

Thermal Fixation Properties of Disperse Dyes

Developed in 1978 by AATCC Committee RA87; reaffirmed 1981; editorially revised and reaffirmed 1986, 1991, 1996, 2001.

1. Purpose and Scope

1.1 This test method is used to determine the fixation properties of disperse dyes on polyester/cellulose blends as a function of fixation conditions. The variable described herein is temperature; however, variations in time and/or concentration of dyes and/or auxiliaries may be studied by this method.

2. Principle

2.1 The dye is padded onto the blend fabric at a specified concentration; the fabric is dried and the dye is fixed by exposure to controlled conditions of time and temperature. The cotton portion of the blend is dissolved in concentrated sulfuric acid followed by neutralization and thorough rinsing.

2.2 The reflectance values of the dyed fabrics, exposed to the several fixation conditions, are determined by spectrophotometry and the concentrations of dye relative to the heaviest dyeing (as 100%) are evaluated by applying the Kubelka-Munk function (K/S). These results express the relative dye fixation for a particular set of dyeing conditions. Alternatively, the dye on the fabric can be extracted with a suitable solvent and the concentrations of dye determined by transmittance spectrophotometry. When the dye concentrations on the fixed samples are compared to the corresponding concentration on a padded but *unfixed* sample, a measure of the true dye fixation, as a function of applied dye, may be obtained.

3. Terminology

3.1 **disperse dye**, n.—an essentially water insoluble dye having affinity, when properly dispersed, for polyester, polyamide and some other manufactured polymeric fibers.

3.2 **thermal fixation**, n.—the use of dry heat to achieve a degree of permanence when applying colorants to textile materials.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedure

and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 Perform leaching procedure (see 7.3) using sulfuric acid, 70%, in a hood. In preparing, dispensing and handling sulfuric acid, use chemical goggles or face shield, impervious gloves and an impervious apron. Concentrated acids should be handled only in an adequately ventilated hood. CAUTION: Always add acid to water.

4.4 In preparing, dispensing and handling ammonium hydroxide, use chemical goggles or face shield, impervious gloves and an impervious apron. Dispense, mix and handle ammonium hydroxide only in an adequately ventilated hood.

4.5 Perform the extraction procedure (see 8.2) using chlorobenzene inside an adequately ventilated hood. CAUTION: Chlorobenzene vapors are toxic and flammable.

4.6 Chlorobenzene is a flammable liquid and should be stored in the laboratory only in small containers away from heat, open flame and sparks.

4.7 An eyewash/safety shower should be located nearby and a self-contained breathing apparatus should be readily available for emergency use.

4.8 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000 of January 1, 1989). In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 11.1).

5. Uses and Limitations

5.1 Evaluations by reflectance mea-

surements are limited to relatively low dye concentrations because of the non-linearity of the K/S function at high dye concentrations (see 11.2).

5.2 Fixation values obtained by reflectance measurements can differ from those determined by extraction techniques because of differences in dye distribution within the fiber (see 11.2).

5.3 Every effort should be made during padding, drying and fixation to avoid two-sided dyeings. Should this occur to any significant extent, the preferred method of measuring fixation is extraction.

6. Apparatus and Materials

6.1 Laboratory padder.

6.2 Forced air thermofixation unit (see 11.1).

6.3 Analytical balance.

6.4 Spectrophotometer (see 11.3).

6.5 Volumetric pipettes and flasks.

6.6 65/35 polyester/cotton fabric; e.g., Testfabrics Inc.'s Style 7409 or equivalent fabric adequately pre-scoured and ready for dyeing (see 11.4).

6.7 Sulfuric acid, 70%.

6.8 Chlorobenzene.

6.9 Ammonium hydroxide, 5%.

6.10 Acetic acid, 56%.

7. Procedure

7.1 Pad polyester/cotton blend cloth at 50-60% wet pickup with:

10 g/L—Disperse dye

20 g/L—Antimigrant (see 11.5)

Acetic acid (56%) to pH 5.5-6.0

Use the same pad liquor for all the dyeings in the set.

7.2 Dry each sample thoroughly under minimum migration conditions (see 11.5), then thermofix for 90 s at 196°C (385°F), 205°C (400°F), 213°C (415°F) and 221°C (430°F), respectively, in a properly calibrated oven (see 11.6).

7.3 Leach out the cotton portion of the blend with 70% sulfuric acid for 3-4 min at 55°C (130°F). Rinse the sample in cold running water and neutralize with 5% ammonium hydroxide solution for 1 min followed by another cold water rinse.

7.4 In cases where a change in shade of a disperse dye is suspected due to leaching, the following procedure may be employed: Attach a 100% polyester rider to the blended cloth to be dyed by the method in 7.1. Leach a portion of the rider separately by the method described in 7.3 and evaluate for any changes that may occur in the leaching process.

8. Evaluation

8.1 Make reflectance measurements in the visible portion of the spectrum (see 11.2) to determine relative strengths on the leached polyester using the deepest dyeing fixed at 221°C (430°F) as 100%. K/S values of the minimum reflectance value are obtained using the Kubelka-Munk equation:

$$K/S = (1 - R)^2/2R$$

where:

K/S = absorption function

R = minimum reflectance value (see 11.2)

Calculate the relative fixation values in comparison to the deepest dyeing using the following equation:

$$C_i = 100 \times \frac{(K/S)_i}{(K/S)_{max}} \quad (\text{see 11.2})$$

where:

C_i = percent of dye fixed on sample i

$(K/S)_i$ = absorption function of dyeing i

$(K/S)_{max}$ = absorption function of the deepest dyeing

8.2 As an alternate method of evaluation, an accurately weighed sample (250 mg) may be individually extracted in several small portions of chlorobenzene at the boil (132°C or 270°F). When the dye is completely removed from the substrate, transfer the extract quantitatively to a volumetric flask and dilute with additional chlorobenzene (see 11.7). Transmittance measurements made at the point of minimum transmittance or maximum absorption in the visible area of the spectrum of the extracts of fixed samples are compared to the corresponding measurements of extracts of the padded, unfixed sample (as 100%). Transmittance values

are converted to absorbance values from tables (see 11.8) or as follows:

$$A = \log_{10} (1/T)$$

where:

A = the absorbance value

T = the transmittance value

Fixation values are calculated by the following equation:

$$C_i = 100 \times \frac{A_i}{A_u} \quad (\text{see 11.9})$$

where:

C_i = percent of dye fixed on sample i

A_i = absorbance of the extract of dyeing i

A_u = absorbance of the extract of the unfixed dyeing

9. Report

9.1 Fixation data obtained by the above methods may be presented in graphic form or in tabular form expressed as the percent of fixation versus temperature. The effects of the time and/or dye concentration may be shown as a family of curves depending upon the objectives of the particular study under consideration.

10. Precision and Bias

10.1 *Precision.* Precision for this test method has not been established. Until a precision statement is generated, use standard statistical techniques in making any comparisons of test results for either *within-laboratory* or *between-laboratory* averages.

10.2 *Bias.* The thermal fixation property of a disperse dye can be defined only in terms of a test method. There is no independent method for determining the true value. As a means of estimating this property, the method has no known bias.

11. Notes

11.1 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: 513/742-2020.

11.2 Refer to AATCC Evaluation Procedure 6, Instrumental Color Measurement, paragraph 4.3, Color Strength Value Using Reflectance Measurement, and 4.5, Relative Strength, available from AATCC Technical Center, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.

11.3 A continuous or abridged reflectance spectrophotometer is suitable for measurements at the wavelength of maximum absorption (minimum reflectance).

11.4 Available from Testfabrics Inc., P.O. Box 26, 415 Delaware St., W. Pittston PA 18643; tel: 570/603-0432; fax: 570/603-0433; e-mail: testfabric@aol.com.

11.5 A number of suitable gums for this purpose are available; e.g., natural gums, alginates and synthetic acrylic acid polymers. The use of electrolytes in the pad liquor is to be avoided since the resulting agglomeration is very difficult to control.

11.6 Several fixation units are available for laboratory studies. In all cases the equipment used should be carefully calibrated with respect to temperature, residence time and uniformity of air flow, and should be named in the written report.

11.7 Some disperse dyes may be partially decomposed during the extraction and are not suitable for this procedure.

11.8 Refer to AATCC Evaluation Procedure 6, Instrumental Color Measurement, paragraph 4.4, Color Strength Value Using Transmittance Measurement, and 4.5, Relative Strength, available from AATCC Technical Center, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.

11.9 As a general guide for obtaining transmittance measurements, refer to A General Procedure for the Determination of Relative Dye Strength by Spectrophotometric Transmittance Measurements, *Textile Chemist and Colorist*, Vol., 4, No. 5, p43, May 1972.