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Standard Test Methods for Relative Tinting Strength of Paste-Type Printing Ink Dispersions[1]

This standard is issued under the fixed designation D2066; 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 These test methods cover procedures for determining the relative tinting strength of paste-type printing ink dispersions by visual or instrumental evaluation of tints prepared by manual or automated mixing.

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 D387, D2745, D4838, and D6531.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D16 Terminology for Paint, Related Coatings, Materials, and Applications

D387 Test Method for Color and Strength of Chromatic Pigments with a Mechanical Muller

D2244 Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

D2745 Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements

D4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints

D6531 Test Method for Relative Tinting Strength of Aqueous Ink Systems by Instrumental Measurement

E284 Terminology of Appearance

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1331 Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry

E1347 Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry

E1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional (45°:0° or 0°:45°) Geometry

2.2 *ANSI Standards:*[3]

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 D16 and E284.

3.2 *Definitions of Terms Specific to This Standard:*

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

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.56 on Printing Inks.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2.2 *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.

3.2.3 *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.

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 a 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 *FlackTek Speed Mixer*[4,5](optional, for automated mixing). Essential accessories include:

6.2.1 *Plastic Cup,* preferably Max 15, for mixing 10 to 15 g. A larger cup, such as Max 40, may be useful for mixing 20 or more g of tinting base prior to use.

6.2.2 *Cup Holder,* of a size appropriate to the cup used in 6.2.1.

6.2.3 *Thermometer,* small, reading close to room temperature, for measuring temperature of tints prepared on the FlackTek.

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

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

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

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

6.7 *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 E1331, a spectrophotometer with bidirectional (45/0 or 0/45) geometry conforming to Test Method E1349, or a tristimulus (filter) colorimeter with either geometry conforming to Test Method E1347. Alternatively, a reflection densitometer conforming to ANSI Standard PH 2.17 and having a set of Status T or Status E filters[5,6] (see 12.3.2), conforming to ANSI Standard PH 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.

7. Materials

7.1 *Reference (Standard) Dispersion,* having the same pigmentation and consistency as the test (unknown) dispersion.

7.2 *Tinting Base,* as agreed upon between the producer and user, consisting of a suitable pigment well dispersed in a vehicle that is compatible with the vehicle in the test dispersion. The consistency of the base should not be appreciably lower than that of the test dispersion. Driers are not generally used because they may affect the color of the base and corresponding tints.

7.2.1 *White Base,*[5,7]for testing colored and black dispersions. A suitable white base may contain by weight 30 to 60 % of either zinc oxide or titanium dioxide and 40 to 70 % vehicle.

7.2.2 *Black Base,* for testing white dispersions. A suitable black base may contain by weight 4 % black pigment (preferably non-flocculating), 43 % precipitated calcium carbonate, and 53 % vehicle. Alternatively, a neutral black nondrying printing ink such as a news ink.

7.2.3 *Dark Blue Base* (optional), for visual testing of white dispersions. A suitable dark blue base may contain by weight 42 % ultramarine blue, 18 % precipitated calcium carbonate, and 40 % vehicle.

[4] The sole source of supply of the apparatus known to the committee at this time is FlackTek Inc., 1708 Highway 11, Building G, Landrum, SC 29356, http://www.speedmixer.com/.

[5] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] Status T filters are available in the USA, Status E in other countries. The major difference is in the peak transmission of the blue filter.

[7] The sole source of supply of the spectrophotochemically controlled NPIRI Bleach White X-1025 known to the committee at this time is Colorcon, No-Tox Products, 171 New Britain Boulevard, Chalfont, PA 18914–1833, http://www.colorcon.com/no-tox/index.html.

7.2.4 *Light Blue Base* (optional), for visual testing of yellow dispersions. A suitable light blue base may contain by weight 1 % phthalocyanine blue dispersion and 99 % white base.

NOTE 3—Mixtures of a light blue base with yellow samples produce green tints, differences between which are more easily detected by eye than are mixtures of white and yellow. However, false results may be obtained. The use of a blue base is not recommended for visual tests on greenish-yellow colorants and is not permitted for instrumental evaluation of any yellow colorant.

7.3 *Weighing Substrate* (for manual mixing), nonabsorbent, such as skin paper or small glass plates ca 75 to 100 mm square.

7.4 *Drawdown Substrates,* one consisting of white bond paper at least 50-mm wide and 150-mm long with a black bar at least 20-mm wide imprinted across the short dimension about half way down the length of the sheet, and a second one consisting of white coated paper.

7.5 *Microscope Cover Glasses* (for instrumental measurements), made of fine optical glass, 50 by 45 mm, 0.13 to 0.17 mm thick.

7.6 *Standard Spacer* (for instrumental measurements), such as cardstock the same size as the cover glass described in 7.5, about 1-mm thick, with a 35-mm diameter hole.

8. Sampling

8.1 These test methods do not include a method for preparation of dispersions. If colorants from different batches or sources are being evaluated, it is important that the standard and unknown samples be dispersed either in the identical manner or to the maximum degree, as agreed upon between the producer and the user.

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 D50 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 hue 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 E1331, E1347, or E1349 and calculating color differences by the 1976 CIELAB equations in accordance with Practice D2244.

10. Preparation of Tints

10.1 *Manual Mixing:*

10.1.1 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.

10.1.2 Tare or counterbalance a weighing substrate. Using guidelines suggested in Table 1, prepare 5 g of the tint if evaluation is to be visual, 10 g if evaluation is to be instrumental; weigh out the desired amount of the standard dispersion and the tinting base by one of the following methods.

10.1.2.1 *Weighing Method 1*—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. For example, 10 g of a 1 % tint is specified and the weight of the specimen is 0.1122 g. Dividing that quantity by 0.1 gives 11.22 g. This is the total weight of the tint. Add bleaching base accordingly.

10.1.2.2 *Weighing Method 2*—The weight of specimen and tinting base must both be exact to ±0.001 g. For example, for 10 g of a 1 % tint, the weight of specimen must be exactly 0.1

TABLE 1 Suggested Tint Concentrations for Strength Testing of Printing Ink Dispersions[A]

Type of Dispersion	Dispersion Concentration in Tint	Ratio Dispersion	Content of Tint,[B,C] g		
			Dispersion	Tinting Base	Total
Flush or concentrate	0.005	1:199	0.05	9.95	10.0
Process color ink	0.01	1:99	0.10	9.90	10.0
Laked or low strength color	0.05	1:19	0.50	9.50	10.0
Titanium dioxide					
with lamp black base	0.85	6:1	8.50	1.50[D]	10.0
with carbon black base	0.98	49:1	8.80	0.20[D]	10.0

[A] In NPIRI Bleach White X-1025 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, that is, 20 to 55 % reflectance for instrumental evaluation.
[B] Materials should be weighed to three significant figures. Increase weights by a factor contingent on the balance sensitivity.
[C] Half the quantity may be used if evaluation is visual only.
[D] For white dispersions, weigh tinting base first.

g ± 0.001 g, and the weight of tinting base must be exactly 9.9 g ± 0.001 g. For a nominal 1 % tint, the weight of tinting base may be 10 g ± 0.001 g.

10.1.3 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.

10.1.4 Repeat 10.1.2 and 10.1.3 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.

10.1.5 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.

10.2 *Automated Mixing on the FlackTek:*

10.2.1 Select a tinting base appropriate to the sample being tested (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 preparing two tints (20+ g) to a Max 40 cup and run on the FlackTek at 3000 RPM for one or two minutes.

10.2.2 Tare or counterbalance the FlackTek Max 15 plastic cup. Using guidelines in Table 1, weigh out the standard and tinting base by Weighing Method 1 (10.1.2.1) or Method 2 (10.1.2.2). When weighing the specimen, try to place it in the center of the cup. When adding the tinting base, make sure no material adheres to the side of the cup above the ridge line. Total tint weight may not exceed 12 g.

10.2.3 Fit the cup securely with the lid, label appropriately and place in the holder on the mixing machine. Set the speed for 3000 RPM and the timer for two minutes. Turn on the mixer.

10.2.4 At the end of mixing, remove the cup and examine the tint for unmixed tinting base or pigment streaks, or both. If not completely mixed, return to the mixer for another minute, or until complete mixing is achieved.

10.2.5 At the end of mixing, remove the cup. Insert a clean thermometer into the tint and record the temperature to the nearest degree.

10.2.6 Repeat 10.2.2 – 10.2.5 with the test specimen. Use precautions as prescribed in 10.1.4 and 10.1.5.

10.2.7 Make sure that the standard and unknown tints are both at room temperature prior to evaluation.

TEST METHOD A—TINTING STRENGTH BY VISUAL EVALUATION

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 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:

$$TS_u,\ \% = \frac{1+(b/a)_u}{1+(b/a)_s} \times 100 \qquad (1)$$

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