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Caseins and caseinates — Determination of moisture content (Reference method)

Caséines et caséinates — Détermination de la teneur en humidité (Méthode de référence)

Reference numbers
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IDF 78:2006(E)

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Contents

Page

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Foreword

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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 5550 | IDF 78 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 edition of ISO 5550 | IDF 78 cancels and replaces ISO 5550:1978, which has been technically revised.

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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 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.

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ISO 5550 | IDF 78 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 Joint ISO-IDF Action Team *Water*, of the Standing Committee on *Main components in milk*, under the aegis of its project leader, Mrs M. Nicolas (FR).

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Caseins and caseinates — Determination of moisture content (Reference method)

1 Scope

This International Standard specifies the reference method for the determination of the moisture content of all types of caseins and caseinates.

2 Terms and definitions

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

2.1
moisture content
loss of mass determined by the procedure described in this International Standard

NOTE The moisture content is expressed as a mass fraction in percent.

3 Principle

A test portion is dried at 102 °C ± 2 °C then weighed to determine its loss of mass.

4 Apparatus

Usual laboratory equipment and, in particular, the following.

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

4.2 Drying oven, well ventilated, capable of being maintained at a temperature of 102 °C ± 2 °C throughout the working space.

4.3 Flat bottomed dish, made of a material that is non-corrodible under the conditions of the test (e.g. a glass dish with ground-glass cover, or aluminium or stainless-steel dish), of diameter at least 65 mm (preferably 75 mm) and depth of at least 25 mm, equipped with a tight-fitting lid which can be readily removed.

4.4 Desiccator, containing an effective desiccant (e.g. freshly dried silica gel), with hygrometric indicator.

4.5 Grinding device, for grinding the laboratory sample (if necessary, see 6.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

4.6 Test sieve, wire cloth, of diameter 200 mm and nominal size of aperture 500 µm, with receiver.

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5 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.

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

6 Preparation of test sample

6.1 Thoroughly mix the test sample by repeatedly shaking and inverting the container (if necessary, after having transferred all test sample to an airtight container of sufficient capacity to allow this operation to be carried out).

6.2 Transfer about 50 g of thoroughly mixed test sample to the test sieve (4.6).

6.3 If the 50 g of test sample directly passes or almost completely passes through the sieve, for the determination use the sample as prepared in 6.1.

6.4 Otherwise, grind the 50 g of test sample using the grinding device (4.5) until it passes through the sieve. Immediately transfer all the sieved sample to an airtight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting.

During these operations, take precautions to avoid any change in the moisture content of the product.

6.5 After the test sample has been prepared, proceed with the procedure (Clause 7) as soon as possible.

7 Procedure

7.1 Preparation of the dish

7.1.1 Heat the uncovered dish and its lid (4.3) in the drying oven (4.2) set at 102 °C for at least 1 h.

7.1.2 Place the lid on the dish and transfer the covered dish to the desiccator (4.4). Allow the covered dish to cool to the temperature of the balance room. Then weigh it to the nearest 1 mg, recording the mass to 0,1 mg.

7.2 Test portion

7.2.1 Caseins

Transfer approximately 5 g of prepared test sample (6.5) to the dish. Cover the dish with the lid and weigh the whole to the nearest 1 mg, recording the mass to 0,1 mg.

7.2.2 Caseinates

Transfer approximately 2 g of prepared test sample (6.5) to the dish. Cover the dish with the lid and weigh the whole to the nearest 1 mg, recording the mass to 0,1 mg.

7.3 Determination

7.3.1 Uncover the dish (7.2.1 or 7.2.2) and place it with its lid in the drying oven (4.2) set at 102 °C for 3 h.

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7.3.2 Replace the lid on the dish and transfer the covered dish to the desiccator (4.4). Allow the covered dish to cool to the temperature of the balance room. Then weigh it to the nearest 1 mg, recording the mass to 0,1 mg.

7.3.3 Uncover the dish and heat it again with its lid in the drying oven (4.2) set at 102 °C for 1 h. Then repeat step 7.3.2.

7.3.4 Repeat the heating and weighing procedure (7.3.1 to 7.3.3) until the mass of the dish with its lid decreases by 1 mg or less, or increases between two successive weighings. Take for the calculation the lowest mass recorded.

NOTE The total drying time will normally not exceed 6 h.

8 Calculation and expression of results

8.1 Calculation

Calculate the moisture content of the sample, w, using the following equation:

$$w = \frac{(m_1 - m_2)}{(m_1 - m_0)} \times 100\ \%$$

where

w is the moisture content of the test sample, expressed as a mass fraction in percent;

m_0 is the mass of the dish and the lid (7.1.2), in grams;

m_1 is the mass of the dish, the lid and the test portion before drying (7.2.1 or 7.2.2), in grams;

m_2 is the mass of the dish, the lid and the test portion after drying (7.3.4), in grams.

8.2 Expression of results

Report the results to two decimal places.

9 Precision

9.1 Interlaboratory tests

Details of two interlaboratory tests on the precision of the method are summarized in Annex A, and have been published for caseinates [4]. The values derived from these tests may not be applicable to concentration ranges and matrices other than those given.

Repeatability and reproducibility values depend on the size of the grains of casein (see Annex B).

9.2 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:

— for caseins 0,34 %;

— for caseinates 0,32 %.

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9.3 Reproducibility

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

— for caseins 0,53 %;

— for caseinates 0,41 %.

10 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.

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Annex A
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Interlaboratory testing

A.1 Caseins

An international collaborative test involving ten laboratories from eight different countries was carried out on eight caseins: five rennet caseins and three acid ones. The French Agency for Food Safety and Security organized the trial. 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. All values in Table A.1 are expressed as mass fraction in percent.

Table A.1 — Results of interlaboratory test on caseins

	Test samples (for sample identification, see foot of table)								
	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**Mean**
No. of participants after eliminating outliers	10	9	10	10	10	10	10	10	
Mean value, %	9,97	6,82	8,14	8,92	8,50	8,42	7,85	6,78	
Repeatability standard deviation, s_r, %	0,173	0,017	0,131	0,185	0,129	0,155	0,102	0,072	0,12
Coefficient of variation of repeatability, %	1,737	0,247	1,613	2,073	1,512	1,847	1,297	1,055	1,42
Repeatability limit, r (= 2,8 s_r), %	0,490	0,048	0,372	0,523	0,364	0,440	0,288	0,202	0,34
Reproducibility standard deviation, s_R, %	0,296	0,123	0,163	0,232	0,127	0,205	0,198	0,150	0,19
Coefficient of variation of reproducibility, %	2,968	1,802	2,008	2,597	1,494	2,442	2,527	2,210	2,26
Reproducibility limit, R (= 2,8 s_R), %	0,837	0,348	0,463	0,655	0,360	0,582	0,562	0,424	0,53
Samples: A = rennet casein; B = acid casein; C = rennet casein; D = acid casein; E = rennet casein; F = acid casein; G = rennet casein; H = rennet casein.									

A.2 Caseinates

An international collaborative test, organized by the Swiss Dairy Research Institute, was carried out on six double-blind samples of caseinates. The results obtained were subjected to statistical analysis in accordance with ISO 5725[1)] to give the precision data shown in Table A.2. The test results for caseinates have been published [4]. All values in Table A.2 are expressed as mass fraction in percent.

1) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by interlaboratory tests* (now withdrawn), was used to obtain the precision data.

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Table A.2 — Results of interlaboratory test on caseinates

	Test samples (for sample identification, see Table A.1)						
	A	**B**	**C**	**D**	**E**	**F**	**Mean**
No. of participants after eliminating outliers	12	12	12	12	13	12	
Mean value, %	5,54	6,38	6,38	6,98	5,39	8,39	
Repeatability standard deviation, s_r, %	0,16	0,09	0,09	0,15	0,09	0,10	0,11
Coefficient of variation of repeatability, %	2,84	1,48	1,46	2,07	1,63	1,17	1,81
Repeatability limit, r (= 2,8 s_r), %	0,44	0,26	0,26	0,41	0,25	0,27	0,32
Reproducibility standard deviation, s_R, %	0,21	0,12	0,14	0,16	0,11	0,14	0,15
Coefficient of variation of reproducibility, %	3,75	1,89	2,14	2,29	2,11	1,63	2,31
Reproducibility limit, R (= 2,8 s_R), %	0,58	0,34	0,38	0,45	0,32	0,38	0,41

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Annex B
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Influence of the size of the casein grains on precision figures

Table B.1 — Influence of casein grain size on precision figures

	Samples (for sample identification, see foot of table)							
	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Initial mass, g	100,30	100,22	100,29	100,00	100,23	100,24	93,87	100,22
Mass retained by a 500 µm sieve aperture, g	0,38	0,88	1,09	0,32	0,82	0,24	0,93	1,15
(as mass fraction of initial mass, %)	(0,4)	(0,9)	(1,1)	(0,3)	(0,8)	(0,2)	(1,0)	(1,1)
Mass retained by a 315 µm sieve aperture, g	0,78	39,79	40,11	1,44	37,47	0,11	41,40	51,76
(as mass fraction of initial mass, %)	(0,8)	(39,7)	(40,0)	(1,4)	(37,4)	(0,1)	(44,1)	(51,6)
Mass retained by a 250 µm sieve aperture, g	21,92	21,50	26,18	25,60	16,87	5,15	21,18	19,42
(as mass fraction of initial mass %)	(21,9)	(21,5)	(26,1)	(25,6)	(16,8)	(5,1)	(22,6)	(19,4)
Mass retained by a 160 µm sieve aperture, g	31,91	24,79	21,92	33,62	25,06	71,24	19,59	17,95
(as mass fraction of initial mass, %)	(31,8)	(24,7)	(21,9)	(33,6)	(25,0)	(71,1)	(20,9)	(17,9)
Mass passing the 160 µm sieve aperture, g	45,08	12,98	10,83	38,86	19,75	23,31	10,34	9,6
(as mass fraction of initial mass, %)	(44,9)	(13,0)	(10,8)	(38,9)	(19,7)	(23,3)	(11,0)	(9,6)
Repeatability limit, r	0,490	0,048	0,372	0,523	0,364	0,440	0,288	0,202
Reproducibility limit, R	0,837	0,348	0,463	0,655	0,360	0,582	0,562	0,424
Samples: A = rennet casein; B = acid casein; C = rennet casein; D = acid casein; E = rennet casein; F = acid casein; G = rennet casein; H = rennet casein.								
Note that the obtained repeatability and reproducibility values are inversely proportional to the diameter of the casein grains.								

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