

Methods for

The chemical analysis of ice cream

Co-operating organizations

The Dairying Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Public Analysts*	National Institute for Research in Dairying*
Chemical Society	National Physical Laboratory (Ministry of Technology)
Dairy and Ice Cream Equipment Association	Office of the High Commissioner for Australia*
Dairy Engineers' Association	Office of the High Commissioner for Canada
Department of Agriculture and Fisheries for Scotland	Office of the High Commissioner for New Zealand*
Embassy of the Republic of South Africa	Royal Association of British Dairy Farmers*
Hannah Dairy Research Institute*	Royal Institute of Public Health & Hygiene
Milk Distributive Council*	Royal Society for the Promotion of Health
Milk Marketing Board*	Scottish Milk Marketing Board*
Milk Marketing Board for Northern Ireland*	Scottish Milk Trade Federation
Ministry of Agriculture, Fisheries and Food*	Society for Analytical Chemistry*
Ministry of Health	Society of Chemical Industry
Ministry of Technology — Laboratory of the Government Chemist*	Society of Dairy Technology
National Association of Creamery Proprietors and Wholesale Dairymen*	University of Reading*
National Farmers' Union	West of Scotland Agricultural College

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Association of British and Dominion Condensed Milk Manufacturers	Ice Cream Alliance Limited
British Food Manufacturing Industries Research Association	Ice Cream Federation Limited
Coated Paper and Board Makers' Association	London Provision Exchange
Department of Agriculture for Canada	Ministry of Agriculture, Government of Northern Ireland
Food Manufacturers' Federation Incorporated	National Dairymen's Association (Inc.)
	Individual manufacturers

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Foreword

This standard makes reference to the following British Standards:

BS 410, *Test sieves*.

BS 809, *Methods for sampling milk and milk products*.

BS 1792, *One-mark volumetric flasks*.

BS 2648, *Performance requirements for electrically-heated laboratory drying ovens*.

BS 3591, *Industrial methylated spirits*.

BS 3978, *Water for laboratory use*.

This British Standard, prepared under the authority of the Dairying Industry Standards Committee, is a revision of BS 2472:1954.

It is not possible to lay down a simple and accurate method for the determination of the milk-solids-not-fat (MSNF), in view of the possibility of wide variations in the formulation of ice cream. This fraction is generally computed from a knowledge of the lactose, casein, calcium and phosphorus contents of the ice cream, but these constituents are not present in constant proportions in MSNF and furthermore some part may not originate from the MSNF.

A reasonably accurate assessment of the MSNF content can only be made if the composition of the ingredients of the particular ice cream product is known; this British Standard is therefore restricted to a description of analytical methods, leaving interpretation of results to the analyst.

These methods comprise those for determining total solids, fat, sucrose, reducing sugars, total protein (estimated from the nitrogen content), casein estimated under prescribed empirical conditions, ash, calcium and phosphorus.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 14, 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.

1 Scope

1.1 This British Standard provides methods for the determination of those constituents of ice cream for which reliable procedures are possible.

2 Sampling

2.1 Sampling of the ice cream shall be carried out in accordance with the provisions of BS 809¹⁾.

3 Reagents

3.1 All reagents used shall be of recognized analytical quality and, wherever water is mentioned, water complying with BS 3978²⁾ shall be used.

4 Storage of sample

4.1 Store the sample at a temperature not higher than $-15\text{ }^{\circ}\text{C}$ until the analysis is started.

5 Preparation of sample

5.1 Transfer the sample to a stoppered bottle. Place in a water bath at a temperature not exceeding $45\text{ }^{\circ}\text{C}$ until the sample is completely liquid. Remove any particulate matter which has been added to the basic ice cream. Mix thoroughly by shaking and cool to room temperature within 15 minutes. All analyses shall be started within 1 hour of melting the sample.

6 Determination of total solids

6.1 **Definition.** The total solids content of ice cream is the residue obtained on drying under the conditions specified in 6.3.

6.2 Apparatus

Dishes. Dishes shall be of metal (nickel, aluminium or stainless steel is suitable) and shall be flat bottomed, suitably flanged and with readily removable but close-fitting lids. A suitable size is a diameter of about 7.5 cm and a depth of about 2.5 cm. Each dish shall be provided with a short glass stirring rod having a flattened end and a hooked handle to rest over the side of the dish. The total length of the rod shall be less than the diameter of the dish.

Sand. White quartz sand which passes through a BS 30-mesh sieve³⁾ and is retained by a BS 85-mesh sieve³⁾. It shall be prepared by treatment with hot concentrated hydrochloric acid followed by a thorough washing with water. It shall then be heated to a dull red heat.

The suitability of the prepared sand shall be checked by carrying out a duplicate total solids determination of 1 g of sucrose in aqueous solution, when the recovered weight of sucrose shall not differ from the weight originally taken by more than $\pm 0.002\text{ g}$, and the difference between these duplicate estimations shall not exceed 0.0005 g.

Drying oven. A suitable electrically heated drying oven complying with the requirements of BS 2648⁴⁾.

6.3 **Procedure.** Place the dish, containing about 25 g of the prepared sand, in an oven at $100 \pm 1\text{ }^{\circ}\text{C}$ alongside its corresponding lid holding the short glass stirring rod. Dry to constant weight. After each period of drying replace the lid with the rod still on it, before removing the dish from the oven. Allow the dish to cool to room temperature (usually 30–45 minutes) in a desiccator charged with an efficient desiccant, such as silica gel, before weighing.

¹⁾ BS 809, "Methods for sampling milk and milk products".

²⁾ BS 3978, "Water for laboratory use".

³⁾ BS 410, "Test sieves".

⁴⁾ BS 2648, "Performance requirements for electrically-heated laboratory drying ovens".

Tilt the sand to one side of the dish; place in the clear space about 3.5 g of the well-mixed sample and weigh rapidly. Add 3 ml of water and mix by means of the rod, heating, if necessary, to dissolve lactose crystals, then mix the diluted ice cream thoroughly with the sand. Leave the stirring end of the rod in the mixture, the other end resting on the side of the dish. Place the dish on a steam bath for 20 minutes, carefully stirring during the early period, so that the mass when dry shall not form a cake, but shall be well aerated. Lay the rod flat in the dish.

Transfer the dish, with its lid alongside, to the oven at 100 ± 1 °C.

After 2½ hours cover the dish with the lid, transfer it immediately to the desiccator, allow to cool as before and weigh. Repeat the heating for periods of 1 hour, cooling and weighing until the loss of weight between successive weighings does not exceed 1 mg.

The non-volatile matter derived from the ice cream shall be regarded as the total solids and shall be expressed as a percentage by weight.

7 Determination of fat

7.1 Two methods are provided: viz. Röse-Gottlieb (using either the siphon-type apparatus, Figure 1 or Figure 2, or the Mojonnier apparatus, Figure 3) and a modified Werner-Schmid method. Investigation has shown these to be of equal validity in respect of ice cream.

7.2 Röse-Gottlieb Method

7.2.1 Apparatus

Fat-extraction apparatus. A fat-extraction tube conforming to the details given in Figure 1 or Figure 2, fitted as shown, with a two-holed bark cork and either a siphon or wash-bottle top, and provided also with a solid bark cork, or ground-glass stopper. The cork used to close the tube during shaking shall be sound and free from pores and channels which would allow leakage of solvent, and shall be previously extracted with ether. It is essential that the cork (or stopper) be wetted with water before each insertion, and washed with solvent (as directed) during each removal. Also, before each removal, to avoid spurting of the solvent, a slightly reduced pressure should be induced in the tube by cooling. Rubber stoppers shall not be used. The narrow tube with the hook-shaped lower end (see Figure 1 or Figure 2) is a sliding fit in the cork and long enough for the opening at its lower end to be placed, if necessary, at a distance of 25 mm from the bottom of the tube.

or

A Mojonnier-type fat-extraction apparatus of the dimensions and capacity given in Figure 3, provided with a ground-glass stopper or a solid bark cork. It is essential that the cork (or stopper) be wetted with water before each insertion, and washed with solvent (as directed) during each removal. Also, before each removal, to avoid spurting of the solvent, a slightly reduced pressure should be induced in the tube by cooling.

Drying oven. A suitable drying oven complying with the requirements of BS 2648⁵⁾.

Flasks. 150 ml short-necked, flat-bottomed flasks preferably of 40–50 g in weight.

7.2.2 Reagents

Aqueous ammonia, $d = 0.880$.

*Ethanol*⁶⁾, 95–96 per cent (v/v).

Diethyl ether, $d = 0.720$, peroxide-free⁷⁾.

Light petroleum, boiling range 40–60 °C.

Mixed solvent, equal parts of diethyl ether and light petroleum.

⁵⁾ BS 2648, "Performance requirements for electrically-heated laboratory drying ovens".

⁶⁾ Ethanol may be replaced by industrial methylated spirits of equivalent strength complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations 1952 (S.I. 1952, No. 2230).

⁷⁾ The diethyl ether may be maintained free from peroxide by adding wet zinc foil (approximately 80 cm² per litre, cut in strips long enough to reach at least half way up the container) that has been completely immersed in dilute acidified copper sulphate solution for 1 minute and subsequently washed with water.