Lactic acid standard solution: dissolve, shortly before use, 0,1067 g...
 - 4.6. Standard reconstituted milk: analyse in advance several samples of high...
5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Spectrophotometer suitable for readings at a wavelength of 570 nm....
 - 5.3. Waterbath at 30 oC ± 2 oC.
 - 5.4. Mortar and pestle.
 - 5.5. Filter paper (Schleicher and Schull 595, Whatman 1 or equivalent)....
 - 5.6. Test tubes, pyrex or equivalent (dimensions 25 x 150 mm)....
6. PROCEDURE
 - 6.1. Blank test
 - 6.2. Determination
 - 6.2.1. Determine the solids-non-fat content (a) g of the sample by...
 - 6.2.2. Weigh 1000 a-10 g of the sample to the nearest...
 - 6.2.3. Pipette 5 ml of the solution obtained into a 50...
 - 6.2.4. Add slowly while shaking, 5 ml of the copper (II)...
 - 6.2.5. Add slowly while shaking, 5 ml of the calcium hydroxide...
 - 6.2.6. Dilute to 50 ml with water, shake vigorously, allow to...
 - 6.2.7. Pipette 1 ml of the filtrate into a test tube...
 - 6.2.8. Add to the tube by means of a burette or...
 - 6.2.9. Heat in the boiling water bath for five minutes. Cool...
 - 6.2.10. Add two drops of p-hydroxydiphenyl reagent (4.4) and shake vigorously...
 - 6.2.11. Place the tube in the boiling waterbath for 90 seconds....
 - 6.2.12. Measure the optical density against the blank test (6.1) within...
 - 6.2.13. If the optical density exceeds that of the highest point...
 - 6.3. Preparation of the standard
 - 6.3.1. Pipette 5 ml of the reconstituted milk (4.6) into five...
 - 6.3.2. Dilute with water to about 30 ml and treat as...
 - 6.3.3. Measure the optical densities of the standards (6.3.1) against the...
7. EXPRESSION OF RESULTS
 - 7.1. Method of calculation
 - 7.2. Repeatability

METHOD 7: DETERMINATION OF PHOSPHATASE ACTIVITY (MODIFIED SANDERS AND SAGER...

1. SCOPE AND FIELD OF APPLICATION
2. DEFINITION

3. PRINCIPLE
4. REAGENTS
 - 4.1. Solution A
 - 4.2. Solution B:
 - 4.3. Solution C
 - 4.3.1. Dissolve 0,5 g of disodiumphenylphosphate ($\text{Na}_2\text{C}_6\text{H}_5\text{PO}_4 \cdot 2\text{H}_2\text{O}$) in 4,5 ml of...
 - 4.3.2. Pipette 1 ml of this solution into a 100 ml...
 - 4.4. Solution D
 - 4.5. Solution E
 - 4.6. Colour dilution buffer
 - 4.7. Copper sulphate solution
 - 4.8. Phenol standard solution
 - 4.9. Boiled distilled water.
 - 4.10. n-Butanol.
5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Waterbath, thermostatically controlled at $37 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$.
 - 5.3. Spectrophotometer suitable for readings at a wavelength of 610 nm....
 - 5.4. Filter paper (Schleicher and Schull 597, Whatman 42 or equivalent...
 - 5.5. Waterbath, boiling.
 - 5.6. Aluminium foil.
6. PROCEDURE
 - 6.1. Preparation of the sample
 - 6.1.1. Weigh, to within 0.1 g, 10 g of the sample...
 - 6.2. Determination
 - 6.2.1. Introduce in each of two test tubes 1 ml of...
 - 6.2.2. Heat one of the tubes in boiling water for two...
 - 6.2.3. Add 10 ml of Solution C (4.3.2). Mix and place...
 - 6.2.4. Incubate for 60 minutes in the waterbath shaking periodically.
 - 6.2.5. Transfer the tubes immediately to a boiling waterbath (5.5) and...
 - 6.2.6. Add 1 ml of Solution D (4.4), mix and filter...
 - 6.2.7. Put 5 ml of each filtrate into test tubes, add...
 - 6.2.8. Allow the colour to develop at room temperature for 30...
 - 6.2.9. Measure the optical density of the sample solution, against the...
 - 6.2.10. Repeat the determination if the optical density of the solution...
7. PREPARATION OF THE STANDARD CURVE
 - 7.1. Pipette into four 100 ml volumetric flasks, 1, 3, 5...
 - 7.2. Pipette 1 ml of water or 1 ml of each...
 - 7.3. Pipette successively into each test tube 1 ml of the...
 - 7.4. Leave the test tubes for 30 minutes at room temperature...
 - 7.5. Measure the absorbance of the solutions in each of the...
 - 7.6. Prepare the standard curve by plotting the absorbance values against...
8. EXPRESSION OF THE RESULTS
 - 8.1. Calculation
 - 8.1.1. Convert the figures as determined under 6.2.9 to μg of...
 - 8.1.2. Calculate the phosphatase activity expressed as μg of phenol per...
 - 8.1.3. If it was necessary to dilute as indicated under 6.2.10...
 - 8.2. Repeatability

METHOD 8: DETERMINATION OF PHOSPHATASE ACTIVITY (ASCHAFFENBURG AND MÜLLEN PROCEDURE)...

1. SCOPE AND FIELD OF APPLICATION
2. DEFINITION
3. PRINCIPLE
4. REAGENTS
 - 4.1. Sodium carbonate-bicarbonate buffer solution.
 - 4.2. Buffer substrate.
 - 4.3. Clarification solutions.
 - 4.3.1. Zinc sulphate solution.
 - 4.3.2. Potassium hexacyanoferrate (II) solution.
5. APPARATUS
 - 5.1. Analytical balance.
 - 5.2. Waterbath, thermostatically controlled at $37\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.
 - 5.3. Comparator, with special disc containing standard colour glasses calibrated in...
6. PROCEDURE
 - 6.1. Preparation of sample
 - 6.2. Determination
 - 6.2.1. Pipette 15 ml of buffer substrate (4.2) into a clean,...
 - 6.2.2. At the same time, place in the water bath a...
 - 6.2.3. After two hours remove both tubes from the water bath,...
 - 6.2.4. Transfer the filtrate to a 25 mm cell and compare...
7. EXPRESSION OF RESULTS
 - 7.1. Calculation
 - 7.2. Repeatability

Status: EU Directives are being published on this site to aid cross referencing from UK legislation. After IP completion day (31 December 2020 11pm) no further amendments will be applied to this version.

(1) [OJ No L 24, 30. 1. 1976, p. 49.](#)