SRI LANKA STANDARD 735 : PART 18 : 2017

(ISO 5534:2004)

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METHODS OF
TEST FOR MILK AND MILK PRODUCTS
PART 18: DETERMINATION OF THE TOTAL SOLIDS CONTENT OF CHEESE AND PROCESSED CHEESE (REFERENCE METHOD)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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PART 18: DETERMINATION OF THE TOTAL SOLIDS CONTENT OF CHEESE AND PROCESSED CHEESE (REFERENCE METHOD)

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Sri Lanka.

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

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 18: DETERMINATION OF THE TOTAL SOLIDS CONTENT OF CHEESE AND PROCESSED CHEESE (REFERENCE 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 2017-05-04.

The Standard prescribes the test methods for determining whether the material conforms to the requirements of the relevant individual Standards and thus from a necessary adjunct to series of Sri Lanka Standard Methods of tests for milk and milk products. However, keeping in view the experience gained during the years and various International Standards brought out by the International Organization for Standardization (ISO) on the subject of testing of milk and milk products; it is decided to publish.

In order to accommodate large number of test methods within the scope of one standard, this Standard is published in several Parts.

This Part of the Standard is identical with **ISO 5534: 2004 (IDF 4)** Cheese and processed cheese-Determination of the total solids content (Reference method) published by International Organization for Standardization.

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as a 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, they 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 at the base line as the decimal marker.

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

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SLS 735-18:2017

INTERNATIONAL STANDARD

ISO 5534

IDF 4

Second edition
2004-05-15

Cheese and processed cheese — Determination of the total solids content (Reference method)

Fromages et fromages fondus — Détermination de la teneur totale en matière sèche (Méthode de référence)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 5534 | IDF 4 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

This edition of ISO 5534 | IDF 4 cancels and replaces the first edition of ISO 5534:1985, which has been technically revised.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50% of IDF National Committees casting a vote.

ISO 5534 | IDF 4 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

All work was carried out by the Joint ISO/IDF/AOAC Action Team on *Water*, of the Standing Committee on *Main components in milk*, under the aegis of its project leader, Mr G.J. Beutick (NL).

This edition of ISO 5534 | IDF 4 cancels and replaces the first edition of IDF 4A:1982, which has been technically revised.

INTERNATIONAL STANDARD

ISO 5534:2004(E)
IDF 4:2004(E)

Cheese and processed cheese — Determination of the total solids content (Reference method)

1 Scope

This International Standard specifies the reference method for the determination of the total solids content of cheese and processed cheese.

NOTE This method may not be applicable to processed cheese preparations as defined in the FAO/WHO Code of Principles Standard A-8.

2 Terms and definitions

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

2.1
total solids content of cheese
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The total solids content is expressed as a percentage by mass.

3 Principle

A weighed test portion mixed with sand is dried by heating it in a drying oven at 102 °C. The dried test portion is then weighed to determine the loss of mass.

4 Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water or water of at least equivalent purity.

4.1 **Dilute hydrochloric acid** (HCl), with a mass fraction of 25 %.

4.2 **Quartz sand** or **sea sand**.

4.2.1 The sand shall pass through a woven wire cloth sieve with nominal size of aperture of 600 µm but is retained by a sieve with a nominal size of aperture of 150 µm.

The sand shall comply with the suitability test given in 4.2.2.

4.2.2 Put approximately 20 g of sand in a flat-bottomed dish (5.4) with a stirring rod (5.5). Heat the opened dish with sand, stirring rod and its lid in the oven (5.3) set at 102 °C for at least 2 h. Close the dish and allow to cool in the desiccator (5.2) to the temperature of the balance room. Weigh the closed dish to the nearest 1 mg, recording the mass to four decimal places.

Open the dish and moisten the sand with approximately 5 ml of water. Mix the sand and the water using the rod. Heat the opened dish, stirring rod and its lid in the oven (5.3) set at 102 °C for at least 4 h. Close the dish and allow it to cool in the desiccator (5.2) to the temperature of the balance room. Weigh the closed dish to the nearest 1 mg, recording the mass to four decimal places. The difference between the two weighings shall not exceed 1,0 mg.

4.2.3 If this requirement is not met, treat the sand as follows.

Leave the sand immersed in dilute hydrochloric acid (4.1) for 3 days, stirring occasionally. Decant the supernatant liquid as much as possible. Wash the sand with water until the chloride reaction on the supernatant is negative. Heat the sand at approximately 160 °C for at least 4 h. Then repeat the suitability test as described in 4.2.2.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

5.2 Desiccator, provided with efficient drying agent (e.g. freshly dried silica gel with hygrometric indicator).

Alternatively, a plate of metal or glass, suitable for fast cooling of the dishes may be used. The plate shall be installed in a closed cupboard through which a stream of dried air is passed.

5.3 Forced ventilation drying oven, electrically heated, with ventilation port(s) fully open, capable of being maintained at 102 °C ± 2 °C throughout its working space. The oven shall be fitted with a suitable thermometer.

5.4 Flat-bottomed dishes, made of an appropriate material such as stainless steel, nickel or aluminium, of height 20 mm to 25 mm and diameter 60 mm to 80 mm, provided with well-fitting, readily removable lids.

5.5 Stirring rods, made of glass or metal, with flattened ends, having a length such that each rod can rest at an angle against the inner wall just below the rim of the dish.

5.6 Grinding or **grating devices**, easy to clean, appropriate for preparing the test sample.

6 Sampling

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

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

Store the test samples at a temperature of between 0 °C and 20 °C from the time of sampling to the time of commencing the procedure. During storage of the samples, take care that their composition is not adversely affected.

7 Preparation of test samples

Prior to analysis, remove the rind, the smear or the mouldy surface layer of the cheese in such a way as to obtain a test sample representative of the cheese as it is usually consumed.

Grind or grate the test sample by using an appropriate grinding or grating device (5.6). Mix the ground mass quickly and, if necessary for semi-hard and hard cheeses, grind it a second time and again mix thoroughly.

For hard and semi-hard cheeses, preferably cut these into cubes of about 15 mm × 15 mm × 15 mm. Mix the cubes by shaking in a container. Grind or grate the test sample as described before. Clean the device after preparing each sample.

If the test sample cannot be ground or grated, mix it thoroughly by intensive kneading, for example with a pestle in a mortar. Care shall be taken to avoid moisture loss.

Store the test sample in an airtight container until commencing the analysis, which shall be carried out as soon as possible after grinding.

If, however, a delay is unavoidable, take all precautions to ensure proper preservation of the test sample. If refrigerated, bring the test sample to room temperature. Thoroughly mix the sample to obviate a well-documented transfer of moisture within the cheese that occurs during cooling and warming. Ensure that any condensation of moisture on the inside surface of the container is thoroughly and uniformly re-incorporated into the test sample. Do not examine ground cheese showing unwanted mould growth or signs of deterioration.

8 Procedure

8.1 Blank test

Simultaneously with the determination of the test portion (8.3), carry out a blank test using the same procedure for the preparation of the dish (8.2) and the determination (8.3), but omitting the test portion.

8.2 Preparation of the dish

8.2.1 Heat the opened dish (5.4) containing approximately 20 g of sand (4.2), with its lid and a suitable stirring rod (5.5) in the drying oven (5.3) set at 102 °C. First allow the contents of the dish to reach 102 °C, then dry the contents for 1 h maximum.

The drying period mentioned in 8.2.1, 8.3.3 and 8.3.5 starts at the moment when the temperature of the contents of the dish reaches 102 °C. The time to reach 102 °C depends on the heating capacity, ventilation frequency and size of the oven. It also depends on the number, mass and material of the dishes placed in the oven, and should be determined experimentally.

8.2.2 Place the lid on the dish and immediately transfer the dish to the desiccator (5.2). Allow the dish to cool to room temperature in the closed desiccator. After cooling, open the desiccator and weigh the cooled dish, with its lid and stirring rod, to the nearest 1 mg, recording the mass to four decimal places.

The cooling time mentioned in 8.2.2, 8.3.4 and 8.3.5 depends on the cooling capacity of the desiccator but also on the number, mass and material of the dishes placed in it and should be determined experimentally.

8.3 Determination

8.3.1 Tilt the dish to move the sand to one side. Put about 3,0 g of test sample (Clause 7) on a clear space in the dish and weigh the dish, with its lid and stirring rod, to the nearest 1 mg, recording the mass to four decimal places.

8.3.2 Thoroughly mix together the test portion and the sand, and spread the mixture evenly over the bottom of the dish. Leave the stirring end of the rod in the mixture with the other end resting against the wall of the dish.

NOTE Adding approximately 3 ml of distilled water to saturate the sand can facilitate its mixing with hard cheeses.

8.3.3 Heat the dish, keeping its lid alongside, in the drying oven (5.3) set at 102 °C. First allow the contents of the dish to reach 102 °C, then dry the contents for 3 h maximum.

8.3.4 Place the lid on the dish. Allow the dish to cool to room temperature in the desiccator (5.2). Weigh the closed dish to the nearest 1 mg, recording the mass to four decimal places.

8.3.5 Heat the dish and lid again as described in 8.3.3 but for 1 h instead of 3 h. Place the lid on the dish and allow it to cool to room temperature in the desiccator (5.2). Weigh the closed dish again to the nearest 1 mg, recording the mass to four decimal places.

8.3.6 Repeat the procedure described in 8.3.5 until the mass of the closed dish decreases by 2,0 mg or less, or until it increases between two successive weighings. Record the minimum mass of the dish.

9 Calculation and expression of results

9.1 Calculation

Calculate the total solids content, w_t, of the test sample, expressed as a percentage by mass, using the following equation:

$$w_t = \frac{(m_2 - m_0) - (m_3 - m_4)}{m_1 - m_0} \times 100\ \%$$

where

m_0 is the numerical value of the mass of the prepared dish (8.2.2), in grams;

m_1 is the numerical value of the mass of the test portion and the dish before drying (8.3.1), in grams;

m_2 is the numerical value of the mass of the test portion and the dish after drying (8.3.6), in grams;

m_3 is the numerical value of the mass of the dish used in the blank test (8.1), in grams, for the same drying time (8.3.6) as m_2;

m_4 is the numerical value of the mass of the prepared dish (8.2.2) used in the blank test (8.1), in grams.

9.2 Expression of results

Express the obtained results to two decimal places.

10 Precision

10.1 Interlaboratory test

Details of the interlaboratory test on the precision of the method are given in Annex A. The values derived from this test may not be applicable to concentration ranges and matrices other than those given. In practice, higher values for repeatability and reproducibility might be found for certain types of cheese.

10.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,35 %.

10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,55 %.

11 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained and, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Interlaboratory trial

An international collaborative test involving 12 laboratories was carried out on two different samples of four types of cheese. The thus-obtained 8 test samples were divided again into 16 blind duplicated samples. The test was organized by COKZ (NL). All values are expressed as mass fractions.

The obtained results were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Table A.1.

Table A.1 — Results of interlaboratory tests

	Cheese 1 [a]		Cheese 2 [b]		Cheese 3 [c]		Cheese 4 [d]	
	A	B	A	B	A	B	A	B
No. of participating laboratories after eliminating outliers	12	12	12	12	12	12	12	12
Mean value, %	57,94	58,59	58,87	59,45	46,06	41,38	52,43	51,55
Repeatability standard deviation, s_r, %	0,088	0,096	0,113	0,118	0,102	0,107	0,142	0,125
Coefficient of variation of repeatability, %	0,15	0,16	0,19	0,20	0,22	0,25	0,27	0,24
Repeatability limit, r (= 2,8 s_r), %	0,251	0,273	0,321	0,335	0,288	0,303	0,402	0,353
Reproducibility standard deviation, s_R, %	0,135	0,139	0,150	0,154	0,248	0,253	0,165	0,181
Coefficient of variation of reproducibility, %	0,23	0,24	0,25	0,26	0,54	0,61	0,31	0,35
Reproducibility limit, R (= 2,8 s_R), %	0,383	0,393	0,444	0,457	0,735	0,751	0,489	0,536

[a] Cheese in which a propionic acid fermentation step occurs during the production process.
[b] Gouda cheese.
[c] Processed cheese.
[d] Brie cheese.

Bibliography

[1] ISO 707, *Milk and milk products — Guidance on sampling* [1)]

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

[3] ISO 5725-2, *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*

1) Equivalent to IDF 50.

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