Designation: D387 – 00 (Reapproved 2017)

Standard Test Method for Color and Strength of Chromatic Pigments with a Mechanical Muller[1]

This standard is issued under the fixed designation D387; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method is intended to be used to compare the color and strength of a pigment under test with a reference standard of the same type and grade.

1.2 This test method does not apply to white pigments.

NOTE 1—Test Method D3022 is similar to this test method, but it utilizes a miniature sandmill rather than a mechanical muller, to disperse the chromatic pigment.

NOTE 2—Test Method D332 and Test Method D2745 are similar to this test method, but they are intended for use with white pigments, rather than chromatic pigments.

1.3 The values stated in SI units are the preferred unit of measurement. 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.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D332 Test Method for Relative Tinting Strength of White Pigments by Visual Observation

D1729 Practice for Visual Appraisal of Colors and Color Differences of Diffusely-Illuminated Opaque Materials

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

D3022 Test Method for Color and Strength of Color Pigments by Use of a Miniature Sandmill

D3964 Practice for Selection of Coating Specimens for Appearance Measurements

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

E284 Terminology of Appearance

3. Terminology

3.1 Definitions of appearance terms used in this test method may be found in Terminology E284.

4. Summary of Test Method

4.1 Pigments are dispersed in a suitable vehicle with a mechanical muller. Test and standard pigments are treated identically. Opaque drawdowns are made from the dispersions and compared, either visually or instrumentally, for color and strength differences.

5. Significance and Use

5.1 Color and tinting strength are the most important properties of a color pigment. This test method provides a means of testing these properties for quality control.

5.2 This test method is intended as a referee method so that such matters as the vehicle for preparing the dispersions and the white for making tints have been suggested. However, other vehicles and whites may be suitable for quality control purposes, and changes in this test method are allowed by agreement between the parties to a test.

5.3 It is assumed that the most exact comparison of mass color and tinting strength occurs when the pigment is completely dispersed. By following the procedure described in Annex A1, the conditions for achieving the maximum practical degree of dispersion with a mechanical muller may be determined. Color and strength tests should be carried out under these conditions.

5.4 The results obtained with a mechanical muller do not necessarily correlate directly with an industrial situation where different dispersing conditions exist. However, dispersion with

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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.

a mechanical muller is a quick and inexpensive way of testing the color and strength of a pigment for routine quality control.

6. Apparatus

6.1 *Balances*—(*1*) A balance sensitive to 10 mg and (*2*) an analytical balance sensitive to 1.0 mg.

6.2 *Muller, Mechanical,* equipped with ground-glass plates to which a variable but known force may be added in 220-N (50-lbf) increments. The driven glass plate shall have a speed of rotation of between 70 and 120 r/min and the apparatus shall have an arrangement for pre-setting the number of revolutions in multiples of 50.

6.3 *Rubbing Surfaces*—The rubbing surfaces of the ground glass plates shall be kept sharp by removing them from the muller and grinding them face-to-face with No. 303 optical emery, or its equivalent, and water.

6.4 *Small Glass Slab* or other nonabsorbent material, suitable for weighing and mixing pigment pastes.

6.5 *Spatula*—A flexible spatula having a 75 to 150-mm (3 to 6-in.) blade.

6.6 *Paper Charts,* white with a black band and a surface impervious to paint liquids.

6.7 *Film Applicator,* at least 75 mm (3 in.) wide with a clearance of 100 µm (4 mils) to produce wet films about 50 µm (2 mils) thick.

6.8 *Color-Measuring Instrument,* meeting the requirements of Practice D2244.

7. Materials

7.1 *Reference Standard*—A standard pigment of the same type and grade as the pigment to be tested, as agreed upon between the purchaser and the seller.

7.2 *Vehicle*—A solvent-free vehicle, such as No. 1 lithographic varnish, with 0.8 % each of cobalt and manganese driers (6 % types).

7.3 *White Tinting Paste*—A white paint compatible with the dispersion vehicle, such as 57 parts of rutile titanium dioxide dispersed in 43 parts of the vehicle described in 7.2.

NOTE 3—Because the choice of vehicle and white tinting pigment may affect the results, they should be agreed upon between the purchaser and the seller.

8. Hazards

8.1 Some pigments are potentially toxic and therefore should be handled with care. Obtain specific precautions from the manufacturer or supplier.

8.2 Many solvents and paint vehicles present explosion, fire, and toxicity hazards, so they should accordingly be handled with care. Again, obtain specific precautions from the manufacturer or supplier.

9. Dispersing Conditions

9.1 The conditions for dispersing the pigment on the mechanical muller should be such that the maximum tinting strength is developed. For each pigment and each dispersing vehicle the development of tinting strength by the mechanical muller is influenced by the force applied, the number of revolutions, the mass of the pigment, and the mass of the vehicle. The conditions for obtaining the maximum tinting strength with the mechanical muller can be determined by following the procedure in Annex A1.

9.2 If these conditions are known for a particular pigment with a particular vehicle, or if the purchaser and seller agree upon a particular set of conditions, there is no need to carry out the procedure in Annex A1.

10. Dispersion Procedure

10.1 Decide, by agreement or by experimentation, as discussed in Section 9, the following dispersing conditions:

10.1.1 Force applied to the muller plates;

10.1.2 Number of revolutions;

10.1.3 Mass of the pigment; and

10.1.4 Mass of the vehicle.

10.2 Applying these decisions, prepare a dispersion of the reference standard pigment. Weigh onto a glass slab to within 2 mg, the appropriate quantities of the standard pigment and the dispersing vehicle. Mix the pigment and vehicle together thoroughly with the spatula and transfer the mixture to the lower plate of the muller. Spread the mixture in a path approximately 100 mm wide and halfway between the center and rim of the lower plate, and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the mulling stages of 50 revolutions; after each stage collect the paste from both plates with the spatula and spread it around the 100-mm path on the lower plate, wiping the spatula on the upper plate as before. When the mulling has been carried out for the prescribed number of revolutions, collect the paste and store it. Clean the glass slab, the muller plates, and the spatula, and repeat the procedure with exactly the same quantities of the test sample and vehicle. Collect the paste from this sample and store it. Clean the glass slab, the muller plates, and the spatula.

NOTE 4—The most common sources of error in this procedure are inaccurate weighing, incomplete transfer of the pigment and vehicle mixture, and contamination of the plates by previous samples.

11. Masstone Color Procedure

11.1 Draw down a portion of the test and standard pastes in juxtaposition on a paper chart over a vacuum-drawdown plate or other suitable plane surface with the film applicator. Make sure that the coating is opaque.

11.2 Immediately compare the colors visually while still wet, using Practice D1729, and record the results. Set the drawdowns aside in a dust-free area to dry. When dry, repeat the visual color difference evaluation and record the results. See Practice D3964.

11.3 If desired, evaluate the color difference instrumentally using Practice D2244, and report the color difference in units as agreed upon between the purchaser and seller.

NOTE 5—Wet color difference evaluations may not agree with dry color difference evaluations because of such phenomena as flooding and flocculation. In the case of a difference between the wet and dry

evaluations, the purchaser and the seller should agree upon which condition is the standard.

NOTE 6—Color difference measurements of wet paints may require a special adapter to protect the instrument from fouling. Because color difference-measuring instruments differ widely in their design, the user may have to develop his own adapter.

12. Tint Color Procedure

12.1 Determine by calculation the amount of white pigment paste that must be added to 0.5 g of the color pigment paste so that the mixture contains 1 part of dry color pigment to 10 parts of dry white pigment. For stronger or weaker pigments this ratio may be adjusted accordingly, for example, 1:20 or 1:5, respectively.

12.2 Weigh 500 ± 2 mg of the standard color pigment paste onto a glass slab. Then weigh the amount of white pigment paste determined in 12.1, and place it next to the color pigment paste on the glass slab. Thoroughly mix the two pastes together with the spatula until a uniform color is observed.

12.3 Prepare a tint mixture of the test color pigment paste and the white pigment paste on a separate glass slab by the procedure described in 12.2.

12.4 Draw a portion of the test and standard tint pastes down in juxtaposition on a paper chart as in 11.1. Evaluate the color difference visually as in 11.2 and, if desired, instrumentally as in 11.3. Clean the spatula blade and glass slabs.

13. Calculation of Tinting Strength

13.1 If the colors of the test tint paste and the standard tint paste are visually the same, the tinting strength of the test pigment is equal to that of the standard pigment, and the relative tinting strength of the test pigment is 100 %. However, if the test and standard colors are not the same, the difference may be due to either tinting strength or hue (shade).

13.2 To determine the relative tinting strength of the test pigment, repeat the operations of Section 12, but this time use an amount of the test pigment paste that is estimated to give the closest color match to the standard pigment paste. Repeat this procedure until satisfied that the closest color match has been obtained. At this point any residual color difference between the test and the standard pigments is attributed to a shade difference, rather than a strength difference. Note and record this shade difference.

13.3 Calculate the relative tinting strength of the test pigment by dividing the mass of the standard paste by the mass of the test paste used to obtain the closest color match; multiply by 100 to express the result in percent.

13.4 If desired, the relative tinting strength of the sample pigment can be calculated from instrumental measurements using the following equation:

$$\mathrm{TS} = \left[(1 - R_\infty)^2/2R_\infty\right]_u / \left[(1 - R_\infty)^2/2R_\infty\right]_s (T)$$

where:

TS = tinting strength of test pigment,
R_∞ = measured reflectant factor (as a decimal),
T = assigned tinting strength of standard, usually 100 %, and subscripts “u” and “s” refer to the test and standard pigments, respectively.

13.4.1 To determine R_∞, follow instructions in the first paragraph of the Summary section and the second paragraph of the Specimen Preparation section of Test Method D4838.

14. Report

14.1 Report the following information:

14.1.1 Type and identification of the test pigment, reference standard pigment, white tinting pigment, and dispersing vehicle.

14.1.2 Mass ratio of pigment to vehicle, and for tints, mass ratio of color pigment mass to white pigment.

14.1.3 Manufacturer and model number of the mechanical muller employed.

14.1.4 Total force applied to the muller plates and total number of revolutions.

14.1.5 Results of the visual evaluation of the color difference (masstone and tint) in accordance with Practice D1729.

14.1.6 If an instrument was used to evaluate the color difference, the results of the instrumental evaluation in accordance with Practice D2244.

14.1.7 Relative tinting strength and method by which it was determined (visual or instrumental). Also, for the instrumental method, the parameter used as the measure of *Rin*.

14.1.8 Any deviation, by agreement or otherwise, from the test procedure just described.

15. Precision

15.1 *Precision*—The precision of this test method depends on several factors such as the type of pigment, the level of tinting, and the magnitude and direction of the color difference. This point is illustrated by the results in Table 1, which contains the between-laboratories standard deviations obtained in an interlaboratory study involving five different laboratories and four different pigments. The dispersing conditions used to obtain these results are listed in Table 2.

15.2 Table 3 lists the maximum acceptable differences, calculated at the 95 % confidence level from the results in Table 1.

TABLE 1 Between-Laboratories Standard Deviations for Various Color Difference[A] and Tinting Strength[B] Parameters

Pigment Type	Masstone Color				Tint Color				Tinting Strength			
	Δ*a*	Δ*b*	Δ*L*	Δ*E*	Δ*a*	Δ*b*	Δ*L*	Δ*E*	*Y*	T	R	V
Yellow Iron Oxide	0.10	0.46	0.22	0.42	0.13	0.20	0.19	0.08	2.0	2.2	3.0	0.7
BON Red	0.12	0.37	0.20	0.28	0.29	0.31	0.07	0.24	0.7	1.8	1.1	2.5
Molybdate Orange	0.09	0.14	0.06	0.11	0.11	0.12	0.05	0.14	0.5	0.8	0.6	1.0
Phthalocyanine Green[C]	0.29	0.65	1.43	0.51	0.25	0.07	0.13	0.21	1.6	1.8	2.6	1.8

[A] Color difference values were calculated with the CIE 1976 L*a*b* (CIELAB) equation.
[B] Tinting strengths were calculated four different ways with the equation in 13.2: *Y*, based on *Y* tristimulus value; *T*, based on lowest tristimulus value; *R*, based on lowest reflectance factor between 420 nm and 680 nm; and *V*, based on visual observation.
[C] Severe bronzing occurred with the masstone of this pigment (more in the batch than the standard), which probably affected the color difference measurements made with different types of instruments.

TABLE 2 Dispersing Conditions Used in Interlaboratory Study

Pigment type	Pthalocyanine Green	Yellow Iron Oxide	BON Red	Molybdate Orange
Force applied to the muller plates, lb (N)	100 (440)	100 (440)	100 (440)	100 (440)
Total number of revolutions	400 (8 × 50)	100 (2 × 50)	200 (4 × 50)	100 (2 × 50)
Mass of color pigment, g	0.75	1.0	0.6	2.0
Mass of dispersing vehicle, g	1.8	1.7	1.4	1.0

TABLE 3 Maximum Acceptable Differences for Various Color Difference[A] and Tinting Strength[B] Parameters

Pigment Type	Masstone Color				Tint Color				Tinting Strength			
	Δ*a*	Δ*b*	Δ*L*	Δ*E*	Δ*a*	Δ*b*	Δ*L*	Δ*E*	*Y*	T	R	V
Yellow Iron Oxide	0.28	1.30	0.62	1.19	0.37	0.57	0.54	0.23	5.7	6.2	8.5	2.0
BON Red	0.34	1.05	0.57	0.79	0.82	0.88	0.20	0.68	2.0	5.1	3.1	7.1
Molybdate Orange	0.25	0.40	0.17	0.31	0.31	0.34	0.14	0.40	1.4	2.3	1.7	2.8
Pthalocyanine Green[C]	0.82	1.84	4.04	1.44	0.71	0.20	0.37	0.59	4.5	5.1	7.4	5.1

[A] Color difference values were calculated with the CIE 1976 L*a*b* (CIELAB) equation.
[B] Tinting strengths were calculated four different ways with the equation in 13.2: *Y*, based on *Y* tristimulus value; *T*, based on lowest tristimulus value; *R*, based on lowest reflectance factor between 420 and 680 nm; and *V*, based on visual observation.
[C] Severe bronzing occurred with the masstone of this pigment (more in the batch than the standard), which probably affected the color difference measurements made with different types of instruments.

16. Keywords

16.1 chromatic pigment; color; muller

ANNEX

(Mandatory Information)

A1. DISPERSING CONDITIONS FOR MAXIMUM TINTING STRENGTH

A1.1 The following describes a test method for determining the conditions for achieving the maximum level of tinting strength with the mechanical muller.

A1.2 Determine the appropriate ratio of color pigment to dispersing vehicle by performing the following operations: Tare off the weight of a glass slab on a balance. Weigh 1.00 ± 0.01 g of the standard pigment on to the glass slab. Add dispersing vehicle to the pigment in small amounts and mix them together with the spatula. Keep adding the vehicle and mixing the paste until the pigment is completely wetted and a workable paste is obtained. At this point the consistency of the paste should be such that a dab of the paste will drop from the spatula when it is gently tapped with the finger. Weigh the paste, and subtract the mass of the pigment to determine the mass of the vehicle. Calculate the pigment to vehicle mass ratio. Repeat the operations described above for the test pigment.

A1.3 Determine the appropriate amount of pigment to use by estimating, to within 0.2 mL, the volume of that paste prepared in A1.2 that has the smallest pigment-to-vehicle mass ratio. Calculate the masses of pigment and vehicle needed to give a paste having a volume of about 2.0 mL. Round the amount of pigment down and the amount of vehicle up to the nearest 0.1 g.

A1.4 Apply 100 lbf (440 N) to the muller plates and prepare a tint of the standard pigment in accordance with the procedure in Sections 10 and 12. Use the amounts of the color pigment and dispersing vehicle determined in A1.3 and mull the paste for 100 revolutions in two stages of 50 revolutions each.

A1.5 Prepare three more specimens from the same sample following the procedure described in A1.4, but mull these specimens, in stages of 50 revolutions, for 200, 300, and 400 revolutions, respectively.

A1.6 Compare each of the four specimens, one to the other, for tinting strength using one of the methods described in Section 13, and determine the minimum number of revolutions necessary to achieve full tinting strength. If the tinting strength is still developing after 400 revolutions, repeat A1.4 – A1.6 with 50 lbf (220 N) more force on the mechanical muller plates.

A1.7 Record the appropriate amounts of pigment and vehicle (by A1.3), the force applied to the mechanical muller plates and the minimum number of revolutions required for maximum tinting strength.

A1.8 Table 2 lists, as examples, the dispersing conditions used in the interlaboratory study that established the precision given in Table 1 and Table 3. The vehicle used was No. 1 lithographic varnish with 0.8 % each of cobalt and manganese driers (6 % types).