INTERNATIONAL STANDARD

ISO 3433

IDF 222

Second edition
2008-01-15

Cheese — Determination of fat content — Van Gulik method

Fromages — Détermination de la teneur en matière grasse — Méthode Van Gulik

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-a1af-4f8a-a638-f3255b0b4c9b/iso-3433-2008

Reference numbers
ISO 3433:2008(E)
IDF 222:2008(E)

© ISO and IDF 2008

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. Neither the ISO Central Secretariat nor the IDF accepts any liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies and IDF national committees. In the unlikely event that a problem relating to it is found, please inform the ISO Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-a1af-4f8a-a638-f3255b0b4c9b/iso-3433-2008

COPYRIGHT PROTECTED DOCUMENT

© ISO and IDF 2008

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO or IDF at the respective address below.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

International Dairy Federation
Diamant Building • Boulevard Auguste Reyers 80 • B-1030 Brussels
Tel. + 32 2 733 98 88
Fax + 32 2 733 04 13
E-mail info@fil-idf.org
Web www.fil-idf.org

Published in Switzerland

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 3433|IDF 222 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.

This second edition of ISO 3433|IDF 222 cancels and replaces the first edition (ISO 3433:1975), of which it constitutes a minor revision.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ea-a1af-4f8a-a638-f3255b0b4c9b/iso-3433-2008

© ISO and IDF 2008 – All rights reserved

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented at the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO 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 the IDF National Committees casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. IDF shall not be held responsible for identifying any or all such patent rights.

ISO 3433|IDF 222 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the former Joint ISOIDF-AOAC Group of Experts (E31-E301) which is now part of the Joint ISO-IDF Action Team on *Fat*, of the Standing Committee on *Main components in milk*.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-a1af-4f8a-a638-f3255b0b4c9b/iso-3433-2008

© ISO and IDF 2008 – All rights reserved

Cheese — Determination of fat content — Van Gulik method

1 Scope

This International Standard specifies the Van Gulik method for the determination of the fat content, as a mass fraction, of cheese.

This method is applicable to all types of cheese. However, it may not give completely satisfactory results when applied to cheeses with an internal mould (blue-veined cheeses).

NOTE For blue-veined cheeses, see Note to 8.3.11.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-01af-4f8a-a638-f3755b0bc9b/iso-3433-2008

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2446, *Milk — Determination of fat content (Routine method)*

ISO 3432|IDF 221:2007, *Cheese — Determination of fat content — Butyrometer for Van Gulik method*

3 Terms and definitions

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

3.1
Van Gulik method
an empirical procedure which, when applied to a cheese, gives a value for fat content, expressed in grams per 100 g of cheese, that is equivalent to that obtained by the reference method (ISO 1735|IDF 5 [2])

3.2
fat content of cheese
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The fat content is expressed in grams per 100 g, numerically equivalent to a percentage mass fraction.

4 Principle

The protein is dissolved in sulfuric acid, then the fat of the cheese is separated in a Van Gulik butyrometer by centrifuging, the separation being assisted by the addition of a small quantity of amyl alcohol.

The fat content is then read directly from the butyrometer scale.

© ISO and IDF 2008 – All rights reserved

5 Reagents

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

5.1 Sulfuric acid

The sulfuric acid shall have a density at 20 °C of (1,522 ± 0,005) g/ml, which corresponds to a H_2SO_4 mass fraction of 61,72 % to 62,63 %. The acid shall be colourless or no darker in colour than pale amber, and shall not contain any impurity likely to affect results.

5.2 Amyl alcohol

5.2.1 Composition

A volume fraction of at least 98 % of the "amyl"[1)] alcohol shall consist of the primary alcohols pentan-1-ol and 2-methylbutan-1-ol, the only permissible major impurities being 2-methylpropan-1-ol and butan-1-ol. It shall be free from secondary pentanols, 2-methylbutan-2-ol, furan-2-al (furfural, furan-2-carboxaldehyde, 2-furaldehyde), gasoline (petrol), and derivatives of benzene. Not more than a trace of water shall be present.

5.2.2 Physical appearance

The amyl alcohol shall be clear and colourless.

5.2.3 Density

The amyl alcohol shall have a density at 20 °C of 0,808 to 0,818 g/ml.

5.2.4 Furan-2-al and other organic impurities

When 5 ml of the amyl alcohol is added to 5 ml of the sulfuric acid (5.1), no more than a yellow or light brown colour shall develop.

5.2.5 Distillation range

When the amyl alcohol is distilled at a pressure of 101,3 kPa[2)], a volume fraction of not less than 98 % shall distil below 132 °C and a volume fraction of not more than 5 % below 128 °C. There shall be no solid residue after distillation.

If the atmospheric pressure during the distillation is lower or higher than 101,3 kPa, the specified temperatures should be respectively decreased or increased by 3,3 °C/kPa.

5.2.6 Suitability test

Amyl alcohol may satisfy the requirements of 5.2.1 to 5.2.5, yet be unsuitable for the Van Gulik method. Therefore, check the suitability of the amyl alcohol before use by means of the following comparative test with a standard amyl alcohol.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-a1af-4f8a-a048-f3255b0b4c0b/iso-3433-2008

1) Outside the scope of this International Standard, this term, whose use is deprecated by IUPAC, can be applied to the eight isomeric forms of C_5H_{11}.

2) 1 kPa = 10 mbar.

© ISO and IDF 2008 – All rights reserved

5.2.6.1 Standard amyl alcohol

Distil an amyl alcohol satisfying the requirements of 5.2.1 to 5.2.5, using a suitable fractionation column, and collect a fraction within a boiling range of 2 °C between 128,9 °C and 131,5 °C (see Note to 5.2.5). Apply the following tests to the fraction:

a) When analysed by gas-liquid chromatography, a volume fraction of at least 99 % shall consist of 3-methylbutan-1-ol and 2-methylbutan-1-ol. Only traces of impurities other than 2-methylpropan-1-ol and butan-1-ol shall be present.

b) When fractionally distilled, the first 10 % by volume and the last 10 % by volume collected, when compared using the procedure described in 5.2.6.2, shall give values for the fat content of milk that do not differ by more than 0,015 % by mass of fat.

If the fraction satisfies both these tests, it can be regarded as standard amyl alcohol. The standard amyl alcohol can be used for several years, provided that it is kept in the dark in a cool place.

5.2.6.2 Comparison procedure

Determine in duplicate the fat content of four samples of whole milk average fat content by the Gerber method specified in ISO 2446, using butyrometers whose scale errors have been determined, and sulfuric acid of suitable quality. In one of each pair of duplicates, use 1 ml of the amyl alcohol under test and in the other use 1 ml of the standard amyl alcohol (5.2.6.1).

Keep the butyrometers in a random order from the shaking stage onwards. Take the readings to the nearest 0,02 % by mass of fat (read by at least two persons) and correct for the scale errors of the butyrometers.

The mean fat content of the four milk samples obtained with the amyl alcohol under test shall not differ by more than 0,015 % by mass of fat from the mean value obtained using the standard amyl alcohol.

Instead of the amyl alcohol specified, an artificial amyl alcohol or an amyl alcohol substitute, coloured if desired, may be used, provided that it is found to be satisfactory when tested by the procedure described in this subclause.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b2eaa-a1af-4f8a-a638-3255b0b4a9b/iso-3433-2008

6 Apparatus

Usual laboratory apparatus, and in particular the following.

6.1 Van Gulik butyrometers, complying with ISO 3432|IDF 221.

6.2 Weighing device (see ISO 3432|IDF 221, Clause 6), which can be fitted to the large stopper of the butyrometer. Alternatively, a dish, a capsule or plastics sheet may be used.

6.3 Pipette or **automatic measure**, to deliver sulfuric acid (5.1).

6.4 Pipette or **automatic measure**, to deliver (1 ± 0,05) ml of amyl alcohol (5.2).

6.5 Analytical balance, capable of weighing to the nearest 0,001 g.

6.6 Centrifuge, in which the butyrometers can be spun, provided with a rotational frequency indicator, graduated in revolutions per minute, with a maximum tolerance of ± 50 r/min, and preferably of the vertical-loading type rather than the horizontal-loading type.

When fully loaded, the centrifuge shall be capable of producing, within 2 min, a relative centrifugal acceleration of (350 ± 50)g at the outer end of the butyrometer stopper. This acceleration is produced by centrifuges with 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 indicated in Table 1.

© ISO and IDF 2008 – All rights reserved

Table 1 — Centrifuge effective radius and rotational frequency to produce centrifugal acceleration of $(350 \pm 50)g$

Effective radius mm	**Rotational frequency** ± 70 r/min
240	1 140
245	1 130
250	1 120
255	1 110
260	1 100
265	1 090
270	1 080
275	1 070
300	1 020
325	980

NOTE The relative centrifugal acceleration produced in a centrifuge is given by Formula (1):

$$1{,}12 r n^2 \times 10^{-6} \quad (1)$$

where

r is the effective horizontal radius, in millimetres;

n is the rotational frequency, in revolutions per minute.

6.7 Water bath for butyrometers, capable of being maintained at (65 ± 2) °C and such that the butyrometers (6.1) can be supported in a vertical position with their scales completely immersed.

6.8 Thermometer, suitable for insertion in the water bath (6.7).

6.9 Grater, or other device for grinding the cheese.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ca-a1af-4f8a-a638-f3255b0b4c9e/iso-3433-2008

7 Sampling

A representative sample should have been sent to the laboratory. It should not have 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|IDF 50 [1].

8 Procedure

8.1 Preparation of the test sample

Remove the rind, smear or mouldy surface layer of the cheese, in such a way as to provide a test sample representative of the cheese as it is usually consumed. Grind the test sample by means of an appropriate device (6.9). Quickly mix the whole mass and preferably grind the mass again quickly.

If the test sample (e.g. soft cheese) cannot be ground, mix the whole sample thoroughly by intensive stirring and kneading.

Transfer the pretreated sample, or a representative part of it, immediately into a container provided with an airtight lid.

© ISO and IDF 2008 – All rights reserved

Analyse the test sample without delay, as soon as possible after grinding or mixing. If delay is unavoidable, take all precautions to ensure proper preservation of the sample and to prevent condensation of moisture on the inside surface of the container. Ground or mixed cheese showing unwanted mould growth or initial signs of deterioration should not be examined.

Clean the device after grinding each sample.

8.2 Test portion

Weigh, to the nearest 0,005 g, 3,000 g of the test sample (8.1) into the weighing device (6.2) fitted to a suitable stopper, or into a capsule, or on plastics sheet.

8.3 Determination

8.3.1 If a stopper with a weighing device is used, close the neck of the butyrometer (6.1) with this stopper, including weighing device and test portion, and add sulfuric acid (5.1) to the small opening until the acid level reaches a height of about two-thirds of the body of the butyrometer and the weighing device is completely surrounded with sulfuric acid.

If no weighing device in the large stopper is used, close the small opening of the butyrometer with the small stopper and let sulfuric acid run into the butyrometer through the neck until the acid level reaches a height of about half the body of the butyrometer.

Transfer the cheese to the butyrometer. If plastics sheet is used, transfer the cheese with the sheet. Close the neck with the large stopper, invert the butyrometer and remove the small stopper.

8.3.2 Place the butyrometer with its neck (i.e. large opening) downwards in the water bath (6.7) maintained at 65 °C for 5 min.

8.3.3 Remove the butyrometer from the water bath and shake it thoroughly for 10 s.

8.3.4 Repeat the operations specified in 8.3.2 and 8.3.3 until the protein is completely dissolved: 1 h is usually needed. Repeat the procedure for 15 min after the protein has been dissolved.

NOTE Mechanical shaking devices may be used provided that their use produces the same results as the manual procedure specified above.

8.3.5 Remove the butyrometer from the water bath and add, after thorough shaking of the butyrometer, 1 ml of amyl alcohol (5.2) to the small opening. Immediately shake the butyrometer for at least 3 s.

8.3.6 Add sulfuric acid to the small opening until the level reaches the 35 % graduation mark. Close immediately with the small stopper and invert the butyrometer.

8.3.7 As soon as the fat has ascended into the body, shake the butyrometer thoroughly for 10 s. Invert again so that the acid drains out of the stem. Repeat the shaking and inversion twice.

8.3.8 Place the butyrometer neck downwards for 5 min in the water bath; the water level should be above the top of the fat column in the butyrometer.

8.3.9 Remove the butyrometer from the water bath, adjust the large stopper to bring the fat column on the scale and centrifuge the butyrometer at a relative centrifugal acceleration of $(350 \pm 50)g$ for 10 min.

8.3.10 Place the butyrometer neck downwards in the water bath for 5 min. Maintain the water level above the top of the fat column in the butyrometer.

8.3.11 Remove the butyrometer from the water bath and carefully adjust the large stopper to bring the bottom of the fat column, with the minimum movement of the column, to a graduation mark, preferably a main graduation mark. This should preferably be done by slightly withdrawing the stopper and not by forcing it further into the neck. Note the scale reading coincident with the bottom of the fat column and then, taking care

© ISO and IDF 2008 – All rights reserved

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/4a6b23ce-a1a4-4f8a-a63e-f3255d0b4c9b/iso-3433-2008