Designation: D 185 – 84 (Reapproved 1999)

Standard Test Methods for Coarse Particles in Pigments, Pastes, and Paints[1]

This standard is issued under the fixed designation D 185; 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 These test methods cover the determination of the amount of coarse particles in dry pigments and of coarse particles and skins in mixtures of pigments and vehicles.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[2]

3. Significance and Use

3.1 In production of paints, smoothness of the paint film is of paramount importance. Agglomerates or coarse particles larger than 45 µm are difficult to disperse and may prevent obtaining a smooth film. These test methods are a valuable quality control test for grading raw materials.

4. Apparatus

4.1 The apparatus shall consist of a 3-in. (75-mm) 45-µm (No. 325) sieve conforming to Specification E 11. A3-in. 45-µm sieve for comparison purposes should be retained in the laboratory as a reference standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it and on the reference standard sieve a comparison test, using a pigment that has a considerable amount of coarse particles. A reserve stock of such a pigment should be kept for this purpose.

5. Procedure for Insoluble Dry Pigments, Except Metallic Aluminum and Bronze Powders

5.1 Dry the sieve in an oven at 105 ± 2°C, cool, and then weigh on an analytical balance, recording the weight to 1 mg.

5.2 Weigh a specimen (25 g for basic carbonate and basic sulfate white leads, 25 g for red lead and mercuric oxide, 2 g for black pigments of low specific gravity, 3 g for Prussian blues and graphite, and 10 g for all other pigments) of the pigment to be tested on an analytical balance to 1 mg. Wet the sieve on both sides with alcohol and transfer the specimen of pigment to the sieve and wet with alcohol.

5.3 Hold the sieve under a tap delivering about 300 to 500 mL of the wash liquid (water) per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's-hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment gradually collects at one edge during the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the sieve.

5.4 After most of the finely divided portion of the pigment has passed through the sieve (from 2 min to 1 h, according to the kind of pigment), place the sieve in an 8-in. (200-mm) porcelain dish containing 250 mL of the wash liquid so that the sieve is covered to a depth of about ½ in. Brush the pigment remaining on the sieve with a soft 1-in. (25-mm) camel's-hair brush at the rate of two strokes per second during two periods of 10 s each. Raise the sieve from the dish after each 10-s period to let the liquid on the sieve run through. Change the liquid in the dish after every two brushing periods. Continue this operation until the wash liquid passing over the residue and through the sieve is clear and free from solid particles. When the washing appears to be complete, collect about 200 mL of the wash liquid, after passing over the residue and through the sieve, in a clean 400-mL beaker. Stir the liquid vigorously, and set the beaker on a black surface in the case of white pigments and on a white surface in the case of colored pigments. The washing is not considered complete until such a test fails to show any particles collected about the middle of the bottom of the beaker.

NOTE 1—Occasionally, pigments will be found that foam when water is used as the wash liquid. In such instances, during the last washing in the porcelain dish the use of a liquid that breaks down the foaming and is

[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications, and are the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

readily miscible with water, such as alcohol, will usually overcome this difficulty.

5.5 When the washing is complete wash the pigment particles adhering to the brush back onto the sieve and wipe off the water below the sieve. Add a few drops of alcohol and then of ether to expedite drying. Dry the sieve for 1 h at 105°C, cool, and weigh as described in 5.1. Calculate the percent of coarse particles.

6. Procedure for Metallic Aluminum and Bronze Powders

6.1 In the case of metallic aluminum and bronze powders follow the procedure described in Section 5 except use 5 g of the material as the specimen and denatured alcohol, instead of water, as the wash liquid.

7. Procedure for Carbon Black in Pellet Form

7.1 Dry, cool, and weigh the sieve as described in 5.1.

7.2 Prepare a stock solution of dispersing agent[3] in water, using as high a concentration of dispersing agent as possible without losing fluidity. When solution has been obtained, filter through coarse filter paper. A quart of this solution will be sufficient for several tests.

7.3 Crush the pellets between two glass plates approximately 12-in. (305-mm) square, using a gentle rotating motion. If 10 g of carbon black are taken, crush about 2 g at a time. Weigh into a 600-mL beaker 10 g of the crushed particles. Add enough of the dispersing solution to make a heavy paste and mix well to incorporate thoroughly the carbon black. Dilute with water to about 300 mL; then pour into the clean 45-µm (No. 325) sieve. First pass the tap water through a 45-µm sieve.

7.4 Using a small camel's-hair brush, gently brush the mix through the sieve, running tap water slowly through at the same time. When it appears that all the dispersed black has gone through, stop the tap water and continue brushing until most of the water remaining on the sieve has gone through. Add a few millilitres of the stock solution of the dispersing agent, thoroughly mix with the brush, then turn on the tap water again and wash through as before. Repeat this operation until no dispersed black comes through the sieve. When about 0.5 g of sand-like material is left on the sieve, the end point is being approached. At this point, adding the dispersing solution and gently rubbing it into the residue with the finger still produces a colloidal dispersion of black which easily passes through the sieve. From this point on, before each addition of dispersing agent, work the residue into the center of the sieve with the tap water. Many washings and many additions of dispersing solution are required in order to reach the end point. Take care to make certain that all the black has been washed through the sieve.

7.5 Dry the sieve in an oven at 105°C for 1 h, cool, and weigh the residue. Calculate the percent of coarse particles. If the value thus obtained is greater than that specified, proceed as follows to remove any adhered carbon that will pass through the 45-µm (No. 325) sieve: Transfer the particles on the sieve to a piece of white bond paper and gently rub the carbon onto the paper with the finger. When no further real blackening of the paper occurs, carefully transfer the residue to the balance pan, weigh, and recalculate the percent of coarse particles.

8. Procedure for Water-Soluble Pigments, Pastes in Oil, Pastes in Japan, and Mixed Paints

8.1 Dry, cool, and weigh the sieve as described in 5.1.

8.2 For water-soluble pigments use 10 ± 1 g as the specimen. For pastes in oil, pastes in Japan, and mixed paints use 25± 1 g as the specimen. For white leads and red lead use 50 ± 1 g as the specimen.

8.3 Weigh the specimen to 1 mg and transfer to a 250-mL beaker. Slowly add 100 mL of kerosine to the contents of the beaker, mixing thoroughly by use of a stirring rod with flattened end. Break up all lumps but do not grind the material.

8.4 Wet the sieve on both sides with kerosine; then transfer the contents of the beaker to the sieve using a wash bottle filled with kerosine.

8.5 Remove small particles retained on the stirring rod or beaker walls with a camel's-hair brush. Rinse the brush with kerosine.

8.6 When the washing is complete, wash the pigment particles adhering to the brush back onto the sieve and wipe off kerosine below the sieve. Dry the sieve for 1 h at 105°C, cool, and weigh as described in 5.1. Calculate the percent of particles.

9. Procedure for Ship-Bottom Paints Containing Resins and Alcohol

9.1 In the case of ship-bottom paints containing resins and alcohol follow the procedure described in Section 8, with specimen weight of 25 ± 1 g, but use denatured alcohol, instead of kerosine, as the wetting medium, for mixing with paint, and as the wash liquid.

10. Procedure for Cellulose Ester Lacquers

10.1 In the case of cellulose ester lacquers follow the procedure described in Section 8, with specimen weight of 25 ± 1 g of the material but use a mixture of equal parts of ethyl acetate, toluene, and denatured alcohol, instead of kerosine, as the wetting medium, for mixing with the lacquer, and as the wash liquid.

11. Procedure for Latex and Emulsion Paints

11.1 *Apparatus*—As described in Section 4 except that sieves used shall be as follows:

11.1.1 *Flat Paints*, a 3-in. (75-mm) 90-µm (No. 170) sieve.

11.1.2 *Gloss and Semi-Gloss Paints*, a 3-in. (75-mm) 75-µm (No. 200) sieve.

11.2 Dry, cool, and weigh the sieve as described in 5.1.

11.3 Weigh 25 g of paint to ±1 g into a tared 250-mL beaker. Without delay, start adding 100 mL of water to the paint in the beaker, slowly and with hand stirring sufficient to thoroughly mix the paint with the water. Wet the sieve on both sides with water and transfer the contents of the beaker gradually to the sieve, using a wash bottle containing water. Small particles retained on the stirrer or beaker walls may be removed with a camel's-hair brush. Instead of tap water, use reagent water for diluting the paint sample, in the wash bottle,

[3] Any dispersing agent specific for carbon black may be used.

and for washing the paint through the sieve. The water may be delivered from a reservoir for the last step.

11.4 Follow the procedure described in 5.3. In using the camel's-hair brush, take care not to crush agglomerates that would not be broken down in normal application of the paint. Rinse the brush into the sieve at end of use.

11.5 Finally, dry, cool, and weigh the sieve for 1 h, as described in 5.1. Then calculate the percent of coarse particles and skins.

12. Precision

12.1 Precision data are not available at this time. When available the appropriate precision statements will be added.

13. Keywords

13.1 agglomerates; coarse particles; paints; paste; pigments; skins