Designation: D 2067 – 97 (Reapproved 2003)

Standard Test Method for Coarse Particles in Printing Ink Dispersions[1]

This standard is issued under the fixed designation D 2067; 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.

1. Scope

1.1 This test method covers the determination of the weight concentration of coarse particles in printing ink dispersions by sieve retention.

1.2 This test method is applicable to printing inks, flushed pigments, and other pigment dispersions that contain particles larger than 45 µm. With proper choice of solvent, it is applicable both to paste and liquid inks.

NOTE 1—This test method is similar in principle to Test Methods D 185. For particles under 25 µm, see Test Method D 1316.

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 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 185 Test Methods for Coarse Particles in Pigments, Pastes and Paints[2]

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

D 1316 Test Method for Fineness of Grind of Printing Inks by the NPIRI Grindometer[4]

E 11 Specification for Wire Cloth and Sieves for Testing Purposes[5]

E 145 Test Method for Gravity-Convection and Forced-Ventilation Ovens[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Summary of Test Method

3.1 A 50-g specimen of the test dispersion is diluted, if necessary, with a reducing varnish, mixed with 200 g of mineral spirits or other mutually agreeable solvent, mixed in a paint shaker for 30 min, and passed through a tared 325-mesh wire cloth sieve. The sieve is dried in an oven and reweighed. The residue is reported either as a percentage or in parts per million of the specimen.

3.2 The nature of the coarse particles may be identified through the use of a magnet and visual or microscopic examination.

4. Significance and Use

4.1 Coarse particles in printing inks reduce the efficiency of the dispersion process, requiring not only extra milling passes, but also frequent changes in pump filters. In printing processes, they may cause excessive wear to metal plates, piling or localized retention of ink on blankets and plates, and water balance problems. Coarse particles also reduce color strength and the gloss of printed matter.

4.2 This test method is suitable for quality control. The precision may be improved by the use of a specimen size larger than that prescribed.

4.3 Test results are sensitive to the type of washout solvent used. Strong solvents are to be avoided because they may dissolve large particles of resin in the ink vehicle.

5. Apparatus

5.1 *Wire-Cloth Sieve*, preferably disposable, 325 mesh (45 µm), 60.3 mm in diameter; alternatively, a 75-mm No. 325 sieve conforming to Specification E 11.

NOTE 2—A disposable sieve is preferred for precision because it weighs only 0.5 g compared to about 70 g for a conventional sieve.

5.2 *Washout Cup Assembly* (for use with the disposable sieve), assembled according to the manufacturer's instructions.

5.3 *Balance*, sensitive to 1.0 mg, preferably 0.1 mg.

5.4 *Paint Shaker*.

5.5 *Oven*, gravity-convection type conforming to Type 1B in Test Method E 145 and maintained at 110° ± 5°C.

5.6 *Mixing Container*, such as a widemouth bottle or jar, with screw cap, capacity 473 mL (1 pt), preferably plastic. If a glass jar is used, a metal container such as a 1-lb coffee can

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

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

[4] *Annual Book of ASTM Standards*, Vol 06.02.

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

with lid and foam packing are recommended for protecting the jar while on the shaker.

5.7 *Spatula*, suitable for mixing 100 g of material.

6. Reagents and Materials

6.1 *Washout Solvent*, as mutually agreed upon between the producer and the user. Type I mineral spirits conforming to Specification D 235 has been found useful for oleoresinous systems. The solvent should be filtered through a 325-mesh screen prior to use.

6.2 *Reducing Varnish*, for high-viscosity or stiff dispersions. The varnish must be compatible with the test dispersion and of a consistency such that a 50:50 mix will have sufficient flow to disperse in the washout solvent. An 800–1200 poise-reducing varnish has been found useful for flushed pigments. The reducing varnish should be washed or filtered, or both prior to use; otherwise, a blank must be run (see 8.11).

6.3 *Plastic Sheeting*, about 150 mm square and 0.001 mm thick.

7. Test Specimen

7.1 Approximately 100 g of dispersion is sufficient to conduct two tests. If a larger specimen is preferred, increase the amount of reducing varnish (if used), the washout solvent, and the size of the mixing container accordingly.

7.2 Make sure the test specimen is representative of the lot. If the dispersion is a liquid ink in a sample container, hand stir vigorously for 2 min before removing the specimen.

8. Procedure

8.1 Weigh out 50 ± 0.1 g of the test dispersion into a widemouth jar or other mixing container. Using a spatula, spread the specimen into a thin film around the bottom and lower sides of the container.

8.2 If the test material is too stiff to spread out or to mix with the solvent (in 8.3) make a reduction in one of the following manners:

8.2.1 Weigh out 50 ± 0.1 g of reducing varnish and add in small increments to the 50-g specimen in the jar while stirring with a spatula. Mix thoroughly until no lumps are present.

8.2.2 Alternatively, discard the specimen in the jar. Weigh out 55 g of the test specimen and transfer to a slab. Weigh out an equal quantity of reducing varnish and mix in small increments with the specimen until no lumps are present. Transfer to the jar 100 g ± 0.1 g (of which 50 g will be specimen) and spread out as in 8.1.

8.3 With the spatula still in the container, slowly add, while stirring, about 200 mL of the prescribed washout solvent. Scrape the spatula clean into the jar.

8.4 Place a plastic sheet over the mouth of the jar and tighten the lid. If made of glass, set the jar into a metal container lined with foam or other soft material before locking between the jaws of the shaker.

8.5 Turn the shaker on and let run for 30 min.

8.6 When the shaker stops, remove the jar, unscrew the lid and carefully remove the plastic sheet. Using a wash bottle containing the same solvent as in 8.3, wash down the plastic sheet into the jar containing the dissolved specimen.

8.7 Tare a clean dry sieve to 1 mg, preferably 0.1 mg, and, if disposable, set in place on the washout cup assembly. Pour the contents of the jar quickly through the sieve. Invert the jar over the screen and rinse out with solvent from the wash bottle until no color remains in the jar. Make sure that no residue remains in the jar and that all solvent from the jar passes over the screen.

8.8 Wash down the sides of the washout cup assembly (if used) and the sides of the screen, collecting the residue close to the center of the sieve.

8.9 Place the sieve in a convection oven at 110°C for a few min or until the screen and residue are dry. Cool in a desiccator and weigh. Record the weight of the residue.

8.10 Repeat 8.1 or 8.2 to 8.9 with a second specimen.

8.11 If a reducing varnish was used (in 8.2) and it was not washed and sieved, run a blank on two replicate specimens.

8.12 Valuable information may be obtained on the nature of the coarse particles by visual or microscopic examination of the residue. Passing a magnet under the residue can help to identify the presence of iron particles. Fibers can be removed with tweezers and their number counted.

9. Calculation

9.1 Calculate the weight concentration, C, of coarse particles either in percent or in parts per million of the specimen as follows:

$$C, \% = \left(\frac{W - w}{S} - \frac{B - w'}{S'}\right) \times 100 \quad (1)$$

$$C, ppm = \left(\frac{W - w}{S} - \frac{B - w'}{S'}\right) \times 10^6 \quad (2)$$

where:

W = weight of the test residue and screen, g,
w = weight of the screen, g,
S = weight of the test specimen, g,
B = weight of the screen and the residue in the reducing varnish (if used), g,
w' = weight of the screen used for the blank, g, and
S' = weight of the specimen of reducing varnish, g.

10. Report

10.1 Report the following information:

10.1.1 Sample identification and type of dispersion,

10.1.2 Percent or ppm of coarse particles as the mean of two determinations,

10.1.3 The specimen size,

10.1.4 The nature of the washout solvent and the reducing varnish (if used), and

10.1.5 Qualitative information, if obtained, about the nature of the coarse particles.

11. Precision and Bias [6]

11.1 *Precision*—An interlaboratory study of this test method was conducted in which two operators in each of four laboratories tested in duplicate on each of two days three

[6] Supporting data are available from ASTM International Headquarters. Request RR:D1–1075.

pigment flushes ranging in concentration of coarse particles from about 40 to 90 ppm. The same lot of washout solvent (Type I mineral spirits) and reducing varnish were distributed to all participants. The test data were analyzed in accordance with Practice E 691. One laboratory was considered an outlier and was deleted from the analysis. The within-laboratory pooled standard deviation was 18.6 ppm, and the between-laboratories pooled standard deviation was 23.4 ppm. Based on these standard deviations, the following criteria should be used to judge the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each of mean of two runs obtained by one operator, should be considered suspect if they differ by more than 82 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of two runs obtained by other operators in the same or different laboratories, should be considered suspect if they differ by more than 128 % relative.

11.2 *Bias*—Bias cannot be determined as there are no standard materials.

12. Keywords

12.1 coarse particles; dispersions; pigment dispersions; printing inks; wire-cloth sieve