

Chelating Agents: Disperse Dye Shade Change Caused by Metals; Control of

Developed in 1983 by AATCC Committee RA90; editorially revised 1985, 1988 (with title change), 2004; editorially revised and reaffirmed 1986, 1987, 1992, 2002.

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 which can cause shade change.

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

2. Principle

2.1 The effectiveness of chelating agents in sequestering metals in a dye bath, thereby preventing metal-induced shade 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 inactivating metal ions by formation of a water soluble complex. SYN: *sequestering agent*.

3.2 **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.

3.3 **shade change**, n.—a change from the normal or expected color of a textile material.

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.

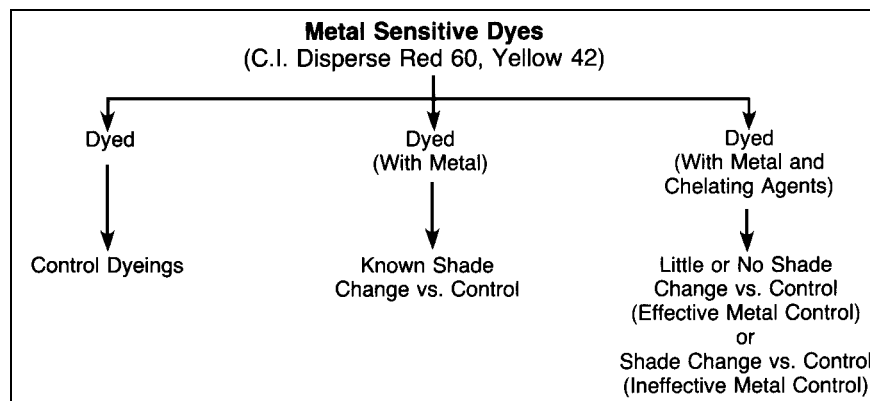


Fig. 1

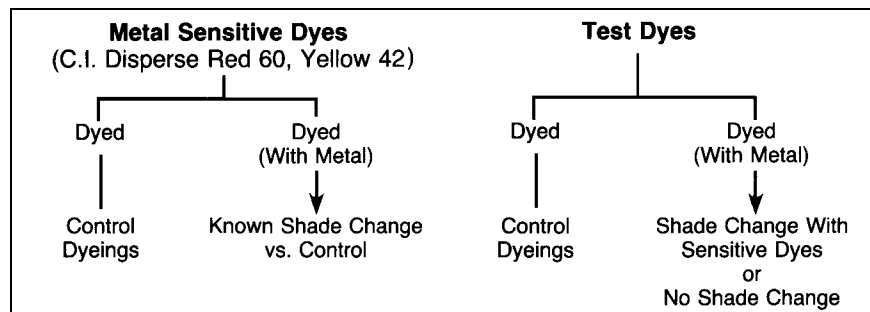


Fig. 2

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 handling 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, MSDS 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 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 12.19).

5. Uses and Limitations

5.1 Accurate results and expected shade changes depend on the particular 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. Particular 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 which may contain metal complexes that are potentially damaged by chelating agents.

6. Apparatus and Materials

6.1 Test fabric. Clean 100% polyester fabric (filament or staple), desized (as needed) and prescoured (see 12.1).

6.2 Control dyes (C. I. Disperse Red 60 and C. I. Disperse Yellow 42) (see 12.2 and 12.3).

6.3 Test dyes.

6.4 ACS Reagent CuSO_4 (cupric sulfate) MW 159.606.

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

6.6 Carrier (if used).

6.7 Chelating agent.

6.8 Distilled water.

6.9 ACS Reagent CH_3COOH (acetic acid) 99.7%.

6.10 ACS Reagent H_3PO_4 (phosphoric acid) 85%.

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

6.12 Gray Scale for Color Change (see 12.20).

7. Test Specimens

7.1 For each test, cut an appropriate number of equal weight specimens of polyester fabric. A convenient weight for each specimen is 10 g.

8. Procedure

8.1 Weigh accurately 2.0 g of the test and control dyes.

8.2 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 from 8.1. Paste well with a stirring rod until the dye is dispersed. Add several aliquots of this water with stirring. Dilute to 1000 mL with distilled water to give a 2.0 g/L dye dispersion.

8.3 Weigh accurately 2.51 g of cupric sulfate and 7.02 g of ferrous ammonium sulfate.

8.4 Prepare a solution of each of the metal salts from 8.3 in 1000 mL of distilled water. This will result in a solution of 1.0 g/L metal ions (see 12.4 and 12.5).

8.5 Weigh accurately the carrier, if used (see 12.6).

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

8.7 Weigh accurately the chelating agent (see 12.7).

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

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

8.10 Prepare dyebaths according to Table I. (If test dyes are to be evaluated, use test dye amounts to give a depth of shade comparable to that of C. I. Disperse Yellow 42 dyes.)

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

8.12 Use a pH meter to adjust dyebath including specimen to pH 5.0 with acetic acid (see 12.8 and 12.9).

8.13 Load pressure dyeing containers into Atlas Launder-Ometer (or comparable equipment). Load atmospheric boil dyeings into suitable dyeing apparatus.

8.14 Raise to dyeing temperature at 2.2°C/min (4°F). Run atmospheric dyeings at the boil for 60 min and pressure

dyeings for 30 min at 130°C (265°F).

8.15 Cool and remove dye containers.

8.16 Afterscour specimens with:

1.0% owf nonionic detergent

2.0% owf sodium carbonate

scour 10 min at 71°C (160°F)

Rinse

Neutralize to pH 7 ± 0.5 with acetic acid

Rinse

(Distilled water optional for after scour).

8.17 Dry.

9. Evaluation

9.1 Mount the specimens side by side, face up with the warps in the same direction.

9.2 Visually determine the shade change for the test dyeings by comparing to control dyeings utilizing a Gray Scale for Color Change (AATCC Evaluation Procedures 1 and 7).

10. Report

10.1 Report the Gray Scale ratings for color change.

10.2 Report the instrumental evaluation data.

11. Precision and Bias

11.1 *Precision.* Precision of this method is being established. Until a precision statement is generated for this method, use standard statistical techniques in making any comparisons of test results for either *within-laboratory* or *between-laboratory* averages.

11.2 *Bias.* The true value of disperse dye shade change cause by metals can only be defined in terms of a test method. Within this limitation, this method has no known bias.

Table I—Composition of Dyebath and Conditions of Use for Evaluating Disperse Dye Shade Change and Chelating Agents (See 12.10)

Parameters	Dye at Boil, Metals Tested		Dye Under Pressure (130°C [265°F]) Metals Tested		
	Cupric C.I.	Ferrous C.I.	Cupric C.I.	Cupric C.I.	Ferrous C.I.
	Disperse Red 60	Disperse Red 60	Disperse Red 60	Disperse Yellow 42	Disperse Red 60
Fabric Weight	10 g	10 g	10 g	10 g	10 g
Water Amount (see 12.11)	251 mL	176 mL	267 mL	267 mL	184 mL
Chelating Agent Amount	0.5% (5 mL)	0.5% (5 mL)	0.5% (5 mL)	0.5% (5 mL)	0.5% (5 mL)
Metal Amount	10 ppm (3 mL)	10 ppm (3 mL)	10 ppm (3 mL)	10 ppm (3 mL)	10 ppm (3 mL)
Carrier Amount (see 12.12 and 12.13)	8.0% (16 mL)	8.0% (16 mL)	No carrier	No carrier	4.0% (8 mL)
Dye Amount (see 12.14)	0.5% (25 mL)	2.0% (100 mL)	0.5% (25 mL)	0.5% (25 mL)	2.0% (100 mL)
Adjust pH with Acetic Acid to	5.0	5.0	5.0	5.0	5.0
Liquor-To-Goods Ratio (see 12.16)	30/1	30/1	30/1	30/1	30/1
Dyeing Temperature	Boil	Boil	130°C (265°F)	130°C (265°F)	130°C (265°F)
Dyeing Time	60 min	60 min	30 min	30 min	30 min

In addition to the formulations listed above, needed dyeings include: (a) Control dyeing: eliminate trace metal and chelating agent and increase water by 8 mL. (b) Dyeing with metal: eliminate chelating agent and increase water by 5 mL. Solution Concentrations: Dye, 2 g/L; Cupric or Ferrous ions, 1 g/L; Chelating agent, 10 g/L; Carrier, 50 g/L.