

## CHAPTER III

### EXPERIMENTS

#### INSTRUMENTS

1. Infrared Spectrophotometers
  - : Perkin-Elmer model FT-IR 1760X
  - : Perkin-Elmer model 1720 FTIR
  - : Perkin-Elmer model 283
2. Nuclear Magnetic Resonance Spectrophotometer
  - : Jeol JNM-A500
  - : Bruker BZH 200/52
3. GC-MS Spectrometer
  - : JMS-DX300
4. Melting Point Apparatus
  - : Buchi capillary melting point apparatus

#### CHEMICALS

- 1-Bromopropane (Fluka Chemika)
- Chloroacetic acid (Fluka Chemika)
- Chlorosulfonyl isocyanate (Sigma)
- Ethanol, absolute (Merck)
- Formic acid, 85% (Carlo Erba)
- Glycerol (Widhyasom)

Hydrochloric acid, concentrated (BDH  
Laboratory-supplies)  
Magnesium, turnings (Merck-Schuchardt)  
Oxalic acid, dihydrate (Mallinckrodt  
Chemical-Works)  
Sodium hydride, 80% in mineral oil  
(Merck-Schuchardt)  
Sulfuric acid, concentrated (Merck)  
p-Toluenesulfonic acid, monohydrate (Sigma)  
Triethyl orthoformate (Fluka Chemika)  
Zinc chloride, anhydrous (Merck)  
All solvents used were either B.P. or  
laboratory-grade

#### Ethyl formate

A mixture of 145.1 ml (2.68 mol) of formic acid (85%) and 79.0 ml (1.34 mol) of absolute ethanol was refluxed for 24 hours. Then a fractionating column was attached to the flask. The reaction mixture was distilled and the liquid passing over at 52-54 °C was collected. The yield was about 75.8 ml (71%) (Furniss et al., 1991).

#### 4-Heptanol

A solution of propylmagnesium bromide was prepared from 28.2 g. (1.16 mol) of magnesium turnings, 142.8 g.

(105.5 ml, 1.16 mol) of propylbromide and 500 ml of dry diethyl ether in 1-litre flask. The flask containing the resulting Grignard reagent was cooled in an ice bath. A solution of 43 g. (46.9 ml, 0.58 mol) of ethyl formate in 100 ml of dry ether was placed in a separatory funnel. The solution of the Grignard reagent was stirred and the ethyl formate solution was run in at such a rate that the ether refluxed gently. The ice bath was removed and the stirring was continued for one hour.

About 100 ml of water was placed in the separatory funnel and run in into the vigorously stirred reaction mixture at such a rate that rapid refluxing occurred and was then followed by a cold solution of 400 ml of 10% sulfuric acid. Two practically clear layers would be present in the flask. The ethereal layer was decanted as much as possible into a separatory funnel and washed well with water. The solution was dried over anhydrous sodium sulfate and then the solvent was evaporated on a rotary evaporator. The residue was distilled and 4-heptanol was collected at 153-155 °C. The yield was about 27 g. (40 %).

4-Heptanol obtained was confirmed by comparing its IR spectrum with that of authentic compound.

IR. : 3600-3100  $\text{cm}^{-1}$  ( $\nu$  O-H)  
 (Neat) 3000-2840  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 1480-1350  $\text{cm}^{-1}$  ( $\sigma$  C-H)  
 1160-980  $\text{cm}^{-1}$  ( $\nu$  C-O)

(Figure 19)

#### 4-Chloroheptane

A mixture of 104.1 g. (73.3 ml, 0.9 mol) of 4-heptanol and a solution of 135 g. of anhydrous zinc chloride in 105 g. of concentrated hydrochloric acid was refluxed for 12 hours. The reaction mixture was cooled and extracted with chloroform. The combined chloroform solution was washed well with water. The solution was dried over anhydrous sodium sulfate and then the solvent was evaporated. The residue was distilled and 4-chloroheptane was collected at 148-150.5°C. The yield was about 67 g. (55%).

IR. : 3000-2800  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 (Neat) 1480-1360  $\text{cm}^{-1}$  ( $\sigma$  C-H)  
 1320-1200  $\text{cm}^{-1}$  ( $\sigma$  (wagging)  $\text{CH}_2$ )  
 780-590  $\text{cm}^{-1}$  ( $\nu$  C-Cl)

(Figure 20)



### Ethyl chloroacetate

A mixture of 50 g. (0.53 mol) of chloroacetic acid and 250 ml of absolute ethanol was refluxed in the presence of 1 ml of concentrated sulfuric acid for 10 hours. An azeotropic mixture of water formed and ethanol was distilled at 78°C. The residue was distilled and ethyl chloroacetate was collected at 144.5°C.

IR.	:3000-2800	cm <sup>-1</sup>	(ν C-H)
(Neat)	1757	cm <sup>-1</sup>	(ν C=O)
	1470-1370	cm <sup>-1</sup>	(δ C-H)
	1313-1098	cm <sup>-1</sup>	(ν <u>C(=O)-O-C</u> )
	1028	cm <sup>-1</sup>	(ν <u>C(=O)-O-C</u> )
	782-699	cm <sup>-1</sup>	(ν C-Cl)

(Figure 21)

### Ethyl ethoxyacetate

A solution of sodium ethoxide was prepared by dissolving 18.76 g. (0.82 mol) of sodium in 300 ml of absolute ethanol. The stirrer was started, and to the solution of sodium ethoxide, 100 g. (103.1 ml, 0.82 mol) of ethyl chloroacetate was run in from the dropping funnel during 2 hours and reaction mixture was refluxed for 30 minutes with stirring. The stirrer was stopped and sodium chloride formed was let to precipitate. The supernatant

liquor was decanted and the solvent was evaporated. If there was some precipitate left in the residue, it was filtered until a clear solution was obtained. The residue was distilled and ethyl ethoxyacetate was collected at 156-157°C. The yield was about 65.4 g. (61%).

IR.	:3000-2800	cm <sup>-1</sup>	(ν C-H)
(Neat)	1756	cm <sup>-1</sup>	(ν C=O)
	1277-1205	cm <sup>-1</sup>	(ν <u>C(=O)-O-C</u> )
	1138	cm <sup>-1</sup>	(ν <sub>as</sub> C-O-C)
	1034	cm <sup>-1</sup>	(ν <u>C(=O)-O-C</u> )

(Figure 22)

## 2-Propylpentanal

This compound was prepared from 2 different methods.

A. A Grignard reagent of 4-heptylmagnesium chloride was prepared as described below. To the flask was added 10 g. of magnesium turnings, 25 ml of dry THF and a magnetic stirrer bar. About 1 g. (0.007 mol) of 4-chloroheptane was introduced into the flask and was then followed by 6-10 drops of methyl iodide and a crystal of iodine to initiate the reaction. The flask was warmed gently. The onset of the reaction was accompanied by the disappearance of the iodine colour. When the reaction was progressing well, a sufficient amount of dry THF was added

to cover the magnesium and the stirrer was set in motion. The remainder (6.7 g, 0.05 mol) of 4-chloroheptane dissolved in THF was then added dropwise at such a rate that the reaction proceeded smoothly. When the addition of 4-chloroheptane was complete, the heating and stirring was continued until the reaction was complete. The flask was cooled and 8.45 g. (9.5 ml, 0.057 mol) of triethyl orthoformate was run in during about 10 minutes. The reaction mixture was refluxed for 6 hours, then the condenser was arranged for distillation and the ether was then removed on a water bath. The reaction mixture was allowed to cool. After standing overnight, to the reaction mixture was added about 60 ml of ice-bathed 5N hydrochloric acid with stirring; the contents of the flask was kept cool by the occasional addition of a little crushed ice. When all the white solid had passed into solution, it was transferred to a separatory funnel and the upper layer of 2-propylpentanal diacetal was removed. The acetal was hydrolyzed by refluxing with a solution of 2.5 ml of concentrated sulfuric acid in 30 ml. of water for 30 minutes. The aldehyde was then steam distilled. The distillate was extracted with ether. The combined ether extract was then washed well with water, dried over anhydrous sodium sulfate and the solvent was then evaporated. The yield was about 0.5 g. (7%).



B. A solution of propylmagnesium bromide was prepared from 130 g. (96 ml, 1.06 mol) of propylbromide magnesium turnings 27 g. (1.10 mol) and 500 ml of dry ether in a 1-litre flask. The flask containing the resulting Grignard reagent was cooled in an ice bath. A solution of 67.6 g. (69.7 ml, 0.53 mol) of ethyl ethoxyacetate in 100 ml of dry ether was placed in the separatory funnel. The solution of the Grignard reagent was stirred and the ethyl ethoxyacetate was run in into it at such a rate that the ether refluxed gently. The ice bath was removed and the stirring was continued for 6 hours.

About 100 ml of water was placed in the separatory funnel and run in into the vigorously stirred reaction at such a rate that rapid refluxing occurred. This was followed by a cold solution of 300 ml of 10% sulfuric acid. Two practically clear layers were present in the flask. The ethereal layer was decanted as much as possible into a 500-ml round-bottomed flask. The remainder, including the aqueous layer was transferred into a separatory funnel. The residual solid was then washed with two 50 ml portions of ether and these washings were combined with the liquid in the separatory funnel which was then washed well with water, dried over anhydrous sodium sulfate and the solvent was evaporated. The residue was crude 1,1-dipropyl-2-ethoxyethanol (Figure 23). It was not further purified. The yield was 72 g. (80%). Then crude 1,1-



dipropyl-2-ethoxyethanol was transformed to 2-propyl-pentanal as described below.

The mixture of 72 g. (0.41 mol) of crude 1,1-dipropyl-2-ethoxyethanol and 75 g. (0.82 mol) of anhydrous oxalic acid was refluxed at 110-115°C for 4 hours. The reaction mixture was cooled and filtered. The filtrate was washed with sodium bicarbonate solution. The residue was washed well with water, dried over anhydrous sodium sulfate and the solvent was then evaporated. The aldehyde was isolated by steam distillation. The distillate was extracted with ether. The combined ether extract was washed well with water, dried over anhydrous sodium sulfate and the solvent was then evaporated. The yield was about 43 g. (80%).

IR. : 3600-3300  $\text{cm}^{-1}$  ( $\nu$  O-H)  
(Neat) 3000-2800  $\text{cm}^{-1}$  ( $\nu$  C-H)  
1462-1380  $\text{cm}^{-1}$  ( $\sigma$  C-H)  
1200-1000  $\text{cm}^{-1}$  ( $\nu$  C-O, ether)  
1200-878  $\text{cm}^{-1}$  ( $\nu$  C-O, alcohol)

(Figure 23)

IR. : 2960-2872  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 (Neat) 2810, 2696  $\text{cm}^{-1}$  ( $\nu$  aldehydic hydrogen)  
 1728  $\text{cm}^{-1}$  ( $\nu$  C=O)  
 1463-1305  $\text{cm}^{-1}$  ( $\sigma$  C-H)  
 1183-1071  $\text{cm}^{-1}$  ( $\nu$  C-O)  
 (Figure 24)

$^1\text{H-NMR}$  : 0.8-0.9 (t, 6H)  
 ( $\text{CDCl}_3$ ) 1.15-1.65 (m, 8H)  
 2.10-2.25 (m, 1H)  
 9.48-9.50 (d, 1H),  $\left| J \right|_{\text{HC-CHO}} = 3.1$   
 (Figure 25)

### [2-(1-Propylbutyl)-1,3-dioxolan-4-yl]methanol

A mixture of 23.6 g. (28.3 ml, 0.18 mol) of 2-propylpentanal, 23 g. (0.2 mol) of glycerol, 2 g. of p-toluenesulfonic acid monohydrate and 250 ml of benzene was refluxed for 20 hours. During refluxing the water produced from the reaction was azeotropically removed from the mixture and collected in a Dean and Stark water separator. The mixture was then cooled. The upper layer was removed and washed with sodium bicarbonate solution. The residue was washed well with water, dried over anhydrous sodium sulfate and the solvent was then evaporated. The purification was performed by column chromatographic technique, mobile phase used was solution

of hexane and ethyl acetate (2.3:1) and stationary phase was silica gel. The yield was about 14.8 g. (40%).

IR. : 3650-3200  $\text{cm}^{-1}$  ( $\nu$  O-H)  
 (Neat) 3000-2800  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 1480-1380  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 1200-900  $\text{cm}^{-1}$  ( $\nu$  O-C-O, acetal and C-O,  
 alcohol)

(Figure 26)

$^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectra : Figure 27-39.

( $\text{CDCl}_3$ )

#### [2-(1-Propylbutyl)-1,3-dioxolan-4-yl]methyl sulfamate

A solution of sulfamoyl chloride ( $\sim 0.033$  mol) in acetonitrile was prepared by adding a solution of 85% formic acid (1.79 ml), 0.033 mol) in 12 ml of acetonitrile over 15 minutes to a solution of chlorosulfonyl isocyanate (2.88 ml, 0.033 mol) in 60 ml of acetonitrile in an ice bath. The gas ( $\text{CO}$  and  $\text{CO}_2$ ) evolution ceased in 3 hours.

To a solution of [2-(1-propylbutyl)-1,3-dioxolan-4-yl]methanol (4.05 g, 0.02 mol) in 10 ml of DMF cooled to  $0-5^\circ\text{C}$  was added the suspension of 80% sodium hydride in mineral oil (0.75 g, 0.025 mol) in 22.5 ml DMF. The mixture was stirred under nitrogen for 30 minutes.

The solution of sulfamoyl chloride was then added portionwise into the solution of sodium alkoxide and the reaction mixture was stirred for an additional 15 minutes.

The mixture was then poured into ice water and extracted with ether. The combined ether extracts were washed well with water, dried over anhydrous sodium sulfate and the solvent was evaporated. The purification was performed by column chromatographic technique, mobile phase used was a solution of hexane and ethyl acetate (2.5:1), stationary phase was silica gel. The yield was about 3 g. (53%).

IR. : 3400-3200  $\text{cm}^{-1}$  ( $\nu$  N-H)  
 (KBr disc) 3000-2800  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 1650-1550  $\text{cm}^{-1}$  ( $\nu$  N-H)  
 1480-1450  $\text{cm}^{-1}$  ( $\nu$  C-H)  
 1400-1350  $\text{cm}^{-1}$  ( $\nu$ as S(=O)<sub>2</sub>)  
 1180-1165  $\text{cm}^{-1}$  ( $\nu$ sym S(=O)<sub>2</sub>)  
 1150-900  $\text{cm}^{-1}$  ( $\nu$  C-O, acetal)  
 (Figure 40)

<sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra : Figure 41-53.

(CDCl<sub>3</sub>)

MS spectrum : Figure 54



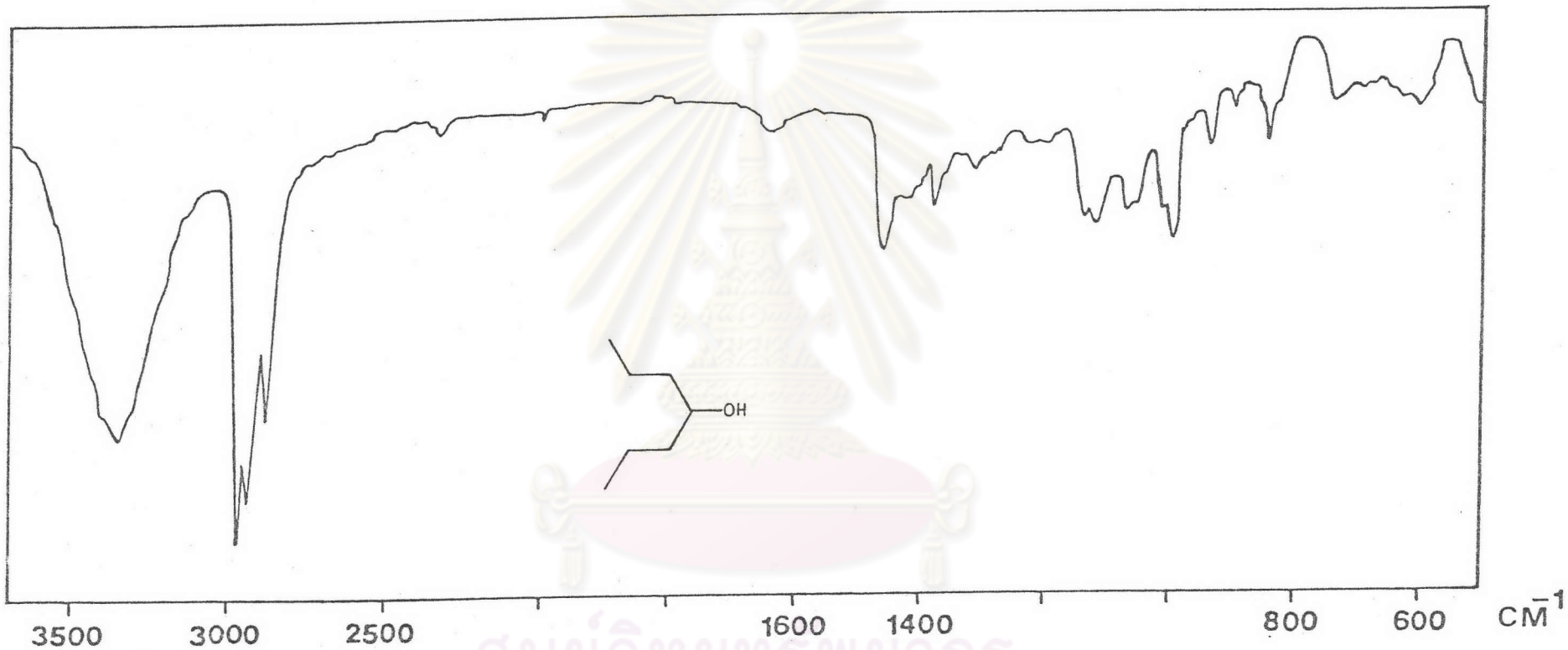


Figure 19. The IR spectrum (Neat) of 4-heptanol

ศูนย์วิทยทรัพยากร  
จุฬาลงกรณ์มหาวิทยาลัย

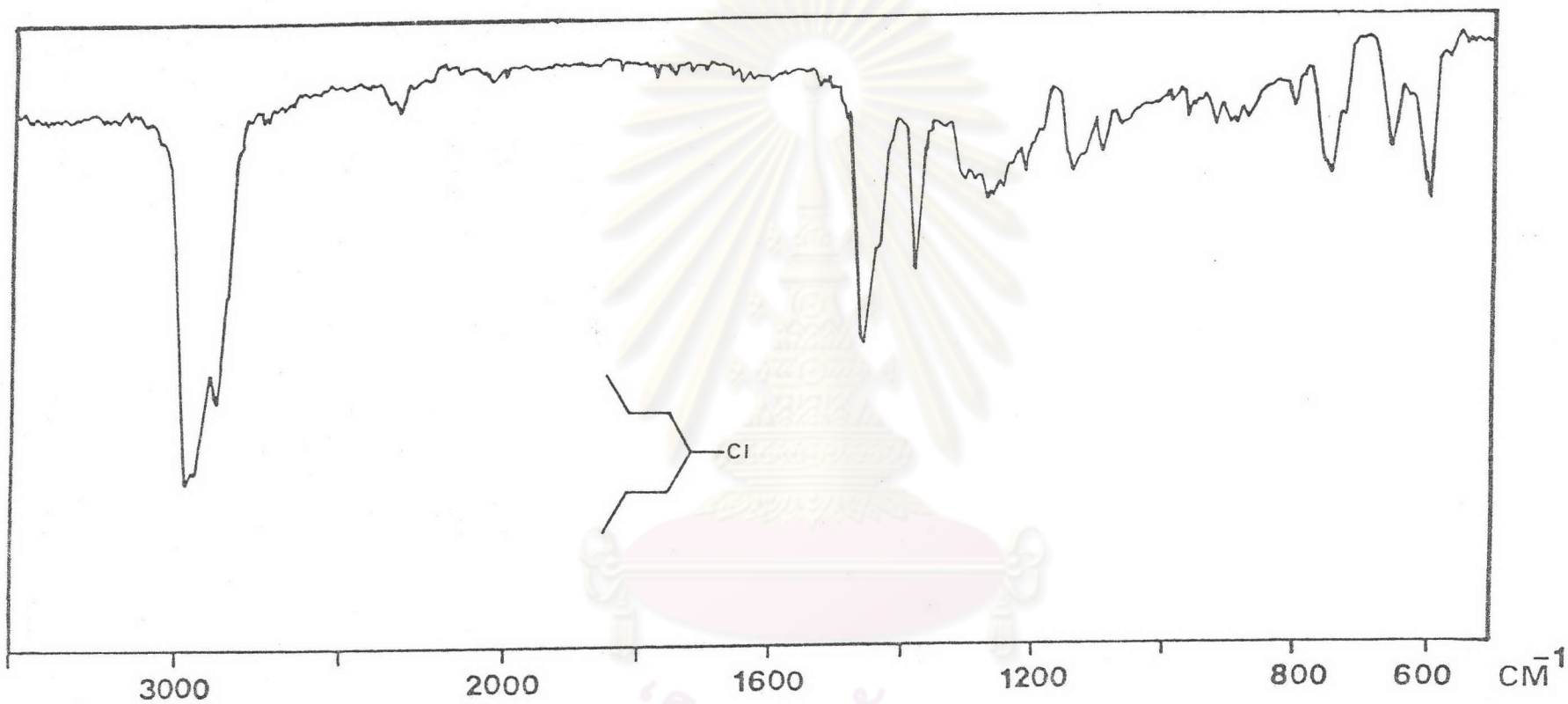


Figure 20. The IR spectrum (Neat) of 4-chloroheptane

ศูนย์วิทยุทรัพยากร  
จุฬาลงกรณ์มหาวิทยาลัย

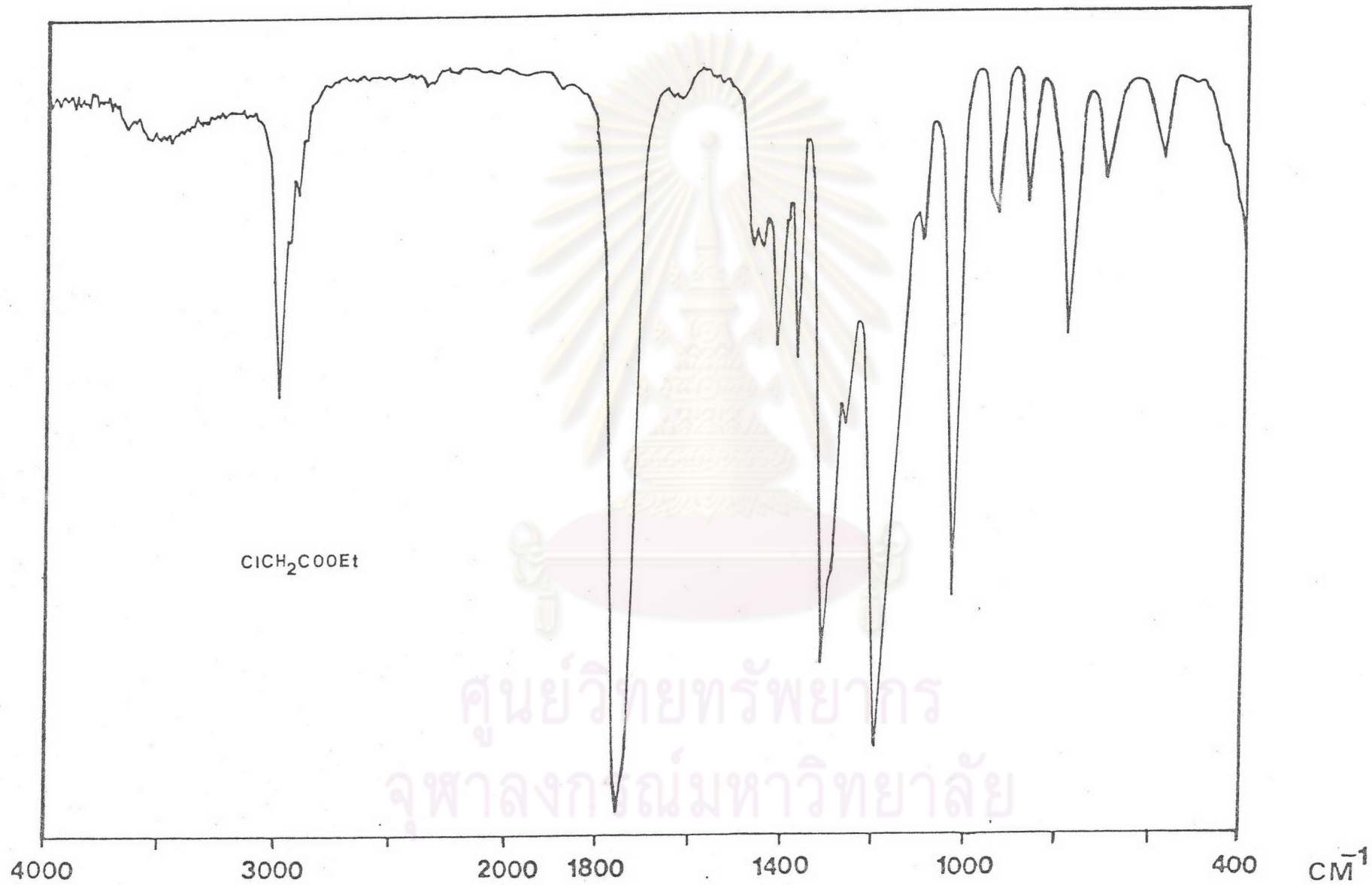


Figure 21. The IR spectrum (Neat) of ethyl chloroacetate

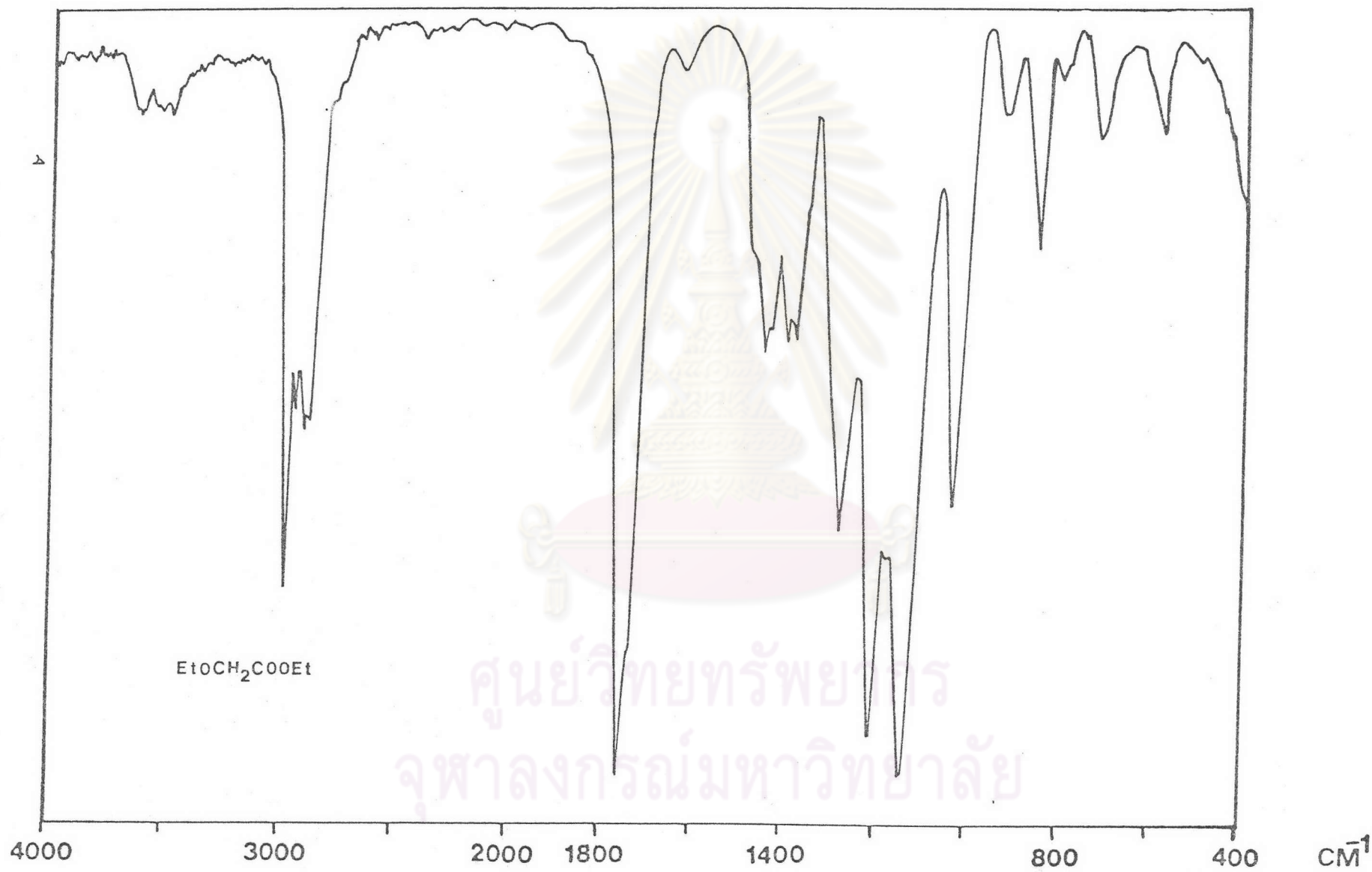


Figure 22. The IR spectrum (Neat) of ethyl ethoxyacetate



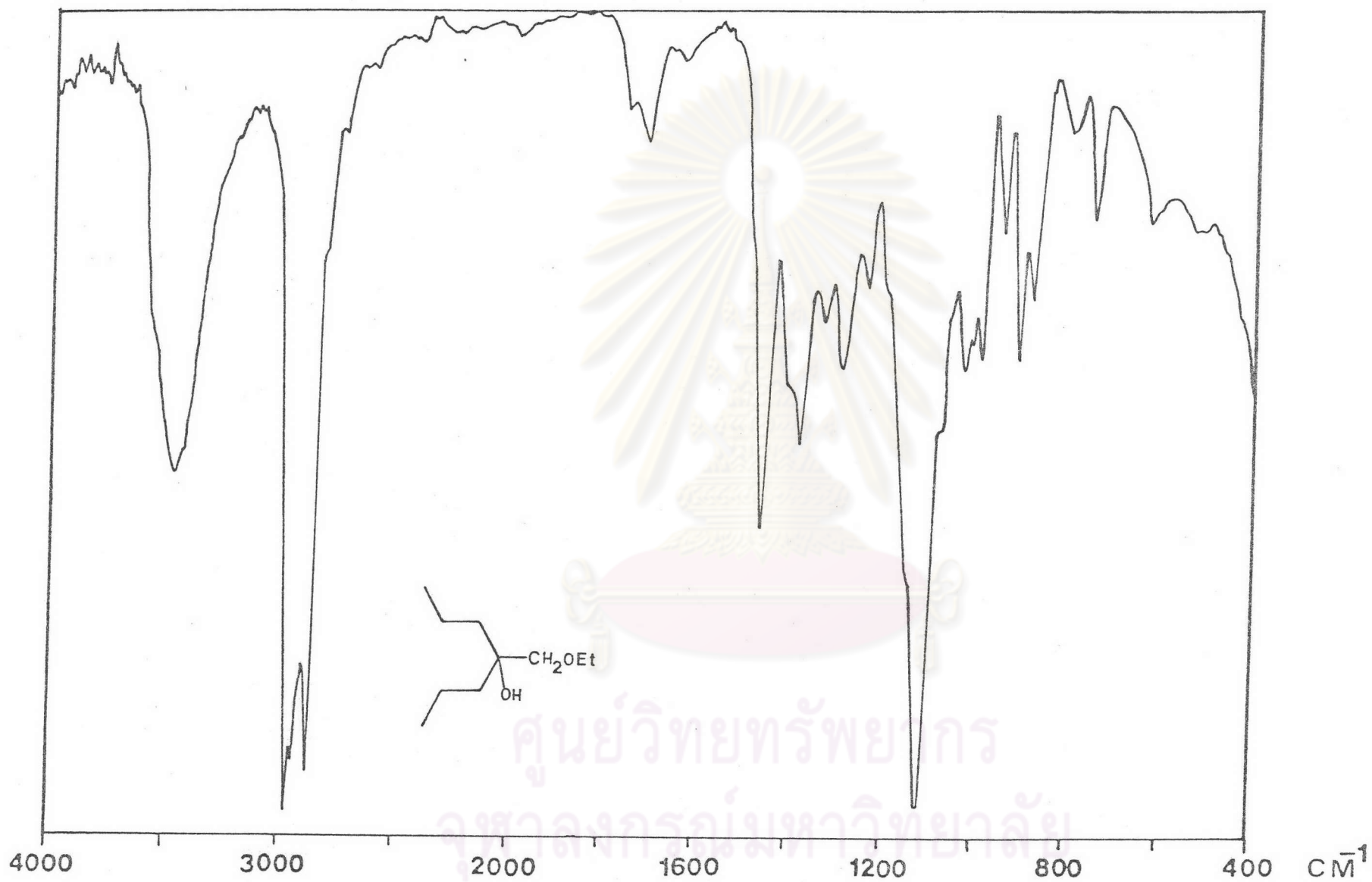


Figure 23. The IR spectrum (Neat) of crude 1,1-dipropyl-2-ethoxyethanol

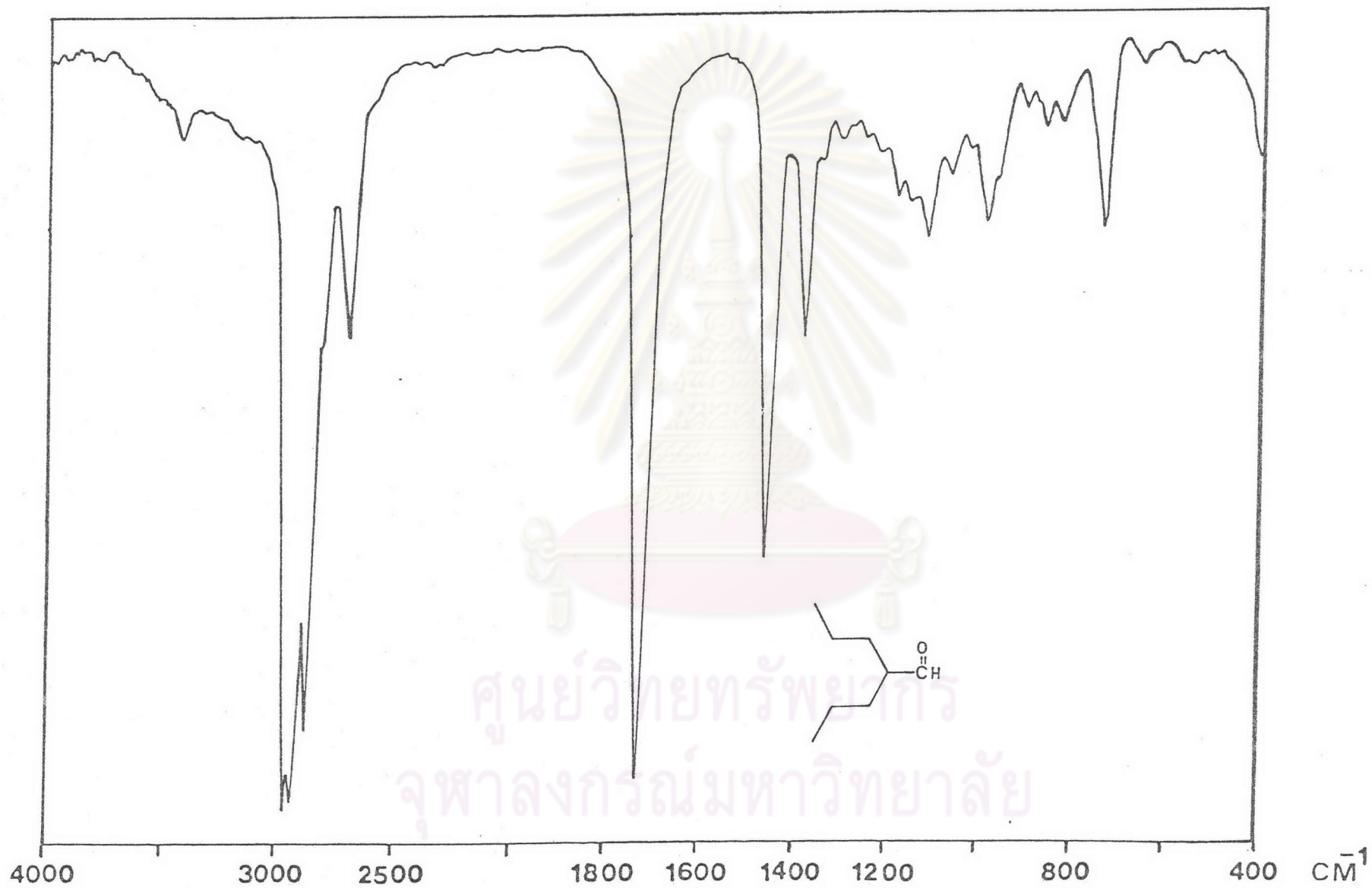
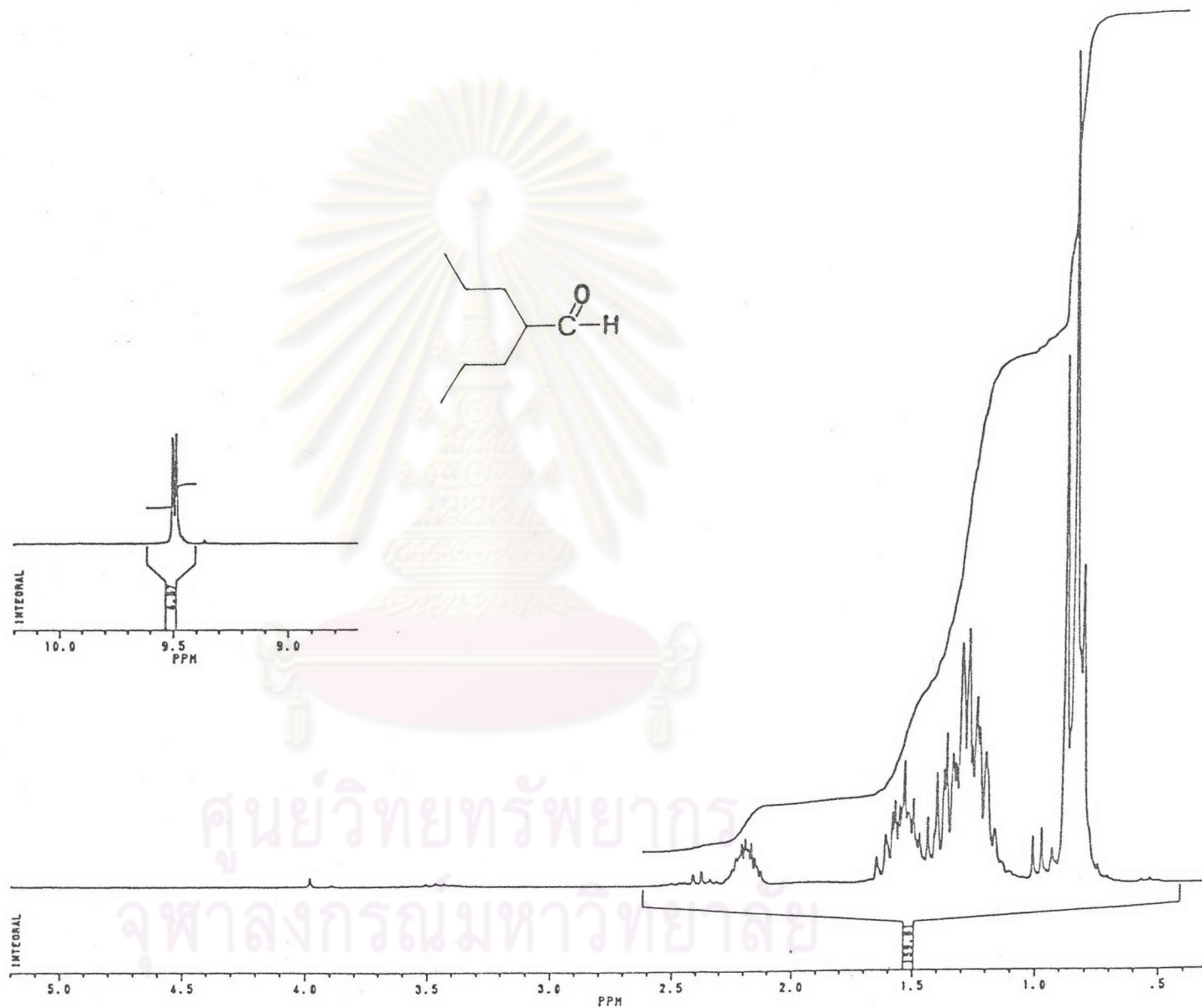


Figure 24 The IR spectrum (Neat) of 2-propylpentanal



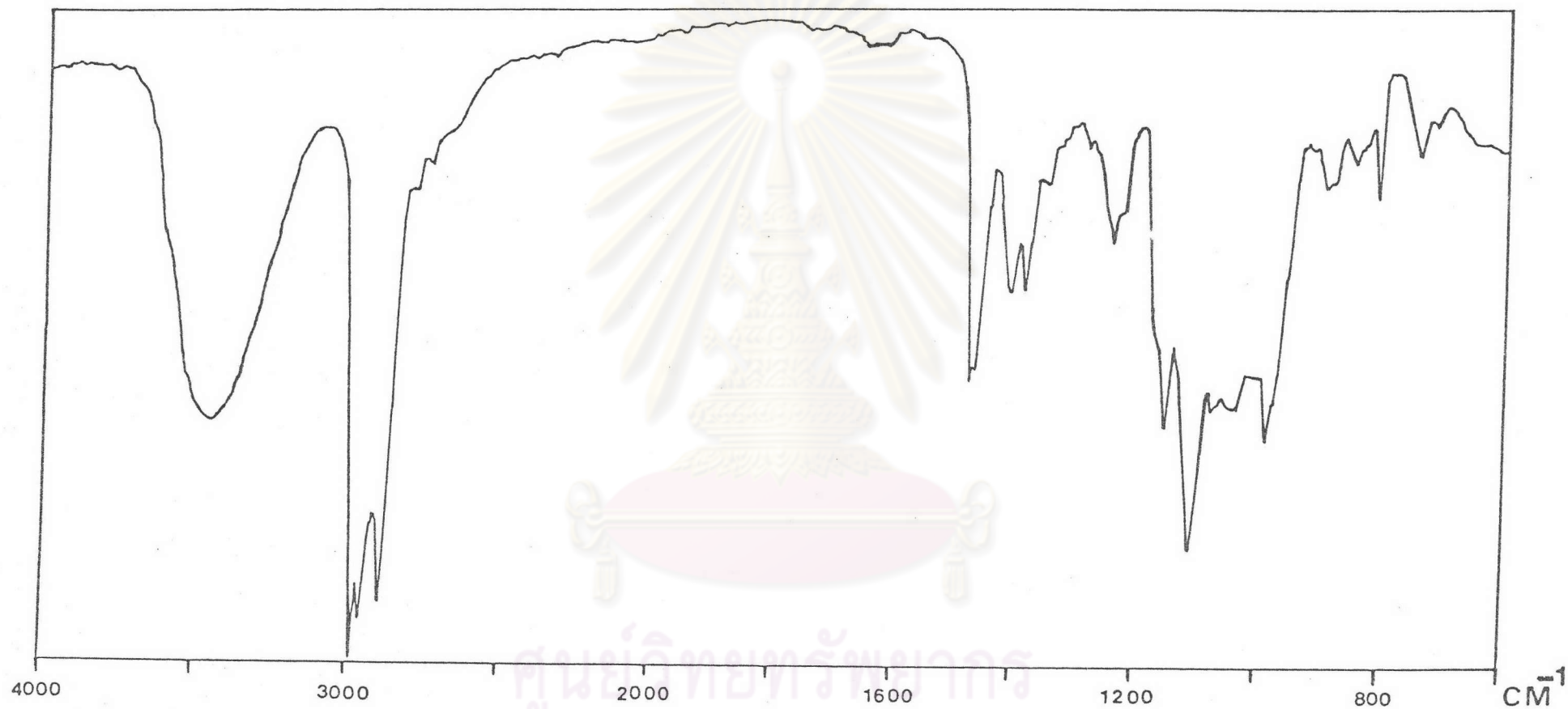
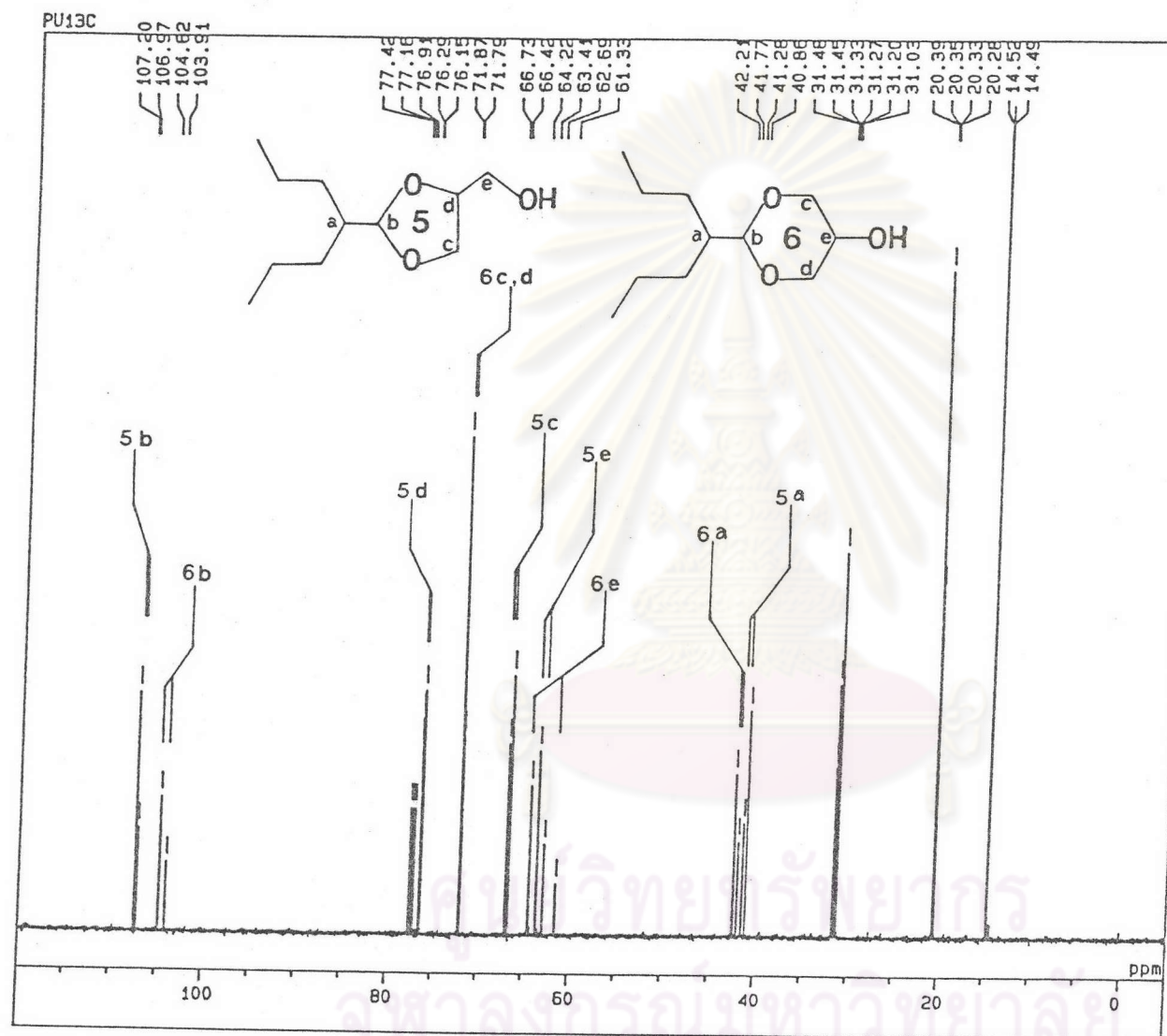


Figure 26. The IR spectrum (Neat) of the product from the condensation of glycerol and 2-propylpentanal.





JEOL

7-SEP-1993 16: 18: 08.29

DFILE : ALPHA  
SFILE : WPU13C

COMNT : PU13C  
EXMOD : SINGL  
IRMOD : BCM  
POINT : 32768  
FREQU : 33898.31 Hz  
SCANS : 128  
DUMMY : 4  
ACQTM : 0.9667 sec  
PD : 1.5000 sec  
RGAIN : 23

PW1 : 4.65 usec

OBNUC : 13C  
OBFRQ : 125.65 MHz  
OBSET : 127958.00 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162663.30 Hz  
IRATN : 200  
IRRPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRRNS : 0

ADBIT : 16  
CTEMP : 27.0 c  
CSPED : 0 Hz  
SLVNT : CDCL3

RESOL : 1.03 Hz  
BF : 0.01 Hz  
T1 : 0.00 %  
T2 : 0.00 %  
T3 : 90.00 %  
T4 : 100.00 %  
REFVL : 0.00 ppm  
XE : 15719.14 Hz  
XS : 5429.54 Hz  
operator

Figure 27. The C-13 decoupled spectrum of the product from the condensation of glycerol and 2-propylpentanal.

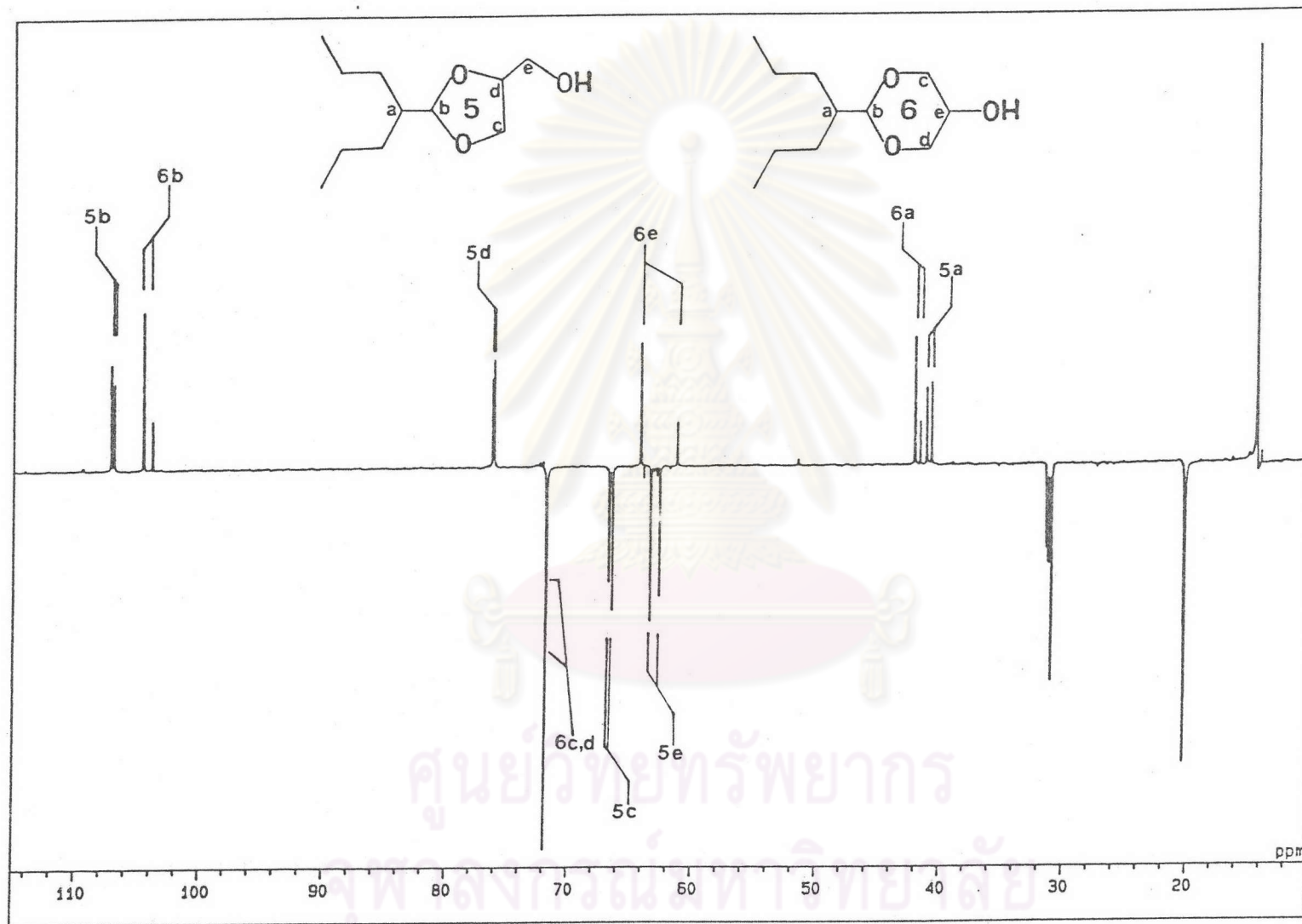


Figure 28. The DEPT-135 spectrum of the product from the condensation of glycerol and 2-propylpentanal

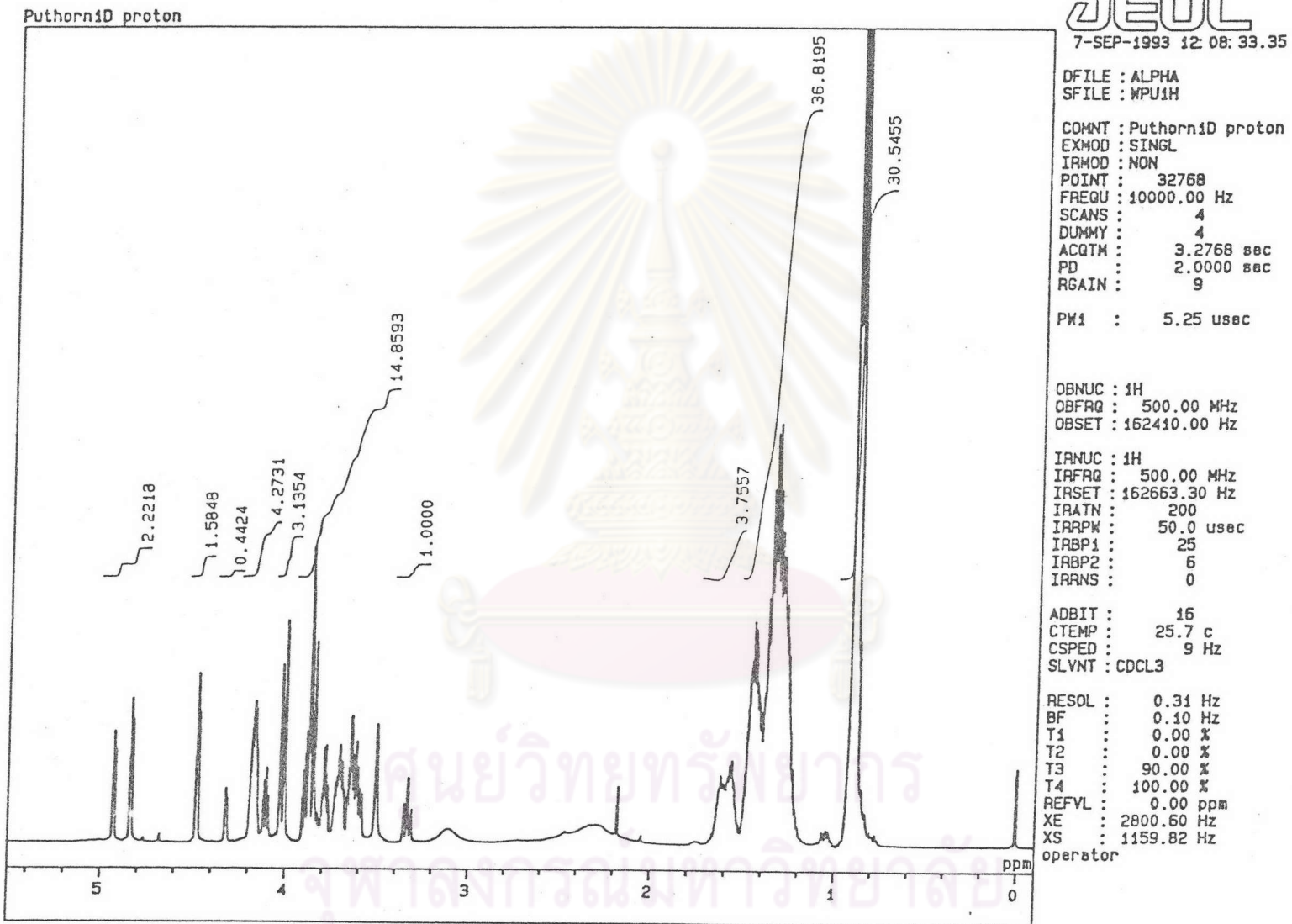
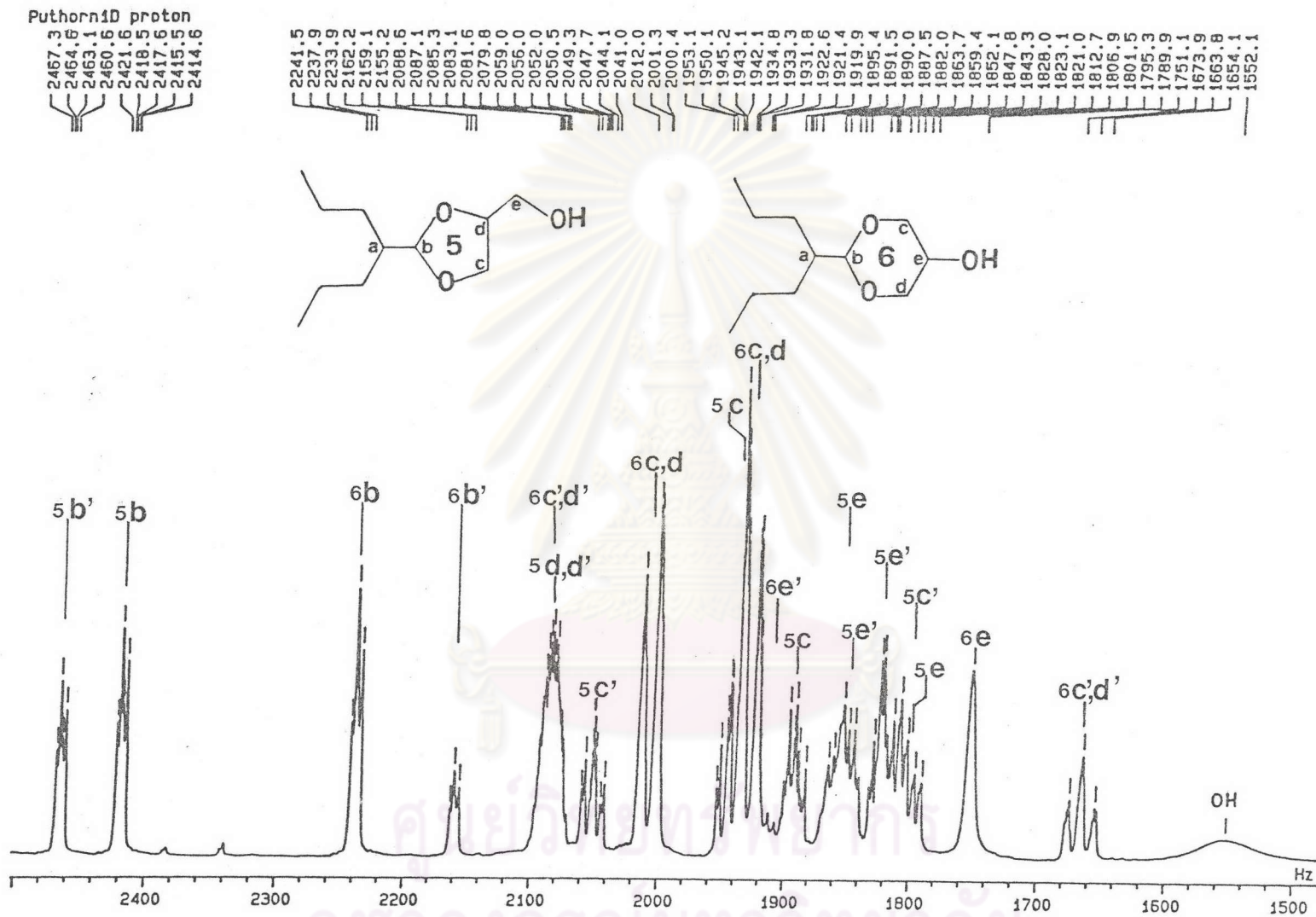


Figure 29. The H-1 spectrum of the product from the condensation of glycerol and 2-propylpentanal





PUTHORN HH COSY

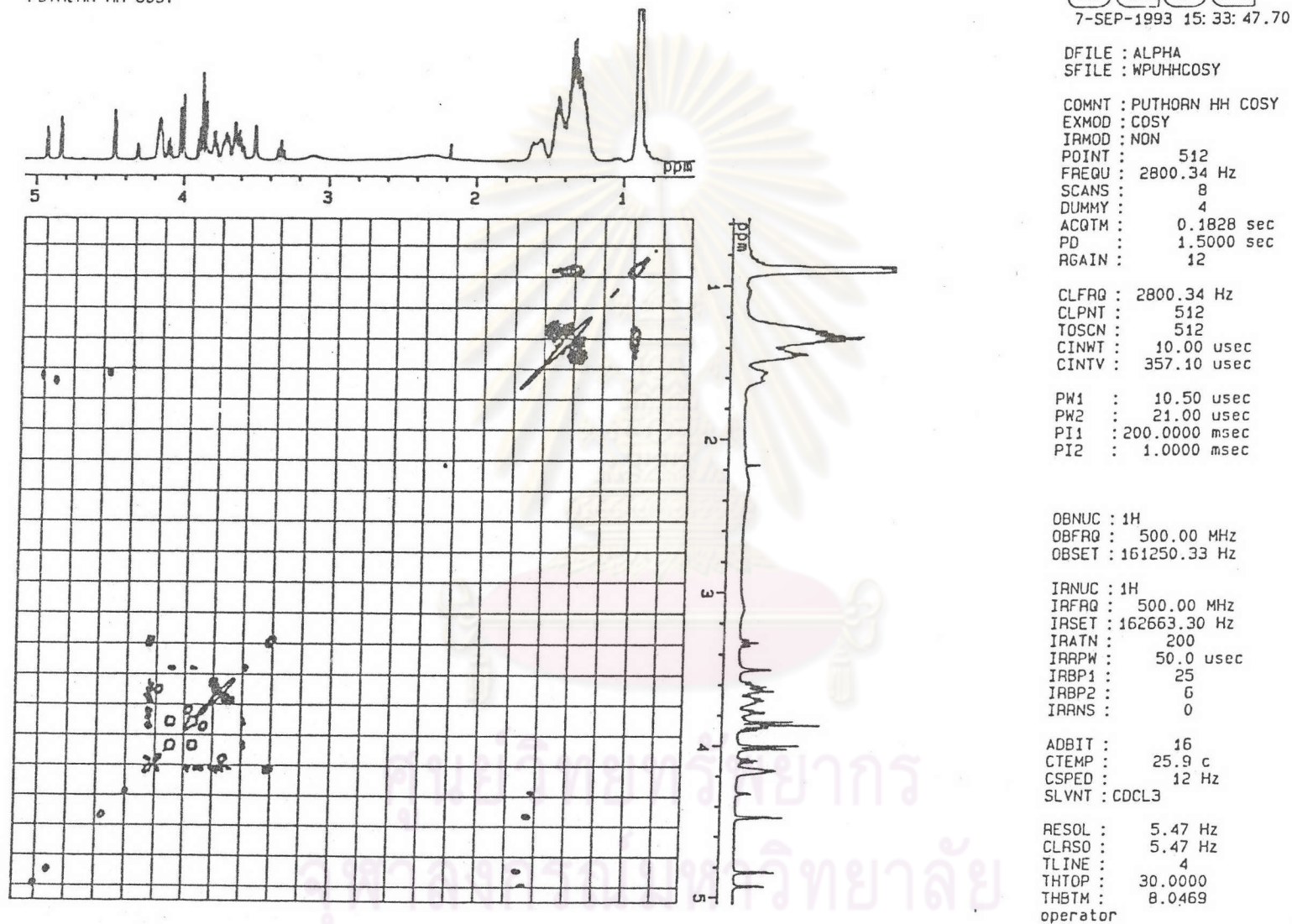


Figure 31. The COSY spectrum of the product from the condensation of glycerol and 2-propylpentanal

# JEOL

7-SEP-1993 15: 25: 34.52

DFILE : ALPHA  
SFILE : WPUHHCOSY

COMNT : PUTHORN HH COSY  
EXMOD : COSY  
IRMOD : NON  
POINT : 512  
FREQU : 2800.34 Hz  
SCANS : 8  
DUMMY : 4  
ACQTM : 0.1828 sec  
PD : 1.5000 sec  
RGAIN : 12

CLFRQ : 2800.34 Hz  
CLPNT : 512  
TOSC : 512  
CINWT : 10.00 usec  
CINTV : 357.10 usec

PW1 : 10.50 usec  
PW2 : 21.00 usec  
PI1 : 200.0000 msec  
PI2 : 1.0000 msec

OBNUC : 1H  
OBFRQ : 500.00 MHz  
OBSET : 161250.33 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162663.30 Hz  
IRATN : 200  
IRAPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRRNS : 0

ADBIT : 16  
CTEMP : 25.9 c  
CSPED : 12 Hz  
SLVNT : CDCL3

RESOL : 5.47 Hz  
CLASO : 5.47 Hz  
TLINE : 4  
THTOP : 30.0000  
THBTM : 8.0469  
operator

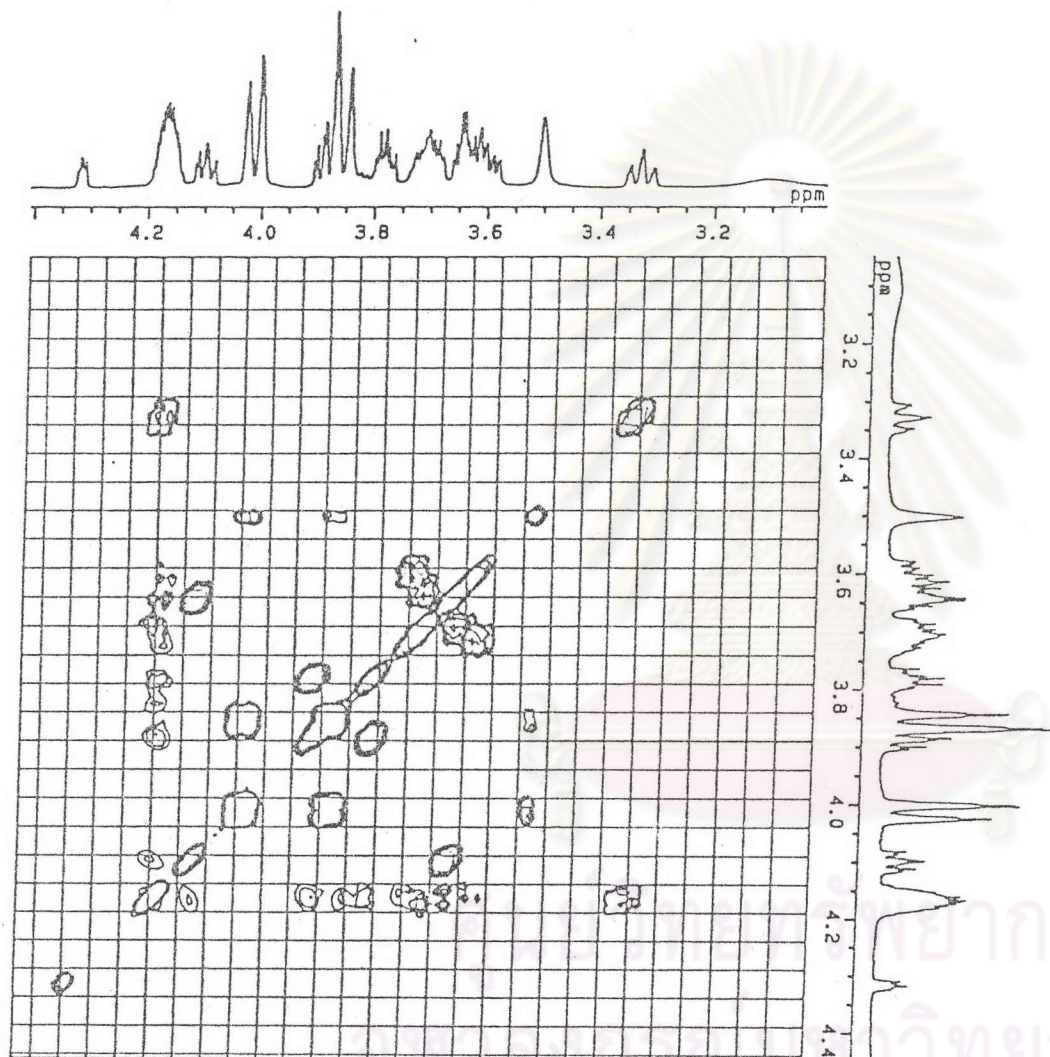
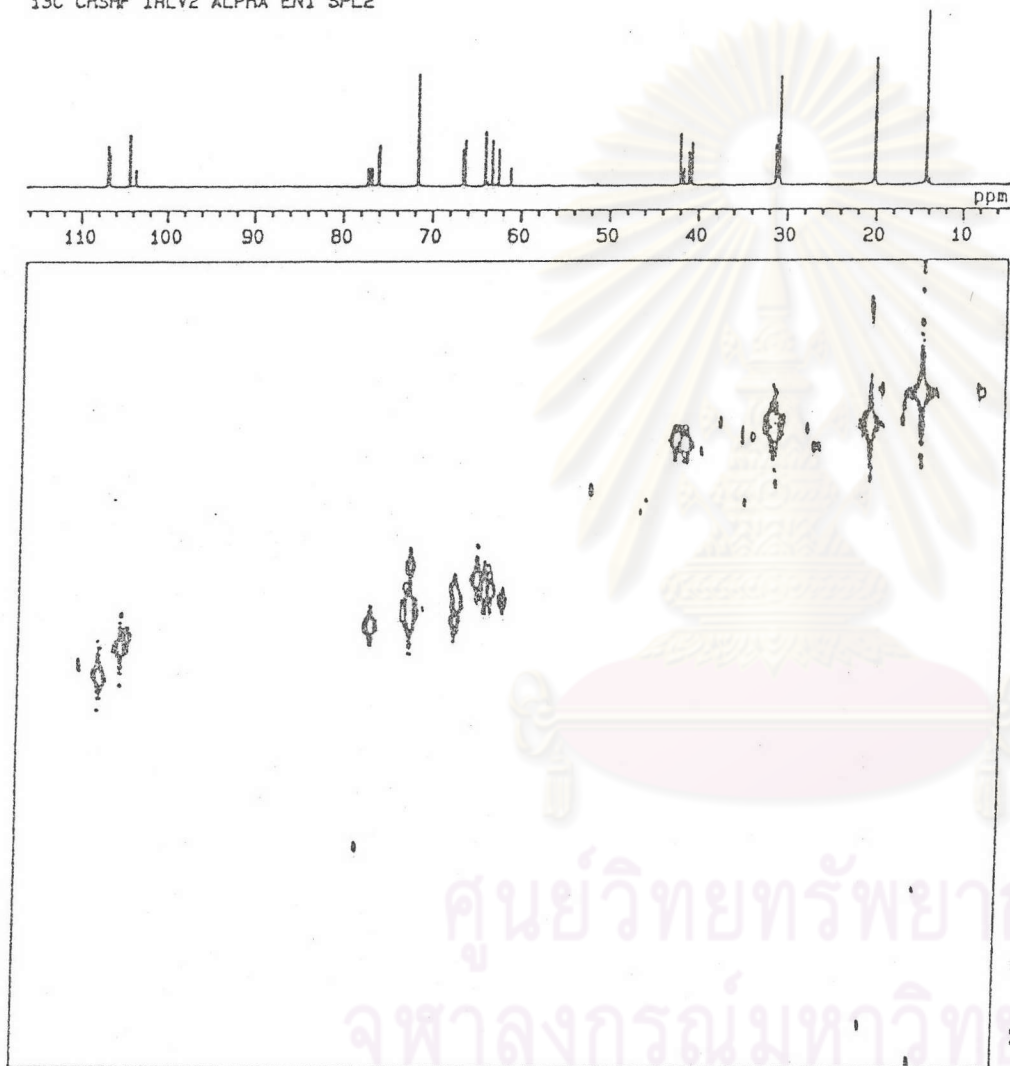


Figure 32. The COSY spectrum of the product from  
the condensation of glycerol and  
2-propylpentanal

13C CHSHF IRLV2 ALPHA EN1 SPL2



JEOL

13-SEP-1993 19:54:14.52

DFILE : ALPHA2CHSHF\_E1S2  
SFILE : ALPHA400

COMNT : 13C CHSHF IRLV2 ALPHA EN:  
EXMOD : CHSHF  
IRMOD : IRLV2  
POINT : 1024  
FREQU : 14044.94 Hz  
SCANS : 64  
DUMMY : 4  
ACQTH : 0.0365 sec  
PD : 1.4635 sec  
RGAIN : 20

CLFRQ : 5733.94 Hz  
CLPNT : 256  
TOSCN : 128  
CINWT : 10.00 usec  
CINT2 : 87.20 usec

PW1 : 9.30 usec  
PW3 : 10.50 usec  
PI1 : 120.0000 msec  
PI3 : 5.6930 msec  
JCNST : 145.00 Hz

OBNUC : 13C  
OBFRQ : 125.65 MHz  
OBSET : 122925.70 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162276.03 Hz  
IRATN : 511  
IRAPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRANS : 0

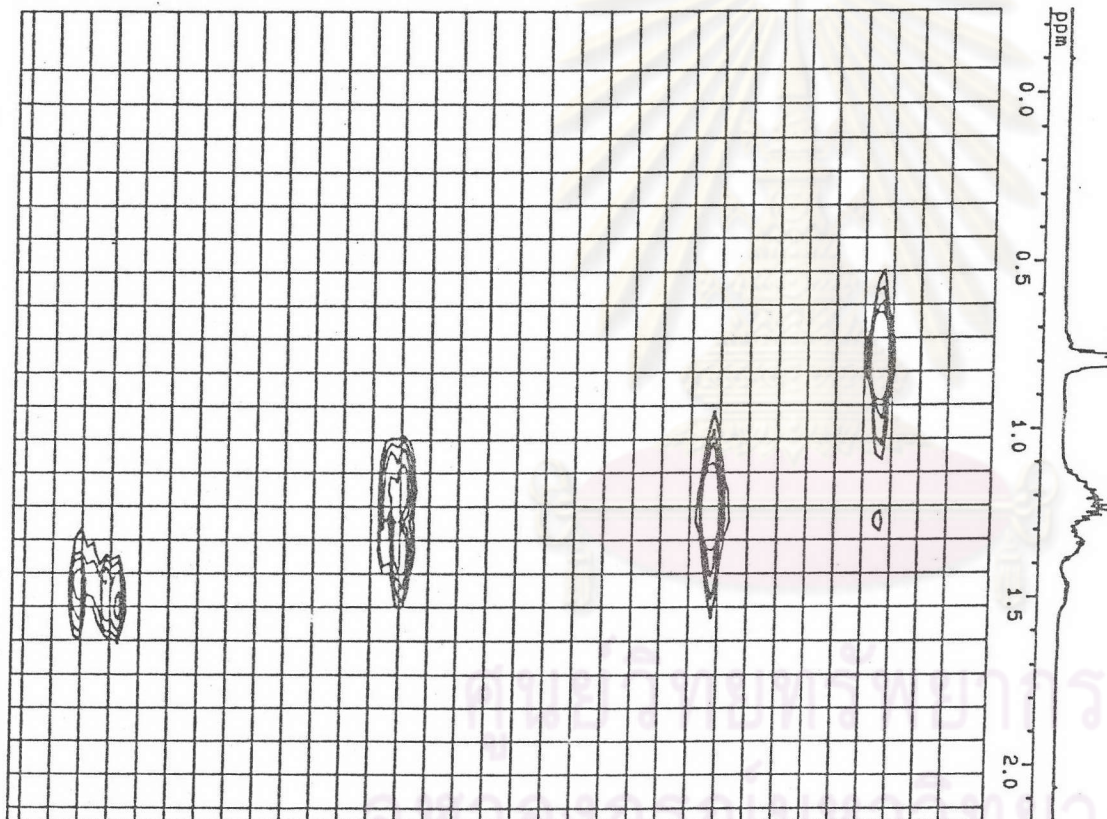
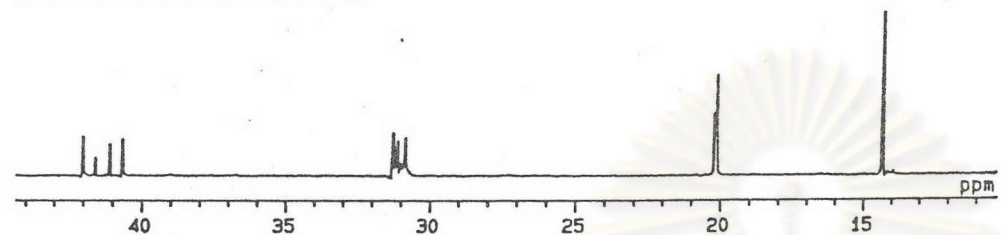
ADBIT : 16  
CTEMP : 25.9 c  
CSPED : 13 Hz  
SLVNT : CDCL3

RESOL : 13.72 Hz  
CLASO : 22.40 Hz  
TLINE : 4  
THTOP : 11.9042  
THBTM : 0.9696  
operator

Figure 33. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal



13C CHSHF IRLV2 ALPHA EN1 SPL2



JEOL

30-SEP-1993 09:39:41.53

DFILE : ALPHA2CHSHF\_E1S2  
SFILE : ALPHA1D135 E1S2

COMNT : 13C CHSHF IRLV2 ALPHA EN:  
EXMOD : CHSHF  
IRMOD : IRLV2

POINT : 512  
FREQU : 14044.94 Hz  
SCANS : 64  
DUMMY : 4  
ACQTM : 0.0365 sec  
PD : 1.4635 sec  
RGAIN : 20

CLFRQ : 5733.94 Hz  
CLPNT : 128  
TOSCN : 128  
CINWT : 10.00 usec  
CINT2 : 87.20 usec

PW1 : 9.30 usec  
PW3 : 10.50 usec  
PI1 : 120.0000 msec  
PI3 : 5.6930 msec  
JCNST : 145.00 Hz

OBNUC : 13C  
OBFRO : 125.65 MHz  
OBSET : 122925.70 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162276.03 Hz  
IRATN : 511  
IRRPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRRNS : 0

ADBIT : 16  
CTEMP : 25.9 c  
CSPED : 13 Hz  
SLVNT : CDCL3

RESOL : 27.43 Hz  
CLHSO : 44.80 Hz  
TLINE : 4  
THTOP : 30.0000  
THBTM : 5.3906  
operator

Figure 34. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Contour plot between 10-45 ppm)



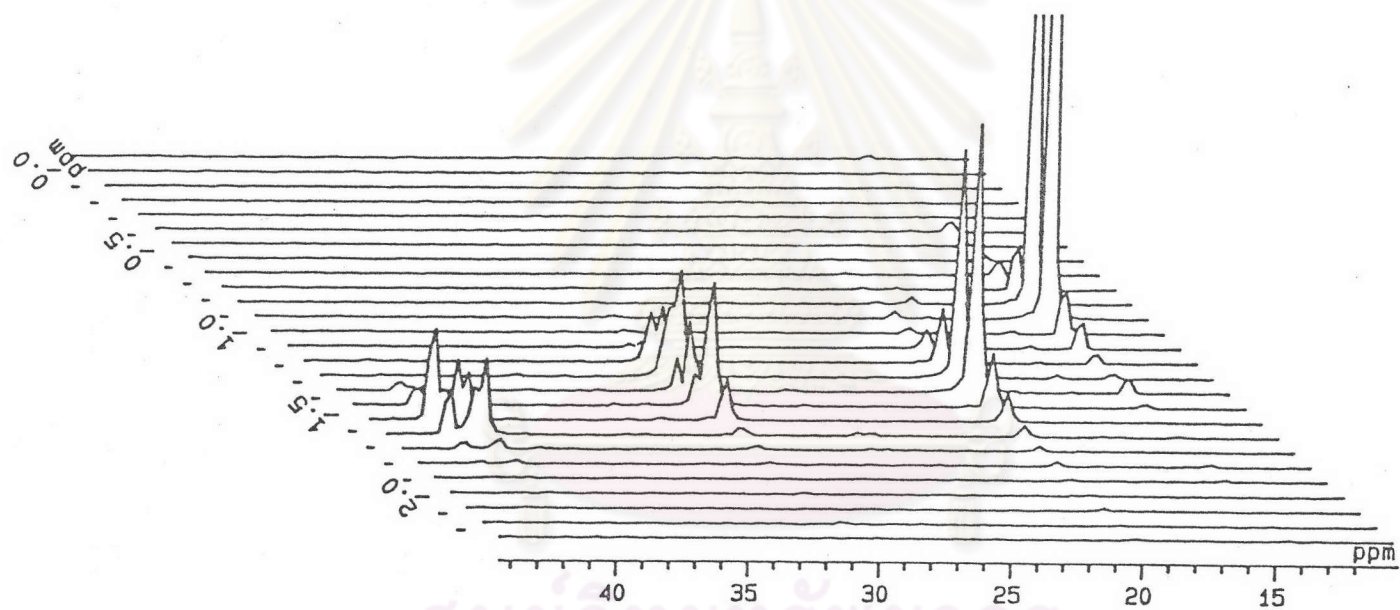
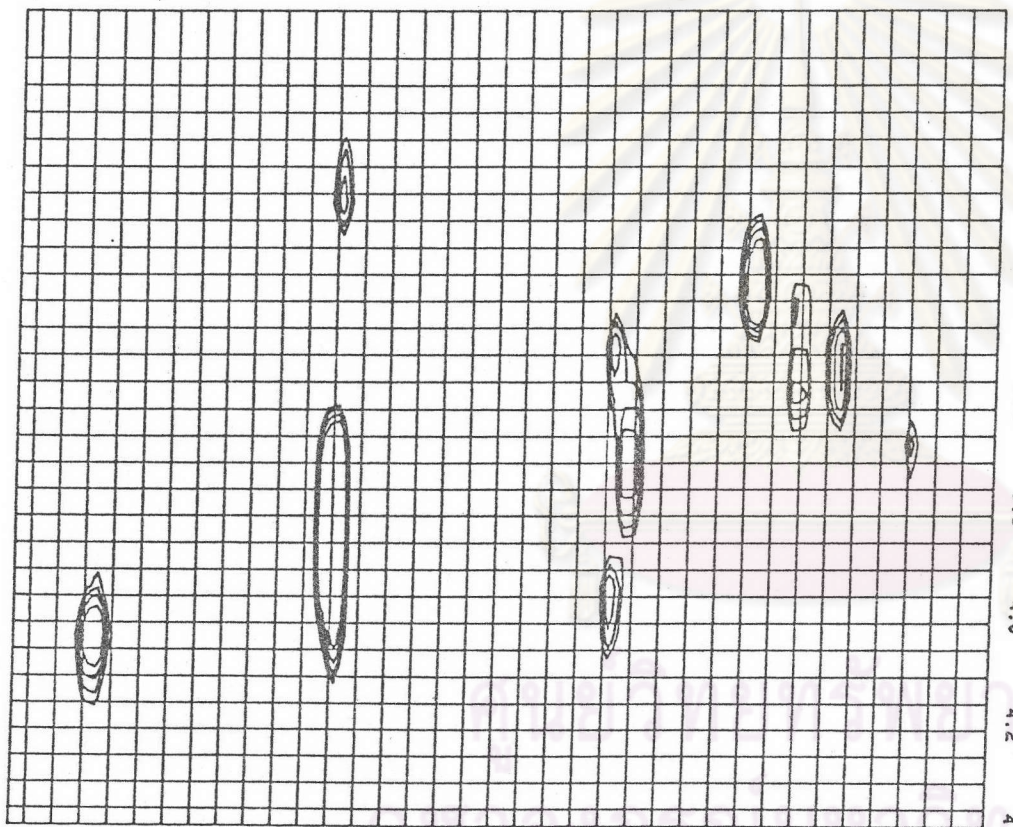
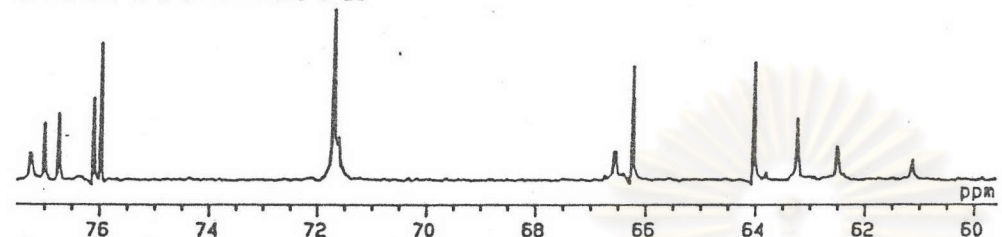


Figure 35. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Stacked plot between 10-45 ppm)

13C CHSHF IRLV2 ALPHA EN1 SPL2



JEOL

30-SEP-1993 09: 56: 26.02

DFILE : ALPHA2CHSHF E1S2  
SFILE : ALPHA1D135 E1S2

COMNT : 13C CHSHF IRLV2 ALPHA EN:

EXMOD : CHSHF  
IRMOD : IRLV2  
POINT : 512  
FREQU : 14044.94 Hz  
SCANS : 64  
DUMMY : 4  
ACQTM : 0.0365 sec  
PD : 1.4635 sec  
RGAIN : 20

CLFRQ : 5733.94 Hz  
CLPNT : 128  
TOSCN : 128  
CINWT : 10.00 usec  
CINT2 : 87.20 usec

PW1 : 9.30 usec  
PW3 : 10.50 usec  
PI1 : 120.0000 msec  
PI3 : 5.6930 msec  
JCNST : 145.00 Hz

OBNUC : 13C  
OBFRQ : 125.65 MHz  
OBSET : 122925.70 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162276.03 Hz  
IRATN : 511  
IRRPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRRNS : 0

ADBIT : 16  
CTEMP : 25.9 c  
CSPED : 13 Hz  
SLVNT : CDCL3

RESOL : 27.43 Hz  
CLASO : 44.80 Hz  
TLINE : 4  
THTOP : 30.0000  
THBTM : 12.1094  
operator

Figure 36. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Contour plot between 60-77.5 ppm)

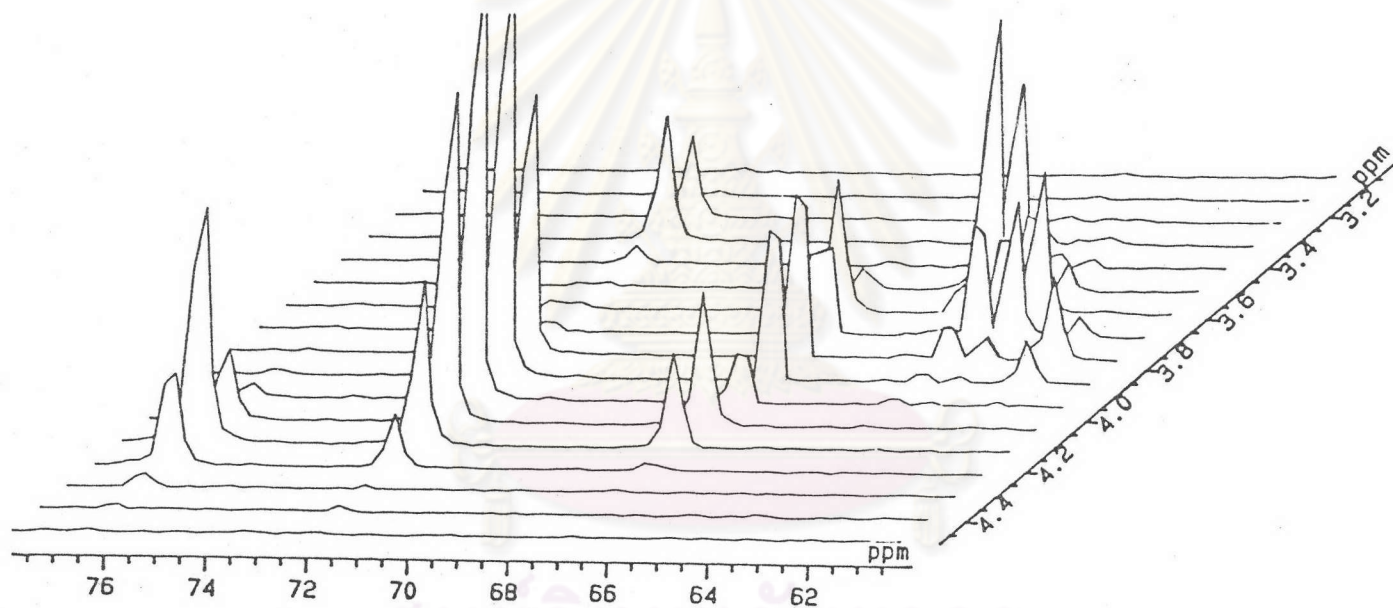
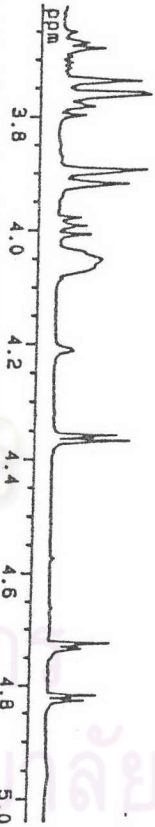
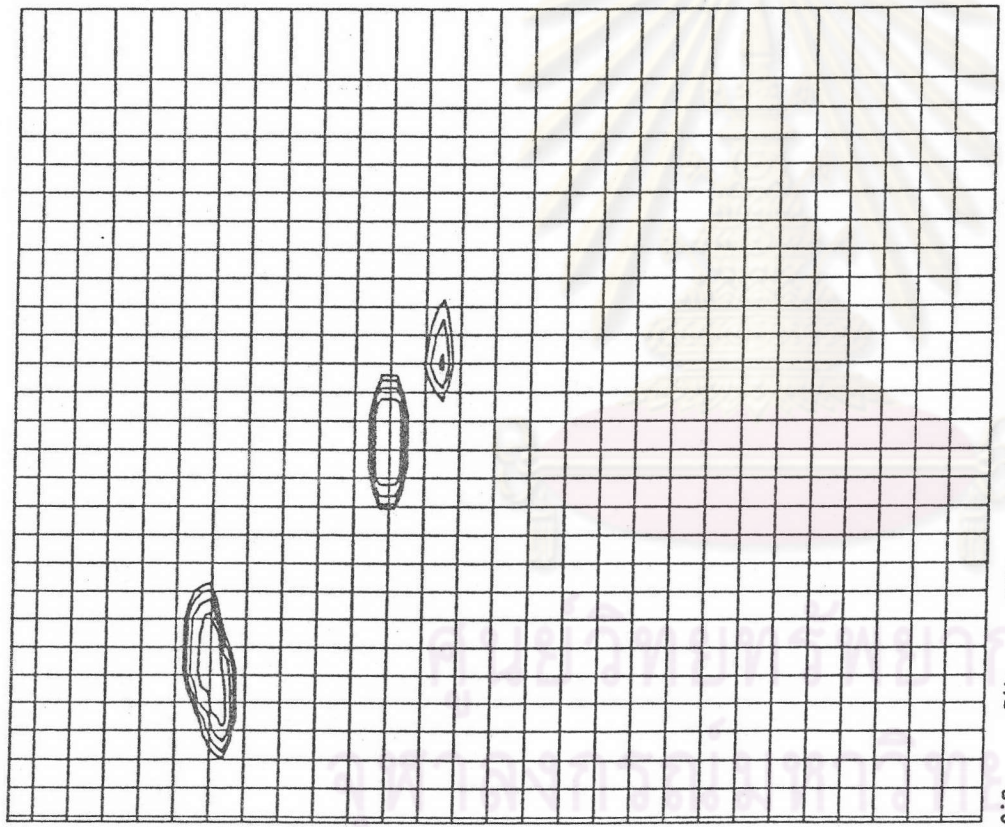
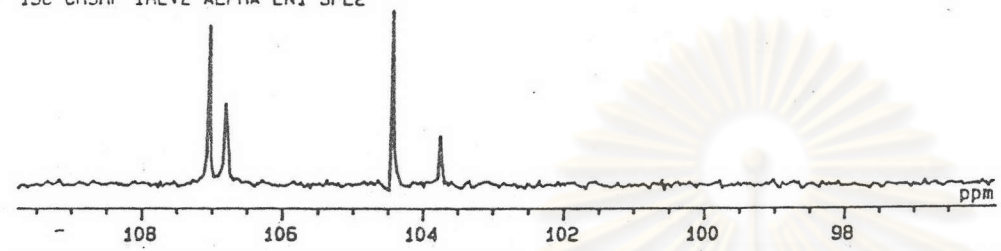


Figure 37. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Stacked plot between 60-77.5 ppm)



13C CHSHF IRLV2 ALPHA EN1 SPL2



**JEOL**  
30-SEP-1993 10:19:30.85

DFILE : ALPHA2CHSHF\_E1S2  
SFILE : ALPHA1D135\_E1S2

COMNT : 13C CHSHF IRLV2 ALPHA EN:  
EXMOD : CHSHF  
IRMOD : IRLV2  
POINT : 512  
FREQU : 14044.94 Hz  
SCANS : 64  
DUMMY : 4  
ACGTM : 0.0365 sec  
PD : 1.4635 sec  
RGAIN : 20

CLFRQ : 5733.94 Hz  
CLPNT : 128  
TOSCN : 128  
CINWT : 10.00 usec  
CINT2 : 87.20 usec

PW1 : 9.30 usec  
PW3 : 10.50 usec  
PI1 : 120.0000 msec  
PI3 : 5.6930 msec  
JCNST : 145.00 Hz

OBNUC : 13C  
OBFRO : 125.65 MHz  
OBSET : 122925.70 Hz

IRNUC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162276.03 Hz  
IRATN : 511  
IRAPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRANS : 0

ADBIT : 16  
CTEMP : 25.9 c  
CSPED : 13 Hz  
SLVNT : CDCL3

RESOL : 27.43 Hz  
CLASO : 44.80 Hz  
TLINE : 4  
THTOP : 30.0000  
THBTM : 9.9219  
operator

Figure 38. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Contour plot between 96-110 ppm)



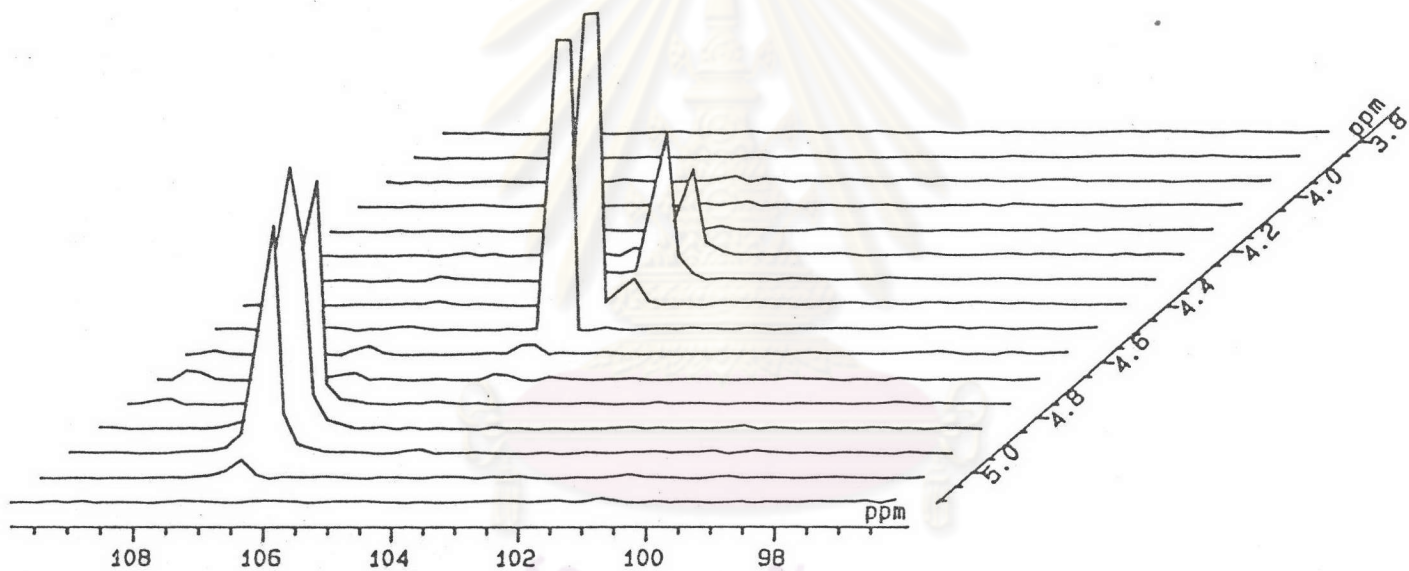


Figure 39. The HETCOR spectrum of the product from the condensation of glycerol and 2-propylpentanal (Stacked plot between 96-110 ppm)

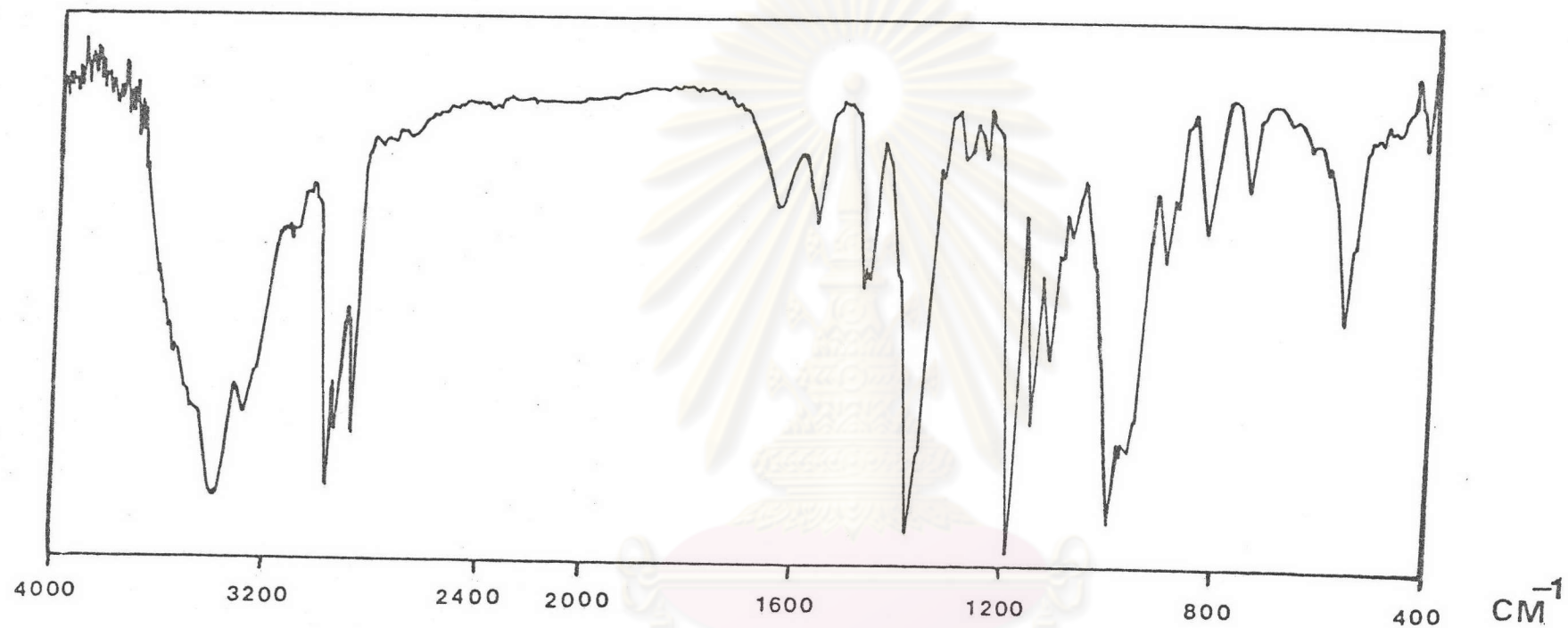


Figure 40. The IR spectrum (KBr disc) of the sulfamate derivative of the product from the condensation of glycerol and 2-propylpentanal

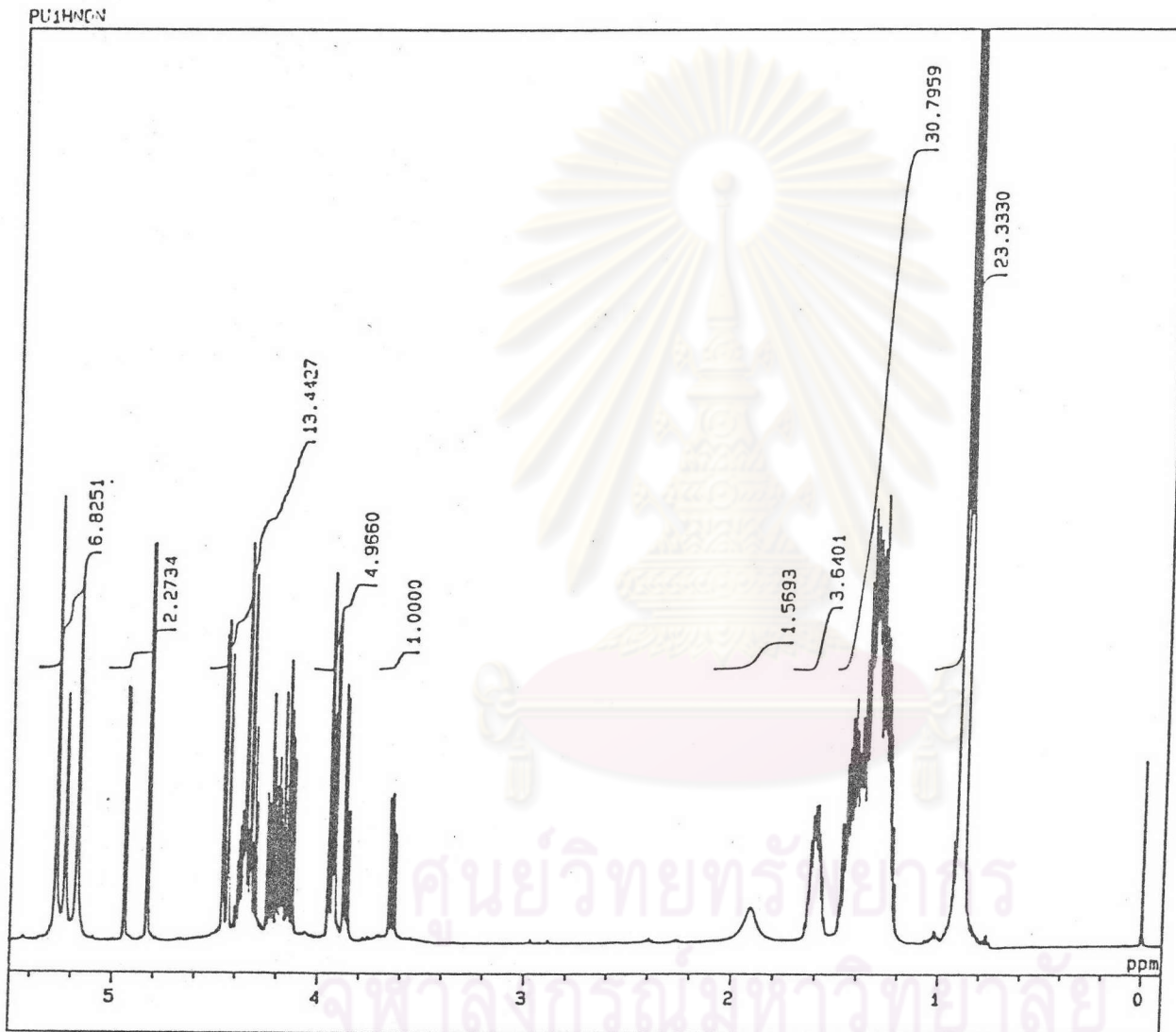


Figure 41. The H-1 spectrum of the sulfamate derivative

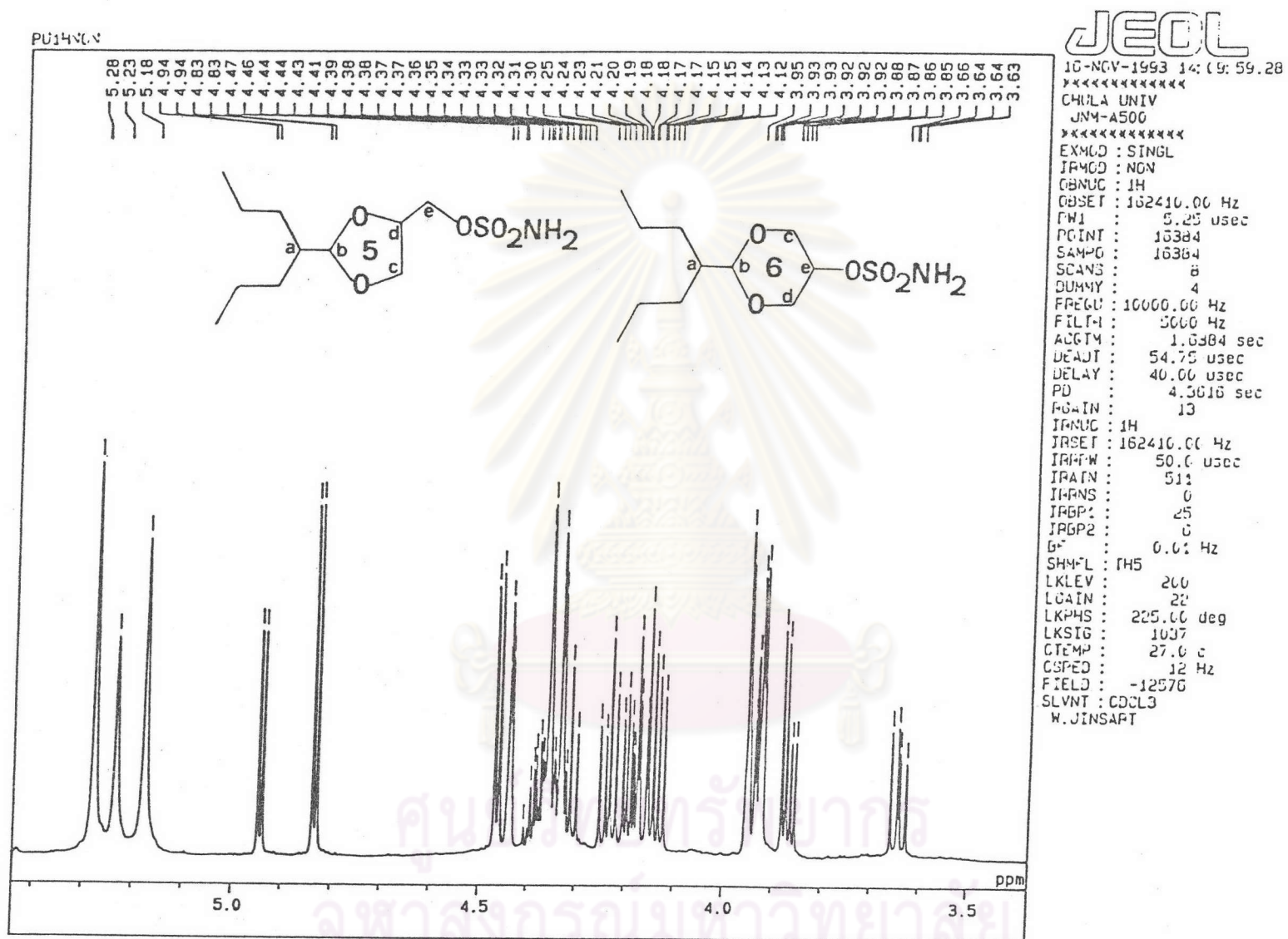


Figure 42. A. The H-1 spectrum of the sulfamate derivative  
 (Enlarged scale)



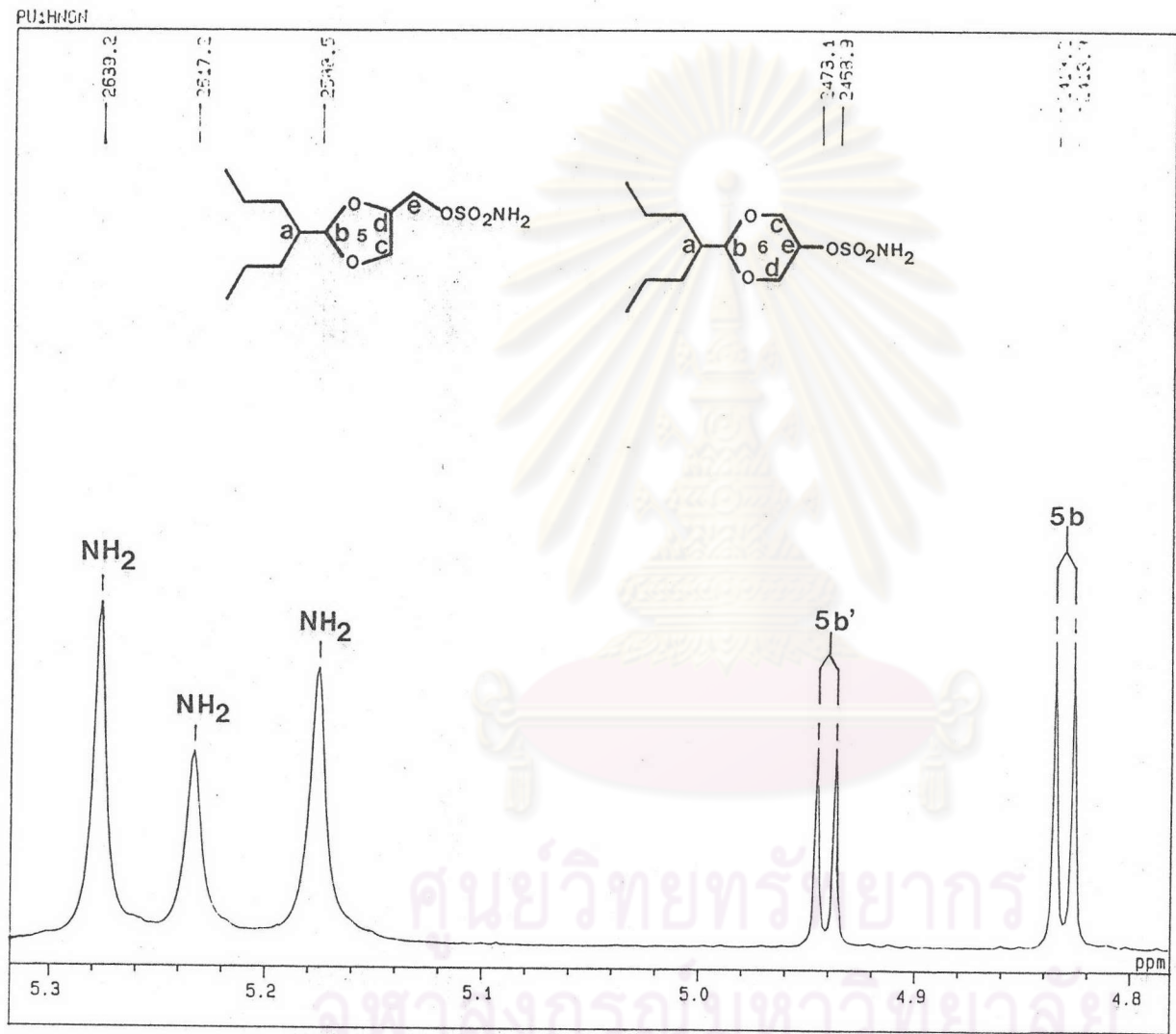


Figure 42. B. The H-1 spectrum of the sulfamate derivative  
 (Enlarged scale : 4.8-5.3 ppm)



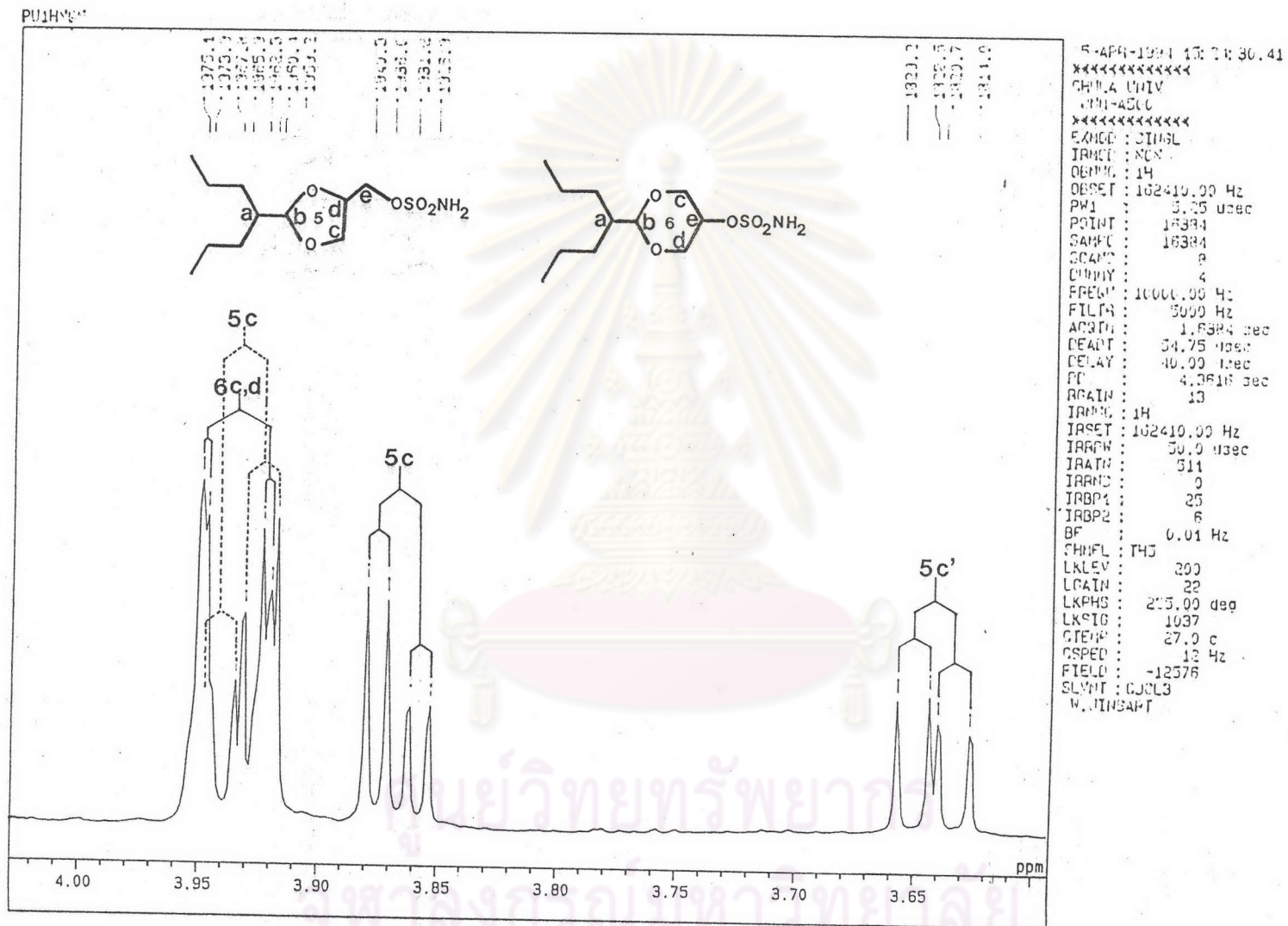


Figure 42. D.The H-1 spectrum of the sulfamate derivative  
(Enlarged scale :3.6-4.00 ppm)

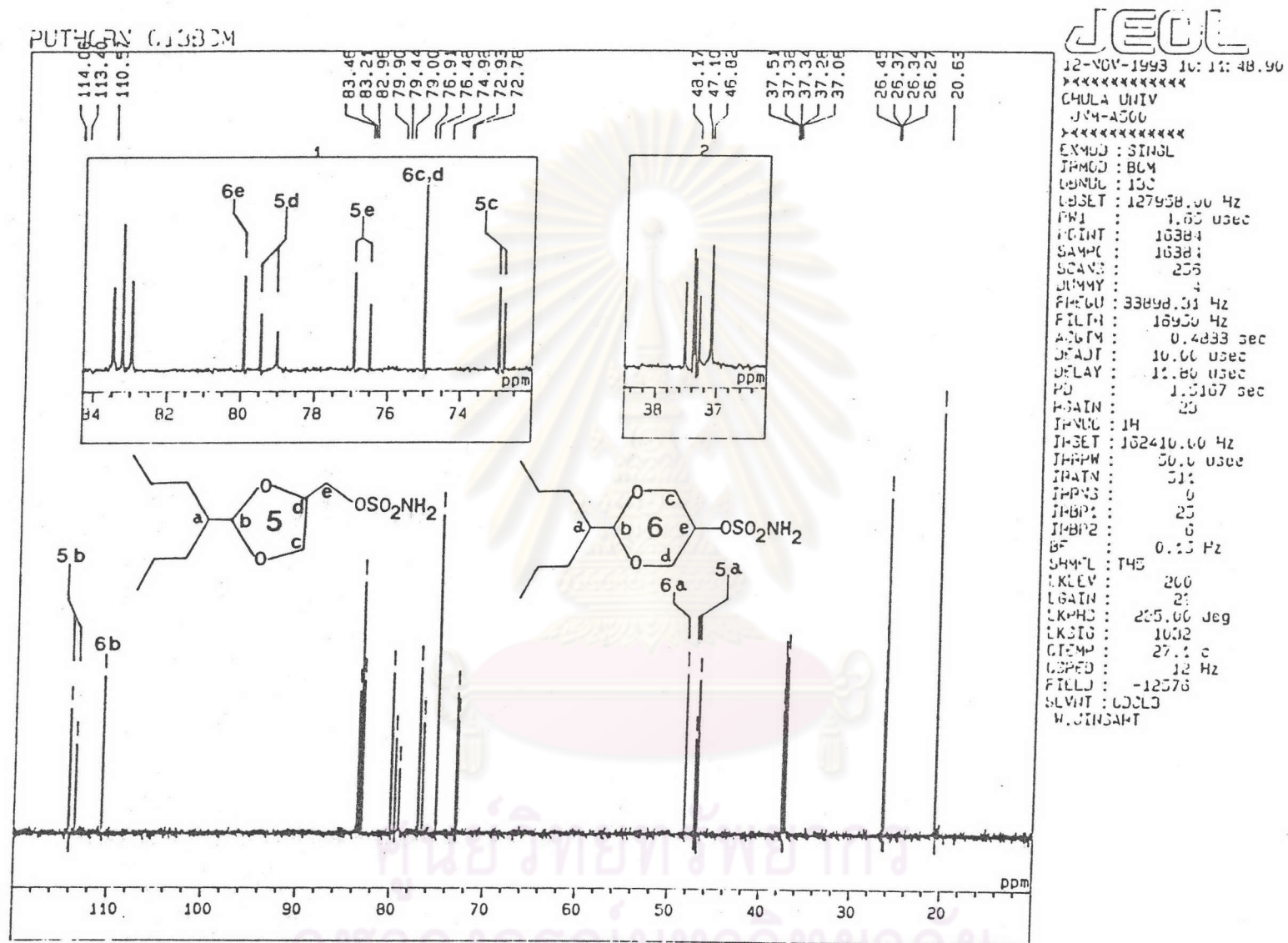
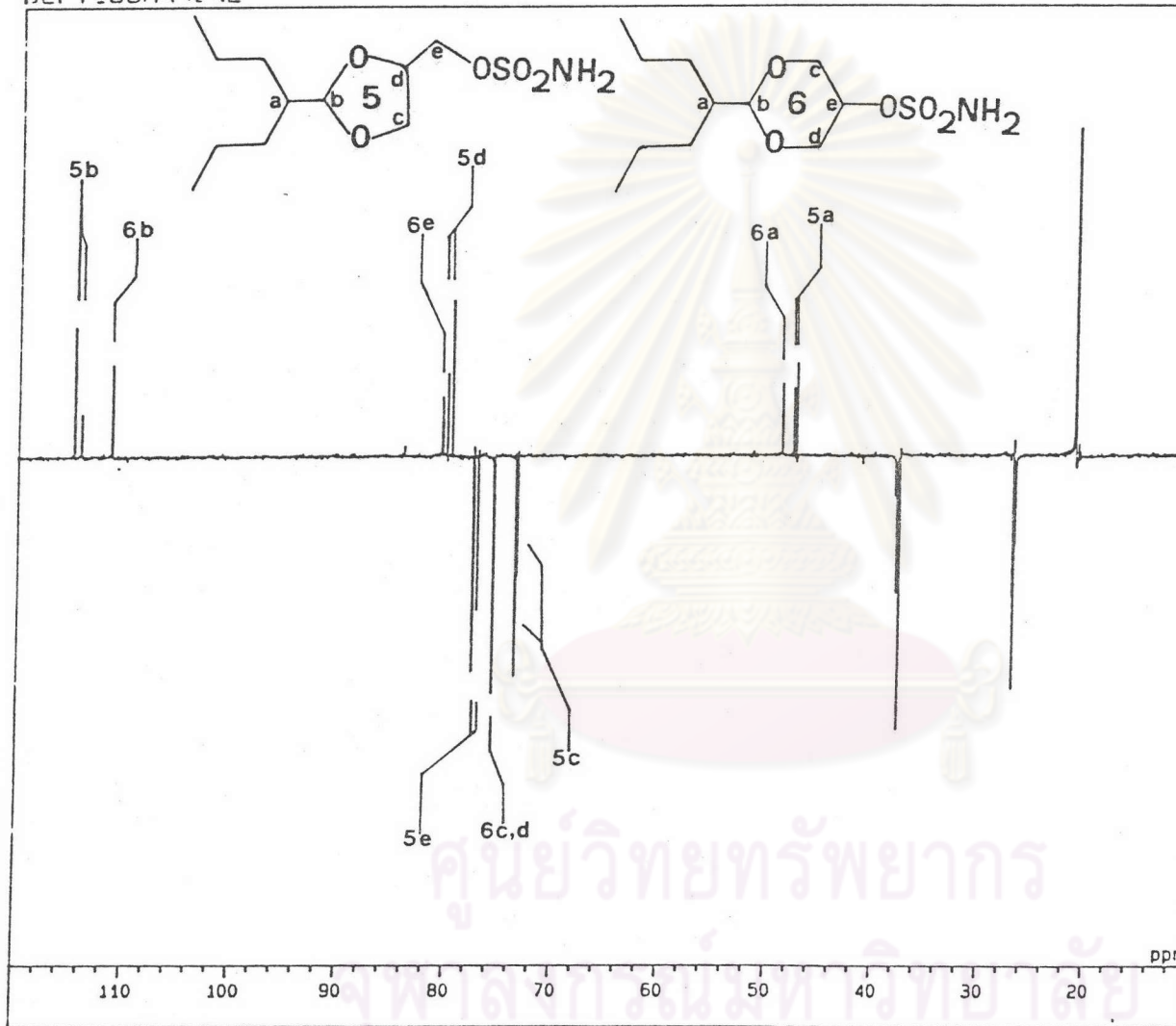


Figure 43. The C-13 decoupled spectrum of the sulfamate derivative



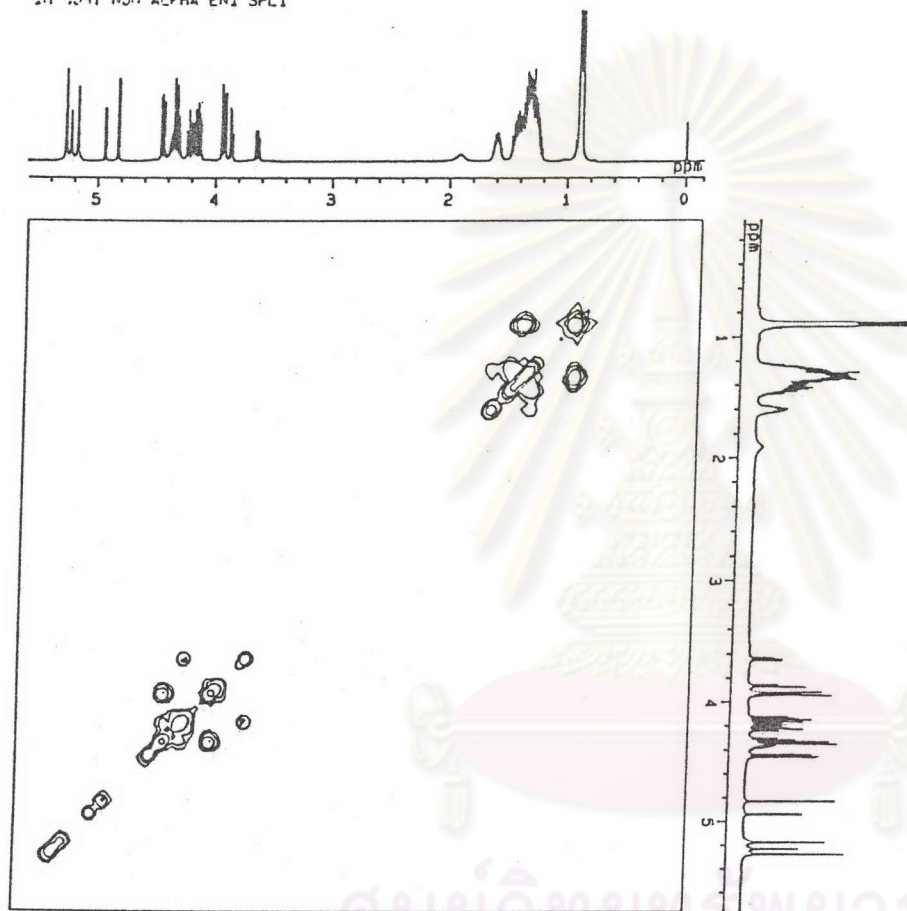
DEPT-135 MANUAL



**JEOL**  
12-NOV-1993 1: 24: 20.10  
\*\*\*\*\*  
CHULA UNIV  
JNM-A500  
\*\*\*\*\*  
EXHDJ : DEPTD  
FPCD : IPLV2  
GPNOC : 13C  
GSDJ : 127958.00 Hz  
PW1 : 9.30 usec  
PPOINT : 16381  
SAMPD : 16381  
SCAND : 1024  
DUMMY : 1  
FREQD : 33098.31 Hz  
FILTR : 16950 Hz  
ACQTM : 0.4833 sec  
DEADT : 10.00 usec  
DELAY : 11.00 usec  
PD : 3.0000 sec  
PSTAT : 23  
IPNOC : 14  
IRSET : 102410.00 Hz  
IRHPW : 50.0 usec  
IRPATN : 120  
IRPHS : 0  
IRBP1 : 25  
IRBP2 : 0  
BF : 0.40 Hz  
SMMFL : TH5  
MKLEV : 200  
LGAIN : 21  
MKPHS : 205.00 deg  
MKSTG : 125  
CTEMP : 25.5 C  
CSPED : 12 Hz  
FIELD : -12554  
GLVNT : C3CL3  
W. JINSANT

Figure 44. The DEPT-135 spectrum of the sulfamate derivative

1H COSY NON ALPHA EN1 SPL1



JEOL  
16-NOV-1993 16:38:12.53

DFILE : ALPHA4COSY.F1S1  
SFILE : ALPHA4COSY.E1S1

COMNT : 1H COSY NON ALPHA EN1 SPL1  
EXMOD : COSY  
JAMOD : NON  
PPOINT : 256  
FREQ1 : 5737.23 Hz  
SCANS : 16  
DUMMY : 4  
ACQTH : 0.0446 sec  
PD : 1.4554 sec  
RGAIN : 10

CLFRQ : 5737.23 Hz  
CLPNT : 256  
TOSCN : 256  
CINWT : 10.00 usec  
CINTV : 174.30 usec

PW1 : 10.50 usec  
PW2 : 21.00 usec  
PI1 : 120.0000 msec  
PI2 : 1.0000 msec

DEVIC : 1H  
DBFRQ : 500.00 MHz  
DBSET : 162304.41 Hz

IRVIC : 1H  
IRFRQ : 500.00 MHz  
IRSET : 162410.00 Hz  
IRATN : 511  
IRAPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRANS : 0

ADBIT : 16  
CTEMP : 27.0 c  
CSPE0 : 13 Hz  
SLVNT : CDCL3

RESOL : 27.41 Hz  
CLAS0 : 22.41 Hz  
TLINE : 4  
THTOP : 50.0000  
THBTM : 1.3281  
operator

Figure 45. The COSY spectrum of the sulfamate derivative  
(Contour plot)

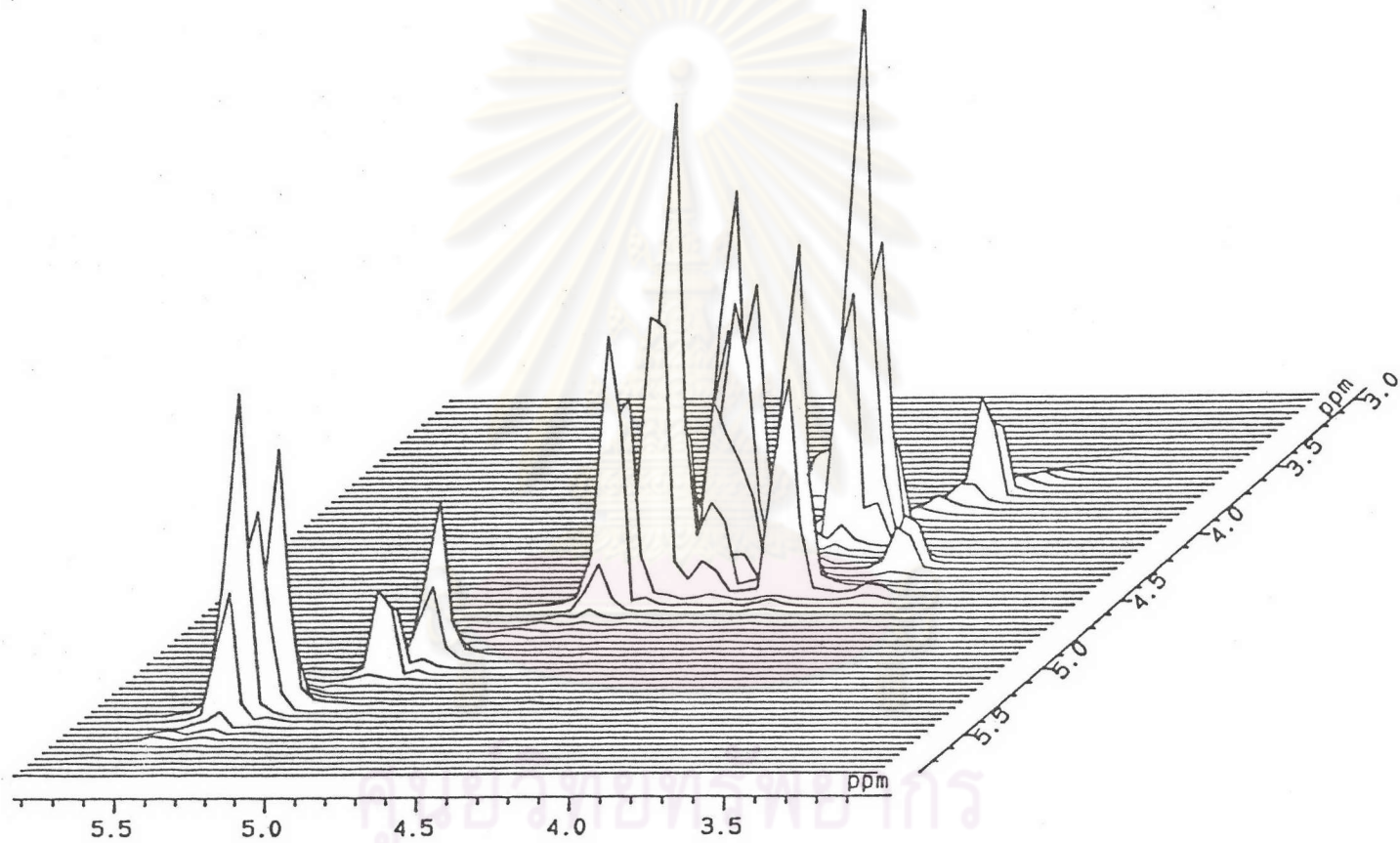
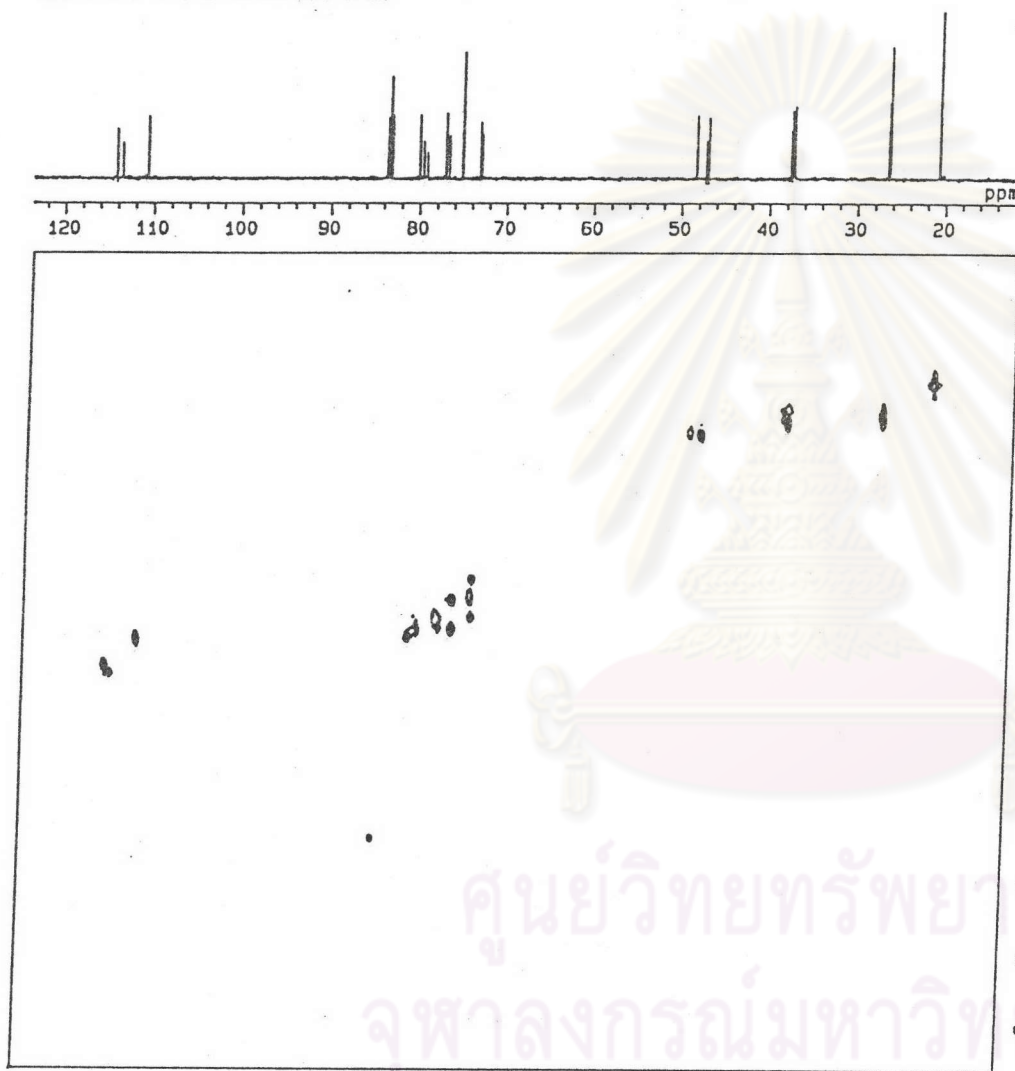


Figure 46. The COSY spectrum of the sulfamate derivative  
(Stacked plot)

<sup>13</sup>C CHSHF IRLV2 ALPHA EN1 SPL1



JEOL

16-NOV-1993 15: 57: 06.06

DFILE : ALPHA5CHSHF\_F1S1  
SFILE : PU-CHSHF

COMNT : <sup>13</sup>C CHSHF IRLV2 ALPHA EN:  
EXMOD : CHSHF  
IRMOD : IRLV2  
POINT : 512  
FREQU : 14104.37 Hz  
SCANS : 64  
DUMMY : 4  
ACQTM : 0.0363 sec  
PD : 1.4637 sec  
RGAIN : 20

CLFRQ : 5737.89 Hz  
CLPNT : 256  
TOSCN : 256  
CINWT : 10.00 usec  
CINT2 : 87.14 usec

PW1 : 9.30 usec  
PW3 : 10.50 usec  
PI1 : 120.0000 msec  
PI3 : 5.6930 msec  
JCNST : 145.00 Hz

OBNUC : <sup>13</sup>C  
OBFRO : 125.65 MHz  
OBSET : 123000.70 Hz

IRNUC : <sup>1</sup>H  
IRFRQ : 500.00 MHz  
IRSET : 162304.41 Hz  
IRATN : 511  
IRRPW : 50.0 usec  
IRBP1 : 25  
IRBP2 : 6  
IRRNS : 0

ADBIT : 16  
CTEMP : 26.8 c  
CSPED : 12 Hz  
SLVNT : CDCL3

RESOL : 27.55 Hz  
CLASO : 22.41 Hz  
TLINE : 4  
THTOP : 30.0000  
THBTM : 1.1719  
operator

Figure 47. The HETCOR spectrum of the sulfamate derivative



13C CHSHF IRLV2 ALPHA EN1 SPL1

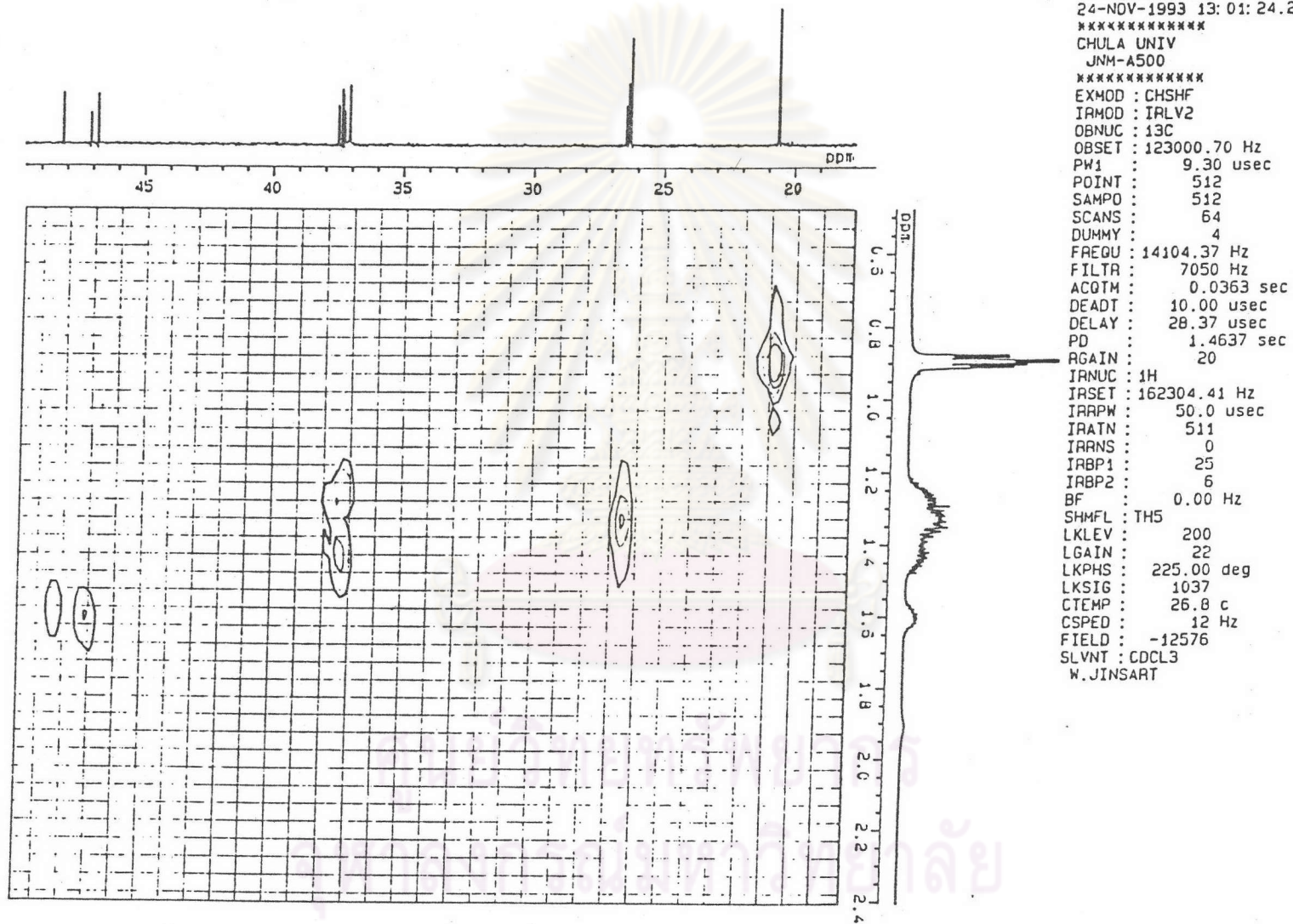


Figure 48. The HETCOR spectrum of the sulfamate derivative  
(Contour plot between 20-50 ppm)

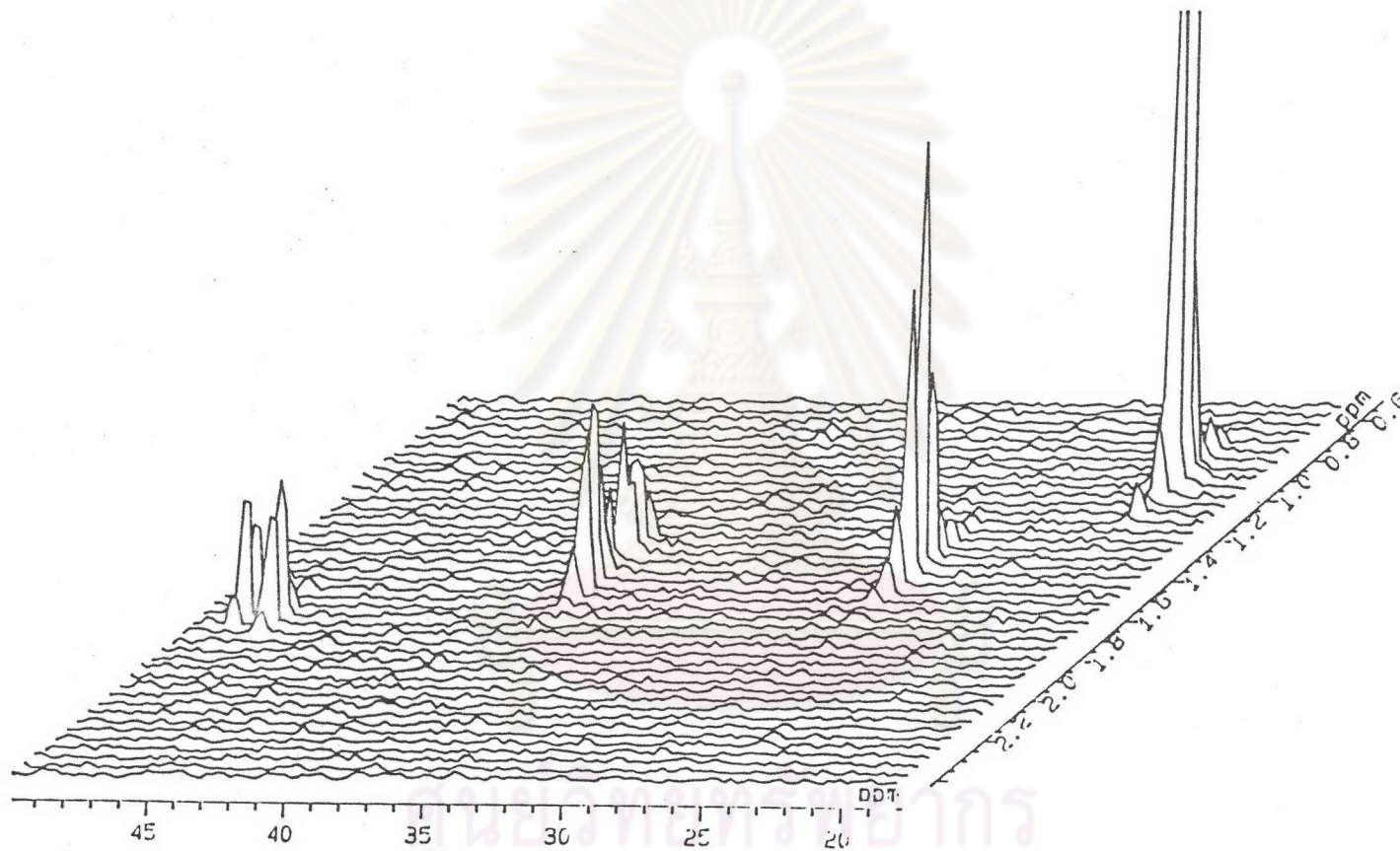
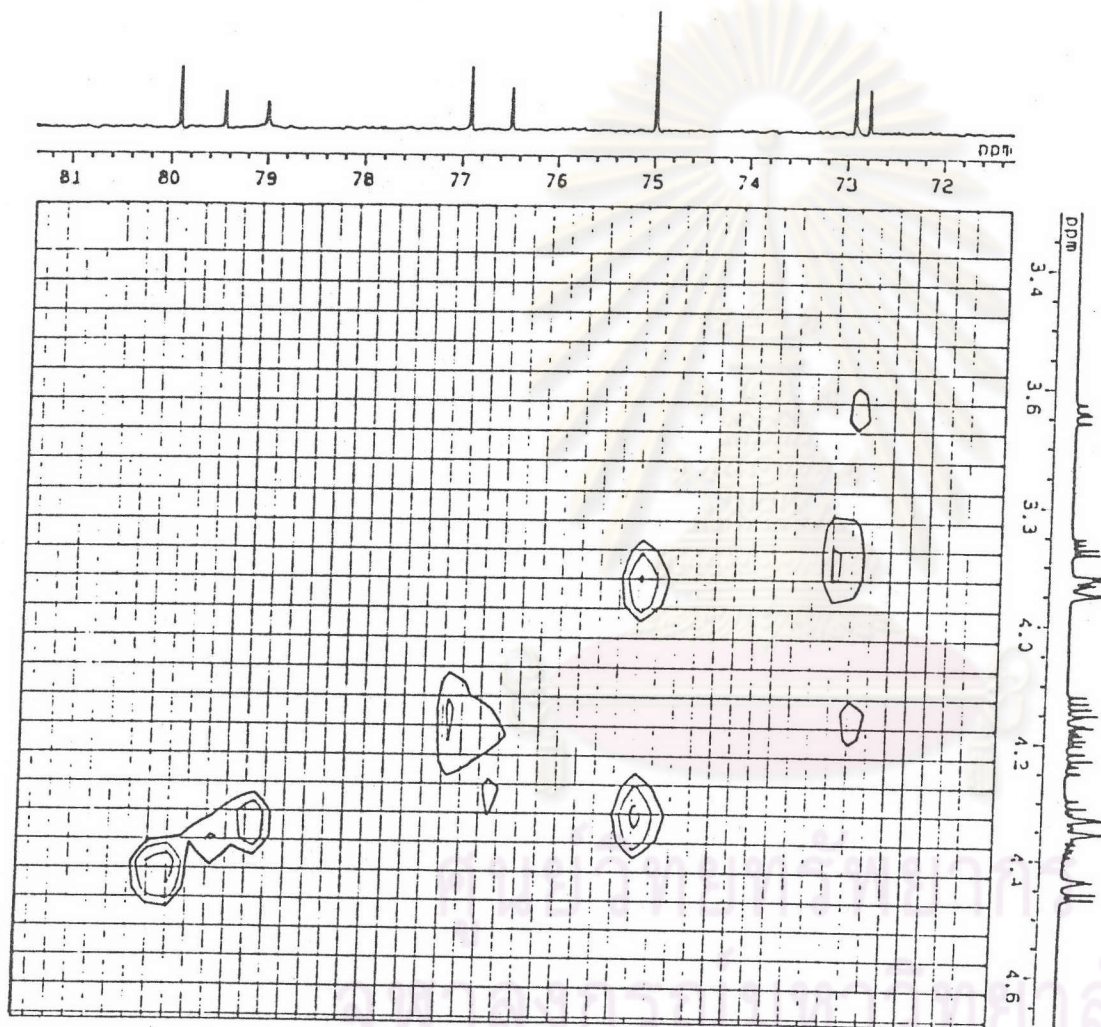


Figure 49. The HETCOR spectrum of the sulfamate derivative  
(Stacked plot between 20-50 ppm)

13C CHSHF IRLV2 ALPHA EN1 SPL1



JEOL

24-NOV-1993 13:54:16.27

\*\*\*\*\*

CHULA UNIV

JNM-A500

\*\*\*\*\*

EXMOD : CHSHF

IRMOD : IRLV2

OBNUC : 13C

OBSET : 123000.70 Hz

PW1 : 9.30 usec

POINT : 512

SAMPO : 512

SCANS : 64

DUMMY : 4

FREQU : 14104.37 Hz

FILTR : 7050 Hz

ACQTM : 0.0563 sec

DEADT : 10.00 usec

DELAY : 28.37 uscc

PD : 1.4637 sec

RGAIN : 20

IRNUC : 1H

IRSET : 162304.41 Hz

IRRPW : 50.0 usec

IRATN : 511

IRANS : 0

IRBP1 : 25

IRBP2 : 6

BF : 0.00 Hz

SHMFL : TH5

LKLEV : 200

LGAIN : 22

LKPHS : 225.00 deg

LKSIG : 1037

CTEMP : 26.8 c

CSPED : 12 Hz

FIELD : -12576

SLVNT : CDCL3

W. JINSART

Figure 50. The HETCOR spectrum of the sulfamate derivative (Contour plot between 72-80 ppm)



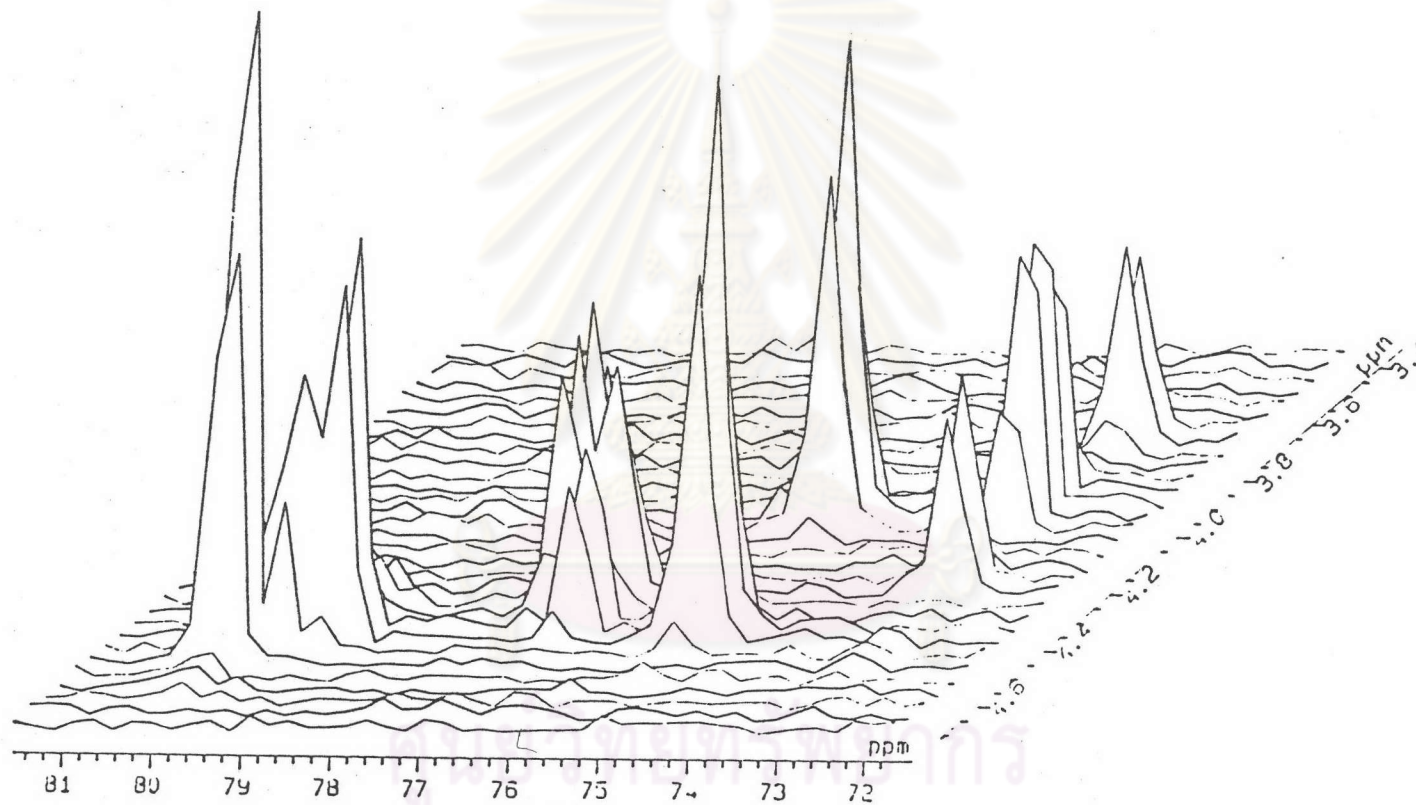
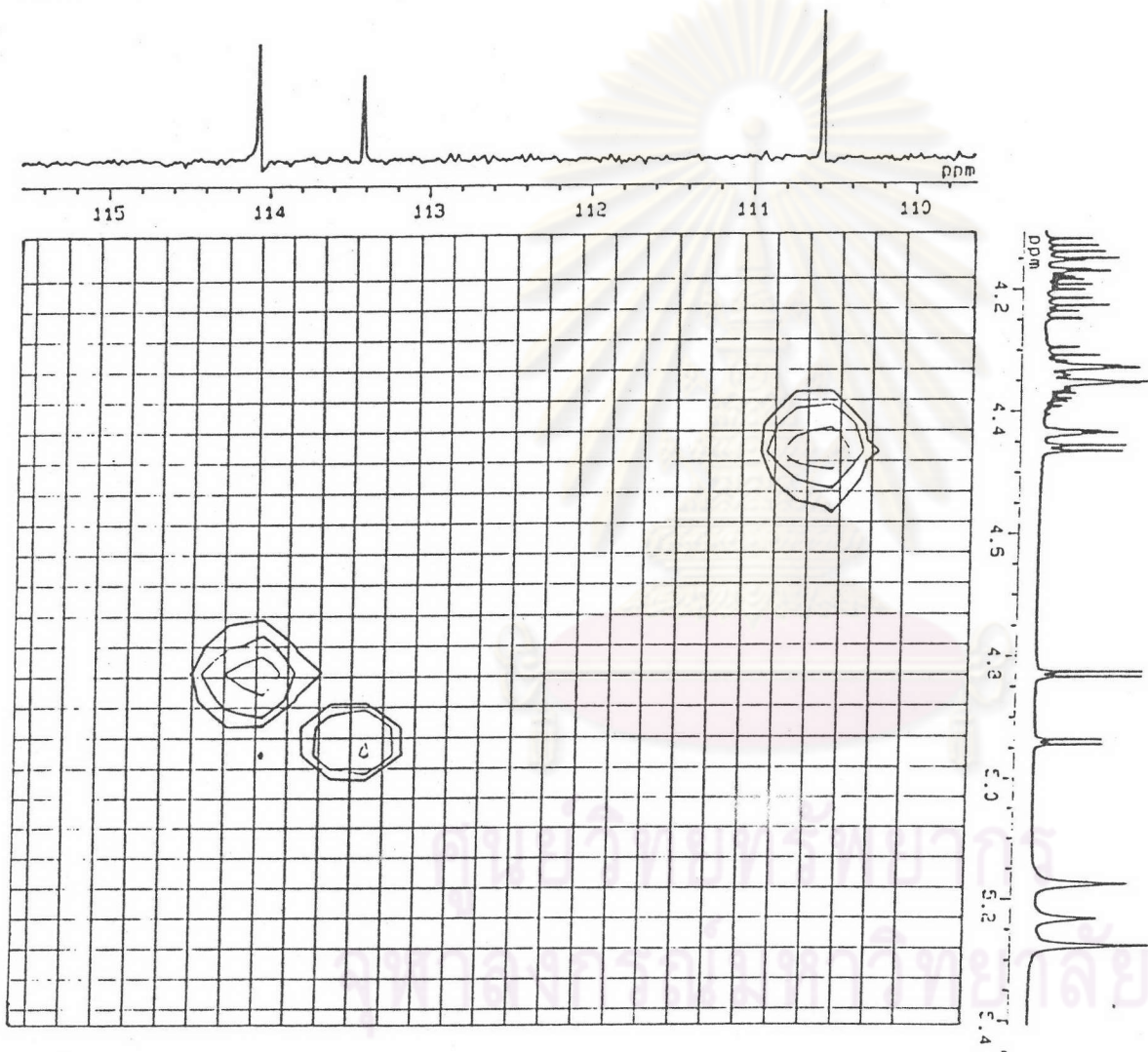


Figure 51. The HETCOR spectrum of the sulfamate derivative (Stacked plot between 72-80 ppm)



13C CHSHF IRLV2 ALPHA EN1 SPL1



JEOL

24-NOV-1993 14:13:56.34

\*\*\*\*\*

CHULA UNIV

JNM-A500

\*\*\*\*\*

EXMOD : CHSHF

IRMOD : IRLV2

OBNUC : 13C

OBSET : 123000.70 Hz

PW1 : 9.30 usec

POINT : 512

SAMPO : 512

SCANS : 64

DUMMY : 4

FREQU : 14104.37 Hz

FILTA : 7050 Hz

ACQTM : 0.0303 sec

DEADT : 10.00 usec

DELAY : 28.37 usec

PD : 1.4637 sec

RGAIN : 20

IRNUC : 1H

IRSET : 162304.41 Hz

IRRPW : 50.0 usec

IRATN : 511

IRRNS : 0

IRBP1 : 25

IRBP2 : 6

BF : 0.00 Hz

SHMFL : TH5

LKLEV : 200

LGAIN : 22

LKPHS : 225.00 dcg

LKSIG : 1037

CTEMP : 26.8 c

CSPED : 12 Hz

FIELD : -12576

SLVNT : CDCL3

W.JINSART

Figure 52. The HETCOR spectrum of the sulfamate derivative (Contour plot between 110-115 ppm)

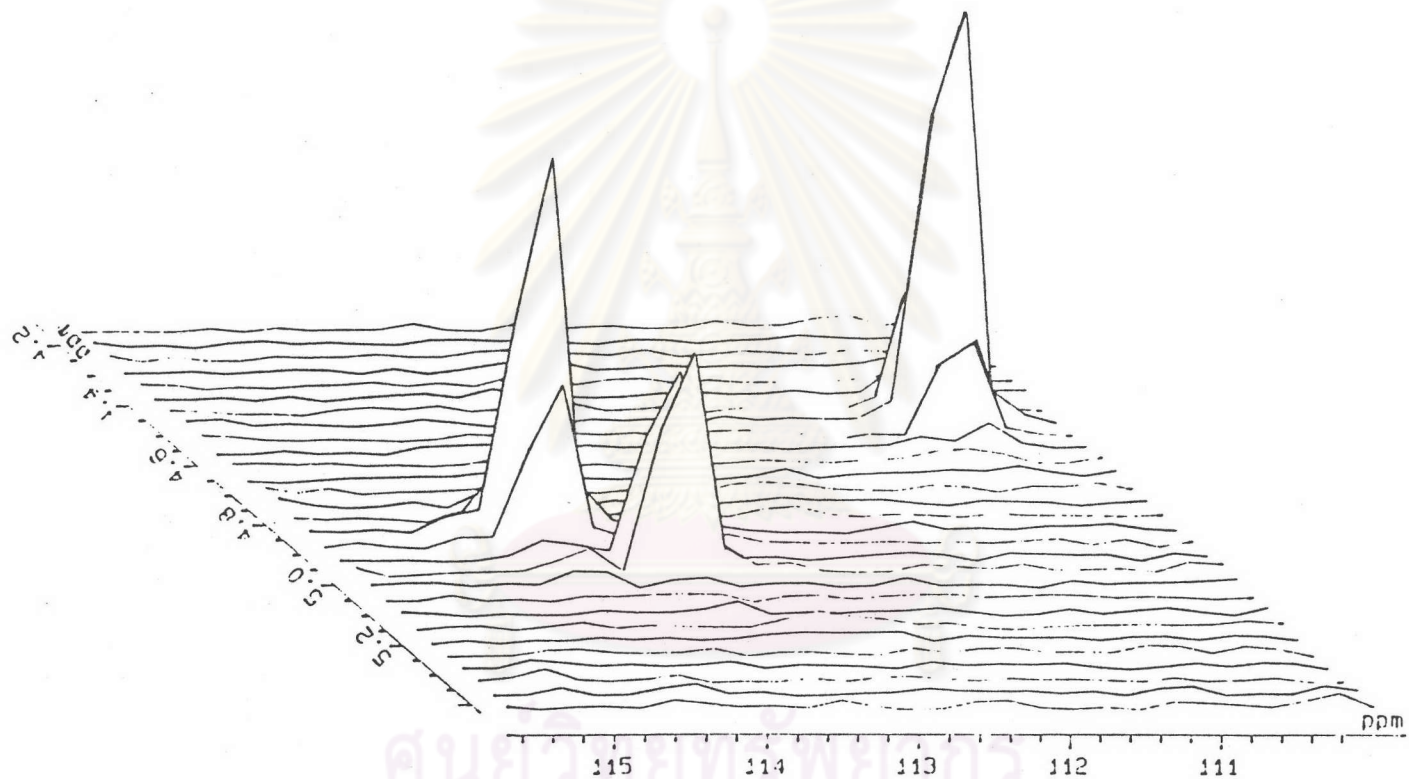


Figure 53. The HETCOR spectrum of the sulfamate derivative (Stacked plot between 110-115 ppm)

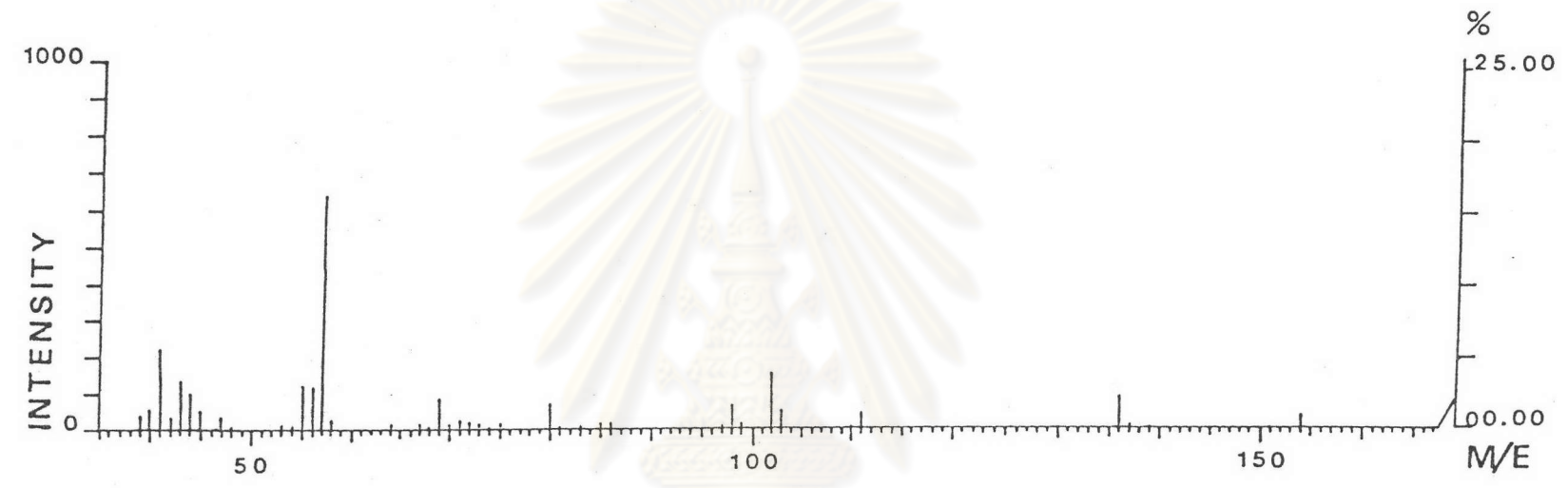


Figure 54. The MS spectrum of the sulfamate derivative

ศูนย์วิทยทรัพยากร  
จุฬาลงกรณ์มหาวิทยาลัย