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ภาคผนวก

ภาคผนวก ก

การทดสอบสมบัติของอะคริลิกเรซิน

ก.1 ค่าของกรดของอะคริลิกเรซิน (ความเข้มข้นของ KOH = 0.1 N)

ครั้งที่	ปริมาณ KOH (ml)	น้ำหนักสารตัวอย่าง (g)	ค่าของกรด
1	20.5	1.01	113.9
2	23.6	1.02	113.9
3	21.9	0.98	113.3
เฉลี่ย			113.7

การทดสอบสมบัติของน้ำมัน

ก.2 ค่าของกรดของน้ำมันปาล์ม น้ำมันทัง และ น้ำมันปาล์มที่ผ่านการดัดแปร

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
น้ำมันปาล์ม			
ครั้งที่ 1	20.29	0.6	0.2
ครั้งที่ 2	20.02	0.6	0.2
ครั้งที่ 3	20.02	0.6	0.2
เฉลี่ย			0.2
น้ำมันทัง			
ครั้งที่ 1	10.16	13.25	7.3
ครั้งที่ 2	10.37	13.75	7.4
ครั้งที่ 3	10.58	14.00	7.4
เฉลี่ย			7.4

ก.2 (ต่อ) ค่าของกรดของน้ำมันปาล์ม น้ำมันทัง และ น้ำมันปาล์มที่ผ่านการดัดแปร

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
น้ำมันปาล์ม ที่ผ่านการดัดแปร			
ครั้งที่ 1	20.00	10.5	2.9
ครั้งที่ 2	20.00	10.6	3.0
ครั้งที่ 3	19.99	10.6	3.0
เฉลี่ย			3.0

ก.3 ค่าความถ่วงจำเพาะของน้ำมันปาล์ม น้ำมันทัง และ น้ำมันปาล์มที่ผ่านการดัดแปร

		น้ำหนักของถ้วยวัด ความถ่วงจำเพาะ	น้ำหนักของถ้วยวัด ความถ่วงจำเพาะ ที่บรรจุน้ำมันเต็มแล้ว	ความถ่วงจำเพาะ
น้ำมันปาล์ม	ครั้งที่ 1	49.22	140.68	0.91
	ครั้งที่ 2	49.22	140.66	0.91
	ครั้งที่ 3	49.22	140.73	0.92
	เฉลี่ย			0.91
น้ำมันทัง	ครั้งที่ 1	49.22	143.68	0.94
	ครั้งที่ 2	49.22	143.59	0.94
	ครั้งที่ 3	49.22	143.65	0.94
	เฉลี่ย			0.94
น้ำมันปาล์ม ที่ผ่านการดัดแปร				
	ครั้งที่ 1	49.22	141.85	0.93
	ครั้งที่ 2	49.22	141.78	0.93
	ครั้งที่ 3	49.22	141.77	0.93
	เฉลี่ย			0.93

ก.4 ค่าความหนืดของน้ำมันปาล์ม น้ำมันทัง และ น้ำมันปาล์มที่ผ่านการดัดแปร

	หมายเลขเข็ม	ความเร็วที่ใช้	ค่าที่อ่านได้	แฟกเตอร์	ความหนืด (พอยส์)
น้ำมันปาล์ม					
ครั้งที่ 1	4	20	0.6	100	60
ครั้งที่ 2	4	20	0.6	100	60
ครั้งที่ 3	4	20	0.6	100	60
ครั้งที่ 4	4	50	1.5	40	60
ครั้งที่ 5	4	50	1.5	40	60
เฉลี่ย					60
น้ำมันทัง					
ครั้งที่ 1	4	20	2.25	100	225
ครั้งที่ 2	4	20	2.25	100	225
ครั้งที่ 3	4	20	2.25	100	225
ครั้งที่ 4	4	50	5.50	40	220
ครั้งที่ 5	4	50	5.50	40	220
เฉลี่ย					223
น้ำมันปาล์มที่ผ่านการดัดแปร					
ครั้งที่ 1	4	2.5	9.5	800	7,600
ครั้งที่ 2	4	2.5	9.5	800	7,600
ครั้งที่ 3	4	2.5	9.5	800	7,600
ครั้งที่ 4	5	20	38	200	7,600
ครั้งที่ 5	5	20	37.5	200	7,500
เฉลี่ย					7,580

การทดสอบสมบัติของอะคริลิก-อัลคิดเรซิน

ก.5 ค่าความหนืดของอะคริลิก-อัลคิดเรซิน

สูตร	หมายเลขเข็ม	ความเร็วที่ใช้	ค่าที่อ่านได้	แฟกเตอร์	ความหนืด (พอยส์)
20% acrylic					
ครั้งที่ 1	5	5	9.8	800	7,840
ครั้งที่ 2	5	5	10.3	800	8,240
ครั้งที่ 3	5	5	10.0	800	8,000
ครั้งที่ 4	5	10	19.8	400	7,920
ครั้งที่ 5	5	10	20.1	400	8,040
เฉลี่ย					8,008
25% acrylic					
ครั้งที่ 1	5	5	10.6	800	8,480
ครั้งที่ 2	5	5	10.5	800	8,400
ครั้งที่ 3	5	5	10.6	800	8,480
ครั้งที่ 4	5	10	21.3	400	8,520
ครั้งที่ 5	5	10	21.2	400	8,480
เฉลี่ย					8,472
30% acrylic					
ครั้งที่ 1	5	5	11.3	800	9,040
ครั้งที่ 2	5	5	11.2	800	8,960
ครั้งที่ 3	5	5	11.3	800	9,040
ครั้งที่ 4	5	10	22.5	400	9,000
ครั้งที่ 5	5	10	22.5	400	9,000
เฉลี่ย					9,008

ก.5 ค่าความหนืดของอะคริลิก-อัลคิดเรซิน (ต่อ)

สูตร	หมายเลขเข็ม	ความเร็วที่ใช้	ค่าที่อ่านได้	แฟกเตอร์	ความหนืด (พอยส์)
35% acrylic					
ครั้งที่ 1	5	5	12.5	800	10,000
ครั้งที่ 2	5	5	12.4	800	9,920
ครั้งที่ 3	5	5	12.7	800	10,160
ครั้งที่ 4	5	10	25.2	400	10,080
ครั้งที่ 5	5	10	25.1	400	10,040
เฉลี่ย					10,040

ก.6 ค่าของกรดของอะคริลิก-อัลคิดเรซิน
(ความเข้มข้นของ KOH = 0.1 N)

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
20% acrylic			
ครั้งที่ 1	5.06	15.7	17.4
ครั้งที่ 2	5.05	15.6	17.3
ครั้งที่ 3	5.07	15.7	17.4
เฉลี่ย			17.4
25% acrylic			
ครั้งที่ 1	5.08	20.20	22.3
ครั้งที่ 2	5.02	20.00	22.4
ครั้งที่ 3	5.06	20.20	22.4
เฉลี่ย			22.4

ก.6 ค่าของกรดของอะคริลิก-อัลคิดเรซิน (ต่อ)

(ความเข้มข้นของ KOH = 0.1 N)

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
30% acrylic			
ครั้งที่ 1	5.04	22.20	24.7
ครั้งที่ 2	5.02	22.10	24.7
ครั้งที่ 3	5.03	22.20	24.8
เฉลี่ย			24.7
35% acrylic			
ครั้งที่ 1	5.02	25.3	28.3
ครั้งที่ 2	5.02	25.4	28.4
ครั้งที่ 3	5.04	25.5	28.4
เฉลี่ย			28.4

การทดสอบสมบัติของอะคริลิก-อัลคิดเรซินสูตรน้ำ

ก.7 ค่าของกรดของอะคริลิก-อัลคิดเรซินสูตรน้ำและอัลคิดเรซินทางการค้า (ความเข้มข้นของ KOH = 0.1 N)

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
20% acrylic			
ครั้งที่ 1	20.16	1.10	0.3
ครั้งที่ 2	20.05	1.09	0.3
ครั้งที่ 3	20.07	1.10	0.3
เฉลี่ย			0.3
25% acrylic			
ครั้งที่ 1	20.08	1.57	0.5
ครั้งที่ 2	20.12	1.57	0.5
ครั้งที่ 3	20.06	1.57	0.5
เฉลี่ย			0.5
30% acrylic			
ครั้งที่ 1	20.14	2.50	0.7
ครั้งที่ 2	20.02	2.48	0.7
ครั้งที่ 3	20.08	2.49	0.7
เฉลี่ย			0.7
35% acrylic			
ครั้งที่ 1	20.02	3.33	1.0
ครั้งที่ 2	20.08	3.34	1.0
ครั้งที่ 3	20.04	3.33	1.0
เฉลี่ย			1.0

ก.7 ค่าของกรดของอะคริลิก-อัลคิดเรซินสูตรน้ำและอัลคิดเรซินทางการค้า (ความเข้มข้นของ KOH = 0.1 N)

	น้ำหนักสารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
อัลคิดเรซินทางการค้า			
ครั้งที่ 1	10.08	9.04	5.3
ครั้งที่ 2	10.10	9.06	5.3
ครั้งที่ 3	10.07	9.03	5.3
เฉลี่ย			5.3

การทดสอบสมบัติของสารเคลือบผิว

ก.8 ปริมาณร้อยละของสารที่ระเหยไม่ได้ของอะคริลิก-อัลคิดเรซิน (%NV)

สูตร	น.น. สารก่อนอบ (g)	น.น. สารหลังอบ (g)	% NV
20% (w) acrylic	2.2195	2.1029	94.75
25% (w) acrylic	2.0729	1.9487	94.01
30% (w) acrylic	2.0313	1.8922	93.15
35% (w) acrylic	2.1573	2.0499	93.52
อัลคิดเรซินทางการค้า	2.0618	1.8878	91.56

ก.9 การทดสอบความแข็งด้วยวิธีการขูดขีด

สูตร	ความทนต่อการขูดขีด (กรัม)			
	1	2	3	เฉลี่ย
20% (w) acrylic	100	100	100	100
25% (w) acrylic	100	100	100	100
30% (w) acrylic	100	100	100	100
35% (w) acrylic	100	100	100	100
อัลคิดเรซินทางการค้า	100	100	100	100

ก.10 ความอ่อนตัวของฟิล์ม

สูตร	ความอ่อนตัวของฟิล์ม (Φ,mm)			
	1	2	3	เฉลี่ย
20% (w) acrylic	3	3	3	3
25% (w) acrylic	3	3	3	3
30% (w) acrylic	3	3	3	3
35% (w) acrylic	3	3	3	3
อัลคิดเรซินทางการค้า	3	3	3	3

ก.11 ความติดแน่น (cross-cut , tape taste , 1 x 1 mm.)

สูตร	1	2	3	เฉลี่ย
20% (w) acrylic	5B	5B	5B	5B
25% (w) acrylic	5B	5B	5B	5B
30% (w) acrylic	5B	5B	5B	5B
35% (w) acrylic	5B	5B	5B	5B
อัลคิดเรซินทางการค้า	5B	5B	5B	5B

ก.12 ความทนทานแรงกระแทก

สูตร	ความทนทานแรงกระแทก (นิ้ว – ปอนด์)			
	1	2	3	เฉลี่ย
20% (w) acrylic	160	160	160	160
25% (w) acrylic	160	160	160	160
30% (w) acrylic	160	160	160	160
35% (w) acrylic	160	160	160	160
อัลคิดเรซินทางการค้า	160	160	160	160

ก.13 ความทนกรด 5% H_2SO_4

สูตร	ความทนกรด 5% H_2SO_4 (ชั่วโมง)		
	1	2	3
20% (w) acrylic	24	24	24
25% (w) acrylic	24	24	24
30% (w) acrylic	24	24	24
35% (w) acrylic	24	24	24
อัลคิดเรซินทางการค้า	4	4	4

ก.14 ความทนน้ำ

สูตร	ความทนน้ำ (ชั่วโมง)		
	1	2	3
20% (w) acrylic	24	24	24
25% (w) acrylic	24	24	24
30% (w) acrylic	24	24	24
35% (w) acrylic	24	24	24
อัลคิดเรซินทางการค้า	22	22	22

ก.15 ความทนด่าง 5% NaOH (นาที)

สูตร	ความทนด่าง 5% NaOH (นาที)			
	1	2	3	เฉลี่ย
20% (w) acrylic	29	31	30	30
25% (w) acrylic	184	178	179	180
30% (w) acrylic	207	213	210	210
35% (w) acrylic	252	251	248	250
อัลคิดเรซินทางการค้า	13	12	11	13

ภาคผนวก ข

ข.1 การหาปริมาณ Fatty Acid Composition

โดยวิธี Gas Chromatrograph ใช้ตัวอย่างน้ำมันประมาณ 1 กรัม เติม เบนซีน : เมทานอล : กรดซัลฟูริก ในอัตราส่วน 60 : 180 : 2 โดยปริมาตรตัวอย่างละ 120 มิลลิลิตร กลั่นรีฟลักซ์ (reflux) 2 ชั่วโมง 30 นาที ทำให้เย็นแล้วล้างด้วยน้ำ ใช้ปิโตรเลียมอีเทอร์เป็นตัวแยก ครั้งละ 100 มิลลิลิตร 5 ครั้ง หรือจนส่วนน้ำมันที่แยกออกเป็นกลาง ทดสอบด้วยเมทิลออเรนจ์ ใช้ แอนไฮดรัสโซเดียมซัลเฟต ดูดน้ำที่ตกค้างในส่วนของเมทิลเอสเทอร์ที่ละลายอยู่ในปิโตรเลียมอีเทอร์ แยกปิโตรเลียมอีเทอร์ออกโดย rotary evaporater ที่อุณหภูมิ 80°C ส่วนที่เหลือคือเมทิลเอสเทอร์ ทำให้เจือจาง 1% ด้วยนอร์มัลเฮกเซน ฉีดเข้าไปในเครื่อง gas chromatograph

ข.2 การวัดและการคำนวณความหนืดของน้ำมันโดย Brookfield viscometer

ใช้ตารางตามที่ควบมากับเครื่อง ซึ่งจะผ่านการตรวจสอบอย่างแม่นยำมาแล้ว และใช้อุณหภูมิในการวัดตรงตามข้อกำหนดของเครื่อง

ถ้าพิจารณาว่าน้ำมันหรือวาร์นิชที่จะวัดมีความเข้มข้นน้อย ใช้หมายเลขเข็มต่ำ เริ่มที่ความเร็วต่ำ แล้วเพิ่มสูงขึ้นเรื่อย ๆ

ตัวอย่างตารางที่ควบมากับเครื่อง

เบอร์เข็ม					
4		5		6	
ความเร็ว	factor	ความเร็ว	factor	ความเร็ว	factor
0.5	4 M	0.5	8 M	0.5	20 M
1.0	2 M	1.0	4 M	1.0	10 M
2.0	1 M	2.0	2 M	2.0	5 M
2.5	800	2.5	1.6 M	2.5	4 M
4.0	500	4.0	1 M	4.0	2.5 M

เบอร์เข็ม					
4		5		6	
ความเร็ว	factor	ความเร็ว	factor	ความเร็ว	factor
5.0	400	5.0	800	5.0	2 M
10	200	10	400	10	1 M
20	100	20	200	20	500
50	40	50	80	50	200
100	20	100	40	100	100

ค่าความหนืดคำนวณได้จาก

ความหนืด = ค่าที่อ่านได้จากเครื่อง x factor ที่เบอร์เข็มและความเร็วนั้น ๆ

ตัวอย่างการคำนวณ เช่น ใช้เข็มวัดความหนืดเบอร์ 4 อ่านค่าได้จากเครื่อง 10 ความเร็วที่ใช้ 2.5 ค่าความหนืดคำนวณได้ดังนี้

ความหนืด = 10 x 800

= 8,000 พอยส์

ภาคผนวก ค

Designation: D 1640 – 95

Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature[1]

This standard is issued under the fixed designation D 1640; 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.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the various stages and rates of film formation in the drying or curing of organic coatings normally used under conditions of ambient room temperature.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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. Referenced Documents

2.1 *ASTM Standards:*

D 202 Test Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation[2]

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels[3]

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers[3]

D 2091 Test Method for Print Resistance of Lacquers[4]

2.2 *U.S. Government Standards:*

Fed. Spec. No. CCC-C-440, Cheesecloth[5]

Fed. Spec. No. CCC-C-419b, Type III, Army Duck[5]

2.3 *TAPPI Standards:*[6]

T 402 Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products

3. Significance and Use

3.1 These test methods are used to determine the various stages and rates of drying, curing, and film formation of organic coatings for the purpose of comparing types of coatings or ingredient changes, or both. This is significant in the development of organic coatings for various end uses and also for production quality control.

4. Coatings and Recommended Film Thicknesses

4.1 Whenever tests are to be performed on coatings not listed in Table 1, there should be a prior agreement between the purchaser and seller as to the substrate, film thickness, and application method for testing the specific coating involved.

5. Test Conditions

5.1 Conduct all drying tests in a well-ventilated room or chamber, free from direct drafts (Note 1), dust, products of combustion, laboratory fumes and under diffused light (see 5.4). Make all measurements at a temperature of 23 ± 2°C and 50 ± 5 % relative humidity with the coated panels in a horizontal position while drying.

NOTE 1—A device to equalize air change conditions has been developed by F. Scofield.[7] Relative humidity should be controlled for moisture-cured and two-package urethane coatings, since their cure is greatly affected by the existing moisture conditions.

5.2 Tests should be carried out at practical viscosities at which films can be applied to the proper film thickness with resultant good flow and leveling properties. In the absence of any specific material specification, instructions for preparation of the film should be determined and agreed upon between the purchaser and the seller.

5.3 Films to be tested should have practical thicknesses commensurate with performance characteristics expected under actual usage for the type under test. All testing should be done within an area, any point of which is not less than ½ in. (15 mm) from the film edge.

5.4 *Light Conditions*—Illumination of the films during the entire drying test period should be about 25 ft-candles (270 1x) from normal laboratory or sky sources, never from direct sunlight or other sources high in nonvisible radiant energy.

6. Preparation of Test Specimens

6.1 Carry out all tests as described in 6.1.1, 6.1.2 and 6.1.3, unless otherwise noted.

6.1.1 All test specimens shall be prepared and tested by

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Film.

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[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[6] Available from Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

[7] Gardner and Sward, *Paint Testing Manual*, ASTM STP 500, ASTM, 13th edition, 1972, p. 269.

one operator properly skilled in the methods to be used. Apply the specimens in duplicate at a time arranged so that examination intervals will fall within the normal working hours of the operator.

6.1.2 Apply the materials to be tested on clean glass panels or other specific substrate of suitable dimensions agreed upon between the purchaser and the seller. Ground-glass plates are more suitable for certain types of coatings that tend to crawl, such as low-viscosity drying oils. Suitable plates can be prepared by roughening the surface of polished glass by grinding a paste of silicon carbide (grit 1-F) and water between two glass plates.

6.1.3 The test films perferably shall be cast with a doctor blade having a clearance sufficient to give the recommended dry film thickness indicated in Table 1. When a suitable doctor blade is not available, or it has been agreed upon to apply the film in some other manner, the various conventional and automatic methods of spray, dip, flow, and brush application may be used, provided dry film thicknesses conform to the requirements given in Table 1. See Practices D 823 for a description of the spray and dip methods of application.

6.1.4 Measure the dry film thickness of test films with the proper film thickness gage. This shall be a micrometer, dial comparator, or dial indicator as described in Test Methods D 1005. When plates of small area are used, measurement of dry film thickness can be made by weighing plates before and after coating and calculating from plate area and coating solids.

7. Procedure

7.1 When test methods or end points other than those listed in 7.2 to 7.9 are used, there shall be a prior agreement between the purchaser and the seller.

7.2 *Set-To-Touch Time*—To determine set-to-touch time, lightly touch the test film with the tip of a clean finger and immediately place the fingertip against a piece of clean, clear glass. Observe if any of the coating is transferred to the glass. For the purpose of this test, the pressure of the fingertip against the coating shall not be greater than that required to transfer a spot of the coating from ⅛ to 3⁄16 in. (3 to 5 mm) in cross section. The film is set-to-touch when it still shows a tacky condition, but none of it adheres to the finger.

7.3 *Dust-Free Times:*

7.3.1 *Cotton Fiber Test Method*—Separate a number of individual fibers from a mass of absorbent cotton with the aid of tweezers. At regular drying intervals, drop several of the cotton fibers from a height of 1 in. (25 mm) onto a marked section of the film. The film is considered to hav dried dust free when the cotton fibers can be removed b blowing lightly over the surface of the film.

TABLE 1 Recommended Film Thickness of Materials to be Tested[A]

Material	Dry Film Thickness
Drying oils	1.25 ± 0.25 mil (32 ± 6 µm)[B]
Varnishes	1 ± 0.1 mil (25 ± 2 µm) (See 7.3.2)
Lacquers	0.5 ± 0.1 mil (12.5 ± 2 µm) (See 7.4.2)
Resin solutions	0.5 ± 0.1 mil (12.5 ± 2 µm)
Enamels	1.5 ± 0.25 mil (36.5 ± 6 µm)
Oil paints	1.8 ± 0.2 mil (45 ± 2.5 µm) (see 6.2)
Water paints	1 ± 0.1 mil (25 ± 2 µm)

[A] This table is a general guide to be used when nothing more specific is agreed upon between the purchaser and the seller.

[B] See 6.1.2 and 7.4.1 Add driers a minimum of 24 h before test.

7.4 *Tack-Free Times:*

7.4.1 *Paper Test Method:*

7.4.1.1 *Test Paper*—The test paper shall be K-4 Powe Cable Paper[8] that when conditioned in accordance with th TAPPI Standard Method T 402, conforms to the followin requirements:

Basis weight (24 by 36/500), lb	90 ± 5
Thickness, mils (µm)	6.65 (17)
Air resistance (s/100 cm²/in.²)	350
Coefficient of static friction[A]	0.5
Friction angle, °	22
Tensile strength, machine direction/cross direction	119/32
Tear, machine direction/cross direction	180/250
Elongation, machine direction/cross direction, %	3.0/7.0
pH of water extract	7.4
Ash content, max, %	0.6

[A] All tests except this one shall be run in accordance with Test Method D 20 All values for properties are typical values and not specification limits.

7.4.1.2 Lay a 2 by 3-in. (50 by 75-mm) piece of the specia test paper on the film and place upon it a steel cylinder 2 in in diameter, and of such weight 6.28 lb, (2.85 kg), as tc produce a pressure of 2 psi (13.8 kPa). At the end of 5 s remove the cylinder and invert the test panel. The film is considered free from after-tack when the paper drops off o the test film within 10 s.

7.4.2 A variation of the test method described in 7.4.1 using the same test paper can be used to test the tack-free time of insulating varnishes. In this method the piece of paper shall be 1½ in. (40 mm) in width and 6 in. (150 mm) in length. The varnish is considered tack-free when this strip of paper does not adhere to it when it is pressed on the surface of the varnish for 1 min by a cylindrical 1-lb (450-g) weight, 1 in. (25 mm) in diameter. In this test, apply the paper in the vicinity of the center of the specimen at right angles to the length of the coated specimen.

7.4.3 *Mechanical Test Method (Tack Tester[9])*—The tack tester to be used in this method comprises essentially a base or surface-contacting portion 1-in. (25-mm) square and a counter-balancing portion 1 by 2 in. (25 by 50 mm) in area. Both portions are made up from a continuous metal strip 0.016 to 0.018 in. (0.41 to 0.46 mm) in thickness. To prepare the apparatus for use (see 7.4.3.1), fit the base with several thicknesses of masking tape and paper strips to provide a means of attaching the aluminum foil and so adjust the angle of the 1 by 2-in. counter-balancing strip so that a weight of 5 g placed in the geometric center of the base portion is just sufficient to overcome the unbalanced force.

7.4.3.1 The tester is prepared for use by carrying out the following steps in sequence:

(*1*) Wrap the metal base with three thicknesses of masking tape, sticky side out,

(*2*) Cover the outer layer with a good grade of paper, except for two exposed strips, equally spaced, about ¼ by 1 in. (6.4 by 25 mm) in area on the top of the tester, and

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[8] Paper meeting these requirements may be obtained from Crocker Technical Papers, Inc., 431 Westminster St., Fitchburg, MA 01420, their Grade R 20–34.

[9] The standard tack tester is fully described in the U. S. Patent 2,406,989, Sept. 3, 1946

(*3*) Cover the paper on the contact side of the base with one thickness of pressure-sensitive cellulose tape previously fixed to the metal base of the tester. The cellulose tape serves two purposes:

First, to pull the layers of masking tape firmly against the front of the metal base, and

Second, to provide a smooth surface for the foil. Attach the aluminum foil to the base of the tester by pressing gently but firmly a 1 by 2-in. (25 by 50-mm) piece of foil, 0.0005 in. (13 µm) in thickness against one of the ¼ by 1-in. (6.4 by 25-mm) exposed strips of masking tape on the top surface of the base. Wrap the foil tightly and smoothly around the base, exposing the shiny side, and finally press the outer end gently against the remaining exposed strip of masking tape. When it finally becomes necessary to replace wrinkled or soiled aluminum foil, the ends are easily removed from the masking tape by exerting a slow, even, upward pull sufficient to overcome the tack of the tape without tearing the foil.

7.4.3.2 A film is considered to have dried tack-free when the tack tester tips over immediately on removing a 300-g weight allowed to act for 5 s on the counter-weighted metal square base fitted with masking tape and aluminum foil.

7.5 *Dry-To-Touch Time:*

7.5.1 *Drying Oils*—Continue testing after the set-to-touch time has been observed. The film is considered dry when it no longer adheres to the finger and does not rub up appreciably when the finger is lightly rubbed across the surface.

7.5.2 *Lacquers (and Sealers)*—Touch the film lightly at varying intervals of time. The film is considered dry when no pronounced marks are left by the finger touching the film in the same area on each observation. Test sealers on wood or other porous substrates as agreed upon between the purchaser and the seller.

7.6 *Dry-Hard Time:*

7.6.1 With the end of the thumb resting on the test film and the forefinger supporting the test panel, exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film is considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

7.7 *Dry-Through (or Dry-To-Handle) Time:*

7.7.1 Place the test panel in a horizontal position at a height such that when the thumb is placed on the film, the arm of the operator is in a vertical line from the wrist to the shoulder. Bear down on the film with the thumb, exerting the maximum pressure of the arm, at the same time turning the thumb through an angle of 90° in the plane of the film. The film is considered dry-through or dry-to-handle when there is no loosening, detachment, wrinkling, or other evidence of distortion of the film.

7.8 *Dry-To-Recoat:*

7.8.1 A film is considered dry for recoating when a second coat or specified topcoat can be applied without the development of any film irregularities such as lifting or loss of adhesion of the first coat, and the dry time of the second coat does not exceed the maximum specified (if any) for the first coat.

7.9 *Print-Free Time:*

NOTE 2—This procedure is similar to Test Method D 2091, except that the time to reach the print-free condition is determined, while Test Method D 2091 is used to evaluate whether a film is print free at a specified time.

7.9.1 *Test Panels*—Apply the material under test to clean plane panels, at least 3 by 6 in. (75 by 150 mm) in size, made of wood, metal, glass, plastic or other material as agreed upon between the purchaser and the seller.

7.9.2 *Imprinting Fabric*—Eight-ounce Army duck conforming to Type III of U.S. Fed. Spec. No. CCC-C-419b or cheesecloth conforming to Fed. Spec. No. CCC-C-440.

7.9.2.1 A pad should be used with the cheesecloth only, made of nonwoven felt cloth at least 0.05 in. (1.3 mm) thick, weighing 7 oz/yd^2 (0.24 kg/m^2) and larger than the plane end of the weight.

7.9.3 *Weights*—Consisting of metal cylinders not less than 2 in. (50 mm) in diameter with plane ends perpendicular to the axis and of a length to give a pressure of ½ or 1 lb/in.^{2} (3.5 or 6.9 kPa).

7.9.4 *Procedure:*

7.9.4.1 Apply the test material to several of the specified or agreed-upon panels by a film applicator, or other specified method, as described in Practices D 823 in either single or multiple coats, as agreed upon between the purchaser and the seller. In the absence of a specified dry film thickness, the values listed in Table 1 should be used.

7.9.4.2 Allow the coated panels to dry under the conditions specified in Section 5, unless otherwise agreed. At appropriate intervals, starting shortly before the coating is expected to be print-free, carry out the print-free test as described in Test Method D 2091, comparing the appearance with the photographic standards appearing therein, until the test shows the coating to be print-free.

8. Frequency of Testing

8.1 It is suggested that test intervals be set at periods of approximately 10 % of the total test time. If frequency varies considerably from the 10 % interval or such time interval is impractical, the intervals used shall be reported.

9. Report

9.1 Reports of tests shall include all applicable conditions that deviated from the standards as outlined or special conditions or tests used and the results of the test.

10. Precision and Bias

10.1 Because of the subjective nature of the drying time tests, the agreement to be expected between laboratories depends upon their understanding of the terms used, and is difficult to establish with certainty. Within any laboratory, the agreement depends upon the material being tested, some coatings being much sharper in their end point than others, but duplicate determinations should agree within 10 % of the time of drying.[10]

10.2 *Bias*—These test methods have no bias because the value for dry times are defined only in terms of these test methods.

11. Keyword

11.1 drying time

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[10] See Prane, J. W., "A Latin Square Drying Time Study," *Paint Industry Magazine* (August 1961), for a study of precision of drying time measurements.

Designation: D 3359 – 95

Standard Test Methods for Measuring Adhesion by Tape Test[1]

This standard is issued under the fixed designation D 3359; 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.

These methods have been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film.

1.2 Test Method A is primarily intended for use at job sites while Test Method B is more suitable for use in the laboratory. Also, Test Method B is not considered suitable for films thicker than 5 mils (125 µm).

NOTE 1—Subject to agreement between the purchaser and the seller, Test Method B can be used for thicker films if wider spaced cuts are employed.

1.3 These test methods are used to establish whether the adhesion of a coating to a substrate is at a generally adequate level. They do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required.

NOTE 2—It should be recognized that differences in adherability of the coating surface can affect the results obtained with coatings having the same inherent adhesion.

1.4 In multicoat systems adhesion failure may occur between coats so that the adhesion of the coating system to the substrate is not determined.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address 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:*

- D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products[2]
- D 823 Practice for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels[2]
- D 1000 Test Methods For Pressure-Sensitive Adhesive-Coated Tapes Used for Electrical and Electronic Applications[3]
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting[4]
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting[5]
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion[2]
- D 2370 Test Method for Tensile Properties of Organic Coatings[2]
- D 3330 Test Method for Peel Adhesion of Pressure-Sensitive Tape of 180° Angle[6]
- D 3924 Specification for Standard Environment for Conditioning and Tesing Paint, Varnish, Lacquers, and Related Materials[2]
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser[2]

3. Summary of Test Methods

3.1 *Test Method A*—An X-cut is made in the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale.

3.2 *Test Method B*—A lattice pattern with either six or eleven cuts in each direction is made in the film to the substrate, pressure-sensitive tape is applied over the lattice and then removed, and adhesion is evaluated by comparison with descriptions and illustrations.

4. Significance and Use

4.1 If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because the substrate and its surface preparation (or lack of it) has a drastic effect on the adhesion of coatings, a method of evaluation adhesion of a coating to different substrates or surface treatments, or of different coatings to the same substrate and treatment, is of considerable usefulness in the industry.

4.2 The limitations of all adhesion methods and the specific limitation of this test method to lower levels of adhesion (see 1.3) should be recognized before using it. The intra- and inter-laboratory precision of this test method is similar to other widely-accepted tests for coated substrates

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

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[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 10.01.

[4] *Annual Book of ASTM Standards*, Vol 02.05.

[5] *Annual Book of ASTM Standards*, Vol 06.02.

[6] *Annual Book of ASTM Standards*, Vol 15.09.

(for example, Test Method D 2370 and Test Method D 4060), but this is partly the result of it being insensitive to all but large differences in adhesion. The limited scale of 0 to 5 was selected deliberately to avoid a false impression of being sensitive.

TEST METHOD A—X-CUT TAPE TEST

5. Apparatus and Materials

5.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting devices. It is of particular importance that the cutting edges be in good condition.

5.2 *Cutting Guide*—Steel or other hard metal straightedge to ensure straight cuts.

5.3 *Tape*–One-inch (25-mm) wide semitransparent pressure-sensitive tape with an adhesion strength agreed upon by the supplier and the user is needed[7]. Because of the variability in adhesion strength from batch-to-batch and with time, it is essential that tape from the same batch be used when tests are to be run in different laboratories. If this is not possible the test method should be used only for ranking a series of test coatings.

5.4 *Rubber Eraser*, on the end of a pencil.

5.5 *Illumination*—A light source is helpful in determining whether the cuts have been made through the film to the substrate.

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the adhesion is to be evaluated.

6.2 For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the adhesion.

NOTE 3—Applicable test panel description and surface preparation methods are given in Practice D 609 and Practices D 1730 and D 2092.

NOTE 4—Coatings should be applied in accordance with Practice D 823, or as agreed upon between the purchaser and the seller.

NOTE 5—If desired or specified, the coated test panels may be subjected to a preliminary exposure such as water immersion, salt spray, or high humidity before conducting the tape test. The conditions and time of exposure will be governed by ultimate coating use or shall be agreed upon between the purchaser and seller.

7. Procedure

7.1 Select an area free of blemishes and minor surface imperfections. For tests in the field, ensure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

7.2 Make two cuts in the film each about 1.5 in. (40 mm) long that intersect near their middle with a smaller angle of between 30 and 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion.

7.3 Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another X in a different location. Do not attempt to deepen a previous cut as this may affect adhesion along the incision.

7.4 Remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 3 in. (75 mm) long.

7.5 Place the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of a pencil. The color under the transparent tape is a useful indication of when good contact has been made.

7.6 Within 90 ± 30 s of application, remove the tape by seizing the free end and pulling it off rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

7.7 Inspect the X-cut area for removal of coating from the substrate or previous coating and rate the adhesion in accordance with the following scale:

5A	No peeling or removal,
4A	Trace peeling or removal along incisions or at their intersection,
3A	Jagged removal along incisions up to 1/16 in. (1.6 mm) on either side,
2A	Jagged removal along most of incisions up to 1/8 in. (3.2 mm) on either side,
1A	Removal from most of the area of the X under the tape, and
0A	Removal beyond the area of the X.

7.8 Repeat the test in two other locations on each test panel. For large structures make sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

7.9 After making several cuts examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone before using again. Discard cutting tools that develop nicks or other defects that tear the film.

8. Report

8.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred that is, between first coat and substrate, between first and second coat, etc.

8.2 For field tests report the structure or article tested, the location and the environmental conditions at the time of testing.

8.3 For test panels report the substrate employed, the type of coating, the method of cure, and the environmental conditions at the time of testing.

8.4 If the adhesion strength of the tape has been determined in accordance with Test Methods D 1000 or D 3330, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

9. Precision and Bias[8]

9.1 In an interlaboratory study of this test method in which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering

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[7] Permacel 99 manufactured by Permacel, New Brunswick, NJ 08903, and available from various Permacel tape distributors, is reported to be suitable for this purpose. The manufacturer of this tape and the manufacturer of the tape used in the interlaboratory study (see RR: D01-1008), have advised this subcommittee that the properties of these tapes were changed. Users of it should, therefore, check whether current material gives comparable results to previous supplied material.

[8] Supporting data are available from ASTM Headquarters. Request RR: D01-1008.

a wide range of adhesion, the within-laboratories standard deviation was found to be 0.33 and the between-laboratories 0.44. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than 1 rating unit for two measurements.

9.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 1.5 rating units.

9.2 Bias cannot be established for these test methods.

TEST METHOD B—CROSS-CUT TAPE TEST

10. Apparatus and Materials

10.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between 15 and 30° that will make either a single cut or several cuts at once[9]. It is of particular importance that the cutting edge or edges be in good condition.

10.2 *Cutting Guide*—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straightedge or template to ensure straight cuts.

10.3 *Rule*—Tempered steel rule graduated in 0.5 mm for measuring individual cuts.

10.4 *Tape*, as described in 5.3.

10.5 *Rubber Eraser*, on the end of a pencil.

10.6 *Illumination*, as described in 5.5.

10.7 *Magnifying Glass*—An illuminated magnifier to be used while making individual cuts and examining the test area.

11. Test Specimens

11.1 Test specimens shall be as described in Section 6. It should be noted, however, that multitip cutters provide good results only on test areas sufficiently plane[10] that all cutting edges contact the substrate to the same degree. Check for flatness with a straight edge such as that of the tempered steel rule (10.3).

12. Procedure

12.1 Where required or when agreed upon, subject the specimens to a preliminary test before conducting the tape test (see Note 3). After drying or testing the coating, conduct the tape test at room temperature as defined in Specification D 3924, unless D 3924 standard temperature is required or agreed.

12.2 Select an area free of blemishes and minor surface imperfections, place on a firm base, and under the illuminated magnifier, make parallel cuts as follows:

12.2.1 For coatings having a dry film thickness up to and including 2.0 mils (50 µm) space the cuts 1 mm apart and make eleven cuts unless otherwise agreed upon.

12.2.2 For coatings having a dry film thickness between 2.0 mils (50 µm) and 5 mils (125 µm), space the cuts 2 mm apart and make six cuts. For films thicker than 5 mils use Test Method A.

12.2.3 Make all cuts about ¾ in. (20 mm) long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area.

12.3 After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings.

12.4 Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts.

12.5 Brush the area as before and inspect the incisions for reflection of light from the substrate. If the metal has not been reached make another grid in a different location.

12.6 Remove two complete laps of tape and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 3 in. (75 mm) long.

12.7 Place the center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser

CLASSIFICATION OF ADHESION TEST RESULTS

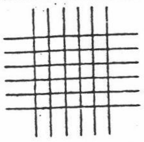
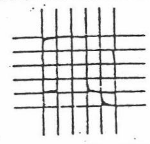
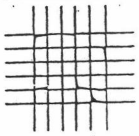
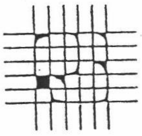
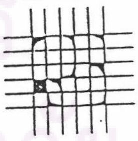
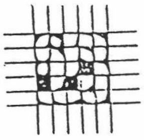
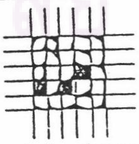
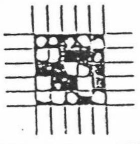
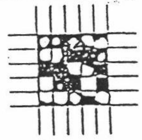
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	 
2B	15 - 35%	 
1B	35 - 65%	 
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

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[9] Multiblade cutters are available from a few sources that specialize in testing equipment for the paint industry. One supplier that has assisted in the refinement of these methods and of Test Methods D 2197 is given in footnote 10.

[10] A multitip cutter for coated pipe surfaces is now available from Paul N. Gardner Co., 316 NE First St., Pompano Beach, FL 33060.

on the end of a pencil. The color under the tape is a useful indication of when good contact has been made.

12.8 Within 90 ± 30 s of application, remove the tape by seizing the free end and ~~rapidly~~ (not jerked) back upon itself at as close to an angle of 180° as possible.

12.9 Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. Rate the adhesion in accordance with the following scale illustrated in Fig. 1:

5B	The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
4B	Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
3B	Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
2B	The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
1B	The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
0B	Flaking and detachment worse than Grade 1.

12.10 Repeat the test in two other locations on each test panel.

13. Report

13.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred, that is, between first coat and substrate, between first and second coat, etc.

13.2 Report the substrate employed, the type of coating and the method of cure.

13.3 If the adhesion strength has been determined in accordance with Test Methods D 1000 or D 3330, report t results with the adhesion rating(s). If the adhesion strength the tape has not been determined, report the specific ta used and its manufacturer.

14. Precision and Bias[8]

14.1 On the basis of two interlaboratory tests of this te method in one of which operators in six laboratories ma one adhesion measurement on three panels each of thr coatings covering a wide range of adhesion and in the oth operators in six laboratories made three measurements c two panels each of four different coatings applied over tw other coatings, the pooled standard deviations for withi and between-laboratories were found to be 0.37 and 0. Based on these standard deviations, the following criter should be used for judging the acceptability of results at th 95 % confidence level:

14.1.1 *Repeatability*—Provided adhesion is uniform ov a large surface, results obtained by the same operator shou be considered suspect if they differ by more than one ratin unit for two measurements.

14.1.2 *Reproducibility*—Two results, each the mean c duplicates or triplicates, obtained by different operato should be considered suspect if they differ by more than tw rating units.

14.2 Bias cannot be established for these test methods.

15. Keywords

15.1 adhesion, tape; crosscut adhesion test method; tap adhesion test method; X-cut adhesion test method

APPENDIX

(Nonmandatory Information)

X1. COMMENTARY

X1.1 Introduction

X1.1.1 Given the complexities of the adhesion process, can adhesion be measured? As Mittal (1)[11] has pointed out, the answer is both yes and no. It is reasonable to state that at the present time no test exists that can precisely assess the actual physical strength of an adhesive bond. But it can also be said that it is possible to obtain an indication of relative adhesion performance.

X1.1.2 Practical adhesion test methods are generally of two types: "*implied*" and "*direct*". "Implied" tests include indentation or scribe techniques, rub testing, and wear testing. Criticism of these tests arises when they are used to quantify the strength of adhesive bonding. But this, in fact, is not their purpose. An "implied" test should be used to assess coating performance under actual service conditions. "Direct" measurements, on the other hand, are intended expressly to measure adhesion. Meaningful tests of this type are highly sought after, primarily because the results are expressed by a single discrete quantity, the force required to rupture the coating/substrate bond under prescribed conditions. Direct tests include the Hesiometer and the Adherometer (2). Common methods which approach the direct tests are peel, lap-shear, and tensile tests.

X1.2 Test Methods

X1.2.1 In practice, numerous types of tests have been used to attempt to evaluate adhesion by inducing bond rupture by different modes. Criteria deemed essential for a test to warrant large-scale acceptance are: use of a straightforward and unambiguous procedure; relevance to its intended application; repeatability and reproducibility; and quantifiability, including a meaningful rating scale for assessing performance.

X1.2.2 Test methods used for coatings on metals are: peel adhesion or "tape testing"; Gardner impact flexibility testing; and adhesive joint testing including shear (lap joint) and direct tensile (butt joint) testing. These tests do not strictly meet all the criteria listed, but an appealing aspect of these tests is that in most cases the equipment/instrumentation is readily available or can be obtained at reasonable cost.

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[11] The boldface numbers in parentheses refer to the list of references at the end of this test method.

X1.2.3 A wide diversity of tests methods have been developed over the years that measure aspects of adhesion (1–5). There generally is difficulty, however, in relating these tests to basic adhesion phenomena.

X1.3 The Tape Test

X1.3.1 By far, the most used test for evaluating coating "adhesion" is the peel test. In use since the 1930's, in its simplest version a piece of adhesive tape is pressed against the paint film. The test consists of observing if the film is peeled off when the tape is removed. The method can be refined to measure the force required for film removal. However, if the coating is removed simply by pulling off the tape, the strength of the adhesive bond is at such a low level as to be practically useless. Consequently, in most cases to assess coatings of appreciable adhesion, crosses or a cross-hatched pattern are cut into the coating, a tape applied and removed. The coating removed is assessed against an established rating scale. If a coating debonds from merely cutting an "X" and not applying tape, the adhesion is also very poor.

X1.3.2 The current widely-used version was first published in 1974; two test methods are covered in this standard. Both test methods are used to establish whether the adhesion of a coating to a substrate is at an adequate level; however they do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required. Major limitations of the tape test are its low sensitivity, applicabiity only to coatings of relatively low bond strengths, and non-determination of adhesion to the substrate where failure occurs within a single coat, as when testing primers alone, or within or between coats in multicoat systems. For multicoat systems where adhesion failure may occur between or within coats, the adhesion of the coating system to the substrate is not determined.

X1.3.3 Repeatability within one rating unit is generally observed for coatings on metals for both methods, with reproducibility of one to two units. The tape test enjoys widespread popularity and is viewed as "simple" as well as low in cost. Applied to metals, it is economical to perform, lends itself to job site application, and most importantly, after decades of use, people feel comfortable with it.

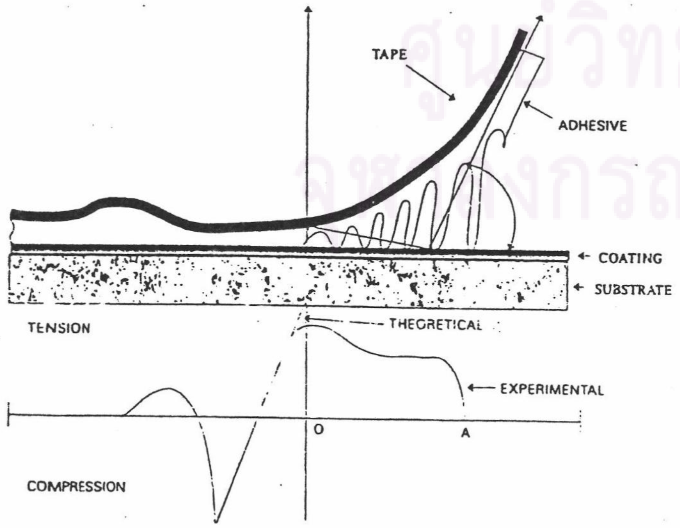

FIG. X1.1 Peel Profile (6)

X1.3.4 When a flexible adhesive tape is applied to a coated rigid substrate surface and then removed, the removal process has been described in terms of the "peel phenomenon," as illustrated in Fig. X1.1.

X1.3.5 Peeling begins at the "toothed" leading edge (at the right) and proceeds along the coating adhesive/interface or the coating/substrate interface, depending on the relative bond strengths. It is assumed that coating removal occurs when the tensile force generated along the latter interface, which is a function of the rheological properties of the backing and adhesive layer materials, is greater than the bond strength at the coating-substrate interface (or cohesive strength of the coating). In actuality, however, this force is distributed over a discrete distance (O-A) in Fig. X1.1, which relates directly to the properties described, not concentrated at a point (O) in Fig. X1.1 as in the theoretical case—though the tensile force is greatest at the origin for both. A significant compressive force arises from the response of the tape backing material to being stretched. Thus both tensile and compressive forces are involved in adhesion tape testing.

X1.3.6 Close scrutiny of the tape test with respect to the nature of the tape employed and certain aspects of the procedure itself reveal several factors, each or any combination of which can dramatically affect the results of the test as discussed (6).

X1.4 Peel Adhesion Testing on Plastic Substrates

X1.4.1 Tape tests have been criticized when used for substrates other than metal, such as plastics. The central issues are that the test on plastics lacks reproducibility and does not relate to the intended application. Both concerns are well founded: poor precision is a direct result of several factors intrinsic to the materials employed and the procedure itself. More importantly, in this instance the test is being applied beyond its intended scope. These test methods were designed for relatively ductile coatings applied to metal substrates, not for coatings (often brittle) applied to plastic parts (7). The unique functional requirements of coatings on plastic substrates cause the usual tape tests to be unsatisfactory for measuring adhesion performance in practice.

X1.5 The Tape Controversy

X1.5.1 With the withdrawal from commerce of the tape specified originally, 3M No. 710, current test methods no longer identify a specific tape. Differences in tapes used can lead to different results as small changes in backing stiffness and adhesive rheology cause large changes in the tension area. Some commercial tapes are manufactured to meet minimum standards. A given lot may surpass these standards and thus be suitable for general market distribution; however, such a lot may be a source of serious and unexpected error in assessing adhesion. One commercially available tape test kit had included a tape with adhesion strength variations of up to 50 % claimed by the manufacturer. Also, because tapes change on storage, bond strengths of the tape may change over time (7, 8).

X1.5.2 While there are tapes available that appear to deliver consistent performance, a given tape does not adhere equally well to all coatings. For example, when the peel removal force of the tape (from the coating) used earlier by Task Group D01.23.10 to establish precision of the method.

by 3M No. 710 was examined with seven different electromagnetic interference/radio frequency interference (EMI/RFI) coatings, it was found that, while peel was indeed consistent for a given coating, the value varied by 25 % between the highest and lowest ratings among coatings. Several factors that contribute to these differences include coating composition and topology: as a result, no single tape is likely to be suitable for testing all coatings. Further, the tape test does not give an absolute value for the force required for bond rupture, but serves only as an indicator that some minimum value for bond strength was met or exceeded **(7, 8)**.

X1.6 Procedural Problems

X1.6.1 The tape test is operator intensive. By design it was made as simple as possible to perform, and requires a minimum of specialized equipment and materials that must meet certain specifications. The accuracy and precision depend largely upon the skill of the operator and the operator's ability to perform the test in a consistent manner. Key steps that directly reflect the importance of operator skill include the angle and rate of tape removal and the visual assessment of the tested sample. It is not unexpected that different operators might obtain different results **(7, 8)**.

X1.6.2 *Peel Angle and Rate:* The standard requires that the free end of the tape be removed rapidly at as close to a 180° angle as possible. If the peel angle and rate vary, the force required to remove the tape can change dramatically. Nearly linear increases were observed in peel force approaching 100 % as peel angle was changed from 135 to 180, and similar large differences can be expected in peel force as peel rate varies. These effects are related as they reflect certain rheological properties of the backing and adhesive that are molecular in origin. Variation in pull rate and peel angle can effect large differences in test values and must be minimized to assure reproducibility **(9)**.

X1.6.3 *Visual Assessment:* The final step in the test is visual assessment of the coating removed from the specimen, which is subjective in nature, so that the coatings can vary among individuals evaluating the same specimen **(9)**.

X1.6.3.1 Performance in the tape test is based on the amount of coating removed compared to a descriptive scale. The exposure of the substrate can be due to factors other than coating adhesion, including that arising from the requirement that the coating be cut (hence the synonym "cross-hatch adhesion test"). Justification for the cutting step is reasonable as cutting provides a free edge from which peeling can begin without having to overcome the cohesive strength of the coating layer.

X1.6.3.2 Cutting might be suitable for coatings applied to metal substrates, but for coatings applied to plastics or wood, the process can lead to a misleading indication of poor adhesion due to the unique interfacial zone. For coatings on soft substrates, issues include how deep should this cut penetrate, and is it possible to cut only to the interface?

X1.6.3.3 In general, if adhesion test panels are examined microscopically, it is often clearly evident that the coating removal results from substrate failure at or below the interface, and not from the adhesive failure between the coating and the substrate. Cohesive failure within the coating film is also frequently observed. However, with the tape test, failures within the substrate or coating layers are rare because the tape adhesive is not usually strong enough to exceed the cohesive strengths of normal substrates and organic coatings. Although some rather brittle coatings may exhibit cohesive failure, the tape test adhesion method does not make provision for giving failure locality **(7, 8)**.

X1.6.4 Use of the test method in the field can lead to variation in test results due to temperature and humidity changes and their effect upon tape, coating and substrate.

X1.7 Conclusion

X1.7.1 All the issues aside, if these test methods are used within the Scope Section and are performed carefully, some insight into the approximate, relative level of adhesion can be gained.

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Standard Test Methods for Resistance of Dried Films of Varnishes to Water and Alkali[1]

This standard is issued under the fixed designation D 1647; 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.

. Scope

1.1 These test methods cover the determination of the resistance of dried varnish films to immersion in water and dilute alkali at room temperature.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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:*

D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products[2]

D 1193 Specification for Reagent Water[3]

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials[2]

3. Summary of Test Methods

3.1 *Test Method A*—The material under test is flowed onto tinplate panels and dried for 48 h. The panels are then immersed to half their length in reagent water for 18 h, or other agreed upon time, removed, and examined visually.

3.2 *Test Method B*—The material under test is applied to glass test tubes by dipping and dried for 72 h. The tubes are then suspended in dilute sodium hydroxide for periods ranging from 1 to 24 h, removed, rinsed, and after drying for 30 min, examined visually.

4. Significance and Use

4.1 Dried Varnish Films are a source of primary protection for surfaces. Exposure to water and dilute alkali solutions are two factors which tend to break down this protective coating. This test method can be used as a comparison basis between manufacturer and consumer to determine the ability of the varnish to resist water and dilute alkali.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is inte that all reagents conform to the specifications of the C mittee on Analytical Reagents of the American Che Society, where such specifications are available.[4]

5.2 *Purity of Water*—Unless otherwise indicated, ences to water shall be understood to mean reagent defined by Type IV of Specification D 1193.

TEST METHOD A—WATER RESISTANCE OF DRIED FI

6. Apparatus and Materials

6.1 *Beaker*, glass, 600 mL or larger.

6.2 *Tinplate Panels*, 3 by 5 in. (75 by 125 mm) cut commercial No. 31 gage (0.225-mm) bright tin weighing 0.4 to 0.5 lb/ft^2 (1.90 to 2.50 kg/m^2) and car cleaned and dried before use in accordance with Meth or C of Methods D 609.

7. Procedure

7.1 Flow the varnish onto the tin panels, allow to d a nearly vertical position, and dry for 48 h in the sta atmosphere described in Specification D 3924.

7.2 Place the panels in a beaker containing about (65 mm) of water at room temperature, immersing th that were uppermost during drying, and allow to rem the water for 18 h, or other suitable period as agree between the purchaser and the seller.

7.3 Remove the panels from the water, wipe careful allow to dry at room temperature. Note the time requ whitening, if any, to disappear. Blooming, which som occurs on immersion, is considered a type of whiteni

8. Report

8.1 Report the results of the water test as foll

8.1.1 Not visibly affected,

8.1.2 Whitening disappears within 20 min,

8.1.3 Whitening does not disappear within 20 m disappears within 2 h,

8.1.4 Whitening does not disappear within 2 disappears within 24 h, or

8.1.5 Whitening does not disappear within 24 h.

TEST METHOD B—DILUTE ALKALI RESISTANCE OF FILMS

9. Reagent

9.1 *Sodium Hydroxide Solution* (30 g/L)—Disso

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Polymers and Resins.

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[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Reagent Chemicals, American Chemical Society Specifications*, Chemical Society, Washington, DC. For suggestions on the testing of r listed by the American Chemical Society, see *Analar Standards for Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Ph and National Formulary*, U.S. Pharmaceutical Convention, In Rockville, MD.

of sodium hydroxide (NaOH) in water and dilute to 1 L.

10. Procedure

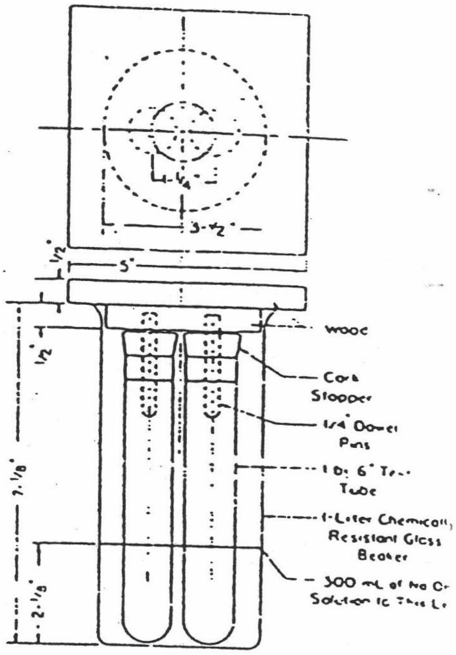
10.1 Thoroughly clean and dry twenty 1 by 6-in. (25 by 150-mm) test tubes in toluene. Dip the tubes into the varnish under test, remove immediately, invert the tubes, and allow the varnish to dry for 72 ± 1 h in the standard atmosphere described in Specification D 3924. A suggested test schedule to fit into normal working hours is shown in Table 1.

10.2 Into each of ten 1000-mL, tall-form lipless, chemically resistant plastic or glass beakers, place 300 mL of the NaOH solution. Suspend a set of two varnish-coated tubes in each beaker so that the tubes do not touch the bottom or sides of the beaker and are immersed for a distance of approximately 2 in. (50 mm). As a suspending device (see Fig. 1), use a wooden cover plate and two dowels, and two one-hole cork stoppers, the dowels and the corks fitting into the tubes and the cover plate fitting the beaker as tightly as possible. Maintain the NaOH solution at a temperature of 23 ± 2°C.

10.3 Remove a set of two varnish-coated tubes after immersion for each of the following time periods: 1, 2, 3, 4, 5, 6, 7, 8, 16, and 24 h. Rinse the tubes under a gentle stream of water, allow to air-dry for 30 min, and examine for film whitening, blistering, or removal. The end point the number of hours immersion at which the film whitening, blistering, or removal are n vertical sides of the tubes or on the spherical bo of the tube. The results may be compared with materials known to give acceptable performance work, the test on materials known to be accepta made simultaneously.

TABLE 1 Suggested Test Schedule

Day	Hour	Operation
Monday	8:30 a.m.	coat 16 tubes
	4:00 p.m.	coat 4 tubes
		dry tubes for 72 h
Thursday	8:30 a.m.	start alkali immersion on first 16 tubes
	9:30 a.m. and each hour thereafter to 4:30 p.m.	remove one set of tubes from alkali, rinse, and air-dry for 30 min
	10:00 a.m. and each hour thereafter to 5:00 p.m.	examine varnish film on set of tubes removed ½ h earlier
	4:00 p.m.	start alkali immersion on last 4 tubes
Friday	8:00 a.m.	remove one set of tubes from alkali, rinse, and air dry for 30 min
	8:30 a.m.	examine varnish film on set of tubes removed and rinsed ½ h earlier
	4:00 p.m.	remove last set of tubes, rinse, and air-dry for 30 min
	4:30 p.m.	examine varnish film on last set of tubes



NOTE—1 in. = 25.4 mm.

FIG. 1 Apparatus for Alkali Resistance Te

11. Report

11.1 Report the results of the alkali test as f

11.1.1 Type of varnish, and

11.1.2 End point of the test, h.

12. Precision

12.1 Precision has not been determined due plicity of ambient test conditions.

ประวัติผู้เขียนวิทยานิพนธ์

นายภาณุพันธ์ ผาพันธุ์ เกิดเมื่อวันที่ 7 ธันวาคม พ.ศ.2522 สำเร็จการศึกษาระดับปริญญาตรี วิทยาศาสตรบัณฑิต สาขาวิชาวัสดุศาสตร์ แขนงวิชาพอลิเมอร์และสิ่งทอ จากภาควิชาวัสดุศาสตร์ คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย ปีการศึกษา 2542 หลังจากนั้นเข้าศึกษาต่อในหลักสูตรวิทยาศาสตรมหาบัณฑิต สาขาวิทยาศาสตร์พอลิเมอร์ประยุกต์และเทคโนโลยีสิ่งทอ ภาควิชาวัสดุศาสตร์ คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย เมื่อภาคต้นของปีการศึกษา 2543 และสำเร็จการศึกษาในภาคต้นปีการศึกษา 2545 รวมระยะเวลาในการศึกษา 2.5 ปี