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**Caseins and caseinates — Determination  
of pH (Reference method)**

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

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## Foreword

**ISO (the International Organization for Standardization)** is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5546|IDF 115 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 5546|IDF 115 cancels and replaces the first edition (ISO 5546:1979), of which it constitutes a minor revision.

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## 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 on 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.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50% of IDF National Committees casting a vote.

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ISO 5546|IDF 115 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 Action Team on *Minor compounds*, now part of the Standing Committee on *Analytical methods for composition*.

This edition of ISO 5546|IDF 115 cancels and replaces IDF 115A:1989, of which it constitutes a minor revision.

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# Caseins and caseinates — Determination of pH (Reference method)

## 1 Scope

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 Terms and definitions

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

### 2.1

#### **pH of caseins and caseinates**

pH, at 20 °C, of an aqueous extract of casein or an aqueous solution of caseinate, as determined by the procedure specified in this International Standard

## 3 Principle

The pH of an aqueous extract of casein or an aqueous solution of caseinate is determined electrometrically using a pH meter.

## 4 Reagents

Unless otherwise stated, use only reagents of recognized analytical grade and recently distilled water that has been protected from carbon dioxide absorption.

### 4.1 **Buffer solutions**, for calibration of the pH meter (5.2).

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

## 5 Apparatus

Usual laboratory equipment and in particular the following.

### 5.1 **Balance**, accurate to the nearest 0,1 g.

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

### 5.3 **Thermometer**, accurate to the nearest 0,5 °C.

- 5.4 **Conical flask**, capacity 100 ml, fitted with a ground-glass stopper.
- 5.5 **Beaker**, capacity 50 ml.
- 5.6 **Grinding device**, for grinding the laboratory sample, if necessary (see 7.4), without producing undue heat and loss or absorption of moisture. A hammer mill shall not be used.
- 5.7 **Test sieve**, wire cloth, diameter 200 mm, nominal size of openings 500 µm, with receiver, ISO 3310-1<sup>[2]</sup>.
- 5.8 **Mixer**.
- 5.9 **Beaker**, for the mixer (5.8), capacity at least 250 ml.

## 6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50<sup>[1]</sup>.

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

## 7 Preparation of test sample

- 7.1 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).
- 7.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (5.7).
- 7.3 If the 50 g portion passes through the sieve completely or almost completely, use for the determination the sample as prepared in 7.1.
- 7.4 Otherwise, grind the 50 g portion, using the grinding device (5.6), until it passes through the sieve. Immediately transfer all 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.
- 7.5 After the test sample has been prepared, initiate the procedure (see Clause 8) as soon as possible.

## 8 Procedure

### 8.1 Calibration of pH meter

Adjust the temperature of the buffer solution (4.1) to 20 °C and calibrate the pH meter in accordance with the manufacturer's instructions.

The calibration should be carried out while the flasks are standing for 20 min (see 8.2.1 and 8.2.2).

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 Preparation of test solution

### 8.2.1 Caseins

Weigh, to the nearest 0,1 g, into the conical flask (5.4), 5,0 g of the test sample (see Clause 7), 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 may be taken.)

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

### 8.2.2 Caseinates

Transfer to the beaker (5.9) 95 ml of water, add 5,0 g of the test sample (see Clause 7), and mix using the mixer (5.8) for 30 s.

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

## 8.3 Measurement of pH

### 8.3.1 Caseins

Decant the supernatant liquid into the beaker (5.5) and immediately determine the pH of this liquid, using the pH meter (5.2), after having rinsed the glass electrode carefully with water.

### 8.3.2 Caseinates

Pour about 20 ml of the solution into the beaker (5.5) and immediately determine the pH of this liquid, using the pH meter (5.2), after having rinsed the glass electrode carefully with water.

## 9 Expression of results

### 9.1 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.

### 9.2 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.

## 10 Precision

### 10.1 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.

## 10.2 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.

## 11 Test report

The test report shall contain at least the following information:

- a) all the information necessary for the complete identification of the sample;
- b) the sampling method used, if known;
- c) the test method used, including a reference to this International Standard (ISO 5546|IDF 115:2010);
- d) any operating conditions not specified in this International Standard, or regarded as optional, as well as details of any incidents that may have influenced the result(s);
- e) the test result(s) obtained;
- f) if the repeatability has been checked, the final quoted result obtained.

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