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Caseins — Determination of “fixed ash” (Reference method)

Caséines — Détermination des «cendres fixes» (Méthode de référence)

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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.

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ISO 5544 | IDF 89 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 5544 | IDF 89 cancels and replaces the first edition (ISO 5544:1978), of which it constitutes a minor revision.

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.

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ISO 5544 | IDF 89 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 ISO-IDF-AOAC Group of Experts (E11-E701) which is now part of the Joint ISO-IDF Action Team on *Physical properties and rheological tests* of the Standing Committee on *Minor components and characterization of physical properties*.

This edition of ISO 5544 | IDF 89 cancels and replaces IDF 89:1979, of which it constitutes a minor revision.

Caseins — Determination of “fixed ash” (Reference method)

1 Scope

This International Standard specifies a reference method for the determination of the “fixed ash” of caseins, as a percentage by mass, obtained by acid precipitation or lactic fermentation, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type.

NOTE For the determination of ash of rennet caseins and caseinates (except ammonium caseinates), see ISO 5545 | IDF 90[2].

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 648, *Laboratory glassware — Single volume pipettes*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 5550 | IDF 78, *Caseins and caseinates — Determination of moisture content (Reference method)*

3 Terms and definitions

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

3.1
“fixed ash” of caseins
substances determined by the procedure specified in this International Standard

NOTE "Fixed ash" of caseins is expressed as a percentage by mass.

4 Principle

The test portion is incinerated at (825 ± 25) °C in the presence of magnesium acetate to bind all phosphorus of organic origin. The residue is then weighed and the mass of ash originating from magnesium acetate subtracted.

5 Reagents

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

5.1 Magnesium acetate tetrahydrate, $[Mg(CH_3CO_2)_2 \cdot 4H_2O]$, 120 g/l solution.

6 Apparatus

Usual laboratory apparatus, and in particular the following.

6.1 Analytical balance, capable of weighing to the nearest 0,000 1 g.

6.2 One-mark pipette, of capacity 5 ml, complying with the requirements of ISO 648, class A.

6.3 Silica or **platinum dishes**, of diameter about 70 mm and depth 25 mm to 50 mm.

6.4 Drying oven, capable of being controlled at (102 ± 2) °C.

6.5 Electrical furnace, with air circulation, capable of being controlled at (825 ± 25) °C.

6.6 Water bath, capable of being maintained at boiling point.

6.7 Desiccator, containing an effective desiccant.

6.8 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

6.9 Test sieve, wire cloth, of diameter 200 mm, of nominal size of aperture 500 µm, with receiver, complying with ISO 3310-1.

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

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

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.9).

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.8), until it passes the sieve. Immediately transfer all of 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 water content of the product.

8.1.5 After the test sample has been prepared, the determination (8.4) should proceed as soon as possible.

Clean the device after grinding each sample.

8.2 Preparation of the dishes

Heat two dishes (6.3) in the electrical furnace (6.5), maintained at (825 ± 25) °C, for 30 min. Allow the dishes to cool in the desiccator (6.7) to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.3 Test portion

Weigh, to the nearest 0,1 mg, directly in or by difference approximately 3,000 0 g of the test sample (8.1) into one of the prepared dishes, designated dish A.

8.4 Determination

Using the pipette (6.2), add 5 ml of the magnesium acetate solution (5.1) to dish A so as to wet all of the test portion, and allow to stand for 20 min.

Using the pipette (6.2), add 5 ml of the magnesium acetate solution (5.1) to the other prepared dish, designated dish B.

Evaporate the contents of dish A and dish B to dryness on the water bath (6.6).

Place dish A and dish B in the oven (6.4), maintained at 102 °C, for 30 min.

Heat dish A with its contents on a low flame until the test portion is completely charred, taking care that it does not burst into flame.

Transfer dish A and dish B to the electrical furnace (6.5), maintained at 825 °C, and heat for at least 1 h until all carbon has disappeared from dish A. Allow dish A and dish B to cool in the desiccator (6.7) to the temperature of the balance room and weigh to the nearest 0,1 mg.

Repeat the operations of heating in the electrical furnace (6.5), cooling and weighing, until the mass remains constant to within 1 mg or begins to increase. Record the minimum mass.

9 Expression of results

9.1 Calculation

9.1.1 The “fixed ash” of the sample, including phosphorus, w_{fa}, as a percentage by mass, is given by Equation (1):

$$w_{\text{fa}} = \frac{(m_1 - m_2) - (m_3 - m_4)}{m_0} \times 100 \qquad (1)$$

where

m_0 is the mass, in grams, of the test portion (8.3);

m_1 is the mass, in grams, of dish A and residue (8.4);

m_2 is the mass, in grams, of dish A (8.2);

m_3 is the mass, in grams, of dish B and residue (8.4);

m_4 is the mass, in grams, of dish B (8.2).

Calculate the “fixed ash” to the nearest 0,01 % by mass and report the final result to the nearest 0,1 % by mass.

9.1.2 To calculate the "fixed ash" of the sample on the dry basis, as a percentage by mass, multiply the result obtained from Equation (1) by Factor (2)

$$\frac{100}{100 - w_{\mathrm{w}}} \qquad (2)$$

where w_{w} is the water content, as a percentage by mass, of the sample determined according to ISO 5550 | IDF 78.

9.2 Precision

9.2.1 Repeatability

The absolute difference between two independent single 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 0,1 g of "fixed ash" per 100 g of product (numerically equivalent to 0,1 % by mass).

9.2.2 Reproducibility

The absolute difference between two independent 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 0,2 g of "fixed ash" per 100 g of product (numerically equivalent to 0,2 % by mass).

10 Test report

The test report shall specify:

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

b) the sampling method used, if known;

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

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

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

Bibliography

[1] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*

[2] ISO 5545 | IDF 90, *Rennet caseins and caseinates — Determination of ash (Reference method)*

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