Designation: D2371 – 85 (Reapproved 2010)

Standard Test Method for Pigment Content of Solvent-Reducible Paints[1]

This standard is issued under the fixed designation D2371; 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 Department of Defense.

1. Scope

1.1 This test method covers the procedure for the quantitative separation of the vehicle from the pigment in solvent-reducible coatings.

1.2 This test method has been proven to be applicable to the following types of paints: white linseed oil outside house paint, white soya and phthalic alkyd enamel, white linseed *o*-phthalic alkyd enamel, red lead primer, zinc chromate primer, flat white inside enamel, white epoxy enamel, white vinyl toluene modified alkyd, and white amino modified baking enamel. It is considered to be applicable to most solvent-reducible paints.

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.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging

3. Significance and Use

3.1 This test method is suitable for setting specifications for the pigment content of solvent-reducible paints as well as for monitoring manufacturing quality control.

3.2 This test method provides the isolated pigment fraction from solvent-reducible paints that may be used for pigment analysis.

4. Apparatus

4.1 *Centrifuge,* explosion-proof, capable of developing 1000 to 2000 *g*.

NOTE 1—The centrifuge should be equipped with a suitable head to take the proper size trunnion cups necessary for use of the 90-mL tubes or 120-mL (4-oz) bottles. A two, four, or six-place head can be used with the bottles and an eight-place head can be used with the tubes.

4.2 *Centrifuge Tube,* 90-mL, heavy-walled. In place of the 90-mL centrifuge tube a 120-mL (4-oz) screw cap bottle with vinyl-lined screw cap may be used.[3]

4.3 *Laboratory Oven,* vented and capable of maintaining a temperature of 105 ± 2°C.

4.4 *Syringe,* 5-mL.

4.5 *Water Bath.*

5. Solvents

5.1 *Ethyl Ether or Petroleum Ether:* (**Warning—**See 6.1).

5.2 *Extraction Mixture*—Mix 10 volumes of ethyl ether, 6 volumes of benzene or toluene, 4 volumes of methyl alcohol, and 1 volume of acetone (**Warning—**See 6.2, 6.3, and 6.4).

6. Hazards

6.1 *Ethyl Ether and Petroleum Ether*— Flammable. Vapor is harmful. May be fatal if inhaled or swallowed. Use only with adequate ventilation. Avoid prolonged contact with skin. Avoid contact with flame, hot surface, or sparks. Do not get in eyes, on skin, or on clothing. Refer to suppliers' Material Safety Data Sheet.

6.2 *Acetone*—Flammable. Vapor is harmful. May be fatal if inhaled or swallowed. Use only with adequate ventilation.

[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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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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] The sole source of supply of bottles and caps, Cat. No. S9185C, 4-oz bottle, known to the committee at this time is Sargent Welch Scientific, 7300 N. Linder Ave., Skokie, IL 60076. 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.

Avoid prolonged contact with skin. Avoid contact with flame, hot surface, or sparks. Do not get in eyes, on skin, or on clothing. Refer to suppliers' Material Safety Data Sheet.

6.3 *Methyl Alcohol*—Flammable. May be fatal or cause blindness if swallowed. Cannot be made nonpoisonous. Harmful if inhaled. Keep away from heat, sparks, or open flame. Avoid breathing vapor. Use only with adequate ventilation. Refer to suppliers' Material Safety Data Sheet.

6.4 *Benzene* has been declared carcinogenic.

6.4.1 *Benzene and Toluene*—Flammable. Vapors and liquid are harmful and may be fatal if swallowed. Keep away from heat, sparks, or open flame. Keep in a well-closed container. Avoid breathing vapor. Avoid contact with eyes, skin, or clothing. Refer to suppliers' Material Safety Data Sheet.

7. Procedure

7.1 Mix the sample until it is homogeneous, preferably on a mechanical shaker.

7.2 Weigh to 1 mg from a syringe, 5 to 10 g of sample into a tared 90-mL centrifuge tube with a screw-type glass stirring rod (the rod is tared with the centrifuge tube), or a glass bottle as described in 4.2. The weight of the specimen is the weight of syringe and paint minus the weight of the syringe after transferring the specimen to the tube or bottle.

7.3 Add 60 mL of extraction mixture (5.2) and stir well with the glass, screw-type stirring rod, preferably using a power stirrer. Wash the rod clean with a stream of extraction mixture into the tube, and reserve the rod. If the glass bottle is used, mixing may be accomplished by hand, shaking vigorously, or by use of a mechanical shaker.

7.4 Centrifuge at 1000 to 2000 *g* (Note 2) until the pigment is clearly separated. A faster speed may cause the pigment to pack too hard for future mixing. (See Test Method D2698.)

NOTE 2—Calculate the gravity developed by the centrifuge as follows:

$$g = 1.118 \times 10^{-6} \times r \times n^{2} \quad (1)$$

where:

r = rotating radius, mm, and
n = rotating speed, r/min.

7.5 Discard the upper layer by decantation.

7.6 Repeat the extraction twice as described in 7.3, 7.4, and 7.5, making certain that the settled pigment is sufficiently stirred or shaken so that no lumps can be seen sticking to the side of the centrifuge tube or bottle and all pigment can easily be washed from the stirring rod into the tube. It may be necessary to break up the pigment cake before stirring. The bottle and contents may be mixed by tapping at an angle on a cloth pad on a bench top to break up the pigment cake and then shaking vigorously to complete the pigment dispersion in the extraction mixture.

7.7 Make a fourth extraction using ethyl ether or petroleum ether following the same precautions noted in 7.6. Decant the upper layer. Drive off the ether by heating (gently at first to avoid spattering) on a steam bath in a hood away from open flames or sparks. The pigment should be broken up by tapping the tube or bottle on a cloth pad on a bench top. Dry the tube or the bottle at 105 ± 2°C for 2 h.

7.8 Cool and weigh the tube (including stirring rod) or bottle, plus pigment.

8. Calculation

8.1 Calculate the percent of pigment, P, as follows:

$$P = \left[\left(W_1 - W_2\right)/S\right] \times 100 \quad (2)$$

where:

W_1 = weight of the tube (including stirring rod) plus pigment, g,
W_2 = weight of the tube (including stirring rod), g, and
S = weight of paint sample used, g.

NOTE 3—All pigment obtained using this test method has minute amounts of vehicle adsorbed on the pigment. However, this will not significantly affect the precision of this test method.

9. Precision

9.1 Data are not available to determine the precision of this test method. There are no plans at present to obtain such data. This test method has been in use for several years and is considered acceptable.

10. Keywords

10.1 high speed centrifuging; pigment content; pigment separation; solvent-reducible paint