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**Milk and milk products — Determination  
of milk fat purity by gas chromatographic  
analysis of triglycerides (Reference  
method)**

*Lait et produits laitiers — Détermination de la pureté des matières  
grasses laitières par analyse chromatographique en phase gazeuse des  
triglycérides (Méthode de référence)*



Reference numbers  
ISO 17678:2010(E)  
IDF 202:2010(E)

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ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

International Dairy Federation  
Diamant Building • Boulevard Auguste Reyers 80 • B-1030 Brussels  
Tel. + 32 2 733 98 88  
Fax + 32 2 733 04 13  
E-mail [info@fil-idf.org](mailto:info@fil-idf.org)  
Web [www.fil-idf.org](http://www.fil-idf.org)

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17678|IDF 202 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

## Foreword

**IDF (the International Dairy Federation)** is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50 % of the IDF National Committees casting a vote.

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ISO 17678|IDF 202 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the Joint ISO-IDF Project Group on *Foreign fats* of the Standing Committee on *Analytical methods for composition* under the aegis of its project leader, Dr J. Molkenin (DE).

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# Milk and milk products — Determination of milk fat purity by gas chromatographic analysis of triglycerides (Reference method)

## 1 Scope

This International Standard specifies a reference method for the determination of milk fat purity using gas chromatographic analysis of triglycerides. Both vegetable fats and animal fats such as beef tallow and lard can be detected. By using defined triglyceride equations, the integrity of milk fat is determined.

Basically, the method applies to bulk milk, or products made thereof, irrespective of feeding, breed or lactation conditions. In particular, the method is applicable to fat extracted from milk products purporting to contain pure milk fat with unchanged composition, such as butter, cream, milk, and milk powder.

However, under the circumstances listed hereafter, a false positive result can be obtained. Hence, the method is not applicable to milk fat:

- a) obtained from bovine milk other than cow's milk;
- b) obtained from single cows;
- c) obtained from cows which received an exceptionally high feeding of pure vegetable oils such as rapeseed oil;
- d) obtained from colostrum;
- e) subjected to technological treatment such as removal of cholesterol or fractionation;
- f) obtained from skim milk or buttermilk;
- g) extracted by using the Gerber, Weibull–Berntrop or Schmid–Bondzynski–Ratzlaff methods, or that has been isolated using detergents (e.g. the Bureau of Dairy Industries method).

With the extraction methods specified in g), substantial quantities of partial glycerides or phospholipids can pass into the fat phase. Consequently, the scope of this International Standard excludes certain products and particularly cheese, whose ripening process can also affect the fat composition to such a degree that a false positive result is obtained.

**NOTE 1** In nature, butyric (*n*-butanoic) acid (C4) occurs exclusively in milk fat and enables quantitative estimations of low to moderate amounts of milk fat in vegetable and animal fats to be made. However, due to the large variation of C4, whose approximate content ranges from 3,1 % mass fraction to 3,8 % mass fraction, it is difficult to provide qualitative and quantitative information for foreign fat to pure milk fat ratios of up to 20 % mass fraction (see Reference [11]).

**NOTE 2** In practice, quantitative results cannot be derived from the sterol content of vegetable fats, because they depend on production and processing conditions. Furthermore, the qualitative determination of foreign fat using sterols is ambiguous.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1211|IDF 1, *Milk — Determination of fat content — Gravimetric method (Reference method)*

ISO 2450|IDF 16, *Cream — Determination of fat content — Gravimetric method (Reference method)*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7328|IDF 116, *Milk-based edible ices and ice mixes — Determination of fat content — Gravimetric method (Reference method)*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

**3.1 milk fat purity**  
absence of vegetable and animal fats determined by the procedure specified in this International Standard

NOTE The purity is determined using *S*-values, which are calculated from the content of triglycerides. Triglyceride mass fractions are expressed as percentages.

## 4 Principle

Fat extracted from milk or milk products is analysed by gas chromatography (GC) using a packed or a short capillary column to determine triglycerides (TGs), separated by total carbon numbers. By inserting the mass fraction, expressed as a percentage, of fat molecules of different sizes (C24 to C54, using even C numbers only) into suitable TG equations, *S*-values are calculated. If the *S*-values exceed the limits established with pure milk fat, the presence of foreign fat is detected.

NOTE 1 The suitability and equivalence of both packed and capillary columns have been demonstrated previously (see References [8] to [10]).

NOTE 2 An *S*-value is the sum of weighted TG mass fractions.

## 5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

**5.1 Water** complying with the requirements of ISO 3696, grade 2.

**5.2 Carrier gas**, nitrogen or, alternatively, helium or hydrogen, all with a purity of at least 99,995 % volume fraction.

**5.3 Fat standards**, purity at least 99 % mass fraction, for standardizing the milk fat standard described in 8.3.3.

**5.3.1 Triglyceride standards**, saturated; suitable products are available commercially.

**5.3.2 Cholesterol standard**.

**5.4 Methanol** (CH<sub>3</sub>OH), with a water content of not more than 0,05 % mass fraction.