Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supplementary Information

Dual Visible-light Photoredox and Palladium(II) Catalysis for Dehydrogenative C-2 Acylation of Indoles at Room Temperature

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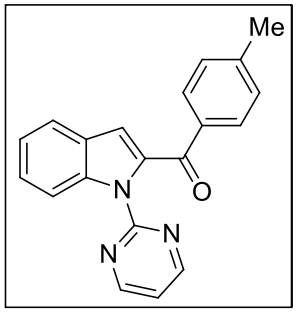
General Information: All manipulations with air-sensitive reagents were carried out under a dry nitrogen atmosphere. Unless otherwise stated, all commercial reagents were used without additional purification. Solvents were dried using standard methods and distilled before use. The starting materials were prepared using literature reported method.[1] TLC was performed on silica gel plates (Merck silica gel 60, f_{254}), and the spots were visualized with UV light (254 and 365 nm) or by charring the plate dipped in $KMnO_4$ or vanillin charring solution. ^1H NMR was recorded at 300 MHz and 600 MHz frequency and ^{13}C NMR spectra were recorded at 75 MHz and 150 MHz frequency in $CDCl_3$ solvent using TMS as the internal standard. Chemical shifts were measured in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br. = broad. Coupling constants, *J* were reported in Hertz unit (Hz). HRMS (m/z) were measured using EI and ESI techniques. Infrared (IR) spectra were recorded on Fourier Transform Infrared Spectroscopy, only intense peaks were reported.

Figure 1. Reaction setup for acylation under dual catalysis

General Experimental Procedure for the C-H Acylation of *N*-pyrimidylindole Derivatives:

To an oven dried 5 mL reaction vial, a mixture of *N*-pyrimidylindole derivative (0.2 mmol), aldehyde (0.3 mmol, 1.5 equiv), $Pd(OAc)_2$ (0.02 mmol, 10 mol %) and photoredox catalyst $Ru(bpy)_3Cl_2 \cdot 6H_2O$ (0.005 mmol, 2.5 mol %) was taken and 1 mL dry acetonitrile was added to it. The reaction vial was purged with nitrogen for 3 min to remove oxygen from the reaction mixture and the vial was immediately closed with teflon cap. A decane solution of TBHP (0.6 mmol, 3.0 equiv) was added to the reaction mixture via micro-liter syringe and the reaction vial was irradiated with blue LED at room temperature. The progress of the reaction was monitored by TLC. After completion the reaction, the reaction mixture was quenched with a saturated solution of $NaHCO_3$ (5 mL) and was extracted with EtOAc (2x25 ml). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired acylation product.

Spectral data:

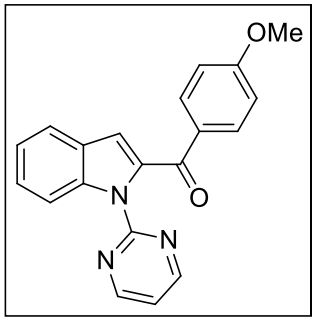


(1-(Pyrimidin-2-yl)-1*H*-indol-2-yl)(*p*-tolyl)methanone, 2a, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (57 mg, 92%), mp 118-120 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.64 (d, *J* = 4.8 Hz, 2H), 8.40 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.24-7.32 (m, 3H), 7.10 (s, 1H), 7.07

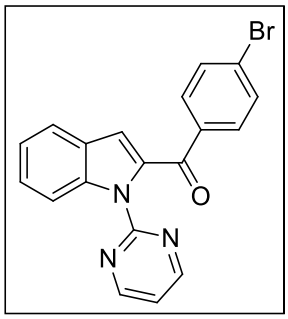
(t, *J* = 4.8 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.4, 158.0, 157.3, 143.6, 138.2, 137.3, 135.3, 129.8, 129.1, 128.0, 126.4, 122.8, 122.4, 117.4, 115.0, 114.2, 21.7; IR (neat): υ_{max} 1639, 1565, 1431, 1281, 1213, 750 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{20}H_{15}N_3ONa$ $[M + Na]^+$: 336.1113; found: 336.1122.



(4-Methoxyphenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2b, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as white solid, (51 mg, 78%), mp 102-104 ºC.

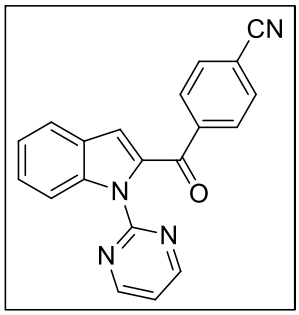
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.66 (d, *J* = 4.8 Hz, 2H), 8.42 (d, *J* = 8.4 Hz, 1H), 8.00 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.08-7.10 (m, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 3.89 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.5, 163.3, 157.9, 157.3, 138.1, 137.3, 131.9, 130.8, 128.0, 126.2, 122.7, 122.3, 117.3, 114.6, 114.2, 113.6, 55.5; IR (neat): υ_{max} 1646, 16021567, 1428, 1267, 1164, 740 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{20}H_{15}N_3O_2[M]^+$: 329.1164; found: 329.1172.



(4-Bromophenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2c, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (55 mg, 73%), mp 96-98 ºC.

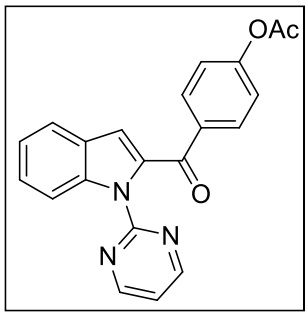
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.62 (d, *J* = 4.8 Hz, 2H), 8.41 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.56 (d, *J* = 8.7 Hz, 2H), 7.42-7.47 (m, 1H), 7.29 (t, *J* = 7.2 H, 1H), 7.10 (s, 1H), 7.06 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.4, 157.9, 157.0, 138.2, 136.7, 136.6, 131.6, 130.9, 127.8, 127.7, 126.6, 122.9, 122.4, 117.4, 115.3, 114.3; IR (neat): υ_{max} 1651, 1572, 1427, 1211, 748 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{19}H_{12}BrN_3ONa$ [M + Na]$^{+}$: 400.0061; found: 400.0049.



4-(1-(Pyrimidin-2-yl)-1*H*-indole-2-carbonyl)benzonitrile, 2d, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (26 mg, 40%), mp 156-158 ºC.

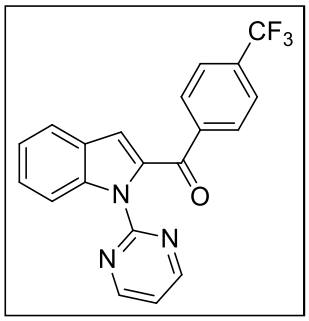
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.63 (d, *J* = 4.8 Hz, 2H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.03 (d, *J* = 8.4 Hz, 2H), 7.73-7.75 (m, 3H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.17 (s, 1H), 7.09 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 185.8, 158.0, 157.0, 141.5, 138.3, 136.2, 132.2, 129.5, 127.1, 123.2, 122.6, 118.0, 117.4, 115.9, 115.7, 114.6; IR (neat): υ_{max} 2226, 1646, 1564, 1433, 1209, 956, 761 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{12}N_4ONa$ [M + Na]$^{+}$: 347.0909; found: 347.0927.



4-(1-(Pyrimidin-2-yl)-1*H*-indole-2-carbonyl)phenyl acetate, 2e, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (60 mg, 85%), mp 122-124 ºC.

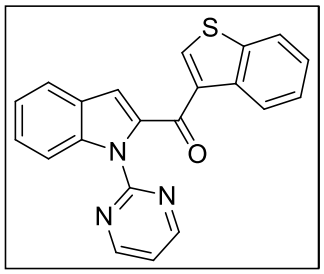
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.66 (d, *J* = 4.8 Hz, 2H), 8.42 (d, *J* = 8.4 Hz, 1H), 8.04 (d, *J* = 9.0 Hz, 2H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 1H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.20 (d, *J* = 9.0 Hz, 2H), 7.15 (s, 1H), 7.09 (t, *J* = 4.8 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.3, 168.9, 158.0, 157.2, 154.1, 138.3, 136.9, 135.5, 131.1, 127.9, 126.5, 122.8, 122.5, 121.6, 117.4, 115.3, 114.2, 21.2; IR (neat): υ_{max} 1762, 1646, 1570, 1421, 1187, 956, 705 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{15}N_3O_3Na$ $[M + Na]^+$: 380.1011; found: 380.1009.



(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(4-(trifluoromethyl)phenyl)methanone, 2f, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as white solid, (62 mg, 69%), mp 118-120 ºC.

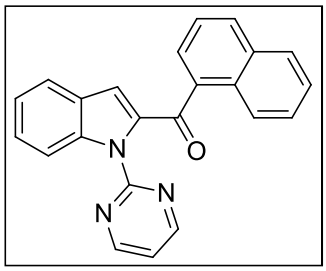
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.63 (d, *J* = 4.8 Hz, 2H), 8.45 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 7.8 Hz, 2H), 7.70-7.73 (m, 3H), 7.48 (t, *J* = 7.8 Hz, 1H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.16 (s, 1H), 7.07 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.3, 158.0, 157.1, 141.0, 138.4, 136.6, 133.9 (q, *J* = 32.3 Hz), 129.7, 127.9, 127.0, 125.4 (q, J = 3.8 Hz), 123.7 (q, *J* = 271.1 Hz), 123.1, 122.7, 117.5, 115.9, 114.5; IR (neat): υ_{max} 1662, 1572, 1435, 1324, 1169, 1121, 1065, 703 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{13}F_3N_3O$ $[M + H]^+$: 368.1011; found: 368.1010.



Benzo[*b*]thiophen-3-yl(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2g, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as white solid, (44 mg, 62%), mp 162-164 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.65-8.68 (m, 3H), 8.41 (d, *J* = 8.4 Hz, 1H), 8.30 (s, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.45-7.53 (m, 3H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.23 (s, 1H), 7.08 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 181.8, 158.0, 157.3, 140.1, 138.4, 138.03, 137.97, 137.1, 135.8, 127.9, 126.5, 125.6, 125.5, 125.2, 122.8, 122.5, 122.3, 117.5, 115.2, 114.1; IR (neat): υ_{max} 1634, 1569, 1421, 743 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{21}H_{13}N_3OSNa$ $[M + Na]^+$: 378.0677; found: 378.0677.

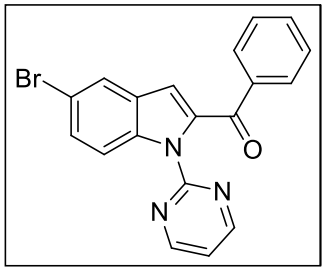


Naphthalen-1-yl(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2h, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (52 mg, 75%), mp 142-144 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.71 (d, *J* = 8.4 Hz, 1H), 8.54 (d, *J* = 4.8 Hz, 2H), 8.38 (d, *J* = 8.7 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.76 (dd, J_1 = 6.9 Hz, J_2 = 1.2 Hz, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.52-7.64 (m, 2H), 7.36-7.49 (m, 2H), 7.27-7.32 (m, 1H), 7.16 (s, 1H), 6.96 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 188.8, 157.8, 157.3, 138.9, 138.5,

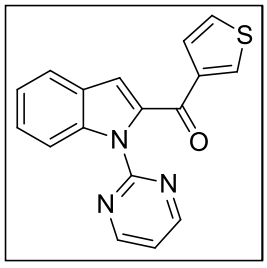
136.0, 133.7, 132.3, 131.1, 129.0, 128.2, 127.9, 127.6, 126.7, 126.4, 126.0, 124.1, 122.8, 122.6, 117.3, 116.2, 114.2; IR (neat): υ_{max} 1654, 1572, 1447, 779 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{23}H_{15}N_3O[M]^+$: 349.1215; found: 349.1202.



(5-Bromo-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone, 2i, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (54 mg, 72%), mp 122-124 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.63 (d, *J* = 4.8 Hz, 2H), 8.35 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 7.8 Hz, 2H), 7.84 (d, *J* = 1.8 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.53 (dd, J_1 = 9.0 Hz, J_2 = 1.8 Hz, 1H), 7.47 (m, 2H), 7.08 (t, *J* = 4.2 Hz, 1H), 7.04 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.4, 158.0, 156.9, 138.0, 137.6, 136.6, 132.9, 129.7, 129.5, 129.1, 128.4, 124.7, 117.6, 116.0, 115.9, 113.6; IR (neat): υ_{max} 1665,1571, 1442, 1266, 798, 715 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{19}H_{12}BrN_3ONa$ $[M + Na]^+$: 400.0061; found: 400.0068.

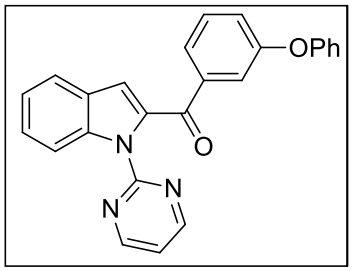


(1-(Pyrimidin-2-yl)-1*H*-indol-2-yl)(thiophen-3-yl)methanone, 2j, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (45 mg, 74%), mp 152-154 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.71 (d, *J* = 4.8 Hz, 2H), 8.36 (dd, J_1 = 8.4 Hz, J_2 = 0.6 Hz, 1H), 8.13-8.14 (m, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.67 (dd, J_1 = 4.8 Hz, J_2 = 1.2 Hz, 1H), 7.45-7.47

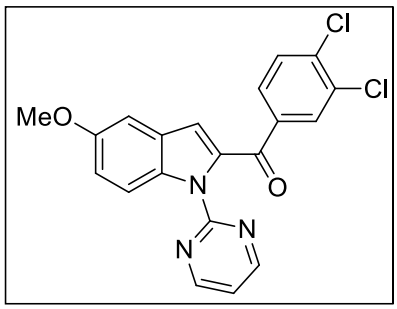
(m, 1H), 7.36-7.37 (m, 1H), 7.30-7.32 (m, 1H), 7.24 (d, *J* = 1.2 Hz, 1H), 7.13 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 180.8, 158.0, 157.4, 142.2, 138.5, 137.5, 133.5, 127.9, 127.8, 126.6, 126.2, 122.8, 122.5, 117.5, 115.1, 114.0; IR (neat): υ_{max} 1638, 1567, 1424, 1341, 824, 740 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{17}H_{11}N_3OSNa$ [M + Na]$^+$: 328.0521; found: 328.0506.



(3-Phenoxyphenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2k, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 8:2 hexane/ethyl acetate) afforded the desired product as colorless gummy liquid, (51 mg, 65%).

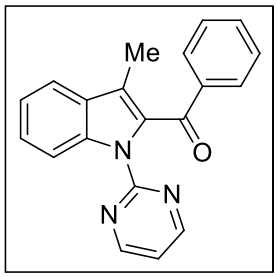
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.65 (d, *J* = 4.8 Hz, 2H), 8.39 (d, *J* = 8.7 Hz, 1H), 7.65-7.72 (m, 3H), 7.27-7.48 (m, 5H), 7.19-7.23 (m, 1H), 7.12-7.16 (m, 2H), 7.08 (t, *J* = 4.8 Hz, 1H), 7.00-7.04 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.7, 157.9, 157.5, 157.2, 156.6, 139.7, 138.3, 136.8, 129.9, 129.7, 127.9, 126.6, 124.4, 123.7, 122.9, 122.8, 122.5, 119.2, 119.0, 117.4, 115.7, 114.2; IR (neat): υ_{max} 1653, 1572, 1428, 1241, 747 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{25}H_{17}N_3O_2Na$ [M + Na]$^+$: 414.1218; found: 414.1215.



(3,4-Dichlorophenyl)(5-methoxy-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2l, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as `white solid, (43 mg, 54%), mp 152-154 ºC.

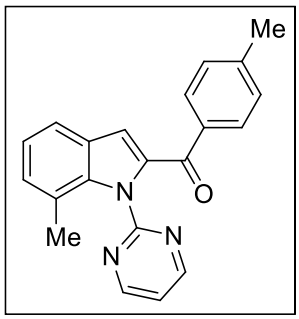
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.62 (d, *J* = 4.8 Hz, 2H), 8.39 (d, *J* = 9.0 Hz, 1H), 8.08 (d, *J* = 2.4 Hz, 1H), 7.76 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.14 (d, *J* = 2.4 Hz, 1H), 7.12 (dd, J_1 = 9.6 Hz, J_2 = 3.0 Hz, 1H), 7.08 (t, *J* = 4.8 Hz, 1H), 7.06 (s, 1H), 3.91 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 185.1, 157.9, 156.1, 137.8, 137.1, 136.6, 133.3, 133.0, 131.1, 130.4, 128.6, 128.3, 117.2, 116.9, 115.7, 115.1, 103.4, 55.7; IR (neat): υ_{max} 1659, 1569, 1428, 1216, 807 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{20}H_{13}Cl_2N_3O_2[M]^+$: 397.0385; found: 397.0391.



(3-Methyl-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone, 2m, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (52 mg, 83%), mp 154-156 ºC.

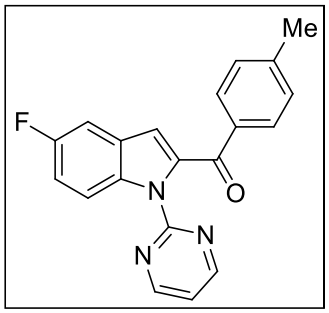
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.64 (d, *J* = 8.4 Hz, 1H), 8.46 (d, *J* = 4.8 Hz, 2H), 7.78 (d, *J* = 7.5 Hz, 2H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.40-7.50 (m, 2H), 7.30-7.37 (m, 3H), 6.86 (t, *J* = 4.8 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 189.4, 157.5, 157.0, 139.2, 136.3, 133.2, 132.2, 130.2, 128.5, 128.3, 126.1, 122.5, 121.7, 120.1, 116.1, 115.1, 9.3; IR (neat): υ_{max} 1657, 1563, 1435, 1262, 948, 746 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{15}N_3ONa$ $[M + Na]^+$: 336.1113; found: 336.1109.



(7-Methyl-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*p*-tolyl)methanone, 2n, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as yellow gummy liquid, (59 mg, 91%).

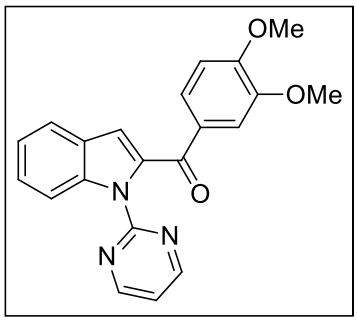
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.87 (d, *J* = 4.8 Hz, 2H), 7.86 (d, *J* = 7.8 Hz, 2H), 7.60 (t, *J* = 4.8 Hz, 1H), 7.41 (t, *J* = 4.8 Hz, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.21 (s, 1H), 7.13-7.14 (m, 2H), 2.44 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.0, 160.0, 158.3, 143.4, 138.4, 136.3, 135.7, 130,0, 129.3, 129.2, 127.4, 122.7, 122.0, 121.2, 120.4, 116.2, 21.8, 19.6; IR (neat): υ_{max} 2922, 1639,1529, 1422, 1233, 960, 749 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{17}N_3ONa$ [M + Na]$^+$: 350.1269; found: 350.1266.



(5-Fluoro-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*p*-tolyl)methanone, 2o, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 8:2 hexane/ethyl acetate) afforded the desired product as white solid, (50 mg, 75%), mp 124-126 ºC.

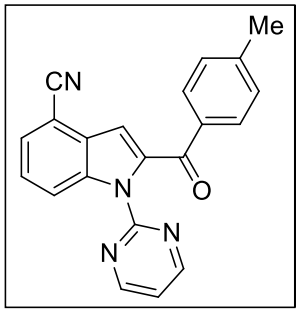
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.63 (d, *J* = 4.8 Hz, 2H), 8.42-8.44 (m, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.35 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 7.26 (d, *J* = 7.8 Hz, 2H), 7.19, (td, J_1 = 9.6 Hz, J_2 = 3.0 Hz, 1H), 7.08 (t, *J* = 4.8 Hz, 1H), 7.04 (s, 1H), 2.43 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.3, 159.2 (d, *J* = 237.9 Hz), 157.9, 157.0, 143.7, 138.6, 135.1, 134.4, 129.7, 129.1, 128.7 (d, *J* = 9.9 Hz), 117.4, 115.6 (d, *J* = 9.0 Hz), 114.4 (d, *J* = 25.2 Hz), 113.8 (d, *J* = 4.5 Hz), 107.1 (d, *J* = 23.4 Hz), 21.7; IR (neat): υ_{max} 1646, 1567, 1424,1169, 816, 749 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{14}FN_3ONa$ [M + Na]$^+$: 354.1019; found: 354.1017.



(3,4-Dimethoxyphenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2p, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (62 mg, 87%), mp 178-180 ºC.

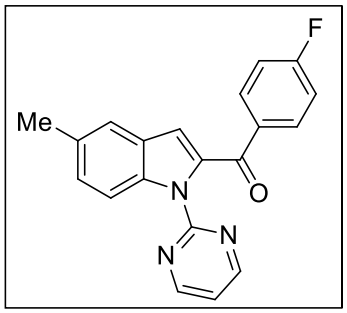
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.67 (d, *J* = 5.4 Hz, 2H), 8.42 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.64 (dd, J_1 = 7.8 Hz, J_2 = 2.4 Hz, 1H), 7.61 (d, *J* = 1.8 Hz, 1H), 7.44-7.47 (m, 1H), 7.30-7.32 (m, 1H), 7.11 (s, 1H), 7.09 (t, *J* = 4.8 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 3.96 (s, 3H), 3.95 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.5, 157.9, 157.3, 153.1, 148.9, 138.1, 137.1, 130.9, 128.0, 126.2, 124.8, 122.7, 122.3, 117.3, 114.7, 114.2, 111.3, 109.9, 56.1, 56.0; IR (neat): υ_{max} 1641, 1514, 1427, 1266, 1139, 760 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{17}N_3O_3Na$ [M + Na]$^+$: 382.1168; found: 382.1171.



2-(4-Methylbenzoyl)-1-(pyrimidin-2-yl)-1*H*-indole-4-carbonitrile, 2q, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (45 mg, 67%), mp 188-190 ºC.

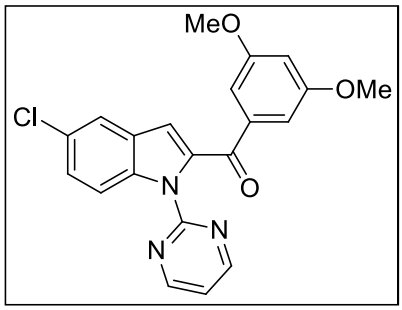
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.67-8.69 (m, 3H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.48-7.51 (m, 1H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.25 (s, 1H), 7.17 (t, *J* = 4.8 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.9, 158.2, 156.7, 144.3, 139.2, 137.4, 134.5, 129.9, 129.4, 129.3, 127.7, 125.6, 119.3, 118.1, 117.7, 111.4, 104, 9, 21.7; IR (neat): υ_{max} 2219, 1666, 1571, 1431, 1266, 754 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{14}N_4ONa$ $[M + Na]^+$: 361.1065; found: 361.1071.



(4-Fluorophenyl)(5-methyl-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2r, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as yellow gummy liquid, (56 mg, 80%).

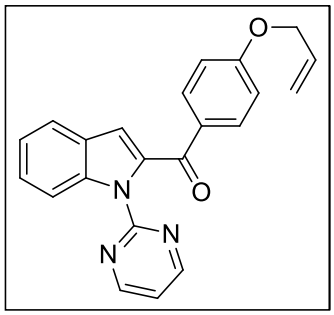
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.61 (d, *J* = 4.8 Hz, 2H), 8.31 (d, *J* = 8.4 Hz, 1H), 7.98-8.00 (m, 2H), 7.48 (s, 1H), 7.28 (dd, J_1 = 8.4 Hz, J_2 = 1.8 Hz, 1H), 7.10-7.13 (m, 2H), 7.03-7.05 (m, 2H), 2.48 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.2, 165.5 (d, *J* = 253.5 Hz), 157.9, 157.3, 136.9, 136.6, 134.5 (d, *J* = 3.2 Hz), 132.4, 132.0 (d, *J* = 9.2 Hz), 128.3, 128.2, 122.1, 117.2, 115.5 (d, *J* = 21.8 Hz), 115.1, 114.1, 21.4 IR (neat): υ_{max} 1653, 1568, 1428, 1222, 1154, 757, 597 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{15}FN_3O$ $[M + H]^+$: 332.1199; found: 332.1200.



(5-Chloro-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(3,5-dimethoxyphenyl)methanone, 2s, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 8:2 hexane/ethyl acetate) afforded the desired product as white solid, (49 mg, 63%), mp 52-54 ºC.

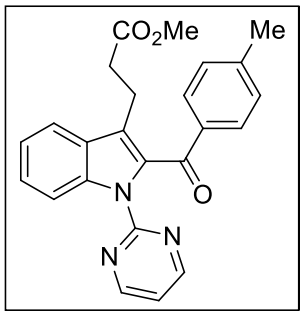
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.63 (d, *J* = 5.1 Hz, 2H), 8.37 (d, *J* = 9.0 Hz, 1H), 7.66 (d, *J* = 2.1 Hz, 1H), 7.38 (d, *J* = 9.0 Hz, 1H), 7.05-7.11 (m, 4H), 6.64-6.66 (m, 1H), 3.80 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.1, 160.8, 158.1, 157.1, 139.6, 138.2, 136.5, 129.2, 128.4, 126.7, 121.8, 117.7, 115.8, 114.0, 107.4, 105.7, 55.7; IR (neat): υ_{max} 1657, 1593, 1430, 1159, 757 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{17}ClN_3O_3$ $[M + H]^+$: 394.0958; found: 394.0965.



(4-(Allyloxy)phenyl)(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2t, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (46 mg, 65%), mp 84-86 ºC.

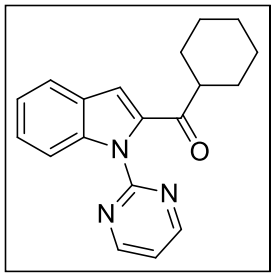
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.65 (d, *J* = 4.8 Hz, 2H), 8.40 (d, *J* = 8.4 Hz, 1H), 7.99 (d, *J* = 9.0 Hz, 2H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.09 (s, 1H), 7.07 (t, *J* = 4.8 Hz, 1H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.00-6.12 (m, 1H), 5.43 (d, *J* = 17.4 Hz, 1H), 5.32 (d, *J* = 10.5 Hz, 1H), 4.60 (d, *J* = 5.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.4, 162.3, 157.9, 157.2, 138.0, 137.2, 132.4, 131.8, 130.7, 127.9, 126.2, 122.7, 122.3, 118.1, 117.3, 114.6, 114.2, 114.1, 68.8; IR (neat): υ_{max} 1647, 1567, 1428, 1270, 1164, 746 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{22}H_{17}N_3O_2Na$ $[M + Na]^+$: 378.1218; found: 378.1190.



Methyl 3-(2-(4-methylbenzoyl)-1-(pyrimidin-2-yl)-1*H*-indol-3-yl)propanoate, 2u, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 4:1 hexane/ethyl acetate) afforded the desired product as white solid, (60 mg, 76%), mp 130-132 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.68 (d, *J* = 8.4 Hz, 1H), 8.47 (d, *J* = 4.8 Hz, 2H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.48 (t, *J* = 8.4 Hz, 1H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 7.8 Hz, 2H), 6.87 (t, *J* = 4.8 Hz, 1H), 3.59 (s, 3H), 3.16 (t, *J* = 7.2 Hz, 2H), 2.73 (t, *J* = 8.4 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.0, 173.2, 157.5, 156.9, 143.0, 136.5, 136.2, 133.7, 129.1, 129.0, 128.5, 125.9, 123.6, 122.7, 120.0, 116.2, 115.4, 51.5, 35.1, 21.6, 19.7; IR (neat): υ_{max} 1737, 1654, 1566, 1435, 1266, 746 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{24}H_{21}N_3O_3Na$ $[M + Na]^+$: 422.1481; found: 422.1498.

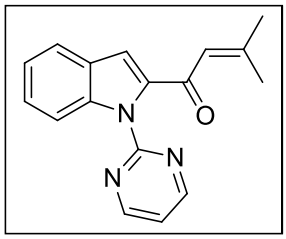


Cyclohexyl(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)methanone, 2v, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (52 mg, 94%), mp 142-144 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.79 (d, *J* = 4.8 Hz, 2H), 8.11 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 1H), 7.21-7.30 (m, 3H), 2.97-3.07 (m, 1H), 1.99-2.03 (m, 2H), 1.83-

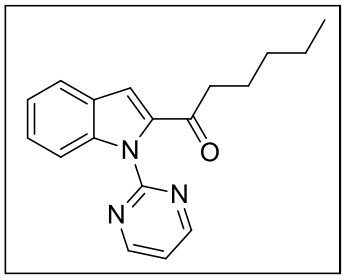
1.88 (m, 2H), 1.51-1.75 (m, 4H), 1.25-1.41 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 197.7, 158.2, 157.9, 138.9, 137.6, 127.7, 126.4, 122.53, 122.49, 117.9, 113.5, 112.7, 48.7, 29.4, 25.9, 25.8; IR (neat): υ_{max} 2923, 1676, 1571, 1437, 1337, 1250, 1150, 806, 747 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{19}H_{20}N_3O$ $[M + H]^+$: 306.1606; found: 306.1604.



3-Methyl-1-(1-(pyrimidin-2-yl)-1*H*-indol-2-yl)but-2-en-1-one, 2w, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as white solid, (35 mg, 63%), mp 90-92 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.78 (d, *J* = 5.1 Hz, 2H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.19-7.28 (m, 3H), 6.69 (s, 1H), 2.17 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 183.4, 158.1, 156.4, 139.6, 139.0, 127.5, 126.4, 122.6, 122.4, 122.3, 118.0, 113.3, 112.9, 27.9, 21.0; IR (neat): υ_{max} 1652, 1607, 1564, 1428, 1006, 750 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{17}H_{15}N_3O[M]^+$: 277.1215; found: 277.1216.

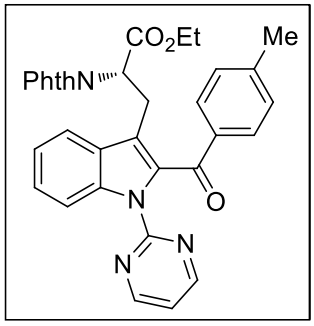


1-(1-(Pyrimidin-2-yl)-1*H*-indol-2-yl)hexan-1-one, 2x, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 9:1 hexane/ethyl acetate) afforded the desired product as yellow gummy liquid, (51 mg, 88%).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.79 (d, *J* = 4.8 Hz, 2H), 8.02 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 1H), 7.31 (s, 1H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.22 (t, *J* = 4.8 Hz, 1H),

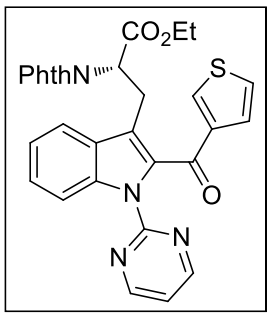
2.94 (t, *J* = 7.8 Hz, 2H), 1.76-1.81 (m, 2H), 1.35-1.41 (m, 4H), 0.92 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 193.9, 158.2, 157.8, 139.1, 137.9, 127.5, 126.7, 122.7, 122.5, 118.2, 113.5, 113.2, 40.5, 31.4, 24.4, 22.5, 14.0; IR (neat): υ_{max} 2924, 2859, 1677, 1569, 1427, 810, 746 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{19}N_3ONa$ $[M + Na]^+$: 316.1426; found: 316.1416.



Ethyl 2-(1,3-dioxoisoindolin-2-yl)-3-(2-(4-methylbenzoyl)-1-(pyrimidin-2-yl)-1*H*-indol-3-yl)propanoate, 2y, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 1:1 hexane/ethyl acetate) afforded the desired product as white solid, (106 mg, 72%), mp 190-192 ^{o}C.

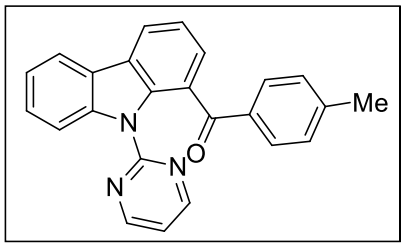
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.59 (d, *J* = 8.4 Hz, 1H), 8.38 (d, *J* = 4.8 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.54-7.55 (m, 4H), 7.36-7.44 (m, 3H), 7.21-7.27 (m, 1H), 6.78-6.84 (m, 3H), 5.27 (t, *J* = 7.5 Hz, 1H), 4.21 (q, *J* = 7.2 Hz, 2H), 3.85 (d, *J* = 7.5 Hz, 2H), 2.19 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 188.0, 168.6, 167.2, 157.5, 142.6, 135.94, 135.89, 134.8, 133.4, 131.6, 129.0, 128.7, 128.3, 126.0, 123.1, 122.8, 120.2, 119.6, 116.3, 115.3, 62.0, 52.7, 23.6, 21.5, 14.1; IR (neat): υ_{max} 1720, 1569, 1428, 1383, 1252, 1029, 720 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{33}H_{26}N_4O_5[M]^+$: 558.1903; found: 558.1898.



Ethyl 2-(1,3-dioxoisoindolin-2-yl)-3-(1-(pyrimidin-2-yl)-2-(thiophene-3-carbonyl)-1*H*-indol-3-yl)propanoate, 2z, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 6:4 hexane/ethyl acetate) afforded the desired product as white solid, (73 mg, 50%), mp 140-142 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.54 (d, *J* = 8.4 Hz, 1H), 8.44 (d, *J* = 4.8 Hz, 2H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.55-7.63 (m, 4H), 7.48-7.49 (m, 1H), 7.35-7.40 (m, 1H), 7.19-7.25 (m, 2H), 6.94 (dd, J_1 = 5.1 Hz, J_2 = 3.0 Hz, 1H), 6.85 (t, *J* = 4.8 Hz, 1H), 5.26 (t, *J* = 7.5 Hz, 1H), 4.23 (q, *J* = 7.2 H, 2H), 3.90 (d, *J* = 7.5 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 181.8, 168.6, 167.2, 157.6, 157.0, 143.3, 136.2, 135.1, 133.7, 131.6, 128.9, 126.8, 126.3, 125.6, 123.2, 122.9, 120.9, 119.8, 116.4, 115.1, 62.1, 52.8, 23.6, 14.1; IR (neat): υ_{max} 1717, 1568, 1437, 1030, 719 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{30}H_{22}N_4O_5S[M]^+$:550.1311; found: 550.1307.



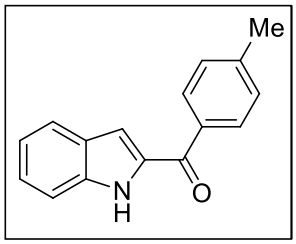
(9-(Pyrimidin-2-yl)-9*H*-carbazol-1-yl)(*p*-tolyl)methanone, 2aa, (Table 2)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 8:2 hexane/ethyl acetate) afforded the desired product as white solid, (49 mg, 68%), mp 204-206 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.54 (d, *J* = 8.4 Hz, 1H), 8.38 (d, *J* = 4.8 Hz, 2H), 8.22 (d, *J* = 7.8 Hz, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.47-7.53 (m, 2H), 7.36-7.43 (m, 2H), 7.18 (d, *J* = 7.8 Hz, 2H), 6.91 (t, *J* = 4.8 Hz, 1H), 2.93 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 195.1, 158.0, 157.4, 143.1, 140.0, 136.2, 134.9, 130.0, 128.9, 127.4, 127.1, 127.0, 126.8, 125.1, 122.4, 122.2, 121.5, 119.7, 117.2, 114.4, 21.7; IR (neat): υ_{max} 1641, 1564, 1416, 1275, 751 cm^{-1}; HRMS (EI, m/z) calcd.for $C_{24}H_{17}N_3O[M]^+$: 363.1372; found: 363.1373.

General procedure for the deportation of the directing group:[1]

To an oven dried 10 ml sealed tube a mixture of the acylation product **2a** (0.2 mmol) and sodium ethoxide (0.6 mmol, 3 equiv) were taken and dry DMSO (1 mL) was added to it. The reaction tube was placed into a preheated oil bath at 100 ^{o}C and allowed to stir for 24h. After completion the reaction, the reaction mixture was extracted with ethyl acetate (3x20 mL). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired product.



(1*H*-indol-2-yl)(*p*-tolyl)methanone, 3a, (Scheme 1b)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 19:1 hexane/ethyl acetate) afforded the desired product as white solid, (36 mg, 76%), mp 190-192 ^{o}C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 9.45 (br. s, 1H), 7.93 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.49 (d, *J* = 8.1 Hz, 1H), 7.33-7.40 (m, 3H), 7.14-7.19 (m, 2H), 2.47 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.9, 143.1, 137.4, 135.3, 134.4, 129.4, 129.2, 127.7, 126.3, 123.1, 120.9, 112.4, 112.2, 21.7;

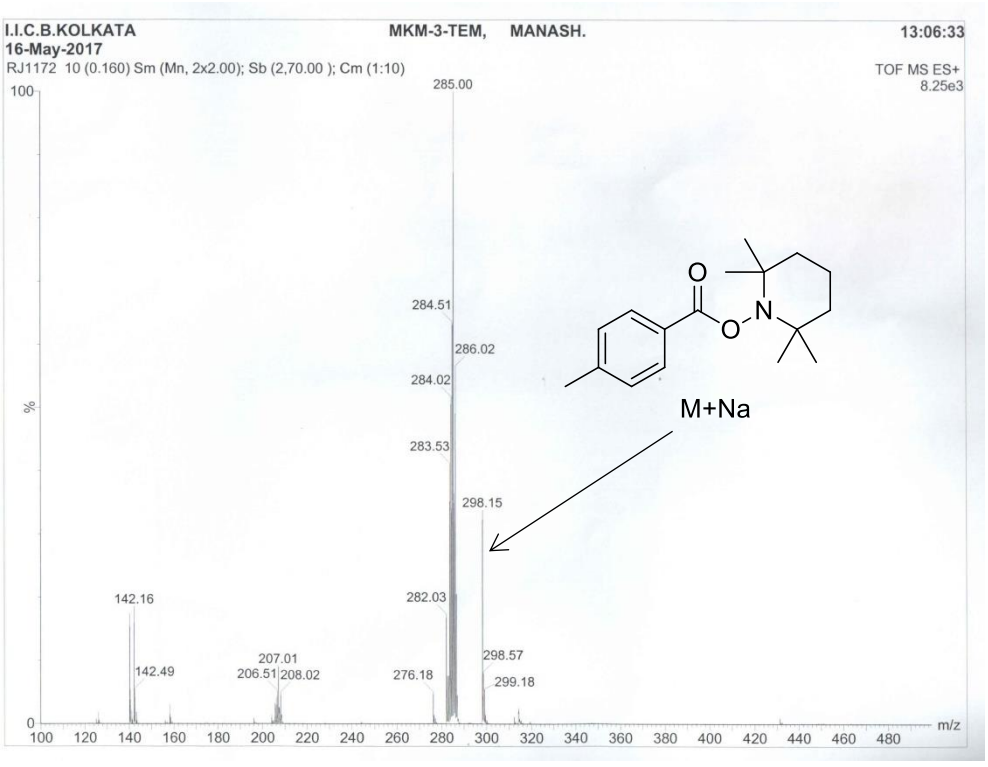
Gram scale experiment:

To an oven dried 100 mL round bottom flask, a mixture of *N*-pyrimidylindole (1.0 gm, 5.12 mmol), *p*-tolualdehyde (15.4 mmol, 1.5 equiv), $Pd(OAc)_2$ (0.51 mmol, 10 mol %) and photoredox catalyst $Ru(bpy)_3Cl_2{\cdot}6H_2O$ (0.13 mmol, 2.5 mol %) was taken and 25 mL dry acetonitrile was added to it. The reaction vessel was purged with nitrogen for 3 min to remove oxygen from the reaction mixture. After purging a decane solution of TBHP (0.6 mmol, 3.0 equiv) was added to the reaction mixture via syringe and the reaction vessel was immediately closed with glass stopper. The reaction vessel was irradiated with blue LED at room temperature.

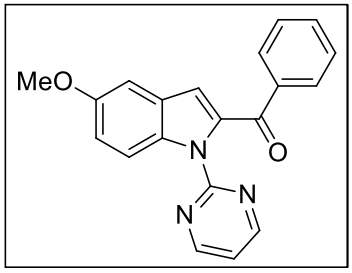
The progress of the reaction was monitored by TLC. After completion the reaction (36 h), the reaction mixture was quenched with a saturated solution of $NaHCO_3$ (50 mL) and was extracted with EtOAc (2x100 ml). The combined organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired acylation product in78% yield.

Control experiment:

The standard reaction was performed in presence of 2 equivalent of TEMPO as a radical scavenger and the reaction did not afford the desire product. The ESI mass spectroscopy of the crude reaction mixture shows an adduct formation of the aldehyde with TEMPO.



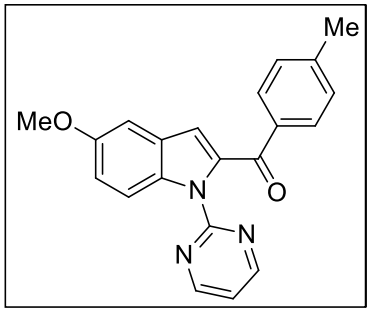
Synthesis of anticancer D-64131 and D-68144:



(5-Methoxy-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(phenyl)methanone, 2ab, (Scheme 3)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as white solid, (48 mg, 73%), mp 128-130 ºC.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.60 (d, *J* = 4.8 Hz, 2H), 8.34 (d, *J* = 9.0 Hz, 1H), 7.93-7.96 (m, 2H), 7.51-7.56 (m, 1H), 7.40-7.45 (m, 2H), 7.12 (d, *J* = 2.4 Hz, 1H), 7.08 (dd, J_1 = 9.0 Hz, J_2 = 2.4 Hz, 1H), 7.05 (s, 1H), 7.02 (t, *J* = 4.8 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.8, 158.0, 156.1, 138.3, 137.8, 133.4, 132.7, 129.6, 128.9, 128.4, 117.2, 116.6, 115.6, 115.0, 103.7, 55.9; IR (neat): υ_{max} 1653, 1592, 1435, 1207, 753 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{20}H_{15}N_3O_2Na$ $[M + Na]^+$: 352.1062; found: 352.1063.

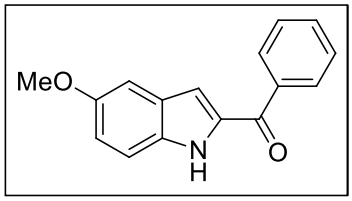


(5-Methoxy-1-(pyrimidin-2-yl)-1*H*-indol-2-yl)(*p*-tolyl)methanone, 2ac, (Scheme 3)

The same general procedure was followed. Column chromatography (SiO_2, eluting with 17:3 hexane/ethyl acetate) afforded the desired product as white solid, (45 mg, 65%), mp 164-166 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.62 (d, *J* = 4.8 Hz, 2H), 8.35 (d, *J* = 9.6 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.13 (d, *J* = 2.4 Hz, 1H), 7.09 (dd, J_1 = 9.0 Hz, J_2 = 3.2 Hz, 1H), 7.04 (t, *J* = 4.8 Hz, 1H), 7.03 (s, 1H), 3.90 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.4, 157.9, 157.2, 155.9, 143.5, 137.7, 135.4, 133.1, 129.7, 129.0, 128.7, 117.1, 116.3, 115.4, 114.5, 103.4, 55.7, 21.7; IR (neat): υ_{max} 1657, 1567, 1444, 1247, 1117, 734 cm^{-1}; HRMS (ESI, m/z) calcd.for $C_{21}H_{17}N_3O_2$ $[M + H]^+$: 344.1399; found: 344.1392.

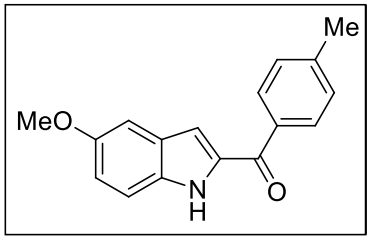
Deprotection:



(5-Methoxy-1*H*-indol-2-yl)(phenyl)methanone, (D-64131), 3b, (Scheme 3)

The same general deportation procedure was followed. Column chromatography (SiO_2, eluting with 19:1 hexane/ethyl acetate) afforded the desired product as white solid, (33 mg, 66%), mp 164-166 ºC.

^{1}H NMR (600 MHz, $CDCl_3$): δ 9.20 (br. s, 1H), 8.00 (d, *J* = 7.2 Hz, 2H), 7.64 (t, *J* = 7.8 Hz, 1H), 7.53-7.56 (m, 2H), 7.39 (d, *J* = 9.0 Hz, 1H), 7.07-7.11 (m, 3H), 3.87 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.9, 154.8, 138.0, 134.7, 132.9, 132.3, 129.2, 128.4, 128.0, 118.4, 113.0, 112.2, 102.8, 55.7;



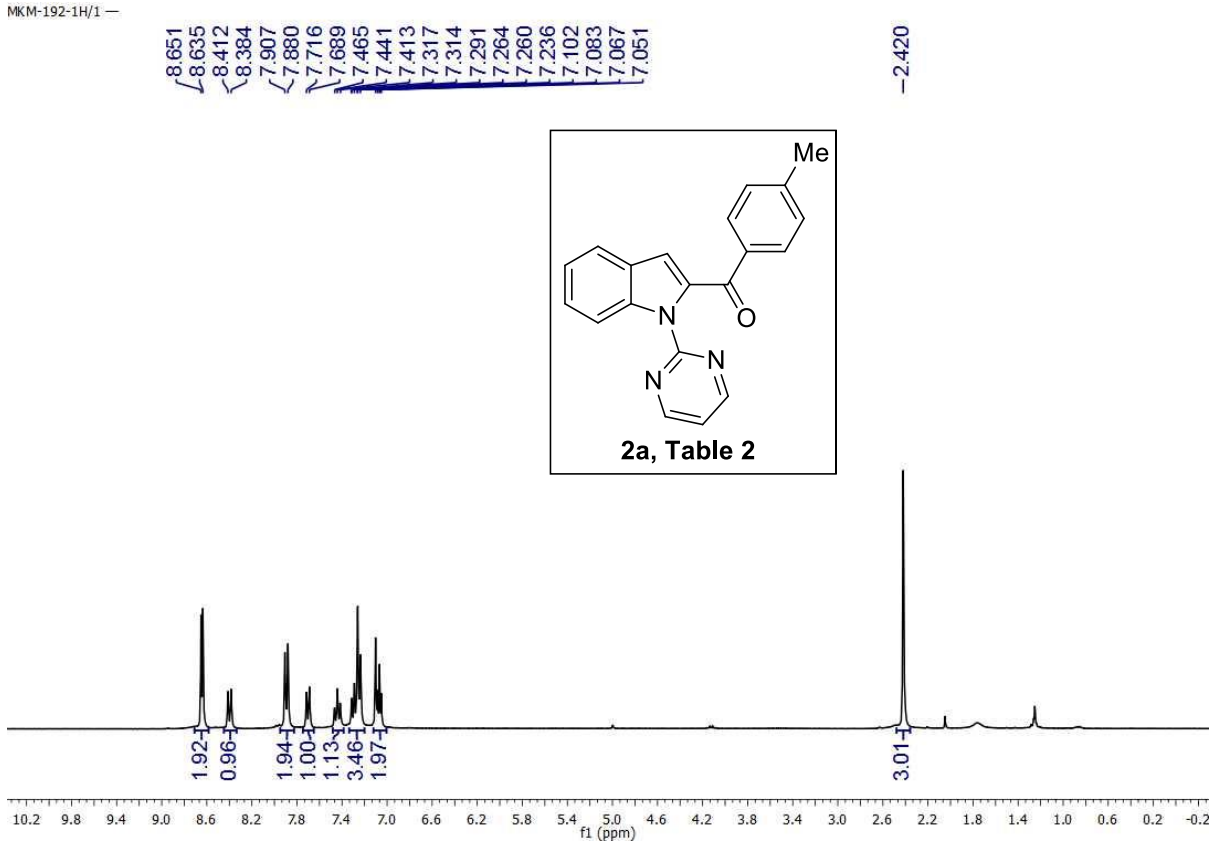
(5-Methoxy-1*H*-indol-2-yl)(*p*-tolyl)methanone, (D-68144), 3c, (Scheme 3)

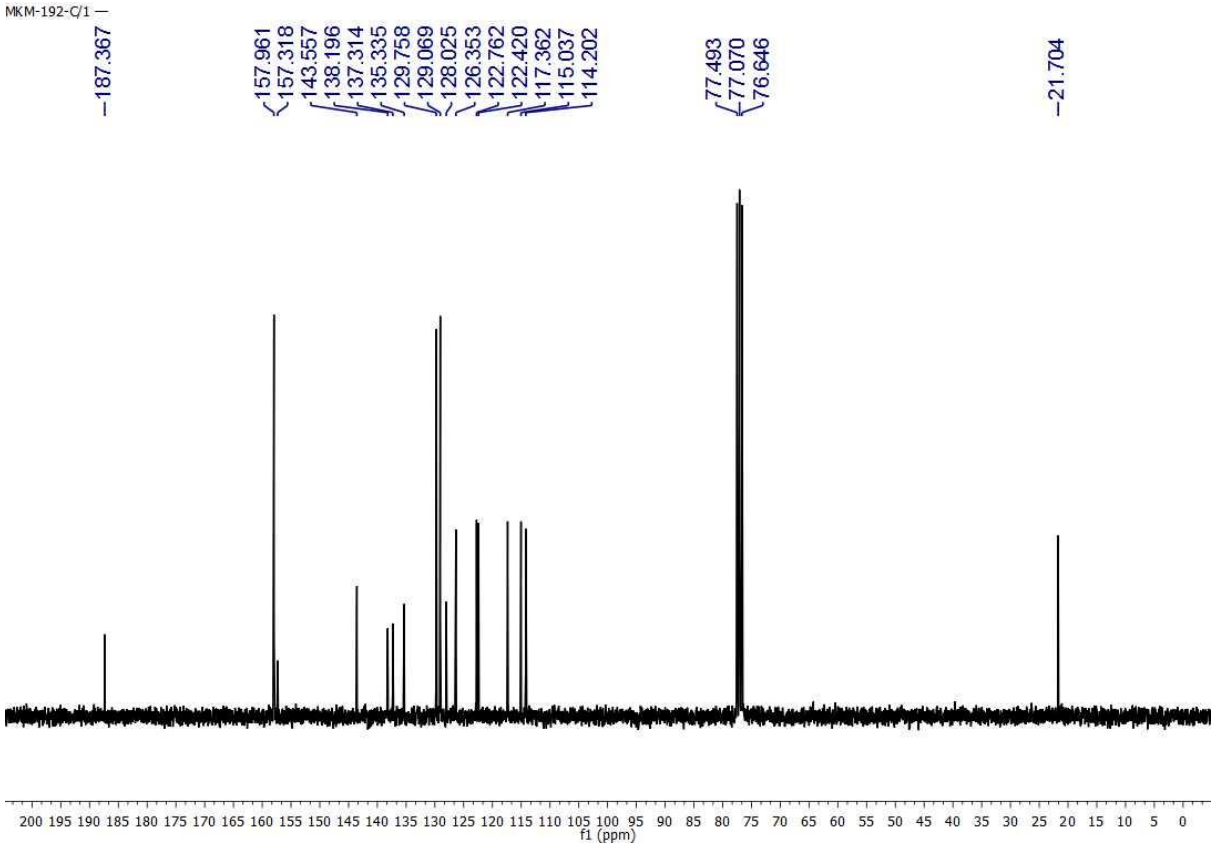
The same general deportation procedure was followed. Column chromatography (SiO_2, eluting with 19:1 hexane/ethyl acetate) afforded the desired product as white solid, (37 mg, 71%), mp 208-210 ºC.

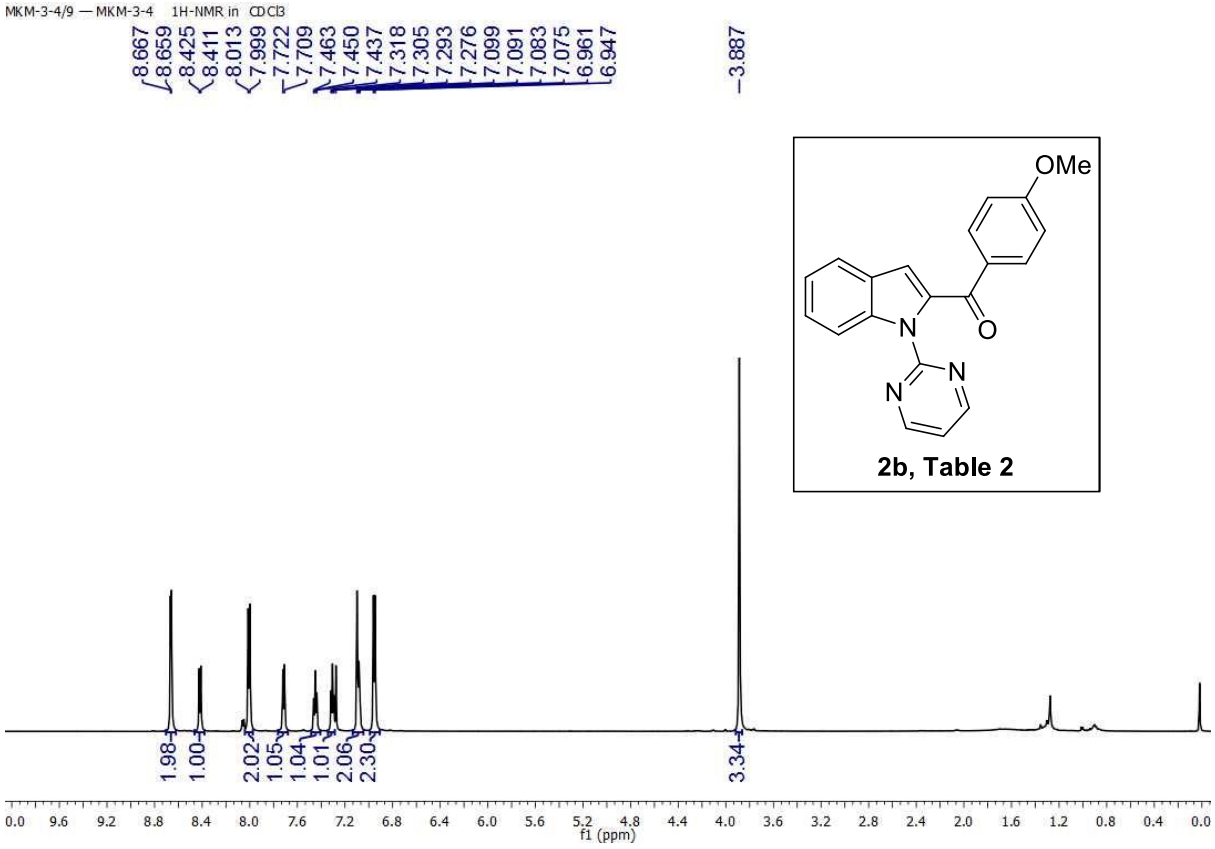
^{1}H NMR (600 MHz, $CDCl_3$): δ 9.21 (br. s, 1H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 9.0 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.09-7.11 (m, 2H), 7.07 (dd, J_1 = 9.0 Hz, J_2 = 2.4 Hz, 1H), 3.87 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 186.6, 154.8, 143.0, 135.3, 134.9, 132.8, 129.3, 129.1, 128.1, 118.2, 113.0, 111.8, 102.7, 55.7, 21.7;

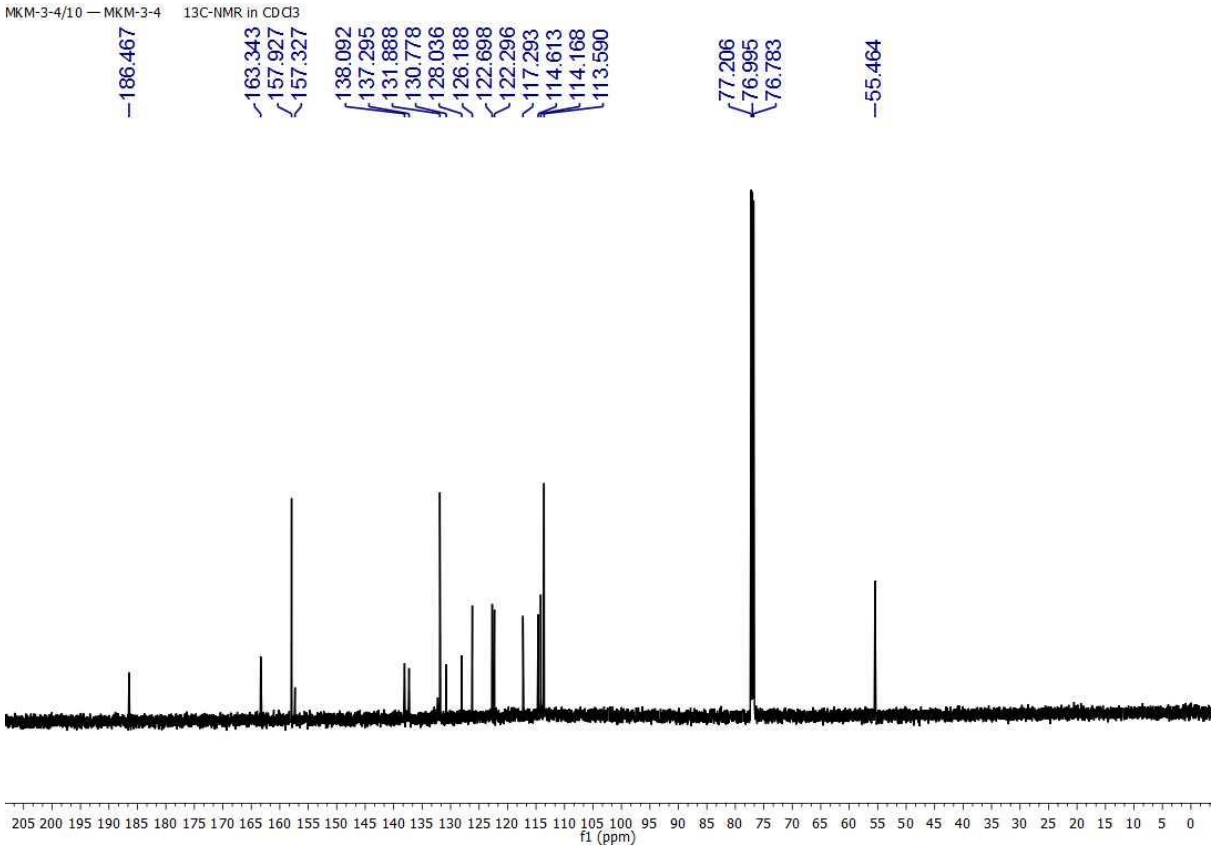
References:

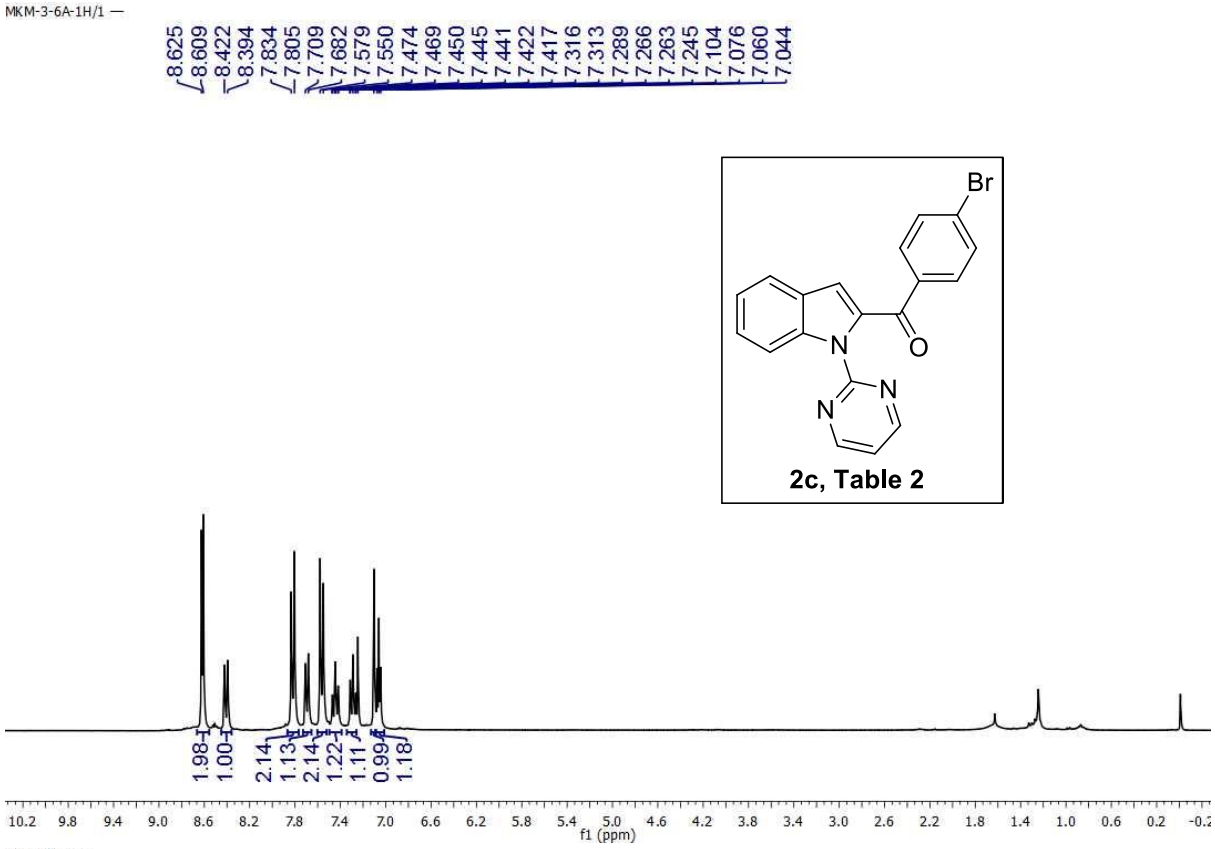
1) L. Ackermann and A. V. Lygin, *Org. Lett.*, 2011, **13**, 3332.

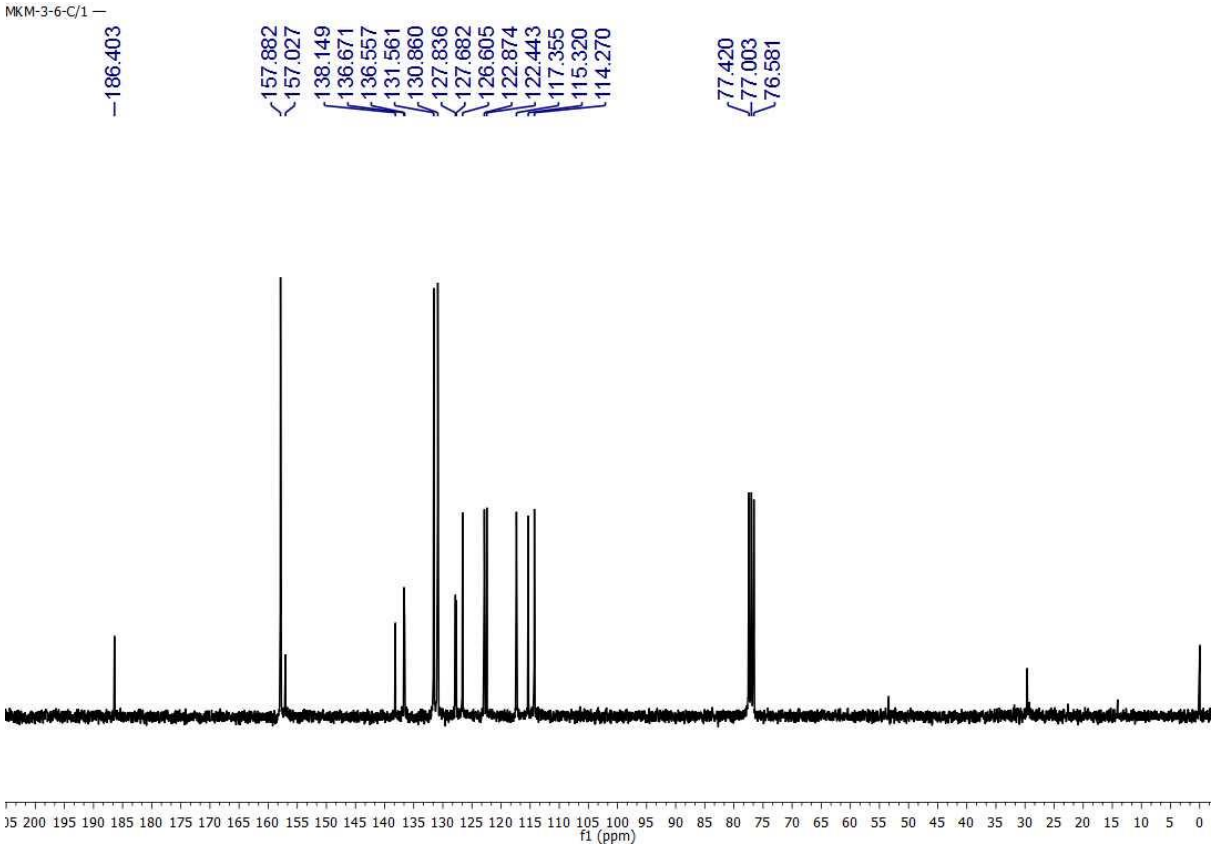

MKM-192-1H/1
8.651
8.635
8.412
8.384
7.907
7.880
7.716
7.689
7.465
7.441
7.413
7.317
7.314
7.291
7.264
7.260
7.236
7.102
7.083
7.067
7.051
2.420
Me
O
N
N
N
2a, Table 2
1.92
0.96
1.94
1.00
1.13
3.46
1.97
3.01
f1 (ppm)

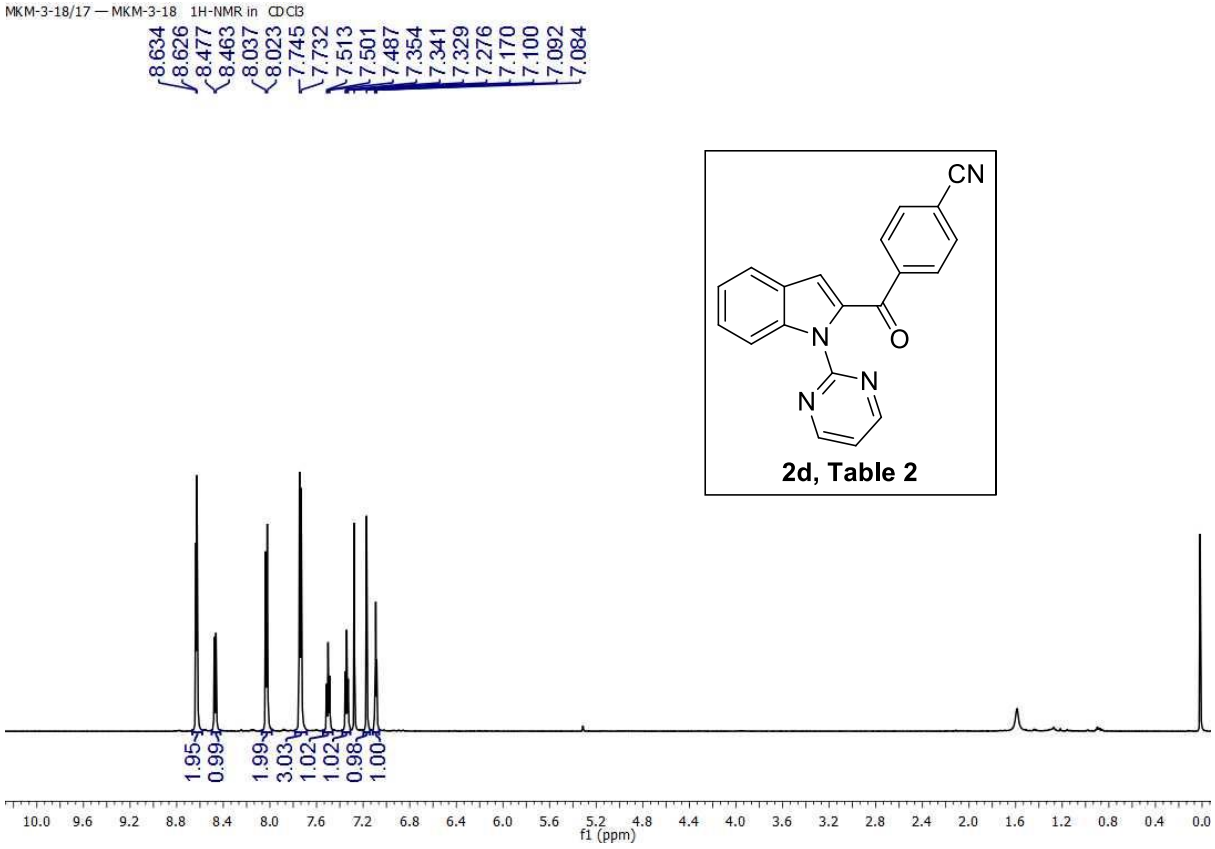

MKM-192-C/1
187.367
157.961
157.318
143.557
138.196
137.314
135.335
129.758
129.069
128.025
126.353
122.762
122.420
117.362
115.037
114.202
77.493
77.070
76.646
21.704
f1 (ppm)

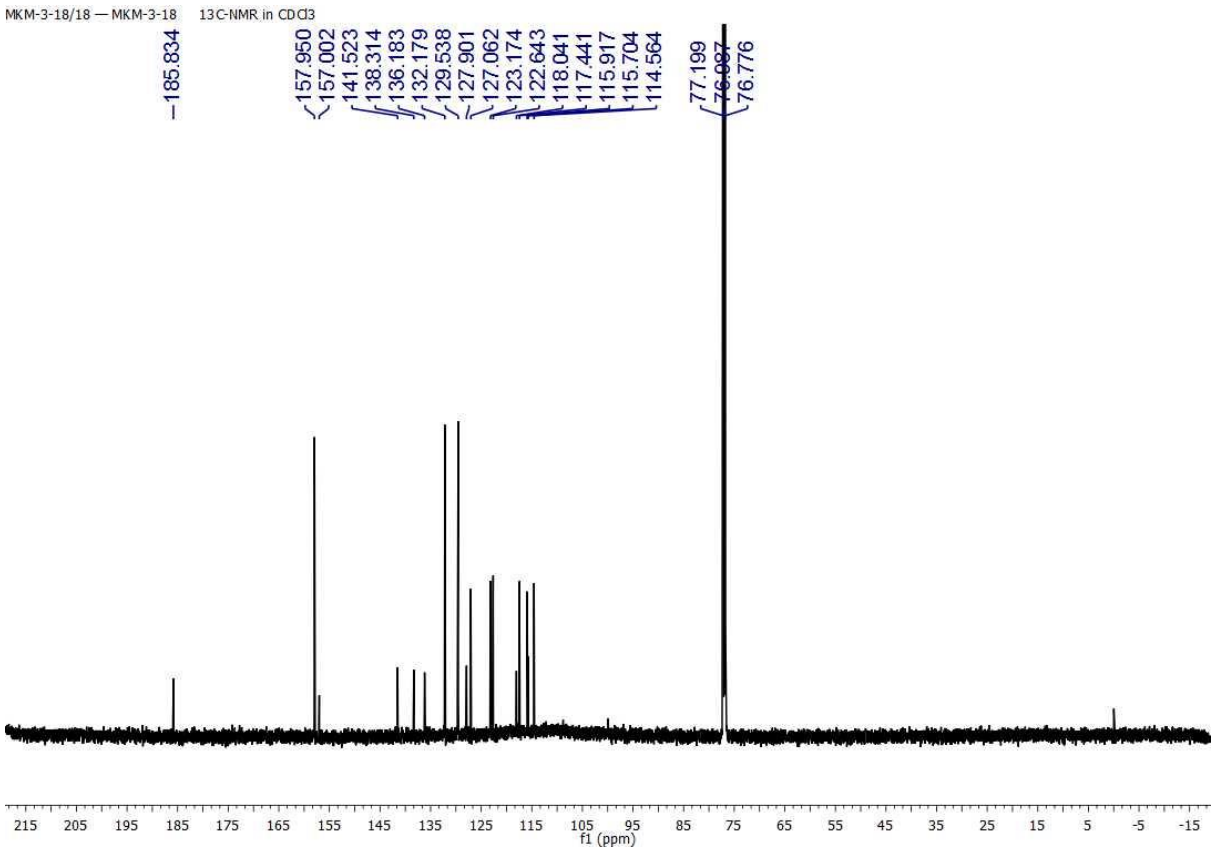

MKM-3-4/9 — MKM-3-4 1H-NMR in CDCl3
8.667
8.659
8.425
8.411
8.013
7.999
7.722
7.709
7.463
7.450
7.437
7.318
7.305
7.293
7.276
7.099
7.091
7.083
7.075
6.961
6.947
3.887
OMe
N
O
N
N
2b, Table 2
1.98
1.00
2.02
1.05
1.04
1.01
2.06
2.30
3.34
f1 (ppm)

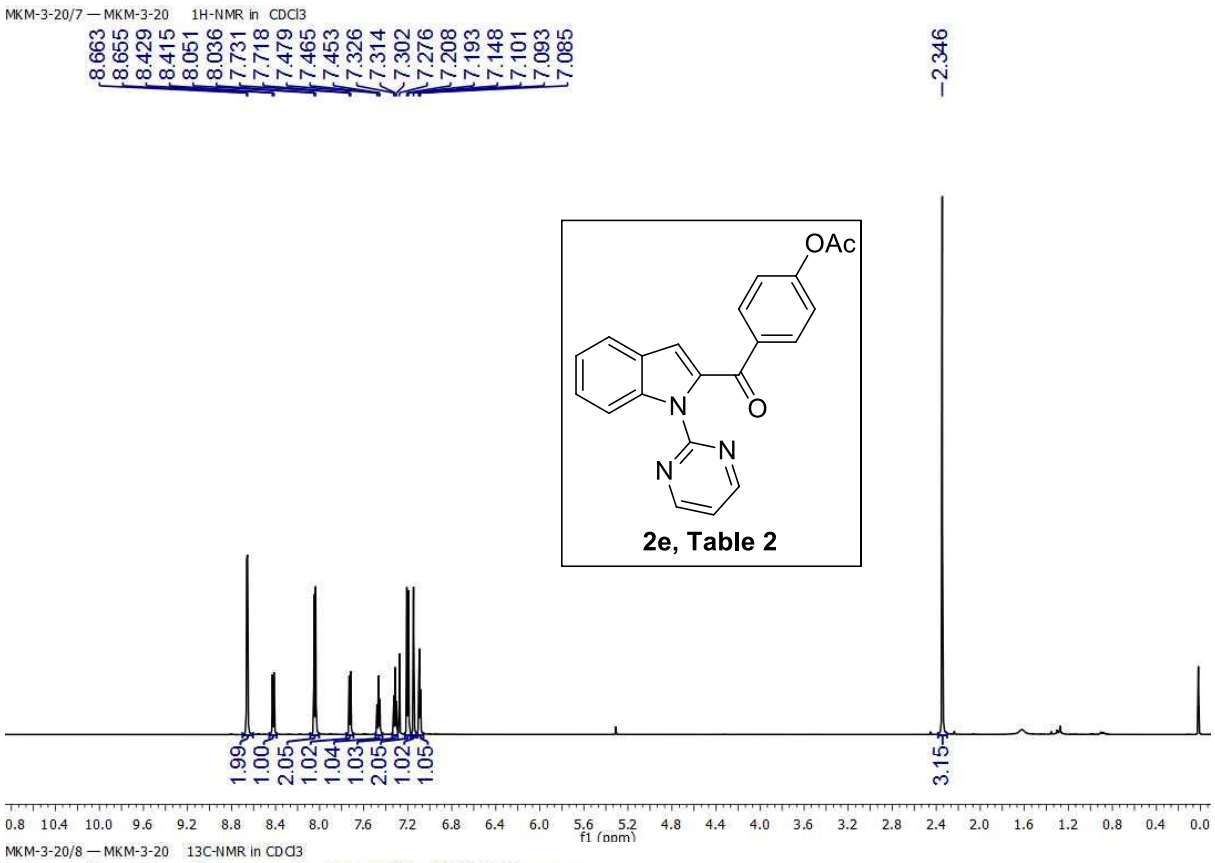

MKM-3-4/10 — MKM-3-4 13C-NMR in CDCl3
186.467
163.343
157.927
157.327
138.092
137.295
131.888
130.778
128.036
126.188
122.698
122.296
117.293
114.613
114.168
113.590
77.206
76.995
76.783
55.464
f1 (ppm)

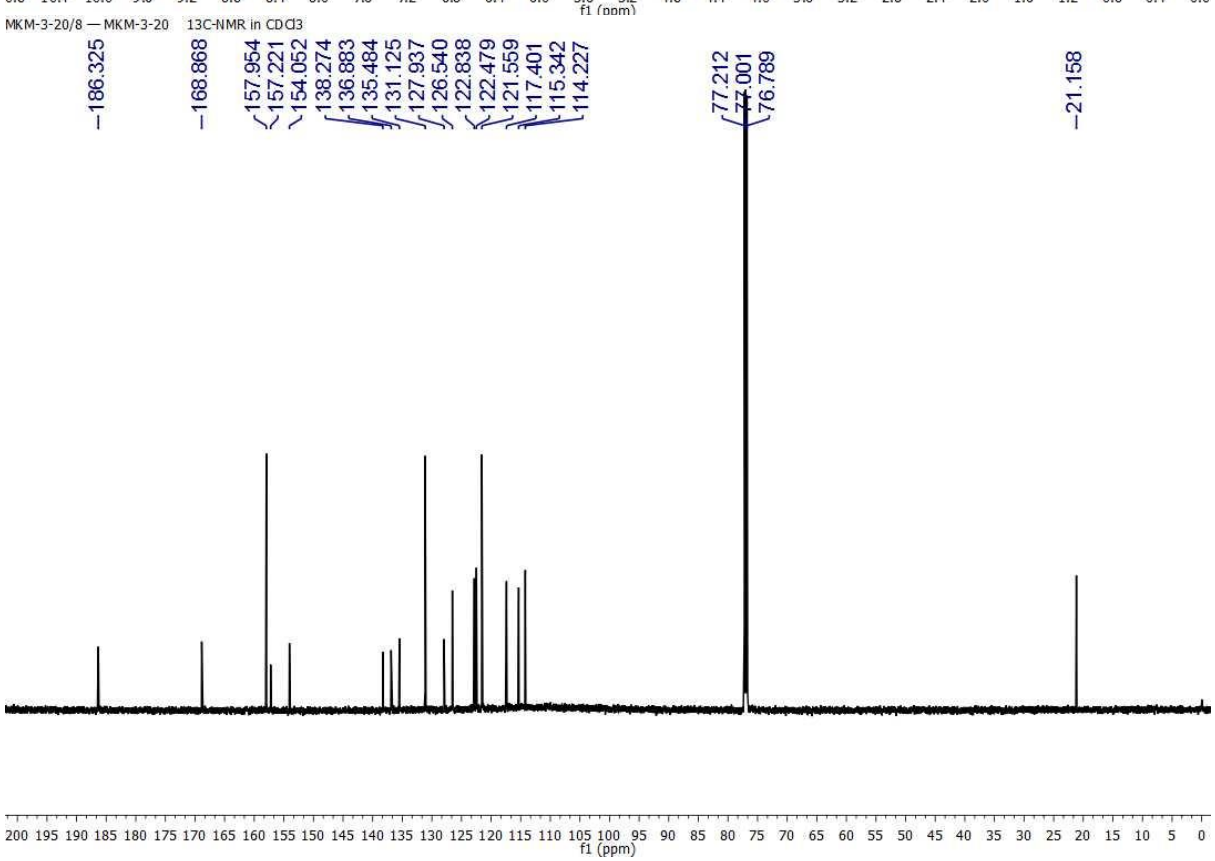

MKM-3-6A-1H/1
8.625
8.609
8.422
8.394
7.834
7.805
7.709
7.682
7.579
7.550
7.474
7.469
7.450
7.445
7.441
7.422
7.417
7.316
7.313
7.289
7.266
7.263
7.245
7.104
7.076
7.060
7.044
Br
O
N
N
N
2c, Table 2
1.98
1.00
2.14
1.13
2.14
1.22
1.11
0.99
1.18
f1 (ppm)

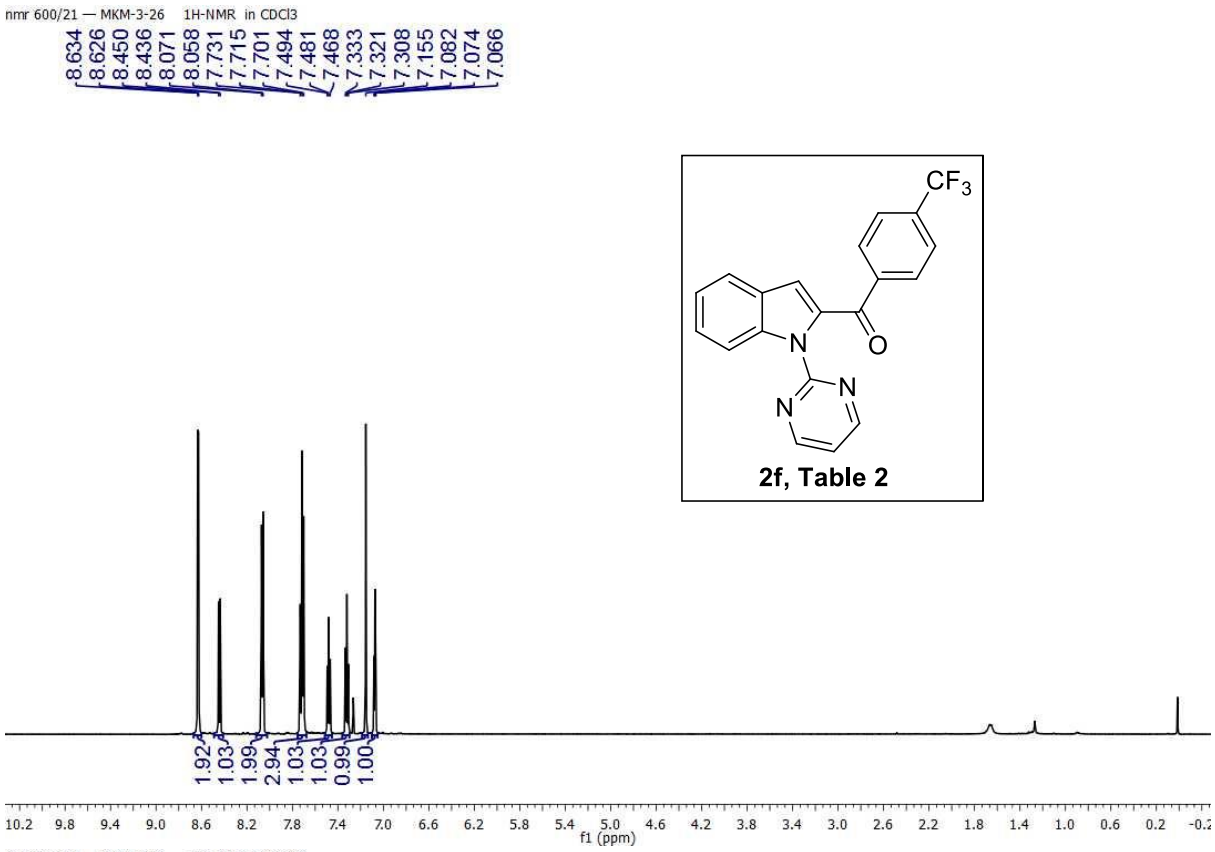

MKM-3-6-C/1
186.403
157.882
157.027
138.149
136.671
136.557
131.561
130.860
127.836
127.682
126.605
122.874
122.443
117.355
115.320
114.270
77.420
77.003
76.581
f1 (ppm)


MKM-3-18/17 — MKM-3-18 1H-NMR in CDCl3
8.634
8.626
8.477
8.463
8.037
8.023
7.745
7.732
7.513
7.501
7.487
7.354
7.341
7.329
7.276
7.170
7.100
7.092
7.084
CN
O
N
N
N
2d, Table 2
1.95
0.99
1.99
3.03
1.02
1.02
0.98
1.00
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)


MKM-3-18/18 — MKM-3-18 13C-NMR in CDCl3
185.834
157.950
157.002
141.523
138.314
136.183
132.179
129.538
127.901
127.062
123.174
122.643
118.041
117.441
115.917
115.704
114.564
77.199
76.987
76.776
215 205 195 185 175 165 155 145 135 125 115 105 95 85 75 65 55 45 35 25 15 5 -5 -15
f1 (ppm)

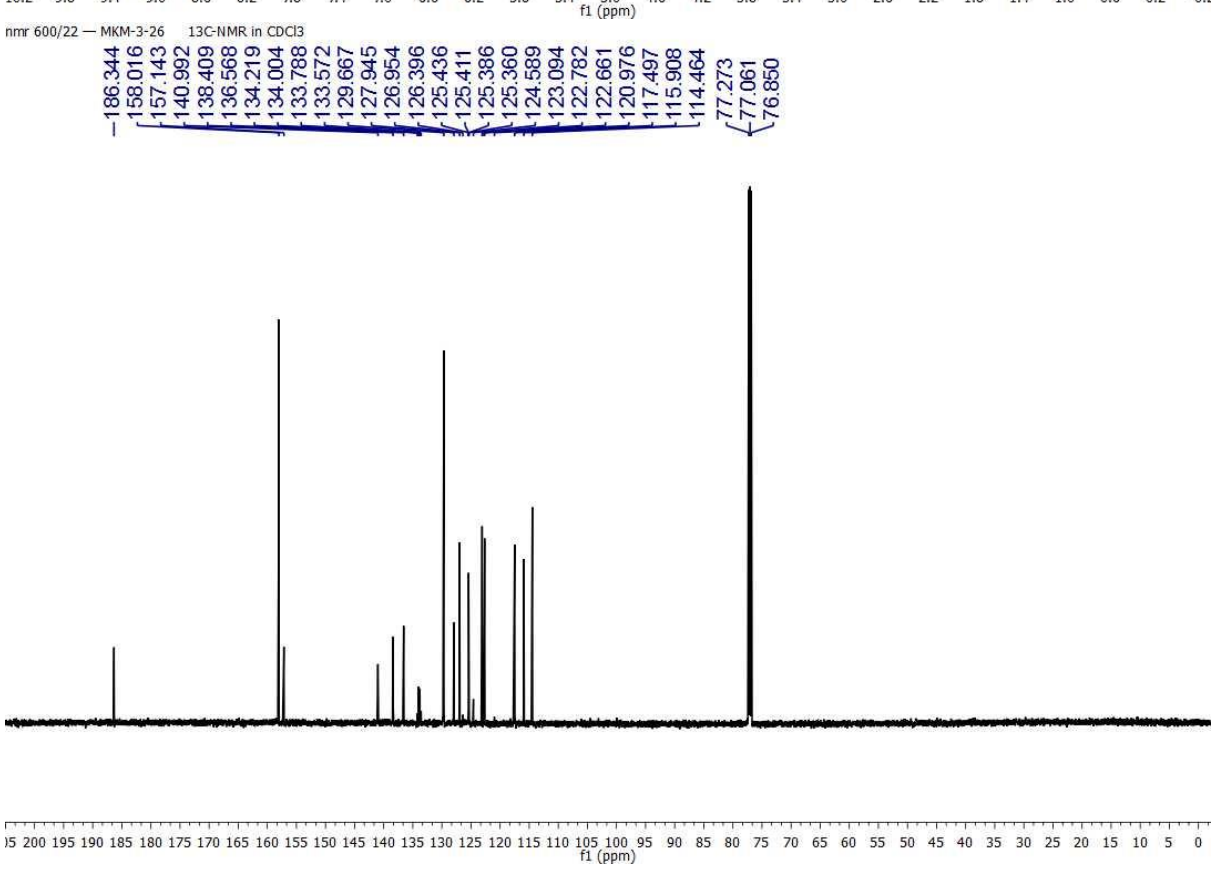

MKM-3-20/7 — MKM-3-20 1H-NMR in CDCl3
8.663
8.655
8.429
8.415
8.051
8.036
7.731
7.718
7.479
7.465
7.453
7.326
7.314
7.302
7.276
7.208
7.193
7.148
7.101
7.093
7.085
2.346
OAc
O
N
N
N
2e, Table 2
1.99
1.00
2.05
1.02
1.04
1.03
2.05
1.02
1.05
3.15
f1 (ppm)

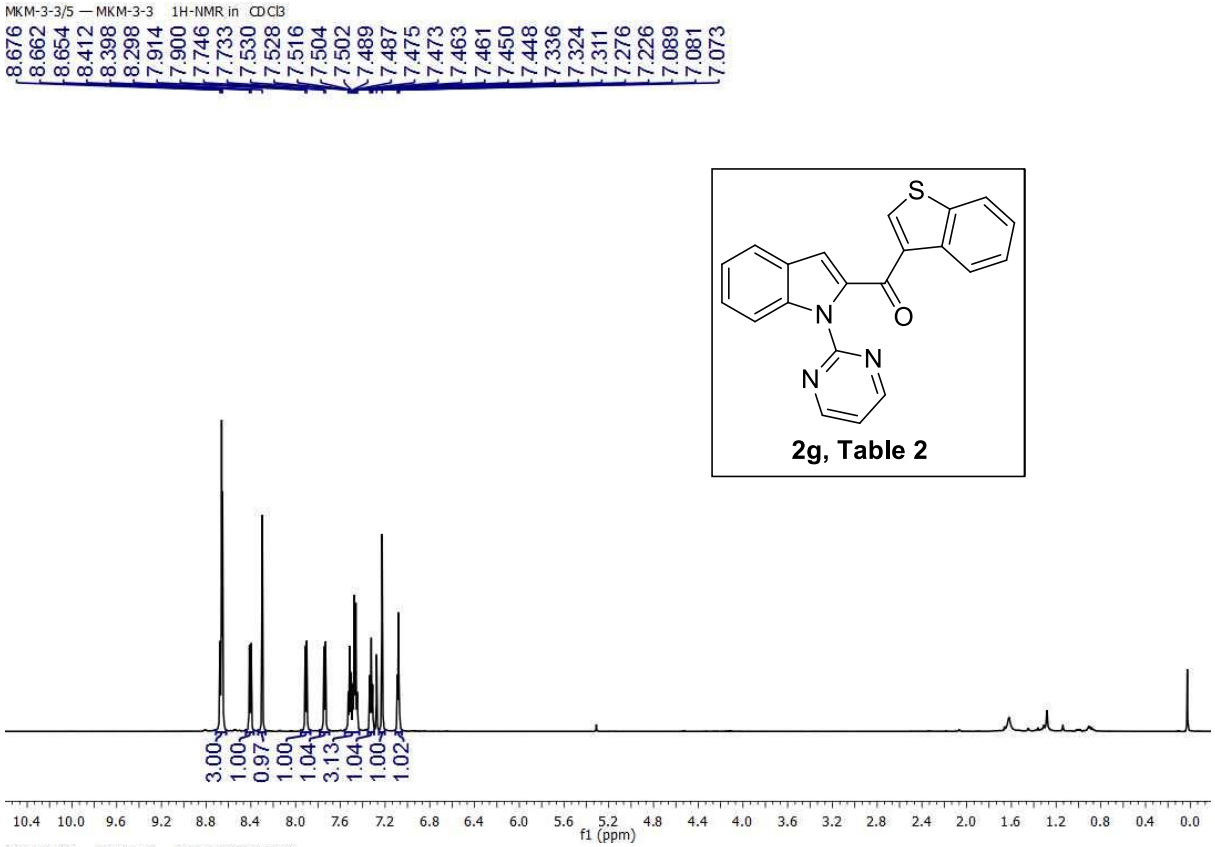

MKM-3-20/8 — MKM-3-20 13C-NMR in CDCl3
186.325
168.868
157.954
157.221
154.052
138.274
136.883
135.484
131.125
127.937
126.540
122.838
122.479
121.559
117.401
115.342
114.227
77.212
77.001
76.789
21.158
f1 (ppm)

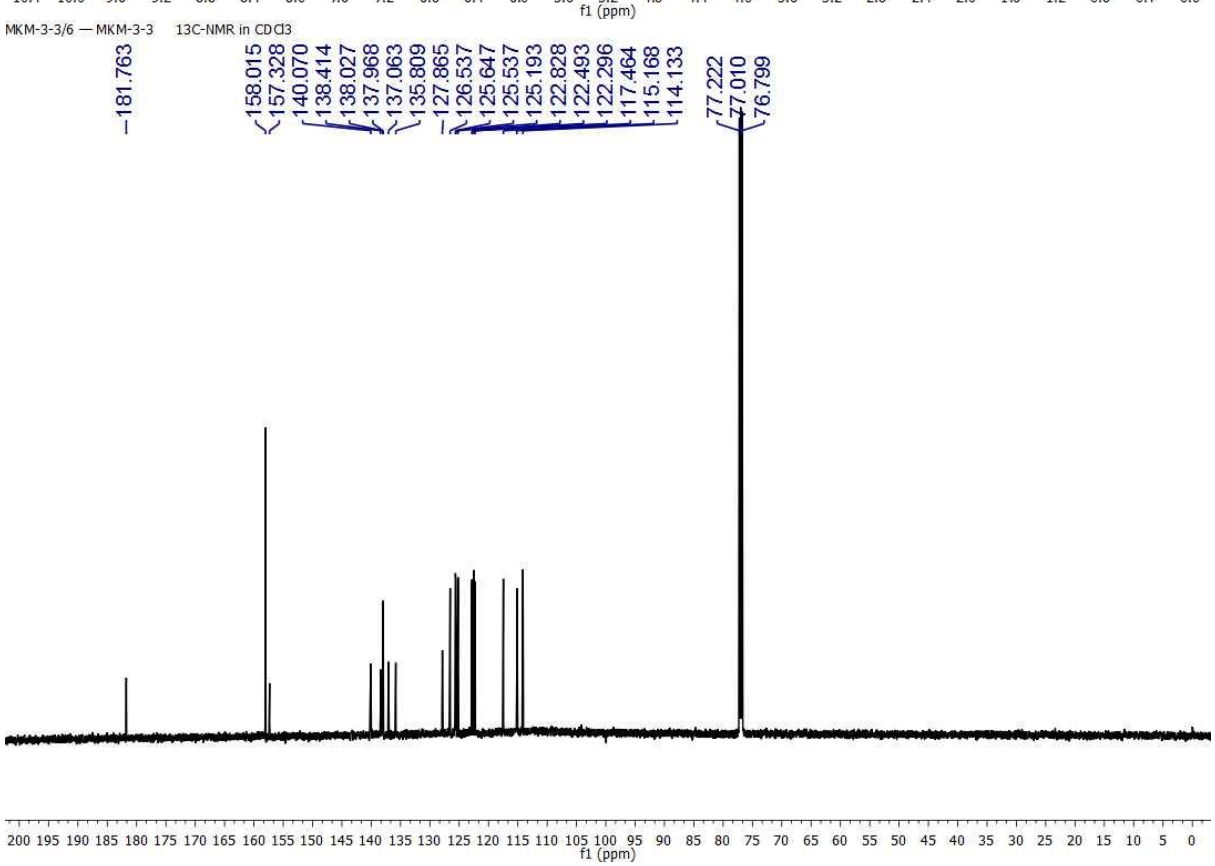

nmr 600/21 — MKM-3-26 1H-NMR in CDCl3
8.634
8.626
8.450
8.436
8.071
8.058
7.731
7.715
7.701
7.494
7.481
7.468
7.333
7.321
7.308
7.155
7.082
7.074
7.066
1.92
1.03
1.99
2.94
1.03
1.03
0.99
1.00
f1 (ppm)

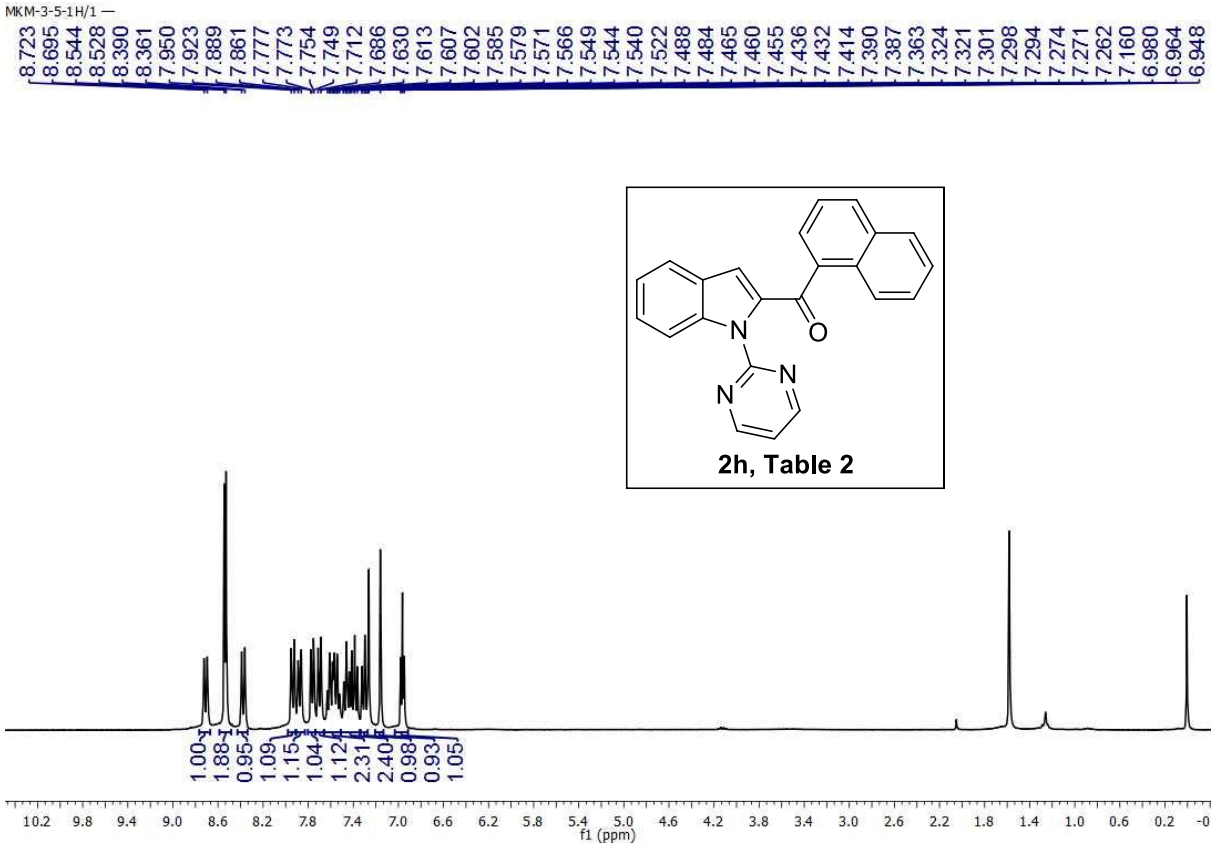
CF3
O
N
N
N

2f, Table 2


nmr 600/22 — MKM-3-26 13C-NMR in CDCl3
186.344
158.016
157.143
140.992
138.409
136.568
134.219
134.004
133.788
133.572
129.667
127.945
126.954
126.396
125.436
125.411
125.386
125.360
124.589
123.094
122.782
122.661
120.976
117.497
115.908
114.464
77.273
77.061
76.850
f1 (ppm)

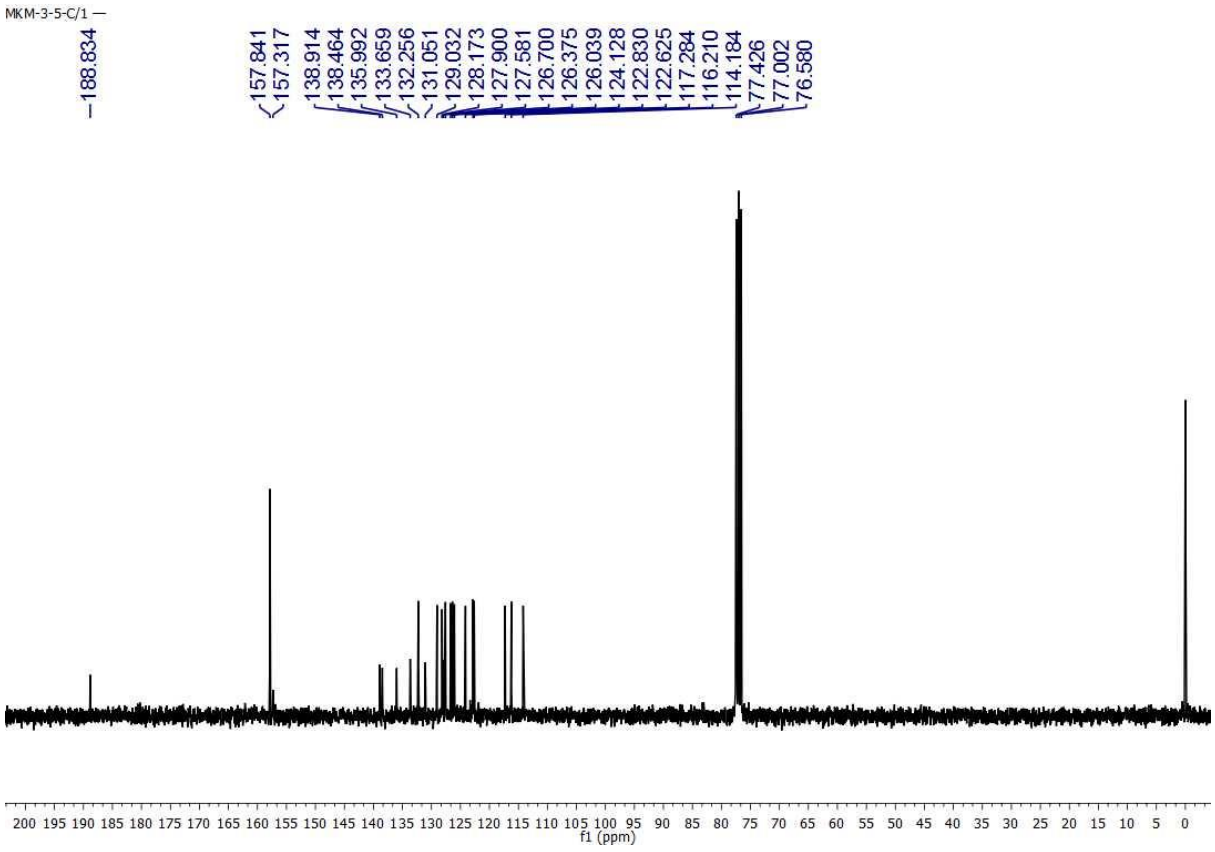

MKM-3-3/5 — MKM-3-3 1H-NMR in CDCl3
8.676
8.662
8.654
8.412
8.398
8.298
7.914
7.900
7.746
7.733
7.530
7.528
7.516
7.504
7.502
7.489
7.487
7.475
7.473
7.463
7.461
7.450
7.448
7.336
7.324
7.311
7.276
7.226
7.089
7.081
7.073
S
O
N
N
N
2g, Table 2
3.00
1.00
0.97
1.00
1.04
3.13
1.04
1.00
1.02
f1 (ppm)

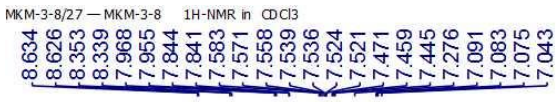

MKM-3-3/6 — MKM-3-3 13C-NMR in CDCl3
181.763
158.015
157.328
140.070
138.414
138.027
137.968
137.063
135.809
127.865
126.537
125.647
125.537
125.193
122.828
122.493
122.296
117.464
115.168
114.133
77.222
77.010
76.799
f1 (ppm)

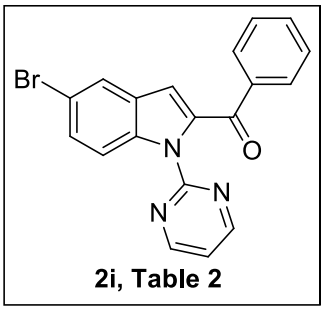

MKM-3-5-1H/1
8.723
8.695
8.544
8.528
8.390
8.361
7.950
7.923
7.889
7.861
7.777
7.773
7.754
7.749
7.712
7.686
7.630
7.613
7.607
7.602
7.585
7.579
7.571
7.566
7.549
7.544
7.540
7.522
7.488
7.484
7.465
7.460
7.455
7.436
7.432
7.414
7.390
7.387
7.363
7.324
7.321
7.301
7.298
7.294
7.274
7.271
7.262
7.160
6.980
6.964
6.948
1.00
1.88
0.95
1.09
1.15
1.04
1.12
2.31
2.40
0.98
0.93
1.05
f1 (ppm)

N
O
N
N

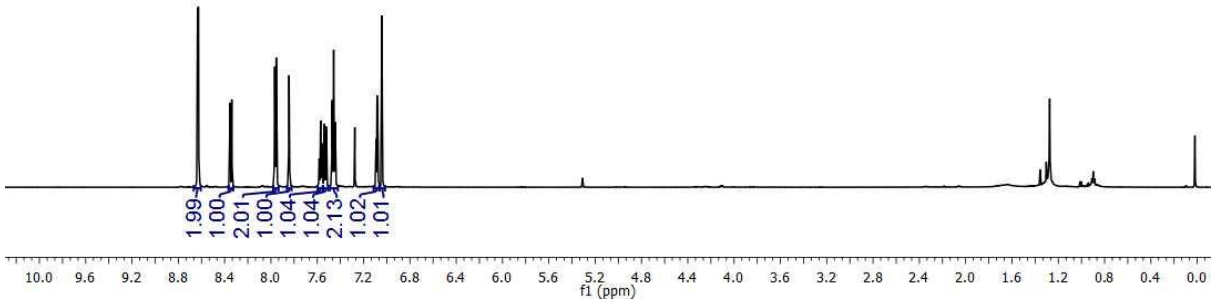
2h, Table 2

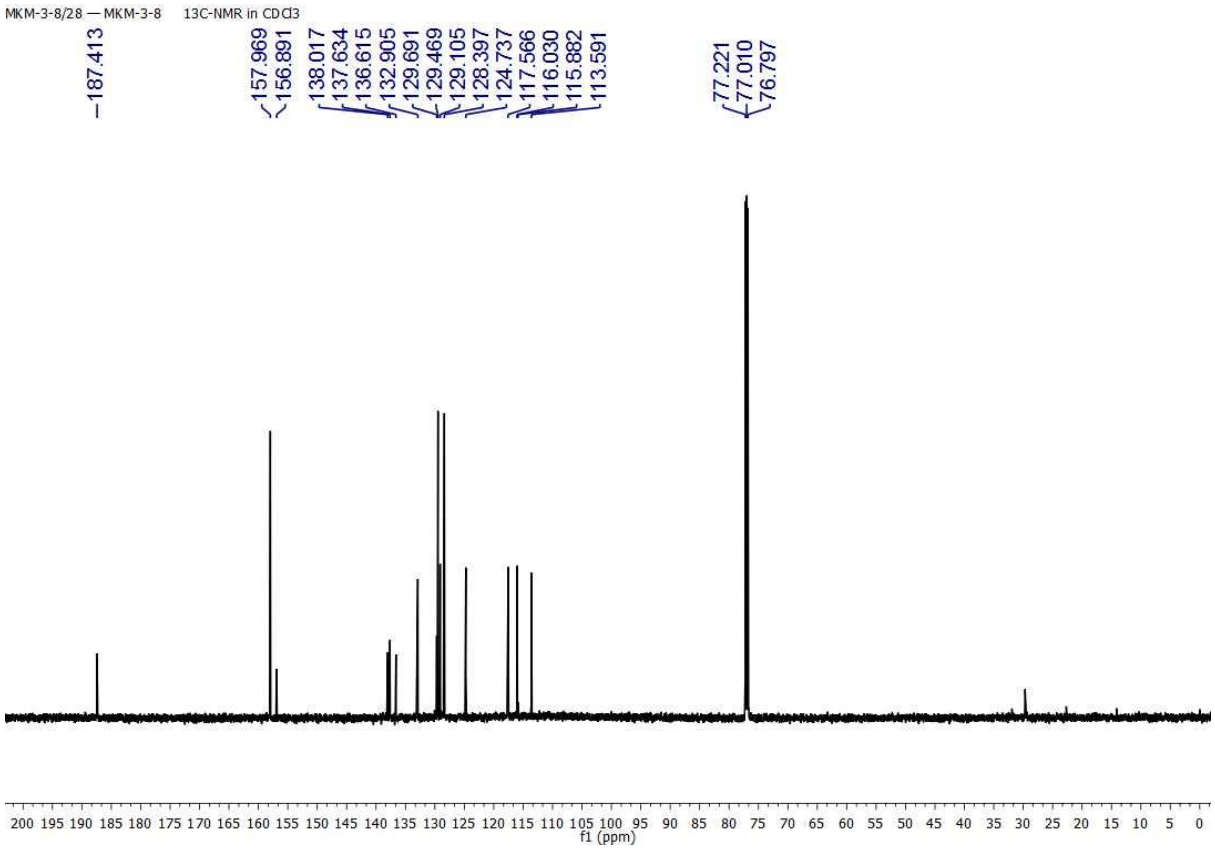

MKM-3-5-C/1
188.834
157.841
157.317
138.914
138.464
135.992
133.659
132.256
131.051
129.032
128.173
127.900
127.581
126.700
126.375
126.039
124.128
122.830
122.625
117.284
116.210
114.184
77.426
77.002
76.580
f1 (ppm)

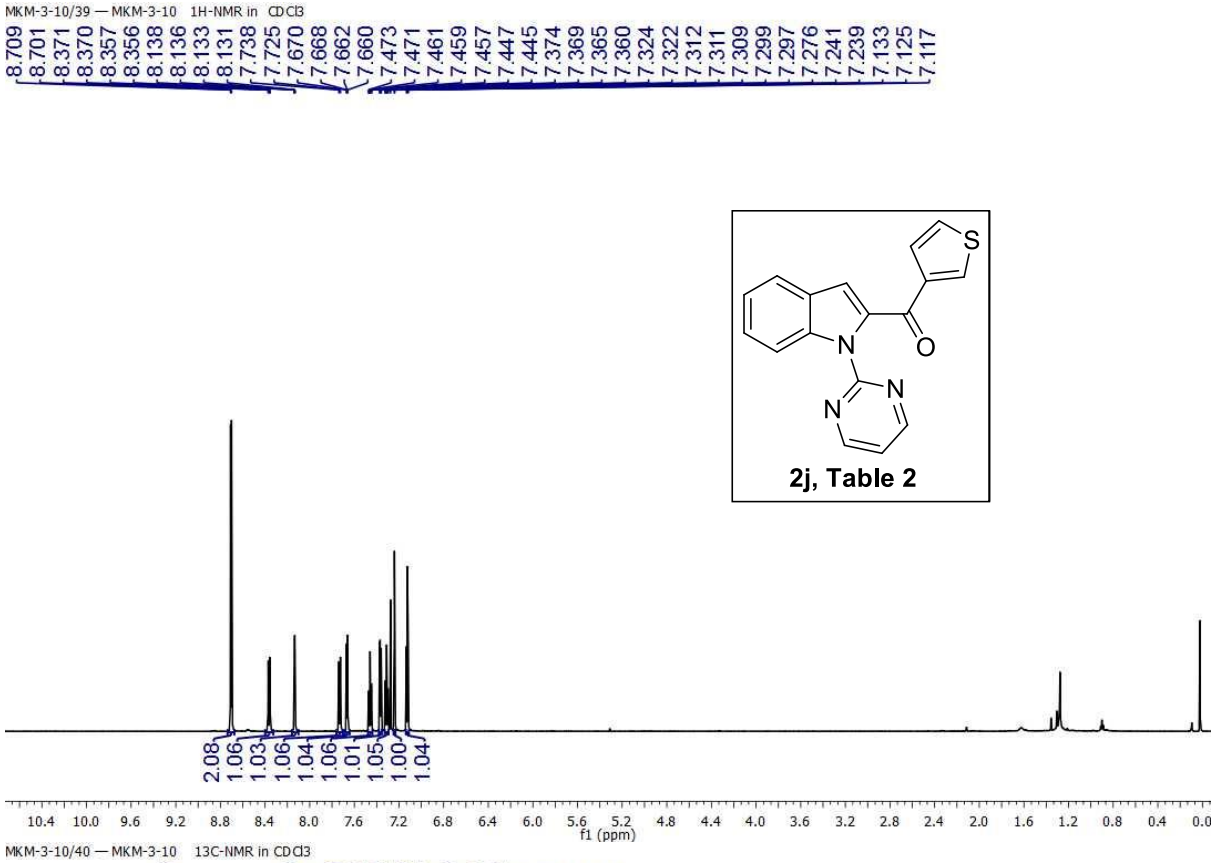

MKM-3-8/27 — MKM-3-8 1H-NMR in CDCl3
8.634
8.626
8.353
8.339
7.968
7.955
7.844
7.841
7.583
7.571
7.558
7.539
7.536
7.524
7.521
7.471
7.459
7.445
7.276
7.091
7.083
7.075
7.043

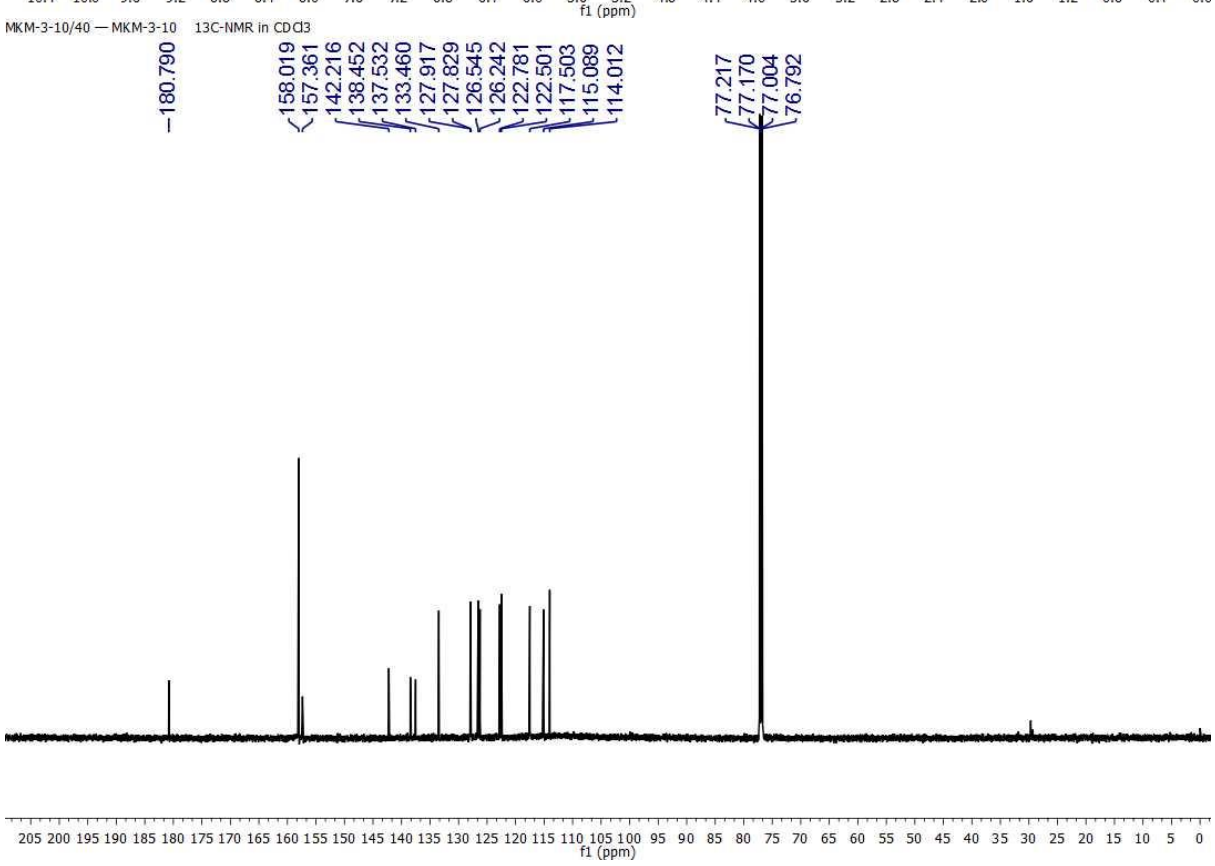

Br
N
O
N
N

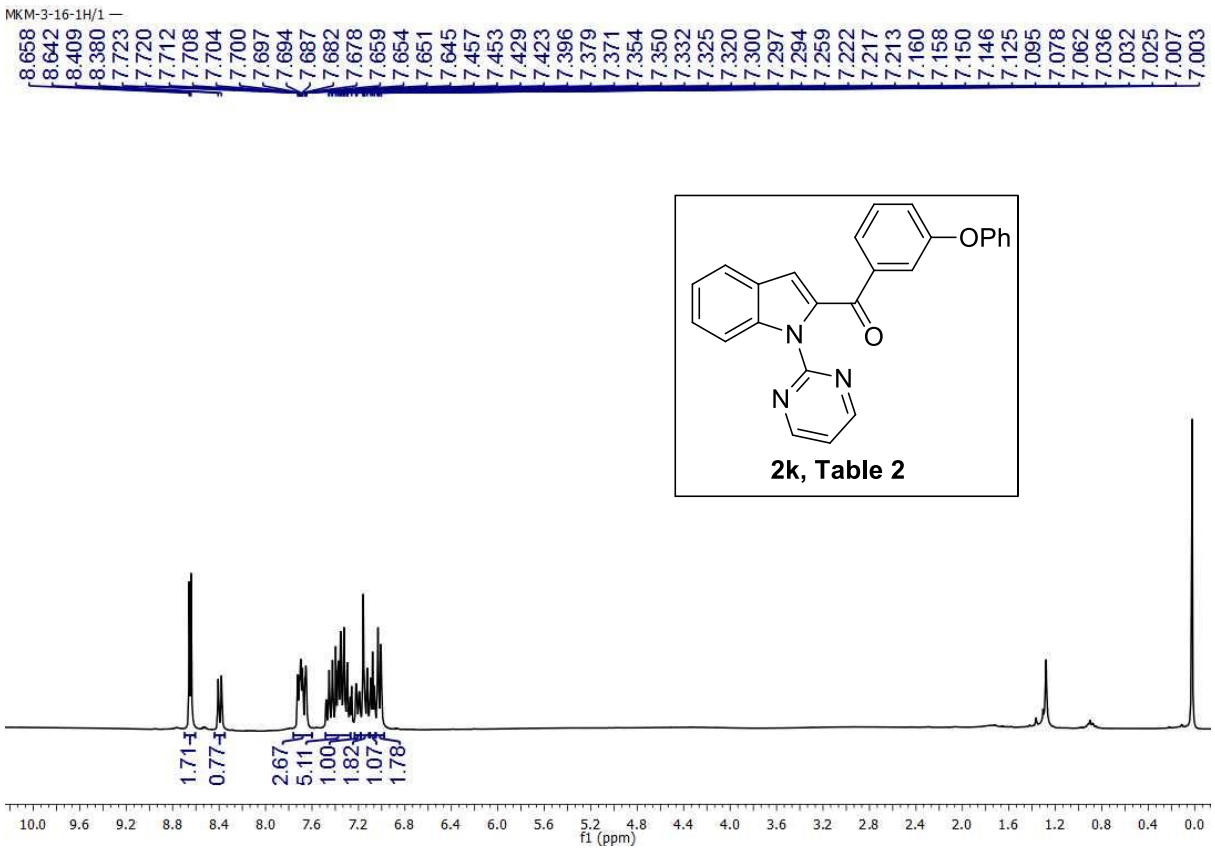
2i, Table 2

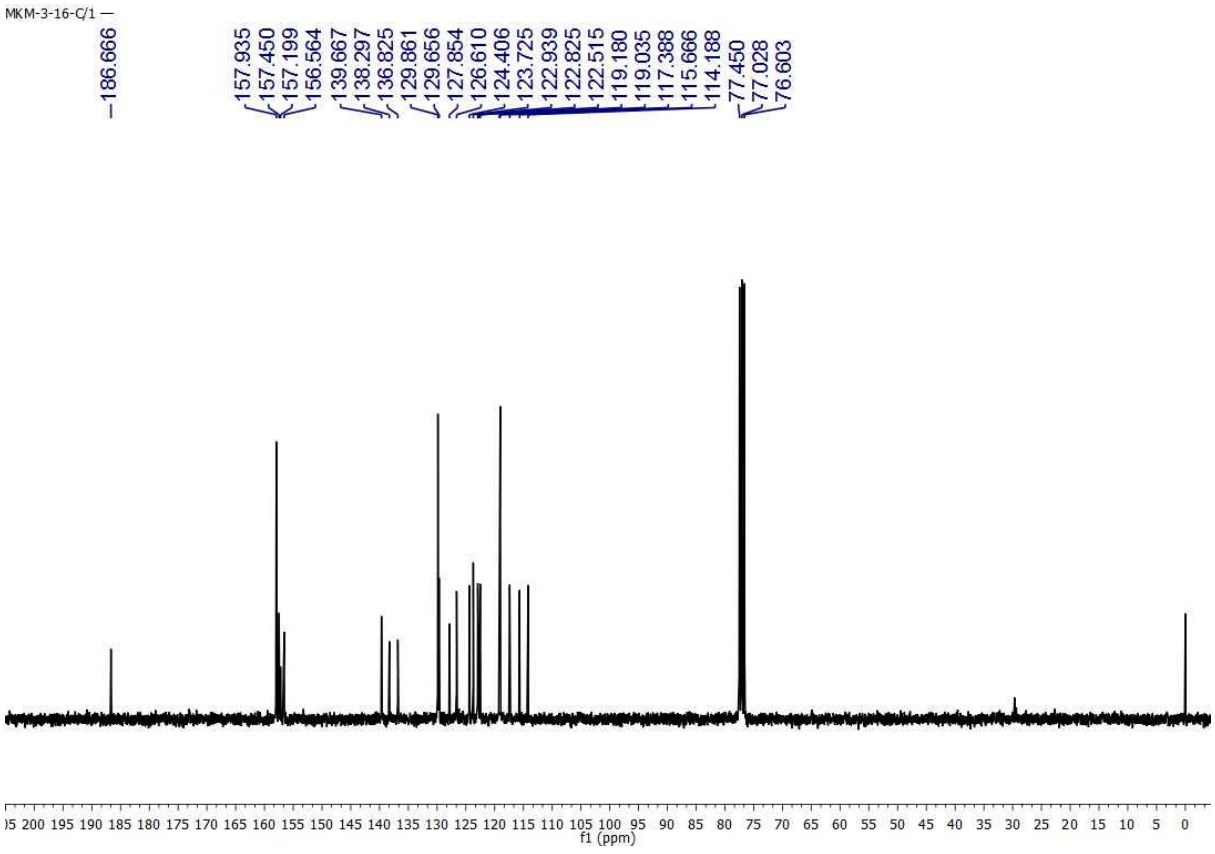

1.99
1.00
2.01
1.00
1.04
1.04
2.13
1.02
1.01
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)

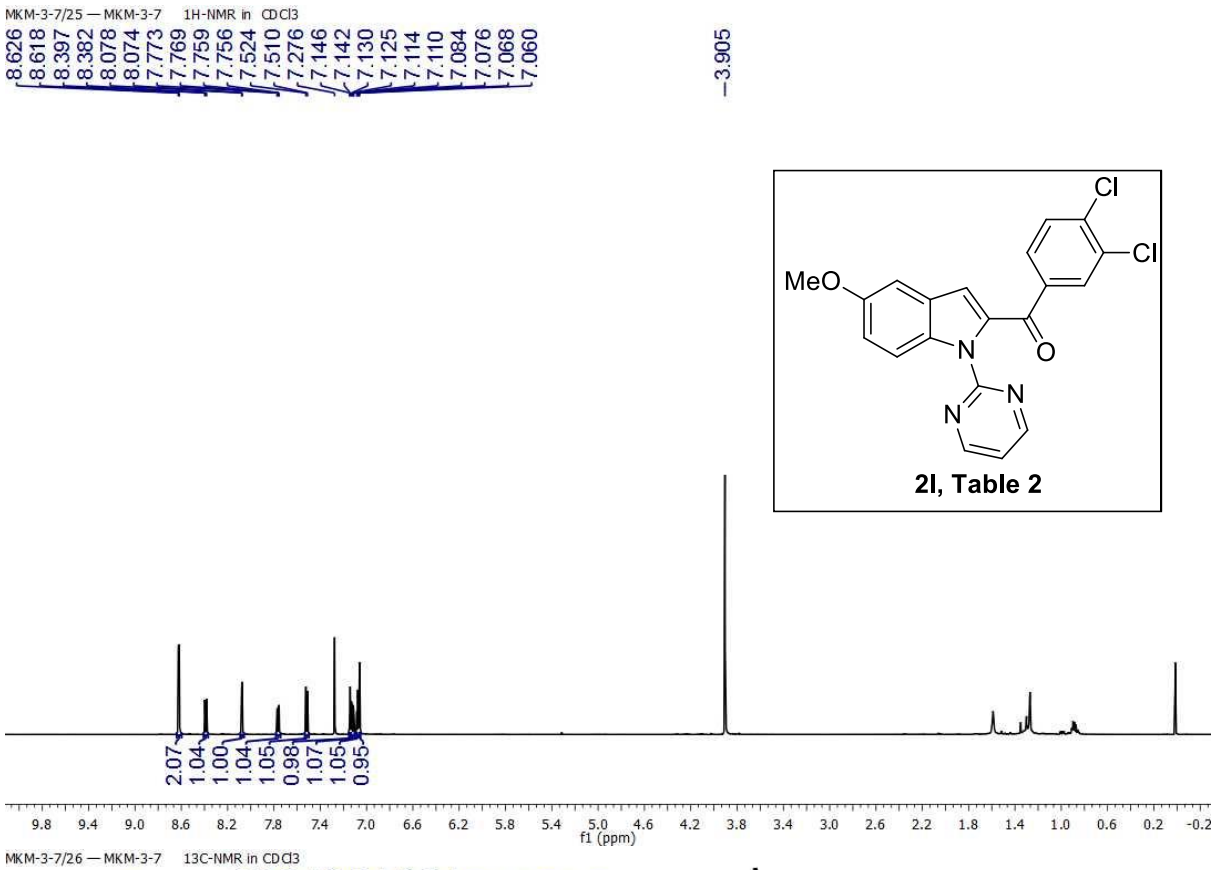

MKM-3-8/28 — MKM-3-8 13C-NMR in CDCl3
187.413
157.969
156.891
138.017
137.634
136.615
132.905
129.691
129.469
129.105
128.397
124.737
117.566
116.030
115.882
113.591
77.221
77.010
76.797
200 195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

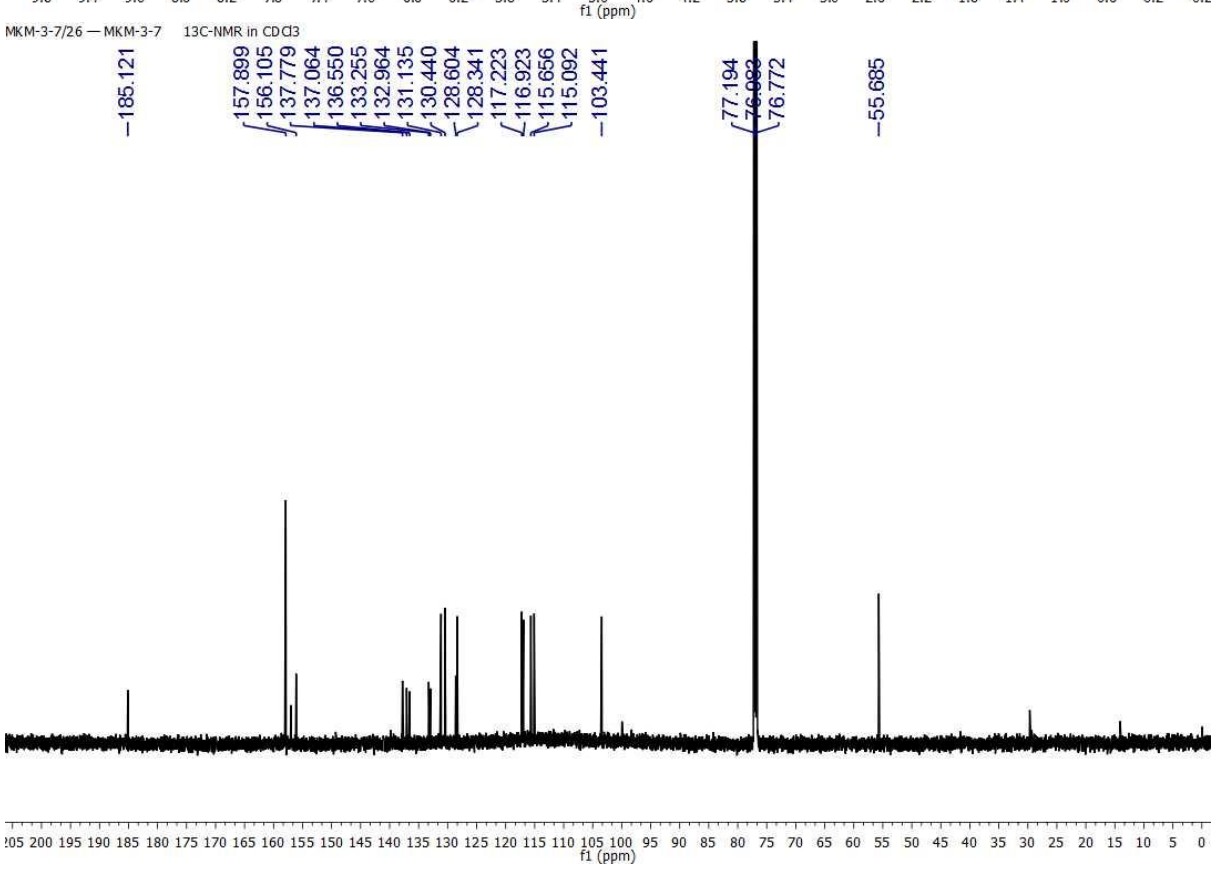

MKM-3-10/39 — MKM-3-10 1H-NMR in CDCl3
8.709
8.701
8.371
8.370
8.357
8.356
8.138
8.136
8.133
8.131
7.738
7.725
7.670
7.668
7.662
7.660
7.473
7.471
7.461
7.459
7.457
7.447
7.445
7.374
7.369
7.365
7.360
7.324
7.322
7.312
7.311
7.309
7.299
7.297
7.276
7.241
7.239
7.133
7.125
7.117
S
O
N
N
N
2j, Table 2
2.08
1.06
1.03
1.06
1.04
1.06
1.01
1.05
1.00
1.04
f1 (ppm)

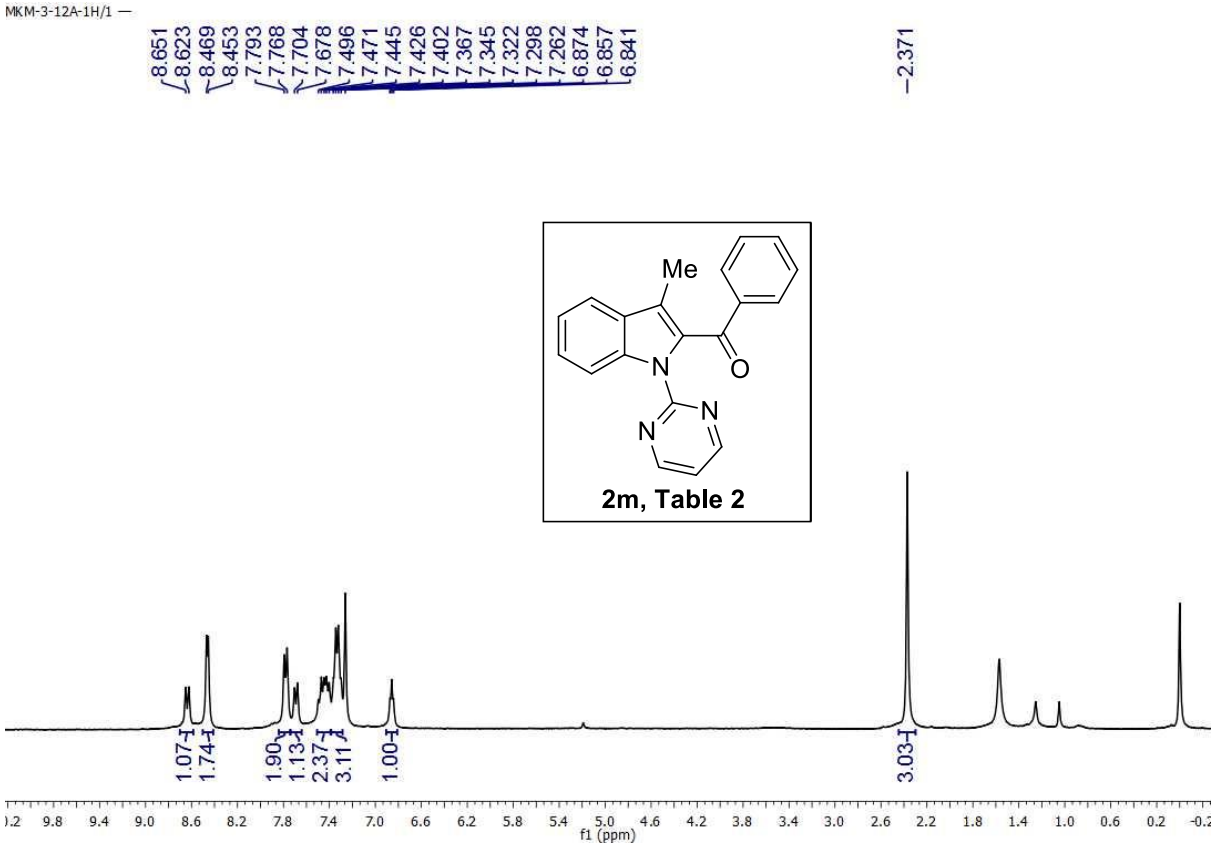

MKM-3-10/40 — MKM-3-10 13C-NMR in CDCl3
180.790
158.019
157.361
142.216
138.452
137.532
133.460
127.917
127.829
126.545
126.242
122.781
122.501
117.503
115.089
114.012
77.217
77.170
77.004
76.792
f1 (ppm)

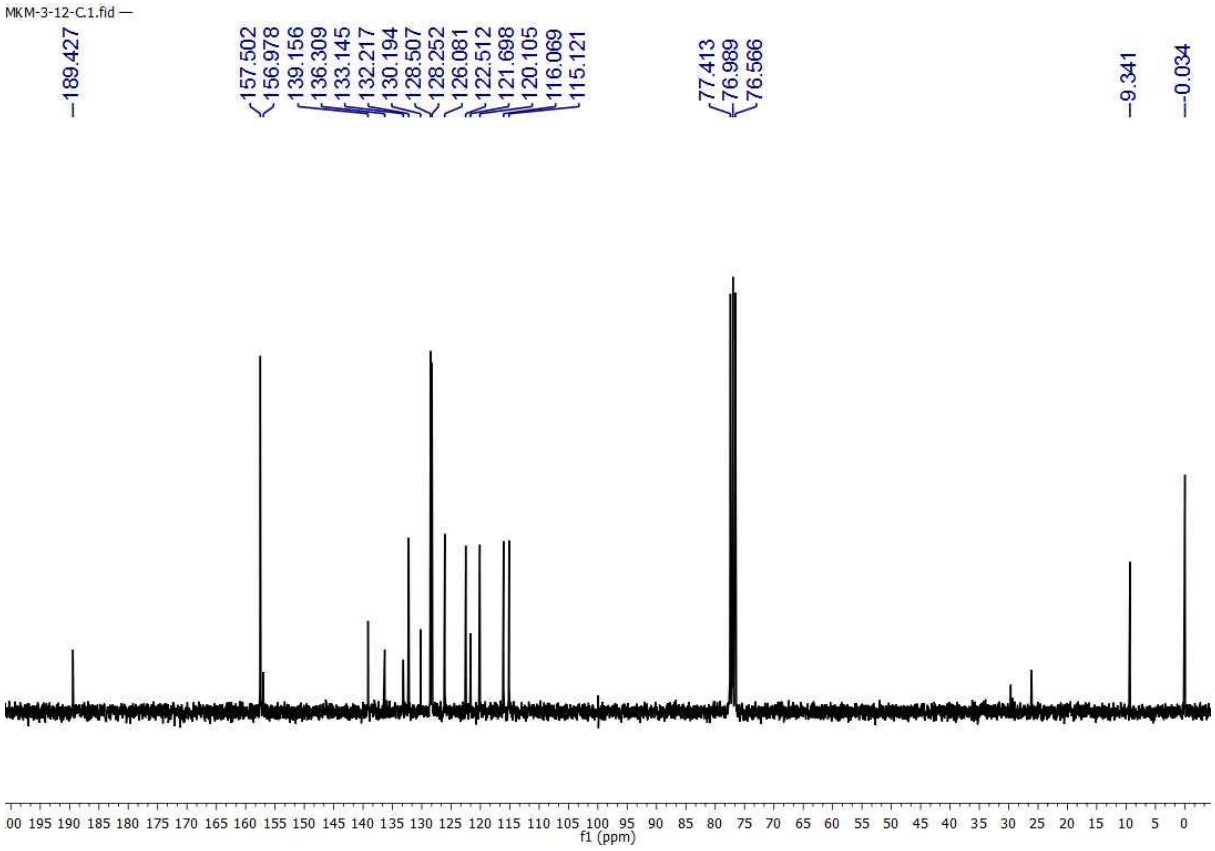

MKM-3-16-1H/1
8.658
8.642
8.409
8.380
7.723
7.720
7.712
7.708
7.704
7.700
7.697
7.694
7.687
7.682
7.678
7.659
7.654
7.651
7.645
7.457
7.453
7.429
7.423
7.396
7.379
7.371
7.354
7.350
7.332
7.325
7.320
7.300
7.297
7.294
7.259
7.222
7.217
7.213
7.160
7.158
7.150
7.146
7.125
7.095
7.078
7.062
7.036
7.032
7.025
7.007
7.003
OPh
O
N
N
N
2k, Table 2
1.71
0.77
2.67
5.11
1.00
1.82
1.07
1.78
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)

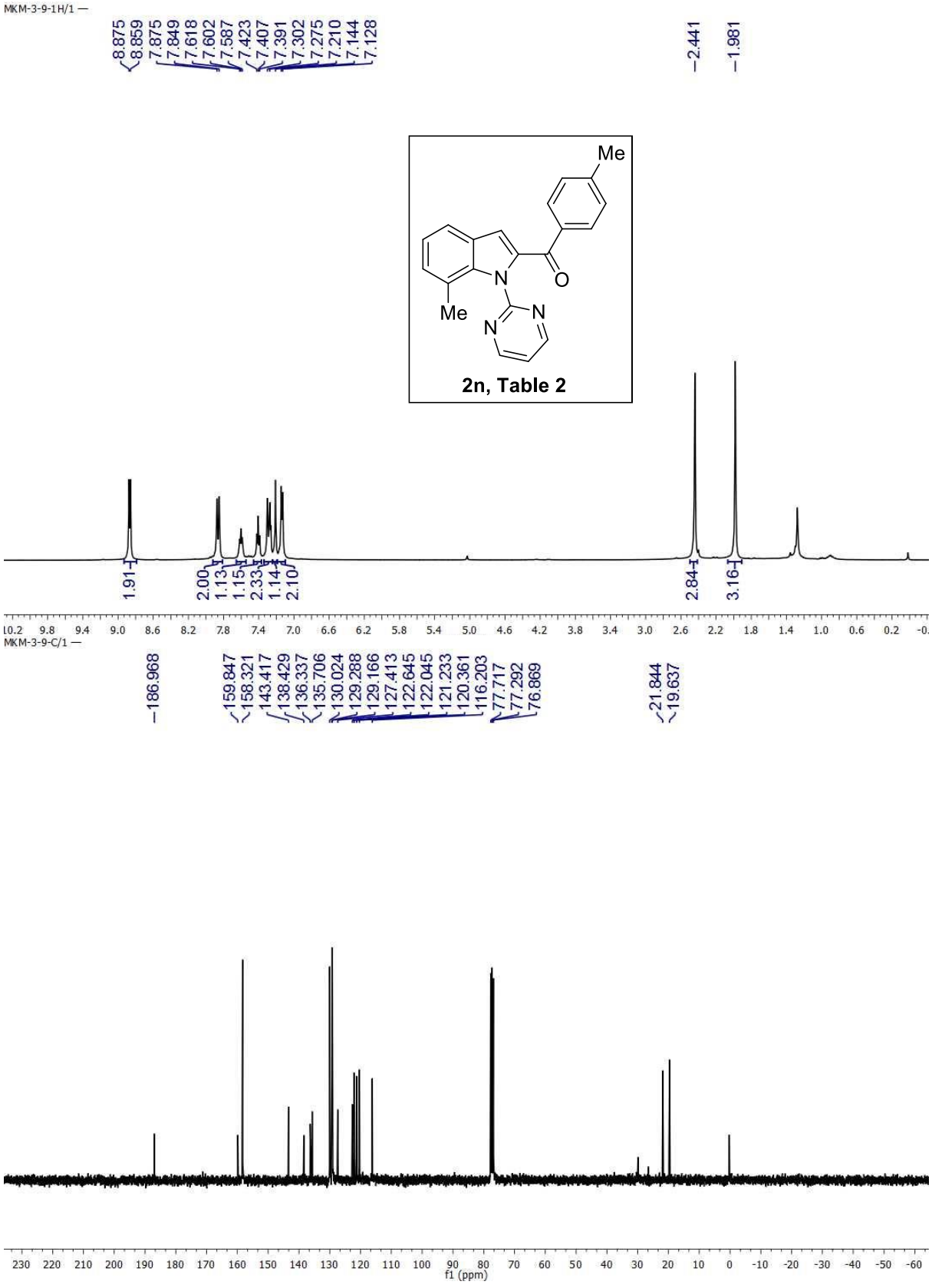

MKM-3-16-C/1
186.666
157.935
157.450
157.199
156.564
139.667
138.297
136.825
129.861
129.656
127.854
126.610
124.406
123.725
122.939
122.825
122.515
119.180
119.035
117.388
115.666
114.188
77.450
77.028
76.603
f1 (ppm)

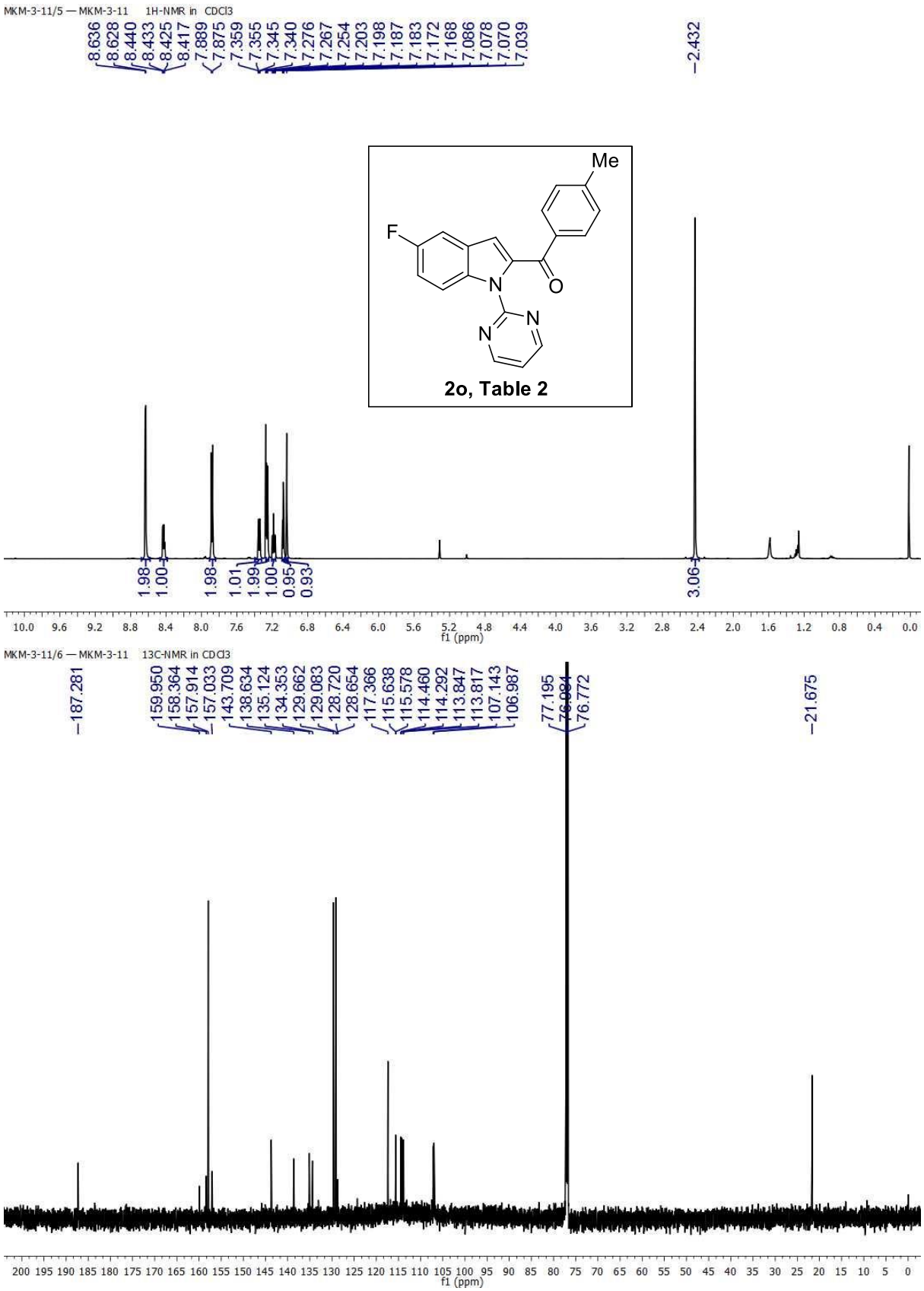

MKM-3-7/25 — MKM-3-7 1H-NMR in CDCl3
8.626
8.618
8.397
8.382
8.078
8.074
7.773
7.769
7.759
7.756
7.524
7.510
7.276
7.146
7.142
7.130
7.125
7.114
7.110
7.084
7.076
7.068
7.060
3.905
Cl
Cl
MeO
O
N
N
N
2I, Table 2
2.07
1.04
1.00
1.04
1.05
0.98
1.07
1.05
0.95
f1 (ppm)

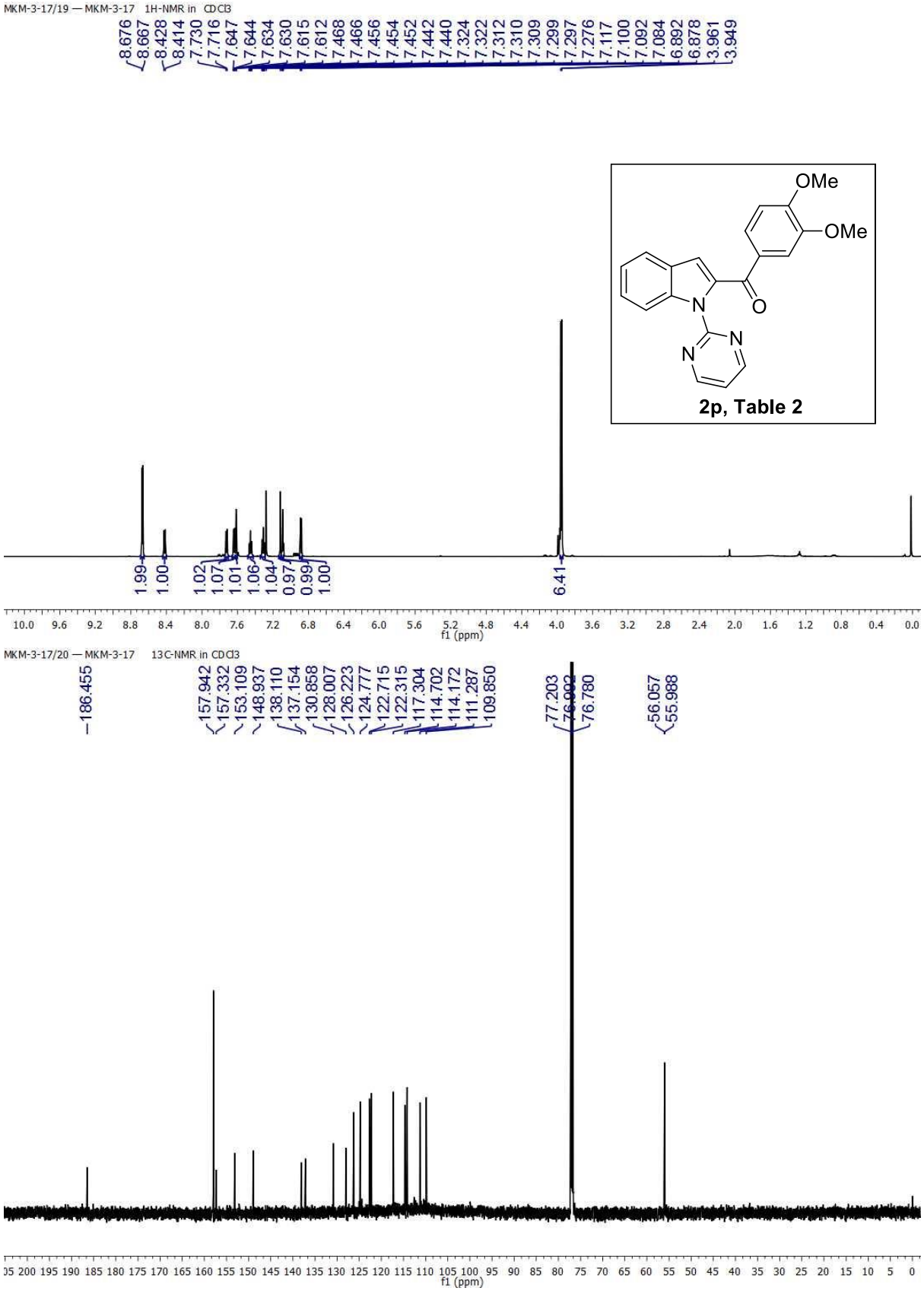

MKM-3-7/26 — MKM-3-7 13C-NMR in CDCl3
185.121
157.899
156.105
137.779
137.064
136.550
133.255
132.964
131.135
130.440
128.604
128.341
117.223
116.923
115.656
115.092
103.441
77.194
76.982
76.772
55.685
f1 (ppm)

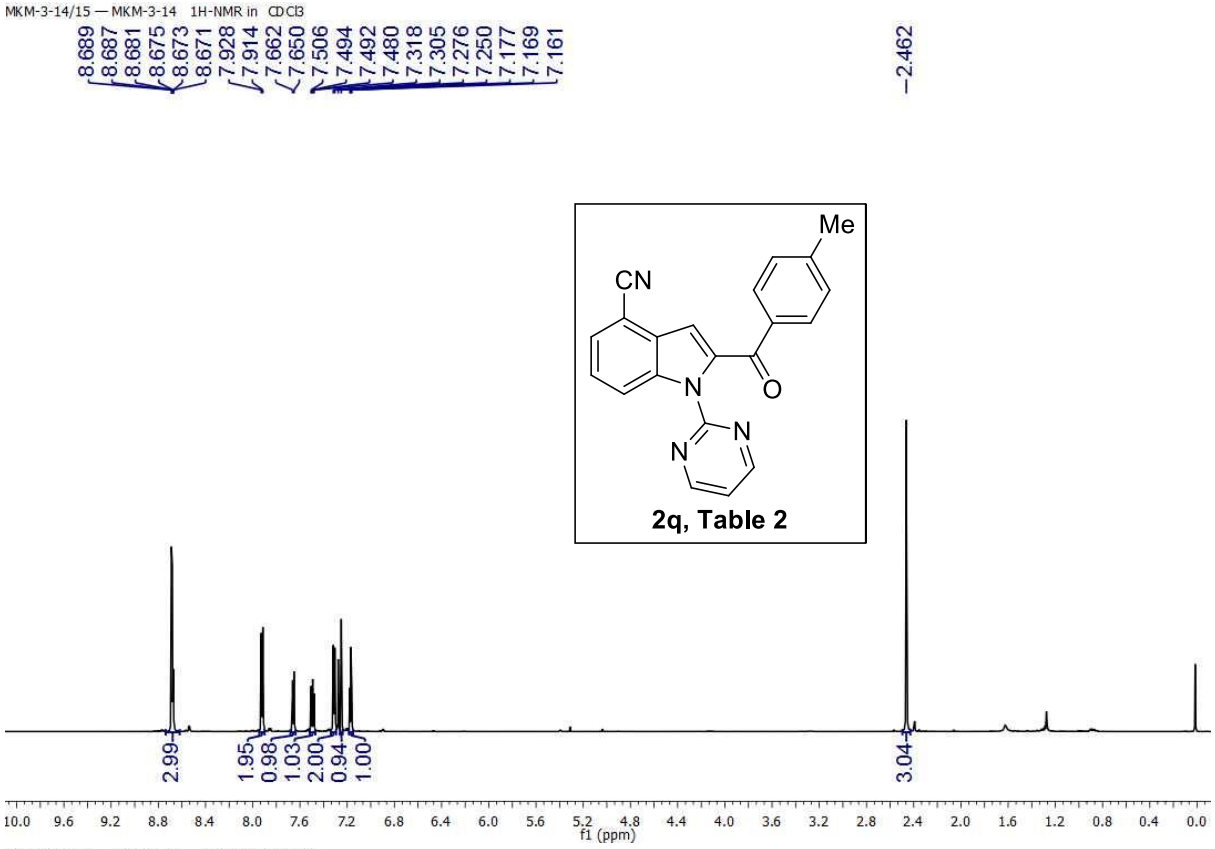

MKM-3-12A-1H/1
8.651
8.623
8.469
8.453
7.793
7.768
7.704
7.678
7.496
7.471
7.445
7.426
7.402
7.367
7.345
7.322
7.298
7.262
6.874
6.857
6.841
2.371
Me
O
N
N
N
2m, Table 2
1.07
1.74
1.90
1.13
2.37
3.11
1.00
3.03
f1 (ppm)

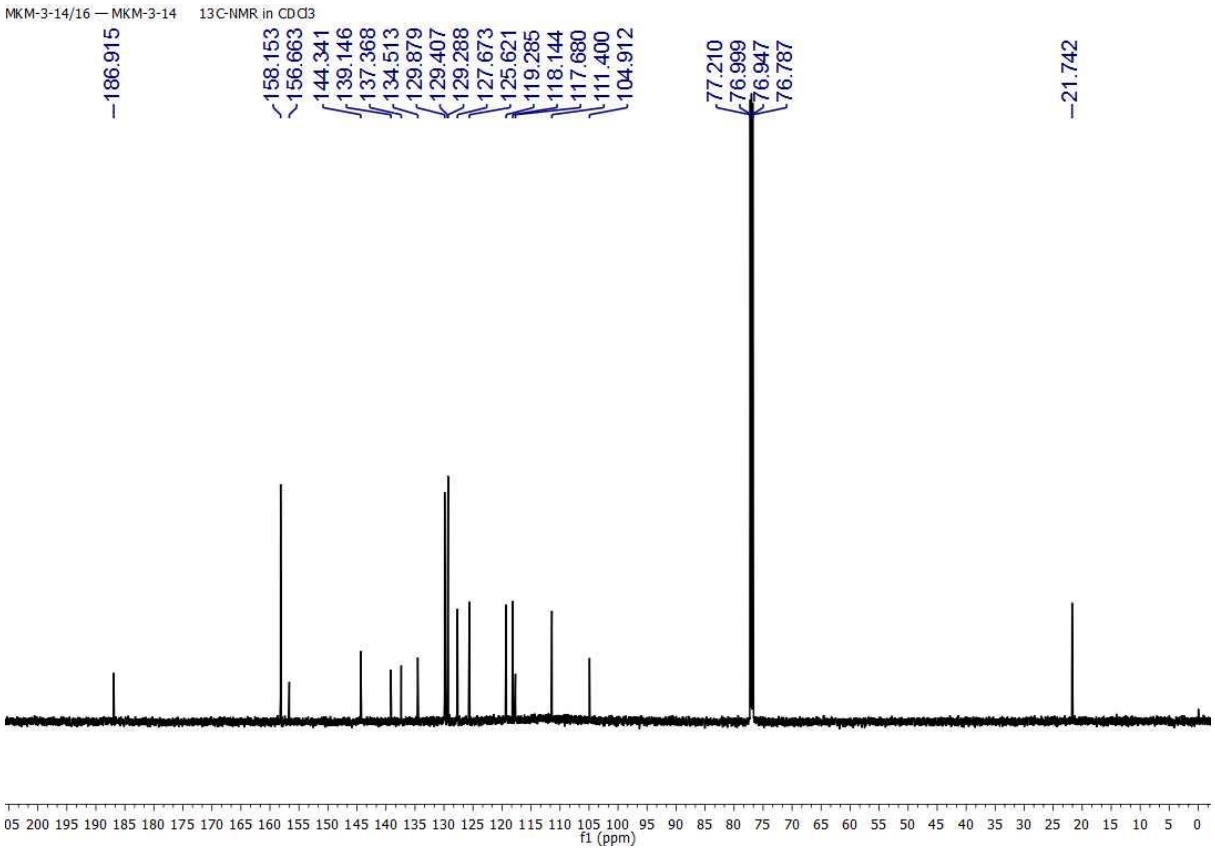

MKM-3-12-C.1.fid
189.427
157.502
156.978
139.156
136.309
133.145
132.217
130.194
128.507
128.252
126.081
122.512
121.698
120.105
116.069
115.121
77.413
76.989
76.566
9.341
0.034
f1 (ppm)

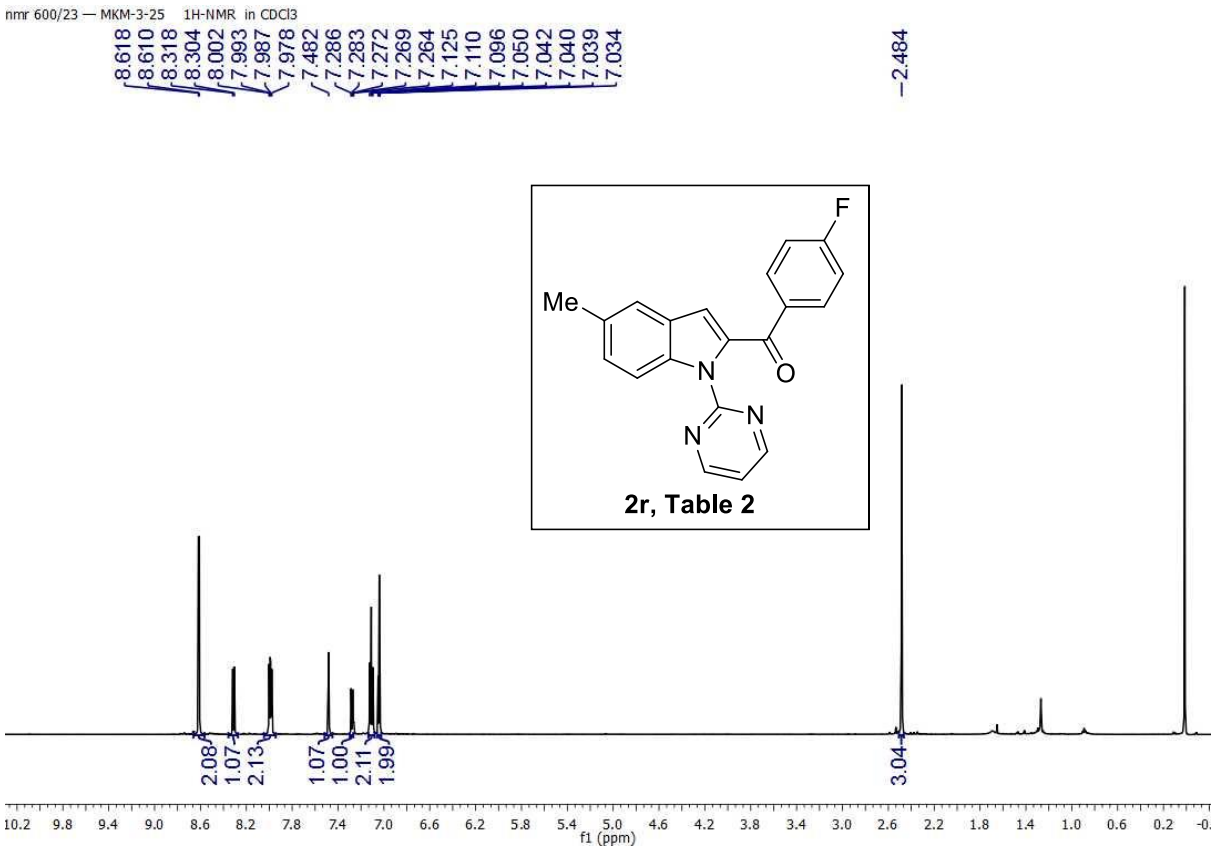

MKM-3-9-1H/1
8.875
8.859
7.875
7.849
7.618
7.602
7.587
7.423
7.407
7.391
7.302
7.275
7.210
7.144
7.128
2.441
1.981
Me
N
O
Me
N
N
2n, Table 2
1.91
2.00
1.13
1.15
2.33
1.14
2.10
2.84
3.16
MKM-3-9-C/1
186.968
159.847
158.321
143.417
138.429
136.337
135.706
130.024
129.288
129.166
127.413
122.645
122.045
121.233
120.361
116.203
77.717
77.292
76.869
21.844
19.637
f1 (ppm)

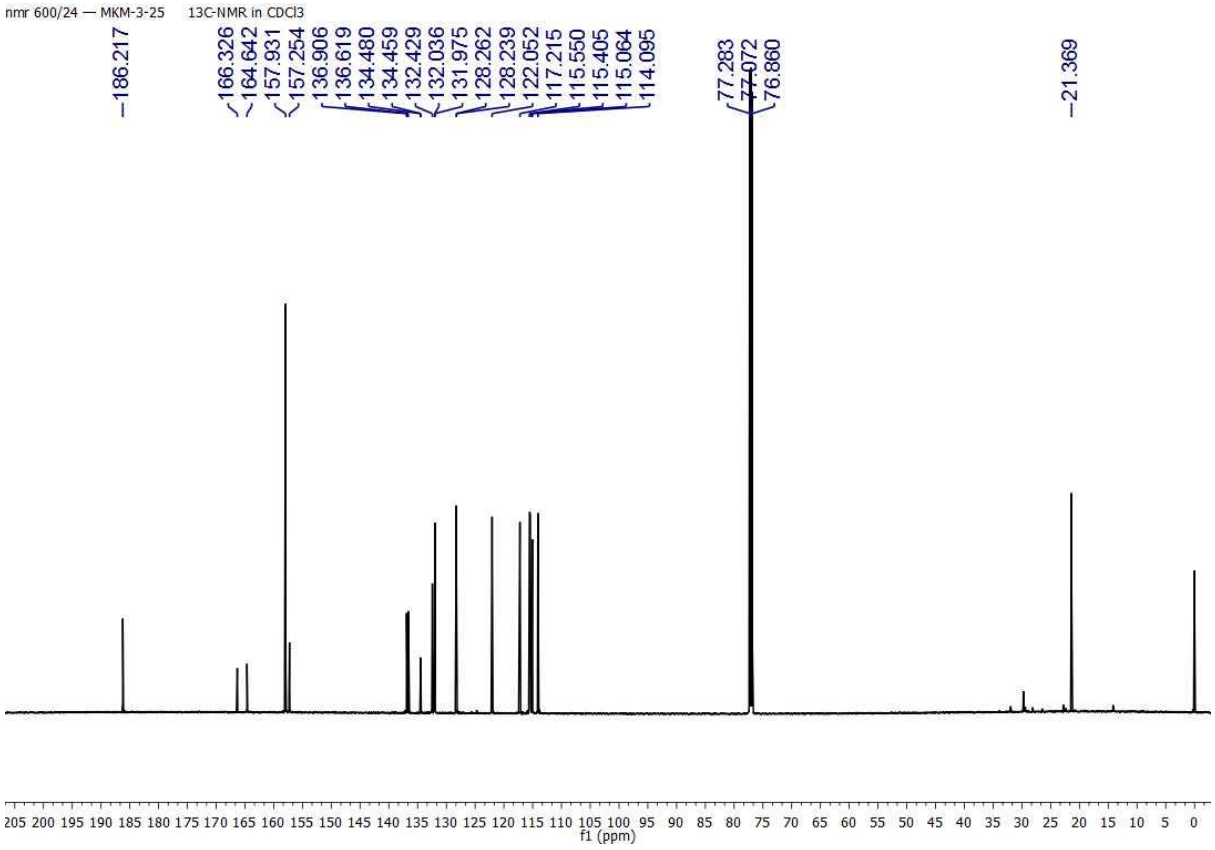

MKM-3-11/5 — MKM-3-11 1H-NMR in CDCl3
8.636
8.628
8.440
8.433
8.425
8.417
7.889
7.875
7.359
7.355
7.345
7.340
7.276
7.267
7.254
7.203
7.198
7.187
7.183
7.172
7.168
7.086
7.078
7.070
7.039
2.432
Me
F
O
N
N
N
2o, Table 2
1.98
1.00
1.98
1.01
1.99
1.00
0.95
0.93
3.06
f1 (ppm)
MKM-3-11/6 — MKM-3-11 13C-NMR in CDCl3
187.281
159.950
158.364
157.914
157.033
143.709
138.634
135.124
134.353
129.662
129.083
128.720
128.654
117.366
115.638
115.578
114.460
114.292
113.847
113.817
107.143
106.987
77.195
76.772
21.675
f1 (ppm)

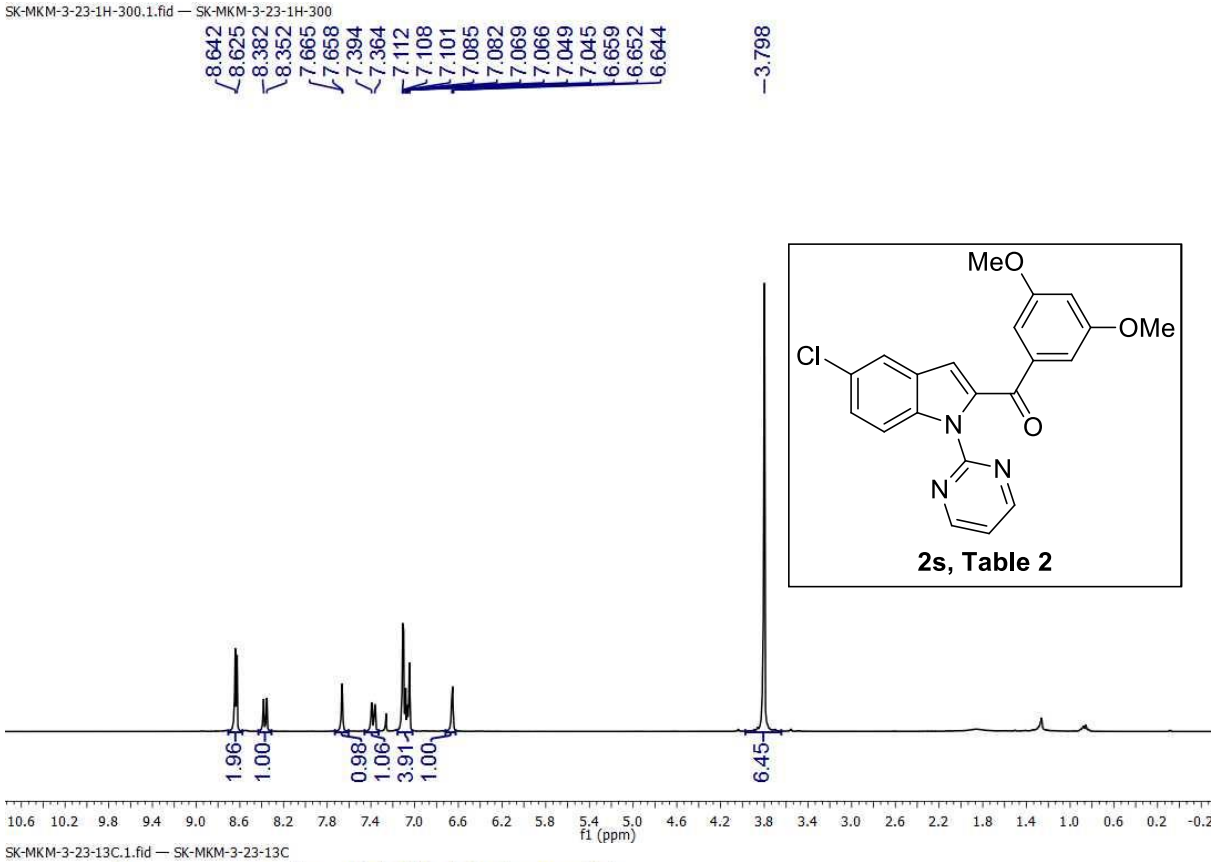

MKM-3-17/19 — MKM-3-17 1H-NMR in CDCl3
8.676
8.667
8.428
8.414
7.730
7.716
7.647
7.644
7.634
7.630
7.615
7.612
7.468
7.466
7.456
7.454
7.452
7.442
7.440
7.324
7.322
7.312
7.310
7.309
7.299
7.297
7.276
7.117
7.100
7.092
7.084
6.892
6.878
3.961
3.949
OMe
OMe
N
O
N
N
2p, Table 2
1.99
1.00
1.02
1.07
1.01
1.06
1.04
0.97
0.99
1.00
6.41
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)
MKM-3-17/20 — MKM-3-17 13C-NMR in CDCl3
186.455
157.942
157.332
153.109
148.937
138.110
137.154
130.858
128.007
126.223
124.777
122.715
122.315
117.304
114.702
114.172
111.287
109.850
77.203
77.002
76.780
56.057
55.988
205 200 195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

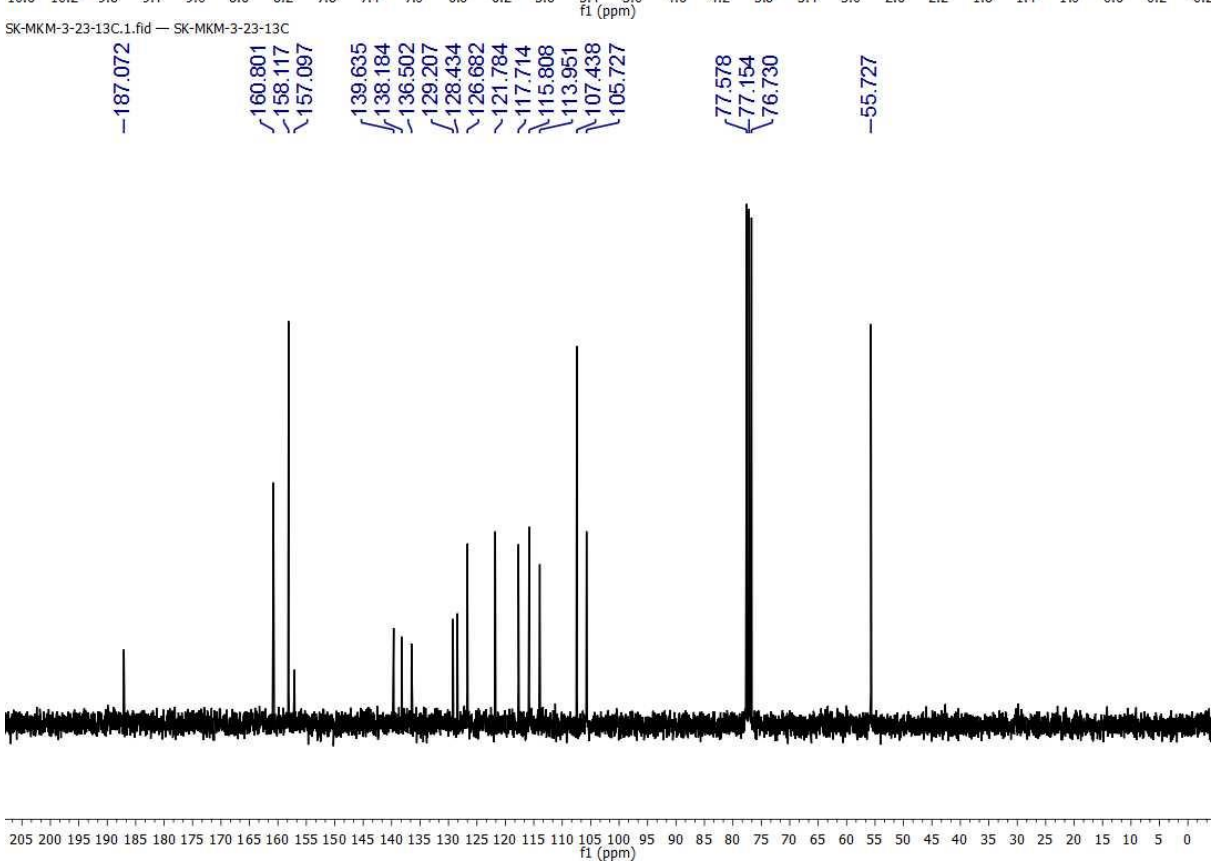

MKM-3-14/15 — MKM-3-14 1H-NMR in CDCl3
8.689
8.687
8.681
8.675
8.673
8.671
7.928
7.914
7.662
7.650
7.506
7.494
7.492
7.480
7.318
7.305
7.276
7.250
7.177
7.169
7.161
2.462
Me
CN
O
N
N
N
2q, Table 2
2.99
1.95
0.98
1.03
2.00
0.94
1.00
3.04
f1 (ppm)


MKM-3-14/16 — MKM-3-14 13C-NMR in CDCl3
186.915
158.153
156.663
144.341
139.146
137.368
134.513
129.879
129.407
129.288
127.673
125.621
119.285
118.144
117.680
111.400
104.912
77.210
76.999
76.947
76.787
21.742
f1 (ppm)

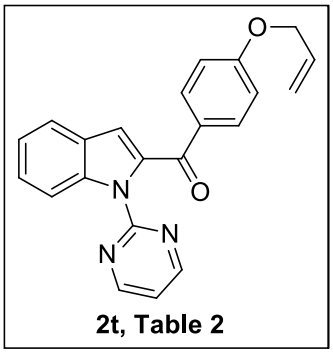

nmr 600/23 — MKM-3-25 1H-NMR in CDCl3
8.618
8.610
8.318
8.304
8.002
7.993
7.987
7.978
7.482
7.286
7.283
7.272
7.269
7.264
7.125
7.110
7.096
7.050
7.042
7.040
7.039
7.034
2.484
F
Me
N
O
N
N
2r, Table 2
2.08
1.07
2.13
1.07
1.00
2.11
1.99
3.04
f1 (ppm)


nmr 600/24 — MKM-3-25 13C-NMR in CDCl3
186.217
166.326
164.642
157.931
157.254
136.906
136.619
134.480
134.459
132.429
132.036
131.975
128.262
128.239
122.052
117.215
115.550
115.405
115.064
114.095
77.283
77.072
76.860
21.369
f1 (ppm)

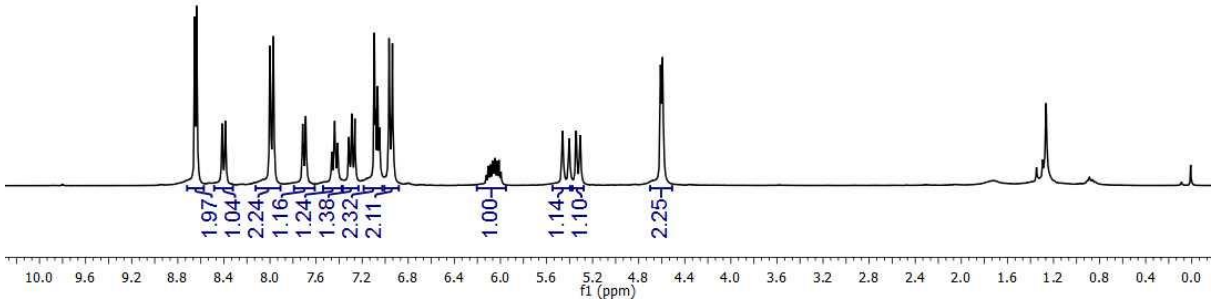

SK-MKM-3-23-1H-300.1.fid — SK-MKM-3-23-1H-300
8.642
8.625
8.382
8.352
7.665
7.658
7.394
7.364
7.112
7.108
7.101
7.085
7.082
7.069
7.066
7.049
7.045
6.659
6.652
6.644
3.798
MeO
OMe
Cl
O
N
N
N
2s, Table 2
1.96
1.00
0.98
1.06
3.91
1.00
6.45
f1 (ppm)

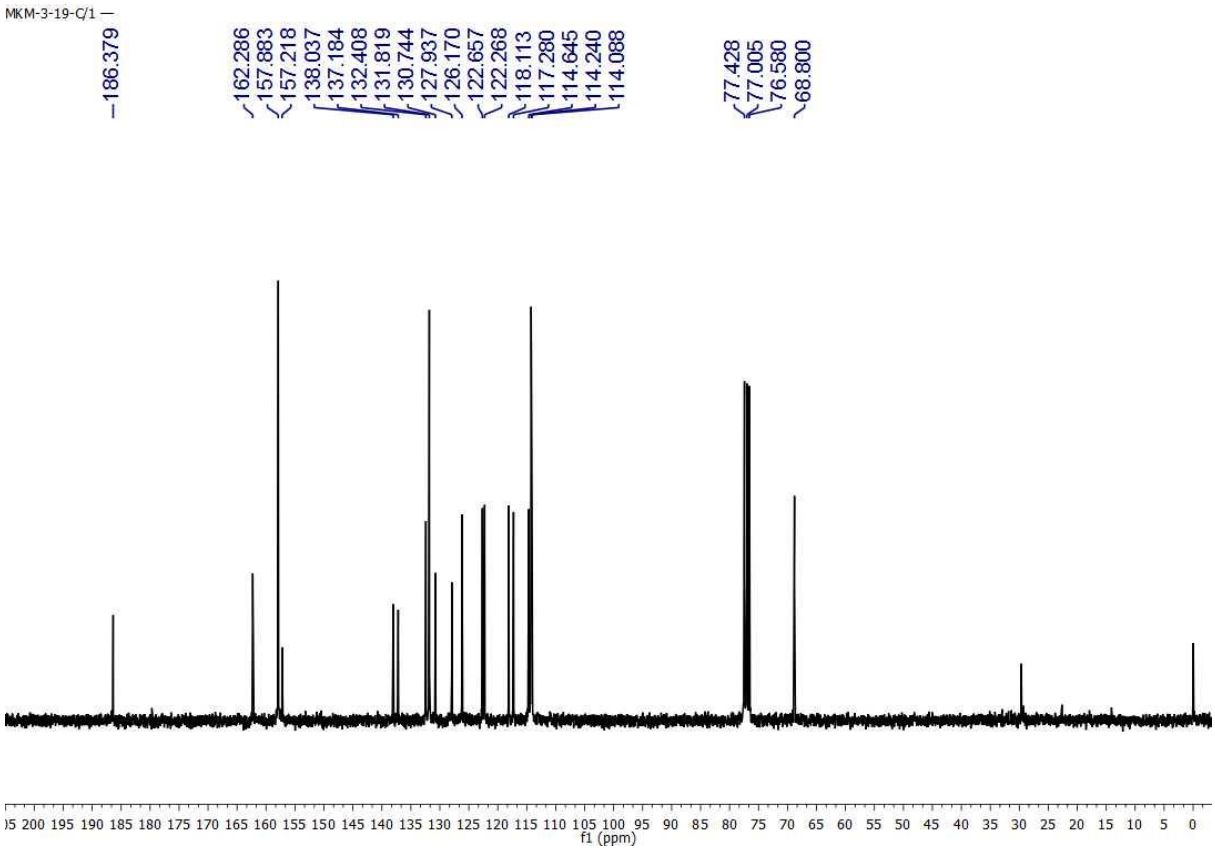

SK-MKM-3-23-13C.1.fid — SK-MKM-3-23-13C
187.072
160.801
158.117
157.097
139.635
138.184
136.502
129.207
128.434
126.682
121.784
117.714
115.808
113.951
107.438
105.727
77.578
77.154
76.730
55.727
f1 (ppm)

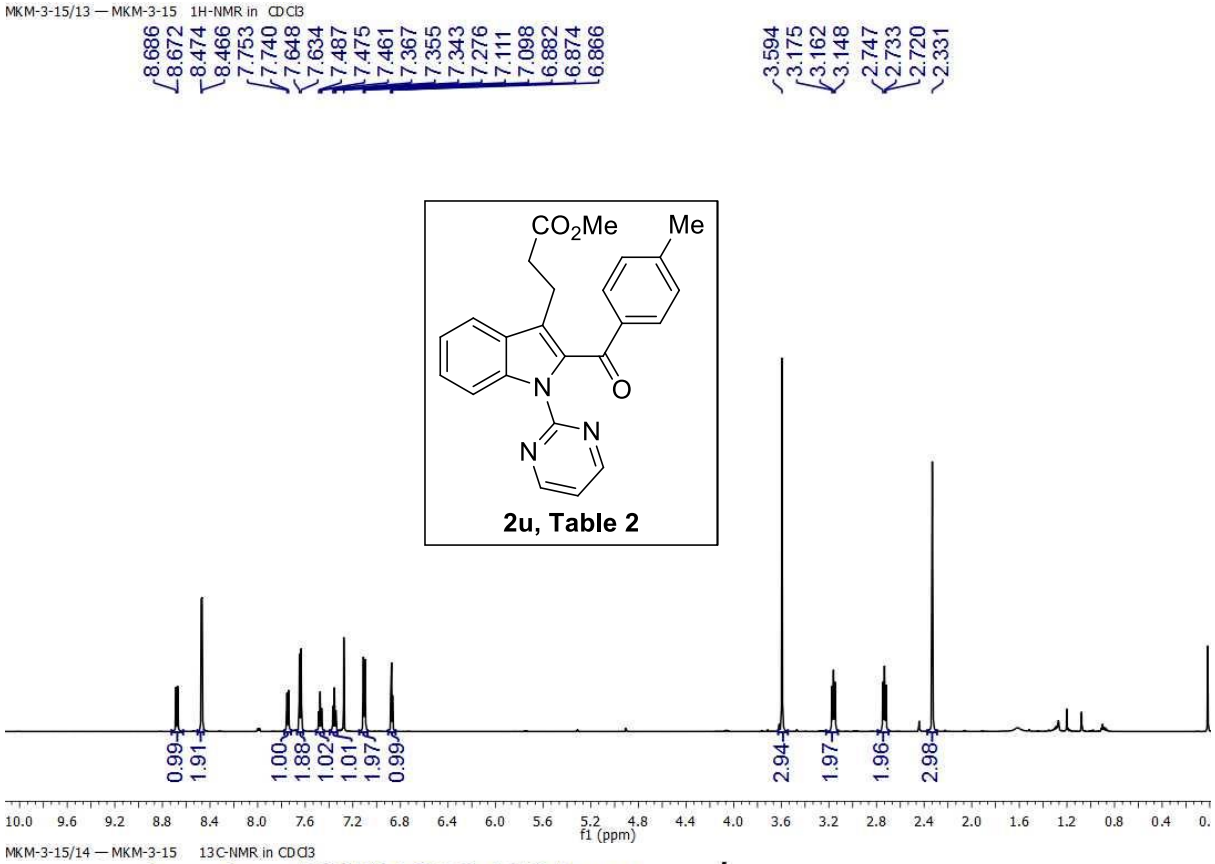

MKM-3-19-1H/1
8.653
8.637
8.416
8.388
8.001
7.971
7.716
7.690
7.439
7.315
7.290
7.263
7.094
7.082
7.066
7.050
6.966
6.937
6.122
6.105
6.087
6.070
6.065
6.053
6.047
6.030
6.012
5.995
5.460
5.402
5.342
5.307
4.611
4.593

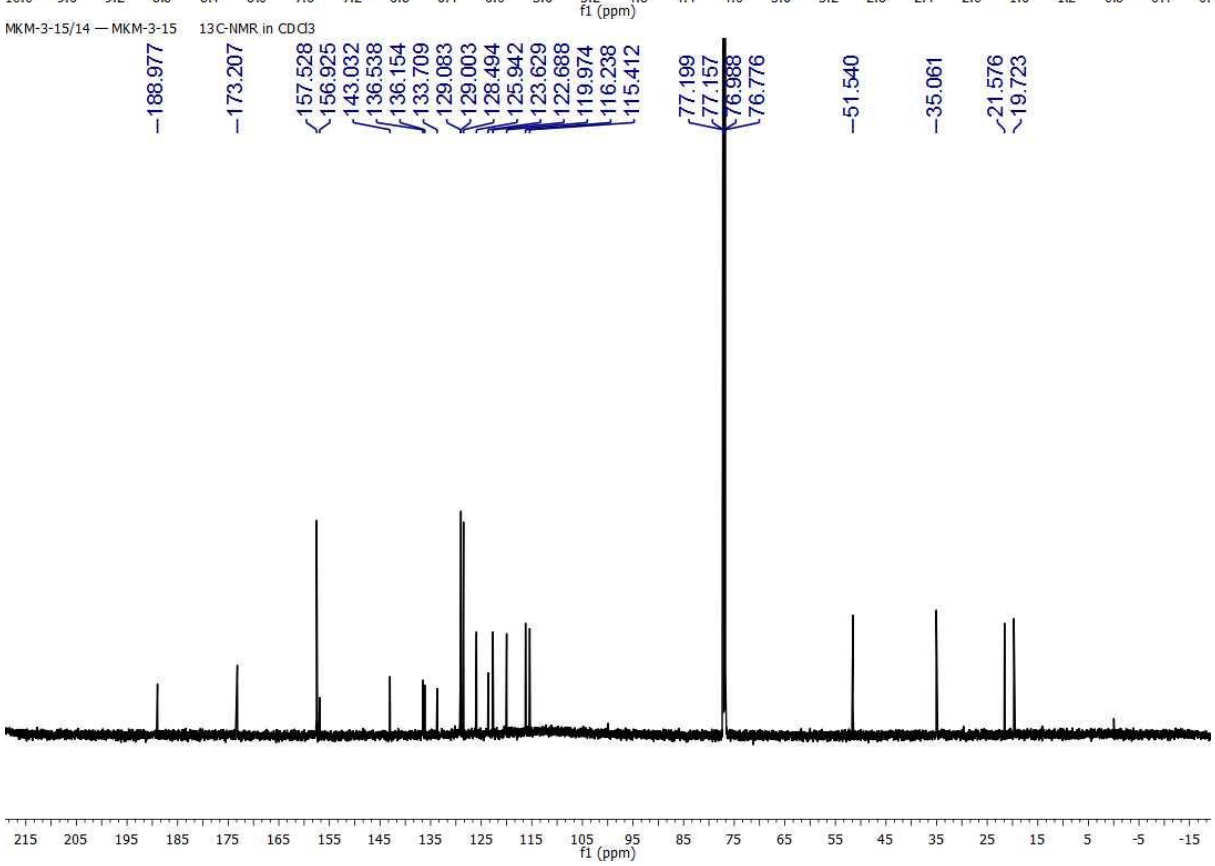

O
N
O
N
N

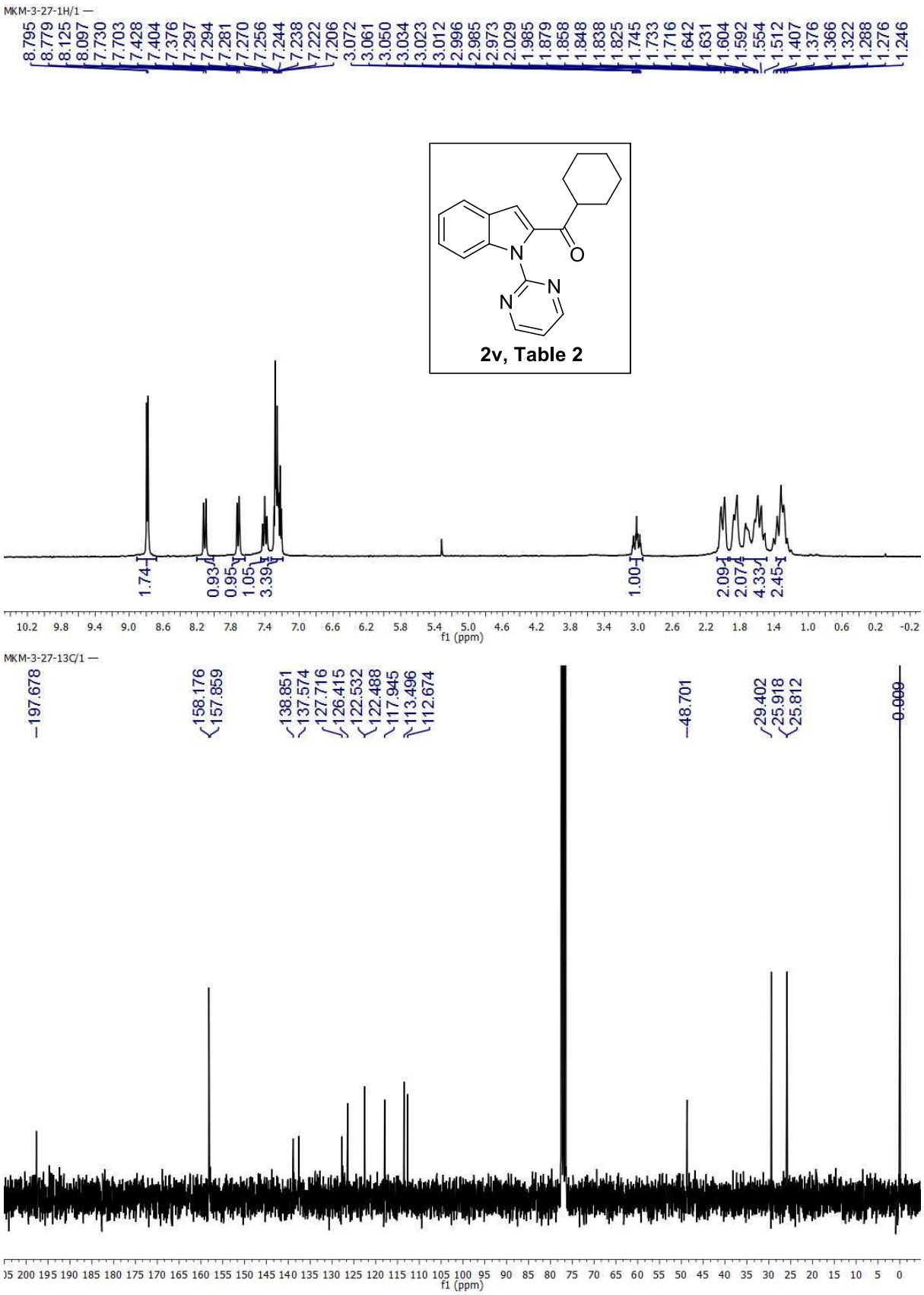
2t, Table 2

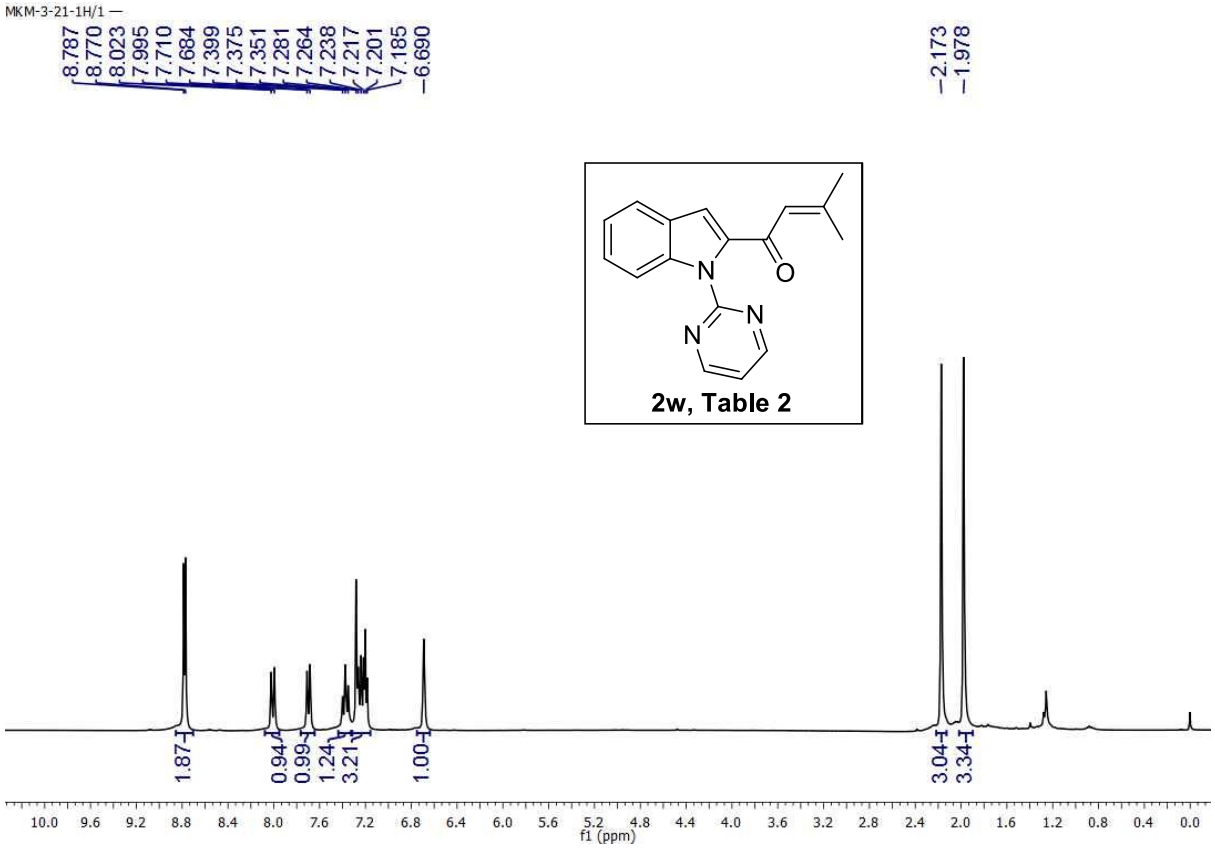

1.97
1.04
2.24
1.16
1.24
1.38
2.32
2.11
1.00
1.14
1.10
2.25
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)

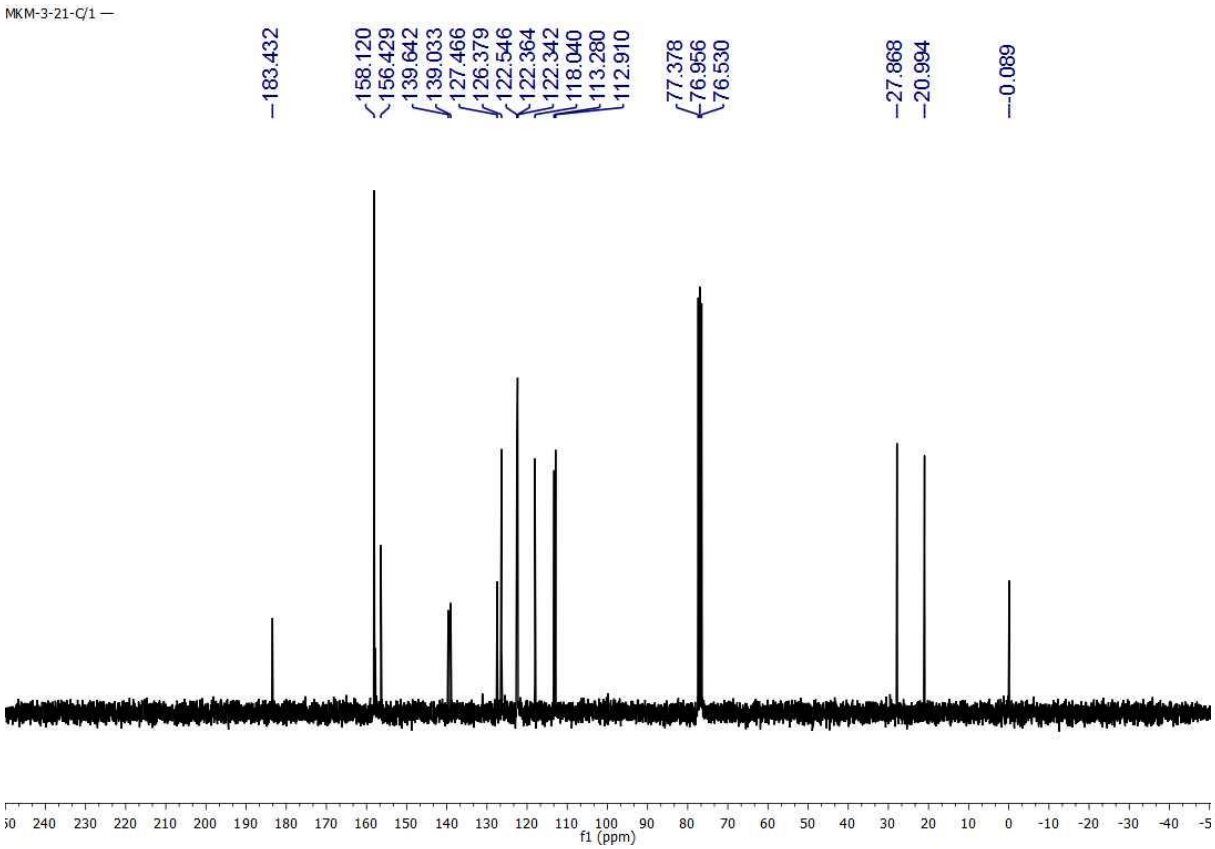

MKM-3-19-C/1
186.379
162.286
157.883
157.218
138.037
137.184
132.408
131.819
130.744
127.937
126.170
122.657
122.268
118.113
117.280
114.645
114.240
114.088
77.428
77.005
76.580
68.800
f1 (ppm)


MKM-3-15/13 — MKM-3-15 1H-NMR in CDCl3
8.686
8.672
8.474
8.466
7.753
7.740
7.648
7.634
7.487
7.475
7.461
7.367
7.355
7.343
7.276
7.111
7.098
6.882
6.874
6.866
3.594
3.175
3.162
3.148
2.747
2.733
2.720
2.331
CO2Me
Me
N
O
N
N
2u, Table 2
0.99
1.91
1.00
1.88
1.02
1.01
1.97
0.99
2.94
1.97
1.96
2.98
f1 (ppm)


MKM-3-15/14 — MKM-3-15 13C-NMR in CDCl3
188.977
173.207
157.528
156.925
143.032
136.538
136.154
133.709
129.083
129.003
128.494
125.942
123.629
122.688
119.974
116.238
115.412
77.199
77.157
76.988
76.776
51.540
35.061
21.576
19.723
f1 (ppm)


MKM-3-27-1H/1
8.795
8.779
8.125
8.097
7.730
7.703
7.428
7.404
7.376
7.297
7.294
7.281
7.270
7.256
7.244
7.238
7.222
7.206
3.072
3.061
3.050
3.034
3.023
3.012
2.996
2.985
2.973
2.029
1.985
1.879
1.858
1.848
1.838
1.825
1.745
1.733
1.716
1.642
1.631
1.604
1.592
1.554
1.512
1.407
1.376
1.366
1.322
1.288
1.276
1.246
N
O
N
N
2v, Table 2
1.74
0.93
0.95
1.05
3.39
1.00
2.09
2.07
4.33
2.45
f1 (ppm)
MKM-3-27-13C/1
197.678
158.176
157.859
138.851
137.574
127.716
126.415
122.532
122.488
117.945
113.496
112.674
48.701
29.402
25.918
25.812
0.000
f1 (ppm)

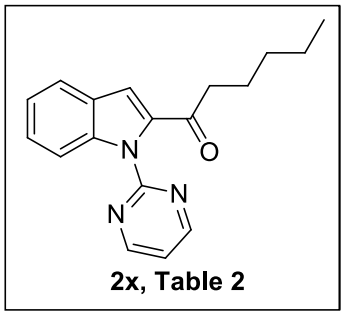

MKM-3-21-1H/1
8.787
8.770
8.023
7.995
7.710
7.684
7.399
7.375
7.351
7.281
7.264
7.238
7.217
7.201
7.185
6.690
2.173
1.978
2w, Table 2
1.87
0.94
0.99
1.24
3.21
1.00
3.04
3.34
f1 (ppm)


MKM-3-21-C/1
183.432
158.120
156.429
139.642
139.033
127.466
126.379
122.546
122.364
122.342
118.040
113.280
112.910
77.378
76.956
76.530
27.868
20.994
0.089
f1 (ppm)

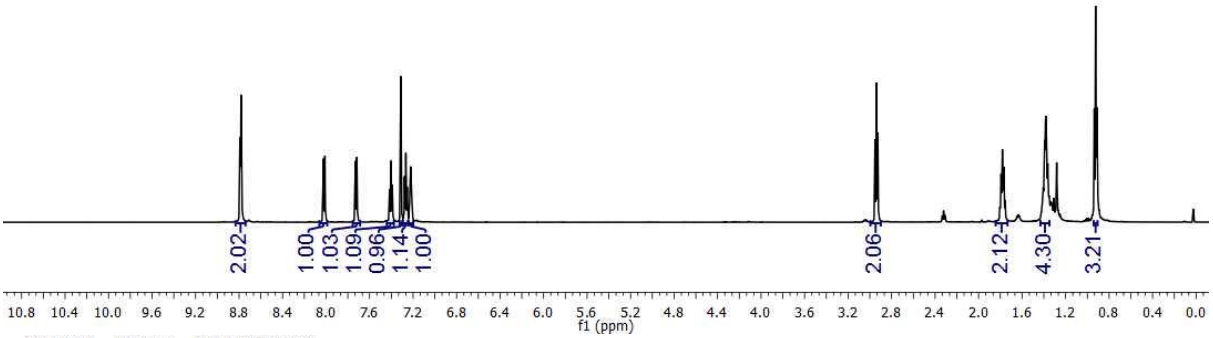
MKM-3-2/7 — MKM-3-2 1H-NMR in CDCl3

8.790 8.782 8.025 8.011 7.730 7.717 7.415 7.403 7.391 7.313 7.279 7.276 7.266 7.254 7.230 7.222 7.212

2.952 2.939 2.927 1.805 1.793 1.781 1.768 1.756 1.410 1.405 1.399 1.396 1.391 1.386 1.384 1.380 1.375 1.372 1.364 1.361 1.352 0.935 0.924 0.913

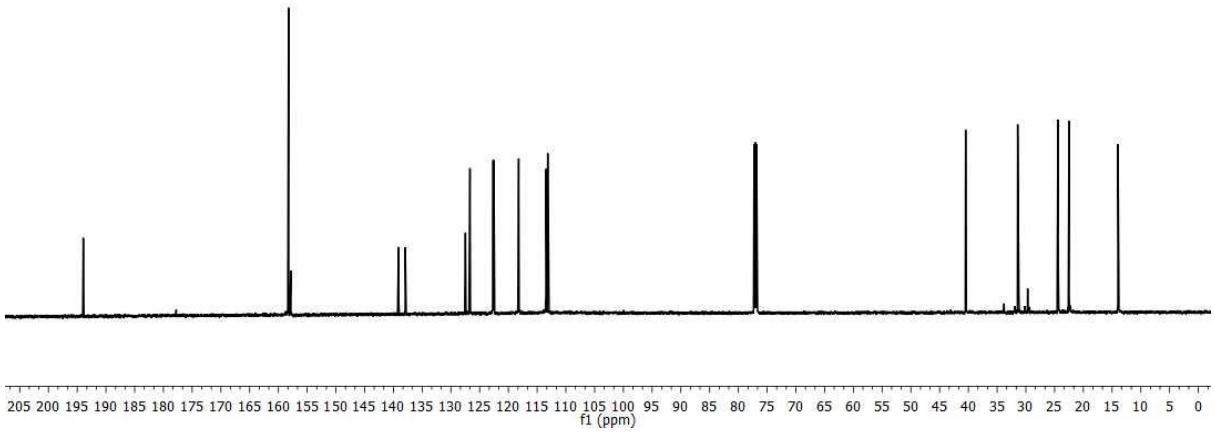


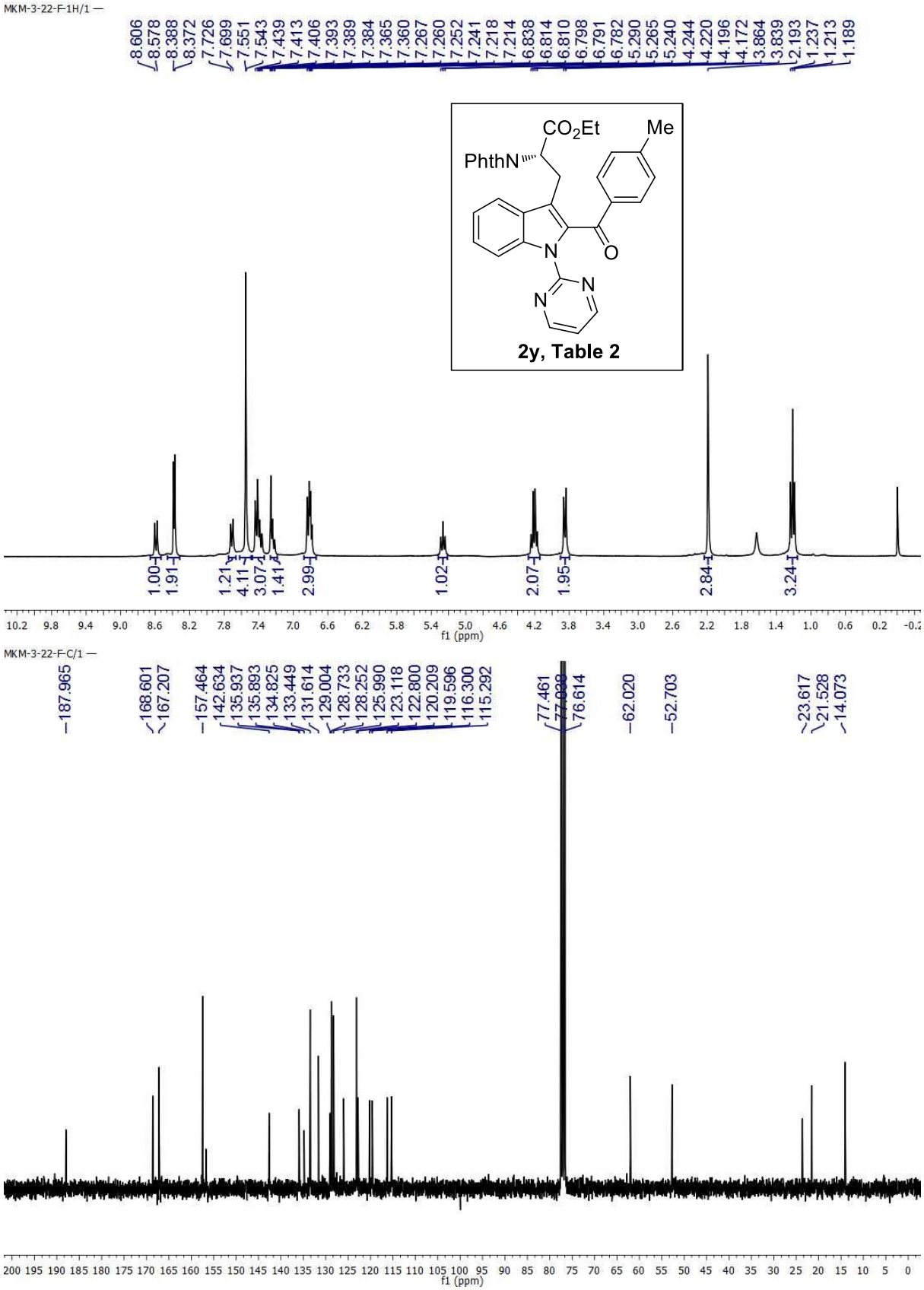
2x, Table 2

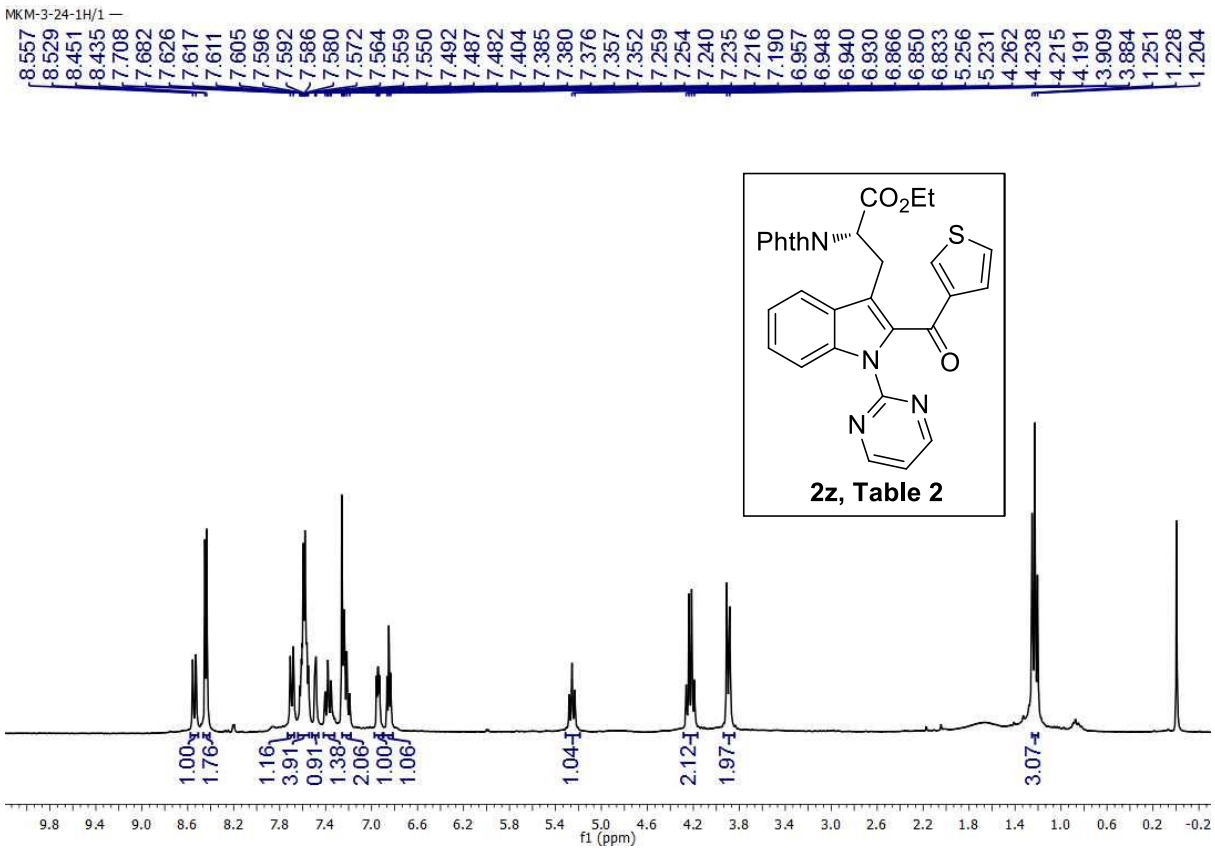


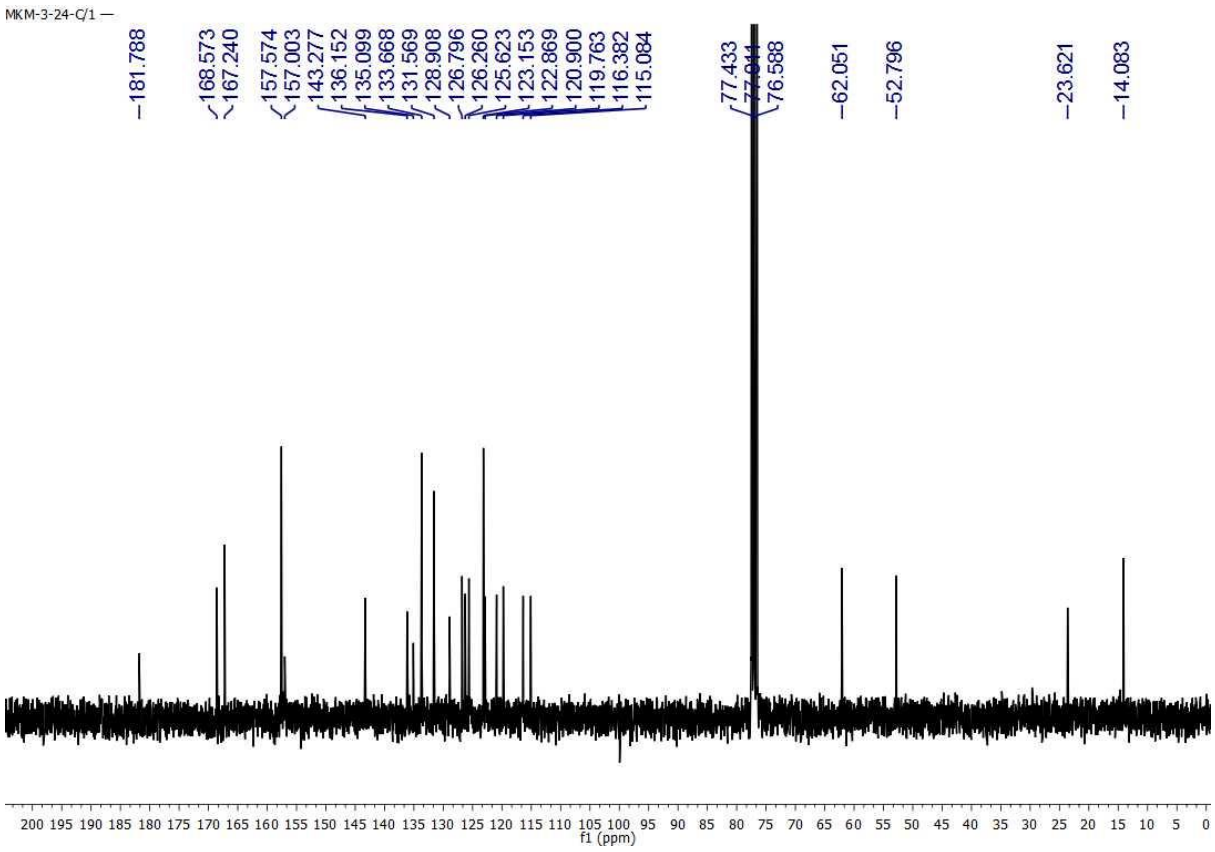
MKM-3-2/8 — MKM-3-2 13C-NMR in CDCl3

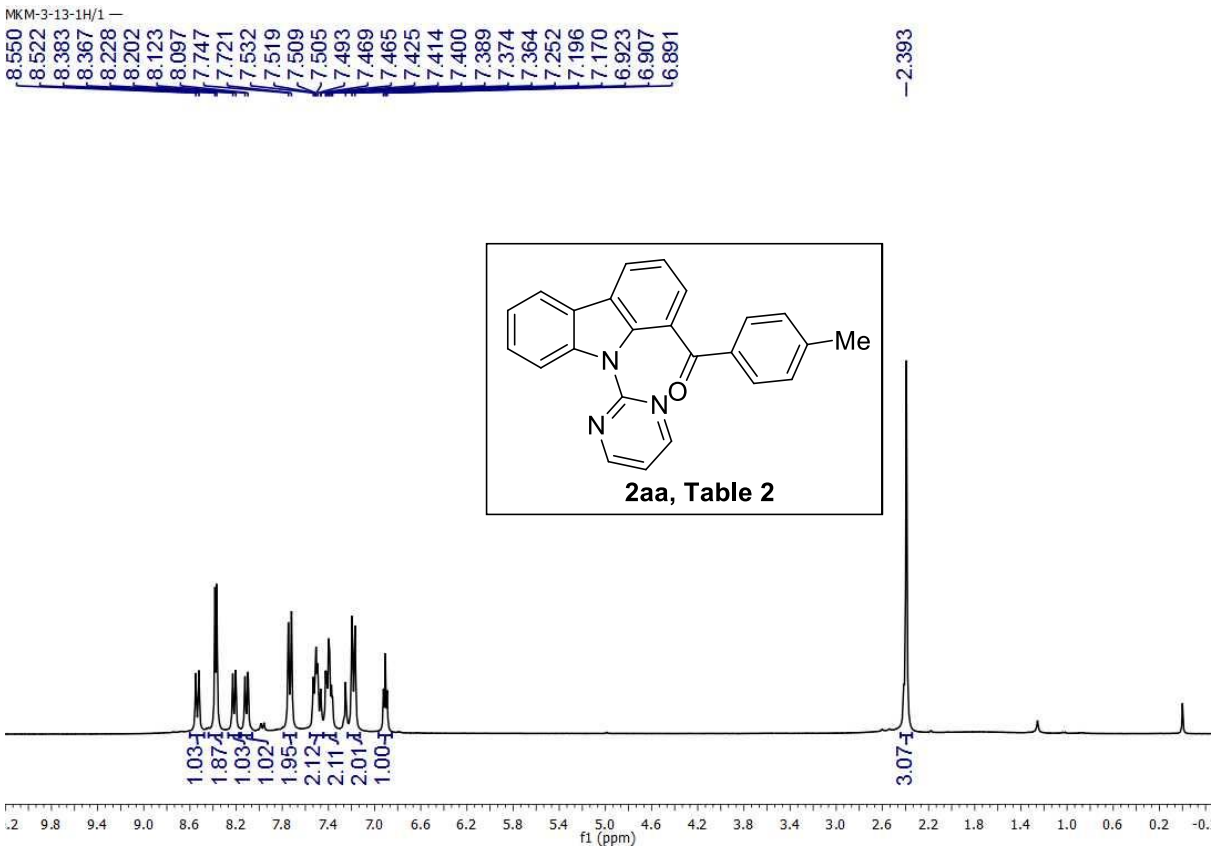
193.937 158.229 157.841 139.118 137.932 127.472 126.679 122.653 122.523 118.209 113.455 113.158 77.262 77.050 76.839 40.473 31.420 24.409 22.496 13.974

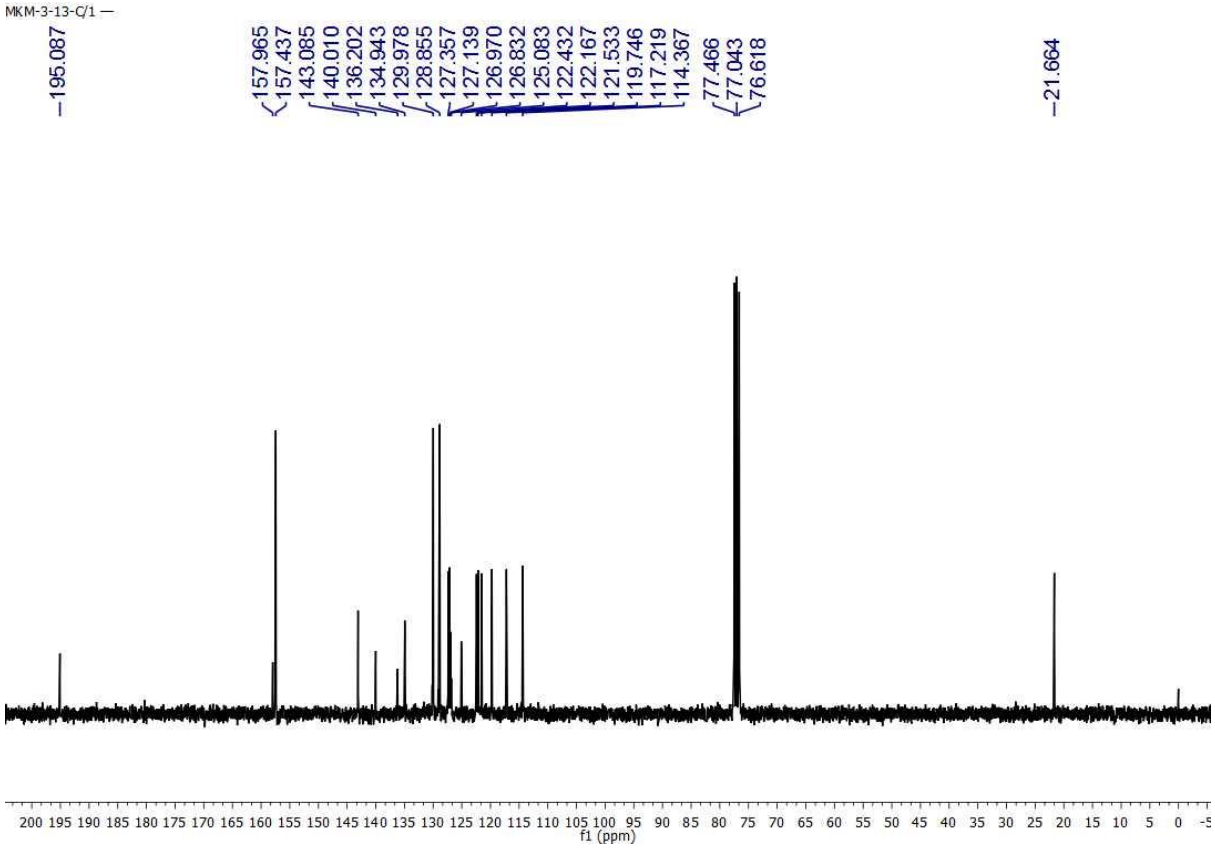


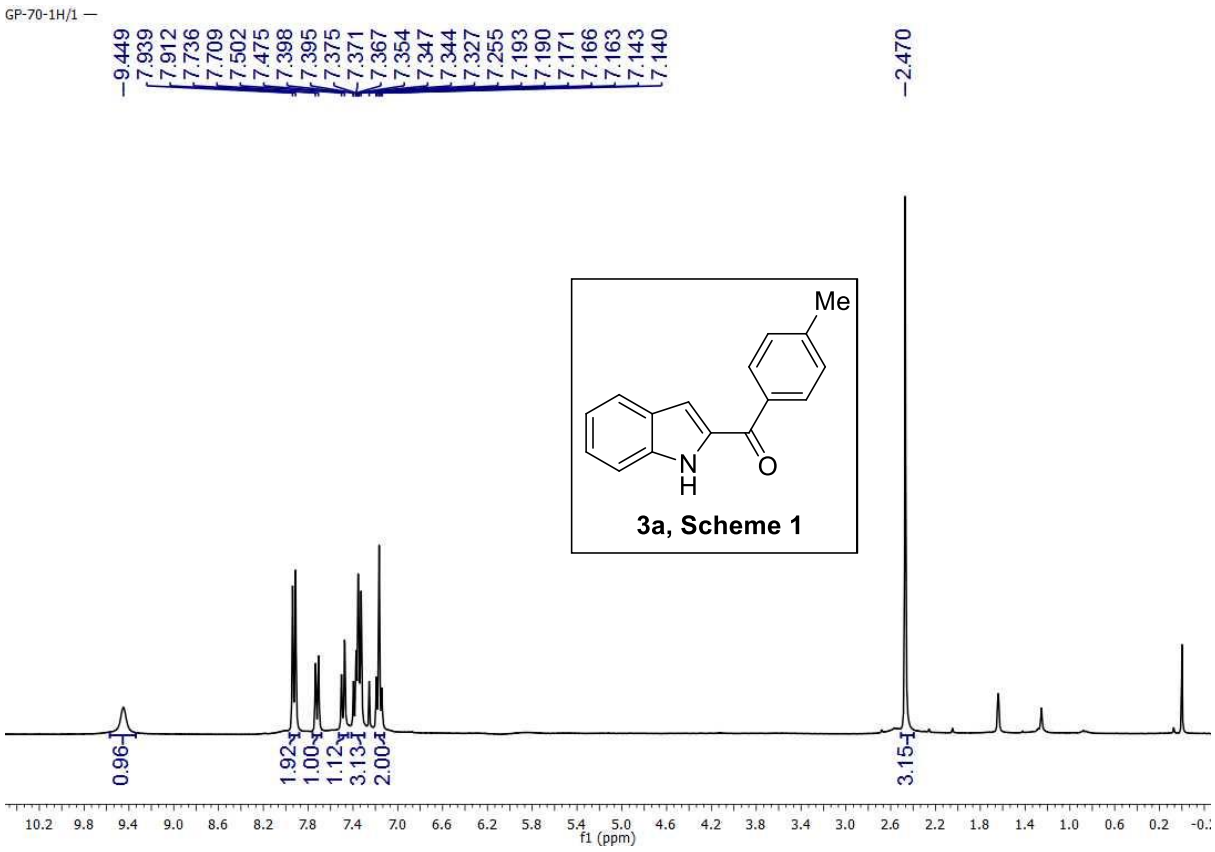

MKM-3-22-F-1H/1
8.606
8.578
8.388
8.372
7.726
7.699
7.551
7.543
7.439
7.413
7.406
7.393
7.389
7.384
7.365
7.360
7.267
7.260
7.252
7.241
7.218
7.214
6.838
6.814
6.810
6.798
6.791
6.782
5.290
5.265
5.240
4.244
4.220
4.196
4.172
3.864
3.839
2.193
1.237
1.213
1.189
CO2Et
Me
PhthN
O
N
N
N
2y, Table 2
1.00
1.91
1.21
4.11
3.07
1.41
2.99
1.02
2.07
1.95
2.84
3.24
f1 (ppm)
MKM-3-22-F-C/1
187.965
168.601
167.207
157.464
142.634
135.937
135.893
134.825
133.449
131.614
129.004
128.733
128.252
125.990
123.118
122.800
120.209
119.596
116.300
115.292
77.461
77.038
76.614
62.020
52.703
23.617
21.528
14.073
f1 (ppm)

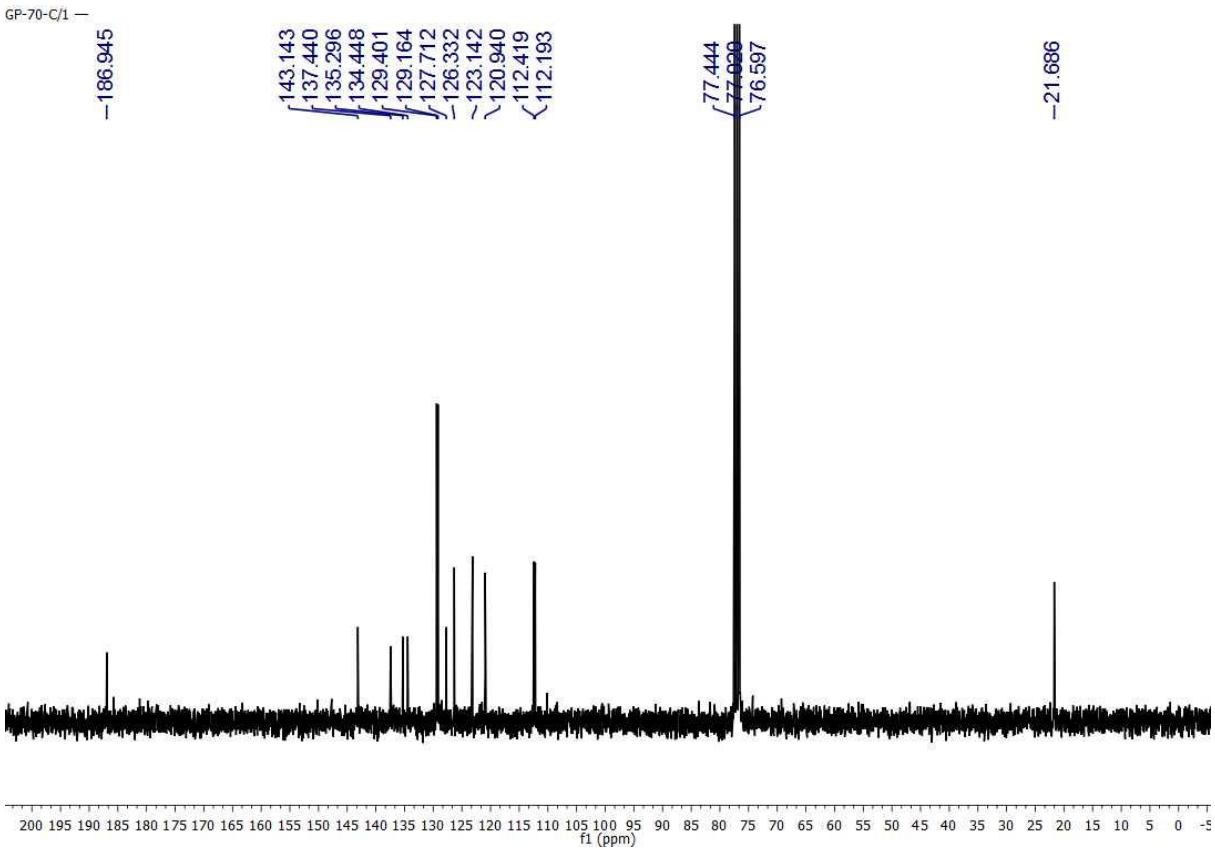

MKM-3-24-1H/1
8.557
8.529
8.451
8.435
7.708
7.682
7.626
7.617
7.611
7.605
7.596
7.592
7.586
7.580
7.572
7.564
7.559
7.550
7.492
7.487
7.482
7.404
7.385
7.380
7.376
7.357
7.352
7.259
7.254
7.240
7.235
7.216
7.190
6.957
6.948
6.940
6.930
6.866
6.850
6.833
5.256
5.231
4.262
4.238
4.215
4.191
3.909
3.884
1.251
1.228
1.204
CO2Et
PhthN
S
N
O
N
N
2z, Table 2
1.00
1.76
1.16
3.91
0.91
1.38
2.06
1.00
1.06
1.04
2.12
1.97
3.07
f1 (ppm)

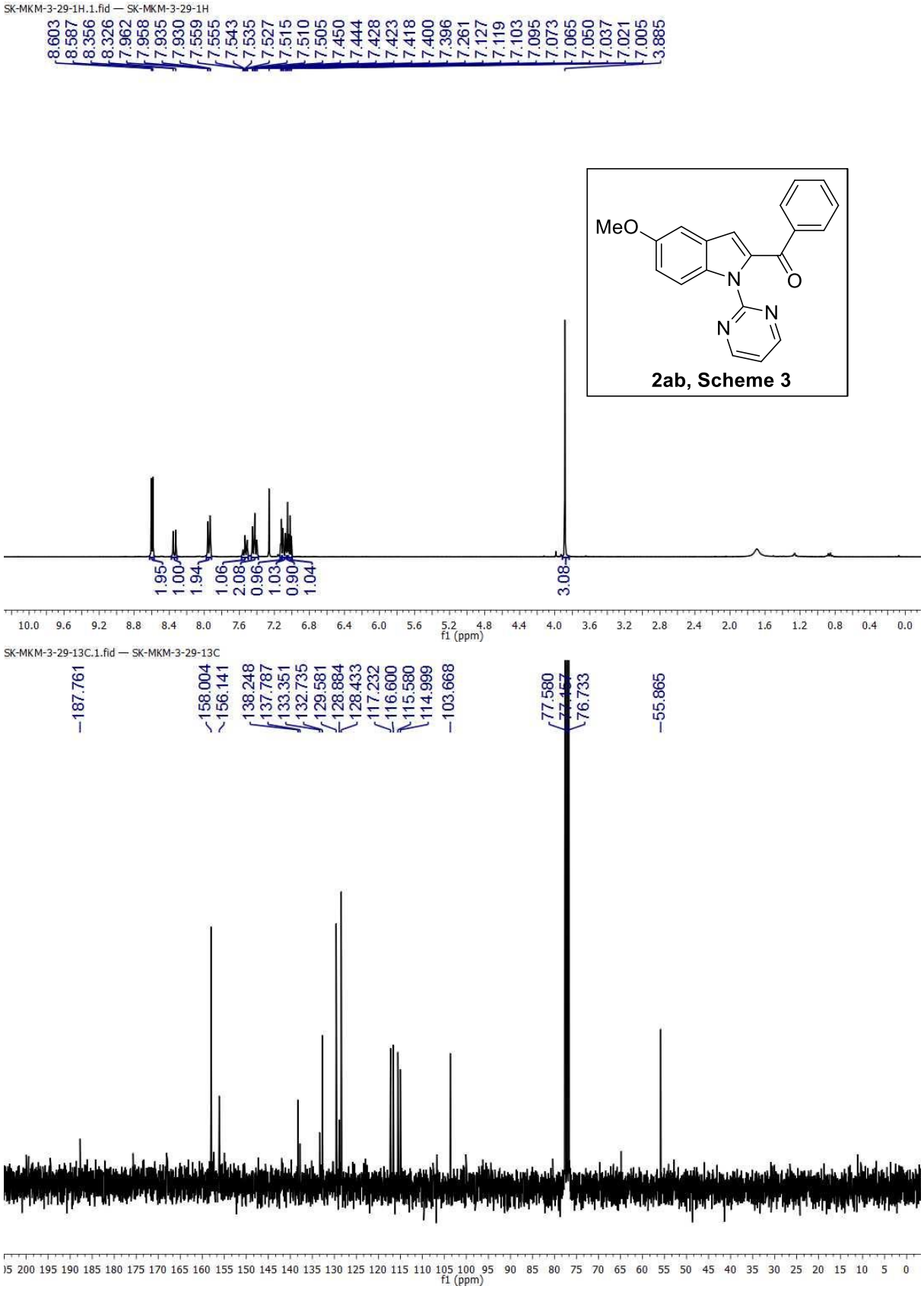

MKM-3-24-C/1
181.788
168.573
167.240
157.574
157.003
143.277
136.152
135.099
133.668
131.569
128.908
126.796
126.260
125.623
123.153
122.869
120.900
119.763
116.382
115.084
77.433
77.011
76.588
62.051
52.796
23.621
14.083
f1 (ppm)

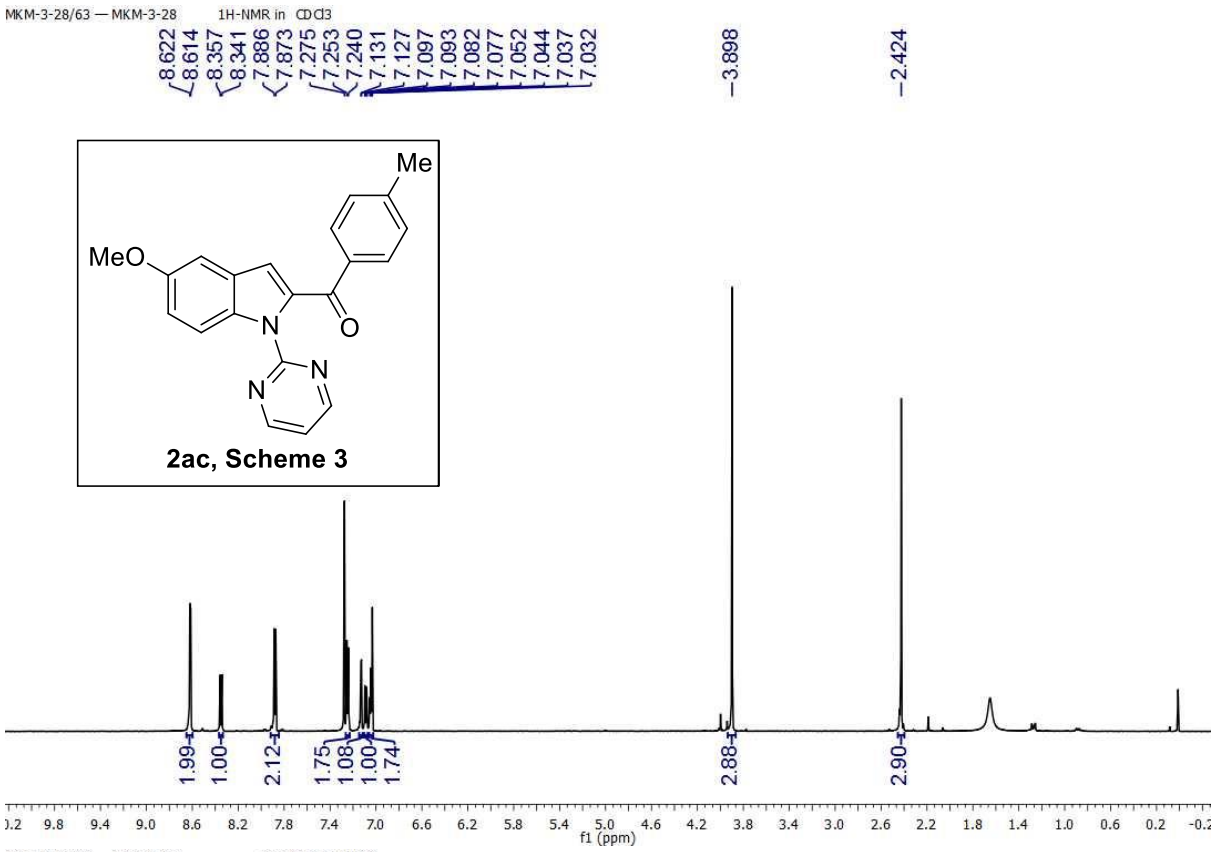

MKM-3-13-1H/1
8.550
8.522
8.383
8.367
8.228
8.202
8.123
8.097
7.747
7.721
7.532
7.519
7.509
7.505
7.493
7.469
7.465
7.425
7.414
7.400
7.389
7.374
7.364
7.252
7.196
7.170
6.923
6.907
6.891
2.393
Me
N
O
N
N
2aa, Table 2
1.03
1.87
1.03
1.02
1.95
2.12
2.11
2.01
1.00
3.07
f1 (ppm)

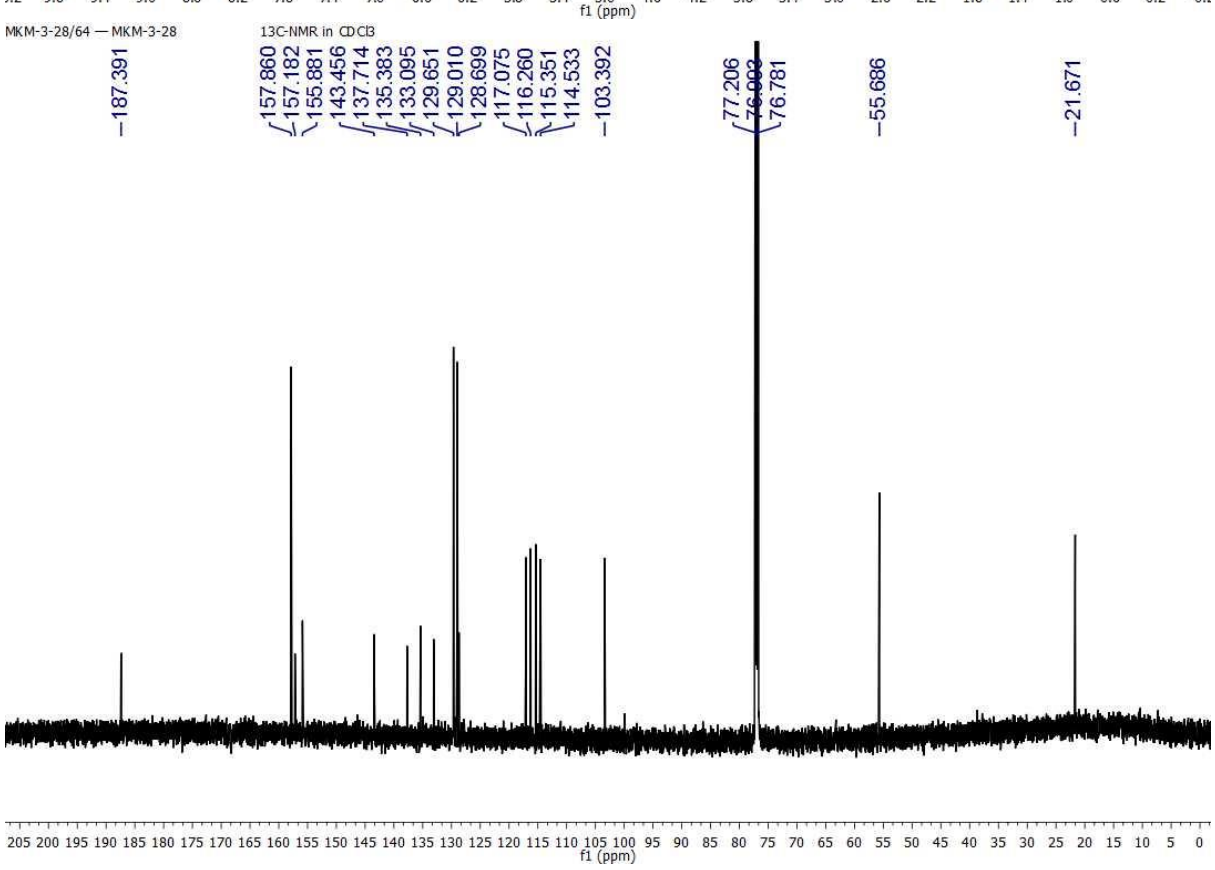

MKM-3-13-C/1
195.087
157.965
157.437
143.085
140.010
136.202
134.943
129.978
128.855
127.357
127.139
126.970
126.832
125.083
122.432
122.167
121.533
119.746
117.219
114.367
77.466
77.043
76.618
21.664
f1 (ppm)

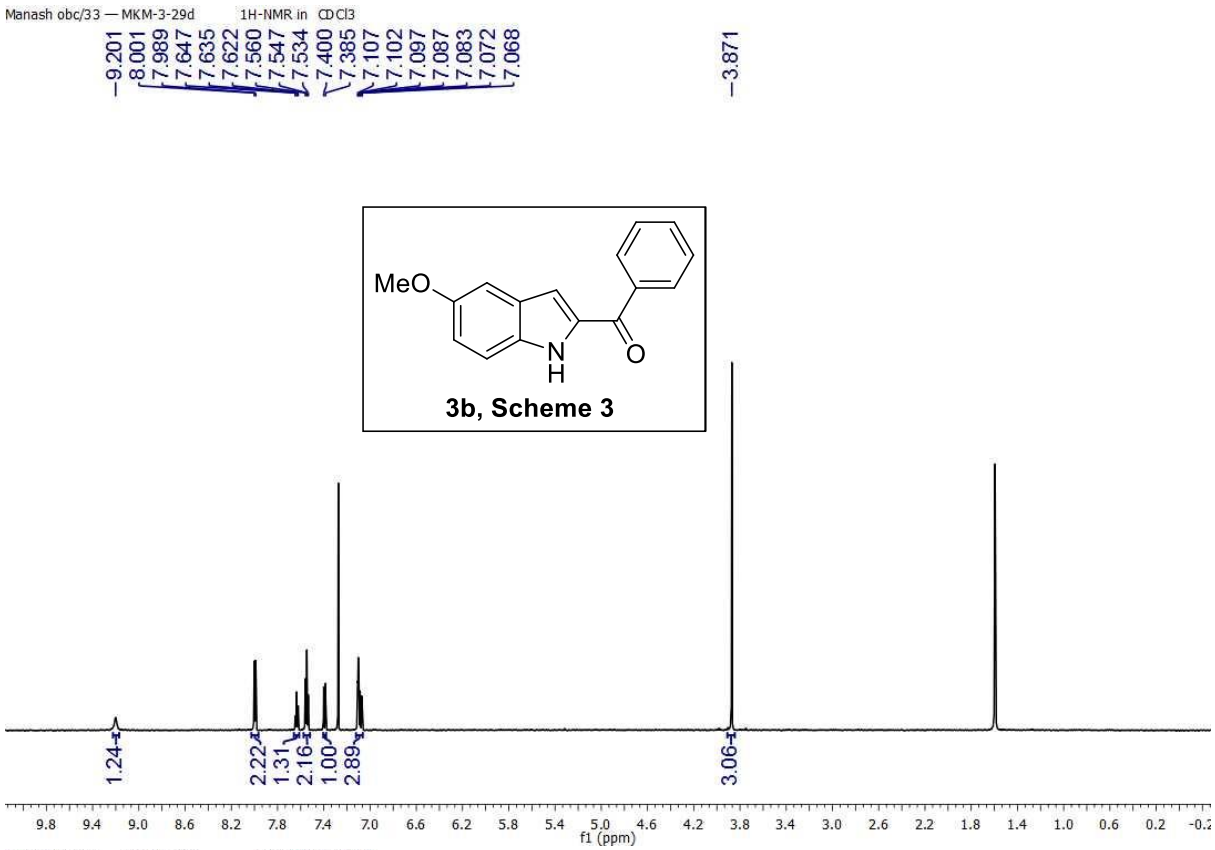

GP-70-1H/1
9.449
7.939
7.912
7.736
7.709
7.502
7.475
7.398
7.395
7.375
7.371
7.367
7.354
7.347
7.344
7.327
7.255
7.193
7.190
7.171
7.166
7.163
7.143
7.140
2.470
Me
N
H
O
3a, Scheme 1
0.96
1.92
1.00
1.12
3.13
2.00
3.15
f1 (ppm)

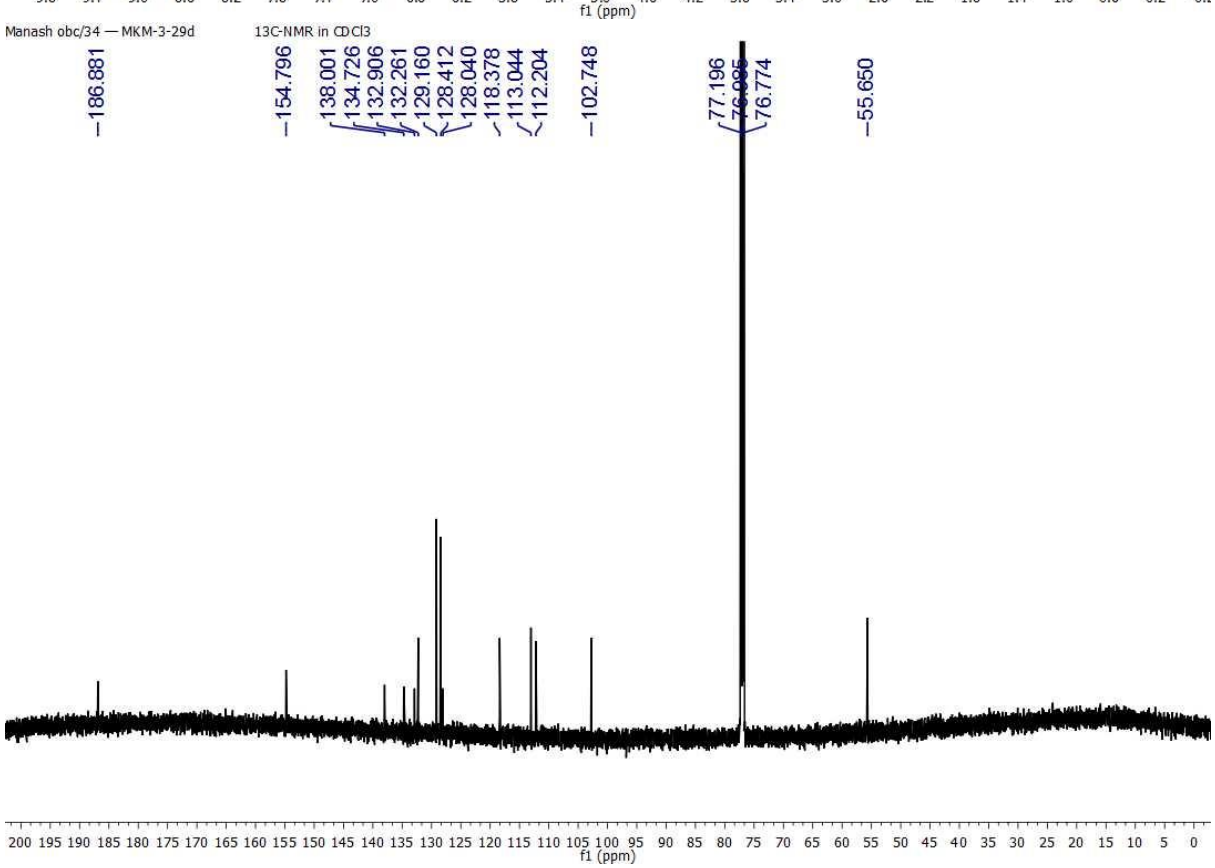

GP-70-C/1
186.945
143.143
137.440
135.296
134.448
129.401
129.164
127.712
126.332
123.142
120.940
112.419
112.193
77.444
77.030
76.597
21.686
f1 (ppm)

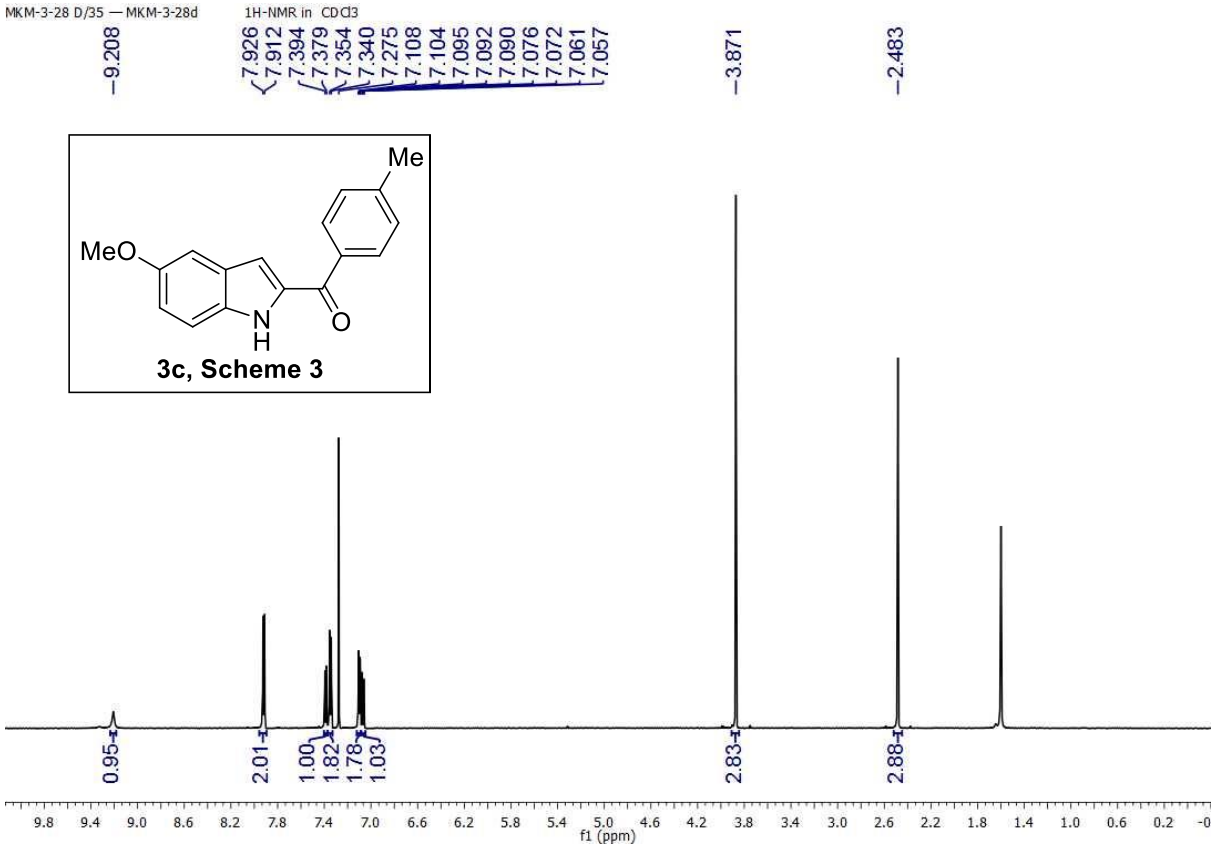

SK-MKM-3-29-1H.1.fid — SK-MKM-3-29-1H
8.603
8.587
8.356
8.326
7.962
7.958
7.935
7.930
7.559
7.555
7.543
7.535
7.527
7.515
7.510
7.505
7.450
7.444
7.428
7.423
7.418
7.400
7.396
7.261
7.127
7.119
7.103
7.095
7.073
7.065
7.050
7.037
7.021
7.005
3.885
MeO
O
N
N
N
2ab, Scheme 3
1.95
1.00
1.94
1.06
2.08
0.96
1.03
0.90
1.04
3.08
10.0 9.6 9.2 8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
f1 (ppm)
SK-MKM-3-29-13C.1.fid — SK-MKM-3-29-13C
187.761
158.004
156.141
138.248
137.787
133.351
132.735
129.581
128.884
128.433
117.232
116.600
115.580
114.999
103.668
77.580
77.157
76.733
55.865
f1 (ppm)

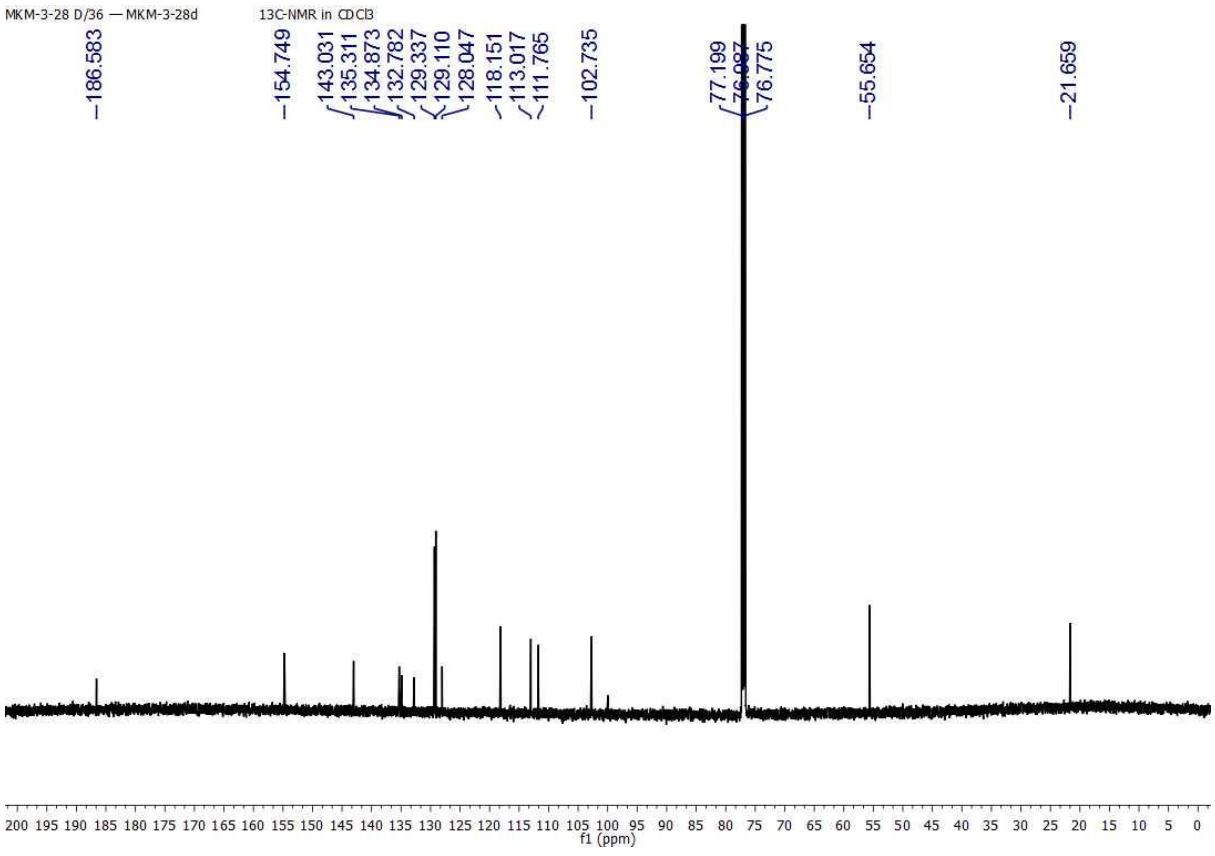

MKM-3-28/63 — MKM-3-28
1H-NMR in CDCl3
8.622
8.614
8.357
8.341
7.886
7.873
7.275
7.253
7.240
7.131
7.127
7.097
7.093
7.082
7.077
7.052
7.044
7.037
7.032
3.898
2.424
Me
MeO
N
O
N
N
2ac, Scheme 3
1.99
1.00
2.12
1.75
1.08
1.00
1.74
2.88
2.90
f1 (ppm)


MKM-3-28/64 — MKM-3-28
13C-NMR in CDCl3
187.391
157.860
157.182
155.881
143.456
137.714
135.383
133.095
129.651
129.010
128.699
117.075
116.260
115.351
114.533
103.392
77.206
76.781
55.686
21.671
f1 (ppm)


Manash obc/33 — MKM-3-29d 1H-NMR in CDCl3
9.201
8.001
7.989
7.647
7.635
7.622
7.560
7.547
7.534
7.400
7.385
7.107
7.102
7.097
7.087
7.083
7.072
7.068
3.871
MeO
N
H
O
3b, Scheme 3
1.24
2.22
1.31
2.16
1.00
2.89
3.06
f1 (ppm)


Manash obc/34 — MKM-3-29d 13C-NMR in CDCl3
186.881
154.796
138.001
134.726
132.906
132.261
129.160
128.412
128.040
118.378
113.044
112.204
102.748
77.196
76.774
55.650
f1 (ppm)


MKM-3-28 D/35 — MKM-3-28d
1H-NMR in CDCl3
9.208
7.926
7.912
7.394
7.379
7.354
7.340
7.275
7.108
7.104
7.095
7.092
7.090
7.076
7.072
7.061
7.057
3.871
2.483
Me
MeO
N
H
O
3c, Scheme 3
0.95
2.01
1.00
1.82
1.78
1.03
2.83
2.88
f1 (ppm)


MKM-3-28 D/36 — MKM-3-28d
13C-NMR in CDCl3
186.583
154.749
143.031
135.311
134.873
132.782
129.337
129.110
128.047
118.151
113.017
111.765
102.735
77.199
76.987
76.775
55.654
21.659
f1 (ppm)