SRI LANKA STANDARD 1256 : PART 7 : 2004 UDC 667.612

METHODS OF TEST FOR PAINTS AND VARNISHES PART 7 : DETERMINATION OF WATER BY THE DEAN AND STARK METHOD

SRI LANKA STANDARDS INSTITUTION

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SLS 1256 : Part 7 : 2004 (Superceding SLS 535: 1981 Part 2 – Sections 2.1 and 2.2)

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SRI LANKA STANDARD METHODS OF TEST FOR PAINTS AND VARNISHES PART 7 : DETERMINATION OF WATER BY THE DEAN AND STARK METHOD

FOREWORD

This Sri Lanka Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 2004-01-23.

This standard was published in 1981 which superceded CS 70 : 1969. In this revision each test method is given as a separate part in order to facilitate updating. This standard supercedes SLS 535 : Part 2 : Tests involving chemical examination of liquid paints and dried paint films : Section 2.1. Introduction and section 2.2 Determination of water by the Dean and Stark method.

1 SCOPE

This standard specifies a method of test for the determination of water in liquid paints, varnishes and allied products and dried films of these products using the Dean and Stark apparatus.

2 REFERENCES

- SLS 489 Glossary of terms for paints
- SLS 523 Methods of sampling paints
- SLS 1256 Methods of test for paints Part 1 Examination and preparation of samples for testing

3 DEFINITIONS

For the purpose of this standard, the terms defined in SLS 489 shall apply.

4 **PRINCIPLE**

The determination of water in paints or paint materials (other than water based paints) is carried out using the Dean and Stark apparatus.

5 APPARATUS

The Dean and Stark apparatus having the following essential features shall be used :

5.1 Flask

A flask of 500-ml capacity as shown in **Fig. 1** and made of hard resistance glass, well annealed and as free as possible from striae and similar defects shall be used.

5.2 Condenser

The condenser shall be a glass water-cooled reflux type, of the design and dimension shown in **Fig. 2**.

In the construction of the condenser, the following points should be noted.

a) The joints A and B should be neatly finished, in particular the bore at B should have the minimum disturbance.

b) The shoulder above the cone of the B 19 joint at C should be elongated as shown in **Fig. 2**, thus avoiding a sharp re-entrant shape which may restrict the free flow of liquid down the inner wall.

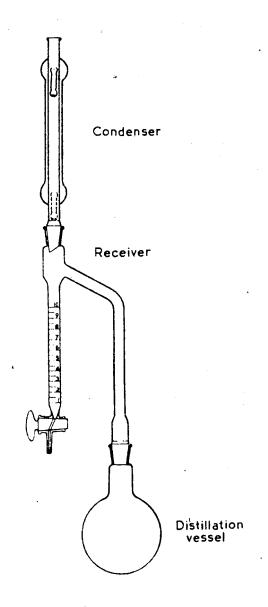
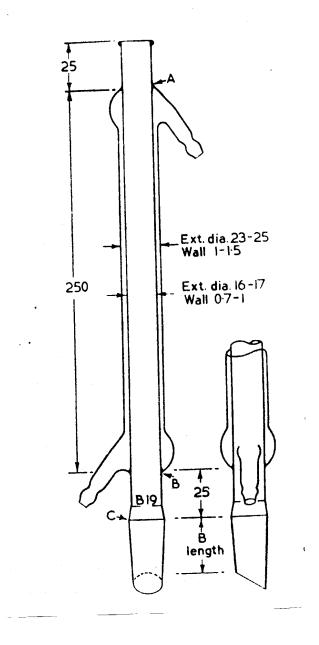


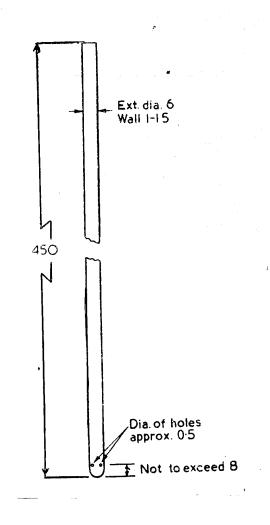
FIGURE 1 – Typical assembly of Dean and Stark apparatus

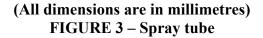


(All dimensions are in millimetres) FIGURE 2 – condenser

5.3 Spray tube

The spray tube, shall be sealed at one end and shall have four small holes spaced regularly round its circumference near the closed end, and shall be of the form shown in **Fig. 3**.





5.4 2-ml Receiver

5.4.1 The receiver shall be made of hard resistance glass, well annealed and as free as possible from striae and similar defects, provided with ground glass joints, and of shape and dimensions given in **Fig. 4**.

5.4.2 The graduated portion shall have a capacity of 2-ml at 27 ! 2 0 C when filled to the highest graduation mark. The scale shall cover the range of 0.1 ml to 2 ml and shall be divided into intervals of 0.05 ml. The numbered graduation marks shall be carried completely round the tube. The graduation marks corresponding to 0.15 ml, 0.25 ml, 0.35 ml and so on up to and including 1.95 ml shall be carried half way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end and beyond the shortest graduation marks. The error at any point on the scale shall not exceed ! 0.02 ml and the difference between the errors at any two points shall not exceed 0.02 ml.

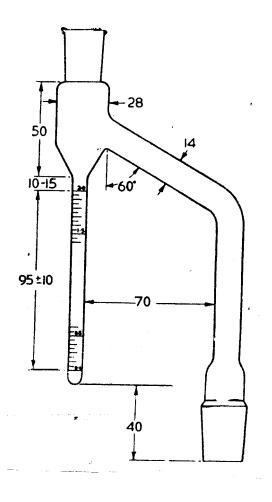


FIGURE 4 – 2-ml receiver (All dimensions are in millimetres)

6 **REAGENTS**

6.1 Toluene, the toluene used shall at $27 \cdot 2 \,^{0}$ C be free from undissolved water and other separated impurities, when viewed by transmitted light.

6.2 Ethyl acetate or amyl acetate

7 SAMPLE

- 7.1 Sample for testing shall be obtained in accordance with SLS 523.
- 7.2 Samples obtained as in 7.1 shall be prepared for testing as prescribed in SLS 1256 Part 1.

8 **PROCEDURE**

8.1 Weigh to the nearest 0.1 g about 100 g of the material under test into the flask (m_o) , add 100 ml toluene and 1 ml of amyl or ethyl acetate and mix the contents of the flask well Pour toluene into the receiver up to the level of the side tube. Connect the flask to the apparatus and immerse it in an oil bath containing sufficient heavy mineral oil to cover the flask nearly to the neck.

8.2 Heat the oil bath to such a temperature as to keep the contents of the flask boiling briskly, continue the distillation until no further water collects in the receiver.

8.3 Remove the persistent ring of condensed water in the condenser tube, if any, by increasing the rate of distillation by a few drops per second. Wash droplets of water which adhere to the lower end of the condenser tube into the receiver with toluene, using the spray tube.

8.4 Read off the amount of water collected in the receiver to the nearest 0.05 ml(v η m₁). Report the result as the percentage of water in the material.

9 CALLCULATION

Water content, per cent by mass $\frac{m_1}{m_0} \times 100$

where,

m₁ is the mass in grammes, of water collected

 m_0 is the mass in grammes, of the sample taken for test

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