



# Standard Test Method for Wetting Tension of Polyethylene and Polypropylene Films<sup>1</sup>

This standard is issued under the fixed designation D 2578; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method covers the measurement of the wetting tension of a polyethylene or polypropylene film surface in contact with drops of specific test solutions in the presence of air.

1.2 *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. Specific hazards statements are given in Section 8.*

NOTE 1—This test method is equivalent to ISO 8296.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D 618 Practice for Conditioning Plastics for Testing

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

### 2.2 ISO Standard:

ISO 8296<sup>3</sup>

## 3. Summary of Test Method

3.1 In this test method drops of a series of mixtures of formamide and ethyl Cellosolve<sup>4</sup> of gradually increasing surface tension are applied to the surface of the polyethylene or polypropylene film until a mixture is found that just wets the film surface. The wetting tension of the polyethylene or polypropylene film surface will be approximated by the surface tension of this particular mixture.

## 4. Significance and Use

4.1 When a drop of liquid rests on the surface of a solid, and a gas is in contact with both, the forces acting at the interfaces

must balance. These forces can be represented by surface energies acting in the direction of the surfaces and it follows that:

$$\gamma_{GL} \cos \theta = \gamma_{GS} - \gamma_{SL} \quad (1)$$

where:

$\theta$  = angle of contact of the edge of the drop with the solid surface,

$\gamma_{GL}$  = surface energy of the gas - liquid interface,

$\gamma_{GS}$  = surface energy of the gas - solid interface, and

$\gamma_{SL}$  = surface energy of the solid - liquid interface.

4.1.1 The right side of the above equation (the difference between the surface energies of the gas - solid and solid - liquid interfaces) is defined as the wetting tension of the solid surface. It is not a fundamental property of the surface but depends on interaction between the solid and a particular environment.

4.1.2 When the gas is air saturated with vapors of the liquid,  $\gamma_{GL}$  will be the surface tension of the liquid. If the angle of contact is  $0^\circ$  the liquid is said to just wet the surface of the solid, and in this particular case (since  $\cos \theta = 1$ ) the wetting tension of the solid will be equal to the surface tension of the liquid.

4.2 The ability of polyethylene and polypropylene films to retain inks, coatings, adhesives, etc., is primarily dependent upon the character of their surfaces, and can be improved by one of several surface-treating techniques. These same treating techniques have been found to increase the wetting tension of a polyethylene or a polypropylene film surface in contact with mixtures of formamide and ethyl Cellosolve in the presence of air. It is therefore possible to relate the wetting tension of a polyethylene or a polypropylene film surface to its ability to accept and retain inks, coatings, adhesives, etc. The measured wetting tension of a specific film surface can only be related to acceptable ink, coating, or adhesive retention through experience. Wetting tension in itself is not a completely acceptable measure of ink, coating, or adhesive adhesion.

NOTE 2—A wetting tension of 35 dynes/cm or higher has been generally found to reveal a degree of treatment normally regarded as acceptable for tubular film made from Type 1 polyethylene and intended for commercial flexographic printing. It is, however, possible that some other level of wetting tension may be required to indicate the acceptability of polyethylene films made by other processes, or from other types of polyethylene, or intended for other uses.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film and Sheeting.

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<sup>2</sup> 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.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

<sup>4</sup> Registered trademark of Union Carbide Corp. for ethylene glycol monoethyl ether.

\*A Summary of Changes section appears at the end of this standard.

At the present date, insufficient experience has been gained to state a generally acceptable level of wetting tension for polypropylene films for commercial flexographic printing.

## 5. Interferences

5.1 Since the wetting tension of a polyethylene or polypropylene film in contact with a drop of liquid in the presence of air is a function of the surface energies of both the air - film and film - liquid interfaces, any trace of surface-active impurities in the liquid reagents or on the film may affect the results. It is, therefore, important that the portion of the film surface to be tested not be touched or rubbed, that all equipment be scrupulously clean, and that reagent purity be carefully guarded. Glass apparatus in particular is likely to be contaminated with detergents having very strong surface tension reducing ability unless specific precautions are taken to ensure their absence such as cleaning with chromic-sulfuric acid and rinsing with distilled water.

## 6. Apparatus

- 6.1 *Cotton-Tipped Wooden Applicators*, approximately 150 mm [6 in.] in length.
- 6.2 *Burets*, two, 50-mL.
- 6.3 *Bottles*, 100-mL, with caps and labels.

## 7. Reagents and Materials

7.1 Prepare mixtures of reagent grade formamide ( $\text{HCONH}_2$ ) and reagent grade ethyl Cellosolve ( $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2\text{OH}$ ) in the proportions shown in Table 1 for the integral values of wetting tension in the range over which measurements are to be made. For extremely precise work, mixtures for determining fractional values of wetting tension may be made up by interpolating between the concentrations shown in Table 1.

**TABLE 1 Concentration of Ethyl Cellosolve—Formamide Mixtures Used in Measuring Wetting Tension of Polyethylene and Polypropylene Films**

Formamide, Volume %	Ethyl Cellosolve, <sup>A</sup> %	Wetting Tension, <sup>B</sup> dynes/cm
0	100.0	30
2.5	97.5	31
10.5	89.5	32
19.0	81.0	33
26.5	73.5	34
35.0	65.0	35
42.5	57.5	36
48.5	51.5	37
54.0	46.0	38
59.0	41.0	39
63.5	36.5	40
67.5	32.5	41
71.5	28.5	42
74.7	25.3	43
78.0	22.0	44
80.3	19.7	45
83.0	17.0	46
87.0	13.0	48
90.7	9.3	50
93.7	6.3	52
96.5	3.5	54
99.0	1.0	56

<sup>A</sup> Cellosolve is the registered trademark of Union Carbide Corp. for ethylene glycol monoethyl ether.

<sup>B</sup> Measured under conditions of  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity.

NOTE 3—Although the mixtures of ethyl Cellosolve and formamide used in this test method are relatively stable, exposure to extremes of temperature or humidity should be avoided.

7.2 If desired, add to each of the formamide ethyl Cellosolve mixtures a very small amount of dye of high tinctorial value. The dye used should be of such color as to make drops or thin films of the solutions clearly visible on the surface of polyethylene or polypropylene film and must be of such chemical composition that it will not measurably affect the wetting tension of the solutions in the concentration used.<sup>5</sup>

7.3 Fully prepared mixtures of these reagents in varying concentrations are available from a number of commercial sources. Pens containing these mixtures are also commercially available. However, correlations between these options and freshly prepared solutions have not been established.

## 8. Hazards

8.1 Formamide may cause skin irritation and is particularly dangerous in direct contact with the eyes. Safety goggles should be worn when making up new test mixtures.

8.2 Ethyl Cellosolve is a highly flammable solvent.

8.3 Both ethyl Cellosolve and formamide are toxic and pose exposure risks. Consult the applicable MSDS before use and ensure that the appropriate Personal Protective Equipment (PPE) is used, especially when preparing new solutions.

8.4 All tests should be performed with adequate ventilation.

## 9. Sampling

9.1 The minimum amount of film required for this test depends upon the skill of the operator. This usually amounts to one sample across the entire width of a roll in order to obtain a representative value.

9.2 Extreme care must be taken to prevent the surface of the film sample from being touched or handled in the areas upon which the test is to be made.

9.3 Usually it will be adequate to make one determination at each location  $\frac{1}{4}$ ,  $\frac{1}{2}$ ,  $\frac{3}{4}$  of the way across the width of the film, to arrive at an average value for the sample or to determine when treatment is uneven.

## 10. Conditioning

10.1 *Conditioning*—Condition the test specimens at  $23 \pm 2^\circ\text{C}$  [ $73.4 \pm 3.6^\circ\text{F}$ ] and  $50 \pm 5\%$  relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be  $\pm 1^\circ\text{C}$  [ $\pm 1.8^\circ\text{F}$ ] and  $\pm 2\%$  relative humidity.

10.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of  $23 \pm 2^\circ\text{C}$  [ $73.4 \pm 3.6^\circ\text{F}$ ] and  $50 \pm 5\%$  relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the tolerances shall be  $\pm 1^\circ\text{C}$  [ $\pm 1.8^\circ\text{F}$ ] and  $\pm 2\%$  relative humidity.

<sup>5</sup> "DuPont Victoria Pure Blue BO" at a maximum concentration of 0.03 % has been found satisfactory.

## 11. Procedure

11.1 Wet the very tip of a cotton applicator with one of the mixtures. Use only a minimum amount of liquid as an excess of reagent can affect the end point of the test.

11.2 Spread the liquid lightly over an area of approximately  $6.5 \text{ cm}^2$  [1 in.<sup>2</sup>] of the test specimen. Do not try to cover a larger area lest there be insufficient liquid to give complete coverage.

11.3 Note the time required for the continuous film of liquid formed in 11.2 to break up into droplets. If the continuous film holds for 2 s or more, proceed to the next higher surface tension mixture, but if the continuous film breaks into droplets in less than 2 s, proceed to the next lower surface mixture. A clean, new cotton applicator must be used each time to avoid contamination of the solutions (even for successive dips into the same solution).

NOTE 4—The solution is considered as wetting the test specimen when it remains intact as a continuous film of liquid for at least 2 s. The “reading” of the liquid film behavior should be made in the center of the liquid film. Shrinking of the liquid film about its periphery does not indicate lack of wetting. Breaking of the liquid film into droplets within 2 s indicates lack of wetting. Severe peripheral shrinkage may be caused by too much liquid being placed upon the film surface. Experience with the test will give excellent insight into “reading” the liquid film behavior.

11.4 Proceeding in the direction indicated by the results of 11.3, continue repeating 11.1 through 11.3 until it is possible to select the ethyl Cellosolve-formamide mixture that comes nearest to wetting the film surface for exactly 2 s. The surface tension of this mixture in dynes per centimetre is called the wetting tension of the polyethylene or polypropylene film specimen.

NOTE 5—Since the surface tension of the formamide/ethyl cellosolve solutions can change, for example, through evaporation of one or both components, or through contamination, and is dependent upon temperature, the actual surface tension of the solution that wet the film for exactly 2 s should be measured. This can be accomplished very simply with a surface tensiometer.<sup>6</sup>

11.5 Experience with this test has shown that on occasion erroneous wetting tension results can be obtained when the final wetting tension is determined by working progressively to lower surface tension mixtures when practicing 11.1-11.4. It is recommended that the test analyst should check the reported wetting tension of the film by working progressively to higher surface tension mixtures.

## 12. Report

12.1 Report the average value of the wetting tension of each sample to the nearest 0.5 dynes/cm.

12.2 If the polyethylene or polypropylene film has been unevenly treated, it may not be possible to arrive at a single value of wetting tension. In this case, report the individual values whenever the largest value exceeds the smallest by more than 1 dyne/cm.

## 13. Precision and Bias

### 13.1 Precision:

13.1.1 Table 2 is based on an interlaboratory study conducted in 1995 in accordance with Practice E 691 involving three materials and thirteen laboratories<sup>7</sup>. Each test result was the average of three individual determinations. Each laboratory obtained three test results for each material on two days.

13.1.2 The values of the repeatability standard deviation  $S_r$ , reproducibility standard deviation  $S_R$ , the 95 % repeatability and reproducibility limits on the difference between two test results,  $r$  and  $R$ , respectively, for different treatment levels have been determined and are shown in Table 2. The results demonstrate that the precision of the results depends on the treatment level (wetting tension) of the film, the higher the treatment level, the higher the variability between single test results obtained in different laboratories, or the lower the precision.

### 13.1.3 Definitions:

**Warning**—The following explanations of  $r$  and  $R$  are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the interlaboratory study and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles 13.1.3-13.2 would then be valid for such data.

13.1.3.1  $S_r$  = the within laboratory standard deviation for the individual material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories.

13.1.3.2  $S_R$  = the between-laboratories reproducibility, expressed as standard deviation.

<sup>6</sup> The Fisher Surface Tensiometer, Model 20, has been found satisfactory for this application.

<sup>7</sup> Supporting data are available from ASTM Headquarters, Request RR:D20-1009.

**TABLE 2 Interlaboratory Study Results.**

Material	Treatment Power, W/ft <sup>2</sup> /min	Average Wetting Tension, dyne/cm	Repeatability Standard Deviation, $S_r$	Reproducibility Standard Deviation, $S_R$	$r$	$R$
PP film	20	39.45	0.74	1.97	2.06	5.52
PP film	1.6	36.27	0.32	1.85	0.90	5.19
PP film	0.5	33.07	0.38	0.87	1.06	2.43

13.1.3.3  $r$  = the within-laboratory critical interval between two results =  $2.8 \times S_r$ . The two test results should be judged not equivalent if they differ by more than the  $r$  dyne/cm for that treatment level.

13.1.3.4  $R$  = the between-laboratories critical interval between two results =  $2.8 \times S_R$ . The two test results should be judged not equivalent if they differ by more than the  $R$  dyne/cm for that treatment level.

13.1.3.5 Any judgment in accordance with 13.1.3.1 and 13.1.3.2 would have an approximate 95 % (0.95) probability of being correct.

13.2 *Bias*—No statement can be made about the bias of this test method because wetting tension is defined in terms of the test method.

## SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

*D 2578 - 04:*

- (1) Added references to commercial mixtures and pens in 7.3.
- (2) Modified hazard statement in 8.3.

(3) Corrected spacing issue in 9.3.

*D 2578 - 99a:*

- (1) An ISO equivalency statement was added as Note 1.

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