SRI LANKA STANDARD 912: 2019

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SPECIFICATION FOR RED PHOSPHORUS (*First Revision*)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 912: 2019

Gr. 6

SRI LANKA STANDARDS INSTITUTION
No. 17, Victoria Place,
Elvitigala Mawatha,
Colombo 08.
SRI LANKA

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

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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 2019-10-22.

Red phosphorus is one of the important raw materials used in the manufacture of safety matches.

This Specification was first published in 1990. In this first Revision, All the amendments issued after the publication of **SLS 912: 1990** were incorporated and the requirement for particle size was removed as there is no significant impact of the particle size for the manufacturing process of safety matches.

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 **SLS 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 formulation of this Specification, the assistance derived from the following publication is gratefully acknowledged:

IS 2012 :2006 Red phosphorus – Specification

1 SCOPE

This specification prescribes the requirements and methods of sampling and test for red phosphorus for use in the safety match industry.

2 REFERENCES

SLS 102 Rounding off numerical values
SLS 428 Random sampling methods.
SLS 692 Safety colours and safety signs

3 REQUIREMENTS

3.1 General requirement

The material shall be in the form of a dark red amorphous powder. It shall be free from visible impurities.

3.2 Other requirements

The material shall also comply with the requirements given in Column **3** of Table **1** when tested by the method given in Column **4** of the table.

TABLE 1 - Requirements for red phosphorus

SI. No.	**Characteristic**	**Requirement**	**Method of test**
(1)	(2)	(3)	(4)
i)	Red phosphorus, per cent by mass, min.	97.5	Appendix B
ii)	Moisture, per cent by mass, max.	0.3	Appendix C
iii)	Acidity, as H_3PO_4 per cent by mass, max.	0.3	Appendix D

3.3 White phosphorus

The material shall not contain white phosphorus when tested by the method prescribed in Appendix **E**.

3.4 Shelf life

Shelf life shall be minimum of 6 months from the date of manufacture.

4 PACKAGING AND MARKING

4.1 Packaging

The material shall be packed in suitable air-tight containers.

4.2 Marking

Each container shall be legibly and indelibly marked or labelled with the following:

a) Name of the product;
b) The word "Flammable" with the appropriate symbols for labelling dangerous goods; (see **SLS 692**);
c) Name and address of the manufacturer and/or supplier and country of origin;
d) Registered trade mark, if any;
e) Net content, in kilograms;
f) Batch or code Number; and
g) Date of manufacture and shelf life.

5 METHODS OF TEST

Tests shall be carried out according to Appendices **B** to **E** of this specification. Unless otherwise stated, reagents of analytical grade and distilled water or water of equivalent purity shall be used.

5.1 Precaution

Necessary precautions shall be taken during testing, since red phosphorus is a flammable substance.

APPENDIX A
COMPLIANCE OF A LOT

The sampling scheme given in this Appendix shall 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 Specification is to be assured, appropriate schemes of sampling and inspection shall be adopted based on manufacturer's control systems coupled with Type Tests and Testing Procedures.

A.1 LOT

In any consignment all the containers of red phosphorus of the same size belonging to one batch of manufacture or supply shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

In drawing, preparing and handling of samples the following precautions and directions shall be observed:

A.2.1 The sampling thief and sample containers shall be protected from adventitious contamination.

A.2.2 The Aluminium sampling thief used shall be clean and dry.

A.2.3 The samples shall be placed in clean, dry air-tight glass or other suitable containers on which the material has no action.

A.2.4 The sample containers shall be of such a size that they are almost completely filled by the sample.

A.2.5 Each sample container shall be marked with relevant details.

A.2.6 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from normal atmospheric temperature.

A.3 SCALE OF SAMPLING

A.3.1 Samples shall be tested from each lot for ascertaining its conformity to the requirements of this specification.

A.3.2 The number of containers to be selected from the lot shall be in accordance with Table **2**.

TABLE 2 - Scale of sampling

Number of packages in the lot (1)	Number of packages to be selected (2)
Up to 500	3
501 to 1 200	4
1201 to 3 200	5
3201 and above	7

A.3.3 The containers 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.4 PREPARATION OF SAMPLES

A.4.1 Individual samples

A representative sample of material shall be drawn from each container selected as in **A.3.2** using an Aluminium sampling thief. The material taken from each container shall be transferred to separate sample containers. The minimum size of a sample shall be 150 g.

A.4.2 Composite sample

Approximately equal quantities of representative samples shall be drawn from each container selected as in **A.3.2** using an Aluminium sampling thief, mixed together and reduced by the method of coning and quartering to get a composite sample of about 100 g. The composite sample shall be transferred to a sample container.

A.5 NUMBER OF TESTS

A.5.1 Each container selected as in A**.3.2** shall be inspected for packaging and marking requirements.

A.5.2 The individual samples prepared as in A**.4.1** shall be tested for acidity.

A.5.3 The composite sample prepared as in A**.4.2** shall be tested for moisture, red phosphorus, and for the presence/absence of white phosphorus.

A.6 CRITERIA FOR CONFORMITY

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

A.6.1 Each container inspected as in A**.5.1** satisfies the relevant requirements.

A.6.2 Each individual sample when tested as in A**.5.2** satisfies the relevant requirements.

A.6.3 The composite sample when tested as in A**.5.3** satisfies the relevant requirements.

APPENDIX B
DETERMINATION OF RED PHOSPHORUS

B. l REAGENTS

B.1.1 Absolute alcohol

B.1.2 Diethyl ether

B.1.3 Iodine, crystals

B.1.4 Nitric acid, concentrated (rel.den. =1.42)

B.1.5 Ammonium citrate, solid

B.1.6 Ammonium hydroxide,20 per cent (V/V) solution

B.1.7 Hydrochloric acid, 5 mol/1 solution

B.1.8 Magnesia mixture

Dissolve 110 g of Magnesium chloride ($MgCl_2.6H_20$) in a small amount of water. Add 288 g of Ammonium chloride and 700 ml of Ammonium hydroxide (rel. den. = 0.90). Dilute the solution to 2 litres with water. Allow to stand for several hours, filter and transfer the solution to a 2-litre glass-stoppered bottle.

NOTE
Filter the solution before use.

B.1.9 Ammonium nitrate solution, saturated.

B.2 PROCEDURE

B.2.1 Weigh about 20 g of the material in an extraction thimble. Place the thimble in a soxhlet extraction apparatus and extract for 1 hour with 100 ml of water on a sand bath. Remove water, cool the system and repeat the extraction for 1 hour with 100 ml of absolute alcohol (**B.1.1**) on a water bath. Remove alcohol/ cool the system and repeat the extraction for 1 hour with 100 ml of diethyl ether **(B.1.2**). Remove the thimble and allow to evaporate slowly any ether left. Dry the residue for 30 minutes in a desiccator containing Phosphorus pentoxide.

B.2.2 Weigh, to the nearest milligram, 1 g of the residue transfer into a 250-ml conical flask, add about 5 ml of water and mix well. Add in small portions 12 g of iodine **(B.1.3)** to dissolve the material. Gently heat the flask and add carefully 50 ml of concentrated Nitric acid **(B.1.4)**. After the dissolution of the Phosphorus iodide add another 20 ml of concentrated Nitric acid and boil gently until all the iodine is expelled. Dilute and filter through a medium filter paper into a 500-ml volumetric flask and add water upto the mark.

B.2.3 Pipette 25 ml of the solution and add to 5 g of Ammonium citrate **(B.l.5)** dissolved in 25 ml of water in a 200-ml beaker. Make the solution alkaline with Ammonium hydroxide **(B.1.6)** and then acidic with dilute Hydrochloric acid **(B.1.7)** with using Methyl red indicator or litmus paper. Add dropwise 25 ml of cold magnesia mix **(B.1.8)** with constant stirring. Stop stirring when the solution becomes, cloudy and allow the precipitate to settle for about 10 minutes. Add 20 ml of Ammonium hydroxide **(B.1.6)** with stirring and leave the precipitate to settle for at least 4 hours.

B.2.4 Filter through a medium filter paper and wash the precipitate several times with small quantities of dilute Ammonium hydroxide **(B.1.6)**. Dissolve the precipitate in Hydrochloric acid **(B.1.7)** and dilute approximately to 50 ml in a 200-ml beaker. Precipitate Magnesium ammonium phosphate as described in **(B.2.3)**. Filter the precipitate through an ashless medium filter paper and wash the precipitate with Ammonium hydroxide **(B.1.6)** until free of chloride. Place the filter paper with the precipitate in a tared porcelain crucible, add a few drops of Ammonium nitrate solution **(B.1.9)** and heat the crucible over a low flame until the paper is charred.

Place the crucible in a muffle furnace at 1 100 ± 20°C for 1 hour, cool in a desiccator and weigh the residual Magnesium pyrophosphate. Repeat the process of heating, cooling and weighing until a constant mass is obtained.

B.3 CALCULATION

$$\text{Red phosphorus, as P, per cent by mass} = 556.8\ \frac{m_2}{m_1}$$

where,

m_1 is the mass, in grams, of the material taken for the test; and
m_2 is the mass, in grams, of the residue.

APPENDIX C
DETERMINATION OF MOISTURE CONTENT

C.1 APPARATUS

C.1.1 Flat bottomed glass dish, approximately 60 mm diameter and 30 mm height with ground glass lid

C.1.2 Vacuum desiccator, with phosphorous pentoxide as desiccant

C.2 PROCEDURE

Heat the dish with lid in the oven for about 30 minutes. Cool it and weigh. Weigh accurately 5g of the sample, replace the lid and weigh accurately.

Uncover the dish and place it in a vacuum desiccator (10 mm or less of mercury) over phosphorus pentoxide for three hours. Weigh it with lid.

C.3 CALCULATION

$$\text{Moisture, per cent by mass} = \frac{(m_2 - m_3)x100}{(m_2 - m_1)}$$

where,

m_1 is the mass, in g, of the empty dish;
m_2 is the mass, in g, of the material and the dish; and
m_3 is the mass, in g, of the material and the dish after drying.

APPENDIX D
DETERMINATION OF ACIDITY

D.1 REAGENTS

D.1.1 *Phenolphthalein indicator*

Dissolve 0.1 g of phenolpbathalein in 100 ml of 95 per cent (V/V) ethyl alcohol

D.1.2 *Sodium chloride,* solid

D.1.3 *Sodium hydroxide,* standard solution, c(NaOH) = 0.1 mol/1

D.2 PROCEDURE

Weigh, to the nearest milligram, 5 g of the material, transfer into a 250-ml conical flask and add 100 ml of water. Heat the mixture to boiling and filter through a Buchner funnel using G5 crucible with gentle suction. Wash the residue several times with hot water collecting the washings in the flask. Add a few drops of phenolphthalein indicator **(D.1.1)** to the filtrate and 10 to 15 g of Sodium chloride **(D.1.2)**. Cool the solution in an ice bath and titrate with Sodium hydroxide solution **(D.1.3)**.

D.3 CALCULATION

$$\text{Acidity, as } H_3PO_4\text{, per cent by mass} = 4.9\ \frac{Vxc}{m}$$

where,

V is the volume, in millilitre, of standard Sodium hydroxide solution used for the titration;
c is the concentration, in mol/1, of standard Sodium hydroxide solution; and
m is the mass, in grams, of the material taken for the test.

APPENDIX E
DETERMINATION FOR THE PRESENCE OF WHITE PHOSPHORUS

E.1 PROCEDURE

Take 10 g of the sample in a stoppered measuring cylinder and add 20 ml of Carbon disulphide. Shake well. Allow it to settle for 2 h. Place a few drops of the clear supernatant liquid upon a piece of the Copper sulphate paper (made by soaking filter paper in 20 per cent Copper sulphate solution and drying in the oven at 60 ^{0}C). Absence of any brown stain indicates the absence of white phosphorus.

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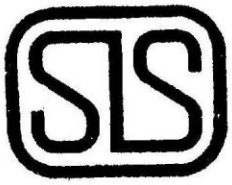

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SRI LANKA STANDARDS INSTITUTION

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