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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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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

International Dairy Federation
Silver Building • Bd Auguste Reyers 70/B • B-1030 Brussels
Tel. + 32 2 325 67 40
Fax + 32 2 325 67 41
info@fil-idf.org
www.fil-idf.org

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Contents

Page

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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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 C20 of the Standing Committee on Analytical Methods for Composition under the aegis of its project leader, Mr Philippe Trossat (FR).

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 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 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 ± 0,2 ml of sulphuric acid (5.1).

6.5 Automatic system or pipette, 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, 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 ±70 r/min^{-1}.

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 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:

$1{,}12\ RN^2\ 10^{-6}$

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 °C ± 2 °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 °C ± 2 °C.

6.9 Thermometer, to determine the temperature of the water bath to within ±1 °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 °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 test portion

Weigh into the previously tared weighing system (6.2) 5 g ± 0,005 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.