BS 3568 : 1988
UDC [677.31.01 : 541.3]

British Standard Method for

Determination of solubility of wool in alkali

Méthode de détermination de la solubilité de la laine dans l'alcali

Verfahren zur Bestimmung der Löslichkeit von Wolle in Alkalien

1989

British Standards Institution

Foreword

This British Standard has been prepared under the direction of the Textiles and Clothing Standards Committee and is a revision of BS 3568 : 1962, which is withdrawn. The appendix, which described a method for determining acid content, has been omitted and is published separately as BS 6981 : 1988. This is because there may be reasons for wishing to determine acid content other than for correcting alkali solubility values, e.g. when investigating finishing faults, cases of skin irritation, etc.

The solubility of wool in alkali provides a useful index of the extent of the change in its chemical properties brought about by certain agencies. Treatment with acids or with oxidizing or reducing agents, or exposure to heat or light, causes an increase in the solubility, whereas treatment with mild alkali, as used in normal processing, or with cross-linking agents causes the solubility to decrease. The more severe the treatment, the greater is the change in solubility. The test is most useful when an untreated control sample is available and when the nature of the treatment of the sample under test is known.

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.

This British Standard is technically equivalent to ISO 3072-1975.

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

Contents

Method

1 Scope

This British Standard describes a method for determination of the solubility of wool in alkali. 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 the inside back cover.

2 Principle

A test specimen is immersed in a solution of sodium hydroxide of known concentration 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

3.1 *Sodium hydroxide solution, c* (NaOH), 0.1 mol/L.

3.2 *Acetic acid solution.* Dilute 10 mL of glacial acetic acid, approximately 99.7 % (*m/m*), ρ approximately 1.06 g/mL, to 1 L with water (**3.3**).

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*, thermostatically controlled at 66 ± 0.5 °C.

4.3 *Conical flasks*, 100 mL capacity, fitted with stoppers.

4.4 *Sintered-glass filter crucibles*, 30 mL capacity, of porosity P160 complying 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*, for weighing the test specimens.

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 alkali (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, with samples from bleached wool, or with samples having an alkali solubility of 20.0 % (*m/m*), the variations within the batch may 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**) with 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 it to come to equilibrium within 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 alkali

Measure 100 mL portions of the sodium hydroxide 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 sodium hydroxide 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 exceeding 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 remaining in each flask into the crucibles with water (**3.3**). Wash each residue in each crucible six times with water (**3.3**), draining completely after each wash, and release the suction.

Fill each crucible twice successively with the acetic acid solution (**3.2**). Allow to stand for 1 min and drain each crucible by suction. Finally, wash each residue six times

with water (3.3), draining completely between each wash. Dry the crucibles and contents at 105 ± 3 °C for 3 h minimum, cool them in the desiccator and weigh them.

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.

6.4 Determination of acid content

Determine the pH of the aqueous extract by the method described in BS 3266. If an aqueous extract of the material has a pH value less than 4.0, determine the acid content by the method described in BS 6981.

7 Expression of results

7.1 Samples not containing acid

The solubility in alkali, S, calculated as the loss in mass of the test specimen and expressed as a percentage of its calculated dry mass, is given by the equation:

$$S = \frac{m_1 - m_2}{m_1} \times 100$$

where

m_1 is the dry mass of the test specimen determined as described in 6.2 (in g);

m_2 is the dry mass of the residue determined as described in 6.3 (in g).

7.2 Samples containing acid

The corrected solubility in alkali, S', calculated as the loss in mass of the test specimen and expressed as a percentage of its calculated dry, acid-free mass, is given by the equation:

$$S' = (S - s)\left(\frac{100}{100 - s}\right)$$

where

S is the solubility in alkali calculated as described in 7.1;

s is the percentage of acid determined as described in BS 6981.

Express the results to one decimal place.

8 Test report

The test report shall include the following information:

(a) the number and date of this British Standard, i.e. BS 3568 : 1988;

(b) a description or reference of the sample tested;

(c) the individual results and their mean, expressed as in clause 7;

(d) any departure from the method described, e.g. owing to insufficient material being available.