BS 3424 : Part 19 : 1989

UDC [677.077.652 : 678.066.2] :
677.017 : 677.014.55 : 543.845

British Standard

Testing coated fabrics

Part 19. Methods 22A, 22B and 22C. Determination of sulphur staining

Essais des supports textiles revêtus
Partie 19. Méthodes 22A, 22B et 22C. Détermination de la décoloration par le soufre

Prüfung beschichteter Gewebe
Teil 19. Verfahren 22A, 22B und 22C. Bestimmung der Verfärbung durch Schwefelverbindungen

IMPORTANT NOTE. It is recommended that this Part be read in conjunction with the information in Part 0 'Foreword and general introduction'.

Foreword

Some coated fabrics contain substances in the coating, or the substrate, such as copper and lead pigments or compounds, that are liable to discolour as a result of reaction with sulphur or its compounds. Method 22A gives an indication of the discoloration of a coating that may eventually occur under the most severe conditions of use. This method is intended for use when the appearance or aesthetic properties of the coating are important. Consideration was given to a gaseous method using hydrogen sulphide, but as hydrogen sulphide is extremely toxic and desensitizing, greater skill would be required in its handling and more sophisticated equipment would be needed. It was therefore decided to specify only the aqueous method, particularly as it provides a test that is sufficiently stringent for most purposes.

Methods 22B and 22C are designed to assess the degree of staining caused by free sulphur in the coating polymer to silver and copper components, respectively. The tests are essentially concerned with protecting electrical components from the corrosive effects of free sulphur in the coating polymer.

This Part supersedes method 22 of BS 3424 : 1973.

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

1 Scope

This Part of BS 3424 describes a method for assessing the resistance of coated fabrics to discoloration by sulphur or its compounds and methods for assessing staining caused by free sulphur in the coating polymer of coated fabrics to silver and copper components.

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

2 Method 22A. Sodium sulphide method

2.1 Principle

A test specimen of coated fabric is immersed in acidified sodium sulphide solution for a given duration. The colour of the test specimen is compared with the original sample immediately after removal from the sodium sulphide solution, and then again after 24 h. Any change in colour is reported by reference to the grey scale for assessing staining.

2.2 Apparatus and reagents

2.2.1 *Hydrated sodium sulphide* ($Na_2S.9H_2O$), analytical reagent grade.

2.2.2 *Concentrated hydrochloric acid,* standard laboratory reagent grade.

2.2.3 *Fume cupboard.*

2.2.4 *Three* 250 mL *beakers.*

2.2.5 *Grey scale for assessing staining* complying with BS 1006 : A03.

2.2.6 *Glass rod* for stirring the test solution.

2.2.7 *Clock glasses.*

2.2.8 *Measuring cylinders.*

2.2.9 *Balance* accurate to ± 10 mg.

2.2.10 *Distilled water* complying with BS 3978.

2.3 Test specimen

Cut a test specimen measuring 50 mm × 50 mm from the sample, but not within 50 mm of the selvedge.

2.4 Preparation of test solution

Caution. Hydrogen sulphide, given off by the following acidified solution, is poisonous and desensitizing. Extreme care should therefore be taken in handling the solutions, and all the operations should be carried out in a fume cupboard.

2.4.1 Dissolve 11 ± 0.1 g of hydrated sodium sulphide (**2.2.1**) in 100 mL of distilled water (**2.2.10**) in a 250 mL beaker (**2.2.4**).

2.4.2 Add, with stirring, 6 mL of concentrated hydrochloric acid (**2.2.2**) to a second 250 mL beaker containing 100 mL of distilled water (**2.2.10**).

2.4.3 Transfer 50 mL of each of the solutions prepared at **2.4.1** and **2.4.2** into a 250 mL beaker, stir and cover with a clock glass (**2.2.7**).

2.5 Procedure

Immerse the test specimen in the solution prepared at **2.4.3.** Cover the beaker with a clock glass, stirring the solution occasionally with a glass rod (**2.2.6**).

After 30 min immersion, remove the test specimen from the acidified sodium sulphide solution, and wash it thoroughly in distilled water. Absorb the surplus water from the test specimen by pressing it between filter papers.

Immediately compare the colour of both surfaces of the test specimen with the original sample, and record any change in shade or colour by reference to the grey scale for assessing staining (**2.2.5**).

Expose the test specimen for 24 h to the normal laboratory atmosphere, and re-examine it to assess whether any permanent staining has occurred by comparing it with the original sample.

Record any contrast between the test specimen and the original sample by reference to the grey scale for assessing staining.

Where necessary, repeat the test to ensure that all colours in a multicoloured sample have been tested.

2.6 Expression of results

2.6.1 *No staining.* If there is no contrast in shade or colour between the test specimen and the original sample, express the result as 'unstained'.

2.6.2 *Staining.* If there is any contrast in shade or colour between the test specimen and the original sample, express the result as 'stained', followed by the numerical rating for the contrast by reference to the grey scale for assessing staining. If this lies between two contrasts on the scale, give an intermediate assessment, e.g. 3-4.

2.6.3 *Temporary staining.* If there is any staining immediately after removal of the test specimen from the test solution which then disappears after 24 h exposure to the standard atmosphere for conditioning and testing specified in BS 3424 : Part 2, express the result as 'temporary staining'.

2.7 Test report

The test report shall include the following particulars:

(a) the description of the sample, including details of the number of colours contained;

(b) whether the test specimen was unstained (see **2.6.1**) or stained (see **2.6.2**)/temporarily stained (see **2.6.3**), and if so, the numerical rating for the contrast in shade or colour between the test specimen and the original sample and the colour to which the result applies;

(c) reference to this method of test, i.e. method 22A of BS 3424 : Part 19;

(d) details of any deviation from the standard test procedure.

3 Method 22B. Corrosive sulphur test (Silver stain test)

3.1 Principle

A test specimen of coated fabric is placed on a polished and degreased piece of assay quality silver foil. The foil and coated fabric are placed superposed inside an oven maintained at 70 °C for 30 min. After removal from the oven and return to normal temperature, the silver foil is inspected and staining of the foil is assessed.

3.2 Apparatus and reagents

3.2.1 *Two or three pieces of silver foil* (see **3.4**) assaying at 99.9 % silver, each measuring at least 30 mm × 30 mm.

3.2.2 *Jeweller's rouge.*

3.2.3 *Propan-2-ol (isopropyl alcohol)* complying with BS 1595 : Part 1.

3.2.4 *Polishing and drying cloth* of pharmaceutical grade absorbent material.

3.2.5 *Grey scale for assessing staining* complying with BS 1006 : A03.

3.2.6 *An oven* capable of maintaining a dry atmosphere of 70 ± 1 °C.

3.2.7 *Glass plate* measuring 30 mm × 30 mm × 4 mm.

3.2.8 *Filter papers* each measuring 30 mm × 30 mm.

3.2.9 *Clean glass jar with stopper.*

3.3 Test specimen

Cut a test specimen measuring 25 mm × 25 mm from the sample, but not within 50 mm of the selvedge.

3.4 Procedure

Thoroughly clean and polish both pieces of silver foil (**3.2.1**) with the jeweller's rouge (**3.2.2**) and isopropyl alcohol (**3.2.3**). Rub dry with the polishing and drying material (**3.2.4**). If double-faced coated fabric is to be tested, three pieces of silver foil will be required to be cleaned and polished.

Place one of the cleaned and polished pieces of silver foil in the glass jar (**3.2.9**) to avoid contamination.

Carefully place a specimen of coated fabric, coated side down, onto one of the polished pieces of silver foil. Cover the coated fabric with a filter paper (**3.2.8**), and cover the filter paper with a glass plate (**3.2.7**). If double-faced coated fabric is to be tested, place the second piece of silver foil on the upper coated surface, and cover with the filter paper and glass plate.

Place the test assembly of coated fabric, foil, filter and glass plate carefully in a horizontal position inside an oven (**3.2.6**) pre-heated to 70 ± 1 °C.

Remove the test assembly from the oven 30 min after the oven has regained its temperature of 70 ± 1 °C. Remove the coated fabric from the silver foil, and immediately compare the silver foil removed from the oven with the polished silver foil removed from the glass jar, reporting any staining by reference to the grey scale for assessing staining (**3.2.5**).

3.5 Expression of results

3.5.1 *No staining.* If there is no contrast in shade or colour between the silver foil removed from the oven and the reference sample, express the result as 'unstained'.

3.5.2 *Staining.* If there is any contrast in shade or colour between the silver foil removed from the oven and the reference sample, express the result as 'stained', followed by the numerical rating for the contrast by reference to the grey scale for assessing staining. If this lies between two contrasts on the scale, give an intermediate assessment, e.g. 3-4.

3.6 Test report

The test report shall include the following particulars:

(a) the description of the sample;

(b) whether the silver foil was unstained (see **3.5.1**) or stained (see **3.5.2**), and if so, the numerical rating for the contrast in shade or colour between the silver foil removed from the oven and the reference sample of foil;

(c) reference to this method of test, i.e. method 22B of BS 3424 : Part 19;

(d) details of any deviation from the standard test procedure.

4 Method 22C. Corrosive sulphur test (Copper stain test)

4.1 Principle

A cleaned, polished and degreased pure copper sheet is immersed at 40 °C for 5 h in a test solution of toluene and trimethylpentane together with strips of a test specimen of coated fabric. The test strips of coated fabric and the copper sheet are then removed, and the copper sheet is compared with a similar one immersed for 5 h in a clean test solution containing no coated fabric specimen. Staining of the copper sheet immersed with the coated fabric specimens is assessed against the clean copper sheet and any staining is reported by reference to the grey scale for assessing staining.

4.2 Apparatus and reagents

4.2.1 *Two stoppered clean glass vessels* of at least 200 mL capacity.

4.2.2 *Stainless steel surgical forceps.*

4.2.3 *Two pieces of pure copper sheet* each measuring 13 mm × 75 mm.

4.2.4 *Jeweller's rouge.*

4.2.5 *Polishing and drying cloth* of pharmaceutical grade absorbent material.

4.2.6 *Propan-2-ol (isopropyl alcohol)* complying with BS 1595 : Part 1.

4.2.7 *Sulphur-free test fluid mixture* of 35 % toluene complying with BS 135, 458, 805 and 65 % 2,2,4-trimethylpentane.

4.2.8 *Means of maintaining an atmosphere of* 40 ± 1 °C.

4.2.9 *Grey scale for assessing staining* complying with BS 1006 : A03.

4.3 Test specimens

Cut a test specimen with a mass of 5 ± 0.5 g into pieces measuring approximately 3 mm x 3 mm.

4.4 Procedure

Thoroughly clean and polish both copper sheets (**4.2.3**) using a paste of jeweller's rouge (**4.2.4**) and isopropyl alcohol (**4.2.6**) and the polishing cloth (**4.2.5**). Continue rubbing the copper sheets with successive clean polishing cloths until a clean pad remains unsoiled. Wash both copper sheets in isopropyl alcohol, remove them using the forceps (**4.2.2**), and allow to dry. Do not handle the copper sheets other than with the forceps.

Completely immerse one of the copper sheets together with the pieces of test specimen (see **4.3**) in approximately 100 mL of the test fluid (**4.2.7**) in the first glass vessel (**4.2.1**), and maintain the mixture at 40 ± 1 °C for 5 h. Simultaneously, immerse the second copper sheet in approximately 100 mL of the test fluid in the second glass vessel at 40 ± 1 °C for 5 h.

Using the forceps, remove the copper sheet from the glass vessel containing the pieces of test specimen, and wash in isopropyl alcohol.

Again using the forceps, remove the copper sheet from the glass vessel containing the uncontaminated test fluid, and wash in fresh isopropyl alcohol.

As soon as they are dry, compare the two copper sheets, and report any staining of the one from the glass vessel containing the strips of test specimen by reference to the grey scale for assessing staining (**4.2.9**).

4.5 Expression of results

4.5.1 *No staining.* If there is no contrast in shade or colour between the copper sheet immersed with the pieces of test specimen and the other copper sheet immersed in uncontaminated test fluid, express the result as 'unstained'.

4.5.2 *Staining.* If there is any contrast in shade or colour between the copper sheet immersed with the pieces of test specimen and the other copper sheet immersed in uncontaminated test fluid, express the result as 'stained', followed by the numerical rating for the contrast by reference to the grey scale for assessing staining. If this lies between two contrasts on the scale, give an intermediate assessment, e.g. 3-4.

4.6 Test report

The test report shall include the following particulars:

(a) the description of the sample;

(b) whether the copper sheet was unstained (see **4.5.1**) or stained (see **4.5.2**), and if so, the numerical rating for the contrast in shade or colour between the copper sheet immersed with the pieces of test specimen and the other copper sheet immersed in uncontaminated test fluid;

(c) reference to this method of test, i.e. method 22C of BS 3424 : Part 19;

(d) details of any deviation from the standard test procedure.

This British Standard, having been prepared under the direction of the Rubber Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 30 November 1989

ISBN 0 580 17561 8

The following BSI references relate to the work on this standard:
Committee reference RUM/13 Draft for comment 87/37204 DC

Publications referred to

BS 135, 458, 805	Specifications for benzene, xylenes and toluenes
BS 1006	Methods of test for colour fastness of textiles and leather A03 Grey scale for assessing staining (including half-steps)
BS 1595	Propan-2-ol (isopropyl alcohol) for industrial use Part 1 Specification for propan-2-ol (isopropyl alcohol)
BS 3424	Testing coated fabrics Part 0 Foreword and general introduction Part 2 Method 4. Conditioning and selection of test specimens
BS 3978	Specification for water for laboratory use

Amendments issued since publication

Amd. No.	Date of issue	Text affected

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8911–2–1.1k–B

RUM/13