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APPENDICES

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

APPENDIX A

Table A1 Effect of talcum clay content on %yield of dimer at 230 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	4.10	5.00	5.89	6.14	7.87	8.95
2	5.95	6.89	7.64	8.32	9.13	10.26
3	7.05	8.30	8.99	10.46	11.09	12.08
4	7.86	8.79	9.76	10.99	11.69	11.48
5	8.60	8.68	10.20	11.05	12.98	11.95
6	8.85	8.95	10.55	11.14	12.45	11.08

Table A2 Effect of Talcum Clay content on %yield of dimer at 250 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	5.36	6.64	6.49	9.25	10.32	8.47
2	7.28	8.59	9.14	10.96	12.05	11.38
3	8.15	8.48	10.38	12.60	12.68	12.95
4	9.10	10.40	12.47	17.20	16.28	15.40
5	9.48	11.48	14.02	16.95	16.00	16.00
6	10.87	12.34	15.40	17.02	15.43	15.75

Table A3 Effect of Talcum Clay content on %yield of dimer at 270 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	6.98	8.08	9.24	10.40	9.47	10.02
2	8.67	9.14	10.89	9.39	10.98	11.95
3	10.09	10.75	10.50	11.37	13.04	14.30
4	9.48	10.38	11.86	12.40	15.40	15.76
5	10.45	11.84	11.49	12.78	15.96	15.00
6	10.70	11.48	11.45	12.45	15.60	14.75

Table A4 Effect of Ball Clay content on %yield of dimer at 230 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	3.84	4.34	5.74	6.48	7.87	8.38
2	4.79	6.08	7.91	7.90	8.96	9.47
3	5.68	6.74	8.05	9.17	9.75	10.04
4	7.14	7.20	9.68	10.38	10.89	11.35
5	8.68	8.97	9.49	11.62	12.41	10.49
6	9.61	9.70	10.74	11.49	12.49	12.45

Table A5 Effect of Ball Clay content on %yield of dimer at 250⁰C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	6.58	7.13	7.54	7.65	9.00	11.04
2	7.64	8.08	8.63	9.52	10.40	11.84
3	9.58	10.21	10.55	10.60	11.96	13.12
4	9.95	10.42	9.80	12.55	14.55	14.30
5	10.36	11.05	11.84	12.35	15.60	14.92
6	10.54	11.51	11.25	12.55	15.55	13.60

Table A6 Effect of Ball Clay content on %yield of dimer at 270⁰C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	7.18	8.60	9.46	10.20	9.50	10.65
2	7.85	8.50	10.25	10.85	10.48	11.25
3	9.31	10.32	11.90	12.75	12.39	12.94
4	10.40	11.85	12.64	12.56	13.85	12.48
5	10.55	10.90	11.45	12.90	13.47	13.61
6	10.68	10.95	12.15	12.75	13.50	13.50

Table A7 Effect of China Clay content on %yield of dimer at 230 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	3.25	3.60	5.20	6.24	6.46	7.24
2	3.85	3.49	6.31	7.26	7.35	8.28
3	4.16	5.35	6.85	7.40	7.49	9.45
4	5.45	6.28	7.96	9.09	8.40	9.49
5	6.28	8.10	8.58	8.47	9.45	10.35
6	7.14	8.74	8.90	9.15	9.20	11.15

Table A8 Effect of China Clay content on %yield of dimer at 250 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	4.42	5.64	6.86	6.10	6.85	6.90
2	5.16	7.24	8.62	7.03	6.45	7.45
3	6.75	7.64	8.48	8.96	8.50	9.30
4	7.30	8.42	9.45	10.47	9.80	9.45
5	8.12	8.89	9.43	9.49	10.80	9.50
6	8.10	9.75	10.25	9.56	10.55	9.60

Table A9 Effect of China Clay content on %yield of dimer at 270 °C under various times.

Time(hr)	%Yield of Dimer					
	10%wt	15%wt	20%wt	25%wt	30%wt	35%wt
1	5.30	6.32	7.05	6.48	7.85	6.55
2	7.25	8.41	8.48	7.43	7.48	6.98
3	8.64	9.25	9.36	9.45	8.45	8.24
4	8.49	10.10	10.55	9.60	10.13	10.54
5	9.60	11.55	10.00	11.64	10.62	9.63
6	8.60	10.85	9.85	10.58	10.20	9.55

Table A10 Effect of cobalt naphthenate content on %yield of dimer at room temperature under various times.

Time(hr)	%Yield of Dimer			
	0.03%wt	0.05%wt	0.07%wt	0.09%wt
72	16.36	20.18	22.12	24.35
96	32.40	38.46	34.40	37.12
120	40.16	44.45	44.65	44.85
144	37.89	42.36	42.68	42.25
168	33.24	37.29	36.75	38.40

Table A11 Effect of cobalt naphthenate content on %yield of dimer at 40 °C under various times.

Time(hr)	%Yield of Dimer			
	0.03%wt	0.05%wt	0.07%wt	0.09%wt
72	19.36	22.95	25.35	24.40
96	30.35	35.64	35.41	36.62
120	39.40	42.14	40.44	41.05
144	37.25	40.36	39.40	39.30
168	33.22	36.42	33.39	34.25

Table A12 Effect of cobalt-naphthenate content on %yield of dimer at 50 °C under various times.

Time(hr)	%Yield of Dimer			
	0.03%wt	0.05%wt	0.07%wt	0.09%wt
24	19.00	21.40	23.05	22.48
48	24.32	32.15	30.38	31.36
72	32.41	38.36	39.24	38.40
96	30.05	37.25	36.44	36.48
120	30.45	32.40	30.39	29.34

Table A13 Effect of cobalt naphthenate content on %yield of dimer at 60 °C under various times.

Time(hr)	%Yield of Dimer			
	0.03%wt	0.05%wt	0.07%wt	0.09%wt
12	13.45	15.28	18.37	20.24
24	22.36	24.75	26.10	27.48
36	26.95	30.62	31.05	30.48
48	32.45	35.55	34.45	35.08
60	31.38	34.15	32.56	33.00

Table A14 Effect of cobalt naphthenaate content on %yield of dimer at 70 °C under various times.

Time(hr)	%Yield of Dimer			
	0.03%wt	0.05%wt	0.07%wt	0.09%wt
12	17.42	19.45	22.40	21.49
24	25.58	26.68	28.19	30.15
36	29.34	32.36	31.29	35.26
48	31.46	33.95	33.41	31.48
60	29.30	28.49	27.37	26.30

Table A15 Effect of TBHP content on %yield of dimer at room temperature, cobalt-naphthenate content 0.05%wt under various times.

Time(hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
24	21.42	25.00	27.34
48	42.31	46.84	49.28
72	56.85	61.08	63.40
96	54.95	58.00	60.05
120	50.00	50.88	51.00

Table A16 Effect of TBHP content on %yield of dimer at 60 °C, cobalt-naphthenate content 0.05%wt under various times.

Time(hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
8	24.40	26.43	28.00
16	33.05	32.40	34.20
24	38.74	44.28	43.49
32	36.95	40.35	41.05
40	35.00	37.14	38.44

Table A17 Effect of TBHP content on %yield of dimer at room temperature, cobalt-naphthenate content 0.07%wt under various times.

Time(hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
24	23.45	29.30	26.35
48	46.32	51.28	48.25
72	60.00	66.09	64.18
96	56.15	62.45	59.00
120	49.95	57.15	53.49

Table A18 Effect of TBHP content on %yield of dimer at 60 °C, cobalt-naphthenate content 0.07%wt under various times.

Time(hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
8	21.42	23.00	20.40
16	29.36	31.40	30.05
24	38.45	40.28	40.98
32	36.30	37.36	35.46
40	32.25	31.42	30.08

Table A19 Effect of TBHP content on %yield of dimer at room temperature, cobalt naphthenate content 0.09%wt under various times.

Time(hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
24	25.40	27.39	29.25
48	43.35	48.28	51.46
72	61.23	63.75	62.49
96	58.45	59.47	56.80
120	49.15	52.04	49.40

Table A20 Effect of TBHP content on %yield of dimer at 60 °C, cobalt naphthenate content 0.09%wt under various times.

Time (hr)	%Yield of Dimer		
	1.0%wt	1.5%wt	2.0%wt
8	20.40	24.16	21.40
16	29.35	32.30	31.27
24	37.15	41.05	39.09
32	36.00	35.42	34.48
40	32.08	31.25	29.45

Table A21 Effect of Clay content on %yield of dimer at 60 °C, 24 hr., Cobalt Naphthenate 0.05%wt and TBHP 1.5%wt to use Talcum clay, Ball clay and China clay.

Clay (%wt)	%Yield of Dimer		
	Talcum	Ball	China
10	32.25	28.45	25.65
15	35.28	29.38	28.05
20	34.45	31.27	28.89
25	34.05	28.09	28.36
30	29.05	26.16	26.68
35	22.16	20.00	20.05

Table A22 Effect of clay content on %yield of dimer at 250 °C for 4 hr.

%Weight	%Yield of Dimer		
	Talcum	Ball	China
10	9.10	7.95	7.00
15	9.95	8.42	7.75
20	12.40	9.80	9.45
25	17.10	12.55	10.47
30	15.85	14.55	9.80
35	15.46	14.30	9.45

Table A23 Effect of temperature on %yield of dimer at 4 hr. and 25%wt clay.

Temp. (°C)	%Yield of Dimer		
	Talcum	Ball	China
230	11.46	9.85	9.09
250	17.20	12.55	10.47
270	13.45	11.55	9.60

Table A24 Effect of reaction time on %yield of dimer at 250 °C and 25%wt clay.

Time (hr.)	%Yield of Dimer		
	Talcum	Ball	China
1	9.40	7.65	6.10
2	10.96	9.52	7.03
3	12.60	10.60	8.96
4	17.20	12.55	10.47
5	16.95	12.35	9.49
6	17.02	12.55	9.56

Table A25 Average data of Iodine Value.

Properties	Iodine Value (I.V)			
	1	2	3	Average
Methyl oleate	80.91	81.58	81.32	81.27±0.32
Dimer A	51.92	52.01	51.86	51.93±0.32
Dimer B	95.53	95.41	95.13	95.36±0.32
Dimer C	96.89	96.07	96.42	96.46±0.32
Dimer D	95.76	95.46	95.81	95.67±0.32
Dimer E	96.36	96.13	96.64	96.37±0.32
Dimer F	96.47	97.02	96.73	96.74±0.32

Dimer A = Dimer of methyl oleate using clay.

Dimer B = Dimer of methyl oleate using cobalt naphthenate at room temperature.

Dimer C = Dimer of methyl oleate using cobalt naphthenate at 60 °C.

Dimer D = Dimer of methyl oleate using TBHP and cobalt naphthenate at room temperature.

Dimer E = Dimer of methyl oleate using TBHP and cobalt naphthenate at 60 °C.

Dimer F = Dimer of methyl oleate using TBHP, cobalt naphthenate and clay at 60 °C.

APPENDIX B

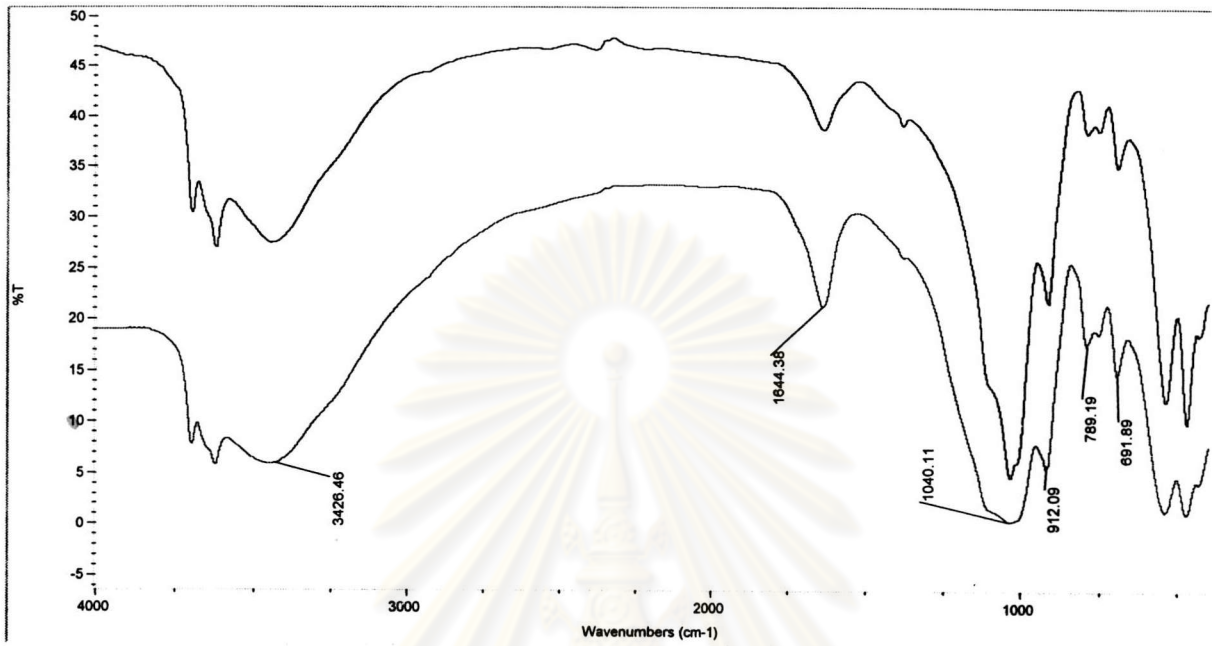


Figure B1 FTIR spectra of china (a) non-acid activated ; (b) acid activated with H₂SO₄

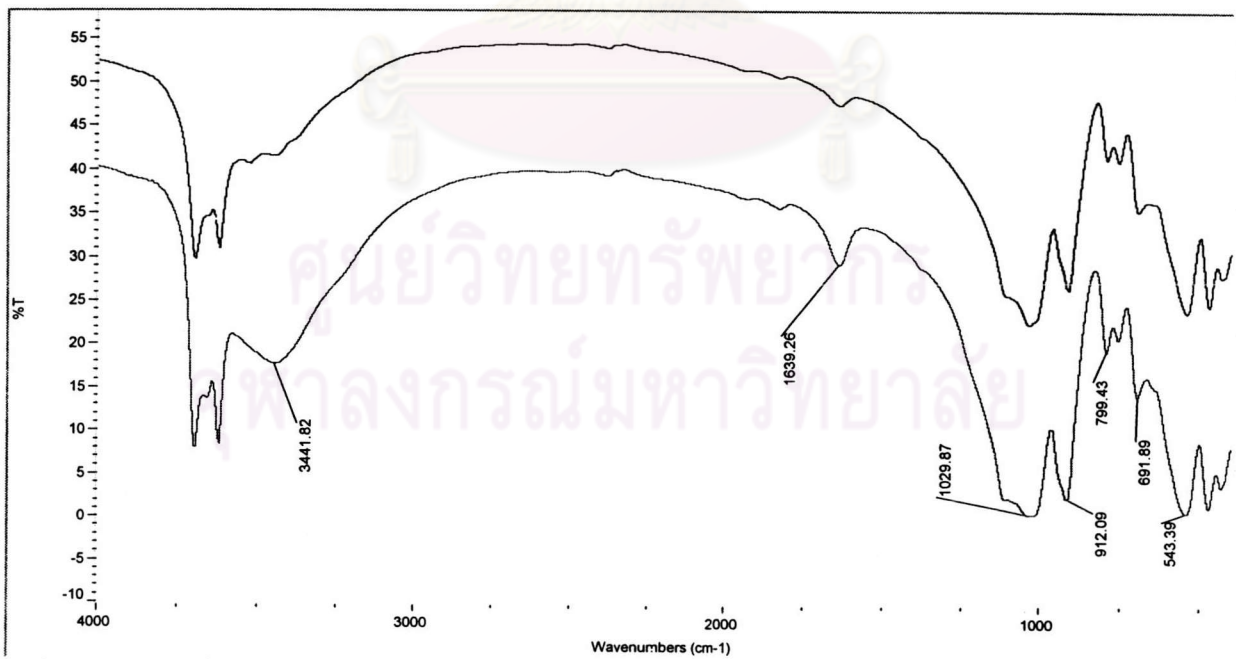


Figure B2 FTIR spectra of ball clay (a) non-acid activated ; (b) acid activated with H₂SO₄

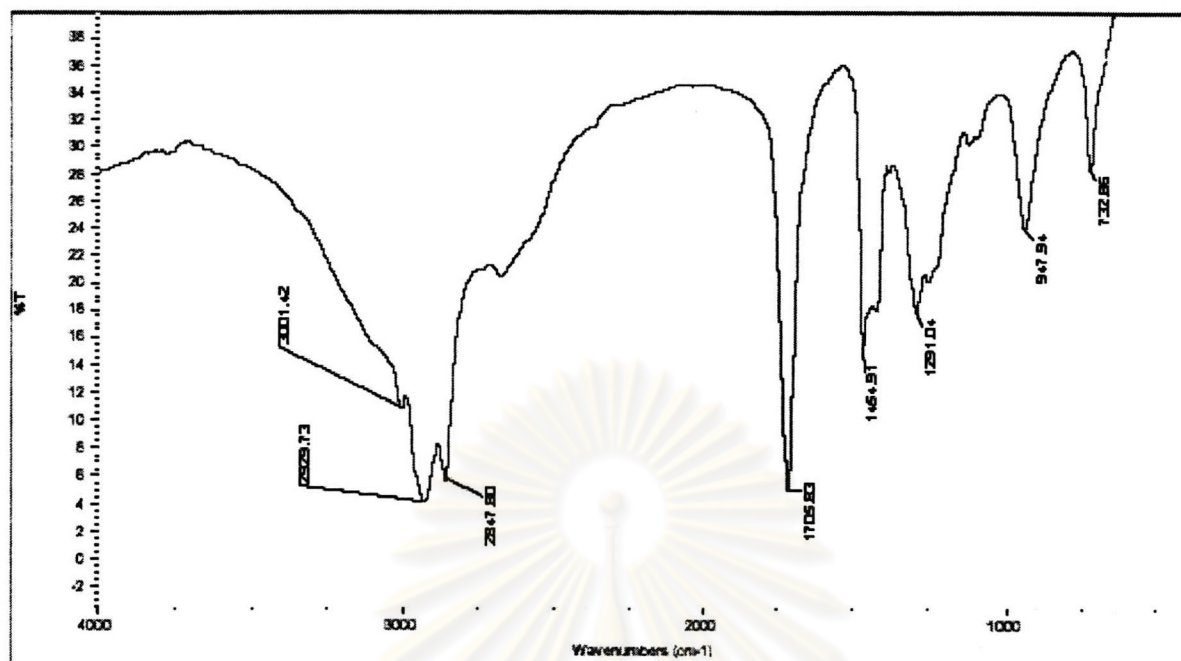


Figure B3 FTIR spectrum of oleic acid (NaCl)

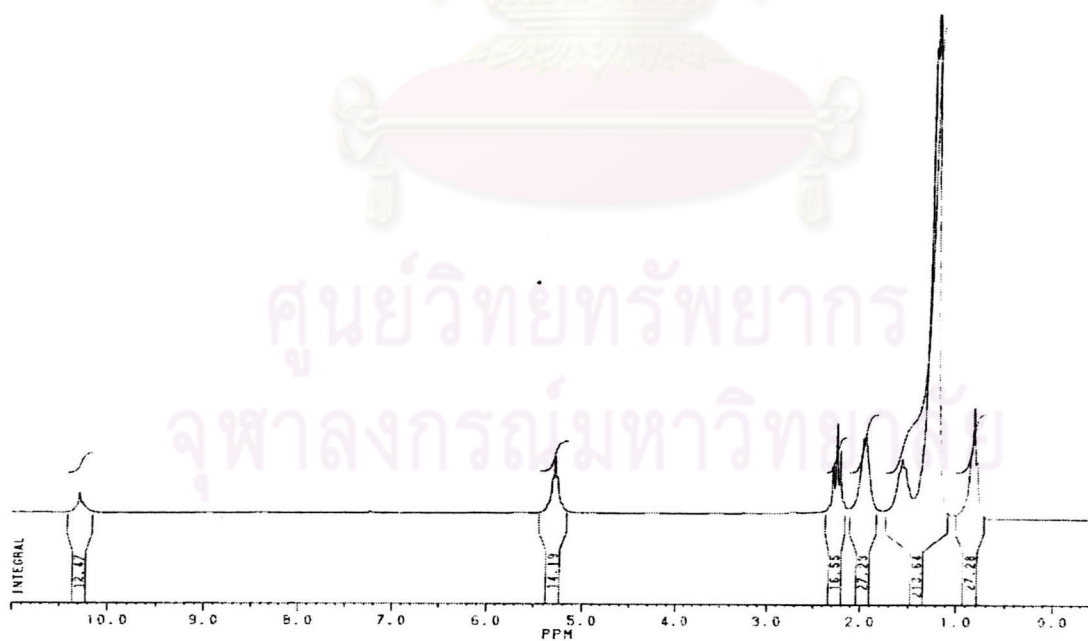


Figure B4 ¹H-NMR spectrum of oleic acid (CDCl₃)

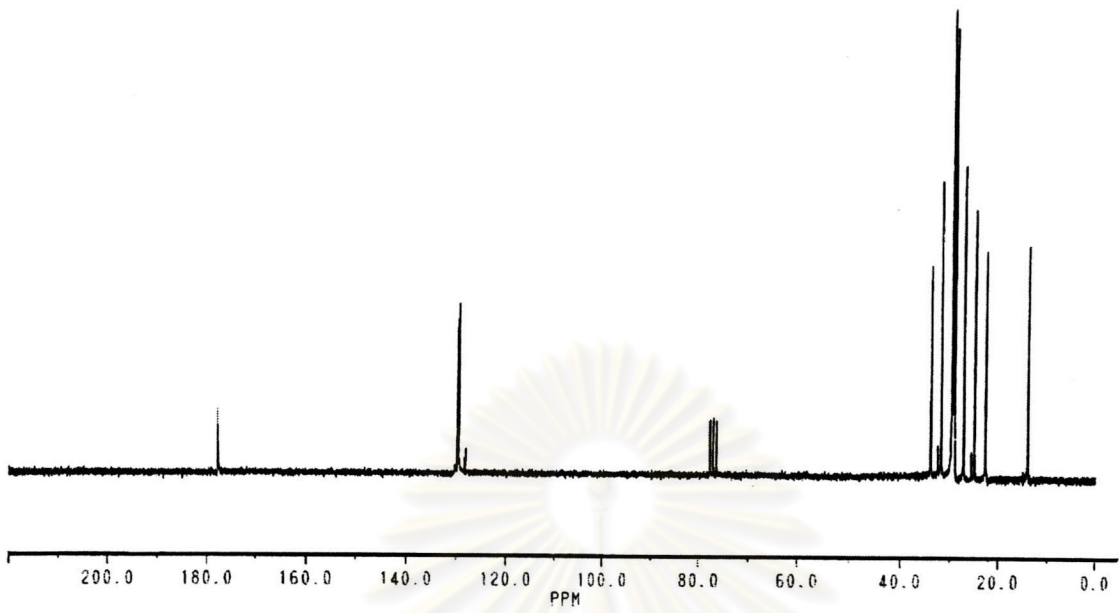


Figure B5 ^{13}C -NMR spectrum of oleic acid (CDCl_3)

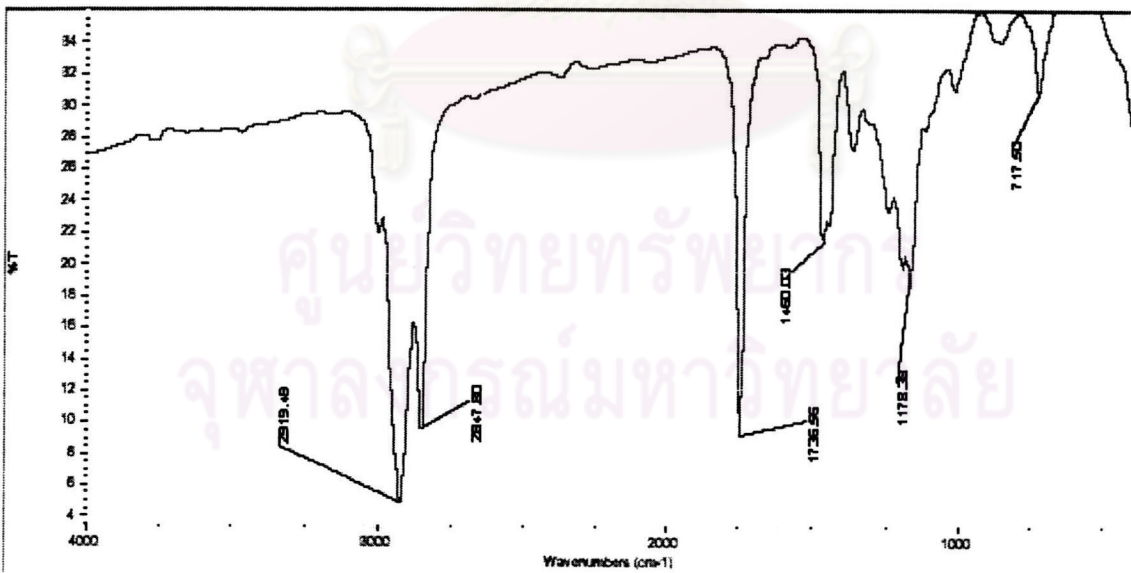


Figure B6 FTIR spectrum of methyl oleate (NaCl)

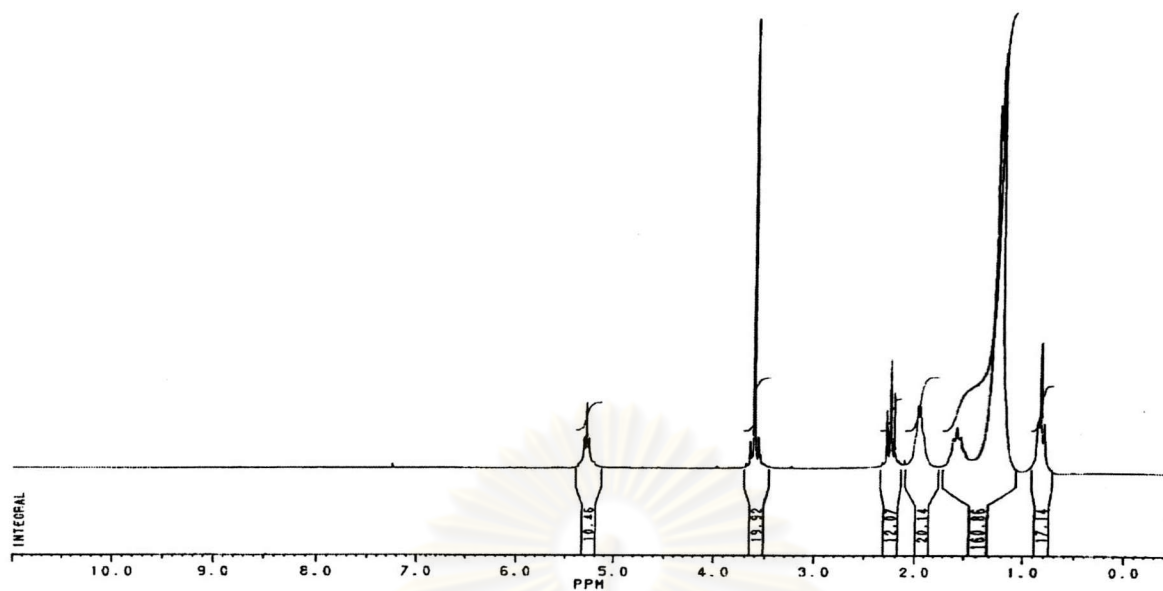


Figure B7 ^1H -NMR spectrum of methyl oleate (CDCl_3)

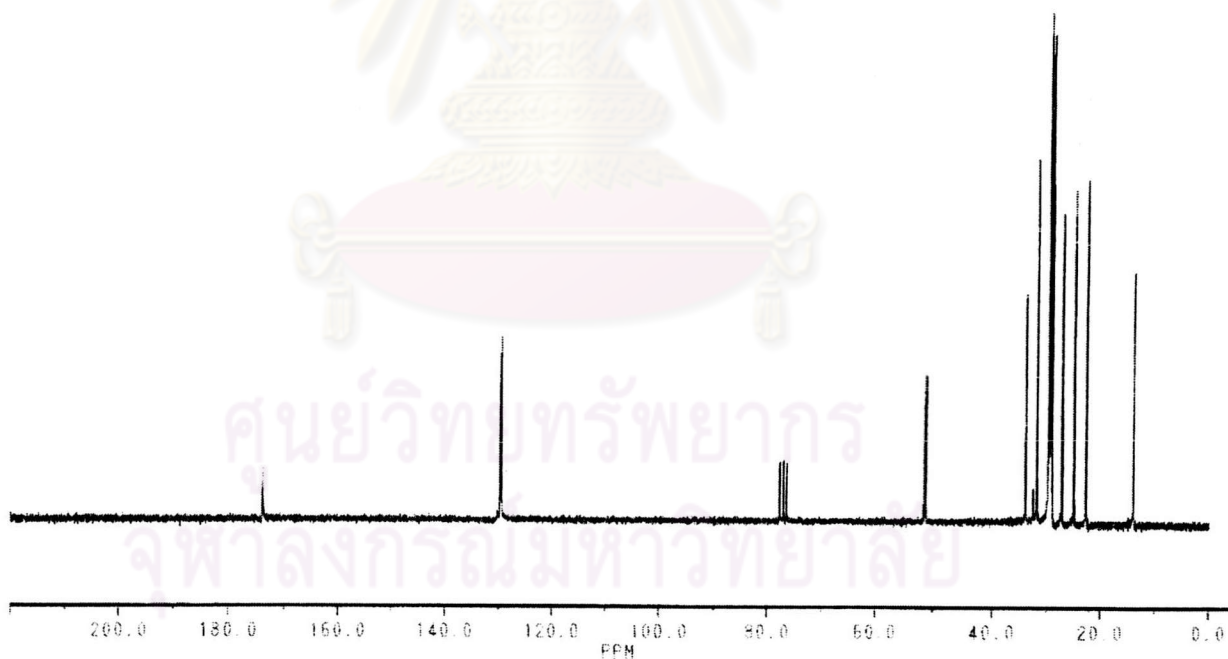


Figure B8 ^{13}C -NMR spectrum of methyl oleate (CDCl_3)

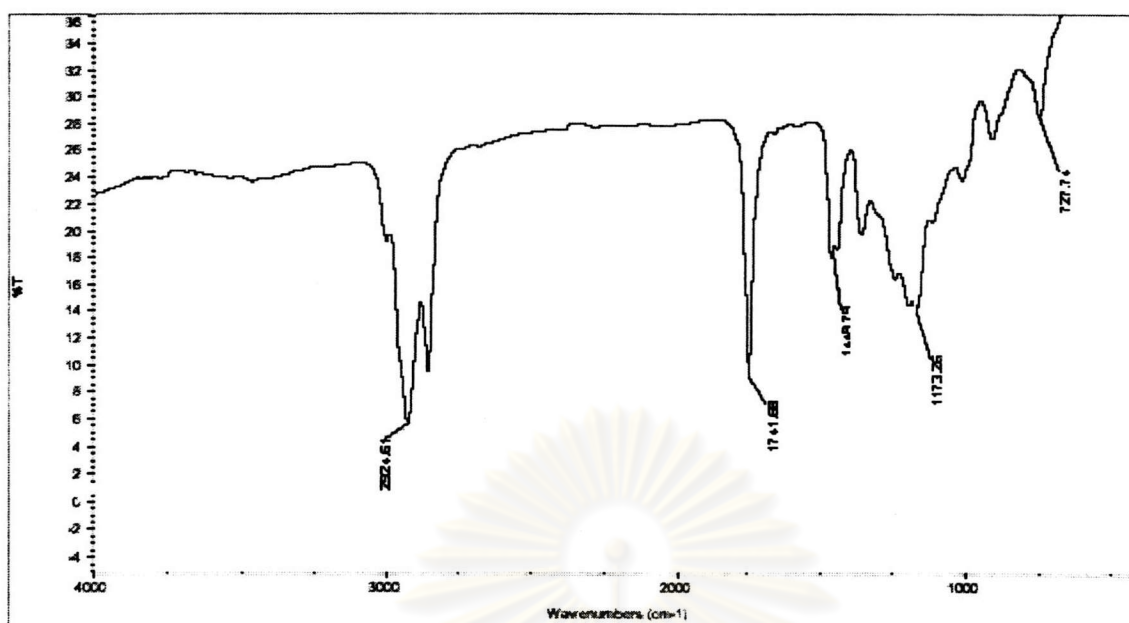


Figure B9 FTIR spectrum of dimer using clay (NaCl)

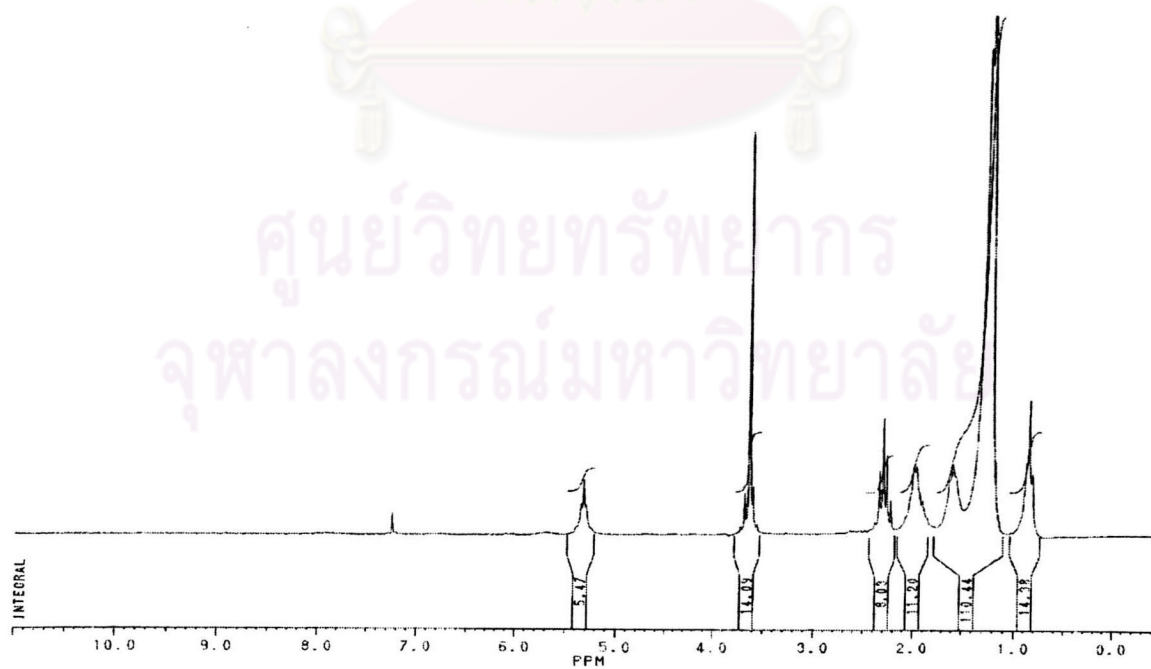


Figure B10 ¹H-NMR spectrum of dimer using clay (CDCl₃)

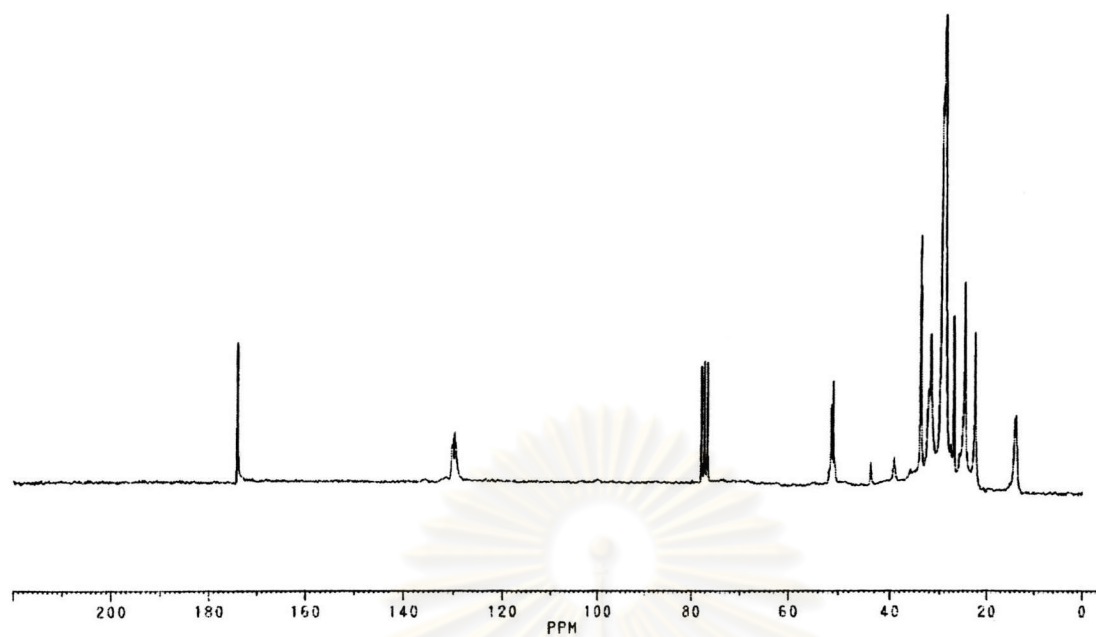


Figure B11 ^{13}C -NMR spectrum of dimer using clay (CDCl_3)

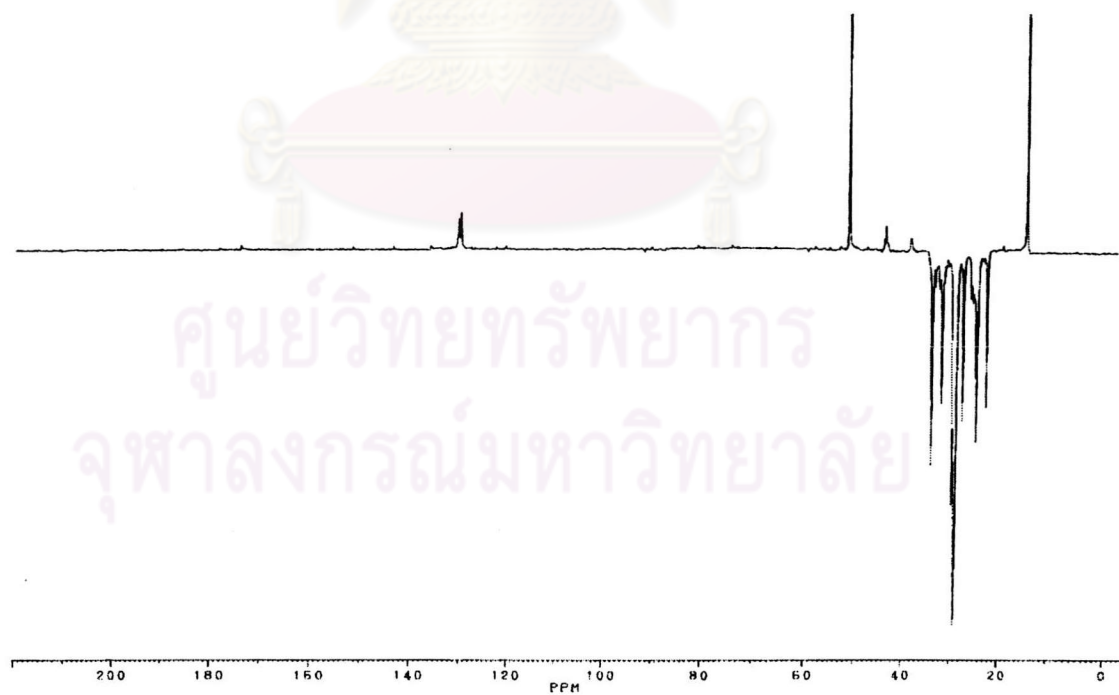


Figure B12 DEPT 135 spectrum of dimer using clay (CDCl_3)

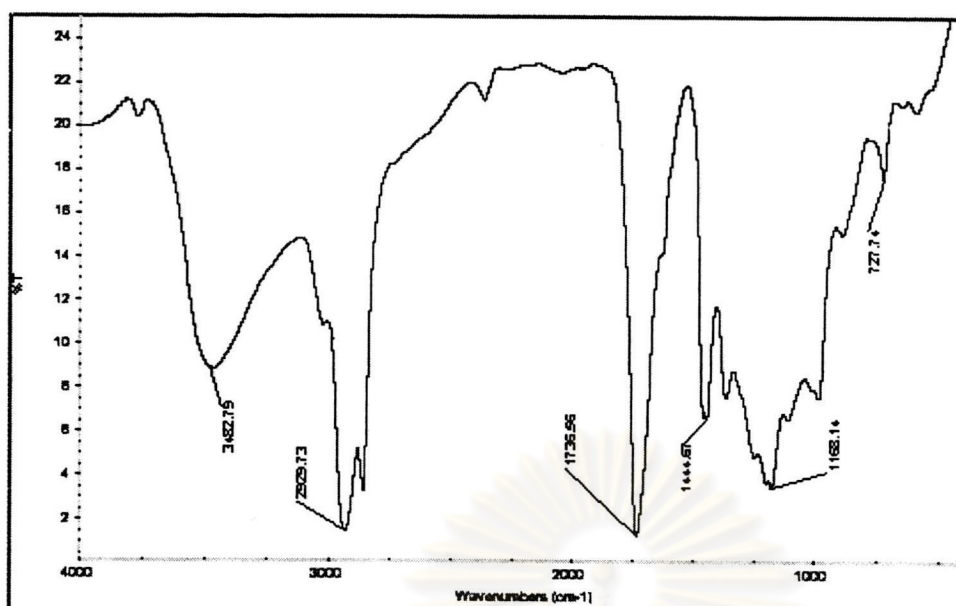


Figure B13 FTIR spectrum of dimer using cobalt naphthenate at room temperature (NaCl)

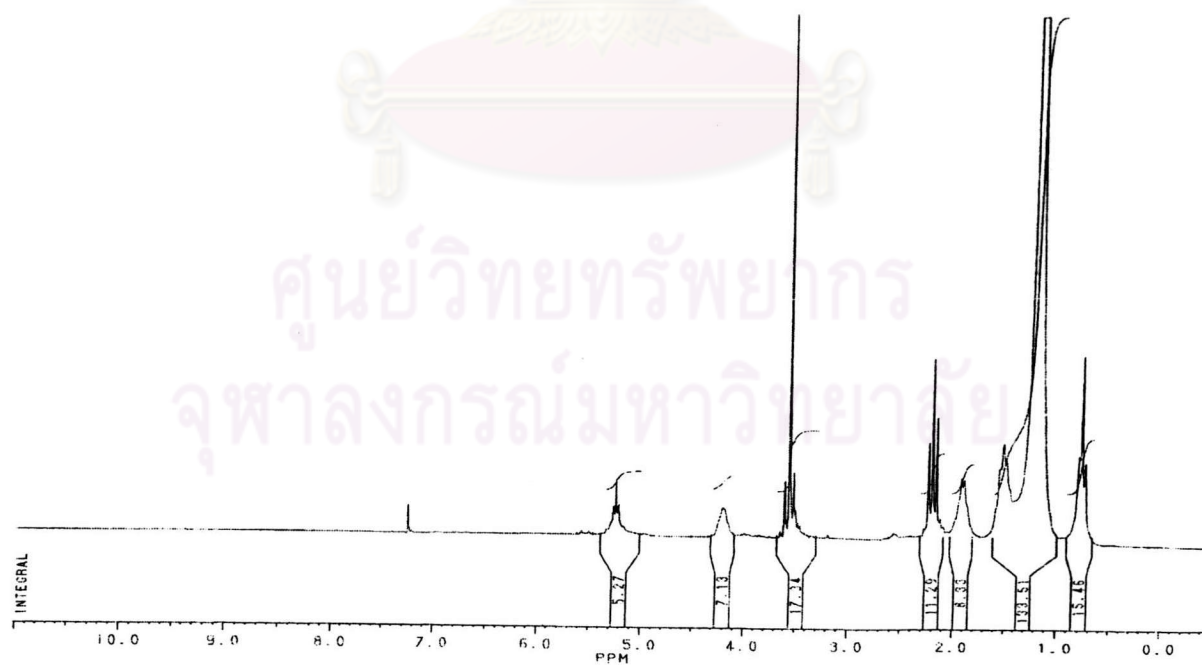


Figure B14 ¹H-NMR spectrum of dimer using cobalt naphthenate at room temperature (CDCl₃)

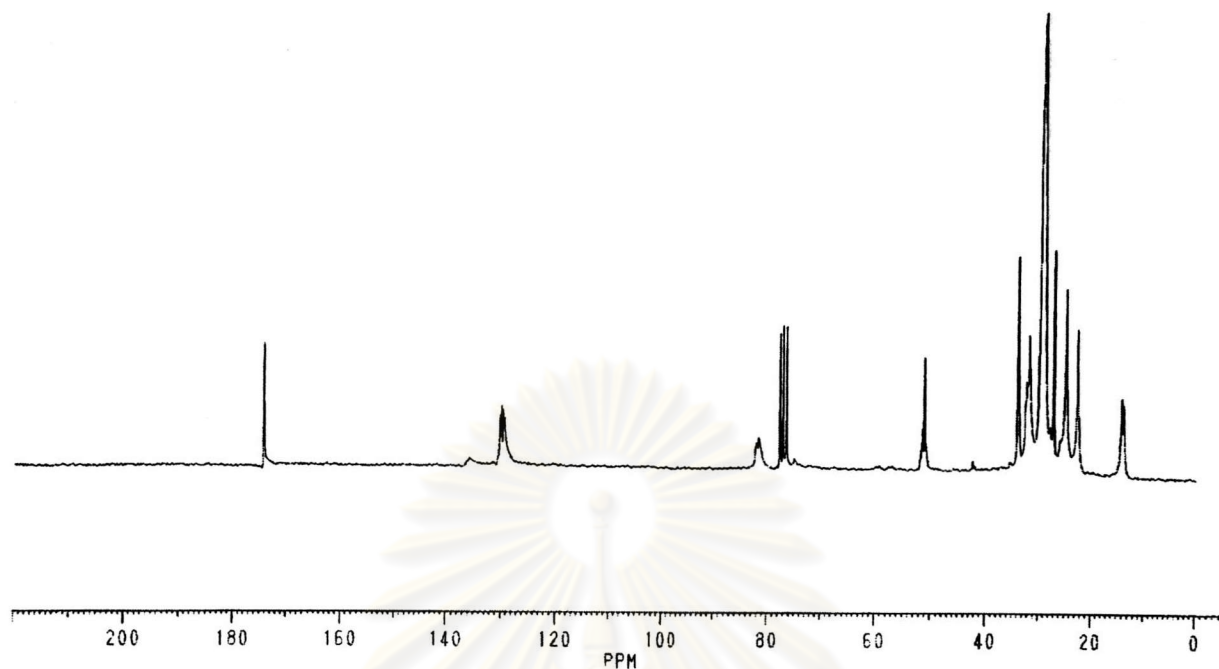


Figure B15 ^{13}C -NMR spectrum of dimer using cobalt naphthenate at room temperature (CDCl_3)

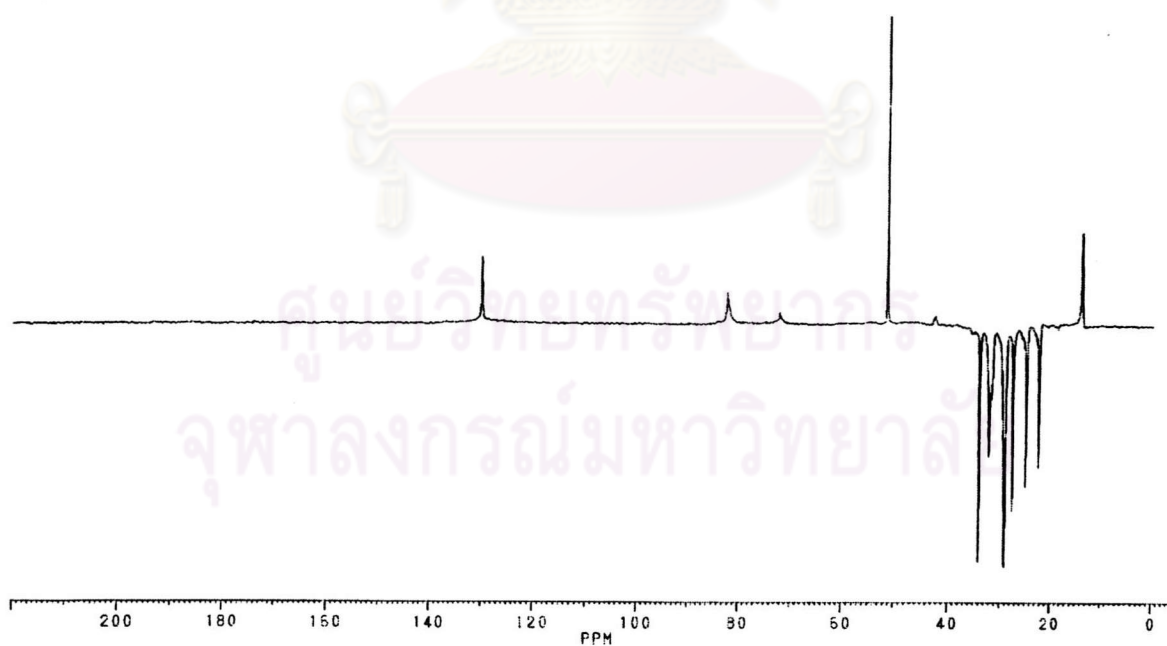


Figure B16 DEPT 135 spectrum of dimer using cobalt naphthenate at room temperature (CDCl_3)

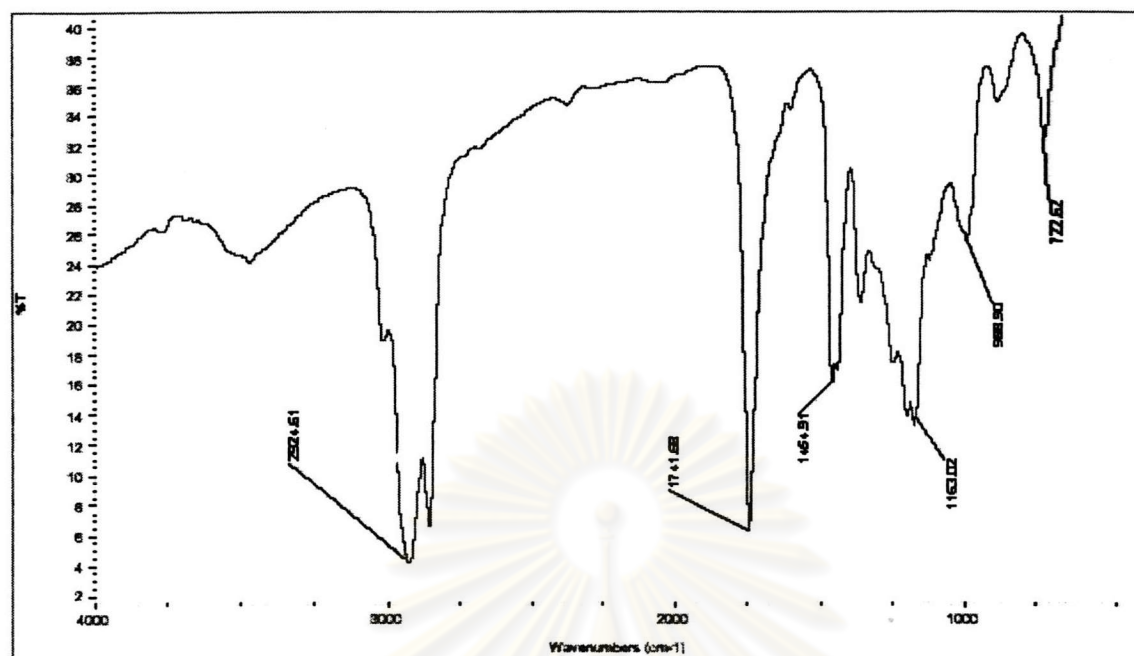


Figure B17 FTIR spectrum of dimer using cobalt naphthenate at 60 °C (NaCl)

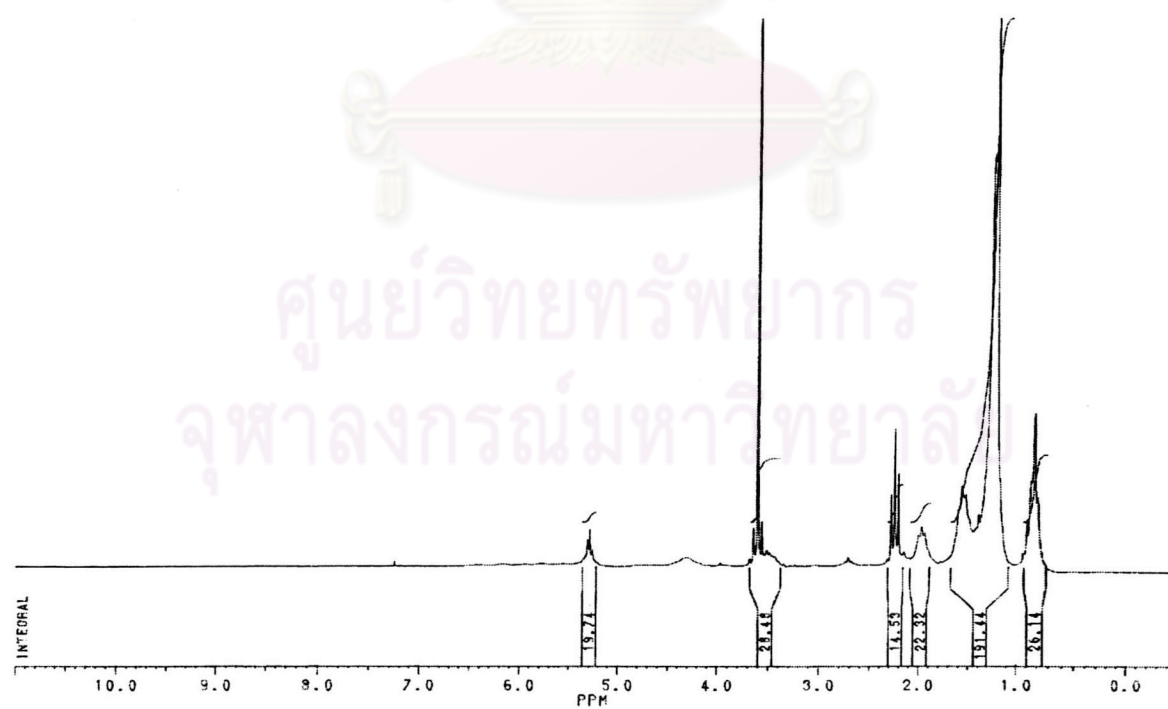


Figure B18 $^1\text{H-NMR}$ spectrum of dimer using cobalt naphthenate at 60 °C (CDCl_3)

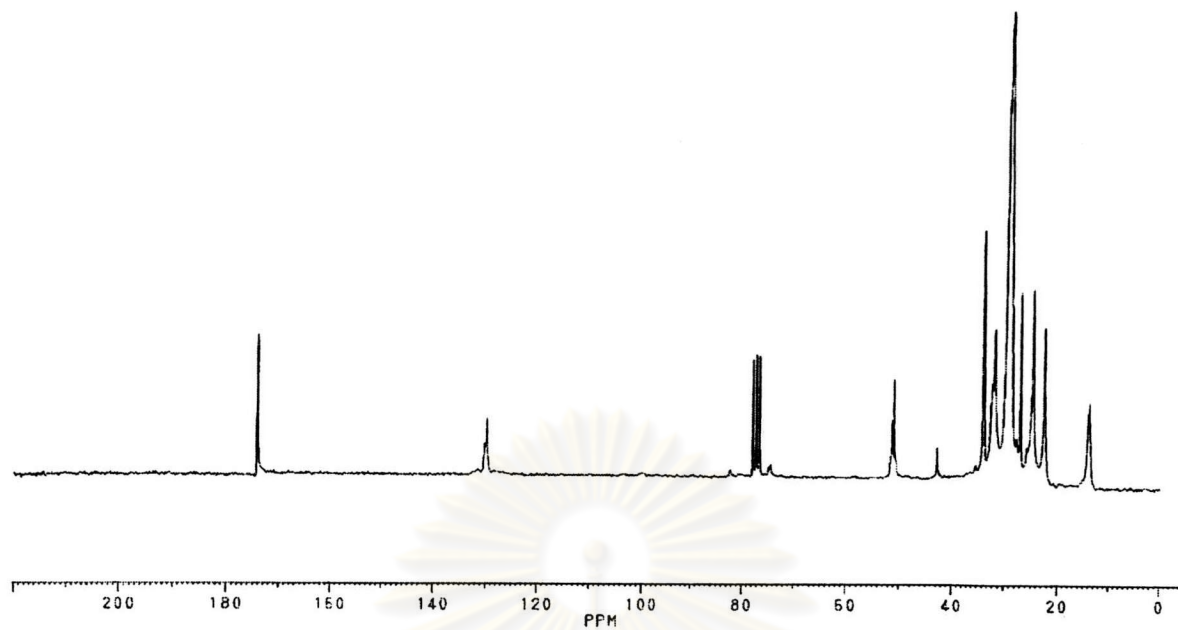


Figure B19 ^{13}C -NMR spectrum of dimer using cobalt naphthenate at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

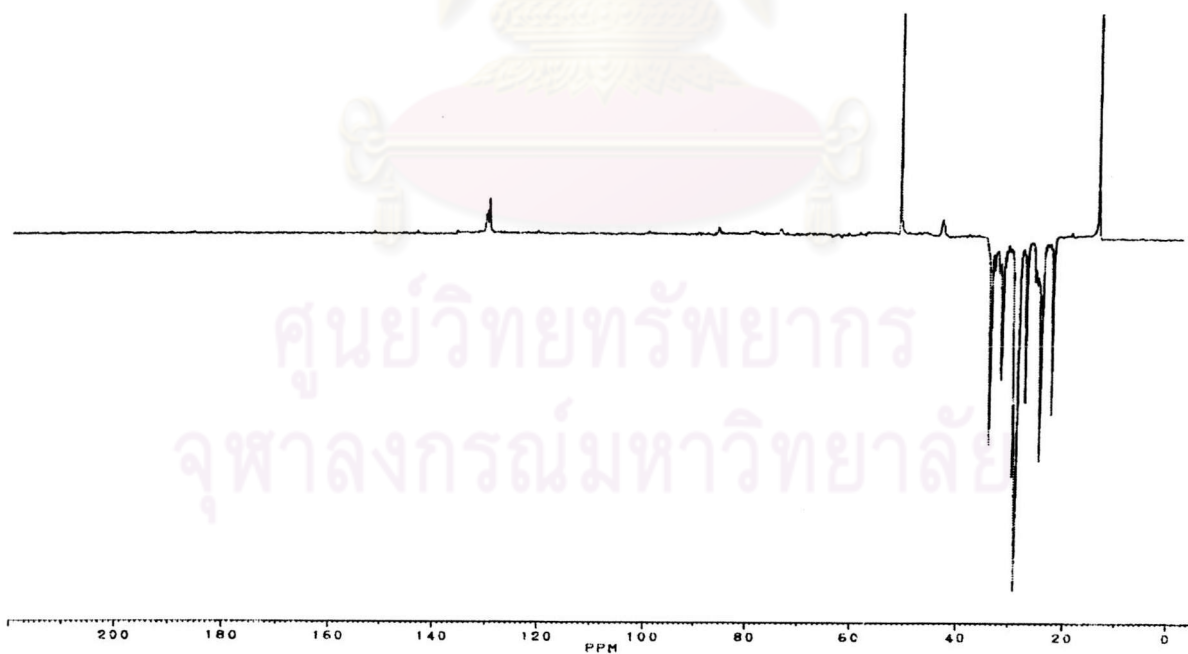


Figure B20 DEPT 135 spectrum of dimer using cobalt naphthenate at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

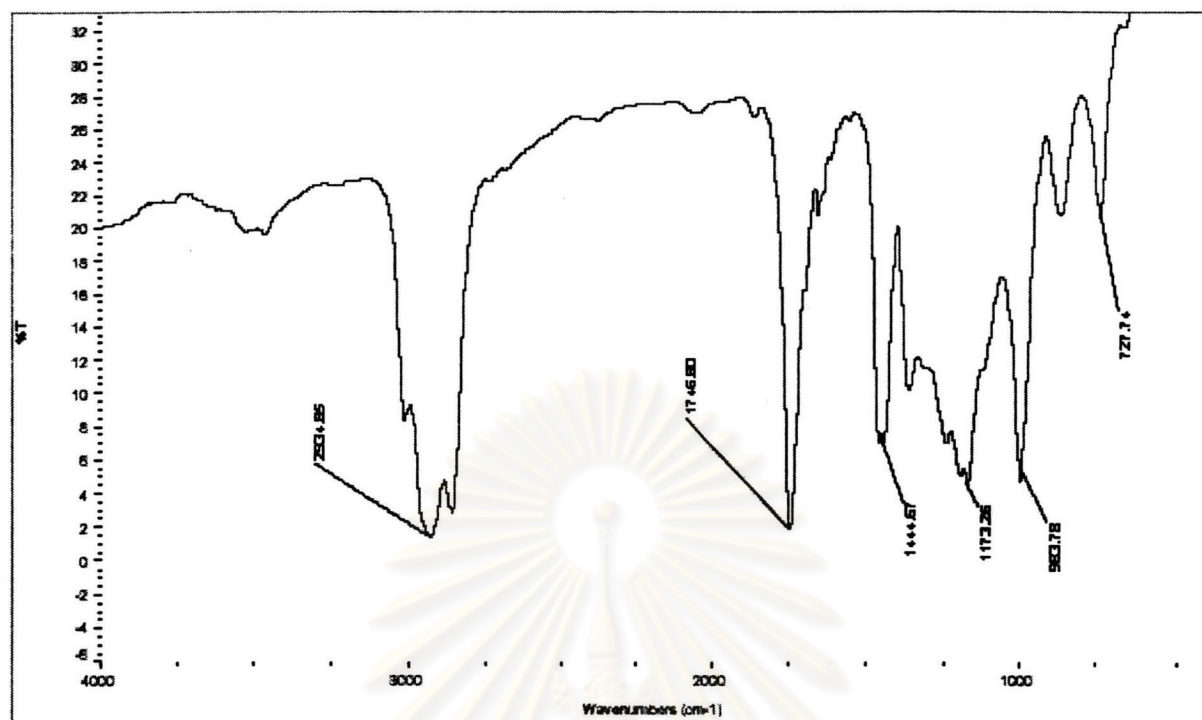


Figure B21 FTIR spectrum of dimer using TBHP and cobalt naphthenate at 60 °C (NaCl)

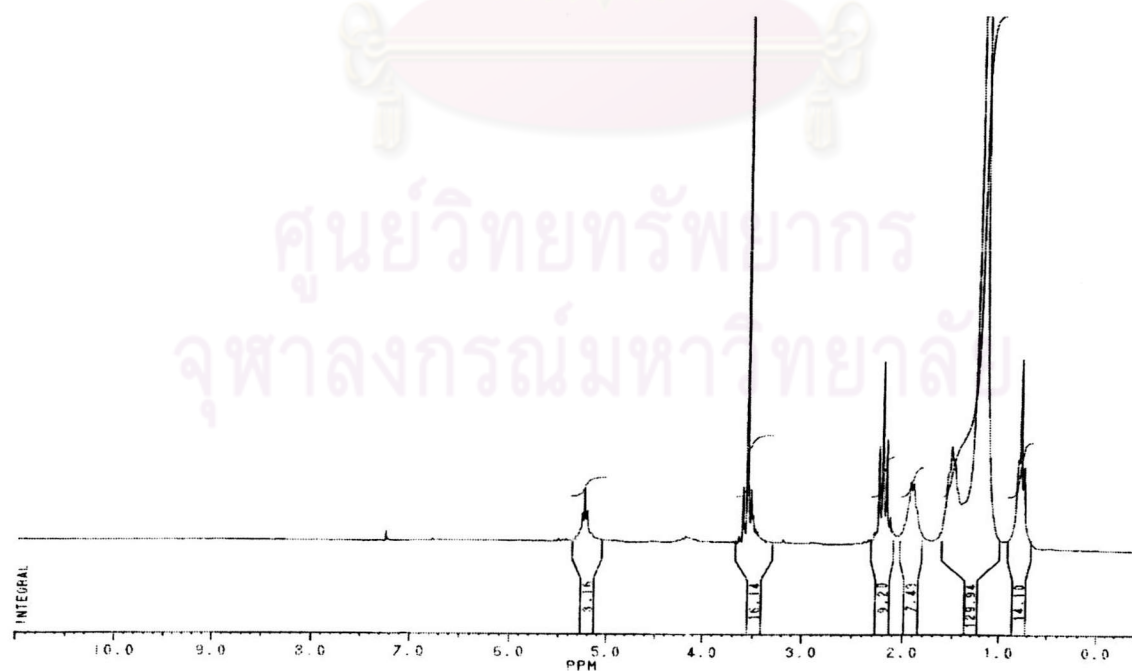


Figure B22 ¹H-NMR spectrum of dimer using TBHP and cobalt naphthenate at 60 °C (CDCl₃)

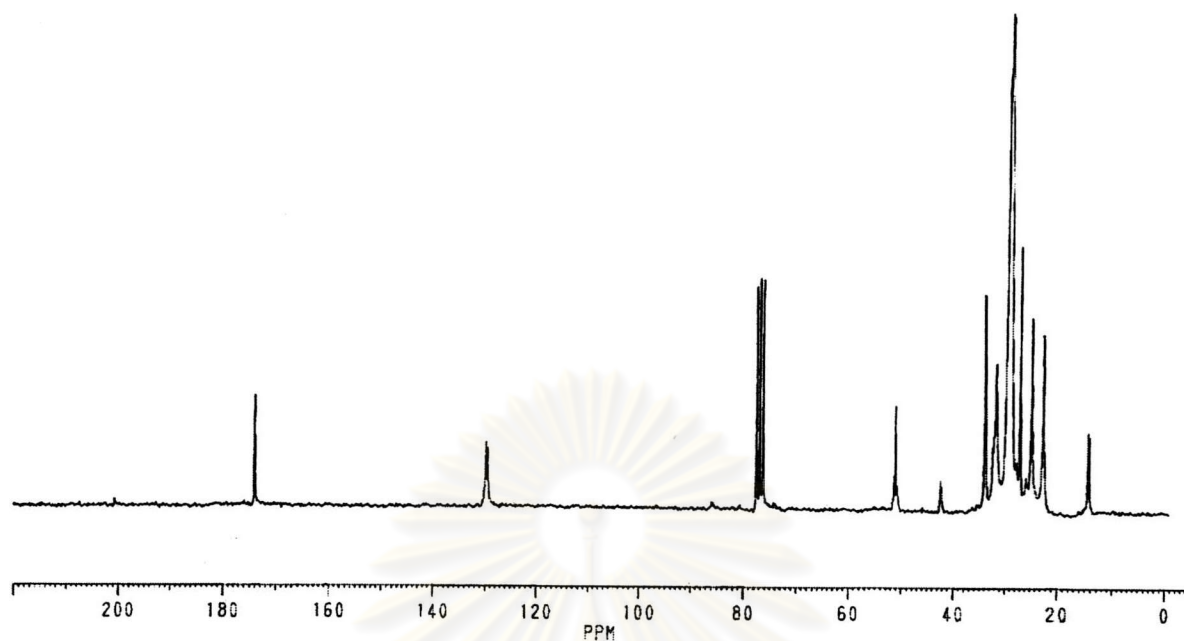


Figure B23 ^{13}C -NMR spectrum of dimer using TBHP and cobalt naphthenate at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

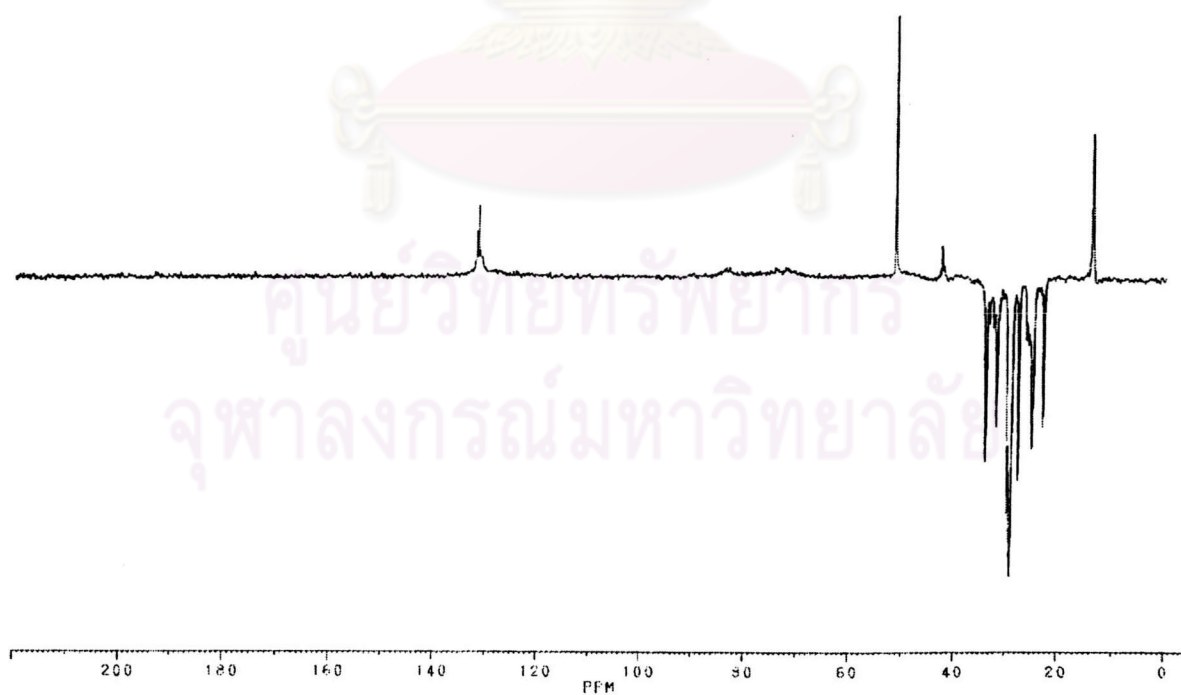


Figure B24 DEPT 135 spectrum of dimer using TBHP and cobalt naphthenate at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

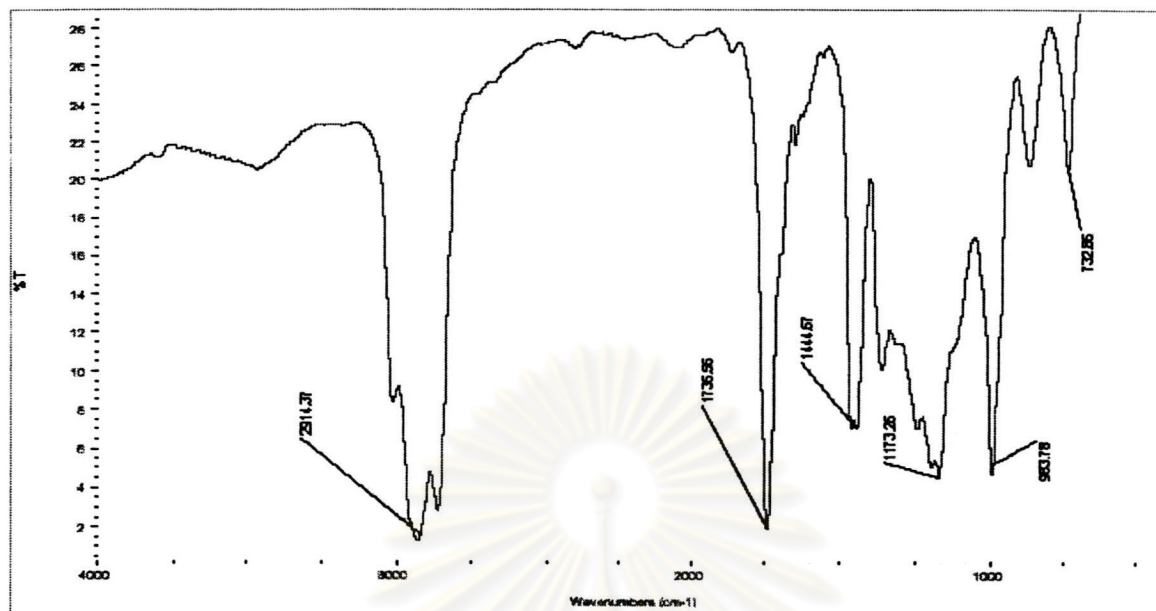


Figure B25 FTIR spectrum of dimer using TBHP, cobalt naphthenate and clay at 60 °C (NaCl)

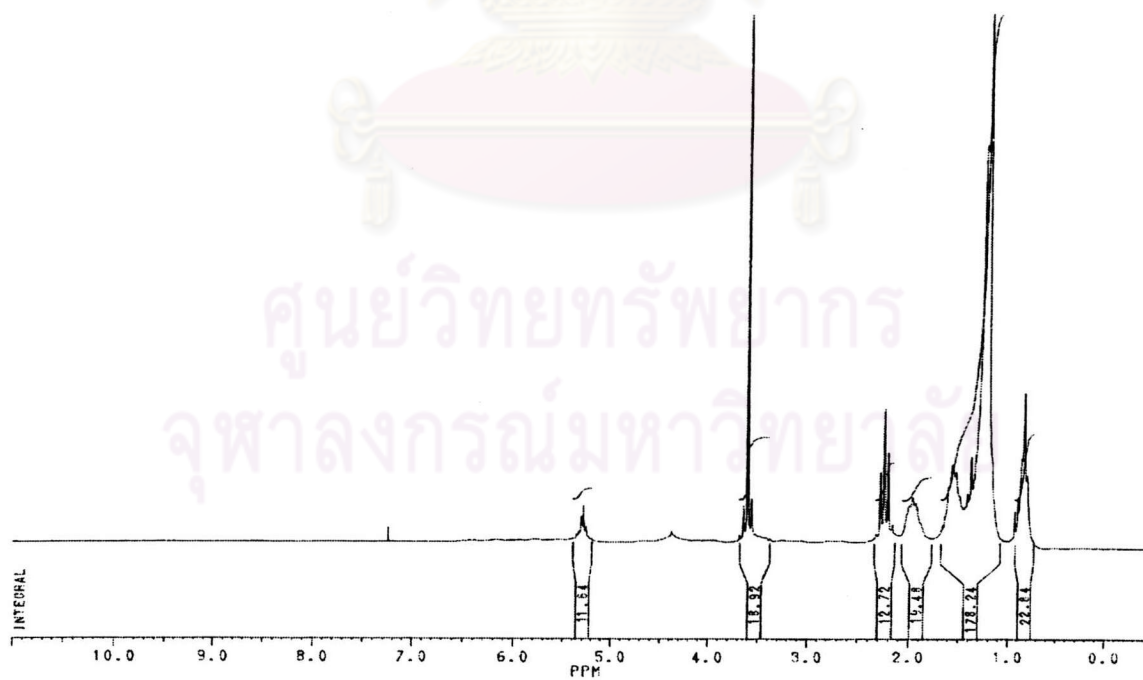


Figure B26 ¹H-NMR spectrum of dimer using TBHP, cobalt naphthenate and clay at 60 °C (CDCl₃)

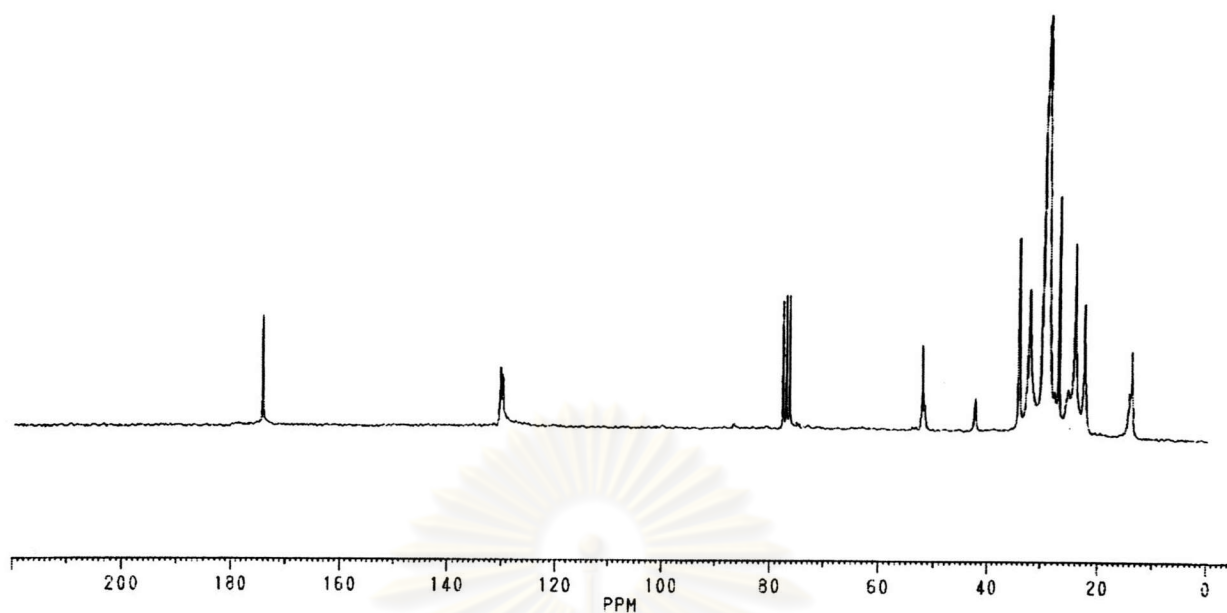


Figure B27 ^{13}C -NMR spectrum of dimer using TBHP, cobalt naphthenate and clay at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

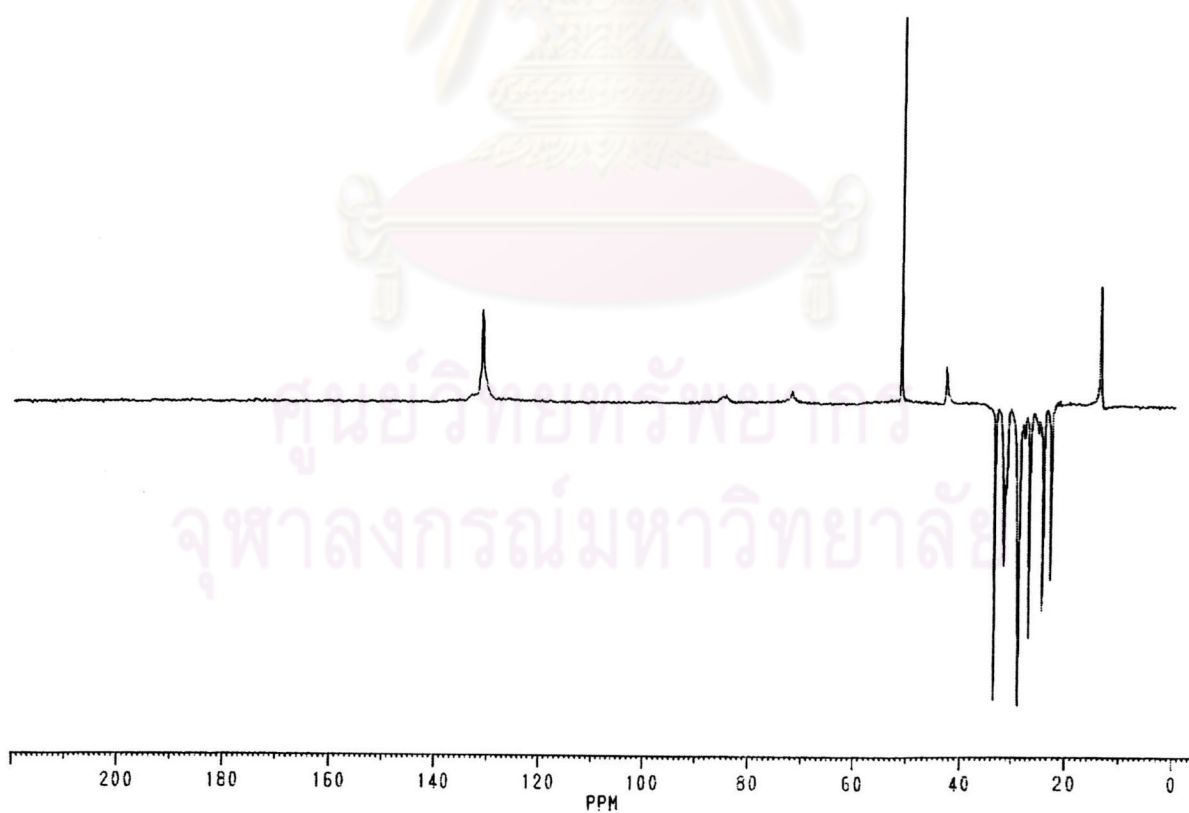


Figure B28 DEPT 135 spectrum of dimer using TBHP, cobalt naphthenate and clay at $60\text{ }^{\circ}\text{C}$ (CDCl_3)

APPENDIX C

JAPANESE INDUSTRIAL STANDARD

JIS K 0070-1966

1. Scope

This standard specifies testing methods for acid value, ester value, iodine value, and hydroxyl value of chemical products.

2. Method of test

Iodine value : the iodine value is defined by the amount of halogen (calculated to the number of grams of iodine) absorbed by 100 g of the sample under the prescribed condition.

3. Reagents

a) Wijs' solution : Weight 7.9 g of iodine trichloride and 8.7 g of iodine into separate flasks. Dissolve them in acetic acid, mix well and dilute with acetic acid into 1000 ml.

This solution should be stored in a brown bottle, in a dark place. When it might freeze at winter, it should be heated to a temperature not higher than 40 °C prior to use.

- b) Potassium iodine solution (10%w/v) : Dissolve 100 g of potassium iodine in 1000 ml of water.
- c) N/10 Sodium thiosulfate solution : Dissolve 24.6 g of sodium thiosulfate in water and dilute with water to 100 ml. This solution should be standardized as follows.
- d) Starch solution : weight 1 g of solution starch with a small amount of water and pour slowly with constant stirring, into 200 ml of boiling water. Allow cooling to room temperature and supernatant liquid or the filtrate should be for test. Pulverize potassium dichromate specified in JIS K 8005 and heat at 100 to 110°C for 3-4 hours. Dissolve 4.9035 g (on the basis of 100%) of this reagent in ester. Transfer the solution to a 1000 ml volumetric flask and diluted with water to mark.
- e) N/10 Potassium dichromate solution (primary standard substance) : Pulverize potassium dichromate specified in JIS K 8005 and heat at 100 to 110°C for 3-4 hours. Dissolve 4.9035 g (on the basis of 100%) of this reagent in ester. Transfer the solution to a 1000 ml volumetric flask and diluted with water to mark.

4. Standardization

Take 10 ml of potassium iodine solution (10% w/v) into the glass-stopper Erlenmeyer flask and 5 ml of hydrochloric acid and shake well. And exactly 25 ml of N/10 potassium dichromate solution (primary standard substance), tightly stopper

with a glass stopper wet with potassium iodine solution (10% w/v) and gently shake the flask. Add 100 ml of water shake and titrate with N/10 sodium thiosulfate solution until the yellow color almost disappears.

Add 1 ml of starch solution and continue the titration until the blue color of iodine –starch changes to green. Run the blank titration and calculate the factor of N/10 sodium thiosulfate solution, f, by the follow formula :

$$f = \frac{25}{A - B}$$

Where : A = Volume of N/10 sodium thiosulfate solution consumed in actual titration (cm³).

B = Volume of N/10 sodium thiosulfate solution consumed in blank titration (cm³).

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