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Caseins and caseinates — Determination of nitrate and nitrite contents — Method by cadmium reduction and spectrometry

Caséines et caséinates — Détermination des teneurs en nitrates et en nitrites — Méthode par réduction au cadmium et spectrométrie

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8195 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

NOTE — The method specified in this International Standard has been developed jointly with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC) and will also be published by these organizations.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Caseins and caseinates — Determination of nitrate and nitrite contents — Method by cadmium reduction and spectrometry

1 Scope and field of application

This International Standard specifies a method by cadmium reduction and spectrometry for the determination of the nitrate and nitrite contents of caseins and caseinates.

2 Reference

ISO 707, *Milk and milk products — Methods of sampling.*

3 Definition

nitrate and nitrite contents of caseins and caseinates: The contents of substances determined by the procedure specified in this International Standard and expressed respectively as milligrams of nitrate ion (NO_3^-) and of nitrite ion (NO_2^-) per kilogram.

4 Principle

Dispersion of the casein or caseinate in warm water, precipitation of the fat and proteins, and filtration.

Reduction of the nitrate to nitrite in a portion of the filtrate by means of copperized cadmium.

Development of a red colour, in portions of both unreduced filtrate and of the reduced solution, by addition of sulfanilamide and *N*-1-naphthyl ethylenediamine dihydrochloride, and spectrometric measurement at a wavelength of 538 nm.

Calculation of the nitrite content of the sample and of the total nitrite content after reduction of nitrate, by comparing the measured absorbances with those of a set of sodium nitrite calibration solutions; calculation of the nitrate content from the difference between these two contents.

5 Reagents

All reagents shall be of recognized analytical grade. The water used shall be distilled or deionized water, free from nitrate and nitrite.

NOTE — In order to avoid possible inclusion of small gas bubbles in the copperized cadmium column (6.10), the distilled or deionized water used for the preparation of the column (8.1), for checking the reducing capacity of the column (8.2), and for regeneration of the column (8.3) should preferably be freshly boiled and afterwards cooled to room temperature.

5.1 Cadmium, granules, diameter 0,3 to 0,8 mm.

If cadmium granules are not available commercially, they may be prepared as follows.

Place a suitable number of zinc rods in a beaker and cover with a 40 g/l solution of cadmium sulfate octahydrate ($CdSO_4 \cdot 8H_2O$). From time to time scrape the cadmium sponge from the rods over a period of 24 h. Remove the zinc rods and decant the liquid until only sufficient remains to cover the cadmium. Wash the sponge two or three times with water. Transfer the cadmium to a laboratory blender together with 400 ml of hydrochloric acid solution, $c(HCl) \approx 0{,}1$ mol/l, and blend for a few seconds to obtain granules of the required size. Return the contents of the blender to the beaker and leave to stand for several hours, occasionally stirring to remove bubbles. Decant most of the liquid and immediately copperize as described in 8.1.1 to 8.1.5.

5.2 Copper(II) sulfate solution.

Dissolve 20 g of copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in water and dilute to 1 000 ml.

5.3 Hydrochloric acid solution, $c(HCl) \approx 2$ mol/l.

Dilute 160 ml of concentrated hydrochloric acid (ϱ_{20} 1,19 g/ml) to 1 000 ml with water.

5.4 Hydrochloric acid solution, $c(HCl) \approx 0{,}1$ mol/l.

Dilute 50 ml of the hydrochloric acid (5.3) to 1 000 ml with water.

5.5 Solutions for precipitation of proteins and fat.

5.5.1 Zinc sulfate solution.

Dissolve 53,5 g of zinc sulfate heptahydrate ($ZnSO_4 \cdot 7H_2O$) in water and dilute to 100 ml.

5.5.2 Potassium hexacyanoferrate(II) solution.

Dissolve 17,2 g of potassium hexacyanoferrate(II) trihydrate ($K_4[Fe(CN)_6] \cdot 3H_2O$) in water and dilute to 100 ml.