Standard Test Methods for Relative Tinting Strength of Paste-Type Printing Ink Dispersions

1. Scope

1.1 These test methods cover procedures for determining the relative tinting strength of paste-type printing ink dispersions by visual or instrumental evaluation.

1.2 These test methods are applicable to paste-type printing inks, flushed pigments, and other pigment dispersions that are essentially nonvolatile under ordinary room conditions and for which there is a wet reference standard of the same pigmentation and consistency. With proper choice of tinting base, they are applicable to dispersions of any color, including black and white.

NOTE 1—The instrumental procedures for tinting strength are similar in principle to those described in Test Methods D 387, D 2745, and D 4838.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 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 prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 16 Terminology For Paint, Related Coatings, Materials, and Applications
D 387 Test Method for Color and Strength of Chromatic Pigments with a Mechanical Muller
D 2244 Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

D 2745 Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements
D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints
E 284 Terminology of Appearance
E 1331 Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry
E 1347 Test Method for Color and Color-Difference Measurement by Tristimulus (Filter) Colorimetry
E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry

2.2 ANSI Standards:

PH 2.17 Geometric Conditions for Reflection Density
PH 2.18 Spectral Conditions for the Measurement of Optical Density
PH 2.30 Viewing Conditions for Graphic Arts and Photography—Color Prints, Transparencies and Photomechanical Reproductions

3. Terminology

3.1 Definitions relating to color attributes and color differences are covered in Terminology D 16 and E 284.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 tinting strength, \( n \)—the ability of a material to impart its color to a standard base; the reciprocal of the relative concentration required to match the reference material in a standard base.

3.2.2 masstone (or masscolor), \( n \)—the color of a material that is thick enough to mask any background.

3.2.3 undertone (or undercolor), \( n \)—the color of a thin film of a material.

4. Summary of Test Methods

4.1 Thin and thick films of the standard and unknown dispersions are drawn down in juxtaposition on bond and on coated paper. Visual evaluation of the relative undertone and masstone provides a check on color equivalency.

*A Summary of Changes section appears at the end of this standard.

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4.2 The standard and unknown dispersions are each reduced to the same concentration in a suitable tinting base. Thick wet drawdowns of the two tints are evaluated for tinting strength by Test Methods A or B.

4.2.1 Test Method A—Visual Evaluation: If the strength of the tints is judged unequal, aliquots of the stronger tint are further reduced until equivalence is obtained. The tinting strength of the unknown dispersion is calculated from the weight of extra tinting base added per unit weight of the stronger tint.

4.2.2 Test Method B—Instrumental Evaluation: Reflectance measurements are made on thick wet films of the original tints. The tinting strength of the unknown dispersion is calculated according to the Kubelka-Munk equation.

4.3 Preparation of a confirming tint is recommended as an unbiased method of verification. The preferred approach is to prepare a new tint of the unknown at a concentration calculated to match the standard tint.

5. Significance and Use

5.1 Tinting strength is an essential property of printing ink dispersions. Although test results on wet drawdowns and tints do not guarantee equivalency of dry printed ink films, they provide useful parameters for quality assurance of established formulations, gaging relative degree of dispersion, and estimating the color value of colorants from different batches, sources, or grades.

6. Apparatus

6.1 Laboratory Balance, sensitive to at least 1.0 mg, preferably 0.1 mg.

6.2 Spatulas, (2) with flexible blades 80 to 120 mm in length (for weighing and mixing).

6.3 Mixing Surface, such as a glass or similar slab fixed to a work bench.

6.4 Putty Knife, with an 80–120 mm wide blade having a smooth straight edge (for use as a drawdown blade).

6.5 Standard Daylight, preferably a D50 light source conforming to ANSI Standard PH 2.30.

6.6 Reflectance Measuring Instrument, (for instrumental evaluation). Unless otherwise agreed upon, the instrument shall be a spectrophotometer with hemispherical (integrating-sphere) geometry conforming to Test Method E 1331, a spectrophotometer with bidirectional (45/0 or 0/45) geometry conforming to Test Method E 1349, or a tristimulus (filter) colorimeter with either geometry conforming to Test Method E 1347. Alternatively, a reflection densitometer conforming to ANSI Standard PH 2.17 and having a set of Status T or Status E filters (see 12.3.2), conforming to ANSI Standard 2.18 may be used for certain colors.

Note 2—The filter systems in typical densitometers are suitable only for use with black, white, and the three process colors (yellow, magenta and cyan). Instrumental evaluation of other colors requires a spectrophotometer or a colorimeter.

5 A spectrophotometrically controlled Universal White Bleach, containing by weight 60 % zinc oxide and 40 % hydrogenated methyl abietate is available from National Printing Ink Research Institute (NPIRI), Lehigh University, 7 Asa Drive, Bethlehem, PA 18015-3192.
8.2 Carefully select a dispersed sample that is free of skin and other contamination and representative of the lot being evaluated. Transfer to a clean container, protect with skin paper, close and seal.

9. Evaluation of Masstone and Undertone for Relative Color

9.1 Using the bond paper with the black bar, place small portions of the standard and unknown dispersions close together, but not touching, in the center at one end of the sheet in the long dimension.

9.2 Place the blade of the drawdown knife behind the pastes and, using heavy pressure, draw down a thin film of the pastes in juxtaposition. When the middle of the black bar is reached, raise the blade slightly and draw down the remaining pastes in a layer sufficiently thick that the black bar is not visible. Remove excess material.

9.3 Immediately examine the drawdowns under the standard D 50 light or other agreed upon light source. Judge the hue, depth, cleanliness, transparency and other properties of the unknown dispersion relative to the standard dispersion. Record qualitative observations of the thin film over white paper as the relative undertone, the thin film over the black bar as the relative transparency, and the thick film as the relative masstone.

9.4 Repeat 9.1 and make a tight drawdown on a sheet of coated paper. Make an immediate visual judgment of the relative undertone. Include relative gloss and bronzing in the evaluation.

NOTE 4—When the consistencies of the standard and unknown dispersions are significantly different, the film thicknesses of the tight drawdowns may not be comparable. In such cases, judgments regarding relative undertone should be reserved until the tints are examined (see Note 8 in 11.6).

NOTE 5—If the hue or cleanliness of the test dispersion is significantly different from the standard dispersion, tinting strength cannot be tested by the procedures covered in this test method. A numerical assessment of such systems may be obtained by making color measurements according to Test Methods E 1331, E 1347, or E 1349 and calculating color differences by the 1976 CIELAB equations in accordance with Practice D 2244.

10. Preparation of Tints

10.1 Select a tinting base appropriate to the sample being tested (see 7.2). Examine the base for uniformity. If there are signs of separation or settling, stir thoroughly in container. If necessary, transfer the quantity required for testing to a slab and mix to ensure that the same composition of base will be used for both the standard and the unknown samples.

10.2 Tare or counterbalance a weighing substrate. Using guidelines suggested in Table 1, weigh out the desired amount of the standard dispersion. The quantity of specimen need not be exactly as listed in Table 1 but must be weighed to at least three significant figures. Divide the actual weight by the desired decimal concentration to obtain the total tint weight. The difference between the total weight and the specimen weight represents the weight of bleaching base to be added.

10.3 Gently mix the specimen and tinting base on the weighing substrate until the tint is uniform. Use a circular stirring motion, periodically scraping all material from the surface of the substrate. Do not use so much energy that further dispersion will result. If necessary, transfer all material to a glass slab and continue mixing with a gentle scraping and stirring motion until a uniform color with no specks or streaks is achieved. With a clean putty knife, push the tint to one side of the slab. Clean the putty knife and remainder of the slab.

NOTE 6—With flushes and other high viscosity dispersions, it is recommended that the tinting base be mixed into the specimen in small increments.

10.4 Repeat 10.2 and 10.3 with the unknown dispersion. Be sure the specimen concentration in the tint and the type of tinting base are identical to that used for the standard dispersion.

10.5 If there will be a delay in the evaluation process, transfer the tints to small clean containers and label appropriately. Always gently restir immediately before subsequent use in order to minimize problems of flooding or floating.

11. Procedure

11.1 Using separate ink knives, gently stir the standard and the test tints. Place a small quantity of each tint close together, but not touching, at one end of a small glass plate or other drawdown substrate. Hold the drawdown knife at a low angle (5 to 15° from horizontal) and, using light pressure, draw down the tints in juxtaposition. The two films must be in contact with each other, smooth, and sufficiently thick so as to mask any background.

11.2 Immediately examine the drawdowns under the standard light. If the two tints appear equal, record the tinting strength of the unknown as 100 %. If the tints are unequal in strength, estimate the strength difference between the stronger and weaker color either from experience or from instrumental measurements (see Eq 6 or Eq 7 in 13.2.2).

NOTE 7—With colored and black samples, the stronger tint will be darker. With white samples, the stronger tint will be lighter.

11.3 Weigh to three significant figures an aliquot of about 1 g (or a quantity representing about 10 to 20 %) of the stronger

<p>| TABLE 1 Suggested Tint Concentrations for Strength Testing of Printing Ink Dispersions |
|------------------------------------------|-----------------|-----------------|-----------------|</p>
<table>
<thead>
<tr>
<th>Type of Dispersion</th>
<th>Dispersion Concentration in Tint</th>
<th>Ratio Dispersion</th>
<th>Content of Tint, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flush or concentrate</td>
<td>0.01</td>
<td>1:99</td>
<td>0.05</td>
</tr>
<tr>
<td>Process color ink</td>
<td>0.02</td>
<td>1:49</td>
<td>0.10</td>
</tr>
<tr>
<td>Laked or low strength color</td>
<td>0.05</td>
<td>1:19</td>
<td>0.25</td>
</tr>
<tr>
<td>Titanium dioxide with lamp black base</td>
<td>0.85</td>
<td>6:1</td>
<td>4.25</td>
</tr>
<tr>
<td>with carbon black base</td>
<td>0.98</td>
<td>49:1</td>
<td>4.90</td>
</tr>
</tbody>
</table>

*In NPIRI Universal Bleaching Base except where noted. Figures are given as a guide. It is recommended that standard batches be checked first to establish tint concentrations that give proper lightness levels.

*Materials should be weighed to three significant figures. Increase weights by a factor contingent on the balance sensitivity.

Use double the quantity for instrumental tinting strength conducted by spectrophotometry and confirmed by aliquot reduction.

For white dispersions, weigh tinting base first.
tint. Multiply the exact weight by the estimated strength difference in decimal units; add tinting base accordingly. For example, for an estimated 10 % difference, add 0.10 g base/g aliquot of the stronger tint.

11.4 Gently mix the adjusted tint until uniform. Gently remix the original tint of the weaker dispersion, make a thick drawdown versus the adjusted tint as in 11.1, and examine as in 11.2.

11.5 If the drawdowns are still unequal, discard the adjusted tint. Weigh out a new aliquot of the stronger tint and add more or less tinting base than in 11.3.

11.6 Repeat 11.3 and 11.4 until the drawdowns show that the adjusted tint equals the strength of the lighter tint. When equivalency is obtained, record whether the standard or unknown tint was stronger, the weight of the final aliquot, and the weight of added tinting base.

**NOTE 8**—If there is a difference in color between the unknown and standard dispersions, a situation will result wherein, as dilution progresses, the darker tint will revert to the lighter tint without obtaining a match. In such cases, this method cannot be used (see Note 5).

11.7 Compute the strength of the unknown dispersion \( (u) \) as a percentage of a standard dispersion(s) as follows:

\[
TS_u \% = \frac{1 + (b/a)_{s}}{1 + (b/a)_{u}} \times 100
\]  

(1)

where:

\( TS_u \% \) = tinting strength of the unknown dispersion,
\( b \) = weight of extra tinting base added to an aliquot of the stronger tint to obtain equivalence, g, and
\( a \) = weight of the aliquot, g.

The term \( b/a \) represents the strength difference between the stronger and weaker colorant. For the weaker dispersion, \( b/a = 0 \) and drops out of Eq 1. When the unknown dispersion is stronger, Eq 1 reduces to:

\[
TS_u \% = 1 + (b/a)_{u} \times 100
\]  

(2)

When the standard dispersion is stronger, Eq 1 reduces to:

\[
TS_u \% = \frac{1}{1 + (b/a)_{s}} \times 100
\]  

(3)

**NOTE 9**—Tinting strength is always expressed as a decimal or a percentage of the unknown relative to the standard. The practice of expressing results as a strength difference may lead to erroneous calculations of the replacement concentration. See Eq 5 in 13.1.

11.8 Since replication of visual tinting strength tests inherently suffers from bias, prepare a confirming tint in accordance with the procedure given in 13.1.

**TEST METHOD B—TINTING STRENGTH BY INSTRUMENTAL EVALUATION**

12. Procedure

12.1 Set the instrument for the large area of view or illumination and standardize in accordance with Test Methods E 1331, E 1347, or E 1349, or, in the case of a densitometer, the manufacturer’s instructions. If it is the intent to make measurements directly on wet tints, it may be useful to protect the instrument with a material such as plastic wrap with the porthole cut out.

12.2 Gently remix the standard tint prepared in Section 10. Place a sufficient quantity on a small glass plate or other rigid surface so that the material is at least 30 to 35 mm in diameter and thick enough to mask any background. Alternatively, use a standard spacer (see 7.6) to prepare a thick sandwich between two microscope cover glasses.

12.3 Measure the reflectance factor in one of the following manners:

12.3.1 **Spectrophotometer:** Following the procedure given in Test Method E 1331 or E 1349, quickly mount the tint on the porthole of the spectrophotometer and, within or at 45 s, measure the reflectance factor between 420 and 680 nm. If hemispherical geometry is used, the specular component may be either included or excluded, as long as the same condition is consistently used. Make a minimum of two measurements, moving or rotating the specimen between runs. Record the spectral reflectance factor in decimal units at the wavelength of maximum absorption (minimum reflectance) and compute the mean.

12.3.2 **Densitometer:** If the tint involves black, white, or a process color, select the filter having the appropriate Status T or Status E response in accordance with ANSI Standard PH 2.18. The peak transmission of the visual response filter should be at 555 nm for blacks and whites; of the blue filter, at 460 nm (Status T) or 440 nm (Status E) for process yellows; of the green filter, at 530 nm for magentas; of the red filter, at 600 nm for cyans. Make measurements as in 12.3.1 at two or three different locations. If the readout is density, convert to the reflectance factor as follows:

\[ R = 10^{-D} \]

If the readout is percent reflectance, record in decimal units.

12.4 Alternatively, measure the CIE tristimulus values of the specimen on a spectrophotometer in accordance with Test Method E 1331 or Test Method E 1349 or on a tristimulus colorimeter in accordance with Test Method E 1347. Make the measurements as in 12.3.1. If hemispherical geometry is used, the specular component may be either included or excluded as long as the same condition is consistently used. The tristimulus values may be based on either 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, for example, \( X \) with blue colors, \( Y \) with reds, blacks, and whites, or \( Z \) with yellows.

12.5 Repeat 12.2 and 12.3 or 12.4 with the unknown tint.

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

\[
TS_u \% = \frac{[(1 - R_{so})^2/2R_{so}]}{[(1 - R_{uo})^2/2R_{uo}]} \times 100
\]  

(4)

where:

\( R_u \) = spectral reflectance factor, expressed as a decimal fraction, of an infinitely thick layer of material (at the wavelength of maximum absorption), or, by mutual agreement, an appropriate tristimulus value.

**NOTE 10**—If the tint represents a white pigment, use Eq 4 in the inverted form.

**NOTE 11**—The term \( [(1 - R_{uo})^2/2R_{uo}] \) 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. Therefore, if the pigmentation of the unknown dispersion is different from that in the standard dispersion, that is, the dispersions are metameric, Eq 4 no longer applies. The equation is also reported to work best when the reflectance factor or tristimulus value of the tints used is about 0.40 (range 0.20–0.55) and the tinting strength of the unknown is within 10 % of the standard.

12.7 If the tinting strength result for the unknown is not within 10 % of the standard, reduce an aliquot of the stronger tint by the procedure given in 13.2. Remeasure the adjusted tint and calculate a new tinting strength value (see section 13.2.5).13.2.4.2.

12.8 Prepare a confirming tint following 13.1 if the original tinting strength value was not within 10 % of the standard (or 13.2 if within 10 %).

13. Preparation of Confirming Tint

13.1 Replacement Concentration:

13.1.1 In this method of confirmation, a new tint of the unknown dispersion is prepared at a concentration calculated to match the standard tint.

13.1.2 Compute the replacement concentration, also called color value, as follows:

$$C_u = C_s/TS_u$$

(5)

where:

$C_u$ = concentration of the unknown dispersion required to match the standard dispersion.

$C_s$ = concentration of the standard dispersion in the original tint prepared in 10.2, and

$TS_u$ = tinting strength result for the known dispersion in decimal units.

13.1.3 Weigh out a quantity of the unknown dispersion similar to that employed in 10.2. Divide the actual weight by $C_u$ to obtain the total tint weight. The difference between the total and the specimen weight represents the weight of bleaching base to be added. Mix as in 10.3.

13.1.4 Gently remix the original tint of the standard dispersion. Make a thick drawdown of both tints as in 11.1. If the two tints match, the tinting strength result computed for the unknown dispersion is correct.

13.1.5 Failure of the two tints to match suggests a weighing error or inadequate mixing of the tints. Clean up and start over from Section 10.

13.1.6 Use Eq 5 to compute the color/money/value of a colorant from different sources or grades (optional). Multiply $C_u$ and $C_s$ by their respective unit costs. Add to each the costs of other components in a formulation, represented by $(1 - C_s)$ and $(1 - C_u)$. The lower total cost figure is the better value.

13.2 Reduction of the Stronger Tint:

13.2.1 In this method of confirmation, the instrumental tinting strength result is used to calculate the strength difference between the stronger and weaker dispersion. An aliquot of the stronger tint is reduced accordingly.

13.2.2 Compute the strength difference, $b/a$ by rearrangement of Eq 2 or Eq 3. If the test dispersion is the stronger, that is, $TS_u$ in decimal units is greater than 1.0 then:

$$(b/a)_u = TS_u - 1$$

(6)

13.2.3 Weigh out an aliquot of the stronger tint as in 11.3. Multiply the exact weight by $b/a$ to obtain the weight of bleaching base to be added. Mix the adjusted tint until uniform.

13.2.4 Evaluate the adjusted tint by either of the following procedures:

13.2.4.1 Visual Evaluation—Draw down the adjusted tint versus the original tint of the weaker color as in 11.1.4. Failure to obtain equivalency on the first cut may suggest a weighing error or the inapplicability of the Kubelka-Munk equation (Eq 4), or both. If necessary, reduce a new aliquot until equivalency is obtained. Calculate the correct tinting strength according to Eq 2 or Eq 3.

13.2.4.2 Instrumental Evaluation—Prepare a thick film of the adjusted tint, remeasure the reflectance, and calculate the adjusted tinting strength by Eq 4. If 100 %, the original tinting strength value is correct. If not 100 %, compute the correct tinting strength by multiplying the original tinting strength percentage by the adjusted tinting strength in decimal units.

14. Report

14.1 Report the following information:

14.1.1 The type and identification of the test dispersion, the reference standard dispersion, and the nature of the tinting base,

14.1.2 The results of the visual evaluation of the relative color difference (masstone and undertone) of the drawdowns on bond and coated paper,

14.1.3 The relative tinting strength and the method by which it was determined (visual or instrumental). If the instrumental method was used, the manufacturer and type of instrument, the geometry (including for hemispherical geometry whether the specular component was included or excluded) and, if used, the basis for the calculation of tristimulus values,

14.1.4 The method, if any, by which the tinting strength result was confirmed, and

14.1.5 Any deviation, by agreement or otherwise, from the procedures given in these test methods.

15. Precision and Bias

15.1 Precision:

15.1.1 An interlaboratory study of these test methods was conducted in which 3 sets of process color printing inks ranging in tinting strength from 75 to 85 % were tested by operators in 6 different laboratories. The tests were conducted as blind duplicates on each of 2 days. In addition to visually evaluated tinting strength, the same reductions were measured spectrophotometrically or densitometrically, or both. The estimated standard deviations and the degrees of freedom are given in Table 2. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:
15.1.1 Repeatability—Two results obtained by the same operator on different days should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

15.1.1.2 Reproducibility—Two results, each the mean of results obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

15.2 Bias—The tinting strength results obtained in the interlaboratory study of these test methods were higher than the formulated values by the amounts shown in Table 3.

### TABLE 2 Precision of Tinting Strength Determinations

<table>
<thead>
<tr>
<th>Method of Evaluation</th>
<th>Standard Deviation, %absolute</th>
<th>Degrees of Freedom</th>
<th>Maximum Allowable Difference, %absolute</th>
</tr>
</thead>
<tbody>
<tr>
<td>Visual</td>
<td>1.3</td>
<td>12</td>
<td>3.7</td>
</tr>
<tr>
<td>Spectrophotometer</td>
<td>1.8</td>
<td>8</td>
<td>5.0</td>
</tr>
<tr>
<td>Densitometer</td>
<td>3.6</td>
<td>10</td>
<td>10.1</td>
</tr>
<tr>
<td><strong>Reproducibility</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Visual</td>
<td>2.8</td>
<td>12</td>
<td>8.0</td>
</tr>
<tr>
<td>Spectrophotometer</td>
<td>1.7</td>
<td>8</td>
<td>4.7</td>
</tr>
<tr>
<td>Densitometer</td>
<td>4.8</td>
<td>12</td>
<td>13.4</td>
</tr>
</tbody>
</table>

### TABLE 3 Accuracy of Tinting Strength Results

<table>
<thead>
<tr>
<th>Method of Evaluation</th>
<th>Difference Between Overall Mean Test Results and Formulated Tinting Strength, %absolute</th>
</tr>
</thead>
<tbody>
<tr>
<td>Visual</td>
<td>+0.73</td>
</tr>
<tr>
<td>Spectrophotometer**</td>
<td>+3.87</td>
</tr>
<tr>
<td>Densitometer**</td>
<td>+1.90</td>
</tr>
</tbody>
</table>

**If the unknowns had been stronger than the standard, the plus signs would be minus signs.

**Values are based on the original tints, not on adjusted tints as recommended in 12.6.

16. Keywords

16.1 colorimeter; densitometer; Kubelka-Munk equation; pigment dispersions; printing inks; relative tinting strength; spectrophotometer

### SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D 2066 – 97) that may impact the use of this standard.

(1) Addition of part of speech in Section 3.
(2) Deletion of White Bleach 95 from footnote 6.
(3) Addition of range to Note 11.

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