Designation: D 3022 – 84 (Reapproved 1996)ε1

Standard Test Method for Color and Strength of Color Pigments by Use of a Miniature Sandmill[1]

This standard is issued under the fixed designation D 3022; 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.

ε[1] NOTE—Editorial changes and Keywords were added in December 1996.

1. Scope

1.1 This test method covers the determination, through the use of a miniature sandmill, of the color and strength of dry color pigments.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.* For specific hazard statements, see Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

C 778 Specification for Standard Sand[2]

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)[3]

D 523 Test Method for Specular Gloss[4]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[4]

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry[5]

E 308 Practice for Computing the Colors of Objects by Using the CIE System[4]

2.2 *Federal Specification:*[6]

TT-R-266 Resin, Alkyd; Solutions

2.3 ASTM Adjuncts:

Miniature sandmill[7]

3. Significance and Use

3.1 This test method is a way of testing the color and strength of pigments by use of a miniature sandmill. It correlates well with industrial practice and is used for routine quality control.

4. Apparatus

4.1 *Balance*, sensitive to 10 mg with a capacity in excess of 300 g.

4.2 *Miniature Sandmill*—A laboratory disperser equipped with a 1⅝-in. (41-mm) diameter fiber rotary disk impeller rotating at a constant 8000 r/min under varying load conditions. The shaft upon which the impeller is mounted shall be sufficiently balanced so no whip of the shaft is observed between 0 and 10 000 r/min.

4.3 *Cylinder*, 100-mL graduated.

4.4 *Beakers*, 200-mL tall-form, stainless steel, or polyethylene (approximately 60-mm inside diameter).

4.5 *Strainers*, paper cone, disposable, about 40 mesh.

4.6 *Fiber Disks*, 1⅝-in. (41-mm) diameter phenolic laminated, ¼ in. (6 mm) thick.[8]

4.7 *Paper Charts*, smooth, surface-coated, the surface of which should be impervious to paint liquids.[9]

4.8 *Film Applicator*, with an 8-mil (200-µm) clearance at least 3 in. (75 mm) wide.

4.9 *Color-Measuring Instruments*, as defined in Test Method E 97 or Practice E 308.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 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 04.01.

[3] *Annual Book of ASTM Standards*, Vol 06.04.

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

[5] Discontinued; see *1992 Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

[7] Drawings are available from ASTM Headquarters. Order Adjunct ADJD3022.

[8] The sole source of supply of the disks known to the committee at this time is Gardner/BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[9] The sole source of supply of the white and black charts known to the committee at this time is the Leneta Co., 15 Whitney Rd., Mahwah, NJ 07430. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

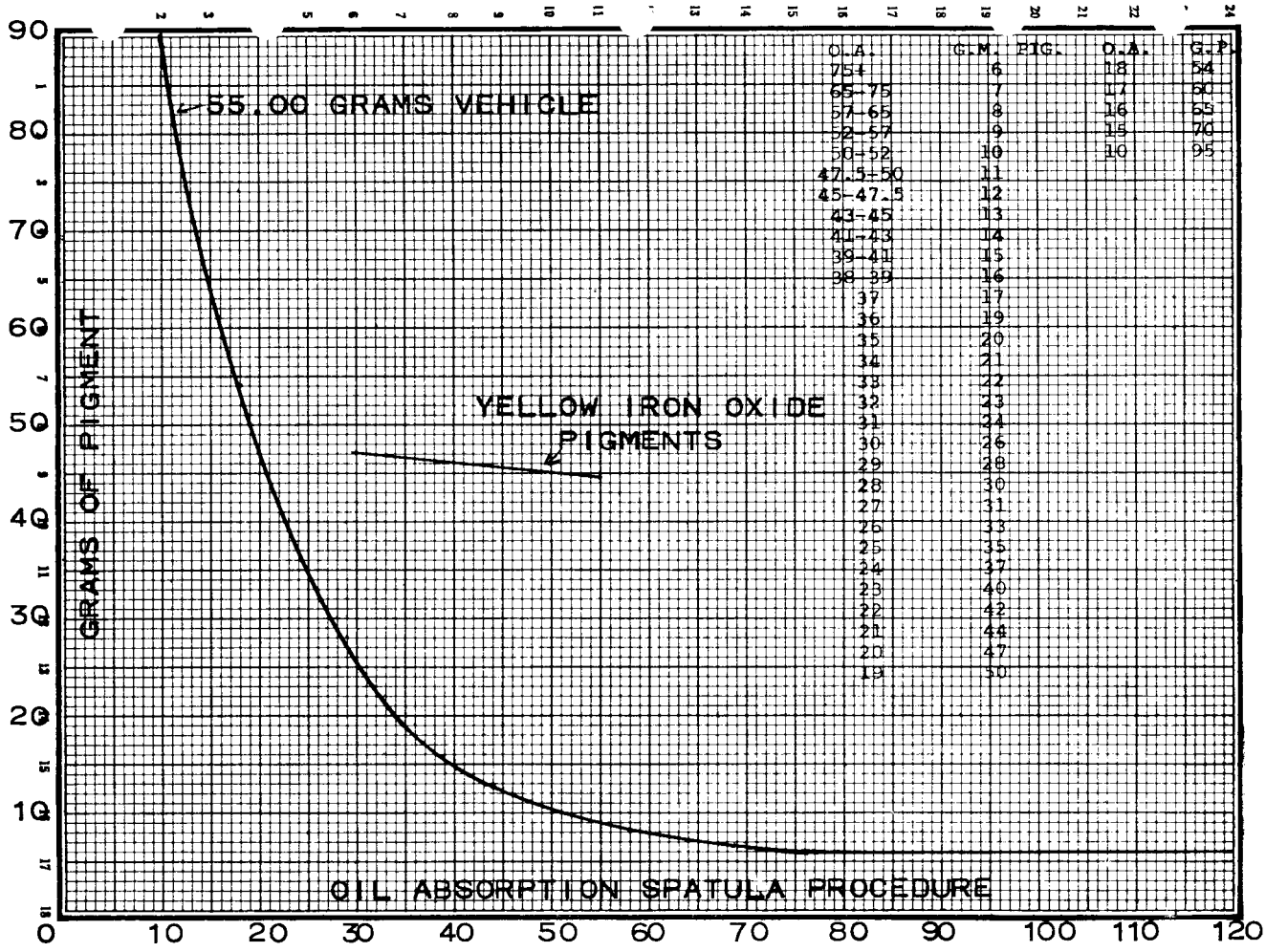

FIG. 1 Oil Absorption Spatula Procedure

5. Materials

5.1 Samples of standard reference pigments as agreed upon between the purchaser and the seller.

5.2 *Standard Sand*[10]—Regular 20 to 30-mesh (850 to 600-µm) cement testing sand conforming to Specification C 778. The sand shall be screened to remove all sub 30-mesh particles. Twenty to thirty-mesh glass beads may be used instead of sand.[11]

5.3 *Grinding Vehicle*—A long oil alkyd[12] meeting U.S. Fed. Spec. TT-R-266 Type 1-A reduced to 47 % solids with mineral spirits conforming to Specification D 235 (2 parts alkyd and 1 part mineral spirits by weight).

5.4 *White Tinting Paint*—A flat white tinting paint compatible with the dispersion vehicle meeting the following requirements.

5.4.1 *Gloss (60°)* less than 4, as determined by Test Method D 523.

5.4.2 *Contrast Ratio* (2-mil (51-µm) dry film): 99.2 min.

5.5 *Drier Blend*—One part 6 % manganese naphthenate, 2 parts 6 % cobalt naphthenate, and 4 parts 24 % lead naphthenate by weight.

6. Hazards

6.1 While operating the mill, keep hands well away from the shaft and disk and be sure that no article of clothing (for example, necktie or long hair) will catch on moving parts. This precaution applies to all cases where the mill is in operation.

7. Procedure for Mass Color

7.1 Weigh to the nearest 10 mg an appropriate amount of pigment to be tested into a 200-mL tall-form beaker. (The amount of pigment needed can be calculated from the graph shown in Fig. 1 or obtained from the table on the graph.) To the pigment in the beaker carefully weigh in 55.00 ± 0.01 g of the long oil alkyd grinding vehicle (Note 1). Then add 60 mL of 20 to 30-mesh sand from a graduated cylinder and 1.00 ± 0.01 g of the drier blend. Stir the sand, vehicle, and pigment with a

[10] The sole source of supply of the sand known to the committee at this time is Agsco Division, American Graded Sand Co., 189 E. 7th St., Patterson, NJ 07524. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[11] The sole source of supply of the glass beads known to the committee at this time is Quackenbusch Co., P. O. Box 607, Palatine, IL 60067. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[12] A long oil soya or safflower alkyd at 70 % solids with the following characteristics: Nonvolatile 70 ± 1 %, phthalic anhydride 23 % min, fatty acids 60 to 65 %, dihydric alcohol 4 % max, acid number 5 to 10, specific gravity 0.950–0.970, Gardner color 10.

spatula until the pigment is completely wetted.

NOTE 1—For routine control, grinding vehicle, drier blend, and white tint paint can be placed in the type of dispensers used in stores to tint custom colors. The following amounts are recommended: 2 fluid oz each of vehicle and of white paint, ⅓ oz of drier. The dispenser shall be reproducible to 2 parts per thousand when dispensing 1-oz charges.

7.2 Carefully clamp the beaker on the miniature sandmill and adjust the height of the disk so that it is between ¾ and 1 in. (20 and 25 mm) above the bottom of the beaker. Turn on the mill and disperse for exactly 10 min, unless otherwise agreed upon, at a shaft speed of 8000 r/min, accurately timing the length of dispersion with a stopwatch or other suitable timing device.

NOTE 2—For normal control procedure, a 10-min dispersion interval will be sufficient. For ultimate color development use the procedure in Annex A1.

7.3 Shut off the mill and raise the impeller until it is just clear of the liquid in the beaker and turn the mill on again for a few seconds to remove excess mill base from the impeller.

7.4 Remove the material from the mill and filter into a suitable container through a cone strainer or equivalent 40-mesh (420-µm) disposable screen.

7.5 Repeat the foregoing procedure with the standard reference pigment.

7.6 Draw the test and standard dispersions down simultaneously on a paper chart over a vacuum drawdown plate or other suitable plane surface using the applicator bar specified in 4.8. Compare the color visually while still wet and then set aside in a dust-free area to dry. If desired, evaluate the color difference instrumentally by Test Method D 2244. Report color difference in units agreed upon between the purchaser and the seller.

8. Procedure for Tint Color

8.1 From the receiving container used in 7.4, weigh 5.000 ± 0.010 g of the strained mill base into a 200-mL beaker and add 100.00 ± 0.05 g of white tinting paint.

8.2 Place the beaker on the mill (without sand) and mix at 8000 r/min for 5 min.

NOTE 3—Some mill bases require vigorous agitation when being dispersed into the paint. Normal agitation on a paint shaker is not satisfactory.

8.3 Repeat the above procedure with the standard reference material and then proceed as directed in 7.6.

8.4 To determine the relative tinting strength of the test material as compared to the standard at the wavelength of maximum absorbance, or at a wavelength agreed upon between the purchaser and the seller, employ the following procedure.

8.4.1 If the spectrophotometric curves cross or otherwise deviate from a relationship characteristic of effectively identical materials, the method is inapplicable (that is, the method is not applicable to metameric specimens).

8.4.2 When spectrophotometers or abridged spectrophotometers are used as color-measuring instruments, determine the lowest reflectance reading between 420 and 680 mm, or at the agreed upon band of wavelengths, and use it in the calculation. Read and record the reflectance to at least the nearest 0.001 reflectance unit.

8.4.3 With color-measuring instruments that employ broad wavelength filters, use the lowest reflectance obtained, or the reflectance from the agreed upon filter, in the calculations. Read and record the reflectance to at least the nearest 0.001 reflectance unit.

NOTE 4—The calculation is based on theory that, strictly interpreted, requires for the types of material being tested, use of a spectrophotometer or abridged spectrophotometer. However, many users of this test method obtain satisfactory results using color-measuring instruments equipped with broad wavelength filters.

8.4.4 Calculate the tinting strength of color pigments as follows:

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

where:

TS = tinting strength,

R = measured reflectance and subscript "u" means reflectance of material under test (that is, unknown) and "s" means reflectance of standard reference material, and

T = assigned TS of the standard reference material, frequently assigned at "100" so that TS is in percent of standard.

9. Precision

9.1 On the basis of an interlaboratory study of this test method in which two operators in five laboratories tested five pigments on each of two days, the precision was found to be as shown.

9.2 *Mass Color*—The within-laboratory standard deviation was found to be 0.97 MacAdam color-difference units and the between-laboratory standard deviation 1.14 units. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.2.1 *Repeatability*—Two results obtained in the same laboratory should be considered suspect if they differ by more than 2.7 MacAdam units.

9.2.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 3.2 MacAdam units.

9.3 *Tint Strength*—The within-laboratory standard deviation was found to be 2.74 % and the between-laboratory standard deviation 3.46 %. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

9.3.1 *Repeatability*—Two results obtained in the same laboratory should be considered suspect if they differ by more than 7.7 %.

9.3.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 9.7 %.

10. Keywords

10.1 color; miniature sandmill; strength

ANNEX

(Mandatory Information)

A1. DETERMINATION OF MAXIMUM COLOR

A1.1 In some cases it may be desirable to determine the maximum color obtainable by this procedure. In this case Section 7 may be modified as follows:

A1.1.1 Carefully determine the total weight of the dispersion mixture and container. Place on the mill and disperse for 10 min. After spinning the impeller free of excess material, reweigh the container and bring back to the original weight with solvent. Carefully mix the added solvent back into the dispersion and extract 2 g of sandfree base, reduce with 50 g of white paint, and prepare a drawdown. Repeat the preceding process at 10-min intervals until no further increase in tint strength occurs. Note the time at which maximum tinting strength occurs and record for future reference.

A1.1.2 Subsequent samples of this pigment can then be run for the optimum length of dispersion time. However, solvent lost during dispersing must be replaced periodically so that viscosity variations will not affect the rate of dispersion.