## International Standard



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО CTAHДАРТИЗАЦИИ⊕ORGANISATION INTERNATIONALE DE NORMALISATION

### Surface active agents — Mercerizing agents — Evaluation of the activity of wetting products for mercerization by determination of the shrinkage rate of cotton

Agents de surface — Adjuvants de mercerisage — Évaluation de l'activité des produits mouillants pour mercerisage par détermination de la vitesse de rétraction du coton

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#### **Foreword**

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International Standard ISO 6836 was developed by Technical Committee ISO/TC 91, Surface active agents, and was circulated to the member bodies in March 1982.

It has been approved by the member bodies of the following countries:

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| Egypt, Arab Rep. of | Italy       | Romania     |
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Belgium

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# Surface active agents — Mercerizing agents — Evaluation of the activity of wetting products for mercerization by determination of the shrinkage rate of cotton

#### 0 Introduction

The effectiveness of strongly alkaline mercerizing liquor used for mercerization (or causticization) depends, amongst other things, on the rapidity with which the sodium hydroxide solution impregnates the material and penetrates into the cellulose fibre the swelling of which causes shrinkage in the longitudinal direction of the fibre under tension which is transmitted to the material and allows the mercerization effect as such to be achieved. In order to shorten the time required for penetration by the sodium hydroxide solution, special wetting agents are added to the mercerizing or causticizing liquor.

#### 1 Scope and field of application

This International Standard specifies a method for evaluating the activity of wetting products for mercerization by determination of the shrinkage rate of cotton threads.

The method is only applicable to mercerizing or causticizing baths.

#### 2 Reference

ISO 607, Surface active agents and detergents — Methods of sample division.

#### 3 Definitions

For the purpose of this International Standard, the following definitions apply.

- **3.1** mercerizing agent: A product used to improve the wetting power of mercerizing liquor and thus to speed up its uniform penetration into the fibres.
- **3.2 shrinkage rate**: Modification of the length of the cotton yarn, expressed in millimetres per second under the influence of the mercerizing liquor.

#### 4 Principle

Immersion in the liquor containing the agent being studied of a hank of smooth cotton held by a hook and with a weight fixed on the end of it.

Determination of the shrinkage of the hank at different intervals of time using one of the two pieces of apparatus described.

#### 5 Reagents

During the test, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

**5.1** Sodium hydroxide solution,  $\varrho_{20}$  1,24 g/ml.

Dissolve 270 g of sodium hydroxide in 1 litre of water.

NOTE — For each agent to be examined 1,5 litres of sodium hydroxide solution are required.

#### 6 Apparatus and material

**6.1** Cotton yarn, the type and designation of which are exactly defined in the test report. For comparative tests, see the note.

NOTE — As the shrinking ability depends upon the type of raw cotton, the fineness of the yarn (tex number), its twist and also, possibly, on the type of twist, comparative tests should only be carried out on one and the same yarn. Tests with different yarns of the same type but from different consignments or from different sources may give divergent values.

#### 6.1.1 Preparation of the hanks of yarn

Using a winder with a perimeter of 1 m under the lowest possible tension, make the number of revolutions necessary to obtain a test hank of mass 1  $\pm$  0,2 g. Then remove this hank from the winder, still without tension, and fold it in two so that it is approximately 24 cm in length.

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#### 6.1.2 Conditioning of the hanks of the yarn

Store the hanks prepared according to 6.1.1 for at least 24 h before testing in a standard atmosphere at a temperature of 20  $\pm$  2 °C and a relative humidity of 65 %  $\pm$  2 %.

#### 6.2 Apparatus for measuring shrinkage.

#### 6.2.1 Hintzmann apparatus

The apparatus (see figure 1) consists of a plastics support  $\bigcirc$ 1, fitted with a device  $\bigcirc$ 2 for holding a glass cylinder  $\bigcirc$ 6 closed on one side and intended to contain the mercerizing liquor. Adjustable screws  $\bigcirc$ 5 at the base of the support keep it correctly in the vertical position. The glass cylinder  $\bigcirc$ 6 is bunged at the top by a removable fitted plastics lid  $\bigcirc$ 7. To this lid  $\bigcirc$ 7 is fitted a hook  $\bigcirc$ 8 which holds the hank of yarn  $\bigcirc$ 9 weighted with a weight  $\bigcirc$ 9 of mass 10  $\pm$  0,1 g. To the support  $\bigcirc$ 1 is fixed a ring  $\bigcirc$ 3 to hold the lid  $\bigcirc$ 7 and also a millimetre scale  $\bigcirc$ 4 to measure the length of the hank before and during the test.

The material of which the apparatus is made shall be alkali resistant

#### 6.2.2 Servo apparatus

The apparatus (see figure 2) consists of a support ① comprising a vertically movable stem the height of which can be adjusted with the screw ③, an arm ④ with two right-angle bends integral with the stem ② to which is fixed a hook ⑤ and a guiding pulley ⑥, and a disc ⑦ graduated in percentage of shrinking with a moving needle ⑧ which can be reset to zero. At the top of the arm ④ a guide ⑪ is attached through which passes a small metal plate ⑨ which moves with virtually no resistance and is fitted with a hook. At the foot of the support ① there is a small tray ⑫ containing filter paper to absorb drops of mercerizing liquor if necessary. The hank is placed under tension by means of a counterweight ⑪ of mass approximately 50 g.

NOTE — The choice of the mass of the weight 11 depends on the nature of the yarn and its value should be stated in d) of the test report (clause 10).

#### 6.3 Stop-watch.

#### 7 Sampling

The laboratory sample of mercerizing agent shall be prepared and stored in accordance with the requirements of ISO 607.

#### 8 Procedure

#### 8.1 Test portion

Weigh, to the nearest 0,01 g, the quantity of the laboratory sample required to obtain a concentration of 3 g/l after dissolution in the sodium hydroxide solution (5.1). Carry out the dissolution immediately before the test.

#### 8.2 Determination

#### 8.2.1 General

For each test, carry out a series of five measurements. These tests shall be carried out first with a solution prepared no more than 15 min in advance and the other tests shall then follow very quickly one after the other.

The temperature of the solution shall be 20  $\pm$  2 °C. It is thus advisable to bring the solution under examination to a temperature of 18 °C before putting it into the glass cylinder so that it is at the temperature required during the tests.

## 8.2.2 Determination of the rate of shrinkage (Servo apparatus)

Bring the vertical stem 2 to its maximum height, fix the hank (6.1.1) to the hooks 5 and 9 and set the needle 8 to zero.

Place the glass cylinder, filled with the mercerizing liquor under examination, on the tray (12), lower the vertical stem (2) until the hank is totally immersed and fix in position by means of the adjustable screw (3). Start the stop-watch (6.3) immediately and record the time at which shrinkage reaches a value of 3 %, 6 %, 9 %, 12 %,...

# 8.2.3 Determination of the time corresponding to 50 % of the total shrinkage and of the shrinkage as a percentage of the total shrinkage (Hintzmann apparatus)

Place the glass cylinder 6 in the supporting device 2 and fill it to just over the zero mark with the mercerizing liquor under examination. Hook the hank (6.1.1) onto the hook 8, and attach the weight 9 to the lower part of the hank.

Put the whole of this assembly into the support ① so that the lid ⑦ is firmly fixed in the support. Read the initial length of the hank on the millimetre scale level with the lower edge of the weight.

Then remove the lid 7 with the hank and the weight on the end of it and, with the weight still attached, put it into the glass cylinder 6, containing the mercerizing liquor under examination; do this within 1 s.

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Start the stop-watch (6.3) as soon as immersion has taken place.

Note the shrinkage of the hank at intervals of 10 s up to 90 s, by reading the lengths on the millimetre scale level with the lower edge of the weight. Finally, note the shrinkage value after 300 s (5 min). When interpreting the results, consider this value as the total shrinkage.

#### 9 Expression of results

#### 9.1 Rate of shrinkage

The rate of shrinkage for the mercerizing agent under examination, expressed in millimetres per unit of time, is calculated by the tangent of the linear part of the curve obtained by plotting the values of the arithmetic mean of the percentage of shrinkage of the five series of tests as ordinates and the corresponding values of the time as abscissae.

## 9.2 Time corresponding to 50 % of the total shrinkage

From the arithmetic mean value of the values obtained during the five series of tests, the percentage of shrinkage at a given time in relation to the total shrinkage is given by the formula

$$\frac{l_{\rm o}-l_{\rm t}}{l_{\rm o}-l_{\rm \infty}}\times 100$$

where

- $l_{\rm o}$  is the length, in millimetres, of the hank with the weight attached, before the test;
- $l_t$  is the length, in millimetres, of the hank with the weight at a given time t;
- $l_{\infty}$  is the length, in millimetres, of the hank with the weight after 5 min.

The time corresponding to 50 % of the total shrinkage is determined by means of a logarithmic scale by plotting a graph, having the values of the percentage of shrinkage as ordinates and the corresponding values of the time as abscissae.

The perpendicular dropped on the axis of abscissae from the point of intersection of the straight line with the ordinate corresponding to 50 % of the total shrinkage shows the time in seconds required to reach 50 % of the total shrinkage.

#### 9.3 Percentage of shrinkage at a given time

The percentage of shrinkage after a given time is given by the formula

$$\frac{l_{\rm o}-l_{\rm t}}{l_{\rm o}-h}\times 100$$

where

- $l_{\rm o}$  is the length, in millimetres, of the hank with the weight attached, before the test;
- $l_t$  is the length, in millimetres, of the hank with the weight at a given time t;
- h is the height, in millimetres, of the weight with the hook.

#### 10 Test report

The test report shall contain the following information:

- a) all the information necessary for the complete identification of the sample;
- b) the type and designation of the cotton yarn used;
- c) the concentration of the solution;
- d) the apparatus used;
- e) the reference of the method used (reference to this International Standard);
- f) the results obtained and the method of expression used :
  - 1) percentage of shrinkage at a given time in relation to the total shrinkage,
  - 2) percentage of shrinkage at a given time,
  - 3) time corresponding to 50 % of the total shrinkage,
  - 4) rate of shrinkage;
- g) any operational details not provided for in this International Standard or in the International Standard to which reference is made or regarded as optional, as well as any incidents which may have influenced the results.

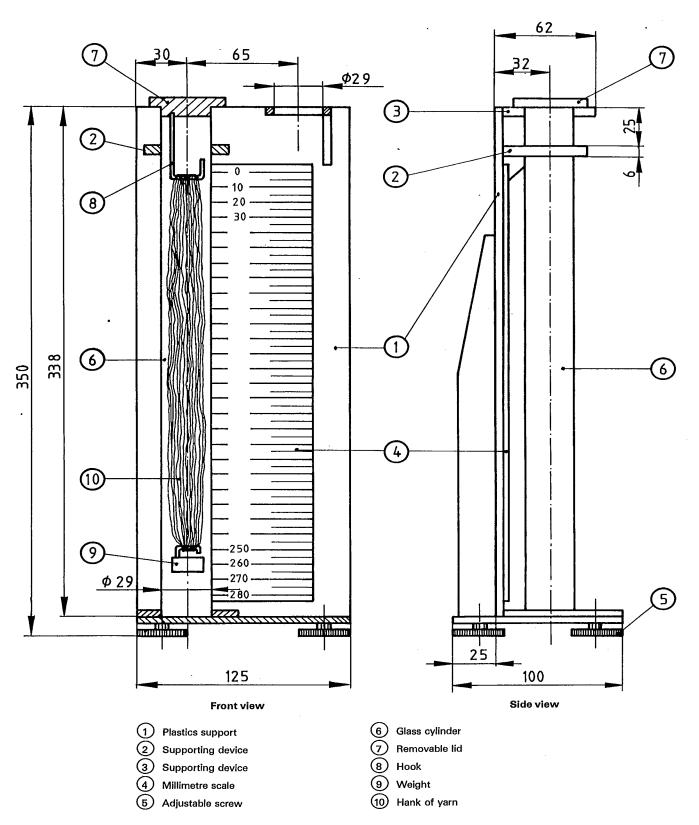


Figure 1 — Hintzmann apparatus

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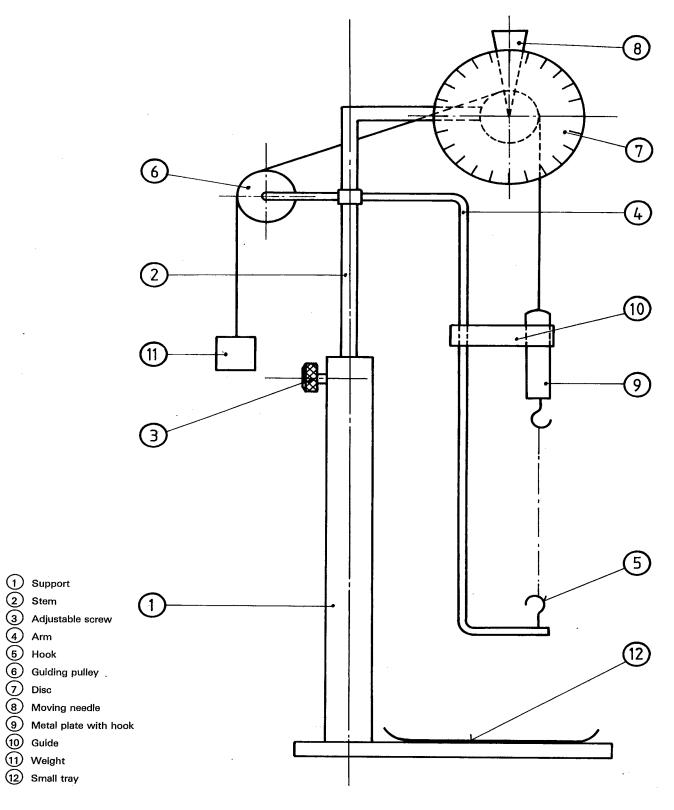


Figure 2 — Servo apparatus