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DETERMINATION OF FAT BY THE GERBER METHOD

PART I MILK

(First Revision)

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Indian Standard

DETERMINATION OF FAT BY THE GERBER METHOD

PART I MILK

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part I) (First Revision) was adopted by the Indian Standards Institution on 30 May 1977, after the draft finalized by the Dairy Products Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 The Gerber method for determination of fat in milk and milk products depends on the liberation of the fat by the action of sulphuric acid on milk or milk products in specially shaped, calibrated glass containers, called butyrometers which are then centrifuged to aid the separation of fat, the volume of which is finally read off against the pereentage scale etched on the butyrometer. This method is widely used in India for the rapid determination of fat in milk and other milk products when a large number of samples is to be analysed at a time.

0.2.1 With the increasing application of the Gerber method for determination of fat in milk and milk products for commercial need in India, it has become necessary to prescribe a uniform method, using standard apparatus designed to meet the Indian conditions, so that the results obtained in different laboratories and by different workers are comparable within a reasonable degree of accuracy.

0.3 This standard was first issued in 1958, as IS : 1224-1958*. The standard prescribed the use of 11·04 ml pipette for determination of fat in milk, with which corrections of the readings of fat column in the butyrometers had to be applied to obtain the value of fat closer to the gravimetric method. In this revision, these correction tables have been omitted as the use of 10·75 ml milk pipettes has been prescribed which have been found to give results closest to the gravimetric method without using correction factor. It has now been decided to issue the revised version of the standard into two parts. This part (Part I) deals with the determination of fat in milk

*Determination of fat in milk, evaporated (unsweetened) milk, separated milk, skim milk, buttermilk and cream by the Gerber method.

and incorporates the latest thinking on the subject of the International Organization for Standardization (ISO). Part II deals with the determination of fat in milk products.

0.4 It should be emphasized that the methods prescribed in this standard are designed for routine purposes only, and while these give results comparable with gravimetric method, these cannot be substituted for gravimetric methods, which will have to be used for reference purposes.

0.5 In the formulation of this standard, considerable assistance has been derived from the following:

ISO/R 2446 Milk — Determination of fat content (routine method). International Organization for Standardization.

BS 696 : Part II : 1969 Gerber method for the determination of fat in milk and milk products : Part II Methods. British Standards Institution.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes the procedure for the determination of fat in whole milk, evaporated (unsweetened) milk, standardized milk, toned milk, double toned milk, separated milk, skim milk, buttermilk and homogenized milk by the Gerber method.

2. APPARATUS

2.1 The apparatus given in **2.1.1** to **2.1.8** conforming to the provisions of IS : 1223 (Part I)-1970†, IS : 1223 (Part II)-1973‡, and IS : 1223 (Part III)-1977§ shall be used.

2.1.1 *Butyrometers, 6 Percent, 8 Percent and 10 Percent Scales* — for estimating fat in whole milk and evaporated (unsweetened) milk, double toned milk, toned milk and homogenized milk.

*Rules for rounding off numerical values (*revised*).

†Specification for apparatus for determination of milk fat by Gerber method : Part I Butyrometers and stoppers (*first revision*).

‡Specification for apparatus for determination of milk fat by Gerber method : Part II Pipettes and automatic measures (*first revision*).

§Specification for apparatus for determination of milk fat by Gerber method : Part III Centrifuges and water-baths (*first revision*).

2.1.2 *Butyrometers, 1 Percent and 4 Percent Scale* — for estimating fat in separated milk, skim milk and buttermilk.

2.1.3 *10-ml Pipette or Automatic Measure for Sulphuric Acid*

2.1.4 *10·75-ml Pipette for Milk*

2.1.5 *1-ml Pipette or Automatic Measure for Amyl Alcohol*

2.1.6 *Stoppers for Butyrometers*

2.1.7 *Centrifuge*

2.1.8 *Water-Bath*

3. REAGENTS

3.1 Sulphuric Acid — Sulphuric acid shall have a density of 1·807 to 1·812 g/ml at 27°C corresponding with a concentration of sulphuric acid from 90 to 91 percent by mass.

3.1.1 The sulphuric acid shall be colourless or not darker than pale amber in colour.

3.1.2 When diluted with distilled water to a density of 1·4 g/ml not more than a very slight turbidity shall occur.

3.2 Amyl Alcohol — The amyl alcohol shall conform to Grade 1 of IS : 360-1964*.

4. PROCEDURE

4.1 Preparation of Butyrometers — Mark the number of the sample to be tested legibly on the bulb of the butyrometer.

4.2 Transfer 10 ml of sulphuric acid (*see* **3.1**) into the butyrometer by means of the 10-ml pipette or the automatic measure for sulphuric acid (*see* **2.1.3**) taking care not to wet the neck of the butyrometer with the sulphuric acid.

4.3 Mixing of Preparation of Sample — If the sample is fresh, warm it to approximately 27°C and mix thoroughly but do not shake it so vigorously as to cause to under frothing or churning of the fat. Pour the sample into another clean dry vessel and back to the original. Repeat this process of pouring back and forth until a homogeneous mixture is obtained. Allow the sample to stand for three or four minutes after mixing to allow air bubbles to rise; invert the sample bottle three or four times immediately prior to taking milk for the test.

NOTE 1 — If the sample has aged and there is difficulty in dispersing the cream layer by the above method, warming to 30°C may be necessary for adequate mixing.

*Specification for amyl alcohol (*revised*).

NOTE 2 — If the sample shows evidence of slight churning, shown by the presence of white flakes, it should be slowly warmed to 34°C to 40°C before mixing as described in 4.3. If after this treatment a sample appears not to be homogeneous, it shall be rejected.

4.4 Addition of the Sample — Measure 10·75 ml of sample into the required butyrometer by means of the 10·75-ml pipette (*see* **2.1.4**), the temperature of sample should be broght to approximately 27°C when it is measured.

4.4.1 The procedure to be followed in using the pipette for measuring sample into the butyrometer shall be as follows:

Dip the tip of the pipette in the well-mixed sample contained in the bottle and suck in the sample, until the sample rises to a short distance above the graduation mark. Close the upper end of the pipette and withdraw it from the sample. Wipe the outside of the delivery tube of the pipette, dry with a clean piece of filter paper, hold the pipette vertically and run out the milk until the top of the milk meniscus, not the bottom of meniscus, which is difficult to see is on the graduation mark. When this is achieved, insert the jet of the pipette into the neck of the butyrometer, holding the butyrometer vertically. Touch the tip of the jet to the base of the neck of the butyrometer and slant the pipette so that the delivery tube of the pipette rests on the top neck (*see* Fig. 1). In this position, the vertical axis of the pipette makes an angle of 45° with the vertical axis of the butyrometer. Holding the pipette in this position, release the finger from the other end of the pipette, directing the flow of milk against the wall of the body of the butyrometer. When emptying the pipette, take care to have a gentle flow of the milk on to the surface of the sulphuric acid, preventing, as far as possible, the mixing of the two liquids. When the outflow has ceased, wait for 3 seconds, raise the pipette and then gently touch the jet of the pipette once against the base of the neck of the butyrometer and then remove the pipette. At any stage of transfering the milk, take care not to wet the neck of the butyrometer with milk.

4.4.2 If the same pipette is used, take care to rinse the pipette with a portion of the next sample to be analyzed. Take care to measure the sample always in the correct sequence.

4.5 Addition of Amyl Alcohol — Measure one millilitre of amyl alcohol (*see* **3.2**) into the butyrometer by means of the 1-ml pipette or the automatic measure for amyl alcohol (*see* **2.1.5**). Do not wet the neck of the butyrometer with alcohol.

4.6 Insertion of Stopper — Close the neck of the butyrometer firmly with the stopper (*see* **2.1.6**) without disturbing the contents. When a double-ended stopper is used, screw it in until the widest part is at least level with the top of the neck. When a lock stopper is used insert it until the rim is in contact with the neck of the butyrometer.

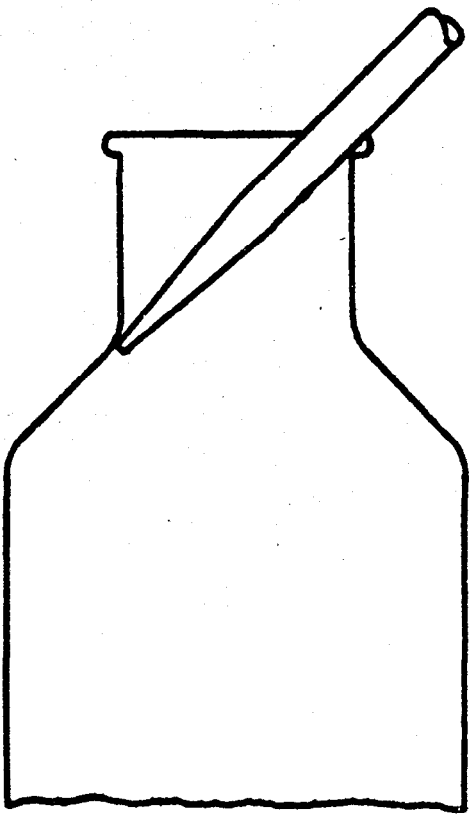

FIG. 1 POSITION OF PIPETTE DELIVERING SAMPLE

4.7 Mixing of Contents — Shake the butyrometer carefully, without inverting it, until the contents are thoroughly mixed, the curd is dissolved and no white particles are seen in the liquid. Then invert the butyrometer a few times to mix the contents thoroughly.

NOTE — When a large number of samples is to be mixed, shake the butyrometers in a protected stand until the contents are thoroughly mixed and no white particles are seen. Invert once or twice during the process.

4.8 Temperature Adjustment — Transfer the butyrometer quickly, with the bulb uppermost, into a water-bath (*see* **2.1.8**) having a temperature of $65 \pm 2°C$ and leave it there for not less than 5 minutes. Take care to have the water level in the bath above the top of the fat colum in the butyrometer. Meanwhile, adjust the stopper so that the fat column shall be on the scale after centrifuging.

4.9 Centrifuging — Take the butyrometer out of the water, dry it with a cloth and transfer it to the centrifuge, placing two butyrometers diametrically opposite so as to balance the rotating disc. Centrifuge at the maximum speed for 4 minutes. Bring the centrifuge to stop gradually. Transfer the butyrometers, stoppers downwards, into a water-bath having a temperature of 65 $\pm$ 2°C as in **4.8**, and allow the butyrometers to stand in the water-bath for not less than 3 minutes and not more than 10 minutes.

4.10 Reading of Butyrometer — 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. Note the scale readings corresponding to the lowest point of the fat meniscus and the surface of separation of the fat and acid; the difference between the two readings gives the percentage by mass of fat in the milk. When readings are being taken, hold the butyrometer with the graduated portion vertical, keep the point read in level with the eye, and then read the butyrometer to the nearest half of the smallest scale division.

4.11 Procedure for Skim Milk, Separated Milk, Buttermilk, and Evaporated (Unsweetened Milk) — When these are being tested, repeat the temperature adjustment (*see* **4.8**) and centrifuging (*see* **4.9**) before taking the reading as prescribed in **4.10**.

NOTE — If there is insufficient fat in the butyrometer to enable the fat content to be read, record the apparent fat content, for example; nil, trace, or fraction of meniscus.

4.12 Procedure for Homogenized Milk — In case of homogenized milk, obtain the second value of fat content (*see* **4.11**). If the second value does not exceed the first value by more than half a smallest scale division of the butyrometer, the second value shall be recorded as the fat content of the milk.

4.12.1 If the second value exceeds the first value by more than half a smallest scale division, repeat the procedure (*see* **4.11**) and obtain a third value for the fat content. If the third value does not exceed the second value by more than half a smallest scale division, the third value shall be recorded as the fat content of the milk.

4.12.2 If the third value exceeds the second value by more than half a smallest scale division, repeat the procedure (*see* **4.11**) and obtain fourth value for the fat content. The fourth value shall be recorded as the fat content of the milk, but if this value exceeds the third value by more than half a smallest scale division, it should be regarded as of doubtful accuracy.

NOTE 1 — If even after the several centrifugings, the fat is turbid or dark in colour or if there is white or black material at the bottom of the fat column the value for fat content would not be accurate.

NOTE 2 — The results obtained may be slightly high.

4.13 Procedure for Milk Containing Preservatives

4.13.1 If the milk containing preservatives has gone through the process of homogenization, follow the procedure described in **4.12**. In case the milk containing preservatives is skim milk, follow the procedure described in **4.11**.

4.13.2 In milk containing preservatives, there may be some difficulty in achieving complete solution of the protein. In such cases, place the butyrometer, stopper downwards in the water-bath maintained at 65 $\pm$ 2°C with occasionally shaking and the inversion of the butyrometer until no white particles are seen. Then proceed as described in **4.8**, **4.9** and **4.10**.

NOTE — If the time required in the water-bath to dissolve the protein exceeds 10 minutes, the method would not give an accurate result and would not be applicable to the sample.

4.14 Precautions — 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.

4.14.1 If the fat column is so dark as to make reading difficult, reject the test and check the strength of the sulphuric acid.

4.14.2 If a large number of sample has to be tested, it is preferable to use automatic measures for measuring the sulphuric acid and amyl alcohol, especially the latter, otherwise there is a possibility of injurious effects to the health arising from the inhalation of amyl alcohol vapours by the use of 1-ml pipette.

4.15 Repeatability — The difference between the results of two determinations carried out simultaneously, or in rapid succession, by the same analyst shall not exceed the value corresponding to one smallest scale division of the butyrometers. When butyrometers with scale errors less than 0·01 percent are used, the difference between the results of two determinations obtained shall not exceed the value corresponding to half a smallest scale division of the butyrometer.

4.16 Test Report — The test report shall show the method used and the result obtained, including the following:

a) The capacity of the milk pipette,

b) The scale range of the butyrometer, and

c) Any observation that indicates that the result is of doubtful accuracy.

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