SLOVENSKI STANDARD
SIST EN ISO 1211:1998

01-avgust-1998

Mleko - Določevanje maščobe - Gravimetrijska metoda (Referenčna metoda) (ISO 1211:1984)

Milk - Determination of fat content - Gravimetric method (Reference method) (ISO 1211:1984)

Milch - Bestimmung des Fettgehaltes - Gravimetrisches Verfahren (Referenzverfahren) (ISO 1211:1984)

Lait - Détermination de la teneur en matiere grasse - Méthode gravimétrique (Méthode de référence) (ISO 1211:1984)

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ICS:

67.100.10	Mleko in predelani mlečni proizvodi	Milk and processed milk products

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English version

Milk - Determination of fat content - Gravimetric method (Reference method) (ISO 1211:1984)

Lait - Détermination de la teneur en matière grasse - Méthode gravimétrique (Méthode de références) (ISO 1211:1984)

Milch - Bestimmung des Fettgehaltes - Gravimetrisches Verfahren (Referenzverfahren) (ISO 1211:1984)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

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Foreword

The text of the International Standard from ISO/TC 34 "Agricultural food products" of the International Organization for Standardization (ISO) has been taken over as a European Standard by the Technical Committee CEN/TC 302 "Milk and milk products - Methods of sampling and analysis".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 1995, and conflicting national standards shall be withdrawn at the latest by December 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 1211:1984 has been approved by CEN as a European Standard without any modification.

International Standard 1211

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Milk — Determination of fat content — Gravimetric method (Reference method)

Lait — Détermination de la teneur en matière grasse — Méthode gravimétrique (Méthode de référence)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1211 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

It cancels and replaces ISO Recommendation R 1211-1970, of which it constitutes a technical revision.

NOTE — The method specified in this International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, USA). The text as approved by the above organizations will also be published by FAO/WHO (Code of principles concerning milk and milk products and associated standards), by the IDF and by the AOAC (Official Methods of Analysis).

Printed in Switzerland

Milk — Determination of fat content — Gravimetric method (Reference method)

0 Introduction

This International Standard constitutes a revision of ISO/R 1211. Attempts to produce a single International Standard specifying the use of the Röse-Gottlieb method applicable to all milk products have been found impracticable for the time being. Therefore, it has been decided to revise and harmonize the existing standardized methods for individual products or groups of products and to standardize similar methods for those products for which methods had not been prepared previously.

The main modifications as compared to ISO/R 1211 are :

a) preference is given to the use of Mojonnier-type fat-extraction flasks and the use of a centrifuge to separate the solvent layers;

b) addition of ethanol before the second extraction;

c) emphasis is placed on the necessity of cooling the fat-collecting vessels to ambient temperature before performing weighings.

The use of a centrifuge enables rapid and complete separation of the solvent layers and minimizes the need for redissolution of the extracted fat or a repetition of the determination.

The addition of ethanol before the second extraction has been specified to reduce the risk of formation of a viscous or gelified aqueous layer, especially in the case of products containing sucrose (for example sweetened condensed milk, edible ices and, to a lesser extent, milk powder). It has been found that this addition distinctly improves the precision of the method, also for milk.

Emphasis has been placed on the necessity of cooling fat-collecting vessels to ambient temperature before weighing, as errors from this source of the order of 0,01 % fat per degree Celsius have been reported for liquid milk. The use of a desiccator is therefore not recommended. The use of an empty control vessel will compensate for these errors to some extent. It has been found, however, that the use of such a vessel together with a blank test on 10 ml of water carried out with the determination is complicated and produces no improvement in precision.

An empty control vessel is, however, necessary, and is therefore specified when performing the blank test to check the reagents in order to avoid a false impression of the presence or absence of non-volatile matter.

1 Scope and field of application

This International Standard specifies the reference method for the determination of the fat content of raw and processed liquid milk, partly skimmed milk and skimmed milk in which no appreciable separation or splitting of fat has occurred (see the note to 8.1).

NOTE — When greater accuracy is required for skimmed milk, for instance to establish the operating efficiency of cream separators, the special method for skimmed products, specified in ISO 7208, *Skimmed milk, whey and buttermilk — Determination of fat content — Gravimetric method (Reference method)*, should be used.

2 References

ISO 707, *Milk and milk products — Methods of sampling.*

ISO 3889, *Milk and milk products — Determination of fat content — Mojonnier-type fat extraction flasks.*

3 Definition

fat content of milk : All the substances determined by the method specified in this International Standard.

It is expressed as a percentage by mass.

4 Principle

Extraction of an ammoniacal ethanolic solution of a test portion with diethyl ether and light petroleum, removal of the solvents by distillation or evaporation, and determination of the mass of the substances extracted which are soluble in light petroleum. (This is usually known as the Röse-Gottlieb principle.)

5 Reagents

All reagents shall be of recognized analytical grade and shall leave no appreciable residue when the determination is carried out by the method specified. The water used shall be distilled water or water of at least equivalent purity.

To test the quality of the reagents, carry out a blank test as specified in 8.3. Use an empty fat-collecting vessel, prepared as specified in 8.4, for mass control purposes. The reagents shall leave no residue greater than 0,5 mg (see 10.1).

If the residue of the complete reagent blank test is greater than 0,5 mg, determine the residue of the solvents separately by distilling 100 ml of the diethyl ether and light petroleum respectively. Use an empty control vessel to obtain the real mass of residue which shall not exceed 0,5 mg.

Replace unsatisfactory reagents or solvents, or redistil solvents.

5.1 Ammonia solution, containing approximately 25 % (m/m) of NH_3, $\varrho_{20} \approx 910$ g/l.

NOTE — If ammonia solution of this concentration is not available, a more concentrated solution of known concentration may be used (see 8.5.1).

5.2 Ethanol, or ethanol denatured by methanol, at least 94 % (V/V).

(See 10.5.)

5.3 Congo-red solution.

Dissolve 1 g of Congo-red in water and dilute to 100 ml.

NOTE — The use of this solution, which allows the interface between the solvent and aqueous layers to be seen more clearly, is optional (see 8.5.2). Other aqueous colour solutions may be used provided that they do not affect the result of the determination.

5.4 Diethyl ether, free from peroxides (see 10.3) and containing no or not more than 2 mg/kg of antioxidants and complying with the requirements for the blank test (see clause 5, and also 10.1 and 10.4).

5.5 Light petroleum, having any boiling range between 30 and 60 °C.

5.6 Mixed solvent, prepared shortly before use by mixing equal volumes of the diethyl ether (5.4) and the light petroleum (5.5).

6 Apparatus

WARNING — Since the determination involves the use of volatile flammable solvents, electrical apparatus employed shall be required to comply with legislation relating to the hazards in using such solvents.

Usual laboratory equipment, and in particular

6.1 Analytical balance.

6.2 Centrifuge, in which the fat-extraction flasks or tubes (6.6) can be spun at a rotational frequency of 500 to 600 min^{-1} to produce a gravitational field of 80 g to 90 g at the outer end of the flasks or tubes.

NOTE — The use of the centrifuge is optional but recommended (see 8.5.5).

6.3 Distillation or evaporation apparatus, to enable the solvents and ethanol to be distilled from the flasks or to be evaporated from beakers and dishes (see 8.5.12) at a temperature not exceeding 100 °C.

6.4 Drying oven, electrically heated, with ventilation port(s) fully open, capable of being maintained at a temperature of 102 ± 2 °C throughout the working space. The oven shall be fitted with a suitable thermometer.

6.5 Water-bath, capable of being maintained at 35 to 40 °C.

6.6 Mojonnier-type fat-extraction flasks, as specified in ISO 3889.

NOTE — It is also possible to use **fat-extraction tubes**, with **siphon or wash-bottle fittings**, but the procedure is then different and is specified in the annex.

The flasks (or tubes, see the note) shall be provided with good quality bark corks or stoppers of other material (for example silicone rubber) unaffected by the reagents used. Bark corks shall be extracted with the diethyl ether (5.4), kept in water at 60 °C or more for at least 15 min, and shall then be allowed to cool in the water so that they are saturated when used.

6.7 Rack, to hold the fat-extraction flasks (or tubes) (see 6.6).

6.8 Wash bottle, suitable for use with the mixed solvent (5.6). A plastic wash bottle shall not be used.

6.9 Fat-collecting vessels, for example boiling flasks (flat-bottomed), of capacity 125 to 250 ml, conical flasks, of capacity 250 ml, or metal dishes. If metal dishes are used, they shall preferably be of stainless steel, shall be flat-bottomed, preferably with a spout, and shall have a diameter of 80 to 100 mm and a height of approximately 50 mm.

6.10 Boiling aids, fat-free, of non-porous porcelain or silicon carbide (optional in the case of metal dishes).

6.11 Measuring cylinders, of capacities 5 and 25 ml.

6.12 Pipettes, graduated, of capacity 10 ml.

6.13 Tongs, made of metal, suitable for holding flasks, beakers or dishes.

7 Sampling

See ISO 707.

All laboratory samples shall be kept at a temperature of 3 to 6 °C from the time of sampling to the time of commencing the procedure.

8 Procedure

NOTE — The alternative procedure using fat-extraction tubes with siphon or wash-bottle fittings (see the note to 6.6) is described in the annex.

8.1 Preparation of the test sample

Adjust the temperature of the laboratory sample (clause 7) to 35 to 40 °C, by means of the water-bath (6.5) if necessary. Mix the sample thoroughly, but gently, by repeatedly inverting the sample bottle without causing frothing or churning, and cool quickly to approximately 20 °C.

Churned milk should not be cooled as it has to be weighed at 30 to 40 °C in 8.2.

NOTE — A reliable value for the fat content cannot be expected :

a) if the milk is churned;

b) when a distinct smell of free fatty acids is perceptible;

c) if during, or after, preparation of the sample, white particles are visible on the walls of the sample bottle or fat droplets float on the surface of the sample.

8.2 Test portion

Mix the test sample (8.1) by gently inverting the bottle three or four times and immediately weigh, to the nearest 1 mg, 10 to 11 g of the test sample, directly or by difference, into one of the extraction flasks (6.6).

The test portion shall be delivered as completely as possible into the lower (small) bulb of the extraction flask.

8.3 Blank test

Carry out a blank test simultaneously with the determination, using the same procedure and same reagents, but replacing the test portion by 10 ml of water (see 10.2).

8.4 Preparation of fat-collecting vessel

Dry a vessel (6.9) with a few boiling aids (6.10) in the oven (6.4) for 1 h. (See note 1.)

Allow the vessel to cool (protected from dust) to the temperature of the weighing room (glass vessel for at least 1 h, metal dish for at least 0,5 h). (See note 2.)

Using tongs (to avoid, in particular, temperature variations), place the vessel on the balance and weigh to the nearest 0,1 mg.

NOTES

1 Boiling aids are desirable to promote gentle boiling during the subsequent removal of solvents, especially in the case of glass vessels; their use is optional in the case of metal dishes.

2 The vessel should not be placed in a desiccator, to avoid insufficient cooling or unduly long cooling times.

8.5 Determination

8.5.1 Add 2 ml of the ammonia solution (5.1), or an equivalent volume of a more concentrated ammonia solution (see the note to 5.1), and mix thoroughly with the test portion in the small bulb of the flask. After the addition of the ammonia, carry out the determination without delay.

8.5.2 Add 10 ml of the ethanol (5.2) and mix gently but thoroughly by allowing the contents of the flask to flow backward and forward between the two bulbs; avoid bringing the liquid too near to the neck of the flask. If desired, add 2 drops of the Congo-red solution (5.3).

8.5.3 Add 25 ml of the diethyl ether (5.4), close the flask with a cork (see 6.6) saturated with water or with a stopper of other material (see 6.6) wetted with water, and shake the flask vigorously, but not excessively (in order to avoid the formation of persistent emulsions), for 1 min with the flask in a horizontal position and the small bulb extending upwards, periodically allowing the liquid in the large bulb to run into the small bulb. If necessary, cool the flask in running water, then carefully remove the cork or stopper and rinse it and the neck of the flask with a little of the mixed solvent (5.6) using the wash bottle (6.8) so that the rinsings run into the flask.

8.5.4 Add 25 ml of the light petroleum (5.5), close the flask with the rewetted cork or rewetted stopper (by dipping in water), and shake the flask gently for 30 s as described in 8.5.3.

8.5.5 Centrifuge the closed flask for 1 to 5 min at a rotational frequency of 500 to 600 min^{-1}. If a centrifuge is not available, allow the closed flask to stand in the rack (6.7) for at least 30 min until the supernatant layer is clear and distinctly separated from the aqueous layer. If necessary, cool the flask in running water.

8.5.6 Carefully remove the cork or stopper and rinse it and the inside of the neck of the flask with a little of the mixed solvent so that the rinsings run into the flask.

If the interface is below the bottom of the stem of the flask, raise it slightly above this level by gently adding water down the side of the flask (see figure 1) to facilitate the decantation of solvent.

NOTE — In figures 1 and 2, one of the three types of flasks as specified in ISO 3889 has been chosen, but this does not imply any preference over the other types.

8.5.7 Holding the extraction flask by the small bulb, carefully decant as much as possible of the supernatant layer into the prepared fat-collecting vessel (see 8.4) containing a few boiling aids (6.10) in the case of flasks (optional with metal dishes), avoiding decantation of any of the aqueous layer (see figure 2).

8.5.8 Rinse the outside of the neck of the extraction flask with a little of the mixed solvent, collecting the rinsings in the fat-collecting vessel and taking care that the mixed solvent does not spread over the outside of the extraction flask.