BS 1743 : Section 16.3 : 1987
ISO 8151 : 1987

UDC 637.143 : 664.97 : 543 : [546.48.173.175 : 620.168.32]



British Standard Methods for

# Analysis of dried milk and dried milk products

Part 16. Determination of nitrate and nitrite contents

Section 16.3 Screening method for nitrate content by cadmium reduction and spectrometry

[ISO title : Dried milk — Determination of nitrate content — Method by cadmium reduction and spectrometry (Screening method)]

Méthodes d'analyse du lait sec et des produits laitiers secs
Partie 16. Détermination de la teneur en nitrates et en nitrites
Section 16.3 Méthode rapide par réduction au cadmium et spectrométrie

Verfahren zur Prüfung von Trockenmilch und Trockenmilchprodukten
Teil 16. Bestimmung des Nitrat- und Nitritgehalts
Abschnitt 16.3 Schnellverfahren zur Bestimmung des Nitratgehalts mit Hilfe der Cadmiumreduktion und der Spektralphotometrie

**NOTE. This Part is to be read in conjunction with Part 1 'General introduction, including preparation of laboratory samples', published separately.**

## National foreword

BS 1743 : Section 16.3, which has been prepared under the direction of the Dairying Standards Committee, is identical with ISO 8151 : 1987 'Dried milk — Determination of nitrate content — Method by cadmium reduction and spectrometry (Screening method)', prepared by ISO/TC 34 Agricultural food products, of the International Organization for Standardization (ISO). It should be noted that any nitrite present will be determined as nitrate.

The method described in ISO 8151 was developed jointly with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC).

**Terminology and conventions.** The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

The symbol 'ml' has been used to denote millilitre. In British Standards it is current practice to use the symbol 'mL'.

In British Standards it is current practice to use the spelling 'sulphur', etc. instead of 'sulfur', etc.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'Section of BS 1743'.

In footnote 1) to **5.7** 'ISO' should be read as 'BSI'.

**Cross-references**

| International standard | Corresponding British Standard |
|---|---|
| ISO 707 : 1985 | BS 809 : 1985 Methods for sampling of milk and milk products (Identical) |
| ISO 835/1 : 1981 | BS 700 Graduated pipettes Part 1 : 1982 Specification for general requirements (Identical) |
| ISO 1042 : 1981 | BS 1792 : 1982 Specification for one-mark volumetric flasks (Identical) |

The Technical Committee has reviewed the provisions of ISO 648, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard to ISO 648 is BS 1583 : 1986 'Specification for one-mark pipettes'.

British Standards Institution

**Additional information.** BS 1743 : Section 16.1 describes a method of determining the nitrate and nitrite contents of dried milk using cadmium reduction and photometry.

With reference to clause **5**, water complying with grade 3 of BS 3978 'Specification for water for laboratory use' may be used.

The wording of the first sentence of **5.6** could be interpreted as an instruction to add water to concentrated sulphuric acid. The wording should be construed as meaning 'To approximately 600 ml of water in a 1000 ml one-mark volumetric flask, add carefully 17 ml of the concentrated sulphuric acid.'

Experience in using the method indicates that in order to achieve a clear filtrate (see **8.5.2**), it is necessary to allow the solution to stand for at least 15 min but no longer than 1 h. After standing, the solution should be filtered through the fluted filter paper.

It is recommended that analysts should periodically undertake duplicate determinations to verify that results complying with the repeatability requirement of **9.2** are being obtained.

NOTE. *Typographical error.* Paragraph 2 of clause **4** should refer to 'cadmium ions'.

**Compliance with a British Standard does not of itself confer immunity from legal obligations.**

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## 1 Scope and field of application

This International Standard specifies a screening method by cadmium reduction and spectrometry for the determination of the nitrate content of dried milk.

## 2 Reference

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

## 3 Definition

**nitrate content of dried milk**: The content of substances determined by the procedure specified in this International Standard and expressed as milligrams of nitrate ion ($NO_3^-$) per kilogram.

NOTE — Any nitrite present in the dried milk will be determined as nitrate.

## 4 Principle

Dissolution of the dried milk in water, precipitation of the fat and proteins, and filtration.

Reduction of the nitrate to nitrite in a portion of the filtrate by means of zinc powder and cadmium ion.

Development of a red colour in a portion of the supernatant liquid by addition of sulfanilamide and *N*-1-naphthyl ethylenediamine dihydrochloride, and spectrometric measurement at a wavelength of 538 nm.

## 5 Reagents

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

### 5.1 Solutions for precipitation of proteins and fat.

#### 5.1.1 Zinc sulfate solution.

Dissolve 267,5 g of zinc sulfate heptahydrate ($ZnSO_4 \cdot 7H_2O$) in water and dilute to 500 ml.

#### 5.1.2 Potassium hexacyanoferrate(II) solution.

Dissolve 86 g of potassium hexacyanoferrate(II) trihydrate ($K_4[Fe(CN)_6] \cdot 3H_2O$) in water and dilute to 500 ml.

### 5.2 Cadmium acetate solution.

Dissolve 0,5 g of cadmium acetate dihydrate [$(CH_3COO)_2Cd \cdot 2H_2O$] in water, add 1 ml of acetic acid ($CH_3COOH$) and dilute to 100 ml.

### 5.3 Zinc, suspension.

Just before use, introduce 10 ml of water and 2 g of zinc powder into a small beaker. Maintain the zinc in suspension by magnetic stirring.

### 5.4 Solutions for colour development.

#### 5.4.1 Solution I.

Dilute 450 ml of concentrated hydrochloric acid ($\varrho_{20}$ 1,19 g/ml) to 1 000 ml with water.

#### 5.4.2 Solution II.

Dissolve, by heating on a water-bath, 0,5 g of sulfanilamide ($NH_2C_6H_4SO_2NH_2$) in a mixture of 75 ml of water and 5 ml of concentrated hydrochloric acid ($\varrho_{20}$ 1,19 g/ml). Cool to room temperature and dilute to 100 ml with water. Filter if necessary.

#### 5.4.3 Solution III.

Dissolve 0,1 g of *N*-1-naphthyl ethylenediamine dihydrochloride ($C_{10}H_7NHCH_2CH_2NH_2 \cdot 2HCl$) in water. Dilute to 100 ml with water. Filter if necessary.

The solution may be stored for up to 1 week in a well-stoppered brown bottle in a refrigerator.

### 5.5 Potassium nitrate, standard solution corresponding to 0,03 g of $NO_3^-$ per litre.

#### 5.5.1 Stock solution.

Dry a few grams of potassium nitrate ($KNO_3$) at 110 to 120 °C to constant mass, i.e. until the difference between two suc-