
INTERNATIONAL STANDARD



5544

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

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

First edition — 1978-06-15

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UDC 637.147.2 : 543.822

Ref. No. ISO 5544-1978 (E)

Descriptors : caseins, chemical analysis, determination of content, ashes, gravimetric analysis.

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).

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 $825 \pm 25^\circ\text{C}$ 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

[$\text{Mg}(\text{CH}_3\text{CO}_2)_2 \cdot 4\text{H}_2\text{O}$], 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 $102 \pm 2^\circ\text{C}$.

6.5 Electrical furnace with air circulation, capable of being controlled at $825 \pm 25^\circ\text{C}$.

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.