

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN ISO 17678:2010 sisaldab Euroopa standardi EN ISO 17678:2010 ingliskeelset teksti.

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ICS 67.100.99; 67.200.10

English Version

Milk and milk products - Determination of milk fat purity by gas chromatographic analysis of triglycerides (Reference method)
(ISO 17678:2010)

Lait et produits laitiers - Détermination de la pureté des
matières grasses laitières par analyse chromatographique
en phase gazeuse des triglycérides (Méthode de référence)
(ISO 17678:2010)

Milch und Milcherzeugnisse - Bestimmung der Reinheit des
Milchfetts durch gaschromatographische Triglyceridanalyse
(Referenzverfahren) (ISO 17678:2010)

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Foreword

This document (EN ISO 17678:2010) has been prepared by Technical Committee ISO/TC 34 "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 NEN.

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of an identical text or by endorsement, at the latest by August 2010, and conflicting national standards shall be withdrawn at the latest by August 2010.

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Endorsement notice

The text of ISO 17678:2010 has been approved by CEN as an EN ISO 17678:2010 without any modification.

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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 (CH₃OH), with a water content of not more than 0,05 % mass fraction.