INTERNATIONAL STANDARD 5547

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Caseins — Determination of free acidity (Reference method)

Caséines — Détermination de l'acidité libre (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 5547 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 free acidity (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the free acidity of caseins obtained by acid precipitation or lactic fermentation and of rennet caseins.

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

free acidity of caseins : Volume, in millilitres, of a 0,1 N standard volumetric sodium hydroxide solution required to titrate an aqueous extract of 1 g of the product.

4 PRINCIPLE

Aqueous extraction of a test portion at 60 °C. Filtration. Titration of the filtrate with a standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

5 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled or deionized water, freed from carbon dioxide by boiling for 10 min before use.

5.1 Sodium hydroxide, approximately 0,1 N standard volumetric solution.

5.2 Phenolphthalein, 10 g/l ethanolic solution.

6 APPARATUS

6.1 Analytical balance.

6.2 Conical flask, 500 ml capacity, with ground neck and fitted with a ground glass stopper.

6.3 One-mark pipette, 100 ml capacity.

6.4 Pipette, suitable for measuring 0,5 ml of indicator solution (5.2).

6.5 Conical flask, 250 ml capacity.

6.6 Measuring cylinder, 250 ml capacity.

6.7 Burette, graduated in 0,1 ml.

6.8 Water bath, capable of being controlled at a temperature of 60 ± 2 °C.

6.9 Appropriate filter.

6.10 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.

6.11 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).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.11).

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.

1) At present at the stage of draft.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.10), 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 (8.3) should be proceeded with as soon as possible.

8.2 Test portion

Weigh about 10 g of the test sample (8.1) to the nearest 10 mg and transfer it to the conical flask (6.2).

8.3 Determination

Using the 250 ml measuring cylinder (6.6), add 200 ml of freshly boiled water, previously heated to 60 °C. Stopper the flask, mix by swirling and place in the water bath at 60 °C (6.8) for 30 min. Shake the flask at intervals of about 10 min.

Filter, and cool the filtrate to about 20 °C. The filtrate must be clear.

Transfer 100 ml of the cooled filtrate into the conical flask (6.5), using the pipette (6.3). Add 0,5 ml of the ethanolic phenolphthalein solution (5.2), using the pipette (6.4). Titrate with the standard volumetric sodium hydroxide solution (5.1), until the appearance of a faint pink colour, persisting for at least 30 s. Record the volume used to the nearest 0,01 ml.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

9.1.1 The free acidity of the casein is equal to

$$\frac{20 \times V \times T}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (5.1) used;

T is the normality of the standard volumetric sodium hydroxide solution (5.1);

m is the mass, in grams, of the test portion.

Calculate the free acidity to the nearest 0,01.

9.1.2 To calculate the free acidity of the sample on the dry basis, 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 0,02 ml of 0,1 N sodium hydroxide solution per 1 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 0,04 ml of 0,1 N sodium hydroxide solution per 1 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.