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Cream — Determination of fat content — Acido-butyrometric method

*Crème — Détermination de la teneur en matière grasse — Méthode
acido-butyrométrique*

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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents	1
6 Apparatus	2
7 Sampling	3
8 Procedure	3
8.1 Preparation of the test sample	3
8.2 Preparation of the test portion	4
8.3 Dissolution of the proteins	4
8.4 Centrifuging	4
8.5 Reading	4
9 Expression of results	5
9.1 Method of calculation	5
9.2 Precision	5
9.2.1 General	5
9.2.2 Repeatability	5
9.2.3 Reproducibility	5
10 Test report	5
Annex A (normative) Characteristics of butyrometers	6
Annex B (normative) Characteristics of weighing system	10
Annex C (normative) Characteristics of stoppers	11
Annex D (informative) Collaborative trial	13
Bibliography	14

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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All work was carried out by the ISO/IDF Project Group C20 of the Standing Committee on Analytical Methods for Composition under the aegis of its project leader, Mr Philippe Trossat (FR).

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Cream — Determination of fat content — Acido-butyrometric method

1 Scope

This document specifies an acidobutyrometric method for determining the fat content of cream. The reference method remains the gravimetric method (by ammoniacal ether extraction) described in ISO 2450 | IDF 16.

This method is applicable to cream having a fat content between 20 % and 50 % inclusive:

- intended for manufacturing butter;
- sweet, unmatured and non-inoculated;
- raw or having undergone a heat treatment;
- non-homogenized;
- with or without preservatives (2-bromo-2-nitropropane, 1,3 diol or bronopol).

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 cream having a fat content between 20 % and 50 % inclusive, gives a fat content expressed in grams per 100 g of cream that is equivalent to that obtained by the gravimetric reference method

4 Principle

Dissolution of the proteins by the addition of sulphuric acid, followed by separation of the cream'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 g of cream by direct reading on the butyrometer scale.

The cream shall not present any physical anomalies at the time of analysis.

5 Reagents

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

5.1 Concentrated sulfuric acid, density $\rho_{20} = 1,820 \text{ g/ml} \pm 0,005 \text{ g/ml}$, 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-ol¹⁾ and butan-1-ol. The ratio of the two isomers shall be 91 % $\pm$ 2 % of 3-methylbutan-1-ol to 9 % $\pm$ 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-ol²⁾, furfural, petroleum and benzene derivatives. Only traces of water can be tolerated.

5.2.2 Physical appearance

Clear and colourless.

5.2.3 Density

$\rho_{20} = 0,813 \text{ g/ml} \pm 0,005 \text{ g/ml}$ 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 Analytical balance, accurate to within 1 mg.

6.2 Cream butyrometer, 0 % to 50 % in accordance with [Annex A](#), equipped with a weighing system in accordance with [Annex B](#) and appropriate stoppers in accordance with [Annex C](#).

6.3 Syringe, 5 ml volume approximately or pipette.

6.4 Automatic system or pipette, capable of delivering 10,0 ml $\pm$ 0,2 ml of sulphuric acid ([5.1](#)).

6.5 Automatic system or pipette, capable of delivering 1,00 ml $\pm$ 0,05 ml of amyl alcohol ([5.2](#)).

1) Iso-butyl alcohol.

2) Tertiary amyl alcohol.

6.6 Centrifuge, in which the butyrometers can be placed, provided with a speed indicator giving the number of revolutions per minute to within a maximum tolerance of $\pm 70 \text{ r/min}^{-1}$.

When fully loaded, the centrifuge shall be capable of producing, within 2 min, a centrifugal acceleration of $(350 \text{ g} \pm 50 \text{ g})$ 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 centrifugal force

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:
 $1,12 \text{ RN}^2 10^{-6}$
 where
 R is the effective horizontal radius, in millimetres;
 N is the rotational speed, in revolutions per minute.

6.7 Water bath for butyrometers, equipped with a temperature regulation device, capable of being maintained at $65 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ and with a device enabling to support the butyrometers in a vertical position.

6.8 Water bath, capable of being maintained at a temperature of $40 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$.

6.9 Thermometer, to determine the temperature of the water bath to within $\pm 1 \text{ }^{\circ}\text{C}$.

7 Sampling

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

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 $40 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$. 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 test portion

Weigh into the previously tared weighing system (6.2) $5 \text{ g} \pm 0,005 \text{ g}$ of the sample taken using the syringe (6.2). Place the system inside the butyrometer. Using the automatic system (6.4), introduce 10 ml of sulphuric acid (5.1) taking care not to wet the neck of the butyrometer and allowing the reagent to flow down along the wall of the butyrometer tube.

Using the automatic system (6.5), introduce 1 ml of amyl alcohol (5.2) into the butyrometer, without wetting the butyrometer neck nor mixing the liquids, allowing the alcohol to flow down along the wall of the butyrometer tube.

Using a pipette, add water without mixing the liquids allowing adjustment to the level to graduation 45 by means of the bottom stopper (in general around 6,5 ml to 7,0 ml is enough).

Stopper the top part of the butyrometer.

8.3 Dissolution of the proteins

Shake and invert the butyrometer, the operator being suitably protected against the risk of breakage, until its contents are thoroughly mixed, and until the proteins are completely dissolved. Place the butyrometer in the water bath (6.7) and keep it there for 5 min.

8.4 Centrifuging

Place the butyrometer in the centrifuge (6.6). Centrifuge for 5 min at room temperature as soon as the required rotational speed is reached.

8.5 Reading

Remove the butyrometer from the centrifuge and place it, wide stopper downwards, in the water bath (6.7) 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.

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.

Do not centrifuge a second time. The result obtained risks being excessively false.

9 Expression of results

9.1 Method of calculation

The fat content is expressed in grams per 100 g of cream.

The fat content of the cream 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 summarized in [Annex D](#).

9.2.2 Repeatability

The difference between two single results, obtained on an identical product submitted to testing by the same operator using the same equipment within a short interval of time, shall not exceed 0,35 g per 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, shall not exceed 0,65 g per 100 g.

10 Test report

The test report shall indicate the method used 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.

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 a 0 % to 50 % graduated butyrometer used for the determination of the fat content of cream using the acido-butyrometric method.

The graduation scale expresses the result in grams of fat per 100 g of cream.

A.2 Classification

Cream butyrometers are classified in one type:

- butyrometers for testing cream 0 to 50 (100 divisions).

A.3 Description

Butyrometers used for the determination of the fat content of cream using the acido-butyrometric method are glass appliances comprising the following (see [Figure A.1](#)):

- a cylindrical chamber terminated at one end by a neck;
- a graduated hollow flat tube, closed at the end of the chamber and coaxial to the latter, the other end of the tube being open.

A.4 Designation

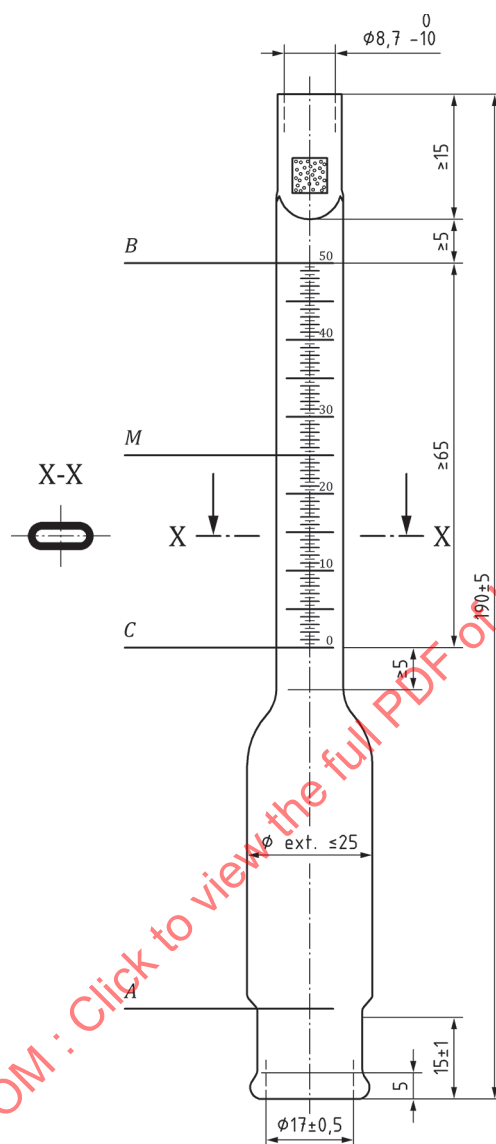
A cream butyrometer is designated by its name followed by the graduation scale and the reference to this document

EXAMPLE For designation: cream butyrometer, 0 to 50, ISO 19660:2018.

A.5 Manufacture

Cream 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

$\leq \phi 25$ Maximum internal diameter of the chamber: ≤ 25 mm.

Figure A.1 — Cream butyrometer

A.6 Geometric characteristics

A.6.1 General dimensions

Cream butyrometers shall have the following dimensions (see [Figure A.1](#)):

Total length	: 190 mm ± 5 mm
Neck internal diameter	: 17 mm ± 0,5 mm
Neck length	: 15 mm ± 1,0 mm
Maximum external diameter of the chamber	: 25 mm
Internal diameter of the opening opposite to the neck	: 8,7 mm to 10 mm
Minimum length of the cylindrical part of the end opposite to the neck	: 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 cream 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 of the cream butyrometer 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 C in [Figure A.1](#)) shall be equal to 22,0 ml ± 0,5 ml.

A.6.4 Graduated tube

The graduated tube of cream 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 Opening opposite to the neck

The opening opposite to the neck shall be cylindrical, with possibility of a tarnished area.

A.7 Graduation

A.7.1 Graduation scale unit

The volume of a graduation unit is 0,057 2 ml.

The basis of the scale is 0 to 50; the scale is graduated in divisions of 0,5.

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](#) and in [Figure A.1](#).

The main graduation lines 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 cream butyrometers

Characteristics	Values
Graduation in percent of fat	0 to 50
Number of divisions	100
Volume of a graduation unit	0,057 2 ml corresponding to 1 % of fat
Chamber capacity	22 ml $\pm$ 0,5 ml
Minimum length of graduation	65 mm
Capacity of the 0 to 50 scale zone	2,860 ml
Short lines every (divisions)	0,5
Average length lines at all the multiple percentages of i.e. every	1 2 divisions
Long lines at all the multiple percentages of i.e. every	5 10 divisions
Marking at all the multiple percentages of i.e. every	10 20 divisions

A.7.6 Graduation line numbering

The numbering shall figure for the main graduations (0 – 10 – 20 – 30 – 40 – 50), in compliance with the indications given 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 graduated tube uppermost, and the neck at the bottom.

A.8 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 $\pm 0,020$ ml.

Annex B (normative)

Characteristics of weighing system

B.1 Material

The weighing systems (see [Figure B.1](#)) shall be made of a material which withstands the thermal shocks and chemicals inherent to the method (glass, plastics, etc.).

B.2 Glass container

In the case where a glass container is used, the thickness of the walls shall be at least 1 mm.

Other weighing systems may be used provided that they give the same results.

Dimensions in millimetres

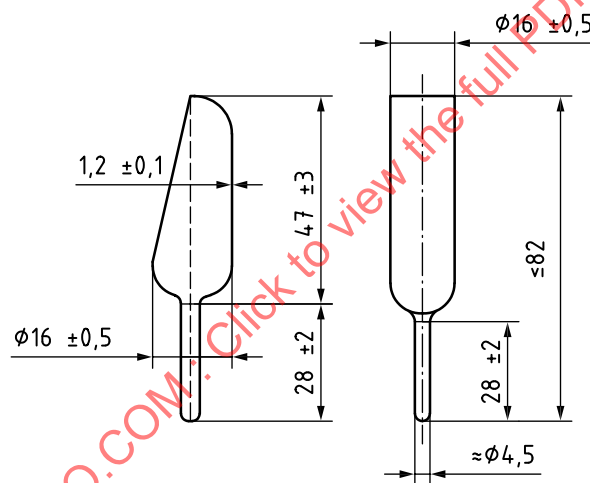


Figure B.1 — Weighing systems

Annex C **(normative)**

Characteristics of stoppers

C.1 General

The purpose of this annex is to define the characteristics of the stoppers.

C.2 Shape and dimensions

Stoppers shall comply with [Figure C.1](#) and with its dimensions.

C.3 Material

The quality of the material used shall be chosen taking into account the products with which the stoppers are in contact (sulfuric acid, amyl alcohol, fat) and the operating temperature.

The hardness of the material used shall be 38 ± 5 international rubber hardness degrees (IRHD), see ISO 48^[1].