INTERNATIONAL STANDARD

ISO 11870

IDF 152

Second edition
2009-10-01

Milk and milk products — Determination of fat content — General guidance on the use of butyrometric methods

Lait et produits laitiers — Détermination de la teneur en matière grasse — Lignes directrices générales pour l'utilisation des méthodes butyrométriques

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Reference numbers
ISO 11870:2009(E)
IDF 152:2009(E)

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Published in Switzerland

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Foreword

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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 11870|IDF 152 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.

This second edition of ISO 11870|IDF 152 cancels and replaces the first edition (ISO 11870:2000), of which it constitutes a minor revision.

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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 Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the IDF National Committees casting a vote.

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

ISO 11870|IDF 152 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 former Joint ISO-IDF Group of Experts (E301 — *Fat*) which is now part of the Joint ISO-IDF Action Team on *Fat* of the Standing Committee on *Main components in milk*.

This edition of ISO 11870|IDF 152 cancels and replaces IDF 152A:1997, of which it constitutes a minor revision.

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Introduction

Reference methods for the determination of fat in milk and milk products are time-consuming to apply and require some experience if reliable results are to be obtained.

Butyrometric techniques, which are simpler to apply, make it possible to obtain fat contents for various milk products quickly. This is why they are used in a great number of industrial laboratories as a fast method for routine checks.

Two acid-butyrometric methods used in many countries to determine the fat content of milk (Gerber method) and of cheese (Van Gulik method) are the subject of International Standards. The apparatus has also been standardized.

In addition, there are other butyrometric methods and butyrometers which have been described or applied in various countries for other types of products (cream, milk powder, etc.).

Whilst only one procedure exists as a reference method for a particular product type, this is not the case for butyrometric methods. Depending upon the country, different butyrometric methods may exist for one single type of product, presenting many problems for the harmonization of such procedures.

Another problem relates to the applicability of such methods. Indeed, with evolving manufacturing technologies, the variety of milk products is such that it is not possible to determine a method which can be applied to all varieties of a single type of product (milk, cheese, cream, etc.). Tests have confirmed this and have shown that the butyrometric methods already standardized have been attributed fields of application which are far too wide-ranging.

Thus this general guide has been prepared to be used in conjunction with existing International Standards.

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Milk and milk products — Determination of fat content — General guidance on the use of butyrometric methods

1 Scope

This International Standard gives guidance on:

a) existing standardized methods (both reference and butyrometric) for the determination of fat in various milk products;

b) the principles underlying any acid-butyrometric analysis and the main operating requirements;

c) a validation procedure for a butyrometric method in relation to the relevant reference method.

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 2446|IDF 226:2008, *Milk — Determination of fat content*

ISO 3433|IDF 222, *Cheese — Determination of fat content — Van Gulik method*

3 Principle

The principles of any butyrometric method remain constant, independent of the product to be analysed. Protein is digested with sulfuric acid. The fat in the product is separated by centrifuging it in a butyrometer. The separation is enhanced by the addition of a small quantity of isoamyl alcohol. The butyrometer scale is then read directly with or without correction.

4 Methods for the determination of fat content

Methods for the determination of fat content are based upon acid-butyrometric and reference gravimetric methods.

The Gerber method is specified in ISO 2446|IDF 226 and the Van Gulik method in ISO 3433|IDF 222. Existing butyrometric and reference methods for most dairy products are listed in Table A.1.

5 Reagents

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

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5.1 Sulfuric acid, pure, colourless or pale amber and containing no impurities.

5.2 Isoamyl alcohol. A volume fraction of at least 99 % of the isoamyl alcohol shall consist of the primary alcohols 3-methylbutan-1-ol and 2-methylbutan-1-ol, the only permissible major impurities being 2-methylpropan-1-ol and butan-1-ol. It shall be free from secondary pentanols, 2-methylbutan-2-ol, furan-2-al (furfural, furan-2-carboxaldehyde, 2-furaldehyde), gasoline (petrol), and derivatives of benzene. Not more than a trace of water shall be present.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Butyrometer and **stopper**, suitable for the method used.

6.2 Dispensers, for acid and alcohol, to deliver the requisite volumes precisely and with sufficient repeatability.

6.3 Centrifuge, to accommodate a butyrometer, provided with a speed indicator which indicates the rotational frequency with a maximum tolerance of ± 70 r/min, preferably of the vertical-loading type rather than the horizontal-loading type.

The centrifuge should be capable of maintaining the temperature of the butyrometer contents at between 30 °C and 50 °C after centrifuging.

The use of a heated centrifuge is permitted provided that the results obtained agree with the reference method.

When loaded, the centrifuge should be capable of producing, within 2 min, a relative centrifugal acceleration of $350g \pm 50g$ at the outer end of the butyrometer stopper. This acceleration is produced by centrifuges with an effective radius (horizontal distance between the centre of the centrifuge spindle and the outer end of the butyrometer stopper) as given in Table 1, operated at the speed indicated.

Table 1 — Centrifuge accelerations

Effective radius mm	Revolutions per minute ± 70 r/min
240	1 140
245	1 130
250	1 120
255	1 110
260	1 100
265	1 090
270	1 080
275	1 070
300	1 020
325	980

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The relative centrifugal acceleration produced in a centrifuge, α, is given by:

$$\alpha = 1{,}12\ rn^2 \times 10^{-6}$$

where

r is the effective horizontal radius, in millimetres;

n is the rotational frequency, in number of revolutions per minute.

6.4 Pipette or **analytical balance**, precise enough to ensure accurate distribution when preparing the test sample.

6.5 Water bath, thermostatically controlled, capable of maintaining the whole apparatus at the desired uniform temperature, and offering sufficient depth for the butyrometers to be supported in a vertical position with their scale graduations completely immersed.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707 | IDF 50[2].

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

8 Preparation of test sample

For each product to be analysed, refer to the relevant reference method.

9 Procedure

Accurately and quickly, take a test portion from a homogeneous sample. Prepare the test portion by dissolving the protein by shaking, and record the type of shaking (vertical or horizontal, frequency and amplitude, etc.).

Centrifuge for a specified time with a specified centrifugal force. Take readings rapidly, immediately on removal from the water bath. If the fat cools, its volume decreases and the results obtained are incorrect.

If readings are being taken by hand, hold the butyrometer vertically with the point of reading at eye level. During this process, hold the stopper absolutely still.

If the fat is turbid or dark in colour, or if there is white or black material at the bottom of the fat column, the value for fat content is not reliable.

If phase separation is not clear-cut, centrifuging twice would produce too high a result. In such cases, repeat the analysis.

10 Care of butyrometers

After the reading has been taken, invert the butyrometers, stoppers upwards, on a rack. In approximately 30 min, the fat from the bulb and the graduation tube rise upward under the stopper. As the butyrometers are still hot, remove the stoppers carefully, holding the open end close to the bottom of a sink.

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Wash the emptied butyrometers, which are still hot, without using a bottle brush, by shaking them vigorously with an appropriate detergent. Plunge the butyrometers into water containing a detergent, then fill and empty them several times and shake them vigorously, paying special attention to the small bulb.

Rinse the butyrometers three times with hot water (i.e. three separate amounts, vigorously shaking and emptying each time).

Finally, shake the butyrometers out very vigorously and allow to drain with their open end downwards. They may be used again immediately, whilst still damp. However, it is important to shake them out again, immediately prior to use, in order to remove to the maximum extent any water droplets still inside.

11 Validation principle for a butyrometric method by comparison with the corresponding reference method

Whatever the butyrometric method used and whatever the product analysed, the method is only an empirical one. The result obtained shall be comparable with the result obtained by the reference method. All laboratories, therefore, should validate their butyrometric methods by comparison with the corresponding reference method.

Adjusting the results from the acid-butyrometric method to those of the reference method is accomplished by varying a number of parameters and especially the following:

a) concentration of the acid;

b) temperature of the water bath;

c) the physical properties of the butyrometer, such as the volume of the large bulb, the length and/or width of the graduated tube, the form of the graduated tube and the graduation scale.

The criterion for optimal adjustment is the absolute difference between the result obtained with the (modified) routine method and the result from the reference method, which should be minimized.

Once a set of conditions for the routine method has been found that gives equivalent results, the equivalence should be confirmed by comparing duplicate determinations with the two methods on several samples. The results from each set of samples can be compared using the Student t-test.

CAUTION — This test presupposes that the variances of the two methods are equal. This should be checked in case of doubt.

As the optimal conditions found may only be valid for a limited concentration range of the analyte, the whole range for which the routine method is used should be tested. Analysing in duplicate can do this by both methods (routine and reference) with samples spanning the whole range of fat content. The equivalence of both methods should then be established by comparing the results for each sample using the t-test. If necessary, the results from testing the whole range can be used to establish a correction table [see also ISO 8196 (all parts)|IDF 128 (all parts)[13]].

It should be stressed that, in laboratories which always verify the same type of product produced by the same process, the adjustment of the results obtained by the butyrometric method to the results obtained by the reference method shall be as perfect as possible. The absolute difference shall approximate to zero.

If a laboratory has to determine the fat content of products of the same type but of different origin, there are problems in adjusting its butyrometric method so as to obtain a value identical in every case to that of a reference method.

Even for standardized butyrometric methods, regular checks are recommended because such methods, in addition to all the causes for variations already listed, have their own limitations (see Annex A).

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Annex A
(normative)

Limitations of butyrometric methods

A.1 Gerber method

See ISO 2446|IDF 226.

This method is applicable to raw or pasteurized, whole or partially skimmed, liquid milk and comprises modifications applicable to:

a) milk containing preservatives;

b) homogenized milk;

c) skimmed milk.

However, it should be noted that:

— the volume of test portion to be used has never been internationally agreed, therefore it is not harmonized (whatever the volume of sample used, the result of the analysis shall agree with the reference method; see also ISO 2446|IDF 226:2008, 6.1.2);

— the modified procedures for homogenized and skimmed milk have not proved satisfactory, therefore, several countries have developed their own procedures;

— the presence of added substances, such as sugar, flavourings, chocolate, etc., interferes with the results.

A.2 Van Gulik method

See ISO 3433|IDF 222.

This method is applicable to all types of cheese. However, it has been shown that the method is not wholly satisfactory for:

a) blue-veined cheeses — interference is produced by the presence of smaller or larger deposits at the base of the fat column;

b) long-matured cheeses — lipolysis alters the composition of the triacylglycerols, which distorts the fat content result obtained;

c) cheeses made from homogenized milks — results obtained are always too low;

d) low- or high-fat content cheeses — the results are not always in conformity with those obtained by the Schmid-Bondzynski-Ratzlaff reference method;

e) cheeses containing added substances — these may interfere and may agglutinate at the bottom of the column, producing results which are too high or too low in comparison with the Weibull-Berntrop reference method;

f) cheeses made from milk other than cow's milk — the milkfat composition is different and, therefore, any butyrometric determination produces results which, to varying degrees, are incorrect.

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