

## รายการอ้างอิง

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ภาคผนวก

## ภาคผนวก ก

การย่อยสลายขวดเพตที่ใช้แล้วเพื่อให้ได้ไกลโคไลซ์โพรดักส์

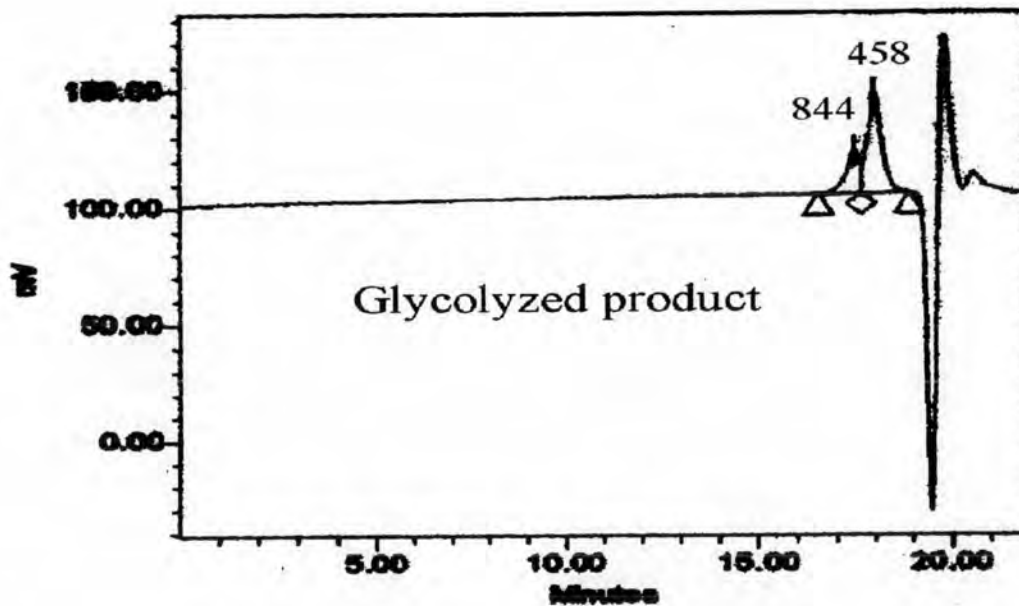
ก.1 ค่าไฮดรอกซิลของไกลโคไลซ์โพรดักส์

ครั้งที่	ปริมาณ NaOH (ml)	น้ำหนักสารตัวอย่าง (g)
1	74.4	1.00
2	74.7	0.99
3	76.6	1.00

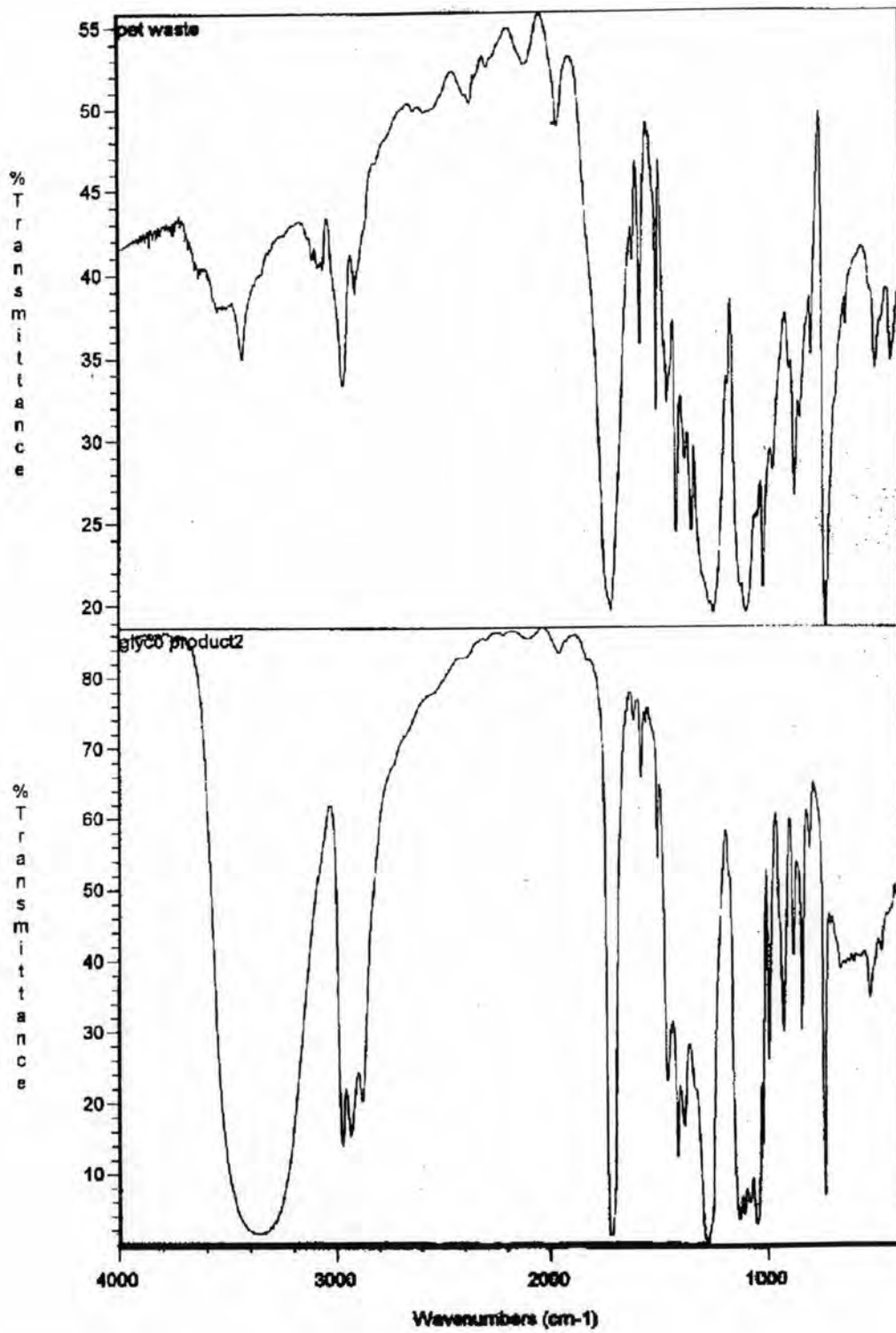
Normality ของ NaOH = 0.4951

ปริมาณของ NaOH ที่ใช้ไตเตรต blank = 94.3 ml

ก.2 โคโรมาโตแกรมจากการศึกษาน้ำหนักโมเลกุลของไกลโคไลซ์โพรดักส์ด้วยเทคนิค GPC

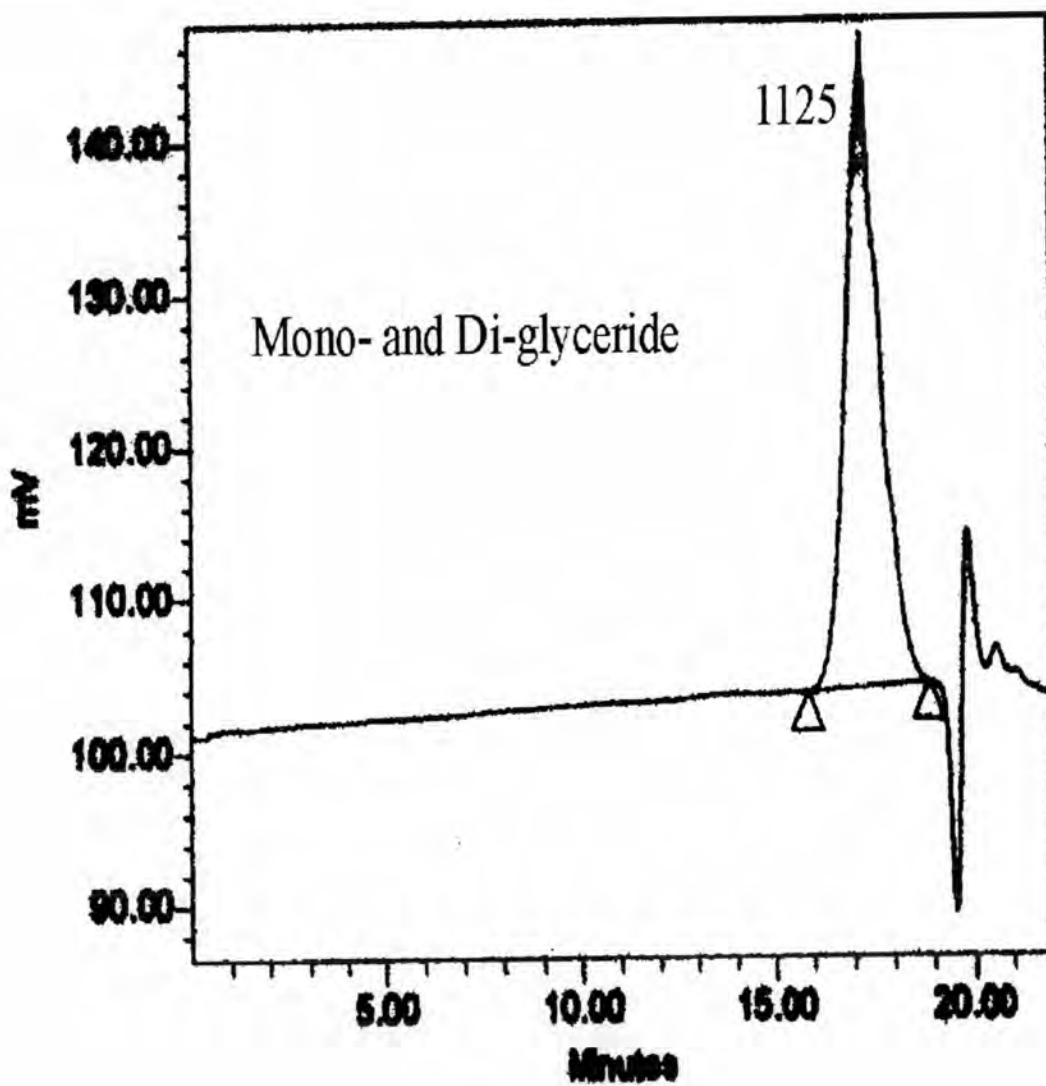


ก.3 FT-IR สเปกตรัมแสดงหมู่ฟังก์ชันของไกลโคไลซิโพรดักส์ และเศษขวดเพตที่ใช้แล้ว

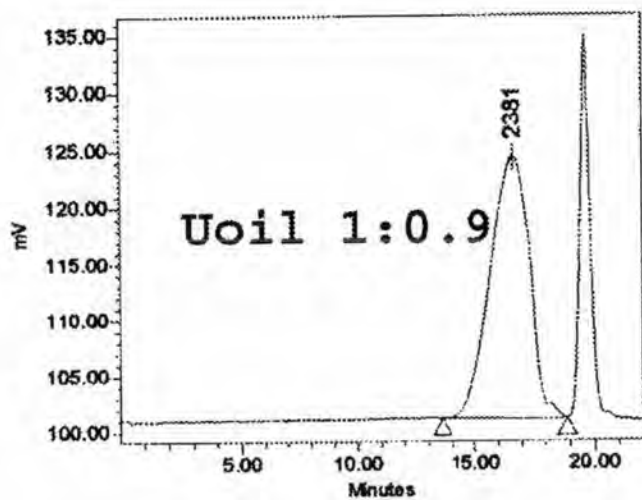
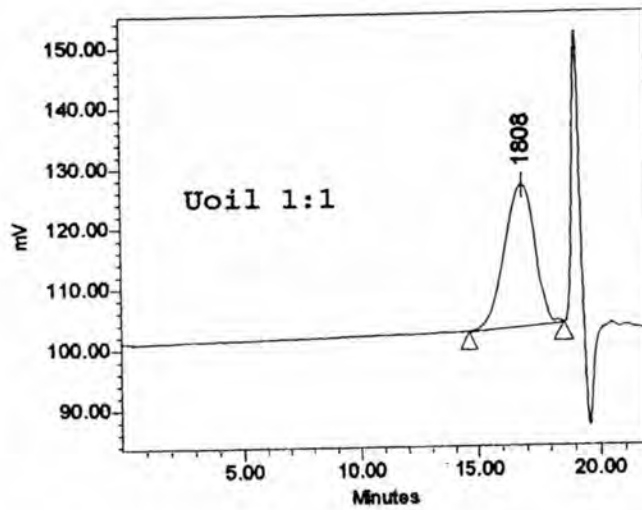
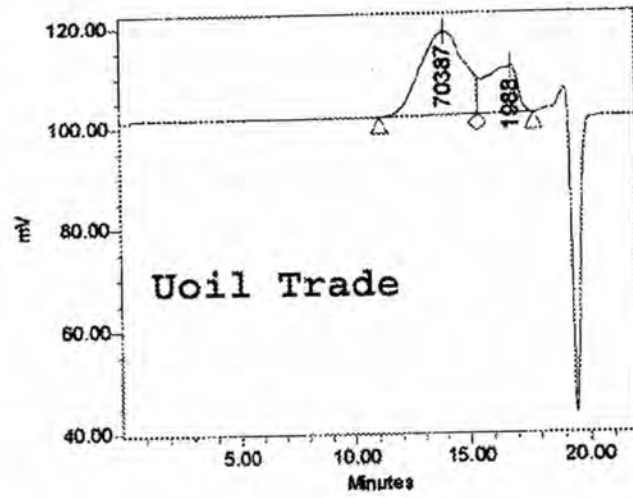


การสังเคราะห์ยูรีเทนออยล์

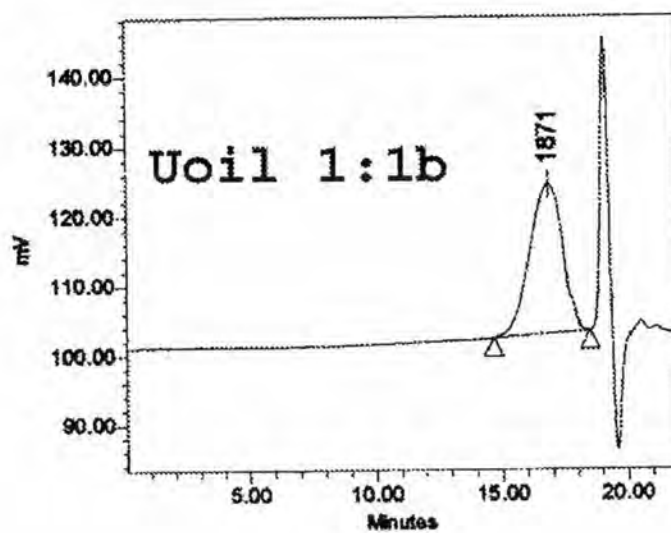
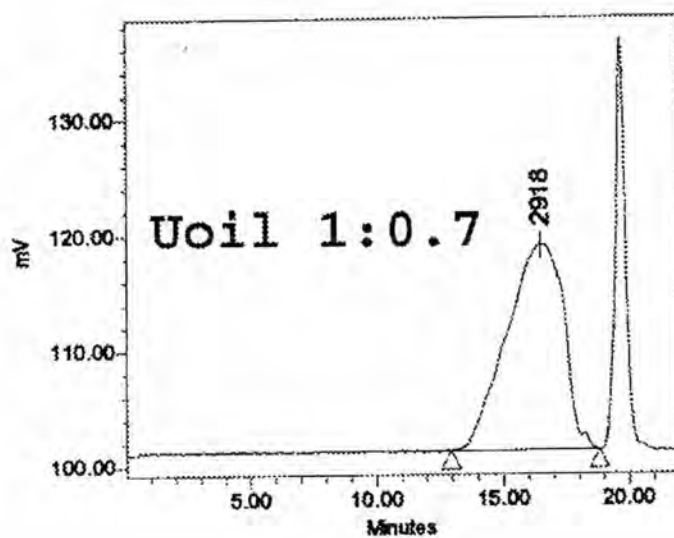
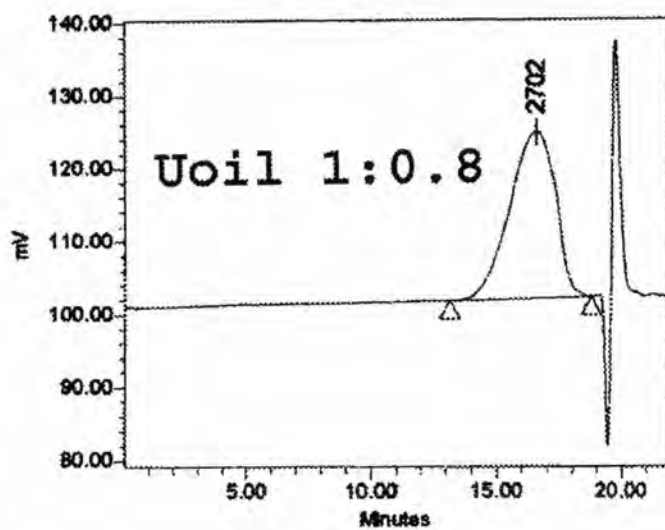
ก.4 โครมาโตแกรมจากการศึกษาน้ำหนักโมเลกุลและการกระจายตัวของน้ำหนักโมเลกุลของมอนอและไดกลีเซอไรต์ด้วยเทคนิค GPC

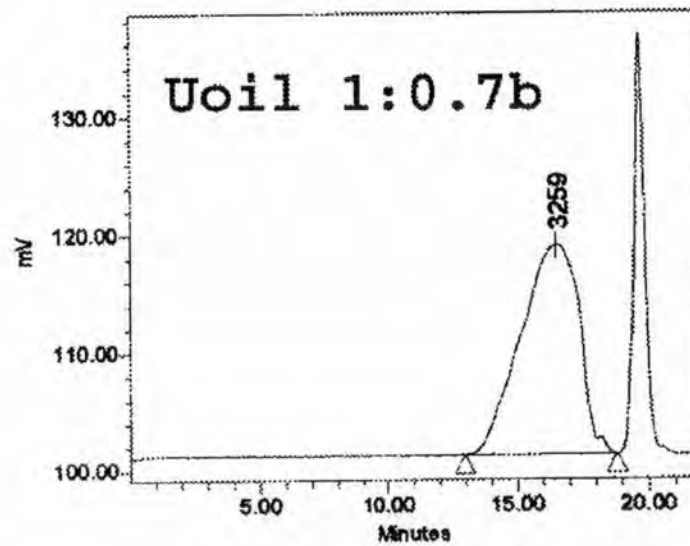
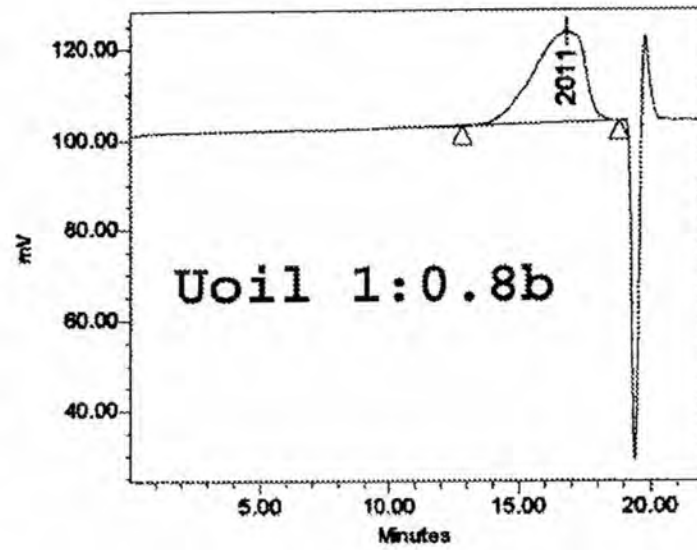
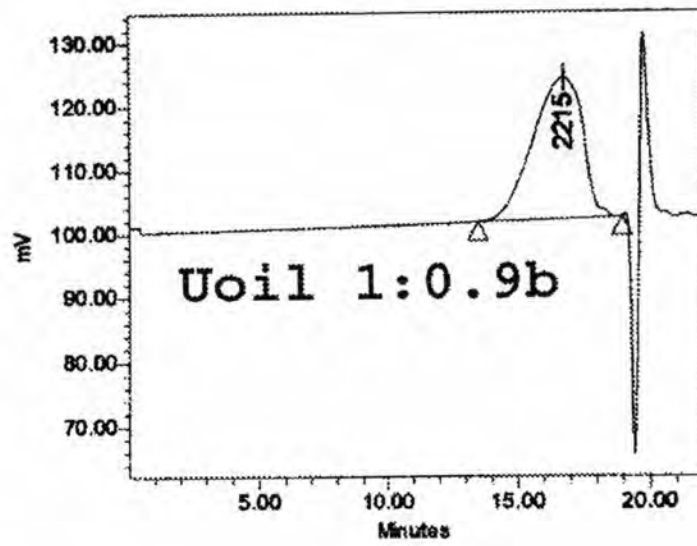


ก.5 โคโรมาโตแกรมจากการศึกษาน้ำหนักโมเลกุลและการกระจายตัวของน้ำหนักโมเลกุลของยูรีเทนออยล์ทางการค้าและที่สังเคราะห์ด้วยเทคนิค GPC



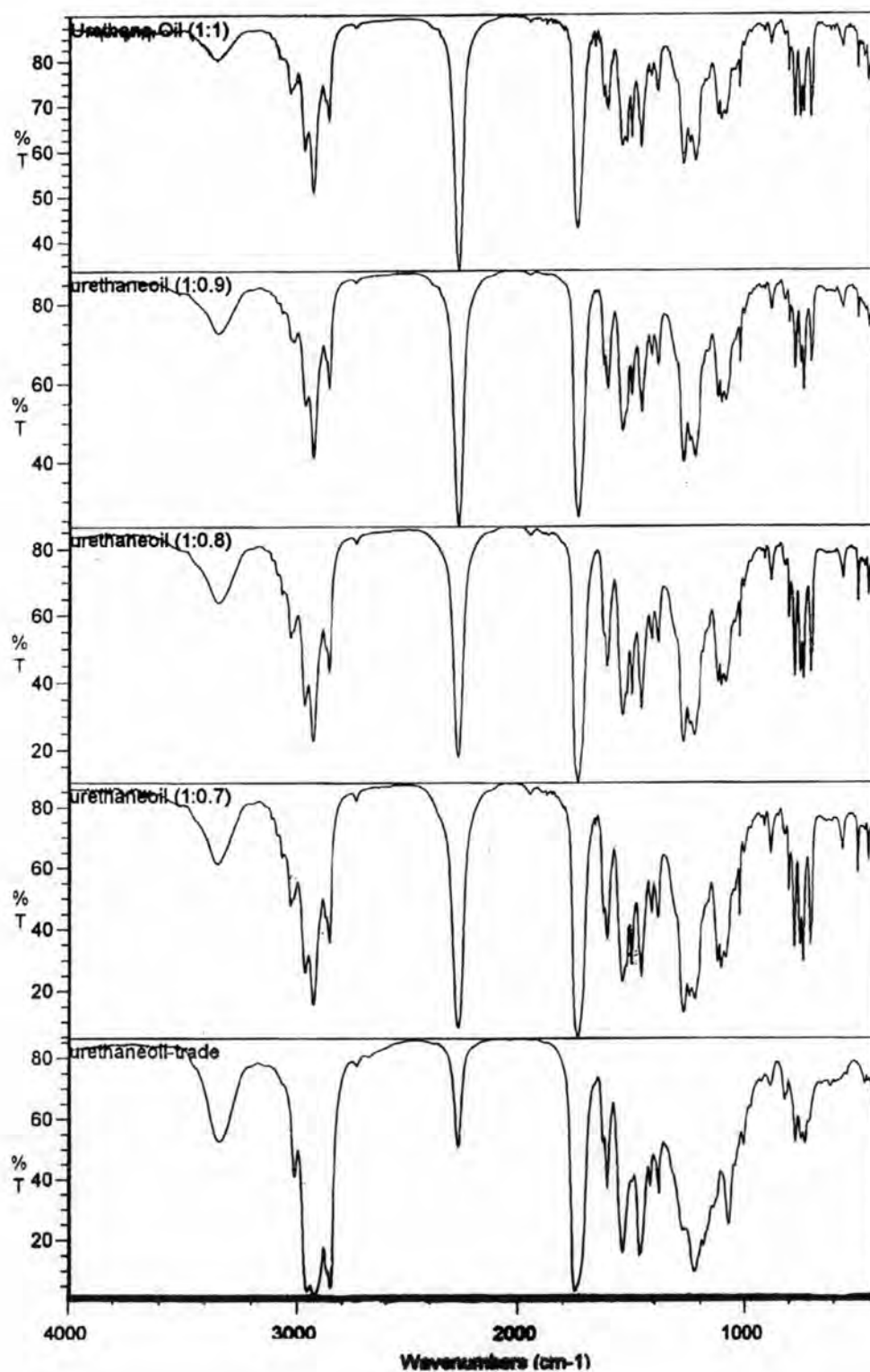






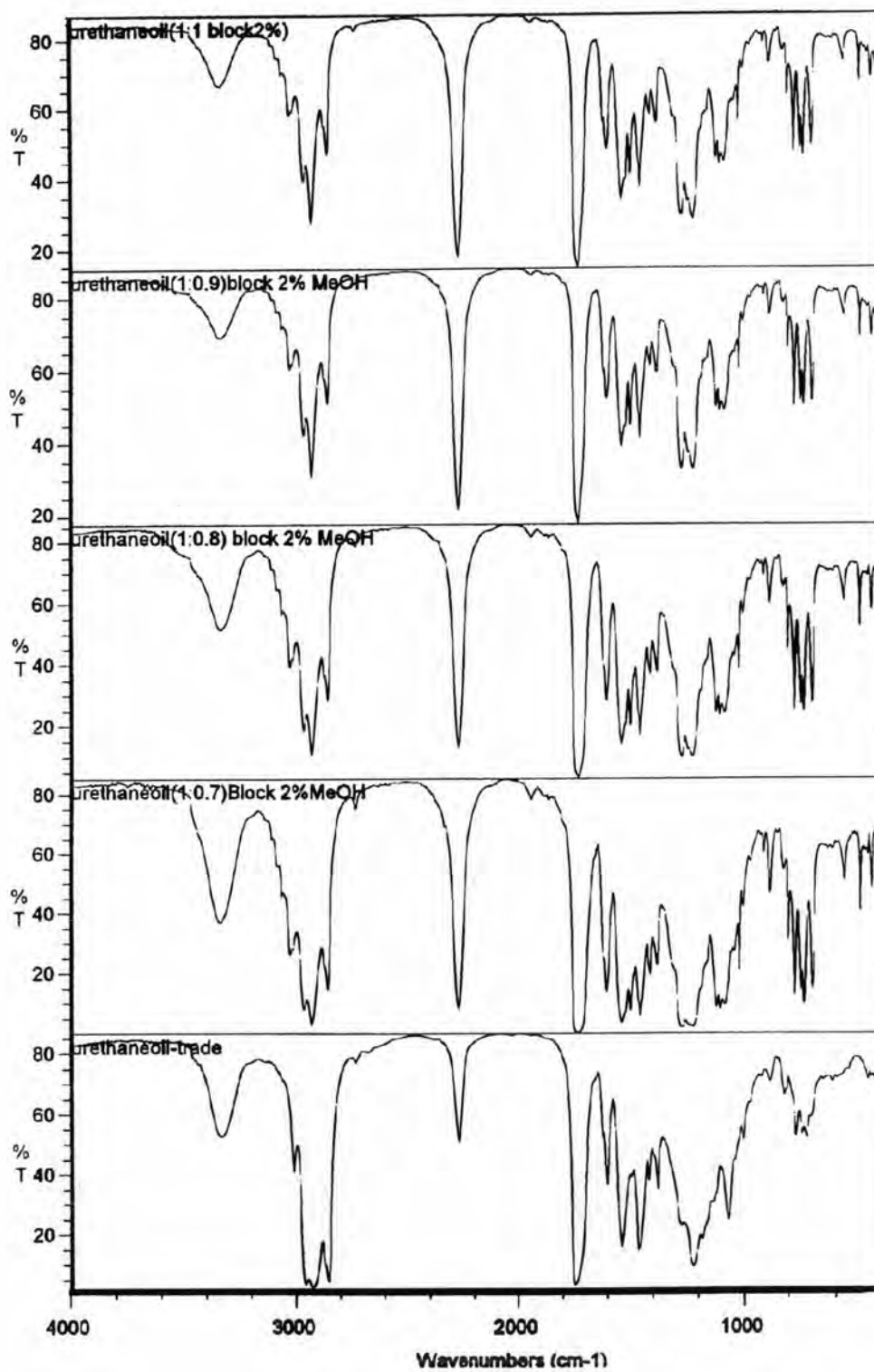
ก.6 FT-IR สเปกตรัมของยูรีเทนออยล์สูตรที่ไม่บดด้วยเมทานอล และยูรีเทนออยล์ทาง

การค้า

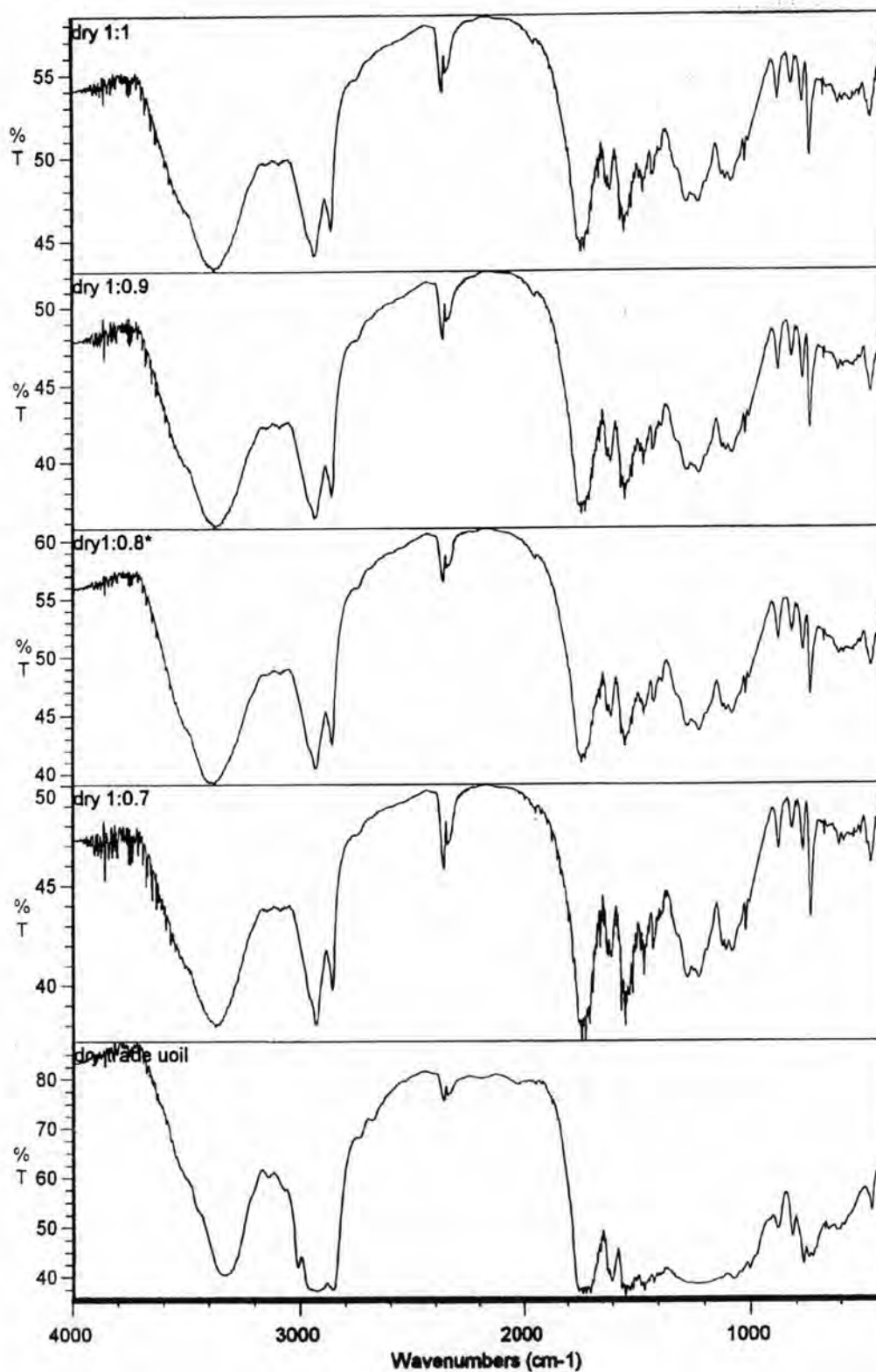


## ก.7 ผล FT-IR สเปกตรัมของยูรีเทนออยล์สูตรบล็อกด้วยเมทานอลและยูรีเทนออยล์ทาง

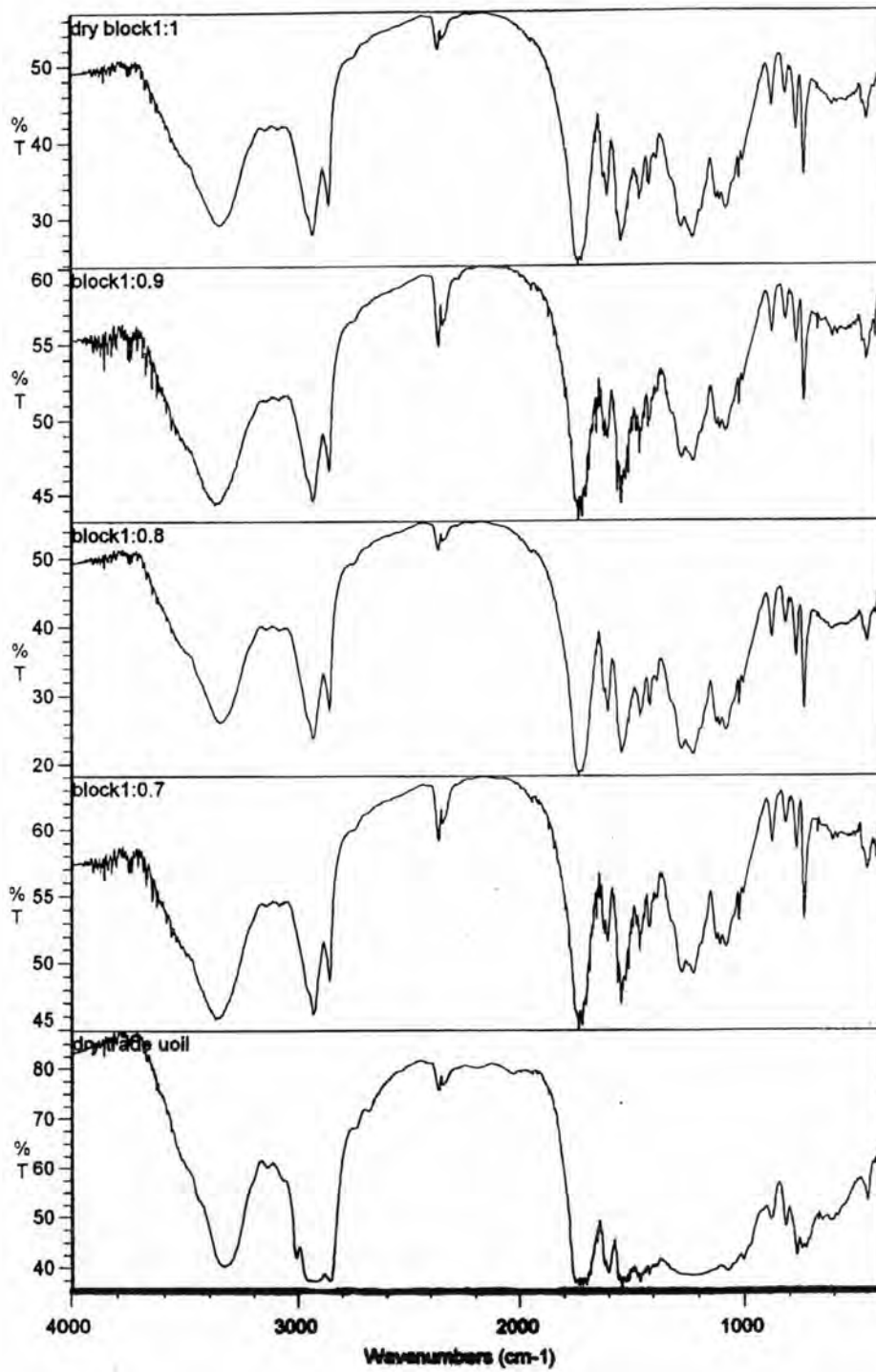
การค้า



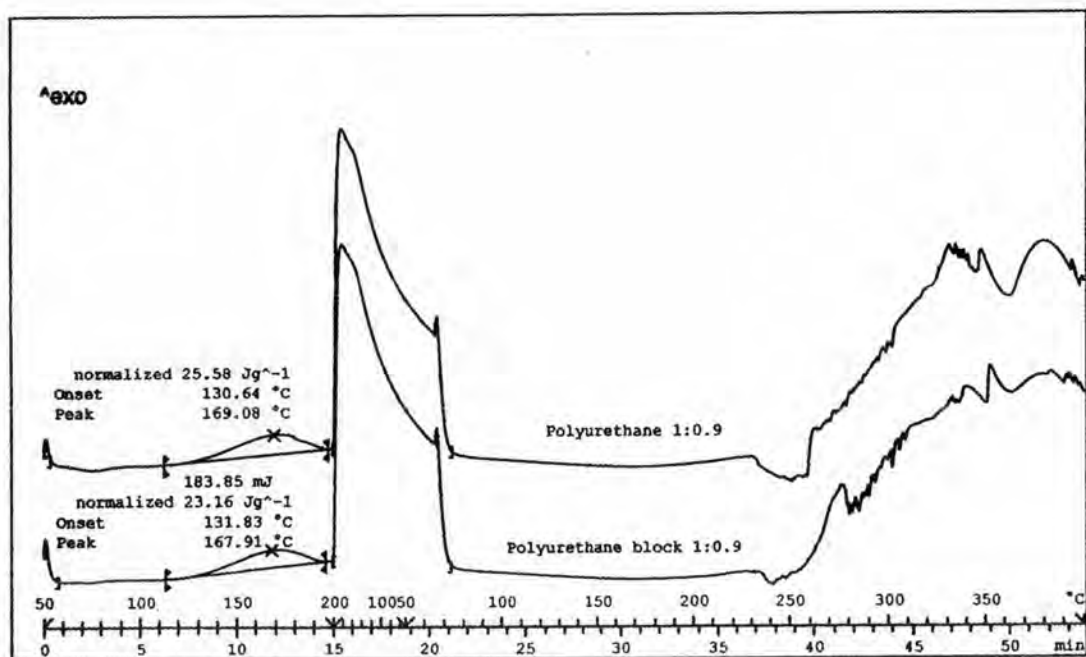
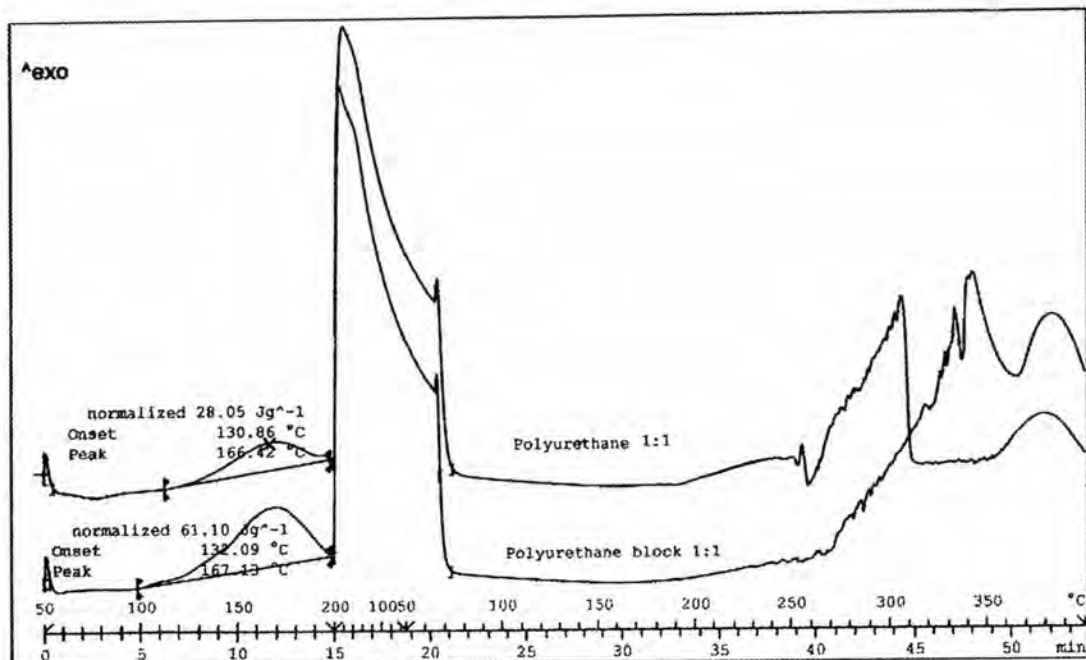
ก.8 FT-IR สเปกตรัมของฟิล์มแห้งของยูรีเทนออยล์สูตรที่ไม่บดด้วยเมทานอลและยูรีเทนออยล์ทางการค้า

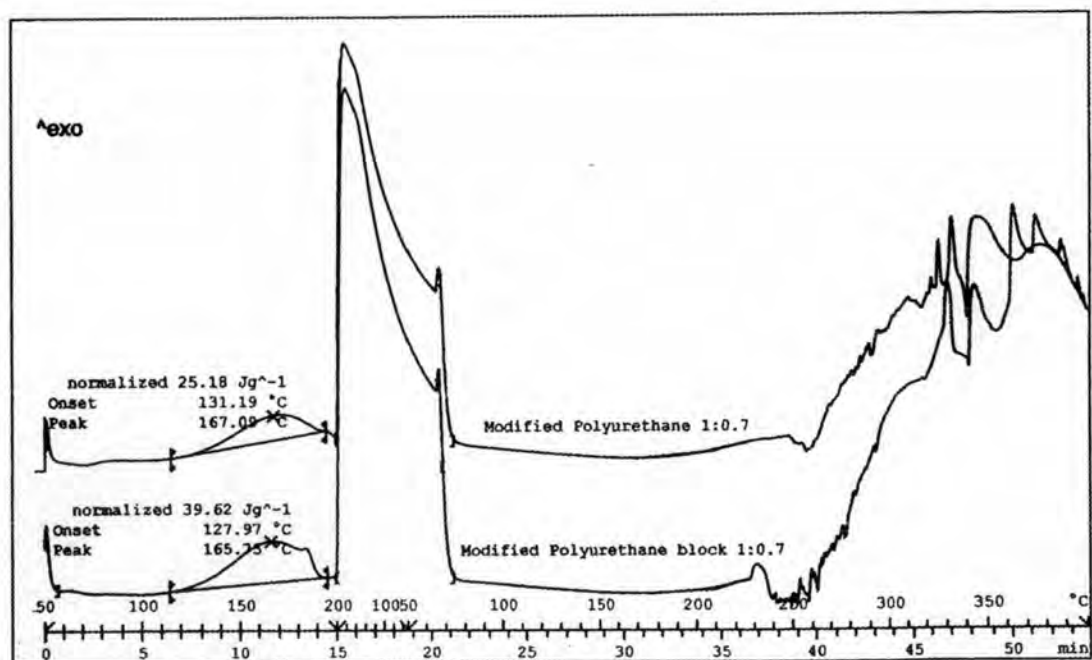
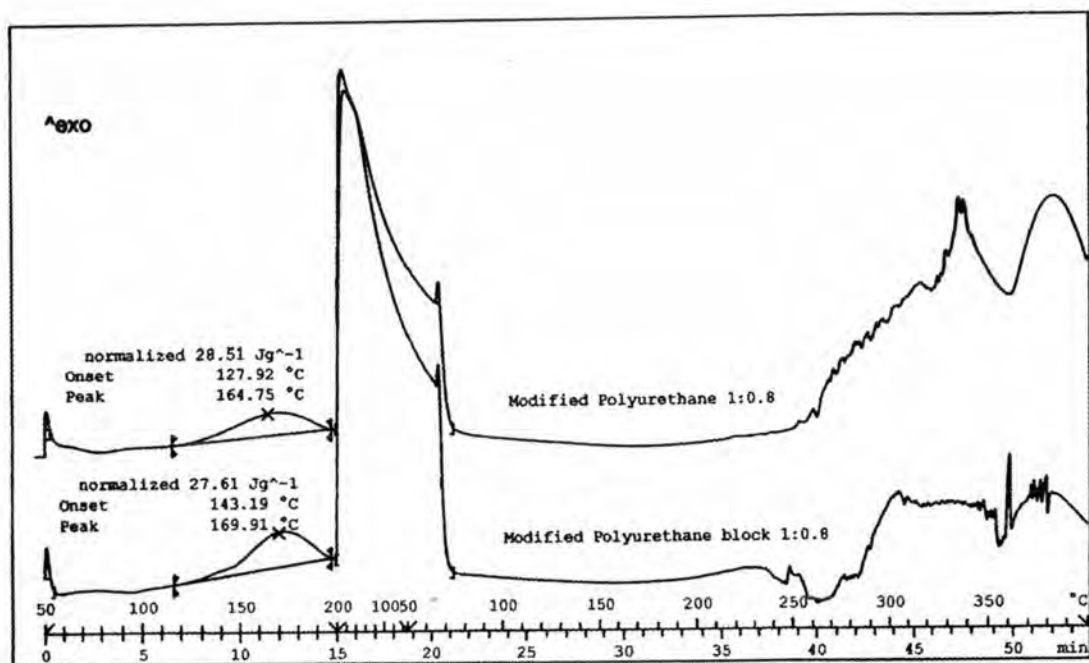


ก.9 FT-IR สเปกตรัมของฟิล์มแห้งยูรีเทนออยล์สูตรบล็อกด้วยเมทานอล และยูรีเทนออยล์  
ทางการค้า

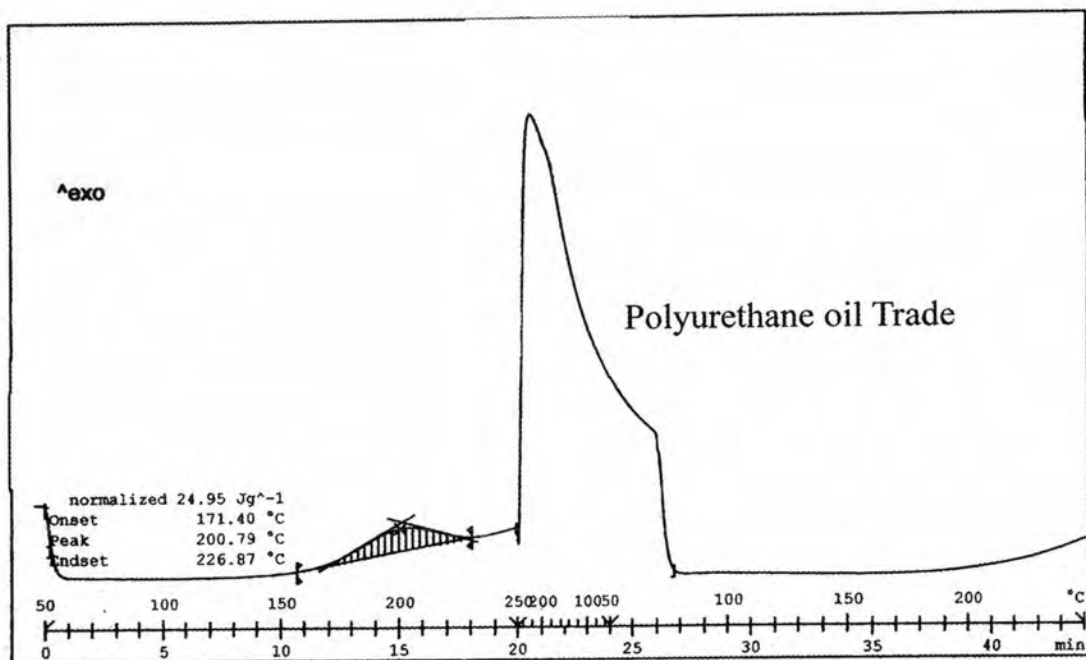


ก.10 เทอร์โมแกรมจากการศึกษาสมบัติทางความร้อนด้วยเทคนิค DSC ของฟิล์มแข็ง ยูรีเทนออยล์ที่สังเคราะห์ได้ และยูรีเทนออยล์ทางการค้า









## ภาคผนวก ข

### การทดสอบสมบัติยูรีเทนออยล์

ข.1 ปริมาณร้อยละของสารที่ระเหยไม่ได้ของยูรีเทนออยล์ (%NV)

สูตร (OH:NCO)	น.น. สารก่อนอบ (g)	น.น. สารหลังอบ (g)	% NV
1:1	2.0200	0.9997	49.49
1:0.9	2.0306	1.0425	51.34
1:0.8	2.0877	1.0904	52.23
1:0.7	2.0094	1.0622	52.86
1:1 b	2.0117	1.0236	50.88
1:0.9 b	2.0350	1.0467	51.43
1:0.8 b	2.1080	1.1184	53.05
1:0.7 b	2.0958	1.1086	52.90

## ข.2 ค่าของกรดของยูรีเทนออยล์

ความเข้มข้นของ KOH = 0.100475 N.

สูตร (OH:NCO)	น.น.สารตัวอย่าง (g)	ปริมาตร KOH (ml)	ค่าของกรด
1:1	ครั้งที่ 1	5.40	3.008
	2	5.35	2.991
	3	5.70	3.187
	เฉลี่ย		3.062
1:0.9	ครั้งที่ 1	4.20	2.339
	2	3.80	2.132
	3	4.00	2.236
	เฉลี่ย		2.236
1:0.8	ครั้งที่ 1	3.70	2.069
	2	3.90	2.166
	3	3.50	1.941
	เฉลี่ย		2.059
1:0.7	ครั้งที่ 1	3.40	1.896
	2	3.90	2.175
	3	3.20	1.793
	เฉลี่ย		1.955
1:1 b	ครั้งที่ 1	3.90	2.184
	2	4.10	2.272
	3	4.00	2.239
	เฉลี่ย		2.232
1:0.9 b	ครั้งที่ 1	3.20	1.766
	2	3.10	1.725
	3	3.30	1.845
	เฉลี่ย		1.779
1:0.8 b	ครั้งที่ 1	1.90	1.061
	2	2.30	1.270
	3	2.20	1.213
	เฉลี่ย		1.181

## ข.2 (ต่อ)

สูตร (OH:NCO)	นน.สารตัวอย่าง (g)	ปริมาตร KOH (g)	ค่าของกรด
1:0.7 b ครั้งที่ 1	10.2049	3.90	2.144
2	10.0999	3.40	1.888
3	10.0725	3.40	1.894
เฉลี่ย			1.198

## ข.3 ระยะเวลาการแห้งตัวของยูรีเทนออยล์ (นาทีก)

สูตร (OH:NCO)	แห้งแตะได้ (touch dry)	แห้งแตะไม่ติด (tack-free dry)	แห้งแข็ง (hard dry)
1:1 ครั้งที่ 1	108	224	426
2	110	240	432
3	103	220	423
เฉลี่ย	107	228	427
1:0.9 ครั้งที่ 1	13	48	281
2	14	49	280
3	12	49	270
เฉลี่ย	13	48	277
1:0.8 ครั้งที่ 1	14	38	127
2	9	35	115
3	11	34	117
เฉลี่ย	11	36	120
1:0.7 ครั้งที่ 1	9	33	110
2	9	31	106
3	12	35	115
เฉลี่ย	10	31	110
1:1 b ครั้งที่ 1	72	193	304
2	77	198	309
3	85	204	311
เฉลี่ย	78	198	308

## ข.3 ( ต่อ )

สูตรยูรีเทนออยล์	แห้งแตะได้ (touch dry)	แห้งแตะไม่ติด (tack free dry)	แห้งแข็ง (hard dry)
1:0.9 b ครั้งที่ 1	10	24	100
2	10	27	108
3	10	22	103
เฉลี่ย	10	24	104
1:0.8 b ครั้งที่ 1	8	19	91
2	9	21	104
3	9	15	100
เฉลี่ย	9	18	98
1:0.7 b ครั้งที่ 1	7	11	94
2	6	13	95
3	7	14	97
เฉลี่ย	7	13	95

## ข.4 ความต้านทานต่อการสึกหรอ

สูตร (OH:NCO)	ความต้านทานต่อการสึกหรอ (รอบ)			
	1	2	3	เฉลี่ย
1:1	47	50	53	50
1:0.9	42	38	40	40
1:0.8	37	40	39	39
1:0.7	48	52	50	50
1:1 b	49	48	51	49
1:0.9 b	50	50	50	50
1:0.8 b	48	53	54	52
1:0.7 b	52	50	48	50

ข.5 การทดสอบความแข็งด้วยวิธีการทุบตี

สูตร (OH:NCO)	ความทนต่อการทุบตี (กรัม)			
	1	2	3	เฉลี่ย
1:1	200	200	200	200
1:0.9	200	200	200	200
1:0.8	100	100	100	100
1:0.7	100	100	100	100
1:1 b	100	100	100	100
1:0.9 b	100	100	100	100
1:0.8 b	100	100	100	100
1:0.7 b	100	100	100	100

ข.6 ความอ่อนตัวของฟิล์ม

สูตร (OH:NCO)	ความอ่อนตัว ( $\phi$ ,mm)			
	1	2	3	เฉลี่ย
1:1	20	20	20	20
1:0.9	20	20	20	20
1:0.8	20	20	20	20
1:0.7	20	20	20	20
1:1 b	20	20	20	20
1:0.9 b	20	20	20	20
1:0.8 b	20	20	20	20
1:0.7 b	20	20	20	20

ข.7 ความตืดแน่น (cross-cut , tape taste, 1 × 1 mm.)

สูตร (OH:NCO)	ครั้งที่ 1	ครั้งที่ 2	ครั้งที่ 3
1:1	5B	5B	5B
1:0.9	5B	5B	5B
1:0.8	4B	4B	4B
1:0.7	3B	3B	3B
1:1 b	4B	4B	4B
1:0.9 b	4B	4B	4B
1:0.8 b	4B	4B	4B
1:0.7 b	4B	4B	4B

ข.9 ความทนต่าง 5% NaOH (นาที)

สูตร (OH:NCO)	ครั้งที่ 1	ครั้งที่ 2	ครั้งที่ 3	เฉลี่ย
1:1	25	28	37	30
1:0.9	28	32	30	30
1:0.8	32	39	34	35
1:0.7	22	25	28	25
1:1 b	30	34	36	33
1:0.9 b	30	33	27	30
1:0.8 b	28	24	23	25
1:0.7 b	25	23	27	25

## ภาคผนวก ค

### การคำนวณ

#### การคำนวณค่าไฮดรอกซิล

ค่าไฮดรอกซิลสามารถคำนวณได้จากความสัมพันธ์ดังต่อไปนี้

$$\text{ไฮดรอกซิลนัมเบอร์} = \frac{(B-A) N \times 56.1}{W}$$

W

เมื่อ B คือ ปริมาตรของ NaOH ที่ใช้ไตเตรต blank (ml)

A คือ ปริมาตรของ NaOH ที่ใช้ไตเตรตสารตัวอย่าง (ml)

N คือ normality ของ NaOH

W คือ น้ำหนักของสารตัวอย่าง (g)

โดยที่ Normality ของ NaOH สามารถคำนวณได้จาก

$$\text{Normality} = \frac{W}{V \times 0.2046}$$

เมื่อ W คือ น้ำหนักของ  $\text{KHC}_8\text{H}_4\text{O}_4$  (g)

V คือ ปริมาตรของ NaOH ที่ใช้ในการไตเตรต  $\text{KHC}_8\text{H}_4\text{O}_4$  (ml)



### การคำนวณปริมาณโทลิลีนไดไอโซไซยาเนต

จากค่าไฮดรอกซิลสามารถคำนวณหาจำนวน mole ของ OH ได้จากความสัมพันธ์

$$\text{จำนวน mole OH} = \frac{\text{ค่าไฮดรอกซิล (mg KOH/g)} \times 10 \times \text{ปริมาณสารตัวอย่าง (g)}}{56.1}$$

เนื่องจาก mole OH = 2 mole TDI

ถ้าใช้อัตราส่วนโดย mole ระหว่าง OH : NCO = 1:1 (สูตร 1:1)

$$\text{จำนวน mole NCO} = \frac{\text{จำนวน mole OH}}{2}$$

$$\frac{\text{ปริมาณ TDI (g)}}{\text{Mw ของ TDI}} = \frac{\text{จำนวน mole OH}}{2}$$

$$\text{ดังนั้นปริมาณ TDI ที่ใช้} = \frac{\text{จำนวน mole OH} \times \text{Mw ของ TDI}}{2}$$

สูตร 1:0.9 (อัตราส่วนโดย mole OH : NCO = 1:0.9)

$$\text{จำนวน mole NCO} = \frac{0.9 \times \text{จำนวน mole OH}}{2}$$

$$\text{ดังนั้นปริมาณ TDI ที่ใช้} = \frac{0.9 \times \text{จำนวน mole OH} \times \text{Mw ของ TDI}}{2} \quad (\text{g})$$

สูตร 1:0.8 (อัตราส่วนโดย mole OH : NCO = 1:0.8)

$$\text{ดังนั้นปริมาณ TDI ที่ใช้} = \frac{0.8 \times \text{จำนวน mole OH} \times \text{Mw ของ TDI}}{2} \quad (\text{g})$$

สูตร 1:0.7 (อัตราส่วนโดย mole OH : NCO = 1:0.7)

$$\text{ดังนั้นปริมาณ TDI ที่ใช้} = \frac{0.7 \times \text{จำนวน mole OH} \times \text{Mw ของ TDI}}{2} \quad (\text{g})$$

## ตัวอย่างสูตรยูรีเทนออยล์

	ร้อยละโดยน้ำหนัก
น้ำมันถั่วเหลือง	20.0
เพนตะอิริททอล	8.9
แคลเซียมออกไซด์	0.015
มิเนอรัลสปิริต	26.0
โทลีนไดไอโซไซยาเนต	19.0
มิเนอรัลสปิริต	26.0
สารเร่งแห้ง	0.06

จากการอ้างอิงสูตรของยูรีเทนออยล์ในข้างต้น สามารถคำนวณปริมาณของส่วนประกอบอื่นๆ เทียบกับปริมาณ TDI ที่ใช้ด้วยความสัมพันธ์ต่อไปนี้

$$\text{ปริมาณน้ำมันถั่วเหลือง (g)} = \frac{20}{19} \times \text{ปริมาณ TDI (g)}$$

$$\text{ปริมาณแคลเซียมออกไซด์ (g)} = \frac{0.015}{19} \times \text{ปริมาณ TDI (g)}$$

$$\text{ปริมาณสารเร่งแห้ง (g)} = \frac{0.06}{19} \times \text{ปริมาณ TDI (g)}$$

$$\text{ปริมาณโทลีน (g)} = \frac{26}{19} \times \text{ปริมาณ TDI (g)}$$

ภาคผนวก ง



## Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature<sup>1</sup>

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 ( $\epsilon$ ) 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<sup>2</sup>

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels<sup>3</sup>

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers<sup>3</sup>

D 2091 Test Method for Print Resistance of Lacquers<sup>4</sup>

#### 2.2 U.S. Government Standards:

Fed. Spec. No. CCC-C-440, Cheesecloth<sup>5</sup>

Fed. Spec. No. CCC-C-419b, Type III, Army Duck<sup>5</sup>

#### 2.3 TAPPI Standards:<sup>6</sup>

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  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  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.<sup>7</sup> 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  $\frac{1}{2}$  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 lx) 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

<sup>1</sup> 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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<sup>2</sup> Annual Book of ASTM Standards, Vol 10.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 06.02.

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

<sup>6</sup> Available from Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

<sup>7</sup> 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 preferably 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 1/8 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

TABLE 1 Recommended Film Thickness of Materials to be Tested<sup>a</sup>

Material	Dry Film Thickness
Drying oils	1.25 ± 0.25 mil (32 ± 6 μm) <sup>b</sup>
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)

<sup>a</sup> This table is a general guide to be used when nothing more specific is agreed upon between the purchaser and the seller.

<sup>b</sup> See 6.1.2 and 7.4.1. Add driers a minimum of 24 h before test.

marked section of the film. The film is considered to have dried dust free when the cotton fibers can be removed by blowing lightly over the surface of the film.

7.4 *Tack-Free Times:*

7.4.1 *Paper Test Method:*

7.4.1.1 *Test Paper*—The test paper shall be K-4 Power Cable Paper<sup>8</sup> that when conditioned in accordance with the TAPPI Standard Method T 402, conforms to the following requirements:

Basis weight (24 by 36/500), lb	90 = 5
Thickness, mils (μm)	6.65 (17)
Air resistance (s/100 cm <sup>2</sup> /in. <sup>2</sup> )	350
Coefficient of static friction <sup>4</sup>	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

<sup>4</sup> All tests except this one shall be run in accordance with Test Method D 202. 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 special 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 to 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 of 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 1 1/2 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<sup>9</sup>)*—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 1/4 by 1 in. (6.4 by 25 mm) in area on the top of the tester, and

<sup>8</sup> Paper meeting these requirements may be obtained from Crocker Technical Papers, Inc., 451 Westminster St., Fitchburg, MA 01420, their Grade R 20-34.  
<sup>9</sup> 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  $\mu$ m) in thickness against one of the 1/4 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<sup>2</sup> (0.24 kg/m<sup>2</sup>) 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 1/2 or 1 lb/in.<sup>2</sup> (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.<sup>10</sup>

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

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

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This standard is subject to revision at any time by the responsible technical committee and is usually reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

# Standard Test Methods for Resistance of Dried Films of Varnishes to Water and Alkali<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. 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<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials<sup>2</sup>

## 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 intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup>

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

## TEST METHOD A—WATER RESISTANCE OF DRIED FILMS

### 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 from commercial No. 31 gage (0.225-mm) bright tin plate, weighing 0.4 to 0.5 lb/ft<sup>2</sup> (1.90 to 2.50 kg/m<sup>2</sup>) and carefully cleaned and dried before use in accordance with Methods B or C of Methods D 609.

### 7. Procedure

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

7.2 Place the panels in a beaker containing about 2.5 in. (65 mm) of water at room temperature, immersing the ends that were uppermost during drying, and allow to remain in the water for 18 h, or other suitable period as agreed upon between the purchaser and the seller.

7.3 Remove the panels from the water, wipe carefully, and allow to dry at room temperature. Note the time required for whitening, if any, to disappear. Blooming, which sometimes occurs on immersion, is considered a type of whitening.

### 8. Report

8.1 Report the results of the water test as follows:

8.1.1 Not visibly affected,

8.1.2 Whitening disappears within 20 min,

8.1.3 Whitening does not disappear within 20 min, but disappears within 2 h,

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

8.1.5 Whitening does not disappear within 24 h.

## TEST METHOD B—DILUTE ALKALI RESISTANCE OF DRIED FILMS

### 9. Reagent

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

<sup>1</sup> 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 D01.33 on Polymers and Resins.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



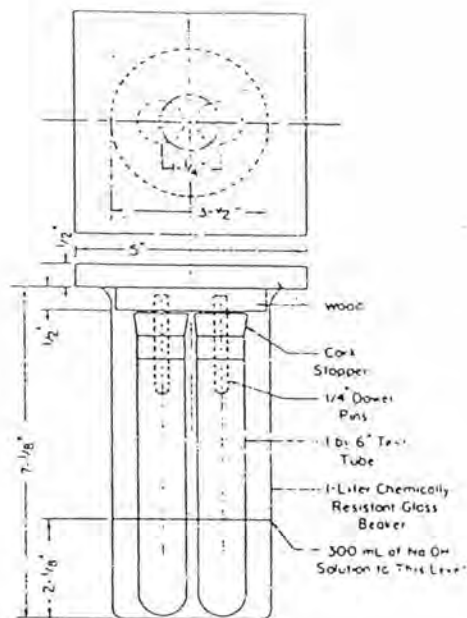
of sodium hydroxide (NaOH) in water and dilute to 1 L.

## 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  $72 \pm 1$  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  $23 \pm 2^\circ\text{C}$ .

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



NOTE—1 in. = 25.4 mm.

FIG. 1 Apparatus for Alkali Resistance Test

TABLE 1 Suggested Test Schedule

Day	Hour	Operation
Monday	8:30 a.m.	coat 16 tubes
	4:00 p.m.	coat 4 tubes
Thursday		dry tubes for 72 h
	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 1/2 h earlier
Friday	4:00 p.m.	start alkali immersion on last 4 tubes
	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 1/2 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

whitening, blistering, or removal. The end point of the test is the number of hours immersion at which the first signs of film whitening, blistering, or removal are noted on the vertical sides of the tubes or on the spherical bottom portion of the tube. The results may be compared with those for materials known to give acceptable performance. For reference work, the test on materials known to be acceptable should be made simultaneously.

## 11. Report

11.1 Report the results of the alkali test as follows:

- 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 to the multiplicity of ambient test conditions.

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## ประวัติผู้วิจัย

นางสาวบัวแก้ว เวสบุตร เกิดเมื่อวันที่ 17 กันยายน พ.ศ.2520 สำเร็จการศึกษา  
ระดับปริญญาตรี วิทยาศาสตร์บัณฑิต (เกียรตินิยมอันดับสอง) สาขาพอลิเมอร์และสิ่งทอ  
จากภาควิชาวัสดุศาสตร์ คณะวิทยาศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย ปีการศึกษา 2540 หลังจากนั้น  
นั้นเข้าศึกษาต่อในหลักสูตรวิทยาศาสตรมหาบัณฑิต สาขาวิทยาศาสตร์พอลิเมอร์ประยุกต์และ  
เทคโนโลยีสิ่งทอ ภาควิชาวัสดุศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย เมื่อภาคต้นของปีการศึกษา 2541  
และสำเร็จการศึกษาในภาคปลายปีการศึกษา 2543 รวมระยะเวลาในการศึกษา 3 ปี