CHAPTER III

EXPERIMENTS

Instruments

1. Infrared spectrophotometer
   : Perkin Elmer model 2000
2. Nuclear Magnetic Resonance Spectrophotometer
   : Bruker Spectrospin 300 (300 MHz)
   Jeol JNM-A 500 (500 MHz)
3. Mass Spectrometer
   : Platform II
4. Melting Point Apparatus
   : Buchi capillary melting point apparatus
5. CHNS/O Analyser
   : Perkin Elmer PE 2400 Series II

Chemicals

Acetic acid, glacial (Merck)

Acetic anhydride (Merck)

Ammonia solution, concentrated (25%) (Merck)

Benzylamine (Fluka)

Dicyclohexylcarbodiimide (Fluka)

Ethanol, anhydrous (Merck)

Formaldehyde solution, 37% (Merck)

Glycine (Merck)

Hydrochloric acid, concentrated (Merck)

Mrthanol, anhydrous (Merck)

Morpholine (Fluka)

Potassium carbonate (Merck)

L-Proline (Fluka)

Pyridine (Merck)

DL-Serine (Fluka)

Sodium bicarbonate (Merck)

Sodium hydroxide (Merck)

Sodium sulfate, anhydrous (Merck)

Thionyl chloride (Laboratory grade)

Triethylamine (Fluka)

Valproic acid (Sigma)

All solvent used was either B.P. or laboratory grade.

2-Propylpentanoyl chloride

Redistilled thionyl chloride (17.7 g, 10.9 ml, 150 mmol) was placed in a 100-ml round bottom flask fitted with a reflux condenser. Valproic acid (14.4 g, 8.8 ml, 100 mmol) was placed in a separatory funnel and added through the condenser during the course of 30 minutes. The reaction mixture was heated at reflux in the steam bath for 5 hours. The excess thionyl chloride was distillated, the residual crude acid chloride was attained and allowed to use without further purification.

N-(2-propylpentanoyl)-L-proline (CU-763-15-01)

A solution of L-proline (4.6 g, 40 mmol) in 10% aqueous sodium hydroxide solution (50 ml) was stirred in an ice-cooled bath. To the solution was added dropwise 2-propylpentanoyl chloride (6.5 g, 40 mmol) and was stirred at 0-4°C for 6 hours. The reaction mixture was neutralized with 10% hydrochloric acid solution and extracted with ethyl acetate (60 ml). The organic layer was washed with water (3x30 ml) and then with brine (30 ml), dehydrated with anhydrous sodium sulfate and evaporated to dryness in vacuo. Recrystallization from hexane afforded white needles of N-(2-propylpentanoyl)-L-proline 9.2 g (95%), m.p.79-80°C.

Anal.calcd. for $C_{13}H_{23}NO_3$: C, 64.700: H, 9.605: N, 5.805

Found : C, 64.711: H, 9.501: N, 5.897

IR	:	3446-2457 cm^{-1}	(ν O-H)
(KBr)		2959-2870 cm^{-1}	(ν C-H, aliphatic)
		1749 cm^{-1}	(ν C=O, acid)
		1734 cm^{-1}	(ν C=O, amide)
		1594 cm^{-1}	(ν N-C=O)
		1218 cm^{-1}	(ν C-O)
		918 cm^{-1}	(δ O-H, out-of-plane)
		(Figure 18)	

^{1}H-NMR	:	0.86 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.18-1.34 ppm	(4H, m, -CH$_2$-C*H*$_2$-CH$_3$)
		1.39, 1.61 ppm	(2H, m, -C*H*$_2$-CH$_2$-CH$_3$)
		1.93-2.06 ppm	(3H, m, -NH-CH$_2$-C*H*$_2$-C*H*$_2$-CH-C(O)-)
		2.36 ppm	(1H, m, -NH-CH$_2$-CH$_2$-C*H*$_2$-CH-C(O)-)

	2.52 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
	3.50, 3.61 ppm	(2H, ddd, -NH-CH_2-CH_2-CH_2-CH-C(O)-)
	4.57 ppm	(1H, dd, -N-C*H*-C(O)-)
	9.71 ppm	(1H, broad, -COO*H*)
	(Figure 19)	
^{13}C-NMR :	14.14 ppm	(-*C*H_3)
($CDCl_3$)	20.49, 20.80 ppm	(-CH_2-*C*H_2-CH_3)
	24.72 ppm	(-N-CH_2-*C*H_2-)
	27.47 ppm	(-N-CH_2-CH_2-*C*H_2-)
	34.79, 35.13 ppm	(-*C*H_2-CH_2-CH_3)
	43.59 ppm	(-*C*H-CH_2-CH_2-CH_3)
	47.81 ppm	(-N-*C*H_2-CH_2-)
	59.74 ppm	(-N-*C*H-C(O)-OH)
	173.03 ppm	(-*C*(O)-N-)
	178.45 ppm	(-*C*(O)-OH)
	(Figure 22)	
EIMS :	242 (2.95%), 199 (21.97%), 170 (33.77%), 155 (29.44%),	
	154 (51.95%), 127 (15.80%), 126 (84.42%), 99 (7.25%),	
	83 (92.64%), 57 (91.34%)	
	(Figure 25)	

L-Proline ethyl ester hydrochloride

Redistilled thionyl chloride (1.6 g, 1.0 ml, 14 mmol) was added to anhydrous ethanol (5.0 ml) at the ice-cooled bath temperature. L-Proline (1.2 g, 10 mmol) was then added and the mixture was heated to reflux for 2 hours. Evaporation of the excess ethanol yielded L-proline ethyl ester hydrochloride as oily liquid and was not further purified.

IR	:	3682-2287 cm^{-1}	(ν N-H, HCl salt)
(Neat)		2959-2924 cm^{-1}	(ν C-H, aliphatic)
		1738 cm^{-1}	(ν C=O, ester)
		1240 cm^{-1}	(ν C-C(=O)-O-)
		(Figure 26)	

N-(2-propylpentanoyl)-L-proline ethyl ester (CU 763-15-02)

2-Propylpentanoylchloride (1.6 g, 10 mmol) was dissolved in dry dichloromethane (10 ml) and placed in a separatory funnel. The mixture was added dropwise through the condenser to an ice-cooled suspension of L-proline ethyl ester hydrochloride (1.8 g, 10 mmol) in dry dichloromethane (20 ml) containing triethylamine (2.0 g, 20 mmol). Stirring was continued at 0-4°C for 4 hours. The mixture was extracted with 1 N hydrochloric acid (30 ml), 1N sodium bicarbonate (30 ml), water (30 ml), dehydrated with anhydrous sodium sulfate and evaporated to dryness in vacuo. The colorless liquid residue was purified by column chromatography using silica gel column and eluted with hexane: ethyl acetate (20:1). The analytically pure ester weighted 1.5 g (68%).

Anal.calcd. for $C_{15}H_{27}NO_3$: C, 66.878: H, 10.104: N, 5.200

Found : C, 66.867: H, 10.054: N, 5.159

IR	:	2957-2872 cm^{-1}	(ν C-H, aliphatic)
(Neat)		1743 cm^{-1}	(ν C=O, ester)
		1650 cm^{-1}	(ν C=O, amide)
		1187 cm^{-1}	(ν C-C-(=O)-O-, ester)
		1045, 1031 cm^{-1}	(ν O-C-C)
		(Figure 27)	
^{1}H-NMR	:	0.84 ppm	(6H, m, -C*H*$_3$)
($CDCl_3$)		1.16-1.40 ppm	(9H, m, -C*H*$_2$-C*H*$_2$-CH$_3$, -C(O)-O-CH$_2$-C*H*$_3$)
		1.59 ppm	(2H, m. -C*H*$_2$-CH$_2$-CH$_3$)
		1.84-2.06 ppm	(3H, m, -N-CH$_2$-C*H*$_2$-C*H*$_2$-CH-C(O)-)
		2.13 pmm	(1H, m, -N-CH$_2$-CH$_2$-C*H*$_2$-CH-C(O)-)
		2.47 ppm	(1H, m, -C*H*-CH$_2$-CH$_2$-CH$_3$)
		3.52,3.63 ppm	(2H, m, -N-C*H*$_2$-CH$_2$-CH$_2$-CH-C(O)-))
		4.11 ppm	(2H, q, -C(O)-O-C*H*$_2$-CH$_3$)
		4.43 ppm	(1H, dd, -N-C*H*-C(O)-O-)
		(Figure 28)	
^{13}C-NMR	:	14.01, 14.12 ppm	(-CH$_2$-CH$_2$-*C*H$_3$)
($CDCl_3$)		14.19 ppm	(-O-CH$_2$-*C*H$_3$)
		20.41, 20.79 ppm	(-CH$_2$-*C*H$_2$-CH$_3$)
		24.75 ppm	(-N-CH$_2$-*C*H$_2$-)
		29.11 ppm	(-N-CH$_2$-CH$_2$-*C*H$_2$-)
		34.97, 35.28 ppm	(-*C*H$_2$-CH$_2$-CH$_3$)

43.29 ppm	(-*C*H-CH_2-CH_2-CH_3)
46.99 ppm	(-N-*C*H_2-CH_2-)
58.74 ppm	(-C(O)-O-*C*H_2-CH_3)
60.81 ppm	(-N-*C*H-C(O)-O-)
172.37 ppm	(-*C*(O)-N-)
175.21 ppm	(-*C*(O)-O-CH_2-CH_3)

(Figure 31)

EIMS : 270 (80.43%), 227 (24.89%), 196 (19.57%), 154 (7.55%), 142 (16.17%), 127 (5.19),99 (9.57%),57 (88.09%)

(Figure 34)

(2-Propylpentanoyl)-L-proline benzylamide (CU 763-15-03)

Benzylamine (2.1 g, 20 mmol) was added to a solution of N-(2-propylpentanoyl)-L-proline (2.4 g, 10 mmol) in dichloromethane (40 ml) followed by the addition of dicyclohexylcarbodiimide (2.1 g, 10 mmol). A precipitate, N,N′-dicyclohexylurea started to separate almost immediately and its amount gradually increased. After 20 hours at room temperature, 5% acetic acid (30 ml) was added and stirred for 30 minutes in order to react with the unreacted carbodiimide. The urea derivative was removed by filtration and washed with dichloron ethane (20 ml). The combined filtrate and washings were extracted with 1 N hydrochloric acid (30 ml), saturated aqueous sodium bicarbonate solution (30 ml), water (30 ml), dried over anhydrous sodium sulfate and evaporated to dryness in vacuo. The residue was purified by column chromatography with chloroform : methanol (80:1) as eluent yields 1.8 g (56%) of (2-propylpentanoyl)-L-proline benzylamide as clear liquid.

Anal.calcd. for $C_{20}H_{30}N_2O_2$: C, 72.690: H, 9.151: N, 8.476

Found : C, 72.727: H, 9.109: N, 8.630

IR	:	3295 cm^{-1}	(ν N-H)
(Neat)		3084-3033 cm^{-1}	(ν C-H, aromatic)
		2955-2871 cm^{-1}	(ν C-H, aliphatic)
		1952-1749 cm^{-1}	(overtone or combination)
		1679 cm^{-1}	(ν C=O, amide)
		1621 cm^{-1}	(ν C=O, amide)
		736, 699 cm^{-1}	(δ out-of-plane, γ)
		(Figure 35)	
^{1}H-NMR	:	0.80, 0.89 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.16, 1.26 ppm	(4H, m, -CH$_2$-C*H*$_2$-CH$_3$)
		1.37, 1.58 ppm	(4H, m, -C*H*$_2$-CH$_2$-CH$_3$)
		1.79 ppm	(1H, m, -N-CH$_2$-CH$_2$-C*H*$_2$-CH-C(O)-)
		1.98, 2.13 ppm	(2H, m, -N-CH$_2$-C*H*$_2$-CH$_2$-CH-C(O)-)
		2.52 ppm	(2H, m, -N-CH$_2$-CH$_2$-C*H*$_2$-CH-C(O), C*H*-CH$_2$-CH$_2$-CH$_3$)
		3.49, 3.57 ppm	(2H, m, -N-C*H*$_2$-CH$_2$-CH$_2$-CH-C(O)-)
		4.39 ppm	(2H, m, -NH-C*H*$_2$-Ph)
		4.70 ppm	(1H, dd, -N-C*H*-C(O)-NH-)
		7.21-7.31 ppm	(5H, m, aromatic H)
		7.70 ppm	(1H, t, -N*H*-CH$_2$-Ph)
		(Figure 36)	

^{13}C-NMR	:	14.23 ppm	(-CH_3)
($CDCl_3$)		20.66, 20.91 ppm	(-CH_2-CH_2-CH_3)
		25.07 ppm	(-N-CH_2-CH_2-)
		26.75 ppm	(-N-CH_2-CH_2-CH_2-)
		34.89, 35.45 ppm	(-CH_2-CH_2-CH_3)
		43.46 ppm	(-NH-CH_2-Ph)
		43.51 ppm	(-CH-CH_2-CH_2-CH_3)
		47.56 ppm	(-N-CH_2-CH_2-)
		59.50 ppm	(-N-CH-C(O)-NH-)
127.18, 127.56,128.53, 138.39 ppm			(aromatic C)
		171.18 ppm	(-C(O)-N-CH_2)
		177.13 ppm	(-C(O)-NH-CH_2-Ph)
		(Figure 39)	

EIMS : 330 (4.46%), 288 (6.20%), 203 (4.56%), 154 (9.02%), 127 (10.20%), 99 (17.65%), 91 (84.31%), 57 (86.27%) (Figure 42)

DL- Serine methyl ester hydrochloride

The DL-serine methyl ester hydrochloride was prepared from thionyl chloride (4 ml), anhydrous methanol (25 ml) and DL–serine (5.3 g, 50mmol) as described in the preparation of L-proline ethyl ester hydrochloride. After concentration of the mixture in vacuo, anhydrous ether was added in order to precipitate DL–Serine methyl ester hydrochloride as white solid.

IR	: 3682-2287 cm^{-1}	(ν N-H, HCl salt)	
(Nujol, mull)	3402 cm^{-1}	(ν O-H)	
	2952-2948 cm^{-1}	(ν C-H, aliphatic)	
	1738 cm^{-1}	(ν C=O, ester)	
	1583 cm^{-1}	(δ N-H, HCl salt)	
	1251 cm^{-1}	(ν C-C(=O)-O-)	(Figure 43)

N-(2-propylpentanoyl)-DL-serine methyl ester (CU 763-15-15)

The compound was prepared from 2-Propylpentanoylchloride (2.4 g, 15 mmol) and DL-serine methyl ester hydrochloride (2.3 g, 15 mmol) in the presence of triethylamine (3.0 g, 30 mmol) in dried dichloromethane (20 ml) for 4 hours as described for N-(2-propylpentanoyl)-L-proline ethyl ester. Recrystallization from hexane gave 3.1 g (83%) of N-(2-propylpentanoyl)-DL-serine methyl ester, m.p. 57-58°C.

Anal.calcd. for $C_{12}H_{23}NO_4$: C, 58.752: H, 9.449: N, 5.711

Found : C, 58.749: H, 9.350: N, 5.751

IR	: 3579-3143 cm^{-1}	(ν O-H)	
(KBr)	3372 cm^{-1}	(ν N-H)	
	2957-2872 cm^{-1}	(ν C-H, aliphatic)	
	1755 cm^{-1}	(ν C=O, ester)	
	1649 cm^{-1}	(ν C=O, amide)	
	1548 cm^{-1}	(δ N-H)	
	1217 cm^{-1}	(ν C-C-(=O)-O-, ester)	
	1050 cm^{-1}	(ν O-C-C)	(Figure 44)

^{1}H-NMR	:	0.90 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.20-1.47 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH_3)
		1.60 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.17 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		2.61 ppm	(1H, broad, -CH_2-O*H*)
		3.80 ppm	(3H, s, -O-C*H*$_3$)
		3.96 ppm	(2H, m, -C*H*$_2$-OH)
		4.71 ppm	(1H, m, -NH-C*H*-C(O)-)
		6.39 ppm	(1H, d, -C(O)-N*H*-CH-)
		(Figure 45)	
^{13}C-NMR	:	14.08 ppm	(-CH_2-CH_2-*C*H$_3$)
($CDCl_3$)		20.67, 20.76 ppm	(-CH_2-*C*H$_2$-CH_3)
		35.15 ppm	(-*C*H$_2$-CH_2-CH_3)
		47.43 ppm	(-*C*H-CH_2-CH_2-CH_3)
		52.74 ppm	(-O-CH_3)
		54.58 ppm	(-NH-*C*H-C(O)-)
		63.94 ppm	(-*C*H$_2$-OH)
		171.01 ppm	(-*C*(O)-NH-)
		176.71 ppm	(-*C*(O)-O-)
		(Figure 47)	
EIMS	:	246 (25.11%), 215 (16.24%), 203 (85.65%), 185 (48.52%), 174 (100.00%), 156 (37.13%), 144 (94.09%), 127 (85.65%), 99 (87.76%), 57 (97.89%)	
		(Figure 48)	

N-(2-propylpentanoyl)-DL-serine (CU 763-15-04)

N-(2-propylpentanoyl)-DL-serine methyl ester (4.9 g, 20 mmol) was added to a mixture of methanol (30 ml) and 1 N sodium hydroxide solution (30 ml). The mixture was stirred at room temperature for 2 hours. A clear solution appeared. After distillation of methanol, the residue was neutralized with 1 N hydrochloric acid and acidified with concentrated hydrochloric acid. extracted by ethyl acetate (3x30 ml). The organic layers were combined and washed with water (30 ml) and brine (30 ml), dried over anhydrous sodium sulfate and evaporated to dryness. Recrystallization in acetone afforded 3.5 g (76%) of N-(2-propylpentanoyl)-DL-serine, m.p.107-108° C.

Anal.calcd. for $C_{11}H_{21}NO_4$: C, 57.121: H, 9.153: N, 6.057

Found : C, 57.144: H, 9.070: N, 6.082

IR	:	3586-2206 cm^{-1}	(ν O-H)
(KBr)		3372 cm^{-1}	(ν N-H)
		2960-2870 cm^{-1}	(ν C-H, aliphatic)
		1736 cm^{-1}	(ν C=O, acid)
		1647 cm^{-1}	(ν C=O, amide)
		1540 cm^{-1}	(δ N-H, amide)
		1234 cm^{-1}	(ν C-O, acid)
		1087 cm^{-1}	(ν C-C-O, asym.)
		919 cm^{-1}	(δ O-H, out-of-plane)
		(Figure 49)	

^{1}H-NMR	:	0.82 ppm	(6H, t, -C*H*$_3$)
(DMSO-d$_6$)		1.10-1.32 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH$_3$)
		1.41 ppm	(2H, m, -C*H*$_2$-CH$_2$-CH$_3$)
		2.27 ppm	(1H, m, -C*H*-CH$_2$-CH$_2$-CH$_3$)
		3.35 ppm	(1H, broad, -CH$_2$-O*H*)
		3.63 ppm	(2H, m, -CH-C*H*$_2$-OH)
		4.26 ppm	(1H, m, -N-C*H*-C(O)-O-)
		7.90 ppm	(1H, d, -C(O)-N*H*-)
		(Figure 50)	
^{13}C-NMR	:	16.85 ppm	(-*C*H$_3$)
(DMSO-d$_6$)		20.13, 20.27 ppm	(-CH$_2$-*C*H$_2$-CH$_3$)
		35.00, 35.14 ppm	(-*C*H$_2$-CH$_2$-CH$_3$)
		45.01 ppm	(-*C*H-CH$_2$-CH$_2$-CH$_3$)
		54.60 ppm	(-NH-*C*H-C(O)-O-)
		61.70 ppm	(-CH-*C*H$_2$-OH)
		172.31 ppm	(-*C*(O)-NH-)
		175.28 ppm	(-*C*(O)-OH)
		(Figure 52)	
EIMS	:	232 (79.17%), 214 (16.88%), 201 (11.25%), 189 (85.42%), 183 (24.38%), 160 (97.92%), 142 (70.00%), 127 (97.08%), 99 (94.58%), 57 (100.00%)	
		(Figure 54)	

DL-Serine ethyl ester hydrochloride

The compound was prepared from thionyl chloride (4 ml), anhydrous ethanol (25 ml) and DL–serine (5.3 g, 50mmol) as described for L–Proline ethyl ester hydrochloride. After concentration of the mixture in vacuo, anhydrous ether was added to percipitate DL–serine ethlyl ester hydrochloride as white solid.

IR	:	3380 cm^{-1}	(ν O-H)
(Nujol, mull)		3616-2243 cm^{-1}	(ν N-H, HCl salt)
		1742 cm^{-1}	(ν C=O, ester)
		1582 cm^{-1}	(δ N-H, HCl salt)
		1240 cm^{-1}	(ν C-C(=O)-O-)
		1027 cm^{-1}	(ν O-C-C)
		(Figure 55)	

N-(2-Propylpentanoyl)-DL-serine ethyl ester (CU 763-15-05)

The compound was prepared from 2-Propylpentanoylchloride (2.4 g, 15 mmol) and DL-serine ethyl ester hydrochloride (2.5 g, 15 mmol) in dry dichloromethane (20 ml) in the presence of triethylamine (3.0 g, 30 mmol) for 4 hours. Recrystallization from benzene gave 0.3 g (73%) of N-(2-propylpentanoyl)-DL-serine ethyl ester, m.p. 99-101°C.

Anal.calcd.for $C_{13}H_{25}NO_4$: C, 60.204: H, 9.717: N, 5.402

Found : C, 60.254: H, 9.757: N, 5.494

IR	:	3282 cm^{-1}	(ν O-H)
(KBr)		3476 cm^{-1}	(ν N-H)
		2957-2870 cm^{-1}	(ν C-H, aliphatic)
		1748 cm^{-1}	(ν C=O, ester)
		1645 cm^{-1}	(ν C=O, amide)
		1287 cm^{-1}	(ν C-C-(=O)-O-, ester)
		1079 cm^{-1}	(ν O-C-C)
		(Figure 56)	
^{1}H-NMR	:	0.90 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.25-1.47 ppm	(9H, m, -CH_2-C*H*$_2$-CH_3, -C(O)-O-CH_2-C*H*$_3$)
		1.62 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.16 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		2.66 ppm	(1H, t, J=5.8 Hz, -CH_2-O*H*)
		3.95 ppm	(2H, m, -CH-C*H*$_2$-OH)
		4.25 ppm	(2H, q, -C(O)-O-C*H*$_2$-CH_3)
		4.69 ppm	(1H, m, -N-C*H*-C(O)-O-)
		6.40 ppm	(1H, d, -C(O)-N*H*-)
		(Figure 57)	
^{13}C-NMR	:	14.00 ppm	(-CH_2-CH_2-*C*H_3)
($CDCl_3$)		20.92 ppm	(-O-CH_2-*C*H_3)
		35.21 ppm	(-CH_2-*C*H_2-CH_3)
		47.42 ppm	(-*C*H-CH_2-CH_2-CH_3)
		54.84 ppm	(-NH-*C*H-C(O)-O-)
		62.16 ppm	(-C(O)-O-*C*H_2-CH_3)

64.05 ppm (-CH_2-OH)

170.81 ppm (-C(O)-N-)

176.88 ppm (-C(O)-O-CH_2-CH_3)

(Figure 59)

EIMS : 260 (5.51%), 242 (0.94%), 229 (3.57%), 217 (20.36%), 188 (39.52%), 183 (8.81%), 142 (7.74%), 102 (13.45%), 99 (28.33%), 57 (100.00%)

(Figure 60)

N-(2-Propylpentanoyl)-DL-serine benzylamide (CU 763-15-06)

The N-(2-propylpentanoyl)-DL-serine benzylamide was prepared from N-(2-propylpentanoyl)-DL-serine (2.3 g, 10 mmol) and benzylamine (2.14 g, 20 mmol) in the presence of dicyclohexylcarbodiimide (2.06 g, 10 mmol) for 20 hours as described for the preparation of N-(2-propylpentanoyl)-L-proline benzylamide. Purification by column chromatography with chloroform: methanol (80:1) as eluent yielded white precipitate of N-(2-propylpentanoyl)-DL-serine benzylamide (1.8 g, 56%), m.p. 145-146°C.

Anal.calcd. for $C_{18}H_{28}N_2O_3$: C, 67.472: H, 8.807: N, 8.741

Found : C, 67.576: H, 8.838: N, 8.697

IR : 3275 cm^{-1} (ν N-H and ν O-H)

(Neat) 3092-3025 cm^{-1} (ν C-H, aromatic)

2959-2878 cm^{-1} (ν C-H, aliphatic)

1955-1745 cm^{-1} (overtone or combination)

1639 cm^{-1} (ν C=O, amide)

	1621 cm^{-1}	(ν C=O, amide)
	1553 cm^{-1}	(δ N-H)
	1048 cm^{-1}	(ν C-O)
	742, 694 cm^{-1}	(δ ring C-H, out-of-plane)
	(Figure 61)	

^{1}H-NMR	:	0.85 ppm	(6H, m, -C*H*$_3$)
($CDCl_3$)		1.10-1.44 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH_3)
		1.44-1.61 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.13 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		3.63, 4.16 ppm	(2H, d, C*H*$_2$-OH)
		3.69 ppm	(1H, broad, -CH_2-O*H*)
		4.34-4.50 ppm	(3H, m, -NH-C*H*-C(O)-NH-C*H*$_2$-Ph)
		6.69 ppm	(1H, d, -C(O)-N*H*-CH-C(O)-)
		7.23-7.31 ppm	(5H, m, aromatic H)
		7.32 ppm	(1H, t, -C(O)-N*H*-CH_2-Ph)
		(Figure 62)	

^{13}C-NMR	:	14.04 ppm	(-*C*H_3)
($CDCl_3$)		20.72 ppm	(-CH_2-*C*H_2-CH_3)
		35.06-35.08 ppm	(-*C*H_2-CH_2-CH_3)
		43.43 ppm	(-NH-*C*H_2-Ph)
		47.24 ppm	(-*C*H-CH_2-CH_2-CH_3)
		53.40 ppm	(-NH-*C*H-C(O)-NH-)
		62.75 ppm	(-*C*H_2-OH)
	127.52, 127.55,128.69, 137.61 ppm		(aromatic C)
		171.20 ppm	(-*C*(O)-NH-CH)
		177.44 ppm	(-*C*(O)-NH-CH_2-Ph) (Figure 64)

EIMS : 320 (9.88%), 278 (2.80%), 249 (1.49%), 214 (18.52%), 193 (10.08%), 186 (16.56%), 169 (29.63%), 127 (64.61%), 106 (58.02%), 99 (44.86%), 91 (93.83%), 57 (97.94%) (Figure 67)

Glycine ethyl ester hydrochloride

This compound was prepared from redistilled thionyl chloride (1.6 g, 1.0 ml, 14 mmol), anhydrous ethanol (5.0 ml) and glycine (0.8g, 10 mmol) as described for L-proline ethyl ester hydrochloride for 2 hours. The excess ethanol was evaporated. The oily residue was added anhydrous ether yielding glycine ethyl ester hydrochloride as white solid and was not further purified.

IR :	3682-2265 cm^{-1}	(ν N-H, HCl salt)
(Nujol,mull)	2959-2924 cm^{-1}	(ν C-H,aliphatic)
	1744 cm^{-1}	(ν C=O,ester)
	1255 cm^{-1}	(ν C-C(=O)-O-)
	1048 cm^{-1}	(ν O-C-C)

(Figure 68)

N-(2-Propylpentanoyl)-glycine ethyl ester (CU 763-15-07)

This compound was prepared from 2-propylpentanoylchloride (1.6 g, 10 mmol) , glycine ethyl ester hydrochloride (1.4 g, 10 mmol) in dried dichloromethane (20 ml) containing triethylamine (2.0 g, 20 mmol) for 4 hours. Recrystallization from hexane gave 1.4 g (61%) of N-(2-propylpentanoyl) glycine ethyl ester as white needles, m.p. 79-80°C.

Anal.calcd.for $C_{15}H_{27}NO_3$: C, 66.878: H, 10.104: N, 5.200

Found : C, 66.867: H, 10.054: N, 5.159

IR	:	3296 cm^{-1}	(ν N-H)
(KBr)		2953-2870 cm^{-1}	(ν C-H, aliphatic)
		1731 cm^{-1}	(ν C=O, ester)
		1641 cm^{-1}	(ν C=O, amide)
		1550 cm^{-1}	(δ N-H)
		1243 cm^{-1}	(ν C-C-(=O)-O-, ester)
		1040 cm^{-1}	(ν O-C-C)
		(Figure 69)	
^{1}H-NMR	:	0.90 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.20-1.46	(9H, m, -C*H*$_2$-C*H*$_2$-CH_3, -C(O)-O-CH_2-C*H*$_3$)
		1.62 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.12 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		4.05 ppm	(2H, d, -N-C*H*$_2$-C(O)-O-)
		4.23 ppm	(2H, q, -C(O)-O-C*H*$_2$-CH_3)
		5.92 ppm	(1H, broad, -C(O)-N*H*-)
		(Figure 70)	
^{13}C-NMR	:	14.08 ppm	(-CH_2-CH_2-*C*H_3)
($CDCl_3$)		14.12 ppm	(-O-CH_2-*C*H_3)
		20.74 ppm	(-CH_2-*C*H_2-CH_3)
		35.19 ppm	(-*C*H_2-CH_2-CH_3)
		41.19 ppm	(-NH-*C*H_2-C(O)-O-)
		47.44 ppm	(-*C*H-CH_2-CH_2-CH_3)

61.48 ppm (-C(O)-O-CH_2-CH_3)

176.21 ppm (-C(O)-N-)

(Figure 72)

EIMS : 230 (0.69%), 187 (29.71%), 184 (4.08%), 158 (60.88%), 127 (11.84%), 99 (7.94%), 85 (61.76 %), 83 (100.00%), 57 (45.29%)

(Figure 73)

N-(2-Propylpentanoyl)-glycine benzylamide (CU 763-15-08)

This compound was prepared from N-(2-propylpentanoyl) glycine (2.0 g, 10 mmol) and benzylamine (2.1g, 20 mmol) in the presence of dicyclohexylcarbodiimide (2.1 g, 10 mmol) in tetrahydrofuran (50 ml) for 20 hours as described for the preparation of N-(2-propylpentanoyl)-L-proline benzylamide. Recrystallization in hexane gave 1.7 g (58%) of N-(2-propylpentanoyl)-glycine benzylamide, m.p.108-110°C.

Anal.calcd.for $C_{17}H_{25}N_2O_2$: C, 70.556: H, 8.708: N, 9.679

Found : C, 70.583: H, 8.835: N, 9.690

IR : 3293 cm^{-1} (ν N-H)

(KBr) 3089 cm^{-1} (ν C-H, aromatic)

2944-2870 cm^{-1} (ν C-H, aliphatic)

1963-1815 cm^{-1} (overtone or combination)

1639 cm^{-1} (ν C=O, amide)

1558 cm^{-1} (δ N-H)

1454 cm^{-1} (ν C=C)

		1256 cm^{-1}	(interaction between δN-H and ν C-N)
		744, 694 cm^{-1}	(δ ring C-H, out-of-plane)
		(Figure 74)	
^{1}H-NMR	:	0.85 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.15-1.42 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH_3)
		1.55 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.11 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		3.96 ppm	(2H, d, -NH-C*H*$_2$-C(O)-NH)
		4.43 ppm	(2H, d, -C(O)-NH-C*H*$_2$-Ph)
		6.33 ppm	(1H, broad, -C(O)-N*H*-CH_2-C(O)-)
		6.66 ppm	(1H, broad, -C(O)-N*H*-CH_2-Ph)
		7.29 ppm	(5H, m, aromatic H)
		(Figure 75)	
^{13}C-NMR	:	14.06 ppm	(-*C*H_3)
($CDCl_3$)		20.78 ppm	(-CH_2-*C*H_2-CH_3)
		35.13 ppm	(-*C*H_2-CH_2-CH_3)
		43.47 ppm	(-NH-*C*H_2-Ph)
		47.29 ppm	(-*C*H-CH_2-CH_2-CH_3)
		127.61, 127.75,128.73 ppm	(aromatic C)
		168.96 ppm	(-*C*(O)-NH-CH_2-C(O)-)
		176.91 ppm	(-*C*(O)-NH-CH_2-Ph)
		(Figure 77)	
EIMS	:	290(21.92%), 248 (11.45%), 219 (12.89%), 184 (62.11%), 163 (79.30%), 127 (48.46%), 106 (95.59%), 99 (32.16%), 91 (100.00%), 77 (18.83%), 57 (89.43%)	(Figure 79)

2-Propylpentamide

2-Propylpentanoylchloride (4.9 g, 30 mmol) was added, dropwise, over a period of about 30 minutes, to a stirred 25% ammonia solution (30 ml), cooled in an ice-bath. Stirring was continued until the white fuming disappeared. The mixture was extracted with ethyl acetate (3x20 ml), the organic extracts were pooled, washed with water (3x20 ml), dried over anhydrous sodium sulfate and evaporated to dryness. The crude product was purified by recrystallization from hexane to gave 3.2 g (74%) 2-propylpentamide as white needles.

IR	:	3390, 3195cm^{-1}	(ν N-H)
(KBr)		2957-2870 cm^{-1}	(ν C-H, aliphatic)
		1654 cm^{-1}	(ν C=O, amide)
		1465 cm^{-1}	(ν C-N)
		(Figure 80)	

N-Hydroxymethyl-2-propylpentamide (CU 763-15-09)

A solution of 2-propylpentamide (3.0 g, 21 mmol) and potassium carbonate (1.4 g, 10 mmol) in ethanol (30 ml) and water (3 ml) was mixed with 37% formaldehyde solution (3.4 ml, 42 mmol). The mixture was stirred at room temperature for 24 hours. After evaporation of the solvents in vacuo, the residue was taken up in ethyl acetate and washed with water until pH7. The organic layer was dried over anhydrous sodium sulfate and evaporated to dryness. Recrystallization from benzene gave 2.9 g (80%) N-hydroxymethyl-2-propylpentamide, m.p.110-111°C.

Anal.calcd. for $C_9H_{19}NO_2$: C, 62.428: H, 10.983: N, 8.092

Found : C, 62.446: H, 10.933: N, 8.042

IR	:	3302 cm^{-1}	(ν N-H)
(KBr)		3217 cm^{-1}	(ν O-H)
		2959-2870 cm^{-1}	(ν C-H, aliphatic)
		1655 cm^{-1}	(ν C=O, amide)
		1546 cm^{-1}	(δ N-H)
		1049, 1015 cm^{-1}	(ν C-O)
		(Figure 81)	
^{1}H-NMR	:	0.88 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.19-1.45 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH_3)
		1.55 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.05 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		3.36 ppm	(1H, t, J=7.7 Hz, -CH_2-O*H*)
		4.73 ppm	(2H, t, J=7.1 Hz, -C*H*$_2$-OH)
		6.39 ppm	(1H, broad, -C(O)-N*H*-)
		(Figure 82)	
^{13}C-NMR	:	14.07 ppm	(-CH_2-CH_2-*C*H_3)
($CDCl_3$)		20.72 ppm	(-CH_2-*C*H_2-CH_3)
		35.05 ppm	(-*C*H_2-CH_2-CH_3)
		47.40 ppm	(-*C*H-CH_2-CH_2-CH_3)
		64.92 ppm	(-NH-*C*H_2-OH)
		177.96 ppm	(-*C*(O)-N-)
		(Figure 83)	
EIMS	:	174 (2.38%), 156 (3.67%), 144 (8.90%), 113 (76.00%),	
		101 (42.00%), 72 (82.00%), 57 (100.00%)	
		(Figure 84)	

N-Acetoxymethyl-2-propylpentamide (CU 763-15-10)

A mixture of 2-propylpentamide (1.7 g, 10 mmol), acetic anhydride (4 g, 4 ml, 10 mmol) and pyridine (0.8 g, 0.8 ml) was stirred at room temperature for 4 hours and then concentrated in vacuo. The residue was taken up in ethyl acetate (30 ml) and water(30 ml). The organic phase was washed with 1 M HCl, 5% aqueous sodium bicarbonate, and water. dried over anhydrous sodium sulfate, and evaporated under reduced pressure. The residue obtained was recrystallized from hexane and was unstable upon standing.

IR	:	3307 cm^{-1}	(ν N-H)
(KBr)		2958-2870 cm^{-1}	(ν C-H, aliphatic)
		1744 cm^{-1}	(ν C=O, ester)
		1673 cm^{-1}	(ν C=O, amide)
		1537 cm^{-1}	(δ N-H)
		1200 cm^{-1}	(ν C-C(=O)-O)
		1017 cm^{-1}	(ν -O-C-C-)
		(Figure 85)	
^{1}H-NMR	:	0.80 ppm	(6H, t, -CH_3)
($CDCl_3$)		1.12-1.38 ppm	(6H, m, -CH_2-CH_2-CH_3)
		1.50 ppm	(2H, m, -CH_2-CH_2-CH_3)
		1.98 ppm	(3H, s, -C(O)-CH_3)
		2.06 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		5.18 ppm	(2H, d, -NH-CH_2-O)
		7.02 ppm	(1H, t, -C(O)-N*H*-)
		(Figure 86)	

N-Methoxymethyl-2-propylpentamide (CU 763-15-11)

Sodium hydride (0.4 g, 8 mmol) was suspended in dry tetrahydrofuran (15 ml) in a 100-ml two-neck round bottom flask placing in an ice bath. A clear solution of 2-propylpentamide (1.1 g, 8 mmol) in 20 ml dried tetrahydrofuran was added dropwise to the mixture through a dropping funnel. The mixture was stirred until hydrogen gas ceased. Methoxymethyl chloride (0.6 g, 8 mmol) was added. Stirring was continued in an ice bath for 2 hours. The salt generated in the reaction was removed by filtration and washed with dried tetrahydrofuran. The combined filtrate and washings was evaporated to dryness. The residual white solid was purified by column chromatography with dichlorometrhane: ethyl acetate (4:1) as eluent yielded 0.6 g (41%) white solid of N-methoxymethyl-2-propylpentamide, m.p.51-52°C.

Anal.calcd. for $C_{10}H_{21}NO_2$: C, 64.130: H, 11.303: N, 7.480

Found : C, 64.149: H, 11.290: N, 7.485

IR	:	3298 cm^{-1}	(ν N-H)
(KBr)		2959-2870 cm^{-1}	(ν C-H, aliphatic)
		1655 cm^{-1}	(ν C=O, amide)
		1544 cm^{-1}	(δ N-H)
		1069 cm^{-1}	(ν C-O)
		(Figure 87)	
^{1}H-NMR	:	0.89 ppm	(6H, t, -C*H*$_3$)
($CDCl_3$)		1.20-1.46 ppm	(6H, m, -C*H*$_2$-C*H*$_2$-CH_3)
		1.60 ppm	(2H, m, -C*H*$_2$-CH_2-CH_3)
		2.08 ppm	(1H, m, -C*H*-CH_2-CH_2-CH_3)
		3.34 ppm	(1H, s, -OC*H*$_3$)

	4.70 ppm	(2H, d, -NH-C*H*$_2$-O)
	6.06 ppm	(1H, broad, -C(O)-N*H*-)
	(Figure 88)	
^{13}C-NMR :	14.07 ppm	(-CH_2-CH_2-*C*H_3)
($CDCl_3$)	20.78 ppm	(-CH_2-*C*H_2-CH_3)
	35.11 ppm	(-*C*H_2-CH_2-CH_3)
	47.89 ppm	(-*C*H-CH_2-CH_2-CH_3)
	56.07 ppm	(-O-*C*H_3)
	71.25 ppm	(-NH-*C*H_2-OH)
	176.83 ppm	(-*C*(O)-N-)
	(Figure 89)	
EIMS :	188 (26.40%), 172 (38.80%), 156(54.00%), 145 (86.40%),	
	127 (94.80%), 113 (100.00%), 99 (88.00%), 84 (97.60%),	
	57 (91.60%)	
	(Figure90)	

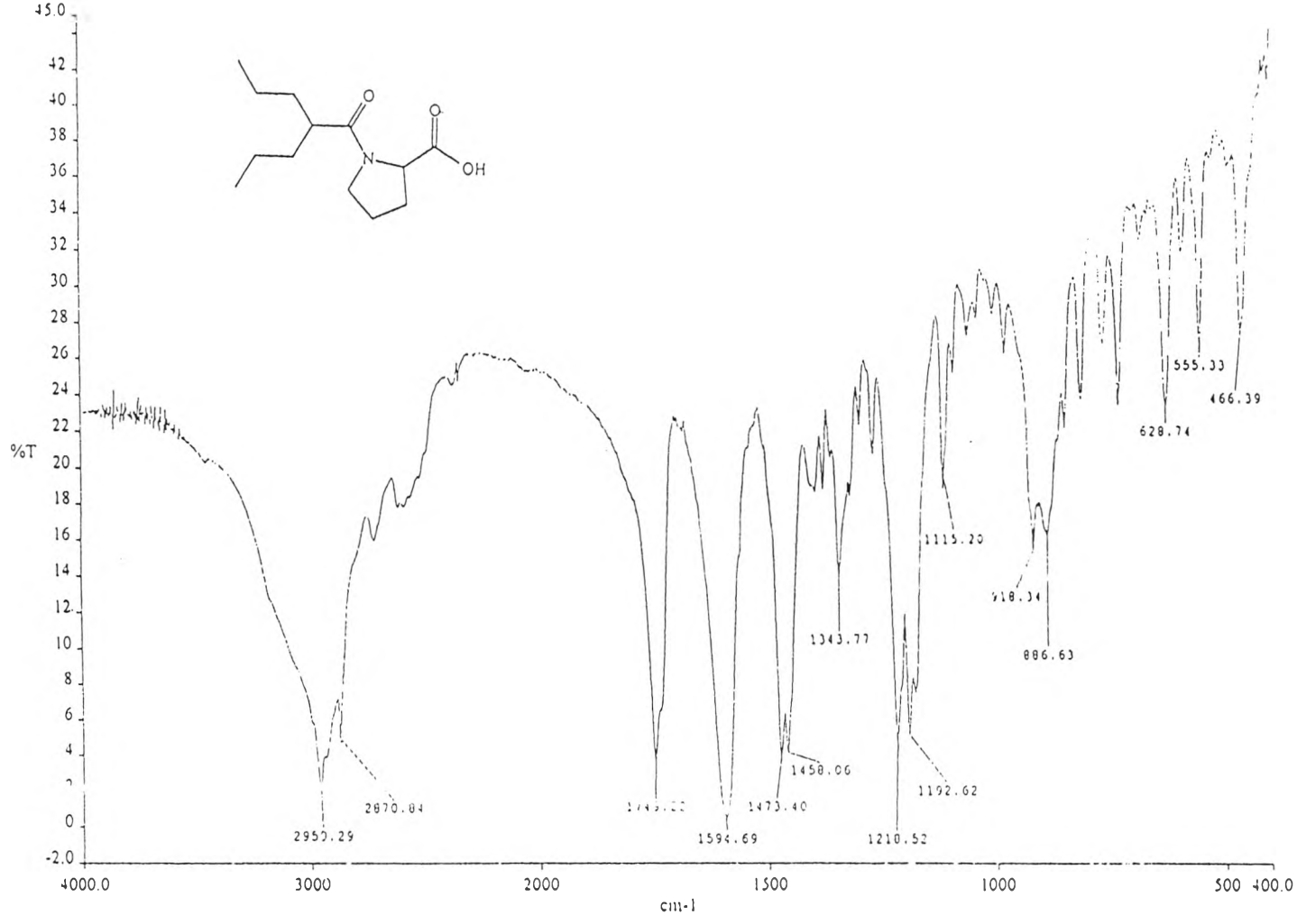

Figure 18. The IR spectrum (KBr) of N-(2-propylpentanoyl)-L-proline

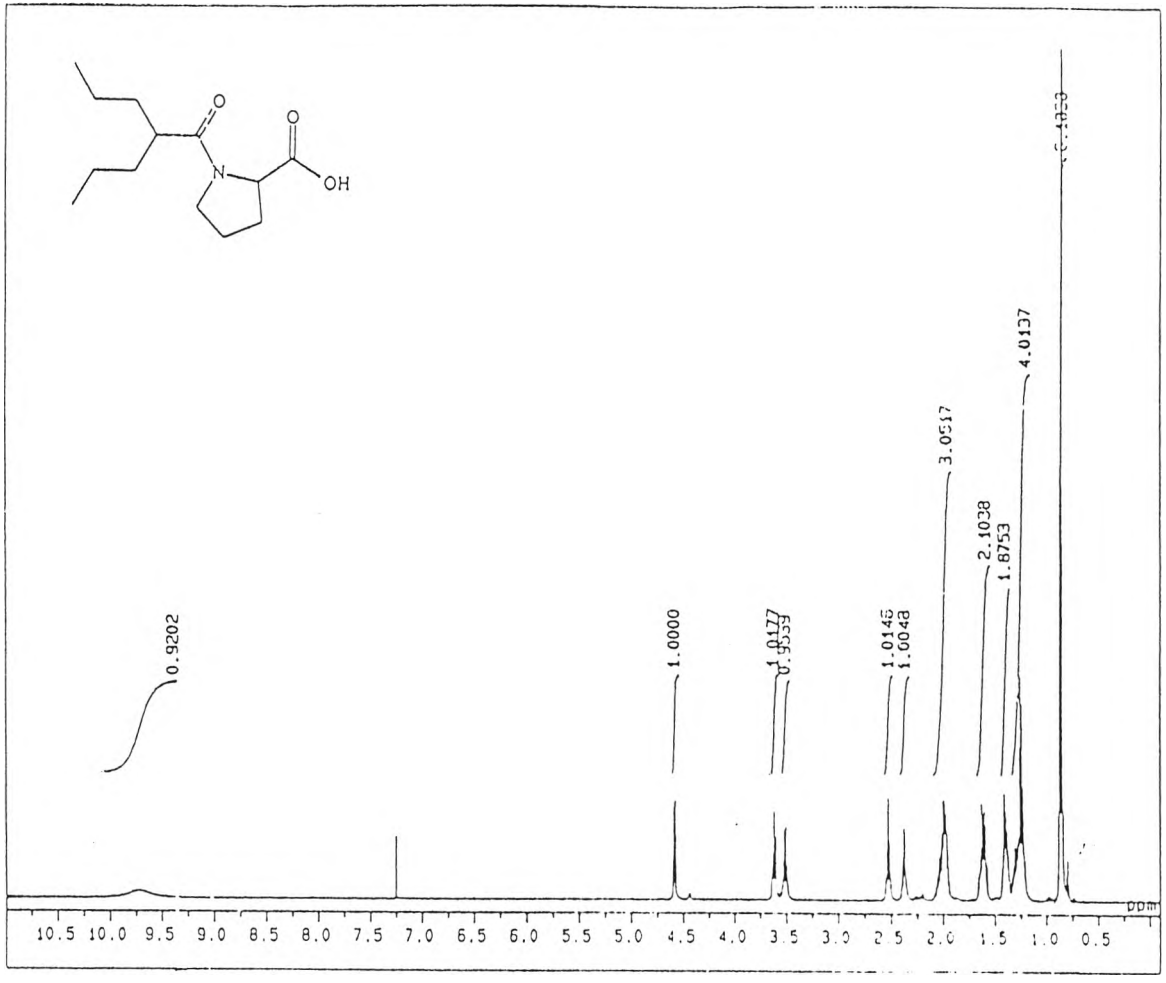

Figure 19. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$

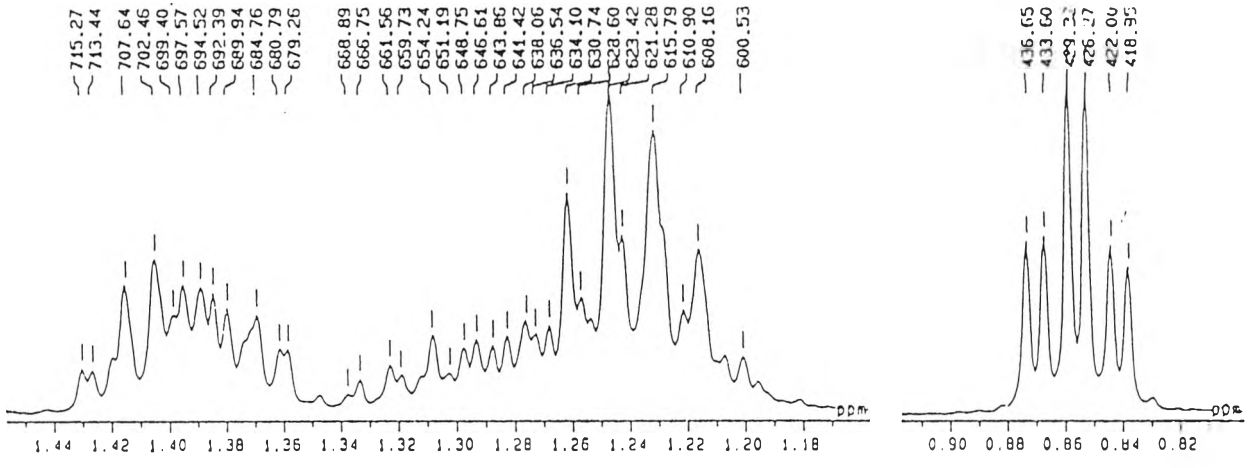

Figure 20. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$ (Enlarged scale)

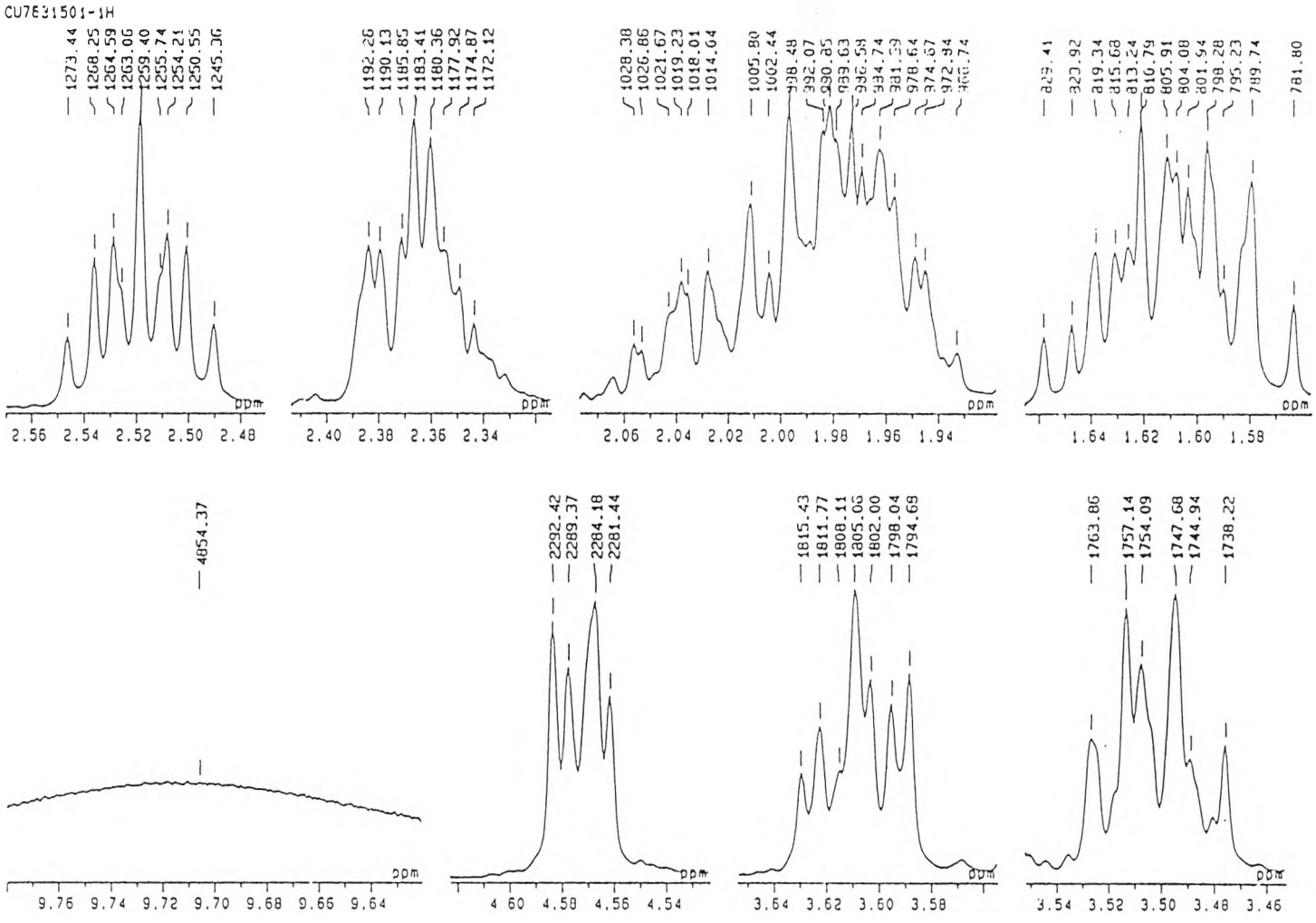

Figure 21. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$ (Enlarged scale)

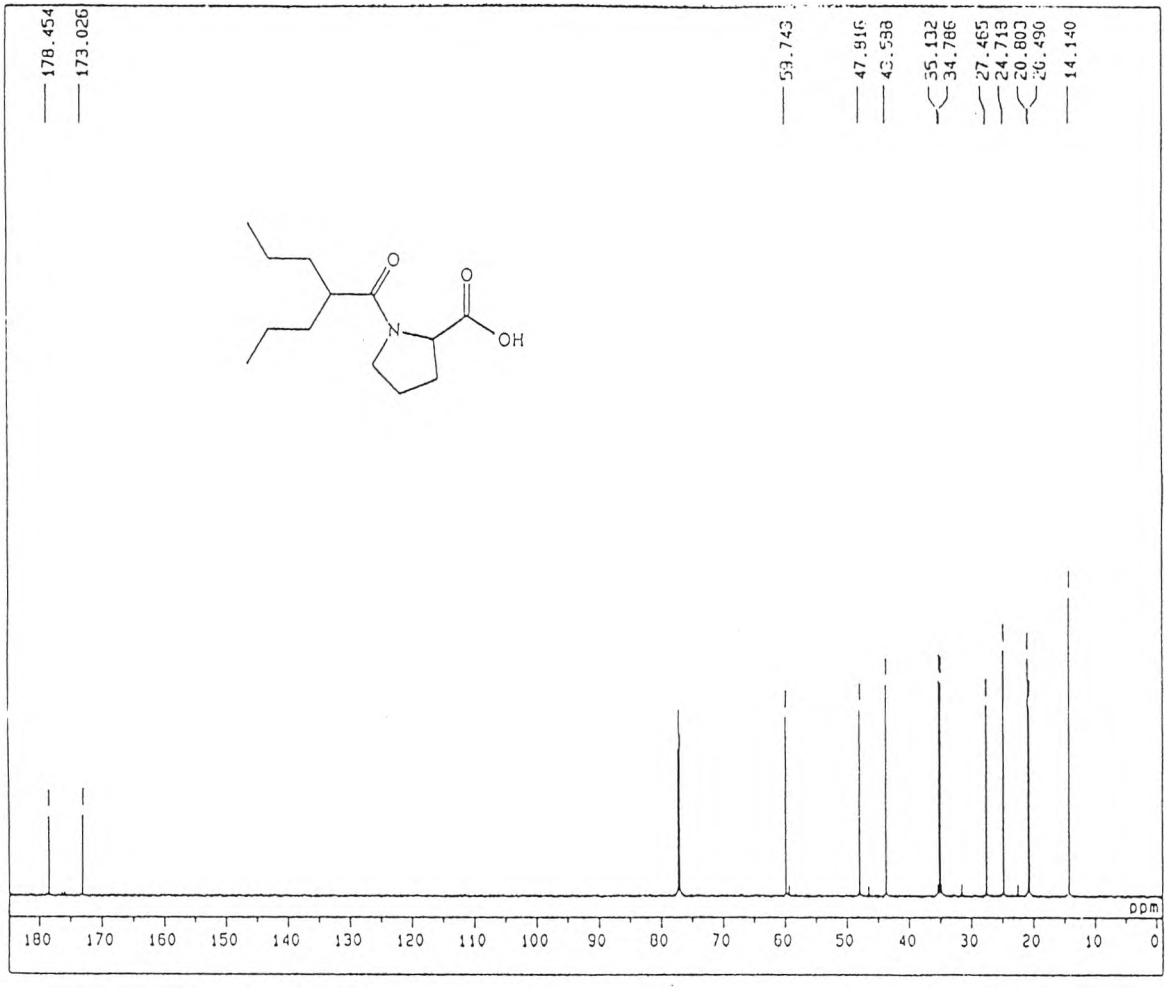

Figure 22. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$

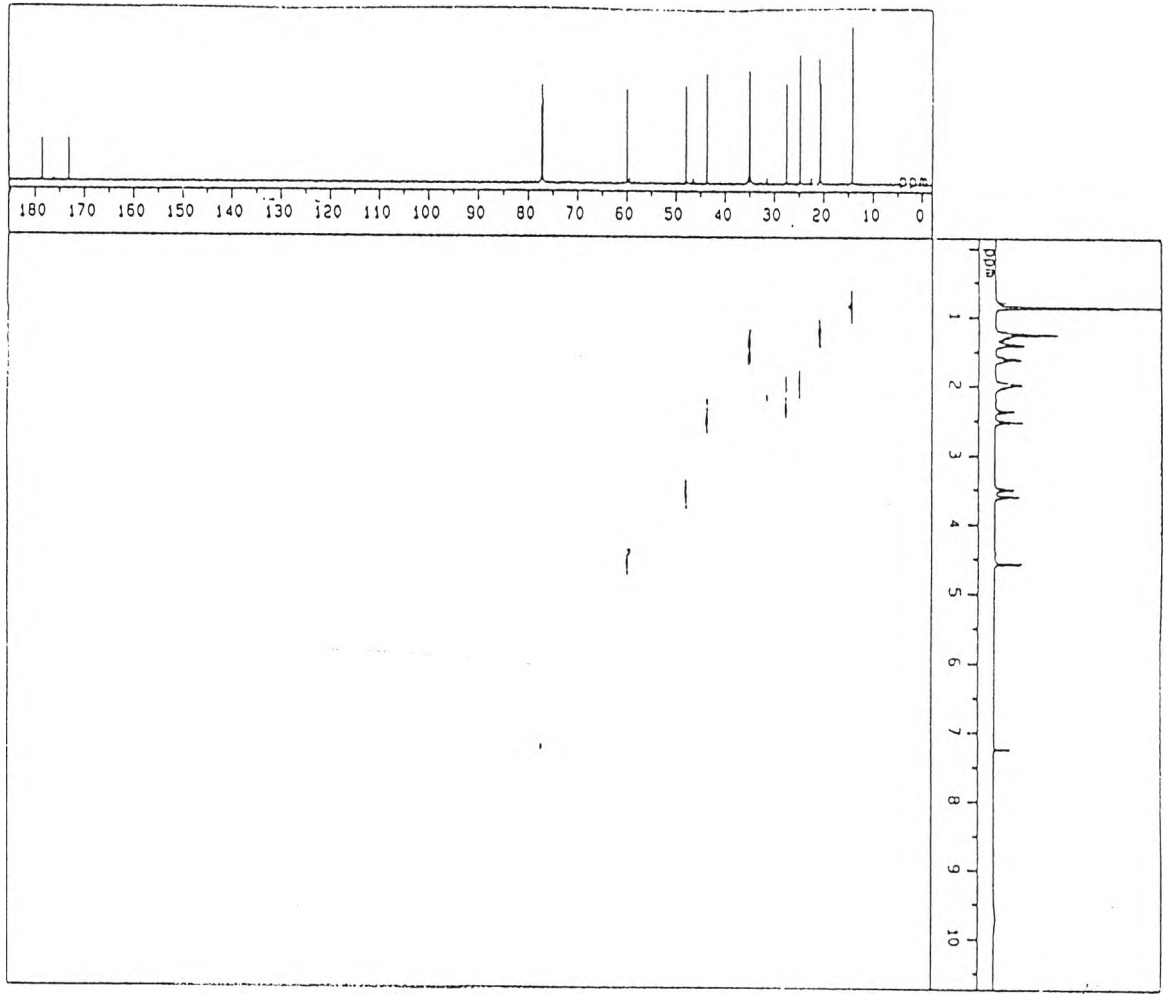

Figure 23. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$

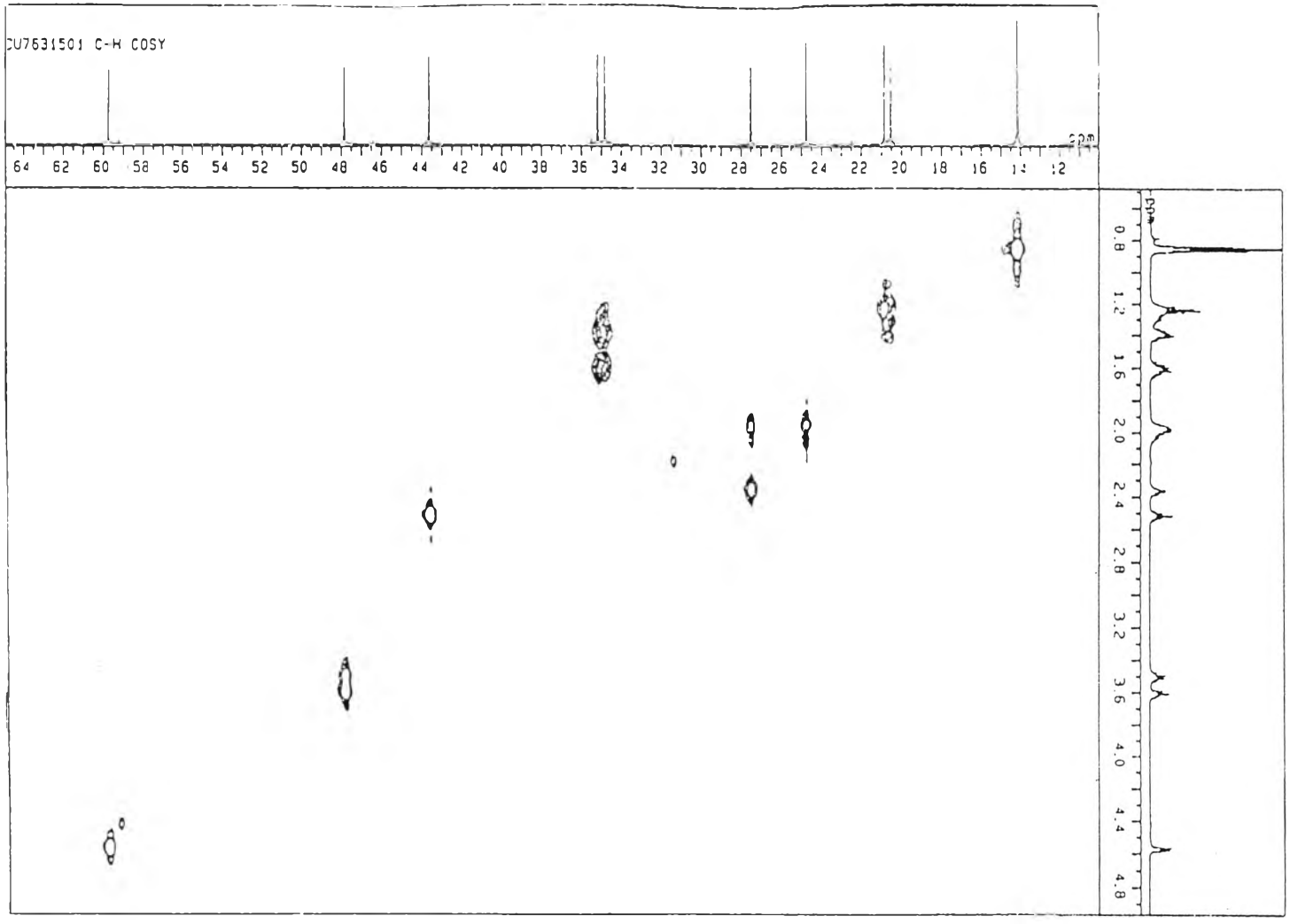

Figure 24. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline in $CDCl_3$ (Enlarged scale)

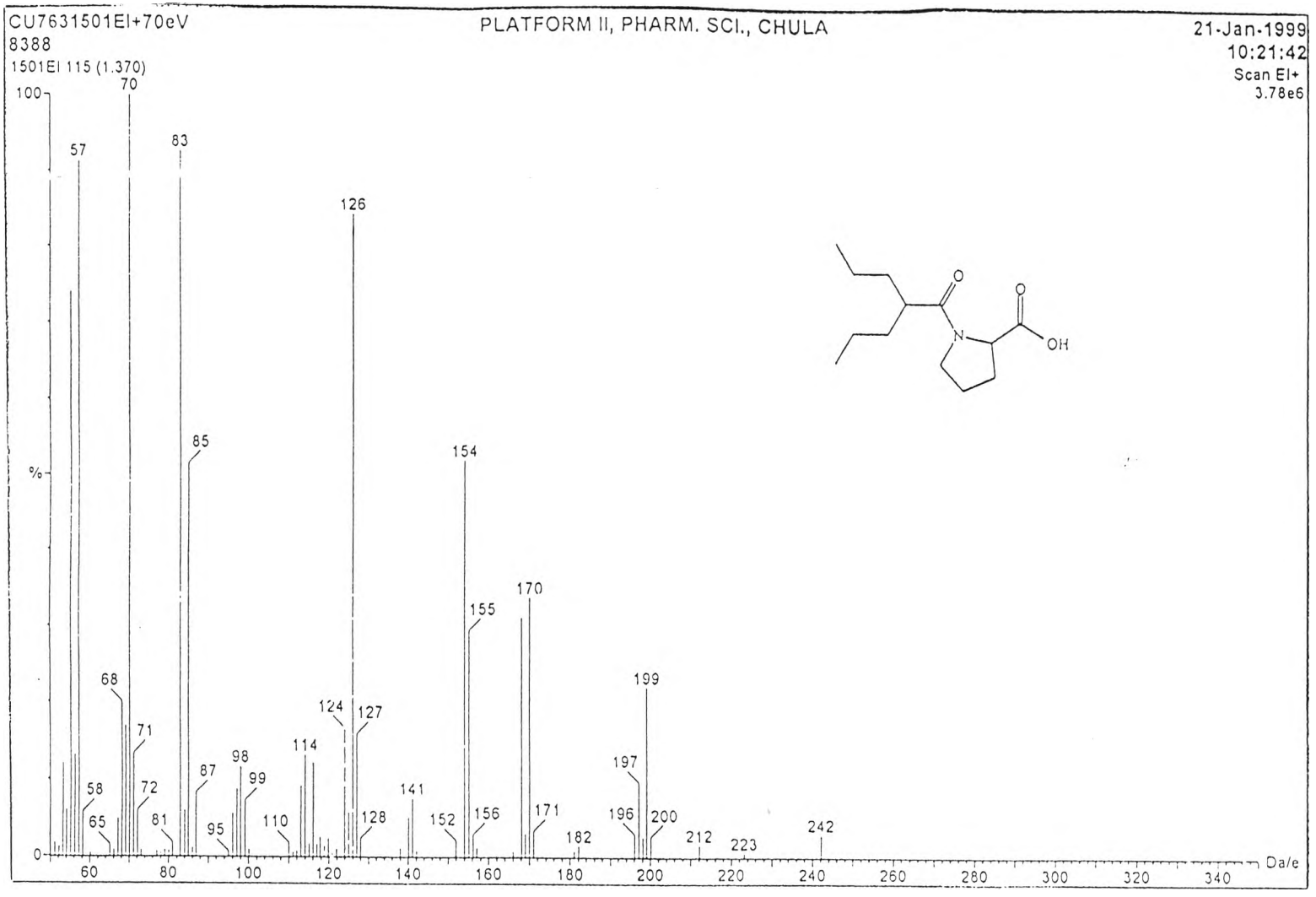

Figure 25. The mass spectrum of N-(2-propylpentanoyl)-L-proline

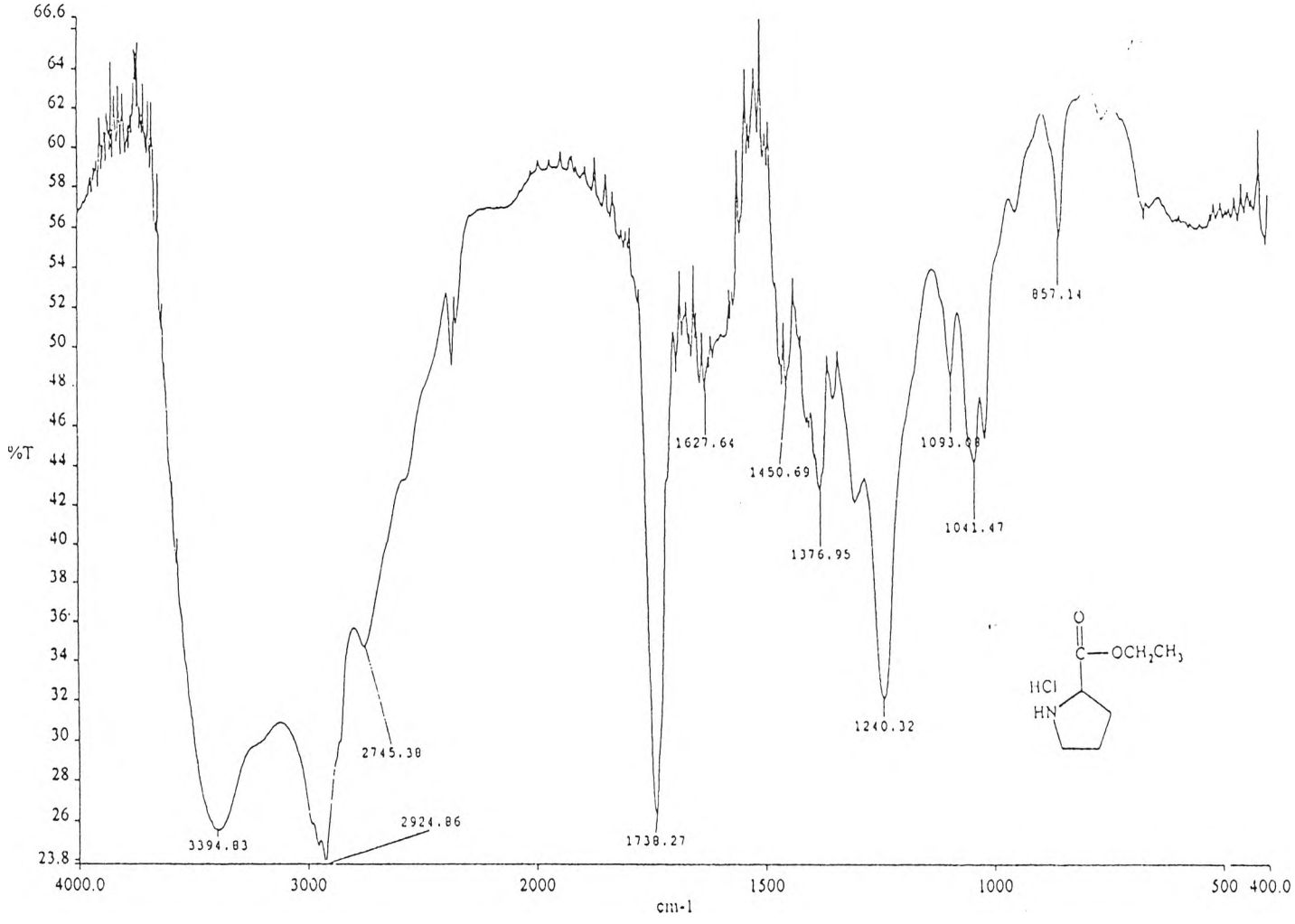

Figure 26. The IR spectrum (Neat) of L-proline ethyl ester hydrochloride

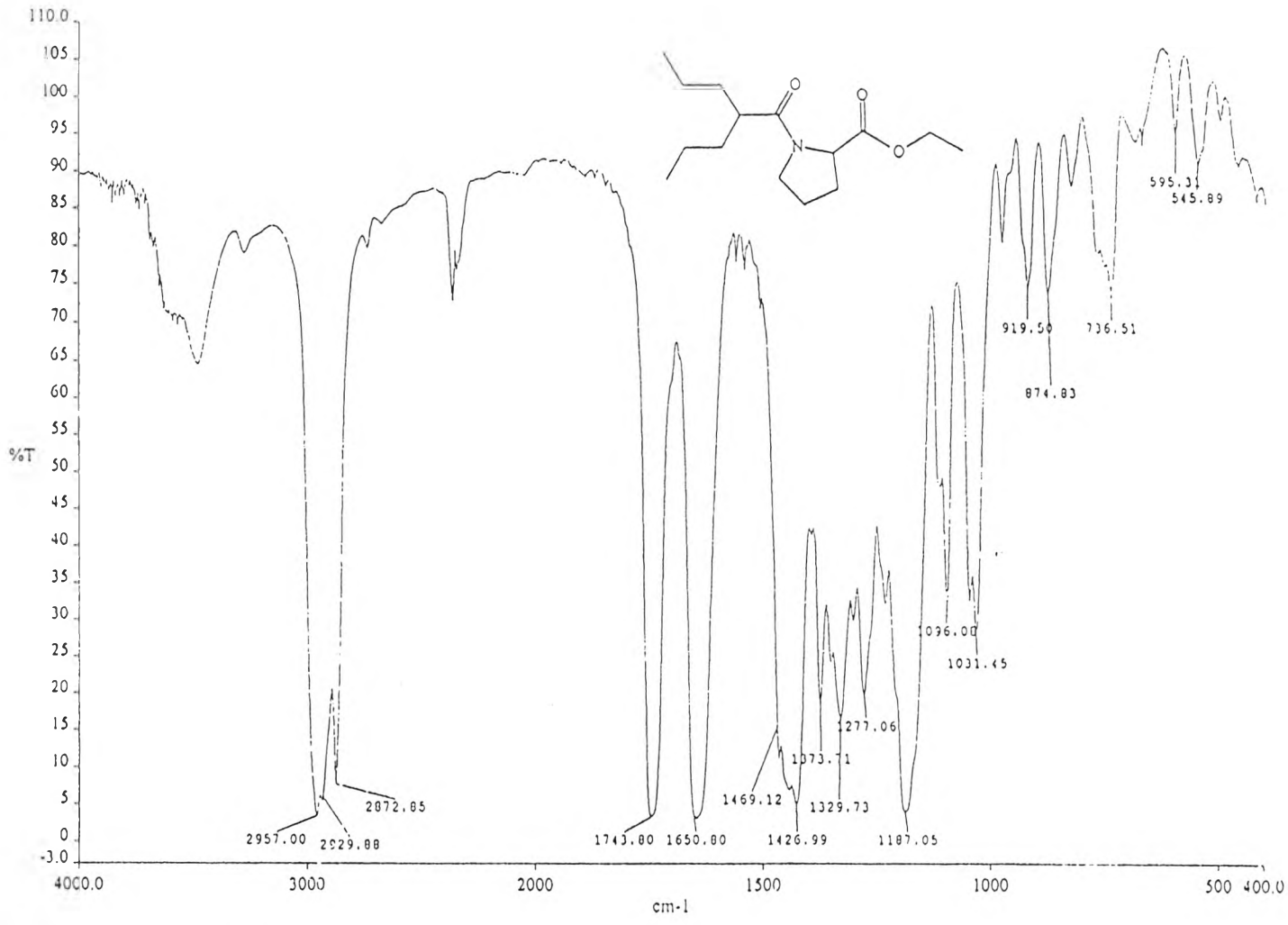

Figure 27. The IR spectrum (Neat) of N-(2-propylpentanoyl)-L-proline ethyl ester

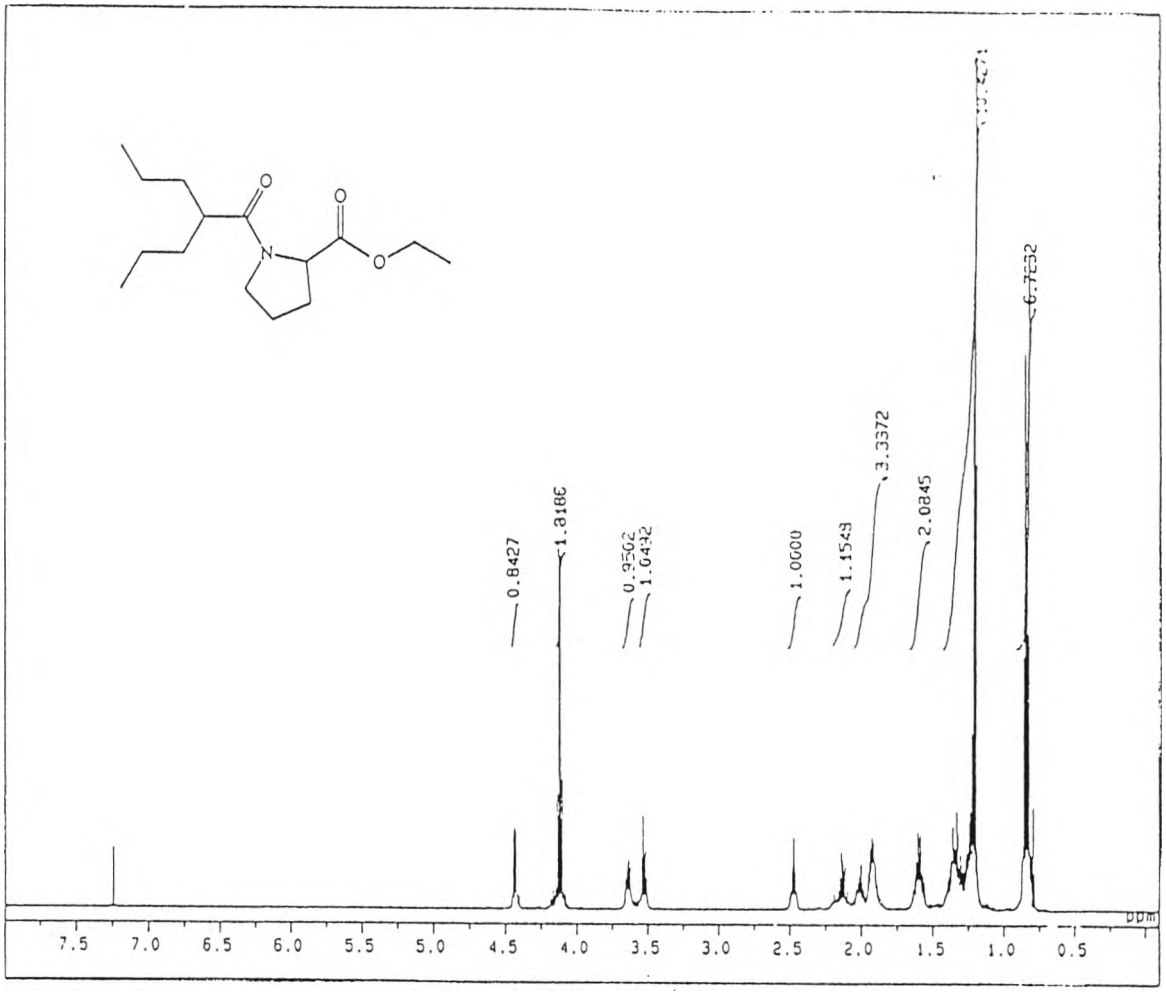

Figure 28. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$

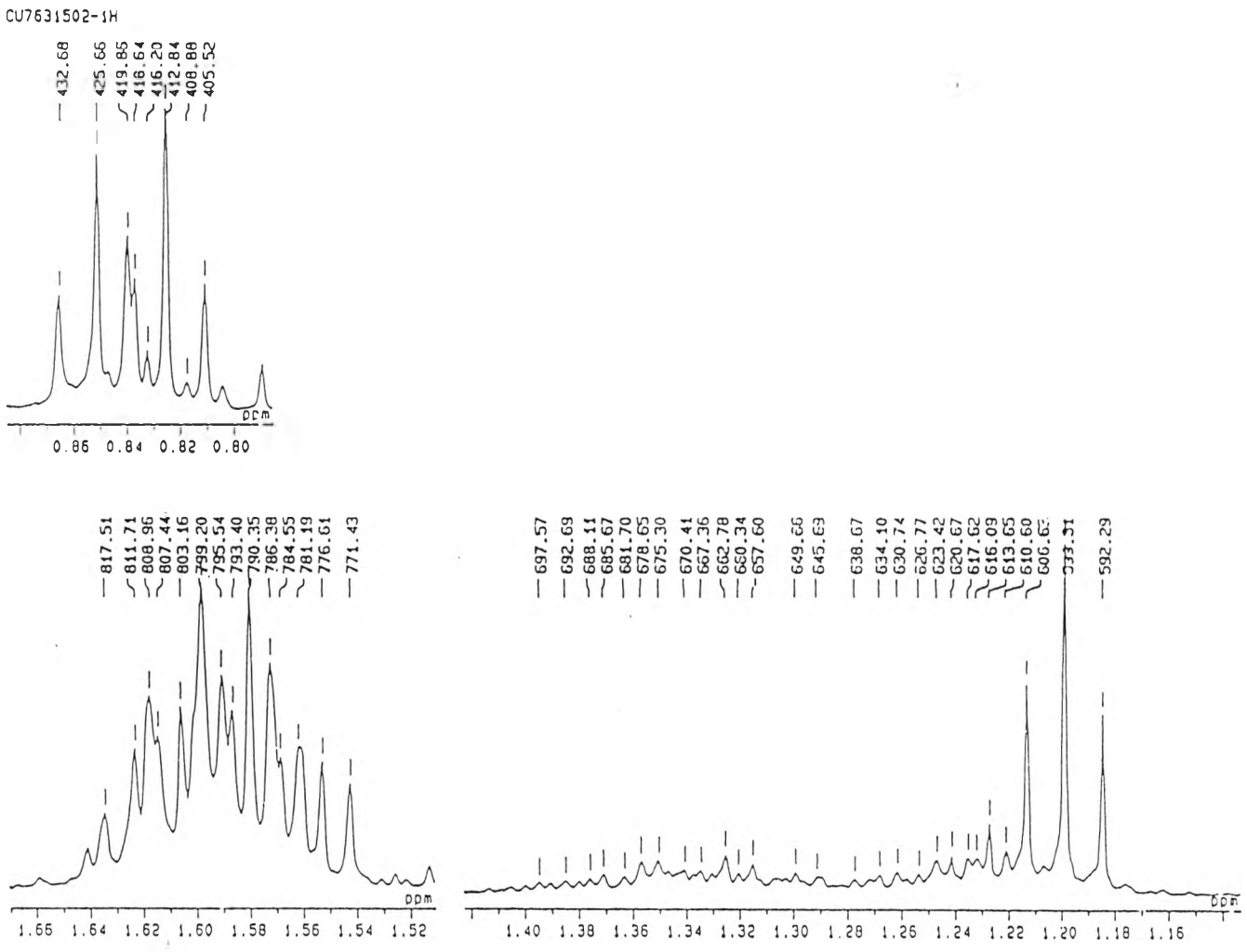

Figure 29. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$(Enlarged scale)

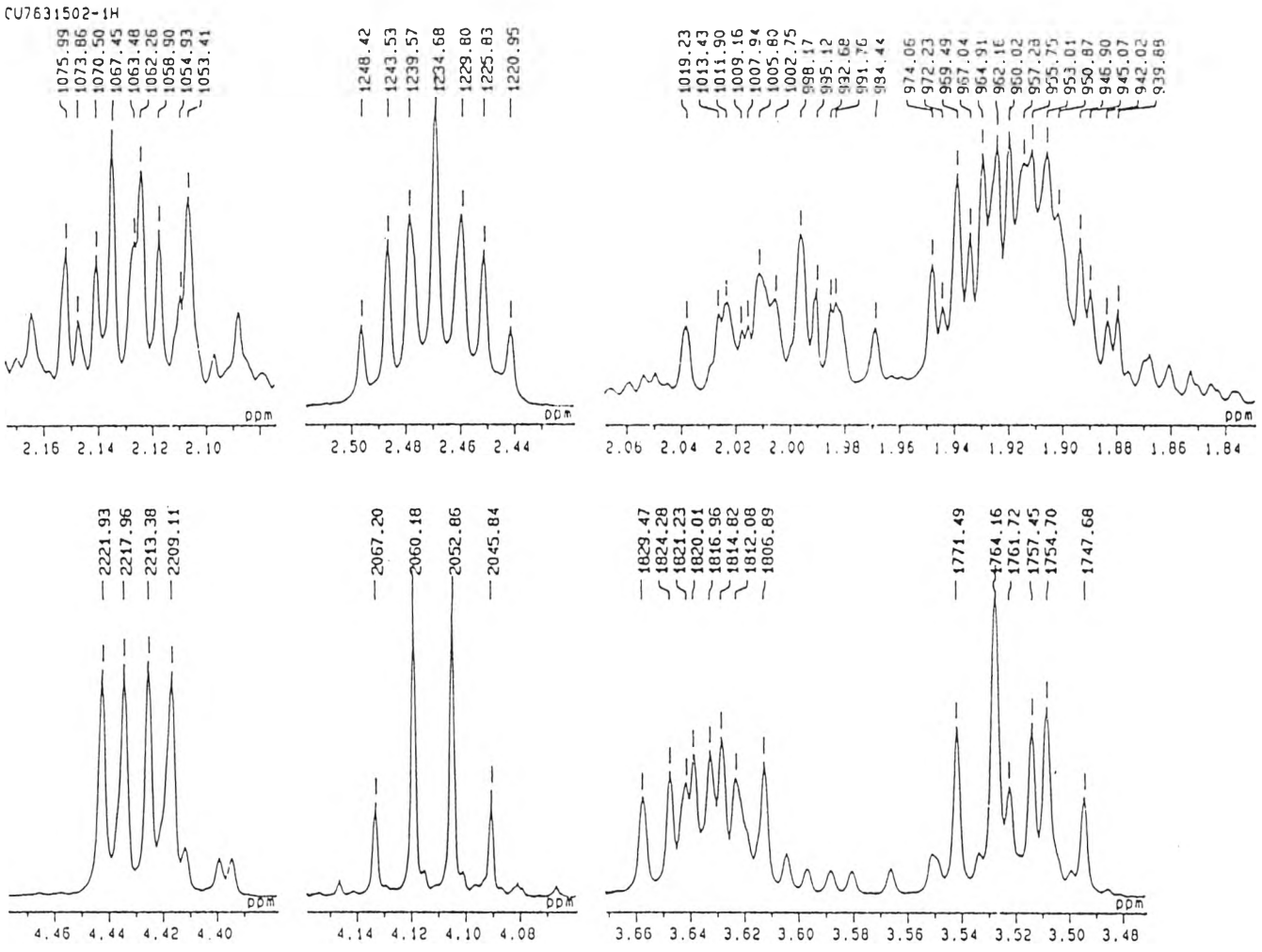

Figure 30. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$ (Enlarged scale)

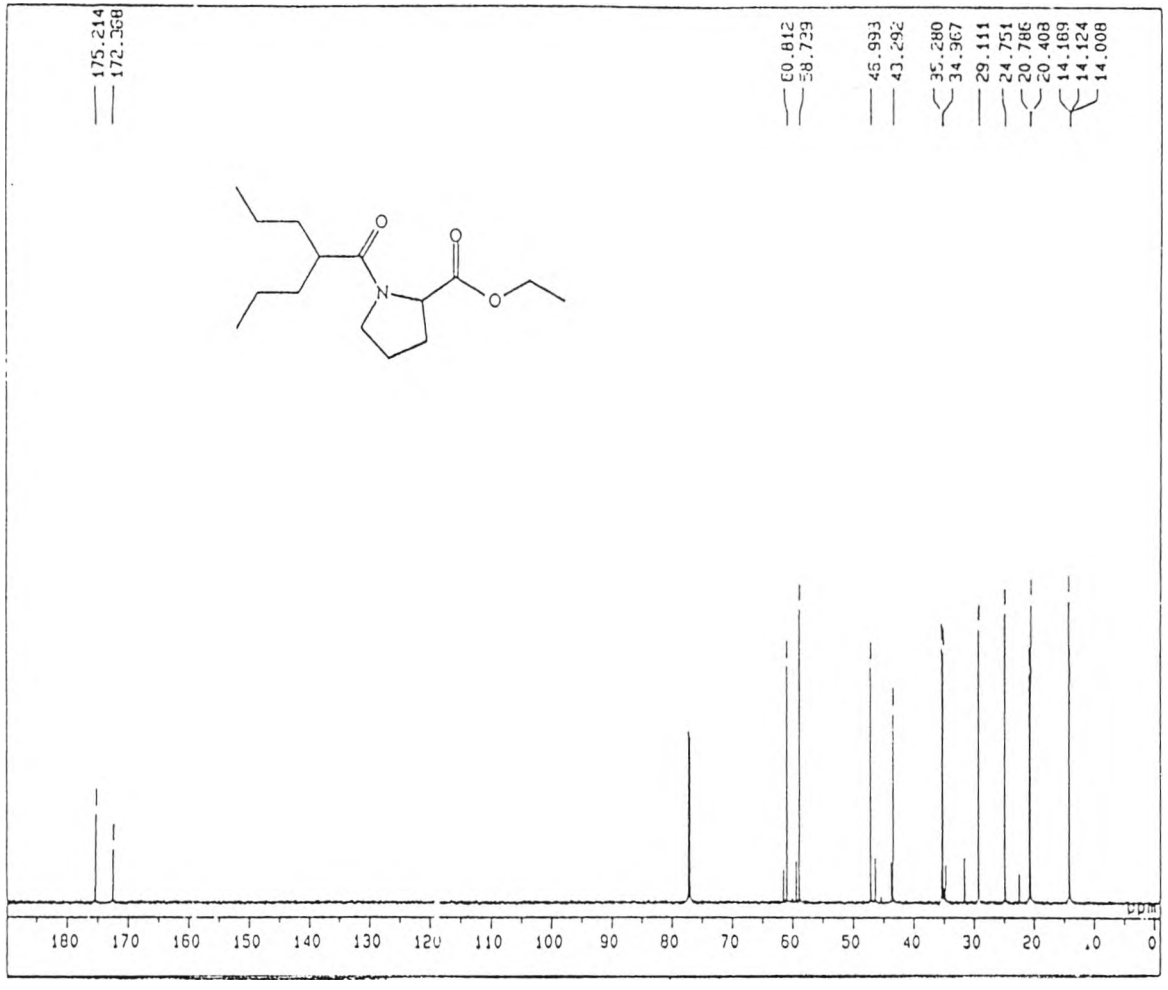

Figure 31. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$

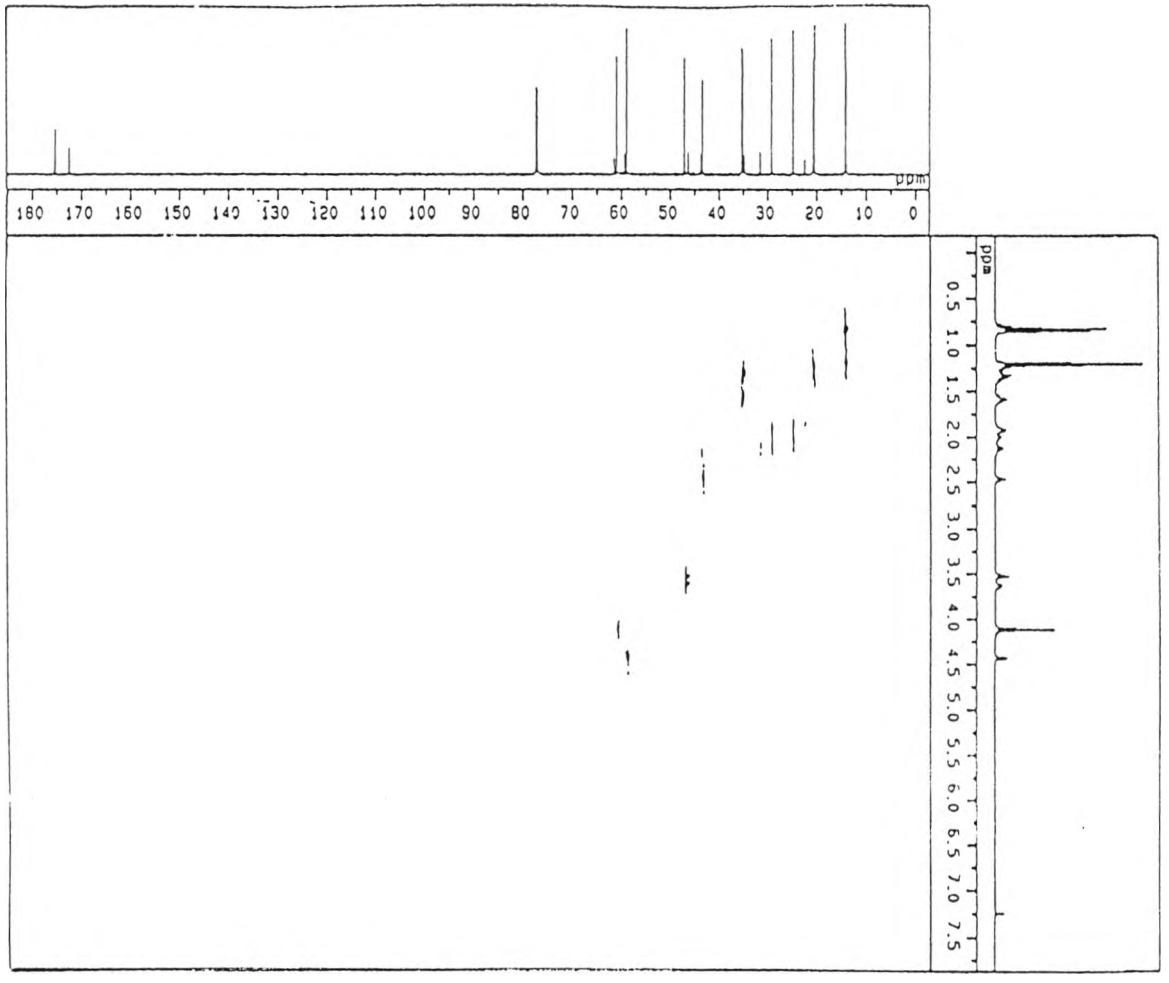

Figure 32. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$

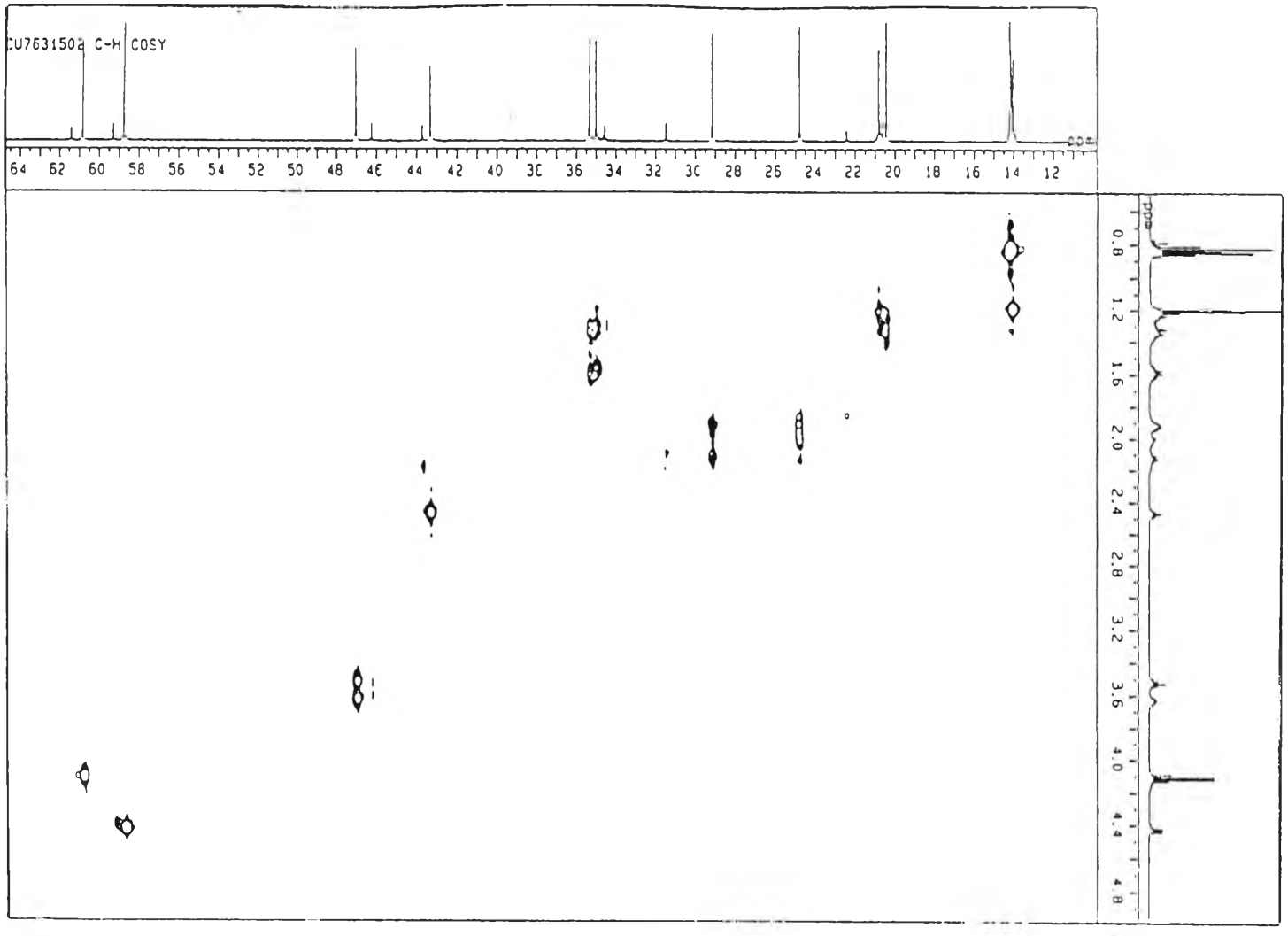

Figure 33. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester in $CDCl_3$ (Enlarged scale)

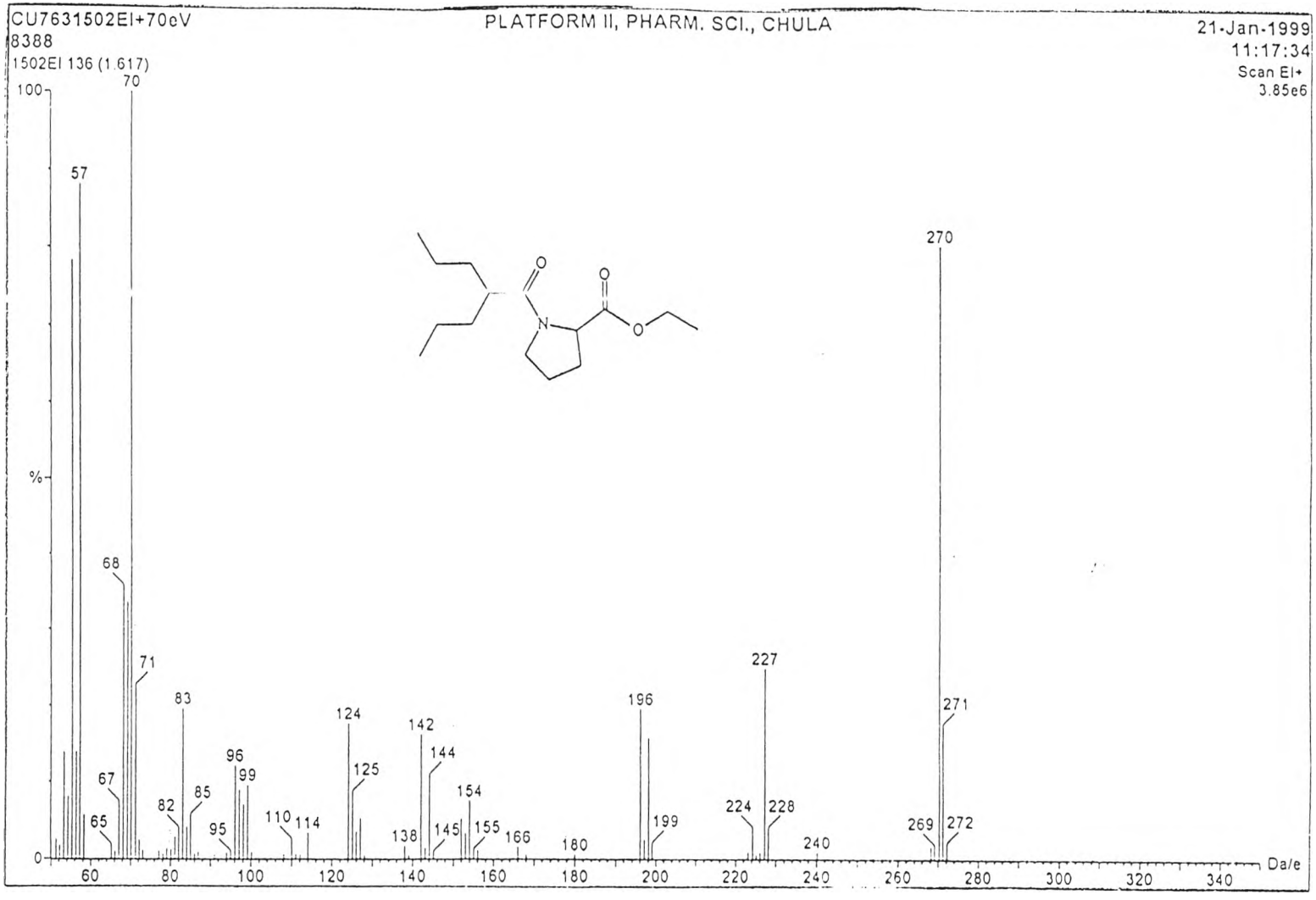

Figure 34. The mass spectrum of N-(2-propylpentanoyl)-L-proline ethyl ester

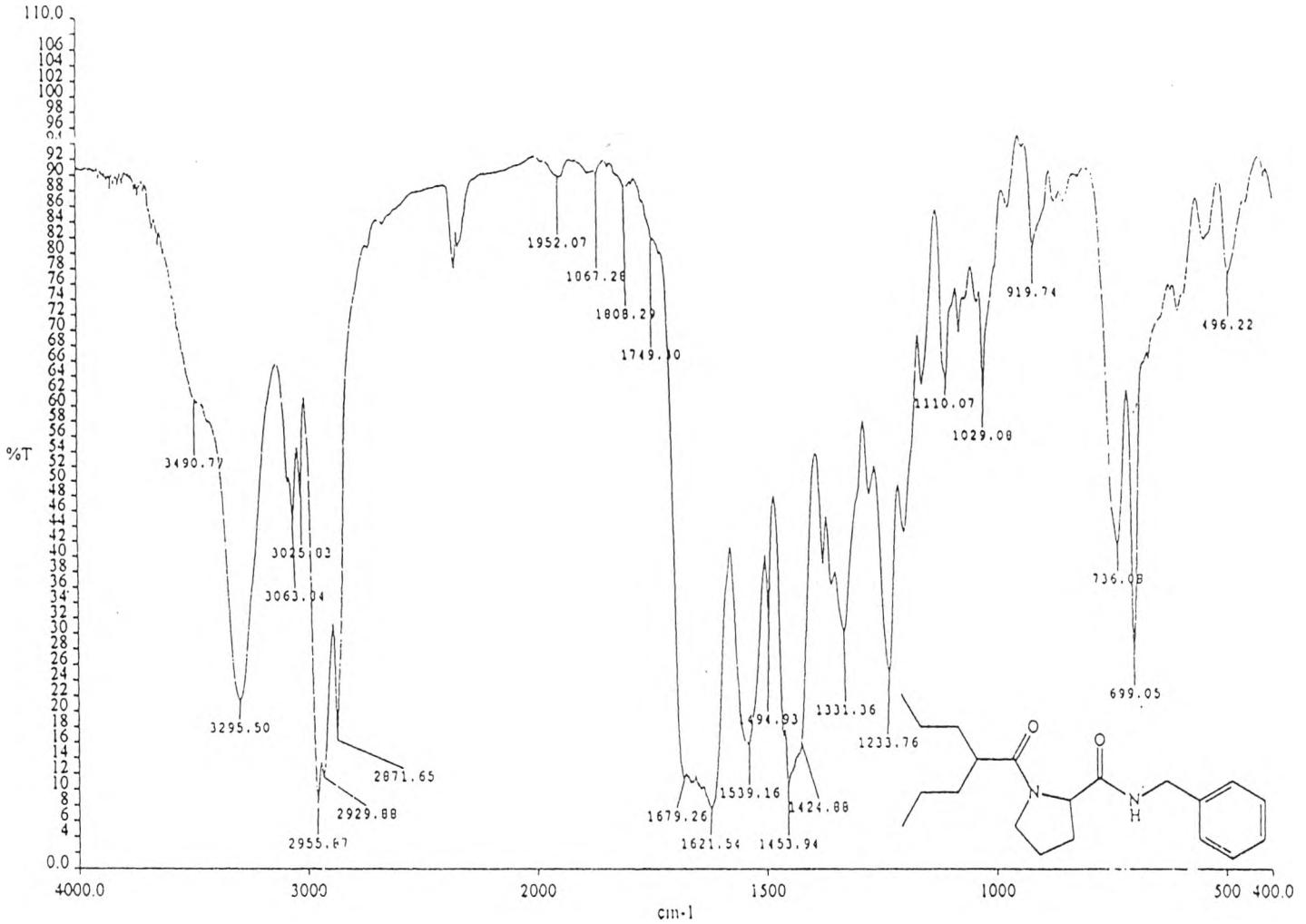

Figure 35. The IR spectrum (Neat) of N-(2-propylpentanoyl)-L-proline benzylamide

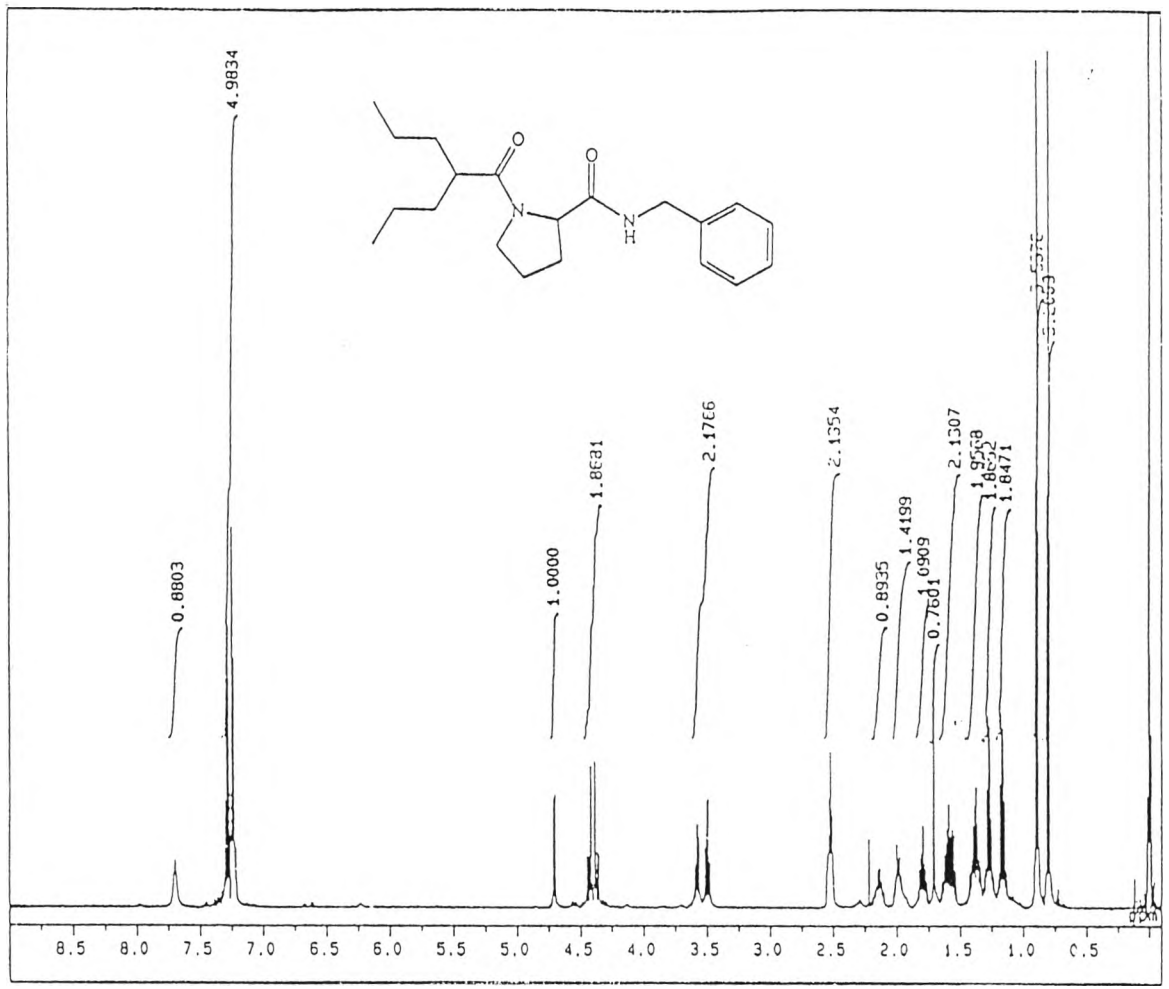

Figure 36. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$

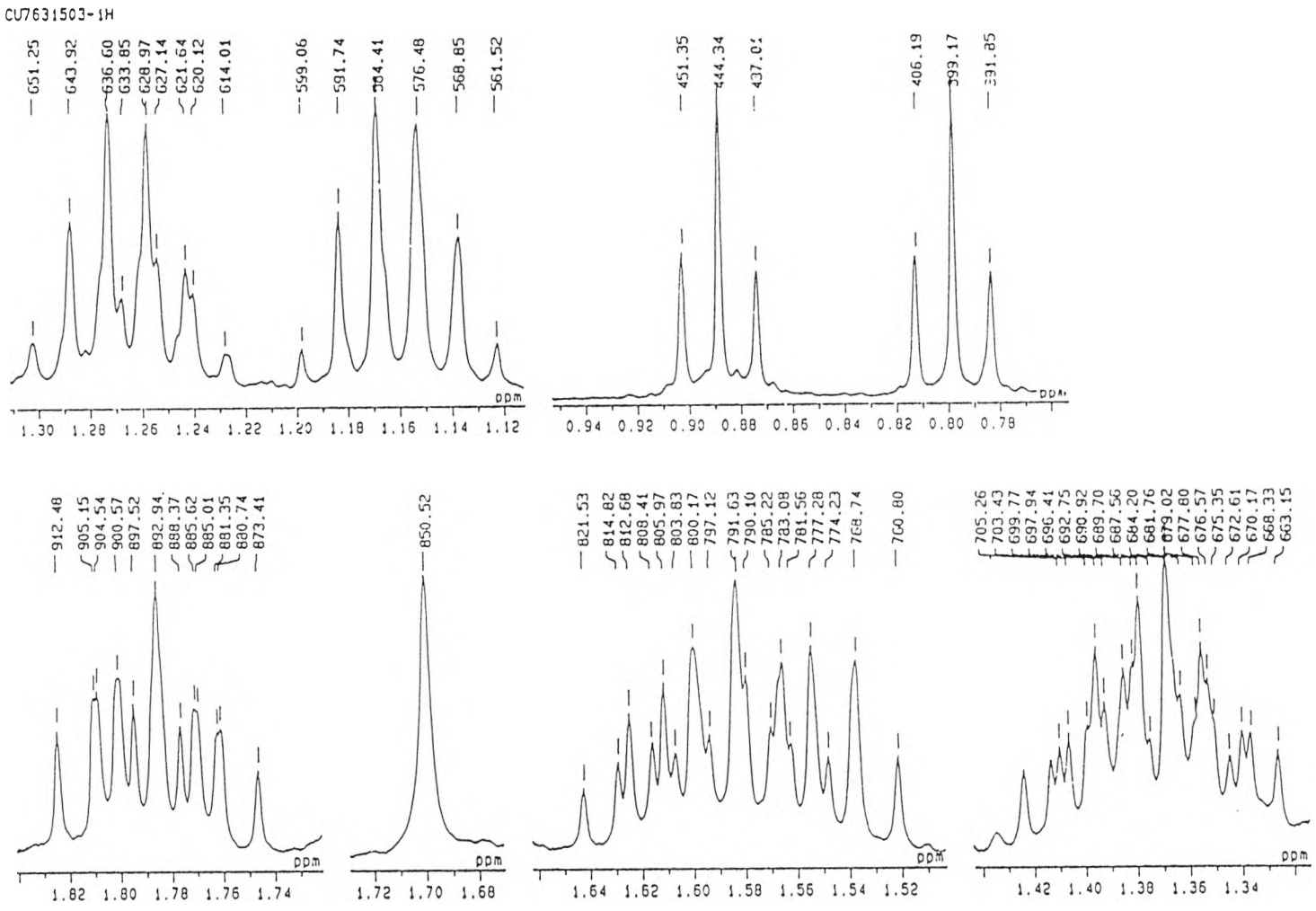

Figure 37. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$(Enlarged scale)

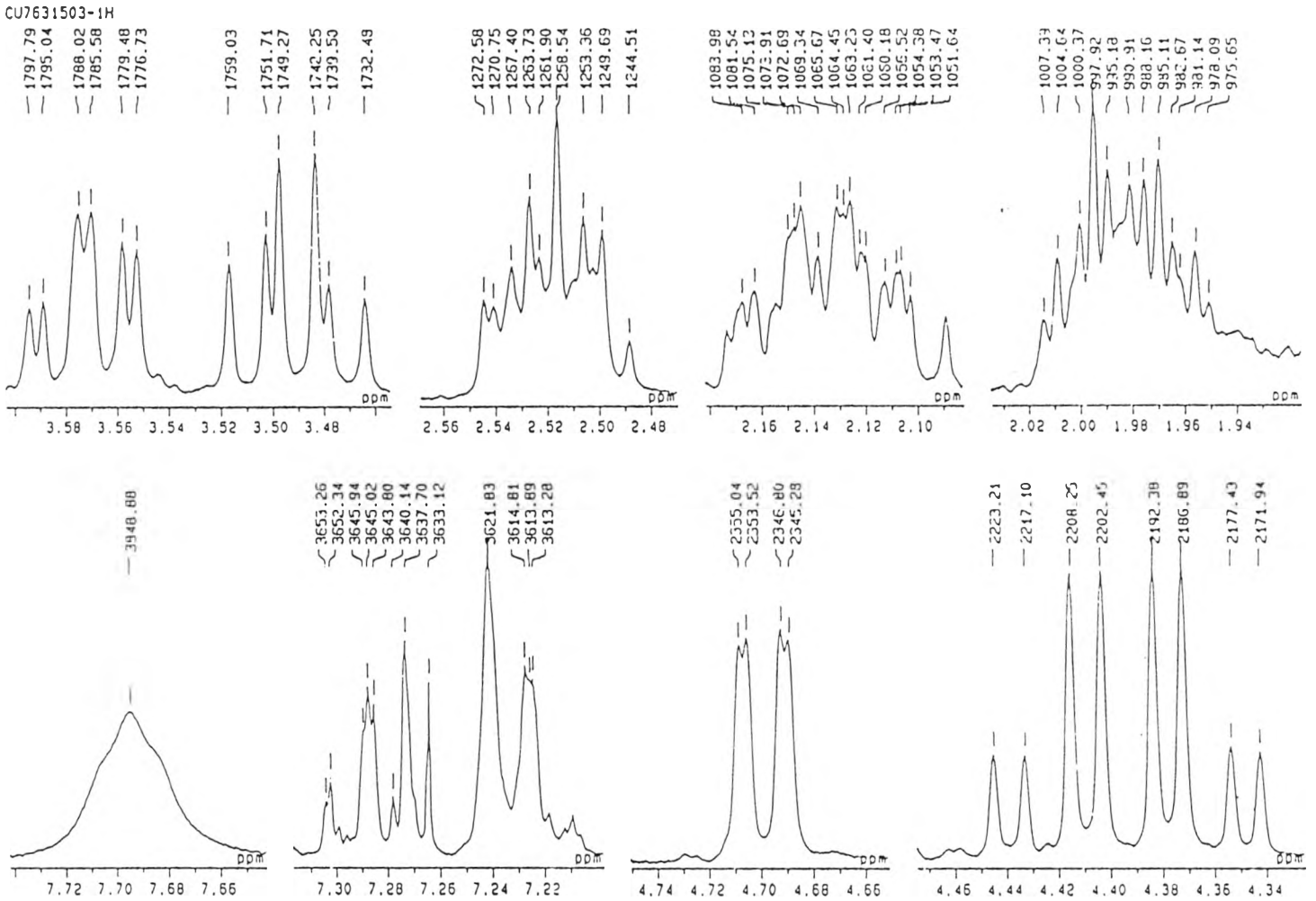

Figure 38. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$ (Enlarged scale)

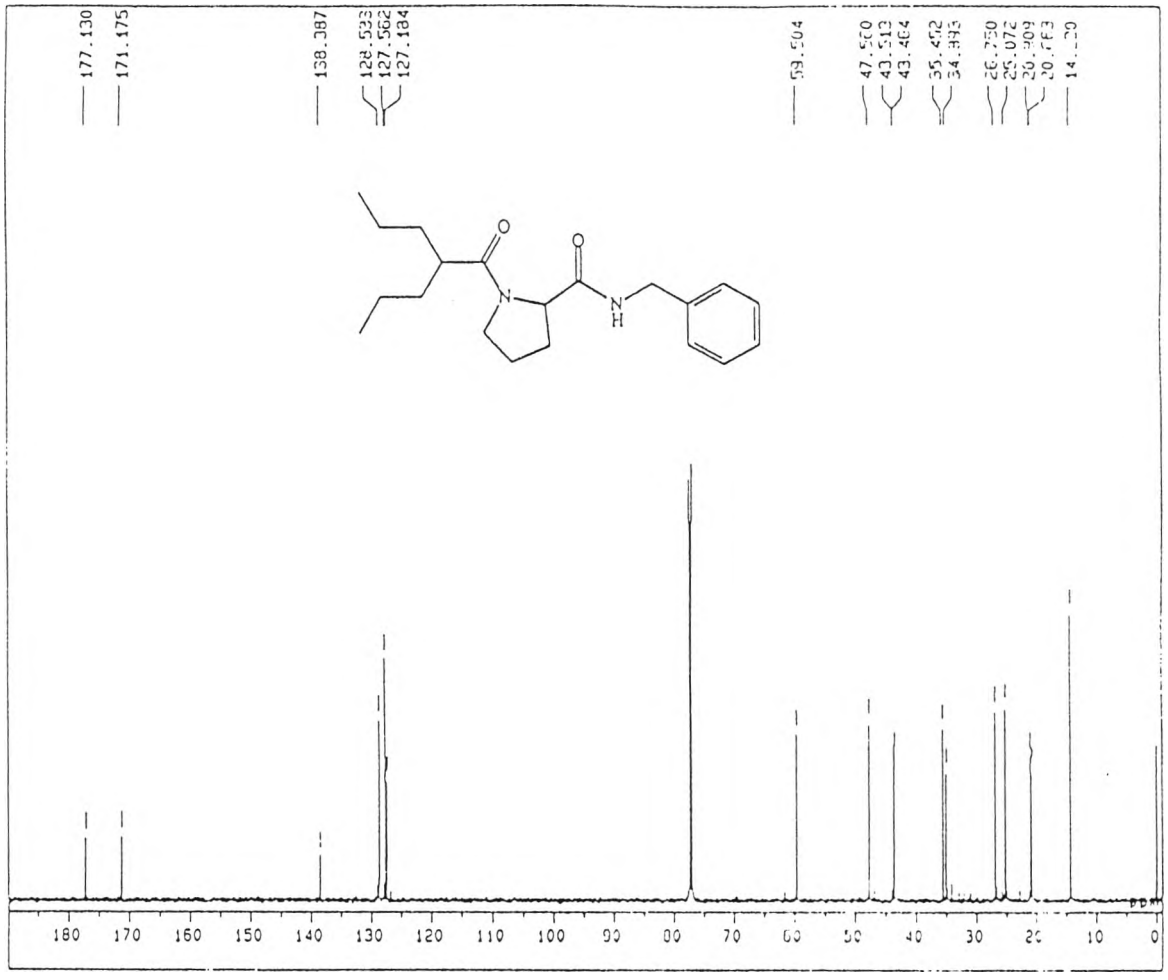

Figure 39. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$

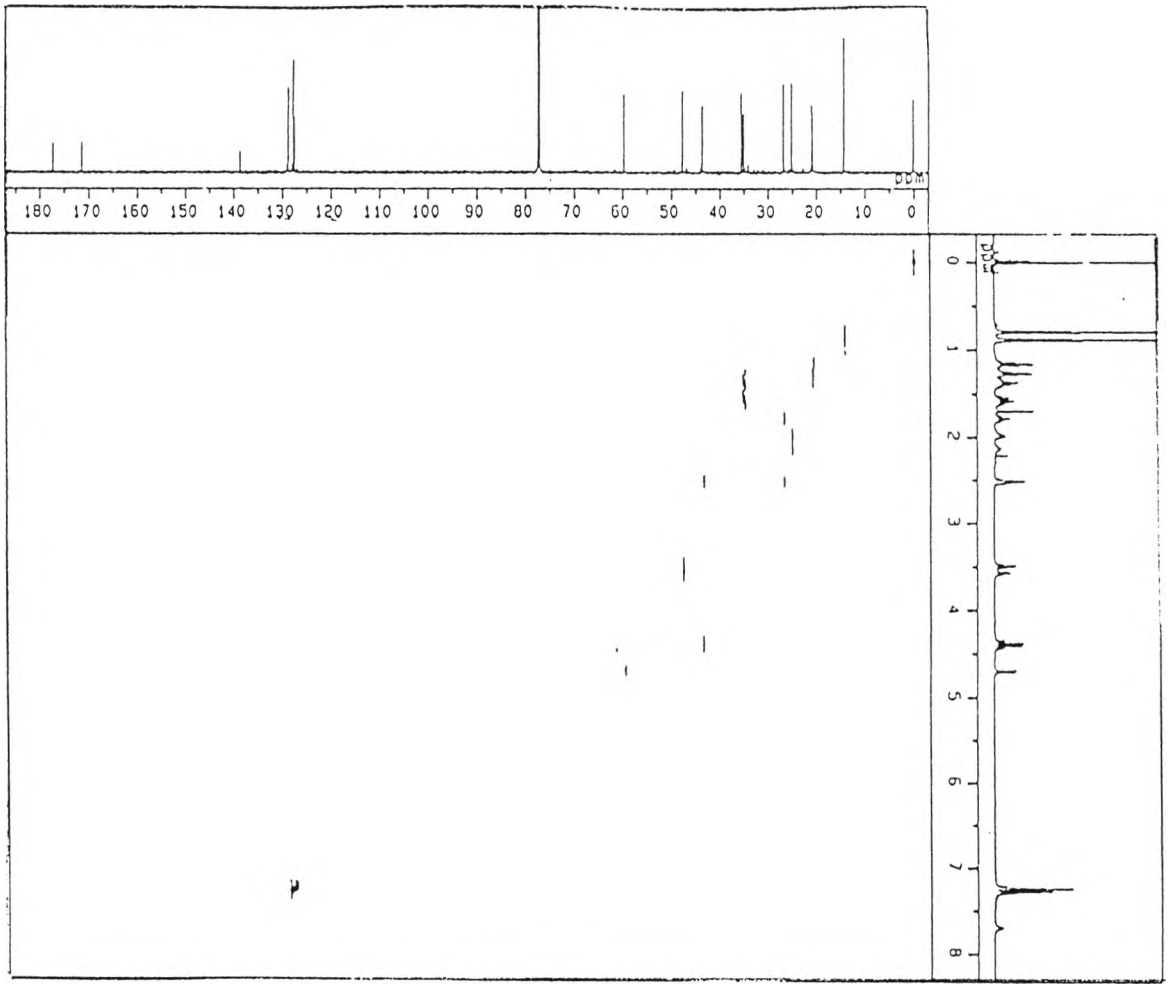

Figure 40. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$

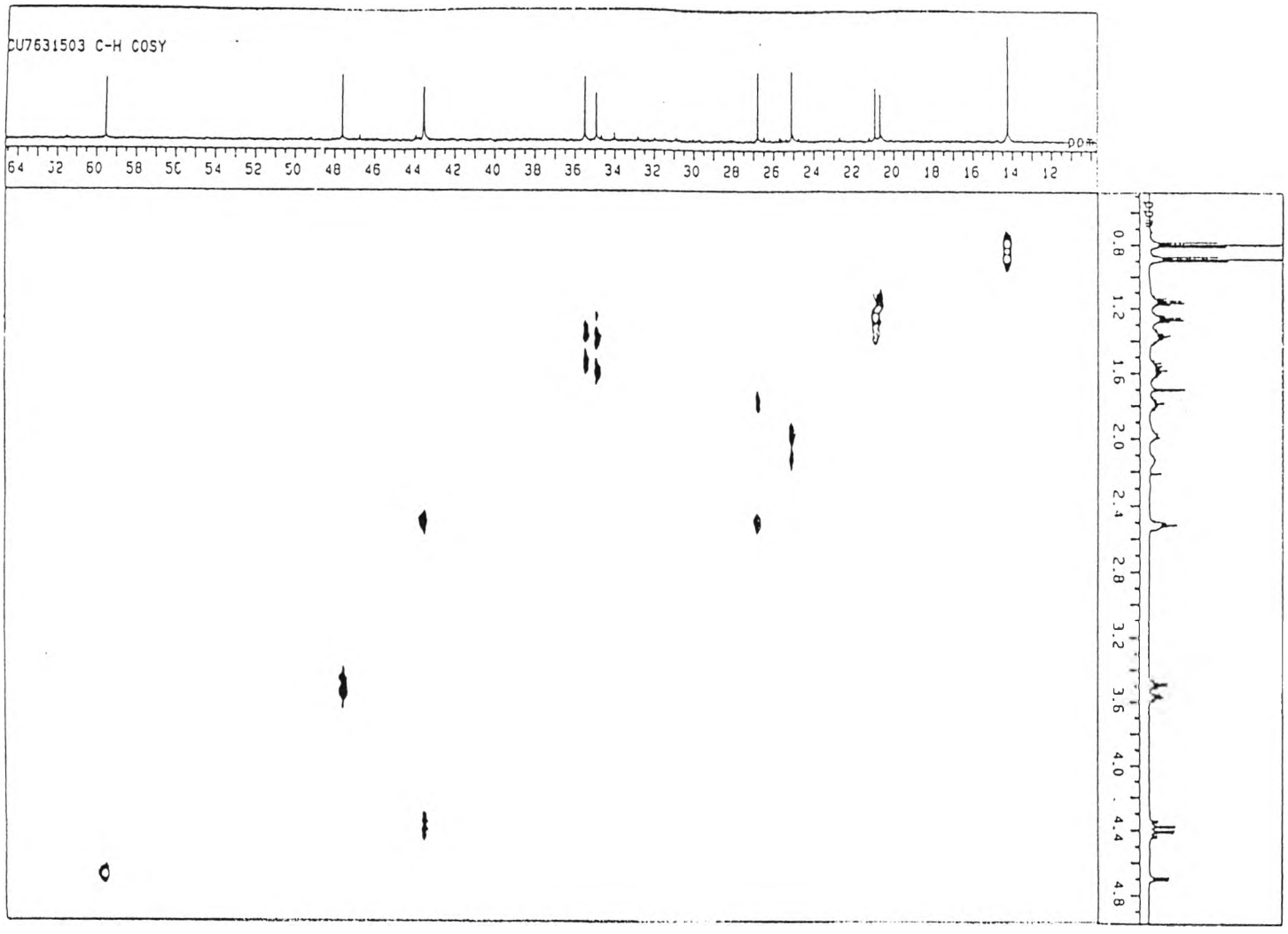

Figure 41. The CH-COSY spectrum of N-(2-propylpentanoyl)-L-proline benzylamide in $CDCl_3$ (Enlarged scale)

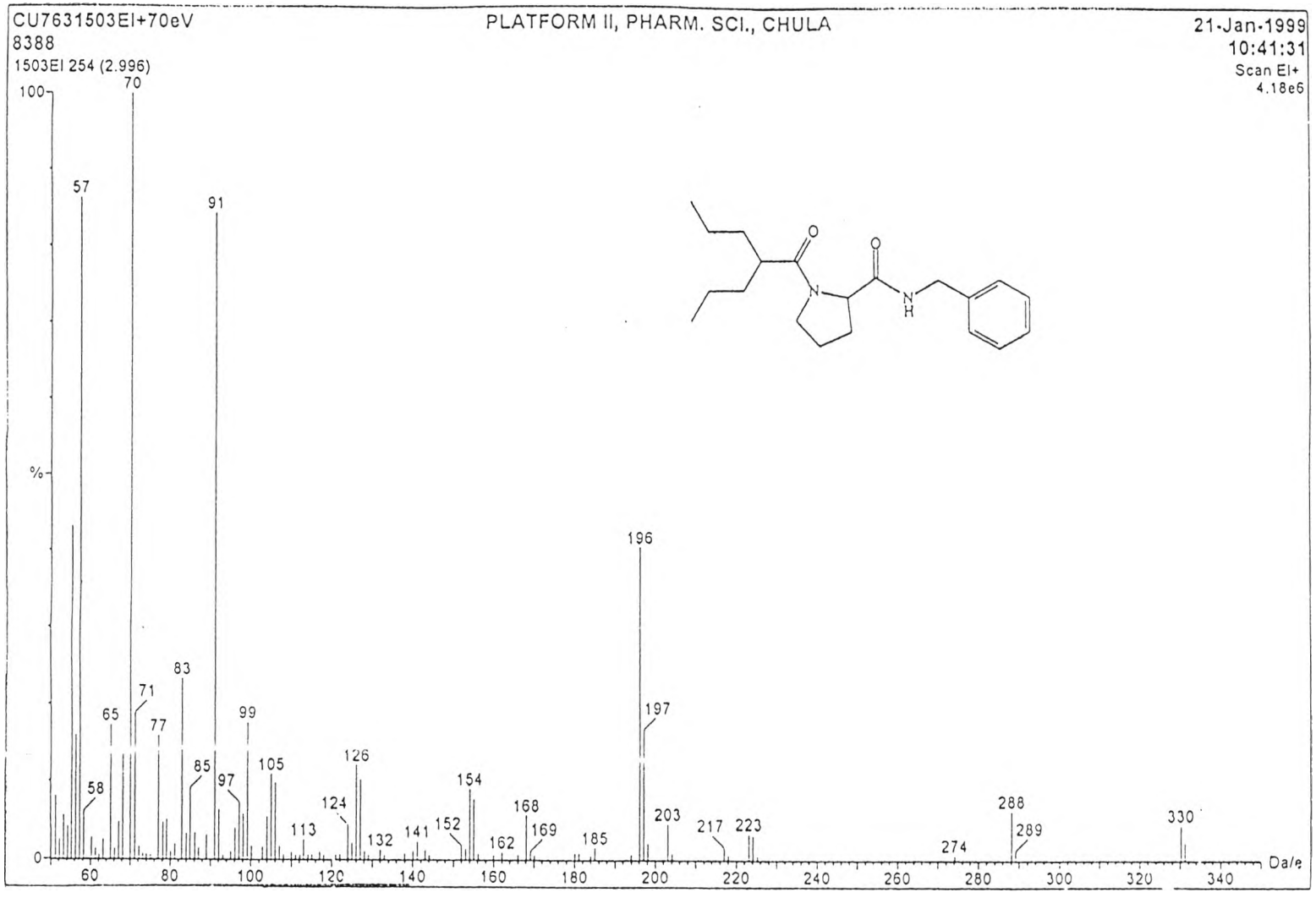

Figure 42. The mass spectrum of N-(2-propylpentanoyl)-L-proline benzylamide

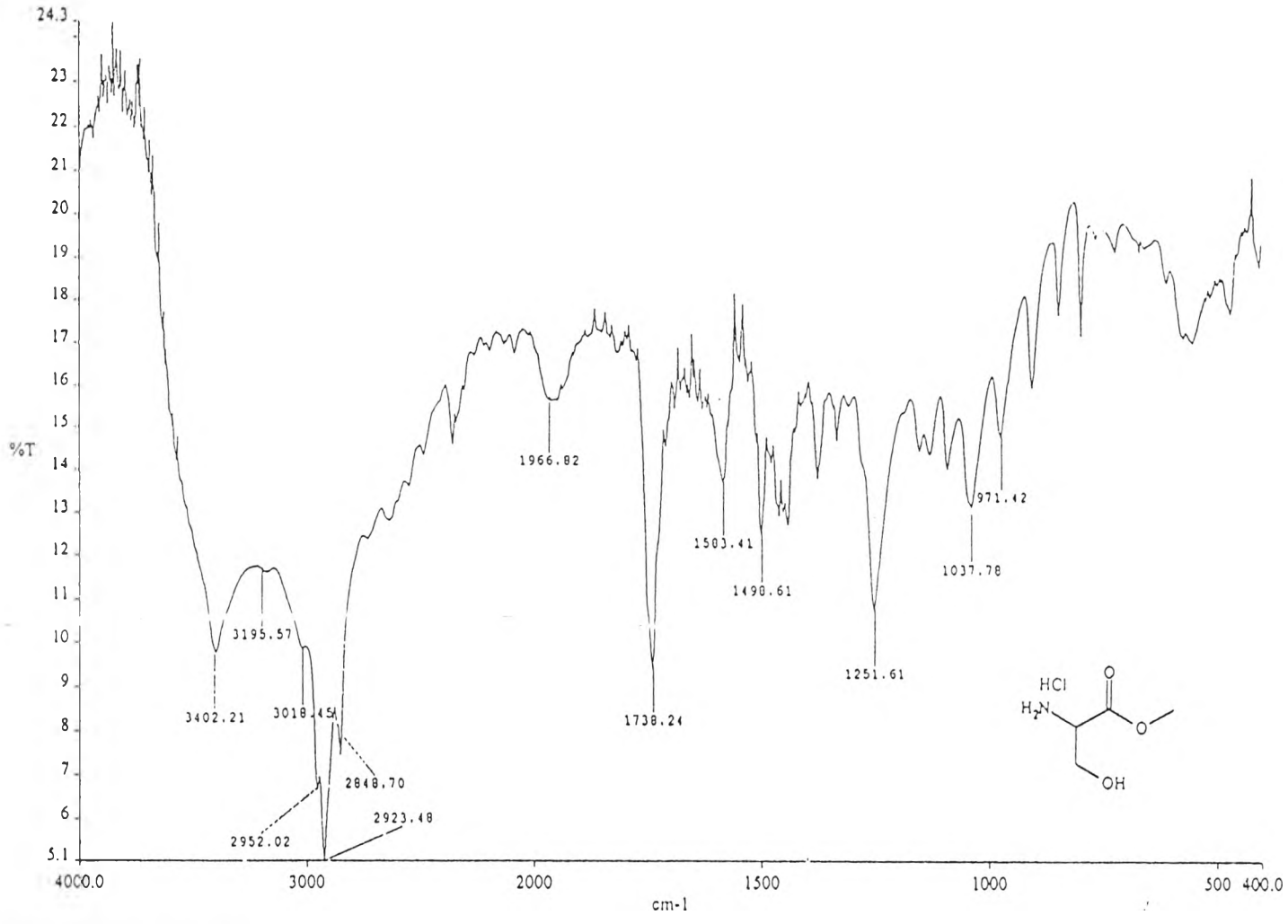

Figure 43. The IR spectrum (Nujol, mull) of DL-serine methyl ester hydrochloride

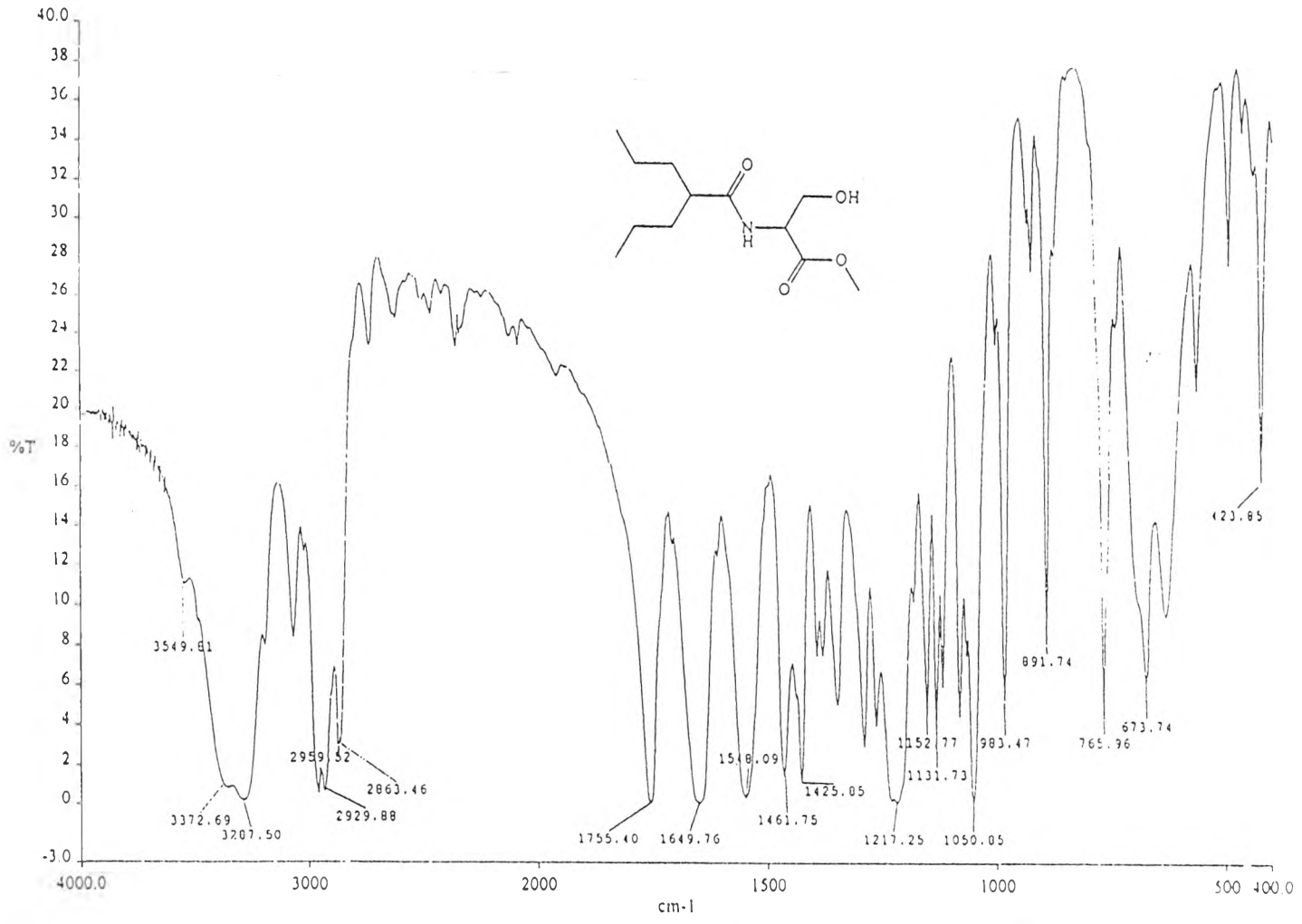

Figure 44. The IR spectrum (KBr) of N-(2-propylpentanoyl)-DL-serine methyl ester

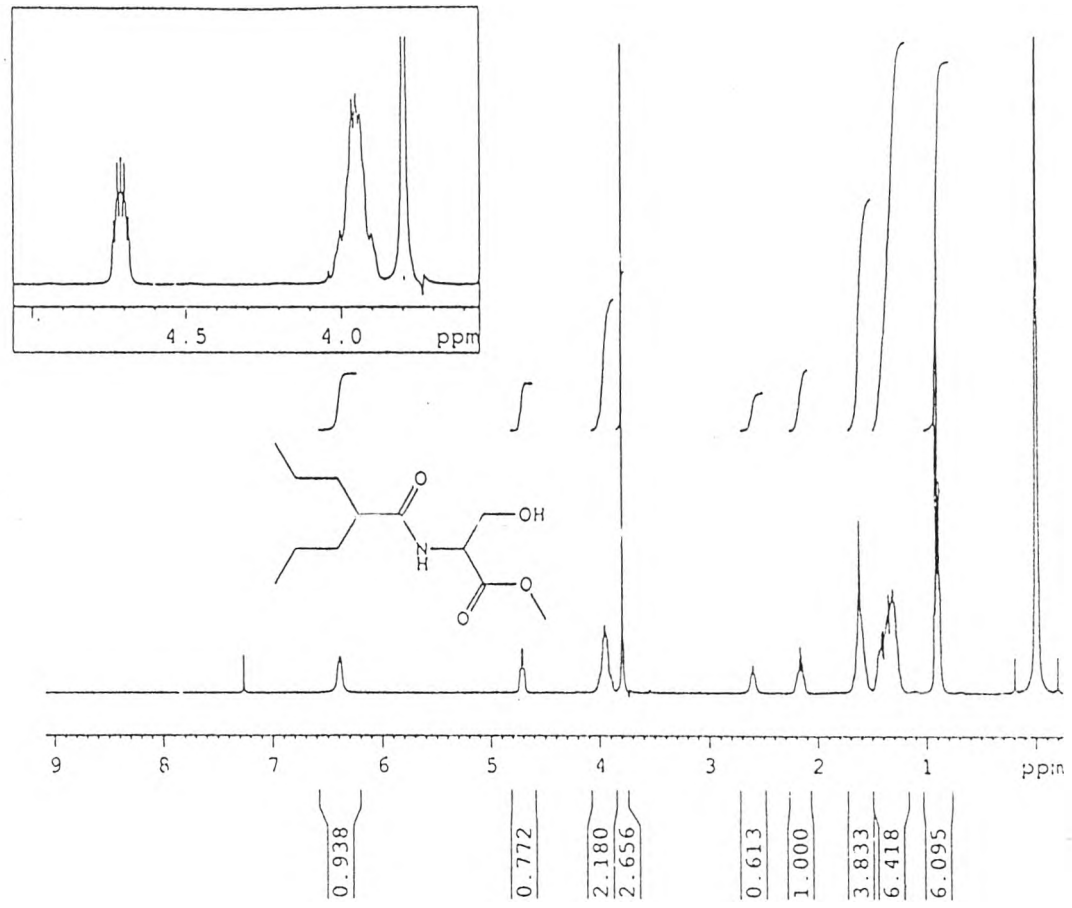

Figure 45. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine methyl ester in $CDCl_3$

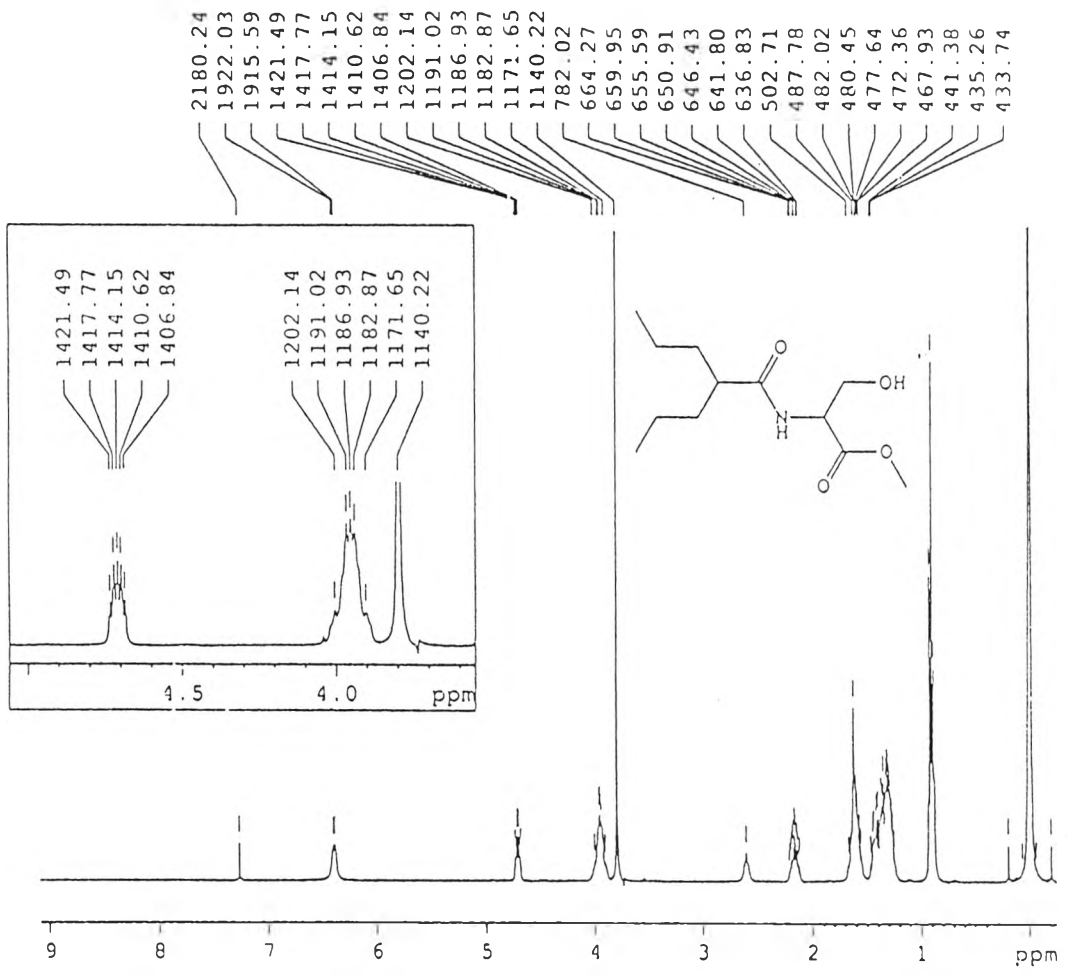

Figure 46. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine methyl ester in $CDCl_3$ (Show peaks in Hz)

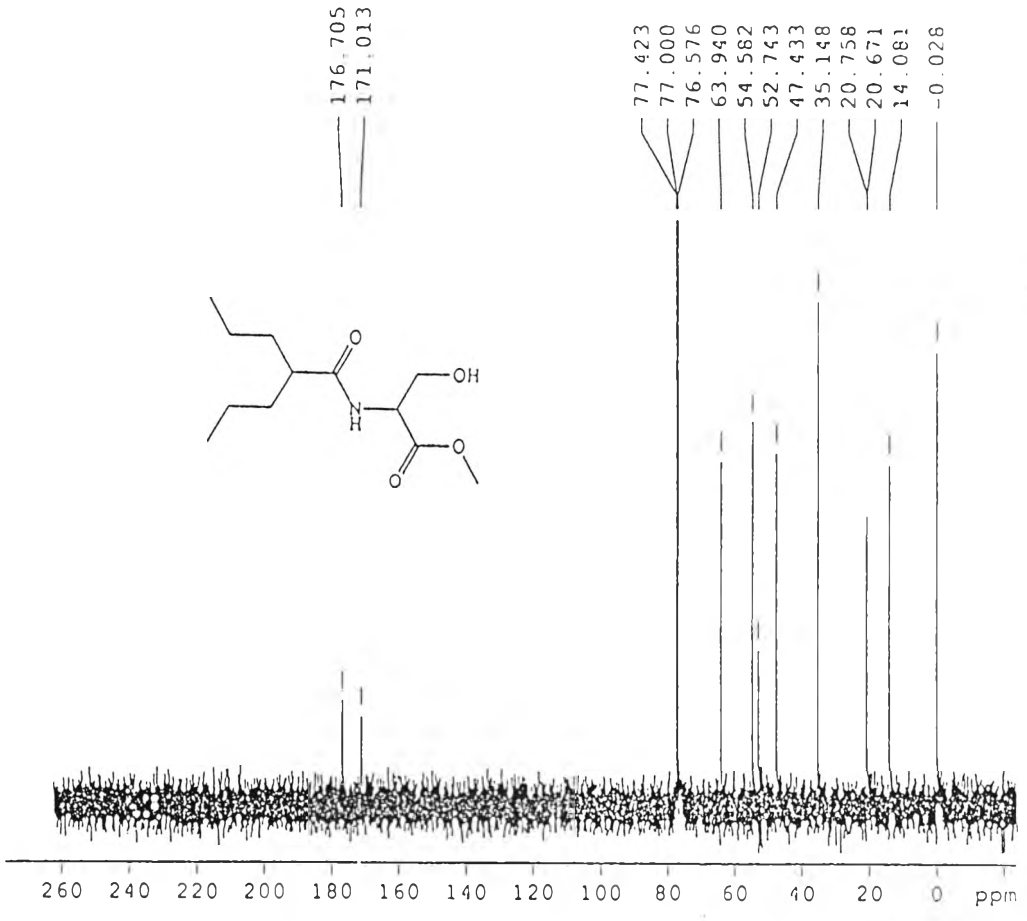

Figure 47. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-DL-serine methyl ester in $CDCl_3$

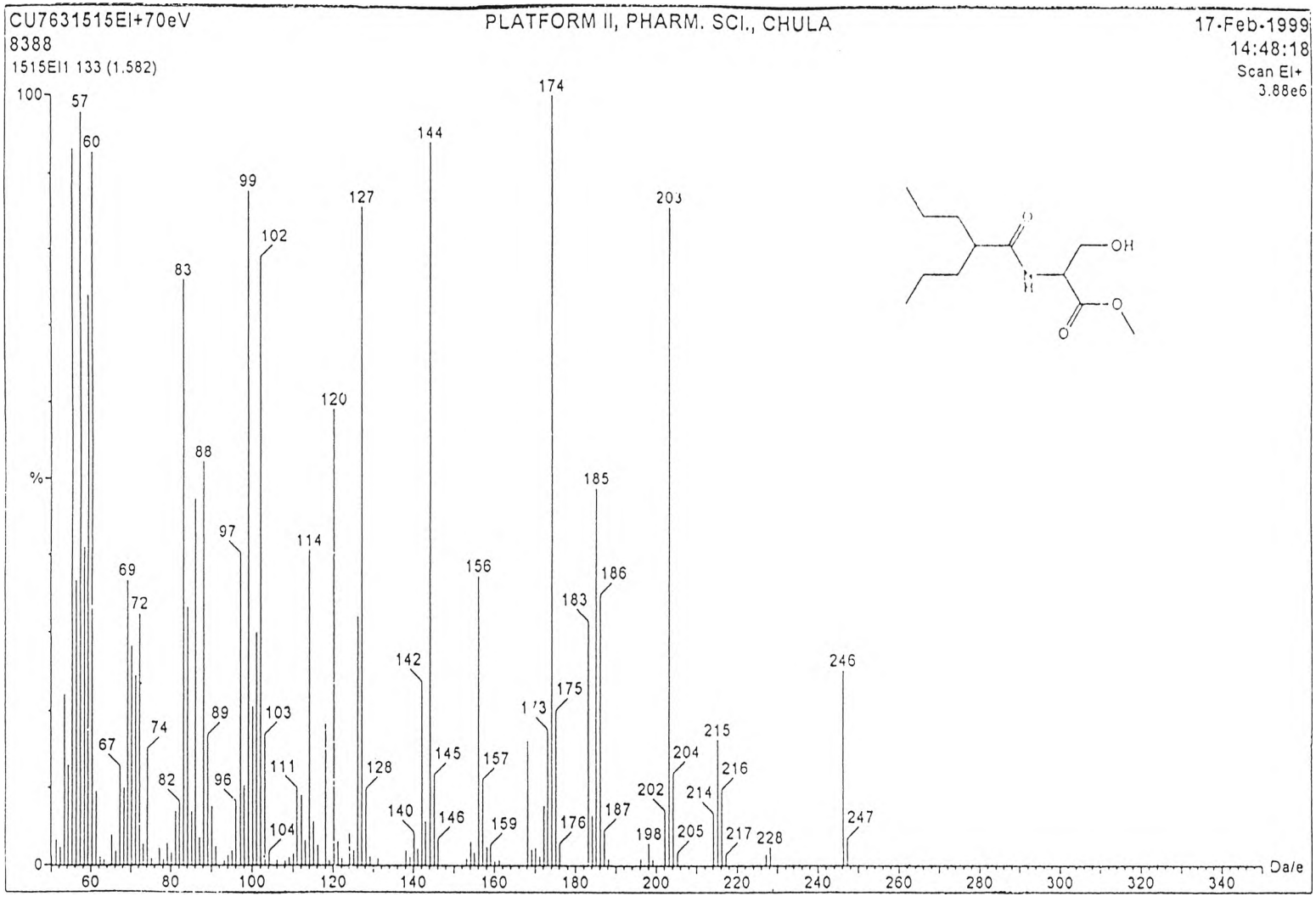

Figure 48. The mass spectrum of N-(2-propylpentanoyl)-DL-serine methyl ester

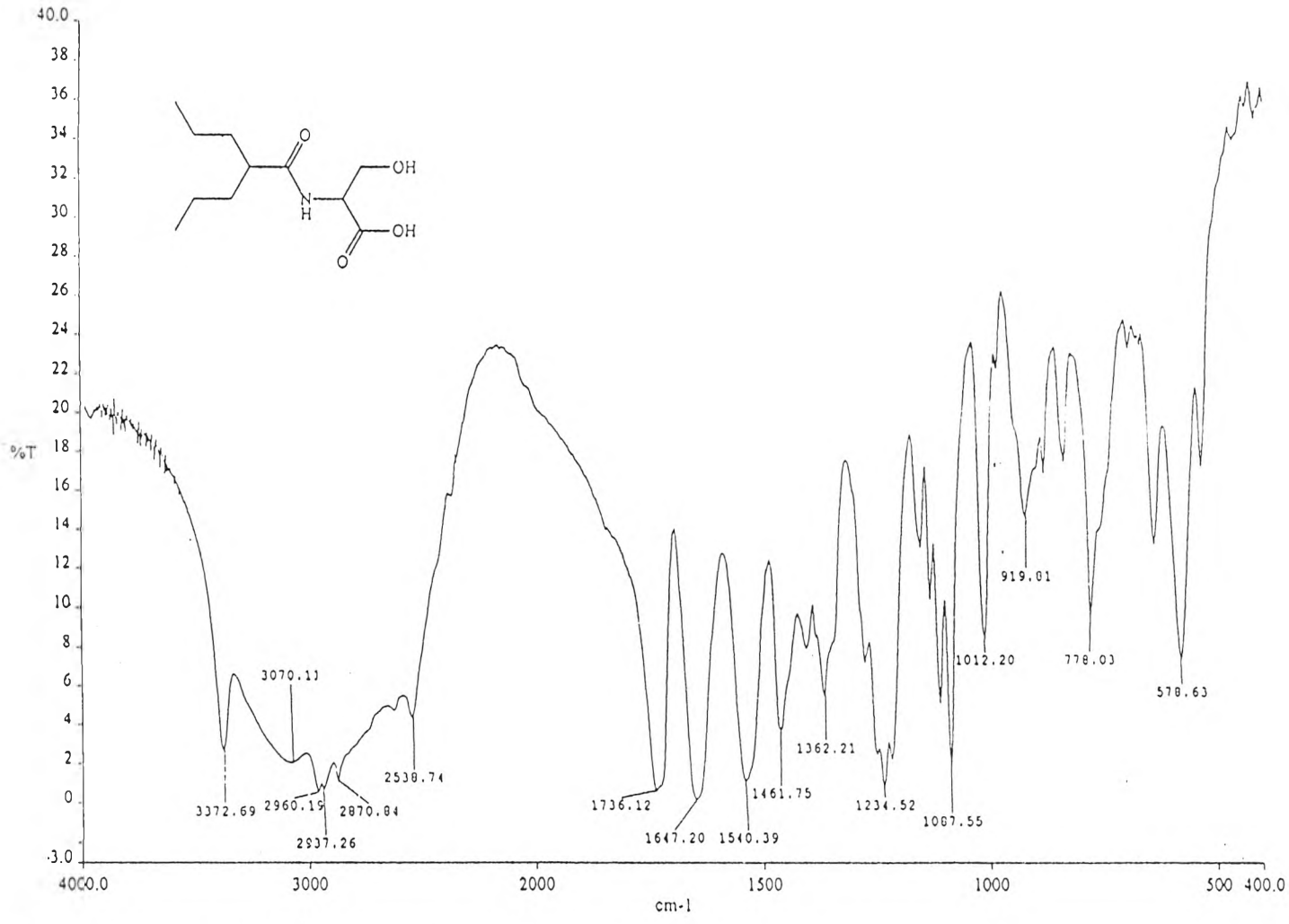

Figure 49. The IR spectrum (KBr) of N-(2-propylpentanoyl)-DL-serine

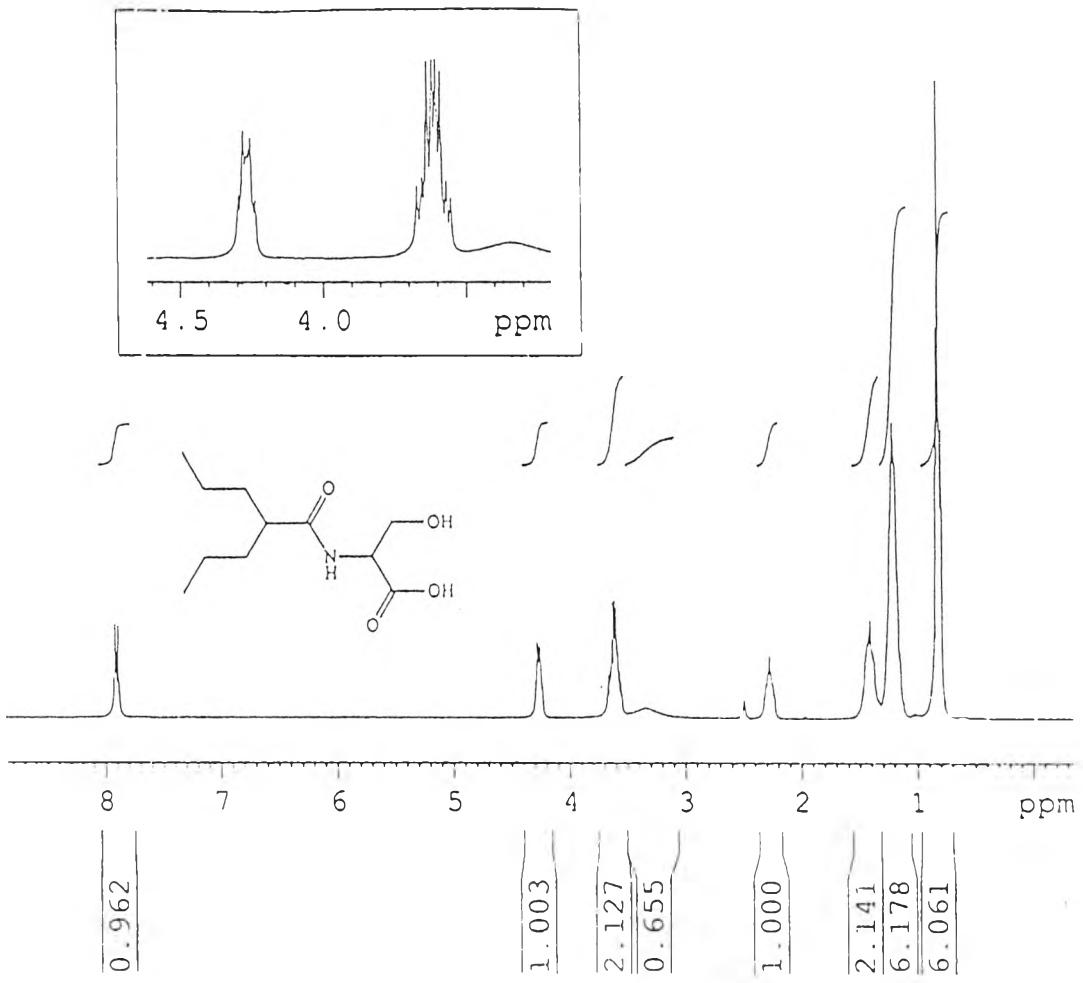

Figure 50. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine in DMSO-d_6

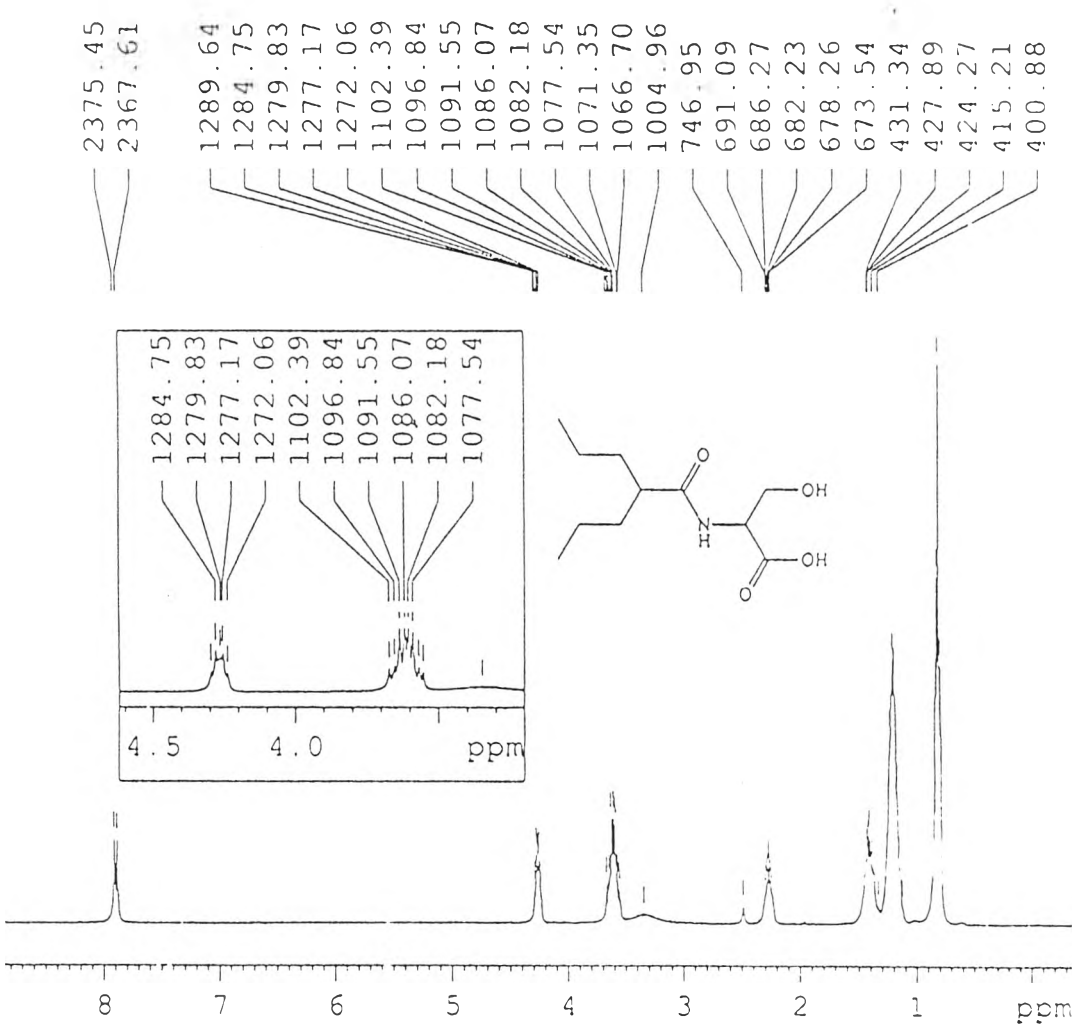

Figure 51. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine in DMSO-d_6(show peak in Hz)

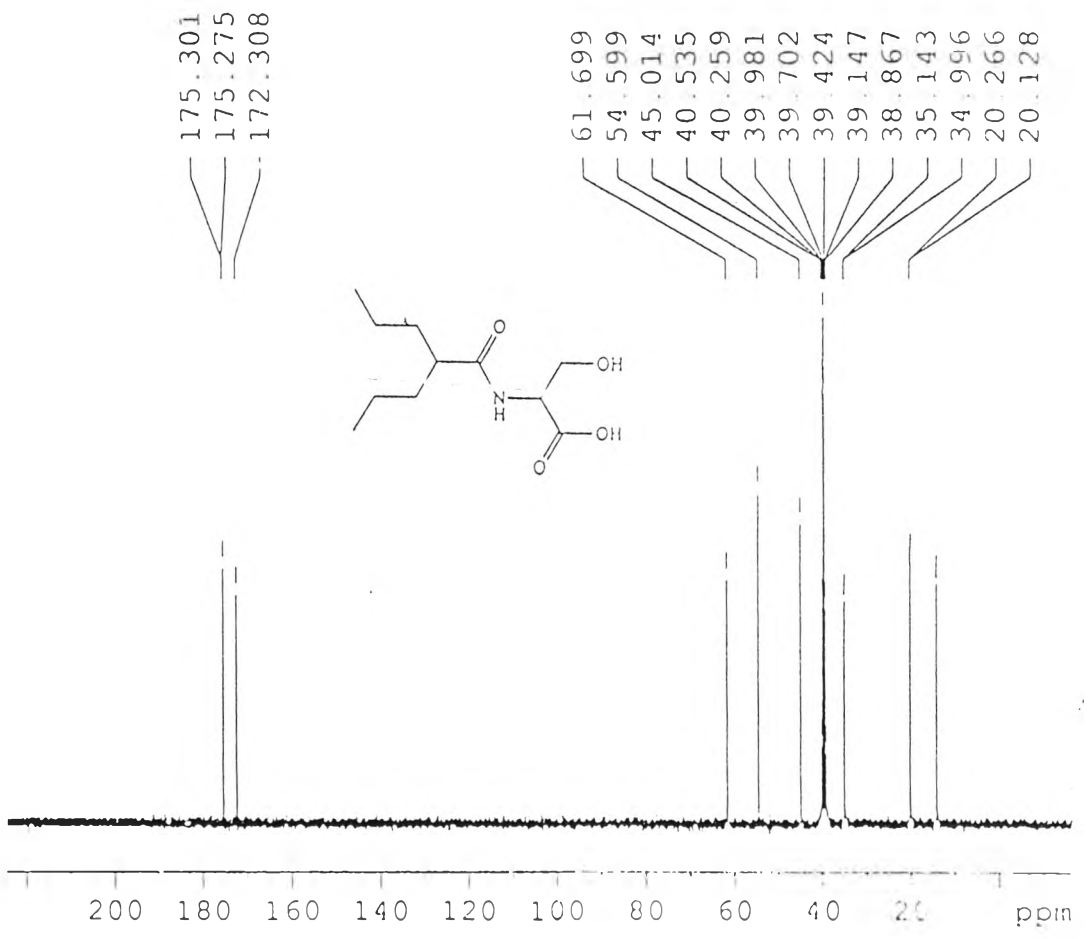

Figure 52. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-DL-serine in DMSO-d_6

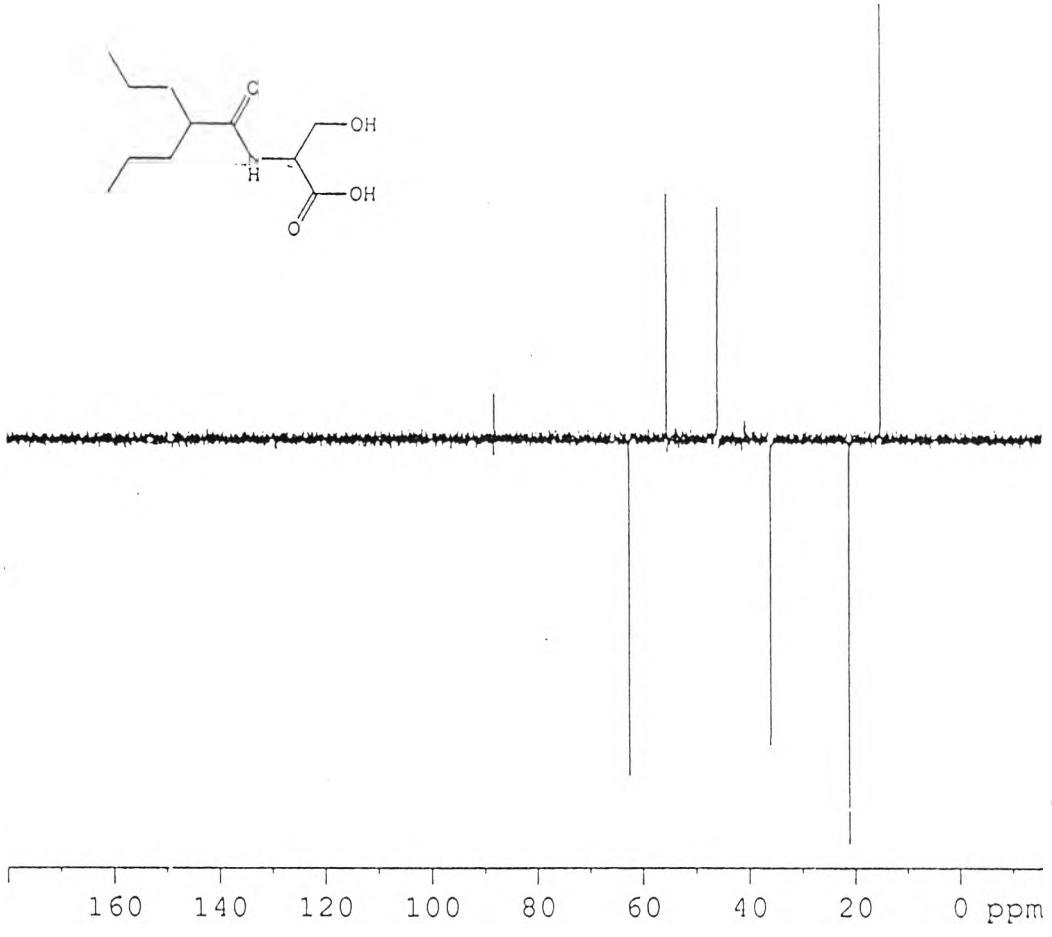

Figure 53. The DEPT 135 spectrum of N-(2-propylpentanoyl)-DL-serine in DMSO-d_6

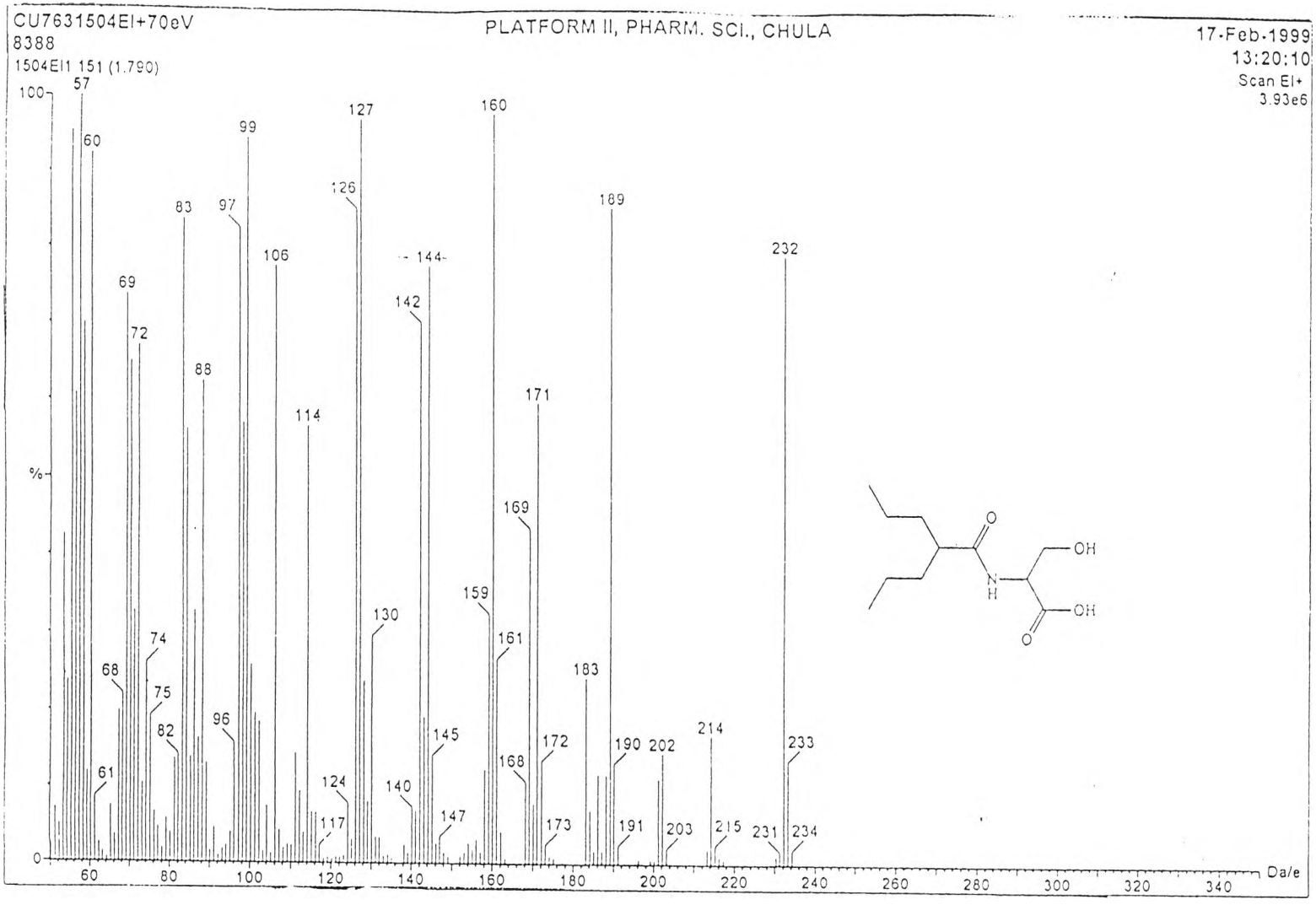

Figure 54. The mass spectrum of N-(2-propylpentanoyl)-DL-serine

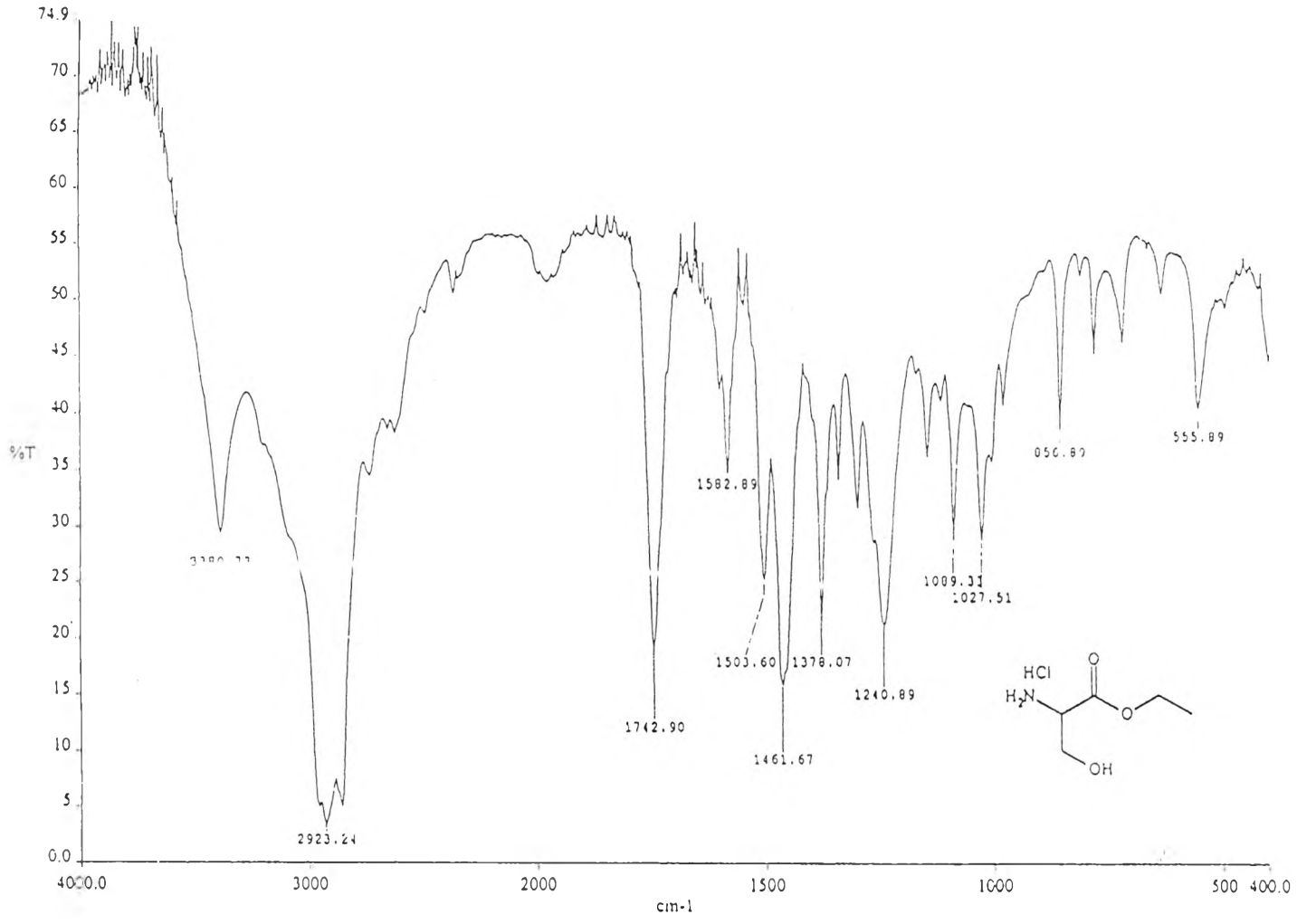

Figure 55. The IR spectrum (Nujol, mull) of DL-serine ethyl ester hydrochloride

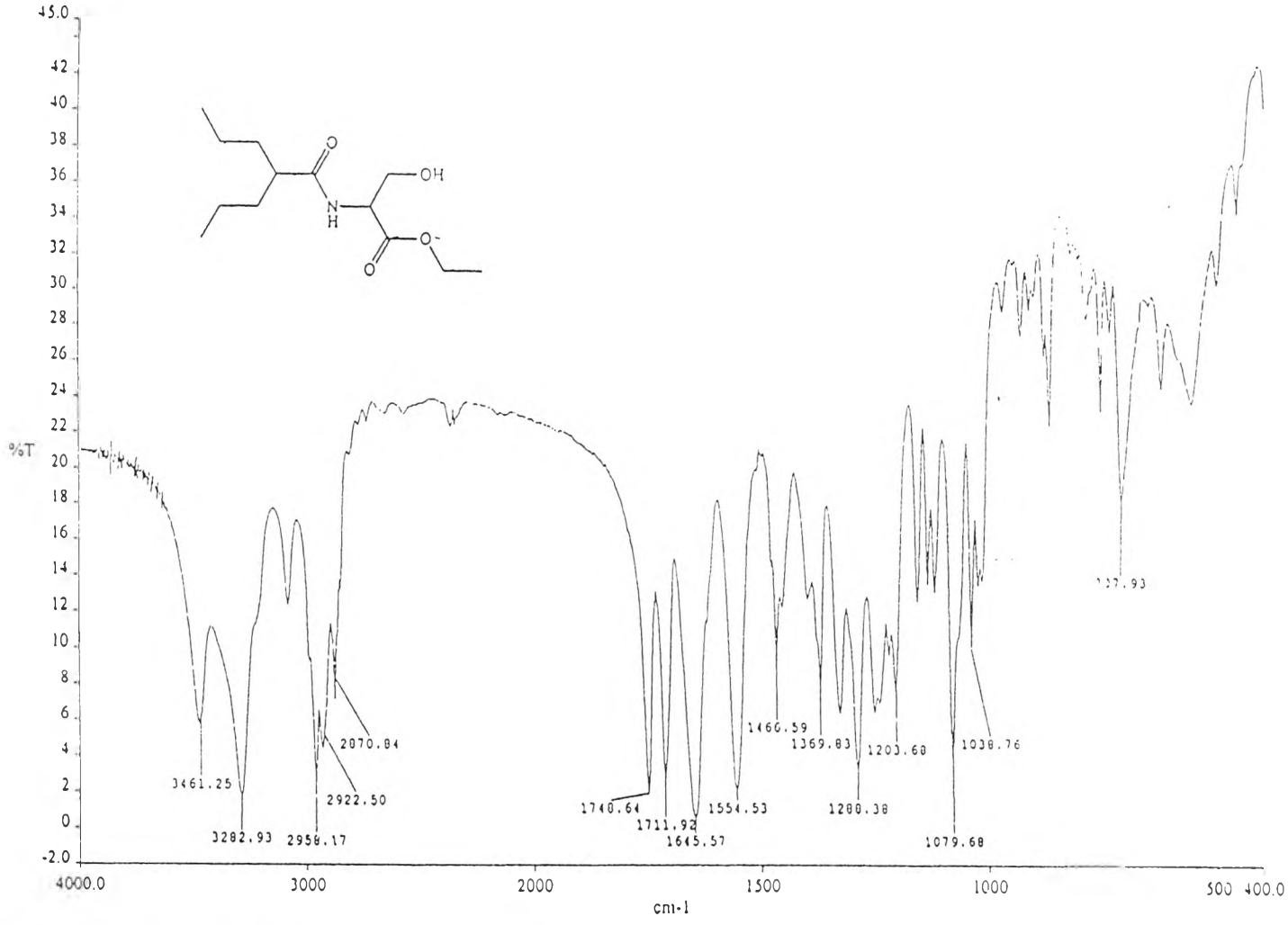

Figure 56. The IR spectrum (KBr) of N-(2-propylpentanoyl)-DL-serine ethyl ester

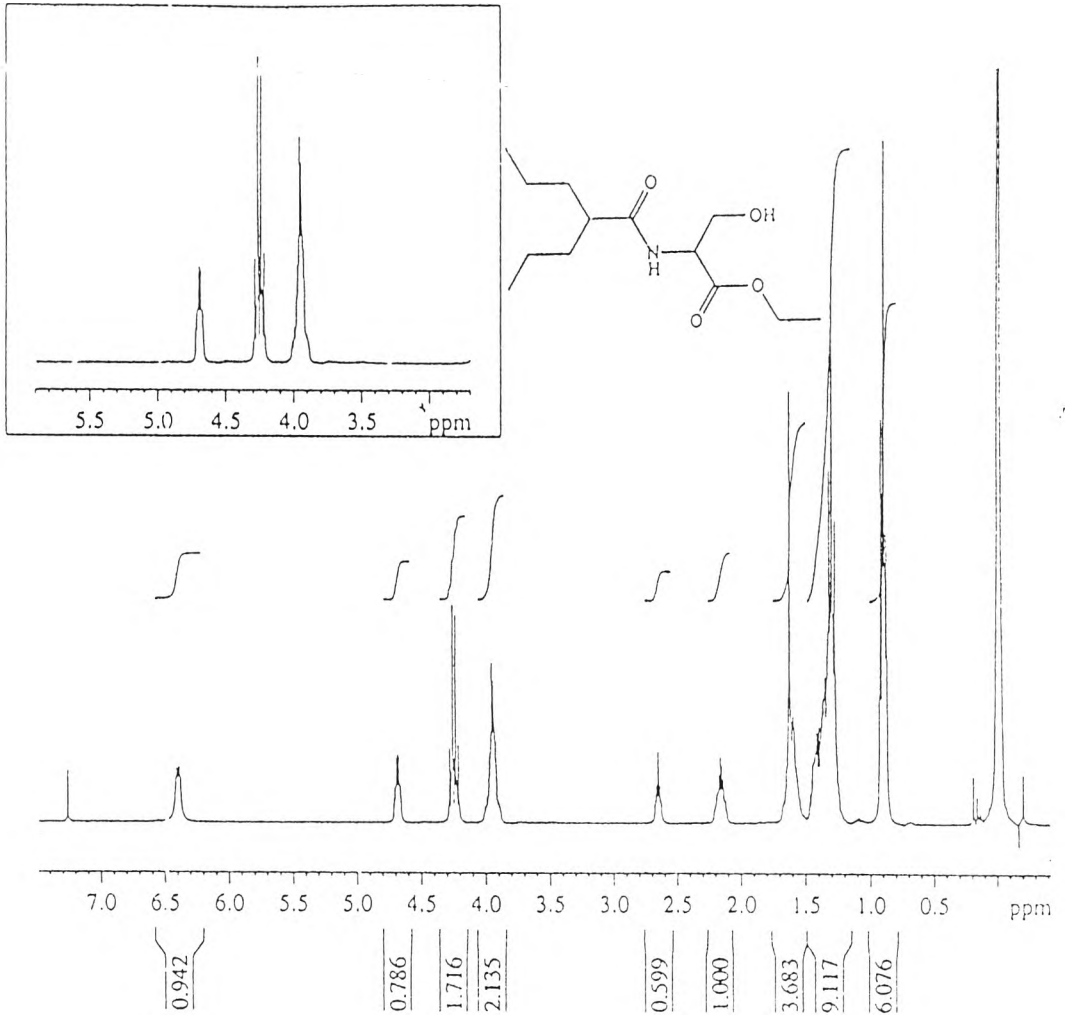

Figure 57. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine ethyl ester in $CDCl_3$

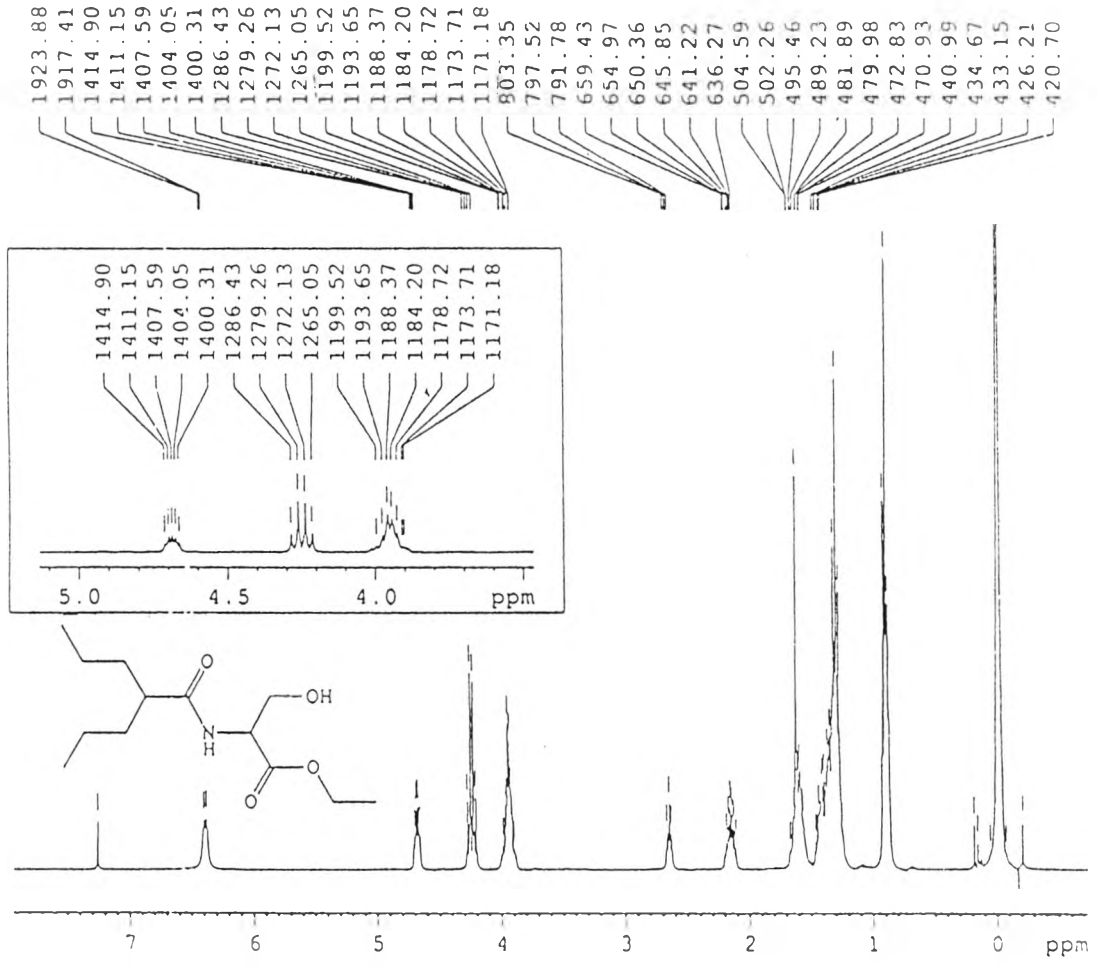

Figure 58. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine ethyl ester in $CDCl_3$ (Show peaks in Hz)

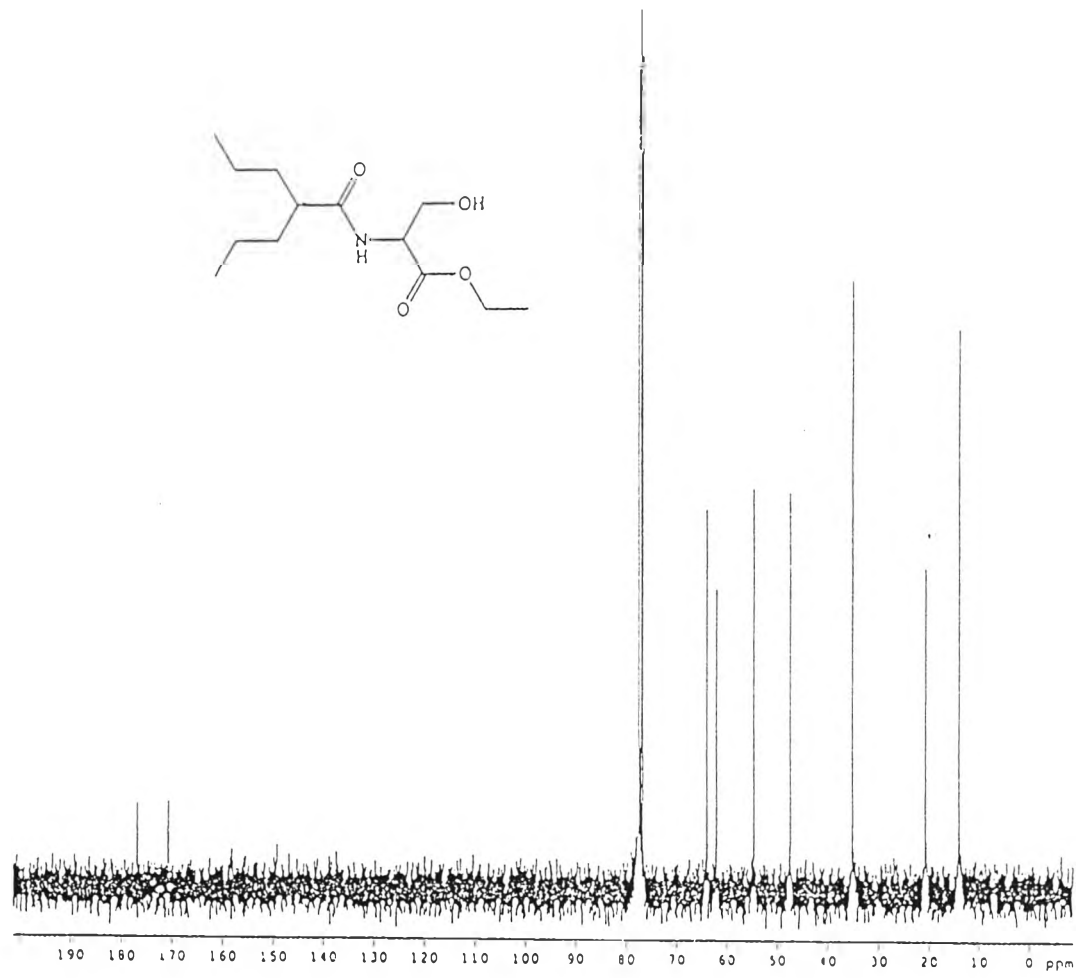

Figure 59. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-DL-serine ethyl ester in $CDCl_3$

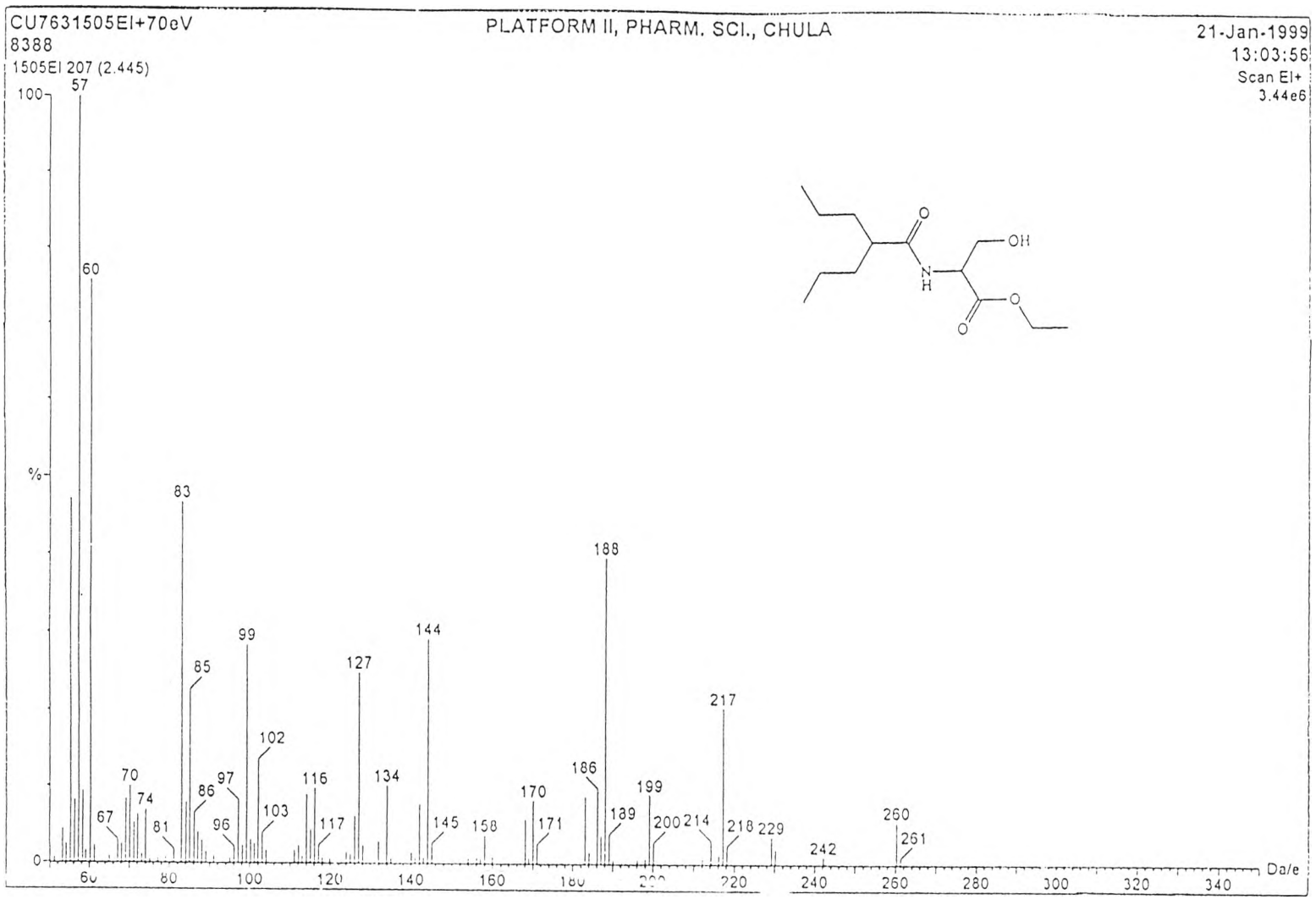

Figure 60. The mass spectrum of N-(2-propylpentanoyl)-DL-serine ethyl ester

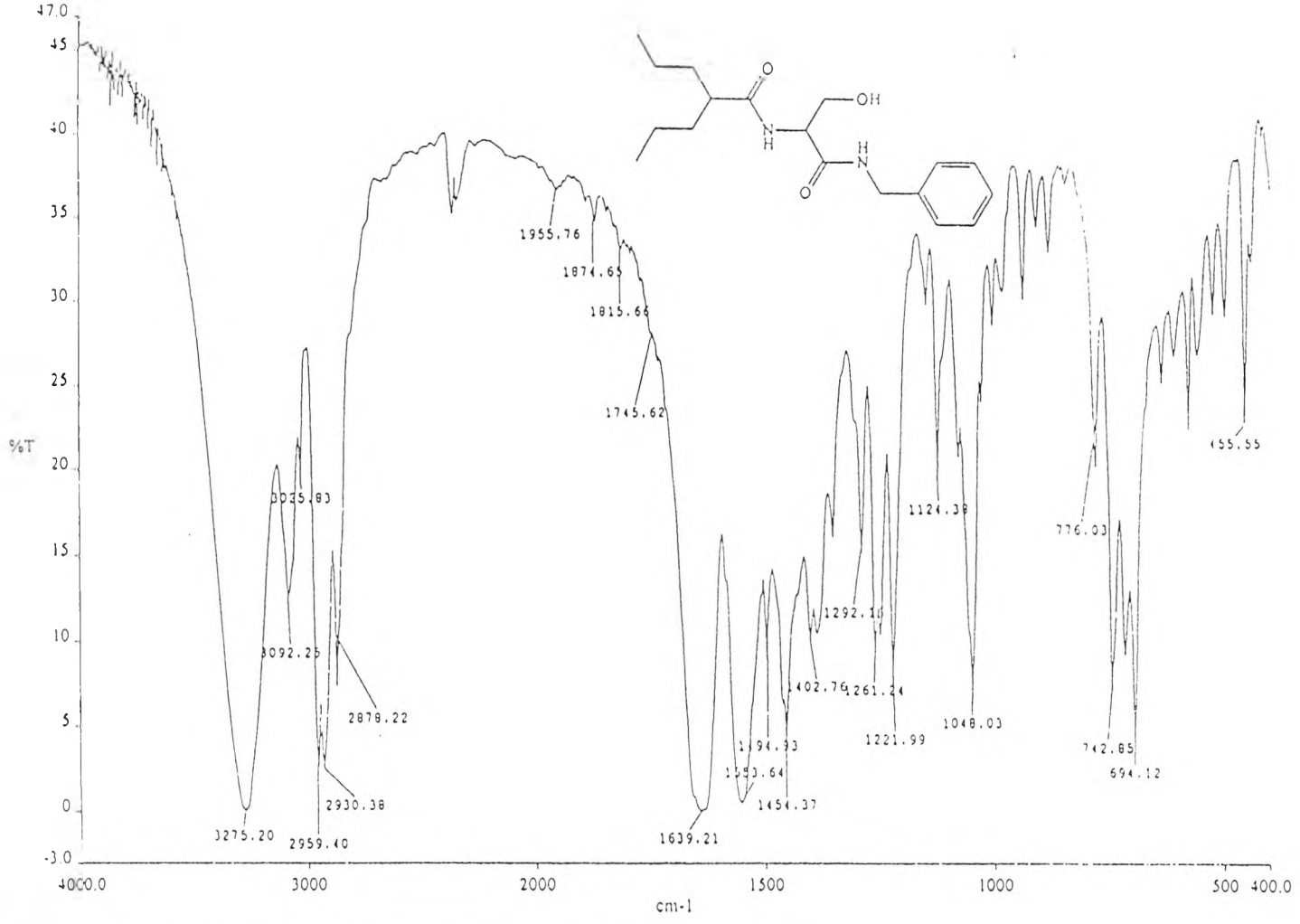

Figure 61. The IR spectrum (KBr) of N-(2-propylpentanoyl)-DL-serine benzylamide

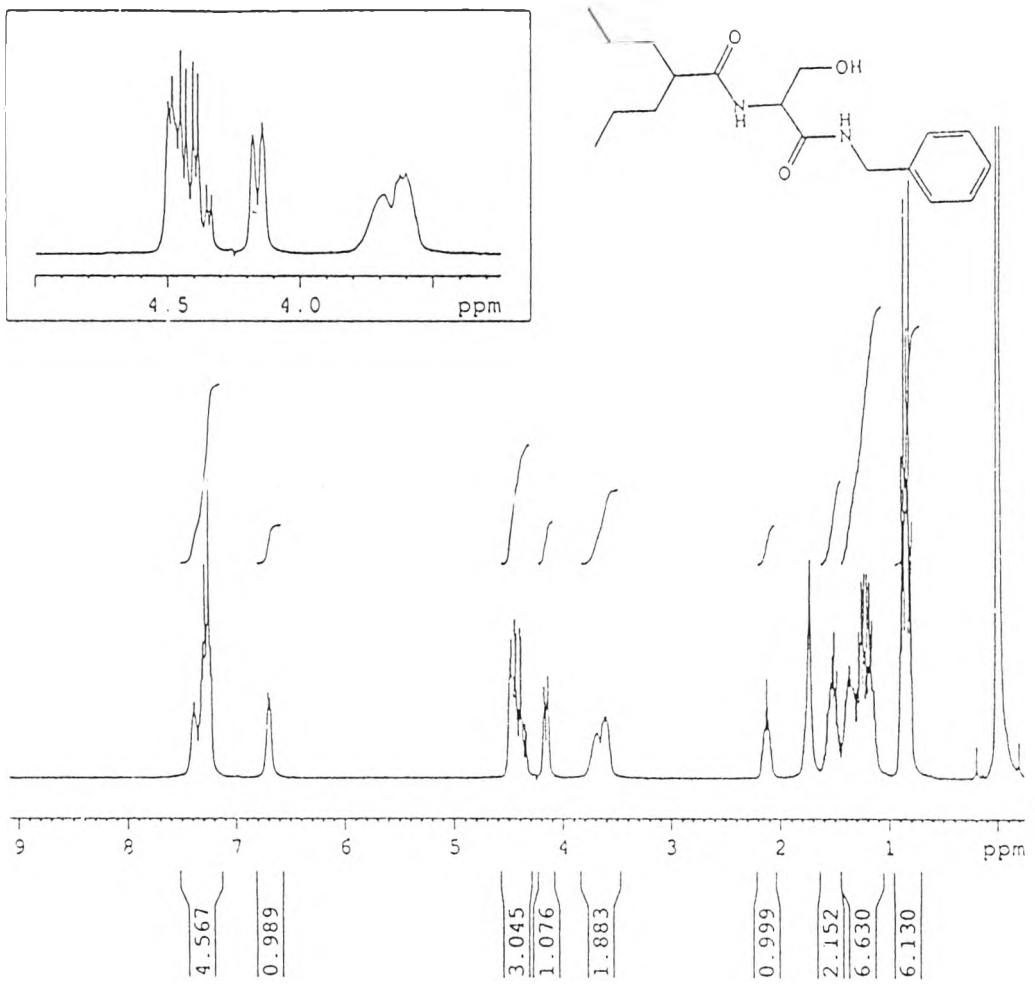

Figure 62. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide in $CDCl_3$

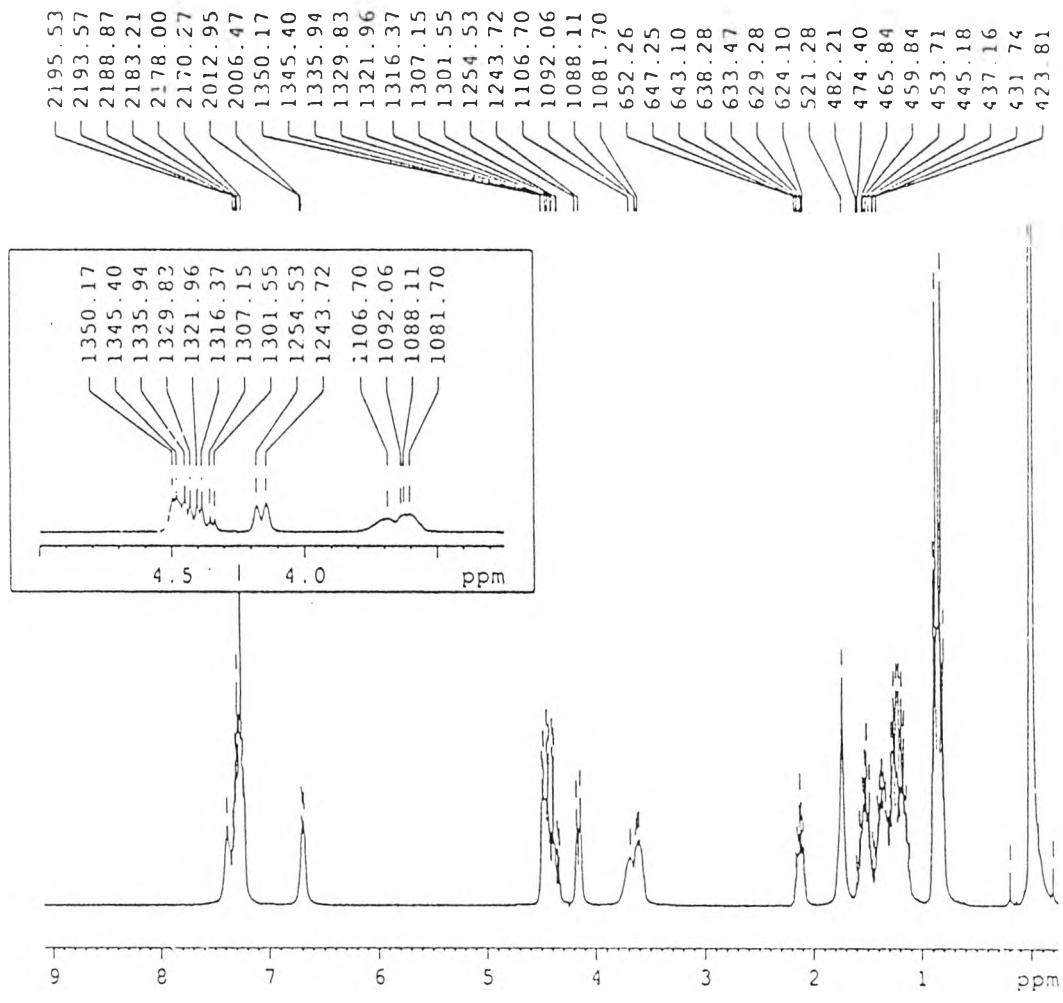

Figure 63. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide in $CDCl_3$ (Show peaks in Hz)

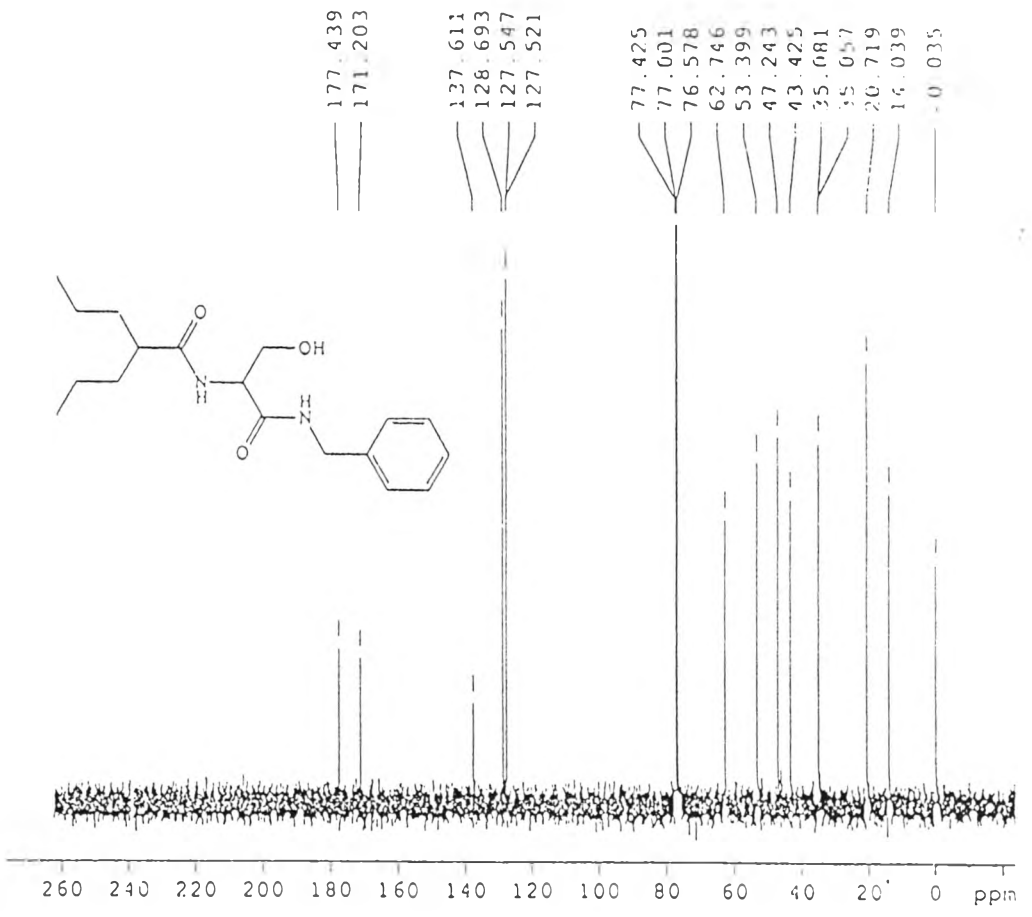

Figure 64. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide in $CDCl_3$

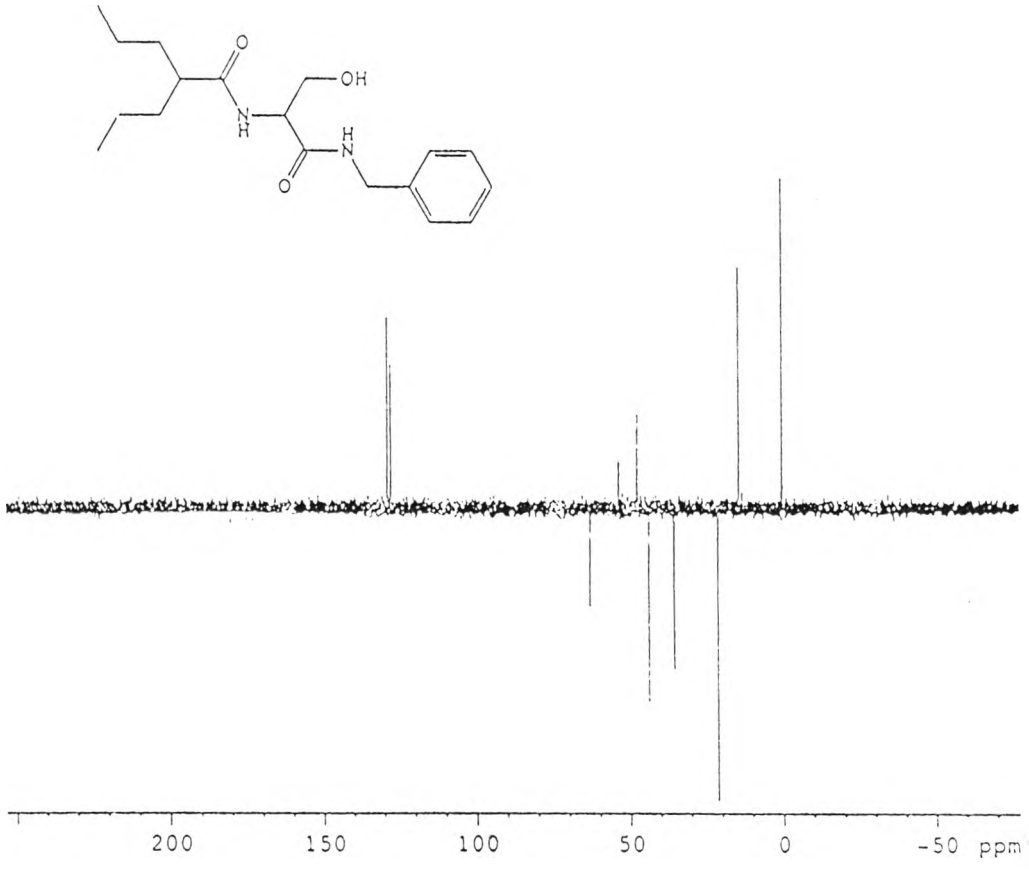

Figure 65. The DEPT 135 spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide in $CDCl_3$

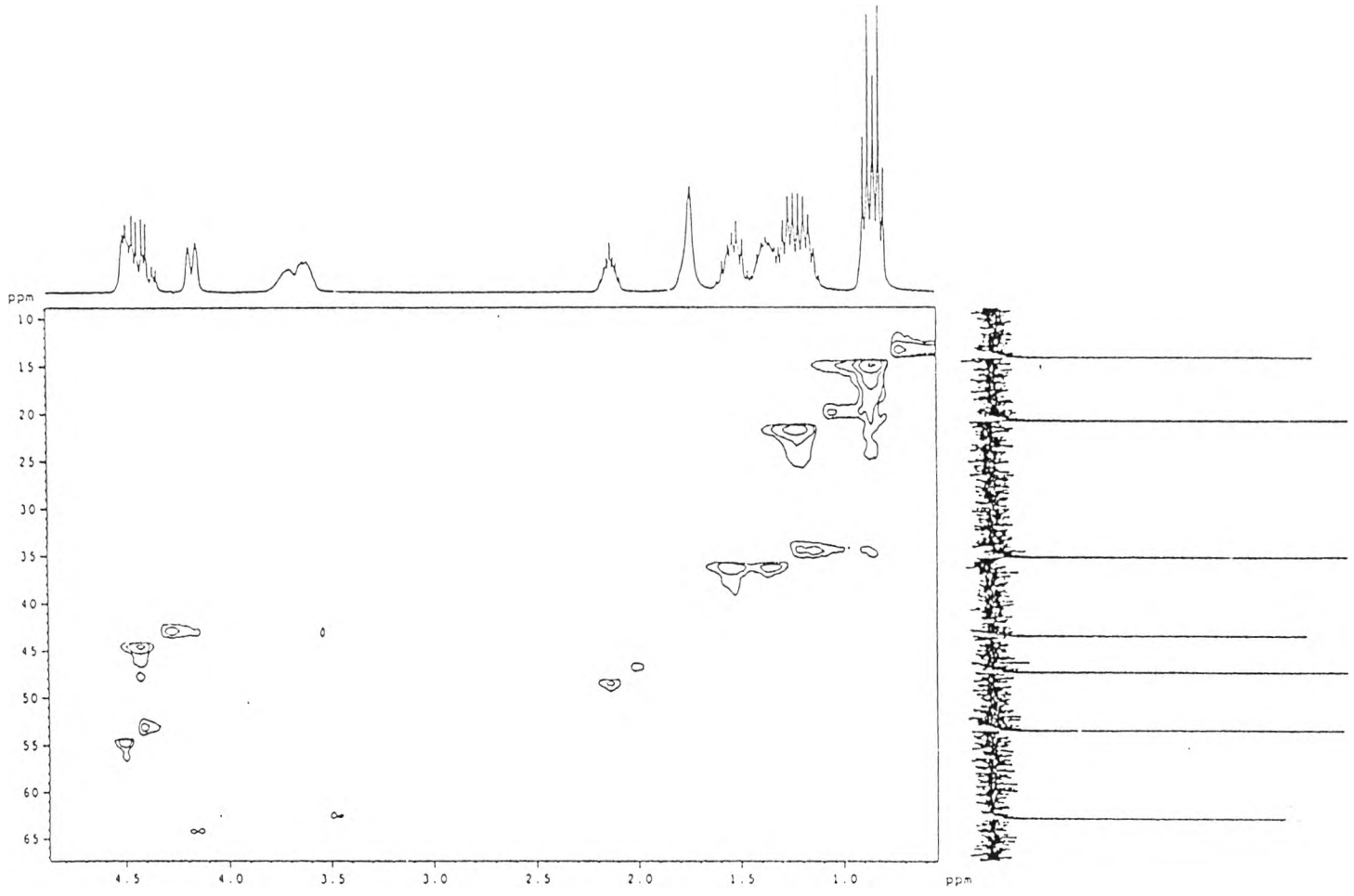

Figure 66. The HMQC spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide in $CDCl_3$

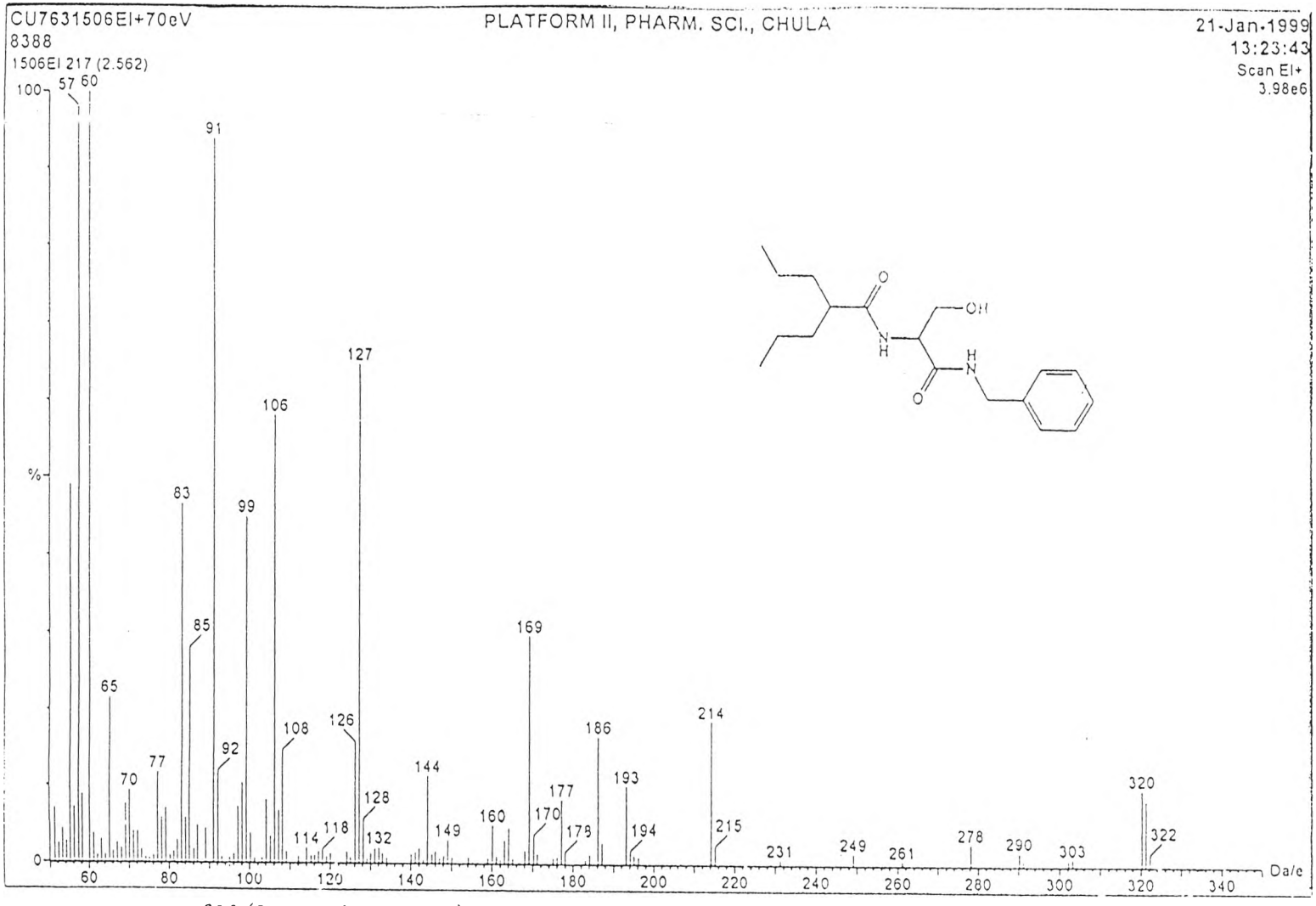

Figure 67. The mass spectrum of N-(2-propylpentanoyl)-DL-serine benzylamide

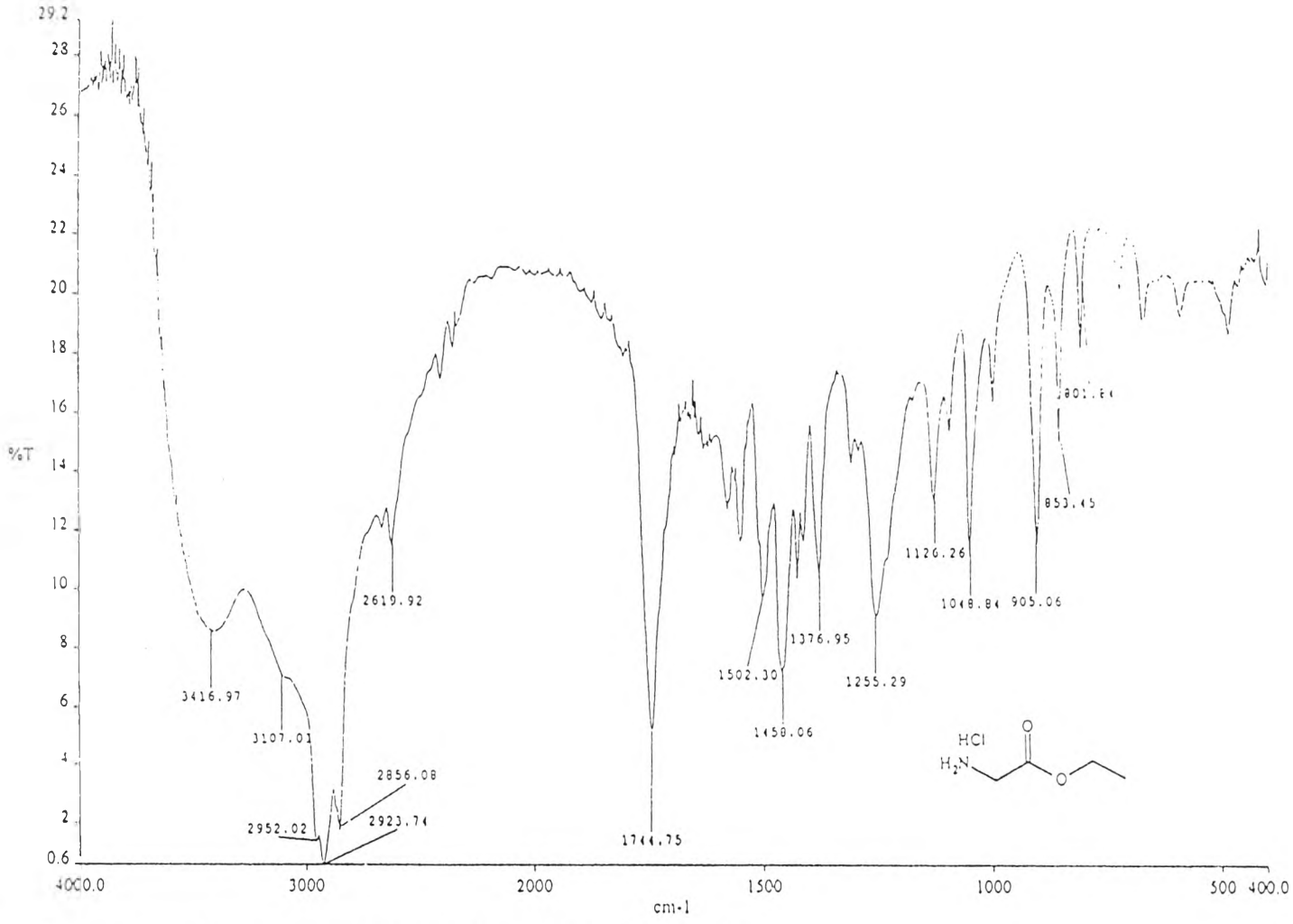

Figure 68. The IR spectrum (Nujol, mull) of glycine ethyl ester hydrochloride

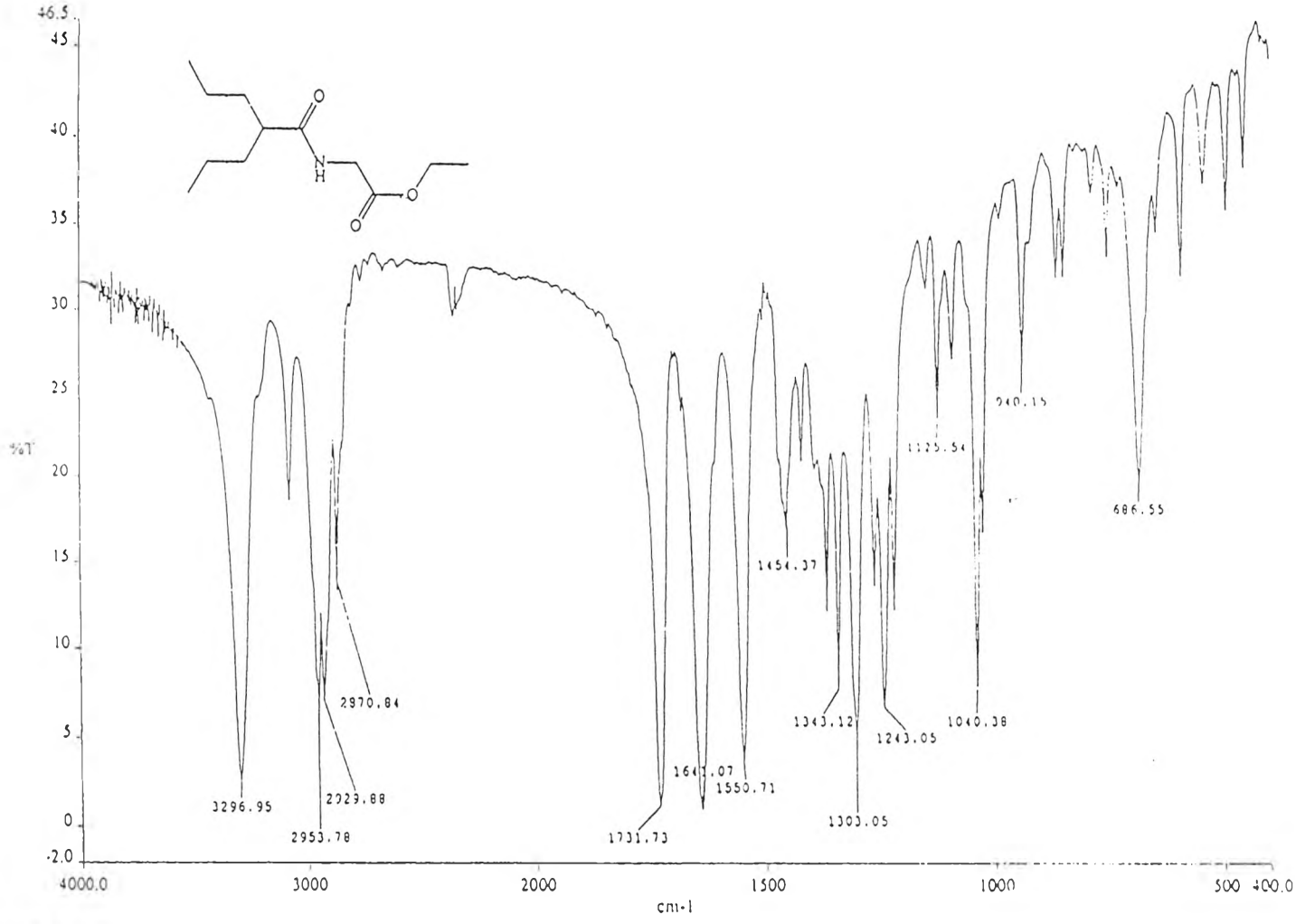

Figure 69. The IR spectrum (KBr) of N-(2-propylpentanoyl)-glycine ethyl ester

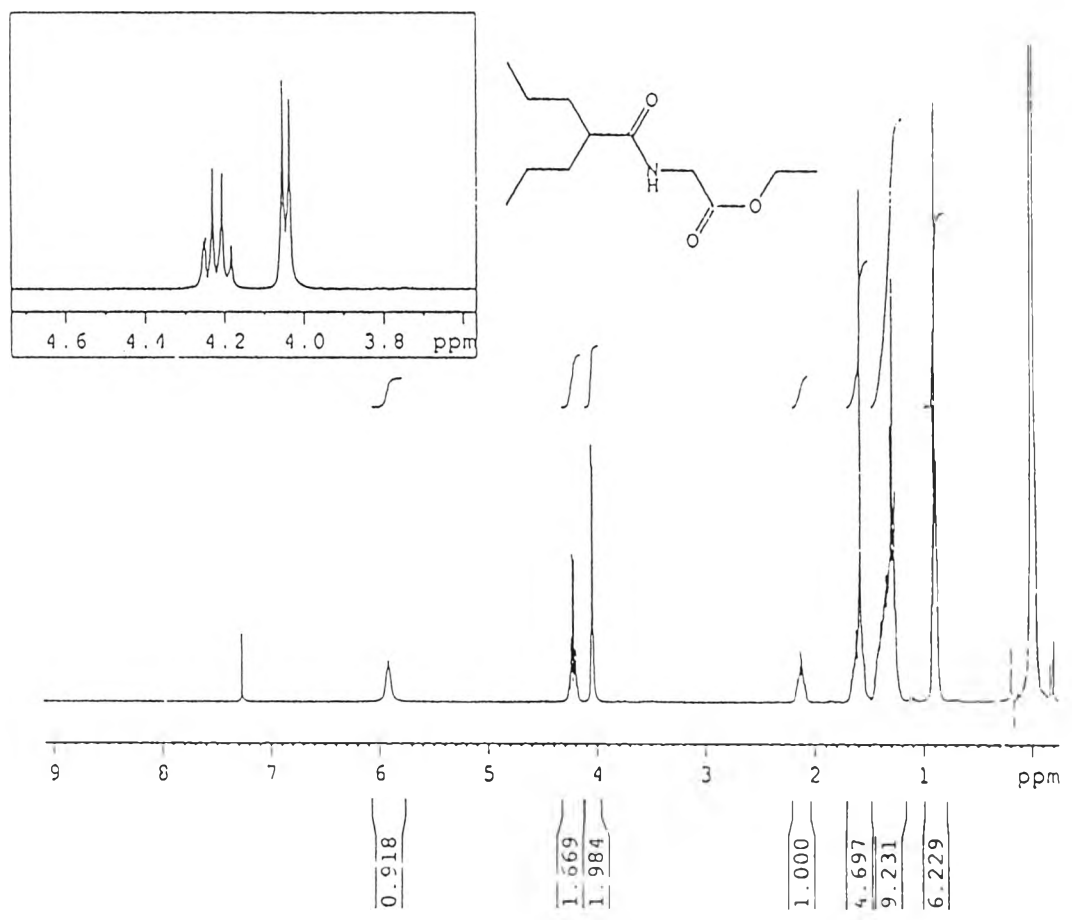

Figure 70. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-glycine ethyl ester in $CDCl_3$

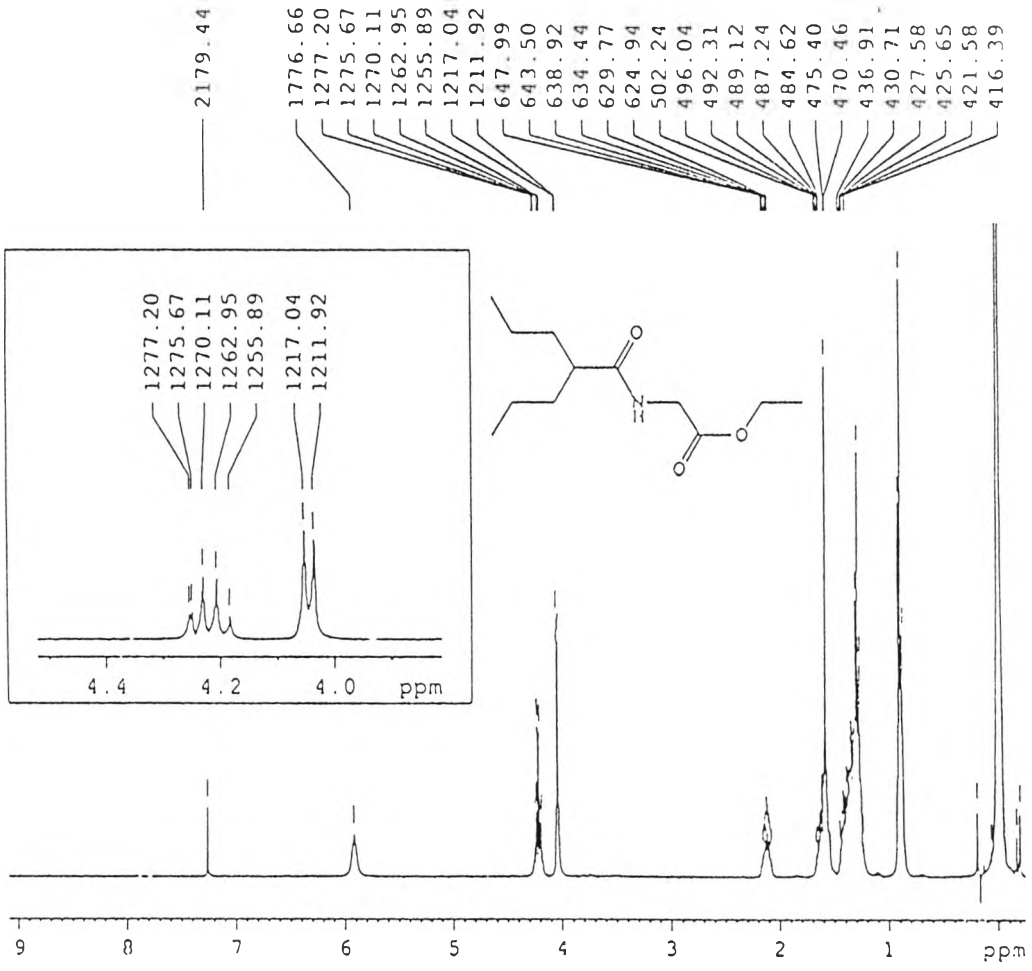

Figure 71. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-glycine ethyl ester in $CDCl_3$ (Show peaks in Hz)

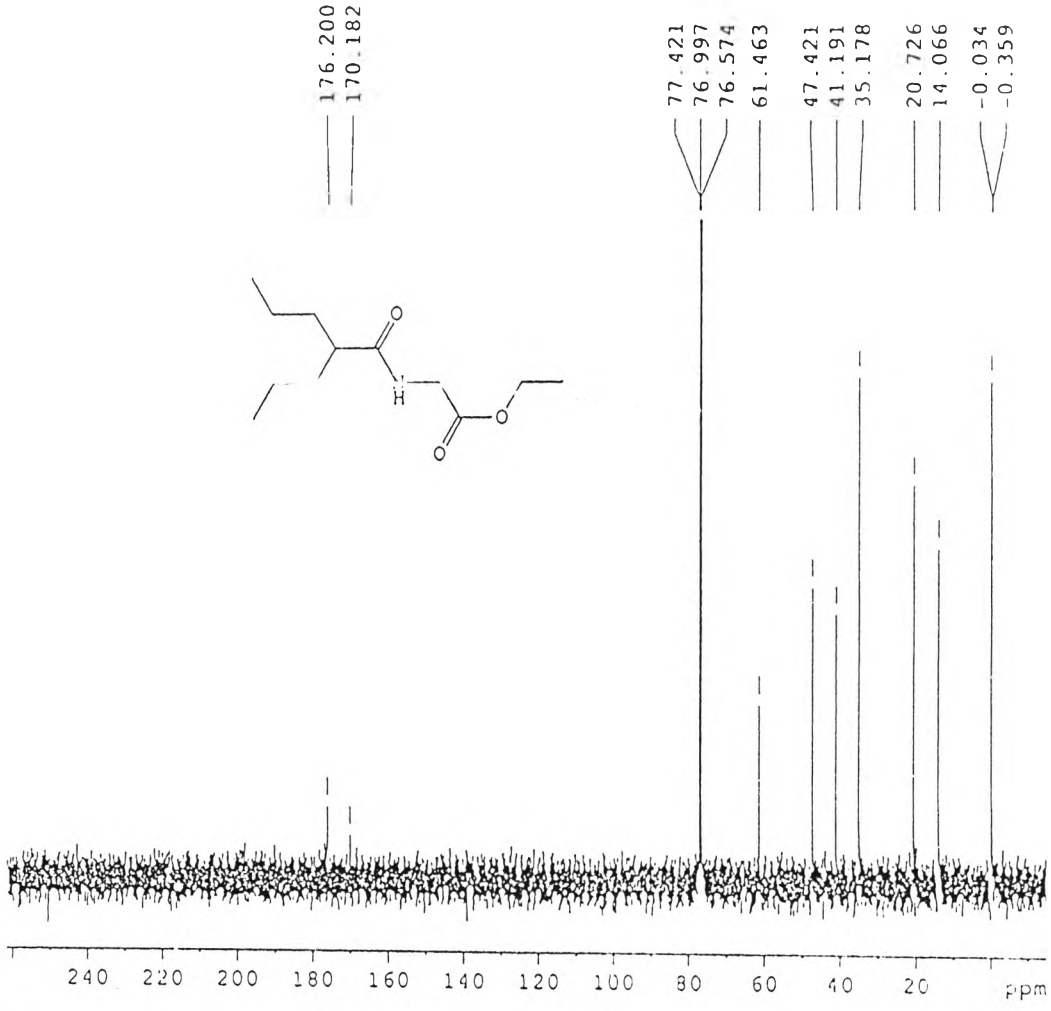

Figure 72. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-glycine ethyl ester in $CDCl_3$

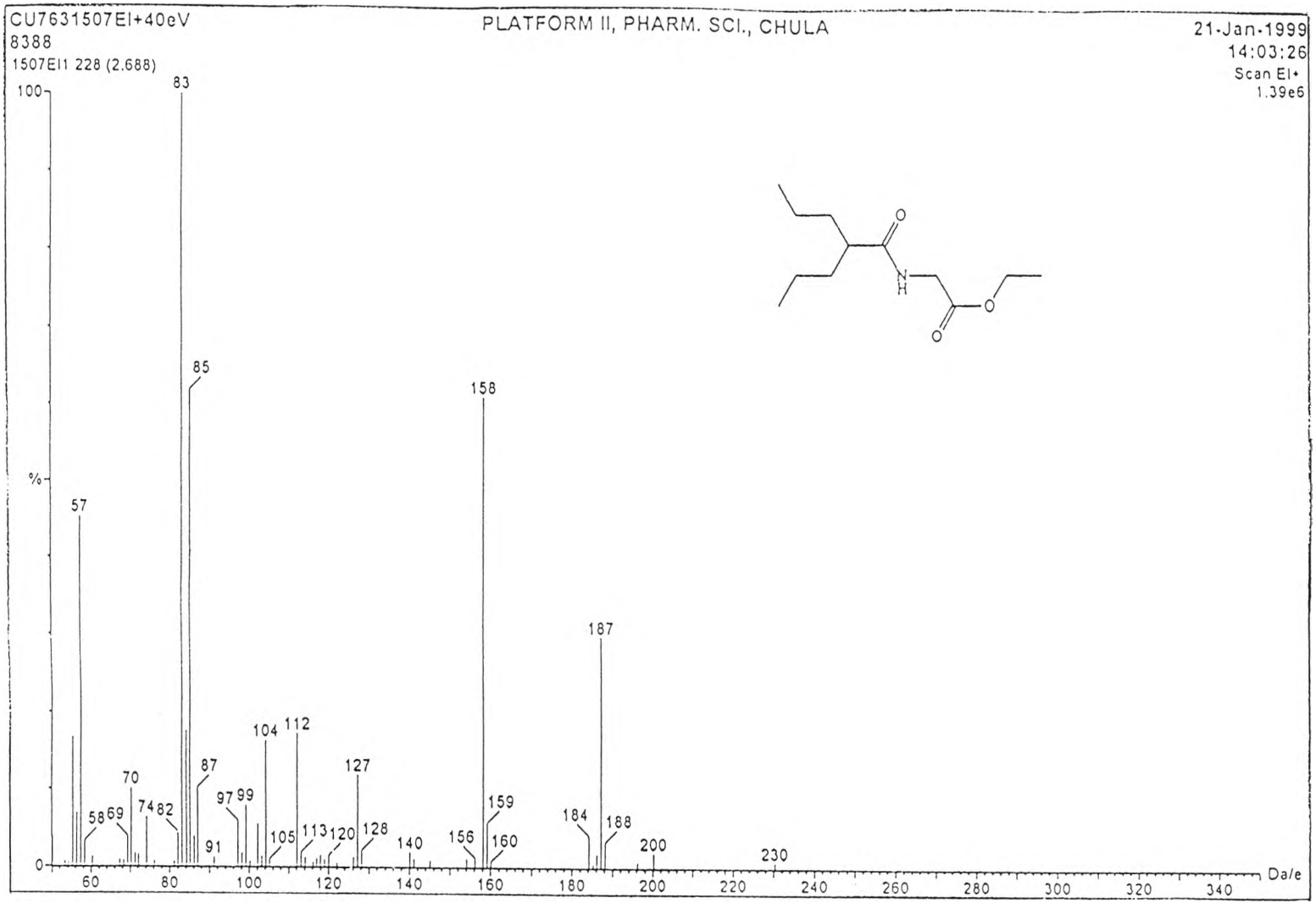

Figure 73. The mass spectrum of N-(2-propylpentanoyl)-glycine ethyl ester

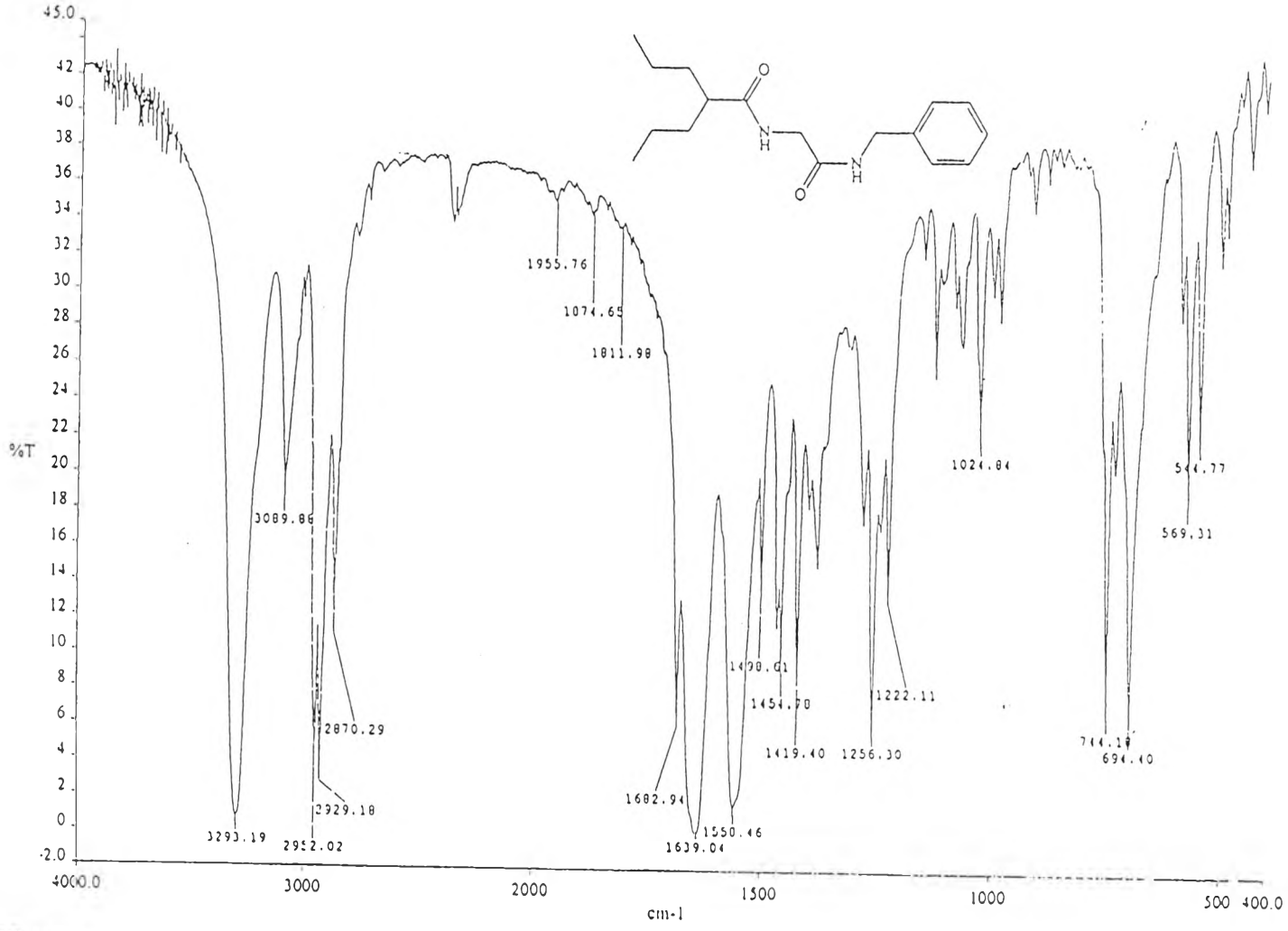

Figure 74. The IR spectrum (KBr) of N-(2-propylpentanoyl)-glycine benzylamide

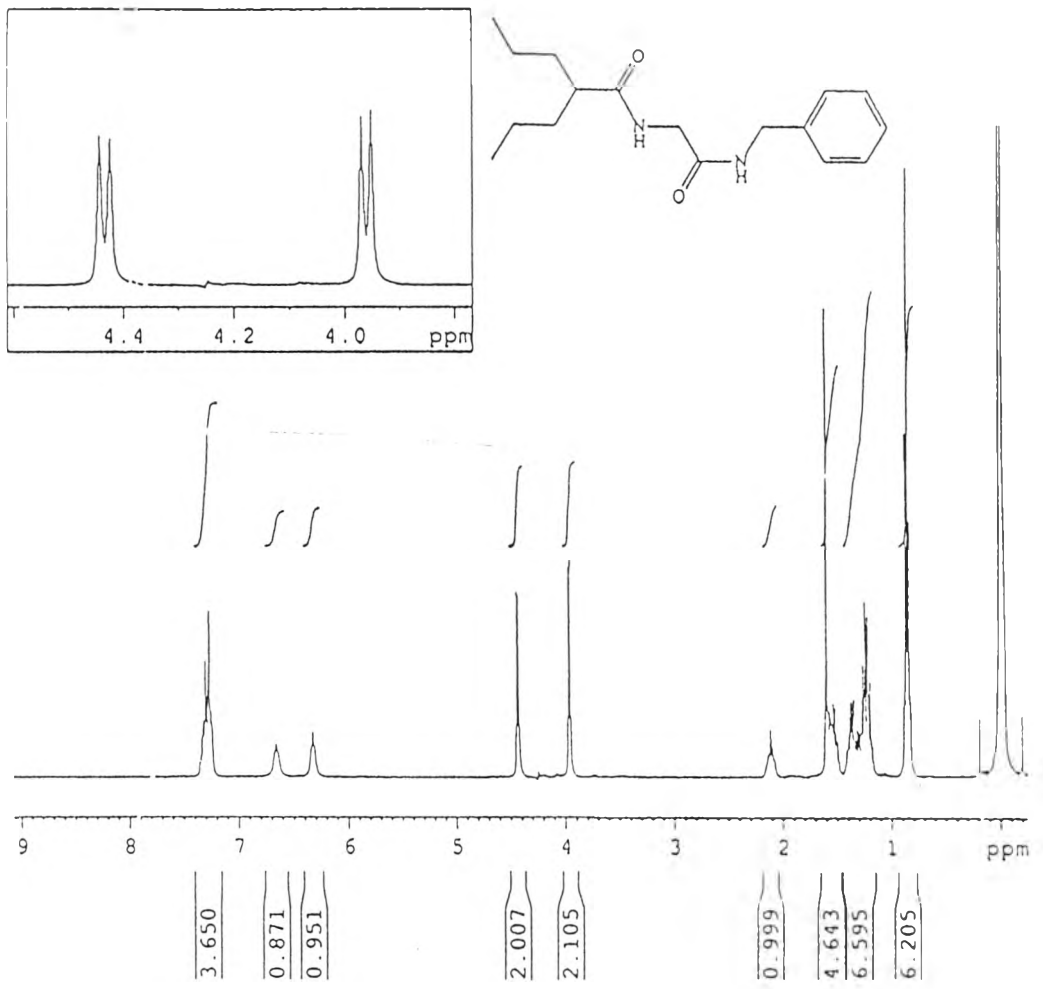

Figure 75. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-glycine benzylamide in $CDCl_3$

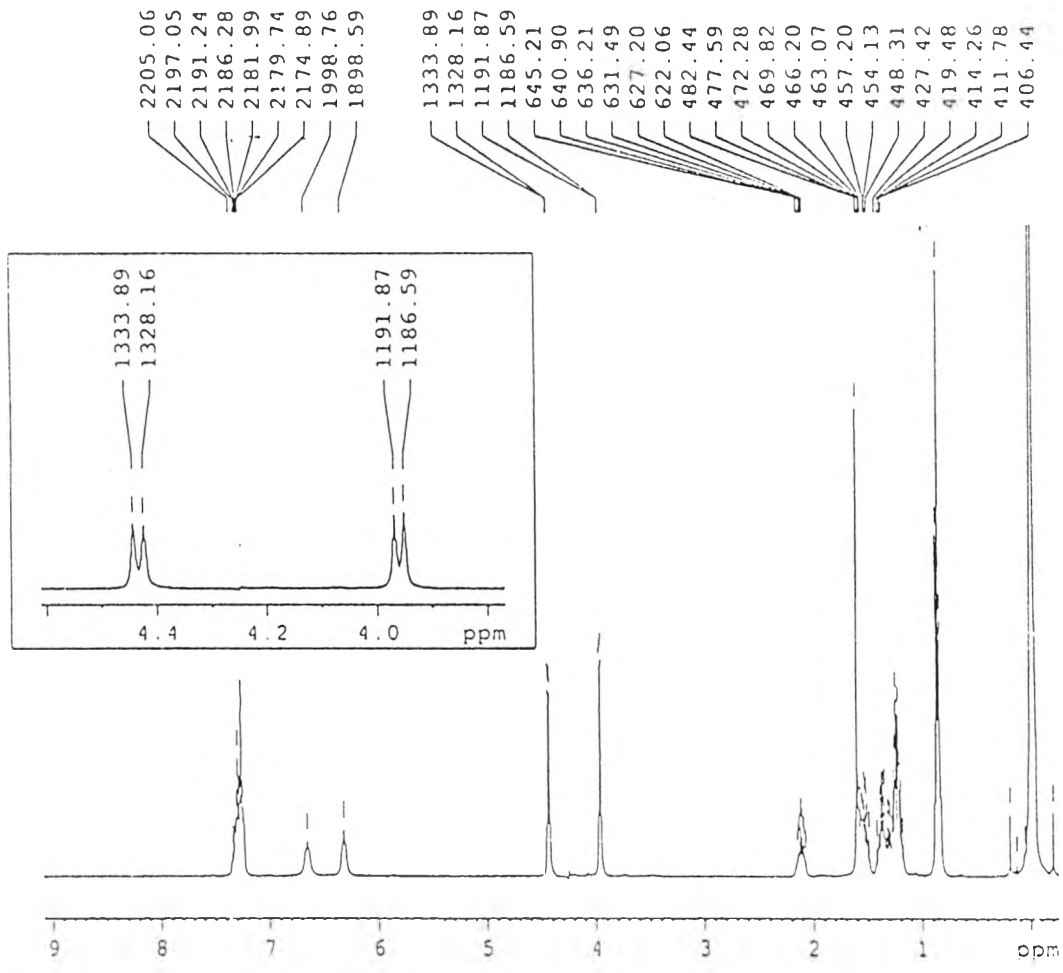

Figure 76. The ^{1}H-NMR spectrum of N-(2-propylpentanoyl)-glycine benzylamide in $CDCl_3$ (Show peaks in Hz)

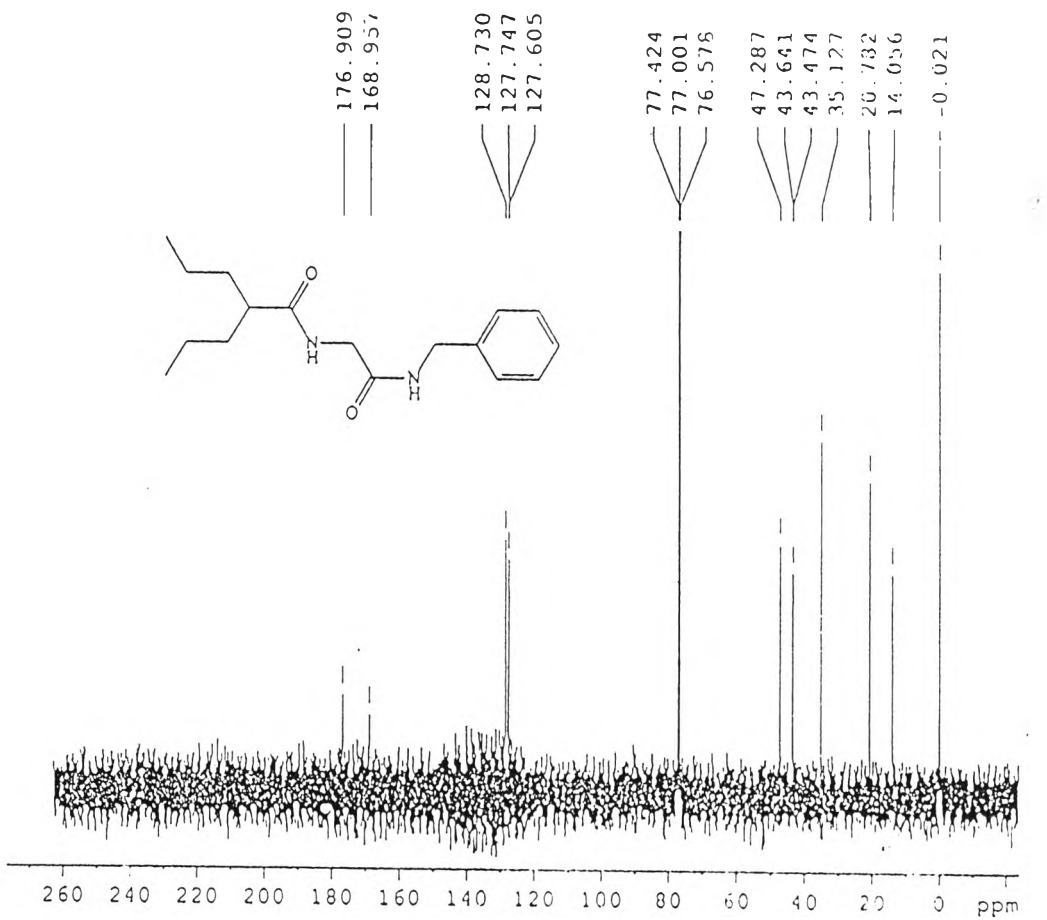

Figure 77. The ^{13}C-NMR spectrum of N-(2-propylpentanoyl)-glycine benzylamide in $CDCl_3$

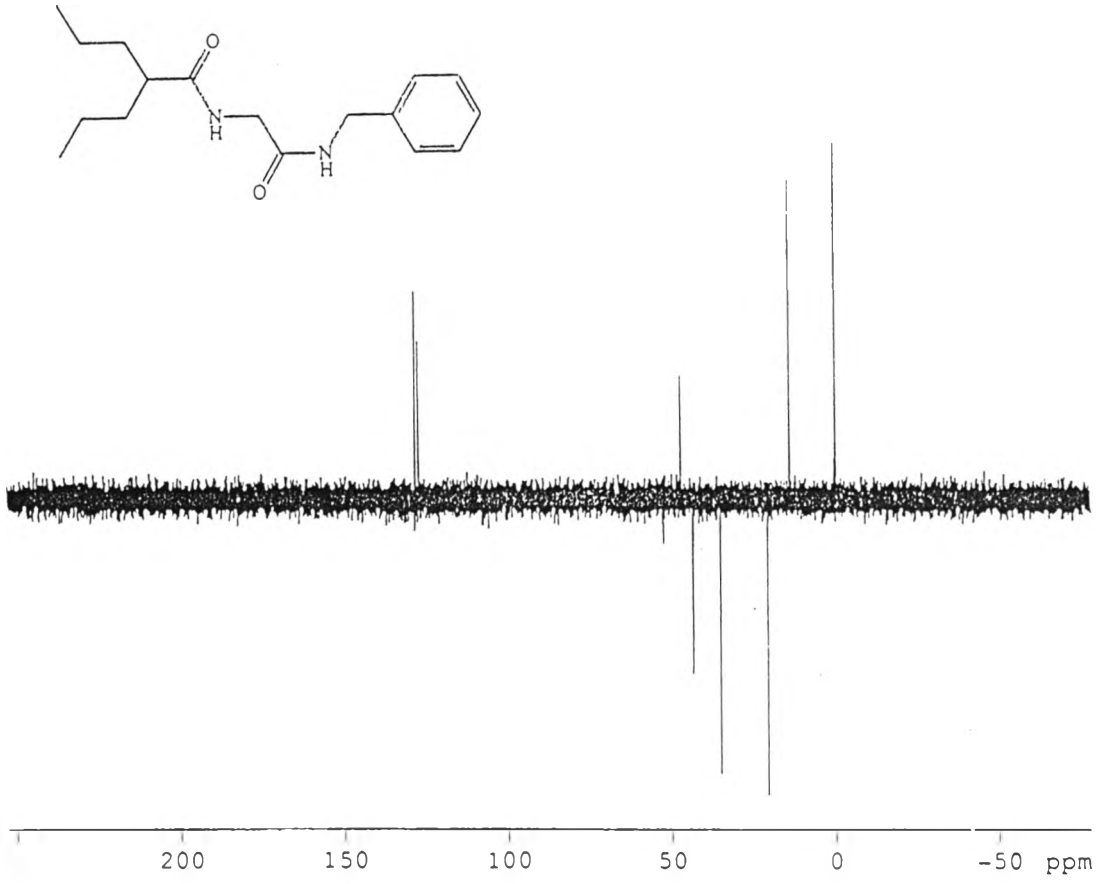

Figure 78. The DEPT 135 spectrum of N-(2-propylpentanoyl)-glycine benzylamide in $CDCl_3$

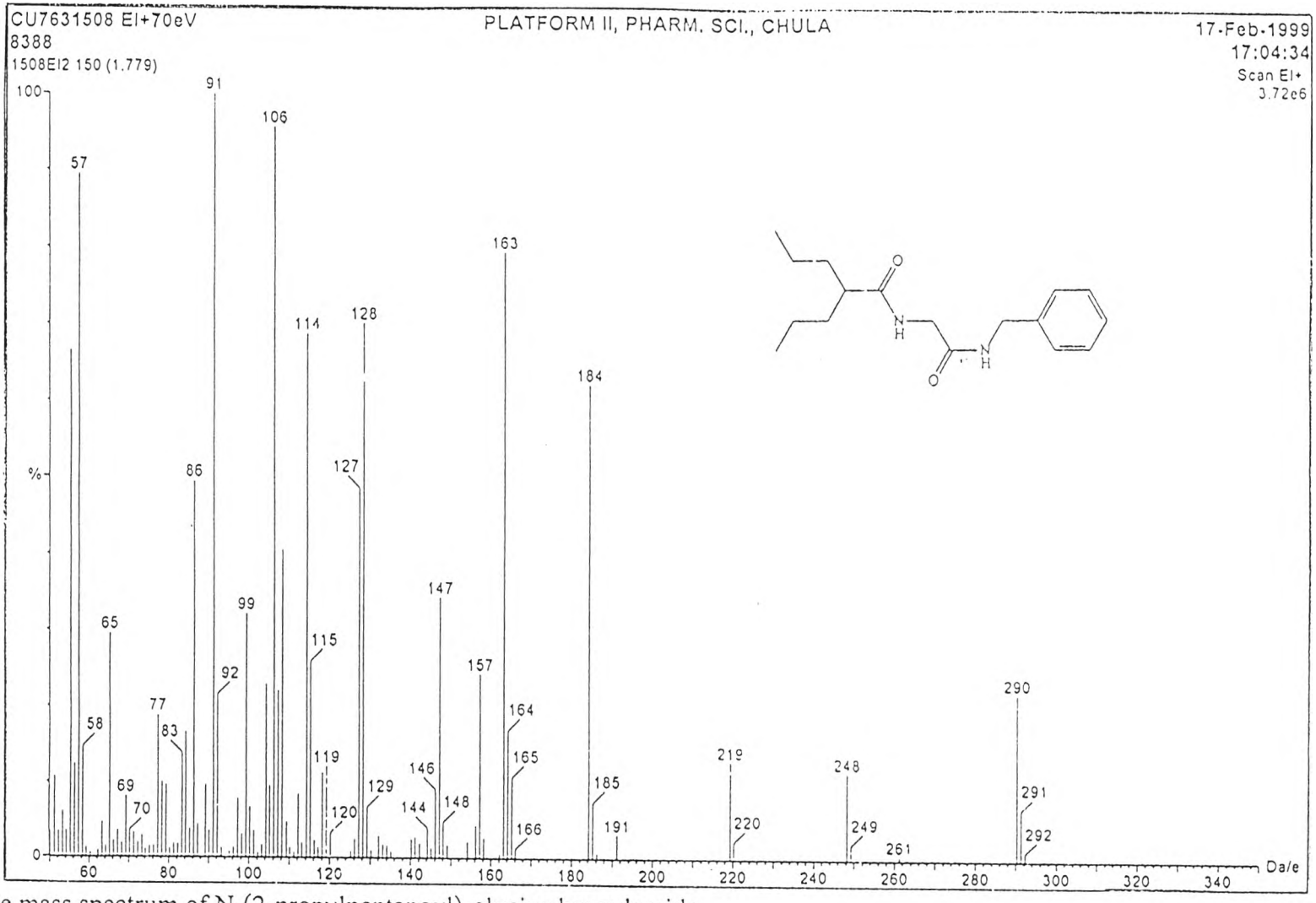

Figure 79. The mass spectrum of N-(2-propylpentanoyl)-glycine benzylamide

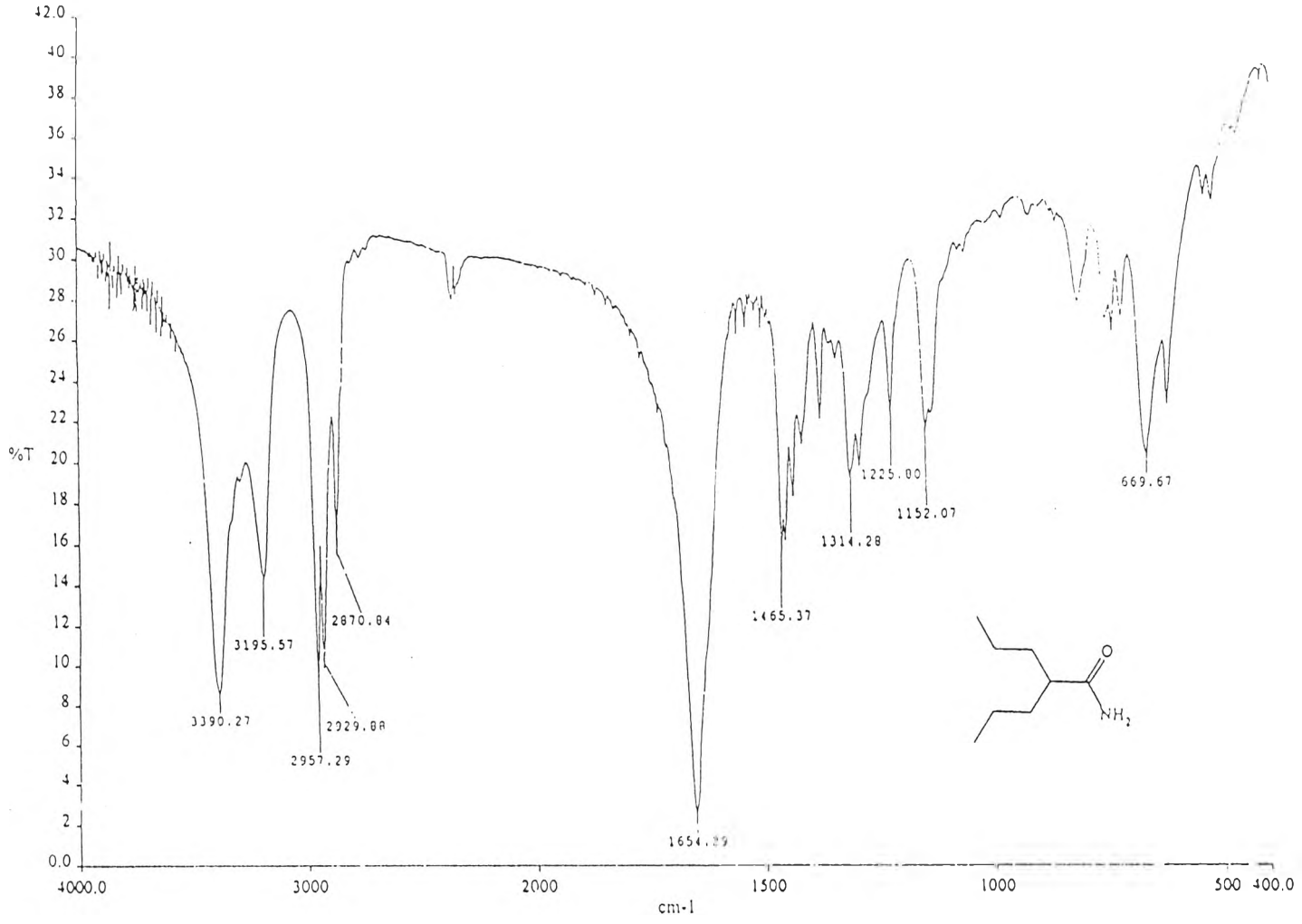

Figure 80. The IR spectrum (KBr) of 2-propylpentamide

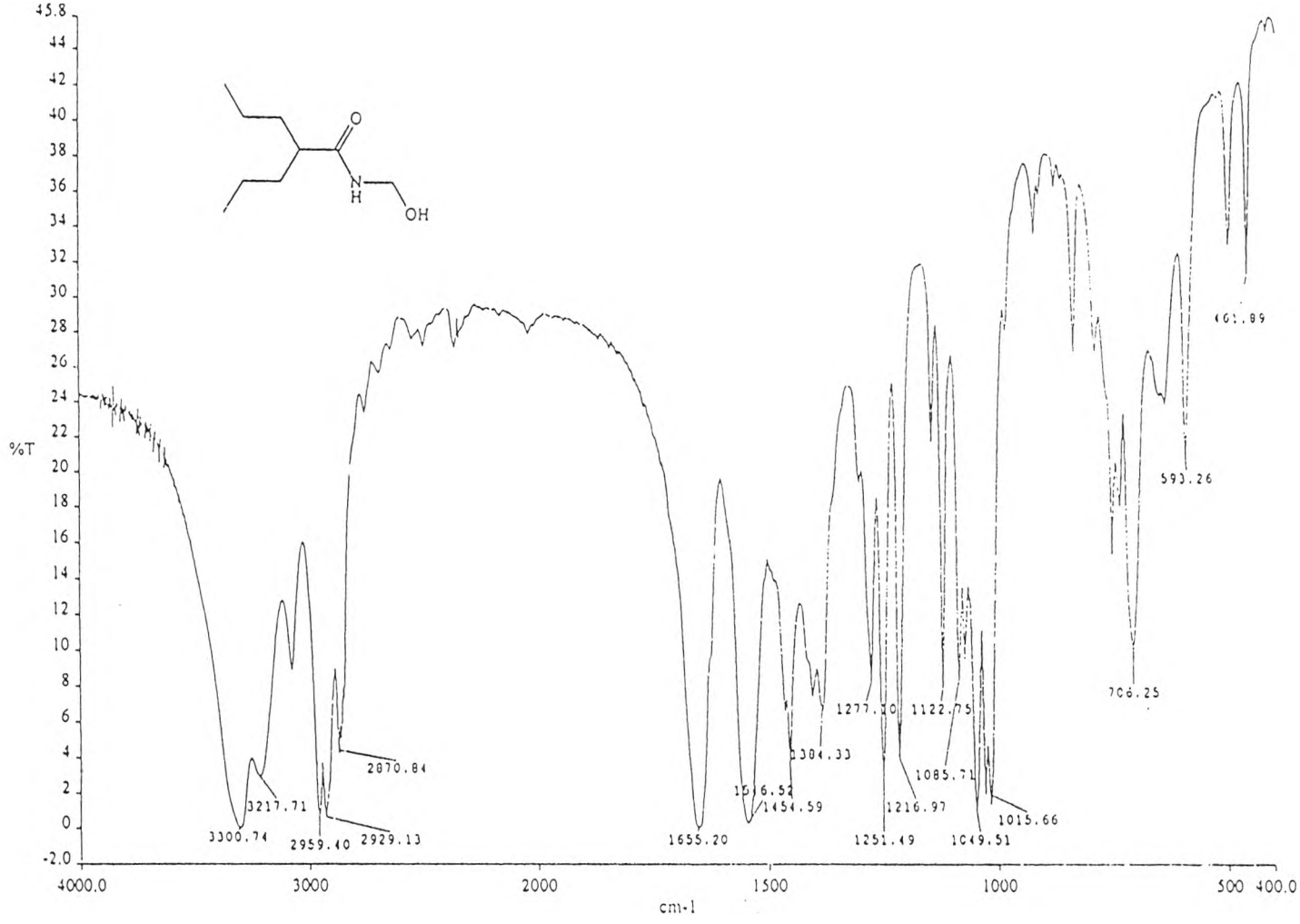

Figure 81. The IR spectrum (KBr) of N-hydroxymethyl-2-propylpentamide

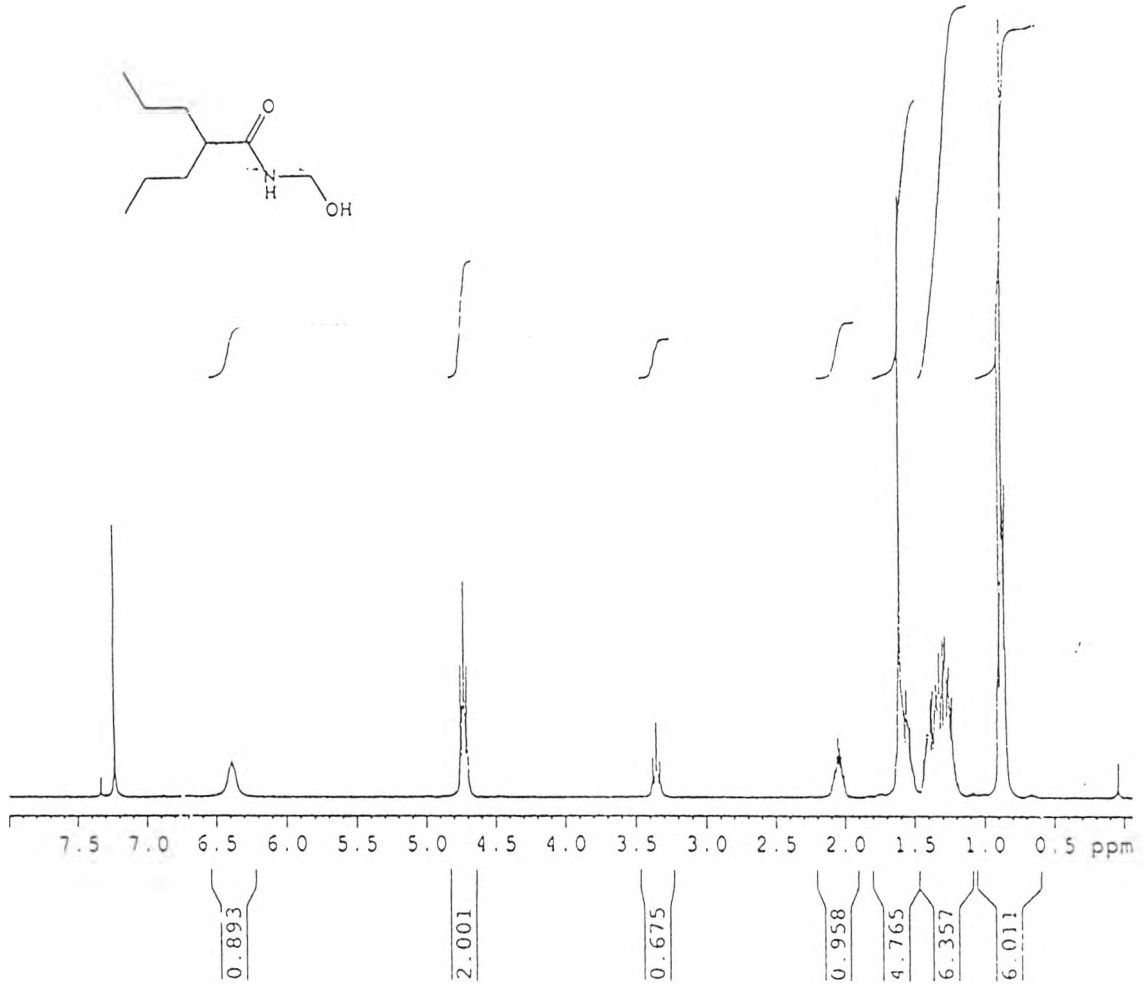

Figure 82. The ^{1}H-NMR spectrum of N-hydroxymethyl-2-propylpentanamide in $CDCl_3$

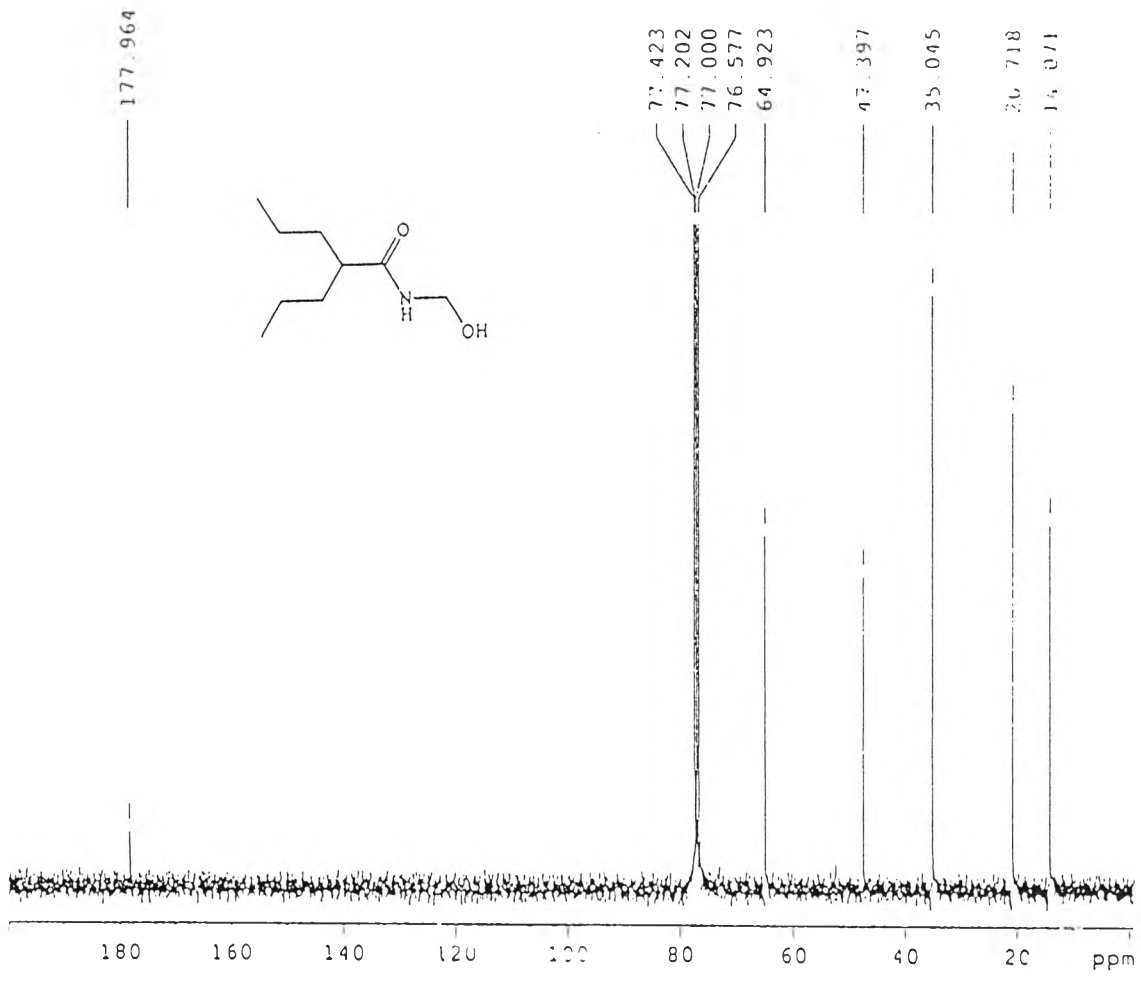

Figure 83. The ^{13}C-NMR spectrum of N-hydroxymethyl-2-propylpentamide

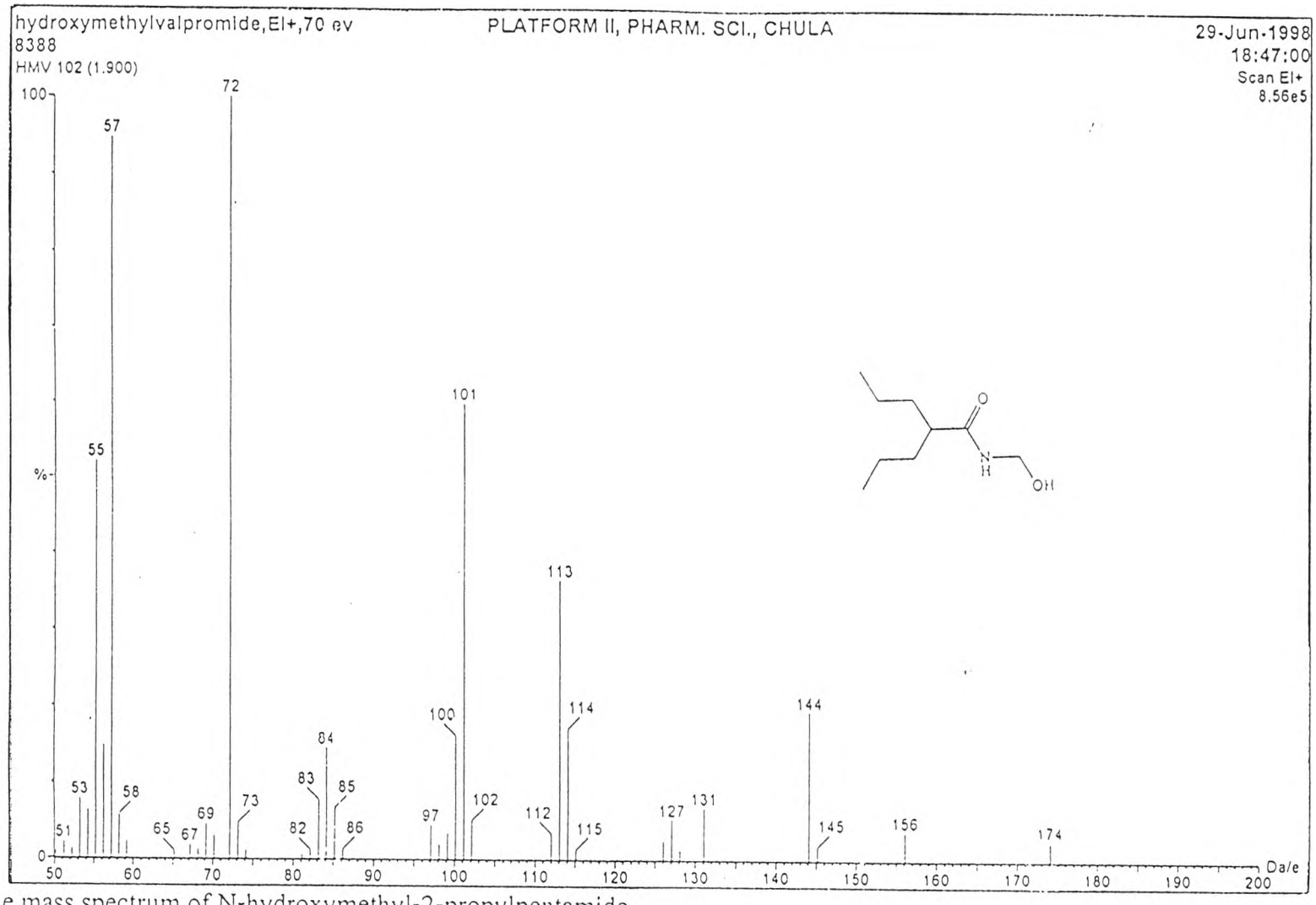

Figure 84. The mass spectrum of N-hydroxymethyl-2-propylpentamide

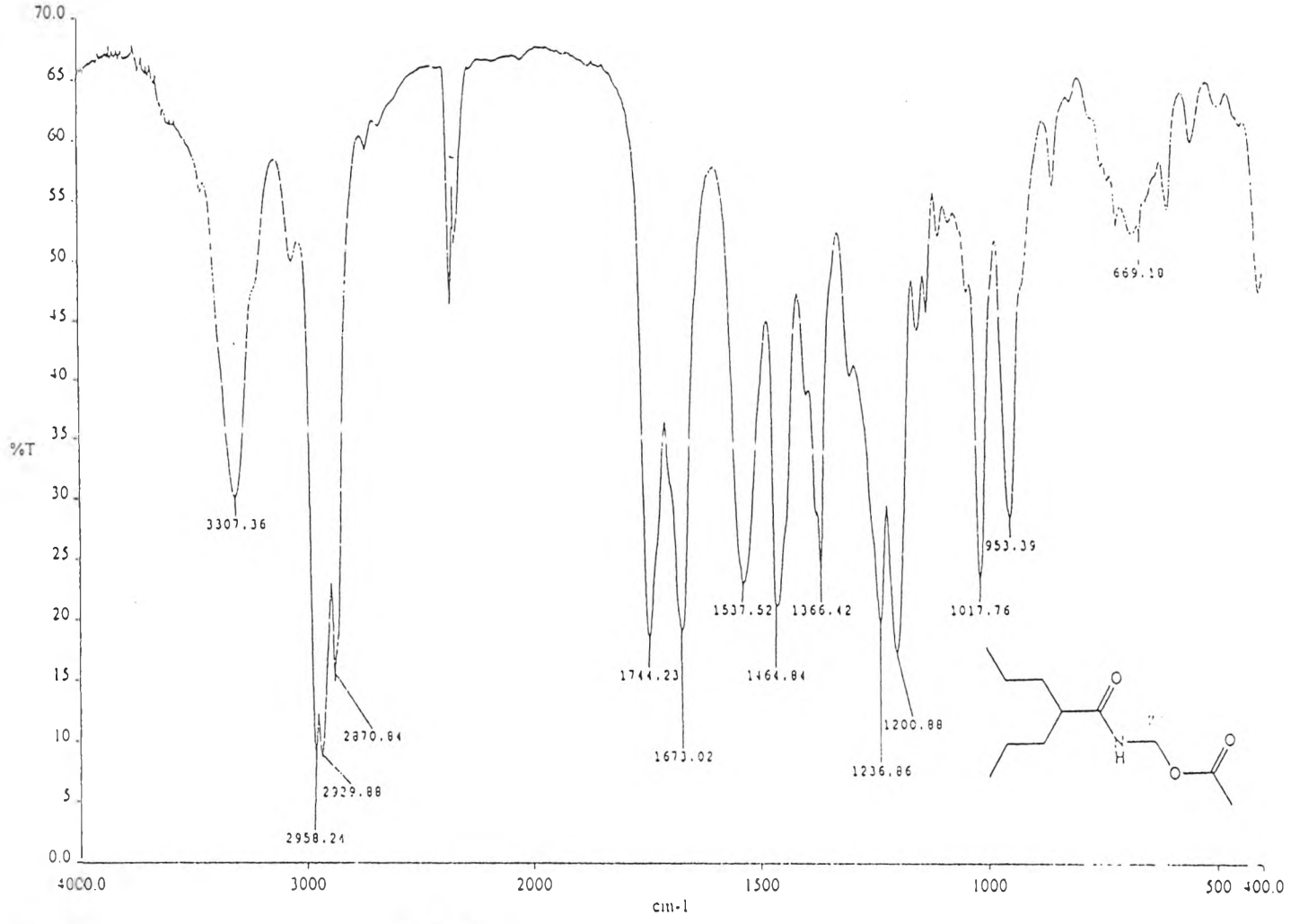

Figure 85. The IR spectrum (KBr) of N-acetoxymethyl-2-propylpentamide

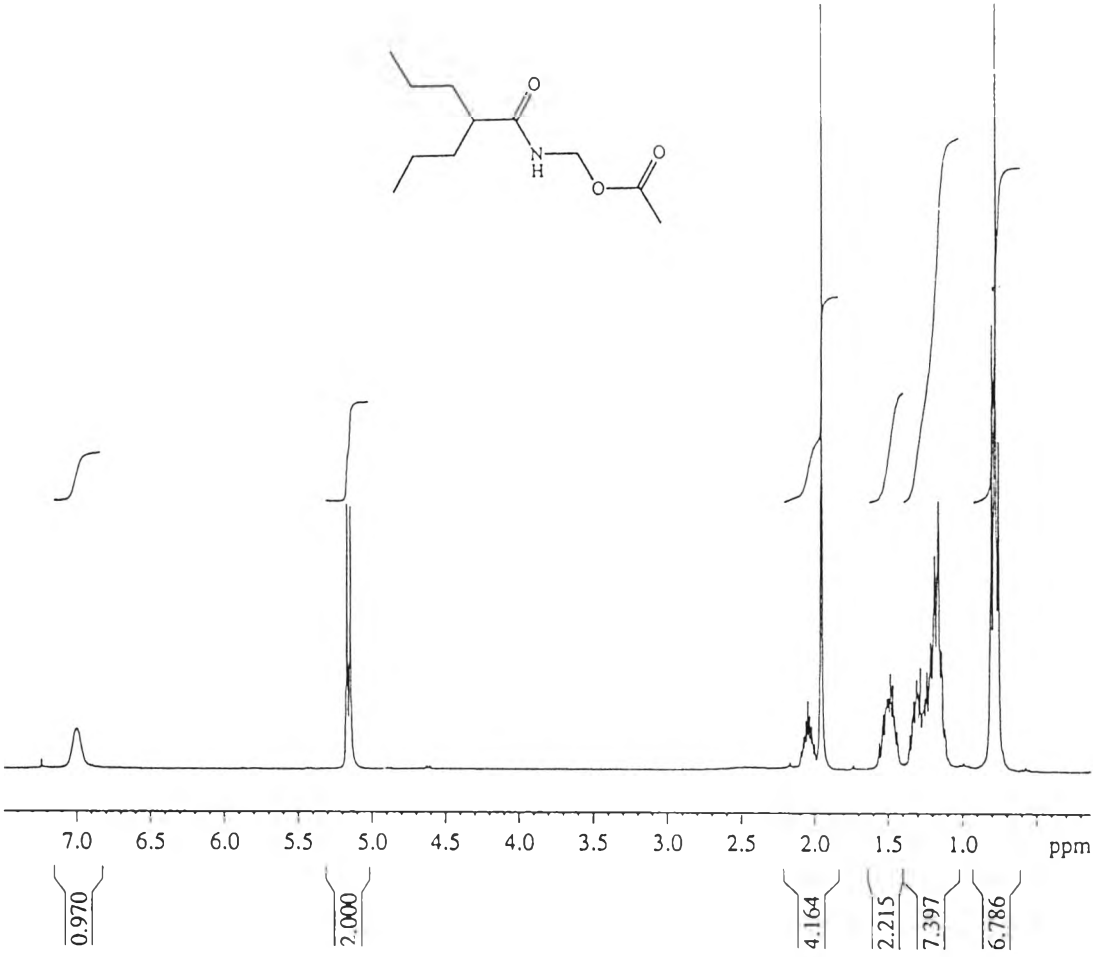

Figure 86. The ^{1}H-NMR spectrum of N-acetoxymethyl-2-propylpentamide in $CDCl_3$

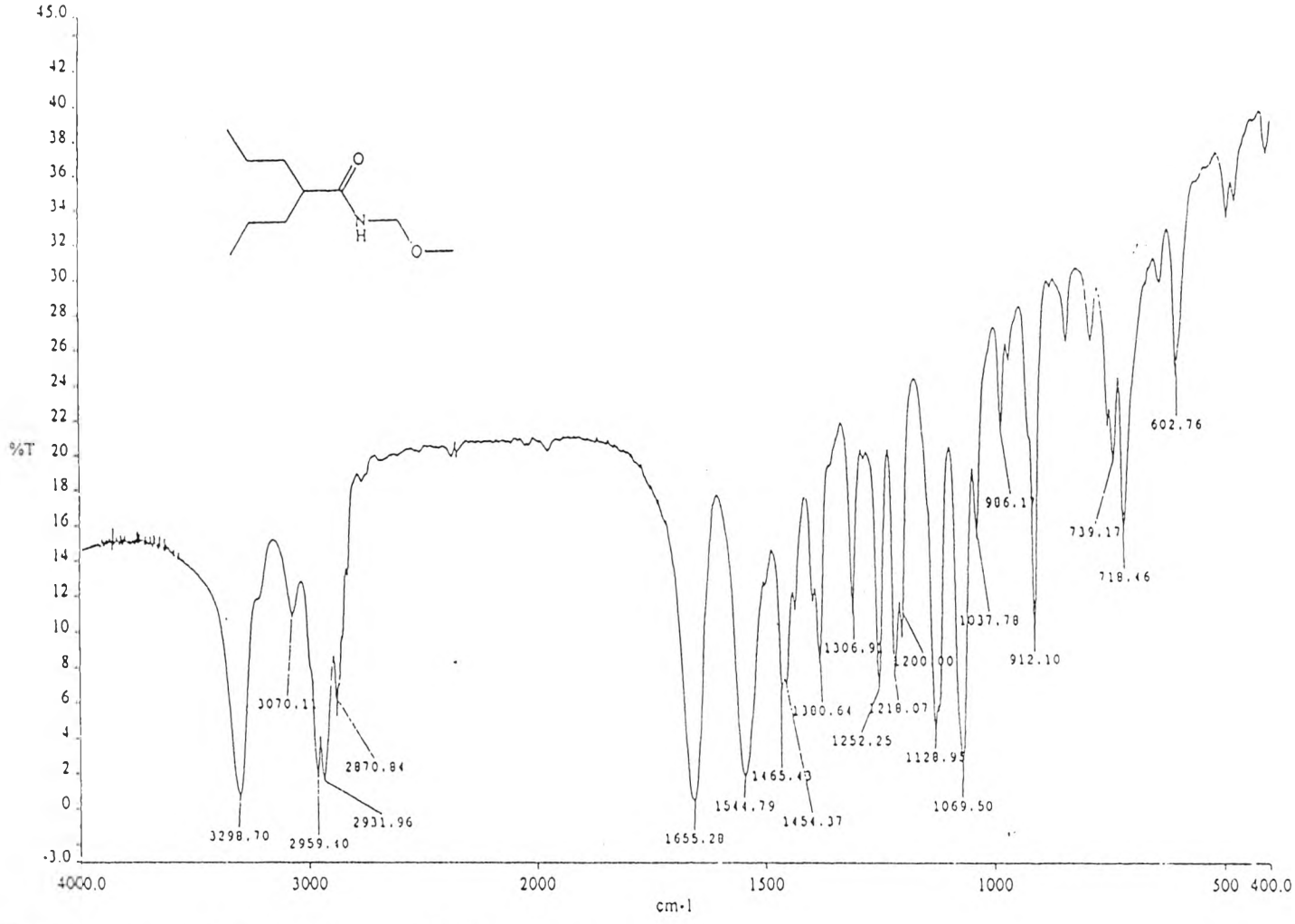

Figure 87. The IR spectrum (KBr) of N-methoxymethyl-2-propylpentamide

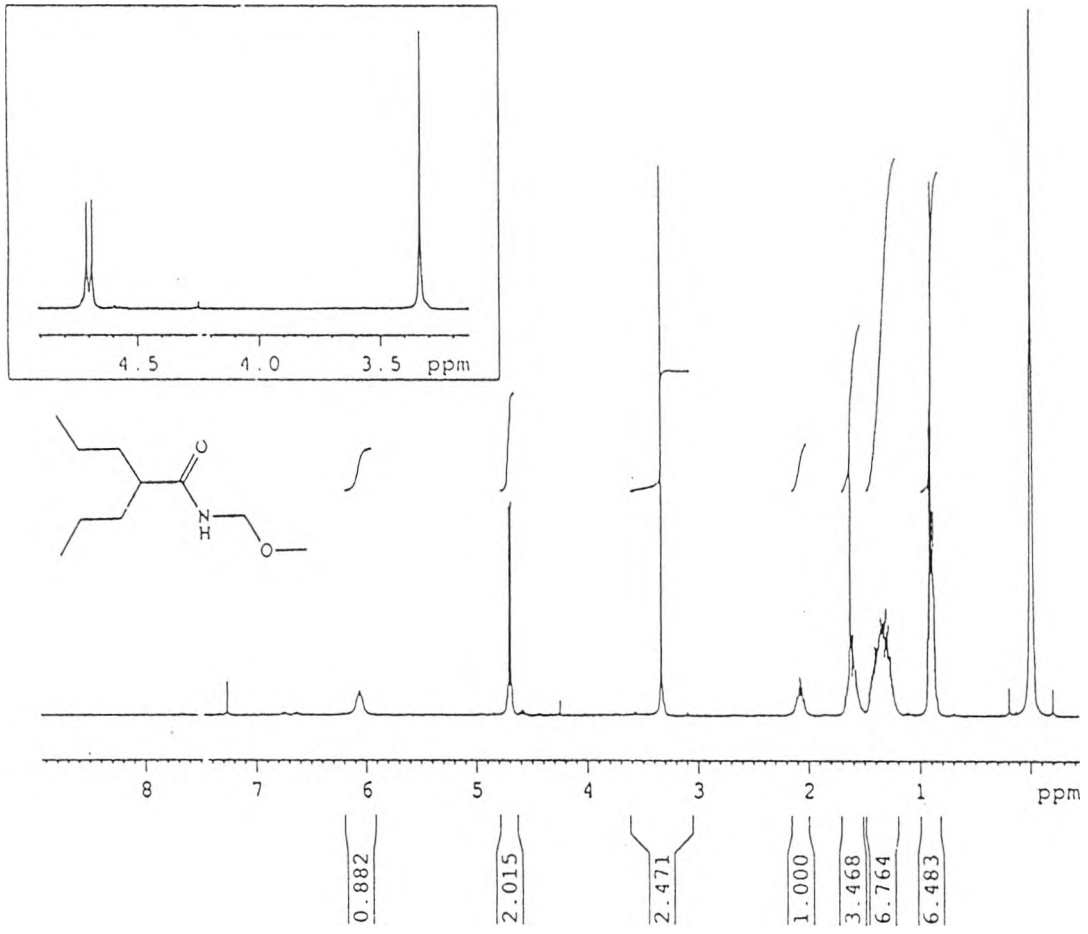

Figure 88. The ^{1}H-NMR spectrum of N-methoxymethyl-2-propylpentamide in $CDCl_3$

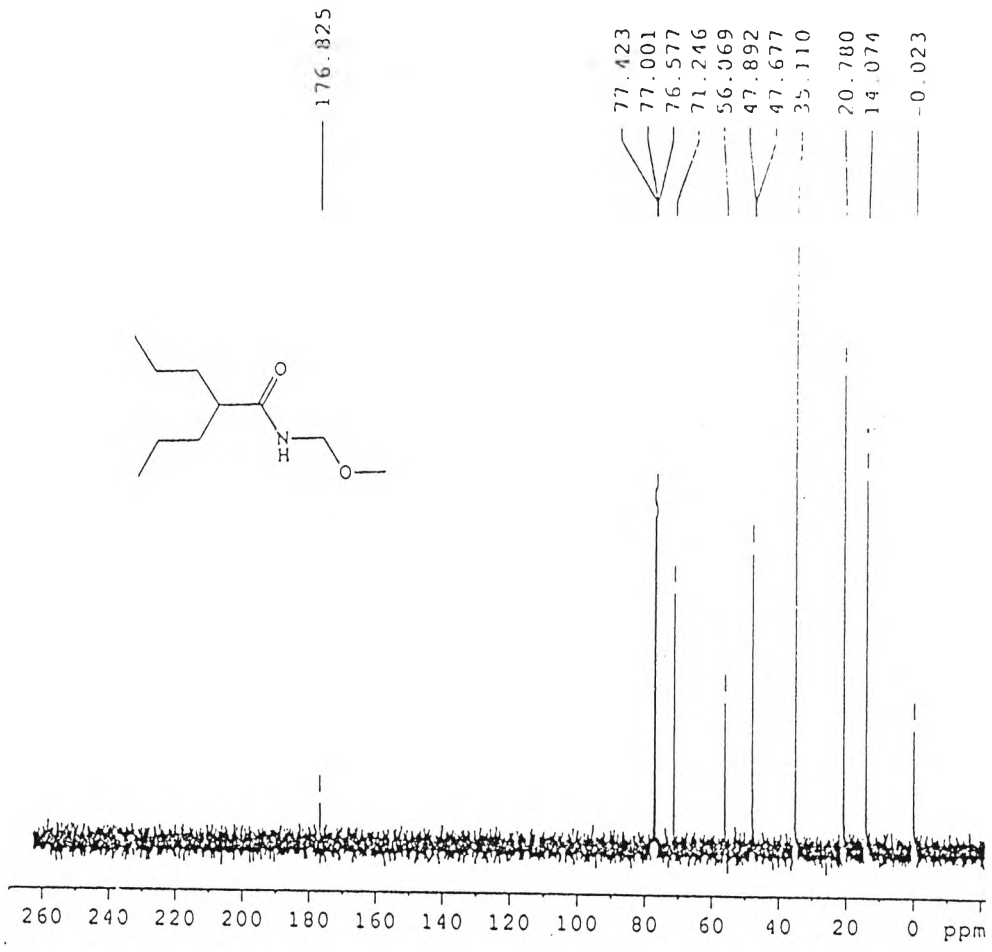

Figure 89. The ^{13}C-NMR spectrum of N-methoxymethyl-2-propylpentamide

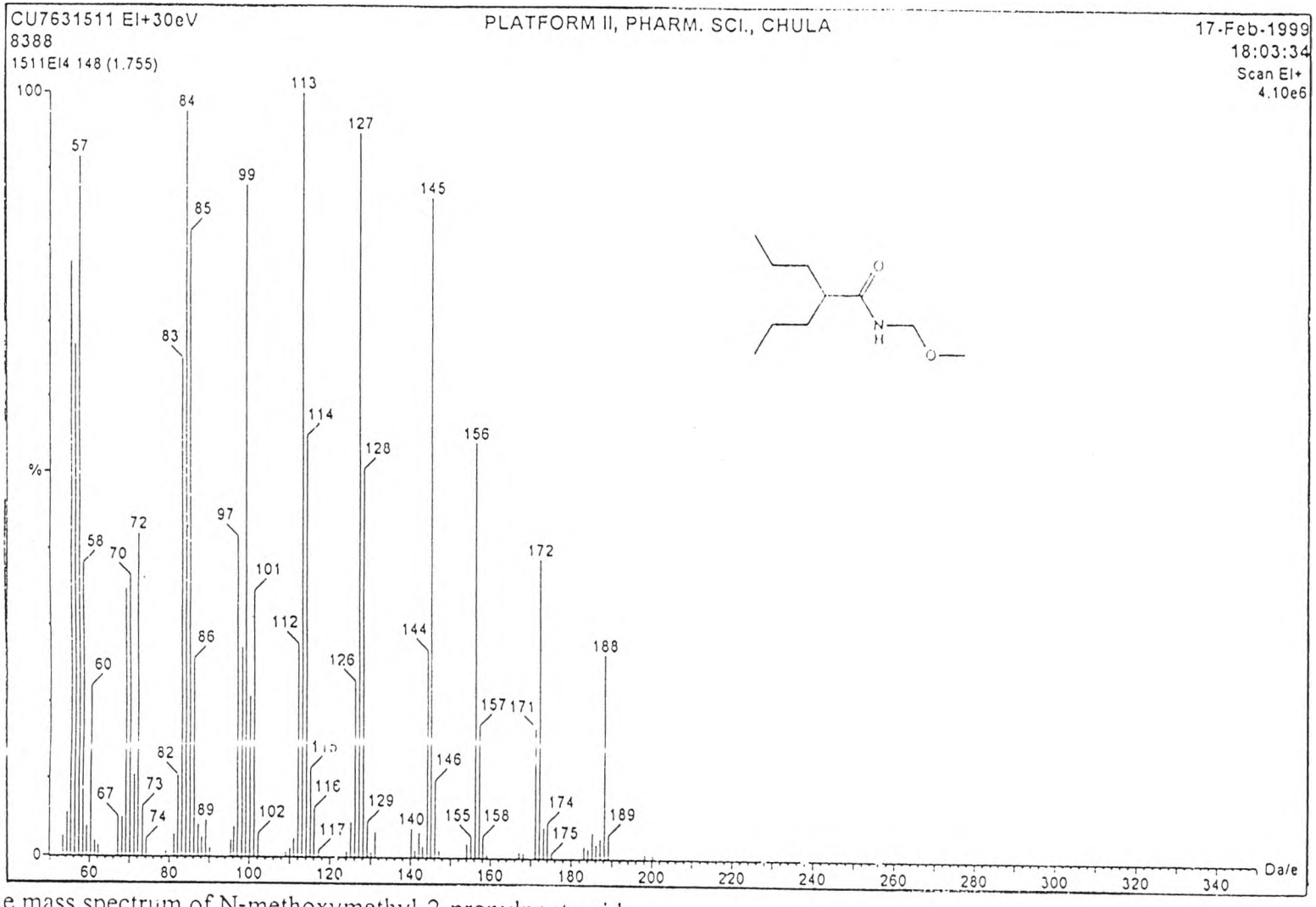

Figure 90. The mass spectrum of N-methoxymethyl-2-propylpentamide