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SPECIFICATION FOR THINNER FOR CELLULOSE NITRATE BASED PAINTS AND LACQUERS (First Revision)

SRI LANKA STANDARDS INSTITUTION

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(First Revision)

SLS 749 : 2013

Gr.7

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Sri Lanka Standard SPECIFICATION FOR THINNER FOR CELLULOSE NITRATE BASED PAINTS AND LACQUERS (First Revision)

FOREWORD

This standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2013 - 04 - 30.

This specification was first published in 1986. In this First Revision new test methods have been introduced for the determination of distillation and acidity.

This product is used as a thinner for cellulose nitrate based paints and lacquers generally employed for finishing of aircrafts, automobiles and furniture.

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

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

ASTM I	D 86	Standard	test	method	for	distillation	of	petroleum	products	at
		atmosphe	ric pi	ressure						
IS	5667	Cellulose	nitra	te based	pain	ts and lacqu	ers			

1 SCOPE

This specification prescribes the requirements, and methods of sampling and test for thinner for cellulose nitrate based paints and lacquers.

2 **REFERENCES**

- SLS 102 Rules for rounding off numerical values
- CS 124 Test sieves
- SLS 351 Rectified spirits
- SLS 428 Random sampling methods
- SLS 523 Sampling of paints
- SLS 692 Safety colours and safety signs

3 REQUIREMENTS

3.1 General requirements

The material shall consist of organic solvents only and shall be clear and free from sediment and suspended matter. It shall have no objectionable odours.

3.2 Availability of toxic material

The material shall be free from benzene, Methanol and chlorinated hydrocarbons when tested as prescribed in **B**.

NOTE : *Method* 1 *of Appendix* **B** *is recommended for more accurate results.*

3.3 Compatibility

When a paint or lacquer based on cellulose nitrate is thinned in the ratio of one part of cellulose nitrate paint or lacquer with five parts by volume of thinner, the mixed material shall be smooth and uniform. It shall not show the tendency of breakdown, coagulation or precipitation of the paint or lacquer.

3.4 Keeping qualities

The material when stored in unopened containers under normal conditions shall retain its original characteristics for at least 12 months from the date of manufacture.

NOTE: *This requirement is optional for testing.*

3.5 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.

SI. No.	Characteristic	Requirement	Method of test	
(1)	(2)	(3)	(4)	
i) ii)	Colour Odour	Not darker than a freshly prepared standard potassium dichromate solution No appreciable residual odour after drying on a filter paper	Appendix C	
iii)	Residue on spontaneous evaporation	Shall completely evaporate from white filter paper without leaving a stain or oily spot	-	
iv)	Relative density at 27 °C/27 °C	0.81 to 0.85	Appendix D	
v)	Distillation °C a) Initial boiling point, min.	80 °C	ASTM D 86	
	b) 10 per cent volume recovery, min.	83 °C to 91 °C		
	 c) 50 per cent volume recovery d) 90 per cent volume recovery, max. 	92 °C to 112 °C 113 °C to 132 °C		
	e) Stop point, max.	150 °C		
vi)	Residue on evaporation, mg/100 ml, max.	5	Appendix E	
vii)	Copper corrosion	The material shall not blacken or corrode clean metallic copper	Appendix F	
viii)	Total hydrocarbon content, per cent by volume, max.	56	Appendix G	
ix)	Aromatic hydrocarbon content, per cent by volume, min.	30	Appendix G	
x)	Aliphatic hydrocarbon content, per cent by volume, max.	25	Appendix G	
xi)	Alcohols, esters, ketones, per cent by volume, min.	44	Appendix G	
xii)	Acidity (as acetic acid) per cent, by mass, max.	0.03	Appendix H	

TABLE 1 – Requirements for thinner

4 PACKAGING

The material shall be packed in suitable containers and the containers shall be securely closed.

5 MARKING

- 5.1 The containers shall be legibly and indelibly marked with the following:
- a) Name of the material;
- b) Name and address of the manufacturer for products manufactured in Sri Lanka. Name and address of the manufacturer and the distributor in Sri Lanka/ Importer need to be marked on imported products including country of origin ;
- c) Registered trade mark, if any;
- d) Batch or code number;
- e) Net volume, in millilitres or litres;
- f) Month and year of manufacture; and
- g) The words "HIGHLY FLAMMABLE LIQUID" in red letters and pictorial marking for flammable material as given in **B.3.2** of **SLS 692:1985.**

6 SAMPLING

Representative samples of the material for ascertaining conformity to the requirements of this specification shall be drawn as prescribed in Appendix A.

7 METHODS OF TEST

7.1 Tests shall be carried out as prescribed in Appendix **B** to Appendix **H** and **ASTM D 86.**

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

APPENDIX A COMPLIANCE OF A LOT

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

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

A.1 LOT

In any consignment all the containers of the same size and containing thinner of one batch of manufacture shall constitute a lot.

A.2 SCALE OF SAMPLING

A.2.1 Representative samples of the material shall be taken in accordance with relevant clauses of **SLS 523.**

A.2.2 Sufficient quantity of the material shall be taken from each container selected and transferred to separate sample containers.

A.3 NUMBER OF TESTS

A.3.1 Each container selected as in 7.2.1 of SLS 523:1981 shall be inspected for packing and marking requirements.

A.3.2 Material of each sample container obtained as in **A.2.2** shall be tested separately for the requirements given in **3.3** and characteristics (i) and (ii) of Table **1**.

A.3.3 Equal quantities of material shall be taken from each container selected as in **7.2.1** of **SLS 523:1981** and mixed to form a composite sample. The composite sample thus obtained shall be tested for requirements given in **3.2** and **3.5** other than colour and odour.

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 container inspected as in A.3.1 satisfies the relevant requirements.

A.4.2 The material of each sample container tested as in A.3.2 satisfies the relevant requirements.

A.4.3 The test results of the composite sample when tested as in A.3.3 satisfies the relevant requirements.

APPENDIX B METHOD OF TEST FOR AVAILABILITY OF TOXIC MATERIALS

- **B.1 METHOD 1** Gas Chromatography method
- **B.2** METHOD 2
- **B.2.1** Apparatus
- **B.2.1.1** Distillation assembly
- **B.2.1.2** Bunsen burner
- **B.2.2** Reagents
- **B.2.2.1** *Concentrated nitric acid*, rel. den. = 1.42
- **B.2.2.2** *Concentrated sulfuric acid*, rel. den. = 1.84.
- **B.2.2.3** *Copper wire,* of diameter 0.914 mm to 1.219 mm.

B.2.3 Procedure

B.2.3.1 *Test for benzene*

Place about 100 ml of sample, 100 ml of distilled water and a few boiling aids in the distillation flask. Slowly heat the contents at a uniform rate so that the first drop is obtained in about half an hour if benzene is present. Collect the fraction boiling up to about 80 °C (see Note). Mix 1 ml of nitric acid (**B.2.2.1**) and 1 ml of sulfuric acid (**B.2.2.2**) in a test tube and cool. Add 10 drops of the distillate and shake. Examine the tube for the smell of nitrobenzene, which is given by 5 per cent of benzene or larger amounts of toluene. Boil the contents for at least 30 seconds, allow to cool and add 10 ml water. Mix the contents and examine for smell of nitrobenzene which should no longer be present if the sample was boiled enough. Allow the tube to stand. A yellow cloudy solution or flocculent precipitate changing to yellow needles is the evidence of the presence of benzene. A white cloudy solution that separates to form a yellow liquid, perhaps with white needles, is evidence of the presence of toluene, which may obscure smaller amounts of benzene. The presence of benzene may further be confirmed by finding out the melting of the yellow compound which should be between 89 °C to 90 °C.

NOTE : *If there is no distillate to this point, benzene is presumed to be absent.*

APPENDIX C DETERMINATION OF COLOUR

C.1 APPARATUS

C.1.1 *Nessler cylinders*, of 50-ml capacity

C.1.2 *Standard potassium dichromate solution*, dissolve 0.003 g potassium dichromate in 1 litre of water.

C.2 PROCEDURE

Filter the material through a 150-mm diameter Whatman No. 31 filter paper and reject the first 10 ml of filtrate. Pour 50 ml of the material into a cylinder (C.1.1). Pour 50 ml of the standard solution (C.1.2) into another Nessler cylinder. Hold the Nessler cylinders in a comparator above the surface of an opaque glass sheet reflecting diffused daylight and examine visually.

APPENDIX D DETERMINATION OF RELATIVE DENSITY

D.1 APPARATUS

D.1.1 A pyknometer, of 25-ml capacity

D.2 PROCEDURE

Weigh, to the nearest milligram, a clean, dry pyknometer (**D.1.1**). Fill up to the mark with distilled water at the specified temperature and weigh to the nearest milligram. Empty the pyknometer, dry and fill with the material at the same temperature and weigh to the nearest milligram.

D.3 CALCULATION

Relative density =
$$\frac{m_1 \cdot m_2}{m_3 \cdot m_2}$$

where,

 m_1 is the mass, in grams, of the pyknometer filled with the material; m_2 is the mass, in grams, of the dry pyknometer; and m_3 is the mass, in grams, of the pyknometer filled with distilled water.

APPENDIX E DETERMINATION OF RESIDUE ON EVAPORATION

E.1 PROCEDURE

Evaporate 100 ml of the sample to dryness in a weighed glass or silica crucible of 150-ml capacity on water-bath. Dry the residue further in an air-oven at a temperature of 100 ± 2 °C for 90 minutes. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing until the difference in mass between two successive weighings does not exceed 0.1 mg.

E.2 CALCULATION

Residue on evaporation, mg/100 ml = $(m_1 - m_0) \times 1000$

Where,

m_0	is the weight of the empty crucible ; and
m_1	is the weight of the crucible with residue

APPENDIX F TEST FOR COPPER CORROSION

F.1 APPARATUS AND MATERIALS

- **F.1.1** Liebeg condenser, having a water jacket
- **F.1.2** Test tubes, thick walled, about 40 mm in diameter and 175 mm in length
- F.1.3 Test sieve, of aperture size 75 µm conforming to CS 124
- F.1.4 Copper foil, of electrolytic copper, size 40 mm x 30 mm x 0.8 mm
- **F.1.5** Emery powder, passing through 75-µm sieve

F.2 **PROCEDURE**

Clean the copper foil on both sides until the surface is highly polished, by rubbing it with a cork moistened with distilled water and dipped in dry emery powder. Wipe the foil with filter paper moistened with acetone, care being taken that it is not touched by fingers. Cut the cleaned foil into two strips of 30 mm x 20 mm each. Place one strip in a clean test tube and cork the tube. Introduce the other strip into another test tube, containing 50 ml of the material under

examination. Fit this test tube to the reflux condenser and allow it to remain for two hours in a water bath maintained at 50 ± 2 °C.

F.2.1 Compare the appearance of the two copper strips.

APPENDIX G DETERMINATION OF HYDROCARBON CONTENT (TOTAL, AROMATIC AND ALIPHATIC) AND ALCOHOLS, ESTERS AND KETONES

G.1 APPARATUS

G.1.1 Stoppered graduated measuring cylinders, 100-ml capacity

G.2 REAGENTS

G.2.1 Sulfuric acid, 80 per cent (V/V) solution, prepared by using concentrated sulfuric acid (rel. den. = 1.84).

G.2.2 *Dimethyl sulfate*

G.3 PROCEDURE

G.3.1 Total hydrocarbon content

Transfer 50 ml of the material into a measuring cylinder. Add to this 50 ml of diluted sulfuric acid (G.2.1) which has been cooled previously in ice. Mix thoroughly. Allow the contents of the cylinder to stand undisturbed until the mixture separates into two well distinct layers. The top layer insoluble in sulfuric acid consists of hydrocarbons. Carefully record the volume of the upper layer. Preserve the acid soluble portion for the determination of alcohols, esters and ketones.

G.3.1.1 Compute the percentage of total hydrocarbon content from the volume of the top layer and volume of material taken for determination.

G.3.2 Aromatic and aliphatic hydrocarbon content

Separate out the insoluble hydrocarbon layer obtained in **G.3.1** into a 100-ml measuring cylinder. Add an equal volume of dimethyl sulfate. Mix well and allow to settle.

G.3.2.1 Aromatic hydrocarbons are soluble. Compute the percentage by volume of aromatic hydrocarbon from the dimethyl sulfate layer.

G.3.2.2 Aliphatic hydrocarbons are insoluble. Measure the volume of the liquid and compute the percentage by volume of aliphatic hydrocarbon.

G.3.3 Alcohols, esters and ketones

Alcohols, esters and ketones are soluble in sulfuric acid. Separate the sulfuric acid soluble portion of the material (**G.3.1**) into a 100-ml measuring cylinder and calculate the percentage of alcohols, esters and ketones from the volume.

G.3.3.1 CALCULATION

Alcohols, esters and ketones per cent by volume = $\underline{V}_2 \times 100$

 V_{I}

Where,

- V_1 is the volume, in ml, of material taken ; and
- V_2 is the volume, in ml, of alcohols, esters and ketones.

APPENDIX H DETERMINATION OF ACIDITY

H.1 GENERAL

This method covers a procedure for the determination of total acidity as acetic acid. The material is mixed with an equal volume of ethyl alcohol and titrated against aqueous sodium hydroxide solution.

H.2 REAGENTS

- H.2.1 Rectified spirit conforming to SLS 351
- H.2.2 Phenolphthalein Indicator Solution
- **H.2.3** Standard sodium hydroxide solution, 0.1 mol/1

H.3 PROCEDURE

Measure into a 250-ml long necked flask 50 ml of rectified spirit and add 0.5 ml of indicator solution. Titrate the alcohol with standard sodium hydroxide solution to first perceptible pink colour. Pipette out 50 ml of the sample into the flask. Titrate with standard sodium hydroxide solution to the first appearance of permanent light pink colour. Note the volume of alkali used.

H.4 CALCULATION

Calculate the acidity of the material as follows :

Acidity (as acetic acid) per cent by weight $= \frac{V \times M \times 0.12}{d}$

where,

V is the volume, in ml, of standard sodium hydroxide solution used in titration ;

M is the molarity of standard sodium hydroxide solution ; and

d is the relative density of the material at 20 °C/20 °C.

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