Nederlandse

Ontwerp

norm

NEN-EN-ISO 14673-1

Melkproducten - Bepaling van het nitraat- en nitrietgehalte - Deel 1: Methode met cadmiumreductie en spectrometrie (ISO/DIS 14673-1:1999)

Publicatie uitsluitend voor commentaar

Milk products - Determination of nitrate and nitrite content - Part 1: Method using cadmium reduction and spectrometry (ISO/DIS 14673-1:1999)

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Normcommissie 370 005 "Melk en zuivelproducten"

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DRAFT INTERNATIONAL STANDARD ISO/DIS 14673-1

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Milk products — Determination of nitrate and nitrite content —

Part 1:
Method using cadmium reduction and spectrometry

Produits laitiers — Détermination de la teneur en nitrates et nitrites —
Partie 1: Méthode par réduction au cadmium et spectrométrie

ICS 67.100.01

ISO/CEN PARALLEL ENQUIRY

The CEN Secretary-General has advised the ISO Secretary-General that this ISO/DIS covers a subject of interest to European standardization. **In accordance with subclause 5.1 of the Vienna Agreement, consultation on this ISO/DIS has the same effect for CEN members as would a CEN enquiry on a draft European Standard.** Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month FDIS vote in ISO and formal vote in CEN.

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 3.

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.

This part of ISO 14673 was prepared by Technical Committee ISO/TC 34, Agricultural food products, Subcommittee SC 5, Milk and milk products, in collaboration with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC), and will also be published by these organizations.

1 Scope

This part of ISO 14673 specifies a method for the determination of the nitrate and nitrite content in milk products by cadmium reduction and spectrometry.

The method is applicable to whole, partly skimmed and skimmed dried milk; hard, semi-hard and soft cheeses; processed cheese; whey cheese, caseins and caseinates and dried whey.

NOTE The method can be performed using automatic equipment, in particular by segmented flow analysis (SFA) or flow injection analysis (FIA), thus reducing cadmium contamination of laboratory work place and waste water. These methods are described in part 2 and part 3 of ISO 14673 respectively.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreement based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings.*

ISO 648:1977, *Laboratory glassware - One-mark pipettes.*

ISO 835-1/4:1981, *Laboratory glassware - Graduated pipettes.*

ISO 1042:1983, *Laboratory glassware - One-mark volumetric flasks*

3 Term(s) and definition(s)

For the purposes of this part of ISO 14673 the following definition applies.

3.1
Nitrate content:
mass fraction of substances determined by the procedure specified in this part of this International Standard.

NOTE The nitrate content is expressed as the mass in milligrams of nitrate-ion (NO_3^-) per kilogram.

3.2
Nitrite content:
mass fraction of substances determined by the procedure specified in this International Standard.

NOTE The nitrite content is expressed as the mass in milligrams of nitrite-ion (NO_2^-) per kilogram.

4 Principle

Dispersion of a test portion 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

Use only reagents of recognized analytical grade unless otherwise specified, and distilled or deionized water or water of equivalent purity, free from nitrate and nitrite.

To avoid possible inclusion of small gas bubbles in the copperized cadmium column (9.1.8), freshly boil the distilled or deionized water and cool to room temperature. Use the thus prepared water for the preparation of the column (9.1), to check the reducing capacity of the column (9.2), and to regenerate the column (9.3).

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

Prepare cadmium granules, not being available commercially, as follows.

Place a suitable number of zinc rods in a beaker. Cover the rods with cadmium-sulfate solution (5.2). Scrape the cadmium sponge from the rods from time to time over a period of 24 h. Remove the zinc rods and decant the liquid until only sufficient remains to cover the cadmium sponge. Wash the sponge two or three times with water. Transfer the cadmium sponge to a laboratory blender together with 400 ml of the hydrochloric acid solution. (5.6) and blend to obtain granules of the required size for a few seconds. Return the contents of the blender to the beaker and leave to stand for several hours, while stirring occasionally to remove bubbles. Decant most of the liquid and immediately copperize the granules as described in 9.1.

5.2 Cadmium sulfate solution, $c(CdSO_4 \cdot 8H_2O)$ = 40 g/l.

5.3 Copper (II) sulfate solution, $c(CuSO_4 \cdot 5H_2O)$ = 20 g/l.

Dissolve 20 g of copper (II) sulfate in water in a 1000 ml volumetric flask (6.4). Dilute to the 1.000 ml mark with water and mix.

5.4 Hydrochloric acid (HCl), (ρ20 = 1.19 g/ml).

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

Add 160 ml of hydrochloric acid (5.4) into a 1000 ml volumetric flask (6.4). Dilute to the 1.000 ml mark with water and mix.

5.6 Hydrochloric acid working solution, $c(HCl) \approx 0,1$ mol/l.

Add 50 ml of hydrochloric acid solution (5.5) into a 1000 ml volumetric flask (6.4). Dilute to the 1.000 ml mark with water and mix.

5.7 Zinc sulfate solution, $c(ZnSO_4 \cdot 7H_2O)$ = 535 g/l.

Dissolve 53,5 g of zinc sulfate in water in a 1000 ml volumetric flask (6.4). Dilute to the 1.000 ml mark with water and mix.

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