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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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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.

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.

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.

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.

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

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.