

**SRI LANKA STANDARD 645:PART 2:1984**  
**UDC 631.8:543.71**

**METHODS OF TEST FOR**  
**FERTILIZERS**  
**PART 2—DETERMINATION OF MOISTURE CONTENT**

**SRI LANKA STANDARDS INSTITUTION**



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PART 2 : DETERMINATION OF MOISTURE CONTENT

SLS 645:Part 2:1984

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SRI LANKA STANDARD  
METHODS OF TEST FOR FERTILIZERS

PART 2 : DETERMINATION OF MOISTURE CONTENT

**FOREWORD**

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1984-12-20, after the draft, finalized by the Drafting Committee on Fertilizers, had been approved by the Agricultural and Food Products Divisional Committee.

This part is one of a series of standards on testing of fertilizers. Other parts covering the determination of nitrogen, buret, potassium and phosphorous are being issued. A complete list of standards may be obtained from the Sri Lanka Standards Institution.

This part of the standard describes three methods for the determination of moisture in fertilizers. Methods 1 and 2 are recommended for use in routine analysis and their application is determined by the composition and nature of the fertilizers being tested. Method 3 is a general method based on ISO 760: Determination of water, Karl Fischer Method and is recommended for use as a reference method.

The values given in this standard are in SI units.

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

In the preparation of this standard, the assistance derived from the publications of the International Organization for Standardization and the Indian Standards Institution is gratefully acknowledged.

**1 SCOPE**

This part of the standard prescribes methods for the determination of moisture in fertilizers including fertilizer mixtures.

## 2 REFERENCES

CS 102 Presentation of numerical values

SLS 559 Sampling of fertilizers.

## 3 QUALITY OF REAGENTS

Unless specified otherwise, chemicals of a recognized analytical grade and distilled water shall be employed in tests.

## 4 PREPARATION OF TEST SAMPLE

Reduce the test sample as specified in Clause 6 of SLS 559:1972, to a quantity sufficient for analysis and grind not less than 225 g of the reduced sample without previously sieving. For fertilizer materials and moist fertilizer mixtures, grind to pass through a sieve of 1 mm aperture size. For dry mixtures that tend to segregate, grind to pass through a sieve of 350  $\mu\text{m}$  aperture size. Grind as rapidly as possible to avoid loss or gain of moisture during the operation. Mix thoroughly and store in tightly stoppered bottles.

## 5 METHOD 1

### 5.1 Field of application

This method is applicable to all types of fertilizers and fertilizer mixtures except those that yield volatile substances other than water at drying temperature.

### 5.2 Apparatus

5.2.1 *Weighing bottle*, 70 mm - 80 mm diameter, with lid;

5.2.2 *Desiccator*, holding silica gel as absorbing agent;

5.2.3 *Oven*, in which drying can take place at  $105 \pm 2$  °C.

### 5.3 Procedure

Weigh, to the nearest milligram, approximately 10 g of the sample prepared as in 4 in a dried and tared weighing bottle (see 5.2). Place the uncovered weighing bottle holding the test portion in an oven (see 5.2) and dry it at  $105 \pm 2$  °C for 5 hours. Allow the uncovered weighing bottle to cool for one hour inside a desiccator (see 5.2). At the same time keep the lid also for one hour in the desiccator. Cover the weighing bottle with the lid and weigh. Repeat the drying operation in the oven until the results of two consecutive weighings at an interval of about one hour do not differ by more than 5 mg.

#### 5.4 Expression of results

$$\text{Moisture, per cent by mass} = \frac{m_o - m_1}{m_o} \times 100$$

where,

$m_o$  = mass, in g, of the test portion before drying, and

$m_1$  = mass, in g, of the test portion after drying.

### 6 METHOD 2

#### 6.1 Field of application

This method is applicable to ammonium chloride and diammonium phosphate.

#### 6.2 Apparatus

6.2.1 *Porcelain dish.*

6.2.2 *Vacuum desiccator.*

#### 6.3 Procedure

Weigh, to the nearest milligram, approximately 5 g of the prepared sample in a weighed shallow porcelain dish and dry for 24 hours in a vacuum desiccator over sulphuric acid and reweigh. Preserve the dried material for subsequent tests.

#### 6.3 Calculation of results

$$\text{Moisture, per cent by mass} = \frac{m_o - m_1}{m_o} \times 100$$

where,

$m_o$  = mass, in g, of the prepared sample taken for the test; and

$m_1$  = mass, in g, of the sample after drying.

## 7 METHOD 3 (KARL FISCHER METHOD)

## 7.1 Field of application

This is a general method suitable for the determination of free water or water of crystallization in most solid or liquid fertilizer products, both organic and inorganic.

## 7.2 Principle

This method makes use of the reaction of any water present in the test portion with a solution of iodine and sulfur dioxide in a pyridine/methanol mixture (Karl Fischer reagent), previously standardized by titration with an exactly known mass of water.

*NOTE - Methanol may be replaced by 2-methoxyethanol (ethylene glycol monoethyl ether). With this solvent, a more constant titration is obtained and the reagent can be used with aldehydes and ketones without using any special technique.*

*Two platinum electrodes subjected to a potential difference are immersed in the solution. While vapour is present in the solution, the polarization of the cathode opposes the passage of the current. The depolarization of the cathode accompanied by a sudden increase in the current indicates the end point.*

## 7.3 Reactions



## 7.4 Reagents

7.4.1 *Methanol*, not containing more than 0.05 per cent (m/m) of water. If the reagent contains more than this quantity of water, dry it by distillation from magnesium turnings activated with iodine. Collect the distillate in a receiver protected from atmospheric moisture by means of a guard tube filled with the desiccant (7.4.9).

7.4.2 *2-methoxyethanol (Ethylene glycol monoethyl ether)*, not containing more than 0.05 per cent (m/m) of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate, which contains the water.

7.4.3 *Pyridine*, not containing more than 0.05 per cent (m/m) of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate, which contains the water.



7.4.4 *Sample solvent*, either a mixture containing 4 parts by volume of the methanol (7.4.1) and 1 part by volume of the pyridine (7.4.3), or (preferably for determination with compounds containing carbonyl groups) a mixture containing 4 parts by volume of the 2-methoxyethanol (7.4.2) and 1 part by volume of the pyridine (7.4.3). In special cases, other solvents may be recommended, for example acetic acid, pyridine or a mixture containing 1 part by volume of the methanol (7.4.1) and 3 parts by volume of chloroform.

7.4.5 *Karl Fischer reagent*. Place 670 ml of the methanol (7.4.1) or the 2-methoxyethanol (7.4.2) in a dry brown glass flask, fitted with a ground glass stopper and having a capacity slightly greater than 1 litre.

Add about 85 g of iodine. Stopper the flask and shake it occasionally until the iodine is completely dissolved. Then add approximately 270 ml of the pyridine (7.4.3), stopper the flask again and mix thoroughly. Using the method described below, dissolve 65 g of sulfur dioxide in this solution, cooling to ensure that the temperature of the liquid does not exceed 20 °C.

*NOTE - As the reaction is exothermic, it is necessary to cool the flask from the beginning and to maintain it at about 0 °C, for example by immersing it in an ice bath or in crushed solid carbon dioxide.*

Replace the ground glass stopper by an attachment for introducing sulfur dioxide, consisting of a cork bearing a thermometer and a glass inlet tube 6 mm x 8 mm, reaching to within 10 mm of the bottom of the flask, and a small capillary tube for connecting to the atmosphere.

Place the whole assembly with the ice bath on a balance and weigh to the nearest gram. Connect the inlet tube to a cylinder of sulfur dioxide by means of a flexible connection and a drying tube filled with the desiccant (7.4.9) and gently open the tap on the cylinder.

Adjust the rate of flow of sulfur dioxide so that all the gas is absorbed without the liquid showing any sign of rising in the inlet tube.

Maintain the equilibrium of the balance by gradually increasing the tare, and ensure that the temperature of the liquid does not rise above 20 °C. Close the tap on the cylinder as soon as the increase in mass reaches 65 g.

Immediately remove the flexible connection and reweigh the flask and its inlet attachment. The mass of dissolved sulfur dioxide should be between 60 g and 70 g. A slight excess is not harmful.

Stopper the flask, mix the solution and leave for at least 24 hours before using it. In fact, as the result of imperfectly understood reactions which occur in the fresh reagent, the water equivalent of the reagent decreases rapidly to begin with and then much more slowly.

This water equivalent is between 3.5 mg/ml and 4.5 mg/ml. It shall be determined daily if methanol has been used, but may be determined less frequently if 2-methoxyethanol has been used.

It is possible to prepare the Karl Fischer reagent having a lower water content by diluting the solution prepared as described above with the sample solvent (7.4.4).

Store the reagent in the dark and protected from atmospheric moisture. It should preferably be stored in the reagent bottle.

7.4.6 *Sodium tartrate dihydrate* ( $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$ ), or water (see Note under 7.4.7).

7.4.7 *Water/methanol*, standard solution corresponding to 10 g of water per litre.

Using a microburette or a pipette, place 1 ml of water in a perfectly dry 100-ml one-mark volumetric flask containing approximately 50 ml of the methanol (7.4.1). Dilute to the mark with the methanol and mix.

1 ml of this standard solution contains 10 mg of water.

*NOTE - Standardization of Karl Fischer reagent with sodium tartrate dihydrate is given in 7.6.1 and standardization of Karl Fischer reagent with water/methanol solution is given in Appendix A.*

7.4.8 *Water/methanol*, standard solution corresponding to 2 g of water per litre.

Using a microburette or a pipette, place 1 ml of water in a perfectly dry 500 ml one-mark volumetric flask containing approximately 100 ml of the methanol (7.4.1). Dilute to the mark with the same methanol and mix. (See the correspondence by volume of this solution with the Karl Fischer reagent in 7.4.5).

1 ml of this standard solution contains 2 mg of water.

7.4.9 *Aluminium sodium silicate*, anhydrous, in the form of granules of diameter 1.7 mm, for use as a desiccant. The granules may be regenerated by washing with water and drying at 350 °C for at least 48 hours.

Alternatively, activated silica gel may be used as a desiccant.

7.4.10 *Silicone-base grease*, for lubricating the ground glass joints.

## 7.5 Apparatus

All the glassware used shall be previously dried for 30 minutes in an oven controlled at approximately 130 °C, then allowed to cool and stored in a desiccator containing the desiccant (7.4.9).

A suitable form of apparatus for this method is given in Figures 1, 2, and 3 should no commercial apparatus be available. This standard apparatus comprises the parts described in 7.5.1 to 7.5.9.

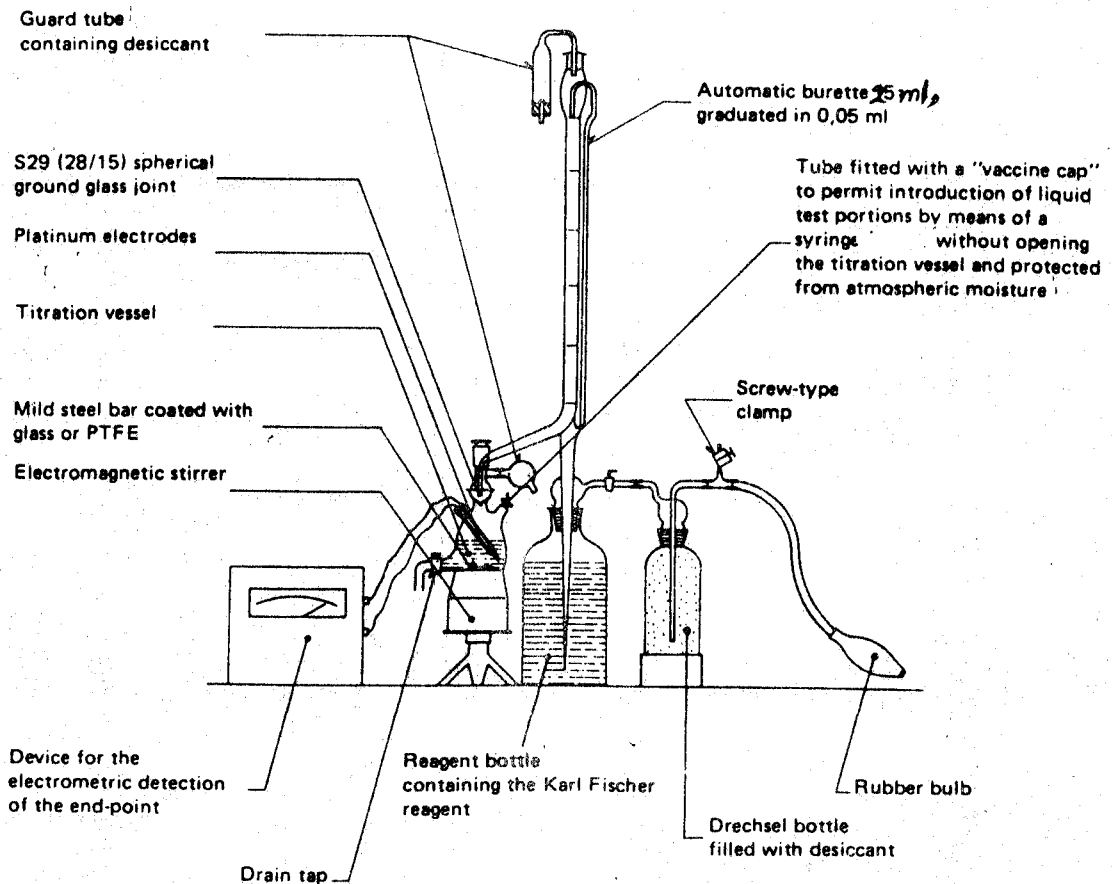


FIGURE 1 - Typical apparatus for direct electrometric titration

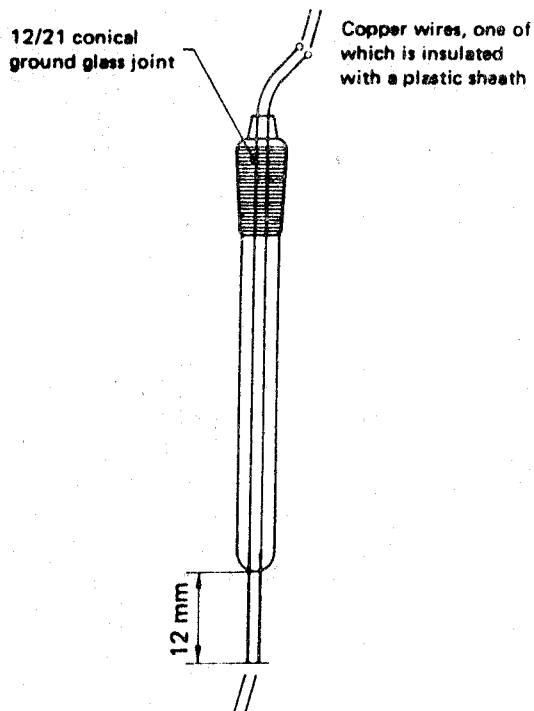


FIGURE 2 - Platinum electrodes

7.5.1 *Automatic burette*, of capacity 25 ml, with a fine point, graduated in 0.05 ml and protected from atmospheric moisture by a gaurd tube filled with the desiccant (7.4.9).

7.5.2 *Titration vessel*, of effective capacity 100 ml, connected to the tap of the automatic burette (7.5.1) by a ground glass joint and having two side tubes, one permitting the introduction of platinum electrodes and the other fitted with a "vaccine cap" to permit the introduction of liquid test portions by means of a syringe without opening the vessel.

7.5.3 *Platinum electrodes*, (see Figures 1 and 2), fused to a glass tube enabling them to be introduced into the bottom of the titration vessel (7.5.2) and joined to two copper wires which in turn are connected to the device for the electrometric detection of the end-point (7.5.7).

7.5.4 *Electromagnetic stirrer*, operating at a rotational frequency of  $150 \text{ min}^{-1}$  to  $300 \text{ min}^{-1}$  with a mild steel bar coated with glass or polytetrafluorethylene (PTFE), and fixed on a base of adjustable height.

7.5.5 *Reagent bottle for the Karl Fischer reagent*, of capacity approximately 3 litres, of brown glass, in which the filling tube of the automatic burette (7.5.1) is immersed through the ground glass stopper.

7.5.6 *Rubber bulb*, connected to a Drechsel bottle filled with the desiccant (7.4.9), for admission of dry air under pressure into the reagent bottle (7.5.5) in order to fill the automatic burette (7.5.1).

7.5.7 *Device for the electrometric detection of the end-point*, shown diagrammatically in Figure 3 .

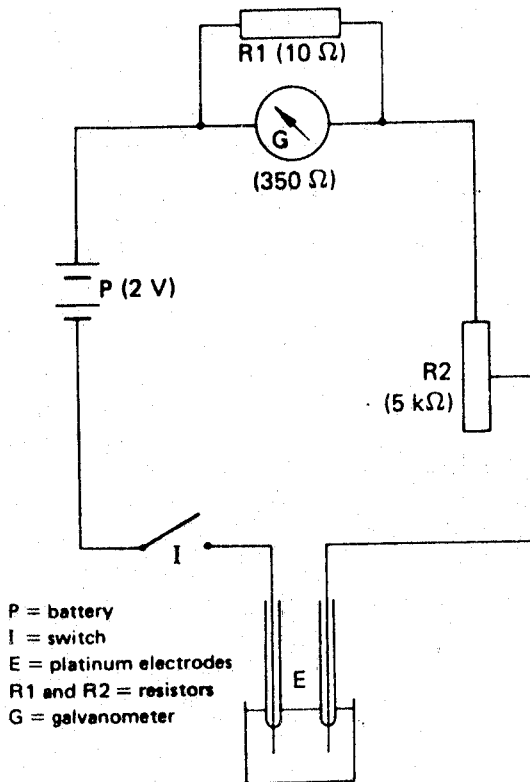
7.5.8 *Medical syringes*, of suitable capacity, the volume of which is calibrated.

7.5.9 *Small glass tube*, closed at one end and fitted at the other end with a rubber stopper, used for weighing and introducing into the titration vessel, for example, the mass of sodium tartrate (7.4.6) (approximately 0.250 g) used to standardize the Karl Fischer reagent (7.4.5), or possibly test portions of solid products.

## 7.6 Procedure

### 7.6.1 Standardization of the Karl Fischer reagent

7.6.1.1 Assemble the apparatus as shown in Figure 1 , lubricating the joints with the grease (7.4.10). Introduce by means of a syringe (7.5.2) 25 ml of the methanol (7.4.1) into the titration vessel (7.5.2) through the "vaccine cap". Switch on the electromagnetic stirrer (7.5.4) and close the circuit of the device for the electrometric detection of the end-point (7.5.7).



**FIGURE 3 - Circuit for the device for the electrometric detection of the end-point (direct titration: increase of current)**

Adjust the apparatus so that a potential difference of 1 V to 2 V is applied to the electrodes and the galvanometer shows a low current, usually a few microamperes. In order to cause reaction of the traces of water present in the methanol, add the Karl Fischer reagent (7.4.5) until the galvanometer shows a sudden increase in current of about  $10\ \mu\text{A}$  to  $20\ \mu\text{A}$ , which remains stable for at least one minute.

7.6.1.2 In the small glass tube (7.5.3), weigh, to the nearest 0.1 milligram, approximately 0.250 g of the sodium tartrate (7.4.6). Place this in the titration vessel very quickly, removing the "vaccine cap" for a few seconds; then weigh the small glass tube, so as to determine, by difference, the mass ( $m_1$ ) of the sodium tartrate used.

Standardization may also be effected by introducing a mass ( $m_2$ ) of water of approximately 0.040 g from a dropping bottle; weighed before and after introduction into the titration vessel.

Titrate the known quantity of water introduced with the Karl Fischer reagent (7.4.5) to be standardized, until the same deflection of the pointer of the galvanometer is reached and remains stable for at least one minute. Note the volume ( $V_1$ ) of reagent used.

### 7.6.2 Determination

Empty the titration vessel (7.5.2) by means of its drain tap. Place in it 25 ml (or the volume specified in the procedure for the product to be analysed) of the methanol (7.4.1) or other solvent (7.4.3 or 7.4.4) using a syringe (7.5.2) passing through the "vaccine cap". Switch on the electromagnetic stirrer (7.5.4). In order to cause reaction of the traces of water present in the solvent used, and the Karl Fischer reagent (7.4.5), proceeding as specified in 7.6.1 until there is a sudden and constant deflection lasting for at least one minute.

Introduce the specified test portion by means of a syringe in the case of a liquid or weighing to the nearest 0.1 milligrams in a small glass tube (7.5.3) in the case of a solid powder. Titrate with the Karl Fischer reagent using the same electrometric procedure for detecting the end-point of the reaction.

Note the volume ( $V_1$ ) of Karl Fischer reagent used for the determination.

*NOTE - It is advisable to use a test portion such that its water content corresponds to a volume of Karl Fischer reagent that can be measured with sufficient accuracy. If necessary, increase in proportion the quantities of solvent and test sample used and then use a titration vessel of suitable capacity.*

### 7.6.3 Calculation of results

#### 7.6.3.1 Water equivalent of the Karl Fischer reagent

The water equivalent  $T$  of the Karl Fischer reagent (7.4.5), expressed in milligrams of water per millilitre of reagent, is given by the formula

$$T = \frac{m_1 \times 0.1566}{V_1} \quad \text{or} \quad T = \frac{m_2}{V_1}$$

where

$m_1$  = mass, in milligrams, of the sodium tartrate (7.4.6) introduced if this reagent is used for the standardization (7.6.1);

$m_2$  = mass, in milligrams, of water introduced if pure water is used for the standardization;

$V_1$  = volume, in millilitres, of the Karl Fischer reagent (7.4.5) used for the standardization; and

0.156 6 = factor for calculating the water of the sodium tartrate dihydrate.

For standardization with the standard water/methanol solution, (see Appendix A).

### 7.6.3.2 Water content of the sample

The water content of the sample, expressed as a percentage by mass, is given by the formula:

$$\frac{V_2 \times T}{m_o \times 10} \quad \text{or} \quad \frac{V_2 \times T}{V_o \times p \times 10}$$

where

$m_o$  = mass, in grams, of the test portion (in the case of solid products);

$V_o$  = volume, in millilitres, of the test portion (in the case of liquid products);

$p$  = density of the sample, in grams per millilitre, at 20 °C (in the case of liquid products);

$V_2$  = volume, in millilitres, of the Karl Fischer reagent (7.4.5) used for the determination (7.6.2); and

$T$  = water equivalent, in milligrams per millilitre, of the Karl Fischer reagent, calculated in accordance with 7.6.3.1.

## APPENDIX A

### STANDARDIZATION OF KARL FISCHER REAGENT WITH STANDARD WATER/METHANOL SOLUTION

A.1 If the standard water/methanol solution (7.4.7) is used instead of the sodium tartrate (7.4.6) to standardize the Karl Fischer reagent (7.4.5), sub-clause 7.6.1.2 relating to direct electrometric titration is amended as follows:

Using a syringe (7.5.2) add 10.0 ml of the methanol (7.4.1) to the titration vessel; titrate with the Karl Fischer reagent (7.4.5) until the same deflection of the pointer of the galvanometer is reached and remains stable for at least one minute and note the volume ( $V_3$ ) of reagent used.

In the same way, add 10.0 ml of the standard water/methanol solution (7.4.7). Titrate the known quantity of water thus introduced with the Karl Fischer reagent (7.4.5) to be standardized until the same deflection of the pointer of the galvanometer is reached and remains stable for at least one minute. Note the volume ( $V_4$ ) of reagent used.

In addition, sub-clause 7.6.3.1 is amended as follows:

The water equivalent  $T$  of the Karl Fischer reagent (7.4.5), expressed in milligrams of water per millilitre of reagent, is given by the formula:

$$T = \frac{100}{V_3 - V_4}$$

where,

100 = mass, in milligrams, of water contained in 10 ml of the standard water/methanol solution (7.4.7);

$V_3$  = volume, in millilitres, of the Karl Fischer reagent (7.4.5) used for the titration of the 10 ml of the methanol (7.4.1); and

$V_4$  = volume, in millilitres, of the Karl Fischer reagent (7.4.5) used for the titration of the 10 ml of the standard water/methanol solution (7.4.7).

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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.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

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All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.