Electronic Supplementary Material (ESI) for Green Chemistry.

Acylation of indoles via photoredox catalysis: A route to 3-acylindoles

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Supporting Information

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(A) Materials and equipment

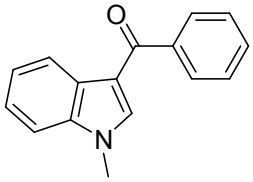
Reagents were obtained commercially and used as received. Solvents were purified and dried by standard methods. All title products were characterized by Infrared (IR), MS, ^{1}H NMR, ^{13}C NMR and High Resolution mass spectrometer (HRMS). IR spectra were reported in frequency of the absorption (cm^{-1}). ^{1}H NMR spectra were recorded on 400 MHz in $CDCl_3$ or DMSO-d_6, and ^{13}C NMR spectra were recorded on 100 MHz in $CDCl_3$ or DMSO-d_6 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) Typical experimental procedure

Typical Experimental Procedure for the Synthesis of compounds 3:

To a Schlenk tube were added α-oxo acids **1** (0.3 mmol), indoles **2** (0.3 mmol), **PC1** (3 mol%), DMF (2.0 mL), dtbbpy (15 mol%), $NiCl_2$·glyme (10 mol%), I_2 (0.3 mmol), Cs_2CO_3 (0.6 mmol), LiF (0.3 mmol). Then the tube was charged with argon, and was stirred at room temperature with the irradiation of a 35 W blue LED for about 60 h. After the reaction was finished, the reaction mixture was diluted in 35 mL ethyl acetate, washed with a saturated solution of brine (8 mL ×2), a solution of $Na_2S_2O_3$ (8 mL), a saturated solution of brine (8 mL ×2), saturated $NaHCO_3$ (10 mL), a saturated solution of brine (8 mL), dried (Na_2SO_4) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired products **3**.

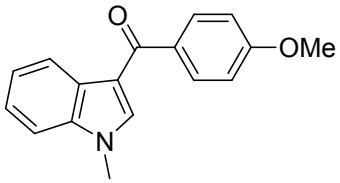
(C) Analytical data



3aa

(1-Methyl-1H-indol-3-yl)(phenyl)methanone (3aa):

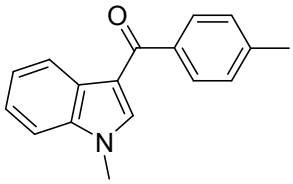
^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.37-8.34 (m, 1H), 7.73 (d, *J* = 6.8 Hz, 2H), 7.73-7.38 (m, 4H), 7.29 (dd, *J* = 3.6 Hz, *J* = 4.0 Hz, 3H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 190.9, 140.9, 137.9, 137.5, 131.1, 128.6, 128.3, 127.1, 123.6, 122.74, 122.73, 115.5, 109.6, 33.6, IR (neat cm^{-1}): 1657 (C=O); LRMS (EI 70 ev) *m/z* (%): 235 (M^+, 100); HRMS m/z (ESI) calcd for $C_{16}H_{13}NNaO$ $(M+Na)^+$ 258.0889, found 258.0897.



3ab

(4-Methoxyphenyl)(1-methyl-1H-indol-3-yl)methanone (3ab):

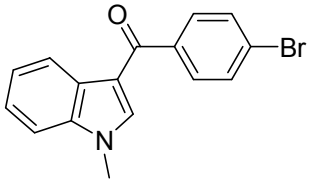
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.31 (d, J = 7.6 Hz, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.47 (s, 1H), 7.31-7.23 (m, 3H), 6.92 (d, J = 8.4 Hz, 2H), 3,81 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.7, 162.1, 137.4, 137.0, 133.4, 130.8, 127.3, 123.4, 122.6, 122.4, 115.6, 113.4, 109.5, 55.4, 33.5; IR (neat cm^{-1}): 1643 (C=O); LRMS (EI 70 ev) *m/z* (%): 265 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{15}NNaO_2$ $(M+Na)^+$ 288.0994, found 288.1002.



3ac

(1-Methyl-1H-indol-3-yl)(p-tolyl)methanone (3ac):

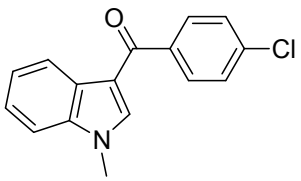
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.43-8.41 (m, 1H), 7.76 (d, J = 8.0 Hz, 2H), 7.50 (s, 1H), 7.37-7.34 (m, 3H), 7.30 (d, J = 7.2 Hz, 2H), 3.80 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.5, 141.4, 138.0, 137.5, 137.2, 128.7, 128.5, 127.1, 123.6, 122.6, 122.4, 115.5, 109.6, 33.2, 21.3; IR (neat cm^{-1}): 1649 (C=O); LRMS (EI 70 ev) *m/z* (%): 249 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{15}NNaO$ $(M+Na)^+$ 272.1046, found 272.1041.



3ad

(4-Bromophenyl)(1-methyl-1H-indol-3-yl)methanone (3ad):

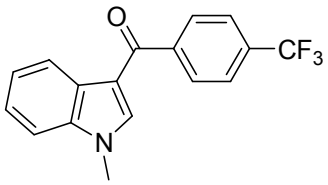
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.40 (d, J = 5.2 Hz, 1H), 7.68 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 8.0 Hz, 2H), 7.48 (s, 1H), 7.35 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.3, 139.5, 137.6, 137.4, 131.4, 130.1, 126.9, 125.6, 123.7, 122.7, 122.5, 115.2, 109.6, 33.5; IR (neat cm^{-1}): 1663 (C=O); LRMS (EI 70 ev) *m/z* (%): 313 (M^+, 71); HRMS m/z (ESI) calcd for $C_{16}H_{12}BrNNaO$ $(M+Na)^+$ 335.9994, found 336.0001.



3ae

(4-Chlorophenyl)(1-methyl-1H-indol-3-yl)methanone (3ae):

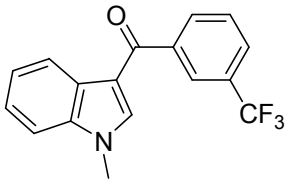
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.39-8.36 (m, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.49 (s, 1H), 7.45 (d, J = 8.4 Hz, 2H), 7.35-7.32 (m, 3H), 3.86 (s, 3H); 189.1, 138.9, 137.6, 137.5, 137.1, 130.1, 128.5, 127.1, 123.6, 122.6, 122.3, 115.3, 109.8, 33.2; IR (neat cm^{-1}): 1664 (C=O); LRMS (EI 70 ev) *m/z* (%):269 (M^+, 100); HRMS m/z (ESI) calcd for $C_{16}H_{12}ClNNaO$ $(M+Na)^+$ 292.0500, found 292.0504.



3af

(4-(Trifluoromethyl)phenyl)(1-methyl-1H-indol-3-yl)methanone (3af):

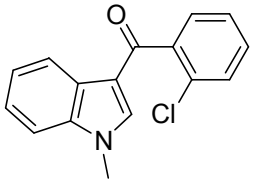
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.42 (d, J = 5.6 Hz, 1H), 7.90 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H), 7.49 (s, 1H), 7.39 (d, J = 8.0 Hz, 3H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.4, 144.0, 138.0, 137.6, 129.8, 129.7, 128.8, 128.55, 128.52, 126.9, 126.89, 126.86, 125.37, 125.33, 124.0, 123.1, 122.7, 115.4, 109.7, 33.6; IR (neat cm^{-1}): 1666 (C=O); LRMS (EI 70 ev) *m/z* (%): 303 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{12}F_3NNaO$ $(M+Na)^+$ 326.0763, found 326.0771.



3ag

(3-(Trifluoromethyl)phenyl)(1-methyl-1H-indol-3-yl)methanone (3ag):

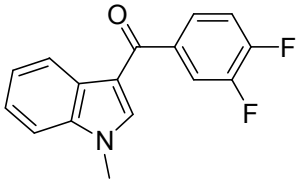
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.37 (d, J = 6.8 Hz, 1H), 8.07 (s, 1H), 7.99 (d, J = 7.2 Hz, 1H), 7.81 (d, J = 7.6 Hz, 1H), 7.64 (t, J = 7.6 Hz, 1H), 7.49 (s, 1H), 7.38 (s, 3H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.0, 137.7, 137.6, 131.7, 130.6, 128.9, 127.5, 127.0, 125.4, 125.3, 123.9, 123.0, 122.6, 122.5, 115.2, 109.7, 33.6; IR (neat cm^{-1}): 1654 (C=O); LRMS (EI 70 ev) *m/z* (%): 303 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{12}F_3NNaO$ $(M+Na)^+$ 326.0763, found 326.0769.



3ah

(2-Chlorophenyl)(1-methyl-1H-indol-3-yl)methanone (3ah):

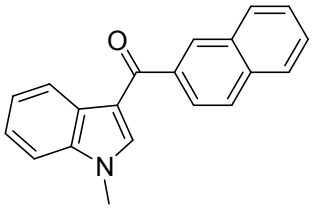
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.37 (d, J = 5.6 Hz, 1H), 7.48-7.42 (m, 3H), 7.40-7.36 (m, 4H), 7.34-7.30 (m, 1H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.7, 140.5, 138.8, 137.7, 130.9, 130.2, 130.0, 128.6, 126.44, 126.41, 123.8, 123.0, 122.6, 116.4, 109.7, 33.6; IR (neat cm^{-1}): 1649 (C=O); LRMS (EI 70 ev) *m/z* (%): 269 (M^+, 100); HRMS m/z (ESI) calcd for $C_{16}H_{12}ClNNaO$ $(M+Na)^+$ 292.0500, found 292.0508.



3ai

(3,4-Difluorophenyl)(1-methyl-1H-indol-3-yl)methanone (3ai):

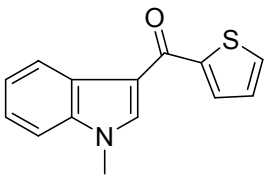
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (d, J = 7.2 Hz, 1H), 7.58 (d, J = 9.2 Hz, 1H), 7.48 (s, 1H), 7.42 (s, 1H), 7.28 (s, 3H), 7.19 (dd, J = 10.0 Hz, J = 8.4 Hz, 1H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 187.7; 153.5, 153.4, 151.3, 151.2, 151.0, 150.9, 148.8, 148.7, 137.7, 137.66, 137.62, 137.5, 126.9, 125.3, 125.26, 125.23, 125.1, 123.8, 122.8, 122.5, 118.0, 117.8, 117.2, 117.0, 114.9, 109.7, 33.5; IR (neat cm^{-1}): 1649 (C=O); LRMS (EI 70 ev) *m/z* (%): 271 (M^+, 100); HRMS m/z (ESI) calcd for $C_{16}H_{11}F_2NNaO$ $(M+Na)^+$ 294.0701, found 294.0708.



3aj

(1-Methyl-1H-indol-3-yl)(naphthalen-3-yl)methanone (3aj):

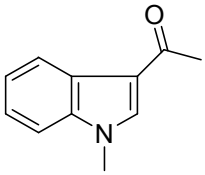
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.42 (s, 1H), 8.21 (s, 1H), 7.87-7.84 (m, 4H), 7.51-7.47 (s, 3H), 7.30 (s, 3H), 3.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.5, 138.0, 137.9, 137.2, 134.6, 132.4, 129.1, 128.8, 128.1, 127.6, 127.0, 126.4, 125.4, 123.6, 122.5, 122.4, 115.5, 109.6, 33.5; IR (neat cm^{-1}): 1650 (C=O); LRMS (EI 70 ev) *m/z* (%): 241 (M^+, 100); HRMS m/z (ESI) calcd for $C_{20}H_{15}NNaO$ $(M+Na)^+$ 308.1046, found 308.1050.



3ak

(1-Methyl-1H-indol-3-yl)(thiophen-2-yl)methanone (3ak):

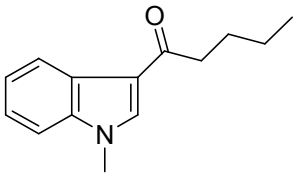
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.43-8.41 (m, 1H), 7.77 (s, 1H), 7.71 (d, J = 3.6 Hz, 1H), 7.57 (d, J = 4.4 Hz, 1H), 7.32 (s, 3H), 7.15 (t, J = 4.0 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.5, 145.1, 137.3, 136.1, 131.2, 130.7, 127.5, 127.0, 123.4, 122.2, 120.1, 115.1, 109.8, 33.2; IR (neat cm^{-1}): 1638 (C=O); LRMS (EI 70 ev) *m/z* (%): 263 (M^+, 100); HRMS m/z (ESI) calcd for $C_{14}H_{11}NNaOS$ $(M+Na)^+$ 264.0453, found 264.0459.



3al

1-(1-Methyl-1H-indol-3-yl)ethanone (3al):

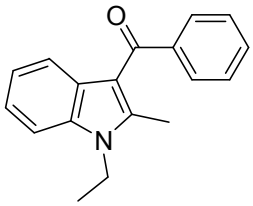
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.38 (dd, J = 8.0 Hz, J = 2.4 Hz, 1H), 7.63 (s, 1H), 7.31-7.27 (m, 3H), 3.77 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 192.9, 137.0, 135.5, 125.9, 123.2, 122.6, 122.1, 116.5, 109.6, 33.4, 27.2. IR (neat cm^{-1}): 1647 (C=O); LRMS (EI 70 ev) *m/z* (%): 173 (M^+, 100); HRMS m/z (ESI) calcd for $C_{11}H_{11}NNaO$ $(M+Na)^+$ 196.0734, found 196.0730.



3am

1-(1-Methyl-1H-indol-3-yl)pentan-1-one (3am):

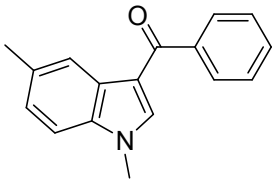
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.43 (d, J = 5.2 Hz, 1H), 7.66 (s, 1H), 7.17 (d, J = 8.4 Hz, 3H), 3.77 (s, 3H), 2.83 (dd, J = 1.6 Hz, J = 6.8 Hz, 2H), 1.80-1.73 (m, 2H), 1.46-1.41 (m, 2H), 0.99 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.8, 137.2, 135.1, 126.1, 123.0, 122.3, 122.2, 116.3, 109.4, 39.4, 33.2, 27.2, 22.5, 13.8; IR (neat cm^{-1}): 1651 (C=O); LRMS (EI 70 ev) *m/z* (%): 215 (M^+, 100); HRMS m/z (ESI) calcd for $C_{14}H_{17}NNaO$ $(M+Na)^+$ 238.1205, found 238.1212.



3ba

(1-Ethyl-2-methyl-1H-indol-3-yl)(phenyl)methanone (3ba):

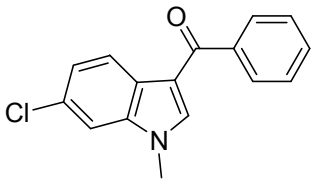
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.78 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H),7.47 (t, J = 7.6 Hz, 2H), 7.35 (dd, J = 8.4 Hz, J = 8.4 Hz, 2H), 7.23 (t, J = 7.6 Hz, 1H), 7.09 (t, J = 7.6 Hz, 1H), 4.24 (q, J = 7.2 Hz, 2H), 2.61 (s, 3H), 1.43 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 192.8, 143.8, 141.4, 135.3, 131.3, 128.9, 128.1, 127.2, 121.9, 121.2, 120.9, 113.6, 109.1, 37.9, 14.7, 12.1; IR (neat cm^{-1}): 1662 (C=O); LRMS (EI 70 ev) *m/z* (%): 263 (M^+, 100); HRMS m/z (ESI) calcd for $C_{18}H_{17}NNaO$ $(M+Na)^+$ 286.1203, found 286.1210.



3ca

(1,5-Dimethyl-1H-indol-3-yl)(phenyl)methanone (3ca):

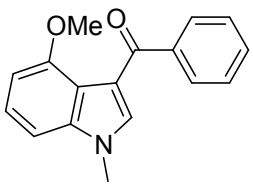
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.23 (s, 1H), 7.77 (d, J = 6.4 Hz, 2H), 7.51 (d, J = 7.6 Hz, 1H), 7.43 (t, J = 6.4 Hz, 3H), 7.24-7.18 (m, 2H), 3.77 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.4, 140.3, 138.1, 135.7, 132.2, 130.6, 128.4, 128.0, 127.2, 125.2, 122.2, 114.5, 109.0, 33.1, 21.2; IR (neat cm^{-1}): 1649 (C=O); LRMS (EI 70 ev) *m/z* (%): 249 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{15}NNaO$ $(M+Na)^+$ 272.1046, found 272.1053.



3da

(6-Chloro-1-methyl-1H-indol-3-yl)(phenyl)methanone (3da):

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.33 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 7.2 Hz, 2H), 7.53 (d, J = 6.8 Hz, 1H), 7.47 (s, 3H), 7.31-7.27 (m, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.4, 140.2, 138.0, 137.8, 131.2, 129.3, 128.5, 128.1, 125.4, 123.5, 123.1, 115.2, 109.6, 33.2; IR (neat cm^{-1}): 1671 (C=O); LRMS (EI 70 ev) *m/z* (%): 269 (M^+, 71); HRMS m/z (ESI) calcd for $C_{16}H_{12}ClNNaO$ $(M+Na)^+$ 292.0500, found 292.0494.

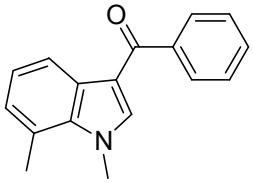


3ea

(4-Methoxy-1-methyl-1H-indol-3-yl)(phenyl)methanone (3ea):

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, J = 7.6 Hz, 2H), 7.47 (t, J = 7.4 Hz, 1H), 7.31-7.26 (m,

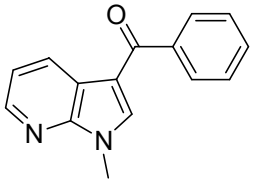
3H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 3.72 (s, 3H), 3.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 191.0, 154.3, 140.7, 139.1, 135.4, 131.3, 129.7, 127.4, 124.4, 116.6, 116.5, 102.8, 102.1, 55.4, 33.5; IR (neat cm^{-1}): 1649 (C=O); LRMS (EI 70 ev) *m/z* (%): 265 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{15}NNaO_2$ $(M+Na)^+$ 288.0994, found 288.0999.



3fa

(1,7-Dimethyl-1H-indol-3-yl)(phenyl)methanone (3fa):

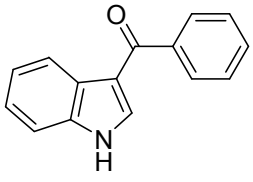
^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.79 (d, *J* = 7.2 Hz, 1H), 7.76 (d, *J* = 7.6 Hz, 2H), 7.55-7.51 (m, 1H), 7.45 (t, *J* = 7.2 Hz, 2H), 7.33 (s, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 7.2 Hz, 1H); 4.03 (s, 3H), 2.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 190.5, 140.7, 139.4, 136.2, 130.8, 128.6, 128.2, 128.0, 126.4, 122.7, 121.5, 120.6, 115.1, 37.5, 19.4; IR (neat cm^{-1}): 1667 (C=O); LRMS (EI 70 ev) *m/z* (%): 249 (M^+, 100); HRMS m/z (ESI) calcd for $C_{17}H_{15}NNaO$ $(M+Na)^+$ 272.1046, found 272.1055.



3ga

(1-Methyl-1H-pyrrolo[2,3-b]pyridin-3-yl)(phenyl)methanone (3ga):

^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.65 (d, *J* = 7.6 Hz, 1H), 8.44 (dd, *J* = 4.8 Hz, *J* = 0.4 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.67 (s, 1H), 7.56-7.53 (m, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.29-7.26 (m, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 190.3, 148.0, 144.3, 139.7, 137.2, 131.4, 131.0, 128.6, 128.3, 119.4, 118.2, 113.6, 32.0; IR (neat cm^{-1}): 1641 (C=O); LRMS (EI 70 ev) *m/z* (%): 236 (M^+, 100); HRMS m/z (ESI) calcd for $C_{15}H_{12}N_2NaO$ $(M+Na)^+$ 259.0842, found 259.0851.

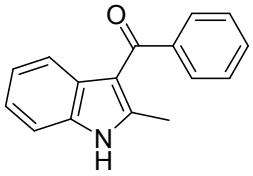


3ha

(1H-indol-3-yl)(phenyl)methanone (3ha):

^{1}H NMR (400 MHz, DMSO-d_6) *δ*: 12.06 (brs, 1H), 8.26 (t, *J* = 5.8 Hz, 1H), 7.91 (s, 1H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.62-7.52 (m, 4H), 7.29 (dd, *J* = 6.0 Hz, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) *δ*: 190.6, 140.8, 137.1, 136.3, 131.6, 128.9, 128.8, 126.6, 123.8, 122.5, 121.9, 115.4, 112.8;

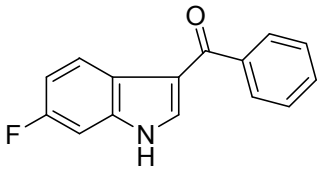
IR (neat cm^{-1}): 1648 (C=O); LRMS (EI 70 ev) *m/z* (%): 221 (M^+, 100); HRMS m/z (ESI) calcd for $C_{15}H_{11}NNaO$ $(M+Na)^+$ 244.0733, found 244.0740.



3ia

(2-Methyl-1H-indol-3-yl)(phenyl)methanone (3ia):

^1H NMR (400 MHz, DMSO-d_6) δ: 11.96 (brs, 1H), 7.60 (t, J = 7.2 Hz, 3H), 7.52 (t, J = 7.4 Hz, 2H), 7.40 (d, J = 8.0 Hz, 1H), 7.33 (d, J = 8.0 Hz, 1H), 7.13 (t, J = 7.6 Hz, 1H), 7.02 (t, J = 7.6 Hz, 1H); 2.37 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 192.3, 145.0, 142.0, 135.4, 131.5, 128.8, 128.4, 127.7, 122.3, 121.4, 120.4, 112.9, 111.7, 14.6; IR (neat cm^{-1}): 1640 (C=O); LRMS (EI 70 ev) *m/z* (%): 235 (M^+, 100); HRMS m/z (ESI) calcd for $C_{16}H_{13}NNaO$ $(M+Na)^+$ 258.0889, found 258.0881.

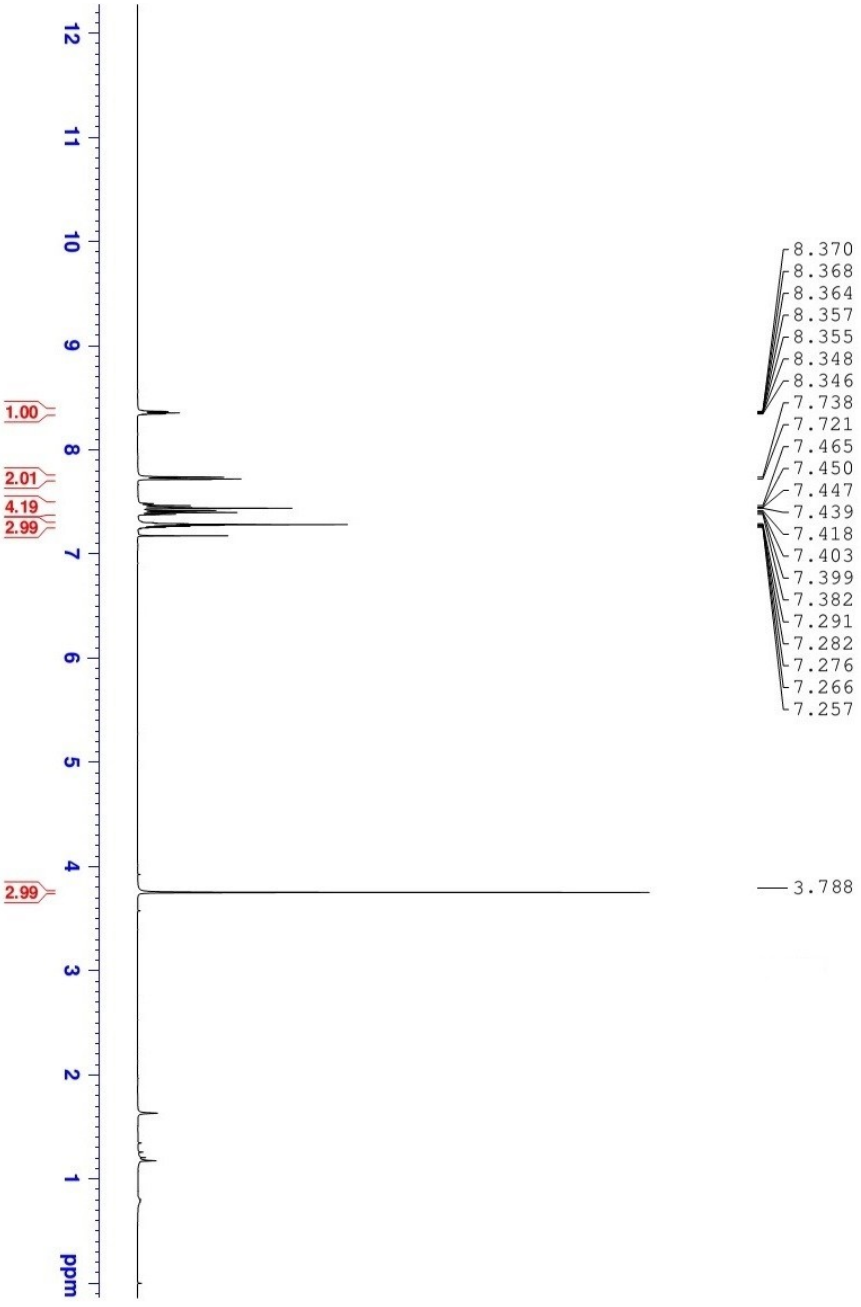


3ja

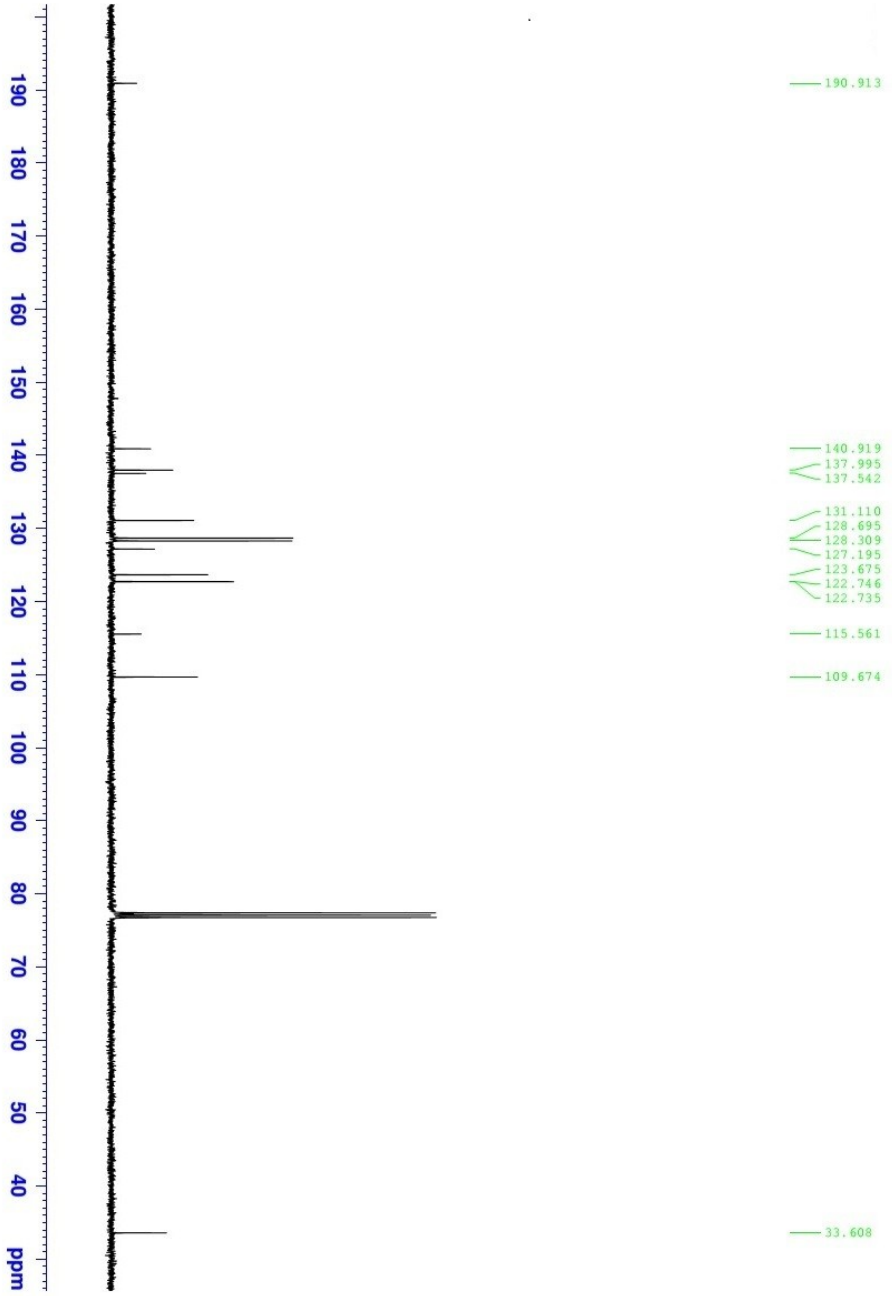
(6-Fluoro-1H-indol-3-yl)(phenyl)methanone (3ja):

^1H NMR (400 MHz, DMSO-d_6) δ: 12.10 (brs, 1H), 8.25 (t, J = 7.2 Hz, 1H), 7.93 (s, 1H), 7.91 (d, J = 7.2 Hz, 2H), 7.61-7.52 (m, 3H), 7.33 (d, J = 6.0 Hz, 1H), 7.13 (t, J = 9.4 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 190.5, 161.0, 158.7, 140.5, 137.3, 137.1, 136.9, 131.7, 128.9, 128.8, 123.3, 123.1, 123.0, 115.3, 110.8, 110.6, 99.1, 98.8; IR (neat cm^{-1}): 1655 (C=O); LRMS (EI 70 ev) *m/z* (%): 239 (M^+, 100); HRMS m/z (ESI) calcd for $C_{15}H_{10}FNNaO$ $(M+Na)^+$ 262.0638, found 262.0644.

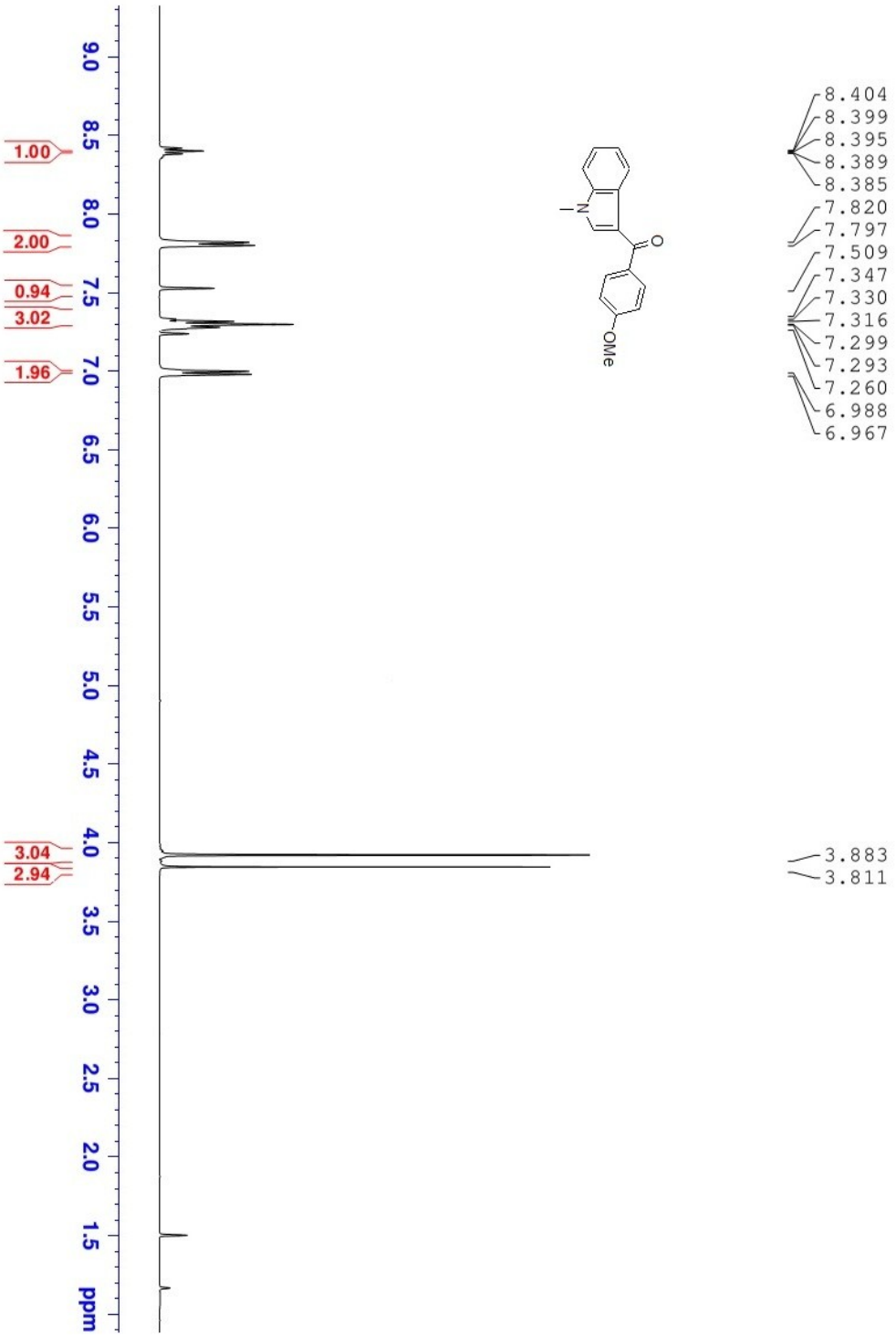
(D) **Spectra**



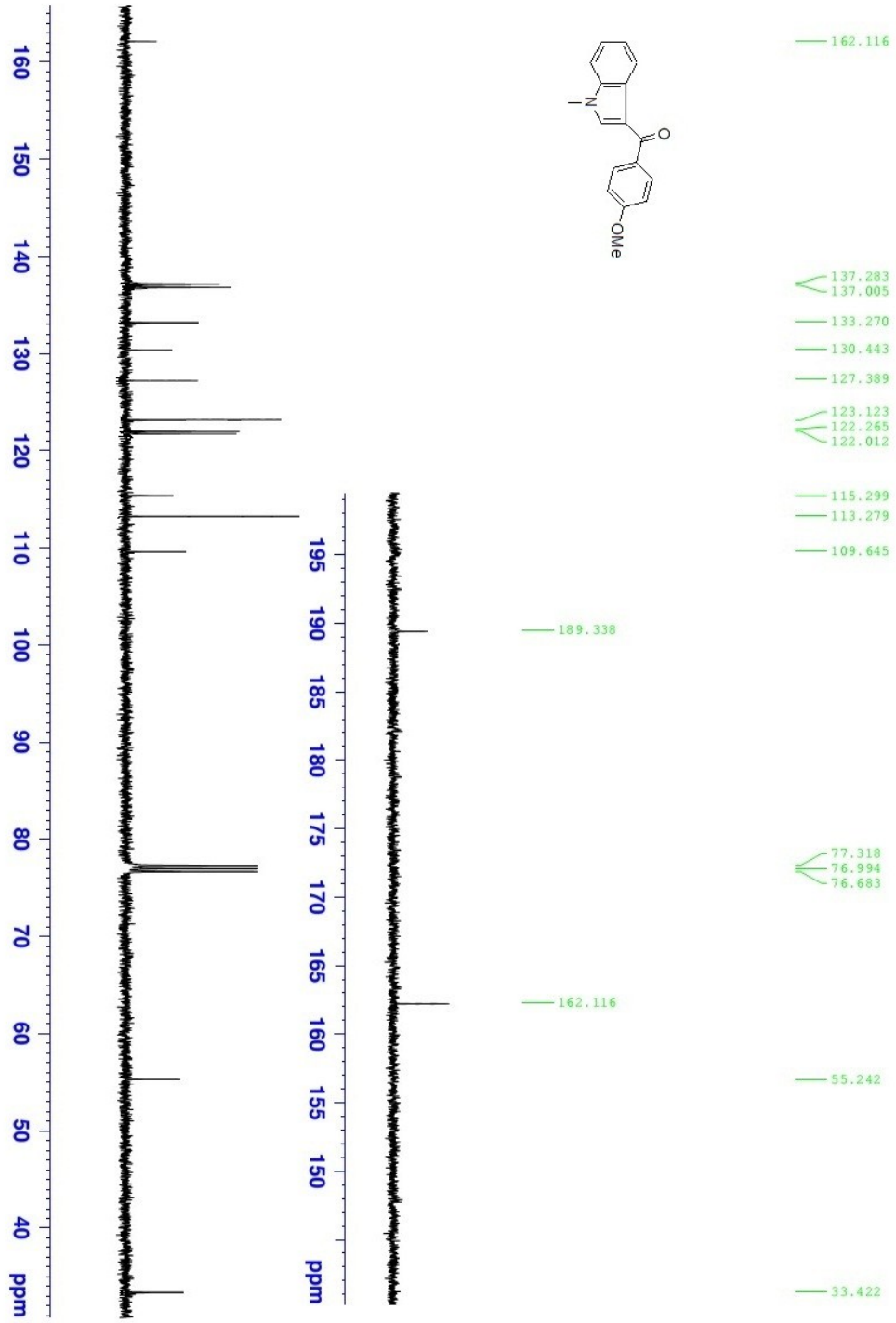
^{1}H NMR of Compound 3aa



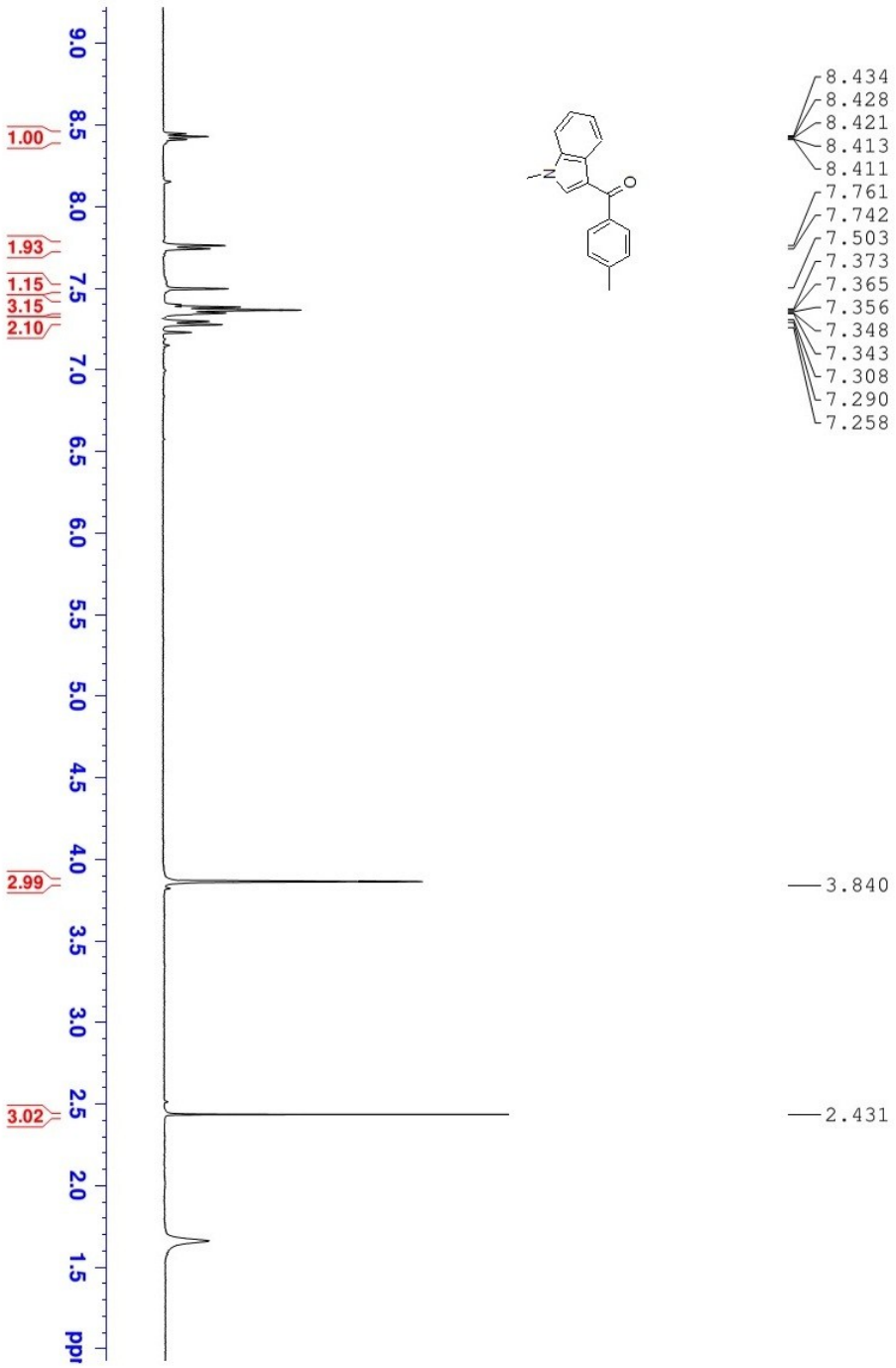
^{13}C NMR of Compound 3aa



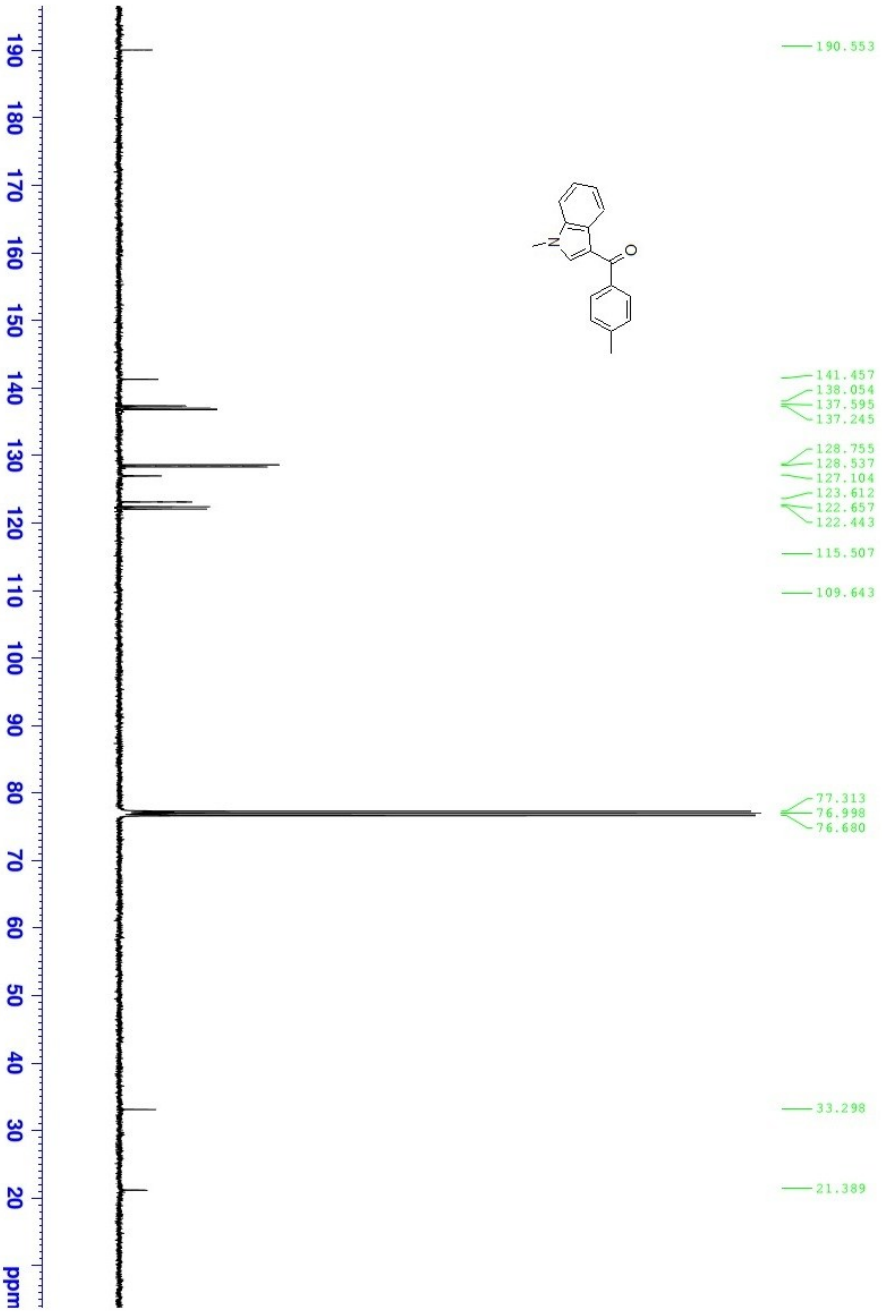
^{1}H NMR of Compound 3ab



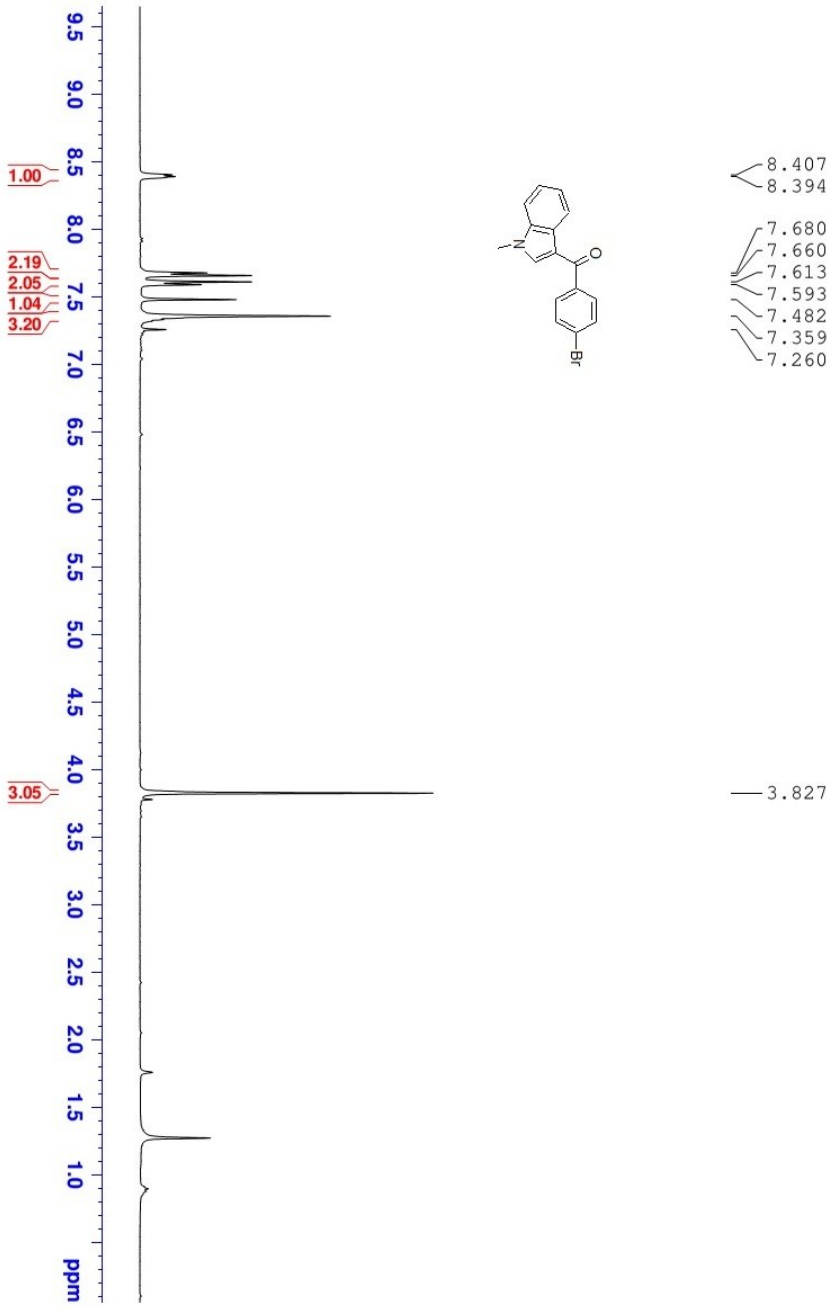
^{13}C NMR of Compound 3ab



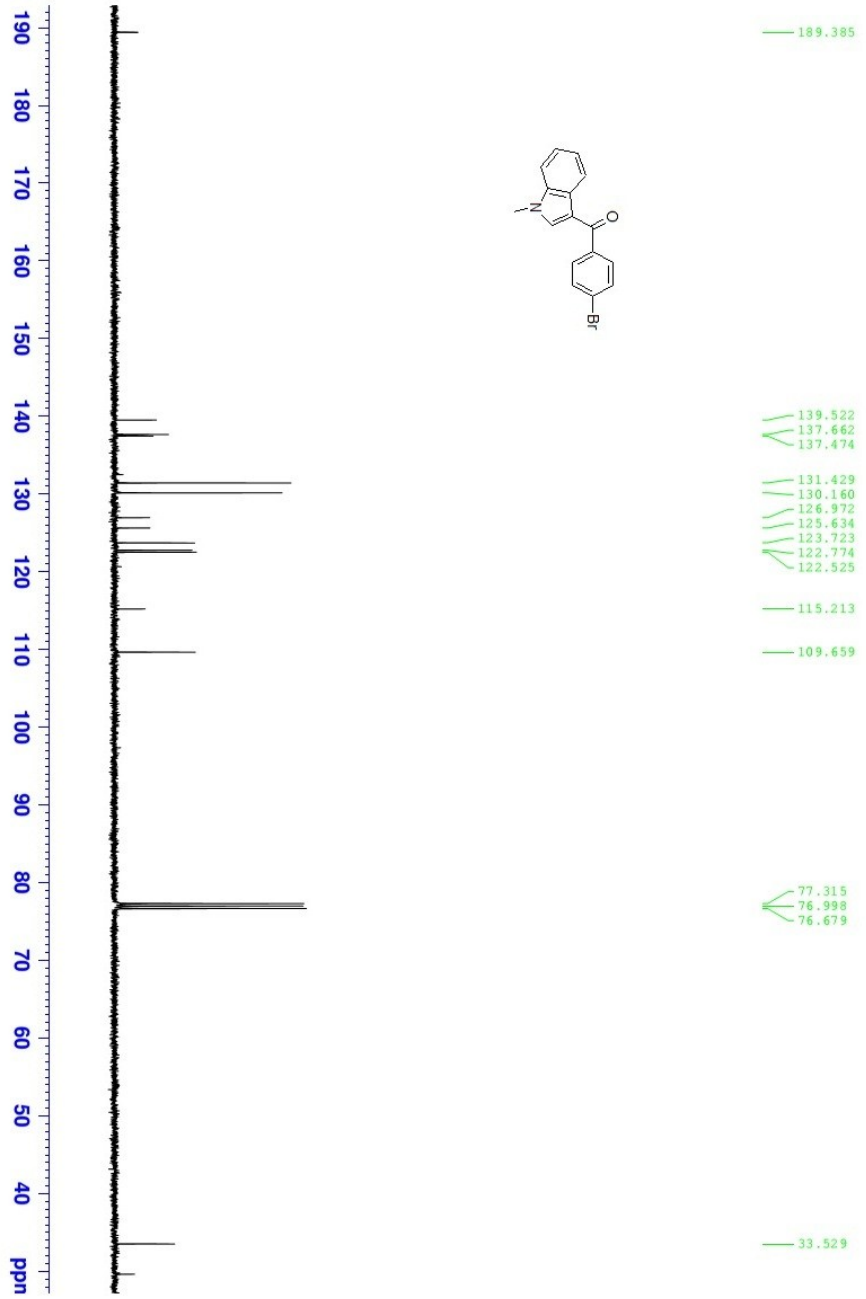
^{1}H NMR of Compound 3ac



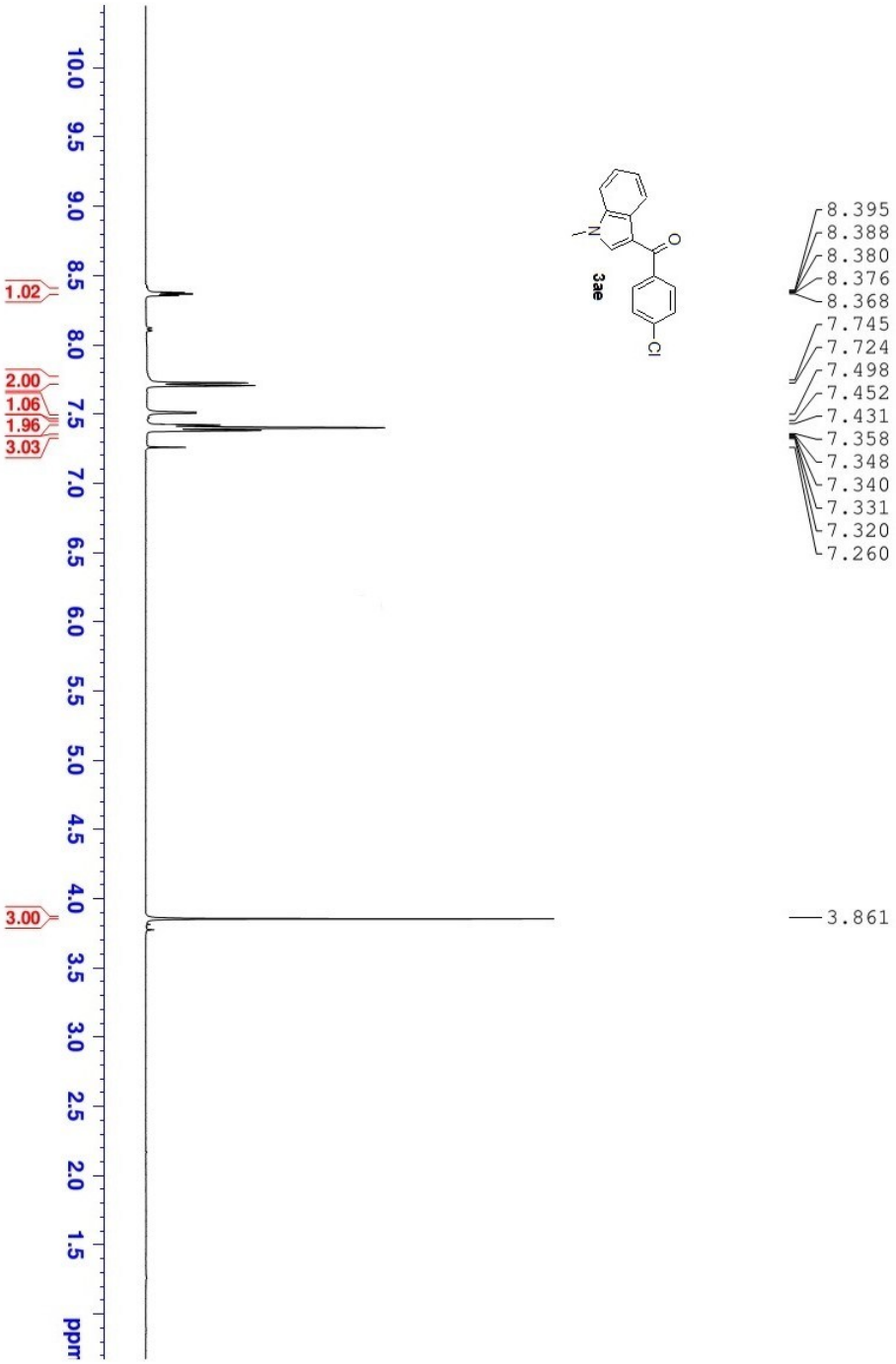
^{13}C NMR of Compound 3ac



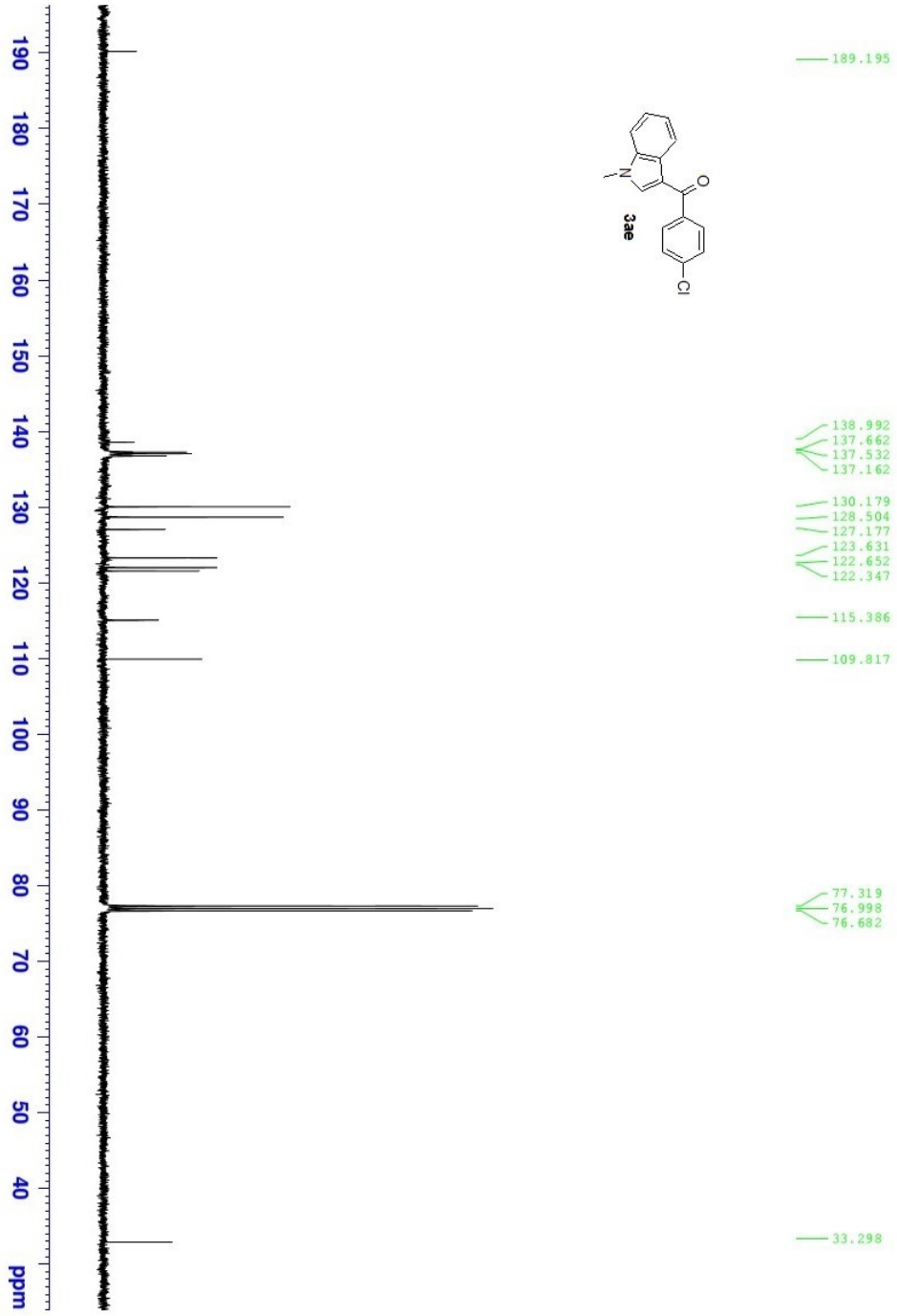
^{1}H NMR of Compound 3ad



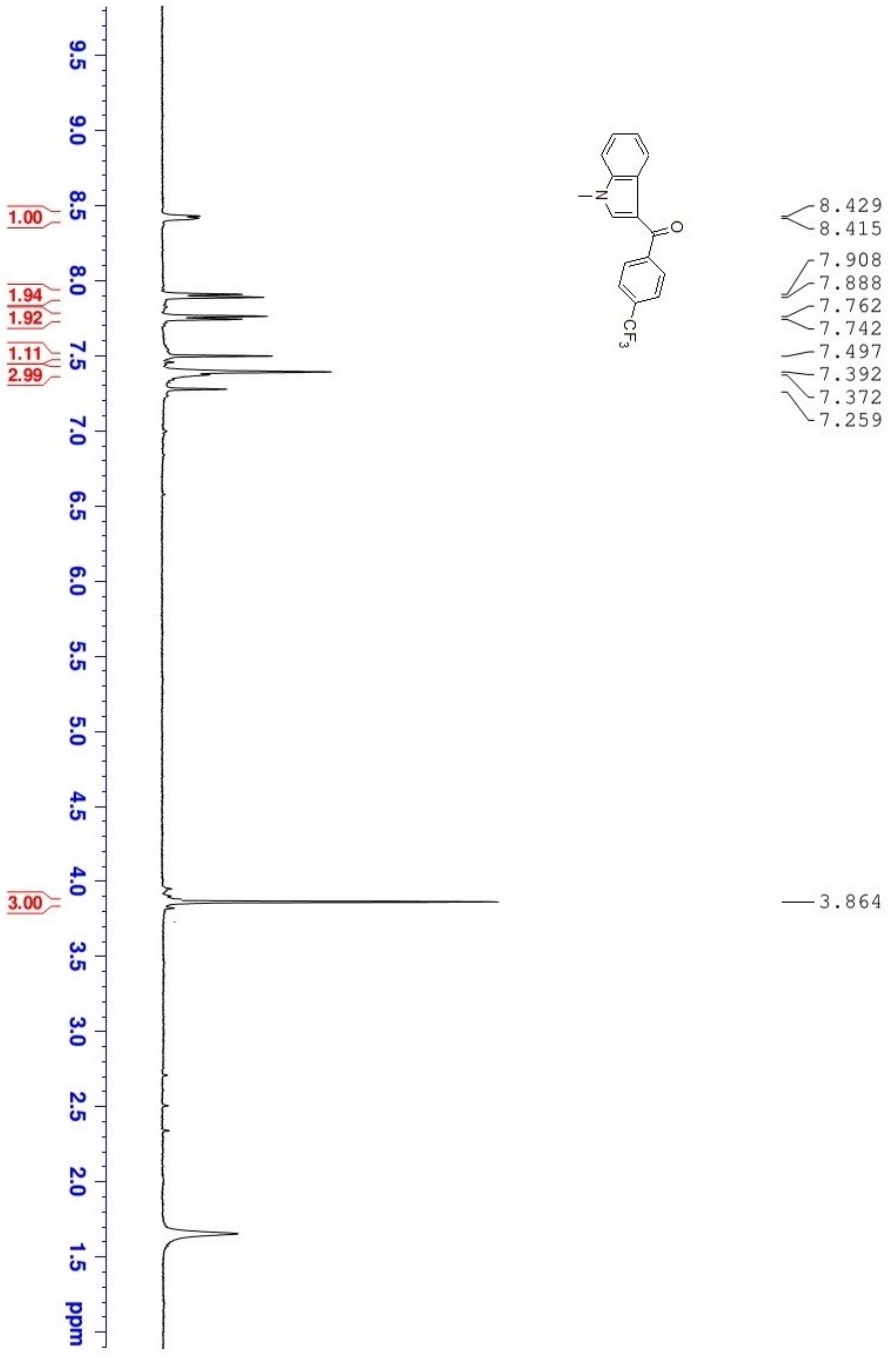
^{13}C NMR of Compound 3ad



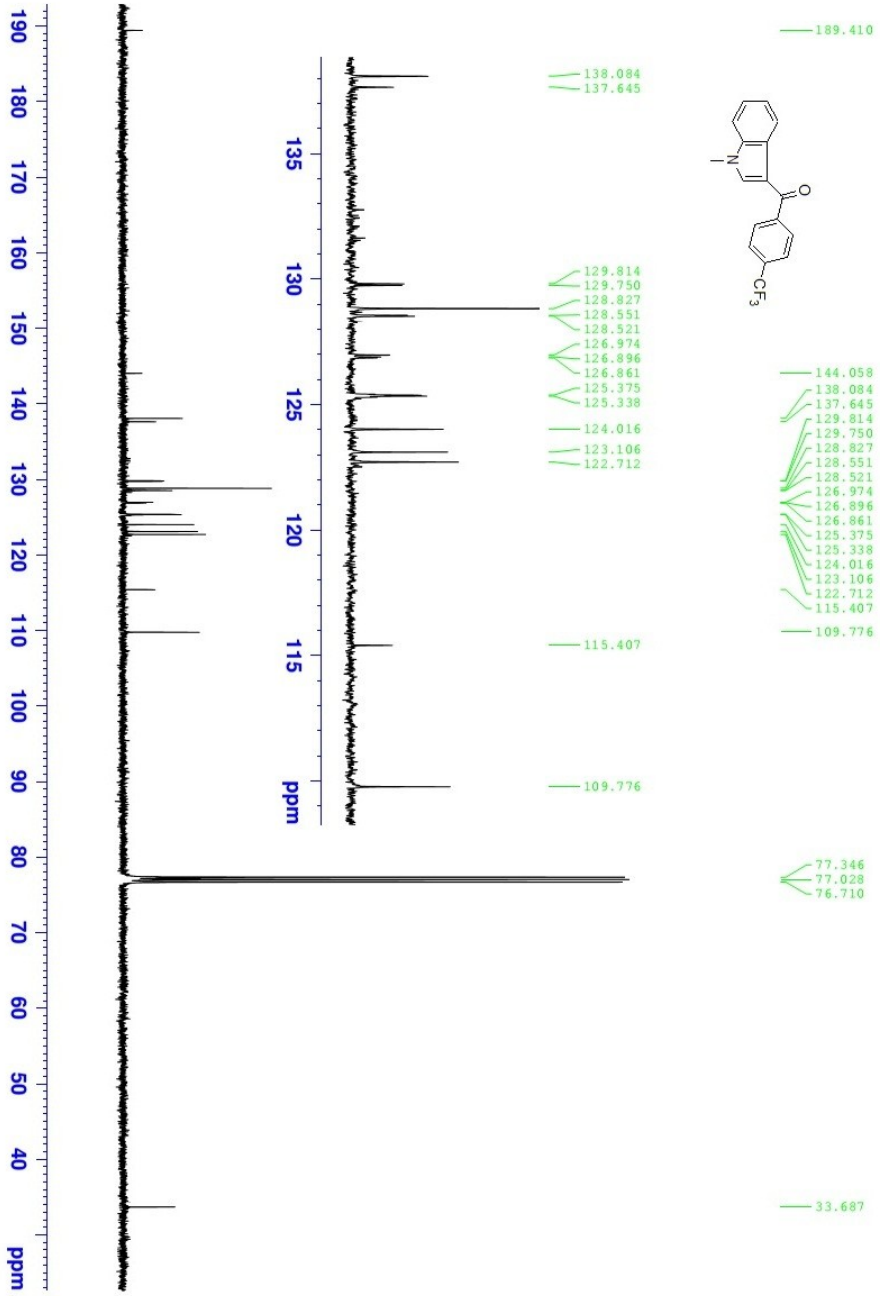
^{1}H NMR of Compound 3ae



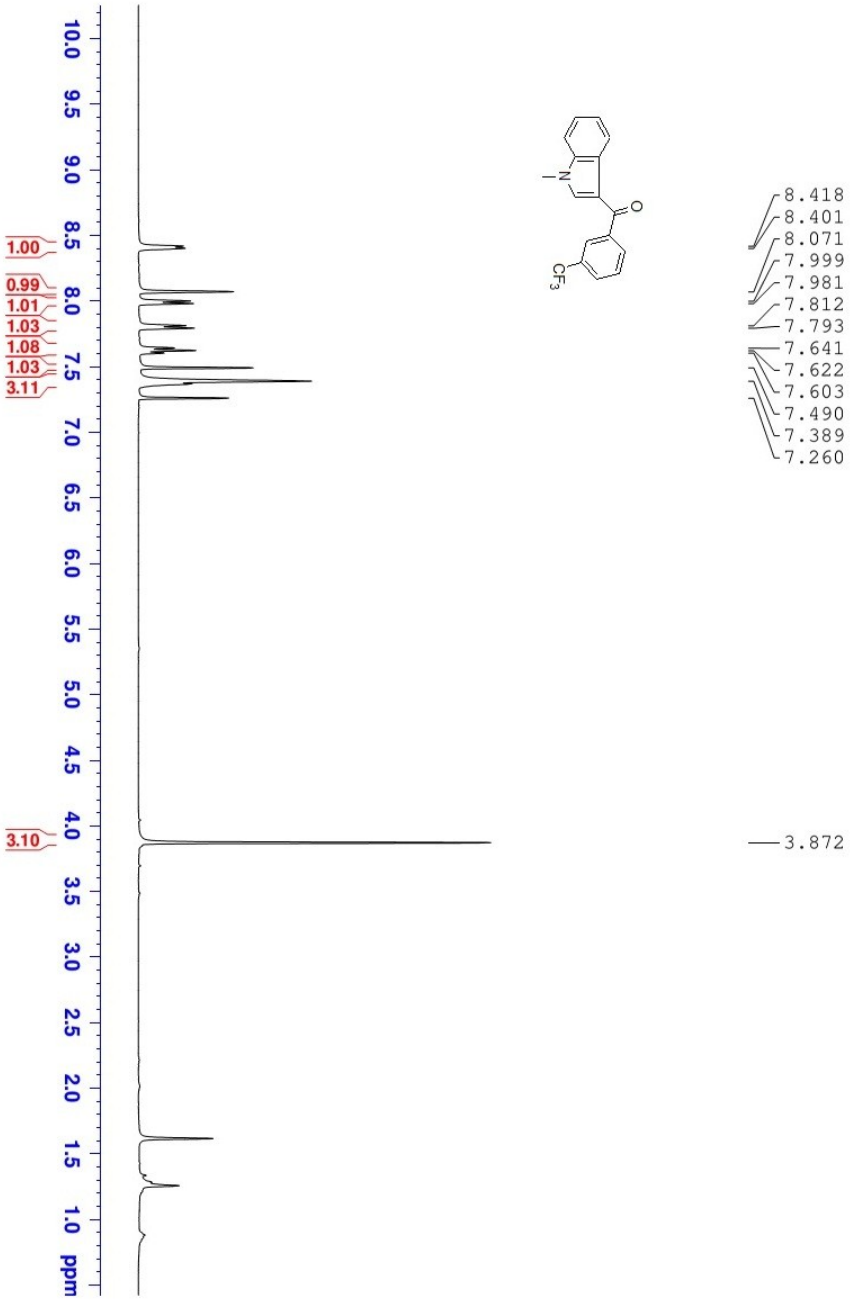
^{13}C NMR of Compound 3ae



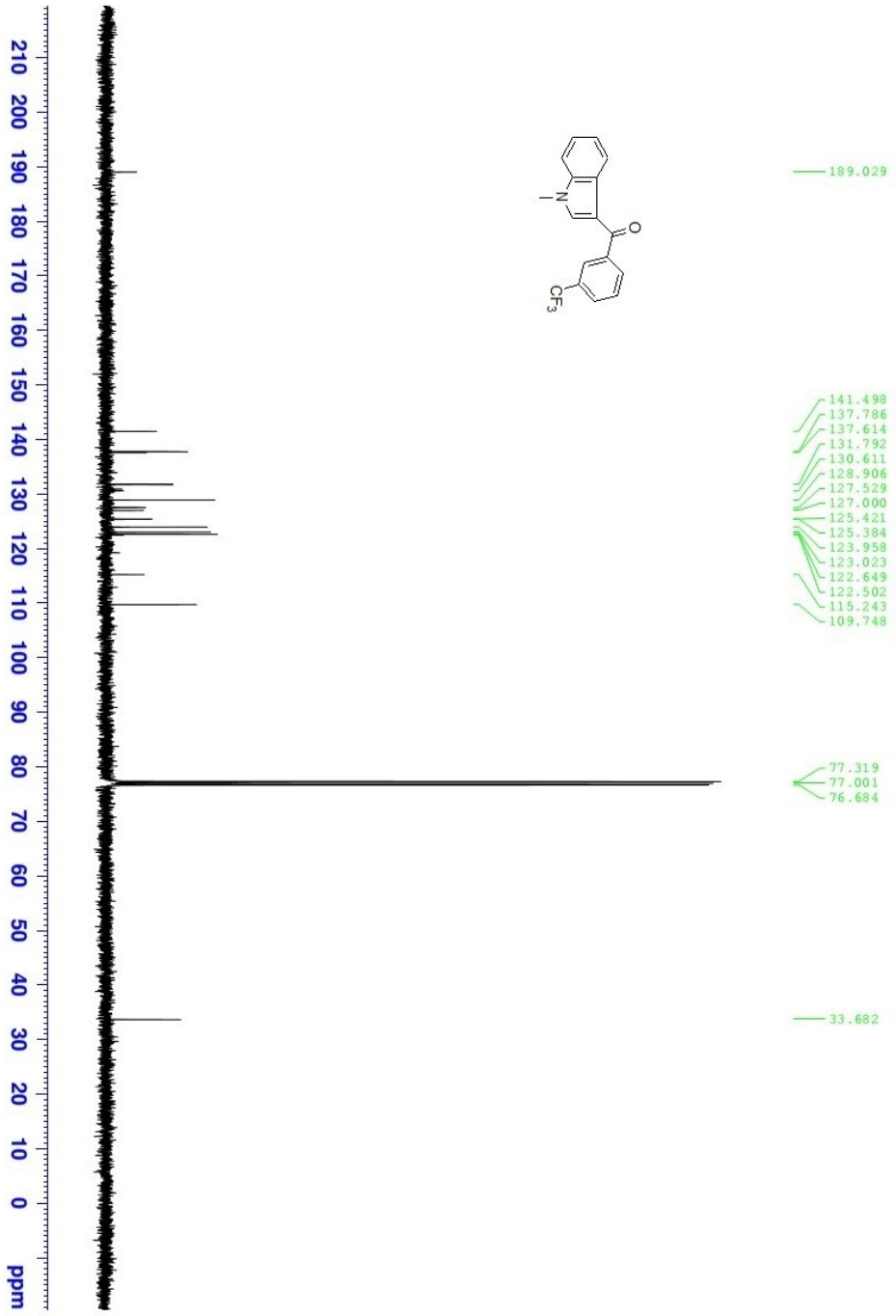
^{1}H NMR of Compound 3af



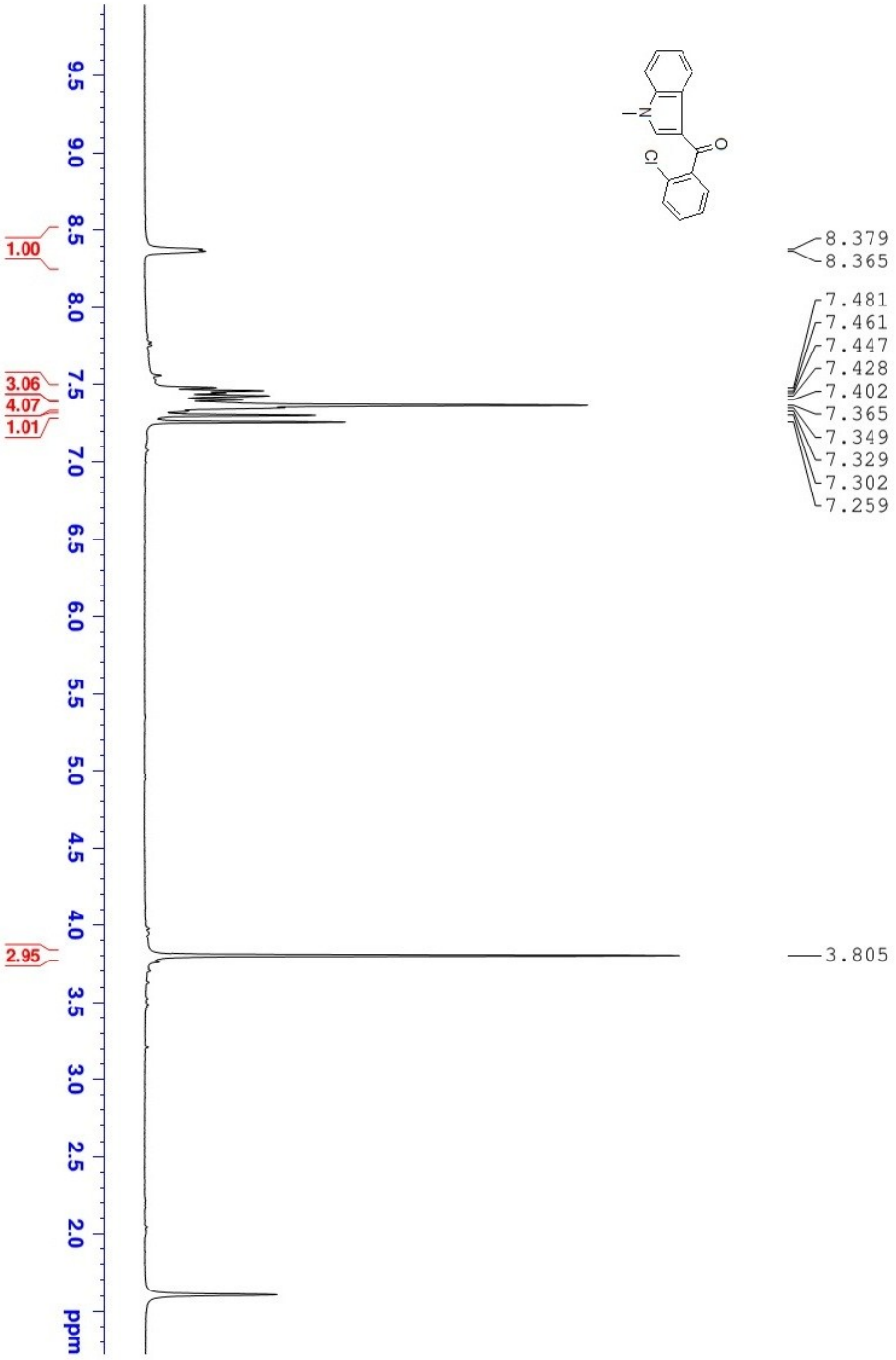
^{13}C NMR of Compound 3af



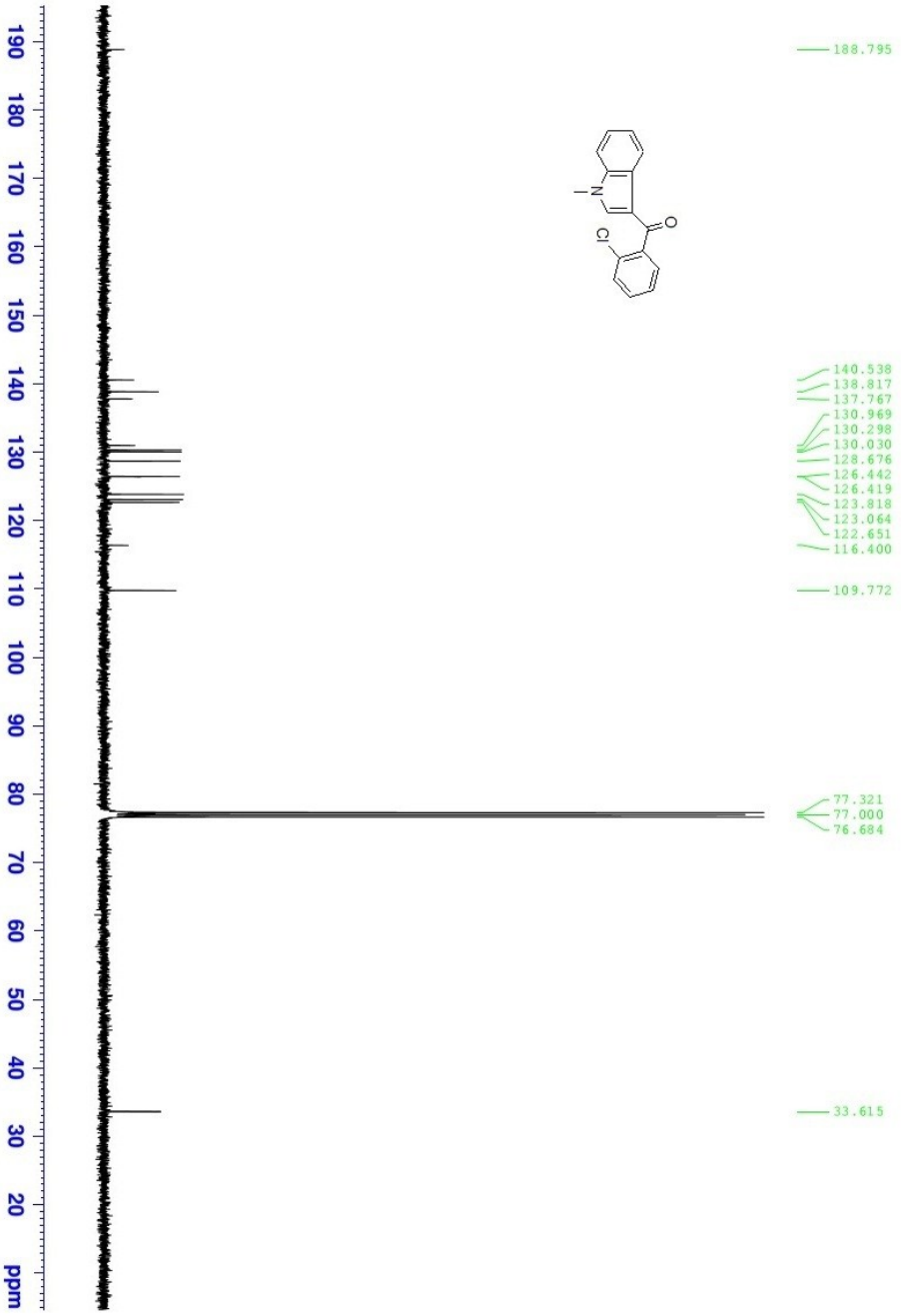
^{1}H NMR of Compound 3ag



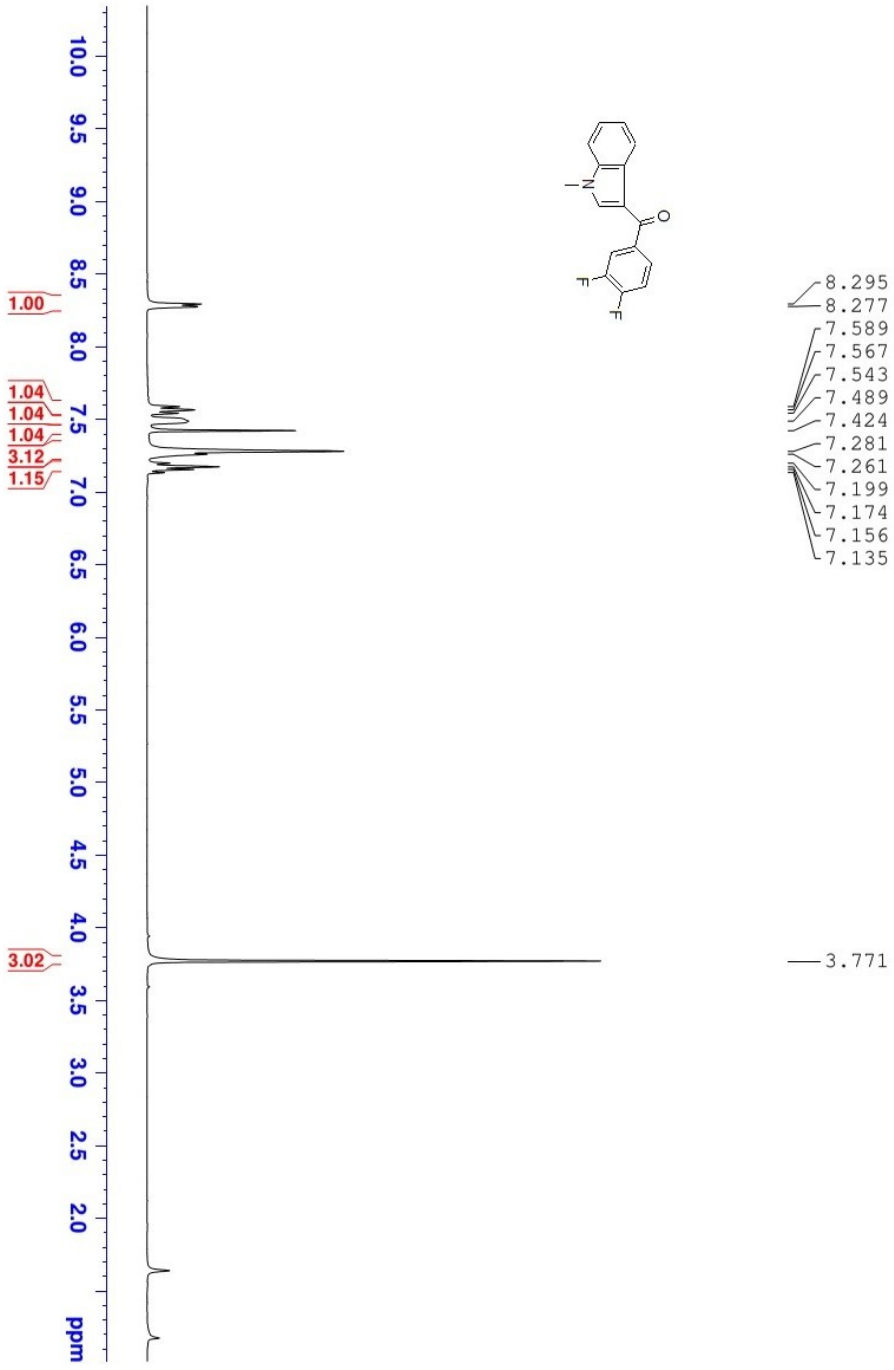
^{13}C NMR of Compound 3ag


9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
1.00
3.06
4.07
1.01
2.95
8.379
8.365
7.481
7.461
7.447
7.428
7.402
7.365
7.349
7.329
7.302
7.259
3.805
N
O
Cl

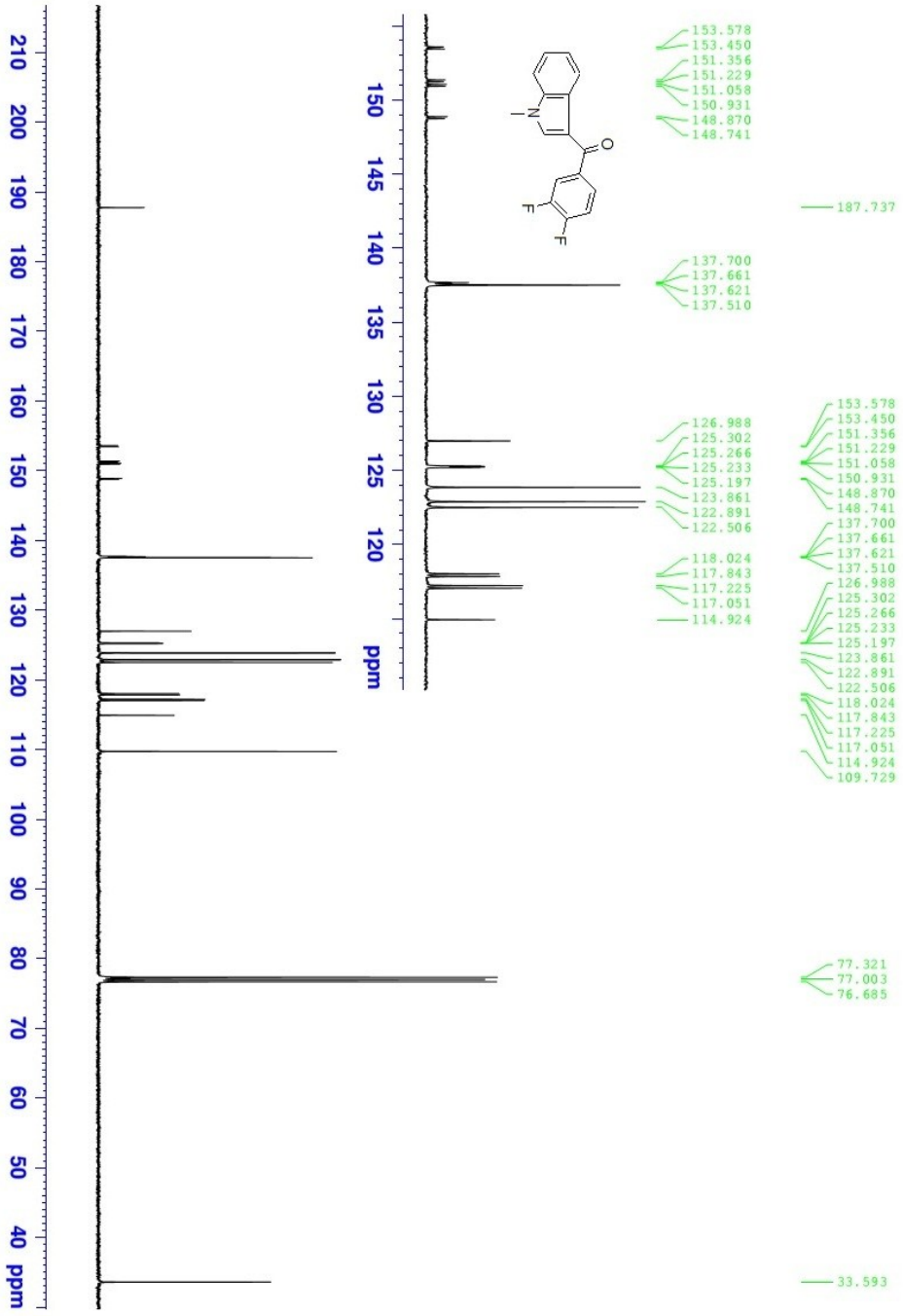
^{1}H NMR of Compound 3ah



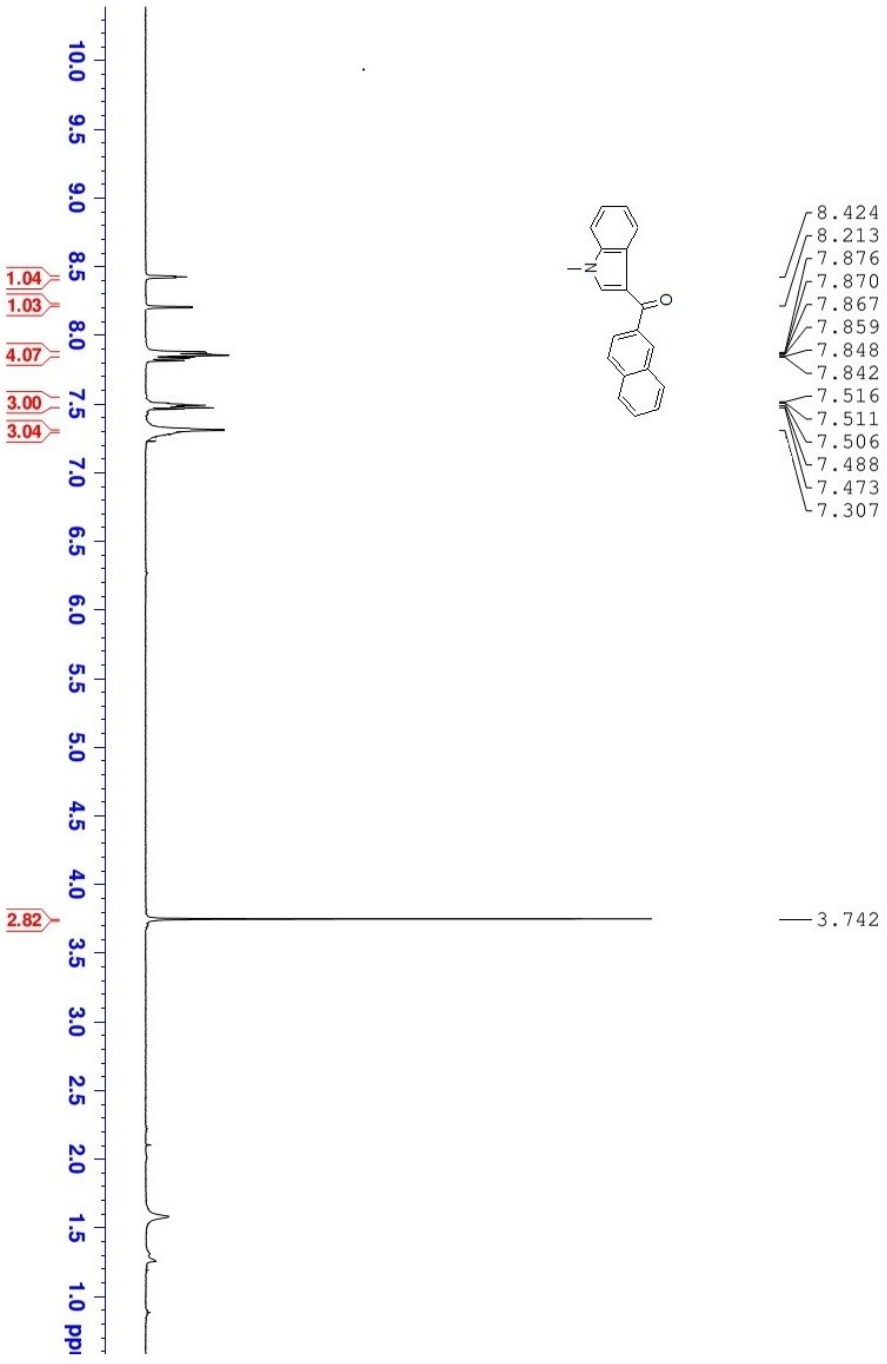
^{13}C NMR of Compound 3ah



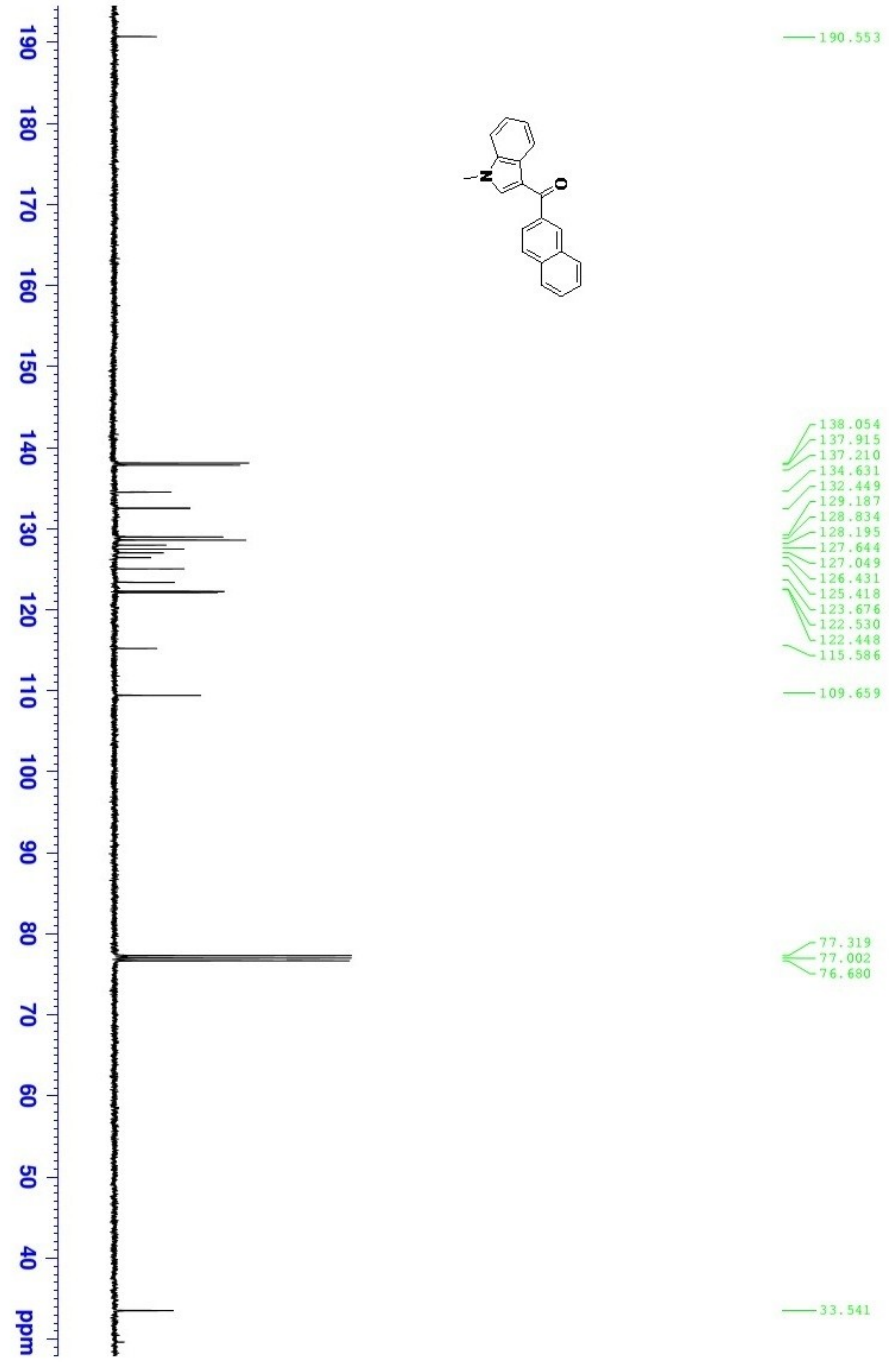
^{1}H NMR of Compound 3ai



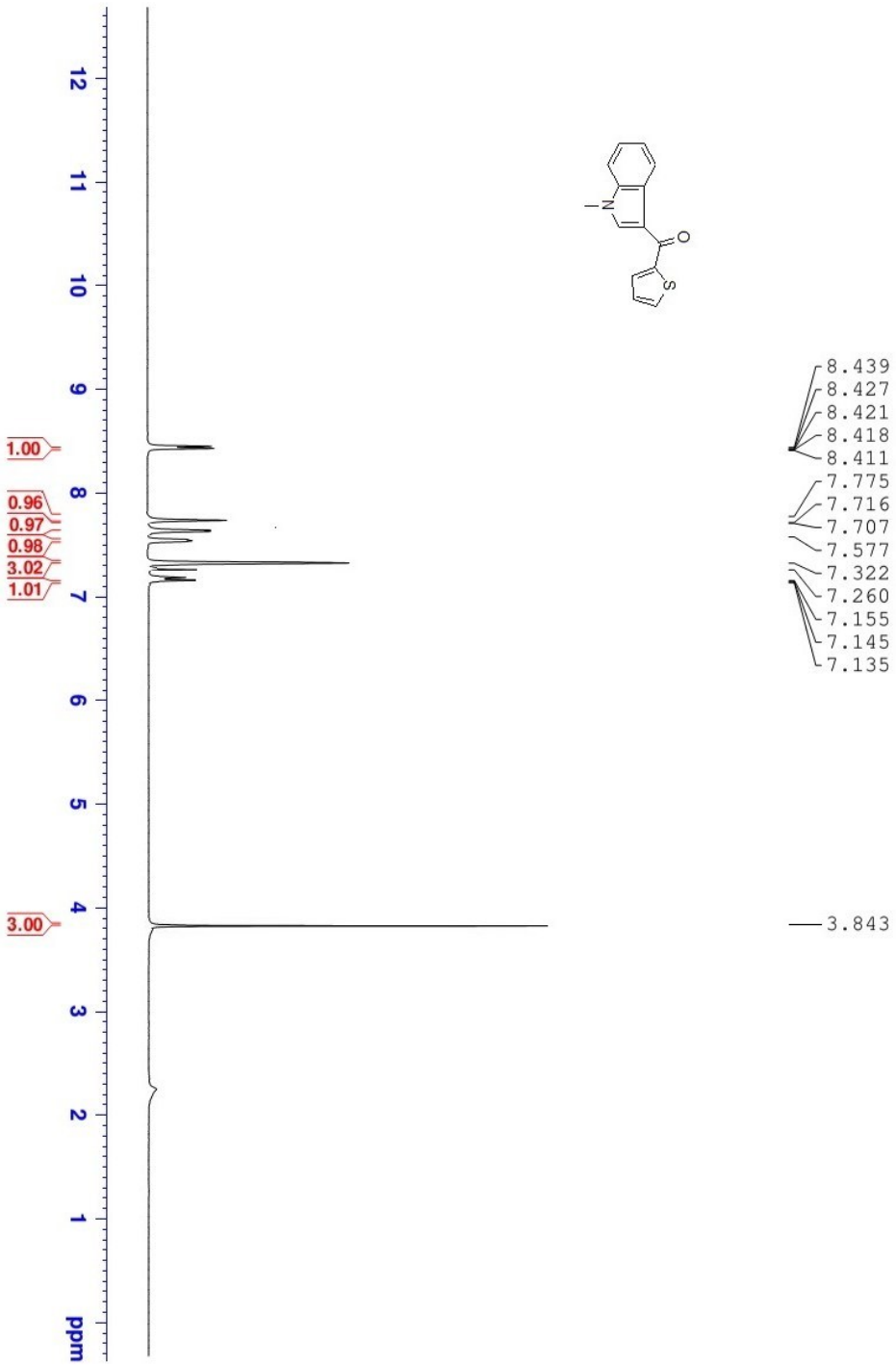
^{13}C NMR of Compound 3ai



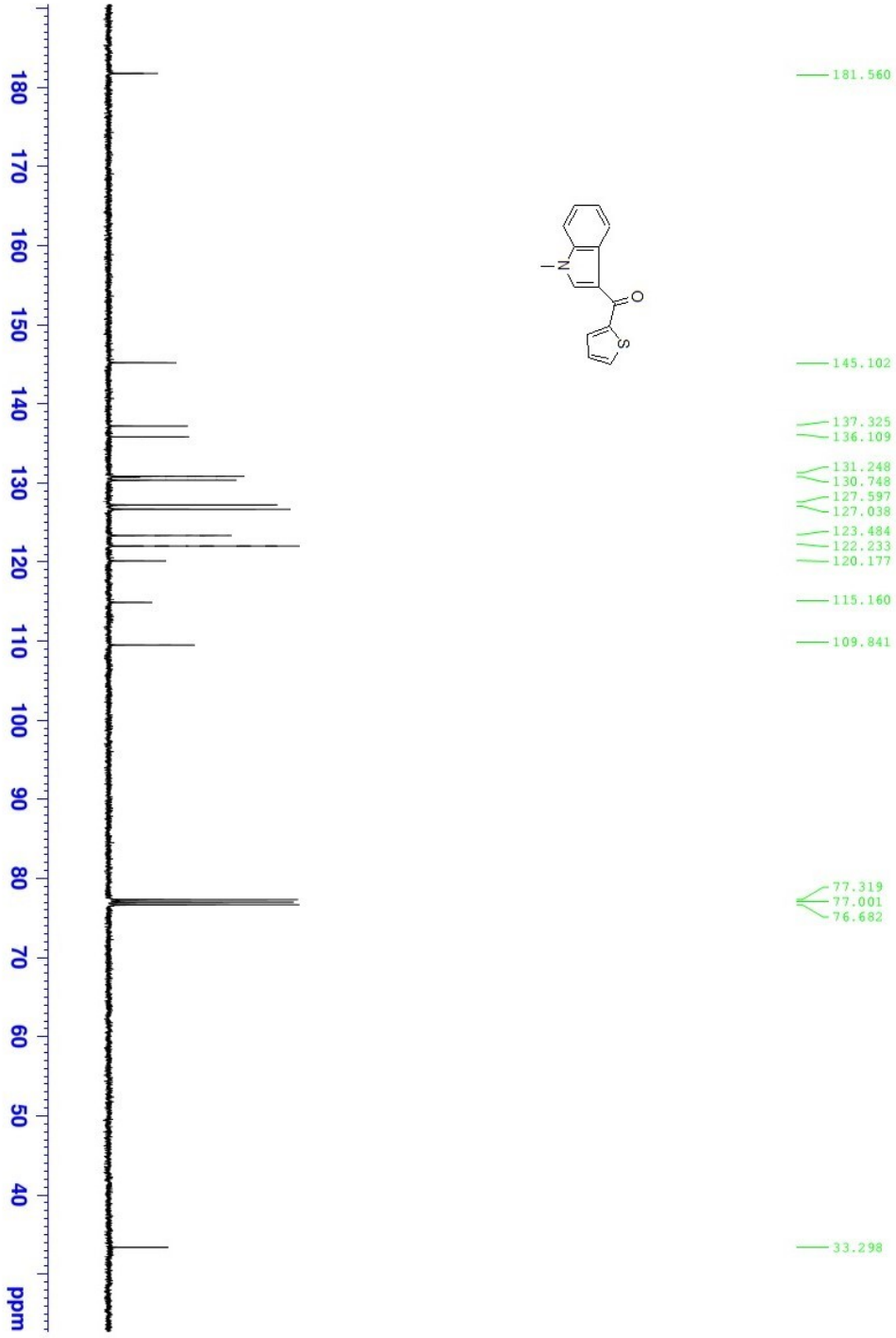
^{1}H NMR of Compound 3aj



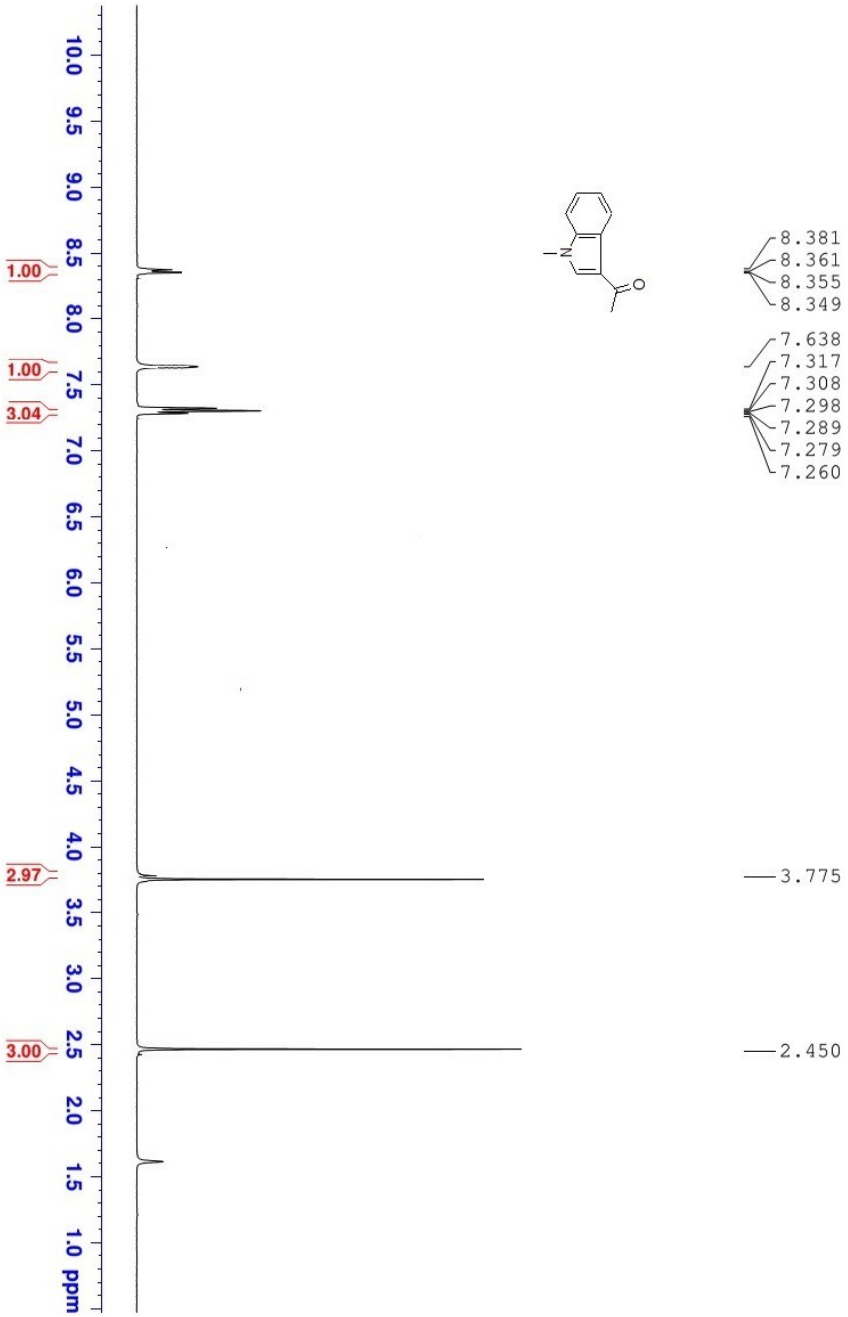
^{13}C NMR of Compound 3aj



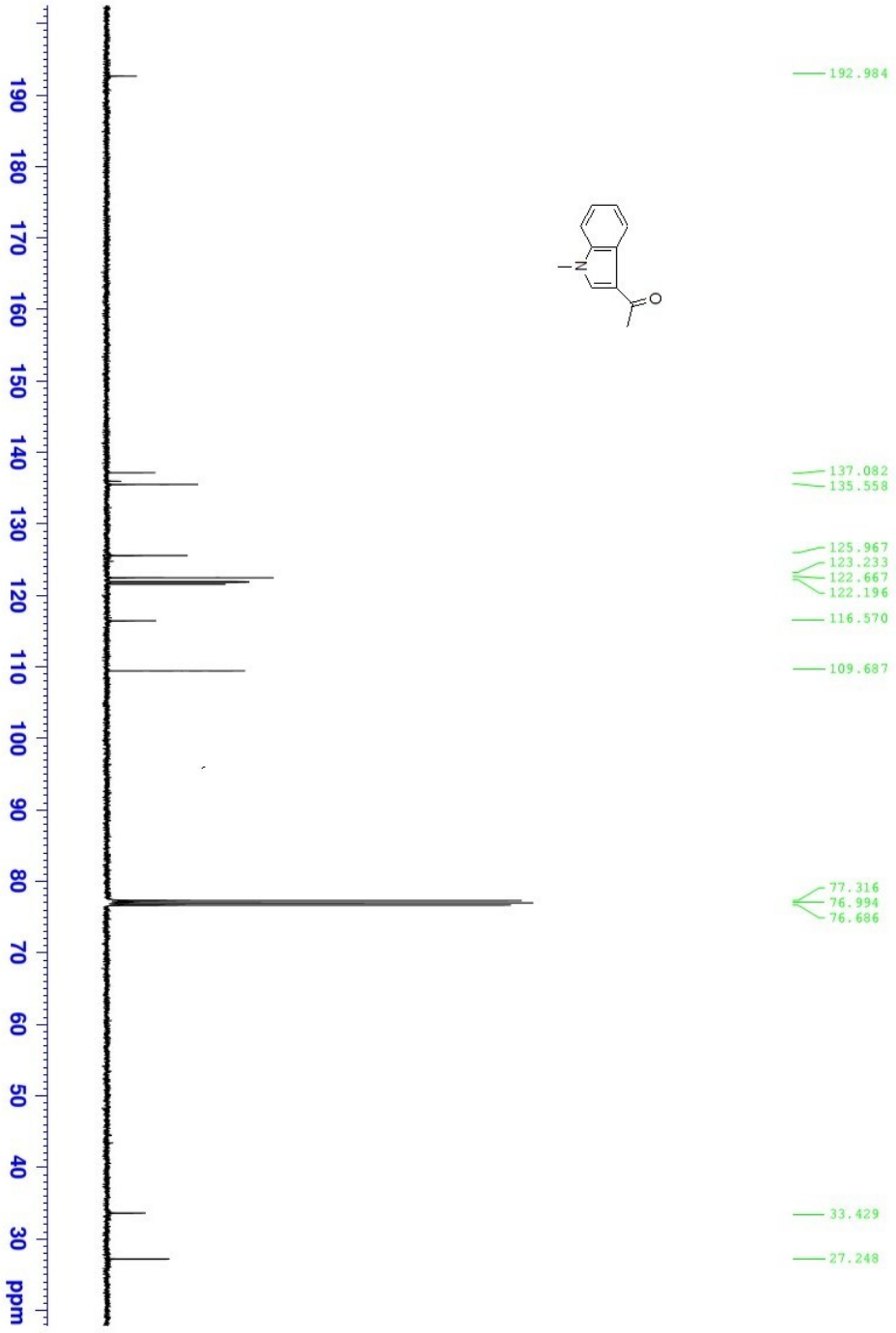
^{1}H NMR of Compound 3ak



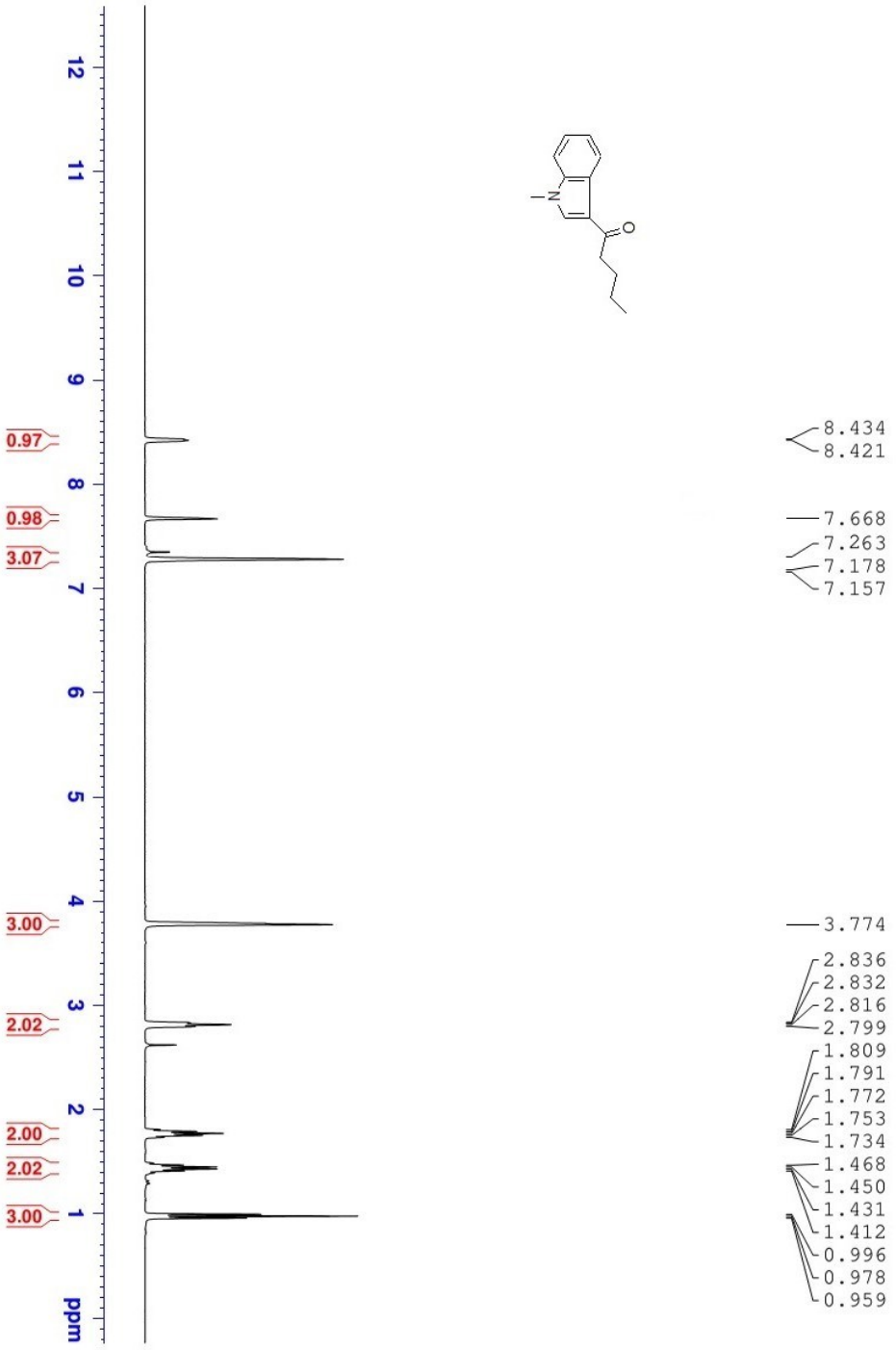
^{13}C NMR of Compound 3ak



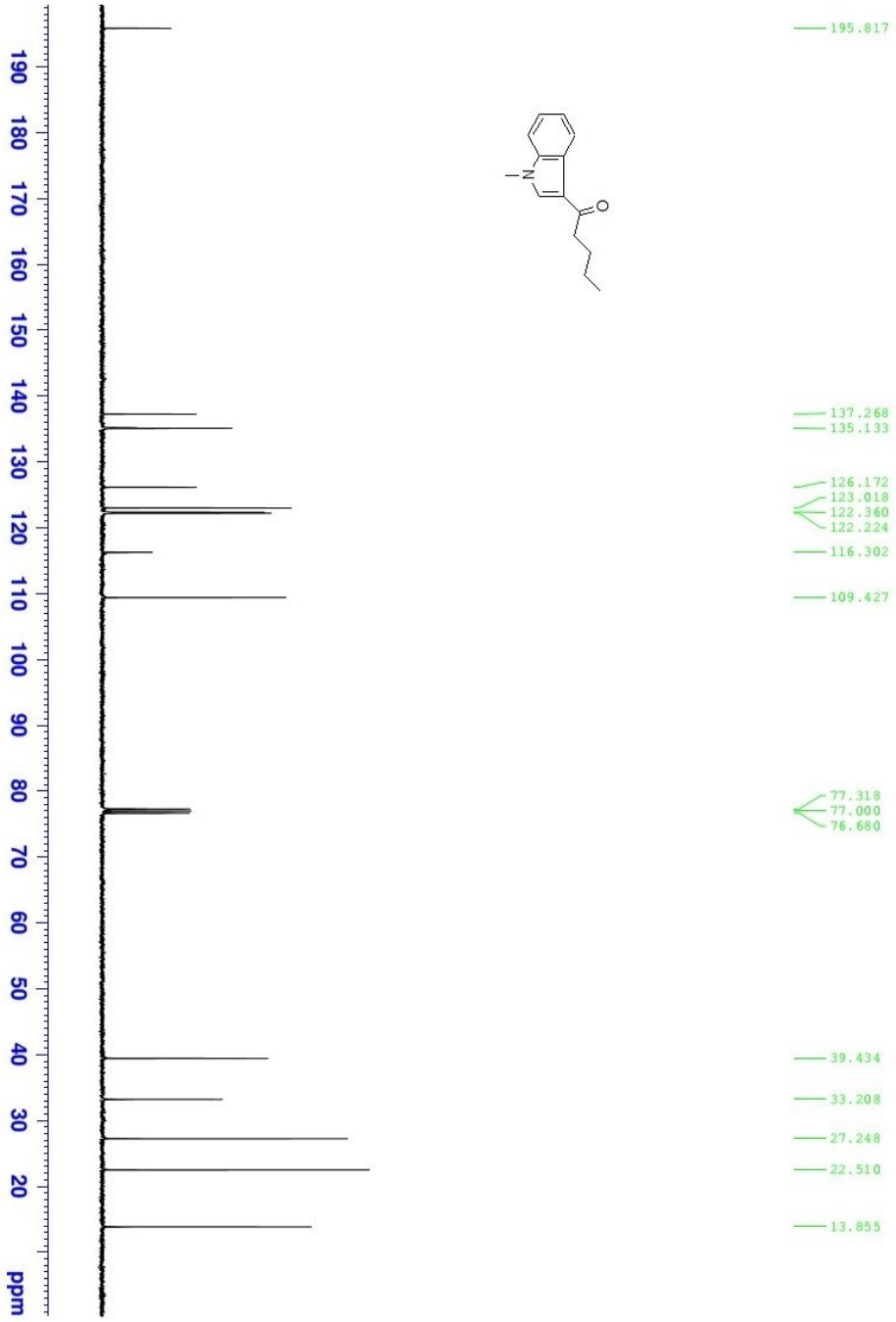
^{1}H NMR of Compound 3al



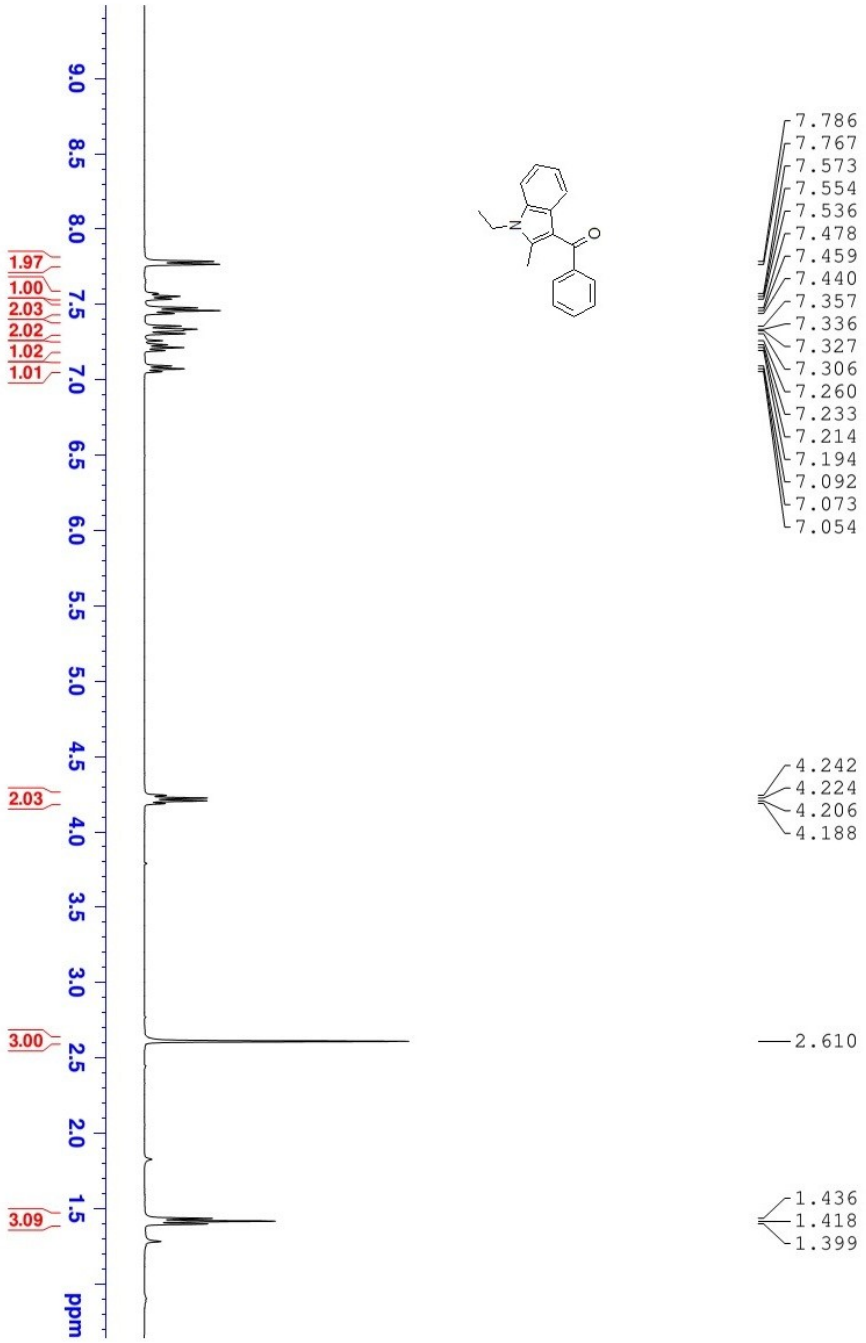
^{13}C NMR of Compound 3al



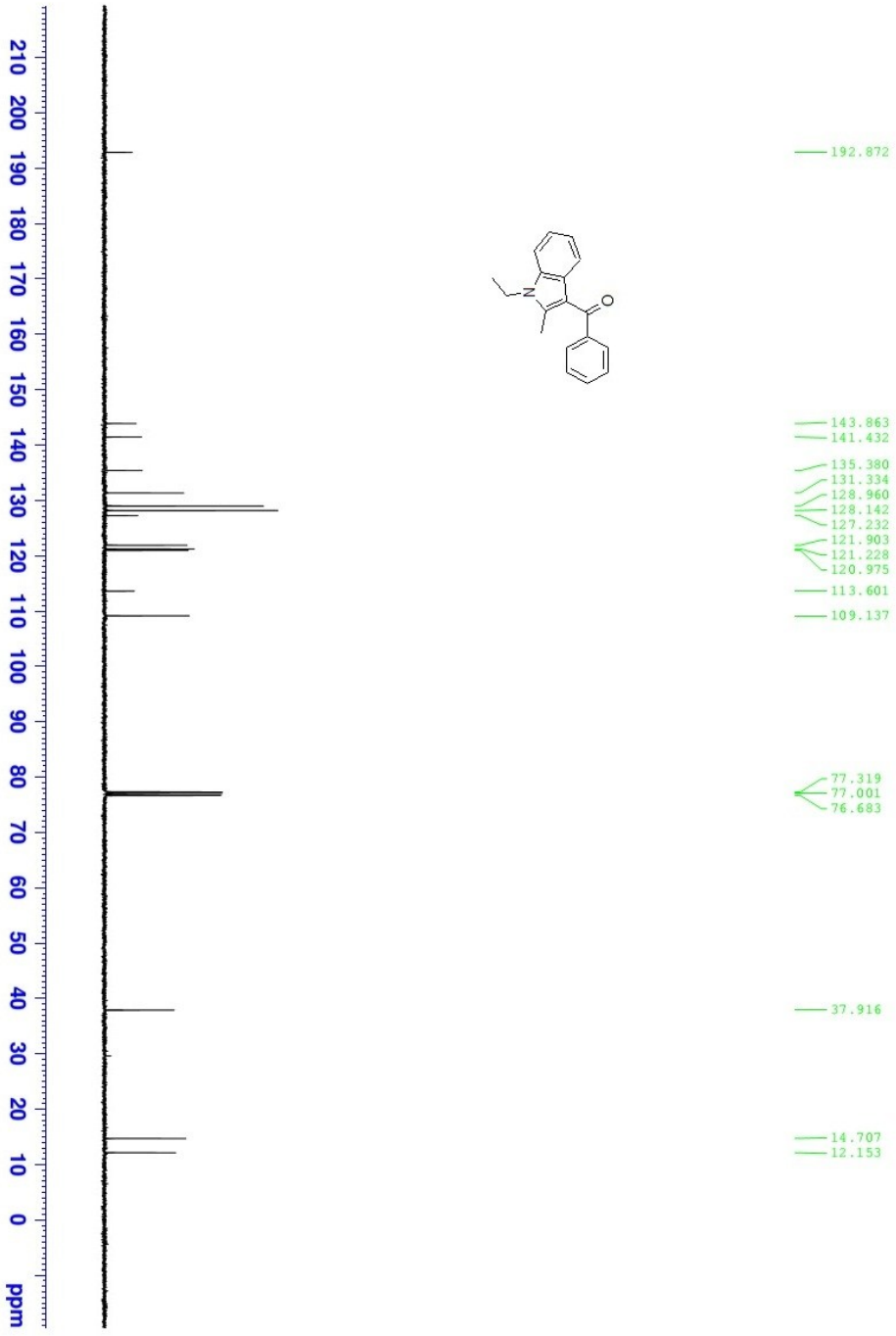
^{1}H NMR of Compound 3am



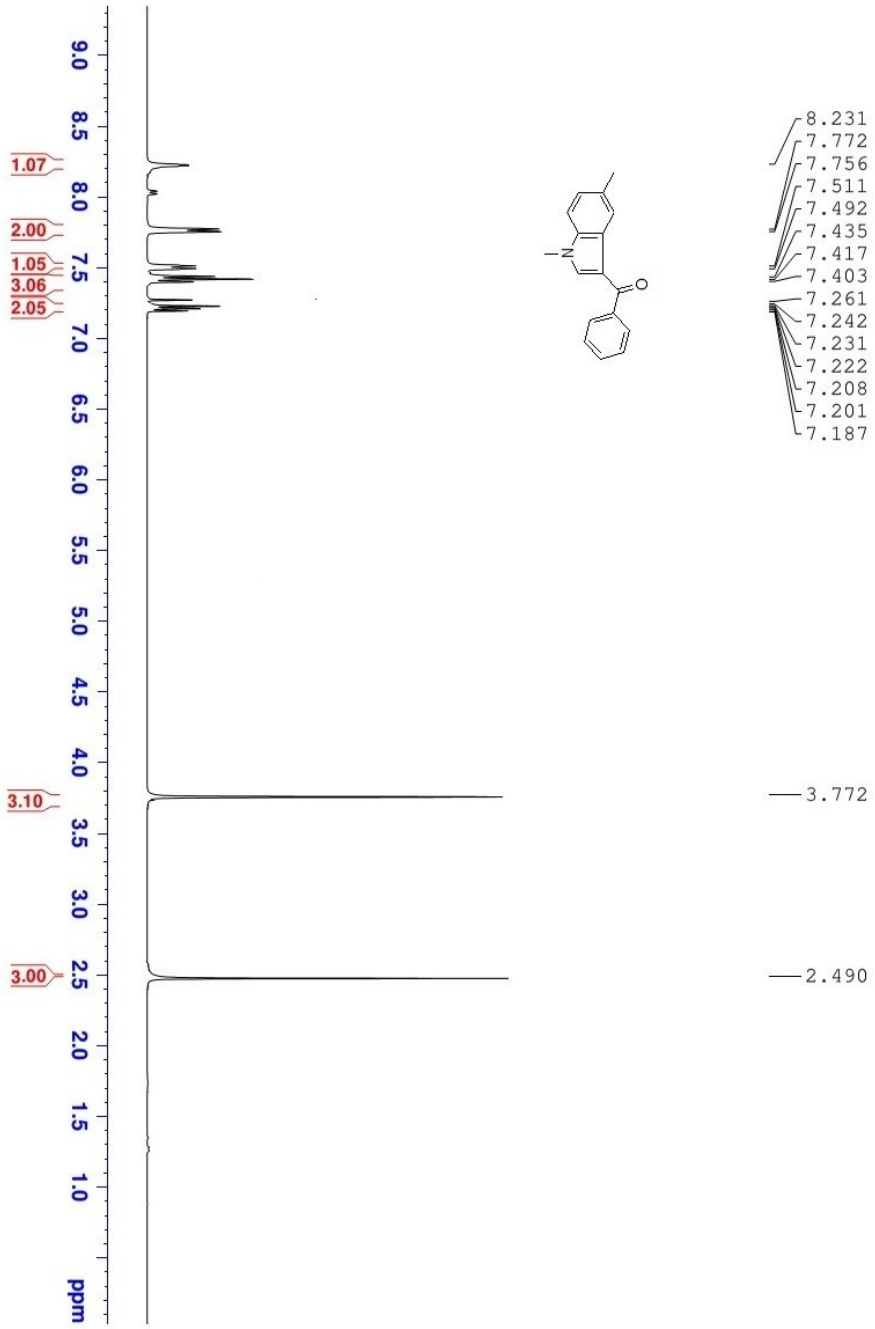
^{13}C NMR of Compound 3am



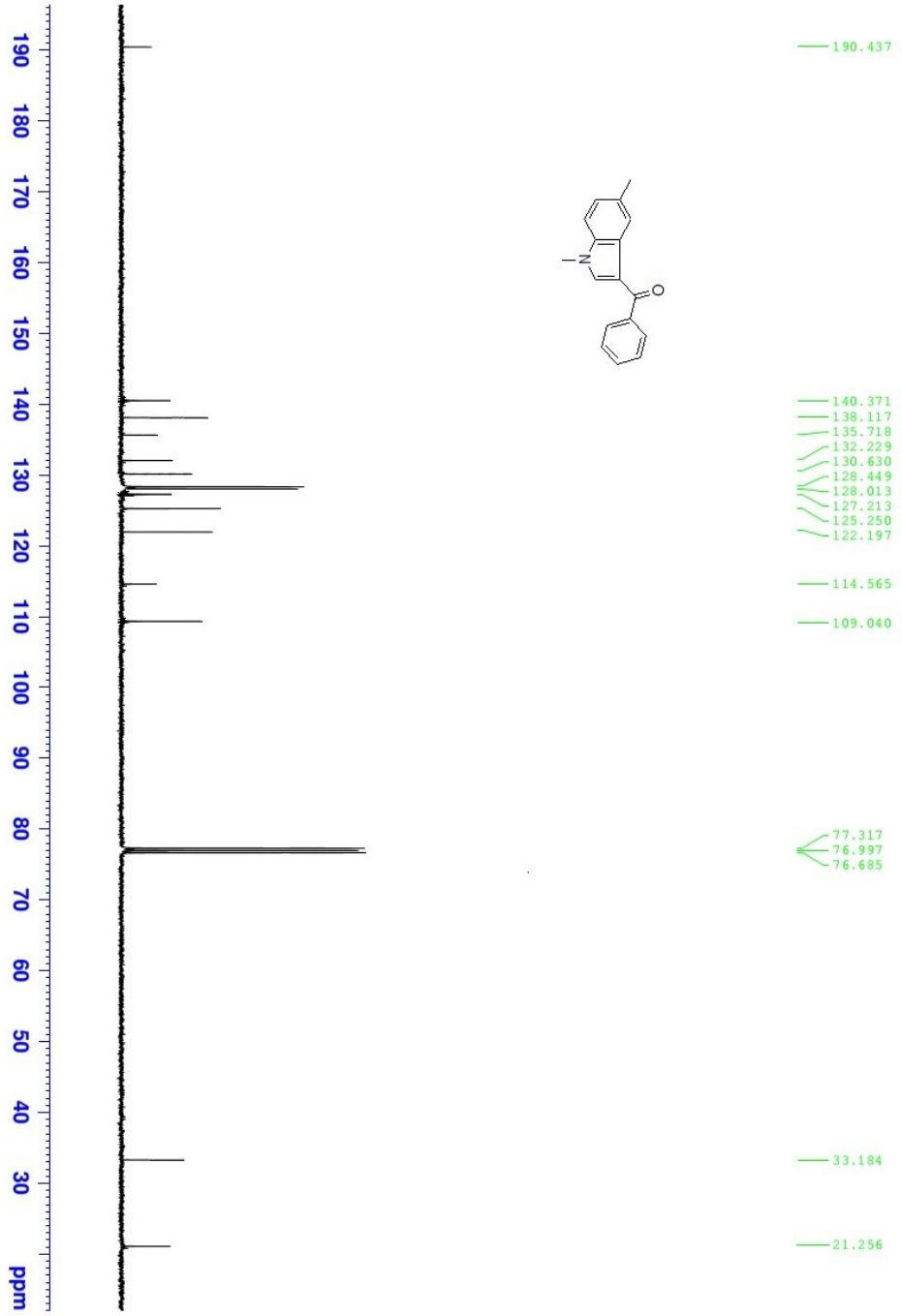
^{1}H NMR of Compound 3ba



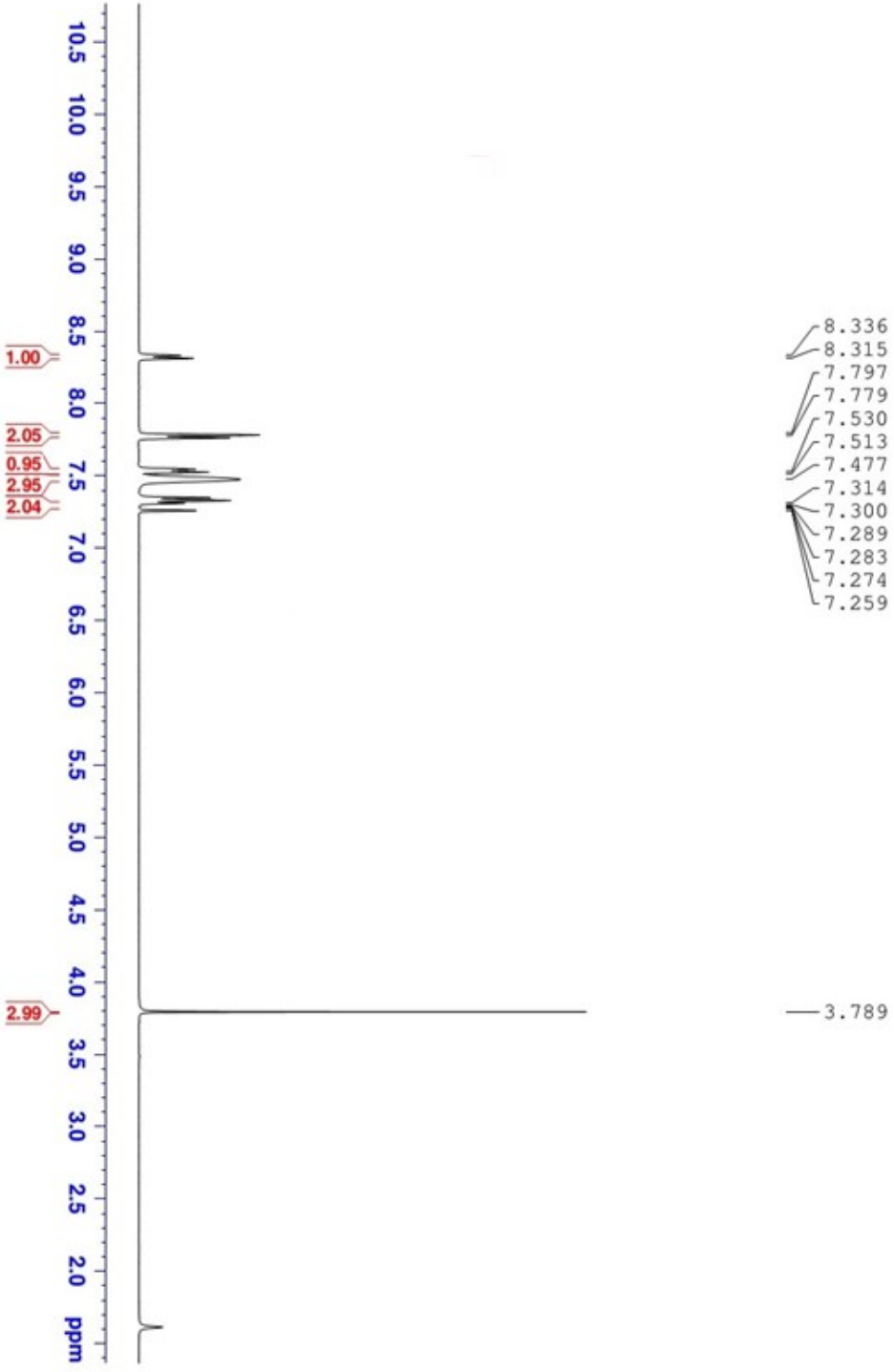
^{13}C NMR of Compound 3ba



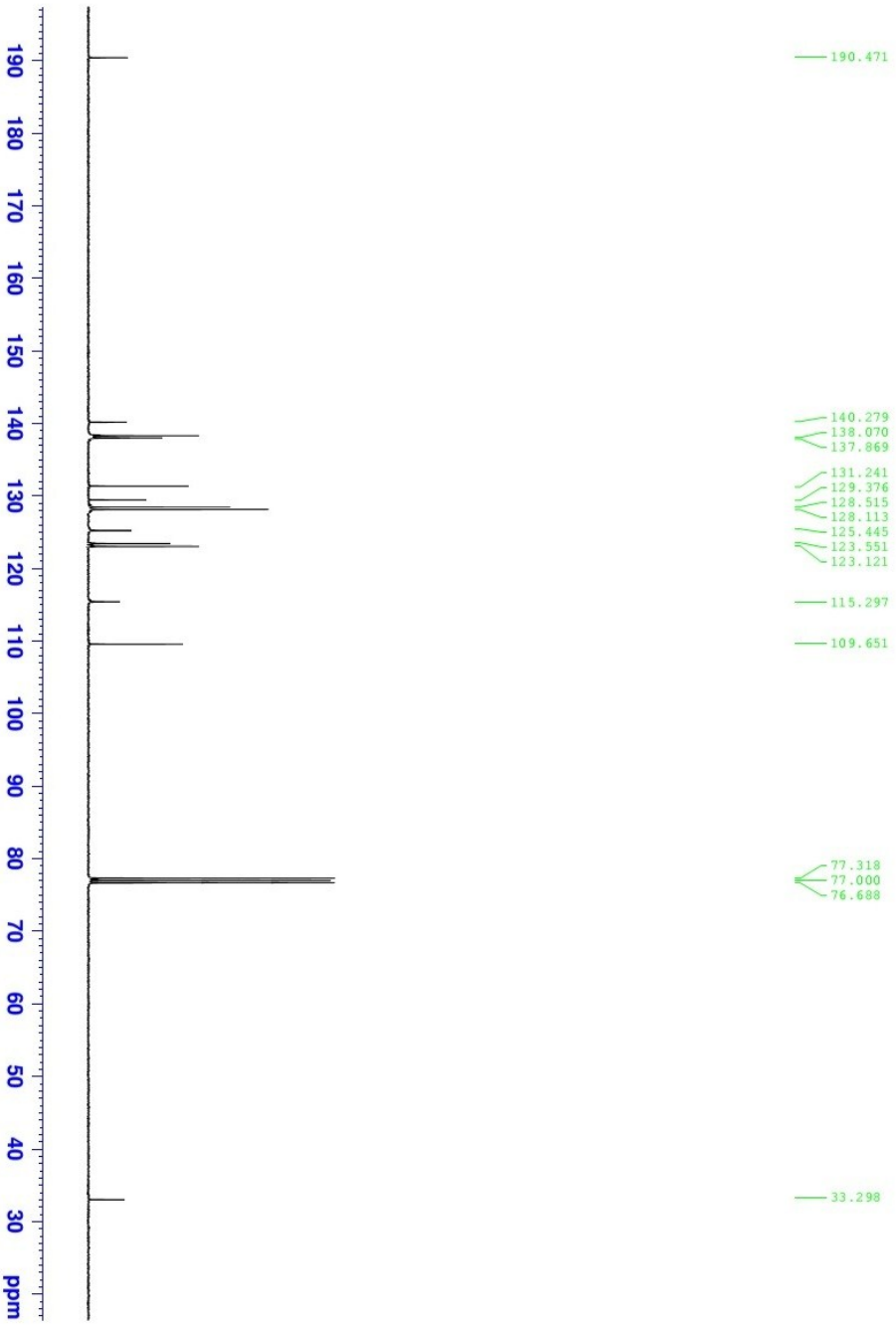
^{1}H NMR of Compound 3ca



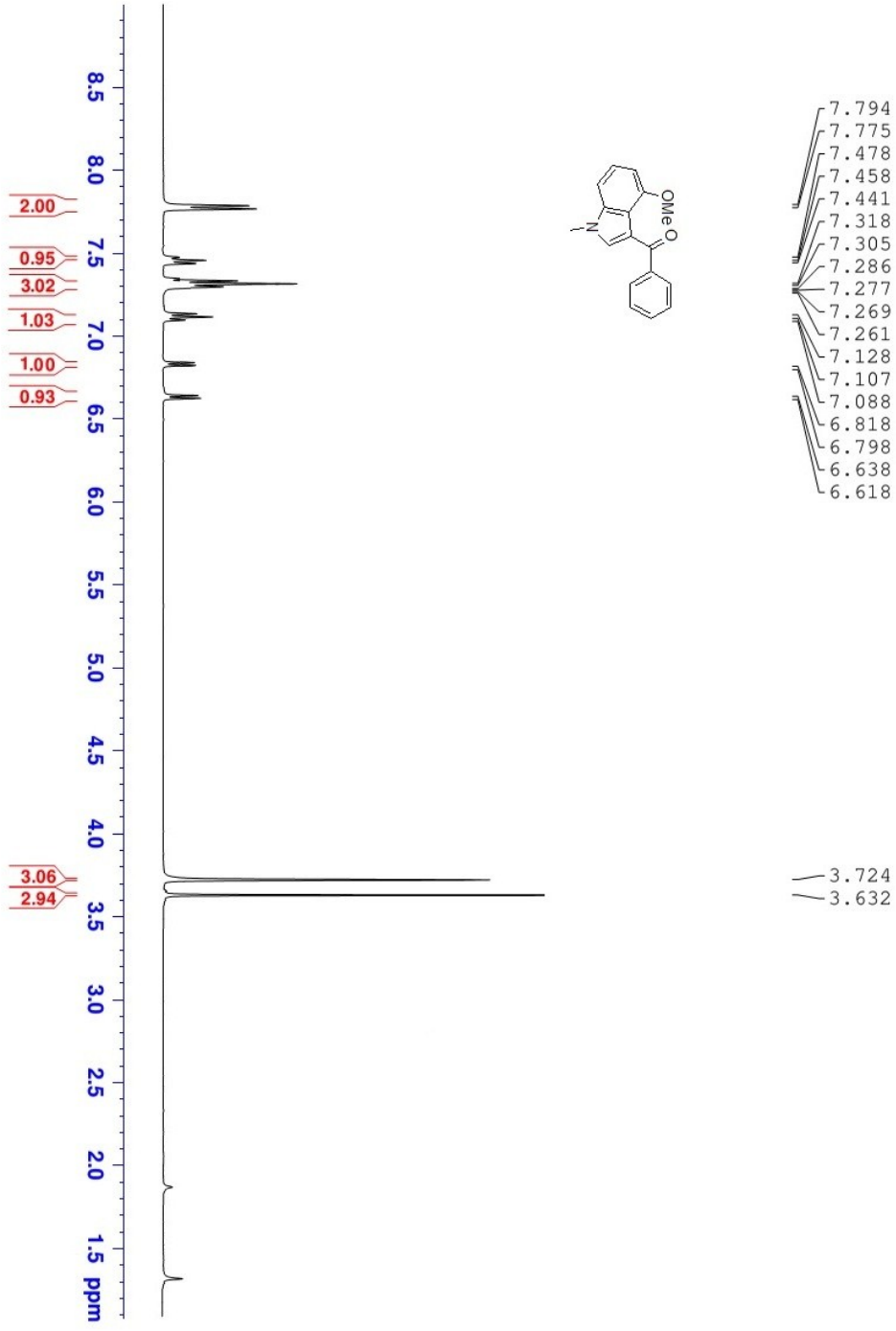
^{13}C NMR of Compound 3ca



^{1}H NMR of Compound 3da



^{13}C NMR of Compound 3da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7.261
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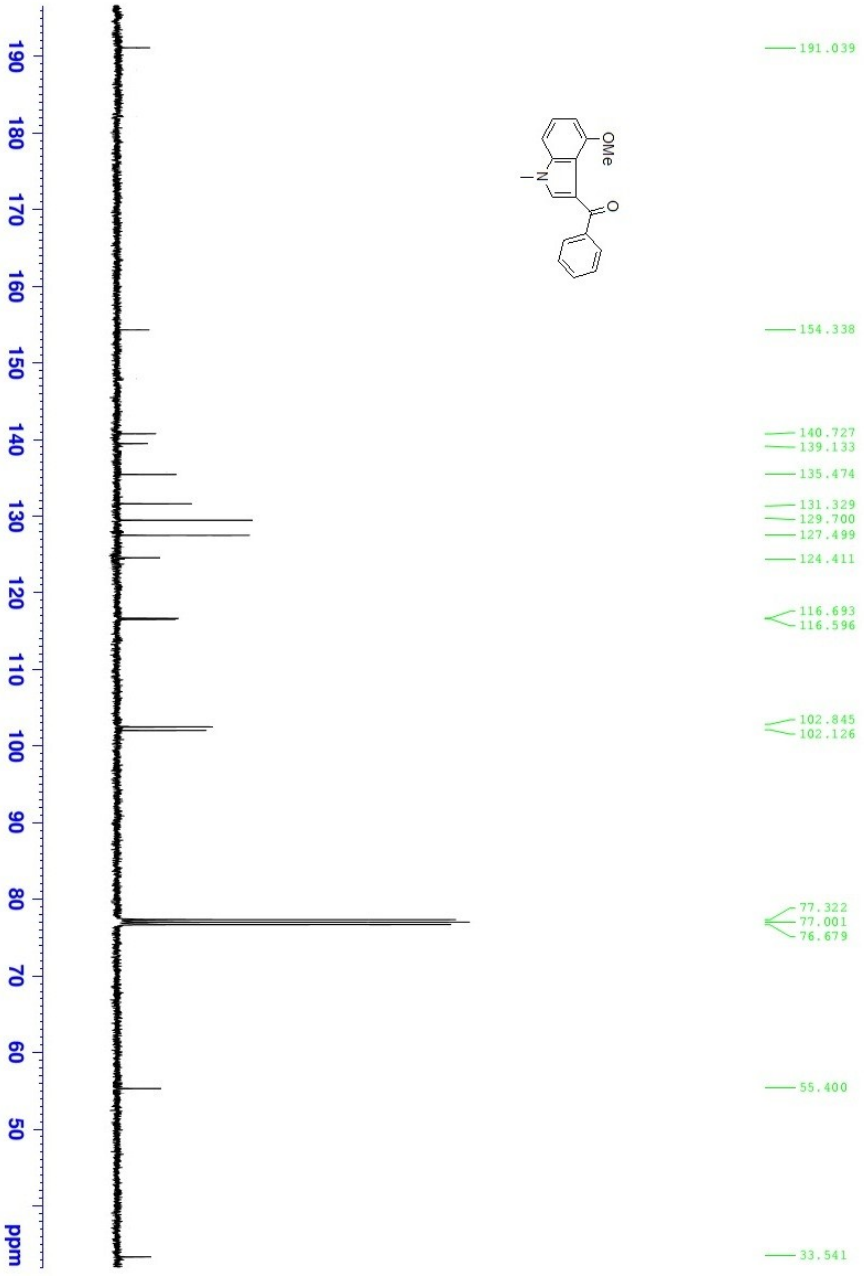
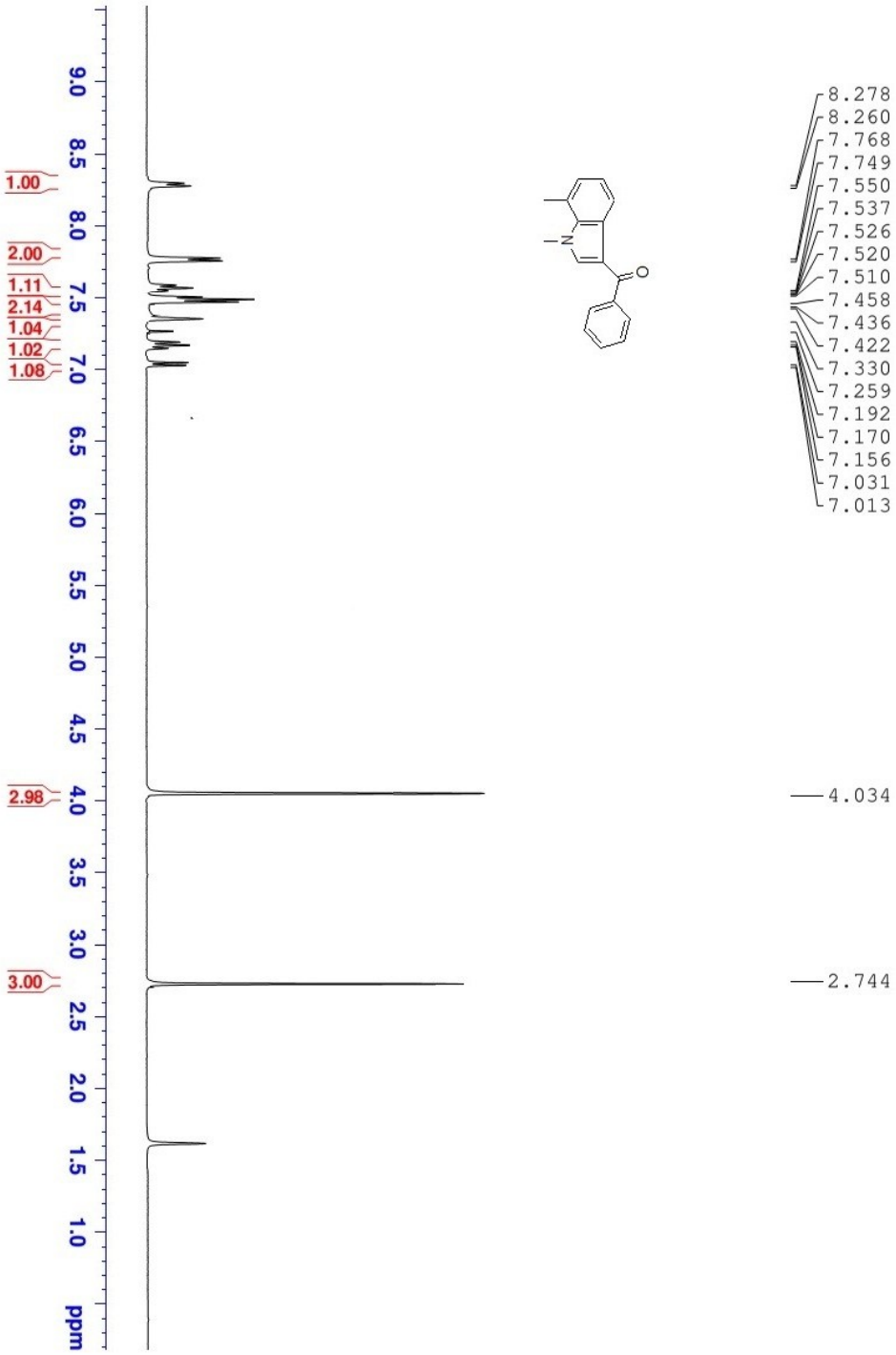
7.107
7.088
6.818
6.798
6.638
6.618
3.724
3.632
2.00
0.95
3.02
1.03
1.00
0.93
3.06
2.94
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
OMe
O
N

^{1}H NMR of Compound 3ea



^{13}C NMR of Compound 3ea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7.259
7.192
7.170
7.156
7.031
7.013
4.034
2.744
1.00
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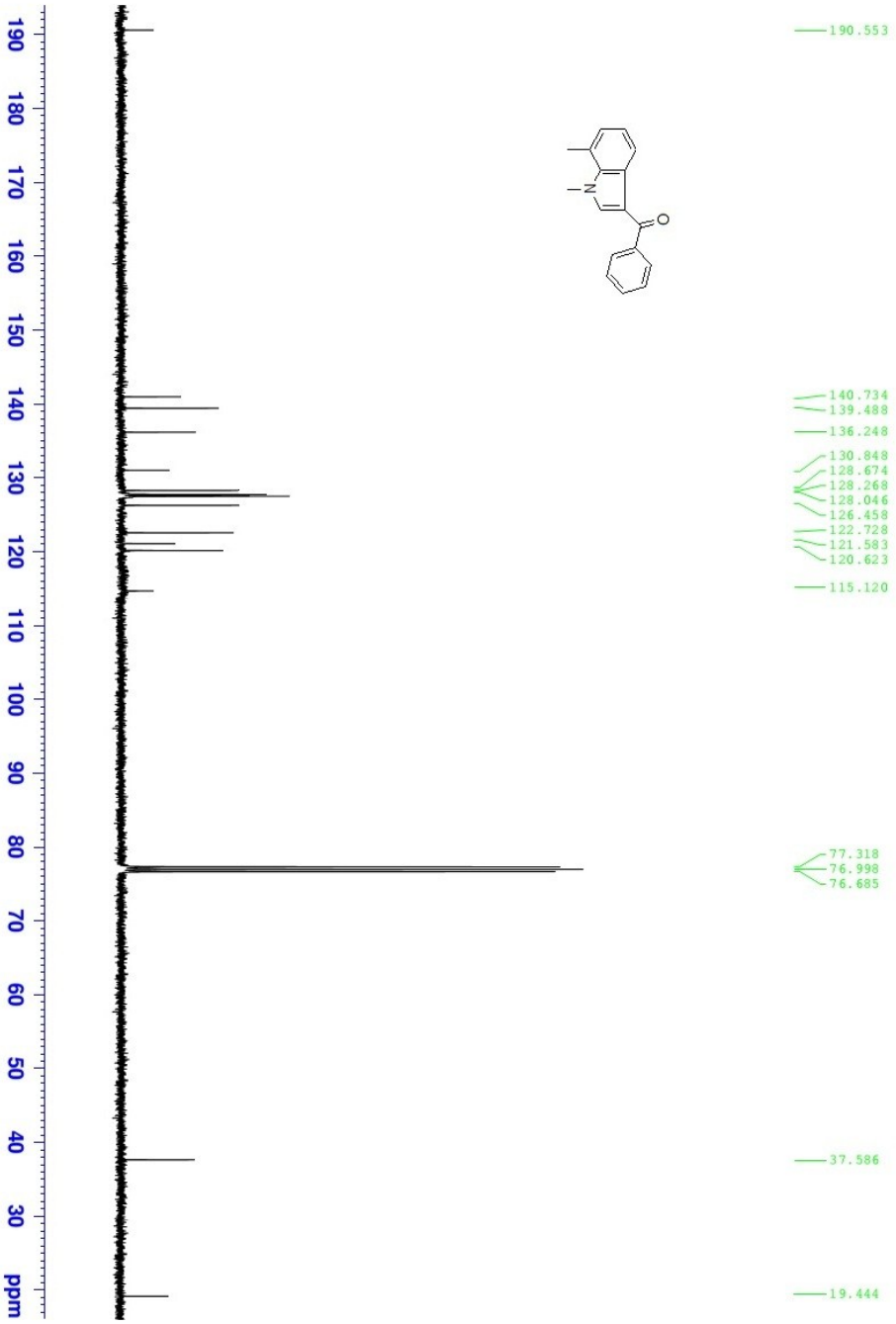
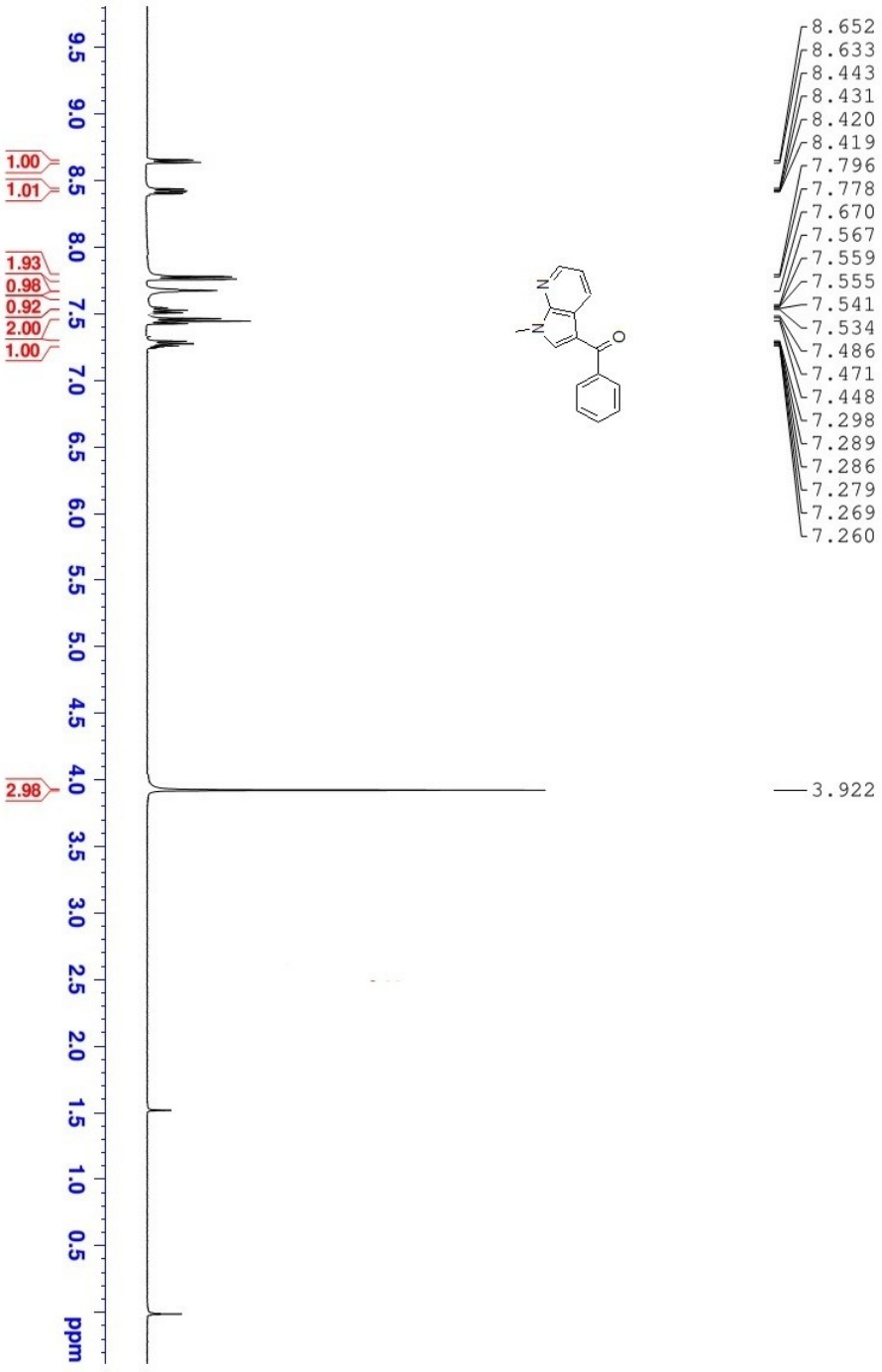
1.11
2.14
1.04
1.02
1.08
2.98
3.00
ppm

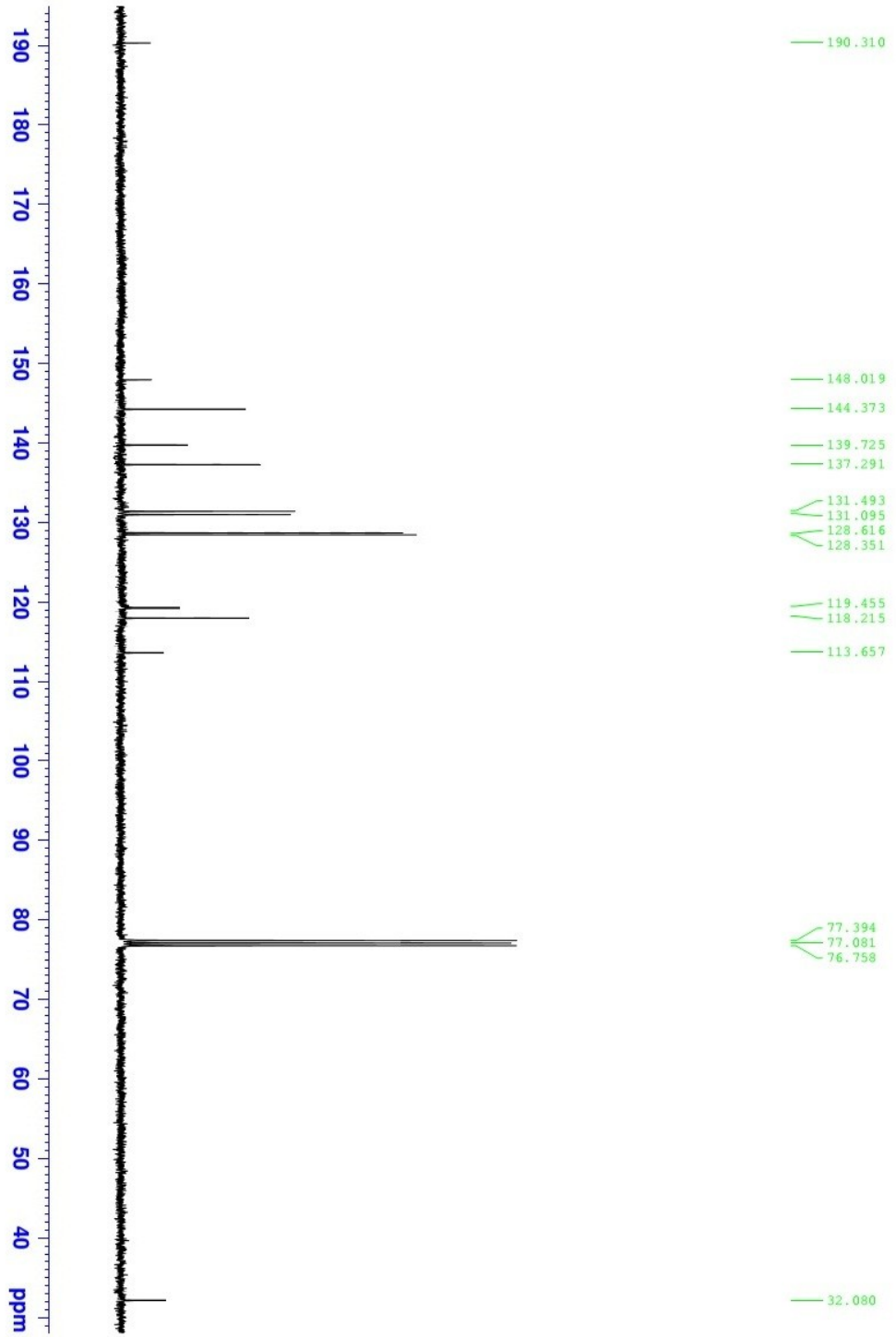
^{1}H NMR of Compound 3fa



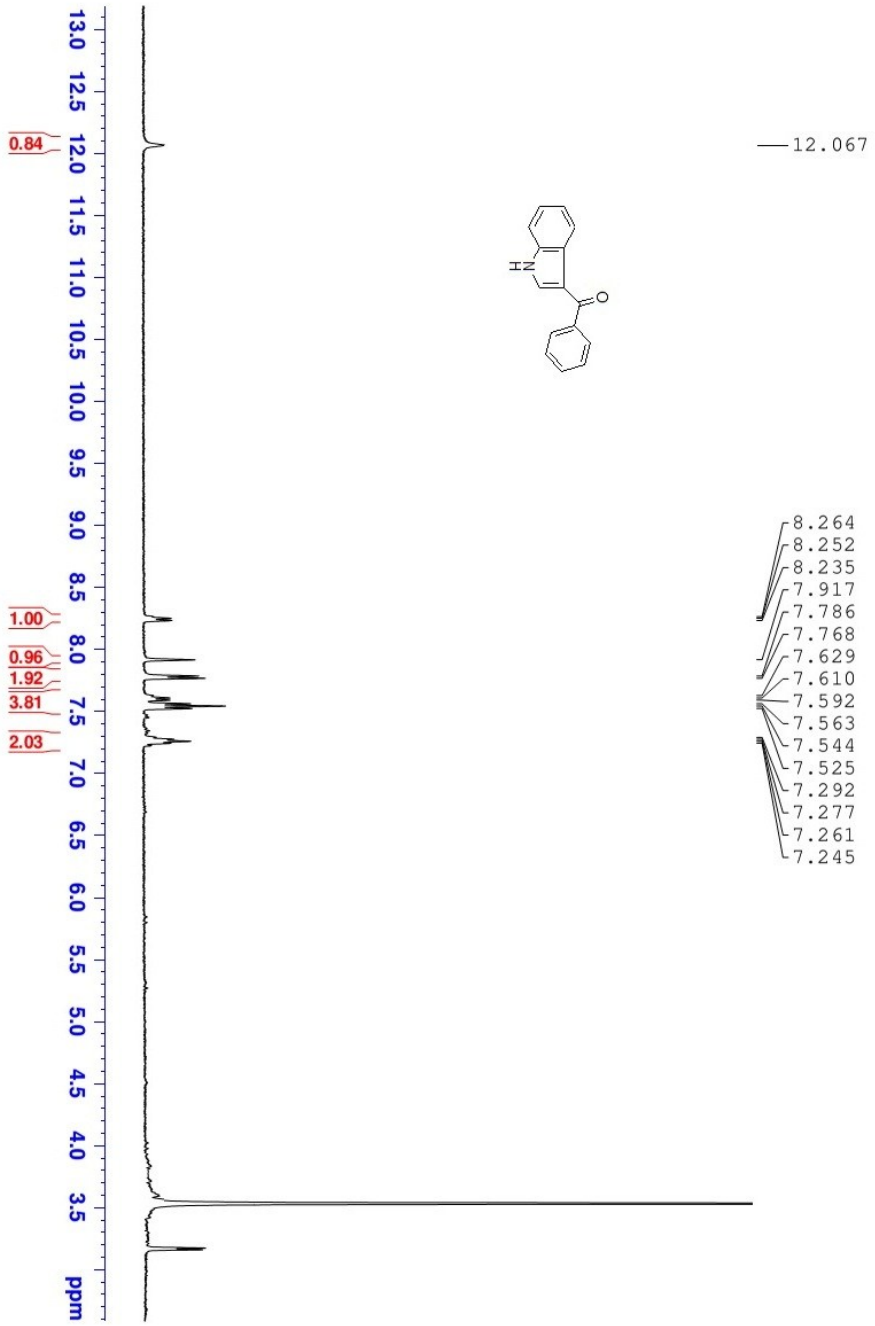
^{13}C NMR of Compound 3fa



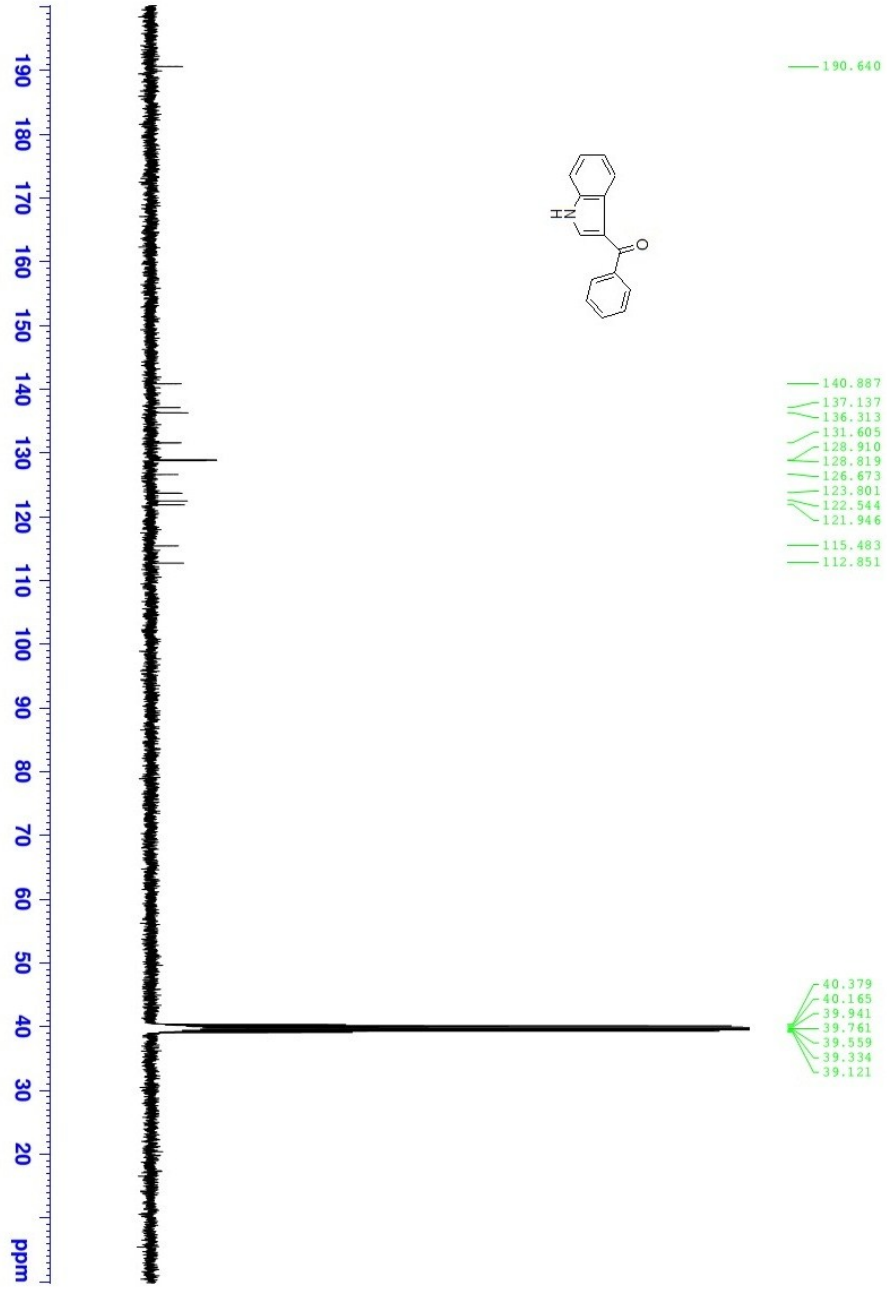
^{1}H NMR of Compound 3ga



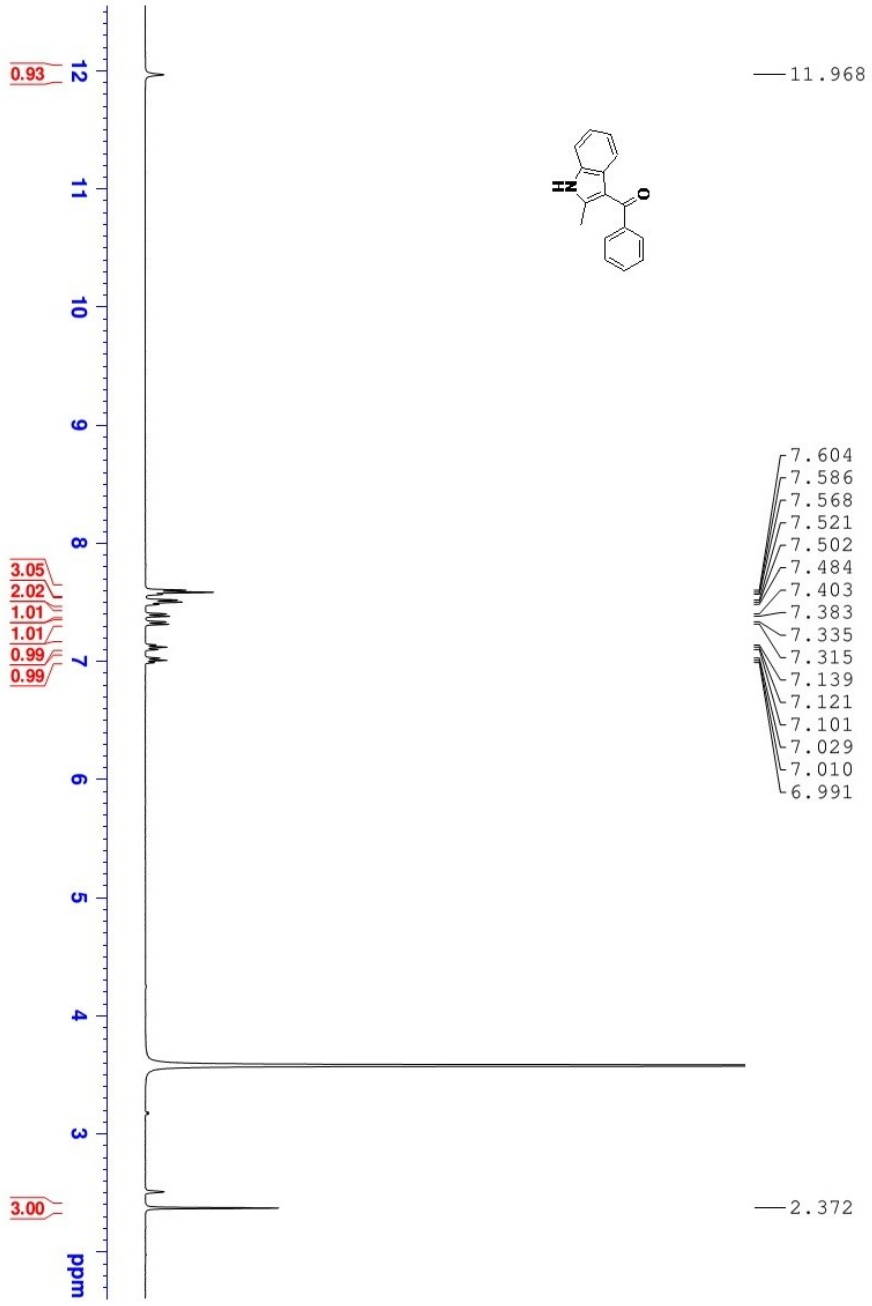
^{13}C NMR of Compound 3ga



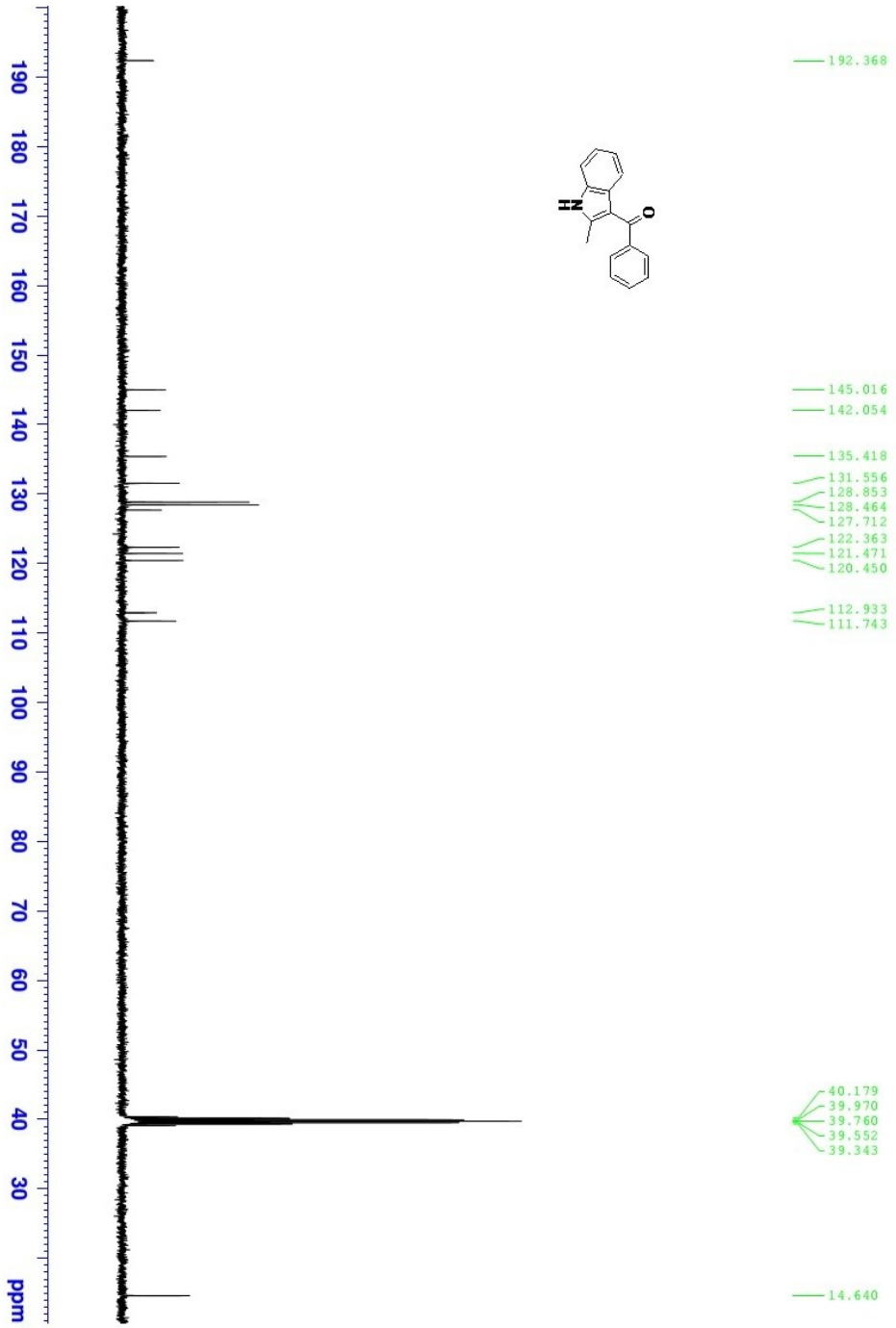
^{1}H NMR of Compound 3ha



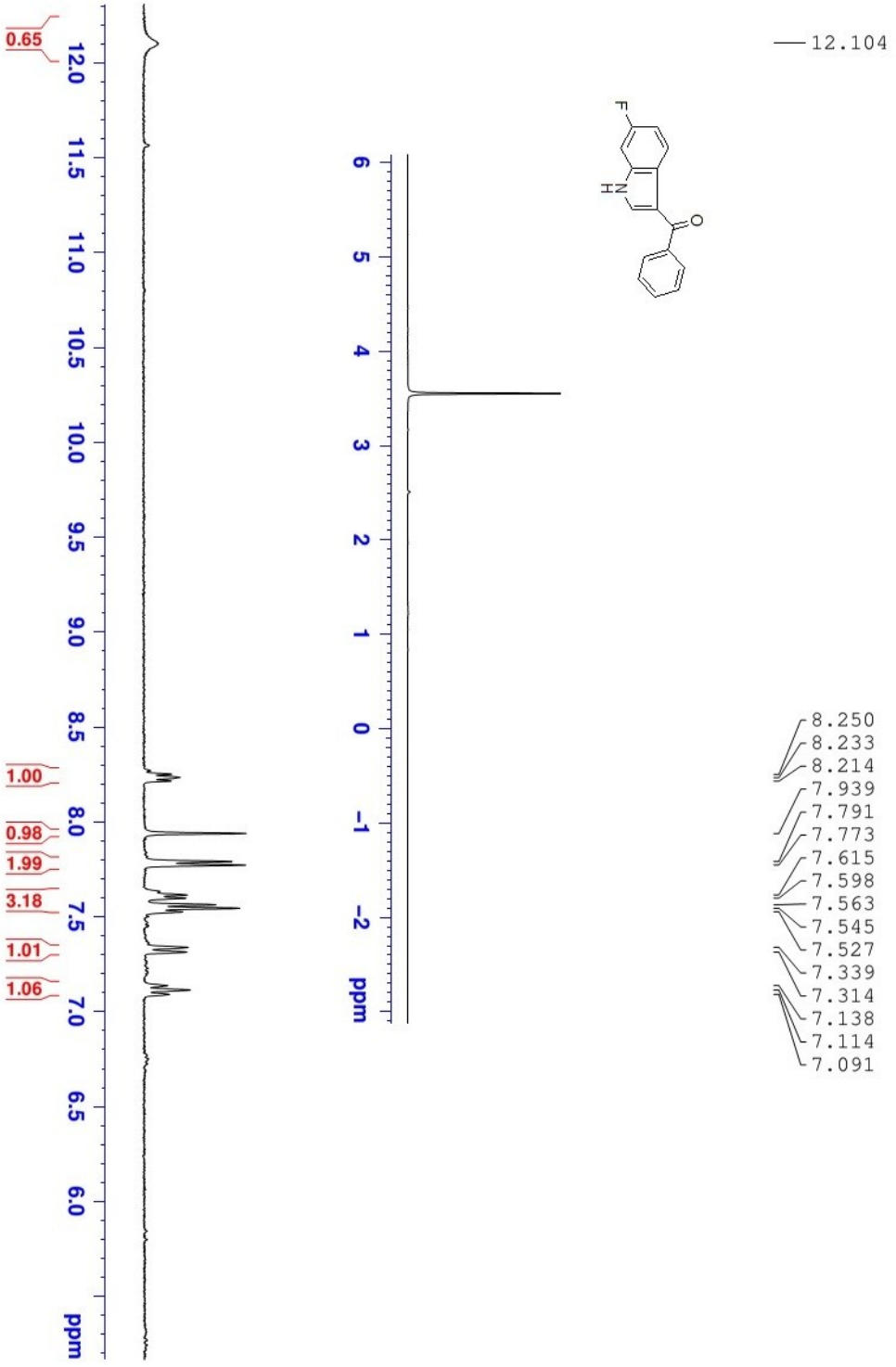
^{13}C NMR of Compound 3ha



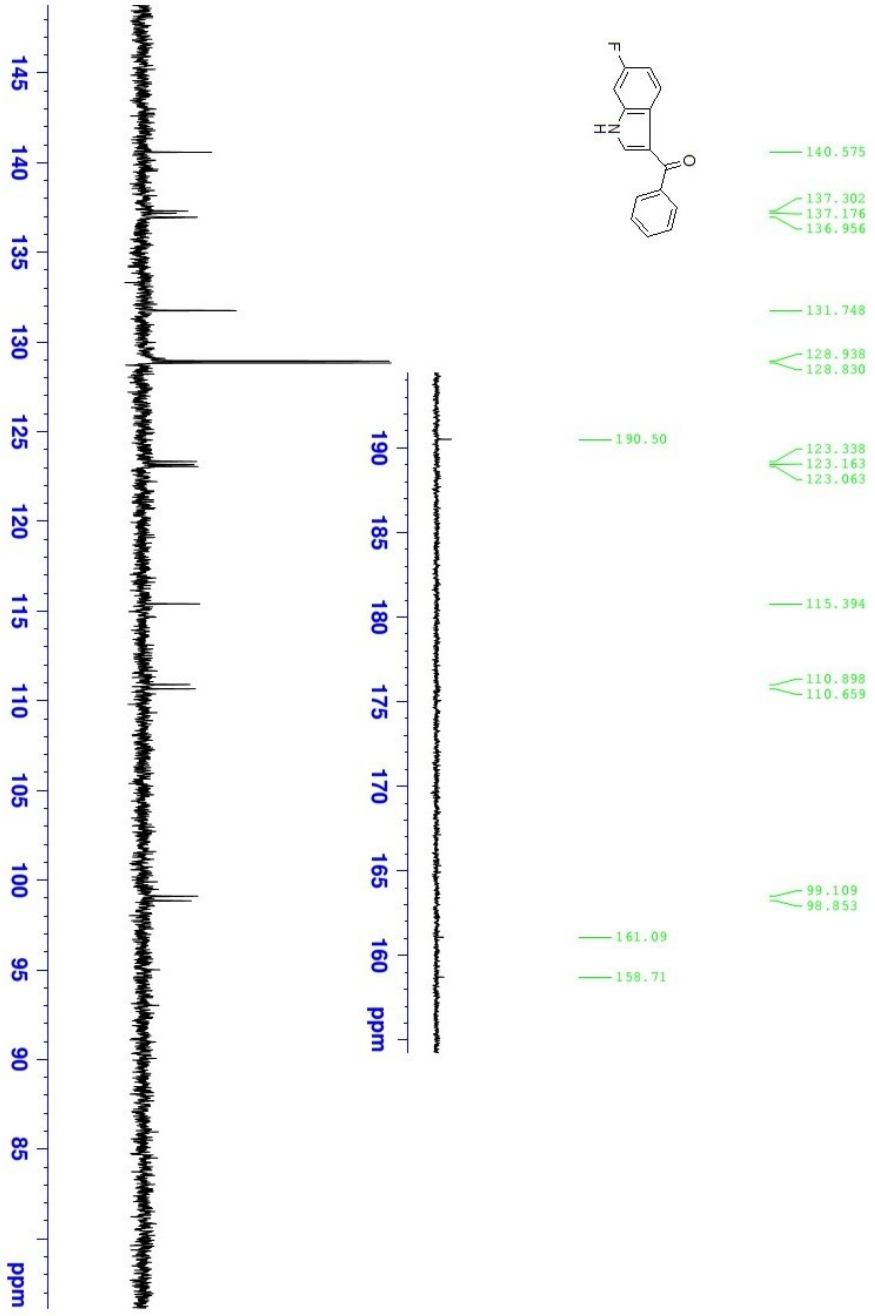
^{1}H NMR of Compound 3ia



^{13}C NMR of Compound 3ia



^{1}H NMR of Compound 3ja



^{13}C NMR of Compound 3ja