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SRI LANKA STANDARD 351:1983
UDC 661.772

SPECIFICATION FOR
RECTIFIED SPIRIT
(FIRST REVISION)

BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR RECTIFIED SPIRIT

(FIRST REVISION)

SLS 351:1983

(Attached AMD 286)

Gr.9

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BUREAU OF CEYLON STANDARDS
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This standard does not purport to include all the necessary provisions of a contract.

SRI LANKA STANDARD
SPECIFICATION FOR RECTIFIED SPIRIT
(FIRST REVISION)

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Bureau of Ceylon Standards on 1983-04-08, after the draft, finalized by the Drafting Committee on Rectified Spirit, had been approved by the Chemicals Divisional Committee.

This specification was first published in 1975. In this revision, a minimum value has been specified for the ethanol content of rectified spirit as against the range of values specified in the original standard. This amendment was made in the light of current trade practice both locally and abroad. The relative density value has also been changed in keeping with the amendment to the ethanol content of the material.

This specification is subject to the provisions of the Cosmetics, Devices and Drugs Act No. 27 of 1980, the regulations framed thereunder and the Excise Regulations of Sri Lanka, wherever applicable.

All standard values given in this specification are 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 shall be the same as that of the specified value in this specifications.

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

1 SCOPE

This specification prescribes the requirements, the methods of sampling and test for rectified spirit for use in the chemical, pharmaceutical and cosmetic industries and for production of potable alcoholic beverages.

2 REFERENCES

CS 102 Presentation of numerical values

SLS 428 Random sampling methods.

3 DEFINITIONS

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

3.1 **spirit** : Mixture of ethanol and water.

3.2 **proof spirit** : Mixture of ethanol and water which shall at a temperature of 10.6 °C, weigh twelve thirteenth part of an equal volume of distilled water at the same temperature. It has a relative density of 0.916 9 at 20 °C and contains 57.10 per cent by volume or 49.28 per cent by mass ethanol at 20 °C.

3.3 **overproof spirit (OP)** : Mixture of ethanol and water containing a greater percentage of ethanol than is contained in proof spirit. If an overproof strength is added to 100, the sum represents the volume of spirit at proof strength which 100 volumes of spirit of that particular overproof strength would yield when diluted with water. For example 100 volumes of 60 ° overproof spirit would yield 160 volume of proof spirit.

4 REQUIREMENTS

4.1 General requirements

The material shall be a colourless, clear, volatile, homogeneous liquid, miscible with water, solvent ether and chloroform. It shall be free from suspended matter and shall consist essentially of ethanol admixed with water, boiling at about 78 °C. Its odour shall be characteristic and spirituous and its taste burning. It shall be readily flammable, burning with a blue smokeless flame.

4.1.1 *Identification*

To 5 ml of 0.5 per cent by volume solution of the material add 1 ml of 0.1 N sodium hydroxide; on adding slowly 2 ml of iodine solution (see Note) the odour of iodoform should develop and a yellow precipitate should form.

NOTE - Iodine solution is made by dissolving 0.2 g of iodine and 0.3 g of potassium iodide in sufficient water to produce 10 ml.

4.2 Other requirements

The material shall also comply with the requirements given in Table 1 when tested according to the relevant methods given in Column 4 of the table.

TABLE 1 - Requirements for rectified spirit

Sl. No.	Characteristic	Requirement	Method of test reference to Appendix
(1)	(2)	(3)	(4)
i	Relative density at 20 °C/20 °C, max.	0.830 7	B
ii	Ethanol content		
	a) Per cent by volume at 20 °C, min.	90.00	C
	b) Per cent by mass, min.	85.68	C
	c) Degrees overproof, min.	57.80	C
iii	Acidity or alkalinity	To satisfy the requirements of the test.	D
iv	Aldehydes and ketones	- do -	E
v	Fusel oil and allied impurities	- do -	F
vi	Ketones, isopropyl alcohol and t-butyl alcohol	- do -	G
vii	Methyl alcohol	- do -	H
viii	Oily or resinous substances (clarity)	- do -	J
ix	Non volatile matter	- do -	K
x	Reducing substance	- do -	L

5 PACKAGING AND MARKING

5.1 Packaging

The material shall be packed in suitable clean, dry and securely closed containers.

5.2 Marking

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

a) The words *Rectified spirit*;

b) The manufacturer's name and address;

c) The volume in millilitres or litres as the case may be;

d) Batch or code number;

e) Pictorial marking for *Flammable liquid* the text of which shall be in Sinhala, Tamil and English (see **5.2.2**, **5.2.2.1** and Fig. 1);

f) Pictorial marking for *This way up* (see Fig. 2); and

g) The country of origin.

5.2.2 The marking for *Flammable liquid* may appear on a label or may preferably be stencilled on the container. It shall be in the form of a square set at 45°, the dimensions depending on the size of the container. It shall also be divided into two equal triangles, the upper being reserved for the symbol and the lower for the text.

5.2.2.1 It is recommended that the shaded portions of the symbol, the border of the marking as shown in Fig. 1 and the text be in

a) red, on a contrasting background,

b) black, if the background is white.

FIGURE 1 - Pictorial marking for "Flammable liquid"

FIGURE 2 - Pictorial marking for "This way up"

5.2.3 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.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in Appendix A.

7 METHODS OF TEST

Test shall be carried out as prescribed in Appendices B to L.

8 CONFORMITY TO STANDARD

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

8.1 Packaging and marking

Each container examined as in A.7.1 satisfies the packaging and marking requirements.

8.2 Ethanol content and relative density

8.2.1 From the results of testing individual samples as in A.7.2 the calculated expression $\bar{x} - 0.6R$ for the requirement for the ethanol content is greater than or equal to the corresponding value.

8.2.2 For the results of testing individual samples as in **A.7.2** the calculated expression $\bar{x} + 0.6R$ for the requirement for the relative density is less than or equal to the corresponding value.

NOTES

1 *Mean* $(\bar{x})$ = $\frac{\text{Sum of the test results}}{\text{Number of test results}}$

2 *Range* (R) = *Difference between the maximum and the minimum of test results.*

8.3 Requirement other than the ethanol content and relative density

The composite sample tested as in **A.7.3** satisfies the relevant requirements.

APPENDIX A

SAMPLING

A.1 LOT

All containers of the same size in a single consignment of the material drawn from one batch of manufacture shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

A.2.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

A.2.2 The sampling instrument shall be clean and dry.

A.2.3 To draw a representative sample the contents of each container selected for sampling shall be mixed as thoroughly as possible.

A.2.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from contamination.

A.2.5 The samples shall be placed in suitable clean, dry and air-tight glass containers.

A.2.6 The sample containers shall be of such size that an ullage of at least 10 per cent is left after pouring in the sample.

A.2.7 Each sample container shall be sealed air-tight with the stopper after filling and marked with necessary details of sampling.

A.2.8 Samples shall be stored in a cool and dry place.

A.2.9 Samples shall be obtained from freshly opened containers.

A.3 SAMPLING INSTRUMENTS

The following forms of sampling instruments may be used.

a) **Sampling bottle or can for taking samples from various depths in large tanks**

It consists of a weighted bottle or metal container, with removable stopper or top, to which is attached a light chain (see Fig. 3). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

b) **Sampling tube**

It is made of metal or thick glass and, is 20 mm to 40 mm in diameter and 350 mm to 750 mm in length (see Fig. 4). The upper and lower ends are conical and reach 6 mm to 12 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

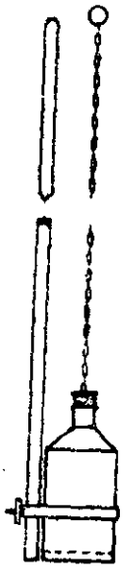

FIGURE 3 - Sampling bottle or can

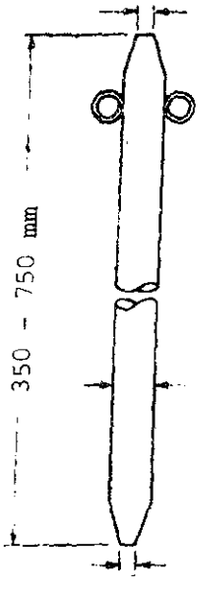

FIGURE 4 - Sampling tube

A.4 SCALE OF SAMPLING

A.4.1 The conformity of a lot to the requirements of this specification shall be determined on the basis of tests carried out on the samples selected from the lot.

A.4.2 The number of containers to be selected from a lot for sampling shall be as given in Table 2.

TABLE 2 - Scale of sampling

Number of containers in the lot (1)	Number of containers to be selected (2)
2 - 15	2
16 - 40	3
41 - 65	4
66 - 110	7
Over 110	10

A.4.3 The containers shall be selected at random. In order to ensure randomness of selection random number tables as given in **SLS 428** shall be used.

A.5 INDIVIDUAL AND COMPOSITE SAMPLES

A.5.1 An equal quantity of material shall be drawn from the top, middle and bottom portions of a container using an appropriate sampling instrument. The material so obtained shall be mixed to form an individual sample of not less than 60 ml. Individual samples shall be obtained from each container selected as in **A.4.2.**

A.5.2 An equal quantity of material shall be drawn from the top, middle and bottom portions of each container selected as in **A.4.2** and the material so obtained shall be mixed to form a composite sample of not less than 300 ml.

A.6 REFERENCE SAMPLE

If a reference sample is required, the required individual and composite samples, for reference shall be obtained as follows :

a) Size of the individual sample shall be 180 ml and each individual sample so obtained shall be divided into three equal parts. One set of individual samples shall be marked for the purchaser, another for the supplier and the third set to be used as a reference sample.

b) The size of the composite sample shall be 900 ml and the composite sample so obtained shall be divided into three equal parts. One set of composite samples shall be marked for the purchaser another for the supplier and the third to be used as a reference sample.

A.7 NUMBER OF TEST

A.7.1 Each container selected as in A.4.2 shall be examined for packaging and marking requirements (This may be done at a place of inspection).

A.7.2 Test for determination of relative density and ethanol content shall be carried out on the each individual samples.

A.7.3 Test for determination of other characteristics specified in Table 1 shall be carried out on the composite sample.

APPENDIX B

DETERMINATION OF RELATIVE DENSITY

B.1 APPARATUS

B.1.1 *Pycnometer*, of 25 ml capacity with a ground-glass stopper having a capillary opening, a chamber to provide for expansion up to room temperature and a cap to prevent evaporation.

B.1.2 *Water bath*, capable of maintaining a temperature of 20 °C during the test.

B.2 PROCEDURE

B.2.1 Clean the pycnometer by filling it with a saturated solution of chromic acid in sulphuric acid, allowing it to stand for a few hours, emptying and rinsing well with distilled water. Fill the pycnometer with freshly boiled distilled water. Place it in the water bath maintained at 20 °C until the pycnometer and its contents are at a constant volume at 20 °C. After immersion in the bath for at least 30 min, adjust the level of liquid to the proper point on the pycnometer, put the stopper in place, remove from the bath, wipe dry, and weigh.

B.2.2 Empty the pycnometer, rinse successively with alcohol and ether, remove the ether vapour, immerse in the bath, and bring to 20 °C as was done before. After immersion at 20 °C for at least 30 min, put the stopper in place, remove from the bath, wipe dry, and weigh.

B.2.3 Fill the pycnometer with the material, immerse in the bath and bring to 20 °C as was done before. After immersion at 20 °C for at least 30 min, adjust the liquid level, put the stopper in place, remove from the bath, wipe dry, and weigh.

B.3 CALCULATION

$$\text{Relative density at } 20\ ^{\circ}\text{C}/20\ ^{\circ}\text{C} = \frac{m_1 - m_2}{m_3 - m_2}$$

where,

m_1 = mass, in g, of the pycnometer with the material at 20 °C;

m_2 = mass, in g, of the pycnometer; and

m_3 = mass, in g, of the pycnometer with water at 20 °C.

APPENDIX C

DETERMINATION OF ETHANOL CONTENT

C.1 PROCEDURE

Determine the relative density of the material at 20 °C/20 °C according to the method prescribed in Appendix B and find out the strength of alcohol by mass and by volume, and also its proof strength from Table 3.

TABLE 3 - Relation between relative density, percentage of alcohol by mass and by volume, and proof strength

Relative density in air at 20 °C/20 °C (1)	Percentage of ethanol		Degrees overproof (4)
	By mass (2)	By volume at 20 °C (3)	
0.8330	84.77	89.30	56.35
0.8329	84.81	89.33	56.60
0.8328	84.85	89.37	56.70
0.8327	84.89	89.40	56.75
0.8326	84.93	89.43	56.80
0.8325	84.97	89.46	56.85
0.8324	85.01	89.49	56.90
0.8323	85.05	89.52	56.95
0.8322	85.09	89.55	57.00
0.8321	85.13	89.58	57.05
0.8320	85.17	89.61	57.10
0.8319	85.21	89.64	57.15
0.8318	85.24	89.67	57.20
0.8317	85.28	89.70	57.25
0.8316	85.32	89.73	57.30
0.8315	85.36	89.76	57.35
0.8314	85.40	89.79	57.40
0.8313	85.44	89.82	57.45
0.8312	85.48	89.85	57.50
0.8311	85.52	89.88	57.60

TABLE *(Contd.)*

Relative density in air at 20 °C/20 °C (1)	Percentage of ethanol		Degrees overproof (4)
	By mass (2)	By volume at 20 °C (3)	
0.8310	85.56	89.91	57.65
0.8309	85.60	89.94	57.70
0.8308	85.64	89.97	57.75
0.8307	85.68	90.00	57.80
0.8306	85.71	90.04	57.85
0.8305	85.75	90.07	57.90
0.8304	85.79	90.10	57.95
0.8303	85.83	90.13	58.00
0.8302	85.87	90.16	58.05
0.8301	85.91	90.19	58.10
0.8300	85.95	90.22	58.15
0.8299	85.99	90.25	58.20
0.8298	86.03	90.28	58.25
0.8297	86.07	90.31	58.30
0.8296	86.10	90.34	58.40
0.8295	86.14	90.37	58.45
0.8294	86.18	90.40	58.50
0.8293	86.22	90.43	58.55
0.8292	86.26	90.46	58.60
0.8291	86.30	90.49	58.65
0.8290	86.34	90.52	58.70
0.8289	86.38	90.55	58.75
0.8288	86.42	90.58	58.80
0.8287	86.46	90.61	58.85
0.8286	86.49	90.64	58.90
0.8285	86.53	90.67	58.95
0.8284	86.57	90.70	59.00
0.8283	86.61	90.73	59.05
0.8282	86.65	90.75	50.10
0.8281	86.69	90.78	50.15

NOTE – The percentage of proof spirit may be obtained by adding 100 to the number of degrees overproof.

APPENDIX D

TEST FOR ACIDITY OR ALKALINITY

D.1 PROCEDURE

D.1.1 Acidity

D.1.1.1 Take 20 ml of the material and titrate with 0.1N sodium hydroxide using phenolphthalein as the indicator.

D.1.1.2 The material shall be taken as satisfying the requirements of this test if the volume of 0.1N sodium hydroxide required to give a pink colour does not exceed 0.2 ml.

D.1.2 Alkalinity

D.1.2.1 Take 20 ml of the material and titrate with 0.1N hydrochloric acid using methyl red as the indicator.

D.1.2.2 The material shall be taken as satisfying the requirements of this test if the volume of 0.1N hydrochloric acid required to give a red colour does not exceed 0.1 ml.

APPENDIX E

TEST FOR ALDEHYDES AND KETONES

E.1 REAGENTS

E.1.1 *Metaphenylenediamine hydrochloride*

E.1.2 *Aldehyde - free alcohol*

Re-distil rectified spirit over solid caustic soda or caustic potash, add 2 g to 3 g of metaphenylenediamine hydrochloride per litre of rectified spirit, digest at ordinary temperature for several days or under a reflux condenser on a steam bath, for several hours and distil slowly, rejecting the first 100 ml and the last 200 ml of the distillate.

E.1.3 *Bromophenol blue solution*

Dissolve 0.1 g of bromophenol blue in 1.5 ml of 0.1N sodium hydroxide solution and dilute with water to 250 ml.

E.1.4 *Hydroxyammonium chloride solution*

Dissolve 1 g of hydroxyammonium chloride in 50 ml of water and add 50 ml of aldehyde-free alcohol 1 ml of bromophenol blue solution and 0.1N sodium hydroxide solution until the solution becomes green.

E.2 PROCEDURE

E.2.1 Heat 100 ml of hydroxy ammonium chloride solution in a loosely stoppered flask on a water bath for thirty minutes, cool and if necessary, and sufficient 0.1N sodium hydroxide to restore the green colour. To 50 ml of this solution add 25 ml of the material and heat on a water bath for ten minutes in a loosely stoppered flask. Cool, transfer to a Nessler cylinder and titrate with 0.05N sodium hydroxide until the colour matches that of the remainder of the hydroxy - ammonium chloride solution contained in a similar cylinder, both solutions being viewed down the axis of the cylinder.

E.2.2 The material shall be taken as satisfying the requirements of this test if not more than 0.9 ml of 0.05N sodium hydroxide is required for the titration.

APPENDIX F

TEST FOR FUSEL OIL

F.1 PROCEDURE

F.1.1 Allow 25 ml of the material to evaporate spontaneously in a porcelain dish, protected from dust until the surface of the dish is barely moist. Observe if foreign odour is perceptible.

F.1.2 Add 1 ml of sulphuric acid.

F.1.3 The material shall be taken as satisfying the requirements of this test if no foreign odour is perceptible and no red or brown colour is produced on treatment with sulphuric acid.

APPENDIX G

TEST FOR KETONES, ISOPROPYL ALCOHOL AND T-BUTYL ALCOHOL

G.1 REAGENTS

G.1.1 *Mercuric sulphate solution*, mix 5 g of yellow mercuric oxide with 40 ml of water and while stirring, add 20 ml of sulphuric acid; add 40 ml of water and stir until completely dissolved.

G.2 PROCEDURE

G.2.1 Dilute to approximately 20 per cent (V/V) of ethyl alcohol with water and heat 5 ml of the dilution with 10 ml of mercuric sulphate solution for three minutes on a water bath.

G.2.2 The material shall be taken as satisfying the requirements of this test, if no precipitate appears.

APPENDIX H

TEST FOR METHYL ALCOHOL

H.1 REAGENTS

H.1.1 *Potassium permanganate and phosphoric acid solution*

Dissolve 3 g of potassium permanganate in a mixture of 15 ml of phosphoric acid containing approximately 89 per cent (*m/m*) of phosphoric acid (H_3PO_4) and 70 ml of water; add sufficient water to produce 100 ml.

H.1.2 *Oxalic acid and sulphuric acid solution*

A 5 per cent (*m/v*) solution of oxalic acid in a cooled mixture of equal volumes of sulphuric acid and water.

H.1.3 *Magenta solution,* decolourised

Dissolve 1 g of basic magenta (roseniline hydrochloride of fuchaine) in 600 ml of water and cool in ice; add 20 g of sodium sulphite dissolved in 100 ml of water, cool in ice and add, slowly and with constant stirring 10 ml of hydrochloric acid; dilute to 1 000 ml.

H.1.3.1 If the resulting solution is turbid, it should be filtered and if brown in colour, it should be shaken with sufficient animal charcoal (0.2 g to 0.3 g) to render it colourless and then filtered immediately. Occasionally it is necessary to add 2 ml or 3 ml of hydrochloric acid, followed by shaking, to remove a little residual pink colour. The solution resulting from any of the foregoing modifications should be allowed to stand overnight before use. Decolourised Magenta solution should be protected from light.

H.2 PROCEDURE

H.2.1 Dilute with water to approximately 10 per cent (*v/v*) of ethyl alcohol and to 5 ml add 2.0 ml of potassium permanganate and phosphoric acid solution. Set aside for ten minutes and add 2.0 ml of oxalic acid and sulphuric acid solution. To the colourless solution add 5 ml of decolourised Magenta solution, allow to stand at a temperature between 15 °C and 30 °C and examine after 30 minutes.

H.2.2 The material shall be taken as satisfying the requirements of this test if no colour is produced.

APPENDIX J

TEST FOR OILY OR RESINOUS SUBSTANCES

J.1 PROCEDURE

J.1.1 Dilute 5 ml of the material to 100 ml with water in a cylinder.

J.1.2 The material shall be taken as satisfying the requirements of this test if the solution remains clear when examined against a black background.

APPENDIX K

TEST FOR NON VOLATILE MATTER

K.1 PROCEDURE

K.1.1 Evaporate 100 ml of the material and dry at 105 ^{o}C.

K.1.2 The material shall be taken as satisfying the requirements of this test if not more than 0.005 per cent (*m/V*) of residue is left.

APPENDIX L

TEST FOR REDUCING SUBSTANCES

L.1 PROCEDURE

L.1.1 To 20 ml of the material add 1 ml of 0.01N potassium permanganate. Allow the solution to stand at 20 ^{o}C for 10 minutes protected from light.

L.1.2 The material shall be taken as satisfying the requirements of this test if the colour of the solution is not completely discharged.

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DRAFT AMENDMENT NO. 1 APPROVED ON 2001-10-29
TO SLS 351 : 1983

SRI LANKA STANDARD SPECIFICATION FOR RECTIFIED SPIRIT

Clause 2 REFERENCES

Insert the following as line 1

AOAC Volume 1, Drugs Part 1 - Official Methods of Analysis of the Association of Official Analytical Chemists

Clause 7 METHODS OF TEST

Delete line 1 and substitute the following

7.1 Tests shall be carried out as prescribed in Appendices B to L and AOAC - Official Methods of Analysis of the Association of Official Analytical Chemists (1999) 16th edition Vol. 1, Drugs Part 1, 18.2.02 Acetone and alcohol in drugs 973 : 69.

Appendix C DETERMINATION OF ETHANOL CONTENT

C.1 Delete 'PROCEDURE' and substitute **'METHOD' 1 - RELATIVE DENSITY METHOD**

Insert C.1.1 **Procedure**

Insert C.2 **METHOD 2 – GAS CHROMATOGRAPHIC METHOD** at the end of Table 3

Insert C.2.1 **Procedure**

Carry out the determination as given in AOAC, Vol. 1, Drugs Part 1.

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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.*

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SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

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