

Methods for

# Analysis of dried milk and dried milk products —

Part 6: Assessment of the heat class of  
dried milk —

Section 6.1 Heat number  
method (reference method)

**IMPORTANT NOTE.** It is essential that this Part be read in conjunction with Part 1 “General introduction, including preparation of laboratory samples”, published separately.

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# Foreword

This Section of Part 6 of BS 1743, which has been prepared under the direction of the Dairying Standards Committee, describes one of a new series of methods for the analysis of dried milk and dried milk products.

The heat number reference method for assessing the heat class of dried milk is technically the same as that being prepared by a joint working group of the International Dairy Federation (IDF), the International Organization for Standardization (ISO) and the Association of Official Analytical Chemists (AOAC), in which the UK actively participated.

The heat number method does not enable Ultra Low Heat dried milk (minimal heat treatment) to be identified, and may be considered unsuitable for routine use. Accordingly, further Sections of Part 6 of BS 1743 will describe a cysteine number reference method for identifying Ultra Low Heat dried milk and also one or more routine methods of heat classification.

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## Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

## Amendments issued since publication

| Amd. No. | Date of issue | Comments |
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## 0 Introduction

**0.1** The heat treatment applied in the manufacture of spray-dried milk, predominantly in the preheating of the liquid milk, can be varied to produce a product suitable for a particular usage. It is therefore necessary, both for the manufacturer and the buyer or user, to be able to assess the degree of overall heat treatment so that a dried milk can be classified as, for example, Low Heat, Medium Heat, etc. The fact that an increasing proportion of the milk serum protein (whey protein) in milk is denatured (and becomes associated with the casein) as the degree of heat treatment increases has provided a basis for heat classification methods. These methods involve either the direct measurement of residual undenatured milk serum protein, or the indirect measurement of denatured milk serum protein, and the concentration found in the dried milk in relation to the range of possible concentrations indicates heat class. Ideally, because of the small seasonal variations in the concentration and relative proportions of the proteins in raw bulk cows' milk, a sample of the original liquid milk should be available for comparison when assessing the heat class of a dried milk but this is only possible for the manufacturer. In practice, heat class is usually assessed solely by examining the dried milk and this inevitably imposes some limitation on the accuracy of all protein-based heat classification methods.

The most widely used heat classification method is probably that published by the American Dry Milk Institute (ADMI) (1971), in which the concentration in the dried milk of undenatured milk serum protein nitrogen (UMSPN) is determined turbidimetrically so that the dried milk can be placed in one of the following heat classes: Low Heat (not less than 6.00 mg UMSPN/g), Medium Heat (1.51 mg to 5.99 mg UMSPN/g) or High Heat (not more than 1.50 mg UMSPN/g). But the procedure for determining the so-called "ADMI undenatured whey protein nitrogen value" of dried milk has several drawbacks and alternative procedures utilizing dye-binding have been developed (e.g. Sanderson, 1970; McGann, O'Connell & McFeely, 1975).

The heat classification of dried milk by the indirect measurement of denatured milk serum protein (DMSP) is possible by isolating the complex of casein and DMSP and determining its content of cystine plus cysteine (as total cysteine; derived mostly from the DMSP) by the method of de Koning, van Rooijen & Draaisma (1976) or Mrowetz & Klostermeyer (1977). However, it has been recognized for some time that another method of this general type would be to utilize the procedure of Rowland (1938a) for determining the casein number of raw liquid milk (Rowland, 1938b); casein number is the ratio of casein nitrogen content to total nitrogen content multiplied by 100 and is useful in diagnosing subclinical mastitis (Rowland & Zein-El-Dine, 1938). When Rowland's procedure is applied to heated milk or to reconstituted dried milk, the "casein number" obtained is in fact 100 times the ratio of casein nitrogen content plus DMSP nitrogen content (or more accurately, acid-insoluble (pH 4.8) protein nitrogen content) to total nitrogen content, and thus increases with the degree of heat treatment sustained by the milk or dried milk.

Consideration of all the available heat classification methods for dried milk and extensive trials of most of these, including examination by gel electrophoresis of the casein/DMSP complex and the UMSP isolated from dried milks, led to the conclusion that a method based on the determination of "casein number", renamed "heat number" to avoid confusion, would be the best overall general-purpose method and should be regarded as the reference method. During these trials, it became evident that for the accurate and precise determination of the heat number of dried milk existing international and national standard methods for determining the total nitrogen and casein nitrogen contents of milk (e.g. in BS 1741:1963) were unsuitable and accordingly, modified methods were developed. That the heat number method of heat classification incidentally determines the total nitrogen content of the dried milk may often be advantageous.

**0.2** Since the Kjeldahl method for the determination of total nitrogen and casein nitrogen in milk described in clause 7 of BS 1741:1963 (digestion mixture: 20 mL  $\text{H}_2\text{SO}_4$ , 10 g  $\text{K}_2\text{SO}_4$ , 0.5 g  $\text{HgO}$ ) is unsuitable for the larger amounts of material to be digested when determining heat number, a modified Kjeldahl method was developed. The amounts of sulphuric acid and potassium sulphate were increased to 30 mL and 15 g respectively and the 0.5 g of mercury oxide was replaced as catalyst with only 0.05 g of copper sulphate hydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), a change in accord with current views (e.g. Rexroad & Cathey, 1976; White, 1980).