

[bmIm]OH-catalyzed amidation of azides and aldehydes: An efficient route to amides

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Supporting Information

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(A) Materials and equipment

Reagents were obtained commercially and used as received. Solvents were purified and dried by standard methods. Substrates **1** were prepared according the literature methods.¹ [bmIm]OH was prepared according to our previous reported method.² All title products were characterized by Infrared (IR), MS, ¹H NMR, ¹³C NMR and High Resolution mass spectrometer (HRMS). ¹H NMR spectra were recorded on 400 MHz in CDCl₃, and ¹³C NMR spectra were recorded on 100 MHz in CDCl₃ using tetramethylsilane (TMS) as an internal standard. Chemical shift values (δ) are given in ppm. Coupling constants (J) were measured in Hz. Mass spectra were obtained with ionization voltages of 70 eV. HRMS spectra were obtained by ESI on a TOF mass. 200-300 mesh silica gel was used for column chromatography.

(B) Experimental procedure

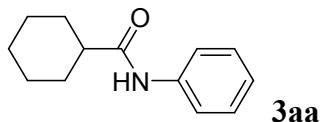
Typical Experimental Procedure for the Synthesis of compounds **3**:

To a Schlenk tube were added aryl azides **1** (0.3 mmol), aldehydes **2** (0.36 mmol), [bmIm]OH (10% mmol), DMSO (2 mL). Then the tube was charged with argon, and was stirred at 30 °C for about 5 h, then 10 mL saturated NH₄Cl was added. the reaction mixture was stirred at 25 °C for about 0.5 h. The reaction mixture was extracted with 40 mL ethyl acetate, The extract was washed with brine, dried (Na₂SO₄) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.

Experimental Procedure for the Synthesis of compounds **4**:

To a Schlenk tube were added aryl azides **1** (0.3 mmol), aldehydes **2** (0.36 mmol), [bmIm]OH (4 mmol). Then the tube was charged with argon, and was stirred at 25 °C for about 5 h. After the reaction was finished, the reaction mixture was extracted with 8 mL CDCl₃ to give the crude 1,2,3-triazolines **4**.

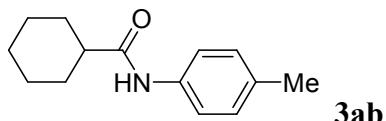
(C) Analytical data



N-Phenylcyclohexanecarboxamide (**3aa**):

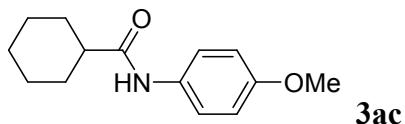
¹H NMR (400 MHz, CDCl₃) δ : 7.53 (d, J = 7.6 Hz, 2H), 7.32-7.21 (m, 3H), 7.10 (t, J = 7.4 Hz, 1H), 2.25 (t, J = 11.0 Hz, 1H), 1.96 (d, J = 12.4 Hz, 2H), 1.84 (d, J = 10.4 Hz, 2H), 1.71 (s, 1H), 1.58 (q, J = 11.2 Hz, 2H), 1.35-1.22 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 174.3, 138.0, 128.9, 124.0,

119.7, 46.6, 29.6, 25.6; LRMS (EI 70 ev) m/z (%): 203 (M^+ , 100); HRMS m/z (ESI) calcd for $C_{13}H_{18}NO$ ($M+H$)⁺ 204.1362, found 204.1368.



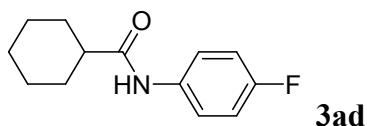
N-p-Tolylcyclohexanecarboxamide (3ab):

¹H NMR (400 MHz, CDCl₃) δ : 7.41 (d, J = 7.6 Hz, 2H), 7.15 (s, 1H), 7.11 (d, J = 8.0 Hz, 2H), 2.33-2.17 (m, 4H), 1.96 (d, J = 12.0 Hz, 2H), 1.84 (d, J = 11.2 Hz, 2H), 1.71 (s, 1H), 1.54-1.48 (m, 2H), 1.31-1.21 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 174.2, 135.4, 133.6, 129.4, 119.7, 46.5, 29.6, 25.7, 20.8; LRMS (EI 70 ev) m/z (%): 217 (M^+ , 100); HRMS m/z (ESI) calcd for $C_{14}H_{20}NO$ ($M+H$)⁺ 218.1519, found 218.1514.



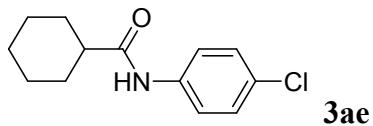
N-(4-Methoxyphenyl)cyclohexanecarboxamide (3ac):

¹H NMR (400 MHz, CDCl₃) δ : 7.43 (d, J = 8.4 Hz, 2H), 7.03 (s, 1H), 6.83 (d, J = 8.0 Hz, 2H), 3.81 (s, 1H), 2.22 (d, J = 13.0 Hz, 1H), 1.97 (d, J = 16.8 Hz, 2H), 1.85 (d, J = 11.2 Hz, 2H), 1.72 (s, 1H), 1.59-1.48 (m, 2H), 1.33-1.19 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 174.4, 156.8, 131.3, 121.8, 115.0, 55.7, 46.4, 29.2, 25.4; LRMS (EI 70 ev) m/z (%): 233 (M^+ , 100); HRMS m/z (ESI) calcd for $C_{14}H_{20}NO_2$ ($M+H$)⁺ 234.1468, found 234.1471.



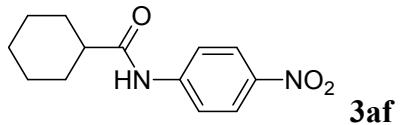
N-(4-Fluorophenyl)cyclohexanecarboxamide (3ad):

¹H NMR (400 MHz, CDCl₃) δ : 7.49 (dd, J = 4.8 Hz, J = 5.2 Hz, 2H), 7.21 (s, 1H), 7.01 (t, J = 8.6 Hz, 2H), 2.24 (d, J = 11.6 Hz, 1H), 1.96 (d, J = 12.4 Hz, 2H), 1.84 (d, J = 10.4 Hz, 2H), 1.71 (s, 1H), 1.63-1.48 (m, 2H), 1.34-1.22 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 174.3, 160.4 (d, J = 240 Hz), 134, 121.6 (d, J = 10 Hz), 115.7 (d, J = 20 Hz), 46.4, 29.7, 25.7; LRMS (EI 70 ev) m/z (%): 221 (M^+ , 100); HRMS m/z (ESI) calcd for $C_{13}H_{17}FNO$ ($M+H$)⁺ 222.1268, found 222.1276.



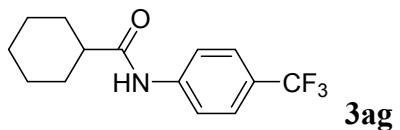
N-(4-chlorophenyl)cyclohexanecarboxamide (3ae):

¹H NMR (400 MHz, CDCl₃) δ: 7.49 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 8.4 Hz, 3H), 2.24 (t, *J* = 11.2 Hz, 1H), 1.95 (d, *J* = 12.8 Hz, 2H), 1.84 (d, *J* = 10.8 Hz, 2H), 1.71 (s, 1H), 1.57 (d, *J* = 12.4 Hz, *J* = 12.0 Hz, 2H), 1.34-1.92 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.4, 136.6, 129.1, 128.9, 120.9, 46.5, 29.6, 25.6; LRMS (EI 70 ev) *m/z* (%): 237 (M⁺, 67); HRMS *m/z* (ESI) calcd for C₁₃H₁₇ClNO (M+H)⁺ 238.0973, found 238.0979.



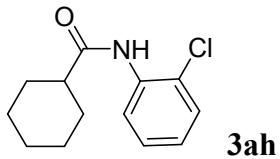
N-(4-Nitrophenyl)cyclohexanecarboxamide (3af):

¹H NMR (400 MHz, CDCl₃) δ: 8.21 (d, *J* = 8.4 Hz, 2H), 7.78 (d, *J* = 8.8 Hz, 2H), 7.55 (brs, 1H), 2.31 (t, *J* = 13.8 Hz, 1H), 2.02 (d, *J* = 10.0 Hz, 2H), 1.93 (d, *J* = 11.2 Hz, 2H), 1.84-1.71 (m, 1H), 1.57 (dd, *J* = 8.0 Hz, *J* = 8.8 Hz, 2H), 1.35-1.27 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.8, 144.0, 143.1, 125.0, 119.1, 46.6, 29.3, 25.4; LRMS (EI 70 ev) *m/z* (%): 248 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₃H₁₇N₂O₃ (M+H)⁺ 249.1146, found 249.1150.



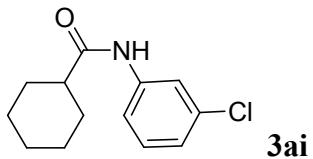
N-(4-(Trifluoromethyl)phenyl)cyclohexanecarboxamide (3ag):

¹H NMR (400 MHz, CDCl₃) δ: 8.11 (d, *J* = 7.6 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.49 (s, 1H), 2.33 (t, *J* = 13.2 Hz, 1H), 2.00 (d, *J* = 10.4 Hz, 2H), 1.81 (d, *J* = 12.0 Hz, 2H), 1.64 (s, 1H), 1.52-1.43 (m, 2H), 1.31-1.24 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 175.0, 148.4, 128.9, 126.7 (q, *J* = 3.7 Hz), 126.2, 123.5, 120.9, 120.4 (q, *J* = 32.4 Hz), 114.1, 46.2, 28.9, 25.8; LRMS (EI 70 ev) *m/z* (%): 271 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₄H₁₇F₃NO (M+H)⁺ 272.1236, found 272.1245.



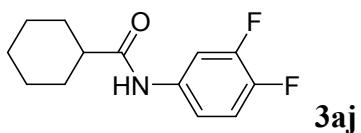
N-(2-Chlorophenyl)cyclohexanecarboxamide (3ah):

¹H NMR (400 MHz, CDCl₃) δ: 8.41 (d, *J* = 8.4 Hz, 1H), 7.76 (s, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 2.35 (t, *J* = 11.6 Hz, 1H), 2.03 (d, *J* = 11.2 Hz, 2H), 1.87 (d, *J* = 12.0 Hz, 2H), 1.74 (d, *J* = 11.2 Hz, 1H), 1.59 (q, *J* = 12.0 Hz, 2H), 1.47-1.36 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.2, 134.5, 128.7, 127.5, 124.3, 122.5, 121.4, 46.4, 29.5, 25.53, 25.50; LRMS (EI 70 ev) *m/z* (%): 237 (M⁺, 63); HRMS *m/z* (ESI) calcd for C₁₃H₁₇ClNO (M+H)⁺ 238.0973, found 238.0981.



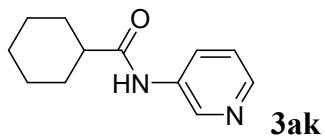
N-(3-Chlorophenyl)cyclohexanecarboxamide (3ai):

¹H NMR (400 MHz, CDCl₃) δ: 7.51 (s, 1H), 7.42 (dd, *J* = 4.0 Hz, *J* = 4.4 Hz, 2H), 7.28 (t, *J* = 2.4 Hz, 2H), 2.28 (t, *J* = 10.0 Hz, 1H), 1.96 (d, *J* = 12.8 Hz, 2H), 1.84 (d, *J* = 12.4 Hz, 2H), 1.74 (d, *J* = 7.6 Hz, 1H), 1.54-1.49 (m, 2H), 1.32-1.23 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.8, 133.6, 130.4, 128.1, 124.2, 119.1, 118.0, 46.7, 29.5, 25.3; LRMS (EI 70 ev) *m/z* (%): 237 (M⁺, 63); HRMS *m/z* (ESI) calcd for C₁₃H₁₇ClNO (M+H)⁺ 238.0973, found 238.0977.



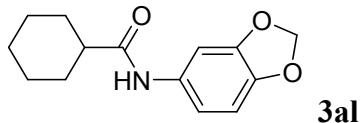
N-(3,4-Difluorophenyl)cyclohexanecarboxamide (3aj):

¹H NMR (400 MHz, CDCl₃) δ: 7.49 (dd, *J* = 0.8 Hz, *J* = 2.4 Hz, 1H), 7.41-7.38 (m, 1H), 7.27-7.22 (m, 2H), 2.31 (t, *J* = 13.2 Hz, 1H), 2.00 (d, *J* = 13.0 Hz, 2H), 1.86 (d, *J* = 9.6 Hz, 2H), 1.72 (d, *J* = 3.2 Hz, 1H), 1.57-1.51 (m, 2H), 1.34-1.24 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.8, 151.8, 151.7, 149.4, 149.3, 144.8, 144.7, 143.5, 143.4, 142.4, 142.3, 117.56, 117.55, 117.38, 117.37, 110.32, 110.29, 110.27, 110.24, 103.9, 103.7, 46.9, 29.7, 25.5; LRMS (EI 70 ev) *m/z* (%): 239 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₃H₁₆F₂NO (M+H)⁺ 240.1174, found 240.1181.



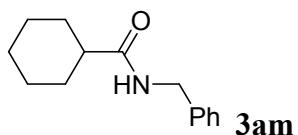
N-(Pyridin-3-yl)cyclohexanecarboxamide (3ak):

¹H NMR (400 MHz, CDCl₃) δ: 8.59 (s, 1H), 8.29 (s, 1H), 8.13 (d, *J* = 6.4 Hz, 1H), 7.77 (s, 1H), 7.29 (d, *J* = 4.8 Hz, 1H), 2.33-2.26 (m, 1H), 1.97 (d, *J* = 12.8 Hz, 2H), 1.87 (d, *J* = 10.4 Hz, 2H), 1.73 (d, *J* = 12.4 Hz, 1H), 1.57-1.50 (m, 2H), 1.34-1.24 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 175.0, 144.4, 140.7, 136.1, 126.3, 123.7, 46.7, 29.6, 25.66, 25.62; LRMS (EI 70 ev) *m/z* (%): 204 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₂H₁₇N₂O (M+H)⁺ 205.1247, found 205.1255.



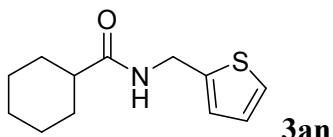
N-(Benzo[d][1,3]dioxol-5-yl)cyclohexanecarboxamide (3al):

¹H NMR (400 MHz, CDCl₃) δ: 7.79 (d, *J* = 8.4 Hz, 1H), 7.59 (s, 1H), 7.21 (s, 1H), 6.93 (d, *J* = 8.4 Hz, 1H), 3.95 (s, 2H), 2.32-2.24 (m, 1H), 2.04 (d, *J* = 10.8 Hz, 2H), 1.73 (d, *J* = 8.4 Hz, 2H), 1.57 (s, 1H), 1.53-1.48 (m, 2H), 1.38-1.24 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 171.8, 153.6, 148.6, 124.6, 121.6, 112.1, 110.2, 56.1, 46.7, 30.2, 25.8; LRMS (EI 70 ev) *m/z* (%): 247 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₄H₁₈NO₃ (M+H)⁺ 248.1261, found 248.1269.



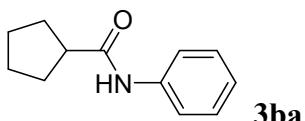
N-Benzylcyclohexanecarboxamide (3am):

¹H NMR (400 MHz, CDCl₃) δ: 7.31 (t, *J* = 7.0 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 3H), 6.16 (s, 1H), 4.38 (d, *J* = 5.2 Hz, 2H), 2.14 (t, *J* = 11.8 Hz, 1H), 1.86 (d, *J* = 12.8 Hz, 2H), 1.77 (d, *J* = 9.6 Hz, 2H), 1.65 (s, 1H), 1.47 (dd, *J* = 7.6 Hz, *J* = 12.0 Hz, 2H), 1.27-1.15 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 176.1, 138.4, 128.4, 127.5, 127.2, 45.3, 43.1, 29.5, 25.5; LRMS (EI 70 ev) *m/z* (%): 217 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₄H₂₀NO (M+H)⁺ 218.1519, found 218.1523.

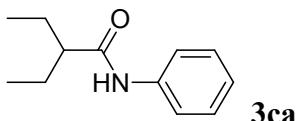


N-((Thiophen-2-yl)methyl)cyclohexanecarboxamide (3an):

¹H NMR (400 MHz, CDCl₃) δ: 7.79 (d, *J* = 8.4 Hz, 1H), 7.59 (s, 1H), 6.93 (d, *J* = 8.4 Hz, 1H), 6.23 (s, 1H), 3.95 (s, 2H), 2.22 (d, *J* = 12.8 Hz, 1H), 1.94 (d, *J* = 8.4 Hz, 2H), 1.85 (d, *J* = 10.0 Hz, 2H), 1.73 (s, 1H), 1.55 (d, *J* = 7.6 Hz, *J* = 12.4 Hz, 2H), 1.34-1.23 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 171.7, 146.5, 126.9, 125.8, 125.1, 45.4, 38.2, 29.6, 25.7; LRMS (EI 70 ev) *m/z* (%): 223 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₂H₁₈NOS (M+H)⁺ 224.1083, found 224.1090.

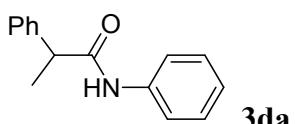


N-Phenylcyclopentanecarboxamide (3ba): ¹H NMR (400 MHz, CDCl₃) δ: 7.55 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 7.8 Hz, 2H), 7.14 (s, 1H), 7.11 (d, *J* = 7.0 Hz, 1H), 2.57-2.51 (m, 1H), 1.95-1.89 (m, 4H), 1.78 (t, *J* = 3.8 Hz, 2H), 1.64-1.61 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.8, 138.6, 129.7, 124.4, 120.1, 47.2, 30.3, 26.3; LRMS (EI 70 ev) *m/z* (%): 189 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₂H₁₆NO (M+H)⁺ 190.1205, found 190.1213.



2-Ethyl-N-phenylbutanamide (3ca):

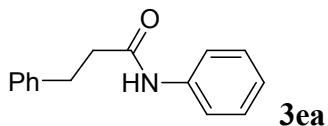
¹H NMR (400 MHz, CDCl₃) δ: 7.56 (d, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.17 (s, 1H), 7.12 (t, *J* = 7.2 Hz, 1H), 2.02 (d, *J* = 4.4 Hz, 1H), 1.76-1.66 (m, 2H), 1.59-1.53 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ: 174.2, 137.8, 128.9, 124.1, 119.7, 52.5, 25.8, 12.1; LRMS (EI 70 ev) *m/z* (%): 191 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₂H₁₈NO (M+H)⁺ 192.1361, found 192.1356.



N,2-Diphenylpropanamide (3da):

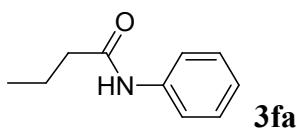
¹H NMR (400 MHz, CDCl₃) δ: 7.44-7.33 (m, 6H), 7.32-7.27 (m, 1H), 7.25 (d, *J* = 4.0 Hz, 1H), 7.08 (s, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 3.76 (q, *J* = 5.6 Hz, 1H), 1.48 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 172.3, 140.8, 137.9, 129.4, 129.1, 127.8, 127.7, 124.2, 119.6, 48.1, 18.6; LRMS (EI 70 ev) *m/z* (%): 225 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₅H₁₆NO (M+H)⁺ 226.1293, found

226.1297.



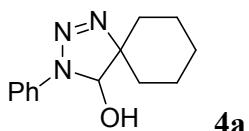
N,3-Diphenylpropanamide (3ea):

¹H NMR (400 MHz, CDCl₃) δ: 7.46 (d, *J* = 8.0 Hz, 2H), 7.32 (s, 4H), 7.23 (s, 3H), 7.13 (t, *J* = 5.6 Hz, 2H), 3.10 (t, *J* = 7.4 Hz, 2H), 2.70 (t, *J* = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.3, 140.5, 137.6, 128.9, 128.6, 128.3, 126.3, 124.2, 119.8, 39.5, 31.5; LRMS (EI 70 ev) *m/z* (%): 225 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₅H₁₆NO (M+H)⁺ 226.1293, found 226.1299.



N-Phenylbutyramide (3fa):

¹H NMR (400 MHz, CDCl₃) δ: 7.53 (t, *J* = 10.0 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.11 (t, *J* = 7.2 Hz, 1H), 2.34 (t, *J* = 7.4 Hz, 2H), 1.79-1.72 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 171.4, 137.9, 128.9, 124.1, 119.7, 39.5, 19.0, 13.7; LRMS (EI 70 ev) *m/z* (%): 163 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₀H₁₄NO (M+H)⁺ 164.1047, found 164.1041.



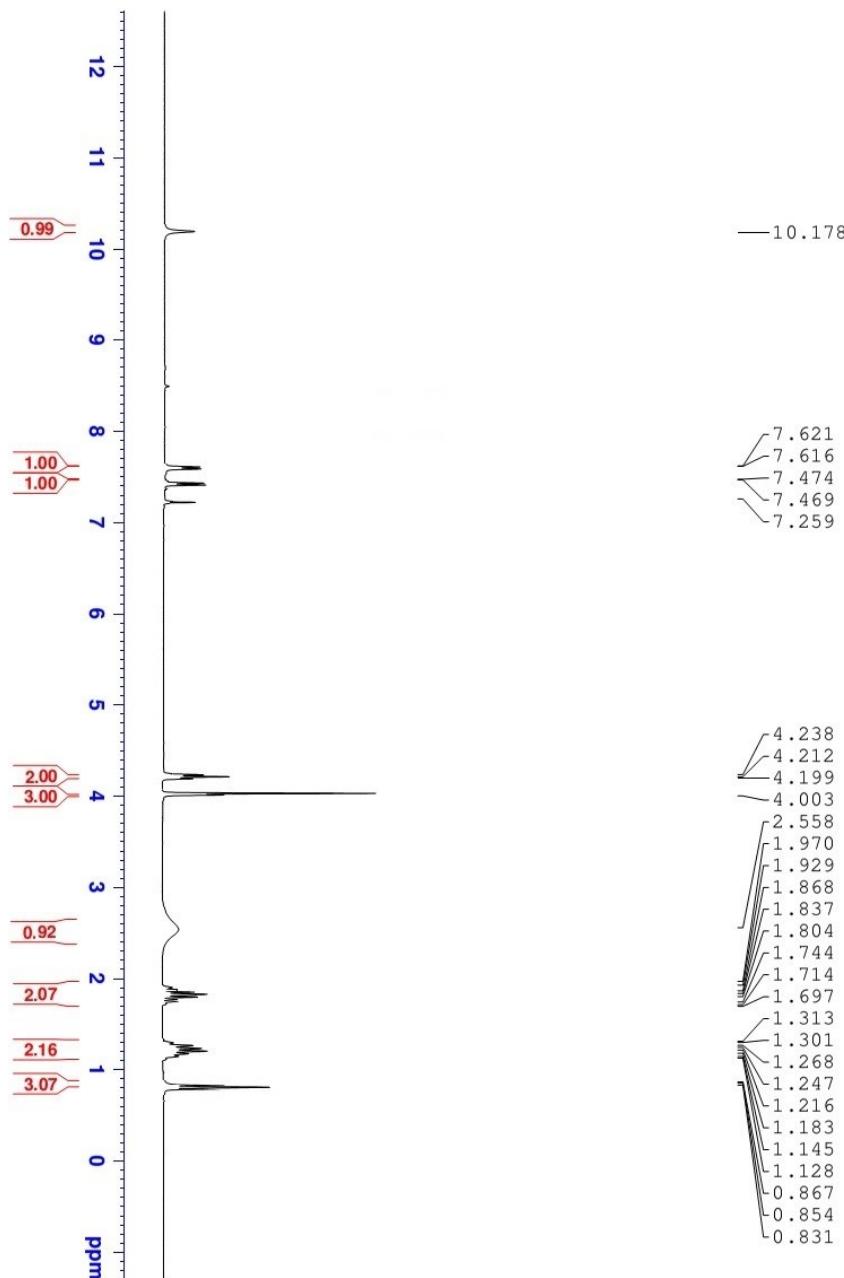
1,2,3-triazolines (4):

¹H NMR (400 MHz, CDCl₃) δ: 7.45 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.80 Hz, 2H), 6.99 (t, *J* = 7.6 Hz, 1H), 5.28 (s, 1H), 4.97 (brs, 1H), 1.86-1.79 (m, 4H), 1.59-1.49 (m, 4H), 1.29-1.19 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 140.6, 129.2, 121.9, 115.8, 82.8, 82.1, 32.0, 27.4, 25.3, 22.9, 22.3.

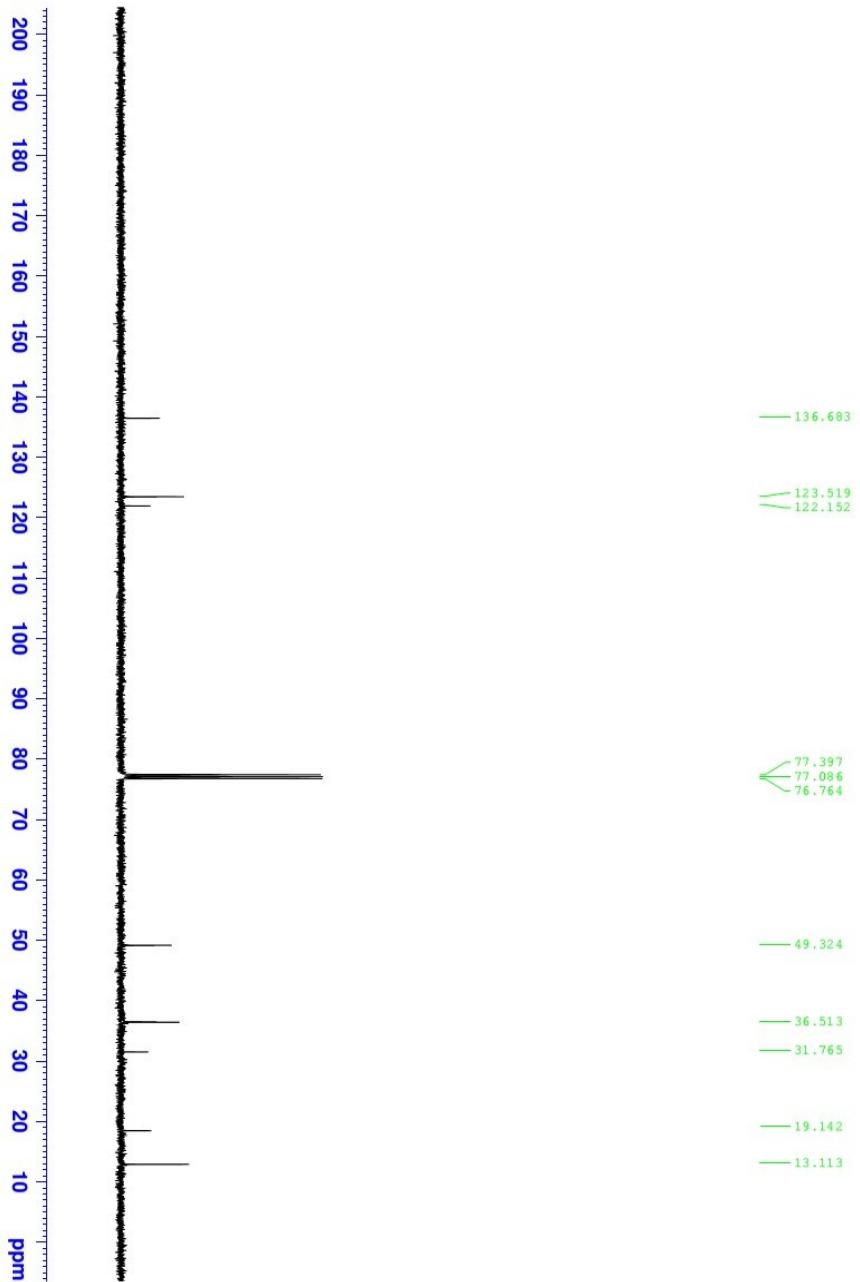
(D) References

1. Keana, J. F. W.; Cai, S. X., *J. Org. Chem.* 1990, 55, 3640
2. X. Li, C. Jin and L. Gu, *J. Org. Chem.* 2005, 80, 2443

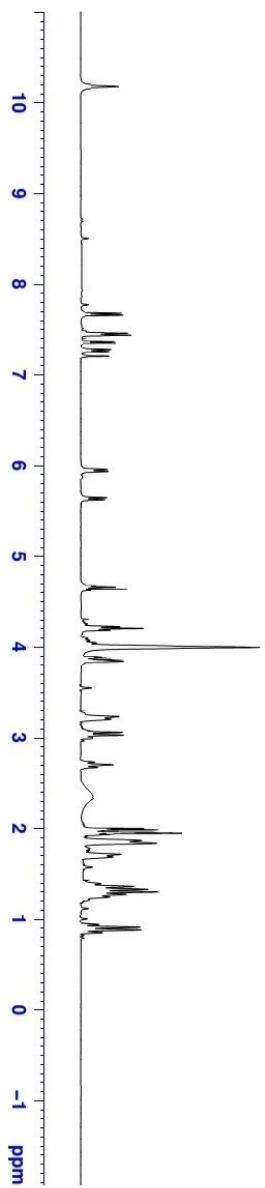
(E) Spectra



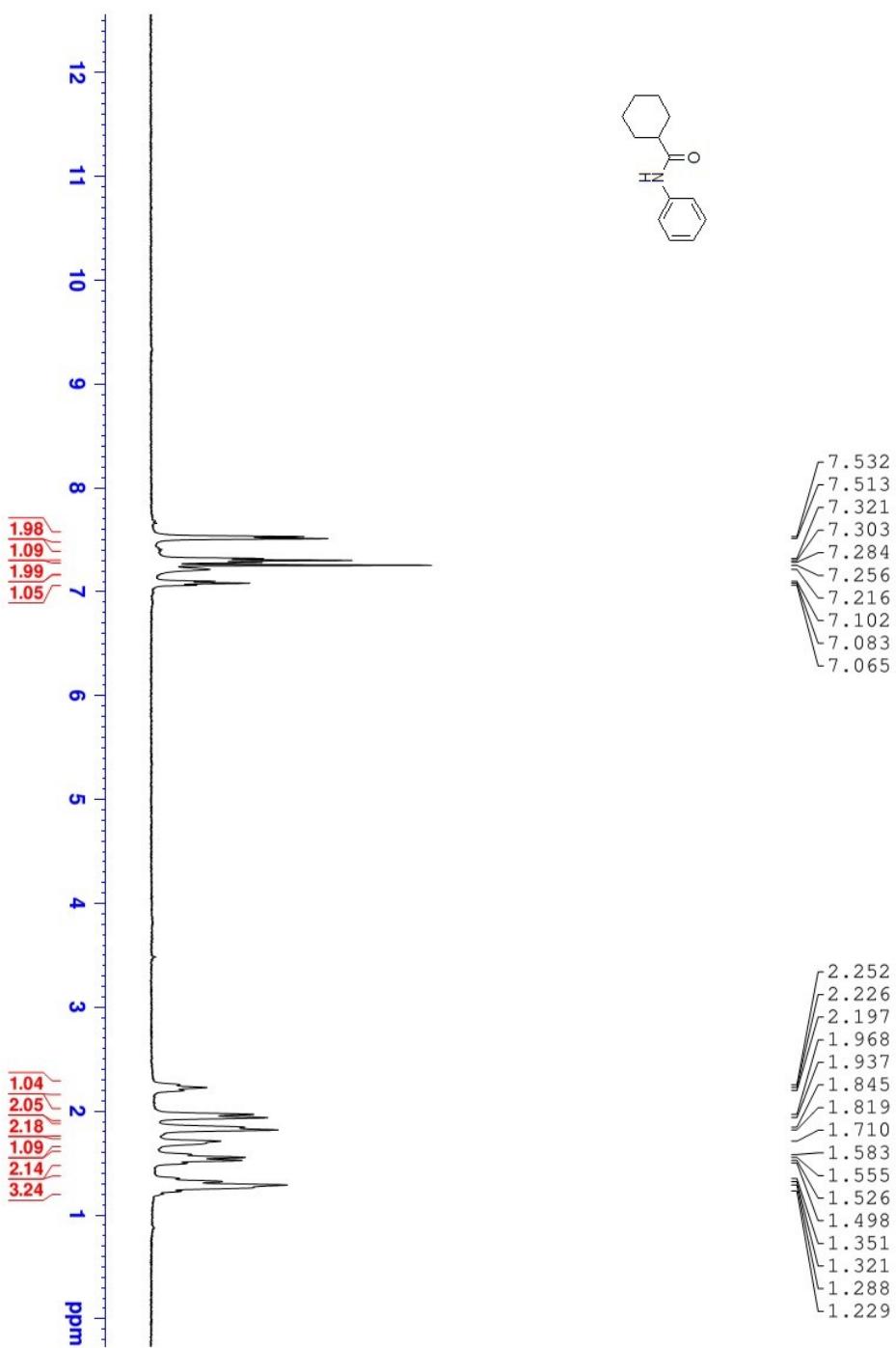
¹H NMR of pure [bmim][OH]



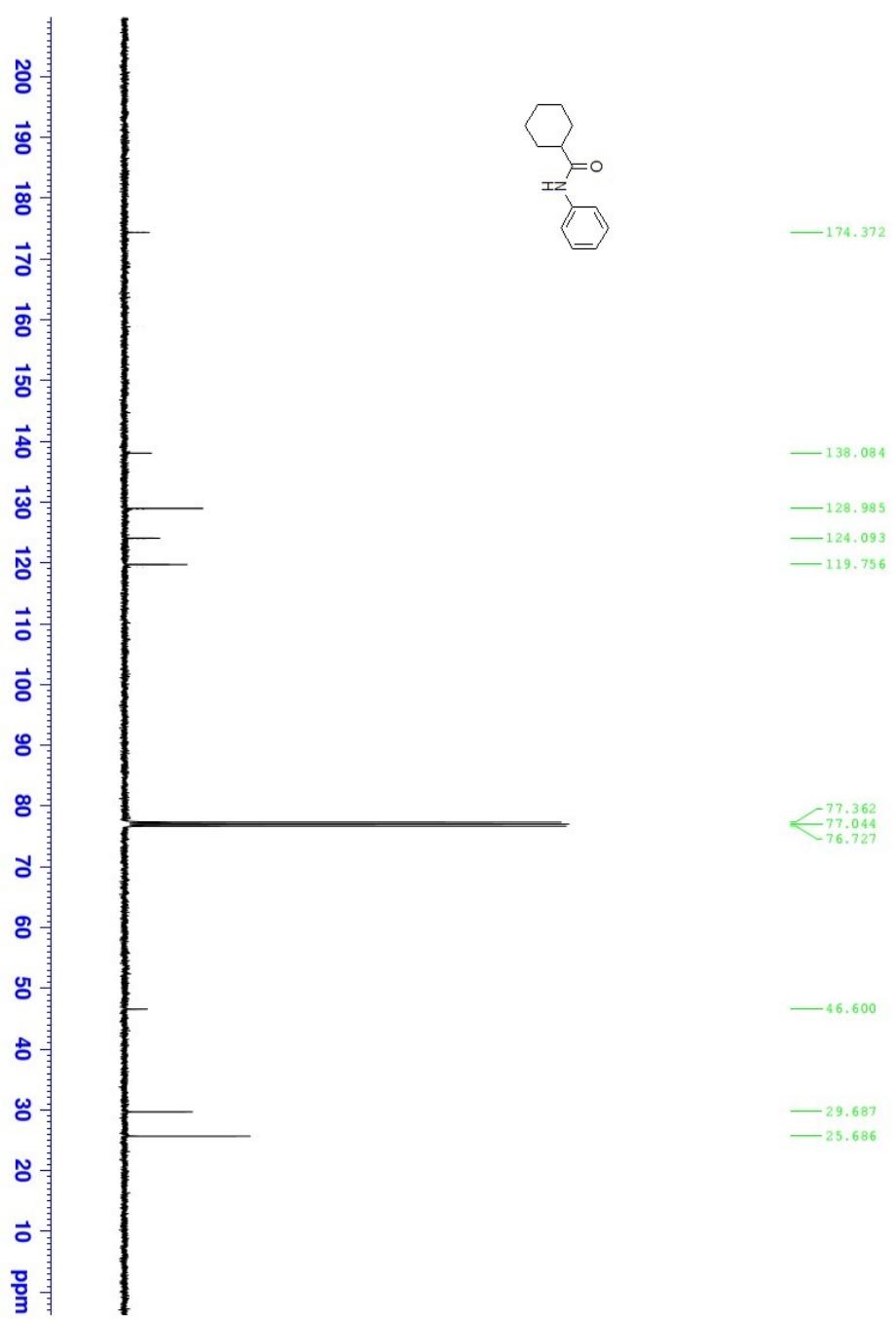
^{13}C NMR of pure [bmim][OH]



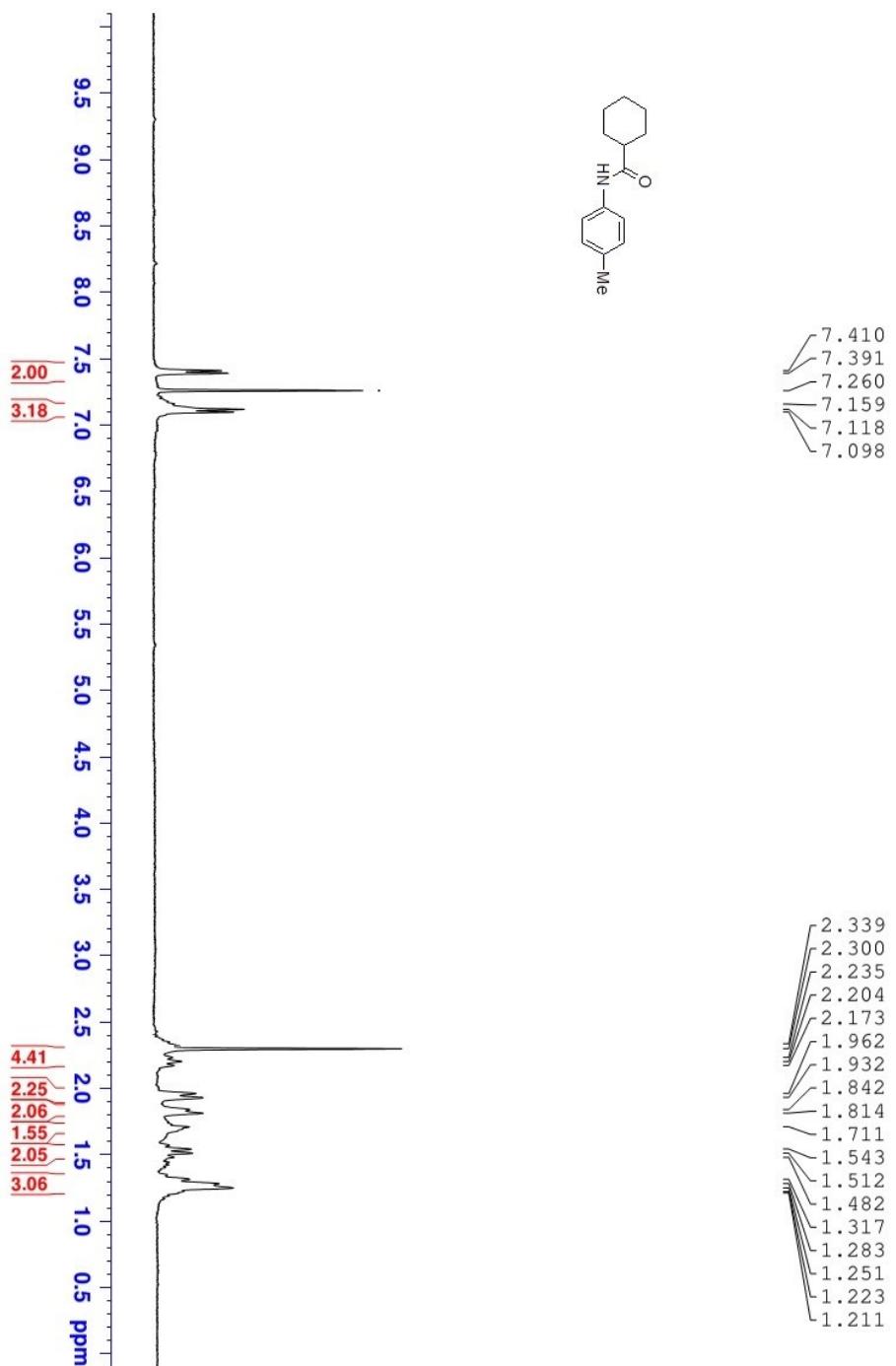
¹H NMR of [bmim][OH] after 5th cycle of reaction



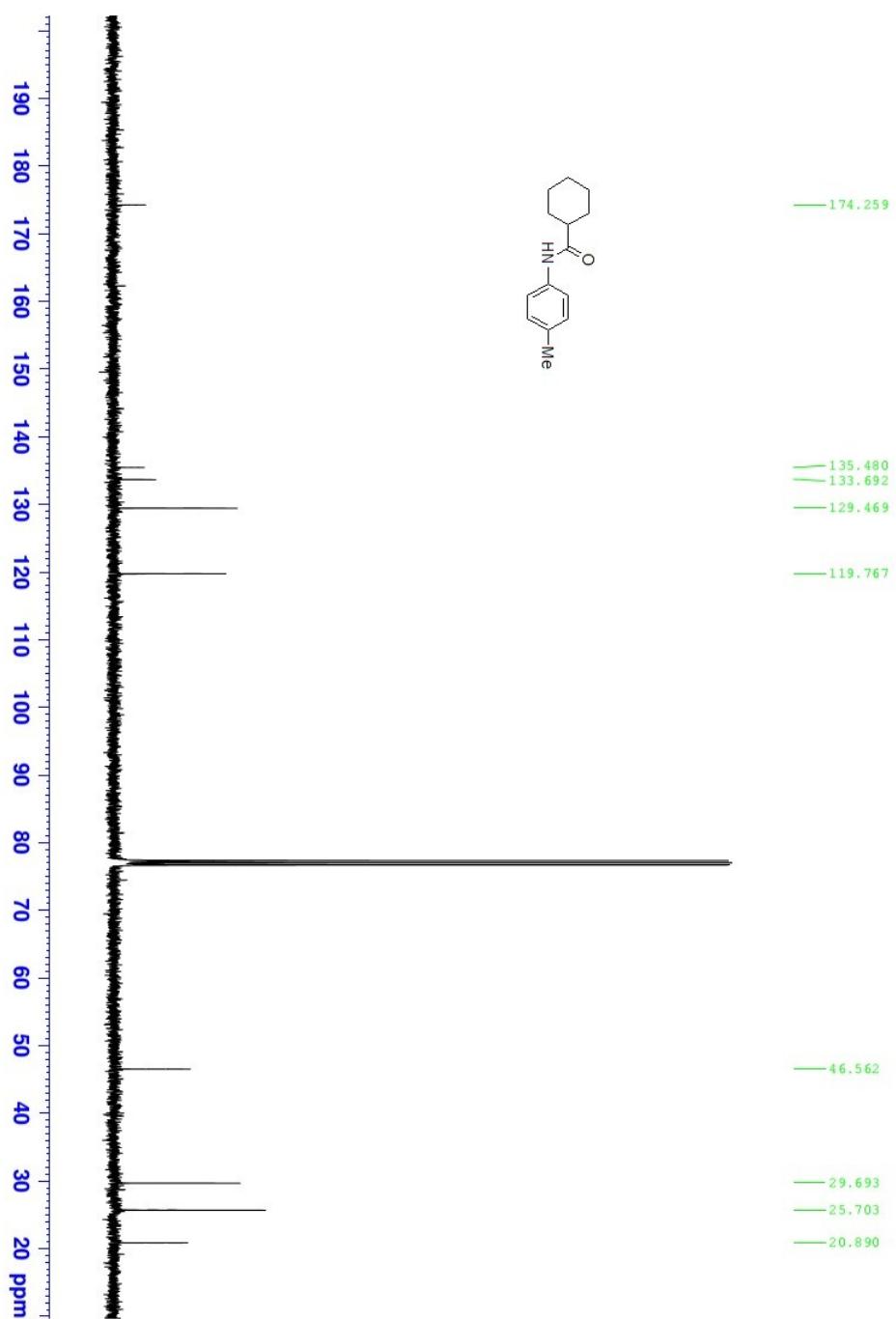
¹H NMR of Compound 3aa



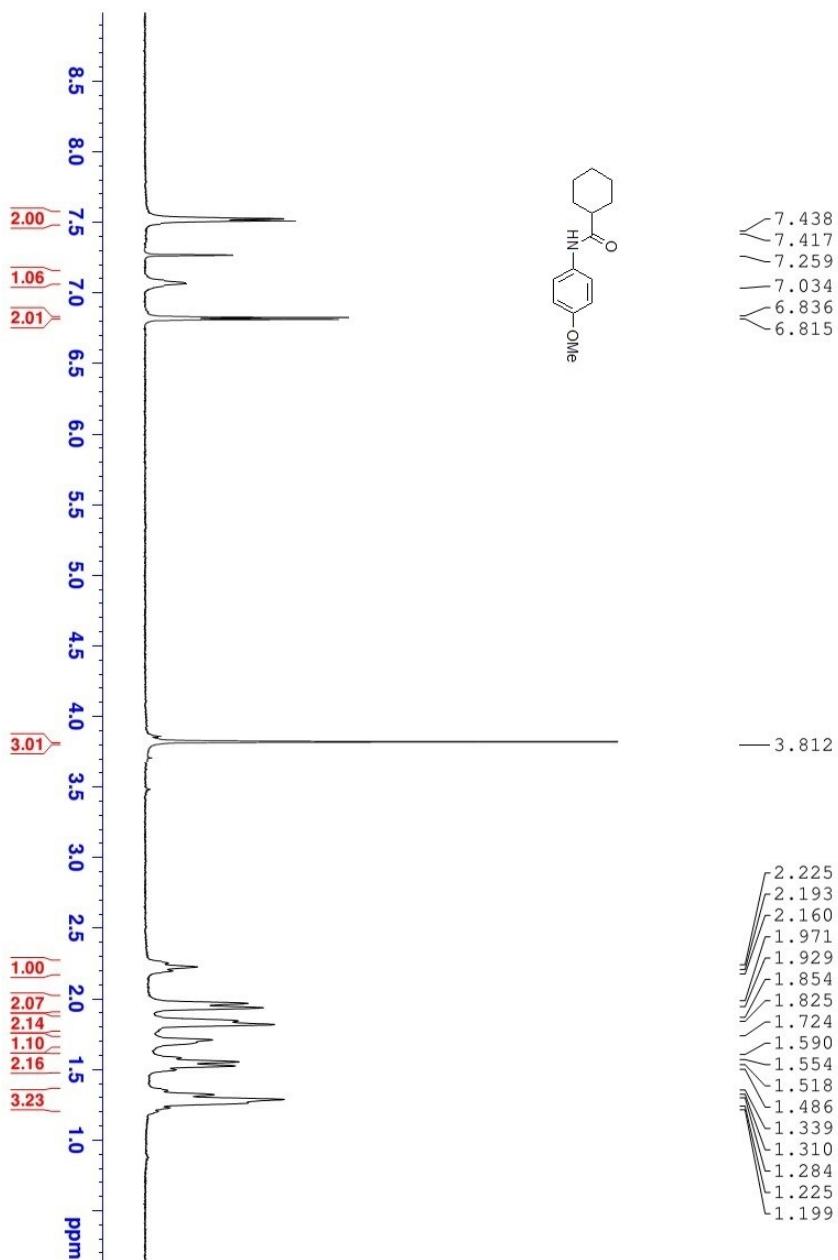
¹³C NMR of Compound 3aa



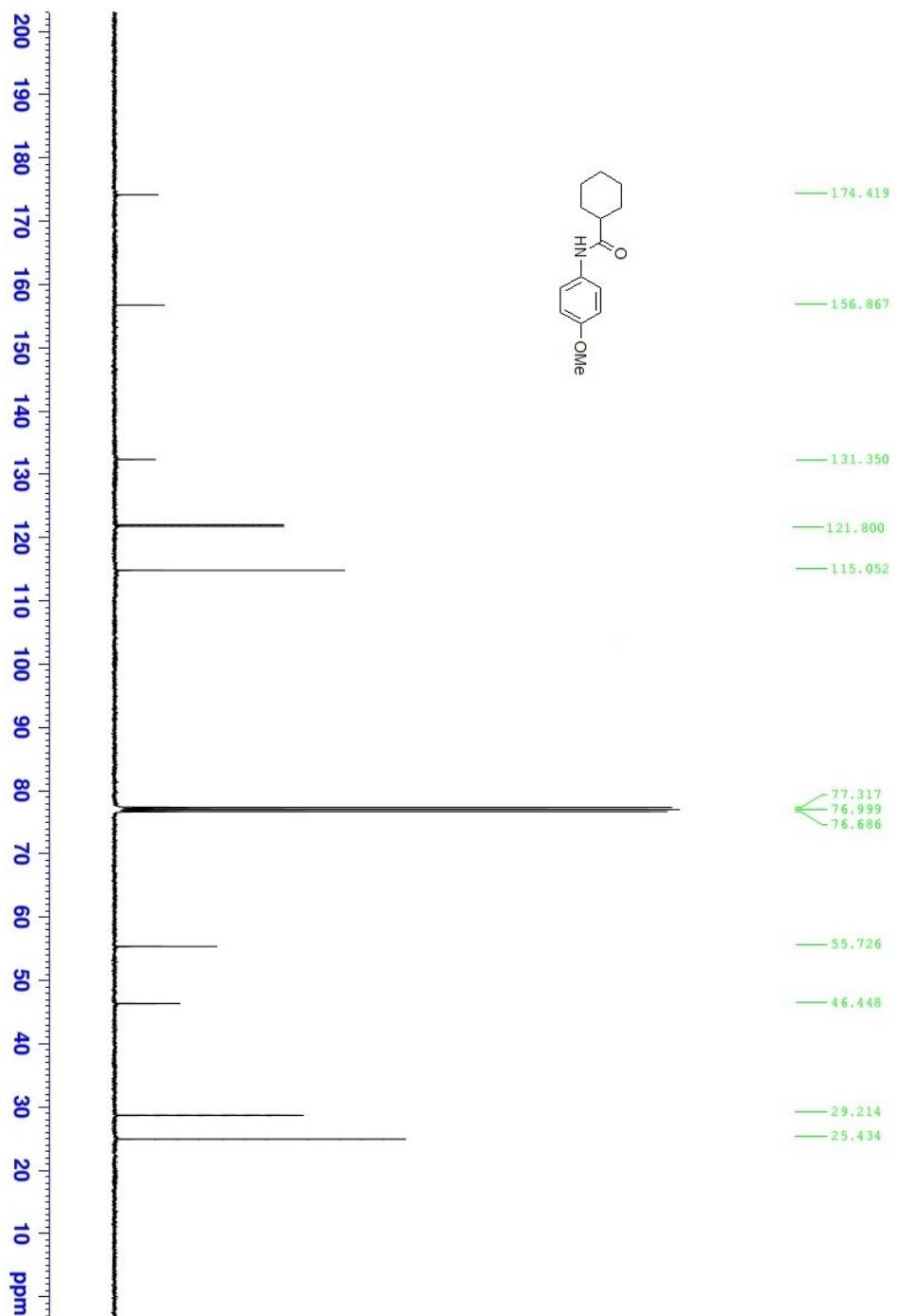
¹H NMR of Compound 3ab



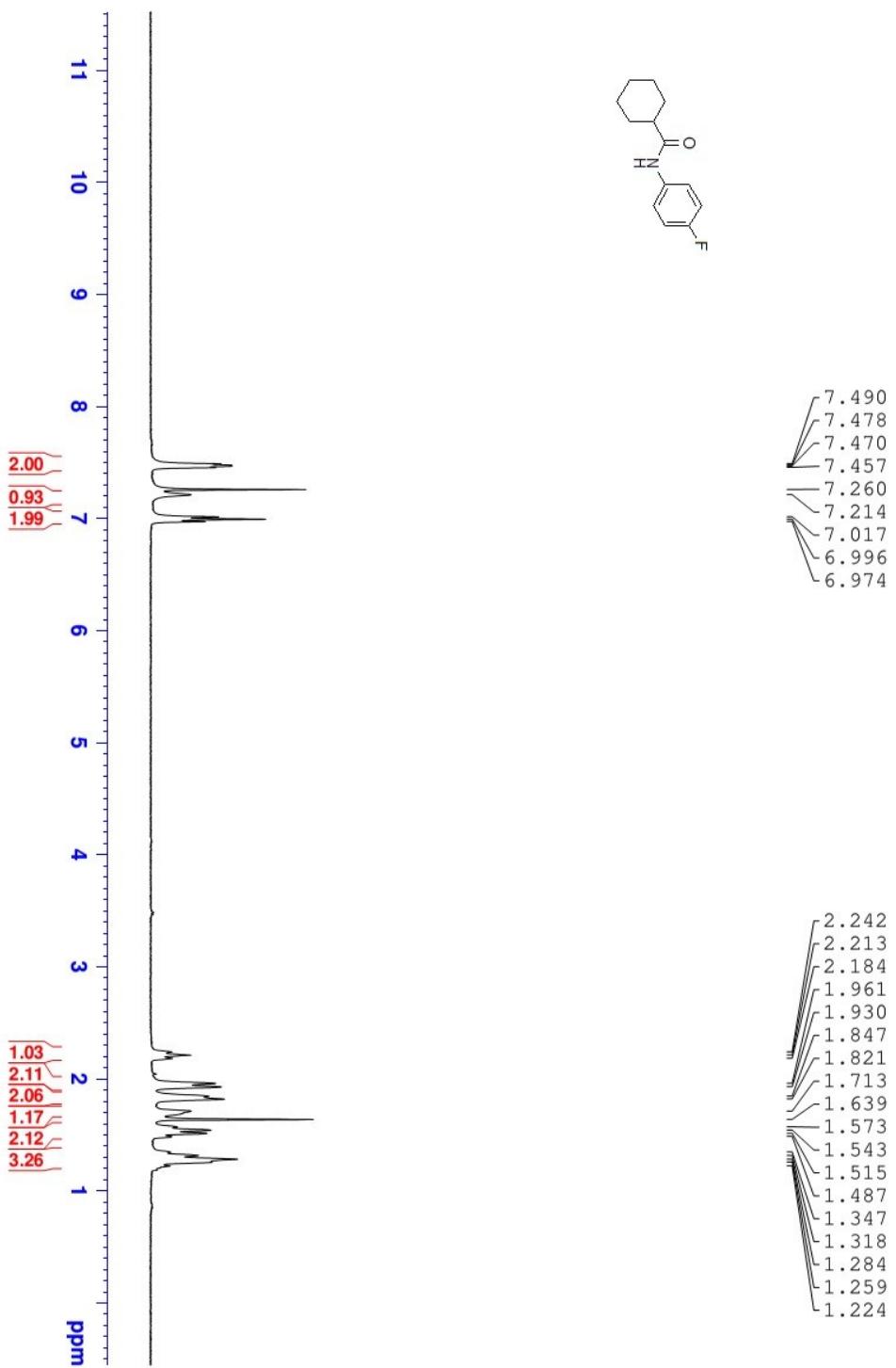
^{13}C NMR of Compound 3ab



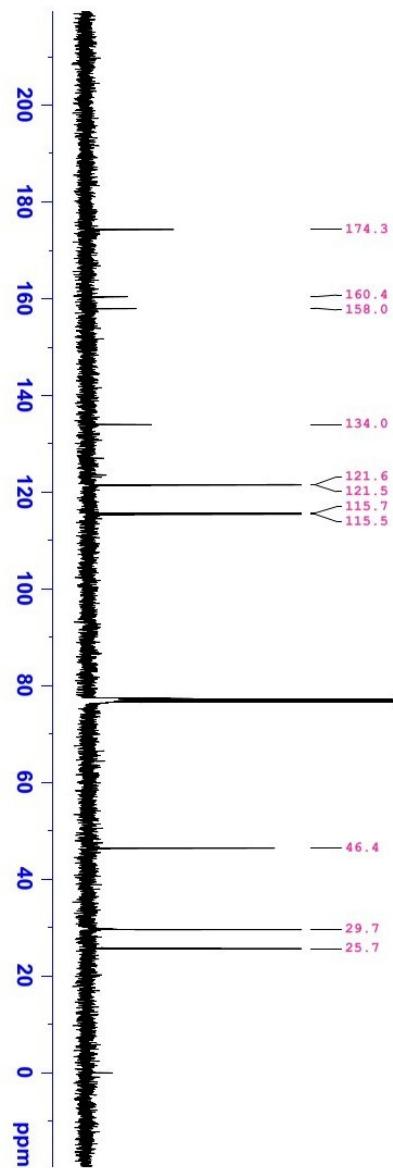
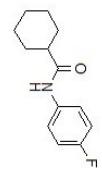
¹H NMR of Compound 3ac



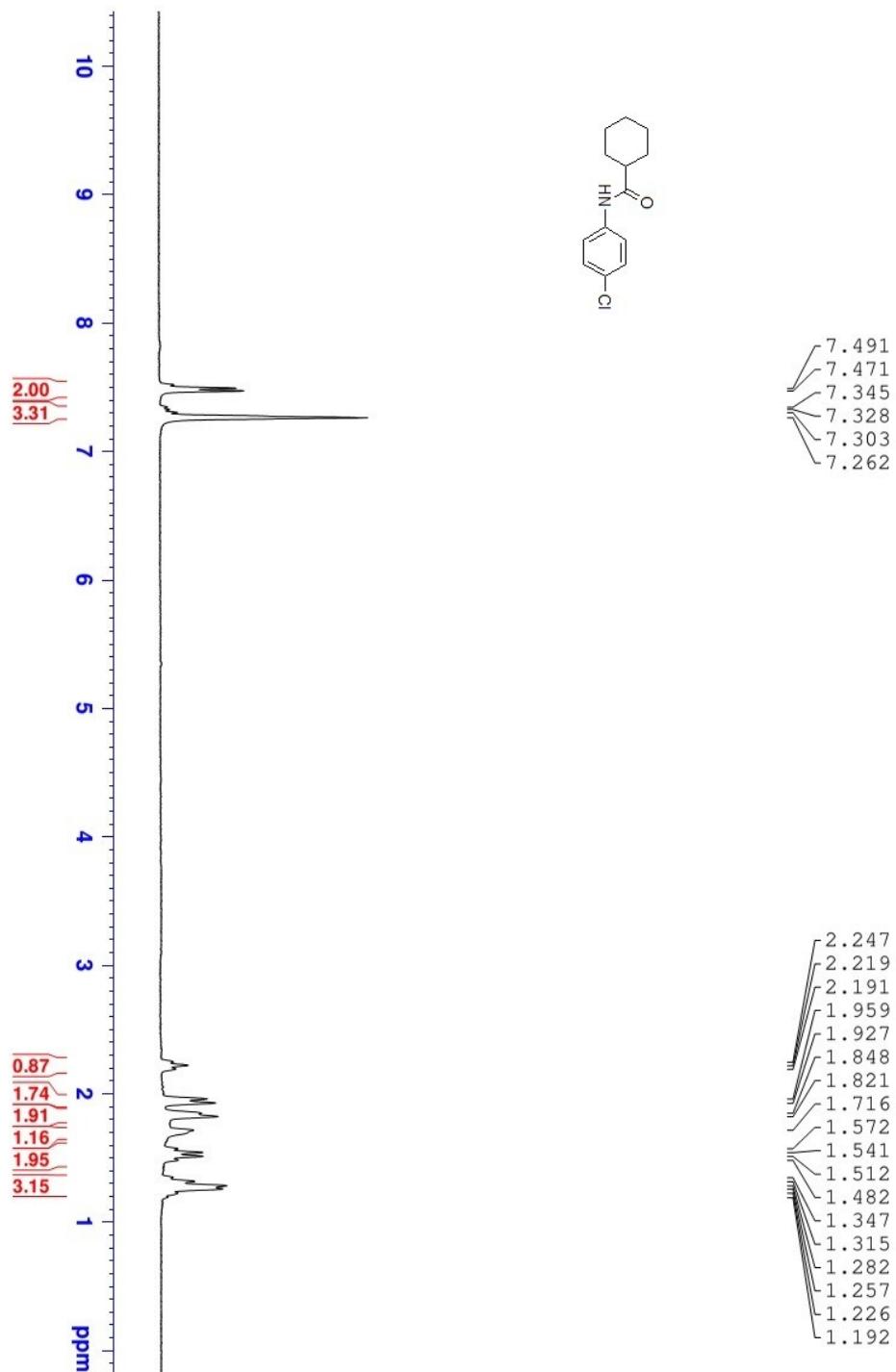
^{13}C NMR of Compound 3ac



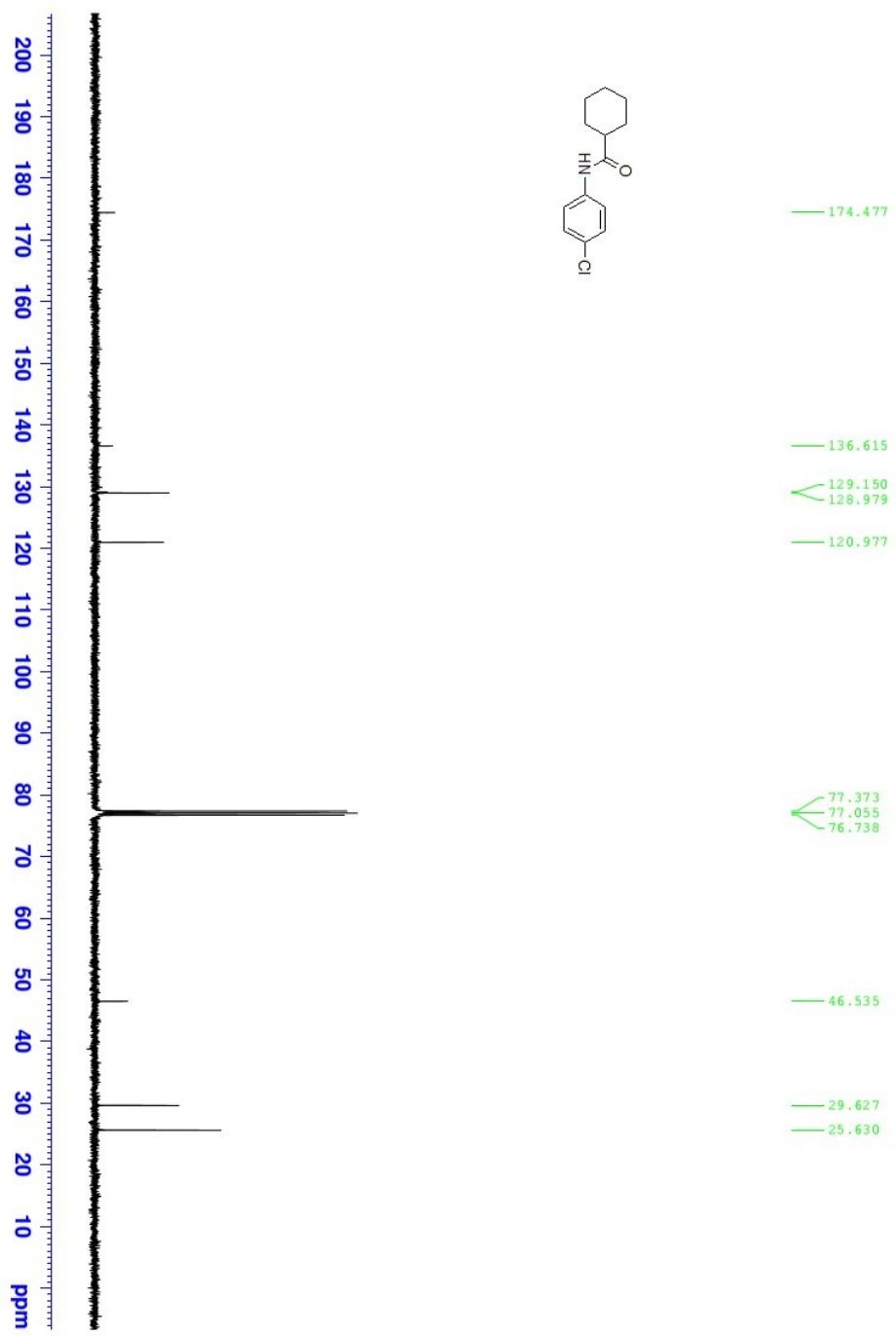
¹H NMR of Compound 3ad



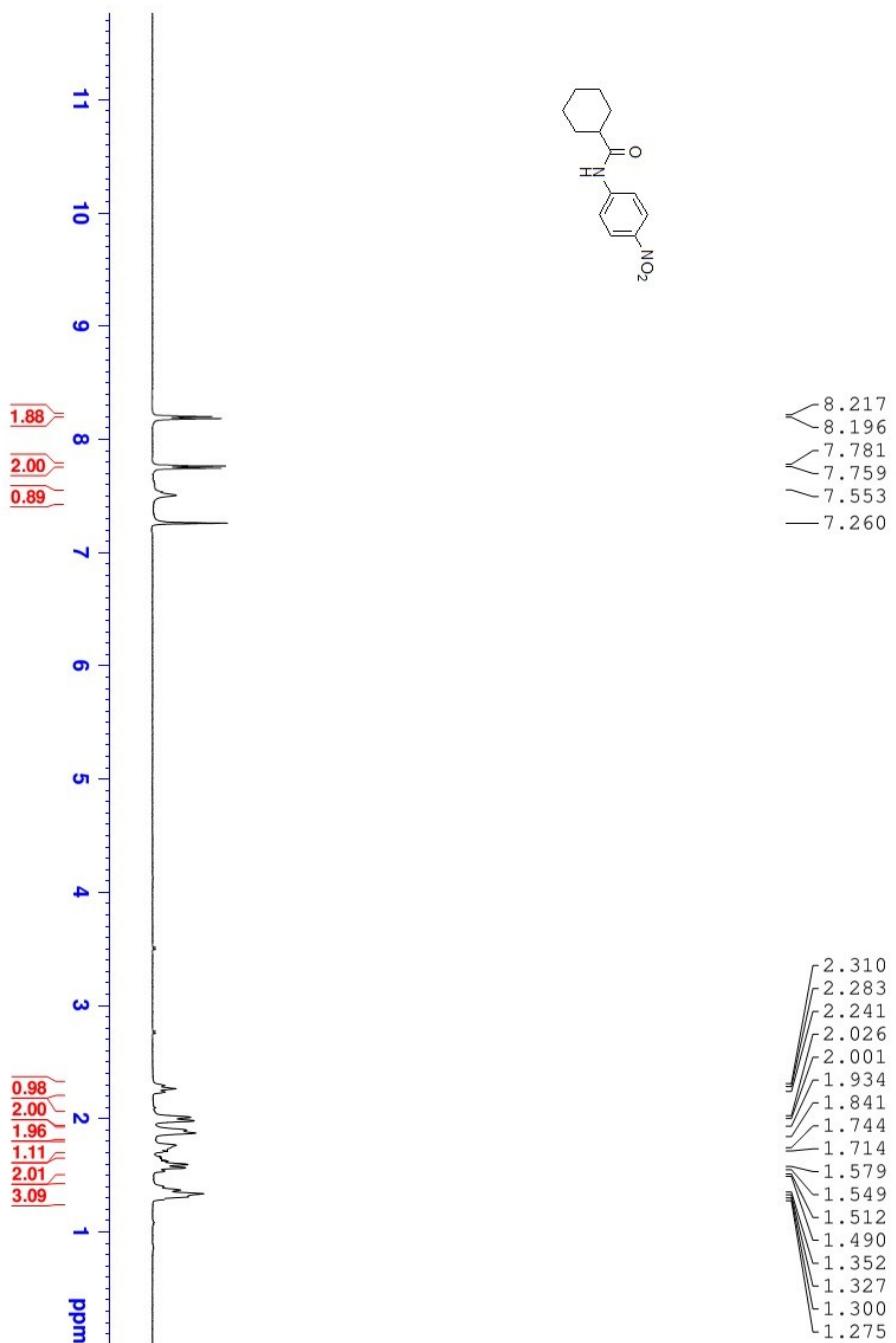
¹³C NMR of Compound 3ad



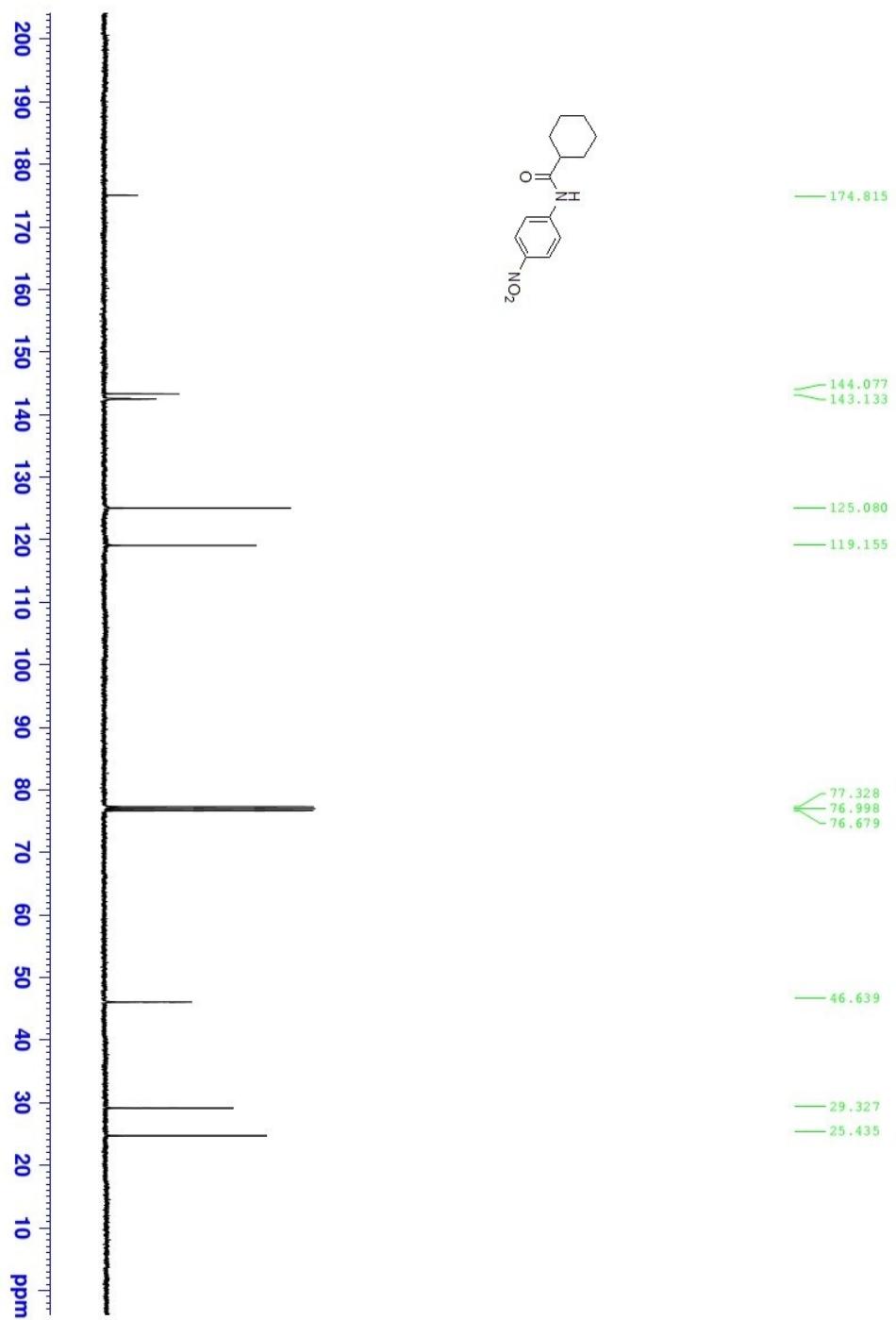
¹H NMR of Compound 3ae



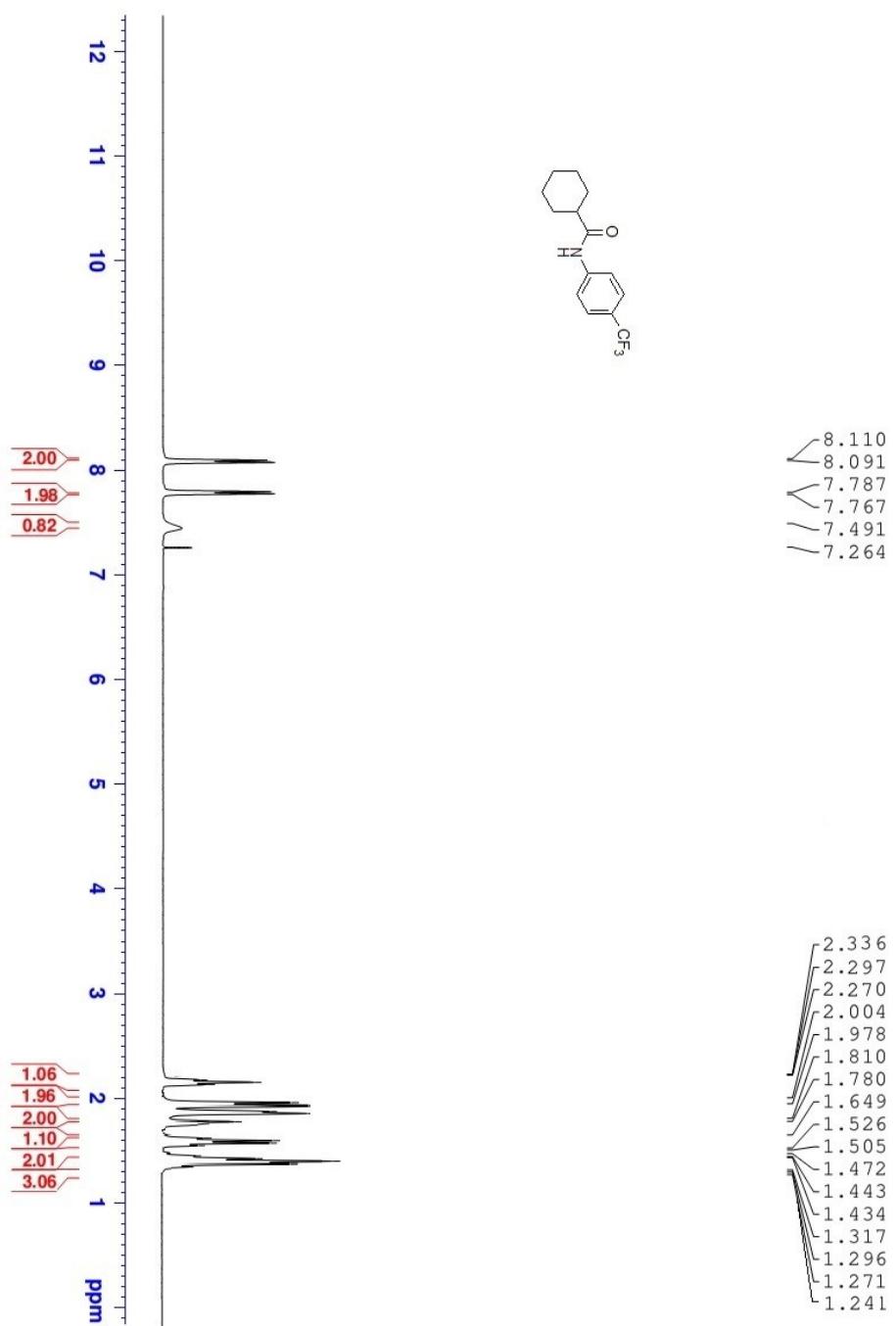
^{13}C NMR of Compound 3ae



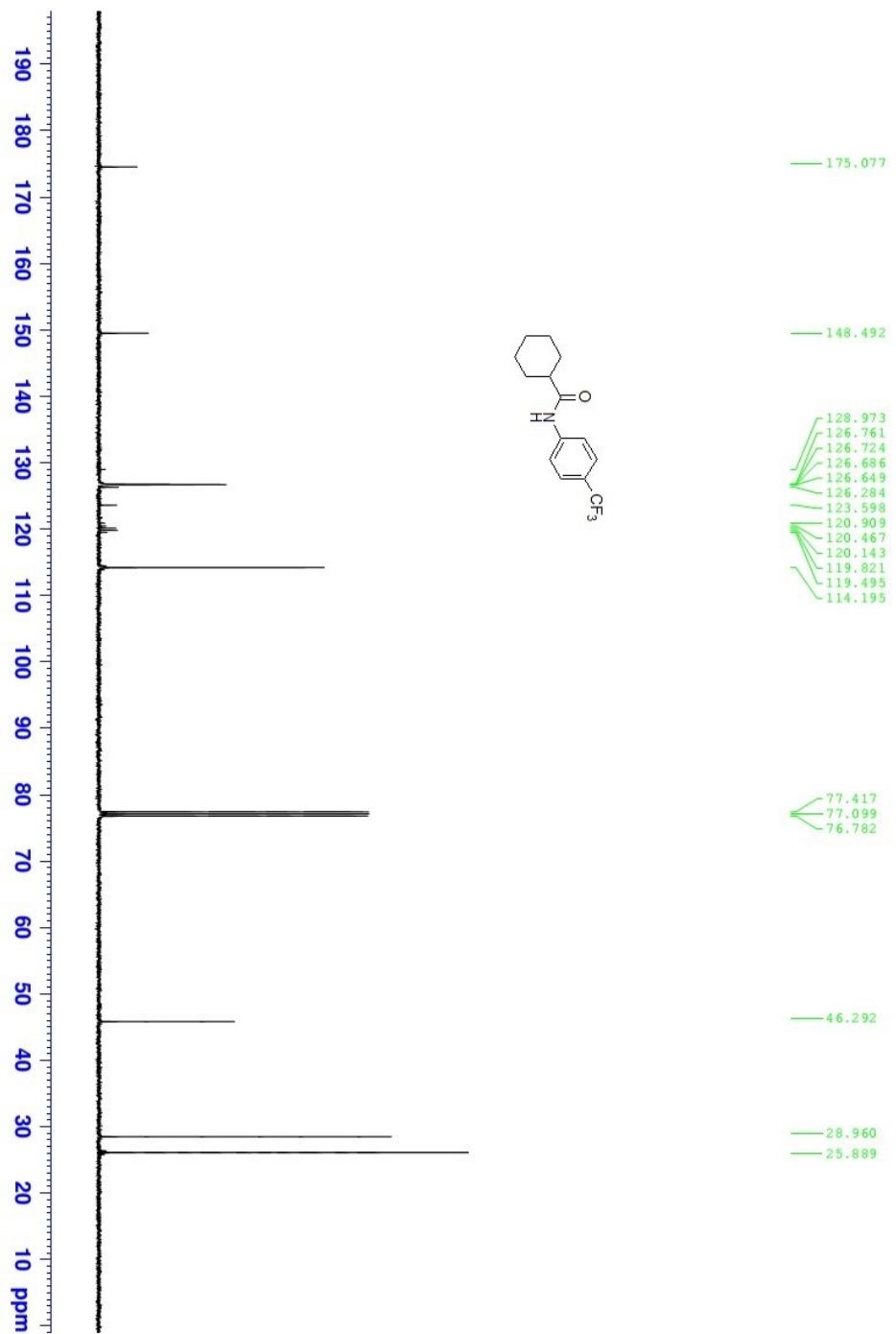
¹H NMR of Compound 3af



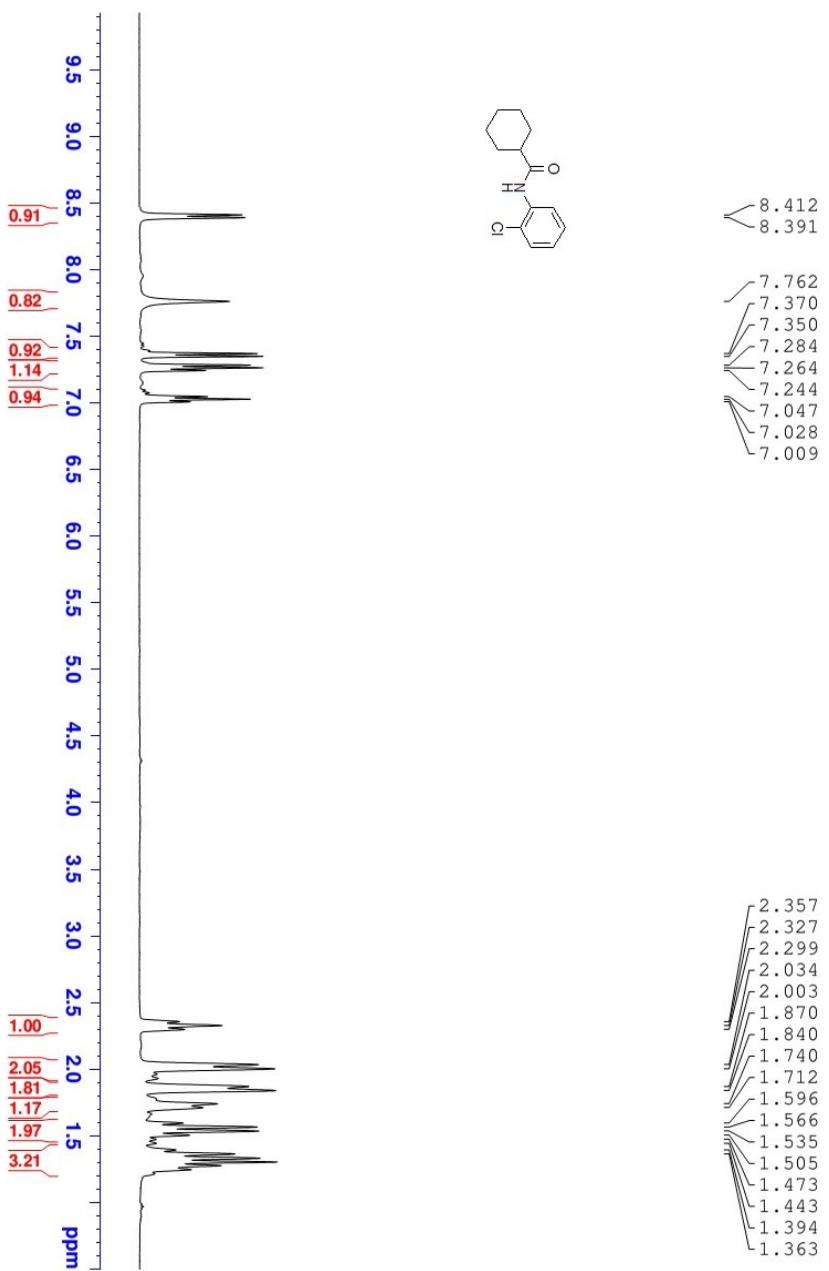
^{13}C NMR of Compound 3af



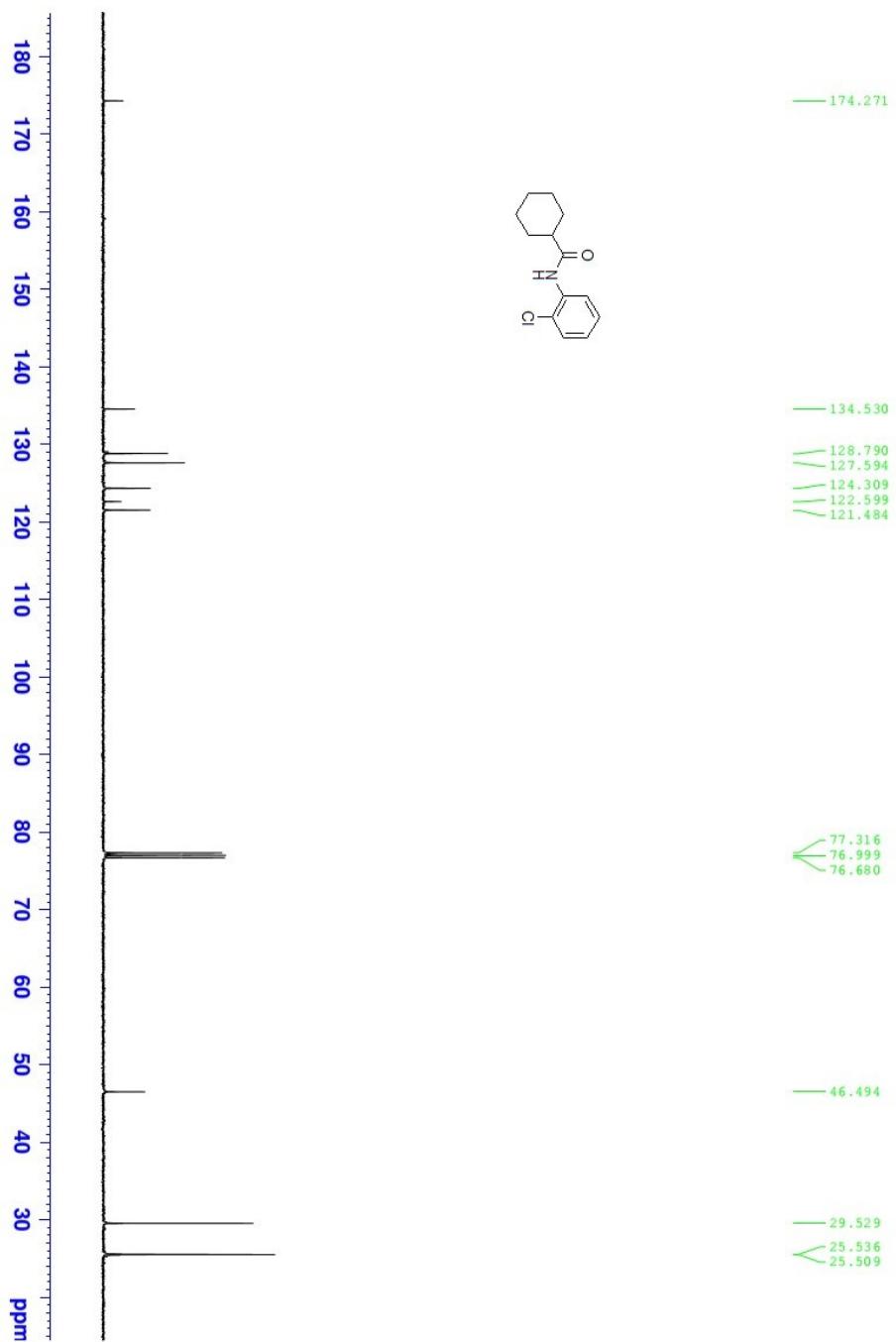
¹H NMR of Compound 3ag



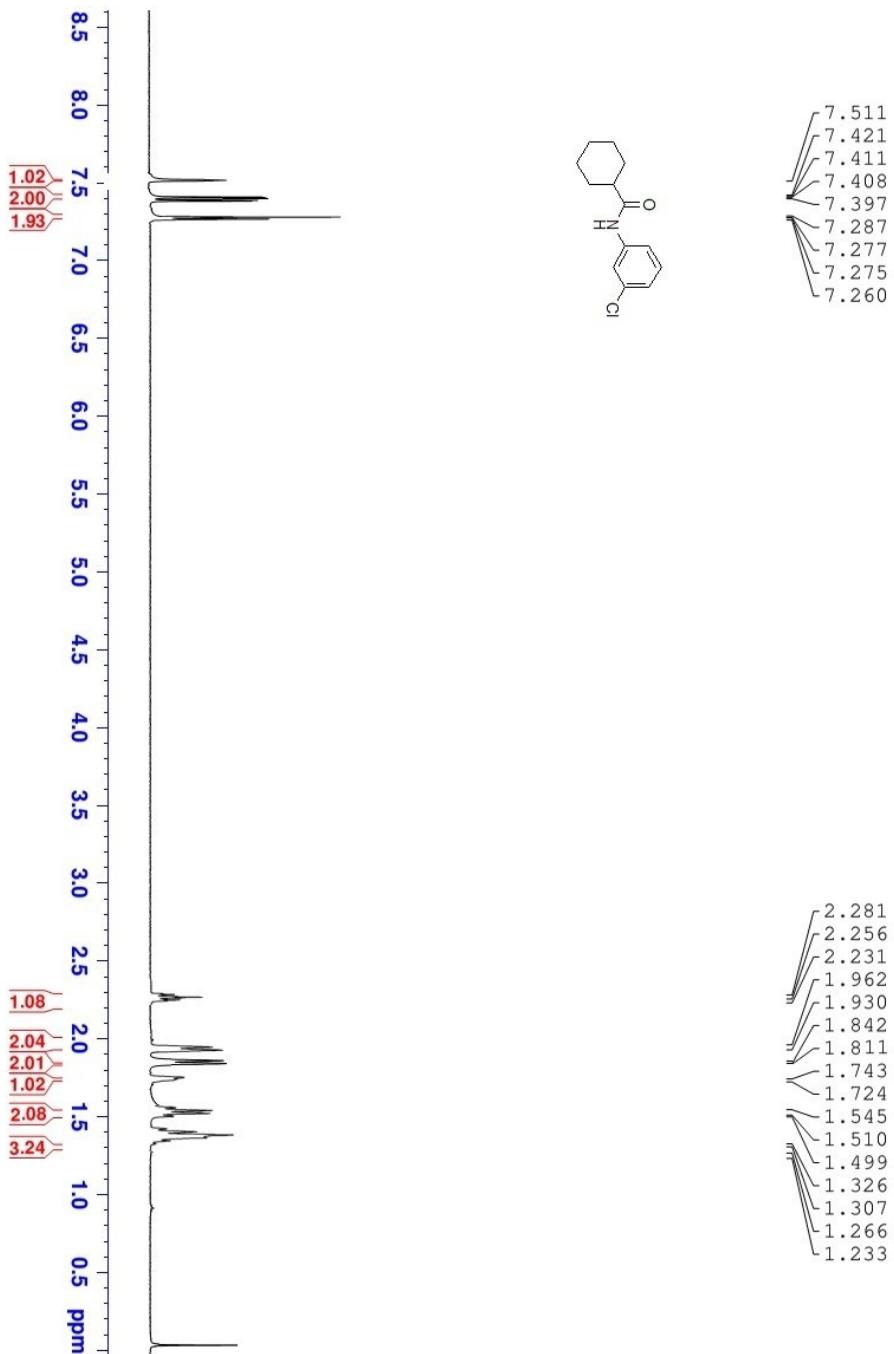
^{13}C NMR of Compound 3ag



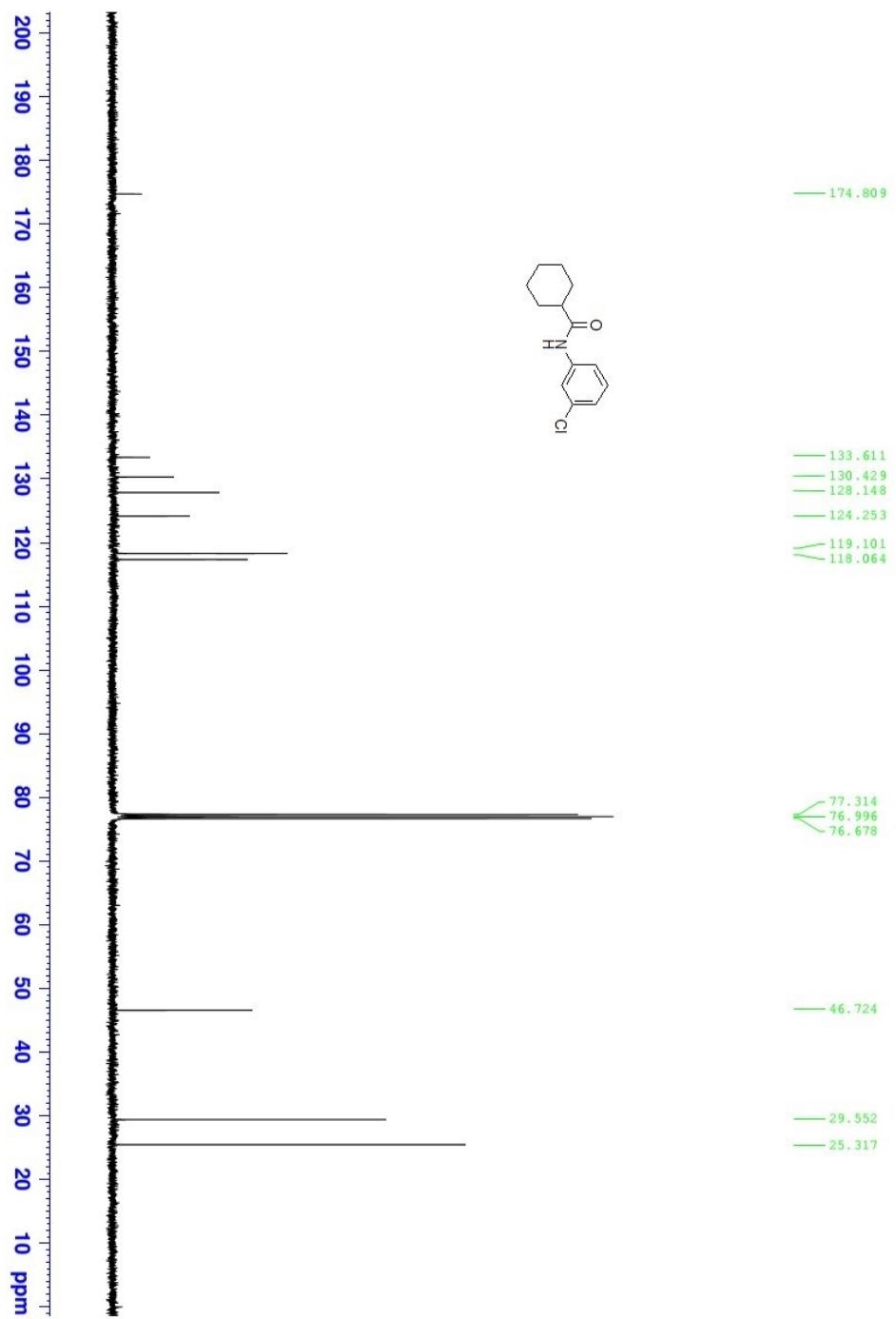
¹H NMR of Compound 3ah



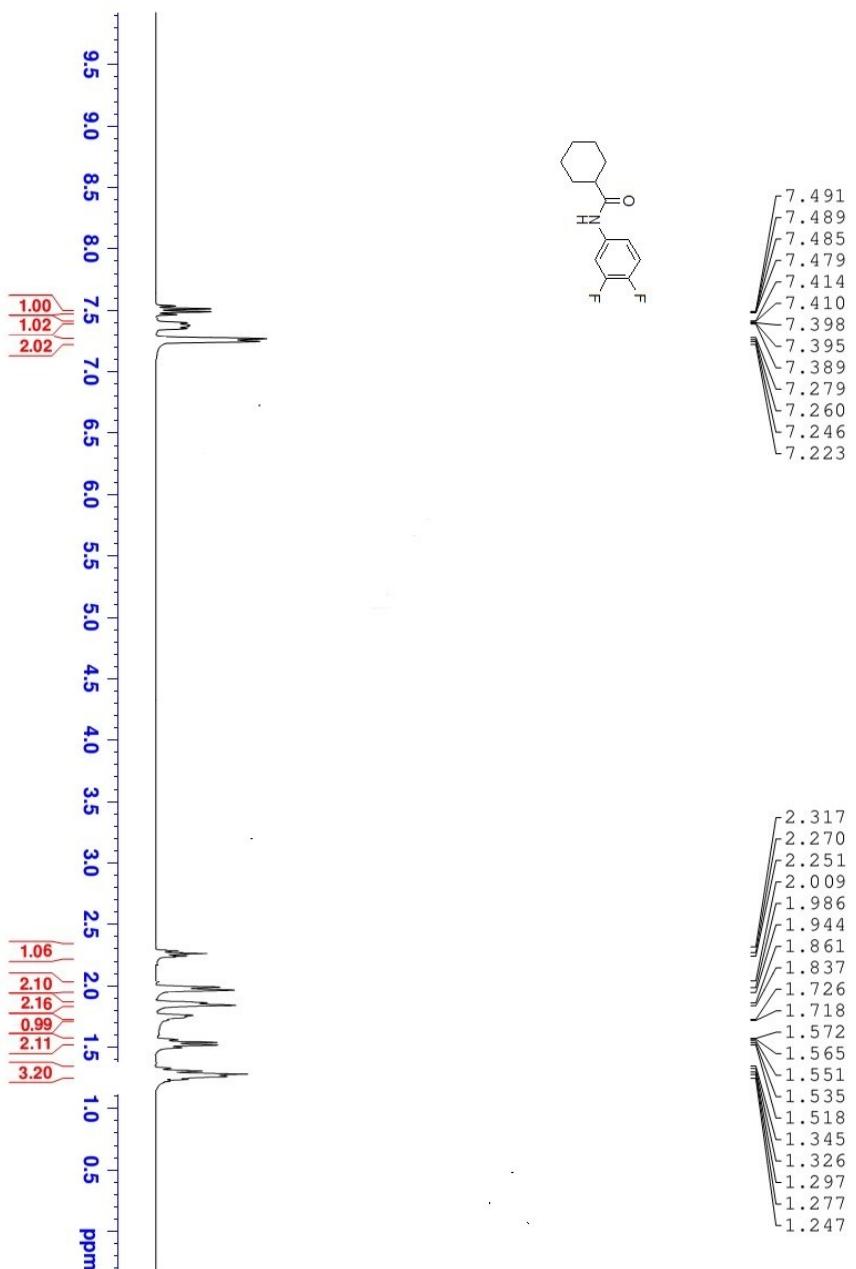
^{13}C NMR of Compound 3ah



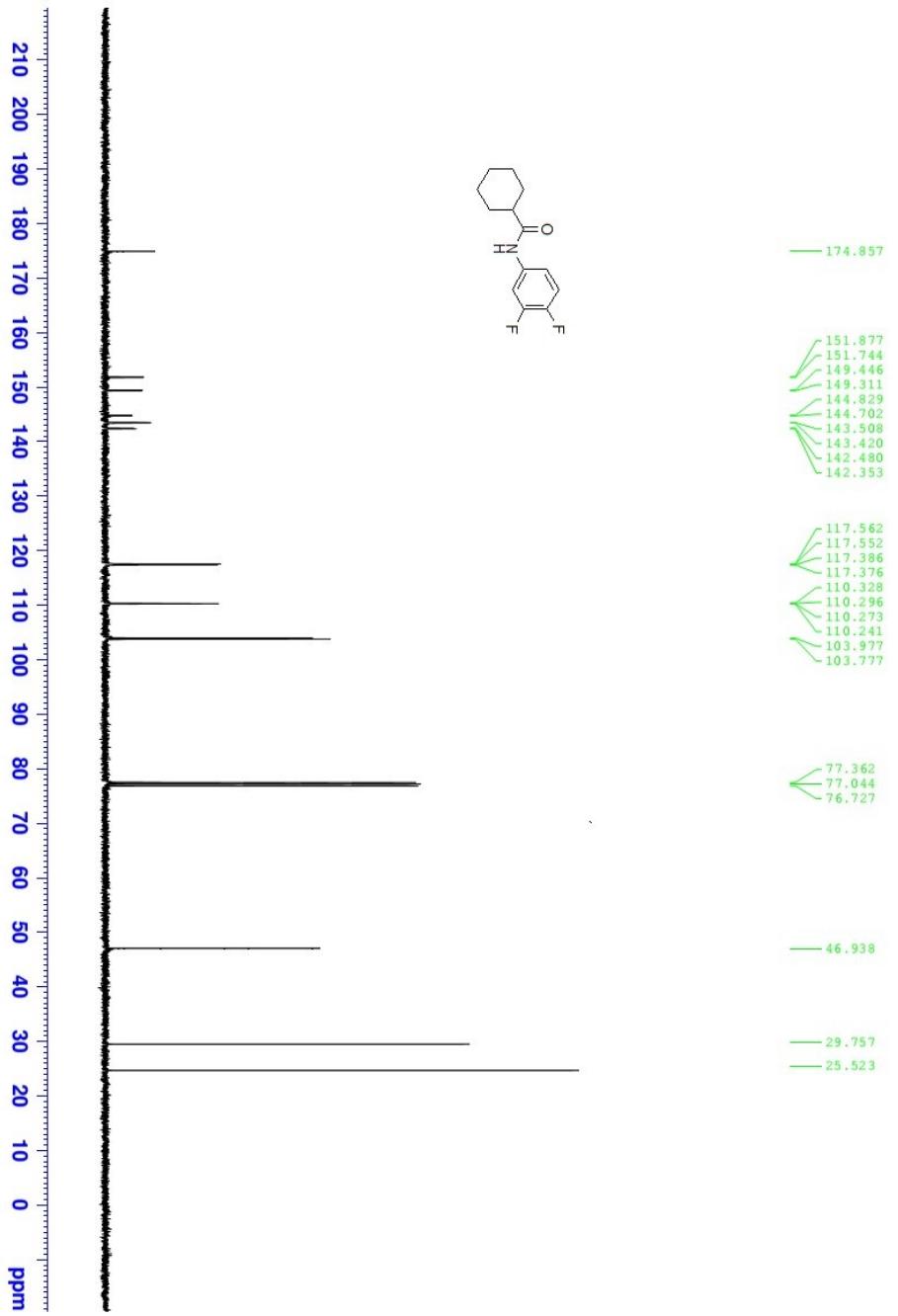
¹H NMR of Compound 3ai



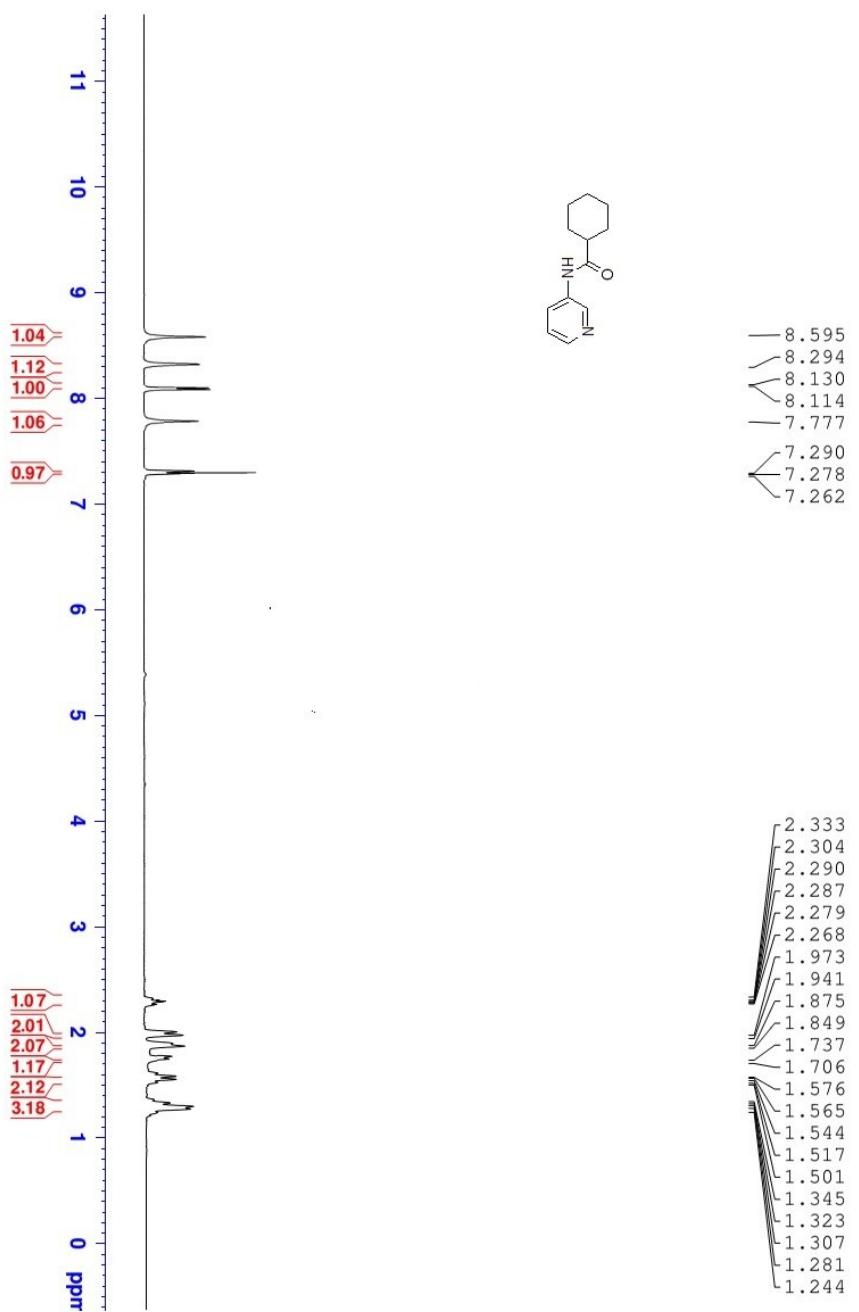
^{13}C NMR of Compound 3ai



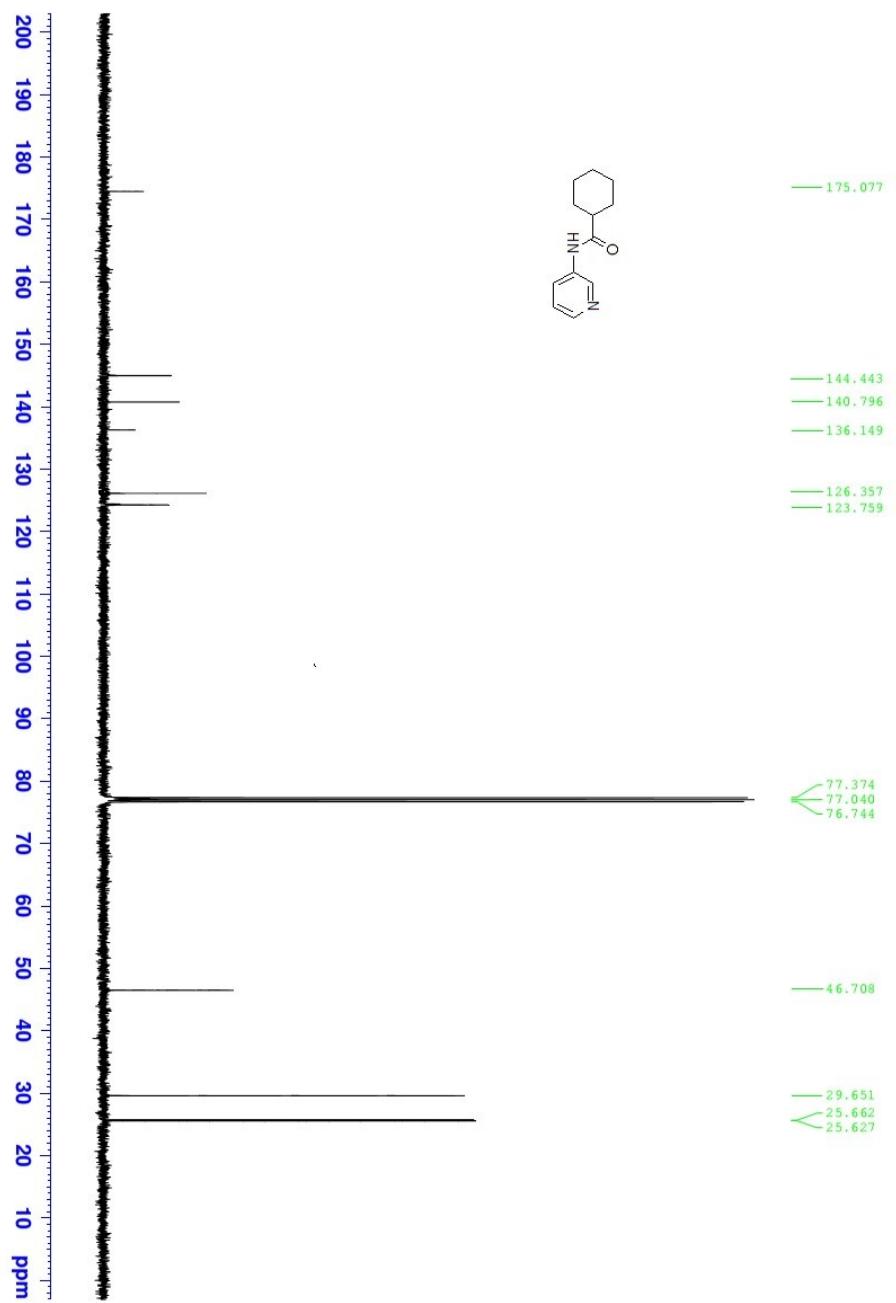
¹H NMR of Compound 3aj



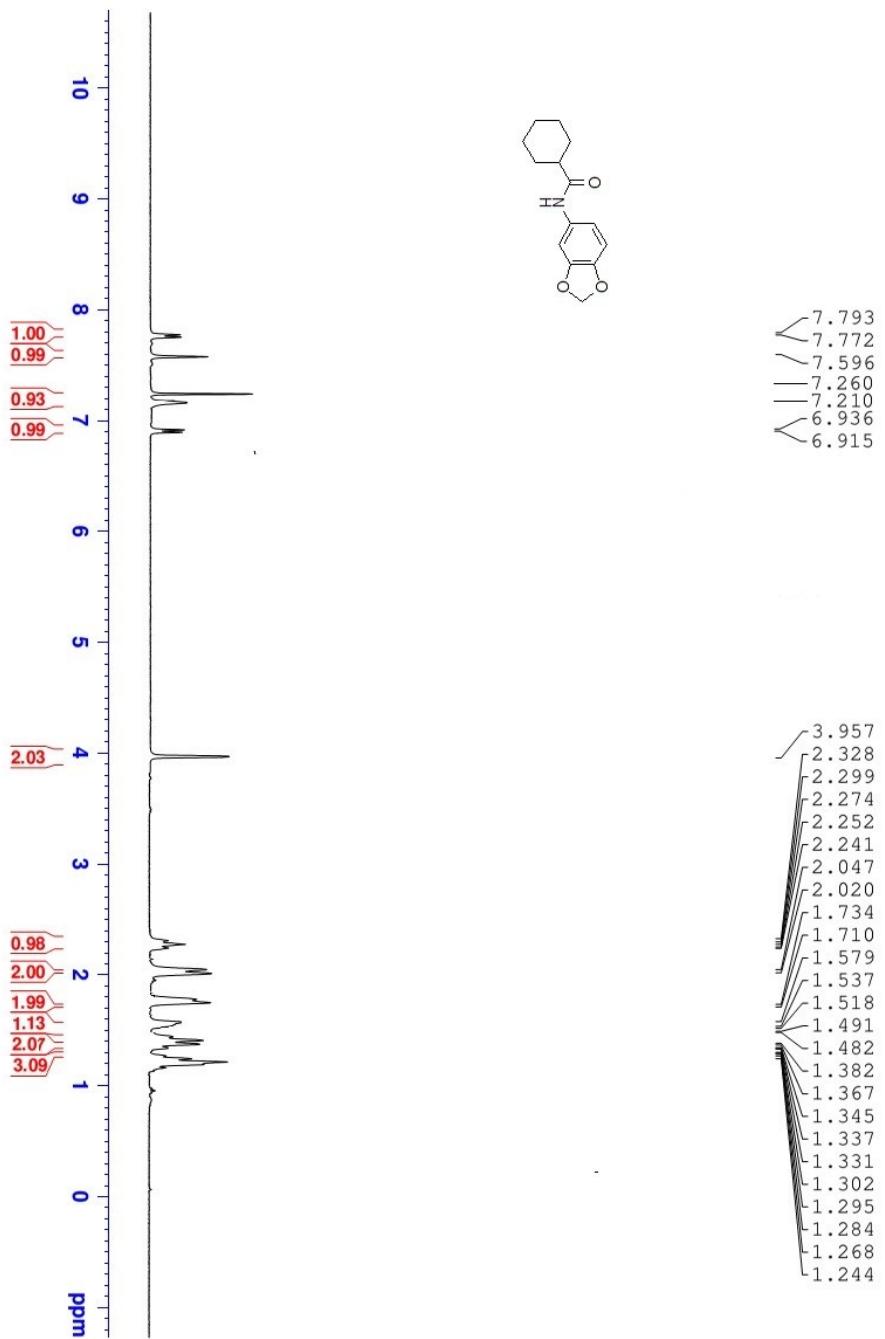
^{13}C NMR of Compound 3aj



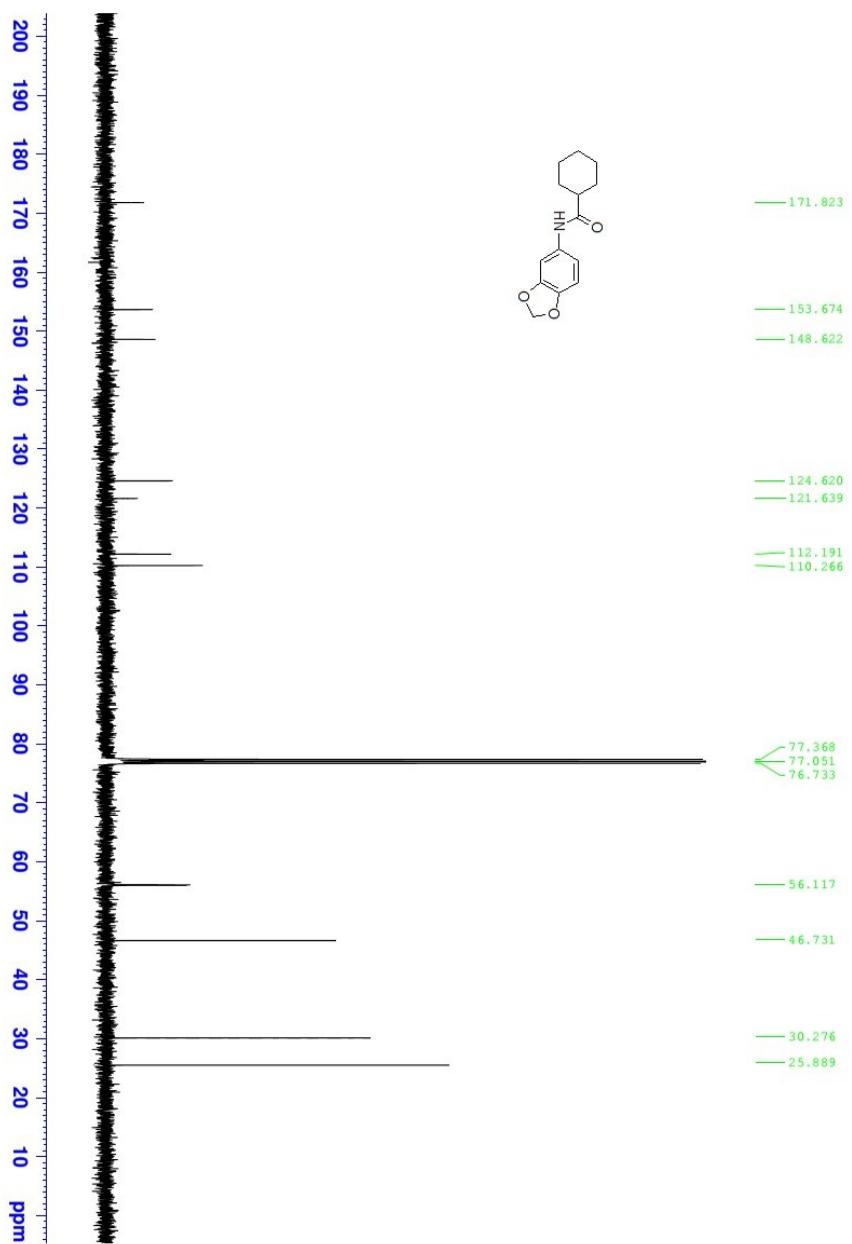
¹H NMR of Compound 3ak



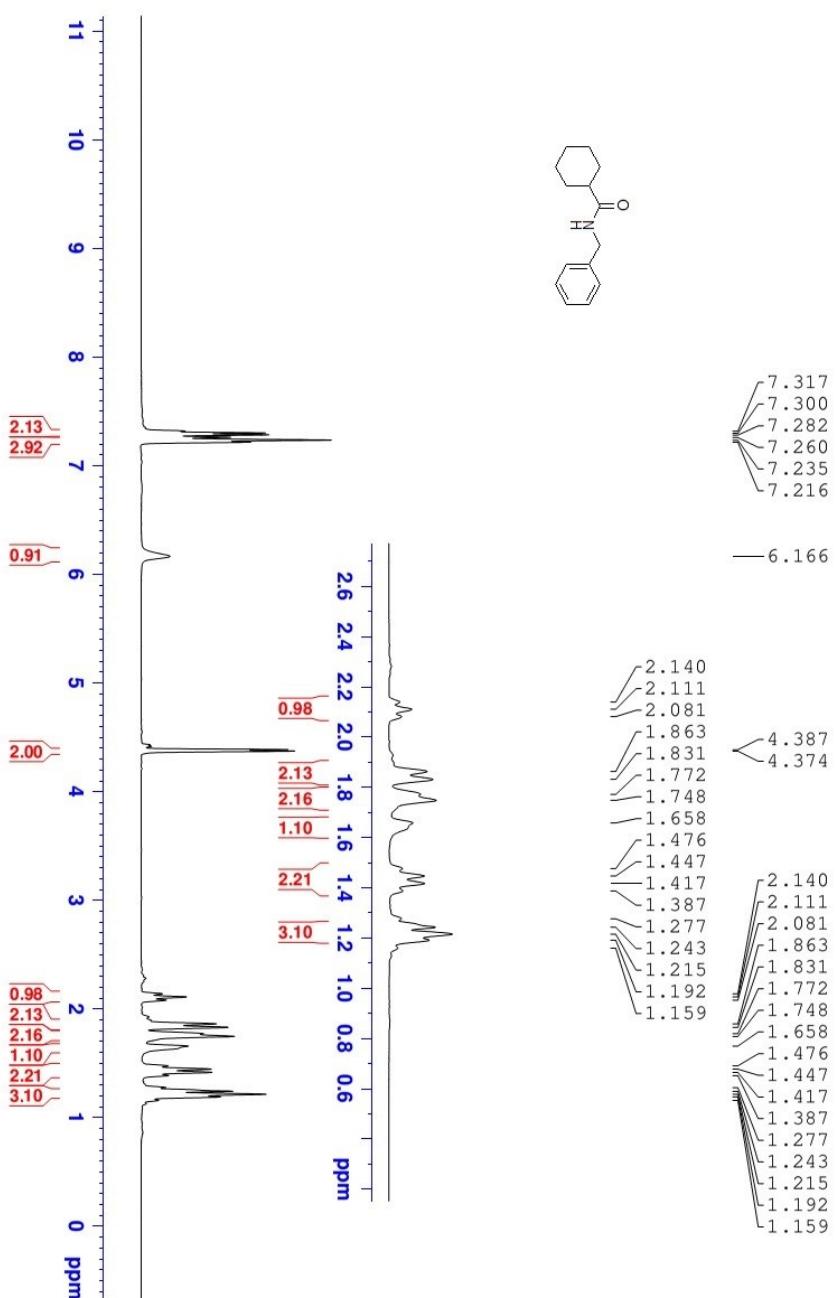
^{13}C NMR of Compound 3ak



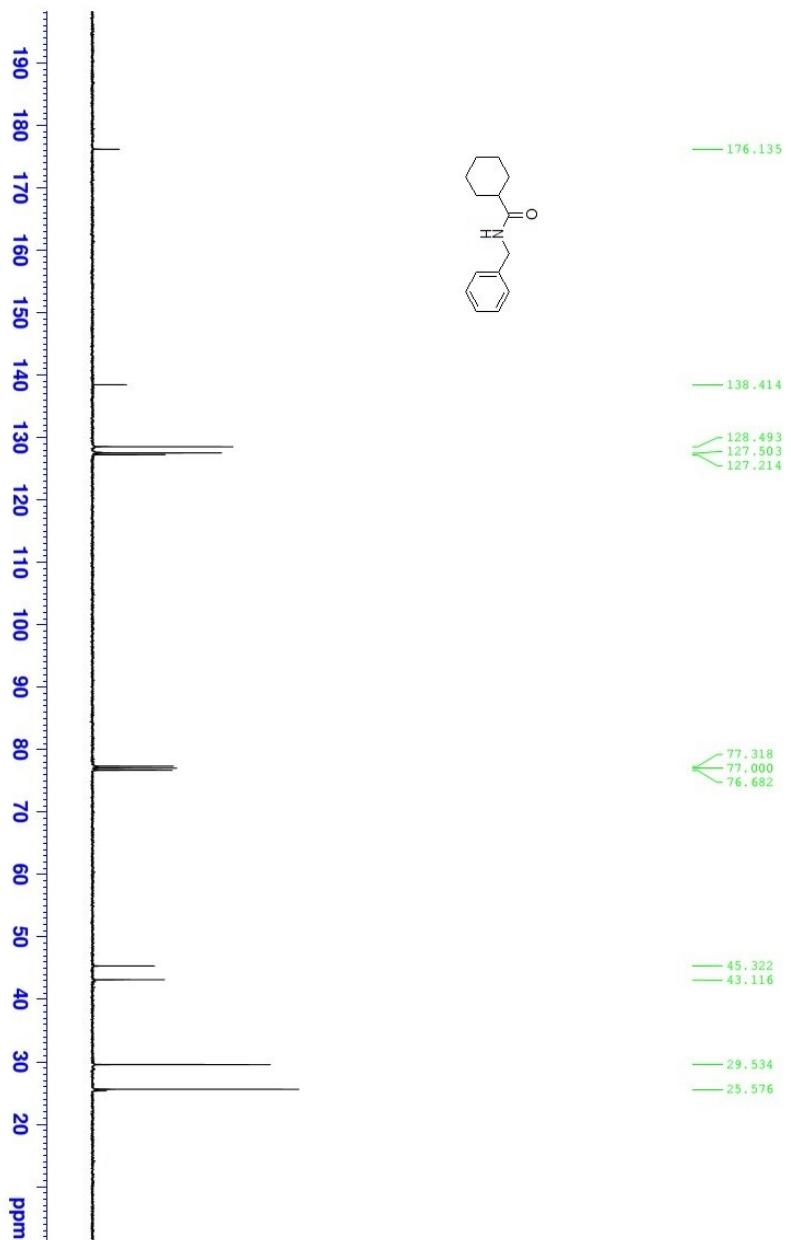
¹H NMR of Compound 3al



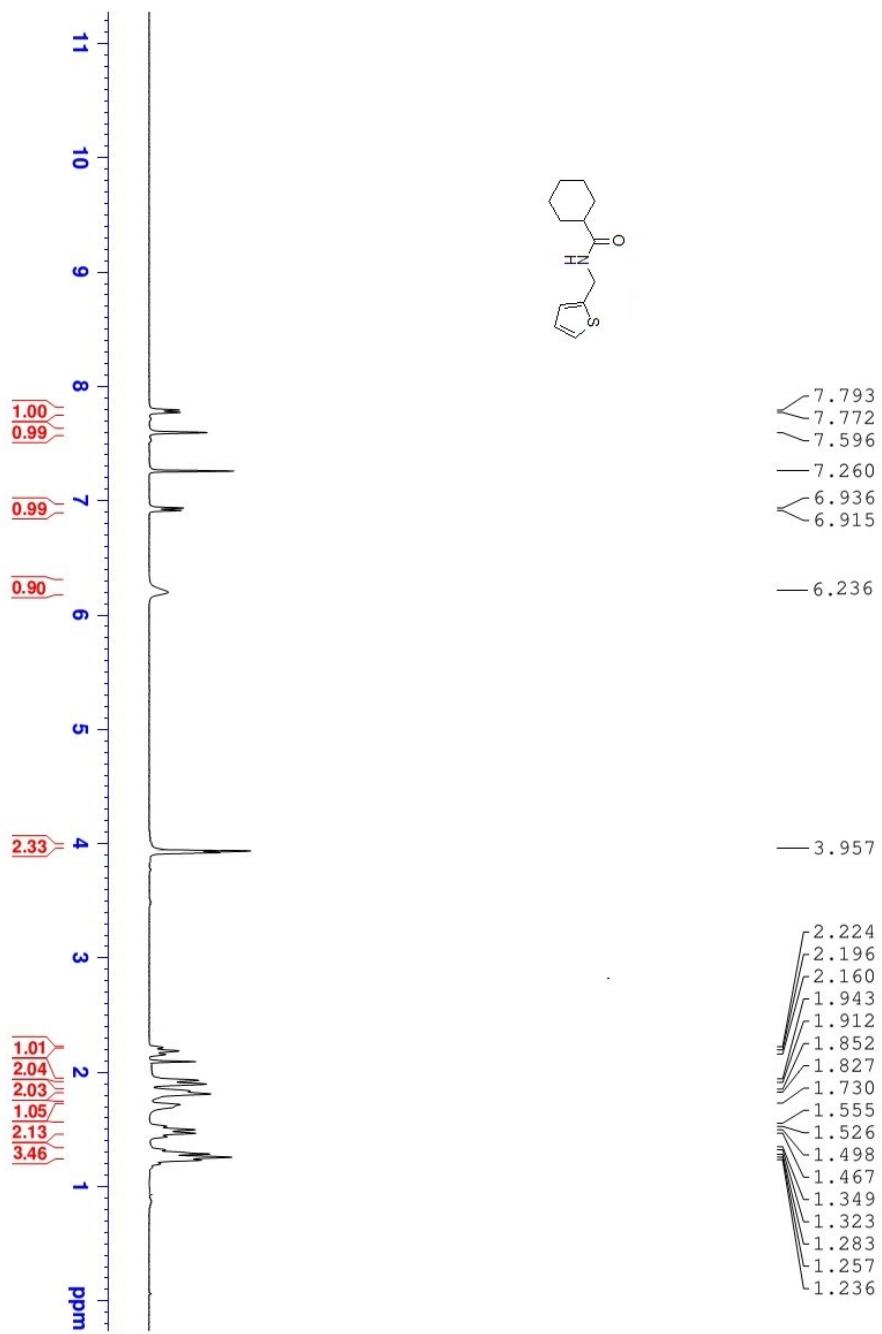
^{13}C NMR of Compound 3al



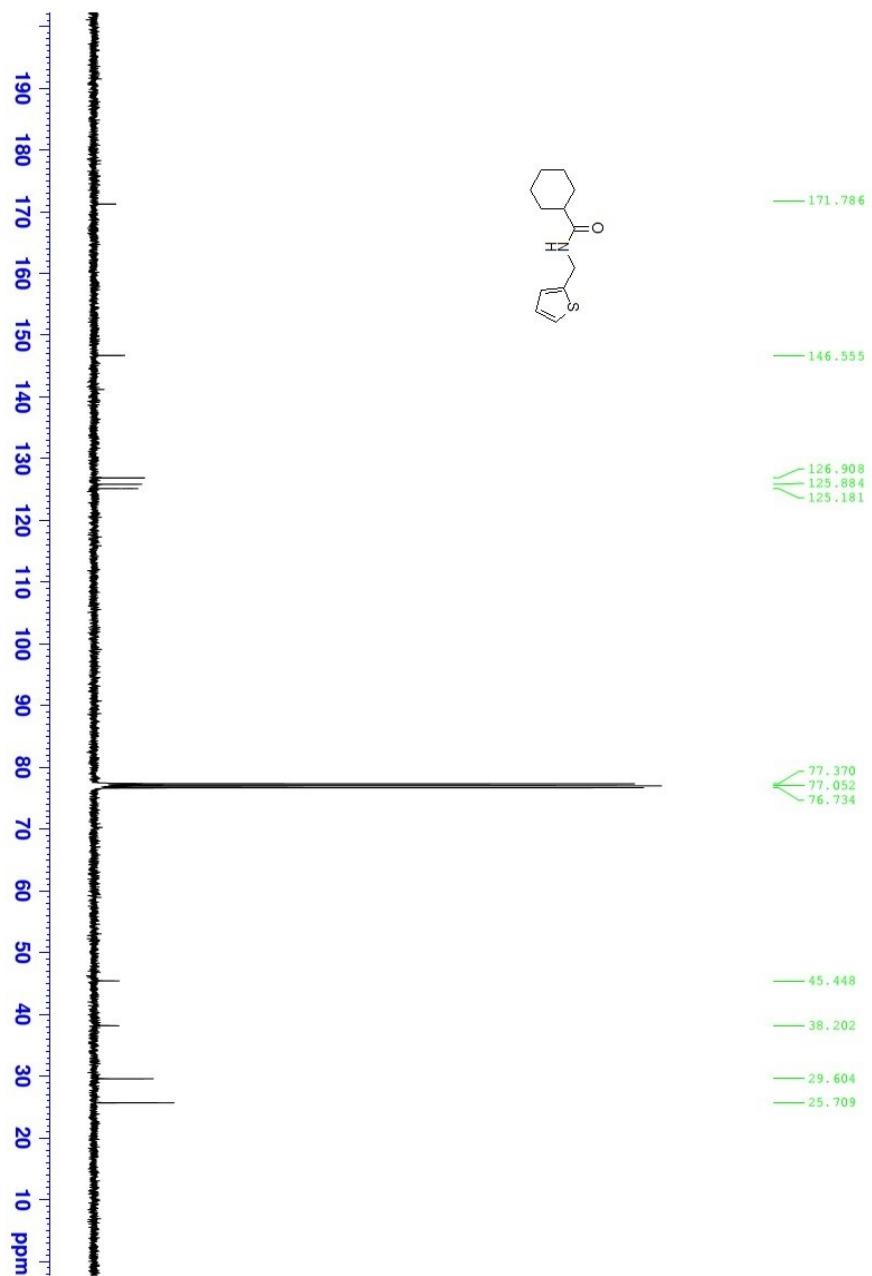
¹H NMR of Compound 3am



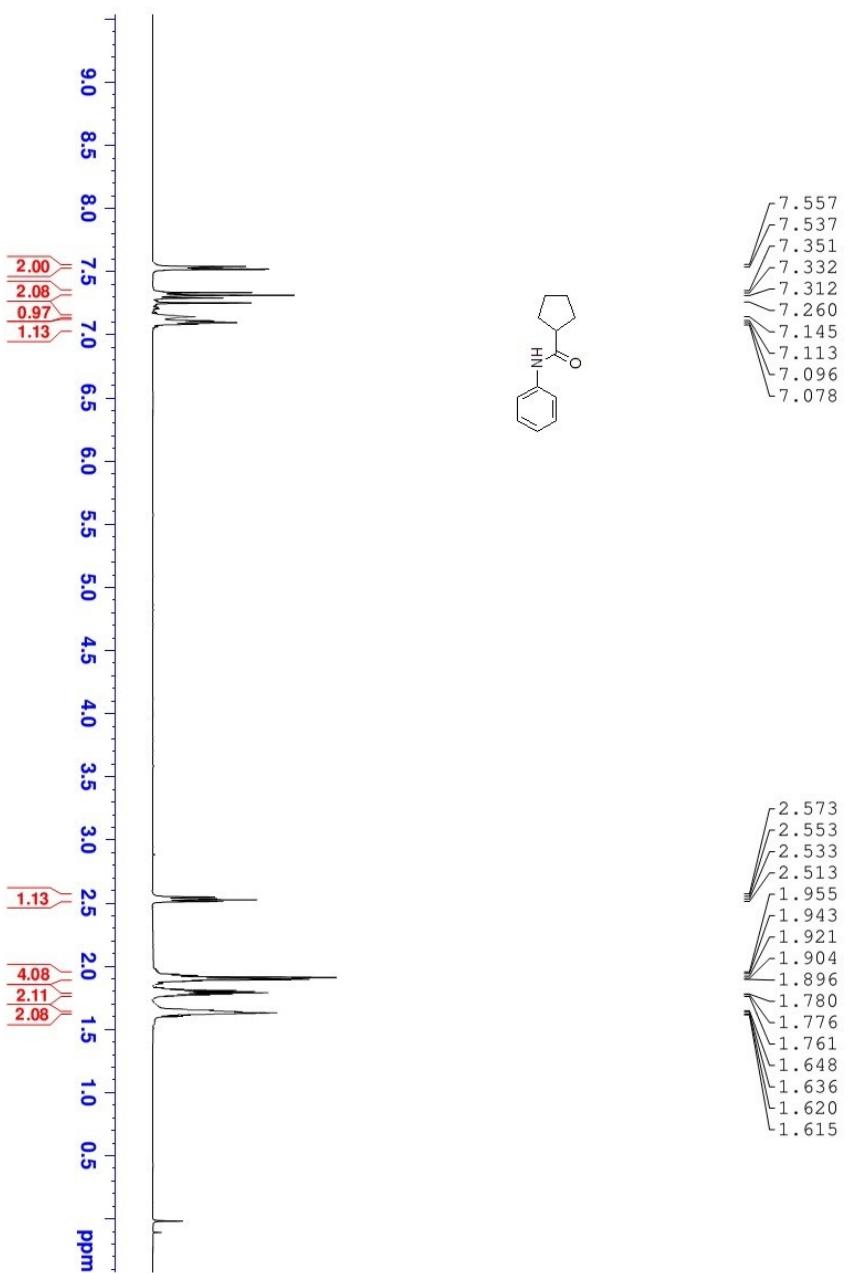
^{13}C NMR of Compound 3am



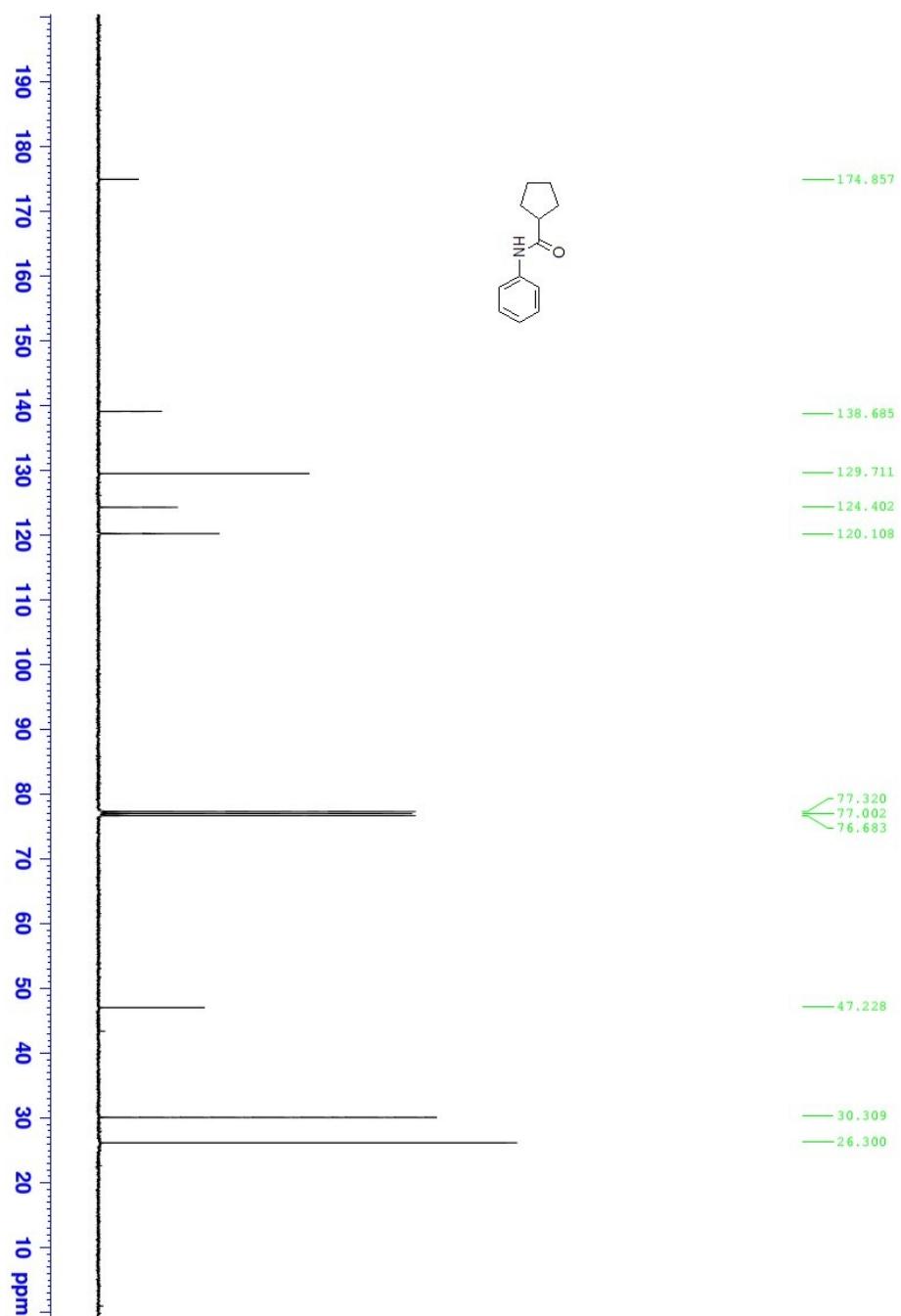
¹H NMR of Compound 3an



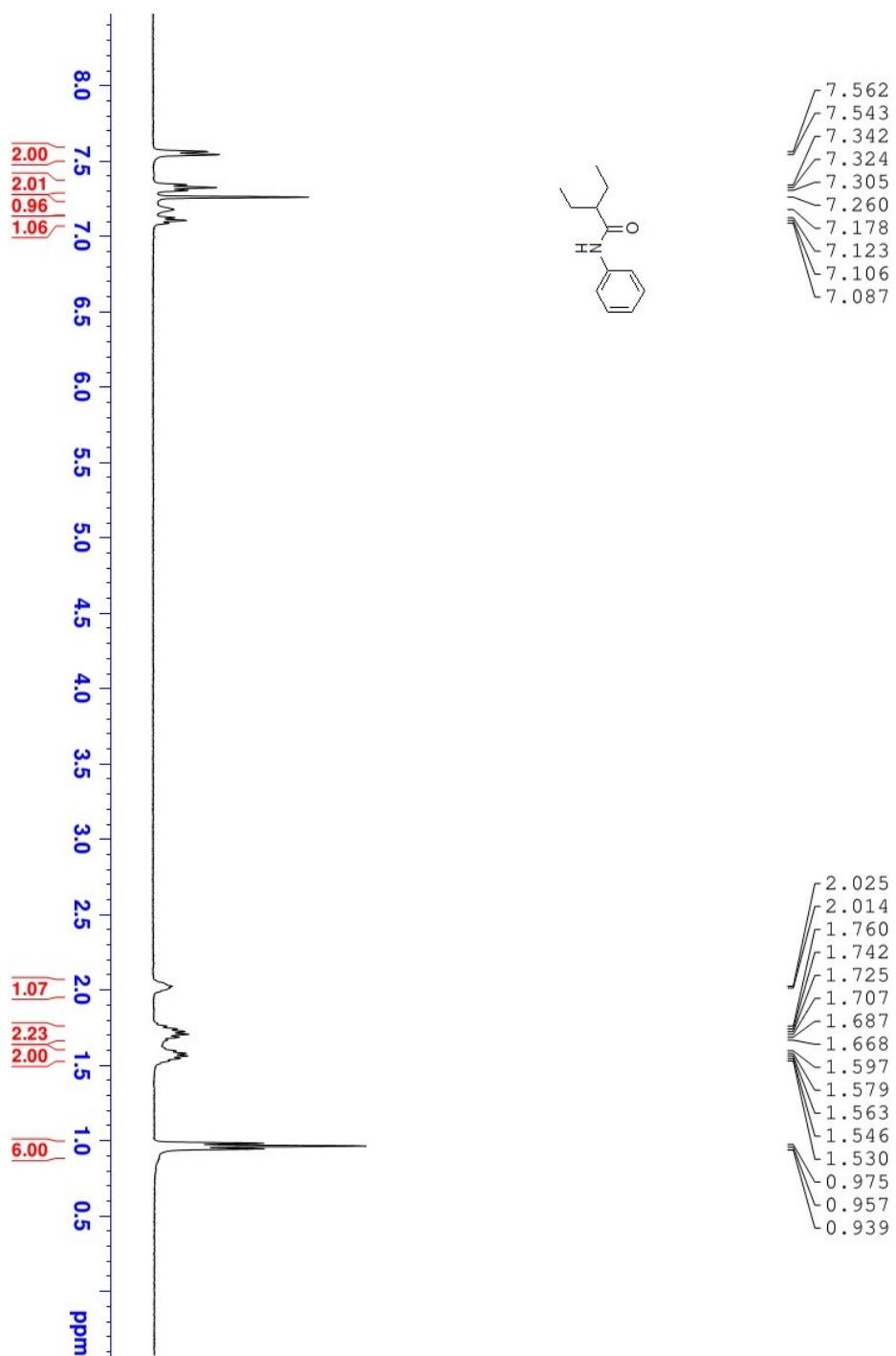
^{13}C NMR of Compound 3an



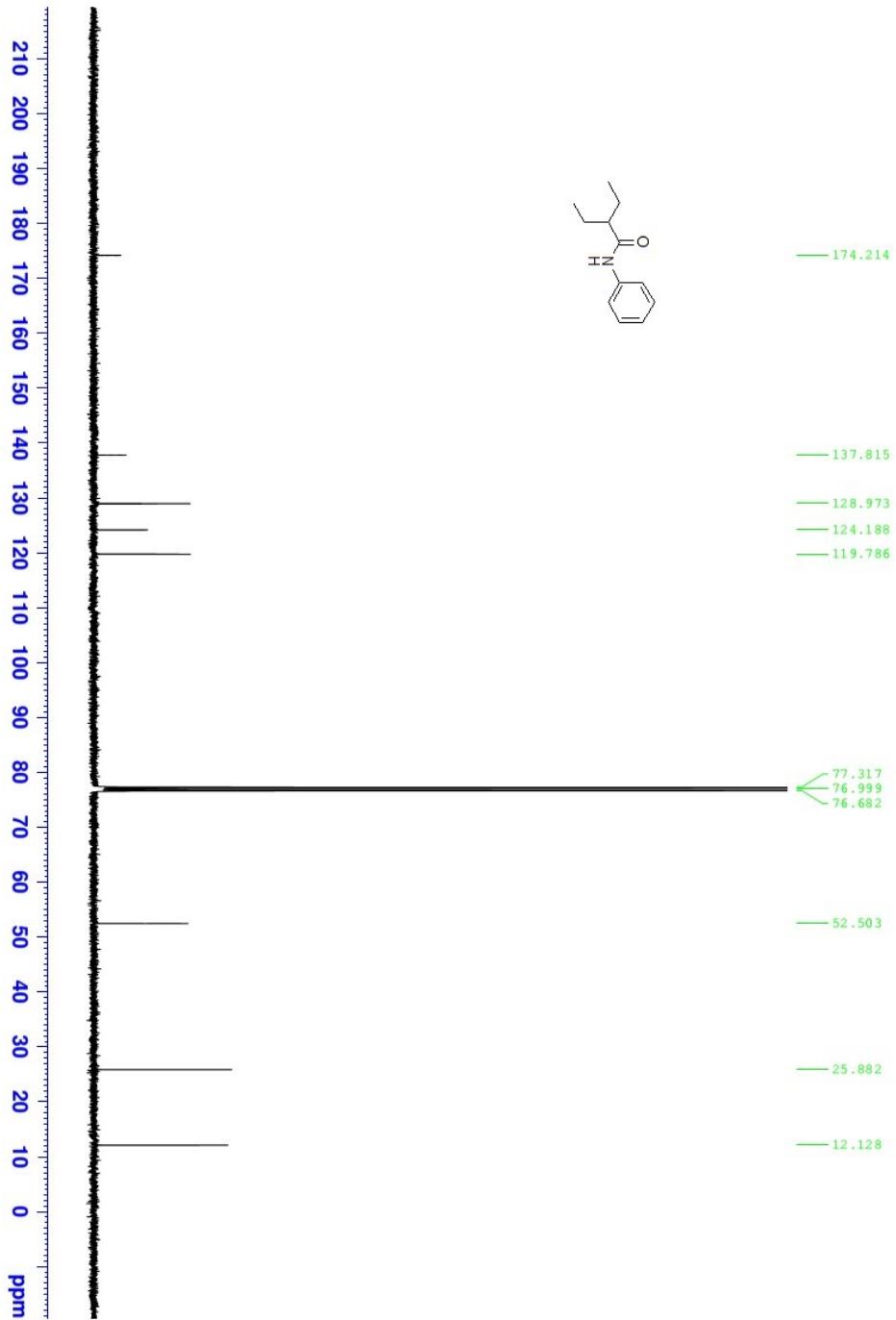
¹H NMR of Compound 3ab



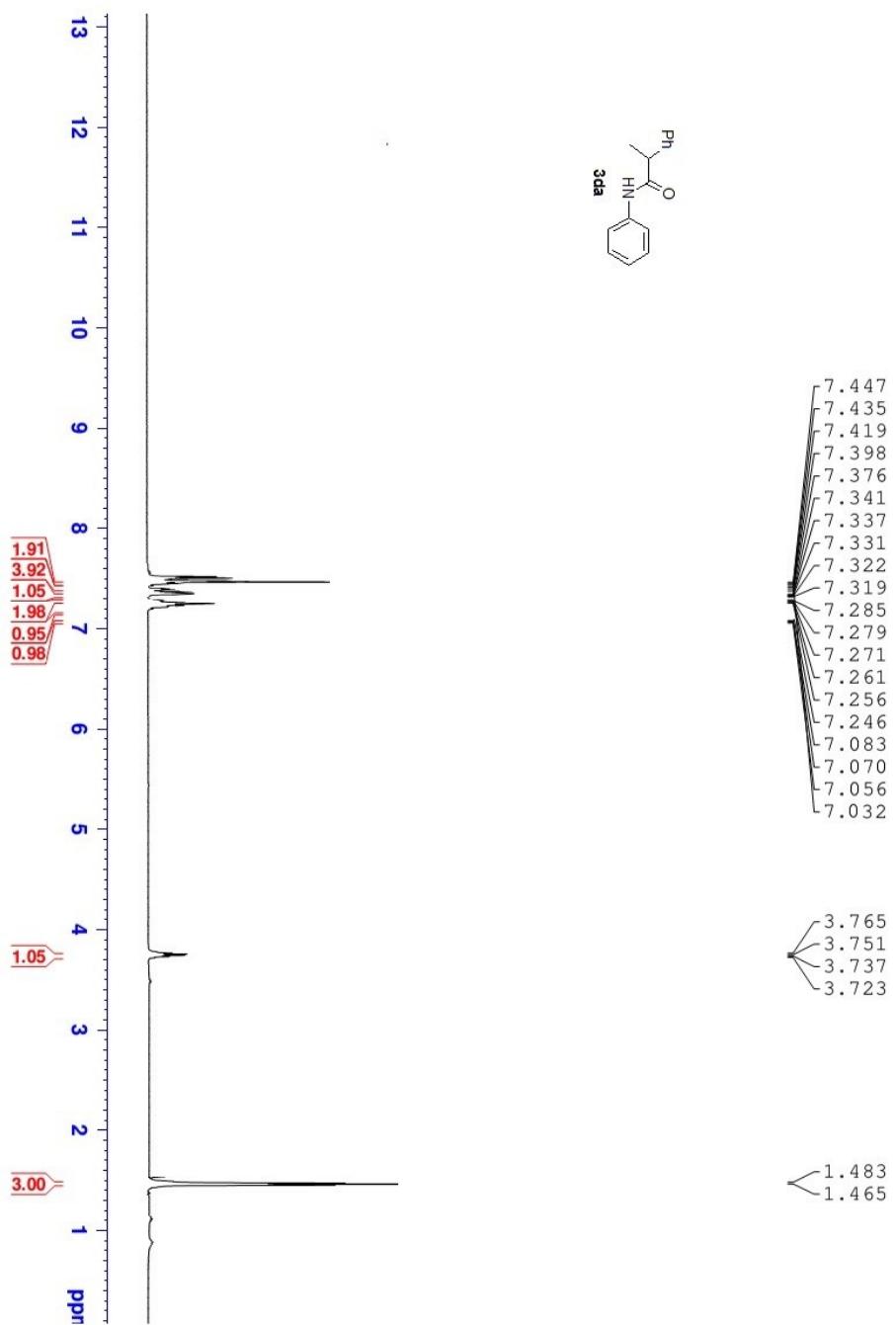
^{13}C NMR of Compound 3ba



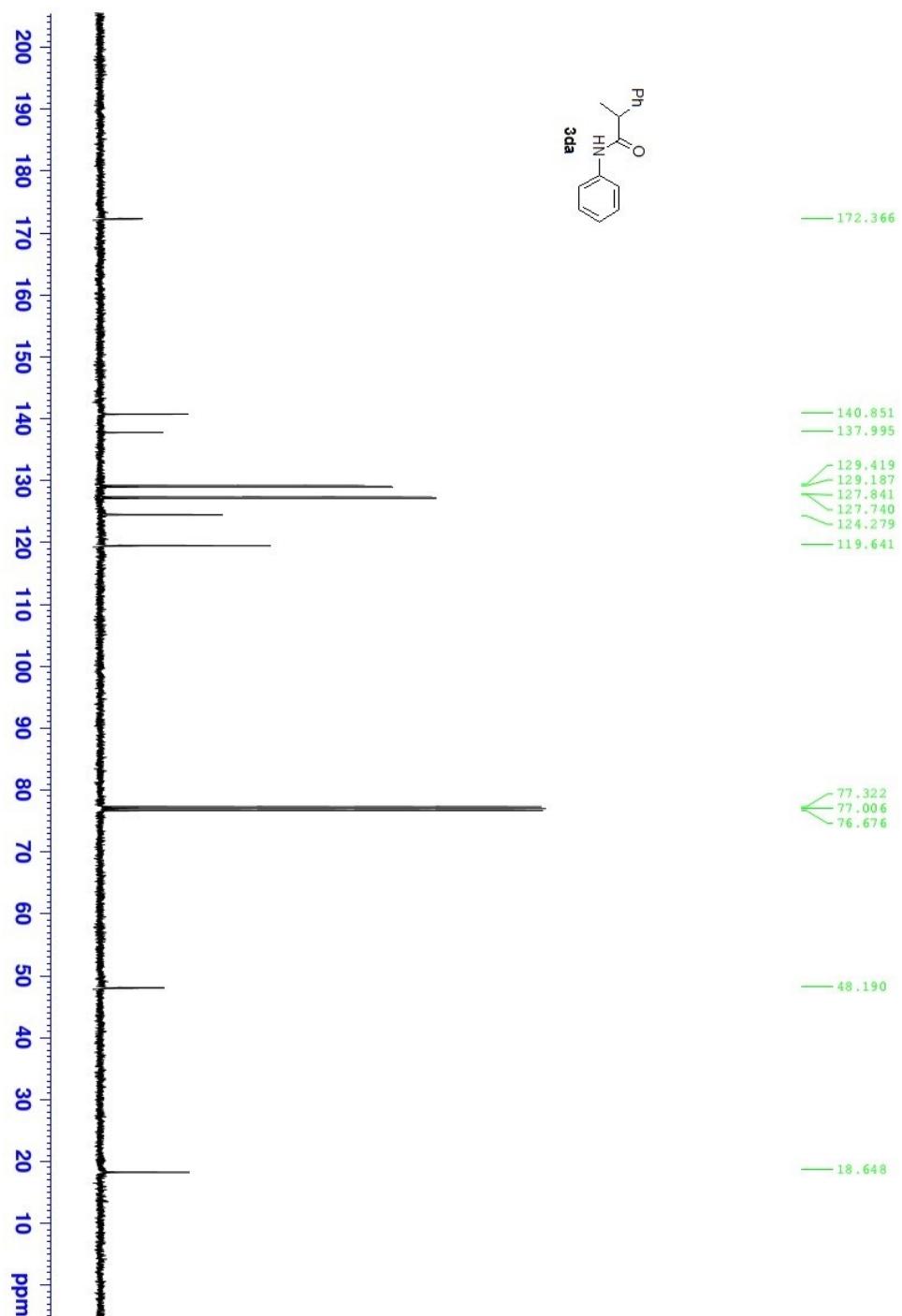
^1H NMR of Compound 3ca



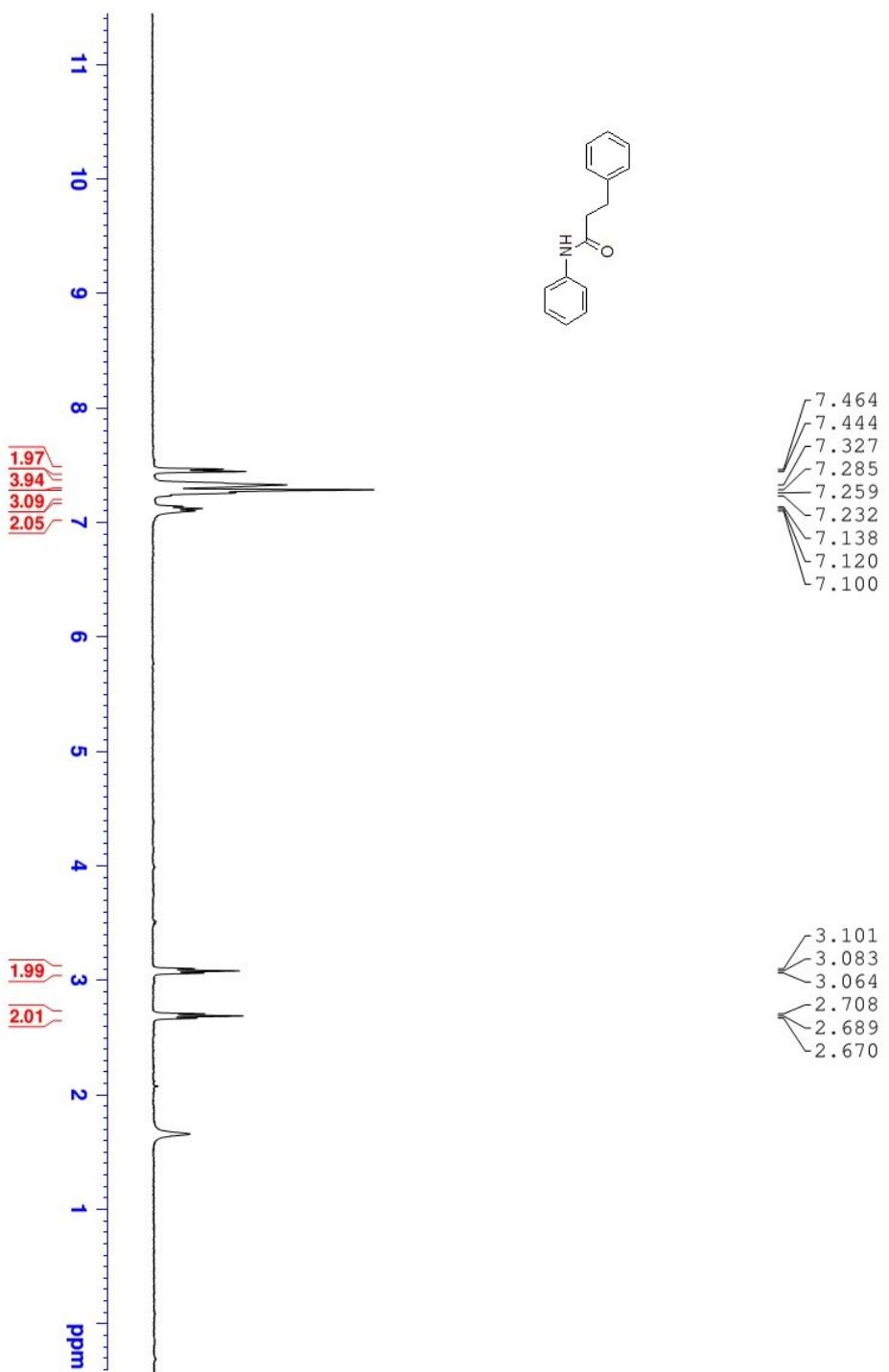
^{13}C NMR of Compound 3ca



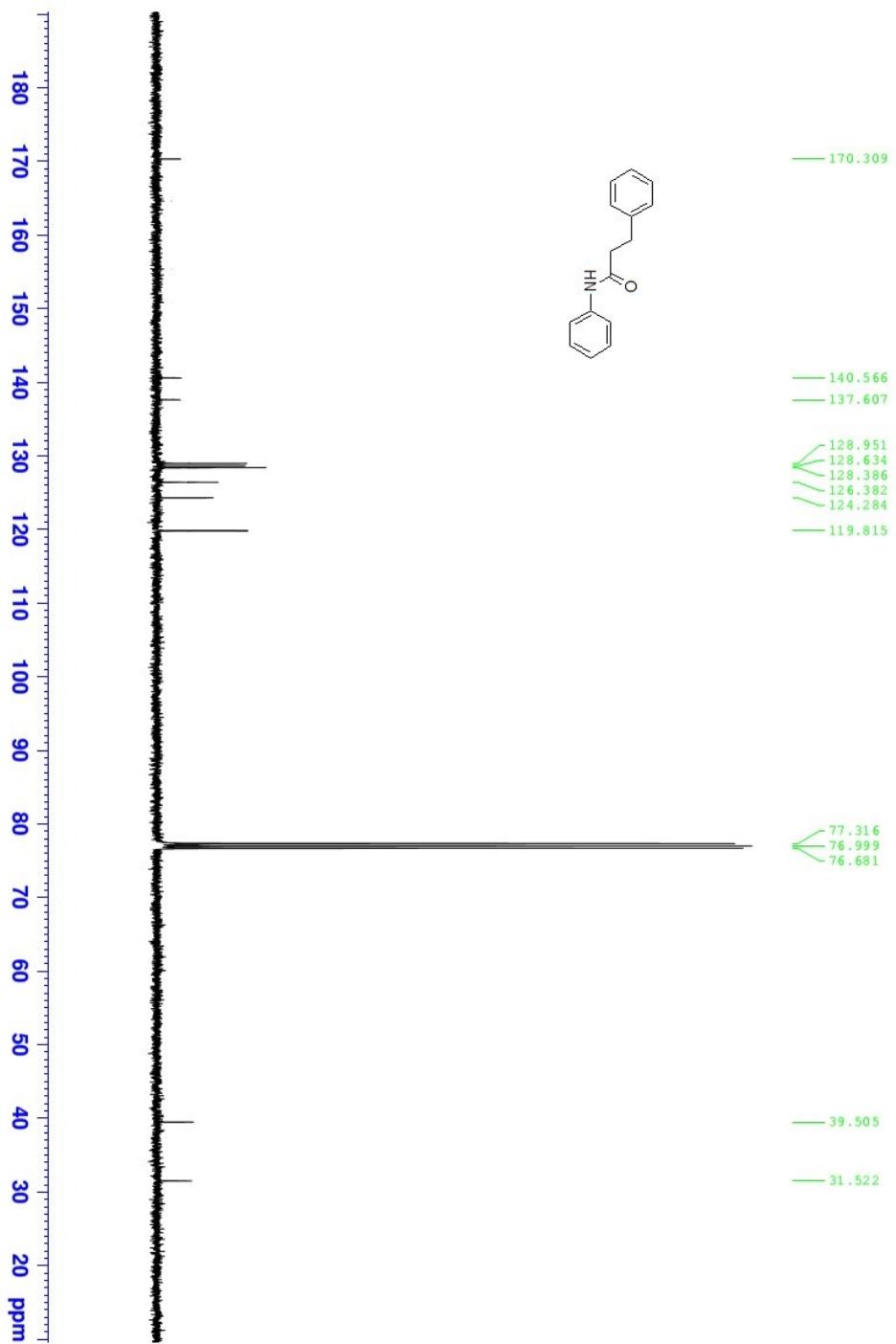
¹H NMR of Compound 3da



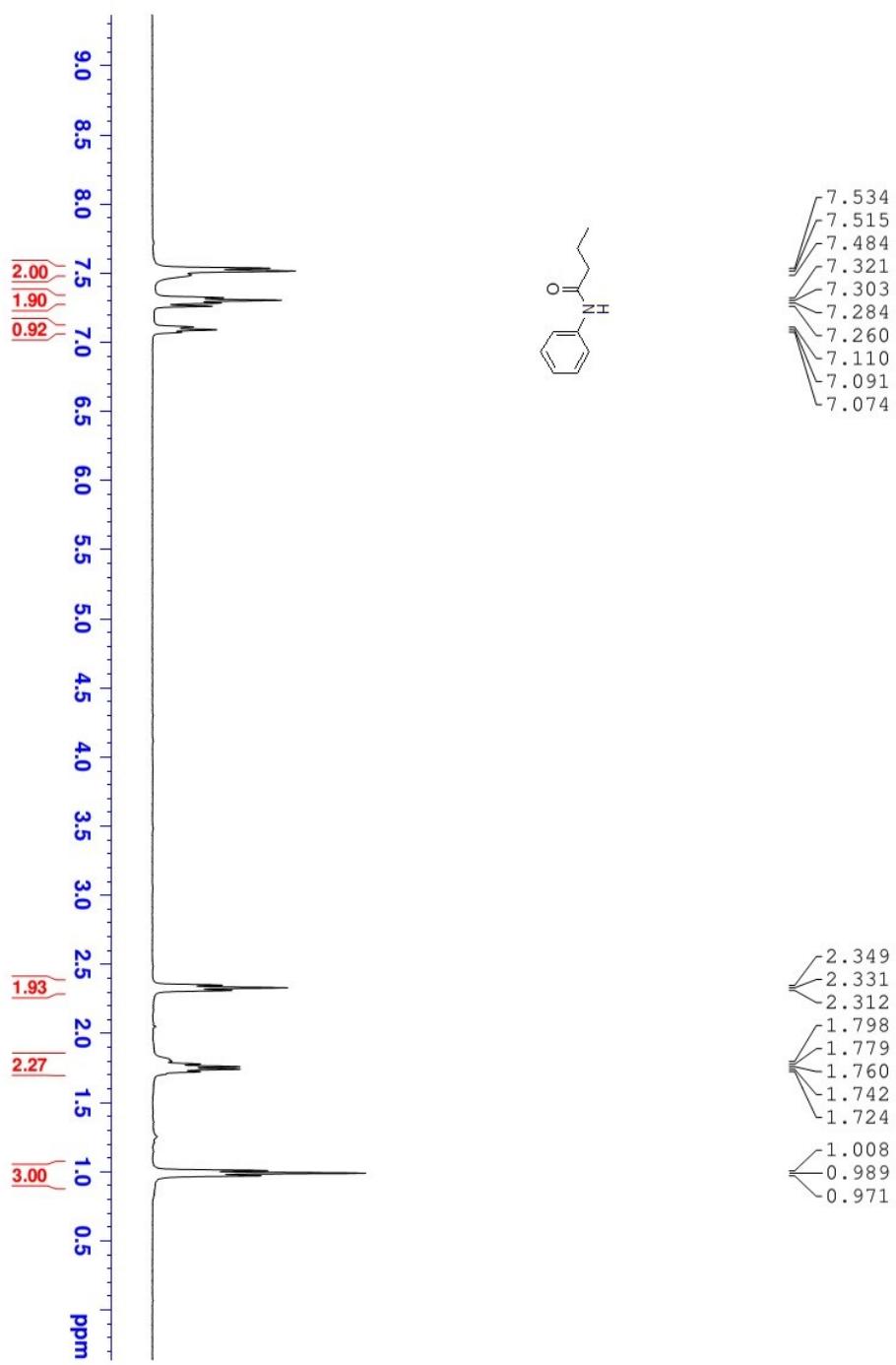
¹³C NMR of Compound 3da



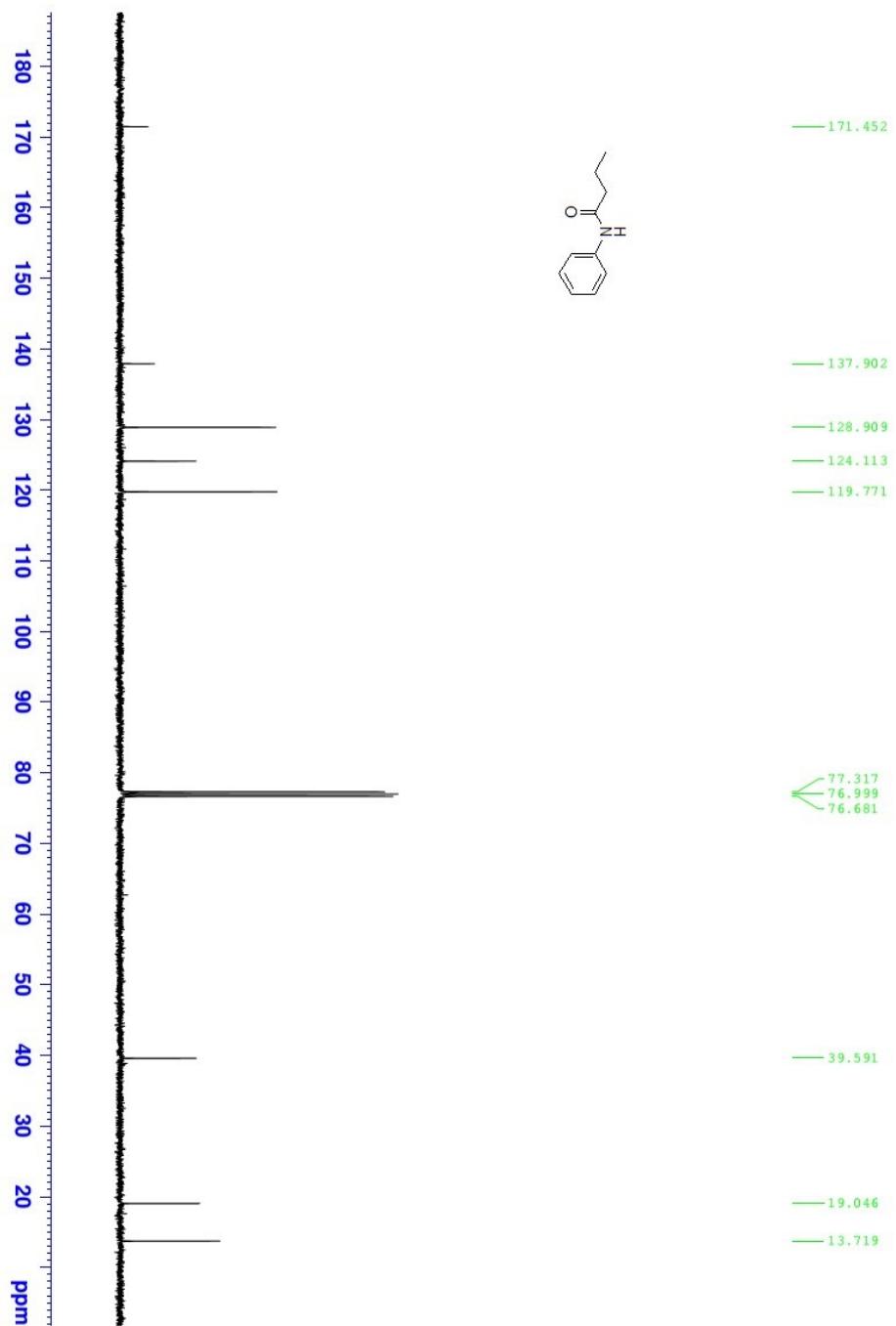
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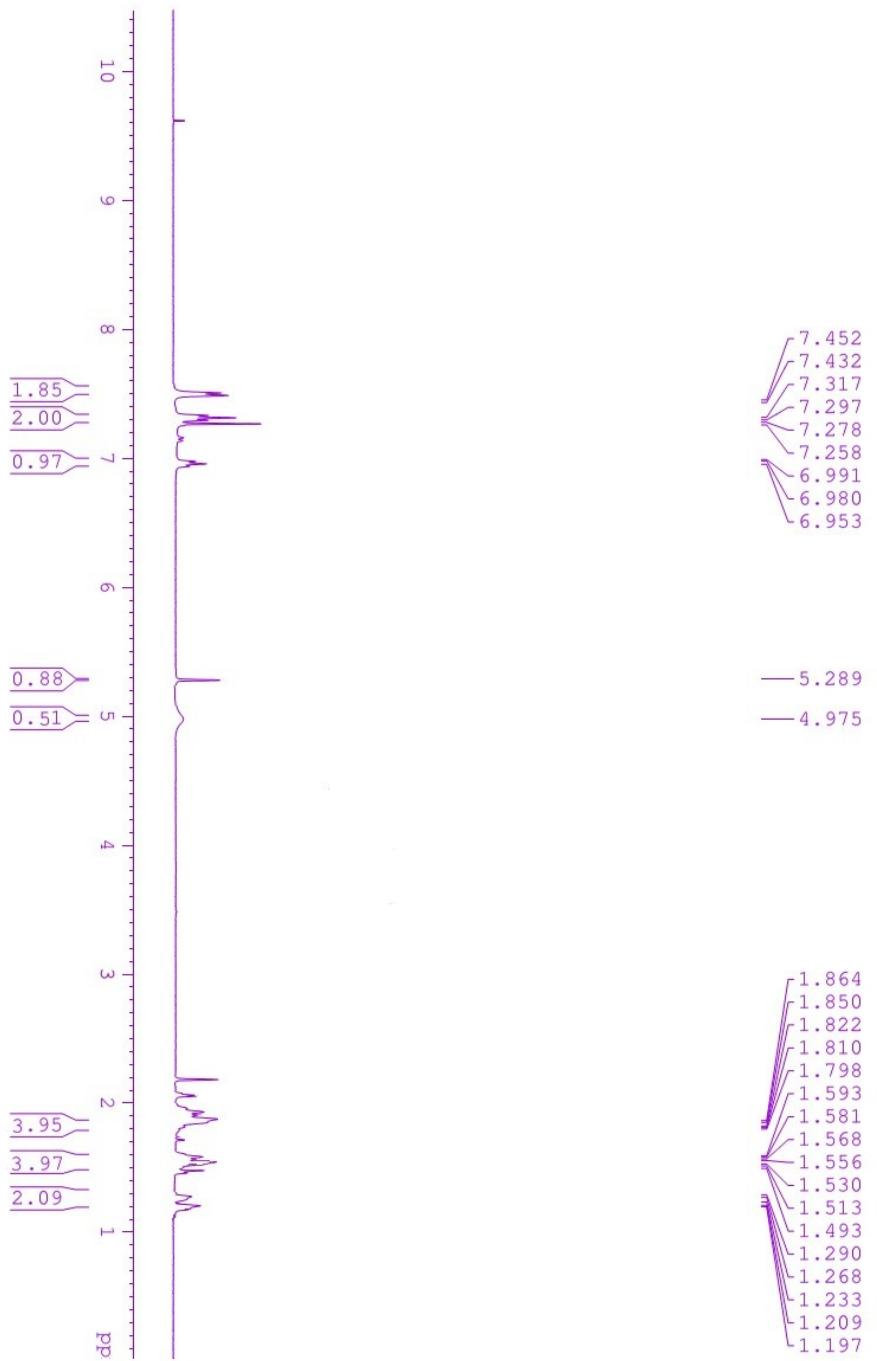
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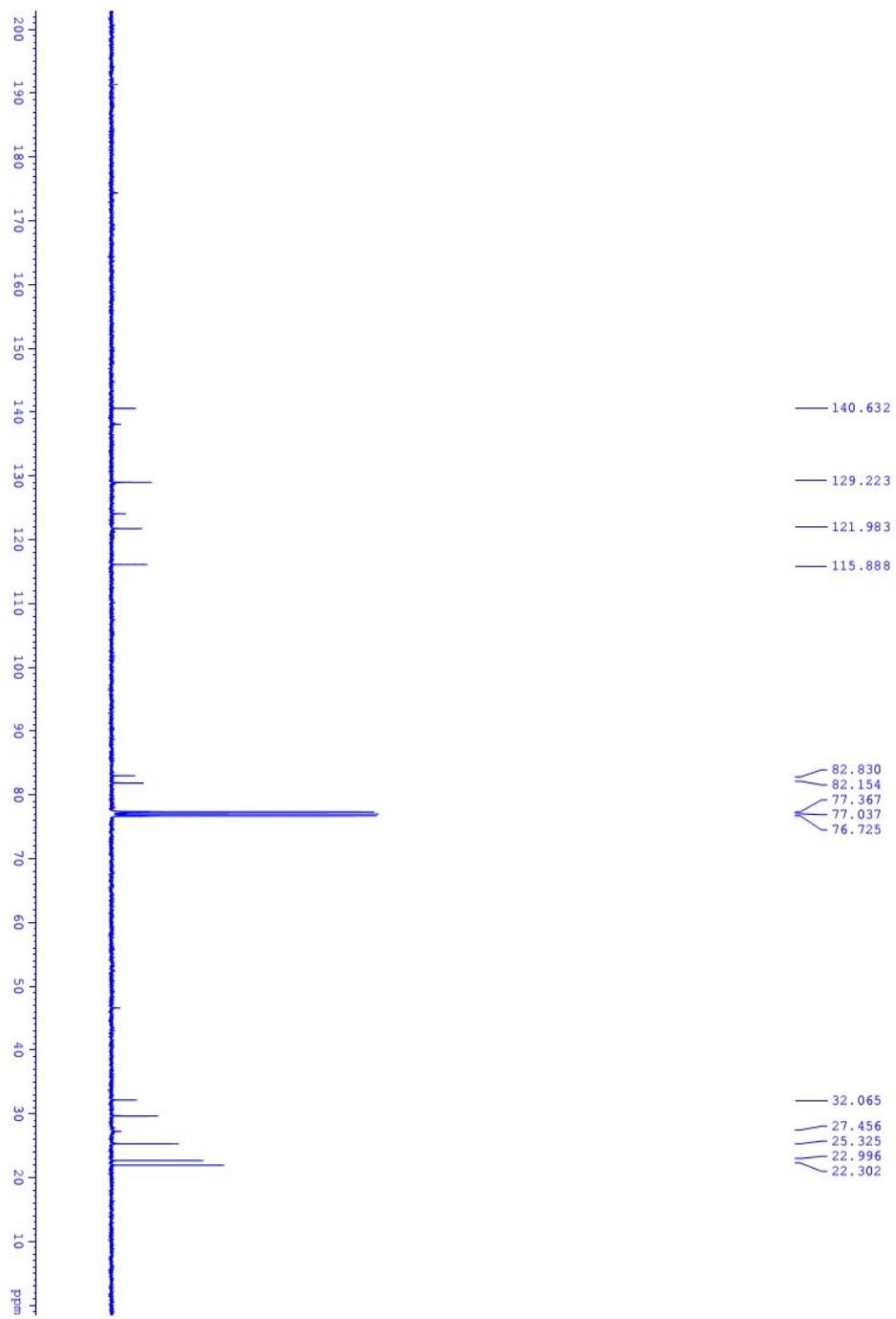
¹H NMR of Compound 3fa



^{13}C NMR of Compound 3fa



¹H NMR of Compound 4



¹³C NMR of Compound 4