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APPENDICES

APPENDIX A

JAPANESE INDUSTRIAL STANDARD

JIS K 0070 – 1996

Iodine value

Dedinition:

The iodine value is a measure of the unsaturation of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample (% iodine absorbed).

Reagents:

a) Wijs'solution

Wijs'solution is more stable if iodine is continued in slightly excess and it often gives high result of iodine value if chlorine is contained in excess. This solution shall be stored in a brown bottle, in a dark place. When it migh freeze at winter, it shall be heated to a temperature not higher than 40 ^{o}C prior to use. This solution can prepared alternatively by Method A or Method B as described below.

Method A:

Dissolve 13 g of iodine in 1000 cm^3 of acetic acid. Pipette 20 cm^3 of the solution and titrate with N/10 sodium thiosulfate solution to determine the concentration of iodine. After dried chloride is introduced in the solution, pipette 20 cm^3 of the solution, add 15 cm^3 of potassium iodine solution (10 % w/v) and 100 cm^3

of water, and titrate with N/10 sodium thiosulfate solution so that the titre measures twice the initia titre. The titration shall be made after chlorine gas is introduced in the solution until the color of liberated iodine disappears, reversing small portions of the solution aside before introducing chlorine. If chlorine is contained excessively, it shall be removed by adding appropriate amount of iodine solution reversed.

Method B:

Weigh 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 to 1000 cm^3.

b) Potassium iodide solution (10 % w/v)

Dissolve 100 g of potassium iodine in 1000 cm^3 of water.

c) N/10 Sodium thiosulfate solution

Dissolve 24.8 g of sodium thiosulfate in water and dilute with water to 1000 cm^3. This solution shall be standardized as follows.

Standardization:

Take 10 cm^3 of potassium iodide solution (10 % w/v) into a glass-stoppered Erlenmeyer flask and add 5 cm^3 of hydrochloric acid and shake well. And exactly 25 cm^3 of N/10 potassium dichromate solution (primary standard substance), tightly stopper with a glass stopper wet with potassium iodide solution (10 % w/v) and gently shake the flask. Add 100 cm^3 of water, shake and titrate with N/10 sodium thiosulfate solution until the yellow color disappears. Add 1 cm^3 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 following formular:

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

A : volume of N/10 sodium thiosulfate solution consumed in actual titration (cm^3)

B : volume of N/10 sodium thiosulfate solution consumed in blank titration (cm^3)

d) Starch solution

Titrate 1 g of soluble starch with small amount of water and pour slowly with constant stirring, into 200 cm^3 of boiling water. Allow cooling to room temperature and the supernatant liquid or the filtrate shall be used for test.

e) N/10 potassium dichromate solution (primary standard substance)

Pulverize potassium dichromate specified in JIS K 8005 and heat at 100 to 110 ^{o}C for 3 to 4 hours. Dissolve 4.9035 g (on the basis of 100 %) of this reagent in water. Transfer the solution to a 1000 cm^3 volumetric flask and dilute with water to the mark.

Procedure: (see section 5.3 in Chapter III)

In this method the sample weighed accurately proper amount shall be taken as directed below in such amount that not more than one half of Wijs'solution is consumed.

Expected iodine value	Weight of sample to be taken (g)
Less than 5	2.00 (to 2 significant digits)
5 to 30 excl.	1.00 (to 3 significant digits)
30 to 50 excl.	0.60 (to 3 significant digits)
50 to 100 excl.	0.30 (to 3 significant digits)
100 to 150 excl.	0.20 (to 3 significant digits)
150 to 200 excl.	0.15 (to 4 significant digits)
200 and over	0.10 (to 4 significant digits)

Calculation of iodine value: (see section 5.3 in Chapter III)

APPENDIX B

Analyte	Concentration Unit
NiO	11.86 Wt %

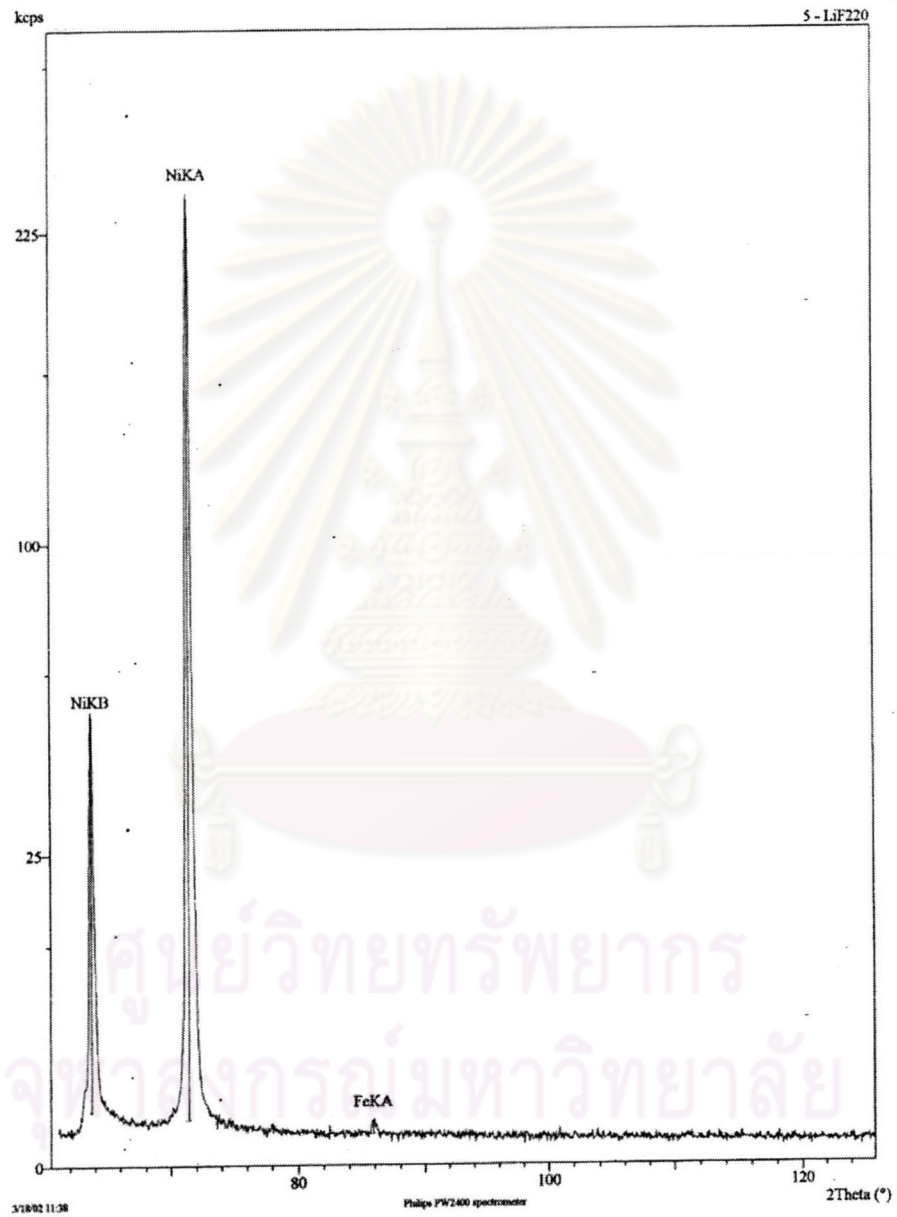


Figure B1 A plot of X-ray fluorescence data of 10% prepared catalyst.

Analyte	Concentration Unit
NiO	16.53 Wt %

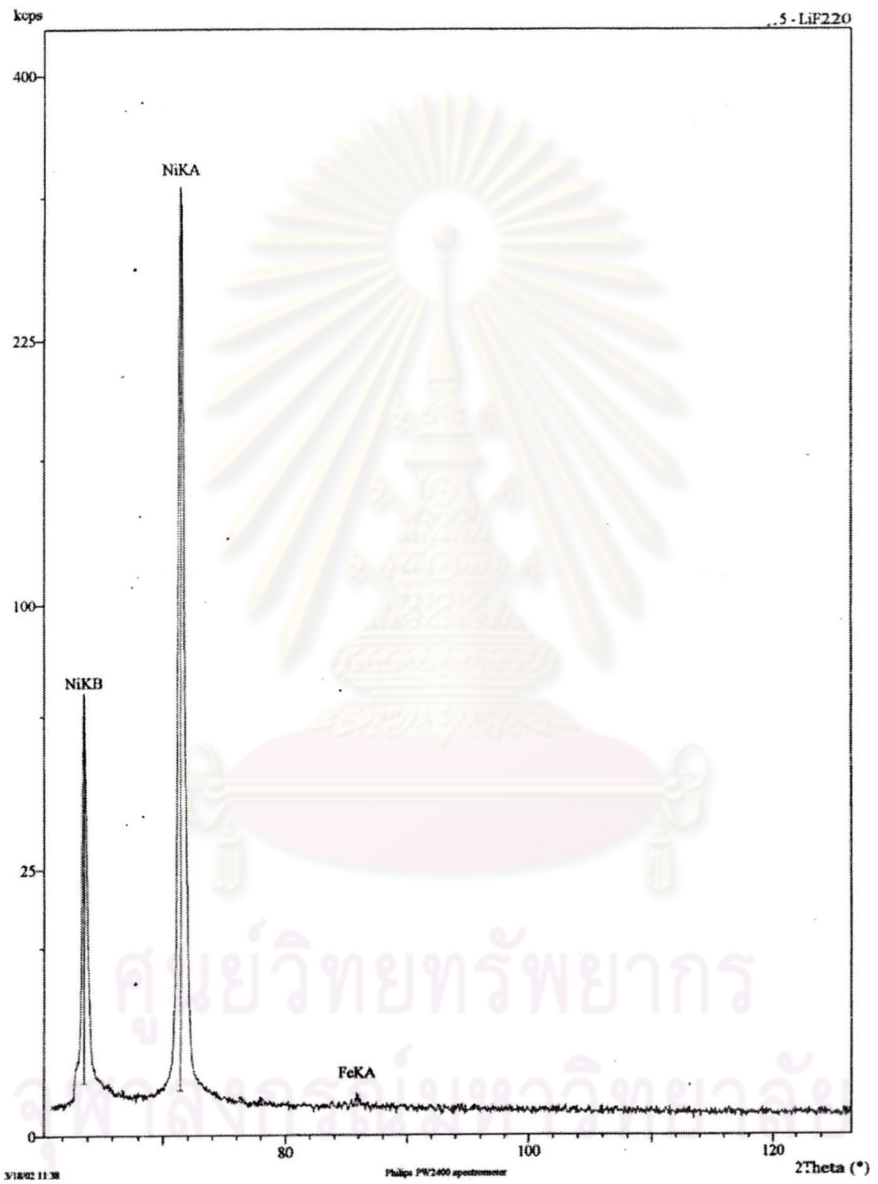


Figure B2 A plot of X-ray fluorescence data of 15% prepared catalyst.

Analyte	Concentration Unit
NiO	21.82 Wt %

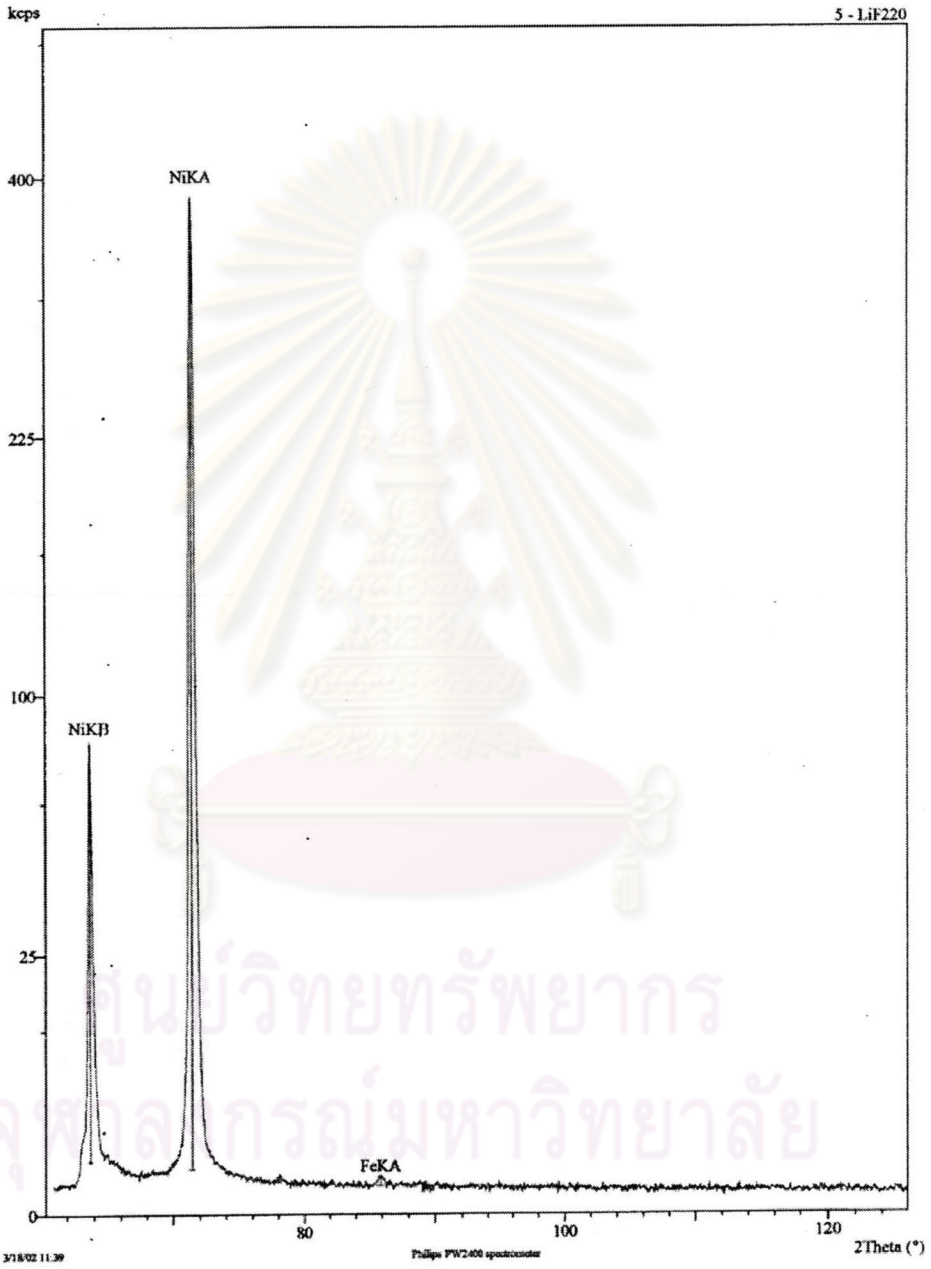
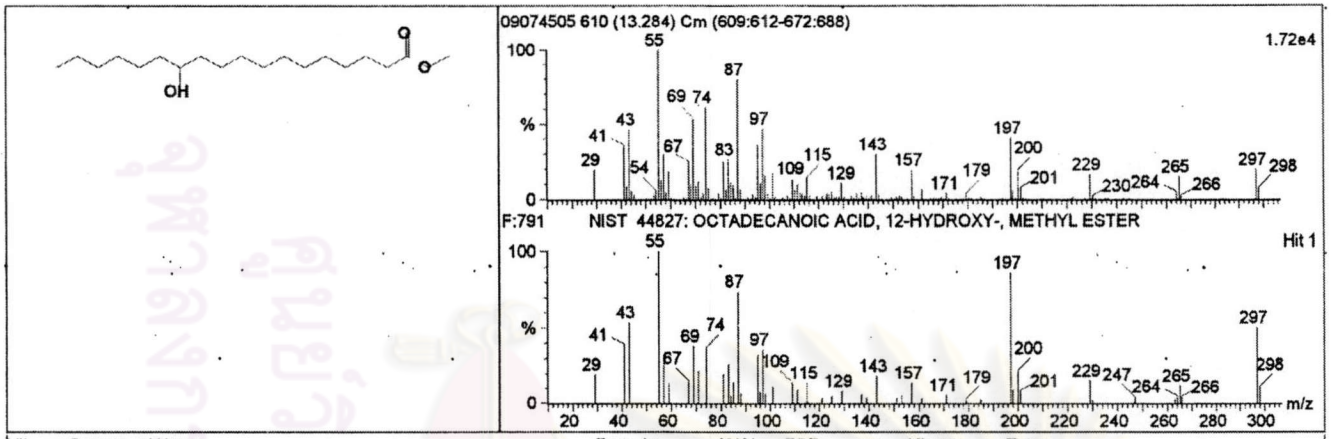
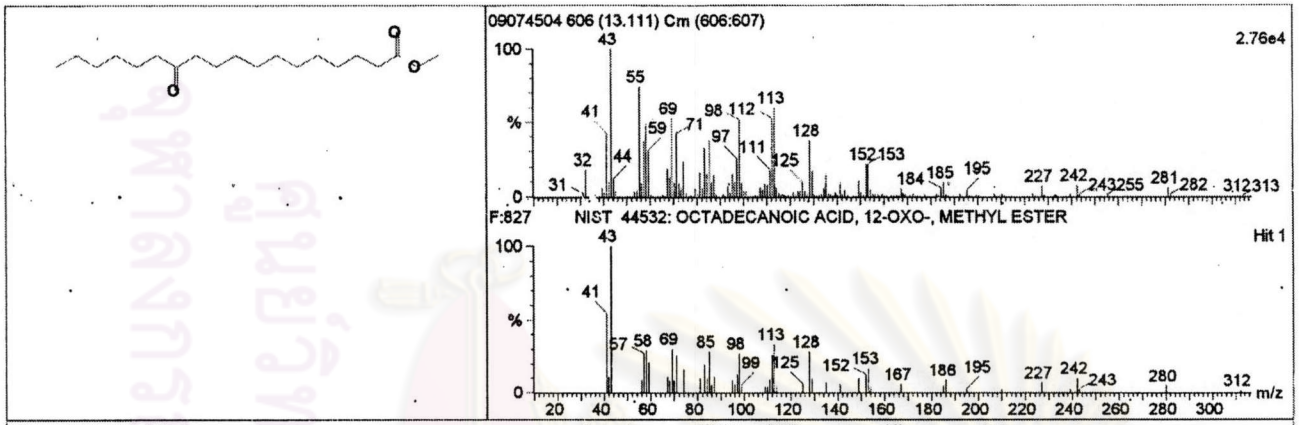


Figure B3 A plot of X-ray fluorescence data of 20% prepared catalyst.



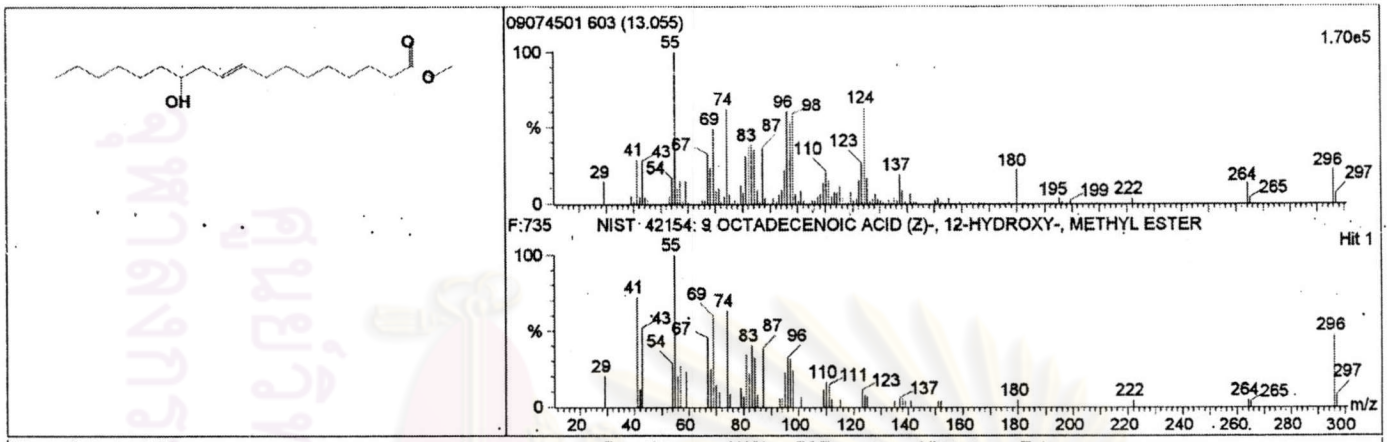
Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	OCTADECANOIC ACID, 12-HYDROXY-, METHYL ESTER	C19H38O3	314	911	955	NIST	44827
2	CYCLOPENTANETRIDECANOIC ACID, METHYL ESTER	C19H36O2	296	823	904	NIST	42140
3	HEXADECANOIC ACID, 14-METHYL-, METHYL ESTER	C18H36O2	284	655	763	NIST	40192
4	DODECANOIC ACID, 10-METHYL-, METHYL ESTER	C14H28O2	228	655	732	NIST	29647
5	TRIACONTANOIC ACID, METHYL ESTER	C31H62O2	466	651	778	NIST	57791
6	HEPTADECANOIC ACID, 16-METHYL-, METHYL ESTER	C19H38O2	298	640	751	NIST	42494
7	PENTACOSANOIC ACID, METHYL ESTER	C26H52O2	396	607	741	NIST	53581
8	EICOSANOIC ACID, METHYL ESTER	C21H42O2	326	597	744	NIST	46374
9	OCTADECANOIC ACID, 8-METHYL-, METHYL ESTER	C20H40O2	312	579	720	NIST	44564
10	UNDECANOIC ACID, METHYL ESTER	C12H24O2	200	574	668	NIST	22934
11	TRIDECANOIC ACID, METHYL ESTER	C14H28O2	228	569	664	NIST	29642
12	DECANOIC ACID, 9-OXO-, METHYL ESTER	C11H20O3	200	532	654	NIST	22901
13	METHYL 9-ACETOXYHENEICOSANOATE	C24H46O4	398	512	661	NIST	53712
14	NONANEDIOIC ACID, DIMETHYL ESTER	C11H20O4	216	487	613	NIST	26670
15	TETRADECANOIC ACID, 12-METHYL-, METHYL ESTER, (S)-	C16H32O2	256	446	837	NIST	35200
16	OCTADECANOIC ACID, 11-METHYL-, METHYL ESTER	C20H40O2	312	432	845	NIST	44550
17	HEXADECANOIC ACID, 9-METHYL-, METHYL ESTER	C18H36O2	284	432	869	NIST	40187
18	HEXADECANOIC ACID, 10-HYDROXY-, METHYL ESTER	C17H34O3	286	430	567	NIST	40530
19	HEPTACOSANOIC ACID, 25-METHYL-, METHYL ESTER	C29H58O2	438	417	834	NIST	56435
20	HEPTACOSANOIC ACID, 26-METHYL-, METHYL ESTER	C29H58O2	438	415	849	NIST	56436

Figure C6 Mass spectrum of Methyl 12-hydroxystearate



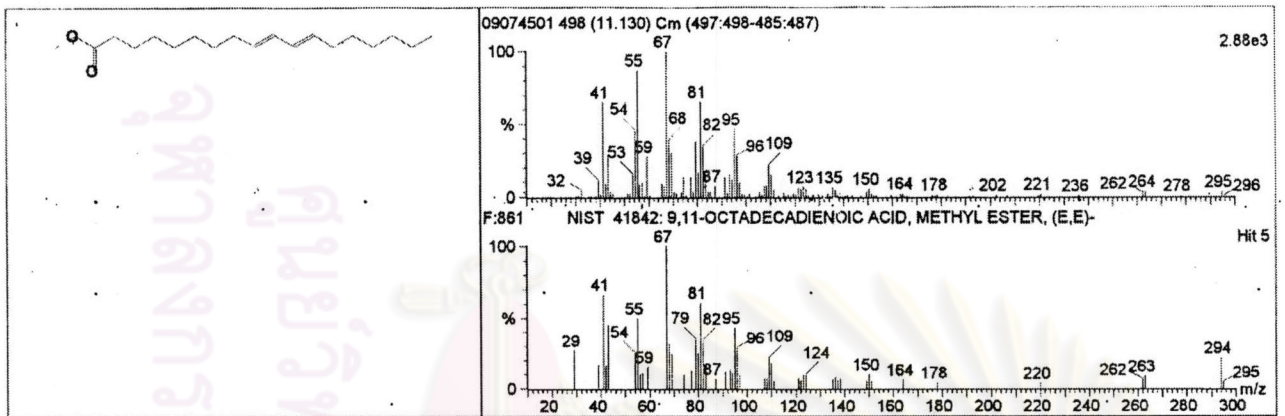
Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	OCTADECANOIC ACID, 12-OXO-, METHYL ESTER	C19H36O3	312	827	925	NIST	44532
2	OCTADECANE, 1-ISOCYANATO-	C19H37ON	295	520	688	NIST	41947
3	2-METHYL-1-ETHYLPYRROLIDINE	C7H15N	113	471	600	NIST	2796
4	UNDECANE, 2,6-DIMETHYL-	C13H28	184	428	621	NIST	19058
5	DODECANE, 1-ISOCYANATO-	C13H25ON	211	419	550	NIST	25612
6	HEXANOIC ACID, 5-(ACETYLOXY)-, METHYL ESTER	C9H16O4	188	383	764	NIST	19775
7	2-PYRROLIDINONE, 1-(3,7,11-TRIMETHYLDODECYL)-	C19H37ON	295	376	559	NIST	41949
8	1-PROPENE, 2-METHYL-, TETRAMER	C16H32	224	366	483	NIST	28774
9	2-PENTENOIC ACID, 4-OXO-, METHYL ESTER, (E)-	C6H8O3	128	332	771	NIST	4875
10	2-PYRROLIDINONE, 1-OCTADECYL-	C22H43ON	337	304	547	NIST	47815
11	3-HEXEN-2-ONE, 5-METHYL-	C7H12O	112	299	544	NIST	2621
12	1,3-CYCLOPENTANEDIONE, 2-ISOPENTYL-	C10H16O2	168	280	604	NIST	14580
13	1,3-CYCLOPENTANEDIONE, 4-METHYL-	C6H8O2	112	216	358	NIST	2504

Figure C5 Mass spectrum of Methyl 12-ketostearate



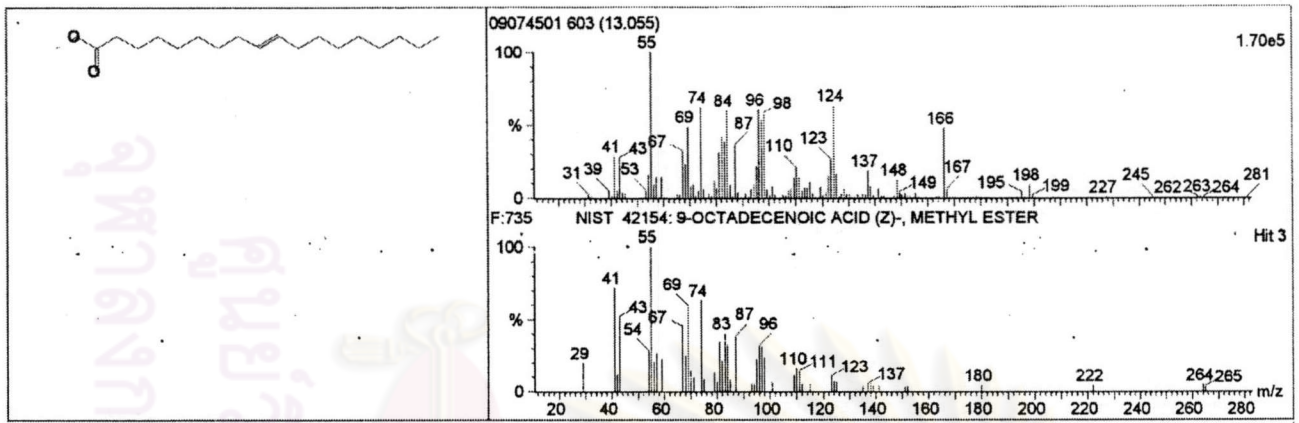
Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	9-OCTADECENOIC ACID (Z)-, 12-HYDROXY-, METHYL ESTER	C19H36O3	312	899	952	NIST	23327
2	7-HEXADECENOIC ACID, METHYL ESTER, (Z)-	C17H32O2	268	753	842	NIST	37409
3	9-OCTADECENOIC ACID (Z)-, METHYL ESTER	C19H36O2	296	735	833	NIST	42154
4	10-UNDECENOIC ACID, METHYL ESTER	C12H22O2	198	728	769	NIST	22425
5	7-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	715	785	NIST	42155
6	8-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	699	776	NIST	42131
7	11-HEXADECENOIC ACID, METHYL ESTER	C17H32O2	268	678	764	NIST	37407
8	14-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	672	750	NIST	42153
9	15-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	670	748	NIST	42130
10	16-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	668	746	NIST	42159
11	4H-CYCLOPENTACYCLOOCTEN-4-ONE, DECAHYDRO-	C11H18O	166	655	808	NIST	14123
12	11-EICOSENOIC ACID, METHYL ESTER	C21H40O2	324	653	771	NIST	46146
13	DECANEDIOIC ACID, DIMETHYL ESTER	C12H22O4	230	552	653	NIST	29910
14	2H-PYRIDO 1,2-F 1,6 DIAZACYCLOOCTADECIN-11(6H)-ONE, 10	C23H45ON	379	487	723	NIST	52144
15	PIPERIDINE, 1-CYCLOHEXYL-	C11H21N	167	484	614	NIST	14317
16	7,14-METHANO-2H,6H-DIPYRIDO 1,2-A:1',2'-E 1,5 DIAZOCIN-15-	C15H24ON	248	431	577	NIST	33528
17	2H-QUINOLIZIN-1-OL, 1-ETHENYLOCTAHYDRO-	C11H19ON	181	414	711	NIST	18097
18	2H-QUINOLIZIN-1-OL, OCTAHYDRO-, TRANS-	C9H17ON	155	402	611	NIST	11206
19	2H-QUINOLIZIN-1-OL, OCTAHYDRO-, CIS-	C9H17ON	155	389	579	NIST	11204
20	2H-QUINOLIZIN-1-OL, OCTAHYDRO-	C9H17ON	155	384	583	NIST	11215

Figure C4 Mass spectrum of Methyl ricinoleate



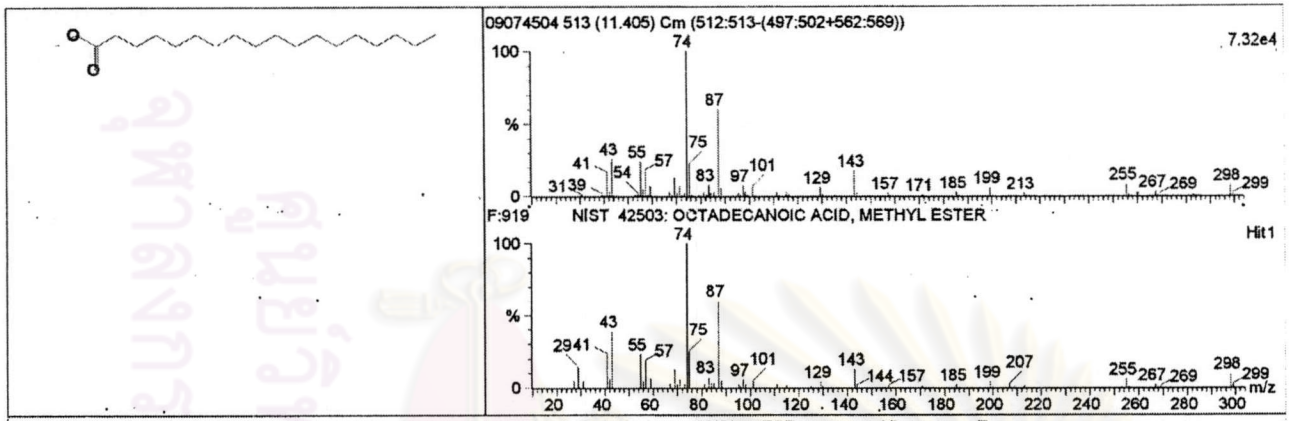
Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	11,14-EICOSADIENOIC ACID, METHYL ESTER	C21H38O2	322	946	970	NIST	45900
2	7,10-HEXADECADIENOIC ACID, METHYL ESTER	C17H30O2	266	907	943	NIST	37015
3	9,12-HEXADECADIENOIC ACID, METHYL ESTER	C17H30O2	266	886	925	NIST	37018
4	CYCLOPROPANEOCTANOIC ACID, 2- 2- (2-ETHYLCYCLOPRO	C22H38O2	334	880	915	NIST	47540
5	9,11-OCTADECADIENOIC ACID, METHYL ESTER, (E,E)-	C19H34O2	294	861	901	NIST	41842
6	9,12-OCTADECADIENOIC ACID, METHYL ESTER, (E,E)-	C19H34O2	294	857	875	NIST	41833
7	9,12-OCTADECADIENOYL CHLORIDE, (Z,Z)-	C18H31OCl	298	849	896	NIST	42460
8	(Z)6,(Z)9-PENTADECADIEN-1-OL	C15H28O	224	846	924	NIST	28747
9	9,17-OCTADECADIENAL, (Z)-	C18H32O	264	841	924	NIST	36671
10	1,E-11,Z-13-OCTADECATRIENE	C18H32	248	840	894	NIST	33611
11	8-OCTADECYNOIC ACID, METHYL ESTER	C19H34O2	294	833	871	NIST	41844
12	1,E-11,Z-13-HEPTADECATRIENE	C17H30	234	828	910	NIST	30779
13	6,9-OCTADECADIENOIC ACID, METHYL ESTER	C19H34O2	294	818	883	NIST	41826
14	9-OCTADECYNOIC ACID, METHYL ESTER	C19H34O2	294	817	847	NIST	41834
15	1,E-8,Z-10-HEXADECATRIENE	C16H28	220	810	893	NIST	27740
16	1,E-11,Z-13-HEXADECATRIENE	C16H28	220	808	891	NIST	27737
17	9,12-OCTADECADIEN-1-OL	C18H34O	266	806	891	NIST	37038
18	7,10-OCTADECADIENOIC ACID, METHYL ESTER	C19H34O2	294	803	872	NIST	41827
19	1,E-8,Z-10-PENTADECATRIENE	C15H26	206	803	885	NIST	24459
20	1,E-11,Z-13-PENTADECATRIENE	C15H26	206	777	845	NIST	24463

Figure C3 Mass spectrum of Methyl linoleate



Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	10-UNDECENOYL CHLORIDE	C11H19OCl	202	799	852	NIST	23327
2	7-HEXADECENOIC ACID, METHYL ESTER, (Z)-	C17H32O2	268	753	842	NIST	37409
3	9-OCTADECENOIC ACID (Z)-, METHYL ESTER	C19H36O2	296	735	833	NIST	42154
4	10-UNDECENOIC ACID, METHYL ESTER	C12H22O2	198	728	769	NIST	22425
5	7-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	715	785	NIST	42155
6	8-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	699	776	NIST	42131
7	11-HEXADECENOIC ACID, METHYL ESTER	C17H32O2	268	678	764	NIST	37407
8	14-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	672	750	NIST	42153
9	15-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	670	748	NIST	42130
10	16-OCTADECENOIC ACID, METHYL ESTER	C19H36O2	296	668	746	NIST	42159
11	4H-CYCLOPENTACYCLOOCTEN-4-ONE, DECAHYDRO-	C11H18O	166	655	808	NIST	14123
12	11-EICOSENOIC ACID, METHYL ESTER	C21H40O2	324	653	771	NIST	46146
13	DECANEDIOIC ACID, DIMETHYL ESTER	C12H22O4	230	552	653	NIST	29910
14	2H-PYRIDO 1,2-F 1,6 DIAZACYCLOOCTADECIN-11(6H)-ONE, 10	C23H45ON	379	487	723	NIST	52144
15	PIPERIDINE, 1-CYCLOHEXYL-	C11H21N	167	484	614	NIST	14317
16	7,14-METHANO-2H,6H-DIPYRIDO 1,2-A:1',2'-E 1,5 DIAZOCIN-15-	C15H24ON	248	431	577	NIST	33528
17	2H-QUINOLIZIN-1-OL, 1-ETHENYLOCTAHYDRO-	C11H19ON	181	414	711	NIST	18097
18	2H-QUINOLIZIN-1-OL, OCTAHYDRO-, TRANS-	C9H17ON	155	402	611	NIST	11206
19	2H-QUINOLIZIN-1-OL, OCTAHYDRO-, CIS-	C9H17ON	155	389	579	NIST	11204
20	2H-QUINOLIZIN-1-OL, OCTAHYDRO-	C9H17ON	155	384	583	NIST	11215

Figure C2 Mass spectrum of Methyl oleate



Hit	Compound Name	Formula	M.W.	FOR	rev	Library	Entry
1	OCTADECANOIC ACID, METHYL ESTER	C19H38O2	298	935	953	NIST	42494
2	HEPTADECANOIC ACID, 16-METHYL-, METHYL ESTER	C19H38O2	298	919	932	NIST	42503
3	HEPTADECANOIC ACID, 15-METHYL-, METHYL ESTER	C19H38O2	298	895	925	NIST	42496
4	HEXADECANOIC ACID, 15-METHYL-, METHYL ESTER	C18H36O2	284	845	892	NIST	40193
5	HEPTADECANOIC ACID, 10-METHYL-, METHYL ESTER	C19H38O2	298	830	856	NIST	42495
6	TETRADECANOIC ACID, METHYL ESTER	C15H30O2	242	812	873	NIST	32399
7	HEPTADECANOIC ACID, METHYL ESTER	C18H36O2	284	808	855	NIST	40190
8	METHYL 9-METHYLTETRADECANOATE	C16H32O2	256	808	898	NIST	35185
9	HEPTADECANOIC ACID, 14-METHYL-, METHYL ESTER, (.+/-.)-	C19H38O2	298	794	835	NIST	42490
10	HEPTADECANOIC ACID, 14-METHYL-, METHYL ESTER	C19H38O2	298	793	835	NIST	42492
11	HENEICOSANOIC ACID, METHYL ESTER	C22H44O2	340	787	819	NIST	48206
12	HEPTADECANOIC ACID, 9-METHYL-, METHYL ESTER	C19H38O2	298	692	721	NIST	42486
13	OCTADECANOIC ACID, 16-METHYL-, METHYL ESTER	C20H40O2	312	675	726	NIST	44556

Figure C1 Mass spectrum of Methyl stearate

APPENDIX D

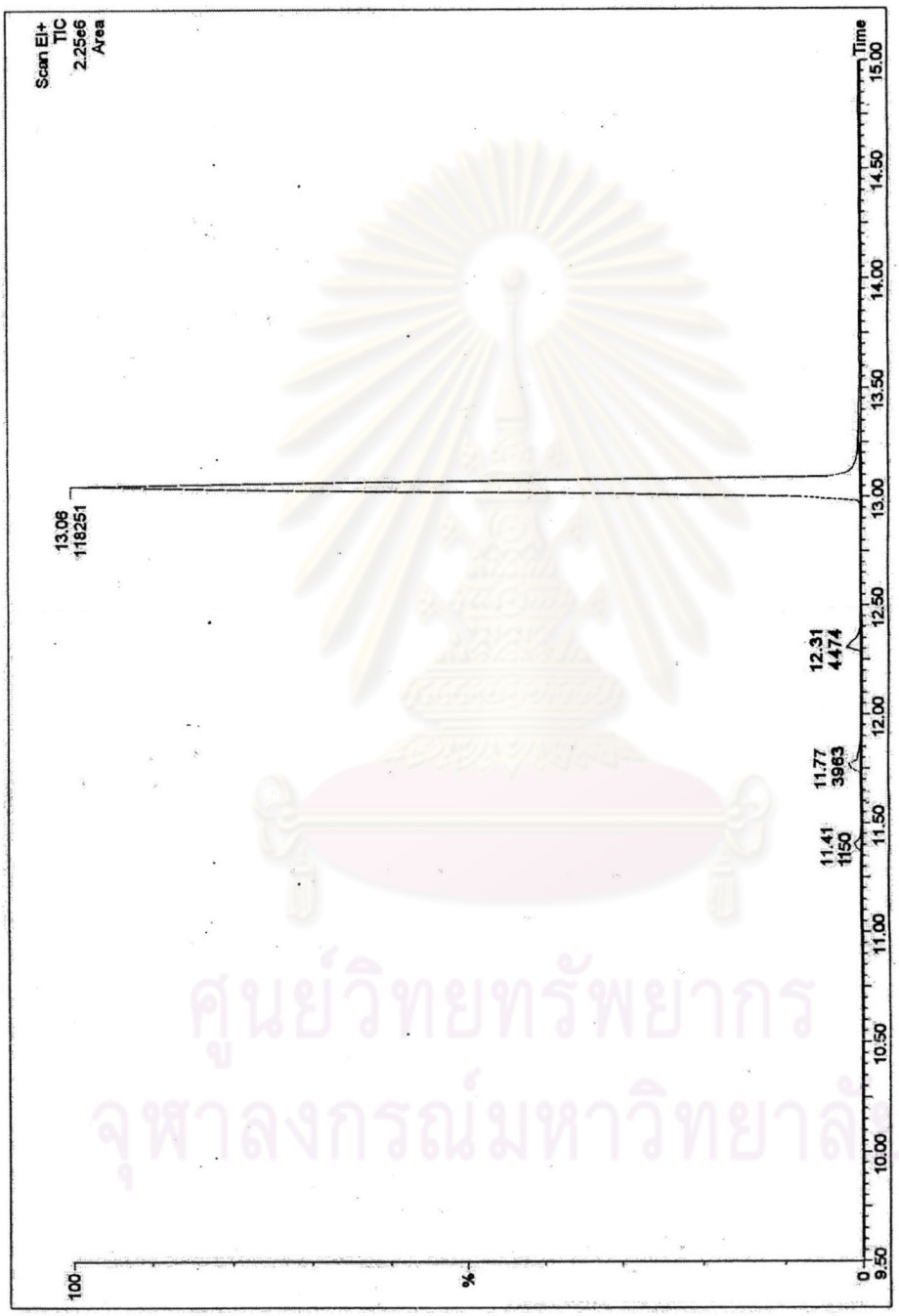


Figure D1 GC chromatogram of hydrogenated methyl ricinoleate

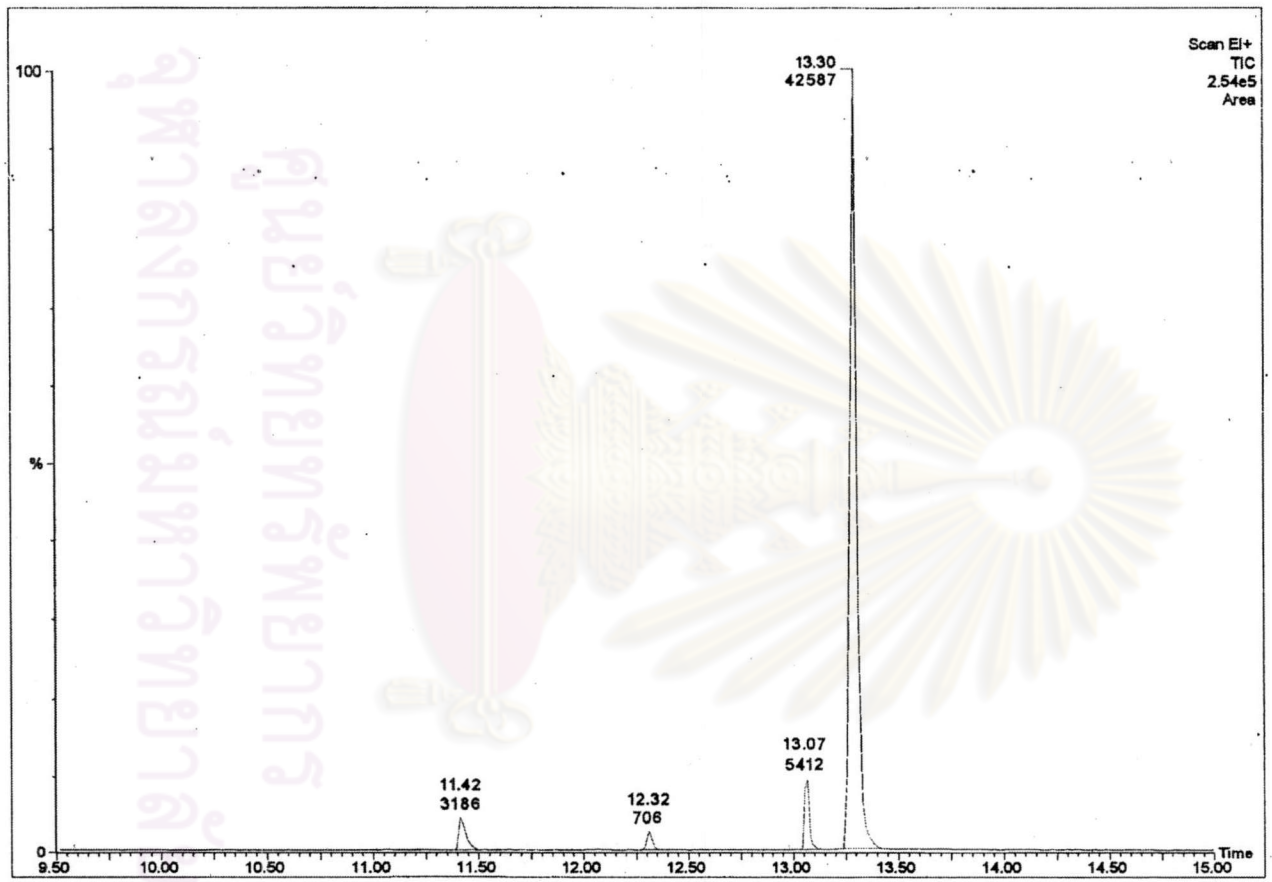


Figure D2 GC chromatogram of hydrogenated methyl ricinoleate using 10% Ni catalyst at 90 ^{o}C and 20 psig hydrogen pressure

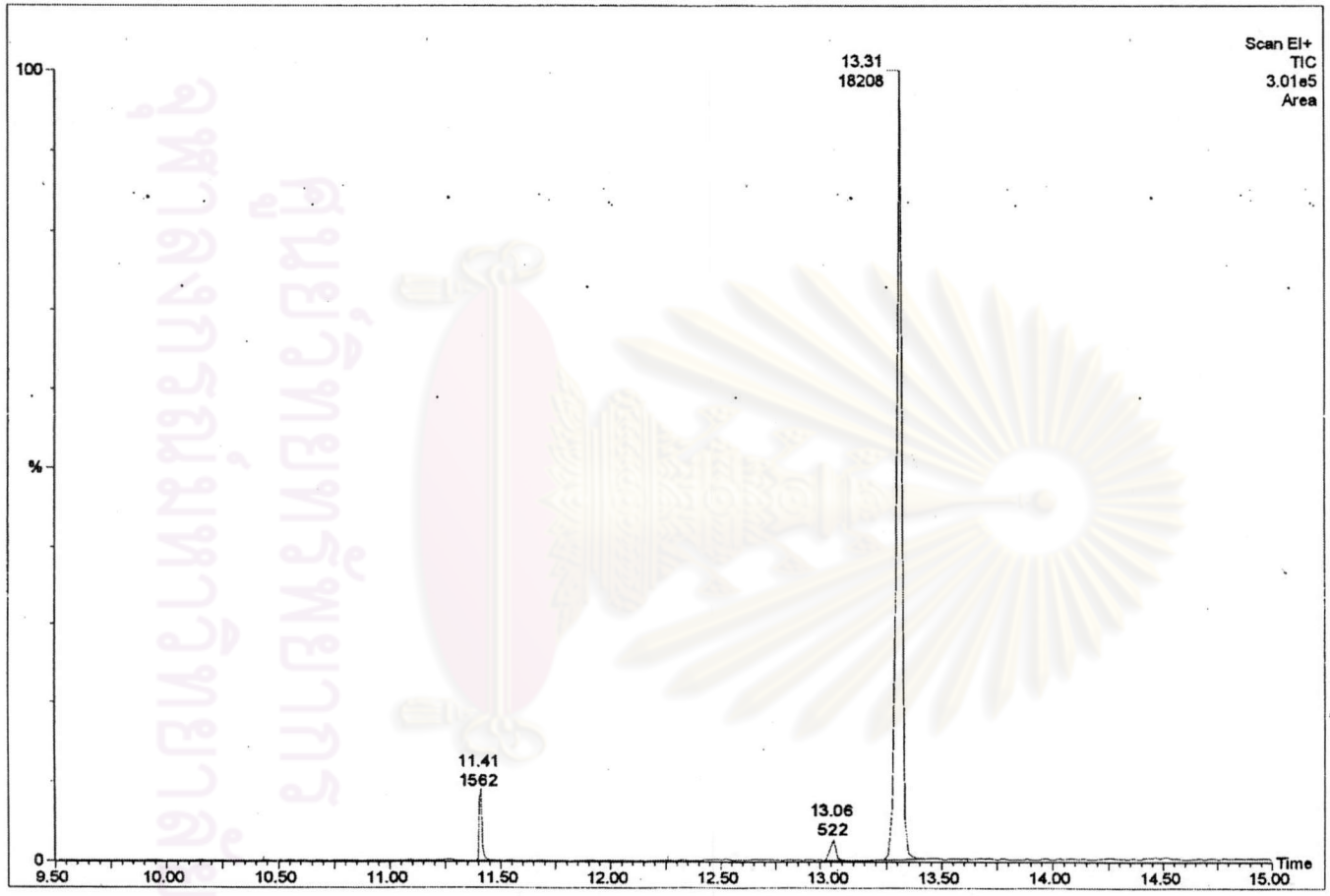


Figure D3 GC chromatogram of hydrogenated methyl ricinoleate using 10% Ni catalyst at 120 ^{o}C and 20 psig hydrogen pressure

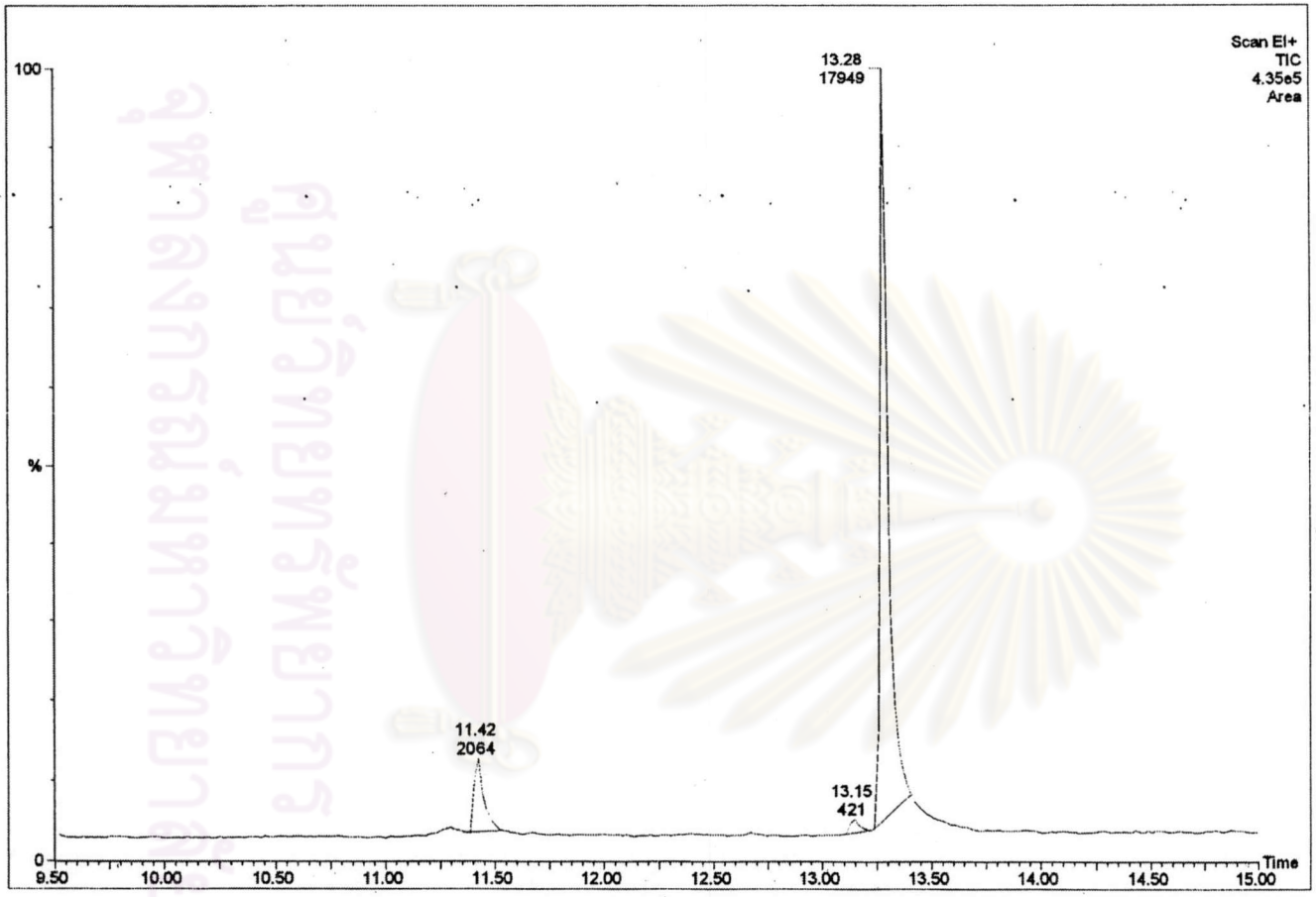


Figure D4 GC chromatogram of hydrogenated methyl ricinoleate using 10% Ni catalyst at 150 ^{o}C and 20 psig hydrogen pressure

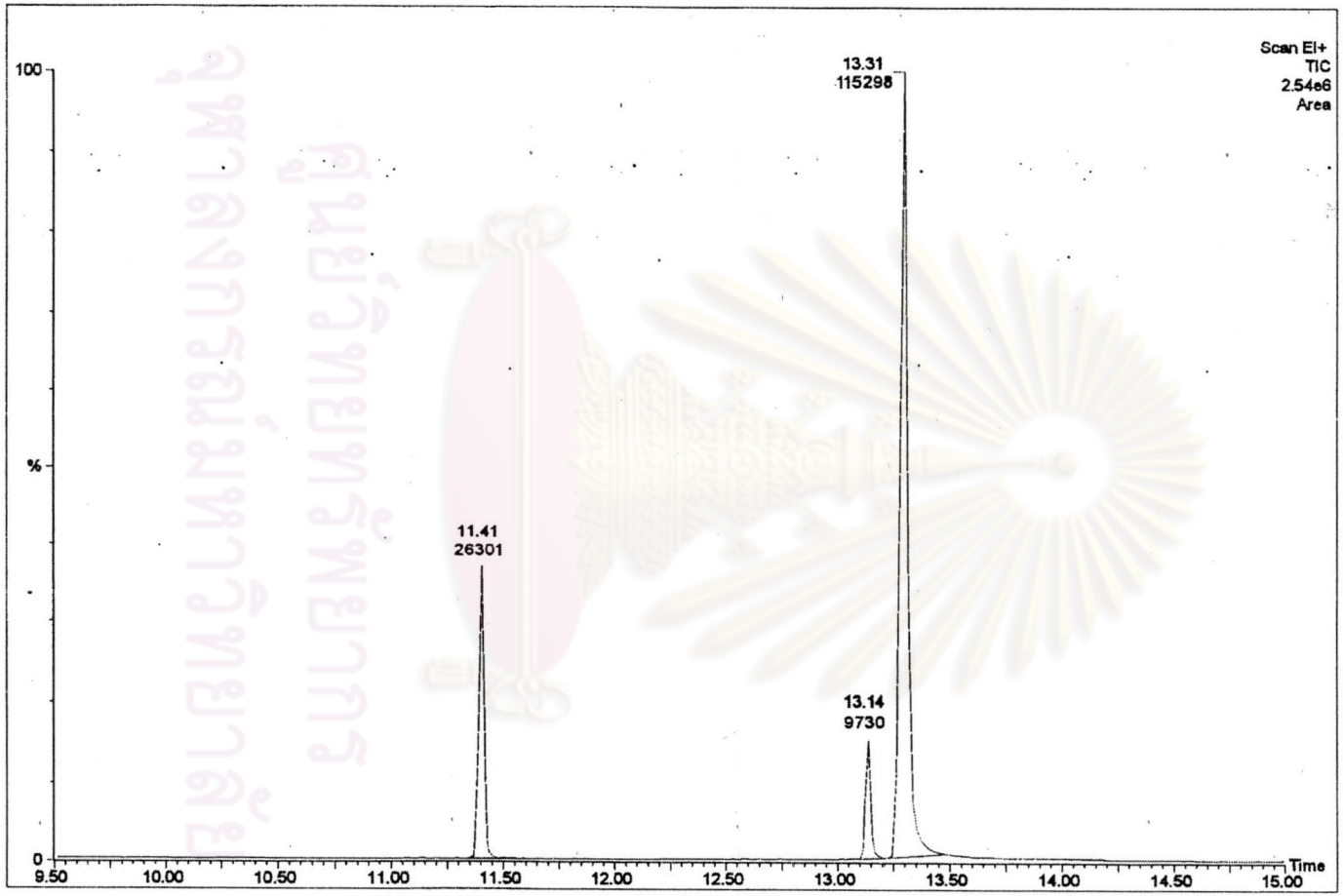


Figure D5 GC chromatogram of hydrogenated methyl ricinoleate using 10% Ni catalyst at 180 °C and 20 psig hydrogen pressure

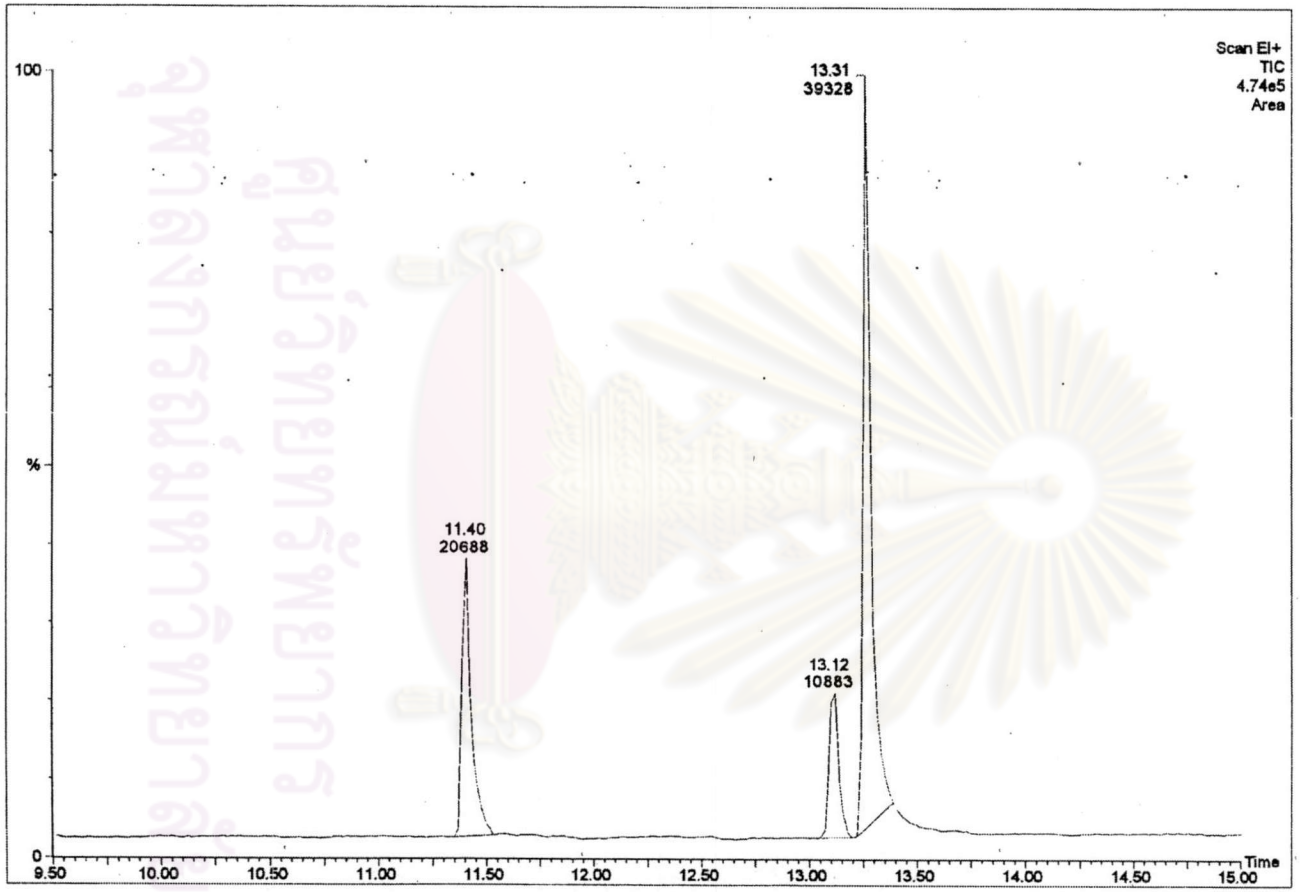


Figure D6 GC chromatogram of hydrogenated methyl ricinoleate using 10% Ni catalyst at 200 ^{o}C and 20 psig hydrogen pressure

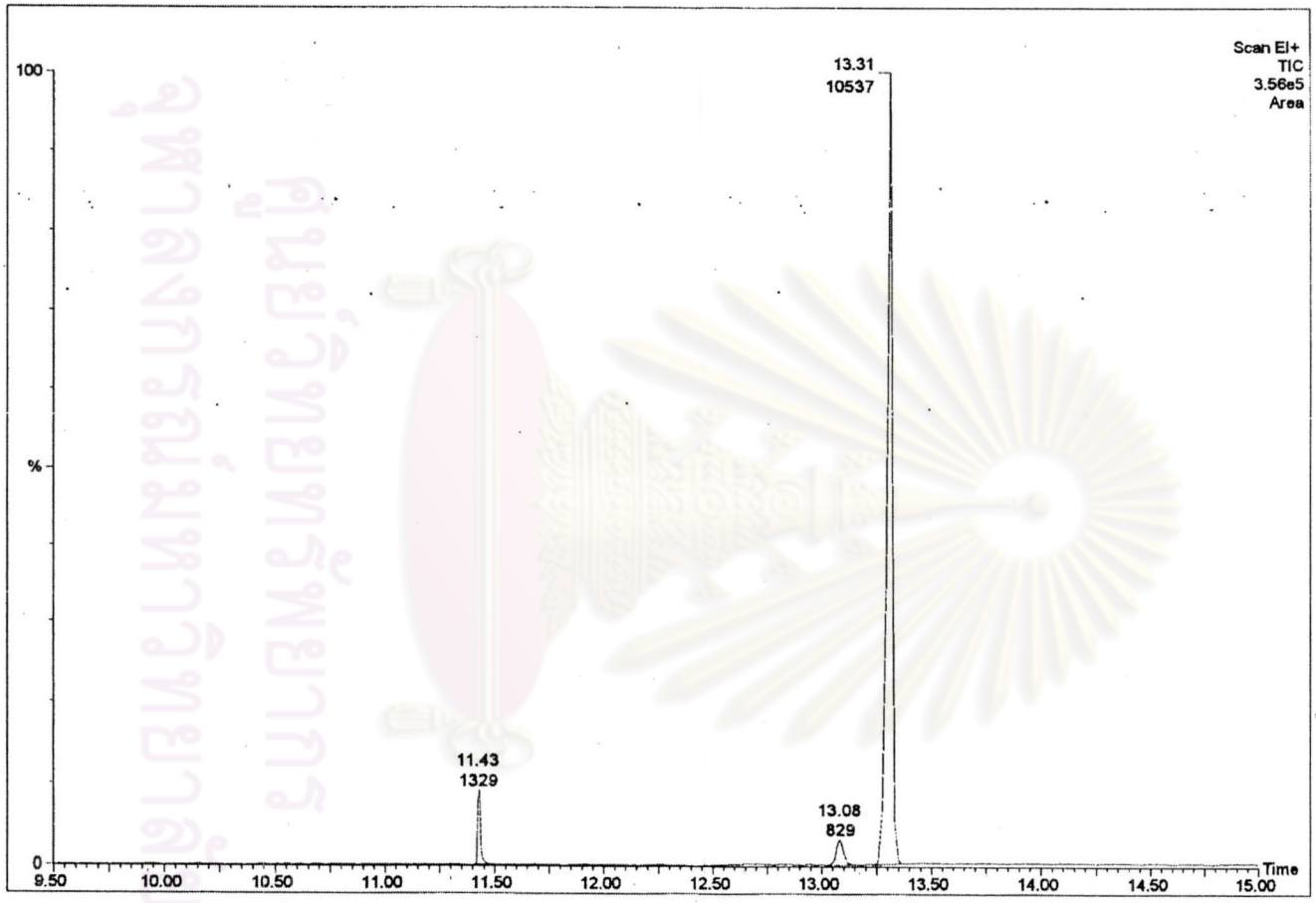


Figure D7 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 90 ^{o}C and 20 psig hydrogen pressure

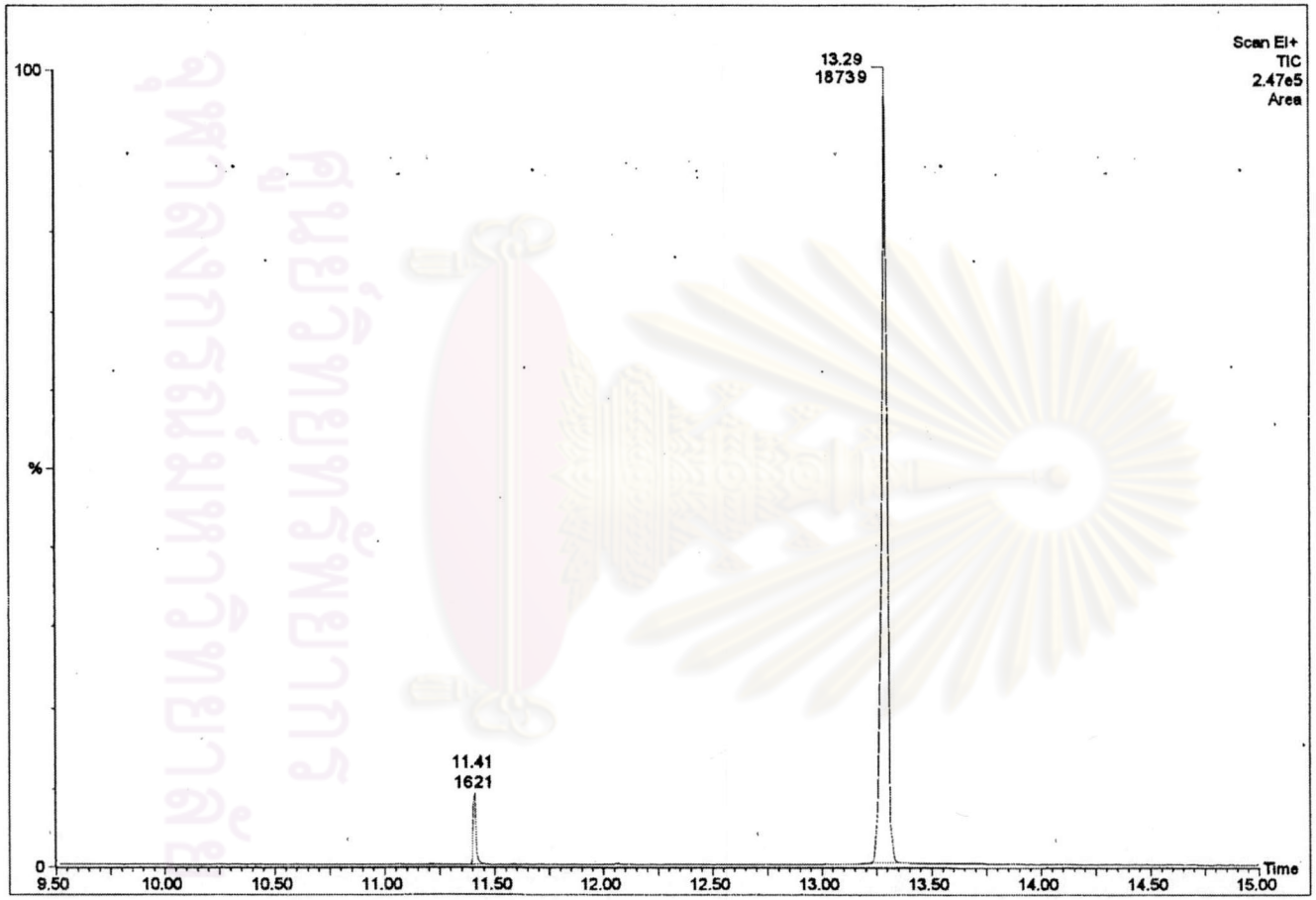


Figure D8 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 120 ^{o}C and 20 psig hydrogen pressure

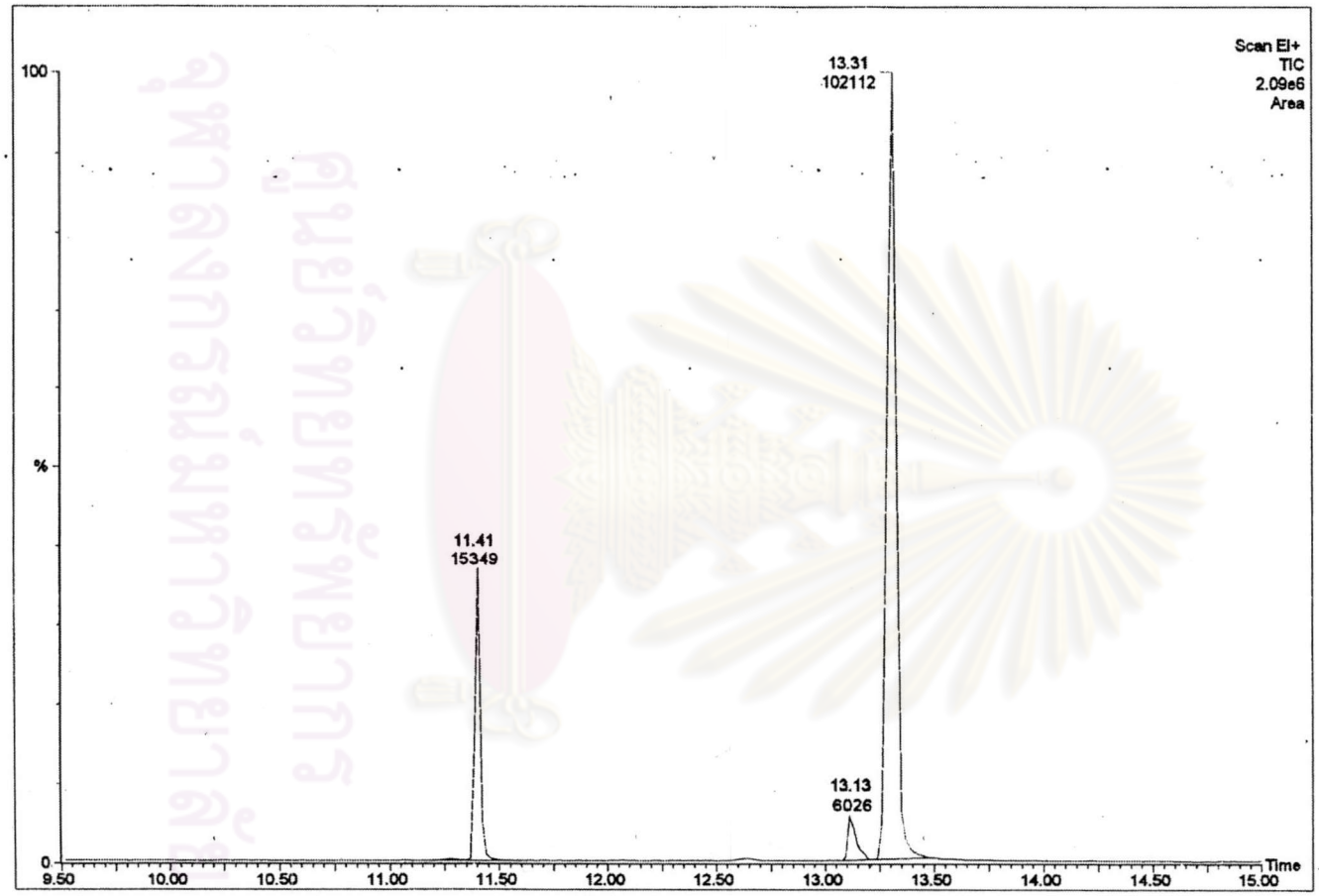


Figure D9 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 150 ^{o}C and 20 psig hydrogen pressure

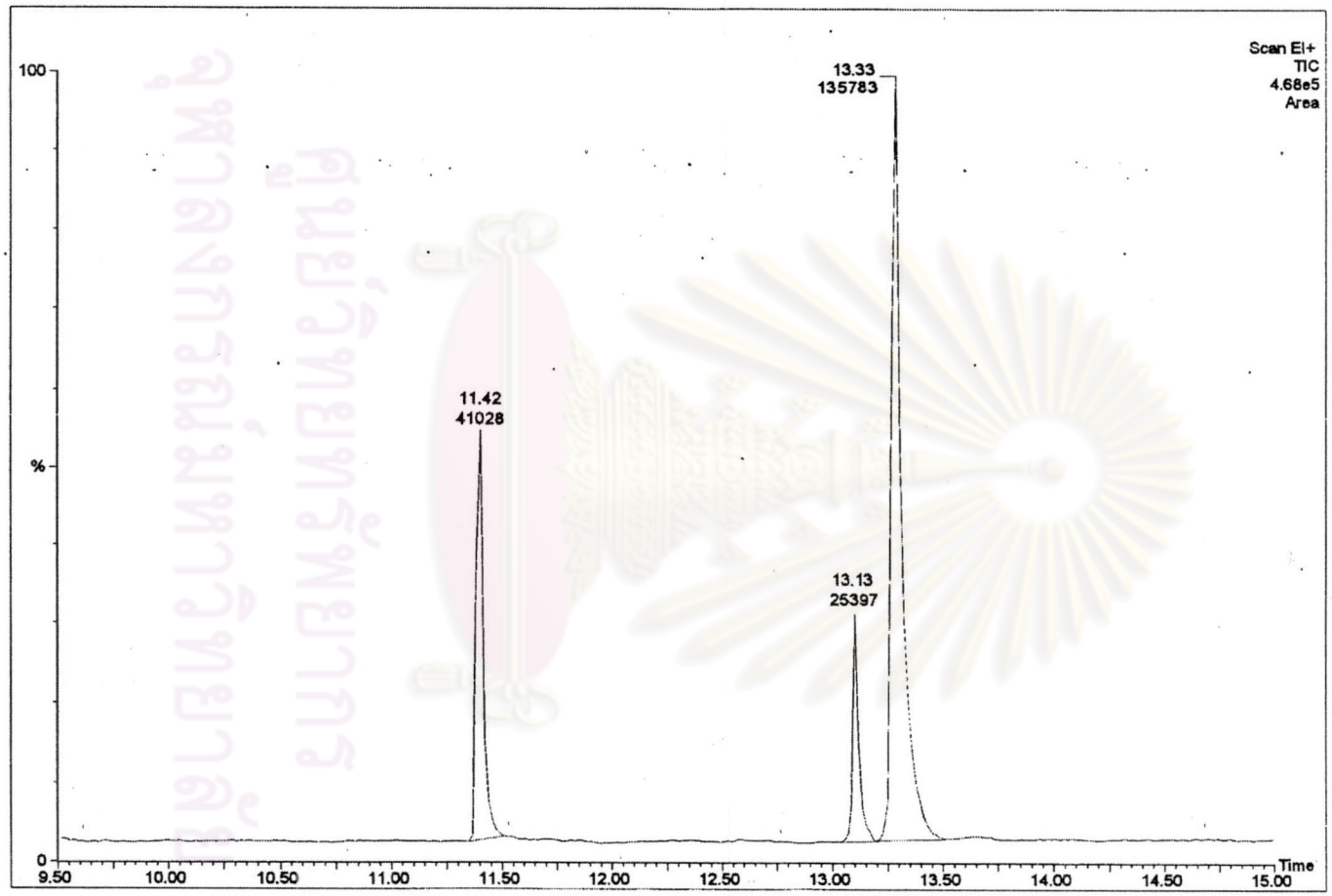


Figure D10 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 180 ^{o}C and 20 psig hydrogen pressure

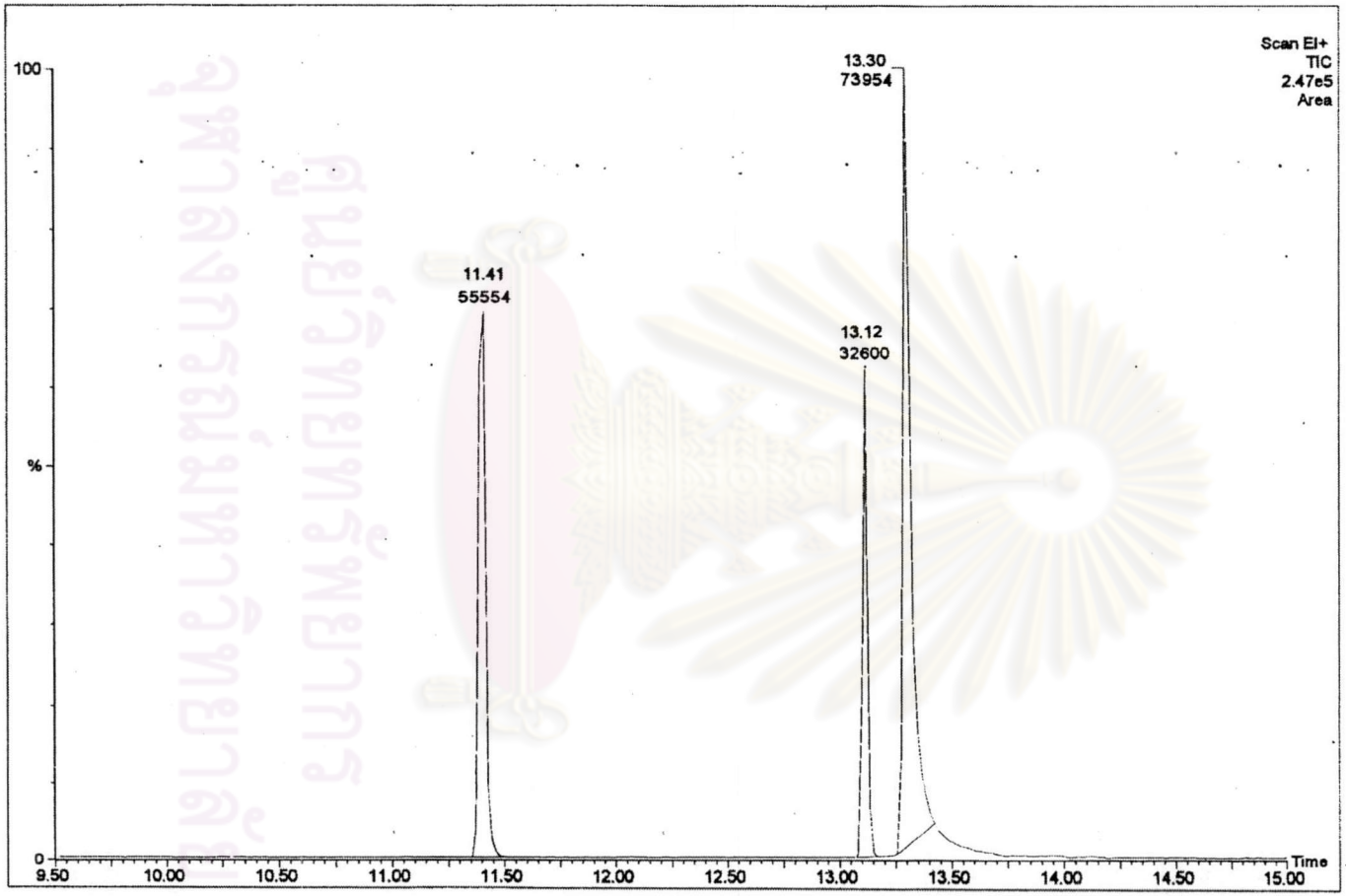


Figure D11 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 200 ^{o}C and 20 psig hydrogen pressure

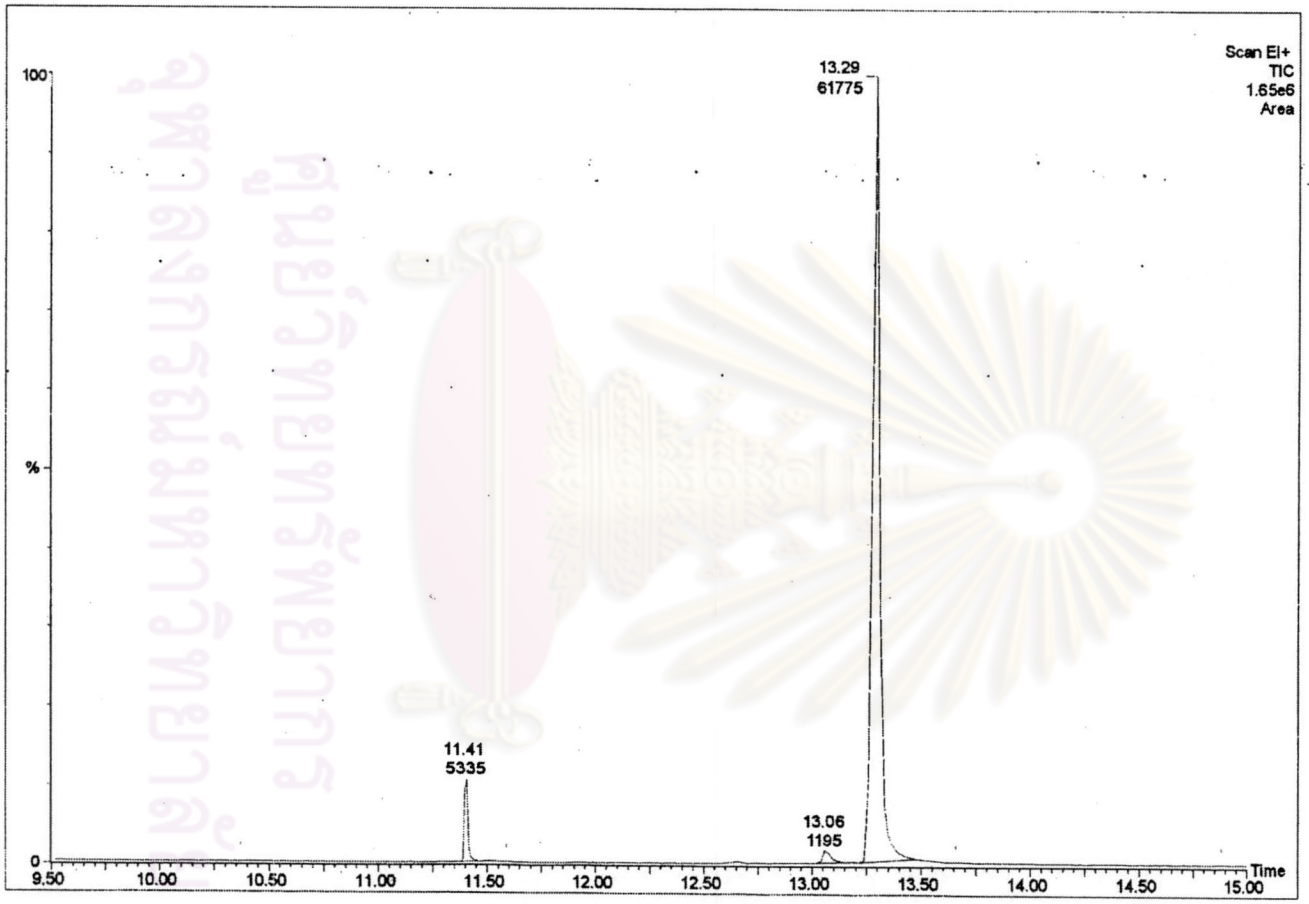


Figure D12 GC chromatogram of hydrogenated methyl ricinoleate using 20% Ni catalyst at 90 ^{o}C and 20 psig hydrogen pressure

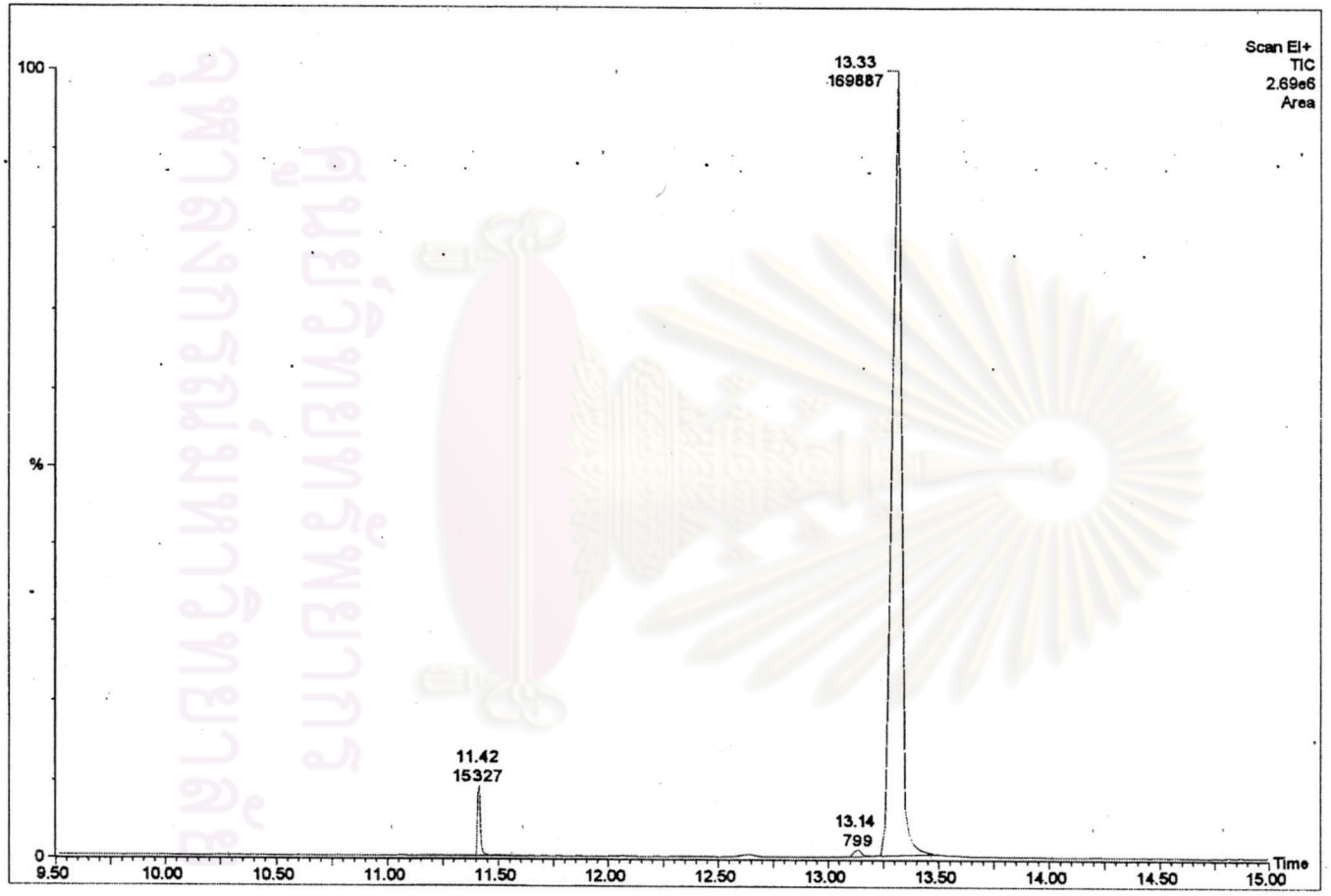


Figure D13 GC chromatogram of hydrogenated methyl ricinoleate using 20% Ni catalyst at 120 °C and 20 psig hydrogen pressure

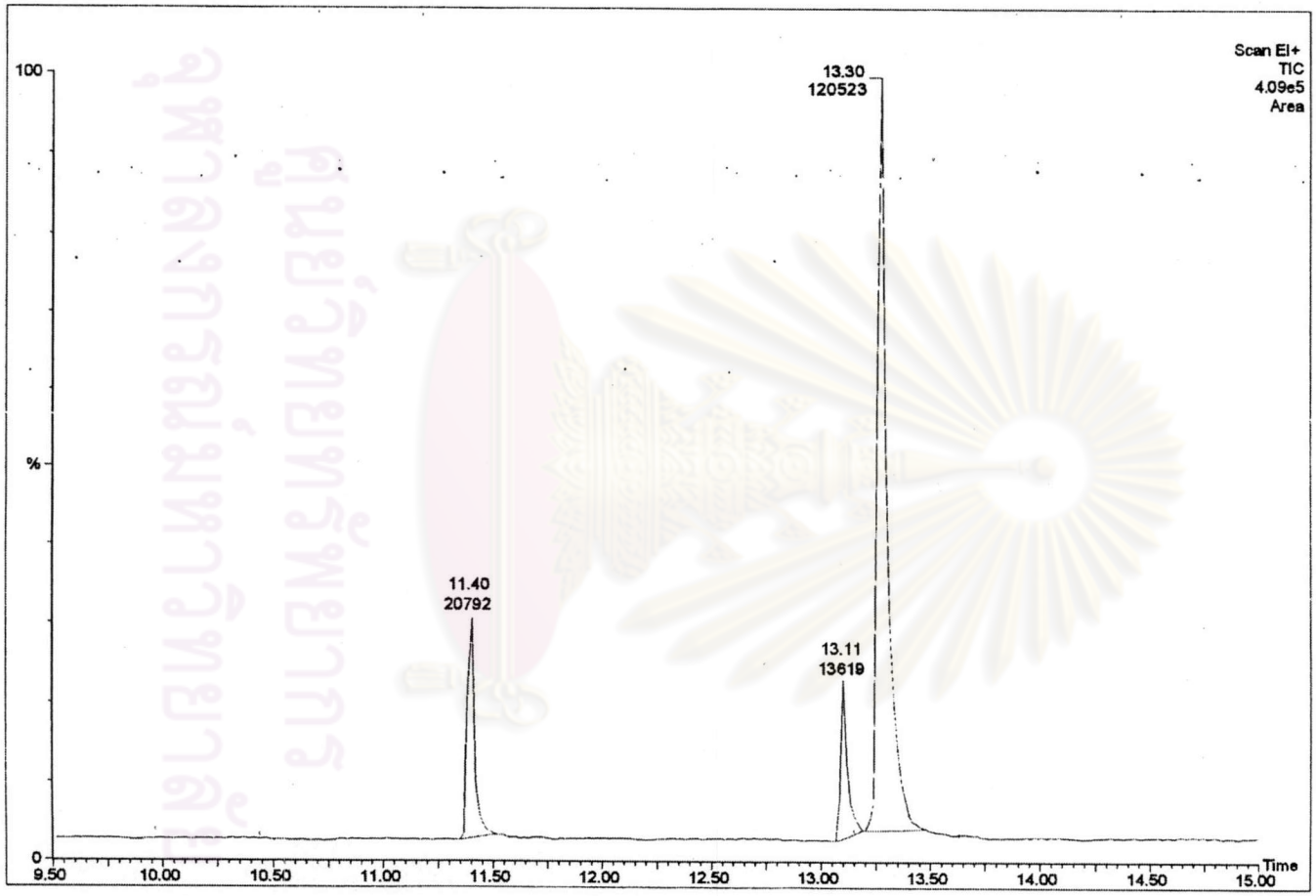


Figure D14 GC chromatogram of hydrogenated methyl ricinoleate using 20% Ni catalyst at 150 ^{o}C and 20 psig hydrogen pressure

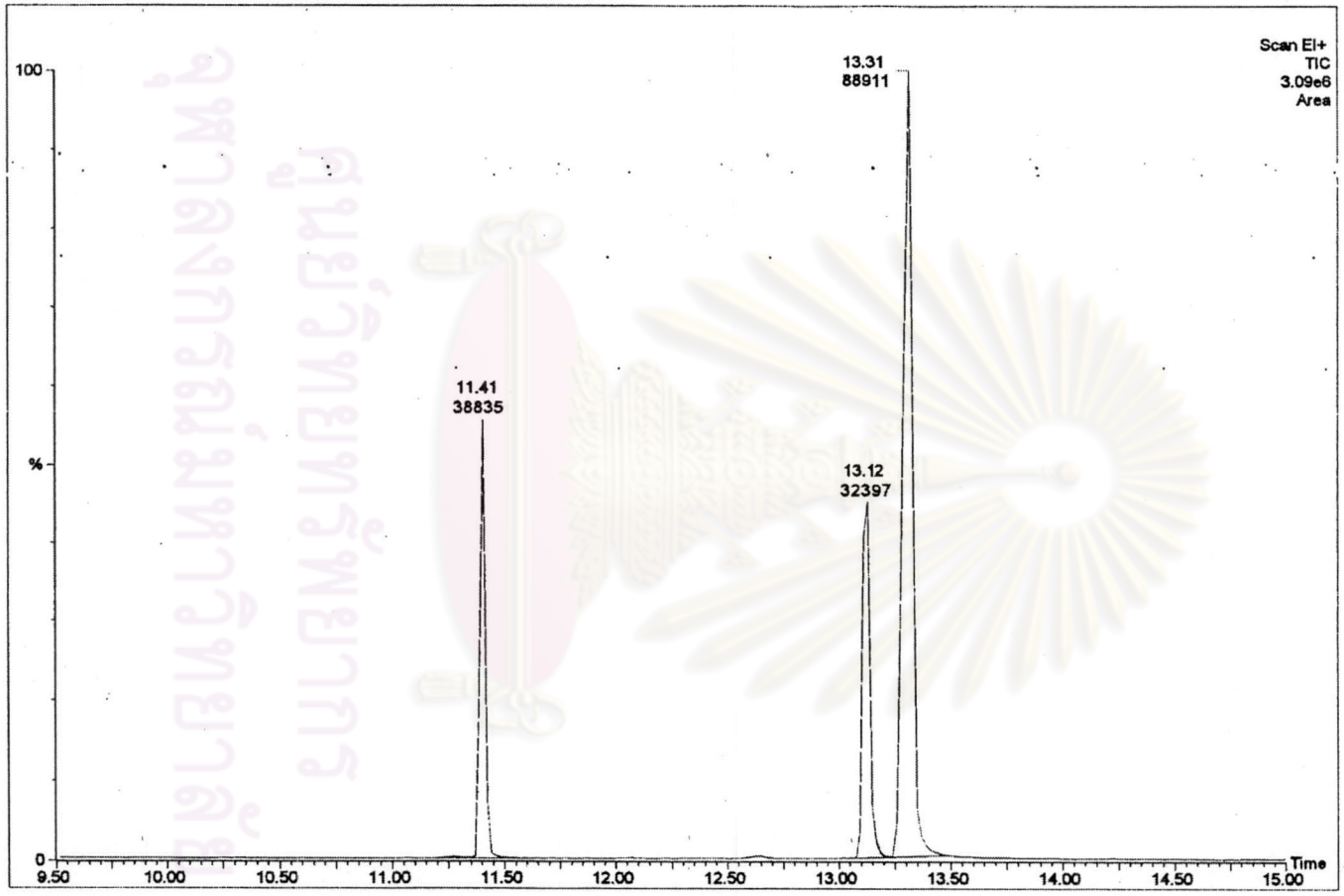


Figure D15 GC chromatogram of hydrogenated methyl ricinoleate using 20% Ni catalyst at 180 ^{o}C and 20 psig hydrogen pressure

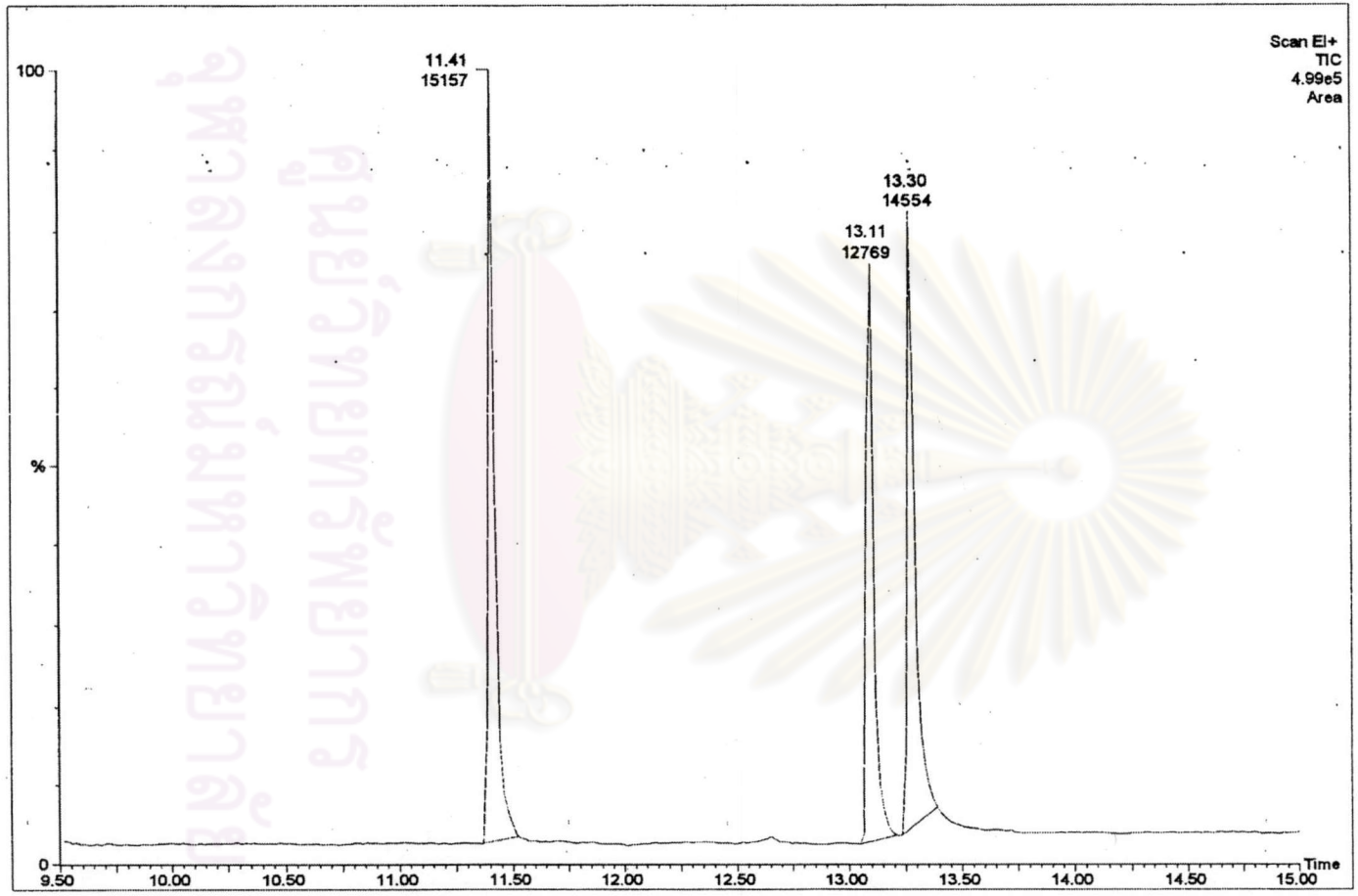


Figure D16 GC chromatogram of hydrogenated methyl ricinoleate using 20% Ni catalyst at 200 ^{o}C and 20 psig hydrogen pressure

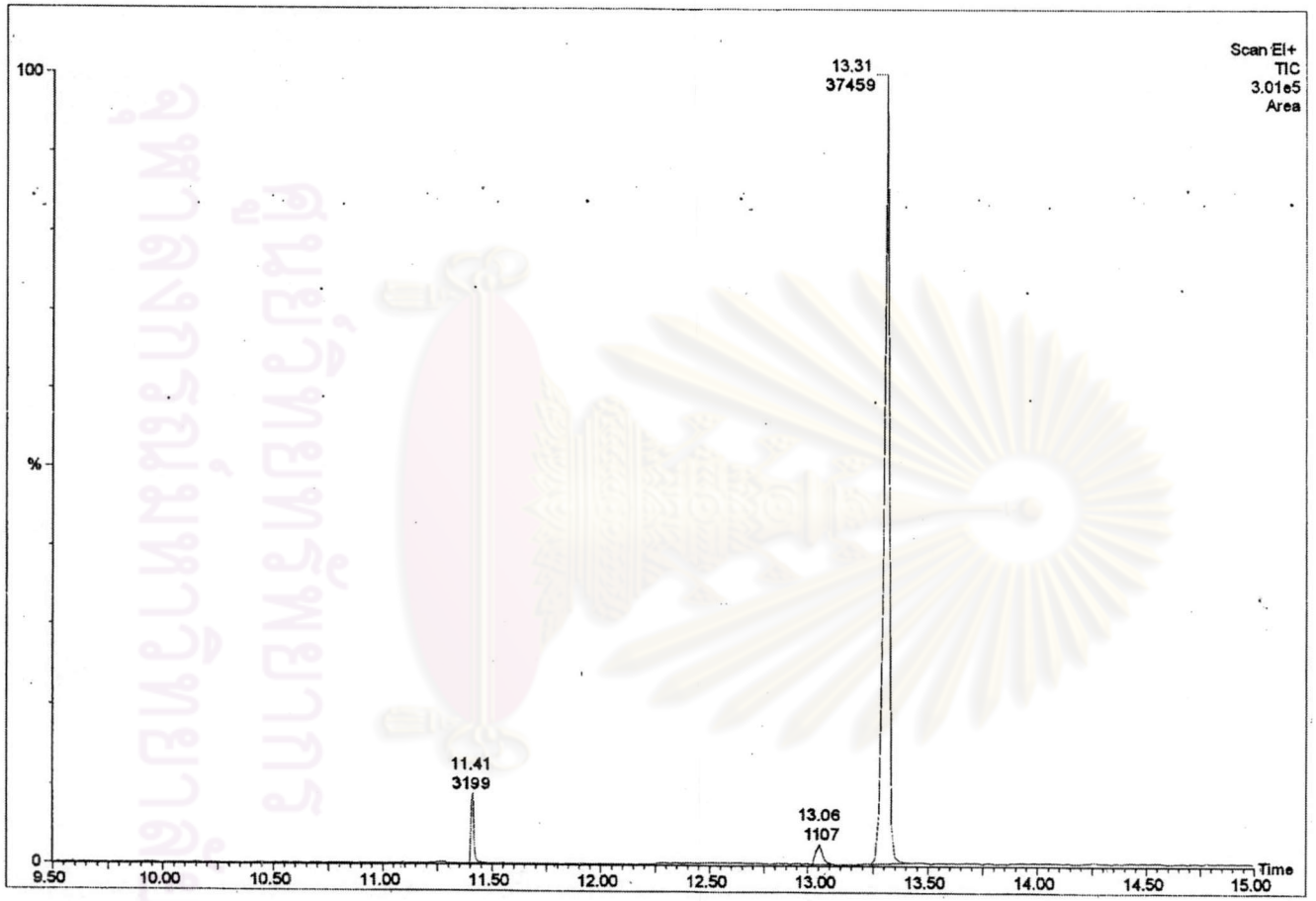


Figure D17 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 90 ^{o}C and 40 psig hydrogen pressure

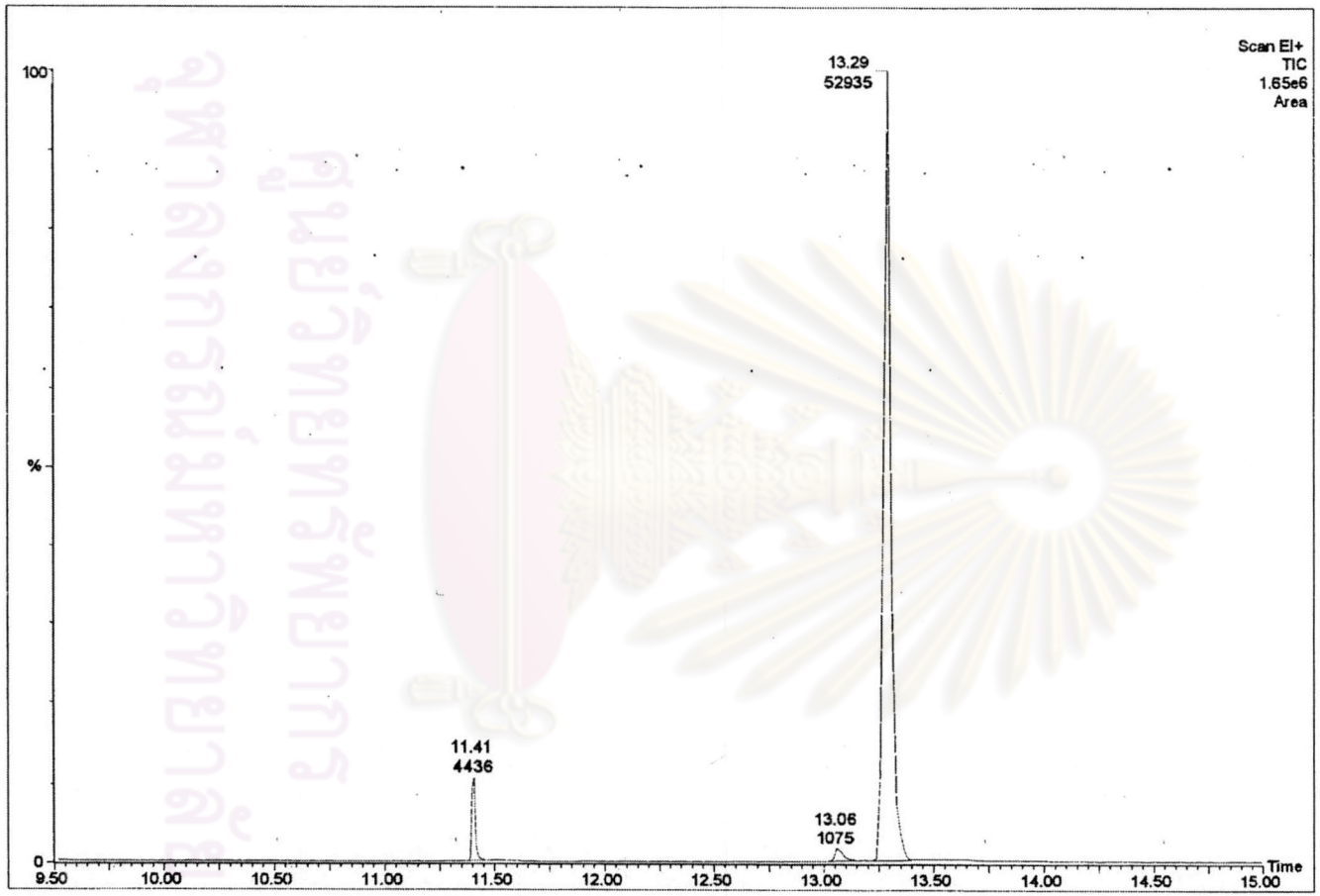


Figure D18 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 90 ^{o}C and 60 psig hydrogen pressure

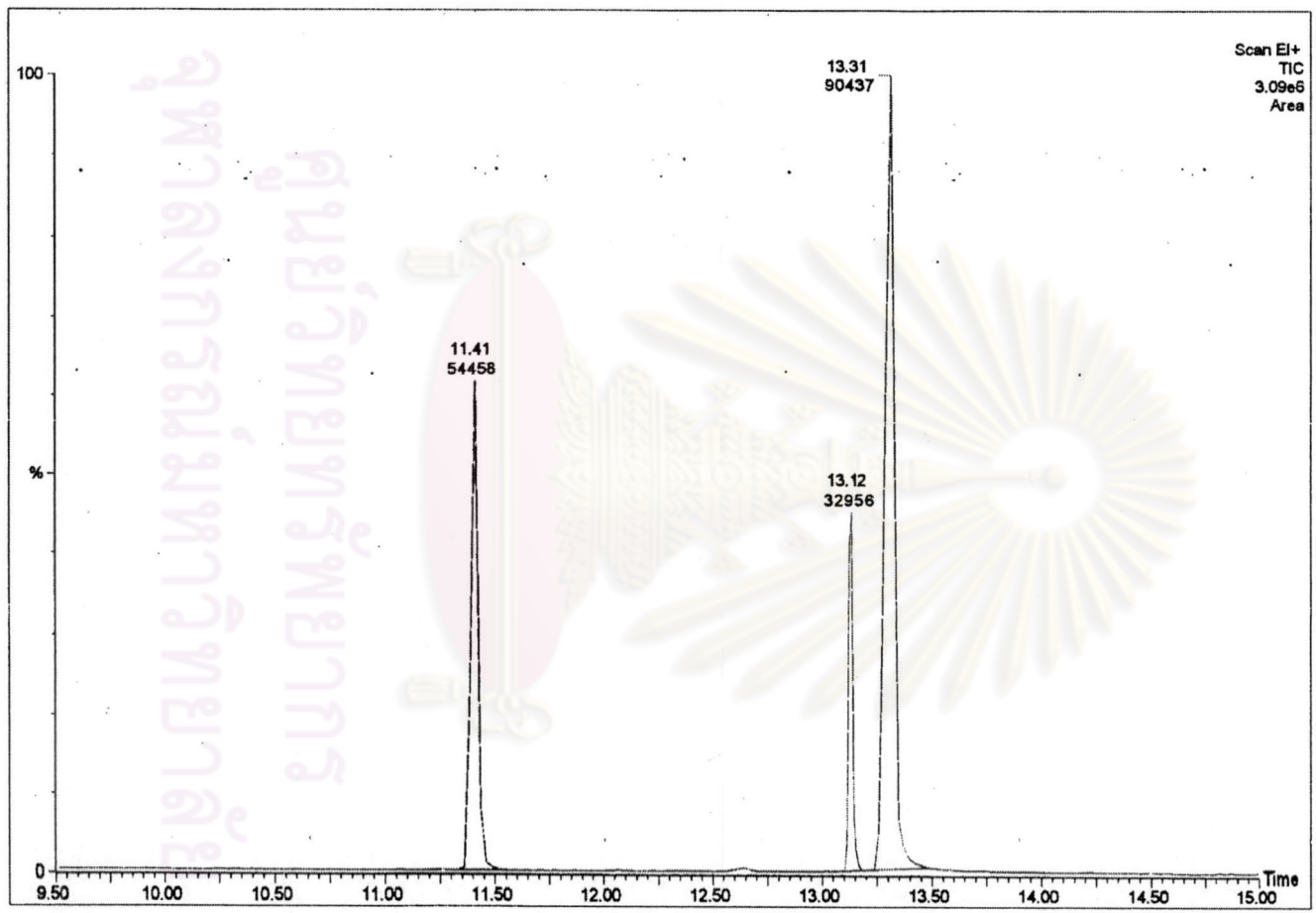


Figure D19 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 200 ^{o}C and 40 psig hydrogen pressure

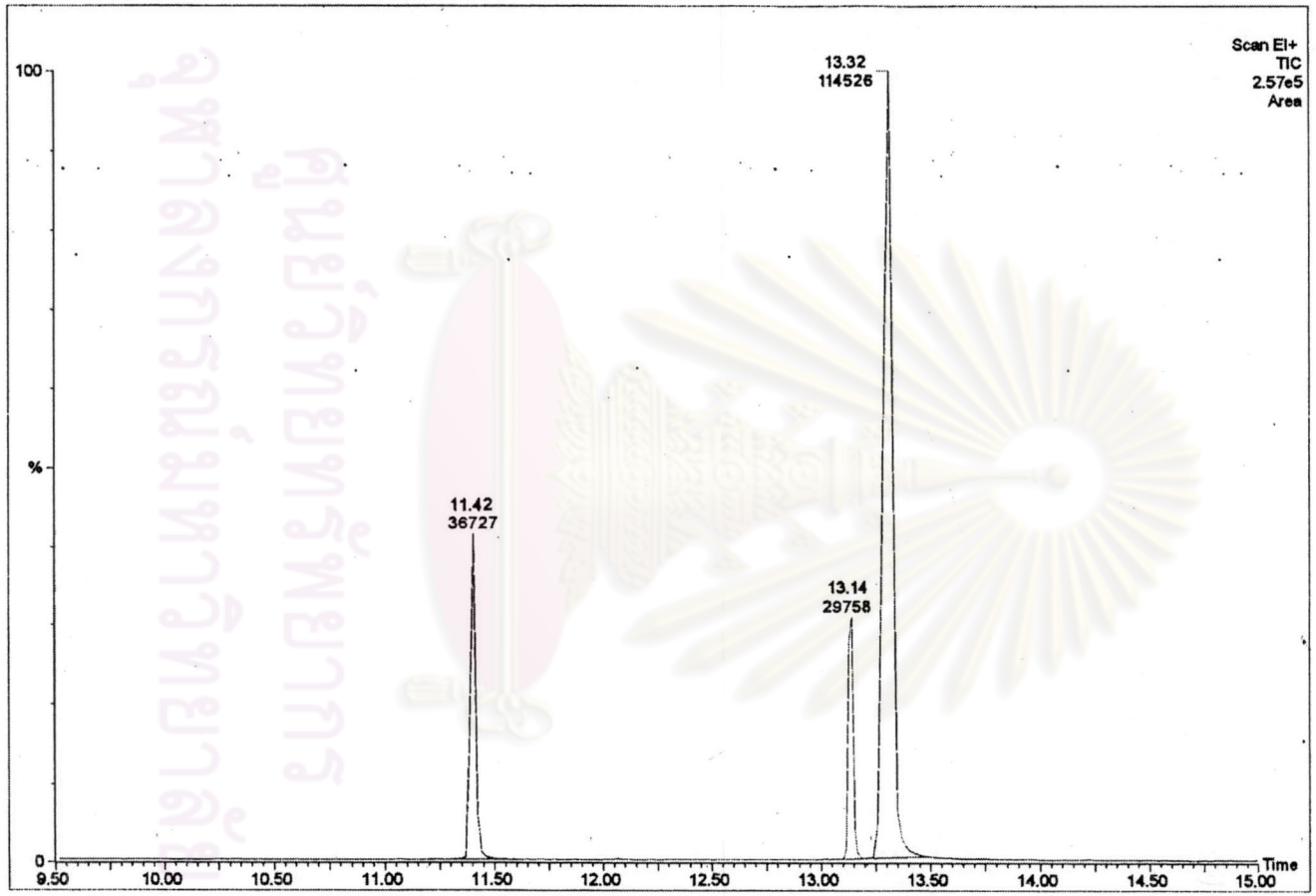


Figure D20 GC chromatogram of hydrogenated methyl ricinoleate using 15% Ni catalyst at 200 ^{o}C and 60 psig hydrogen pressure

APPENDIX E

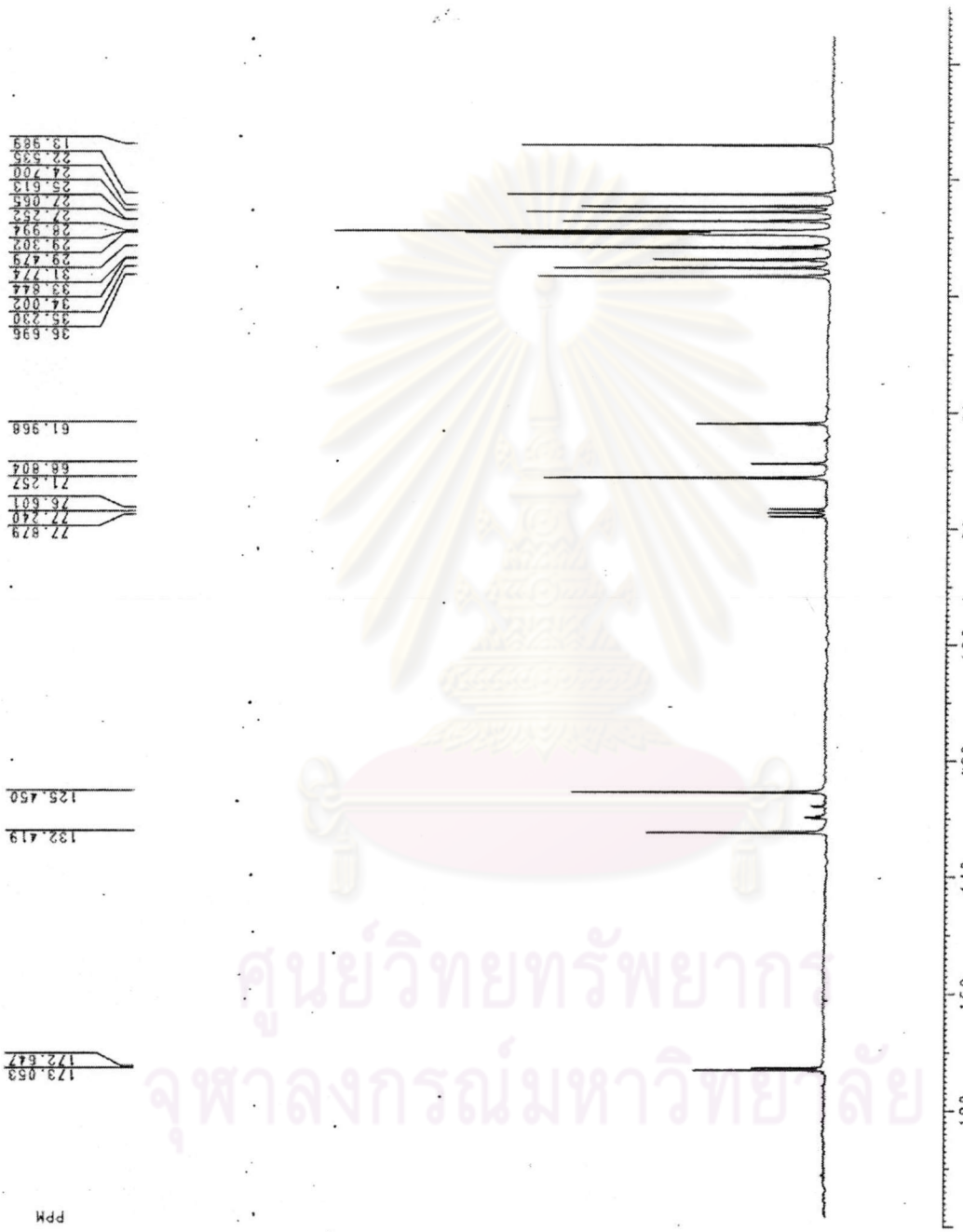


Figure E1 ^{13}C-NMR spectrum of castor oil

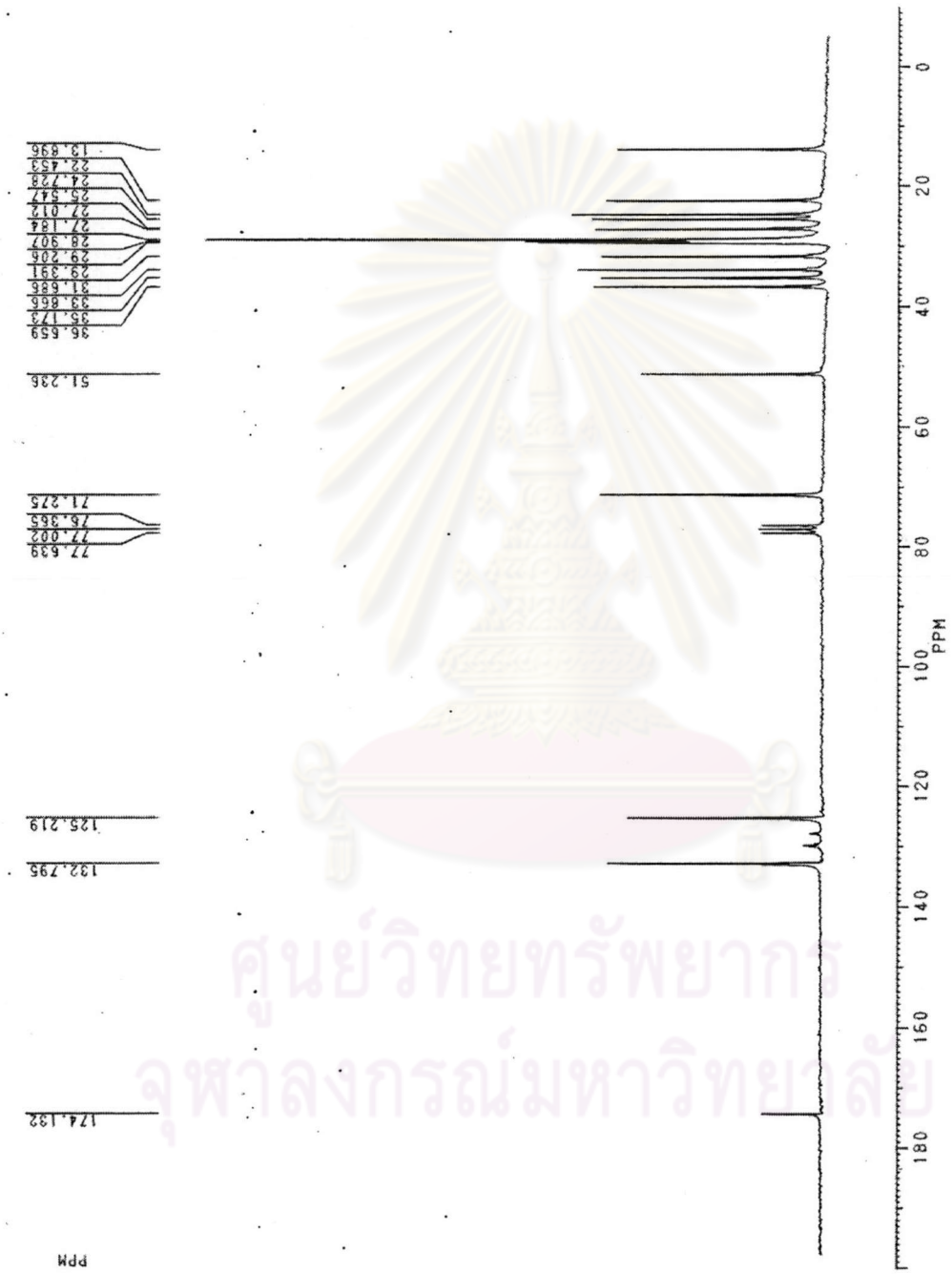


Figure E2 ^{13}C-NMR spectrum of castor oil methyl ester

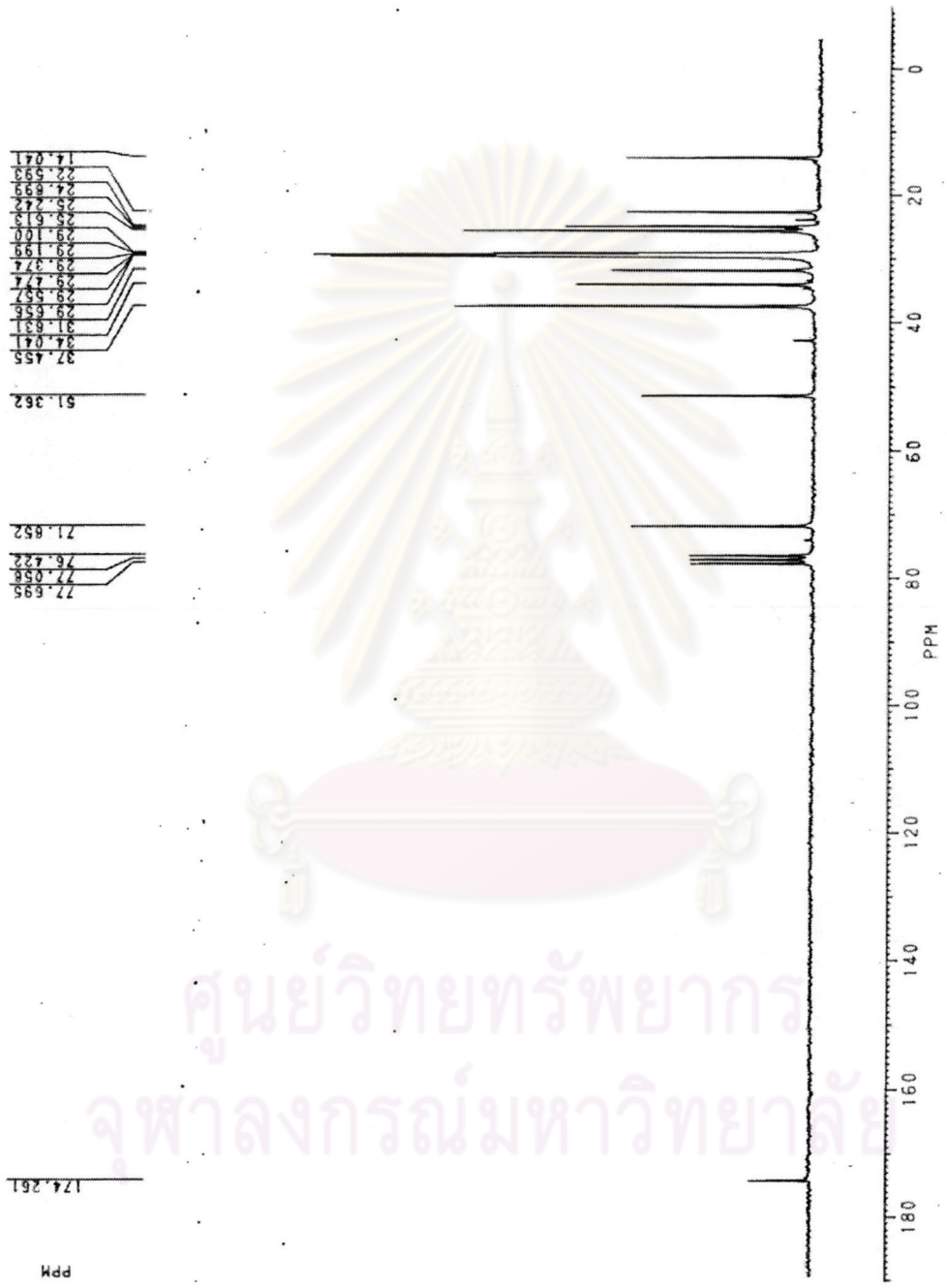


Figure E3 ^{13}C-NMR spectrum of hydrogenated product at 90 ^{o}C

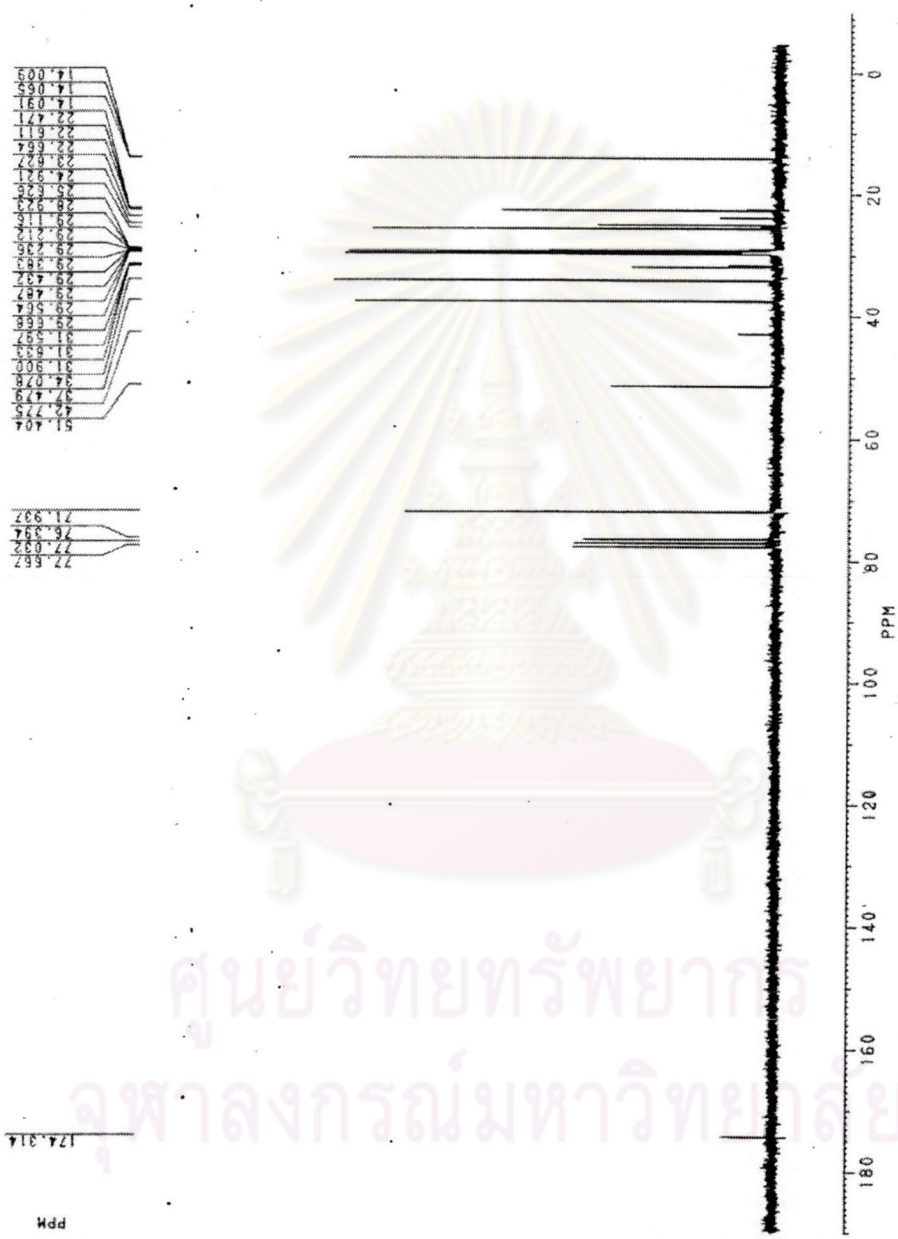


Figure E4 ^{13}C-NMR spectrum of hydrogenated product at 120 ^{o}C

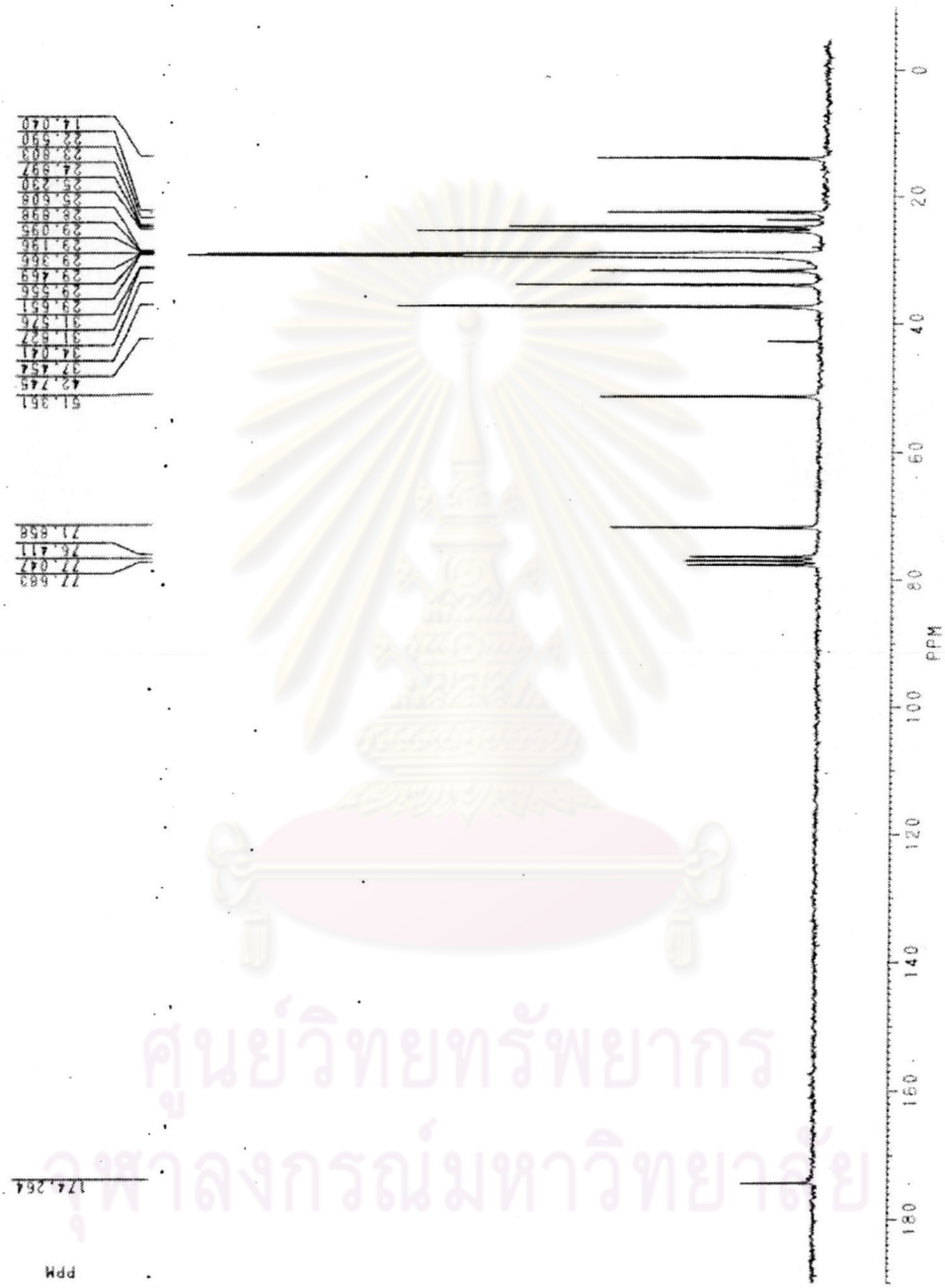


Figure E5 ^{13}C-NMR spectrum of hydrogenated product at 150 ^{o}C

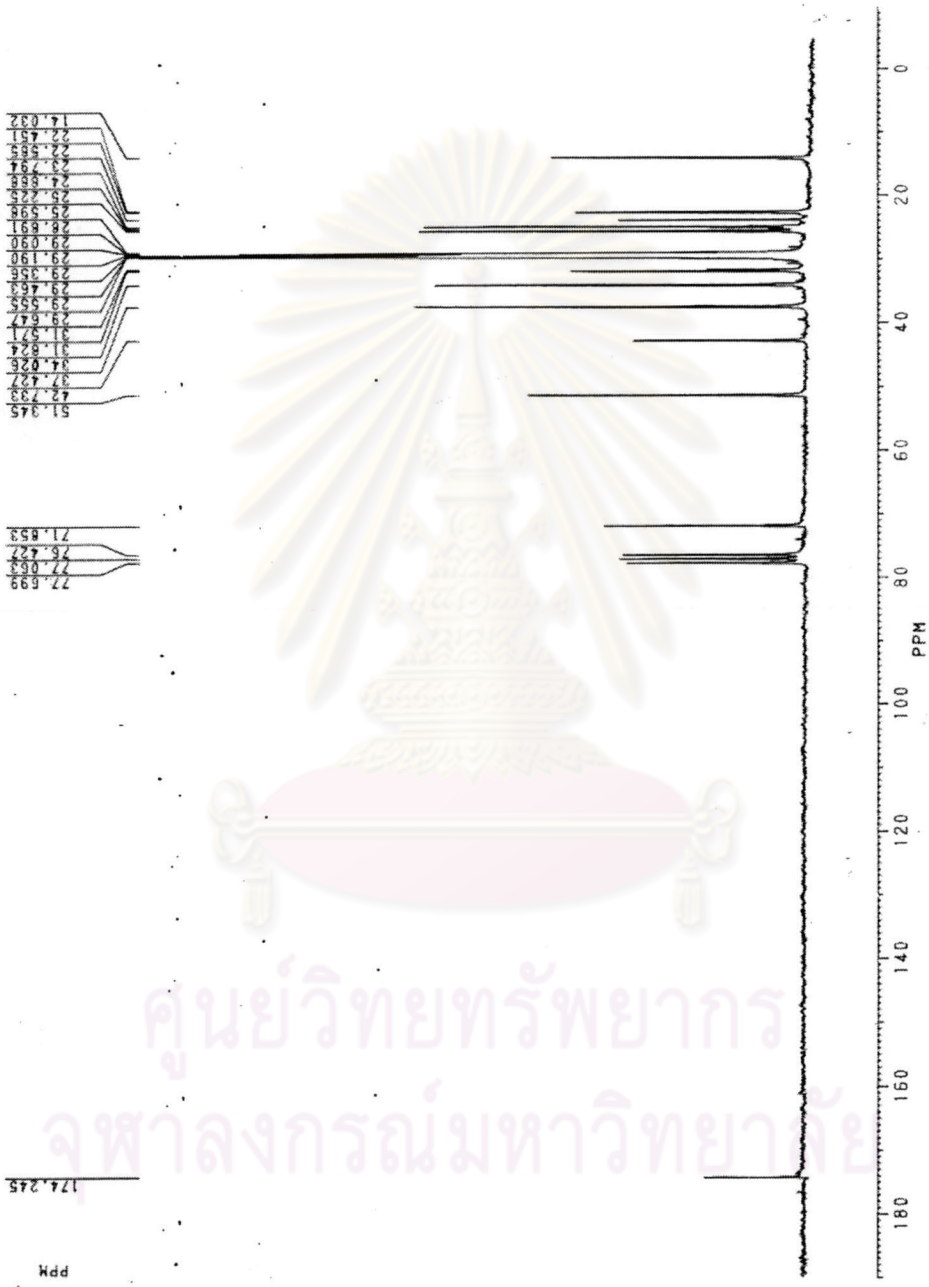


Figure E6 ^{13}C-NMR spectrum of hydrogenated product at 180 ^{o}C

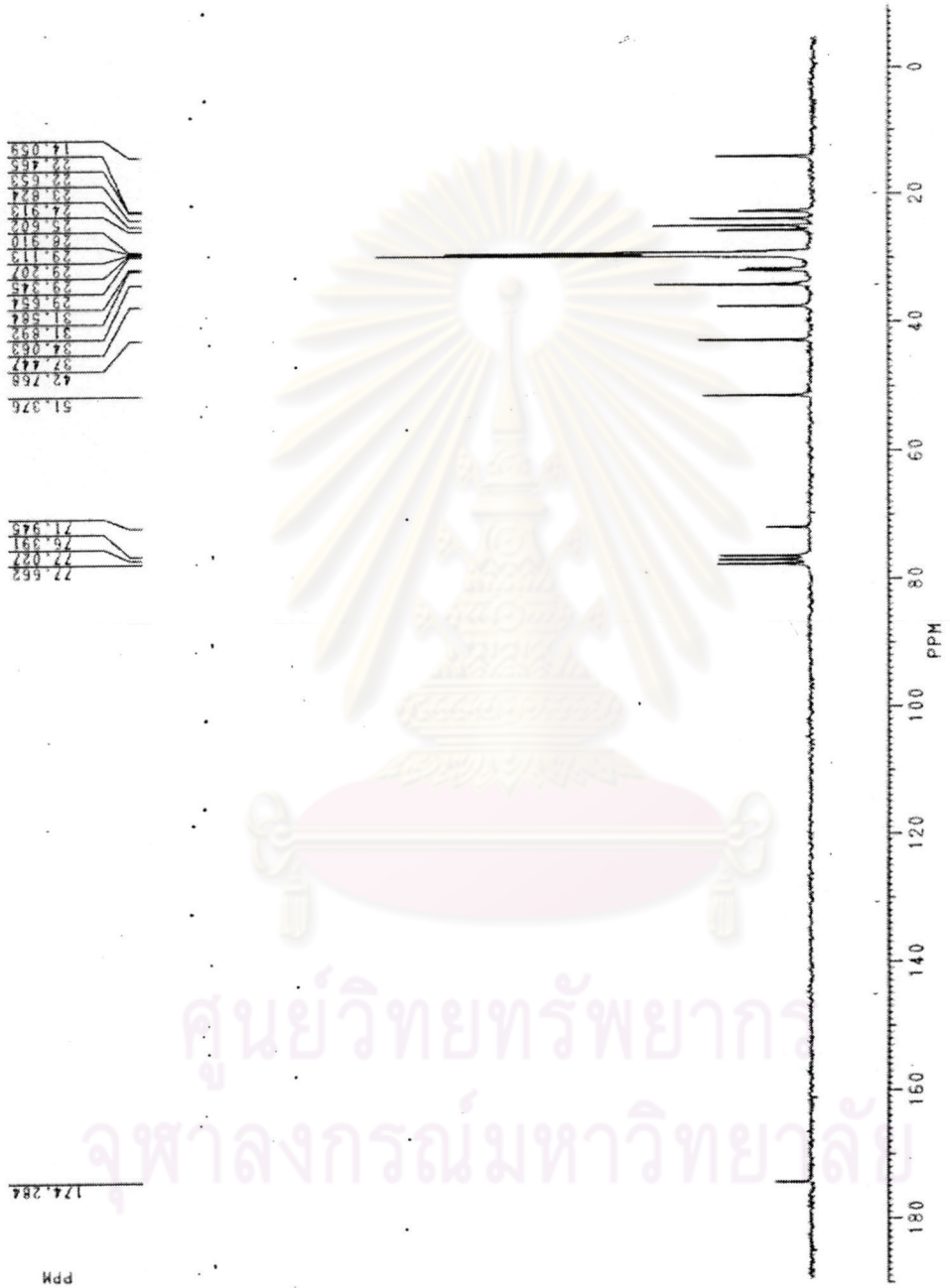


Figure E7 ^{13}C-NMR spectrum of hydrogenated product at 200 °C

VITA

Mr. Niti Leangjan was born on November 14, 1978 in Chonburi. He received his Bachelor Degree of Science in Chemistry from Chulalongkorn University, in 1999. He continued his Master Degree of Science in Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University in 1999 and completed the program in 2002.