
Changes to legislation: There are currently no known outstanding effects for the 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), ANNEX I. (See end of Document for details)

ANNEX I

I. GENERAL PROVISIONS

1. INTRODUCTION

General provisions with respect to reagents, equipment, expression of results, precision and test reports are described. Competent authorities of Member States and enforcement laboratories charged with the sampling and testing of milk must respect these provisions.

2. REAGENTS

2.1. Water

2.1.1. Wherever mention is made to water for solution, dilution or rinsing purposes, distilled water, deionized water or demineralized water of at least equivalent purity, shall be used unless otherwise specified.

2.1.2. Wherever reference is made to 'solution' or 'dilution' without further indication, 'solution in water' or 'dilution with water' is meant.

2.2. Chemicals

All chemicals used shall be of recognized analytical quality unless otherwise specified.

3. EQUIPMENT

3.1. Lists of equipment

The lists of equipment given in the different reference methods contain only those items with a specialized use and items to a particular specification.

3.2. Analytical balance

'Analytical balance' means a balance capable of weighing at 0,1 mg.

4. EXPRESSION OF RESULTS

4.1. Results

Unless otherwise specified, the results stated in the test report (6) shall be the mean arithmetic value obtained from two tests which satisfy the repeatability-criterion (5.1.1.) stated for that method. If the repeatability-criterion is not satisfied, the test must be repeated, if possible, or the result declared invalid.

4.2. Calculation of percentage

Except when otherwise specified, the result shall be calculated as a percentage by mass of the sample.

5. PRECISION CRITERIA: REPEATABILITY AND REPRODUCIBILITY

5.1. The precision criteria given in each method is defined as follows:

5.1.1. *Repeatability (r)* is the value below which the absolute difference between two single test results obtained with the same procedure on identical test material, under the same conditions (same operator, same apparatus, same laboratory, and a short interval of time) lies.

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- 5.1.2. *Reproducibility (R)* is the value below which the absolute difference between two single test results obtained with the same procedure on identical test material, under different conditions (different operators, different apparatus, different laboratories and/or different time) lies.
- 5.1.3. Unless otherwise specified for each individual method the values for the repeatability- and reproducibility-criteria of each procedure represent the 95% confidence level intervals as defined by ISO 5725: 2'ed. 1986.
- 5.1.4. The necessary collaborative trials and studies should be planned and conducted in accordance with international guidelines.
6. TEST REPORT

The test report shall specify the method of analysis used as well as the results obtained. In addition, it shall give any details of procedure used not specified in the method of analysis or which are optional, as well as any other circumstances that may have influenced the results obtained. The test report shall give all the information necessary for the complete identification of the sample.

II. SAMPLING OF HEAT-TREATED MILK

1. SCOPE AND FIELD OF APPLICATION

This procedure specifies the reference method of sampling, transport and storage of samples of heat treated milk.

2. GENERAL

Sampling of heat-treated milk in tanks etc., shall be carried out by a skilled operator who has had suitable training before undertaking the sampling of milk.

If they consider it appropriate, the competent authorities or testing laboratory shall instruct sampling personnel in sampling techniques to ensure that the sample is representative of, and in conformity with, the entire batch.

If they consider it appropriate, the competent authorities or testing laboratory shall instruct sampling personnel on marking the sample to ensure the unambiguous identity of the sample.

3. SAMPLING EQUIPMENT

3.1. General

Sampling equipment shall be made of stainless steel, or other suitable material of adequate strength and of a construction suitable for the intended purpose (mixing, sampling etc.). Plungers and agitators for mixing liquids in containers shall have a sufficient area to produce adequate mixing of the product, but without causing the development of a rancid flavour. Dippers must have a solid handle of sufficient length to enable a sample at any depth of the container to be obtained. The capacity of the dipper shall be not less than 50 ml.

Sample containers and closures should be of glass, suitable metals or plastics.

The materials of which sampling equipment (including containers and closures) is constructed must not cause any change in the sample which could affect the results of the examinations. All surfaces of sampling equipment and sample containers shall be clean and dry, smooth and free from crevices, and corners shall be rounded.

4. SAMPLING TECHNIQUE

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4.1. General

Irrespective of the tests to be performed, the milk shall be thoroughly mixed prior to sampling, by either manual or mechanical means.

The sample shall be taken immediately after mixing while the milk is still agitated.

The volume of the sample shall be adequate to the testing requirements. The capacity of the sample containers used shall be such that they are filled almost completely by the sample, thus allowing proper mixing of the contents before testing, but avoiding churning during transport.

4.2. Manual sampling

4.2.1. *Sampling a divided bulk*

Where the quantity of milk to be sampled is in more than one container, take a representative quantity from each container and note the quantity of milk to which each sample relates. Unless the samples from each container are to be tested individually, mix portions of these representative quantities in amounts which are proportional to the quantity in the container from which each sample was taken. Take sample(s) from these bulked proportionate amounts after mixing.

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.

To mix the contents of large vessels or of storage, rail or road tanks, the use of mechanical agitation is advised (4.2.2.2.).

The extent of mixing shall be appropriate to the period of time over which the milk has been at rest. The efficiency of the procedure of mixing applied in any particular circumstances shall be demonstrated as being adequate for the purposes of the analysis envisaged; the criterion of mixing efficiency particularly influences the similarity between analytical results from samples taken either from different parts of the consignments, or from the outlet of the tank at intervals during discharge. A procedure of mixing milk (untreated milk or whole milk) shall be considered efficient if the difference in fat content between two samples, taken under these conditions, is less than 0,1 %.

In a large vessel with a bottom discharge outlet there may be, at the discharge point, a small quantity of milk which is not representative of the whole contents even after mixing. Therefore samples should preferably be taken through a manhole. If samples are taken from the discharge outlet, run off sufficient milk to ensure that the samples are representative of the whole.

4.2.2.2. Mixing of the contents of large vessels or of storage, rail or road tanks can be carried out:

- by a mechanical agitator built into the tanks and driven by an electric motor;
- by a propellor or agitator driven by an electric motor and placed on the manhole with the agitator suspended in the milk;
- in the case of rail or road tankers by recirculation of the milk through the transfer hose attached to the tanker unloading pumps and inserted through the manhole;
- by clean filtered compressed air. In this case, minimal air pressure and volume should be used to prevent the development of rancid flavour.

4.3. Sampling of heat-treated milk for direct consumption in retail-packings

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Samples of heat-treated milk for direct consumption in retail packages are to be the complete sealed package. If possible, the samples must be taken from the packaging machine or cold room in the treatment establishment as soon as possible after processing (for pasteurized milk on the same day as processing).

The samples are taken from each type of heat-treated milk (pasteurized, UHT-treated and sterilized) in numbers corresponding to the examinations which will be made and in accordance with instructions laid down by the testing laboratory or other competent authority.

5. IDENTIFICATIONS OF THE SAMPLE

The sample shall be marked with an identification code so that it can be readily identified using instructions given by the testing laboratory or competent authority.

6. PRESERVATION, TRANSPORT AND STORAGE OF SAMPLES

In accordance with the competent national authority, instructions concerning the conditions of preservation (chemical, temperature), transport, storage and time between sampling and analysis of milk shall be prepared by the testing laboratory according to the type of milk and the procedure of analysis to be used.

In the instruction the following points shall be included:

- During transport and storage, precautions shall be taken to prevent exposure to contaminating odours and to direct sunlight. If the container used for samples is transparent, it shall be stored in a dark place.

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