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Butter — Determination of salt content

Beurre — Détermination de la teneur en sel

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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

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Foreword

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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 1738|IDF 12 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 1738|IDF 12 cancels and replaces ISO 1738:1997, of which it constitutes a minor revision.

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 the National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Group of Experts on *Minerals and minor compounds* (E602), under the aegis of its chairman, Mr G. Bråthen (NO).

This edition of ISO 1738|IDF 12 cancels and replaces IDF 12C:2000, of which it constitutes a minor revision.

Butter — Determination of salt content

1 Scope

This International Standard specifies a method for the determination of the salt content of butter. The method is applicable to all types of butter containing more than 0,1 % (mass fraction) of salt.

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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

3 Terms and definitions

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

3.1
salt content
mass fraction of substances determined by the method specified in this International Standard

NOTE It is expressed as the equivalent content of sodium chloride as a mass fraction in percent.

4 Principle

A test portion of butter is melted by adding boiling water. The dissolved chlorides in the mixture are titrated with a solution of silver nitrate, using potassium chromate as indicator (the Mohr procedure).

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity, free from chlorides.

5.1 Silver nitrate standard volumetric solution ($AgNO_3$), of known concentration in the range 0,08 mol/l to 0,10 mol/l.

Dissolve an amount of between 13,6 g to 20,4 g of silver nitrate in water which is practically free from carbon dioxide in a 1 000 ml volumetric flask. Dilute to the mark with water. Calibrate the silver nitrate solution against 100 ml of a solution containing 0,400 g/l of sodium chloride (NaCl), which has previously been dried at 300 °C,

following the procedure specified in 9.3.2 and 9.4. Express the concentration of the silver nitrate solution in moles per litre to four decimals. Store the solution away from direct sunlight.

NOTE If a solution containing 14,53 g/l (0,085 5 mol/l) of silver nitrate is used, 1 ml of this solution is equivalent to 5 mg of sodium chloride, thus making the calculation of the salt content of the butter easier.

5.2 Potassium chromate indicator solution

Dissolve 50 g of potassium chromate (K_2CrO_4) in 1 000 ml of water.

WARNING — Hexavalent chromium is a carcinogenic agent.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Analytical balance, capable of being read to the nearest 0,001 g.

6.2 Titration vessel, made of glass, for example, a conical flask or beaker of capacity 250 ml.

6.3 Graduated measuring cylinder, of capacity 100 ml, complying with ISO 4788.

6.4 Pipette, capable of delivering 2,0 ml, complying with ISO 648.

6.5 Burette, of capacity 50 ml, complying with ISO 385:1984, Class B.

6.6 Greaseproof paper or **plastic film**, chloride free or of sufficiently low chloride content as to not affect the results. The use of filter paper is not recommended.

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.

Store the sample in such a way that deterioration and change in composition are prevented.

8 Preparation of test sample

Take a representative sample of butter to be tested.

If the test sample is visibly not homogeneous, or if the history of the test sample (age, storage conditions) is such that inhomogeneity is to be expected, mix the test sample as follows. Warm the test sample in the original unopened container, which should be from one-half to two-thirds full, to a temperature that preferably should not exceed 30 °C. At this temperature the sample will be soft enough to facilitate thorough mixing to a homogeneous state (either by a mechanical shaker or by hand).

Cool the sample to ambient temperature, continuing to mix until cooling is complete. Immediately after cooling, open the sample container and stir briefly with a suitable device, for example a spoon or spatula, for no longer than 10 s before weighing.

9 Procedure

9.1 Number of determinations

If it is required to check whether the repeatability limit (11.1) is met, carry out two single determinations in accordance with 9.2 and 9.3.

9.2 Test portion

Weigh, to the nearest 0,05 g, a test portion of between 4,5 g to 5,5 g either directly into the titration vessel (6.2) or on a piece of greaseproof paper or plastic film (6.6) which is transferred with the test sample to the titration vessel. Add 100 ml of boiling water or 100 ml of cold water and heat to boiling. Mix the contents of the vessel.

9.3 Determination

9.3.1 The titration may be carried out on the hot solution or after cooling. It is essential, however, that each laboratory standardize its own procedure for the determination by always bringing the contents of the titration vessel to approximately the same temperature before titration.

The titration should preferably be carried out a temperature of about 50 °C. This temperature may (partially) prevent coagulation of the butterfat which influences the orange tint.

9.3.2 Cool, while mixing, the contents of the titration vessel to the laboratory standardized temperature. Add 2,0 ml of the potassium chromate indicator (5.2).

Titrate the solution with the silver nitrate solution (5.1), with stirring, until an orange tint is obtained which persists for 30 s. Record the volume, in millilitres, of silver nitrate used.

9.4 Blank test

Carry out a blank test using all the reagents but omitting the test portion.

10 Calculation and expression of results

10.1 Calculation

10.1.1 Calculate the salt content of butter, w, expressed as a mass fraction in percent, using the following equation:

$$w = \frac{5{,}844\ c_{\mathrm{s}} \left(V_{\mathrm{s}} - V_0 \right)}{m}\ \%$$

where

V_{s} is the numerical value of the volume, in millilitres, of silver nitrate solution used in the titration of the test portion (9.3.2);

V_0 is the numerical value of the volume, in millilitres, of silver nitrate solution used in the titration of the blank test (9.4);

c_{s} is the numerical value of the concentration of the silver nitrate solution, in moles per litre;

m is the numerical value of the mass, in grams, of the test portion;

5,844 is the mass of NaCl equivalent of 1 ml of standard volumetric solution, $c(AgNO_3)$ = 1 mol/l, divided by a factor 10 [obtained by dividing 100 (%) by 1 000 (ml)].

10.1.2 If a solution containing 14,53 g/l of silver nitrate is used and 5 g of the test portion is weighed to the nearest 0,01 g, the calculation of w, the salt content of butter, may be simplified using the following equation:

$$w = \frac{V_s - V_0}{10} \%$$

where V_s is the numerical value of the volume, in millilitres, of a silver nitrate solution containing 14,53 g/l of silver nitrate.

10.2 Expression of results

Round the result to the nearest 0,01 % (mass fraction).

11 Precision

Details of an interlaboratory test on the precision of the method are summarized in Reference [5]. The study was carried out in accordance with ISO 5725:1986 (now withdrawn). The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

11.1 Repeatability

The absolute difference between two independent single test results, obtained using 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 0,03 % (mass fraction).

11.2 Reproducibility

The absolute difference between two single test results, obtained using 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 0,05 % (mass fraction).

12 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 that may have influenced the test result(s);

e) the test results obtained;

f) if the repeatability has been checked, the final quoted result obtained.

Bibliography

[1] ISO 707:1997, *Milk and milk products — Guidance on sampling*[1)]

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn)

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

[4] 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*

[5] BRÅTHEN, G. and MARTENS, R. *Bulletin of the IDF,* **235**, 1988, pp. 20-33

1) Equivalent to IDF 50.