SRI LANKA STANDARD 1027 : 1994

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SPECIFICATION FOR
OXYGEN, TECHNICAL GRADE

SRI LANKA STANDARDS INSTITUTION

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SLS 1027 : 1994

Gr. 12

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SPECIFICATION FOR OXYGEN, TECHNICAL GRADE

FOREWORD

This standard was approved by the Sectoral Committee on Chemicals 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 1994-12-22 .

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**.

The safety considerations for handling of oxygen is given in Appendix **G** as a guidance.

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 an 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 specification.

In the preparation of this standard, the valuable assistance derived from the following publications is gratefully acknowledged:

BS 4364 : 1993 Industrial oxygen
SS 153 : 1976 Industrial oxygen.

1 SCOPE

1.1 This specification prescribes the requirements and methods of test for oxygen in gaseous and liquid forms, intended for use in industrial applications.

1.2 It does not apply to material intended for medical use and for use in aircrafts for breathing purposes.

2 REFERENCES

CS 102	Presentation of numerical values.
SLS 428	Random sampling methods
SLS 875	Identification markings of the contents of industrial gas cylinders.

3 REQUIREMENTS

The product shall comply with the requirements specified in Table **1** when tested by the methods prescribed in Column **4** of the table.

TABLE 1 - Requirements for oxygen

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Oxygen, as O_2 , per cent by volume, min.	99.5	Appendix C*
ii)	Carbon dioxide, as CO_2 , ppm (V/V) , max.	10	Appendix D*
iii)	Total hydrocarbons, as CO_2 or methane, ppm (V/V) , max.	50	Appendix E*
iv)	Water content** as ppm, (V/V), for oxygen in :		
	a) gaseous form, max.	30	Appendix F*
	b) liquid form, max.	2	

* One of the two methods given , may be used.

** As product is removed from a fully charged container, the contribution of water from the container wall may increase.

4 PACKAGING AND MARKING

4.1 Oxygen in gaseous form

4.1.1 Oxygen in gaseous form shall be supplied in properly maintained, clean cylinders painted externally black in colour.

4.1.2 Each cylinder shall be legibly and indelibly marked or labelled with the following information:

a) Name of the product and its chemical formula
b) Name and address of the manufacturer and /or supplier of the gas (including the country of origin) ;
c) Net volume, in m^3, at standard temperature and pressure ;
d) Brand name, if any ;
e) Trade mark, if any ; and
f) Batch identification mark.

4.2 Oxygen in liquid form

4.2.1 Oxygen in liquid form shall be supplied in properly maintained, clean, double walled vacuum insulated containers.

4.2.2 Each container shall be legibly and indelibly marked or labelled with the following information;

a) Name of the product and its chemical formula;
b) Name and address of the manufacturer ; and
c) Batch identification mark.

4.3 The method of marking on cylinders containing oxygen in gaseous form shall comply with the requirements specified in **SLS 875**.

NOTE – Attention is drawn to the certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of this standard.

5 METHODS OF TEST

5.1 Tests for the requirements given in 3 shall be carried out as prescribed in Appendices **B** to **F** of this specification. The Appendix **B** gives the method of treatment of liquid samples.

5.2 Unless otherwise specified, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

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, and appropriate scheme of sampling and inspection should be adopted.

A.1 LOT

In any consignment, all the cylinders/ containers of the same size and containing product of the same form(ie. gaseous or liquid) belonging to one batch of manufacture or supply should constitute a lot.

A.2 SCALE OF SAMPLING

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

A.2.2 The number of cylinders/containers to be selected from a lot shall be in accordance with Table **2**.

TABLE 2 - Scale of sampling

Number of cylinders/ containers in the lot (1)	Number of cylinders/ containers to be selected (2)	Size of sub sample (3)
Up to 150	8	3
151 to 280	10	4
281 to 500	12	5
501 to 1200	15	6
1201 and above	20	8

A.2.3 The cylinders/ containers should 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 cylinder/container selected as in **A.2.2** shall be inspected for packaging and marking requirements.

A.3.2 Each cylinder/container in the sub sample selected in accordance with Column **3** of Table **2** shall be tested for the requirements given in clause **3**.

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 cylinder/container inspected as in **A.3.1** satisfies the relevant requirements.

A.4.2 Each cylinder/container tested as in **A.3.2** satisfies the relevant requirements.

APPENDIX B

METHOD OF TREATMENT OF LIQUID SAMPLES

B.1 OUTLINE OF METHOD

The liquid is forced under pressure into an evaporating coil. By use of small bore tubing complete evaporation of the liquid is ensured, and hence the gas produced has the same composition as the liquid.

CAUTION

Liquid oxygen requires careful handling and all flames should be extinguished in the vicinity during sampling operations. Care could be taken to avoid spilling it on to clothing. Liquid oxygen boils at - 183 ^{0}C so precautions should be taken to prevent it coming into contact with the skin and the eyes.

B.2 APPARATUS

As shown in Figure **1**, comprising of :

B.2.1 Mercury lute, **A**, arranged in such a way so that the head of mercury lute can easily be adjusted.

B.2.2 Metal Dewar flask, **B**, designed to withstand an internal pressure of 70 kPa, having a capacity of about 2.5 litres, fitted with a screw-on brass cap carrying a bursting disk set at 35 kPa and two connections of drawn metal tubing having an internal diameter of 1.5 mm.

B.2.3 Evaporator, C consisting of a drawn metal tubing of 500 mm in length and 1.5 mm internal diameter, formed into a spiral and immersed in water at 50 ^{0}C to 60 ^{0}C .

B.3 PROCEDURE

Cool the Dewar flask (**B.2.2**) , by introducing a small quantity of the liquid oxygen and after a few minutes discard the liquid. Immediately introduce more liquid oxygen until the flask is about two-thirds full. With stopcock **T$_1$** (see Figure **1**) open, screw on the brass cap and make the connections as shown in Figure **1**. Control the rate of gas flow to the analysis apparatus by adjusting the control valve and the head of mercury in the lute.

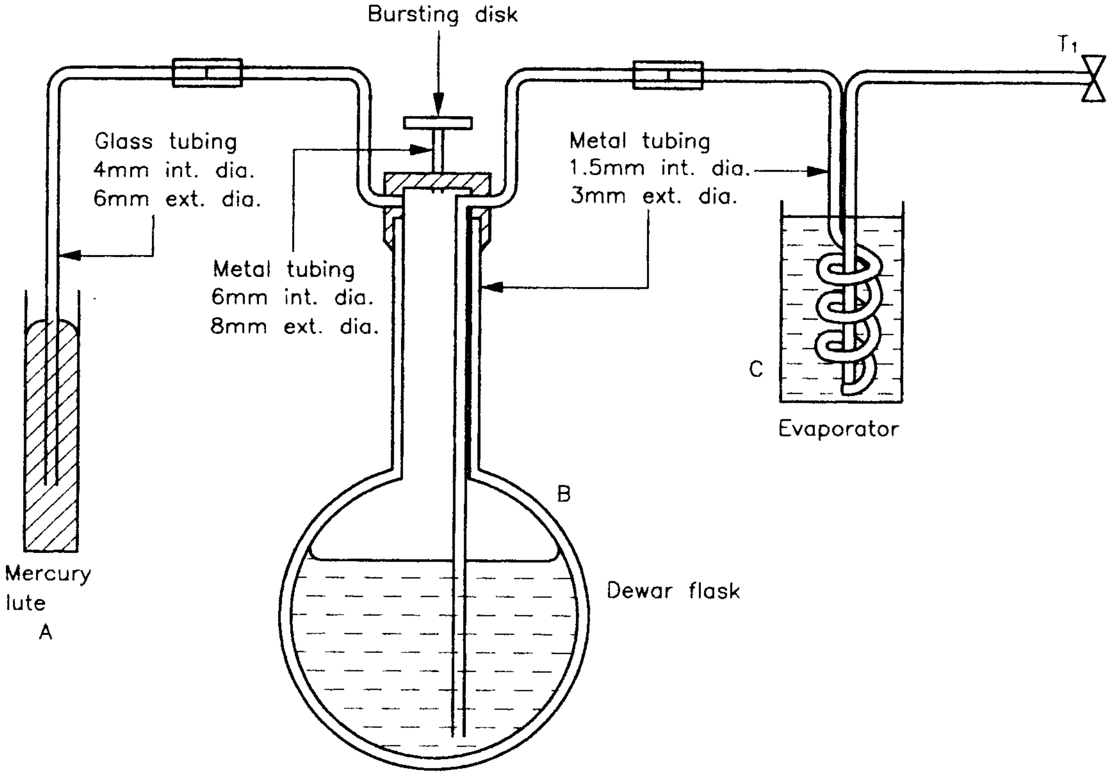


FIGURE 1 - Apparatus for treatment of liquid samples

APPENDIX C
DETERMINATION OF OXYGEN CONTENT

C.1 METHOD I

C.1.1 Outline of method

The oxygen content is determined by measuring the volume of a sample before and after absorption of the oxygen by copper, in the presence of ammoniacal ammonium chloride solution.

C.1.2 Apparatus

As shown in Figure **2**, comprising of :

C.1.2.1 *Levelling bottle , A.*

C.1.2.2 *Graduated burette and stopcock,* B, as shown in Figure **3**, fitted with a two-way burette stopcock, T_1 .

NOTE - In graduating the burette the lowest point of the liquid meniscus shall be adjusted to the top edge of the graduation mark. The meniscus can be clearly defined by folding a strip of black paper round the vessel, the top edge of the paper being horizontal and not more than 1 mm below the mark on which the setting is to be made. The meniscus, so shaded, should be viewed against a white background. When reading the burette, sufficient time shall be allowed for all the liquid to drain from the walls.

The same technique shall be used in making all readings of the burette.

C.1.2.3 *Container for copper wire* , **C**, as shown in Figure **4**.

C.1.2.4 *Reservoir,* **D**.

C.1.2.5 *Gas inlet*, **E**, with three-way stucpcock, $\mathbf{T_2}$.

C.1.2.6 *Lute*, **F,** sealed with a head of about 50 mm of water.

C.1.2.7 *Glass tube*, **G,** outside diameter of 6 mm and a length of 260 mm.

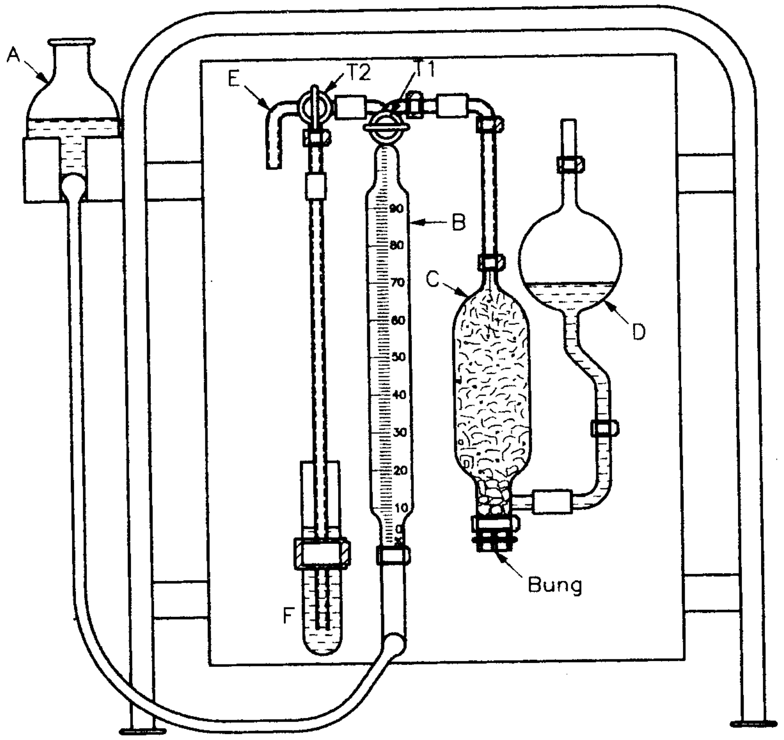
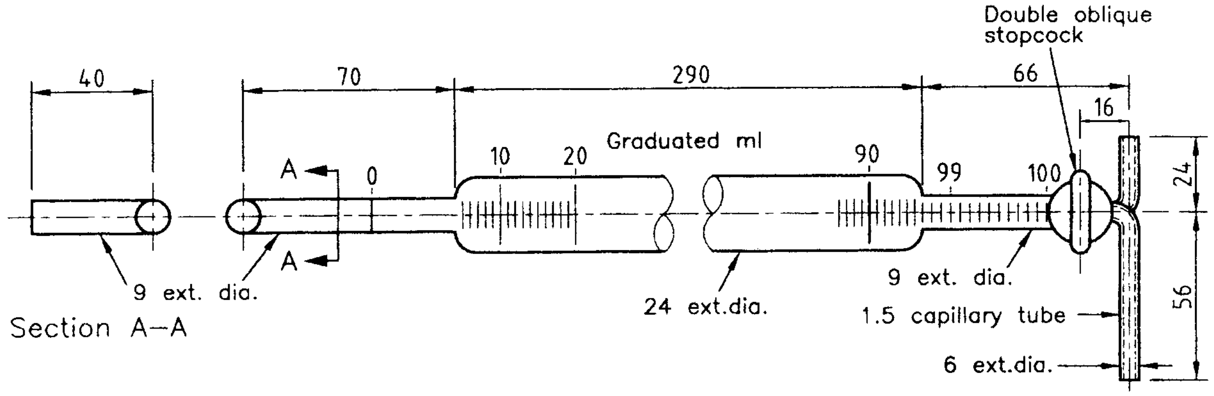


FIGURE 2 - Appartus for the determination of oxygen content

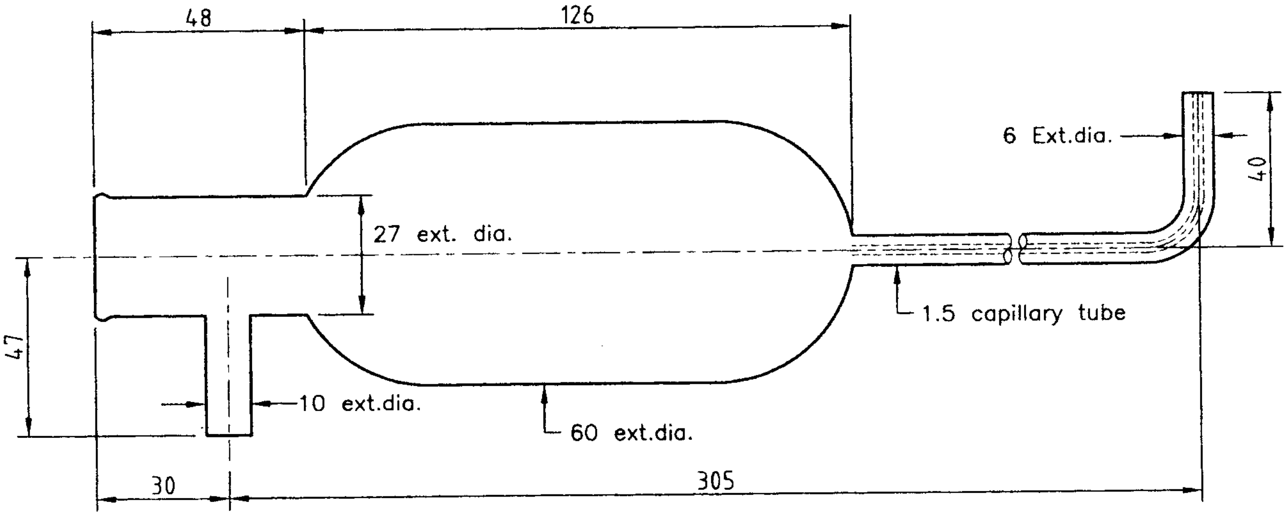


Dimensions in millimetres

Limits ± 1 mm

Material – glass

FIGURE 3 - Graduated burette for the determination of oxygen content



Dimensions in millimetres

Limits ± 1 mm

Material – glass

FIGURE 4 - Copper wire container for the determination of oxygen content

C.1.3 Reagent and material

C.1.3.1 *Ammoniacal ammonium chloride solution.*

Dissolve 550 g of ammonium chloride in 1 250 ml of water and add 750 ml of concentrated ammonia solution, (rel. den. = 0.88).

NOTES -

1. Freshly prepard solution in contact with the copper spirals is initially very pale in colour, and the absorption of oxygen in the determination may be incomplete, leading to errors in test results. Before it is used for a determination, it should be aged to a deep blue colour by carrying out several dummy operations.

*2. The solution loses its efficiency with use and the apparatus may be recharged by the method given in **Note 1** at the end of this Appendix.*

C.1.3.2 *Copper wire spirals*, 200-mm lengths of copper wire of diameter 0.9 mm, wound into open spirals of about 6-mm diameter and about 20 mm long.

C.1.4 Procedure

C.1.4.1 *Preparation of the apparatus*

Fill in the container **(C.1.2.3)** , with the copper spirals **(C.1.3.2)** and connect to the rest of the apparatus as shown in Figure **2**. Lubricate the stopcocks with the minimum amount of a suitable vacuum stopcock grease (silicone greases are unsuitable).

Lower the reservoir **D**, **(C.1.2.4)** to the bench and connect the lute **(C.1.2.6)** and invert the rest of the apparatus with stopcock **T**$_1$ closed. Fill the container **C**, **(C.1.2.3)** with the solution **(C.1.3.1)** and close it with a rubber bung. Return the apparatus to the upright position. Fill leveling bottle **A** **(C.1.2.1)** with the solution. Operate stopcocks **T**$_1$ and **T**$_2$ so that burette **B**, **(C.1.2.2)** is open to the atmosphere, and by raising **A**, completely fill burette **B** with the solution **(C.1.3)** . Operate stopcock **T**$_1$ so that **B** is in communication with **C**, and keeping **A** raised , allow the solution to pass through **C** until reservoir **D** is about one third full. Close stopcock **T**$_2$. With **A** still raised, fill the capillary between stopcocks **T**$_1$ and **T**$_2$ with the solution, close stopcock **T**$_2$ and return **A** to its resting position. Add more solution if necessary, until **A** is half full.

C.1.4.2 *Determination*

C.1.4.2.1 Connect the sample gas supply, either from a cylinder or from the liquid evaporator to **E**, **(C.1.2.5)** and with stopcock **T**$_2$ closed, purge the tubing by allowing the gas to escape through the water lute **F** **(C.1.2.6)**.

C.1.4.2.2 With the gas still bubbling through **F**, operate stopcock **T$_1$** to connect **B** to **E** and lower the levelling bottle **A** to maintain the same liquid level in **A** and **B**, until the level in **B** reaches to zero mark.

C.1.4.2.3 Raise **A** and expel all the gas from **B** through the lute. Lower **A** to refill **B**, as in **C.1.4.2.2** Close stopcocks **T$_1$** and **T$_2$** .

C.1.4.2.4 Operate stopcock **T$_1$** to connect **B** to **C**, and by raising A, pass the gas sample from **B** to **C**. Close stopcock **T$_2$** and rock the apparatus gently until absorption appears to be complete. Lower **A** and operate stopcock **T$_1$** to connect **C** to **B** so as to pass any unabsorbed gas into **B**, taking care that the connection capillary is filled with solution. Close stopcock **T$_1$** . Raise **A** until the liquid levels in **A** and **B** are the same and read the volume of residual gas (see note in **C.1.2.2** regarding reading the meniscus).

C.1.4.2.5 Repeat the operation in **C.1.4.2.4** until a constant reading is obtained.

NOTES

1. When the absorption of oxygen becomes slow or deposits form in the ammoniacal ammonium chloride solution, replace approximately half the solution in the apparatus by the following sequence of operations :

- *a) Operate stopcock **T$_1$** , so that **B** is connected to **C** and raise **A** until **B** is full of liquid.*
- *b) Close stopcock **T$_1$** , remove the bung from **C** and allow the solution in **C** and **D** to run to waste.*
- *c) Replace the bung in C.*
- *d) Operate stopcock **T$_2$** to connect **B** to **C** and by raising **A** pass the solution from **B** to **C** until the latter is half full.*
- *e) Add fresh solution to **A** , and lower **A** to draw gas from **C** into **B**, taking care not to draw in air through **D** . Operate stopcocks **T$_1$** and **T$_2$** to connect **B** to atmosphere and expel the air from **B** by raising **A**.*
- *f) Repeat operations (d) and (e) until all levels are correct.*

*2. It is essential to keep **C** completely full of copper spirals as any fall in the level of the copper will slow down the rate of absorption of oxygen. To recharge with additional spirals, close the top of **D** with bung, lower **A** to the bench and disconnect the lute, invert the apparatus and remove the bung from **C**. Add more spirals until **C** is full and then reassemble the apparatus.*

C.2 METHOD II

C.2.1 Principle

The method uses the principle of paramagnetic susceptibility, a physical property which is considerably greater in oxygen than in other common gases. Oxygen molecules are more strongly attracted by a magnetic field than are molecules of other gases, most of which are slightly diamagnetic (repelled by a magnetic field).

Magneto-dynamic oxygen analysers are based on Faraday's method of determining the magnetic susceptibility of a gas by measuring the force developed by a strong non-uniform magnetic field on a diamagnetic test body suspended in the sample gas. The test body of all measuring cells in paramagnetic oxygen analysers consists of two nitrogen-filled quartz spheres arranged in the form of a dumb-bell, as shown in Figure **5a** single turn of fine platinum wire (the feedback coil) is secured in place around the dumb-bell. A rugged, taut band platinum ribbon suspension attached to the mid-point of the dumb bell, positions the dumb-bell in the strong non uniform magnetic field existing between the specially shaped pole pieces of the permanent magnetic structure (see Figure **5b**) .

C.2.2 Apparatus

A variety of analytical equipment suppliers provide simple, ready to run portable units for this purpose, which may be powered either by battery or mains electricity.

Figure **5c** illustrates the configuration of a typical unit designed for this purpose.

C.2.3 Calibration

Calibration of these units shall be carried out using a calibration standard that is assured by analysis traceable gravimetrically of purity greater than 99.95% oxygen and the manufacturer's instructions.

C.2.4 Procedure

After calibrating the instrument, introduce the sample of the product in accordance with the manufacturer's instruction, record the oxygen content that is displayed by the instrument.

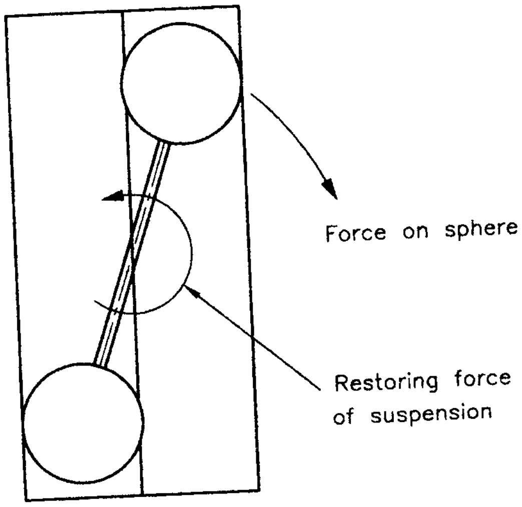


FIGURE 5a - Dumb-bell system : forces with the cell

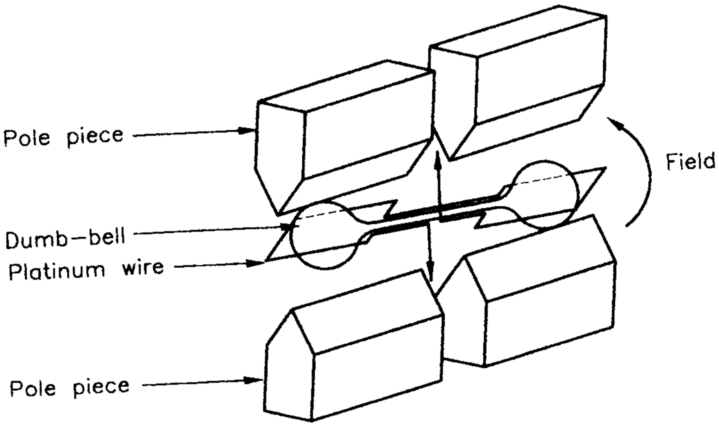


FIGURE 5b - Measuring cell configuration

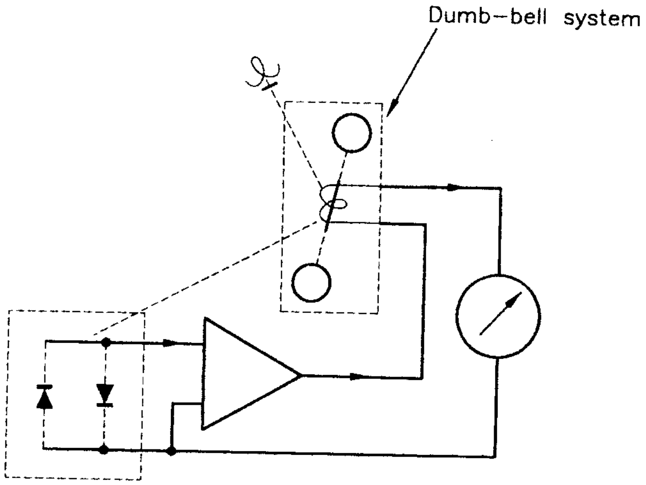


FIGURE 5c - Typical analyser configuration (electronic)

APPENDIX D
DETERMINATION OF CARBON DIOXIDE CONTENT

D.1 METHOD 1

D.1.1 Outline of method

A measured volume of gas is passed through an absorber containing barium hydroxide and the carbon dioxide absorbed is determined by titrating the excess barium hydroxide with standard hydrochloric acid solution.

D.1.2 Apparatus

As shown in Figure 6, comprising of;

D.1.2.1 *Mercury lute , A,* with, a head of mercury of approximately 100 mm.

D.1.2.2 *Regulating valve , B.*

D.1.2.3 *Spiral absorber, C,* as shown in Figure 7.

D.1.2.4 *Integrating wet gas meter, D ,* 2.5 litres per revolution.

NOTE - The spiral absorber is efficient at a flow rate of 5 litres per hour ; higher rates must be avoided as this tends to lift all the liquid into the upper portion of the absorber.

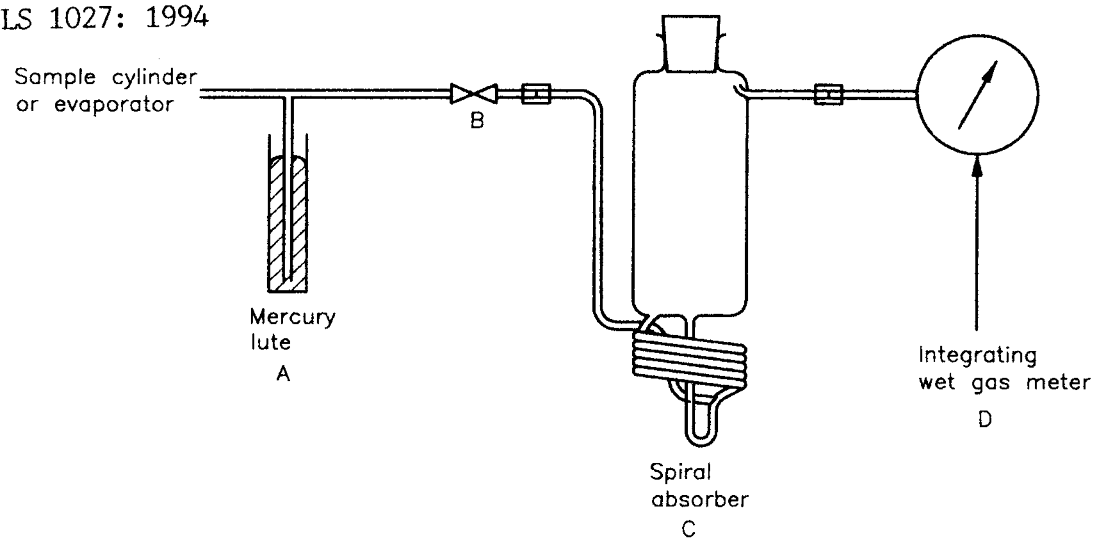


FIGURE 6 - Apparatus for the determination of carbon dioxide content

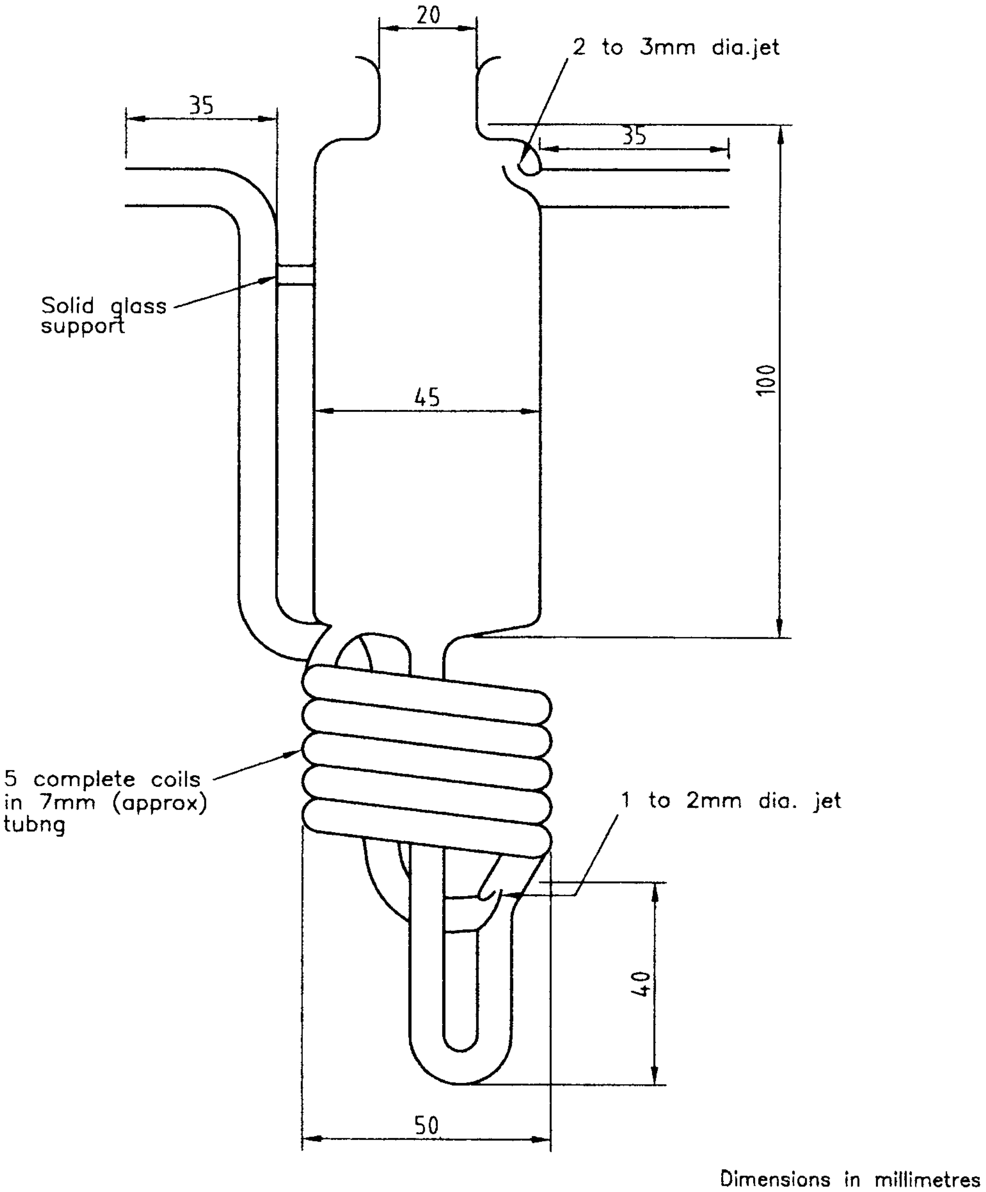


FIGURE 7 - Spiral absorber

D.1.3 Reagents

Water freed from carbon dioxide by passing through it, a stream of carbon dioxide free nitrogen, for at least 30 minutes or by other suitable means, shall be used throughout.

D.1.3.1 *Nitrogen,* carbon dioxide free

D.1.3.2 *Thymol blue indicator solution,* 0.4 g/1.

D.1.3.3 *Bariium hydroxide,* approximately 0.01 mol/1 solution.
This solution must be protected from atmospheric carbon dioxide, for instance, by storing under nitrogen.

D.1.3.4 *Hydrochloric acid, 0.02 mol/1 standard solution.*

D.1.4 Procedure

D.1.4.1 *Preparation of the absorber*

To ensure that the absorber and the water are free from acidic impurities, place 10 ml of the water in the absorber, pass the nitrogen (**D.1.3.1**) through the absorber at 5 litres per hour for 5 minutes and then add two to three drops of the indicator solution (**D.1.3.2**) . On the addition of one drop of the barium solution (**D.1.3.3**) the colour shall turn to violet-blue.

NOTE *– If the solution does not turn violet-blue, thoroughly clean the absorber and test again.*

D.1.4.2 *Standardization*

Maintaining the rate of nitrogen flow at 5 litres per hour, carefully add from a burette, 20.0 ml of the barium hydroxide (**D.1.3.3**) to the solution in the absorber prepared as described in **D.1.4.1.** Rinse down the mouth of the absorber with water and then titrate the contents with the hydrochloric acid solution (**D.1.3.4**) until the colour just changes.

D.1.4.3 *Determination*

Adjust the flow rate of the sample gas flow rate, either from a sample cylinder or from the liquid evaporator (see Appendix **B**), to 5 litres per hour and thoroughly purge the connections until all air is removed.

Empty the absorber and recharge with 20 ml of the barium hydroxide solution as described above and insert a rubber bung into its mouth. Immediately connect into the analysis train, as shown in Figure **5**.

NOTE *– Care must be taken to prevent the pick up of atmospheric carbon dioxide by the barium hydroxide solution by preventing any ingress of air into the absorber.*

Regulate the gas flow through the absorber at 5 litres per hour. Record the initial meter reading and continue passing the sample gas for 16 hours. At the end of this time read the meter again, remove the absorber from the train and wash down any liquid in its side arms with water. Titrate the contents with the hydrochloric acid solution **(D.1.3.4)** , while maintaining a flow of the nitrogen through the absorber.

Determine the mean temperature and barometric pressure during the 16 hours of sample flow by any suitable means.

D.1.5 Calculation

Carbon dioxide content, parts per million by volume at 27°C and

$$101.3 \text{ kPa pressure} = \frac{V_1 - V_2}{V_3} \times 246 \times \frac{(273 + t)}{300} \times \frac{101.3}{P}$$

Where,

V_1 is the volume, in millilitres, of 0.02 mol/l hydrochloric acid used in the standardization ;
V_2 is the volume, in millilitres, of 0.02 mol/l hydrochloric acid used in the determination;
V_3 is the volume, in lilitres, of sample passed ;
t is the mean temperature, in °C, of the gas passed through the gas meter; and
P is the mean barometric pressure, in kPa, during the determination less the vapour pressure of water, in kPa, at temperature t.

***NOTE** – The factor 246 used in the above calculation is derived as follows :*

1 ml of 0.01 mol/l barium hydroxide

$$\text{solution} = \frac{0.01 \times 44}{1000} \text{ g of carbon dioxide}$$

Since at 0 °C and 101.3 kPa, 44 g of carbon dioxide occupy 22.4 litres ;

1 ml of 0.01 mol/l barium hydroxide solution

$$= \left(\frac{0.01 \times 44}{1000} \times \frac{22.4}{44}\right) \text{ litres of}$$

carbon dioxide at standard temperature and pressure.

Therefore, at 27 °C and 101.3 kPa :
1 ml of 0.01 mol/l barium hydroxide solution

$$= \frac{0.01 \times 44}{1000} \times \frac{22.4}{44} \times \frac{300}{273} \text{ litres of carbon dioxide}$$

$$= 246 \times 10^{-6} \text{ litres of carbon dioxide.}$$

D.2 METHOD II

D.2.1 Principle

The method uses the principle of infra-red absorption by different molecules at specific wavelengths in the infra-red spectrum.

D.2.2 Apparatus

D.2.2.1 Scanning infra-red spectrometer, fitted with a gas cell of 10 m minimum optical path length.

D.2.3 Calibration

Calibration of these units shall be carried out using a calibration mixture.

D.2.4 Procedure

After calibrating the instrument, introduce the sample of the product in accordance with the manufacturer's instructions, and calculate carbon dioxide content.

APPENDIX E
DETERMINATION OF TOTAL HYDROCARBON CONTENT

E.1 METHOD 1

E.1.1 Outline of method

The gas is freed from carbon dioxide and then any hydrocarbons present are oxidized at 600 °C to 650 °C in the presence of copper oxide catalyst. The carbon dioxide formed is absorbed by barium hydroxide solution, the excess barium hydroxide being determined by titration with standard hydrochloric acid solution.

E.1.2 APPARATUS

As shown in Figure 8, comprising of :

E.1.2.1 *U – tube, A,* filled with soda lime **(E.1.3.1)**

E.1.2.2 *Silica furnace tube,* **B,** at least 150 mm long, containing a plug of Arneil catalyst.

E.1.2.3 *Electric furnace,* **C,** maintained at 600 °C to 650 °C.

E.1.2.4 *Spiral absorber,* **D,** as shown in Figure 7, in Appendix **C.**

E.1.2.5 *Integrating wet gas meter,* **E,** 2.5 litres per revolution.

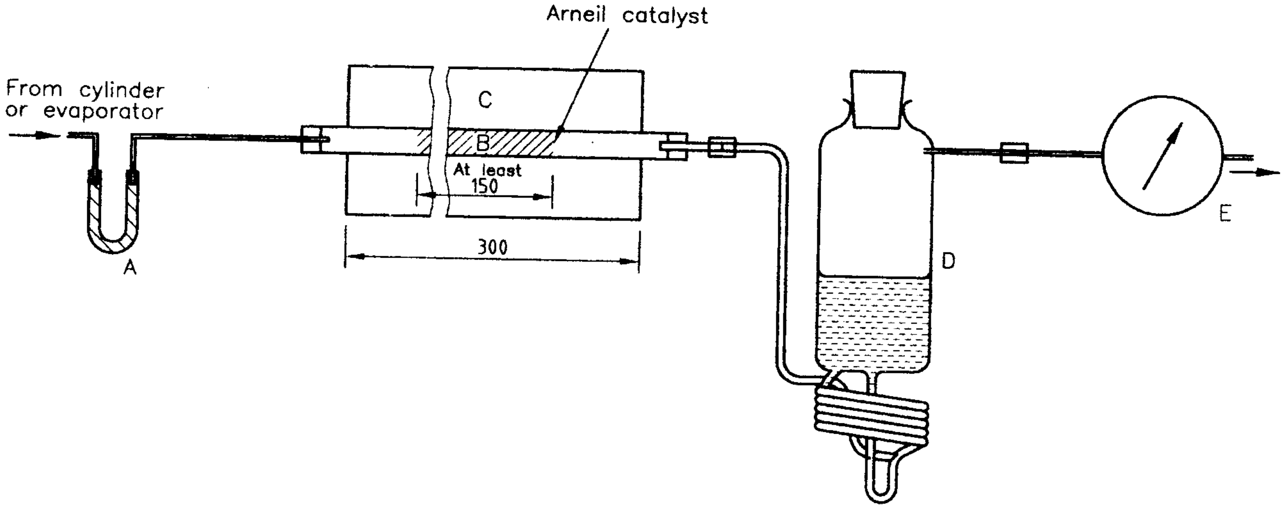


FIGURE 8 – Apparatus for the determination of total hydrocarbon content

E.1.3 Reagents

Water freed from carbon dioxide by passing a stream of carbon dioxide free nitrogen, for at least 30 minutes or by other suitable means, shall be used throughout.

E.1.3.1 *Soda lime,* passing through a sieve of 1.20 mm nominal aperture width and retaining on a sieve of 710 µm nominal aperture width.

E.1.3.2 *Nitrogen*, carbon dioxide free

E.1.3.3 *Thymol blue indicator 0.4 g/l solution.*

E.1.3.4 *Barium hydroxide,* approximately 0.01 mol/l solution.
This solution must be protected from atmospheric carbon dioxide, for instance, by storing under nitrogen.

E.1.3.5 *Hydrochloric acid,* 0.02 mol/l standard solution.

E.1.3.6 *Oxygen,* hydrocarbon free.

This can be prepared by passing the gas through a furnace tube similar to that used in the determination and absorbing the carbon dioxide formed in soda lime.

E.1.3.7 *Arneil catalyst.*
Dissolve 1.4 g of electrolytic iron powder in concentrated nitric acid and evaporate the solution almost to dryness. Dissolve the resulting ferric nitrate in 100 ml of water and mix this solution with 198 g of cupric oxide powder sieved through a sieve of 75 µm nominal aperture width, 50 g of kaolin and 3g of sodium silicate powder. Add more water to form a smooth paste and extrude through a 5 mm nozzle. Dry the resulting threads at 110 °C. Break into approximately 6 mm lengths and heat at 900 °C.

NOTE *– There are several methods of preparaing this reagent. The above method is recommended.*

E.1.4 PROCEDURE

E.1.4.1 *Preparation of the absorber*

To ensure that the absorber and the water are free from acidic impurities, place 10 ml of the water in the absorber, pass the nitrogen **(E.1.3.2)** through the absorber at 5 litres per hour for 5 minutes and then add two to three drops of the indicator solution **(E.1.3.3)**. On the addition of 1 drop of the barium hydroxide solution **(E.1.3.4)** the colour shall turn violet – blue.

NOTE *– If the solution does not turn violet – blue, thoroughly reclean the absorber and test again.*

E.1.4.2 *Standardization*

Maintaining the nitrogen flow at 5 litres per hour, carefully add from a burette 20.0 ml of the barium hydroxide **(E.1.3.4)** to the solution in the absorber, prepared as described in **E.1.4.1.** Rinse down the mouth of the absorber with water and titrate the contents with the standard hydrochloric acid **(E.1.3.5)** until the colour just changes.

E.1.4.3 *Blank determination*

Thoroughly purge the catalyst tube, maintained at 600 °C to 650 °C with the reagent oxygen **(E.1.3.6)**. After all the air has been displaced by the oxygen, adjust the flow rate to 5 litres per hour.

Charge as described in **E.1.4.2,** the absorber prepared as described in **E.1.4.1,** with 20.0 ml of the barium hydroxide solution. Stopper the absorber mouth with a rubber bung and close stopcock $\mathbf{T_1}$. Immediately, connect this to the catalyst tube as shown in Figure 6, with cocks $\mathbf{T_1}$ closed and $\mathbf{T_2}$ opened.

NOTE – Care must be taken to prevent the absorption of atmospheric carbon dioxide by the barium hydroxide solution.

Quickly open $\mathbf{T_1}$ and close $\mathbf{T_2}$ and record the initial meter reading. Continue passing the reagent oxygen for at least 4 hours. At the end of this time read the meter again, close $\mathbf{T_1}$ and open $\mathbf{T_2}$. Disconnect the absorber from the catalyst tuber, wash down any liquid in its side arms with water, and titrate the contents with the standard hydrochloric acid, while maintaining a flow of the reagent oxygen at 5 litres per hour through the absorber.

The difference in titration between the standardization and the blank determination shall not exceed 0.2 ml of 0.02 mol/l hydrochloric acid solution. If this value is exceeded then :

a) Check the apparatus for leaks ;
b) Re-activate the catalyst by reduction and re-oxidation ; and
c) Examine the soda lime.

E.1.4.4 *Determination*

Carry out the procedure given in **E.1.4.3** but use the sample gas instead of the reagent oxygen.

Determine the mean temperature and barometric pressure during the time of sample flow by any suitable means.

E.1. 5 CALCULATION

Total hydrocarbon content, as carbon dioxide, parts per million by volume, at 27 °C and 101.3 kPa

$$= \frac{V_1 - V_2}{V_3} \times 246 \times \frac{273 + t}{300} \times \frac{101.3}{P}$$

Where,

V_1 is the volume, in millilitres, of 0.02 mol/l hydrochloric acid used in the blank;
V_2 is the volume, in millilitres, of 0.02 mol/l hydrochloric acid used in the determination;
V_3 is the volume, in litres, of sample passed;
t is the mean temperature, in °C, of the gas passed through the gas meter; and
P is the mean barometric pressure, in kPa, during the determination less the vapour pressure of water, in kPa, at tempretrature t.

NOTE – It is convenient to determine the carbon dioxide content and total hydrocarbon content simultaneously by passing the oxygen stream form the first determination, straight to the second. In this case it is important not to put the indicator solution into the absorber for the carbon dioxide determination because this ethanolic solution can give rise to ethanol vapour which will be carried over into the hydrocarbon determination train.

E.2 METHOD II

E.2.1 Principle

The method uses the principle of ionizing the hydrocarbon molecules that are present in a particular sample in a flame, and monitoring the change in potential across two electrodes in close proximity to the flame, as illustrated in Figure **9.** The change in potential is proportional to the quantity of hydrocarbons present.

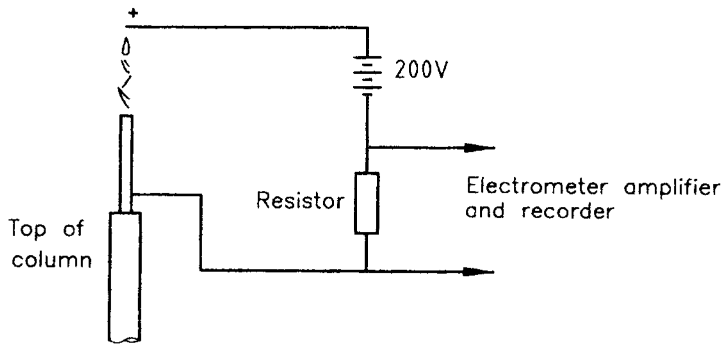


FIGURE 9 – Flame ionization detector (schematic)

E.2.2 Apparatus

E.2.2.1 Gas *chromatograph,* fitted with a flame ionization detector, a gas sampling valve, and a suitable valve to enable hydrocarbons to be backflushed to the detector. Nitrogen carrier gas shall be used.

E.2.3 Calibration

Calibration of these units shall be carried out using a calibration mixture. The mixture shall contain only methane in a balance of nitrogen, and the result of the calibration shall be expressed as 'total hydrocarbons calibrated as methane'.

E.2.4 Procedure

After calibrating the instrument, introduce the sample of the product in accordance with the manufacturer's instructions. Record the output signal on a computing integrator and calculate the concentration of hydrocarbons in accordance with the computing integrator manufacturer's instructions.

APPENDIX F
DETERMINATION OF WATER CONTENT

F.1 APPARATUS

Direct reading hygrometer, of one of the following types :

a) Cooled mirror dew point;
b) Electrolytic;
c) Capacitance; or
d) Piozoelectric.

F.2 PROCEDURE

Operate the direct reading hygrometer (**E.1**) in accordance with the manufacturer's instructions. Keep all sample lines as short as practicable and, together with all ancillary equipment, ensure that they have only polytetrafluoroethylene or stainless steel wetted parts. Introduce the sample of the product in accordance with the manufacturer's instructions and record the water content that is displayed by the instrument.

APPENDIX G
SAFETY CONSIDERATIONS FOR THE HANDLING OF OXYGEN

The product is non-toxic, but vigorously supports combustion of many materials, some of which would not burn in air.

The liquid product and the cold gaseous product can cause severe burns or frostbite when in contact with the skin or respiratory tract.

The product should not be used in confined spaces without ventilation. Personnel should not enter areas enriched with oxygen.

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.*

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