Standard Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements¹

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1. Scope

- 1.1 This test method describes the procedure for determining the relative tinting strength of white pigments by reflectance measurements of black tints.
- 1.2 This test method is applicable only for comparing the test pigment with a reference standard of the same type and grade.

Note 1—Test Method D 332 describes a procedure for visual assessment of blue-tinted samples.

- 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 332 Test Method for Relative Tinting Strength of White Pigments by Visual Observation²
- D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates²

3. Summary of Test Method

3.1 Pigment is dispersed in a vehicle and let down with additional vehicle that has been tinted. Dispersion and letdown are accomplished with a mechanical muller. Both the test and standard pigments are treated identically. Opaque drawdowns are made of the paint and the *Y* tristimulus values (green-filter reflectance) of the wet films are measured. The relative tinting strength of the test pigment is calculated directly from the reflectance values.

4. Significance and Use

- 4.1 Tinting strength is one of the most important properties of a white pigment. This test method provides a means of testing this property for quality control.
- 4.2 This test method is a referee method, and the vehicle for preparing the dispersion and the black for tinting are suggested, but others may be used provided both the purchaser and the seller agree to the changes.
- 4.3 The results obtained with a muller do not necessarily agree with industrial situations where different dispersing conditions exist. However, dispersing with a muller is a fast, relatively inexpensive way of testing tinting strength for routine quality control.

5. Apparatus and Materials

- 5.1 *Muller*, automatic, equipped with a weight that exerts a permanent 220-N (50-lbf) and an additional weight exerting a 50-lbf making a total of 445-N (100-lbf). The two glass plates shall be kept sharp by removing from the machine and grinding them face-to-face with No. 303 optical emery or equivalent, and water.
- 5.2 *Spatula*—A flexible spatula having a chromium-plated or plastic blade 75 to 150 mm (3 to 6 in.) long, and another with a 75 mm (3-in.) tapered blade.
- 5.3 *Balances*—(1) A balance sensitive to 20 mg and (2) an analytical balance sensitive to 0.4 mg.
- 5.4 *Vehicle*—Because the choice of vehicle may affect results, a solvent-free vehicle (excluding refined or low-bodied linseed oil) should be agreed upon by the purchaser and the seller.
- 5.5 *Tinting Black*—A lamp black predispersed in a vehicle similar in nature to the test vehicle. Tinting black should be agreed upon between the purchaser and the seller.
- 5.6 *Chart*—Either gray or white lacquered charts cut to a convenient size.
- 5.7 *Color Measuring Instruments*, meeting the requirements of Test Method D 2244.
- 5.8 Film Applicator—50 or 75 mm (2 or 3 in.) wide with a clearance of 150 μ m (6 mils) to produce wet films about 75 μ m (3 mils) thick. This applicator when used as suggested in the test method will produce an R_{∞} film.

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

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² Annual Book of ASTM Standards, Vol 06.01.

TABLE 1 Examples of Preparation of Black Letdown Vehicles

Pigment Type	Approximate Pigment Density	Pigment, g	Clear Vehicle, g	Black Letdown Vehicle, g	Approximate PVC of Final Paint, %	Predispersed Black, ^A g	Clear Vehicle, ^B g
Titanium dioxide:							
rutile	4.1	3.000	2.000	1.000	20	6.80	93.20
anatase	3.8	3.000	2.250	1.000	20		
Lithopone	4.3	5.000	1.220	1.000	35	2.25	250.00
Zinc oxide, leaded	5.9	5.000	2.460	1.000	20		
Zinc oxide	5.6	5.000	2.660	1.000	20		
White lead	6.8	3.000	0.810	1.000	20		

^ADaniel Products Co. Lampblack F is used for this example.

6. Standard

6.1 A standard white pigment of the same type as the sample to be tested, as agreed upon by the purchaser and the seller.

7. Procedure

- 7.1 Place two concentric circles under the base plate of the muller so that they can be clearly seen through the plate. The circles can be drawn on a paper and inserted under the plate, or they can be drawn directly on the underside of the bottom plate. The inner circle should be 63 mm ($2\frac{1}{2}$ in.) in diameter and the outer circle 114 mm ($4\frac{1}{2}$ in.) in diameter.
- 7.2 Make a preliminary test by dispersing standard white pigment in a clear vehicle at the pigment volume concentration (PVC) given in Table 1 and adding tinting black to determine the amount of black required to provide a reflectivity of 0.42 ± 0.03 . Prepare a "black letdown vehicle" by adding tinting black to clear vehicle so that $1.000~\rm g$ of the black letdown vehicle will contain the same amount of black that provided this reflectivity. Three examples are given in Table 1.
- 7.3 Weigh the pigment to the nearest g with the analytical balance in accordance with Table 1 and transfer to the lower plate of the muller. Add clear vehicle to the pigment in accordance with Table 1 (Note 2). Mix with a spatula until a paste is obtained. "Pick up" the paste with the spatula and spread it within the area between the two concentric circles on the plate. Close the muller, mull 25 times, and collect the paste. Thoroughly mix the black letdown vehicle in its container. Add 1.000 g of black letdown vehicle as specified in Table 1 to the white paste and mix in thoroughly with a spatula. Again spread the paste within the two concentric circles, mull 25 times, pickup the paste, spread within the area, and mull a final 25 times more. Prepare a reference standard with each series of test pigments by this same procedure.

Note 2—An analytical balance is specified for referee-type of testing; for routine work, weighing to 20 mg has been found satisfactory as an alternative procedure. Among the methods of accurately weighing vehicles (either the clear or black letdown vehicles) are these two alternatives: (*I*) Place a small beaker of vehicle on the weighing pan of the balance. Remove and transfer vehicle employing the spatula ultimately intended for preparing a paste on the muller. Use the weigh-by-difference method. (2) Add the vehicle to a tared metal or plastic plate till the correct weight is reached; transfer to the muller by scraping off the plate with the spatula. If the vehicle tends to run, weigh both pigment and vehicle on the plate making a crater in the pile of pigment to contain the vehicle.

7.4 Standardize the color measuring instrument for measurement of the green filter reflectance (that is, *Y*-value) with, if necessary, a wet-film adapter in place. Adjust the color

instrument to the approximate expected reading. Draw down the sample paint on the chart (lacquered side) with the applicator blade (5.8). Place the film immediately on the reading port, cover the back of the chart with a flat, black panel (for example, black structural glass) to keep the chart flat and prevent exterior light from entering the color measuring instrument, and read the reflectance immediately (Note 3), recording the value. Clean the port of the colorimeter thoroughly with a rag. Draw down the standard paint and read immediately as before (Note 4). When the reflectance reading of the sample is greater than the standard, the sample is stronger in tinting strength. When it is lower it is weaker.

Note 3—Flooding will cause drifting in the reflectance reading of some films. When the color measuring instrument is already adjusted to the expected reading, an experienced operator can read the reflectance with high precision in a very short interval. Consequently the time lapse between draw-down and reflectance reading should be as short as possible.

Note 4—Although the method is primarily intended as a rapid method, it obviously can be adapted to measuring the tinting strength in a dry film of paint. For this an appropriate amount of cobalt drier is added to the black letdown vehicle during its preparation (see 7.2), and the films are allowed to dry as needed before making the reflectance measurements. It should be cautioned that tinting-strength values measured on dry films may differ from those measured on wet films.

8. Calculation of Tinting Strength

8.1 Calculate the tinting strength as follows:

$$TS = (T)[(1 - R_{\infty})^{2}/2R_{\infty}] / [(1 - R_{\infty})^{2}/2R_{\infty}]_{u} (T)$$

where:

TS = tinting strength of test pigment,

 R_{∞} = measured reflectance 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.

9. Report

- 9.1 Report the following information:
- 9.1.1 Tinting strength rounded to three significant figures,
- 9.1.2 Identification of the pigment, the reference standard, and the arbitrary assigned value, T, of the tinting strength for the reference standard. (Also identify the vehicle and colorant employed.), and
- 9.1.3 Name and model number of the color measuring instrument employed.

10. Precision

10.1 On the basis of an interlaboratory test of this test

^BA 50-50 (by weight) composite vehicle, Aroplaz 1271 and 1278, is the "vehicle" used for this example.



method in which seven laboratories tested four materials covering a broad range in tinting strength using five types of colorimeters, the standard deviation was found to be 1.16 within laboratories and 1.72 among laboratories. Based on this, the following criteria should be used for judging the acceptability of results at the 95% confidence level when the standard is assigned a value of 100:

10.1.1 Repeatability—Two results obtained by the same

operator on the same pigment should be considered suspect if they differ by more than 3.3 units.

10.1.2 *Reproducibility*—Two reported results, obtained by operators in different laboratories, should be considered suspect if they differ by more than 4.9 units.

11. Keywords

11.1 Muller; tinting strength; white pigments

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