Supporting information:

Oxidative Conversion of α,α-Disubstituted Acetamides to Corresponding One Carbon Shorter Ketones Using Hypervalent Iodine (λ^5) Reagents in Combination With Tetraethylammonium Bromide

*Eknath V. Bellale, Dinesh S. Bhalerao and Krishnacharya G. Akamanchi**

General Methods:

All ^{1}H NMR and ^{13}C NMR spectra were recorded on 60MHz or 300 MHz. Chemical shift values are expressed in δ units relative to tetramethylsilane (TMS) signal as internal reference in CCl_4 or $CDCl_3$. IR spectra were recorded neat or as KBr pallet. All the solvents were purchased from commercial sources and used without further purification. Wherever necessary the solvents were dried by standard literature procedures. Gas chromatography (GC) is used with packed column 5% OV-17, carrier gas N_2, flow rate 2.6 cm^3/min, Injector Temperature 300 ^{o}C, FID detector. High Performance Liquid Chromatography (HPLC) is used with Column RP-18e (5μm), with UV detector, flow rate 1ml/min.

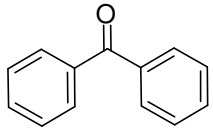
General experimental procedure for conversion of α,α-disubstituted acetamides to ketones:

To a stirred suspension of 10 mmol of hypervalent iodine (λ^5) reagent in 30 ml of CH_3CN was added 5 mmol TEAB and stirred for 5 min, a yellow suspension was observed. To which was added α,α-disubstituted acetamide (5 mmol) in one portion at RT. The temperature of the reaction mixture was raised to 60 ºC and stirred until complete consumption of starting material as observed by TLC. Acetonitrile was removed under reduced pressure and resultant residue was suspended in 30 ml of chloroform, stirred and filtered. The filtrate was washed with 30 ml of 10% sodium bisulfite solution, 30 ml of saturated sodium carbonate solution and 30 ml of brine. The organic layer was dried over anhydrous sodium sulfate and concentrated to give crude ketone. Pure ketone was isolated after silica gel column chromatography (ethyl acetate: hexane = 5: 95)

Spectral Data:

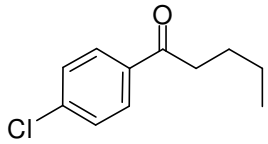
IR and NMR data were identical with those of authentic sample and data are given below: -

Benzophenone 2a



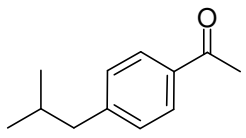
White solid, mp 47-48 ºC (lit.[1] mp 47.5 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.80-7.78(d, *J* = 7.8Hz, 4H), 7.59-7.54(t, *J* = 7.35Hz, 2H) 7.49-7.44(t, *J* = 7.5Hz, 4H) ppm. IR (KBr) ν_{max}: 3060, 3030 and 1659 cm^{-1}.

1-(4-Chlorophenyl)pentan-1-one 2b



Colorless liquid, (lit.[2] bp 126-129 ºC). ^{1}H NMR (60 MHz, CCl_4): δ 1.82-0.96(m, 7H), 2.86(t, *J* = 6.6 Hz, 2H),7.36(d, *J* = 9Hz, 2H), 7.86(d, *J* = 9Hz, 2H,) ppm. IR (neat)ν_{max}: 2959, 1688, 1590, 1400 and 1092 cm^{-1}. HPLC Purity: 99.94 %

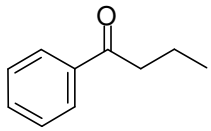
1-(4-Isobutylphenyl)ethanone 2c



Colorless liquid, (lit.[3] $bp_{1.5}$ 94-100 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89-7.86(d, *J* = 7.8Hz, 2H),

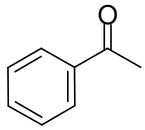
7.24-7.21(d, *J* = 7.8Hz, 2H), 2.58(s, 3H), 2.54-2.52(d, *J* = 7.2Hz, 2H), 1.96-1.81(m, 1H), 0.92-0.89(d, *J* = 6.6Hz, 6H) ppm. IR (KBr) ν_{max}: 3031, 2956, 2925, 1683 and 796 cm^{-1}.

Butyrophenone 2d



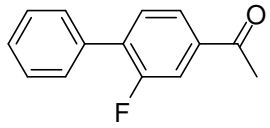
Colorless liquid, (lit.[1] bp 228 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95-7.92(d, *J* = 7.5Hz, 2H), 7.54-7.49(t, *J* = 7.05Hz, 1H), 7.45-7.40(t, *J* = 7.35Hz, 2H), 2.94-2.89(t, *J* = 7.2Hz, 2H), 1.81-1.68(m, 2H), 1.00-0.95(t, *J* = 7.35Hz, 3H)ppm. IR (neat) ν_{max}: 1684, 1599, 1583, 1492, 761 and 691 cm^{-1}.

Acetophenone 2e



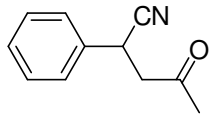
Colorless liquid, (lit.[1] bp 202 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94-7.92(d, *J* = 7.8Hz, 2H), 7.56-7.51(t, *J* = 7.35Hz, 1H), 7.45-7.40(t, *J* = 7.5Hz, 2H), 2.57(s, 3H) ppm. IR (KBr) ν_{max}: 1683, 1490, 3066, 3031 and 1691cm^{-1}.

1-(2-Fluorobiphenyl-4-yl)ethanone 2f



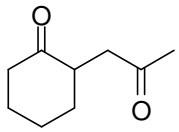
Solid, mp 94-95 ºC, (lit.[4] mp 97 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82-7.72(m, 2H), 7.59-7.42(m, 6H), 2.63(s, 3H) ppm. IR (KBr) ν_{max}: 1686, 1646, 1599, 1583, 1492, 761 and 691 cm^{-1}.

4-Oxo-2-phenylpentanenitrile 2g



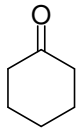
Colorless oil, (lit.[5] mp 47 ºC), ^{1}H NMR (60 MHz, CCl_4): δ 7.36-7.47(s, 5H), 4.35(t, J=7Hz, 1H), 3.22(dd, J=16Hz, 1H), 2.98(dd, J=7.16Hz, 1H), 2.18(s, 3H) ppm. IR (neat) ν_{max}: 1584, 1491, 2236 and 1708 cm^{-1}.HPLC Purity: 98.74 %

2-(2-Oxopropyl)cyclohexanone 2h



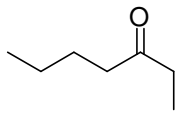
Colorless liquid, (lit.[6] bp 91-93 ºC), ^{1}H NMR (60 MHz, CCl_4): δ 2.170(s, 3H), 1.759(m, 5H), 1.2-0.92 (m, 6H) ppm. IR (KBr) ν_{max}: 2956, 1710, 1696 and 1411 cm^{-1}. HPLC Purity: 98.17 %

Cyclohexanone 2i



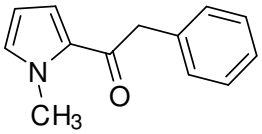
Colorless liquid, (lit.[1] bp 154-156 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.28-2.24(t, *J* = 6.6Hz, 4H), 1.83-1.75(m, 4H), 1.68-1.61(m, 2H) ppm. IR (neat) ν_{max} : 2940, 2867 and 1717cm^{-1}.

3-Heptanone 2j



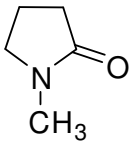
Colorless liquid, (lit.[1] bp 146-149 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.02-1.99(m, 4H), 1.73(m, 2H), 1.53(m, 2H), 1.08-0.83(m, 8H) ppm. IR (KBr) ν_{max} : 2962, 2940, 2867 and 1716 cm^{-1}.

1-(1-methyl-1H-pyrrol-2-yl)-2-phenylethanone 2k



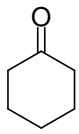
Oil, (lit.[12] oil), ^{1}H NMR (60 MHz, $CDCl_3$): δ 3.95(s, 3H), 4.1(s, 2H), 6.67 (m, 2H), 6.22(m, 8H), 7.1(m, 1H), 7.4(m, 5H) ppm. IR (KBr) ν_{max} : 3011, 2967,1665 cm^{-1}. HPLC Purity: 98.56 %

N-Methyl Pyrrolidone 2l



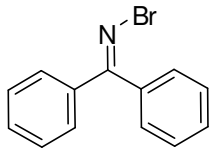
Colorless liquid, (lit.[1] bp 202 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.41-3.37(t, *J* = 8.1Hz, 2H), 2.85(s, 3H), 2.40-2.35(t, *J* = 6.9Hz, 2H), 2.08-1.98(m, 2H), ppm. IR (KBr) ν_{max} : 2927, 2880, 1667 and 1603 cm^{-1}.

Cyclohexanone 2i (Reaction of isocynate with IBX/TEAB)



Colorless liquid, (lit.[1] bp 154-156 ºC), ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.36-2.32(t, *J* = 6.6Hz, 4H), 1.91-1.83(m, 4H), 1.76-1.1.69(m, 2H) ppm. IR (neat) ν_{max} : 2945, 2862 and 1716 cm^{-1}.

N-Bromo-1,1-diphenylmethanimine



Colorless oil, (lit.[7] mp 37 ^{0}C), ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.84-7.81(d, *J* = 7.22Hz, 4H), 7.63-

7.58(t, *J* = 7.35Hz, 2H), 7.52-7.47(t, *J* = 7.5Hz, 4H) ppm. IR (neat) ν_{max}: 3057, 1602, 1282, 842 and 692 cm^{-1}. MS (EI): *m/z* 262 & 260 $[M+H]^+$, 182 $[(Ph)_2C=N^+H.H]^+$, 104$[(Ph)_2C=NH]^+$.

Preparation of α,α-disubstituted acetamides

Method A: Alkylation of arylacetonitrile[9]

To a stirred solution of 10 ml of 50% NaOH solution were added 30 mmol of arylacetonitrile and 10 mole% of benzyltriethylammonium chloride. By maintaining temperature at 30 ^oC, 30 mmol of alkyl bromide was added slowly and stirring continued for 2 h. Then temperature of the reaction mixture was increased to 40 ^oC and maintained for 30 min. The reaction mixture was diluted by addition of ethyl acetate 40 ml. Organic layer was separated, washed with 10% HCl (2 x 20 ml), water (2 x 20 ml), dried over sodium sulphate and concentrated to get crude product and was purified by silica gel column chromatography (ethyl acetate: hexane = 5: 95) afforded the pure product.

Method B: Hydrolysis of nitriles to carboxylic acids

To a solution of NaOH (0.72 g, 18 mmol) in 20 ml of MeOH/Water (1:1) was added 15 mmol of nitrile and the mixture was refluxed for 12h. Methanol was removed under reduced pressure and residue was diluted with 10 ml ethyl acetate and 10 ml water. The aqueous layer was separated and acidified with 10% HCl solution to pH 2 and extracted with ethyl acetate (2 x 10 ml), organic layer washed with water (3 x 30 ml), dried over anhydrous sodium sulfate and concentrated to give crude acid. The crude acid was purified by silica gel column chromatography (ethyl acetate: hexane = 15: 85).

Method C: Amidation of carboxylic acid

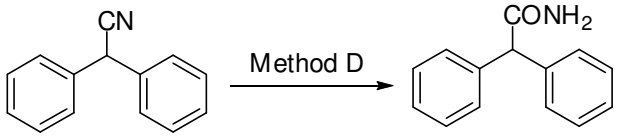
To a stirred thionyl chloride (15 mmol) was slowly added 10 mmol of acid and reaction mixture was warmed to 50 ºC and maintained for 1 h. The reaction mixture was cooled to 10 ºC in ice cold water and carefully, slowly 10 ml of 25% aq NH_3 was added drop wise and stirred for 30 min. The solid

separated out was colleted by filtration, washed with 10% $NaHCO_3$ solution and water. Dried and purified by crystallization.

Method D: Amidation of nitrile

To a stirred solution of (26 mmol) of nitrile in 40 ml of ethanol: water (9:1) was added (26 mmol) of KOH and refluxed for 12 h. Ethanol was distilled off and residue was extracted with ethyl acetate. Organic layer was washed with water (2 x 30 ml), dried over sodium sulphate and concentrated *in vacuo*. The residue was column chromatographed over silica gel (ethyl acetate: hexane = 20:80) to afford pure amide.

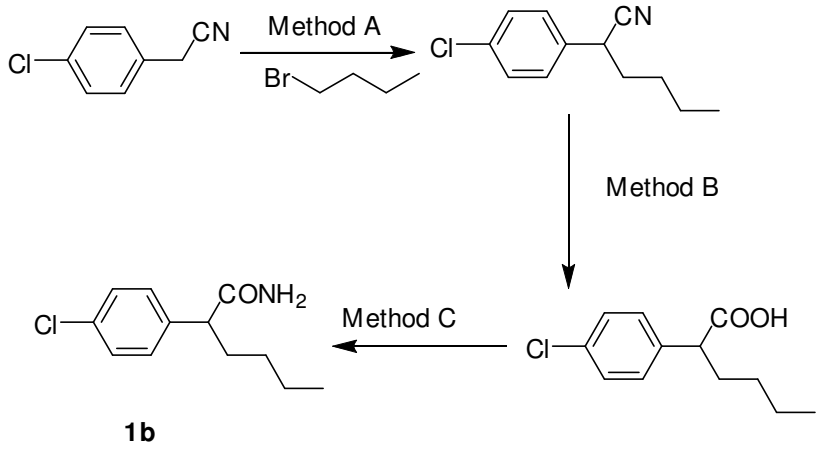
2,2-Diphenylacetamide 1a



1a

White solid. mp 169 ºC (lit.[1] mp 167-168 ºC), ^{1}H NMR (60 MHz, CCl_4): δ 7.48-7.18(m, 10H), 5.615(s, 2H) ppm. IR (KBr) ν_{max}: 3389, 3180, 1654, 1596 and 1494 cm^{-1}.

2-(4-Chlorophenyl)hexanamide 1b

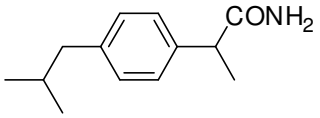


1b

White Solid, mp 127-129 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.31-7.22(m, 4H), 5.63-5.39(bs, 2H),

3.35-3.29(t, J = 7.2 Hz, 1H), 2.07(m, 1H), 1.71(m, 1H), 1.29-1.25(m, 4H), 0.87-0.83(t, 3H) ppm. ^{13}C NMR (300 MHz, $CDCl_3$): 175.6, 138.5, 133.2, 128.9, 129.3, 52.2, 32.9, 29.8, 22.4, 13.9 ppm. IR (KBr) ν_{max}: 3385, 3190 and 1656 cm^{-1}. Elemental analysis calcd for $C_{12}H_{16}ClNO$: C, 63.8; H, 7.1; N,6.2. Found: C, 63.98; H, 7.33; N, 6.19.

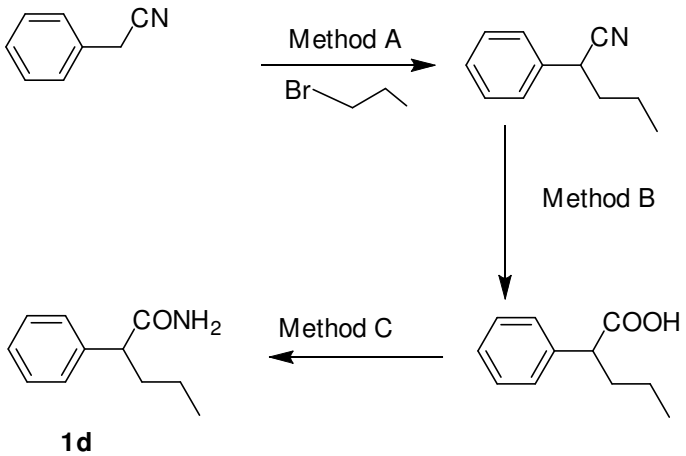
2-(4-isobutylphenyl)propanamide 1c



The amide was prepared by the procedure described in Method C from the corresponding acid.

White Solid, mp 106-107 °C (lit.[8] mp 107-108 ºC), ^{1}H NMR (60 MHz, $CDCl_3$): δ 7.28(d, J = 7.9 Hz, 2H), 7.18(d, J = 7.9 Hz, 2H), 5.5-5.3(bs, 2H), 3.78-3.42(q, J = 7.3 Hz, 1H), 2.53-2.42(q, J = 7.2 Hz, 2H), 1.97-1.88(m, 1H), 1.59-1.49(d, J = 7.3 Hz, 3H), 0.96-0.86(d, J = 6.6 Hz, 6H) ppm. IR (KBr) ν_{max}: 3354, 3201, 1657 and 1641 cm^{-1}.

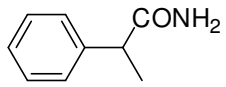
2-Phenylpentanamide 1d



1d

White solid, mp 80-81 °C (lit.[1] mp 83-85 ºC), ^{1}H NMR (60 MHz, $CDCl_3$): δ 7.31(s, 5H), 5.47(bs, 2H), 3.58-3.27(t, 1H), 2.21-0.90(m, 7H) ppm. IR (KBr) ν_{max}: 3409, 3185, 1650, 1617 and 1492 cm^{-1}.

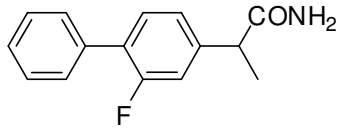
2-Phenylpropanamide 1e



The amide was prepared as per the procedure described in Method C from the corresponding acid.

White solid, mp 93-95 °C (lit.[8] mp 94-95 ºC), ^{1}H NMR (60 MHz, $CDCl_3$): δ 7.21(s, 5H), 5.63-5.73(bs, 2H), 3.31(q, *J* = 7.3 Hz, 1H), 1.1(d, *J* = 7.3 Hz, 3H) ppm. IR (KBr) ν_{max}: 3376, 3174 and 1665 cm^{-1}.

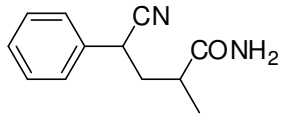
2-(2-fluorobiphenyl-4-yl)propanamide 1f



The amide was prepared as procedure described in Method C from the corresponding acid.

White solid, mp 123-125 °C (lit.[10] mp 122-124 ºC) ^{1}H NMR (60 MHz, $CDCl_3$): δ 7.46-7.06(m, 8H), 5.53(bs, 2H), 3.69-3.45(q, 1H), 1.61-1.49(d, 3H) ppm. IR (KBr) ν_{max}: 3400, 3189, 1659, and 1627, 1560 and 1482 cm^{-1}.

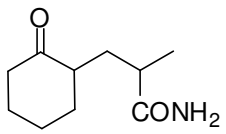
4-Cyano-2-methyl-4-phenylbutanamide 1g



To a stirred solution of 1.34 g (56 mmol) of NaH in 20 ml of DMF was added slowly 6.0 g (51 mmol) of phenylacetonitrile and 4.34 g (51 mmol) of methacrylamide and refluxed for 7 h. The reaction mixture was cooled to room temperature, poured in water, neutralized with 10% dil HCl and extracted with ethyl acetate (2 x 20 ml), organic layer was separated, washed with (3 x 20 ml) water, dried over anhydrous sodium sulfate and concentrated to give crude amide. Purification was carried by silica gel

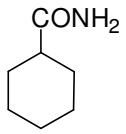
column chromatography (ethyl acetate: hexane = 15: 85) afforded **1g** as a White solid, mp 119-121 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08-8.00(bs, 2H), 7.4-7.2(m, 5H), 3.79-3.73(dd, 1H), 2.78-2.56(m, 1H), 2.30-2.00(m, 2H), 1.40-1.23(dd, 3H) ppm. ^{13}C NMR (300 MHz, $CDCl_3$): 174.9, 137.5, 128.8, 128.5, 127.7, 127.6, 49.6, 37.6, 35.6, 15.0 ppm. IR (KBr) ν_{max}: 3349, 3239, 2185 and 1664 cm^{-1}. Elemental analysis calcd for $C_{12}H_{14}N_2O$: C, 71.29; H, 6.93; N,13.86. Found: C, 71.1; H, 6.83; N, 13.62.

2-Methyl-3-(2-oxo-cyclohexyl)propanamide 1h



To a stirred solution of 1.34 g (56 mmol) of NaH in 20 ml of DMF was added slowly 5.0 g (51 mmol) of cyclohexanone and 4.34 g (51 mmol) of methacrylamide and refluxed for 7 h. The reaction mixture was cooled to room temperature, poured in water, neutralized with 10% dil HCl and extracted in ethyl acetate (2 x 20 ml), organic layer was separated, washed with (3 x 20 ml) water, dried over anhydrous sodium sulfate and concentrated to give crude amide. Purification was carried by silica gel column chromatography (ethyl acetate: hexane = 15: 85) afforded **1h** as a White solid, mp 109-111 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.85 (bs, 2H), 2.55-2.47(m, 1H), 2.23-2.16(m, 1H), 2.06-2.00(m, 5H) 1.74-1.66(m, 5H), 1.21-1.19(d, 3H) ppm.^{13}C NMR (300 MHz, $CDCl_3$): 174.6, 128.1, 109.4, 35.0, 34.2, 27.9, 25.9, 22.7, 22.3, 15.7 ppm. IR (KBr) ν_{max} : 3217, 3175, 2936, 2833, 1706 and 1659 cm^{-1}. Elemental analysis calcd for $C_{10}H_{17}NO_2$: C, 65.5; H, 9.3; N, 7.7. Found: C, 65.72; H, 9.27; N, 7.5.

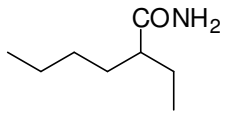
Cyclohexanecarboxamide 1i



The amide was prepared as procedure described in Method C from the corresponding acid.

White solid, mp 180-182 °C (lit.[1] mp 186-187 ºC) ^{1}H NMR (60 MHz, $CDCl_3$): δ 5.51(bs, 2H), 1.84-1.36(m, 11H) ppm. IR (KBr) ν_{max}: 3380, 3185, 2927, 2853 and 1655 cm^{-1}.

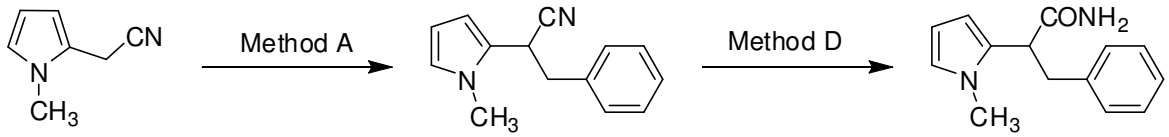
2-Ethylhexanamide 1j



The amide was prepared as procedure described in Method C from the corresponding acid.

White solid, mp 101-104 °C (lit.[1] mp 102-103 ºC) ^{1}H NMR (60 MHz, $CDCl_3$): δ 5.60(bs, 2H), 1.86-1.84(m, 1H), 1.51-0.88(m, 14H) ppm. IR (KBr) ν_{max}: 3342, 3165, 2927, 2851 and 1669 cm^{-1}.

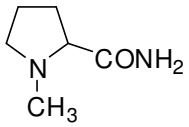
2-(1-methyl-1H-pyrrol-2-yl)-3-phenylpropanamide 1k



1k

White solid, mp 111-112 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25-7.12 (m, 5H), 6.54(s, 1H), 6.13-6.07(m, 2H), 5.56(bs, 2H) 3.79-3.73(m, 1H), 3.48-3.34(m, 4H), 3.15-3.08(m, 1H) ppm.^{13}C NMR (300 MHz, $CDCl_3$): 174.8, 139.2, 130.1, 128.9, 128.3, 126.4, 122.9,107.3,107.31, 47.1, 38.4, 33.7 ppm. IR (KBr) ν_{max} : 3217, 3175, 2936, 2833, 1655 cm^{-1}. Elemental analysis calcd for $C_{14}H_{16}N_2O$: C, 73.66; H, 7.06; N, 12.27. Found: C, 73.57; H, 7.02; N, 12.23.

1-methylpyrrolidine-2-carboxamide 1l



White solid, mp 139-141 °C (lit.[11] mp 140-141 ºC) ^{1}H NMR (300 MHz, $DMSOd_6$): δ 1.75-1.63 (m, 3H), 1.90-2.15 (m, 6H), 3.14-2.99(m, 1H), 7.20 (bs, 2H) ppm. IR (KBr) ν_{max}: 2925, 2853 and 1659 cm^{-1}.

References:

1. *Dictionary of Organic compounds*, 6th ed.; Bukiyahan J., Macdonald, F., Chapman and Hall Electric Publishing House, London, 1996.

2. Friour, G.; Normant, J. F. *Synthesis* **1984**, 37.

3. Parrinello, G.; Stille, J. K. *J. Am. Chem. Soc.* **1987**, *109*, 7122.

4. Takeda, M.; Taniyama, E.; Ozawa, Y.; Imanari, M.; Takahashi, K.; Iwane, H. U.S.Patent 4 263 457, 1981.

5. Agami, C.; Levisalles, J.; Puchot, C. *J. Org. Chem*. **1982**, *47*, 3561.

6. Jacobson, R. M.; Raths, R. A.; McDonald, J. H. *J. Org. Chem.* **1977**, *42*, 2545.

7. Theilacker, W.; Fauser, K. *Justus Liebigs Ann. Chem*. **1939**, *539*, 103.

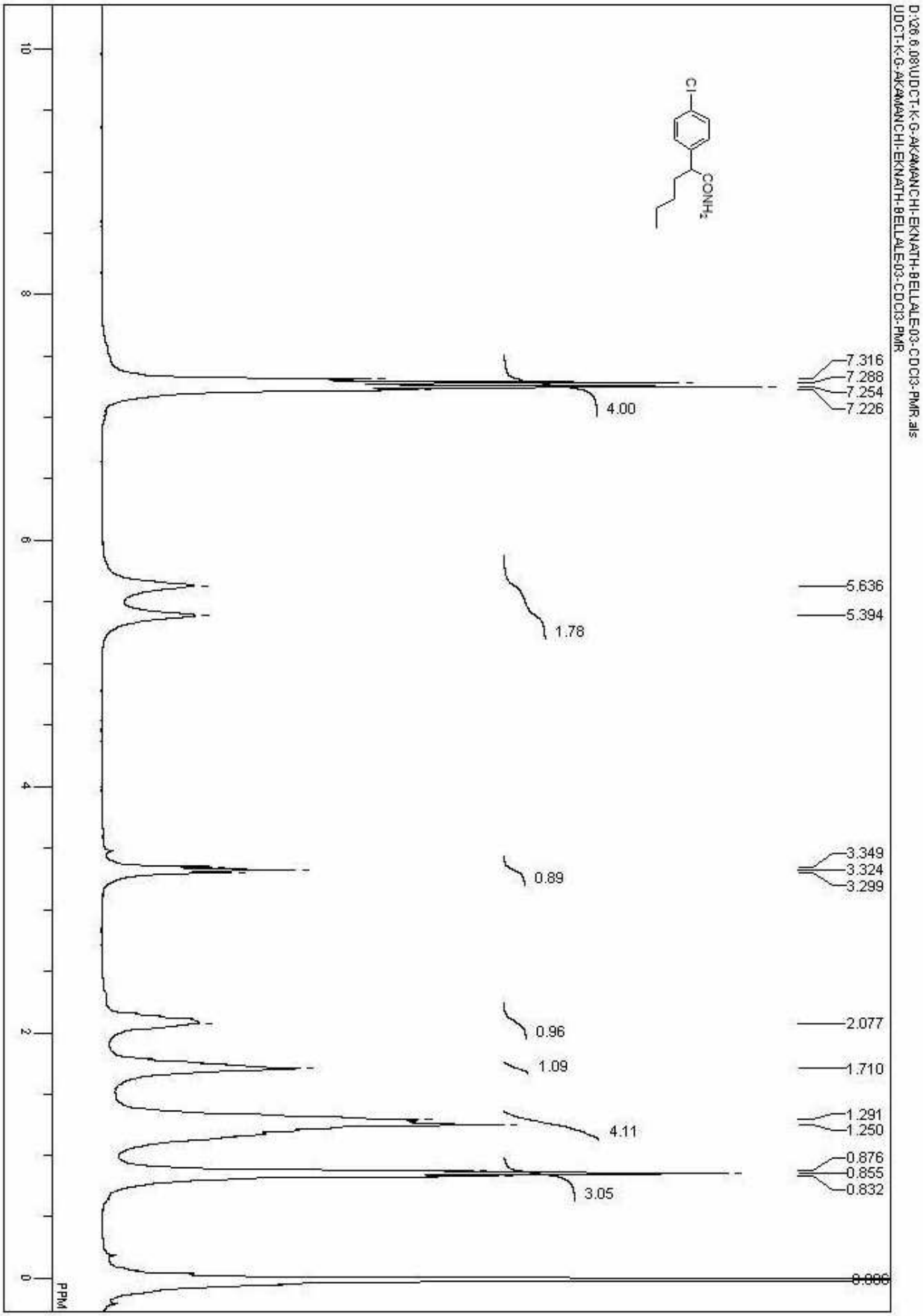
8. Snell, D.; Colby, J. *Enzyme Microb. Technol.* **1999**, *24*, 160.

9. Makosza, M. and Jonczyk, A. *Organic Syntheses, Coll. Vol. 6,* **1988**, 897.

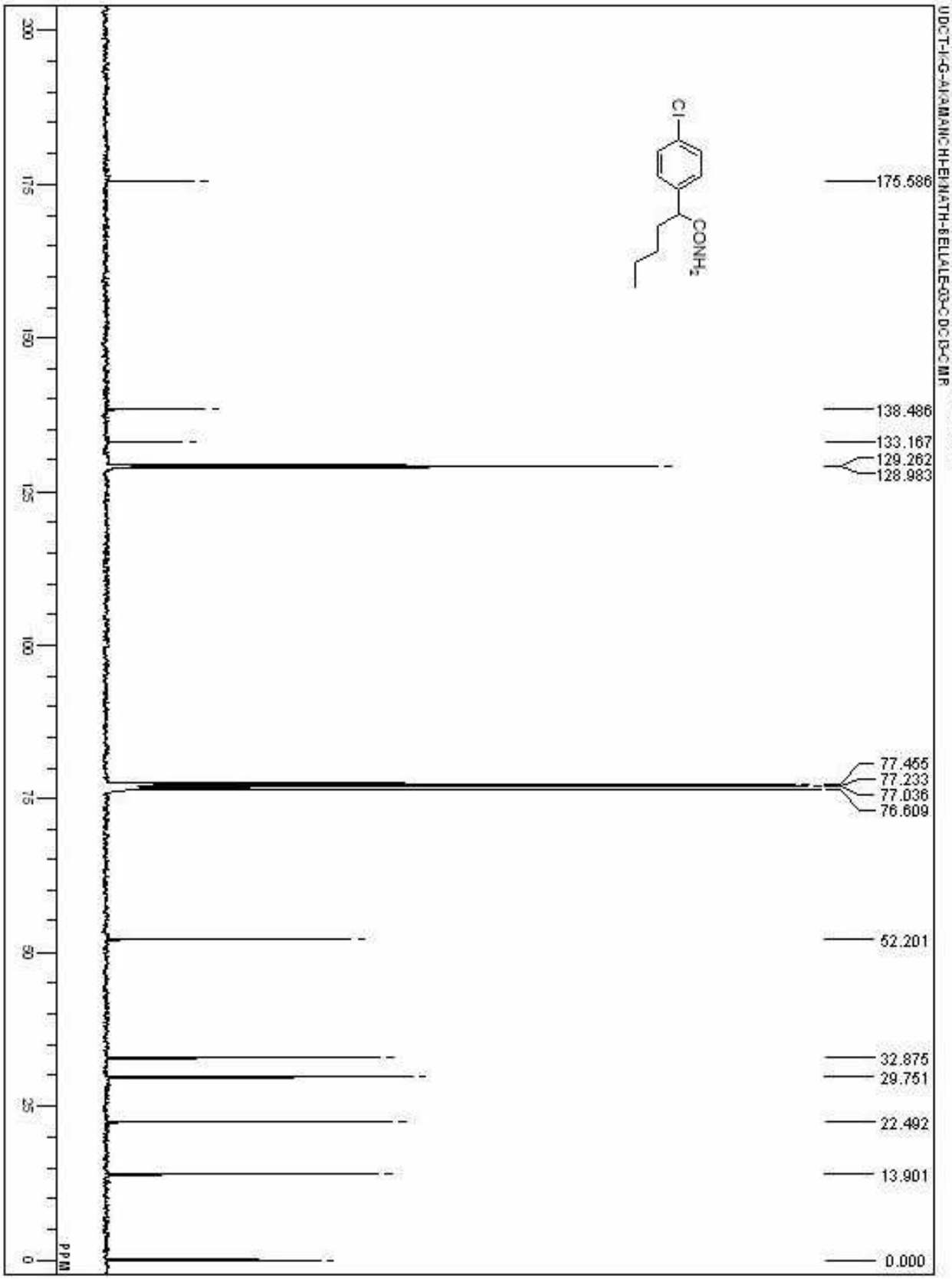
10. Satyam, A. World Patent 027711, 2006.

11. Sassaman, M. B.; Giovanelli, J.; Sood, V. K.; Eckelman, W. C. *Bioorg. Med. Chem.* **1998**, *6*, 1759.

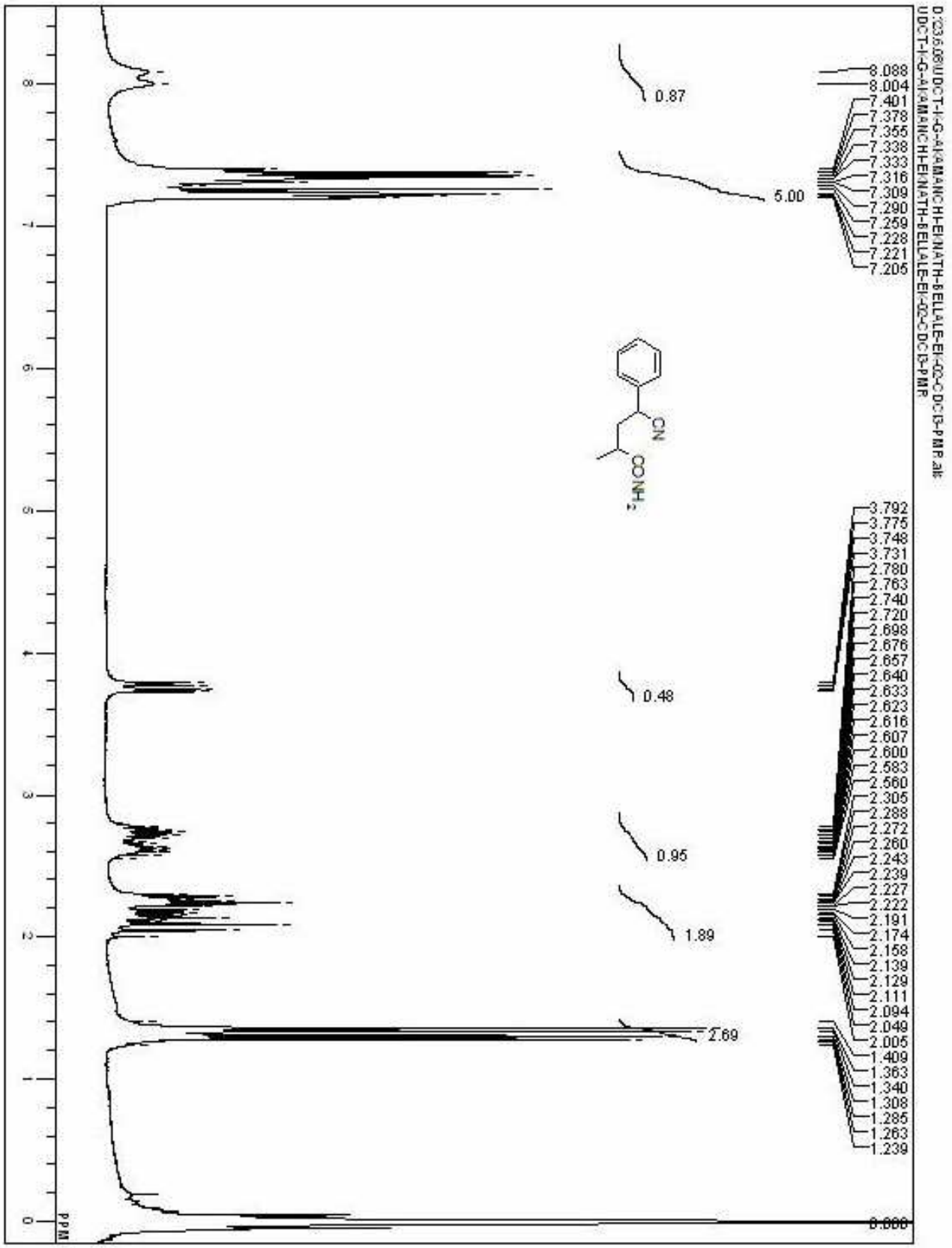
12. Greenhouse, R.; Ramirez, C. *J. Org. Chem.* **1985**, *50*, 2961.



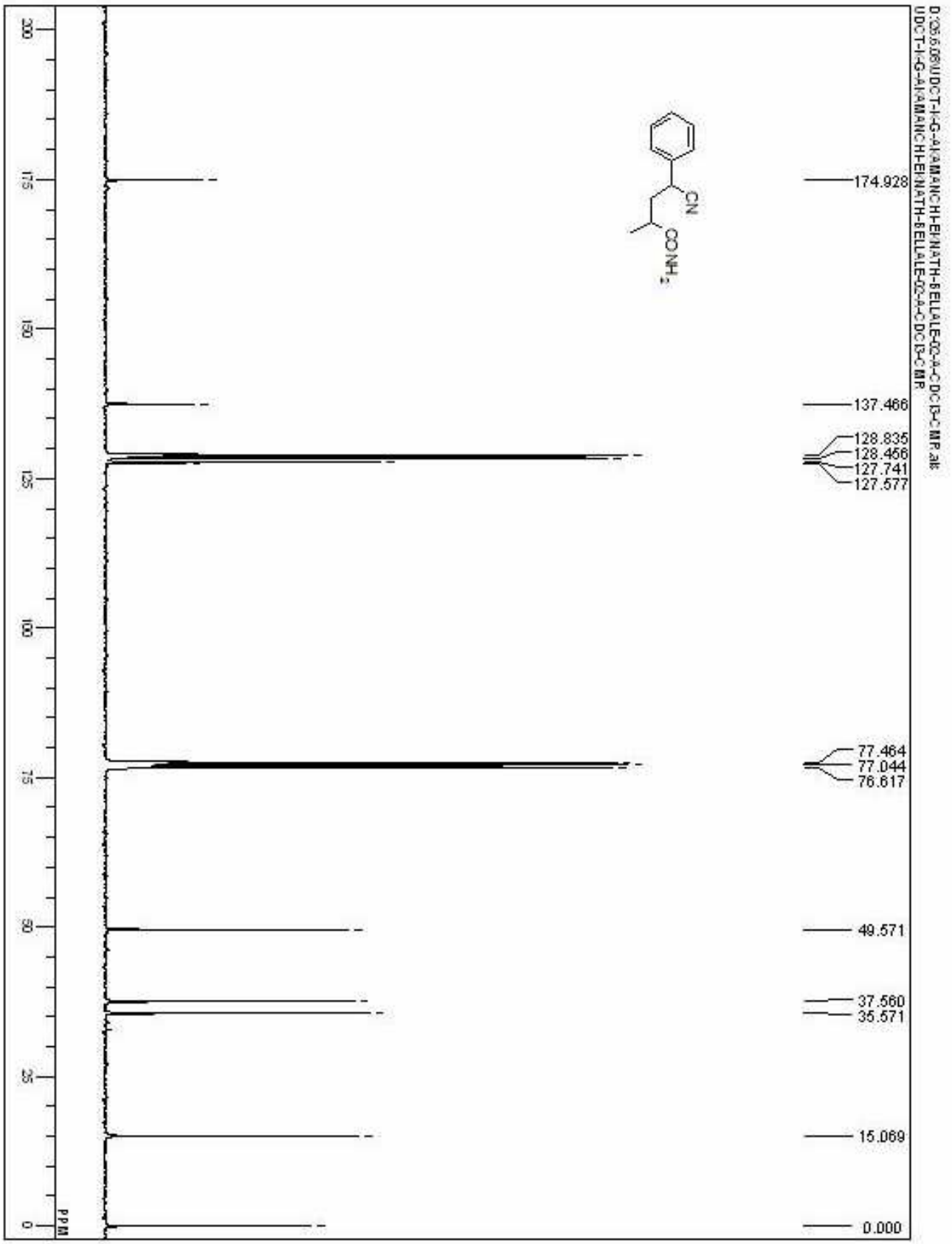
^{1}H NMR of 2-(4-Chlorophenyl)hexanamide **1b**



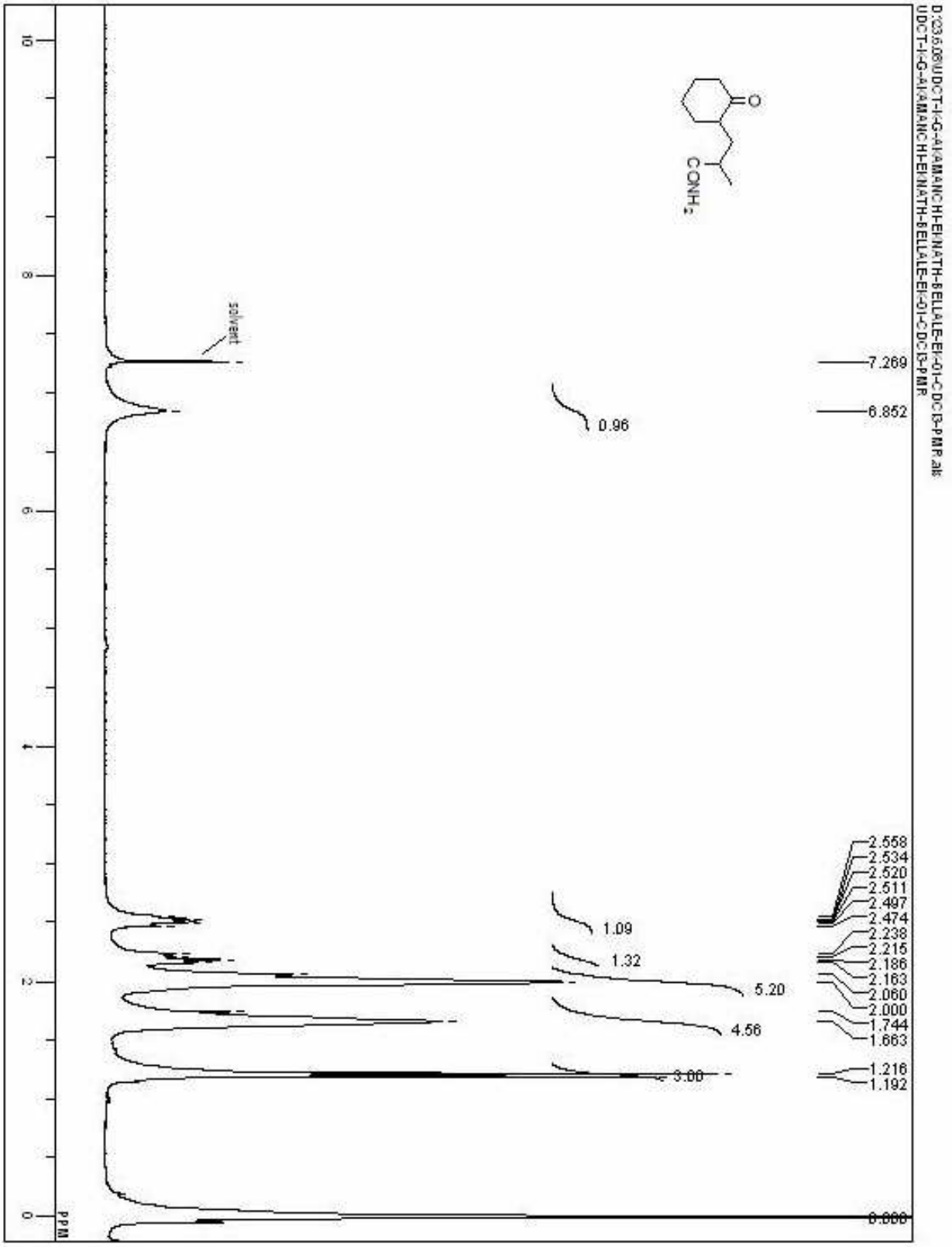
^{13}C NMR of 2-(4-Chlorophenyl)hexanamide **1b**



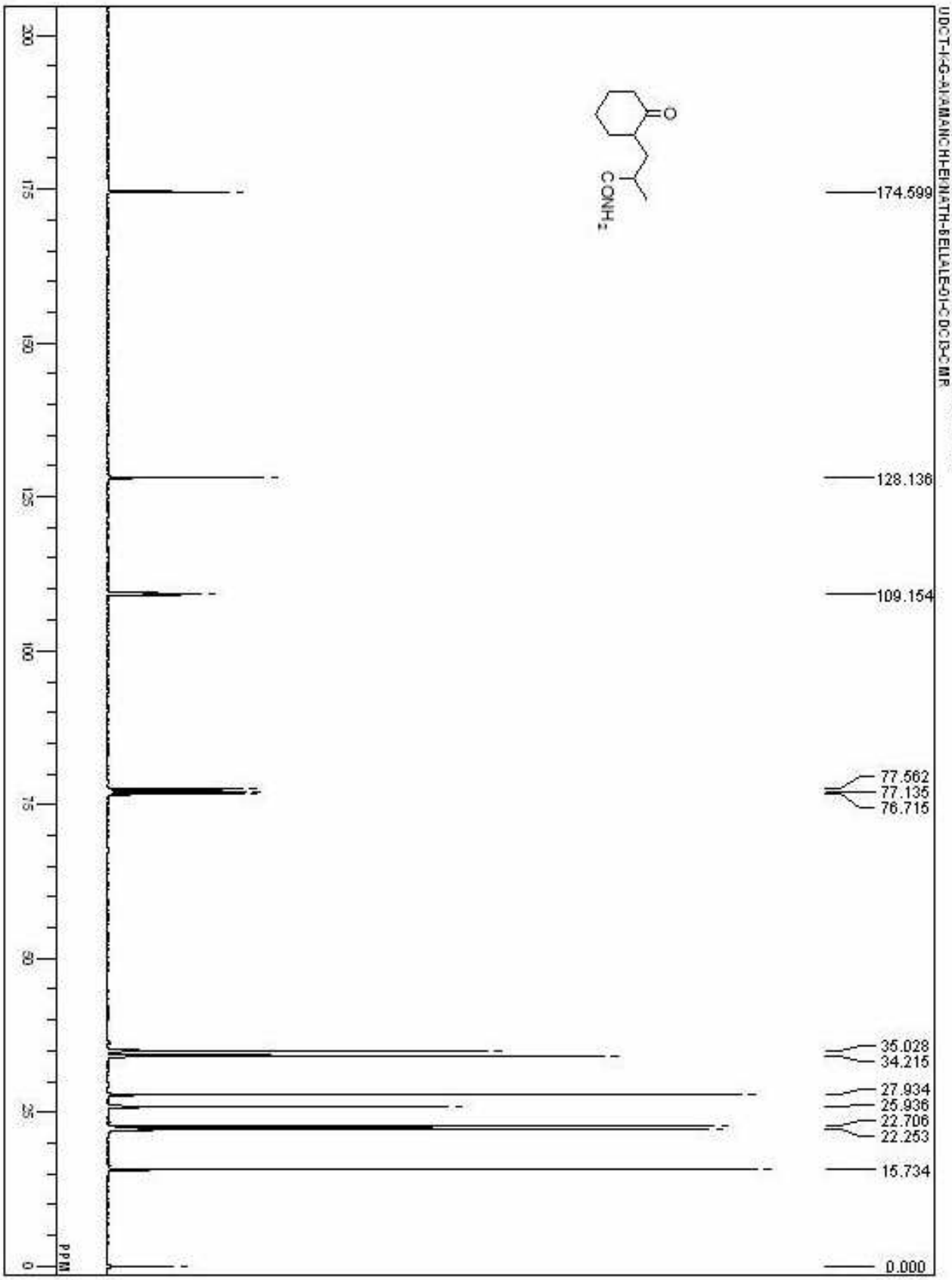
^{1}H NMR of 4-Cyano-2-methyl-4-phenylbutanamide **1g**



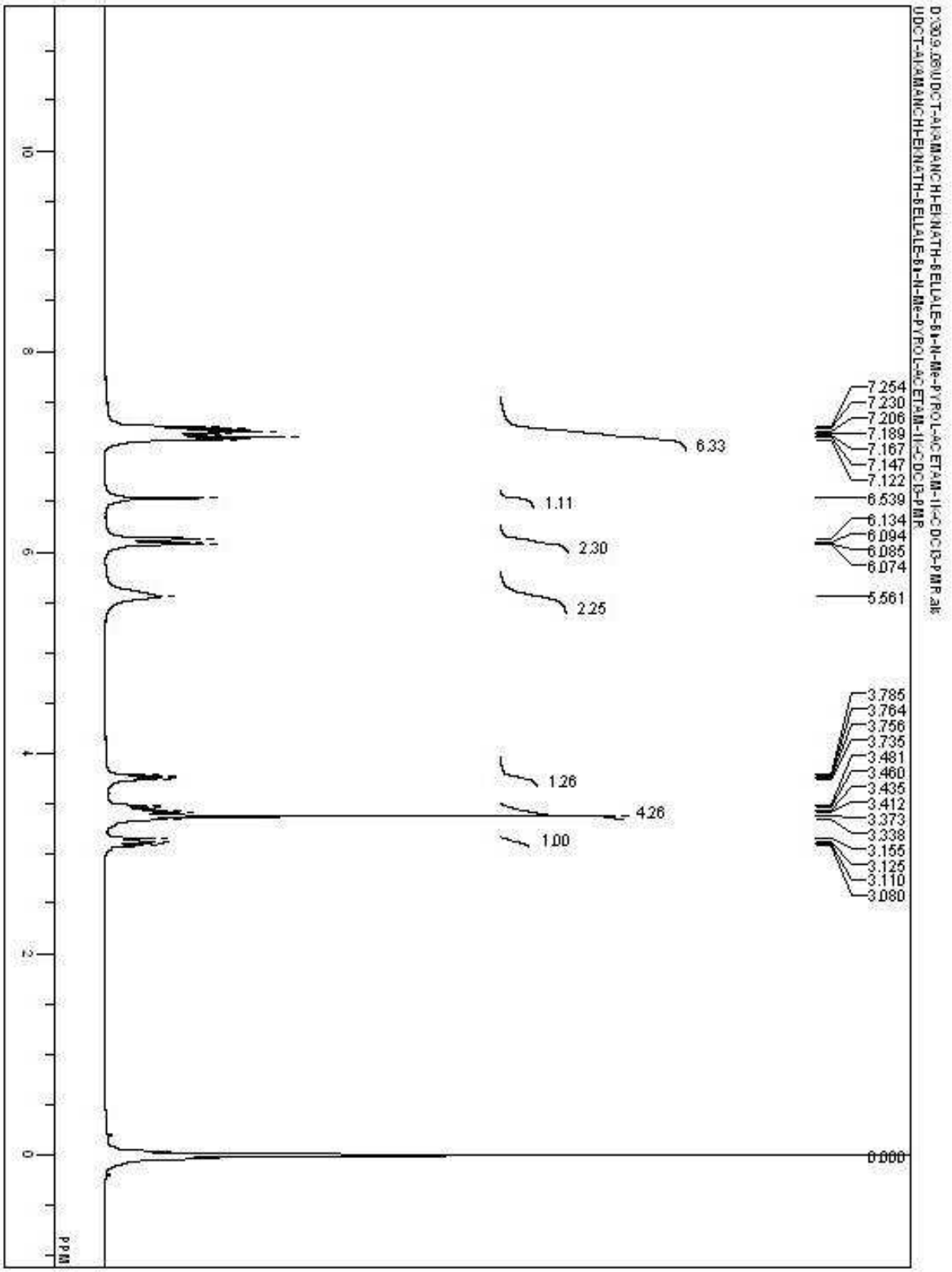
^{13}C NMR of 4-Cyano-2-methyl-4-phenylbutanamide **1g**



^{1}H NMR of 2-Methyl-3-(2-oxo-cyclohexyl)propanamide **1h**

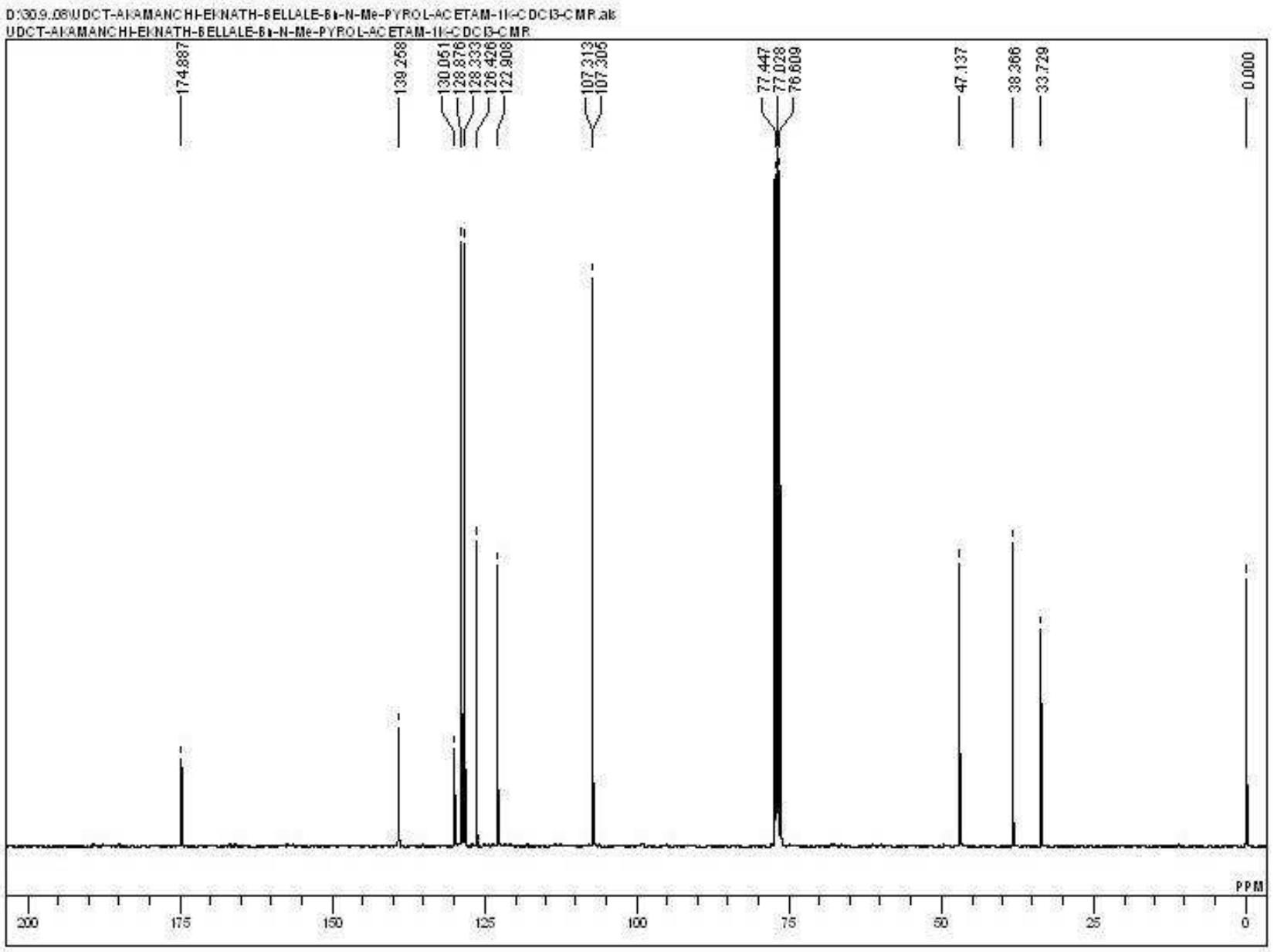


^{13}C NMR of 2-Methyl-3-(2-oxo-cyclohexyl)propanamide **1h**

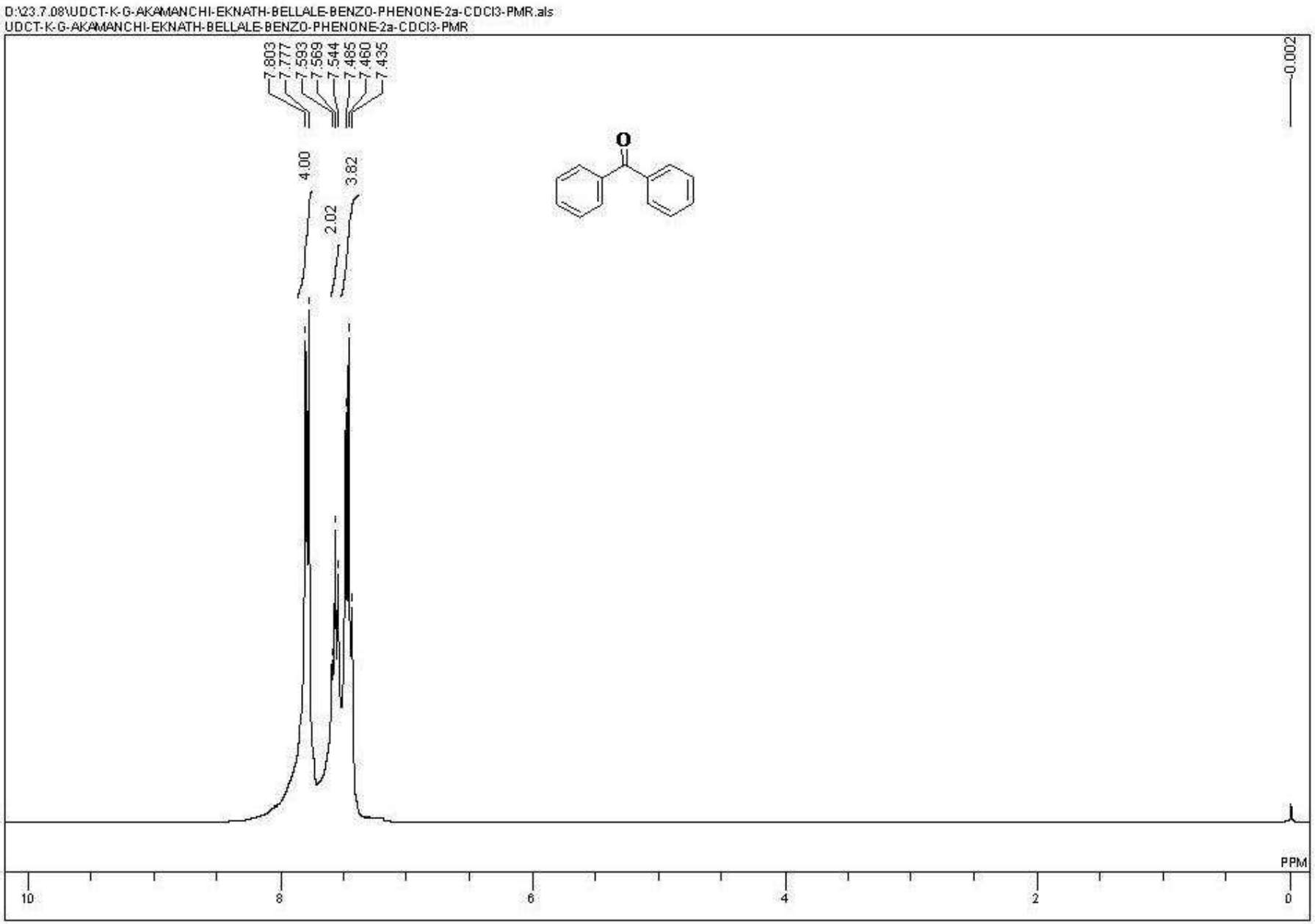


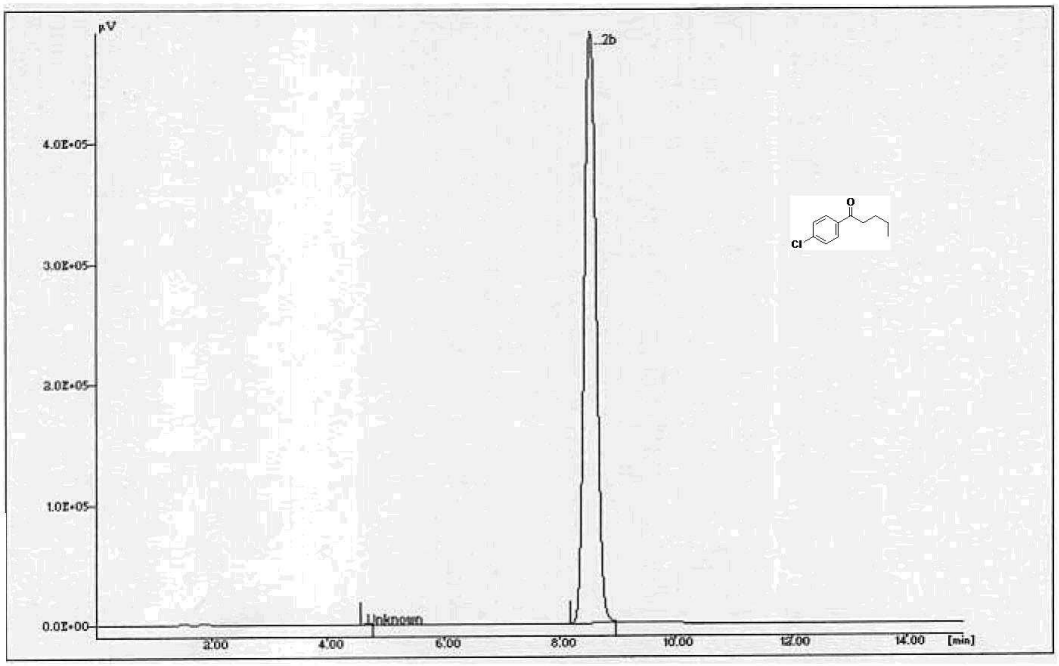
^{1}H NMR of 2-(1-methyl-1H-pyrrol-2-yl)-3-phenylpropanamide **1k**

^{13}C NMR of NMR of 2-(1-methyl-1H-pyrrol-2-yl)-3-phenylpropanamide **1k**



^{1}H NMR of Benzophenone **2a**





File name : **1-(4-Chlorophenyl)pentan-1-one 2b**

Info :

SYSTEM : ACN:Water (3:1) flow : 1 ml/min.

WAVELENGTH : 254

Injection Date :29-Jul-2008 4:44:14

Curr. Date : 29-Jul-2008 9:01:28

User : EKNATH

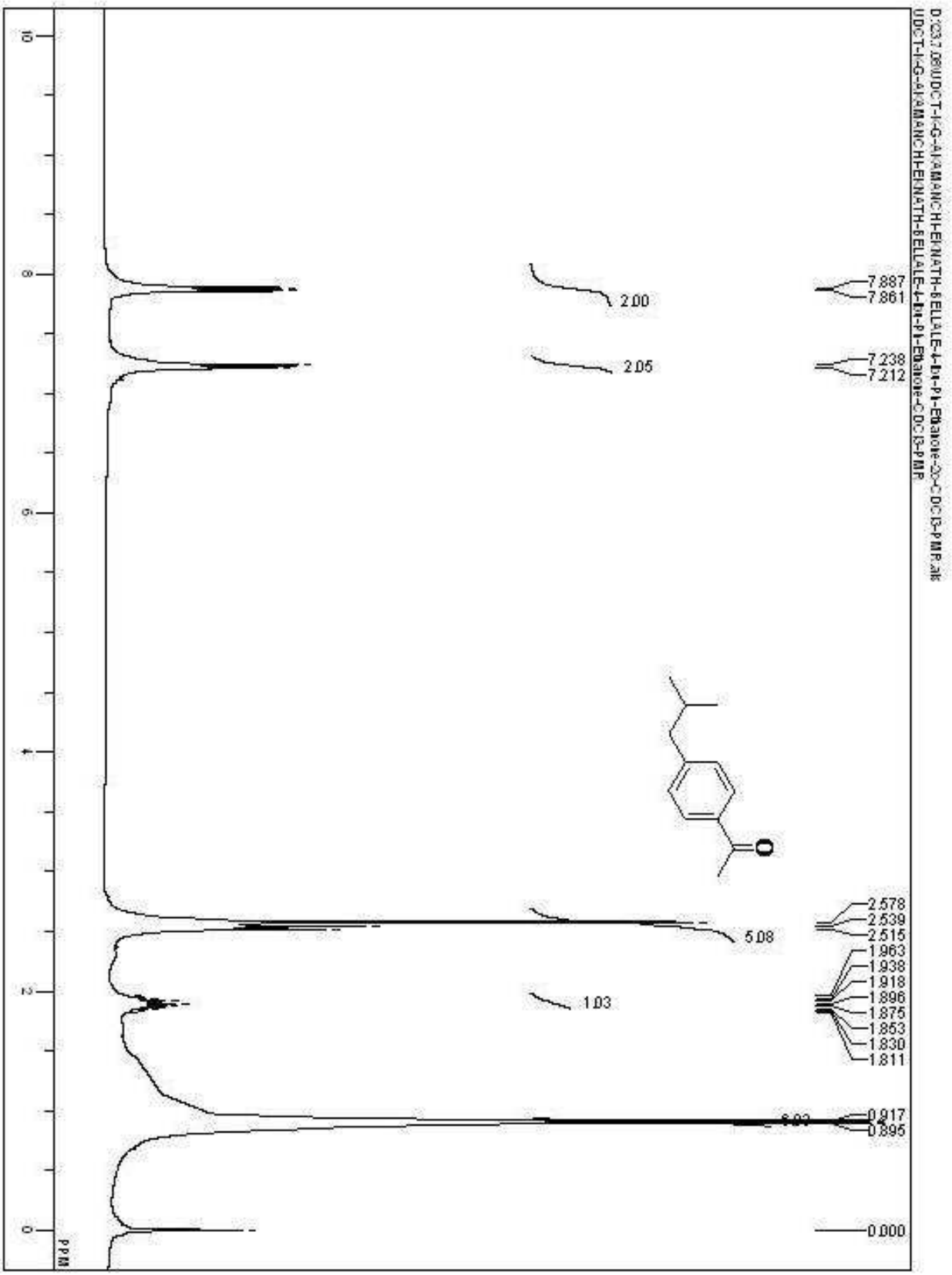
Group : KGA

Control Method :

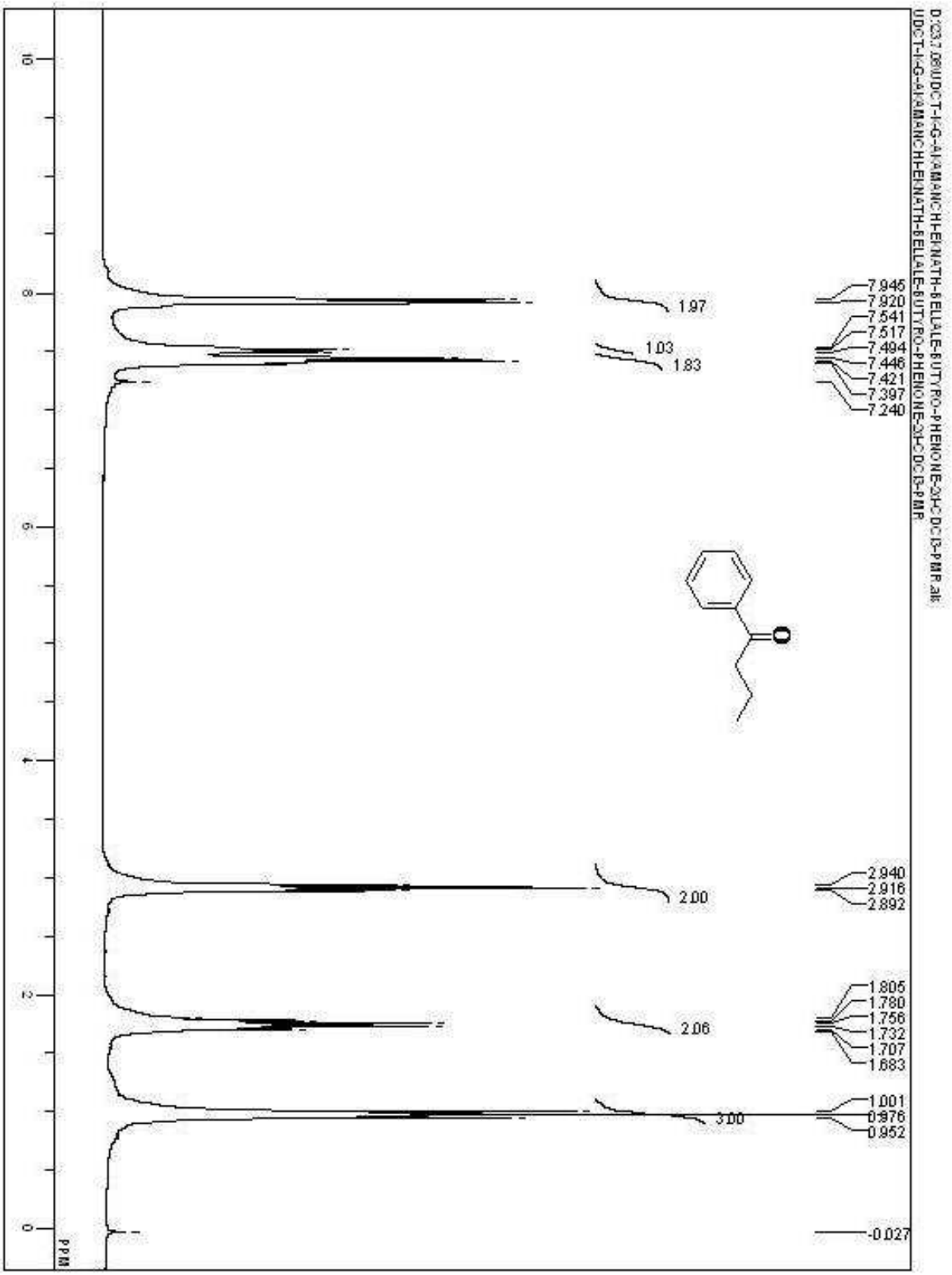
Sr.No.	Name	RT	Area(μV.Sec)	%Area
1		4.662	3821.250	0.062
2	2b	8.515	6184749.410	99.938

Total Area of Peak = 6188570.660 [μV.Sec]

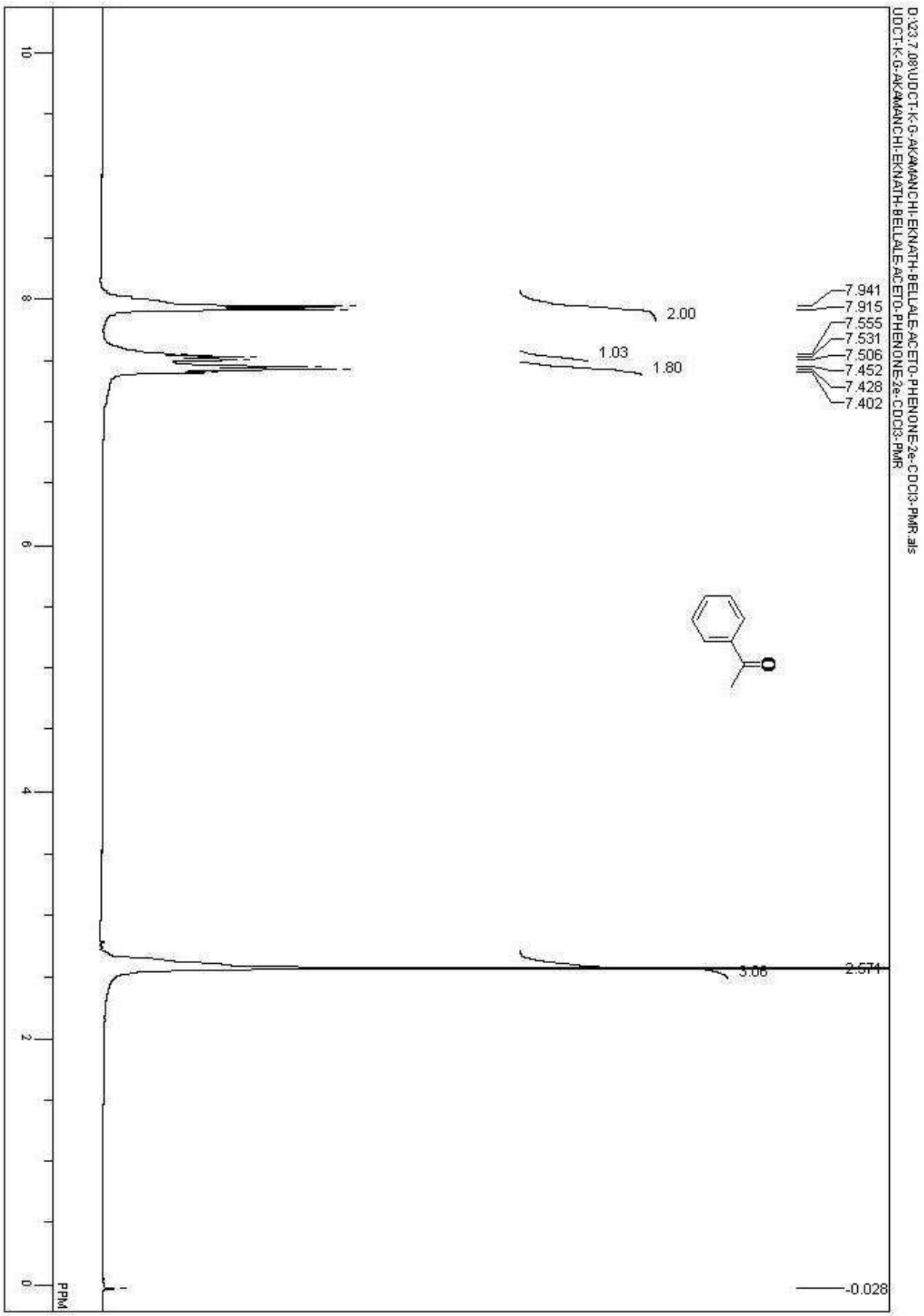
HPLC Chromatogram of 1-(4-Chlorophenyl)pentan-1-one **2b**



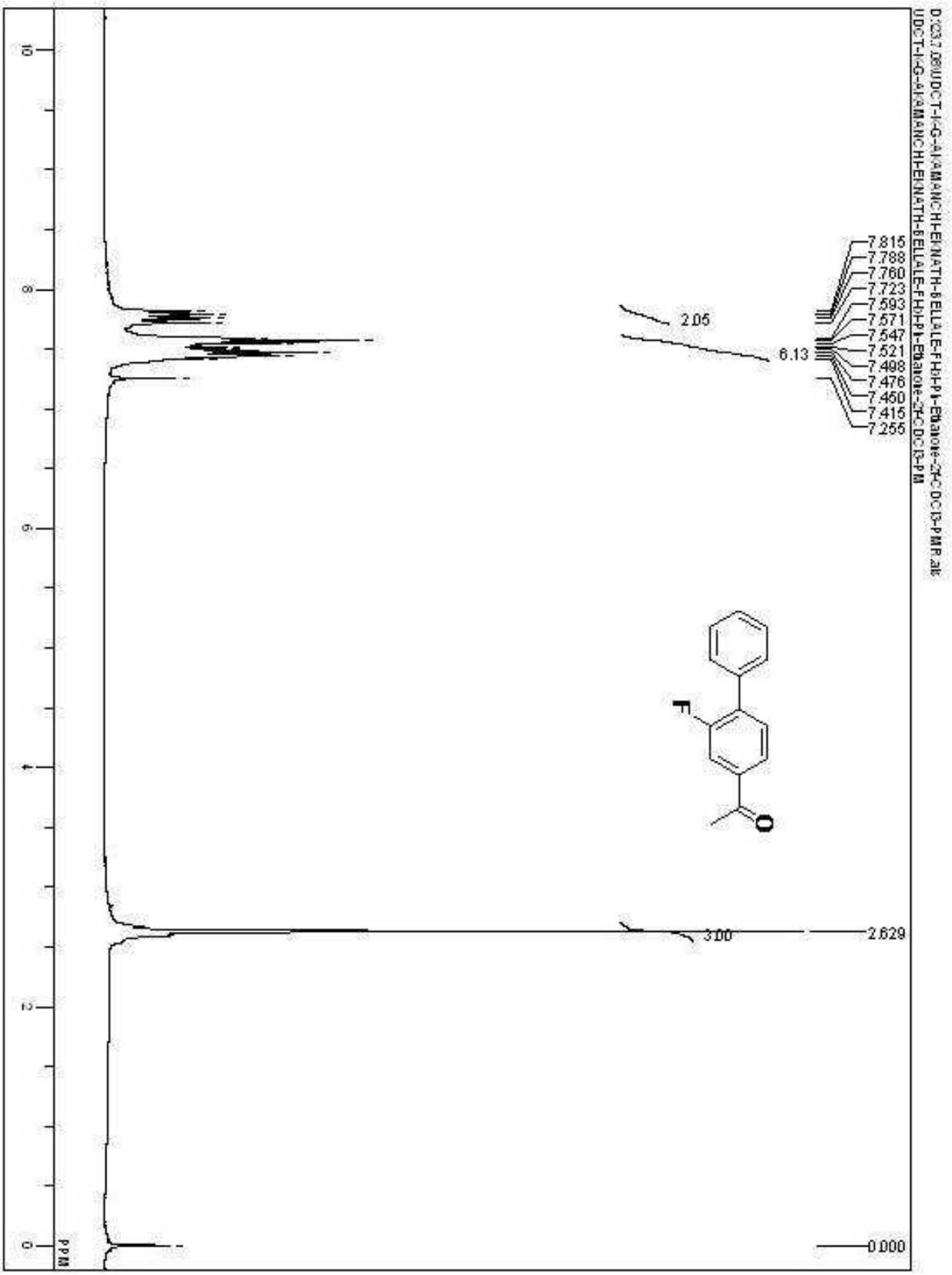
^{1}H NMR of 1-(4-Isobutylphenyl)ethanone **2c**



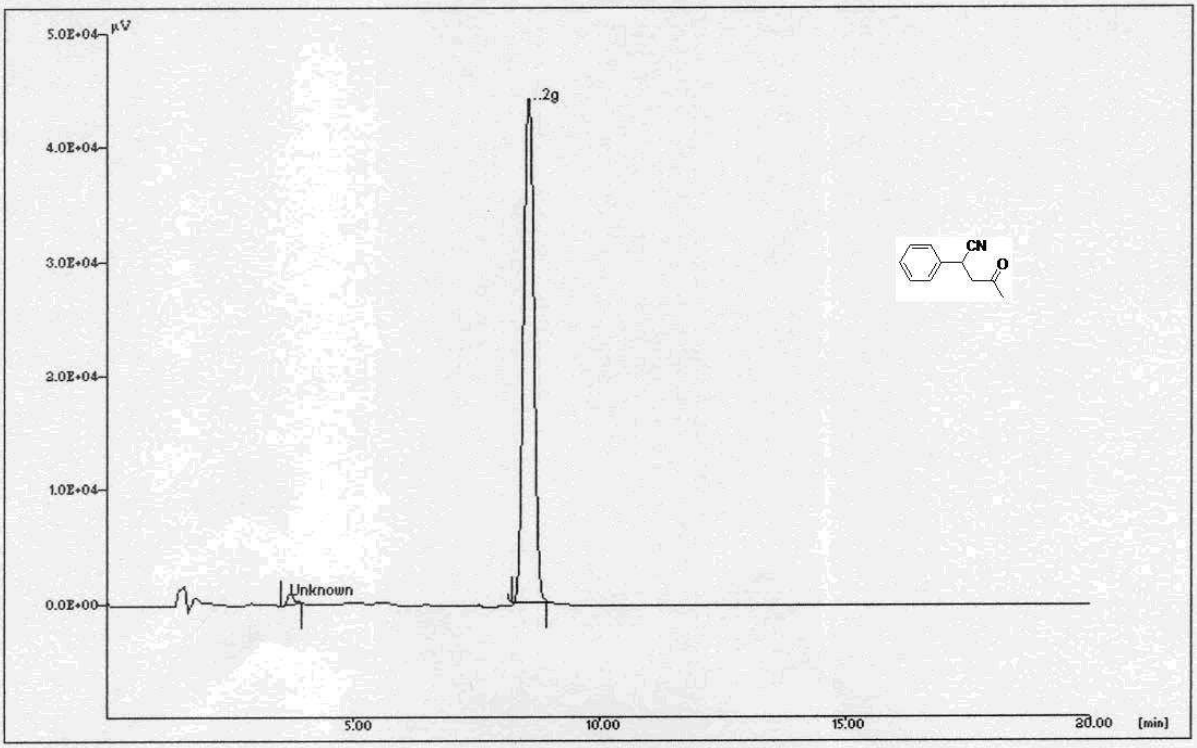
^{1}H NMR of Butyrophenone **2d**



^{1}H NMR of Acetophenone **2e**



^{1}H NMR of 1-(2-Fluorobiphenyl-4-yl)ethanone **2f**



File name : **4-Oxo-2-phenylpentanenitrile 2g**

Info :

SYSTEM : ACN:Water (3:1) flow : 1 ml/min.

WAVELENGTH : 254

Injection Date :29-Jul-2008 6:04:36

Curr. Date : 29-Jul-2008 9:11:14

User : EKNATH

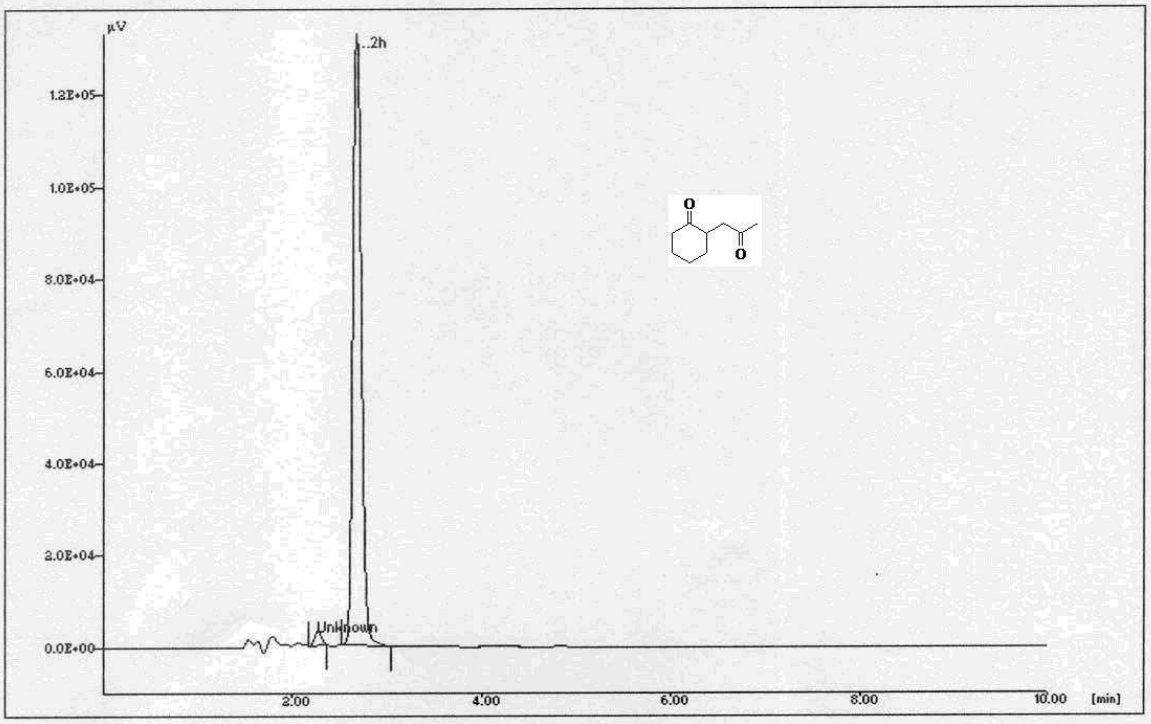
Group : KGA

Control Method :

Sr.No.	Name	RT	Area(μV.Sec)	%Area
1		3.713	7033.975	1.259
2	2g	8.535	551792.581	98.741

Total Area of Peak = 558826.556 [μV.Sec]

HPLC Chromatogram of 4-Oxo-2-phenylpentanenitrile **2g**



File name : **2-(2-oxopropyl) cyclohexanone 2h**

Info :

SYSTEM : ACN:Water (3:1) flow : 1 ml/min.

WAVELENGTH : 280

Injection Date :29-Jul-2008 8:19:08

Curr. Date : 29-Jul-2008 9:04:28

User : EKNATH

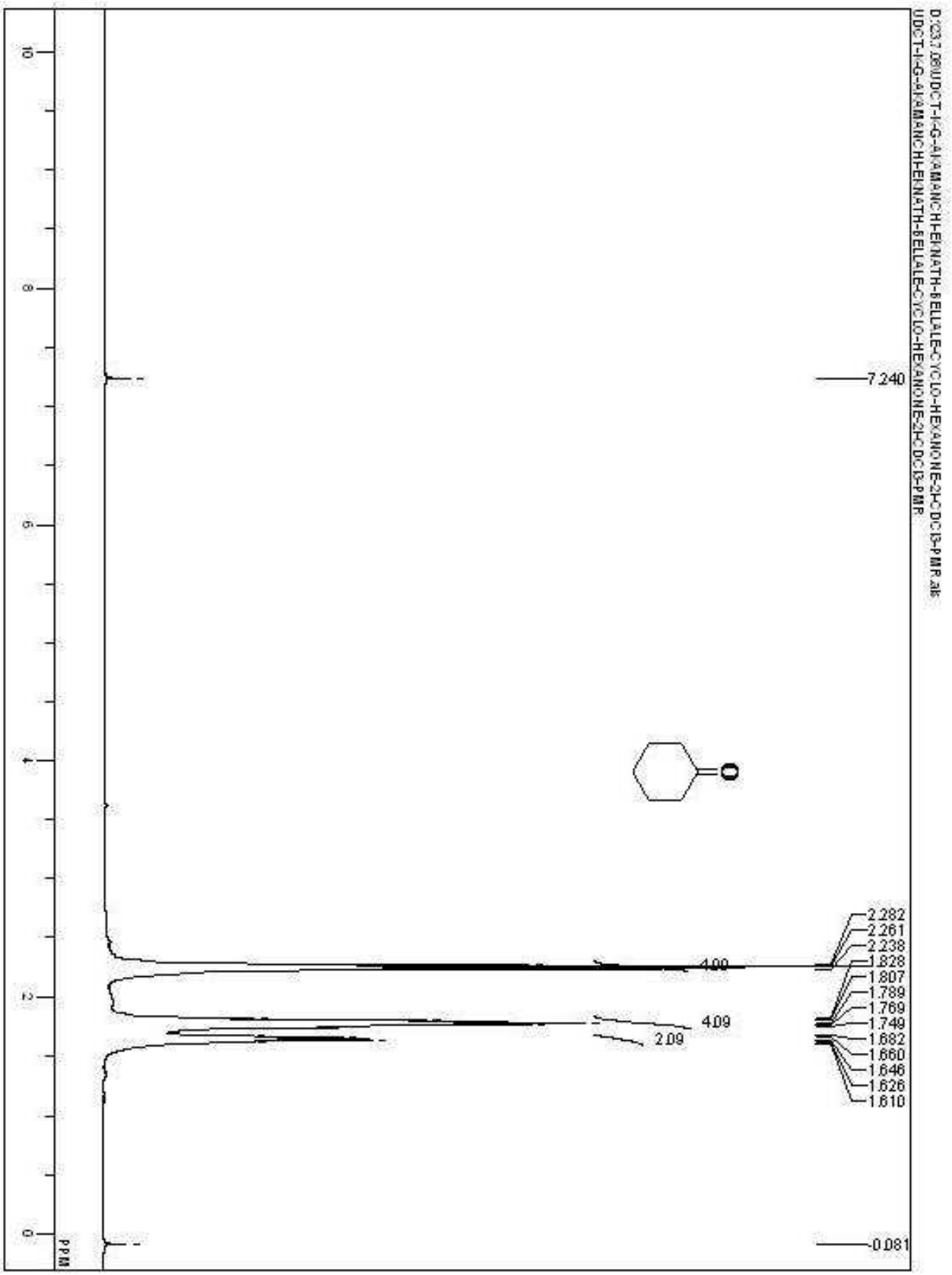
Group : KGA

Control Method :

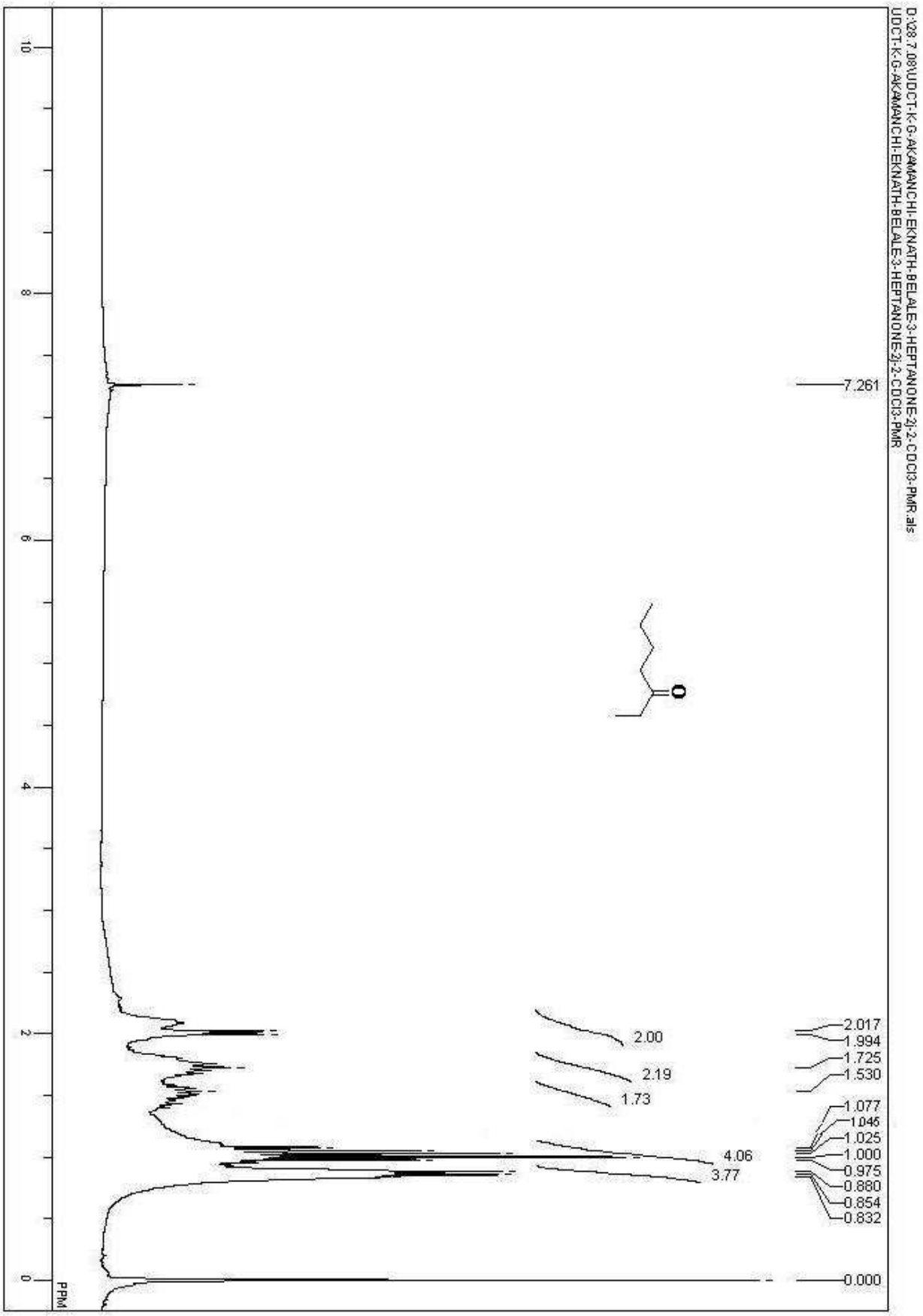
Sr.No.	Name	RT	Area(µV.Sec)	%Area
1		2.260	13011.187	1.832
2	2h	2.668	697306.113	98.168

Total Area of Peak = 710317.300 [µV.Sec]

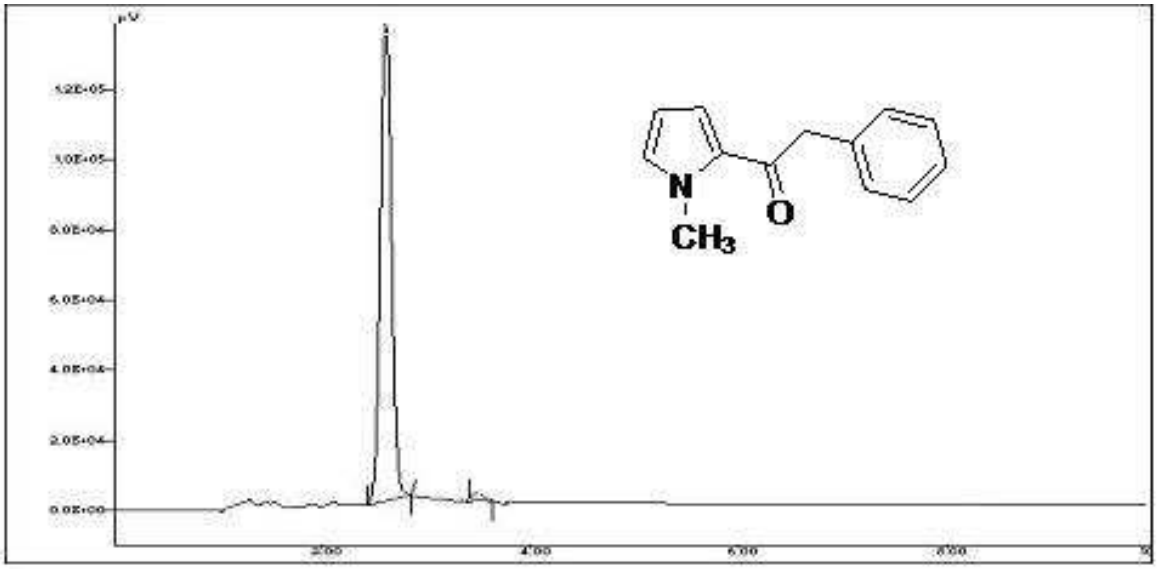
HPLC Chromatogram of 2-(2-Oxopropyl)cyclohexanone **2h**



^{1}H NMR of Cyclohexanone **2i**



^{1}H NMR of 3-Heptanone **2j**



File name : **1-(1-methyl-1H-pyrrol-2-yl)-2-phenylethanone 2k**

Info :

SYSTEM : ACN:Water (3:1) flow : 1 ml/min.

WAVELENGTH : 254

Injection Date :30-SEP-2008 5:30:36

Curr. Date : 30-SEP-2008 10:11:14

User : EKNATH

Group : KGA

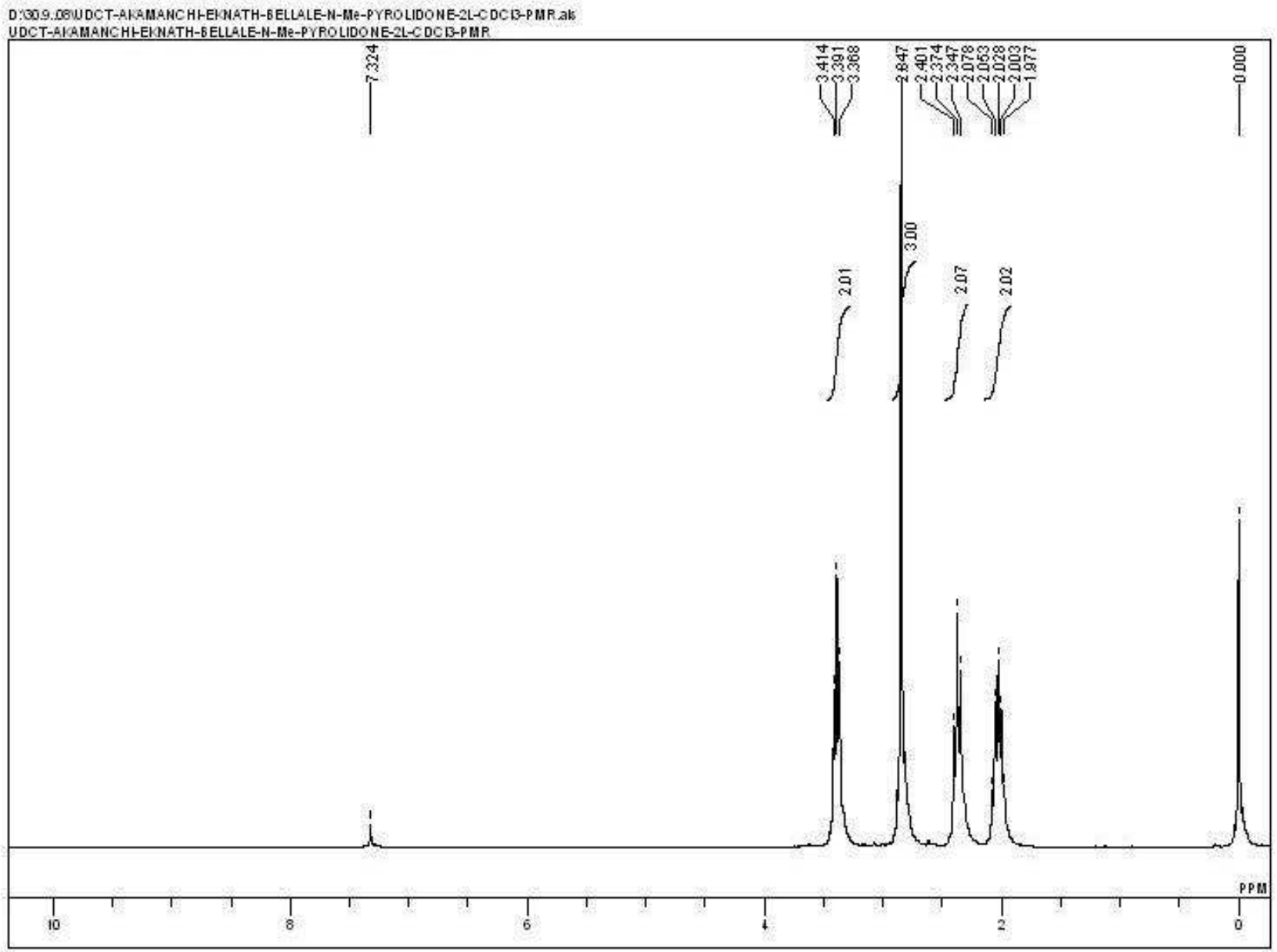
Control Method :

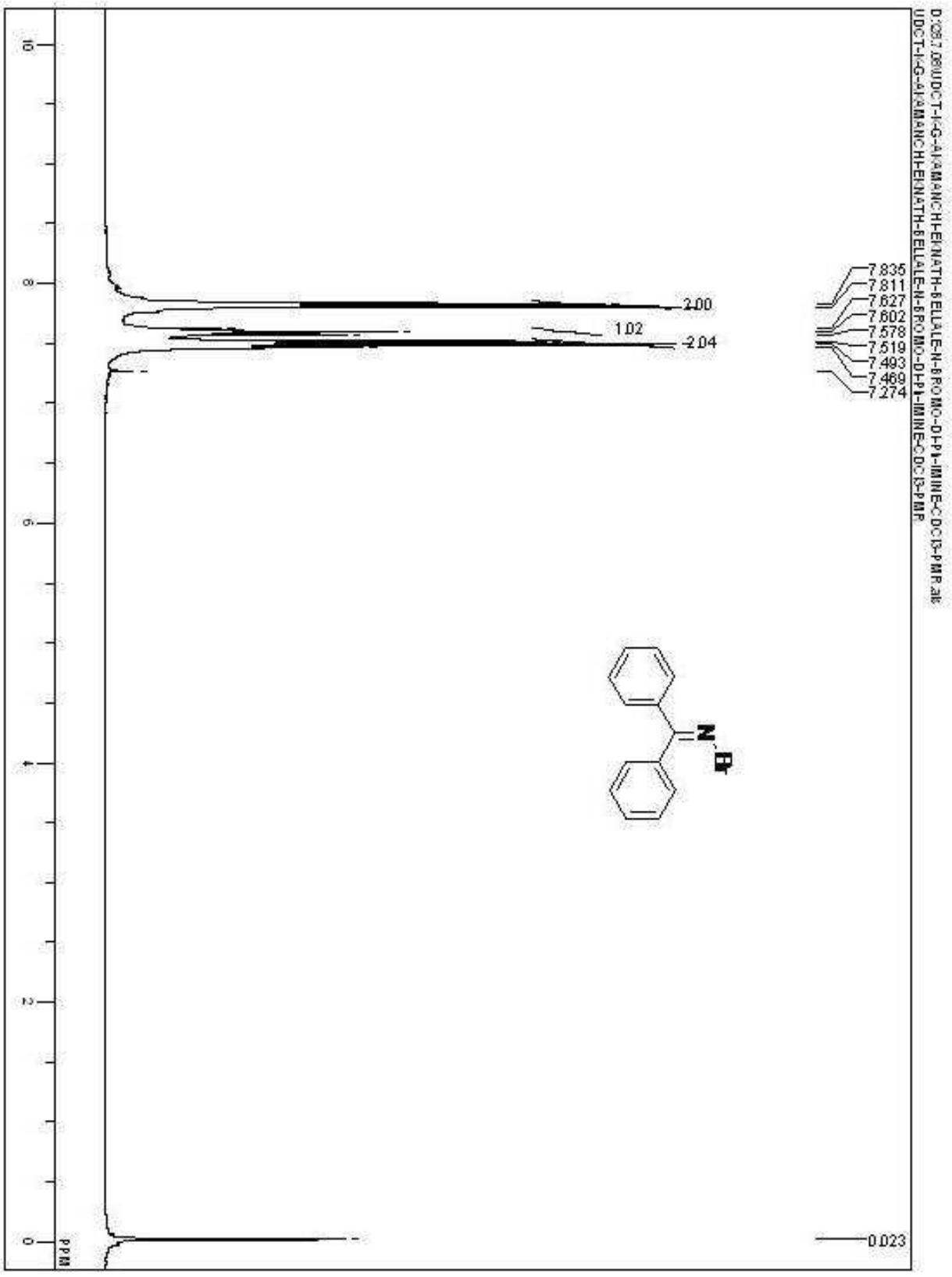
Sr.No.	Name	RT	Area(μV.Sec)	%Area
1	2k	2.602	880642.200	98.556
2		3.480	12904.700	1.444

Total Area of Peak = 893546.900 [μV.Sec]

HPLC Chromatogram of 1-(1-methyl-1H-pyrrol-2-yl)-2-phenylethanone **2k**

^{1}H NMR of N-Methyl Pyrrolidone **2l**





^{1}H NMR of N-Bromo-1,1-diphenylmethanimine

^{1}H NMR of Cyclohexanone from Reaction of Isocynate with IBX/TEAB