BS 3584 : 1989

British Standard Method for

Determination of solubility of wool in urea-bisulphite solution

Méthode de détermination de la solubilité de la lame dans une solution d'urée et de bisulfite

Verfahren zur Bestimmung der Harnstoffbisulfit-Löslichkeit von Wolle

Foreword

This British Standard has been prepared under the direction of the Textiles and Clothing Standards Policy Committee and is a revision of BS 3584 : 1963, which is withdrawn. The appendix describing a method for determining acid content formerly given in BS 3584 : 1963 has been published separately as BS 6981 : 1988 because there may be other reasons for wishing to determine acid content other than correcting bisulphite solubility values, e.g. when investigating finishing faults, cases of skin irritation.

The solubility of wool in urea-bisulphite solution provides an index of the extent of the change in its chemical properties brought about by certain agencies. Treatment in neutral or alkaline solution, or steaming wool in a neutral or alkaline condition, usually leads to a decrease in solubility. Hence the method is particularly useful for investigating setting processes. Dry heating or treatment with cross-linking agents also causes the solubility to decrease, whereas oxidation or acid-dyeing increases the solubility. The more severe the treatment, the greater is the change in solubility. The test is most useful when an untreated sample is available and when the treatment of the sample under test is known, i.e. as a method of control.

When the sample has been treated by two agencies having opposite effects on the solubility, the interpretation of the results, even when an untreated control sample is available, is difficult, and other tests are necessary to supplement the information.

At the time of publication of this British Standard, no corresponding international standard exists.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

British Standards Institution

Method

1 Scope

This British Standard describes a method for determination of the solubility of wool in urea-bisulphite solution. This procedure is applicable to wool textiles in any form, i.e. loose fibre, sliver, roving, yarn or fabric, but is not applicable if the specimen has been dyed with any metallized dye.

NOTE. The titles of the publications referred to in this standard are listed on page 3.

2 Principle

A test specimen is immersed in a solution containing urea and disodium disulphite (also known as sodium meta-bisulphite) of known concentration and specified composition, under specified conditions of time, temperature and volume. The loss in mass is determined as the difference between the dry masses of the test specimen before and after treatment.

3 Reagents

NOTE. All reagents should be of analytical reagent quality.

3.1 *Urea-bisulphite solution,* freshly prepared on day of use, containing 50 g of urea, 3 g of disodium disulphite and 2 mL of 5 mol/L sodium hydroxide per 100 mL.

Dissolve the urea in boiling water, add the disodium disulphite, cool, add 2 mL sodium hydroxide solution and make-up to volume. Check the pH value using a glass electrode pH meter and adjust to 7.0 ± 0.1 if necessary.

3.2 *Urea solution,* containing 25 g urea per 100 mL. Dissolve 250 g urea in 1000 mL of water.

3.3 *Water,* complying with grade 3 of BS 3978.

3.4 *Petroleum spirit,* boiling range 40 °C to 60 °C.

4 Apparatus

4.1 *Soxhlet extraction apparatus* complying with BS 2071.

4.2 *Water bath,* or other means for controlling the temperature of the flask and contents at 65 ± 0.5 °C.

4.3 *Conical flasks,* 250 mL capacity, fitted with stoppers.

4.4 *Sintered-glass filter crucibles,* 30 mL capacity, of porosity P 160 in accordance with BS 1752.

NOTE. If possible, these crucibles should have ground glass stoppers

4.5 *Filter-flask, filter-pump and adaptor.*

4.6 *Ventilated oven,* for drying specimens at 105 ± 3 °C.

4.7 *Stoppered weighing bottles.*

4.8 *Balance,* accurate to ± 0.0002 g.

4.9 *Desiccator.*

5 Test specimens

Take a sample representative of the bulk and not less than 10 g sufficient to provide wool for the following test specimens:

(a) one test specimen weighing approximately 1 g for determining the dry mass (see **6.2**);

(b) two test specimens each weighing approximately 1 g for determining the solubility in urea-bisulphite (see **6.3**).

NOTE 1. If it is suspected that the sample contains acid (see **6.4**) two further test specimens each weighing approximately 2 g may be taken from the sample for determining acid content by the method described in BS 6981.

NOTE 2. Useful information on sampling is given in BS 2545.

NOTE 3. The precision of results may be quite high given a very uniform sample, but, for example, in testing dyed loose wool the variations within the batch could be ± 2 % or more and fresh test specimens may be needed.

6 Procedure

6.1 Preparation of sample

Extract the sample in the Soxhlet extraction apparatus (**4.1**) using petroleum spirit (**3.4**) for 1 h at a minimum rate of six cycles per hour. Allow the petroleum spirit to evaporate and then remove all vegetable and other obvious foreign matter. Disintegrate the sample into short lengths of approximately 10 mm and allow them to come to equilibrium with the laboratory atmosphere.

6.2 Determination of dry mass

Place the 1 g test specimen (see clause **5**) in a weighing bottle (**4.7**) and dry it in the ventilated oven (**4.6**) at 105 ± 3 °C. Stopper the bottle, cool it in the desiccator (**4.9**) and weigh it. Repeat these drying, cooling and weighing operations until the results of two consecutive weighings do not differ from each other by more than 0.0005 g, i.e. the mass is virtually constant.

Remove the test specimen, weigh the weighing bottle and hence calculate the dry mass of the test specimen.

6.3 Determination of solubility in urea-bisulphite

Measure 100 mL portions of the urea-bisulphite solution (**3.1**) into separate flasks (**4.3**), stopper loosely, and fix the flasks in the water-bath (**4.2**) by any suitable means so that the level of the water outside the flasks is at least 50 mm higher than the level of the solution inside.

NOTE. This procedure is essential for precise control of temperature.

When the temperature of the urea-bisulphite solution reaches 65 ± 0.5 °C, introduce separate test specimens of known mass (see clause **5**) into the flasks, replace the stoppers and gently shake the flasks to ensure complete wetting of the test specimens. Shake the flasks gently again after 15, 30 and 45 min, the time of shaking not to exceed 5 s on each occasion.

After 1 h transfer the contents of each flask to separate sintered-glass filter crucibles (**4.4**) of known mass and drain the crucibles by suction. Wash any fibrous material remain-