Designation: D 6531 – 00

Standard Test Method for Relative Tinting Strength of Aqueous Ink Systems by Instrumental Measurement[1]

This standard is issued under the fixed designation D 6531; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the procedure for determining the relative tinting strength of waterbased ink systems using a computer-aided spectrophotometer.

1.2 This test method is applicable to waterbased printing inks and bases to be used primarily in flexographic and gravure printing applications.

1.3 This test method applies only to single, non-fluorescent pigmented colors and black ink systems for which there is a reference standard containing a pigment of the identical color index name and number.

1.4 The procedure in this test method specifies placing tinted samples in a cuvette for spectrophotometric measurements. The use of thick wet drawdowns as in Test Methods D 2066 are inappropriate due to severe floating problems with aqueous systems.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 2066 Test Methods for Relative Tinting Strength of Paste-Type Printing Ink Dispersions[2]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

E 1331 Test Method for Reflectance Factor and Color by Spectrophotometer Using Hemispherical Geometry[3]

E 1347 Test Method for Color and Color-Difference Measurement by Tristimulus (Filter) Colorimetry[3]

E 1349 Test Method for Reflectance Factor and Color by Spectrophotometer Using Bidirectional Geometry[3]

3. Terminology

3.1 Definition related to color differences are covered in Test Method D 2244. The definitions of tinting strength terms are given in Test Methods D 2066.

4. Summary of Test Method

4.1 A standard and unknown samples are each reduced to the same concentration in a white tinting base then dispersed on a paint shaker.

4.2 The dispersed samples are transferred to cuvettes for reflectance measurements on a spectrophotometer. Hue and strength relative to the standard tint are computed.

NOTE 1—The hue readings indicate the closeness of the unknown sample to that of the standard.

5. Significance and Use

5.1 Tinting strength is an essential property of printing ink dispersions. Although results on bulk tints do not guarantee equivalency of dry printed films, they provide useful parameters for quality control of production batches of bases and finished inks. Test results may also be used for color matching purposes.

6. Apparatus

6.1 *Glass Jars*, capacity approx. 64 g, 33 mm in diameter by 70 mm in height, with screw cap lids.

6.2 *Paint Shaker*.

6.3 *Spectrophotometer*, with small area view (10-mm aperture, diffuse/8° geometry) and with a computer and software to measure reflectance factor, calculate color differences by CIELAB 1976 equation. Tinting strength is calculated by a Kubelka-Monk equation transformed from the lowest tristimulus value. Alternate instrumentation and software may be used by agreement between producer and user, or different plant locations.

6.4 *Disposable Cuvettes*, 10 mm in diameter, with at least one optically true face.

6.5 *Disposable Pipettes* 7-mL capacity, 3.5-mL bulb draw or suitable substitute.

6.6 *Balance*, capacity 100 g, accurate to 10 mg, preferably 1 mg.

6.7 *Black Electrical Tape*.

6.8 *Black Cover*, 90 mm in diameter such as 1-lb ink can,

[1] This specification is under the jurisdiction of ASTM Committee D 1 on Paint and Related Coatings, Materials, and Applications, and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved March 10, 2000. Published May 2000.

[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

with interior painted flat black.

6.9 *Ink Knives*, small.

6.10 *Medicine Droppers*.

7. Materials

7.1 *Standard Ink or Base*—The standard should be 100 % intended strength of the same pigmentation and similar in viscosity to the unknown sample.

7.2 *White Tinting Base*, compatible with and of a viscosity similar to samples being tested.

8. Sampling

8.1 Carefully select a sample that is free of skin and other contamination. The minimum sample per test is 1.0 g of ink, or 0.5 g of base.

8.2 When ready to conduct the test, remove enough sample for the test then reseal the container.

9. Preparation of Tints

NOTE 2—Work quickly, as volatile loss during tint preparation may bias test results.

9.1 Weigh approximately 25 g ± 1 g of white tinting base and put into the jar. Record the weight to two decimal places. Coat the sides of the jar with the tinting base.

9.2 Put the lid on the jar then tip and rotate the jar so that the sides are completely covered with the tinting base.

9.3 Using a medicine dropper, weigh approximately 1 g± 0.1 g of the unknown ink or 0.5 g ± 0.1g of test base into the jar. Record weight to two decimal points.

9.4 Divide exact weight of ink by 0.02. The resulting number represents the total weight of sample plus tinting base for a 2 % tint. Add tinting base accordingly. If the sample is a base, divide the exact weight by 0.01. Add tinting base accordingly to make a 1 % tint. In both cases, total weight should be approximately 50 g. The amount of colorant should be adjusted so that, at the wavelength of calculation, the reflectance is approximately 0.40.

9.5 Place cap on jar and close tightly.

9.6 Repeat 9.1-9.5 with the standard ink or base.

9.7 Securely position the samples in the paint shaker. Position the samples at the leading edge of the shaker to get the most vigorous shaking action.

9.8 Shake sample jars for five minutes.

9.9 Stop the paint shaker and invert the sample jars. Shake for another five minutes.

9.10 Visually check the jars to make sure that the tints are uniform with no white remaining, especially in the corners of the jar. If any white remains, repeat 9.8 until it is completely mixed.

9.11 Let the samples sit for five minutes so the air bubbles can dissipate.

10. Procedure for Spectrophotometric Measurements

10.1 Calibrate the spectrophotometer with D65 light source, 10° observer, and specular component included. (See 10.9.)

10.2 Referring to the manual of the software that is being used, set the computer to measure the reflectance of the standard sample.

10.3 Remove port cover and center an empty cuvette over the port with the optically true side facing the light source. Tape it in place using the black electrical tape.

10.4 Open the tint of standard sample and stir it with a disposable pipet to insure homogeneity. Fill the pipet with the tint.

10.5 Touch the tip of the pipet to the inside surface of the cuvette that the spectrophotometer will read, and fill the cuvette to above the level of the spectrophotometer port in one continuous motion. If it is not, the specimen will not read properly.

10.6 Attach the port cover to the front of the spectrophotometer and place the black cover over the cuvette on the port.

10.7 Take reflectance factor readings of the standard tint at spectral measurements of 20 nm or smaller intervals.

10.8 Remove cuvette from the sample port.

10.9 Repeat 10.1-10.9 with the unknown tint sample.

10.10 Alternatively, measure the CIE tristimulus values of the specimen on a spectrophotometer or tristimulus colorimeter in accordance with Test Method E 1331, Test Method E 1347 or Test Method E 1349. Make measurements as in 10.3-10.8. If hemispherical geometry is used, the specular components may be either included or excluded as long as the same condition is consistently used. The tristimulus values may be based on the CIE 1964 (10°) supplementary standard observer and standard illuminant D65 or the CIE 1931 (2°) standard observer and standard illuminant C, as long as the same basis is consistently used. Record in decimal units the lowest appropriate value (two decimal points), for instance, *X* with blue colors, *Y* with reds, blacks and whites or *Z* with yellows.

11. Calculation

11.1 Referring to the software manual, obtain the computer determined CIELAB values (L*a*b*h) for the tint of the standard ink, or standard base, and the unknown ink or unknown base.

11.2 Calculate the shade difference between the two tints (DL*, Da*, Dc* and Dh).

11.3 Calculate the tinting strength of the unknown according to the Kubelka-Munk equation as follows:

$$TS, \% = \frac{[(1 - R\infty)^2/2R\infty]\,u}{[(1 - R\infty)^2/2R\infty]s} \times 100 \quad (1)$$

where:

$R\infty$ = spectral reflectance factor, expressed as a decimal fraction or by mutual agreement, an appropriate tristimulus value.

NOTE 3—The term $[(1 - R\infty)^2/2R\infty]$ represents *K/S* of the colorant, where *K* is the absorption coefficient and *S* is the scattering coefficient, both of which are specific to a colorant. The equation is reported to work best when the tristimulus value of the tints is about 0.40 and the tinting strength of the unknown is within 10 % of the standard.

12. Precision and Bias

12.1 *Precision*—An interlaboratory study was conducted in which five laboratories tested in triplicate, three aqueous inks

versus standard aqueous ink.[5] The round robin data were analyzed according to Practice E 691. The strength varied from 90 to 107 %. There was one outlier. The within-laboratory-pooled standard deviation was 1.66 % at 6 df, and the between-laboratories pooled standard deviation was 3.02 % at 9 df. Based on these standard deviations, the following criteria should used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of three results obtained by one operator should be considered suspect if they differ by more than 4.74 % of valued measured.

12.1.2 *Reproducibility*—Two results, each the mean of three results obtained by operators in different laboratories should be considered suspect if they differ by more than 7.90 % of valued measured.

12.2 *Bias*—No statement on bias can be made because no suitable reference material exists for determining bias.

13. Keywords

13.1 aqueous printing inks; colorimeter; Kubelka-Munk equation; printing inks; spectrophotometer; tinting strength; waterbased ink systems

[5] Many commercially available white latex paints were investigated. Recommended for its combination of appropriate properties in order to get the results used in this test method, is Sherwin Williams "SuperPaint" Interior Latex Flat White Wall Paint (X3056-99993), Titanium Dioxide (Type III) = 25 %, Silicates = 12 %). If you are aware of alternative suppliers, that would produce the same results, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.