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SP 15-4 (1988): Handbook of Textile Testing Part-4
identification and Testing of Dyestuffs and Their Colour
Fastness of Textile Materials [TXD 1: Physical Methods of
Tests]



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“Knowledge is such a treasure which cannot be stolen”

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DDP/SC

Handbook of Textile Testing

**Part 4 Identification and Testing of Dyestuffs and Their
Colour Fastness on Textile Materials**

(First Revision)



BUREAU OF INDIAN STANDARDS

HANDBOOK OF TEXTILE TESTING

**Part 4 Identification and Testing of Dyestuffs and Their
Colour Fastness on Textile Materials**

(First Revision)

**BUREAU OF INDIAN STANDARDS
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FOREWORD

Each part/section of the Handbook on Textile Testing covers the methods of tests relating to that group. It is, however, felt essential to bring out fundamental and general principles governing the Handbook together with common aspects applicable to this part.

Textile dyes cover a wide range of application classes and chemical composition, such as direct, acid, sulphur, vat, solubilized vat, reactive, disperse, chrome, per-metallized and textile pigments, etc. When applied to textiles by dyeing or printing, they not only enhance the appeal of the textile product but also increase the sale value of a product to a considerable extent. In order to give the best end use suitability and full satisfaction to the consumers, it is essential that the dyestuffs applied on the textile products, in addition to increasing its appeal, should withstand the effects of light, washing, perspiration, heat, etc, to which the textile is subsequently exposed in actual use. It is in this context that standard methods of testing colour fastness of textile materials are undoubtedly very important.

Textile dyes from different manufacturers differ very widely in their chemical constitution, strength and quality even when the dyestuff is same. These variations in the dyestuffs are subsequently transformed into various dyeing defects, such as lot to lot variation, variation within the same lot (listing, ending, etc) and lead to wide scale rejection of the textile product. This situation is further exaggerated when the dyestuffs from different manufacturers differ widely in their colour fastness ratings. The variations in the strength of dyestuffs compel the dyer or printer to adjust his recipe every now and then and lead to increased cost of dyeing or printing. Therefore, in order to minimize lot to lot variations and the malpractice in the trade, development of standard methods of test for identification and testing of dyestuffs needs no emphasis.

The methods of test included in this part for colour fastness are based on the current national and international practice and have been found very useful for assessing the quality of dyed and printed textiles. Similarly, the methods of test for identification and testing of dyestuffs are presently prevalent and found useful in controlling the various requirements of production batches and dyeing recipes.

Various standards included in this part have been brought out by the Dyestuffs Sectional Committee (TDC 38) and Chemical Methods of Test Sectional Committee (TDC 5) of the Textile Division Council.

INTRODUCTION

Handbook of Textile Testing (SP : 15-1981) was first published in 1982 and was taken up for revision to incorporate new standards which have since been published. Opportunity has also been taken to incorporate the new versions of the standards which have since been revised. The Handbook is now being brought out in four parts wherein standards have been grouped on the basis of application and use. The four parts are:

Part 1 Testing and grading of textile fibres

Part 2 Testing of yarns and fabrics (excluding colour fastness)

Part 3 Testing of textile products other than yarns and fabrics

Part 4 Identification and testing of dyestuffs and their colour fastness on textile materials

The Handbook is basically a compilation of various Indian Standards on methods of test published by various sectional committees under the Textile Division Council. There are more than 300 standards covering a wide range of physical and chemical characteristics of textiles. Such methods of test for which separate standards have not been published and which are included in the product specifications, have also been included in the revision of the Handbook, wherever appropriate. The methods of test included in the Handbook would now be able to satisfy the requirement of various sectors of textile industry like testing laboratories, research institutions, educational institutions in as far as the testing of the products like handloom and khadi, powerloom, hosiery, carpets, readymade garments, dyestuffs, textile auxiliaries, ropes and cordage, industrial textiles, aerospace textiles, etc, is concerned.

The object of the Handbook is to:

- a) give the user first-hand information on all published national standards on methods of test for textiles and their use;
- b) help the various users to establish a suitable quality assurance system in their organizations;
- c) serve as a guide for the ordinary consumer to know what characteristics of textile are important for its best use and care; and

- d) assist the textile students, and educational and research institutions in the methods of test for effective studies and research.

Some of the important aspects covered in the Handbook are:

- a) identification and grading of various textile fibres and yarns;
- b) quantitative chemical analysis and tests for various physical and chemical characteristics of textile fibres;
- c) identification and strength of various dyestuffs used for textiles;
- d) colour fastness properties of coloured textile towards various agencies, such as light, washing, heat, perspiration, hot water and dry cleaning, etc;
- e) code of practice for stains removal from textiles and clothing;
- f) tests for various physical and chemical characteristics of yarns and fabrics, such as strength parameters, dimensional stability, water repellency, soil resistance, flame resistance, flammability, biological degradation, etc;
- g) tests for various sizing and finishing treatments; and
- h) tests for ropes and cordages, coir products, textile floor coverings, industrial textiles, fishing gear materials, hosiery products, tapes, webbings, narrow fabrics and aerospace textiles.

Every effort has been made to make the various parts and sections self-contained but in certain cases, relevant provisions have been extracted and reproduced. In all such cases, for detailed guidance, reference should be made to individual standard and, in case of any contradiction observed between the Indian Standards and those reproduced (here in) the provisions of the former should be considered accurate. The purpose of the Handbook is to make it self-contained reference document and, at the same time, keeping it less voluminous. The present version of the Handbook is the judicious choice with respect to these two aspects.

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METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS — COTTON AND OTHER CELLULOSIC FIBRES

[Source : IS 4472 (Part 1) : 1967]

The methods prescribed are simple and meant for rapid identification of the application classes of dyestuffs. Successful identification of the application classes of dyestuff depends on

experience and familiarity of the dyestuffs. In general, the identification of the application classes of dyestuffs is not dependent on any single test.

1 SCOPE

1.1 These methods are intended for the identification of application classes of dyes on cotton and other cellulosic fibres.

1.1.1 The methods are applicable to dyes normally used for dyeing and printing cotton and other cellulosic fibres.

2 PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is fibre or yarn, take a tuft of fibre or yarn of about 3 cm length.

2.2 If the sample under test is fabric, take a 3×3 cm test piece.

NOTE — In case of multi-coloured fabric, the specimens shall be taken from different coloured portions of the sample and the different coloured fibres/yarns present therein shall be identified separately for their respective classes of dyes.

2.3 In the case of finished textiles, the sample should be treated twice with 1 percent hydrochloric acid at boil for 5 minutes.

3 REAGENTS

3.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

3.1 Ammonium Hydroxide

(a) dilute (1 percent w/v), and (b) concentrated [25 percent (w/v)] of sp gr 0.88.

3.2 Sodium Hydroxide Solution

(a) 5 percent (w/v), (b) 10 percent (w/v), and (c) 44 percent (w/v) (or 70°Tw).

3.3 Sodium Carbonate

(a) 5 percent solution (w/v), and (b) solid.

3.4 Ammonium Chloride Solution

5 percent (w/v).

3.5 Vat Dye Developer Solution

prepared by dissolving 8 g of ammonium chloride and 2 g of ammonium persulphate in 100 ml of water.

3.6 Hydrogen Peroxide

3 percent (w/v) (10 volumes).

3.7 Sodium Sulphoxylate Formaldehyde (Glycol Solution)

prepared by dissolving 20 g of sodium sulphoxylate formaldehyde in 75 ml of warm water and diluted with cold water. To this is added 50 g of mono- or diethylene glycol.

NOTE — Sodium sulphoxylate formaldehyde is commercially available as 'Formosul', 'Rongalite', etc.

3.8 Ethylenediamine

sp gr 0.97, b.p. 117°C.

3.9 Acetic Acid

(a) 10 percent (w/v), (b) 20 percent (w/v), and (c) glacial.

3.10 Hydrochloric Acid

(a) 1 percent (w/v), and (b) 10 percent (w/v).

3.11 Sodium Hypochlorite Solution

2 to 3 g/l of available chlorine.

3.12 Sodium Sulphide

(a) 5 percent solution (w/v), and (b) solid.

3.13 Tannin Reagent

prepared by dissolving 10 g of tannic acid and 10 g of anhydrous sodium acetate in 200 ml of water.

3.14 Lead Acetate Solution

5 percent (w/v).

3.15 Stannous Chloride Solution

prepared by dissolving 100 g of stannous chloride in 100 ml of concentrated hydrochloric acid at boil.

3.16 Rectified Spirit

3.17 Ferric Chloride Solution

1 percent (w/v).

3.18 Glucose

solid.

3.19 Sulphuric Acid

(a) 5 percent (w/v), (b) 12 N, and (c) concentrated (sp gr 1.84).

3.20 Sodium Nitrate

solid.

3.21 Nitric Acid

concentrated (sp gr 1.42).

3.22 Carbazol Solution

1 percent solution in concentrated sulphuric acid.

3.23 Chromotropic Acid

5 percent solution in distilled water.

3.24 Pyridine

b.p. 112 to 114°C.

3.25 Sodium Hydrosulphite

solid.

3.26 Dimethylformamide

(a) 50 percent (w/v), and (b) concentrated, b.p. 152 to 154°C.

3.27 Phenol

3.28 Monoethyl Ether of Ethylene Glycol

b.p. 132 to 136°C.

3.29 Mordanted Cotton

mordanted with tannic acid or any other mordant suitable for basic dyes.

3.30 Bleached Cotton

3.31 Undyed Wool

3.32 Sodium Chloride

solid.

3.33 Methyl Pyrrolidone

b.p. 202°C.

4 PROCEDURE

4.1 For preliminary identification of dyes on fibres, follow the procedure given in Annex A.

4.2 Confirm, wherever necessary, the class of dyes by the tests prescribed in Annex B.

NOTE — While identifying the dyes used for dyeing pale shades, it is advisable to use large specimen and larger quantities of reagents, and to concentrate the extract before making the test.

4.3 For the dyes which are not easily identified by the procedure given in Annexes A and B, follow the procedure given in Annex C.

ANNEX A

PRELIMINARY TESTS FOR IDENTIFICATION OF DYES ON COTTON AND OTHER CELLULOSIC FIBRES

The test specimen is successively treated with (a) 50 percent dimethylformamide, (b) concentrated dimethylformamide and (c) mixture of glacial acetic acid and rectified spirit (1 : 1, v/v) at boil for 3 to 4 minutes with intermediate washing with water and squeezing.

If the dyes of Group I are absent, take a fresh test specimen and boil it for 1 to 2 minutes in 1 percent ammonium hydroxide solution.

No stripping or partial stripping of dye. (Some of the basic dyes are not stripped)	<i>Reactive Dyes and Ingrain Dyes (Except Azoics)</i> The specimen bleeds off the dye. The solution is distinctly coloured. Remove the test specimen and add a few pieces of white bleached cotton, 25 mg of sodium chloride and boil for 2 minutes. Cool and rinse the added bleached cotton. The white cotton is dyed to approximately original shade. <i>Direct Dyes</i> The specimen bleeds off the colour and the white cotton is left undyed or slightly stained, neutralize the solution with acetic acid and add 1 ml of 10 percent acetic acid. Add undyed wool pieces and boil for 1 minute. Cool and rinse the added wool pieces. The wool is dyed. <i>Acid Dyes (If Direct and Basic Dyes Absent)</i> Mordanted cotton is dyed. <i>Basic Dyes</i> Test the specimen (treated with 1 percent hydrochloric acid) for the presence of direct dyes if the specimen responds to direct dyes test. <i>Direct Dyes After-treated with Resin</i>	<p>If the test specimen does not bleed (or bleeds slightly), take a fresh test specimen, and add 1 ml of glacial acetic acid and warm. Add 3 to 5 ml of water and boil. Remove the specimen, add 25 mg of mordanted cotton and boil for 2 minutes.</p> <p>If direct, reactive, ingrain (except azoics) acid and basic dyes are absent, take a fresh test specimen, add 10 to 15 ml of 1 percent hydrochloric acid. Boil for 1 minute. Discard the acid solution and repeat this once or twice.</p> <p>If the dyes of Group II are absent, a fresh test specimen is boiled for 1 to 2 minutes in solution containing 10 to 30 mg of sodium hydrosulphite in 5 to 10 ml of water to which 4 to 6 drops of 44 percent sodium hydroxide solution are added.</p> <p>Group III dyes decolourize or change shade radically. The colour of the test specimen is restored on exposure to air or with vat dye developer.</p> <p>All Group IV dyes are destroyed and, on re-oxidation, do not restore to the original colour.</p>	<p>Take a fresh test specimen, add 2 to 3 ml of water, 1 to 2 ml of 5 percent sodium carbonate solution, 500 mg of solid sodium sulphate and boil for 2 minutes. Remove the specimen. Add 25 mg of sodium chloride and a few pieces of white bleached cotton. Boil for 2 minutes; the white cotton is dyed. Place the test specimen and white cotton on filter paper and reoxidize the dye and dyeings on white cotton. White cotton is redyed to original shade (except strength) and the test specimen restores the colour.</p> <p><i>Sulphur Dyes</i></p> <p>Cotton is not redyed from sodium carbonate-sodium sulphide solution. Take a fresh sample in an evaporating dish, pour 2 to 3 ml of concentrated sulphuric acid. Shake just enough to extract the dye. Pour the extract into the test tube, add 25 ml of water and filter. Wash the filter paper with water. Spot the filter paper with 10 percent sodium hydroxide solution. Spot turns red violet.</p> <p><i>Oxidation Blacks (Aniline Black)</i></p> <p>If sulphur and oxidation blacks are absent, take a fresh test specimen; boil it with sodium sulphoxylate formaldehyde-glycol solution containing few drops of sodium hydroxide solution (44 percent). Test specimen is decolourized or altered in shade. The solution is yellow, bluish red, etc. Original colour restored by vat-dye developer.</p> <p><i>Vat Dyes</i></p> <p>Take a fresh test specimen (about 6 g) and ash it in a porcelain crucible. Add 200 mg of flux (equal parts of sodium carbonate and sodium nitrate) and fuse. Fused mass is orange yellow when hot and permanent greenish yellow when cold.</p> <p><i>Direct Dyes After-treated with Chromium Salts</i></p> <p>If chromium is absent, ash the test specimen as above and dissolve the ash in few drops of concentrated nitric acid. Add 2 ml of water, boil and cool. Add 2 ml of concentrated ammonium hydroxide. Blue colour indicates:</p> <p><i>Direct Dyes After-treated with Copper Salts</i></p> <p>If chromium or copper salts treated direct dyes are absent, take a fresh test specimen, treat it with 5 percent boiling sulphuric acid. Cool and add dropwise 1-percent carbazol solution. Blue precipitate indicates presence of formaldehyde.</p> <p><i>Direct Dyes After-treated with Formaldehyde</i></p> <p>Specimen bleeds and continues to do so in subsequent treatment.</p> <p><i>Azoic Dyes</i></p> <p>Specimen does not bleed or bleed slightly; bleeding decreases or in most cases terminates.</p> <p><i>Diazotized and Developed Dyes</i></p>	<p>The dyes of Group V usually do not respond to the reactions of the first four groups (if they do the reactions are slower and often incomplete).</p> <p><i>Vat and Azoic Pigments</i></p>
Group I	Group II	Group III	Group IV	Group V

ANNEX B

CONFIRMATORY TESTS FOR IDENTIFICATION OF DYES ON COTTON AND OTHER CELLULOSIC FIBRES

B-1 DIRECT DYES

B-1.1 Take a test specimen. Boil it for half a minute in 5 percent sodium hydroxide solution (see Note under B-1.2). Add little mercerized cotton to the solution. Dye the mercerized cotton for 10 minutes. The dye on the mercerized cotton is not stripped by 1 percent ammonium hydroxide solution.

B-1.2 Take a test specimen. Add to it little cold ethylenediamine and shake for some time. Dilute the coloured extract with water. Introduce a few pieces of white cotton and heat to 80°C. Add little sodium chloride and allow it to cool. The white cotton is stained evenly and the stain is not removed by boiling with 1 percent ammonium hydroxide solution.

NOTE — Certain pale blue dyeings are not appreciably affected by boiling in 5 percent sodium hydroxide solution. However, they are stripped to white in about 15 minutes by cold ethylenediamine. Dyes of this type belong to 'dioxazine' series.

B-1.3 Direct Dyes After-treated with Formaldehyde

Take a test specimen. Extract it with 12 N sulphuric acid for 5 minutes. Add 1 to 2 ml of concentrated sulphuric acid and 4 to 5 drops of chromotropic acid. Reddish violet colour is obtained.

B-2 BASIC DYES

B-2.1 Take a test specimen. Add to it 1 ml of 5 percent sodium hydroxide solution and boil for a few seconds. To this add 4 ml of 5 percent ammonium chloride solution and boil again.

B-2.1.1 Take small amount of extract obtained in B-2.1. Add to it a few pieces of undyed wool and allow the solution to cool. Most of the dye is taken up by the wool.

B-2.1.2 Take small amount of extract obtained in B-2.1. Acidify the solution with 10 percent acetic acid and add tannin reagent. A coloured precipitate is obtained.

B-2.1.3 Take small amount of extract obtained in B-2.1. Acidify the solution with 10 percent acetic acid. Add to it 1 percent solution of ferric chloride. A black precipitate is obtained.

NOTE — The tannin mordanted dyeings would give the above test.

B-3 SULPHUR DYES

B-3.1 Take a test specimen. Boil with stannous chloride solution in a test tube, the mouth of

which is covered with filter paper moistened with lead acetate solution. Brown stains on filter paper will indicate sulphur dyes.

NOTES

1 Deep brown stains should be regarded as positive test.

2 Prolonged boiling may discolour the brown stains due to action of hydrochloric acid.

B-3.2 Take a test specimen. Boil it with ethylenediamine. The dye is readily stripped.

NOTE — Indocarbon black dyeings give reddish solution which revert to original shade on dilution with water whereas sulphur black dyeings give greenish solution.

B-3.3 Take a test specimen. Treat it with sodium hypochlorite solution. The specimen is bleached to white or buff colour.

NOTE — Indocarbon blacks are not affected by this test.

B-4 VAT DYES

B-4.1 Take a test specimen. Boil it with 5 to 10 ml of sodium sulfoxylate formaldehyde-glycol solution containing a little of 44 percent sodium hydroxide solution. A distinct change in colour is observed. Take out the test specimen and wash with fresh water. The original colour is restored, if not, treat the test specimen with vat dye developer or hydrogen peroxide.

B-4.2 Take a test specimen. Treat it with little ethylenediamine and glucose. The colour is more or less completely removed when treated near the boiling point.

B-5 AZOIC DYES

B-5.1 Take a test specimen, add sufficient amount of ethylenediamine and boil for few minutes. A considerable amount of dye is extracted. Divide the extract in two parts and proceed as below.

B-5.1.1 To one part of the extract, add a little sodium hydrosulphite. Warm, if necessary, permanent decolourization occurs.

B-5.1.2 Dilute the other part of extract with water and boil. The liquid becomes turbid and the flakes of coloured pigment settle down on standing.

B-5.2 Take a test specimen. Add to it little sodium sulfoxylate formaldehyde-glycol solution containing 44 percent sodium hydroxide solution and boil vigorously. Most of the dyeings are reduced to colourless or yellow compounds.

NOTE — If no signs of reduction appear after boiling for 1 or 2 minutes, treat the test specimen in boiling 5 percent sodium hydroxide solution, to which little sodium hydrosulphite is added. All azoic dyeings including the yellow ones are reduced to pale yellow or white by this method. Some of the alkaline reduction products exhibit fluorescence when exposed to ultra-violet rays. This phenomenon is of value in confirming the presence of certain pale azoic dyeings and prints.

B-5.3 Take a test specimen. Dip it in liquid phenol. Take out the specimen and squeeze it lightly. Lay the test specimen between two filter papers, and press it either with hot iron or on steam pipe. The filter paper is stained.

B-5.4 Take a test specimen. Add to it a solution of equal volumes of 10 percent sodium hydroxide and monoethyl ether of ethylene glycol, and little sodium hydrosulphite. The azoic dyeings are reduced to colourless or yellow compound on boiling.

NOTE — The above test is carried out, especially when the dyeings are on the viscose rayon.

B-6 PIGMENTS

B-6.1 Vat Pigments

Take a test specimen and treat it with methyl pyrrolidone which will plasticize the resin binder. Carry out tests for the presence of vat dyes.

B-6.2 Azoic Pigments

Take a test specimen weighing about 200 mg and treat it with 1 ml of methyl pyrrolidone for 30 seconds and cool. Add 5 percent sodium hydroxide solution and 25 to 50 mg of sodium hydrosulphite. Boil till the sample is white, light yellow or orange. Filter the solution, add 25 mg of sodium chloride and a few pieces of cotton. Boil for 1 minute and cool. Take out the white cotton and dry. Naphthol AS pigments would be yellow while β -naphthol pigments would darken and turn brown.

B-7 OXIDATION BLACK (ANILINE BLACKS)

B-7.1 Take a test specimen and digest it with concentrated sulphuric acid in cold. Dilute the solution with water, a green colour is obtained.

B-7.2 Take a test specimen. Treat it for 1 minute with sodium hypochlorite solution. The specimen turns brown.

B-7.3 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron or copper; a positive test for the presence of iron or copper is obtained.

B-8 INGRAIN DYES (EXCEPT AZOICS)

B-8.1 Phthalogen Green

B-8.1.1 Take a test specimen. Add 1 ml of methyl pyrrolidone. Heat to boil and cool to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen reduces to dark violet. Remove the test specimen and put it in 20 percent acetic acid solution. The colour remains violet.

B-8.2 Phthalogen Blue

B-8.2.1 Take a test specimen add 1 ml of methyl pyrrolidone. Heat to boil and cool to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen does not reduce.

B-8.2.2 Take a test specimen and spot it with concentrated nitric acid. The colour changes to violet.

B-8.2.3 Take a test specimen and spot it with concentrated sulphuric acid. The colour changes to bright green.

B-8.3 Alcian Blue

B-8.3.1 Take a test specimen. Add 1 ml of methyl pyrrolidone. Heat to boil and cool it to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen changes to violet. Remove the test specimen and put it in 20 percent acetic acid solution. The colour of the test specimen changes to green.

B-8.3.2 Take a test specimen and spot it with concentrated nitric acid. The colour of the specimen changes to violet.

B-8.3.3 Take a test specimen and spot it with concentrated sulphuric acid. The colour of the test specimen changes to green.

ANNEX C

METHOD OF TEST FOR MINERAL KHAKI, VEGETABLE KHAKI, CATECHU, ETC

C-1 MINERAL KHAKI

C-1.1 Take a test specimen. Boil it for 5 minutes with 10 percent hydrochloric acid. Observe the test specimen. The colour of the test specimen is completely stripped off and the solution is green.

C-1.2 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron and chromium. The positive test for iron and chromium indicates the presence of mineral khaki.

C-2 VEGETABLE KHAKI

C-2.1 Take a test specimen. Boil it for 5 minutes with 10 percent hydrochloric acid. Observe the test specimen. The colour of the test specimen is not completely stripped off and the solution is yellow.

C-2.2 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron and chromium. The positive test for iron and chromium indicates the presence of vegetable khaki.

C-2.3 Take a test specimen and treat it with 5 percent sodium hydroxide solution at boil for 1 to 2 minutes; the specimen as well as the solution turns brownish.

C-3 CATECHU

C-3.1 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of copper and chromium. The positive test for copper and chromium indicates the presence of catechu.

C-3.2 Take a test specimen. Put a drop of concentrated nitric acid on it, an orange colour is obtained.

C-4 CHROME YELLOW AND CHROME ORANGE

C-4.1 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of lead. The positive test of lead indicates the presence of chrome yellow or chrome orange.

NOTE — Chrome yellow dyeing on treatment with 5 percent sodium hydroxide solution becomes orange and chrome orange dyeing on treatment with 10 percent hydrochloric acid becomes yellow.

C-5 PRUSSIAN BLUE

C-5.1 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron. The positive test for iron indicates presence of prussian blue.

C-5.2 Take a test specimen and treat it with hot 5 percent sodium hydroxide solution. The specimen turns brown.

METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS — WOOL, SILK AND OTHER PROTEIN FIBRES

[Source : IS 4472 (Part 2) : 1968]

The methods of identification of dyes on wool, silk and other protein fibres prescribed are simple and meant for rapid identification of application classes of dyes. Successful identification of application classes of dyes depends on experience and familiarity of the dyestuffs. The knowledge of a fibre eliminates dyes which are

not applicable to the particular fibre and the identification is made somewhat easier.

In general, the identification of application classes of dyes is not dependent on any single test and final confirmation of the identity of an unknown class of dye should preferably be made by comparison with an authentic sample.

1 SCOPE

1.1 These methods are intended for the identification of application classes of dyestuff on wool, silk and other protein fibres.

1.1.1 The methods are applicable to types of dyes normally used for dyeing and printing wool, silk and other protein fibres.

2 PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is fibre or yarn, take a tuft of fibre or yarn of about 3 cm length.

2.2 If the sample under test is fabric, take a 3 × 3 cm test piece.

NOTE — In case of multi-coloured fabric, the specimens shall be taken from different coloured portions of the sample and the different coloured fibres/yarns present therein shall be identified separately for their respective classes of dyes.

2.3 To remove finishing materials, if any, present in textiles, the sample should be treated twice with 1 percent hydrochloric acid at boil for 5 minutes and washed.

3 REAGENTS

3.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

3.1 Acetic Acid

(a) 5 percent (w/v) and (b) glacial.

3.2 Ammonium Hydroxide Solution

prepared by diluting 1 ml of concentrated ammonia (sp gr 0.88) to 100 ml with water.

3.3 Sodium Chloride

solid.

3.4 Sulphuric Acid

10 percent (w/v).

3.5 Sodium Carbonate

(a) 5 percent solution (w/v) and (b) solid.

3.6 Sodium Nitrate

solid.

3.7 Sodium Hydroxide Solution

1, 5, 10 and 30 percent solutions.

3.8 Sodium Hydrosulphite

(a) 10 percent (w/v) freshly prepared and (b) solid.

3.9 Sodium Bisulphite

30 percent (w/v).

3.10 Rectified Spirit

3.11 Hydrogen Peroxide

3 percent (w/v) (10 volumes).

3.12 Hydrochloric Acid

(a) prepared by diluting 15 ml of concentrated hydrochloric acid to 100 ml and (b) concentrated (sp gr 1.16).

3.13 Ethylenediamine

sp gr 0.97, b.p. 117°C.

3.14 Ether

3.15 Bleached Cotton

3.16 Scoured Wool (Undyed)

3.17 Dimethyl Formamide

(a) 50 percent (w/v) and (b) concentrated (b.p. 152 to 154°C).

3.18 Tannin Reagent

prepared by dissolving 10 g of tannic acid and 10 g of anhydrous sodium acetate in 200 ml of water.

3.19 EDTA-Glycerine Solution

4 percent (w/v) solution of disodium salt of ethylenediamine tetra acetic acid in glycerine.

3.20 Paraffin Wax

3.21 Blank Vat Solution

prepared by dissolving 15 g of sodium hydroxide and 20 g of sodium hydrosulphite in sufficient water and made to 1 litre.

4 PROCEDURE

4.1 For identification of dyes on fibres, follow the procedure given in Annex A.

NOTE — While identifying the dyes used for dyeing pale shades, it is advisable to use large specimen and large quantities of reagents, and to concentrate the extract before making the test.

ANNEX A

TESTS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON WOOL, SILK AND OTHER PROTEIN FIBRES

The test specimen is successively treated with : (a) 50 percent dimethylformamide, (b) concentrated dimethylformamide, and (c) a mixture of glacial acetic acid and rectified spirit (1 : 1, v/v) at boil for 3 to 4 minutes with intermediate washing with water and squeezing.

No stripping or partial stripping of dye. REACTIVE DYES	In dyes of Group I are absent, treat the test specimen with 0.1 ml of glacial acetic acid and warm. Add 5 ml of water.				
	The acetic acid extract is distinctly coloured. BASIC DYES	If dyes of Groups I and II are absent, take a fresh test specimen and add to it 5 to 10 ml of 1 percent ammonium hydroxide solution. If extract is coloured, remove the stripped specimen and divide the extract into two portions: (a) Add to the first portion 30 mg of sodium chloride and 10 to 30 mg each of bleached cotton and scoured wool, and boil. (b) Neutralize the other portion by adding 10 percent sulphuric acid and few drops in excess. Add bleached cotton and scoured wool and boil.			
		Cotton is dyed in first portion. DIRECT DYES	If dyes of Groups I, II and III are absent, take a test specimen (about 5 g) and ash it completely in a porcelain crucible. Add 200 mg of flux (equal parts of sodium carbonate and sodium nitrate) and fuse.		
			<p>Test for the Presence of Metal (Cr or Co present) <i>Metal Complex or Mordant Dyes</i> (<i>Chrome</i>)</p>		
			<p>Strip the dye in hot 1 percent ammonium hydroxide. After cooling acidify the solution with hydrochloric acid. Shake up the extract with ether.</p>		
GROUP I	GROUP II	GROUP III	GROUP IV		
			<p>Ether is coloured. <i>Metal Complex Dyes</i></p>	<p>Ether is not coloured. <i>Mordant Dyes</i> (<i>Chrome</i>)</p>	<p>If dyes of Groups I to IV are absent, take a test specimen (about 200 to 300 mg) and add 2.5 ml of 10 percent sodium hydroxide and boil until it is dissolved. Add 25 to 30 mg of sodium hydrosulphite, 20 to 50 mg of sodium chloride and 10 to 15 mg of bleached cotton. Keep near boil for 2 minutes and cool. Take out the cotton and place it on filter paper for 1 to 2 minutes and oxidize with sodium nitrate and acetic acid solution.</p>
			<p>Cotton is dyed in second portion. ACID DYES (<i>If Direct and Basic Dyes Absent</i>)</p>		
			<p>Cotton is dyed. In case of very pale dyeings, it may be necessary to redye the cotton in 2 to 4 successive solutions stripped from the original dyeing.</p>		
			<p>Cotton is undyed and colour is destroyed.</p>		
			GROUP V		

ADDITIONAL TESTS

1) *Basic Dyes*

- i) Take a test specimen. Add to it 1 ml of glacial acetic acid and warm. Add 5 ml of water. To the extract add tannin reagent. A coloured precipitate is obtained.
- ii) Take a test specimen, boil it with rectified spirit; a coloured extract is obtained.
- iii) Take a test specimen, boil it in 2 ml glacial acetic acid. Add 30 percent sodium hydroxide till the solution is alkaline. A change in colour or complete decolourization is obtained. Acidify the solution with 5 percent acetic acid. The original colour is restored.

2) *Direct Dyes*

Take a test specimen and boil it with 5 percent sodium carbonate solution for half a minute in the presence of few pieces of bleached cotton. The cotton is stained. The stained cotton is practically unaffected on treatment with 1 percent ammonium hydroxide solution at boil.

NOTES

- 1 In case of silk dyeings 5 to 10 percent of sodium hydroxide should be used instead of sodium carbonate solution.
 - 2 In the above test, certain dyes which are closely allied to substantive azo dyes in chemical structure, stain the cotton to a comparatively slight extent. Identification of such dyeings may be achieved by boiling the specimen in 1 percent sodium hydroxide solution when dull yellowish colour is obtained. This yellowish colour may also be obtained on boiling the dyeings with 30 percent sodium bisulphite solution.
- 3) *Acid, Metal Complex and Mordant Dyes (Chrome)*
- i) Take a test specimen. Boil it with dimethylformamide. Bleeding indicates *acid dyes*, slight bleeding indicates *metal complex dyes*. No bleeding indicates *mordant dyes (chrome)*.
 - ii) Heat a test specimen in solution of EDTA in glycerine. At 140°C : No change — *Acid and mordant dyes*. Rapid change (in 1 to 2 minutes) — 1 : 1 *metal complex dyes (acid dyeing)*. Slow change (in 20 minutes) — 1 : 2 *metal complex dyes (neutral dyeing)*. At 160°C : No change — *Acid Dyes*.
 - iii) Take a test specimen. Boil it with dilute hydrochloric acid [see 3.12(a)]. Take out the specimen and wash and treat it with 10 percent sodium hydro-sulphite solution. The colour is destroyed.
 - iv) Most of the after chrome dyes are not stripped. This fact should be taken as a clue for mordant dyes (chrome).

4) *Vat and Azoic Dyes*

- i) Warm some paraffin wax in a white porcelain crucible until faint vapours appear. Take a test specimen and hold it in molten wax for about a minute. Remove the specimen. After cooling any staining of paraffin wax is readily seen against white background of the porcelain.
- ii) Take a test specimen and treat it with a blank vat solution at 60°C in a test-tube. Oxidize the specimen with hydrogen peroxide (3 percent, w/v).
 - a) Change in colour and original colour restored on oxidation — *vat dyes*.
 - b) Test solution becomes yellow. Colour of the pattern usually remains unchanged. If the colour is changed, original colour not restored on oxidation — *azoic dyes*.
- iii) Take a test specimen. Warm it with ethylenediamine. Add aqueous solution of sodium hydrosulphite [see 3.8(a)] to ethylenediamine extract. The coloured extract is decolorized readily and permanently.

NOTE — Most azoic dyeings on wool and silk yield slimy residues of the same intense colours as the original dyes, on boiling dyeings in 5 and 10 percent sodium hydroxide solution respectively (distinction from mordant dyes). Most yellow dyeings and prints change to orange or red colours.

METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS : MAN-MADE FIBRES

[Source : IS 4472 (Part 3) : 1973]

The methods prescribed are simple and meant for rapid identification of application classes of dyes used for man-made fibres. Successful identification of application classes of dyes depends on experience and familiarity of the dyestuffs. The knowledge of a fibre eliminates dyes which are not applicable to the particular

fibre and the identification is made somewhat easier.

In general, the identification of application classes of dyes is not dependent on any single test and final confirmation of the identity of an unknown class of dye should preferably be made by comparison with an authentic sample.

1 SCOPE

1.1 These methods are intended for the identification of application classes of dyes on man-made fibres, such as secondary acetate, triacetate, acrylic polyester, polyamide, polyvinyl alcohol, polyvinyl chloride, polyvinyl acetate, polyurethane and polyolefin fibres; their blends with each other and with natural and regenerated-cellulosic fibres.

1.1.1 The methods are not applicable to protein fibres or blends thereof.

1.2 The methods are applicable to types of dyes normally used for dyeing and printing man-made fibres.

2 PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is in the form of fibres, take a tuft of fibre. If the sample is in the form of yarn, take a bundle of yarn about 3 cm length.

2.2 If the sample is in the form of fabric, take a 3 × 3 cm test specimen.

NOTE — In case of multi-coloured fabric, the specimens shall be taken from different coloured portions of the sample and the different coloured fibres/yarns present therein shall be identified separately for their respective classes of dyes.

2.3 Any finish present in the sample shall be removed prior to identification of application classes of dyes by the procedure given in 2.3.1 to 2.3.6. If the extract is appreciably coloured at any stage, it should be analyzed individually for the application classes of dyes as given in Annex A.

NOTE — These procedures are given only as guide and it must be stressed that a number of finishes are likely to be encountered which will not be removed by these treatments and for which certain other treatments may be necessary.

2.3.1 Treat the specimen with 1 g/l of a non-ionic detergent at 60 to 70°C for 15 to 20 minutes. Wash well first with warm and then with cold water, and dry.

2.3.2 Boil the specimen obtained in 2.3.1 with 50 ml of carbon tetrachloride, under reflux, for 5 minutes.

2.3.3 Boil the specimen obtained in 2.3.2 with 50 ml of ethyl alcohol, under reflux, for 5 minutes.

2.3.4 Boil the specimen obtained in 2.3.3 with 50 ml of distilled water, under reflux, for 5 minutes.

2.3.5 Boil the specimen obtained in 2.3.4 with dioxane, under reflux, for 5 minutes.

2.3.6 Boil the specimen obtained in 2.3.5 with 50 ml of distilled water containing 2 ml of concentrated hydrochloric acid, under reflux, for 5 minutes.

3 APPARATUS, MATERIALS AND REAGENTS

3.1 Apparatus

3.1.1 Microscope

3.1.2 Test Tubes

3.1.3 Separating Funnels

3.1.4 Porcelain Crucible

3.2 Materials

3.2.1 Lead Acetate Paper

3.2.2 Mordanted Wool

3.2.3 Scoured Acetate Fabric

3.2.4 Scoured Cotton

3.2.5 Scoured Wool

3.2.6 Magnesium Ribbon

3.2.7 Zinc Dust, Pure

3.3 Reagents

3.3.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be

used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

3.3.1 Acetic Acid

- (a) 5 percent (m/v) and
- (b) 30 percent (m/v).

3.3.2 Ammonia Solution

- (a) 1 percent (v/v) and
- (b) concentrated (sp gr 0.88).

3.3.3 Carbazol

0.1 percent.

3.3.4 Chromotropic Acid Solution

5 percent (aqueous).

3.3.5 Ether

3.3.6 Ethylene Diamine Hydrate

sp gr 0.960.

3.3.7 Ethylene Diamine Tetra-acetic Acid Disodium Salt

3.3.8 Formic Acid

85 percent.

3.3.9 Formosul G

3.3.10 Glycerol

3.3.11 Hydrochloric Acid

- (a) 16 percent (v/v) and
- (b) Concentrated (sp gr 1.18).

3.3.12 Hydrogen Peroxide

30 percent (m/v) (100 volumes).

3.3.13 Nitric Acid

concentrated.

3.3.14 Non-ionic Detergent

3.3.15 o-Chlorophenol

3.3.16 Paraffin, Liquid

3.3.17 Pyridine

3.3.18 Sodium Carbonate

3.3.19 Sodium Hydroxide Solution

- (a) 5 percent and
- (b) 20 percent.

3.3.20 Sodium Hydrosulphite

- (a) 0.2 percent (m/v) solution and (b) solid.

3.3.21 Sodium Hypochlorite Solution

containing 2 g/l available chlorine.

3.3.22 Sodium Nitrate

3.3.23 Sodium Sulphate Solution

0.2 percent (m/v).

3.3.24 Solution of Dispersing Agent

10 percent (m/v).

3.3.25 Sulphuric Acid

- (a) 5 percent (v/v) and
- (b) concentrated.

3.3.26 Tannic Acid

3.3.27 Toluene

4 PROCEDURE

4.1 Microscopic Examination

Examine the test specimen under the microscope. If the dye is found to be present on the surface of the fibre as particles, it indicates pigment dyes, namely, carbon black, vat, azoic or phthalocyanine [see 3(b) under Additional Tests in Annex A].

4.2 For identification of application classes of dyes, follow the procedure as given in Annex A.

NOTES

1 While identifying the dyes used for dyeing pale shades, it is advisable to use large specimens and large quantities of reagents, and concentrate the extract before making the tests.

2 Before identification, the fibres in the blend may be separated by a suitable method, if necessary.

ANNEX A

IDENTIFICATION OF APPLICATION CLASSES OF DYES ON MAN-MADE FIBRES

TEST SPECIMEN

FIRST PORTION	SECOND PORTION	THIRD PORTION	FOURTH PORTION	FIFTH PORTION
Boil about 0.5 g of test specimen in 1 percent ammonia solution for one minute.	Boil 0.5 g of test specimen in glacial acetic acid for 5 minutes.	Reflux 0.5 g of fresh test specimen with dioxane-water mixture in the proportion of 1 : 5 or 1 : 10 for 2 to 3 hours. If the dioxane extract is coloured, take 20 ml of the extract and add to it 5 to 10 ml of 20 percent sodium hydroxide and 10 to 15 mg of formal G and boil for 5 to 10 minutes. Note any change in colour. Then add to it 2 ml of 100 volume hydrogen peroxide. If the original colour of the solution is now restored, it indicates VAT DYE (the exceptions are with certain blue dyes, the leuco compounds, which are also blue in colour). If not, it indicates AZOIC or CERTAIN DIAZOTISABLE AZO DYES (direct dye, diazotised and developed; and developed disperse dyes).	Treat 0.5 g of fresh test specimen with 57 : 43 pyridine-water in cold for 5 to 10 minutes.	Dissolve 4 g of ethylene diamine tetra-acetic disodium salt in 100 g of glycerol. Heat the test specimen in this mixture at 140°C.
<p><i>A considerable amount of colour bleeds into the solution</i></p> <p>Discard the specimen. Acidify the solution with 30 percent acetic acid. Add 0.5 g of scoured wool and dye for 5 to 10 minutes at 80 to 90°C.</p> <p>If the wool is dyed, it indicates ACID DYE.</p>	<p><i>Slight or no colour bleeds into solution</i></p> <p>Boil 0.5 g of fresh test specimen with 5 percent acetic acid for one minute</p>	<p><i>Boil 0.5 g of fresh test specimen with 5 percent sodium hydroxide solution for 1 to 2 minutes.</i></p> <p>a) If some of the colour is stripped, cool, add ether to the solution and shake well. If the ether layer is coloured, it indicates DISPERSE DYE.</p> <p>b) Treat a fresh specimen in hot liquid paraffin at 160°C for 5 minutes. If the colour is stripped add scoured acetate fabric. Tinting of the acetate fabric confirms DISPERSE DYE.</p>	<p><i>If the liquor is stained, it indicates SOLUBLE DISPERSE DYE.</i></p>	<p>Observe change in colour of the specimen after 1 to 2 minutes and after 20 minutes.</p> <p>If the colour of the specimen changes in 1 to 2 minutes, it indicates 1:1 METAL COMPLEX DYE.</p> <p>If the colour of the specimen changes after 20 minutes, it indicates 1:2 METAL COMPLEX DYE.</p>
<p><i>The solution is distinctly coloured</i></p> <p>Divide the solution into two parts:</p> <p>a) To one part add tannic acid. Formation of precipitate indicates BASIC or MODIFIED BASIC DYE.</p> <p>b) To the other part add scoured wool and warm for 4 to 5 minutes.</p> <p>Staining of wool indicates BASIC or MODIFIED BASIC DYE.</p>	<p><i>The solution is not coloured</i></p> <p>Boil 0.5 g of fresh test specimen with 5 percent sodium hydroxide solution for 1 to 2 minutes.</p> <p>If considerable amount of colour is stripped, divide the solution into two parts:</p> <p>a) To one part add scoured cotton and warm for 5 to 10 minutes. Deep staining indicates DIRECT DYE. A little or no staining indicates REACTIVE DYE.</p> <p>b) To another part add formal G. The solution is decolorized and original colour is not restored. DIRECT DYE is confirmed (Non-dischargeable direct dyes will not respond to this treatment).</p> <p>NOTE — For determining whether the dyeing has been after-treated with formaldehyde or copper, chromium or nickel, proceed as follows:</p> <p>a) <i>Direct Dyes After-Treated with Formaldehyde</i> — Warm 0.5 g original test specimen (that is, before deffining) in 5 percent sulphuric acid. Allow it to cool. Discard the sample and test as in (i) or (ii):</p> <p>i) Add 0.1 percent of carbazol dissolved in concentrated sulphuric acid to the solution drop by drop.</p> <p>The formation of a blue colour precipitate indicates the presence of FORMALDEHYDE.</p> <p>OR</p> <p>ii) Add 1 ml concentrated sulphuric acid and 1 ml of 5 percent aqueous chromotropic acid solution to the solution.</p> <p>The formation of violet or red-violet colour either immediately or warming at 60 to 70°C for 5 to 10 minutes indicates presence of FORMALDEHYDE.</p> <p>b) <i>Direct Dyes After-Treated with Copper, Nickel or Chromium</i> — Take 1 ml of 100 volume hydrogen peroxide in a watch glass and add 2 to 3 drops of concentrated ammonia solution. After the evolution of bubbles has ceased, add about 0.1 g of fresh test specimen to it.</p> <p>Any vigorous action indicates the presence of COPPER, NICKEL or CHROMIUM.</p>	<p><i>Boil 0.5 g of fresh test specimen with 16 percent hydrochloric acid for 30 seconds. Cool and add 3 mg of magnesium ribbon or pure zinc dust and warm for 2 to 3 minutes. Place lead-acetate paper on the mouth of the test tube.</i></p> <p>If lead acetate paper turns black and the vapours give smell of hydrogen sulphide gas, SULPHUR or SULPHURIZED VAT DYE is confirmed.</p> <p>Treat a fresh test specimen with cold dilute hypochlorite solution for 5 to 10 minutes.</p> <p>If the specimen becomes colourless, SULPHUR DYE is confirmed.</p> <p>(a) If the colour of the test specimen is destroyed or changed to another tone and is restored on exposure to air, it indicates SULPHUR or SULPHURIZED VAT DYE.</p> <p>(b) If the colour of the test specimen readily becomes brown and changes to black when exposed to air, it indicates ANILINE BLACK.</p> <p>Warm a fresh test specimen with concentrated sulphuric acid for a few seconds and pour the liquid to little cold water.</p> <p>If a dull green colour is produced, ANILINE BLACK is confirmed.</p> <p>(c) If the colour of the test specimen is reduced to another tone and on exposure to air original colour is restored, it indicates a VAT or ANTHRAQUINONE REACTIVE DYE.</p> <p>NOTE — The exceptions are with certain blue dyestuffs where the colour of the reduced vat dye or leuco compound is also blue like the original dyestuff.</p> <p>Treat a fresh test specimen with cold dilute sodium hypochlorite solution for 5 to 10 minutes.</p> <p>If the test specimen is decolorized or its colour becomes light in tone and the original tone is not restored on treatment with 0.2 percent sodium hydro-sulphite solution in cold for 2 minutes, it indicates ANTHRAQUINONE REACTIVE DYE. If not, it indicates VAT DYE.</p> <p>(d) If the color of the test specimen is permanently decolorized or is changed to another tone and does not come back to the original colour on oxidation, it indicates AZOIC or CERTAIN DIAZOTISABLE AZO DYES or NON-ANTHRAQUINONE REACTIVE DYES.</p> <p>1) <i>Acid and Certain Diazotisable Azo Dyes</i> — Boil 0.5 g of fresh test specimen with 5 ml of pyridine for 1 to 2 minutes.</p> <p>Profuse bleeding of colour indicates AZOIC DYE.</p> <p>Slight bleeding of colour indicates DEVELOPED DISPERSE DYE.</p> <p>No bleeding of colour indicates DIRECT DYE (diazotised and developed).</p> <p>Treat 0.5 g of fresh test specimen with non-ionic detergent at 85 to 90°C for 2 to 3 minutes. Discard the test specimen, add 0.5 g of scoured cotton to the solution and boil for another 2 to 3 minutes.</p> <p>If the cotton is stained, DIRECT DYE (diazotised and developed) is confirmed. If there is no staining of the cotton, DEVELOPED DISPERSE DYE is confirmed.</p> <p>2) <i>Reactive Dyes</i> — Reflux 0.5 g of fresh test specimen with a solution containing 1 ml of concentrated sulphuric acid and 2 g/l sodium sulphate diluted to 1 litre with water for 15 minutes. If colour bleeds into solution, add scoured wool. If wool is stained, it indicates REACTIVE DYE of the heterocyclic halogenated type.</p> <p>No bleeding of colour indicates REACTIVE DYES of types other than the heterocyclic halogenated.</p> <p>(e) If the colour of the test specimen does not change, it indicates CHROME DYE.</p> <p>Warm the test specimen with 16 percent hydrochloric acid for 1 to 2 minutes. Cool and add 57 : 43 pyridine water. If colour bleeds into solution, add mordanted wool to the solution and warm for 2 to 3 minutes.</p> <p>If the wool is dyed, it indicates CHROME DYE.</p>	<p>If the colour of the specimen changes in 1 to 2 minutes, it indicates 1:1 METAL COMPLEX DYE.</p> <p>If the colour of the specimen changes after 20 minutes, it indicates 1:2 METAL COMPLEX DYE.</p>	<p>Observe change in colour of the specimen after 1 to 2 minutes and after 20 minutes.</p> <p>If the colour of the specimen changes in 1 to 2 minutes, it indicates 1:1 METAL COMPLEX DYE.</p> <p>If the colour of the specimen changes after 20 minutes, it indicates 1:2 METAL COMPLEX DYE.</p>

ADDITIONAL TESTS

1) *Extraction Test*

Extract 0.5 g of fresh test specimen with 15 ml of 57 : 43 pyridine-water in test tube by keeping it in a beaker of boiling water for 10 to 15 minutes or until sufficient amount of colour bleeds into the reagent. Discard the test specimen and note the colour of the extract. Pour the solution into a separating funnel, add 15 ml of toluene, shake well and allow the two layers to separate. The distribution of dyes between the two layers is as follows:

<i>Toluene Layer</i>	<i>Pyridine-Water Layer</i>
All disperse dyes	All direct dyes
Some neutral dyeing metallized dyes (1 : 2 metal complex dyes)	All basic dyes
Some vat dyes	All acid dyes
Some reactive disperse dyes	All acid dyeing metallized dyes (1 : 1 metal complex dyes)
All azoic combinations	All chrome dyes Logwood: Some neutral dyeing metallized dyes (1 : 2 metal complex dyes)

If the toluene layer is coloured, wash it with water thrice. Separate the toluene layer again and evaporate it. Disperse the residue with a few drops of 10 percent solution of a dispersing agent in water. Add scoured wool and scoured acetate fabric to this and warm for 15 minutes.

If only wool is dyed, it indicates **NEUTRAL DYEING METALLIZED DYE** (that is, 1 : 2 METAL COMPLEX DYE).

If both wool and acetate fabric are dyed, it indicates **DISPERSE DYE**.

If the pyridine-water layer is coloured dark cherry-red, it indicates *Logwood*. Add 1 to 2 ml of concentrated hydrochloric acid, it turns yellowish brown; shake with toluene, the colour remains in pyridine-water layer.

NOTE — In case of chrome dyes, pyridine-water layer is coloured. But sometimes the toluene layer is also stained to a different colouration than original dyeing.

2) *Ash Test*

Ash 0.2 to 0.3 g of fresh test specimen in a porcelain crucible. Add 0.2 to 0.3 g of flux composed of equal parts by weight of powdered sodium carbonate and sodium nitrate. Fuse the mixture and allow it to cool. The presence of any metals is indicated by the colour of the fused mass as follows:

<i>Colour of Fused Mass</i>	<i>Metal Present</i>
Yellow colour	Chromium
Royal blue	Cobalt
Faint blue-green	Copper
Blue-green	Manganese
Brown	Nickel

The presence of cobalt or manganese indicates **NEUTRAL DYEING (1 : 2 METAL COMPLEX DYES)**.

The presence of chromium indicates **DIRECT DYE** after-treated with chromium salt, chrome dyes or metallized dyes (that is, 1 : 1 metal complex dyes and 1 : 2 metal complex dye).

The presence of copper or nickel indicates **DIRECT DYE** after-treated with copper or nickel salt respectively.

3) *Miscellaneous Tests*a) *Test for reactive disperse dye on nylon 6 and 66*

Dissolve 0.5 g of fresh test specimen in formic acid or *o*-chlorophenol and pour the resulting solution into 1 ml of ethylene diamine hydrate diluted with 5 to 10 ml of water. Warm for 5 to 10 minutes and then filter. If the dye remains along with the precipitate, it is a **REACTIVE DISPERSE DYE**.

b) *Test for pigment dye*

If pigment dye is found to be present by the microscopic examination (*see 4.1*), and azoic and vat pigments are found to be absent by the relevant subsequent tests, the pigment dye present may be carbon black or phthalocyanine pigment.

Treat a test specimen with sodium hydroxide and sodium hydrosulphite solution, no discolouration of the specimen indicates **CARBON BLACK**.

Spot a test specimen with concentrated nitric acid, appearance of bright-green tone indicates **PHTHALOCYANINE PIGMENT**.

EVALUATION OF STRENGTH OF WATER SOLUBLE AZO DYES BY REDUCTION WITH TITANIUM TRICHLORIDE

(Source : IS 3859 : 1966)

Water soluble azo (direct and acid) dyes are available in the market generally in diluted form. These dyes are usually checked by dyeing method against standard samples as agreed to between the buyer and the seller. These standard samples are usually designated arbitrarily as hundred percent. This creates difficulties in reporting records of production, import-export statistics or comparing dyes where, due to slight shade differences, the assessment of strength by dyeing method becomes difficult.

This method is mainly useful for production control, production statistics and import-export

statistics; where one normally deals with unblended dyestuffs which are straight dilutions of crude dyes and where one requires an agreed method of test for comparison.

This method is useful for comparing the strengths of dyestuffs which have slight shade differences but have the same colour index number. In case of mixtures, this method may be useful provided the blending composition of the mixtures is the same. However, the strength calculated will be an arbitrary figure because of the differing molecular weights of dyes used in the mixtures.

1 SCOPE

1.1 This method is intended for the determination of strength of water soluble azo dyes listed in Annex A. This method is not applicable to mixtures of dyestuffs and the dyestuffs containing reducible impurities.

2 PRINCIPLE

2.1 The azo dye is reduced by titanium trichloride knowing the amount of titanium trichloride used for reducing the dye, the strength of dye is calculated.

3 PREPARATION OF TEST SAMPLE

3.1 From each container selected, draw small quantities of the dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

4 APPARATUS

4.1 Titanium Trichloride Storage Bottle

See Fig. 1.

4.2 Reduction Flask

See Fig. 2.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in test and distilled water shall be used where the use of water or distilled water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Hydrochloric Acid

(a) concentrated and (b) 25 percent (*m/v*).

5.2 Ammonium Thiocyanate Solution

20 percent (*m/v*).

5.3 Titanium Trichloride Solution

approximately 0.025 N, prepared as follows:

Prepare a 15 percent (*m/v*) solution of titanium trichloride. Take 30 ml of the solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of one litre of water and 60 ml of concentrated hydrochloric acid. Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen for about 15 minutes. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus. The solution should be stored in a bottle painted black to protect it from light.

5.4 Stannous Chloride Solution

prepared by dissolving 30 g of stannous chloride in 100 ml of concentrated hydrochloric acid and diluting it to 200 ml with water. Keep the solution in air-tight bottle.

NOTE — This solution should be prepared fresh after one week.

5.5 Mercuric Chloride Solution

prepared by dissolving 5 g of mercuric chloride in 100 ml of water and filtered, if necessary.

5.6 Diphenylamine Solution

prepared by melting 1 g of diphenylamine and adding to this 100 ml of concentrated sulphuric acid. Dissolve diphenylamine by stirring.

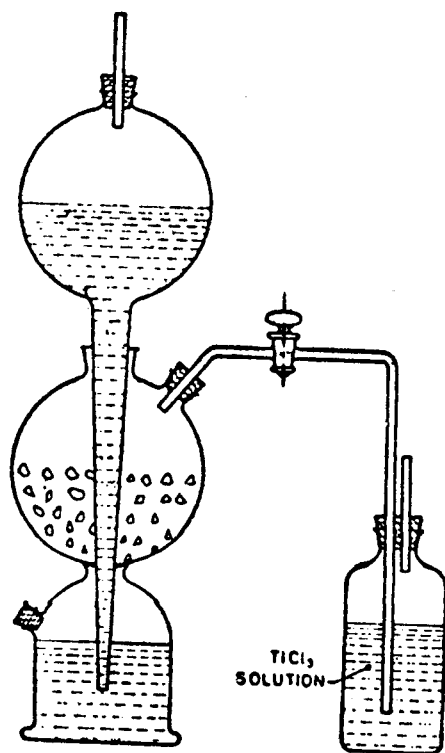


FIG. 1 TiCl_3 SOLUTION STORAGE BOTTLE (PAINTED BLACK)
WITH KIPP'S APPARATUS FOR CO_2 GAS

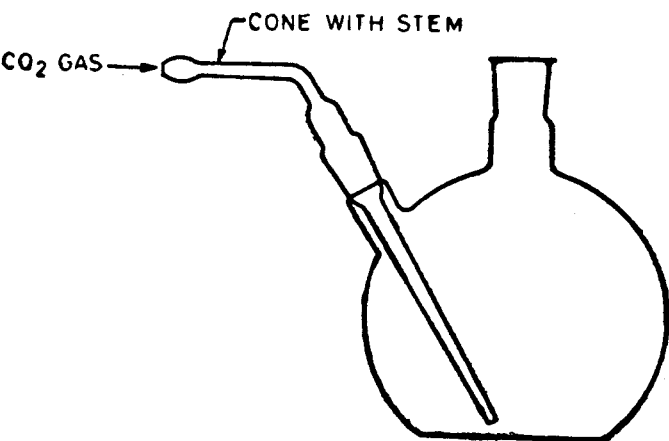


FIG. 2 500-ml REDUCTION FLASK (TWO NECKS, SHORT CENTRE
NECK AND SIDE NECK AT AN ANGLE)

5.7 Sulphuric Acid

1 N.

5.8 Orthophosphoric Acid

free from iron.

5.9 Potassium Dichromate Solution

0.1 N.

5.10 Ferric Ammonium Sulphate Solution

approximately 0.025 N, prepared as follows:

Dissolve 9.803 g of pure ferrous ammonium sulphate [$\text{FeSO}_4 (\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6 \text{H}_2\text{O}$] in 100 ml of water. Add 10 ml of concentrated sulphuric acid and mix well. Dissolve 0.79 g of potassium permanganate in 100 ml of water. Add this solution to the solution of ferrous ammonium sulphate with stirring. At the end, add the permanganate solution carefully so that a ferrous salt is just oxidized. Cool the solution and dilute it to one litre. Determine the normality of the solution by the method given in Annex B.

6 PROCEDURE

6.1 Weigh accurately 1 g of dyestuff from the test sample. Dissolve it in hot water. Pour the solution into a 500-ml graduated flask and make the volume to 500 ml. Mix well.

NOTE — In the case of some azo dyes (see Annex A) 0.5 g of dyestuff should be taken for the test.

6.2 Pipette 50 ml of the solution into a 500-ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml of water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

6.3 Pass carbon dioxide in the storage bottle containing titanium trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously. Add this solution to the dye solution (see 6.2). Immediately boil the contents of the flask for complete reduction (see Note 1). Cool the flask in ice-bath. Maintain continuous flow of carbon dioxide throughout the test. Add 10 ml of 20 percent ammonium thiocyanate indicator solution. Titrate the mixture against 0.025 N ferric ammonium sulphate solution to a permanent change of colour. Note

the reading of ferric ammonium sulphate solution (see Note 2).

NOTES

1 The reduction time for various dyes is given in Annex A.

2 The volume of ferric ammonium sulphate required for titration should not be less than 10 ml.

6.4 Carry out a blank by following the procedure given as above using 50 ml of water in place of dye solution.

7 CALCULATION

7.1 Calculate the titanium value, in percent of the dye, by the following formula:

$$T = \frac{(B - A) \times N \times M \times V}{E \times C \times W \times 10}$$

where

T = titanium value, percent, of the dye;

B = volume in ml of ferric ammonium sulphate solution required for blank (see 6.4);

A = volume in ml of ferric ammonium sulphate solution required for the dye (see 6.3);

N = normality of ferric ammonium sulphate solution (see B-1.3);

M = molecular mass of dye;

V = volume of dye solution (see 6.1);

E = number of hydrogen atoms consumed per molecule of dye (see Note);

C = volume in ml of aliquot taken for titration (see 6.2); and

W = mass in g of the sample taken for test.

NOTE — Each azo group requires 4, nitro group 6 and hydrozo group 2 atoms of hydrogen for reduction.

7.2 Repeat the test prescribed in 6.3 twice and calculate the titanium value in percent of the dye.

7.3 Calculate the average of the values obtained in 7.1 and 7.2.

8 REPORT

8.1 The report shall include the strength of the dye (titanium value) in percent.

ANNEX A

GENERAL INFORMATION ABOUT ANALYSIS OF AZO DYES

Sl No.	Colour Index Number	Name of Dye	Molecular Mass of Dye as Acid	Hydrogen Equivalent	Concentration of Dye in g/500 ml	Reduction Time in Minutes
1.	13065	Acid yellow 36	353	4	1	12
2.	14710	Acid red 4	358	4	1	12
3.	14720	Acid red 14	458	4	1	10
4.	15510	Acid orange 7	328.5	4	1	10
5.	15620	Acid red 88	378	4	1	12
6.	16230	Acid orange 10	408	4	0.5	10
7.	16255	Acid red 18	538	4	1	10
8.	18055	Acid violet 7	522	4	1	10
9.	18065	Acid red 35	479	4	1	13
10.	20470	Acid black 1	572	14	0.5	10
11.	22120	Direct red 28	652	8	1	16
12.	22145	Direct red 10	653	8	1	16
13.	22245	Acid red 85	744	8	0.5	12
14.	22310	Direct red 1	581	8	1	10
15.	22345	Direct brown 59	659	8	1	10
16.	22590	Direct blue 2	764	8	1	10
17.	23500	Direct red 2	680	8	1	10
18.	24895	Direct yellow 12	636	8	1	8
19.	26370	Acid black 24	701	8	0.5	13
20.	27290	Acid red 73	512	8	1	8
21.	27855	Direct violet 7	659	8	0.5	12
22.	27905	Direct violet 51	675	8	1	8
23.	29100	Direct red 31	669	8	0.5	8
24.	29150	Direct orange 26	712	8	1	15
25.	29160	Direct red 23	769	8	1	8

ANNEX B

DETERMINATION OF NORMALITY OF FERRIC AMMONIUM SULPHATE

B-1 PROCEDURE

B-1.1 Pipette 100 ml of ferric ammonium sulphate solution (see 5.10) into a 500-ml conical flask and add 5 ml of concentrated hydrochloric acid. Heat it to a about 85°C. Add stannous chloride solution dropwise from a burette while swirling the flask over a white background until the yellow colour of the solution has nearly disappeared. Complete the reduction by adding dilute solution of stannous chloride (see Note 1) dropwise with agitation after each addition until the liquid has a faint green colour free from any tinge of yellow. Dilute the solution in the flask to thrice its volume with water. Rapidly cool the solution under the tap with stopper on and remove the excess of stannous chloride by adding 10 ml of mercuric chloride solution rapidly in one lot. A slight silky white precipitate of mercurous chloride is obtained (see Note 2). Add 50 ml of 1 N sulphuric acid, 2.5 ml of *o*-phosphoric acid and three drops of diphenylamine solution as indicator. Titrate slowly against 0.1 N potassium dichromate solution while stirring constantly to the first permanent tinge of purple or violet blue colouration.

NOTES

1 Dilute stannous chloride solution should be prepared by diluting stannous chloride solution (see 5.4)

with two volumes of 10 percent hydrochloric acid.

2 Excess of stannous chloride solution should not be added. A heavy, grey or black precipitate is the indication of too much stannous chloride solution. Discard the solution and start with a fresh solution of ferric ammonium sulphate.

B-1.2 Carry out a blank by following the procedure prescribed in B-1.1 but using 100 ml of water in place of 100 ml of ferric ammonium sulphate solution.

B-1.3 Calculate the normality of ferric ammonium sulphate solution by the formula given below:

$$N = \frac{(A - B) \times N_1}{100}$$

where

N = normality of ferric ammonium sulphate solution;

A = volume in ml of potassium dichromate solution required to titrate the solution (see B-1.1);

B = volume in ml of potassium dichromate solution required to titrate the blank (see B-1.2); and

N_1 = normality of potassium dichromate solution.

DETERMINATION OF STRENGTH OF DIRECT DYESTUFFS BY DYEING TEST

(Source : IS 4459 : 1967)

This method is a general one and, wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. In order to compare the exhaust property of the dyestuff under test and the standard dyestuff, it is recommended to carry out the exhaust dyeings. Normally the yellow

dyeings are difficult to compare visually. It is, therefore, advisable to add a constant amount of similar type of blue dyestuff and get the dyeings in green colour. The green dyeings would facilitate the visual comparison in determining the strength of the dyestuff.

1 SCOPE

1.1 This method is intended for the determination of strength of water-soluble direct dyes by normal dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the experimental results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Before dyeing the hanks, carry out a preliminary test to determine the approximate strength of the dyestuff under test by the method given in 5.1.1.

5.1.1 Take separately 10 ml of 0.1 percent solution of the dyestuff under test and the standard dyestuff (see A-4.1). Put a spot each of the solutions of the dyestuff under test and the standard dyestuff on the filter paper. Visually examine the spots. If they are not equal, dilute the stronger solution with water to such an extent as to get approximately equal depth of spot on filter paper. Calculate the approximate strength of the dyestuff under test by the following formula:

Approximate strength of dyestuff under test,

$$\text{percent} = \frac{V_t}{V_s} \times 100$$

where

V_t = final volume in ml of solution of the dyestuff under test, and

V_s = final volume in ml of solution of the standard dyestuff.

5.2 Prepare dyeings of 100 and 95 percent of 1 percent depth of the dyestuff under test by following the procedure given in Annex A.

NOTE — Depending on the strength of dyestuff under test, the hanks may be dyed in other suitable depths. One of the two dyeings may be 0.5, 1.5 or 2 percent depth (instead of 1 percent) so as to give medium shade which is convenient for visual comparison. The other dyeing would be of 95 percent strength of the first dyeing.

5.3 Similarly, prepare dyeings of different percentages (see Notes 1 and 2) of standard dyestuff by following the procedure given in Annex A.

NOTES

1 The percentages of strength of dyeings of standard dyestuffs should be so arranged that the difference between the two consecutive dyeings is approximately 5 percent and the dyeings are well-distributed on either side of the approximate percentage strength determined by the spot test.

Example

If the strength of the dyestuff, as determined by the spot test, is 60 percent, then the different dyeings of the standard dyestuff should be of 60 percent, 60 ± 5 percent (57 and 63 percent) and 60 ± 10 percent (54 and 66 percent).

2 The dyeings obtained with the dyestuff under test should fall within the range of dyeings obtained with the standard dyestuff.

5.4 Carry out the dyeings of the fresh hanks in the exhaust liquor of the above dyeings by following the procedure given in A-4.3.

5.5 Compare the dyeings obtained in 5.2 and 5.3 (see Note). Select a dyeing of the standard

dyestuff which exactly matches with one of the dyeings of the dyestuff under test. Note the percentages of the dyeings which match exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a thickness to avoid the effects of other backing on the appearance. The hanks should be compared, if possible in north skylight. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

6 CALCULATION

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test matching with A .

7 REPORT

7.1 The report shall include the following information:

- Strength of dyestuff in percent,
- Shade of the dyestuff in comparison with that of the standard dyestuff, and
- Shade of the exhaust dyeings of the dyestuff under test in comparison with that of the standard dyestuff.

ANNEX A

GENERAL METHOD FOR DYEING OF DIRECT COLOURS

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel beakers or dye-vessels provided for mechanically agitated dye baths.

A-1.2 Watch Glass

A-1.3 Graduated Pipettes

capable of measuring correct to 0.1 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Soda Ash Solution

10 percent (w/v).

A-2.3 Sodium Chloride Solution

10 percent (w/v).

NOTE — 10 percent solution (w/v) of sodium sulphate, anhydrous may be used.

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, bleached, unmercerized cotton yarn having no

finishing chemical or blueing agent shall be used in this test. Each hank should weigh 10 ± 0.1 g (see Note 2).

NOTES

1 Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

- Count — 10 tex \times 2 (or 60s/2),
- Twist per metre — 750, and
- Cuprammonium fluidity not more than 5 rhes.

2 If the mass of the hank is not 10 ± 0.1 g, then it should be weighed accurately and the amount of dyestuff and the chemicals to be taken should be calculated accordingly.

A-3.2 Preparation of Test Hanks

The hanks shall be treated in boiling water for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of Dyestuff Solutions

A-4.1.1 Weigh accurately 1.0 g of dyestuff under test. Paste it thoroughly with cold water. Add hot water and dissolve the dyestuff. If necessary, heat the solution till it becomes clear. Dilute the solution to 1 litre with cold water.

A-4.1.2 Similarly, prepare solution of the standard dyestuff by following the procedure given in A-4.1.1 but taking the standard dyestuff instead of the dyestuff under test.

A-4.2 Dyeing (for 10 ± 0.1 g Hank)

A-4.2.1 Pipette out separately the required amount of solution of dyestuff under test (see Note 1) in the dye vessels so as to give 100 and 95 percent dyeings of the 1 percent depth (see 5.2 and Note under it). Add requisite quantity of water to make the volume of dye liquor 300 ml (see Note 2). Add 5 to 10 ml of sodium chloride solution depending on the nature of the dyestuffs (see Note 3). Stir the dye liquors and enter the wetted hanks at 40°C. Turn the hanks frequently so as to obtain level dyeings. Slowly raise the temperature of water bath to 80°C within 20 minutes. Remove the hanks from the dye bath, add second lot of 5 to 10 ml of sodium chloride solution and stir. Enter the hanks into the dye bath and raise the temperature of the water bath to boil and continue to dye at boil for 40 minutes (see Notes 4 and 5). At the end of dyeing, squeeze the dyed hanks returning the squeezed out solutions back to respective dye baths and rinse them well in cold water. Dry the hanks in oven at a temperature not exceeding 70°C.

NOTES

1 The amount of dye solution to be taken for dyeing to give required percentage may be calculated as follows:

Volume in ml of 0.1 percent solution of dyestuff = $p \times y$
where

p = percentage depth of dyeing (for example, 0.5, 1 or 2 percent); and

y = percentage dyeing (for example, 95 or 100 percent).

General formula for the calculation is as follows :

Volume in ml of stock solution

$$\text{of dyestuff required for dyeing} = \frac{w \times p}{y} \times \frac{y}{100}$$

where

w = mass in g of the hank;

p = percentage depth (for example, 0.5 or 1 percent);

y = strength in percent of the dyestuff solution (0.10 percent in this case); and

y = percentage dyeing (for example, 95, 100 or 105 percent).

2 The liquor ratio should be 1:30.

3 One percent soda ash on the mass of the hanks should also be added, if dyestuff is sensitive to acid.

4 During dyeing, water should be added to make up the volume lost by evaporation.

5 The total time taken for dyeing should be 70 minutes and the temperature should be raised to boil during first 30 minutes.

A-4.2.2 Similarly, pipette out separately different amounts of solution of the standard dyestuff in different dye vessels (see Note). Dye the hanks by following the method given in A-4.2.1.

NOTE — The dyeings with the solutions of the dyestuff under test and of the standard dyestuff should be done simultaneously in the same water bath.

A-4.3 Exhaust Dyeing

Make up the volumes of the exhaust liquors (see A-4.2.1 and A-4.2.2) to the original volume (300 ml). Enter fresh wetted hanks in the exhausted dye baths and continue dyeing for 30 minutes at boil. Squeeze the hank, rinse well in cold water and dry.

DETERMINATION OF STRENGTH OF FAST BASES

(Source : IS 4360 : 1967)

Fast bases are marketed in different strengths. This method is useful for production control, production and import-export statistics, where one normally deals with unblended fast bases.

1 SCOPE

1.1 This method is intended for the determination of strength of fast bases as listed in Annex A.

1.2 This method is not applicable to mixtures of fast bases.

2 PRINCIPLE

2.1 The fast bases (aromatic primary amines) are quantitatively diazotized with nitrous acid. Knowing the quantity and strength of the sodium nitrite used in the reaction, the strength of fast bases is determined.

3 PREPARATION OF TEST SAMPLE

3.1 From each container selected, draw small quantities of the fast base by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 50 g.

4 APPARATUS

4.1 Mechanical Stirrer

4.2 Burette

with a small attachment as shown in Fig. 1.

4.3 Beakers

of 1 litre capacity.

4.4 Water Bath

5 REAGENTS

5.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water or distilled water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 Sodium Nitrite Solution

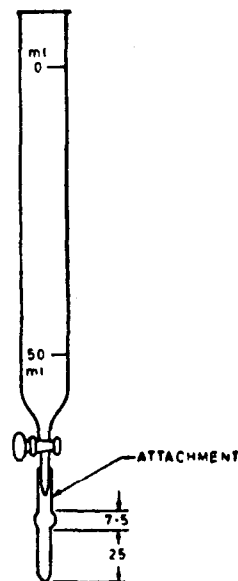
0.5 N.

5.3 Hydrochloric Acid

concentrated.

5.4 Potassium Bromide Solution

25 percent (w/v).



All dimensions in millimetres.

FIG. 1 BURETTE WITH AN ATTACHMENT

5.5 Standard Sulphanilic Acid Solution

0.5 N.

6 PROCEDURE

6.1 Take about 5 g of fast base (see Note 1) from the test sample and weigh it accurately. Transfer it to 1-litre beaker. Add 50 ml of hydrochloric acid and 500 ml of distilled water. Dissolve the base completely by heating, if necessary (see Note 2). Cool it to room temperature and add 20 ml of potassium bromide solution (see Note 3). Keep the beaker in the water bath containing chopped ice and water. Bring down the temperature of the contents in the beaker to about 5°C (see Notes 4 and 5).

NOTES

1 For CI azoic diazo component 48, 3 g of test sample is sufficient.

2 For CI azoic diazo component 4 and 8, the base is first dissolved in 100 ml of glacial acetic acid by warming, if necessary. After cooling the solution to 20°C, a mixture of 500 ml of water and

30 ml of concentrated hydrochloric acid is added. The solution is titrated immediately against sodium nitrite. Any precipitate formed initially will dissolve on addition of nitrite solution.

3 Potassium bromide is added as catalyst.

4 The temperature is brought to 5°C to avoid the loss of nitrous acid.

5 CI azoic diazo component 1 and 5 may precipitate on cooling the solution to about 5°C. The diazotization should be carried out immediately as with time lapse, crystals may aggregate and lower the diazotization rate considerably.

6.2 Immerse the tip of the burette well under the surface of the solution. Keep the solution under agitation with mechanical stirrer. Add the nitrite solution from the burette in small portions, and test the reaction mixture by putting a drop on starch iodide paper. Note the reading when the reaction mixture gives instantaneous blue colour with starch iodide paper (see Note 1).

NOTES

1 The rate of addition of nitrite solution depends on how rapidly the base consumes the nitrous acid. There should be no large excess of nitrite present at any time, since this may cause loss of nitrous acid. At first, the nitrite should be added in small portions and the solution tested by putting a drop on starch iodide paper. If the base consumes the nitrous acid rapidly, nitrite should be added more rapidly and *vice versa*. As the end-point is approached, nitrite will be consumed more slowly. The end-point is recorded when an immediate blue colour appears on starch iodide paper which can be obtained repeatedly during a period of 5 minutes without further addition of sodium nitrite.

2 For the fast bases CI azoic diazo component 4, reaction mixture is strongly coloured. To observe the end-point, it is necessary to rinse the starch

iodide paper with distilled water immediately after spotting. A blue ring on the starch iodide paper indicates excess of sodium nitrite.

6.3 Determine the normality of the sodium nitrite solution by titrating against standard sulphanilic acid solution (see 5.5).

7 CALCULATION

7.1 Calculate the strength of the fast base by the following formula:

$$P = \frac{A \times N \times M}{10 \times W \times B}$$

where

P = strength in percent by weight of the fast base;

A = volume in ml of sodium nitrite solution;

N = normality of sodium nitrite solution (see 6.3).

M = molecular mass of the fast base;

W = mass in g of the fast base (see 6.1); and

B = number of amino groups per molecule.

7.2 Repeat the test prescribed in 6.1 and 6.2 twice and calculate the strength of fast base by the formula given in 7.1.

7.3 Calculate the average of the values obtained in 7.1 and 7.2.

8 REPORT

8.1 Report the value obtained in 7.3 as the strength of the fast base in percent.

ANNEX A

GENERAL INFORMATION ABOUT ANALYSIS OF FAST BASES

SL No.	*COLOUR INDEX DESIGNATION	COMMERCIAL OR TRADE NAME	COLOUR INDEX NUMBER	MOLECULAR MASS†	NO. OF AMINO GROUPS PER MOLECULE
1	Azoic diazo component 1	Fast bordeaux GP base	CI 37135	168	1
2	Azoic diazo component 2	Fast orange GC base	CI 37005	164 (hydrochloride)	1
3	Azoic diazo component 3	Fast scarlet GG base	CI 37010	162	1
		Fast scarlet GGS base		422 (Sulphate)	(2)
4	Azoic diazo component 4	Fast garnet GB base	CI 37210	225	1
		Fast garnet GBC base		261.5 (hydrochloride)	1
5	Azoic diazo component 5	Fast red B base	CI 37125	168	1
6	Azoic diazo component 8	Fast red GL base	CI 37110	152	1
7	Azoic diazo component 10	Fast red R base	CI 37120	157.5	1
		Fast red RC base		194.0 (hydrochloride)	1
8	Azoic diazo component 11	Fast red TR base	CI 37085	178 (hydrochloride)	1
9	Azoic diazo component 12	Fast scarlet G base	CI 37105	152	1
10	Azoic diazo component 13	Fast scarlet R base	CI 37130	168	1
		Fast scarlet RC base		204.5 (hydrochloride)	1
11	Azoic diazo component 32	Fast red KB base	CI 37090	178 (hydrochloride)	1
12	Azoic diazo component 44	Fast yellow GC base	CI 37000	164 (hydrochloride)	1
13	Azoic diazo component 48	Fast blue B base	CI 37235	244	2

*Colour Index (1975), ed 3, Society of Dyers and Colourists, UK; and American Association of Textile Chemists and Colourists, USA.

†The molecular mass of the bases varies, depending upon whether the base is a free base, hydrochloride or a sulphate. The common commercial names of the bases are also given as industry is familiar with it.

EVALUATION OF STRENGTH AND SHADE OF FAST BASES BY DYEING TEST

(Source : IS 6526 : 1971)

Fast bases are marketed in different strengths. This method prescribed would be useful for assessing both the strength and shade of the fast bases against mutually accepted standard. Unlike other types of dyes, the exhaustion of fast bases from the

developing bath is comparatively less. In order to reduce the chances of error in evaluating the strength of fast bases, two additional exhaust dyeings are recommended instead of a single dyeing and the average of the three dyeings is determined.

1 SCOPE

1.1 This method is intended for the evaluation of strength and shade of fast bases listed in Table 1 by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of the fast base by suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 50 g.

3 STANDARD FAST BASE

3.1 The standard sample of fast base, against which the strength and shade of fast base under test is evaluated shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH

5.1 Out of the sets of conditions of test laid down in Table 1, choose the set of conditions applicable to the fast base under test.

5.2 Prepare dyeing of the recommended depth of shade of fast base under test by following the procedure given in Annex A. Prepare simultaneously additional dyeing of the fast base under test with the depth of shade 90 percent of the first by taking 90 percent of the quantity of base taken for the first bath.

5.3 Simultaneously prepare dyeings of different depths of shade of standard fast base by following the procedure given in Annex A.

NOTE — The depth of shade of each of the standard fast base should be so arranged that the difference between the two consecutive dyeings is the same and the dyeings are well distributed on either side of the recommended depth of shade taking into consideration the expected strength of the sample.

5.4 Carry out second set of dyeings with fresh naphtholated hanks in the exhaust liquor of the above dyeings by following the procedure given in A-4.5.

5.4.1 Carry out third set of dyeings with naphtholated hanks in the exhaust liquor obtained in 5.3 by following the procedure given in A-5.1.

5.5 Compare the dyeings as obtained in 5.2 and 5.3. Select dyeing of the standard fast base which exactly matches with one of the dyeings of the fast base under test. Note the depths of the shade of dyeings which match exactly.

NOTE — The dyeings should be spread out properly and compared in North daylight.

6 CALCULATION

6.1 Calculate the strength of fast base under test by comparing the first set of dyeings by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of fast base in percent;

A = depth of shade in grams per litre of the dyeing of standard fast base; and

B = depth of shade in grams per litre of the dyeing of fast base under test, comparing with that of standard.

6.1.1 Similarly, calculate the strength of the fast base under test from the other two sets of exhaust dyeings.

6.2 Calculate the average of the values obtained in 6.1 and 6.1.1.

7 REPORT

7.1 The report shall include the following information:

- a) Strength of the fast base in percent, and
b) Shade of the fast base in comparison with the shade of the standard fast base.

TABLE 1 GENERAL INFORMATION ABOUT DYEING OF FAST BASES

(Clauses 1.1 and 5.1)

Sl. No.	COLOUR INDEX DESIGNATION (COMMON COMMERCIAL NAME OF THE BASE)	COLOUR INDEX NO. OF BASE	NAPHTHOLATING BATH			DEVELOPING BATH			SOAP-ING TEMPERATURE
			Common Commercial Name of Naphthol	Concentration of Naphthol	Concentration of NaCl	Concentration of Base	Concentration of NaCl	pH	
(1)	(2)	(3)	(4)	(5) g/l	(6) g/l	(7) g/l	(8) g/l	(9)	(10)
i)	CI azoic diazo component (Yellow GC) 44	37000	Naphthol ASG	2	15	1.1	20	4 to 5	Boil
ii)	CI azoic diazo component (Orange GC) 2	37005	Naphthol AS	2	20	1.0	20	4 to 5	65°C
iii)	CI azoic diazo component (Orange GR) 6	37025	Naphthol AS	2	20	0.9	20	4 to 5	Boil
iv)	CI azoic diazo component (Scarlet G) 12	37105	Naphthol AS	2	20	0.9	20	4 to 5	Boil
v)	CI azoic diazo component (Scarlet GGS) 3	37010	Naphthol AS	2	20	1.4	20	4 to 5	Boil
vi)	CI azoic diazo component (Scarlet GG) 3	37010	Naphthol AS	2	20	1.1	20	4 to 5	Boil
vii)	CI azoic diazo component (Scarlet RC) 13	37130	Naphthol AS	2	20	1.1	20	4 to 5	Boil
viii)	CI azoic diazo component (Scarlet R) 13	37130	Naphthol AS	2	20	1.1	20	4 to 5	Boil
ix)	CI azoic diazo component (Red B) 5	37125	Naphthol ASBO	1	20	0.9	20	4 to 5	Boil
x)	CI azoic diazo component (Red GL) 8	37110	Naphthol AS	2	20	0.9	20	4 to 5	Boil
xi)	CI azoic diazo component (Red 3 GL) 9	37040	Naphthol AS	2	20	0.9	20	4 to 5	Boil
xii)	CI azoic diazo component (Red KB) 32	37090	Naphthol AS-SW	1	—	1.0	20	5.5 to 6.5	Boil
xiii)	CI azoic diazo component (Red RC) 10	37120	Naphthol ASOL	1.5	20	1.1	20	5.5 to 6.5	Boil
xiv)	CI azoic diazo component (Red R) 10	37120	Naphthol ASOL	1.5	20	0.9	20	5.5 to 6.5	Boil
xv)	CI azoic diazo component (Red TR) 11	37085	Naphthol ASTR	1	20	1.0	20	5.5 to 6.5	Boil
xvi)	CI azoic diazo component (Bordeaux GP) 1	37135	Naphthol AS	2	20	1.1	20	4 to 5	Boil
xvii)	CI azoic diazo component (Garnet GBC) 4	37210	Naphthol AS	2	20	1.7	10	4 to 5	Boil
xviii)	CI azoic diazo component (Violet B) 41	37165	Naphthol AS	2	20	1.8	20	6 to 7	Boil
xix)	CI azoic diazo component (Blue B) 48	37235	Naphthol AS	2	20	0.8	20	6 to 7	Boil
xx)	CI azoic diazo component (Blue BB) 20	37175	Naphthol AS	2	20	2.0	20	6 to 7	Boil

ANNEX A

DYEING PROCEDURE

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel beakers or dye vessels of 250 to 300 ml capacity.

A-1.2 Watch Glass

A-1.3 Pipettes, Graduated

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of dye bath.

NOTE — For rinsing, water having hardness of not more than 60 ppm, expressed as calcium carbonate may be used.

A-2.2 Hydrochloric Acid

32°Tw.

A-2.3 Sodium Nitrite

98 percent.

A-2.4 Sodium Acetate

A-2.5 Acetic Acid

50 percent (w/v).

A-2.6 Sodium Chloride

10 percent solution (w/v).

A-3 PREPARATION OF HANKS

A-3.1 Test Hanks

Hanks shall be of scoured, bleached, unmercerized cotton yarn having no finishing chemical or blueing agent. Each hank shall weigh 10 ± 0.1 g.

NOTE — Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for the test:

- Count 10 tex \times 2 (or 60 $\frac{1}{2}$),
- Twist 750/m, and
- Cuprammonium fluidity not more than 5 rhes.

A-3.2 Preparation of Test Hanks

The hanks shall be treated in boiling water for 10 minutes and squeezed evenly to contain approximately its own mass of water, cooled and entered in the naphtholating bath.

A-4 PROCEDURE

A-4.1 For naphtholation, developing and soap-
ing, the liquor ratio shall be 1 : 20.

A-4.2 Naphtholation

Prepare a naphtholating bath in water containing requisite amounts of suitable naphthol and the recommended quantity of sodium chloride (see Table 1). Naphtholate the required number of hanks at room temperature for 30 minutes. Take out the hanks, squeeze them to contain liquor about equal to their mass and develop separately in the fast base diazo solutions.

A-4.3 Preparation of Developing Solution

A-4.3.1 Prepare a developing solution in water containing a suitable concentration of fast base (see Table 1) under test by following the procedure given in Annex B.

A-4.3.2 Similarly, prepare the solution of the standard fast base following the procedure given above but taking a standard fast base instead of fast base under test.

A-4.4 Developing

A-4.4.1 Take in each dye vessel required quantity of diazo solution of the fast base under test. Make up the volume to 160 ml with cold water, add with stirring 40 ml of sodium chloride solution. Enter the naphtholated and squeezed hanks separately in each dye bath and work for 30 minutes. Squeeze the hanks to contain liquor about equal to their own mass. Preserve the developing bath for carrying out exhaust dyeings. Wash the dyed hanks.

A-4.4.2 Similarly, develop the naphtholated hanks with the diazo solution of the standard fast base.

A-4.5 Make up the volumes of the developing baths to 200 ml with cold water (see Note under A-4.5.1). Enter in each dye bath freshly naphtholated hanks and work them for 30 minutes. Take out the dyed hanks, squeeze them and wash.

A-4.5.1 Repeat the developing third time in the same bath by taking freshly naphtholated hanks (see Note).

NOTE — Add acetic acid (10 percent) to get pH of the developing bath as given in Table 1.

A-4.6 Soap all the three sets of dyeings (see A-4.4.1, A-4.4.2, A-4.5 and A-4.5.1) and boil for 30 minutes in a bath containing 5 g/l of soap and 2 g/l of soda ash. Wash thoroughly and dry.

ANNEX B

METHOD FOR DIAZOTIZATION OF FAST BASES

1) Fast Yellow GC Base

Paste thoroughly 10 g fast yellow GC base with 50 ml hot water. To this, add 150 ml cold water and 12 ml hydrochloric acid 32°Tw. Cool the solution to 10°C and add at a moderate rate 5 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring. After 15 to 20 minutes of diazotizing, neutralize the solution with 10 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 10°C.

NOTE — For naphthol ASG combination, 4.0 ml/l of acetic acid (50 percent) should be added.

2) Fast Orange GC Base

Paste thoroughly 10 g fast orange GC base with 50 ml hot water. To this, add a mixture of 150 ml cold water and 12 ml hydrochloric acid 32°Tw. Cool the solution to 10°C and add at a moderate rate 5 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring. After 15 to 20 minutes of diazotizing, neutralize the solution with 10 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 10°C.

3) Fast Orange GR Base

Paste thoroughly 10 g fast orange GR base with 15 ml cold water and add 5.5 g sodium nitrite (98 percent). When the sodium nitrite has dissolved completely, cool the paste and add quickly with vigorous stirring 300 ml cold water containing 22.5 ml hydrochloric acid 32°Tw. Allow the diazo to stand for 15 to 20 minutes with occasional stirring. Filter and neutralize the excess acid with 12.5 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 12°C.

4) Fast Scarlet G Base

Dissolve 10 g fast scarlet G base in a mixture of 20 ml hydrochloric acid 32°Tw, and 100 ml boiling water. Cool the solution 10 to 15°C by

adding 150 ml ice cold water. Add slowly 5 g sodium nitrite (98 percent) dissolved in 25 ml cold water with vigorous stirring. After 30 minutes, dilute the solution and neutralize with 10 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in about 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 10°C.

5) Fast Scarlet GGS Base

Paste 10 g fast scarlet GGS base with 40 ml cold water containing 20 ml hydrochloric acid 32°Tw. After standing for 5 minutes run in, in a thin jet 3.6 g sodium nitrite (98 percent) dissolved in 25 ml cold water, whilst stirring the paste vigorously. Immediately after the solution becomes clear, filter through a hair sieve and make up to about 200 ml total volume with ice cold water. Neutralize with 25 g sodium acetate dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 10 to 15°C.

6) Fast Scarlet GG Base

Add to 10 g fast scarlet GG base 54 ml near boiling water. Stir to melt the base completely. Add whilst stirring 36 ml hydrochloric acid 32°Tw. Cool the paste to 10 to 15°C with ice. Run in, in a thin jet 5.5 g sodium nitrite (98 percent) dissolved in 35 ml cold water whilst stirring the paste vigorously. Immediately after the solution becomes clear, filter through a hair sieve and make up to about 300 ml total volume with ice cold water. Neutralize with 37.5 g sodium acetate dissolved in 75 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 10 to 15°C.

7) Fast Scarlet RC Base

Paste thoroughly 10 g fast scarlet RC base with 12.5 ml cold water containing 10 ml hydrochloric acid 32°Tw. To this paste add 200 ml cold water whilst stirring. Cool the solution to 12°C and add 4 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring. After 20 to 30 minutes of diazotizing, neutralize the solution with 7.5 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 5 ml acetic acid 50 percent or 7.5 g aluminium sulphate crystals dissolved in 40 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 12°C.

8) Fast Scarlet R Base

Paste 10 g fast scarlet R base with a mixture of 20 ml hydrochloric acid 32°Tw and 20 ml cold water. Add to this 300 ml near boil water and stir to dissolve. Cool the solution to 10 to 15°C by adding 100 ml cold water and ice. Add to this quickly whilst stirring 60 g sodium nitrite (98 percent) dissolved in 30 ml cold water. After 20 to 30 minutes, neutralize the solution with 11 g sodium acetate dissolved in 35 ml cold water. Finally, add the alkali binding agent 3.5 ml acetic acid 50 percent or 11.0 g aluminium sulphate crystals dissolved in about 80 ml cold water. Make up the final volume to 500 ml with cold water.

Diazotizing temperature — 10 to 15°C.

9) Fast Red B Base

Paste thoroughly 10 g fast red B base with 15 ml hot water, and 5 g sodium nitrite (98 percent) after obtaining complete solution of the sodium nitrite, cool the paste to 15°C, and add in small quantities with vigorous stirring to a mixture of 200 ml cold water and 17.5 ml hydrochloric acid 32°Tw. Allow to stand for 30 minutes with frequent stirring. Filter and neutralize with 8.5 g sodium acetate dissolved in 25 ml cold water. Then finally add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 15°C.

10) Fast Red GL Base

Paste thoroughly 10 g fast red GL base with 20 ml hot water and 5 g sodium nitrite (98 percent). When the nitrite is dissolved completely, cool the paste to 15°C and add in small quantities with vigorous stirring to a mixture of 200 ml cold water and 20 ml hydrochloric acid 32°Tw. After 30 minutes of frequent stirring dilute the solution to about 400 ml with cold water and neutralize by either method (a) or (b):

- a) Add 5 g sodium formate dissolved in 10 ml cold water. Finally, add the alkali binding agent 7.5 ml formic acid 50 percent or 17.5 g aluminium sulphate crystals dissolved in 75 ml cold water. Make the final volume to 500 ml with cold water.
- b) Dissolve 4 g soda ash and 16.6 g aluminium sulphate separately in four times the quantity of water and stir the soda ash solution in the aluminium solution. Add this mixture to the diazo solution. The diazo solution should react neutrally to Congo Red paper. If necessary, add a little more soda ash. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 15°C.

11) Fast Red 3 GL Base

Paste thoroughly 10 g fast red 3 GL base with 40 ml hot water and add 5 g sodium nitrite (98

percent). After sodium nitrite has completely dissolved, cool the paste and add it in portions with vigorous stirring in 200 ml ice cold water containing 18 ml hydrochloric acid 32 Tw. Allow the diazo to stand for 30 minutes with occasional stirring. Filter the diazo and dilute with 200 ml cold water. Neutralize the excess acid using 10 g sodium acetate dissolved in about 25 ml cold water. Finally, add to the alkali binding agent 6 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 10°C.

12) Fast Red KB Base

Dissolve 10 g fast red KB base by adding 100 ml hot water containing 10 ml hydrochloric acid 32°Tw. Filter the hot solution, crush the lumps and dissolve in the solution. Dilute the solution with water and ice to 250 ml volume at 10°C. Add slowly 4 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring. After 20 to 30 minutes of diazotizing, neutralize the solution with 7.5 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 6 ml acetic acid 50 percent or 7.5 g aluminium sulphate crystals dissolved in about 40 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 10°C.

13) Fast Red RC Base

Paste 10 g fast red RC base with 12.5 ml hot water. To this, add a mixture of 100 ml cold water and 10 ml hydrochloric acid 32°Tw. Cool the solution to 10°C and add at a moderate rate 5 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring vigorously. After diazotization for 30 minutes, the solution is neutralized with 7.5 g sodium acetate dissolved in 25 ml cold water. Then finally, add the alkali binding agent 3 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in about 50 ml cold water. Make to dye-bath a further addition of 7.5 g/l sodium acetate before use. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 10°C.

14) Fast Red R Base

Paste 10 g fast red R base with 20 ml hot water. To this add a mixture of 200 ml cold water and 20 ml hydrochloric acid 32°Tw and add at a moderate rate 7.5 g sodium nitrite (98 percent) dissolved in about 40 ml cold water, whilst stirring vigorously. After diazotizing for about 30 to 40 minutes, the solution is neutralized with 11.0 g sodium acetate dissolved in 35 ml cold water. Then finally, add the alkali binding agent 3.5 ml acetic acid 50 percent or 15 g aluminium sulphate

crystals dissolved in about 75 ml cold water. Add to the dye-bath a further addition of 7.5 g/l sodium acetate before use. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 10°C.

15) Fast Red TR Base

Paste thoroughly 10 g fast red TR base with 200 ml containing 10 ml hydrochloric acid 32°Tw. The solution is cooled to 12°C and add at a moderate rate 4 g sodium nitrite (98 percent) dissolved in about 25 ml cold water whilst stirring the paste vigorously. After 20 to 30 minutes of diazotizing, neutralize the solution with 7.5 g sodium acetate dissolved in about 25 ml cold water. Finally, add the alkali binding agent 1.5 ml acetic acid 50 percent or 5 g aluminium sulphate crystals dissolved in 25 ml cold water. Add to the dye-bath 10 g/l sodium acetate before use. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 12°C.

16) Fast Bordeaux GP Base

Paste 10 g fast bordeaux GP base with 20 ml hot water and 5 g sodium nitrite (98 percent). After obtaining complete solution of the sodium nitrite, cool the paste to 15°C and add in small quantities with vigorous stirring to a mixture of 200 ml cold water and 17.5 ml hydrochloric acid 32°Tw. Allow to stand for 30 to 45 minutes with frequent stirring. Filter and neutralize with 8.5 g sodium acetate dissolved in 20 ml cold water. Then add the alkali binding agent 7.5 ml acetic acid 50 percent or 10 g aluminium sulphate crystals dissolved in 50 ml cold water. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 15°C.

17) Fast Garnet GBC Base

Paste 10 g fast garnet GBC base with 12 ml hydrochloric acid 32°Tw. Allow to stand for 5 minutes and add 20 ml hot water. Allow to stand for 5 minutes and then add to the paste 150 ml cold water and bring the temperature to about 5°C by adding ice. Then slowly stir in 3.2 g sodium nitrite (98 percent) dissolved in 15 ml cold water. After standing for 20 to 30 minutes with occasional stirring, filter and neutralize with 12.5 g sodium acetate dissolved in 20 ml cold water. Above solution contains sufficient alkali binding agent for dyeing normal strengths of shades. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 5°C.

18) Fast Violet B Base

Paste 10 g fast violet B base with 25 ml hot water and 3 g sodium nitrite (98 percent). When sodium nitrite is dissolved, cool the paste

to 15°C and then add slowly with vigorous stirring to a mixture of 150 ml cold water and 11.5 ml hydrochloric acid 32°Tw. Allow to stand for 20 to 30 minutes with occasional stirring. Then neutralize by either method (a) or (b).

- a) Dilute the solution with 100 ml cold water and neutralize by adding 2 g zinc oxide pasted with water. Filter the solution and add the alkali binding agent 10.5 g zinc sulphate. Make the final volume to 500 ml with cold water.
- b) Neutralize with 5.5 g sodium acetate dissolved in 25 ml cold water. Add to the dye-bath 10 g/l sodium acetate before use. Make the final volume to 500 ml with cold water.

Diazotizing temperature — 15°C.

19) Fast Blue B Base

Dissolve 10 g fast blue B base in 200 ml hot water containing 25 ml hydrochloric acid 32°Tw. Then add 50 ml cold water and cool solution with ice to 10 to 15°C. A solution of 6.5 g sodium nitrite in 25 ml cold water is then added slowly whilst stirring vigorously. Neutralize by either method (a) or (b).

- a) After standing for 10 to 15 minutes at 12°C with occasional stirring, the excess hydrochloric acid is neutralized by the addition of about 2.5 g sodium bicarbonate dissolved in 12.5 ml cold water. Add the required alkali binding agent 5 g sodium bicarbonate dissolved in 25 ml cold water. Make the final volume to 500 ml with cold water.
- b) Dissolve 12.5 g sodium acetate dissolved in 25 ml cold water. Before use an addition of 10 g/l sodium acetate is made to the dye-bath, make the final volume to 500 ml with cold water.

Diazotizing temperature — 12°C.

20) Fast Blue BB Base

Paste 10 g fast blue BB base with 100 ml cold water containing 12 ml hydrochloric acid 32°Tw. Cool to 18°C. Add 2.5 g sodium nitrite (98 percent) dissolved in 12 ml cold water to the paste whilst stirring vigorously. After 20 to 30 minutes, neutralize by either method (a) or (b).

- a) Add 2 g zinc oxide pasted with 5 ml cold water to the diazo solution. Then add 7.5 ml zinc sulphate dissolved in 50 ml cold water as alkali binding agent. Make the final volume to 500 ml with cold water.
- b) Dissolve 5 g sodium acetate in 12.0 ml cold water. Make to the dye-bath a further addition of 10 g/l sodium acetate before use. Make the final volume to 500 ml with cold water.

Diazotizing temperature — about 15 to 20°C.

DETERMINATION OF STRENGTH OF NAPHTHOLS (AZOIC COUPLING COMPONENTS) (GRAVIMETRIC AND VOLUMETRIC METHODS)

(Source : IS 4471 : 1981)

Naphthols are being marketed in different strengths. The methods prescribed are useful in assessing the strength of naphthols. Volumetric methods are not applicable to mixtures of naph-

thols. In case of dispute in the results by the volumetric and gravimetric method, the latter method shall be preferred.

1 SCOPE

1.1 The methods, gravimetric and volumetric, are intended for the determination of strength of naphthols. Both the methods are applicable to the naphthols given in Annex A.

1.2 Volumetric method prescribed in the standard is not applicable to the mixtures of naphthols.

1.2.1 In case of dispute, the gravimetric method shall be used for determination of strength of naphthols.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected draw small quantities of the naphthol by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 50 g.

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals shall be employed in tests, and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

4 GRAVIMETRIC METHOD

4.1 Apparatus

4.1.1 Beaker

of 1 000 ml capacity.

4.1.2 Sintered Glass Funnel

of appropriate size:

a) with pore size 15 to 40 microns (G-3 size), and

b) with pore size 5 to 15 microns (G-4 size).

4.2 Reagents

4.2.1 Methanol

pure.

4.2.2 Sodium Hydroxide Solution

30 percent (*m/m*) and 1 percent (*m/m*).

4.2.3 Hydrochloric Acid

1 : 1 (*v/v*).

4.3 Procedure

4.3.1 Weigh accurately about 1 g of naphthol sample and transfer to 1 000 ml dry beaker. Paste it with 10 ml methanol and add 1 ml of 30 percent sodium hydroxide and stir to dissolve. Add 400 ml warm (about 50°C) 1 percent sodium hydroxide solution and stir till the solution becomes clear. Filter through sintered glass funnel G-3, wash the residual material first with 3 to 5 ml methanol and finally with two 25-ml portions of warm 1 percent sodium hydroxide solution. Transfer the filtrate and washing quantitatively into a 1 000 ml beaker. Add, while stirring with a glass rod, hydrochloric acid drop by drop until the solution is acidic to Congo red paper. Digest the precipitated mass on water bath for about 2 to 3 hours. Allow the precipitate of naphthol to settle down. Filter through a sintered glass funnel G-4. Wash the precipitate with hot water till the filtrate is free from chlorides.

4.3.2 Dry the residue at 100 to 110°C to constant mass.

4.3.3 Calculate the percentage of naphthol by the following formula:

$$p = \frac{b}{a} \times 100$$

where

p = percentage by mass of the naphthol under test,

b = mass in g of the residue of the naphthol, and

a = mass in g of the naphthol taken for test.

4.4 Repeat the test twice as given in 4.3.1 and 4.3.2, and calculate the percentage of naphthol by the formula given in 4.3.3.

4.5 Calculate the average of the values obtained in 4.3.3 and 4.4.

5 VOLUMETRIC METHOD

5.1 Apparatus

5.1.1 Amber-Coloured Jacketed Burette

5.1.2 Amber-Coloured Graduated Flask of 500 ml capacity.

5.1.3 Mechanical Stirrer

5.1.4 Beakers

of 1 000 and 500 ml capacities.

5.1.5 Water Bath

5.2 Reagents

5.2.1 Amino Acetanilide Diazonium Chloride Solution (Diazo Solution)

0.05 N, prepared as follows:

Weigh accurately 3.77 g of *p*-amino acetanilide and transfer it to a 500 ml beaker. Add 55 ml of 1 N hydrochloric acid and 100 ml of water. Cool externally with ice to 5 to 10°C while stirring mechanically. Add 25.5 ml of 1 N sodium nitrite solution in about 5 minutes. Continue stirring for 10 minutes and test for excess of nitrite with starch iodide paper (see Note). Transfer the solution to a 500 ml amber-coloured graduated flask and make up the volume with ice cold water. Keep the diazo solution in ice bath.

NOTE — The starch iodide paper should give a positive faint test of excess of nitrous acid.

5.2.2 Diazotized Aniline Solution

0.05 N, prepared as follows:

Weigh accurately 2.325 g of redistilled aniline and diazotize it in the same manner as for *p*-amino acetanilide (see 5.2.1).

NOTE — This solution should be used for estimating the strength of CI azoic coupling component 18.

5.2.3 Dianisidine Diazonium Chloride

0.02 N, prepared as follows:

Diazotize 5 g of the dianisidine base with 10 ml of hydrochloric acid and 3 g of solid sodium nitrite. Neutralize the solution with a fresh sodium acetate solution and dilute it to 1 000 ml.

5.2.4 H-Acid (1-Naphthol-3.6, Disulphonic Acid, 8 Amino)

0.1 percent, prepared as follows:

Dissolve 0.1 g of H-acid in 100 ml of water containing 5 ml of 2 N sodium carbonate.

5.2.5 Hydrochloric Acid

1 N and 0.1 N.

5.2.6 Sodium Hydroxide Solution

1 N.

5.2.7 Dilute Sodium Hydroxide Solution

2.5 ml of 40 percent sodium hydroxide in 100 ml of water.

5.2.8 Dilute Acetic Acid

25 percent (*m/v*).

5.2.9 Pyridine

5.3 Procedure

5.3.1 Take about 1 g of naphthol from the composite sample. Weigh it accurately and dissolve it in 40 ml of dilute sodium hydroxide solution (see 5.2.7). Heat the solution, if necessary, to dissolve the naphthol completely. Cool and add dilute acetic acid till the pH of the solution is neutral (pH 6 to 7). Dissolve the precipitated naphthol in 80 ml of pyridine with constant stirring. Add more pyridine, if necessary. Cool it to 25°C and keep under mechanical agitation.

5.3.2 Titrate the solution obtained in 5.3.1 against diazotized *p*-amino acetanilide solution (diazo solution) (see 5.2.1) from an amber-coloured burette equipped with a water jacket through which water at about 5°C is circulated. Spot a drop of the reaction mixture on a filter paper. Spot a drop each of dianisidine diazonium chloride solution and H-acid solution near the spot of the reaction mixture. Take the end point as the mean of the two readings, one of which gives a negative test for naphthol with dianisidine diazonium chloride and the other gives a positive test for *p*-amino acetanilide diazonium chloride solution with H-acid. Note the volume of the *p*-amino acetanilide diazonium chloride solution required.

NOTES

1 The *p*-amino acetanilide diazonium chloride solution should be added initially in portions of 1 to 2 ml, testing the reaction mixture after each addition for excess of diazonium chloride or naphthol.

2 If the bleed of reaction mixture, when spotted on filter paper is not clear, a small amount of solid common salt should be kept on filter paper and the drops of reaction mixture should be dropped on it.

3 If naphthol is in excess, a colouration is seen at the junction of the spots of dianisidine diazonium chloride and the reaction mixture. If diazo solution is in excess, a pink colour is seen at the junction of the spot of H-acid and the reaction mixture. The addition of *p*-amino acetanilide diazonium chloride solution should be in instalments of 0.2 to 0.3 ml near the end point.

4 In case of azoic coupling component 18, diazotized aniline solution (see 5.3.2) should be used.

5 CI azoic coupling component 5 is dissolved in pyridine at 40°C. No alkali is added. The solution is cooled to 5 to 10°C and titrated against diazotized *p*-amino acetanilide.

5.3.3 Calculate the strength of naphthol by the following formula:

$$P = \frac{0.005 \times M \times A \times 100}{B}$$

NOTE — In case of azoic coupling component 5, the coupling takes place at two positions. Therefore, the amount of diazo solution consumed is double and hence the strength should be calculated as:

$$P = \frac{0.005 \times M \times A}{2} \times \frac{100}{B}$$

where

P = percentage by mass of naphthol,

M = molecular mass of naphthol under test (see Annex A),

A = volume in millilitres of *p*-acetanilide diazonium chloride solution (or diazotized aniline solution) required for titration, and

B = mass in g of the naphthol taken for the test (see 4.3.1).

5.4 Repeat the procedure prescribed in 5.3.1 and 5.3.2 twice and calculate the strength of naphthol in each case.

5.5 Calculate the average of the values obtained in 5.3.3 and 5.4.

6 REPORT

6.1 Report the value obtained in 4.5 or 5.5 as the strength of naphthol under test in percent.

6.2 Report also the method used, whether gravimetric or volumetric.

ANNEX A

GENERAL INFORMATION ABOUT ANALYSIS OF NAPHTHOLS

SL No.	*CI DESIGNATION OF NAPHTHOL	COMMON COMMERCIAL NAME OF NAPHTHOL	COLOUR INDEX No.	MOLECULAR MASS	STRUCTURAL FORMULA AND CHEMICAL NAME
(1)	(2)	(3)	(4)	(5)	(6)
i)	Azoic coupling component 2	Naphthol AS	CI 37505	263.0	
ii)	Azoic coupling component 4	Naphthol AS-BO	CI 37560	313.0	
iii)	Azoic coupling component 5	Naphthol AS-G	CI 37610	380.0	
iv)	Azoic coupling component 8	Naphthol AS-TR	CI 37525	311.5	
v)	Azoic coupling component 17	Naphthol AS-BS	CI 37515	308.0	
vi)	Azoic coupling component 18	Naphthol AS-D	CI 37520	277.0	

*Colour Index (1956). Ed 3. Society of Dyers and Colourists, UK; and American Association of Textile Chemists and Colorists, USA.

EVALUATION OF STRENGTH AND SHADE OF NAPHTHOL

(Source : IS 4946 : 1968)

Unlike other types of dyes, the exhaustion of naphthols from the naphtholating bath is comparatively less. In order to reduce the chances of error in evaluating the strength of naphthols, two

additional exhaust dyeings are recommended instead of a single dyeing and the average of the three dyeings is determined.

1 SCOPE

1.1 This method is intended for the determination of strength of naphthols listed in Table 1 by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of naphthol by suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 50 g.

3 STANDARD NAPHTHOL

3.1 The standard sample of naphthol against which the strength and shade of naphthol under

test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH

5.1 Out of the sets of conditions of test laid down in Table 1, choose the set of conditions applicable to the naphthol under test.

TABLE 1 GENERAL INFORMATION ABOUT DYEING OF NAPHTHOLS

Sl. No.	COLOUR INDEX DESIGNATION (COMMON COMMERCIAL NAME)	COLOUR INDEX No.	DEPTH OF SHADE	FOR NAPHTHOL DISSOLUTION, NAPHTHOL OF			FINAL CONCENTRATIONS IN NAPHTHOLATING BATH			COLOUR INDEX DESIGNATION OF DIAZO COMPONENT (COMMON COMMERCIAL NAME)		CONCENTRATION OF BASE IN DEVELOPING BATH	pH OF DEVELOPING BATH	PERCENT NITRIC VALUE, ABOUT
				TRO	NaOH	ml/g	TRO	NaOH	NaCl	Diazo Component	Molecular Mass			
(1)	(2)	(3)	(4) g/l	(5) ml/g	(6) ml/g	(7) ml/l	(8) ml/l	(9) ml/l		(10)	(11)	(12)	(13)	(14)
i)	Azoic coupling component 2 (naphthol AS)	37505	2.0	3	1.5	8	6	20		Azoic diazo component 13 (Fast Scarlet RC)	204.5	1.5	4.5	80
ii)	Azoic coupling component 17 (naphthol AS-BS)	37515	1.0	3	1.5	8	6	20		Azoic diazo component 13 (Fast Scarlet R)	204.5	1.2	4.5	80
iii)	Azoic coupling component 4 (naphthol AS-BO)	37560	1.0	3	2	8	6	20		Azoic diazo component 5 (Fast Red B)	168	1.2	4.5	95
iv)	Azoic coupling component 18 (naphthol AS-D)	37520	2.0	3	5.1	8	6	20		Azoic diazo component 13 (Fast Scarlet R)	204.5	1.2	4.5	80
v)	Azoic coupling component 8 (naphthol AS-TR)	37525	1.0	3	2	8	6	20		Azoic diazo component 11 (Fast Red TR)	178	1.2	4.5	80
vi)	Azoic coupling component 5 (naphthol AS-G)	37610	2.0	3	2.5	8	6	15		Azoic diazo component 44 (Fast Yellow GC)	164	1.2	4.5	80
vii)	Azoic coupling component 13 (naphthol AS-SG)	37595	2.0	3	1.0	8	7	—		Azoic diazo component 5 (Fast Red B)	168	1.2	4.5	95
viii)	Azoic coupling component 15 (naphthol AS-LB)	37600	1.0	3	0.5	8	6	20		Azoic diazo component 2 (Fast orange GC)	164	0.6	4.5	80

NOTE — Quantities mentioned under column (11) are based on the nitrite value of column (13).

5.2 Prepare dyeing of the recommended depth of shade of naphthol under test by following the procedure given in Annex A. Prepare simultaneously additional dyeing of the naphthol under test with the depth of shade 90 percent of the first.

5.3 Simultaneously, prepare dyeings of different depths of shade of standard naphthol by following the procedure given in Annex A.

NOTE — The depths of shade of dyeings of standard naphthols should be so arranged that the difference between two consecutive dyeings is the same and the dyeings are well distributed on either side of the recommended depth of shade (see Table 1), taking into consideration the expected strength of the sample.

5.4 Carry out second set of dyeings with fresh hanks in the exhaust liquor of the above dyeings by following the procedure given in A-4.5.

5.4.1 Carry out third set of dyeings with fresh hanks in the exhaust liquor obtained in 5.4 by following the procedure given in A-4.5.1.

5.5 Compare the dyeings obtained in 4.2 and 5.3 (see Note). Select dyeing of the standard naphthol which exactly matches with one of the dyeings of the naphthol under test. Note the depths of the shade of the dyeings which match exactly.

NOTE — The dyeing should be spread out properly and compared in the north light.

6 CALCULATION

6.1 Calculate the strength of naphthol under test

by comparing the first set of dyeings by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of naphthol in percent,

A = depth of shade in grams per litre of the dyeing of standard naphthol, and

B = depth of shade in grams per litre of the dyeing of naphthol under test comparing with that of standard.

6.1.1 Similarly calculate the strength of naphthol under test from the other two sets of exhaust dyeings.

6.2 Calculate the average of the three values obtained in 6.1 and 6.1.1.

7 REPORT

7.1 The report shall include the following information:

- Strength of naphthol in percent, and
- Shade of naphthol in comparison with the shade of the standard.

NOTE — The remarks regarding shade would hold good only for the specific combination of naphthol and base mentioned in Table 1.

ANNEX A DYEING PROCEDURE

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain, glass or stainless steel beakers or dye vessels of 250 to 300 ml capacity.

NOTE — For diazotization, only porcelain or glass beakers should be used.

A-1.2 Watch Glass

A-1.3 Pipettes, Graduated

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of dye baths.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Hydroxide Solution

430 g/l or 70°Tw.

A-2.3 Turkey Red Oil

50 percent solution (v/v).

A-2.4 Soap-Neutral

A-2.5 Soda Ash

A-2.6 Sodium Chloride Solution

10 percent (w/v).

A-2.7 Hydrochloric Acid

32°Tw.

A-2.8 Sodium Nitrite

solid (98 percent).

A-2.9 Sodium Acetate

solid (98 percent).

A-2.10 Acetic Acid

50 percent (w/v).

A-3 PREPARATION OF HANKS**A-3.1 Test Hanks**

Hanks shall be of scoured, bleached, unmercerized cotton yarn having no finishing chemical or blueing agent. Each hank shall weigh 10 ± 0.1 g.

NOTE — Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

- a) Count — 10 tex $\times 2$ (or 60s/2)
- b) Twist — 750/m
- c) Cuprammonium fluidity — not more than 5 rhes.

A-3.2 Preparation of Test Hanks

The hanks shall be treated in boiling water for 10 minutes and squeezed evenly to contain approximately its own mass of water, cooled and entered in the naphtholating bath.

A-4 PROCEDURE**A-4.1 Preparation of Naphthol Solution**

A-4.1.1 Weigh accurately 5.0 g of naphthol under test. Paste it thoroughly with Turkey red oil. Add requisite quantity of sodium hydroxide solution (see Table 1). Add 100 ml of water near boil and boil the whole solution till it becomes clear. Dilute the solution to 500 ml with water containing 1 ml/l each of sodium hydroxide and Turkey red oil solutions.

NOTES

1 CI azoic coupling component 13 (naphthol AS-SG) is dissolved by following the procedure given below:

'Paste 5.0 g of naphthol with equal amount of Turkey red oil. Add to it 5 ml of hot water and 5 ml of sodium hydroxide solution. Heat the paste for 15 minutes and then add 30 ml of boiling water. Boil the solution and add 40 ml of boiling water. Boil the solution again. Dilute the clear solution to 500 ml with water containing 1 ml/l each of Turkey red oil and sodium hydroxide solution.

2 In case of CI azoic coupling component 8 (naphthol AS-TR), 200 ml of water is used for naphthol dissolution. Also caustic soda is added to the boiling suspension of naphthol in water.

A-4.1.2 Similarly prepare solution of the standard naphthol by following the procedure given in **A-4.1.1** but taking standard naphthol instead of naphthol under test.

A-4.2 For naphtholation, developing and soaping, the liquor ratio shall be 1 : 20.

A-4.3 Naphtholation (for 10 ± 0.1 g Hank)

A-4.3.1 Take in each dye vessel required quantity of water (see Note). Add the necessary amount of Turkey red oil and sodium hydroxide (subtracting the amount of sodium hydroxide and

Turkey red oil coming with naphthol solution). Add the necessary amount of naphthol solution under test in one dye vessel so that the final concentration of the naphthol under test shall be as given in Table 1. Add the necessary amount of sodium chloride solution with stirring. Similarly, add in other dye vessel solution of naphthol under test so as to give 90 percent depth of shade of first dyeing. Enter the wetted and squeezed hanks into the dye vessels and naphtholate for 30 minutes at room temperature. Turn the hanks so as to obtain level naphtholation. After naphtholation, take out the hanks and squeeze them evenly. Preserve the naphtholating bath for carrying out exhaust dyeing (see **A-4.5**).

NOTE — The volume of water should be calculated taking into consideration the volume of sodium hydroxide, Turkey red oil, sodium chloride and naphthol solution taken for naphtholation.

A-4.3.2 Similarly, prepare naphtholated hanks with the solution of standards naphthol in different depths (see also Note under 5.3).

NOTE — The naphtholation with the solutions of standard naphthol and the naphthol under test should be done simultaneously.

A-4.4 Developing

Prepare a developing bath in water containing suitable diazotized fast base (see Table 1 and Notes below). Develop all the naphtholated hanks obtained in **A-4.3.1** and **A-4.3.2** in the same developing bath at room temperature for 30 minutes with frequent stirring. Wash the dyed hanks thoroughly with water.

NOTES

1 The details of the diazotization are given in Annex B.

2 The pH of the developing bath can be adjusted to the desired value by addition of acetic acid or sodium bicarbonate solution, as the case may be.

3 In case of CI diazo component 11 (fast red TR base), sodium acetate crystals 10 g/l are added to the developing bath before use.

4 For developing bath of CI azoic coupling component 5 (naphthols AS-G), an addition of 4 ml/l of acetic acid (50 percent) is made over and above the normal quantity of alkali binding agent.

A-4.5 Make up the volumes of each naphtholating bath obtained in **A-4.3.1** and **A-4.3.2** to 200 ml with water containing 1 ml/l each of sodium hydroxide solution and Turkey red oil. Carry out the naphtholation of the fresh hanks and develop the naphtholated hanks in the fresh baths.

A-4.5.1 Repeat naphtholation and developing of fresh hanks according to the method given in **A-4.5**.

A-4.6 Soaping

Soap all the three sets of dyeings (see **A-4.4**, **A-4.5** and **A-4.5.1**) together and boil for 30 minutes in a bath containing 3 g/l of soap and 2 g/l of soda ash. Wash thoroughly and dry.

ANNEX B

DIAZOTIZATION RECIPES FOR FAST BASES

B-1 CI AZOIC DIAZO COMPONENT 13 (FAST SCARLET RC BASE)

B-1.1 Paste 10 g fast scarlet RC base with a mixture of 10 ml hydrochloric acid and 10 ml cold water. Add to this 200 ml cold water and stir to dissolve. Cool the solution to 10 to 15°C. Add to this quickly, while stirring, 4 g sodium nitrite dissolved in 25 ml cold water. After 20 to 30 minutes, neutralize the solution with 7.5 g sodium acetate dissolved in 25 ml cold water. Add finally the alkali binding agent 5 ml acetic acid (50 percent).

Diazotizing temperature: 10 to 15°C.

B-2 CI AZOIC DIAZO COMPONENT 5 (FAST RED B BASE)

B-2.1 Paste 10 g fast red B base with 15 ml hot water and 5 g sodium nitrite. After the nitrite is completely dissolved, cool the paste to 15°C and add in small quantities with vigorous stirring to a mixture of 200 ml cold water and 17.5 ml hydrochloric acid. Allow to stand for 30 minutes with frequent stirring. Filter the solution and neutralize with 8.5 g sodium acetate dissolved in about 25 ml cold water. Finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing temperature: 15°C.

B-3 CI AZOIC DIAZO COMPONENT 11 (FAST RED TR BASE)

B-3.1 Dissolve 10 g fast red TR base in a mixture of 200 ml cold water and 10 ml hydrochloric acid. Cool the solution to 10°C while vigorously

stirring and fairly slowly add 4 g sodium nitrite dissolved in 25 ml cold water. After 20 to 30 minutes, neutralize with 7.5 g sodium acetate dissolved in about 25 ml cold water. Add the alkali binding agent 1.5 ml acetic acid (50 percent). Add further 10 g sodium acetate to the dye bath before use.

Diazotizing temperature: 10°C.

B-4 CI AZOIC DIAZO COMPONENT 44 (FAST YELLOW GC BASE)

B-4.1 Paste 10 g fast yellow GC base with 50 ml hot water and 12 ml hydrochloric acid. Add 150 ml cold water and cool to 10°C. To the solution, add 5 g sodium nitrite dissolved in about 25 ml cold water with stirring. After 15 to 20 minutes, neutralize with about 10 g sodium acetate dissolved in 25 ml cold water. Then finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing temperature: 5 to 10°C.

B-5 CI AZOIC DIAZO COMPONENT 2 (FAST ORANGE GC BASE)

B-5.1 Paste 10 g fast orange GC base with 50 ml hot water and 12 ml hydrochloric acid. Add 150 ml cold water to dissolve the paste. Cool to 10°C. Add with stirring 5 g sodium nitrite dissolved in about 25 ml cold water. After 15 to 20 minutes, neutralize with 10 g sodium acetate dissolved in about 25 ml cold water. Finally add the alkali binding agent 7.5 ml acetic acid (50 percent).

Diazotizing temperature: 10°C.

EVALUATION OF STRENGTH AND SHADE OF ACID DYES BY DYEING TEST

(Source : IS 7843 : 1975)

This method is a general one and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. In order to compare the exhaust property of the dyestuff under test and the standard dyestuff, it is recommended to carry out the exhaust

dyeings. It is generally better to use medium shade (about 1 percent) as they show any variations in strength more clearly than dark shades. Wherever necessary, it is advisable to carry out evaluation in deep shades also as recommended by the manufacturers (for example, blacks, navy blues, etc.).

1 SCOPE

1.1 This method is intended for the evaluation of strength and shade of acid dyes (level-dyeing acid dyes, acid-milling dyes, neutral-dyeing acid dyes, 1 : 1 and 2 : 1 metal complex acid dyes) by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuffs under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Preliminary Test to Determine Strength

Before dyeing the hanks, carry out a preliminary test to determine the approximate strength of the dyestuff under test as given in 5.1.1.

5.1.1 Take separately 10 ml of 0.1 percent solution of the dyestuff under test and standard dyestuff (see A-4.1). Put a spot or streak or pour each of the solutions of the dyestuff under test and the standard dyestuff on filter paper. Visually examine the spots. If they are not equal in strength, dilute the stronger solution with water to such an extent as to get approxi-

mate equal strength of dyestuff spotted on filter paper. Calculate the approximate strength of the dyestuff under test by the following formula:

Approximate strength (S_s)

$$\text{of dyestuff under test} = \frac{V_t \times 100}{V_s}$$

where

V_t = final volume in millilitres of the solution of dyestuff under test, and

V_s = final volume in millilitres of solution of the standard dyestuff.

5.2 Prepare solutions of standard dyestuff and dyestuff under test as given in A-4.1.

5.3 Prepare dyeings of 0.95, 1.00 and 1.05 percent depths of shade of the standard dyestuff as given in A-4.2.1.

5.4 Similarly, prepare dyeings of 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade of dyestuff under test as given in A-4.2.2.

5.5 Carry out the dyeings of the fresh hanks in the exhaust liquors of the above dyeings by following the procedure given in A-4.3.

5.6 Assessment

Compare the dyeings of dyestuff under test (see 5.4) and standard dyestuff (see 5.3) in North skylight (see Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentages of the dyeings which match exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of the backing on the appearance. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeing should be repeated.

5.7 For Deep Shades

Wherever necessary, evaluation shall also be carried out in deep shades on similar lines as for

one percent shade, using dyeing assistants in accordance with the manufacturer's recommendations.

B = percentage shade of the dyestuff under test matching with A .

6 CALCULATIONS

6.1 Calculate to strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percentage,

A = percentage shade of the standard dyestuff, and

7 REPORT

7.1 The report shall include the following information:

- Strength of dyestuff in percent (see 6.1),
- Shade of the dyestuff in comparison with that of the standard dyestuff, and
- Shade of the exhaust dyeings in comparison with that of the exhaust dyeing of the standard dyestuff.

ANNEX A

GENERAL METHOD FOR DYEING OF ACID DYES ON WOOL

A-1 APPARATUS

A-1.1 Dye Vessel

porcelain or stainless steel dye vessels.

A-1.2 Graduated Pipettes

capable of measuring correct up to 0.1 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing water, having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Sulphate (Calcined) Solution

10 percent (m/v).

NOTE — Quality of sodium sulphate is extremely important. Commercial brands may be acidic or alkaline. Therefore, they shall be neutralized.

A-2.3 Acetic Acid Solution

10 percent (m/v).

A-2.4 Formic Acid Solution

10 percent (m/v).

A-2.5 Sulphuric Acid Solution

10 percent (m/v).

A-2.6 Ammonium Sulphate

10 percent (m/v).

A-2.7 Ammonium Acetate

10 percent (m/v).

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, 100 percent pure wool knitting yarn having no finishing chemical, blueing agent or fluorescent brightening agent shall be used. Each hank shall weigh 5.0 ± 0.1 g (see Note).

NOTE — If the mass of the hank is not 5.0 ± 0.1 g, then it shall be weighed accurately and the amount of dyestuff and the chemicals to be taken shall be calculated accordingly.

A-3.2 Preparation of Test Hanks

Treat the required number of hanks in warm water (not higher than 60°C) for 10 minutes, squeeze evenly to contain approximately its own mass of water, cool and enter into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of Dyestuff Solutions

A-4.1.1 Weigh accurately 1.00 g of standard dyestuff. Paste it thoroughly with cold water. Add hot water and dissolve the dyestuff. If necessary, heat the solution till it becomes clear. Dilute the solution to 1 000 ml with cold water.

A-4.1.2 Similarly weigh accurately $\frac{1.00 \times 100}{S_a}$ (see 5.1.1) of dyestuff under test and prepare solution following the procedure given in A-4.1.1.

A-4.2 Dyeing (for 5.0 ± 0.1 g Hank)

A-4.2.1 Pipette out separately 47.5, 50.0 and 52.5 ml of standard dyestuff solution in the dye

vessels so as to give 0.95, 1.00 and 1.05 percent depths of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml (liquor ratio 1 : 60). Add requisite quantity of sodium sulphate (calcined) solution, wherever necessary, and half the quantities of acid solution or acid salt solution depending on the nature of the dyestuff (see Note 1). Stir the dye liquor and raise the temperature to 50°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this temperature. Remove the hank from the dye bath, add second lot of requisite quantity of acid solution or acid salt solution and stir well. Enter the hanks into the dye bath and turn the hanks frequently. Slowly raise the temperature of water bath to boil within 20 minutes. Continue the dyeing at boil for 45 to 60 minutes (see Note 2). Remove the hank. If necessary, add weak or strong acid solution (see Note 1). After addition, stir the bath and enter the hanks. Continue the dyeing further for 15 minutes. At the end of dyeing, squeeze the dyed hank returning the squeezed out solutions to the respective dye baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at a temperature not exceeding 70°C.

NOTES

1 Addition of strong acid, weak acid or acid salt individually or in combination should be made with or without sodium sulphate (calcined) according to the recommendations given below or the recommendations of the dyestuff manufacturers or by mutual agreement between the buyers and the sellers. Recommended quantities of dyeing assistants for medium shades (about 1 percent shade) are given below:

- a) *Level dyeing acid dyes* — 5 ml sodium sulphate (calcined) solution and 1.75 ml sulphuric acid solution or formic acid solution.
- b) *Acid milling dyes and neutral dyeing dyes*

- 1) 5 ml sodium sulphate (calcined) solution and 0.75 ml acetic acid solution.

If necessary, for exhaustion, add additional amount of 0.75 ml sulphuric acid solution or formic acid solution. Continue the dyeing further according to the recommended procedure.

OR

- 2) 5 ml sodium sulphate (calcined) solution and 3 ml ammonium acetate solution or ammonium sulphate solution.

For exhaustion, add additional amount of 1 ml acetic acid solution.

- c) *1 : 1 metal complex acid dyes* — 5 ml sodium sulphate (calcined) solution and 4.0 ml sulphuric acid solution.
- d) *2 : 1 metal complex acid dyes* — 1.0 ml ammonium sulphate solution.

- 2 During dyeing, water should be added to make up the volume lost by evaporation.

A-4.2.2 Similarly, pipette out separately 45.0, 47.5, 50.0, 52.5 and 55.0 ml of solution of dyestuff under test in the dye vessels so as to give 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade (in terms of equivalent standard strength). Dye the hanks by following the method given in A-4.2.1 (see Note).

NOTE — The dyeings with the solutions of the dyestuffs under test and of the standard dyestuff should be done simultaneously in the same water bath.

A-4.3 Exhaust Dyeing

Make up the volumes of the exhaust liquor (see A-4.2.1 and A-4.2.2) to the original volume (300 ml). Enter the fresh wetted hanks in the exhausted dye baths and continue the dyeing for 30 minutes at boil. Squeeze the hanks, rinse in warm water (50 to 60°C) and dry.

EVALUATION OF STRENGTH AND SHADE OF CHROME DYES BY DYEING TEST

(Source : IS 7844 : 1975)

This method is a general method and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. In order to compare the exhaust property of the dyestuff under test and the standard dyestuff, it is recommended to carry out the exhaust dyeings. It is generally better to use medium shades (above 1 percent) as they show any variations in strength more clearly than dark shades. Wherever necessary, it is advisable to carry out evaluation in deep shades also as recommended by the manufacturers (for example, blacks, navy blues, etc).

Chrome dyes are generally dyed by the following three methods:

- a) *After Chrome Process (or Top Chrome Process)* — In this process, dyeing is first carried out on the wool and subsequently chroming is done in the same bath.
- b) *One-Bath Chrome Process (Chromal Process or Chromate Process or Metachrome Process)* — In this process, dyeing and chroming take place simultaneously.
- c) *Bottom Chrome Process (Chrome Mordant Process)* — In this process, a chrome mordant is first fixed on the wool and subsequently dyed in a fresh bath.

This method is based on 'After Chrome Process' as it is applicable to wide range of dyes. However, an outline of other two methods of dyeing is given in Annex B.

1 SCOPE

1.1 This method is intended for the evaluation of strength and shade of chrome dyes by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFFS

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test result.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Preliminary Test to Determine Strength

Before dyeing the hanks, carry out a preliminary test to determine the approximate strength of the dyestuff under test as given in 5.1.1.

5.1.1 Take separately 10 ml of 0.1 percent solution of the dyestuff under test and standard dyestuff (see A-4.1). Put a spot or streak or pour each of the solution of the dyestuff under test and the standard dyestuff on filter paper. Visually examine the spots. If they are not equal in strength, dilute the stronger solution with water to such an extent as to get approximate equal strength of dyestuff spotted on filter paper. Calculate the approximate strength of the dyestuff under test by the following formula:

Approximate strength (S_a) of dyestuff under

$$\text{test} = \frac{V_t}{V_s} \times 100$$

where

V_t = final volume in millilitres of the solution of dyestuff under test, and

V_s = final volume in millilitres of solution of the standard dyestuff.

5.2 Prepare solutions of standard dyestuff and dyestuff under test as given in A-4.1.

5.3 Prepare dyeings of 0.95, 1.00 and 1.05 percent depths of shade of the standard dyestuff as given in A-4.2.1.

5.4 Similarly, prepare dyeings of 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade of the dyestuff under test as given in A-4.2.2.

5.5 Carry out the dyeings of the fresh hanks in the exhaust liquors of the above dyeings by following the procedure given in A-4.3.

5.6 Assessment

Compare the dyeings of dyestuff under test

(see 5.4) and standard dyestuff (see 5.3) in North sky-light (see Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentages of the dyeings which match exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of the backing on the appearance. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variation between the two consecutive dyeings is not constant, the dyeings should be repeated.

5.7 For Deep Shades

Wherever necessary, evaluation shall also be carried out in deep shades on similar lines as for 1 percent shade using dyeing assistants in accordance with the manufacturers' recommendations.

6 CALCULATION

6.1 Calculate the strength of the dyestuff under

test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percentage,

A = percentage shade of the standard dyestuff, and

B = percentage shade of the dyestuff under test matching with A .

7 REPORT

7.1 The report shall include the following information:

- a) Strength of dyestuff in percent,
- b) Shade of the dyestuff in comparison with that of the standard dyestuff, and
- c) Shade of the exhaust dyeings in comparison with that of the exhaust dyeing of the standard dyestuff (A-4.3).

ANNEX A

GENERAL METHOD FOR DYEING OF CHROME DYES ON WOOL

A-1 APPARATUS

A-1.1 Dye Vessel

porcelain or stainless steel dye vessels.

A-1.2 Graduated Pipettes

capable of measuring correct up to 0.1 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Sulphate (Calcined) Solution

10 percent (m/v).

NOTE — Quality of sodium sulphate is extremely important. Commercial brands may be acidic or alkaline. Therefore, they shall be neutralized.

A-2.3 Acetic Acid Solution

10 percent (m/v).

A-2.4 Formic Acid Solution

10 percent (m/v).

A-2.5 Potassium or Sodium Dichromate Solution

5 percent (m/v).

A-2.6 Ammonium Sulphate Solution

10 percent (m/v).

A-2.7 Ammonium Acetate Solution

10 percent (m/v).

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, 100 percent pure wool knitting yarn having no finishing chemical, blueing agent or fluorescent brightening agent shall be used. Each hank shall weigh 5.0 ± 0.1 g (see Note).

NOTE — If the mass of the hank is not 5.0 ± 0.1 g, then it shall be weighed accurately and the amount of dyestuff and the chemicals to be taken shall be calculated accordingly.

A-3.2 Preparation of Test Hanks

Treat the required number of hanks in warm water (not higher than 60°C) for 10 minutes,

squeeze evenly to contain approximately its own mass of water, cool and enter into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of Dyestuff Solutions

A-4.1.1 Weigh accurately 1.00 g of standard dyestuff. Paste it thoroughly with cold water. Add hot water and dissolve the dyestuff. If necessary, heat the solution till it becomes clear. Dilute the solution to 1 000 ml with cold water.

A-4.1.2 Similarly weigh accurately $\frac{1.00}{S_a} \times 100$ g (see 5.1.1) of the dyestuff under test and prepare solution by following the procedure given in A-4.1.1.

A-4.2 Dyeing

for 5 ± 0.1 g hank.

A-4.2.1 Pipette out separately 47.5, 50.0 and 52.5 ml of standard dyestuff solution in the dye vessels so as to give 0.95, 1.00 and 1.05 percent depth of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml (liquor ratio of 1 : 60). Add 2.5 ml of sodium sulphate (calcined) solution and 0.5 ml acetic acid solution. Stir the dye liquor and raise the temperature to 60°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this temperature. Remove the hank from the dye bath, add further 0.5 ml of acetic acid solution and stir well. Enter the hanks into the dye bath and turn the hanks frequently. Slowly

raise the temperature of water bath to boil within 20 minutes. Continue the dyeing at boil for 30 minutes (see Note). Remove the hanks from the dye bath and add 0.5 ml formic acid solution, enter the hanks, and boil further for 30 minutes. Then cool down the water bath to 85°C and add 0.5 ml of sodium or potassium dichromate solution. Raise the temperature to boil and continue dyeing for further 45 minutes. At the end of dyeing, squeeze the dyed hank, returning the squeezed out solutions to the respective dye baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at a temperature not exceeding 70°C.

NOTE — During dyeing, water should be added to make up the volume lost by evaporation.

A-4.2.2 Similarly, pipette out separately 45.0, 47.5, 50.0, 52.5 and 55.0 ml of solution of dyestuff under test in the dye vessels so as to give 0.90, 0.95, 1.00, 1.05, and 1.10 percent depths of shades (in terms of equivalent standard strength). Dye the hanks by following the method given in A-4.2.1 (see Note).

NOTE — The dyeings with the solutions of the dyestuffs under test and of the standard dyestuff should be done simultaneously in the same water bath.

A-4.3 Exhaust Dyeing

Make up the volumes of the exhaust liquors (see A-4.2.1 and A-4.2.2) to the original volume (300 ml). Enter the fresh wetted hanks in the exhausted dye baths and continue the dyeing for 30 minutes at boil. Squeeze the hanks, rinse in warm water (50 to 60°C) and dry.

ANNEX B

B-1 ONE-BATH CHROME PROCESS

B-1.1 Pipette out separately the required amount of standard dyestuff solution (see A-4.2.1) to get different depths of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml. Add requisite quantities of sodium sulphate solution, 2 ml sodium or potassium dichromate and 3 ml ammonium sulphate or ammonium acetate solution. Stir the dye liquor and raise the temperature to 60°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this temperature and slowly raise the temperature of water bath to boil within 30 minutes. Continue the dyeing at boil for 60 minutes. At the end of dyeing, squeeze the dyed hank returning the squeezed out solution to the respective

dye baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at the temperature not exceeding 70°C.

B-1.2 Similarly, pipette out separately required amount of solution of the dyestuff under test in different dye vessels (see A-4.2.2) to get different depths of shade and carry out dyeing as in B-1.1.

B-2 BOTTOM CHROME PROCESS

B-2.1 Chroming

Prepare chroming bath using 1.5 ml sodium or potassium dichromate and 0.70 ml formic acid solution, and finally make the volume to 300 ml. Stir the liquor of the bath and raise the temperature to 50°C. Enter the wetted hanks and turn them frequently so as to obtain uniform

chroming. Slowly raise the temperature to boil within 20 minutes and continue the chroming at boil for further 60 minutes. At the end of chroming period, squeeze the hanks and rinse the same in cold water. Carry out the dyeing immediately.

B-2.2 Dyeing

Pipette out separately the required amount of standard dyestuff solution in the dye vessels to get different depths of shade (*see* A-4.1.1). Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the volume to 300 ml. Add 0.6 ml acetic acid solution, stir

the dye liquor and raise the temperature to 40°C. Enter the chromed hanks and turn them frequently so as to obtain level dyeing. Slowly raise the temperature of water bath to boil within 30 minutes. Continue the dyeing at boil for 90 minutes. At the end of dyeing, squeeze the dyed hanks and rinse the hanks with warm water (50 to 60°C). Dry the hanks in oven or in drier at the temperature not exceeding 70°C.

B-2.3 Similarly, pipette out separately required amount of solution of the dyestuff under test (*see* A-4.1.2) in different dye vessels to get different depths of shade and carry out chroming and dyeing as in B-2.1 and B-2.2.

DETERMINATION OF STRENGTH OF HOMOGENEOUS VAT DYESTUFFS

(Source : IS 4394 : 1988)

Vat dyes are marketed in a large number of varieties in powder or paste form with different strengths. They are mainly used for colouring cellulosic fibres and most favoured for applications wherever the fastness demands are of prime importance.

The evaluation of strength of a dye is useful for the manufacturer to standardize the product batches and for the user for matching of shades,

evaluating cost-benefit ratio and adjustment of dyeing recipes to minimize dyeing irregularities.

The method prescribed in this standard is a general method and, wherever special instructions are given by manufacturers, these should be followed while carrying out the dyeing. In order to compare the strength of the dyestuff under test and the standard dyestuff, dyeings of standard depths are recommended as given in Table 1.

1 SCOPE

1.1 This method is intended for evaluating the strength of homogeneous vat dyestuffs by exhaust dyeing.

1.2 The method is not applicable to mixtures of dyestuffs. The qualitative tests are given in Annex A to distinguish a homogeneous dyestuff (with or without corrective shade components) from a mixture of dyestuff.

2 TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Depth of Shade

The amount of dyestuff in g in the dye bath per 100 g of the fibre, expressed as a percentage.

2.2 Strength of Dyestuff

The percentage ratio of the strength of dyestuff under test to that of the standard dyestuff, the strength of which is assumed to be 100 percent.

2.3 Liquor Ratio

The ratio of the mass of the material to be dyed to the mass of the liquor (dye solution or dye bath in which the material is to be dyed).

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 DETERMINATION OF STRENGTH OF DYESTUFF

5.1 Carry out the qualitative tests given in Annex A to determine whether the dyestuff under test is a mixture of dyestuffs or a single homogeneous dyestuff (with or without corrective shade components added).

NOTE — Two qualitative tests are prescribed in Annex A. If necessary, both the tests should be carried out to find out the components in the mixture of dyestuffs. If corrective shade components are added to the dyestuff, they are also detected by these tests.

5.2 Out of the sets of conditions of test laid down in Table 1, choose the set of conditions applicable to the dyestuff under test.

5.3 Prepare dyeings for both the recommended percentages of standard sample of dyestuff (see 3.1) by following the procedure given in Annex B; prepare simultaneously additional dyeings of the standard sample, with the percentage variations of dyeing strength by 5 percent on either side of each recommended percentage.

5.4 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex B.

5.5 Compare the dyeings obtained in 5.4 with the dyeings obtained in 5.3. Select a dyeing of the dyestuff under test which visually appears to be equal in strength to the dyeing of the standard dyestuff for each recommended percentage and note the percentage of these dyeings.

NOTES

1 The dyeings should be compared in North skylight. The evaluation of the hanks dyed with the standard dyestuff and the dyestuff under test should be carried out by at least three observers for consistency in the results. This may also be done instrumentally, if facilities exist.

2 Before comparing the dyeings, they should be spread out properly. The consistency in different

dyeings of standard dyestuff and the sample of dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

5.6 Calculate the strength of the dyestuff under test for each recommended percentage by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff under test in percent,

A = percentage dyeing of standard dyestuff, and

B = percentage dyeing of dyestuff under test (see 5.5).

5.7 Calculate the average of the two values obtained in 5.6.

6 REPORT

6.1 Report the value obtained in 5.7 as the strength of the dyestuff under test as compared to the standard dyestuff.

7 PREPARATION OF TEST SAMPLE

7.1 If the dye is marketed in powder form, draw from each container selected, a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

7.2 For dyes marketed in liquid emulsion or paste form, shake each container as selected in 7.1 thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 7.1.

TABLE 1 VATTING AND DYEING CONDITIONS FOR TEST

(Clauses 5.2, B-4.1 and Note Under B-4.3.1)

SL No.	DYEING METHOD	PERCENT-AGE DYE-ING	VATTING CONDITIONS					BULKING AND DYEING CONDITIONS				
			Quan- tity of Dye- stuff (g)	Vatt- ing Volume (ml)	Sodium Hydro- xide 53°Tw (ml)	Sodium Hydro- sulphite (g)	Vatting Tem- pera- ture (°C)	Sodium Hydro- xide 53°Tw (ml)	Sodium Hydro- sulphite (g)	Sodium Chloride (g)	Total of Dye Liquor (ml)	Dyeing Tem- pera- ture (°C)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	A/Q1	1.0 and 2.5	1.0	75	4.5	1.5	55-60	1.5	0.5	—	300	55-60
ii)	A/Q2		and	75	2.0	1.5	45-50	1.5	0.5	6.0	300	45-50
iii)	A/Q3		2.5	75	2.0	1.5	35-40	1.5	0.5	10.0	300	25-30
iv)	A/Q1+			75	—	—	55-60	—	—	—	300	60-65

NOTES

1 The temperatures mentioned in col 8 and 13 refer to the temperatures of dye liquor in the dye vessels and not to the temperatures of water bath.

2 The conditions given in are for guidance only. Wherever special instructions are supplied by the manufacturers, these should be followed.

3 For a particular dye, the method giving maximum yield, as recommended by the manufacturer, should be followed.

4 The recommended percentage dyeings of standard dyestuff is shown under col 3. Suitable dyeing percentage of the dyestuff under test should be employed to obtain a shade within the range of standard dyeing, which is convenient for visual comparison.

5 The vatting, bulking and dyeing conditions would remain unchanged within 10 percent in the percentages of dyeings taken.

ANNEX A

QUALITATIVE METHOD FOR DETERMINATION OF COMPONENTS IN
VAT DYESTUFF

A-1 PAPER CHROMATOGRAPHY TEST

A-1.1 Apparatus

A-1.1.1 Two Plate Glasses

Evenly smooth, each of size 25×25 cm. The thickness of each plate shall be 1 cm. One plate shall have a centre hole of 1.5 cm diameter.

A-1.1.2 Conical Flasks

of 250 ml capacity.

A-1.1.3 Pipettes

of 1, 10 and 25 ml capacities.

A-1.1.4 Watch Glasses

A-1.1.5 Measuring Cylinder

of 100 ml capacity.

A-1.1.6 Water Bath

A-1.1.7 Filter Paper

of 3 mm thickness and of the size of plate glasses.

NOTE — Whatman filter paper No. 1 (for chromatography) is suitable for this test.

A-1.2 Reagents

A-1.2.1 Sodium Hydroxide Solution

sp gr 1.27 (or 53°Tw).

A-1.2.2 Sodium Hydrosulphite

of minimum 85 percent strength.

A-1.2.3 Pyridine

boiling range 114 to 116°C .

A-1.2.4 Lint Dust or any Other Suitable Filtering Aid

A-1.3 Procedure

A-1.3.1 Place the filter paper in between the two plate glasses, the one with centre hole coming on top. Pack tightly and uniformly half the height of the centre hole with lint dust.

A-1.3.2 Weigh about 0.05 g of the dyestuff under test on a watch glass. Transfer the dyestuff to the 250 ml conical flask using 10 ml of distilled water. Add 2 ml of sodium hydroxide solution followed by 4 ml of pyridine. Make up the volume to 100 ml. Heat the contents in the flask in the water bath to the vatting temperature as given in Table 1. Add 0.5 g of sodium hydrosulphite and vat the dyestuff for 10 minutes.

A-1.3.3 Prepare an eluting solution by taking 80 ml of distilled water in a 250 ml conical flask

and adding to it 5 ml of sodium hydroxide solution, 2.5 g sodium hydrosulphite and 15 ml of pyridine in the same order. Stopper the flask with a rubber bung.

A-1.3.4 Put 1 ml dye solution (see A-1.3.2) on lint dust in the centre hole of the glass plate. Immediately add eluting solution in the centre hole so that there shall be no oxidation of leuco vat dyestuff. Cover the centre hole with a small watch glass to avoid oxidation of leuco vat dyestuff by exposure to air. Continue the elution till the wet halo nearly reaches the limits of the filter paper (see Note). Remove the filter paper from the plate glasses. Dry the filter paper and simultaneously oxidize the leuco vat dye on it by hanging the filter paper from a peg in air. Examine the filter paper for the presence of components.

NOTE — During elution, the central portion of the filter paper should not be allowed to dry. The watch glass should be removed only for the addition of eluting solution. By the time the wet halo reaches the limits of the paper, the components in the mixture get clearly separated into different coloured bands. Even though the chromatograph obtained during elution would indicate the presence of components, the results should be assessed on the oxidized dyestuff on chromatograph.

A-2 BLOW TEST

A-2.1 Apparatus

A-2.1.1 White Enamelled Tray

$25 \times 20 \times 4.5$ cm in size.

A-2.1.2 Filter Paper

of 3 mm thickness and of 30×18 cm size.

NOTE — Whatman filter paper No. 1 is suitable for the test.

A-2.1.3 Glass Rod

of 5 mm diameter.

A-2.1.4 Folded Filter Paper

of size 10×1.5 cm and folded lengthwise.

A-2.2 Reagents

A-2.2.1 Sodium Hydroxide Solution

sp gr 1.27 (or 53°Tw).

A-2.2.2 Sodium Hydrosulphite

of minimum 85 percent strength.

A-2.2.3 Turkey Red Oil

A-2.3 Procedure

A-2.3.1 Pour 1 500 ml of distilled water at 70 to 75°C into the clean enamelled tray. Add 30 ml of sodium hydroxide solution, 7.5 g of sodium hydrosulphite and 3 drops of Turkey red oil. Stir the solution well.

A-2.3.2 Introduce one filter paper (*see* A-2.1.2) into the tray. Take enough of dyestuff under test on the folded filter paper and blow it on the surface of the solution in the tray (*see* Note 1). Vat the dyestuff for 5 to 10 minutes (*see* Note 2).

Take out the filter paper from the tray. Wash it in cold running water. Oxidize the leuco vat dyestuff in air and dry the filter paper in an atmosphere free from dust. Examine the filter paper for the presence of colour components.

NOTES

1 While blowing the dyestuff, care should be taken so that the dyestuff is not allowed to fall as a lump on the surface of the solution, in which case the identification of the constituents is rendered difficult.

2 Careful observation of leuco vat dyestuff would be useful in the identification.

ANNEX B

GENERAL METHOD FOR DYEING VAT DYESTUFFS

B-1 APPARATUS

B-1.1 Dye Vessels

porcelain or stainless steel beakers or dye vessels provided for mechanically agitated dye baths (*see* B-4.3).

B-1.2 Watch Glass

B-1.3 Graduated Pipette

capable of measuring correct to 0.1 ml.

B-1.4 Water Bath

B-2 DYEING ASSISTANTS

B-2.1 Water

Distilled water shall be used in the preparation of the dye bath (*see* B-4.3).

NOTE — For rinsing and soaping, water having hardness of not more than 50 ppm expressed as calcium carbonate may be used.

B-2.2 Sodium Hydroxide Solution

sp gr 1.27 (or 53°T_w).

B-2.3 Sodium Hydrosulphite

of minimum 85 percent strength.

B-2.4 Vat Yellow G Paper

B-2.5 Phenolphthalein Paper

B-2.6 Wetting Agent

methylated spirit or highly sulphonated castor oil.

B-2.7 Dilute Hydrochloric Acid

containing 2 ml/l of hydrochloric acid (sp gr 1.16).

B-2.8 Soap Solution

5 g/l.

B-3 PREPARATION OF HANKS FOR DYEING

B-3.1 Test Hanks

A sufficient number of hanks of scoured, bleached, unmercerized cotton yarn having no finishing chemical or blueing agent shall be used for the test. Each hank should weigh 10 ± 0.1 g.

NOTES

1 Any yarn normally used in laboratories for carrying out dyeing trials or yarn of the following requirements is suitable for this test:

- a) Count — 10 tex \times 2 (or 60s/2),
- b) Twist per metre — 750, and
- c) Cuprammonium fluidity not more than 5 rhes.

2 If the mass of a hank is not 10 ± 0.1 g. it should be weighed accurately, and the amount of dyestuffs and chemicals to be taken should be calculated accordingly.

B-3.2 Preparation of Test Hanks

The hanks shall be treated in boiling water for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered in the dye bath.

B-4 PROCEDURE

B-4.1 Vatting and Dyeing Conditions

The vatting and dyeing conditions for the recommended percentage dyeing shall be as given in Table I.

B-4.2 Liquor Ratio

The liquor ratio shall be 1 : 30 in all operations, unless otherwise stated.

B-4.3 Preparation of the Dye Bath

B-4.3.1 Weigh accurately the requisite quantity of dyestuff in a watch glass and transfer it to

the dye vessel with little water. Paste the dyestuff with the necessary amount of wetting agent and add sufficient amount of hot water with constant stirring. Add more hot water to make up the volume to 75 ml for vatting. Add the recommended quantity of sodium hydroxide solution with constant stirring and heat the dye liquor to the recommended temperature for vatting. Add the recommended quantity of sodium hydrosulphite with constant stirring.

Allow the dye to reduce at this temperature for 15 minutes with occasional stirring.

NOTE — The required amount of dyestuff should be weighed accurately for each hank separately; in case a sensitive balance for the purpose is not available, a large amount of dyestuff (such as 5.0 g) may be weighed and a stock vat of the dyestuff prepared. The quantities of sodium hydroxide and sodium hydrosulphite shall be such as to ensure complete reduction of the stock vat. Aliquot amounts of the stock vat shall be pipetted out for each dyeing as required. The final dyeing volume shall contain the recommended quantities of sodium hydroxide and sodium hydrosulphite (*see* Table 1).

B-4.3.2 Prepare the bulk solution by adding the recommended quantities of sodium hydroxide and sodium hydrosulphite in the remaining amount of water kept at the recommended temperature, and add this to the dye bath with stirring.

B-4.4 Dyeing

Stir the dye liquor and enter the wetted hank (*see* B-3.2). Agitate the liquor to obtain a level dyeing. Dye the hank for 45 minutes at the

recommended temperature agitating the dye liquor at frequent intervals. Care should be taken to keep the hank immersed below the surface of the liquor. Avoid excessive aeration of the hank to prevent oxidation of the reduced dye. Remove the hank from the dye bath. Squeeze it and allow it to oxidize in air for 5 minutes. Rinse it in cold running water. Treat the hank for 2 minutes in cold dilute hydrochloric acid. Remove the hank and wash it well in cold running water and soap it as in B-4.5.

NOTES

1 During dyeing, the dye liquor shall be tested frequently for the presence of sodium hydrosulphite and sodium hydroxide by spotting vat yellow G paper and phenolphthalein paper, respectively. Vat yellow G paper turns blue in the presence of sodium hydrosulphite and phenolphthalein paper turns pink in the presence of sodium hydroxide.

2 All dyeings should be carried out simultaneously in the same dye bath in separate vessels to ensure identical conditions.

3 After air oxidation, the dyed hanks shall be developed for 15 minutes in a cold aqueous solution of sodium hypochlorite containing 3 g/l available chlorine. The hanks are then washed thoroughly, scoured, washed and soaped as usual.

B-4.5 Soaping

Enter the hank in a boiling soap solution (*see* B-2.8) containing 2 g/l sodium carbonate, anhydrous and continue the treatment at boil for 15 minutes with frequent stirring. Remove the hank and rinse it well in running water. Dry the hank in an oven at a temperature not exceeding 70°C.

ESTIMATION OF STRENGTH (VAT CONTENT) OF SOLUBILIZED VAT DYESTUFFS

(Source : IS 5970 : 1970)

Solubilized vat dyestuffs are increasingly being used in the industry for dyeing and printing of fabrics. The strength of solubilized vat dyes is generally determined by comparison with an agreed standard by dyeing test which creates difficulties in reporting records of production, import or export statistics, or comparing dyes, where due to slight shade difference, the assessment of strength by dyeing method becomes

difficult. To overcome this difficulty, this standard has been prepared to determine the strength by gravimetric method which agrees with the dyeing tests. The method prescribed is applicable to individual dyes but not to a mixture of dyes. It takes care of dyes which are precipitated due to over oxidation. However, it may not be very much suitable for dyes which are partially oxidized and pass through the filtering media.

1 SCOPE

1.1 This gravimetric method is intended for the estimation of strength (vat content) of homogeneous solubilized vat dyestuffs.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 APPARATUS AND REAGENTS

3.1 Sintered Glass Funnel and Crucible porosity G4.

3.2 Quality of Reagents

Unless otherwise specified, pure chemicals shall be used in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

3.2.1 Sodium Nitrite Solution

10 percent (w/v).

3.2.2 Dilute Sulphuric Acid

prepared by diluting 10 ml of concentrated sulphuric acid to 100 ml.

4 PROCEDURE

4.1 Take about 2 g of solubilized vat dyestuff and weigh it accurately. Dissolve the dyestuff in 200 ml of distilled water at 60°C. Filter the solution while hot through sintered glass funnel and wash

the funnel with hot distilled water until the dissolved dyestuff goes into the filtrate.

4.2 Add 10 ml of sodium nitrite solution and 10 ml of dilute sulphuric acid to the solution obtained in 4.1. Boil for 5 minutes and filter it through a previously weighed sintered glass crucible. Wash the residue on the crucible with distilled water till free from acid using an appropriate indicator, such as bromocresol purple. Dry the crucible along with the residue at $100 \pm 3^\circ\text{C}$ to constant mass, Determine the weight of the residue.

4.3 Repeat the test with at least two more test specimens.

5 CALCULATIONS

5.1 Calculate the strength of the solubilized vat dyestuff by the following formula:

$$P = \frac{W_2}{W_1} \times 100$$

where

P = percentage by mass of the vat content of solubilized vat dyestuff,

W_2 = mass in g of the residue obtained as in 4.2, and

W_1 = mass in g of the solubilized vat dyestuff taken for the test (see 4.1).

5.2 Similarly, calculate the strength of the other test specimens and calculate the average of the results.

6 REPORT

6.1 The report shall include the following:

- Strength of dyestuff in percent, and
- Number of tests.

DETERMINATION OF STRENGTH OF INDIGO IN SUBSTANCE

(Source : IS 11636 : 1986)

Synthetic indigo, which now forms the chief source of supply, comes in the form of a fine powder containing 96 to 98 percent pure indigo (indigotine). It is also sold as a thin paste containing 20 percent of dyestuff or in the form of a concentrated solution of the leuco compound (reduced indigo). Natural indigo is less pure and contains indigo red (indirubine), small quantities of certain brown and yellow colouring matter, glutinous material and mineral matter (clay and sand).

The methods given in this standard are based on the oxidation of indigo to isatine by use of a standard solution of potassium permanganate.

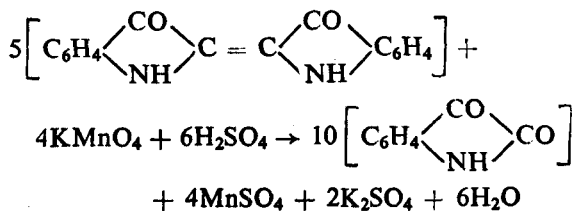
Disulphonic acid method also gives an approximate estimation of indirubine present in natural indigo and is less accurate. Tetrasulphonic acid method is not as rapid as the disulphonic acid method but is more accurate and is to be preferred when impure indigos are to be estimated. This method also gives exact estimation of indirubine present in natural indigo. With indigos of high purity, both the methods agree well. In the case of both methods, however, the presence of starch in the product may lead to erroneous (too low) results. In such cases, it is necessary to previously boil the sample with dilute hydrochloric acid (4 percent) for an hour before proceeding to analysis.

1 SCOPE

1.1 These methods are intended for the estimation of pure colouring matter (indigotine) present in the commercially available natural and synthetic indigo in powder or paste form or in the form of concentrated solution of leuco compound.

2 PRINCIPLE

2.1 A test sample of commercial indigo is first converted into a disulphonic acid or into a tetrasulphonic acid to render it soluble in water and is then oxidized by a standard solution of potassium permanganate to form isatinmono- or disulphonic acid. The reaction proceeds theoretically according to the equation:



On account of the high dilution or for other unascertained reasons, the reaction does not take place strictly in accordance with the above equation and a factor is, therefore, used which represents the results obtained experimentally with pure indigotine under the same conditions. This factor varies somewhat with the method of estimation employed.

3 PREPARATION OF TEST SAMPLE

3.1 If the indigo is in powder form, draw randomly small quantities of dyestuff by a suitable sampling instrument from at least five different parts of each selected container and mix them thoroughly to get a test sample of about 20 g.

If the indigo is in paste or concentrated solution form, stir the contents of each container selected thoroughly with a wooden stick so as to homogenize the contents. Then draw randomly small quantities of dyestuff by a suitable sampling instrument from at least five different parts of each selected container and mix them thoroughly by stirring with a glass rod so as to get a test sample of about 20 g.

4 STANDARD INDIGO

4.1 The standard sample of indigo, against which the strength of indigo under test is evaluated, shall be as agreed to between the buyer and the seller.

5 APPARATUS**5.1 Drying Oven**

capable of drying at $110 \pm 2^\circ\text{C}$.

5.2 Weighing Balance

with an accuracy of 1 mg.

5.3 Glass Rod

for stirring purpose.

5.4 Glass Beakers

capacity of 250 ml.

5.5 Water Bath

capable of heating at 70 to 75°C .

5.6 Conical Flasks

500 and 200 ml capacity.

5.7 Titration Flask

500 ml capacity.

5.8 Burette

graduated in ml.

5.9 Pipette

25 ml capacity.

5.10 Gooch Crucible

30 ml capacity

5.11 Funnel**5.12 Shallow Porcelain Dish**

of suitable size.

6 REAGENTS**6.0 Quality of Reagents**

Unless otherwise specified, pure reagents shall be employed. Whenever the use of water as a reagent is intended, distilled water shall be used.

NOTE — Pure reagents shall mean reagents which do not contain impurities which affect the results of test or analysis.

6.1 20 g of White Quartz Sand**6.2 Pure Concentrated Sulphuric Acid**98 percent (*m/v*).**6.3 Barium Chloride Solution**10 percent (*m/v*).**6.4 Potassium Permanganate Solution**

N/50, prepared by dissolving 0.632 g potassium permanganate per litre of the solution with water.

6.5 Fuming Sulphuric Acid

20 percent SO₃ for fairly pure indigos and 25 percent SO₃ for crude indigos.

6.6 Potassium Acetate Solution

450 g/l.

6.7 Ice**6.8 Glacial Acetic Acid****6.9 Hydrochloric Acid Solution**4 percent (*m/v*).**7 PROCEDURE COMMON TO BOTH METHODS**

7.1 Take the required quantity of test specimen (*see* 7.2 and 7.3) and indigo from the test sample selected (*see* 3.1) in a beaker and boil the test specimen with dilute hydrochloric acid (4 percent, *m/v*) for an hour to remove any starch if present. Extract the boiled specimen with distilled water thoroughly and proceed for estimation as given in 7.2 and 7.3.

7.2 Disulphonic Acid Method

7.2.1 Weigh accurately 0.5 g of the test specimen and treat as in 7.1.

7.2.2 Dry it at $110 \pm 2^\circ\text{C}$ in the drying oven for 2 to 4 hours till a constant mass is obtained. Take it into a beaker and add to it about 3 g of white quartz sand and 20 ml of pure concentrated sulphuric acid.

7.2.3 Mix the contents with a glass rod and heat in a water bath at a temperature of 70 to 75°C for an hour, the mixture being meanwhile occasionally stirred.

7.2.4 Pour off the sulphuric acid solution from the sand into a 500 ml flask.

7.2.5 Transfer the washing of the remaining sand, glass rod and the beaker with water into the flask.

7.2.6 Dilute the contents of flask (*see* 7.2.5) to about 400 ml and add 10 ml of a 10 percent barium chloride solution and make the whole up to 500 ml with water.

NOTE — The barium sulphate formed settles quickly and carries down with it impurities present in the indigo which would otherwise mask the end point of the titration.

7.2.7 After keeping the contents of flask as in 7.2.6 for half an hour, draw 50 ml of clear solution in the titration flask, dilute it with 300 ml of distilled water and titrate the contents with N/50 potassium permanganate solution. Note the end point when the solution has a pale yellow or orange colour free from any bluish or greenish tint. Note the volume of potassium permanganate used to reach the end point.

7.2.8 Calculate the quantity of indigotine present in the test sample by factor : 1 ml of N/50 KMnO₄ = 0.001 47 g indigotine.

7.2.9 Repeat the procedure from 7.2.1 to 7.2.8 for the standard indigo (*see* 3.1).

7.2.10 Calculate the percentage strength of indigo under test by the following formula:

$$S = \frac{V_2}{V_1} \times 100$$

where

S = percentage strength of indigo under test,

*V*₁ = volume of N/50 KMnO₄ used in titration of standard indigo, and

*V*₂ = volume of N/50 KMnO₄ used in titration of indigo under test.

NOTES

1 With natural indigo containing more than 1 or 2 percent of indirubine, the end point, instead of being yellow, is orange or red whose tint, however, changes to yellow upon further addition of potassium permanganate. The quantity of indirubine present thus may be calculated approximately as follows:

Percentage of indirubine on pure indigotine basis

$$= \frac{V_2 - V_1}{V_2} \times 100$$

where

V_3 = volume of potassium permanganate used when the end point is yellow, and

V_4 = volume of potassium permanganate used when the end point is orange or red.

2 For more exact estimation of indirubine, tetrasulphonic acid method should be used.

7.3 Tetrasulphonic Acid Method

7.3.1 Weigh accurately 1 g of the test specimen and treat as given in 7.1. Take it into a beaker and dry in the drying oven at $110 \pm 2^\circ\text{C}$ for 2 to 4 hours till a constant mass is obtained. Add to it 3 g of white quartz sand and 5 ml of fuming sulphuric acid (20 percent SO_3 for fairly pure indigos or 25 percent SO_3 for crude indigos). Stir the mixture with a glass rod, cover with a watch glass and heat in the water bath for 45 minutes at 96 to 98°C stirring at intervals.

7.3.2 Then cool the contents to room temperature, transfer the clear sulphonic acid solution to a 500 ml flask containing a little water and add to it the washings of the beaker, sand and the glass rod. Make the volume up to 500 ml with water.

7.3.3 Take 100 ml of a solution obtained in 7.3.2 into a conical flask and add to it 100 ml of a solution of potassium acetate (450 g/l). Heat the mixture just to boiling point in order to dissolve the precipitate first formed. Then cool it at once in running water and finally allow to stand for an hour upon ice.

NOTE — If the acetate solution is boiled for any length of time, there is a liability for destruction of indigo to occur.

7.3.4 Collect the potassium tetrasulphonate, which separated out upon cooling as a crystalline precipitate, in a Gooch crucible and wash it with an ice cold solution containing 225 g/l of potassium acetate and 125 g/l of glacial acetic acid.

NOTE — If the indigo is pure, the filtrate is nearly colourless, or at most has only a pale blue tint. With impure indigos the colour of the filtrate will depend upon the character of impurities. Thus, if indirubine is present, a red filtrate is obtained.

7.3.5 Place the Gooch crucible containing potassium tetrasulphonate in a funnel resting in the neck of a 200 ml flask and wash the contents with hot water, adding any precipitate which

may have remained adhering to conical beaker. Make the contents of the flask up to 200 ml.

7.3.6 Transfer 200 ml of the solution as obtained in 7.3.5 to a shallow porcelain dish. Add 0.5 ml of pure concentrated sulphuric acid, dilute with 80 ml of water and titrate with a potassium permanganate solution (N/50) till the end point is reached (see 7.2.7 and NOTE 1 under 7.2.10).

7.3.7 Calculate the quantity of indigotine present by the factor : 1 ml of N/50 KMnO_4 = 0.001 47 g indigotine.

7.3.8 Repeat the procedure from 7.3.1 to 7.3.7 for the standard indigo (see 4.1).

7.3.9 Calculate the percentage strength of indigo as follows:

$$\text{Percentage strength} = \frac{V_6}{V_5} \times 100$$

where

V_5 = volume of N/50 KMnO_4 used to reach the end point for the standard indigo (see 4.1), and

V_6 = volume of N/50 KMnO_4 used to reach the end point for the indigo under test.

7.3.10 Percentage of indirubine can be calculated by noting the volumes of N/50 KMnO_4 used, when the end point is orange red and yellow (see Note 1 under 7.2.10), by the following formula:

Percentage of indirubine on pure indigotine basis

$$= \frac{V_7 - V_8}{V_7} \times 100$$

where

V_7 = volume of N/50 KMnO_4 used when the end point for indigo under test is yellow, and

V_8 = volume of N/50 KMnO_4 used when the end point for indigo under test is orange or red.

8 REPORT

8.0 The report shall indicate the following:

- Percentage strength of indigo as obtained in 7.2.10 and 7.3.9.
- Percentage of indirubine present in the indigo on the basis of pure indigotine.

DETERMINATION OF STRENGTH OF INDIGO ON DYED TEXTILES

(Source : IS 11678 : 1986)

The methods prescribed in this standard are based on the extraction of indigo from the dyed textile material by using pyridine or cresol

mixture at the boiling point of the solvent. The accuracy of the method is not affected due to the presence of other dyestuffs in addition to indigo.

1 SCOPE

1.1 This method is intended for determining the percentage strength of indigo on dyed wool, cotton and linen textiles, when present alone or with other dyestuffs.

2 PRINCIPLE

2.1 A suitable quantity of the textile material is extracted with pyridine or cresol mixture at its boiling point. The precipitate obtained on filter is washed successively with hot 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution (1 percent), hot water, alcohol, and finally alcohol and ether. The precipitate is then dried and weighed. An alternative method is to render the precipitate of indigo obtained soluble in water by sulphonation with pure concentrated sulphuric acid and titrating with N/50 potassium permanganate (KMnO_4) solution, and then calculating the amount of indigo by the factor — 1 ml of N/50 KMnO_4 is equal to 0.001 47 g of indigotine.

3 PREPARATION OF TEST SPECIMEN

3.1 The mass of the specimen of the cloth or yarn or fibre to be taken shall be such that it gives 0.03 to 0.10 g of indigo after extraction. This will vary from 3 to 15 g depending upon the depth of indigo present on the textile material. The material shall be loosely packed into the inner tube of the Soxhlet apparatus (see 4.1). If the material to be tested is cloth, it shall be cut into small strips or pieces and then packed into the inner tube in the form of roll.

4 APPARATUS

4.1 Soxhlet Apparatus

As shown in Fig. 1 which consists of air condenser, in which is placed the inner tube containing the textile material. The inner tube may have the various forms (A, B or C) as shown in Fig. 2. In the apparatus of form A, the hot extract is siphoned intermittently into the distilling flask. With form B, the solvent is directed to the bottom of the tube by means of a long funnel, and then rises through the material overflowing through the side opening near the top. In the form C, it percolates continuously through the material, flowing out by the small hole at the bottom of tube. The simplest and preferable form is tube C, as it gives good

results with all cloths and is easy to make. The tube is drawn out at the end, and some glass points are fused on to the outer surface so as to

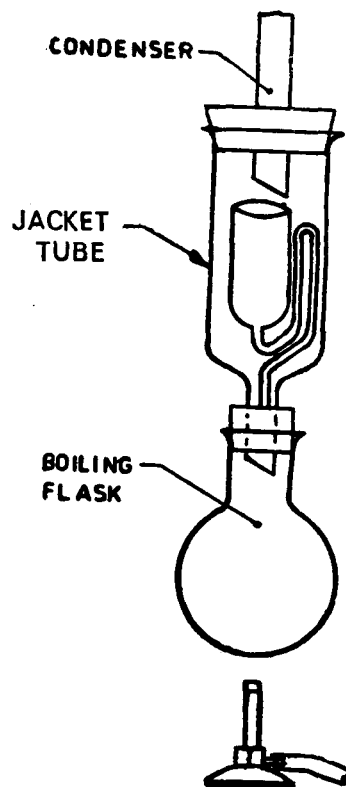
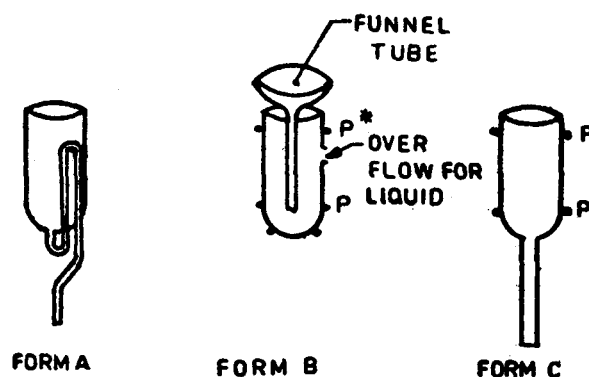


FIG. 1 EXTRACTION APPARATUS



*P — Glass points fixed on to furnish passage for vapour round the inner tube.

FIG. 2 FORMS OF INNER TUBE

furnish a passage for the vapour of the solvent. A little white wool, cotton wool or crushed quartz is placed at the bottom of the tube, as a filtering and regulating medium.

4.2 Conical Flask

500 ml capacity.

4.3 A Gooch Crucible

provided with filter paper or asbestos.

4.4 Drying Oven

capable of heating up to $110 \pm 2^\circ\text{C}$.

4.5 Beaker

250 ml capacity.

4.6 Water Bath

capable of heating up to 70 to 80°C .

4.7 Burette

graduated in 0.1 ml.

4.8 Graduated Cylinder

100 ml capacity.

4.9 Titration Flask

500 ml capacity.

4.10 Steam Oven

for drying.

4.11 Thermometer

capable of measuring a temperature up to 120°C .

4.12 Porcelain Beaker

250 ml capacity.

4.13 Burner

5 REAGENTS

5.0 Quality of Reagents

Unless otherwise specified, pure reagents and distilled water shall be employed in the tests.

NOTE — Pure reagents shall mean reagents which do not contain impurities that affect the test results.

5.1 Commercial Pyridine

5.2 Alcohol

50 percent (v/v).

5.3 Sodium Hydroxide Solution

2 percent (m/v).

5.4 Dilute Hydrochloric Acid

1 percent (v/v).

5.5 Ether (Diethylether)

5.6 Pure Concentrated Sulphuric Acid (AR Grade)

5.7 Potassium Permanganate Solution

N/50.

5.8 Solvent Mixture

a mixture of 75 parts of cresol with 25 parts of heavy petroleum spirit (b. p. 155 to 170°C).

5.9 Sulphuric Acid

80 percent (m/v).

5.10 Dilute Ammonia

10 percent (m/v).

6 PROCEDURE

6.1 Estimation of Indigo on Textiles

6.1.1 Weigh the required quantity of the dyed textile (*see* 3.1) accurately after heating it in an air oven at $105 \pm 3^\circ\text{C}$ for 2 to 4 hours to constant mass and pack it loosely into the inner tube of the Soxhlet apparatus (*see* Fig. 1).

6.1.2 Take 100 ml of commercial pyridine in the boiling flask of the Soxhlet apparatus and heat it over wire-gauze or upon an air bath. Continue the extraction until the extract no longer has a blue colour. This usually requires two hours. The thickest material can be completely extracted in four hours at most.

NOTE — Instead of pyridine, a cresol mixture (*see* 5.8) may also be used for extraction.

6.1.3 Distil down the extract to about 20 or 30 ml and cool the extraction flask along with contents when the greater part of the indigo separates in well-formed bronzy crystals. To complete the precipitation, add to it 100 ml of 50 percent alcohol.

6.1.4 Heat the contents of the flask to boil for about 10 to 20 minutes and filter through a Gooch crucible provided with filter paper or asbestos into a beaker.

6.1.5 Wash the precipitate on filter successively with hot (50 to 60°C) 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution (1 percent), hot water, alcohol, and finally with alcohol and ether.

NOTE — The appearance of the indigo is a guide to its purity. It should form a bronzy crystalline powder testing 100 percent by the tetrasulphonic acid method (*see* IS 11636 : 1986). A dull appearance shows the presence of impurities.

6.1.6 Collect the washed precipitate on the Gooch crucible, the bottom of which is covered with a little asbestos and dry it for 10 to 20 minutes. Place the crucible in a small beaker containing 15-20 ml of pure concentrated

sulphuric acid and heat in water bath at 70 to 80°C for about 45 minutes.

6.1.7 Take the solution in the titrating flask of 500 ml capacity and then make it up to the mark with distilled water. Titrate 100 ml of this solution in 200 ml water against N/50 potassium permanganate solution till end point is obtained (see Note). Note the volume of N/50 potassium permanganate solution required to reach the end point.

NOTE — The end point is obtained when the solution has a pale yellow or orange colour free from any bluish or greenish tint.

6.1.8 Repeat the procedure given in 6.1.7 for remaining 400 ml solution 4 times and find the average volume of potassium permanganate used.

6.1.9 Calculate the percentage strength of indigo on the textile by the following formula:

$$S = \frac{0.00147 \times V \times 5 \times 100}{M}$$

where

S = percentage strength of indigo on the textile,

V = average volume of N/50 KMnO_4 used (see 6.1.8), and

M = mass of the test specimen (oven dry basis) (see 6.1.1).

7 REPORT

7.1. Report the percentage strength of indigo (see 6.1.9) present on the textile.

TESTING OF VAT DYESTUFFS

(Source : IS 12547 : 1988)

Vat dyestuffs are most commonly used for dyeing and printing of cellulosic textile materials because of their very good all round fastness properties. Commercial vat dyestuffs are marketed in different forms, such as paste, supra paste, double paste, powder, powder concentrate, powder highly concentrated, 400 powder, 800 powder, 2 100 powder, 4 200 powder, highly concentrated, ultra concentrated, microfine, ultra disperse, colloidal and FD grains. These various forms of vat dyestuffs differ from one another in respect of dye content, filtering capacity, limit of water suspension, wettability and spreading of dye in water, rate of development and fixation, redox potential, migration properties, strike and levelling properties, etc. Depending upon the level of impurities present

in the dyestuff, the same dyestuff obtained from different manufacturers may differ in: (a) ease of reduction, (b) affinity of the sodium salt of the leuco vat dye towards cellulose, (c) ease of dissolution of the leuco vat dye in alkali, (d) rate and extent of dye uptake by cellulose, (e) ease of re-oxidation of the sodium salt of leuco vat dye into the original vat dye, (f) stability of dye, (g) sensitivity towards retarding or exhausting agents during dyeing and (h) the ability to respond towards certain aftertreatments such as soaping at the boil.

In order to ascertain the exact end use and optimum suitability for a particular process, the testing of various characteristics of the vat dyestuffs is of considerable importance.

1 SCOPE

1.1 The methods of determining dye content, filtering capacity limit of water suspension, wettability and spreading of dye in water, rate of development and fixation, migration and strike of vat dyestuffs are described.

2 DETERMINATION OF DYE CONTENT

2.1 Take a test specimen of dyestuff of about 1 g in a weighing bottle and dry it to constant mass in a drying oven at $105 \pm 3^\circ\text{C}$. Cool the specimen in a desiccator charged with self indicating silica gel or calcium chloride and obtain the oven-dry mass of the specimen to an accuracy of 0.000 2 g with the help of analytical balance.

2.2 Prepare leuco vat of the dye as prescribed below:

Prepare a paste of 0.1 g of dye in Turkey red oil or a suitable wetting agent in a beaker. Add 20 ml of hot water (50°C) to the paste with constant stirring. Then add the required quantity of sodium hydroxide and sodium hydrosulphite with constant stirring and allow to stand for 5-10 min (see IS 4394 : 1988).

2.3 Filter the solution on a Buchner funnel through Whatman filter paper No. 41 under low pressure of 320 mm of mercury. Repeat the procedure twice more with the residue left and collect all the filtered solution in a beaker.

2.4 Oxidize the solution obtained in 2.3 by bubbling a strong current of air through it for about 20 min and filter the solution through a weighed sintered filter crucible. Repeat the procedure twice more with the solution left and collect all the residues on sintered filter crucible.

Dry the crucible and the residue to constant mass in a drying oven at $105 \pm 3^\circ\text{C}$, cool in a desiccator charged with self-indicating silica gel or calcium chloride and weigh them to obtain the oven-dry mass of the residue.

2.5 Calculate the dye content, percent (P) by the formula:

$$P = \frac{m_1}{m_2}$$

where

m_1 = oven-dry mass of the residue, and

m_2 = oven-dry mass of the specimen taken.

3 DETERMINATION OF FILTERING CAPACITY LIMIT OF WATER SUSPENSION OF THE DYE

3.1 Take 0.5 to 1.0 g samples of the dye in steps of 0.1 g and weigh them correct to 0.01 g. Introduce the samples in cups of 150 ml capacity containing 50 ml of distilled water heated to 70°C . Add the dye in each cup and then make the volume in each cup up to 100 ml by adding distilled water at 70°C with constant stirring. Place the suspension in each cup for 5 minutes on a water bath at 70°C with constant stirring. Then cool the suspension and filter on a Buchner funnel through a Whatman filter paper No. 41 under low pressure of 320 mm of mercury.

3.2 Take that mass of the sample as filtering capacity limit for which the filter paper is uniformly coloured and the residues of the dye are visible in the place over the holes of the funnel.

4 DETERMINATION OF WETTABILITY AND SPREADING OF DYE IN WATER

4.1 Introduce 0.02 g of the test dye and the same amount of the standard sample of dye in separate glass cups of 200 ml capacity, in each add 100 ml of distilled water. The test dye shall get wet and spread in the water, at least, as much as the standard sample of dye (*see* IS 12547 : 1988).

5 DETERMINATION OF RATE OF DEVELOPMENT AND FIXATION OF DYE

5.1 Carry out dyeing of a hank with the dye as prescribed in Method A/Q1 of IS 4394 : 1988.

5.2 Take another five hanks and dye as given in 5.1 except that the amount of dye and chemicals shall be double to that taken in 5.1 and time of dyeing shall be 5, 10, 15, 20, 25 minutes for each hank respectively.

5.3 Match the samples obtained in 5.2 with that of 5.1 and note the time of dyeing for the sample obtained in 5.2 which exactly matches with the sample in 5.1.

5.4 Record the time of dyeing noted in 5.3 as the time of half dyeing for the dye.

NOTE — The time of half dyeing is a measure of rate of development and fixation of the dye.

6 DETERMINATION OF MIGRATION PROPERTIES

6.1 Carry out the treatments of an already dyed hank and a white hank of cotton yarn at 50°C for 40 minutes at a liquor ratio of 1 : 30 in a blank dye bath simultaneously by the method A/Q1 as prescribed in IS 4394 : 1988. Also after-treat the hanks as prescribed in IS 4394 : 1988.

NOTE — The blank dye bath contains all other chemicals except the dyestuff and the common salt.

6.2 Similarly carry out treatment of another pair of a dyed and white hank of cotton yarn at 80°C in a blank bath for 40 minutes at a liquor ratio of 1 : 30 and after-treat as given in 6.1.

6.3 Assess the staining of the white-treated hank by comparing with the dyed-treated hank with the help of grey scale for staining.

7 DETERMINATION OF STRIKE

7.1 Prepare a dye bath of 5 percent depth for a 5 g cotton yarn hank as prescribed in IS 4394 : 1988. Enter the hank in dye bath, and turn steadily and continuously. After 2 minutes, enter a second 5 g hank in the dye bath and turn both the hanks in the same way. After next 3 minutes, remove both the hanks and after-treat them by the procedure given in IS 4394 : 1988.

EVALUATION OF STRENGTH OF REACTIVE DYES (MONOCHLOROTRIAZINYL TYPE) BY DYEING TEST

(Source : IS 7478 : 1974)

Reactive dyes are marketed in different strengths. This method for determining the strength of dyestuff against a mutually accepted

standard would be useful for assessing the strength of dyestuff.

1 SCOPE

1.1 This method is intended for the determination of strength of reactive dyes (monochlorotriazinyl type) by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye from at least three different parts by a suitable sampling instrument and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in test and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Prepare dyeings of the standard sample of dyestuff (see 3.1) by following the procedure given in Annex A; prepare simultaneously additional dyeings of the standard sample with the percentage variation of dyeing strength by 5 percent on the lower side of the recommended percentage.

5.2 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex A.

5.3 Compare the dyeings obtained in 5.2 and 5.1. Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentage of the dyeings which matches exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of backing on the appearance. The hanks should be compared, if possible, in North skylight. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

6 CALCULATION

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test.

7 REPORT

7.1 The report shall include strength of dyestuff in percent.

ANNEX A

METHOD FOR DYEING REACTIVE DYES (MONOCHLOROTRIAZINYL TYPE)

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel beakers, or dye vessels provided with mechanically agitated dye baths.

A-1.2 Graduated Pipettes

capable of measuring correct to 0.1 ml.

A-1.3 Volumetric Flask

calibrated to 500 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Chloride Solution or Anhydrous Sodium Sulphate Solution

20 percent (m/v).

NOTE — Quality of sodium sulphate is extremely important. Commercial brands may be acidic or alkaline, therefore, they shall be neutralized.

A-2.3 Sodium Carbonate Solution

10 percent (m/v).

A-2.4 Soap Solution

containing 0.3 percent (m/v) of neutral detergent.

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, bleached, unmercerised cotton yarn, having no finishing chemicals or blueing agent, shall be used in this test. Each hank should weigh 10 ± 0.1 g (see Note 2).

NOTES

1 Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

- a) Count — 10 tex \times 2 or (60 $\frac{2}{2}$),
- b) Twist per metre — 750, and
- c) Cuprammonium fluidity not more than 5 rhes.

2 If the mass of the hank is not 10 ± 0.1 g, then it should be weighed accurately and the amount of dyestuff and the chemicals to be taken should be calculated accordingly.

A-3.2 Preparation of the Test Hanks

The hanks shall be treated in boiling water for 10 minutes, squeezed evenly to contain approxi-

mately its own mass of water, cooled and entered into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of the Dyestuff Solution

Weigh accurately 1.0 g of the dyestuff under test. Paste the dyestuff with cold water and dissolve by adding warm water (not higher than 80°C). The total volume of water used for dissolution of the dyestuff should not exceed 100 ml. Dilute the solution with cold water and make up to 500 ml in a volumetric flask.

A-4.1.1 Similarly, prepare a solution of the standard dyestuff by following the procedure given in A-4.1 but taking the standard dyestuff instead of the dyestuff under test.

A-4.2 Dyeing (for 10 ± 0.1 g Hank)

A-4.2.1 Pipette out separately the required amount of solution of dyestuff under test in the dye vessels so as to give 1.9, 2.0 and 2.1 percent depth. Add the requisite quantity of water and 100 ml of sodium chloride solution or anhydrous sodium sulphate solution to make the volume of the dye liquor 300 ml leaving sufficient margin for the addition of alkali. Heat the dye bath to 40°C, stir the dye liquor and enter the wetted hanks. Raise the temperature to 80°C in 15 minutes and continue dyeing for further 30 minutes. Turn the hanks frequently so as to obtain level dyeings. Add 60 ml of anhydrous sodium carbonate solution and dye for a further 30 minutes. Remove the dyeings, squeeze the dyed hanks evenly. Rinse the dyeing first in cold water and then in hot water. Treat the dyed hanks at boil for 15 minutes in a soap solution at liquor ratio of 1 : 30; rinse in cold water and again treat the hanks at boil for 15 minutes in soap solution at liquor ratio of 1 : 30. Finally, rinse the dyeings in cold water and dry.

NOTE — For reactive blue 5, blue 25 and similar products — Dissolve these dyes at 50°C. Use 50 parts (maximum) anhydrous sodium sulphate (do not use sodium chloride) and dye at temperature of 90°C.

Further addition of alkali — In addition to anhydrous sodium carbonate solution, add predissolved caustic soda (100 percent), 2 parts per 1 000 parts of dye liquor soon after the dye bath has attained the final dyeing temperature.

A-4.2.2 Similarly, pipette out separately required amounts of solution of the standard dyestuff in separate dye vessels (see Note). Dye the hanks by following the method given in A-4.2.1.

NOTE — The dyeings with the solutions of the dyestuff under test and of the standard dyestuff should be done simultaneously in the same water bath.

EVALUATION OF STRENGTH OF REACTIVE DYES (DICHLOROTRIAZINYL TYPE) BY DYEING TEST

(Source : IS 7447 : 1974)

Reactive dyes are marketed in different strengths. This method for determining the strength of dyestuff against a mutually accepted

standard would be useful for assessing the strength of dyestuff.

1 SCOPE

1.1 This method is intended for the determination of strength of reactive dyes (dichlorotriazinyl type) by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Prepare dyeings of the standard sample of dyestuff (see 3.1) by following the procedure given in Annex A; prepare simultaneously additional dyeings of the standard sample, with the percentage variations of dyeing strength by 5 percent on the lower side of the recommended percentage.

5.2 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex A.

5.3 Compare the dyeings obtained in 5.2 and 5.1. Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentage of the dyeing which matches exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a thickness to avoid the effects of other backing on the appearance. The hanks should be compared, if possible, in North skylight. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

6 CALCULATIONS

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test.

7 REPORT

7.1 The report shall include strength of dyestuff in percent.

ANNEX A

METHOD FOR DYEING REACTIVE DYES (DICHLOROTRIAZINYL TYPE)

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel beakers or dye vessels provided with mechanically agitated dye baths.

A-1.2 Graduated Pipettes

capable of measuring correct to 0.1 ml.

A-1.3 Volumetric Flask

calibrated to 500 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Chloride Solution or Anhydrous Sodium Sulphate Solution

20 percent (m/v).

NOTE — Quality of sodium sulphate is extremely important. Commercial brands may be acidic or alkaline. Therefore, they shall be neutralized.

A-2.3 Sodium Carbonate Solution

10 percent (m/v).

A-2.4 Soap Solution

containing 0.3 percent (m/v) of neutral detergent.

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, bleached, unmercerised cotton yarn, having no finishing chemicals or blueing agent, shall be used in this test. Each hank should weigh 10 ± 0.1 g (see Note 2).

NOTES

1 Any yarn normally used in the laboratories for carrying out trial or yarn of the following requirements is suitable for this test:

- a) Count — 10 tex \times 2 or (60s/2),
- b) Twist per metre — 750, and
- c) Cuprammonium fluidity not more than 5 rhes.

2 If the mass of the hank is not 10 ± 0.1 g, then it should be weighed accurately, and the amount of dyestuff and the chemicals to be taken should be calculated accordingly.

A-3.2 Preparation of the Test Hanks

The hanks shall be treated in boiling water for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of the Dyestuff Solutions

Weigh accurately 1.0 g of the dyestuff under test. Paste the dyestuff with cold water and dissolve by adding warm water (not higher than 50°C). The total volume of water used for dissolution of dyestuff should not exceed 100 ml. Dilute the solution with cold water and make up to 500 ml in a volumetric flask.

A-4.1.1 Similarly, prepare a solution of the standard dyestuff by following the procedure given in A-4.1 but taking the standard dyestuff instead of the dyestuff under test.

A-4.2 Dyeing (for 10 ± 0.1 g Hank)

A-4.2.1 Pipette out separately the required amount of the solution of dyestuff under test in the dye vessels so as to give 1.9, 2.0 and 2.1 percent depth. Add the requisite quantity of water and 60 ml of sodium chloride solution or anhydrous sodium sulphate solution to make the volume of the dye, liquor to 300 ml, leaving sufficient margin for the addition of alkali. Heat the dye bath to 35°C, stir the dye liquor and enter wetted hanks. Turn the hanks frequently so as to obtain level dyeings and dye for 30 minutes. Add 12 ml of anhydrous sodium carbonate solution and dye for further 30 minutes. Remove the dyeings and squeeze the dyed hanks evenly. Rinse the dyeings first in cold water and then in hot water. Treat the dyed hanks at boil for 15 minutes in a soap solution at liquor ratio of 1 : 30; rinse in cold water and again treat the hanks at boil for 15 minutes in soap solution at liquor ratio of 1 : 30. Finally, rinse the dyeings in cold water and dry.

A-4.2.2 Similarly, pipette out separately required amounts of solution of the standard dyestuff in separate dye vessels (see Note). Dye the hanks by following the method given in A-4.2.1.

NOTE — The dyeings with the solution of the dyestuff under test and of the standard dyestuff should be done simultaneously in the same water bath.

EVALUATION OF STRENGTH OF REACTIVE DYES (VINYL SULPHONE TYPE) BY DYEING TEST

(Source : IS 7842 : 1975)

Reactive dyes are marketed in different strengths. This method for determining the strength of vinyl sulphone type reactive dyestuffs against a mutually accepted standard would be

useful for assessing the comparative strength of dyestuffs only. This methods may not necessarily be the most economical method of dyeing.

1 SCOPE

1.1 This method is intended for the determination of strength of reactive dyes (vinyl sulphone type) by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantites of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Prepare dyeings of the standard sample of dyestuff (see 3.1) by following the procedure given in Annex A; prepare simultaneously additional dyeings of the standard sample, with the percentage variations of dyeing strength by 5 percent on the lower side of the recommended percentage.

5.2 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex A.

5.3 Compare the dyeing obtained in 5.2 and 5.1 (see Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentage of the dyeing which matches exactly.

NOTE — Before comparing the dyeings, they shall be spread out properly. The dried hanks shall be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of backing on the appearance. The hanks shall be compared, if possible, in North skylight. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test shall be observed. If the strength variations between the two consecutive dyeings are not constant; the dyeings shall be repeated. If dyeings of the test sample and the standard do not fall within the range, fresh set of dyeings shall be taken.

6 CALCULATION

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test which matches with A .

7 REPORT

7.1 The report shall include the following information:

- Type of dye, and
- Strength of the dyestuff in percent.

ANNEX A

METHOD FOR DYEING REACTIVE DYES (VINYL SULPHONE TYPE)

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel dye vessels.

A-1.2 Graduated Pipettes

capable of measuring correct to 0.1 ml.

A-1.3 Volumetric Flask

calibrated to 500 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the dye bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Sulphate (Calcined) Solution

20 percent (*m/v*).

NOTE — Quality of sodium sulphate solution is extremely important. Commercial brands may be acidic or alkaline therefore, they shall be neutralized.

A-2.3 Sodium Carbonate Solution

10 percent (*m/v*).

A-2.4 Trisodium Phosphate Solution

10 percent (*m/v*).

A-2.5 Soap Solution

containing 0.3 percent (*m/v*) of neutral detergent.

A-2.6 Acetic Acid Solution

1 percent (*v/v*).

A-3 PREPARATION OF THE HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, bleached, unmercerised cotton yarn (*see* Note 1), having no finishing chemicals or blueing agent, shall be used in this test. Each hank shall weigh 10 ± 0.1 g (*see* Note 2).

NOTES

1 Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

- Count — 10 tex \times 2 (or 60s/2),
- Twist per metre — 750, and
- Cuprammonium fluidity not more than 5 rhes.

2 If the mass of the hank is not 10 ± 0.1 g then it shall be weighed accurately, and the amount

of dyestuff and the chemicals to be taken shall be calculated accordingly.

A-3.2 Preparation of the Test Hanks

Treat the hanks in boiling water for 10 minutes, squeeze evenly to contain approximately its own mass of water, cool and enter into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of the Dyestuff Solutions

Weigh accurately 1.0 g of the dyestuff under test. Paste the dyestuff with cold water and dissolve by adding hot water (not higher than 50°C). The total volume of water used for dissolution of dyestuff shall not exceed 100 ml. Dilute the solution with cold water and make up to 500 ml in a volumetric flask.

A-4.1.1 Similarly, prepare a solution of the standard dyestuff by following the procedure given in A-4.1 but taking the standard dyestuff instead of the dyestuff under test.

A-4.2 Dyeing (for 10 ± 0.1 g Hank)

A-4.2.1 Pipette out separately the required amount of solution of standard dyestuff in the dye vessels so as to give 1.9, 2.0 and 2.1 percent depth. Add the requisite quantity of water and 75 ml sodium sulphate (calcined) solution to make the volume of the dye liquor 300 ml (liquor ratio of 1 : 30) leaving sufficient margin for the addition of alkali. Set the dye bath at room temperature, stir the dye liquor and enter the wetted hanks. Turn the hanks frequently so as to obtain level dyeings. Raise temperature of the dye bath to 40°C within 10 minutes. Then add 30 ml trisodium phosphate solution to the dye bath and dye for further 90 minutes at 40°C (*see* Note). Remove the dyeings and squeeze the dyed hanks evenly. Rinse the dyeings in cold water, treat with acetic acid, rinse in cold water and then in hot water. Treat the dyed hanks at boil for 15 minutes in a soap solution at liquor ratio of 1 : 30. Rinse in cold water and again treat the hanks at boil for 15 minutes in soap solution at liquor ratio of 1 : 30. Finally, rinse the dyeings in cold water and dry.

NOTE — 1 or Reactive Blue 21 and similar products, dyeing shall be done at 80°C for 60 minutes after addition of 15 ml sodium carbonate solution.

A-4.2.2 Similarly, pipette out separately required amounts of solution of the dyestuff under test in separate dye vessels (*see* Note). Dye the hanks by following the method given in A-4.2.1.

NOTE — The dyeings with the solutions of the dyestuff under test and of the standard dyestuff should be done simultaneously in the same water bath.

EVALUATION OF STRENGTH OF REACTIVE DYES (TRICHLOROPYRIMIDYL TYPE) BY DYEING TEST

(Source : IS 7845 : 1975)

Reactive dyes are marketed in different strengths. This method for determining the strength of trichloropyrimidyl type (reactone/ drimarene) dyestuffs against a mutually accepted

standard would be useful for assessing the comparative strength of dyestuffs only. This method may not necessarily be the most economical method of dyeing.

1 SCOPE

1.1 This method is intended for the determination of strength of reactive dyes (trichloropyrimidyl type) by dyeing test.

2 PREPARATION OF TEST SAMPLE

2.1 From each container selected, draw small quantities of dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite test sample weighing about 20 g.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 EVALUATION OF STRENGTH OF DYESTUFF

5.1 Prepare dyeings of the standard sample of dyestuff (see 3.1) by following the procedure given in Annex A; prepare simultaneously additional dyeings of the standard sample, with the percentage variations of dyeing strength by 5 percent on the lower side of the recommended percentage.

5.2 Simultaneously, prepare dyeings of different percentages of the dyestuff under test by following the procedure given in Annex A.

5.3 Compare the dyeings obtained in 5.2 and 5.1 (see Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentage of the dyeing which matches exactly.

NOTE — Before comparing the dyeings, they shall be spread out properly. The dried hanks shall be laid side by side in the same plane and oriented in the same direction. They shall be combed to a uniform thickness to avoid the effects of backing on the appearance. The hanks shall be compared, if possible, in North skylight. The consistency in strength variation of different dyeing of standard dyestuff and the dyestuff under test shall be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings shall be repeated. If the dyeings of the test sample and the standard do not fall within the range, fresh set of dyeings shall be taken.

6 CALCULATION

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff in percent,

A = percentage dyeing of the standard dyestuff, and

B = percentage dyeing of the dyestuff under test which matches with A .

7 REPORT

7.1 The report shall include the strength of dyestuff in percent.

ANNEX A

METHOD FOR DYEING REACTIVE DYES (TRICHLOROPYRIMIDYL TYPE)

A-1 APPARATUS

A-1.1 Dye Vessels

porcelain or stainless steel vessels.

A-1.2 Graduated Pipettes

capable of measuring correct to 0.1 ml.

A-1.3 Volumetric Flask

calibrated to 500 ml.

A-2 DYEING ASSISTANTS

A-2.1 Water

Distilled water shall be used in the preparation of the bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm, expressed as calcium carbonate, may be used.

A-2.2 Sodium Sulphate (Calcined) Solution

20 percent (*m/v*).

NOTE — Quality of sodium sulphate solution is extremely important. Commercial brands may be acidic or alkaline. Therefore, they shall be neutralized.

A-2.3 Sodium Carbonate Solution

10 percent (*m/v*).

A-2.4 Mild Oxidant Solution

such as *m*-nitrobenzene sulphonic acid sodium salt, 10 percent (*m/v*).

A-2.5 Soap Solution

containing 0.3 percent (*m/v*) of neutral detergent.

A-3 PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, bleached, unmercerized cotton yarn (*see* Note 1) having no finishing chemicals or blueing agent shall be used in this test. Each hank shall weigh 10.0 ± 0.1 g (*see* Note 2).

NOTES

1 Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test:

- Count — 10 tex \times 2 or (60 \times 2),
- Twist per metre — 750, and
- Cuprammonium fluidity not more than 5 rhes.

2 If the mass of the hanks is not 10.0 ± 0.1 g, then it shall be weighed accurately, and the amount of dyestuff and chemicals to be taken should be calculated accordingly.

A-3.2 Preparation of the Test Hanks

Treat the hanks in boiling water for 10 minutes,

squeeze evenly to contain approximately their own mass of water, cool and enter into the dye bath.

A-4 PROCEDURE

A-4.1 Preparation of the Dyestuff Solution

Weigh accurately 1.0 g of the standard dyestuff. Paste the dyestuff with cold water and dissolve by adding warm water (not higher than 50°C.) The total volume of water used for dissolution of the dyestuff shall not exceed 100 ml. Dilute the solution with cold water and make up to 500 ml in a volumetric flask.

A-4.1.1 Similarly prepare a solution of the dyestuff under test by following the procedure given in A-4.1.

A-4.2 Dyeing (for 10.0 ± 0.1 g Hanks)

A-4.2.1 Pipette out separately the required amount of the solution of the standard dyestuff in the dye vessels so as to give 1.9, 2.0 and 2.1 percent depth. Add the requisite quantity of water, 30 ml of sodium sulphate (calcined) solution, 3 ml of mild oxidant solution to make the volume of the dye liquor 300 ml (liquor ratio of 1 : 30) leaving sufficient margin for the addition of alkali and remaining salt solution. Set the dye bath at 40°C, stir the dye liquor and enter the wetted hanks and keep for 5 minutes. Turn the hanks frequently so as to obtain level dyeings. Add 60 ml of sodium carbonate solution and raise the temperature from 40 to 70°C in 15 minutes turning the hanks at regular intervals. Add 30 ml sodium sulphate (calcined) solution and raise the temperature from 70 to 95°C in 15 minutes. Add further 30 ml of sodium sulphate (calcined) solution and maintain the temperature at 95°C for 1 hour, turning the hanks frequently to obtain level dyeings.

Remove the dyeings and squeeze the dyed hanks evenly. Rinse the dyed hanks first in cold water and then in hot water. Treat the dyed hanks at boil for 15 minutes in soap solution at liquor ratio of 1 : 30; rinse in cold water and again treat the hanks at boil for 15 minutes in soap solution at liquor ratio of 1 : 30. Finally, rinse the dyeings in cold water and dry.

A-4.2.2 Similarly, pipette out separately required amounts of the solution of the dyestuff under test in separate dye vessels to give required depth. Dye the hanks by following the method given in A-4.2.1.

NOTE — The dyeings with the solutions of the dyestuff under test and of the standard dyestuff should be done simultaneously in the same water bath.

QUANTITATIVE ESTIMATION OF CATIONIC DYE-FIXING AGENTS

(Source : IS 11660 : 1986)

Cationic dye-fixing agents are used for after-treatment of direct or reactive dyeings to minimize the bleeding trouble. Many such agents available in the market are of different chemical nature and may vary in active matter content. Assessment of active matter content is important to the manufacturer to standardize the product batches as well as to the user to evaluate cost-benefit ratio.

A number of methods are available for determining the active matter content. However, the method suggested should neither be too complicated nor be unsuitable for cationic compounds

under consideration. Determination of solid content is not always a valid indication of active matter content as it can be adjusted by adding some other inactive, cheaper and compatible ingredient.

The method prescribed in this standard is based on titration of known volume of direct dye solution (Congo red) against solution of the agent and comparing it with titration of cetrimide solution as a standard solution. The method is useful in estimating accurately the extent of absorption of dye-fixing agents by cotton under different conditions.

1 SCOPE

1.1 This method is intended for the comparative estimation of active matter content of cationic dye-fixing agents. The lowest concentration of a commercial sample which can be estimated by this method is 0.2 ml/l.

2 PRINCIPLE

2.1 A known volume of dye (Congo red) solution is titrated against the solution of cationic dye-fixing agent, the end point being indicated by complete precipitation of the dye. The volume of agent thus required is compared with that for a solution of known strength of standard solution of cetrimide. Results are expressed as equivalent of cetrimide in the stock solution of the dye-fixing agent under test.

3 PREPARATION OF TEST SAMPLE

3.1 Stir the contents of the containers selected thoroughly for not less than five minutes with a clean stick to make them homogeneous. Draw a sample of about 10 g from each selected container in a clean bottle and mix thoroughly. This shall constitute the test sample. The test sample should be stirred again with a clean glass rod before testing.

4 STANDARD AGENT SOLUTION

4.1 For the purpose of comparative estimation of dye-fixing agent under test, a standard solution (4 g/litre) of cetrimide, prepared by dissolving 4 g of cetrimide (100 percent solid content) in 1 000 ml of distilled water, shall be used.

5 APPARATUS

5.1 Titration Flask

100 ml capacity.

5.2 Graduated Burette

with an accuracy of 0.1 ml.

5.3 Pipette

20 ml capacity.

5.4 Weighing Balance

with an accuracy of 1 mg.

5.5 Whatman Filter Paper No. 41 or Its Equivalent

5.6 Glass Rod

for spotting and stirring purposes.

6 REAGENTS

6.1 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6.2 Congo Red Direct Dye

6.3 Cetrimide with Known Strength (Solid Content)

7 PROCEDURE

7.1 Prepare 2 g/l Congo red dye solution by dissolving accurately weighed 2 g dye in 50 ml of water and then making the final volume up to 1 000 ml.

7.2 Prepare 4 ml/litre solution of the cationic dye-fixing agent under test by dissolving 4 ml of stock solution of the agent under test in 50 ml water and making the final volume up to 1 000 ml. Fill this solution in the burette up to the mark.

7.3 Pipette out 20 ml of Congo red solution as prepared in 7.1 in the titrating flask.

7.4 Titrate the solution of the agent under test as prepared in 7.2 against Congo red solution (7.3) till the end point is reached. End point is indicated by complete precipitation of dye present in the flask when, by spotting a drop of solution on filter paper, dye appears as small specks in the spot with no coloured ring around. Note the volume of the solution of the agent under test required to achieve the end point.

7.5 Prepare 4 g/litre solution of standard cetrimide by dissolving x g of it in 50 ml distilled water (see Note) and making the final volume up to 1000 ml. Titrate this solution against 2 g/litre Congo red solution similar to the procedure given in 7.4 till the end point is reached. Note the volume of 4 g/litre cetrimide solution required to achieve the end point.

NOTE — The value of x can be calculated as follows:

$$x = \frac{4 \times 100}{y}$$

where

y = solid content of the cetrimide used.

8 CALCULATIONS

8.1 Calculate the active matter content in the stock solution of the cationic dye-fixing agent under test in terms of equivalent of cetrimide by the following formula:

$$C_a = \frac{V_c}{V_a}$$

where

C_a = active matter content of the stock solutions of the agent under test as equivalent of cetrimide,

V_c = volume of the standard cetrimide solution (4 g/litre) required to achieve the end point, and

V_a = volume of the solution (4 ml/litre) of the agent under test required to achieve the end point.

9 REPORT

9.1 Report the active matter content in the stock solution of the agent under test in terms of equivalent of cetrimide.

DETERMINATION OF STRENGTH OF BASIC DYES

(Source : IS 12387 : 1988)

Basic dyes are generally marked by brilliance of shade and high tinctorial value. They can be applied on cellulosic fibres with the help of mordants. They are also used for dyeing and printing of protein fibres (wool, silk, etc) and polyacrylonitrile fibres as well as for topping of cellulosic materials dyed with direct dyes.

Since the range of basic dyes for textile use is very wide and the quality of these dyes differs from manufacturer to manufacturer, it is very essential to know their strength so as to minimize cost-benefit ratio. This will also help to adjust recipes for dyeing and printing which

minimize variation in dyeing from one lot to the other and reduces chances of rejection of the dyed or printed material. Method 1 prescribed in this standard is based on the comparison of transmission of the solution of dyes under test with that of the standard dye whose strength is assumed to be 100 percent. It is suitable for dye solutions which do not scatter light and which obey the Bauger-Lambert-Beer Law. Method 2 is based on the reduction of the dye with titanium trichloride and then calculating the strength of dye by the amount of titanium trichloride used up in the reduction.

1 SCOPE

1.1 This standard prescribes two methods for the determination of strength of basic dyes.

2 TERMINOLOGY

2.1 Depth of Shade

the amount of dyestuff in g in the dye bath per 100 g of the fibre, expressed as a percentage.

2.2 Strength of Dyestuff

the percentage ratio of the strength of the dyestuff under test to that of the standard dyestuff, the strength of which is assumed to be 100 percent.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5 METHOD 1

5.1 Apparatus

5.1.1 Suitable Colorimeter

capable of measuring transmission from 400 to 700 nm.

5.1.2 Volumetric Flask

250 ml capacity.

5.1.3 Weighing Balance

with an accuracy of 1 mg.

5.1.4 Watch Glass

for weighing the dye.

5.1.5 Glass Rod

for preparing dye solution.

5.1.6 Pipette

with an accuracy of 0.05 ml.

5.2 Reagent

5.2.1 Methylated Spirit

5.3 Procedure

5.3.1 Weigh accurately 250 mg of the standard dye (see 3.1). Paste the weighed amount of dye with 5-10 ml of methylated spirit in a volumetric flask. Pour 50 ml of boiling water with constant stirring. Cool the contents and make the solution up to 250 ml. Shake the flask thoroughly so as to homogenize the contents.

5.3.2 Pipette out 0.1, 0.2, 0.3, 0.4 and 0.5 ml of the solution prepared in 5.3.1 in separate test tubes and make the total volume in each test tube to 100 ml before measurement.

5.3.3 Calculate the concentration C_a in g/litre of each dye solution by the formula:

$$C_a = \frac{X}{100}$$

where X is the volume of dye solution taken (see 5.3.2).

5.3.4 Measure the transmission of the dye solutions prepared in 5.3.2 on the colorimeter at wavelengths of minimum transmission (maximum absorption) and then record the transmission of all the dye solutions prepared in 5.3.2 at this selected wavelength.

5.3.5 From values of transmission of dye solutions obtained in 5.3.4, calculate the optical density of each solution separately by the formula:

$$\text{Optical density} = \log \left(\frac{1}{T} \right)$$

where T is the percent transmission.

5.3.6 Plot a graph between the concentration of dye solution (C_s) along X-axis against the optical density along Y-axis, which shall be a straight line.

5.3.7 Carry out the procedure given from 5.3.1 to 5.3.6 for the dyestuff under test.

5.3.8 Record the concentration (C_u) of each solution of dyestuff under test from the graph prepared in 5.3.6 against their optical densities.

5.3.9 Calculate the strength (S) of dyestuff under test by the formula:

$$S = \frac{C_s}{C_u}$$

where

C_s = concentration of solution of standard dyestuffs, and

C_u = concentration of solution of dyestuff under test having same optical density as that of standard dyestuff solution.

5.3.10 Calculate the average of all the values obtained in 5.3.9 and round off the result to the nearest whole number.

5.3.11 If the absolute content of the standard dyestuff is known, then calculate the percentage absolute strength S_a of dyestuff under test by the formula:

$$S_a = \frac{C_s \times B}{C_u}$$

where

C_s = concentration of a solution of standard dyestuff,

B = absolute content of the standard dyestuff in percentage, and

C_u = concentration of a solution of dyestuff under test having the same optical density as that of standard dyestuff solution.

5.3.12 For quick determination of strength of dyestuff under test with respect to that of standard dyestuff, prepare similar solutions of both the dyestuffs under similar conditions and measure their optical densities as described in 5.3.4 and 5.3.5. Then calculate the strength of dyestuff under test (S) with respect to that of the standard dyestuff by the formula:

$$S = \frac{D_1 \times Y}{D}$$

where

D_1 = optical density of the solution of standard dyestuff,

Y = nominal strength of the standard dyestuff, and

D = optical density of the solution of the dyestuff under test.

NOTE — The arithmetic mean of two parallel determinations, permissible difference between which shall not exceed 4 percent at confidence level of 95 percent shall be taken as the test result.

6 METHOD 2

6.1 Apparatus

6.1.1 Titanium Trichloride Storage Bottle

See Fig. 1.

6.1.2 Reduction Flask

See Fig. 2.

6.2 Reagents

6.2.1 Hydrochloric Acid

(a) concentrated and (b) 25 percent (m/v).

6.2.2 Ammonium Thiocyanate Solution

20 percent (m/v).

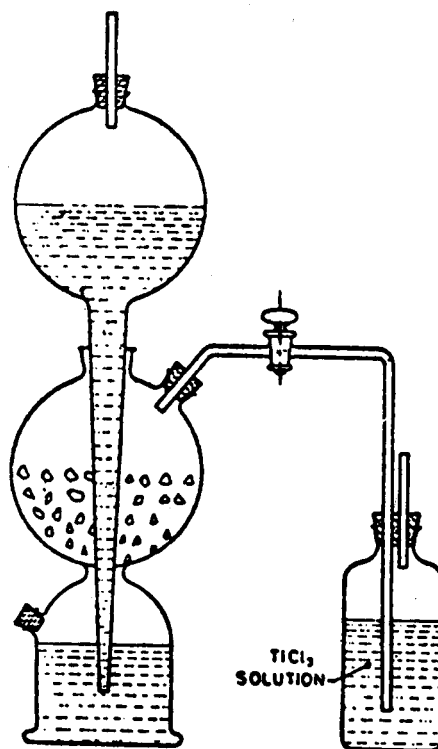


FIG. 1 $TiCl_3$ SOLUTION STORAGE BOTTLE (PAINTED BLACK) WITH KIPP'S APPARATUS FOR CO_2 GAS

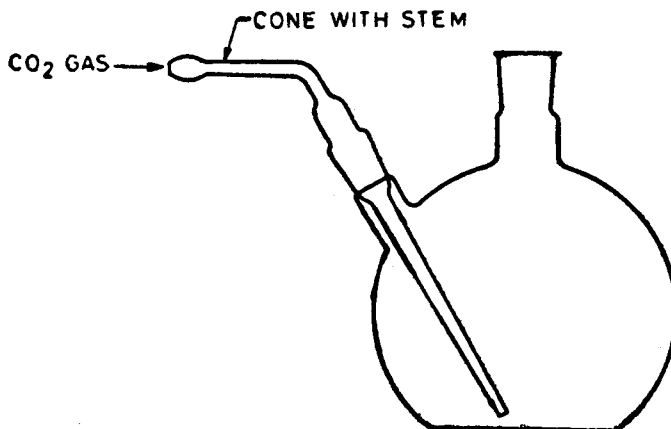


FIG. 2 500-ml REDUCTION FLASK (TWO NECKS, SHORT CENTRE NECK AND SIDE NECK AT AN ANGLE)

6.2.3 Titanium Trichloride Solution

approximately 0.025 N, prepared as follows:

Prepare a 15 percent (*m/v*) solution of titanium trichloride. Take 30 ml of solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of one litre of water and 60 ml of concentrated hydrochloric acid. Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen, for about 15 minutes. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus. The solution should be stored in a bottle painted black to protect it from light.

6.2.4 Stannous Chloride Solution

prepared by dissolving 30 g of stannous chloride in 100 ml of concentrated hydrochloric acid and diluting it to 200 ml with water. Keep the solution in air-tight bottle.

NOTE — This solution should be prepared fresh after one week.

6.2.5 Mercuric Chloride Solution

prepared by dissolving 5 g of mercuric chloride in 100 ml of water and filtered, if necessary.

6.2.6 Diphenylamine Solution

prepared by melting 1 g of diphenylamine and adding to this 100 ml of concentrated sulphuric acid. Dissolve diphenylamine by stirring.

6.2.7 Sulphuric Acid

1 N.

6.2.8 o-Phosphoric Acid

free from iron.

6.2.9 Potassium Dichromate Solution

0.1 N.

6.2.10 Ferric Ammonium Sulphate Solution

approximately 0.025 N, prepared as follows:

Dissolve 9.803 g of pure ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 100 ml of water. Add 10 ml of concentrated sulphuric acid and mix well. Dissolve 0.70 g of potassium permanganate in 100 ml of water. Add this solution to the solution of ferrous ammonium sulphate with stirring. At the end, add the permanganate solution carefully so that a ferrous salt is just oxidized. Cool the solution and dilute it to one litre. Determine the normality of the solution by the method given in Annex A.

6.3 Procedure

6.3.1 Weigh accurately 1 g of dyestuff from the test sample. Dissolve it in hot water. Pour the solution into a 500 ml graduated flask and make the volume to 500 ml. Mix well.

6.3.2 Pipette 50 ml of the solution into a 500 ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml of water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

6.3.3 Pass carbon dioxide in the storage bottle containing titanium trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously. Add this solution to the dye solution (*see 6.2.2*). Immediately boil the contents of the flask for complete reduction (*see Note 1*). Cool the flask in ice-bath. Maintain the continuous flow of carbon dioxide throughout the test. Add 10 ml of 20 percent ammonium thiocyanate indicator solution. Titrate the mixture against 0.025 N ferric ammonium sulphate solution to a permanent change of colour. Note the reading of ferric ammonium sulphate solution (*see Note 2*).

NOTES

1 The reduction time for various dyes ranges from 8 to 15 minutes.

2 The volume of ferric ammonium sulphate required for titration should not be less than 10 ml.

6.3.4 Carry out a blank by following the procedure given as above using 50 ml of water in place of dye solution.

6.3.5 Calculate the titanium value, in percent, of the dye by the following formula:

$$T = \frac{(B - A) \times N \times M \times V}{E \times C \times W \times 10}$$

where

T = titanium value in percent of the dye,

B = volume in ml of ferric ammonium sulphate solution required for blank (see 6.3.4),

A = volume in ml of ferric ammonium sulphate solution required for the dye (see 6.3.3),

N = normality of ferric ammonium sulphate solution (see A-1.3),

M = molecular mass of dye,

V = volume of dye solution (see 6.3.1),

E = number of hydrogen atoms consumed per molecule of dye (see Note),

C = volume of dye solution (see 6.3.1), and

W = mass in g of the sample taken for test.

NOTE — Each azo group requires 4, nitro group 6 and hydroazo 2 atoms of hydrogen for reduction.

6.3.6 Repeat the test prescribed in 6.3.3 twice and calculate the titanium value, in percent, of the dye.

7 REPORT

7.1 Report the value obtained in 5.3.10 or 5.3.11 or 5.3.12 and 6.3.5 as strength of dye-stuff under test. Report also the method used, either Method 1 or Method 2.

8 PREPARATION OF TEST SAMPLE

8.1 If the dye is marketed in powder form, draw from each container selected, a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

8.2 For dyes marketed in liquid, emulsion or paste form, shake each container selected thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 8.1.

ANNEX A

DETERMINATION OF NORMALITY OF FERRIC AMMONIUM SULPHATE

A-1 PROCEDURE

A-1.1 Pipette 100 ml of ferric ammonium sulphate solution (see 6.2.10) into a 500 ml conical flask and add 5 ml of concentrated hydrochloric acid. Heat it to about 85°C. Add stannous chloride solution dropwise from a burette while swirling the flask over a white background until the yellow colour of the solution has nearly disappeared. Complete the reduction by adding dilute solution of stannous chloride (see Note 1) dropwise with agitation after each addition until the liquid has a faint green colour free from any tinge of yellow. Dilute the solution in the flask to thrice its volume with water. Rapidly cool the solution under the tap with stopper on and remove the excess of stannous chloride by adding 10 ml of mercuric chloride solution rapidly in one lot. A slight silky white precipitate of mercurous chloride is obtained (see Note 2). Add 50 ml of 1 N sulphuric acid, 2.5 ml of *o*-phosphoric acid and three drops of diphenylamine solution as indicator. Titrate slowly against 0.1 N potassium dichromate solution while stirring constantly to the first permanent tinge of purple or violet blue colouration.

NOTES

1 Dilute stannous chloride solution should be prepared by diluting stannous chloride solution (see 6.2.4) with two volumes of 10 percent hydrochloric acid.

2 Excess of stannous chloride solution should not be added. A heavy, grey or black precipitate is the indication of too much stannous chloride solution. Discard the solution and start with a fresh solution of ferric ammonium sulphate.

A-1.2 Carry out a blank by following the procedure prescribed in A-1.1 but by using 100 ml of water in place of 100 ml of ferric ammonium sulphate solution.

A-1.3 Calculate the normality of ferric ammonium sulphate solution by the formula given below:

$$N = \frac{(A - B) \times N_1}{100}$$

where

N = normality of ferric ammonium sulphate solution,

A = volume in ml of potassium dichromate solution required to titrate the solution (see A-1.1),

B = volume in ml of potassium dichromate solution required to titrate the blank (see A-1.2), and

N_1 = normality of potassium dichromate solution.

DETERMINATION OF STRENGTH OF DISPERSE DYES FOR POLYESTER BY SCREEN PRINTING

(Source : IS 11635 : 1986)

The method prescribed in this standard is a general method and, wherever special instructions are given by the manufacturers, these should be followed while carrying out screen printing using the dyestuff. In order to compare the strength,

and shade of the dyestuff under test and the standard dyestuff, screen printing on 67/33 polyester/cotton fabric is recommended in two standard depths (see Annex A).

1 SCOPE

1.1 This method is intended for evaluating the strength and shade of disperse dyes for polyester by hand screen printing.

2 PREPARATION OF TEST SAMPLE

2.1 If the dye is marketed in powder form, draw from each container selected, a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

2.2 For dyes marketed in liquid emulsion or paste form, shake each container selected thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 2.1.

3 STANDARD DYESTUFF

3.1 The standard dyestuff, against which the strength of the dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 APPARATUS

4.1 Laboratory Scale Screen Printing Table Together with a Screen and Squeegee to Give Stripe Design

4.2 Steel Rod

15-20 cm long for stirring purpose.

4.3 Stainless Steel Vessel of 2 Litres Capacity

to prepare stock thickening.

4.4 Glass or Stainless Steel Containers

twelve, each of 500 ml capacity, for preparation of printing paste.

4.5 Steaming Chamber

capable for steaming at 2.1 kg/cm² (30 psi) pressure and with capacity to hold 24 specimens of 20 × 20 cm² size.

4.6 Water Bath

provided with heating arrangement.

4.7 Electric Iron with Energy Regulator

4.8 Weighing Balance

capable of weighing accurately up to 1 mg.

4.9 Weighing Scale

capacity up to 5 kg and capable of weighing accurately up to 1 g.

5 REAGENTS

5.1 Quality of Reagents

5.1.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 A Suitable Thickener

preferably modified guar gum with pH adjusted to 5.5 by adding citric acid or tartaric acid.

5.3 Sodium Chlorate Powder or Aqueous Solution

5.4 Citric Acid

5.5 Sodium Hydroxide Flakes

5.6 Sodium Hydrosulphite Powder (Na₂S₂O₄)

5.7 Sulphuric Acid

70 percent (m/m), specific gravity 1.629 at room temperature.

5.8 A 5-Metre Well Scoured, Bleached and Heat-Set Piece of 67/33 Polyester/Cotton Fabric of Plain Weave

The fabric shall not be treated with optical whitening agents.

NOTE — 100 percent well scoured, bleached and heat-set polyester fabric in plain weave may also be used. In this case, carbonization treatment as given in 6.10 is not required.

5.9 Non-ionic Synthetic Detergent

based on polyethylene glycol.

6 EVALUATION OF STRENGTH OF DYESTUFF

6.0 Procedure

6.1 Prepare stock thickening using following reagents:

- Suitable thickener — 80 parts,
- Sodium chlorate (solid content) — 2 parts,
- Citric acid — 4 parts, and
- Water — to make up to 1 000 parts.

Slowly add the thickener (*see* 5.2), sodium chlorate and citric acid to water. Stir for 15 minutes at room temperature so as to obtain a homogeneous paste.

6.2 Prepare the stock printing paste using following reagents for the standard dyestuff and the dyestuff under test separately:

- Standard disperse dye — 10 g, and
- Warm water (40 to 50°C) — 30 g and stir well.

Add 60 g of stock thickening as prepared in 6.1 so as to make total stock printing paste of the 100 g. For dyestuff under test, prepare stock printing paste of 400 g with 40 g dye, 120 g warm water and 240 g stock thickening.

6.3 Prepare separately the required printing paste for the standard dyestuff for the depths as recommended in Annex A as follows:

Stock printing paste — X parts by mass
where X = recommended depth $\times 10$,

Stock thickening — 35 parts by mass, and

Warm water — To make up to 100 parts by mass and stir thoroughly.

6.4 Take 5 m of 67/33 polyester/cotton fabric and print it with the printing pastes of two recommended depths of standard dyestuff as prepared in 6.3 by hand screen printing (in two strokes in forward and backward direction) on one side of the fabric and mark the printed area for identification.

NOTES

1 The polyester/cotton fabric used should be well desized, scoured, bleached, scoured and heat-set. The pH of the cloth ready for printing should be neutral or slightly acidic and never alkaline. The fabric shall not be finished and treated with optical whitening agent. Before printing, it shall be ironed to remove all creases and folds.

2 The pressure applied on the screen by hand through squeegee should be normal and uniform throughout while printing so as to get uniform and even prints. Excessive pressure should be avoided.

6.5 Prepare suitable quantity of 24 printing pastes in 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5,

5.0, 5.5, 6.0, 6.5, 7.0, 7.5, 8.0, 8.5, 9.0, 9.5, 10.0, 10.5, 11.0, 11.5 and 12 percent depth of dyestuff under test in a similar way as in 6.3 except that the amount of stock printing paste to be taken is calculated by using the following formula:

$$\text{Amount of stock printing paste required} = \frac{\text{Percent depth of printing paste required} \times 10}{100}$$

6.6 Print with printing pastes of different depths as in 6.5 separately similar to the procedure followed in 6.4 side by side on the polyester/cotton fabric (*see* 6.4) and mark the printed areas for identification.

6.7 Dry the printed fabric on screen printing table. Cut 20 \times 20 cm specimens of printing fabric with all the different depths as printed in 6.4 and 6.6, and steam them in the steaming chamber at 2.1 kg/cm² (30 psi) pressure for 30 minutes.

6.8 Treat all the specimens of printed fabric separately after steaming in baths containing 2 g/l sodium hydroxide and 2 g/l sodium hydro-sulphite powder at 60-70°C for 15-20 minutes, and at liquor ratio of 1 : 20.

6.9 Rinse all the specimens well in tap water and dry them on screen printing table.

6.10 Carbonize all the printed specimens separately in baths each containing 70 percent (m/m) sulphuric acid and 10 to 20 g/l of urea or sulphamic acid at room temperature for 30 minutes.

6.11 Rinse the carbonized specimens thoroughly in tap water and wash them separately in 2 g/l suitable detergent (*see* 5.9) at boil for about 20 minutes. Rinse well, dry and iron all the specimens (on the reverse).

6.12 Select two prints of the dyestuff under test which exactly match with the prints of the standard dyestuff. Note the percentage depth of the prints of the dyestuff under test which match with the two prints of recommended depth of the standard dyestuff. Also find out the variation in depths, if any, from the standard depths.

NOTES

1 Before comparison of prints, the specimen should be spread out, ironed properly and laid side by side in the same plane and oriented in the same direction. The consistency in depth variation of different prints of standard dyestuff and the dyestuff under test should be observed. If the depth variations between the two consecutive printings are not constant, the printing should be repeated. Moreover, it should be ensured that prints are uniform and free from printing defects like colour spreading, overlapping of design, etc. Matching should be done on a perfectly white background.

2 The strength of the sample should be expressed on comparisons of printings obtained in 6.11. The variation in shade should be reported in the reduction printings as obtained in 6.11.

7 CALCULATIONS

7.1 Calculate the strength of dyestuff under test for each recommended depth of standard dyestuff by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff under test in percent,

A = depth of printing of the standard dyestuff, and

B = depth of printing of the dyestuff under test which exactly matches with that of the standard dyestuff.

7.2 Calculate the average of the two values of S as obtained in 7.1.

8 REPORT

8.1 Report the value obtained in 7.2 as the strength in percent of the dyestuff under test as compared to the standard dyestuff.

8.1.1 Report also the variation in depth in comparison with the depth of the printing paste of standard dyestuff.

ANNEX A**RECOMMENDED STANDARD DEPTHS FOR EVALUATION OF STRENGTH OF DYESTUFF**

<i>Hue of Dyestuff</i>	<i>Recommended Percent Standard Depth</i>
Black and navy blue	1 and 6
Others	1 and 4

DETERMINATION OF STRENGTH OF DISPERSE DYES FOR POLYESTER BY EXHAUST DYEING

(Source : IS 12149 : 1987)

Disperse dyes are marketed in a large number of varieties and even as mixture in powder or paste form with different strengths. These dyes are mainly used for colouring hydrophobic fibres, such as cellulose acetate, triacetate, polyamide, polyester and polyacrylonitrile. These have become especially important for polyester fibre which is widely used as a component of blends with cotton, wool or any other man-made fibre. The method laid down in this standard for determining the strength of dyestuff against a mutually

accepted standard would be useful for assessing both strength and shade of disperse dyes for polyester.

The method prescribed in this standard is a general method and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. In order to compare the strength of the dyestuff under test and the standard dyestuff, dyeings of two standard depths are recommended as given in Annex A.

1 SCOPE

1.1 This method is intended for evaluating the strength and shade of disperse dyes for polyester by exhaust dyeing.

2 PREPARATION OF TEST SAMPLE

2.1 If the dye is marketed in powder form, draw from each container selected, a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

2.2 For dyes marketed in liquid, emulsion or paste form, shake each container selected thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 2.1.

3 STANDARD DYESTUFF

3.1 The standard sample of dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 APPARATUS

4.1 High Temperature Bomb/Beaker Dyeing Machine (Laboratory Type)

consisting of a number of bombs/beakers made of thick stainless steel and provided with a rotating mechanism to rotate these beakers/bombs in forward or backward direction in glycerine bath. The machine shall be provided with electric heaters (with cut-off system) to heat the glycerine bath gradually up to $132 \pm 2^\circ\text{C}$ at a rate of 1 to 2°C per minute. The machine should also be provided with a running tap water circulation system to cool the bombs/beakers.

NOTE — Any other machine giving identical and reproducible results may also be used.

4.2 Graduated Pipette

capable of measuring correct to 0.1 ml.

4.3 Volumetric Flask

calibrated to 500 ml with an accuracy up to 1 ml.

4.4 pH Meter

4.5 Weighing Balance

capable of weighing accurately up to 1 mg.

4.6 Comb

for combing the dyed hanks.

4.7 Glass Rod

for preparing dispersion of dye.

5 REAGENTS

5.1 Quality of Reagents

5.1.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 Suitable Dispersing Agent

stable at high temperature up to $132 \pm 2^\circ\text{C}$.

5.3 Acetic Acid

(30 percent *m/v*).

5.4 Sodium Hydroxide Flakes

5.5 Sodium Hydrosulphite Powder

conforming to IS 1919: 1982.

5.6 An Appropriate Number of Hanks

of well scoured, uniform, 100 percent polyester spun yarn from the same lot, each weighing 5.00 ± 0.02 g and free from optical brightening agents.

NOTE — For blended yarns, the hanks may be carbonized before dyeing according to the method given in IS 11220 : 1984.

6 EVALUATION OF STRENGTH OF DYESTUFF

6.1 Procedure

6.1.1 Prepare stock dispersions of the standard dyestuff and the dyestuff under test separately as follows:

Sprinkle x parts of the disperse dyestuff over 15-20 times the quantity of distilled water at 40°C under constant stirring and finally make to the required volume.

NOTE — In general, 1.0 g of the disperse dyestuff is used to prepare stock dispersion and the dispersion is made to 100 ml using distilled water at 40°C which gives 1.0 percent dispersion.

6.1.2 Take the required volume of the stock dispersion of the standard dyestuff according to the depth recommended in Annex A and calculated as given below:

$$\begin{array}{l} \text{Volume of stock} \\ \text{dispersion required} \\ \text{in ml} \end{array} = \frac{\text{recommended} \\ \text{percent depth}}{\text{percent strength} \\ \text{of stock disper-} \\ \text{sion}} \times m$$

where m is the mass of each hank of polyester yarn (see 5.6).

6.1.3 Prepare as follows six dye baths of standard dyestuff, three each for the lower and higher recommended standard depth such that one dye bath has concentration as given in Annex A and the other two differ from this value by 5 percent, one on lower side and the other on higher side. For example, in case of 0.5 percent standard depth, three dye baths of 0.475, 0.500 and 0.525 percent concentration shall be prepared. Add required volume of stock dispersion as in 6.1.2 to the bombs/beakers of high temperature dyeing machine set with 0.5 g/l of dispersing agent and 0.5 g/l of acetic acid (30 percent). Adjust the pH of each dye bath to 5.0 ± 0.2 and make the volume up to 100 ml per dye bath with distilled water to give a liquor ratio of 1 : 20.

6.1.4 Prepare suitable number of dye baths of the dyestuff under test for each standard depth as recommended in Annex A such that each differ in concentration from the other by 5 percent of the value specified in Annex A. For example, in case of 0.5 percent standard depth, prepare required number of dye baths of 0.425,

0.450, 0.475, 0.500, 0.525, 0.550, 0.575 percent concentration, etc, similarly as given in 6.1.3.

6.1.5 Take required number of hanks of well scoured polyester yarn (see 5.6), each weighing 5.00 ± 0.02 g. Treat them in boiling water for 10 minutes, squeeze them evenly to cent percent pick up. Cool the hanks and enter in separate dye baths already prepared in 6.1.3 and 6.1.4. Close the bombs/beakers of the high temperature dyeing machine.

6.1.6 Starch each dyeing at 60°C and raise the temperature to $132 \pm 2^\circ\text{C}$ in about 40 minutes at the rate of 1 to 2°C per minute. Continue dyeing at $132 \pm 2^\circ\text{C}$ for 60 minutes more. Cool the bombs/beakers under tap water on the machine itself and take them out for opening.

6.1.7 Rinse the dyed hanks in distilled water and treat each dyed hank separately in a bath containing 2 g/l sodium hydroxide and 2 g/l sodium hydrosulphite at $60-70^\circ\text{C}$ for 15-20 minutes at liquor ratio of 1 : 20.

6.1.8 Wash the after-treated hanks in distilled water and treat them separately in fresh baths containing 0.5-1 ml/l glacial acetic acid at room temperature for about 15-20 minutes.

6.1.9 Finally, rinse all the hanks thoroughly and dry them in air at a temperature not exceeding 60°C .

6.1.10 Select two dyed hanks of the dyestuff under test which exactly match with the dyed hanks of the standard dyestuff. Note the percentage depths of shade of the dyestuff under test which exactly match with the recommended depths of standard dyestuff (see Notes 1, 2 and 3).

NOTES

1 Before comparing the dyed specimens, it should be ensured that the dyeings are uniform and free from dyeing defects. The uniformly dyed hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of the backing on the appearance. Matching should be done on a white background and in North skylight, if possible.

2 The consistency in strength variations of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variations between the two consecutive dyeings are not constant, the dyeings should be repeated.

3 In case of dispute due to total deviations, spectrophotometer may be used to set right any doubt.

7 CALCULATIONS

7.1 Calculate the strength of dyestuff under test for each recommended depth of standard dyestuff by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff under test in percentage,

A = percentage depth of shade of standard dyestuff, and

B = percentage depth of shade of the dyestuff under test matching with *A*.

7.2 Calculate the average of the two values of *S* obtained in 7.1.

8 REPORT

8.1 Report the value obtained in 7.2 as strength of the dyestuff under test in percentage.

ANNEX A

RECOMMENDED STANDARD DEPTHS FOR EVALUATION OF STRENGTH OF DYESTUFF

<i>Hue of Dyestuff</i>	<i>Recommended Standard Depth of Shade</i> (g of dye/100 g of fibre)
Yellow, orange, scarlet, red and blue (excluding navy blue and turquoise blue)	0·5 and 1·5 or 2·0
Violet	0·3 and 1·0
Turquoise blue	1·0 and 4·0
Navy blue	2·0 and 6·0
Black	2·0 and 8·0

DETERMINATION OF STRENGTH OF DISPERSE ACETATE DYES BY EXHAUST DYEING

(Source : IS 12150 : 1987)

Disperse dyes are marketed in large number of varieties in powder or paste form with different strengths and sometimes even as mixtures also. These dyes are mainly used for colouring hydrophobic fibres, such as cellulose acetate, triacetate, polyester, polyamide and polyacrylonitrile. Disperse acetate dyes are specially used for colouring acetate fibres. With a few exceptions, disperse acetate dyes do not build up well on polyester fibres (they do not produce deep shades on polyester fibres). Further, they are not sufficiently fast to sublimation.

The methods laid down in this standard for determining the strength of dyestuff against a mutually accepted standard would be useful for assessing both strength and shade of disperse dyes for acetate fibre.

The method prescribed in this standard is a general method and, wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings. In order to compare the strength of dyestuff under test and the standard dyestuff, dyeings of two standard depths of shade are recommended (see Annex A).

1 SCOPE

1.1 This method is intended for evaluating the strength of disperse dyes for acetate fibre by exhaust dyeing.

2 PREPARATION OF TEST SAMPLE

2.1 If the dye is marketed in powder form, draw from each container selected, a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

2.2 For dyes marketed in liquid, emulsion or paste form, shake each container selected thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 2.1.

3 STANDARD DYESTUFF

3.1 The standard dyestuff, against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

4 APPARATUS

4.1 High Temperature Bomb/Beaker Dyeing Machine (Laboratory Type)

consisting of a number of bombs/beakers made of thick stainless steel and provided with a rotating mechanism to rotate these beakers/bombs in forward or backward direction in glycerine bath. The machine shall be provided with electric heaters (with cut-off system) to heat the glycerine bath gradually up to $85 \pm 2^\circ\text{C}$ at a rate of 1 to 2°C per minute. The machine should also be provided with a running tap water circulation system to cool the bombs/beakers.

NOTE — Any other machine giving identical and reproducible results may also be used.

4.2 pH Meter

4.3 Glass Rod

for preparing dispersion of dye.

4.4 Graduated Pipette

with an accuracy up to 0.1 ml.

4.5 Graduated Glass Cylinder

500 ml capacity and accuracy up to 1 ml.

4.6 Weighing Balance

with accuracy up to 1 mg.

4.7 Comb

for combing the dyed hanks.

5 REAGENTS

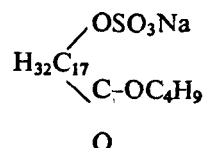
5.1 Quality of Reagents

5.1.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 Suitable Dispersing Agent

may be based on sulphated ricinoleic acid ester such as butyl sulphoricinoleate having the formula:



5.3 Acetic Acid

30 percent (m/v).

5.4 Non-ionic Synthetic Detergent Based on Polyethylene Glycol

5.5 An Appropriate Number of Hanks

an appropriate number of hanks of well scoured, uniform, 100 percent acetate spun yarn from the same lot, each weighing 5.00 ± 0.02 g and free from optical brightening agents.

6 EVALUATION OF STRENGTH OF DYESTUFF

6.1 Procedure

Prepare stock dispersion of the standard dyestuff and the dyestuff under test separately as given below:

Sprinkle 5.0 g of the standard dyestuff over 15-20 times the quantity of distilled water at 40° under constant stirring and make up to 100 ml using distilled water.

Take the required volume of the stock dispersion of the standard dyestuff according to the depth recommended in Annex A and calculated as follows:

$$\text{Volume of stock dispersion (ml)} = \frac{\text{Recommended percent depth}}{5} \times m$$

where m is the mass of each hank of acetate yarn (see 5.5).

Prepare as follows six dye baths of the standard dyestuff, three each for lower and higher recommended standard depths such that one dye bath has concentration as given in Annex A and other two differ from this value by 5 percent, one on lower side and the other on higher side. For example, in case of 0.5 percent standard depth, three dye baths of 0.475, 0.500 and 0.525 percent concentration shall be prepared.

Add the required volume of dye dispersion for the depths of shades as recommended in Annex A in the bombs/beakers already set with 0.5-1 g/l of dispersing agent in 50 ml of distilled water at 40°C. Add 1 ml of acetic acid (30 percent) to adjust the pH of each dye bath to 6.0-6.5. Finally make the total volume of dye liquor to 100 ml by adding distilled water at 40°C to give a liquor ratio of 1 : 20.

Prepare suitable number of dye baths of the dyestuff under test for each standard depth as recommended in Annex A such that each differ in concentration from the other by 5 percent of the value specified in Annex A. For example, in case of 0.5 percent standard depth, prepare required number of dye baths of 0.425,

0.450, 0.475, 0.500, 0.525, 0.550 and 0.575 percent concentration, etc, similarly as given in 6.1.3.

Take required number of hanks of well scoured acetate yarn (see 5.5) and treat them in hot water at 60°C for 10 minutes. Squeeze them individually so that pick up for each hank is approximately cent percent.

NOTE — The hanks should have neutral or slightly acidic pH but never alkaline.

Cool all the hanks and enter in separate dye baths already prepared, and close the bombs/beakers. Start each dyeing at 40°C and raise the temperature gradually to 85°C in about 35-40 minutes at the rate of 1 to 2°C per minute.

Continue dyeing at 85°C for 60 minutes more. After dyeing, rinse the hanks thoroughly in distilled water.

Wash each hank separately in bombs/beakers containing 2 g/l non-ionic synthetic detergent at 40°C for about 10 minutes at liquor ratio of 1 : 20 and then rinse them thoroughly in water and dry them in air at a temperature not exceeding 60°C.

Compare the dyeings of the dye under test with those of the standard dyestuff and select dyed hanks of dye under test which exactly match with those of the standard dyestuff. Note the percentage depths of dyestuff under test which exactly match with those of the standard dyestuff (see Notes 1, 2 and 3).

NOTES

1 The dyed hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of the backing on the appearance.

Matching should be done on a white background in North skylight, if possible. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variation between the two consecutive dyeings are not constant, the dyeings should be repeated. In all cases, ± 5 percent deviation in dye strength is taken for accurate strength assessment in the dye trials.

2 Before comparing the dyed specimens, it should be ensured that the dyeings are uniform and free from dyeing defects, otherwise dyeings should be repeated.

3 In case of dispute due to total deviations, spectrophotometer may be used to set right any doubt.

7 CALCULATIONS

7.1 Calculate the strength of dyestuff under test for both recommended depths of standard dyestuff by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of dyestuff under test in percentage,

A = percentage depth of shade of standard dyestuff, and

B = percentage depth of shade of the dyestuff under test matching with A .

7.2 Find out the average of the two values of S obtained in 7.1.

8 REPORT

8.1 Report the value obtained in 7.2 as strength of the dyestuff under test in percentage.

ANNEX A

RECOMMENDED STANDARD DEPTHS FOR EVALUATING STRENGTH OF DYESTUFF

<i>Hue of Dyestuff</i>	<i>Recommended Standard Depth of Shade</i> (g of dye/100 g of fibre)
Yellow, orange, scarlet red and blue (excluding navy blue and turquoise blue)	0.5 and 1.5 or 2.0
Violet	0.3 and 1.0
Turquoise blue	1.0 and 4.0
Navy blue	2.0 and 6.0
Black	2.0 and 8.0

TESTING OF DISPERSE DYES

(Source : IS 12386 : 1988)

Disperse dyes are extensively used for dyeing and printing of hydrophobic fibres, such as polyester, cellulose acetate and nylon, and their blends with other fibres. These have become especially important for polyester fibres as they are widely used as a component of blends with cotton, wool or any other man-made fibre.

The quality of dyeing and printing with disperse dyes depends upon the characteristics of

the dyestuff, nature of dispersing agent, nature of carrier used for dyeing, and the method and conditions employed for dyeing or printing. In order to achieve optimum quality with minimum lot to lot variations and variations in dyeing due to same dyes of different manufacturers, it is essential to test the various characteristics of these dyestuffs. This will also lead to minimize cost-benefit ratio and help the user to adjust his dyeing or printing recipes suitably.

1 SCOPE

1.1 The methods of determining dye content, filtering capacity limit of water suspensions, chromatographic test, wettability and time of half dyeing are described.

2 DETERMINATION OF DYE CONTENT

2.1 Take a test specimen of the dyestuff of about one gram in a weighing bottle and dry it to constant mass in a drying oven at $105 \pm 3^\circ\text{C}$. Cool the specimen in a desiccator charged with self-indicating silica gel or calcium chloride and obtain the oven-dry mass of the specimen to an accuracy of 0.0002 g with the help of an analytical balance.

2.2 Dissolve the specimen in 100 ml of benzene or acetone or ethanol solution with constant stirring and filter the solution on a Buchner funnel through a Whatman filter paper No. 41 under low pressure of 320 mm of mercury. Repeat the procedure for the residue left twice more and collect all the filtered solutions.

2.3 Evaporate the solvent from the solution obtained in 2.2 and dye the residue obtained at $105 \pm 3^\circ\text{C}$ to constant mass. Cool the residue in a desiccator and weigh it.

2.4 Calculate the dye content, percent (P), by the formula:

$$P = \frac{m_2}{m_1} \times 100$$

where

m_1 = oven-dry mass of the specimen of dyestuff taken, and

m_2 = oven-dry mass of the residue obtained.

3 DETERMINATION OF FILTERING CAPACITY LIMIT OF WATER SUSPENSION OF THE DYE

3.1 Take 0.5 to 1.0 g of samples of the dye in steps of 0.1 g and weigh them correct to 0.01 g.

Introduce the samples in beakers of 150 ml capacity containing some distilled water at 70°C and under stirring add in each beaker distilled water to complete 100 ml. Place the suspension in each beaker and filter on a Buchner funnel through a Whatman filter paper No. 41 under low pressure of 320 mm of mercury.

3.2 Note the mass of that sample of dye for which the filter is uniformly coloured as the filtering capacity limit, however, the residues of dye may be visible in the places over the holes of the funnel when an extremely thin layer of dye may be formed.

4 CHROMATOGRAPHIC TEST

4.1 In two beakers of 50 mm dia, introduce 10 ml of 1 percent water suspension of the test dye and of the standard sample of dye as agreed to between the buyer and the seller, respectively. In a third beaker, take 10 ml of distilled water.

4.2 In each beaker, as in 4.1, after stirring the suspension, insert a cylinder made of 70×90 mm rectangular chromatographic paper.

4.3 When, in the third beaker, the water reaches the top of the paper cylinder, remove the cylinders, in the other two beakers and dry them in air.

4.4 Compare the chromatographs of the test dye and that of standard sample of dye, and record the height of rise of the suspension, intensity of colouring and appearance of strips at the bottom portion of the paper cylinder at the level of the surface of the liquid.

5 DETERMINATION OF WETTABILITY IN WATER

5.1 Weigh about 0.05 g of test dye and the same quantity of standard sample of dyestuff correct to 0.01 g, and add them simultaneously in separate glass cylinders containing 500 ml distilled water at the required temperature or at 25 or 70°C . By comparing with the standard sample of dye, note the solubility of test dye, rate

of its spreading in water, intensity of colouration and the turbidity of the suspension.

6 DETERMINATION OF TIME OF HALF DYEING

6.1 Carry out dyeings of the dye on polyester and acetate fibres as prescribed in IS 12149 : 1987 and IS 12150 : 1987.

6.2 Carry out dyeings on five samples each of polyester and acetate as in 6.1 except that the

amount of dye taken shall be double to that taken in 6.1 and times of dyeing for each sample of each fibre shall be 5, 10, 15, 20 and 25 minutes respectively.

6.3 Match the samples obtained in 6.2 with those of 6.1 separately for each fibre and note the time of dyeing for the sample obtained in 6.2 separately for each fibre. Record this time as the time of half dyeing for polyester and acetate separately.

EVALUATION OF STRENGTH AND SHADE OF PIGMENT DISPERSION BY PRINTING METHOD

(Source : IS 10010 : 1981)

The pigments for textile printing are marketed in the form of a dispersion/suspension of a finely divided pigment in water. The pigment content of such dispersion may vary from product to

product. The method of printing prescribed is a general method and wherever special instructions are given by the manufacturers, these should be followed.

1 SCOPE

1.1 This method is intended for the determination of strength and shade of a pigment dispersion by printing.

2 PREPARATION OF TEST SAMPLE

2.1 It is very important that the content of each container should be stirred thoroughly for not less than five minutes with a clean stick before sampling. The stirring should be prolonged particularly if settling of the pigment has taken place at the bottom of the container and this can be felt by probing with the stick. After ensuring that the contents in the container are completely homogenized, draw a sample of about 50 g in a clean bottle which should be corked or closed immediately after filling. This shall constitute the test sample. This test sample should be stirred again with a clean glass rod just before weighing for the preparation of printing paste (see Note 2 under A-3.3).

3 STANDARD PIGMENT DISPERSION

3.1 The standard sample of pigment dispersion, against which the strength and shade of pigment dispersion under test is evaluated, shall be as agreed between the buyer and the seller.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in the test and distilled water shall be used where use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the experimental results.

5 EVALUATION OF STRENGTH AND SHADE

5.1 Prepare a printing paste of a recommended percentage of a standard pigment dispersion (see 3.1) by exactly following the procedure given in Annex A. Prepare simultaneously a printing paste of the test sample of pigment dispersion using the same percentage as in the printing paste with the standard as also percentages varying by 5 and 10 percent on either side of the recommended percentage.

5.2 Print the printing pastes of standard and the test sample, and also 5 and 10 percent variation prints side by side by machine or screen on a cotton cloth (see Annex A), and then dry and fix the prints cloth as detailed out in Annex A. Immediately after printing, give identification marks to the prints. After fixing the prints, iron the printed cloth on the reverse before evaluating the strength and shade.

5.3 Make 1:3 reductions of the above printing pastes by using reduction thickening (see Annex A) and print them against 1:3 reduction paste of the standard. Then dry and fix as in 5.1 and 5.2.

5.4 Compare carefully the above set of prints (after ironing on the reverse) and determine by visual observation, which of the sample print tallies with the standard print. Also find out the variation in shade, if any, from the standard shade.

NOTE — The strength of the sample should be expressed on the comparisons of printings obtained in 5.1 and 5.2. The variation in shade should be reported on reduction printings obtained in 5.3.

5.5 Calculate the pigment under test by comparing the printings by the following formula:

$$S = \frac{A}{B} \times 100$$

where

S = strength of pigment in percent,

A = depth of printing of the standard pigment, and

B = depth of printing of the pigment under test comparing with that of standard.

6 REPORT

6.1 Report the value obtained in 5.5 as the strength in percent of the pigment under test as compared to the standard.

6.1.1 Report also the variation in shade in comparison with the shade of the standard.

ANNEX A

METHOD FOR PIGMENT PRINTING

A-1 APPARATUS

A-1.1 Balance

capable of weighing accurately up to 1 mg.

A-1.2 Weighing Scale

capacity up to 2.5 kg and capable of weighing accurately up to 1 g.

A-1.3 Glass Containers or Beakers

capacity 200 ml and capable to withstand high stirring.

A-1.4 High Speed Stirrer (Laboratory Model)

speed 2 000 3 000 rev/min with speed regulator, spindle and agitator blades.

A-1.5 Electric Oven

with an outlet for exhaust gases and heating range up to 160°C.

A-1.6 Electric Iron

one.

A-1.7 Laboratory Sample Printing Machine

with a roller engraved to give vertical stripes, when printed. (The width of the stripes should be minimum 1.25 cm and the stripes should be not less than 1.25 cm apart.)

A-1.8 A screen printing table together with a screen and squeegee to give stripe design as in A-1.7.

A-2 PRINTING AUXILIARIES

A-2.1 Binder for Pigment Printing

a synthetic resin based on an acrylic copolymer emulsion of the oil in water type. A binder with non-volatile matter content of 30 ± 2 percent should be used.

A-2.2 Soluble Thickening

carboxy methyl cellulose, preferably of low viscosity giving practically colourless thickening with good flow property and neutral pH.

A-2.3 Mineral Turpentine-Solvent

125/240 [see IS 1745 : 1966 Specification for petroleum hydrocarbon solvents (*first revision*)].

or

A-2.4 Kerosene

See IS 1459 : 1974 Specification for kerosines (*second revision*).

A-2.5 Diammonium Phosphate Solution

prepared by dissolving diammonium phosphate (see IS 6448 : 1971 Specification for diammonium phosphate) in water in the proportion 1 : 3 by mass.

A-2.6 Urea

technical grade [see IS 1781 : 1975 Specification for urea, technical (*first revision*)].

A-2.7 Cotton Fabric

The cotton cloth for printing test should be unmercerized poplin. It shall be fully desized, scoured, bleached and soured. The pH of the cloth ready for pigment printing should be neutral or slightly acidic and never alkaline. It shall not be resin finished and shall not be treated with optical whitening agent.

A-3 PROCEDURE

A-3.1 Preparation of Soluble Thickening

Prepare 5 percent thickening by weighing 5 g of carboxy methyl cellulose powder (see A-2.2) accurately and gradually sprinkle it over 95 ml cold water with constant stirring. Allow it to soak overnight. This should be sieved through a fine mesh cloth before use.

A-3.2 Preparation of 10 Percent Stock Thickening (Emulsion)

Weigh, in a stainless steel or plastic container (capacity about 2 kg), 100 g binder (see A-2.1) and 100 ml water. Then add to this 50 g soluble thickening (see A-3.1) and 20 g urea. Mix the ingredients under high speed stirrer with gradual addition of 7.0 g mineral turpentine or kerosene (see A-2.3 or A-2.4) and continue stirring till a homogeneous emulsion thickening is obtained. The total mass of the components of mixture shall be 1 000 g.

A-3.3 Preparation of a Printing Paste

Weigh accurately, in a glass container or beaker (see A-1.3), 4 g pigment dispersion (see Notes 1 and 2). Then place the beaker containing the pigment dispersion on a weighing scale together with a glass rod for mixing purpose and counterpoised. Add 89 g of 10 percent stock thickening (see A-3.2) followed by 3 g binder [extra addition to adjust the ratio of colour : binder, 1 : 3 (see Note 3)] and mix the whole mixture thoroughly. Add 4 g diammonium phosphate solution. Stir under a high speed stirrer for about 30 to 60 seconds. The paste is then ready for printing. The total mass of ingredients shall be 100 g.

NOTES

1 Normally 4 percent shade for printing shall be used. For black pigments, it is necessary to have deeper shade and 6 to 8 percent of pigment is taken in preparing of printing paste.

2 Some pigment dispersions on long storage show separation due to settling. Hence, all pigment dispersions must be thoroughly stirred before sampling as well as before weighing.

3 The strength of a pigment print also depends, among other factors, on the quantity of binder used. Therefore, to get a maximum strength, a colour to binder ratio of 1 : 3 should be maintained and also minimum of 10 percent binder in 100 parts of printing paste.

A-3.4 Preparation of Reduction Printing Paste

To make 1 : 3 reduction printing paste, weigh in a glass container or a beaker, 25 g printing paste (*see* A-3.3) and add 75 g of 10 percent stock thickening (*see* A-3.2) containing 0.5 ml diammonium phosphate solution and mix initially whole mixture with a glass rod and thereafter stir it for about 60 seconds under a high speed stirrer.

NOTE — For black pigments, the reduction paste should contain 0.5 to 1.0 percent of the pigment.

A-3.5 Printing

The printing pastes prepared with the sample and standard in the manner described in A-3.3 and A-3.4 are printed side by side in full shade and reduction on plain white cotton cloth pieces (*see* A-2.7) with a sample printing machine or a screen giving uniform and adequate pressure to get even prints. Excessive pressure should be avoided.

NOTE — If the screen printing is used, the concentration of the pigment should be reduced by 25 percent than that of the concentration used in roller printing.

A-3.6 Drying

The printed cloth pieces are evenly dried at a temperature of about 80°C in an oven (*see* A-1.5) till such time that the dried prints do not retain any trace of mineral turpentine or kerosene.

A-3.7 Fixing (Curing)

The dried prints are fixed by curing the prints at a temperature of 140°C for 5 minutes. The prints should not smell of kerosene/mineral turpentine when they are ready for curing. Over-curing should be avoided since, in certain cases, it causes a dullening of the prints.

GENERAL PRINCIPLES OF TESTING TEXTILES FOR COLOUR FASTNESS TESTS

(Source : IS 10251 : 1982)

This standard is based on ISO 105/A-1984
Textiles — Tests for colour fastness A01 —

General principles of testing, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This standard provides general information about the methods for testing colour fastness of textiles for the guidance of users. The uses and limitations of the methods are pointed out, several terms are defined, an outline of the form of the methods is given, and the contents of the clauses constituting the methods are discussed. Procedures common to a number of methods are discussed briefly.

1.2 By colour fastness is meant the resistance of the colour of textiles to the different agencies to which textiles may be exposed during manufacture and subsequent use. The change of colour and staining of undyed adjacent fabrics are assessed as fastness ratings. Other visible changes of the textile material under test, for example, surface effects, change in gloss or shrinkage, should be considered as separate properties and reported as such. Any loose fibres from the specimen adhering to the adjacent fabrics shall be removed before assessment of staining.

1.3 The individual test methods may be used not only for assessing colour fastness of textiles, but also for assessing colour fastness of dyes. When a method is so used, the dye is applied to the textile in specified depths of colour by stated procedures and the textile is then tested in the usual way.

1.4 For the most part, individual methods are concerned with colour fastness to a single agency, as the agencies of interest in a particular case, and the order of application, will generally vary. It is recognized that experience and future developments in practice may justify procedures in which two or more agencies are combined.

1.5 The conditions in the tests have been chosen to correspond closely to treatments usually employed in manufacture and to conditions of ordinary use. At the same time, they have been kept as simple and reproducible as possible. As it cannot be hoped that the tests will duplicate all the conditions under which textiles are processed or used, the fastness ratings should be interpreted according to the particular needs of each user. They, however, provide a common basis for testing and reporting colour fastness.

2 GENERAL PRINCIPLES

2.1 A specimen of the textile to be tested, with adjacent fabric attached, if staining is to be assessed, is subjected to the action of the agency in question. The extent of any change in colour and that of any staining of the adjacent fabric are assessed and the rating is expressed in fastness numbers.

3 OUTLINE OF FORM OF THE METHODS

3.1 The headings of the principal clauses of the individual test methods are as follows:

- Foreword
- Scope
- Principles
- References
- Sampling
- Apparatus and Reagents (or Standards and Apparatus)
- Test Specimen
- Procedure
- Test Report
- Note(s)

4 SCOPE OF INDIVIDUAL TEST METHODS

4.1 Under this heading, in each method, are given intended use of the method, its limitation and the definitions of any terms that may not be clear.

4.2 Details of the principal natural and man-made fibres which can be submitted to each test are given. These lists are by no means exclusive, and any dyed or printed cloth not mentioned in the method (whether manufactured wholly from one fibre or from a mixture of fibres) may be submitted to test. In such cases, it is necessary to verify and note whether the procedure is likely to cause any alteration in the fibre under test. This applies particularly to all man-made fibres (acrylic, pure or copolymer; polyvinyl, pure or copolymer; polyester, etc) currently being developed, of which any list is always likely to be incomplete.

5 PRINCIPLE (OF METHOD)

5.1 Under this heading, in each method, is given a concise statement of the principle of the method to enable the user to decide whether the method is the one that he is seeking.

6 REFERENCES

6.1 Under this heading, in each method, is given a complete list of other documents which are indispensable for the application of the method.

7 APPARATUS AND REAGENTS

7.1 Under this heading, in each method, are described the equipment and supplies required for the test.

7.2 Test Solutions

Should be prepared using distilled water. The concentrations of baths are given in millilitres per litre (ml/l) or grams per litre (g/l). The qualities of chemicals to be used are given in each method. For crystalline substances, the amount of water of crystallization is given and, for liquids, the relative density at 27°C.

7.3 By Adjacent Fabric

Is meant a small piece of undyed cloth of a single or of several generic kinds of fibres to be used during the tests for assessing staining.

7.3.1 Single Fibre Adjacent Fabrics

If not specified otherwise, should be of plain weave, medium mass per unit area, scoured and bleached, and free from chemically damaged fibres, finishes, residual chemicals, dyes of fluorescent whitening agents. The fluidity value of cotton adjacent fabric may not exceed 5 rhes.

7.3.2 Properties of the single fibre adjacent fabrics are given in Annex A.

7.3.3 Multi-fibre Adjacent Fabrics

Are made of yarns of various generic kinds of fibres each of which forms a stripe of at least 1.5 cm width providing even thickness of the fabric. The staining properties of generic fibres used in single-fibre and multi-fibre adjacent fabrics must be identical.

7.4 By Standard of Fading

Is meant a dyed fabric of similar appearance as the test control (8.4), showing the colour to which the test control should fade during the test.

NOTE — Information regarding the availability of the test controls may be obtained from the Bureau of Indian Standards, New Delhi.

7.5 Selection and Use of Adjacent Fabrics

Two alternative procedures for selection of adjacent fabrics are provided, and details of the type

of adjacent fabric used shall be given in the test report, including dimensions.

7.5.1 Types of Adjacent Fabric

Either of the following procedures may be used:

- a) Two single-fibre adjacent fabrics. The first adjacent fabric should be of the same generic kind of fibre as the material under test or that predominating in the case of blends. The second adjacent fabric should be that indicated in the individual test or as specified otherwise.
- b) One multi-fibre adjacent fabric. In this case, no other adjacent fabric may be present, as this can affect the degree of staining of the multi-fibre fabric.

7.5.2 Dimensions and Use of Adjacent Fabrics

7.5.2.1 When single-fibre adjacent fabrics are used, these shall be of the same dimensions as the specimen under test (normally 10×4 cm). As a general principle, each surface of the specimen is completely covered by each of the adjacent fabrics. Special requirements are outlined in 8.6.

7.5.2.2 When a multi-fibre adjacent fabric is used, this shall be of the same dimensions as the specimen under test (normally 10×4 cm). As a general principle, it shall cover the face side only of the specimen.

8 TEST SPECIMEN

8.1 Under the heading, in each method, are given the particular requirements for the specimen to be used in the test.

8.1.1 Definitions of the following terms are given in 8.2 to 8.4:

- a) Specimen,
- b) Composite specimen, and
- c) Test-control specimen.

8.1.2 General directions for their preparation are also given in 8.5 and 8.6.

8.2 By Specimen

Is meant a small piece of material that is to be submitted to the test; it is usually taken from a large sample representing a lot of the dyed or printed textile material.

8.3 By Composite Specimen

Is meant the specimen together with one or two selected adjacent fabrics used for assessing staining.

8.4 By Test-Control Specimen

Is meant a specimen of known extent of colour change during the test, used to ensure that the

test, is conducted correctly. Details of the preparation of the test-control specimens are given as appropriate in each individual test method. The test-control specimen is treated in parallel with the test specimen under the same conditions as prescribed in the individual test method.

8.5 Preparation of Specimens

8.5.1 *Fabrics*

Specimens of specified dimensions are cut from woven and knitted fabrics, felts and other piece goods. The fabrics should be free from creases, so that the treatment they receive may result in uniform action over the whole area.

8.5.2 *Yarns*

Yarns to be tested may be knitted into fabric, from which a specimen is taken. They may also be wound parallel, for example, on a U-shaped wire frame. For dry treatments, close winding on a card is recommended. For certain wet treatments without accompanying undyed adjacent material, hanks of yarn tied at both ends may be used.

8.5.2.1 The method of preparation used shall be specified in the test report.

8.5.3 *Loose Fibres*

Loose fibre may be tested after combing and compressing into a sheet.

8.6 Preparation of Composite Specimens

8.6.1 Sewing threads must be free from fluorescent brightening agents.

8.6.2 Composite specimens using two single-fibre adjacent fabrics.

8.6.2.1 If the specimen is fabric, it is placed between the two adjacent fabrics and sewn along one short side, except in the case of certain methods involving a mechanical action, when the composite specimen is sewn along all four sides.

8.6.2.2 When testing unions in which one fibre predominates on one side and another fibre on the other side, the specimen should be placed between the adjacent fabrics so that the predominant fibre is next to the adjacent fabric of the same generic kind of fabric.

8.6.2.3 If the specimen is a printed fabric, the composite specimen is arranged in such a manner that the face is in contact with half or each of the adjacent fabrics; depending on the design of the print, more than one composite specimen may be required.

8.6.2.4 If the specimen is yarn or loose fibre, an amount approximately equal to one-half the combined mass of the adjacent fabrics is evenly

spread over one adjacent fabric, covered with the other, and sewn around all four side with additional stitching at about 1 cm intervals; in the case of yarn specimens, this stitching shall be at right angles to the direction of the yarns.

8.6.3 Composite specimens using one multi-fibre adjacent fabrics.

8.6.3.1 If the specimen is fabric, it is placed with its face in contact with the multi-fibre fabric and sewn along one short side, except in the case of certain methods involving mechanical action, when the composite specimen is sewn along all four sides.

8.6.3.2 When testing unions in which one fibre predominates on one side and another fibre on the other side, two composite specimens must be so prepared that each side is in contact with the multi-fibre adjacent fabric.

8.6.3.3 If the specimen is yarn or loose fibre and amount approximately equal to the mass of the multi-fibre adjacent fabric is evenly spread over the multi-fibre adjacent fabric, yarns being laid at right angles to the individual stripe. This is then covered with an equal size piece of a light-weight, non-staining quality of polypropylene fabric, stitched along all four sides and in addition between each pair of adjacent stripes comprising the multi-fibre adjacent fabric.

9 CONDITIONING

9.1 Special conditioning of specimens and of adjacent fabrics used with them is not usually necessary, but they should be neither moist nor very dry.

9.2 For tests in which differences in moisture content of the specimen and adjacent fabric will influence the results, all fabrics should be in a standard condition, that is, in moisture equilibrium with air having a temperature of $27 \pm 2^\circ\text{C}$ and relative humidity of 65 ± 2 percent.

10 PROCEDURE

10.1 Under this heading, in each method, are described the series of operations through which the specimen is taken, including the assessment of colour change and staining of adjacent fabrics. The use of test-control specimens is specified in some of the methods to ensure that the tests have been conducted correctly.

10.2 Tolerances

are given for numerical values of dimensions, temperatures and times that are considered critical. If no tolerance is given, the precision of the measurements need be only to be expected when using common instruments and reasonable care. The precision is further indicated by the number of significant figures in the values given.

10.3 By Liquor Ratio

is meant the ratio of the volume of the liquor used in the treatment, expressed in millilitres (ml), to the mass of the specimen or the composite specimen (specimen plus adjacent fabric) expressed in grams (g).

10.4 Wetting Out

When wetting specimens, special care should be taken to ensure that they are uniformly saturated. In particular, when wool or materials containing wool are wetted out, it is necessary for them to be immersed in softened, distilled or deionized water and either thoroughly kneaded by hand or with a mechanical device, for example, the flattened end of a glass rod, or placed in a suitable flask, evacuated using a water suction pump or a similar apparatus.

10.5 Spotting

For tests in which the material is spotted with water or a reagent and the surface rubbed with a glass rod to ensure penetration, care should be taken not to raise the surface of the materials; otherwise, there will be change in the reflection of light and hence in appearance.

10.6 Wetting to 100 Percent Increase in Mass

When the material is to be wetted to contain its own mass of liquor, it may be saturated with the liquor and then drawn between two rubber rollers or squeezed by means of a rubber roller on a glass plate, or centrifuged. Wringing by hand does not give uniform wetting.

10.7 Temperatures

are given in degrees celsius (°C), usually with a tolerance of $\pm 2^{\circ}\text{C}$. For reliable results, continuous temperature control is essential.

11 FASTNESS OF DYES

11.1 As the fastness of a dye depends on the depth of colour, it is necessary to specify standard depths which are to be used by dye manufacturers. A main range of standard depths, referred to as 1/1 standard depths, is recommended in 18 hues and is to be used whenever possible.

11.2 Supplementary ranges twice as deep (referred to as 2/1 standard depths), and weaker (referred to as 1/3, 1/6, 1/12 and 1/25 standard depths), are also recommended and are to be used whenever additional fastness data are required.

11.3 In the case of navy blues and blacks, only two standard depths are recommended, namely:

Navy blue/light (N/L) Black/light (B/L)
Navy blue/dark (N/Dk) Black/dark (B/Dk)

Whenever possible, dye manufacturers' pattern cards should provide fastness ratings at both depths.

11.4 These standard depths should be prepared on a matt fabric (wool gaberdine or printed card).

NOTE — Information regarding the master sets of dyeings may be obtained from the Bureau of Indian Standards, New Delhi.

11.5 These standard depths are not subjected to any fastness test, but merely indicate at what depth of colour the fastness figures of dye manufacturers have been obtained, irrespective of dye or fibre.

12 ASSESSMENT OF COLOUR FASTNESS

12.1 Colour fastness is assessed separately with respect to change in colour of the specimen in the test and with respect to staining of adjacent fabrics. Specimens and adjacent fabrics should have cooled after drying and should have regained their normal moisture content before the assessment, except when specified otherwise. In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

12.2 Colour Fastness with Respect to Change in Colour

The change which occurs in a test may be a change in depth of colour (that is, saturation), a change in hue: a change in brightness or any combination of these.

Regardless of the character of the change, the assessment is based upon the magnitude of the visual contrast between the specimen after the test and a specimen of the original material. This contrast is compared visually with the contrast represented by the five or nine pairs of colour chips (or swatches of cloth), ranging in both cases from 5, which relates no contrast, to 1, which illustrates a large contrast, these scales being specified in IS 768 : 1982.

The fastness rating of the specimen is that number of the grey scale which corresponds to the contrast between the original and the treated specimens in the case of the nine-step scale; in the case of the five-step scale, if the contrast is judged to be nearer to the non-existent half-step, the appropriate half-step rating shall be given.

Assessments to less than one half-step are not permitted. A rating of 5 is given only when there is no difference between the tested specimens and the original material.

In assessing the results of test for colour fastness to light, the exposed specimens are compared with the scale of eight standard dyed blue cloths,

which have been exposed simultaneously. In addition, the grey scale for assessing change in colour is used as a means of defining the extent to which fading should be carried out in the test.

In some tests, changes occur in the surface appearance of the specimen (for example, pile arrangement, structure, lustre, etc), in addition to change in colour. In such cases, the surface of the specimen shall be restored, if possible, to its original condition by combing or brushing, etc. If this restoration is not possible, the fact that the resultant rating gives not only the change in colour but also the overall change in appearance should be noted in the test report.

For certain types of textile products, a simple wetting can create an apparent difference in colour compared with a fabric which has not been wetted, and this is not due to a real change in colour. This colour difference should be attributed to a modification of the surface of the fabric or to a migration of the finish. In this case, evaluation should be made by comparison with the original fabric which has been wetted and not with the original fabric. The original fabric, placed horizontally, is wetted superficially and evenly by spraying with distilled water, avoiding formulation of drips, and is left to dry. If this procedure has been conducted, details shall be included in the test report.

12.3 Colour Fastness with Respect to Staining

The degree of staining of the adjacent fabric, either by absorption of dye from the bath or by direct transfer of colour from the specimen, is assessed by visual inspection of the side of the adjacent fabric which was in contact with the specimen, the colour of the bath is ignored if not specified otherwise (for example, in the dry cleaning test).

For assessing staining, the five- or nine-step scale specified in IS 769 : 1982 is used in a manner similar to that described for assessing change in colour in 12.2. Staining is assessed for each type of adjacent fabric in the test, staining at the stitching being ignored. If the adjacent fabric undergoes any visual change when subjected to a test in the absence of a specimen, a sample of the adjacent fabric which has been treated in this manner shall be used as the reference sample for the assessment of staining.

12.4 Influence of Pre-treatments and After-treatments

It is well known that the fastness of dyeing obtained with a given dyestuff depends on the amount of dye, the substrate and the treatments applied to the textile material before, during or after dyeing. It follows that fastness is not a property of the dye itself, but only of a given dyeing.

12.5 Simple Checks on Colour Fastness

The existence of a simple method of checking the colour fastness of a dyed cloth by reference to a standard dyeing is extremely useful for authorities receiving goods in large quantities, who wish to determine whether these are comparable with an accepted piece.

Where it is not essential to determine the exact colour fastness rating, it may be sufficient to compare the cloth being investigated with a standard dyeing. This method is used when it is necessary to establish only whether the colour fastness of the specimen to a particular agency is greater than, equal to or less than that of reference dyeing.

13 CONDITIONS OF VIEWING AND ILLUMINATION IN ASSESSING COLOUR FASTNESS

13.1 In assessing colour fastness, a piece of the original material and the tested specimen, or the undyed and stained adjacent fabrics are arranged side by side in the same plane and oriented in the same direction. Two or more layers are used, if necessary to avoid effects of other backing on the appearance. The appropriate grey scale is placed nearly in the same plane. For optimum precision, the areas of contrast to be compared should be approximately of the same size and shape, if necessary; this can be achieved by the use of a mask of neutral grey colour approximately mid-way between that illustrating grade 2 of the grey scale for assessing change in colour (this is approximately Munsell N 5) and of a size equal to that of the grey scale steps. The surrounding field is the same uniform grey. The surfaces to be compared are illuminated by North skylight in the Northern hemisphere, South skylight in the Southern hemisphere or an equivalent source with an illumination of 600 lx or more. The light is incident upon the surfaces at an angle of approximately 45°, and the direction of viewing is approximately along the perpendicular to the plane of the surfaces.

14 TEST REPORT

14.1 Under this heading, in each method, is indicated the information to be supplied in the test report. In addition to fastness ratings, information is required to be given on the adjacent fabrics used and any deviations from the test procedure.

15 NOTE(S)

15.1 Under this heading in each method are given references to literature and other information not considered necessary for inclusion in the body of the test method.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass in g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
Cotton	115 ± 5	1/1 Plain	35	31	16·5	14 0
Viscose	140 ± 5	1/1 Plain	28	22	20·0	33·0
Wool	125 ± 5 — 0	1/1 Plain	21 ± 0·5	18 ± 0·5	15·6 × 2 Worsted	15·5 × 2 Worsted
Polyamide	130 ± 5	1/1 Plain	17·5	20	10 × 2	20
Polyester	130 ± 5	1/1 Plain	23·5	20·5	7·5 × 2	20
Acrylic	135 ± 5	1/1 Plain	17·5	16	10 × 2	10 × 2

NOTES

1 For wool adjacent fabric, additional requirements are: (a) pH value of aqueous extract 6·5 to 7·5, (b) residual fat content $0·4 \pm 0·1$ percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic adjacent fabrics, additional requirement are: (a) pH value of aqueous extract $7 \pm 0·5$, and (b) residual oil content less than 1·0 percent.

3 For polyester adjacent fabric, additional requirement is residual oil content less than 0·5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

METHOD FOR EVALUATING CHANGE IN COLOUR

(Source : IS 768 : 1982)

This method is in line with ISO 105/A-1984 Textiles — Tests for colour fastness A02 — Grey scale for assessing change in colour, issued by the International Organization for Standardization

(ISO). It has a provision for 9-step scale and includes colorimetric data for the grey scale for evaluating change in colour.

1 SCOPE

1.1 This method is intended for evaluating, by means of grey scale, change in colour of textile materials during fastness tests.

1.2 A precise colorimetric specification of the scale is given as a permanent record against which newly prepared working standards and standards that may have changed should be compared.

2 PRINCIPLE

2.1 The essential or 5-step scale consists of five pairs of non-glossy grey colour chips (or swatches of grey cloth) which illustrate the perceived colour differences corresponding to fastness ratings 5, 4, 3, 2 and 1. This essential scale may be augmented by the provision of similar chips or swatches illustrating the perceived colour differences corresponding to the half-step fastness ratings 4-5, 3-4, 2-3 and 1-2, such scales being termed 9-step scales. The first member of each pair is neutral grey in colour and the second member of the pair illustrating fastness ratings 5 is identical with the first member. The second members of remaining pairs are increasingly lighter in colour so that each pair illustrates increasing contrasts or perceived colour differences which are defined colorimetrically. The full colorimetric specification is given below.

2.2 The chips or swatches shall be neutral grey in colour and shall be measured with a spectrophotometer with the specular component included. The colorimetric data shall be calculated using CIE 1964 supplementary standard colorimetric system (10° observer data) for Illuminant D₆₅.

2.3 The Y tristimulus value of the first member of each pair shall be 12 ± 1 .

2.4 The second member of each pair shall be such that the colour difference between it and the adjacent first member is as follows:

Fastness Grade	CIELAB Difference	Tolerance
5	0	0.2
(4-5)	0.8	± 0.2
4	1.7	± 0.3
(3-4)	2.5	± 0.35
3	3.4	± 0.4
(2-3)	4.8	± 0.5
2	6.8	± 0.6
(1-2)	9.6	± 0.7
1	13.6	± 1.0

(bracketed values apply only to the nine-step scale).

3 PROCEDURE

3.1 Use of the Scale

Place a piece of the original textile and the tested specimen of it side by side in the same plane and oriented in the same direction. Place the grey scale nearby in the same plane. The surrounding field should be neutral grey colour approximately midway between that illustrating Grade 1 and that illustrating Grade 2 of the grey scale for assessing change in colour (this is approximately Munsell N5). If it is necessary to avoid the effects of the backing on the appearance of the textiles, use two or more layers of the original textile under both original and tested specimens. Illuminate the surfaces with north skylight in the Northern hemisphere, South skylight in the Southern hemisphere, or an equivalent source with an illumination of 600 lx or more. The light should be incident upon the surfaces at approximately 45° and the direction of viewing approximately perpendicular to the plane of the surfaces. Compare the visual difference between original and tested textiles with the differences represented by the grey scale.

3.1.1 If the 5-step scale is used, the fastness rating of the specimen is that number of the grey scale which has a perceived colour difference equal in magnitude to the perceived colour difference between the original and treated specimens; if the latter is judged to be nearer the imaginary contrast lying midway between the two adjacent pairs than it is to either, the specimen is given an intermediate assessment, for example, 4-5 or 2-3. A rating of 5 is given only when there is no perceived difference between the tested specimen and the original material.

3.1.2 If the 9-step scale is used, the fastness rating of the specimen is that number of the grey scale which has a perceived colour difference nearest in magnitude to the perceived colour difference between the original and the tested specimens. A rating of 5 is given only when there is no perceived difference between the tested specimen and the original material.

NOTE — When a number of assessments have been made, it is very useful to compare all the pairs of original and tested specimens which have been given

the same numerical rating. This gives a good indication of the consistency of the assessments, since any errors become prominent. Pairs which do not appear to have the same degree of contrast as the remainder of their groups should be re-checked against the grey scale and, if necessary, the rating should be changed.

3.2 Describing Colour Change in Fastness Tests

3.2.1 In using the grey scale, as outlined in 3.1 to 3.1.2, the character of the change in colour, whether, in hue, depth, brightness or any combination of these, is not rated; the overall difference or contrast between original and tested specimens is the basis for the assessment.

3.2.2 If, as in rating dyes on textiles, for example, it is desired to record the character of the change in colour of the textile in the test, appropriate qualitative terms may be added to the numerical rating, as illustrated by the following example:

Rating	Meaning	
	Contrast Corresponding to the Following Grades of Grey Scales	Character of Changes
3	Grade 3	Loss in depth of colour only
3 redder	Grade 3	No significant loss in depth but tone redder
3 weaker yellower	Grade 3	Loss in depth and change in hue

Rating	Meaning	
	Contrast Corresponding to the Following Grades of Grey Scales	Character of Changes
3 weaker bluer duller	Grade 3	Loss in depth and changes in both hue and brightness
4-5 redder	Intermediate between grades 4 and 5	No significant loss in depth but tone slightly redder

3.2.3 When changes in colour occur in two or three directions, it is considered neither feasible nor necessary to indicate the relative magnitude of each change.

3.2.4 When the space available for recording qualitative terms is restricted, as on pattern cards, the following abbreviations may be used:

Abbreviation	Meaning
Bl	Bluer
G	Greener
R	Redder
Y	Yellower
W	Weaker
Str	Stronger
D	Duller
Br	Brighter

METHOD FOR EVALUATING STAINING

(Source : IS 769 : 1982)

This method is in line with the corresponding ISO Standard, ISO 105/A-1984 Textiles — Tests for colour fastness A03 — Grey scale for assessing staining, issued by the International

Organization for Standardization (ISO). It has a provision for 9-step scale and includes colorimetric data for the grey scale for evaluating staining.

1 SCOPE

1.1 This standard prescribes the method for evaluating the staining of undyed fabrics by means of grey scale (geometrical staining scale) during colour fastness tests.

1.2 A precise colorimetric specification of the scales is given as a permanent record against which newly prepared working standards and standards that may have changed should be compared.

2 PRINCIPLE

2.1 The essential or 5-step scale consists of five pairs of non-glossy grey or white colour chips (or swatches of grey cloth) which illustrate the perceived colour differences corresponding to fastness ratings 5, 4, 3, 2 and 1. This essential scale may be augmented by the provision of similar chips or swatches illustrating the perceived colour differences corresponding to the half-step fastness ratings 4.5, 3.4, 2.3 and 1.2, such scales being termed 9-step scales. The first member of each pair is white in colour and the second member of the pair illustrating fastness rating 5 is identical with the first member. The second members of the remaining pairs are increasingly darker in colour so that each pair illustrates increasing contrasts or perceived colour differences which are defined colorimetrically. The full colorimetric specification is given below.

2.2 The chips or swatches shall be white or neutral grey in colour and shall be measured with a spectrophotometer with the specular component included. The colorimetric data shall be calculated using the CIE 1964 supplementary standard colorimetric system (10° observer data) for illuminant D₆₅.

2.3 The Y tristimulus values of the first member (white) of each pair shall not be less than 85.

2.4 The second member of each pair shall be such that the colour difference between it and the adjacent first member is as follows:

Fastness Grade	CIELAB Difference	Tolerance
5	0	0.2
(4-5)	2.2	± 0.3
4	4.3	± 0.3
(3-4)	6.0	± 0.4
3	8.5	± 0.5
(2-3)	12.0	± 0.7
2	16.9	± 1.0
(1-2)	24.0	± 1.5
1	34.1	± 2.0

(bracketed values apply only to the nine-step scale).

3 PROCEDURE

3.1 Use of the Scale

Place a piece of the unstained adjacent fabric (the original piece) and the piece which has been part of a composite specimen in a fastness test (the tested specimen) side by side in the same plane. The surrounding field should be neutral grey colour approximately midway between that illustrating Grade 1 and that illustrating Grade 2 of the grey scale for assessing change in colour (this is approximately Munsell N5). If it is necessary to avoid the effects of the backing on the appearance of the textiles, use two or more layers of the unstained, undyed textile under both original and treated pieces. Illuminate the surface with North skylight in the Northern hemisphere, South skylight in the Southern hemisphere or an equivalent source with an illumination of 600 lx or more. The light should be incident upon the surfaces at approximately 45° and the direction viewing approximately perpendicular to the plane of the surfaces. Compare the visual difference between the original piece and the tested specimen with the differences represented by the grey scale.

3.1.1 If the 5-step scale is used, the degree of staining of the tested specimen is that number of the grey scale which has a perceived colour difference equal in magnitude to the perceived colour difference between the original and the tested specimens; if the latter is judged to be nearer the imaginary contrast lying midway between two adjacent pairs than it is to either, the tested specimen is given an intermediate assessment, for example 4.5 or 2.3. A rating of 5 is given only when there is no perceived difference between the tested specimen and the original piece.

3.1.2 If the 9-step scale is used, the degree of staining of the tested specimen is that number of the grey scale which has a perceived colour difference nearest in magnitude to the perceived colour difference between the original piece and the tested specimen. A rating of 5 is given only when there is no perceived difference between the tested specimen and the original piece.

NOTE — When a number of assessments have been made, it is very useful to compare all the pairs of original and tested specimens which have been given the same numerical rating. This gives a good indication of the consistency of the assessments, since any error becomes prominent. Pairs which do not appear to have the same degree of contrast as the remainder of their groups should be re-checked against the grey scale and, if necessary, the rating should be changed.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO DAYLIGHT

(Source : IS 686 : 1985)

Colour fastness of textile materials to daylight is of considerable importance to the consumer. Though a quicker method of determining colour fastness to light by xenon arc exists yet, in cases of dispute, the methods prescribed in this standard should be preferred. Two methods of mounting and testing of specimens for colour fastness to daylight have been prescribed. Method 1 is considered preferable and should be used in cases of dispute but it requires one set of standard patterns for each test specimen and is, therefore, impracticable when a large

number of test specimens have to be tested concurrently. Method 2 enables a number of test specimens of differing light fastness ratings to be rated against a single set of standard patterns. This method is used when a large number of test specimens have to be tested concurrently. The methods prescribed are based on ISO 105/B-1984 Textiles — Tests for colour fastness B01 — Colour fastness to light : (Daylight), issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 These methods are intended for determining the colour fastness of textile materials of all kinds and in all forms to the action of daylight.

2 PRINCIPLE

2.1 A specimen of the textile material is exposed to daylight under prescribe conditions including protection from rain along with eight blue dyed wool standard patterns. The fastness is assessed by comparing the change in colour of the specimen with that of the standard patterns.

3 SAMPLING

3.1 Sample shall be drawn from the lot so as to be representative of the lot. Sample drawn in accordance with the material specification for the coloured textile material or as agreed to between the buyer and the seller shall be held to be representative of the lot.

4 PREPARATION OF TEST SPECIMENS

4.1 An area of the textile product not less than 1 cm × 6 cm should be used for Method 1 (see 7.1.1) or 1 cm × 10 cm for Method 2 (see 7.1.2) so that each exposed portion is not less than 1 cm × 2 cm. The specimen may be a strip of cloth, yarns wound close together on a card or laid parallel and fastened on a card, or a mat of fibres combed and compressed to give a uniform surface and fastened on a card. Draw one such specimen from each piece in the test sample.

4.2 To facilitate handling, the specimen or specimens to be tested and the similar strips of the standard patterns may be mounted on a card in an arrangement as indicated in Fig. 1 or 2 (see 7.1.1 or 7.1.2).

4.3 The specimens to be tested and the blue strips of the standard patterns should be of equal size and shape in order to avoid errors in assessment due to over-rating the visual contrast between exposed and unexposed parts on the larger specimens as against the narrower standard patterns.

4.4 While the testing carpets, the standard patterns shall be arranged in such a way that they are in the same plane as the surface of the carpet specimens. This can be achieved by underlaid cardboard.

5 STANDARD PATTERNS

5.1 Standard patterns of blue wool cloth ranging in light fastness from rating No. 1 (very low light fastness) to rating No. 8 (very high light fastness) and dyed with the dyes, as prescribed below, shall be used:

Fastness Rating No.	Dye [Colour Index (CI), Designation (see Note 1)]
1	CI acid blue 104
2	CI acid blue 109
3	CI acid blue 83
4	CI acid blue 121
5	CI acid blue 47
6	CI acid blue 23
7	CI solubilized vat blue 5 (see Note 2)
8	CI solubilized vat blue 8 (see Note 2)

NOTES

1 The Colour Index (third edition) published by the Society of Dyers and Colourists, P.O. Box 244, Perkins House, 83 Grattan Road, Bradford BD 1 2JB, West Yorks, United Kingdom.

2 The patterns dyed with these dyes should be decatized after dyeing.

3 Sets of standard patterns of light fastness rating No. 1 to 8 may be obtained from the Bureau of Indian Standards or the British Standards Institution. They are specially dyed to match a master set of standard patterns in colour and in fading behaviour. It has been found that, when repeated dyeings for the dyed standard patterns are made, the amount of dye required to match a previous lot is often different from that originally used. The dyeing strengths would, therefore, be misleading and they are intentionally omitted from this list.

6 APPARATUS

6.1 Exposure Rack

Facing South in the Northern hemisphere, North in the Southern hemisphere and slopping at an angle from the horizontal approximately equal to the latitude of the place where the exposure is made. The rack shall be sited, preferably in a non-residential, non-industrial area free from dust and automobile exhaust fumes.

6.1.1 The rack shall be placed so that shadows of surrounding objects, including any framing, will not fall on the exposed specimen and constructed so that the latter are firmly held. There shall be adequate ventilation behind the mounted specimens and the rack shall be covered with window glass to protect the specimens from rain and other elements of the weather. The transparency of the glass used shall be at least 90 percent between 380 and 750 nm, falling to 0 percent between 310 and 320 nm.

6.1.2 The minimum permissible distance between the glass and the specimens is 5 cm. In order to minimize shadows due to the varying angle of the sun, the usable exposure area under the glass is limited to that of the glass cover reduced on each side by twice the distance from the glass cover to the specimen.

6.2 Opaque Cardboard

or other thin opaque material, for example, thin sheet aluminium or cardboard covered with aluminium foil or in the case of pile fabrics, a cover that avoids surface compression.

6.3 Grey Scale

for evaluating change in colour.

7 PROCEDURE FOR MOUNTING, EXPOSURE AND PRELIMINARY ASSESSMENT OF LIGHT FASTNESS

7.1 Exposure of Specimens

Expose the specimen (or group of specimens) and the standard patterns simultaneously to daylight every day from sunrise to sunset under con-

ditions described in 6.1 in such a manner and for such times as are necessary to evaluate fully the light fastness of each specimen relative to that of the standard patterns, by successively covering the specimens and exposed standard patterns throughout the duration of the test. The suggested methods of procedure follows.

7.1.1 Method 1

7.1.1.1 Arrange the specimen to be tested and the standard patterns as shown in Fig. 1 with an opaque cover *AB* across the middle third of the specimen and standard patterns. Expose to daylight under the conditions described in 6.1. Follow the effect of light by removing the cover *AB* and inspecting the specimen frequently. When a change in colour of the exposed portion of the specimen can just be perceived, equal to grey scale grade 4-5, note the number of the standard pattern showing a similar change. (This is preliminary assessment of light fastness of the specimens).

7.1.1.2 Continue to expose the specimen and the standard patterns until the contrast between the exposed and the unexposed portions of the specimen or the standard pattern 7 is equal to grey scale grade 4.

7.1.1.3 If the standard pattern 7 fades to a contrast equal to grey scale grade 4 before the specimen does, terminate the exposure at this stage. If the specimen fades to a contrast equal to grey scale grade 4 before the standard pattern 7 does, replace the cover *AB* exactly in the previous position, cover the left hand one-third of the specimen and the standard patterns with another opaque cover (*CD* in Fig. 1) and continue to expose until the contrast between the fully exposed and unexposed portions of the specimen is equal to grey scale grade 3. At this stage, terminate the exposure.

NOTE — When a specimen has light fastness rating equal to or greater than 7, it would require unduly long exposure to produce a contrast equal to grey scale grade 3; moreover, this contrast would be impossible to obtain when the light fastness rating is 8. Assessment in the region of 7 to 8 are made, therefore, when the contrast produced on standard pattern 7 is equal to grey scale grade 4, the time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.

7.1.1.4 Assess the light fastness of the specimen as given in 8.

7.1.2 Method 2

7.1.2.1 Arrange the specimens to be tested and the standard patterns as shown in Fig. 2 with an opaque cover *AB*, covering one-quarter of the total length of each specimen and standard pattern. Expose to daylight under the conditions described in 6.1. Follow the effect of light by lifting cover *AB* periodically and inspecting the

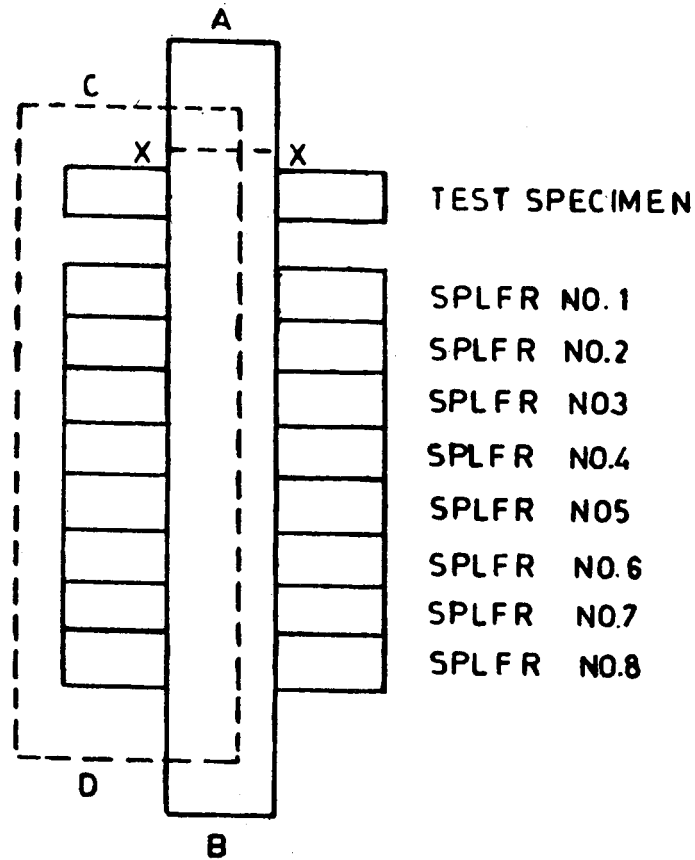


FIG. 1 MOUNTING OF TEST SPECIMEN AND STANDARD PATTERNS FOR METHOD 1

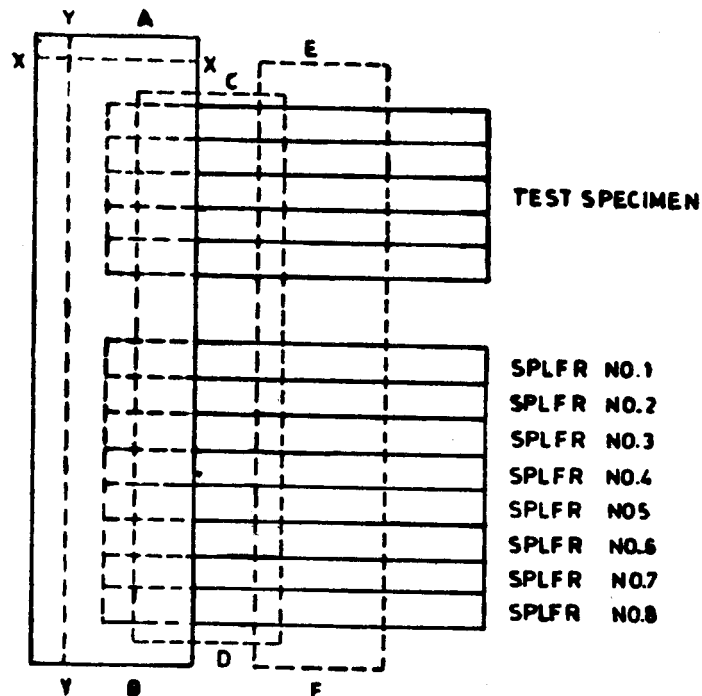


FIG. 2 MOUNTING OF TEST SPECIMENS AND STANDARD PATTERNS FOR METHOD 2

standard patterns. When a change in colour of the exposed portion of the standard pattern 3 can just be perceived, equal to grey scale grade 4-5, inspect the specimens and rate their light fastness by comparing any change that has occurred with the changes that have occurred in standard patterns 1, 2 and 3 (This is a preliminary assessment of light fastness.)

7.1.2.2 Replace the lifted cover *AB* in exactly the same position and continue to expose until a change in colour of the exposed portion of the standard pattern 4 can just be perceived, equal to grey scale grade 4-5; at this stage, fix an additional cover *CD* in the position shown in Fig. 2, overlapping the cover *AB*.

7.1.2.3 Continue to expose until a change in colour of the exposed portion of the standard pattern 6 can just be perceived, equal to grey scale grade 4-5; then fix the final cover *EF* in the position shown in Fig. 2, the other three covers remaining in position. Expose until either

- a) a contrast is produced on standard pattern 7 equal to the contrast illustrated by grey scale grade 4, or
- b) a contrast equal to grey scale grade 3 on the grey scale has been produced on the most resistant specimen,

whichever occurs first.

7.1.2.4 Compare the changes in the colour of the specimens and those of the standard patterns and assess the light fastness of each specimen as given in 8.

7.2 Other Permissible Exposure

Where the test is to be used to check conformity with a performance specification, it is permissible to expose the specimens with two standard patterns only; that specified as minimum and the one below it. Continue exposure until grey scale grade 4 and grey scale grade 3 contrasts have been produced on separate areas of the minimum standard pattern.

8 ASSESSMENT OF LIGHT FASTNESS

8.1 The final assessment in numerical ratings is based on contrasts equal to grey scale grade 4 and/or grade 3 between exposed and unexposed portions of the specimen(s).

8.2 Compare the change in colour of the specimen with that of the standard patterns under suitable illumination. The light fastness of the specimen is the number of the standard pattern which shows similar changes in colour (visual contrast between exposed and unexposed parts of the specimen). If the specimen shows changes in colour which are nearer to the imaginary standard pattern midway between any two

consecutive standard patterns, the intermediate rating, for example, 3-4 shall be given.

NOTES

1 In cases of doubt in the colour fastness rating as assessed by an observer, assessment may be made by three observers and the overall average rating may be reported.

2 The term change in colour includes not only true fading, that is, destruction of dyes but also changes in hue, depth, brightness or any combination of these characteristics of colour. If the difference in colour is a change of hue or brightness, this may be indicated by adding abbreviations as follows to the numerical fastness ratings:

Bl = bluer, Y = yellower, D = duller, and
R = redder, G = greener, Br = brighter.

If the change in hue is accompanied by change in depth, this may also be indicated as follows:

W = weaker, and Str = stronger.

3 In order to avoid a misrating of the light fastness of the specimen due to photochromism, the phototropic specimens shall be allowed to condition in the dark at room temperature for 24 h before the light fastness is assessed.

8.3 If different assessments are obtained at different degrees of contrast, the light fastness of the specimen is the arithmetic mean of these expressed to the nearest whole or half grade. When three areas are being rated, take the mean of the contrasts closest to grades 4 and 3. Assessments, however, shall be confined to whole or midway ratings only. When the arithmetic mean gives a quarter or three quarter rating, the assessment is defined as the next higher half or whole grade.

8.4 If the colour of the specimen is more fugitive than that of the standard pattern 1, the rating shall be 1.

8.5 Comparison of the changes in the specimen with changes in the standard pattern may be facilitated by surrounding the specimen with a mask of neutral grey colour (approximately the shade of the lightest chip of the grey scale for assessing change in colour), and the standard patterns, in turn, with a similar mask of equal aperture.

8.6 If the light fastness is equal to or higher than 4, any preliminary assessment based on the contrast equal to grey scale 4-5 (see 7.1.1.1 and 7.1.2.1) becomes significant; if this preliminary assessment is 3, it shall be included in the rating in brackets. For example, rating of 6 (3) indicates that the specimen changes very slightly in the test when standard pattern 3 just begins to fade but that on continuing the exposure, the resistance to light is equal to that of standard pattern 6.

8.7 If specimens larger than the standard patterns were exposed because of certain other requirements, a mask of a neutral grey colour midway between grade 1 and grade 2 of the grey scale (approximately Munsell N5) should be used in assessment which covers the surplus area of the specimens leaving an area equal to that of the standard patterns open for comparative evaluation.

9 REPORT

9.1 Report individually the numerical light fastness rating of specimens.

9.2 If the rating is equal to 4 or higher and the preliminary assessment is equal to 3 or lower, report the latter figure in brackets.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ARTIFICIAL LIGHT (XENON LAMP)

(Source : IS 2454 : 1985)

Colour fastness of textile materials to daylight is of considerable importance to the consumer. As the daylight exposure takes long time to complete a test, a quick method of determining colour fastness to light is by the use of artificial light (xenon lamp). However, there are cases where the results obtained by artificial light exposure differ from the results obtained with daylight exposure. In case of dispute, method based on daylight exposure should be used.

Two methods for mounting and testing of specimens for colour fastness to artificial light have been prescribed in this standard Method 1

is considered preferable and should be used in cases of dispute. But it requires one set of standard patterns for each test specimen and is, therefore, impracticable when a large number of test specimens have to be tested concurrently. Method 2 enables a number of test specimens of differing light fastness ratings to be rated against a single set of standard patterns. This method is used when a large number of test specimens have to be tested concurrently. The methods prescribed are based on ISO 105/B-1984 Textiles — Tests for colour fastness F02 — Colour fastness to light : Xenon arc, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 These methods are intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of artificial light source, namely, xenon arc lamp.

2 PRINCIPLE

2.1 A specimen of the textile material is exposed under prescribed condition along with eight dyed wool standard patterns to the light from a xenon arc. The fastness is assessed by comparing the change in colour of the specimen with that of the standard patterns.

3 SAMPLING

3.1 Sample shall be drawn from the lot so as to be representative of the lot. Samples drawn in accordance with the material specification for the coloured textile material or as agreed to between the buyer and the seller shall be representative of the lot.

4 PREPARATION OF TEST SPECIMENS

4.1 If the material to be tested is fabric, draw a specimen of area not less than $1\text{ cm} \times 4.5\text{ cm}$ from each piece in the test sample.

4.2 If the material to be tested is yarn, wind enough of the sample closely on a card or lay it parallel and fasten on a card to make a test specimen of the area not less than $1\text{ cm} \times 4.5\text{ cm}$ taking care that all knots present in the yarn, while preparing the test specimen, are under the back of the card. Draw one such specimen from each piece in the test sample.

4.3 If the material to be tested is loose fibre, comb and compress enough of the sample into the form of a sheet to make a test specimen of area not less than $1\text{ cm} \times 4.5\text{ cm}$ and fasten it

on a card. Draw one such specimen from each piece in the test sample.

4.4 To facilitate handling, the specimen(s) and strips of standard patterns of similar size may be mounted on a card in some such arrangement as indicated in Fig. 1 or 2. Each exposed and unexposed area shall not be less than $1\text{ cm} \times 1\text{ cm}$.

4.5 The specimens and the standard patterns should be of equal size and shape in order to avoid errors in assessment due to over-rating the visual contrast between exposed and unexposed parts on the longer specimens as against the narrower standard patterns.

4.6 When testing carpets, the standard patterns shall be arranged in such a way that they are at the same distance from the light source as the surface of the carpet specimens. This can be achieved by underlaid cardboard.

5 STANDARD PATTERN

5.1 Standard patterns of blue wool cloth ranging in light fastness from rating No. 1 (very low light fastness) to rating No. 8 (very high light fastness) and dyed with the dyes, as prescribed below shall be used:

Fastness Rating No.	Dye [Colour Index (CI), Designation (see Note 1)]
1	CI acid blue 104
2	CI acid blue 109
3	CI acid blue 83
4	CI acid blue 121
5	CI acid blue 47
6	CI acid blue 23
7	CI solubilized vat blue 5 (see Note 2)
8	CI solubilized vat blue 8 (see Note 2)

NOTES

1 The Colour Index (third edition) published by the Society of Dyers and Colourists, P.O. Box 244, Perkins House, 83 Gratton Road, Bradford BD1 2JB, West Yorks, United Kingdom.

2 The patterns dyed with these dyes should be decatized after dyeing.

3 Sets of standard patterns of light fastness rating No. 1 to 8 may be obtained from the Bureau of Indian Standards or the British Standards Institution. They are specially dyed to match a master set of standard patterns in colour and in fading behaviour. It has been found that, when repeated dyeings for the dyed standard patterns are made, the amount of dye required to match a previous lot is often different from that originally used. The dyeing strengths would, therefore, be misleading and they are intentionally omitted from this list.

5.2 The humidity test control of red azoic dyed cotton cloth of an area not less than $1\text{ cm} \times 4.5\text{ cm}$ shall be used.

NOTE — The humidity test control may be prepared by impregnating bleached mercerized poplin with 4 g/l brenthol AN (CI azoic coupling component 4) and developing with 20 g/l brentamine fast scarlet R salt (CI azoic diazo component 13). The above humidity test control may be obtained from the Bureau of Indian Standards.

6 APPARATUS

6.1 Light Source

Xenon arc lamp of correlated colour temperature of 5 500 to 6 500 K.

6.2 Light Filter

A filter to be placed between the light source and the assembly of test specimen(s) and standard patterns so that the ultraviolet spectrum is steadily reduced. Window glass may be used as filter. The glass used shall have a transmission of at least 90 percent between 380 and 750 nm, falling to zero percent between 310 and 320 nm.

6.3 Opaque Cardboard or Opaque Material

Such as thin sheet aluminium or cardboard covered with aluminium foil or, in the case of pile fabrics, a cover that avoids surface compression.

6.4 Grey Scale

For assessing the change in colour.

6.5 Heat Filters

The spectrum of xenon arc contains an appreciable amount of infra red radiations, which should be minimized by heat filters. The temperature conditions (see 7.1.1 and 7.1.2) can then be satisfied. The filters should be cleaned regularly to avoid undesirable reduction in light intensity by dirt.

6.6 Black Panel Thermometer

See Note 2 under 7.1.2.

7 EXPOSURE CONDITIONS

7.1 The test specimens are exposed in a well ventilated exposure chamber.

7.1.1 Normal Conditions

7.1.1.1 Moderate effective humidity

Light fastness of the humidity test control 5, black-panel temperature, *Max* 45°C.

7.1.2 Extreme Conditions

For testing the sensitivity of specimens to humidity, the following extreme conditions are useful:

a) Low effective humidity

light fastness of the humidity test control 6-7, black-panel temperature, *Max* 60°C.

b) High effective humidity

light fastness of the humidity test control 3, black-panel temperature, *Max* 40°C.

NOTES

1 The variation of the light intensity over the area covered by the test specimens and standard patterns should not exceed ± 10 percent of the mean.

2 The black panel thermometer should consist of a metal panel at least $4.5 \times 10\text{ cm}$ whose temperature is measured with a thermometer or a thermocouple with its sensitive portion located in good contact with the panel. The side of the panel facing the light source shall be black with a reflectance of less than 5 percent throughout the spectrum reaching the specimen, the side of the panel not facing the light source shall be thermally isolated.

8 PROCEDURE COMMON TO METHODS 1 AND 2

8.1 Humidity Control

Expose partially covered strips of the humidity test control and standard patterns mounted on to a card simultaneously until a contrast is produced on the humidity test control equal to grade 4 on the grey scale. Assess the light fastness of the humidity test control and, if necessary, adjust the controls on the lamp to give the selected exposure conditions (see 7.1.1 and 7.1.2).

NOTE — Ensure that the strip of humidity test control is mounted uppermost in the sample holder.

8.2 Mounting and Testing of Test Specimens

8.2.1 Expose the specimen(s) and the standard patterns simultaneously for 24 hours per day under the conditions enumerated in 7.1.1 or 7.1.2 in such a manner and for such times as are necessary to evaluate fully the light fastness of each test specimen relative to that of the standard patterns, by successively covering both the test specimens and exposed standard patterns throughout the test duration (either by Method 1 or Method 2).

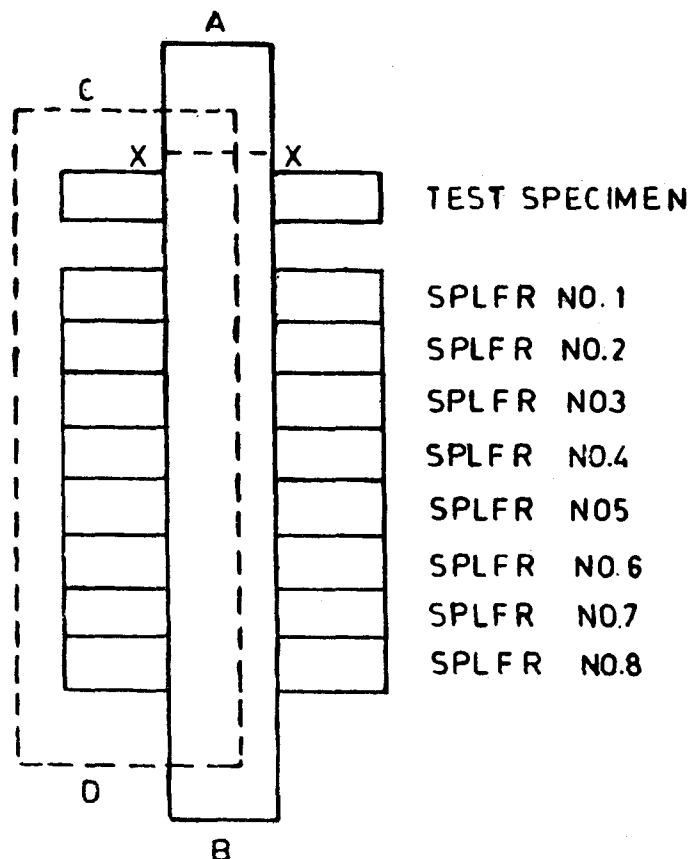


FIG. 1 MOUNTING OF TEST SPECIMEN AND STANDARD PATTERNS FOR METHOD 1

8.3 Method 1

8.3.1 Mount the test specimen and the standard patterns as shown in Fig. 1 with an opaque cover *AB* across the middle one-third of the specimen and the standard patterns. Expose the specimen and the standard patterns to artificial light under the conditions enumerated in 7.1.1 or 7.1.2. Follow the effect of light by lifting the cover *AB* and inspecting the specimen frequently. When a change in colour of the exposed portion of the specimen can just be perceived equal to grey scale grade 4-5, note the number of the standard pattern showing a similar change. (This is preliminary assessment of light fastness of the specimen).

8.3.2 Continue to expose the specimen and the standard patterns until the contrast between the exposed and the unexposed portions of the specimen or the standard pattern 7 is equal to grey scale grade 4.

8.3.3 If the standard pattern 7 fades to a contrast equal to grey scale grade 4 before the specimen does, terminate the exposure at this stage. If the specimen fades to a contrast equal to grey scale grade 4 before the standard pattern 7 does, replace the cover *AB* exactly in the previous position, cover the left hand one-third

of the specimen and the standard patterns with another opaque cover (*CD* in Fig. 1) and continue to expose until the contrast between the fully exposed and unexposed portions of the specimen is equal to grey scale grade 3. At this stage, terminate the exposure.

NOTE — When a test specimen has light fastness rating of 7 or higher, it would require unduly long exposure to produce a contrast equal to grade 3 on the grey scale. Moreover, this contrast would be impossible to obtain when the light fastness rating is 8. Assessments in the region 7 to 8 are made, therefore, when the contrast produced on standard pattern 7 is equal to grade 4 on the grey scale. The time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.

8.3.4 Assess the light fastness of the specimen as given in 8.5.

8.4 Method 2

8.4.1 Mount the test specimens and the standard patterns as shown in Fig. 2. Cover one quarter of the total length of each test specimen and standard pattern with opaque cover *AB*. Expose the assembly to artificial light under the conditions enumerated in 7.1.1 or 7.1.2. Follow the effect of light by lifting the cover *AB* periodically and inspecting the standard patterns. When a

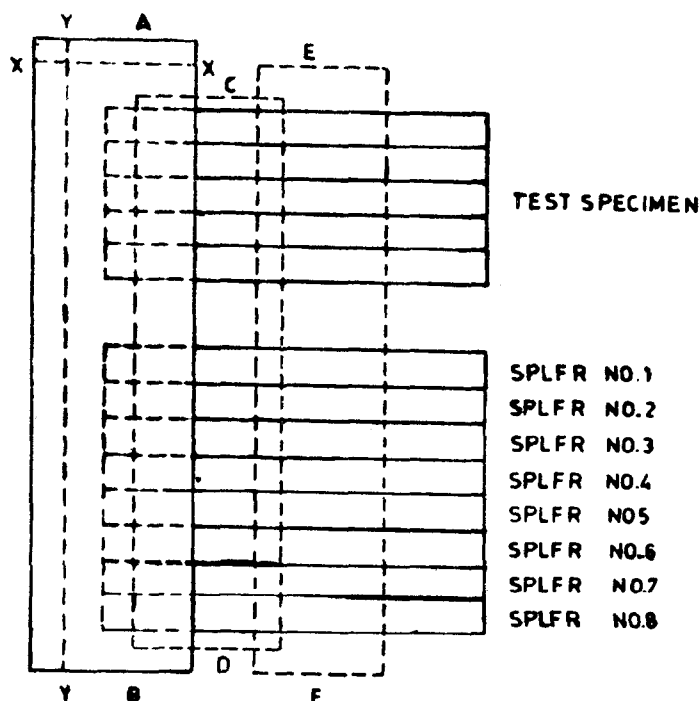


FIG. 2 MOUNTING OF TEST SPECIMENS AND STANDARD PATTERNS FOR METHOD 2

change in colour of the exposed portion of the standard pattern 3 can just be perceived equal to grey scale grade 4-5, inspect the specimens and assess their preliminary light fastness by comparing any change that has occurred with the changes that have occurred in standard patterns 1, 2 and 3.

8.4.2 Replace the opaque cover *AB* in exactly the same position and continue to expose until a change in colour of the exposed portion of the standard pattern 4 can just be perceived, equal to grey scale grade 4-5. Place an additional opaque cover *CD* in the position as shown in Fig. 2, overlapping the first cover *AB*.

8.4.3 Continue to expose until a change in colour of the exposed portion of the standard pattern 6 can just be perceived equal to grey scale grade 4-5. Place an opaque cover *EF* in the position as shown in Fig. 2, the other two covers remaining in position.

8.4.4 Expose the assembly until either:

- a contrast is produced on standard pattern 7 equal to the contrast illustrated by grade 4 on the grey scale, or
- a contrast equal to grade 3 on the grey scale has been produced on the most resistant specimen,

whichever occurs first.

Remove the three covers and conclude the experiment.

8.4.5 Compare the changes in the colour of the specimens and those of the standard patterns and assess the light fastness of each specimen as given in 8.5.

8.5 Assessment of Light Fastness

8.5.1 The final assessment in numerical ratings is based on contrast equal to grey scale grade 4 and/or grade 3 between exposed and unexposed portions of the specimen(s).

8.5.2 Compare the change in colour of the specimen with the changes which have occurred in the standard patterns under suitable illumination. The light fastness of the specimen is the number of standard pattern which shows similar changes in colour (visual contrast between the exposed and unexposed portions of the specimen). If the specimen shows changes in colour approximately half way between two standard patterns, the intermediate rating, for example, 3-4 shall be given.

NOTES

1 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment may be made by three observers and the overall average rating may be reported.

2 The term 'change in colour' includes not only true fading, that is, destruction of dyes but also changes in hue, depth, brightness or any combination of these characteristics of colour. If the difference in colour is a change of hue or brightness, this may

be indicated by adding abbreviations as follows to the numerical fastness ratings:

Bl = bluer, Y = yellower, D = duller, and
R = redder, G = greener, Br = brighter.

If the change in hue is accompanied by change in depth, this may also be indicated as follows:

W = weaker, and Str = stronger.

3 In order to avoid a misrating of the light fastness of the specimen due to photochromism, the phototropic specimens shall be allowed to remain in the dark at room temperature for 24 h before the light fastness is assessed.

8.5.3 If the different assessments are obtained at the different degrees of contrast, the light fastness of the specimen is the arithmetic mean of these, expressed to the nearest whole or half grade. When three areas are being rated, take the mean of the contrasts closest to grade 4 and 3. Assessments, however, shall be confined to whole or midway ratings only. When the arithmetic mean gives a quarter or three-quarter rating, the assessment is defined as the next higher half or whole grade.

8.5.4 If the colour of the specimen is more fugitive than that of standard pattern No. 1, a rating of 1 shall be given.

8.5.5 If the light fastness is of rating 4 or higher, assign the test specimen its light fastness rating number but also add within parenthesis its

preliminary light fastness rating number, if any (see 8.3.1).

Example

A light fastness rating of 6 (2) indicates that the test specimen changes very slightly in colour when the standard pattern of light fastness rating No. 2 just begins to fade but that on continuing the exposure, the resistance to light of the test specimen is equal to that of the standard pattern of light fastness rating No. 6.

8.5.6 If specimens larger than the standard patterns were exposed because of certain other requirements, a mask of a neutral grey colour midway between the grade 1 and grade 2 of the grey scale (approximately Munsell N5) should be used in assessment which covers the surplus area of the specimens leaving an area equal to that of the standard patterns open for comparative evaluation.

9 REPORT

9.1 Report, individually, the numerical light fastness rating of test specimens.

9.2 If the rating is equal to 4 or higher and the preliminary assessment is 3 or lower, report the latter figure in brackets.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WEATHERING BY OUTDOOR EXPOSURE

(Source : IS 5951 : 1985)

Two methods for mounting and testing of specimens for colour fastness to weathering by outdoor exposure have been prescribed. Method 1 is considered preferable and should be used in cases of dispute but it requires one set of standard patterns for each test specimen and is, therefore, impracticable when a large number of test specimens have to be tested concurrently. Method 2 enables a larger number of test speci-

mens of differing light fastness ratings to be rated against a single set of standard patterns. This method is used when a large number of test specimens have to be tested concurrently. The methods prescribed are based on ISO 105/B-1984 Textiles—Test for colour fastness B03—Colour fastness to weathering: Outdoor exposure, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 These methods are intended for the determination of colour fastness of textile materials of all kinds and in all forms except loose fibres to the action of weathering by outdoor exposure.

2 PRINCIPLE

2.1 Specimens of textiles are exposed under specified conditions in the open air without any protection from weathering. At the same time and in the same place, eight standard patterns of blue wool cloth are exposed to daylight but are protected from rain, snow, etc, by a sheet of window glass. The fastness is assessed by comparing the change in colour of the specimen with that of the standard patterns.

2.1.1 The wide variations in conditions under which outdoor exposures are usually carried out, make it desirable to make replicate exposures starting at different times of the year. The most reliable indication of weathering fastness is obtained by taking the mean of several exposures.

3 SAMPLING

3.1 Samples shall be drawn from the lot so as to be representative of the lot. Samples drawn as prescribed in material specification for the coloured textile materials or as agreed to between the buyer and the seller to evaluate the colour fastness of the material in the lot shall be representative of the lot.

4 PREPARATION OF TEST SPECIMENS AND STANDARD PATTERNS

4.1 If the material to be tested is a fabric, draw two specimens from each lot measuring at least 10 cm × 4 cm and sew along each side on to a piece of scoured, undyed cloth made of hydrophobic fibre such as polyester or acrylic.

4.1.1 If the material to be tested is yarn, knit or weave it into a fabric, then treat it as in 4.1. If facilities do not exist for knitting or weaving the yarn into fabric, the yarn may be wound over an inert material covered with a piece of scoured, undyed cloth made of hydrophobic fibre, such as polyester or acrylic. Draw two such specimens from each lot.

NOTE — Loose fibres are not suitable for weathering tests.

4.2 Standard Patterns

Mount strips of light fastness standard patterns (see 5) of 10 cm × 4 cm size on cardboard and cover the middle one-third (see Fig. 1 and 2) with an opaque cardboard.

NOTES

1 To facilitate handling, the test specimens and strips of the standard patterns of similar size may be mounted on a card in some such arrangement as indicated in Fig. 1 or 2. Each exposed and unexposed area shall not be less than 1 cm × 1 cm.

2 The test specimens and the standard patterns should be of equal size and shape in order to avoid errors in assessment due to over-rating the visual contrast between exposed and unexposed parts on the larger specimens as against the narrower standard patterns.

3 When testing carpets, the standard patterns shall be arranged in such a way that they are at the same distance from the light source as the surface of the carpet specimens. This can be achieved by underlaid cardboard.

5 STANDARD PATTERNS

5.1 Standard patterns of blue wool cloth with light fastness ranging from rating No. 1 (very low light fastness) to rating No. 8 (very high light fastness) and dyed with the

dyes, as prescribed below, shall be used for this test:

<i>Fastness Rating No</i>	<i>Dye [Colour Index (CI), Designation (see Note 1)]</i>
1	CI acid blue 104
2	CI acid blue 109
3	CI acid blue 83
4	CI acid blue 121
5	CI acid blue 47
6	CI acid blue 23
7	CI solubilized vat blue 5 (see Note 2)
8	CI solubilized vat blue 8 (see Note 2)

NOTES

1 The Colour Index (third edition) published by the Society of Dyers and Colourists, P.O. Box 244, Parkins House, 83 Gratton Road, Bradford BD1 2 JB, West Yorks, UK.

2 The patterns dyed with these dyes should be decatized after dyeing.

3 Sets of standard patterns of light fastness rating No. 1 to 8 may be obtained from the Bureau of Indian Standards or the British Standards Institution. They are specially dyed to match the master set of standard patterns in colour and in fading behaviour. It has been found that, when repeated dyeings for the dyed standard patterns are made, the amount of dye required to match a previous lot is often different from the originally used. The dyeing strengths would, therefore, be misleading and they are intentionally omitted from this list.

6 APPARATUS

6.1 Exposure Rack for Specimens

The rack should be facing due south in the Northern hemisphere or due North in the Southern hemisphere and sloping at an angle from the horizontal approximately equal to the latitude of the place where the exposure is made. It should be located in open at such a place that the shadow of surrounding objects do not fall on the exposed textiles, and constructed such that the specimens or the cloths on which the specimens are sewn or wound are firmly held. There should be free circulation of air behind the mounted specimens.

NOTE — The test may be carried out, preferably, in a non-residential and non-industrial area free from dust and automobile exhaust fumes.

6.2 Exposure Rack for Standard Patterns

The rack should be oriented similarly as given in 6.1 and covered with a glass to protect the standard patterns from weathering, the glass being not less than 5 cm from the standard patterns and shall be provided with adequate ventilation. The glass shall have a transmission

of at least 90 percent between 380 and 750 nm, falling to zero percent between 310 and 320 nm.

6.3 Opaque Cardboard or Thin Opaque Material

For example, a thin sheet of aluminium or cardboard covered with aluminium foil or, in the case of pile fabrics, a cover that avoids surface compression. It is used for covering the standard patterns.

6.4 Grey Scale

for evaluating the change in colour.

7 PROCEDURE COMMON TO METHODS 1 AND 2

7.1 Firmly attach the specimens or the cloth to which the specimens have been sewn or wound, to the exposure rack (see 6.1). Place the mounted and partially covered standard patterns in the glass covered rack (see 6.2). Expose the specimens and the standard patterns simultaneously 24 h per day for such times as are necessary to evaluate the weathering fastness using either Method 1 or Method 2 (see 7.2 and 7.3).

7.2 Method 1

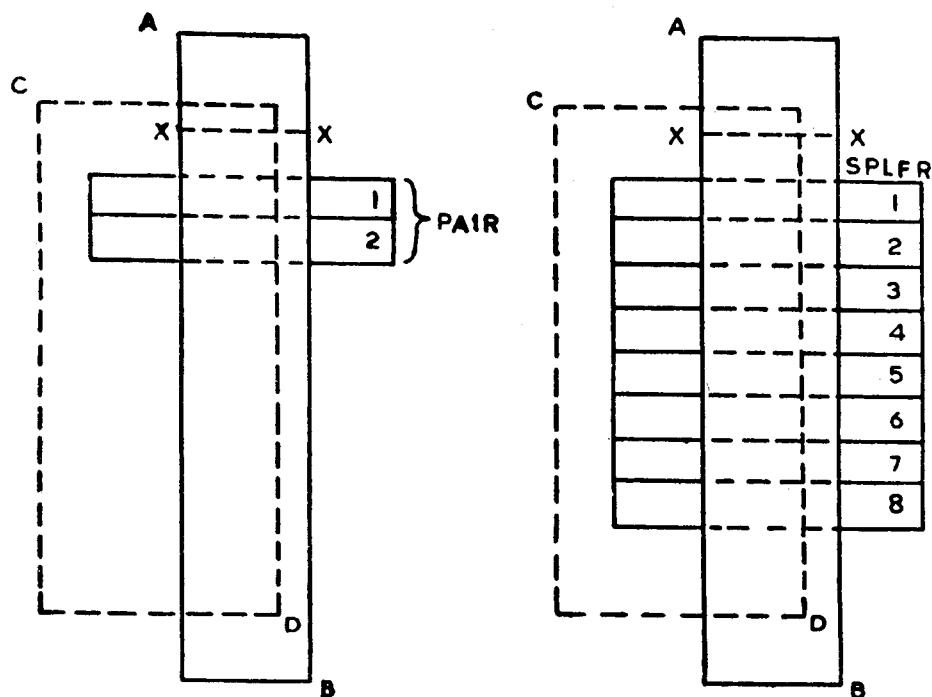
7.2.1 Expose the specimens and the standard patterns (see Fig. 1) under the conditions as given in 7.1 until the contrast between the exposed specimens and a portion of the original fabric is equal to grade 3 on the grey scale. Remove one of the specimens and cover the left hand one-third of the standard patterns with an additional opaque cover.

7.2.2 Continue the exposure until the contrast between the remaining specimen and a portion of the original fabric is equal to grade 2 on the grey scale. If standard pattern 7 fades to a contrast equal to grade 4 on the grey scale before the contrast between the specimen and a portion of the original fabric is equal to grade 2 on the grey scale, the exposure may be concluded at this stage and the remaining specimen and the standard patterns removed.

7.2.3 Wash both specimens as well as a portion of the original fabric and prepare them for assessment to weathering fastness as given in 7.4 and 7.5 respectively.

7.3 Method 2

7.3.1 Expose the specimens and the standard patterns (see Fig. 2) under the conditions given in 7.1 until the contrast between the exposed and the unexposed portion of standard pattern 6 is equal to grade 4 on the grey scale. At this stage, remove one specimen from each pair and cover the left hand one-third of the standard patterns with an additional opaque cover.



1A Exposure of Test Specimens

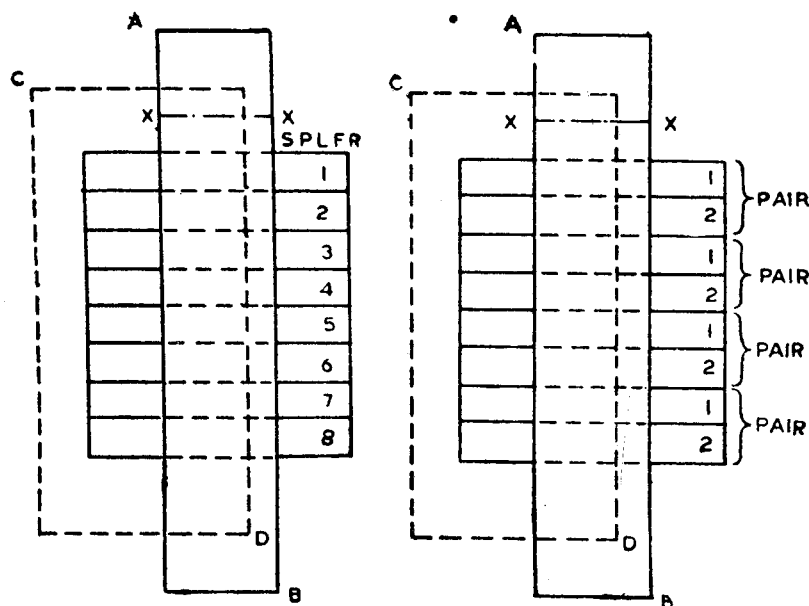
1B Exposure of Standard Patterns

SPLFR — Standard patterns for light fastness rating

AB — Opaque cover — may be hinged at *XX* so that it may be lifted and returned to the same place over the standard patterns

CD — Another opaque cover

FIG. 1 MOUNTING OF TEST SPECIMENS AND STANDARD PATTERNS FOR METHOD 1



2A Exposure of Standard Patterns

2B Exposure of Test Specimens

SPLFR — Standard patterns for light fastness rating

AB — Opaque cover — may be hinged at *XX* so that it may be lifted and returned to the same place over the standard patterns

CD — Another opaque cover

FIG. 2 MOUNTING OF STANDARD PATTERNS AND TEST SPECIMENS FOR METHOD 2

7.3.2 Continue the exposure until the contrast between the fully exposed and unexposed portion of standard pattern 7 is equal to grade 4 on the grey scale. Remove the remaining specimens and the standard patterns.

7.3.3 Wash the exposed specimens and a portion of the original fabric from each specimen and prepare them for assessment to weathering fastness as given in 7.4 and 7.5 respectively.

7.4 Washing of Test Specimens

7.4.1 Wash the exposed specimens and a portion of the original fabric measuring at least 10 cm × 4 cm (in the absence of undyed cloth) under the conditions specified in Annex A.

7.4.2 Trim and mount the washed specimens on each side of the washed original fabric which has been trimmed to the same size and shape as the specimens. The specimens exposed for shorter length of time should be mounted on the left.

7.5 Assessment of Fastness

7.5.1 First Assessment

Compare the magnitude of the contrast between the specimen exposed for the shorter time and the original fabric in terms of the contrasts produced on the standard patterns exposed for the same period. The assessment is the number of the standard pattern showing the contrast closest to that of the specimen. If the specimen shows a change in colour approximately half way between the two standard patterns, an appropriate half rating, for example, 5-6 shall be given.

7.5.2 Second Assessment

Compare the magnitude of the contrast between the specimen exposed for the longer time and the original fabric in terms of the contrast produced in the standard patterns exposed for the same period. The assessment is the number of the standard pattern showing the contrast closest to that of the specimen. If the specimen shows

a change in colour approximately half way between two standard patterns, an appropriate half rating, for example, 3-4 shall be given.

NOTES

1 The terms 'change in colour' includes not only true fading, that is destructions of dyes, but also changes in the hue, depth, brightness or any combination of these characteristics of colour. If the difference in colour is a change of hue or brightness, this may be indicated by adding abbreviations as follows to the numerical fastness rating:

Bl = bluer Y = yellower D = duller

R = redder G = greener Br = brighter

If the change in hue is accompanied by change in depth, this may also be indicated as follows:

W = weaker Str = stronger

2 Phototropic test specimens should be allowed to condition in dark at room temperature for 24 hours before evaluating the change in colour.

3 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.5.3 If specimens larger than the standard patterns are exposed, a mask of a neutral grey colour approximately midway between grade 1 and grade 2 of the grey scale for assessing change in colour (approximately Munsell NS) should be used in the assessment which should cover the surplus area of the specimen and leaving an area equal to that of the standard patterns open for comparative evaluation.

8 REPORT

8.1 The report shall include the following:

- a) The numerical ratings for weathering fastness by outdoor exposure (if the two assessments carried out in 7.5.1 and 7.5.2 are different, report only the lower one); and
- b) Place of exposure and the time of the year.

ANNEX A

METHOD FOR WASHING OF TEST SPECIMENS

A-1 APPARATUS

A-1.1 A suitable mechanical washing device conforming to the following requirements shall be used:

- a) A water bath containing a rotor with which containers (of glass or of stainless steel) are rotated at a speed of 40 ± 2 rev/min.
- a) Means for thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $40 \pm 2^\circ\text{C}$.

NOTE — The Wash-wheel sponsored by the Society of Dyers and Colourists or the Launderometer sponsored by the American Association of Textile Chemists and Colorists or any other mechanical apparatus giving identical results may be used for these tests.

A-2 SOAP SOLUTION

A-2.1 It shall contain 5 g of soap per litre. The soap shall contain not more than 5 percent moisture and shall comply with the following requirement based on dry weight.

The soap shall be free from fluorescent brightening agents:

Free alkali calculated as Na_2CO_3 (<i>m/m</i>) (<i>Max</i>), percent	0.3
Free alkali calculated as NaOH (<i>m/m</i>) (<i>Max</i>), percent	0.1
Combined fatty acids calculated as sodium salt (<i>m/m</i>) (<i>Min</i>), percent	85
Titre of mixed fatty acids prepared from the soap (<i>Max</i>)	39°C
Iodine value of fatty acids (<i>Max</i>)	50

A-3 PROCEDURE

A-3.1 Place one of the specimens in a container and add to it the necessary amount of soap solution previously heated to $40 \pm 2^\circ\text{C}$ to give a liquor ratio of 1 : 50. Treat the specimen for 30 minutes at $40 \pm 2^\circ\text{C}$ in the mechanical washing device. Remove the specimen, rinse it twice in cold water and then in cold running tap water for 10 minutes. Squeeze it and dry in air at a temperature not exceeding 60°C .

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WEATHERING BY XENON ARC LAMP

(Source : IS 6152 : 1985)

This standard is based on ISO 105/B-1984 Textiles — Test for colour fastness B04 — Colour fastness to weathering — Xenon arc, issued by the International Organization for Standardization (ISO).

Colour fastness of textile materials to weathering (out-door exposure) is of considerable strategic importance to consumers especially in case of defence textile stores like camouflage material, tents and tarpaulins, which are especially dyed and printed so as to mix with the surrounding colours of nature. As the out-door exposure method takes long time to complete a test, a quick method of determining colour fastness to weathering is by use of artificial light (xenon lamp) in simulated weather conditions. However,

there is likelihood that the results obtained by this method may differ from the results obtained by out-door exposure. In case of dispute, the latter method should be followed.

Two methods for mounting and testing of specimens for colour fastness to weathering by xenon arc lamp have been prescribed in this standard. Method 1 is considered preferable and should be used in cases of dispute but it requires one set of standard patterns for each test specimen. Method 2 enables a number of test specimens of differing light fastness ratings to be rated against a single set of standard patterns. This method is used when a large number of test specimens have to be tested concurrently.

1 SCOPE

1.1 These methods are intended for the determination of colour fastness of textile materials of all kinds and in all forms except loose fibres to the action of weathering as simulated by exposure in a cabinet equipped with xenon arc lamp.

2 PRINCIPLE

2.1 Specimens of textiles are exposed under specified conditions to light from a xenon arc lamp and to water spray. At the same time and in the same cabinet, eight standard patterns of blue wool cloth are exposed to light but are protected from water spray by a sheet of window glass. The fastness is assessed by comparing the change in colour of the textile with that of the standard patterns.

3 SAMPLING

3.1 Samples shall be drawn from the lot so as to be representative of the lot. Samples drawn as prescribed in material specification for the coloured textile materials or as agreed to between the buyer and the seller, shall be representative of the lot.

4 PREPARATION OF TEST SPECIMENS AND STANDARD PATTERNS

4.1 Test Specimens

If the material to be tested is fabric, draw two test specimens from each lot, each of area not less than $1\text{ cm} \times 4.5\text{ cm}$ mounted on holders or other equipment which will fit the weathering test equipment.

4.1.1 If the material to be tested is yarn, it should be knitted or woven into a fabric and then treated as in 4.1. If facilities do not exist for knitting or weaving the yarn into fabric, the yarn may be wound over an inert material covered with a piece of scoured, undyed cloth made of hydrophobic fibre such as polyester or acrylic. Draw two such specimens from each lot.

NOTE — Loose fibres are not suitable for weathering tests.

4.2 Standard Patterns

Mount strips of light fastness standard patterns (see 5) on a cardboard and cover the middle one-third (see Fig. 1 and 2) with an opaque cardboard.

NOTES

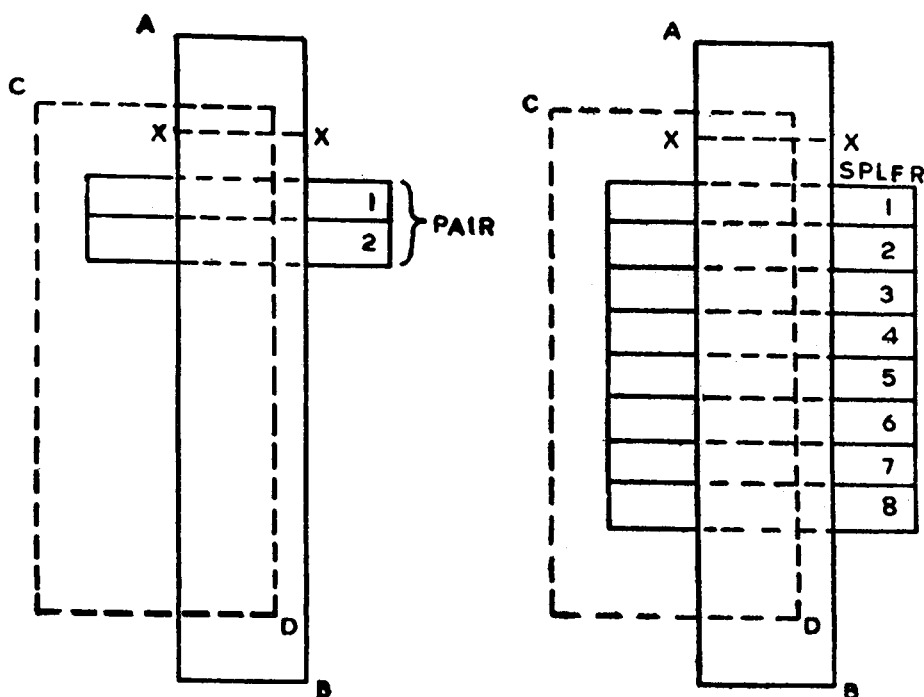
1 To facilitate handling, the test specimens and strips of standard patterns of similar size may be mounted on a card in some such arrangement as indicated in Fig. 1 or 2. Each exposed and unexposed area shall not be less than $1\text{ cm} \times 1\text{ cm}$.

2 The test specimens and the standard patterns should be of equal size and shape in order to avoid errors in assessment due to over rating the visual contrast between exposed and unexposed parts on the larger patterns as against the narrower standard patterns.

3 When testing carpets, the standard patterns shall be arranged in such a way that they are at the same distance from the light source as the surface of the carpet specimens. This can be achieved by underlaid cardboard.

5 STANDARD PATTERNS

5.1 Standard patterns of blue wool cloth with light fastness ranging from rating No. 1



1A Exposure of Test Specimens

1B Exposure of Standard Patterns

FIG. 1 MOUNTING OF TEST SPECIMENS AND STANDARD PATTERNS FOR METHOD 1

(very low light fastness) to rating No. 8 (very high light fastness) and dyed with the dyes as prescribed below, shall be used for this test:

Fastness Rating No.	Dye [Colour Index (CI), Designation (see Note 1)]
1	CI acid blue 104
2	CI acid blue 109
3	CI acid blue 83
4	CI acid blue 121
5	CI acid blue 47
6	CI acid blue 23
7	CI solubilized vat blue 5 (see Note 2)
8	CI solubilized vat blue 1 (see Note 2)

NOTES

1 The Colour Index (third edition) published by the Society of Dyers and Colourists, P.O. Box 244, Parkins House, 83 Gratton Road, Bradford BD1 2 JB, West Yorks, U.K.

2 The patterns dyed with these dyes should be decatized after dyeing.

3 Sets of standard patterns of light fastness rating No. 1 to 8 may be obtained from the Bureau of Indian Standards or the British Standards Institution. They are specially dyed to match the master set of standard patterns in colour and in fading behaviour. It has been found that, when repeated dyeings for the dyed standard patterns are made, the amount of dye required to match a previous lot is often different from that originally used. The dyeing strength would, therefore, be misleading and they are intentionally omitted from this list.

6 APPARATUS

6.1 Light Source

It shall be xenon arc lamp having correlated colour temperature between 5 500 and 6 500 K kept in well ventilated exposure chamber.

6.2 Light Filter

A special kind of glass possessing transparency of 90 percent between 380 and 750 nm and which is opaque between 310 and 320 nm is placed as filter between the light source and the assembly of the test specimen(s) and standard patterns so that ultraviolet spectrum is steadily reduced.

6.3 Heat Filters

These are used to minimize the infra-red radiation (which is present in appreciable amount) of the spectrum of the xenon arc to satisfy temperature conditions. They should be cleaned

regularly to avoid undesirable reduction in light intensity by dirt.

6.4 Opaque Cardboard or Thin Opaque Material

It shall be a thin sheet of aluminium or cardboard covered with aluminium foil or in the case of pile fabrics a cover that avoids surface compression.

6.5 Grey Scale

for evaluating the change in colour.

6.6 Black Panel Thermometer

See Note under 7.1.2.

7 EXPOSURE CONDITIONS

7.1 The following exposure conditions should be satisfied during tests.

7.1.1 The variation of the light intensity over the area covered by the test specimens and standard patterns shall not exceed ± 10 percent of the mean.

7.1.2 The specimens shall be exposed in a well-ventilated exposure chamber. The air temperature in the chamber shall not exceed 40°C during the drying period. The temperature shall be measured with a thermometer whose bulb is shielded from the direct radiation of the arc. The temperature of the black panel which is measured in the same position and under the same illumination as the specimen shall not exceed that of the test chamber by more than 20°C at the maximum drying period.

NOTE — The black panel thermometer should consist of a metal panel at least 4.5 cm × 10 cm whose temperature is measured with a thermometer or a thermocouple with its sensitive portion located in the centre and in good contact with the panel. The side of the panel facing the light source shall be black with a reflectance of less than 5 percent throughout the spectrum reaching the specimen; the side of the panel not facing the light source shall be thermally isolated.

7.1.3 The specimens mounted in rotating holders shall be subjected to an accurately adjusted and reproducible weathering cycle of:

- a) length of spraying — 1 min (rain period), and
- b) length of drying — 29 min (drying period).

NOTES

1 For spraying the specimens, completely ion-free water shall be used. It should be especially noted that this water must not contain any metal salts. Tubing, tanks and spray jets must be made of corrosion resistant material.

2 The specimens should completely enclose the holder and the side to be assessed should not be in contact with metal plates, other specimens or backing fabric.

8 PROCEDURE COMMON TO METHODS 1 AND 2

8.1 Place the test specimen(s) mounted on the holders in the frame (see Fig. 1) and expose them to weathering.

8.2 At the same time, expose the mounted and partially covered blue wool standard patterns (see 4.2) to light in the glass case of the same frame. The standard patterns shall be protected from the water spray by a shield of window glass whilst being exposed to light. The window glass used should have transparency of at least 90 percent between 380 and 750 nm and be completely opaque between 310 and 320 nm. The glass case should be well ventilated, that is, there should be an opening on the top and bottom which allows a good circulation of air.

8.3 Expose the specimens and the standard patterns simultaneously for 24 hours per day, for such times as are necessary to evaluate the weathering fastness, using either Method 1 or Method 2 for mounting the test specimens and standard patterns (see 8.4 and 8.5).

NOTES

1 Only one side of the specimen shall be exposed to weathering and light.

2 Whilst specimens are drying, the air in the test chamber shall not be moistened.

3 The conditions of the weathering test depend on the kind of test apparatus used.

4 Contrary to stipulations for the outdoor exposure test, the specimens must not be washed after the weathering test.

8.4 Method 1

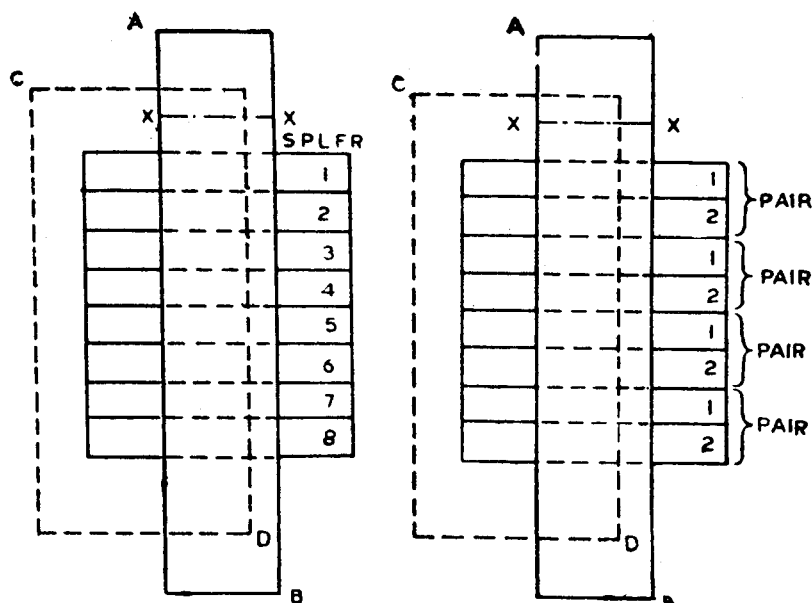
8.4.1 Expose the specimens and the standard patterns (see Fig. 1) under the conditions given in 8.1 to 8.3, until the contrast between the exposed specimens and portion of the original fabric is equal to grade 3 on the grey scale. Remove one of the specimens and cover the left hand one-third of the standard patterns with an additional opaque cover.

8.4.2 Continue the exposure until the contrast between the remaining specimen and a portion of the original fabric is equal to grade 2 on the grey scale. If Standard Pattern 7 fades to a contrast equal to grade 4 on the grey scale before the contrast between the specimen and the portion of the original fabric is equal to grade 2 on the grey scale, the exposure may be concluded at this stage and the remaining specimen and the standard patterns removed.

8.4.3 Prepare the specimens as well as a portion of the original fabric for assessment to weathering fastness (see 8.5.4 and 8.5.5) and assess as given in 8.6.

8.5 Method 2

8.5.1 Expose the specimens and the standard patterns (see Fig. 2) under the conditions given



2A Exposure of Standard Patterns 2B Exposure of Test Specimens

FIG. 2 MOUNTING OF STANDARD PATTERNS AND TEST SPECIMENS FOR METHOD 2

in 8.1 to 8.3, until the contrast between the exposed and unexposed portion of standard pattern 6 is equal to grade 4 on the grey scale. At this stage, remove one specimen from each pair and cover the left hand one-third of the standard patterns with an additional opaque cover.

8.5.2 Continue the exposure until the contrast between the fully exposed and unexposed portion of Standard Pattern 7 is equal to grade 4 on the grey scale. Remove the remaining specimens and the standard patterns.

8.5.3 Prepare the exposed specimens as well as a portion of the original fabric from each specimen for assessment to weathering fastness (see 8.5.4 and 8.5.5) and assess as given in 8.6.

8.5.4 Dry the specimens in air at a temperature not exceeding 60°C before mounting for assessment.

8.5.5 Trim and mount the tested specimens measuring at least 15 mm × 30 mm, one on each side of a portion of the original fabric which has been trimmed to the same size and shape as the specimens. The specimen exposed for shorter length of time should be mounted on the left.

8.6 Assessment of Fastness

8.6.1 First Assessment

Compare the magnitude of the contrast between the specimen exposed in the shorter time and

original fabric in terms of the contrasts produced on the standard patterns exposed for the same period. The assessment is the number of standard pattern(s) showing the contrast closest to that of the specimen. If the specimen shows the change in colour approximately half way between the two standards patterns, an appropriate half rating, for example, 5-6, shall be given.

NOTES

1 The term 'change in colour' includes not only true fading, that is, destruction of dyes but also changes in hue, depth, brightness or any combination of these characteristics of colour. If the difference in colour is a change of hue or brightness, this may be indicated by adding abbreviations as follows to the numerical fastness ratings:

Bl = bluer Y = yellower D = duller,
R = redder G = greener Br = brighter

If the change in hue is accompanied by change in depth, this may also be indicated:

W = weaker, and Str = stronger.

2 Phototropic test specimens should be allowed to condition in dark at room temperature for 24 hours before evaluating the change in colour.

3 In case of doubt in the colour fastness rating as assessed by an observer, the assessment may be made by three observers and the overall average rating may be reported accordingly.

8.6.2 If specimen larger than the standard patterns are exposed, a mask of neutral grey colour midway between grade 1 and grade 2 of the grey scale (approximately Munsell N5) should be

used in the assessment covering the surplus area of the specimens and leaving an area equal to that of the standard patterns open for comparative evaluation.

9 REPORT

9.1 The report shall include the following:

- a) Numerical ratings of weathering fastness — Xenon lamp.

NOTE — If the two assessments (see 8.6.1 and 8.6.2) are different, report only the lower one.

- b) Model of the apparatus used for the test.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO RUBBING WITH ORGANIC SOLVENTS

(Source : IS 3426 : 1982)

This standard is based on ISO 105/D-1982 Textiles — Tests for colour fastness D02 — Colour fastness to rubbing : Organic solvents, issued by

the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms, except loose fibres to the combined action of rubbing and of organic solvents used in spot cleaning, that is, localized spotting carried out by hand.

2 PRINCIPLE

2.1 A specimen of textile is rubbed with undyed rubbing cotton cloth impregnated with solvent, and the change in colour of the specimen and the staining of rubbing cotton cloth are assessed with grey scale.

3 SAMPLING

3.1 The sample of yarns or fabrics shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedures laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 TEST SPECIMEN

4.1 Preparation of Test Pieces

4.1.1 If the material to be tested is fabric, draw from each piece in the test sample two pairs of test specimens, each test specimen in the pair not less than 14×5 cm. One pair shall have the long direction parallel to the warp yarns and the other parallel to the weft yarns.

NOTE — If the material to be tested is of knitted fabric, one pair of the test pieces shall have long direction parallel to the wales and the other pair to the courses.

4.1.2 If the material to be tested is yarn, knit or weave the test sample into a fabric and draw from it six test pieces, each not less than 14×5 cm; alternatively, prepare the test pieces to form a layer of the parallel lengths by wrapping it lengthwise on a glass plate of suitable dimensions.

5 APPARATUS

5.1 Suitable Testing Device

an apparatus equipped with a finger of 1.6 cm diameter moving to and fro in a straight line

along a 10 cm track on the specimen with a downward force of 9 N.

5.2 Rubbing Cotton Cloth

desized, bleached, free from finishes and cut into squares of 5×5 cm.

5.3 Grating of Stainless Steel Wire

made out of stainless steel wire of 1 mm diameter and a width of mesh of about 20 cm.

5.4 Grey Scales

for assessing change in the colour and staining.

6 REAGENTS

6.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the experimental results.

6.1 Perchloroethylene

6.2 White Spirit (Solvent F) (or Some Other Petroleum Hydrocarbon)

7 PROCEDURE

7.1 Soak the rubbing cotton cloth (5.2) in the solvent; squeeze it to retain approximately its own mass of appropriate solvent. Alternatively, wet the rubbing cotton cloth by placing it on the grating (5.3) and dropping levelly on to it, its own mass of appropriate solvent.

7.2 Place the rubbing cotton cloth soaked with the solvent at the end of the finger of the apparatus and rub it to and fro in a straight line along a track 10 cm long on the test piece, having a longer side parallel to the warp, 10 times in 10 seconds with a downward force of 9 N on the finger.

NOTE — If the test piece is of knitted fabric, a test piece having longer side parallel to wales shall be taken for testing.

7.2.1 Treat one test specimen with the longer side parallel to the weft in a similar manner using a fresh piece of rubbing cotton cloth.

NOTE — If the test specimen is of knitted fabric, a test specimen having longer side parallel to courses shall be taken for testing.

7.3 Dry the pieces of rubbing cotton cloth at a temperature not exceeding 60°C.

7.4 Repeat the test prescribed in 7.1 to 7.3 with the remaining two test specimens using white spirit (solvent F) or some other petroleum hydrocarbon in place of perchloroethylene.

7.5 Repeat the test prescribed in 7.1 to 7.4 with the test specimen drawn from the remaining pieces in the test sample.

7.6 If the material to be tested is yarn, test three test specimens with perchloroethylene and the remaining three test specimens with white spirit (solvent F) (or some other petroleum hydrocarbon).

7.7 Evaluate the change in colour of each test specimen and the degree of staining of each piece of rubbing cotton cloth with the help of grey scales and assign the ratings. If the test pieces drawn from the fabric have been used in the test, evaluate the degree of staining of that piece of rubbing cotton cloth which shows heavier staining.

8 REPORT

8.1 Report, individually,

- a) the solvent used,
- b) the numerical ratings for change in colour of the specimens, and

- c) the numerical ratings for staining of piece of rubbing cotton cloth for the direction (warp or weft) showing heavier staining.

9 NOTES

9.1 It is necessary to eliminate dyed fibres pulled out during rubbing and retained on the surface of the rubbing cotton cloth; consider only the colouration due to staining by the dyestuffs.

9.2 A suitable apparatus is the Crockmeter, described in the Technical Manual of the American Association of Textile Chemists and Colorists. Test Methods 8-1972 (Vol 50, 1974, p 112). Other devices may be used, provided the same results are obtained as with the apparatus described in 5.1.

9.3 The finger of the apparatus can be replaced by a moving hollow tube ending in a grill at its base. A plug of cotton is placed in this tube. The outside of the grill is covered with a sample of wool flannel.

With apparatus modified in this way, it is no longer necessary to immerse the rubbing cotton cloth in the solvent (6.1); the dry rubbing cotton cloth is placed on the wool flannel at the end of the tube and 3 ml of the solvent are dropped on to the plug of cotton on the inside of the hollow tube. Then proceed as described in 7.2 onwards.

9.4 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO DRY-CLEANING

(Source : IS 4802 : 1988)

The method prescribed in this standard is based on ISO 105/D-1984 Textiles — Tests for colour fastness D01 — Colour fastness to dry cleaning, issued by the International Organization for Standardization (ISO).

In India, different solvents are used for dry-cleaning of textiles though perchloroethylene is considered as ideal for many of the textiles. Determination of colour fastness of such textiles to dry-cleaning is of considerable importance to the consumer.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to dry-cleaning.

1.2 The test prescribed in this standard is not suitable for evaluation of the durability of textile finishes, nor it is intended for use in evaluating the resistance of colours to spot and stain removal procedures used by the dry-cleaners. (see Notes 1 and 2).

NOTES

1 This test covers colour fastness to dry-cleaning only; commercial dry-cleaning practice normally involves other operations, such as water spotting, solvent spotting and steam pressing, for which other standard test methods are available if the full 'dry-clearability' of the textile is to be assessed.

2 The presence of absorbed water in either the fabric or dry-cleaning solution, or the presence of a detergent water in the dry-cleaning solution, has not been found to be a critical factor in assessing colour fastness. The test gives results which correlate satisfactorily with those obtained in commercial dry-cleaning.

2 PRINCIPLE

2.1 A specimen of the textile in contact with a cotton fabric bag together with non-corrodible steel discs is agitated in dry-cleaning solvent, then squeezed or centrifuged and dried in hot air. The change in colour of the specimen is assessed with the grey scale for change in colour. At the conclusion of the test, the discolouration of the solvent is also assessed by comparing the filtered solvent with unused solvent by transmitted light by means of grey scale for staining.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 PREPARATION OF TEST SPECIMEN

4.1 If the textile to be tested is fabric, draw a test specimen of 10 cm × 4 cm size.

4.2 If the material to be tested is yarn, knit or weave it into fabric and draw from it 10 cm × 4 cm test specimen or make a wick of parallel lengths 10 cm long and about 0.5 cm in diameter, tied near both ends.

4.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm × 4 cm.

5 APPARATUS

5.1 Suitable Mechanical Device

consisting of water bath containing a rotatable shaft which supports, radially, glass or stainless steel containers (75 ± 5 mm diameter × 125 ± 10 mm high) of approximately 550 ± 50 ml capacity, the bottom of the containers being 45 ± 10 mm high from the centre of the shaft. The shaft/container assembly is rotated at a speed of 40 ± 2 rev/min. The temperature of the water bath is thermostatically controlled to maintain the test solvent at 30 ± 2°C.

NOTE — Other mechanical devices may also be used, provided the results are identical to those obtained by the apparatus described above.

5.2 Glass or Stainless Steel Containers

75 ± 5 mm diameter, 125 ± 10 mm high and of 550 ± 50 ml capacity, which shall be closed using solvent resistant gaskets.

5.3 Non-corrodible (Stainless) Steel Discs

30 ± 2 mm × 3 ± 0.5 mm, smooth and free from rough edges of mass 20 ± 2 g.

5.4 Undyed Cotton Twill Cloth

of mass per unit area 270 ± 70 g/m², free from finishes and cut into samples 12 cm × 12 cm.

5.5 Grey Scales

for evaluating change in colour and staining.

5.6 Glass Tubes

of diameter 25 mm.

6 REAGENTS

6.1 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall

be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6.2 Perchloroethylene

or any other solvent used for dry-cleaning.

NOTE — Perchloroethylene should be stored over anhydrous sodium carbonate to neutralize any hydrochloric acid formed.

7 PROCEDURE

7.1 Prepare a bag with inside dimensions of 10×10 cm using the undyed cotton twill cloth by sewing together two squares of this cloth around three sides. Place the specimen and 12 steel discs inside the bag. Close the bag by any convenient means.

7.2 Place the bag containing the specimen and the steel discs in the container and add 200 ml of the dry-cleaning solvent at $30 \pm 2^\circ\text{C}$. Treat the specimen for 30 min at $30 \pm 2^\circ\text{C}$ in the specified equipment (see 5.1).

7.3 Remove the bag from the container, withdraw the specimen, place it between absorbent paper or cloth and squeeze or centrifuge to remove surplus solvent. Dry the specimen by

hanging in air in shade at a temperature of $60 \pm 5^\circ\text{C}$.

7.4 Evaluate the change in colour of the treated test specimen with grey scale (see Notes under 7.5).

7.5 At the conclusion of the test, filter the solvent remaining in the container through filter paper. Compare the colour of the filtered solvent with that of unused solvent in the glass tube placed in front of white card using transmitted light by means of grey scale for assessing staining.

NOTES

1 The treated test specimens should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In case of doubt in the colour fastness ratings as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report the numerical rating for change in colour of the test specimen and the numerical ratings for staining of the solvent.

8.2 Report the solvent used for the test.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING : TEST 1

(Source : IS 687 : 1979)

This method is designed to determine the effect of washing only on the colour fastness of the textiles. It is not intended to reflect the result of the comprehensive laundering procedures. The method prescribed is based on

ISO 105/C-1982 Textiles — Tests for colour fastness C01 — Colour fastness to washing: Test 1, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials irrespective of their composition (cotton, linen, silk, man-made fibres, wool) and their form (fibre, yarn or fabric) to the action of solution at 40°C. (Test 1 of a series of five washing tests.)

2 PRINCIPLE

2.1 A specimen of textile in contact with pieces of specified adjacent fabrics is mechanically agitated in soap solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with the grey scales.

3 SAMPLING

3.1 The sample shall be so drawn as to be the representative of the lot. Sample drawn in accordance with the procedures laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used (see also Annex A):

- A water bath containing a rotor with which containers (of glass or of stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min, and
- Means of thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $40 \pm 2^\circ\text{C}$.

4.2 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends; the second piece made of the fibre as indicated below; and in the case of blends, of the kind of fibre second in order of predominance (see also Annex A of IS 10251 : 1982):

If First Piece is

Cotton
Wool
Silk
Linen
Viscose
Acetate
Polyamide
Polyester
Acrylic

Second Piece to be

Wool
Cotton
Cotton
Cotton or viscose
Wool
Viscose
Wool or viscose
Wool or cotton
Wool or cotton

4.3 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Soap Solution

The soap solution shall contain 5 g of soap per litre. The soap shall conform to the requirements as given below, with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

Characteristic	Requirement
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acids prepared from the soap (Max)	39°C
Iodine value of fatty acid (Max)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen of 10 cm × 4 cm between the two adjacent fabrics (*see* 4.2) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (*see* 4.2), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabric (*see* 4.2) into a sheet 10 × 4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare at least three composite test specimens.

7 PROCEDURE

7.1 Place one composite specimen in a container and add to it the necessary amount of soap solution previously heated to $40 \pm 2^\circ\text{C}$ to give a liquor ratio of 1 : 50. Treat the composite specimen for 30 minutes at $40 \pm 2^\circ\text{C}$ in the mechanical washing device. Remove the composite specimen, rinse it twice in cold water and then in cold running tap water for ten minutes. Squeeze it. Remove the stitchings along the two

long sides and one short side (*see* Note). Open out the composite specimen and dry in air at a temperature not exceeding 60°C with the three pieces (test specimen and two adjacent fabrics) in contact only along the remaining line of stitching.

NOTE — The stitching along the two diagonals, if any, should also be removed.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of Grey scales and assign the ratings.

NOTES

1 Treated test specimens and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture contents before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Report the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test piece, and
- c) Numerical ratings for staining of the two adjacent fabrics.

ANNEX A

MECHANICAL WASHING DEVICE

A-1 Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

A-1.1 Mention of the name of specific or proprietary instrument is not intended to promote or give preference to the use of that instrument over others not mentioned. This is only for the guidance of the users.

*Available with the Silk and Art Silk Mills Industry's Research Association, Bombay.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING : TEST 2

(Source : IS 3361 : 1979)

This method is designed to determine the effect of washing only on the colour fastness of the textiles. It is not intended to reflect the result of the comprehensive laundering procedure.

The method prescribed is based on ISO 105/C-1982 Textiles — Tests for colour fastness C02 — Colour washing : Test 2, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials irrespective of their composition (cotton, linen, silk, wool), man-made fibres and their form (fibre, yarn or fabric) to the action of soap solution at 50°C (Test 2 of a series of five washing tests).

2 PRINCIPLE

2.1 A specimen of textile in contact with specific adjacent fabrics is mechanically agitated in soap solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 The sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedures laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used (*see also* Annex A):

- A water bath containing a rotor with which containers (of glass or stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min, and
- Means of thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $50 \pm 2^\circ\text{C}$.

4.2 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends; the second piece made of the fibre as indicated in the following table; and in the case of blends, of the kind of fibre second in order of predominance (*see also* Annex A of IS 10251 : 1982):

If First Piece is

Cotton
Wool
Silk
Linen
Viscose
Acetate
Polyamide
Polyester
Acrylic

Second Piece to be

Wool
Cotton
Cotton
Cotton or viscose
Wool
Viscose
Wool or viscose
Wool or cotton
Wool or cotton

4.3 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean the chemicals that do not contain impurities which affect the test results.

5.2 Soap Solution

The soap solution shall contain 5 g of soap per litre. The soap shall conform to the requirements as given below with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

<i>Characteristic</i>	<i>Requirement</i>
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acids prepared from the soap (Min)	39°C
Iodine value of fatty acids (Max)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen of 10×4 cm between the two

adjacent fabrics (*see* 4.2) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (*see* 4.2), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (*see* 4.2) into a 10 × 4 cm sheet. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare at least three composite test specimens.

7 PROCEDURE

7.1 Place one composite specimen in a container and add to it the necessary amount of soap solution previously heated to $50 \pm 2^\circ\text{C}$ to give a liquor to material ratio of 1 : 50. Treat the composite specimen for 45 minutes at $50 \pm 2^\circ\text{C}$. Remove the composite specimen, rinse it twice in cold water and then in cold running tap water for ten minutes. Squeeze it. Remove the stitchings along the two long sides and one short side (*see* Note). Open out the composite specimen and

dry in air at a temperature not exceeding 60°C with the three pieces (test specimen and two adjacent fabrics) of the specimen in contact only along the remaining line of stitching.

NOTE — The stitching along the two diagonals, if any, should also be removed.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimens and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test piece, and
- c) Numerical ratings for staining of the two adjacent fabrics.

ANNEX A

MECHANICAL WASHING DEVICE

A-1 Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

*Available with the Silk and Art Silk Mills Industry's Research Association, Bombay.

A-1.1 Mention of the name of a specific or proprietary instrument is not intended to promote or give reference to the use of that instrument over others not mentioned. This is only for the guidance of the users.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING : TEST 3

(Source : IS 764 : 1979)

This method is designed to determine the effect of washing only on the colour fastness of the textiles. It is not intended to reflect the result of the comprehensive laundering procedure. The

method prescribed is based on ISO 105/C-1982 Textiles — Tests for colour fastness C03 — Colour fastness to washing : Test 3, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of all textile materials irrespective of their composition (cotton, linen, silk, manmade fibre, etc) and their form (fibre, yarn or fabric) to the action of soap and sodium carbonate solution at 60°C. (Test 3 of a series of five washing tests).

2 PRINCIPLE

2.1 A specimen of textile in contact with pieces of specified adjacent fabrics is mechanically agitated in a soap soda solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with standard grey scales.

3 SAMPLING

3.1 The sample shall be so drawn as to be the representative of the lot. Sample drawn in accordance with the procedure laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used (see also Annex A):

- a) A water bath containing a rotor with which containers (of glass or of stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min, and
- b) Means of thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $60 \pm 2^\circ\text{C}$.

4.2 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind fibre as that of the textile to be tested, or that predominating in the case of blends; the second piece made of the fibre as indicated below; and in the case of blends, of the kind of fibre second in order of predominance (see also Annex A of IS 10251 : 1982):

If First Piece is

Second Piece to be

Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Cotton or viscose
Viscose	Wool
Acetate	Viscose
Polyamide	Wool or viscose
Polyester	Wool or cotton
Acrylic	Wool or cotton

4.3 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemical shall mean the chemicals that do not contain impurities which affect the test results.

5.1 Soap-Soda Solution

The solution shall contain 5 g of soap and 2 g of anhydrous sodium carbonate per litre. The soap shall conform to the requirements as given below, with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

Characteristic	Requirement
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acids prepared from the soap (Max)	39°C
Iodine value of fatty acids (Max)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen 10×4 cm between the two adjacent fabrics (*see 4.2*) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (*see 4.2*), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (*see 4.2*) into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare the least three composite test specimens.

7 PROCEDURE

7.1 Place one composite specimen in the container. Add the necessary amount of soap-soda solution previously heated to $60 \pm 2^\circ\text{C}$, to give a liquor ratio of 1 : 50. Treat the composite specimen for 30 minutes at $60 \pm 2^\circ\text{C}$ in the mechanical washing device. Remove the composite specimen, rinse it twice in cold water and then in cold running tap water for 10 minutes and squeeze it. Remove the stitching along the two long sides

and one short side (*see Note*). Open out the composite specimen and dry in air at a temperature not exceeding 60°C with three pieces (test specimen and two adjacent fabrics) in contact only at the line of stitching.

NOTE — The stitching along the two diagonals, if any, should also be removed.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scale and assign the ratings.

NOTES

1 Treated test specimens and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test piece, and
- c) Numerical rating for staining of the two adjacent fabrics.

ANNEX A

MECHANICAL WASHING DEVICE

A-1 Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

A-1.1 Mention of the name of a specific or proprietary instrument is not intended to promote or give preference to the use of that instrument over others not mentioned. This is only for the guidance of the users.

*Available with the Silk and Art Silk Mills Industry's Research Association, Bombay.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING : TEST 4

(Source : IS 765 : 1979)

This method is designed to determine the effect of washing only on the colour fastness of the textiles. It is not intended to reflect the result of the comprehensive laundering procedures. The

method prescribed is based on ISO 105/C-1982 Textiles — Tests for colour fastness C04 — Colour fastness to washing : Test 4, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials irrespective of their composition (cotton, linen, man-made fibres, etc) and their form (fibre, yarn or fabric) to the action of soap and sodium carbonate solution at 95°C along with beating action of steel balls. It is applicable only to those fibres which are capable of withstanding such conditions (Test 4 of a series of five washing tests).

2 PRINCIPLE

2.1 A specimen of textile in contact with specified adjacent fabrics is mechanically agitated in a soap-soda solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 The sample, as the case may be, shall be so drawn as to be the representative of the lot. Sample drawn in accordance with the procedures laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used (*see also* Annex A):

- A water bath containing a rotor with which containers (of glass or stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min, and
- Means of thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $95 \pm 2^\circ\text{C}$.

4.2 Stainless Steel Balls

approximately 6 mm in diameter.

4.3 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of

blends; the second piece made of the fibre as indicated below; and in the case of blends of the kind of fibre second in order of predominance (*see also* Annex A of IS 10251 : 1982):

If First Piece is

Second Piece to be

Cotton	Viscose
Linen	Viscose
Viscose	Cotton
Acetate	Viscose
Polyamide	Cotton or viscose
Polyester	Cotton or viscose
Acrylic	Cotton or viscose

4.4 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean the chemicals that do not contain impurities which affect the test results.

5.1 Soap-Soda Solution

The soap solution shall contain 5 g of soap and 2 g of anhydrous sodium carbonate per litre of water. The soap shall conform to the requirements as given below, with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

<i>Characteristic</i>	<i>Requirement</i>
Free alkali calculated as Na_2CO_3 (<i>Max</i>), percent	0.3
Free alkali calculated as NaOH (<i>Max</i>), percent	0.1
Combined fatty acids calculated as Na salts (<i>Min</i>), percent	85
Titre of mixed fatty acids prepared from the soap (<i>Max</i>)	39°C
Iodine value of fatty acids (<i>Max</i>)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen 10×4 cm between the two adjacent fabrics (see 4.3) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as 6.1, or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (see 4.3), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (see 4.3) into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare at least three composite test specimens.

7 PROCEDURE

7.1 Place one composite specimen with 10 stainless steel balls in the container. Add the necessary amount of soap-soda solution previously heated to $95 \pm 2^\circ\text{C}$, to give a liquor ratio of 1:50. Treat the composite specimen for 30 minutes at $95 \pm 2^\circ\text{C}$. Remove the composite specimen, rinse it twice in cold water and then in cold running tap water for 10 minutes and squeeze it. Remove the

stitching along the two long sides and one short side (see Note). Open out the composite specimen and dry in air at a temperature not exceeding 60°C with three pieces (test specimen and two adjacent fabrics) of the specimen in contact only at the line of stitching.

NOTE — The stitching along the two diagonals, if any, should be removed.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimen and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In case of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Report the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical rating for change in colour of the test piece, and
- c) Numerical rating for staining of the two adjacent fabrics.

ANNEX A

MECHANICAL WASHING DEVICE

A-1 Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

A-1.1 Mention of the name of a specific or proprietary instrument is not intended to promote or give preference to the use of that instrument over others not mentioned. This is only for the guidance of the users.

*Available with the Silk and Art Silk Mills Industry's Research Association, Bombay.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING : TEST 5

(Source : IS 3417 : 1979)

This method is designed to determine the effect of washing only on the colour fastness of the textiles. It is not intended to reflect the result of the comprehensive laundering procedures. The

method prescribed is based on ISO 105/C-1982 Textiles — Tests for colour fastness C05 — Colour fastness to washing: Test 5, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials irrespective of their composition (cotton, linen, man-made fibres, etc) and their form (fibre, yarn or fabric) to the action of soap and sodium carbonate solution at 95°C for four hours along with beating action of steel balls. It is applicable only to those fibres which are capable of withstanding such conditions (Test 5 of a series of five washing tests).

2 PRINCIPLE

2.1 A specimen of textile in contact with specified adjacent fabrics is mechanically agitated in a soap-soda solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with standard grey scales.

3 SAMPLING

3.1 The sample, as the case may be, shall be so drawn as to be the representative of the lot. Sample drawn in accordance with the procedures laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used (*see also* Annex A):

- A water bath containing a rotor with which containers (of glass or stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min, and
- Means of thermostatically controlling the temperature of water bath so as to maintain temperature of test solution in the containers at $95 \pm 2^\circ\text{C}$.

4.2 Stainless Steel Balls

approximately 6 mm in diameter.

4.3 Two Adjacent Fabrics

each measuring 13×4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of

blends; the second piece made of the fibre as indicated below; and in the case of blends of the kind of fibre second in order of predominance (*see also* Annex A of IS 10251 : 1982):

<i>If First Piece is</i>	<i>Second Piece to be</i>
Cotton	Viscose
Linen	Viscose
Viscose	Cotton
Acetate	Viscose
Polyamide	Cotton or viscose
Polyester	Cotton or viscose
Acrylic	Cotton or viscose

4.4 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean the chemicals that do not contain impurities which affect the test results.

5.1 Soap-Soda Solution

The soap solution shall contain 5 g of soap and 2 g anhydrous sodium carbonate per litre of water. The soap shall conform to the requirements as given below with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

<i>Characteristic</i>	<i>Requirement</i>
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acids prepared from the soap (Max)	39°C
Iodine value of fatty acids (Max)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen 10×4 cm between the two adjacent fabrics (see 4.3) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (see 4.3), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (see 4.3) into a sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare at least three composite test specimens.

7 PROCEDURE

7.1 Place one composite specimen with 10 stainless steel balls in the container. Add the necessary amount of soap-soda solution previously heated to $95 \pm 2^\circ\text{C}$ to give liquor ratio of 1 : 50. Treat the specimen for four hours at $95 \pm 2^\circ\text{C}$. Remove the composite specimen, rinse it twice in cold water and then in cold running tap water for 10 minutes. Squeeze it. Remove the stitchings along the two long sides and one short side

(see Note). Open out the composite specimen and dry in air at a temperature not exceeding 60°C with the three pieces (test specimen and two adjacent fabrics) of the specimen in contact only along the remaining line of stitching.

NOTE — The stitching along the two diagonals, if any, should be removed.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimen and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test piece, and
- c) Numerical ratings for staining of the two adjacent fabrics.

ANNEX A

MECHANICAL WASHING DEVICE

A-1 Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

A-1.1 Mention of the name of a specific or proprietary instrument is not intended to promote or give preference to the use of that instrument over others not mentioned. This is only for the guidance of the users.

*Available with the Silk and Art Silk Mills Industry's Research Association, Bombay.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WASHING IN THE PRESENCE OF SODIUM HYPOCHLORITE

(Source : IS 984 : 1988)

The colour fastness of textile materials is affected when they are washed at high temperatures in the presence of sodium hypochlorite. The method of test prescribed in this standard is intended to evaluate the colour fastness of textile materials, when treated thus.

Since certain fibres (for example, protein fibres like wool) are damaged by such treatment, this method is applicable only to those fibres which are capable of withstanding such conditions.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to washing at high temperatures in the presence of sodium hypochlorite. It is applicable only to those fibres which are capable of withstanding such conditions.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is agitated in a solution of soap, sodium carbonate and sodium hypochlorite, rinsed and dried. The numerical ratings for change in colour of the specimen and staining of the adjacent fabrics are evaluated with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of same kind of fibre as that of the textile to be tested or that predominating in the case of blends the second piece made of the fibre as indicated below, or in the case of blends, of the kind of fibre second in order of predominance, or as otherwise specified (see Annex A of IS 10251 : 1982):

<i>If First Piece is</i>	<i>Second Piece to be</i>
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Cotton
Viscose	Wool
Acetate	Viscose
Polyamide	Wool or viscose
Polyester	Wool or cotton
Acrylic	Wool or cotton

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, draw a 10 cm × 4 cm specimen from the sample and place it between the two pieces of adjacent fabrics (see 4.1). Sew around all four sides to form a composite specimen.

4.2.2 If the textile to be tested is yarn, knit or weave it into fabric, draw from it a 10 cm × 4 cm specimen and place it between the two pieces of adjacent fabrics (see 4.1). Sew around all four sides to form a composite specimen. Alternatively taking an amount of yarn weighing approximately half the combined mass of the pieces of adjacent fabrics (see 4.1), prepare a 10 cm × 4 cm specimen in the form of parallel lengths of yarn. Place it between the two pieces of adjacent fabrics and sew around all four sides to hold the yarn in place and to form a composite specimen.

4.2.3 If the textile to be tested is loose fibre, comb and compress an amount of fibre weighing approximately half the combined mass of the pieces of adjacent fabrics (see 4.1) into a sheet to make a 10 cm × 4 cm specimen. Place the sheet between the two pieces of adjacent fabrics and sew around all four sides to form a composite specimen.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6 APPARATUS

6.1 Suitable Mechanical Device

consisting of a water bath containing a rotatable shaft which supports, radially, glass or stainless steel containers (75 ± 5 mm diameter \times 125 ± 10 mm high) of approximately 550 ± 50 ml capacity, the bottom of the containers being 45 ± 10 mm from the centre of the shaft. The shaft/container assembly is rotated at a speed of 40 ± 2 rev/min. The temperature of the water bath is thermostatically controlled to maintain the test solution at the prescribed temperature $\pm 2^\circ\text{C}$.

NOTE — The wash wheel sponsored by the Society of Dyers and Colourists, or the Launderometer sponsored by the American Association of Textile Chemists and Colorists, or any other mechanical device giving identical results may be used for this test.

6.2 Stainless Steel Balls

ten, each approximately 6 mm in diameter.

6.3 Grey Scales

for evaluating change in colour and staining.

7 REAGENTS

7.1 Soap-Soda Solution

containing 5 g of soap and 2 g of anhydrous sodium carbonate per litre of solution. The soap shall contain not more than 5 percent moisture and shall comply with the following composition (based upon dry mass):

Free alkali, calculated as Na_2CO_3 (<i>Max</i>), percent	0.3
Free alkali, calculated as NaOH (<i>Max</i>), percent	0.1
Combined fatty acids, calculated as Na salt (<i>Min</i>), percent	85
Titre of mixed fatty acids prepared from the soap (<i>Max</i>)	39°C
Iodine value of fatty acids (<i>Max</i>)	50

7.1.1 The soap shall be free from optical brightening agents.

7.2 Sodium Hypochlorite Solution

containing 10 g/l of available chlorine, freshly prepared, having a pH between 10.8 and 11.2.

7.3 Acetic Acid Solution

containing 0.14 g of glacial acetic acid per litre.

8 PROCEDURE

8.1 Place the composite specimen in one of the containers of the apparatus together with 10 stainless steel balls.

8.2 Take the necessary amount of soap and soda ash solution (*see* 7.1) previously heated to $83 \pm 2^\circ\text{C}$. Add to the hot solution sufficient amount of sodium hypochlorite solution (*see* 7.2) to make a final concentration of 0.1 g/l of available chlorine and to give a liquor ratio of 1 : 50.

8.3 Pour the solution as obtained in 8.2, into the container (*see* 8.1).

8.4 Treat the specimen in the apparatus for 45 minutes at $83 \pm 2^\circ\text{C}$. At the end of this period, drain out the liquor from the container.

8.5 Pour 100 ml of distilled water at $40 \pm 2^\circ\text{C}$ into the container and rinse the specimen in the container for one minute, shaking the composite specimen vigorously with water. Drain out the water from the container. Repeat this procedure once more.

8.6 Put 100 ml of acetic acid solution (*see* 7.3) into the container, shake the container vigorously with the specimen and acetic acid solution at $27 \pm 2^\circ\text{C}$ for one minute. Drain out the acid from the container.

8.7 Rinse the specimen repeatedly in cold water until it is free from acid.

8.8 Wring the specimen and dry it in air in shade at a temperature not exceeding 60°C .

8.9 Remove the specimen, open it out and separate the two pieces of adjacent fabrics.

8.10 Evaluate the change in colour of the treated specimen and the degree of staining of the two pieces of adjacent fabrics with grey scales.

NOTES

1 Treated specimen and the two pieces of adjacent fabrics should have cooled after drying, and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness ratings as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

9 REPORT

9.1 Report the numerical rating for change in colour of the test specimen and the numerical ratings for staining of the two pieces of adjacent fabrics used in the preparation of the composite specimen.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO WATER

(Source : IS 767 : 1988)

The method prescribed in this standard is based on ISO 105/E-1978 Textiles — Tests for colour fastness E01 — Colour fastness to water,

issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to immersion in water.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is immersed in water, drained and placed between two plates under a specified pressure in the testing device. The specimen and the adjacent fabrics are dried separately. The change in colour of the specimen and the staining of adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Testing Device

consisting of a frame of stainless steel into which a weight-piece of mass 5 kg and base 11.5 cm × 6 cm is closely fitted so that a pressure of 12.5 kPa can be applied on test specimens measuring 10 cm × 4 cm placed between glass or acrylic resin plates. If the weight-piece is removed during the test, the testing device shall be so constructed that the pressure of 12.5 kPa remains unchanged.

NOTE — Suitable testing devices are the hydrotest, the perspiration tester and the perspirometer. If the dimensions of the composite specimen differ from the size of 10 cm × 4 cm, such a weight-piece has to be used that a pressure of 12.5 kPa is applied to the specimen. Other devices may be used provided that the same results are obtained as with the apparatus described in 4.1.

4.2 Oven

maintained at $37 \pm 2^\circ\text{C}$.

4.3 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends, the second piece made of the fibre as indicated below, or in the case of blends, of the kind of fibre second in order of predominance, or as otherwise specified (see Annex A of IS 10251 : 1982):

<i>If First Piece is</i>	<i>Second Piece to be</i>
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Wool
Viscose	Wool
Acetate or triacetate	Viscose
Polyamide	Wool or cotton
Polyester	Wool or cotton
Acrylic	Wool or cotton

4.4 Grey Scales

for assessing change in colour and staining.

5 REAGENT

5.1 Distilled Water

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between the two adjacent fabrics (4.3) and sew along one of the shorter sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit or wave it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two adjacent fabrics (4.3), the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics (4.3). Sew along two opposite sides to hold the yarns in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (4.3) into a sheet 10 cm × 4 cm. Place the sheet

between the two adjacent fabrics and sew along all four sides to hold the fibre in place, and to form a composite specimen.

7 PROCEDURE

7.1 Thoroughly wet the composite specimen in distilled water at room temperature. Pour off the water and place the composite specimen between two glass or acrylic resin plates, measuring about 11.5 cm × 6.0 cm under a force of 50 N.

7.2 Place the apparatus containing the composite specimen in the oven for 4 h at $37 \pm 2^\circ\text{C}$.

7.3 Open out the composite specimen and dry it by hanging it in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.4 Assess the change in colour of the specimen and the staining of the adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report the numerical rating for change in colour of test specimen and the numerical rating for staining of each kind of adjacent fabric used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO SEA WATER

(Source : IS 690 : 1988)

The method prescribed in this standard is based on ISO 105/E-1978 Textiles — Tests for colour fastness E02 — Colour fastness to sea

water, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to immersion in sea water.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is immersed in sodium chloride solution, drained and placed between two plates under a specified pressure in a testing device. The specimen and the adjacent fabrics are dried separately. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Testing Device

consisting of a frame of stainless steel into which a weight-piece of mass 5 kg and a base of 11.5 cm × 6 cm is closely fitted with glass or acrylic resin plates of the same size and of 0.15 cm thickness. In this case, the size of the composite specimen must be 10 cm × 4 cm.

NOTES

1 Suitable testing devices are the hydrotest, the perspiration tester and the perspirometer. If the dimensions of the composite specimen differ from the size of 10 cm × 4 cm, such a weight piece has to be used so that a pressure of 12.5 kPa is applied to the specimen.

2 Other devices may be used provided the same results are obtained as with the apparatus described in 4.1.

4.2 Air Oven

maintained at $37 \pm 2^\circ\text{C}$.

4.3 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends, the second piece made of the fibre as indicated below or in the case of blends, of the kind of fibre second in order of predominance, or as otherwise specified (see Annex A of IS 10251 : 1982):

<i>If the First Adjacent Fabric is</i>	<i>Second Piece to be</i>
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Wool
Viscose	Wool
Acetate or triacetate	Viscose
Polyamide	Wool or Cotton
Polyester	Wool or Cotton
Acrylic	Wool or Cotton

4.4 Grey Scales

for evaluating change in colour and staining.

5 REAGENT

5.1 Sodium Chloride Solution

containing 30 g of pure sodium chloride per litre of distilled water.

NOTE — Pure sodium chloride shall mean sodium chloride that does not contain impurities which affect the test results.

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between two adjacent fabrics (4.3) and sew along one of the shorter sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 6.1, or form a layer of parallel lengths of it between the two adjacent fabrics (4.3), the amount of yarn taken being approximately equal to half the combined

mass of the adjacent fabrics, sew along two opposite sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (4.3) into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics sew along all four sides to hold the fibre in place and to form a composite specimen.

7 PROCEDURE

7.1 Wet each composite specimen thoroughly in separate container by immersion in sodium chloride solution (*see Note*) at room temperature. Pour off the solution and place the composite specimen between the two glass or acrylic resin plates measuring 11.5 cm × 6.0 cm × 1.5 cm under a pressure of 12.5 kPa. Keep the glass plates in position in the perspirometer (or its equivalent) and place the apparatus in the air oven maintained at $37 \pm 2^{\circ}\text{C}$ for 4 hours. At the end of this period remove the specimen, separate the specimen and the two pieces of adjacent fabrics, and dry them apart by hanging in air in shade at a temperature not exceeding

60°C with the three parts in contact only at the remaining line of stitching.

NOTE — Special care should be taken when wetting the specimen to see that it is uniformly saturated, in particular, when wool or material containing wool is to be wetted out, it should be kneaded thoroughly by hand with the flattened end of a glass rod, or by a mechanical device.

7.2 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report individually, the numerical rating for change in colour of test specimen and the numerical ratings for staining of the two pieces of adjacent fabrics used in the preparation of the composite specimen.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO CHLORINATED WATER (SWIMMING-BATH WATER)

(Source : IS 4803 : 1985)

The method prescribed is based on ISO 105/E-1978 Textiles — Tests for colour fastness F.03 — Colour fastness to chlorinated water (swimming-

bath water), issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textiles of all kinds and in all forms to the action of active chlorine in concentrations such as are used to disinfect swimming-bath water (break point chlorination).

2 PRINCIPLE

2.1 A specimen of the textile is treated with a very weak chlorine solution and dried. The change in colour of the specimen is assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the material specification or as agreed to between the buyer and the seller to evaluate the colour fastness of the textile material in the lot to chlorinated water shall be representative of the lot.

4 APPARATUS

4.1 A Glass Container or Glazed China Container which can be closed for specimen and chlorine solution.

4.2 Grey Scale

for assessing change in colour.

5 REAGENTS

5.1 Sodium Hypochlorite (NaOCl) Solution of the following composition:

Active chlorine	140 to 160 g/l
Sodium chloride (NaCl)	120 to 170 g/l
Sodium hydroxide (NaOH), Max	20 g/l
Sodium carbonate (Na ₂ CO ₃), Max	20 g/l
Iron (Fe), Max	0.01 g/l

5.2 Sodium Hypochlorite Solution

containing 20 mg of active chlorine per litre at pH 8.5 and may be prepared as follows.

5.2.1 Solution A

Dilute 4 ml of sodium hypochlorite solution (see 5.1) to 1 litre.

5.2.2 Solution B

Dissolve in 51.0 ml of 1 N sodium hydroxide (NaOH) solution, 18.64 g of potassium chloride (KCl) and 15.46 g of boric acid (H₃BO₃) and dilute to 1 litre.

5.2.3 To 50 ml of solution A, add excess of potassium iodide (KI) and hydrochloric acid (HCl) and titrate the liberated iodine with 0.1 N sodium thiosulphate (Na₂S₂O₃) solution using starch as indicator. Let the volume of 0.1 N sodium thiosulphate solution required be *V* ml.

5.2.4 For each 100 ml of working solution required, add (28.2/*V*) ml of solution A to 20 ml of solution B and dilute to 100 ml.

5.2.5 Check the pH of the solution (see 5.2.4) against a standard buffer solution electrometrically.

NOTE — The solution should be prepared just prior to use.

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, draw a 10 cm × 4 cm specimen from each piece in the test sample (see 3.2). Draw at least three such test specimens.

6.2 If the textile to be tested is yarn, knit or weave the test sample (see 3.2) into fabric and draw from it at least three 10 cm × 4 cm test specimens. Alternatively, make a wick or parallel lengths of yarn 10 cm long and about 0.5 cm in diameter, tied near both ends.

6.3 If the material to be tested is loose fibre, from the test sample (see 3.2) comb and compress enough of it to form a sheet of 10 cm × 4 cm, and sew it on to a piece of cotton adjacent cloth to support the fibre. Similarly, prepare at least two more test specimens from the test sample.

7 PROCEDURE

7.1 Weigh one test specimen accurately nearest to 1 mg. Put in the container sufficient amount of sodium hypochlorite solution (see 5.2) such that the liquor ratio is 1 : 100. Immerse the specimen in this solution ensuring that the specimen is thoroughly wetted. Close the container and allow the specimen to remain in solution at $27 \pm 2^{\circ}\text{C}$ for 4 hours in subdued light.

7.2 Remove the specimen from the container, squeeze or hydroextract it till no solution drips out of the specimen and dry it by hanging in air at room temperature. Evaluate the change in

colour of the treated specimen with the help of grey scale on the same day and assign the ratings.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Repeat the test with remaining test specimens.

8 REPORT

8.1 Report individually the numerical ratings for change in colour of the test specimens.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO HOT WATER

(Source : IS 4389 : 1987)

The method prescribed in this standard is based on ISO 105/E-1978 Textiles — Tests for colour fastness E08 — Colour fastness to water:

Hot water, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of hot water.

1.1.1 The method prescribed in this standard is mainly applicable to wool and textiles containing wool.

2 PRINCIPLE

2.1 A specimen of textile in contact with adjacent fabrics is rolled around a glass rod, treated with slightly acidified hot water and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be taken to be representative of the lot.

4 APPARATUS

4.1 Vessel

equipped with reflux condenser to hold a cylindrical specimen, 4 cm long, in hot water.

4.2 Thermostatically Controlled Bath

to maintain the contents of the vessel (4.1) at $70 \pm 2^\circ\text{C}$.

4.3 Glass Rod

5 to 8 mm in diameter.

4.4 Wool Adjacent Fabric

10 cm \times 4 cm (see Annex A).

4.5 Cotton Adjacent Fabric

or adjacent fabric made from the fibre used in a mixture with the wool measuring 10 cm \times 4 cm (see Annex A).

4.6 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Distilled Water

if necessary, acidified with acetic acid to pH 6 ± 0.5 .

6 PREPARATION OF COMPOSITE SPECIMENS

6.1 If the textile to be tested is fabric, draw a 10 cm \times 4 cm test specimen from each piece in the test sample. Prepare a composite specimen by placing the test specimen between two adjacent fabrics (4.4 and 4.5) and sew along one of the shorter sides. Prepare at least three such composite specimens.

6.2 If the textile to be tested is yarn, knit or weave the test sample into fabric and draw from it a 10 cm \times 4 cm test specimen; alternatively prepare a 10 cm \times 4 cm test specimen in the form of parallel lengths of yarn, the amount of yarn taken being approximately equal to half the combined mass of the two adjacent fabrics (4.4 and 4.5). Place the test specimen between two adjacent fabrics and sew around all four sides to hold the yarn in place and to form a composite specimen. Prepare at least three such composite specimens.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (4.4 and 4.5) into sheet of 10 cm \times 4 cm size. Place the sheet between the two adjacent fabrics, and sew around all four sides to hold the fibres in place and to form a composite specimen. Prepare at least three such composite specimens.

7 PROCEDURE

7.1 Roll the composite specimen around the glass rod to form a cylinder 4 cm long, and tie it loosely and uniformly with thread.

7.2 Treat the specimen on the rod for 30 minutes in slightly acidified distilled water (see 5.1) at a temperature of $70 \pm 2^\circ\text{C}$, at a liquor ratio of 1 : 30 under reflux. During the test, ensure that the composite specimen is always submerged in the water.

7.3 Remove the composite specimen from the rod and squeeze it. Open out the composite specimen by breaking the stitching on all sides except one of the shorter sides and dry it in air at a temperature not exceeding 60°C with three parts in contact only along the remaining line of stitching.

7.4 Evaluate the change in colour of the treated test specimen and the degree of staining of the adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimen and the two adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.5 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 Report individually the numerical rating for change in colour of the test specimen and the numerical ratings for staining of the two adjacent fabrics used in the preparation of the composite specimen.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass, g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
Cotton	115 ± 5	1/1 Plain	35	31	16.5	14
Viscose	140 ± 5	1/1 Plain	28	22	20	33
Wool	125 ± 5 — 0	1/1 Plain	21 ± 0.5	18 ± 0.5	15.6×2 Worsted	15.6×2 Worsted
Polyamide	130 ± 5	1/1 Plain	17.5	20	10×2	20
Polyester	130 ± 5	1/1 Plain	23.5	20.5	7.5×2	20
Acrylic	135 ± 5	1/1 Plain	17.5	16	10×2	10×2

NOTES

1 For wool adjacent fabric, additional requirements are: (a) pH value of aqueous extract 6.5 to 7.5, (b) residual fat content 0.4 ± 0.1 percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic, additional requirements are: (a) pH value of aqueous extract 7 ± 0.5 , and (b) residual oil content less than 1.0 percent.

3 For polyester, additional requirement is residual oil content less than 0.5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

5 The constructional and other details of adjacent fabrics as given above are for guidance only.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO PERSPIRATION

(Source : IS 971 : 1983)

This standard is based on ISO 105/E-1978 Textiles — Tests for colour fastness E04 — Colour fastness to perspiration, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of human perspiration.

2 PRINCIPLE

2.1 Specimens of the textiles in contact with adjacent fabrics are treated in two different solutions containing histidine, drained and placed between two plates under a specified pressure in a testing device. The specimens and the adjacent fabrics are dried separately. The change in colour of each specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with an agreement between the buyer and the seller to evaluate the colour fastness to perspiration of the textile material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Testing Device

consisting of a frame of stainless steel into which a weight-piece of mass 5 kg and a base of 11.5×6 cm is closely fitted, with glass or acrylic resin plates of the same size and of 0.15 cm thickness. In this case, the size of the composite specimen shall be 10×4 cm.

NOTES

1 The perspirometer or perspiration tester is a suitable testing device. If the dimensions of the test piece differ from the size of 10×4 cm, such weight piece has to be used so that a pressure of 12.5 kPa is applied to the specimen.

2 Other devices may be used provided that the same test results are obtained as with the apparatus described in 4.1.

4.2 Oven

capable of being maintained at $37 \pm 2^\circ\text{C}$.

4.3 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind of fibre as that of textile to be tested or that predominating in the case of blends, the second piece made of the fibre as indicated below or in the case of blends, of the kinds of fibre second in order of predominance or as otherwise specified (see Annex A of IS 10251 : 1982):

If First Piece is

Cotton
Wool
Silk
Linen
Viscose
Acetate
Polyamide
Polyester
Acrylic

Second Piece to be

Wool
Cotton
Cotton
Wool
Wool
Viscose
Wool or viscose
Wool or cotton
Wool or cotton

4.4 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.1 Alkaline Solution

freshly prepared, containing the following per litre:

- 0.5 g of 1-histidine monohydrochloride monohydrate ($\text{C}_6\text{H}_9\text{O}_2\text{N}_3\text{HCl} \cdot \text{H}_2\text{O}$),
- 5 g of sodium chloride (NaCl), and
- 5 g of disodium hydrogen orthophosphate dodecahydrate ($\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$).

The solution is brought to pH 8 with 0.1 N sodium hydroxide solution.

5.2 Acid Solution

freshly prepared, containing the following per litre:

- 0.5 g of 1-histidine monohydrochloride monohydrate ($\text{C}_6\text{H}_9\text{O}_2\text{N}_3\text{HCl} \cdot \text{H}_2\text{O}$),
- 5 g of sodium chloride (NaCl), and
- 2.2 g of sodium dihydrogen orthophosphate dihydrate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$).

The solution is brought to pH 5.5 with 0.1 N acetic acid solution.

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen 10×4 cm between the two adjacent fabrics (see 4.3) and sew along one of the shorter sides to form a composite specimen. Two such composite specimens are required.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1 or form a layer of parallel lengths of it between the two adjacent fabrics (see 4.3), the amount of yarn taken being approximately equal to half the combined mass of adjacent fabrics. Sew along two composite sides to hold the yarn in place and to form a composite specimen. Two such composite specimens are required.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of adjacent fabrics (see 4.3) into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibres in place and to form a composite specimen. Two such composite specimens are required.

7 PROCEDURE

7.1 Wet one of the composite specimens thoroughly in alkaline solution (see 5.1) at a liquor ratio of 1 : 50 and allow it to remain in the solution at room temperature for 30 minutes. Press and move it from time to time to ensure good and uniform penetration of the liquor. Pour off the solution and wipe the excess liquor off the specimen between two glass rods. Then place the composite specimen between two glass or acrylic

resin plates measuring about 11.5×6 cm under a pressure of 12.5 kPa. Keep the testing device containing the composite specimen in the oven for 4 hours at $37 \pm 2^\circ\text{C}$.

7.2 Open out the composite specimen by breaking the stitching on all sides except one of the shorter sides and dry by hanging in air at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.3 Treat the second composite specimen as in 7.1 and 7.2 using acidic solution (see 5.2) instead of alkaline solution.

7.4 Repeat the test with other composite specimens taken from the other test pieces.

7.5 Evaluate the change in colour of the treated test specimens and the degree of the staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTE — In case of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material tested; and
- b) For each of the test solutions specified in 5.1 and 5.2, report:
 - 1) numerical rating for the change in colour for all the test specimens, and
 - 2) numerical rating for staining of each kind of adjacent fabric used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO DECATIZING

(Source : IS 865 : 1958)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre and the method of dyeing or printing employed; the same colouring matter when used in dyeing or printing different fibres

or, when applied by different methods upon the same fibre, may give vastly different results. The method prescribed is based on ISO 105/E-1978 Textiles — Tests for colour fastness E10 — Colour fastness to decatizing, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 These methods are intended for the determination of colour fastness of textile materials to the action of steam as employed in the decatizing of wool fabrics.

1.2 Two tests, one for mild decatizing and the other for severe decatizing, are prescribed.

2 PRINCIPLE

2.1 A test piece of the textile material is wrapped round a perforated cylinder and steam is passed through it for 15 minutes. The test piece is then dried and the numerical rating for change in colour of the piece is evaluated with grey scale. A test control fabric is used for reference.

3 SAMPLING

3.1 Samples shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the procedure laid down in material specification or as agreed between the buyer and the seller shall be representative of the lot.

4 TEST PIECES

4.1 If the material to be tested is fabric, draw two 10 × 4 cm test pieces from the sample.

4.2 If the material to be tested is yarn, knit or weave some of the sample into fabric and draw from it two 10 × 4 cm test pieces; alternatively, prepare a test piece in the form of parallel lengths of yarn, place it between two 10 × 4 cm pieces of undyed cotton cambric and sew around the four sides to hold the yarns in place. Prepare one more test piece in a similar manner.

4.3 If the material to be tested is loose fibre, comb and compress enough of the sample into the form of a sheet to make a 10 × 4 cm test piece, place it between two pieces of undyed cotton cambric and sew around the four sides to hold the fibres in place. Prepare another test piece in a similar manner.

5 TEST CONTROL

5.1 A one percent dyeing of Diamond Brown RH Extra (Colour Index, second edition, Mordant

Brown 33) on wool cloth shall be used as a test control to ensure that the test is carried out correctly.

NOTE — For preparation of the dyeing, see Annex A.

6 EQUIPMENT

6.1 Decatizing Apparatus

or any other similar apparatus.

6.2 Cotton Blanket

boiled off, weighing about 400 g/m² and napped on both sides and of a length sufficient to wrap six times round the perforated cylinder of the decatizing apparatus.

6.3 Grey Scale

for evaluating change in colour.

7 PROCEDURE

7.1 Mild Decatizing

7.1.1 Wrap the cotton blanket three times around the perforated cylinder of the decatizing apparatus. Place one of the test pieces and a 10 × 4 cm test control around the wrapped cylinder and cover them with three further wrappings of the blanket cloth. Pass saturated but water-free steam at 110°C and at a pressure of 1.5 kg/cm² or 0.5 kg/cm² above atmospheric pressure, through the test piece and the test control for 15 minutes. Dry the test piece and the test control in air in shade at a temperature not exceeding 60°C.

7.1.2 Evaluate the change in colour of the treated test control with the help of grey scales and assign the ratings (see Note). If the numerical rating for change in colour is not equal to 4 yellower, then repeat the test (see 7.1.1) using a fresh test piece and a fresh test control.

NOTES

1 The treated control should have cooled after drying and should have regained its normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.1.3 Evaluate the change in colour of the treated test piece with the help of grey scale and assign the ratings (*see* Notes under 7.1.2).

7.2 Severe Decatizing

7.2.1 Treat the second test piece and a fresh test control in the manner prescribed in 7.1.1 but using steam at 127°C and at a pressure of 2.5 kg/cm² or 1.5 kg/cm² above atmospheric pressure. Dry them in air in shade at a temperature not exceeding 60°C.

7.2.2 Evaluate the change in colour of the treated test control with the help of grey scale and assign the ratings (*see* Notes under 7.1.2). If the numeri-

cal rating for change in colour is not equal to 3 yellower, then repeat the test (*see* 7.2.1) using a fresh test piece and a fresh test control.

7.2.3 Evaluate the change in colour of the treated test piece with the help of grey scale and assign the ratings (*see* Notes under 7.1.2).

8 REPORT

8.1 The report shall include the following information:

The numerical ratings for change in colour of the test piece for:

- 1) both mild, and
- 2) severe decatizing.

ANNEX A

PREPARATION OF DYEING

A-1 Wet out well sufficiently large-sized piece of wool cloth and enter it at 40°C into a dye bath containing one percent Diamond Brown RH Extra (Colour Index, second edition, Mordant Brown 33), 10 percent sodium sulphate crystals (Na₂SO₄.10H₂O) and 3 to 5 percent acetic acid (30 percent), all percentages being calculated on the weight of the piece, and using a liquor ratio of 1 : 40.

A-2 Bring the dye bath to boil within 30 minutes and boil for a further period of 30 minutes. Exhaust the dye bath, if necessary, by adding carefully 3 to 5 percent acetic acid (30 percent). Boil for a further period of 15 minutes after addition of the acid. Cool the dye bath by adding cold water and add 0.5 percent potassium dichromate dissolved in water. Bring it into the boil and continue for 45 minutes. Remove the piece, rinse it in cold running tap water and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO POTTING

(Source : IS 972 : 1988)

This standard is based on ISO 105/E-1978 Textiles — Tests for colour fastness E09 — Colour

fastness to potting, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of boiling water.

1.2 The method is mainly applicable to wool and textile materials containing wool.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is rolled around a glass rod and treated with boiling water. The specimen and adjacent fabrics are dried separately. The change in colour of the specimen and staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Vessel

equipped with reflux condenser and capable of holding a cylindrical specimen 4 cm long in boiling water.

4.2 Glass Rod

5 to 8 mm in diameter.

4.3 Wool Adjacent Fabric

measuring 10 cm × 4 cm.

4.4 Cotton Adjacent Fabric

or in the case of blends, adjacent fabric made from the kind of fibre admixed with wool, measuring 10 cm × 4 cm (see Annex A of IS 10251 : 1982).

4.5 Grey Scales

for assessing change in colour and staining.

5 PREPARATION OF COMPOSITE SPECIMEN

5.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between the two adjacent fabrics (4.3 and 4.4) and sew along one of the shorter sides to form a composite specimen.

5.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 5.1 or form a layer of parallel lengths, of it between the two adjacent fabrics (4.3 and 4.4), the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along two opposite sides to hold the yarn in place and to form a composite specimen.

5.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (4.3 and 4.4) into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics, and sew along all four sides to hold the fibre in place and to form a composite specimen.

6 PROCEDURE

6.1 Roll the composite specimen compactly around the glass rod to form a cylinder 4 cm long, and tie loosely and uniformly with thread.

6.2 Treat the specimen on the rod for 1 h in boiling distilled water at a liquor ratio of 1 : 30 under reflux.

6.3 Open out the composite specimen by breaking the stitching on all sides except one of the shorter sides and dry it by hanging in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

6.4 Assess the change in colour of the specimen and the staining of the adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in colour fastness ratings as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7 REPORT

7.1 Report the numerical rating for change in colour of test specimen and the numerical ratings for staining of the two adjacent fabrics used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ACID MILLING

(Source : IS 981 : 1988)

The method of test prescribed in this standard is intended for evaluating colour fastness of textile materials to the action of dilute and hot

mineral acids as used in the hat-making and felt industries, and also in the manufacture of blanket-ing and service materials.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds, and in all forms to the action of dilute and hot mineral acids as used in milling.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is milled in a container containing steel balls and a solution of sulphuric acid. The severity of the action is standardized by means of a test control dyeing milled separately in the same way. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of the same kind of fibre as that of the textile to be tested, or that predominating in the case of blends, the second piece made of the fibre as indicated below or in the case of blends, of the kind of fibre second in order of predominance, or as otherwise specified (see Annex A of IS 10251 : 1982):

If the First Piece is Second Piece to be

Cotton	Wool
Wool	Cotton
Linen	Wool
Viscose	Wool
Acetate	Wool
Polyamide	Wool
Polyester	Wool
Acrylic	Wool

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between the two adjacent fabrics and sew along all four sides to form a composite specimen.

4.2.2 If the textile to be tested is yarn, knit or weave it into fabric and treat it as in 4.2.1 or form a layer of parallel lengths of it between the two adjacent fabrics, the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

4.2.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics, and sew along all four sides to hold the fibre in place and to form a composite specimen.

4.2.4 Prepare the composite specimen of the test control in the way outlined for fabric in 4.2.1.

5 APPARATUS

5.1 Suitable Mechanical Device

consisting of water bath containing a rotatable shaft which supports, radially, glass or stainless steel containers (75 ± 5 mm diameter × 125 ± 10 mm high) of approximately 550 ± 50 ml capacity, the bottom of the containers being 45 ± 10 mm high from the centre of the shaft. The shaft/container assembly is rotated at a speed of 40 ± 2 rev/min. The temperature of the water bath is thermostatically controlled to maintain the test solution at the prescribed temperature $\pm 2^\circ\text{C}$.

Suitable devices are:

- Laundrometer, as described in the Technical Manual of the American Association of Textile Chemists and Colorists (AATCC).
- Linitest, as described in Mellian Textilverichts 49 (1968), 6, pp. 709-711.
- Wash wheel sponsored by the Society of Dyers and Colourists.

NOTE — Other mechanical devices may be used for the test, provided that the results are identical to those obtained with the apparatus described above.

5.2 Non-corrodible (Stainless) Steel Balls

6 mm diameter.

5.3 Test Control

a dyeing of CI Acid Blue 7 (Colour Index, 3rd edition) on wool cloth. A well wetted-out pattern of wool serge is entered at 40°C into a dye bath containing 3 percent CI Acid Blue 7, 10 percent sodium sulphate decahydrate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and 3 percent sulphuric acid (sp gr 1.84), all percentages being calculated on the mass of the wool, at a liquor ratio of 1 : 40.

The dye bath is raised to the boil in 30 minutes and continued for 45 minutes. The pattern is removed, rinsed and dried.

5.4 Grey Scale

for evaluating change in colour and staining.

6 REAGENTS

6.0 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6.1 Sulphuric Acid Solution

containing 50 g of concentrated sulphuric acid (sp gr 1.84) per litre of distilled water.

7 PROCEDURE

7.1 Carry out the operations described in 7.2 to 7.6 with the composite specimen and the composite test control specimen in parallel in separate baths.

7.2 Put the composite specimen and the composite test control specimen in separate containers in the device each with three times its own mass of the milling solution and 50 stainless steel balls. Run the device for 2 h at $40 \pm 2^\circ\text{C}$.

7.3 Add sufficient distilled water at $40 \pm 2^\circ\text{C}$ to give a liquor ratio of 1 : 50, and run the device for an additional 10 minutes.

7.4 Remove the composite specimen, rinse twice in cold distilled water, then rinse for 10 minutes in cold running tap water. Open out the composite specimen by breaking the stitching on all sides except one of the shorter sides and dry it by hanging in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.5 Assess the change in colour of the test control and the staining of adjacent fabrics with grey scales. If the change in colour is not equal to rating 3 on the appropriate grey scale, the test has not been carried out correctly, and the operations described in 7.1 to 7.4 should be repeated with a fresh composite specimen and a fresh composite test control specimen (see Notes under 7.6).

7.6 Assess the change in colour of the specimen and the staining of adjacent fabrics with grey scales.

NOTES

1 Treated test specimen or the test control should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report the numerical rating for change in colour of the test specimen and the numerical ratings for the staining of two pieces of adjacent fabrics used in the preparation of the composite specimens.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ALKALINE MILLING

(Source : IS 983 : 1980)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre, and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different

fibres or when applied by different methods upon the same fibre, may give vastly different results. The method prescribed is based on ISO 105/E-1978 Textiles — Tests for colour fastness E12 — Colour fastness to milling: Alkaline milling, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials to the action of soap and sodium carbonate used in alkaline milling.

1.2 The method is applicable to materials containing wool.

2 PRINCIPLE

2.1 A specimen of textile in contact with adjacent fabrics is milled in a jar containing stainless steel balls, and solution of soap and sodium carbonate. The severity of the action is standardized by means of a test control milled separately in the same way. The ratings for change in colour of the test specimen and staining of the two pieces of adjacent fabrics are assessed with the grey scales.

3 SAMPLING

3.1 The sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 A suitable mechanical washing device conforming to the following requirements shall be used:

- A water bath containing a rotor with which containers (of glass or of stainless steel) of 500 ml capacity are rotated at a speed of 40 ± 2 rev/min.
- Means of thermostatically controlling the temperature of water bath so as to maintain the temperature of test solution in the containers at $40 \pm 2^\circ\text{C}$.

NOTE — Launderometer* developed by the Silk and Art Silk Mills Industry's Research Association or any other mechanical apparatus giving identical results may be used for this test.

*Available with the Silk and Art Silk Mills Industry's Research Association, Dr. A. B. Road, Worli, Bombay 400025

4.2 Stainless Steel Balls

approximately 6 mm in diameter.

4.3 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends, the second piece made of the fibre as indicated below or, in the case of blends, of the kind of fibre second in order of predominance (see Annex A of IS 10251 : 1982):

<i>If the First Piece Consists of</i>	<i>Second Piece to be</i>
Cotton	Wool
Wool	Cotton
Linen	Wool
Viscose	Wool
Acetate	Wool
Polyamide	Wool
Polyester	Wool
Acrylic	Wool

4.4 Test Control

A dyeing of 3 percent Acid Blue 7 (Colour Index, 3rd edition) on wool cloth shall be used as a test control to ensure that the test was carried out correctly.

NOTE — For preparation of the dyeing, see Annex A.

4.5 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Soap-Soda Solution

The soap solution shall contain 50 g/l soap and 10 g/l anhydrous sodium carbonate. The soap shall conform to the requirements as given below with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

<i>Characteristic</i>	<i>Requirement</i>
Free alkali calculated as Na_2CO_3 (<i>Max</i>), percent	0.3
Free alkali calculated as NaOH (<i>Max</i>), percent	0.1
Combined fatty acids calculated as Na salts (<i>Min</i>), percent	85
Titre of mixed fatty acids prepared from the soap (<i>Max</i>)	39°C
Iodine value of fatty acids (<i>Max</i>)	50

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen of 10×4 cm between the two adjacent fabrics (*see 4.3*) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat it as in 6.1, or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (*see 4.3*), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (*see 4.3*) into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare a composite test control specimen from the test control (*see 4.3*) as given in 6.1.

6.5 Prepare at least three composite test specimens.

7 PROCEDURE

7.1 Carry out the operations described in 7.2 to 7.4 inclusive with the composite specimen and

the composite test control specimen, in parallel in separate baths.

7.2 Put the composite specimen and the composite test control specimen in separate containers in the machine (*see 4.1*), each with three times its own mass of the milling solution and 50 of the stainless steel balls. Run the machine for 2 h at $40 \pm 2^\circ\text{C}$.

7.3 Add sufficient distilled water at $40 \pm 2^\circ\text{C}$ to give a liquor ratio of 1 : 100 and run the machine for an additional 10 minutes.

7.4 Remove the composite specimens, rinse twice in cold distilled water, then rinse for 10 min in cold running water. Open out the composite specimen by breaking the stitching on all but one of the shorter sides and dry it by hanging in air at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of the stitching.

7.5 Assess the change in colour of the test control and the staining of adjacent fabrics with the appropriate grey scales. If the change in colour is not equal to rating 3 on the appropriate grey scales, the test has not been carried out correctly, and the operations described in 7.2 to 7.4 should be repeated with a fresh composite specimen and a fresh composite test control specimen.

7.6 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimens and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.7 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- Type of material,
- Numerical ratings for change in colour of the test piece, and
- Numerical ratings for staining of the two adjacent fabrics.

ANNEX A**PREPARATION OF DYEING**

- A-1** Wet out a sufficiently large-sized piece of wool cloth and enter it at 40°C into a dye bath containing 3 percent Acid Blue 7 (Colour Index, 3rd Edition), 10 percent sodium sulphate crystals ($\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$) and 3 percent sulphuric acid (sp gr 1.84). All percentages being calculated on the mass of the piece of wool cloth and using a liquor ratio of 1 : 40.
- A-2** Bring the dye bath to the boil in 30 minutes and boil for 45 minutes. Remove the piece, rinse it well and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ACID-FELTING : SEVERE

(Source : IS 3425 : 1986)

The method prescribed is based on ISO 105/E-1978 Textiles — Test for colour fastness E13 — Colour fastness to acid felting : Severe, issued by

the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of acids as used under severe conditions in the acid felting process.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is treated with solutions of acetic acid and/or sulphuric acid, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the material specification or as agreed to between the buyer and the seller to evaluate the colour fastness of the textile material in the lot to acid-felting : severe, shall be representative of the lot.

4 APPARATUS

4.1 A suitable open container and a glass rod flattened at one end or appropriate mechanical device.

NOTE — Launderometer of American Association of Textile Chemists and Colorists or Washwheel sponsored by the Society of Dyers and Colourists, may be used. Other similar mechanical device may also be used for the tests, provided the results obtained are identical to those obtained with the apparatus described in 4.1.

4.2 Two adjacent fabrics, each measuring 10 cm × 4 cm, one piece made of wool and the other made of wool or of another fibre to be assessed for staining, as desired (see Annex A).

4.3 Grey Scales

for assessing change in colour and staining.

5 REAGENTS

5.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be

used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the experimental results.

5.2 Sulphuric Acid Solution

containing 1 ml concentrated sulphuric acid (sp gr 1.84) per litre.

5.3 Acetic Acid Solution

containing 5 ml of acetic acid (300 g/l) per litre.

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, draw at least three test specimens 10 cm × 4 cm from the test sample (see 3.2). Place a test specimen between the two adjacent fabrics (see 4.2) and sew along all four sides to form a composite specimen. Similarly, treat other test specimens to form composite specimens.

6.2 If the textile to be tested is yarn, knit or weave the test sample (see 3.2) into a fabric and treat as in 6.1, or form a layer of parallel lengths of yarn between the two adjacent fabrics (see 4.2), the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen. Prepare at least three such composite specimens.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (see 4.2) into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics, and sew along all four sides to hold the fibres in place and to form a composite specimen. Prepare at least three such composite specimens.

7 PROCEDURE

7.1 Take the requisite amount of sulphuric and/or acetic acid solution (see Note 1) in a container to give a liquor ratio of 1 : 40. Raise the temperature of the solution to 90 ± 2°C. Immerse one composite specimen in the solution for 30 minutes at 90 ± 2°C.

NOTES

1 The tests with sulphuric acid and acetic acid are carried out as separate tests or with only one of the two reagents (see 5.2 and 5.3), as required.

2 The mechanical milling device set to give results, identical with those of hand milling, may be used.

3 During milling by hand, move the composite specimen continuously with the glass rod while it is in the milling bath and press it with the rod every two minutes, without removing from the bath.

7.2 Remove the specimen, rinse it for 10 minutes in cold running water and drain it. Remove the stitching along all sides except one of the shorter sides. Open out the composite specimen and dry it by hanging in air at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.3 Evaluate the change in colour of the treated test specimen and the degree of staining of the

two adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test piece and the two adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported accordingly.

7.4 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 Report individually the numerical rating for change in colour of the test specimen and the numerical ratings for staining of the two adjacent fabrics used in the preparation of composite specimen.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass in g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
Cotton	115 ± 5	1/1 Plain	35	31	16.5	14
Viscose	140 ± 5	1/1 Plain	28	22	20	33
Wool	125 ± 5 — 0	1/1 Plain	21 ± 0.5	18 ± 0.5	15.6 × 2 Worsted	15.6 × 2 Worsted
Polyamide	130 ± 5	1/1 Plain	17.5	20	10 × 2	20
Polyester	130 ± 5	1/1 Plain	23.5	20.5	7.5 × 2	20
Acrylic	135 ± 5	1/1 Plain	17.5	16	10 × 2	10 × 2

NOTES

1 For wool adjacent fabric, additional requirements are : (a) pH value of aqueous extract 6.5 to 7.5, (b) residual fat content 0.4 ± 0.1 percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic adjacent fabrics, additional requirements are : (a) pH value of aqueous extract 7 ± 0.5, and (b) residual oil content less than 1.0 percent.

3 For polyester adjacent fabric, additional requirement is residual oil content less than 0.5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ACID-FELTING : MILD

(Source : IS 3857 : 1986)

The method prescribed is based on ISO 105/E-1978 Textiles — Tests for colour fastness E14 — Colour fastness to acid felting: Mild, issued by the

International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of dilute hot mineral acids as used in mild felting conditions in the hat-making and felt industries.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is milled in acid solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot to this specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the material specification or as agreed to between the buyer and the seller to evaluate the colour fastness of the textile material in the lot to acid felting: mild, shall be representative of the lot.

4 APPARATUS

4.1 A suitable receptacle and a glass rod flattened at one end or appropriate mechanical device.

NOTE — Launderometer of American Association of Textile Chemists and Colorists or Washwheel sponsored by the Society of Dyers and Colourists, may be used. Other mechanical devices may also be used for the tests, provided that the results are identical to those obtained with the apparatus described in 4.1.

4.2 Two adjacent fabrics, each measuring 10 cm × 4 cm, one piece made of wool and the other made of wool or of another fibre to be assessed for staining, as desired (see Annex A).

4.3 Grey Scale

for assessing change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be

used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the experimental results.

5.1 Sulphuric Acid Solution

containing 1 ml of concentrated sulphuric acid (sp gr 1.84) per litre.

6 PREPARATION OF TEST SPECIMENS AND COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, draw at least three test specimens 10 cm × 4 cm from the test sample (see 3.2). Place a test specimen between the two adjacent fabrics (see 4.2) and sew along all four sides to form a composite specimen. Similarly, treat other test specimens to form composite specimens.

6.2 If the textile to be tested is yarn, knit or weave the test sample (see 3.2) into a fabric and treat as in 6.1, or form a layer of parallel lengths of yarn between the two adjacent fabrics (see 4.2), the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen. Prepare at least three such composite specimens.

6.3 If the textile to be tested is loose fibre, from the test sample (see 3.2), comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (see 4.2) into a sheet 10 cm × 4 cm. Place the sheet between two adjacent fabrics, and sew along all four sides to hold the fibres in place and to form a composite specimen. Prepare at least three such composite specimens.

7 PROCEDURE

7.1 Take the requisite amount of sulphuric acid in a container to give a liquor ratio of 1 : 40. Raise the temperature of the solution to $60 \pm 2^\circ\text{C}$. Immerse the composite specimen in the solution and mill it for 1 hour at $60 \pm 2^\circ\text{C}$.

NOTES

1 A mechanical milling device set, which gives results, identical with those of hand milling, may be used.

2 During milling by hand, move the composite specimen continuously with the glass rod while it is in the milling bath and press it with the rod every two minutes without removing from the bath.

7.2 Remove the composite specimen, rinse it for 10 minutes in cold running water and drain it. Remove the stitching along all sides except one of the shorter sides. Open out the composite specimen and dry it by hanging in air at a temperature not exceeding 60°C with the three parts in contact only along the remaining line of stitching.

7.3 Evaluate the change in colour of the treated test specimens and the degree of staining of the two adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimens and two adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported accordingly.

7.4 Report the test with the remaining composite specimens.

8 REPORT

8.1 Report, individually, the numerical rating for change in colour of the test specimens and the numerical ratings for staining of the two adjacent fabrics used in the preparation of the composite specimens.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass in g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
Cotton	115 ± 5	1/1 Plain	35	31	16.5	14
Viscose	140 ± 5	1/1 Plain	28	22	20	33
Wool	125 ± 5 — 0	1/1 Plain	21 ± 0.5	18 ± 0.5	15.6 × 2 Worsted	15.6 × 2 Worsted
Polyamide	130 ± 5	1/1 Plain	17.5	20	10 × 2	20
Polyester	130 ± 5	1/1 Plain	23.5	20.5	7.5 × 2	20
Acrylic	135 ± 5	1/1 Plain	17.5	16	10 × 2	10 × 2

NOTES

1 For wool adjacent fabric, additional requirements are : (a) pH value of aqueous extract 6.5 to 7.5, (b) residual fat content 0.4 ± 0.1 percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic adjacent fabrics, additional requirements are : (a) pH value of aqueous extract 7 ± 0.5, and (b) residual oil content less than 1.0 percent.

3 For polyester adjacent fabric, additional requirement is residual oil content less than 0.5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO HYPOCHLORITE BLEACHING

(Source : IS 762 : 1988)

The method prescribed in this standard is based on ISO 105/N-1978 Textiles — Tests for colour fastness N01 — Colour fastness to bleach-

ing: Hypochlorite, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of bleaching baths containing sodium or calcium hypochlorite in concentrations normally used in commercial bleaching.

1.1.1 The method is applicable principally, but not exclusively, to natural and regenerated cellulose textile materials.

2 PRINCIPLE

2.1 A specimen of the textile is agitated in a solution of sodium or calcium hypochlorite, rinsed in water, agitated in a hydrogen peroxide solution or sodium bisulphite solution, rinsed and dried. The change in colour is assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Container

made of glass or glazed porcelain with provision for closing.

4.2 Grey Scale

for evaluating change in colour.

5 REAGENTS

5.0 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Sodium Hypochlorite (NaOCl) Solution

containing 2 g of available chlorine per litre, buffered at pH 11.0 ± 0.2 with 10 g of anhydrous sodium carbonate (Na_2CO_3) per litre at $27 \pm 2^\circ\text{C}$. To prepare this reagent, use sodium hypochlorite of the following composition:

Active chlorine	140 to 160 g/l
Sodium chloride (NaCl)	120 to 170 g/l
Sodium hydroxide (NaOH), Max	20 g/l
Sodium carbonate (Na_2CO_3), Max	20 g/l
Iron (Fe), Max	0.01 g/l

5.2 Hydrogen Peroxide Solution

containing 2.5 ml of hydrogen peroxide [30 percent (*m/m*) H_2O_2] per litre or sodium bisulphite (NaHSO_3) solution (5 g/l).

5.3 Soap Solution

containing 5 g of soap per litre (*see* 5.1 of IS 3361 : 1979) for wetting out water-repellent fabrics.

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, use a specimen $10\text{ cm} \times 4\text{ cm}$.

6.2 If the textile to be tested is yarn, knit or weave it into fabric and use a specimen $10\text{ cm} \times 4\text{ cm}$, or make a wick of parallel lengths 10 cm long and about 0.5 cm in diameter tied near both ends.

6.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet $10\text{ cm} \times 4\text{ cm}$ in order to support the fibres, sew the sheet on a piece of cloth which will not affect the action of the hypochlorite on the specimen.

7 PROCEDURE

7.1 If the textile to be tested has a water-repellent finish, wet out the specimen thoroughly in the soap solution at 25 to 30°C . Drain the

specimen of excess liquor so that it retains approximately its dry mass of wetting-out solution and immediately place the specimen in an open form in the sodium hypochlorite solution at $27 \pm 2^\circ\text{C}$ at a liquor ratio of 1 : 50.

7.2 If the textile to be tested has no water repellent finish, wet out the specimen in distilled water at room temperature, remove the excess water and place the test specimen in an open form in the sodium hypochlorite solution at $27 \pm 2^\circ\text{C}$ at a liquor ratio of 1 : 50.

7.3 Close the container and keep the specimen in the solution at $27 \pm 2^\circ\text{C}$ for 60 min. Avoid exposure to direct sunlight.

7.4 Rinse the specimen thoroughly in cold running tap water and then stir it for 10 min in one of the solutions of H_2O_2 or sodium bisulphite at room temperature.

7.5 Rinse the specimen thoroughly in cold running tap water, remove the excess water and dry

the specimen by hanging it in air in shade at a temperature not exceeding 60°C .

7.6 Evaluation

Assess the change in colour of the specimen with grey scale.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report the numerical rating for change in colour of each specimen tested.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO PEROXIDE BLEACHING

(Source : IS 763 : 1988)

The method prescribed in this standard is based on ISO 105/N-1978 Textiles — Tests for colour fastness N02 — Colour fastness to bleach-

ing: Peroxide, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of bleaching baths containing peroxide in concentrations commonly used in textile processing.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is immersed in the bleaching solution, rinsed and dried. The change in colour of the specimen and the staining of adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Test Tube

having diameter and length such that the composite specimen, when rolled, will have a reasonably snug fit in the tube and be covered by the bleach bath.

4.2 Reflux Condenser

fitting the test tube to reduce evaporation from the bleaching bath during the test.

4.3 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends. the second piece made of the fibre as indicated below or, in the case of blends, of the kind of fibre second in order of predominance or as otherwise specified (see Annex A of IS 10251 : 1982).

If the First Adjacent Fabric is

Second Piece to be

Wool, silk, linen, viscose, polyester, acrylic

Cotton

Cotton, acetate

Viscose

4.4 Grey Scales

for evaluating change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Bleaching Baths

of the composition given below:

<i>Starting Bath</i>	<i>Bath 1</i>	<i>Bath 2</i>	<i>Bath 3</i>	<i>Bath 4</i>
(per litre of distilled water)	(for natural and regenerated cellulose)	(for natural and regenerated cellulose)	(for wool and acetate)	(for silk)
Hydrogen peroxide* solution, ml	3	—	12.2	12.2
Sodium peroxide†, g	—	3	—	—
Sodium silicate‡ solution, ml	5	5	—	5
Sodium pyrophosphate§, g	—	—	5	—

*Containing 602 g/l H₂O₂ (500 g/kg H₂O₂).

†100 percent Na₂O₂.

‡Relative density at 20°C = 1.32: SiO₂/Na₂O ratio = 2.7 : 1.

§Na₂P₂O₇·10H₂O.

Starting Bath (per litre of distilled water)	Bath 1 (for natu- ral and regene- rated cellu- lose)	Bath 2 (for natu- ral and regene- rated cellu- lose)	Bath 3 (for wool and acetate)	Bath 4 (for silk)
Magnesium chloride*, g	0.1	0.1	—	0.1
pH, initial value† ± 0.2	10.5	11.5	9.3‡	10.0
Temperature, °C ± 2°C	90	80	50	70
Duration of treatment, h	1	1	2	2
Liquor ratio	1 : 30	1 : 30	1 : 30	1 : 30

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between two adjacent fabrics (*see 4.3*) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 6.1, or form a layer of parallel lengths of it between the two adjacent fabrics (*see 4.3*), the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics, sew along all four sides to hold the yarn in place and to form a composite specimen.

*MgCl₂.6H₂O.

†Adjust by addition of NaOH solution, if necessary.

‡The pH of the bath at the end of the test should not be less than 9.0.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (*see 6.3*) into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen.

7 PROCEDURE

7.1 Loosely roll the composite specimen in the direction of the long edge, place the 4 cm roll into the test tube containing the appropriate bleaching solution (*see 5.1*) and keep it well covered by the bleaching solution for the time and temperature indicated in 5.1.

7.2 Remove the composite specimen, rinse it for 10 min in cold running tap water and squeeze it. Open the composite specimen by breaking the stitching on all sides except one of the shorter sides and dry by hanging it in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.3 Assess the change in colour of the specimen and the staining of the adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report the bleaching solution used, the numerical rating for change in colour of the specimen and the numerical rating for staining of each kind of adjacent fabric used individually.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO BLEACHING WITH SODIUM CHLORITE

(Source : IS 987 : 1988)

The methods prescribed in this standard are based on ISO 105/N-1978 Textiles — Tests for colour fastness N03 — Colour fastness to bleaching: Sodium chlorite: Mild; and ISO 105/

N-1978 Textiles — Tests for colour fastness N04 — Colour fastness to bleaching: Sodium chlorite, Severe; issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 The methods prescribed are intended for the determination of colour fastness of textile materials composed of natural or regenerated cellulose and synthetic fibres to the action of bleaching with sodium chlorite as employed in the textile processing.

1.2 In the first method (see 6), mainly applicable to textile materials made of regenerated cellulose and synthetic fibres, a comparatively mild test has been prescribed. In the second method (see 7), mainly applicable to textile materials made of natural cellulose textiles, a comparatively severe test has been prescribed.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is treated with sodium chlorite solution, rinsed and dried. The numerical ratings for change in colour of the specimen and staining of the adjacent fabrics are evaluated with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Two Adjacent Fabrics

10 cm × 4 cm, each made of fibre to be assessed for staining (see Annex A of IS 10251 : 1982).

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between two adjacent fabrics and sew along all four sides to form a composite specimen.

4.2.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 4.2.1, or form a layer of parallel lengths of it between the two adjacent fabrics, the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

4.2.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics. Sew along all four sides to hold the fibre in place and to form a composite specimen.

5 APPARATUS

5.1 Glass Container with Reflux Condenser

or other means of reducing evaporation of the bath and so preventing its modification during the test.

5.2 Grey Scales

for evaluating change in colour and staining.

6 FIRST METHOD

6.1 Reagent

6.1.1 Sodium Chlorite Solution

containing one gram of sodium chlorite (800 g/ kg) per litre (see Note) brought to pH 3.5 with acetic acid immediately before use.

NOTE — The concentration of sodium chlorite solution used should be determined by titration with sodium thiosulphate solution. One millilitre of 0.1 N sodium thiosulphate solution is equal to 0.00226 g of sodium chlorite (NaClO₂).

6.2 Procedure

Wet out the composite specimen in sodium chlorite solution and immerse it in the bath containing sodium chlorite solution for one hour without agitation at a temperature of 80 ± 2°C using a liquor ratio of 1 : 50. At the end of the

period, remove the specimen from the bath and rinse it for 10 minutes in cold running tap water. Remove the stitching from two long sides and one short side of the specimen, open it out and dry it in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

6.3 Evaluation

Evaluate the change in colour of the test specimen and the degree of staining of the two pieces of adjacent fabrics with the grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7 SECOND METHOD

7.1 Reagent

7.1.1 Sodium Chlorite Solution

containing 2.5 g of sodium chlorite (800 g/kg) per litre (*see* Note under 6.1.1) and 0.1 g of tetrasodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$) per litre, brought to pH 3.5 with formic acid immediately before the test.

7.2 Procedure

same as in 6.2.

7.3 Evaluation

same as in 6.3.

8 REPORT

8.1 Report the numerical rating for change in colour of test specimen, the numerical ratings for staining of the two adjacent fabrics used in the preparation of the composite specimen and the method used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO STOVING

(Source : IS 980 : 1980)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different fibres

or when applied by different methods upon the same fibres, may give vastly different results.

The method prescribed is based on ISO 105/N-1978 Textiles — Tests for colour fastness N05 — Colour fastness to stoving, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials to the action of sulphur dioxide as used for bleaching animal fibres.

2 PRINCIPLE

2.1 A composite specimen of the textile and a composite test-control specimen (in contact with adjacent fabrics), containing its own mass of soap solution, are exposed in an atmosphere containing sulphur dioxide. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with the grey scales.

3 SAMPLING

3.1 The samples shall be so drawn as to be the representative of the lot. Sample drawn in accordance with the procedure laid down in the material specifications or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 Vessel

of approximately 10-litre capacity for sulphur dioxide atmosphere.

4.2 Grey Scales

for assessing change in colour and staining.

4.3 Test Control

A dyeing of wool of 2.5 percent Acid Red 163 (Colour Index 3rd edition) shall be used to ensure that the test is carried out correctly.

NOTE — For preparation of the dyeing, see Annex A.

4.4 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of wool and the other of the same fibre as the cloth under test or as otherwise specified (see Annex A of IS 10251 : 1982).

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Soap Solution

The solution shall contain 5 g of soap per litre. The soap shall conform to the requirements given below, with moisture regain not more than 5 percent. The soap shall be free from optical brightening agents:

<i>Characteristic</i>	<i>Requirement</i>
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acids prepared from the soap (Max)	39°C
Iodine value of fatty acids (Max)	50

5.2 Sulphur

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is fabric, place a specimen of 10 × 4 cm between the two adjacent fabrics (see 4.4) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit it into fabric and treat as in 6.1, or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (see 4.4), the amount of yarn taken being approximately equal to half of the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half of the combined mass of the adjacent fabrics (see 4.4) into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics, and sew along all four sides to hold the fibre in place and to form a composite specimen. If necessary, sew along the two diagonals also.

6.4 Prepare at least three composite test specimens.

6.5 Test Control

Prepare the composite specimen of the test-control dyeing (see 4.3) as given in 6.1.

7 PROCEDURE

7.1 Thoroughly impregnate the composite specimen and the composite test-control specimen by immersion for 5 minutes at a temperature of $25 \pm 2^\circ\text{C}$ in the soap solution, then squeeze so that each contains its own mass of solution (see Note).

NOTE — When the material is to be wetted to contain its own mass of liquor, it may be saturated with the liquor and then drawn between two rubber rollers, squeezed by means of a rubber roller on a glass plate or centrifuged. Wringing by hand does not give uniform wetting.

7.2 Suspend the composite specimen and the composite test-control specimen for 16 h in the vessel containing an atmosphere of sulphur dioxide obtained by igniting 5 g of sulphur below the composite specimen and the composite test-control specimen and immediately closing the vessel.

7.3 Remove the composite specimen and the composite test-control specimen from sulphur dioxide atmosphere, remove the stitching along three sides of each and allow both to hang in air for at least 2 h without rinsing. The composite specimen and the composite test-control specimen are to be examined, when dry. If the com-

posite specimen contains cellulosic fibres, rinse it immediately after removal from sulphur dioxide atmosphere in distilled water in order to remove the acidity and then in cold running tap water. Remove the stitching along three sides of both specimens and dry them by hanging in air at a temperature not exceeding 60°C with the three parts in contact only at the remaining row of stitching.

7.4 Assess the effect on the test-control specimen with grey scale. If the change in colour is not equal to the contrast illustrated by Grade 3, the test has not been carried out correctly and the operations described in 7.1 to 7.5 should be repeated with a fresh composite specimen and a test-control specimen.

7.5 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with the help of grey scales and assign the ratings.

NOTES

1 Treated test specimens and the two pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.6 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test piece, and
- c) Numerical ratings for staining of the two adjacent fabrics.

ANNEX A

PREPARATION OF DYEING

A-1 Wet out a sufficiently large sized piece of wool cloth and enter it at 40°C into a dye bath containing 2.5 percent Acid Red 163 (Colour Index, 3rd edition), 10 percent sodium sulphate crystals ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and 3 percent acetic acid (30 percent), all percentages being calculated on the mass of the piece and using liquor ratio of 1 : 40.

A-2 Bring the dye bath to boil in 30 minutes and continue for 30 minutes. Exhaust the dye bath, if necessary, by carefully adding 1 to 3 percent acetic acid (30 percent) or 1 percent sulphuric acid (sp gr 1.84) well diluted with water. Boil the bath for a further period of 15 minutes after addition of the acid. Remove the piece, rinse it and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO HOT PRESSING

(Source : IS 689 : 1988)

This standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X11 — Colour

fastness to hot pressing, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to ironing and to processing on hot cylinders. Tests are given for hot pressing when the textile is dry, wet and damp. The end use of the textile usually determines which test should be made.

2 PRINCIPLE

2.1 Dry Pressing

The dry specimen is pressed with a heating device of specified temperature and pressure for a specified time.

2.2 Damp Pressing

The dry specimen is covered with a wet cotton adjacent fabric and pressed with a heating device of specified temperature and pressure for a specified time.

2.3 Wet Pressing

The upper surface of the wet specimen is covered with a wet cotton adjacent fabric and pressed with a heating device of specified temperature and pressure for a specified time.

2.4 The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales immediately and again after a period of exposure to air.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Heating Device

consisting of a pair of smooth and parallel plates equipped with an electrical heating system which is accurately controllable and giving a pressure

on the specimen of 4 ± 1 kPa. Heat should be transferred to the specimen from the upper side only; if the lower plate is equipped with a heating system which cannot be turned off, the insulating sheet (4.2) acts as a heat shield.

NOTES

1 The insulating sheet should be smooth and not warped. It is best to complete the specimen assembly on the insulating sheet before placing it on the heating device. The sheet should be cooled and the wet wool should be dried between tests.

2 If a heating device is not available, a household iron may be used, but its temperature should be measured with a surface pyrometer or with temperature sensitive papers. The iron should be weighed so that its area and total mass are in the appropriate ratio to exert a pressure of 4 ± 1 kPa. However, due to temperature fluctuation during on-off differences over the iron surface, the accuracy and reproducibility are limited. When a hand iron is used, this fact must be stated in the test report.

Under normal gravitational conditions, the area over which the mass of the heating plate should be distributed can be calculated in square centimetres by multiplying the mass in kilograms of the heating plate by the factor 24.525. If the area of the heating plate is less than that of specimen, the required mass is calculated in kilograms by dividing the plate area (expressed in square centimetres) by the same factor. For composite specimen $10 \text{ cm} \times 4 \text{ cm}$, the mass of the heating plate assembly should be between 1.25 and 2.00 kg.

4.2 Smooth Insulating Sheet

of thickness 3 to 6 mm (see Note 1 under 4.1).

4.3 Wool Flannel

of mass approximately 260 g/m^2 . Two layers of this material are used to make a pad of thickness approximately 3 mm. Similar smooth wool fabrics or felt can be used to give a pad of thickness approximately 3 mm.

4.4 Undyed, Bleached and Unmercerized Cotton Cloth

of mass 100 to 130 g/m^2 and with a smooth surface.

4.5 Cotton Adjacent Fabric

measuring $10 \text{ cm} \times 4 \text{ cm}$ (see Annex A of IS 10251 : 1982).

4.6 Grey Scales

for assessing change in colour and staining.

5 PREPARATION OF TEST SPECIMEN

5.1 If the textile to be tested is fabric, use a specimen of 10 cm × 4 cm.

5.2 If the textile to be tested is yarn, knit or weave it into fabric and use a piece 10 cm × 4 cm or wind it closely round a piece of thin inert material measuring 10 cm × 4 cm to form a layer having only the thickness of the yarn.

5.3 If the textile to be tested is loose fibre, comb and compress enough of it to form a sheet 10 cm × 4 cm and sew the sheet on to a piece of cotton adjacent fabric to support the fibre.

6 PROCEDURE

6.1 The following temperatures are used (*see Note*):

110 ± 2°C

150 ± 2°C

200 ± 2°C

When necessary, other temperatures may be used, provided that they are specially noted in the test report.

NOTE — The choice of pressing temperature used depends, to a large extent, on the type of fibre and on the construction of the fabric or garment. In the case of the blends, it is further suggested to use the temperature appropriate to the fibre with the lowest heat resistance. The indicated temperatures cover three commonly used pressing conditions.

6.2 Specimens of materials that have been subjected to any heat or drying treatment must be allowed to condition in the standard atmosphere for testing that is, 65 ± 2 percent relative humidity and a temperature of 27 ± 2°C before they are tested.

6.3 The bottom plate of the heating device is covered with insulating sheet (*see 4.2*), wool flannel (*see 4.3*) and dry undyed cotton cloth (*see 4.4*), whether it is heated or not.

NOTE — In order to obtain the pressure per unit area of 4 ± 1 kPa, the total area of the wool flannel padding should bear a suitable relationship to the mass of the plate pressing down on the padding. If the fabric to be tested has an appreciable thickness, it is necessary either to increase the area of the test specimen or to augment the pressure-bearing surface using a suitable template made from the same material as the test specimen. If the plates of the heating device are smaller than the specimen size, the pressure depends on the design of the apparatus (ratio of mass to area of the top plate).

6.4 Dry Pressing

Place the dry specimen on the top of the cotton cloth covering the wool flannel padding. Lower the top plate of the heating device and leave the specimen for 15 s at the specified pressing temperature.

6.5 Damp Pressing

Place the dry specimen on top of the cotton cloth covering the wool flannel padding. Soak a piece of cotton adjacent fabric measuring 10 cm × 4 cm in distilled water, and squeeze or extract it to contain its own mass of water. Place the wet fabric on top of the dry specimen. Lower the top plate of the heating device and leave the specimen for 15 s at the specified pressing temperature.

6.6 Wet Pressing

Soak the specimen and a piece of cotton adjacent fabric 10 cm × 4 cm in distilled water and squeeze or extract them to contain their own mass of water. Place the wet specimen on top of the dry cotton cloth covering the wool flannel pad and place the wet, adjacent fabric on the specimen. Lower the top plate of the heating device and leave the specimen for 15 s at the specified pressing temperature.

6.7 Assess the change in colour of the specimen with the appropriate grey scale immediately and again after the specimen has been allowed to condition for 4 h in the standard atmosphere for testing textiles.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

6.8 Assess the staining of the cotton adjacent fabric with appropriate grey scale. Use the more heavily stained side of the cotton adjacent fabric for the assessment (*see Note under 6.7*).

7 REPORT

7.1 The report shall include the following:

- a) Test procedure (dry, damp or wet);
- b) Temperature of the heating device;
- c) Numerical rating for change in colour immediately after testing and after conditioning for 4 h in the standard atmosphere for testing textiles, and
- d) Numerical rating for the staining of cotton adjacent fabric.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO STEAMING UNDER ATMOSPHERIC PRESSURE

(Source : IS 974 : 1984)

The method prescribed is based on ISO 105/E-1978 Textiles — Tests for colour fastness E11 —

Colour fastness to steaming issued, by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials, irrespective of their composition (cotton, silk, man-made fibres, etc) and form (fibre, yarn or fabric) to the action of steaming under atmospheric pressure.

2 PRINCIPLE

2.1 A specimen of textile in contact with pieces of specified adjacent fabrics is rolled into the form of a cylinder and placed in the neck of the flask through the means of glass tube. The specimen is then subjected to steaming by boiling water contained in the flask. The staining of the pieces of adjacent fabrics is assessed with standard grey scale.

3 SAMPLING

3.1 The sample shall be so drawn as to be

representative of the lot. Sample drawn in accordance with the procedure laid down in the material specifications or as agreed to between the buyer and the seller shall be representative of the lot.

4 APPARATUS

4.1 Glass Tube Open at Both Ends

with 3 cm inside diameter, mounted in a cork stopper and fitted into the neck of a wide-neck conical flask of about 2-litre capacity. A wire ring shall be fixed in the cork stopper with the loop covered with a thin fabric to catch spray. The flask shall contain about 0.5 litre of distilled water, to which some small beads are added (see Fig. 1).

4.2 Adjacent Fabric (10 cm × 4 cm)

undyed, free from size and other finishing materials and of the same fibre as the specimen (see Annex A).

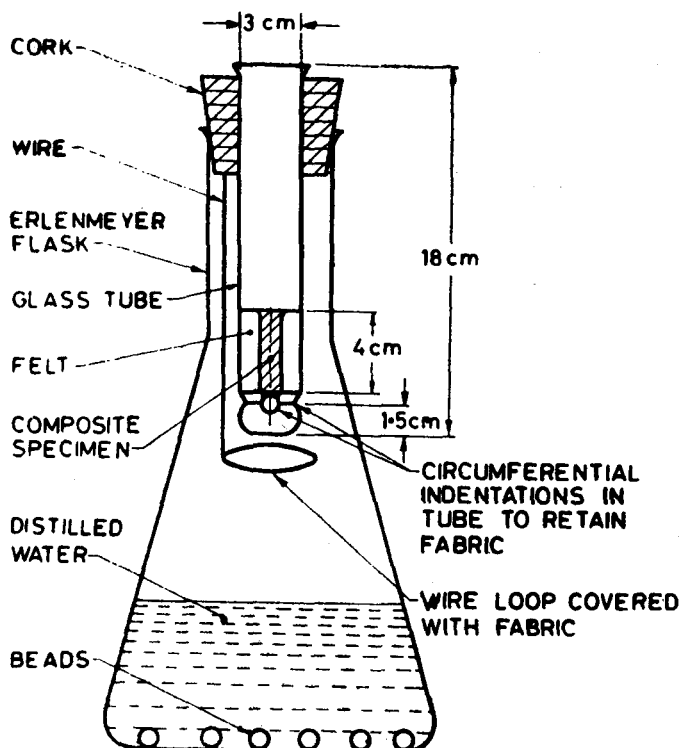


FIG. 1 APPARATUS FOR COLOUR FASTNESS TO STEAMING UNDER ATMOSPHERIC PRESSURE

NOTES

1 The length of 10 cm may be reduced if the cylindrical specimen to be tested is too thick for the cylinder to be inserted into the tube (see 4.1).

2 The constructional and other requirements for adjacent fabrics, as given in Annex A, are for guidance only.

4.3 Two Cotton Adjacent Fabrics (Each 10 cm × 4 cm)

undyed, free from size and other finishing materials and of plain weave (see Note 2 under 4.2 and Annex A).

4.4 Undyed Scoured Wool Felt

4.5 Grey Scale

for assessing staining.

5 REAGENTS

5.1 Distilled Water

6 COMPOSITE SPECIMEN

6.1 Preparation of Test Pieces

6.1.1 If the material to be tested is fabric, draw a 10 cm × 4 cm test piece from each piece in the test sample (see Note 1 under 4.2).

6.1.2 If the material to be tested is yarn, knit or weave the test sample into fabric and draw from it three 10 cm × 4 cm test pieces; alternatively, prepare three 10 cm × 4 cm test pieces in the form of parallel lines of yarn.

6.1.3 If the material to be tested is loose fibre, comb and compress the amount of material weighing approximately equal to the mass of the cotton adjacent fabric into a sheet to make a 10 cm × 4 cm test piece. Similarly, prepare two more test pieces from the test sample.

6.2 Preparation of Composite Specimen

6.2.1 Prepare a composite specimen measuring 10 cm × 4 cm by placing successively on a piece

of cotton adjacent fabric (4.3), a piece of test specimen (6.1), a piece of adjacent fabric (4.2) and a further piece of cotton adjacent fabric (4.3). Roll this composite specimen into the form of a cylinder, with the cloth being tested as near the middle as possible.

NOTE — To facilitate rolling, one short side of the composite specimen may be stitched.

6.2.2 Similarly, prepare the other two composite specimens.

7 PROCEDURE

7.1 Bring the water in the conical flask to boil. Wrap the cylindrical composite specimen in felt (see 4.4) so that it fits easily in the glass tube in the neck of the flask and is retained by the indentations in the lower part of the tube (see Fig. 1). Boil for 30 minutes.

7.2 Remove the composite specimen from the tube, separate the components and dry by hanging in air at a temperature not exceeding 60°C.

7.3 Evaluate the staining of the adjacent fabrics with the help of grey scale and assign the ratings.

NOTES

1 The pieces of adjacent fabrics should be allowed to cool after drying and to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.4 Repeat the test with the remaining composite specimens.

8 REPORT

8.1 Report individually the numerical ratings for staining of the adjacent fabrics used in the preparation of the composite specimen.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass in g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
Cotton	115 ± 5	1/1 Plain weave	35	31	16.5	14
Viscose	140 ± 5	1/1 Plain weave	28	22	20	33
Wool	125 ± 5	1/1 Plain weave	21 ± 0.5	18 ± 0.5	15.6 × 2	15.6 × 2
	— 0				Worsted	Worsted
Polyamide	130 ± 5	1/1 Plain weave	17.5	20	10 × 2	20
Polyester	130 ± 5	1/1 Plain weave	23.5	20.5	7.5 × 2	20
Acrylic	135 ± 5	1/1 Plain weave	17.5	16	10 × 2	10 × 2

NOTES

1 For wool adjacent fabric, additional requirements are: (a) pH value of aqueous extract 6.5 to 7.5, (b) residual fat content 0.4 ± 0.1 percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic, additional requirements are: (a) pH of aqueous extract 7 ± 0.5, and (b) residual oil content less than 1.0 percent.

3 For polyester additional requirement is residual oil content less than 0.5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO DRY-HEAT TREATMENTS (EXCLUDING PRESSING)

(Source : IS 4636 : 1988)

The method prescribed in this standard is based on ISO 105/P-1978 Textiles — Tests for colour fastness P01 — Colour fastness to dry heat

(excluding pressing), issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for determination of colour fastness of textile materials of all kinds and in all forms to the action of dry heat as employed in processes designed to give textiles stability of dimensions or shape such as pleating and setting. It is mainly applicable to synthetic fibres or textiles containing synthetic fibres.

1.1.1 Three tests differing in temperature are provided; one or more of them shall be used depending on the requirements and the stability of the fibres.

1.2 When this method is used for assessing colour change and staining in dyeing, printing and finishing processes; it should be recognized that other chemical and physical factors may influence the results.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is heated by close contact with a medium which in turn, is heated to the required temperature. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in accordance with the relevant material specifications as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 PREPARATION OF COMPOSITE SPECIMEN

4.1 If the textile to be tested is fabric, place a test specimen of suitable size from the sample (see Note) adapted to that of heating device (see 5.1) between the two pieces of adjacent fabrics and sew along one of the shorter sides to form a composite specimen.

NOTE — The size of the specimen should be such that the pressure on the composite specimen would amount to 4 ± 1 kPa.

4.2 If the textile to be tested is yarn, knit or weave it into a fabric and prepare the composite specimen as in 4.1 or alternatively, form a layer of parallel lengths of yarn between the two pieces of adjacent fabrics; the amount of yarn taken being approximately half of the combined mass of the adjacent fabrics, sew along two opposite sides to hold the yarn in place and to form a composite specimen.

4.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics into a sheet of required size (see Note under 4.1). Place the sheet between the two pieces of adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen.

5 APPARATUS

5.1 A Suitable Heating Arrangement

providing even heat transfer at controlled temperature by close contact with the composite specimen.

NOTES

1 A pair of hot-plates equipped with an electrical heating system, which is accurately controllable, is suitable. In order to obtain a pressure of 4 ± 1 kPa the total area of the composite specimen, shall bear a suitable relationship to the mass of the plate pressing down on the specimen.

2 A molten metal-bath, in which a holder (see Fig. 1) containing the composite specimen is immersed, is also suitable. The metal alloy of the following composition should be used:

Alloy	Percent
Bismuth	50
Lead	26.6
Tin	13.4
Cadmium	10.0

3 Other devices may also be used provided that the same results are obtained as with the apparatus described in 5.1.

5.2 Aluminium Foil

of thickness 0.001 to 0.002 cm.

5.3 Two Adjacent Fabrics

each of size adapted to that of heating device, one piece made of the same fibre as that in the sample to be tested or that predominating in case of blends, the second piece made of polyester fibre, unless otherwise specified (see Annex A of IS 10251 : 1982).

5.4 Grey Scales

for evaluating change in colour and staining.

6 PROCEDURE

6.1 Place the composite specimen in the heating device with a pressure of 4 ± 1 kPa on it or, alternatively, cover both sides of the composite specimen with aluminium foil and mount in the holder (see Fig. 1 and 2). Leave it there for 30 seconds at one or more of the following temperatures:

Test	Temperature
Mild	$150 \pm 2^\circ\text{C}$
Intermediate	$180 \pm 2^\circ\text{C}$
Severe	$210 \pm 2^\circ\text{C}$

NOTES

- 1 The composite specimen should be placed after the required temperature is attained.
- 2 When desired, other temperatures and/or times may be used, provided that they are specially noted in the test report.

6.2 Remove the composite specimen and leave it in air at $27 \pm 2^\circ\text{C}$ and 65 ± 2 percent relative humidity for 4 hours.

6.3 Evaluate the change in colour of the test specimen and the staining of the two pieces of adjacent fabrics with grey scales.

NOTES

- 1 Treated test specimens and the piece of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.
- 2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

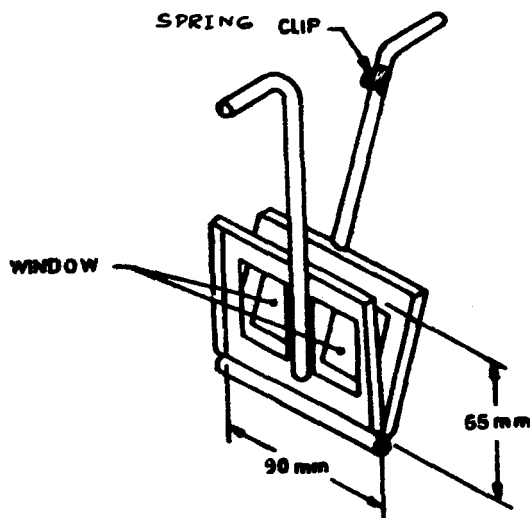


FIG. 1 HOLDER

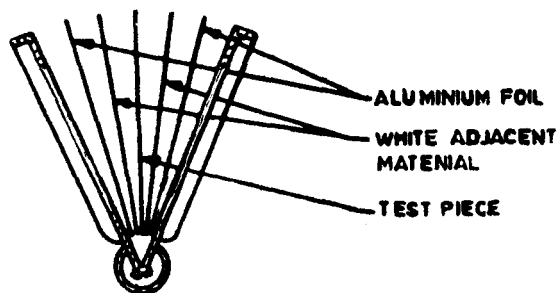


FIG. 2 LOADING ARRANGEMENT

7 REPORT

7.1 Report individually the numerical ratings for the change in colour of the specimen and for staining of each kind of adjacent fabric used in the preparation of composite specimen.

7.2 Report also the test used, that is, mild, intermediate or severe.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO STEAM UNDER PRESSURE

(Source : IS 4637 : 1988)

The method prescribed in this standard is based on ISO 105/P-1978 Textiles — Tests for colour fastness P02 — Colour fastness to pleating: Steam pleating, issued by the International Organization for Standardization (ISO).

The process of pleating is carried out to give textiles the stability of dimensions or shape. The colour of textile materials is likely to be affected when they are pleated by the action of dry heat or steam under pressure.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of steam under pressure. The materials are not pleated during test and it is emphasized that it is not intended for assessing the quality of pleating process.

1.2 Three tests, differing in severity, are prescribed. One or more of them shall be used depending on the requirements.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is steamed under pressure and dried. The numerical ratings for change in colour of the specimen and staining of the adjacent fabrics are evaluated with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Two Adjacent Fabrics

each measuring 10×4 cm, made of same kind of fibre as that of the textile to be tested or otherwise specified. In the case of blends, two different adjacent fabrics are required corresponding to the two predominant fibres of the specimen or as otherwise specified (see Annex A of IS 10251 : 1982).

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, place a specimen 10×4 cm between two adjacent fabrics and sew along all four sides to form a composite specimen.

4.2.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 4.2.1, or form a layer of parallel lengths of it between the two adjacent fabrics, the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics, sew along all four sides to hold the yarn in place and to form a composite specimen.

4.2.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics, into a sheet 10×4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen.

5 APPARATUS

5.1 Specimen Holder

consisting of a copper tube 80 mm in external diameter and 1.5 mm in thickness. The copper tube is wrapped with six layers of bleached cotton cloth (mass 125 g/m^2 approximately). The outside layer is made of bleached cotton cloth (mass 185 g/m^2 approximately). The rods at each end are made from 6 mm diameter mild steel. The strength of the springs is not critical but it must be sufficient to hold the layer tightly against the tube. The springs are fastened to one of the steel rods and should easily hook on to the other steel rod (see Fig. 1).

5.2 Jacketed Steamer

provided with an instrument to measure the pressure accurately and that no water splashes on to the specimen during the test.

or

5.3 Domestic Pressure Cooker

sufficiently large to avoid water splashing on the specimen during test.

NOTE — It is suggested that the minimum size should be 230 mm in diameter and 260 mm in height. It must be fitted with an accurate pressure gauge. If the domestic pressure cooker is used, the holder must be loosely wrapped in one layer of polyester film which projects 10 mm over each end of the holder and is not closed at the ends. The holder should then be placed in a rectangular metal container having ten holes of 1 mm diameter equally spaced along

the centre of the bottom and should be sufficiently deep to reach 10 mm from the top of the specimen holder (see Fig. 2). The bottom of the container should be slightly concave to ensure that condensed water rapidly drains out. The container is then placed on a stand which holds it 50 mm from the surface of the water. It is suggested that the depth of water should be 30 mm. Before raising the pressure in the cooker, it is suggested that all the air should be expelled over a period of 2 minutes.

5.4 Grey Scale

for evaluating change in colour and staining.

6 PROCEDURE

6.1 Mount the composite specimen in a holder between two adjacent fabrics as shown in Fig. 1.

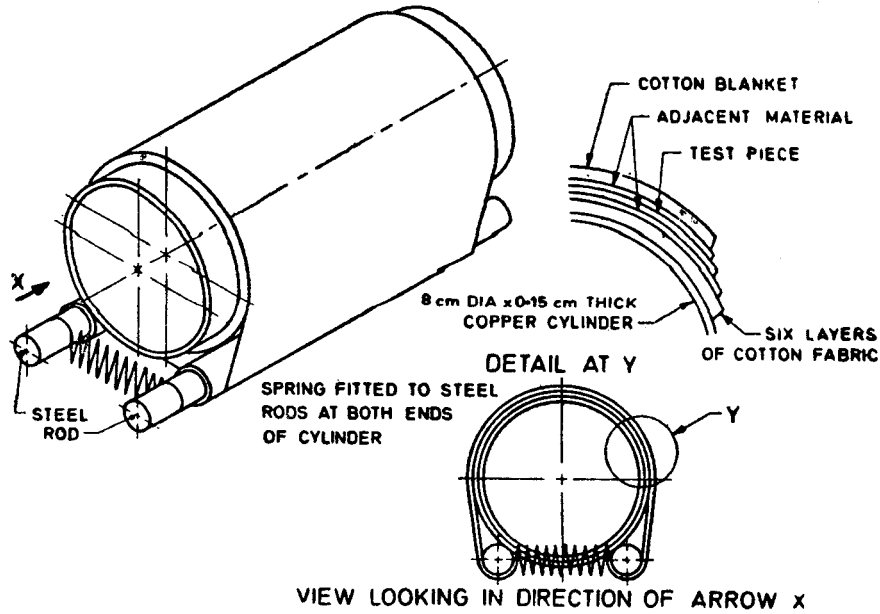


FIG. 1 SPECIMEN HOLDER

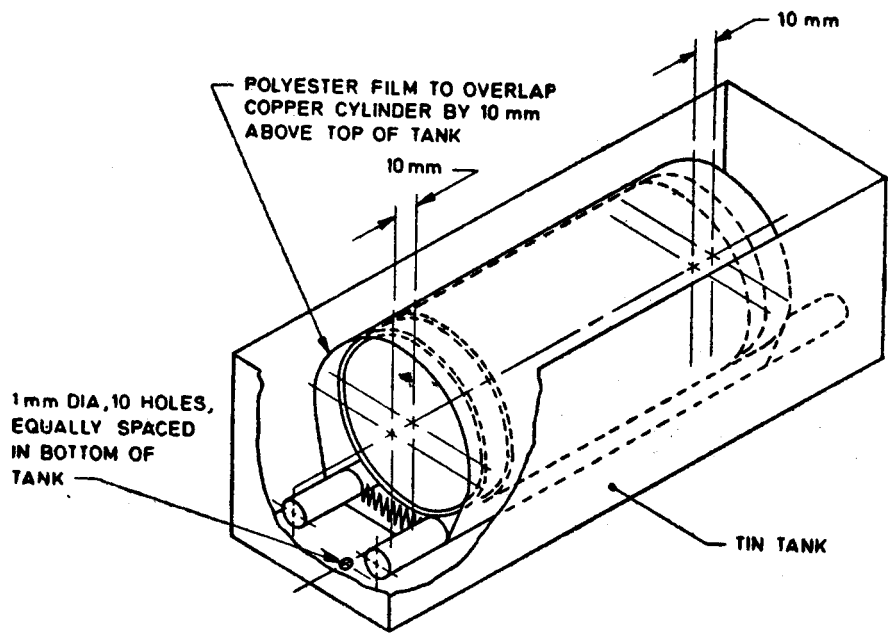


FIG. 2 ASSEMBLY

6.2 Place the holder containing the composite specimen in a jacketed steamer or pressure cooker. Steam under one of the following sets of conditions:

<i>Test</i>	<i>Maximum Duration of Heat- ing up Period (min)</i>	<i>Duration of Time at Speci- fied Tem- perature (min)</i>	<i>Press- ure (kPa)</i>	<i>Tempe- rature (°C)</i>
Mild	5	5	135	108
Inter- mediate	8	10	170	115
Severe	15	20	270	130

NOTES

1 The severe test is intended primarily for 100 percent synthetic textiles such as those made from polyamide and polyester fibres. It should not be used for textile containing wool.

2 Specimens which liberate formaldehyde under steam pleating conditions should be tested separately.

3 If wool is used as one of the adjacent fabrics, it may have an adverse effect on the dye in the specimen, particularly under alkaline conditions.

4 It should be noted that the papers used in commercial pleating occasionally contain reducing agents which, with certain colouring matters, can produce a much greater change in colour than occurs under test conditions.

6.3 After completing the steaming, release the pressure over a period not exceeding two minutes. Open out the composite specimen and dry in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the line of stitching. Condition the test specimen at $27 \pm 2^\circ\text{C}$ and 65 ± 2 percent relative humidity for four hours.

6.4 Evaluation

Evaluate the change in colour of the test specimen and the degree of staining of the two pieces of adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7 REPORT

7.1 Report the numerical rating for change in colour of test specimen and the numerical ratings for staining of the two adjacent fabrics used in the preparation of the composite specimen.

7.2 Report also the test whether mild, intermediate or severe, which has been used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ORGANIC SOLVENTS

(Source : IS 688 : 1988)

This standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X05 — Colour fastness to organic solvents, issued by the

International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to organic solvents commonly used in cleaning or mill processing. If dry-cleaning is involved, use the method prescribed in IS 4802 : 1988.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is agitated in the solvent and dried. The change in colour of the specimen and staining of the adjacent fabrics are evaluated with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Suitable Container

with means of agitation.

NOTE — A 500 ml beaker or other suitable open container may be used for the test, agitation being done by hand with a glass rod flatted at one end. A closed vessel agitated by shaking or tumbling by hand or motor-driven machine may also be used.

4.2 Two Adjacent Fabrics

each measuring 10 cm × 4 cm, one piece made of same kind of fibre as that of the textile to be tested, or that predominating in the case of blends, the second piece made of the fibre as indicated below or in the case of blends, of the kind of fibre second in order of predominance or as otherwise specified (see Annex A of IS 10251 : 1982).

If the First Piece is

Cotton

Wool

Silk

Linen

Viscose

Acetate or triacetate

Polyamide

Polyester

Acrylic

Second Piece to be

Wool

Cotton

Cotton

Wool

Wool

Viscose

Wool or viscose

Wool or cotton

Wool

4.3 Grey Scales

for evaluating change in colour and staining.

5 REAGENT

5.1 Solvent

any one of the solvents commonly used in cleaning or mill processing.

NOTES

1 It is anticipated that the test will be made with trichloroethylene or with perchloroethylene and with Stoddard solvent (specially specified naphtha for use of dry-cleaners). Other solvents, for which information is desired, may be used. The use of trichloroethylene is recommended for rating dye-stuffs.

2 Halogenated hydrocarbons should be stored over anhydrous sodium carbonate to neutralize hydrochloric acid.

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 If the textile to be tested is fabric, place a specimen 10 cm × 4 cm between two adjacent fabrics and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 6.1, or form a layer of parallel lengths of it between the two adjacent fabrics, the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides

to hold the yarn in place and to form a composite specimen.

6.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics into a sheet 10 cm × 4 cm. Place the sheet between the two adjacent fabrics and sew along all four sides to hold the fibre in place and to form a composite specimen.

7 PROCEDURE

7.1 Place the composite specimen and the solvent in the container, the liquor ratio being 1 : 40. Agitate the specimen by hand or by a machine continuously for 30 minutes in the solvent at room temperature. If the agitation is by hand, the specimen should be pressed against the container every 2 min with a glass rod without removing the specimen from the solvent. Remove the specimen, squeeze excess solvent from it and dry in air in shade at $80 \pm 2^\circ\text{C}$ without unstitching. Take the precautions necessary for safety in drying inflammable or explosive solvent. Remove the stitching.

7.2 Evaluate the change in colour of the treated specimen and the degree of staining of the two pieces of adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report shall include the following:

- a) Solvent used,
- b) Numerical rating for change in colour of test specimen, and
- c) Numerical ratings for staining of the two pieces of adjacent fabrics used in the preparation of the composite specimen.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO RUBBING

(Source : IS 766 : 1988)

This standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X12 — Colour

fastness to rubbing, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms including textile floor coverings and other pile fabrics, to rubbing off and staining other materials. The method is applicable to a lid textile floor covering or to a detached sample or yarns. Two tests are made, one with a dry rubbing cloth and the other with a wet rubbing cloth.

2 PRINCIPLE

2.1 Specimens of the textile are rubbed with dry rubbing cloth and with wet rubbing cloth. Two alternative sizes of rubbing finger are specified, one for pile fabrics and the other for textiles. The staining of the rubbing cloth is assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Suitable Testing Device

for determining the colour fastness to rubbing. Such a device has one of two alternative sizes of rubbing finger, depending upon the type of textile to be tested as follows.

4.1.1 *For Pile Fabrics Including Textile Floor Covering*

a rubbing finger of 3.2 cm diameter and flat area of 2.5 cm diameter with a circular transition of 0.32 cm radius. The rubbing finger exerts a downward force of 22 N, moving to and fro in a straight line along a 10-cm track.

NOTE — A suitable device is the crockmeter of the type developed by the American Association of Textile Chemists and Colorists (AATCC) — USA.

4.1.2 *For All Other Textiles*

a rubbing finger comprising a cylinder of 1.6 cm diameter moving to and fro in a straight line along a 10.0 cm track on the specimen with downward force of 9 N.

4.2 Grating of Stainless Steel Wire

1 mm diameter and a width of mesh of about 20 mm.

4.3 Rubbing Cotton Cloth

desized, bleached, without finish and cut into 5 cm × 5 cm squares.

4.4 Grey Scale

for evaluating staining.

5 PREPARATION OF COMPOSITE SPECIMEN

5.1 If the textile to be tested is a fabric or textile floor covering, draw from the sample two pieces each not less than 14 cm × 5 cm for dry rubbing and two pieces for wet rubbing. One specimen of each pair has the direction parallel to the warp yarns (or in the direction of manufacture) and the other parallel to the weft or filling yarns (or at right angles to the direction of manufacture).

5.2 If the textile to be tested is yarn or thread, knit or weave it into fabric to provide four specimens at least 14 cm × 5 cm, two for dry rubbing and two for wet rubbing. Alternatively, prepare four specimens by forming a layer of parallel strands by wrapping it lengthways on a cardboard rectangle of suitable dimensions.

6 PROCEDURE

6.1 Fix the test specimen to the rubbing device by means of clamps such that the long direction of the specimens follows the track of the device. Test the specimens according to the procedure given in 6.3 and 6.4.

6.2 When testing multicoloured textiles, care should be taken to position the specimen in such a way that all colours of the design are rubbed in the test. Alternatively, if the areas of colour are sufficiently large, more test specimens may be taken and individual colour assessed separately.

6.3 Dry Rubbing

with the dry rubbing cloth flat in place over the end of the finger of the testing device, rub it to and fro in a straight line along a track 10 cm long on the dry specimen, 10 times to and fro in 10 seconds, with a downward force of 22 N or 9 N on the finger (*see* 4.1.1 and 4.1.2).

NOTE — It is necessary to eliminate dyed fibres pulled out during rubbing and retained on the surface of the rubbing cotton cloth; consider only the colouration due to staining by the dyestuff.

6.4 Wet Rubbing

Repeat the test described in 6.3 with a fresh dry specimen and with a rubbing cloth that has been wetted with water by placing it on the grating and dropping evenly on to it its own mass of water, or use any method to ensure a take up of about 100 percent. After rubbing, dry the cloth at room temperature (*see* Note under 6.3).

6.5 Assess the staining of the rubbing cotton cloths with grey scale for evaluating staining.

NOTES

1 Difficulty may be experienced in making assessments of the degree of staining on the rubbing cloth when pile fabrics are tested using 1.6 cm diameter finger due to heavier staining occurring on the circumference of the stained area, that is, haloing. The 3.2 cm diameter rubbing finger will eliminate the haloing in many types of pile fabrics. Even with the use of the larger diameter rubbing finger, difficulty may be experienced in assessing staining when fabrics with high pile are tested.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and overall average rating should be reported.

7 REPORT

7.1 Report the numerical rating for dry staining and for wet staining of the rubbing cotton cloth for each direction of manufacture.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO ACID CHLORINATION

(Source : IS 967 : 1956)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different fibres or when applied by different methods upon the same fibre, may give vastly different results.

This method of test is intended for evaluating colour fastness of textile materials to the manufacturing operation in which an acid hypochlorite solution is used to prevent woollen textile materials from shrinking. The method prescribed is based on ISO 105/X-1984 Textiles — Tests for colour fastness X03 — Colour fastness to chlorination, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials to the action of acid hypochlorite solution.

1.2 The method is mainly applicable to materials containing wool.

2 SAMPLING

2.1 Sample shall be selected so as to be representative of the lot.

2.2 Sample drawn in compliance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be representative of the lot.

3 COMPOSITE SPECIMEN

3.1 Undyed Material

Take sufficient lengths of yarns of scoured unbleached undyed wool, undyed bleached cotton and other fibres, as desired for testing yarns and fabrics; take sufficiently large sized comparable pieces of adjacent fabrics for use in testing loose fibre.

3.2 Test Piece

3.2.1 If the material to be tested is fabric, draw a 10 cm × 4 cm test piece from the sample.

3.2.2 If the material to be tested is yarn, knit or weave some of the sample into fabric and draw from it a 10 cm × 4 cm test piece.

3.2.3 If the material to be tested is loose fibre, comb and compress enough of the sample into the form of a sheet to make a 10 cm × 4 cm test piece.

3.3 Preparation of Composite Specimen

3.3.1 If the test piece is in the form of fabric (see 3.2.1 and 3.2.2), sew stitches of the undyed yarns (see 3.1) at intervals of approximately

1 cm into the test piece to form a composite specimen.

3.3.2 If the test piece is in the form of a sheet of fibre, place it between the two pieces of adjacent fabric (see 3.1) and sew the three together with stitching at intervals of 1 cm to form a composite specimen. The mass of the test piece should approximate to that of the wool adjacent fabric.

4 COMPOSITE TEST CONTROL

4.1 Test Control

a dyeing of 1 percent Solway Blue GS on woollen cloth shall be used as a test control to ensure that the test is carried out correctly.

NOTE — For preparation of the dyeing, see Annex A.

4.2 Preparation of Composite Test Control

4.2.1 Prepare a composite test control in the way outlined for fabric (see 3.3.1).

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

6 APPARATUS

6.1 Container

made of glass or glazed porcelain with provision for closing.

6.2 Grey Scales

for evaluating change in colour and staining.

7 REAGENTS

7.1 Hydrochloric Acid Solution

containing 6 ml of hydrochloric acid (sp gr 1.16) per litre.

7.2 Sodium Hypochlorite Solution

containing 1 g of available chlorine per litre. Sodium hypochlorite shall be of the following composition:

	Percent
Sodium hypochlorite (NaOCl)	14 to 16
Sodium chloride (NaCl)	12 to 17
Sodium hydroxide (NaOH), <i>Max</i>	2.0
Sodium carbonate (Na ₂ CO ₃), <i>Max</i>	2.0
Iron, <i>Max</i>	0.001

7.3 Sodium Sulphite Solution

containing 3 g of sodium sulphite (Na₂SO₃.7H₂O) per litre.

8 PROCEDURE

8.1 Immerse the composite specimen (*see also 8.1.1*) in the hydrochloric acid solution for 10 minutes at room temperature, liquor ratio being 1 : 25. Add an equal volume of sodium hypochlorite solution, close the container and keep the specimen immersed for a further period of 10 minutes. Remove the composite specimen, rinse it thoroughly in cold running tap water and then immerse in sodium sulphite solution for 10 minutes at 35 to 40°C at a liquor ratio of 1 : 50. Remove the specimen, rinse it thoroughly in cold running water and dry in air in shade at a temperature not exceeding 60°C.

8.1.1 Treat the composite test-control in the manner prescribed in 8.1 in parallel but in a

separate bath.

8.2 Evaluate the change in colour of the treated test control with the help of grey scale and assign the rating. If the numerical rating for change in colour is not equal to 3, repeat the whole test (*see 8.1 and 8.1.1*) using a fresh composite specimen and fresh composite test control.

NOTES

1 Treated test control and the undyed adjacent material should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8.3 Evaluate the change in colour of the treated test piece and the degree of staining of each type of undyed adjacent material with the help of grey scale and assign the rating.

NOTES

1 Treated piece and the undyed adjacent material should have cooled after drying and regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

9 REPORT

9.1 The report shall include the following information:

- Numerical rating for change in colour of the test piece, and
- Numerical rating for staining of each kind of undyed adjacent material.

ANNEX A

PREPARATION OF DYEING

A-1 Wet out a sufficiently large sized piece of woollen cloth and enter it at 40°C into a dye bath containing 1 percent Solway Blue GS (Colour Index, Blue 37); 10 percent sodium sulphate crystals (Na₂SO₄.10H₂O) and 3 percent sulphuric acid (sp gr 1.84), all percentages

being calculated on the mass of the piece, at a liquor ratio of 1 : 40.

A-2 Raise the dye bath to the boil in 15 minutes and continue further for 45 minutes. Remove the piece, rinse it in cold running tap water and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO CROSS-DYEING : WOOL

(Source : IS 969 : 1956)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different fibres

or when applied by different methods upon the same fibre, may give vastly different results. The method prescribed is based on ISO 105/X-1984 Textiles — Tests for colour fastness X07 — Colour fastness to cross-dyeing: Wool, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials to processes used for dyeing wool.

2 SAMPLING

2.1 Sample shall be selected so as to be representative of the lot.

2.2 Sample drawn in compliance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be representative of the lot.

3 COMPOSITE SPECIMEN

3.1 Ten Adjacent Fabrics

each measuring 10 cm × 4 cm, five pieces being made of the same kind of fibre as that in the sample or that predominating in the sample in case of blends, and the other five pieces being made of the fibre as indicated below or as otherwise specified (see also Annex A of IS 10251 : 1982):

<i>If the First Piece Consists of</i>	<i>Second Piece shall be</i>
Cotton	Wool
Wool	Cotton
Silk	Wool
Linen	Wool
Viscose	Wool
Acetate	Wool
Polyamide	Wool
Polyester	Wool
Acrylics	Wool

3.2 Test Pieces

3.2.1 If the material to be tested is fabric, draw five 10 × 4 cm test pieces from the sample.

3.2.2 If the material to be tested is yarn, knit or

weave some of the sample into fabric and draw from it five 10 × 4 cm test pieces.

3.2.3 If the material to be tested is loose fibre, comb and compress enough of the sample into the form of a sheet and draw from it five 10 × 4 cm test pieces.

3.3 Preparation of Composite Specimens

3.3.1 Place the test piece between two 10×4 cm pieces each drawn from one of the two types of adjacent fabrics (see 3.1) and sew along all four sides to form a composite specimen. Prepare five such specimens.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

5 APPARATUS

5.1 A number of dye vessels suitable for treatment shall be used.

5.2 Grey Scales

for evaluating change in colour and staining.

6 REAGENTS

6.0 The following reagents shall be used.

6.1 Sodium Sulphate Crystals ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$)

6.2 Acetic Acid

30 percent.

6.3 Sulphuric Acid

sp gr 1.84.

6.4 Potassium Dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$)

solid.

7 PROCEDURE

7.1 Carry out the operations prescribed in 7.2 to 7.6 at a liquor ratio of 1 : 50 calculating the percentages indicated on the mass of the wool, silk or polyamide or similar (undyed) cloth in the composite specimen.

7.2 Neutral Cross-Dyeing

Enter one of the composite specimens into a bath containing 20 percent sodium sulphate crystals. Raise the bath to $98 \pm 2^\circ\text{C}$ in 30 minutes and keep at this temperature for 90 minutes.

7.3 Acetic Acid Cross-Dyeing

Enter the second composite specimen into a bath containing 5 percent acetic acid and 20 percent sodium sulphate crystals. Raise the bath to $98 \pm 2^\circ\text{C}$ in 30 minutes and keep at this temperature for 90 minutes.

7.4 Sulphuric Acid Cross-Dyeing

Enter the third composite specimen into a bath containing 20 percent sodium sulphate crystals and 4 percent sulphuric acid. Raise the bath to $98 \pm 2^\circ\text{C}$ in 30 minutes and maintain at this temperature for 90 minutes.

7.5 Acetic Acid-Chrome Cross-Dyeing

Enter the fourth composite specimen into a bath containing 20 percent sodium sulphate crystals and 5 percent acetic acid. Raise the bath to $98 \pm 2^\circ\text{C}$ in 30 minutes and maintain at this temperature for 30 minutes. Add 2 percent potassium dichromate and maintain the bath at $98 \pm 2^\circ\text{C}$ for a further 60 minutes.

7.6 Sulphuric Acid-Chrome Cross-Dyeing

Enter the fifth composite specimen into a bath

containing 20 percent sodium sulphate crystals and 5 percent of 30 percent acetic acid solution. Raise the bath to $98 \pm 2^\circ\text{C}$ in 30 minutes and maintain at this temperature for 30 minutes. Add 2 percent sulphuric acid and maintain the bath at $98 \pm 2^\circ\text{C}$ for a further period of 15 minutes. Add 2 percent potassium dichromate and maintain at $98 \pm 2^\circ\text{C}$ for a further period of 60 minutes.

7.7 Rinse the composite specimens in cold running tap water, separate the test piece and the two pieces of adjacent fabric from each of the composite specimens and dry them apart in air at a temperature not exceeding 60°C .

7.8 Evaluate the change in colour of each of the treated test pieces and the degree of staining of the corresponding two pieces of adjacent fabric with the help of grey scales and assign the ratings.

NOTES

1 Treated test pieces and the pieces of adjacent fabric should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 The report shall include the following:

- a) Numerical rating for change in colour of the test piece, and
- b) Numerical ratings for staining of the two pieces of adjacent fabrics for each kind of cross-dyeing liquor used.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO DEGUMMING

(Source : IS 970 : 1988)

This standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X08 — Colour fastness to degumming, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds, except loose fibres, to the action of soap solution such as those used in removing natural gum from raw silk.

2 PRINCIPLE

2.1 A specimen of the textile in contact with adjacent fabrics is treated with soap solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Conical Flask

of 500 ml capacity with a water cooled 'finger' condenser fitting loosely in the neck.

4.2 Two Adjacent Fabrics

10 cm × 4 cm, one piece made of raw silk, the other made of the same kind of fibre as that of the textile to be tested or that predominating in the case of blends (see Annex A of IS 10251 : 1982).

4.3 Grey Scales

for evaluating change in colour and staining.

5 REAGENTS

5.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 Soap Solution

The soap solution shall contain 7 g of soap per litre of distilled water. The soap shall conform to the following requirements based on dry mass and shall not have moisture regain more than 5 percent. The soap shall be free from optical brightening agents:

Characteristic	Requirement
Free alkali, calculated as Na_2CO_3 , <i>Max</i>	3 g/kg
Free alkali, calculated as NaOH , <i>Max</i>	1 g/kg
Total fatty matter, <i>Min</i>	850 g/kg
Titre of mixed fatty acids prepared from the soap, <i>Max</i>	39°C
Iodine value, <i>Max</i>	50

5.3 Sodium Carbonate

anhydrous.

6 PREPARATION OF TEST SPECIMENS

6.1 If the textile to be tested is a fabric, place a specimen of 10 × 4 cm between two adjacent fabrics of the same size (see 4.2) and sew along all four sides to form a composite specimen.

6.2 If the textile to be tested is yarn, knit or weave it into the fabric and treat as prescribed in 6.1, or form a layer of parallel lengths of it between the two pieces of adjacent fabrics (see 4.2); the amount of yarn taken being approximately equal to half the combined mass of the adjacent fabrics. Sew along all four sides to hold the yarn in place and to form a composite specimen.

7 PROCEDURE

7.1 Treat the composite specimen in the flask (see 4.1) for 10 minutes under reflux with a lightly boiling soap solution (see 5.2) at liquor ratio of 1 : 100.

7.2 After 10 minutes, add 0.5 g/l of anhydrous sodium carbonate to the boiling soap solution, and keep boiling lightly for 110 minutes (2 hours in all).

7.3 Remove the composite specimen from the soap solution, rinse twice in cold distilled water and then for 10 minutes in cold running soft water. Squeeze it thoroughly, open out the composite specimen by breaking the stitching on all but one of the shorter sides and dry it by hanging in air in shade at a temperature not exceeding 60°C, with the three parts in contact only at the remaining line of stitching.

7.4 Evaluate the change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics with grey scales.

NOTES

1 Treated test specimens and the pieces of adjacent fabrics should have cooled after drying and should

have regained their normal moisture content before evaluation.

2 In cases of doubt in the assessment of colour fastness ratings by a single observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 The report shall include the following information:

- a) Type of material,
- b) Numerical ratings for change in colour of the test specimen, and
- c) Numerical ratings for staining of the two adjacent fabrics.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO SODA BOILING

(Source : IS 973 : 1988)

The method prescribed in this standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X06 — Colour fastness to soda

boiling, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the action of boiling dilute sodium carbonate solution.

1.1.1 The method is mainly applicable to natural and regenerated cellulosic textile materials.

1.1.2 Two tests, one with and the other without the addition of a reduction inhibitor, are prescribed.

2 PRINCIPLE

2.1 A specimen of the textile between specified undyed cloths is rolled around a glass rod and treated with boiling sodium carbonate solution with and without the addition of a reduction inhibitor. The composite specimen is rinsed, the cloths are separated and dried. The change in colour of the specimen and the staining of the undyed cloths are assessed with grey scales.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 APPARATUS

4.1 Vessel

equipped with water-cooled reflux condenser of the finger type to hold a cylindrical specimen 4 cm long in the boiling solution.

4.2 Glass Rod

5 to 8 mm in diameter.

4.3 Adjacent Fabric

measuring 10×4 cm of the type under test or, if fibre or yarn is being tested, adjacent fabric made from the same kind of fibre (see Annex A of IS 10251 : 1982).

4.4 Desized Undyed Cotton Fabric

measuring 10×4 cm. This material is *not* cotton adjacent fabric.

4.5 Test Control

dyeings of CI Vat Red 1 (Colour Index, 3rd edition).

NOTE — For preparation of the dyeings, see Annex A.

4.6 Grey Scales

for evaluating change in colour and staining.

5 REAGENTS

5.0 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Sodium Carbonate Solution

containing 10 g of anhydrous sodium carbonate per litre.

5.2 Sodium Carbonate-Sodium *m*-Nitrobenzenesulphonate Solution

containing 10 g of anhydrous sodium carbonate per litre and 4 g of sodium *m*-nitrobenzenesulphonate per litre.

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 Two composite specimens, prepared as follows, are required for the tests with and without the addition of a reduction inhibitor.

6.2 If the textile to be tested is fabric, place a specimen of 10×4 cm between one piece of undyed cotton fabric and one piece of adjacent fabric, and sew along one of the shorter sides to form a composite specimen.

6.3 If the textile to be tested is yarn, knit or weave it into fabric and treat as in 6.2, or form a

layer of parallel lengths between the two pieces of undyed cloth, the amount of yarn taken being approximately equal to half the combined mass of the undyed cloths. Sew along two opposite sides to hold the yarn in place and to form a composite specimen.

6.4 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the undyed cloths into a sheet 10×4 cm. Place the sheet between the two undyed cloths, and sew along all four sides to hold the fibre in place and to form a composite specimen.

6.5 Prepare composite specimen of the test control in the way as outlined for fabric in 6.2.

7 PROCEDURE

7.1 Carry out the operations described in 7.2 to 7.4 with each composite specimen and the composite test control specimen in parallel in separate baths.

7.2 Roll the composite specimen compactly around the glass rod to form a cylinder 4 cm long and tie it loosely and uniformly with thread.

7.3 Treat one composite specimen on the rod by boiling gently under reflux for 1 h in sodium carbonate solution at a liquor ratio of 1 : 30. Treat the other composite specimen in the same way and for the same time in boiling sodium carbonate-sodium *m*-nitrobenzenesulphonate solution.

7.4 Remove the composite specimens from the rod immediately, rinse for 10 min in cold running tap water and separate the undyed cloths from them. Open out the composite specimen and dry it by hanging in air in shade at a temperature not exceeding 60°C with the three parts in contact only at the remaining line of stitching.

7.5 Assess the effect on the composite test-control specimen with grey scales. The ratings of the test control specimen after boiling with

sodium *m*-nitrobenzenesulphonate should be:

3-4 weaker, yellower, in respect of change in colour

5 in respect of staining.

The ratings of the test control specimen after boiling without sodium *m*-nitrobenzenesulphonate should be:

2-3 weaker, yellower, in respect of change in colour

2-3 in respect of staining.

If the test control specimens do not yield these values, the test has not been carried out correctly, and the operations described in 7.1 to 7.4 inclusive should be repeated with fresh composite specimen and fresh composite test control specimen.

NOTES

1 Treated test piece or the test control should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.6 Assess the change in colour of the specimen and the staining of the undyed cotton fabric and the adjacent fabric with grey scales (see Notes under 7.5).

8 TEST REPORT

8.1 Report the numerical ratings for change in colour and the numerical ratings for staining of each kind of undyed cloth tested with sodium carbonate alone, and with sodium carbonate and sodium *m*-nitrobenzenesulphonate reduction inhibitor.

8.2 When the two pieces of undyed fabric are the same and the two assessments of staining are different, report only the lower.

ANNEX A

PREPARATION OF DYEING

A-1 Take 3.0 percent of CI Vat Red 1 (Colour Index, 3rd edition) powder, paste it with 150 times its own mass of water using 3 ml of an anionic wetting agent per gram of dye. Add 40 ml of sodium hydroxide (400 g/l), 13 g of sodium hydrosulphite per litre and allow the dye to reduce for 15 minutes at 80°C.

A-2 Take a sufficiently large piece of cotton fabric and set the dye bath at a liquor ratio of

1 : 25. Add to it 2-3 ml of sodium hydroxide (400 g/l), 1 g of sodium hydrosulphite per litre followed by the calculated amount of reduced dye. Enter the piece into the dye bath at 30°C; raise the temperature to 60°C in 15 minutes and continue dyeing at this temperature for 30 minutes. Remove the piece, oxidize it in air, rinse in cold running tap water, soap at the boil, rinse in distilled water, again rinse in cold running tap water and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO SUBLIMATION

(Source : IS 975 : 1988)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of the fibre and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different fibres

or when applied by different methods upon the same fibre, may give vastly different results. Formation of standard methods of test for determining colour fastness of textile materials to different agencies likely to effect change in colour is, therefore, necessary.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to sublimation in storage.

2 PRINCIPLE

2.1 A specimen of the textile in contact with specified adjacent fabrics is rolled in the form of a cylinder and placed in a glass tube, and is heated to the required temperature. The specimen is then removed and cooled, and the degree of staining of the specimen is assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Two Adjacent Fabrics

each measuring 10×4 cm, one piece made of same kind of fibre as that of the textile to be tested or that predominating in the case of blends, the second piece made of polyester fibre, unless otherwise specified (see Annex A of IS 10251 : 1982).

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, place a specimen 10×4 cm between two adjacent fabrics (see 4.1) and sew along one of the shorter sides to form a composite specimen.

4.2.2 If the textile to be tested is yarn, knit or weave it into fabric and draw from it a 10×4 cm specimen. Treat it as in 4.2.1, alternatively, form a layer of parallel lengths of yarn between

the two adjacent fabrics (see 4.1), the amount of yarn taken being equal to half the combined mass of the adjacent fabrics. Sew along two opposite sides to hold the yarn in place and to form a composite specimen.

4.2.3 If the textile to be tested is loose fibre, comb and compress an amount approximately equal to half the combined mass of the adjacent fabrics (see 4.1), into a sheet 10×4 cm size, and sew along all four sides to hold the fibre in place and to form a composite specimen.

5 APPARATUS

5.1 Glass Tube

about 15 mm in diameter.

5.2 Drying Oven

capable of being maintained at required temperature $\pm 2^\circ\text{C}$.

5.3 Grey Scale

for evaluating staining.

6 PROCEDURE

6.1 Roll the composite specimen in the form of a cylinder and place it in the glass tube. Heat the tube (with the specimen) in the oven under either of the following conditions:

Temperature ($^\circ\text{C}$)	Time (seconds)
120 ± 2	120
150 ± 2	30
180 ± 2	30
210 ± 2	30

6.2 Remove the specimen from the tube and unroll it. Evaluate the degree of staining of the pieces of adjacent fabrics with grey scale.

NOTES

1 The treated test specimens and the pieces of adjacent fabric should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7 REPORT

7.1 Report the numerical rating for staining of each piece of adjacent fabric used in the preparation of composite specimen, and the conditions of treatment, that is, the temperature and time.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO CARBONIZING WITH SULPHURIC ACID

(Source : IS 978 : 1988)

The method prescribed in this standard is based on ISO 105/X-1985 Textiles — Tests for colour fastness X02 — Colour fastness to

carbonizing: Sulphuric acid, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials of all kinds and in all forms to the manufacturing operation designed to remove vegetable impurities by a treatment with sulphuric acid at high temperatures.

1.1.1 The method is mainly applicable to wool and textile materials containing wool.

2 PRINCIPLE

2.1 A specimen containing sulphuric acid solution is dried, baked, rinsed and neutralized. The changes in colour after rinsing, neutralizing and drying are assessed with grey scale.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 PREPARATION OF TEST SPECIMEN

4.1 If the textile to be tested is fabric, draw a 10×4 cm specimen from the sample.

4.2 If the textile to be tested is yarn, knit or weave some of the sample into fabric and draw from it a 10×4 cm specimen; alternatively, prepare a specimen in the form of a wick of parallel lengths of yarn 10 cm long and about 0.5 cm in diameter tied near both ends.

4.3 If the textile to be tested is loose fibre, comb and compress enough of the sample into the form of a sheet and draw from it a 10×4 cm specimen.

5 TEST CONTROL

5.1 A dyeing of CI Mordant Red 3 (Colour Index, 3rd edition) treated with potassium

dichromate shall be used as test control to ensure that the test is carried out correctly.

NOTE — For preparation of the dyeings, see Annex A.

6 APPARATUS

6.1 Oven

capable of being maintained at $60 \pm 2^\circ\text{C}$ for drying and at $105 \pm 2^\circ\text{C}$ for baking.

6.2 Grey Scale

for evaluating change in colour.

7 REAGENTS

7.0 Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

7.1 Sulphuric Acid Solution

containing 50 g of concentrated sulphuric acid (sp gr 1.84) per litre.

7.2 Sodium Carbonate Solution

containing 2 g of anhydrous sodium carbonate per litre.

8 PROCEDURE

8.1 Immerse the specimen (see also 8.1.1) in sulphuric acid solution for 15 minutes at room temperature at liquor ratio of 1 : 20. Squeeze it to leave 80 percent of its mass of solution. Dry the specimen by hanging it in the oven for 30 minutes or longer, if necessary, at $60 \pm 2^\circ\text{C}$. Then bake it by heating in the oven for 15 minutes at $105 \pm 2^\circ\text{C}$. Remove the specimen, rinse it for 5 minutes in cold running tap water and then divide it into two equal parts. Dry one half in air in shade at a temperature not exceeding 60°C . Agitate the other half in sodium carbonate solution for 30 minutes at room temperature

using liquor ratio of 1:40, then rinse it for 5 minutes in cold running tap water and dry it in air in shade at a temperature not exceeding 60°C.

8.1.1 Treat the test-control in the manner prescribed in **8.1** in parallel but in a separate bath.

8.2 Evaluate the change in colour of the un-neutralized test control with grey scale (*see* Notes 1 and 2 under **8.2.1**).

8.2.1 The numerical rating for change in colour of the treated test should be 2, yellower. If the test control does not yield this value, then repeat the whole test (*see* **8.1** and **8.1.1**) using a fresh specimen and a fresh test control.

NOTES

1 Treated test specimen or the test control should have cooled after drying and should have regained their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8.3 Evaluate the change in colour of each half of the treated test specimen with grey scale (*see* Notes 1 and 2 under **8.2.1**).

9 REPORT

9.1 Report the numerical ratings for change in colour for both the rinsed and the neutralized portions of the specimen.

ANNEX A

PREPARATION OF DYEING

A-1 Wet out well a sufficiently large sized woollen cloth and enter it at 40°C into a dye bath containing 1 percent of CI Mordant Red 3 Powder (Colour Index, 3rd edition), 10 percent sodium sulphate crystals ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) and 3 percent acetic acid (300 g/l), all percentages being calculated on the mass of the piece and using liquor ratio of 1 : 40.

A-2 Raise the dye bath to boil in 30 minutes and continue further for a period of 30 minutes.

Exhaust the dye bath, if necessary, by adding carefully 1 to 3 percent acetic acid (300 g/l) or 1 percent sulphuric acid (sp gr 1.84) well diluted with water. Boil the bath for a further period of 15 minutes after addition of acid. Cool the dye bath by addition of cold water and 0.5 percent potassium dichromate dissolved in water. Raise the dye bath to the boil and continue for 30 minutes.

A-3 Remove the piece, rinse it in cold running tap water and dry.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO MERCERIZING

(Source : IS 979 : 1988)

The method prescribed in this standard is based on ISO 105/X-1984 Textiles — Tests for colour fastness X04— Colour fastness to mer-

cerizing, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile fabrics, yarns or threads, of all kinds to the action of concentrated solutions of sodium hydroxide used in mercerizing.

1.2 The method is mainly applicable to cotton and mixtures containing cotton.

2 PRINCIPLE

2.1 A specimen of the textile, in contact with specified adjacent fabrics, is treated with sodium hydroxide solution, rinsed, acidified, again rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabric are assessed with grey scales.

2.2 As completely resistant specimens may show an apparent increase in depth of colour, these would not be rated 5 by the normal method of assessment. In such cases, therefore, only the changes in hue and brightness should be assessed using grey scale, without consideration of the increase in depth, and such assessments should be marked with an asterisk(*). The meaning of the asterisk has to be explained in a foot-note.

Examples

5* : Increase in depth (not considered); no change in hue and brightness.

3-4 redder* : Increase in depth (not considered); the hue became redder matching grey scale rating 3-4.

2 bluer, duller* : Increase in depth (not considered); the shade changed in hue and brightness according to grey scale rating 2.

2.3 Specimens which do not increase in depth shall be assessed in the normal manner and the results shall not be marked with an asterisk.

Example

2 weaker, bluer, duller : Loss in depth (considered) and change in both hue and brightness according to grey scale rating 2.

3 SAMPLING

3.1 Sample to determine conformity of a lot of coloured textile material to a specification shall

be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with the relevant material specification or as agreed to between the buyer and the seller to evaluate colour fastness of the material in the lot shall be representative of the lot.

4 COMPOSITE SPECIMEN

4.1 Cotton Adjacent Fabric

at least 10×10 cm for evaluating staining (see Annex A of IS 10251 : 1982).

4.2 Preparation of Composite Specimen

4.2.1 If the textile to be tested is fabric, sew a specimen of at least 10×10 cm to an equal sized piece of the cotton adjacent fabrics (see 4.1) around all four sides. Fasten this composite specimen to a frame firmly but without excessive tension.

4.2.2 If the textile to be tested is yarn or thread, wind an amount of it equal to the mass of adjacent fabric on a rigid frame firmly, but without excessive tension, with the strands close together and parallel to provide an area at least 10×10 cm. Sew an equal size piece of the adjacent fabric (see 4.1) to this area along the two sides across the strands.

5 APPARATUS

5.1 Frame

a metal frame for holding specimen. It consists of two folding wings which, in closed position can be locked by a wing nut. The two wings have fitting open squares of about 8×3 cm. All four sides of the frame are corrugated or contain needle bars in order to fix the composite specimen during the treatment. The rigid frame for yarns and threads should be a little larger than the corrugated frame or the needle bar and fitted into the metal frame.

5.2 Grey Scales

for evaluating change in colour and staining.

6 REAGENTS

6.0. Quality of Reagents

Unless otherwise specified, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTES

- 1 Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.
- 2 In the case of a dispute, chemicals of analytical reagent grade shall be used for the test.

6.1 Sodium Hydroxide (NaOH) Solution

300 g/l.

6.2 Sulphuric Acid Solution

containing 5 ml of concentrated sulphuric acid (sp gr 1.84) per litre.

6.3 Acetic Acid Solution

containing 10 ml of glacial acetic acid per litre.

7 PROCEDURE

7.1 Immerse the composite specimen with the coloured material uppermost in sodium hydroxide solution at $27 \pm 2^\circ\text{C}$ for 5 minutes so as to cover the specimen completely. Rinse the specimen first by pouring on it one litre of water at $70 \pm 2^\circ\text{C}$ over a period of one minute and then rinse in cold running tap water for 5 minutes. Remove it from the frame and immerse it in sulphuric acid solution or in acetic acid solution for five minutes at a liquor ratio of 1 : 50. Rinse it in cold running tap water until neutral. Remove the stitching along three sides (or along one side, in case the test piece is in the form of yarns or threads) and dry in

air in shade at a temperature not exceeding 60°C taking care that the adjacent fabric and the test specimen are kept apart except at the remaining line of stitching.

7.2 If the treated specimen shows increased depth of colour, evaluate its change in hue and/or brightness only, and the degree of staining of the two pieces of adjacent fabrics with the grey scales.

NOTES

- 1 Treated test specimen and the piece of adjacent fabric should have cooled after drying, and should have regained their normal moisture content before evaluation.
- 2 In cases of doubt in the assessment of colour fastness ratings as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 If the treated test piece does not show increased depth of colour, evaluate its change in colour as an overall contrast and the degree of staining of the piece of adjacent fabric (see Notes under 7.2) with grey scales.

8 REPORT

8.1 In the case of evaluation in accordance with 7.2, report the numerical rating for change in hue and/or brightness of the specimen and mark the rating with an asterisk, and the numerical rating for staining of the piece of adjacent fabric used in the preparation of the composite specimen.

8.2 In the case of evaluation in accordance with 7.3, report the numerical rating for change in colour of the specimen and the numerical rating for staining of the piece of adjacent fabric used in the preparation of the composite specimen.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO FORMALDEHYDE

(Source : IS 1807 : 1961)

Colour fastness of textile materials is of considerable importance to the consumer. The fastness depends not only upon the nature and depth of shade of dyestuff used but also upon the nature of fibre and the method of dyeing or printing employed; the same colouring matter, when used in dyeing or printing different fibres or when applied by different methods upon the same fibre, may give vastly different results.

This method of test is intended for evaluating colour fastness of textile materials to the effect of

contamination with formaldehyde, such as may originate from materials treated with a crease-resist finish, filling materials, pleating formers, etc.

The method prescribed is based on ISO 105/X-1984 Textiles — Tests for colour fastness X09 — Colour fastness to formaldehyde, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile materials to the effect of contamination with formaldehyde vapour as may be encountered in storehouses where fabrics have been stored with materials which have undergone a crease-resist treatment.

1.2 The method is not suitable for determining the change in colour which may occur during the anticrease finishing treatment with amino resins or during the after treatment of dyed materials with formaldehyde solutions.

2 PRINCIPLE

2.1 A piece of the material under test is exposed in a closed container to the action of gaseous formaldehyde. The change in colour of the piece is evaluated with grey scale.

3 SAMPLING

3.1 Sample shall be drawn so as to be representative of the lot.

3.2 Sample drawn in compliance with the procedure laid down in the material specification or as agreed between the buyer and the seller shall be representative of the lot.

4 TEST PIECE

4.1 If the material to be tested is fabric, draw a 10×4 cm test piece from the sample.

4.2 If the material to be tested is yarn, knit or weave some of the sample into fabric and draw from it a 10×4 cm test piece; alternatively, prepare a test piece in the form of a wick of parallel lengths of yarn 10 cm long and about 0.5 cm in diameter tied near both ends.

4.3 If the material to be tested is loose fibre, comb and compress enough of the sample into the form of a 10×4 cm sheet and sew it on a 10×4 cm piece of cotton cloth to form a test piece.

5 APPARATUS

5.1 Glass Bell

of 6-litre capacity.

5.2 Glass Frame

for suspending the test piece.

5.3 China Dish

of 50-ml capacity.

5.4 Grey Scale

for evaluating change in colour.

6 REAGENTS

6.1 Formaldehyde Solution

35 percent.

7 PROCEDURE

7.1 Fix the test piece to the glass frame so that it hangs freely over the china dish and does not come into direct contact with formaldehyde solution. Fill the china dish with 15 ml of the formaldehyde solution. Place the glass bell over the glass frame with the test piece and the china dish. Allow the test piece to remain in the formaldehyde-saturated atmosphere at a temperature of $27 \pm 2^\circ\text{C}$ for 24 hours. Remove the test piece and dry in an oven for one hour at a temperature of 100°C . Remove the test piece

from the oven and hang it in the air to cool for one hour.

7.2 Evaluate the change in colour of the treated piece with the help of grey scale and assign the ratings.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment

should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 The report shall include the numerical rating for change in colour.

DETERMINATION OF COLOUR FASTNESS OF DISPERSE DYES ON POLYESTER CELLULOSIC FABRICS TO CARBONIZATION

(Source : IS 11220 : 1984)

Carbonized fabrics are prepared from polyester-cellulosic blends. The blend fabric is dyed or printed and its cellulosic component is removed by treatment with concentrated sulphuric acid. This process is carried out in a textile mill itself with a view to impart the fabric special effects, such as fullness, very soft feel and bulkiness.

It is necessary that the disperse dyes used for dyeing or printing of polyester component should be fast to carbonization treatment. The method prescribed in this standard intends to help the textile mills to find out the colour fastness to carbonization treatment of the disperse dyes used for dyeing or printing.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of disperse dyes on polyester-cellulosic fabrics of 67 : 33 and 80 : 20 percent polyester : cellulose composition, to carbonization treatment using concentrated sulphuric acid [70 percent (*m/m*)].

2 PRINCIPLE

2.1 A test specimen of polyester-cellulosic fabric is dyed with the disperse dye under test, and carbonized with concentrated sulphuric acid [70 percent (*m/m*)], rinsed and dried. The change in colour of the test specimen is then compared with a test control specimen of 100 percent polyester fabric (obtained by pre-carbonization of the same polyester-cellulosic fabric and subsequently dyeing with the same dye) and assessed with the help of grey scale.

3 SAMPLING OF DISPERSE DYE

3.1 When the dye is available in powder form, draw from each container selected, small quantities of the dye by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

3.2 For the dyes available in the form of liquid, emulsion or paste, stir the contents of each container thoroughly and then draw small quantities of dye by a suitable sampling instrument from at least three different parts of each container and mix them thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

4 SAMPLING AND PREPARATION OF SPECIMENS OF FABRIC

4.1 A random sample shall be drawn from each lot so as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed

to between the buyer and the seller of the dyestuff shall be taken to be representative of the lot.

4.2 Prepare a specimen of 10 × 4 cm of fabric from each piece selected as in 4.1. Prepare at least six such specimens from each lot.

4.3 Scour all the specimens according to the procedure given in Annex A (*see* A-1). Mark five specimens as test specimens and the remaining one as test control specimen.

5 APPARATUS

5.1 A Laboratory Scale High Temperature Bomb/Beaker Dyeing Machine

5.2 Glass Beakers

six, of 500 ml capacity.

5.3 Grey Scale

for change in colour.

5.4 Test Tubes

6 REAGENTS

6.0 Quality of Reagents

Unless specified otherwise, pure reagents shall be employed in the tests and distilled water shall be used, wherever water is used as a reagent.

NOTE — Pure reagents shall mean reagents that do not contain impurities which affect the test results.

6.1 Sulphuric Acid Solution

reagent grade, 70 percent (*m/m*), specific gravity 1.629 at room temperature, and free from any nitrogenous impurities such as nitrous acid and nitric acid.

6.2 Sodium Carbonate Solution

containing 2 g/l of anhydrous sodium carbonate.

6.3 A Dispersing Agent

6.4 Acetic Acid

30 percent (*m/m*).

6.5 Sodium Hydrosulphite Powder

6.6 A Suitable Detergent

6.7 Urea

6.8 Diphenylamine

6.9 Polyethylene Glycol

6.10 Sodium Hydroxide Flakes

7 PROCEDURE

7.1 Immerse the test control specimen (*see 4.3*) into a beaker containing 100 ml of 70 percent (*m/m*) sulphuric acid solution per gram of the specimen at room temperature. Shake the beaker carefully to wet the specimen completely. Maintain the beaker at room temperature for 30 minutes with intermittent stirring and then take out the test control specimen.

7.2 Rinse the test control specimen as obtained in 7.1 for 5 minutes in cold running tap water and then dry it in air by hanging at a temperature not exceeding 60°C.

7.3 Carry out the operations as described in 7.4 to 7.6 with the test specimens (*see 4.3*) and the test control specimen as obtained in 7.2 in separate baths (*see Notes under 7.5*).

7.4 Dye the five test specimens (*see 4.3*) and one test control specimen of 100 percent polyester fabric as obtained in 7.2 under identical conditions according to the procedure given in Annex A (*see A-2*).

7.5 Carry out the operations on the five dyed test specimens (*see 7.4*) as given in 7.1 and 7.2

in five different beakers taking fresh solutions of 70 percent (*m/m*) sulphuric acid.

NOTES

1 It has been found that the colour of the fabric dyed with disperse dyes is destroyed, if exposed to an environment having nitrous acid fumes or chlorine gas. The deterioration effect is more pronounced if the nitrous acid fumes or chlorine gas are present in the sulphuric acid bath. Therefore, the fabric should not be exposed to this type of environment during carbonization.

2 The strength of the sulphuric acid should be checked during carbonization to see that it does not differ much.

3 Sulphuric acid may contain nitrogenous impurities, such as nitrous and nitric acids. These impurities are harmful and even traces cause considerable dulling of dyeings. In order to protect disperse dyes against the action of these unwarranted impurities, urea or sulphamic acid to the extent of 10-20 g/l may be incorporated in the carbonizing bath. The addition of these prevents the adverse effect on the dyeing and it also helps in subsidizing the effect of temperature rise during carbonization.

4 The presence of oxidizing impurities (such as nitrous, nitric and nitrosyl sulphuric acids) can be detected by the test given in Annex B.

7.6 Assess the effect of change in colour of the test specimens keeping test control specimen as standard and give all the test specimens appropriate grey scale rating.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 TEST REPORT

8.1 Report the lowest value of the numerical ratings for change in colour of the test specimens.

ANNEX A

PREPARATION OF DYEING OF DISPERSE DYES

A-1 SCOURING

A-1.1 Treat the five test specimens and one test control specimen, in parallel, in separate baths at 70-110°C for 15-30 minutes, each bath containing one part wetting agent, 1-2 parts sodium carbonate per 1000 parts by mass of water keeping liquor ratio of 1 : 40.

A-1.2 Rinse all the specimens well in warm water (40 to 50 C) containing one part of acetic acid (30 percent *m/v*) per 1000 parts of water by mass and then rinse all the specimens in plain water.

A-2 DYEING

A-2.1 Wet out all the test specimens and test control specimen in boiling water for 10 minutes and squeeze them evenly to about 100 percent pick up. Cool the specimens and enter into separate dye baths already prepared at 60°C, each dye bath containing:

a) Dispersing agent — 0.5-1 g/l

b) Disperse dye — *X* (*see Notes 2 and 3*)

and adjusted to pH 5.0 to 6.0 with acetic acid (30 percent *m/v*) keeping a liquor ratio of 1 : 40.

NOTES

1 Dye bath is prepared by first sprinkling the required amount of dyestuff over 15 to 20 times the quantity of distilled water at 40°C under constant stirring and then adding this to a blank bath containing dispersing agent and acetic acid adjusted to pH 5.0 to 6.0. The dyeing is carried out in high temperature bomb/beaker dyeing machine.

2 X is the amount of disperse dye in grams corresponding to the standard depth of the disperse dye under test or the percentage depth of shade as agreed to between the buyer and the seller.

3 While dyeing polyester-cellulosic fabrics (test specimens), the percentage depth shall be considered on the mass of polyester content of the fabric.

A-2.2 Start dyeing at 60°C and raise the temperature to 130°C at the rate of 2 to 3°C per minute in about 30 minutes. Continue dyeing at 130°C for 60 minutes more. Cool the machine to 85°C, take out the bombs/beakers and cool them further under running tap water before opening. Take out all the specimens.

A-2.3 Rinse all the dyed specimens in cold water and give the subsequent treatments as given in **A-2.4** and **A-2.5**.

A-2.4 Reduction Clearing

Treat all the specimens obtained in **A-2.3** in separate baths each containing 2 g/l sodium hydroxide flakes and 2 g/l sodium hydrosulphite powder at 70°C for 30 minutes keeping a liquor ratio of 1 : 40. Rinse all the specimens in cold water.

A-2.5 Soaping

Soap all the specimens obtained in **A-2.4** at 70-80°C for 20 minutes at a liquor ratio of 1 : 40 in separate baths, each containing 1 g/l of wetting agent. Wash the specimens in cold running water and dry them by hanging at a temperature not exceeding 60°C.

ANNEX B**TESTING PRESENCE OF OXIDIZING IMPURITIES IN SULPHURIC ACID**

B-1 Prepare 1 percent (m/v) diphenylamine solution in glycol.

B-1.1 Take 2 ml of sulphuric acid solution in a test tube and add to it 3 to 4 drops of 1 percent diphenylamine solution. A blue colour will indicate the presence of oxidizing impurities.

B-2 In other test tube, take 4-5 ml of the sulphuric acid solution. Add to it a few mg of urea and mix the two. Then add 2 to 3 drops of 1 percent diphenylamine solution. A faint yellowish green colour indicates that the impurities are suppressed/nullified by the urea added in the acid.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE FABRICS TO WET SCRUBBING

(Source : IS 11908 : 1986)

Pigment printing of textile fabrics is very common in India. The pigments are fixed on the fabric surface by the use of binder and there is almost neither any chemical linkage of the pigment particles with the fabric nor any penetration inside the fabric structure. Thus, due to the surface deposition of pigments by the binder

on the textile fabric, the deposited film may break when subjected to wet scrubbing action during home laundering. So it is necessary that the pigment printed fabric, in addition to other colour fastness requirements, should withstand the wet scrubbing action during home laundering.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of pigment printed textile fabrics of all kinds to the action of wet scrubbing during home laundering.

2 PRINCIPLE

2.1 A specimen of the pigment printed textile fabric is dipped in a hot soap solution containing sodium carbonate and scrubbed in wet condition with a nylon bristle brush. The specimen is then rinsed and dried. The change in colour of the specimen is assessed with grey scale for change in colour by comparing with that of original unscrubbed specimen.

3 SAMPLING

3.1 Test sample from the lot shall be drawn so as to be representative of the lot. Test sample drawn in accordance with the relevant material specification or as agreed to between the buyer and the seller shall be representative of the lot. The test sample so selected shall consist of a minimum of three pieces from the lot.

4 APPARATUS

4.1 A Suitable Brushing Device

4.1.1 It consists of a base board over which a small carriage is drawn. This carriage runs on parallel tracks attached to the edge of the upper surface of the base board. The brush is hinged with pin hinges at the rear edge of the base board and rests on the carriage vertically with a load of 150 g.

4.1.2 The brush consists of two rows of a 0.41 mm diameter stiff nylon bristle tufts with 20 bristles per tuft and 4 tufts per 25.0 mm. The two rows of tufts are staggered. The tufts are cut to a uniform length of 19 mm. A clamp is attached to the forward edge of the movable carriage to permit holding the specimen on the carriage during the brushing operation.

4.1.3 After the specimen has been put in place on the carriage and fastened by means of the clamps, the brush is raised, the carriage pushed to the rear and the brush lowered to the face of the specimen. The carriage is then drawn forward by hand at uniform rate, similar to crockmeter.

4.2 Grey Scale

for assessing change in colour.

4.3 A Beaker of 500 ml Capacity

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE—Pure chemicals shall mean the chemicals that do not contain impurities which affect the test results.

5.1 Soap Solution

The soap solution shall contain 5 g/l soap and 2 g/l sodium carbonate. The soap shall not have moisture regain more than 5 percent and shall be free from optical brightening agents.

5.1.1 The soap meeting the following requirements (based on dry mass) is satisfactory:

Characteristic	Requirement
Free alkali calculated as Na_2CO_3 (Max), percent	0.3
Free alkali calculated as NaOH (Max), percent	0.1
Combined fatty acids calculated as Na salts (Min), percent	85
Titre of mixed fatty acid prepared from the soap (Max)	39°C
Iodine value of fatty acids (Max)	50

6 PREPARATION OF TEST SPECIMENS

6.1 From the test sample (3.1), draw at least three test specimens of 250 × 75 mm size taking

care that no two specimens are cut from the same piece.

7 PROCEDURE

7.1 Take a test specimen (6.1) and dip it for 5 to 10 minutes in the soap solution (5.1) taken in a beaker and previously heated to 55 to 60°C, at liquor ratio of 1 : 50.

7.2 Take out the specimen from the beaker and remove excess soap solution just by hanging in air for 5 minutes. The specimen thus obtained should carry sufficient soap solution so that scrubbing is proper.

7.3 Mount the specimen on the carriage of the brushing device with printed side up. While mounting the specimen, keep it flat and smooth without wrinkles or creases and do not stretch or distort it. Fasten the specimen by means of clamp.

7.4 Raise the brush, push the carriage to the rear and lower the nylon bristle brush to rest on the specimen. Draw the carriage under the brush by hand at uniform rate fifty times from each end, that is, a total of hundred strokes.

NOTE — When the brush is lowered to rest on the mounted specimen, it should be positioned approximately 3.0 mm in from the raw cut edge of the specimen so that the specimen will not be distorted

during the brushing operation. The cloth should be maintained properly wet during wet scrubbing.

7.5 Take out the specimen from the brushing device and rinse it in running tap water till all the soap is removed. Then dry the specimen in air without exposing in sunlight.

7.6 Evaluate the change in colour of the treated specimen with the help of grey scale and assign the rating.

NOTES

1 Treated test specimen should be allowed to cool after drying to regain their normal moisture content before evaluation.

2 In cases of doubt in the colour fastness rating as assessed by an observer, the assessment may be made by three observers and the overall average rating may be reported.

7.7 Repeat the test procedure from 7.1 to 7.6 with the remaining test specimens.

8 REPORT

8.1 The test report shall include the following information:

- a) Type and quality of fabric, and
- b) Numerical ratings for change in colour for each individual test specimen.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE FLOOR COVERINGS TO SHAMPOOING

(Source : IS 11969 : 1986)

Textile floor coverings form a large part of export of textiles. Colour fastness to shampooing is an important parameter to check the quality of the product for everyday use.

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textile floor coverings; yarns and loose fibres intended for manufacture of textile floor coverings; and the tufts extracted from textile floor coverings to the action of a standard shampoo solution.

2 PRINCIPLE

2.1 A specimen of the textile floor covering, yarn, loose fibre or tufts; in contact with specified adjacent fabrics, is immersed under pressure in a solution of shampoo buffered to pH 7.5 ± 0.2 . The specimen and adjacent fabrics are dried separately. The change in colour of the specimen and the staining of the adjacent fabrics are assessed with grey scales.

3 SAMPLING

3.1 The sample of textile floor covering, yarn, fibre or tufts shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be held to be representative of the lot.

4 APPARATUS AND REAGENTS

4.1 AATCC Perspirometer

NOTE — This instrument has been developed by the American Association of Textile Chemists and Colorists. Any other equivalent apparatus giving identical results may also be used.

4.2 Flat-Bottomed Dish

about 100×150 mm and 50 mm deep.

4.3 Smooth Glass Plate or Clear Acrylic Resin Plate

about 113×60 mm and 3 mm thick.

4.4 Weight-Piece

of mass about 5 kg.

4.5 pH Meter

equipped with glass calomel electrodes.

4.6 Shampoo Solution

prepared by dissolving 1 g sodium lauryl sulphate (active ingredient) and 0.2 g lauric mono-iso-

propanolamide in about 500 ml of de-ionized or distilled water. Heat, if necessary, to ensure the dissolution of the surfactant. Cool to about 30°C . Prepare buffer solution by adding 14 ml of 0.5 M citric acid to 372 ml of 0.5 M disodium hydrogen phosphate. Add the buffer solution to the surfactant solution. Check the pH to 7.5 ± 0.2 . Finally make the total volume of shampoo solution up to one litre.

4.7 Adjacent Fabrics

4.7.1 When the specimen is in the form a textile floor covering, two adjacent fabrics 50×40 mm are required, one piece made of the same fibre as that in the specimen, or that predominating in the case of blends; the second piece made of any other fibre (see Annex A). If the staining of additional fibres is of interest, two or more specimens shall be tested separately.

4.7.2 When the specimen is in the form of yarn or loose fibre, two adjacent fabrics 100×40 mm are required, one piece made of the same fibre as that in the specimen or that predominating in the case of blends, the second piece made of any other fibre (see Annex A). If the staining of additional fibres is of interest, two or more specimens shall be tested separately.

4.7.3 When the specimen is in the form of tufts extracted from a textile floor covering, two adjacent fabrics 50×40 mm are required, one piece made of cotton, to act solely as a support for the tufts, the second piece made of the same fibre as that in the specimen or that predominating in the case of blends, or any other fibre (see Annex A). If the staining of two or more fibres is of interest, two or more specimens shall be tested.

4.8 Grey Scales

for evaluating change in colour and staining of adjacent fabrics.

5 PREPARATION OF COMPOSITE TEST SPECIMENS

5.1 If a textile floor covering is to be tested, cut a specimen 100×40 mm, from which any integral form underlay has been removed, and cover the use surface with the two pieces of adjacent fabrics 50×40 mm (see 4.7.1).

5.2 If yarn is to be tested, sew a uniform layer of parallel 100 mm length, weighing 0.4 g, to one

edge of one of the pieces of adjacent fabrics 100×40 mm (see 4.7.2). Cover with the other piece 100×40 mm and sew along the same edge to form a composite specimen.

5.3 If loose fibre is to be tested, comb and compress 0.4 g of the fibre into a uniform layer 100×40 mm. Place this between the two pieces of adjacent fabrics 100×40 mm (see 4.7.2) and sew along two opposite sides to form a composite specimen.

5.4 If tufts extracted from textile floor coverings are to be tested, sew a number of identical tufts weighing approximately 0.2 g on to a piece of cotton adjacent fabric 50×40 mm. Cover with a piece of adjacent fabric 50×40 mm (see 4.7.3) and sew along one side to form a composite specimen.

6 PROCEDURE

6.1 Place the composite specimen in the flat-bottomed dish (see 4.2) and pour over the specimen a quantity of the shampoo solution (see 4.6) at $27 \pm 2^\circ\text{C}$ sufficient to give a liquor ratio of 1 : 50. Ensure that the specimen is thoroughly wetted by the solution and lies flat. Cover with the glass plate (see 4.3), press with the fingers to remove air bubbles, and place the weight-piece (see 4.4) on top of the glass plate. A composite specimen of tufts should be placed in the dish with the cotton on the underside.

NOTE — Care should be taken so that the material of the weight does not react with the solution during testing.

6.2 Allow it to stand at $27 \pm 2^\circ\text{C}$ for 15 minutes in the perspirometer. Remove the weight-piece and pour off the shampoo solution without removing the glass plate, replace the weight-piece and allow it to stand for a further period of three hours at $27 \pm 2^\circ\text{C}$.

6.3 Remove the weight-piece and glass plate. Separate the specimen from the adjacent fabrics, and allow the specimen and the adjacent fabrics to dry apart in air at a temperature not exceeding 60°C .

6.4 Assess the change in colour of the specimen and the staining of the adjacent fabrics with the grey scales, and assign the ratings. When tufts are being tested, the staining of the cotton support need not be assessed.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be made by three observers and the overall average rating should be reported accordingly.

7 REPORT

7.1 Report shall include the following information:

- Type of material,
- Numerical rating for change in colour of the test specimen, and
- Numerical rating for staining of each kind of adjacent fabric used.

ANNEX A

CONSTRUCTIONAL DETAILS OF ADJACENT FABRICS

Adjacent Fabric	Mass in g/m ²	Type of Weave	Ends/cm	Picks/cm	Tex of Yarn	
					Warp	Weft
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Cotton	115 ± 5	1/1 Plain	35	31	16.5	14.0
Viscose	140 ± 5	1/1 Plain	28	22	20.0	33.0
Wool	125 ± 5 — 0	1/1 Plain	21 ± 0.5	18 ± 0.5	15.6×2	15.6×2
Polyamide	130 ± 5	1/1 Plain	17.5	20	Worsted	Worsted
Polyester	130 ± 5	1/1 Plain	23.5	20.5	10×2	20
Acrylic	135 ± 5	1/1 Plain	17.5	16	7.5×2	10×2

NOTES

1 For wool adjacent fabric, additional requirements are: (a) pH value of aqueous extract 6.5 to 7.5, (b) residual fat content 0.4 ± 0.1 percent, and (c) alkali solubility less than 18 percent.

2 For polyamide and acrylic adjacent fabrics, additional requirements are: (a) pH value of aqueous extract 7 ± 0.5 , and (b) residual oil content less than 1.0 percent.

3 For polyester adjacent fabric, additional requirement is residual oil content less than 0.5 percent.

4 The adjacent fabrics shall be bleached and free from any sizing or finishing material and optical brightening agents.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO VULCANIZING WITH HOT AIR

[Source : IS 4635 (Part 1) : 1968]

In proofed textile materials, the coloured cloth comes in contact with rubber compounds. During vulcanizing, due to the action of the rubber compounds, the colour of the dyed materials is likely to change. The colour fastness of such textile materials to vulcanizing is, therefore, important.

The method prescribed is based on ISO 105/S-1978 Textiles — Tests for colour fastness S01 — Colour fastness to vulcanizing : Hot air, issued by the International Organization for Standardization (ISO)

1 SCOPE

1.1 This method is intended for determining the resistance of colour of textiles of all kinds and in all forms to the action of a typical rubber compound such as may be used in the proofing industry and its decomposition produced during vulcanization in hot air.

2 PRINCIPLE

2.1 A specimen of textile is heated in air in direct contact with an (initially) unvulcanized rubber compound. The change in colour of the specimen is assessed with Grey Scale.

3 SAMPLING

3.1 The sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 APPARATUS

4.1 An oven maintained at $125 \pm 2^\circ\text{C}$ with a fan to ensure uniformity of air temperature.

4.2 Grey Scale

For evaluating change in colour.

5 REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.2 A sheet of rubber compound 2.5 ± 1.5 mm thick consisting of :

- a) 100 parts pale crepe,
- b) 5 parts zinc oxide,
- c) 1 part stearic acid,

- d) 2 parts sulphur,
- e) 1 part mercaptobenzthiazole,
- f) 0.2 part zincdiethyldithiocarbamate,
- g) 15 parts titanium dioxide, and
- h) 75 parts barium sulphate.

NOTE — If it is necessary to transport the rubber compound, it should be covered with thin polyethylene sheeting.

6 PREPARATION OF COMPOSITE SPECIMEN

6.1 Remove any polyethylene film from the sheet of uncured rubber compound (see 5.2) and moisten the compound with petroleum spirit.

6.2 If the textile to be tested is fabric, cut a piece of 10×4 cm from the sample and place it on the sheet of uncured rubber compound. To ensure uniform degree of adhesion, roll the specimen on the rubber compound with a metal roller. Similarly prepare two more composite specimens.

6.3 If the textile to be tested is yarn, take sufficient amount of yarn and knit it into a fabric and cut a 10×4 cm test piece. Alternatively stick a number sheet to obtain the specified area of 10×4 cm. Similarly, prepare two more composite specimens.

6.4 If the textile is loose fibre, comb and compress enough sample to form a sheet of 10×4 cm and stick it to the rubber compound. Similarly prepare two more composite specimens.

7 PROCEDURE

7.1 Hang one composite specimen in the oven for 30 minutes at $125 \pm 2^\circ\text{C}$. Take out the composite specimen and cool it in air. Separate the test piece from the rubber.

7.2 Evaluate the change in colour of the specimen on the side showing the greater change with the help of grey scale and assign the ratings.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer the assessment should be done by at least three observers and the overall average rating should be reported.

7.3 Repeat the test with the remaining test pieces.

8 REPORT

8.1 The report shall include the numerical rating for the change in colour for each test piece.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO VULCANIZING WITH SULPHUR MONOCHLORIDE

[Source : IS 4635 (Part 2) : 1968]

In proofed textile materials, the coloured cloth is exposed to action of sulphur monochloride under the conditions occurring during cold vulcanizing of rubber. The colour fastness of such textiles to vulcanizing is, therefore, important. This method would be useful for determining the

colour fastness of textiles to vulcanizing with sulphur monochloride. The method prescribed is based on ISO 105/S-1978 Textiles — Tests for colour fastness S02 — Colour fastness to vulcanizing: Sulphur monochloride, issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for determining the resistance of colour of textiles of all kinds and in all forms to the action of sulphur monochloride under the conditions occurring during the cold vulcanizing of rubber.

2 PRINCIPLE

2.1 A specimen of textile is exposed to sulphur monochloride vapours. The change in the colour of the specimen is assessed with grey scale, before and after neutralizing with ammonia.

3 SAMPLING

3.1 The sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 PREPARATION OF TEST PIECES

4.1 If the sample to be tested is fabric, cut out a test piece of 10×4 cm from each piece in the test sample.

4.2 If the sample to be tested is yarn, knit or weave a fabric and cut out a test specimen measuring 10×4 cm or alternatively, wind it round a piece of thin inert material 14×4 cm to obtain an area 10×4 cm of textiles for test. Similarly prepare two more test pieces from the test sample.

4.3 If the sample to be tested is loose fibre, comb and compress enough of it to form a sheet of 10×4 cm and sew it on a piece of undyed bleached cotton cloth to support the fibres. Similarly prepare two more test pieces from the test sample.

5 APPARATUS

5.1 Heated Exposure Chamber

In which a provision is made to hang the test pieces (see Fig. 1).

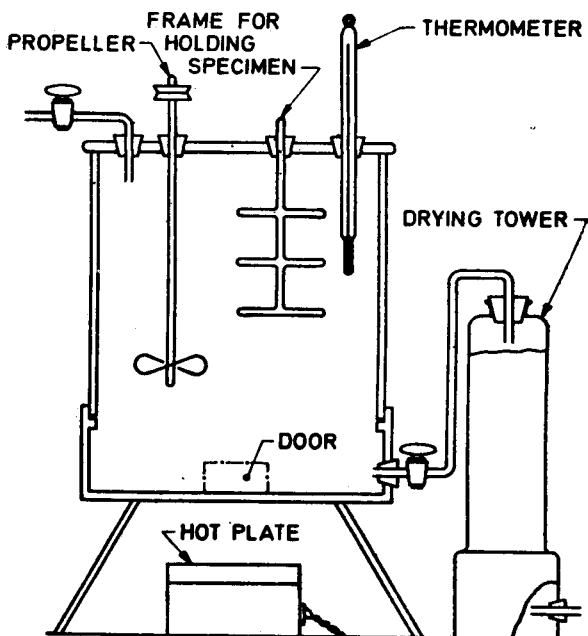


FIG. 1 EXPOSURE CHAMBER

NOTE — Provision is also made so that the dry air is aspirated through the apparatus, space being provided to put a beaker containing sulphur monochloride or ammonia inside the chamber.

5.2 Grey Scale

for evaluating change in colour.

6 REAGENTS

6.1 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6.2 Sulphur Monochloride (S_2Cl_2)

Sulphur monochloride should be yellow and not

brown, as brown colouration indicates decomposition.

6.3 Ammonia Solution

sp gr 0.88 containing about 300 g/l of ammonia.

7 PROCEDURE

7.1 Suspend the test pieces in the exposure chamber. Heat the air in the chamber to $50 \pm 4^{\circ}\text{C}$ and aspirate dry air to the chamber for 15 minutes. Close the tap leading from the drying tower and disconnect the lead to the vacuum line.

7.2 Place a beaker containing 0.01 g sulphur monochloride (*see* 6.2) for each litre of exposure chamber capacity inside the chamber and maintain a temperature at $50 \pm 2^{\circ}\text{C}$ for 1 hour. Switch off the heater and aspirate dry air through the chamber for 2 hours.

7.3 Remove the test pieces and cut them into 2 pieces across the width. Place one portion of each test piece in the exposure chamber. Place a beaker containing 0.05 g of ammonia solution for each litre of exposure chamber capacity inside the chamber and leave at room temperature for 1 hour.

7.4 Evaluate the change in colour of the test pieces immediately before and after neutralizing with ammonia with the help of grey scale and assign the ratings.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

8 REPORT

8.1 Report individually the numerical rating for the change in colour for each test piece before and after neutralizing with ammonia.

DETERMINATION OF COLOUR FASTNESS OF TEXTILE MATERIALS TO VULCANIZING WITH OPEN STEAM

[Source : IS 4635 (Part 3) : 1968]

The method prescribed is based on ISO 105/S-1978 Textiles — Tests for colour fastness S03 — Colour fastness to vulcanizing: Open steam,

issued by the International Organization for Standardization (ISO).

1 SCOPE

1.1 This method is intended for the determination of colour fastness of textiles of all kinds and in all forms to the action of a typical rubber compound, such as may be used in the proofing industry and its decomposition products during vulcanization in open steam either:

- a) under conditions that prevent live steam coming in contact with the textiles to be tested, or
- b) under conditions that allow live steam to infiltrate into textiles to be tested.

2 PRINCIPLE

2.1 A specimen of the textile is heated in a live steam in direct contact with an unvulcanized rubber compound. The textile material should be either:

- a) wrapped in sheeting impermeable to steam or water, so that the live steam or water is prevented from infiltrating into the specimen;

or

- b) wrapped in undyed bleached cotton cloth, so that the live steam is not prevented from infiltrating into the specimen.

2.2 The change in colour of the specimen and the staining of the adjacent fabric are assessed with grey scales.

3 SAMPLING

3.1 The sample shall be so drawn as to be representative of the lot. Sample drawn in accordance with the procedure laid down in the material specification or as agreed to between the buyer and the seller shall be taken as representative of the lot.

4 PREPARATION OF COMPOSITE SPECIMEN

4.1 Adjacent Fabric

4.1.1 Adjacent fabric of the same generic kind of fibre as that in the textile to be tested or that predominating in the case of blends (see Annex A of IS 10251 : 1982).

4.1.2 Undyed Bleached Cotton Cloth

4.1.3 A Sheet Impermeable to Steam and Water

NOTE — A rubber proofed fabric or plastic sheet capable of withstanding a temperature of 140°C. such as polyester.

4.2 Remove any polyethylene film from the sheet of uncured rubber compound and moisten it with petroleum spirit.

4.3 If the textile to be tested is fabric, from the sample cut a piece of 10 × 4 cm and place it on the sheet of uncured rubber compound. To ensure a uniform degree of adhesion, roll the specimen on the rubber compound with a metal roller.

4.4 If the textile to be tested is yarn, take sufficient amount of yarn and knit it into a fabric. Cut a piece of 10 × 4 cm or stick a number of lengths of yarn flat on the rubber sheet to obtain the specified area of 10 × 4 cm.

4.5 If the textile is loose fibre, comb and compress enough of sample to form a sheet of 10 × 4 cm and stick it to the rubber compound.

5 APPARATUS

5.1 Jacketed Autoclave

capable of holding steam pressure on both jacket and pan of 390 kPa.

5.2 Stainless Steel Open-Ended Tube

of diameter 40 ± 3 mm and wall thickness 1.5 ± 0.5 mm.

5.3 Grey Scales

for evaluating change in colour and staining.

6 REAGENTS

6.1 Unless specified otherwise, pure chemicals shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

6.2 A Sheet of Rubber Compound

2.5 ± 1.5 mm thick consisting of:

- a) 100 parts pale crepe,
- b) 5 parts zinc oxide,
- c) 1 part stearic acid,

- d) 2 parts sulphur,
- e) 1 part mercaptobenzthiazole,
- f) 0.2 part zinc diethyldithiocarbamate,
- g) 15 parts titanium dioxide, and
- h) 75 parts barium sulphate.

NOTE — If it is necessary to transport the rubber compound, it should be covered with a thin polyethylene film.

7 PROCEDURE

7.1 Wrap the composite specimen around stainless steel tube with rubber face inside.

7.2 To Prevent Ingress of Steam

Cover the composite specimen by tightly wrapping with one layer of adjacent fabric (see 4.1.1), three layers of undyed bleached cotton cloth (see 4.1.2) and two layers of impermeable sheeting (see 4.1.3) taking care that the wrapping projects beyond both the edges of composite specimen on the tube. Securely bind the edges with string to prevent ingress of steam.

7.3 To Permit Ingress of Steam

Cover the composite specimen by tightly wrapping with one layer of adjacent fabric (see 4.1.1) and two layers of undyed bleached cotton cloth (see 4.1.2).

7.4 Place the tube in the preheated autoclave ensuring that the temperature of the jacket is at $142 \pm 1^\circ\text{C}$ (350 kPa steam pressure) and the pan at $139 \pm 1^\circ\text{C}$ (350 kPa steam pressure). Carry out the test for 20 minutes.

7.5 Remove the tube and cool in air. Remove the wrapping cloth and adjacent fabric, and condition for 4 h in the standard atmosphere for testing.

7.6 Evaluate the change in colour of the test piece on the side not in contact with rubber and the staining of the adjacent fabric on the side in direct contact with the test piece with help of grey scale and assign the ratings.

NOTE — In cases of doubt in the colour fastness rating as assessed by an observer, the assessment should be done by at least three observers and the overall average rating should be reported.

7.7 Repeat the procedure with the remaining test pieces.

8 REPORT

8.1 The report shall include the following information:

- a) Numerical ratings for the change in colour,
- b) Numerical ratings for the staining of the adjacent fabric, and
- c) Method of wrapping followed (see 7.2 or 7.3).

