Designation: D 2066 – 97

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

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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.[2,3]

1.3 *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 Relating to Paint, Varnish, Lacquer, and Related Products[2]

D 387 Test Method for Color and Strength of Colored Pigments with a Mechanical Muller[2]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[2]

D 2745 Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements[2]

D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints[3]

E 284 Terminology of Appearance[2]

E 1331 Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry[2]

E 1347 Test Method for Color and Color-Difference Measurement by Tristimulus (Filter) Colorimetry[2]

E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry[2]

2.2 *ANSI Standards:*

PH 2.17 Geometric Conditions for Reflection Density[4]

PH 2.18 Spectral Conditions for the Measurement of Optical Density[4]

PH 2.30 Viewing Conditions for Graphic Arts and Photography—Color Prints, Transparencies and Photomechanical Reproductions[4]

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*—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)*—the color of a material that is thick enough to mask any background.

3.2.3 *undertone (or undercolor)*—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.

[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D1.56 on Printing Inks.

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

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

[4] Available from American National Standards Institute, 13th Floor, 11 W42nd St., New York, NY 10036.

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[5] (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.

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,*[6] for testing colored and black dispersions. A suitable white base may contain by weight 30–60 % of either zinc oxide or titanium dioxide and 40–70 % 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.

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*, 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* (optional, for instrumental measurements), made of finest optical glass, 50 by 45 mm, 0.13- to 0.17-mm thick.

7.6 *Standard Spacer* (optional, 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.

[5] 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.

[6] Spectrophotometrically controlled white bleaches are available from National Printing Ink Research Institute (NPIRI), Lehigh University, Sinclair Laboratory, 7 Asa Drive, Bethlehem, PA 18015-3192. Universial White Bleach contains by weight 60 % zinc oxide and 40 % hydrogenated methyl abietate plasticizer. White Bleach 95 contains by weight 30 % titanium dioxide and 70 % vehicle consisting of oxidizing oils, petroleum distillates and resins.

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 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 E 1331, E 1347, or E 1349 and calculating color differences by the 1976 CIELAB equations in accordance with Test Method 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.

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	0.01	1:99	0.05	4.95	5.0
Process color ink	0.02	1:49	0.10	4.90	5.0
Laked or low strength color	0.05	1:19	0.25	4.75	5.0
Titanium dioxide					
with lamp black base	0.85	6:1	4.25	0.75[D]	5.0
with carbon black base	0.98	49:1	4.90	0.10[D]	5.0

[A] 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.

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

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

[D] For white dispersions, weigh tinting base first.

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.

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.