SRI LANKA STANDARD 735 : PART 6 : 1989

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METHODS OF TEST FOR

MILK AND MILK PRODUCTS

PART 6 - DETERMINATION OF SUGARS

SRI LANKA STANDARDS INSTITUTION

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Gr. 6

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

SRI LANKA STANDARD
METHODS OF TEST FOR MILK AND MILK PRODUCTS
PART 6 : DETERMINATION OF SUGARS

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1989-08-22, after the draft, finalized by the Drafting Committee on Milk and Milk Products, had been approved by the Agricultural and Food Products Divisional Committee.

In order to accommodate the large number of test methods within the scope of one standard, this standard is published in several parts.

This standard forms Part 6 of Sri Lanka Standard methods of test for milk and milk products.

The values used in this standard are given in SI units.

In reporting the result of a test or an analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with CS 102.

1 SCOPE

This part of the standard prescribes the methods of determination of sugar content of ice cream, milk ice, flavoured milk and sweetened condensed milk.

2 REFERENCES

ISO 707 Milk products sampling.
CS 102 Presentation of numerical values.

3 SAMPLING

Test samples for the use in the tests specified in this part shall be obtained in accordance with ISO 707.

4 DETERMINATION OF SUGAR CONTENT OF ICE CREAM, MILK ICE AND FLAVOURED MILK

4.1 Preparation of the sample

Samples should be analysed immediately after receipt. If this is not possible samples should be placed in a screw capped jar and stored at a temperature below 0 °C. In the case of flavoured milk, samples shall be kept at 6 °C to 10 °C. Samples shall not be tested after 24 hours.

4.1.1 *Plain ice cream and milk ice*

Allow the sample to soften at room temperature. Do not heat the sample. Mix thoroughly by stirring with a spoon, egg beater or by pouring back and forth between beakers.

4.1.2 *Ice cream with insoluble particles*

Allow the sample to soften at room temperature. Blend 100 g to 200 g of the sample using a mixer (for about 10 minutes) until the insoluble particles are finely divided.

4.1.3 *Flavoured milk*

Allow the sample to attain room temperature. Mix thoroughly by continuous slow inversions of the sample bottle or by slowly pouring back and forth between beakers.

4.2 Reagents

4.2.1 *Stock solution of dextrose*, weigh, to the nearest milligram, about 2 g of anhydrous dextrose and dissolve it in 200 ml of water. Prepare this solution fresh every day.

4.2.2 *Standard dextrose solution*, dilute a known aliquot of the stock solution of dextrose (4.2.1) with water 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 (4.2.8). 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 of Fehling's solution is taken for titration, a standard dextrose solution containing 1.1 g/l to 3.0 g/l of anhydrous dextrose is convenient for use.

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

4.2.4 *Zinc acetate solution,* dissolve 21.9 g of zinc acetate, $Zn(C_2H_3O_2)_2.2H_2O$, in water and dilute to 100 ml.

4.2.5 *Potassium ferrocyanide solution,* dissolve 10.6 g of potassium ferrocyanide, $K_4Fe(CN)_6.3H_2O$, in water and dilute to 100 ml.

4.2.6 *Hydrochloric acid,* 6.34 mol/l solution.

4.2.7 *Sodium hydroxide,* 1 mol/l solution.

4.2.8 *Fehling's solution (Soxhlet modification),* Pipette 5 ml of Solution A (4.2.8.1) into 5 ml of Solution B (4.2.8.2) immediately before use and mix.

4.2.8.1 Solution A - Dissolve 34.639 g of copper sulfate, ($CuSO_4.5H_2O$), in water. Add 0.5 ml of concentrated sulfuric acid (rel. den. = 1.84) and dilute to 500 ml in a volumetric flask. Filter if necessary.

4.2.8.2 Solution B - Dissolve 173 g of 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 volumetric flask. Filter if necessary.

4.2.8.3 Standardization of Fehling's solution

Take standard dextrose solution (4.2.2) into a 50-ml burette. (preferably fitted with an outlet tube bent twice at right angles) 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 1 (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 of Fehling's solution (4.2.8) into a 300-ml conical flask. Add almost the whole of the standard dextrose solution (4.2.2) required to effect reduction of all the copper from the burette, 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 for 2 minutes. At the end of 2 minutes of boiling, add few drops of methylene blue indicator solution (4.2.3). While the contents of the flask continue to boil, 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 minutes without interruption.

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 1 and determine correction, if any, to be applied to the dextrose factor derived from Table 1.

EXAMPLE:

Concentration in mg/100 ml of anhydrous dextrose of standard dextrose solution	=	167.0
Titre in millilitres obtained by direct titration	=	30.1
Dextrose factor for 30.1 ml of standard dextrose solution	=	Titre in millilitres 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 1 (calculated by interpolation)	=	50.11
Correction to be applied to the dextrose factor derived from Table 1	=	50.2670 -50.11
	=	+ 0.1570

TABLE 1 - Dextrose factors for 10 ml of Fehling's solution

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.*

4.3 Procedure

4.3.1 *Preparation of the test sample for titration*

4.3.1.1 For the determination of reducing sugars

Weigh, to the nearest milligram, about 10 g of the sample (30 g for flavoured milk) prepared as in 4.1 in a beaker. Trnasfer to a 250-ml volumetric flask washing with successive quantities of distilled water at 60 °C, until the total volume is about 200 ml. Cool to room temperature and add 5 ml each of zinc acetate (4.2.4) and potassium ferrocyanide (4.2.5). Make up to 250 ml. Mix and allow to settle. Filter through No. 1 Whatman filter paper, rejecting the first few millilitres.

4.3.1.2 For the determination of reducing sugars after inversion

Transfer 50 ml of the filterate obtained in 4.3.1.1 into a beaker. Add 10 ml of hydrochloric acid (4.2.6) and bring to boil. Boil for exactly 30 seconds and cool rapidly. Make just neutral to litmus with sodium hydroxide (4.2.7). Transfer quantitatively to 200 ml volumetric flask and dilute upto the mark.

4.3.2 *Incremental method of titration*

Transfer the solution prepared as in 4.3.1 to a 50 ml burette (preferably fitted with an outlet tube bent twice at right angles).

Pipette 10 ml of Fehlings solution (4.2.8) in to a 250-ml Erlenmeyer flask. Add 15 ml of the prepared solution (4.3.1) from the burette. Heat the contents of the flask over a wire gauze. Gently boil for 2 minutes. At the end of 2 minutes add 2 to 3 drops of methylene blue indicator solution (4.2.3) without removing the flask from the flame and complete the titration within 3 minutes, by adding small increments of the prepared solution. The end point is indicated by the decolourization of the indicator. Note the volume of prepared solution required to reduce 10 ml of Fehling's solution.

A second titration shall be carried out by standard method of titration.

4.3.3 *Standard method of titration*

Pipette 10 ml of Fehling's solution into a 300-ml conical flask. Add almost the whole of the volume determined under 4.3.2 from the burette so that, not more than one millilitre will be required to complete the titration. Gently boil the contents of the flask for 2 minutes. Add few drops of methylene blue indicator solution. While the contents of the flask contirue to boil, add the prepared solution (one or two drops at a time) from the burette and complete the titration as in 4.3.2 within one minute.

4.4 Calculation

4.4.1 Refer to Table 1 for the dextrose factor corresponding to the titre determined under 4.3.3 and apply the correction previously determined under 4.2.8.3. Calculate the dextrose content of the prepared solution as follows:

$$\text{Milligrams of anhydrous dextrose present in one millilitre of the prepared solution} = m_1 = \frac{\text{Dextrose factor}}{\text{Titre}}$$

4.4.2 Reducing sugars, expressed as dextrose, per cent by mass $= R = \frac{m_1}{m_2} \times 100$

where,

m_1 is the milligrams of anhydrous dextrose in 1 ml solution of the sample (see 4.4.1); and

m_2 is the mass, in grams, of the prepared sample used for making 100 ml of solution (see 4.3.1.1).

4.4.3 Sucrose, per cent by mass = (T - R) 0.95.

where,

T is the reducing sugars after inversion (4.3.1.2), expressed as dextrose, per cent by mass.

5 DETERMINATION OF SUCROSE CONTENT OF SWEETENED CONDENSED MILK

5.1 Preparation of the test sample

Warm the container in a waterbath at 40 °C for 30 minutes. Open the container and transfer all material adhering to the lid into the container. Mix thoroughly with a spoon or spatula so that the upper and lower layers are mixed well. Transfer the homogenous sample to a jar. Allow to cool.

5.2 Reagents

5.2.1 *Zinc acetate solution*, prepared as in 4.2.4.

5.2.2 *Potassium ferrocyanide solution*, prepared as in 4.2.5.

5.2.3 *Hydrochloric acid*, 6.34 mol/l solution.

5.2.4 *Ammonium hydroxide*, 10 ml of concentrated ammonium hydroxide (rel. den. = 0.880) diluted to 100 ml.

5.2.5 *Dilute acetic acid*, approximately equivalent to the concentration of ammonium hydroxide solution (5.2.4).

5.3 Apparatus

5.3.1 *Polarimeter tubes*, exactly 2-dm in length.

5.3.2 *Polarimeter*, with an accuracy of 0.05 angular degrees.

5.4 Procedure

5.4.1 *Preparation of the sample for direct polarisation*

Weigh, to the nearest milligram, about 40 g of the sample prepared as in 5.1 in a beaker. Add 50 ml of distilled water at 80 °C to 90 °C. Mix, transfer to a 200-ml volumetric flask washing with successive quantities of distilled water at 60 °C until the total volume is between 120 ml to 150 ml. Mix, cool to room temperature and add 5 ml of ammonia solution (5.2.4). Mix again and allow to stand for 15 minutes. Add a sufficient quantity of acetic acid solution (5.2.5) to neutralize the ammonia added and mix again (the exact equivalent is determined previously by titration). Add with gentle mixing 12.5 ml each of zinc acetate (5.2.1) and potassium ferrocyanide solution (5.2.2). Cool to room temperature and make up to 200 ml mark with distilled water. (see Note).

Close the flask with a dry stopper and mix thoroughly by shaking. Allow to stand for few minutes and filter through a dry filter paper rejecting the first 25 ml of the filtrate.

NOTE

All additions of water or reagents up to this stage shall be made in such a manner to avoid formation of air bubbles, and with the same object in view, all mixings shall be made by rotation of the flask rather than by shaking. If bubbles appear before completion of dilution to 200 ml, remove any traces of occluded air by careful temporary attachment of the flask to a vacuum pump and by rotating the flask.

5.4.2 Determine the optical rotation of the filterate obtained at 20 ± 2 °C.

5.4.3 *Preparation of the sample for invert polarization*

Pipette 40 ml of the filtrate, obtained in 5.4.1 into a 50-ml volumetric flask and add 6 ml of hydrochloric acid (5.2.3). Immerse the entire flask in a water bath maintained at 60 °C for 12 minutes with rotary movement during the first 3 minutes, so that the contents of the flask attain the temperature of the bath during this time. Cool to room temperature and dilute to 50 ml with distilled water. Mix and allow to stand for one hour.

5.4.4 Determine the optical rotation of the inverted solution at 20 ± 2 °C.

5.5 Calculation

$$\text{Sucrose content, per cent by mass} = \frac{A - 1.0253}{Q} \times \frac{V - \Delta V}{V} \times \frac{V}{l \times m}$$

$$\Delta V = \frac{m}{100} (1.08F + 1.55P)$$

where,

ΔV is the correction, in millilitres, for the volume of the precipitate formed during clarification;
A is the polarimeter reading obtained in 5.4.2;
B is the polarimeter reading obtained in 5.4.4 (after inversion);
l is the length, in decimetres, of the polarimeter tube;
Q is the inversion division factor (see Table 2);
V is the volume, in millilitres, to which the sample is diluted before filteration (5.4.1);
m is the mass, in grams, of the test sample (5.4.1);
F is the fat, per cent by mass, in the sample; and
P is the protein, per cent by mass, in the sample.

TABLE 2 - Values of the inversion division factor (Q)

Light	Zinc acetate potassium ferrocyanide precipitant
Angular degrees and sodium light	0.8825 + 0.0006 (C-9) - 0.0033 (t-20)
Angular degrees and mercury green light or special Wratten screen, N 77A	1.0392 + 0.0007 (C-9) - 0.0039 (t-20)
International sugar scale degree and (j) light	2.549 + 0.0017 (C-9) - 0.0095 (t-20)

where,

C is the total sugars, per cent by mass, in the inverted solution (according to the polarimetric reading); and
t is the temperature, in celsius, of the inverted solution during the polarimetric determination.

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SRI LANKA STANDARDS INSTITUTION

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