

Council Decision of 14 November 1992 laying down methods for the analysis and testing of heat-treated milk for direct human consumption (92/608/EEC)

- Article 1 The methods for analysing and testing heat-treated milk shall be...  
Article 2 The implementation of the reference methods for analysis and testing, ...  
Article 3 The methods referred to in Article 1 are set out...  
Article 4 This Decision is addressed to the Member States.  
Signature

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ANNEX I

- I. GENERAL PROVISIONS
1. INTRODUCTION
  2. REAGENTS
    - 2.1. Water
      - 2.1.1. Wherever mention is made to water for solution, dilution or...
      - 2.1.2. Wherever reference is made to 'solution' or 'dilution' without further...
    - 2.2. Chemicals
  3. EQUIPMENT
    - 3.1. Lists of equipment
    - 3.2. Analytical balance
  4. EXPRESSION OF RESULTS
    - 4.1. Results
    - 4.2. Calculation of percentage
  5. PRECISION CRITERIA: REPEATABILITY AND REPRODUCIBILITY
    - 5.1. The precision criteria given in each method is defined as...
      - 5.1.1. Repeatability (r) is the value below which the absolute difference...
      - 5.1.2. Reproducibility (R) is the value below which the absolute difference...
      - 5.1.3. Unless otherwise specified for each individual method the values for...
      - 5.1.4. The necessary collaborative trials and studies should be planned and...
  6. TEST REPORT
- II. SAMPLING OF HEAT-TREATED MILK
1. SCOPE AND FIELD OF APPLICATION
  2. GENERAL
  3. SAMPLING EQUIPMENT
    - 3.1. General
  4. SAMPLING TECHNIQUE
    - 4.1. General
    - 4.2. Manual sampling

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- 4.2.1. Sampling a divided bulk
- 4.2.2. Sampling from large vessels — Storage, rail and road tanks...
  - 4.2.2.1. Mix the milk by an appropriate procedure, before sampling.
  - 4.2.2.2. Mixing of the contents of large vessels or of storage,...
- 4.3. Sampling of heat-treated milk for direct consumption in retail-packings
- 5. IDENTIFICATIONS OF THE SAMPLE
- 6. PRESERVATION, TRANSPORT AND STORAGE OF SAMPLES

## ANEX II

- I. DETERMINATION OF TOTAL SOLIDS CONTENT
  - 1. SCOPE AND FIELD OF APPLICATION
  - 2. DEFINITION
  - 3. PRINCIPLE
  - 4. APPARATUS AND GLASSWARE
  - 5. PREPARATION OF THE TEST SAMPLE
  - 6. PROCEDURE
    - 6.1. Preparation of the dish
    - 6.2. Test portion
    - 6.3. Determination
      - 6.3.1. Pre-dry the dish for 30 minutes by heating it on...
      - 6.3.2. Heat the dish, with its lid alongside, in the oven...
      - 6.3.3. Allow to cool in the desiccator (4.2.) to room temperature...
      - 6.3.4. Heat the dish again, with its lid alongside, in the...
      - 6.3.5. Repeat the operations described in 6.3.4. until the difference in...
  - 7. EXPRESSION OF RESULTS
    - 7.1. Calculation and formula
    - 7.2. Precision
      - 7.2.1. Repeatability (r): 0,10 g of total solids per 100 g...
      - 7.2.2. Reproducibility (R): 0,20 g of total solids per 100 g...
- II. DETERMINATION OF FAT CONTENT
  - 1. SCOPE AND FIELD OF APPLICATION
  - 2. DEFINITION
  - 3. PRINCIPLE
  - 4. REAGENTS
    - 4.1. Ammonia solution, containing approximately 25 % (m/m) of NH<sub>3</sub>. A...
    - 4.2. Ethanol, at least 94% (v/v). Ethanol denatured by methanol may...
    - 4.3. Congo red or Cresol red solution
    - 4.4. Diethylether, free from peroxides not containing more than 2 mg/kg...
    - 4.5. Light petroleum, having any boiling range between 30 and 60...
    - 4.6. Mixed solvent, prepared shortly before use by mixing equal volumes...
  - 5. APPARATUS AND GLASSWARE
    - 5.1. Analytical balance
    - 5.2. Centrifuge, in which the fat-extraction flasks or tubes (5.6.) can...
    - 5.3. Distillation or evaporation apparatus, to permit the solvents and ethanol...
    - 5.4. Oven, electrically heated, with ventilation port(s) fully open, capable of...

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- 5.5. Water bath, capable of being maintained at a temperature of...
  - 5.6. Mojonnier-type fat extraction flasks
  - 5.7. Rack, to hold the fat-extraction flasks (or tubes) (see 5.6.)....
  - 5.8. Wash bottle, suitable for use with the mixed solvent (4.6.)....
  - 5.9. Fat-collecting vessels, for example boiling flasks (flat-bottom), or Erlenmeyer flasks...
  - 5.10. Boiling aids, fat-free, of non-porous porcelain or silicon carbide or...
  - 5.11. Measuring cylinders, of capacities 5 and 25 ml.
  - 5.12. Pipettes, graduated, of capacity 10 ml.
  - 5.13. Tongs, made of metal, suitable for holding flasks, beakers or...
  6. PROCEDURE
    - 6.1. Preparations of the test sample
    - 6.2. Test portion
    - 6.3. Blank test
    - 6.4. Preparation of fat-collecting vessel
    - 6.5. Determination
      - 6.5.1. Add 2 ml of the ammonia solution (4.1.) or an...
      - 6.5.2. Add 10 ml of the ethanol (4.2.) and mix gently...
      - 6.5.3. Add 25 ml of diethyl ether (4.4.), close the flask...
      - 6.5.4. Add 25 ml of light petroleum (4.5.), close the flask...
      - 6.5.5. Centrifuge the closed flask for one to five minutes at...
      - 6.5.6. Carefully remove the cork or stopper and rinse it and...
      - 6.5.7. Holding the extraction flask by the small bulb, carefully decant...
      - 6.5.8. Rinse the outside of the neck of the extraction flask...
      - 6.5.9. Add 5 ml of the ethanol (4.2.) to the contents...
      - 6.5.10. Carry out a second extraction by repeating the operations, described...
      - 6.5.11. Carry out a third extraction by further repeating the operations...
      - 6.5.12. Remove the solvents (including ethanol) as completely as possible from...
      - 6.5.13. Heat the fat-collecting vessel (with the flask placed on its...
      - 6.5.14. Repeat the operations described in 6.5.13. until the mass of...
      - 6.5.15. Add 25 ml of light petroleum to the fat-collecting vessel...
      - 6.5.16. If the extracted matter is not wholly soluble in the...
  7. EXPRESSION OF RESULTS
    - 7.1. Calculation and formula
    - 7.2. Precision
      - 7.2.1. Repeatability (r):
      - 7.2.2. Reproducibility (R):
- III. DETERMINATION OF TOTAL NON-FAT SOLIDS
1. SCOPE AND FIELD OF APPLICATION
  2. DEFINITION AND CALCULATION
- IV. DETERMINATION OF TOTAL NITROGEN CONTENT
1. SCOPE AND FIELD OF APPLICATION
  2. DEFINITION
  3. PRINCIPLE
  4. REAGENTS
    - 4.1. Potassium sulphate (K<sub>2</sub>SO<sub>4</sub>).
    - 4.2. Copper sulphate solution. Dissolve 5,0 g of copper (II) sulphate...

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- 4.3. Sulphuric acid, at least 98,0% (m/m) H<sub>2</sub>SO<sub>4</sub>.
- 4.4. Sodium hydroxide solution, 47% (m/m) 704 g NaOH/l (20 °C)....
- 4.5. Boric acid solution. Dissolve 40 g of boric acid (H<sub>3</sub>BO<sub>3</sub>)...
- 4.6. Indicator solution. Dissolve 0,01 g methyl red, 0,02 g bromothymol...
- 4.7. Volumetric solution
- 4.8. Nitrogen-free sucrose.
- 4.9. Ammonium salt, pure, such as ammonium oxalate (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O or ammonium...
- 4.10. Tryptophan (C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>), phenacetin (C<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>CONH<sub>2</sub>) or lysine mono- or di-hydrochloride (C<sub>6</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>...
5. APPARATUS AND GLASSWARE
6. PROCEDURE
  - 6.1. To the Kjeldahl flask (5.1.) add boiling aid (5.2.) (eg....
  - 6.2. Heat each Kjeldahl flask on the digestion apparatus (5.5.), very...
  - 6.3. When the Kjeldahl flasks are cool, add 300 ml of...
  - 6.4. Immediately connect each Kjeldahl flask to a distillation apparatus (5.6.)....
  - 6.5. Titrate each distillate with standard volumetric solution (4.7.) until the...
  - 6.6. Carry out a blank test according to 6.1. to 6.5....
  - 6.7. Regularly check the accuracy of the procedure by using two...
    - 6.7.1. Check that no loss of nitrogen occurs as a result...
    - 6.7.2. Check that the digestion procedure is sufficient to release all...
7. SAFETY PRECAUTIONS
8. EXPRESSION OF RESULTS
  - 8.1. Calculation and formula:
  - 8.2. Precision
    - 8.2.1. Repeatability (r): 0,007 g per 100 g.
    - 8.2.2. Reproducibility (R): 0,015 g per 100 g.
9. MODIFIED PROCEDURES
  - 9.1. Use a block digestion apparatus fitted with cylindrical flasks, instead...
  - 9.2. Use of steam distillation instead of direct heating of the...
  - 9.3. A test portion of 1 g of milk (semi-macro Kjeldahl)...
- V. DETERMINATION OF PROTEIN CONTENT
  1. SCOPE AND FIELD OF APPLICATION
  2. DEFINITION
  3. CALCULATION
- VI. DETERMINATION OF SPECIFIC MASS
  1. SCOPE AND FIELD OF APPLICATION
  2. DEFINITION
  3. PRINCIPLE
  4. APPARATUS AND GLASSWARE
    - 4.1. Hydrometer
    - 4.2. Cylinders (glass or stainless steel).
    - 4.3. Water bath regulated at 20 ± 0,1 °C.
    - 4.4. Water bath regulated at 40 ± 2 °C.
    - 4.5. Thermometer, graduated to 0,5 °C.
  5. PROCEDURE
    - 5.1. Mix the sample by inversion to disperse the fat and...
    - 5.2. Mix the sample thoroughly by careful inversion to avoid inclusion...
    - 5.3. When the hydrometer reaches equilibrium read the graduation at the...

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- 5.4. Immediately after taking the hydrometer reading introduce the thermometer (4.5.)...
6. TEMPERATURE CORRECTION
  - 6.1. If the temperature of the milk sample is not exactly...
7. EXPRESSION OF RESULTS
8. PRECISION
  - 8.1. Repeatability (r): 0,0003 g/ml.
  - 8.2. Reproducibility (R): 0,0015 g/ml.

Appendix  
(to Annex II)

ALTERNATIVE PROCEDURE USING FAT-EXTRACTION TUBES WITH SIPHON OR WASH-BOTTLE FITTINGS...

- A.1. PROCEDURE
  - A.1.1. Preparation of the test sample
  - A.1.2. Test portion
  - A.1.3. Blank test
  - A.1.4. Preparation of fat-collecting vessel
  - A.1.5. Determination
    - A.1.5.1. Add 2 ml of the ammonia solution (4.1.), or an...
    - A.1.5.2. Add 10 ml of the ethanol (4.2.) and mix gently...
    - A.1.5.3. Add 25 ml of diethyl ether (4.4.), close the tube...
    - A.1.5.4. Add 25 ml of light petroleum (4.5.), close the tube...
    - A.1.5.5. Centrifuge the closed tube for one to five minutes at...
    - A.1.5.6. Carefully remove the cork or stopper and rinse it and...
    - A.1.5.7. Insert a siphon fitting or a wash-bottle fitting into the...
    - A.1.5.8. Loosen the fitting from the neck of the tube, slightly...
    - A.1.5.9. Again loosen the fitting from the neck, slightly raise the...
    - A.1.5.10. Carry out the second extraction by repeating the operations described...
    - A.1.5.11. Carry out a third extraction by again repeating the operations...
    - A.1.5.12. Proceed as described in 6.5.12. to 6.5.16.

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- (1) [OJ No L 226, 24. 8. 1985, p. 13](#), as last amended by Directive 89/662/EEC ([OJ No L 395, 30. 12. 1989, p. 13](#)).

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