

SLOVENSKI STANDARD SIST EN ISO 1736:2001

01-februar-2001

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Dried milk and dried milk products - Determination of fat content - Gravimetric method (Reference method) (ISO 1736:2000)

Milchpulver und Trockenmilcherzeugnisse - Bestimmung des Fettgehaltes - Gravimetrisches Verfahren (Referenzverfahren) (ISO 1736:2000)

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Lait sec et produits a base de lait sec - Détermination de la teneur en matiere grasse Méthode gravimétrique (Méthode de référence) (ISO 1736:2000)

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Ta slovenski standard je istoveten z: EN ISO 1736-2001

ICS:

67.100.10 T $|^{\hat{A}} \hat{A}|^{\hat{A}}$ $|^{\hat{A}} \hat{A}|^{\hat{A}}$ Milk and processed milk products

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EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN ISO 1736

March 2000

ICS 67.100.01

English version

Dried milk and dried milk products - Determination of fat content - Gravimetric method (Reference method) (ISO 1736:2000)

Lait sec et produits à base de lait sec - Détermination de la teneur en matière grasse - Méthode gravimétrique (Méthode de référence) (ISO 1736:2000)

Milchpulver und Trockenmilcherzeugnisse - Bestimmung des Fettgehaltes - Gravimetrisches Verfahren (Referenzverfahren) (ISO 1736:2000)

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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

Page 2 EN ISO 1736:2000

Foreword

The text of the International Standard ISO 1736:2000 has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 302 "Milk and milk products - Methods of sampling and analysis", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, 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 1736:2000 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

ISO 1736

Third edition 2000-03-01

Dried milk and dried milk products — Determination of fat content — Gravimetric method (Reference method)

Lait sec et produits à base de lait sec — Détermination de la teneur en matière grasse — Méthode gravimétrique (Méthode de référence)

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Reference number ISO 1736:2000(E)

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 1736 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 5, *Milk and milk products*, in collaboration with the International Dairy Federation (IDF) and AOAC International, and will also be published by these organizations.

This third edition cancels and replaces the second edition (ISO 1736:1985), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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Dried milk and dried milk products — Determination of fat content — Gravimetric method (Reference method)

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies the reference method for the determination of the fat content of dried milk and dried milk products. The method is applicable to dried milk with a fat content of 40 % (mass fraction) or more, dried whole, dried partially skimmed, and dried skimmed milk, dried whey, dried buttermilk and dried butter serum.

NOTE When the powder contains hard lumps which do not dissolve in ammonia solution or contains free fatty acids in significant quantities, noticeable by a distinct smell, the result of the determination will be too low. With such products recourse should be made to a method using the Weibull-Berntrop principle (see ISO 8262-3).

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2 Normative reference

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The following normative document contains provisions which through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreement based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1

fat content of dried milk and dried milk products

mass fraction of substances determined by the procedure specified in this International Standard

NOTE The fat content is expressed as a mass fraction, in percent [formerly given as % (m/m)].

4 Principle

An ammoniacal ethanolic solution of a test portion is extracted with diethyl ether and light petroleum. The solvents are removed by distillation or evaporation. The mass of the substances extracted is determined.

NOTE This is usually known as the Röse-Gottlieb principle.

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5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

The reagents shall leave no appreciable residue when the determination is carried out by the method specified (see 9.2.2).

5.1 Ammonia solution, containing a mass fraction of NH₃ of approximately 25 % (ρ_{20} = 910 g/l).

NOTE If ammonia solution of this concentration is not available, a more concentrated solution of known concentration may be used (see 9.4.2).

5.2 Ethanol (C_2H_5OH), or ethanol denatured by methanol, containing a volume fraction of ethanol of at least 94 %. (See A.5.)

5.3 Congo red solution

Dissolve 1 g of Congo red in water in a 100 ml one-mark volumetric flask (6.14). Dilute to the mark with water.

NOTE The use of this solution, which allows the interface between the solvent and aqueous layers to be seen more clearly, is optional (see 9.4.4). Other aqueous colour solutions may be used provided that they do not affect the result of the determination.

5.4 Diethyl ether (C₂H₅OC₂H₅), free from peroxides (see A.3), containing no more than 2 mg/kg of antioxidants, and complying with the requirements for the blank test (see 9.2.2, A.1 and A.4).

NOTE The use of diethyl ether could lead to hazardous situations. Due to expected changes in safety regulations, studies are ongoing to replace diethyl ether by another reagent provided that it does not affect the end result of the determination.

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5.5 Light petroleum, with any any aboiling arange between is 30 °C2 and 8 604°C2 or as equivalent, **pentane** (CH₃[CH₂]₃CH₃) with a boiling point of 36 °C1 and complying with the requirements for the blank test (see 9.2.2, A.1 and A.4).

NOTE The use of pentane is recommended because of its higher purity and constant quality.

5.6 Mixed solvent

Shortly before use, mix equal volumes of diethyl ether (5.4) and light petroleum (5.5).

6 Apparatus

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

Usual laboratory equipment and, in particular, the following.

- **6.1** Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.
- **6.2 Centrifuge**, capable of holding the fat-extraction flasks or tubes (6.6) and capable of spinning at a rotational frequency of 500 min⁻¹ to 600 min⁻¹ to produce a radial acceleration 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 9.4.7).

6.3 Distillation or evaporation apparatus, for distilling the solvents and ethanol from the boiling or conical flasks, or evaporating from beakers and dishes (see 9.4.14) 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 $^{\circ}$ C \pm 2 $^{\circ}$ C throughout its working space.

The oven shall be fitted with a suitable thermometer.

- **6.5** Water bath, capable of being maintained at a temperature of 65 °C \pm 5 °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 then the procedure is different. The alternative procedure is given in annex B.

The fat-extraction flasks shall be provided with good quality corks or stoppers of another material [e.g. silicone rubber or polytetrafluoroethylene (PTFE)] unaffected by the reagents used. Bark corks shall be extracted with the diethyl ether (5.4), kept in water at a temperature of 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**, for holding the fat-extraction flasks (or tubes) (6.6).
- **6.8 Wash bottle**, suitable for use with the mixed solvent (5.6).

A plastics wash bottle shall not be used.

6.9 Fat-collecting vessels, such as boiling flasks (flat-bottomed), of capacities 125 ml to 250 ml, conical flasks, of capacity 250 ml, or metal dishes. **STANDARD PREVIEW**

If metal dishes are used, they shall be of stainless steel, flat-bottomed with a diameter of 80 mm to 100 mm and a height of approximately 50 mm. (Standards.iten.al)

6.10 Boiling aids, fat-free, of non-porous porcelain or silicon carbide (optional when metal dishes are used).

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- 6.11 Measuring cylinders, of capacities 5 ml and 25 mla-iso-1736-2001
- **6.12 Pipettes**, graduated, of capacity 10 ml.
- **6.13 Tongs**, made of metal, for holding flasks, beakers or dishes.
- **6.14 Volumetric flask**, one-mark, of capacity 100 ml.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Store the samples at a temperature of between 2 °C and 6 °C from the time of sampling.

8 Preparation of test sample

Thoroughly mix the test sample by repeatedly rotating and inverting the sample container. If necessary, transfer all of the test sample to an airtight container of approximately twice the volume of the test sample to allow this operation to be carried out.

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