SRI LANKA STANDARD 48 : 1999

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METHOD FOR DETERMINATION OF CERTAIN WATER OR ALKALI SOLUBLE ADDITIVES IN CELLULOSE OR SYNTHETIC FIBRES,YARNS AND FABRICS OR YARNS AND FABRICS MADE FROM BLENDS OF SUCH FIBRES

(FIRST REVISION)

SRI LANKA STANDARDS INSTITUTION

METHOD FOR DETERMINATION OF CERTAIN WATER OR ALKALI SOLUBLE ADDITIVES IN CELLULOSE OR SYNTHETIC FIBRES,YARNS AND FABRICS OR YARNS AND FABRICS MADE FROM BLENDS OF SUCH FIBRES (FIRST REVISION)

SLS 48 : 1999

Gr. 6

SRI LANKA STANDARDS INSTITUTION
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SRI LANKA

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

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

SRI LANKA STANDARD METHOD FOR DETERMINATION OF CERTAIN WATER OR ALKALI SOLUBLE ADDITIVES IN CELLULOSE OR SYNTHETIC FIBRES, YARNS AND FABRICS OR YARNS AND FABRICS MADE FROM BLENDS OF SUCH FIBRES (FIRST REVISION)

FOREWORD

This standard was approved by the Sectoral Committee on Textiles, Clothing and Leather and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1999-11-11.

This standard was first published in 1969. A revision was considered necessary to update the test method.

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

In the revision of this standard the assistance derived from the following publication is gratefully acknowledged:

BS 4032 : 1978 (1995) British Standard Method of Test for the Determination of certain water or alkali soluble additives in cellulose or synthetic fibres, yarns and fabrics or yarns and fabrics made from blends of such fibres.

1 SCOPE

1.1 This standard describes a procedure for the quantitative removal and determination of fatty matter, size and filling from cotton, viscose and synthetic fibres, yarns and fabrics in which the adhesive is starch, a chemically degraded starch, vegetable gum or some other water or alkali soluble polymer.

1.2 It is not applicable where the starch has been made insoluble as in formaldehyde containing finishes.

1.3 The standard is not intended for use in determining small amounts of residual starch or fatty materials.

2 PRINCIPLE

A weighed amount of the dry sized material is extracted with light petroleum and reweighed accurately. The starch and gum are then removed by treating the extracted specimen first in a suitable enzyme solution, and then in alkaline soap solution followed by hot water. The enzyme treatment is omitted when starch is shown to be absent when tested with iodine. The size content is the loss in dry mass, corrected for losses suffered by corresponding unsized material, and expressed as a percentage of the dry mass of the original unsized material.

3 APPARATUS

3.1 Sintered disk filter crucibles, porosity 1, of capacity not less than 60 ml, fitted with a ground glass stopper or other suitable cover.

3.2 Suitable ignition crucibles and covers

3.3 Muffle furnace, or other equipment for heating crucibles to about 700°C.

3.4 Soxhlet apparatus

3.5 Ventilated oven

3.6 Desiccator, containing self indicating silica gel.

3.7 Analytical balance, accurate to 0.0001g.

3.8 Cellulose extraction thimble, 25 mm X 80 mm.

4 REAGENTS

4.1 light petroleum, boiling range 40°C to 60°C.

4.2 Amylase solution, containing 0.5 per cent of active bacterial amylase (See Note 1) specially prepared according to manufacturers instructions, also containing suitable amount of nonionic wetting agent. (See Note 2).

NOTES

1 It is essential that the hydrolysis be performed at the optimum temperature and pH, and with such additions as are required for maximum activity.

2 Wetting agent should not be an enzyme inhibitor.

4.3 Alkaline soap solution, containing 5g/l of sodium oleate or other suitable high titre soap and 2.5g/1 of anhydrous sodium carbonate, filtered.

4.4 Distilled or deionized water

5 TEST SAMPLE AND TEST SPECIMEN

Take a sample representative of the bulk, sufficient for two or more test specimens, each approximately 2g in mass. Cut these specimens into pieces of convenient size for treatment in the crucible.

6 TEST PROCEDURE

6.1 General

6.1.1 Conduct all drying operations for 4h to 16h at 105 $\pm$ 3°C in a ventilated oven with the oven door closed throughout.

6.1.2 Dry the filter crucibles with its stopper or cover beside it. After drying,cover each crucible and transfer it quickly to a desiccator putting no more than two crucibles in each desiccator.

6.1.3 Conduct all cooling operations for 30 minutes with the desiccator beside the balance.

6.1.4 After cooling, complete the weighing of crucible within two minutes of its removal from the desiccator. Weigh to an accuracy of 0.0001 g.

6.1.5 Do not handle the crucibles with bare hands during the drying, cooling and weighing operations.

6.2 Procedure

6.2.1 Place about 2 g of the prepared specimen in cellulose extraction thimble. Place in a Soxhlet appratus and extract with light petroleum for 1 h at a minimum siphoning rate of 6 cycles per hour. Evaporate the extract in a tared 100-ml flask and determine the mass of residue. Transfer the extracted specimen to the tared crucible, dry the crucible and specimen and determine the mass of the dry specimen.

6.2.2 If starch is shown to be present by the iodine test, transfer the dried specimen to a 250 ml flask and add 100 ml of bacterial enzyme at 70 °C to the flask. Maintain this temperature for 30 min, shaking the contents of the flask by any convenient means, continuously or at 5 min intervals, during this time. Decant the solution through the filter crucible, add a fresh 100 ml portion of enzyme solution at 70 °C to the flask and

repeat the extraction. Decant the second portion of solution through the filter crucible. Transfer the enzyme-treated specimen to a 400 ml beaker with 200 ml of alkaline soap solution.

If starch is not present, transfer the dried specimen from the filter crucible to a 400 ml beaker and add 200 ml of the alkaline soap solution.

Heat to 70 °C with stirring. Maintain this temperature for 30 min. Collect the desized specimen in the filter crucible with suction. Wash with 3 litres of hot tap water, then with 500 ml of distilled water. If the washings are opalescent with mineral filling continue till clear.

6.2.3 Dry the crucible and contents and determine the mass of the desized specimen.

6.2.4 If the sample is assumed to contain substantial amount of insoluble mineral filling, for example china clay, transfer the weighed desized specimen to an ignition crucible, which has been previously ignited with its lid at 700 °C, cooled and weighed. Place the open crucible at the front of the muffle furnace and burn off the fibre while manipulating the crucible lid to prevent the fibre from flaming. Move the crucible to the interior of the furnace, without the lid, and continue the combustion at 700 °C, until no carbonaceous residue remains.

Cool and weigh the crucible and its content, and calculate the mass of ash. Correct the mass of the desized specimen by subtracting the mass of ash.

7 CALCULATION AND EXPRESSION OF RESULTS

Fatty matter and adhesive/filler, if any, may be calculated and reported separately as a percentage of the dry mass of the original unsized material.

$$\text{Total size, per cent by mass} = \frac{100\,(m + F - fm_d)}{fm_d}$$

where,

m is the dry mass of the sized specimen, after solvent extraction;
F is the mass of fatty matter extracted by solvent (See **6.2.1**);
m_d is the dry mass of the desized specimen, corrected where necessary by substracting the mass of residual mineral filler as determined by ashing the residue (See **6.2.4**); and
f is a factor for converting the mass of the dry desized specimen into the mass of the dry specimen before the addition of size.

If an unsized control is available, determine the value of f experimentally using the test procedure given in **6.2** as the ratio;

$$\frac{\text{Dry mass of unsized control}}{\text{Dry mass of control after treatment}}$$

Otherwise f may be assumed to have the following values :

1.03 for grey cotton
1.02 for unbleached viscose
1.00 for all other fibres

For fibre mixtures and union fabrics containing a first component for which the factor is f_1, and a second component for which the factor is f_2, the factor f_m for the mixture of union is

$$f_m = Pf_1 + (1 - P)f_2$$

where,

P is the fractional proportion by mass of the first component in the mixture or union. Express the result to the nearest 0.1 per cent.

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SRI LANKA STANDARDS INSTITUTION

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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.

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The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

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