SRI LANKA STANDARD 275 : 2014

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SPECIFICATION FOR
TOOTHPASTE

(***Third Revision***)

SRI LANKA STANDARDS INSTITUTION

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SLS 275 : 2014

(Attached AMD 532)

Gr. 12

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Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

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

SRI LANKA STANDARD
SPECIFICATION FOR TOOTHPASTE
(***Third Revision***)

FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2014-04-24.

This Standard was first published in 1974, revised in 1980 and 2006. In this Third Revision, all the amendments issued to SLS 275 : 2006 have been incorporated. The requirement and limits for heavy metals have been changed.

Dentifrice is a substance which may be used with a toothbrush for the purpose of cleaning the accessible surface of the teeth and the gum. In addition, toothpaste is used to reduce dental caries, help to maintain healthy gums and delay the development of objectionable mouth odours. The toothpaste when used in the normal manner shall not cause toxic or irritant reaction to the mucous membrane of the oral cavity, make harm to teeth, gums and any part in the mouth, in general.

For the purpose of deciding whether a particular requirement of this specification is complied with the final value, measured or computed, expressing the result of a test or an analysis, shall be rounded off in accordance with **SLS 102**. The number of decimal places retained in the rounded off value shall be the same as that of the specified value in this specification.

Guidelines for the determination of a compliance of a lot with the requirements of this standard based on statistical sampling and inspection are given in Appendix **A**.

This specification is subject to the restrictions imposed under the Cosmetic, Devices and Drugs (CDDA) Act No. 27 of 1980, Consumer Affairs Authority Act No. 09 of 2003 and the Regulations framed there under. Toothpaste is considered as a cosmetic according to the CDDA Act.

In the preparation of this standard assistance obtained from the following publications is gratefully acknowledged :

BS 5136 : 1981	British Specification for Toothpastes
IS 6356 : 2001	Indian Standard Specification for Toothpaste
ISO 11609 : 2010 (E)	Dentistry Toothpastes – Requirements tests methods and marking
Vol. 3, 9th Edition : 1993	Poucher's Perfumes Cosmetics and Soaps

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for toothpaste in the form of paste, cream or gel, with or without herbs/ herbal extracts including medicated toothpastes.

1.2 This standard does not prescribe requirements related to therapeutic/ medicinal claims of toothpastes.

2. REFERENCES

ISO/R 468	Surface roughness
ISO /TR 17276	Cosmetics - Analytical approach for screening and quantification methods for heavy metals in cosmetics
SLS 102	Rules for rounding off numerical values
SLS 124	Test sieves
SLS 428	Random sampling method
SLS 457	Part 1 Dyes pigments and colour additives recognized as safe
SLS 457	Part 2 : Raw materials and adjuncts other than dyes, colours and pigments
SLS 1316	Code of good manufacturing practices for cosmetics industry
SLS 1349	Method for the enumeration and detection of aerobic mesophilic bacteria in cosmetics

3 DEFINITIONS

For the purpose of this specification the following definitions shall apply :

3.1 bio-compatible : Not producing any toxic, injurious or immunological response or rejection in living tissue.

3.2 dentifrice : Any substance or combination of substances specially prepared for the public for hygiene of the accessible surfaces of teeth and surrounding tissues.

3.3 toothpaste : Any semi-solid dentifrice preparation presented in the form of paste, cream or gel used for cleaning of teeth.

4 TYPES

4.1 The toothpaste shall be of the following two types based on the efficacy of the fluoride :

Type 1 - Fluoridated toothpaste (total fluoride, 850 – 1 150 mg/kg or ppm)
Type 2 - Non-Fluoridated toothpaste (maximum total fluoride, 50 mg/kg or ppm)

5. REQUIREMENTS

The toothpaste shall be manufactured by a process adhering to Good Manufacturing Practices (GMP) complying with **SLS 1316.**

The toothpaste shall meet performance and stability specifications based on in-vitro studies for the complete duration of the declared shelf life. The date of expiry / best before / shelf life of the finished product shall be determined and declared by the manufacturer on the results of stability.

5.1 Raw materials

5.1.1 The dyes, colours and pigments, if any, shall comply with the provisions of **SLS 457 : Part 1**.

5.1.2 The raw materials and adjuncts other than dyes, colours and pigments shall comply with the provisions of **SLS 457 : Part 2**.

5.1.3 Diethylene glycol shall not exceed 0.25 per cent by mass.

5.2 Flavour

Flavour(s) shall be in food-grade and be distinct, pleasant and bio-compatible.

5.3 Readily fermentable carbohydrates

The toothpaste shall not contain readily fermentable carbohydrates. Compliance shall be established by the absence of such compounds in the complete formula.

5.4 Extrusive content

When tested in accordance with Appendix **B.1,** it shall be possible to extrude toothpaste not less than 91 per cent of net mass declared from the tube or sachet in a continuous mass without application of excessive force which would cause damage to the tube.

5.5 Consistency

When examined in accordance with Appendix **B.2,** the toothpaste shall be a homogeneous mixture and shall be free of lumps or particles as per Sl. No. iv) of Table **1**, except where such particles are specifically added to the paste for cleaning benefits such as interdental cleaning and whitening.

5.6 Cohesiveness

When tested in accordance with Appendix **C,** the toothpaste extruded at $27^{o} \pm 2^{o}$C shall remain attached to the tube/ sachet for at least 15 seconds.

5.7 Stability

The toothpaste shall not segregate, ferment or deteriorate when cooled to a temperature of 0 ^{o} C for 1 hour and when heated to a temperature of 45 ^{o} ± 2 ^{o} C for a period of 72 hours when tested in accordance with Appendix **D**.

5.8 Tube/ sachet inertness

When tested in accordance with Appendix **E** the filled tube/ sachet and lining (if present) shall not be corroded or otherwise damaged by the toothpaste or the temperature itself when heated to a temperature of 45 ^{o} C for a period of 72 hours.

5.9 The toothpaste shall also comply with the requirements given in Table **1** when tested in accordance with the relevant methods given in Column (**4)** of the Table **1**.

5.9.1 The approval of the relevant authority shall be obtained for the fluoride level(s) of toothpastes with special claims / user category.

5.10 Microbiological limits

The product shall also comply with the microbiological limits given in Table **2**, when tested according to the relevant methods given in Column (**4)** of Table **2**.

5.11 Net mass

The net mass of toothpaste packed shall not be less than the net mass declared on the package when tested in accordance with Appendix **B.1**.The maximum net mass of the content shall be as agreed between the purchaser and the supplier.

TABLE 1 – Requirements for toothpaste

Sl. No. (1)	Characteristic (2)	Requirement (3)	Method of Test (4)
i)	Moisture and volatile matter, per cent by mass	12-55	Appendix **F**
ii)	pH of aqueous suspension	5.8-10.5	Appendix **G**
iii)	Heavy metals		ISO /TR 17276
	a) Lead (as Pb) mg/kg, max.	1	
	b) Arsenic (as As) mg/kg, max.	0.5	
	c) Cadmium (as Cd) mg/kg, max.	0.1	
	d) Mercury (as Hg) mg/kg, max.	0.2	
	e) Antimony (as Sb) mg/ kg, max.	0.5	
iv)	Fineness		Appendix **H**
	a) Particles retained on 150-µm sieve, per cent by mass, max.	1.0	
	b) Particles retained on 75-µm sieve, per cent by mass, max.	2.5	
v)	Total Fluoride (throughout the shelf life), mg/kg		Appendix **J**
	a) for Type 1	850 – 1 150	
	b) for Type 2, max.	50	
vi)	Abrasion	To satisfy the test	Appendix **K**

TABLE 2 – Microbiological limits

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Total aerobic bacteria per gram, max	1 000	SLS 1349
ii)	E.coli per 10 grams	Absent	Appendix L
iii)	Salmonellae per 10 grams	Absent	Appendix L

6 PACKAGING

6.1 The toothpaste shall be filled in collapsible tubes or in properly sealed sachets compatible with the contents. The tubes shall have non-corrosive screw caps which fit the neck externally. The area of the orifice of the tube/ sachet through which the paste is to be extruded shall be greater than 13 mm^2 and less than 50 mm^2 when tested by a suitable method (see Note).

NOTE : *Go-no-go type gauge may be used.*

6.2 Each toothpaste tube shall be in a suitable outer container.

6.3 Paint on the fold or crimp end of the tube shall not extend more than 3 mm from the end of the tube when unfolded.

7 LABELLING AND MARKING

7.1 Each container and sachet shall be marked legibly and indelibly with the following information :

a) Name of the product for Type 1 as “Fluoridated Toothpaste”, and as “Non fluoridated toothpaste” for Type 2 ;
b) In fluoridated toothpaste either the words “use a ‘smear’ for children under 2 years and a pea sized amount for children of 2-6 years” or the same in the pictorial form
(see Note) ;
c) Name and address of the manufacturer for locally manufactured products;
d) Name and address of the distributor in Sri Lanka / importer including the country of origin, in the case of imported products;
e) Brand name / trade name or registered trade mark, if any ;
f) Date of manufacture ;
g) Date of expiry / best before/ shelf life ;
h) Batch or code number ;
j) Net mass of the product, in grammes ;
k) List of ingredients ; and
l) Fluoride content for fluoridated toothpaste, in ppm or mg/kg.

NOTE : *See* **Figure 1** *for illustration of smear and a pea-sized amount of toothpaste.*

7.2 Each tube shall be marked legibly and indelibly with the following information :

a) Name of the product for Type 1, as “Fluoridated Toothpaste” and for Type 2, as “Non fluoridated toothpaste” ;
b) Name and address of the manufacturer for locally manufactured products;

c) Name and address of the distributor in Sri Lanka / importer including the country of origin, in the case of imported products;
d) Brand name / trade name or registered trade mark, if any ;
e) Batch or code number ;
f) Net mass of the product, in grammes;
g) Date of expiry/ best before ; and
h) Fluoride content for fluoridated toothpaste , in ppm or mg/kg.

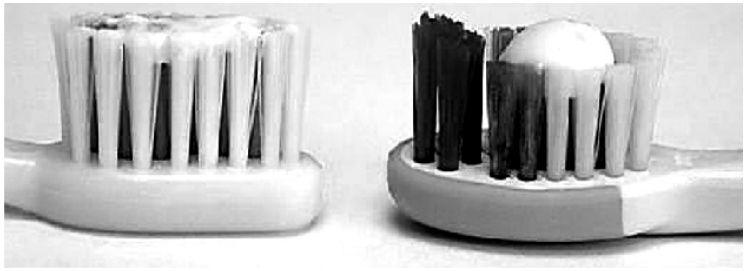

FIGURE 1 – Illustration of a smear (left) and a pea-sized (right) amount of toothpaste

APPENDIX A
COMPLIANCE OF A LOT

The sampling scheme given in this Appendix should be applied where compliance of a lot to the requirements of this standard is to be assessed based on statistical sampling and inspection.

Where compliance with this standard is to be assessed based on manufacturer's control systems coupled with type testing and check tests or any other procedure, an appropriate scheme of sampling and inspection should be adopted.

A.1 LOT

In a single consignment, all the packages containing toothpaste of the same size (mass) representing the same batch of manufacture shall constitute a lot.

A.2 SCALE OF SAMPLING

A.2.1 Samples shall be tested from each lot for ascertaining the conformity of the toothpaste to all the requirements of this specification.

A.2.2 The number of tubes / sachets to be selected from each lot shall be in accordance with Column (**2**) and Column (**3**) of Table **3**.

TABLE 3 - Scale of sampling

Number of tubes/ sachets in the lot (1)	**Number of tubes/ sachets to be selected** (2)	**Sub-sample size** (3)
Up to 1 200	21	5
1 201 to 3 200	25	7
3 201 to 10 000	30	10
10 001 and above	35	15

A.2.3 If the tubes/ sachets are packed in cartons ten per cent of the cartons subject to a minimum of five (05) cartons shall be selected and as far as possible an equal number of tubes shall be drawn from each carton so selected, to form a sample as given in Table **3**.

A.2.4 The cartons and tubes/ sachets shall be selected at random. In order to ensure randomness of selection, tables of random numbers as given in **SLS 428** shall be used.

A.3 NUMBER OF TESTS

A.3.1 Each tube/ sachet selected as in **A.2.3** shall be inspected and tested for packaging and marking requirements given in **6** and **7** of this specification.

A.3.2 Sufficient amount of paste shall be taken under aseptic conditions from five (05) tubes selected as in **A.2.3** and mix to form a composite sample. The composite sample thus obtained shall be tested for microbiological requirements given in **5.10**.

A.3.3 A sub sample of eight tubes/ sachets shall be drawn from the sample selected as in **A.2.3** and tested for the requirement given in **5.6.**

A.3.4 Withdraw the paste from all eight tubes / sachets tested in **A.3.3** and mix thoroughly to give a homogeneous composite sample. This composite sample shall be tested for the requirements given in **5.2, 5.5, 5.7** and **5.9**.

A.3.5 A sub sample of size as given in Column (**3**) of Table **3** shall be selected from the sample and tested for the requirements given in **5.4** and **5.11**.

A.3.6 The remaining tubes/ sachets in the sample shall be tested for the requirement given in **5.8**.

A.4 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied:

A.4.1 Each tube/ sachet inspected and tested as in **A.3.1** satisfies the relevant requirements.

A.4.2 The test results on composite sample tested as in **A.3.2** satisfy the relevant requirements.

A.4.3 The number of tubes/ sachets tested as in **A.3.3** not conforming with the requirement specified in clause **5.6** should not exceed one.

A.4.4 The test results on composite sample tested as in **A.3.4** satisfy all the relevant requirements.

A.4.5 From the results of testing the characteristics specified in **A.3.5** the mean (x) and the range (R) shall be calculated. The value of the expression x - 0.4 R shall not be less than the relevant specification limit.

A.4.6 Each tube/ sachet tested as in **A.3.6** satisfies the relevant requirement.

NOTES :

1 *Mean (x)* = $\frac{\text{Sum of the observed values}}{\text{Number of values}}$

2 *Range (R)* = *The difference between maximum value and minimum value.*

APPENDIX B
DETERMINATION OF NET MASS, EXTRUSIVE CONTENT AND CONSISTANCY

B.1 DETERMINATION OF NET MASS AND EXTRUSIVE CONTENT

B.1.1 Procedure

Weigh the gross mass of the tube or sachet (m_1). After opening the tube or sachet, extrude the bulk of the contents of the tube by the application of light pressure with the fingers. Starting from the crimp end, roll the tube up to the shoulder over a cylindrical surface of diameter 7.5 mm to 8.0 mm approximately. Weigh the remaining of toothpaste with the tube or sachet (m_2). Cut the tube or sachet and remove the remaining toothpaste, wash, dry and weigh the empty tube or sachet (m_3).

B.1.2 Calculation

Net mass packed, g = $m_1 - m_3$

Extrusive content, per cent by mass of declared net mass = $\frac{(m_1 - m_2)}{M} \times 100$

where,

m_1 is the gross mass of tube or sachet of the toothpaste, in grammes;
m_2 is the mass of the remaining of toothpaste with the tube or sachet, in grammes;
m_3 is the mass of empty tube or sachet, in grammes ; and
M is the net mass declared on the tube or sachet.

B.2 DETERMINATION OF CONSISTANCY

Examine the toothpaste extruded as in **B.1.1** for lumps or particles.

APPENDIX C
DETERMINATION OF COHESIVENESS

C.1 PROCEDURE

Hold the tube / sachet in a vertical position with the mouth downwards. Squeeze out a 10-mm ribbon of toothpaste at a temperature of 27 $\pm$ 2 ^{o}C. Observe whether the ribbon remains attached to the tube for at least 15 seconds.

APPENDIX D
DETERMINATION OF STABILITY

D.1 PROCEDURE

Place portions of the toothpaste in four glass test tubes and close them. Heat two of the test tubes/ sachet at 45 ^{o}C for 72 h. Examine the toothpaste for separation, fermentation and deterioration. Cool the remaining two test tubes to 0 ^{o}C for one hour. Examine the toothpaste for separation, fermentation and deterioration.

APPENDIX E
DETERMINATION OF TUBE/ SACHET INERTNESS

E.1 PROCEDURE

Heat the tube of toothpaste to 45 ^{o}C for 72 h. Extrude a part of the toothpaste. Examine for contamination on the paste. Slit the tube and remove the toothpaste. Examine the surface of the tube and cap or sachet for corrosion, chemical attack or other injury.

There shall be no corrosion or damage observed.

APPENDIX F
DETERMINATION OF MOISTURE AND VOLATILE MATTER

F.1 PROCEDURE

Weigh, to the nearest 0.1 g, about 2 g of the toothpaste in a suitable tared dish. Dry it at 105 ^{0} $\pm$ 2 ^{o}C in an oven to constant mass.

Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing until the difference in mass between two successive weighing does not exceed 5.0 mg.

F.2 CALCULATION

$$Moisture\ and\ volatile\ matter,\ per\ cent\ by\ mass\ =\ \frac{m_1 - m_2}{m_1}\ x\ 100$$

Where

m_2 is the mass, in grams, of the material after drying, and

m_1 is the mass, in grams, of the paste taken for the test.

APPENDIX G
DETERMINATION OF pH VALUE

G.1 PROCEDURE

Weigh, to the nearest 0.1 g, about 10 g of the toothpaste, in a 150-ml beaker. Add 45 ml of freshly distilled and cooled water and mix well. Determine the pH with a pH meter using glass calomel electrodes.

APPENDIX H
DETERMINATION OF FINENESS

This Appendix prescribes two test methods for the determination of fineness.

METHOD I

H.1 ON 150 - µm SIEVE

H.1.1 Procedure

Weigh, to the nearest milligram, about 10 g of the toothpaste in a 150 - µm sieve conforming to **SLS 124.** Wash by means of a slow stream of running tap water and finally with a fine stream, from a wash bottle until all the material that can pass through the sieve has passed. Leave the sieve for the water to drain and then dry the sieve on a steam bath. If there is any residue on the sieve carefully transfer it to a tared watch glass and dry it in an oven at $105^{0} \pm 2\ ^{o}C$. Cool in a desiccator and weigh . Repeat the process of drying, cooling and weighing until the difference in mass between two successive weighings does not exceed 5.0 mg.

H.1.2 Calculation

$$Material\ \ retained\ \ on\ \ 150-\mu m\ \ sieve,\ \ per\ \ cent\ \ by\ \ mass\ = \frac{m_2}{m_1}\ x\ 100$$

where

m_2 is the mass, in grams, of the residue retained on the sieve, and

m_1 is the mass, in grams, of the material taken for the test.

H.2 ON 75-µm SIEVE

H.2.1 Procedure

Weigh, to the nearest milligram, about 10 g of the toothpaste in a 75-µm sieve conforming to **SLS 124** and proceed as in **H.1.1**. If there is any residue on the sieve carefully transfer it to a tared watch glass and dry it in an oven at $105^{0} \pm 2\ ^{o}C$. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing until the difference in mass between two successive weighings does not exceed 5.0 mg.

H.2.2 Calculation

Calculate the material retained on the75-µm sieve in a manner similar to that specified in **H.1.2**.

METHOD 2

H.3 APPARATUS

H.3.1 Ultrasonic bath, Trans-O-Sonic Compact model on equivalent.
(60 ± 10 watts power with 35 ± 5 KH_ZUltrasonic frequency, 1 – 2 watts/inch 2 power density, L x B x H 225 x 125 x 60 mm tank)

H.3.2 On 150-µm sieve

Weigh, to the milligram, nearest 0.1 gram, about 20 g of the toothpaste. Place it in a 250-ml beaker. Add 200-ml of water and allow to stand for about 30 minutes with occasional stirring until the toothpaste is completely dispersed free of toothpaste/gel flocks trapping the agglomerates. Transfer the beaker ultrasonic bath. Fill the ultrasonic bath (2 litre capacity) to about three-fourth height with water. Clamp the above beaker in the centre of the bath keeping about 1 cm clearance from the bottom of the bath and subject to ultrasonification for 10 minutes to completely loosen out the constituents.

Transfer this suspension quantitatively to a 150-µm sieve and wash by means of a slow stream of running tap water and finally with a fine stream from a wash bottle until all the matter that can pass through the sieve has passed. Let the water drain out and then dry the sieve containing the residue in an oven. If there is any residue on the sieve, carefully transfer it to a tared watch glass and dry it to constant mass in an oven at $105^{0} \pm 2^{0}$ C. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing until the difference in mass between two successive weighings does not exceed 5.0 mg.

H.3.3 Calculation

Calculate the material retained on 150-µm sieve in a manner similar to that specified in **H.1.2.**

H.3.4 On 75-µm sieve

Weigh to the nearest milligram about 20 g of the toothpaste and proceed as **H.2.1** using a 75-µm sieve. If there is any residue on the sieve carefully transfer it to a tared watch glass and dry it in an oven at $105^{0} \pm 2^{0}$ C. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing until the difference in mass between two success weighings does not exceed 5.0 mg.

H.3.5 Calculation

Calculate the material retained on 75-µm sieve in a manner similar to that specified in **H.1.2**.

APPENDIX J
DETERMINATION OF FLUORIDE

This method has been specified in this appendix for the determination of total fluoride of toothpaste, containing fluoride or monofluorophosphate.

J.1 PRINCIPLE OF METHOD

Fluoride ion is determined in an extract of the product by direct potentiometry with a fluoride ion-selective electrode. The electrode responds only to fluoride ions, but monofluorophosphate can be measured after acid hydrolysis to fluoride and orthophosphate.

J.2 REAGENTS

J.2.1 *Standard Fluoride solutions*, 1 000 mg/1, 100 mg/1, 10 m/1, 1 mg/1 and 0.1 mg/l. Dry a little sodium fluoride Analar at 105 ^{o}C for 1 h and cool to room temperature. Accurately weigh 2.210 g, dissolve in water, transfer quantitatively to a 1 000 ml

volumetric flask, dilute to volume and mix. The exact concentration of fluoride is 452.4 mg/l (NaF contains 45.24 % F), where W is the mass in grams. Using polypropylene pipettes and volumetric flasks, make successive tenfold dilutions of this solution to obtain solutions containing 100, 10, 1 and 0.1 mg/l fluoride ion. Record their exact concentrations.

J.2.2 *Total Ionic Strength Adjusting Buffer* (TISAB). Dissolve 294 g trisodium citrate dehydrate Analar, 68 g sodium acetate trihydrate Analar and 29 g sodium chloride Analar in about 600 ml hot water. Cool, adjust the pH to 6.4 with glacial acetic acid Analar, dilute to 1 000 ml and mix. The function of the TISAB is to ensure that all readings are made on solutions of the same ionic strength and at the same pH, and to sequester metal ions, particularly Al^{3+}, that form stable covalent complexes with F^- ions.

J.2.3 *Sulfuric acid solution*, $c(H_2SO_4)$ = c. 1.0 mol/l. Slowly and with continuous cooling, pour 55 ml concentrated sulfuric acid (CAUTION ; CORROSIVE) into about 500 ml water. Cool, dilute to 1 000 ml and mix.

J.2.4 *Sodium hydroxide solution*, c(NaOH) = c. 1.0 mol/l. Dissolve 40 g sodium hydroxide pellets (CAUTION ; CAUSTIC) in water, cool dilute to 1 000 ml and mix.

J.2.5 *Phenolphthalein indicator.*

J.3 APPARATUS

J.3.1 *Centrifuge capable of 4 000 r.p.m.*
J.3.2 *Plastic centrifuge tubes, 50 ml, with caps*
J.3.3 *Polypropylene 10 and 20 ml pipettes, 100 ml volumetric flasks and 100 ml beakers*
J.3.4 *Potentiometer, or pH meter with millivolt scale*
J.3.5 *Fluoride ion-selective electrode. The Orion 94-01 is suitable*
J.3.6 *Calomel reference electrode. The Orion 94-01 is suitable*
J.3.7 *Magnetic stirrer and plastic-coated stirrer bar.*

J.4 PROCEDURE

J.4.1 Preparation of calibration curve

Using polypropylene pipettes, transfer 20 ml of each of the 100, 10, 1 and 0.1 mg/l fluoride solutions into 100 ml polypropylene beakers. Add to each by pipette 20 ml TISAB. Add a stirrer bar to the beaker containing the 100 mg/l solution, place it on a magnetic stirrer, insert the electrodes and commence stirring. Record the millivolt reading at 1 min intervals until two consecutive readings are equal. Repeat steps 3 and 4 with each of the other three fluoride standards. Let the four readings be A, B, C and D.

Confirm that A - B, B - C and C - D are equal within 1 mV and that all three differences lie between 56 and 59 mV. If this is not so, or if the millivolt readings take an excessive time to become constant, carefully clean the sensing surface of the fluoride electrode and repeat the measurements. On semi-log graph paper, plot millivolts (vertical axis) against concentration (logarithmic scale), or plot millivolts against the logarithm of the concentration on ordinary graph paper. Use actual, not nominal, concentrations. There is no need to divide by two to correct for the dilution with TISAB, because the test solutions will be diluted in the same ratio. Draw the best straight line through the points. The curve has a negative slope, i.e. higher concentrations give lower readings.

J.4.2 Preparation of aqueous extract of toothpaste

Accurately weigh about 5 g (*M* g) toothpaste in a 100 ml beaker. Add 10 ml cold water and work to a smooth cream with a glass rod. Add a further 20 ml cold water and stir until homogeneous. Transfer quantitatively to a 100 ml polypropylene volumetric flask, dilute to volume and mix. Transfer about 40 ml of the suspension to each of the two 50 ml plastic centrifuge tubes and counterbalance them, with caps, to within 0.1 g. Cap the tubes, place them in diametrically opposed positions in the centrifuge and spin at 4 000 r.p.m. until the supernatant liquid is completely clear. Decant the supernatant liquid into a clean, dry stoppered polypropylene flask (solution A). Do not dilute to volume, this has already been done. Pipette 20 ml into a 100 ml polypropylene volumetric flask dilute to volume and mix (solution B).

J.4.3 Hydrolysis of monofluoroophosphate

Pipette 20 ml solution A into a 100 ml beaker, add 5 ml c. 1.0 mol/l sulfuric acid solution, cover with a watch glass and boil very gently for 5 min. Cool to room temperature. Rinse the watch glass with a jet of water, collecting the rinsings in the beaker. Neutralize with phenolphthalein with c. 0.1 mol/l sodium hydroxide solution. Quantitatively transfer to a 100 ml polypropylene volumetric flask, dilute to volume and mix thoroughly (solution C).

J.4.4 Determination of fluoride

For determination of free fluoride, use solution B. For determination of monofluorophosphate use solution C. Pipette 20 ml of the appropriate solution into a 100 ml polypropylene beaker. Add by pipette 20 ml TISAB. Add a stirrer bar, insert the electrodes and commence stirring. Record the millivolt reading at 1-min intervals until two consecutive readings are equal. Final reading = E mV.

NOTE : *If only free fluoride (eg. NaF) is added as declared by the manufacturer, determination of monoflurophosphate should not be required.*

J.4.5 Calculation

Read from the calibration curve the logarithm of fluoride concentration corresponding to E mV. Take its antilog to find the fluoride concentration in the solution measured. Let this be C_1 mg/l for solution B and C_2 mg/l for solution C. Fluoride content in solution A = 5 C_1 and 5 C_2 respectively.

If only free fluoride is added :

Total fluoride content in toothpaste = 500 C_1/M mg/kg (ppm)

If free fluoride and monofluorophosphate are added, or if only monofluorophosphate is added :

Total fluoride content in toothpaste = 500 C_2/M mg/kg (ppm)

where

C_1 is the fluoride concentration of solution B in mg/l ;
C_2 is the fluoride concentration of solution C in mg/l ; and
M is the mass of toothpaste sample in g.

APPENDIX K
DETERMINATION OF ABRASION

K.1 PRINCIPLE OF METHOD

Toothpaste placed on a glass slide is subjected to reciprocating strokes from a metal disc. The glass slide is then examined for any scratching.

K.2 APPARATUS

An apparatus similar to that shown in Figure **2** shall be used. It consists essentially of the following components :

K.2.1 Glass slide, soda lime, microscope slide.

K.2.2 Metal disc, a copper nickel alloy disc 20 mm diameter, 2 mm width, surface finish of 10 µm R_a (see **ISO/R 468**) (**the surface finish shall not be obtained with abrasives**). Inspect the rim carefully by means of a hand lens to ensure the absence of projections capable of scratching glass.

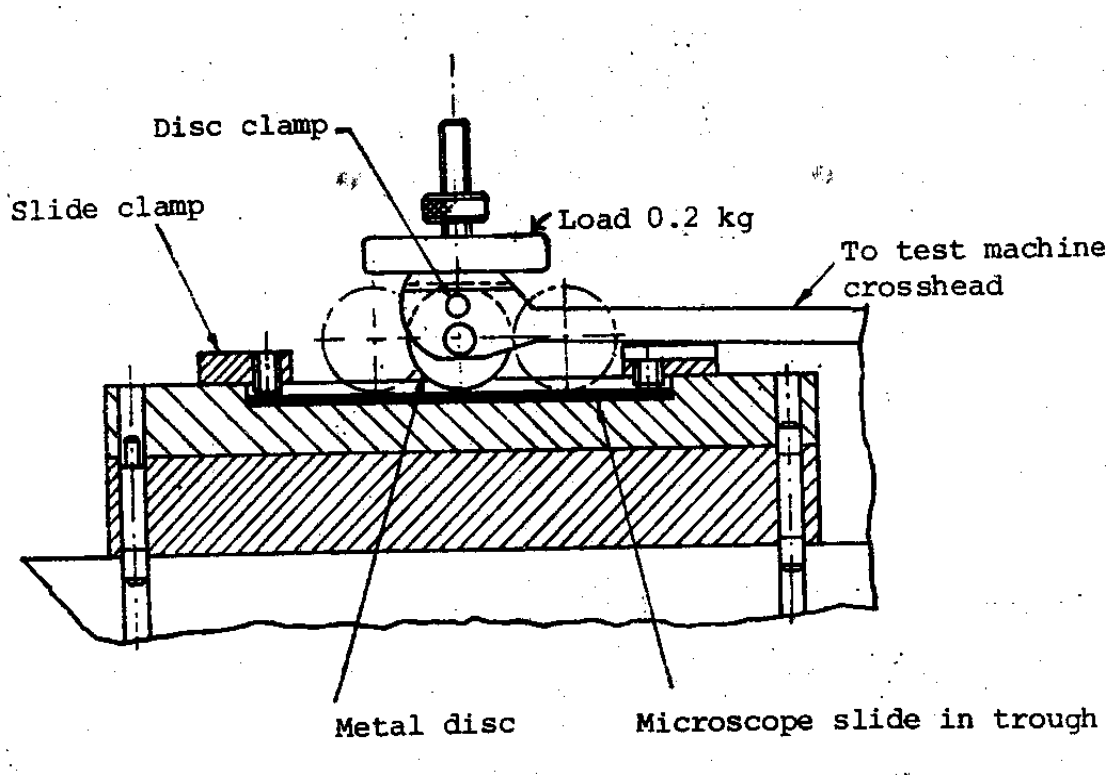

FIGURE 2 - Glass slide disc test apparatus

K.3 PROCEDURE

K.3.1 Clean the glass slide with chromic acid, rinse in water and dry. Place about one gram of the toothpaste on the slide and operate the apparatus shown in Fig. **2** for 100 double strokes using a load of 200 g on the disc. Run a control test along side the previous track using glycerine as the control medium. Rotate the disc to provide a fresh surface for each test. After completion of both tests wash the glass slide and dip into dilute nitric acid to remove any particles of metal alloy adhering to the slide. Wash the slide in water and after drying, view in reflected light.

K.3.2 If the track produced by the toothpaste shows visible scratches, then it shall be considered to have failed the test. Ignore any smooth polished grooves.

K.3.3 An example of this type of grooving will be exhibited by the glycerol test track.

K.3.4 If the glycerol test track shows signs of scratching, then the test shall be repeated using a fresh glass slide and a different position on the rim of the disc.

APPENDIX L
DETERMINATION OF SALMONELLA AND ESCHERICHIA COLI

L.1 OUTLINE OF THE METHOD

The test consists of enrichment of above bacteria from a sample in a suitable culture medium and then in selective culture media individually and after incubation streaking over on selective agar plates for identification.

L.2 APPARATUS AND EQUIPMENT

L.2.1 *Conical flasks with glass-beads-100, 250 and 500* ml

L.2.2 *Sterile pipettes – 1, 5 and 10 ml graduated*

L.2.3 *Test tubes – without rim,*

18 mm diameter x 150 mm height (with durhams tubes)
25 mm diameter x 150 mm height, and
38 mm diameter x 200 mm height

L.2.4 *Petridishes – sterile 15 x 100* mm

L.2.5 *Standard streaking loop, straight inoculative wire*

L.2.6 *Balance, sensitivity of 0.01* g

L.2.7 *Autoclave for steam sterilization at 121* ^{o}C *at 103* kN/m^2 *(15 lb per square inch) pressure*

L.2.8 *Oven – Temperature up to 250* ^{0}C

L.2.9 *Incubator – 30*^{o} *to 35* ^{0}C *and 37*^{o} $\pm$ *2* ^{0}C

L.2.10 *Water bath, - 45* ^{o} $\pm$ *2* ^{0}C *, boiling water bath*

L.2.11 *Laboratory microscope*

L.3 MEDIA AND REAGENTS

L.3.1 Lactose broth

Beef extract	3.0
Pancreatic digest of gelatin	5.0 g

Lactose	5.0	g
Soy-lecithin	5.0	g
Tween 20	40	ml
Distilled water	960	ml
pH after sterilization	6.9 ± 0.2	

Mix well and dispense in 90 ml in conical flasks of 250 ml capacity.

Autoclave at 121 ^{o}C with 103 kN/m^2 (15 lb per square inch) pressure for 15 minutes.

L.3.2 Selenite cystine broth

Pancreatic digest of casein	5.0	g
Lactose	4.0	g
Sodium phosphate	10.0	g
Sodium acid selenite	4.0	g
L-cystine	10	mg
Distilled water	1 000	ml
Final pH .	7.0 ± 0.2	

Mix and heat to effect solution. Heat in flowing steam for 15 minutes. Dispense in sterile 10 ml tubes aseptically. Do not autoclave for sterilization.

L.3.3 Tetrathionate broth

Pancreatic digest of casein	2.5	g
Peptic digest of animal tissue	2.5	g
Bile salts	1.0	g
Calcium carbonate	10.0	g
Sodium thiosulphate	30.0	g
Distilled water	1 000	ml

Heat the solution of solids to boiling. On the day of use, add a solution prepared by dissolving 5 g of potassium iodide and 6 g of iodine in 20 ml of water. Then add 10 ml of solution of Brilliant Green (1 : 1 000), and mix. Do not heat the medium after adding the Brilliant Green solution.

L.3.4 Brilliant Green Agar

Yeast extract	3.0	g
Peptic digest of animal tissue	5.0	g
Pancreatic digest of casein	5.0	g
Lactose	10.0	g
Sodium chloride	5.0	g
Sucrose	10.0	g
Phenol red	80	mg

Agar	20.0	g
Brilliant green	12.5	mg
Distilled water	1 000	ml

Boil the solution of solids for 1 minute. Sterilize just prior to use, melt the medium, pour into sterile petridishes, and allow it to cool.

L.3.5 Bismuth Sulphite Agar

Beef extract	5.0	g
Pancreatic digest of casein	5.0	g
Peptic digest of animal tissue	5.0	g
Dextrose	5.0	g
Sodium phosphate	4.0	g
Ferrous sulphate	300	mg
Bismuth sulphite indicator	8.0	g
Agar	20.0	g
Brilliant green	25	mg
Distilled water	1 000	ml
Final pH .	7.6 ± 0.2	

Heat the mixture of solids and water in a boiling water-bath, with swirling just to the boiling point. Do not over-heat or sterilize. Transfer at once to water-bath maintained at 50 ^{o}C, and pour into sterile petridishes as soon as the medium has cooled up to 50 ^{o}C.

L.3.6 MacConkey's broth

Peptone	20.0	g
Sodium chloride	5.0	g
Sodium taurocholate	5.0	g
Distilled water	1 000	ml
Bromocresol purple	10	mg
Lactose	10	g

Dissolve the peptone, the sodium chloride, and the sodium taurocholate in distilled water with the aid of water-bath. Adjust to pH 8.0 and boil for twenty minutes. Cool, filter and adjust to pH 7.4 ± 0.2. Add the Lactose and indicator solution, mix and distribute in tubes containing inverted Durham's tubes. Autoclave at 121 ^{o}C for 103 kN/m^2 (15 lb per square inch) pressure for 15 minutes.

L.3.7 MacConkey's Agar

Pancreatic digest of gelatin	17.0	g
Pancreatic digest of casein	1.5	g
Peptic digest of animal tissue	1.5	g
Lactose	10.0	g
Bile salts mixture	1.5	g

Sodium chloride	5.0 g
Agar	13.5 g
Neutral red	30 mg
Crystal violet	1.0 mg
Distilled water	1 000 ml
pH after sterilization	7.1 ± 0.2

Boil the mixture of solids and water for 1 minute to effect solution. Autoclave at 121 ^{o}C with 103 kN/m^2 (15 lb per square inch) pressure for 15 minutes. Pour 15 to 20 ml in sterile petridishes aseptically.

L.3.8 Tripple Sugar-Iron-Agar

Pancreatic digest of casein	10.0 g
Pancreatic digest of animal tissue	10.0 g
Lactose	10.0 g
Dextrose	1.0 g
Ferrous ammonium sulphate	200 mg
Sodium chloride	5.0 g
Sodium thiosulphate	200 mg
Agar	13.0 g
Phenol red	25 mg
Distilled water	1 000 ml

After mixing and digesting on a boiling water-bath distribute 10 ml in test tubes of 18 x 150 mm size. Autoclave at 121 ^{o}C with 103 kN/m^2 (15 lb per square inch) pressure for 15 minutes. Cool approximately at 45 ^{o}C and put in slanting position to solidify.

L.4 GRAM STAINING METHOD

L.4.1 Outline of the method

The test consists essentially of applying crystal violet to a fixed smear from the culture, removing the primary stain after a suitable staining period and then applying iodine as a mordant. The mordant in turn is removed and declourizer is added to remove the primary stain where possible. Saffranin is then added as a counter stain.

Organisms are judged to be gram- positive under oil immersion lens of microscope, if they retain the primary stain after decolourization, gram negative organisms are decolourized and appear pink to red because they take up the counter stain.

L.4.2 Gram stain reagents

Stain 1

a) Crystal violet (90 per cent dye content) 2 g
Ethanol (95 per cent) 20 ml

b) Ammonium oxalate 0.8 g
Distilled water 80 ml

Mix **a** and **b**

Stain 2

Iodine 1 g
Potassium iodide 2 g
Ethanol (95 per cent) 25 ml
Distilled water 100 ml

Stain 3

5 ml of Stain –2 added to 95 ml of ethanol (95 per cent)

Stain 4

Saffranin 0.25 g
Ethanol (95 per cent) 10 ml
Distilled water 100 ml

L.4.3 Procedure for preparation of identification plates

Place a small loopful of distilled water on a clean microscope slide. Touch a sterile inoculating loop to a colony, rub it into the droplet to obtain an even suspension of microorganisms and spread the suspension over the surface of the slide to obtain correct density. Let the smear dry at room-temperature. Heat fix by passing the slide 3 times through a low gas flame. Cool to room-temperature before staining.

Flood the stain-1 on fixed smear for 1 minute. Wash excess of stain with tap water. Flood the smear with stain – 2 for 1 minute. Remove stain-2 by gentle washing in cold tap water. Flood off stain-3 until solvent runs colourless from the slide for 30 to 60 seconds. Wash off excess stain with cold tap water. Blot off excess water with paper, air dry and examine under oil immersion lens.

NOTE : *All the above media and reagents in dehydrated form are commercially available.*

L.5 PROCEDURE

Weigh and transfer aseptically 10 g of sample into a sterile conical flask containing 90 ml of Lactose broth. Mix well and incubate at 37 ^{o} ± 2 ^{o} C for 18 to 24 hours.

Examine the broth for growth, and if the growth is present, mix by gently shaking. Transfer aseptically 1 ml portions with the help of sterile pipette into :

a) 10 ml selenite cystine broth
b) 10 ml tetrathionate broth
   Incubate at 37 ^{o} ± 2 ^{o} C for 18 to 24 hours
c) 10 ml Sterile MacConkey's broth tube having Durhams tube
   Incubate at 37 ^{o} ± 2 ^{o} C for 48 hours

L.6 IDENTIFICATION TEST FOR SALMONELLA

By means of an inoculating loop streak portions from broth (a) and (b), on the surface of Brilliant Green Agar and Bismuth Sulphite Agar plates .Cover and invert the petridishes and incubate at 37 ^{o} ± 2 ^{o}C for 18 to 24 hours. Upon examination if none of the colonies conforms to the description given in Table **4**, the sample meets the requirement for the absence of Salmonellae.

Gram stain the portion of suspected colony and if the colonies of Gram-negative rods are found, proceed with further identification by transferring representative suspect colonies individually by means of inoculating wire, to a butt-slant tube of tripple-sugar iron agar by first streaking the surface of the slant and then stabbing the wire well beneath the surface. Incubate at 37 ± 2 ^{o}C for 18 to 24 hours. If examination discloses no evidence of tubes having alkaline (red) slants and acid (yellow) butts (with or without concomitant blackening of butt from hydrogen sulphide production), the sample meets the requirements for the absence of salmonella.

TABLE 4 – Morphological characteristics of salmonellae

Sl No. (1)	**Medium** (2)	**Description of Colony** (3)
i)	Brilliant green agar	Small transparent, colourless or pink to white opaque (Frequently surrounded by pink to red zone)
ii)	Bismuth sulphite agar	Black or Green

L.7 IDENTIFICATION TEST FOR ESCHERICHIA COLI

After incubation of MacConkey's broth tubes if the contents do not show acid and gas the sample meets the requirement of the test for the absence of Escherichia coli.

If there is acid and gas in the Mac Conkey's broth tube, streak a portion of the above growth on the surface of Mac Conkey's Agar Plate - Cover and invert the petridishes and incubate at 37 ^{o} ± 2 ^{o} C for 18 to 24 hours. Upon examination if none of the colonies conforms to the description of Table **5** for the medium, the sample meets the requirements of the test for the absence of Escherichia Coli.

TABLE 5 - Morphological Characteristics of Escherichia Coli

Medium (1)	**Description of colony** (2)
MacConkey's Agar	Brick-red, may have surrounding zone of precipitated bile

If colonies matching the description in Table **5** are found, and are Gram-negative proceed with further identification by transferring the suspect colonies individually by means of inoculating loop to the surface of Levine Eosin-Methylene Blue Agar plates. Divide the surface of the Agar plate, if multiple colonies are observed. Incubate at 37 ^{o} ± 2 ^{o} C for 18 to 24 hours. Upon examination, if none of the colonies exhibits both a characteristic metalic sheet under reflected light and a blue-black appearance under transmitted light, the sample meets the requirements of the test for the absence of Escherichia coli. The presence of Escherichia coli may be confirmed by further suitable cultural and biochemical tests.

L.8 VALIDATION OF TEST PROCEDURE

The validity of the results of the test set forth in this appendix rests largely upon the adequacy of a demonstration that the test samples to which they are applied do not of themselves inhibit the multiplication, under the test conditions, of micro organisms that may be present.

This can be done by adding 1 ml of not less than 10^{-8} dilution of 24 hours broth culture of :

i) Salmonella abony NCTC 6017
ii) Escherichia Coli ATCC 8739

to the first dilution (In Fluid Casein Digest Soy-Lecithin Polysorbate 20 Medium and Lactose broth with 0.5 per cent of soy-Lecithin and 4 per cent of Tween 20) of the test sample and following the test procedure.

If inspite of incorporation of inactivating agents and a substantial increase in the volume of diluent, it is still not possible to recover the viable cultures described above, it can be assumed that failure to isolate the inoculated organisms is attributable to the bactericidal activity of the product. This information serves to indicate that the sample is not likely to be contaminated with the given species of micro organisms. Monitoring should be continued in order to establish the spectrum of inhibition and bactericidal activity of the sample.

To prove the growth promoting ability of different culture media, organisms without sample preparation is run in the same manner.

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Amendment No: 01 approved on 2020-07-22 to SLS 275: 2014

SRI LANKA STANDARD SPECIFICATION FOR TOOTHPASTE (*Third Revision*)

EXPLANATORY NOTE

To incorporate the SLDA latest opinion on the parameter of the “total fluoride in toothpaste”

This amendment is issued accordingly.

Amendment No: 01 approved on 2020-07-22 to SLS 275: 2014

SRI LANKA STANDARD SPECIFICATION FOR TOOTHPASTE
(Third Revision)

2 REFERENCES

Delete the following:

"SLS 457 Part 1 Dyes pigments and colour additives recognized as safe
SLS 457 Part 2 Raw materials and adjuncts other than dyes, colours and pigments"

and

Insert the following.

"SLS 457 Cosmetics- Classification of raw materials
Part 1: Substances permitted subject to restrictions and permitted colourants, preservatives and UV filters (First revision)
Part 2: Prohibited substances (First revision)

Insert the following at the end of references.

"SLS 1587 Cosmetics - packaging and labelling"

4 TYPES

4.1 Delete the text given in type 1 and substitute the following:

"Type 1- Fluoridated toothpaste (total fluoride, 1 000-1 500 mg/kg or ppm.)"

5 REQUIREMENTS

5.1 Raw materials

Delete the Clause **5.1.1** and **5.1.2** and substitute the following:

5.1.1 The raw materials shall comply with the provisions of **SLS 457 Part 1** and **Part 2**.

TABLE1 : Requirements for toothpaste
Delete the limit given in Column (3) S.No. v) (a) “850- 1 150” and substitute the “1 000-1 500”

7 LABELLING AND MARKING

Delete the Clause 7.1 (b) In fluoridated toothpaste either the words use a ‘smear’ for children under 2 years and a pea sized amount for children of 2- 6 years” or the same in the pictorial form (see Note) and substitute the “In fluoridated toothpaste either the words” use a ‘smear’ for children under 3 years and a pea sized amount for children of 3 - 6 years” or the same in the pictorial form (see Note)” .

Insert the new Clause underneath the Figure 1.

7.3 The product labelling shall also be in compliance with **SLS 1587** (see Note).

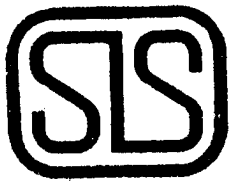
NOTE: Warning of conditions for use and warnings are not applicable to mark on the each container and tube.

……

SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI *which includes surveillance inspection of the factory, testing of both factory and market samples.*

Further particulars of the terms and conditions of the permit may be obtained from the Sri *Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.*



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