SLS 735 PART 1 SECTION 9: 2019
(ISO 19660: 2018)
UDC 637.13

METHODS OF TEST FOR MILK AND MILK PRODUCTS

Part 1: Cream-Determination of fat content

Section 9: Acido-butyrometric method

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard

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This Standard does not purport to include all the necessary provisions of a contract

Sri Lanka Standard
METHODS OF TEST FOR MILK AND MILK PRODUCTS
Part 1: Cream- Determination of fat content
Section 9: Acido-butyrometric method

NATIONAL FOREWORD

This Sri Lanka Standard was approved by the sectoral committee on Food products and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2019-03-07.

This Standard specifies an acido-butyrometric method for determining the fat content of cream. 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.

This Standard is identical with **ISO 19660: 2018** Cream- Determination of fat content-Acido-butyrometric method, published by the International Organization for Standardization (**ISO**).

Terminology and Conventions:

The text of the International Standard has been accepted as suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

a) Wherever the words "International Standard" appear referring to this Standard should be interpreted as "Sri Lanka Standard".

b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point on the base line as the decimal marker.

c) Wherever page numbers are quoted, they are **ISO** page numbers.

…….

SLS 735-1-9: 2019

INTERNATIONAL STANDARD

ISO 19660

IDF 237

First edition
2018-02

Cream — Determination of fat content — Acido-butyrometric method

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

Reference numbers
ISO 19660:2018(E)
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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

INTERNATIONAL STANDARD

ISO 19660:2018(E)
IDF 237:2018(E)

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.

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

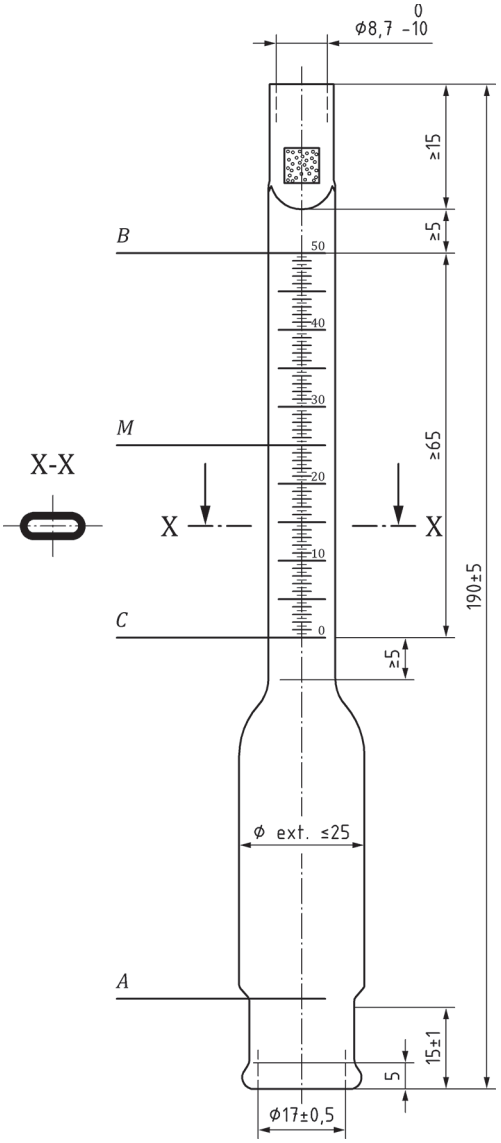
A cream butyrometer is designated by its name followed by the graduation scale and the reference to this document

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

≤ Ø25 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 ± 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 ±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

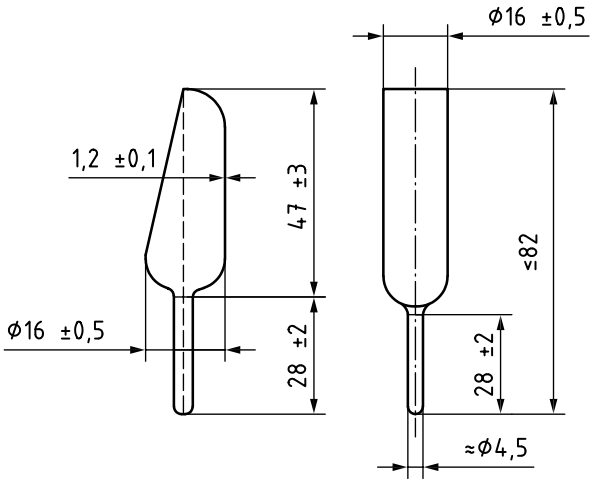


Figure B.1 — Weighing systems

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

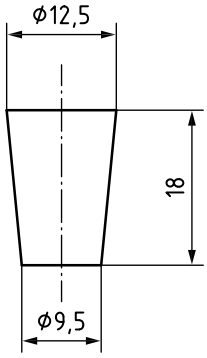
Stoppers shall comply with Figure C.1 and with its dimensions.

C.3 Material

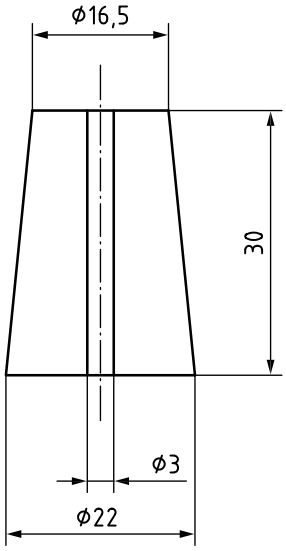
The quality of the material used shall be chosen taking into account the products with which the stoppers are in contact (sulfuric acid, amyl alcohol, fat) and the operating temperature.

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

Dimensions in millimetres



a) For the top of the graduated tube



b) For the neck and the adaptation to the glass container

Figure C.1 — Stoppers for cream butyrometer

Annex D
(informative)

Collaborative trial

An interlaboratory collaborative test involving thirteen laboratories from four countries was carried out on six test samples divided into blind duplicates. The obtained results are expressed in g of fat per 100 g of cream.

The test was organized with test samples prepared and distributed by ACTALIA CECALAIT, Poligny (FR), which also performed the statistical analyses in accordance with ISO 5725-1[4] and ISO 5725-2[5] to give the precision data shown in Table D.1.

Table D.1 — Result of interlaboratory test for fat determination (g of fat/100 g of cream)

Samples	S1	S2	S3	S4	S5	S6	Mean
No. participating laboratories after eliminating outliers [a]	12	12	12	12	11	11	—
Mean value	42,39	37,73	34,65	31,11	27,08	22,14	—
Repeatability standard deviation, s_r	0,17	0,08	0,19	0,11	0,07	0,10	0,126
Repeatability limit, r (2,8 × s_r)	0,46	0,21	0,52	0,30	0,20	0,27	0,35
Reproducibility standard deviation, s_R	0,29	0,24	0,21	0,22	0,23	0,21	0,235
Reproducibility limit, R (2,8 × s_R)	0,80	0,66	0,59	0,60	0,64	0,58	0,65
Coefficient of variation of repeatability, $C_{V,r}$, in %	0,394	0,204	0,544	0,347	0,264	0,444	—
Coefficient of variation of reproducibility, $C_{V,R}$, in %	0,682	0,635	0,613	0,694	0,855	0,954	—

[a] After Grubbs and Cochran 1 % elimination.

Bibliography

[1] ISO 48:2010, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

[2] ISO 707 | IDF 50:2008, *Milk and milk products — Guidance on sampling*

[3] ISO 2450 | IDF 16:2008, *Cream — Determination of fat content — Gravimetric method (Reference method)*

[4] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[5] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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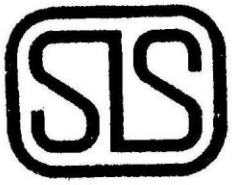

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