

Test Method for Dispersibility of Disperse Dyes: Filter

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Table I. Selection of Applicable Test

Test	Whatman Filter Paper Combination	Application of Dyes	pH of Dispersion
I	#2 over #4	Package dyeing of polyester	4.5-5.0
II	#4 over #4	Beck dyeing of polyester	4.5-5.0
III	#4 over #4	Dyeing of nylon carpet and apparel	9.0-10.0

1. Purpose and Scope

1.1 This test determines the dispersibility as evaluated by filtering time and filter residue of disperse dyes under standard conditions in aqueous media. Additional information such as variables which affect accuracy and repeatability of the test are specified in Section 10.

1.2 This test method is to be used for determining the degree of dispersion at the specified conditions in aqueous media only.

2. Principle

2.1 A quantity of disperse dye is diluted, heated and passed through filter papers of specified micron size. The time needed for the dye dispersion to pass through the filter is then recorded.

2.2 Three tests are outlined according to intended dye application (see 10.5 and Table I).

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 **dispersibility**, *n.*—the degree to which particles can be broken down to some minimum size such that they will pass through the interstices of a standard filter paper.

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 and a single use dust respirator while handling powder dyes.

4.2 All chemicals should be handled with care. The compounds used in this test method at the concentrations specified are considered to present no significant hazard when used in accordance with good laboratory practices.

4.3 If concentrated acetic acid is diluted to prepare the 10% acetic acid, use chemical goggles or face shield, impervious gloves and an impervious apron during preparation. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

4.4 An eyewash/safety shower should be located nearby and an organic vapor respirator with full facepiece should be readily available for emergency use.

4.5 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administrator's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000; see web site: www.osha.gov for latest version). 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 10.15).

5. Apparatus and Materials

5.1 Buchner funnel: Coors glazed one-piece porcelain, 110 mm diameter (see 10.2).

5.2 Filter papers: Whatman #2, 110 mm diameter; Whatman #4, 110 mm di-

ameter or exact equivalent filter paper from other sources (see 10.4).

5.3 Stainless steel ring with approximate dimensions 103 mm (4.05 in.) ID, 111 mm (4.33 in.) OD and 8 mm (0.32 in.) height to hold filter paper in place (see 10.6 and 10.12).

5.4 Filtering flask with side tube, capacity 1000 mL.

5.5 Manometer to measure vacuum (pressure drop).

5.6 Vacuum pump or water aspirator to obtain vacuum of 560 ± 100 mm Hg (22 ± 4 in. Hg).

5.7 Vacuum control valve.

5.8 Rubber tubing, vacuum.

5.9 Stopwatch.

5.10 Beakers, 400 mL or larger.

5.11 Balance, analytical.

5.12 Tetrasodium salt of ethylenediaminetetraacetic acid (EDTA) as 25% solution, such as Cheelox BF-12 or equivalent.

5.13 Tetrasodium pyrophosphate, anhydrous (TSPP) ($\text{Na}_4\text{P}_2\text{O}_7$), 10% solution.

5.14 Acetic acid (CH_3COOH), 10% solution.

5.15 Filter Residue Scale (see Fig. 1 and 10.12).

5.16 Distilled or deionized water, which does not change significantly in pH on heating. This water is modified with 0.25 g/L of chelating agent (EDTA) as specified in 5.12.

5.17 Dye samples (see 6.2).

5.18 Stirrer, laboratory, magnetic.

5.19 Graduated cylinder, 250-mL.

5.20 pH meter.

6. Procedure

6.1 Water Preparation.

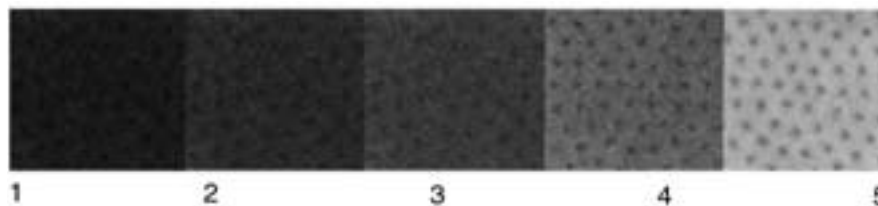


Fig. 1—Filter residue scale.

6.1.1 Preheat 400 mL of water to 71°C (160°F). This will be used to preheat the funnel before conducting test (see 10.7 and 6.6).

6.1.2 Prepare special water (see 5.16) for use in dye dispersion as follows: Add 0.25 g/L EDTA (see 5.12) to distilled or deionized water.

6.2 Weighing of dyes.

6.2.1 Weigh out 2.0 ± 0.1 g of dye powder or 4.0 ± 0.2 g of 50% liquid of the dye sample to be tested. Also weigh out an equal quantity of the standard dye (see 10.3).

6.2.2 If the dyes are of high tinctorial strength, the weighed amount can be reduced to 1 g/200 mL but should be reported as only 1g used (see 10.8).

6.2.3 When evaluating two different dyes of approximately equal shade but of different tinctorial values, one dye is always selected as the standard. Then the other product is run on an equal concentration (grams/200 mL) basis. When evaluating two samples of the same dye, the standard is the one furnished as standard material by the respective dye manufacturer. When evaluating the same dye from two different manufacturers, one should determine if the two products are of equal strength before running the test. If a strength difference is determined, the two products should be evaluated as outlined in the first two sentences of this paragraph.

6.3 Add the weighed dye (see 6.2.1) slowly and completely to 200 mL of the vigorously agitated specially treated water (see 5.16) at 43-49°C (110-120°F) in a 400 mL beaker (see 10.9) using a magnetic stirrer. Adjust the pH of the dye dispersion as follows:

6.3.1 Tests I and II: adjust to pH 4.5-5.0 with a 10% solution of acetic acid (see 5.13 and 10.14).

6.3.2 Test III: adjust to pH 9-10 with TSPP (see 5.13 and 10.14).

6.4 Heat the adjusted solution 5-10 min to 71°C (160°F). Stir by means of a magnetic stirrer to prevent localized heating (see 10.1 and 10.10).

6.5 Pour 200-300 mL of 71°C (160°F) water prepared in 6.1.1 through a 110 mm funnel without filter paper to preheat the funnel. Wait 25 ± 10 s. Turn on the vacuum until the water has passed through the funnel. Quickly wipe funnel dry (see 10.6 and 10.7).

6.6 Cut vacuum off and immediately place Whatman filter papers in dry Buchner funnel (see 10.4, 10.5, 10.6 and Table I):

Test I:	Whatman #2 on top Whatman #4 on bottom
Test II:	Whatman #4 on top Whatman #4 on bottom
Test III:	Whatman #4 on top Whatman #4 on bottom

6.7 Insert stainless steel ring in funnel over filter papers and turn on vacuum.

The vacuum should be 560 ± 100 mm (22 ± 4 in.). This range is obtained by using either a standard vacuum pump or house vacuum. A water aspirator may not give this vacuum until a liquid is introduced into the system. In this event, the test should proceed after pouring the dye dispersion into the funnel and waiting 10-13 s for full vacuum.

6.7.1 With the vacuum on, immediately pour dye dispersion which has been heated to 71°C (160°F) (see 6.4) into funnel and begin timing. The end point is reached when the filter paper changes from a wet to a dry visual appearance. Record time up to 120 s to the nearest s.

6.7.2 In order to achieve more reproducible results, especially with dyes in Class 3 (intermediate values are interpolated) or lower, it may be advisable to rinse the filter paper while it is in the funnel with 10-15 mL of the specially prepared water (see 6.1.2) before drying (see 10.13).

6.8 Allow filter paper to dry and evaluate as outlined in Section 7).

7. Evaluation

7.1 Record time for filtration and rate as follows:

Class A—0-24 s
Class B—25-49 s
Class C—50-74 s
Class D—75-120 s
Class E—> 120 s

7.2 Examine residue on filter paper versus the Filter Residue Scale (standard photographic replicas) (see 10.13, 10.14). Also examine the paper for any visible coarse or granular particles (see 10.13). If present the test is automatically rated as Class 1. If none is present, rate as follows:

Class 5—Excellent
Class 4
Class 3—Intermediate values are interpolated
Class 2
Class 1—Poor

8. Report

8.1 Classify and report the following:

8.1.1 Test Number (see 10.6 and Table I).

8.1.2 Class according to filtration rate (see 7.1).

8.1.3 Class according to amount of residue (see 7.2).

Example: I-A-3, where I is the Test Number (Whatman #2 over Whatman #4); A indicates the time of filtration (0-24 s); and 3 represents amount of residue on filter paper as compared with a standard photographic chart.

9. Precision and Bias

9.1 *Precision.* Precision for this test

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

9.2 *Bias.* The dispersibility of disperse dyes 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.

10. Notes

10.1 The results of this test method can vary widely unless all details are run under the conditions outlined. Any variation in the conditions can cause the test to be invalid. Results have been shown to be repeatable in several laboratories when the specified conditions were met.

10.2 Variations in results can be caused by differences in the diameter of the filter funnel due to differences in surface area.

10.3 The dye standard is always included so that the influence of any operator induced variable on the results is minimized.

10.4 Whatman paper is specified because it was used by all participants in developing this test method. Any other filter paper which gives equivalent results may be used. Two filter papers are used to provide a more uniform vacuum across the filtering surface. The filter paper is used as received in the box with the slick side up to avoid variations due to texture differences on the rough side. The coarser paper should always be on the bottom. Other papers may be used if quality and microretention are exactly the same as the Whatman paper.

Whatman #2—Filters particles above 8 μ m.

Whatman #4—Filters particles above 25 μ m.

10.5 Tests I, II and III are defined as follows:

Test I—Whatman #2 filter paper over Whatman #4 filter paper. This test is designed to evaluate the most critical dispersion requirements such as for package dyeing of polyester.

Test II—Whatman #4 filter paper over Whatman #4 filter paper. This test is designed to evaluate dispersion requirements for dyeing at longer liquor ratios (such as beck dyeing).

Tests I and II are carried out at an acid pH of 4.5-5.0 to reflect common dyeing practices for disperse dyes on polyester fiber.

Test III—Whatman #4 filter paper over Whatman #4 filter paper. This test is designed to evaluate the dispersion requirements of disperse dyes for application on nylon (carpet and apparel dyeing). The pH of 9-10 specified for this test reflects common industrial practice.

10.6 Since the Whatman filter paper is cellulosic in nature, it can swell quite readily when wet out. Therefore, if the paper is prewet, the time of filtration will go up as a result of the wet out temperature and/or time lag. Thus it is *imperative* that the paper not be wet prior to the test, since the micron retention of the paper is reduced. This is the reason for the stainless steel ring, which holds the filter paper in place without prewetting. On dyes which have a very small particle size, the speed of filtration will not be changed to a sizeable degree by a vacuum alteration. However, a reduced vacuum can result in faster filtration