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Standard Practice for Estimating Critical Surface Tensions¹

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1. Scope

- 1.1 This practice covers procedures for estimating values of the critical surface tension of surfaces by observing the wetting and dewetting of a series of liquids (usually organic solvents) applied to the surface in question.
- 1.2 Another technique, measurement of the contact angles, θ , of a series of test liquids and plotting $\cos \theta$ versus surface tension (Zisman plots), provides data that allow the determination of more exact values for critical surface tension.
- 1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.4 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:²

D2578 Test Method for Wetting Tension of Polyethylene and Polypropylene Films

2.2 Nordtest Standards:³

NT poly 176 Spreading Surface Tension by the Applied Droplet Method.

3. Terminology

- 3.1 Definitions:
- 3.1.1 *critical surface tension, n*—the surface tension of a hypothetical liquid that would just spontaneously spread if applied as a drop to the surface in question; any liquid with a

¹ This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

surface tension lower than the critical surface tension will spread spontaneously.

4. Summary of Practice

4.1 In this practice, a series of liquids of gradually increasing surface tension are applied to a surface in the form of drops, narrow strips, or spots. Drops may be applied using a dropper, syringe or other device capable of producing individual drops. Liquid strips or spots are applied to the surface by swabbing with saturated cotton swabs or by another type of applicator, such as one that is similar to a marker pen. In the case of the drop, the observer determines whether the drop stays in place or spreads. In the case of the liquid strip or spot, the question is whether the liquid stays in place or dewets and crawls. In each case, the break point between wetting and dewetting provides the critical surface tension.

5. Significance and Use

- 5.1 Knowledge of the critical surface tension of substrates, primers and other coatings is useful for explaining or predicting wettability by paints and other coatings applied to those surfaces. Surfaces with low critical surface tensions usually are prone to suffer defects such as crawling, picture framing, cratering and loss of adhesion when painted. Low or irregular values, or both, often are indicative of contamination that could reduce adhesion. Surfaces with high critical surface tensions are easy to wet and usually provide an excellent platform for painting.
- 5.2 The swab, marking pen and draw-down tests all simulate the application of a film
- 5.3 The swab and marking pen techniques are simple and rapid and are particularly useful for testing in the field or on curved, irregular or porous surfaces where contact angles cannot be measured. The drop test does not work well on such surfaces and the draw-down method requires a flat specimen that is relatively large.
- 5.4 The estimation of critical surface tension has been useful in characterizing surfaces before and after cleaning processes such as power washes and solvent wipes in order to evaluate the efficiency of the cleaning.
- 5.5 One or more of these techniques could be the basis of a go/no-go quality control test where if a certain liquid wets, the

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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.

³ For Nordtest standards, see www.nordicinnovation.net/nordtest.cfm or contact Nordtest, Tekniikantie 12, FIN-02150 Espoo, Finland.

TABLE 1 Test Liquids and Their Surface Tensions

Liquid	Surfact Tension mN/m (= dynes/cm)
Acetone	23.7
Dimethyl formamide	35.2
2-pyrollidone	37.6
N-methyl-2-pyrollidone	39.0
Dimethyl sulfoxide	43.0
Ethylene cyanohydrin	44.4
Formamide	56.0
Water	72.8

surface is acceptable for painting, but if that liquid retracts and crawls, the surface is not acceptable.

5.6 Another go/no go test is possible where the test liquid is a paint and the surface is a substrate, primer or basecoat. A form of this test has been used for coatings for plastics.

6. Interferences

- 6.1 The specimen must be clean in order for results to be meaningful. The surface must not be touched or rubbed.
- 6.2 The surface tensions of test liquids, especially those that are mixtures, are subject to change with time. Test liquid surface tensions should be confirmed before they are first used, measured periodically after that and whenever change is suspected.
- 6.3 Test liquids may become contaminated, in which case results with them will be meaningless. Test liquid surface tensions should be confirmed before they are first used, measured periodically after that and whenever contamination is suspected.
- 6.4 High ambient temperature may cause rapid evaporation of test liquids and make it difficult to determine whether retraction has occurred or shrinkage is due to evaporation.

7. Apparatus

- 7.1 Several of one of the following types of applicators:
- 7.1.1 *Cotton swab* (small ball of cotton at the end of a stick).
- 7.1.2 Marker pen type applicator.
- 7.1.3 Dropper or syringe.
- 7.1.4 Other device with the ability to apply a drop, strip, or spot.
 - 7.2 Drawdown Bar—only for draw-down test.
- 7.3 Instrument or devices for measuring temperature and humidity.

8. Materials

- 8.1 Swab, drop and draw-down tests.
- 8.1.1 Test liquids.
- 8.1.1.1 The original publication on the swab test⁴ specified the liquids in Table 1 (listed along with their surface tensions). This set of liquids has a large surface tension gap between 24 and 35 dynes/cm.
 - ⁴ Hansen, C. M., J. Coat. Technol., 44 (570), 57 (1972).

- 8.1.1.2 Test Method D2578 specifies mixtures of formamide and ethylene glycol monomethyl ether (both reagent grade). Mixtures can be designed so as to produce any combination of surface tension values.
- 8.1.1.3 The drop test publications⁵, 6 specify mixtures of ethanol and water, but do not give details of the proportions.
- 8.1.1.4 Nordtest Method NT poly 176³ includes a table of surface tensions for ethanol/water mixtures at 23°C, which gives values ranging from 22 to 72 mN/m in 2 mN/m intervals.
- 8.1.1.5 Other liquids covering a range of surface tensions could be used. If possible, highly volatile solvents should be avoided as it is difficult to tell whether they are dewetting or just evaporating.

9. Preparation of Specimens

- 9.1 The areas tested shall not contain visible blemishes or defects and shall not be touched with the fingers or be allowed to be contaminated in any way.
- 9.2 Specimens may be cleaned, bearing in mind that the surface of the specimen may be affected by the cleaning process. Any cleaning method should be chosen only after considering how the process may alter the surface. Cleaning must be clearly indicated in the report.

10. Procedure-Cotton Swab

- 10.1 Beginning with the lowest surface tension liquid in the test series, saturate the cotton ball at the end of the stick and apply three uniform strips 0.5 to 1 cm wide and 2 to 5 cm long to the test specimen.
- 10.2 Observe whether the liquid remains as an intact film, retracts (crawls) or completely beads up. Allow 3 seconds for retraction or beading to occur. Longer times may be used, but evaporation of the liquid may cause retraction that appears to be dewetting, but is not.
- 10.3 If the liquid strip does not retract or bead up within 3 seconds, repeat the procedure on an untested section of the specimen with a clean cotton swab and the next higher surface tension liquid. If the strip does retract or bead in the initial test, a lower surface tension test liquid must be found and used.
- 10.4 Each of the liquids in the series is used to test the specimen until one of them clearly retracts. There are two different conventions for determining the end point and estimating the critical surface tension:
- 10.4.1 The critical surface tension of dewetting is taken as being intermediate between the surface tension value of the last liquid that wet the surface and the first one that did not. For example, if a 34 mN/m liquid wets the specimen and a 36 mN/m liquid does not, the critical surface tension will be considered to be approximately 35 mN/m.
- 10.4.2 The critical surface tension is taken as the highest surface tension liquid that just wets the surface. Using this criterion, the critical surface tension in the example in 10.4.1 would be considered to be approximately 34 mN/m.

⁵ Hansen, C. M., European Coatings J., 94, 838 (1994).

⁶ Hansen, C. M., Pigment and Resin Technol., 27, 374 (1998).

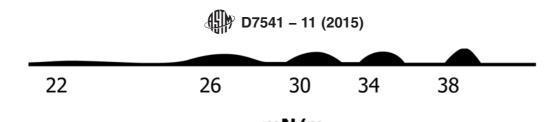


FIG. 1 Drops of Different Surface Tension Liquids on a Low Surface Tension Surface. Left to Right: Low to High Surface Tension Liquids; Spreading to Beading

11. Procedure-Marker Pen Applicator

- 11.1 Begin with the marker pen with the lowest surface tension.
- 11.2 Press the applicator tip firmly on the corner of the specimen until the tip is fully saturated with the ink.
- 11.3 Lightly draw the pen across the specimen in three parallel passes. One of the pen manufacturers specifies only evaluating the last pass.
- 11.4 If the last ink strip remains wetted for 3 seconds or more, repeat steps 11.2 and 11.3 on an untested portion of the specimen with the next higher surface tension marker pen.
- 11.5 Continue testing until the paint strip clearly retracts. As with the cotton swab test, there are two conventions for determining the end point and estimating the critical surface tension. See 10.4.1 and 10.4.2 for details.

12. Procedure-Drop Test

- 12.1 Beginning with the lowest surface tension test liquid, place a small drop of the test liquid on the test specimen.
- 12.2 Observe whether the liquid spreads. If it does, apply a drop of the next higher surface tension liquid.
- 12.3 Continue with higher and higher surface tension liquids until a drop does not spread.
- 12.4 See 10.4.1 and 10.4.2 for the two conventions used in estimating the critical surface tension from the observations.
- 12.5 An example is shown in Fig. 1. The surface has a critical surface tension between 22 and 26 dynes/cm. Liquids with surface tensions equal to or higher than 26 dynes/cm do not wet the surface and, therefore, retain their droplet form.

13. Procedure-Draw-Down Test

13.1 A flat specimen, preferably on the order of 20 by 25 cm is placed on and clamped to a work bench or table.

- 13.2 Several test liquid drops are placed across the top of the specimen with space between them. The liquids should range from low to high surface tension, increasing from left to right. A draw-down bar is placed above the drops and pulled down. The resultant draw-downs are quickly examined for wetting and dewetting behavior.
- 13.3 As with the other methods, there should be a break point between the draw-down strips that wet and those that dewet and retract.

14. Report

- 14.1 The report shall contain the following information when applicable and available.
 - 14.1.1 Date, time, and place of the measurement.
 - 14.1.2 Description of the test specimen.
- 14.1.3 Whether specimen is as-received or whether cleaning has been done. If the latter, then describe the cleaning process.
 - 14.1.4 Temperature and humidity in the area of the test.
- 14.1.5 Identification of the test and applicator that were used.
 - 14.1.6 If the drop test is used, indicate the drop size.
 - 14.1.7 Any deviations from the method.
- 14.1.8 Identification of which convention was used to estimate the critical surface tension.
- 14.1.9 The estimated value for the critical surface tension in mN/m.

15. Keywords

15.1 critical surface tension; dewetting; surface contamination; wettability; wetting

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