Designation: D7244 – 06a (Reapproved 2011)

Standard Test Method for Relative Cure of Energy-Cured Inks and Coatings[1]

This standard is issued under the fixed designation D7244; 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 describes the procedure for evaluating the relative cure of printed energy-cured (ultraviolet or electron beam) ink or coating by a mechanical solvent rub test using a motorized Crockmeter.

1.2 This test method is applicable to laboratory and production prints on any flat substrate that is no thicker than 3 mm (0.125 in.), durable enough to withstand the test conditions, and for which a control (reference) sample is available.

1.3 This test method applies to comparisons between energy-cured inks and coatings of the same chemistry and film weight and should not be used to compare different ink or coating chemistries or various applied film weights without first establishing process performance.

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

2. Referenced Documents

2.1 *Other Standards:*

AATCC Test Method 8 Colorfastness to Crocking: AATCC Crockmeter Method [2]

3. Terminology

3.1 *Definitions:*

3.1.1 *break-through, n*—the point where the ink or coatings film is penetrated by the solvent rub, causing a loss of density.

3.1.2 *cure, n*—the condition of a test sample after conversion to a dried film as measured by tests generally related to end-use performance and mutually agreeable to supplier and purchaser.

3.1.3 *electron beam (EB) curing, n*—conversion of an ink or coating to its final polymerized state by means of a mechanism initiated by electron beam radiation.

3.1.4 *ultraviolet (UV) curing, n*—conversion of an ink or coating to its final polymerized state by means of a mechanism initiated by ultraviolet radiation.

4. Summary of Test Method

4.1 The Crockmeter is set to a predetermined number of rub cycles. The test specimen is attached to the bed of the instrument. The linen covered rubbing finger is laid on the test print. Solvent is applied and the rub cycle started.

4.2 The test specimen is removed, examined for break-through and rated as less, equal, or more than the control.

5. Significance and Use

5.1 This test method is used as a manufacturing or laboratory process control tool by providing a visual comparison of the ability to resist solvent break-through against an established control. The test method is designed to indicate a potential problem caused by an undercured condition but does not identify what caused the condition.

5.2 This test method does not duplicate the conditions on a printing press but does provide a means to determine whether the test sample meets specifications as agreed upon between supplier and customer.

6. Apparatus

6.1 AATCC Motorized Crockmeter[3] equipped with a lightweight aluminum sliding arm that has a nominal weight of 250 g and hollow nylon finger. See Fig. 1 and Fig. 2.

6.2 Ultraviolet or electron beam curing equipment.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

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[2] Available from American Association of Textile Chemists and Colorists (AATCC), P.O. Box 12215, Research Triangle Park, NC 27709, http://www.aatcc.org.

[3] The sole source of supply of the apparatus know to the committee at this time is the Atlas Material Testing Technology LLC, 4114 N. Ravenswood Ave., Chicago, IL 60613 USA. If you are aware of alternate suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at the meeting of the responsible technical committee[1] which you may attend.

FIG. 1 AATCC Motorized Crockmeter Model CM-5

7. Reagents and Materials

7.1 *Control (reference) prints* consisting of energy cured prints that have met all quality control tests and end-use requirements. The control may be a production or laboratory print.

7.2 *Test cloth,* white linen 50 mm^2 (2 $in.^2$) as specified in AATCC Method 8.

7.3 *Solvents:*

7.3.1 Methyl ethyl ketone (MEK).

7.3.2 Isopropyl alcohol 99 % (IPA).

8. Hazards

8.1 Provide adequate ventilation, consistent with accepted laboratory practice, to limit accumulation of solvent vapors.

9. Test Specimen

9.1 This test method does not cover the preparation of print samples. The test print and the control print need to be made at the same film weight from inks or coatings of the same chemistry and similar color. Darker colors and heavier film weights are more difficult to cure and different chemistries may show different solvent rub resistance at the same degree of cure.

9.2 The ideal size of the test specimen is 178 mm (7 in.) long by 127 mm (5 in.) wide. Three tests can be run on this size of print. Alternatively, three specimens 38 mm (1.5 in.) wide by 127 mm (5 in.) long can be used.

9.3 Printed test specimens must have been exposed to an UV or EB energy source.

9.4 If the test print is a clear coating, the coating can be printed over on an ink film to help visual evaluation.

10. Preparation of Apparatus

10.1 Set up the Crockmeter in a ventilated area since solvents are being used.

10.2 Level the instrument if necessary.

10.3 Install the lightweight aluminum sliding arm. The reciprocating stroke length is positioned at the shortest stroke setting of 50 mm (2 in.).

10.4 Insert the hollow nylon finger into the sliding arm and lock into position. The solid end of the nylon finger should be facing the print surface. Position the top hole in the finger to allow easy access by the solvent filled eyedropper. Make sure the nylon finger is set level with the print surface and not at an angle. Otherwise, the rub test will not be uniform.

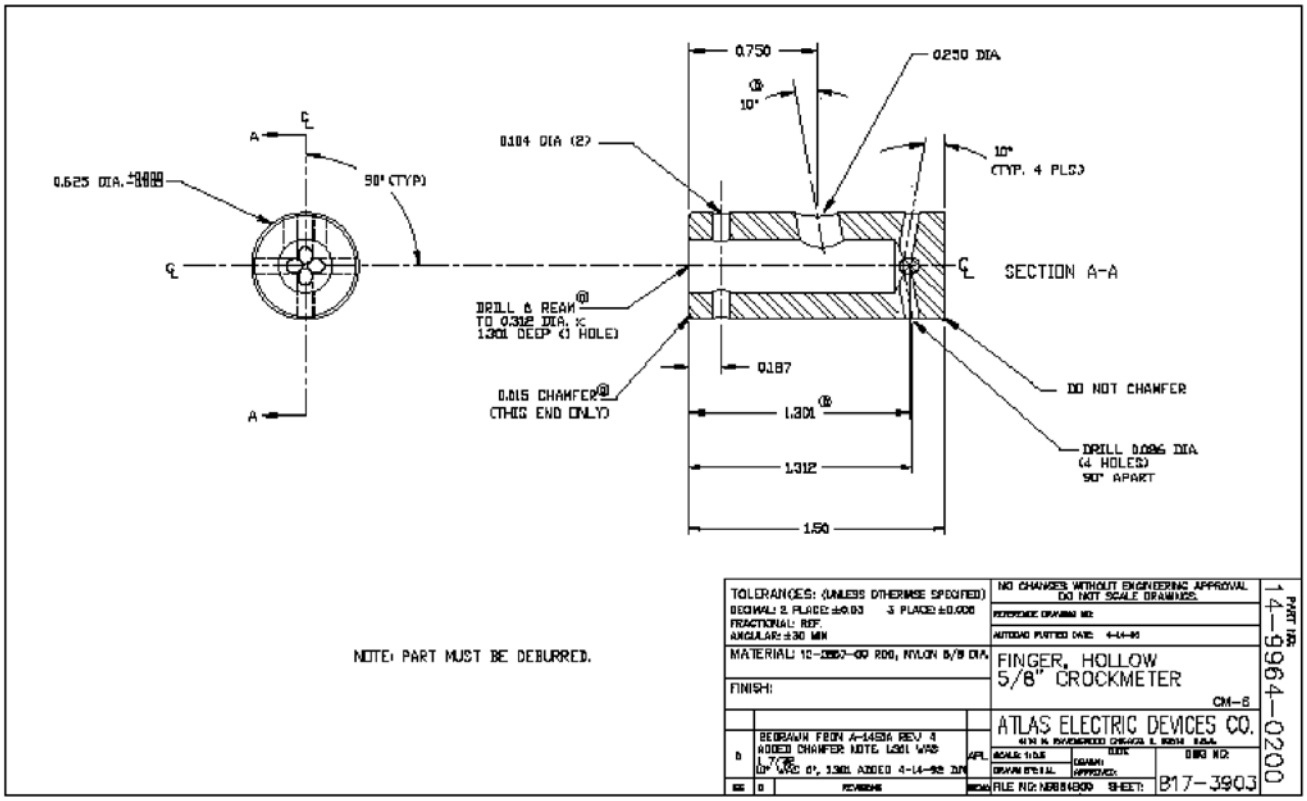

FIG. 2 Hollow Nylon Finger

10.5 Cover the hollow nylon finger with the 50 mm square linen cloth for each test to be run and secure with the spiral spring clip supplied by the manufacturer. Position the linen cloth so the hole in the nylon finger is exposed.

11. Preparation of Test Solutions

11.1 Prepare the solvent solutions in a fume hood with adequate ventilation.

11.2 For energy-cured inks, a starting point of 4 % MEK and 96 % IPA by volume is suggested. The ratio of solvents can be adjusted to provide the appropriate number of rub cycles (maximum 40) for the samples to be tested. The solvent can be changed to another type or concentration to meet the requirements of the ink chemistry as long as the desired results are achieved.

11.3 For energy-cured coatings, use 100 % MEK as the starting point. The solvent can be changed to another type or concentration as long as the desired results are achieved.

12. Standardization

12.1 Using the procedure in Section 13, establish the number of rub cycles needed for the control (reference) prints to exhibit break-through. It is important to ensure that the solvent does not completely evaporate before the rub test cycle is completed. A blend of 4 % MEK and 96 % IPA will provide 40 to 50 rubs at the 50.8 mm (2 in.) stroke setting for energy-cured inks. If 100 % MEK is used for energy-cured coatings, the solvent may evaporate faster than the number of required rubs. Stop the test before the solvent has completely evaporated. Add another 1 mL of solvent and continue the test until the desired number of rubs is achieved.

13. Procedure

13.1 Set the counter on the Crockmeter to the number of rub cycles determined under 12.

13.2 Center the nylon finger by moving the motor swing arm until it faces down between the bottom brackets. This centers the stroke.

13.3 Place the test sample under the linen covered finger and center it. The sample can be taped down to prevent movement during the run cycle or it can be held down by hand.

13.4 Transfer the appropriate test solution to a 10 mL graduated cylinder, and using an eyedropper, extract a maximum of 1 mL test solution. (Less test solution, 0.8 mL, has been found to give similar results and produces less solvent spread.) Inject the solution into the hole at the top of the finger, and turn on the instrument. Do not exceed two seconds for this operation because solvent comes out quickly through four small holes in the bottom of the nylon finger. Solvent must be present during the entire rub cycle.

13.5 When the test is complete, shut off the instrument. The test sample can be moved over to either side by 38 mm (1.5 in.) to allow two more tests to be run if necessary. Approximately 25.4 mm (1 in.) in width is needed for each test to allow for solvent migration.

13.6 Repeat steps 13.1 through 13.5 to continue the test cycle.

13.7 When the test cycle is complete, remove the test sample for visual assessment.

14. Interpretation of Results and Report

14.1 Examine the test sample for a decrease in density (break-through). Report the results as equal to, worse than, or better than the control.

14.2 If the break-through is equal to the control, the degree of cure is similar and is expected to meet end-use requirements. Break-through worse than the control indicates the cure may not be sufficient to meet requirements and the cause of the poor cure needs to be investigated. Less break-through than the control indicates an overcure condition may exist or that a new control standard may need to be established.

15. Precision and Bias

15.1 An interlaboratory study was conducted in which seven laboratories tested 12 print samples of energy-cured ink and coating in quadruplicate. Visual comparisons were conducted against a set of reference standards. Out of 336 tests only 32 were misidentified. Most of the error occurred with two coated samples. However, since the test is non-quantitative, it is not possible to compute repeatability, reproducibility, and bias.

16. Keywords

16.1 coatings; Crockmeter; cure test; curing; electron beam; printing inks; solvent rub; ultraviolet