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

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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.

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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).

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.

It shall, in addition, mention all operating details not specified in this document, or regarded as optional, together with details of any possible incidents that may have influenced the results, and in particular any comment stating whether the accuracy of the result is questionable.

The test report shall provide all the information required for the complete identification of the sample.

Annex A
(normative)

Characteristics of butyrometers

A.1 General

The purpose of this annex is to define the characteristics of butyrometers used for determination of the fat content in milk by the Gerber method.

The graduation scale expresses the result in grams of fat per 100 ml of milk when the test portion is 11 ml and in grams of fat per 100 g of milk when the test portion is 10,75 ml.

A.2 Classification

Milk butyrometers are classified in three types:

— butyrometers for testing milk 0 to 4 (80 increments);

— butyrometers for testing milk 0 to 5 (50 and 100 increments);

— butyrometers for testing milk 0 to 6 (60 increments).

A.3 Description

Butyrometers used for the determination of the fat content of milk using the Gerber method are glass appliances comprising the following (see Figure A.1):

— a cylindrical chamber terminated at one end by a neck;

— a graduated hollow cylindrical tube, closed at the other end of the chamber and coaxial to the latter, the other end of the tube being terminated by a bulb.

A.4 Designation

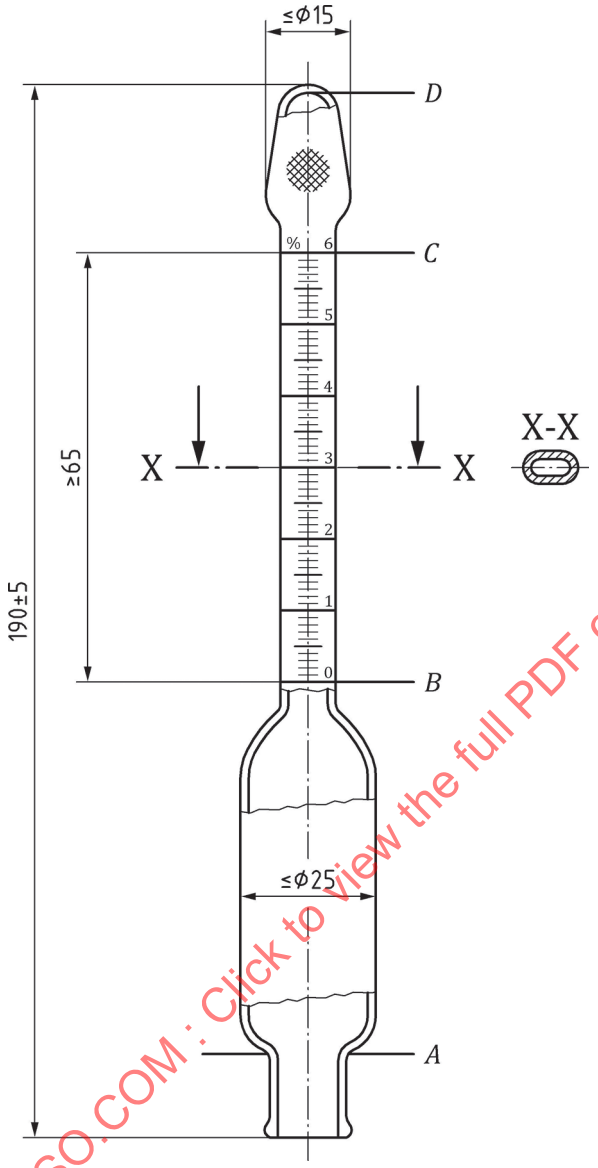
A milk butyrometer is designated by its name followed by its graduation scale limits and the reference to this document.

EXAMPLE For designation: milk butyrometer, 0 to 5, ISO 19662.

A.5 Manufacture

Milk butyrometers shall be manufactured in clear glass, shall be resistant to the thermal shocks and chemicals inherent to the method and shall be as defect-free as possible. They shall have been suitably annealed.

Dimensions in millimetres



Key

≤ Ø25 Maximum internal diameter of the chamber: ≤ 25 mm

Figure A.1 — Milk butyrometer

A.6 Geometric characteristics

A.6.1 General dimensions

Milk butyrometers shall have the following dimensions (see Figure A.1):

Total length	: 190 mm ± 5 mm
Neck internal diameter	: 11 mm ± 0,5 mm
Neck length	: 15 mm ± 1,0 mm
Maximum external diameter of the chamber	: 25 mm
External diameter of the end bulb	: approximately 15 mm

The internal surface shall be smooth and free from any defect that could, during the course of the determination, retain fat elsewhere than in the graduated tube. The appliance shall have a single axis of symmetry.

The wall thickness from one end of the butyrometer to the other shall be such that it renders the butyrometer sufficiently robust for its intended use. This thickness shall be as uniform as possible and at least 1,0 mm.

A.6.2 Neck

The neck shall be smooth, reinforced at the end by an external rim.

A.6.3 Chamber

The capacity of the chamber, measured between the end of the neck and the 0 graduation line (i.e. between levels A and B of Figure A.1) shall be equal to the following:

— 21,7 ml ± 0,3 ml for the 0 to 4 butyrometer;

— 21,5 ml ± 0,4 ml for the 0 to 5 and 0 to 6 butyrometers.

A.6.4 Graduated tube

The graduated tube of milk butyrometers shall be of the flat scale type (see Figure A.1, cross-section X–X); the ungraduated side of the tube shall not have a matt finish.

A.6.5 End bulb

The minimum capacity of the end bulb of the butyrometer up to the graduation nearest to the bulb (i.e. between level C and the end D of the bulb in Figure A.1) shall be 15 ml.

The bulb shall be tapered (see Figure A.1) and the inside of the bulb shall be shaped so that the fat can flow freely from the bulb towards the graduated tube when the butyrometer is inverted.

Provision shall be made on the end bulb for a small matt surface area on which a temporary mark can be made, if required.

A.7 Graduation

A.7.1 Graduation scale unit

The volume of a graduation unit is 0,125 ml.

A graduation unit corresponds to 1 g of fat.

A.7.2 Graduation scale length

The total length of the graduation scale (i.e. between levels B and C, Figure A.1) shall not be less than 65 mm.

A.7.3 Graduation scale position

The length of the flat part beyond each end of the scale shall not be less than 5 mm so as to be certain that the tube has a uniform transversal inner cross-section over at least 3 mm beyond each end of the scale.

A.7.4 Graduation lines

The graduation lines shall be fine, engraved in a distinct, permanent manner, and of a uniform thickness between 0,1 mm and 0,2 mm inclusive. The lines shall lie in planes perpendicular to the axis of the graduated tube, presenting no obvious irregularity in their spacing.

The graduation lines shall be coloured provided that the colour withstands the heat and the chemicals inherent to the method.

A.7.5 Graduation scheme

The graduation scheme shall correspond to the indications given in Table A.1.

The main graduation lines with or without numbers shall occupy the front side of the tube over its entire width.

The shortest graduation lines shall have a length of approximately 3 mm.

The intermediate length graduation lines shall exceed the shortest graduation lines both on the right and left by at least 1 mm.

Table A.1 — Summary table concerning milk butyrometers

Characteristics	Type of butyrometers			
Graduation in percent of fat	0 to 4	0 to 5	0 to 5	0 to 6
Number of divisions	80	50	100	60
Volume of a graduation unit	0,125 ml corresponding to 1 % of fat			
Chamber capacity	21,7 ml ± 0,3 ml	21,5 ml ± 0,4 ml		
Minimum length of graduation	65 mm			
Percentage between two consecutive lines (1 division)	0,05	0,1	0,05	0,1
Long lines at all the multiple percentages of i.e. every	0,5	1	0,5	1
	10 divisions			
Average length lines at all the intermediate percentages of i.e. every	0,1	0.5	0,1	0.5
	1 or 2 divisions	5 or 10 divisions		
Short lines at all the multiple intermediate percentages of i.e. every	0,05 or 0,1			
Marking at all the multiple percentages of i.e. every	1			
	20 divisions	10 divisions		

A.7.6 Graduation line numbering

The numbering locations are specified in Table A.1.

The graduation numbers shall be permanent and clearly legible; each number shall be located immediately above the graduation line to which it refers, to the right of the graduation axis when the butyrometer is placed vertically with the end bulb uppermost, and the neck at the bottom.

A.7.7 Tolerance on the capacity

The accepted tolerance on the capacity of the graduation for each of the two halves of the latter, i.e. between the limit graduation lines (levels B and C) and the line of the middle part of the scale (level M) is equal to the fortieth of the volume of a graduation unit, namely ±0,003 1 ml.