
International Standard



5546

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

Caseins and caseinates — Determination of pH (Reference method)

Caséines et caséinates — Détermination du pH (Méthode de référence)

First edition — 1979-10-01

iTeh STANDARD PREVIEW
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UDC 637.147.2 : 543.257.1

Ref. No. ISO 5546-1979 (E)

Descriptors : caseins, chemical analysis, pH.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 5546 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, F. R.	Poland
Austria	Ghana	Portugal
Bulgaria	Hungary	Romania
Canada	India	South Africa, Rep. of
Chile	Iran	Spain
Czechoslovakia	Israel	Thailand
Egypt, Arab Rep. of	Korea, Rep. of	Turkey
France	Netherlands	Yugoslavia

The member bodies of the following countries expressed disapproval of the document on technical grounds :

New Zealand
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 and caseinates — Determination of pH (Reference method)

1 Scope and field of application

This International Standard specifies a reference method for the determination of the pH of all types of casein (acid caseins and rennet caseins) and of caseinates.

2 References

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

ISO 3310/1, *Test sieves — Technical requirements and testing — Part 1: Metal wire cloth*.

3 Definition

pH of caseins and caseinates: The pH, at 20 °C, of an aqueous extract of casein or an aqueous solution of caseinate as determined by the procedure described in this International Standard.

4 Principle

Electrometric determination of the pH of an aqueous extract of casein or an aqueous solution of caseinate, using a pH meter.

5 Reagents

The reagents shall be of recognized analytical grade. Any water used in their preparation and the water used in the determination (8.2) shall be recently distilled water that has been protected from carbon dioxide absorption.

5.1 Buffer solutions, for calibration of the pH meter (6.2).

Two standard buffer solutions with pH values at 20 °C which are known to the second decimal place and will bracket the pH value of the sample under test, for example phthalate buffer solution of pH approximately 4 and a borax buffer solution of pH approximately 9. In addition, a phosphate buffer solution of pH approximately 7 may be used.

6 Apparatus

6.1 Balance, accuracy 0,1 g.

6.2 pH meter, minimum sensitivity 0,05 pH unit, with a suitable glass electrode and a calomel or other reference electrode.

6.3 Thermometer, accuracy 0,5 °C.

6.4 Conical flask, capacity 100 ml, fitted with a ground glass stopper.

6.5 Beaker, capacity 50 ml.

6.6 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.

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

6.8 Mixer.

6.9 Beaker, for the mixer (6.8), of at least 250 ml capacity.

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 (6.7).

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 (6.6), until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeated 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 (8.2) should be proceeded with as soon as possible.

8.2 Determination

8.2.1 Calibration of pH meter

Adjust the temperature of the buffer solutions (5.1) to 20 °C and calibrate the pH meter in accordance with the manufacturer's instructions.

NOTES

1 The calibration should be carried out while the flasks are standing for 20 min (see 8.2.2.1 and 8.2.2.2).

2 If a series of samples is being tested, check the calibration of the pH meter with one or more of the standard buffer solutions at least every 30 min.

8.2.2 Preparation of test solution

8.2.2.1 Caseins

Weigh to the nearest 0,1 g, into the conical flask (6.4), 5,0 g of the test sample (8.1), add 30 ml of water previously adjusted to 20 °C and stopper the flask. (If desired, 7,0 g of the test sample and 42 ml of water can be taken.)

Shake the flask by hand for 10 s and allow to stand for 20 min at about 20 °C.

8.2.2.2 Caseinates

Transfer to the beaker (6.9) 95 ml of water, add 5,0 g of the test sample (8.1), and mix using the mixer (6.8) for 30 s.

Allow to stand for 20 min at about 20 °C.

8.2.3 Measurement of pH

8.2.3.1 Caseins

Decant the supernatant liquid into the beaker (6.5) and immediately determine the pH of this liquid, using the pH meter (6.2), after having rinsed the glass electrode carefully with water.

8.2.3.2 Caseinates

Pour about 20 ml of the solution into the beaker (6.5) and immediately determine the pH of this liquid, using the pH meter (6.2), after having rinsed the glass electrode carefully with water.

9 Expression of results

9.1 Recording of pH

9.1.1 Caseins

Record, as the pH of the aqueous extract of casein, the value read from the dial of the pH meter to at least one decimal place.

9.1.2 Caseinates

Record, as the pH of the aqueous solution of caseinate, the value read from the dial of the pH meter to at least two decimal places.

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 pH unit for casein extracts and 0,05 pH unit for caseinate solutions 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 0,3 pH unit for casein extracts and 0,15 pH unit for caseinate solutions 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.

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