

Electronic Supporting Information

Acylation of Grignard reagents mediated by N-methylpyrrolidone: A remarkable selectivity for the synthesis of ketones

Maravanhalli Sidde Gowda[†] Sushanth Sudhir Pande,[§] Ramesha Andagar Ramakrishna,^{*, †} and
Kandikere Ramaiah Prabhu^{*, §}

[†]R. L. Fine Chem, No. 15, KHB Industrial Area, Yelahanka Newtown, Bangalore-560 106, Karnataka,
India

[§]Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012, Karnataka, India

E-mails: prabhu@orgchem.iisc.ernet.in; ramesha63@hotmail.com

Table of Contents

Table of Contents	Page No
General	ESI 2
Experimental Section	ESI 2 – ESI 3
Analytical Data	ESI 4-ESI 17
References	ESI 18-ESI 19
¹ H and ¹³ C spectra	ESI 20- ESI 45

General. All solvents were dried and distilled according to standard methods before use. NMR spectra were recorded on a JEOL LA-300, BRUKER-AV400 spectrometer in CDCl_3 and DMSO-d_6 . Tetramethylsilane (TMS; $\delta = 0.00$ ppm) and residual non-deuterated DMSO signal ($\delta = 2.49$ ppm) served as internal standards for ^1H NMR. The corresponding residual non-deuterated solvent signal (CDCl_3 : $\delta = 77.00$ ppm; DMSO : $\delta = 39.50$ ppm) was used as internal standards for ^{13}C NMR. IR spectra were measured using a JASCO FT/IR-410 spectrometer, and Perkin-Elmer FT/IR Spectrum BX, GX. Mass spectra were measured with Micromass Q-ToF (ESI-HRMS), and GCMS shimadzu. Column chromatography was conducted on Silica gel 230-400 mesh (Merck) and preparative thin-layer chromatography was carried out using SILICA GEL GF-254. Elemental analysis was carried out at the Department of Organic Chemistry, Indian Institute of Science, Bangalore, India by using Thermo Finnigan Flash 1112 series analyser.

Experimental Section

Typical procedure adapted to preparation of Grignard Reagents. To a well-stirred dry mixture of Mg (23.1 mmol) in dry THF (10 mL) in nitrogen atmosphere was added iodine (20 mg) and 1,2-dibromoethane (100 mg), and the reaction mixture was heated to $55 - 60^\circ$. To this reaction mixture was added aryl or alkyl bromide (23.1 mmol) in toluene (10 mL) at the same temperature and the reaction mixture was stirred at 60°C for 1h and brought to room temperature and used for the next step.

Acylation of Grignard reagent (general procedure). To a well-stirred solution of NMP (2.51 g, 2.45 mL, 25.4 mmol) in dry toluene (10 mL) was added acid chloride (27.7 mmol) in toluene (10 mL) at 0°C during 15 min, and stirred for 15 min at the same temperature, followed by the addition of RMgX (23.1 mmol, in 20 mL THF) at -5 to -10°C during 15 min. The reaction mixture was stirred for 4h at 0 to -5°C , and was added to aqueous ammonium chloride solution (10g solution in 30 mL water), and extracted with CH_2Cl_2 (3×50 mL), the combined organic layer was washed with dil HCl (20%, 100mL), saturated

Na₂CO₃ solution (100mL), and water (2 × 50 mL), dried over Na₂SO₄ and concentrated in *vacuo*. The resulting crude mixture was purified by column chromatography to furnish the pure compound.

General procedure 2 (For compound 14c and 15c Table 2).

Step 1: To a well-stirred mixture of Mg (878 mg, 36 mmol) and I₂ (100 mg) in THF (25 mL) was added isobutyl chloride (3.22g, 3.65 g, 35 mmol) at room temperature during 20 min, heated at reflux for 1h, cooled to 0 ° in N₂ atmosphere and was added 2-bromopyridine (5g, 3.01 mL, 31.8 mmol,) in toluene (6 mL) at 0 °C and stirred at the same temperature for 30 min to generate the Grignard reagent.

Step 2: To a well-stirred cold solution (0 °C) of NMP (3.4mL, 3.47 g, 35 mmol) in dry toluene (10 mL) was added a solution of acid chloride (38.2 mmol, 1.2 equiv) in dry toluene (10 mL) during 15 min. After stirring the reaction mixture for 15 min at the same temperature was added a solution of RMgX (31.8 mmol) in THF (generated in step 1) at -5 to -10 °C during 15 min and the reaction mixture was stirred for 4 h at the same temperature and was quenched with aqueous ammonium chloride (10 g solution in 30 mL water), extracted with CH₂Cl₂ (3 × 50 mL), combined organic layer was separated and dried over sodium sulphate and the solvent was removed completely. The residue was purified by column chromatography on silica gel to obtain the product.

Analytical Data.

Hexanophenone (1c).¹ ¹H NMR (CDCl₃, 400 MHz); Colourless liquid; IR (Neat, cm⁻¹) = 1684; 7.94-7.96 (m, 2H), 7.51-7.55 (m, 1H), 7.42-7.46 (m, 2H), 2.95 (t, 2H, *J* = 7.4 Hz), 1.73 (t, 2H, *J* = 7.4 Hz), 1.25-1.41(m, 4H), δ 0.90 (t, 3H, *J* = 6.8 Hz); ¹³C NMR (CDCl₃, 75 MHz): δ 200.4, 136.9, 132.7, 128.4, 127.9, 38.4, 31.4, 23.9, 22.4, 13.8; HRMS(ESI) calculated for C₁₂H₁₆O: 177.1279, found 177.1278 (M+H).

4-chloro-1-(4-fluorophenyl)butan-1-one (2c).² Prepared from *p*-fluorophenylmagnesium bromide and 4-chlorobutryl chloride as described in general procedure 1. Colorless liquid; IR (Neat, cm⁻¹): 2964, 1687, ¹H NMR (CDCl₃, 400 MHz); δ 7.90 - 8.00 (m, 2H), 7.0 - 7.10 (m, 2H), 3.68 (t, 2H, *J* = 6.2 Hz), 3.16 (t, 2H, *J* = 6.9 Hz), 2.22 (quintet, 2H, *J* = 6.4 Hz); ¹³C NMR (CDCl₃, 75 MHz): δ 197.2, 166.9, 164.4, 133.0, 130.5, 130.4, 115.7, 115.5, 35.0, 31.0, 26.5; MS (EI) *m/z* 201(M⁺)

4-Chloro-1-(4-chlorophenyl)butan-1-one (3c).³ Prepared from *p*-chlorophenyl magnesium bromide and 4-chlorobutryl chloride as described in general procedure 1. *mp*: 31 °C (29-30 °C)^{lit, 3a}, Yield: 70%; colourless solid; IR (Neat, cm⁻¹) : 2961, 1686, ¹H NMR (CDCl₃, 400 MHz); δ 7.90 - 7.94 (m, 2H), 7.43 - 7.46 (m, 2H), 3.68 (t, 2H, *J* = 6.4 Hz), 3.16 (t, 2H, *J* = 7.2 Hz), 2.23 (p, 2H, *J* = 6.8 Hz); ¹³C NMR (CDCl₃, 75 MHz): δ 197.7, 139.6, 135.0, 129.4, 128.9, 44.5, 35.2, 26.6; MS (EI) *m/z* 217 (M⁺).

Benzophenone (4c).⁴ Prepared from benzoyl chloride and phenylmagnesium bromide as described in general procedure 1. *mp*: 48 °C(47.5 °C)^{lit, 4a}, Yield: 85%; Colourless solid; IR (KBr, cm⁻¹): 1659, ¹H NMR (CDCl₃, 300 MHz): δ 7.42-7.81(m, 10H), ¹³C NMR (CDCl₃, 75 MHz): δ 196.5, 137.5, 132.3, 129.9,128.1; MS (EI) *m/z* 182 (M⁺); HRESI-MS (*m/z*): calculated for C₁₃H₁₀O: 183.0810, found 183.0812 (M+H).

4-Fluoro benzophenone (5c).⁵ Prepared from benzoyl chloride and fluoro-*p*-phenylmagnesium bromide as described in general procedure 1. *mp*: 46-47 °C (45-47 °C)^{lit, 5a}, Yield: 77%; pale yellow solid; IR (KBr, cm⁻¹):1723, 1660, ¹H NMR (CDCl₃, 400 MHz): δ 7.10 - 7.80 (m, 9H), ¹³C NMR (CDCl₃, 100

MHz): δ 195.2, 164.4(d, $J = 252$ Hz), 137.4, 133.7(d, $J = 3$ Hz), 132.6 (d, $J = 9$ Hz), 132.4, 129.8, 128.3, 115.4 (d, $J = 22$ Hz), MS (EI) m/z 200(M^+).

4-Methoxybenzophenone (6c).⁶ Prepared from *p*-methoxybenzoyl chloride and phenylmagnesium bromide as described in general procedure 1. *mp*: 58-60 °C (59-60 °C)^{lit, 6a}; Yield: 76%; Colourless solid; IR (Neat, cm^{-1}): 3059, 1651, ¹H NMR (CDCl₃, 300 MHz): δ , 6.9 - 7.85 (m, 9H), 3.89 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 195.5, 163.2, 138.2, 132.5, 131.8, 130, 129.6, 128.1, 113.5, 55.5, MS (EI) m/z 212(M^+); HRESI-MS (m/z): calculated for C₁₄H₁₂O₂: 213.0916, found 213.0915 (M+H).

2,4-Dichlorobenzophenone (7c).⁷ Prepared from 2,4-dichlorobenzoyl chloride and phenylmagnesium bromide as described in general procedure 1. *mp*: 51-52 °C (50.5-51.5 °C)^{lit, 10a}; Yield: 78%; Colourless solid; IR (KBr, cm^{-1}): 1680, 1673, ¹H NMR (CDCl₃, 400 MHz): δ 7.20 - 7.90 (m, 8H), ¹³C NMR (CDCl₃, 100 MHz): δ 194.1, 136.8, 136.5, 136.1, 133.8, 132.3, 130.0, 129.9, 128.6, 127; MS (EI) m/z 250(M^+); HRESI-MS (m/z): calculated for C₁₃H₈Cl₂O(M+Na) : 272.9850, found 272.9852 (M+Na).

Acetophenone (8c).⁸ Prepared from acetyl chloride and phenyl magnesium bromide as described in general procedure 1. Yield: 72%; Colorless liquid; IR (Neat cm^{-1}): 1684, ¹H NMR (CDCl₃, 400 MHz): δ 7.43-7.96 (m, 5H), 2.60 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 198.1, 137, 133, 128.5, 128.2, 26.5; MS (EI) m/z 120(M^+).

1-(4-chlorophenyl)ethanone (9c).⁹ Prepared from *p*-chlorophenyl magnesium bromide and acetyl chloride as described in general procedure 1. Yield: 83%; colorless liquid; IR (Neat, cm^{-1}): 1742, 1687, ¹H NMR (CDCl₃, 400 MHz): δ 7.87 - 7.89 (d, $J = 8.2$ Hz, 2H), 7.41 - 7.43 (d, $J = 8.2$ Hz, 2H), 2.58 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 196.7, 139.4, 135.3, 129.6, 128.7, 26.4; MS (EI) m/z 154(M^+).

1-(4-(*tert*-butyl)phenyl)-4-chlorobutan-1-one (10c).¹⁰ Prepared from *p-tert*-butylphenyl magnesium bromide and 4-chloro butryl chloride as described in general procedure 1. and *o*-benzoylbenzoyl chloride as described in general procedure 1. *mp*.: 47 °C (46-49 °C)^{lit, 10a}, Yield: 83%; pale yellow solid; IR (KBr, cm^{-1}): 1684. ¹H NMR (CDCl₃, 400 MHz): δ 7.92 (d, 2H, $J = 8.4$ Hz), 7.48 (d, 2H, $J = 8.4$ Hz), 3.67 (t, 2H,

$J = 6.2$ Hz), 3.15 (t, 2H, $J = 6.2$ Hz), 2.21 (quint, 2H, $J = 6.6$ Hz), 1.30 (s, 9H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 198.5, 156.9, 134.1, 127.9, 125.5, 44.6, 35.1, 35.0, 31.2, 26.8; MS (EI) m/z 239 ($\text{M}^+ + 1$).

2-Benzoylbenzophenone (11c).¹¹ Prepared from phenyl magnesium bromide and *o*-benzoylbenzoyl chloride as described in general procedure 1. *mp.*: 139-140 °C (137-139 °C)^{lit.},¹¹ Yield: 83%; pale yellow solid; IR (KBr, cm^{-1}): 1672, 1661. ^1H NMR (CDCl_3 , 400 MHz): δ 7.88 (d, $J = 7.4$ Hz, 4H), 7.79 (s, 4H), 7.69 (t, $J = 7.1$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 4H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 196.5, 139.9, 137.1, 132.9, 130.3, 129.7, 129.6, 128.2; MS (EI) m/z 286 (M^+); HRESI-MS (m/z): calculated for $\text{C}_{20}\text{H}_{14}\text{O}_2$: 309.0891 ($\text{M} + \text{Na}$), found 309.0890 ($\text{M} + \text{Na}$).

Benzoylacetonitrile (12c).¹² Prepared from phenyl magnesium bromide and cyano acetyl chloride as described in general procedure 1. *mp.*: 81-83 °C (80-81 °C)^{lit.},¹² Yield: 73%; colourless crystalline solid; IR (KBr, cm^{-1}): 2263, 1689; ^1H NMR (CDCl_3 , 400 MHz): δ 7.91-7.93 (m, 2H), 7.65-7.69 (m, 1H), 7.51 - 7.55 (m, 2H), 4.10 (s, 2H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 187, 134.7, 134.2, 129.1, 128.4, 113.7, 29.3; HRESI-MS (m/z): calculated for $\text{C}_9\text{H}_7\text{NO}$ ($\text{M} + \text{Na}$): 168.0425, found 168.0427 ($\text{M} + \text{Na}$).

Compound 13c.¹³ Prepared from *p*-chlorophenyl magnesium bromide and methyl-2-(chlorocarbonyl) benzoate as described in general procedure 1. *mp.*: 108-110 °C (101-102 °C)^{lit.},^{10a} Yield: 86%; Colourless solid; IR (KBr, cm^{-1}): 1727, 1673, ^1H NMR (CDCl_3 , 400 MHz): δ 7.30 - 8.00 (m, 8H), 3.6 (s, 3H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 195.5, 194.9, 165.8, 140.9, 139.1, 135.2, 132.2, 130.2, 129.8, 129.4, 128.5, 127.2, 51.9; MS (EI) m/z 274 (M^+).

2-Acetyl pyridine (14c).^{12, 14} Prepared from 2-pyridyl magnesium bromide and acetyl chloride as described in general procedure 2. Yield: 79 %; colorless liquid; IR (Neat, cm^{-1}): 1699, 1584, ^1H NMR (CDCl_3 , 400 MHz): δ 8.69 (br, d, 1H), 8.04 (br, d, 1H), 7.82-7.86 (m, 1H), 7.46 - 7.49 (m, 1H), 2.74 (s, 3H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 199.3, 152.9, 148.4, 136.3, 126.6, 121, 25.1; MS (EI) m/z 121 (M^+); HRESI-MS (m/z): calculated for $\text{C}_7\text{H}_7\text{NO}$: 144.0425 ($\text{M} + \text{Na}$), found 144.0423 ($\text{M} + \text{Na}$).

4-chloro-1-(thiophen-2-yl)butan-1-one (15c).¹⁵ Prepared from 2-thiophenyl magnesium bromide and 4-chlorobutyryl chloride as described in general procedure 2. Yield: 89 %; colorless liquid; IR (Neat, cm⁻¹): 1666, ¹H NMR (CDCl₃, 400 MHz): δ 7.75 (dd, 1H, *J* = 3.7 and 0.8 Hz), 7.64 (dd, 1H, *J* = 5 and 0.8 Hz), 7.13 (dd, 1H, *J* = 4.8 and 3.9 Hz), 3.66 (t, 2H, *J* = 6.2 Hz), 3.12 (t, 2H, *J* =), 2.22 (m, 2H); ¹³C NMR (CDCl₃, 75 MHz): δ 191.8, 143.9, 133.6, 131.9, 128.1, 44.4, 35.9, 26.91; MS (EI) *m/z* 121(M⁺); HRESI-MS (*m/z*): calculated for C₈H₁₀ClOS: 189.0141(M+H), found 189.0141(M+H).

Propiophenone (16c).¹⁶ Colorless liquid; Yield - 54%; IR (Neat, cm⁻¹): 1687; ¹H NMR (CDCl₃, 400 MHz): δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.56-7.53(m, 1H), 7.47-7.43(m, 2H), 3.00(q, *J* = 7.2 Hz, 2H), 1.22(t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 200.81, 136.85, 132.83, 128.49, 127.91, 31.72, 8.18; HRESI-MS (*m/z*): Calculated for C₉H₁₀O (M + Na): 157.0629, found (M + Na): 157.0630

Butyrophenone (17c).¹⁶ Colorless liquid; Yield - 55%; IR (Neat, cm⁻¹): 1686; ¹H NMR (CDCl₃, 400 MHz): δ 7.95(d, *J* = 7.7 Hz), 7.56-7.52 (m, 1H), 7.46-7.43 (m, 2H), 2.94(t, *J* = 7.2 Hz, 2H), 1.79-1.74 (m, 2H), 1.00(t, *J* = 7.4 Hz, 3H), ¹³C NMR (CDCl₃, 100 MHz): δ 136.99, 132.79, 128.45, 127.94, 40.41, 17.67, 13.80; HRESI-MS (*m/z*): Calculated for C₁₀H₁₂O (M + Na): 171.0786, found (M + Na): 171.0788

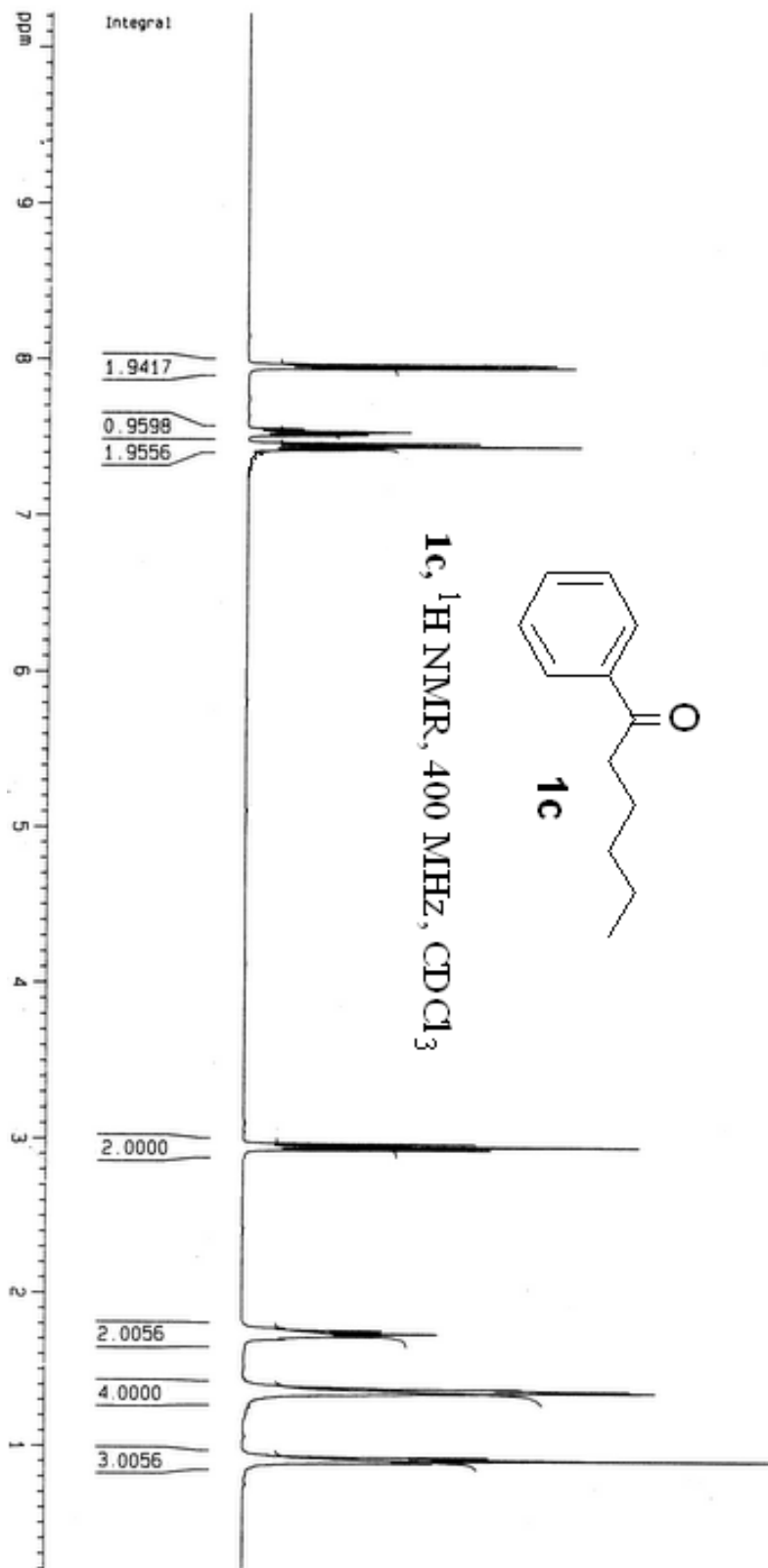
1-Chloroheptan-4-one (18c).¹⁷ 1-chloroheptan-4-one; Colorless liquid; Yield - 65%; IR (Neat, cm⁻¹): 1741; ¹H NMR (CDCl₃, 400 MHz): δ 3.58(t, *J* = 6.2 Hz, 2H), 2.60 (t, *J* = 7 Hz, 2H), 2.40 (t, *J* = 7.3 Hz, 2H), 1.64-1.58(m, 2H), 0.92(t, *J* = 7.4 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 209.92, 44.86, 44.51, 39.18, 26.21, 17.27, 13.69; HRESI-MS (*m/z*): Calculated for C₇H₁₃ClO (M + H): 149.0733, found (M + Na): 149.0732.

References:

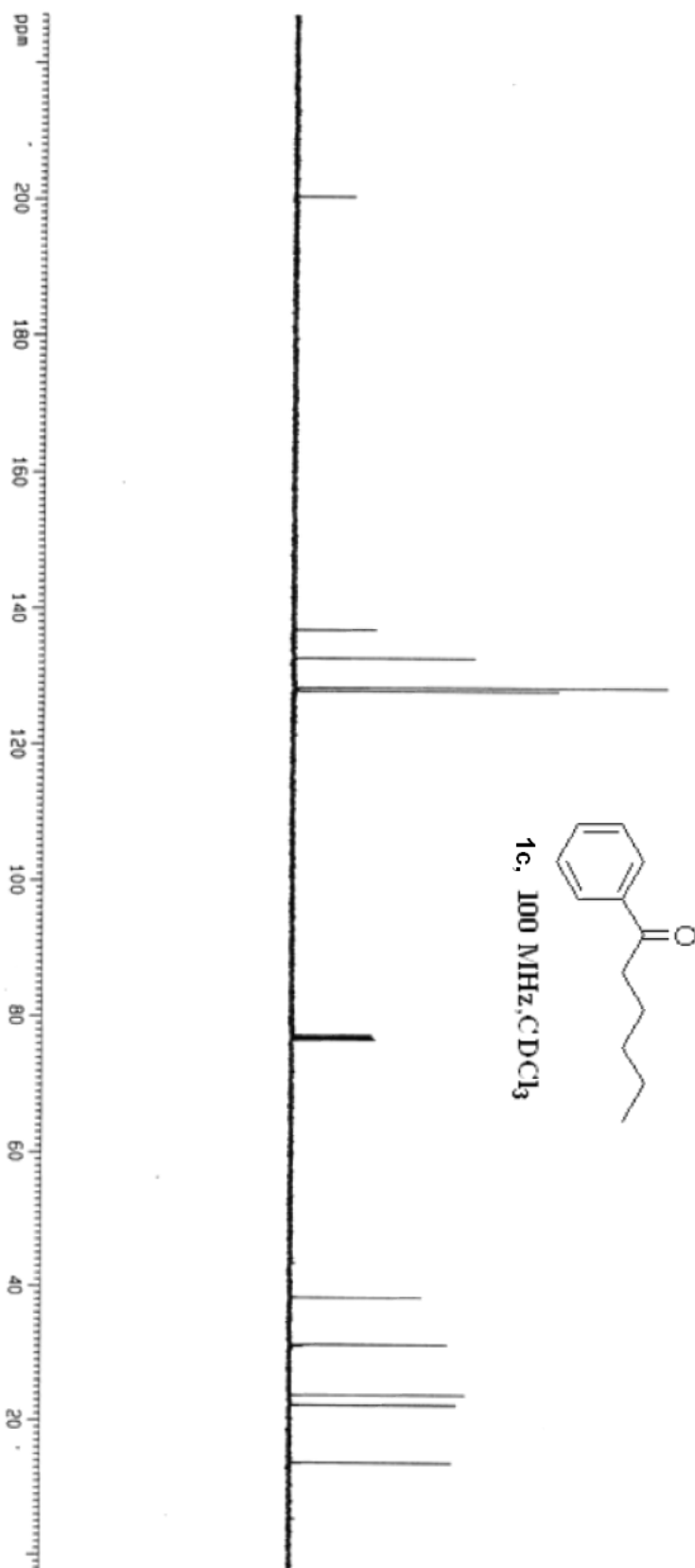
- (1) (a) Fiandanese, V.; Marchese, G.; Ronzini, L. *Tetrahedron Lett.* **1983**, *24*, 3677. (b) Duhamel, L.; Poirier, J. M. *J. Org. Chem.* **1979**, *44*, 3585.
- (2) Gregg, B. T.; Golden, K. C.; Quinn, J. F. *J. Org. Chem.* **2007**, *72*, 5890.
- (3) (a) Schliemann, W.; Buege, A.; Reppel, L. *Pharmazie*, **1980**, *35*, 140. (b) Noguchi, T.; Hasegawa, M.; Tomisawa, K.; Mitsukuchi, M. *Bioorg. Med. Chem.* **2003**, *11*, 4729.
- (4) (a) Dictionary of Organic Compounds, 6th ed.; Bukiyahan, J.; Macdonald, F., Chapman and Hall Electric Publishing House, London, 1996. (b) Bellale, E. V.; Bhalerao, D. S.; Akamanchi, K. G. *J. Org. Chem.* **2008**, *73*, 9473.
- (5) (a) Rao, M. L. N.; Venkatesh, V.; Banerjee, D. *Tetrahedron*, **2007**, *63*, 12917. (b) Xing, D.; Guan, B.; Cai, G.; Fang, Z.; Yang, L.; Shi, Z. *Org. Lett.* **2006**, *8*, 693.
- (6) (a) Cai, M.; Peng, J.; Hao, W.; Ding, G. *Green Chem.*, **2011**, *13*, 190. (b) Nishimoto, Y.; Babu, S. A.; Yasuda, M.; Baba, A. *J. Org. Chem.* **2008**, *73*, 9465.
- (7) (a) Brown, E.; Leze, A.; Touet, J. *Tetrahedron: Asymmetry*, **1992**, *3*, 841. (b) Huang, Y. -C.; Majumdar, K. K.; Cheng, C. -H. *J. Org. Chem.* **2002**, *67*, 1682.
- (8) Uyanik, M.; Akakura, M.; Ishihara, K. *J. Am. Chem. Soc.* **2009**, *131*, 251.
- (9) Kwon, M. S.; Kim, N.; Park, C. M.; Lee, J. S.; Kang, K. Y.; Park, J. *Org. Lett.* **2005**, *7*, 1077.
- (10) (a) Griffith, R. C.; Napier, J. J. USP 4855462A (1989); (b) Singh, P. N. D.; Muthukrishnan, S.; Murthy, R. S.; Klima, R. F.; Mandel, S. M.; Hawk, M.; Yarbrough N.; Gudmundsdóttir, A. D. *Tetrahedron Lett.* **2006**, *44*, 9169. (c) Anilkumar, H. G.; Vijay, T.; Yathirajan, H. S.; Narasimhamurthy, T.; Rathore, R. S. *Acta Cryst.* (2005). **E61**, o3332-o3333.
- (11) Lo Fiego, M. J.; Badajoz, M. A; Silbestri, G. F.; Lockhart, M.T.; Chopa, A. B. *J. Org. Chem.* **2008**, *73*, 9184.
- (12) Puterova, Z.; Andicsova, A.; Vegh, D. *Tetrahedron*. **2008**, *64*, 11262.
- (13) Bhatt, M. V.; Rao, K. S.; Rao, G. V., *J. Org. Chem.* **1977**, *42*, 2697.
- (14) Xu, D.; Liu, Z.; Tong, W.; T.; Xu, L.; Hyder, Z.; Xiao, J. *Tetrahedron Lett.* **2008**, *49*, 6104.

- (15) Borate, H. B.; Gaikwad, A.G.; Maujan, S. R.; Sawargave, S. P.; Kalal, K. M. *Tetrahedron Lett.* **2007**, *48*, 4869
- (16) (a) Zhao, B; Lu, X. *Tetrahedron Lett.* **2006**, *47*, 6765. (b) (2) Wang, D.; Zhang, Z. *Org. Lett.* **2003**, *5*, 4645. (c) Pitts, M. R.; Harrison, J. R.; Moody, J. J. *Chem. Soc. Perkin. Trans 1.* **2001**, 955.
- (17) Hart, H.; Curtis, O. E. Jr. *J. Am. Chem. Soc.* **1957**, *79*, 931.

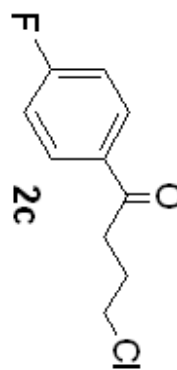
¹H NMR of 1c



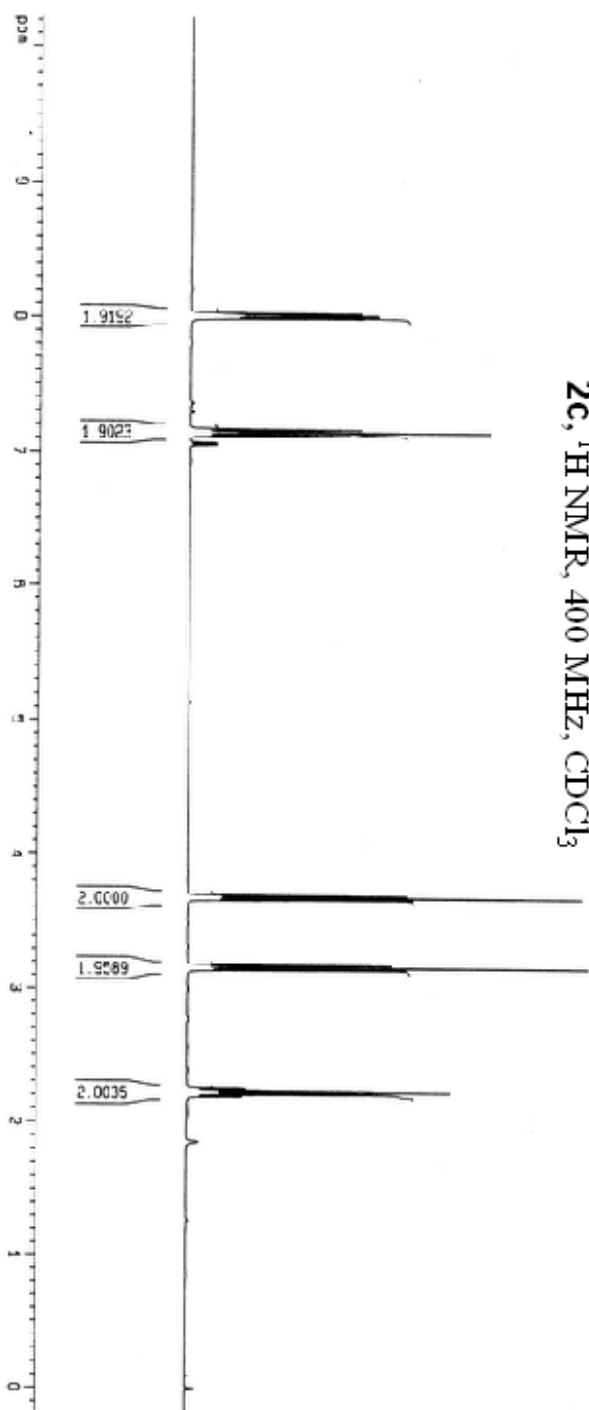
^{13}C NMR of **1c**



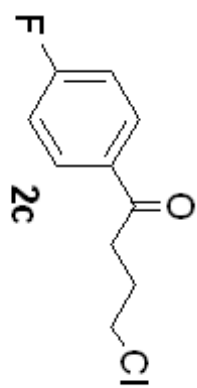
¹H NMR of 2c



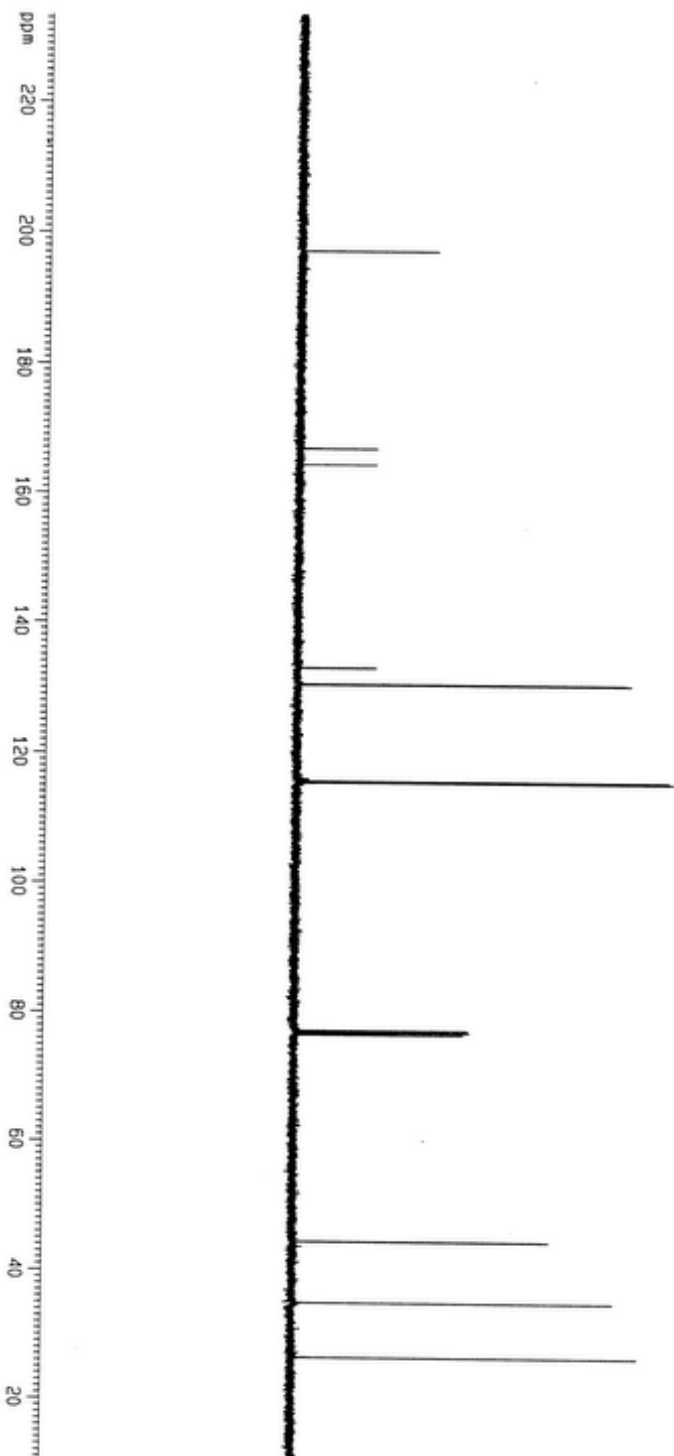
2c, ¹H NMR, 400 MHz, CDCl₃



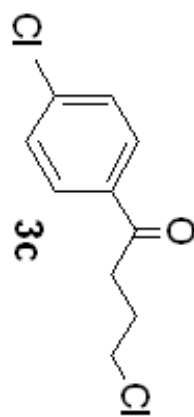
^{13}C NMR of 2c



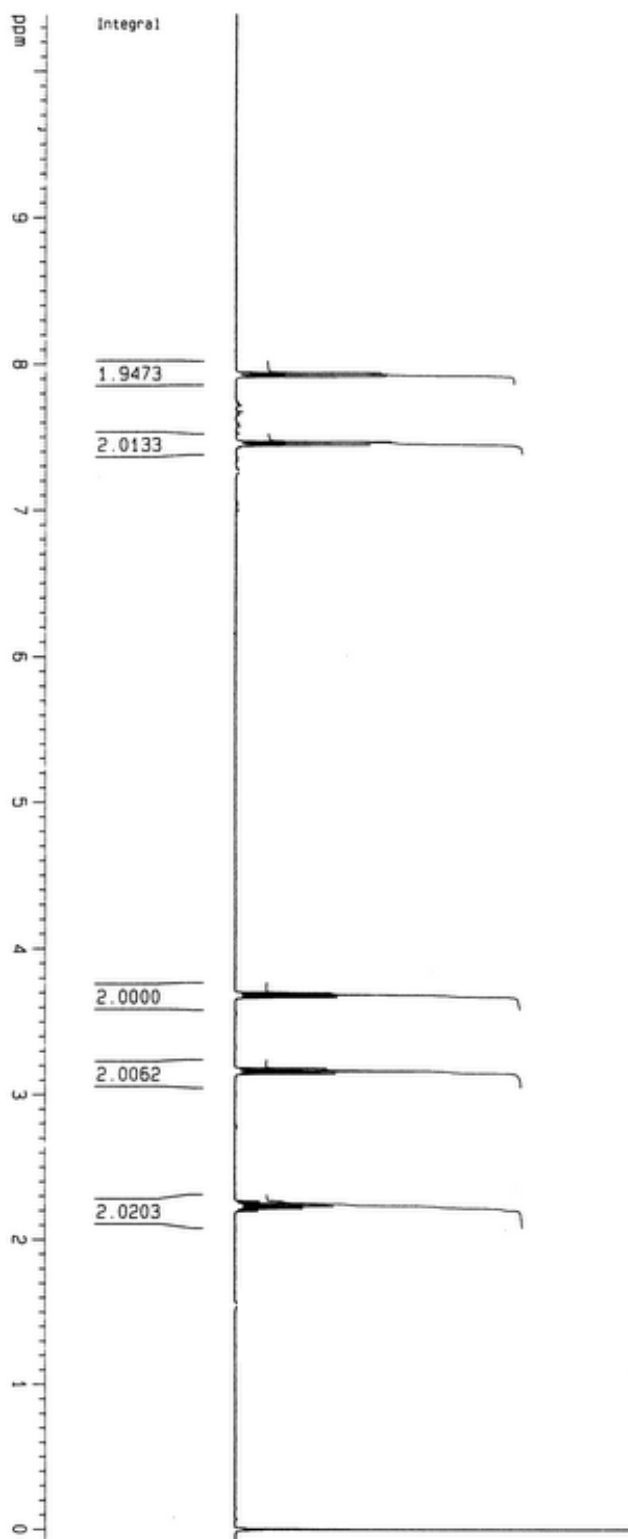
2c, ^{13}C NMR, 100 MHz, CDCl_3

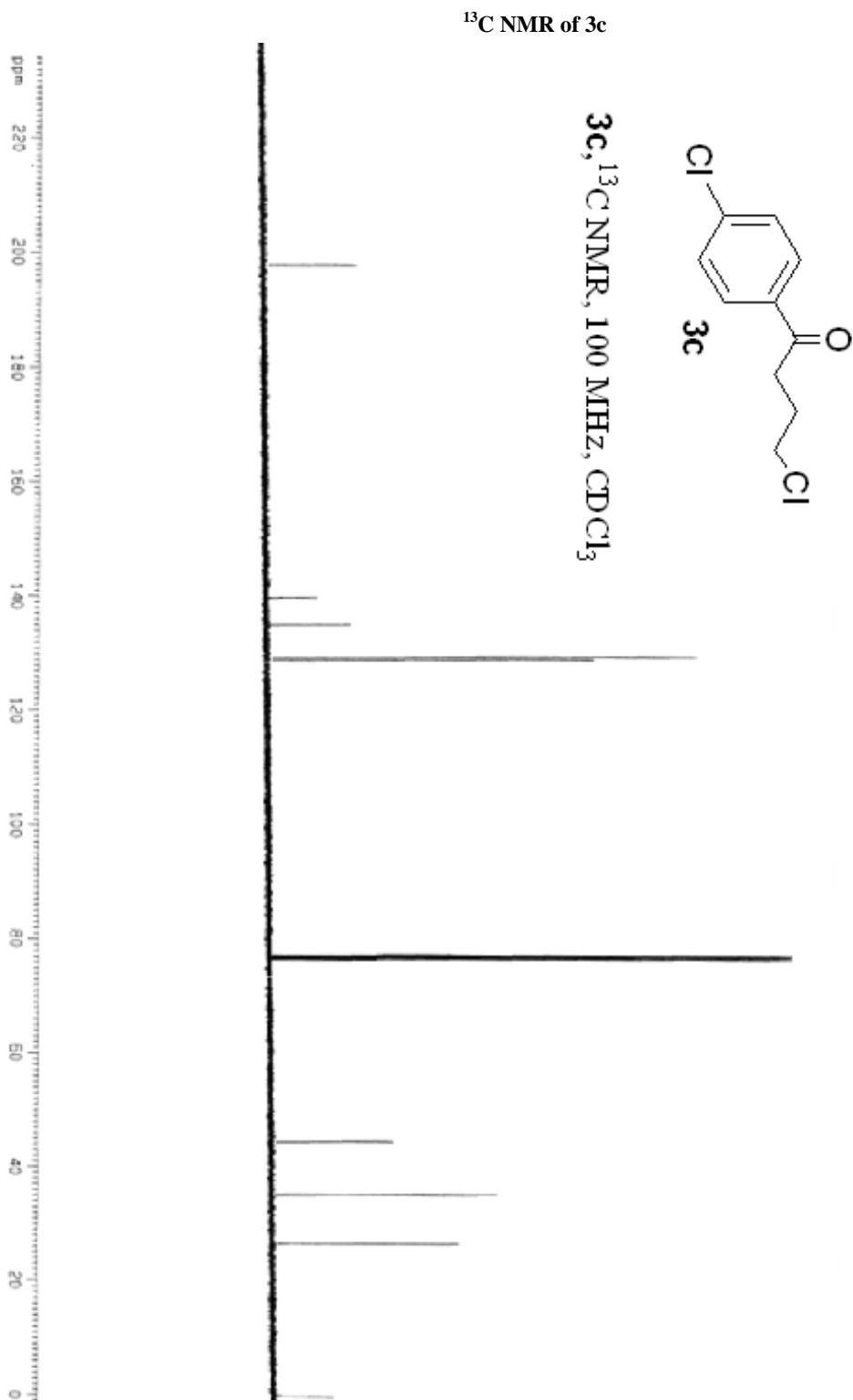


^1H NMR of **3c**

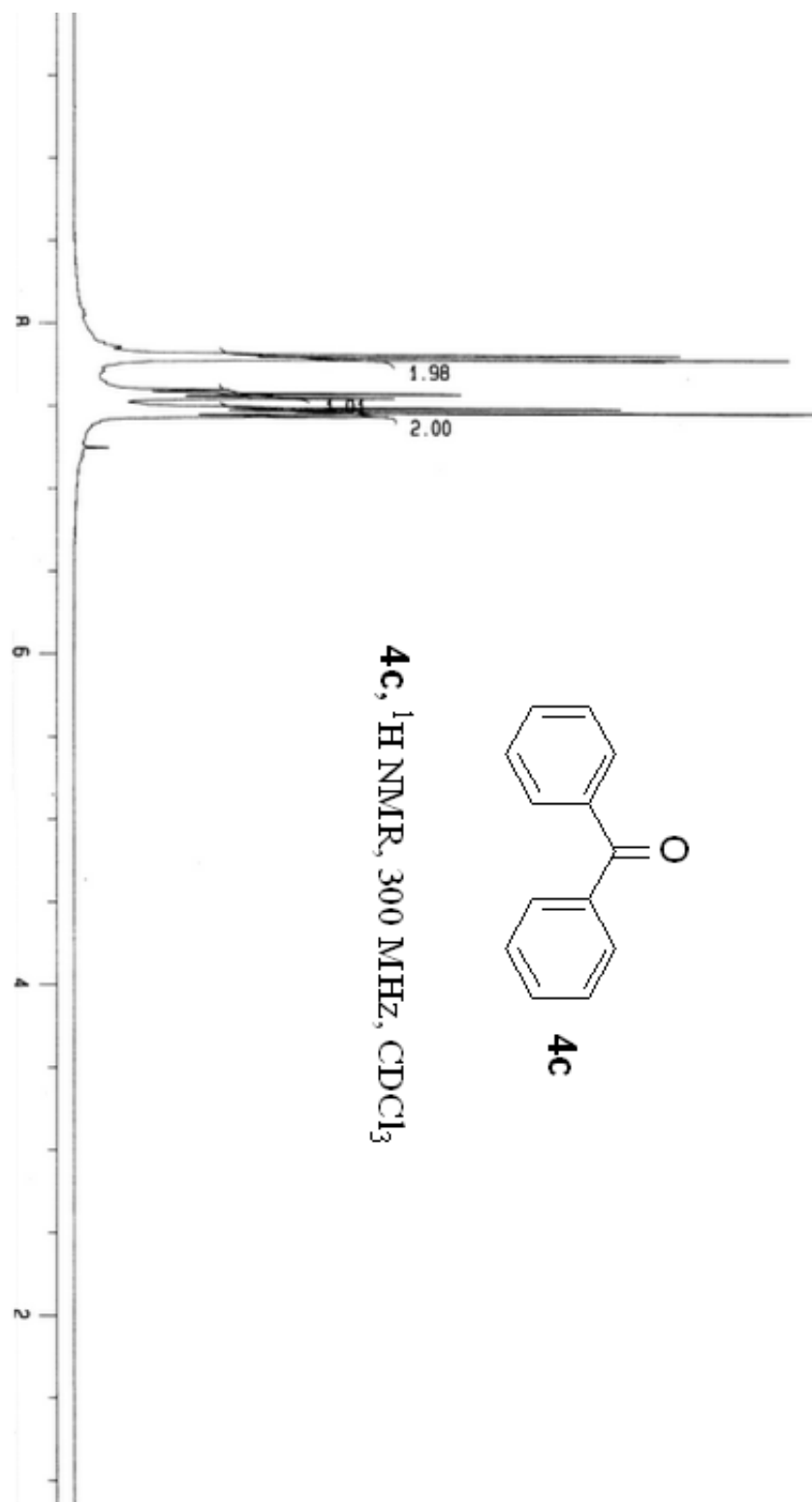


3c, ^1H NMR, 400 MHz, CDCl_3

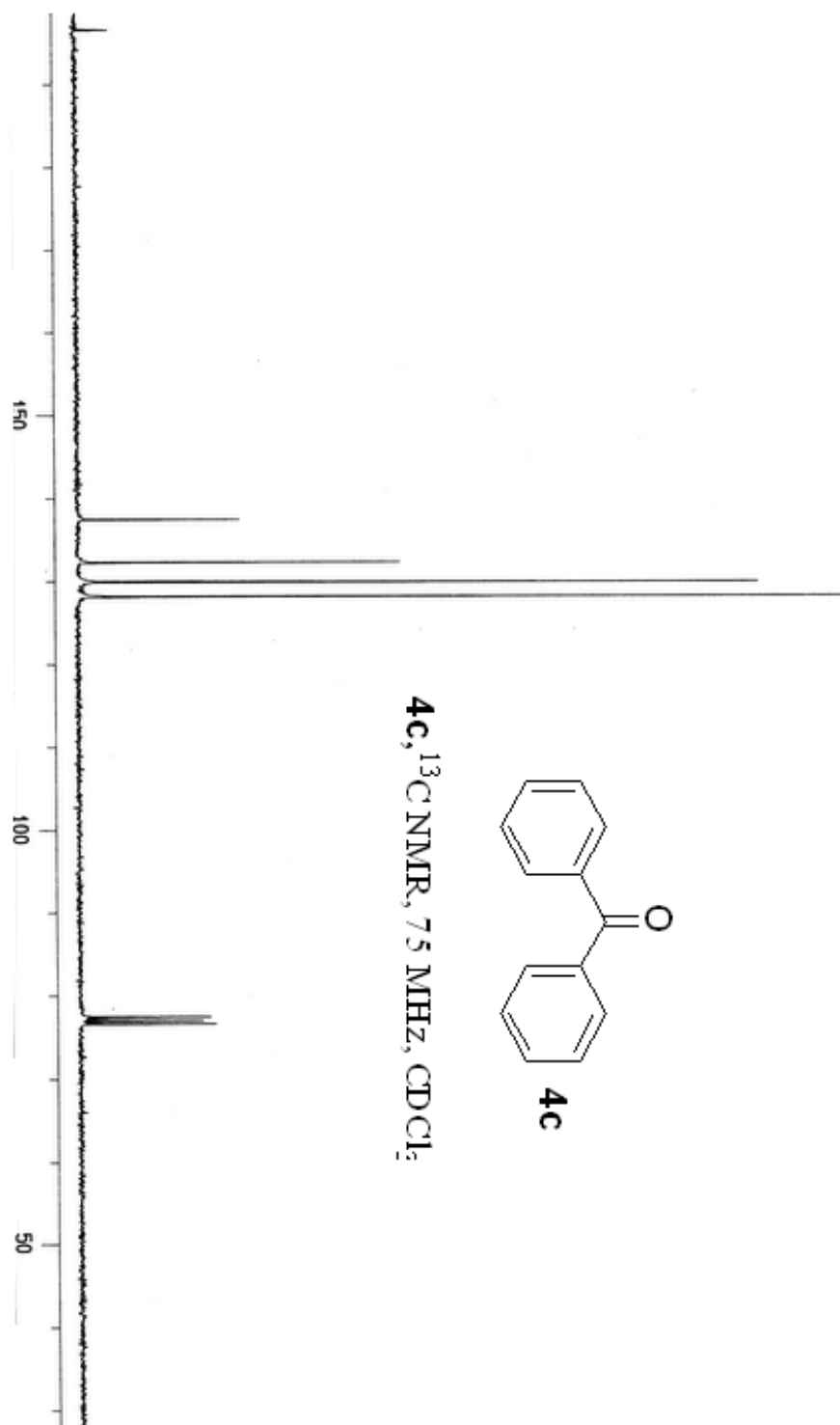




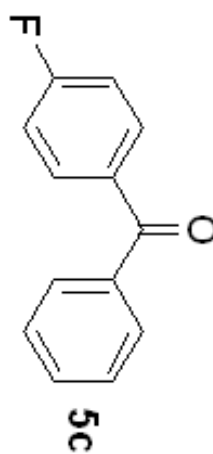
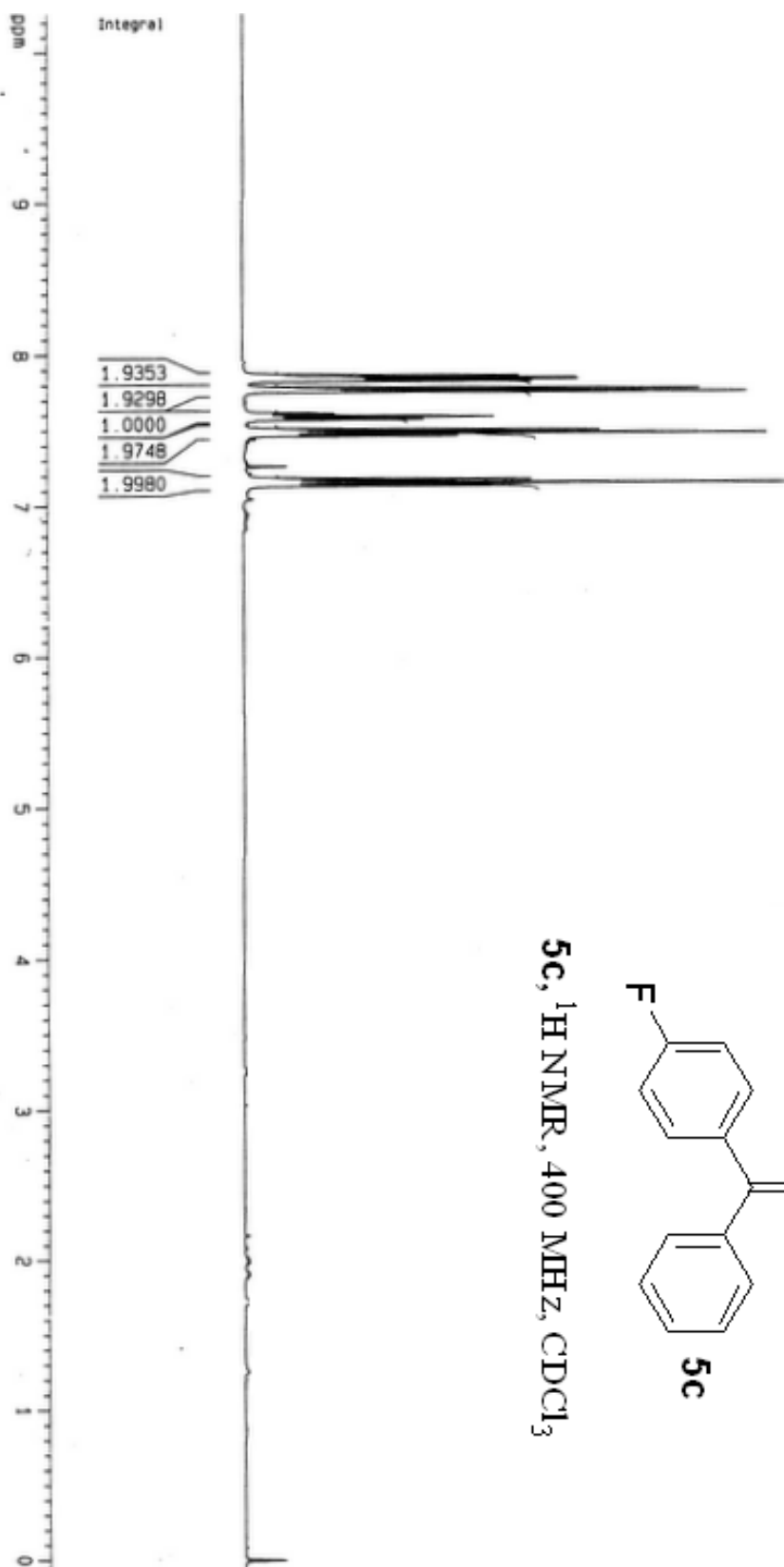
¹H NMR of 4c



^{13}C NMR of **4c**

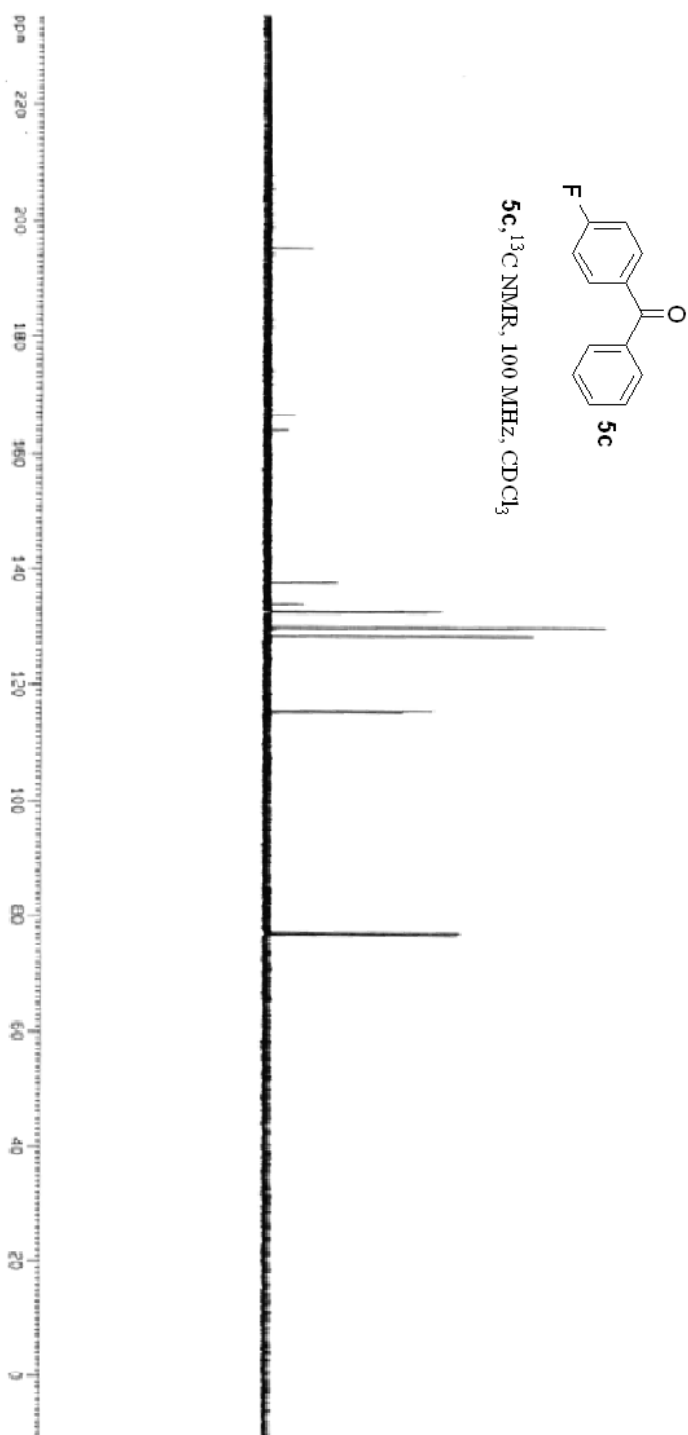


¹H NMR of 5c

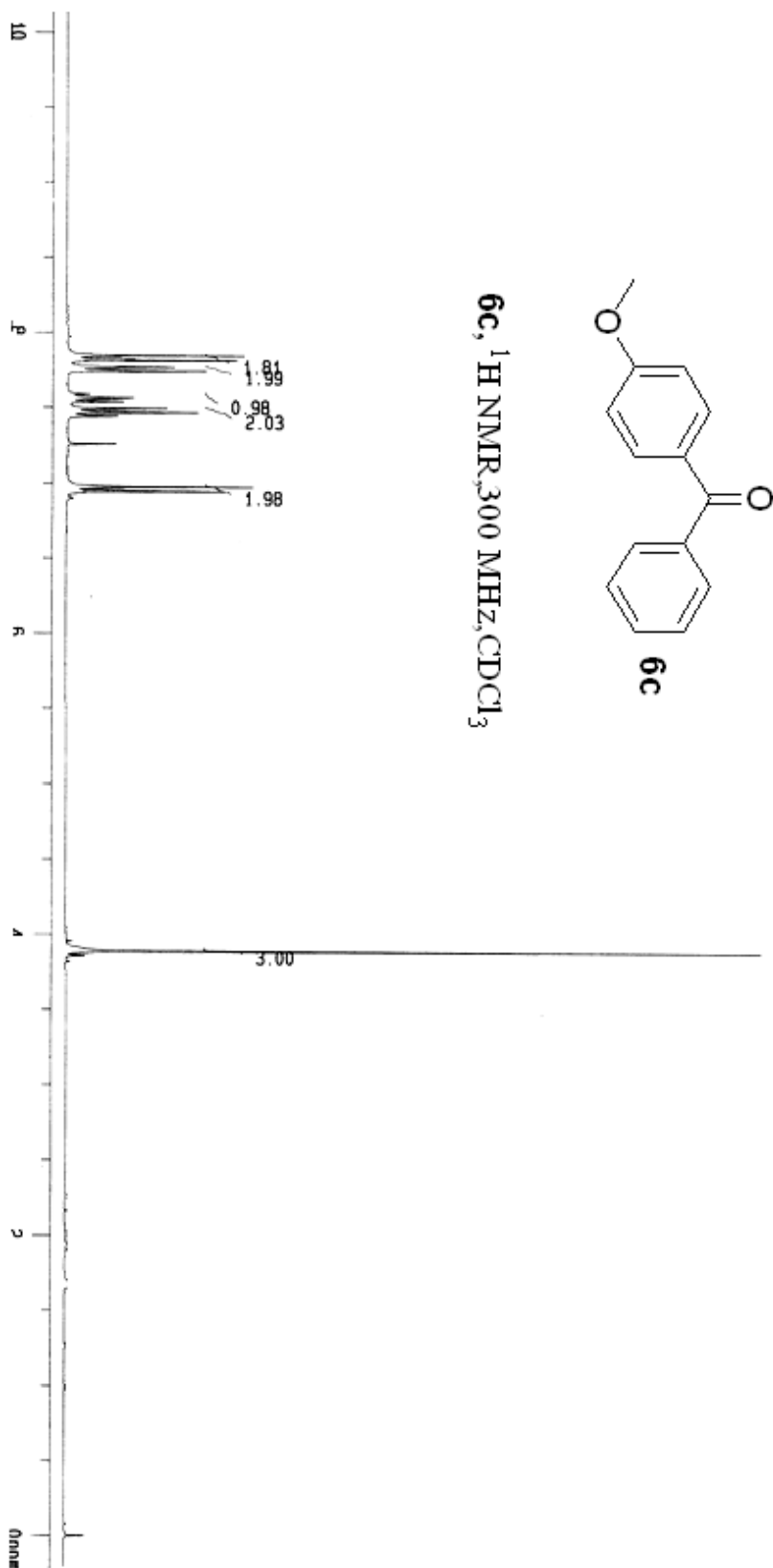


5c, ¹H NMR, 400 MHz, CDCl₃

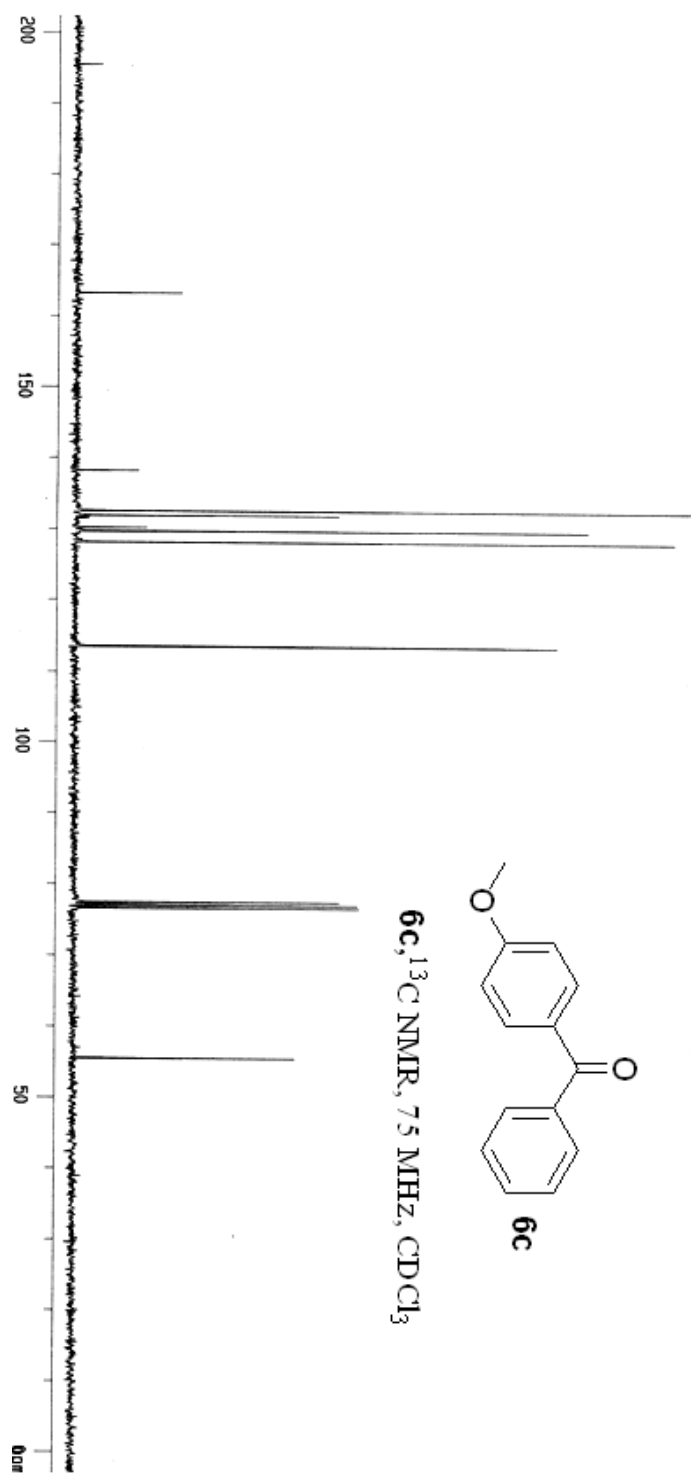
^{13}C NMR of 5c



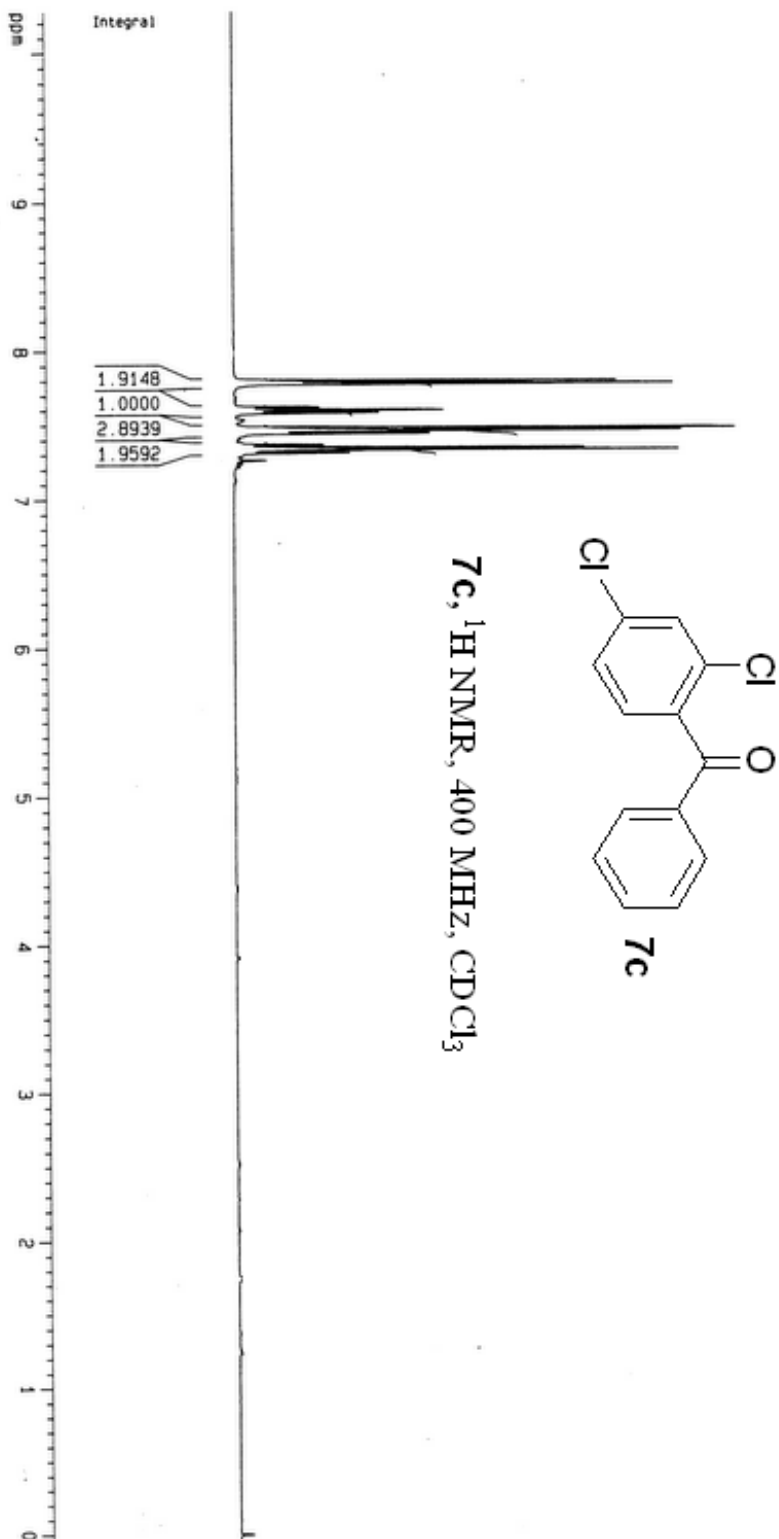
¹H NMR of 6c



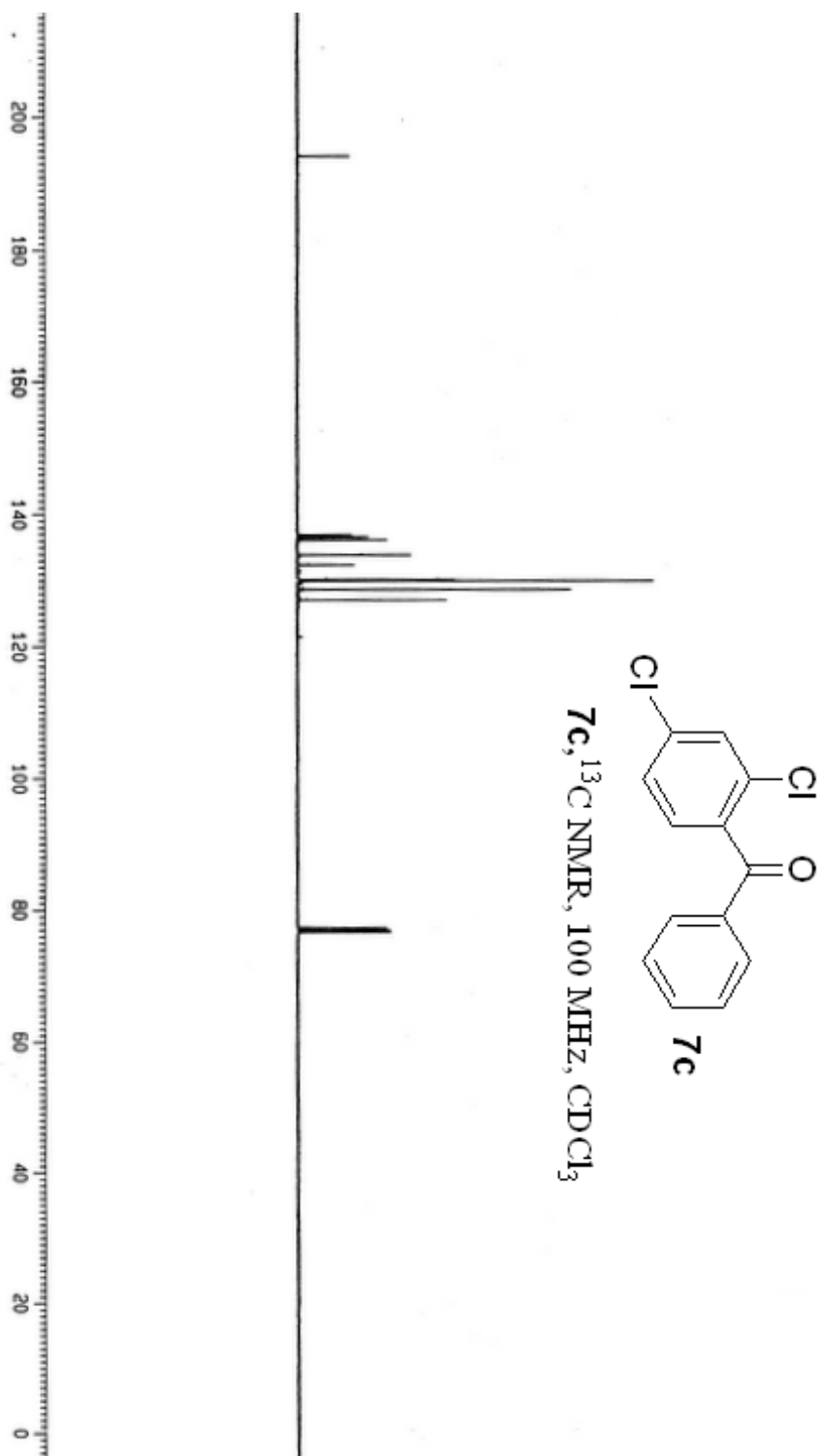
^{13}C NMR of **6c**



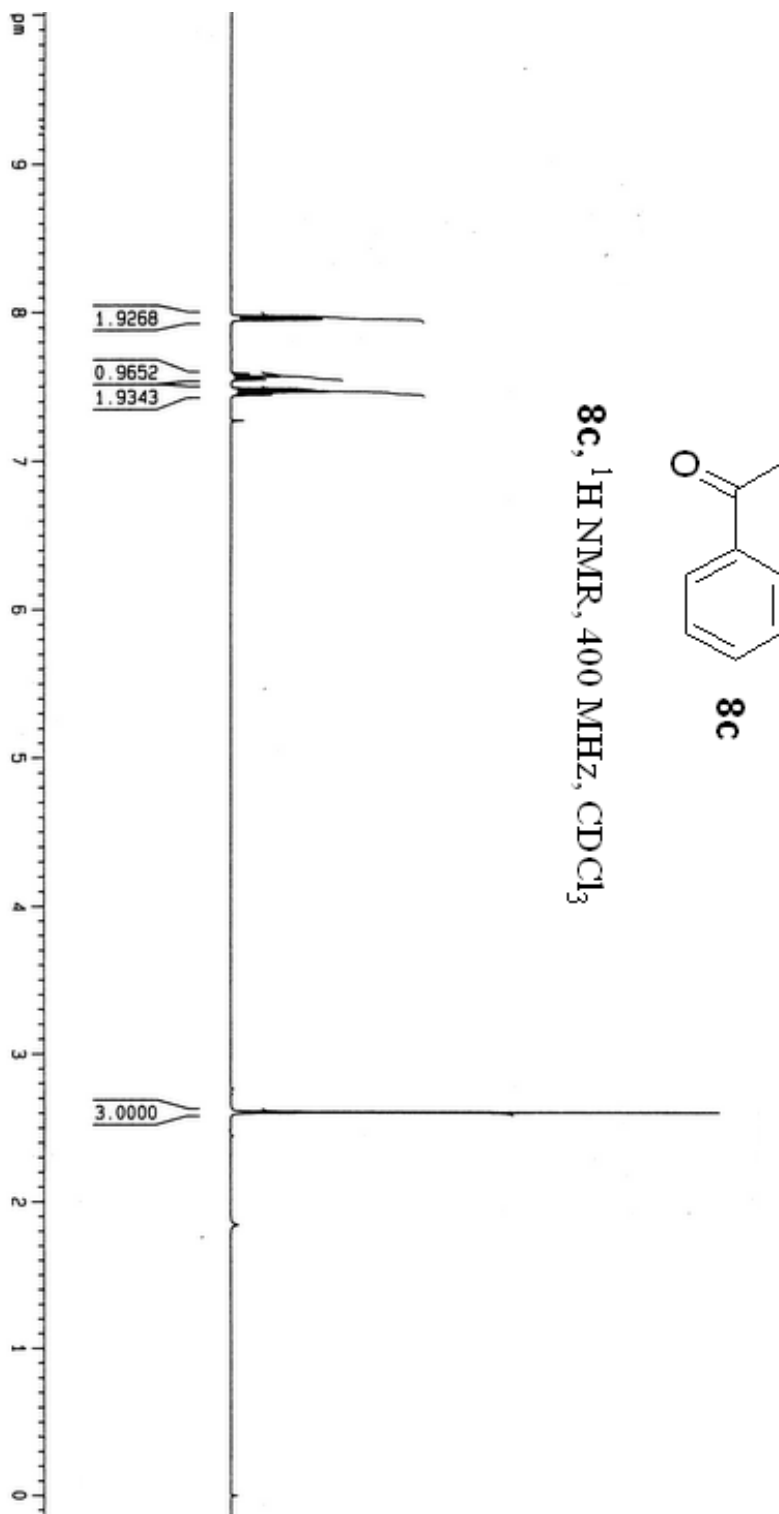
¹H NMR of 7c



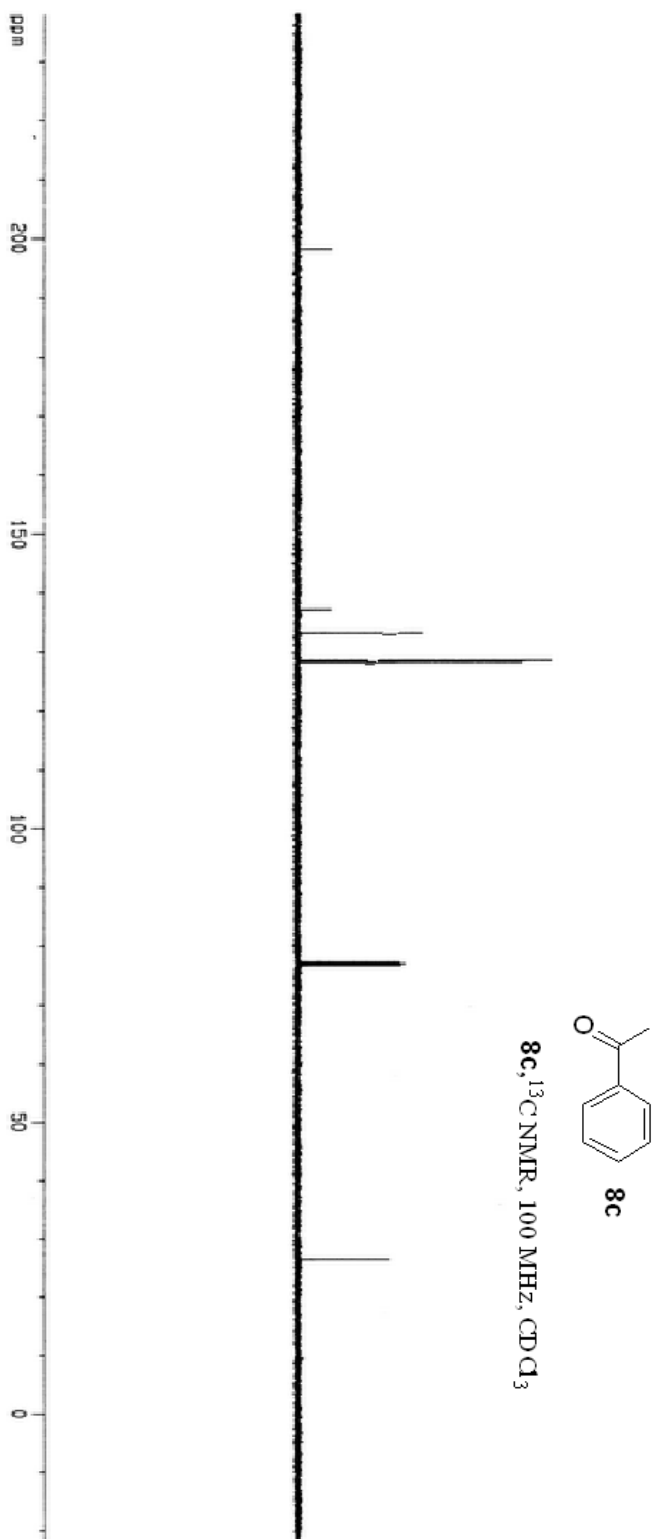
^{13}C NMR of **7c**



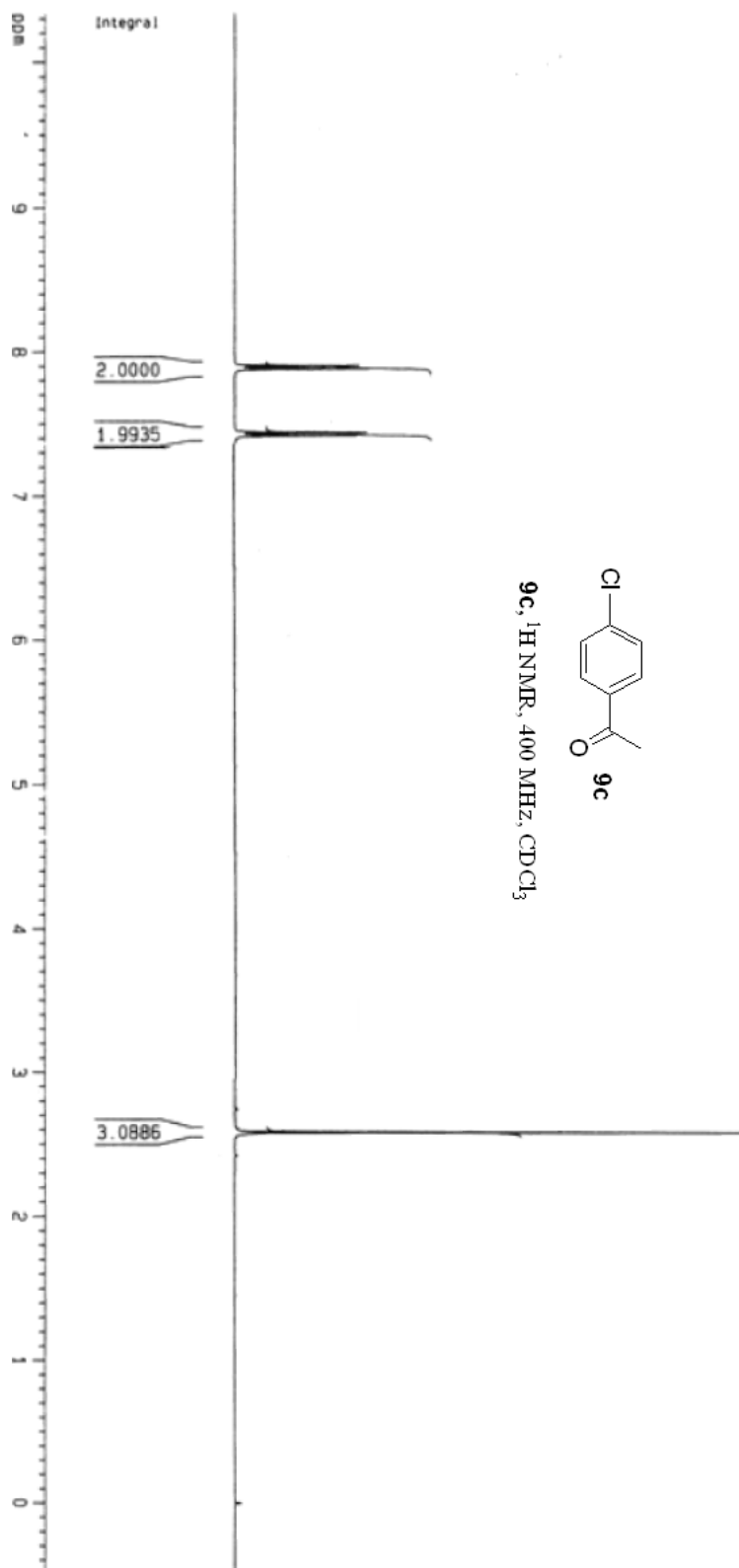
^1H NMR of 8c



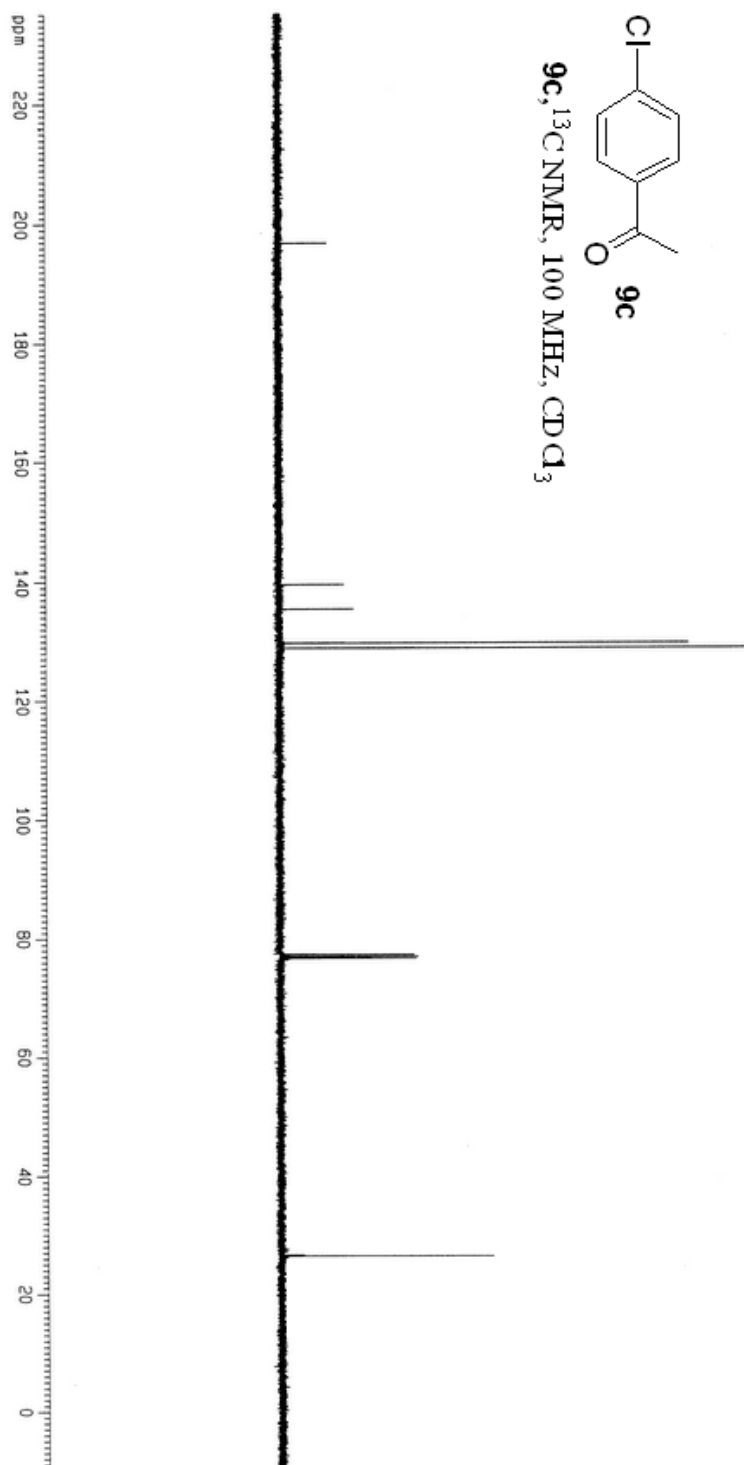
^{13}C NMR of **8c**



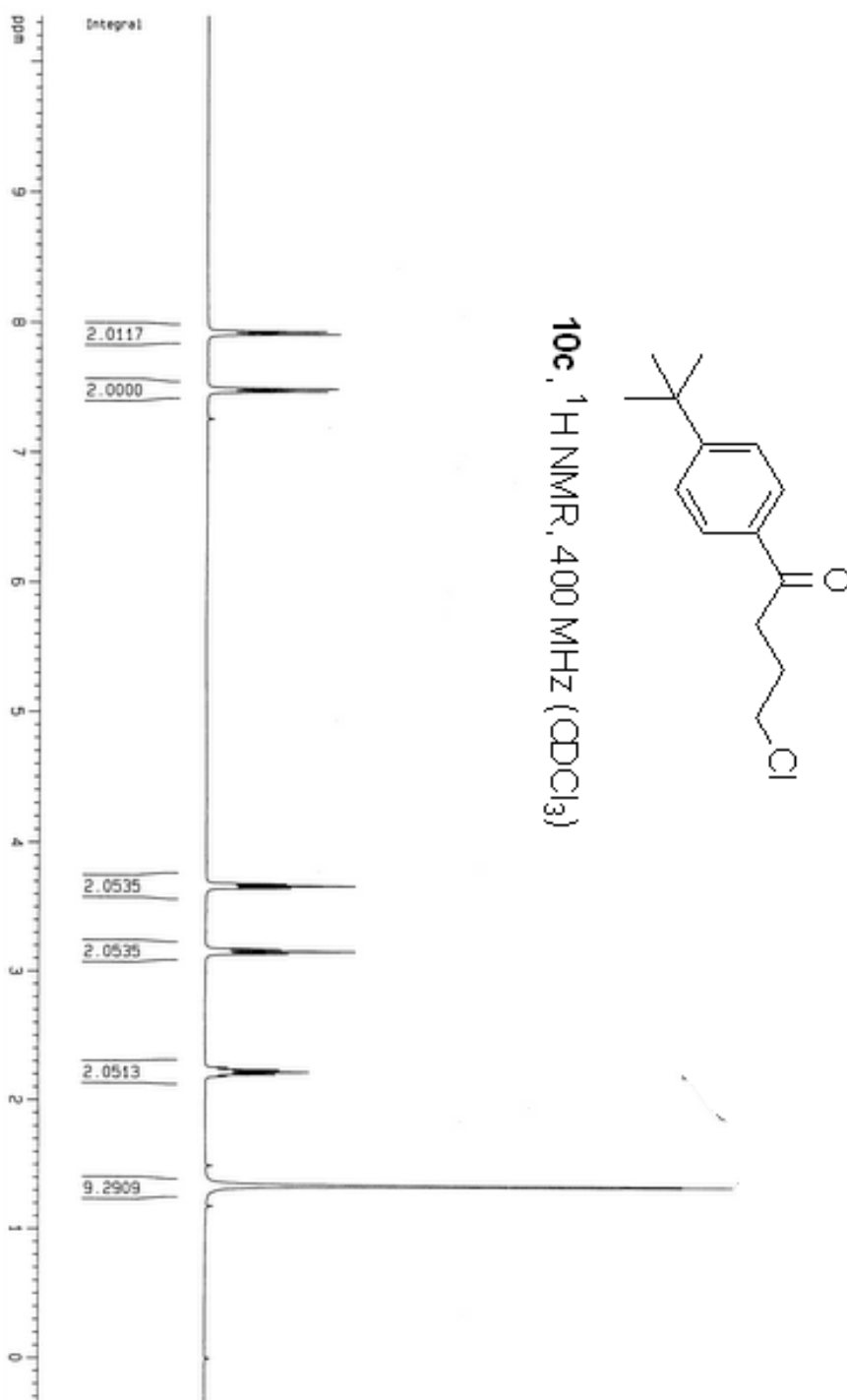
¹H NMR of 9c



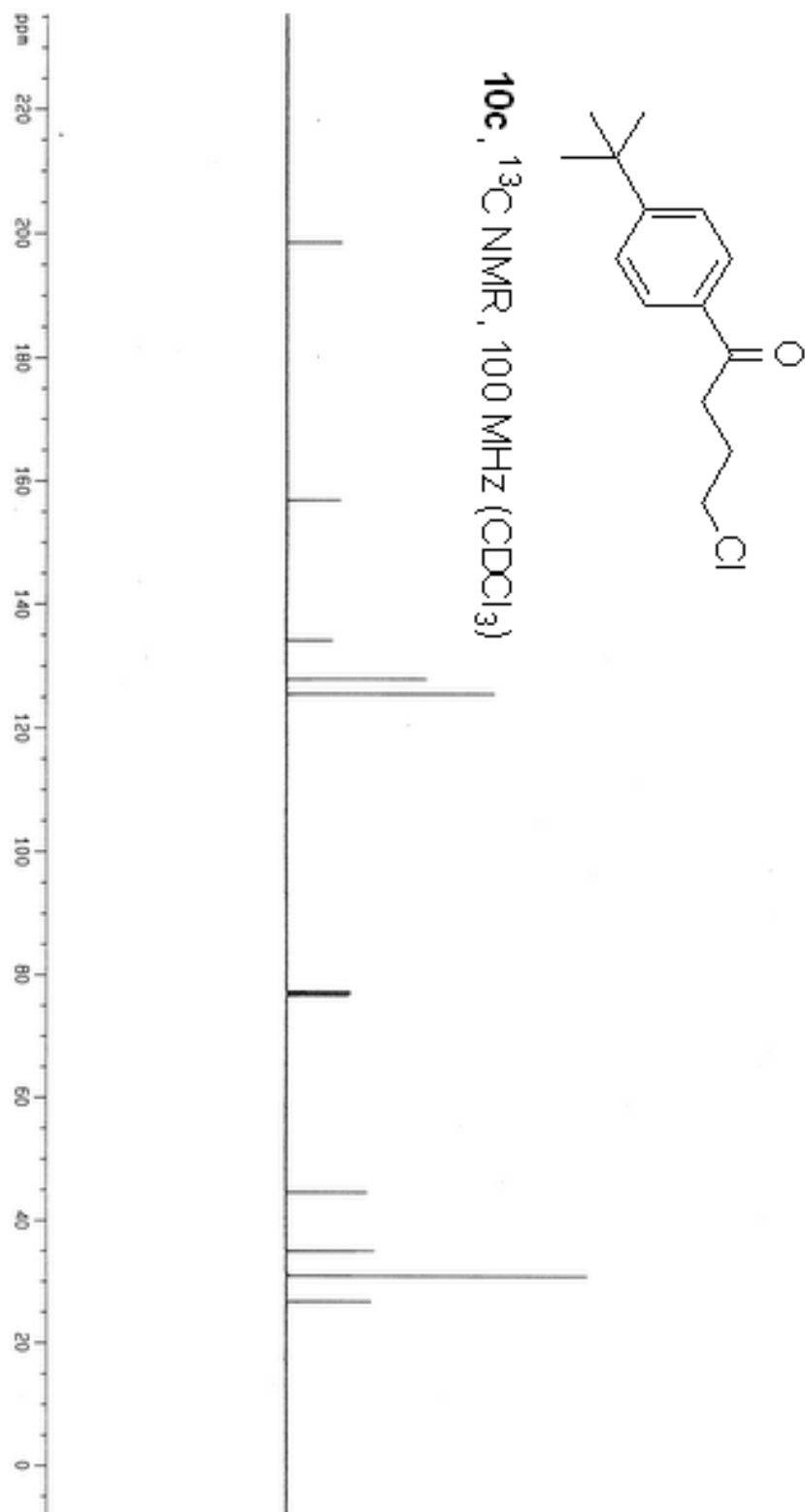
^{13}C NMR of **9c**



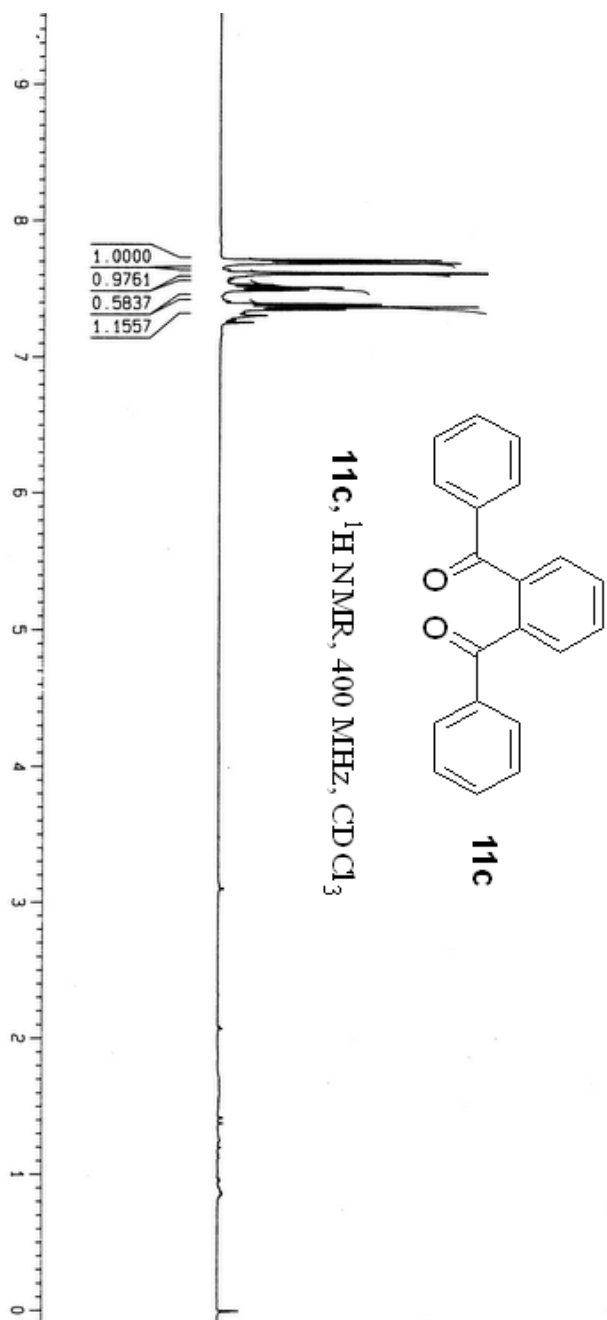
¹H NMR OF 10c



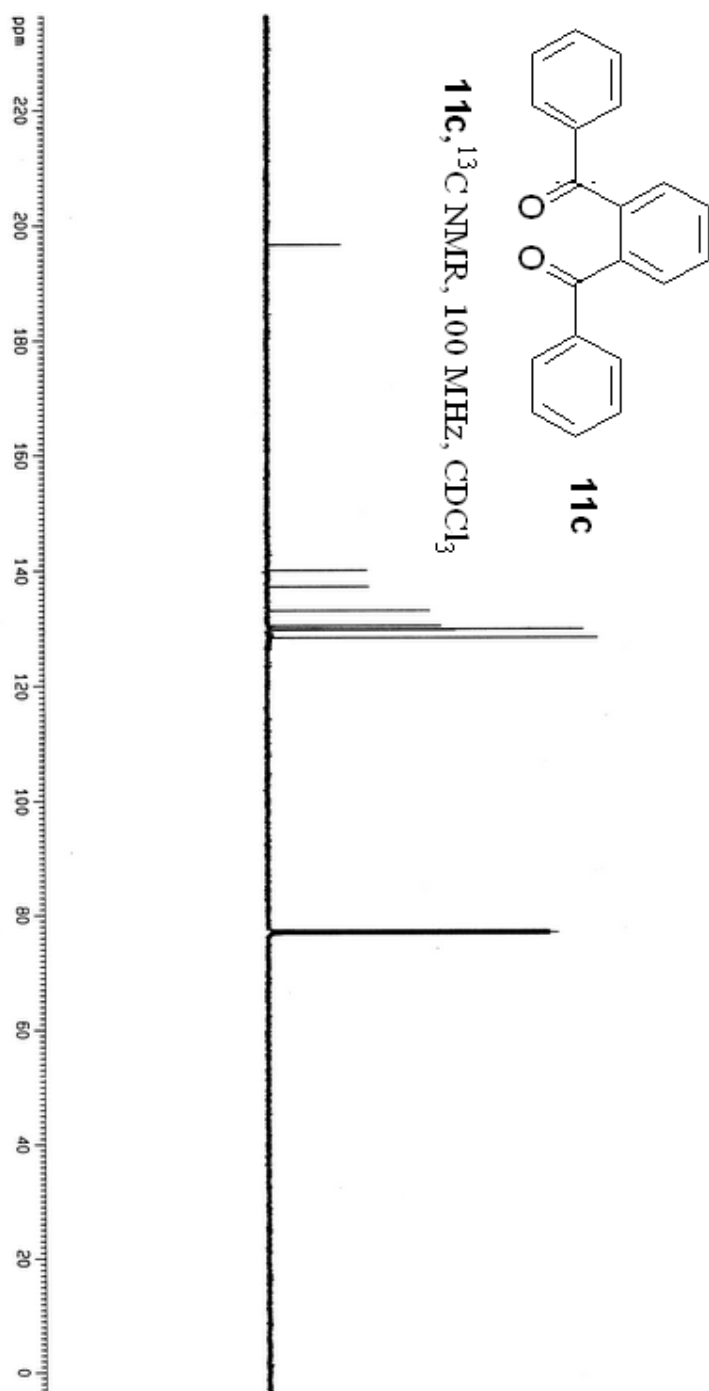
¹³C NMR of 10c



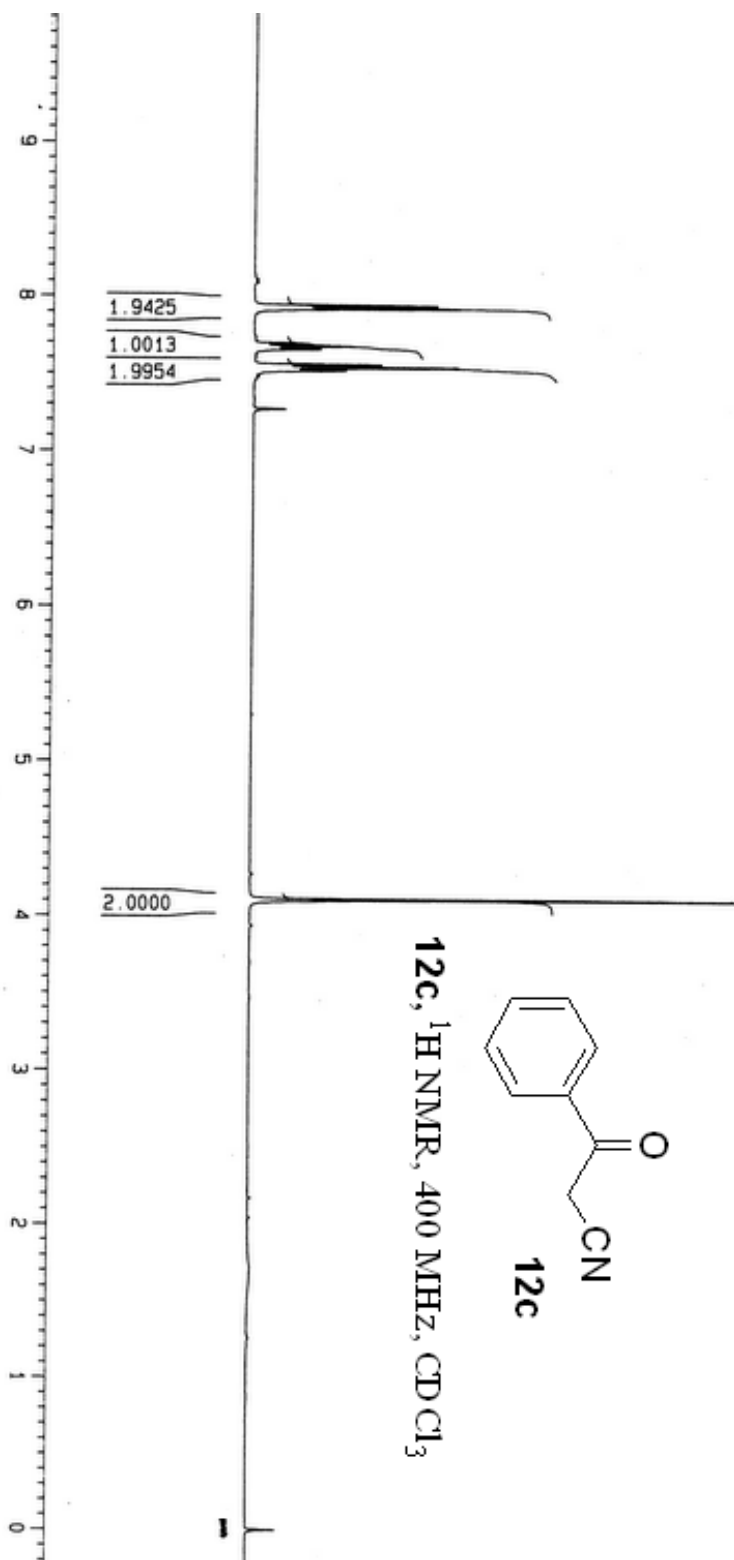
¹H NMR of 11c



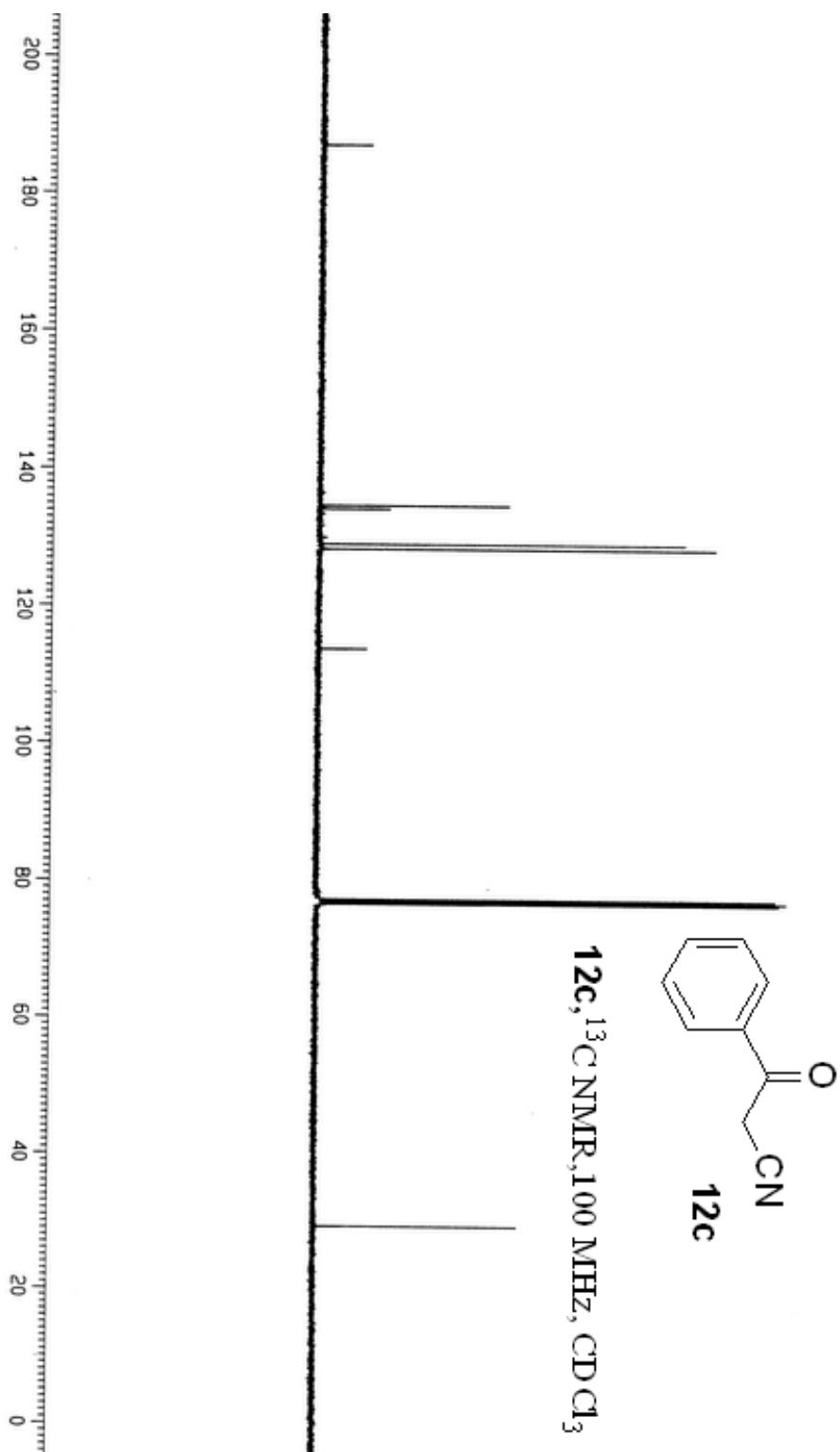
^{13}C NMR of 11c



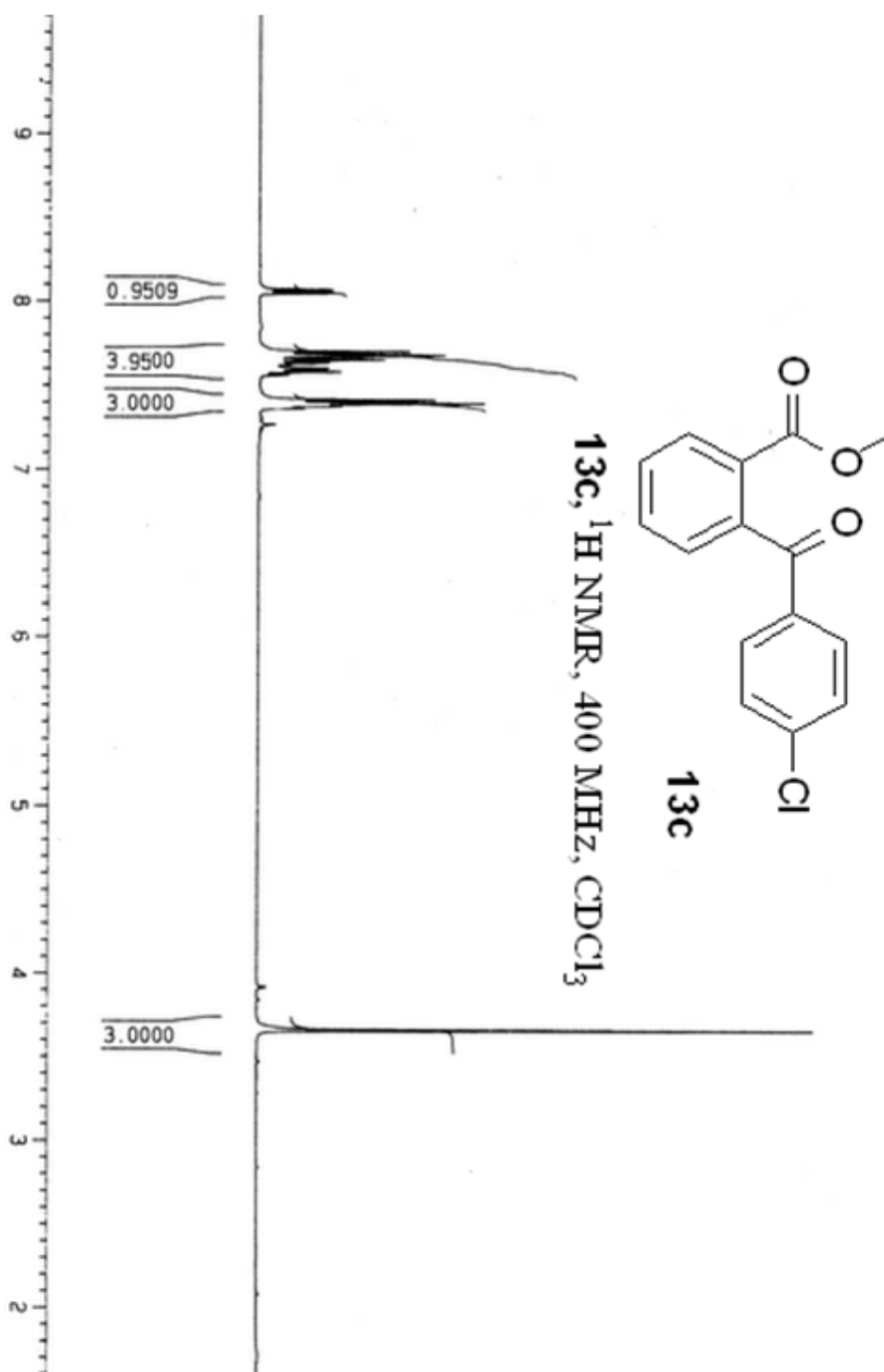
¹H NMR of 12c



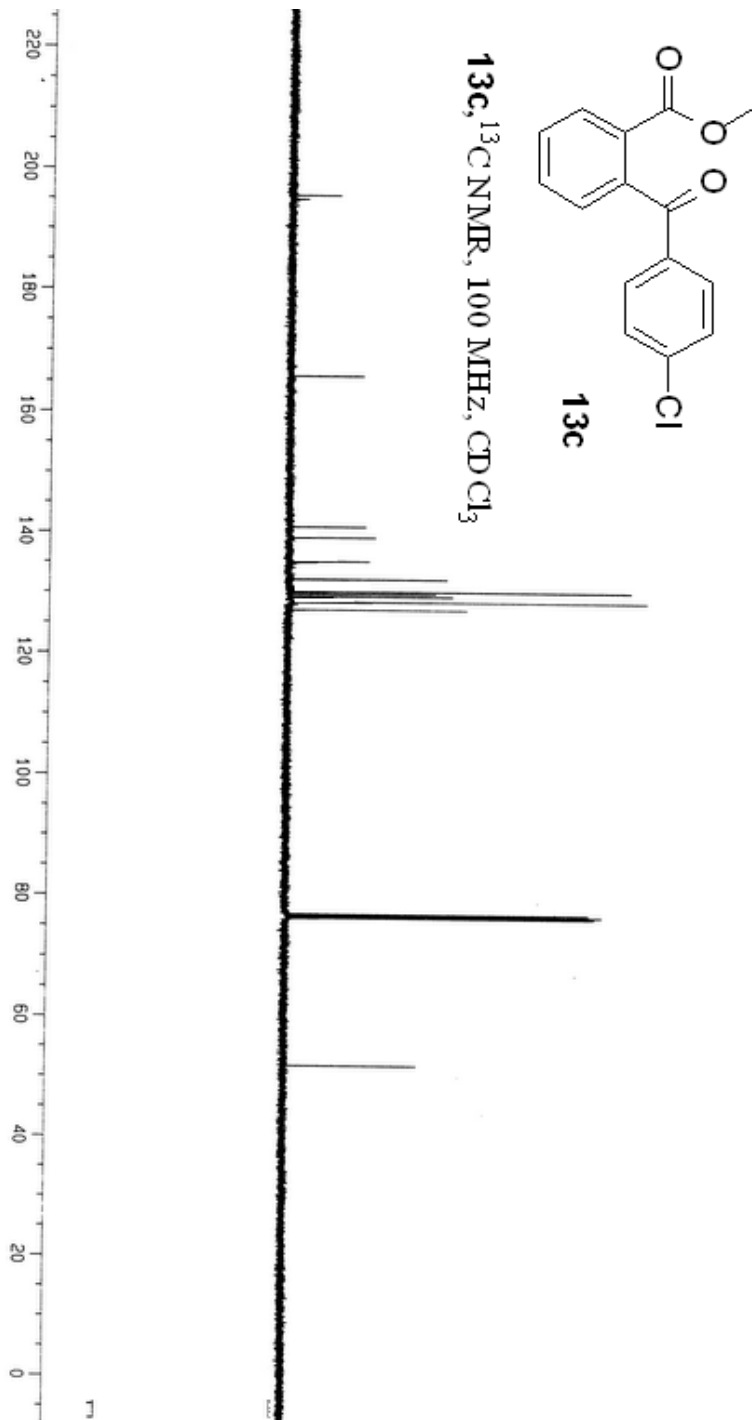
^{13}C NMR of 12c



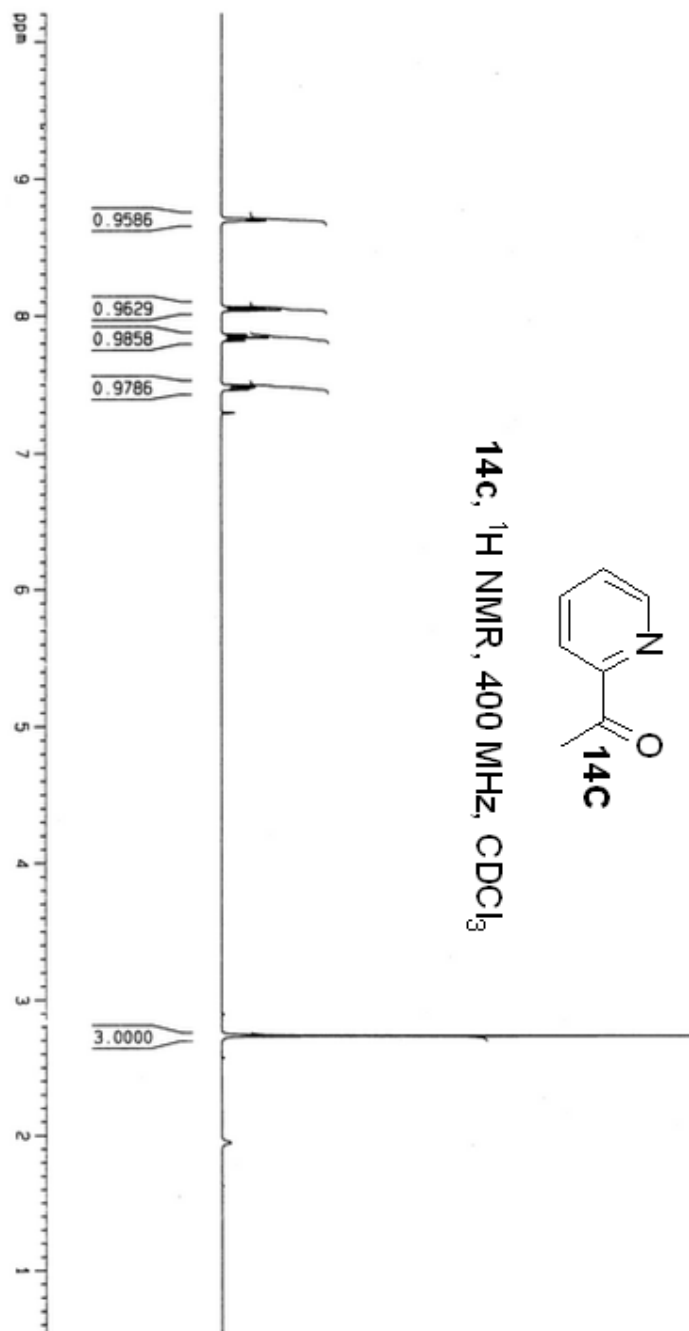
¹H NMR of 13c



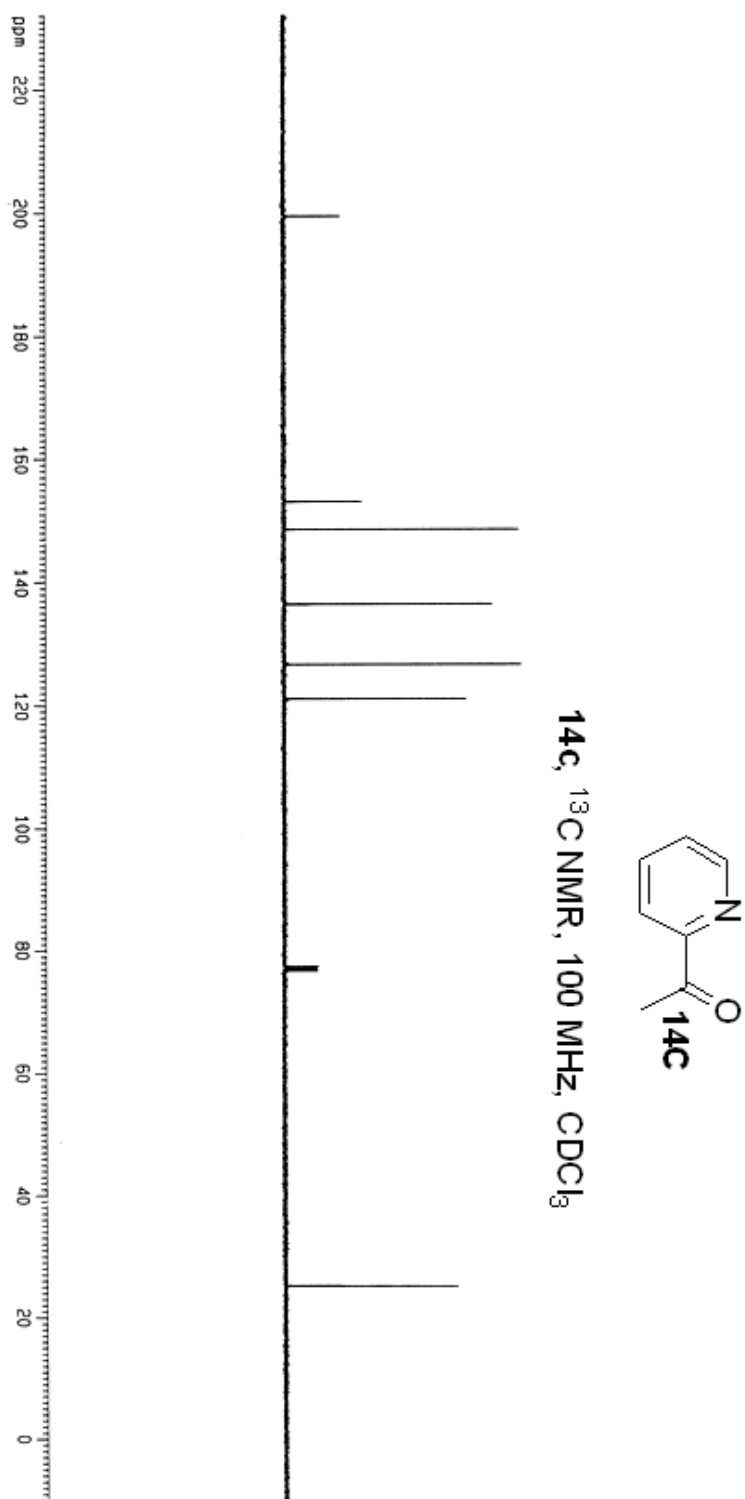
^{13}C NMR of 13c



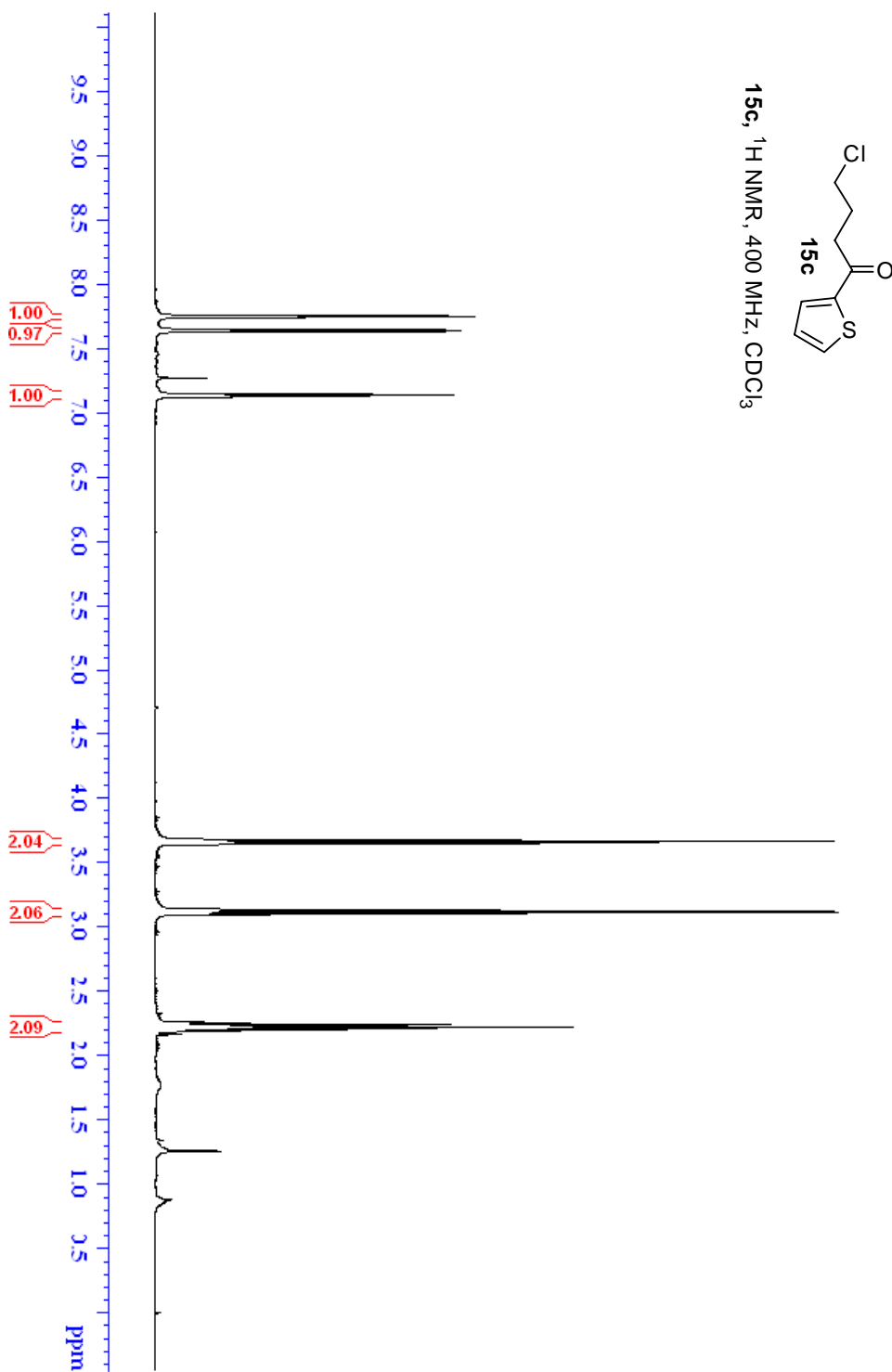
¹H NMR of 14c



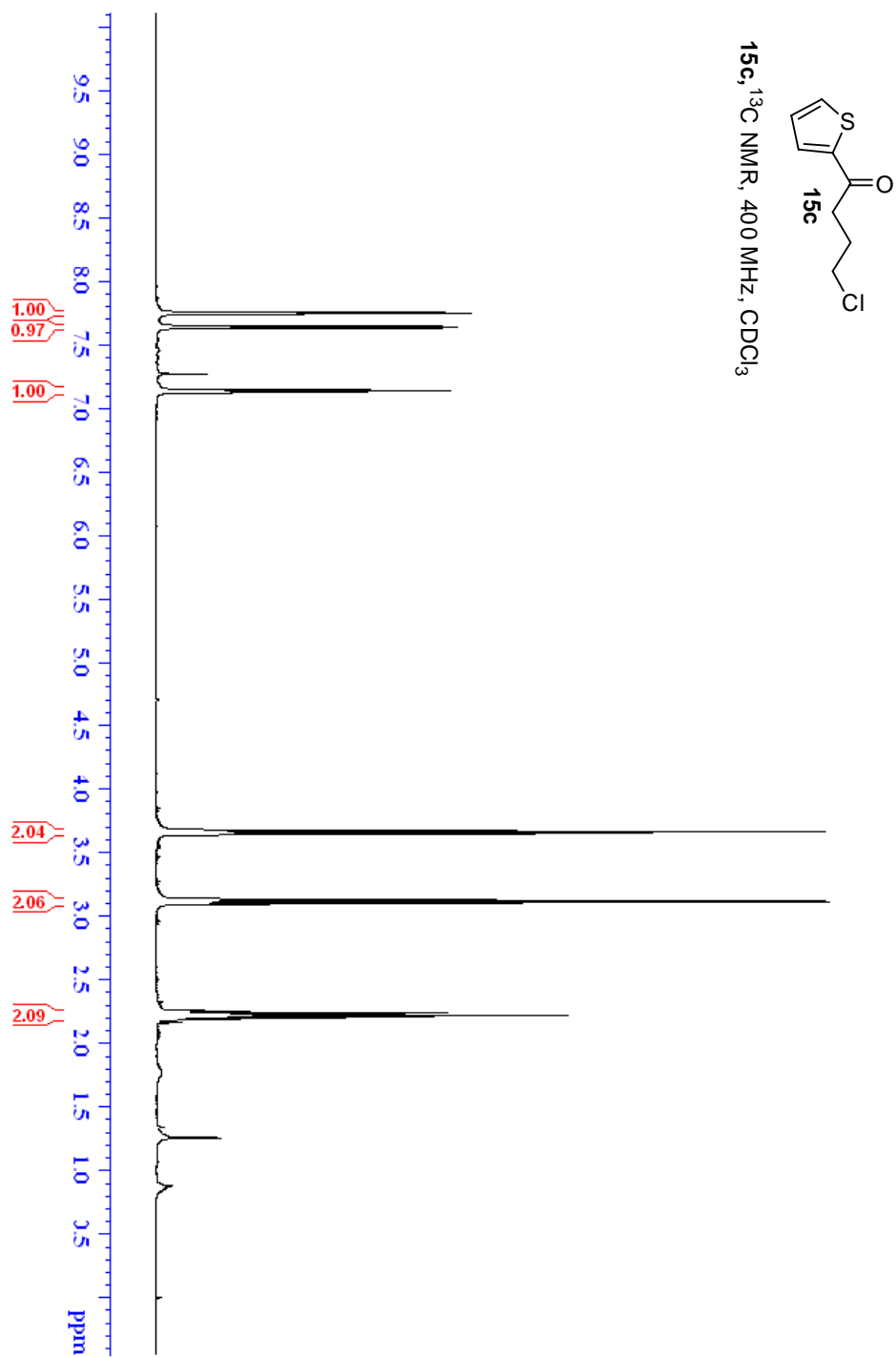
^{13}C NMR of 14c.



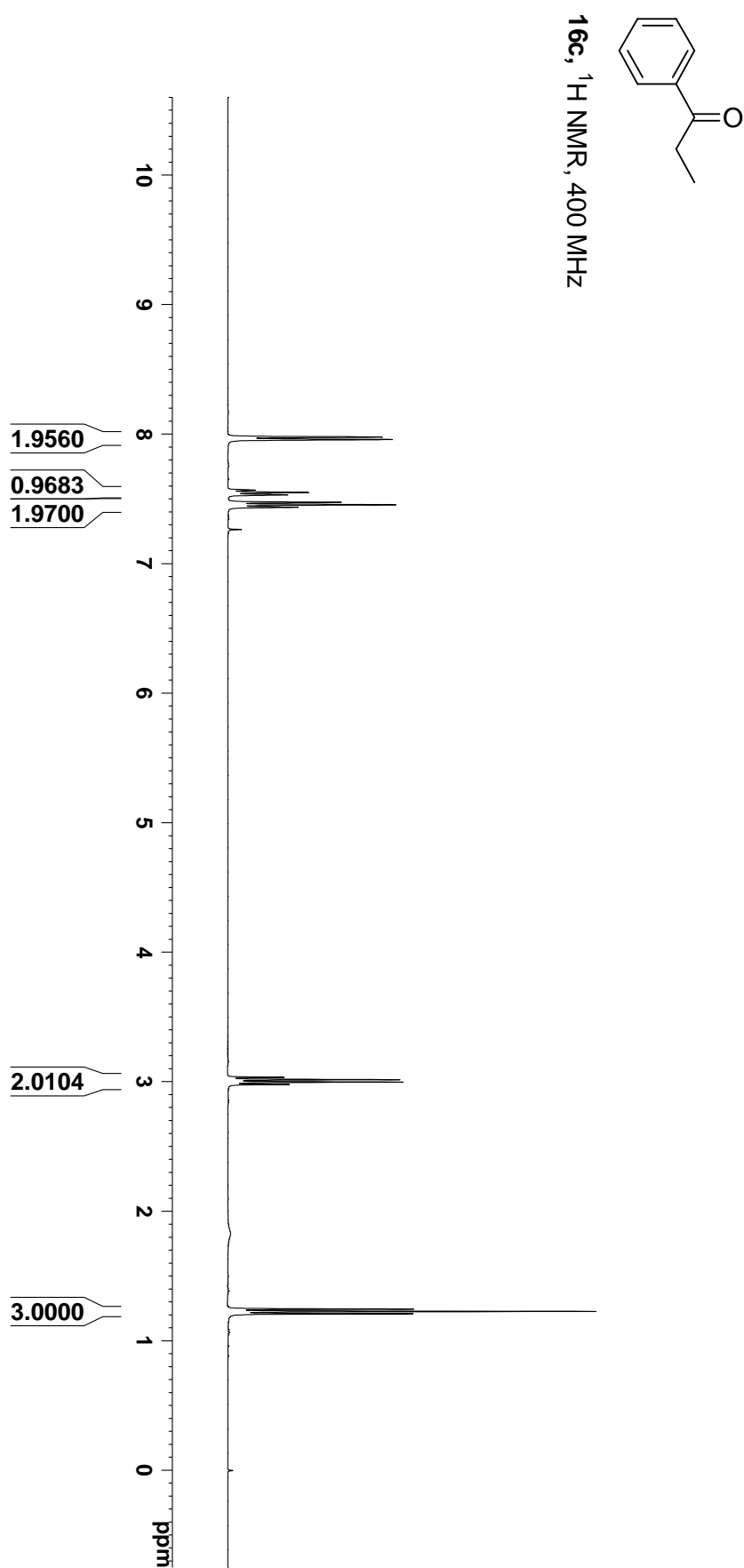
¹H NMR of 15c



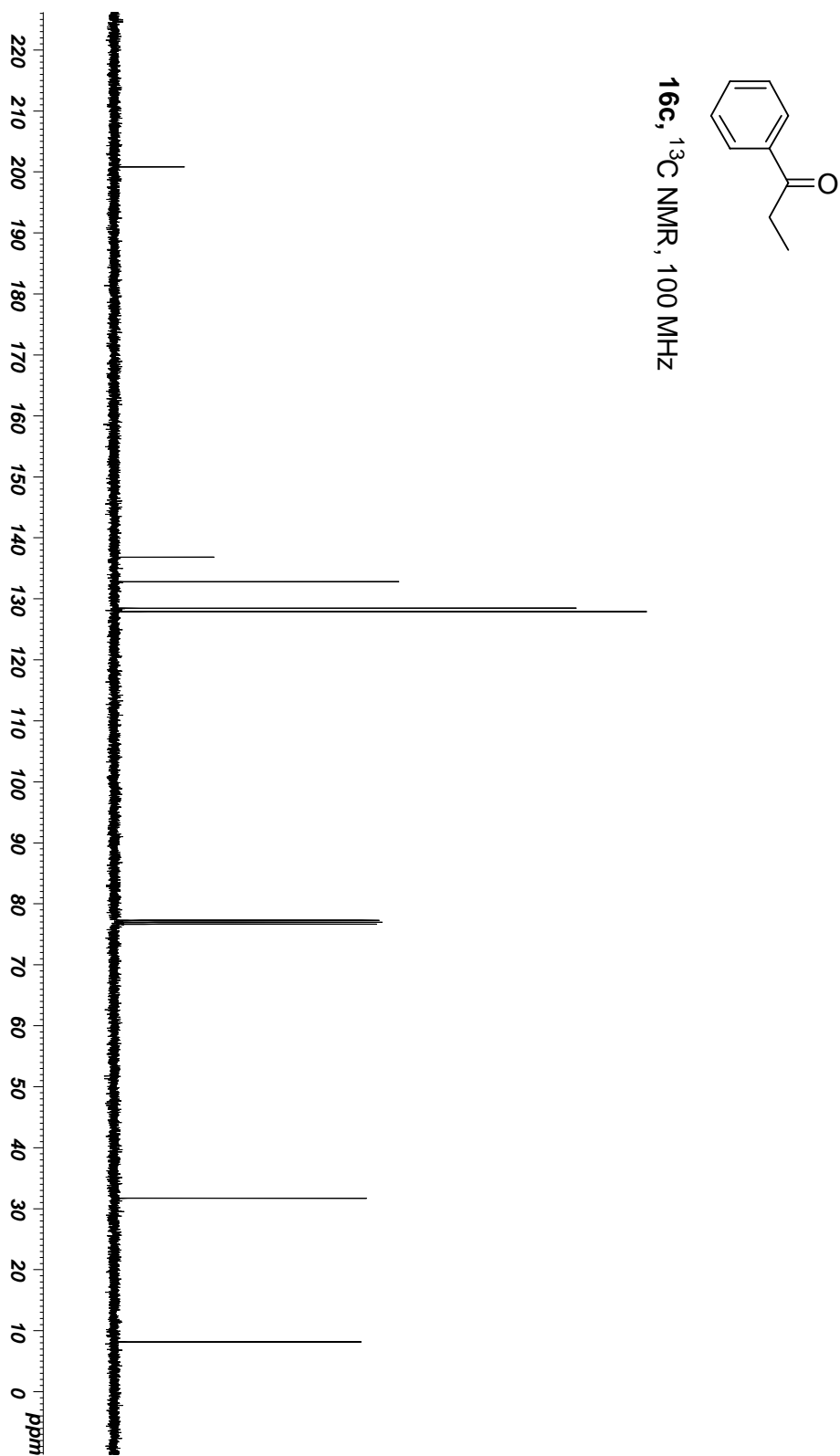
^{13}C NMR of 15c



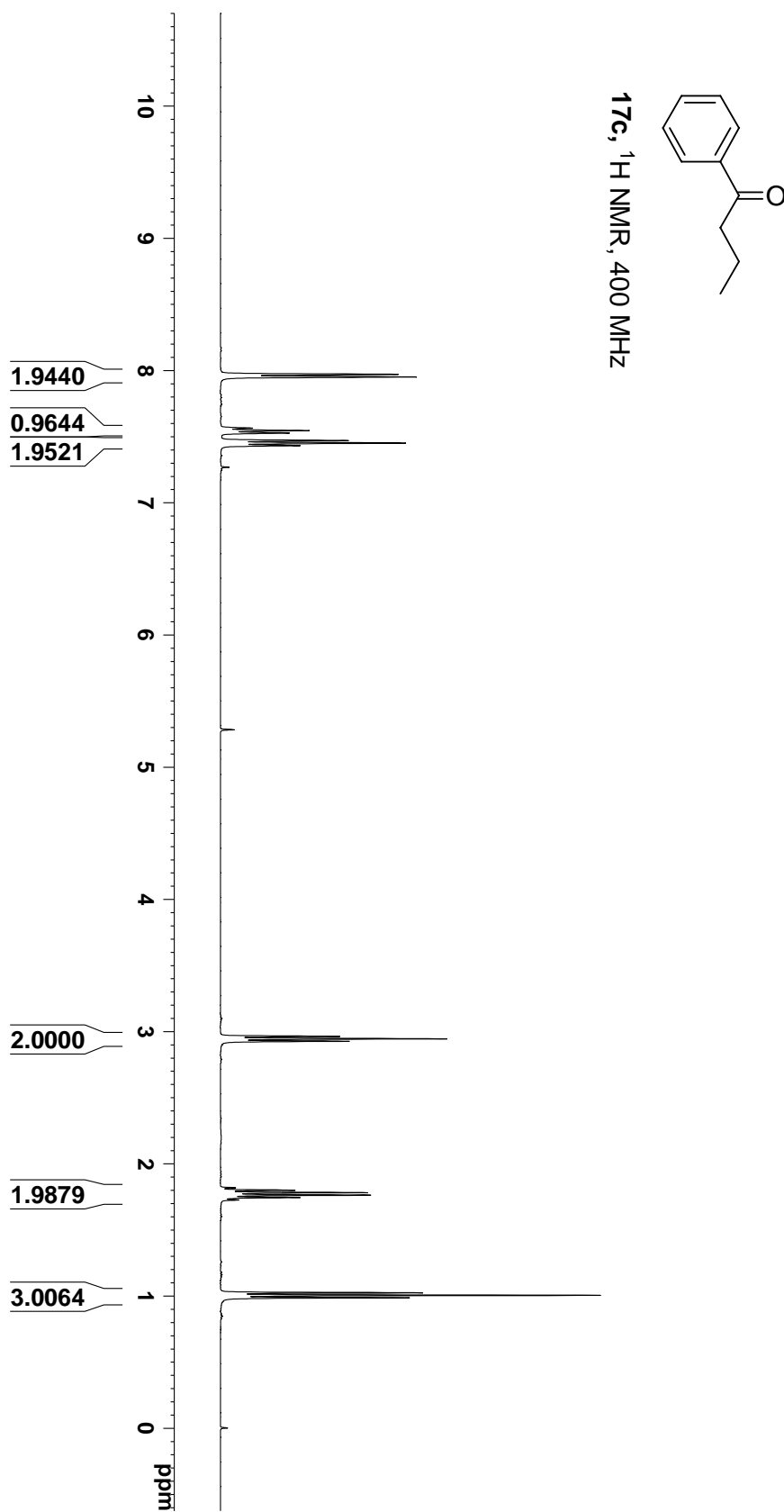
¹H NMR of 16c



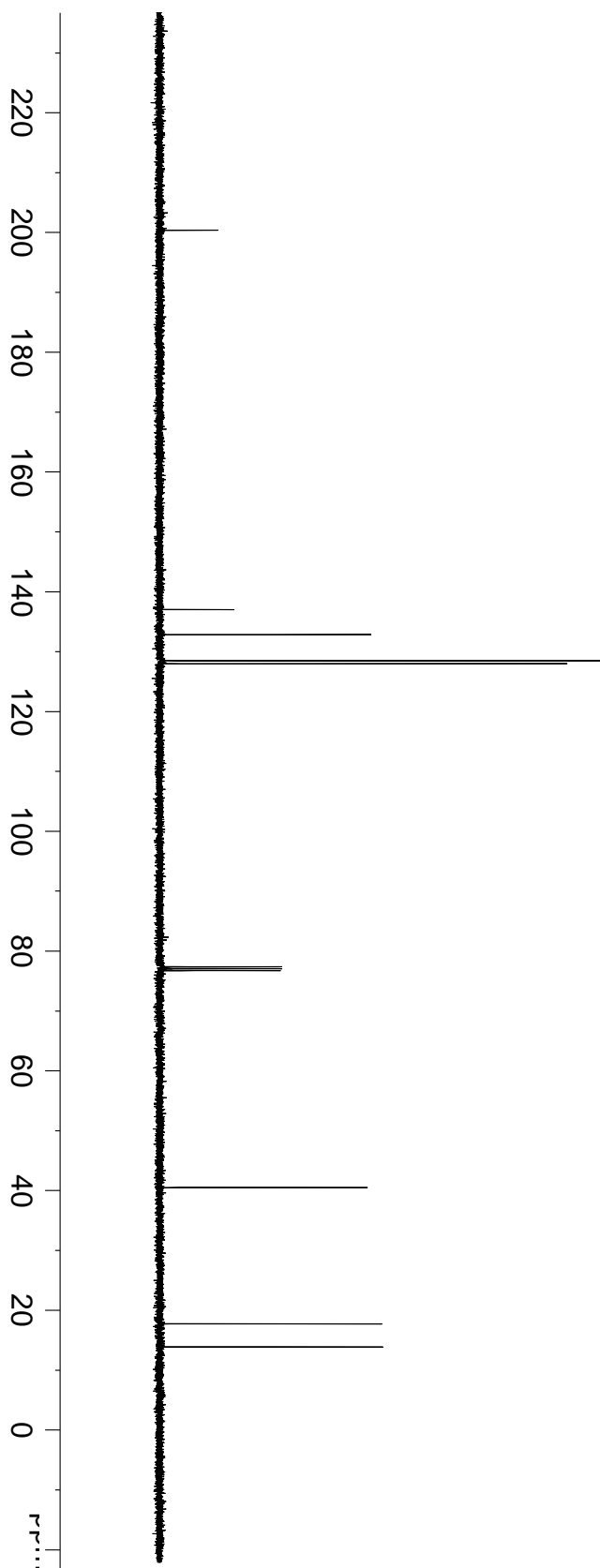
¹H NMR of 16c



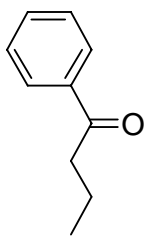
¹H NMR of 17c



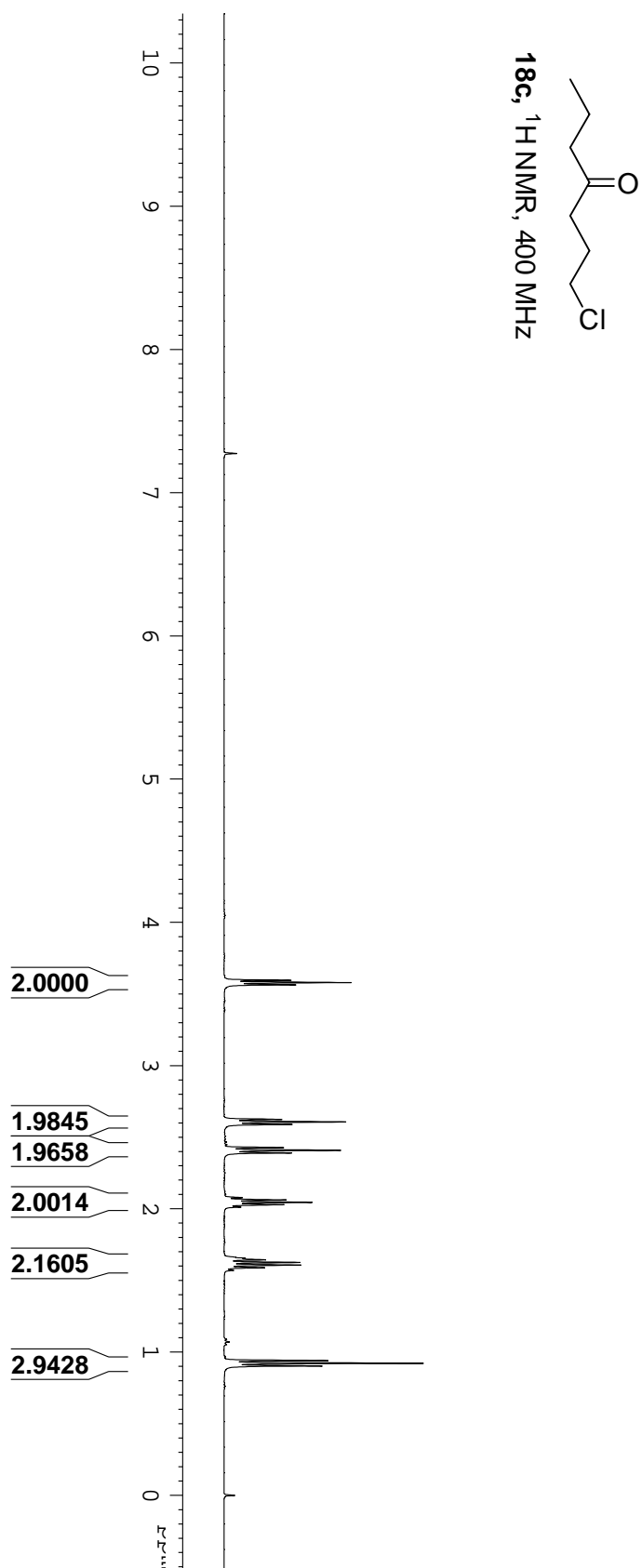
^{13}C NMR of 17c



17c, ^{13}C NMR, 100 MHz



¹H NMR of 18c



^{13}C NMR of 18c

