RSC Advances

Supporting Information

Synthesis of isatins by the palladium-catalyzed intramolecular acylation of unactivated aryl C(sp^2)-H bond†

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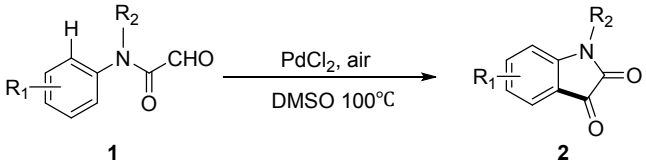
1. General details

All the reactions were monitored by thin-layer chromatography (TLC) and were visualized using UV light. The product purification was done using silica gel column chromatography. Thin layer chromatography (TLC) characterization was performed with precoated silica gel GF254 (0.2mm), while column chromatography characterization was performed with silica gel (100-200mesh).^{1}H and ^{13}C NMR spectra were recorded with tetramethylsilane as the internal standard. ^{1}H NMR spectra were recorded at 400 or 600 MHz (Varian) and ^{13}C NMR spectra were recorded 150 MHz (Varian). Chemical shifts are reported in ppm downfield from $CDCl_3$ (δ = 7.26 ppm) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0 ppm) for ^{13}C NMR spectroscopy. Coupling constants are given in Hz. Melting points were measured with YRT-3 melting point apparatus (Shantou Keyi Instrument & Equipment Co., Ltd., Shantou, China). High resolution mass spectroscopy data of the products were collected on a Waters Micromass GCT or a Bruker Apex IV FTMS instrument. All the N-methyl-2-oxo-N-phenylacetamide **1** were prepared according to the reported procedures.[1]

2. General procedure for the synthesis of 2 (2a as an example)

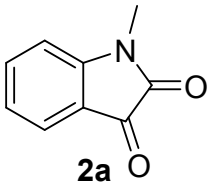


1 **2**

A mixture of N-methyl-2-oxo-N-phenylacetamide **1a** (0.4 mmol) and $PdCl_2$ (10 mol %) were added in 2 mL DMSO and then stirred under air at 100 ^{o}C for 3h. After the completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature, diluted with water and extracted with ethyl acetate. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate and the solvent was evaporated to dryness. The crude residue was purified by flash chromatography on silica (PE/EA=10/1) to afford pure 1-methylindoline-2,3-dione **2a** as a red solid (59.3 mg, 92% yield).

3. Spectroscopic characterization data of products

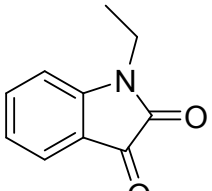
1-methylindoline-2,3-dione (2a)



2a

Yield 92%; red solid; mp. 130-133 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.58 (m, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.3, 158.1, 151.4, 138.4, 125.1, 123.8, 117.3, 109.9, 26.2; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_7NO_2$+Na^+ 184.0369, found 184.0370.

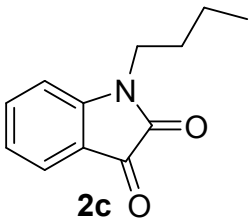
1-ethylindoline-2,3-dione (2b)



2b

Yield 93%; red solid; mp. 92-94 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.58 (m, 2H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 1H), 3.80 (q, *J* = 7.6 Hz, 2H), 1.32 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.3, 158.3, 151.4, 138.2, 125.2, 123.9, 117.0, 109.6, 36.0, 14.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_2$+Na^+ 198.0525, found 198.0528.

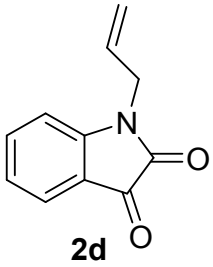
1-butylindoline-2,3-dione (2c)



2c

Yield 86%; red solid; mp. 35-36 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.57 (m, 2H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.90 (m, 1H), 3.76-3.71 (m, 2H), 1.73-1.64 (m, 2H), 1.58 (q, *J* = 7.6 Hz, 1H), 1.42 (q, *J* = 7.6 Hz, 1H), 0.99 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.4, 158.5, 151.3, 138.5, 125.1, 123.5, 116.9, 109.8, 43.1, 31.0, 20.7, 13.8; HRMS (ESI): m/z [M + Na^+] calcd for $C_{12}H_{13}NO_2$+Na^+ 226.0838, found 226.0842.

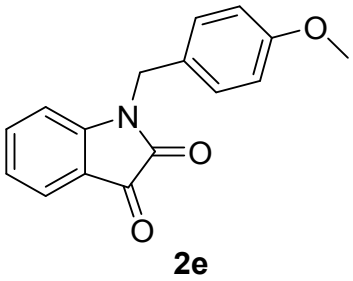
1-allylindoline-2,3-dione (2d)



2d

Yield 75%; red solid; mp. 89-90 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.62 (d, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.90 (d, *J* = 7.2 Hz, 1H), 5.85 (m, 1H), 5.30 (m, 2H), 4.37 (d, *J* = 5.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.2, 157.8, 150.8, 138.3, 130.3, 125.4, 123.8, 118.6, 117.5, 110.8, 42.5; HRMS (ESI): m/z [M + Na^+] calcd for $C_{11}H_9NO_2$+Na^+ 210.0525, found 210.0526.

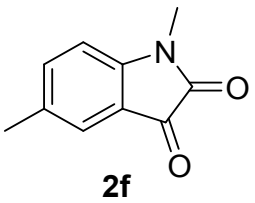
1-(4-methoxybenzyl)indoline-2,3-dione (2e)



2e

Yield 95%; red solid; mp. 169-171 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 7.2 Hz, 1H), 7.51-7.46 (m, 1H), 7.27 (d, *J* = 8.8 Hz, 2H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.0 Hz, 1H), 4.86 (s, 2H), 3.78 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.3, 159.4, 158.2, 150.7, 138.2, 128.9, 126.4, 125.3, 123.7, 117.6, 114.3, 111.0, 55.2, 43.5; HRMS (ESI): m/z [M + Na^+] calcd for $C_{16}H_{13}NO_3$+Na^+ 290.0788, found 290.0790.

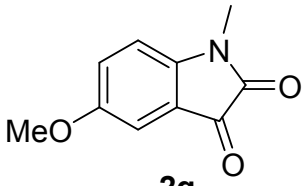
1,5-dimethylindoline-2,3-dione (2f)



2f

Yield 90%; red solid; mp. 149-150 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.28 (m, 2H), 6.79 (d, *J* = 8.0 Hz, 1H), 3.23 (s, 3H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.2, 158.7, 151.5, 150.7, 125.3, 124.4, 115.2, 110.8, 26.2, 22.9; HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_2$+Na^+ 198.0525, found 198.0525.

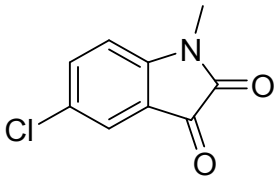
5-methoxy-1-methylindoline-2,3-dione (2g)



2g

Yield 93%; red solid; mp. 175-176 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17-7.15 (m, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 3.81 (s, 3H), 3.23 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.8, 158.2, 156.6, 145.7, 124.0, 117.8, 110.9, 109.8, 55.5, 26.1; HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_3$+Na^+ 214.0475, found 214.0476.

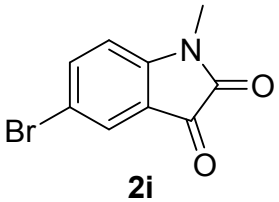
5-chloro-1-methylindoline-2,3-dione (2h)



2h

Yield 84%; red solid; mp. 171-173 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59-7.56 (m, 2H), 6.86 (d, *J* = 9.2 Hz, 1H), 3.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 181.5, 158.2, 152.2, 144.3, 126.4, 124.0, 115.2, 110.5, 26.5; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6ClNO_2$+Na^+ 217.9979, found 217.9982.

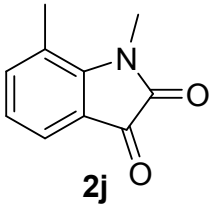
5-bromo-1-methylindoline-2,3-dione (2i)



2i

Yield 89%; red solid; mp. 163-164 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74-7.70 (m, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.1, 157.4, 150.1, 140.6, 128.0, 118.5, 116.6, 111.6, 26.3; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6BrNO_2$+Na^+ 261.9474, found 261.9472.

1,7-dimethylindoline-2,3-dione (2j)

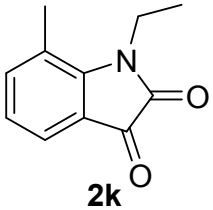


2j

Yield 90%; red solid; mp. 162-164 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 7.6 Hz, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.01 (t, *J* = 7.6 Hz, 1H), 3.53 (s, 3H), 2.57 (s, 3H); ^{13}C NMR

($CDCl_3$, 150 MHz): δ 182.7, 158.0, 151.8, 150.5, 125.5, 124.5, 115.5, 110.9, 26.1, 22.9; HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_2$+Na^+ 198.0525, found 198.0522.

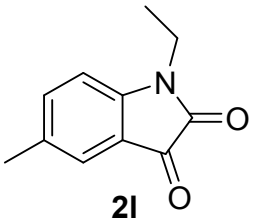
1-ethyl-7-methylindoline-2,3-dione (2k)



2k

Yield 87%; red solid; mp. 188-190 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.01 (t, *J* = 7.6 Hz, 1H), 4.01 (q, *J* = 7.2 Hz, 2H), 2.54 (s, 3H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 184.1, 159.1, 148.4, 142.3, 123.7, 123.5, 121.4, 118.8, 36.9, 18.7, 14.5; HRMS (ESI): m/z [M + Na^+] calcd for $C_{11}H_{11}NO_2$+Na^+ 212.0682, found 212.0685.

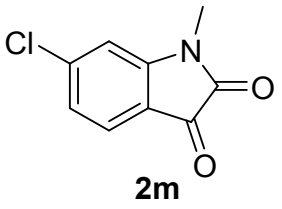
1-ethyl-5-methylindoline-2,3-dione (2l)



2l

Yield 88%; red solid; mp. 74-76 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 7.42-7.38 (m, 2H), 6.80 (d, *J* = 8.0 Hz, 1H), 3.81 (q, *J* = 7.2 Hz, 2H), 2.39 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 184.0, 159.2, 148.3, 142.5, 123.2, 123.1, 121.7, 118.2, 36.9, 18.7, 14.5; HRMS (ESI): m/z [M + Na^+] calcd for $C_{11}H_{11}NO_2$+Na^+ 212.0682, found 212.0684.

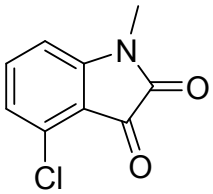
6-chloro-1-methylindoline-2,3-dione (2m)



2m

Yield (2m +2m') total 81%; red solid; mp. 177-178 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.0 Hz, 1H), 7.12 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1H), 6.91 (d, *J* = 1.6 Hz, 1H), 3.27 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 181.7, 158.1, 152.4, 144.8, 126.3, 124.0, 115.7, 110.8, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6ClNO_2$+Na^+ 217.9979, found 217.9983.

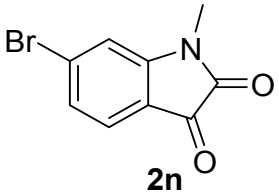
4-chloro-1-methylindoline-2,3-dione (2m')



2m'

red solid; mp. 192-195 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 7.6 Hz, 1H), 3.27 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 181.1, 158.3, 152.3, 144.5, 126.2, 124.0, 115.7, 110.5, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6ClNO_2$+Na^+ 217.9979, found 217.9981.

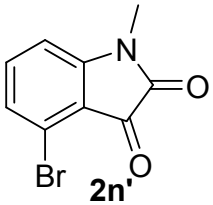
6-bromo-1-methylindoline-2,3-dione (2n)



2n

Yield (2n +2n') total 87%; red solid; mp. 118-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 7.6 Hz, 1H), 7.30 (dd, J_1 = 1.2 Hz, J_2 = 7.6 Hz, 1H), 7.09 (d, *J* = 1.2 Hz, 1H), 3.25 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.3, 157.4, 150.2, 140.6, 128.1, 118.6, 116.5, 111.7, 26.3;HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6BrNO_2$+Na^+ 261.9474, found 261.9470.

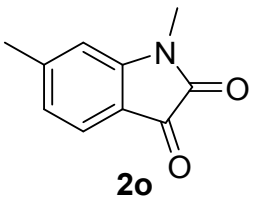
4-bromo-1-methylindoline-2,3-dione (2n')



2n'

red solid; mp. 199-200 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.43 (t, *J* = 8.4 Hz, 1H), 7.26 (d, *J* = 7.8 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 3.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.0, 157.2, 150.3, 140.3, 128.1, 118.5, 116.6, 111.2, 26.3; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6BrNO_2$+Na^+ 261.9474, found 261.9475.

1,6-dimethylindoline-2,3-dione (2o)

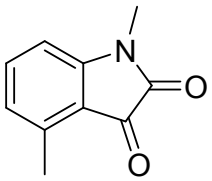


2o

Yield (2o +2o') total 88%; red solid; mp. 150-151 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (d, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.70 (s, 1H), 3.32 (s, 3H), 2.40 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.6, 158.9, 151.8, 150.7, 125.3, 124.4, 115.3, 110.7, 26.1, 22.9;

HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_2$+Na^+ 198.0525, found 198.0527.

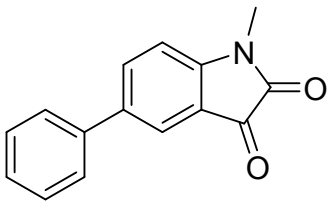
1,4-dimethylindoline-2,3-dione (2o')



2o'

red solid; mp. 164-165 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (t, *J* = 8.0 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 3.24 (s, 3H), 2.57 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.8, 158.8, 151.8, 150.7, 125.4, 124.4, 115.5, 110.8, 26.1, 22.9; HRMS (ESI): m/z [M + Na^+] calcd for $C_{10}H_9NO_2$+Na^+ 198.0525, found 198.0528.

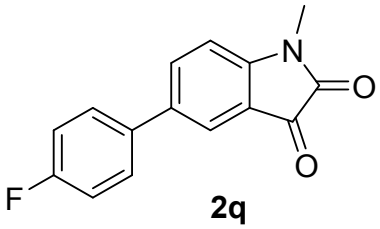
1-methyl-5-phenylindoline-2,3-dione (2p)



2p

Yield 80%; red solid; mp. 182-184 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.82 (m, 2H), 7.54-7.51 (m, 2H), 7.47-7.44 (m, 2H), 7.40-7.37 (m, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 3.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.3, 160.1, 150.5, 139.3, 137.2, 136.5, 129.2, 127.9, 126.6, 123.5, 117.8, 110.5, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_{11}NO_2$+Na^+ 260.0682, found 260.0686.

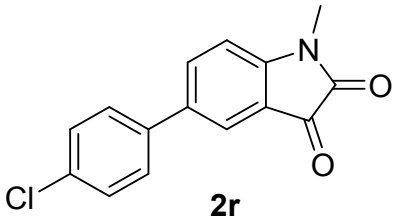
5-(4-fluorophenyl)-1-methylindoline-2,3-dione (2q)



2q

Yield 77%; red solid; mp. 136-139 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.79-7.75 (m, 2H), 7.50-7.46 (m, 2H), 7.14 (t, *J* = 8.8 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 1H), 3.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ183.3, 162.1(d, J_{C-F}=246.8Hz), 158.2, 150.4, 136.6, 136.3, 135.1, 128.2(d, J_{C-F}=7.6Hz), 123.5, 117.8, 115.9(d, J_{C-F}=21.4Hz), 110.3, 26.3; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_{10}FNO_2$+Na^+ 278.0588, found 278.0587.

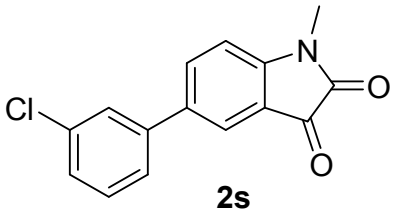
5-(4-chlorophenyl)-1-methylindoline-2,3-dione (2r)



2r

Yield 82%; red solid; mp. 180-182 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1H), 7.60 (d, J = 1.6 Hz, 1H), 7.46-7.40 (m, 4H), 6.98 (d, J = 8.0 Hz, 1H), 3.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.2, 158.2, 150.6, 137.4, 136.6, 136.0, 134.0, 129.2, 127.8, 123.5, 117.8, 110.4, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_{10}ClNO_2$+Na^+ 294.0292, found 294.0290.

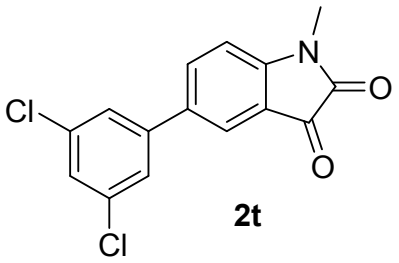
5-(3-chlorophenyl)-1-methylindoline-2,3-dione (2s)



2s

Yield 79%; red solid; mp. 188-189 °C;; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81-7.78 (m, 2H), 7.49 (s, 1H), 7.39-7.35 (m, 3H), 6.98 (d, J = 8.0 Hz, 1H), 3.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.2, 158.2, 150.8, 140.7, 136.7, 135.9, 135.0, 130.3, 127.9, 126.7, 124.7, 123.7, 117.9, 110.4, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_{10}ClNO_2$+Na^+ 294.0292, found 294.0294.

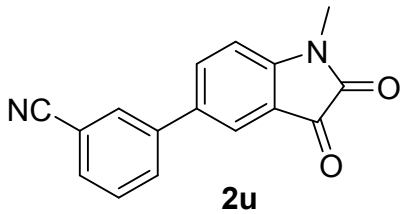
5-(3,5-dichlorophenyl)-1-methylindoline-2,3-dione (2t)



2t

Yield 72%; red solid; mp. 228-230 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.08 (dd, J_1 = 1.6 Hz, J_2 = 8.4 Hz, 1H), 7.94 (d, J = 1.6 Hz, 1H), 7.79 (d, J = 1.2 Hz, 2H), 7.57 (d, J = 1.6 Hz, 1H), 7.24 (d, J = 8.4 Hz, 1H), 3.18 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 183.3, 158.6, 151.6, 142.3, 136.8, 134.9, 132.4, 127.0, 125.2, 122.9, 118.2, 111.3, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_9Cl_2NO_2$+Na^+ 327.9903, found 327.9904.

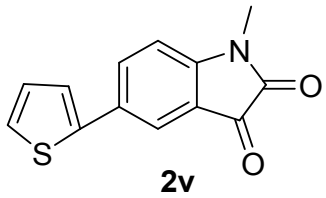
3-(1-methyl-2,3-dioxoindolin-5-yl)benzonitrile (2u)



2u

Yield 74%; red solid; mp. 236-237 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.22 (s, 1H), 8.10-8.04 (m, 2H), 7.94 (d, J = 1.6 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.65 (t, J = 8.0 Hz, 1H), 7.27 (d, J = 8.0 Hz, 1H), 3.20 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 183.4, 158.5, 151.4, 139.8, 136.6, 133.2, 131.2, 130.3, 130.1, 122.7, 118.9, 118.3, 112.3, 111.3, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{16}H_{10}N_2O_2$+Na^+ 285.0634, found 285.0637.

1-methyl-5-(thiophen-2-yl)indoline-2,3-dione (2v)



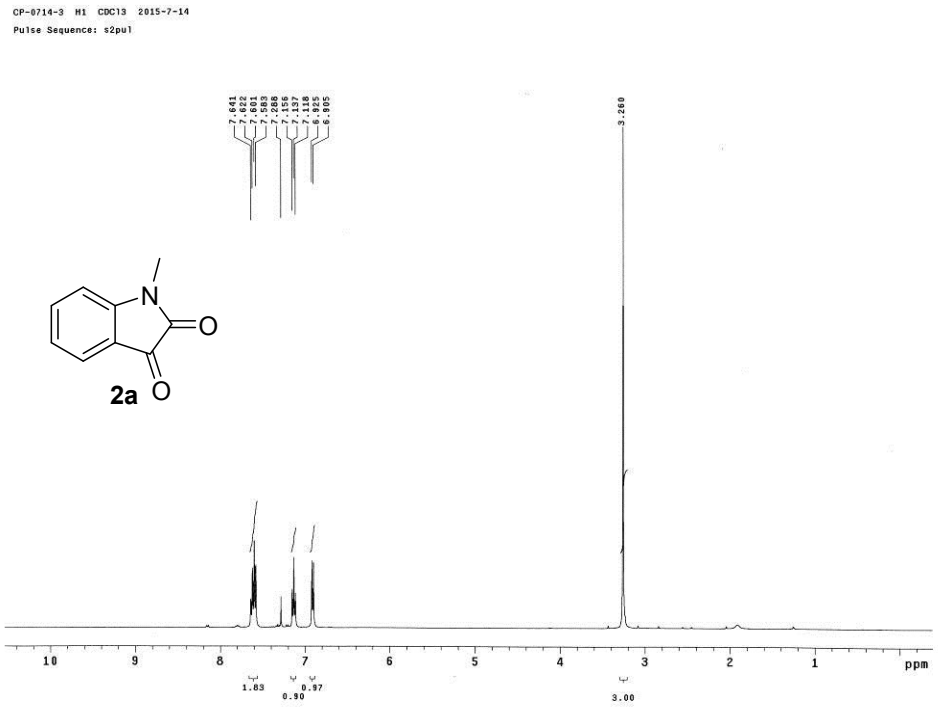
2v

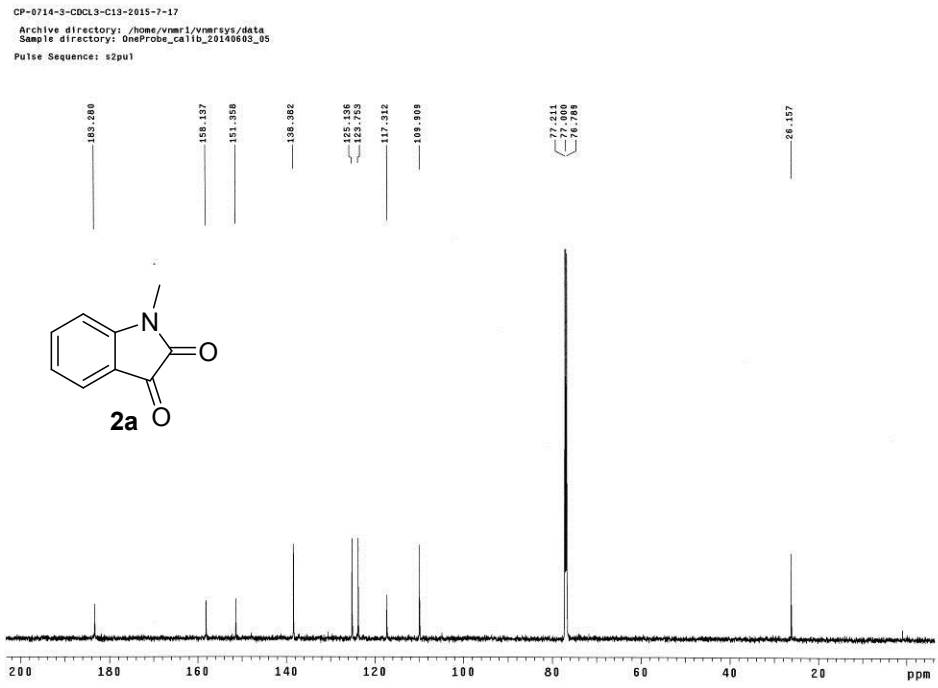
Yield 76%; red solid; mp. 206-208 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 1.6 Hz, 1H), 7.82 (s, 1H), 7.30 (dd, J_1 = 0.8 Hz, J_2 = 4.0 Hz, 1H), 7.27 (d, J = 2.4 Hz, 1H), 7.10-7.08 (m, 1H), 6.92 (d, J = 8.8 Hz, 1H), 3.28 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.1, 158.1, 150.3, 142.3, 135.2, 130.9, 128.2, 125.3, 123.1, 122.4, 117.8, 103.1, 26.4; HRMS (ESI): m/z [M + Na^+] calcd for $C_{13}H_9NO_2S$+Na^+ 266.0246, found 266.0250.

4. References

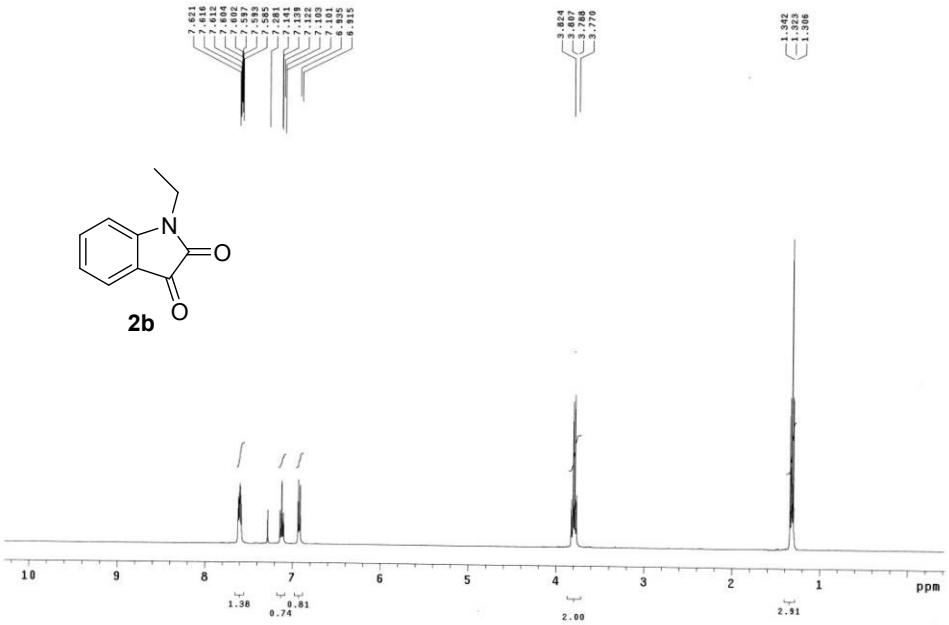
[1] Marc, M.; Johannes, C.; Vogel, W. T. *Org. Biomol. Chem*., **2009**, *7*, 589.

5. ^{1}H NMR and ^{13}C NMR Spectra of Products

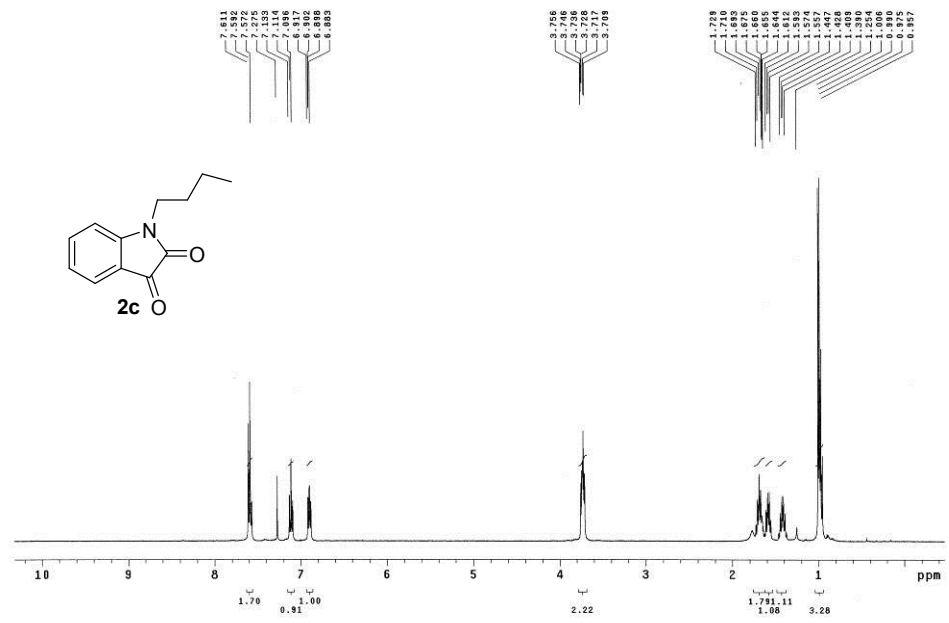


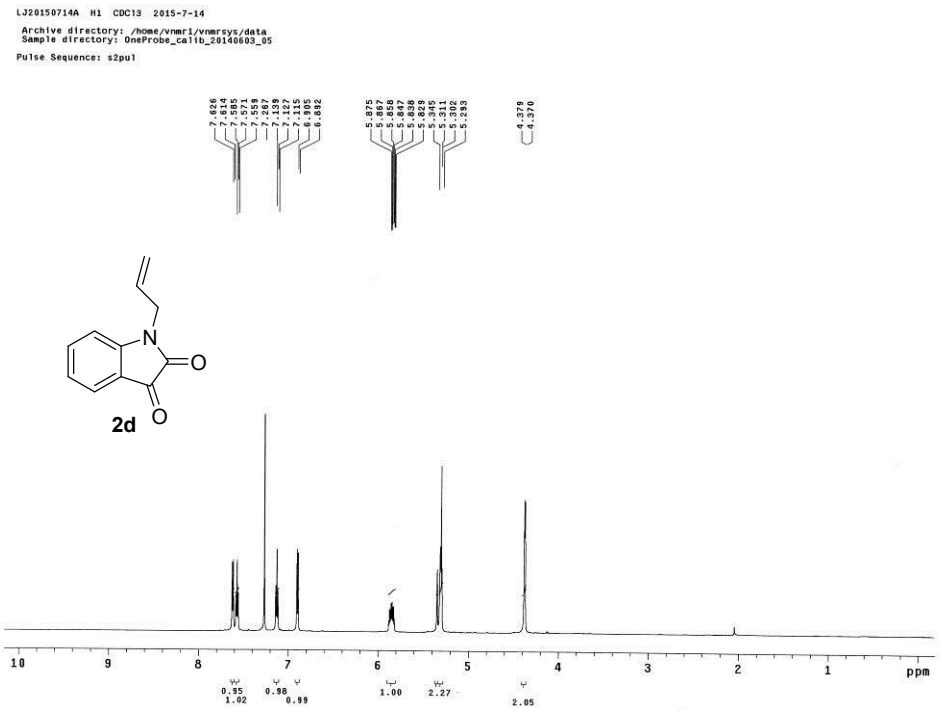


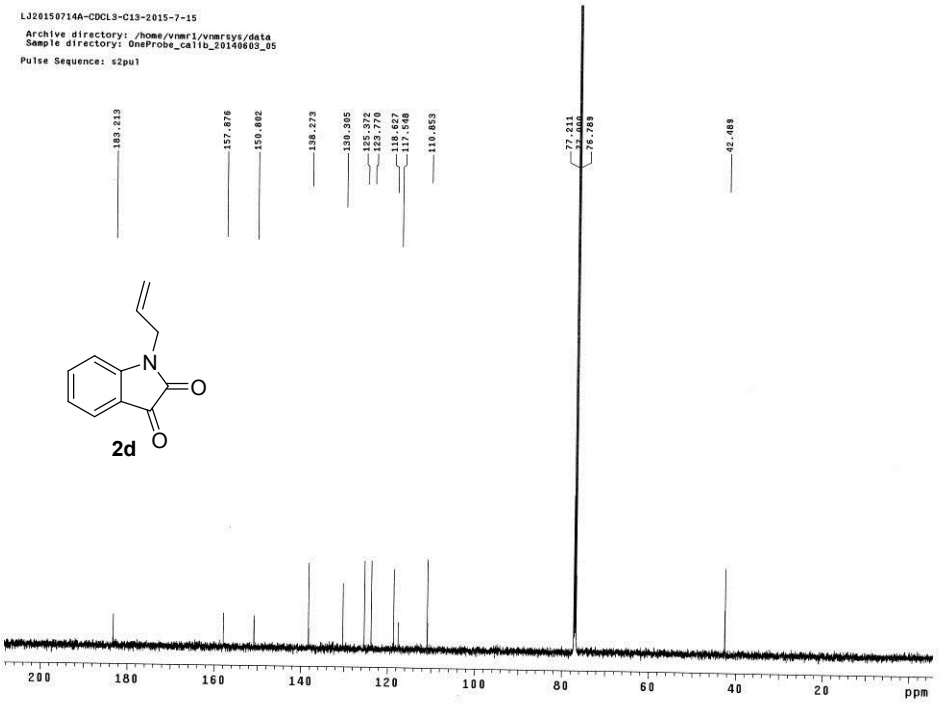
LJ-11 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul

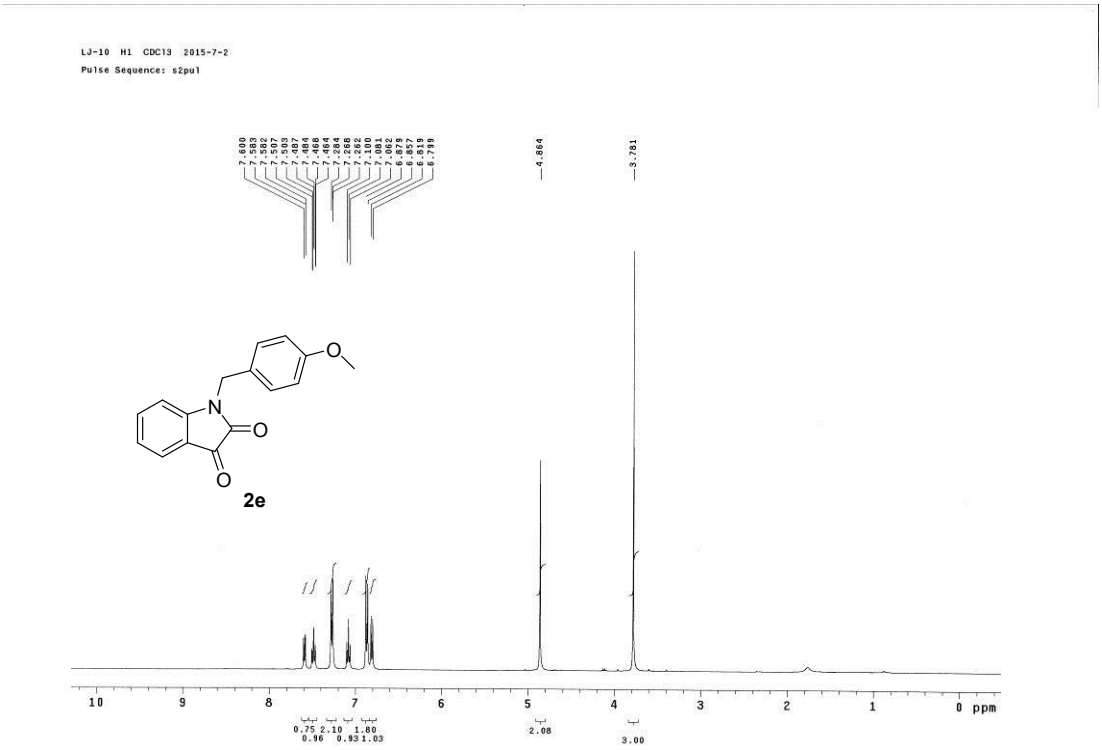


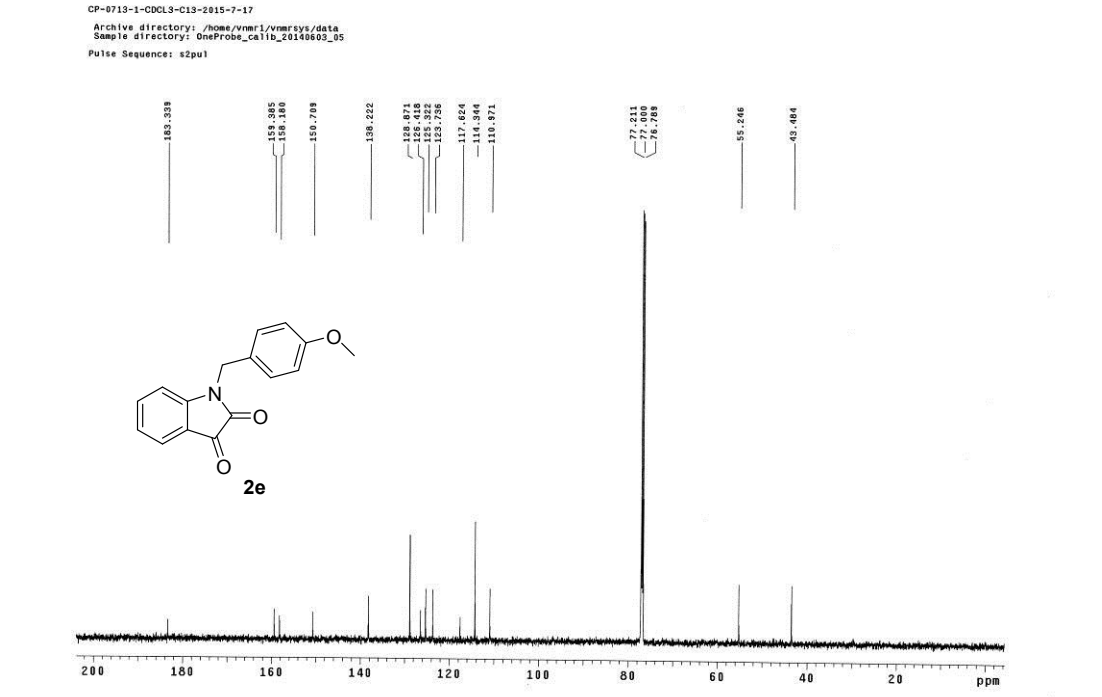
LJ-19 H1 CDCl3 2015-7-8
Pulse Sequence: s2pul

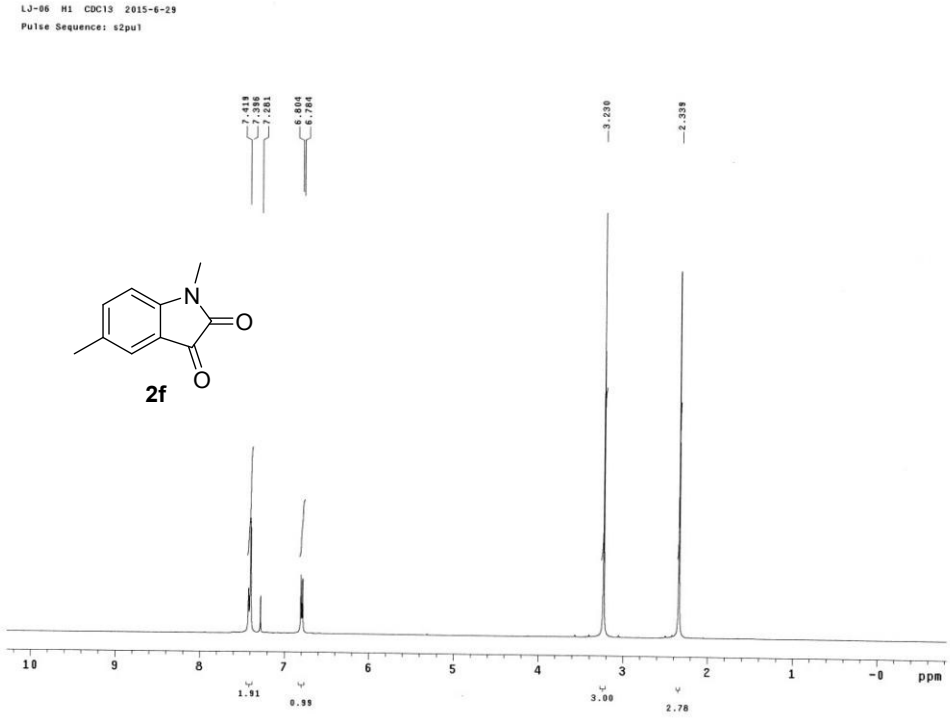


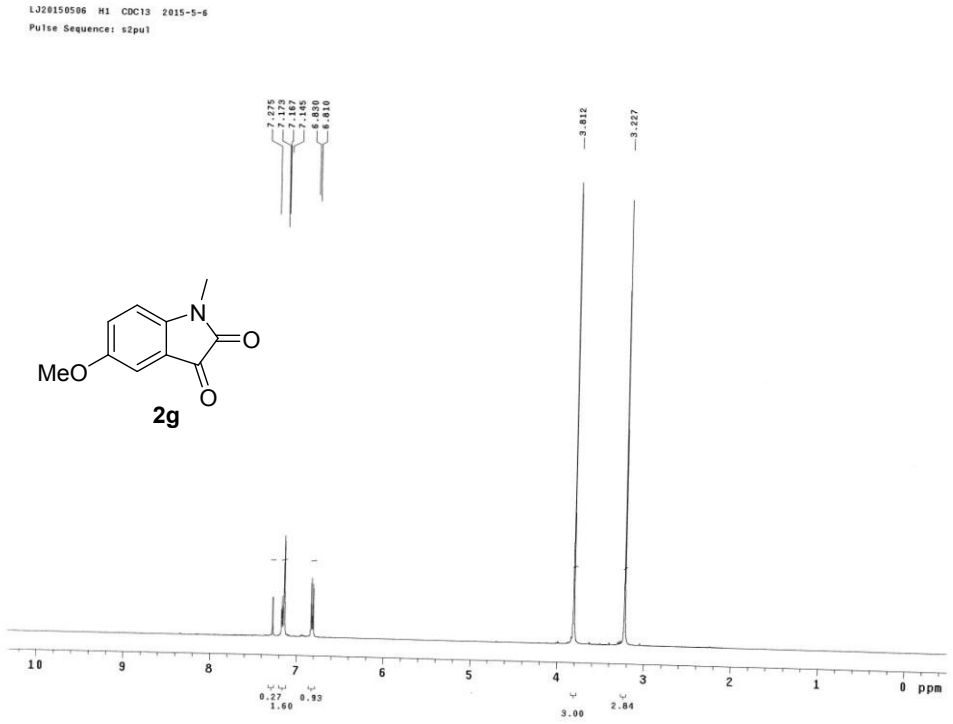

LJ20150714A H1 CDCl3 2015-7-14
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
7.626
7.614
7.585
7.571
7.559
7.207
7.139
7.127
7.115
6.895
6.892
5.875
5.867
5.856
5.847
5.838
5.829
5.585
5.311
5.302
5.293
4.378
4.370
2d
10
9
8
7
6
5
4
3
2
1
ppm
0.95
1.02
0.98
0.99
1.00
2.27
2.05

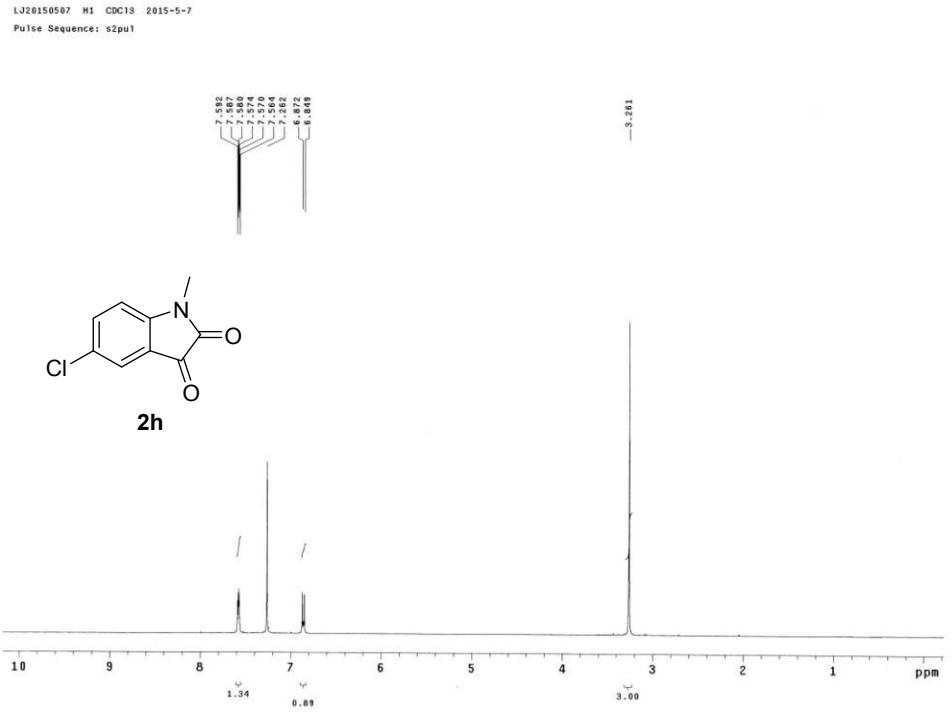

LJ20150714A-CDCL3-C13-2015-7-15
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.213
157.876
150.602
138.273
130.305
125.372
123.770
118.627
117.548
110.653
77.211
76.789
42.489
2d
200
180
160
140
120
100
80
60
40
20
ppm


LJ-10 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
7.600
7.593
7.582
7.507
7.503
7.487
7.466
7.464
7.284
7.268
7.262
7.100
7.081
7.062
6.879
6.857
6.838
6.799
4.864
3.701
O
N
O
O
2e
10
9
8
7
6
5
4
3
2
1
0 ppm
0.75 2.10 1.80
0.96 0.93 1.03
2.08
3.00

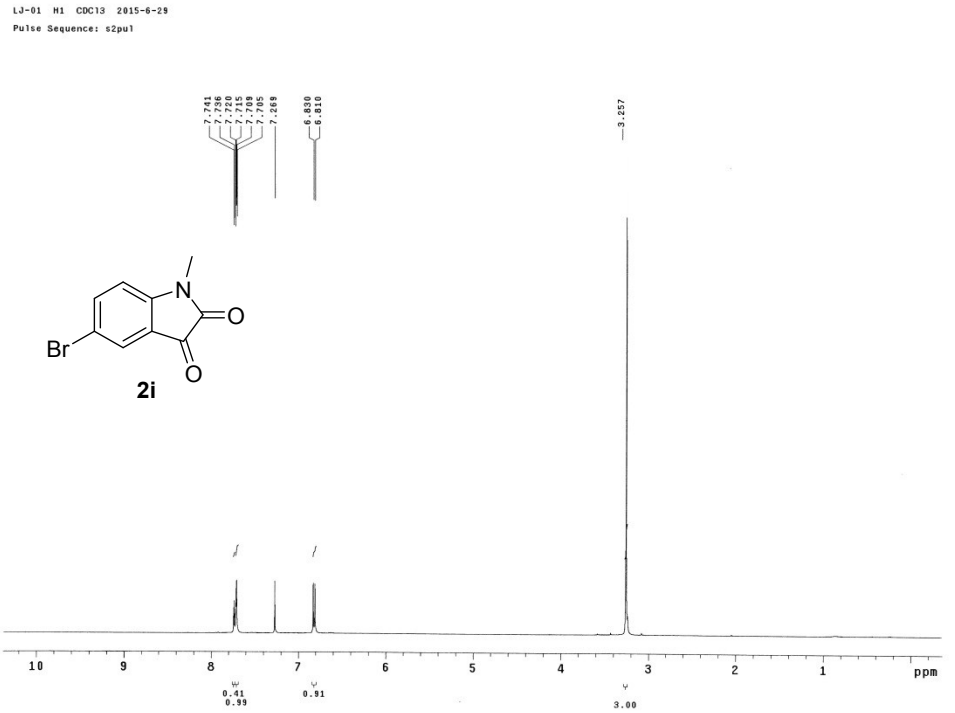

CP-0713-1-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.339
159.385
158.160
150.709
138.222
128.871
126.418
125.322
123.736
117.624
114.344
110.971
77.211
77.000
76.789
55.246
43.484
O
N
O
O
2e
200
180
160
140
120
100
80
60
40
20
ppm


LJ-06 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.419
7.386
7.261
6.804
6.784
3.230
2.339
2f
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.91
0.99
3.00
2.78

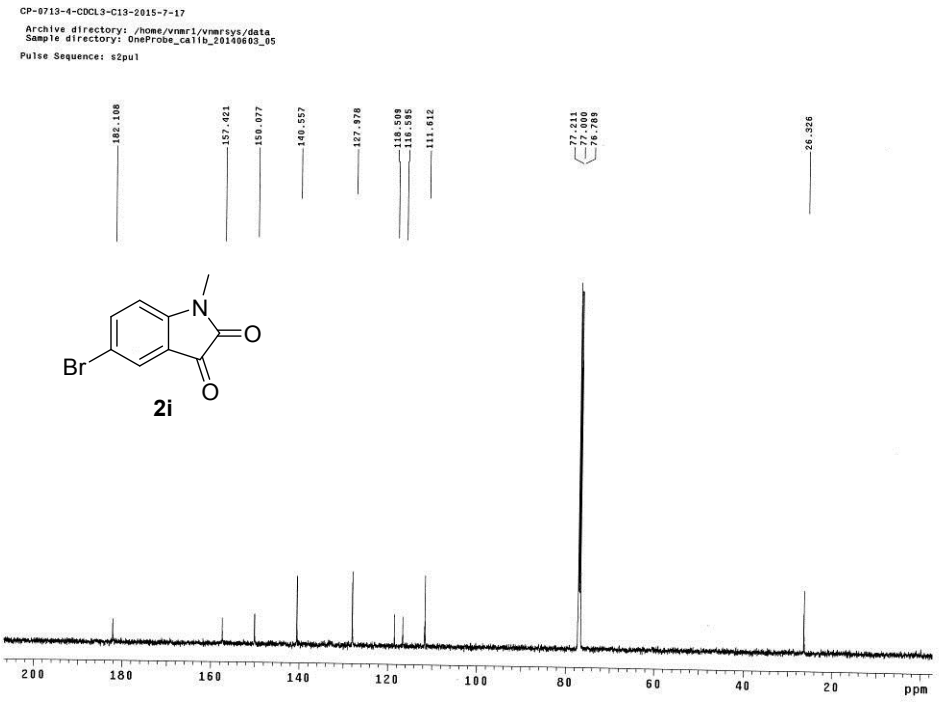

LJ20150506 H1 CDCl3 2015-5-6
Pulse Sequence: s2pul
7.275
7.173
7.167
7.146
6.830
6.810
3.812
3.227
MeO
2g
10
9
8
7
6
5
4
3
2
1
0 ppm
0.27
1.60
0.93
3.00
2.84

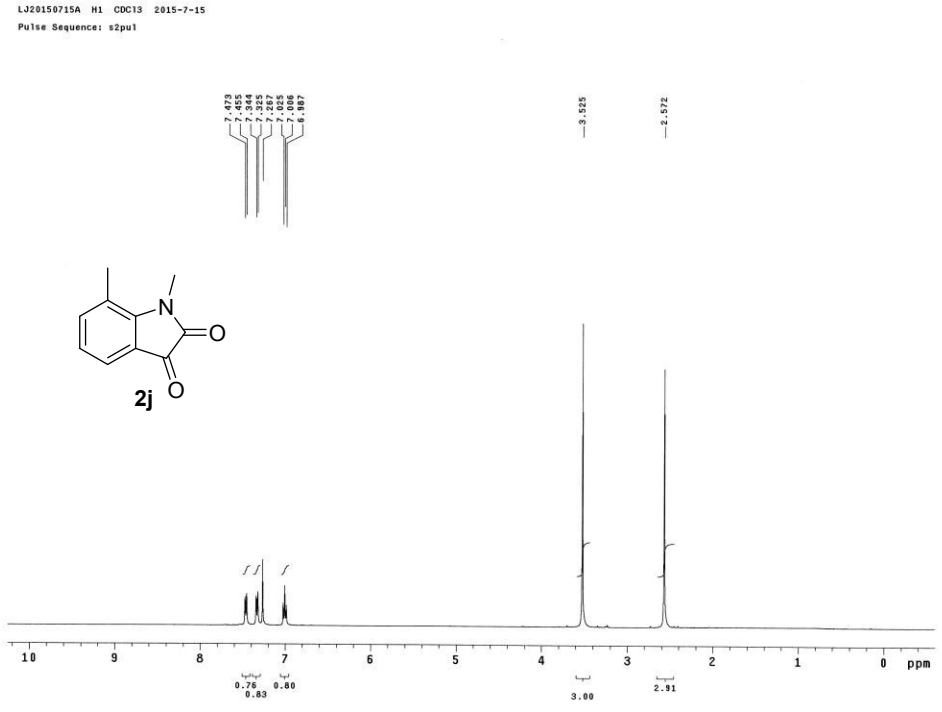

LJ20150507 H1 CDCl3 2015-5-7
Pulse Sequence: s2pul
7.592
7.587
7.580
7.574
7.570
7.564
7.262
6.872
6.849
3.261
N
O
O
Cl
10
9
8
7
6
5
4
3
2
1
ppm
1.34
0.89
3.00

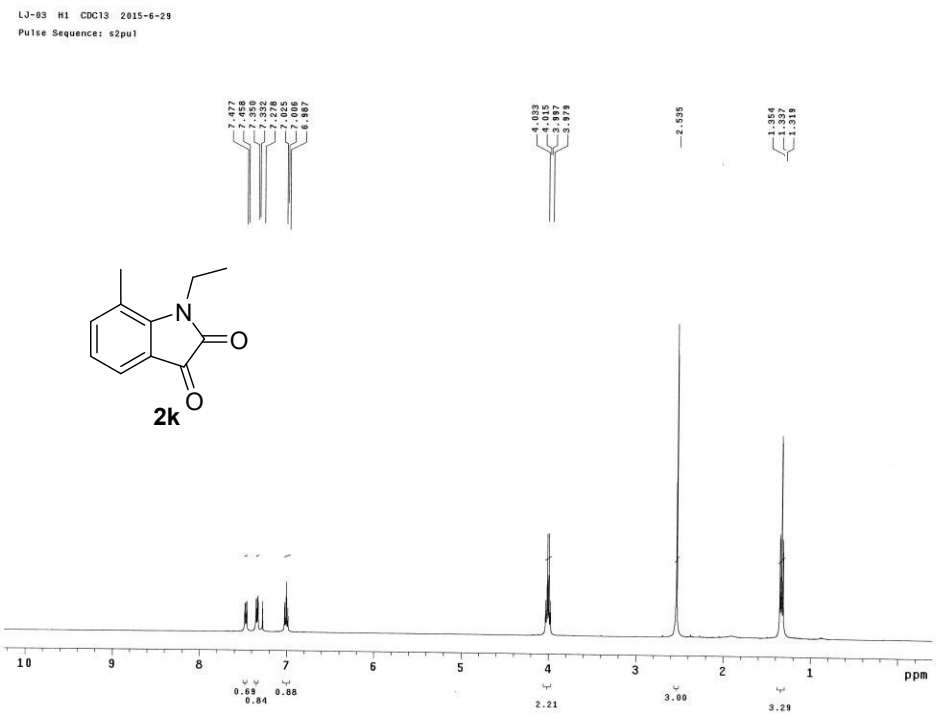
2h

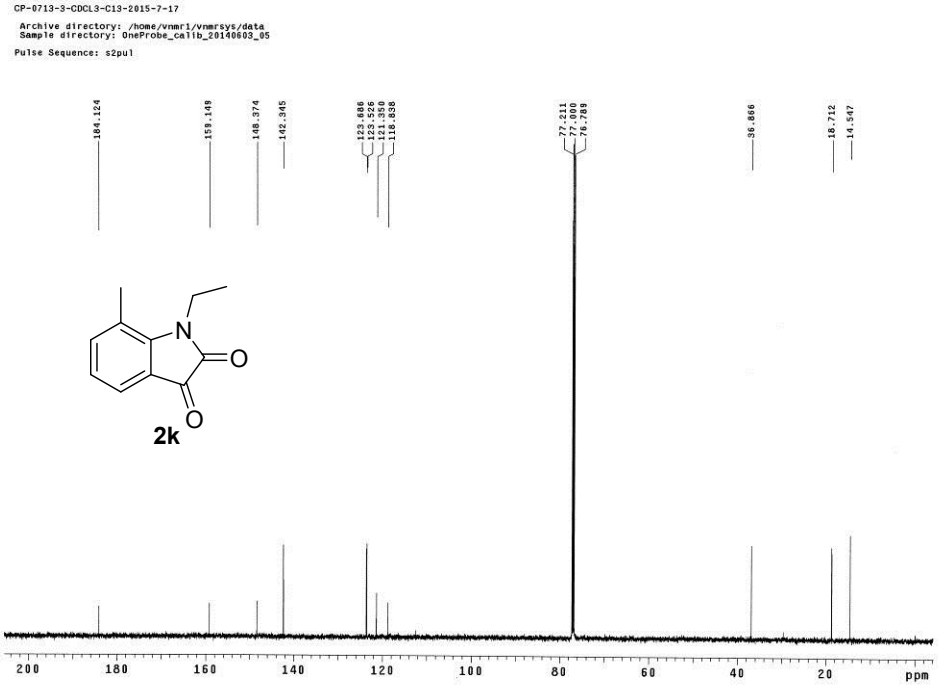

LJ-01 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.741
7.736
7.720
7.715
7.708
7.705
7.269
6.830
6.810
3.257
N
O
O
Br
10
9
8
7
6
5
4
3
2
1
ppm
0.41
0.99
0.91
3.00

2i


CP-0713-4-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
182.108
157.421
150.077
140.557
127.978
118.598
118.585
111.612
77.211
77.000
76.789
26.326
N
O
O
Br
2i
200
180
160
140
120
100
80
60
40
20
ppm

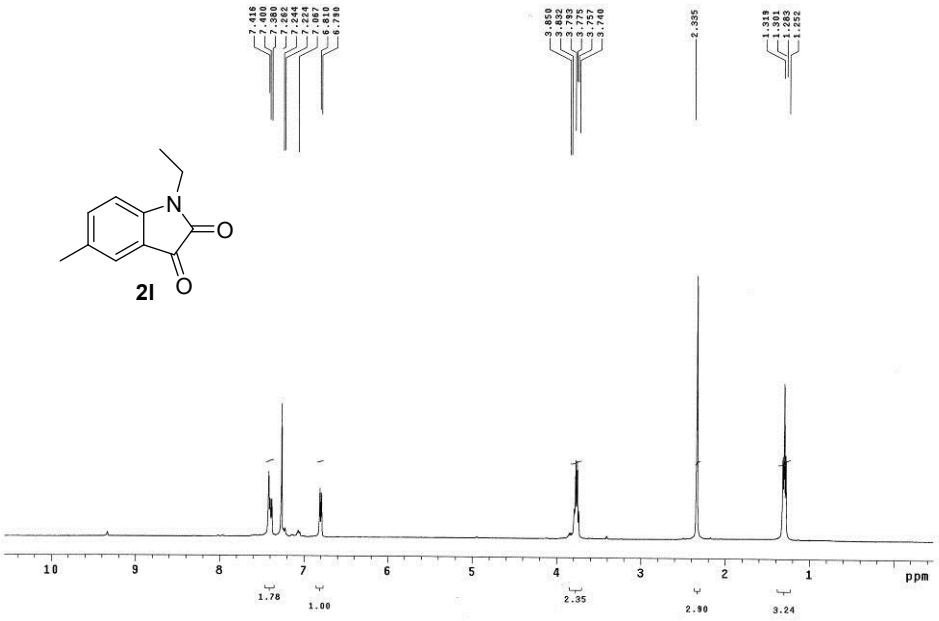

LJ20150715A H1 CDCl3 2015-7-15
Pulse Sequence: s2pul
7.473
7.465
7.344
7.325
7.267
7.035
7.006
6.987
3.525
2.572
N
O
O
2j
10
9
8
7
6
5
4
3
2
1
0
ppm
0.76
0.83
0.80
3.00
2.91


LJ-03 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.477
7.458
7.350
7.332
7.278
7.025
7.006
6.987
4.033
4.015
3.997
3.979
2.535
1.354
1.337
1.319
2k
10
9
8
7
6
5
4
3
2
1
ppm
0.69
0.84
0.88
2.21
3.00
3.29


CP-0713-3-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
184.124
159.149
148.374
142.345
123.686
123.526
121.350
118.838
77.211
77.000
76.789
36.866
16.712
14.547
2k
200
180
160
140
120
100
80
60
40
20
ppm

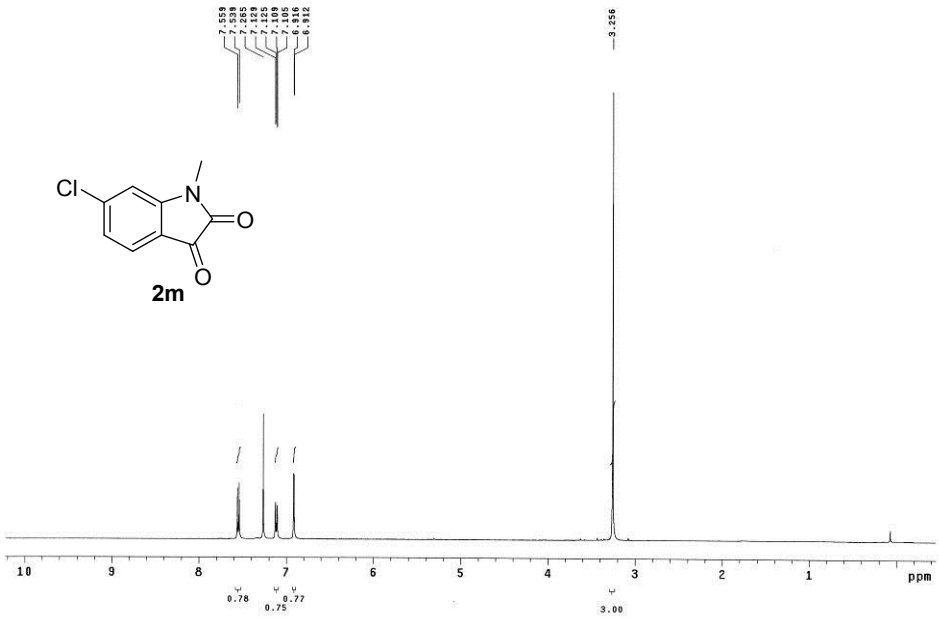
LJ-14-CDCL3-H1-2015-7-7
Pulse Sequence: s2pul

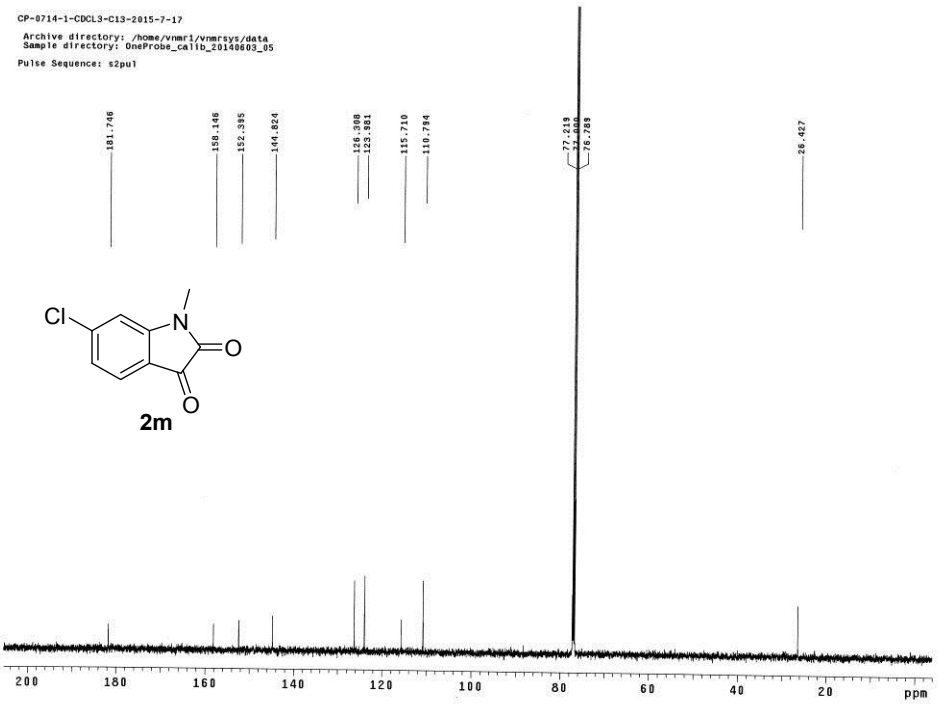
2l

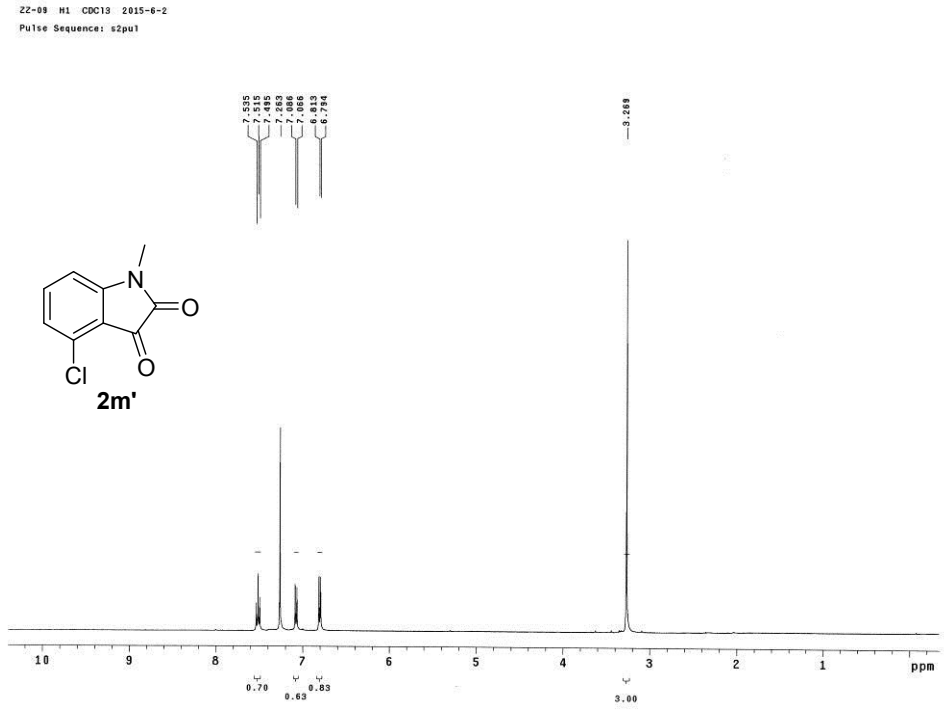


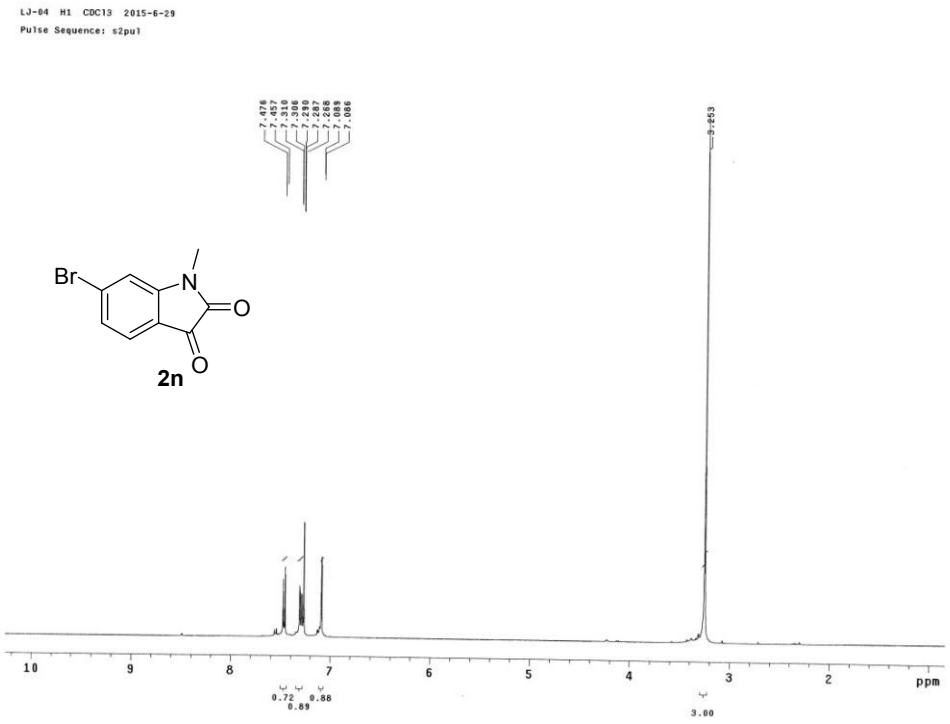
ZZ-08 H1 CDCl3 2015-6-2
Pulse Sequence: s2pul

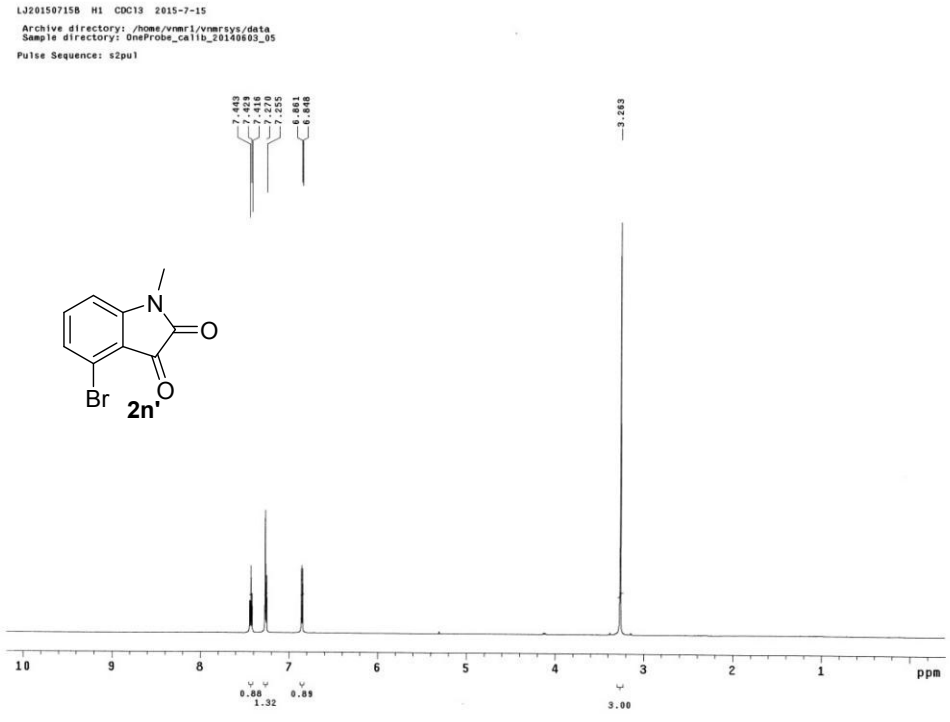
2m

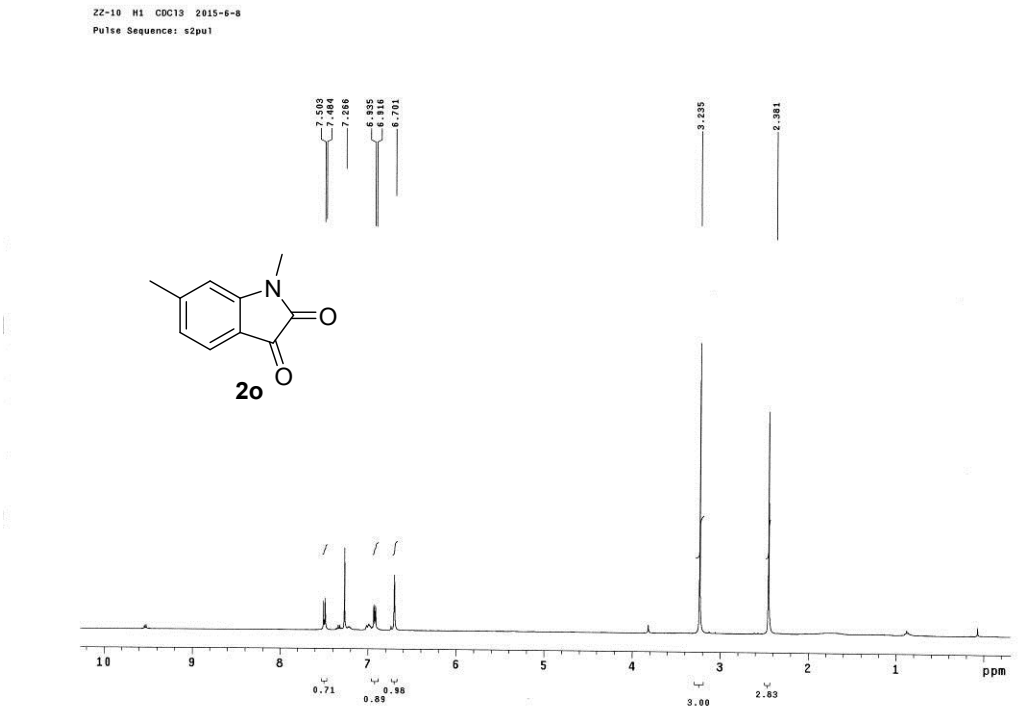


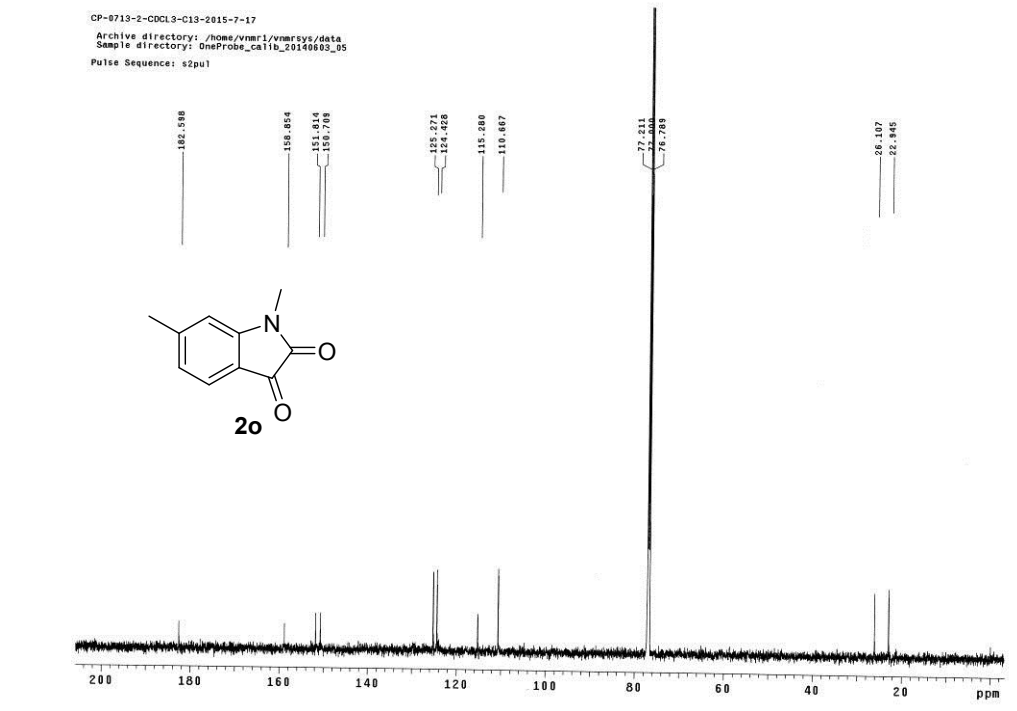

CP-0714-1-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
181.746
158.146
152.395
144.824
126.308
123.981
115.710
110.784
77.219
77.000
76.788
28.427
Cl
N
O
O
2m
200
180
160
140
120
100
80
60
40
20
ppm

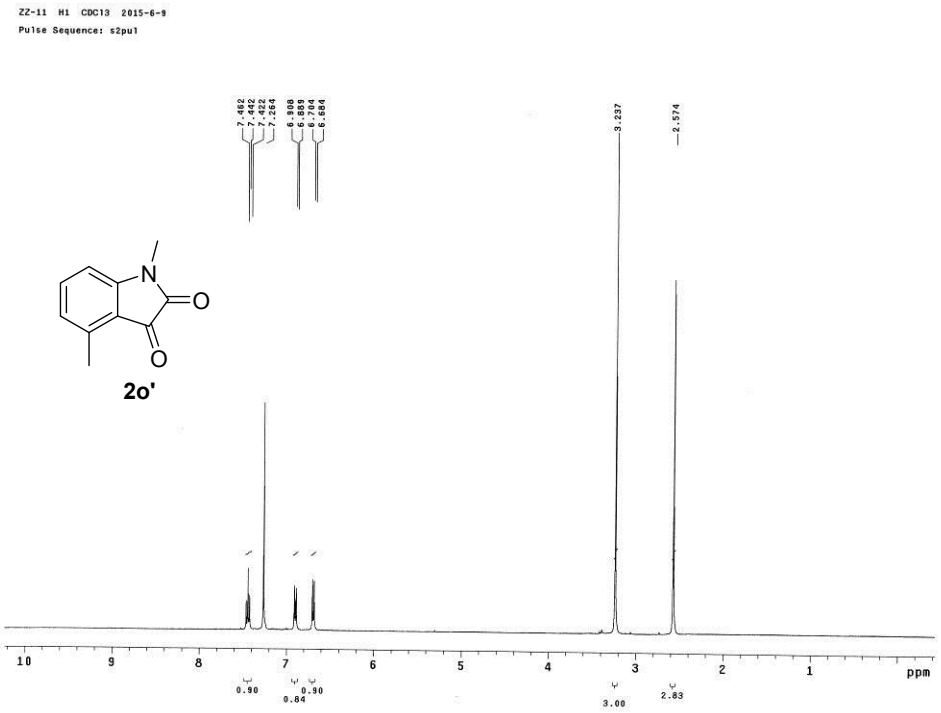

ZZ-09 H1 CDCl3 2015-6-2
Pulse Sequence: s2pul
7.535
7.515
7.495
7.088
7.066
6.813
6.794
3.269
N
O
O
Cl
2m'
10
9
8
7
6
5
4
3
2
1
ppm
0.70
0.63
0.83
3.00

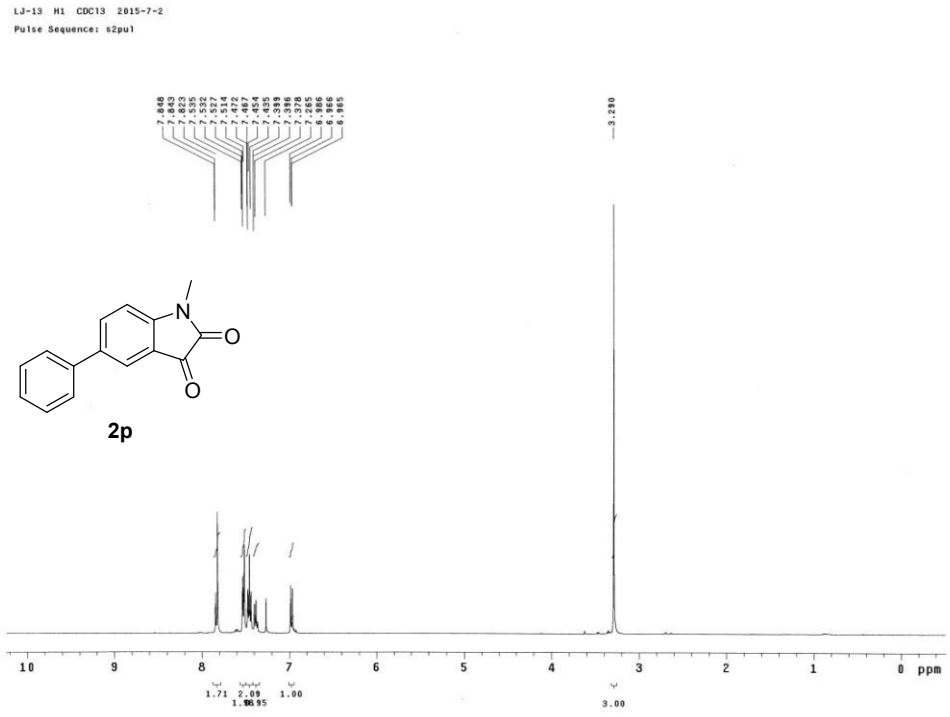

LJ-04 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.476
7.457
7.310
7.306
7.289
7.287
7.268
7.089
7.086
3.253
Br
N
O
O
2n
10
9
8
7
6
5
4
3
2
ppm
0.72
0.89
0.88
3.00

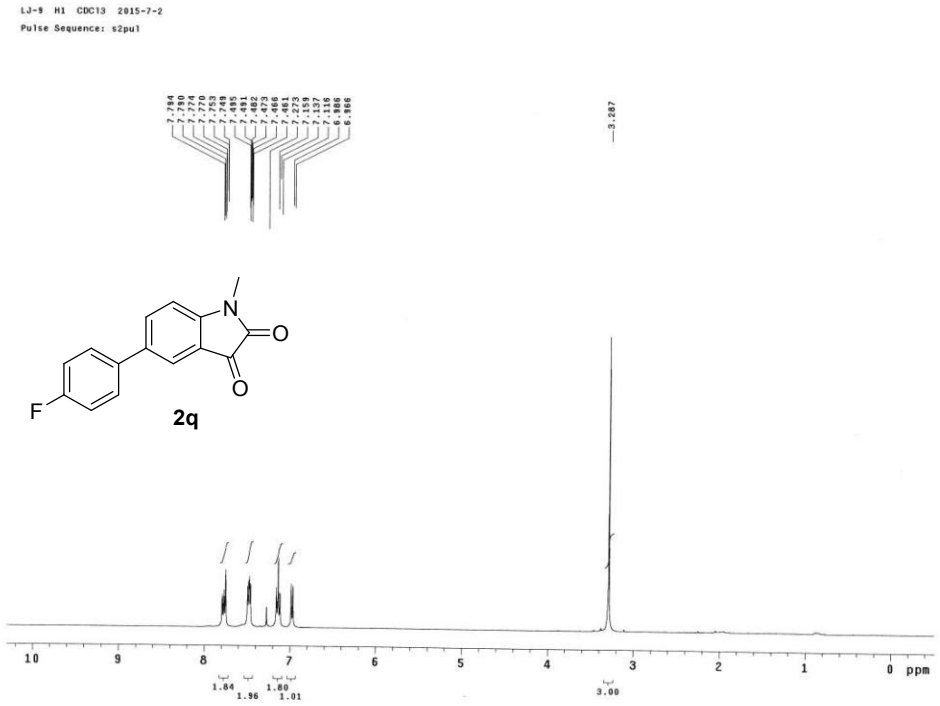

LJ20150715B H1 CDCl3 2015-7-15
Archive directory: /home/vnmrj/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
7.449
7.429
7.416
7.270
7.255
6.881
6.848
3.263
N
O
O
Br
2n'
10
9
8
7
6
5
4
3
2
1
ppm
0.88
1.32
0.89
3.00

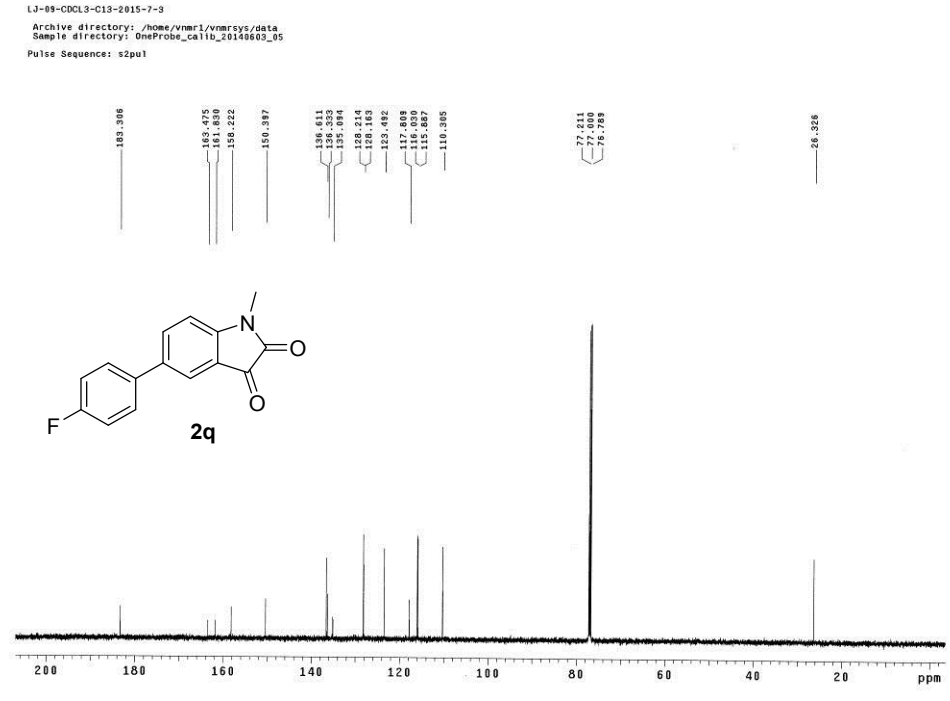

ZZ-10 H1 CDCl3 2015-6-8
Pulse Sequence: s2pul
7.503
7.484
7.266
6.835
6.816
6.701
3.235
2.381
2o
10
9
8
7
6
5
4
3
2
1
ppm
0.71
0.89
0.98
3.00
2.83

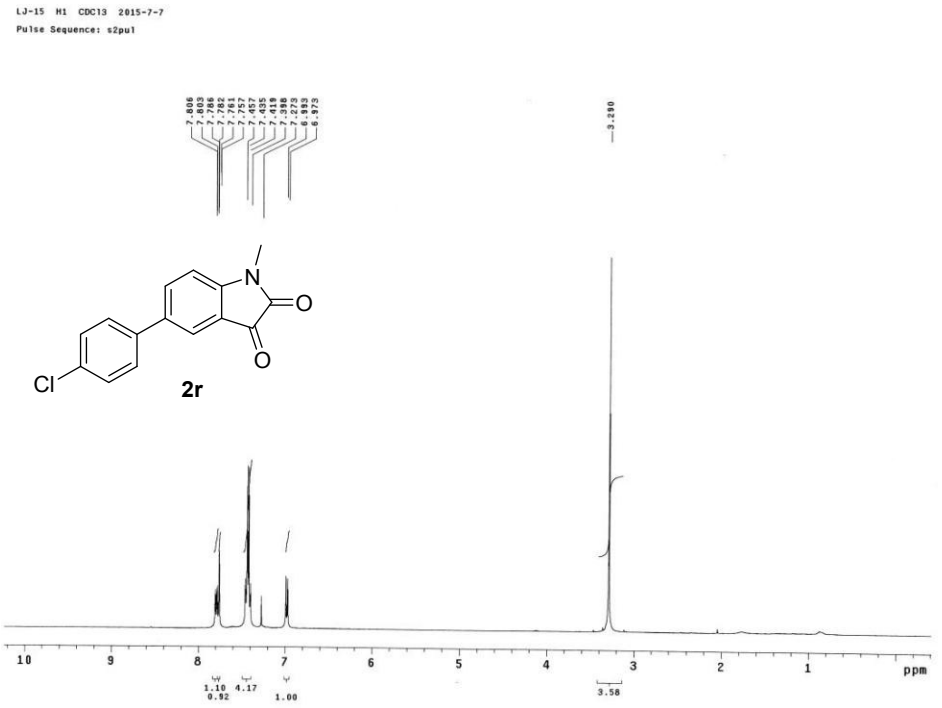

CP-0713-2-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
182.598
158.854
151.614
150.709
125.271
124.428
115.280
110.667
77.211
77.000
76.789
26.107
22.945
2o
200
180
160
140
120
100
80
60
40
20
ppm

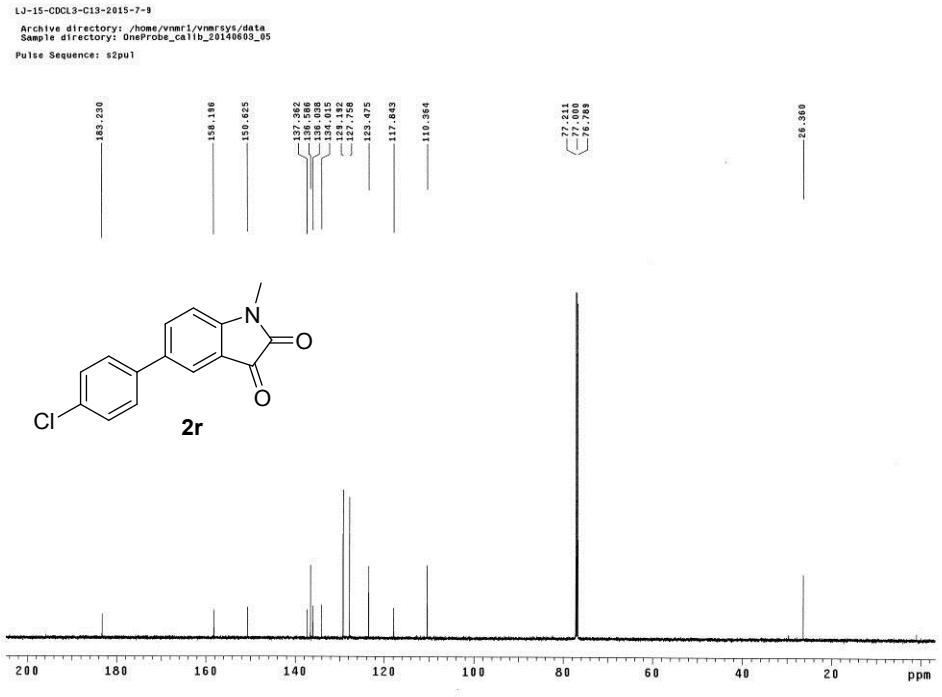

ZZ-11 H1 CDCl3 2015-6-9
Pulse Sequence: s2pul
7.482
7.462
7.422
7.264
6.888
6.884
6.704
6.684
3.237
2.574
N
O
O
2o'
10
9
8
7
6
5
4
3
2
1
ppm
0.90
0.84
0.90
3.00
2.83

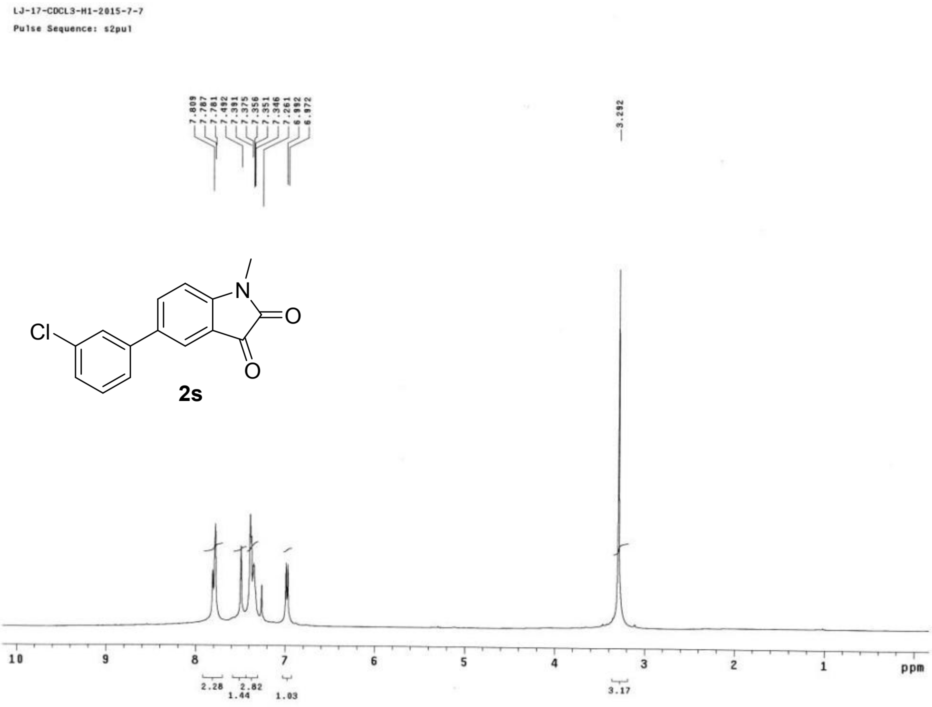

LJ-13 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
7.846
7.843
7.833
7.535
7.532
7.527
7.514
7.472
7.467
7.454
7.435
7.398
7.396
7.378
7.285
6.986
6.985
3.280
N
O
O
2p
10
9
8
7
6
5
4
3
2
1
0 ppm
1.71
2.09
1.00
3.00

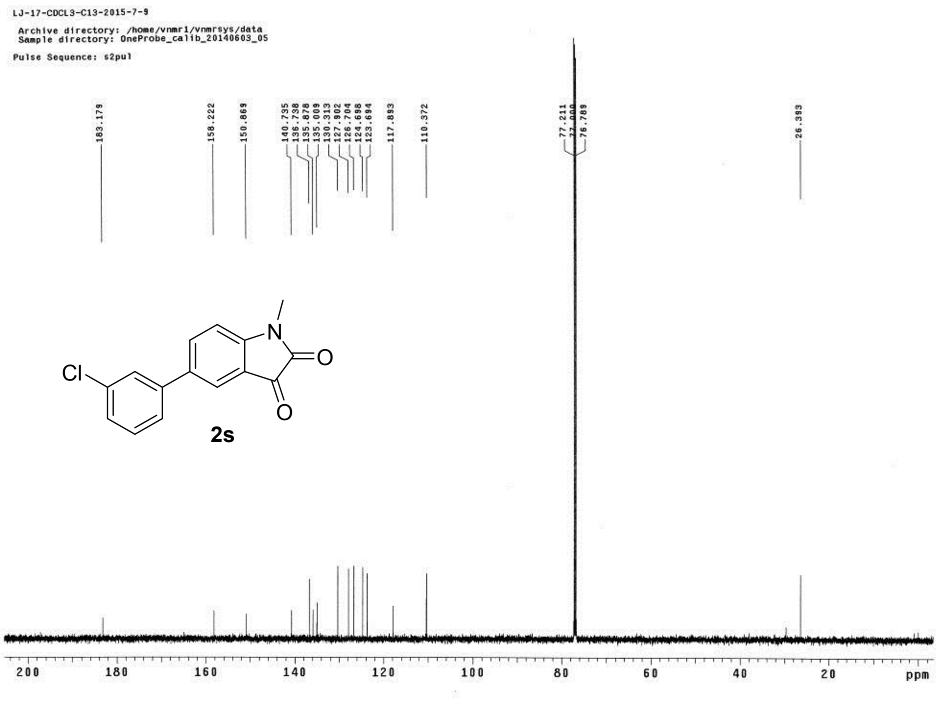

LJ-9 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
7.794
7.790
7.774
7.770
7.753
7.749
7.495
7.491
7.482
7.473
7.466
7.461
7.273
7.159
7.137
7.116
6.986
6.966
3.287
N
O
O
F
2q
10
9
8
7
6
5
4
3
2
1
0 ppm
1.84
1.96
1.80
1.01
3.00

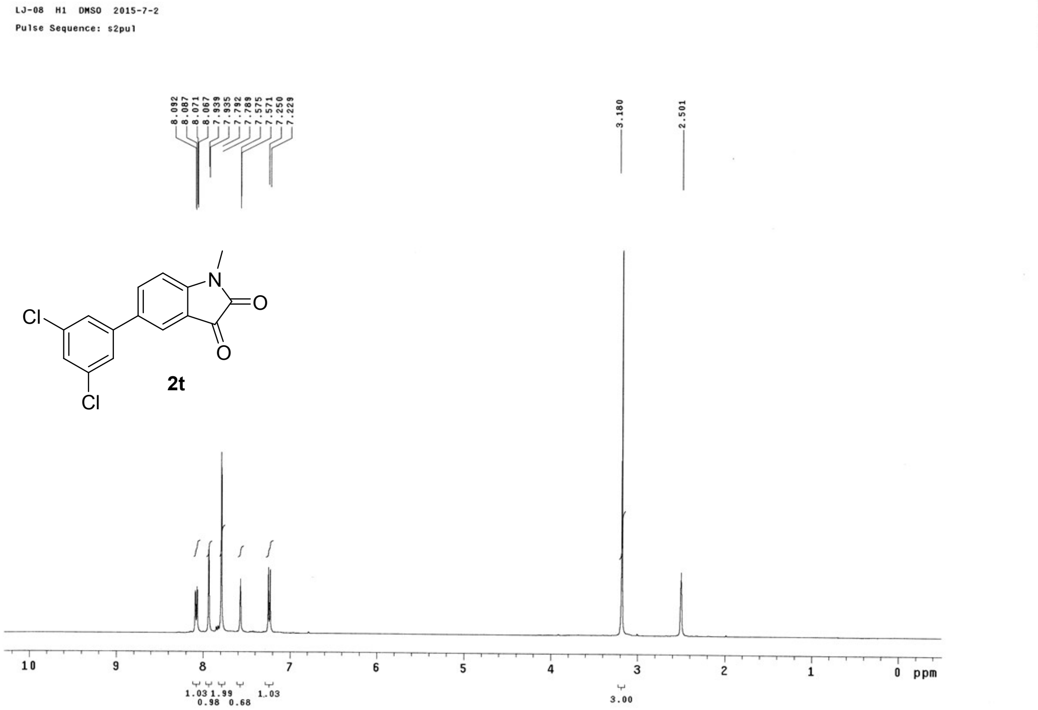

LJ-09-CDCL3-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.306
163.475
161.490
158.222
150.397
136.611
135.353
135.094
128.214
128.163
123.492
117.809
116.030
115.887
110.305
77.211
77.000
76.789
26.326
N
O
O
F
2q
200
180
160
140
120
100
80
60
40
20
ppm

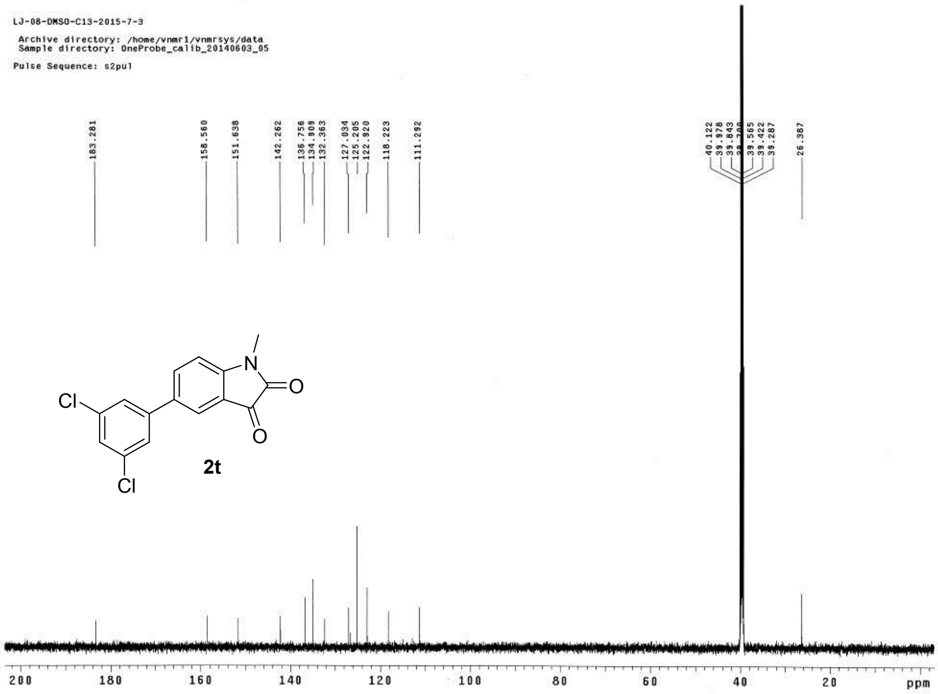

LJ-15 H1 CDCl3 2015-7-7
Pulse Sequence: s2pul
7.806
7.803
7.786
7.782
7.761
7.757
7.757
7.435
7.419
7.398
7.373
6.993
6.973
3.290
N
O
O
Cl
2r
10
9
8
7
6
5
4
3
2
1
ppm
1.10
0.92
4.17
1.00
3.58

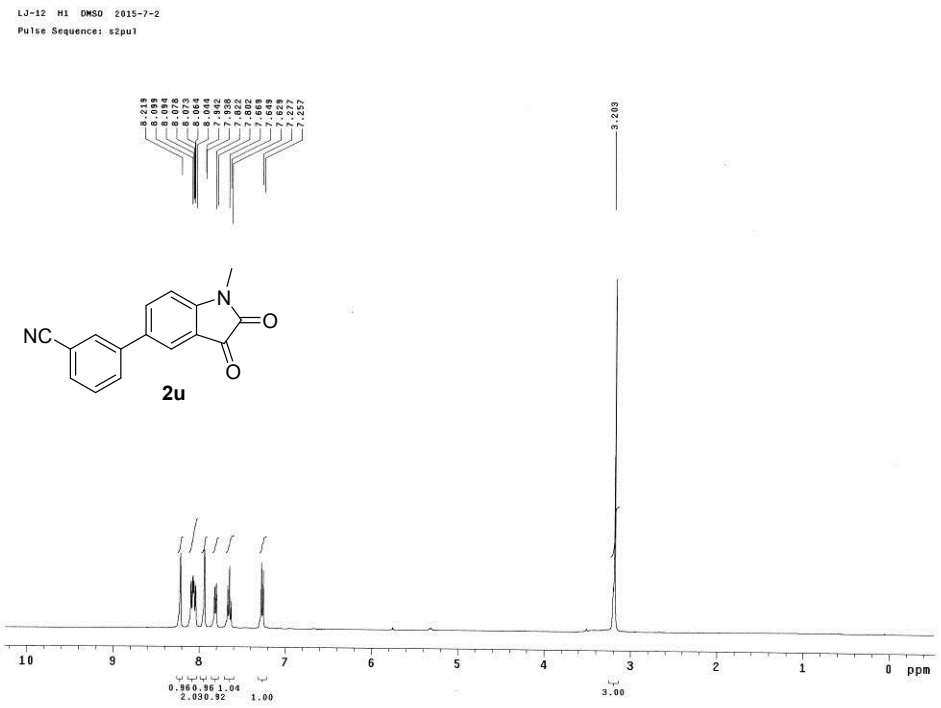

LJ-15-CDCL3-C13-2015-7-9
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.230
158.186
150.625
137.382
135.586
130.038
124.015
129.182
127.758
123.475
117.843
110.364
77.211
77.000
76.788
26.360
N
O
O
Cl
2r
200
180
160
140
120
100
80
60
40
20
ppm

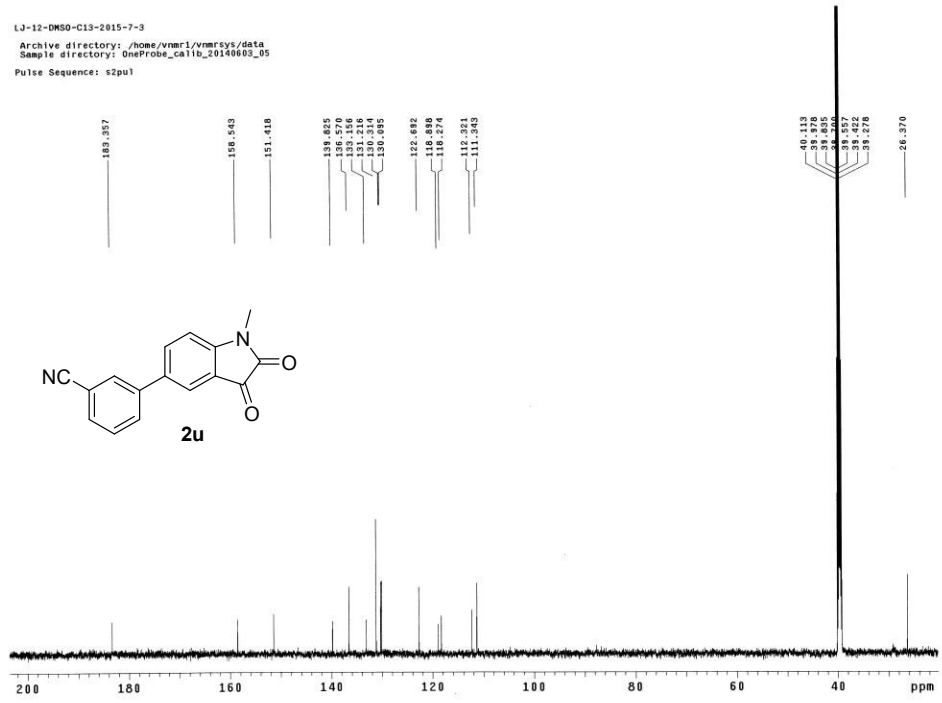

LJ-17-CDCL3-H1-2015-7-7
Pulse Sequence: s2pul
Cl
N
O
O
2s
ppm

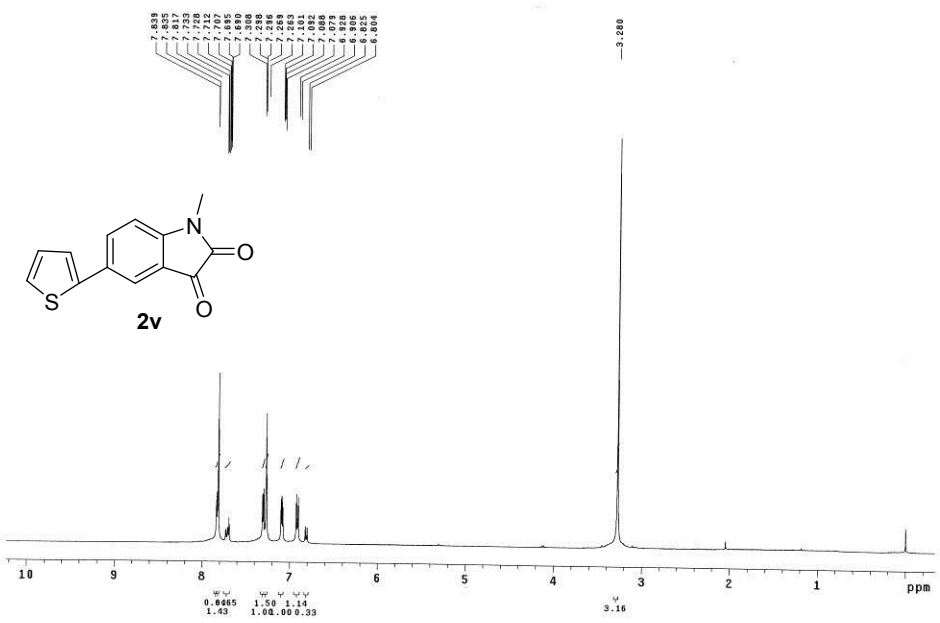

LJ-17-CDCL3-C13-2015-7-9
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
Cl
N
O
O
2s
ppm


LJ-08 H1 DMSO 2015-7-2
Pulse Sequence: s2pul
8.092
8.087
8.071
8.067
7.939
7.935
7.792
7.789
7.575
7.571
7.250
7.229
3.180
2.501
Cl
Cl
N
O
O
2t
1.03
0.98
1.99
0.68
1.03
3.00
ppm


LJ-08-DMSO-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.281
158.560
151.638
142.262
136.756
134.909
132.363
127.034
125.205
122.920
118.223
111.292
40.122
39.978
39.843
39.565
39.422
39.287
26.387
Cl
Cl
N
O
O
2t
ppm


LJ-12 H1 DMSO 2015-7-2
Pulse Sequence: s2pul
8.219
8.098
8.088
8.078
8.073
8.064
8.046
7.942
7.938
7.802
7.802
7.669
7.669
7.277
7.277
7.257
3.203
NC
N
O
O
2u
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96 0.96 1.04 2.03 0.92
1.00
3.00


LJ-12-DMSO-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.357
150.543
151.418
139.825
136.570
133.156
131.216
130.314
130.095
122.692
118.898
118.274
112.321
111.343
40.113
39.978
39.835
39.693
39.557
39.422
39.278
26.370
NC
N
O
O
2u
200
180
160
140
120
100
80
60
40
ppm


LJ-16 H1 CDCl3 2015-7-7
Pulse Sequence: s2pul
7.839
7.835
7.817
7.733
7.728
7.712
7.707
7.695
7.690
7.308
7.295
7.286
7.269
7.263
7.101
7.092
7.086
7.079
6.938
6.986
6.825
6.804
3.280
N
O
O
S
2v
10
9
8
7
6
5
4
3
2
1
ppm
0.9465
1.43
1.50
1.00
1.14
1.00
0.33
3.16