

INTERNATIONAL STANDARD

ISO
6739

Second edition
1988-09-01



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

Whey cheese — Determination of nitrate and nitrite contents — Method by cadmium reduction and spectrometry

Fromage de sérum — Détermination des teneurs en nitrates et en nitrites — Méthode par réduction au cadmium et spectrométrie

Reference number
ISO 6739: 1988 (E)

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.

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 6739 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, in collaboration with the International Dairy Federation (IDF) and the Association of Analytical Chemists (AOAC) and will also be published by these organizations.

This second edition cancels and replaces the first edition (ISO 6739 : 1982), of which it constitutes a minor revision.

Whey cheese — Determination of nitrate and nitrite contents — Method by cadmium reduction and spectrometry

1 Scope

This International Standard specifies a method for the determination of the nitrate and nitrite contents of whey cheese.

The method is suitable for all kinds of whey cheese.

The detection limits of the method are 5 mg of nitrate per kilogram and 0,5 mg of nitrite per kilogram.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 707 : 1985, *Milk and milk products — Methods of sampling*.

3 Definition

For the purposes of this International Standard, the following definition applies.

nitrate and nitrite contents of whey cheese : The mass fractions of substances determined by the procedure specified in this International Standard.

They are expressed respectively as milligrams of nitrate ion (NO_3^-) and of nitrite ion (NO_2^-) per kilogram.

4 Principle

Extraction of the whey cheese with warm water, precipitation of the fat and proteins, and filtration.

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

Development of a red colour, in portions of both the unreduced filtrate and 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 series of standard sodium nitrite 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 (6.11), the distilled or deionized water used for the preparation of the column (9.1), for checking the reducing capacity of the column (9.2) and for regeneration of the column (9.3) should preferably be freshly boiled and afterwards cooled to room temperature.

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

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 ($\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$). 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 distilled water. Transfer the cadmium to a laboratory blender together with 400 ml of 0,1 mol/l hydrochloric acid solution 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 9.1.1 to 9.1.5.

5.2 Copper(II) sulfate solution.

Dissolve 20 g of copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 1000 ml.

5.3 Buffer solution, pH 9,6 to 9,7.

Dilute 50 ml of concentrated hydrochloric acid [ρ_{20} 1,19 g/ml, about 38 % (*m/m*) solution] with 600 ml of water. After mixing,