

# Relative Color Strength of Dyes in Solution

**Established in 1998 by AATCC Committee RA98; under jurisdiction of AATCC Committee RA36; reaffirmed 1999, 2005; editorially revised and reaffirmed 2000. Technically equivalent to ISO 105-Z10.**

## 1. Purpose and Scope

1.1 This is a test method to determine the color strength of a dye sample relative to a reference dye by means of spectrophotometric transmittance measurements on solutions of each. It is a comparison of the color strength of the dyes in a chosen solvent which may or may not correspond to strengths when applied to a textile substrate.

1.2 To be valid as a measurement of the relative color strength of a dye for a particular textile end-use, additional tests must be performed to demonstrate that the relative strength in solution is equal to the relative strength when applied to the textile substrate (see 5.4).

1.3 The test is often used by dye manufacturers and users because of its speed, repeatability and reproducibility.

## 2. Principle

2.1 Solutions of the test dye and the reference dye are prepared in known concentrations and their transmittance values are determined using a spectrophotometer. The relative color strength for the test dye is then calculated using absorbance and concentration values.

## 3. Terminology

3.1 **absorbance**, *n.*—the logarithm to the base 10 of the reciprocal of transmittance.

NOTE: Absorbance is proportional to the mass of absorbing material in the path length of a spectrophotometer cell (syn: *absorbance value*).

3.2 **color strength**, *n.*—a measure of the ability of a dye to impart color to other materials.

NOTE: Color strength is evaluated by light absorption in the visible region of the spectrum (see 13.1).

3.3 **relative color strength**, *n.*—in *spectrophotometric testing of dyes*, the percentage of the color strength of a sample relative to that of a reference dye assigned a color strength of 100% (see 10.2).

3.4 **transmittance**, *n.*—of *light*, that fraction of the incident light of a given wavelength which is not reflected or absorbed, but passes through a substance.

NOTE: In this test, the transmittance of the material is measured on a spectrophotometer, and corrected for that of pure solvent for the same path length.

## 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 the 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 chemical goggles or face shield, impervious gloves, and an impervious apron during dispersing and mixing of acids, alkalis and organic solvents. Concentrated acids, alkalis and organic solvents should be handled in a well ventilated hood. CAUTION: Always add acid to water.

4.4 Acetone, methyl alcohol and ethyl alcohol are highly volatile liquids and should be stored in the laboratory only in small containers away from heat, open flames and sparks. These chemicals should not be used near an open flame.

4.5 Cellosolve (2-ethoxyethanol) is combustible and may be harmful or fatal if ingested or inhaled. It is also a reproductive hazard and should only be used when absolutely necessary.

4.6 An eyewash/safety shower should be located nearby and an organic vapor respirator and a self contained breathing apparatus 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 government 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 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 14.1).

## 5. Uses and Limitations

5.1 This method is not applicable to all dyestuffs; e.g., pigments, due to solubility and/or other limitations. Dyes that are commonly measured include acid, basic, direct and disperse. Many reactive dyes are not easily measured using this procedure.

5.2 Basic requirements for this method are that the dye solutions do not scatter light and do obey the Lambert-Beer or Beer-Bouguer Laws as well as having identical or similar absorption curves of the samples and the reference dyes in the visible region of the spectrum (see 13.9).

5.3 Identical or similar absorption curves are usually obtained if the tests are carried out for the purpose of dye production control of batches/deliveries of the same dye. This method is not applicable for the evaluation of dyes with distinctly different hues or strengths or chemical compositions.

5.4 The method may be useful in the prediction of relative dye strength in process applications; e.g., dyeing. It is generally accepted that some reasonable correlation can be obtained between application and solution measurements. Exceptions occur where there is a significant difference in the relative amount of colored but non-exhausting or non-fixing impurities between the specimen under test and the reference dye; e.g., reactive dyes. Exceptions may also occur for some reactive dyes which exhibit shade differences between their hydrolyzed and unreacted forms (see additional caution for reactive dyes in 8.3.3).

5.5 As the relative color strength is determined by a comparison with a reference dye, the reference dye is assumed to remain constant. It is essential, therefore, to ensure careful storage and control of the reference dye. Many dyes are hygroscopic and sensitive to oxidation. Protect the reference dye from exposure to light in firmly sealed containers that are impervious to moisture.

## 6. Apparatus and Materials

6.1 Flasks, volumetric class A.

6.2 Pipettes, volumetric class A.

6.3 Analytical balance with accuracy of weighing to 0.0005 g.

6.4 Cells for spectrophotometers, 5 mm or 10 mm path length as needed, analytical grade or optical quality. May be cuvette or flow-through cell.

6.5 Spectrophotometer.

## 7. Reagents

- 7.1 Sulfuric acid— $\text{H}_2\text{SO}_4$ .
- 7.2 Acetic acid— $\text{CH}_3\text{COOH}$ .
- 7.3 Sodium hydroxide— $\text{NaOH}$ .
- 7.4 Sodium carbonate— $\text{Na}_2\text{CO}_3$ .
- 7.5 Buffering agents.
- 7.6 Solvents (see 13.3).
  - 7.6.1 For water-soluble dyes:
    - 7.6.1.1 Water, demineralized.
  - 7.6.2 For water-insoluble dyes:
    - 7.6.2.1 Methyl alcohol ( $\text{CH}_3\text{OH}$ , methanol), anhydrous.
    - 7.6.2.2 Acetone ( $\text{CH}_3\text{COCH}_3$ ).
    - 7.6.2.3 N-methyl-2-pyrrolidone.
    - 7.6.2.4 Ethylene glycol monoethyl ether (2-ethoxyethanol, Cellosolve).
    - 7.6.2.5 Mixtures of the above solvents as well as other solvents appropriate for the test dye.

## 8. Preparation of Dye Solutions

- 8.1 Storage of dyes.
  - 8.1.1 Store the samples in tightly sealed containers to avoid erroneous results arising from changes in moisture levels. Condition powder samples in a controlled atmosphere for 4 h as described in ASTM D 49.
- 8.2 Stock solutions.
  - 8.2.1 Weigh an amount of not less than 0.5 g to avoid errors attributable to micro-inhomogeneities to the nearest 0.0005 g (see 13.4 and 13.8).
  - 8.2.2 Transfer the weighed dye quantitatively to a volumetric flask containing approximately one-third of the total volume of solvent, then dissolve/disperse it. In general, pre-dissolve/disperse the dye in about 20 mL of the selected solvent. A mixture of solvents or an additive may be needed.
    - 8.2.2.1 Water-soluble dyes may need to be heated to effect dissolution. If heated, cool the dissolved/dispersed mixture to room temperature (see 13.5).
  - 8.2.3 Make up the volumetric flasks to the mark and homogenize the solution/dispersion by stirring or by turning the flask end over end.
- 8.3 Test solutions.
  - 8.3.1 Dilute the concentrated stock solution as directed (see 13.6) to obtain maximum absorption (the minimum transmittance at the lowest point) within the range of 10-60% transmittance (see 13.5). (The same solvents used to make the concentrated stock solution may not be the appropriate choice for dilution. For example, many disperse dyes are dispersed into water for the stock solution, and then pipetted into a solvent/water mixture for dilution.)
  - 8.3.2 Some additives may be recommended to improve the stability and reproducibility.
  - 8.3.3 To adjust the pH of water soluble dyes use an acid such as acetic or sulfuric, a base such as sodium carbonate or

sodium hydroxide, or a buffering agent. (However, sodium carbonate or sodium hydroxide should not be used with reactive dyes.)

8.3.4 To eliminate the influence of unwanted metal ions use a sequestering agent.

8.3.5 To prevent agglomeration in water use a surfactant; e.g., ethylene oxide adducts to alkanols.

8.3.6 Use a dispersing agent or an antioxidant, if appropriate.

8.3.7 Volumetric equipment and solvents should be at ambient temperature.

## 9. Procedure

9.1 When a dye specimen is measured for the first time, it is advisable to confirm the validity of the relationship (Beer's Law) under the conditions selected (concentration, solvent) within a range of concentrations equal to one-half and twice the concentration actually used.

9.2 Determine the wavelength ( $\lambda$ ) of minimum % transmittance. The test and the reference specimens should have the same wavelength of minimum % transmittance.

9.3 Carry out the measurement as soon as possible after preparation of the solution to exclude the influence of changes in solution.

9.4 Adopt appropriate techniques with light sensitive solutions; e.g., using darkened flasks, or working in dimly lit surroundings (see 13.7).

9.5 Measure the transmittance of the solution in cells (cuvette or flow-through cell) of identical path length (usually 5 mm or 10 mm).

9.5.1 The difference in absorbance between the test dye and reference dye shall not exceed 20%, to minimize the influence of variations in the spectrophotometric response over a relatively wide range of concentrations.

9.5.2 The transmittance,  $T$ , of the dye solution is obtained differently with a double-beam spectrophotometer compared to a single-beam spectrophotometer, but yields the same result. With a double-beam instrument, simultaneous measurements may be made with a cell of pure solvent placed in the reference beam, and the dye solution in the sample beam. Single-beam instruments require that the instrument first be standardized with pure solvent. Then, the dye specimens are measured successively in the same cell.

9.5.3 Some narrow (0.5-2.0 nm) band-pass, analytical spectrophotometers used for dye strength determination measure both transmittance and absorbance, directly. In this case, it is not necessary for the system or the user to calculate the absorbance separately.

## 10. Calculations

10.1 Base the evaluation on Beer's law, using the following equation:

$$A = \log_{10}(1/T_\lambda) \quad (1)$$

10.1.1  $A$  is the absorbance at wavelength of minimum transmittance (see 13.2).

10.1.2  $T_\lambda$  is the transmittance (in decimal fraction form, where 100% = 1.0) at wavelength of minimum transmittance.

10.2 Calculate the relative color strength to the nearest 0.1% using the following equation:

$$F_s = 100 (A_2 C_1 / A_1 C_2) \quad (2)$$

where:

$F_s$  is relative color strength in percent

$C$  is the dye concentration

subscript 1 refers to the reference dye

subscript 2 refers to the dye under test

10.2.1 Assuming  $C_1 = C_2$ , the following simplified equation is obtained:

$$F_s = 100 (A_2 / A_1) \quad (3)$$

## 11. Report

- 11.1 Identification of test dye.
- 11.2 Dye mass.
- 11.3 Solvent used (if solvent system is light-sensitive, note illumination conditions).
- 11.4 Dissolving conditions (e.g., temperature, also include names and concentrations of any additives used to improve stability and reproducibility, per 8.4).
- 11.5 Dilution factor.
- 11.6 Concentration and temperature of measured solution.
- 11.7 Measuring instrument (e.g., type and band width).
- 11.8 Cell path length.
- 11.9 Wavelength of analysis, also note bandpass and reporting interval of spectrophotometer.
- 11.10 Relative color strength.
- 11.11 Conditions under which dye was stored.

## 12. Precision and Bias

### 12.1 Precision.

12.1.1 An interlaboratory study by ten laboratories was conducted in 1987. Absorbance was measured using aqueous visible transmittance spectroscopy. A round-robin test battery yielded the results of the test. Several different spectrophotometers were used to determine variance between instruments as well as laboratories. Sample preparation varied as described by this test method for readily soluble dyes and solvent/aqueous solution soluble dyes (disperse dyes). These data are on file at the AATCC Technical Center.

12.1.2 Intra-Lab Variation: The maxi-

imum percent coefficient of variation (%C.V.) calculated as:

$\%C.V. = s/\text{mean} \times 100$  for any given lab was 5.20% for Disperse Blue 56.

12.1.3 Typical %C.V. for water-soluble dyes are below 1%, provided they are sufficiently dissolved. Acid dyes used in the test had typical %C.V. values of 2.0%. Many of the labs in the study indicated that their internal standards for day-to-day and intra-lab variation is  $\pm 0.5\%$ .

12.1.4 Inter-Lab Variation: The maximum %C.V. for between-lab analysis was 6.55% for Disperse Blue 56. Typical %C.V. for water-soluble dyes are below 5%, provided they are sufficiently dissolved.

12.1.5 Spectroscopic measurement of disperse dyes in aqueous/solvent mixtures often yield worse precision than shown for water-soluble dyes, due largely to variances in the solubilities of the disperse dyes. As solubility decreases, light scattering increases, which will lead to higher error levels. For this reason, a given dye is best characterized when it is best dissolved.

12.1.6 One major source of deviation was the use of different spectrophotometers. In several studies, no one single wavelength of maximum absorptivity (transmittance minimum) was used for any of the dyes. Other possible sources of error often include differences in time after initial dissolution; a dye's absorptivity can increase over time after initial dilution until it reaches a maximum. Solution temperature and cis-trans (and azo-hydrazone) isomerism can lead to variability. It is possible and recommended that laboratories develop correction factors (correlation models) between instruments and

laboratories to reduce these errors.

12.1.7 Table I shows *inter-* and *intra-laboratory variability* for the disperse dyes (worst case) studied. Some data points from the study failed the 1.5 IQR fit test and were not included in the data analysis.

#### 12.2 Bias.

12.1.1 There is no independent referee analytical method for determining a true value of this property. This test method has no known bias.

### 13. Notes

13.1 Color strength is traditionally a coloristic concept, being based essentially on visual assessment. Therefore, color strength determined instrumentally should not contradict visual assessment. Such a contradiction would not be expected if the samples to be compared exhibit concentration-dependent differences in absorption only; i.e., their absorption curves in the visible region are identical or show only small differences when the strength of the absorption curves is made equal.

13.2 If the absorption curves differ more markedly (hue or chroma differences), a better visual assessment may be obtained if the summed weighted color strength in the visible region is used.

13.3 When selecting solvents the following aspects are important:

13.3.1 Solubility of dye.

13.3.2 Stability of solutions.

13.3.3 Reproducibility of test.

13.3.4 Applicability of result to other media or practical applications.

13.4 It is essential to ensure that uptake of moisture by hygroscopic dye samples does not lead to errors during weighing.

13.5 It is essential to ensure that the solubility limit is not exceeded during subsequent cooling. Solubility specifications are available from the dye suppliers.

13.6 To avoid errors in dilution, no pipette of total volume less than 5 mL nor volumetric

flask less than 100 mL should be used.

13.7 In certain instances, the solution to be measured may be influenced adversely by the light energy of the source in the measuring instrument (e.g., heat-sensitive or photo-sensitive products). If so, monochromatic illumination, shutter or flash light source should be used.

13.8 When measuring liquid dye samples, it is essential that the sample be thoroughly mixed before an aliquot is taken. If the sample is retained for future use, it must be sealed in a container protected from exposure to light and impervious to moisture.

13.9 A simple method to determine qualitatively the presence of solid dye/dispersing agents in what may appear to be clear solutions is to filter the dye solution through a sub-micrometer filter using a simple syringe filter as is often used in HPLC and determine the absorbance before and after filtration.

### 14. References

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

14.2 General procedure for the determination of relative dye strength by spectrophotometric transmittance measurement (report of the ISCC): R. G. Kuehni, *Textile Chemist and Colorist*, Vol. 4, 1972, p133.

14.3 Difficulties in preparing dye solutions for accurate strength measurement: T. R. Commerford, *Textile Chemist and Colorist*, Vol. 6, 1974, p14.

14.4 Reproducibility of dye strength evaluation by spectrophotometric transmission measurement (report of the ISCC): C. D. Sweeny, *Textile Chemist and Colorist*, Vol. 8, 1976, p31.

14.5 ISO proposed test method 105-Z10, 94/341270.

14.6 AATCC (Committee RA98) training video tape "Techniques for Solution Measurement Used in Dye Strength Determination," (1995) AATCC Committee RA98, Assessment of Dye Strength and Shade Test Methods.

14.7 Possible Test Method for Determining Strength of Acid Dyes: B. L. McConnell, *Textile Chemist and Colorist*, Vol 24, No. 2, February 1992, p23.

14.8 Can A Standard Test Be Developed For Measuring the Strength of Disperse Dyes?, M. D. Hurwitz, *Textile Chemist and Colorist*, Vol 25, No. 9, September 1993, p71.

Table I—Inter- and Intra-Laboratory Variability

Disperse Dye	Yellow 54	Red 60	Blue 56
Intra-Lab %C.V. Maximum	0.75%	1.27%	5.20%
Inter-Lab %C.V. Maximum	1.07%	1.42%	6.55%