



Standard Test Method for Oil Absorption of Pigments by Spatula Rub-out¹

This standard is issued under the fixed designation D 281; 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 determination of the oil absorption of pigments by the spatula rub-out technique.

1.2 The values stated in SI 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 234 Specification for Raw Linseed Oil²

D 1483 Test Method for Oil Absorption of Pigments by Gardner-Coleman Method³

3. Summary of Test Method

3.1 A stiff, putty-like paste is formed by the dropwise addition of linseed oil to pigment that is being thoroughly rubbed with a spatula. The amount of oil required to produce the end point is used to calculate an oil absorption value.

4. Significance and Use

4.1 The oil absorption value obtained by this test method gives information about the vehicle demand of the pigment when it is used in a pigment paste. Oil absorption values can be used to characterize pigments or batches of a given pigment.

4.2 This test method differs from Test Method D 1483 in that D 1483 involves only a gentle stirring and folding of the pigment, whereas this test method requires a thorough rubbing action. Because the end points are different, the values obtained from the two test methods generally differ.

5. Apparatus and Materials

5.1 *Balance*, capable of weighing to 0.001 g.

5.2 *Dropping Bottle*, fitted with ground-in pipet and rubber bulb or buret, graduated in 0.1-mL divisions.

5.3 *Smooth Glass Rub-up Plate or Marble Slab* (glass should have a surface similar to Hoover Muller Plates).

5.4 *Spatula*, sharp-edged, steel, having a blade of 15 or 20 mm by 100 mm ($\frac{1}{2}$ or $\frac{3}{4}$ by 4 in.).

5.5 *Oil*, linseed, raw, conforming to Specification D 234 except that it shall have an acid number of 3 ± 1 . Linseed oil used in comparative tests must have the same acid value. Other liquids, such as refined oil, may be used by mutual agreement.

6. Procedure

6.1 *Procedure A (Weighing Bottle)*—Weigh exactly 1 g., or any multiple thereof (Note 1), of the thoroughly mixed and air dried pigment and place upon a glass plate or marble slab. Weigh to 0.01 g a dropping bottle containing raw linseed oil along with the pipet and rubber bulb. Add the linseed oil gradually, drop by drop (by means of the pipet), to the pigment. After the addition of each drop, thoroughly incorporate the oil by rubbing up with the spatula. The test is complete when exactly enough oil has been incorporated with the pigment to produce a very stiff, putty-like paste, that does not break or separate. Weigh the bottle and oil to 0.01 g and determine by difference the weight of oil used.

NOTE 1—The specimen weight depends upon the specific gravity, fineness, and other characteristics of the pigment. For example, 20 g is taken for white lead, but about 1 g is sufficient for carbon black. In any event, the specimen size should be large enough so that at least 1 g of oil is required.

6.2 *Procedure B (Buret)*:

6.2.1 Follow Procedure A, except add the linseed oil from a buret rather than a dropping bottle pipet. Calculate the weight of oil in grams by multiplying the volume oil used by its density (0.93 g/mL).

6.3 It is suggested that when a new pigment is to be tested, a preliminary rub-out be made to determine an approximate end point. Once this is established, the actual determination should be made with a slower addition of oil and a more

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² *Annual Book of ASTM Standards*, 06.03.

³ *Annual Book of ASTM Standards*, 06.01.

vigorous rub-out through the critical region, therefore permitting a more precise assessment of the correct oil absorption end point.

7. Calculation

7.1 From the weights of oil and pigment used in the test, calculate the number of grams (pounds) of oil required to exactly wet grams of oil per 100 grams of pigment or 100 pounds of pigment.

8. Report

8.1 Report the oil absorption of the pigment as the number of grams (pounds) of oil required to exactly wet 100 grams (pounds) of pigment.

9. Precision and Bias

9.1 On the basis of an interlaboratory study of this test method in which one operator in each of five laboratories tested two grades of zinc oxide covering a small oil absorption range

by spatula rub-out, the within-laboratory coefficient of variation was found to be 4.6 % at 10 df and the between-laboratories coefficient of variation 11.6 % at 8 df. Based on these coefficients, the following criteria should be used for judging the precision of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results obtained by a single operator should be suspect if they differ by more than 14.3 % relative.

9.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 38.0 % relative.

9.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method, bias cannot be determined.

10. Keywords

10.1 oil absorption; pigments (general properties); spatula rub-out test

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