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the direction of the Textiles and Clothing Standards Policy Committee and is a revision of BS 3584 : 1963, which is withdrawn.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-

ing in each flask into the crucibles with urea solution (10 mL each time) (3.2). Wash each residue in each crucible six times with water (3.3). Allow the liquid to stand in contact with the residue for about 15 s before draining completely after each wash, and release the suction.

Dry the crucibles and contents at 105 ± 3 °C for not less than 3 h and not more than 16 h, 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 value of the aqueous extract by the cold water 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 of urea-bisulphite, 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 urea-bisulphite, S', calculated as the loss in mass of the test specimen 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 uncorrected solubility in urea-bisulphite (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 3584 : 1989;

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

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

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

Publications referred to

BS 1752	Specification for laboratory sintered or fritted filters including porosity grading
BS 2071	Specification for Soxhlet extractors
BS 2545	Methods of fibre sampling for testing
BS 3266	Methods of test for determination of conductivity, pH, water-soluble matter, chloride and sulphate in aqueous extracts of textile materials
BS 3978	Specification for water for laboratory use
BS 6981	Method for determination of acid content of wool

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Method of test for the

Solubility of wool in urea-bisulphite solution

Source. BS 3584 : 1963

Introduction

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 is difficult, even when an untreated control sample is available, and other tests are necessary to supplement the information.

1. Scope

The method is applicable to all wool textiles in any form, viz. loose fibre, sliver, roving, yarn, or cloth.

2. Principle

The wool is immersed in a solution containing urea and sodium metabisulphite of 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 sample before and after treatment.

3. Apparatus and reagents

3.1 Apparatus. The following are required:

(1) *Water-bath*, thermostatically controlled at 66 ± 0.5 °C (see Note). To ensure uniform temperature, the water should be stirred.

(2) *Stoppered flasks*, with a working capacity of 50 ml, all of the same shape and wall thickness.

(3) *Sintered glass filtering crucibles*, 30 ml capacity, porosity 1 (see BS 1752). If possible, these crucibles should have ground-glass stoppers. If ground-glass stoppers are not available, each crucible should be covered with a watch-glass during cooling and determinations of mass.

NOTE. It is essential that the temperature of the urea-bisulphite solution should be maintained at 65 ± 0.5 °C throughout the period of the test. Experience has shown that this temperature is maintained inside the flask when the temperature of the water bath is maintained at 66 ± 0.5 °C.

3.2 Reagents. The following are required:

(1) *Urea-bisulphite solution*, freshly prepared on the day of use, containing 50 g urea, 3 g sodium metabisulphite and 2 ml 5N sodium hydroxide per 100 ml. All reagents should be of analytical grade. Dissolve the urea in boiling distilled water, add the metabisulphite, cool, add 2 ml sodium hydroxide solution, and make up to volume. Check the pH of the solution, using a glass-electrode pH meter, and adjust to 7.0 ± 0.1 if necessary.

(2) *Urea solution*, containing 25 g urea per 100 ml.

(3) *Light petroleum* (boiling range 40 °C to 60 °C).

4. Atmosphere for conditioning and testing

Because dry masses are determined, it is unnecessary to condition the test specimens. The test is conducted under ordinary room conditions.

5. Selection and preparation of test specimens

Take a sample representative of the bulk and sufficient to provide fat- and burr-free wool for the following test specimens:

one test specimen of mass 0.5 g $\pm$ 0.001 g for determining dry mass;

two test specimens of mass 0.5 g $\pm$ 0.001 g for determining solubility; and

two test specimens of mass 2 g $\pm$ 0.001 g for determining acid content (only required when a cold-water extract, liquor ratio 50:1, has a pH value under 4).

Extract the sample in a Soxhlet extractor (see BS 2071) with light petroleum for 1 h at a minimum rate of 6 cycles/h. Allow the petroleum to evaporate and then remove all vegetable and other obvious foreign matter. If the sample is yarn or cloth, disintegrate it into short lengths of yarn (approximately 1 cm); if it is sliver or loose wool, cut it into approximately 1 cm lengths. Allow the sample to come to equilibrium with the laboratory atmosphere.

6. Test procedure

6.1 Determination of mass. Determine the mass of the test specimens under the same conditions detailed in Clause 5.

6.2 Determination of dry mass. Dry one test specimen (of mass 0.5 g) in a weighing bottle at 105 $\pm$ 3 °C for 3 h. Stopper the bottle, cool it in a desiccator, and determine its mass. Remove the test specimen, determine the mass of the weighing bottle, and hence calculate the dry mass of each test specimen.

6.3 Determination of solubility. Measure 50 ml urea-bisulphite solution into a flask, stopper loosely, and fix the flask in the water-bath by any suitable means, so that the level of the water outside the flask is at least 5 cm higher than the level of the solution inside. This procedure is essential for precise control of temperature.

When the temperature of the urea-bisulphite solution reaches 65 $\pm$ 0.5 °C, introduce one test specimen (of mass 0.5 g) into the flask, replace the stopper tightly, shake the flask gently to ensure complete wetting of the test specimen, and replace it in the water-bath. After 60 min, transfer the contents of the flask to a filter crucible of known mass, at the same time draining the crucible by suction. Wash any fibrous material remaining in the flask into the crucible with urea solution. Wash the residue in the crucible three times with urea solution (10 ml each time) and then six times with distilled or de-ionized water, allowing the liquid to stand in contact with the residue for about 15 s before applying suction to drain completely. Dry the crucible and contents at 105 $\pm$ 3 °C for 3 h, stopper the crucible or cover it with a watch-glass, cool it in a desiccator, and determine its mass.

6.4 Determination of acid content. If a water extract (liquor ratio 50:1) of the sample has a pH value under 4.0, determine the acid content by the method given in Appendix A.

7. Calculation and expression of results

Calculate the solubility in urea-bisulphite as the loss in mass of the test specimen, expressed as a percentage of its calculated dry, fat- and acid-free mass. Average the results of two determinations.

(1) *Samples not containing acid* (extract pH greater than 4)

$$S_{ub} = \frac{M_1 - M_2}{M_1} \times 100$$

where S_{ub} is the percentage solubility in urea-bisulphite,
M_1 is the dry mass of the specimen (determined on another specimen as in **6.2**),
M_2 is the dry mass of the residue (determined as in **6.3**).

(2) *Samples containing acid* (extract pH less than 4)

$$S_{ub} = \frac{100\left(100\,\frac{M_1 - M_2}{M_1} - a\right)}{100 - a}$$

where a is the percentage content of acid in the specimen (determined as in Appendix A).

8. Test report

The report shall state:

(1) that the procedure was conducted in accordance with this standard method;

(2) which of the alternatives in Clause 7 was employed:

(3) the average percentage solubility in urea-bisulphite.

Appendix A

Method for determining acid content

A.1 Reagents

The following shall be used:

(1) *Pyridine solution*: 5 g pyridine (analytical grade) in 1 l of distilled water.

(2) 0.1N *sodium hydroxide solution*: this solution should be standardized by titration with standard potassium hydrogen phthalate solution.

(3) *Phenolphthalein indicator*: 0.5 g phenolphthalein in 95 ml ethanol and 5 ml of distilled water.

A.2 Test procedure

Place the two test specimens each of mass 2 ± 0.001 g (see Clause 5), into separate glass-stoppered conical flasks and pipette 100 ml of pyridine solution into each. Stopper each flask and either shake on a mechanical shaker for 1 h, or allow the flask to stand overnight after initial shaking to ensure complete wetting of the specimen. Decant the liquid from the wool, filtering through a plug of glass-wool to retain fibrous material. Pipette 50 ml of the filtered liquid into a conical flask, add three drops of phenolphthalein solution, and titrate with the sodium hydroxide until a faint pink colour persists.

A.3 Calculation and expression of results

Express the mass of acid as a percentage of the dry mass of the specimen.

$$a = \frac{v \times k \times n}{m}$$

where a is the content of acid (%),

m is the dry mass of the 0.5 g specimen (as determined in 6.2),

v is the volume (ml) of the sodium hydroxide solution required to neutralize 50 ml of pyridine extract,

n is the normality of the sodium hydroxide solution,

k is a constant which has the following values:

for calculating as sulphuric acid	2.45
for calculating as formic acid	2.3
for calculating as acetic acid	3.0

Average the results of the two determinations, calculated on the same basis.