

Test Method for Color Change of Disperse Dyes Caused by Metals, with and without Chelating Agent

Developed in 1983 by AATCC Committee RR90; editorially revised 1985, 1988 (with title change), 2004, 2008, 2010, 2019; editorially revised and reaffirmed 1986, 1987, 1992, 2002, 2012; reaffirmed 2007; revised 2018.

1. Purpose and Scope

1.1 This test method provides a laboratory procedure for:

1.1.1 Determining the effectiveness of chelating agents in disperse dye baths for inactivating heavy metals that can cause color change.

1.1.2 Evaluating the sensitivity of disperse dyes to metal-induced color change during dyeing.

2. Principle

2.1 The effectiveness of chelating agents in sequestering metals in a dye bath, thereby preventing metal-induced color change, is determined by the protocol in Fig. 1.

2.2 Metal sensitivity of disperse dyes is determined by comparing control dyes and test dyes by the protocol shown in Fig. 2.

3. Terminology

3.1 **chelating agent**, n.—in *textile chemistry*, a chemical capable of deactivating metal ions by formation of a water-soluble complex. SYN: *sequestering agent*.

3.2 **color change**, n.—as used in *colorfastness testing*, a change in color of any kind whether in lightness, hue or chroma, or any combination of these, discernible by comparing the test specimen with a corresponding untested specimen.

3.3 **grade**, n.—the number assigned to a test specimen resulting from comparison to a scale.

3.4 **Gray Scale**, n.—a scale consisting of pairs of standard gray chips, the pairs representing progressive differences in color or contrast corresponding to numerical colorfastness grades.

NOTE: Gray Scale grades are expressed as half-steps in decimal form (e.g., 1 or 1.5). Dashed form is also acceptable (e.g., 1 or 1-2).

3.5 **metal sensitivity**, n.—the propensity of a dye to produce an abnormal color on a textile material when in the presence of certain metal ions.

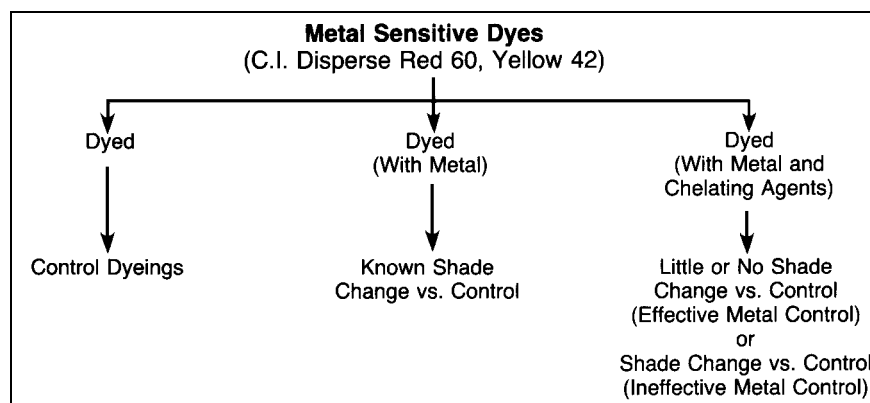


Fig. 1

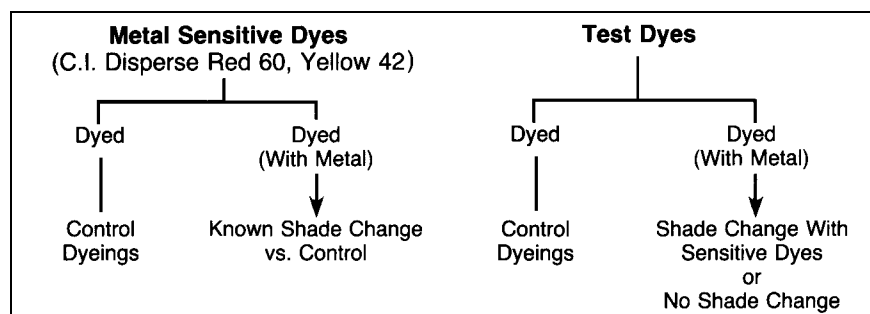


Fig. 2

4. Safety Precautions

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

4.2 Pressurized dyeing equipment is required for some dyeing. Trained personnel should exercise care and follow the equipment manufacturer's instructions for use and safety precautions.

4.3 All chemicals should be handled with care.

4.4 In preparing, dispensing and han-

dling acetic acid and phosphoric acid, use chemical goggles or face shield, impervious gloves and an impervious apron. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

4.5 Carriers vary in toxicity depending on type. Read vendors technical data, SDS and/or product label for contents and/or OSHA hazard classification.

4.6 An eyewash/safety shower should be located nearby and a high efficiency particulate respirator with a full facepiece should be readily available for emergency use.

4.7 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; see www.osha.gov for latest version]. In addition, the American Conference of Governmental Industrial Hy-

gienists (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 14.1).

5. Uses and Limitations

5.1 Accurate results and expected shade changes depend on the metal dye and carrier used, as well as close control of all dyeing parameters.

5.2 Extraneous metal contamination can give misleading results. Use clean fabrics, distilled water, and rinse the dye containers with a 10% phosphoric acid solution followed by a distilled water rinse.

5.3 To avoid erroneous conclusions, test conditions must be established with dyes of known metal sensitivity to verify that the expected shade change can be obtained. Some samples of control dyes or carriers, or certain dyeing conditions, may not produce the expected shade change.

5.4 This test method is not intended for use with disperse dyes in combination with other dye classes that may contain metal complexes potentially damaged by chelating agents.

6. Apparatus and Materials (see 14.2)

6.1 Test fabric, clean 100% polyester fabric (filament or staple), desized (as needed) and prescoured (see 14.3).

6.2 Control dyes, C. I. Disperse Red 60 and C. I. Disperse Yellow 42 (see 14.4).

6.3 Test dyes.

6.4 Cupric sulfate (CuSO_4), MW 159.606.

6.5 Ferrous ammonium sulfate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$), MW 392.158.

6.6 Carrier (see 14.5).

6.7 Chelating agent (see 14.6).

6.8 Distilled water.

6.9 Acetic acid (CH_3COOH), 99.7%.

6.10 Phosphoric acid (H_3PO_4), 85%.

6.11 Dyeing apparatus, capable of providing good agitation and uniform temperature control.

6.12 AATCC Gray Scale for Color Change (AATCC EP1) (see 14.7) or spectrophotometer as specified in AATCC EP7, Instrumental Assessment of the Change in Color of a Test Specimen.

7. Sampling

7.1 Cut one specimen of polyester test fabric for each dyeing required (see Figs. 1 and 2).

7.2 Use the same dye, metal, carrier, and chelating agent solutions for all dyeings, as needed.

8. Specimens

8.1 Cut equal-weight specimens of polyester test fabric. A convenient weight for each specimen is 10 g.

8.2 Create dye and auxiliary solutions as described in Section 9.

9. Preparation of Reagents

9.1 Dye solutions (for *each* control and test dye).

9.1.1 Weight accurately 2.0 g of dye (see 14.8).

9.1.2 Measure 100 mL of distilled water and heat to 49–60°C (120–140°F). Add a small quantity of this water to the dye.

9.1.3 Paste well with a clean stirring rod until the dye is dispersed. Add several aliquots of the warm water with stirring.

9.1.4 Dilute to 1000 mL with distilled water to give a 2.0 g/L dye dispersion.

9.2 Cupric sulfate solution.

9.2.1 Weigh accurately 2.51 g of cupric sulfate.

9.2.2 Prepare a solution in 1000 mL of distilled water. This will result in a solution of 1.0 g/L metal ions (see 13.9).

9.3 Ferrous ammonium sulfate solution.

9.3.1 Weigh accurately 7.02 g of ferrous ammonium sulfate.

9.3.2 Prepare a solution in 1000 mL of distilled water. This will result in a solution of 1.0 g/L metal ions (see 14.9 and 14.10).

9.4 Carrier solution.

9.4.1 Weigh accurately the carrier (see 14.11).

9.4.2 Prepare a 50.0 g/L carrier solution using distilled water.

9.5 Chelating agent solution.

9.5.1 Weigh accurately the chelating agent (see 14.12).

9.5.2 Prepare a 10.0 g/L chelating agent solution using distilled water.

10. Procedure

10.1 To test the *effectiveness of chelating agents* in sequestering metals in a dyebath to prevent metal-induced color change, perform dyeings with C.I. Disperse Red 60 and C.I. Disperse Yellow 42 under “control,” “with metal,” and “with metal and chelating agent” conditions (see Fig. 1 and Tables I and II.)

10.2 To test *metal sensitivity of disperse dyes*, perform dyeings with C.I. Disperse Red 60, C.I. Disperse Yellow 42, and test dyes under “control” and “with metal” conditions (see Fig. 2 and Tables I and II.)

10.3 Prior to dyeing, rinse all dyeing containers with a 10% solution of phosphoric acid followed by distilled water rinse.

10.4 Prepare dyebaths according to Table I and/or Table II.

10.5 Pre-wet specimens in distilled water. Squeeze to remove excess water. Add to the dyebath with stirring.

10.6 Use a pH meter to adjust dyebath, including specimen, to pH 5.0 with acetic acid (see 14.13).

10.7 Load pressure-dyeing containers into appropriate equipment. Load atmospheric boil dyeings into suitable dyeing apparatus.

10.8 Raise to dyeing temperature at 2.2°C/min (4°F/min.)

10.8.1 Run atmospheric dyeings at the boil for 60 min.

10.8.2 Run pressure dyeings for 30 min at 130°C (265°F).

10.9 Cool and remove dye containers.

10.11 Afterscour specimens 10 min at 71°C (160°F) with:

1.0% owf nonionic detergent

2.0% owf sodium carbonate

10.12 Rinse.

10.13 Neutralize to pH 7 ± 0.5 with acetic acid.

10.14 Rinse (distilled water optional for after scour).

10.15 Dry.

11. Evaluation

11.1 Make all comparisons as directed in AATCC EP1 or AATCC EP7. Use “control” dyeings in place of original specimens. Use dyeings “with metal” and “with metal and chelating agent” as test specimens.

11.2 Compare C.I. Disperse Red 60 and C.I. Disperse Yellow 42 dyeings “with metal” to the relevant “control” dyeings (see 14.4). If there is no color difference, repeat the test, with dye from another manufacturer, if necessary.

11.3 To evaluate the *effectiveness of chelating agents*:

11.3.1 Compare C.I. Disperse Red 60 and C.I. Disperse Yellow 42 dyeings “with metal and chelating agent” to relevant “control” dyeings. Little or no color change indicates effective sequestering of metals.

11.4 To evaluate *metal sensitivity of test dyes*:

11.4.1 Compare test-dye dyeings “with metal and chelating agent” to relevant “control” dyeings.

12. Report

12.1 Test report should include, at a minimum:

12.1.1 A description or identification of each dye and chelating agent tested.

12.1.2 Evaluation was performed using AATCC TM161-2018e.

12.1.3 Gray Scale for Color Change grade for each dyeing. If dyeings are performed in replicate, report average grade for each dyebath composition.

12.1.4 Any modifications of the test method.