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Caseins and caseinates — Determination of lactose content — Photometric method

Caséines et caséinates — Détermination de la teneur en lactose — Méthode photométrique

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Foreword

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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.

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ISO 5548|IDF 106 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

This edition of ISO 5548|IDF 106 cancels and replaces ISO 5548:1980, of which it constitutes a minor revision. Only editorial changes have been made.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of the National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Group of Experts, *Methods for caseins and caseinates* (E36), under the aegis of its project leader, Mr J. Eisses (NL).

This edition of ISO 5548|IDF 106 cancels and replaces IDF 106:1982. Only editorial changes have been made.

Caseins and caseinates — Determination of lactose content — Photometric method

1 Scope

This International Standard specifies a photometric method for the determination of the content of lactose and other soluble carbohydrates in caseins and caseinates containing less than 2,0 % of total soluble carbohydrates.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

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

3.1
lactose content of caseins and caseinates
content of total soluble carbohydrates, expressed as anhydrous lactose, determined by the procedure specified in this International Standard

NOTE It is expressed as a mass fraction in percent.

4 Principle

A test portion is dissolved

a) in hot water in the case of caseinates;

b) in hot water with the addition of sodium hydrogen carbonate in the case of acid caseins;

c) in hot water with the addition of pentasodium triphosphate in the case of rennet casein.

The casein is precipitated with a solution of acetic acid and sodium acetate at pH 4,6, then filtered to obtain a protein-free solution of the carbohydrates. Phenol solution and concentrated sulfuric acid are added to an aliquot portion of the filtrate, thus producing a colour which is proportional to the amount of carbohydrate present, which is measured photometrically at a wavelength of 490 nm.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

5.1 Sodium hydrogen carbonate ($NaHCO_3$), for analysis of acid casein.

5.2 Pentasodium triphosphate ($Na_5P_3O_{10}$), for analysis of rennet casein.

5.3 Dilute hydrochloric or **sulfuric acid**, c(HCl) or c(1/2 H_2SO_4) = 0,1 mol/l.

5.4 Dilute acetic acid, $c(CH_3CO_2H)$ = 100 g/l.

5.5 Sodium acetate solution, $c(CH_3COONa)$ = 1 mol/l.

5.6 Phenol solution, 80 % (mass fraction).

Heat a mixture of 8 g of phenol and 2 g of water until the mixture is homogeneous.

5.7 Sulfuric acid, concentrated, ρ_{20} (H_2SO_4)= 1,84 g/ml.

5.8 Lactose standard solution, $\rho_{20}(C_{12}H_{22}O_{11})$ = 20 g/l.

Weigh 2,105 g ± 0,001 g of lactose monohydrate, corresponding to 2,00 g of anhydrous lactose, into a 100 ml volumetric flask. Dissolve in water, make up to the mark with water and mix well. Store the obtained standard solution at 0 °C.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 1 mg.

6.2 Conical flasks, of capacity 100 ml.

6.3 One-mark pipettes, of capacity 1 ml, 2 ml and 10 ml.

6.4 Micropipettes, of capacity 0,2 ml, with 0,001 ml divisions.

6.5 Graduated pipettes, of capacity 25 ml.

6.6 Test tubes, of capacity about 40 ml, with ground necks and fitted with ground glass stoppers.

6.7 Automatic dispenser, capable of dispensing 5 ml of concentrated sulfuric acid within 1 s.

6.8 Water bath, capable of being maintained at 60 °C to 70 °C.

6.9 Photometer, suitable for making measurements at a wavelength of 490 nm, provided with cells of optical path length 1 cm to 2 cm.

6.10 Mixer, suitable for mixing inside the test tubes (6.6), with a stirrer resistant to strong acid.

6.11 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss of moisture. A hammer-mill shall not be used.

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

6.13 Volumetric flasks, of capacity 100 ml.

6.14 Water bath, capable of being maintained at 20 °C.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707[1].

8 Procedure

8.1 Preparation of 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 airtight 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 (6.12).

8.1.3 If the 50 g portion passes completely or almost completely through the sieve, use the sample prepared in 8.1.1 for the determination.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.11), 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.

8.1.5 After the test sample has been prepared, carry out the determination (8.5) as soon as possible.

8.2 Preparation of a blank solution

Prepare a blank solution containing 0,1 g ± 0,001 g of sodium hydrogen carbonate (5.1) or 0,1 g ± 0,001 g of pentasodium triphosphate (5.2), as appropriate, using the same apparatus, the same reagents in the same amounts, and the same procedure as described in 8.4.2 to 8.5.1 inclusive, but omitting the test portion and omitting those operations in connection with the presence of a test portion.

For the most accurate results, prepare the blank solution, the test solution and the lactose standard working solutions for the calibration graph (see 8.6) simultaneously.

8.3 Test portion

Weigh, to the nearest 1 mg, about 1 g of the test sample (8.1) into a conical flask (6.2).

8.4 Test solution

8.4.1 In the case of acid casein, add 0,1 g ± 0,001 g of the sodium hydrogen carbonate (5.1).

In the case of rennet casein, add 0,1 g ± 0,001 g of the pentasodium triphosphate (5.2).