



SLOVENSKI STANDARD SIST EN ISO 8381:2001

01-februar-2001

Hrana za majhne otroke na osnovi mleka - Določevanje maščob - Gravimetrijska metoda (Referenčna metoda) (ISO 8381:2000)

Milk-based infant foods - Determination of fat content - Gravimetric method (Reference method) (ISO 8381:2000)

Säuglingsnahrung auf Milchbasis - Bestimmung des Fettgehaltes - Gravimetrisches Verfahren (Referenzverfahren) (ISO 8381:2000)

Aliments a base de lait pour enfants en bas âge - Détermination de la teneur en matière grasse - Méthode gravimétrique (Méthode de référence) (ISO 8381:2000)

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67.100.99	Drugi mlečni proizvodi	Other milk products
67.230	Predpakirana in pripravljena hrana	Prepackaged and prepared foods

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 8381

March 2000

ICS 67.100.01

English version

Milk-based infant foods - Determination of fat content -
Gravimetric method (Reference method) (ISO 8381:2000)

Aliments à base de lait pour enfants en bas âge -
Détermination de la teneur en matière grasse - Méthode
gravimétrique (Méthode de référence) (ISO 8381:2000)

Säuglingsnahrung auf Milchbasis - Bestimmung des
Fettgehaltes - Gravimetrisches Verfahren
(Referenzverfahren) (ISO 8381:2000)

This European Standard was approved by CEN on 24 December 1999.

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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

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Foreword

The text of the International Standard ISO 8381:2000 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 8381:2000 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

ISO
8381

Second edition
2000-03-01

Milk-based infant foods — Determination of fat content — Gravimetric method (Reference method)

*Aliments à base de lait pour enfants en bas âge — Détermination de la
teneur en matière grasse — Méthode gravimétrique (Méthode de
référence)*

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ISO 8381:2000(E)**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 8381 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 second edition cancels and replaces the first edition (ISO 8381:1987), which has been technically revised.

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

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Milk-based infant foods — 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 milk-based infant foods.

The method is applicable to liquid, concentrated and dried milk-based infant foods with no, or not more than a mass fraction of 5 % (dry matter) of starch or dextrin, or vegetable, fruit, meat, etc.

NOTE 1 Malto-dextrins without higher molecular mass dextrins, which are often present in infant foods, do not disturb the Röse-Gottlieb extraction even when present in high concentrations.

The method is not applicable to products which do not dissolve completely in ammonia owing to the presence of more than a few percent of starch or dextrin, or to the presence of hard lumps. The method is also not applicable to products which contains free fatty acids in significant quantities. The results obtained for these products will be too low.

NOTE 2 For such products, recourse should be made to a method utilizing the Weibull-Berntrop principle (see ISO 8262-1).

2 Normative reference

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 agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions 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 apply.

3.1

fat content of milk

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*)].

ISO 8381:2000(E)**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.

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 NH_3 of approximately 25 % ($\rho_{20} = 910 \text{ g/l}$).

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 ($\text{C}_2\text{H}_5\text{OH}$), or ethanol denatured by methanol, containing a volume fraction of ethanol of at least 94 %. (See A.5.)

5.3 Congo red solution

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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 ($\text{C}_2\text{H}_5\text{OC}_2\text{H}_5$), 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.

5.5 Light petroleum, with any boiling range between 30 °C and 60 °C or, as equivalent, **pentane** ($\text{CH}_3[\text{CH}_2]_3\text{CH}_3$) with a boiling point of 36 °C 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 $100 \text{ }^{\circ}\text{C}$.

6.4 Drying oven, electrically heated, with ventilation port(s) fully open, capable of being maintained at a temperature of $102 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ throughout its working space.

The oven shall be fitted with a suitable thermometer.

6.5 Water baths, capable of being maintained at temperatures of between $30 \text{ }^{\circ}\text{C}$ and $40 \text{ }^{\circ}\text{C}$, $40 \text{ }^{\circ}\text{C}$ and $60 \text{ }^{\circ}\text{C}$, and $60 \text{ }^{\circ}\text{C}$ and $70 \text{ }^{\circ}\text{C}$.

6.6 Mojonier-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 bark corks or stoppers of other 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 $60 \text{ }^{\circ}\text{C}$ 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.

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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.

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.

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

6.11 Measuring cylinders, of capacities 5 ml and 25 ml.

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 all liquid, viscous or pasty samples at a temperature of between $2 \text{ }^{\circ}\text{C}$ and $6 \text{ }^{\circ}\text{C}$ from the time of sampling to the time of commencing the procedure. In the case of samples in sealed cans or bottles, store these samples unopened at a temperature below $20 \text{ }^{\circ}\text{C}$.