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Butter — Determination of salt content — Potentiometric method

Beurre — Détermination de la teneur en sel — Méthode potentiométrique

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Foreword

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ISO 15648|IDF 179 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.

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.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team on *Minor compounds*, of the Standing Committee on *Minor components and characterization of physical properties*, under the aegis of its project leader, Mr G. Bräthen (NO).

Butter — Determination of salt content — Potentiometric method

1 Scope

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

NOTE If the method is used for the analysis of butter with a low salt content, the expected relative precision figure will be higher than for salted butter; see also Reference [4].

2 Terms and definitions

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

2.1
salt content of butter
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The salt content, being the equivalent of sodium chloride, is expressed in percent by mass.

3 Principle

A test portion is suspended in water. Chloride ions are titrated potentiometrically with a standard volumetric solution of silver nitrate.

4 Reagents

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

4.1 Silver nitrate, standard volumetric solution, of concentration in the range of $c(AgNO_3)$ = 0,08 mol/l to 0,12 mol/l.

Dissolve 13,6 g to 20,4 g of silver nitrate in water which is practically free from carbon dioxide. Dilute to 1 000 ml. Standardize the silver nitrate standard volumetric solution against sodium chloride (NaCl), previously dried at 300 °C for 2 h.

Express the concentration of the silver nitrate standard volumetric solution to four decimal places. Store the solution protected from direct sunlight.

Commercially available solutions may be used.

4.2 Nitric acid solution, $c(HNO_3) \approx 4$ mol/l.

5 Apparatus

Usual laboratory apparatus 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 Potentiometer, provided with a measuring electrode suitable for the determination of chloride (e.g. a silver electrode) and a reference electrode [e.g. a mercury(I) sulfate electrode].

5.3 Vessel, suitable for blending and titration.

5.4 Graduated cylinders, of capacities 50 ml and 100 ml.

5.5 Burette, of capacity 50 ml, graduated in 0,1 ml, or, preferably, an **automatic plunger burette**, readable to the nearest 0,01 ml, both made of brown glass or protected from light.

5.6 Stirrer.

Suitable (semi) automatic titration apparatus may be used for 5.2 to 5.6.

5.7 Spoon or spatula.

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

7 Preparation of test sample

Take a representative test sample.

If the test sample is visibly not homogeneous, or if the history of the test sample (age, storage conditions) is such that inhomogeneity is expected, homogenize the sample as follows.

a) Warm the test sample in the original unopened container, which should be from one-half to two-thirds full, to a temperature at which the sample will be soft enough to facilitate thorough mixing to a homogeneous state (either by a mechanical shaker or by hand). Take care that the mixing temperature does not exceed 30 °C.

b) Cool the test sample to ambient temperature with constant mixing until cooling is complete. As soon as possible thereafter, open the sample container and stir briefly (no longer than for 10 s) with a suitable device, for example a spoon or spatula (5.7), before weighing.

8 Procedure

8.1 Test portion

Weigh, to the nearest 1 mg, 2 g to 4 g of the test sample (Clause 7) into the vessel (5.3). Record the mass to 0,1 mg.

8.2 Determination

8.2.1 Add 100 ml of boiling water or 100 ml of cold water and heat to boiling to dissolve the test portion. Cool the obtained suspension to below 55 °C.

Put the measuring electrode and the reference electrode of the potentiometer (5.2) into the suspension.

Titrate the suspension with the silver nitrate standard volumetric solution (4.1) using the burette (5.5). Stir continuously until the endpoint has nearly been reached. Then titrate cautiously until the endpoint is reached.

NOTE The endpoint corresponds to the maximum potential difference which is observed between two successive equal additions (of about 0,05 ml) of the silver nitrate standard volumetric solution.

8.2.2 In the case of a difficult titration, the addition of 2 ml to 3 ml of nitric acid solution (4.2) to the contents of the vessel before titration is allowed. By testing this in the collaborative study (see Annex A), it was shown that the same level of precision was attained.

8.3 Blank test

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

9 Calculation and expression of results

9.1 Calculation

Calculate the salt content of the test sample, w, expressed as a percentage by mass, using the following equation:

$$w = \frac{(V_1 - V_0) \times 5{,}84\, c}{m}$$

where

V_0 is the volume of the silver nitrate standard volumetric solution used in the blank test, in millilitres (see 8.3),

V_1 is the volume of the silver nitrate standard volumetric solution used in the determination, in millilitres (see 8.2);

c is the numerical value of the actual concentration of the silver nitrate standard volumetric solution, in moles per litre;

5,84 is the factor transforming the experimentally obtained amount of Cl^- into salt (NaCl);

m is the mass of the test portion, in grams (see 8.1).

9.2 Expression of results

Express the test results to two decimal places.

10 Precision

10.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A.

The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

10.2 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 a mass fraction of 0,05 %.

10.3 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 a mass fraction of 0,08 %.

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 operational 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)

Results of the interlaboratory test

A.1 An international collaborative test involving eleven laboratories was carried out on six butter samples, each divided in two equal samples (blind duplicates) so as to obtain 12 test samples.

The Norwegian Institute for Food and Environmental Analysis organized the test. The results obtained 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.

Based on the obtained results, the repeatability limit, r, was set at 0,05 %. The reproducibility limit, R, was set at 0,08 %

Table A.1 — Interlaboratory test results

	Butter samples						
	A	**B**	**C**	**D**	**E**	**F**	**Mean**[b]
No. of participating laboratories left after eliminating outliers	10	10	10	11	9	11	
Mean value [a]	1,388	1,306	1,051	0,015	2,027	1,295	
Repeatability standard deviation, s_r [a]	0,016	0,017	0,017	0,003	0,016	0,023	0,018
Coefficient of variation of repeatability [a]	1,13	1,43	1,65	20,73	0,77	1,81	1,36
Repeatability limit, r (= 2,8 s_r) [a]	0,044	0,052	0,048	0,008	0,044	0,066	0,051
Reproducibility standard deviation, s_R [a]	0,023	0,041	0,024	0,013	0,025	0,026	0,028
Coefficient of variation of reproducibility [a]	1,62	3,16	2,32	88,80	1,22	2,04	2,07
Reproducibility limit, R (= 2,8 s_R) [a]	0,063	0,115	0,068	0,036	0,069	0,074	0,078

[a] Mass fraction in percent.

[b] Mean values without butter sample D.

A.2 An additional study was organized using a procedure similar to that described in 8.2.1. This study was organized to show whether the addition of nitric acid before titration could be allowed. No significant difference was shown between the results obtained with the unmodified and the modified method.

The test results obtained with the addition of nitric acid (modified method) are shown in Table A.2.