Designation: D5909 – 20

Standard Test Method for Drying Time of Oxidative-Drying Printing Inks by Squalene Resistance[1]

This standard is issued under the fixed designation D5909; 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 procedure for determining the drying time of oxidative-drying printing inks (also referred to as "sheetfed inks") by squalene resistance of printed ink films.

1.2 This test method is applicable to all paste inks that dry primarily by oxidation regardless of the substrate on which they are printed. With appropriate changes in the test fluid, it may also be used with paste inks that dry by other mechanisms, such as heatset or ultraviolet light.

1.3 This test method utilizes a modified rub tester and is intended to serve as a "referee" procedure when laboratories, using less rigorous test procedures (see Appendix X1), cannot agree on their results.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing

D5264 Practice for Abrasion Resistance of Printed Materials by the Sutherland Rub Tester

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *blanket, n*—a composite structure, normally a rubber-like layer supported by one or more fabric or foam layers, or both.

3.1.2 *squalene oil, n*—an organic liquid ($C_{30}H_{50}$ unsaturated aliphatic hydrocarbon) that imitates the action of skin oil.

3.1.3 *Discussion*—Squalene dissolves vehicle components in a typical oxidative-drying ink system but not when fully polymerized.

4. Summary of Test Method

4.1 The test print is affixed to the base of the rub tester. The removable one-half pound weight of the instrument is fitted with a piece of rubber blanket and covered with an absorbent towel to which a small amount of squalene oil has been added. The weighted, oil-impregnated towel is allowed to rub back and forth over the test print for a prescribed number of cycles.

4.2 The receptor towel is examined for evidence of ink transfer from the print. Results may be related qualitatively to an agreed upon standard, or, the intensity of the stain can be measured quantitatively with a colorimeter, spectrophotometer, or other reflectance type device.

5. Significance and Use

5.1 Inadequate setting or drying of sheetfed inks, or both, can cause blocking of stacked prints with subsequent loss of product. "Setting" of an ink refers to the rapid rise in viscosity of the printed ink film, sometimes accompanied by surface drying, that prevents transference of the undried ink film to adjacent surfaces under light pressure. "Setting" is a property of the ink-substrate combination. Inadequate drying may be due to several factors, but the primary causes are: (*1*) omission of metallic driers from the ink, improper ink formulation (*2*) unusual ink-substrate interactions, and (*3*) use of a fountain solution that is too acidic. If the test prints are made on a

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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.

standard laboratory proof press, where there is no application of fountain solution, then only the first three possible causes can be evaluated. If the prints have been made on a commercial production printing press or some other acceptable means of introducing the fountain solution into the ink agreed upon, then the effect of fountain solution can be determined. It will often be necessary to run several tests to isolate the specific cause of a drying problem.

5.2 This test method is suitable for most combinations of oxidative drying inks and substrates. Because this test method relies on a visual assessment of the extent of drying, very light colors and clear varnishes may present difficulties in quantifying the extent of drying. In such cases, the supplier and the customer should agree upon an alternative method of assessing the drying properties of the ink.

6. Apparatus

6.1 *Rub Tester,* conforming to Test Method D5264 but equipped with a special one-half pound weight and modified as in Section 9.

6.2 *Laboratory Printing Apparatus,* capable of preparing test prints from a blanket or rubber covered-printing disk (that is, by the dry offset mode). The minimum print size is about 47 by 115 mm.

6.3 *Reflection Densitometer,* for use in controlling the ink density of the printed specimens.

6.4 *Spectrophotometer (Optional),* for measuring the reflectance of receptor toweling.

7. Materials

7.1 *Soft Toweling,* cut to fit the sub tester receptor block 50 by 140 mm.

7.2 *Squalene Oil,* [3]

7.3 *Rubber Blanket,* [4] preferably having an adhesive or "sticky" backing, cut to 38 by 38 mm.

7.4 *Substrate,* such as paper, paperboard or other as agreed upon between the supplier and the customer. The substrate should be cut to a size appropriate to the printing apparatus (6.2).

7.5 *Reference Standards (optional),* such as wet production or laboratory prints of inks and substrates having known or desired drying properties.

8. Test Specimen and Conditioning

8.1 Set the rub tester on a sturdy bench, preferably in a room conditioned at 23 ± 1°C (73.4 ± 2°F) and 50 ± 2 % relative humidity, as described in Practice D4332.

9. Preparation of the Rub Tester

9.1 Remove the one-pound weight of the rub tester and replace with the special half-pound weight. Affix the 38-mm square rubber blanket (7.3) to the center portion of the weight.

9.2 Remove the rubber pad from the lower surface of the instrument. Replace with a glass plate of comparable size.

9.3 In order to facilitate proper placement of the test specimen, determine where the center stroke occurs and scribe, or otherwise indicate, a line on the glass plate. Similarly, scribe lines to facilitate placement of the specimen in the left-to-right direction.

10. Procedure

10.1 Using the manufacturer's instructions, ink up the laboratory printing apparatus and pull a print on the agreed upon substrate. Measure the optical density. If not within ±0.05 of the desired value, discard and apply more or less ink to the print maker. Prepare a sufficient number of prints so that, after cutting, there will be a minimum of six specimens each about 47 by 115 mm. Record the time of printing on each specimen.

NOTE 1—The wide rubber disk of the bench type print tester produces a printed strip 47-mm wide and 230-mm long; by cutting in half cross-wise, two 47 by 115 mm specimens per print are obtained. The plate of a common flat-bed proof press is 102 by 153 mm and also gives two 47 by 115 mm specimens per print. In each case, three prints are needed to provide six test specimens.

10.2 A few minutes before an hour has elapsed, clamp a single thickness piece of toweling to the receptor block of the rub tester. With a pipet or eye dropper, place five drops of squalene oil on the towel in the region that lies over the rubber blanket. Using the thumb, work the oil into the toweling so that it is fully saturated, but not dripping in the region over the rubber blanket.

10.3 Center a specimen on the glass plate and tape down. One hour after the print was made, place the weight in its location on top of the specimen and turn the instrument on. Conduct the test for 15 strokes (a stroke consists of one back and forth cycle).

10.4 Remove the specimen and receptor towel. Carefully blot off any excess oil. Mark the number of hours that have elapsed between the time of printing and that of rub testing.

10.5 If inspection (11.1) indicates the print is not dry, set the print and receptor towel aside, and repeat 10.2 to 10.4 on another print specimen at 1-h (or other agreed upon) intervals after printing. If the ink has not completely dried at the end of 6 h, then the time interval must be extended by starting from 10.1 and running the tests at longer intervals or longer times.

11. Evaluation

11.1 Visually inspect the print for evidence of adverse effects and the receptor towel for evidence of ink transfer.

11.2 (Optional) Set the spectrophotometer to the following condition: Illuminant C, large aperture, 2° observer. Determine the reflectance of the receptor towels as a function of time, using an oil-soaked towel as the 100 % reflectance standard. Plot results in the form of L* versus time, noting on the graph the points at which visual drying (11.1) occurred. Drying can be considered complete when L* = 80 %.

[3] Available from chemical supply houses.
[4] Suitable rubber blanket is available from local graphic art supply houses.

12. Report

12.1 Report the time in hours at which there is no visible effect on the test print or the L* value of the receptor towel reaches 80 %.

12.2 If a reference standard was used, report whether the drying time of the test sample was shorter, equal to, or longer than the standard.

13. Precision and Bias[5]

13.1 *Precision*—An interlaboratory study of this test method was conducted in which 5 laboratories tested 3 inks ranging in drying time from about 2 to 5 h. The overall standard deviation was 0.74 h. Based on this result, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

13.1.1 *Repeatability*—Repeatability cannot be determined because the tests were not replicated.

13.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 2 h.

13.2 *Bias*—Bias cannot be determined because there are no standard materials.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1097. Contact ASTM Customer Service at service@astm.org.

14. Keywords

14.1 drying; oxidative drying; paper; paperboard; printing ink; squalene resistance

APPENDIX

(Nonmandatory Information)

X1. MANUAL METHOD

X1.1 The generally accepted industrial practice for determining "drying" of inks involves wiping prints, prepared similarly to the procedure described in this test method, with cotton swabs or balls that have been impregnated with an appropriate fluid at various times.

X1.2 As part of the interlaboratory round-robin conducted in 13.1, four of the participating laboratories concurrently measured the drying time of the same ink/paper combination using the procedure outlined in X1.1. Test results obtained by the various operators differed by as much as 5 h, well beyond the 2 h cited in 13.1.2.

X1.3 The manual procedure is very operator sensitive due primarily to the variation in applied pressure when wiping the print. The present method, involving a rub tester, serves to standardize the applied force and wiping action.