INTERNATIONAL STANDARD 5544

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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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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5544 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in September 1976.

It has been approved by the member bodies of the following countries :

Australia	Germany	New Zealand
Austria	Ghana	Poland
Bulgaria	Hungary	Portugal
Canada	India	Romania
Chile	Iran	South Africa, Rep. of
Czechoslovakia	Israel	Spain
Egypt, Arab Rep. of	Korea, Rep. of	Turkey
France	Netherlands	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

NOTE – The method specified in this International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, U.S.A.). The text as approved by the above organizations will also be published by FAO/WHO (Code of Principles concerning Milk and Milk Products and Associated Standards), by the IDF and by the AOAC (Official Methods of Analysis).

Printed in Switzerland

Caseins — Determination of "fixed ash" (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the "fixed ash" of caseins 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.

2 REFERENCES

ISO/R 707, *Milk and milk products — Sampling.*

ISO 3310/I, *Test sieves — Technical requirements and testing — Part I : Metal wire cloth.*

ISO 5550, *Caseins and caseinates — Determination of water content (Reference method).* [1)]

3 DEFINITION

"fixed ash" of caseins : The substances determined by the procedure described in this International Standard and expressed as a percentage by mass.

NOTE — The designation "fixed ash" is used to indicate that the phosphorus of organic origin is retained in the ash.

4 PRINCIPLE

Incineration of a test portion at 825 ± 25 °C in the presence of magnesium acetate to bind all phosphorus of organic origin. Weighing of the residue and subtraction of the mass of ash originating from the magnesium acetate.

5 REAGENT

The reagent shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

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

6 APPARATUS

6.1 Analytical balance.

6.2 One-mark pipette, 5 ml.

6.3 Silica or platinum dishes, about 70 mm diameter and 25 to 50 mm deep.

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 Boiling water bath.

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, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310/I.

7 SAMPLING

See ISO/R 707.

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 air-tight container of sufficient capacity to allow this operation to be carried out).

1) At present at the stage of draft.

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 the sieved sample to an air-tight 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 be proceeded with as soon as possible.

8.2 Preparation of the dishes

Heat two dishes (6.3) in the electrical furnace (6.5), controlled 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 into one of the prepared dishes (A), approximately 3 g of the test sample (8.1).

8.4 Determination

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

To the other prepared dish (B), add with the pipette (6.2) exactly 5 ml of the magnesium acetate solution (5.1).

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

Place both dishes in the oven (6.4), controlled at 102 ± 2 °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 both dishes (A and B) to the electrical furnace (6.5), controlled at 825 ± 25 °C, and heat for at least 1 h until all carbon has disappeared from dish A. Allow both dishes 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 Method of calculation and formula

9.1.1 The "fixed ash" of the sample, including phosphorus, as a percentage by mass, is equal to

$$\frac{(m_1 - m_2) - (m_3 - m_4)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

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

m_2 is the mass, in grams, of the prepared dish A;

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

m_4 is the mass, in grams, of the prepared dish B.

Calculate the "fixed ash" to the nearest 0,01 % and report the final result to the nearest 0,1 %.

9.1.2 To calculate the "fixed ash" of the sample on the dry basis, as a percentage by mass, multiply the result obtained in accordance with 9.1.1 by

$$\frac{100}{100 - M}$$

where M is the water content of the sample determined according to ISO 5550.

9.2 Precision

9.2.1 *Repeatability*

The difference between two single results obtained on identical test material by one analyst using the same apparatus within a short time interval will exceed 0,1 g of "fixed ash" per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9.2.2 *Reproducibility*

The difference between two single and independent results obtained by two operators working in different laboratories on identical test material will exceed 0,2 g of "fixed ash" per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

10 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.