SRI LANKA STANDARD 181:1983
UDC 637.141

SPECIFICATION FOR
RAW AND PROCESSED MILK
(FIRST REVISION)

BUREAU OF CEYLON STANDARDS

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(FIRST REVISION)

SLS 181:1983
(Attached Corrigendum No1)

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AMENDMENT NO. 1 APPROVED ON 1986-04-30.

SLS 181:1983 SPECIFICATION FOR RAW AND PROCESSED MILK (FIRST REVISION)

Page 4

Clause 3.8

Delete the existing clause and substitute the following:

"**flavoured milk** : The product prepared from liquid milk or milk powder or condensed milk, sugar, chocolate or cocoa or coffee and other permitted flavouring and with or without permitted food colour, stabilizer and buffering agent."

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This standard does not purport to include all the necessary provisions of a contract.

SRI LANKA STANDARD
SPECIFICATION FOR RAW AND PROCESSED MILK
(FIRST REVISION)

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Bureau of Ceylon Standards on 1983-11-29, after the draft, finalized by the Drafting Committee on Milk Products had been approved by the Agricultural and Food Products Divisional Committee.

This revision covers 13 types of milk based on processing, constitution and flavour. A standard code of designation of the different types has also been included to facilitate marking and labelling. This covers revised methods of sampling from retail containers and includes methods of sampling from bulk containers.

This specification is subject to the provisions of Food Act No. 26 of 1980 and the regulations framed thereunder.

All standard values are given in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with **CS 102**. The number of significant places retained in the rounded off value should be the same as that of the specified value in this specification.

In the preparation of this specification, the assistance obtained from the publications of the Australian Standards Institution, the British Standards Institution and the Indian Standards Institution is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements and methods of sampling and test for raw and processed milk.

2 REFERENCES

CS 102 Presentation of numerical values

SLS 428 Random sampling methods

SLS 516 Microbiological test methods

Part 1 General guidance for enumeration of micro-organisms, aerobic plate count

Part 3 Detection and enumeration of coliforms, faecal coliforms and *Escherichia coli*.

3 DEFINITIONS

For the purpose of this specification, the following definitions shall apply.

3.1 raw milk: This is the fresh lacteal secretion (practically free from colostrum) obtained by the complete milking of one or more healthy cows or buffaloes without the addition of any substances or abstraction of fat or any other constituents.

3.2 pasteurized milk: The milk that has been heated to at least 63 °C and held continuously at that temperature for at least 30 minutes or heated to at least 71.5 °C and held at that temperature continuously for at least 15 seconds or any other approved *temperature-time combination* equivalent thereto, that will serve to give a negative phosphatase test and cooled immediately to a temperature of 10 °C or less and kept at that temperature.

3.3 homogenized milk: The milk that has been subjected to such temperature and pressure which will ensure break up of the fat globules into much smaller particles that do not rise to form a fat layer.

3.4 sterilized milk: The milk that has been heated without appreciable loss of volume to a temperature of 100 °C, for a length of time sufficient to kill all organisms present and contained for delivery in hermetically sealed containers.

3.5 standardized milk: The milk that has been standardized to fat and solids other than milk fat.

3.6 toned milk: The milk from which, part of the milk fat has been removed.

3.7 skimmed milk: The milk from which almost all the milk fat has been removed to a limit not more than 0.5 per cent milk fat.

3.8 flavoured milk: The product prepared from milk, sugar and chocolate, cocoa, coffee or other permitted flavouring and with or without permitted food colour, stabilizer and buffering agent.

3.9 ultra heat treated milk: The milk that has been heated without appreciable loss of volume to a temperature of 132.2 °C for not less than one second and shall then be filled and sealed aseptically into sterile containers in which it is to be supplied to the consumer.

4 TYPES

Milk shall be of the following types:

			Abbreviations
a)	Type 1	Raw milk-cow milk;	C
		buffalo milk;	B
b)	Type 2	Pasteurized unhomogenized;	PU
c)	Type 3	Pasteurized homogenized;	PH
d)	Type 4	Sterilized;	S
e)	Type 5	Standardized pasteurized;	SP
f)	Type 6	Standardized sterilized;	SS
g)	Type 7	Toned pasteurized;	TP
h)	Type 8	Toned sterilized;	TS
j)	Type 9	Skimmed pasteurized;	KP
k)	Type 10	Skimmed sterilized;	KS
l)	Type 11	Flavoured pasteurized;	FP
m)	Type 12	Flavoured sterilized;	FS
n)	Type 13	Ultra heat-treated milk;	UHT

5 REQUIREMENTS

5.1 All types of milk other than flavoured milk shall be white or light cream in colour and free from abnormal taste or odour. They shall be free from any added colour, preservative or other foreign substances.

5.1.1 Sterilized milk shall show no evidence of scorching. No visible cream line or cream plug shall form in the milk within 5 days of preparation.

5.1.2 Flavoured milk may be coloured with permitted colours laid down under the Food Act No. 26 of 1980. It shall be free from abnormal taste and odour.

5.2 The milk shall also conform to the requirements given in Table 1.

6 PACKAGING

6.1 Processed milk shall be filled in sanitized glass bottles, cans or any other suitable container and hermetically sealed.

6.2 Raw milk shall be supplied in containers as agreed between buyer and seller.

7 MARKING

7.1 The following particulars shall be marked legible and indelible on each container.

a) Type or abbreviation (see 4);

NOTES

1 Abbreviated marks should be used only in cases where marking of the type name in full is practically difficult.

2 In case of types 2, 3 and 4 the origin of milk shall be indicated as "cow" or "buffalo".

b) Name and address of the manufacturer,

c) Net volume in millilitre;

d) Date of manufacture;

e) Batch or code number.

7.2 The containers may also be marked with the Certification Mark of the Bureau of Ceylon Standards illustrated below on permission being granted for such marking by the Bureau of Ceylon Standards.

NOTE – The use of the Bureau of Ceylon Standards Certification Mark (SLS Mark) is governed by the provisions of the Bureau of Ceylon Standards Act and the regulations framed thereunder. The SLS mark on products covered by a Sri Lanka Standard is an assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control, which is devised and supervised by the Bureau and operated by the producer. SLS marked products are also continuously checked by the Bureau for conformity to that standard as a further safeguard. Details of conditions under which a permit for the use of the Certification Mark may be granted to manufacturers or processors may be obtained from the Bureau of Ceylon Standards.

TABLE 1 - Requirements for milk

Item	Characteristic	Requirements													
		Raw milk Type 1		Type 2 Pasteurized unhomogenized	Type 3 Pasteurized homogenized	Type 4 Sterilized	Type 5 Standardized pasteurized	Type 6 Standardized sterilized	Type 7 Toned pasteurized	Type 8 Toned sterilized	Type 9 Skimmed pasteurized	Type 10 Skimmed sterilized	Type 11 Flavoured pasteurized	Type 12 Flavoured sterilized	Type 13 Ultra heat-treated	Methods of test (Ref. to Appendices)
		Cow	Buffalo													
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)
i	Milk fat per cent by mass, min.	3.5	7	3.5	3.5	3.5	3.25	3.25	2.0	2.0	0.5 (max)	0.5 (max)	2	2	3.25	A
ii	Milk solids other than milk fat (per cent by mass, min.)	8.5	9	8.5	8.5	8.5	8.25	8.25	8.5	8.5	8.5	8.5	7.2	7.2	8.25	B & C
iii	Creaming index, max.	-	-	-	20	20	-	20	-	-	-	-	-	-	20	D
iv	Phosphatase activity	-	-	to satisfy the test		-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	-	E
v	Turbidity test	-	-	-	-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	to satisfy the test	F
vi	Methylene blue reduction test	-	-	to satisfy the test		-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	to satisfy the test	-	-	G
vii	Colony count per ml not more than*	-	-	30000	30000	-	30000	-	30000	-	30000	-	30000	-	-	
viii	Coliform**	-	-	-	absent in 1 ml	-	absent in 1 ml	-	absent in 1 ml	-	absent in 1 ml	-	absent in 1 ml	-	-	

* SLS 516 Part 1

** SLS 516 Part 5

8 SAMPLING

8.1 Batch

All the containers, containing milk of one type and obtained from the same process of manufacture shall be grouped to constitute a batch.

8.2 Lot

Part or whole of the batch as defined in 8.1.

8.3 Sampling equipment (see Appendix J)

8.3.1 Plunger or agitator.

8.3.2 Dipper.

8.3.3 Plunger-dipper.

8.4 General requirements of sampling

In the drawing, preparation, storage and handling of samples, the following precautions shall be observed.

8.4.1 *Sampling for chemical analysis*

8.4.1.1 Samples shall be taken in a protected place not exposed to damp air and dust.

8.4.1.2 The sampling equipment and containers shall be dry and clean.

8.4.1.3 Samples shall be stored such that the temperature of the contents does not vary unduly from the ambient temperature.

8.4.2 *Sampling for microbiological analysis*

8.4.2.1 Samples shall be drawn under aseptic conditions.

8.4.2.2 All sampling equipment and containers shall be sterilized by either of the following methods.

a) Heating in a hot air-oven for not less than 2 h at 160 °C;

OR

b) Autoclaving for not less than 15 min at 120 °C.

8.4.2.3 Samples shall be held between 6 °C and 10 °C and the testing shall be done as soon as possible and never later than 24 h after sampling.

8.4.3 *Sampling from bulk containers*

8.4.3.1 If the samples are taken from bulk containers in addition to the requirements given in 8.4.1 or 8.4.2, the following precautions shall be observed.

a) Milk shall be thoroughly mixed, by pouring from one vessel to another, by plunging or by mechanical agitation.

b) With large containers, particular care is necessary to ensure that the milk is thoroughly mixed and agitation shall be continued until this has been achieved.

c) The sample shall be taken immediately after mixing. When a plunger-dipper is used, the contents of the dipper shall be emptied after thorough mixing of the material to be sampled and the dipper shall then be refilled to obtain a representative sample.

d) Samples shall be obtained separately for chemical and microbiological analysis.

8.5 Scale of sampling

8.5.1 Samples shall be tested from each lot for ascertaining conformity of the lot, to the requirements of this specification.

8.5.2 *Sampling from retail containers*

8.5.2.1 The number of retail containers to be drawn from a lot shall be as given in Table 2.

TABLE 2 - Scale of sampling for retail containers

Number of retail containers in the lot	No. of retail containers to be selected	
	Microbiological examination	Chemical examination
(1)	(2)	(3)
Up to 500	2	3
501 to 1 000	2	4
1 001 to 5 000	3	5
5 001 to 10 000	3	6
10 001 and above	5	8

8.5.2.2 The retail containers shall be selected at random. In order to ensure randomness of selection, random number tables as given in SLS 428 shall be used.

8.5.3 *Sampling from bulk containers*

8.5.3.1 Samples for microbiological analysis

Draw with a sampling instrument a 350 ml from top, middle and bottom portion of bulk container. The material so obtained shall be mixed under aseptic conditions and shall be transferred to the sample container. Samples shall be obtained from each bulk container in the lot.

8.5.3.2 Samples for chemical analysis

Draw with a sampling instrument a 250 ml sample from top, middle and bottom portions of bulk container. The material so obtained shall be mixed and transferred to the sample containers. Samples shall be obtained from each bulk container in the lot.

8.6 Number of tests

8.6.1 *Retail containers*

8.6.1.1 Each container selected as in 8.5.2 shall be examined for packaging and marking requirements.

8.6.1.2 Tests for chemical and microbiological requirements of this specification shall be carried out on each container selected as in 8.5.2.

8.6.2 *Bulk containers*

Samples from each bulk container selected as in 8.5.3 shall be individually tested for chemical and microbiological requirements of this specification.

8.7 Reference sample

8.7.1 If a reference sample is required for chemical examination, number of containers selected from the lot shall be three times the number specified in Table 2 in case of retail containers and containers so obtained, shall be divided into three equal sets. In case of bulk containers, sample, from each bulk container shall be divided into three equal parts to form three sets.

8.7.1.1 One of the three sets shall be for the purchaser, the other for the vendor and the third for the reference.

9 METHODS OF TEST

Tests shall be carried out as prescribed in the appropriate appendices given in Table 1.

10 CONFORMITY TO STANDARD

The lot shall be declared as conforming to the requirements of this specification, if the following conditions are satisfied.

10.1 Retail container

10.1.1 Each container examined as in 8.6.1.1 and marking requirements.

10.1.2 Each container tested as in 8.6.1.2 satisfies the relevant requirements.

10.2 Bulk containers

Samples from each bulk container tested as in 8.6.2 satisfy the relevant requirements.

APPENDIX A

DETERMINATION OF MILK FAT (ROSE-GOTLIEB METHOD)

A.1 APPARATUS

A.1.1 *Fat-extraction apparatus,*

Either of the following apparatus may be used:

a) A fat-extraction tube conforming to the dimensions and capacities given in Fig. 1, 2 or 3 fitted as shown with either a wash-bottle top or a siphon, carrying the two tubes in a two-holed bark cork and provided also with a ground-glass stopper or with a solid bark cork. The solid bark cork used to close the tube shall be sound, free from pores and channels which would allow leakage of solvent, and previously extracted with ether. The narrow tube with the hook-shaped lower end (see Fig. 1, 2 and 3) is a sliding fit in the cork and of such length that the opening at its lower end may be placed, if necessary, at a distance of 25 mm from the bottom of the tube.

NOTE - Other modifications of the Rohrig tube of the same capacity may also be used.

b) A Mojonnier fat-extraction tube of the dimensions and capacity given in Fig. 3, closed with a solid bark or ground-glass stopper.

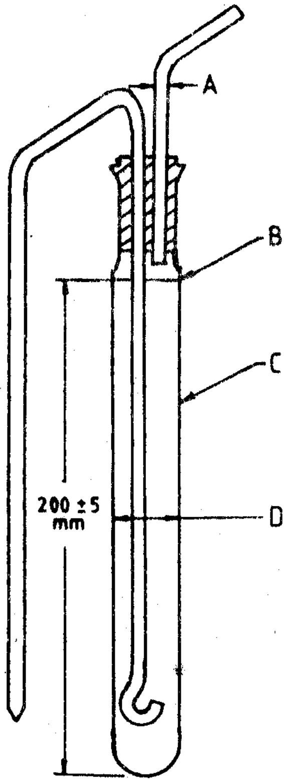

Fig. 1a - With siphon fitting

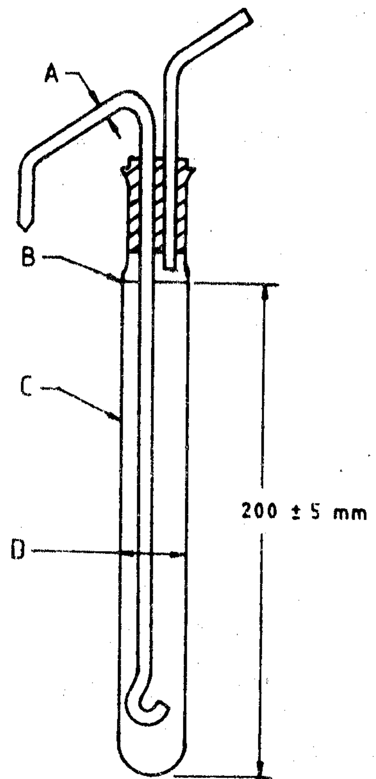

Fig. 1b - With wash-bottle fitting

A External diameter of tubing 3.5 ± 0.5 mm

B Capacity to this level with fittings removed 105 ± 5 ml

C Wall thickness 1.5 ± 0.5 mm

D Internal diameter 26 ± 1 mm

FIGURE 1 Fat-extraction apparatus

Minimum bore 4 mm (Max external diameter 6 mm)

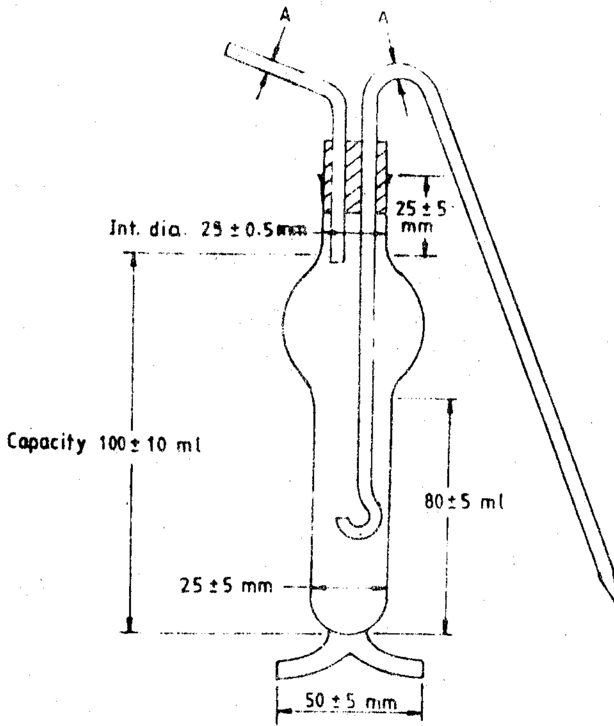

FIGURE 2 Fat extraction apparatus (with siphon fitting)

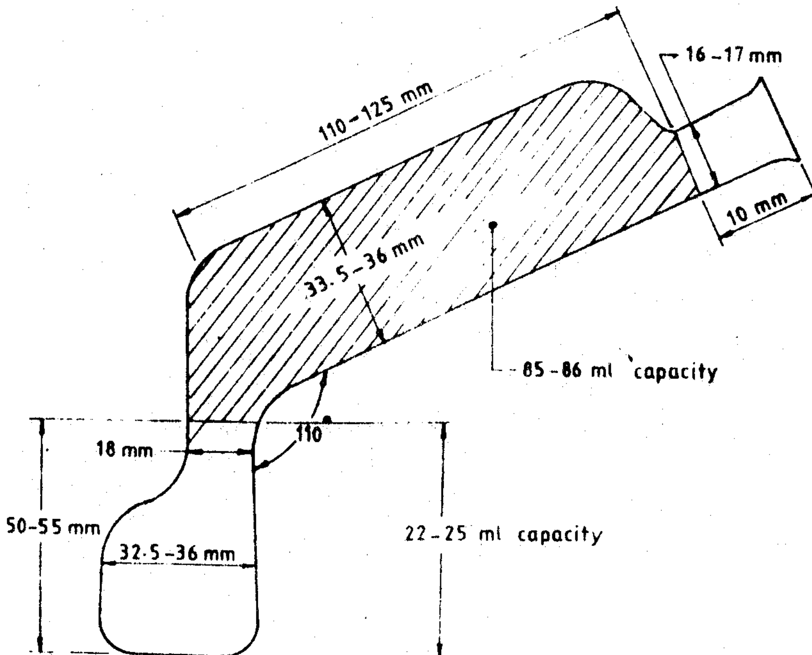

FIGURE 3 Mojonnier-type fat-extraction apparatus

A.1.2 A well ventilated electrically heated oven, set to operate at 98°C.

A.2 REAGENTS

A.2.1 *Aqueous ammonia*, concentrated, approximately 35 per cent *m/m*.

A.2.2 *Ethyl alcohol*, 95 to 96 per cent *V/V*.

A.2.3 *Diethyl ether*, peroxide free. (The ether may be maintained free from peroxide by storing over a moist zinc-copper couple).

A.2.4 *Light petroleum*, boiling range 40°C to 60°C, recently distilled.

A.2.5 *Mixed solvent*, prepared by mixing equal volumes of the diethyl ether and light petroleum.

A.3 PROCEDURE

A.3.1 Using the fat-extraction tube (Fig. 1, 2 or 3), weigh to the nearest milligram about 10 g to 11 g of the prepared sample into the extraction tube. Add 1 ml of aqueous ammonia and mix well. Add 10 ml of alcohol and again mix well. Complete extraction of the fat is dependent on satisfactory mixing at each stage.

Add 25 ml of diethyl ether, close the tube with the cork (or stopper), which is wetted with water before insertion, and shake vigorously for one minute. Remove the cork and, with 25 ml of light petroleum, wash the cork and neck of the tube so that the washings run into the tube. Replace the cork, again wetted with water, and shake vigorously for 30 s.

(It is essential that the cork (or stopper) be wetted with water before each insertion and washed with solvent during each removal. Also,before each removal, to avoid spurting of the solvent, a slightly reduced pressure should be induced in the tube by cooling. Rubber stoppers shall not be used.)

Allow the tube to stand until the ethereal layer is clear and completely separated from the aqueous layer, usually for not less than 30 min or centrifuge at about 1000 r.p.m. for 30 min. Remove the cork and insert the siphon (or wash-bottle) fitting so adjusted for length that the inlet is 2 mm to 3 mm above the interface between the ethereal and aqueous layers and transfer the ethereal layer to a suitable flask. Add 5 ml of mixed solvent to the extraction tube, using it to wash the siphon or wash-bottle fitting which is raised sufficiently to permit this but not removed from the inside of the tube. Lower the fittings and transfer the solvent to the flask without shaking. Repeat this operation with further 5 ml of mixed solvent. Wash the tip of the siphon fitting into the flask with mixed solvent.

Remove the siphon fitting and repeat the extraction of the milk residue, using 15 ml of ether and 15 ml of light petroleum, and repeat the subsequent operations, as before. Use the ether to wash the inner limb of the siphon (or wash-bottle) fitting during its removal from the tube. Finally repeat the extraction once more with 15 ml each of ether and petroleum.

Distil carefully the solvents from the flask and dry the residual fat in the oven at 98 °C to 100 °C for one hour taking precautions to remove all traces of volatile solvent. Cool the flask to room temperature in a desiccator charged with an efficient desiccant and weigh. Repeat this procedure for periods of half an hour until successive weighings do not show a loss in mass by more than one milligram.

Extract completely the fat from the flask by repeated washing with light petroleum, allowing any sediment to settle before each decantation. Dry the flask in the oven, cool and weigh as before. The differences in mass, before and after the petroleum extractions, subject, if necessary, to a correction for the blank described below, is the mass of fat contained in the mass of milk taken.

Make a blank determination using the specified quantities of reagents throughout, and distilled water in place of the milk, and deduct the value found, if any from the apparent mass of fat. A flask, similar to that used to contain the fat, shall receive the same heating and cooling treatments and shall be used as countermass.

A.3.2 Using the Mojonnier fat-extraction tube (Fig. 3)

Weigh to the nearest milligram about 10 g or 11 g of the prepared sample into the tube. Add one ml of aqueous ammonia and mix well in the lower bulb. Add 10 ml of the alcohol and mix by allowing the liquid to flow backwards and forwards between the two bulbs. (Avoid bringing the liquid too near the neck of the tube). Allow the tube to cool in cold, running water or by immersing in chilled water.

Add 25 ml of ether, close with a bark or glass stopper which is wetted with water before insertion, and shake vigorously for one minute.

(It is essential that the cork (or stopper) be wetted with water before each insertion and washed with solvent during each removal. Also, before each removal to avoid spurting of the solvent a slightly reduced pressure should be induced in the tube by cooling. Rubber stoppers shall not be used).

Open the tube and add 25 ml of light petroleum, close the tube, and shake vigorously for one minute. Allow the tube to stand on the flat bottom of the lower bulb until the ethereal layer is clear and completely separated from the aqueous layer, usually for not less than 30 min or centrifuge until clear. Examine the tube to see if the junction of the liquid is at the lower end of the narrow neck of the tube. If it is below this, it should be raised by the addition of a little distilled water run down the side of the tube.

Carefully decant as much as possible of the supernatant layer into a suitable flask by gradually bringing the cylindrical bulb of the tube into a horizontal position. When as much as possible has been poured off, wash the outside of the neck of the tube and the cork or stopper with mixed solvent, collecting the rinsings in the flask. With the Mojonnier tube in a vertical position, wash the inside of the neck with 4 ml to 5 ml of mixed solvent, and decant.

Repeat the extraction of the milk residue and the subsequent operations but using 15 ml of ether and 15 ml of petroleum.

Finally repeat the extraction and subsequent operations once more with 15 ml each of ether and petroleum.

Distil carefully the solvents from the flask and dry the residual fat in the oven at 98 °C to 100 °C for one hour taking precautions to remove all traces of volatile solvent. Cool the flask to room temperature in a desiccator charged with an efficient desiccant and weigh. Repeat this procedure for periods of half an hour until successive weighings do not show a loss in mass by more than one milligram.

Extract completely the fat from the flask by repeated washing with light petroleum, allowing any sediment to settle before each decantation, dry the flask in the oven, cool and weigh as before. The difference in mass before, and after the petroleum extractions, subject, if necessary, to a correction for the blank described below, is the mass of fat contained in the mass of milk taken.

Make a blank determination using the specified quantities of reagents throughout, and distilled water in place of milk, and deduct the value found, if any, from the apparent mass of fat. A flask similar to that used to contain the fat shall receive the same heating and cooling treatments and shall be used as a countermass.

APPENDIX B

DETERMINATION OF MILK SOLIDS OTHER THAN MILK FAT

For the purpose of this standard, the term 'total solids' is applied to the dry residue obtained when the milk is treated as described in the method below. The method is not applicable to milks having a titratable acidity exceeding 0.20 per cent *m/V* expressed as lactic acid.

B.1 APPARATUS

B.1.1 *A flat-bottomed, metal dish,* 70 mm to 80 mm in diameter and 10 mm to 25 mm deep, provided with an easily removable but closely fitting lid. Aluminium, nickel and stainless steel are suitable metals.

B.1.2 *A well ventilated, electrically heated Oven,* set to operate at 100 ± 1 °C.

B.2 PROCEDURE

Heat the uncovered empty dish and lid in the oven for at least 30 min. Place in a desiccator charged with an efficient desiccant, such as silica gel or phosphoric oxide, and allow to cool to room temperature (usually for 30 min to 45 min) and weigh accurately. Pipette 3 ml to 4 ml of the milk into the dish, cover the dish with the lid and weigh again. Place the dish uncovered, on a rapidly boiling water bath so that the bottom of the dish is in direct contact with steam or in close contact with a thin metal plate (e.g. aluminium, copper or stainless steel) fitted on top of the bath. To promote uniform drying, ensure that the base of the dish is horizontal.

After about 30 min, the film of milk solids should appear dry and cracked. Remove the dish from the bath, wipe the outside free of any moisture and transfer it to the oven. Place the lid by the dish. Use a shelf near the middle of the oven, keep the dish away from the walls and insulate the bottom of the dish from the surface of the shelf. The bulb of the thermometer registering the air temperature in the oven should be immediately above the shelf carrying the dish.

After 2.5 h, cover the dish with the lid, transfer it immediately to the desiccator, allow to cool as before and weigh. Repeat the heating for periods of 1 h, cooling and weighing, until the loss of the mass between successive weighings does not exceed 0.5 mg.

B.3 CALCULATION

Solids not fat = Total solids per cent by mass-fat per cent by mass (reading obtained from Appendix A).

$$\text{Total solids, per cent, by mass} = \frac{m_1}{m_2} \times 100$$

where

m_1 = mass, in g, of the residue after drying; and

m_2 = mass, in g, of the prepared sample taken for the test.

APPENDIX C

DETERMINATION OF MILK SOLIDS OTHER THAN MILK FAT IN FLAVOURED MILK

Determine the total solids other than milk fat as given in Appendix B. The total solids determined by this method includes any sucrose added to the flavoured milk. Determine the amount of sucrose present by the methods given below and subtract this value from the value obtained for total solids other than milk fat by the method given in Appendix B.

C.1 DETERMINATION OF REDUCING SUGARS

C.1.1 Reagents

C.1.1.1 *Stock solution of dextrose*, weigh to the nearest milligram about 10 g of anhydrous dextrose into a 1-litre graduated flask and dissolve it in water. Add to this solution 2.5 g of benzoic acid, shake to dissolve the benzoic acid and make up the volume to the mark with water. (This solution shall not be used after 48 h).

C.1.1.2 *Standard dextrose solution*, dilute a known aliquot of the stock solution of dextrose (see C.1.1.1) with water containing 0.25 per cent *(m/V)* of benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it will be required to reduce all the copper in the Fehling's solution taken for titration. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml (see Note), prepare this solution fresh every day.

NOTE – When 10 ml (see C.1.3.1.a) of Fehling's solution are taken for titration, a standard dextrose solution containing 0.11 to 0.30 per cent *(m/V)* of anhydrous dextrose is convenient for use.

C.1.1.3 *Methylene blue indicator solution*, dissolve 0.2 g of methylene blue in water and dilute to 100 ml.

C.1.1.4 *Petroleum ether*, re-distilled below 60°C.

C.1.1.5 *Fehling's solution (Soxhlet modification)*, prepared by mixing immediately before use, equal volumes of Solution A and Solution B.

a) Solution A

Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water, add 0.5 ml of concentrated sulphuric acid of relative density 1.84 and dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

b) Solution B

Dissolve 173 g of Rochelle salt (potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$)) and 50 g of sodium hydroxide, analytical reagent in water, dilute to 500 ml in a graduated flask and allow the solution to stand for two days. Filter the solution through prepared asbestos.

c) Standardization of Fehling's solution

Pour standard dextrose solution (see C.1.1.2) into a 50-ml burette (see Note 3 under C.1.2.3). Find the titre (that is, the volume of standard dextrose solution required to reduce all the copper in 10 ml of Fehling's solution) corresponding to the concentration of standard dextrose solution from Table 2. (If, for example, the standard dextrose solution contains 167.0 mg of anhydrous dextrose per 100 ml, the corresponding titre would be 30 ml). Pipette 10 ml (see C.1.3.1.a) of Fehling's solution into a 300-ml conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper, so that not more than one millilitre will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. (The titration should be completed within one minute, so that the contents of the flask boil altogether for 3 min without interruption (see Note 2 under C.1.2.3). Note the titre (that is the total volume in millilitres of standard dextrose solution used for the reduction of all the copper in 10 ml of Fehling's solution.) Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in one millilitre of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 2 and determine correction, if

any, to be applied to the dextrose factor derived from Table 2.

EXAMPLE

Concentration in mg/100 ml of anhydrous dextrose of standard dextrose solution	=	167.0
Titre in millilitre obtained by direct titration	=	30.1
Dextrose factor for 30.1 ml of standard dextrose solution	=	Titre in millilitre x number of milligrams of anhydrous dextrose in one millilitre of standard dextrose solution
	=	30.1 x 1.670
	=	50.2670
Dextrose factor for 30.1 ml of standard dextrose solution from Table 2 (calculated by interpolation)	=	50.11
Correction to be applied to the dextrose factor derived from Table 2	=	50.2670 - 50.11
	=	+0.1570

C.1.2 Procedure

C.1.2.1 *Preparation of solution*

Weigh to the nearest milligram about 3 g to 4 g of the prepared sample (see C.1.1.1) in Soxhlet extraction thimble and extract the fat in a Soxhlet apparatus using petroleum ether. Take out carefully the thimble along with the fat-free material from the Soxhlet apparatus and dry the same to be free from the petroleum ether. Dissolve carefully the entire fat-free sample in a small quantity of water in a beaker. If necessary, add water to the thimble and dissolve the adhering material. Collect the washings into the beaker. Warm to a temperature of 50 °C to 60 °C. Cool it. Filter through a Whatman filter paper No. 40 or its equivalent, collecting the filtrate in a 100-ml graduated flask. Wash the filter paper and the insoluble starch residue, if any, on the filter paper carefully. Collect the washings in the graduated flask. Make up to the mark with water.

TABLE 2 - Dextrose factors for 10 ml of Fehling's solution (see C.1.1.5.c)

Titre ml (1)	Dextrose factor* (2)	Dextrose content per 100 ml of solution, mg (3)
15	49.1	327
16	49.2	307
17	49.3	289
18	49.3	274
19	49.4	260
20	49.5	247.4
21	49.5	235.8
22	49.6	225.5
23	49.7	216.1
24	49.8	207.4
25	49.8	199.3
26	49.9	191.8
27	49.9	184.9
28	50.0	178.5
29	50.0	172.5
30	50.1	167.0
31	50.2	161.8
32	50.2	156.9
33	50.3	152.4
34	50.3	148.0
35	50.4	143.9
36	50.4	140.0
37	50.5	136.4
38	50.5	132.9
39	50.6	129.6
40	50.6	126.5
41	50.7	123.6
42	50.7	120.8
43	50.8	118.1
44	50.8	115.5
45	50.9	113.0
46	50.9	110.6
47	51.0	108.4
48	51.0	106.2
49	51.0	104.1
50	51.1	102.2

* *Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling's solution.*

C.1.2.2 *Incremental method of titration*

Pour the prepared solution (see C.1.2.1) into a 50-ml burette (see Note 3 below C.1.2.3). Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette 15 ml of the prepared solution. Without further dilution, heat the contents of the flask over a wire gauze, and boil. (After the liquid has been boiling for about 15 sec, it will be possible to judge if almost all the copper is reduced, by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide.) When it is judged that nearly all the copper is reduced, add one millilitre of methylene blue indicator solution (see Note 1), Continue boiling the contents of the flask for one to two minutes from the commencement of ebullition, and then add the prepared solution in small quantities (one millilitre or less at a time), allowing the liquid to boil for about 10 sec between successive additions, till the blue colour of the indicator just disappears (see Note 2 below C.1.2.3). In case there still appears to be much unreduced copper after the mixture of Fehling's solution with 15 ml of the prepared solution has been boiling for 15 sec, add the prepared solution from the burette, in larger increments (more than one millilitre at a time according to judgement), and allow the mixture to boil for 15 sec after each addition. Repeat the addition of the prepared solution at intervals of 15 sec, until it is considered unsafe to add a large increment of the prepared solution. At this stage, continue the boiling for an additional one to two minutes, and one millilitre of methylene blue indicator solution and complete the titration by adding the prepared solution in small quantities (less than one millilitre at a time) (see also Note 2).

NOTES

1) It is advisable not to add the indicator until the end point has been nearly reached because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

2) When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration. For the utmost degree of accuracy of which the method is capable, a second titration should be carried out by the standard method of titration (see C.1.2.3).

C.1.2.3 *Standard method of titration*

Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette almost the whole of the prepared solution required to effect reduction of all the copper (determined under C.1.2.2) so that, if possible, not more than one millilitre will be required later to complete the titration. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add the prepared solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears (see Note 1). (The titration should be completed within one minute so that the contents of the flask boil altogether for 3 min without interruption (see Note 2).

In case of doubt, the flame may be removed from the wire gauze for one or two seconds and the flask held against a sheet of white paper. (A holder of paper, suitably fixed around the neck of the flask, is very convenient for this purpose as it can be left round the neck of the flask, without risk of overbalancing it). The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is inadvisable to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access into the flask, but there is no danger of this as long as a continuous stream of steam is issuing from the mouth of the flask.

NOTES

1) The indicator is so sensitive that it is possible to determine the end point within one drop of the prepared solution in many cases. The complete decolouration of the methylene blue is usually indicated by the whole reaction liquid, in which the cuprous oxide is continuously churned up becoming red or orange in colour.

2) It should be observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

3) In adding sugar solution to the reaction mixture, the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of the steam while adding sugar solution. Burettes with glass taps are unsuitable for this work, as the taps become heated by the steam and are liable to jam.

C.1.3 Calculation

C.1.3.1 Refer to Table 3 for the dextrose factor corresponding to the titre (determine as given under C.1.2.3) and apply the correction previously determined under C.1.1.5.c). Calculate the dextrose content of the prepared solution (see C.1.2.1) as follows:

$$\text{Milligrams of anhydrous dextrose present in one millilitre of the prepared solution} = w = \frac{\text{Dextrose factor}}{\text{Titre}}$$

C.1.3.1.a Instead of using 10 ml of Fehling's solution, a 25 ml portion may also be substituted throughout the procedure (including 'standardization of Fehling's solution' under C.1.1.5.c). In this case, the standard dextrose solution, used in standardizing the Fehling's solution and the prepared solution of the material (see C.1.2.1) shall contain 0.25 per cent to 0.75 per cent (m/V) of anhydrous dextrose, and Table 3 shall be used for all calculations.

C.1.3.2 Reducing sugars, per cent, by mass $= \frac{m_1}{m_2} \times 100$

where

m_1 = milligrams of anhydrous dextrose in 1 ml solution of the material (see C.1.3.1); and

m_2 = mass, in g, of the prepared sample used for making 100 ml of solution (see C.1.2.1).

TABLE 3 - Dextrose factors for 25 ml of Fehling's solution (see C.1.3.1 and C.1.3.1.a)

Titre ml (1)	Dextrose factor* (2)	Dextrose content per 100 ml of solution, mg (3)
15	120.2	810
16	120.2	751
17	120.2	707
18	120.2	668
19	120.3	638
20	120.3	601.5
21	120.3	572.9
22	120.3	547.3
23	120.4	523.6
24	120.5	501.9
25	120.5	482.0
26	120.6	463.7
27	120.6	446.8
28	120.7	431.1
29	120.7	416.4
30	120.8	402.7
31	120.8	389.7
32	120.8	377.6
33	120.9	366.3
34	120.9	355.6
35	121.0	345.6
36	121.0	336.3
37	121.1	327.4
38	121.2	318.8
39	121.2	310.7
40	121.2	303.1
41	121.3	295.9
42	121.4	289.0
43	121.4	282.4
44	121.5	276.1
45	121.5	270.1
46	121.6	264.3
47	121.6	258.8
48	121.7	253.5
49	121.7	248.4
50	121.8	243.6

* *Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling's solution.*

NOTE - Tables 2 and 3 show, for the standard method of titration, the values corresponding to integral millilitres of the sugar solutions, intermediate values being obtained by interpolation.

C.2 DETERMINATION OF SUCROSE

C.2.1 Reagents

C.2.1.1 *Concentrated hydrochloric acid,* relative density 1.16 of analytical reagent grade.

C.2.1.2 *Fehling's solution (Soxhlet modification),* prepared by mixing immediately before use, equal volumes of Solution A and Solution B.

a) Solution A

Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water, add 0.5 ml of concentrated sulphuric acid of relative density 1.84, and dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

b) Solution B

Dissolve 173 g of Rochelle salt (potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$)) and 50 g of sodium hydroxide, analytical reagent in water, dilute to 500 ml in a graduated flask and allow the solution to stand for two days. Filter this solution through prepared asbestos.

C.2.2 Procedure

Take 10 ml of the prepared solution (see C.1.2.1) in a conical flask and add 1.5 ml of the concentrated hydrochloric acid and about 10 ml of water. Heat the flask at 60 °C to 70 °C for 10 min in a water-bath. Cool immediately and neutralize with 30 per cent sodium hydroxide (m/V) and transfer quantitatively the neutralized inverted solution to a graduated flask and make up the volume to 100 ml.

Determine the reducing sugars in the inverted solution as described in Appendix C.

C.2.3 Calculation

C.2.3.1 Sucrose, per cent by mass = $(Q - R)\ 0.95$

where

Q = total sugars (after inverting); and

R = reducing sugars (before inverting) (see C.1.3).

APPENDIX D

DETERMINATION OF CREAMING INDEX

D.1 APPARATUS

Glass centrifuge tubes, *pipette*, and apparatus for the determination of fat.

D.2 PROCEDURE

Place 50 ml of milk at room temperature in each of the 3 tubes. Centrifuge for 15 min at 1 000 r.p.m.

Using the pipette, take 5 ml from the upper part of each of the 3 tubes, carefully removing the cream that adheres to the tube walls. The upper tenth of the volume of milk that has been centrifuged is thus transferred to the vessel (Sample A). Empty the 3 tubes into a single container (Sample B).

Measure the fat content in Samples A and B by the Rose-Gotlieb method, bearing in mind the usual precaution with homogenized milk of centrifuging several times (heating after each centrifuging in a 67 °C water-bath and taking a reading) until the results are identical. Generally three centrifuging cycles will be sufficient.

D.3 CALCULATION

$$\text{Creaming index} = \frac{A - B}{B} \times 100$$

where

A = percentage fat content of Sample A; and

B = percentage fat content of Sample B.

The lower the creaming index, the better the homogenization.

APPENDIX E

PHOSPHATASE TEST

E.1 APPARATUS

E.1.1 *Lovibond all purposes comparator*, with stand and standard discs.

E.1.2 *Fused glass cells*, 25 mm.

E.1.3 *Test tubes*, with ring at 10 ml fitted with rubber stoppers.

E.2 REAGENTS

E.2.1 *Buffer solution,* 3.5 g of sodium carbonate analytical reagent grade and 1.5 g of sodium bicarbonate analytical reagent grade dissolved in one litre of water.

E.2.2 *Substrate,* disodium p-nitrophenylphosphate not less than 95 per cent pure.

E.2.3 *Buffer substrate,* transfer 0.15 g of the substrate into a 100-ml measuring cylinder or stoppered graduated flask and make up to the mark with the buffer solution. The solution should not be stored for long periods but may normally be kept in refrigerator for up to one week. The solution is practically colourless; when viewed through a 25-mm cell in the comparator, it should give a reading of less than 10 on the disc.

E.3 PROCEDURE

Fill 10 ml (5 ml may be used) quantities of the buffer substrate solution into test tubes marked at 10 ml and bring to 37 °C to 38 °C in a water bath. Add 2 ml (1 ml if 5 ml of buffer substrate are used) of the milk to be tested, close the tubes with rubber stoppers and invert to mix. Prepare in the same way a control tube using a portion of the milk under test which has been boiled and cooled. Incubate all the tubes at 37 °C to 38 °C. Read the yellow colour after 30 min, return to the bath, and take a second reading after incubation for a further 90 min. The yellow colour is read in a Lovibond all purposes comparator on a resazurin stand, fitted with the disc calibrated in microgramme p-nitrophenol. The blank is placed on the left of the stand and the sample on the right. Readings are taken by looking down the two apertures with the comparator facing a good source of daylight; the disc is revolved until the colours are matched. Readings falling between two standards are recorded to the nearest reading.

E.4 INTERPRETATION

Milk showing a disc reading of 0 after 30 min and 0 to 10 after 2 h shall be deemed to have satisfied the test.

APPENDIX F

TURBIDITY TEST

F.1 REAGENTS

F.1.1 *Ammonium sulphate,* A.R.

F.1.2 *Whatman filter paper No. 12 or equivalent,* 125 mm disc.

F.2 PROCEDURE

Mix the sample of milk thoroughly and transfer 20-ml to a 50-ml conical flask containing 4 g ammonium sulphate. Shake the contents of the flask for at least 3 min to ensure that all the ammonium sulphate is dissolved. Allow to stand for not less than 5 min and then filter into a test tube using a Whatman No. 12 filter paper. When not less than 5 ml of clear filtrate is obtained, place the tube in a beaker of water which is kept boiling and allow to remain for 5 min. Transfer the test tube to a beaker of cold water and when cold, examine the contents of the tube for turbidity. This is facilitated by moving the tube in front of an electric light shaded from the eyes of the observer and comparing the tube with a control tube. A control tube is prepared by heating 20 ml of milk in a boiling water bath for 20 min. The cooled milk is then tested in the same way as the sample under examination.

F.3 INTERPRETATION

Milk which shows no sign of turbidity shall be deemed to have satisfied the test.

APPENDIX G

METHYLENE BLUE REDUCTION TEST

G.1 APPARATUS

G.1.1 *Test tubes,* 150 mm^{2}x 15 mm with graduation at 10 ml.

G.1.2 *Rubber stoppers,* for test tubes, sterilized.

G.1.3 *Pipettes,* 1.0 ml straight-sided blow-out pipettes plugged with cotton wool at the upper end and sterilized.

G.1.4 *Dipper or pipette,* to deliver 10 ml.

G.1.5 *Water bath,* fitted with a lid and thermostatically controlled at 37.5 ± 0.05^{o}C.

G.1.6 *Sheet metal or wire racks,* corrosion resistant, for holding tubes vertical in single or double rows.

G.2 REAGENTS

G.2.1 *Methylene blue solution,* prepare a solution observing aseptic precautions by adding one tablet (containing the equivalent of 15.8 mg of pure methylene blue and conforming to B.P. Standards) to about 200 ml of cold sterile glass-distilled water in a sterile flask, graduated at 800 ml, shake until the tablet is completely dissolved,

and make up the solution to 800 ml with cold sterile, glass distilled water. Store the resultant solution in a sterile, stoppered bottle in a cool, dark place. Do not use if:

a) it has been exposed to sunlight;

b) a period of 2 months has elapsed since the date of preparation;

c) contamination is suspected.

Pour off the amount of methylene blue required for a day's work with aseptic precautions, from the stock bottle, into a sterile glass container. Do not introduce the pipette to be used later for transferring the methylene blue solution to the tubes of milk into the stock bottle.

G.3 TREATMENT OF SAMPLE

G.3.1 Samples shall be collected off the production line or factory cold room, and left overnight (approximately 18 h) in a refrigerator where the temperature shall not exceed 10 °C.

G.3.2 On the following morning keep the samples at atmospheric shade temperature (approximately 27 °C to 30 °C) for six hours.

G.4 PROCEDURE

Thoroughly mix the sample by inverting and shaking the sample bottle. Remove the stopper or cap of the bottle with aseptic precautions. Flame the mouth of the bottle and pour the milk into a sterile test tube, up to the 10 ml mark, leaving one side of interior of the tube unwetted with milk. Add 1 ml of methylene blue solution, taking care that the pipette does not make contact with the milk, or with the wetted side of the interior of the tube. After a laps of 3 s, blow out the solution remaining in the tip of the pipette. Close the tube aseptically with a sterile stopper. Mix the contents by slowly inverting the tube twice, so that the whole column of contained air rises above the level of the milk, and place within 5 min in a water bath. Keep the water in the bath above the level of the milk in the test tubes, and maintain the temperature at 37.5 ± 0.5 °C. Carry out the incubation, with the lid closed.

To indicate when decolourization is commencing, and when it is complete, use two control tubes for comparison with each batch of experimental tubes. Prepare one control tube immersing in boiling water, for not less than 3 min in a properly stoppered test tube containing 1 ml of tap water and 10 ml of a mixture of milk having a fat content and colour similar to that of the milk being tested.

Inspect the tubes under test and compare with the control tubes at intervals of 30 min. At these inspections:

a) if the tube containing the milk under test has become decolourised, remove it from the water bath.

b) if decolourisation has begun, keep the tube without inversion in the water bath until decolourisation is complete.

c) if decolourisation has not begun, invert the tube once and replace.

The milk shall be regarded as decolourised when the whole column of milk is completely decolourised or is colourised up to within 5 mm of the surface. A trace of colour at the bottom of the tube may be ignored, provided that it does not extend upwards for more than 5 mm.

Record the time taken for complete decolourisation of the sample.

G.5 INTERPRETATION

The test shall be deemed to be satisfied by milk which fails to decolourise methylene blue in two hours.

G.6 NOTES

1) Do not perform this test in bright sunshine.

2) Clean out the bath, scrub the racks and add fresh water at least once a week.

APPENDIX H

MICROBIOLOGICAL EXAMINATION

H.1 AEROBIC PLATE COUNT

Proceed as described in **SLS 516**:Part 1, Clause 7, using Plate Count Agar or Yeast Extract Milk Agar, incubating at 30 ± 1 °C for 72 h.

H.2 ENUMERATION OF COLIFORMS

Proceed as described in **SLS 516**:Part 3, Clause 7, incubating at 30 ± 1 °C for 72 h.

APPENDIX J

SAMPLING EQUIPMENT

J.1 Plungers and dippers shall be of stainless steel or other suitable material of adequate strength, and shall be of sufficiently robust construction to prevent distortion in use. They shall, however, be sufficiently light for the operator to be able to move them rapidly through the liquid. If solder is used in the manufacture of the apparatus, it shall be capable of withstanding sterilization at 180 °C. All surfaces shall be smooth and free from crevices and all corners shall be rounded.

J.2 Plungers or agitators

In general, plungers or agitators for mixing liquids in bulk shall be of sufficient area to produce adequate disturbance of the product.

In view of the differing shapes and sizes of containers, no specific design of plunger can be recommended for all purposes.

A form of plunger recommended as being suitable for the mixing of liquids in buckets or in cans (see Fig. 4) has the following approximate dimensions:

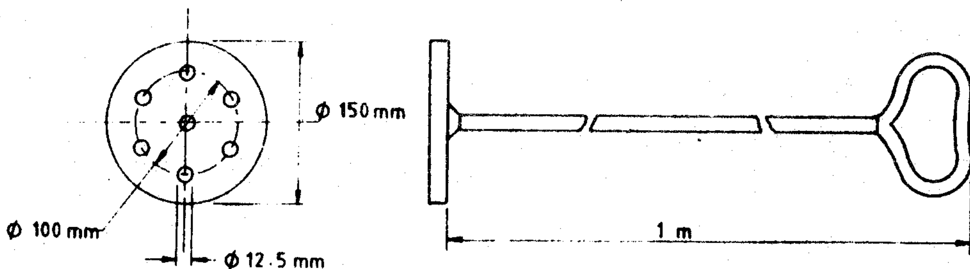

FIGURE 4 Recommended plunger for cans and buckets (see J.2)

a disc 150 mm in diameter, perforated with six holes each 12.5 mm in diameter on a pitch circle of 100 mm diameter, the disc being fixed centrally to a metal rod, the other end of which forms a loop handle. The length of the rod, including the handle, should be approximately 1 m.

A suitable plunger for use with road and rail tanks (see Fig. 5) has the following approximate dimensions:

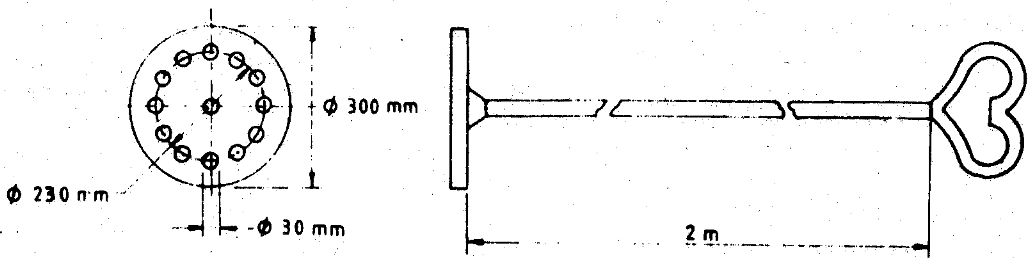

FIGURE 5 Suitable plunger for road and rail tanks (see J.2)

a rod not less than 2 m in length is fitted with a disc 300 mm in diameter, perforated with twelve holes each 30 mm in diameter on a pitch circle of 230 mm diameter.

For mixing the contents of large vessels, mechanical agitation by clean* compressed air or suitable stirring is advisable.

J.3 DIPPERS

A dipper of suitable size and shape for collecting the sample is illustrated in Fig. 6. The dipper shall be fitted with a solid handle at least 150 mm in length. The capacity of the dipper shall be not less than 85 ml. It is an advantage for the handle to be bent over. The tapered form of the cup permits nesting of the dippers.

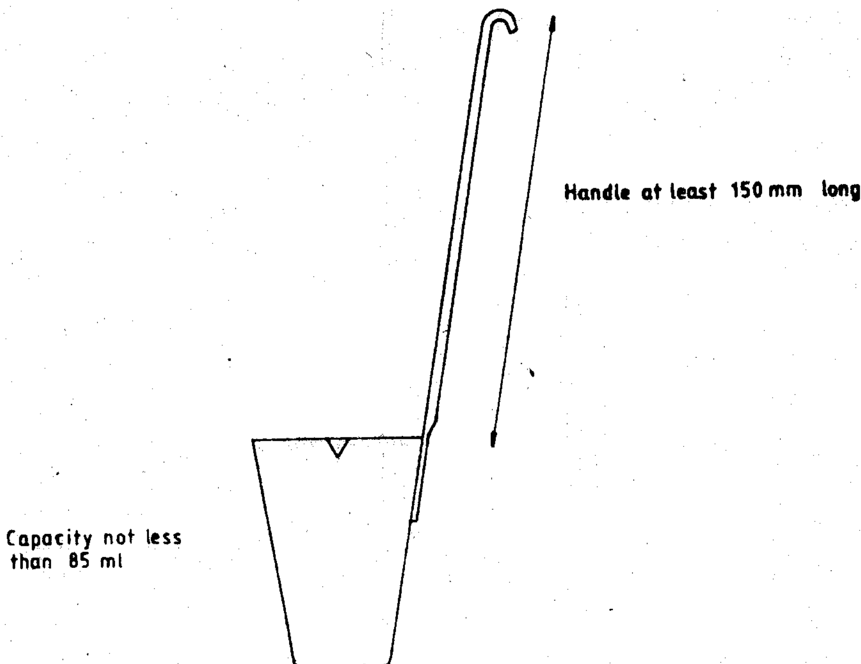

FIGURE 6 Suitable dipper for liquids (see J.3)

**Wherever 'clean compressed' is required by this standard, it is necessary to use compressed air from which all contaminants (including oil, water and dust) have been excluded.*

Alternatively, a dipper may be used which is of similar capacity, but which has parallel sides graduated into 5 equal sections for assistance in sampling proportionately consignments held in more than one container.

J.4 PLUNGER DIPPERS

If desired, the plunger and dipper may be combined.

J.5 SAMPLING FOR MICROBIOLOGICAL EXAMINATION

When the sample is required for microbiological examination, the sampling equipment shall be sterilized as described in 8.4.2.2.

AMD 77

AMENDMENT NO. 1 APPROVED ON 1986-04-30.

SLS 181:1983 SPECIFICATION FOR RAW AND PROCESSED MILK (FIRST REVISION)

Page 4

Clause 3.8

Delete the existing clause and substitute the following:

"**flavoured milk** : The product prepared from liquid milk or milk powder or condensed milk, sugar, chocolate or cocoa or coffee and other permitted flavouring and with or without permitted food colour, stabilizer and buffering agent."

CORRIGENDUM TO SLS 181 : 1983
SRI LANKA STANDARD SPECIFICATION FOR RAW AND PROCESSED MILK
(FIRST REVISION)

Appendix D
Determination of creaming Index

Replace 2nd paragraph of **D.2** to read as:

" Using the separate pipette, take 5 ml from the upper part of the three tubes, carefully taking the cream that adheres to walls of the tube and transfer into a container (sample A). Then empty the three tubes into a separate container (sample B).

....

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