

Speckiness of Colorant Dispersions: Evaluation of

Developed in 1992 by AATCC Committee RA87; reaffirmed 1993, 2006; editorially revised 1994, 2004; revised 1995, 1996 (with title change); editorially revised and reaffirmed 2001.

1. Purpose and Scope

1.1 Agglomerates in colorant dispersions may become apparent as specks on a continuously dyed (padded) or on a printed fabric, especially when pale and light shades are produced.

1.2 This test method is used to determine speckiness, primarily of disperse dye, vat dye and pigment dispersions.

2. Principle

2.1 A dispersion of a dye is filtered through a polyester/cotton blend fabric.

2.2 The degree of speckiness is assessed visually.

3. Terminology

3.1 **disperse dye**, n.—a nonionic dye which is sparingly soluble in water and which has substantivity, when properly dispersed, for polyester, polyamide and some other manufactured polymeric fibers.

3.2 **dispersion**, n.—in *textile wet processing*, a suspension of very fine particles in a liquid phase.

3.3 **pigment**, n.—a colorant in particulate form which is insoluble in the substrate, but which can be dispersed in the substrate to modify its color.

3.4 **speck**, n.—a small particle, such as an agglomerate in a liquid dispersion, or a very small spot of dark color on a dyed substrate.

3.5 **speckiness**, n.—in *textile dyeing and printing*, the quality or state of containing specks.

3.6 **vat dye**, n.—a water-insoluble dye, usually containing keto groups, which is normally applied to the fiber from an alkaline aqueous solution of the reduced enol (leuco) form which is subsequently oxidized in the fiber to the insoluble form.

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 all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 Use heat resistant gloves when working at hot air ovens and thermofixation devices.

4.4 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 12.1).

5. Apparatus and Materials

5.1 Filter cloth: 65/35 polyester/cotton, bleached woven broadcloth (shirting weight), approx. 240 × 240 mm square (see 12.2). A fabric of similar construction and of varying blend ratios may be used, but it must then be stated in the report.

5.2 Funnel, Buchner, polypropylene, 110 mm diameter (see 12.3). Cut out the perforated bottom smoothly and even with the rim using a knife or other suitable implement. Both parts are used in the test.

5.3 Filtering flask, heavy wall with side tabulation, 2 L.

5.4 Rubber stopper, 1 hole, to fit the filtering flask.

5.5 Stirrer, small propeller type, diameter approx. 20 mm, speed up to 2,200 rpm.

5.6 Dishes, evaporating, glass or porcelain, 1 L (approx. 150 mm diameter), three, for testing vat dyes.

5.7 Ovens.

5.7.1 Drying, without air circulation.

5.7.2 Thermofixation.

6. Reagents (only for testing vat dyes)

6.1 Sodium hydroxide (NaOH) (30%).

6.2 Sodium hydrosulfite conc. powder ($\text{Na}_2\text{S}_2\text{O}_4$).

6.3 Hydrogen peroxide (H_2O_2) (30%).

6.4 Acetic acid (CH_3COOH) (80%).

7. Preparation of Liquid Dispersions for Sampling

7.1 On prolonged storage, liquid dispersions tend to settle and may develop a more or less tacky sediment. It is imperative to ensure complete homogeneity of the dispersion prior to testing. Thoroughly mix dispersions in drums using a mechanical mixing device, such as a propeller stirrer or homogenizer. The liquid must be smooth and free of sediment and lumps before removing a sample for testing. Thoroughly shake laboratory samples to ensure that all dried material adhering to the cover and sides of the container is re-incorporated into the liquid. Then remove the cover and stir the sample either mechanically or by hand until all sediment and/or lumps are completely redispersed. Replace the cover and again shake the sample to ensure complete homogeneity. Test the sample immediately after homogenization. When storing the sample for future use, thoroughly clean the lid and the container lip prior to closure.

8. Procedure

8.1 Identify the filter cloth with the experiment or sample number in one of the corners and ensure that it is free of extraneous specks.

8.2 Clean and dry the funnel. Prepare the funnel assembly by placing the top part of the funnel upside down on a clean surface and drape the fabric over it as flat as possible with the identification mark facing down towards the surface. The side of the fabric with the identification will then be on top during filtration and is used for subsequent evaluation (see 9.1). Snap and press the bottom half of the funnel to the top half, causing the fabric to become a tight, smooth filter in the funnel.

8.3 Place the assembled funnel straight on the filtering flask using the rubber stopper to ensure that the funnel remains upright (and the filter fabric horizontal) during filtration and rinsing.

8.4 Weigh 7.5 ± 0.075 g of 100% powder or 15.0 ± 0.15 g of 50% liquid (or an equivalent weight of other concentrations) in a weighing cup and transfer to a 400 mL graduated beaker containing approx. 200 mL deionized or distilled water at room temperature. Rinse the weighing cup with water from a spray bottle.

8.5 With powder and granular forms:

stir 3 min with the propeller of the stirrer in the center and just above the container bottom. Adjust the stirrer and speed to create a vortex terminating at the top of the propeller.

8.6 With liquid forms: stir as in 8.5 except for 30 s.

8.7 After stirring, transfer the dispersion to the 1 L beaker. Rinse the 400 mL beaker with 200 mL deionized or distilled water at room temperature and pour the rinse water into the 1 L beaker. Dilute the dispersion further to 800 mL.

8.8 Pre-wet the fabric in the filter with 200 mL water.

8.9 Stir the dispersion in the beaker for approx. 30 s and pour it into the funnel.

8.10 Rinse the beaker with 200 mL water and pour it into the funnel.

8.11 Rinse the funnel with an additional 200 mL water and let stand for about 1 min until dripping has completely stopped.

8.12 Carefully remove the filter cloth from the funnel and place it on a blotting paper to remove excess water.

8.13 For disperse dyes:

8.13.1 Dry the fabric in an oven without air circulation at $80 \pm 5^\circ\text{C}$.

8.13.2 Thermoflex for 60 s at $210\text{--}220^\circ\text{C}$.

8.14 For vat dyes proceed as follows:

8.14.1 Prepare 400 mL of a fresh reducing solution in an evaporating dish (see 12.4) at $60\text{--}70^\circ\text{C}$ containing 30 mL/L sodium hydroxide, 30%, and 20 g/L sodium hydrosulfite conc. powder.

8.14.2 Fully immerse the filter cloth in

the reducing solution in the evaporating dish for 5 min. Do not move the cloth during this time.

8.14.3 Immerse the filter cloth for 1 min without agitation in an evaporating dish containing deionized or distilled water at $15\text{--}25^\circ\text{C}$.

8.14.4 Prepare 100 mL of an oxidizing solution in an evaporating dish at $40\text{--}50^\circ\text{C}$ containing 10 mL/L hydrogen peroxide, 30%.

8.14.5 Fully immerse the filter cloth in the oxidizing solution in the evaporating dish for 2 min. Do not move the cloth during this time.

8.14.6 Neutralize the filter cloth for 2 min at $15\text{--}25^\circ\text{C}$ with 200 mL/L acetic acid, 80%, in a 400 mL beaker.

8.14.7 Rinse for approx. 30 s in cold running water.

8.14.8 Dry the fabric in an oven at $80 \pm 5^\circ\text{C}$.

8.15 For pigments:

8.15.1 Dry the fabric in an oven without air circulation at $80 \pm 5^\circ\text{C}$.

9. Evaluation

9.1 Visually examine the fabric for specks on the side with the identification mark. Count individual specks. Base the acceptability on user requirements; for example, acceptable, marginal, unacceptable.

10. Report

10.1 Report the dyestuff tested and weight used, the number of specks and

the speckiness rating.

11. Precision and Bias

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

11.2 *Bias*. Speckiness of colorant dispersions 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.

12. Notes

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

12.2 Available as Style 7409 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.

12.3 Available as Cat. #10-362E from Fisher Scientific, 50 Fadem Rd., Springfield NJ 07081; tel: 201/467-6400. May also be available from other laboratory equipment suppliers.

12.4 The reason for using evaporating dishes for the reduction and oxidation of vat dyes is to keep the fabric flat and without agitation. This prevents any specks from dissolving and leveling.