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Standard Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging¹

This standard is issued under the fixed designation D 2698; 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 separation of pigment from solvent-reducible paints and the calculation of the percent pigment from the results of nonvolatile determinations on the total paint and the separated vehicle.

NOTE 1—This test method has been proven to be applicable to most solvent-reducible paints, the exception being those paints containing severely bleeding pigments or pigments such as carbon black that are very difficult or impossible to centrifuge.

1.2 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 2369 Test Method for Volatile Content of Coatings²

3. Significance and Use

3.1 This test method describes a rapid method for separating pigment from solvent-reducible paints.

3.2 This test method is used by paint producers and consumers for product acceptance and process control.

4. Apparatus

4.1 *Laboratory Centrifuge*, capable of developing 32 000 g or higher.³

5. Procedure

5.1 Shake the sample for 10 min on a mechanical shaker,

To calculate the gravities developed by a particular centrifuge use the following calculation:

 $g = \text{rotating radius, cm} \times \text{r/min}^2 \times 1.118 \times 10^{-5}$

stir with a paddle to loosen any caked pigment, and shake again for 10 min. If necessary, repeat until there are no lumps present and the sample is thoroughly mixed.

5.2 Add the mixed paint to the centrifuge container until sufficient paint is added to permit the recovery of 25 mL minimum of clear vehicle. Revolve the bowl at 32 000 g or higher for 15 min or until a clear vehicle is obtained.

5.3 Transfer the vehicle carefully, so as not to dislodge any of the pigment from the walls, into a jar that is immediately capped to prevent any solvent losses by evaporation. Use this vehicle for the determination of the nonvolatile content of the separated vehicle.

6. Percent Paint and Vehicle Solids

6.1 Determine the percent nonvolatile content of the original paint and the separated vehicle in accordance with Test Method D 2369.

7. Calculation

7.1 Calculate the weight percent pigment of paint, P, as follows:

$$P = (A - B) \times 100/100 - B \tag{1}$$

where:

A = nonvolatile content of paint, %, and

B = nonvolatile content of separated vehicle, %.

8. Recovery of Pigment for Analysis

8.1 The pigment may be completely recovered from the centrifuge bowl and washed to remove residual vehicle. This may be done either by a Soxhlet extraction or washing the pigment with a suitable solvent and recentrifuging.

NOTE 2—It has been noted that the removal of the pigment from the bowl of a Sharples supercentrifuge can be facilitated by lining the bowl with a piece of polyester film prior to adding the sample and subsequently just slipping the polyester film along with the pigment out of the bowl.

9. Precision and Bias

9.1 *Precision*—The precision estimates are based on an interlaboratory study of this test method in which air drying materials were analyzed in duplicate or triplicate. The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

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

Current edition approved May 25, 1990. Published July 1990. Originally published as D 2698 – 68. Last previous edition D 2698 – 73 $(1984)^{\epsilon_1}$.

² Annual Book of ASTM Standards, Vol 06.01.

³ A supercentrifuge or a centrifuge equipped with a high-speed head have been found satisfactory for this purpose. Any centrifuge capable of developing 32 000 g and having facilities for sealing the specimen to prevent solvent loss should also prove to be satisfactory.

9.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same operator, will approximate 0.1 % absolute. Two such values should be considered suspect if they differ by more than 0.25 % absolute.

9.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations obtained by operators in different laboratories, will approximate 0.25 % absolute. Two such values should be considered suspect if they differ by more than 0.5 % absolute.

9.1.3 This precision pertains to air-drying materials. Heatreactive vehicles will have a repeatability and reproducibility approximately twice as large, that is, 0.5 % and 1.0 % absolute. 9.1.4 It should be noted that when the solvent content of the total paint is very low (15 % or less) the precision of this test method will not be satisfactory unless the nonvolatile determination is precise to the second decimal place.

9.2 *Bias*—Bias has not been determined for this test method.

10. Keywords

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

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