BS 696-2: 1989

# Determination of fat content of milk and milk products (Gerber method) —

Part 2: Methods

UDC 637.1:543.85:542.3:620.1



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# Foreword

This Part of BS 696, having been prepared under the direction of the Dairying Standards Committee, supersedes BS 696-2:1969 which is withdrawn.

The Gerber is almost universally used for the routine determination of the fat content of milk and milk products in European countries and, to a lesser extent, in America, Australia and New Zealand. It is therefore of great importance that a method using standardized apparatus should be available so that results obtained in different laboratories and by different workers are comparable. This is essential with milk and milk products where there is considerable interchange between different factories, dairies and countries.

This revision of Part 2 takes into account the changes introduced in Part 1, the revision of which is being published simultaneously.

For milks containing about 2.5 % to 4.5 % of butterfat, the Gerber method gives results which are in close agreement with those obtained by the more accurate Röse-Gottlieb method described in BS 1741-3.

BS 696 should enable all laboratories to produce results which are comparable, and of adequate accuracy for commercial needs. It should be remembered, however, that the methods proposed are designed for routine purposes only.

While they give results in general agreement with gravimetric methods, they cannot be considered substitutes for these. Gravimetric methods should still be used for special purposes.

The apparatus for the Gerber test is specified in BS 696-1.

It is assumed in the drafting of this standard that it will be used and applied by those who are appropriately qualified and experienced. The procedures described in this standard are intended to be carried out by suitably trained and/or supervised personnel.

This Part of BS 696 calls for the use of substances and procedures that may be injurious to health if adequate precautions are not taken. In particular, attention is drawn to the need for protection especially of hands and eyes, and avoidance of mouth pipetting of sulphuric acid and amyl alcohol.

This standard refers only to its technical suitability and does not absolve the users from statutory obligations relating to health and safety.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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

#### Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, 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 General

#### 1.1 Scope

This British Standard describes methods for the determination of fat content in the milks and milk products given in Table 1 by the Gerber method.

# Table 1 — Relevant methods for various milks and milk products

Product	Relevant clause
Milk	
raw	2
pasteurized	2
homogenized	3
sterilized	3
UHT	3
Separated milk	
partly skimmed	4
skimmed	5
buttermilk	4
whey	4
Cream	6
Dried milk powder	
whole	7
Reconstituted dried milk powder	
whole	2
partly skimmed	4
skimmed	5
Cheese	8

#### **1.2 Reagents**

1.2.1 Sulphuric acid, density

 $\rho_{20}$  = 1.815  $\pm$  0.003 g/mL, i.e. 89.5 g to 91.0 g of  $\rm H_2SO_4$  per 100 g. The acid shall be colourless, or not darker in colour than pale amber, and shall not contain impurities which will affect the determination.

1.2.2 *Amyl alcohol*, complying with Appendix A.1.2.3 *Water*, complying with grade 3 of BS 3978.

#### **1.3 Apparatus**

**1.3.1** Lock stopper with key, complying with section three of BS 696-1:1989.

**1.3.2** *Pipette*, complying with the appropriate requirements of section four of BS 696-1:1989, or *automatic dispenser*, to deliver 10 mL of sulphuric acid.

**1.3.3** *Pipette*, complying with the appropriate requirements of section four of BS 696-1:1989, or *automatic dispenser* to deliver 1 mL of amyl alcohol.

1.3.4 Shaking stand, for butyrometers.

**1.3.5** *Centrifuge*, complying with section 6 of BS 696-1:1989.

**1.3.6** *Water bath(s),* capable of being controlled at  $20 \pm 2$  °C, at 20 °C to 30 °C, at 35 °C to 40 °C and at  $65 \pm 2$  °C.

**1.3.7** *Thermometers*, nominal range 0 °C to 100 °C, with a maximum error of  $\pm 1$  °C.

NOTE  $\,$  Thermometers of designation D complying with BS 1704:1985 are suitable.

### 2 Milk

NOTE The method described in this clause is applicable to unhomogenized raw and pasteurized milks and to reconstituted whole dried milk powder.

#### 2.1 Apparatus

**2.1.1** *General.* In addition to the apparatus listed in **1.3**, the items given in **2.1.2** and **2.1.3** are required.

**2.1.2** *Gerber* (8 %) *butyrometer* for testing milk, or, where milk or higher fat content (e.g. ewe's milk) is being tested, *Gerber* (10 %) *butyrometer*, complying with section 2 of BS 696-1:1989.

**2.1.3** Standard milk pipette, complying with the appropriate requirements of section 4 of BS 696-1:1989, calibrated to deliver 10.94 mL of water.

# 2.2 Preparation of sample

Adjust the temperature of the milk sample to 20 °C to 30 °C, if necessary using the water bath controlled at 20 °C to 30 °C (**1.3.6**). Mix the milk thoroughly but gently by repeatedly inverting the sample bottle to ensure a homogeneous distribution of the fat without causing undue frothing or any churning of the fat. If there is difficulty in dispersing the cream layer or the milk shows evidence of slight churning, warm the milk slowly to 35 °C to 40 °C, with gentle mixing in the water bath controlled at 35 °C to 40 °C (**1.3.6**).

Quickly adjust the temperature of the milk to about 20 °C, for example by cooling the sample under cold running water.

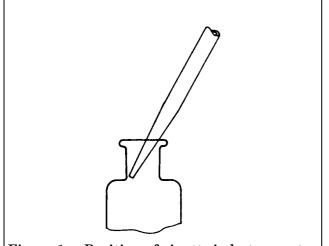
NOTE If at this stage the milk contains white particles or free fat, the determination will not yield a reliable value for fat content.

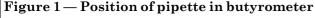
Allow the milk to stand in the water bath controlled at  $20 \pm 2$  °C (**1.3.6**) for 3 min to 4 min after the final temperature adjustment to allow air bubbles to rise. From this point it is necessary to proceed without interruption.

#### 2.3 Procedure

**2.3.1** Addition of acid to butyrometer. Measure 10 mL of the sulphuric acid (**1.2.1**) into the butyrometer (**2.1.2**) using the pipette or automatic dispenser (**1.3.2**). Do not wet the neck of the butyrometer with acid.

**2.3.2** Measurement of sample. Gently invert the bottle containing the prepared sample three or four times, and immediately pipette the milk at a temperature of about 20 °C into the butyrometer in the following manner. Using the standard milk pipette (**2.1.3**), adjust the bottom of the milk meniscus to the calibration line after wiping the outside of the delivery jet free from milk. While delivering the milk, hold the pipette with the jet in the neck of the butyrometer as shown in Figure 1. When the meniscus comes to rest, wait 3 s and then stroke the jet against the base of the neck of the butyrometer<sup>1)</sup>. Do not wet the neck of the butyrometer with milk.





**2.3.3** Addition of amyl alcohol. Measure 1 mL of amyl alcohol into the butyrometer using the amyl alcohol pipette or automatic dispenser (**1.3.3**). Do not wet the neck of the butyrometer with the alcohol. Do not, in any circumstances, add the amyl alcohol to the butyrometer before the milk.

**2.3.4** *Insertion of stopper.* Close the neck of the butyrometer firmly with the lock stopper (1.3.1) without disturbing the contents.

Using the key, stretch the rubber of the lock stopper such that the lock stopper will fit easily into the neck of the butyrometer, and then push it in so that the metal rim of the stopper is in contact with the neck of the butyrometer, as far as it is possible, and then remove the key. **2.3.5** *Mixing of contents.* Shake the butyrometer in the protected stand (**1.3.4**) until the contents are thoroughly mixed and no white particles can be seen. Invert several times during the process.

**2.3.6** Centrifuging. Immediately after mixing, place the butyrometer with the stopper end outermost, in the centrifuge (**1.3.5**). When there are insufficient butyrometers to fill the centrifuge completely, take care that they are placed symmetrically, using a blank tube filled with water if necessary. When the centrifuge has attained the speed necessary to achieve a relative centrifugal acceleration of  $350 \pm 50 g_n$  at the outer end of the butyrometer stopper (see **1.3.5** and clause **20** of BS 696-1:1989), continue centrifuging for at least a further 4 min and not more than 5 min at that speed. Do not heat the centrifuge artificially.

**2.3.7** Temperature adjustment. Remove the butyrometer from the centrifuge, adjusting the stopper, if necessary, to bring the fat column on the scale. Place it, stopper downwards, in the water bath (**1.3.6**) controlled at  $65 \pm 2$  °C for at least 3 min and not more than 10 min. Maintain the water level above the top of the fat column in the butyrometer.

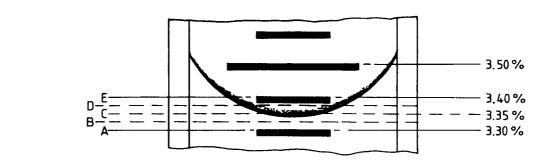
**2.3.8** *Reading of butyrometer*. Insert the key into the lock stopper and apply sufficient pressure to bring the lower end of the fat column on to a main graduation mark. Record the scale readings corresponding to the lowest point of the fat meniscus and to the interface of fat and acid; the difference between the two readings gives the percentage by mass of fat in the milk. When readings are being taken, keep the point to be read adjusted level with the eye, and read the butyrometer to the nearest half a scale division (i.e. 0.05 % or 0.1 %).

NOTE 1 See Figure 2, which shows the imaginary lines between graduation markings which define the limits for an intermediate reading.

NOTE 2 The result will be sufficiently accurate for most purposes without the application of a correction.

**2.3.9** *Check reading.* Replace the butyrometer in the water bath for another 3 min and then take a check reading of the butyrometer immediately on removal from the bath. The check reading should agree with the initial reading. If it does not, again place the butyrometer in the water bath for 3 min and take a further check reading. Repeat the procedure until two identical readings are obtained.

<sup>&</sup>lt;sup>1)</sup> This method of emptying the pipette is not identical with that used in its calibration with water.



NOTE Points A, B, C, D and E are equidistant, and where the bottom of the meniscus lies between line B and D then the reading would be for example, 3.35 % of fat. It should be observed that the thickness of a graduation line is equivalent to about 0.02 % of fat. **Figure 2 — Meniscus reading** 

**2.3.10** Correction of reading. When it is desired, for special purposes, to ensure a closer agreement between the Gerber value and the percentage of fat that would be obtained by the gravimetric Röse-Gottlieb method, use a butyrometer with known scale errors. Read the butyrometer to the nearest 0.02 % and correct for a) the scale error and b) the appropriate correction from Table 2.

# Table 2 — Corrections for butyrometer readings

Butyrometer reading (% fat)	Correction	
%	%	
1.87 to 2.25 2.26 to 2.63 2.64 to 3.02 3.03 to 3.40 3.41 to 3.79 3.80 to 4.17	+ 0.04 + 0.03 + 0.02 + 0.01 0 - 0.01 - 0.01	
4.18 to 4.56 4.57 to 4.95 4.96 to 5.33 5.34 to 5.72 5.73 to 6.10 6.11 to 6.49	$ \begin{array}{r} -0.02 \\ -0.03 \\ -0.04 \\ -0.05 \\ -0.06 \\ -0.07 \end{array} $	

#### NOTES to 2.3

NOTE 1 If a fluffy layer is observed at the base of the fat column in the butyrometer, reject the test. Examine the stopper to see if it is in good condition, repeat the test and take greater care to ensure that the curd is completely dissolved and that the correct volume of amyl alcohol has been used. NOTE 2 If the fat column is so dark as to make reading difficult, reject the test and check the density of the sulphuric acid. Also reject the test if there is charring at the interface. NOTE 3 Thoroughly clean the butyrometer immediately after use.

# 3 Homogenized, sterilized and UHT milks

#### **3.1 Apparatus**

**3.1.1** *General.* In addition to the apparatus listed in **1.3** the items given in **3.1.2** and **3.1.3** are required.

**3.1.2** *Gerber* (8%) *butyrometer*, complying with section two of BS 696-1:1989, for testing milk.

**3.1.3** Standard milk pipette, complying with the appropriate requirements of section 4 of BS 696-1:1989, calibrated to deliver 10.94 mL of water.

### **3.2 Preparation of sample**

Adjust the temperature of the sample to  $20 \pm 2$  °C if necessary using the water bath controlled at  $20 \pm 2$  °C (**1.3.6**). Mix thoroughly but gently, without causing undue frothing.

NOTE If at this stage the sample contains white particles or free fat, the determination will not yield a reliable value for fat content.

### **3.3 Procedure**

**3.3.1** *General.* Follow the same procedure as for the determination of fat in milk as described in **2.3.1** to **2.3.8**, together with the additional treatment given in **3.3.2** and **3.3.3**.

**3.3.2** Second centrifuging. Screw in the stoppers fully if necessary and immediately repeat the centrifuging, temperature adjustment and reading of the butyrometer (see **2.3.6** to **2.3.8**).

**3.3.3** Further centrifuging. If the reading obtained after the second centrifuging differs from that obtained after the first centrifuging, repeat the procedures described in **2.3.6** to **2.3.8** until two successive readings are found to be identical (see notes to **2.3**).

NOTE Homogenization and UHT treatment reduces the size of the fat globules and, consequently, renders complete separation of the fat more difficult; hence the possible need for further centrifuging.

# 4 Partly skimmed milk, buttermilk and whey

NOTE The method described in this clause is applicable partly skimmed milk, reconstituted partly skimmed milk powder, buttermilk and whey containing more than 0.25 % (m/m) fat. For products containing less than that quantity of fat, see clause **5**.

### 4.1 Apparatus

**4.1.1** *General.* In addition to apparatus listed in **1.3** the items given in **4.1.2** and **4.1.3** are required.

**4.1.2** *Gerber* (4 %) *butyrometer*, complying with section 2 of BS 696-1:1989, for testing partly skimmed milk.

**4.1.3** Standard milk pipette, complying with the appropriate requirements of section 4 of BS 696-1:1989, calibrated to deliver 10.94 mL of water.

### 4.2 Preparation of sample

Prepare the sample by the method described in 3.2.

#### 4.3 Procedure

**4.3.1** *General.* Follow the same procedure as for the determination of fat in milk, as described in **2.3.1** to **2.3.7**, with the following additional treatment given in **4.3.2** and **4.3.3**.

NOTE Care should be taken to ensure that there is adequate mixing of the contents of the butyrometer (4.1.2) between the body of the butyrometer and the stem.

**4.3.2** Second centrifuging. Repeat the centrifuging and temperature adjustment once (see **2.3.6** and **2.3.7**) before taking and checking the reading as described in steps **2.3.8** and **2.3.9**.

**4.3.3** Low butyrometer reading. When the butyrometer reading is less than 0.25 %, the method is no longer quantitative, and the product should be tested again using the method described in clause **5**.

# 5 Skimmed milk

NOTE The method described in this clause is applicable to skimmed milk, reconstituted skimmed milk powder and to products containing less than 0.25 % (m/m) fat (see clause 4).

### **5.1 Apparatus**

**5.1.1** *General.* In addition to apparatus listed in **1.3**, the items given in **5.1.2** and **5.1.3** are required.

**5.1.2** Gerber double quantity (0.5 %) butyrometer, complying with section 2 of BS 696-1:1989.

**5.1.3** Double quantity milk pipette, complying with the appropriate requirements of section 4 of BS 696-1:1989, calibrated to deliver 21.88 mL of water.

### **5.2 Preparation of sample**

Prepare the sample by the method described in 3.2.

#### 5.3 Procedure

**5.3.1** Addition of acid to the butyrometer. Measure 20 mL of the sulphuric acid (**1.2.1**) into the butyrometer (**5.1.2**) using two discharges from the pipette or automatic dispenser (**1.3.2**). Do not wet the neck of the butyrometer with acid. Ensure that air bubbles are not trapped in the bulb or stem of the butyrometer.

NOTE Trapped air bubbles can usually be removed by holding the butyrometer at an angle of 45° and gently tapping it to dislodge the air. If this is unsuccessful, the butyrometer may be placed in the water bath (1.3.6) with its stem downwards and the air bubbles should eventually rise to the surface.

**5.3.2** Measurement of sample. Gently invert the bottle containing the prepared sample three or four times, and immediately pipette the milk at a temperature of about 20 °C into the butyrometer in the following manner. Using the double quantity milk pipette (**5.1.3**) adjust the bottom of the milk meniscus to the calibration line after wiping the outside of the delivery jet free from milk. While delivering the milk, hold the pipette with the jet in the neck of the butyrometer as shown in Figure 1. When the meniscus comes to rest, wait 3 s and then stroke the jet against the base of the neck of the butyrometer<sup>2)</sup>. Do not wet the neck of the butyrometer with milk.

NOTE Care should be taken in adding the milk to the butyrometer, in order to prevent charting.

**5.3.3** Addition of amyl alcohol. Measure 2 mL of the amyl alcohol (**1.2.2**) into the butyrometer using two discharges from the pipette or automatic dispenser (**1.3.3**). Do not wet the neck of the butyrometer with the alcohol. Do not, in any circumstances, add the amyl alcohol to the butyrometer before the milk.

**5.3.4** *Insertion of stopper.* Close the neck of the butyrometer firmly with the lock stopper (1.3.1) without disturbing the contents.

Using the key, stretch the rubber of the lock stopper such that the lock stopper will fit easily into the neck of the butyrometer, and then push it in so that the metal rim of the stopper is in contact with the neck of the butyrometer, as far as it is possible, and then remove the key.

 $<sup>^{2)}</sup>$  This method of emptying the pipette is not identical with that used in its calibration with water.

**5.3.5** *Mixing of contents.* Shake the butyrometer in the protected stand (**1.3.4**) until the contents are thoroughly mixed and no white particles can be seen. Invert once or twice during the process. If air bubbles become entrapped remove them (see note to **5.3.1**).

**5.3.6** Centrifuging. Place the butyrometer immediately after mixing, with the stopper end outermost, in the centrifuge (**1.3.5**). When there are insufficient butyrometers to fill the centrifuge completely, take care that they are placed symetically, using a blank tube filled with water if necessary. When the centrifuge has attained the required speed (see **1.3.5**) continue centrifuging for at least a further 4 min and not more than 5 min at that speed. Do not heat the centrifuge artificially.

**5.3.7** Temperature adjustment. Remove the butyrometer from the centrifuge, adjusting the stopper, if necessary, to bring the fat column on the scale. Place it, stopper downwards, in the water bath (1.3.6) controlled at  $65 \pm 2$  °C for at least 3 min and not more than 10 min. Maintain the water level above the top of the fat column in the butyrometer.

**5.3.8** Second centrifuging. Repeat the centrifuging (**5.3.6**) and temperature adjustment (**5.3.7**) before taking a reading.

**5.3.9** *Reading of butyrometer*. Insert the key into the lock stopper and apply sufficient pressure to bring the lower end of the fat column on to a main graduation mark. Record the scale readings corresponding to the lowest point of the fat meniscus and to the interface of fat and acid; the difference between the two readings gives the percentage by mass of fat in the skimmed milk. When readings are being taken, hold the butyrometer with the graduated portion vertical, keep the point to be read adjusted level with the eye, and read the butyrometer to the nearest 0.01 %.

**5.3.10** *Check reading.* Replace the butyrometer in the water bath for another 3 min and then take a check reading of the butyrometer immediately on removal from the bath. The reading should agree with the initial reading. If it does not, again place the butyrometer in the water bath for 3 min and take a further check reading. Repeat the procedure until two identical readings are obtained. NOTE See notes to **2.3**.

#### 6 Cream

NOTE The method described in this clause is applicable to all types of cream.

#### 6.1 Apparatus

**6.1.1** *General.* In addition to the apparatus listed in **1.3**, the items given in **6.1.2** to **6.1.5** are required.

**6.1.2** Gerber (70 %) butyrometer, complying with section 2 of BS 696-1:1989, for testing cream.

**6.1.3** *Balance*, suitable for weighing to 0.005 g, provided with a suitable stand to support a butyrometer.

**6.1.4** *Stoppered funnel,* complying with section 5 of BS 696-1:1989 for weighing the cream.

**6.1.5** *Glass wash bottle*, containing hot water complying with BS 3978, grade 3, at a minimum temperature of 70 °C.

#### 6.2 Preparation of sample

Stir the sample of cream thoroughly but not so vigorously as to cause undue froth or churning. If the cream is very thick, warm to between 30 °C and 40 °C in the water bath (1.3.6) to facilitate mixing. If complete mixing cannot be achieved do not test the sample.

#### **6.3 Procedure**

**6.3.1** Addition of acid to butyrometer. Measure 10 mL of the sulphuric acid (**1.2.1**) into the butyrometer (**6.1.2**) by means of the standard pipette or automatic dispenser (**1.3.2**). Do not wet the neck of the butyrometer with acid.

**6.3.2** Weighing of sample. Counterbalance the weighing funnel, with its stopper inserted. Weigh  $5 \pm 0.01$  g of cream into the funnel (**6.1.4**). Wash all the cream from the funnel and stopper into the butyrometer with 6 mL of the hot water.

Alternatively, mix, and immediately weigh  $5 \pm 0.01$  g of sample into the butyrometer without soiling the neck, using the balance and stand for supporting the butyrometer (6.1.3). Then add about 6 mL of hot water (6.1.5) to the butyrometer.

**6.3.3** Addition of amyl alcohol and water. Measure 1 mL of the amyl alcohol (**1.2.2**) into the butyrometer by means of the standard pipette or automatic dispenser (**1.3.3**). Adjust the level of the contents to about 5 mm below the shoulder by further additions of hot water. Do not, in any circumstances, add the amyl alcohol to the butyrometer before the cream.

**6.3.4** *Insertion of stopper.* Close the neck of the butyrometer firmly with the lock stopper (1.3.1) without disturbing the contents.

Using the key, stretch the rubber of the lock stopper such that the lock stopper will fit easily into the neck of the butyrometer, and then push it in so that the metal rim of the stopper is in contact with the neck of the butyrometer, as far as it is possible, and then remove the key. **6.3.5** *Mixing of contents.* Shake the butyrometer in the protected stand (1.3.4) until the contents are thoroughly mixed and no white particles can be seen. Invert once or twice during the process.

**6.3.6** Temperature adjustment. Place the butyrometer, stopper downwards, in the water bath (1.3.6) controlled at  $65 \pm 2$  °C for not less than 3 min or more than 10 min.

**6.3.7** *Centrifuging.* Immediately centrifuge the butyrometer, with the stopper end outermost. When there are insufficient butyrometers to fill the centrifuge completely, take care that they are placed symmetrically, using a blank tube filled with water if necessary. When the centrifuge has attained the required speed, continue centrifuging for at least a further 5 min and not more than 6 min at that speed. Do not heat the centrifuge artificially.

**6.3.8** Second temperature measurement. Remove the butyrometer from the centrifuge, adjusting the stopper, if necessary, to bring the fat column on the scale. Place it, stopper downwards, in the water bath for at least 3 min and not more than 10 min. Maintain the water level above the top of the fat column.

**6.3.9** *Examination of fat column.* Remove the butyrometer from the water bath and examine the contents. If there is not a sharp dividing line between the fat and the acid or if the acid layer is not clear, adjust the stoppers if necessary and repeat **6.3.5** to **6.3.8**.

**6.3.10** Reading of butyrometer. When a sharp dividing line has been obtained between the fat and the acid, and the acid layer is clear, read the butyrometer immediately after removal from the water bath. Before taking a reading, adjust the position of the fat column to bring the lower end of the column on to a main graduation mark, by inserting the key and applying sufficient pressure to raise the fat column to the required position. Record the scale readings corresponding to the lowest point of the fat meniscus and to the interface of fat and acid; the difference between the two readings gives the percentage by mass of fat in the cream. When readings are being taken, hold the butyrometer with the graduation portion vertical, keep the point to be read adjusted level with the eye, and read the butyrometer to the nearest 0.5 %.

NOTE 1  $\,$  See Figure 2 which shows the imaginary lines between graduation markings which define the limits for an intermediate reading.

NOTE 2 The result will be sufficiently accurate for most purposes without the application of a correction.

Repeat **6.3.5** to **6.3.9** until two successive readings are found to be identical.

If it proves impossible to obtain a sharp dividing line between the fat and acid or to avoid a fluffy layer at the base of the fat column in the butyrometer, reject the test. Examine the stopper to see if it is in good condition, repeat the test and take greater care to ensure that the curd is completely dissolved and that the correct volume of amyl alcohol has been used (see Notes 2 and 3 to **2.3**).

## 7 Dried milk

 $\operatorname{NOTE}$   $\$  The method described in this clause is applicable to whole dried milk powder.

## 7.1 Apparatus

**7.1.1** *General.* In addition to the apparatus listed in **1.3**, the items given in **7.1.2** to **7.1.9** are required.

**7.1.2** Gerber (8 %) butyrometer, complying with section 2 of BS 696-1:1989, for testing milk.

7.1.3 Balance, suitable for weighing to 0.001 g.

**7.1.4** *Small scoop of suitable material,* e.g. aluminium, nickel or plastics for weighing the powder.

NOTE It is convenient if this scoop has a counterpoise.

**7.1.5** *Stemless funnel,* to facilitate the transference of the powder to the butyrometer.

**7.1.6** *Glass rod* of such diameter (6 mm is convenient) that the rod will pass through the hole at the base of the funnel.

7.1.7 Small camel hair brush.

**7.1.8** *Wash bottle,* containing cold water complying with BS 3978.

**7.1.9** Glass wash bottle, containing hot water complying with BS 3978 grade 3, at a minimum temperature of 70 °C.

#### 7.2 Procedure

**7.2.1** Weighing of sample. Mix the sample thoroughly, and using the balance (**7.1.3**) weigh  $1.69 \pm 0.01$  g of milk powder into the scoop (**7.1.4**).

**7.2.2** Addition of acid to butyrometer. Measure 10 mL of the sulphuric acid (**1.2.1**) into the butyrometer (**7.1.2**) using the pipette or automatic dispenser (**1.3.2**). Do not wet the neck of the butyrometer with acid.

**7.2.3** *First addition of water.* Add gently from the wash bottle (**7.1.8**) sufficient cold water to form a layer about 6 mm deep on top of the acid, allowing the water to flow down the side of the bulb.

**7.2.4** Addition of sample. Insert the narrow end of the stemless funnel (**7.1.5**) into the neck of the butyrometer. Transfer the contents of the scoop (**7.1.4**) to the funnel, removing the last particles with the camel hair brush (**7.1.7**). Tap the funnel gently until most of the powder is in the butyrometer. Transfer the powder remaining in the funnel to the butyrometer with the aid of the glass rod (**7.1.6**) and the camel hair brush. Remove the funnel.

**7.2.5** Addition of amyl alcohol. Measure 1 mL of the amyl alcohol (1.2.2) into the butyrometer using the pipette or automatic dispenser (1.3.3). Do not, in any circumstances, add the amyl alcohol to the butyrometer before the dried milk.

**7.2.6** Second addition of water. Add hot water from the wash bottle (**7.1.9**) until the butyrometer is filled to about 5 mm below the shoulder, allowing all air entrained in the powder to escape.

**7.2.7** *Insertion of stopper.* Close the neck of the butyrometer firmly with the lock stopper (1.3.1) without disturbing the contents.

Using the key, stretch the rubber of the lock stopper such that the lock stopper will fit easily into the neck of the butyrometer, and then push it in so that the metal rim of the stopper is in contact with the neck of the butyrometer, as far as it is possible, and then remove the key.

**7.2.8** *Mixing of contents.* Shake the butyrometer in the protected stand (**1.3.4**) in such a manner as to mix thoroughly the contents of the bulb of the tube, before inverting. After inversion continue the shaking and inverting until all the solid particles disappear.

**7.2.9** First temperature adjustment. Place the butyrometer, stopper downwards, in the water bath (1.3.6), controlled at  $65 \pm 2$  °C, for not less than 3 min nor more than 10 min.

**7.2.10** First centrifuging. Immediately place the butyrometer, with the stopper end outermost, in the centrifuge (**1.3.5**). When there are insufficient butyrometers to fill the centrifuge completely, take care that they are placed symmetrically, using a blank tube filled with water if necessary. When the centrifuge has attained the required speed (see **1.3.6**), continue centrifuging for at least a further 5 min and not more than 6 min at that speed. Do not heat the centrifuge artificially.

**7.2.11** Second temperature adjustment. Remove the butyrometer from the centrifuge, adjusting the stopper, if necessary, to bring the fat column on the scale. Place it, stopper downwards, in the water bath for at least 3 min and not more than 10 min. Maintain the water level above the top of the fat column in the butyrometer.

**7.2.12** Reading of butyrometer. Insert the key into the lock stopper and apply sufficient pressure to bring the lower end of the fat column on to a main graduation mark. Record the scale readings corresponding to the lowest point of the fat meniscus and to the interface of fat and acid. Record the difference between the two readings. When readings are being taken, hold the butyrometer with the graduated portion vertical, keep the point to be read adjusted level with the eye, and read the butyrometer to the nearest 0.05 %.

NOTE 1  $\,$  See Figure 2 which shows the imaginary lines between graduation markings which define the limits for an intermediate reading.

NOTE 2 The result will be sufficiently accurate for most purposes without the application of a correction.

Repeat **7.2.9** to **7.2.12** until two successive readings are found to be identical.

**7.2.13** *Calculation of percentage of fat.* Multiply the scale reading by the factor 20/3 to obtain the percentage of fat in the dried milk.

NOTE 1 The above method is applicable to cream powders and to milk powders and buttermilk powders containing not less than 10 % of fat.

NOTE 2 See notes to 2.3.

#### 8 Cheese

 $\operatorname{NOTE}$   $\$  The method described in this clause is applicable to all types of cheese.

#### 8.1 Apparatus

**8.1.1** *General.* In addition to the apparatus listed in **1.3**, the items given in **8.1.2** to **8.1.7** are required.

**8.1.2** *Gerber* (40 %) *butyrometer*, complying with section 2 of BS 696-1:1989, for testing cheese.

8.1.3 Balance, suitable for weighing to 0.001 g.

**8.1.4** *Stoppered funnel*, complying with section 5 of BS 696-1:1989, for weighing the cheese.

8.1.5 Grater or pestle and mortar.

**8.1.6** Wash bottle, containing warm water complying with BS 3978 at 30 °C to 40 °C.

8.1.7 Small camel-hair brush.

#### 8.2 Preparation of sample

Grate samples of hard cheese using the grater (8.1.5). Grind samples of soft cheese using the pestle and mortar (8.1.5). Mix thoroughly.

#### 8.3 Procedure

**8.3.1** Weighing of sample. Counterbalance the weighing funnel, with its stopper inserted (8.1.4) on the balance (8.1.3). Weigh  $3 \pm 0.001$  g of the sample into the funnel.

**8.3.2** Addition of acid to butyrometer. Measure 10 mL of the sulphuric acid into the butyrometer (**8.1.2**) using the pipette or automatic dispenser (**1.3.2**), (**1.2.1**). Do not wet the neck of the butyrometer with acid.

**8.3.3** *First addition of water.* Add gently from the wash bottle sufficient warm water (**8.1.6**) to form a layer about 6 mm deep on top of the acid, allowing the water to flow down the side of the bulb.

**8.3.4** Addition of sample. Insert the neck of the funnel containing the 3 g of cheese into the neck of the butyrometer. Withdraw the stopper from the neck of the funnel and transfer all the cheese to the butyrometer with the aid of the camel-hair brush (**8.1.7**).

**8.3.5** Addition of amyl alcohol. Measure 1 mL of the amyl alcohol (**1.2.2**) into the butyrometer by means of the standard pipette or automatic

dispenser (1.3.3). Do not, in any circumstances, add the amyl alcohol to the butyrometer before the cheese.

**8.3.6** Second addition of water. Add warm water from the wash bottle until the butyrometer is filled to about 5 mm below the shoulder.

**8.3.7** *Insertion of stopper.* Close the neck of the butyrometer firmly with the lock stopper (1.3.1) without disturbing the contents.

Using the key, stretch the rubber of the lock stopper such that the lock stopper will fit easily into the neck of the butyrometer, and then push it in so that the metal rim of the stopper is in contact with the neck of the butyrometer, as far as it is possible, and then remove the key.

**8.3.8** *Mixing of contents.* Shake the butyrometer in the protected stand (1.3.4) in such a manner as to mix thoroughly the contents of the bulb of the tube before inverting. After inversion continue the shaking and inverting until all the solid particles disappear.

**8.3.9** Temperature adjustment. Place the butyrometer, stopper downwards, in the water bath (1.3.6) controlled at  $65 \pm 2$  °C for not less than 3 min or more than 10 min.

**8.3.10** Centrifuging. Place the butyrometer immediately, with the stopper end outermost, in the centrifuge (1.3.5). When there are insufficient butyrometers to fill the centrifuge completely, take care that they are placed symmetrically using a blank tube, filled with water if necessary. When the centrifuge has attained the required speed continue centrifuging (see 1.3.5) for at least a further 5 min and not more than 6 min.

**8.3.11** Second temperature adjustment. Remove the butyrometer from the centrifuge, adjusting the stopper, if necessary, to bring the fat column on the scale. Place it, stopper downwards, in the water bath for at least 3 min and not more than 10 min. Maintain the water level above the top of the fat column in the butyrometer.

**8.3.12** *Examination of fat column.* Remove the butyrometer from the water bath and examine the contents. If there is not a sharp dividing line between the fat and the acid, or if the acid layer is not clear, adjust the stopper if necessary and repeat **8.3.9** to **8.3.12**.

**8.3.13** *Reading of butyrometer.* When a sharp dividing line has been obtained between the fat and the acid, and the acid layer is clear, read the butyrometer immediately after removal from the water bath. Before taking a reading, adjust the position of the fat column to bring the lower end of the column on to a main graduation mark. When double-ended stoppers are used, do this by slightly withdrawing the stopper and not by forcing it further into the neck. When a lock stopper is used, do this by inserting the key and applying sufficient pressure to raise the fat column to the required position. Record the scale readings corresponding to the lowest point of the fat meniscus and to the interface of fat and acid; the difference between the two readings gives the percentage by mass of fat in the cheese. When readings are being taken, hold the butyrometer with the graduated portion vertical, keep the point to be read level with the eye, and read the percentage of fat to the nearest 0.3 % i.e. one-third of the smallest scale division. Repeat 8.3.9 to 8.3.13 until two successive readings are found to be identical.

NOTE For cheese containing more than 40 % of fat 1.5 g of cheese should be taken for the test and the butyrometer reading multiplied by 2. See also Note 1 to **6.3.10** and Notes 2 and 3 to **2.3**.

#### Appendix A Amyl alcohol

#### A.1 Description

The amyl alcohol shall consist principally of 3-methylbutan-1-ol and 2-methylbutan-1-ol. The colour of the material shall not exceed 35 Hazen units, and it shall be free from secondary amyl alcohols, tertiary amyl alcohol and furfural. It shall have a density at 20 °C of  $0.811 \pm 0.002$  g/mL and on distillation at 1.013 bar<sup>3)</sup> shall all distil between 128 °C and 133 °C, leaving no solid residue.

NOTE Amyl alcohol supplied for this purpose or the documents relating thereto, should be labelled as follows. "Amyl alcohol for the Gerber determination of fat (BS 696)".

#### A.2 Suitability for Gerber test

It is possible that an amyl alcohol may comply with **A.1** and yet be unsuitable for the Gerber method. Therefore the only conclusive criterion of the suitability of an amyl alcohol is that when it is used in the method described in this standard, using butyrometers of known scale error, the values obtained with milks of average fat content will not differ from those obtained by the reference method by more than 0.03 % fat. The method of carrying out this test is as follows.

Obtain at least three samples of fresh milk in good physical condition with a fat content of

between 3.4 % and 3.8 %. Determine the fat content of each sample in quadruplicate by the Gerber method described in this standard, taking readings to the nearest 0.02 % fat and correcting for any scale errors of the butyrometers. Calculate the mean of all the values. Determine the fat content of each sample in triplicate by the reference method described in BS 1741-3 and calculate the mean value as before. Compare the two mean values.

#### A.3 Furfural and other organic impurities

The colour developed when 5 mL of the amyl alcohol is added to 5 mL of concentrated sulphuric acid (97 %  $H_2SO_4$ ) of density 1.837 g/mL at 20 °C shall not exceed the following values, using either the chromaticity co-ordinates of the Commission Internationale de l'Eclairage (CIE) or the Lovibond scale<sup>3)4)</sup>. The colour shall be determined using a 12.7 mm cell.

#### Table 3 — Colour development values

Chromaticity co-ordinates using illuminant B			Lovibond units	
x	У	z	yellow	red
0.4391	0.4571	0.1040	10	1

#### A.4 Sampling and size of sample

For the purpose of examination, a representative sample of the reagent measuring not less than 500 mL shall be taken from the bulk. The sample shall be placed in a clean, dry and airtight glass stoppered bottle of such a size that it is nearly filled by the sample.

In cases where it is necessary to seal the container, care shall be taken to avoid the risk of contaminating the contents in any way.

<sup>&</sup>lt;sup>3)</sup> 1 bar =  $10^5$  N/m<sup>2</sup> = 100 kPa.

<sup>&</sup>lt;sup>4)</sup> For information on suitable equipment contact the Enquiry Section, BSI, Linford Wood, Milton Keynes MK14 6LE.

# **Publications referred to**

BS 696, Determination of fat content of milk and milk products (Gerber method).
BS 696-1, Specification for apparatus.
BS 1704, Specification for solid-stem general purpose thermometers.
BS 1741, Methods for chemical analysis of liquid milk and cream.
BS 1741-3, Determination of fat content of liquid milk.
BS 3978, Specification for water for laboratory use.

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