Supporting information

Direct C-2 acylation of indoles with toluene derivatives *via* Pd(II)-catalyzed direct C-H activation

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General experimental procedure

NMR spectra were recorded on a 300 MHz instrument using $CDCl_3$ as solvent unless and otherwise stated (s = singlet, d = doublet, t = triplet, m = multiplet). The ^1H and ^{13}C chemical shifts are reported in parts per million relative to tetramethylsilane as an internal standard. For the Mass spectrometry, ion source temperature was 150-250°C, as required. High-resolution EI-mass spectra were performed with a resolution of 10,000. For chromatography, analytical TLC plates and 70-230 mesh silica gel were used. All the solvents and chemicals were purchased and used as available.

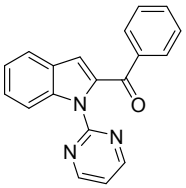
Typical experimental procedure

1-(pyrimidin-2-yl)-1*H*-indole (1 equiv, 0.2 mmol), $PdBr_2$ (10 mol%, 0.02 mmol), PivOH (0.5 equiv), PhCl (0.5 mL), toluene (1.5 mL), TBHP (5 equiv, 1.0 mmol), were consecutively loaded to an oven dried 10 mL screw cap vial equipped with a magnetic stirring bar and then stirred at 120 °C for 24 h. The resulting reaction mixture was cooled to the ambient temperature and subjected to the column chromatography to afford desired 2-oxindole. The products were further identified by ^1H NMR, ^{13}C NMR.

Removal of directing group

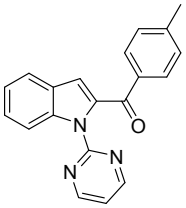
An oven-dried 10 mL screw-cap vial was charged with a mixture of **3c** (65.8 mg, 0.3 mmol), DMSO (2.0 mL) and EtONa (61.2 mg, 0.90 mmol), and the reaction mixture was stirred at 100 °C under nitrogen atmosphere for 24 h. After cooling to ambient temperature, the reaction mixture was diluted with EtOAc and washed with H_2O. The aqueous phase was extracted with EtOAc, and the combined organic phase was dried over Na_2SO_4. After filtration and evaporation under reduced pressure, the residue was purified by flash column chromatography (petroleum ether/ethyl acetate) on silica gel to give the product **3cc**.

Characterization of products



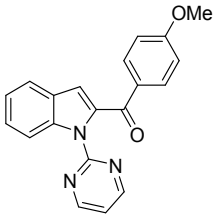
phenyl(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone(3a)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 2H), 8.40 (dd, J = 8.5, 0.8 Hz, 1H), 7.97 (dd, J = 8.3, 1.3 Hz, 2H), 7.71 (d, J = 7.8 Hz, 1H), 7.59–7.52 (m, 1H), 7.48–7.41 (m, 3H), 7.32–7.27 (m, 1H), 7.13 (d, J = 0.7 Hz, 1H), 7.07 (t, J = 4.8 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.6, 157.9, 157.3, 138.3, 138.0, 137.2, 132.7, 129.6, 128.3, 128.0, 126.5, 122.8, 122.5, 117.4, 115.4, 114.2.HRMS (ESI) for C19H14N3O [M+H]+ calculated 300.1131, found 300.1136.



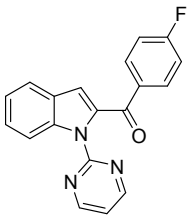
(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*p*-tolyl)methanone(3b)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 2H), 8.40 (dd, J = 8.5, 0.8 Hz, 1H), 7.89 (d, J = 8.2 Hz, 2H), 7.70 (d, J = 7.8 Hz, 1H), 7.44 (ddd, J = 8.5, 7.1, 1.3 Hz, 1H), 7.32–7.27 (m, 1H), 7.25 (d, J = 7.3 Hz, 2H), 7.10 (d, J = 0.7 Hz, 1H), 7.06 (t, J = 4.8 Hz, 1H), 2.42 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.4, 157.9, 157.4, 143.5, 138.2, 137.4, 135.4, 129.7, 129.1, 128.1, 126.3, 122.8, 122.4, 117.3, 115.0, 114.2, 21.7.HRMS (ESI) for C20H15N3O [M+H]+ calculated 314.1288, found 314.1291.



(4-methoxyphenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone (3c)

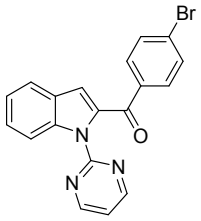
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (d, J = 4.8 Hz, 2H), 8.40 (dq, J = 8.4, 0.9 Hz, 1H), 8.04–7.94 (m, 2H), 7.70 (dt, J = 7.8, 1.0 Hz, 1H), 7.43 (ddd, J= 8.5, 7.1, 1.3 Hz, 1H), 7.34–7.26 (m, 1H), 7.13–7.03 (m, 2H), 6.98–6.90 (m, 2H), 3.87 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 186.5, 163.4, 157.9, 157.4, 138.1, 137.4, 131.9, 130.8, 128.1, 126.2, 122.7, 122.3, 117.3, 114.6, 114.2, 113.6, 55.5. HRMS (ESI) for C20H15N3O2 [M+H]+ calculated 300.1237, found 330.1234.



(4-fluorophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone(3d)

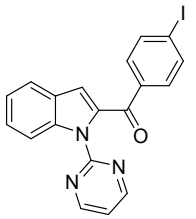
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 2H), 8.41 (dd, J = 8.5, 0.7 Hz, 1H), 8.01 (ddd, J = 8.9, 5.2, 2.5 Hz, 2H), 7.71 (d, J = 7.9 Hz, 1H), 7.46 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H), 7.34–7.27 (m, 1H), 7.17–7.06 (m, 4H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 186.2, 158.0, 157.2, 138.3, 136.9, 132.1, 132.0, 127.9, 126.6, 122.9, 122.5, 117.4, 115.7, 115.4, 115.3,

114.3.HRMS (ESI) for C19H12FN3O [M+H]+ calculated 318.1037, found 318.1042.



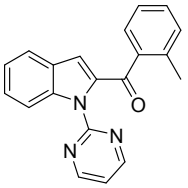
(4-bromophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone(3e)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 2H), 8.40 (dd, J = 8.5, 0.8 Hz, 1H), 7.97 (dd, J = 8.3, 1.3 Hz, 2H), 7.71 (d, J = 7.8 Hz, 1H), 7.59– 7.52 (m, 2H), 7.48–7.41 (m, 1H), 7.32–7.27 (m, 1H), 7.13 (d, J = 0.7 Hz, 1H), 7.07 (t, J = 4.8 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 186.6, 157.9, 157.2, 138.3, 136.9, 136.7, 131.7, 130.9, 128.0, 127.8, 126.7, 122.9, 122.5, 117.4, 115.3, 114.4.HRMS (ESI) for C19H12BrN3O [M+H]+ calculated 378.0237, found 378.0240.



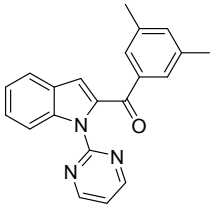
(4-iodophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone(3f)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (s, 2H), 8.42 (d, J = 8.4 Hz, 1H), 7.80 (d, J = 8.0 Hz, 2H), 7.69 (t, J = 10.4 Hz, 3H), 7.46 (t, J = 7.3 Hz, 1H), 7.31 (d, J = 7.4 Hz, 1H), 7.09 (d, J = 11.6 Hz, 2H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 186.8, 157.9, 157.2, 138.3, 137.7, 137.4, 136.7, 130.9, 128.0, 126.7, 122.9, 122.5, 117.4, 115.3, 114.4, 100.5.HRMS (ESI) for C19H12IN3O [M+H]+ calculated 426.0100, found 426.0094.



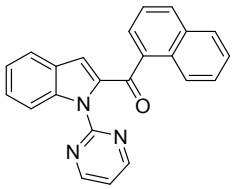
(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*o*-tolyl)methanone(3i)

^{1}H NMR (300 MHz, $CDCl_3$) δ8.68 (d, J = 4.8 Hz, 2H), 8.29 (dq, J = 8.5, 0.9 Hz, 1H), 7.68 (dt, J = 7.9, 1.0 Hz, 1H), 7.56 (dd, J = 7.6, 1.4 Hz, 1H), 7.43 (ddd, J = 8.5, 7.1, 1.3 Hz, 1H), 7.34 (td, J = 7.5, 1.5 Hz, 1H), 7.31–7.26 (m, 1H), 7.26–7.22 (m, 1H), 7.20–7.12 (m, 1H), 7.12–7.05 (m, 2H), 2.56 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 189.2, 157.9, 157.5, 138.7, 138.1, 131.2, 131.1, 130.3, 129.9, 127.8, 127.6, 126.7, 126.1, 125.1, 122.8, 122.7, 117.6, 116.3, 113.9, 20.4.HRMS (ESI) for C20H15N3O [M+H]+ calculated 314.1288, found 314.1293.



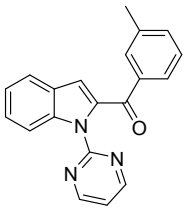
(3,5-dimethylphenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone (3j)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.66 (d, J = 4.8 Hz, 2H), 8.37 (dd, J = 8.5, 0.7 Hz, 1H), 7.71 (d, J = 7.8 Hz, 1H), 7.62 (s, 2H), 7.44 (ddd, J= 8.4, 7.2, 1.2 Hz, 1H), 7.34–7.26 (m, 1H), 7.21 (s, 1H), 7.12–7.05 (m, 2H), 2.36 (s, 6H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.9, 157.9, 157.4, 138.3, 138.0, 137.9, 137.4, 134.5, 127.9, 127.5, 126.4, 122.7, 122.5, 117.4, 115.3, 114.1, 21.2.HRMS (ESI) for C21H17N3O [M+H]+ calculated 328.1444, found 328.1449.



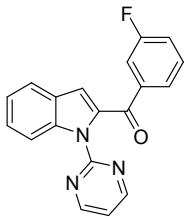
naphthalen-1-yl(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone(3k)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.74–8.67 (m, 1H), 8.54 (d, J = 4.8 Hz, 2H), 8.37 (dq, J = 8.5, 0.9 Hz, 1H), 7.97–7.83 (m, 2H), 7.79–7.66 (m, 2H), 7.65–7.50 (m, 2H), 7.49–7.35 (m, 2H), 7.29 (ddd, J = 8.1, 7.1, 1.0 Hz, 1H), 7.16 (d, J = 0.9 Hz, 1H), 6.96 (t, J = 4.8 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 188.8, 157.8, 157.3, 138.9, 138.5, 136.0, 133.7, 132.3, 131.1, 129.0, 128.2, 127.9, 127.6, 126.7, 126.4, 126.1, 124.2, 122.8, 122.6, 117.3, 116.2, 114.2.HRMS (ESI) for C23H15N3O [M+H]+ calculated 350.1288, found 350.1284.



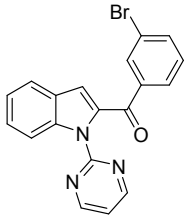
(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*m*-tolyl)methanone(3l)

^{1}H NMR (300 MHz, $CDCl_3$) δ8.65 (d, J = 4.8 Hz, 2H), 8.38 (dd, J = 8.5, 0.9 Hz, 1H), 7.81 (dq, J = 1.7, 0.8 Hz, 1H), 7.78 (dd, J = 7.2, 1.8 Hz, 1H), 7.71 (dt, J = 7.9, 1.0 Hz, 1H), 7.44 (ddd, J = 8.5, 7.1, 1.3 Hz, 1H), 7.40–7.26 (m, 3H), 7.12 (d, J = 0.8 Hz, 1H), 7.08 (t, J = 4.8 Hz, 1H), 2.40 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.7, 157.9, 157.4, 138.3, 138.2, 137.9, 137.3, 133.6, 130.1, 128.2, 128.0, 126.9, 126.5, 122.8, 122.5, 117.4, 115.4, 114.2, 21.3.HRMS (ESI) for C20H15N3O [M+H]+ calculated 314.1288, found 314.1284.



(3-fluorophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone (3m)

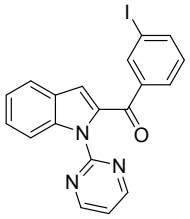
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (d, J = 4.9 Hz, 2H), 8.42 (dd, J = 8.5, 0.9 Hz, 1H), 7.78–7.63 (m, 3H), 7.51–7.36 (m, 2H), 7.31 (ddd, J= 8.1, 7.1, 1.0 Hz, 1H), 7.27–7.21 (m, 1H), 7.15 (d, J = 0.8 Hz, 1H), 7.08 (t, J = 4.8 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 186.2, 163.9, 161.4, 157.9, 157.2, 140.2, 140.1, 138.3, 136.7, 130.0, 129.9, 127.9, 126.7, 125.3, 125.2, 122.9, 122.6, 119.8, 119.5, 117.4, 116.2, 115.9, 115.6, 114.4.HRMS (ESI) for C19H12FN3O [M+H]+ calculated 318.1037, found 318.1035.



(3-bromophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone (3n)

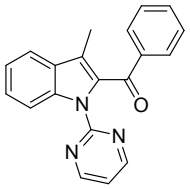
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (d, J = 4.8 Hz, 2H), 8.42 (dq, J = 8.5, 0.9 Hz, 1H), 8.13 (t, J = 1.8 Hz, 1H), 7.87 (ddd, J= 7.7, 1.6, 1.1 Hz, 1H), 7.76–7.65 (m, 2H), 7.47 (ddd, J = 8.5, 7.2, 1.3 Hz, 1H), 7.36–7.28 (m, 2H), 7.14 (d, J = 0.8 Hz, 1H), 7.09 (t, J = 4.9 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 185.9, 157.9, 157.2, 139.9, 138.4, 136.5, 135.5, 132.6, 129.9, 128.0, 127.9, 126.8, 122.9, 122.6, 122.6, 117.4, 115.7, 114.4.HRMS (ESI) for C19H12BrN3O [M+H]+ calculated

378.0237, found 378.0236.



(3-iodophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone (3o)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.66 (d, J = 4.8 Hz, 2H), 8.41 (dq, J = 8.5, 0.9 Hz, 1H), 8.33 (t, J = 1.7 Hz, 1H), 7.95–7.83 (m, 2H), 7.72 (dt, J = 7.9, 1.0 Hz, 1H), 7.46 (ddd, J = 8.5, 7.1, 1.3 Hz, 1H), 7.31 (ddd, J = 8.0, 7.1, 1.0 Hz, 1H), 7.18 (t, J = 7.8 Hz, 1H), 7.13 (d, J = 0.8 Hz, 1H), 7.10 (t, J = 4.8 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 185.9, 157.9, 157.2, 141.4, 139.82, 138.4, 138.2, 136.5, 130.0, 128.7, 127.9, 126.8, 122.9, 122.6, 117.5, 115.8, 114.3, 94.1.HRMS (ESI) for C19H12IN3O [M+H]+ calculated 426.0100, found 426.0097.

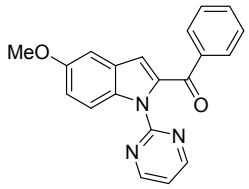


(3-methyl-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone(3p)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, J = 8.4 Hz, 1H), 8.46 (d, J = 4.8 Hz, 2H), 7.78 (d, J = 7.4 Hz, 2H), 7.69 (d, J = 7.8 Hz, 1H), 7.51 – 7.39 (m, 2H), 7.33 (q, J = 7.4 Hz, 3H), 6.85 (t, J = 4.8 Hz, 1H), 2.37 (s, 3H).

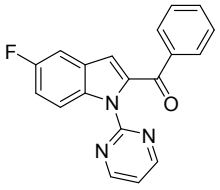
^{13}C NMR (101 MHz, $CDCl_3$) δ 189.47, 157.6, 157.1, 139.3, 136.4, 133.3, 132.3, 130.3, 128.6, 128.3, 126.1, 122.6, 121.7, 120.2, 116.1, 115.2, 9.4.

HRMS (ESI) for C20H15N3O [M+H]+ calculated 314.1288, found 314.1284.



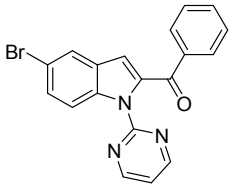
(5-methoxy-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone(3q)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.59 (d, J = 4.8 Hz, 2H), 8.34 (d, J = 9.0 Hz, 1H), 7.98–7.91 (m, 2H), 7.57–7.49 (m, 1H), 7.46–7.39 (m, 2H), 7.13–7.06 (m, 2H), 7.05 (d, J = 0.6 Hz, 1H), 7.02 (t, J = 4.8 Hz, 1H), 3.88 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.6, 157.9, 157.2, 156.0, 138.1, 137.7, 133.2, 132.6, 129.5, 128.7, 128.3, 117.1, 116.5, 115.5, 114.9, 103.5, 55.7.HRMS (ESI) for C20H15N3O2 [M+H]+ calculated 330.1237, found 330.1237.



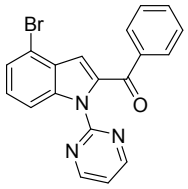
(5-fluoro-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone(3r)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 2H), 8.46–8.37 (m, 1H), 7.94 (d, J = 6.9 Hz, 2H), 7.55 (s, 1H), 7.44 (d, J = 6.8 Hz, 2H), 7.34 (d, J = 9.0 Hz, 1H), 7.17 (t, J = 9.0 Hz, 1H), 7.05 (s, 2H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.6, 160.4, 158.0, 157.9, 157.0, 138.5, 137.8, 134.5, 132.8, 129.5, 128.6, 128.6, 128.6, 128.4, 127.6, 126.9, 117.4, 115.8, 115.7, 114.7, 114.5, 114.2, 114.2, 107.3, 107.1.HRMS (ESI) for C19H12FN3O [M+H]+ calculated 318.1037, found 318.1036.



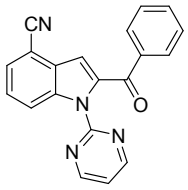
(5-bromo-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone (3s)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 2H), 8.33 (d, J = 8.8 Hz, 1H), 7.94 (d, J = 6.5 Hz, 2H), 7.82 (s, 1H), 7.60–7.48 (m, 2H), 7.48–7.26 (m, 2H), 7.06 (s, 1H), 7.02 (s, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.4, 157.9, 156.9, 138.1, 137.7, 136.6, 132.9, 129.7, 129.5, 129.1, 128.4, 124.7, 117.6, 116.1, 115.9, 113.6.HRMS (ESI) for C19H12BrN3O [M+H]+ calculated 378.0237, found 378.0230.



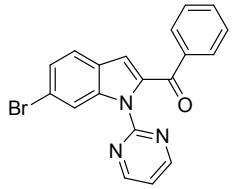
(4-bromo-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone(3t)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 2H), 8.35 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 7.0 Hz, 2H), 7.57 (d, J = 6.6 Hz, 1H), 7.46 (d, J = 7.0 Hz, 3H), 7.29 (t, J = 10.6 Hz, 1H), 7.16 (s, 1H), 7.10 (s, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.3, 158.1, 157.1, 138.3, 137.6, 137.5, 132.9, 129.6, 128.8, 128.5, 127.2, 125.7, 117.8, 116.1, 114.5, 113.4.HRMS (ESI) for C19H12BrN3O [M+H]+ calculated 378.0237, found 378.0240.



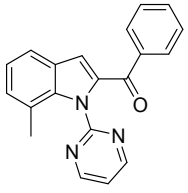
2-benzoyl-1-(pyrimidin-2-yl)-1*H*-indole-4-carbonitrile (3u)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.72–8.63 (m, 3H), 8.04–7.95 (m, 2H), 7.68–7.59 (m, 2H), 7.50 (ddd, J = 8.5, 7.2, 1.1 Hz, 3H), 7.27 (d, J = 0.9 Hz, 1H), 7.16 (t, J = 4.9 Hz, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.20, 158.19, 156.69, 139.00, 137.48, 137.14, 133.35, 129.70, 129.41, 128.59, 127.77, 125.79, 119.35, 118.19, 117.66, 111.76, 105.08.HRMS (ESI) for C20H12N4O [M+H]+ calculated 325.1084, found 325.1085.



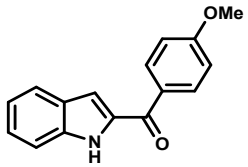
(6-bromo-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone (3v)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67–8.59 (m, 3H), 7.99–7.91 (m, 2H), 7.60–7.51 (m, 2H), 7.49–7.37 (m, 3H), 7.10–7.05 (m, 2H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.3, 158.0, 156.9, 138.6, 137.7, 137.6, 132.8, 129.5, 128.4, 126.8, 126.3, 123.5, 120.3, 117.6, 117.5, 114.6.HRMS (ESI) for C19H12BrN3O [M+H]+ calculated 378.0237, found 378.0238.



(7-methyl-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone(3w)

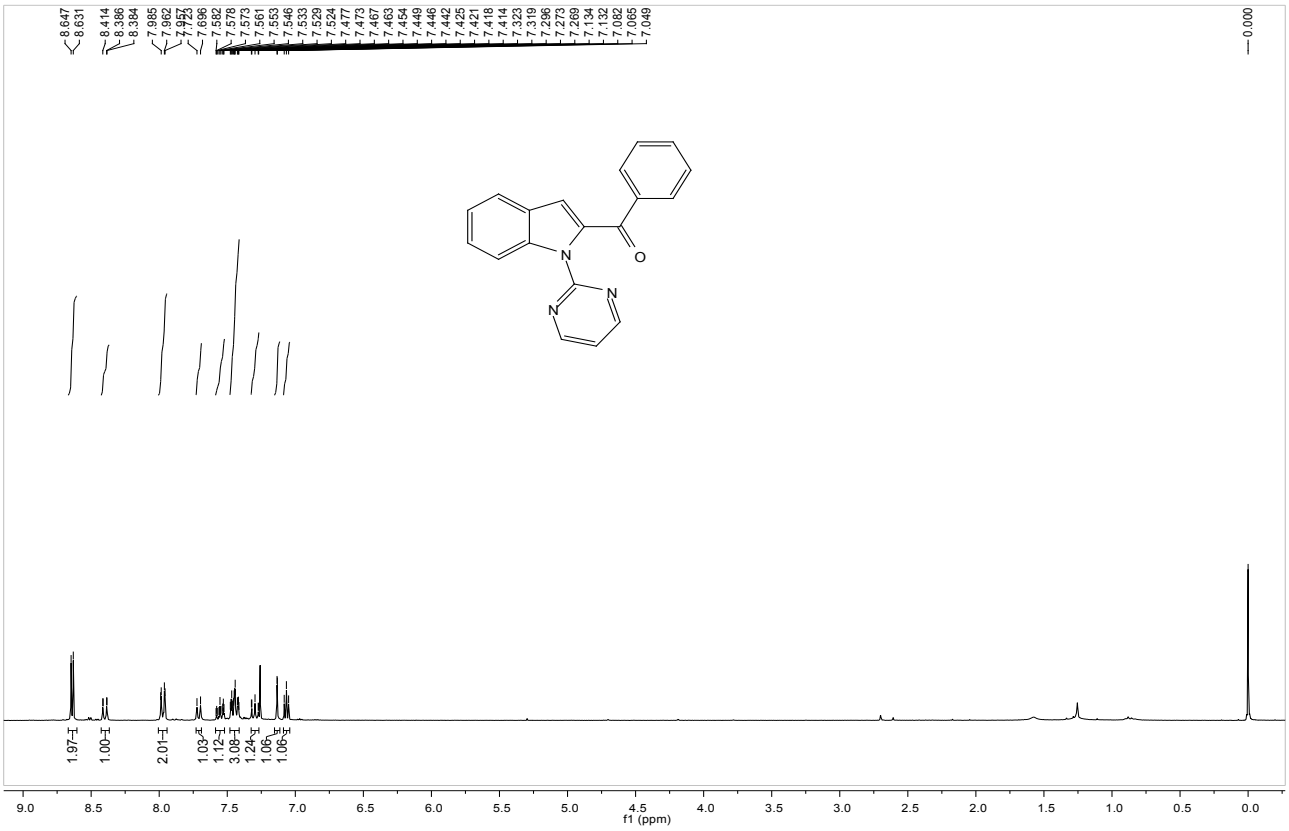
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.88 (d, J = 4.9 Hz, 2H), 7.95–7.90 (m, 2H), 7.62–7.56 (m, 2H), 7.52–7.45 (m, 2H), 7.43 (t, J = 4.9 Hz, 1H), 7.21 (s, 1H), 7.15–7.11 (m, 2H), 1.97 (s, 3H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 187.1, 159.8, 158.2, 138.4, 138.3, 136.1, 132.4, 129.7, 129.3, 128.3, 127.3, 122.5, 121.9, 121.1, 120.2, 116.5, 19.4.HRMS (ESI) for C20H15N3O [M+H]+ calculated 314.1288, found 314.1288.

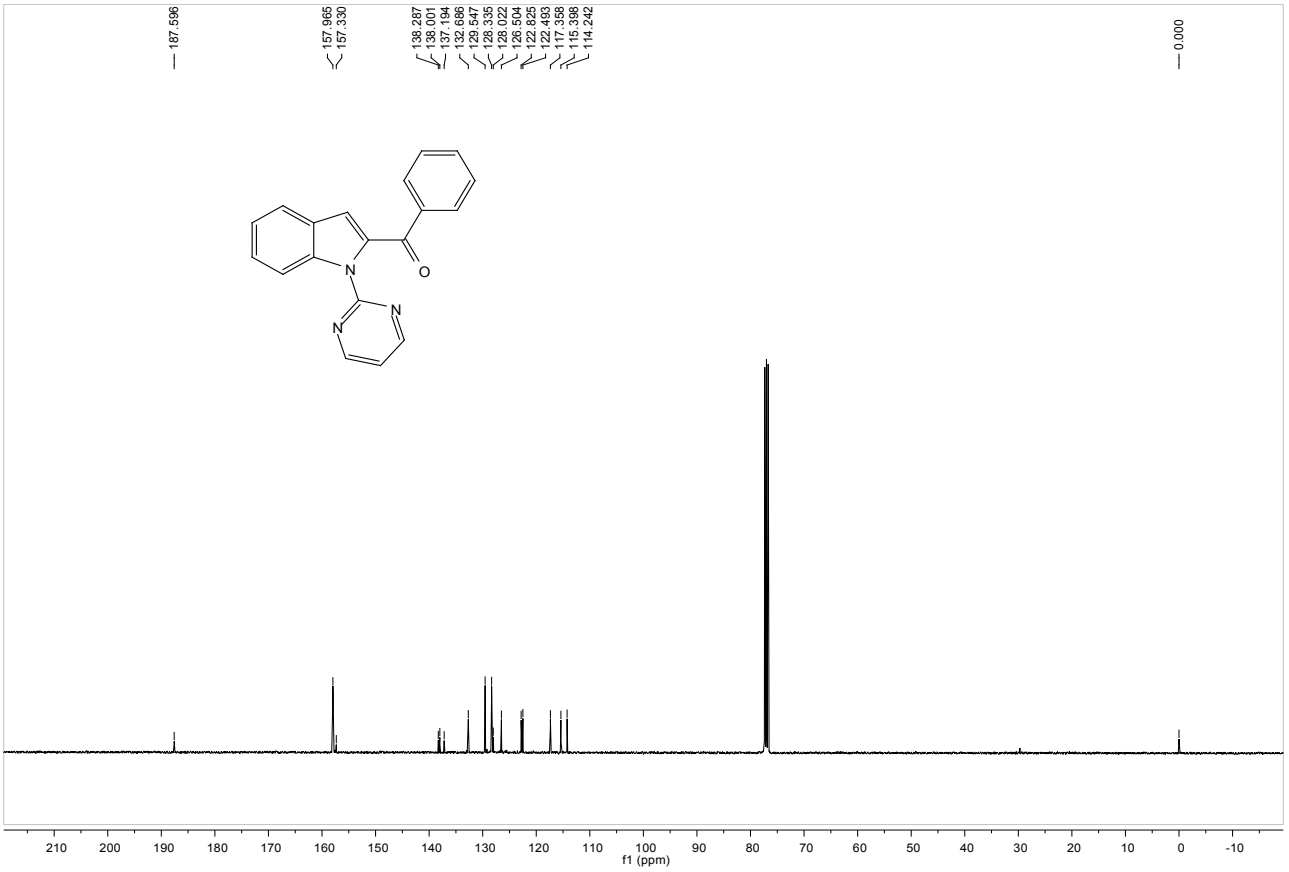


(1H-indol-2-yl)(4-methoxyphenyl)methanone (3cc)

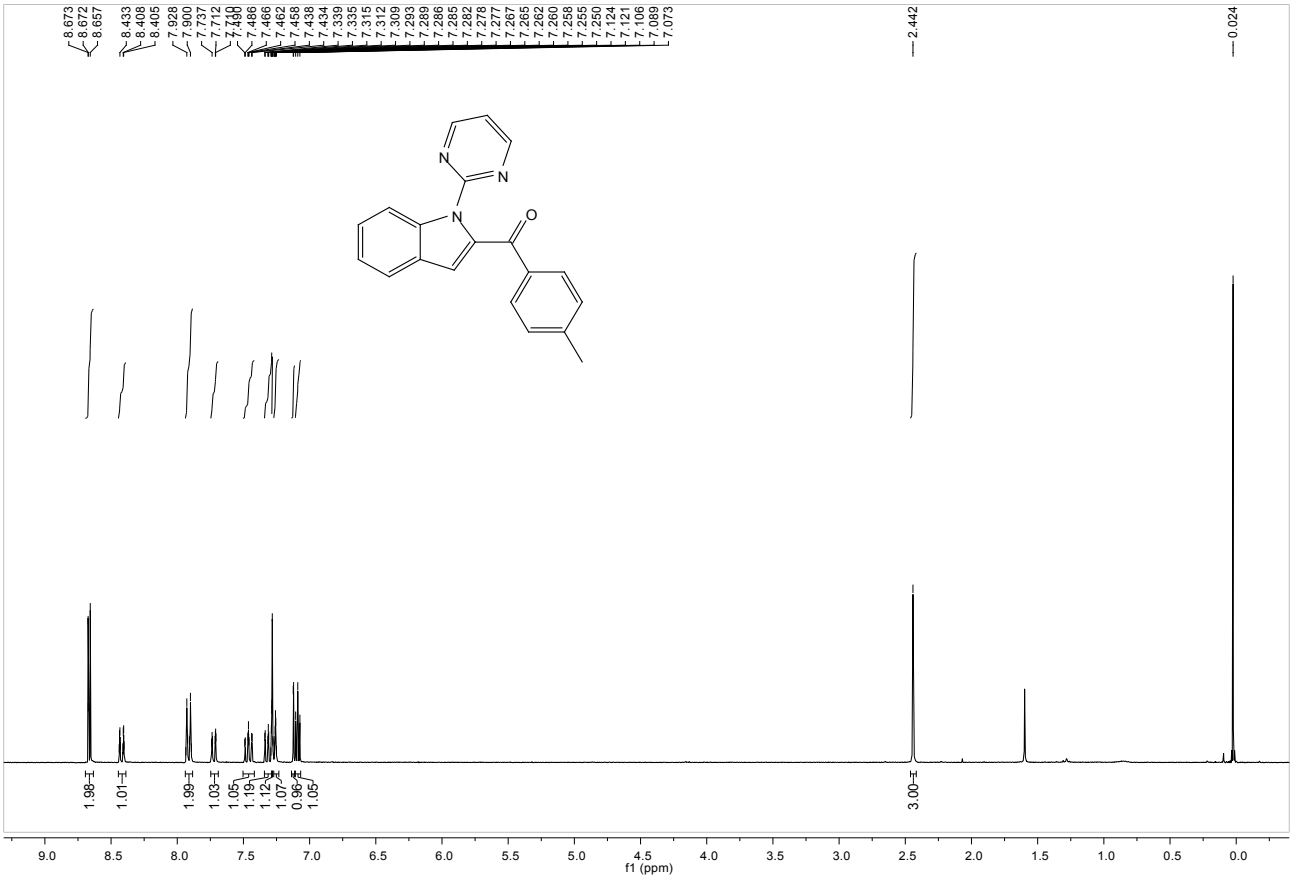
^{1}H NMR (400 MHz, Chloroform-d) δ 9.32 (s, 1H), 8.04 (d, J = 8.8 Hz, 2H), 7.72 (d, J = 8.0 Hz, 1H), 7.48 (d, J = 8.3 Hz, 1H), 7.37 (t, J = 7.7 Hz, 1H), 7.21 – 7.12 (m, 2H), 7.03 (d, J = 8.8 Hz, 2H), 3.91 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 185.9, 163.4, 137.4, 134.6, 131.7, 130.8, 127.9, 126.3, 123.2, 121.1, 113.9, 112.2, 111.9, 77.2, 55.7.

^{1}H and ^{13}C-NMR Spectra of the Products





3a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7.466
7.462
7.458
7.438
7.434
7.339
7.335
7.315
7.312
7.309
7.293
7.289
7.286
7.285
7.282
7.278
7.277
7.267
7.265
7.262
7.260
7.258
7.255
7.250
7.124
7.121
7.106
7.089
7.073
2.442
0.024
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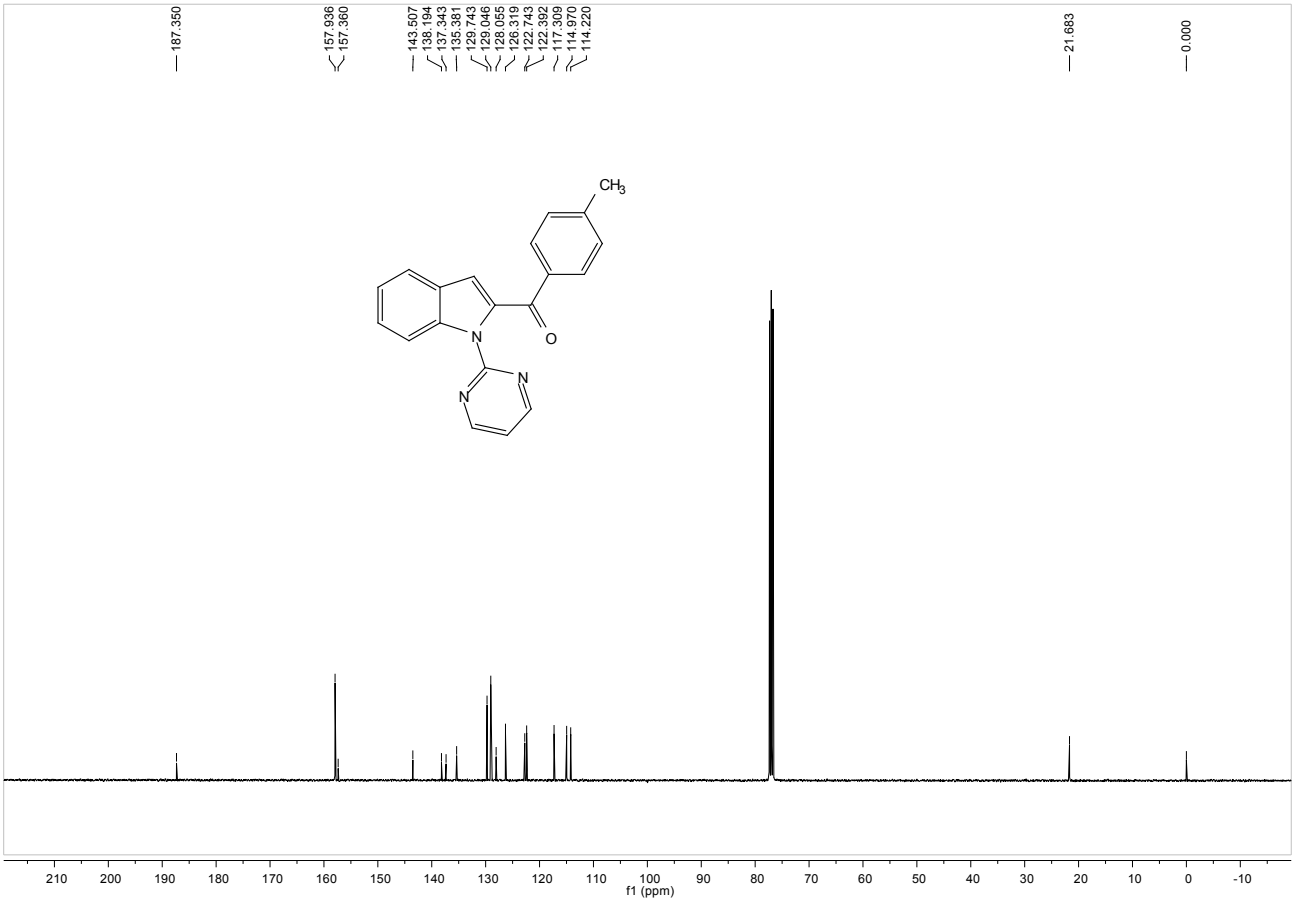
1.01
1.99
1.03
1.05
1.19
1.12
1.07
0.96
1.05
3.00
f1 (ppm)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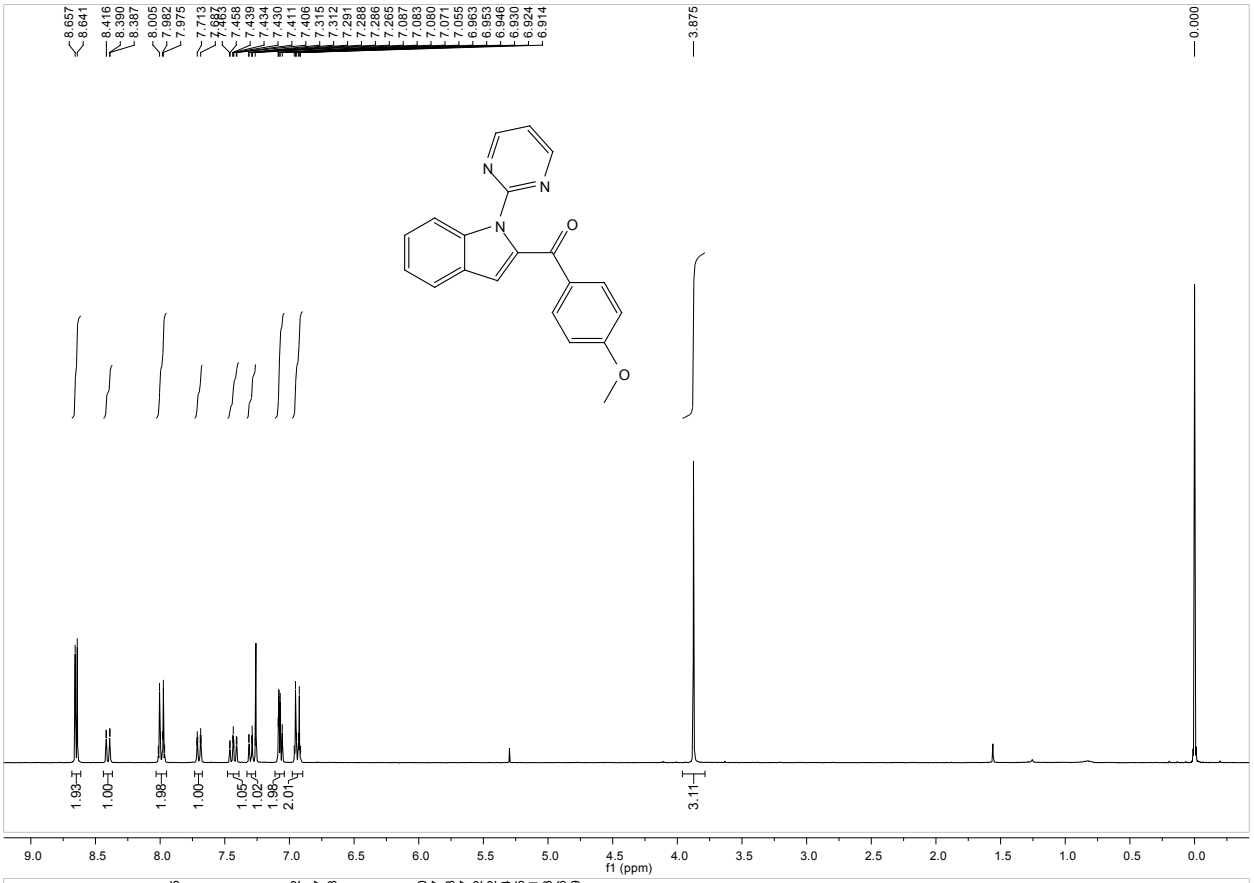
0.000
f1 (ppm)

3b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7.439
7.434
7.430
7.411
7.406
7.315
7.312
7.291
7.288
7.286
7.285
7.087
7.083
7.080
7.077
7.055
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6.953
6.946
6.930
6.924
6.914
3.875
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1.98
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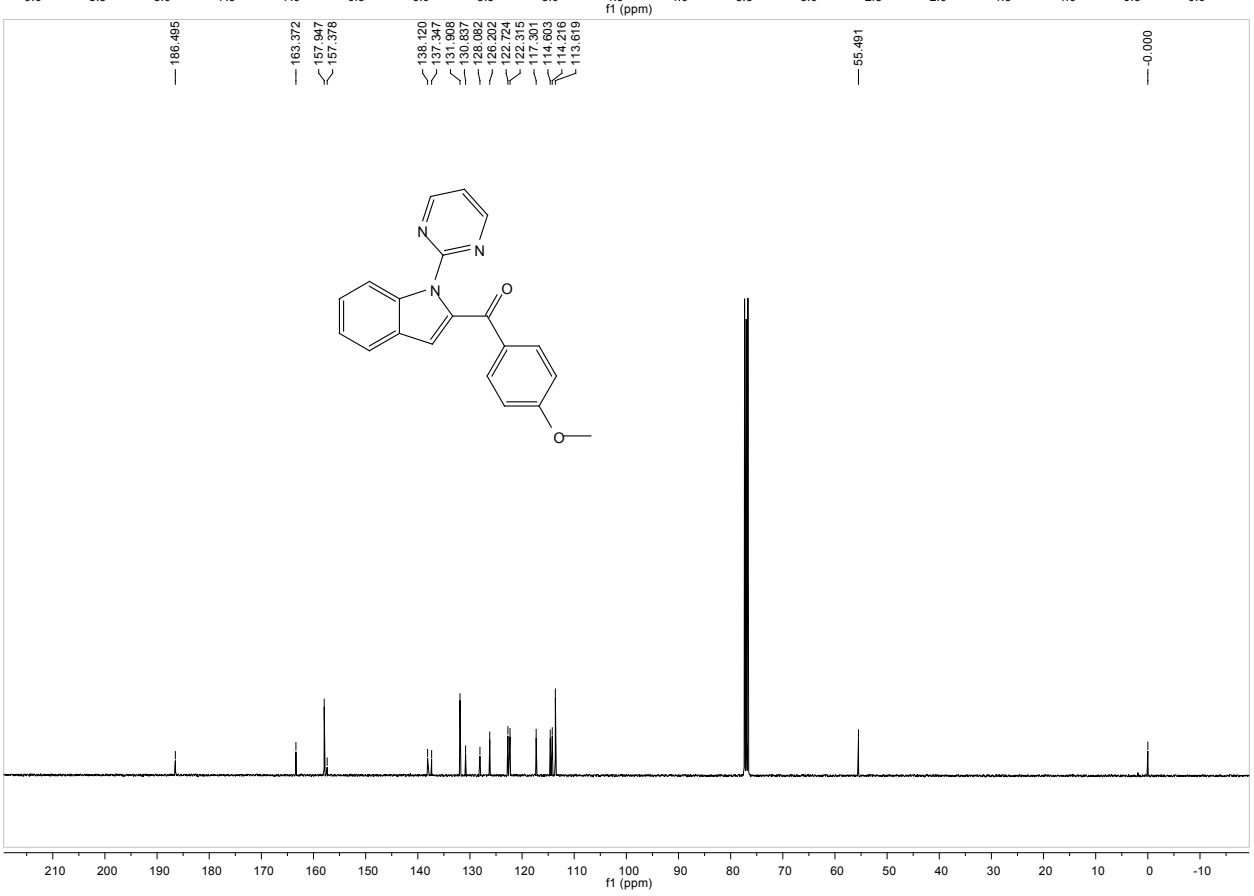
O
O
f1 (ppm)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114.216
113.619
55.491
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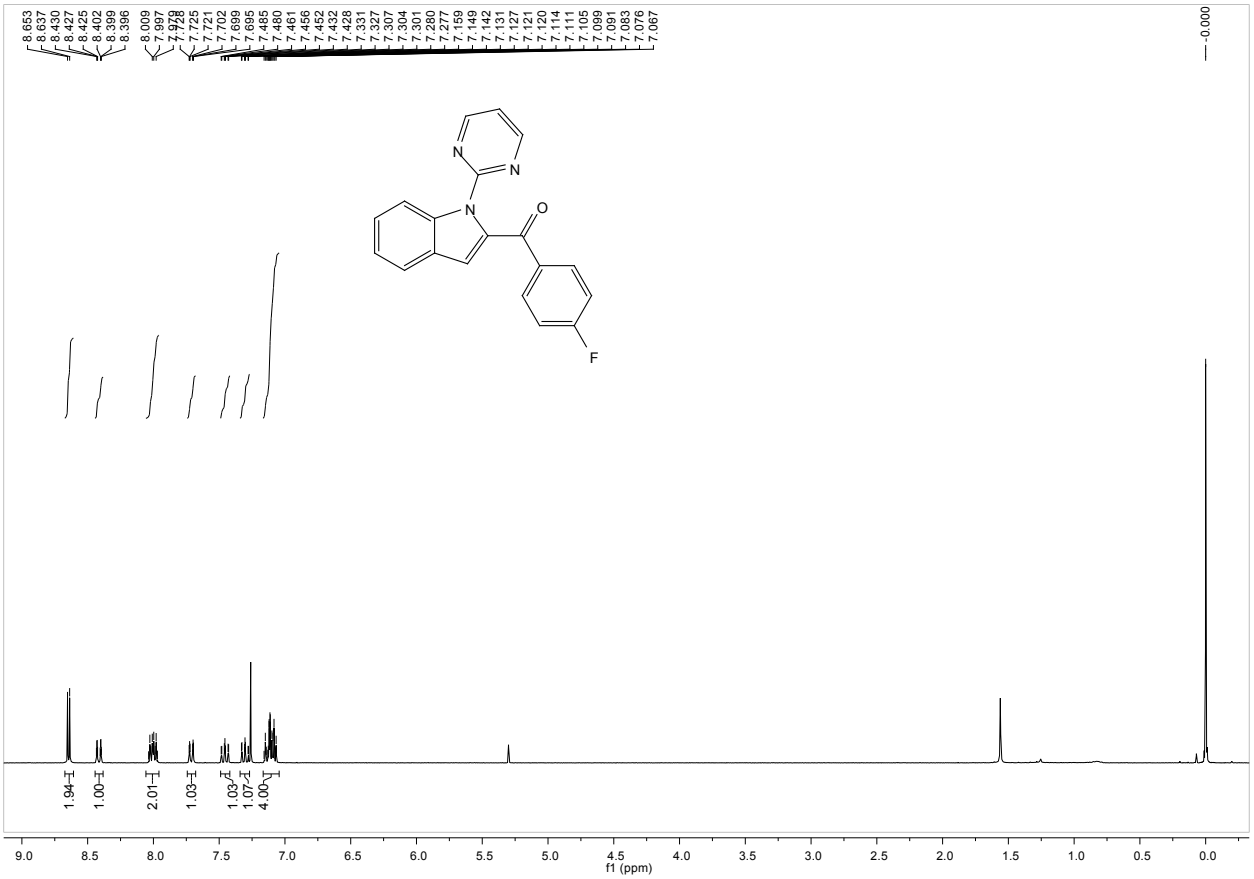
N
N
N
O
O
f1 (ppm)

3c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7.728
7.725
7.721
7.702
7.699
7.695
7.485
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7.461
7.456
7.452
7.432
7.428
7.351
7.327
7.307
7.304
7.301
7.280
7.277
7.159
7.149
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7.127
7.121
7.120
7.114
7.111
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7.083
7.076
7.067
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N
N
O
F
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2.01
1.03
1.03
1.07
4.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
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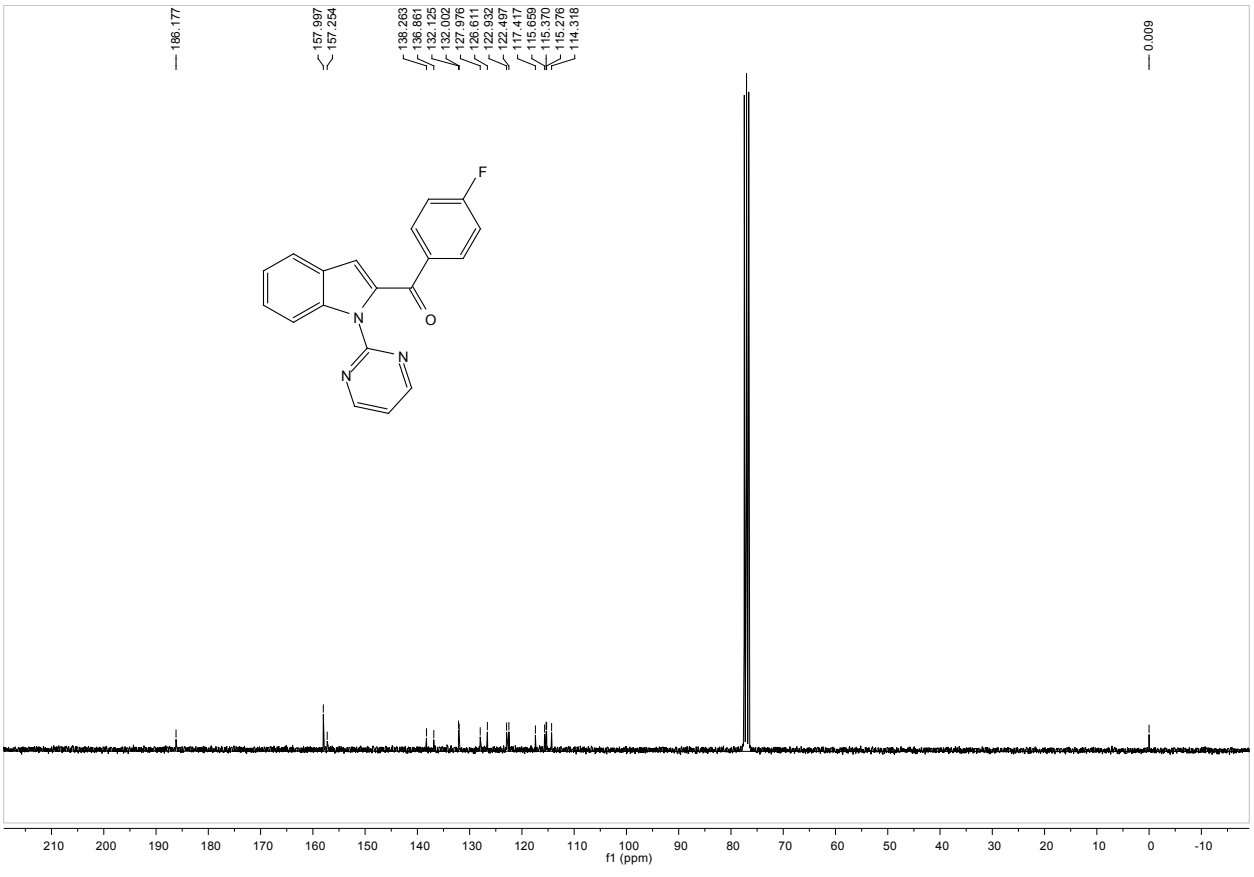
f1 (ppm)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115.370
115.276
114.318
0.009
F
O
N
N
N
210
200
190
180
170
160
150
140
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120
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100
90
80
70
60
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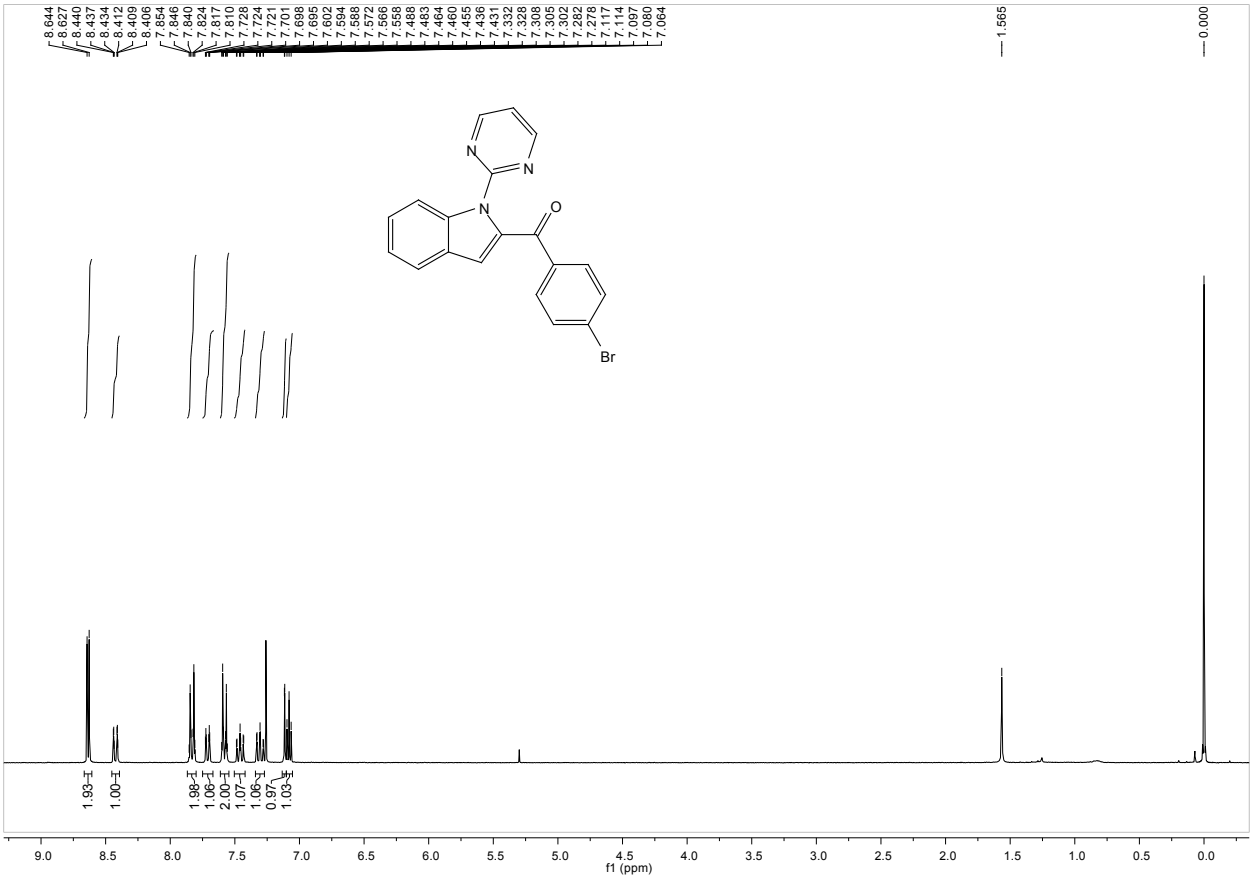
40
30
20
10
0
-10
f1 (ppm)

3d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7.278
7.117
7.114
7.097
7.080
7.064
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N
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1.06
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1.06
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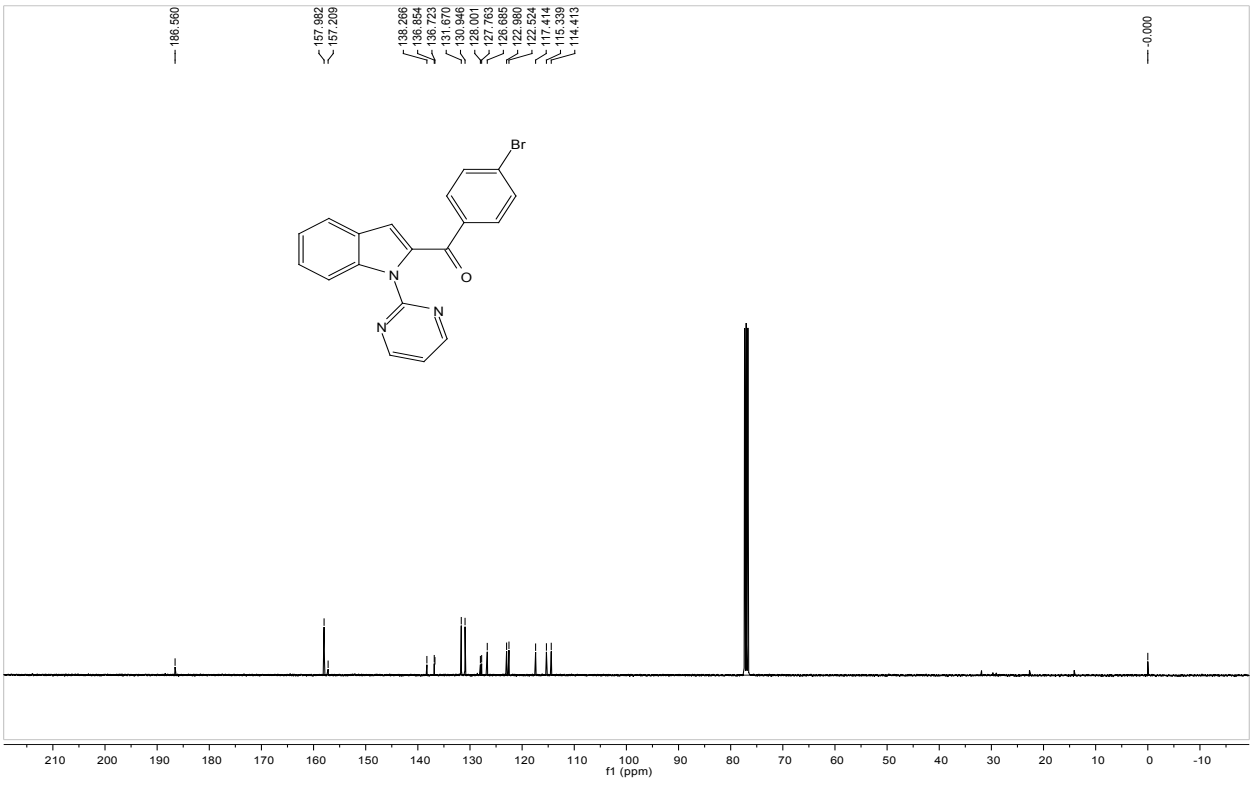
f1 (ppm)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114.413
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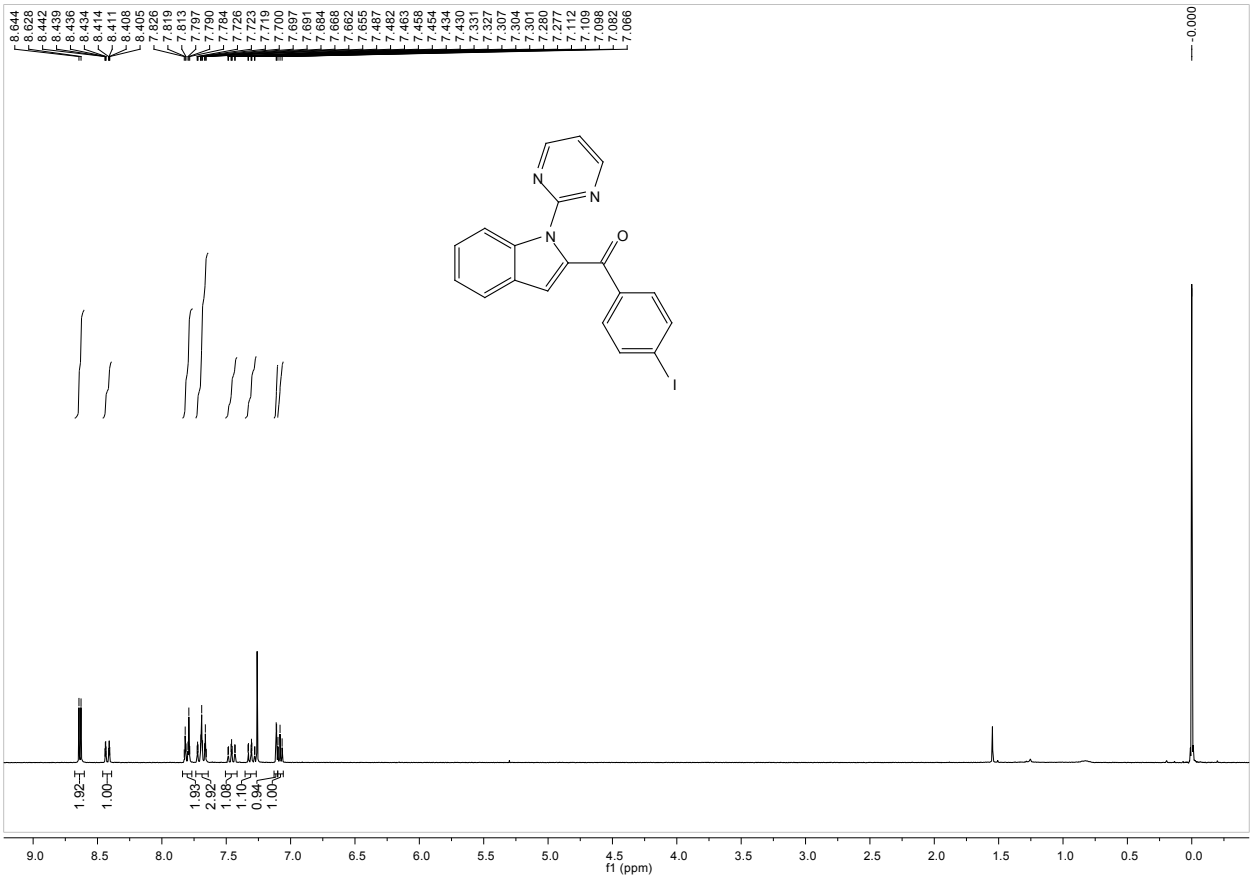
Br
O
N
N
N
f1 (ppm)

3e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7.813
7.797
7.790
7.784
7.726
7.723
7.719
7.700
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7.684
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7.662
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7.066
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N
O
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1.10
0.94
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8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
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1.0
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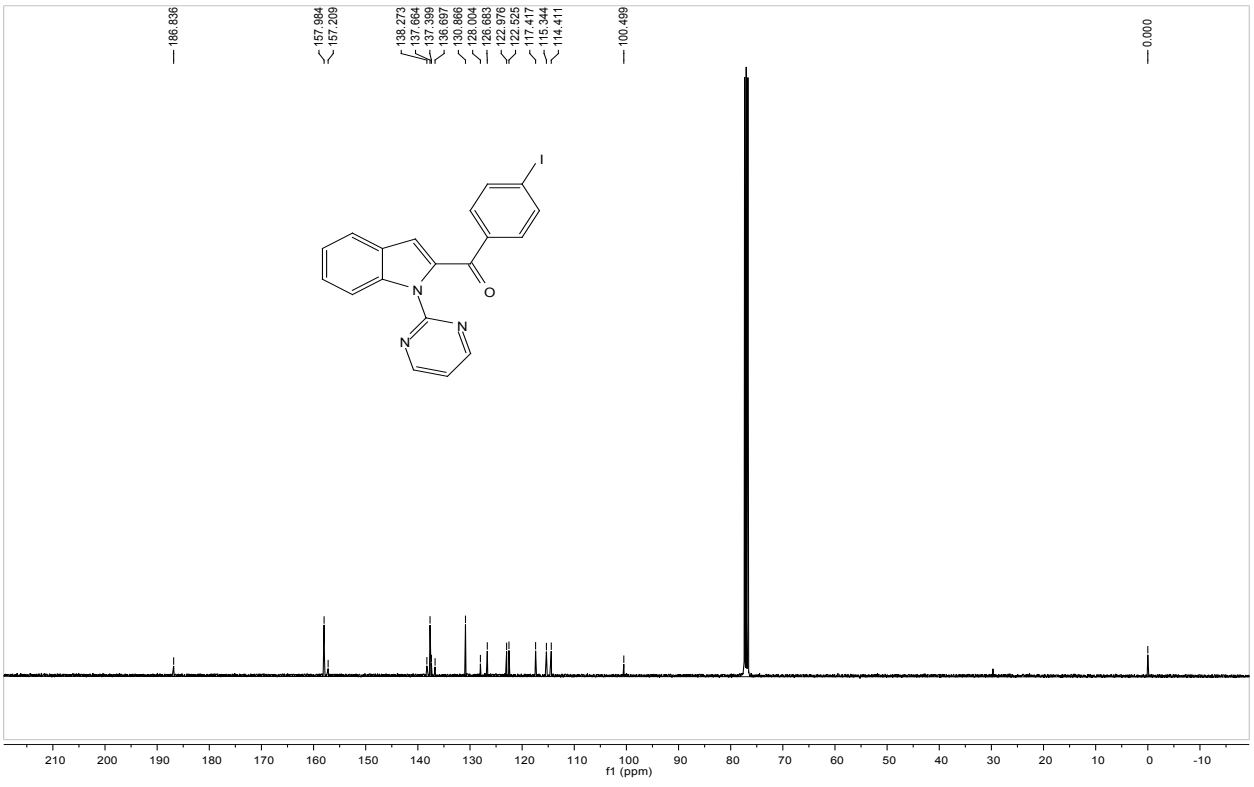
0.0
f1 (ppm)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I
O
N
N
N
210
200
190
180
170
160
150
140
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120
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100
90
80
70
60
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40
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20
10
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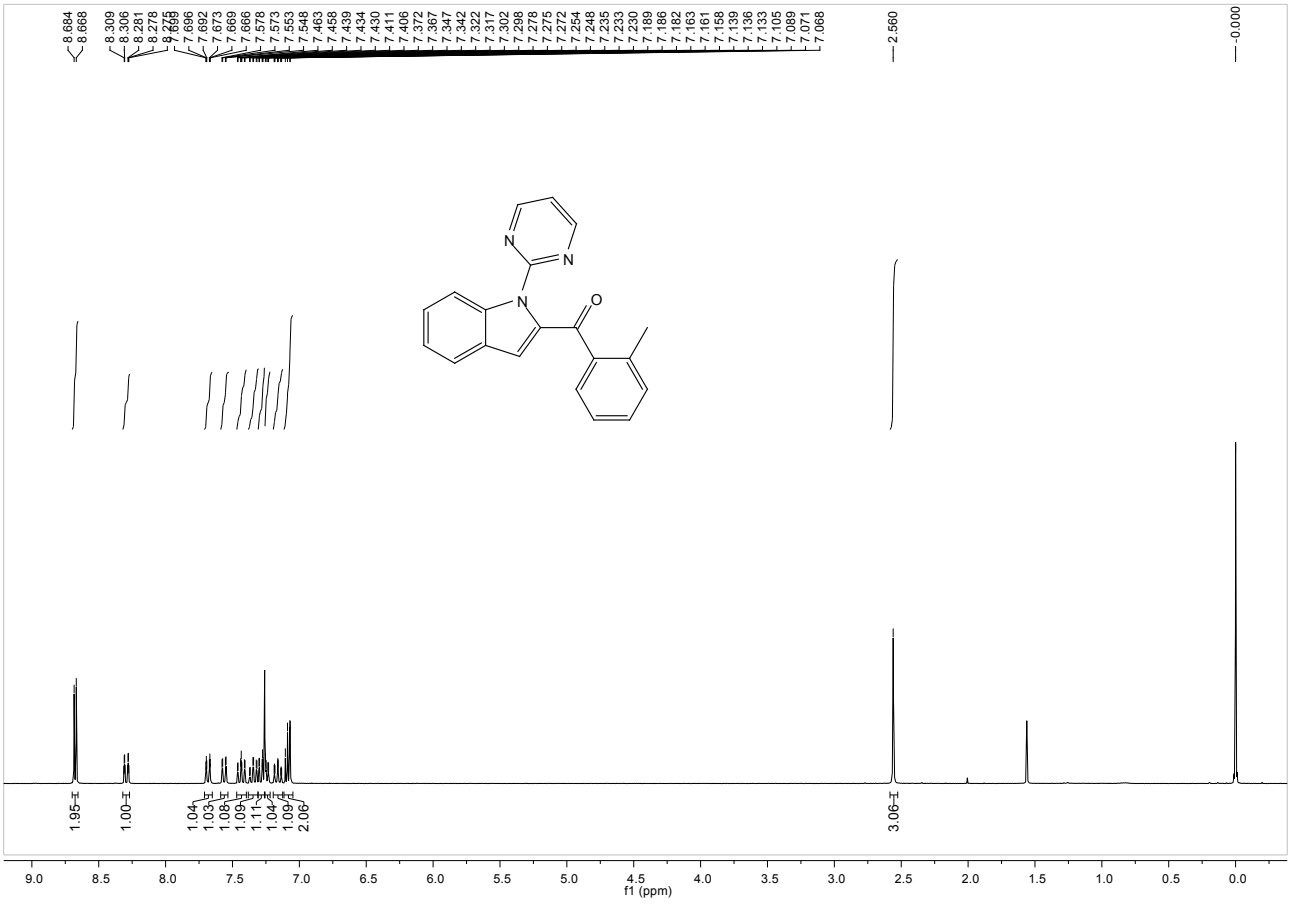
f1 (ppm)

3f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7.233
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7.182
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7.161
7.158
7.139
7.136
7.133
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1.03
1.08
1.09
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1.09
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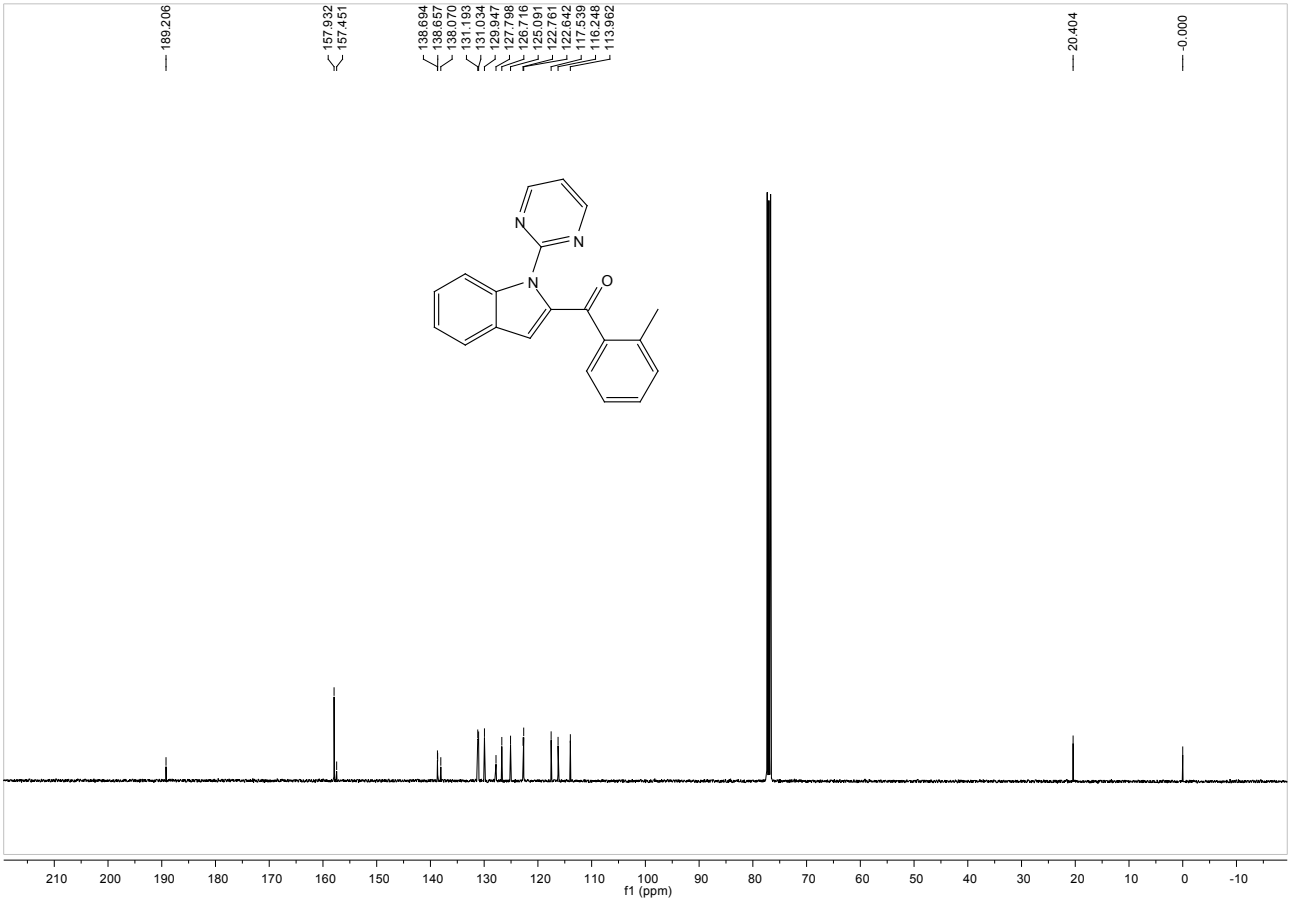
3.06
f1 (ppm)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122.642
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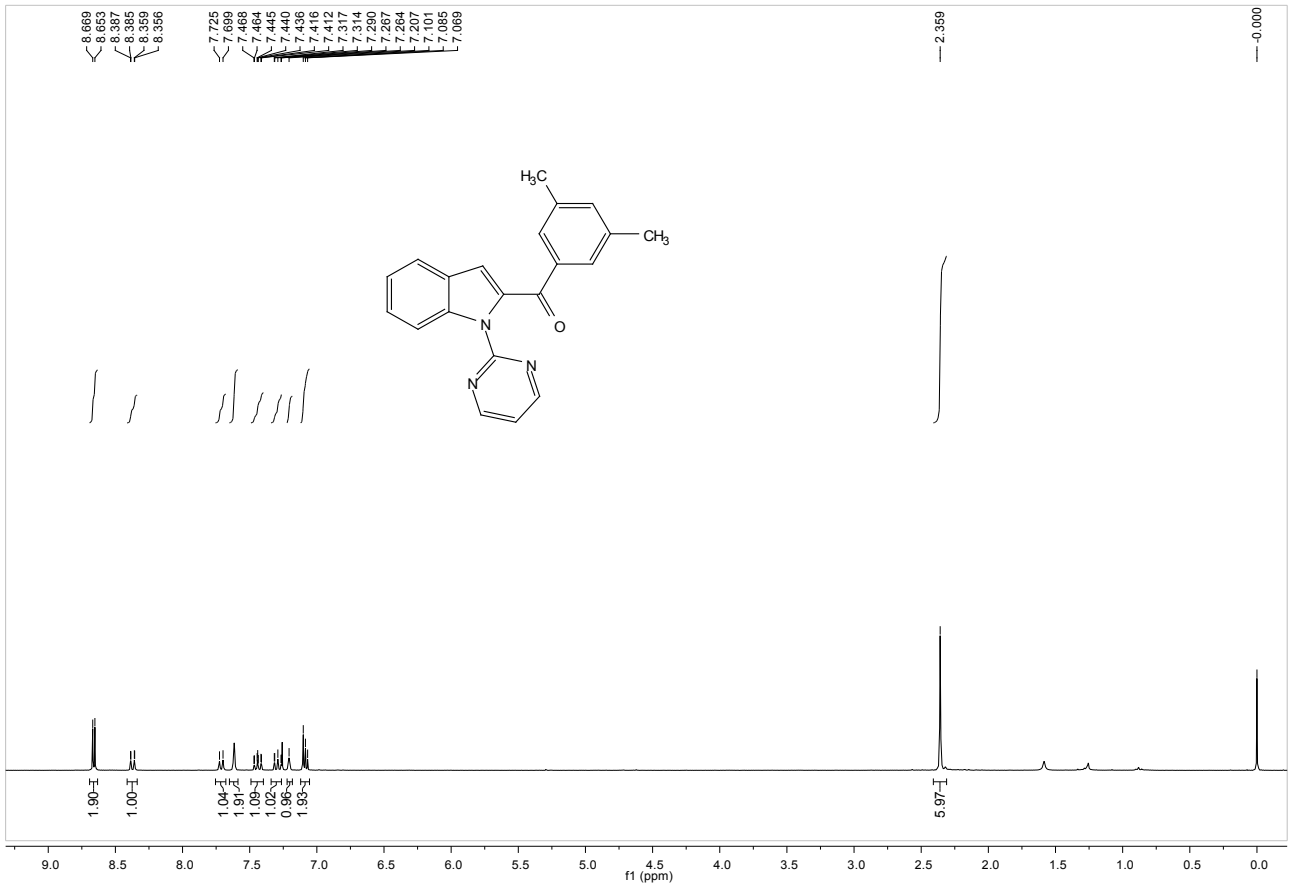
f1 (ppm)

3i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7.436
7.416
7.412
7.317
7.314
7.290
7.267
7.264
7.207
7.101
7.085
7.069
2.359
-0.000
H3C
CH3
O
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N
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1.00
1.04
1.91
1.09
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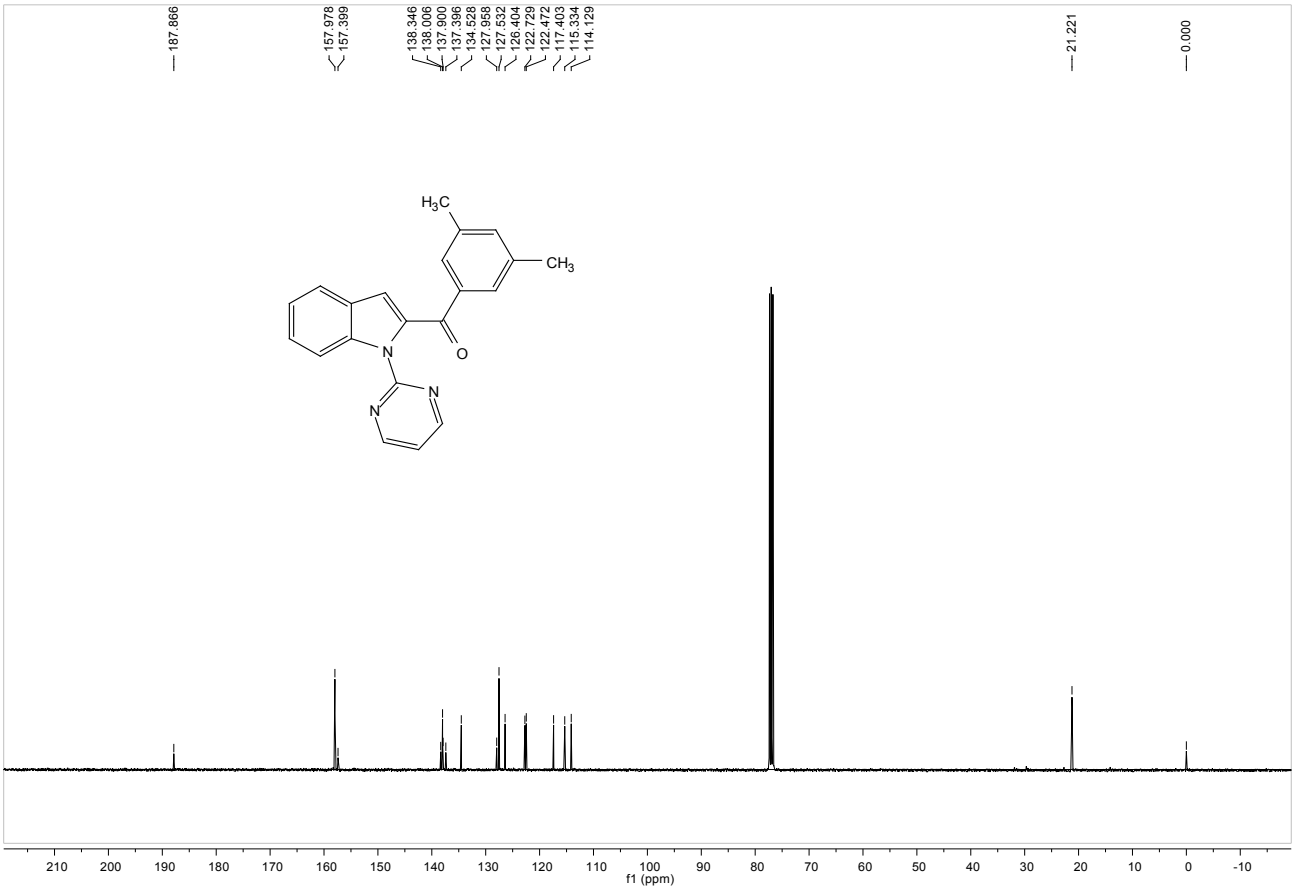
1.93
5.97
f1 (ppm)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114.129
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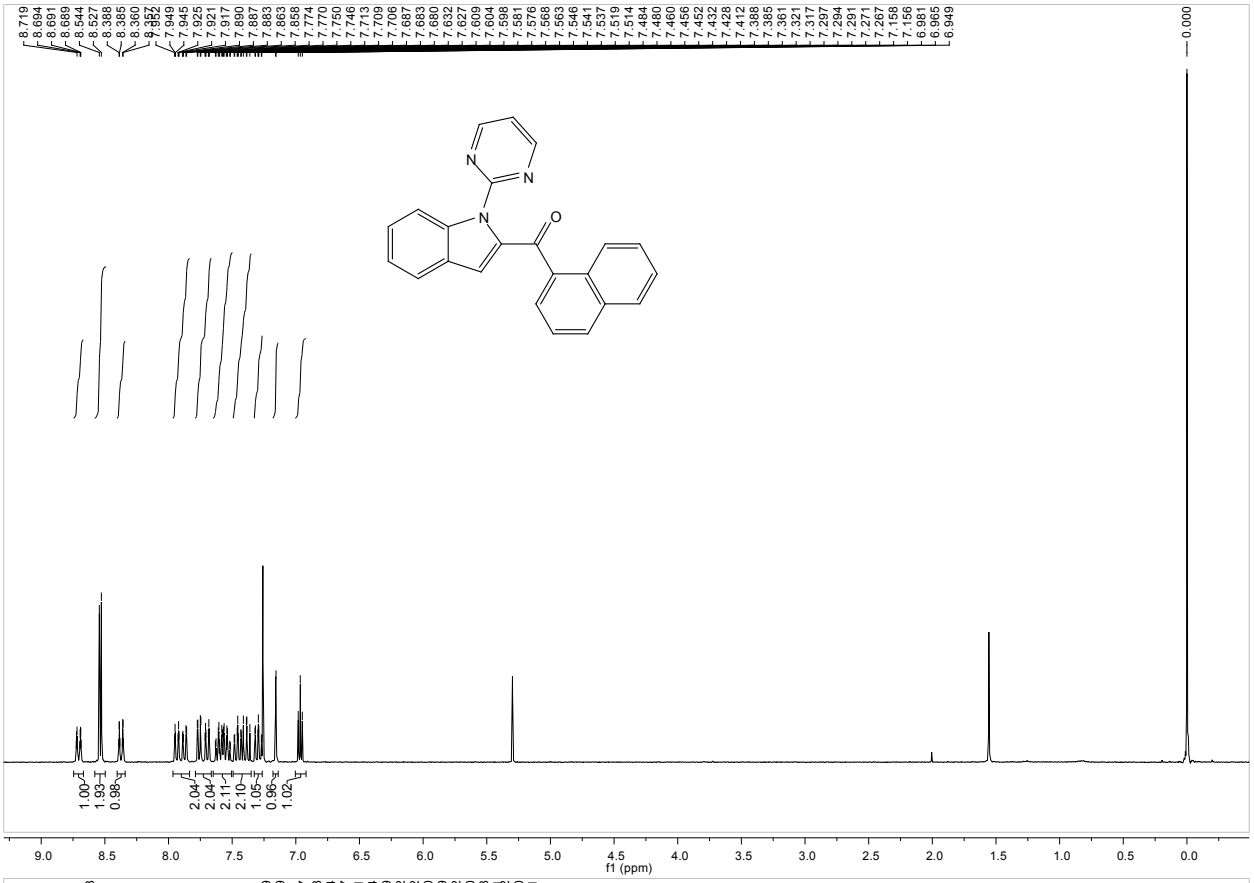
0.000
H3C
CH3
O
N
N
N
f1 (ppm)

3j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2.10
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0.96
1.02
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N
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8.0
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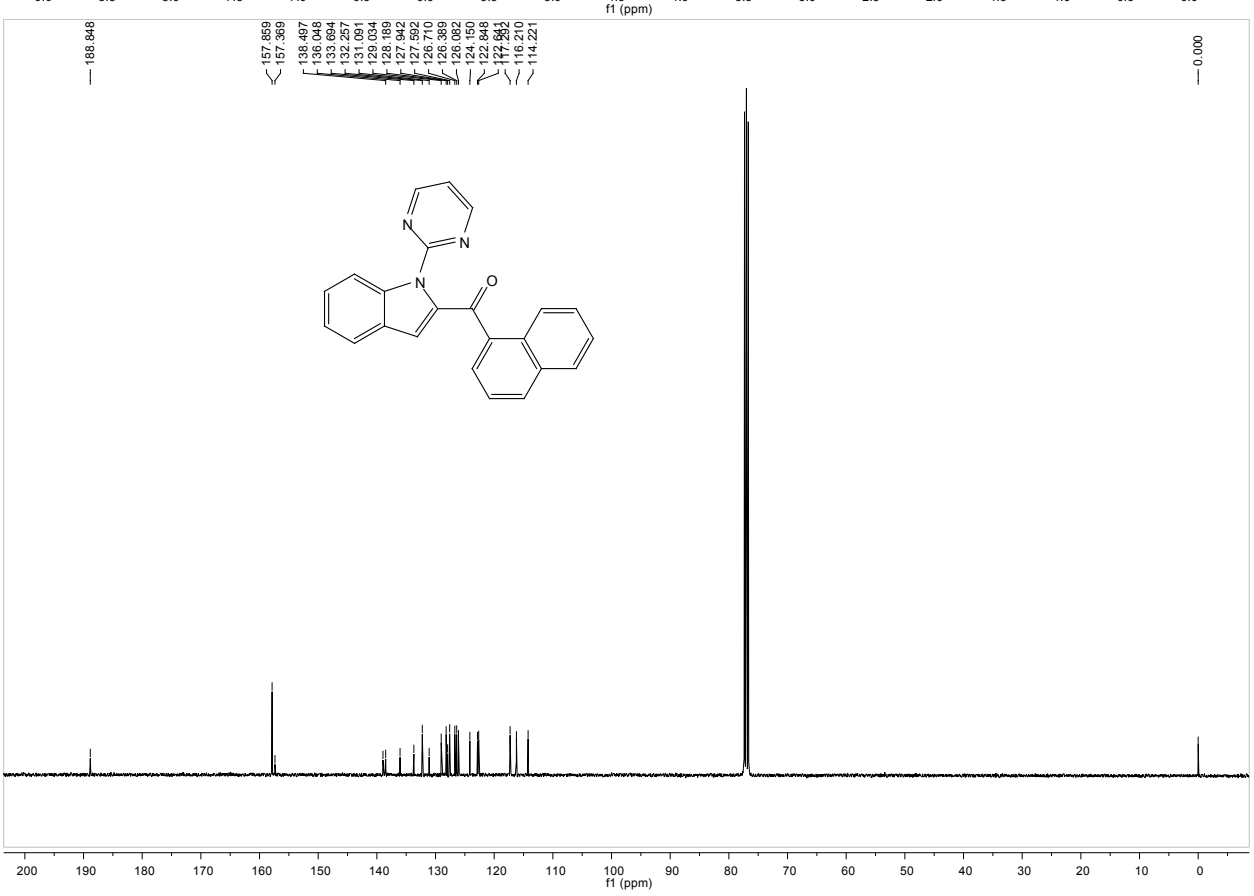
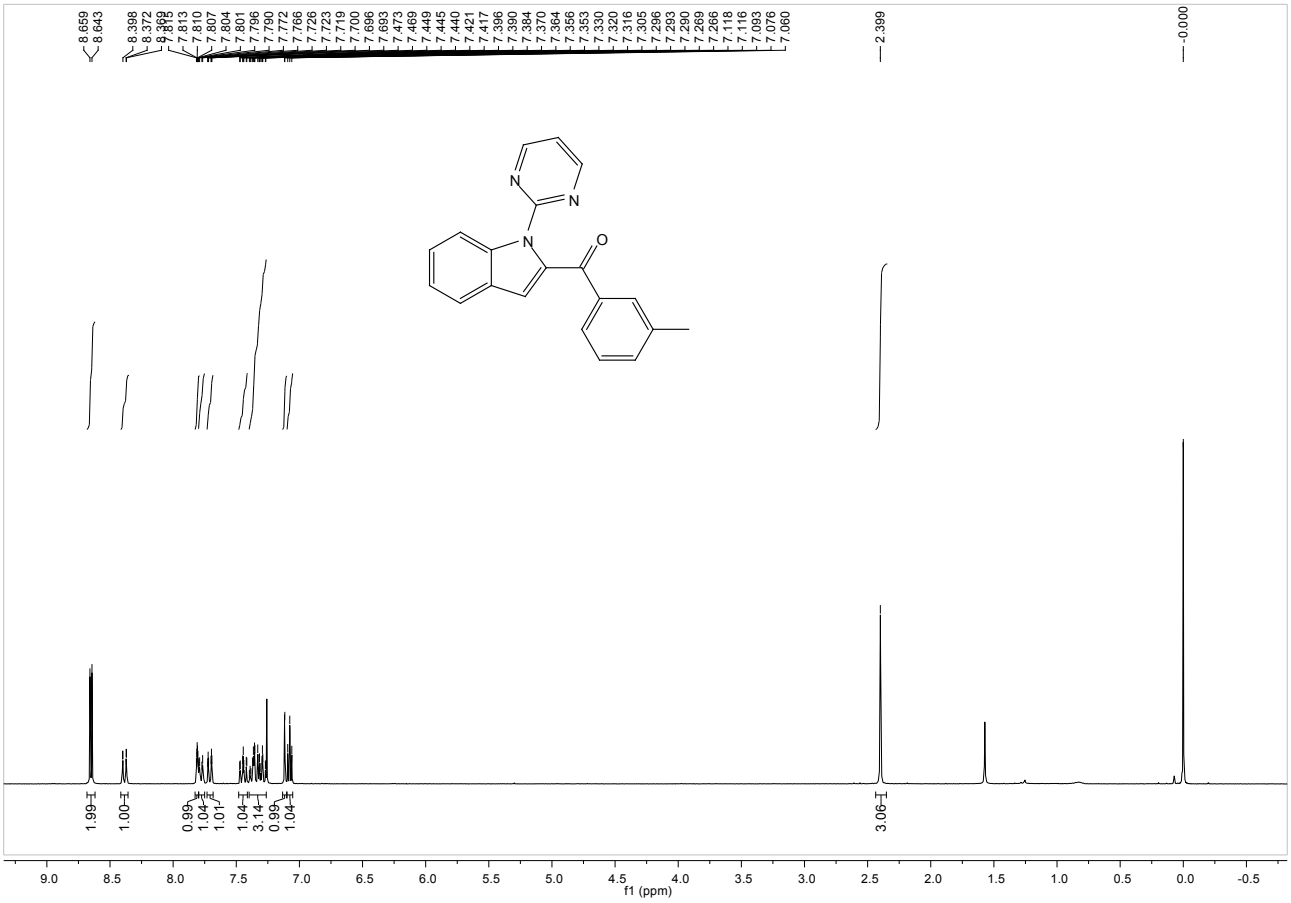
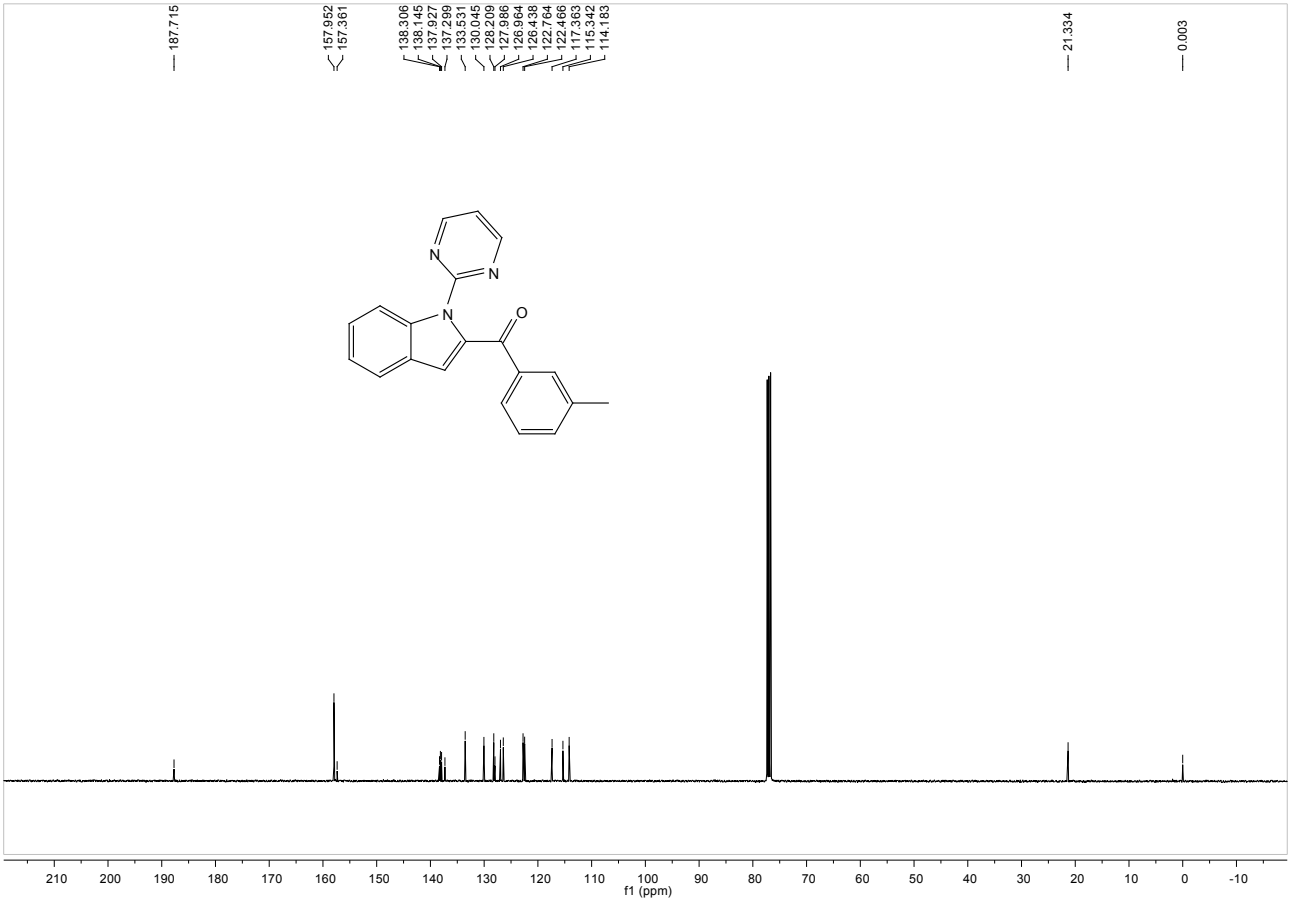
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

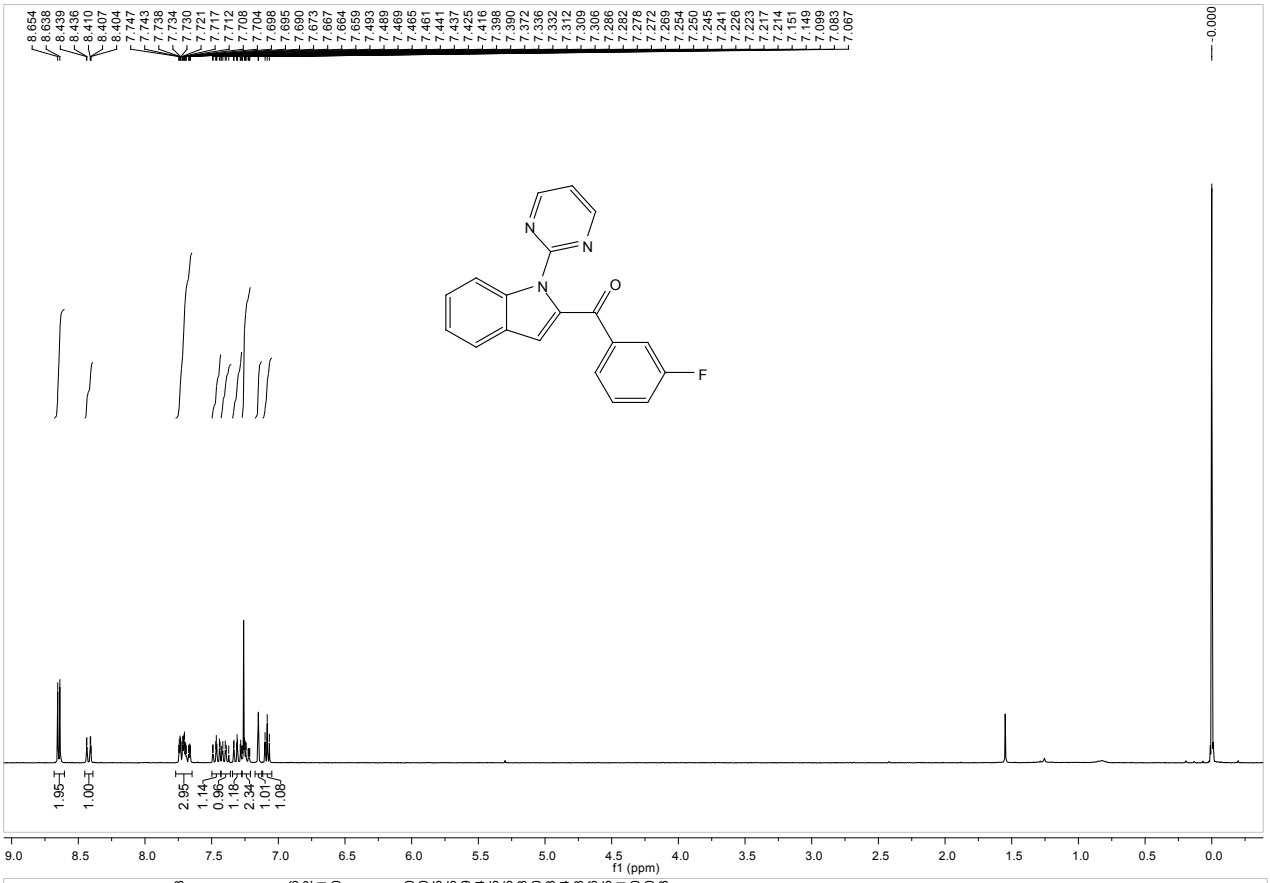

188.848
157.859
157.369
138.497
136.048
133.694
132.257
131.091
129.034
128.189
127.942
127.592
126.710
126.389
126.082
124.150
122.848
122.641
117.252
116.210
114.221
0.000
N
N
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

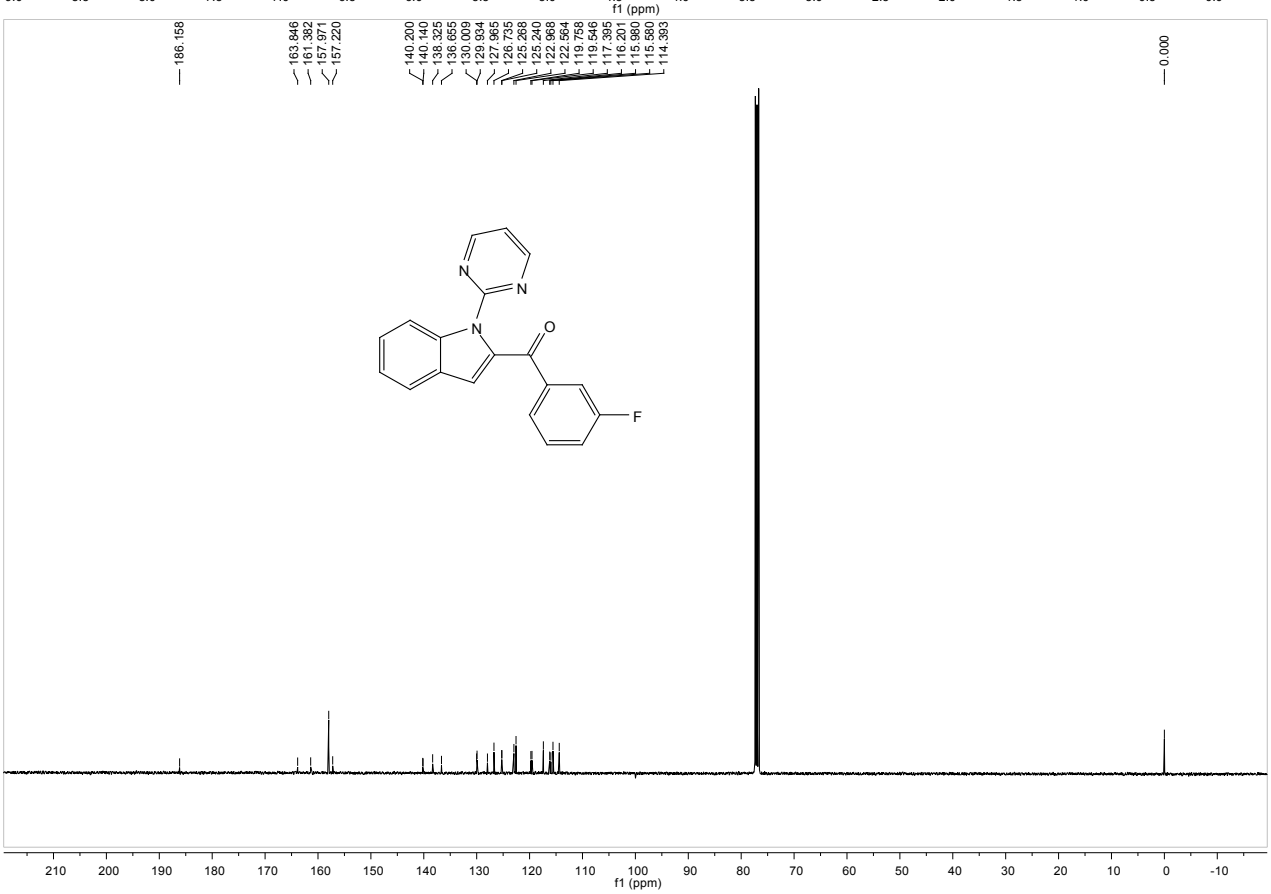
3k


8.659
8.643
8.398
8.372
8.369
7.815
7.813
7.810
7.807
7.804
7.801
7.796
7.790
7.772
7.766
7.726
7.723
7.719
7.700
7.696
7.693
7.473
7.469
7.449
7.445
7.440
7.421
7.417
7.396
7.390
7.384
7.370
7.364
7.356
7.353
7.330
7.320
7.316
7.305
7.296
7.293
7.290
7.269
7.266
7.118
7.116
7.093
7.076
7.060
2.399
-0.000
1.99
1.00
0.99
1.04
1.01
1.04
3.14
0.99
1.04
3.06
f1 (ppm)

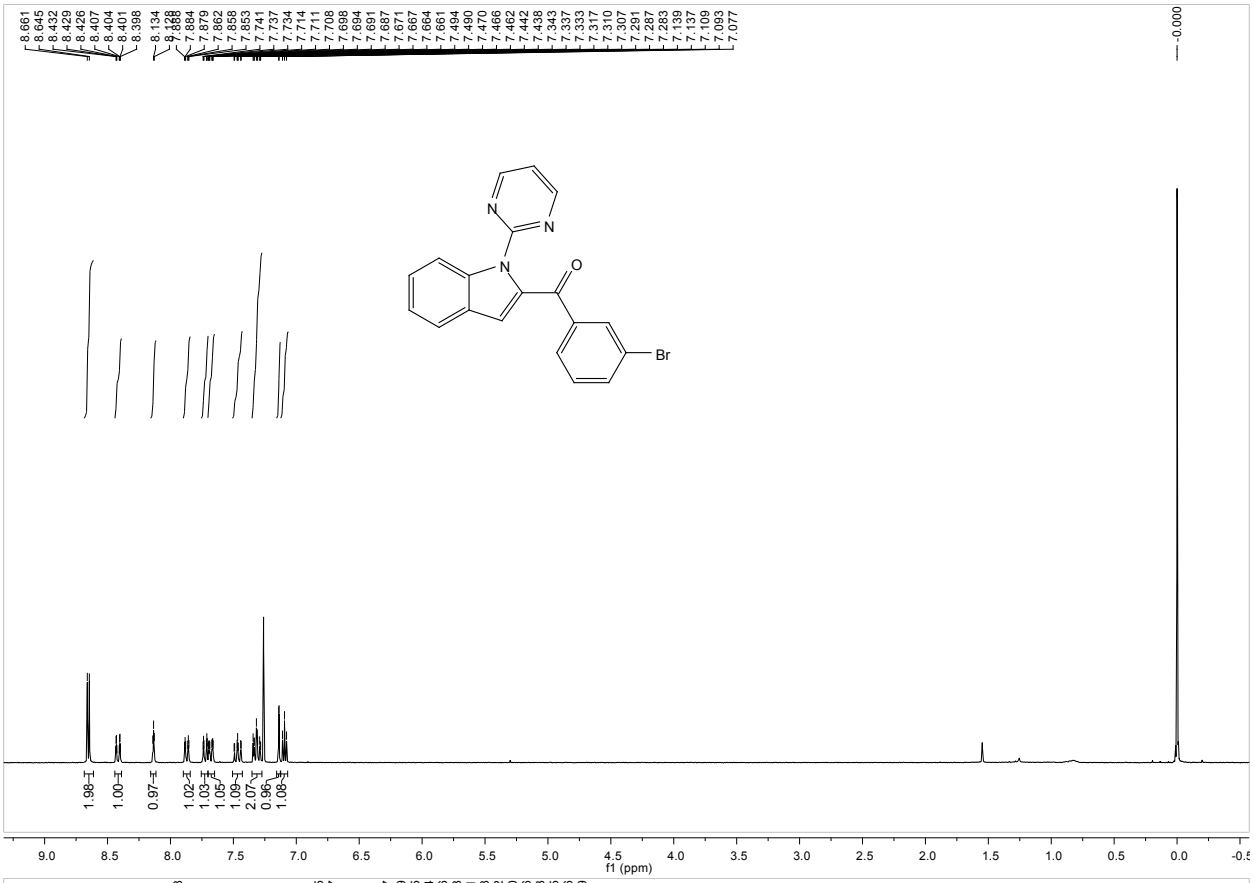

187.715
157.952
157.361
138.306
138.145
137.927
137.299
133.531
130.045
128.209
127.986
126.964
126.438
122.764
122.466
117.363
115.342
114.183
21.334
0.003
f1 (ppm)

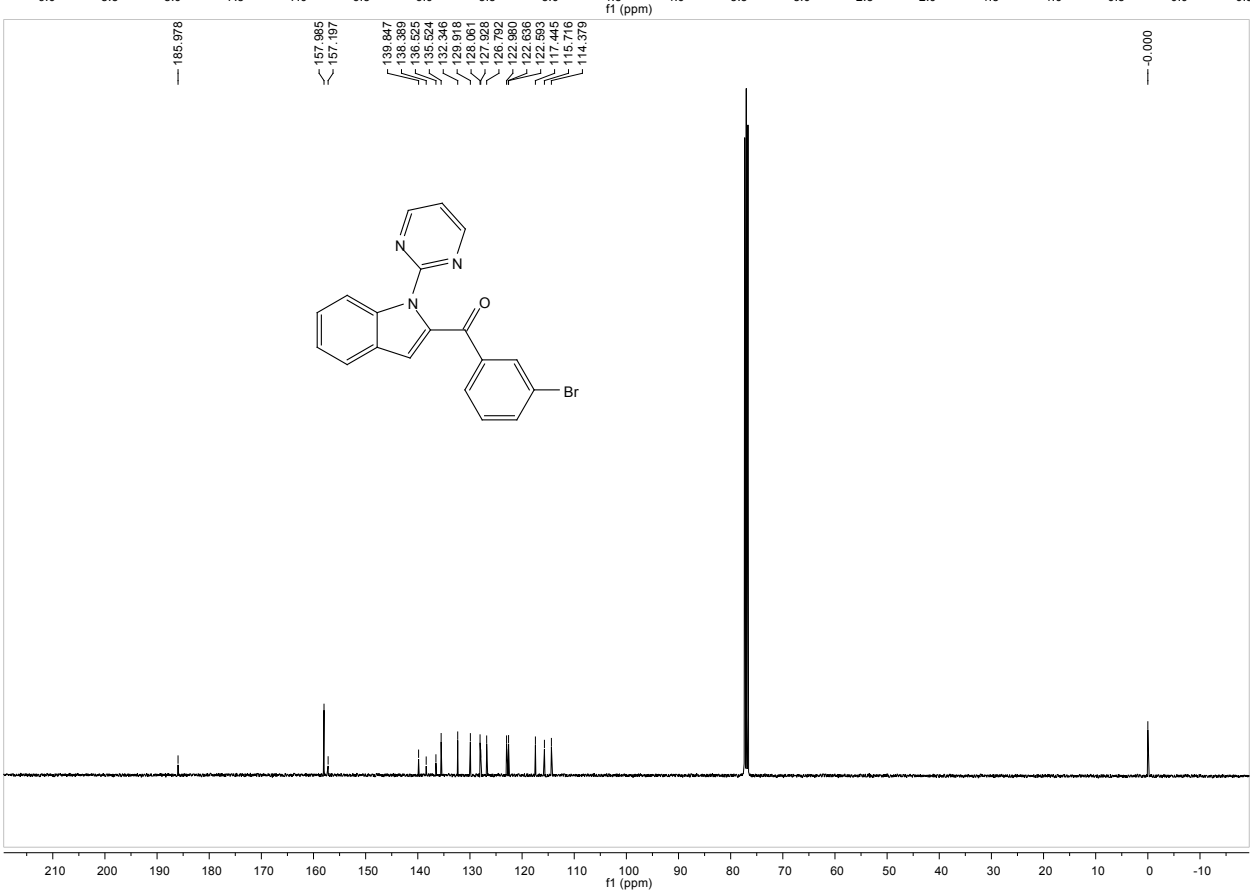
3l


8.654
8.638
8.439
8.436
8.410
8.407
8.404
7.747
7.743
7.738
7.734
7.730
7.721
7.717
7.712
7.708
7.704
7.698
7.695
7.690
7.673
7.667
7.664
7.659
7.493
7.489
7.469
7.465
7.461
7.441
7.437
7.425
7.416
7.398
7.390
7.372
7.336
7.332
7.312
7.309
7.306
7.286
7.282
7.278
7.272
7.269
7.254
7.250
7.245
7.241
7.226
7.223
7.217
7.214
7.151
7.149
7.099
7.083
7.067
0.000
1.95
1.00
2.95
1.14
0.96
1.18
2.34
1.01
1.08
N
N
N
O
F
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
1.5
1.0
0.5
0.0
f1 (ppm)

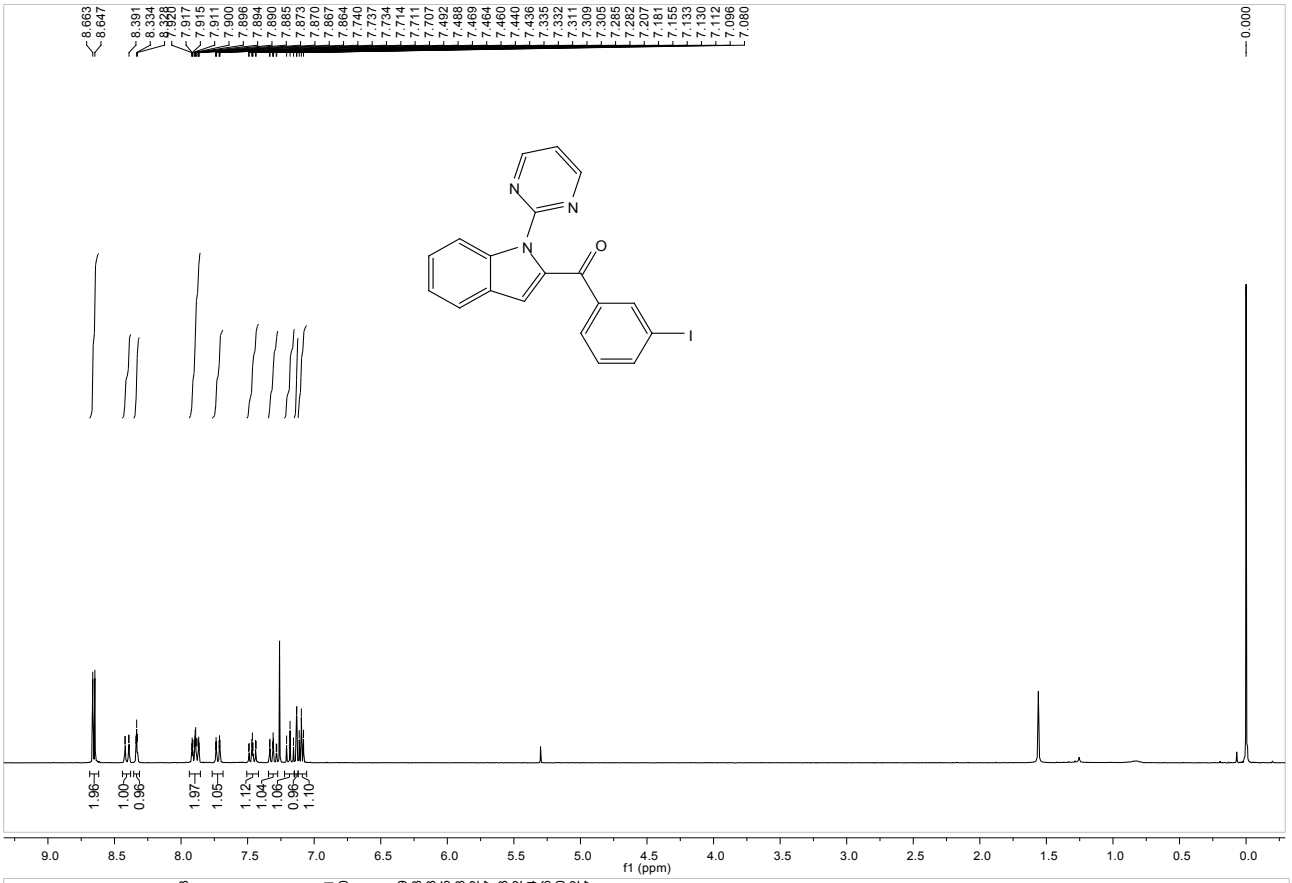

186.158
163.846
161.382
157.971
157.220
140.200
140.140
138.325
136.655
130.009
129.934
127.965
126.735
125.268
125.240
122.968
122.564
119.758
119.546
117.395
116.201
115.980
115.580
114.393
0.000
N
N
N
O
F
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

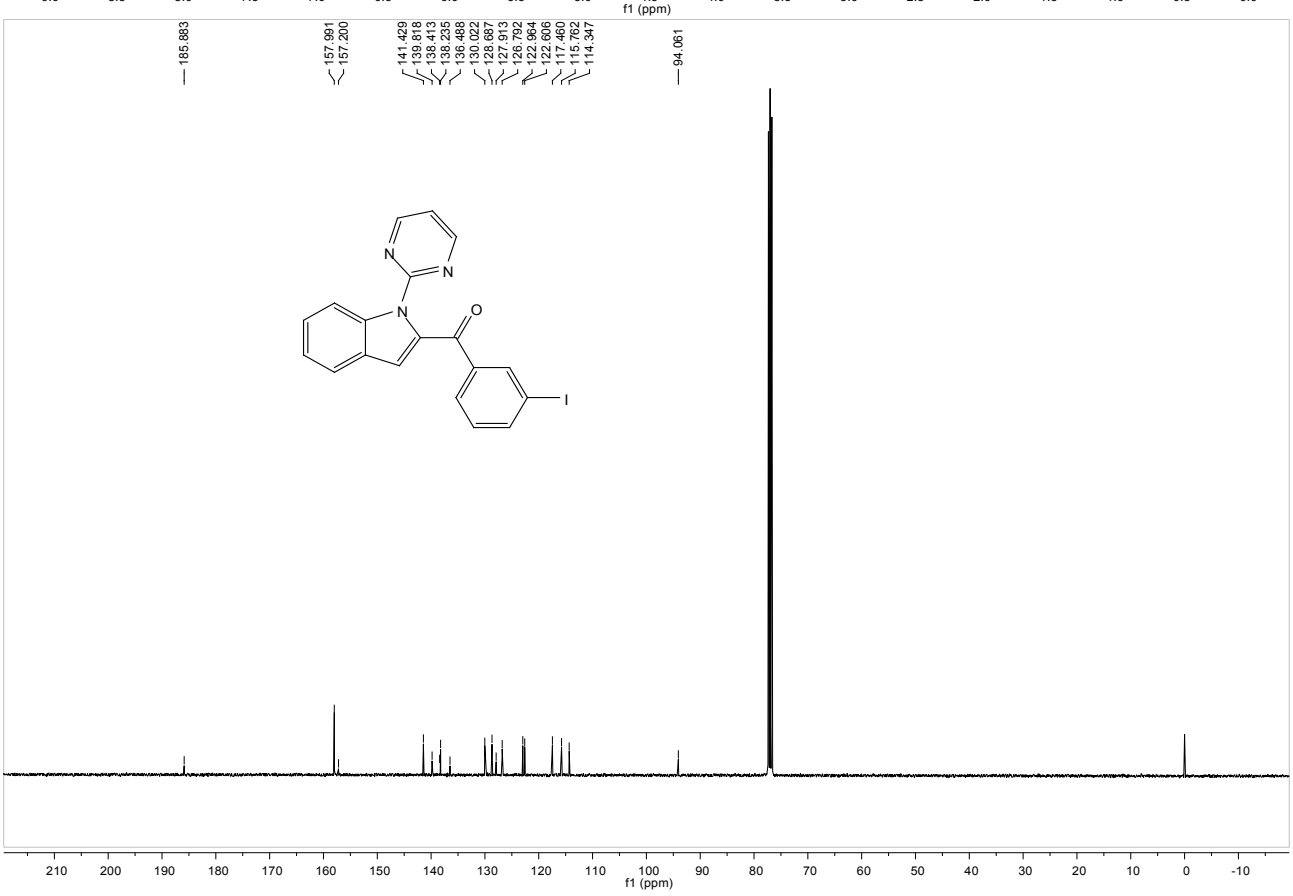
3m


8.661
8.645
8.432
8.429
8.426
8.407
8.404
8.401
8.398
8.134
7.884
7.879
7.862
7.858
7.853
7.741
7.737
7.734
7.714
7.711
7.708
7.698
7.694
7.691
7.687
7.671
7.667
7.664
7.661
7.494
7.490
7.470
7.466
7.462
7.442
7.438
7.343
7.337
7.333
7.317
7.310
7.307
7.291
7.287
7.283
7.139
7.137
7.109
7.093
7.077
-0.000
N
N
N
O
Br
1.98
1.00
0.97
1.02
1.03
1.05
1.09
2.07
0.96
1.08
f1 (ppm)

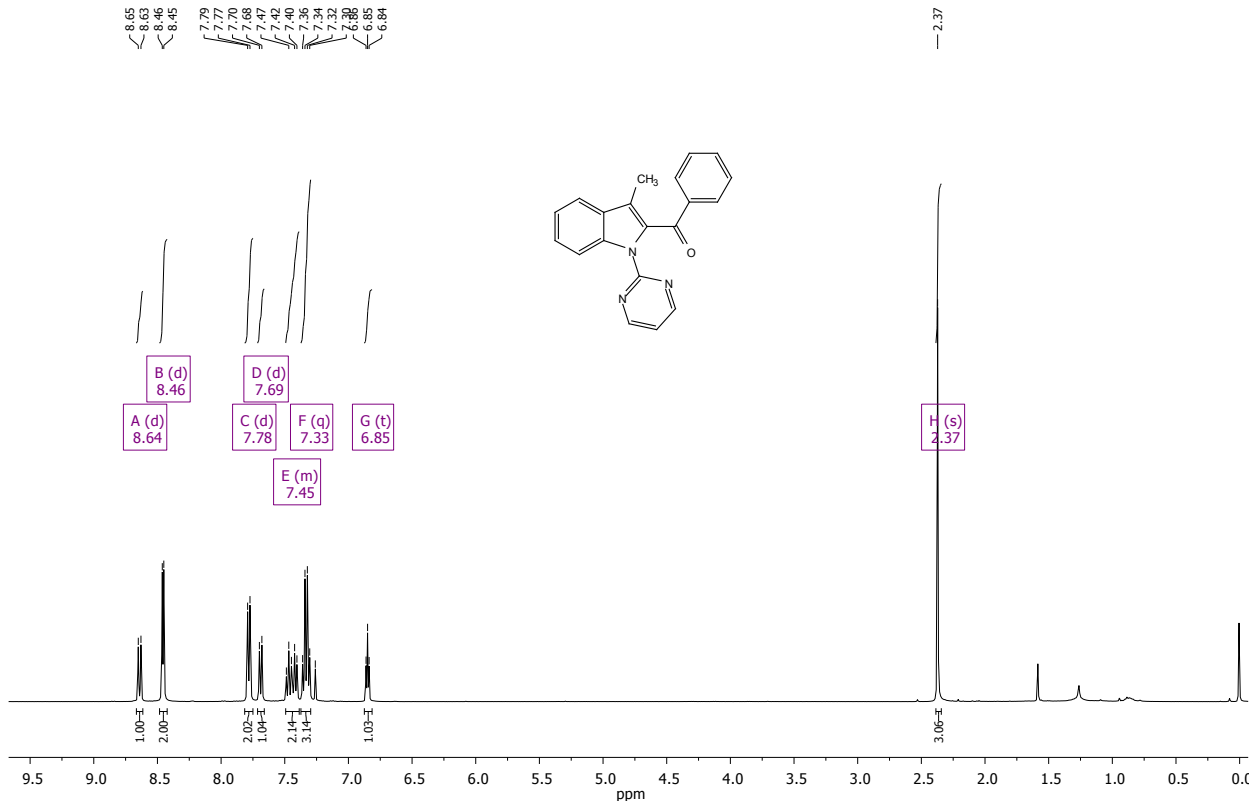

185.978
157.985
157.197
139.847
138.389
136.525
135.524
132.346
129.918
128.061
127.928
126.792
122.980
122.636
122.593
117.445
115.716
114.379
-0.000
N
N
N
O
Br
f1 (ppm)

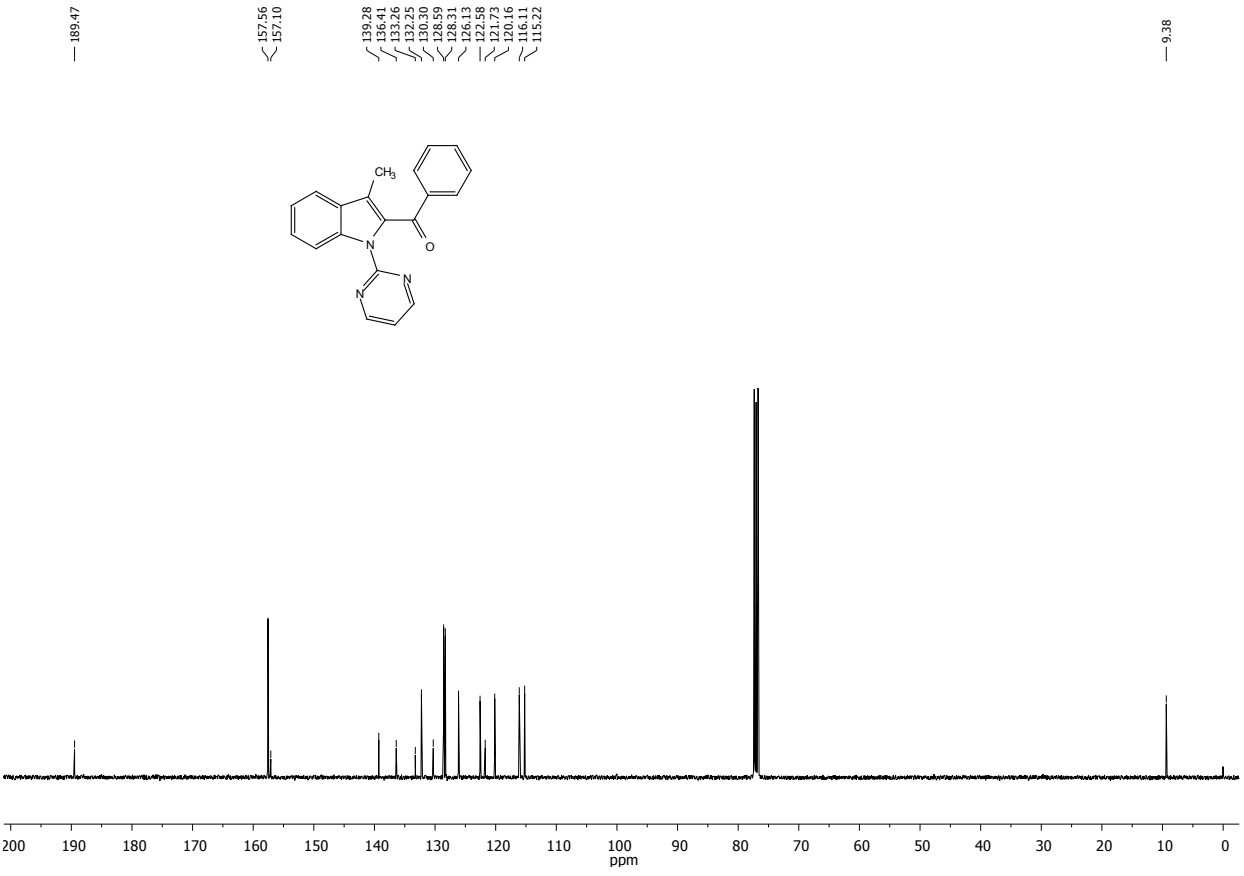
3n


8.663
8.647
8.391
8.334
7.917
7.915
7.911
7.900
7.896
7.894
7.890
7.885
7.873
7.870
7.867
7.864
7.740
7.737
7.734
7.714
7.711
7.707
7.492
7.488
7.469
7.464
7.460
7.440
7.436
7.335
7.332
7.311
7.309
7.305
7.285
7.282
7.207
7.181
7.155
7.133
7.130
7.112
7.096
7.080
0.000
1.96
1.00
0.96
1.97
1.05
1.12
1.04
1.06
0.96
1.10
f1 (ppm)

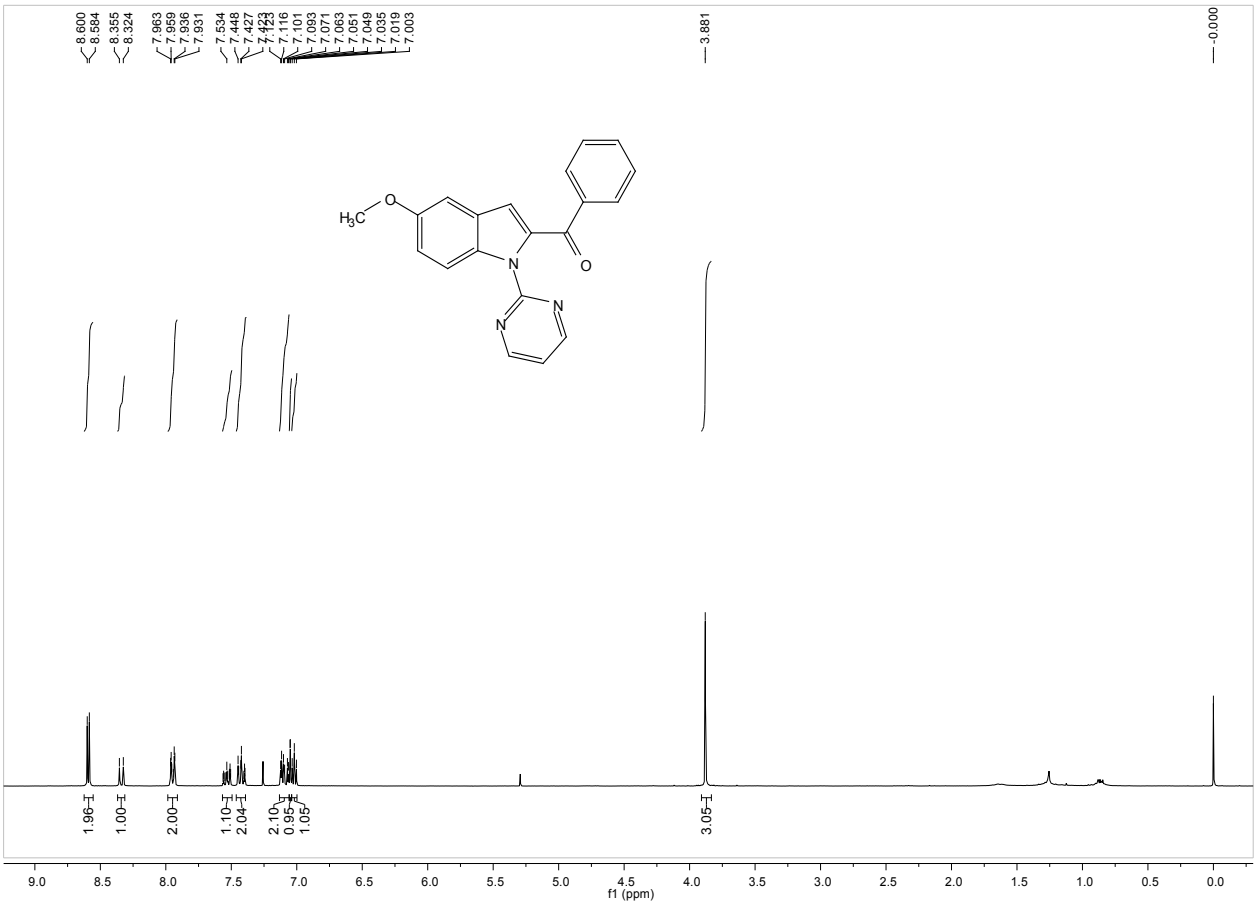

185.883
157.991
157.200
141.429
139.818
138.413
138.235
136.488
130.022
128.687
127.913
126.792
122.964
122.606
117.460
115.762
114.347
94.061
f1 (ppm)

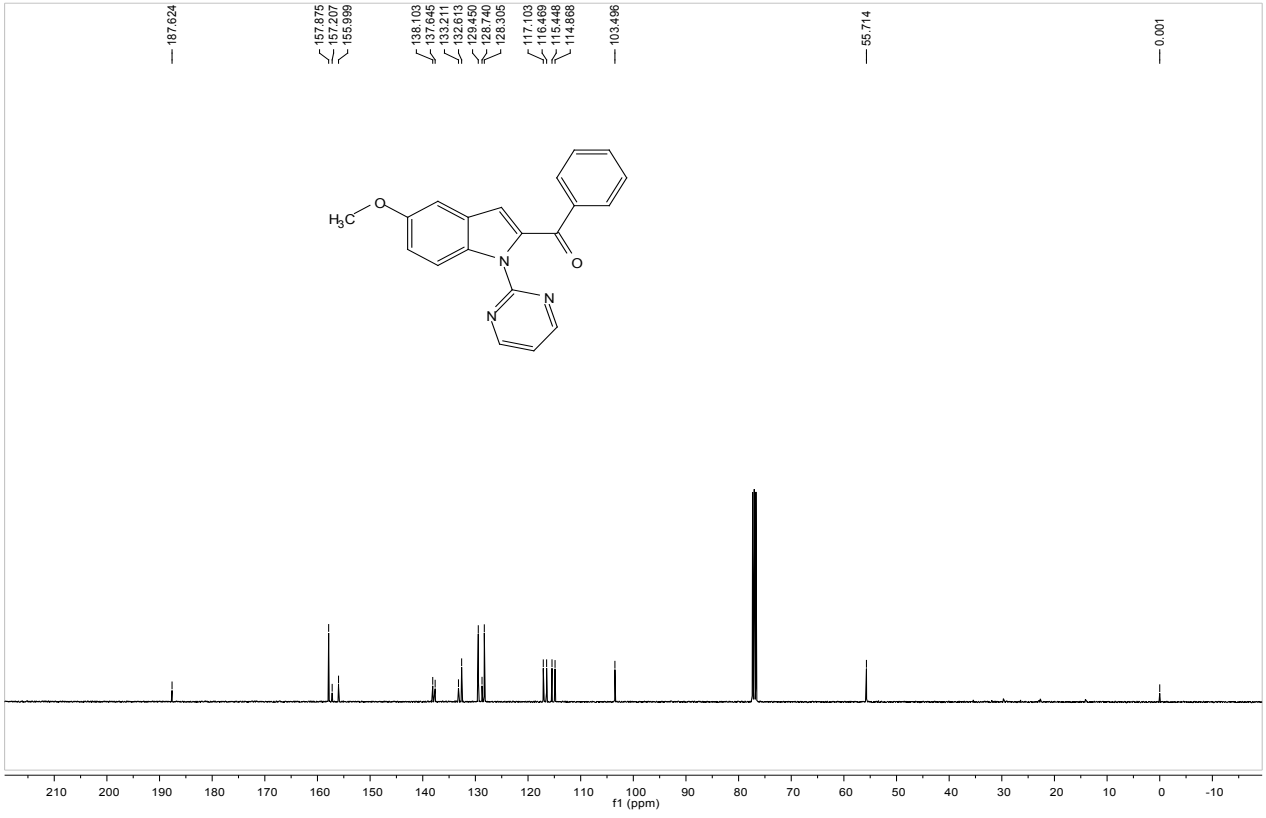
3o


8.65
8.63
8.46
8.45
7.79
7.77
7.70
7.68
7.47
7.42
7.40
7.36
7.34
7.32
6.85
6.84
2.37
CH3
O
N
N
N
B (d)
8.46
D (d)
7.69
A (d)
8.64
C (d)
7.78
F (q)
7.33
G (t)
6.85
E (m)
7.45
H (s)
2.37
1.00
2.00
2.02
1.04
2.14
3.14
1.03
3.06
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
1.5
1.0
0.5
ppm

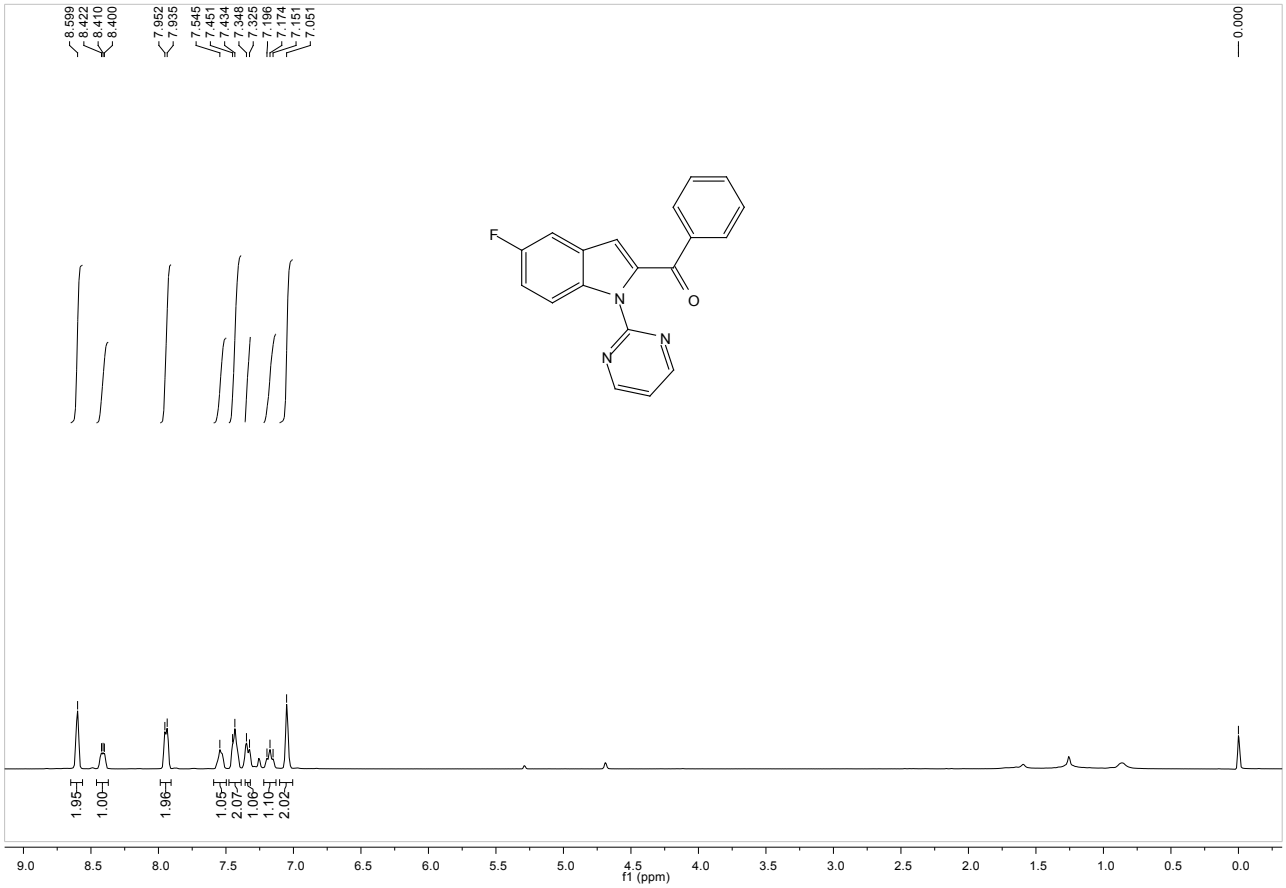

189.47
157.56
157.10
139.28
136.41
133.26
132.25
130.30
128.59
128.31
126.13
122.58
121.73
120.16
116.11
115.22
9.38
CH3
O
N
N
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

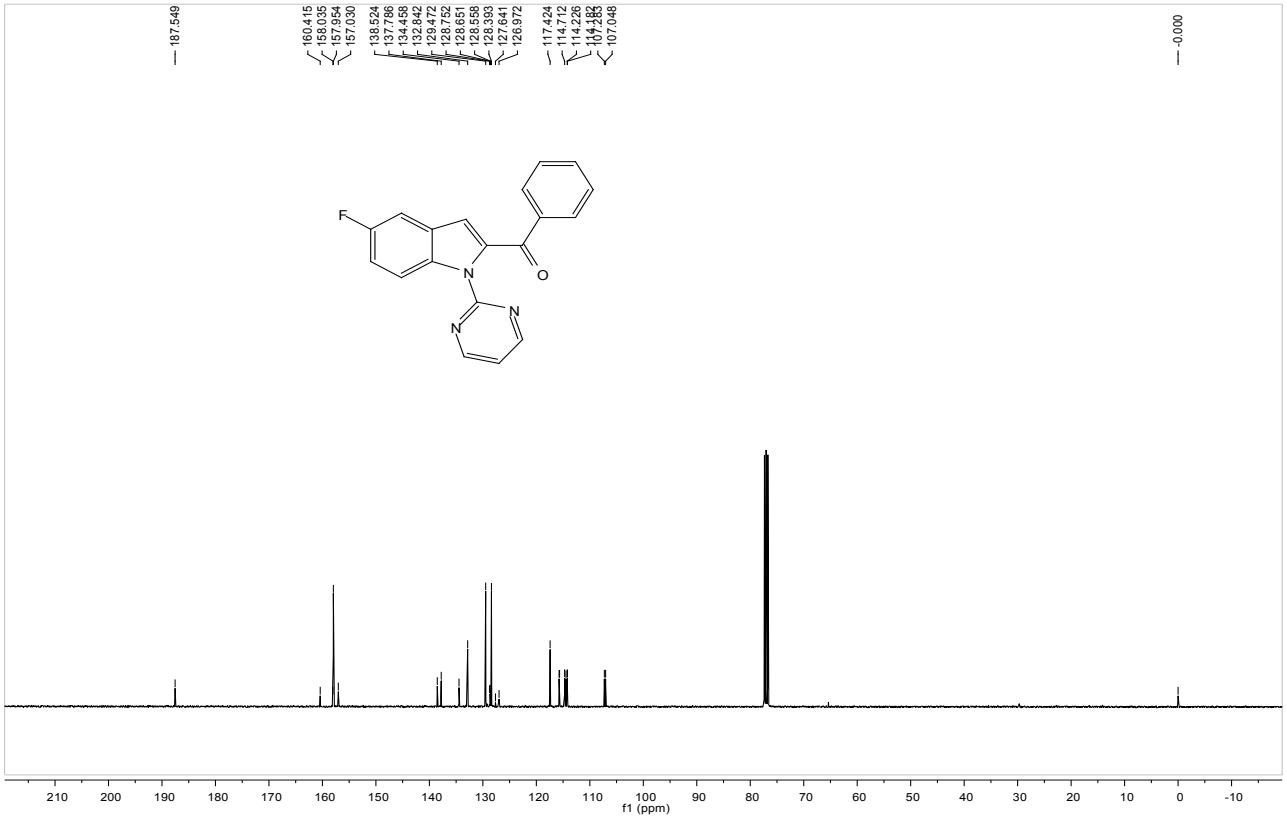
3p


8.600
8.584
8.355
8.324
7.963
7.959
7.936
7.931
7.534
7.448
7.427
7.116
7.101
7.093
7.071
7.063
7.051
7.049
7.035
7.019
7.003
3.881
-0.000
1.96
1.00
2.00
1.10
2.04
2.10
0.95
1.05
3.05
f1 (ppm)

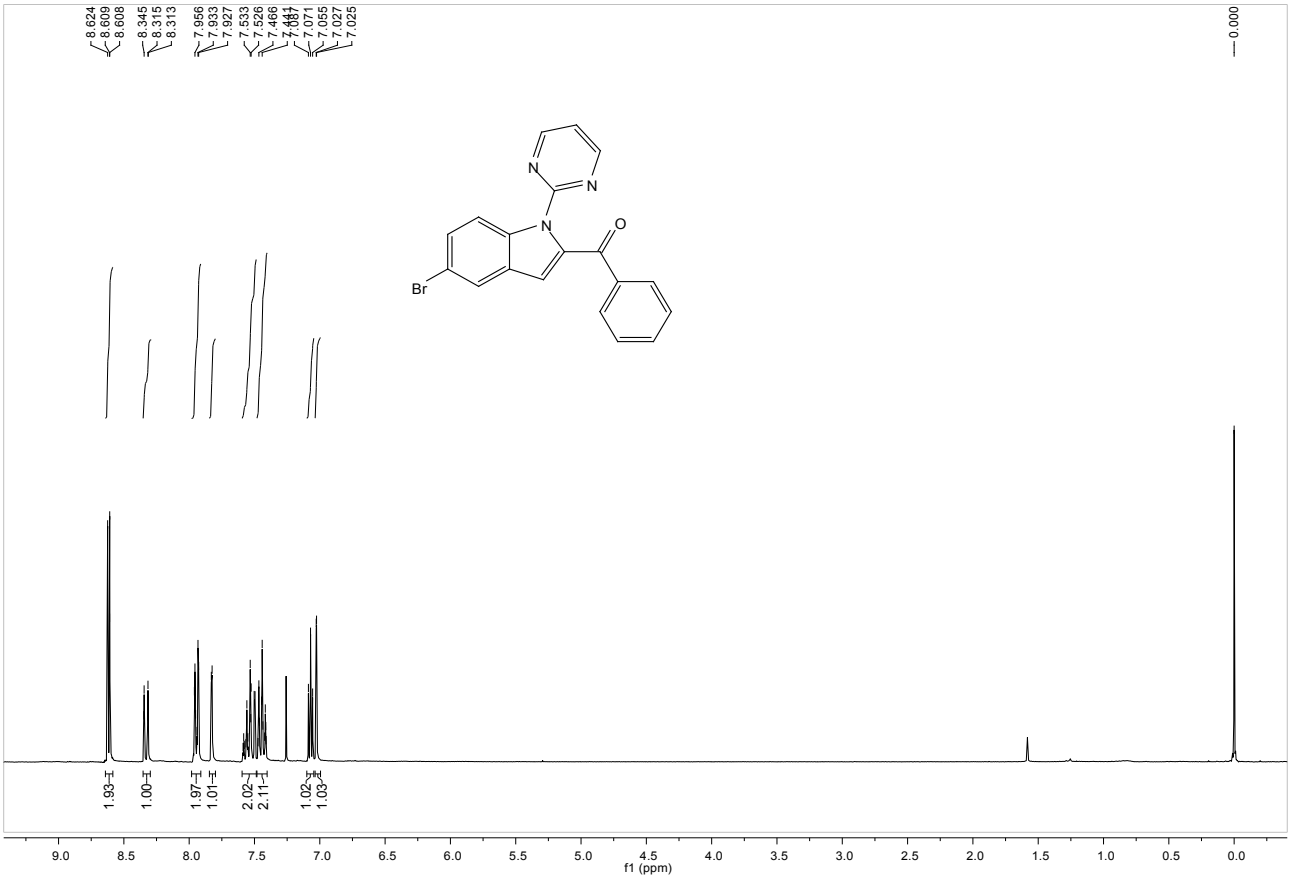

187.624
157.875
157.207
155.999
138.103
137.645
133.211
132.613
129.450
128.740
128.305
117.103
116.469
115.448
114.868
103.496
55.714
0.001
f1 (ppm)

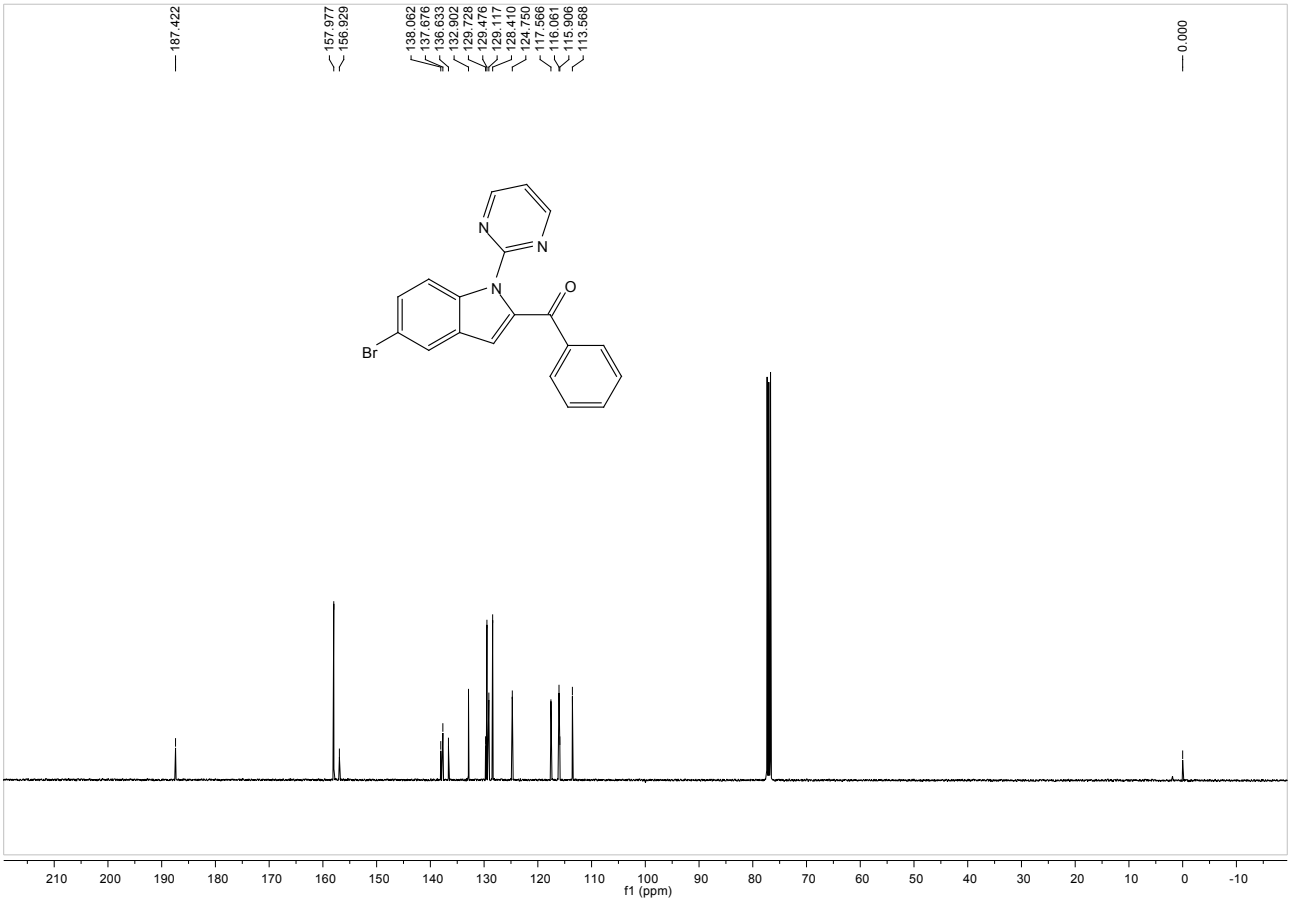
3q


8.599
8.422
8.410
8.400
7.952
7.935
7.545
7.451
7.434
7.348
7.325
7.196
7.174
7.151
7.051
0.000
F
N
O
N
N
1.95
1.00
1.96
1.05
2.07
1.06
1.10
2.02
f1 (ppm)

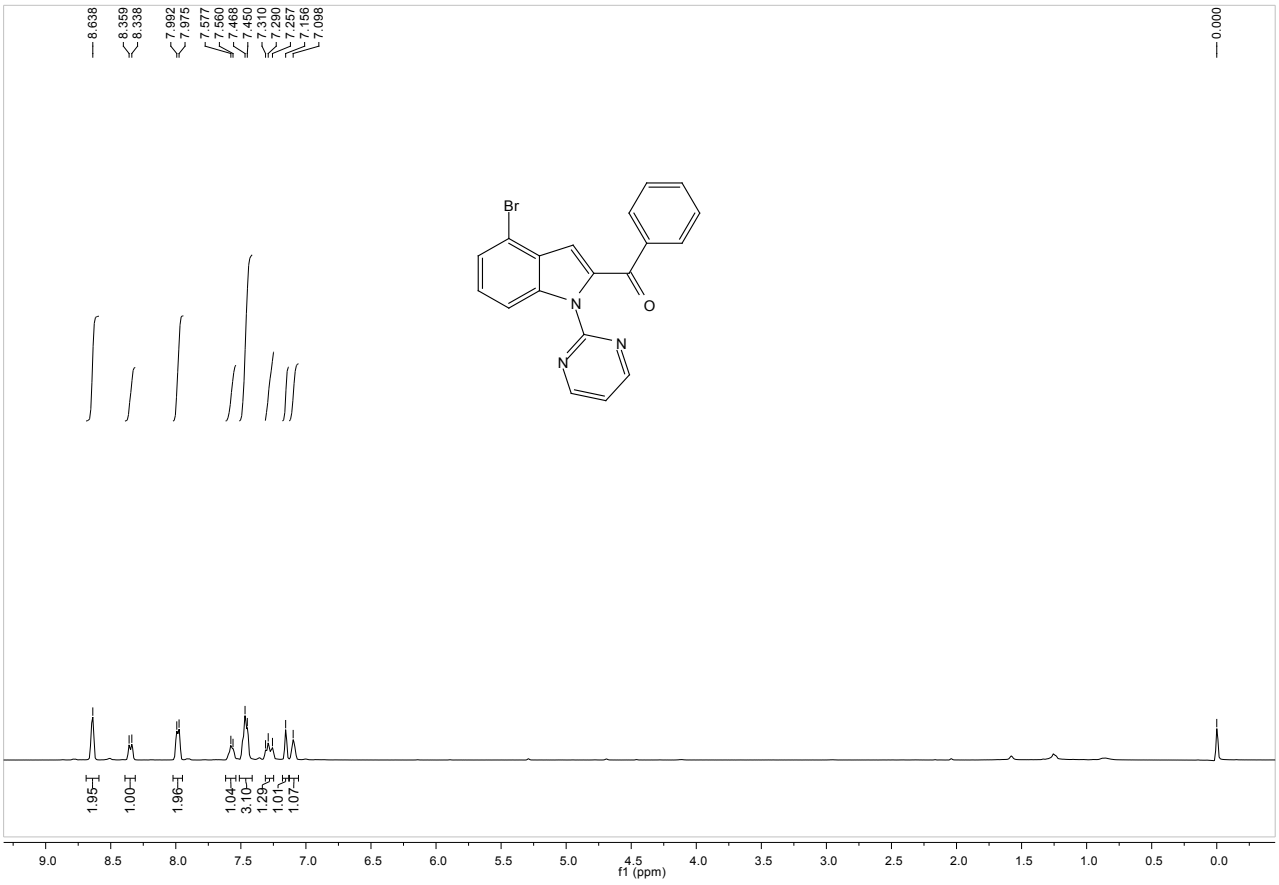

187.549
160.415
158.035
157.954
157.030
138.524
137.786
134.458
132.842
129.472
128.752
128.651
128.558
128.393
127.641
126.972
117.424
114.712
114.226
107.283
107.048
-0.000
F
N
O
N
N
f1 (ppm)

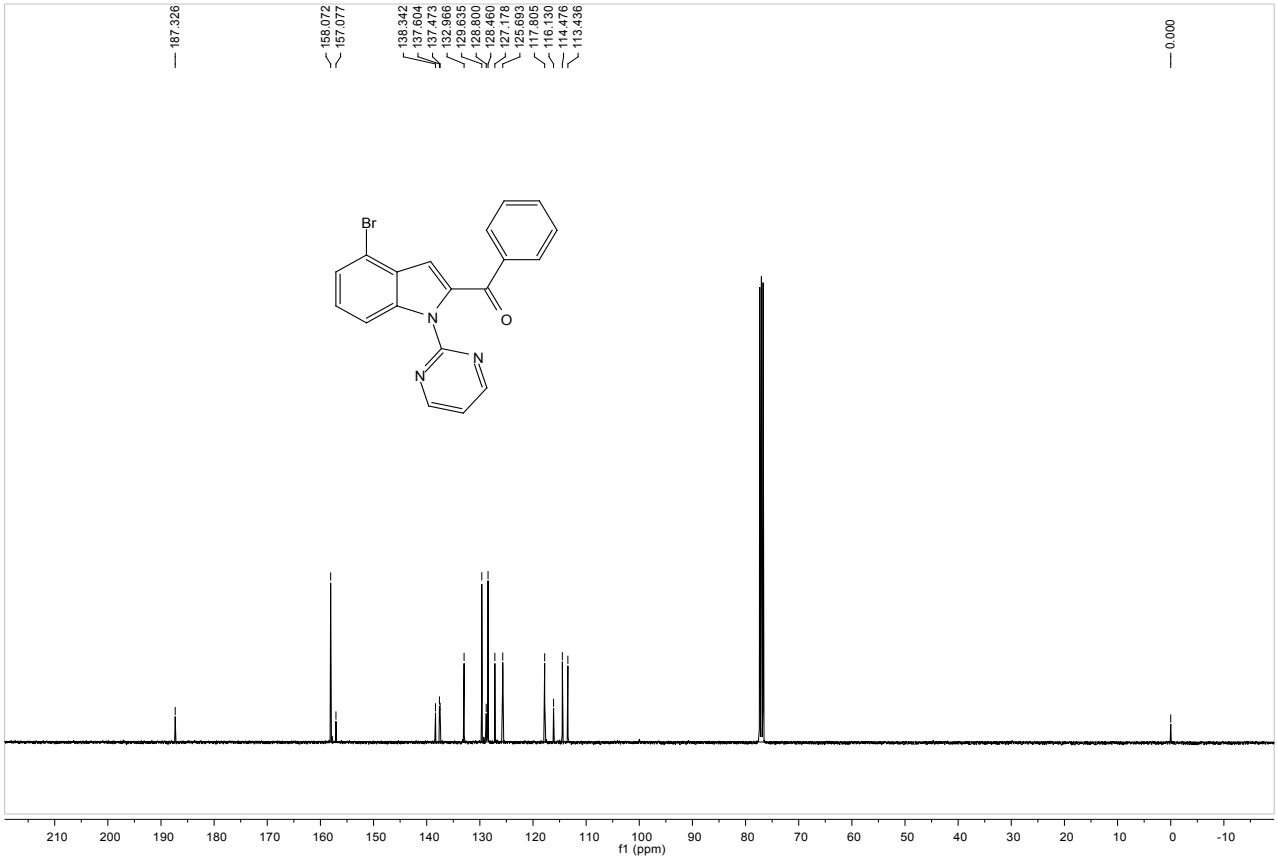
3r


8.624
8.609
8.608
8.345
8.315
8.313
7.956
7.933
7.927
7.533
7.526
7.466
7.441
7.087
7.071
7.055
7.027
7.025
0.000
1.93
1.00
1.97
1.01
2.02
2.11
1.02
1.03
N
N
N
O
Br
f1 (ppm)

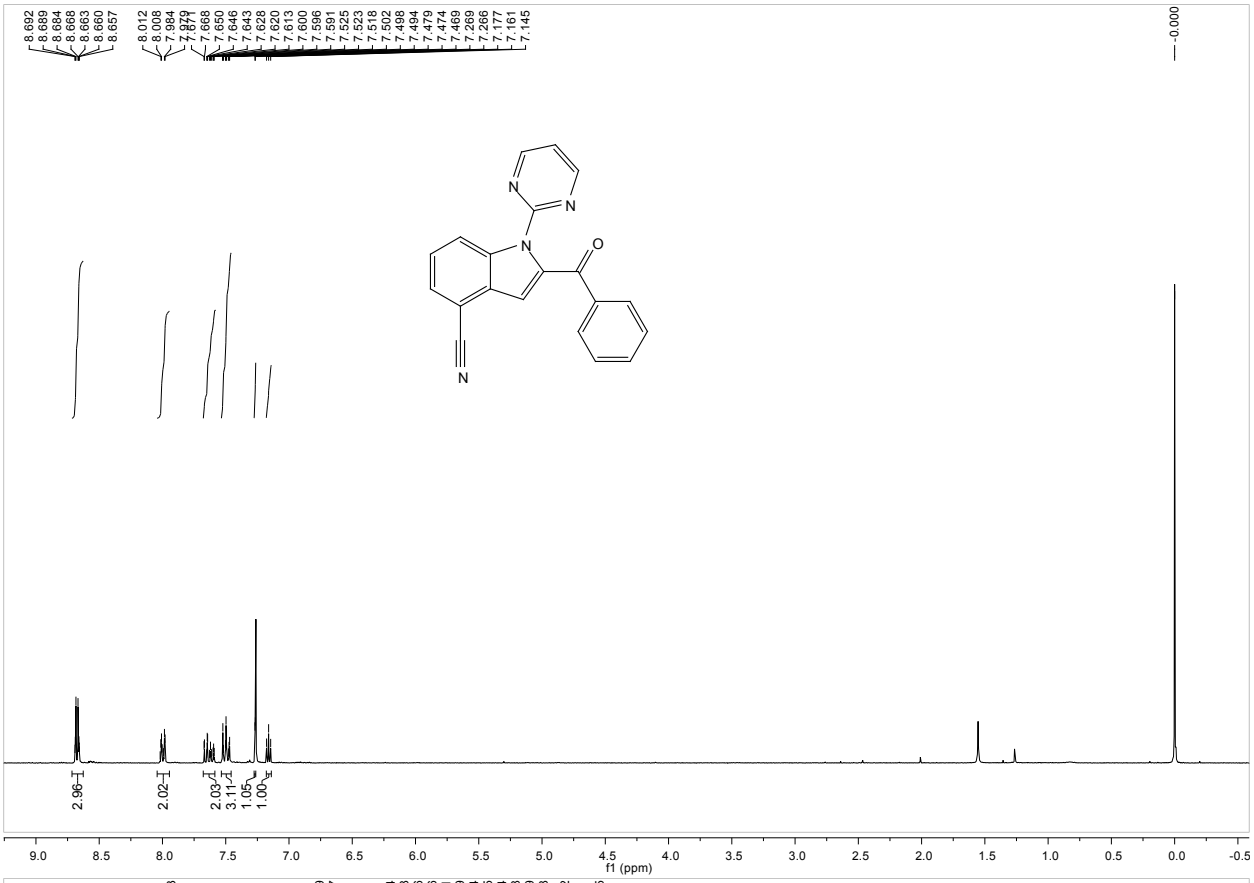

187.422
157.977
156.929
138.062
137.676
136.633
132.902
129.728
129.476
129.117
128.410
124.750
117.566
116.061
115.906
113.568
0.000
N
N
N
O
Br
f1 (ppm)

3s


8.638
8.359
8.338
7.992
7.975
7.577
7.560
7.468
7.450
7.310
7.290
7.257
7.156
7.098
0.000
Br
O
N
N
N
1.95
1.00
1.96
1.04
3.10
1.29
1.01
1.07
f1 (ppm)


187.326
158.072
157.077
138.342
137.604
137.473
132.966
129.635
128.800
128.460
127.178
125.693
117.805
116.130
114.476
113.436
0.000
Br
O
N
N
N
f1 (ppm)

3t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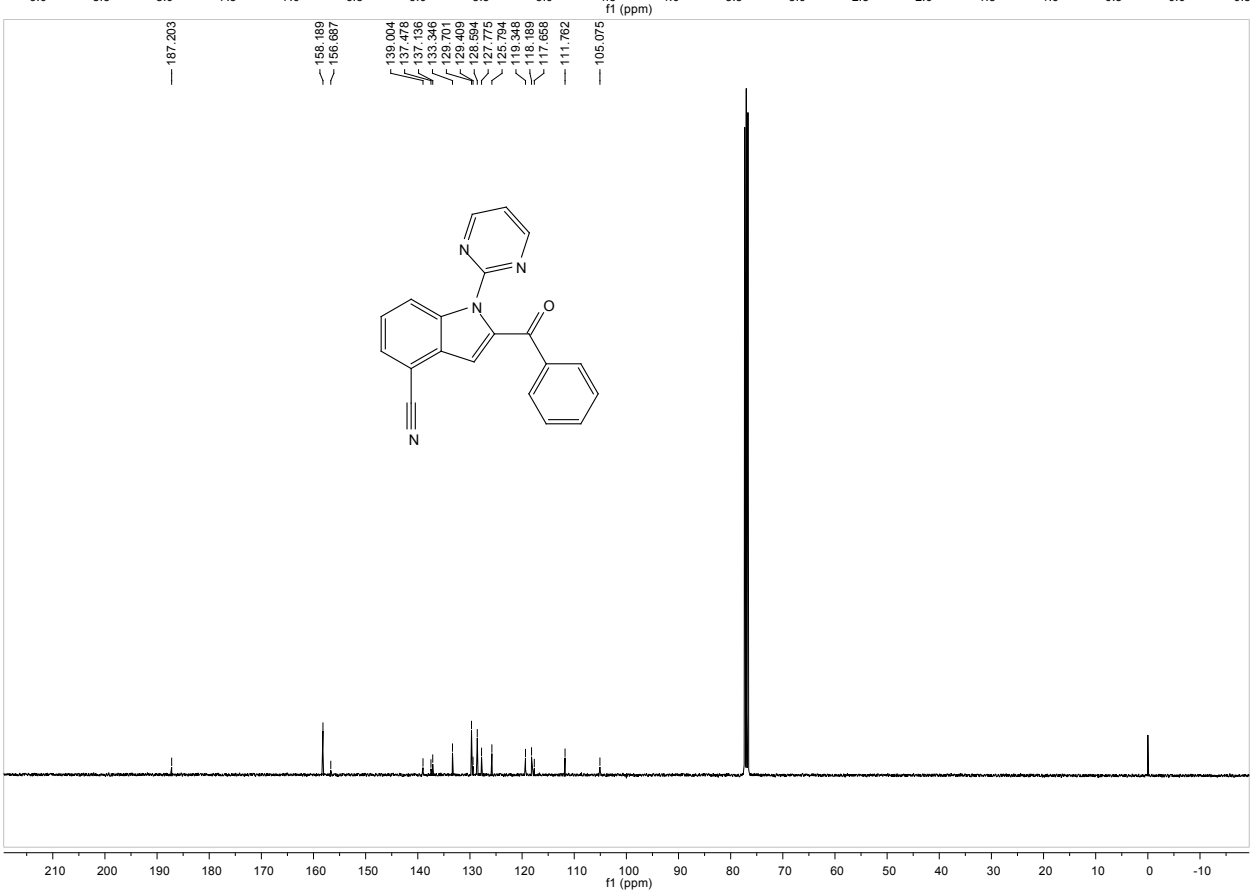
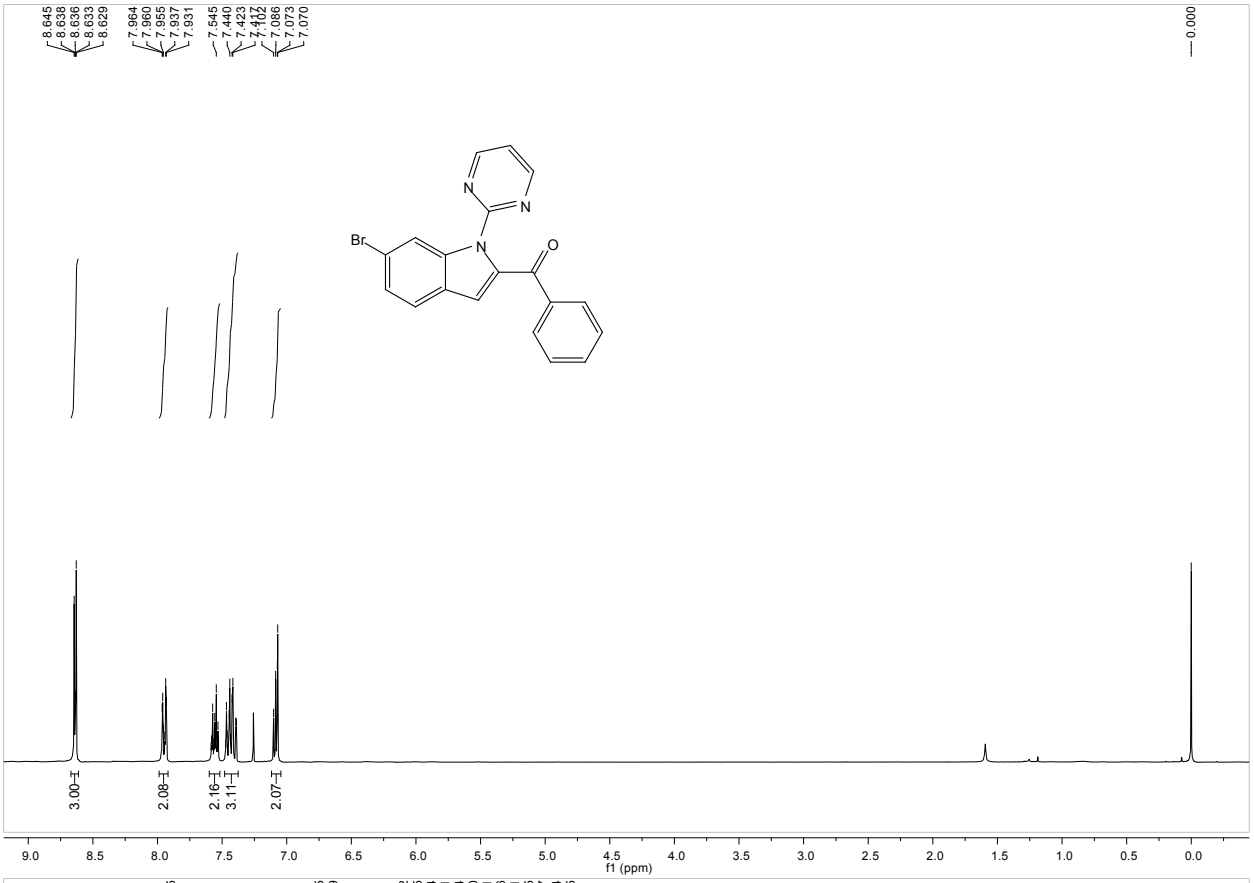
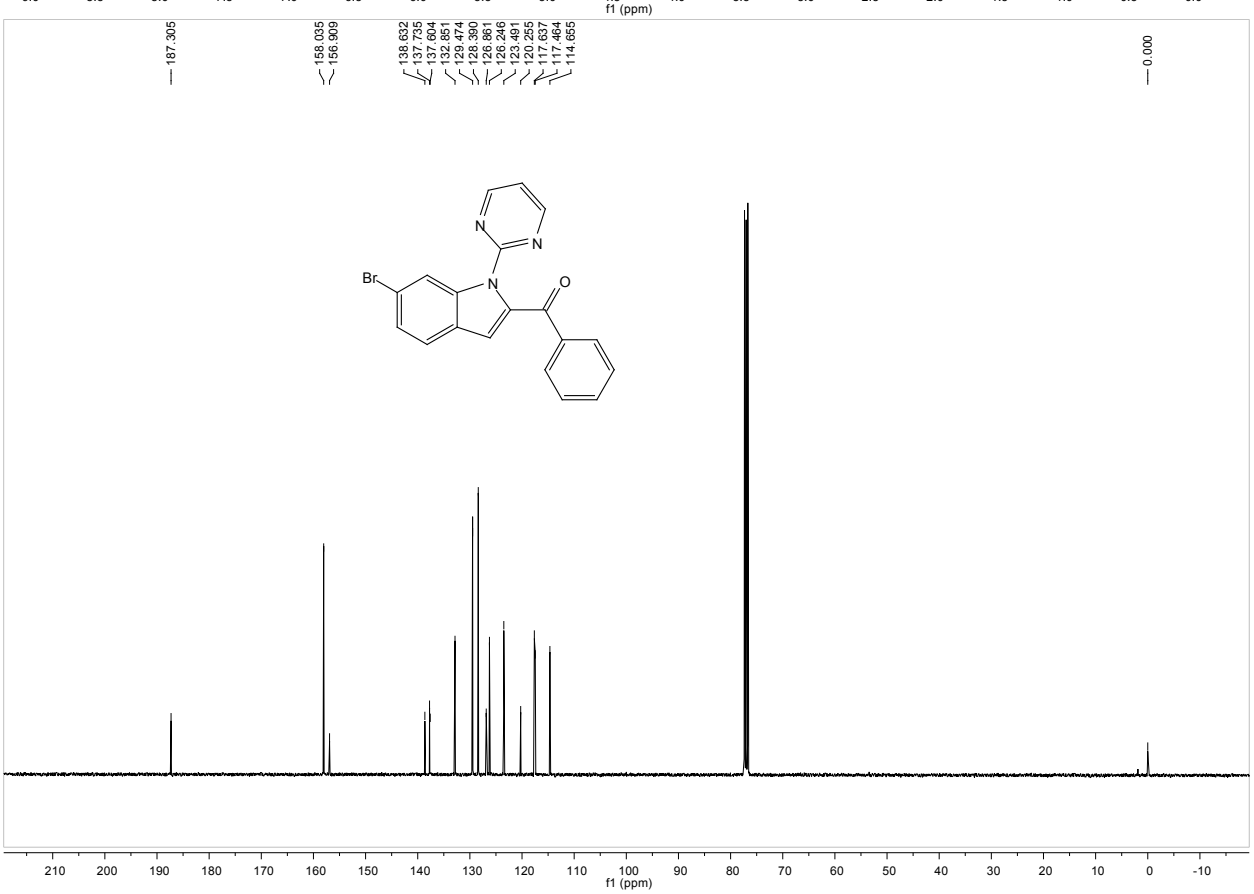
8.689
8.684
8.668
8.663
8.660
8.657
8.012
8.008
7.984
7.671
7.668
7.650
7.646
7.643
7.628
7.620
7.613
7.600
7.596
7.591
7.525
7.523
7.518
7.502
7.498
7.494
7.479
7.474
7.469
7.269
7.266
7.177
7.161
7.145
-0.000
2.96
2.02
2.03
3.11
1.05
1.00
f1 (ppm)

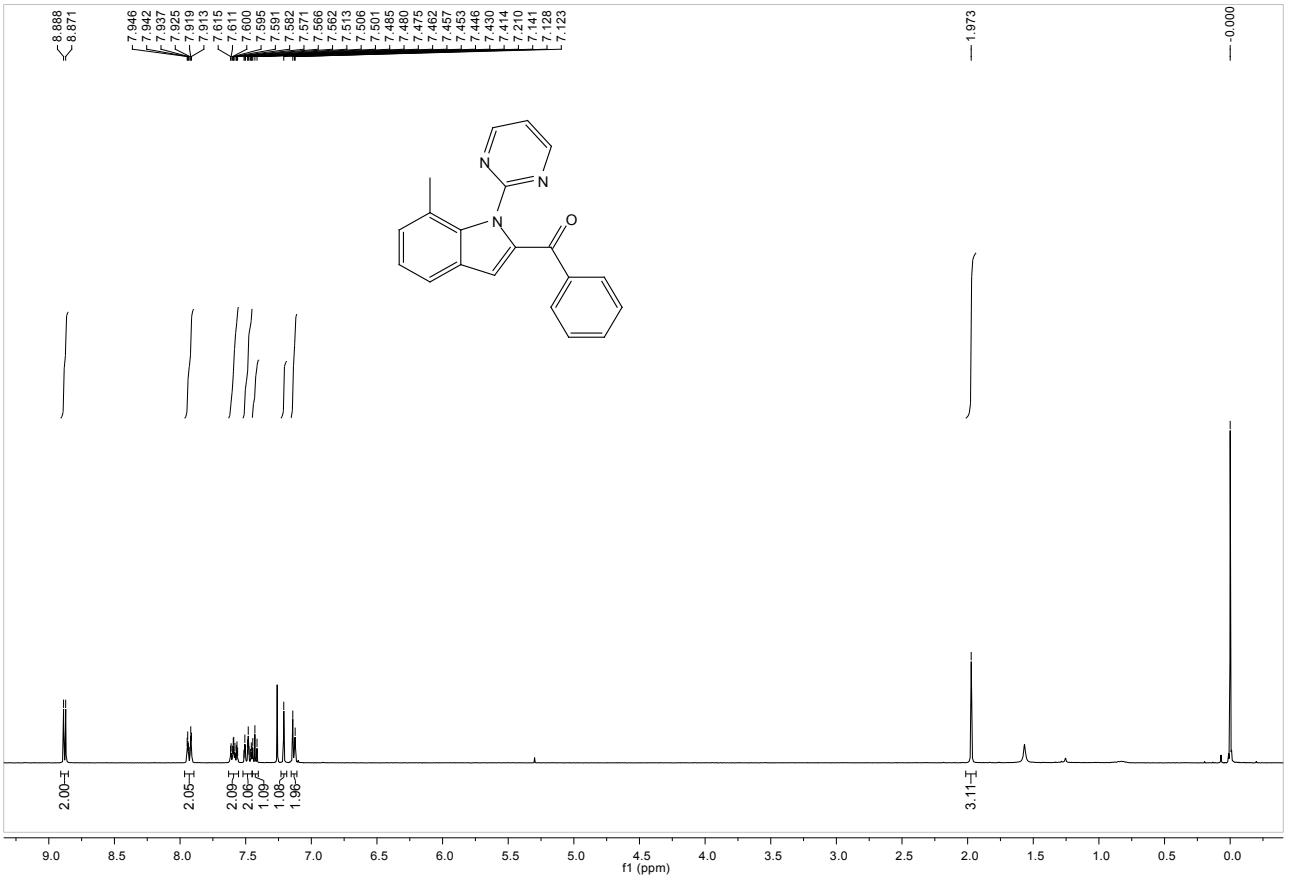

187.203
158.189
156.687
139.004
137.478
137.136
133.346
129.701
129.409
128.594
127.775
125.794
119.348
118.189
117.658
111.762
105.075
f1 (ppm)

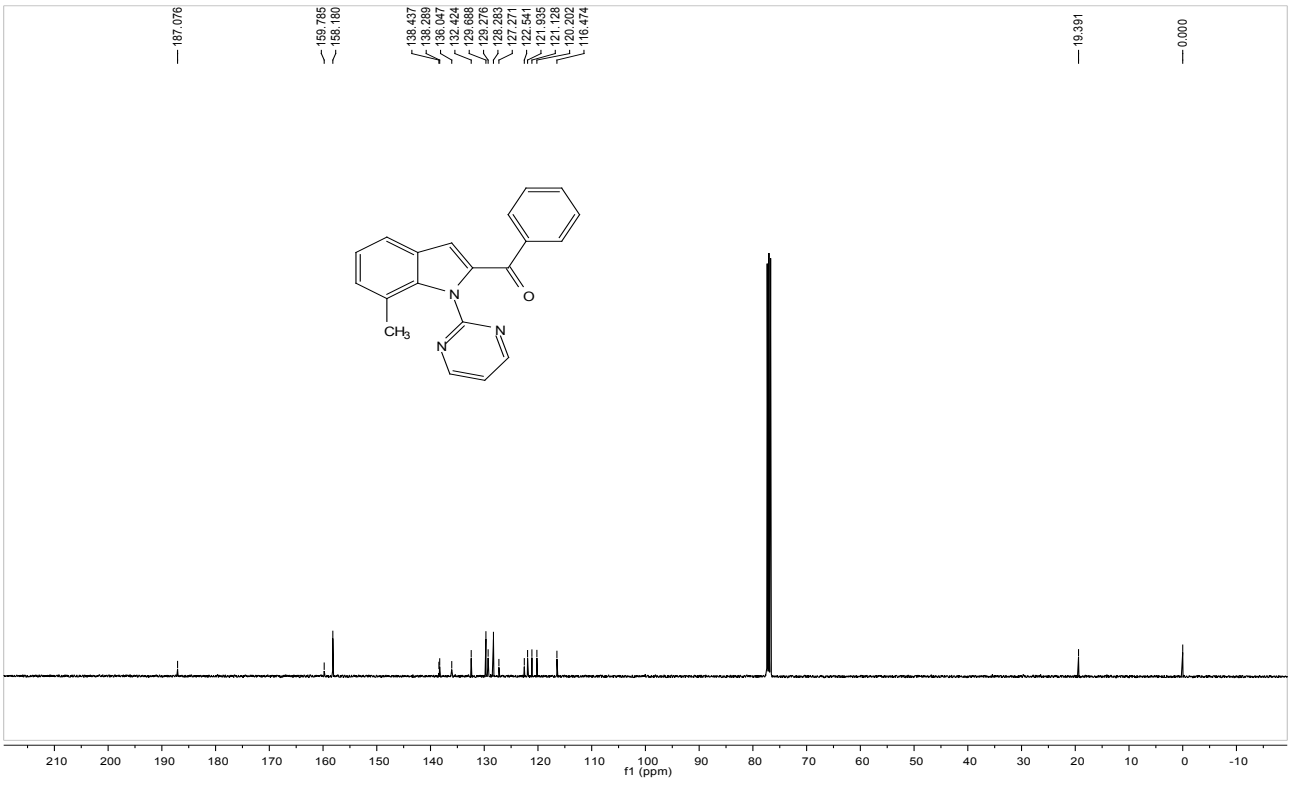
3u


8.645
8.638
8.636
8.633
8.629
7.964
7.960
7.955
7.937
7.931
7.545
7.440
7.423
7.086
7.073
7.070
0.000
Br
N
N
N
O
3.00
2.08
2.16
3.11
2.07
f1 (ppm)

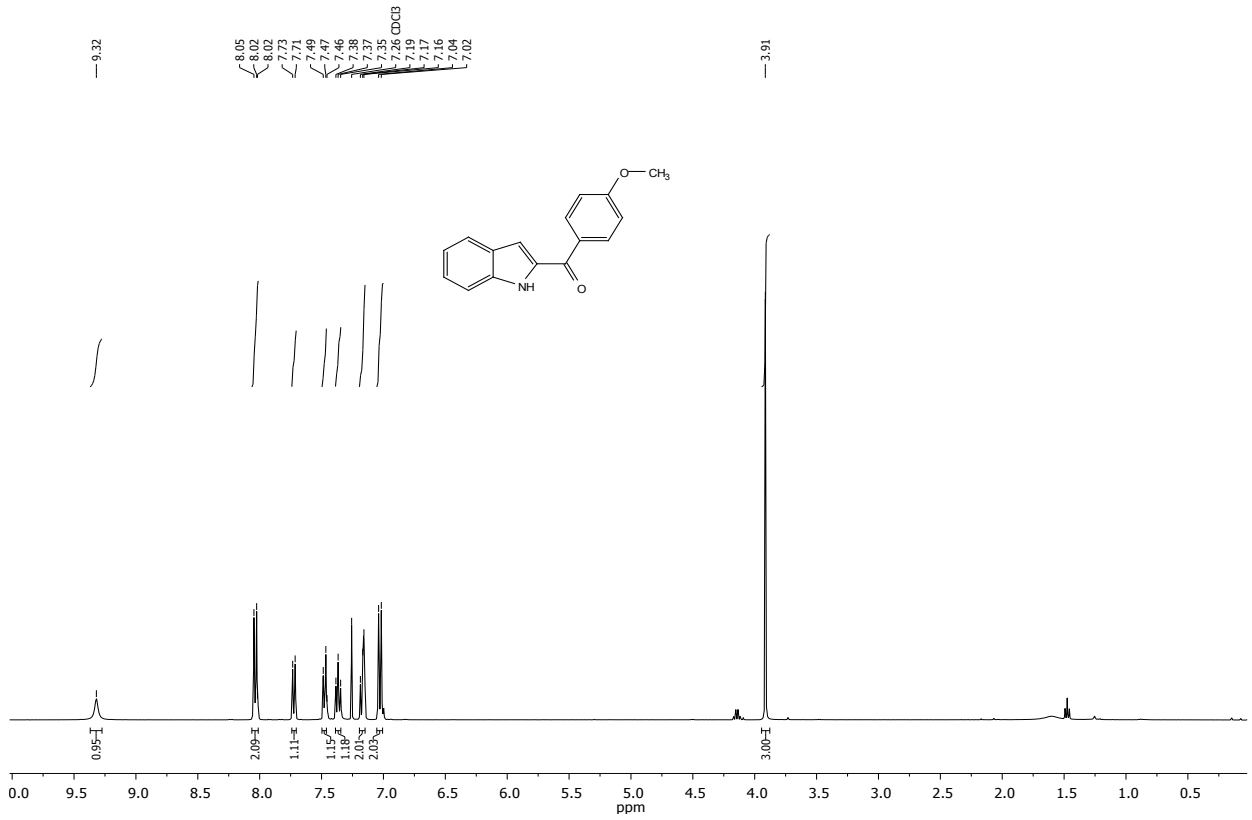

187.305
158.035
156.909
138.632
137.735
137.604
132.851
129.474
128.380
126.861
126.246
123.491
120.255
117.637
117.464
114.655
0.000
Br
N
N
N
O
f1 (ppm)

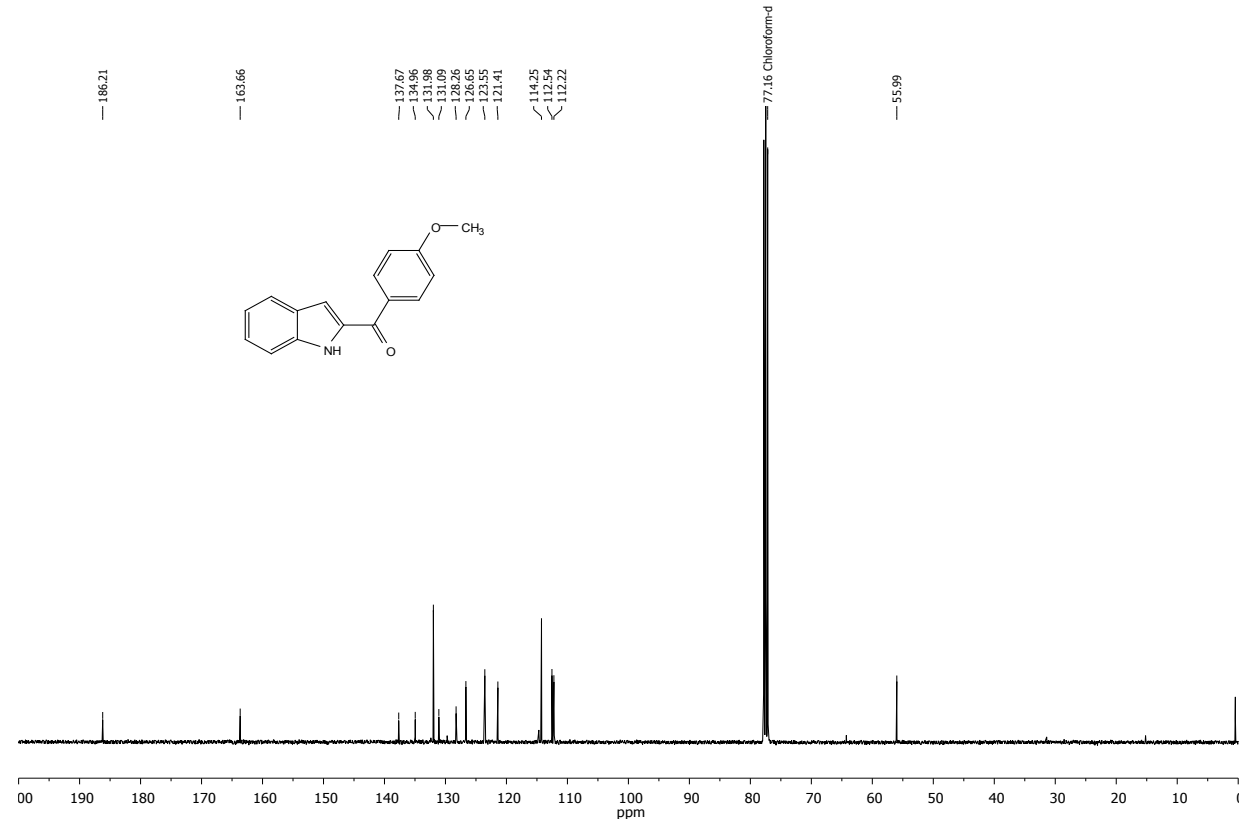
3v


8.888
8.871
7.946
7.942
7.937
7.925
7.919
7.913
7.615
7.611
7.600
7.595
7.591
7.582
7.571
7.566
7.562
7.513
7.506
7.501
7.485
7.480
7.475
7.462
7.457
7.453
7.446
7.430
7.414
7.210
7.141
7.128
7.123
1.973
-0.000
2.00
2.05
2.09
2.06
1.09
1.08
1.96
3.11
f1 (ppm)


187.076
159.785
158.180
138.437
138.289
136.047
132.424
129.688
129.276
128.283
127.271
122.541
121.935
121.128
120.202
116.474
19.391
0.000
f1 (ppm)

3w


9.32
8.05
8.02
8.02
7.73
7.71
7.49
7.47
7.46
7.38
7.37
7.35
7.26 CDCl3
7.19
7.17
7.16
7.04
7.02
3.91
O—CH3
NH
O
0.95
2.09
1.11
1.15
1.18
2.01
2.05
3.00
ppm


186.21
163.66
137.67
134.96
131.98
131.09
128.26
126.65
123.55
121.41
114.25
112.54
112.22
77.16 Chloroform-d
55.99
O—CH3
NH
O
ppm

3cc