Designation: D 4942 – 89 (Reapproved 2001)

Standard Test Methods for Water Pickup of Lithographic Printing Inks and Vehicles in a Laboratory Mixer[1]

This standard is issued under the fixed designation D 4942; 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 These test methods cover two procedures for determining the amount of water picked up by lithographic printing inks in a laboratory mixer.

1.2 Test Method A covers single-point water pickup; Test Method B covers the rate of water pickup. Both test methods are applicable to any printing ink and vehicle intended for the lithographic printing process.

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. Summary of Test Methods

2.1 These test methods utilize a laboratory mixer for beating water or other agreed upon fluid into the test ink.

2.2 For single-point water pickup (Test Method A), 50 mL of water is normally added to 50 g of ink and mixed in for 5 min. The water picked up is determined from volumetric measurements of free water.

2.3 For rate of water pickup (Test Method B), water is added to 50 g of ink in increments of 20 mL and mixed in for 1 min or more over a cumulative time period totaling 10 min. The water taken up by the ink after each mixing interval is determined gravimetrically.

3. Significance and Use

3.1 The lithographic printing process requires that some dampening solution be emulsified into the ink. These test methods provide a rapid means for determining water pickup under laboratory conditions. Test results may be useful for specification acceptance between the supplier and the customer.

3.2 In order that results be comparable, the tests must be run at the same temperature and with the same type and quantity of liquid added prior to mixing.

3.3 The emulsions obtained in these test methods are of larger particle size than those typically produced in printing nips. Because of these and other variables in the printing process, water pickup results do not by themselves predict lithographic printing performance.

4. Apparatus

4.1 *Laboratory Mixer*,[2] such as a Duke Ink-Water Emulsification Tester[2] equipped with a stainless steel specimen bowl 83 mm wide and 88 mm high, mixer blades that rotate at 90 r/min, and a timing device.

4.2 *Balance*, accurate to 0.1 g, 600-g capacity.

4.3 *Palette knives*, two.

4.4 *Thermometer*, quick response.

4.5 *pH Meter* (optional).

4.6 *Conductivity Meter* (optional).

4.7 *Graduated Cylinder*, 50 or 100-mL.

5. Reagents and Materials

5.1 *Water*—Deionized or distilled water, preferably having a pH of 5.0 to 7.0 (100 to 200 mL per sample); alternatively, fountain solution or other aqueous medium as agreed upon between the supplier and the customer may be used.

5.2 *Cleanup Materials*—Naptha and rags or tissues.

6. Test Specimen

6.1 A minimum of 100 g is sufficient for two determinations. Before removing ink from the can, stir or otherwise ensure that the ink specimen is representative. Close the can and replace sealing tape immediately after each ink removal.

7. Conditioning

7.1 Condition the instrument, water, and ink samples in a constant temperature room or bath, preferably at 23 ± 1°C.

7.2 Prior to use, check the alignment of the mixer blades. With the power switch of the mixer in the off position, set the clean bowl into the turntable and engage the locking pin firmly into the slot in the side of the turntable. Tilt the mixer head back and insert the blades, marked left and right, into their respective holders. Lower the mixer head. If the blades hit the side or bottom of the bowl, return the instrument to the

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[2] Available from Duke Custom Systems, 8371 Highway 49, Pleasant View, TN 37146.

manufacturer for realignment.

8. Test Method A—Single Point Water Pickup (by Volumetry)

8.1 Program the counter of the mixer for 5 min mixing time (450 revolutions).

8.2 *Optional*—If the first run of the day, pour test water into a beaker. Measure pH, conductivity, and temperature at the beginning of testing.

8.3 Weigh or tare the clean dry mixing bowl. Add 50 ± 0.1 g of the ink to the center of the bowl.

8.4 Pour 50 mL of water (from 8.2) into a graduated cylinder. If the ink is expected to pick up more than 100 % water, use 100 mL of water. Adjust the volume to ±0.5 mL. Add the entire contents to the bowl.

8.5 With the mixer head tilted back insert the clean blades, marked left and right, into their respective holders. Lock the bowl on the turntable. Lower the mixer head. Press the counter reset button, making sure that 450 is displayed on the face of the counter.

8.6 Turn the mixer on. Examine contents of the bowl as mixing progresses. If 50 mL of water had been added and all of it disappears into the ink, stop, discard the ink in the bowl, clean up, and start over from 8.3, adding 100 mL of water in 8.4. The latter quantity must also be used for all other inks in the series under study.

NOTE 1—With some inks, water pickup is affected by the amount of water added prior to mixing. When 50 mL is insufficient, do not simply add another 50 mL during the run, as test results may differ significantly from those obtained by adding 100 mL at the outset.

8.7 When the mixer stops, turn the power switch off. Tilt the head out of the ink, detach the mixing blades, and add to the bowl.

8.8 Remove the bowl from the turntable and, holding the blades at the side of the bowl, decant the free water into a graduated cylinder. Run the blades *very slowly* through the ink in the bowl. Decant additional free water into the cylinder.

NOTE 2—Do not knock the bowl to force free water from the surface. Always handle the bowl gently to avoid breaking the emulsion.

8.9 Record the returned water level to 0.5 mL.

8.10 *Optional*—Measure the temperature, pH, and conductivity of the returned water. Note the appearance of the water and the consistency of the ink and the appearance of the returned water.

8.11 Discard ink left in the bowl. Clean the bowl and the mixer blades with tissue wetted with naphtha. Discard the returned water and rinse the cylinder clean.

8.12 Repeat 8.3 through 8.10 with a second specimen of the same ink.

9. Test Method B—Rate of Water Pickup (by Gravimetry)

9.1 Program the counter for the first interval of the mixing cycle.

NOTE 3—A commonly used cycle is 1-min intervals (90 revolutions) times ten determinations. Intervals need not be uniform, for example, 1, 2, 3, 5, and 10 min (90 times 3 plus 180 plus 450 revolutions).

9.2 *Optional*—Measure water properties in accordance with 8.2.

9.3 Weigh or tare the clean dry mixing bowl and blades on the balance. Add 50 ± 0.1 g of ink to the center of the bowl.

9.4 Lock the bowl on the platform of the mixer. With the mixer head raised, carefully insert the blades into their respective holders. If ink on one blade touches the upper parts of the other blade or the side of the bowl, carefully remove the ink with two palette knives and transfer to the bottom of the bowl. Lower the mixer head.

9.5 Pour 100 mL of water (from 8.2) into a beaker. Meter out 20 mL and add to the bowl.

9.6 Press the counter reset button, making sure that the desired number of revolutions is displayed on the face of the counter. Turn the mixer on. Examine the contents of the bowl as mixing progresses. If all liquid disappears into the ink, add more as needed to maintain a layer of excess water on the surface of the ink.

NOTE 4—Few specimens will take up more than 20 mL of water within a 1-min mixing interval. If a high-water pickup specimen is being run and the mixing interval is longer than 1 min, another 20 mL should be added prior to each subsequent minute of mixing time.

9.7 When the mixer stops, turn the power switch off. Detach the mixing blades and add to the bowl.

9.8 Remove the bowl from the turntable and, holding the blades at the side of the bowl, decant the free water into the beaker containing the unused water. Run the blades *very slowly* through the ink in the bowl. Decant additional free water into the beaker (see Note 2).

9.9 Weigh the mixing bowl and contents, including the blades.

9.10 Using a palette knife, transfer the ink from the walls to the center of the bowl. Return the bowl to the mixer. Replace the blades as in 8.4.

9.11 For the next mixing interval, swirl the beaker in order to mix the returned and unused water. Meter out 20 mL and add to the bowl. Press the counter reset (or change the counter) and turn the power on. Add more water if needed to maintain an excess layer (see Note 4).

9.12 When the mixer stops, repeat 9.7 through 9.11 until the cumulative mixing time totals at least 10 min.

9.13 *Optional*—At the end of the run, make measurements in accordance with 8.10.

9.14 Discard the ink left in the bowl. Clean the bowl and the mixer blades with tissue wetted with naphtha. Discard returned water and rinse the beaker clean.

9.15 Repeat 9.3 through 9.14 with another specimen of the same ink.

10. Calculation

10.1 Calculate water pickup, P, as follows:

10.1.1 *Test Method A—Volumetric*:

$$P = (V_1 - V_2) \times 2 \quad (1)$$

where:

P = water pickup, % or mL water/100 g ink,
V_1 = volume of water added, mL, and
V_2 = volume of returned water, mL.

10.1.2 *Test Method B—Gravimetric*:

$$P = (W - S) \times 2 \quad (2)$$

where:

P = water pickup, % or g water/100 g ink,
W = weight of the specimen plus water picked up after each mixing interval, g, and
S = weight of initial specimen, g.

NOTE 5—The conversion from water pickup of the ink to water content, C, of the emulsion is $C = P/(100 + P)$. Units are percent or grams of water per 100 grams of emulsion.

11. Report

11.1 Report the following information:

11.1.1 The percent water pickup to the nearest whole number as the mean of the two determinations, the cumulative mixing time, and a description of the water used for testing (for example, tap water, deionized water, or type of fountain solution).

11.1.2 If rate of water pickup was determined, plot the percent of water pickup versus the cumulative mixing time.

11.1.3 *Optional*—The mean temperature, changes in pH, conductivity, appearance of the water, and the change in consistency of the ink.

12. Precision and Bias

12.1 *Precision*:

12.1.1 *Test Method A*—An interlaboratory study of single-point water pickup by Test Method A was conducted in which one operator in each of eleven laboratories tested in duplicate on each of two days three lithographic printing inks ranging in 5-min water pickup from 50 to 65 %. One company was found to be an outlier and was deleted from the analysis. The within laboratory pooled standard deviation was found to be 1.58 % absolute (millilitre of water per 100 grams of ink) at 9 degrees of freedom (df), and the between laboratories pooled standard deviation was 7.1 % absolute at 30 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1.1 *Repeatability*—Two results, each the mean of two runs obtained by one operator, should be considered suspect if they differ by more than 4.5 % absolute.

12.1.1.2 *Reproducibility*—Two results, each the mean of two runs obtained by operators in different laboratories, should be considered suspect if they differ by more than 20 % absolute.

12.1.2 *Test Method B*—In an interlaboratory study of rate of water pickup by Test Method B, water pickup values at 2½, 5, 7½ and 10 min were determined twice on one day by one operator in each of nine laboratories on six inks. The inks ranged in water pickup from 40 to 52 % at 2½min and from 65 to 100 % at 10 min. After rejecting 12 out of 156 replicated test values as outliers, the within laboratory pooled standard deviation was found to be 1.58 % absolute (grams of water per 100 grams ink) with 97 df and the between laboratory standard deviation 3.73 % absolute with 86 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.2.1 *Repeatability*—Repeatability cannot be determined as both runs were conducted on the same day.

12.1.2.2 *Reproducibility*—Two water pickup curves, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 10.5 % absolute.

12.2 *Bias*—Bias cannot be determined because there are no standard materials. The poorer interlaboratory precision of Test Method A compared to Test Method B is believed to be caused by the fact that the gross quantity of water added at one time is picked up as large globules which make it difficult for different operators to release free water in the same manner.

13. Keywords

13.1 lithographic printing inks; printing inks; inks; vehicles; water pickup; water content; emulsification; fountain solution; mixers