
International Standard



6740

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Dried whey — Determination of nitrate and nitrite contents — Method by cadmium reduction and spectrometry

Lactosérum en poudre — Détermination des teneurs en nitrates et en nitrites — Méthode par réduction au cadmium et spectrométrie

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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.

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 6740 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.

Dried whey — 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 dried whey.

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

NOTE — Methods for the determination of the nitrate and nitrite contents of dried milk and whey cheese are specified in ISO 6736 and ISO 6739 respectively.

2 Reference

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

3 Definition

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

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

4 Principle

Dissolution of the dried whey in 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 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 series 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 ($\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$). 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 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 ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 1 000 ml.

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

Dilute 50 ml of concentrated hydrochloric acid [d_{20} 1,19 g/ml, about 38 % (*m/m*) solution] with 600 ml of water. After mixing, add 135 ml of ammonium hydroxide solution [d_{20} 0,91 g/ml, about 25 % (*m/m*) solution]. Dilute to 1 000 ml with water and mix.