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METHOD FOR THE DETERMINATION OF  
GELATINE AND OIL SIZE IN VISCOSE  
RAYON, ACETATE YARN AND FABRIC

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BUREAU OF CEYLON STANDARDS



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**C.S. 174 : 1972**

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# CEYLON STANDARD METHOD FOR THE DETERMINATION OF GELATINE AND OIL SIZE IN VISCOSE RAYON, ACETATE YARN AND FABRIC

## FOREWORD

This Ceylon Standard has been prepared by the drafting Committee on test methods for Textiles. It was approved by the Textiles Divisional Committee of the Bureau of Ceylon Standards, and was authorised for adoption and publication by the Council of the Bureau on 30th November, 1972.

It is common practice for warp yarns of continuous filament viscose rayon or cellulose acetate to be sized with a solution of gelatine in which a non-volatile oil has been emulsified. Unsized yarn loses about 1 per cent in mass in the de-sizing process, and a correction for the loss, in the form of a factor has to be applied to the mass of the residue. The correction can be determined if a specimen of the corresponding unsized yarn is available. In the absence of such a specimen the values for the correction factor are assumed.

All quantities and dimensions specified in this standard are given in the International System of Units (SI). In reporting the result of 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 C. S. 102 : Ceylon Standard on Presentation of Numerical Values.

In the preparation of this standard, considerable assistance derived from publications of the British Standards Institution is gratefully acknowledged.

## 1. SCOPE

This Ceylon Standard describes a procedure for the removal of size from viscose rayon and acetate yarn and fabric in which the size is based on gelatine and a non-volatile non drying oil.

## 2. PRINCIPLE

A weighed amount of the dry sized material is extracted with light petroleum, and the gelatine is removed by hydrolysis in a suitable enzyme solution. The loss in mass is determined after washing and drying the specimen, and expressed as a percentage of the mass of the original (unsized) material.

### 3. APPARATUS

- 3.1 Sintered glass filtering crucibles, porosity 1, capacity at least 60 ml fitted with a ground glass stopper or other suitable cover.
- 3.2 Suitable ignition crucibles and covers.

### 4. REAGENTS

- 4.1 Light petroleum, distillation range 40°—60°C.
- 4.2 **Millon's reagent**—Dissolve 2g of mercury in 2 ml of concentrated nitric acid at room temperature. Add 2ml of cold water. If yellow turbidity develops, stir in a few drops of nitric acid until the solution clears.
- 4.3 **Proteolytic enzyme solution\***—pH 6.0-7.0 freshly prepared and containing 0.5 percent by mass of an active preparation.

### 5. TEST SAMPLE AND TEST SPECIMEN

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

### 6. TEST PROCEDURE

Place the specimen in a crucible, previously weighed after being heated at  $105 \pm 3^\circ\text{C}$  until constant mass\*\* has been attained. Dry at  $105 \pm 3^\circ\text{C}$  to constant mass and calculate the mass of the dry specimen.

Place the crucible in a Soxhlet apparatus, with its side above the level of siphon, and extract with light petroleum for one hour at a minimum siphoning rate of 1/360Hz\*\*. Remove the crucible from the extractor and allow the solvent to evaporate. Attach the crucible to a Buchner flask and wash the extracted specimen by drawing about 100ml of water through it, without allowing it to dry completely. Remove the crucible and place it in a beaker containing sufficient proteolytic enzyme solution at 50°C to cover the specimen but not to submerge the crucible completely. Lift the

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\*Gelatase is suitable. Proteolytic enzymes from other sources may replace Gelatase. If other enzymes are used, the hydrolysis must be performed at the optimum temperature and pH and with such additions as are required for maximum activity. The conditions may differ from those suitable for Gelatase.

\*\*The crucible and specimen shall be deemed to have attained constant mass when, after being heated for 3 hr at  $105 \pm 3^\circ\text{C}$ , cooled in a desiccator and then re-heated at the same temperature for not less than one hour and cooled, the change in mass does not exceed 0.002g.

1 Hz (hertz = 1 cycle per second)

crucible above the liquid level at intervals of about five minutes during one hour and allow the liquid to drain back into the beaker before lowering the crucible. Re-attach the crucible to the Buchner flask and wash with three successive portions of hot water, drain with suction after each wash. Test the washed specimen by spotting with Millon's reagent. If now gelatine remains wash again with hot water and drain with suction. If gelatine is present repeat the enzyme treatment until test with millon's reagent is negative. Dry the crucible and desized specimen at  $105 \pm 3^{\circ}\text{C}$  to constant mass and determine the mass of the desized specimen.

## 7. CALCULATION AND EXPRESSION OF RESULTS

Express the contents of total dry size as a percentage of the dry mass of the original unsized specimen.

The percentage of total size is given by :

$$\text{Total size percentage} = \frac{100 (M - F M_d)}{F M_d}$$

Where

**M** is the dry mass of the sized specimen, and

**M<sub>d</sub>** is the dry mass of desized specimen, and

**F** is a factor 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, the value of F should be determined by the method used to desize the test specimen, as the ratio.

**Dry mass of unsized control**

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**Dry mass of control after treatment**

Otherwise F may be assumed to have the value 1.01.





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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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