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Milk — Determination of fat content — Acido-butyrometric (Gerber method)

Lait — Détermination de la teneur en matière grasse — Méthode acido-butyrométrique (méthode de Gerber)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document 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.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

ISO and IDF collaborate closely on all matters of standardization relating to methods of analysis and sampling for milk and milk products. Since 2001, ISO and IDF jointly publish their International Standards using the logos and reference numbers of both organizations.

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This document was prepared by the IDF Standing Committee on Analytical Methods for Composition and ISO Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by ISO and IDF.

All work was carried out by the ISO/IDF Project Group C21 of the Standing Committee on Analytical Methods for Composition under the aegis of its project leader, Mr Philippe Trossat (FR).

Milk — Determination of fat content — Acido-butyrometric (Gerber method)

1 Scope

This document specifies a method, the acido-butyrometric or "Gerber", for determining the fat content of milk. It is applicable to whole milk and partially skimmed milk.

It is also applicable to milk containing authorized preservatives (potassium dichromate, bronopol).

It does not apply to formalin milk, nor to milks that have undergone a homogenisation treatment.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
acido-butyrometric method
traditional technique which, when applied to a whole milk having a fat content between 3 g and 5 g per 100 ml or 100 g , gives a fat content that is equivalent, after correction by the density at 20 °C, to that obtained by the gravimetric reference method

4 Principle

Dissolution of the proteins by the addition of sulfuric acid, followed by separation of the milk's fat by centrifuging in a butyrometer. The separation is assisted by the addition of amyl alcohol.

Determination of the fat content in grams per 100 ml or 100 g of milk by direct reading on the butyrometer scale.

5 Reagents

All reagents shall be of recognised analytical grade and the water used shall be distilled water or water of at least equivalent purity.

5.1 Concentrated sulfuric acid, density ρ_{20} = 1,820 g/ml ± 0,005 g/ml, colourless or barely amber, free from any impurity that may influence the result.

5.2 Amyl alcohol.

5.2.1 Composition

The amyl alcohol shall be composed of at least 98 % in volume of primary alcohols 3-methylbutan-1-ol (boiling point 131,4 °C) and 2-methylbutan-1-ol (boiling point 128,0 °C), the only permissible impurities being 2-methylpropane-1-ol[1)] and butan-1-ol. The ratio of the two isomers shall be 91 % ± 2 % of 3-methylbutan-1-ol to 9 % ± 2 % of 2-methylbutan-1-ol in the volume of primary alcohols as defined above.

It shall be exempt from all compounds which may have an influence on the result given by the acido-butyrometric method, such as secondary amyl alcohols, 2-methylbutan-2-ol[2)], furfural, petroleum and benzene derivatives. Only traces of water can be tolerated.

5.2.2 Physical appearance

Clear and colourless.

5.2.3 Density

ρ_{20} = 0,813 g/ml ± 0,005 g/ml at 20 °C.

5.2.4 Furfural and other organic impurities

The absence of impurities is revealed if the colour of a volume to volume mixture of amyl alcohol and sulphuric acid remains yellow or light brown.

5.2.5 Distillation range

When distilled at a pressure of 1 013 mbar (760 mm of Hg), a volume fraction of not less than 98 % shall distil below 132 °C and a volume fraction of not more than 5 % at below 128 °C. The alcohol shall not leave any residue after distillation.

6 Apparatus

Ordinary laboratory equipment, and in particular the following.

6.1 Milk butyrometer, in accordance with Annex A, equipped with an appropriate stopper in accordance with Annex B.

Use the butyrometer type with the scale range that corresponds best to the expected fat content of the sample.

6.2 Pipette or automatic system, capable of delivering 11,00 ml ± 0,03 ml of milk for expression in g of fat/100 ml of milk.

6.3 Pipette or automatic system, capable of delivering 10,75 ml ± 0,03 ml of milk for expression in g of fat/100 g of milk.

6.4 Pipette or automatic system, capable of delivering 10,0 ml ± 0,2 ml of sulphuric acid (5.1).

6.5 Pipette or automatic system, capable of delivering 1,00 ml ± 0,05 ml of amyl alcohol (5.2).

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1) Iso-butyl alcohol.

2) Tertiary amyl alcohol.

6.6 Centrifuge, suited for the butyrometer and equipped, preferably, with a timer (optional) and a speed indicator giving the number of revolutions per minute.

When fully loaded, the centrifuge shall be capable of producing, within 2 min, a centrifugal acceleration of (350 g ± 50 g) at the outer end of the butyrometer stopper. Such an acceleration can be obtained with centrifuges having the effective radius (horizontal distance between the centre of the centrifuge spindle and the outer end of the butyrometer stopper) operated at the rotational frequency given in Table 1.

NOTE For centrifuges equipped with a translucent lid, the rotational speed can be checked with an optical tachometer.

Table 1 — Correspondence between the effective radius and the number of revolutions per minute

Effective radius mm	Revolutions per minute	Centrifugal acceleration[a] g
240	1 140	349
245	1 130	350
250	1 120	351
255	1 110	351
260	1 100	352
265	1 090	352
270	1 080	352
275	1 070	352
300	1 020	349
325	980	349

[a] The centrifugal acceleration at the extremity of the radius of a centrifuge is given by the formula: $1{,}12\ RN^2\ 10^{-6}$

where

R is the effective horizontal radius, in millimetres;

N is the rotational speed, in revolutions per minute.

6.7 Water bath, for supporting the butyrometers in a vertical position with their scales completely immersed, maintained at a temperature of 65 °C ± 2 °C.

6.8 Water bath, capable of being maintained at a temperature of 40 °C ± 2 °C.

6.9 Thermometer, for determining the temperature of the water bath to within ±1 °C.

6.10 Timer (optional).

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 707 | IDF 50[3].

It is important that the laboratory receive a sample that is representative and has not been damaged or changed during transport or storage.

8 Procedure

8.1 Preparation of the test sample

Using the water bath (6.8), warm the test sample to a temperature of 40 °C ± 2 °C. Gently mix the test sample thoroughly by repeatedly inverting the sample bottle without causing frothing or churning.

Cool the test sample quickly to approximately 20 °C.

8.2 Preparation of the butyrometer and test portion

Using a pipette or an automatic system (6.4), introduce 10 ml of sulphuric acid (5.1) into the butyrometer (6.1) without wetting the neck.

Gently invert the vessel containing the prepared sample (8.1) three or four times. Immediately sample the required volume of milk using a pipette or automatic system (6.2 or 6.3) and pour it into the butyrometer so that it forms a layer above the acid.

Using a pipette or an automatic system (6.5) measure 1 ml of amyl alcohol (5.2) and introduce it into the butyrometer without wetting the neck of the butyrometer or mixing the liquids.

Securely stopper the butyrometer, taking care to avoid mixing the different phases of the contents.

8.3 Dissolution of the proteins

Shake the butyrometer until the proteins are completely dissolved (there is an absence of white particles), then invert it.

The mixture is exothermic; the operator should take all customary precautions.

8.4 Centrifuging

Centrifuge immediately at room temperature after shaking for 5 min as soon as the required rotational speed is reached.

8.5 Reading

Remove the butyrometer from the centrifuge, adjusting the stopper if necessary, to bring the fat column onto the scale. Place the butyrometer, stopper downwards, in a water bath (6.7) at 65 °C ± 2 °C for 10 min; the water level shall be above the top of the fat column.

Remove the butyrometer from the water bath, stopper still downwards, and carefully adjust the stopper by pulling on it in order to bring the bottom of the fat column, with minimum column movement, in line with the nearest mark, preferably a main graduation line. (It is recommended to choose the 0 graduation line of the butyrometer as mark A.)

Note the graduation line (A) corresponding to the bottom of the fat column, then, taking care not to move the latter, note, as quickly as possible, the graduation line (B) coincident with the lowest point of the meniscus at the top of the fat column.

Carry out the reading to within 0,025 g per 100 ml or 100 g.

While reading, the butyrometer shall be held and moved vertically, in order to obtain the reading point at eye level and avoid parallax error. (Do not move the head.)

No more than 10 s shall elapse between the removal of the butyrometer and the end of the reading.

If it is necessary to verify the obtained result, replace the butyrometer in the water bath for approximately 5 min, then remove it and take the readings as indicated in the previous paragraph.

If the fat is turbid or dark coloured, or if there is a black or white deposit at the bottom of the fat column, the value obtained for the fat content will not be reliable.

9 Expression of results

9.1 Method of calculation

The fat content is expressed in grams per 100 ml of milk (test portion of 11 ml) or in grams per 100 g of milk (test portion of 10,75 ml).

The fat content of the milk is shown in Formula (1):

$$MG = B - A \quad (1)$$

where

A is the reading taken at the bottom of the fat column;

B is the reading taken at the top of the fat column.

9.2 Precision

9.2.1 General

The repeatability and reproducibility values are expressed with a probability level of 95 % and were obtained from interlaboratory tests according to ISO 5725-2[5]. Details on the interlaboratory collaborative tests are summarised in Annex C.

9.2.2 Repeatability

The difference between two single results, obtained on an identical product submitted to the same test by the same operator within a short interval of time, shall not exceed 0,05 g per 100 ml or 100 g.

9.2.3 Reproducibility

The difference between two single independent results, obtained by two operators working in different laboratories on an identical product submitted to a same test, shall not exceed 0,1 g per 100 ml or 100 g.

10 Milks having a fat content between 1,5 and 3,0 g/100 ml or g/100 g and 5,0 to 6,0 g/100 ml or g/100 g

The equivalence between the fat content in 9.1 and the fat content obtained by the reference method is not reached; however, the differences observed remain within the method's repeatability limits.

The difference for a milk having a 1,5 g/100 ml or g/100 g fat content is –0,03 g/100 ml or g/100 g; it is –0,02 g/100 ml or g/100 g for a milk with a 2,0 g/100 ml or g/100 g fat content and +0,02 g/100 ml or g/100 g for a milk with a 6,0 % fat content. These values are below the repeatability of the method. It is therefore not necessary to correct the values read on the butyrometers when the contents are outside the 3 g/100 ml or g/100 g fat content to 5 g/100 ml or g/100 g fat content[1].

11 Test report

The test report shall indicate the method used, the butyrometer scale and the results obtained.