

**Bangladesh Standard  
Specification For  
Soft candy**  
(Draft for Second Revision)

**ICS 67.060**



BANGLADESH STANDARDS AND TESTING INSTITUTION  
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**BSTI**

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## Foreword

This Bangladesh Standard was adopted by the Bangladesh Standards and Testing Institution on ....., after the draft finalized by the Bakery and Confectionary Products Sectional Committee had been approved by the Agricultural and Food Products Divisional Committee.

A broad range of varieties of soft candy are being marketed in the country. The quality of soft candy is governed by its key compositional factors, namely the amount of moisturizer and the type and ratio of component sugars. In order to ensure that the product conforms to safety and quality requirement it was necessary to prepare this standard so as to safeguard the consumer which mainly comprise of children.

This standard was first published in 1982 and then revised in 2001. Earlier this standard was titled as 'BDS 1000 Toffees'. While reviewing this standard, the committee decided to accommodate sugar confectionary soft candy including toffees, gelatin based candy and pectin based candy within the scope of one standard as the basic requirements are common for these products. Thereby the committee agreed to update this standard into a single comprehensive user friendly-standard and changed the title as 'BDS 1000 Soft candy' in keeping with international nomenclature.

Major modifications in this version are as follows:

- a) requirements for various categories of soft candy have been incorporated ;
- b) the ingredient lists have been modified;
- c) clauses for 'hygienic requirements' and 'legal requirements' have been included;
- d) requirements for 'reducing sugar' and 'lactose' have been omitted;
- e) requirements for 'milk fat' has been included;
- f) microbiological limit for *Salmonella* has been introduced; and
- g) requirements for labeling has been modified according to the current practice.

The Sectional Committee responsible for the preparation of this standard has taken into consideration the views of the members of this committee, manufacturers, consumers and technologists. This standard is subject to periodical reviews and amendments, if necessary. Any suggestions for improvement will be recorded and placed before the Committee in due course.

In the formulation of this standard, considerable assistance has been derived from the following publications, which are acknowledged with thanks.

- SLS 1575:2017 Soft Candy  
Sri Lanka Standards Institution
- NTC 3207:2008 Soft Candies  
Colombian Institute of Technical Standards and Certification

For the purpose of deciding, whether a particular requirement of this standard is complied with the final value observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with BDS 103. The number of significant places retained in the rounded off value should be the same as that of the specified value in the standard.

This standard BDS 1000:YYYY Soft Candy (2<sup>nd</sup> Rev.) cancels and replaces BDS 1000:2001 Toffees (1<sup>st</sup> Rev.) that has been technically revised.

# Bangladesh Standard Specification For Soft candy (Draft for Second Revision)

## 1. Scope

1.1 This standard prescribes the requirements and the methods of test for soft candy.

## 2. Normative References

2.1 The Bangladesh Standards listed in Annex-J are necessary adjuncts to this standard. For references, the latest edition of the referenced document including any amendments applies.

## 3. Terminology and Types

For the purpose of this standard the following definitions apply:

3.1 Soft candy shall be of following types.

**3.1.1 Soft Candy** – They are soft, easily chewable products, obtained from cooking of a solution of carbohydrates such as sugar, invert sugar, glucose syrup, polyols, polydextrose, isomaltitol, edible fats and oils, emulsifiers and other permitted ingredients and may or may not contain cocoa and milk.

**3.1.2 Soft Dairy or Milk Candy** – They are the products defined in section 3.1.1 with the addition of milk and/or its derivatives where the final product must have a dairy flavor and the milk protein content must not be less than 2%.

**3.1.2.1 Toffee** - They are the products defined in section 3.1.2, prepared with the addition of butter or other dairy fat and the milk fat content must not be less than 2%.

**3.1.3 Filled Soft Candy (dairy or not)** – These are product made up of soft candy having covering or crust around with a clearly distinct filling inside, both in defined proportions.

The filling has its own flavor and texture that differentiates it from the topping and is obtained from a mixture of variable composition of sugars, sugar syrups, chewing gum, fruit pulp, honey, chocolate, coffee and other permitted edible ingredients. The filling can be solid, liquid, semi-liquid or pasty.

**3.1.4 Coated Soft Candy (Filled or not)** – These are products defined in section 3.1.1 or 3.1.2, covered by a layer of sugar, chocolate or other permitted edible ingredients.

**3.2.5 Marshmallow**- The product of cooked sugar under defined temperature with aerated agent such as albumin or modified proteins or gelatin with optional materials. It may contain gelling or thickening agent of plant-animal origin alone or in combination and characterized by having a spongy texture, which may be coated and/or filled with other components.

**3.2.6 Gummies** - Products obtained from gelling or thickening agents of animal and plant origin, alone or in combination, and a mixture of natural gums, gelatins, agar-agar, starch, sugars and others substances and permitted additives.

**3.2.7 Jelly candy** - Products that are characterized by including in their composition ingredients such as pectins, agar-agar, modified starch or a combination of them and other components and permitted additives.

## 4. Ingredients

4.1 All ingredients shall conform to the relevant Bangladesh Standard specifications, where available. All materials shall be food grade, halal and also free from foreign matter, harmful microorganisms, insect infestation, objectionable flavours and odours, food additives and processing aids.

### 4.2 Essential ingredients –

- a) Sugar/Sugar syrup
- b) Water

### 4.3 Optional ingredients

- a) Edible vegetable fats and oils
- b) Butter
- c) Milk and milk products
- d) Edible salt
- e) Gelling agents, e.g. gelatin, pectin, agar (halal)
- f) Edible gums
- g) Edible starches
- h) Malt and malt extracts
- i) Chocolate and their derivatives
- j) Cocoa and their derivatives
- k) Honey
- l) Jaggery
- m) Coffee and coffee products
- n) Tea extracts
- o) Fruit and fruit products
- p) Candied fruits and peels
- q) Spices, condiments and their extracts
- r) Egg albumin
- s) Vitamin and Minerals
- t) Protein isolates
- u) Eucalyptus oil, camphor, menthol oil crystals, pepper mint oil and
- v) Thymol

**4.3.1** In the preparation of soft candies, the addition of flavourings, colourings, acidulants, preservatives, humectants, emulsifiers required shall be according to the relevant category of the updated version of CXS 192 or permitted by national legislations.

## 5. Requirements

**5.1** The product shall be in any desired colour, distinctive type, uniform size and proper texture. It shall have pleasant taste, flavor, and attractive appearance and shall have a bite ranging from hard and chewy to soft eating. It shall be free from undesirable smell, rancidity, dirt, filth, adulterants and harmful ingredients. It shall be halal.

**5.2 Hygienic requirement** – During processing, handling, storage and transportation, effective measures must be taken to prevent cross contamination with chemicals, microbial or physical contaminants.

**5.2.1** The product shall be processed and packed under strict hygienic conditions in premises maintained in accordance with BDS 822.

**5.3 Legal requirement** – The product shall in all other aspects comply with the requirements of the legislations enforced in the country.

**5.4** The material shall also comply with the requirements given under Table 1 and shall not contain the poisonous metal in excess of the limits specified in Table 2.

## 6. Packing and Marking

**6.1 Packing** – The material, if wrapped, shall be in food grade, plain or printed waxed paper, foil or cellulose film. In case of printed packaging material, the printing ink shall be non-toxic and shall not come in direct contact with the product. The wrapped or unwrapped material shall be bulk packed in clean, food grade, reasonably air-tight and sound containers that have no effect on product properties. Such containers shall be made of tinplate, glass, plastics, moisture proof paper, cellulose film or any other suitable packing material.

**Table-1 Requirements for Soft Candy**

(Sub clause 5.4)

Sl.	Characteristic	Soft Candy	Dairy/ Milk Candy	Toffee	Marshmallow	Gummies	Jelly candy	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i.	Moisture, percent by mass, max.	10	10	10	20	25	30	Annex-A
ii.	Ash, sulphated, percent by mass, max.	2.5	2.5	2.5	1	1	1	Annex-B
iii.	Acid insoluble ash, percent by mass, max.*	0.2	0.2	0.2	0.2	0.2	0.2	Annex-C
iv.	Sucrose, percent by mass, max.	65	65	65	70	65	65	Annex-D
v.	Protein, percent by mass, min.	-	2	2	-	-	-	Annex-E
vi.	Total Fat, percent by mass (on dry basis), min.	1	4	4	-	-	-	Annex-F
vii.	Milk Fat, percent by mass (on dry basis), min.	-	0.5	2	-	-	-	ISO 17678
viii.	Sulphur di oxide, mg/kg, max.	15	15	15	-	-	-	Annex-G

\* When spices are used as centre filling, the acid insoluble ash shall not be more than 0.4 per cent.

**NOTE** - For caramels with coating or filling, soft caramel (dairy or non-dairy) should be taken as the calculation base.

**Table-2 Microbiological and heavy metals limit for Soft Candy**

(Sub clause 5.4)

Sl.No.	Characteristic	Requirement	Method of Test Ref. to
(1)	(2)	(3)	(4)
ii.	<i>Salmonella</i> , cfu/g	Absent	BDS ISO 6579-1
iii.	Arsenic (as As), mg/kg, max.	1	AOAC 986.15
iv.	Lead (as Pb), mg/kg, max.	1	AOAC 994.02
v.	Cadmium (as Cd), mg/kg, max.	1	AOAC 999.11
vi.	Tin (as Sn), mg/kg, max.	2	AOAC 985.16

**6.2 Marking** – The following particulars shall be marked or labeled and indelibly on each container:

- Name of the product;
- Name and address of manufacturer/importer;
- Batch or code number;
- Net mass (g);
- List of ingredients;
- List of food additives, if used;
- Allergen, if any;
- Date of manufacture
- Date of expiry.
- Maximum Retail Price (MRP); and
- Any other requirements as specified in the current Legislations and Regulation enforced in the country.

**NOTE** - Containers having less than 60 g may not be marked with the particulars mentioned under 5.2. Containers having more than 60 g and less than 120 g shall be marked with the particular given under 5.2 (a), (b), (e), (f), (g) and (h), but may not be marked with the particulars under 5.2 (c), (d) and (i).

**5.2.1** Each package may also be marked with the BSTI Certification Mark.

**NOTE** – The use of BSTI Certification Mark is governed by the provisions of Bangladesh Standards and Testing Institution Act, 2018 and the Rules and Regulations made thereunder. Details of conditions, under which a license for

the use of BSTI Certification Mark may be granted to manufacturers or processors, may be obtained from the Bangladesh Standards and Testing Institution.

## 6. Sampling

6.1 Representative samples of the material shall be drawn as prescribed in Annex-H.

## 7. Tests

7.1 Test shall be carried out as prescribed in the appropriate annexes specified in Col. 6 of Table-1 and col 4 of table 2.

7.2 **Quality of reagents** – Unless specified otherwise, pure chemicals and distilled water (see BDS 833) shall be used where the use of water as a reagent is intended.

**NOTE** – 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

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**Annex - A**  
 [(Table 1, item (i))]  
**Determination of Moisture**

**A. Vacuum Oven Method**

**A.1** If composition of entire product is desired, grind and mix thoroughly. If product is composed of layers or of distinctly different portions and it is desired to examine these individually, separate with knife or other mechanical means as completely as possible, and grind and mix each test portion thoroughly.

**A.2 Apparatus** - The following apparatus is required:

**A.2.1** Dish 5.0 cm in diameter and 2.5cm high, made of aluminium and having a close fitting cover provided with a knob.

**A.2.2** Vacuum oven- fitted with necessary accessories as to provide slow current of dry air through the oven to ensure removal of water vapours.

**A.2.3** Desiccator

**A.2.4** Weighing balance (Accuracy of weighing balance should be  $\pm 0.001$  g)

**A.3 Procedure**

Accurately weigh about 5 g of sample, in a flat dish with tight-fit cover having a diameter of about 75 mm and a height of about 25 mm previously dried and weighed. Distribute the material as evenly as possible over the bottom of the dish by gentle sidewise movements.

Place dish in vacuum oven, remove cover of dish and dry the material for 2 h at  $70 \pm 1$  °C at a pressure not exceeding 50 mm of Hg. During heating admit slow current of air into oven.

Cover dish, transfer to desiccator and weigh soon after room temperature is attained.

Re-dry for 1 h and repeat the process till the difference between the two successive weighing is less than 2 mg. Report percent loss in weight as moisture %.

**A.4 Calculation**

$$\text{Moisture (\%)} = \frac{(W_1 - W_2) \times 100}{W_1 - W}$$

Where,

W = Weight in g, of empty Aluminium dish.

$W_1$  = Weight in g, of Aluminium dish + sample before drying.

$W_2$  = Weight in g, of Aluminium dish + dried sample.

**Annex - B**  
 [(Table 1, item (ii))]  
**Determination of Sulphated Ash**

**B.1 Reagent**

**B.1.1** Concentrated Sulphuric acid- sp.gr. 1.84

**B.2 Procedure**

**B.2.1** Accurately weigh about 5 g of the prepared sample into a 9 cm diameter platinum basin.

**B.2.2** Add 5 ml of sulphuric acid to the material in the dish. Gently heat the dish on a hot plate (in exhaust hood) until the material is well carbonized and then increase the heat until the evolution of sulphuric acid fumes ceases.

**B.2.3** Ash the carbonized matter in a muffle furnace at  $550 \pm 25$ °C.

**B.2.4** Cool the ash and moisten it with 2-3 ml of sulphuric acid.

**B.2.5** Heat strongly on a hot plate until sulphuric acid fumes ceases to be evolved and finally ash in the muffle furnace at  $550 \pm 25$  °C for 2h.

**B.2.6** Cool in a desiccator and weigh.

**B.2.7** Heat again in a muffle furnace for 30 min at  $550 \pm 25$  °C. Cool in a desiccator and weigh.

**B.2.8** Repeat the process of heating in the muffle furnace for 30 min, cooling and weighing till the difference between two successive weighing is less than 1 mg. Record the lowest mass.

**B.3 Calculation**

$$\text{B.3.1 Sulphated Ash, percent by mass} = \frac{100 M_1}{M_2}$$

where

$M_1$  = mass in g of the sulphated ash, and

$M_2$  = mass in g of the prepared sample taken for the test.

**Annex - C**

[Table 1, item (iii)]

**Determination of Acid Insoluble Ash****C.1 Reagent**

**C.1.1** Dilute Hydrochloric acid (Approx 5 N)- Concentrated Hydrochloric acid (445 ml) diluted to 1 L using distilled water

**C.1.2** Silver nitrate

**C.1.3** Nitric acid

**C.2 Procedure**

**C.2.1** Weigh accurately about 5 g of the prepared sample in a tared, clean and dry platinum dish of 100 ml capacity.

**C.2.2** Carbonize the material in the dish with the flame of a burner. Complete the ignition by keeping in a muffle furnace at  $550 \pm 25$  °C until gray ash results. Cool in a desiccator.

**C.2.3** To the ash, add 25 ml of the dilute hydrochloric acid, cover with a watch glass and heat on a small flame of a burner to near boiling.

**C.2.4** Allow it to cool and filter the contents of dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper and residue with hot water until the washings are free from chlorides (To check this, add few drops of 2M Nitric acid and 0.1 M Silver nitrate solution to the filtrate obtained. No precipitate or milky turbidity should occur in the solution, if it is chloride-free.)

**C.2.5** Return the filter paper and the residue to the dish. Keep it in an air oven maintained at  $105 \pm 2$  °C for about 3 h. Ignite in the muffle furnace at  $550 \pm 25$  °C for 1h.

**C.2.6** Cool the dish in a desiccator and weigh.

**C.2.7** Heat again for 30 min in the muffle furnace, cool and weigh.

**C.2.8** Repeat this process of heating for 30 min, cooling and weighing till the difference between two successive weighing is less than one milligram. Note the lowest mass.

**C.3 Calculation**

$$\text{Acid insoluble Ash, percent by mass} = \frac{100 M_1}{M_2}$$

where

$M_1$  = mass in g of the acid insoluble ash, and

$M_2$  = mass in g of the prepared sample taken for the test.

**Annex - D**

[Table 1, item (iv)]

**Determination of Sucrose****D.1 Apparatus**

**D.1.1 Analytical balance**

**D.1.2 Normal weight-** 26.000g (brass weight) as weighed in air at 20°C.

**D.1.3 Basin-** made of nickel or german silver, large enough to hold twice the normal weight of sugar.

**D.1.4 Volumetric flasks-** four; One of capacity 200 ml; one of capacity 110 ml with 100 ml and 110 ml marks; and two of capacity 100 ml each; calibrated at 20°C.

**D.1.5 Conical flask** of capacity 250 ml,

**D.1.6 Long-stemmed funnel-** with a stem long enough to extend below the neck of the Volumetric flask (see D.1.4)

**D.1.7 Stemless funnel-** capable of holding 200 ml.

**D.1.8 Glass Cylinder-** capable of holding 200 ml.

**D.1.9 Watch glass-** large enough to cover the stemless funnel

**D.1.10 Pipette-** 50ml, Calibrated at 20°C

**D.1.11 Thermometer-** having a range 0° to 100° C and graduated at every 0.1° C

**D.1.12 Saccharimeter-** graduated in international sugar scale and provided with a 200 mm tube. The Saccharimeter should be stored in a cabinet, the inside of which is maintained at 20°C. Where this is not possible necessary corrections shall be applied based on the characteristics of the particular instrument.

## D.2 Reagents

**D.2.1 Anhydrous lead subacetate-** finely powdered.

**D.2.2 Deleading solution-** Dissolve 7g of disodium hydrogen phosphate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ) and 3g of potassium oxalate ( $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ ) in water and make up the volume to 100ml.

**D.2.3 Sodium chloride solution-** Dissolve 231.50g of sodium chloride in water and make up the volume to one litre in a volumetric flask.

**D.2.4 Hydrochloric acid-** sp. gr. 1.1029, at 20° ± 4°C

**D.3 Test Temperature-** The polarization of the material shall be determined at 20°C, as far as possible.

## D.4 Procedure

**D.4.1 Preparation of the solution-** Weigh accurately in the basin twice the normal weight (see D.1.2) of the powdered material on the analytical balance. Transfer this quantity to the 200 ml volumetric flask with the aid of the long-stemmed funnel. Rinse the basin and the funnel with water, and transfer the washings to the volumetric flask taking care that the volume in the flask does not exceed 160 ml. Bring the sugar into solution by gently swirling the flask and make up the volume to 200ml with water. Clarify the solution by adding anhydrous lead subacetate in small quantities until nearly all the impurities have been precipitated. If necessary add 1-2 ml of alumina cream to clarify the turbidity. Place the stemless funnel over the glass cylinder and fix in the funnel a fluted cone of dry filter paper, large enough to hold 200 ml of the liquid. Pour the whole of the defecated solution on to the fluted cone of dry filter paper and cover it immediately with the watch glass. Collect the filtrate in the cylinder.

**D.4.2 Deleading the filtrate-** Take 100 ml of the filtrate in a 110 ml volumetric flask. Add the deleading solution (see D.2.2) in small quantities at a time to the whole of the filtrate until the excess of lead is completely precipitated. Make up the volume to 110 ml with water and filter.

**D.4.3 Polarization-** Pipette two portions of 50 ml each of the clear filtrate (see D.4.2) separately into a 100 ml volumetric flask and a 250 ml. conical flask and treat them separately as follows:

a) **Polarization before inversion-** To the filtrate contained in the 100 ml volumetric flask, add 1.10 ml sodium chloride solution and make up to volume with water. Polarize this solution in the saccharimeter using 200 mm tube enclosed in a water jacketed tube at the test temperature (see D.3). Note the reading (p) of the saccharimeter for the solution before inversion.

b) **Polarization after inversion-** To the filtrate in the conical flask, add 10 ml of hydrochloric acid solution. Mix the contents of the flask rotating it. Insert a thermometer in the conical flask containing the reaction mixture. Immerse the conical flask in a hot water bath and heat the contents to 60°C. Agitate the solution continuously for 3 minutes and allow it to remain in the bath for a total time of 10 minutes. Cool the contents of the conical flask to 20°C. Transfer quantitatively the same to a 100 ml volumetric flask. Make up the volume to 100ml. Polarize this solution in the saccharimeter at the test temperature (see D.3). Note the reading (Pt) of the saccharimeter for the solution after inversion.

## D.5 Calculation

$$\text{Sucrose content of the material, percent by mass.} = \frac{100(p - p')}{132.56 + 0.0794(m - 13) - 0.53(t - 20)}$$

where

P = reading of the saccharimeter observed at t° C before inversion (see D.4.3 (a), calculated to normal weight basis.

P' = reading of the saccharimeter observed at t° C after inversion (see D.4.3 (b), calculated to normal weight basis.

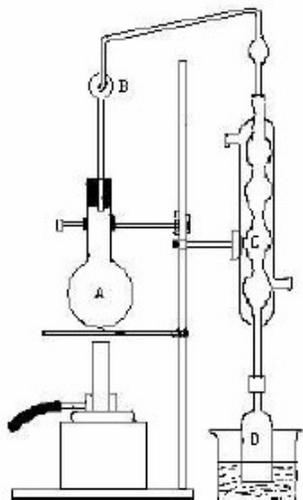
m = in g of the moisture free material per 100 ml of the solution (see D.4.3 (b), and

t = temperature in degrees celsius at which the polarization is carried out.

**Annex - E**  
**[Table 1, Item (v)]**  
**Determination of Total Protein**

### E.1 Apparatus

**E.1.1** A recommended distillation assembly is shown below. The assembly consists of a round bottom flask A of 1000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube B. the other end of the bulb B is connected to the condenser C which is attached, by means of a rubber tube, to a dip tube D which dips into a known quantity of standard sulphuric acid contained in a beaker of 250 ml capacity.



- E.1.2** Kjeldahl flask – 500 ml capacity
- E.1.3** Weighing balance
- E.1.4** Burner
- E.1.5** Round bottom flask

### E.2 Reagents

- E.2.1** Anhydrous Sodium sulphate
  - E.2.2** Copper sulphate
  - E.2.3** Concentrated Sulphuric acid- sp gr 1.84
  - E.2.4** Sodium hydroxide solution- Dissolve about 225 g of sodium hydroxide in 500 ml of water
  - E.2.5** Standard Sulphuric acid- 0.1 N
  - E.2.5** Methyl red indicator solution- Dissolve 1g of methyl red in 200ml of Rectified spirit (95 %v/v)
  - E.2.7** Standard sodium hydroxide solution -0.1 N
- \*Boric acid can also be used instead of sulphuric acid.

### E.3 Procedure

- E.3.1** Transfer carefully about 1 or 2 g of the sample accurately weighed, to the Kjeldhal flask, taking precaution to see that particles of the material do not stick to the neck of the flask.
- E.3.2** Add about 10 g of anhydrous sodium sulphate, 0.2 to 0.3 g of copper sulphate and 20 ml of concentrated sulphuric acid.
- E.3.3** Place the flask in an inclined position. Heat below the boiling point of the acid until frothing ceases. Increase heat until the acid boils vigorously and digests for 30 min after the mixture becomes clear and pale green in colour. Cool the flask.
- E.3.4** Transfer quantitatively to the round-bottomed flask with water, the total quantity of water used being about 200 ml. Add a few pieces of pumice stones to avoid bumping. Add about 50 ml of Sodium hydroxide solution (which is sufficient to make the solution alkaline) carefully through the side of the flask so that it does not mix with the acid solution but forms a separate layer below the acid layer.
- E.3.5** Assemble the apparatus as shown above taking care that the dip tube extends below the surface of the standard sulphuric acid solution contained in the beaker.
- E.3.6** Mix the contents of the flask by shaking and distil until all the ammonia has passed over into the standard sulphuric acid.

**E.3.7** Shut off the burner and immediately detach the flask from the condenser. Rinse the condenser thoroughly with water into the beaker. Wash the dip tube carefully so that all traces of the condensate are transferred to the beaker.

**E.3.8** When all the washings have been drained into the beaker, add two or three drops of methyl red indicator solution and titrate with the standard sodium hydroxide solution.

**E.3.9** Carry out a blank determination using all reagents in the same quantities but without the sample to be tested.

#### **E.4 Calculation**

$$\text{Total Protein (N x 6.25), \% by mass} = \frac{8.75 \times (B-A) \times N}{M}$$

Where,

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in the blank determination

A = volume in ml of the standard sodium hydroxide solution used to neutralize the excess of the acid in the test with the material

N = Normality of the standard sodium hydroxide solution

M = mass in g of the material taken for the test

### **Annex - F** **[Table 1, Item (vi)]** **Determination of Fat**

Two methods for the determination of fat are given. Any of these may be used. If the confectionery is known to contain milk, method No.2 (Roese-Gottlieb Method) wherein ammonia is used to dissolve the milk protein before fat extraction may be used.

#### **F.1 Simple Extraction Method-**

##### **F.1.1 Apparatus**

**F.1.1.1** Mojonnier fat extraction tube or any other similar apparatus

**F.1.1.2** Flasks

**F.1.1.3** Weighing balance

##### **F.1.2 Reagents**

**F.1.2.1** Peroxide free Diethyl ether

**F.1.2.2** Petroleum ether

##### **F.1.3 Procedure**

**F.1.3.1** Dissolve 10g sample in 10 ml warm water, and introduce into Mojonnier fat extraction tube or similar apparatus.

**F.1.3.2** Add 25 ml peroxide free diethyl ether. Cork the tube and shake vigorously for 1 minute.

**F.1.3.3** Add 25 ml of petroleum ether and shake again vigorously for 30 sec. Let it stand for 30 min or until separation is complete.

**F.1.3.4** Draw off the ether layer containing fat in a previously dried and weighed flask. Repeat the extraction twice.

**F.1.3.5** Pool the ether extract, recover excess solvent and dry the fat for 1 h at 100°C. Cool and weigh.

**F.1.3.6** Fat must be dried by keeping the flasks for 30 min and weighed, till constant mass is achieved.

##### **F.1.4 Calculation**

$$\text{Fat, \% on dry basis} = \frac{M_1 \times 100}{M_2 \times (100 - M)}$$

Where,

M<sub>1</sub> = Weight in g of the fat

M<sub>2</sub> = Weight in g of sample taken

M = Moisture % in the sample

#### **F.2 Roese-Gottlieb Method**

##### **F.2.1 Apparatus**

**F.2.1.1** Mojonnier fat extraction tube or any other similar apparatus

**F.2.1.2** Flasks

**F.2.1.3** Weighing balance

**F.2.2 Reagents**

**F.2.2.1** Concentrated Ammonia - sp.gr. 0.88

**F.2.2.2** Ethyl Alcohol - 95 to 96 %percent (v/v)

**F.2.2.3** Diethyl ether - sp.gr. 0.720 (peroxide free)

**F.2.2.4** Petroleum ether – boiling range 40 to 60 C, recently distilled

**F.2.3 Procedure**

**F.2.3.1** Introduce 4g sample into a Mojonnier extraction tube or similar apparatus.

**F.2.3.2** Dilute to 10 ml with water.

**F.2.3.3** Add 1.2 ml Ammonia solution and mix thoroughly.

**F.2.3.4** Add 10 ml alcohol and mix.

**F.2.3.5** Then add 25 ml diethyl ether and shake vigorously for about 30 sec and finally add 25 ml petroleum ether and shake again for about 30 sec.

**F.2.3.6** Let stand for 20 min or until separation of liquids is complete.

**F.2.3.7** Draw off as much as possible of ether fat solution (usually 0.5 to 0.8 ml is left) into a weighed flask through a small rapid filter.

**F.2.3.8** Again extract liquid remaining in tube, this time with 15 ml each of ether and petroleum ether; shake vigorously for about 30 sec with each solvent and let settle. Proceed as above, washing mouth of tube and filter with a few ml of mixture of equal parts of two solvents.

**F.2.3.9** For accuracy, repeat extraction. If previously solvent-fat solution has been drawn off closely, third extraction usually yields approximately up to 1 mg fat or about 0.02 % with 4 g sample.

**F.2.3.1** Slowly evaporate solvent on steam bath and then dry fat in an oven maintained at 100 oC to constant mass.

**F.2.3.1** Test purity of fat by dissolving in a little petroleum ether. If residue remains, wash out fat completely with petroleum ether, dry the residue, weigh and calculate the mass of the fat.

**F.2.3 Calculation**

$$\text{Fat, \% on dry basis} = \frac{M_1 \times 100}{M_2}$$

Where,

$M_1$  = Weight in g of the fat

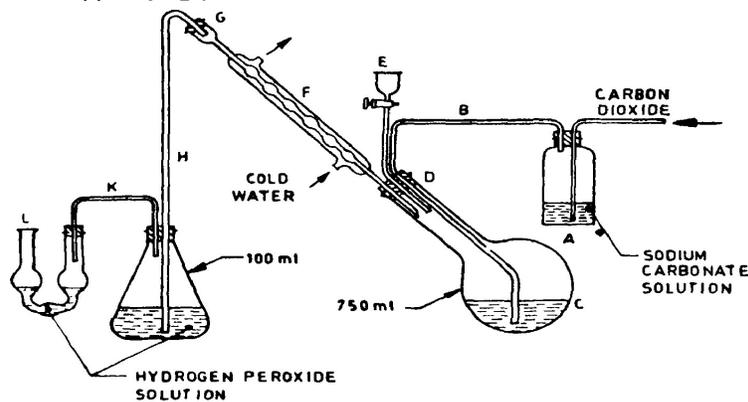
$M_2$  = Weight in g of sample taken

**Annex - G**

[Table 1, Item (vii)]

**Determination of Sulphur Dioxide**

**G.1 Apparatus-** The apparatus as assembled is shown below.



**FIG. 2 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF SULPHUR DIOXIDE**

**G.2 Reagents**

**G.2.1** Concentrated hydrochloric acid- sp.gr. 1.16

**G.2.2** Carbon dioxide gas- from a cylinder

**G.2.3** Bromo phenol blue indicator solution – Dissolve 0.1 g of bromo phenol blue in 3.0 ml of 0.05 N sodium hydroxide solution and 5ml of ethyl alcohol (90%, v/v) by gently warming. Make up the volume of the solution with ethyl alcohol (20 %, v/v) to 250ml in a volumetric flask.

- G.2.4** Sodium carbonate solution [10% (m/v)]- Sodium carbonate (10 g) dissolved in water (100 ml).
- G.2.5** Standard sodium hydroxide solution - approximately 0.1 N, standardized at the time of the experiment using bromo phenol blue indicator solution.
- G.2.6** Hydrogen peroxide solution – Dilute 30 percent (m/v) hydrogen peroxide solution with about twice its volume of water and neutralize the free sulphuric acid that may be present in the hydrogen peroxide solution with barium hydroxide solution, using bromo phenol blue indicator solution. Allow the precipitate of barium sulphate to settle and filter. Determine the concentration of hydrogen peroxide in the filtrate by titrating with standard potassium permanganate solution. Dilute the filtrate with cold water so as to obtain a 3% (m/v) solution of hydrogen peroxide

### G.3 Method of analysis

- G.3.1** Assemble the apparatus as shown in figure. Introduce into the flask C, 300 ml of water and 20 ml of concentrated hydrochloric acid through the dropping funnel E.
- G.3.2** Run a steady current of cold water through the condenser F.
- G.3.3** Boil the mixture contained in the flask for a short time to expel the air from the system in current of carbon dioxide gas previously passed through the wash bottle A.
- G.3.4** Weigh accurately about 100 g of the sample and mix with the minimum quantity of water so as to make the diluted sample easily flow down to the dropping funnel.
- G.3.5** Introduce the diluted material into the flask C through the dropping funnel E. Wash the dropping funnel with a small quantity of water and run the washing into the flask C.
- G.3.6** Again boil the mixture contained in the flask C in a slow current of carbon dioxide gas (passed previously through the wash bottle A) for one hour.
- G.3.7** Just before the end of the distillation, stop the flow of water in the condenser. (This causes the condenser to become hot and drives over residual traces of sulphur dioxide retained in the condenser.) When the delivery tube H, just above the Erlenmeyer flask j, becomes hot to touch, remove the stopper J immediately.
- G.3.8** Wash the delivery tube H and the contents of the Peligot tube L with water into Erlenmeyer flask.
- G.3.9** Cool the contents of the Erlenmeyer flask to room temperature, add a few drops of bromo phenol blue indicator
- G.3.10** Titrate with standard sodium hydroxide solution. (Bromo phenol blue is unaffected by carbon dioxide and gives a distinct change of color in cold hydrogen peroxide solution).
- G.3.11** Carry out a blank determination using 20 ml of conc. hydrochloric acid diluted with 300 ml of water.

### G.4 Calculation

$$\text{Sulphur dioxide, mg/kg} = \frac{0.032000 (V-v) \times 1000 \times 1000 \times N}{W}$$

Where,

V = volume in ml of standard sodium hydroxide solution required for the test with the material

v = volume in ml of standard sodium hydroxide solution required for the blank determination;

N = normality of standard sodium hydroxide solution; and

W = weight in g of the material taken for the test.

## Annex - H

(Clause 6)

### Sampling

**H.1** General requirements for sampling – In drawing, preparing and handling samples, the following precautions and directions shall be observed.

**H.1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**H.1.2** The sampling instruments shall be clean and dry when used.

**H.1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for sampling from adventitious contaminations.

**H.1.4** The samples shall be placed in clean and dry glass or polythene containers. The sample containers shall be of such a size that they are almost completely filled by the sample. The samples shall be filled loose and not pressed in the container.

**H.1.5** Each container shall be sealed air-tight after filling and marked with the name of the material, date of sampling, season of manufacture, batch number, name of manufacturer and other important particulars of the batch. The Samples shall be protected from light as far as possible.

**H.1.6** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

## H.2 Scale of Sampling

**H.2.1 Lot-** All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each shall constitute separate lots.

**H.2.1.1** Samples shall be tested for each lot for ascertaining its conformity to the requirements of the corresponding specification.

**H.2.2** The number of containers to be sampled from each lot shall depend on the size of the lot and it shall be done according to the following table.

**Minimum Number of Containers to be Selected for Sampling from Various Size of Lots**  
(Sub-clause H.2.2)

Lot size N (1)	Number of containers to be selected (n) for size of the containers	
	500g and Above (2)	Less than 500g (3)
Up to 25	3	6
26 to 100	4	6
101 to 300	5	9
301 to 500	7	12
501 and above	9	15

**H.2.3** The containers shall be selected at random from the lot and for this purpose a random number table as agreed to between the purchaser and the supplier shall be used. If such a table is not available, the following procedure shall be adopted:

Starting from any container in the lot count them as 1,2,3,..... up to r in a systematic manner, where r is the integral part of  $N/n$ , N being the total number of containers in the lot, n the number of containers to be selected (see Table I). Every  $r^{\text{th}}$  container thus counted shall be separated until the requisite number n container is obtained from the lot to give the sample for the test.

## H.3 Test and Referee Samples

**H.3.1 Test samples-** From each of the selected containers, with the help of suitable sampling instrument, approximately equal quantity of material shall be taken out so as to make a composite sample of about 1kg. Thus sample shall be thoroughly mixed and divided into three equal parts and transferred to clean and dry glass containers, sealed air tight and labeled with particulars as given in H.1.5. One of these composite samples shall be for the purchaser/inspecting authority, one for the vendor and the third for the referee.

Note- In case the materials of various types are packed in the same container, the material of each type shall be separated. The sample shall be prepared as given in H.3.1 and tested for conformity to requirements carried out separately for each type.

**H.3.2 Referee Sample-** Referee sample shall consist of the composite sample marked for this purpose and shall bear the seal of the purchaser/inspecting authority and the vendor. This shall be kept at a place as agreed to between the two parties and shall be used in case of disputes between them.

## H.4 Number of tests and Criteria for conformity

**H.4.1** All the requirements of the corresponding specification shall be tested on the composite samples (H.3.1).

**H.4.2** The lot shall be declared as conforming to the specification when the composite sample tested for various requirements shall satisfy the corresponding requirements of the specification.

**Annex-J**  
**(Clause-2)**  
**List of relevant standards**

BDS No	Title
BDS 103	Methods of rounding off numerical value
BDS 822	Code of Hygienic Conditions for Food Processing Units.
BDS 833	Water for Laboratory use
BDS ISO 6579-1	Microbiology of the food chain - Horizontal method for the detection, enumeration and serotyping of <i>Salmonella</i> – Part 1: Detection of <i>Salmonella</i> spp
ISO 17678:2019	Milk and milk products — Determination of milk fat purity by gas chromatographic analysis of triglycerides