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Supporting Information

IBX-promoted domino reaction of α-hydroxy amides: A facile one-pot synthesis of isatins

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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. 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 α-hydroxy amides **1** were prepared according to the reported procedures.[1]

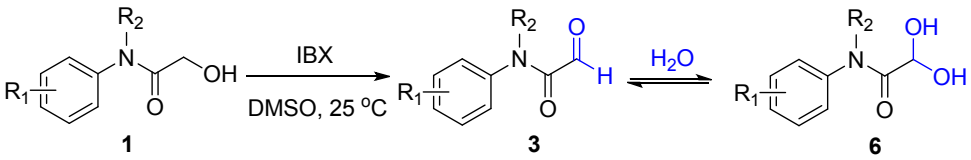
2. Experimental Procedure and Analytical Data of Products

2.1 General procedure for the synthesis of 2

1 **2**

A mixture of α-hydroxy amides **1** (1 mmol) and IBX (1 mmol) were added in 2 ml DMSO and then stirred under air at 100 °C for 3h. After the completion of the reaction (monitored by TLC), the mixture was cooled to room temperature, diluted with water and extracted with ethyl acetate (10 ml x 3). 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 isatins **2** as a red solid.

2.2 General procedure for the synthesis of 3



1 **3** **6**

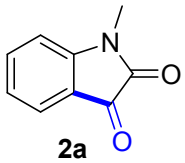
A mixture of α-hydroxy amides **1** (1 mmol) and IBX (1 mmol) were added in 2 ml DMSO and then stirred under air at 25 °C for 3h. After the completion of the reaction (monitored by TLC), the mixture was diluted with water and extracted with ethyl acetate (10 ml x 3). The organic layer was washed with 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=5/1) to afford pure **3**.

Notably, because of α-formyl amide's strong ability of absorbing water, the NMR spectral data of α-formyl amides actually is the data of a mixture of **3** and **6** (see pages 10-11 and 32-35).

2.3 Characterization data for compounds 2

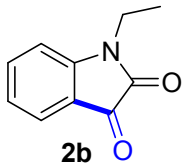
1-methylindoline-2,3-dione (2a)



2a

Yield 91%; red solid; mp. 130-133 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.58 (m, 2H), 7.15-7.11 (m, 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^+ 184.0374, found 184.0370.

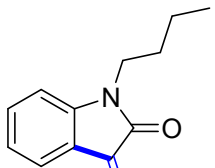
1-ethylindoline-2,3-dione (2b)



2b

Yield 91%; red solid; mp. 92-94 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.58 (m, 2H), 7.14-7.10 (m, 1H), 6.93-6.91 (m, 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^+ 198.0531, found 198.0534.

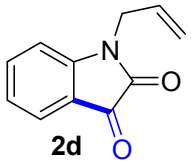
1-butylindoline-2,3-dione (2c)



2c

Yield 91%; red solid; mp. 35-36 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.57 (m, 2H), 7.13-7.09 (m, 1H), 6.91-6.88 (m, 1H), 3.76-3.71 (m, 2H), 1.73-1.64 (m, 2H), 1.61-1.55 (m, 1H), 1.44-1.39 (m, 1H), 0.99 (t, *J* = 6.4 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^+ 226.0844, found 226.0842.

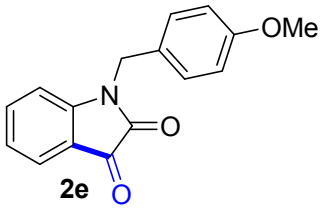
1-allylindoline-2,3-dione (2d)



2d

Yield 74%; red solid; mp. 89-90 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.62 (d, *J* = 7.2 Hz, 1H), 7.58-7.56 (m, 1H), 7.14-7.11 (m, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 5.88-5.82 (m, 1H), 5.31-5.29 (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^+ 210.0531, found 210.0531.

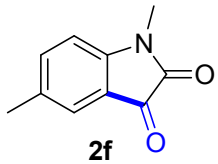
1-(4-methoxybenzyl)indoline-2,3-dione (2e)



2e

Yield 90%; red solid; mp. 169-171 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 6.8 Hz, 1H), 7.51-7.46 (m, 1H), 7.27 (d, *J* = 8.8 Hz, 2H), 7.10-7.06 (m, 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^+ 290.0793, found 290.0790.

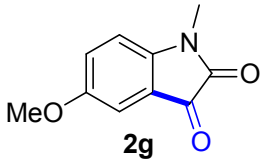
1,5-dimethylindoline-2,3-dione (2f)



2f

Yield 93%; 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^+ 198.0531, found 198.0535.

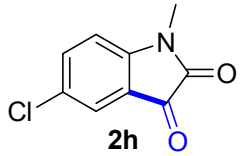
5-methoxy-1-methylindoline-2,3-dione (2g)



2g

Yield 97%; 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^+ 214.0480, found 214.0482.

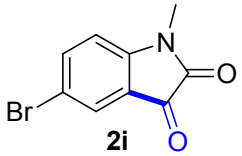
5-chloro-1-methylindoline-2,3-dione (2h)



2h

Yield 74%; 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^+ 217.9985, found 217.9982.

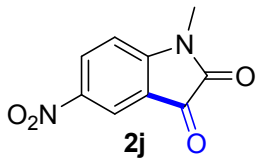
5-bromo-1-methylindoline-2,3-dione (2i)



2i

Yield 80%; 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^+ 261.9480, found 261.9478.

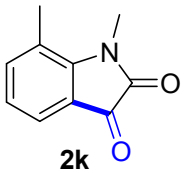
1-methyl-5-nitroindoline-2,3-dione (2j)



2j

Yield 57%; red solid; mp. 201-202 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74-7.71 (m, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 182.1, 157.5, 150.1, 140.6, 128.0, 118.5, 116.6, 111.6, 29.6; HRMS (ESI): m/z [M + Na^+] calcd for $C_9H_6N_2O_4$+Na^+ 229.0225, found 229.0228.

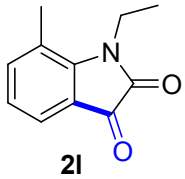
1,7-dimethylindoline-2,3-dione (2k)



2k

Yield 92%; red solid; mp. 162-164 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 7.2 Hz, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.02-6.98 (m, 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^+ 198.0531, found 198.0527.

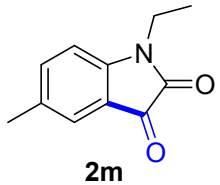
1-ethyl-7-methylindoline-2,3-dione (2l)



2l

Yield 94%; red solid; mp. 188-190 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, J = 7.6 Hz, 1H), 7.34 (d, J = 7.2 Hz, 1H), 7.02-6.98 (m, 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^+ 212.0687, found 212.0683.

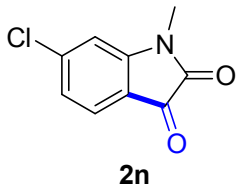
1-ethyl-5-methylindoline-2,3-dione (2m)



2m

Yield 94%; red solid; mp. 74-76 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.38 (m, 2H), 6.80 (d, J = 8.0 Hz, 1H), 3.77 (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^+ 212.0687, found 212.0685.

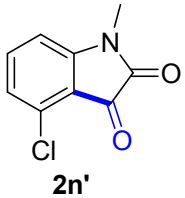
6-chloro-1-methylindoline-2,3-dione (2n)



2n

Yield 46%; red solid; mp. 177-178 °C; ^{1}H 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^+ 217.9985, found 217.9983.

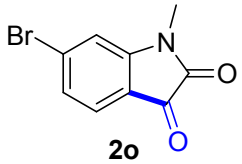
4-chloro-1-methylindoline-2,3-dione (2n')



2n'

Yield 29%; Red solid; mp. 192-195 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.49 (m, 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^+ 217.9985, found 217.9981.

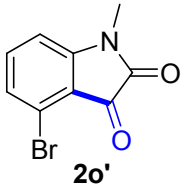
6-bromo-1-methylindoline-2,3-dione (2o)



2o

Yield 44.6%; red solid; mp. 118-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, J = 7.6 Hz, 1H), 7.30 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1H), 7.08 (d, J = 1.6 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^+ 261.9480, found 261.9484.

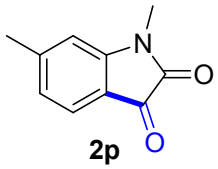
4-bromo-1-methylindoline-2,3-dione (2o')



2o'

Yield 34.4%; Red solid; mp. 199-200 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.44-7.41 (m, 1H), 7.26 (d, J = 9.0 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^+ 261.9480, found 261.9483.

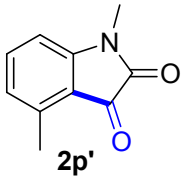
1,6-dimethylindoline-2,3-dione (2p)



2p

Yield 61%; 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), 3.23 (s, 3H), 2.45 (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^+ 198.0531, found 198.0533.

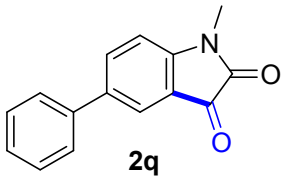
1,4-dimethylindoline-2,3-dione (2p')



2p'

Yield 32%; Red solid; mp. 164-165 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.42 (m, 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^+ 198.0531, found 198.0528.

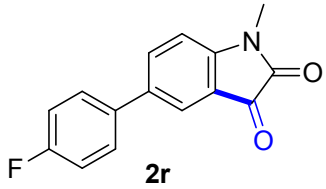
1-methyl-5-phenylindoline-2,3-dione (2q)



2q

Yield 78%; 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^+ 260.0687, found 260.0689.

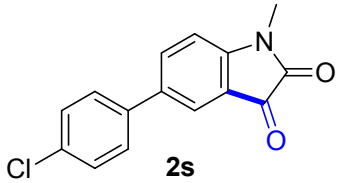
5-(4-fluorophenyl)-1-methylindoline-2,3-dione (2r)



2r

Yield 71%; 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), 7.16-7.11 (m, 2H), 6.98 (d, J = 8.0 Hz, 1H), 3.29 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 183.3, 162.1 (d, $J_{C\text{-}F}$ = 246.8 Hz), 158.2, 150.4, 136.6, 136.3, 135.1, 128.2 (d, $J_{C\text{-}F}$ = 7.6 Hz), 123.5, 117.8, 115.9 (d, $J_{C\text{-}F}$ = 21.4 Hz), 110.3, 26.3; HRMS (ESI): m/z [M + Na^+] calcd for $C_{15}H_{10}FNO_2$+Na^+ 278.0593, found 278.0597.

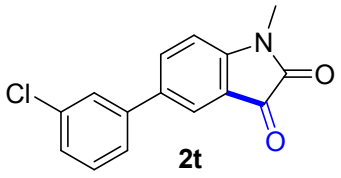
5-(4-chlorophenyl)-1-methylindoline-2,3-dione (2s)



2s

Yield 73%; 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^+ 294.0298, found 294.0298.

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

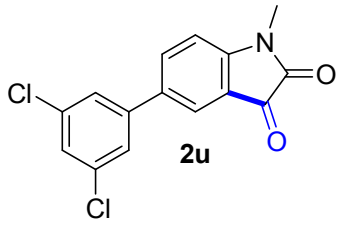


2t

Yield 75%; 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^+ 294.0298, found 294.0294.

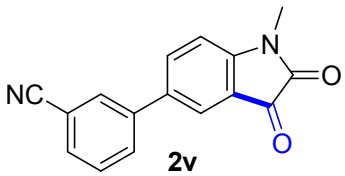
5-(3,5-dichlorophenyl)-1-methylindoline-2,3-dione (2u)



2u

Yield 70%; red solid; mp. 228-230 °C; ^{1}H NMR (400 MHz, $CDCl_3$): 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 = 1.2 Hz, 1H), 7.24 (d, J = 8.4 Hz, 1H), 3.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 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 $C_{15}H_9C_{l2}NO_2$+Na^+ 327.9908, found 327.9911.

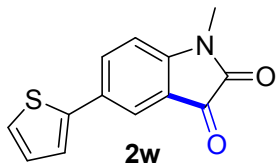
3-(1-methyl-2,3-dioxoindolin-5-yl)benzonitrile (2v)



2v

Yield 67%; red solid; mp. 236-237 °C; ^{1}H NMR (400 MHz, $CDCl_3$): 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), 7.67-7.63 (m, 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^+ 285.0640, found 285.0638.

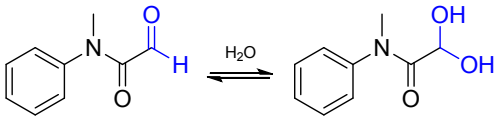
1-methyl-5-(thiophen-2-yl)indoline-2,3-dione (2w)



2w

Yield 72%; 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^+ 266.0252, found 266.0250.

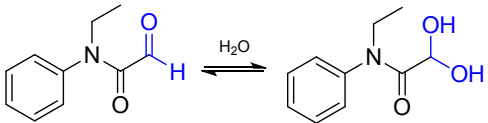
***N*-methyl-2-oxo-*N*-phenylacetamide (3a)** [2]



3a

Yield 93%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 7.47-7.37 (m, 3H), 7.32-7.21 (m, 2H), 4.93 (s, 0.6H), 3.41-3.27 (m, 3H).

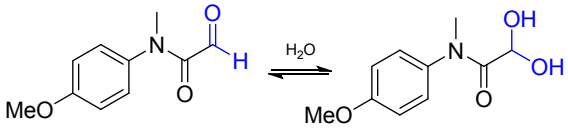
***N*-ethyl-2-oxo-*N*-phenylacetamide (3b)** [2]



3b

Yield 95%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 7.46-7.38 (m, 3H), 7.28-7.18 (m, 2H), 4.84 (s, 0.46H), 3.89-3.68 (m, 2H), 1.21-1.08 (m, 3H).

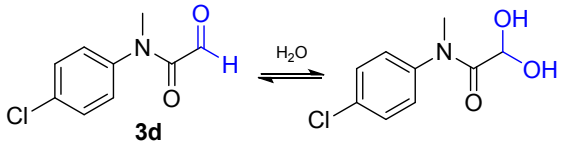
***N*-(4-methoxyphenyl)-*N*-methyl-2-oxoacetamide (3c)** [2]



3c

Yield 92%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 9.38 (s, 0.27H), 7.21-7.12 (m, 2H), 6.95-6.92 (m, 2H), 3.83 (s, 3H), 3.37-3.23 (m, 3H).

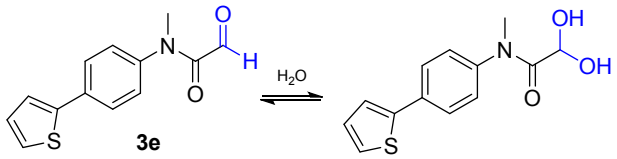
***N*-(4-chlorophenyl)-*N*-methyl-2-oxoacetamide (3d)** [2]



3d

Yield 90%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 7.42-7.40 (m, 2H), 7.27-7.21 (m, 2H), 4.97 (s, 0.53H), 3.48-3.25 (m, 3H).

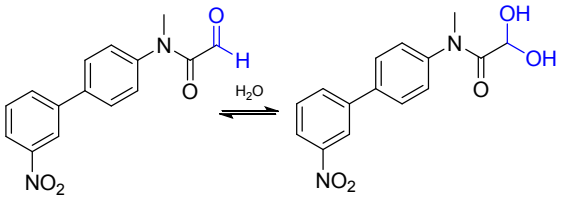
***N*-methyl-2-oxo-*N*-(4-(thiophen-2-yl)phenyl)acetamide (3e)** [2]



3e

Yield 82%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 7.67-7.62 (m, 2H), 7.58-7.52 (m, 1H), 7.51-7.30 (m, 2H), 7.25-7.18 (m, 1H), 7.15-7.08 (m, 1H), 3.42-3.26 (m, 3H).

***N*-methyl-*N*-(3'-nitro-[1,1'-biphenyl]-4-yl)-2-oxoacetamide (3f)** [2]



3f

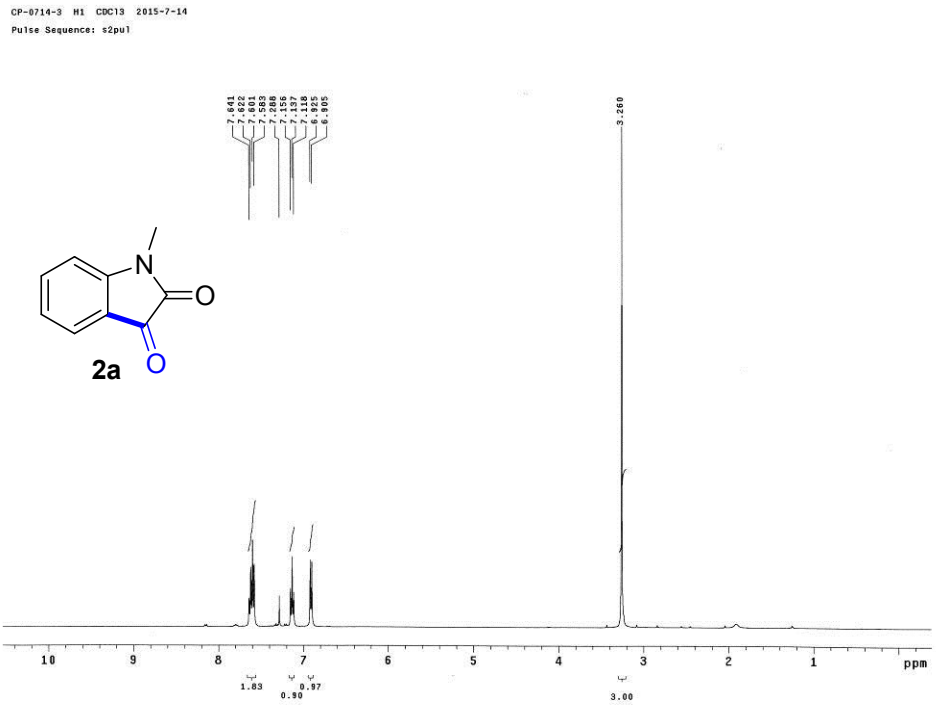
Yield 89%; canary yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$ + D_2O): δ 8.45 (s, 1H), 8.25 (d, *J*=6.4 Hz, 1H), 7.91 (d, *J*=5.2 Hz, 1H), 7.73 (d, *J*=5.2 Hz, 1H), 7.69 (d, *J*=6.4 Hz, 1H), 7.66 (d, *J*=5.2 Hz, 1H), 7.41 (d, *J*=6.4 Hz, 1H), 7.33 (d, *J*=6.4 Hz, 1H), 3.46-3.33 (m, 3H).

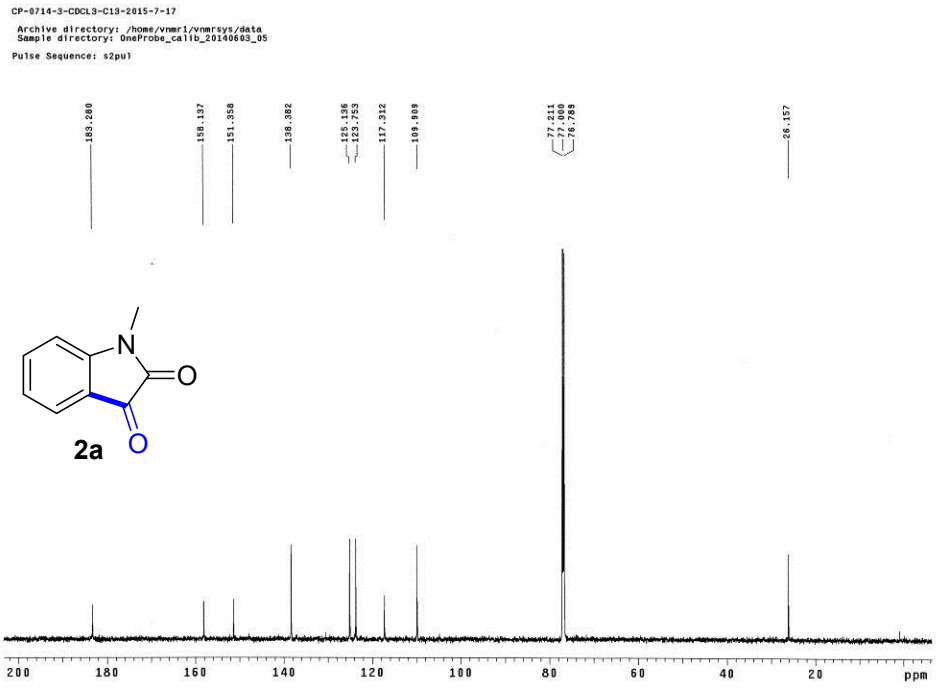
3. References

[1] Miller, M.; Vogel, J. C.; Tsang, W.; Merrit, A.; Procter, D. J. *Org. Biomol. Chem*, **2009**,*7*, 589-597.

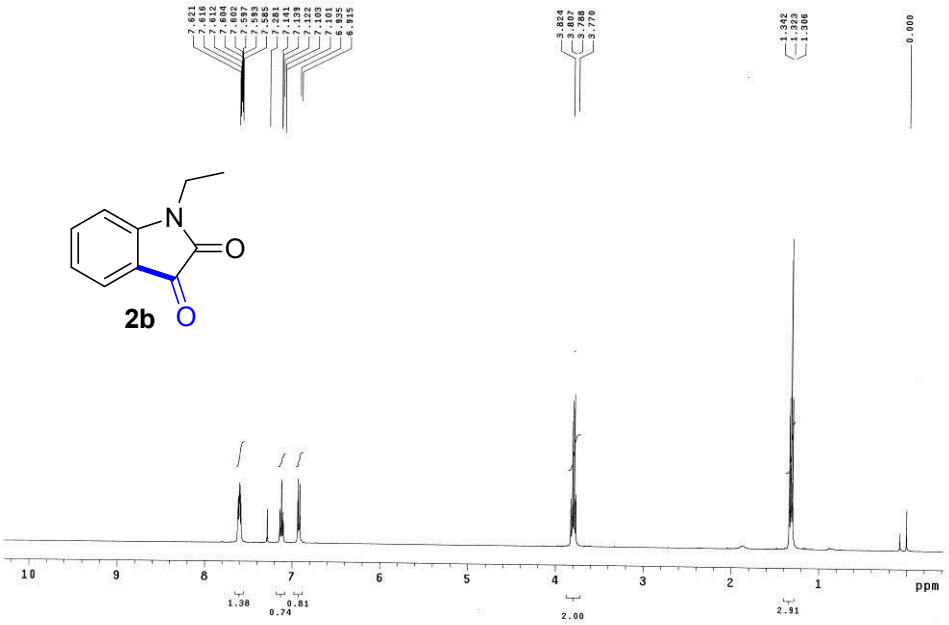
[2] Tang, B. X.; Song, R. J.;Wu, C. Y.; Liu, Y.; Zhou, M. B.; Wei, W. T.; Deng, G. B.; Yin, D. L.; Li, J. H. *J. Am. Chem. Soc*. **2010**, *132*, 8900-8902.

4. ^{1}H NMR and ^{13}C NMR Spectra of Products

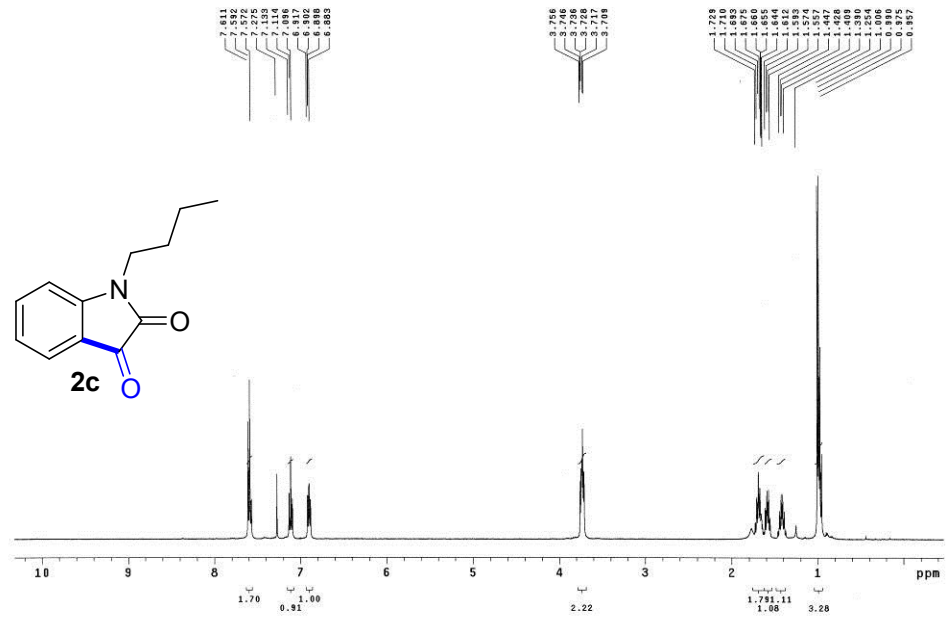


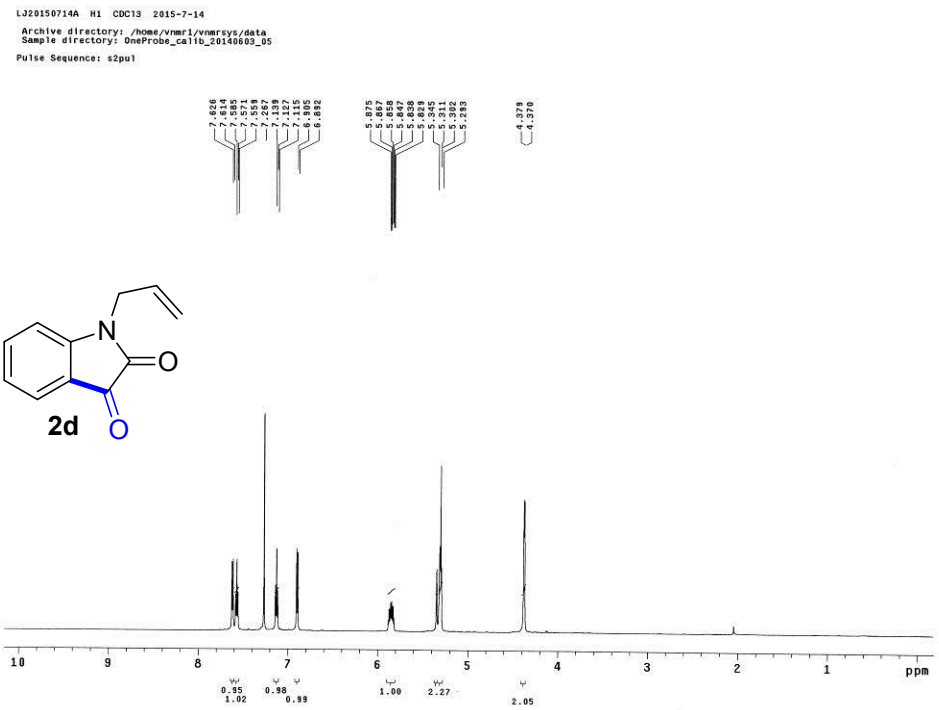


LJ-11 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul



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
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7.614
7.585
7.571
7.559
7.287
7.139
7.127
7.115
6.955
6.892
5.875
5.867
5.858
5.847
5.838
5.828
5.345
5.311
5.302
5.293
4.378
4.370
N
O
O
2d
10
9
8
7
6
5
4
3
2
1
ppm
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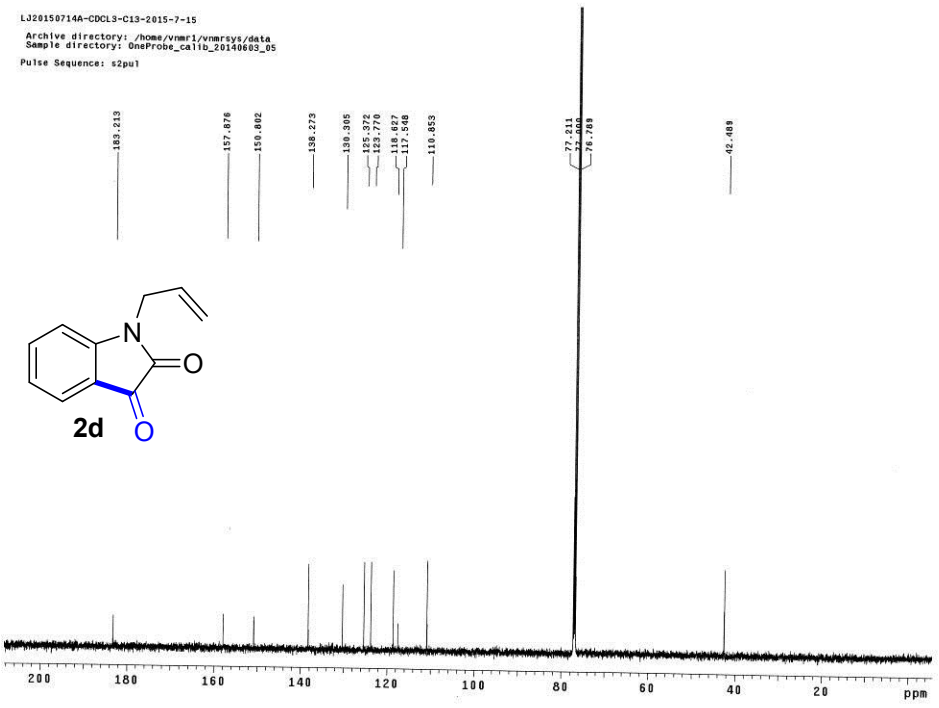
0.98
0.99
1.00
2.27
2.05


LJ20150714A-CDCL3-C13-2015-7-15
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Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
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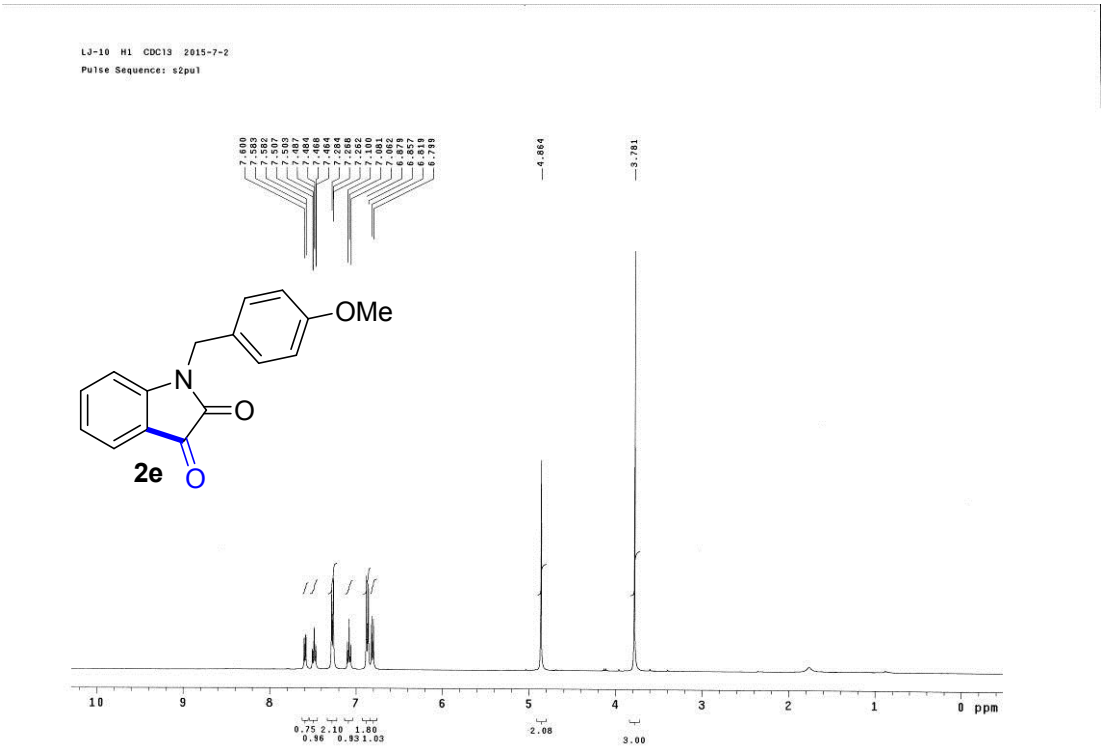
157.876
150.602
136.273
130.305
125.372
123.770
118.627
117.548
110.853
77.211
77.000
76.788
42.489
N
O
O
2d
200
180
160
140
120
100
80
60
40
20
ppm


LJ-10 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
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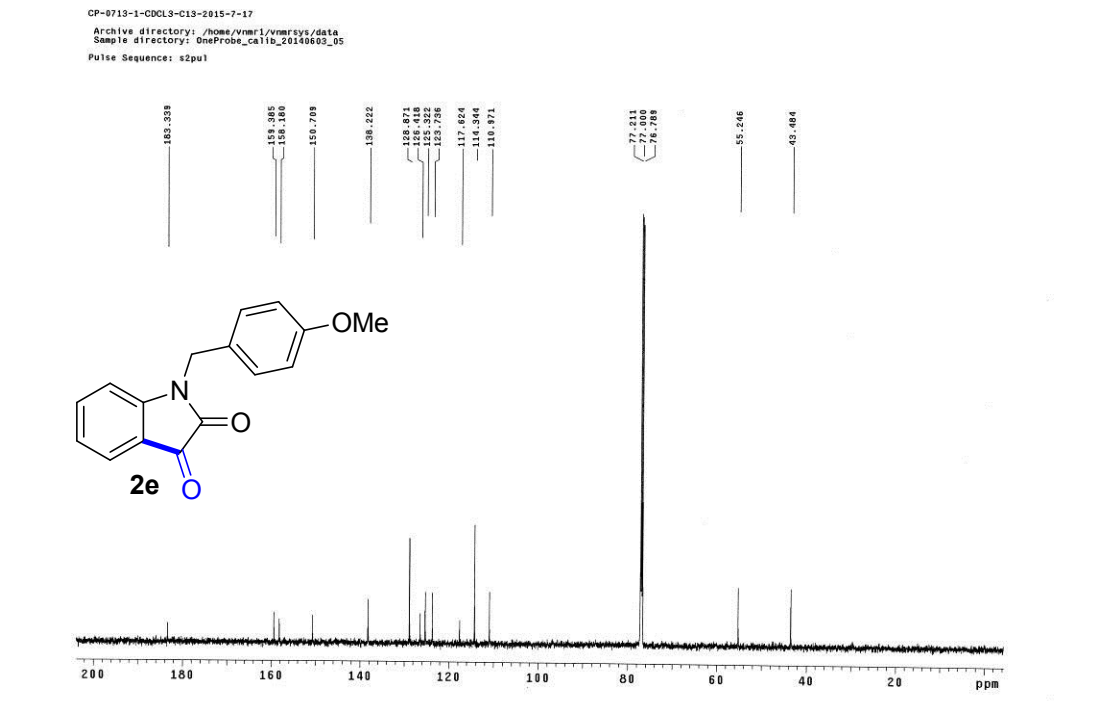
7.593
7.582
7.507
7.503
7.487
7.466
7.468
7.464
7.284
7.266
7.262
7.100
7.081
7.062
6.879
6.857
6.818
6.798
4.864
3.781
OMe
N
O
O
2e
10
9
8
7
6
5
4
3
2
1
0 ppm
0.75 2.10 1.80
0.96 0.93 1.03
2.08
3.00


CP-0713-1-CDCL3-C13-2015-7-17
Archive directory: /home/vnmrj/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
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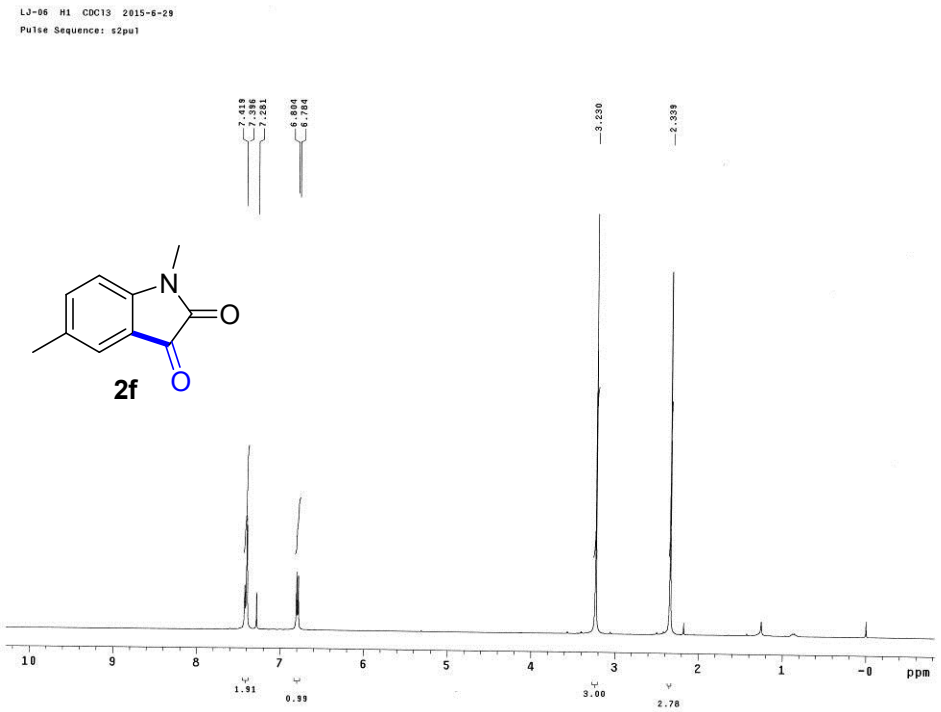
159.305
158.160
150.709
138.222
128.871
126.418
125.322
123.736
117.624
114.344
110.971
77.211
77.000
76.789
55.246
43.484
OMe
N
O
O
2e
200
180
160
140
120
100
80
60
40
20
ppm


LJ-06 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
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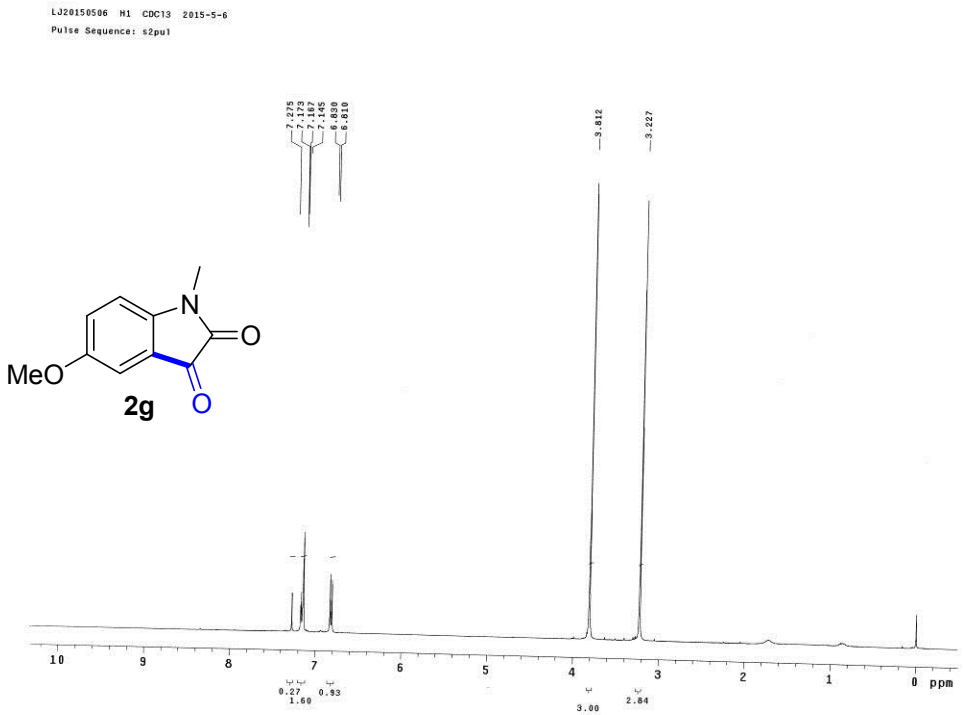
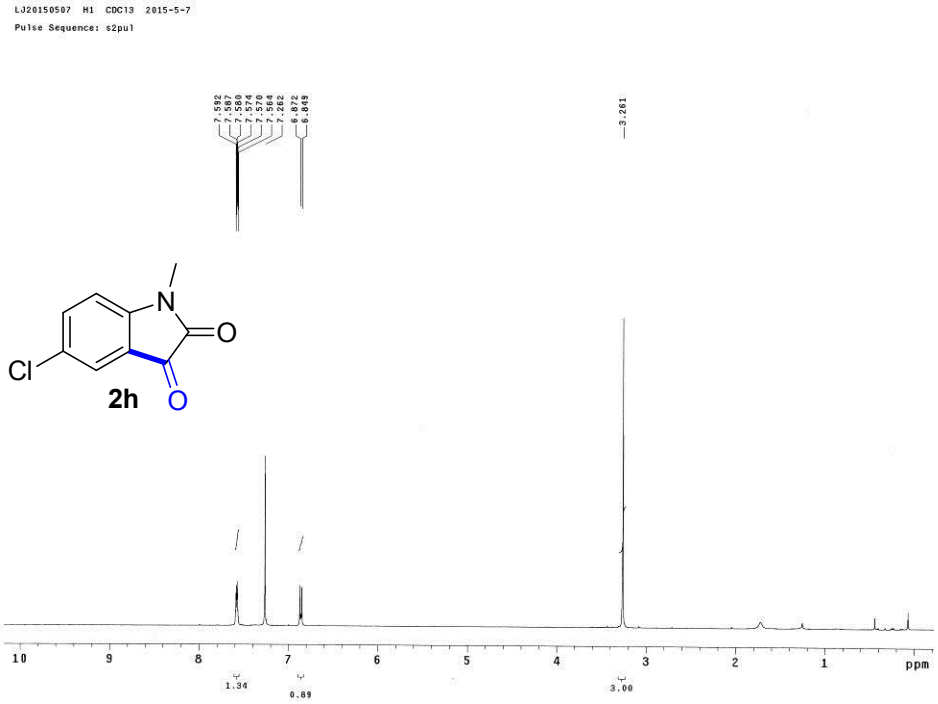
7.396
7.281
6.804
6.784
3.230
2.339
2f
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.91
0.99
3.00
2.78


LJ20150506 H1 CDCl3 2015-5-6
Pulse Sequence: s2pul
7.275
7.179
7.167
7.145
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
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7.580
7.574
7.570
7.564
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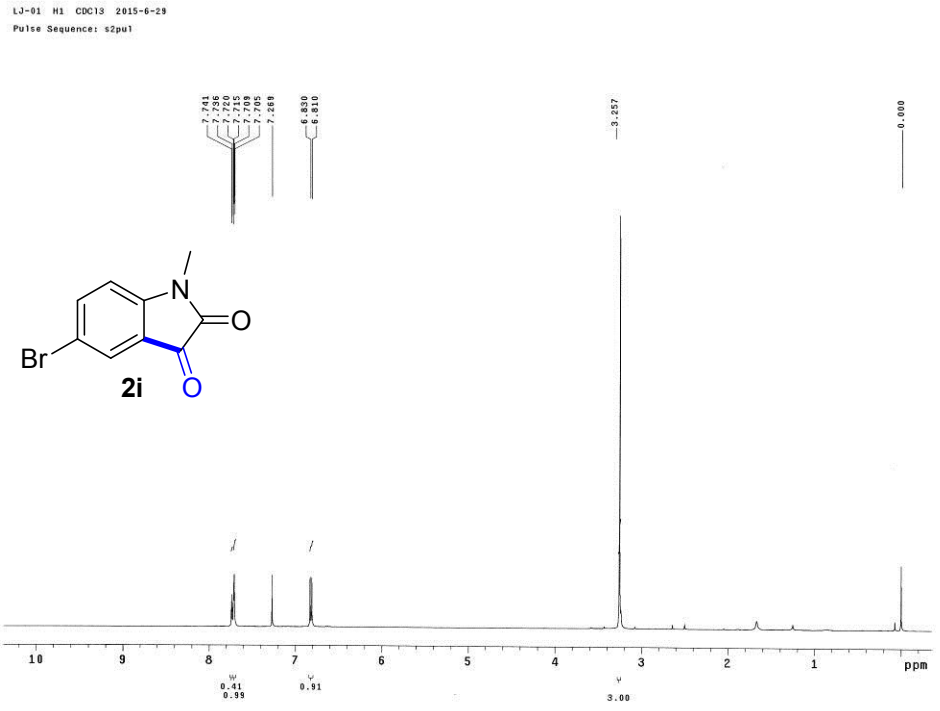
6.049
3.261
N
O
Cl
O
2h
10
9
8
7
6
5
4
3
2
1
ppm
1.34
0.89
3.00


LJ-01 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
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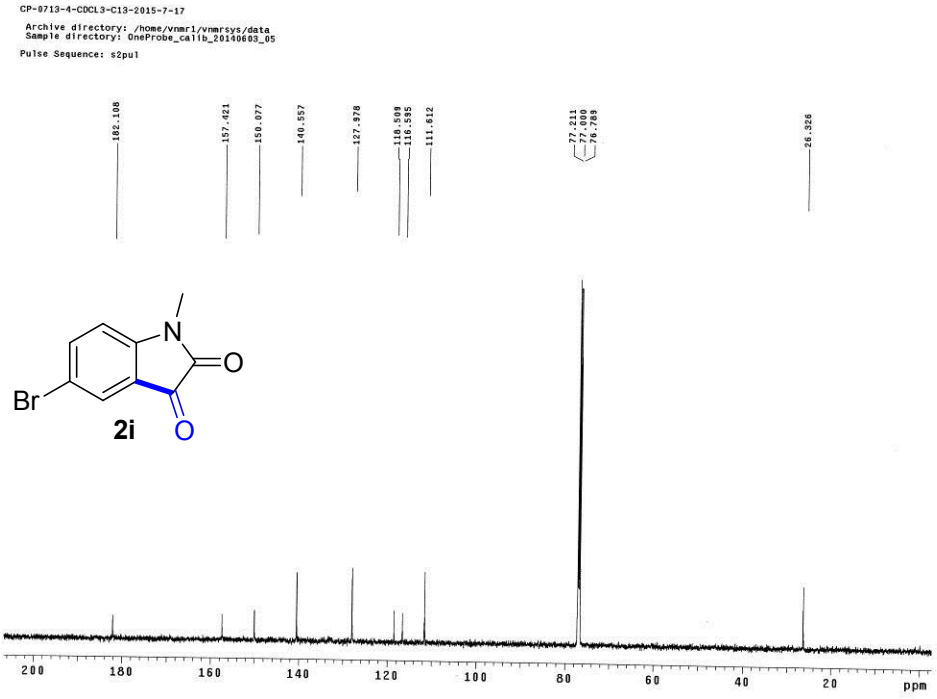
7.736
7.720
7.715
7.709
7.705
7.269
6.830
6.810
3.257
0.000
N
O
Br
O
2i
10
9
8
7
6
5
4
3
2
1
ppm
0.41
0.99
0.91
3.00


CP-0713-4-CDCL3-C13-2015-7-17
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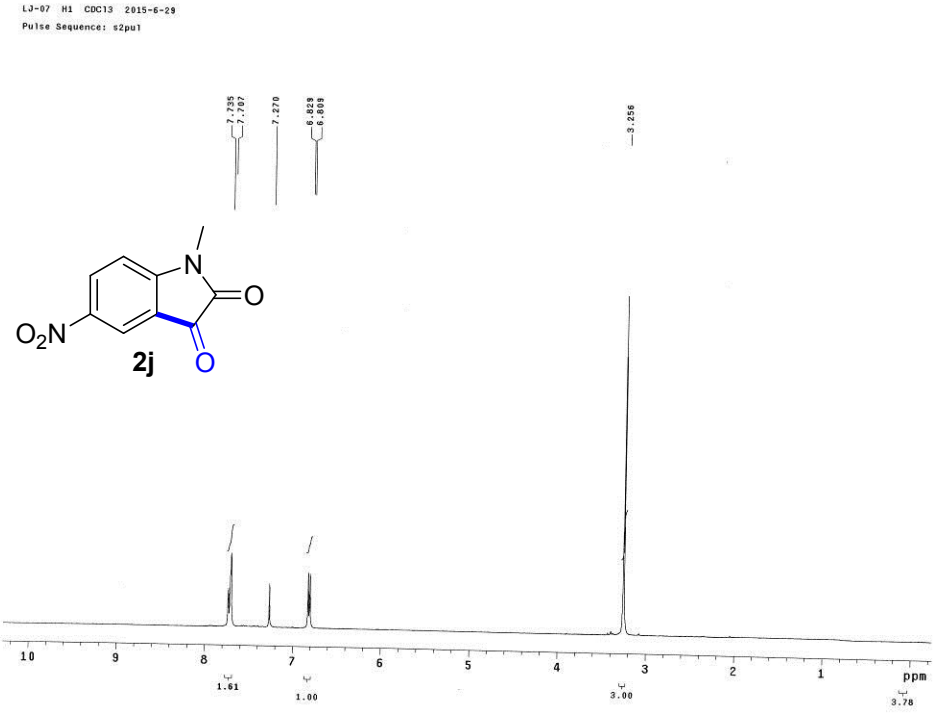
Sample directory: OneProbe_Calib_20140603_05
Pulse Sequence: s2pul
182.108
157.421
150.077
140.557
127.378
118.598
116.595
111.612
77.211
77.000
76.789
26.326
N
O
Br
O
2i
200
180
160
140
120
100
80
60
40
20
ppm


LJ-07 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
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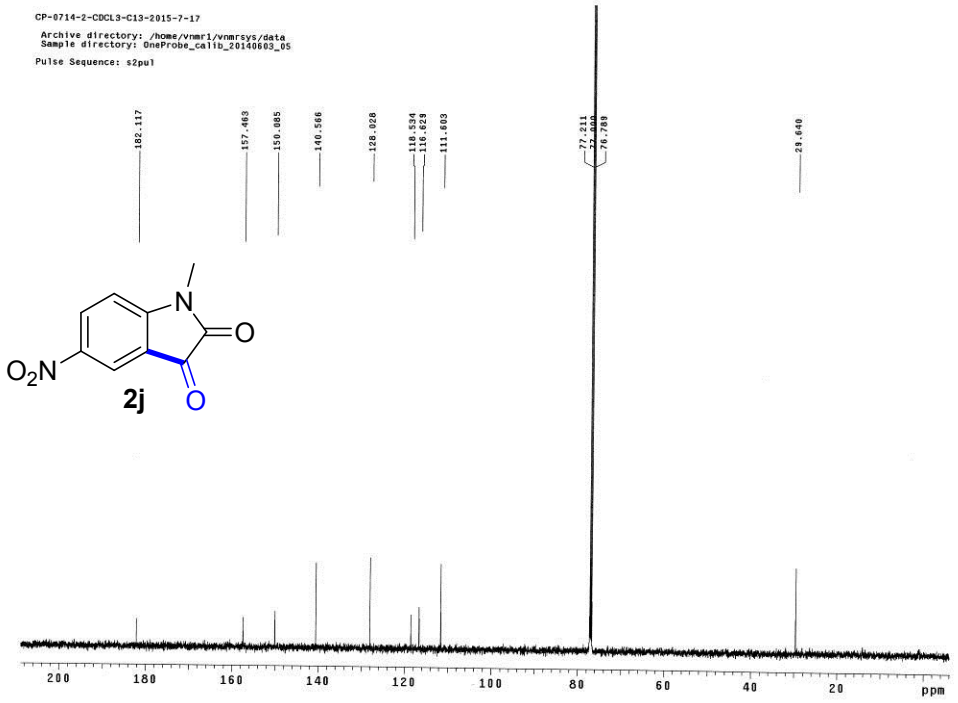
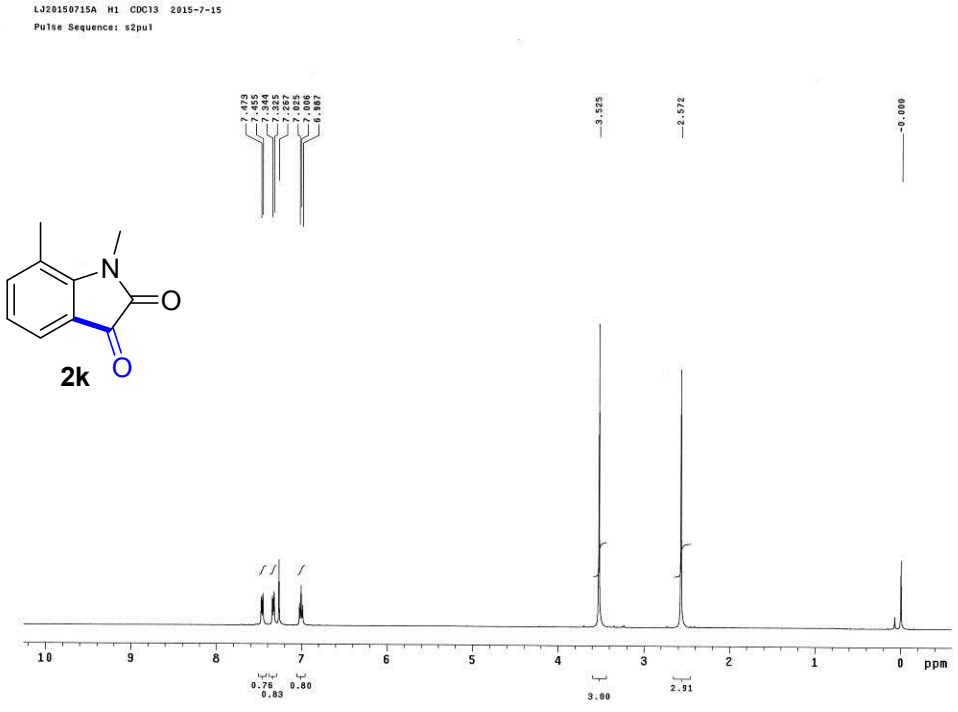
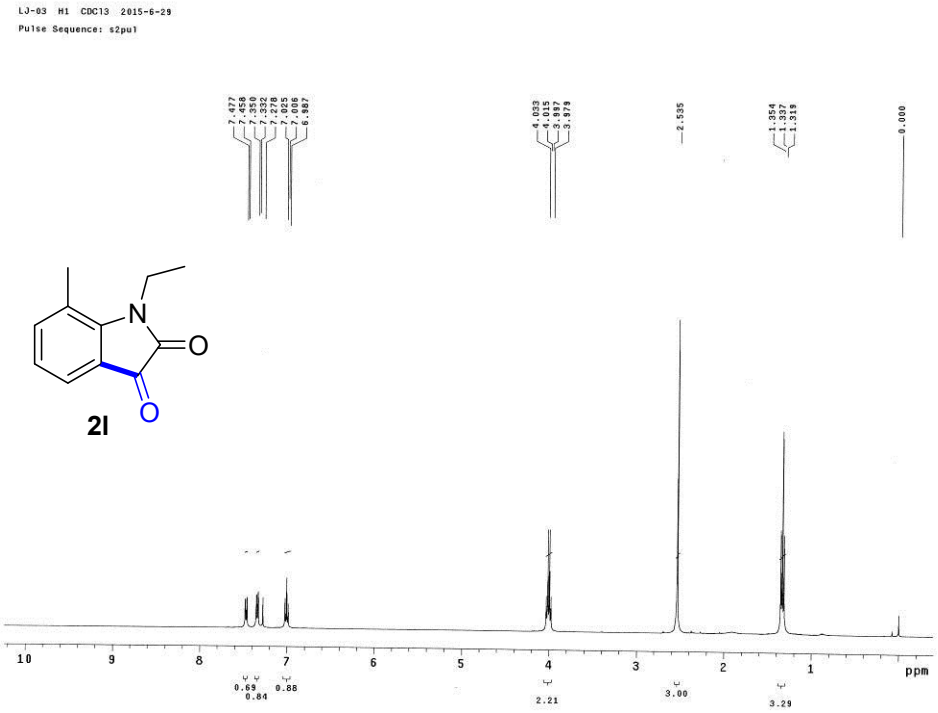
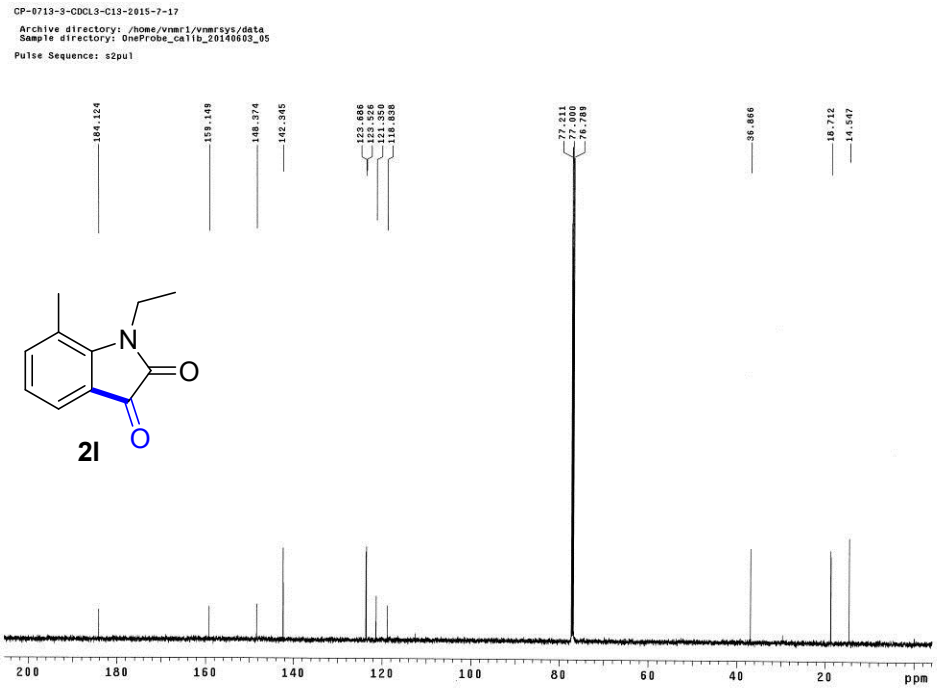
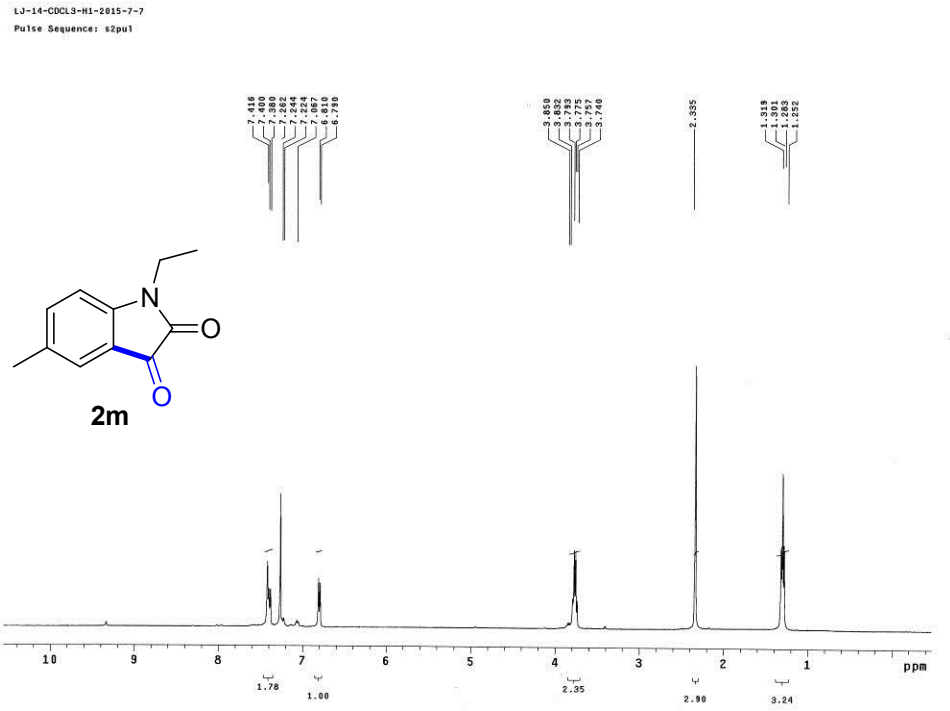
7.707
7.270
6.828
6.808
3.256
N
O
O2N
O
2j
10
9
8
7
6
5
4
3
2
1
ppm
1.61
1.00
3.00
3.78

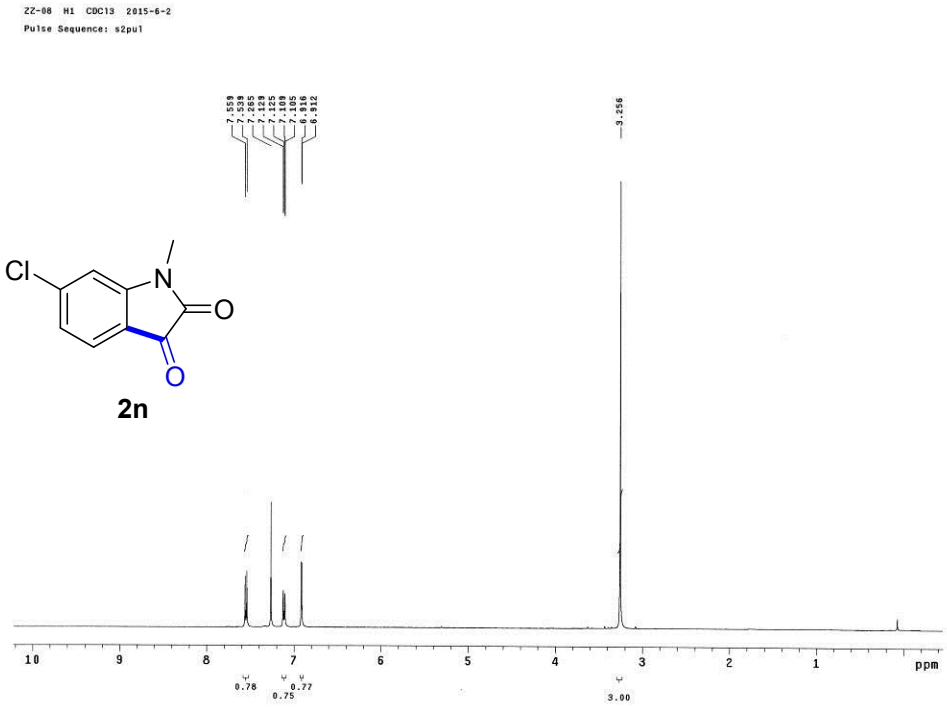

CP-0714-2-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
182.117
157.463
150.085
140.566
128.026
118.534
116.029
111.603
77.211
76.788
29.640
2j
200
180
160
140
120
100
80
60
40
20
ppm

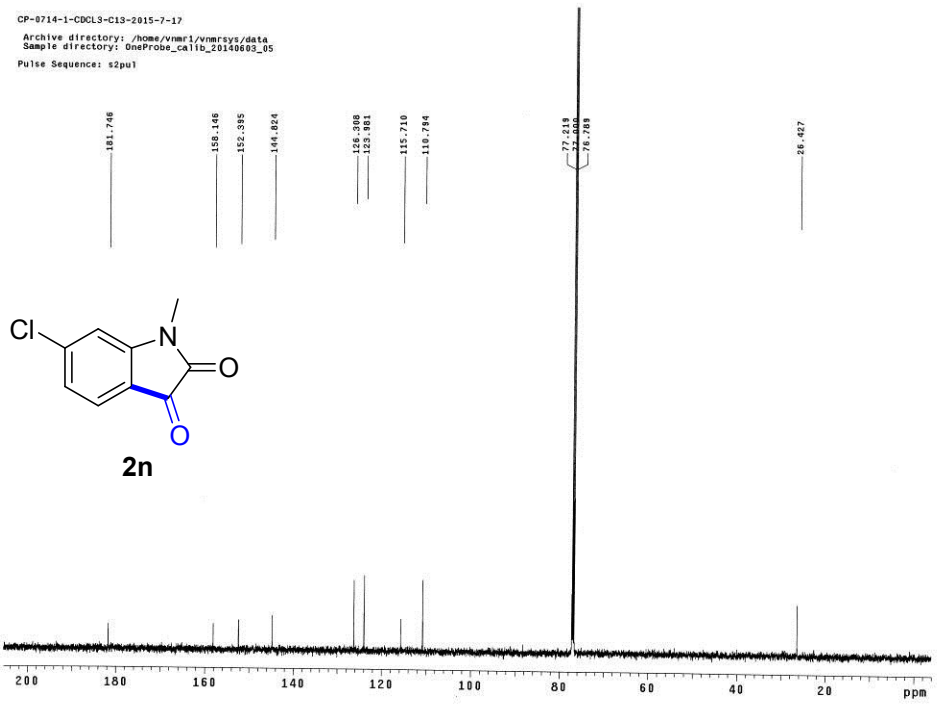

LJ20150715A H1 CDCl3 2015-7-15
Pulse Sequence: s2pul
7.473
7.465
7.344
7.325
7.267
7.088
7.008
6.987
3.525
2.572
0.000
2k
10
9
8
7
6
5
4
3
2
1
0
ppm
0.76
0.83
0.80
3.00
2.91

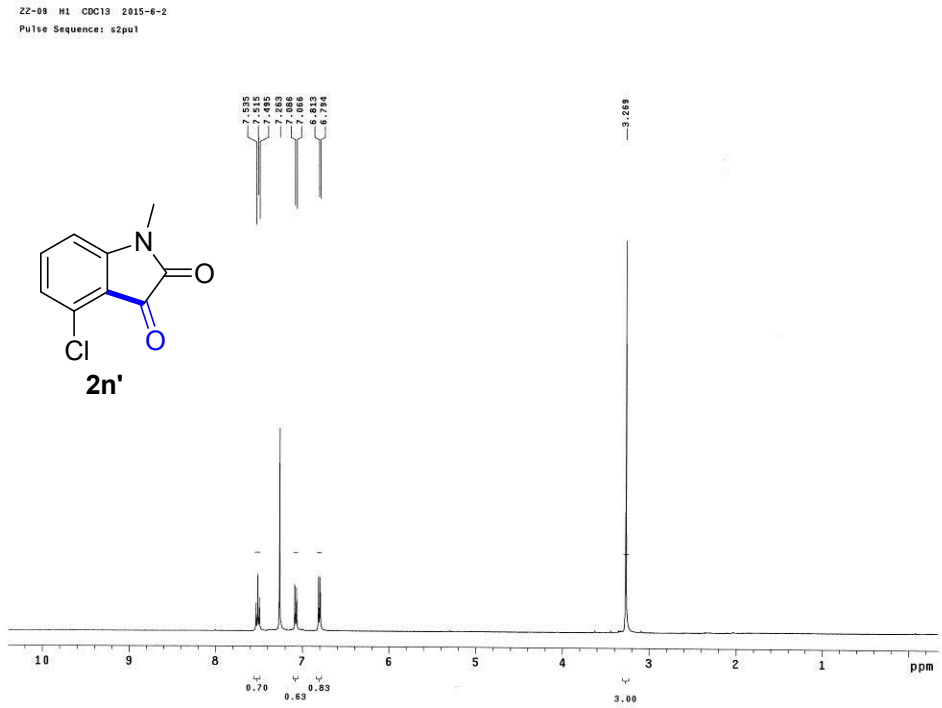

LJ-03 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.477
7.458
7.350
7.332
7.278
7.025
7.006
6.987
4.033
4.015
3.997
3.979
2.535
1.354
1.337
1.319
0.000
N
O
O
2l
10
9
8
7
6
5
4
3
2
1
ppm
0.69
0.84
0.88
2.21
3.00
3.29

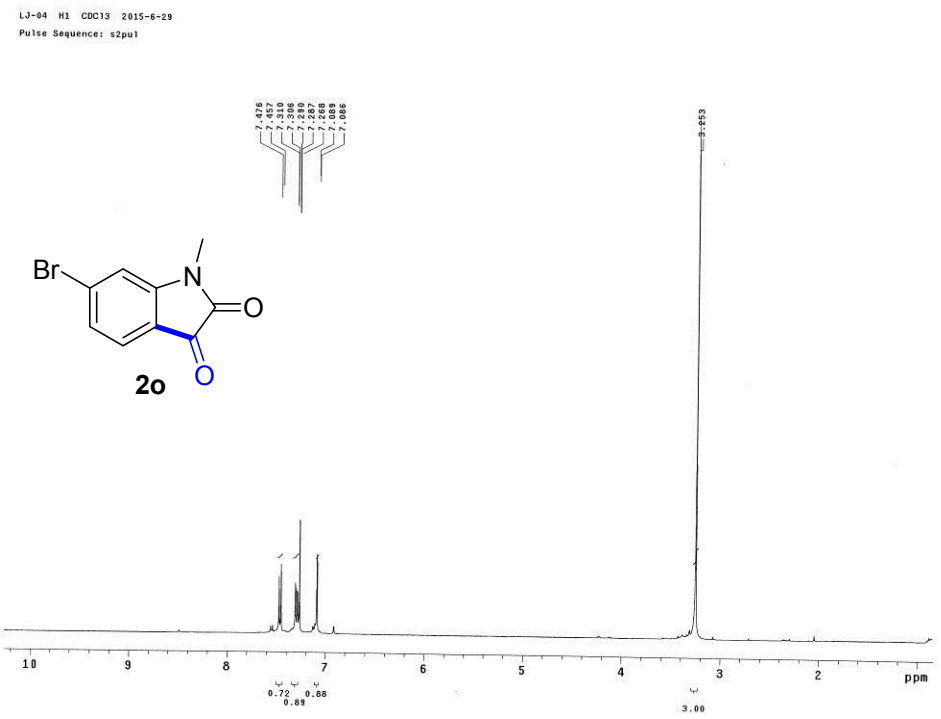

CP-0713-3-CDCl3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
184.124
159.149
140.374
142.345
123.686
123.526
121.350
118.838
77.411
77.000
76.589
36.866
18.712
14.547
N
O
O
2l
200
180
160
140
120
100
80
60
40
20
ppm

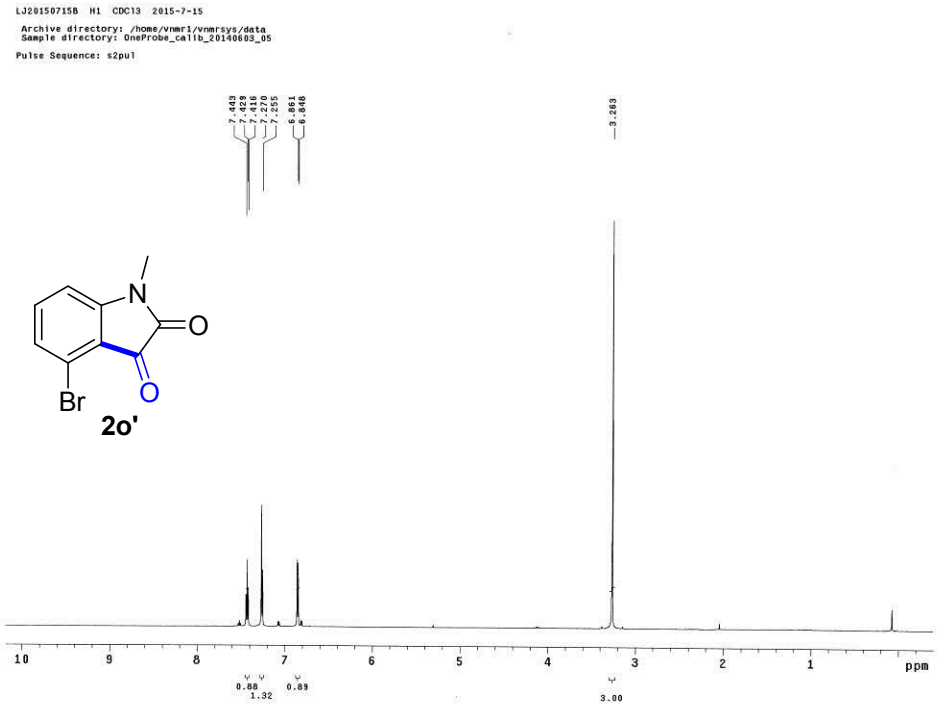

LJ-14-CDCL3-H1-2015-7-7
Pulse Sequence: s2pul
7.416
7.400
7.380
7.262
7.244
7.224
7.087
6.810
6.798
3.850
3.832
3.793
3.775
3.757
3.740
2.335
1.319
1.301
1.283
1.252
N
O
O
2m
10
9
8
7
6
5
4
3
2
1
ppm
1.78
1.00
2.35
2.90
3.24

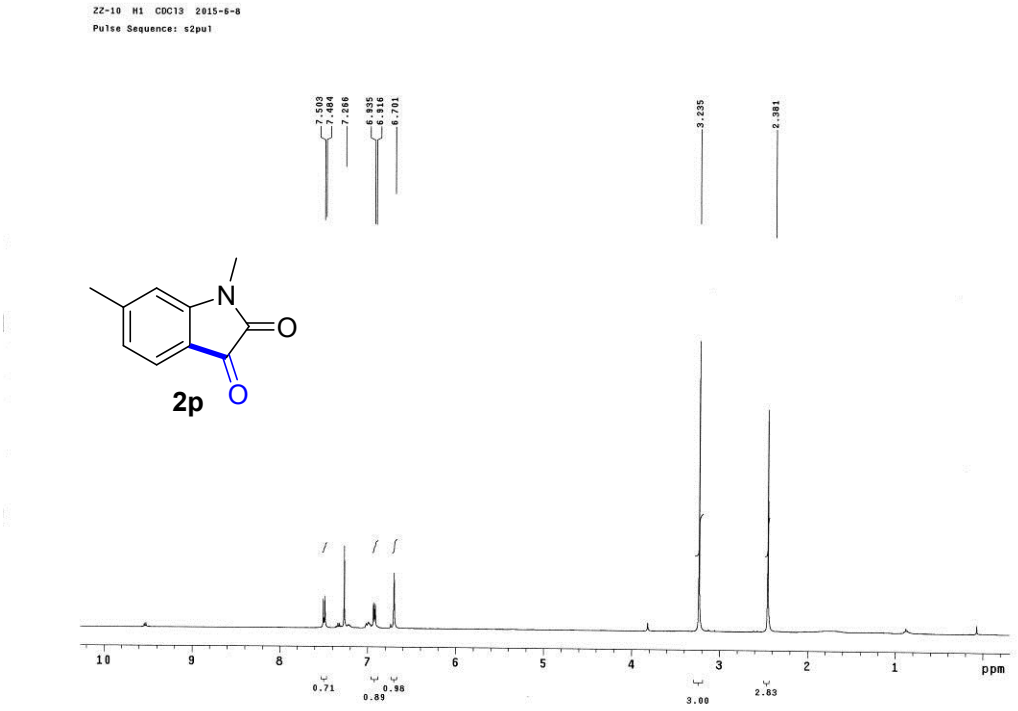

ZZ-08 H1 CDCl3 2015-6-2
Pulse Sequence: s2pul
7.559
7.538
7.265
7.129
7.125
7.108
7.105
6.916
6.912
3.256
Cl
N
O
O
2n
10
9
8
7
6
5
4
3
2
1
ppm
0.78
0.75
0.77
3.00

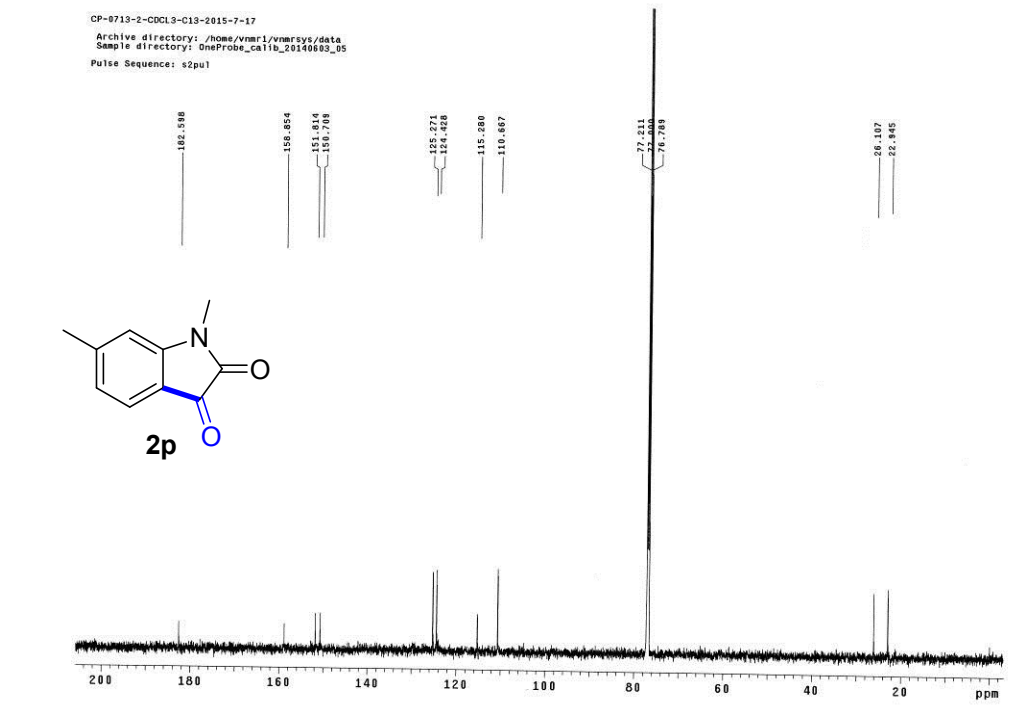

CP-0714-1-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
181.746
158.146
152.395
144.824
126.308
123.981
115.710
110.794
77.219
77.000
76.788
26.427
Cl
N
O
O
2n
200
180
160
140
120
100
80
60
40
20
ppm

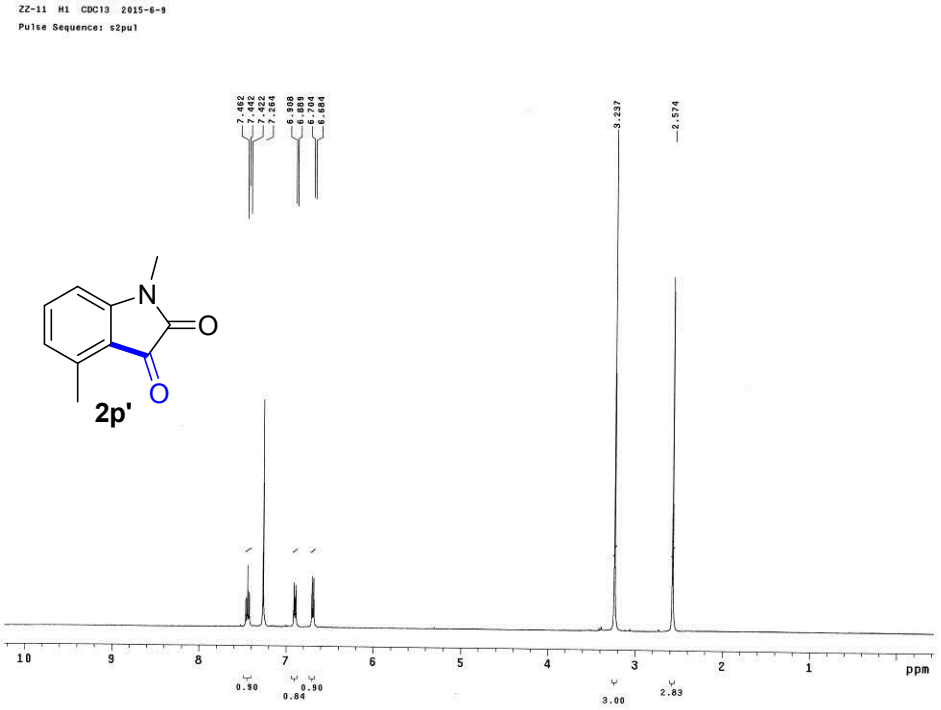

ZZ-08 H1 CDCl3 2015-6-2
Pulse Sequence: s2pul
7.535
7.515
7.495
7.266
7.086
7.066
6.813
6.794
3.269
N
O
O
Cl
2n'
10
9
8
7
6
5
4
3
2
1
ppm
0.70
0.63
0.83
3.00

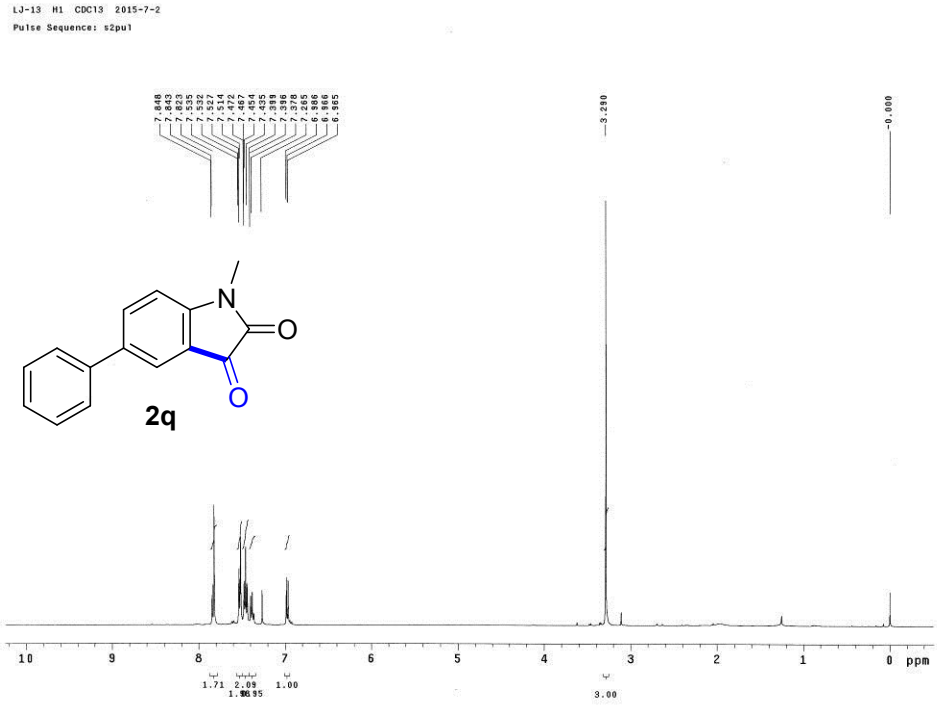

LJ-04 H1 CDCl3 2015-6-29
Pulse Sequence: s2pul
7.476
7.457
7.310
7.306
7.290
7.287
7.088
7.069
7.086
3.253
Br
N
O
O
2o
10
9
8
7
6
5
4
3
2
ppm
0.72
0.89
0.88
3.00

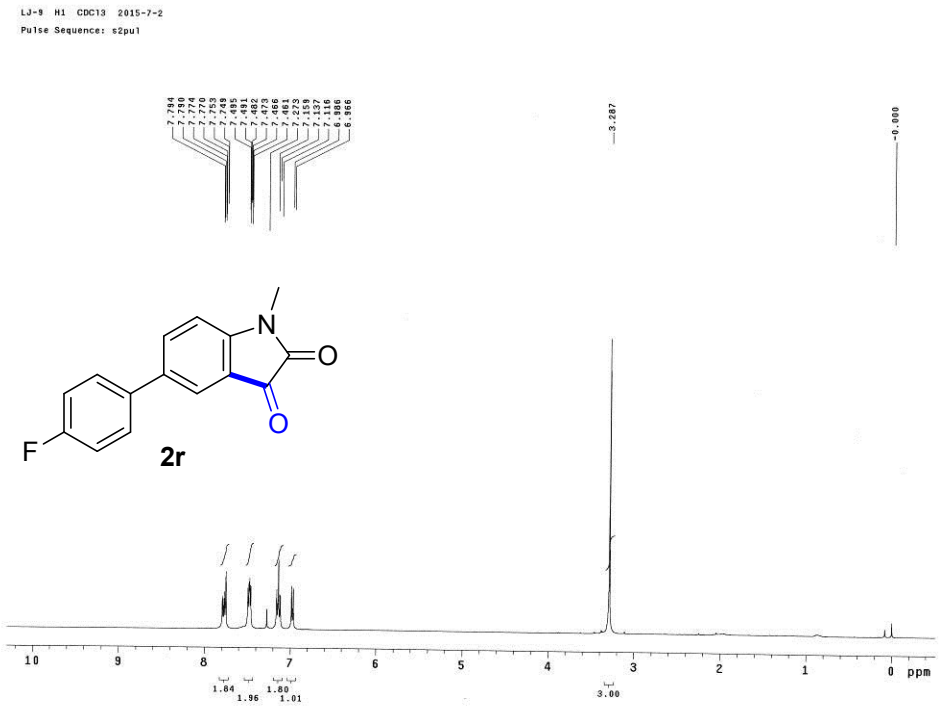

LJ20150715B H1 CDCl3 2015-7-15
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
7.443
7.429
7.416
7.270
7.255
6.861
6.840
3.263
N
O
O
Br
2o'
10
9
8
7
6
5
4
3
2
1
ppm
0.88
1.32
0.89
3.00

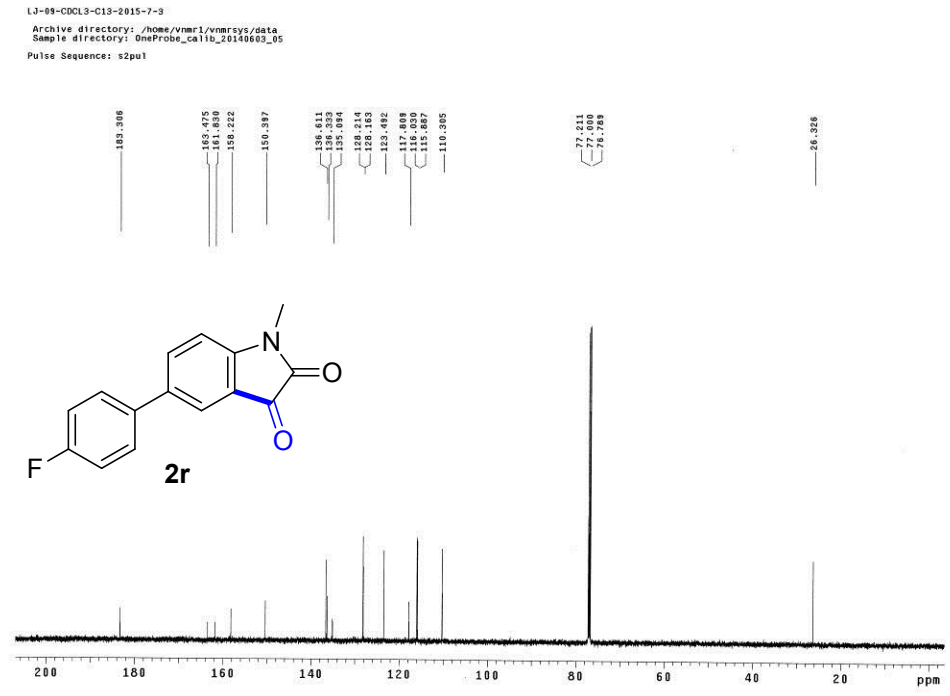

ZZ-10 H1 CDCl3 2015-6-8
Pulse Sequence: s2pul
7.503
7.484
7.266
6.835
6.816
6.701
3.235
2.381
N
O
O
2p
10
9
8
7
6
5
4
3
2
1
ppm
0.71
0.89
0.98
3.00
2.83

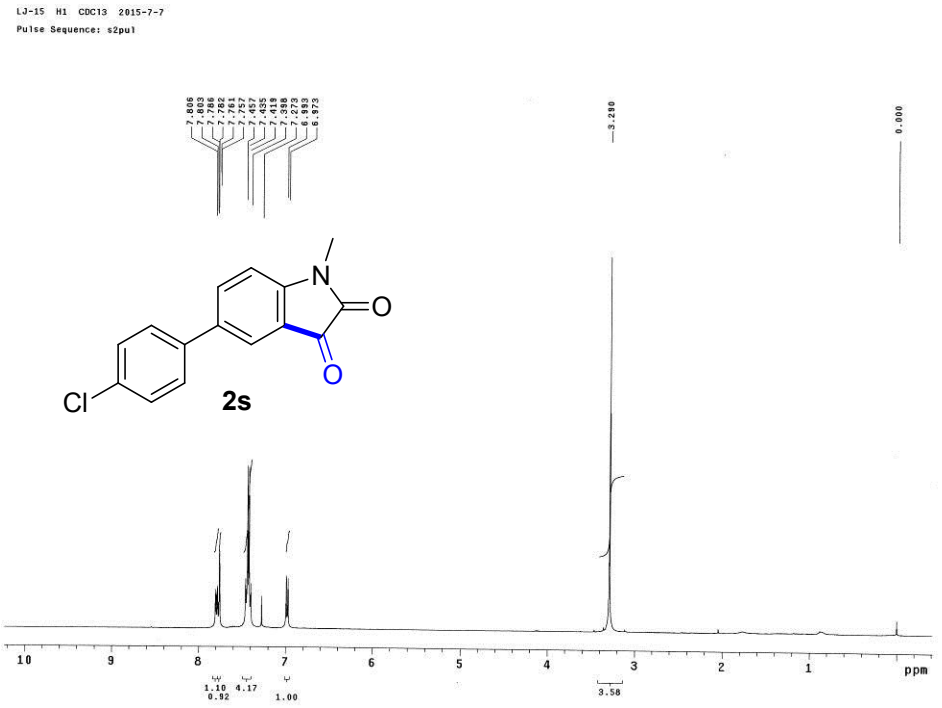

CP-0713-2-CDCL3-C13-2015-7-17
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
182.598
158.854
151.814
150.709
125.271
124.428
115.280
110.667
77.211
77.000
76.789
26.107
22.945
N
O
O
2p
200
180
160
140
120
100
80
60
40
20
ppm

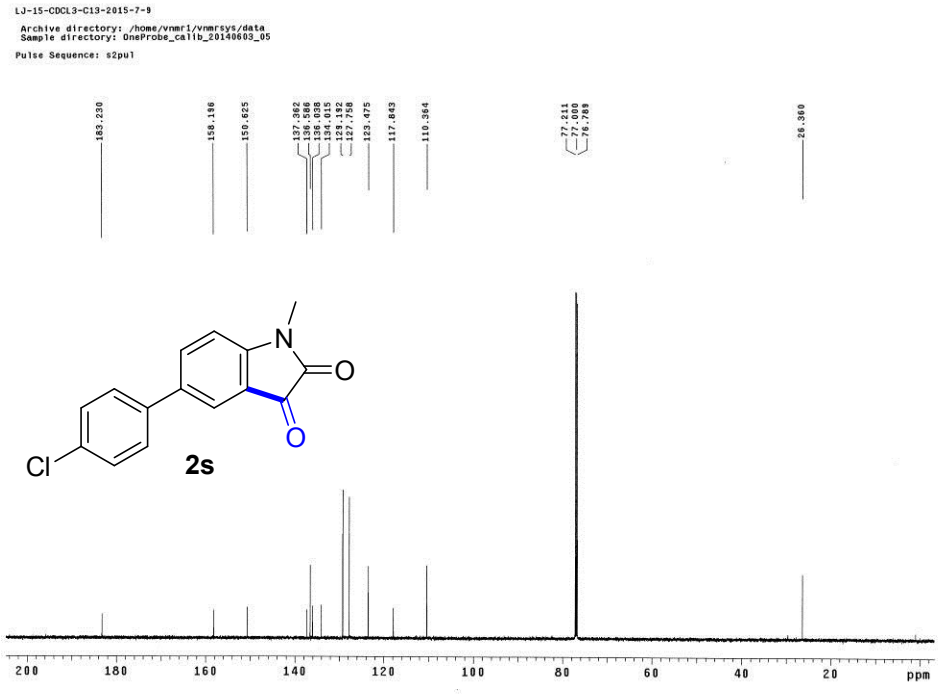

ZZ-11 H1 CDCl3 2015-6-9
Pulse Sequence: s2pul
7.492
7.480
7.422
7.264
6.888
6.885
6.704
6.684
3.237
2.574
N
O
O
2p'
10
9
8
7
6
5
4
3
2
1
ppm
0.90
0.84
0.90
3.00
2.83

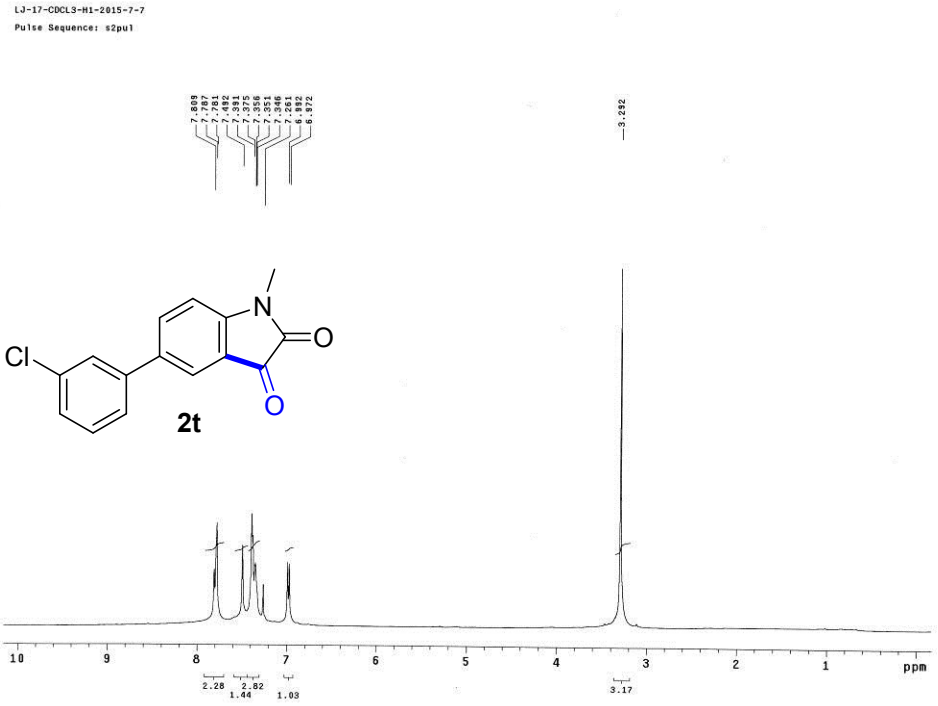

LJ-13 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
7.646
7.643
7.625
7.535
7.532
7.517
7.514
7.472
7.467
7.454
7.435
7.399
7.396
7.378
7.265
6.986
6.985
3.290
0.000
N
O
O
2q
10
9
8
7
6
5
4
3
2
1
0
ppm
1.71
2.09
1.00
3.00

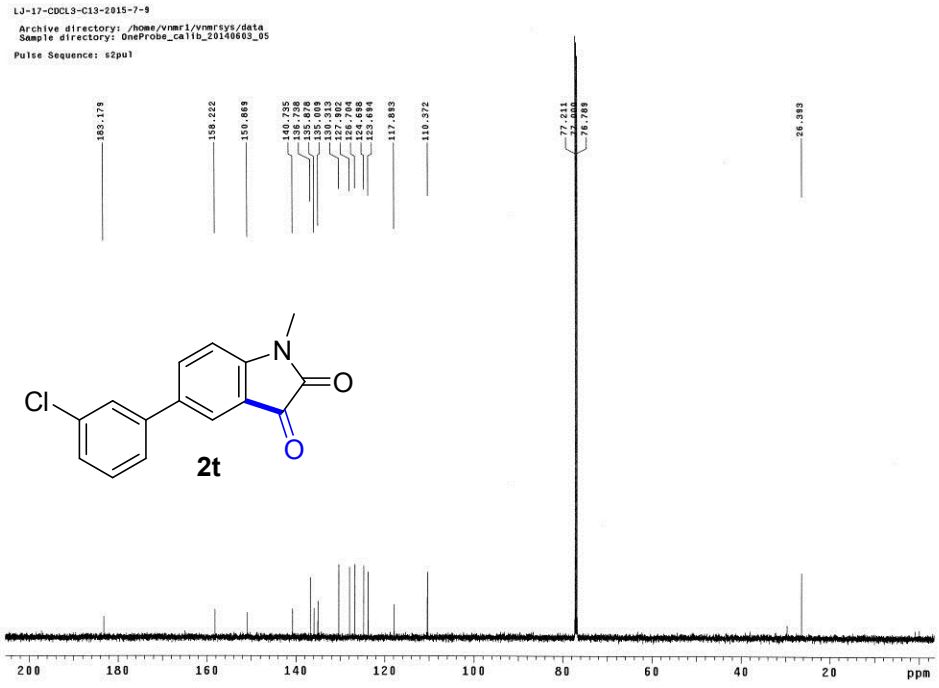

LJ-9 H1 CDCl3 2015-7-2
Pulse Sequence: s2pul
7.794
7.790
7.774
7.770
7.753
7.749
7.495
7.491
7.482
7.473
7.466
7.461
7.223
7.159
7.137
7.116
6.886
6.866
3.287
0.000
N
O
O
F
2r
10
9
8
7
6
5
4
3
2
1
0
ppm
1.84
1.96
1.80
1.01
3.00

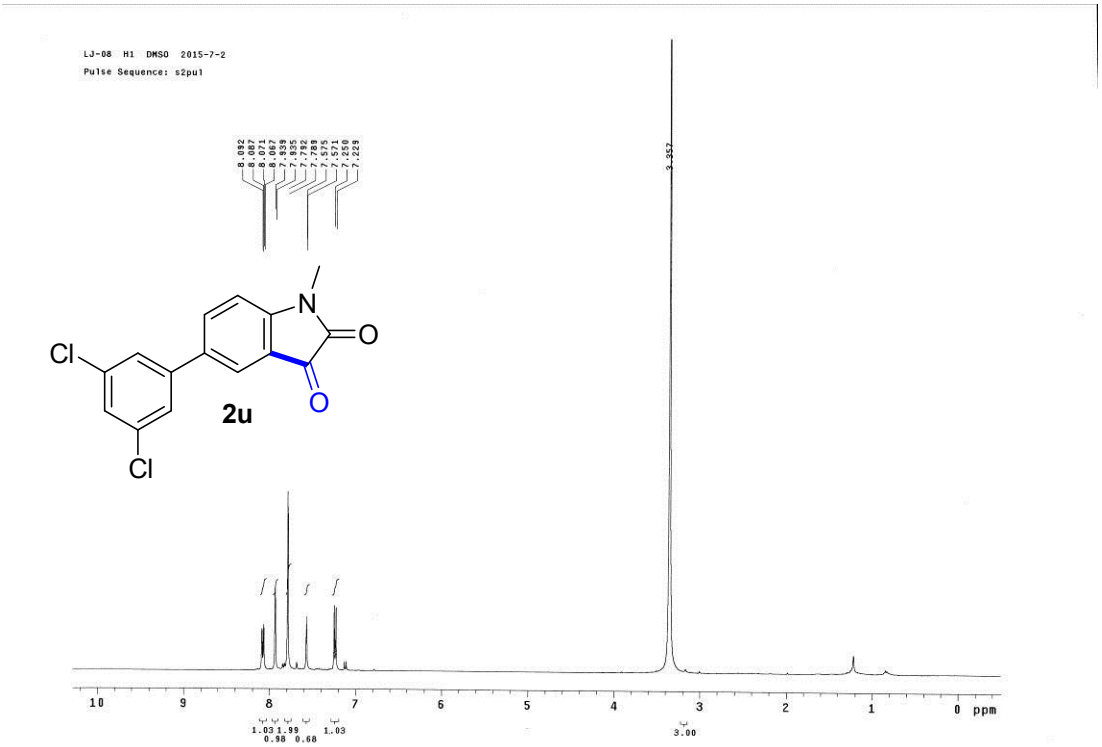

LJ-09-CDCL3-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.366
163.475
161.496
158.222
150.397
136.611
136.553
135.094
128.214
128.163
123.492
117.008
116.880
115.887
110.365
77.211
77.000
76.789
26.326
N
O
O
F
2r
200
180
160
140
120
100
80
60
40
20
ppm

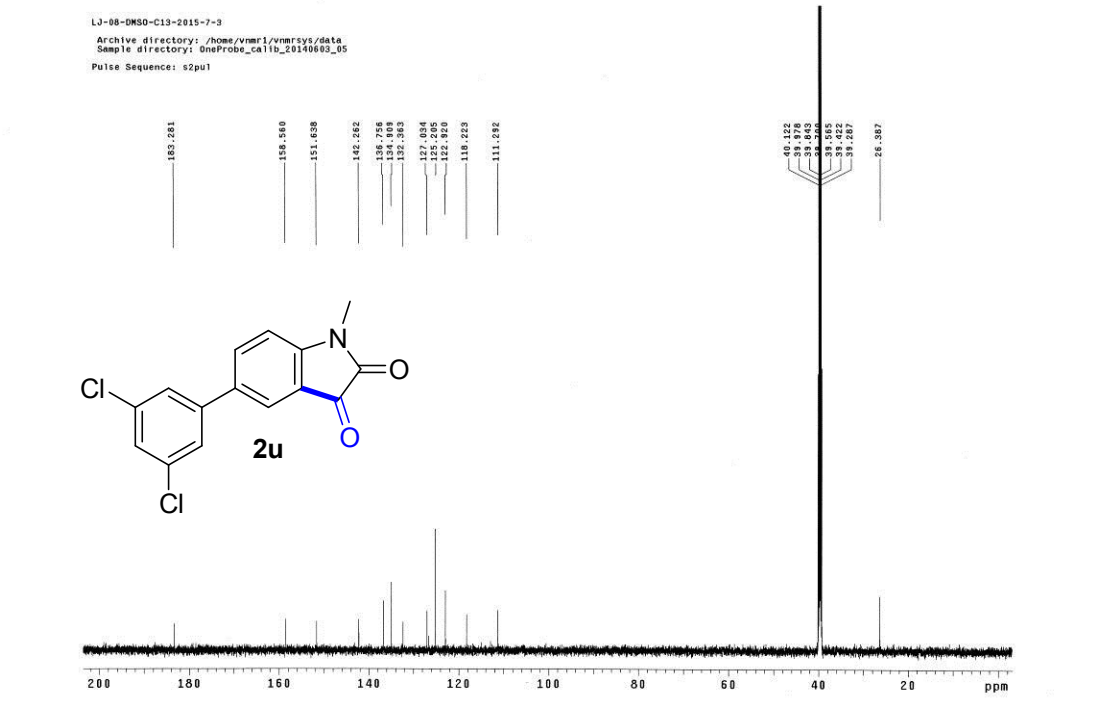

LJ-15 H1 CDCl3 2015-7-7
Pulse Sequence: s2pul
7.806
7.803
7.786
7.780
7.761
7.757
7.657
7.435
7.419
7.398
7.373
6.993
6.973
3.280
0.000
N
O
O
Cl
2s
10
9
8
7
6
5
4
3
2
1
ppm
1.10
0.92
4.17
1.00
3.58

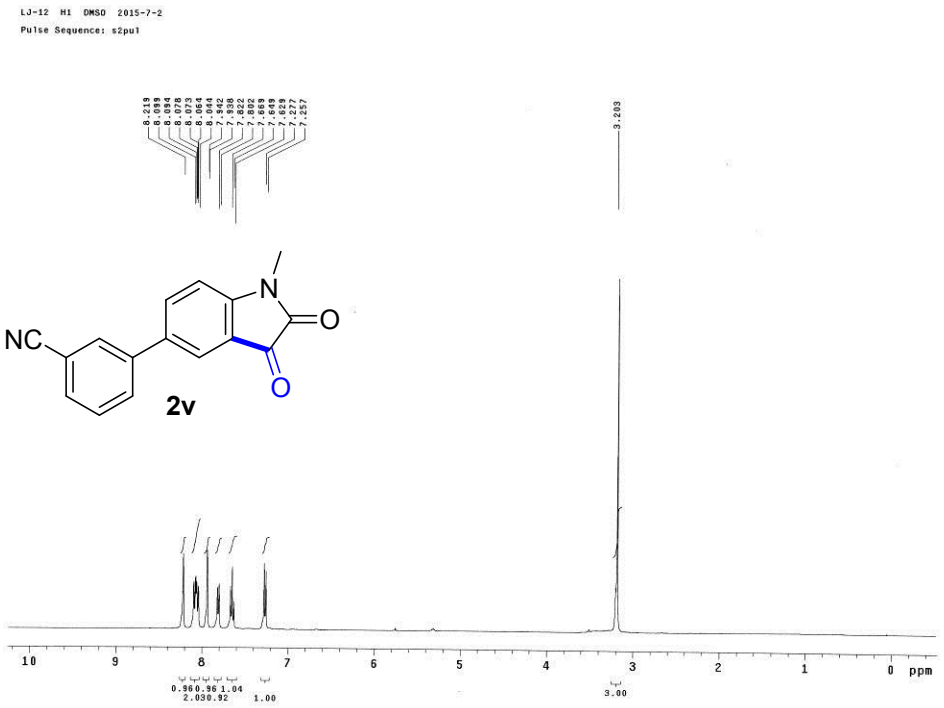

LJ-15-CDCL3-C13-2015-7-8
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
163.230
158.186
150.625
137.382
136.566
136.138
134.615
129.192
127.758
123.475
117.849
110.364
77.211
77.000
76.788
26.360
N
O
O
Cl
2s
200
180
160
140
120
100
80
60
40
20
ppm

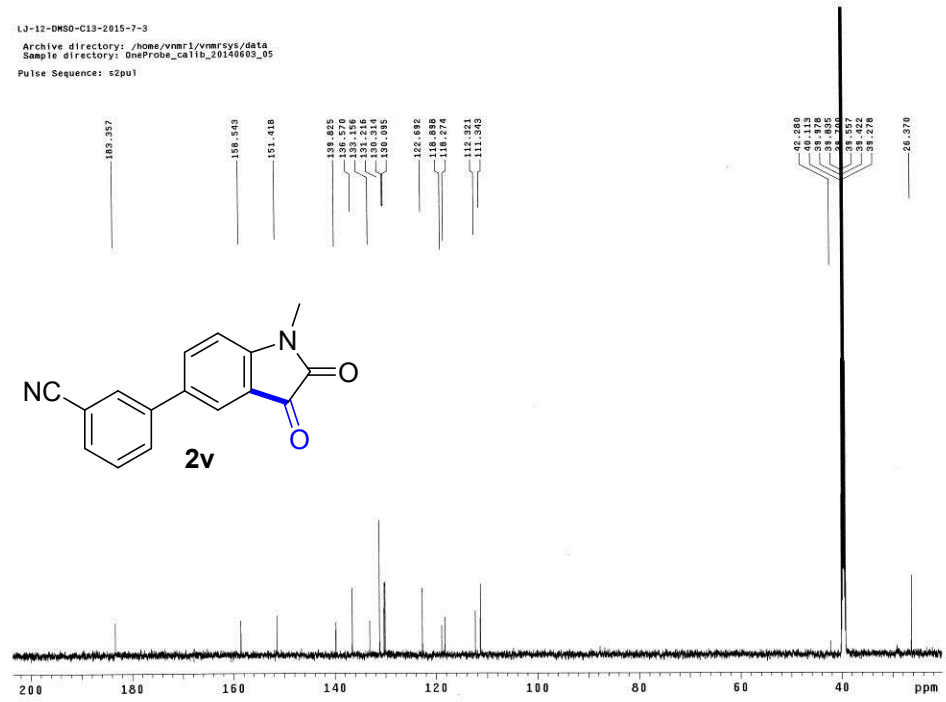

LJ-17-CDCL3-H1-2015-7-7
Pulse Sequence: s2pul
7.809
7.787
7.781
7.482
7.481
7.375
7.356
7.351
7.346
7.261
6.982
6.972
3.292
Cl
N
O
O
2t
10
9
8
7
6
5
4
3
2
1
ppm
2.28
2.82
1.44
1.03
3.17

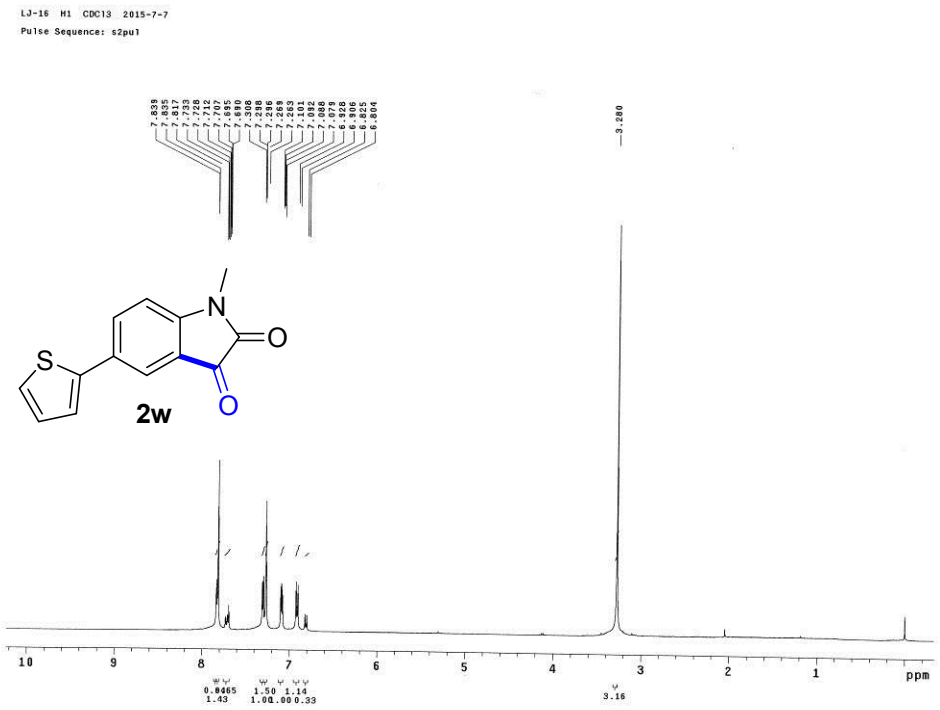

LJ-17-CDCL3-C13-2015-7-9
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.179
158.222
150.869
140.735
136.738
135.988
135.009
130.313
127.902
127.808
124.698
123.604
117.893
110.372
77.211
77.000
76.788
26.983
Cl
N
O
O
2t
200
180
160
140
120
100
80
60
40
20
ppm

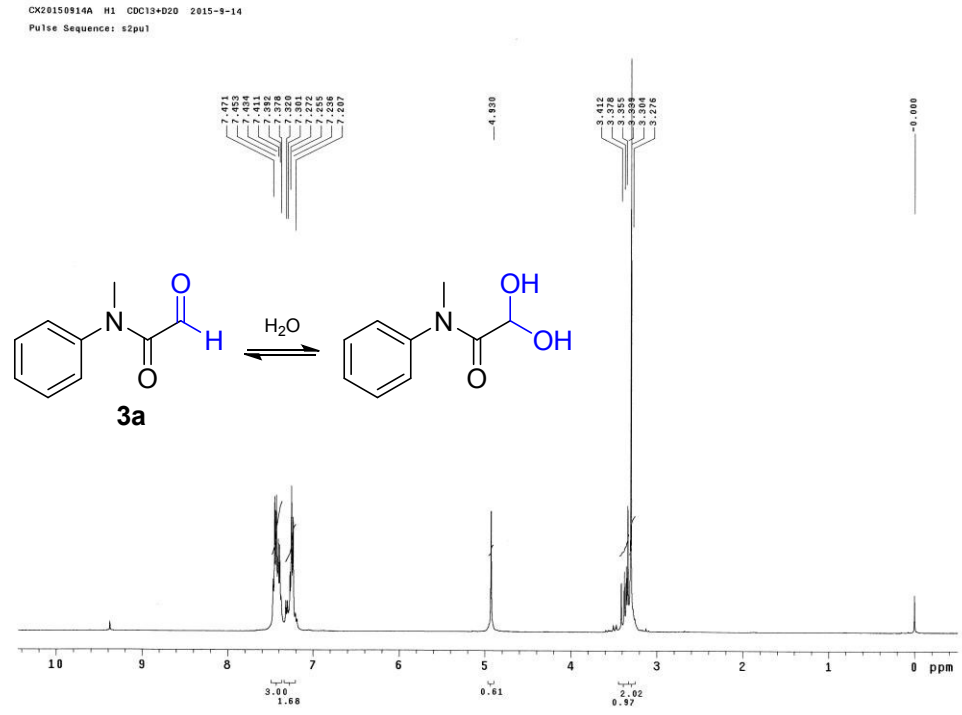

LJ-08 H1 DMSO 2015-7-2
Pulse Sequence: s2pul
8.092
8.087
8.071
8.067
7.938
7.935
7.792
7.789
7.575
7.571
7.250
7.229
3.352
N
O
O
Cl
Cl
2u
10
9
8
7
6
5
4
3
2
1
0 ppm
1.03 1.99 1.03
0.98 0.68
3.00

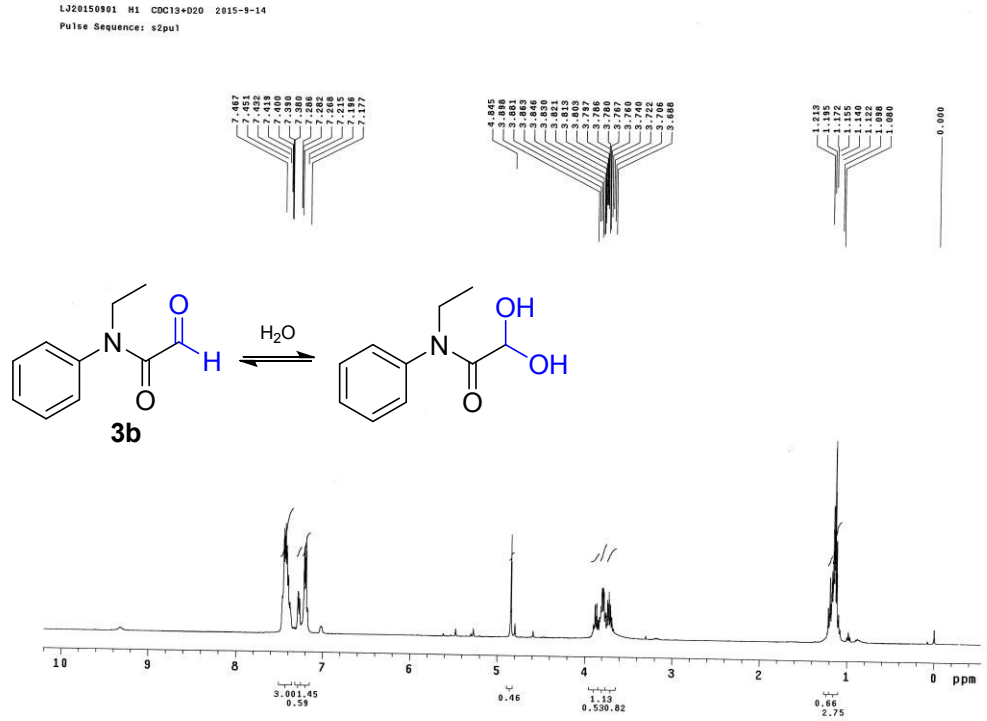

LJ-08-DMSO-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.281
158.560
151.638
142.262
136.758
134.708
132.363
127.034
125.965
122.920
118.223
111.292
40.122
39.918
39.843
39.566
39.355
39.142
38.927
38.287
26.387
N
O
O
Cl
Cl
2u
200
180
160
140
120
100
80
60
40
20
ppm

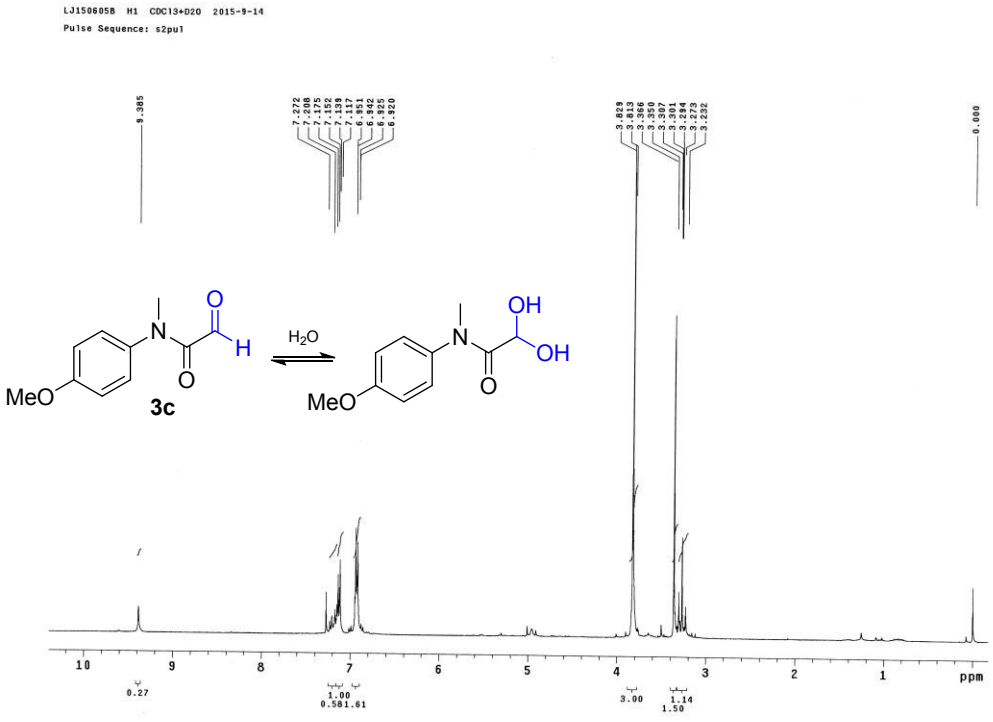

LJ-12 H1 DMSO 2015-7-2
Pulse Sequence: s2pul
8.219
8.086
8.084
8.078
8.073
8.064
8.064
7.942
7.938
7.820
7.802
7.669
7.649
7.639
7.277
7.257
3.283
NC
N
O
O
2v
10
9
8
7
6
5
4
3
2
1
0 ppm
0.96 0.96 1.04
2.03 0.92
1.00
3.00

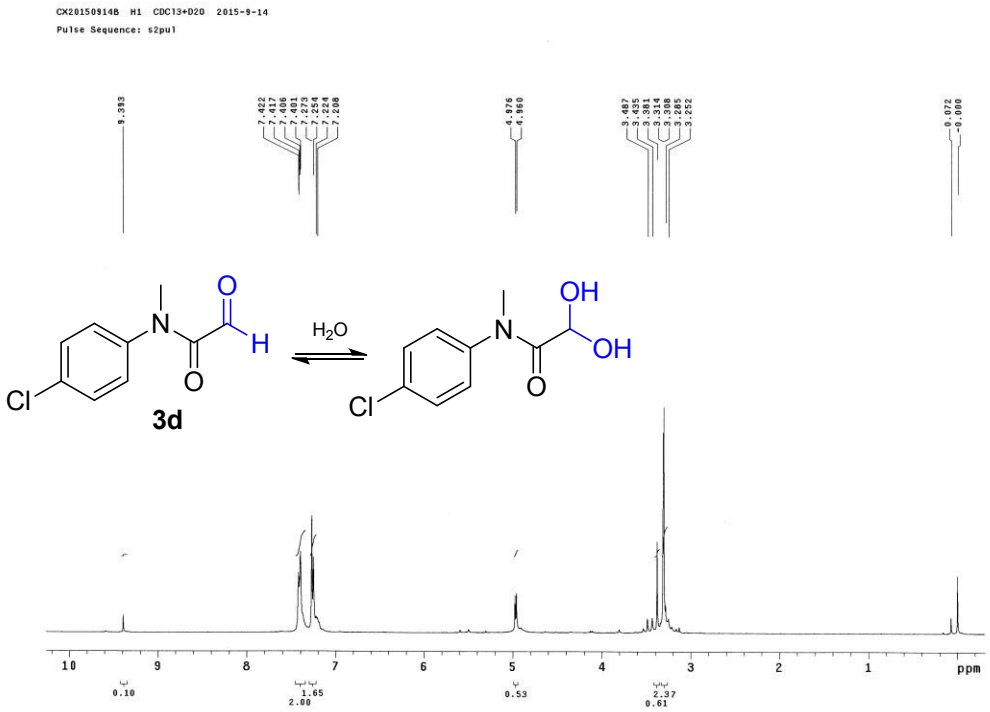

LJ-12-DMSO-C13-2015-7-3
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
183.357
150.543
151.418
139.825
136.570
133.216
131.316
130.314
130.095
122.692
118.898
118.274
112.321
111.343
42.280
40.113
39.978
39.835
39.693
39.557
39.422
39.278
26.370
NC
N
O
O
2v
200
180
160
140
120
100
80
60
40
ppm

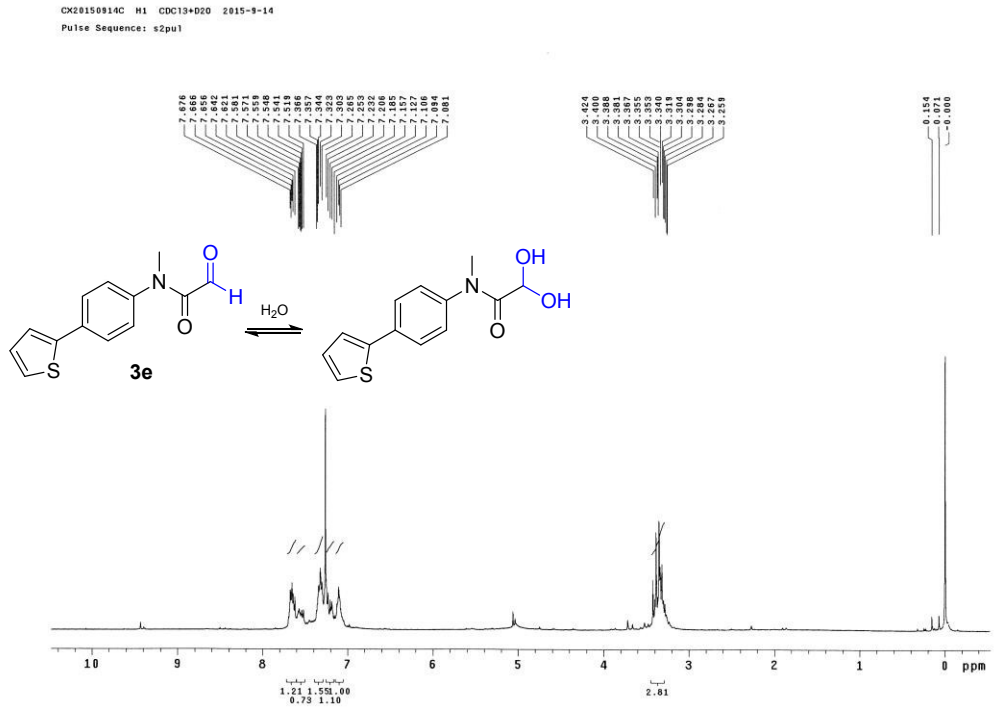

LJ-16 H1 CDCl3 2015-7-7
Pulse Sequence: s2pul
2w
ppm

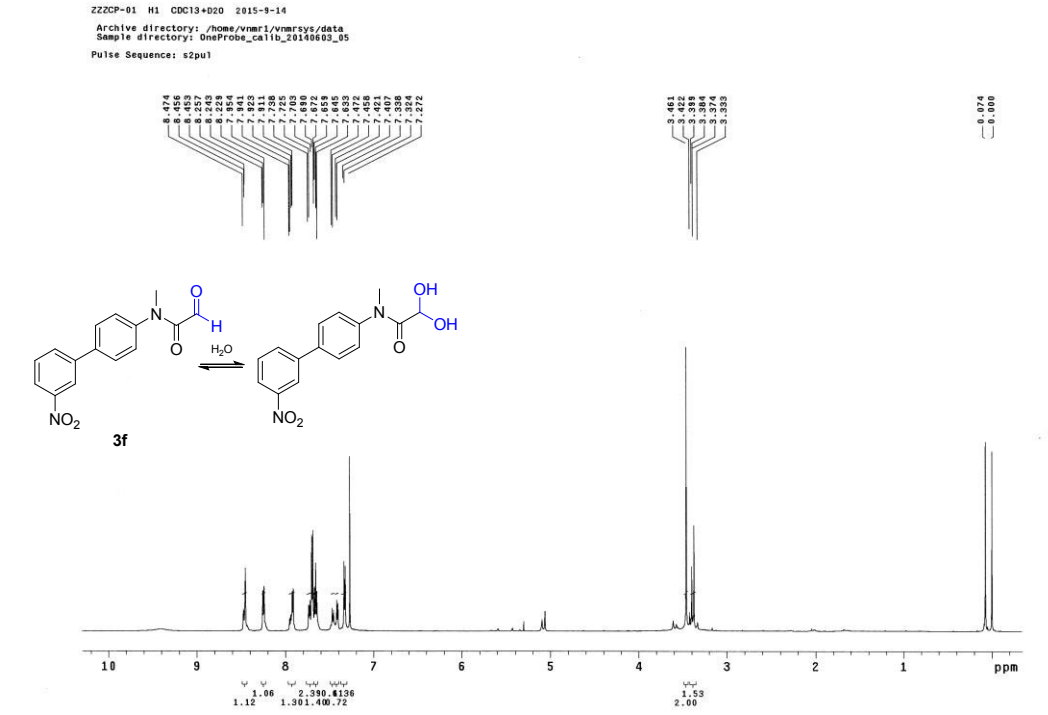

CX20150914A H1 CDCl3+D2O 2015-9-14
Pulse Sequence: s2pul
H2O
3a
ppm


LJ20150901 H1 CDCl3+D2O 2015-9-14
Pulse Sequence: s2pul
H2O
3b
ppm


LJ150605B H1 CDCl3+D2O 2015-9-14
Pulse Sequence: s2pul
H2O
MeO
3c
ppm


CX20150914B H1 CDCl3+D2O 2015-9-14
Pulse Sequence: s2pul
8.393
7.422
7.417
7.406
7.401
7.273
7.254
7.224
7.208
4.976
4.960
3.407
3.435
3.381
3.314
3.300
3.285
3.252
0.072
0.000
O
N
H
O
Cl
3d
H2O
OH
N
OH
O
Cl
10
9
8
7
6
5
4
3
2
1
ppm
0.10
1.65
2.00
0.53
2.37
0.61


CX20150914C H1 CDCl3+D2O 2015-9-14
Pulse Sequence: s2pul
7.676
7.656
7.642
7.621
7.591
7.571
7.559
7.548
7.541
7.519
7.366
7.357
7.344
7.323
7.303
7.265
7.253
7.232
7.206
7.185
7.157
7.127
7.106
7.094
7.081
3.424
3.400
3.388
3.381
3.367
3.355
3.353
3.340
3.318
3.304
3.298
3.284
3.267
3.259
0.154
0.071
0.000
O
N
H
O
S
3e
H2O
OH
N
OH
O
S
10
9
8
7
6
5
4
3
2
1
0
ppm
1.21
1.55
1.00
0.73
1.10
2.81


ZZZCP-01 H1 CDCl3+D2O 2015-9-14
Archive directory: /home/vnmrs1/vnmrsys/data
Sample directory: OneProbe_calib_20140603_05
Pulse Sequence: s2pul
8.474
8.456
8.453
8.257
8.245
8.228
7.954
7.941
7.923
7.911
7.738
7.725
7.703
7.690
7.672
7.659
7.655
7.633
7.472
7.456
7.421
7.407
7.338
7.324
7.272
3.461
3.422
3.389
3.384
3.372
3.339
0.074
0.000
O
H
N
O
H2O
OH
OH
N
O
NO2
NO2
3f
10
9
8
7
6
5
4
3
2
1
ppm
1.06
1.12
2.39
1.30
0.86
1.40
1.36
0.72
1.53
2.00