

## Supporting Information

### Visible Light-Promoted C–H Functionalization of Ethers and Electron-Deficient Arenes

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#### Experimental:

$^1\text{H}$  &  $\text{C}^{13}$  spectra were recorded on Bruker-Avance DPX FT-NMR 500 and 400 MHz instruments. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent ( $\text{CDCl}_3$ , 7.26ppm). Integration, and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz). Carbon nuclear magnetic resonance spectra ( $^{13}\text{C}$  NMR) were recorded at 100 MHz or 125 MHz. chemical data for carbons are reported in parts per million (ppm,  $\delta$  scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent. Reagents and solvents used were mostly AR grade. Silica gel coated plates were used for TLC.

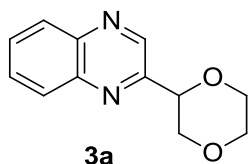
#### Experimental procedure

**General Procedure for the  $\alpha$ -Arylation of Ethers:** Oven dried round bottomed flask was charged with  $\text{K}_2\text{S}_2\text{O}_8$  (2.0 equiv), heteroarene (1.0 equiv), ether (50 equiv) and water (equal volume to ether). The round bottomed flask was closed with septum, and then irradiated with a house hold CFL bulb (27 W). The reaction mixture was kept approximately 2-5 cm away from the light source at room temperature. On reaction completion monitored through TLC (12-72 h), the reaction mixture was diluted with 15 ml of 10%  $\text{NaHCO}_3$  solution, and extracted with DCM ( $3 \times 20$  ml). The combined organic extracts were washed with brine (20 ml), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated on vacuo. Purification of the crude product on silica gel using EtOAc:Hexane as solvent system afforded the desired product.

**Note:** The reaction shouldn't be degassed and sealed with septa. The reaction times are significantly reduced with the use of two CFL bulbs.

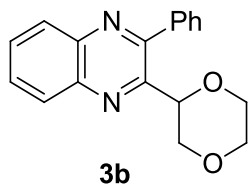
## Characterization data:

**2-(1,4-dioxan-2-yl)quinoxaline (3a):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (207 mg, 0.769 mmol), quinoxaline (50 mg, 0.384 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml).



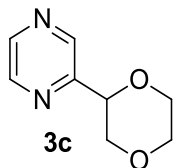
The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 12 h and the product formation was monitored by TLC, purified by column chromatography as solid product (64 mg, 77% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.06 (s, 1H), 8.15 – 8.03 (m, 2H), 7.81 – 7.71 (m, 2H), 4.99 (dd,  $J = 10.0, 2.9$  Hz, 1H), 4.27 (dd,  $J = 11.6, 2.9$  Hz, 1H), 4.02 (dtd,  $J = 14.7, 11.6, 2.8$  Hz, 2H), 3.90 – 3.75 (m, 2H), 3.71 (dd,  $J = 11.6, 10.1$  Hz, 1H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  152.6, 143.4, 142.1, 141.4, 130.2, 129.9, 129.2, 129.2, 77.0, 70.51, 67.0, 66.4; GC-MS (EI)  $m/z$  (%) 216.4 (13.3,  $M^+$ ), 188.4 (21.7), 173.3 (5.9), 157.4 (100), 144.4 (20.1), 129.4 (40.4), 103.3 (41.9).

**2-(1,4-dioxan-2-yl)-3-phenylquinoxaline (3b):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (131 mg, 0.484 mmol), 2-phenylquinoxaline (50 mg, 0.242 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml).



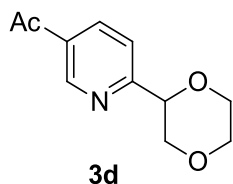
The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 12 h and the product formation was monitored by TLC, purified by column chromatography as solid product (47 mg, 67% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.25 – 8.11 (m, 9H), 7.82 – 7.70 (m, 17H), 7.55 (ddt,  $J = 6.9, 4.5, 2.4$  Hz, 11H), 5.01 (dd,  $J = 9.9, 2.5$  Hz, 4H), 4.25 (dd,  $J = 11.7, 9.9$  Hz, 5H), 3.98 (d,  $J = 9.1$  Hz, 4H), 3.92 – 3.83 (m, 12H), 3.79 (t,  $J = 9.1$  Hz, 5H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  154.7, 150.0, 141.6, 141.1, 137.9, 130.6, 130.0, 129.4, 129.3, 129.2, 129.1, 128.7, 75.0, 69.6, 67.2, 66.2; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{18}H_{17}N_2O_2$  293.1285 found 293.1294.

**2-(1,4-dioxan-2-yl)pyrazine (3c):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (337 mg, 1.25 mmol),



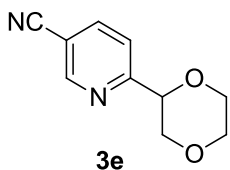
pyrazine (50 mg, 0.625 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 20 h and the product formation was monitored by TLC, purified by column chromatography as solid product (77 mg, 75% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.77 (s, 1H), 8.55 – 8.48 (m, 2H), 4.80 (dd,  $J = 10.0, 2.5$  Hz, 1H), 4.15 (dt,  $J = 7.9, 3.9$  Hz, 1H), 3.95 (dt,  $J = 7.1, 6.5, 5.8$  Hz, 2H), 3.86 – 3.80 (m, 1H), 3.80 – 3.70 (m, 1H), 3.57 (dd,  $J = 11.4, 10.2$  Hz, 1H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  153.2, 143.9, 143.5, 143.0, 76.3, 70.6, 66.8, 66.3; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_8H_{11}N_2O_2$  167.0815 found 167.0821.

**1-(6-(1,4-dioxan-2-yl)pyridin-3-yl)ethanone(3d):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (223 mg, 0.826 mmol), 1-(pyridin-3-yl)ethanone (46  $\mu$ l, 0.413 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house



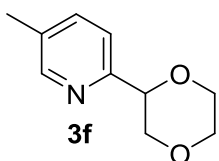
hold CFL bulb (27 W) at room temperature for 24 h and the product formation was monitored by TLC, purified by column chromatography as solid product (55 mg, 65% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.10 – 9.06 (s, 1H), 8.26 (dd,  $J = 8.2, 2.2$  Hz, 1H), 7.61 (d,  $J = 8.2$  Hz, 1H), 4.81 (dd,  $J = 10.0, 2.9$  Hz, 1H), 4.20 (dd,  $J = 11.5, 2.9$  Hz, 1H), 4.02 – 3.92 (m, 2H), 3.86 – 3.80 (m, 1H), 3.73 (ddd,  $J = 11.7, 10.5, 3.7$  Hz, 1H), 3.48 (dd,  $J = 11.5, 10.0$  Hz, 1H), 2.63 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  196.4, 162.4, 149.2, 136.3, 131.4, 120.4, 70.9, 66.8, 66.3, 26.7; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{11}H_{14}NO_3$  208.0968 found 208.0969.

**6-(1,4-dioxan-2-yl)nicotinonitrile (3e):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (259 mg, 0.961 mmol), nicotinonitrile (50 mg, 0.480 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml).



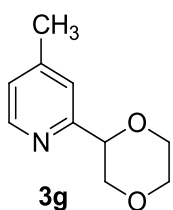
The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 32 h and the product formation was monitored by TLC, purified by column chromatography as oil product (52 mg, 58% from heteroarene).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.80 (dd,  $J = 4.9, 1.7$  Hz, 1H), 8.01 (dd,  $J = 7.9, 1.7$  Hz, 1H), 7.39 (dd,  $J = 7.9, 4.9$  Hz, 1H), 5.08 (dd,  $J = 10.0, 2.8$  Hz, 1H), 4.07 – 3.99 (m, 3H), 3.87 – 3.81 (m, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  159.6, 152.4, 141.2, 122.9, 115.8, 108.9, 76.7, 69.4, 67.1, 66.2; HRMS (TOF)  $m/z$   $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{10}\text{H}_{11}\text{N}_2\text{O}_2$  191.0815 found 191.0819.

**4-(1,4-dioxan-2-yl)-3-methylpyridine (3f):** The title compound was prepared according to



the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (290 mg, 1.07 mmol), 3-methylpyridine (50  $\mu\text{l}$ , 0.537 mmol) in 1,4-dioxane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 32 h and the product formation was monitored by TLC, purified by column chromatography as oil product (50 mg, 52% from heteroarene).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.46 (t,  $J = 7.5$  Hz, 1H), 8.37 (s, 1H), 7.41 (d,  $J = 4.9$  Hz, 1H), 4.77 (dt,  $J = 12.7, 6.4$  Hz, 1H), 3.97 – 3.91 (m, 2H), 3.83 (dd,  $J = 11.0, 7.2$  Hz, 2H), 3.75 (dd,  $J = 8.5, 2.8$  Hz, 1H), 3.33 (dd,  $J = 11.6, 10.2$  Hz, 1H), 2.31 (d,  $J = 8.9$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  150.6, 147.8, 145.2, 129.7, 120.5, 74.20, 70.5, 67.0, 66.3, 15.8; GC-MS (EI)  $m/z$  (%) 180.3 (100,  $[\text{M}+\text{H}]^+$ ), 117.3 (15.3).

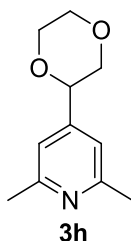
**2-(1,4-dioxan-2-yl)-4-methylpyridine (3g):** The title compound was prepared according to



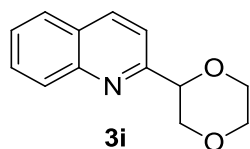
the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (290 mg, 1.07 mmol), 4-methylpyridine (50  $\mu\text{l}$ , 0.537 mmol) in 1,4-dioxane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 48 h and the product formation was monitored by TLC, purified by column chromatography as solid product (51 mg, 54% from heteroarene).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 8.2$  Hz, 1H), 7.28 (s, 1H), 7.02 (t,  $J = 8.1$  Hz, 1H), 4.71 (dd,  $J = 10.1, 2.8$  Hz,

1H), 4.12 (dt,  $J = 6.5, 3.3$  Hz, 1H), 3.96 – 3.91 (m, 2H), 3.82 – 3.77 (m, 1H), 3.73 – 3.68 (m, 1H), 3.54 – 3.47 (m, 1H), 2.35 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.5, 148.6, 148.2, 123.8, 121.6, 78.0, 71.3, 66.9, 66.3, 21.1; HRMS (TOF)  $m/z$   $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{10}\text{H}_{14}\text{NO}_2$  180.1019 found 180.1015.

**4-(1,4-dioxan-2-yl)-2,6-dimethylpyridine (3h):** The title compound was prepared according to the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (252 mg, 0.934 mmol), 2,6-dimethylpyridine (50  $\mu\text{l}$ , 0.467 mmol) in 1,4-dioxane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 18 h and the product formation was monitored by TLC, purified by column chromatography as solid product (50 mg, 56% from heteroarene).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.92 (s, 2H), 4.54 (dd,  $J = 10.1, 2.5$  Hz, 1H), 3.93 (dd,  $J = 11.8, 2.6$  Hz, 1H), 3.91 – 3.82 (m, 2H), 3.79 (dd,  $J = 11.7, 1.8$  Hz, 1H), 3.69 (td,  $J = 11.5, 3.1$  Hz, 1H), 3.36 (dd,  $J = 11.5, 10.3$  Hz, 1H), 2.50 (s, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.9, 147.5, 117.5, 76.5, 71.9, 66.8, 66.3, 24.3; HRMS (TOF)  $m/z$   $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{11}\text{H}_{16}\text{NO}_2$  194.1176 found 194.1171.

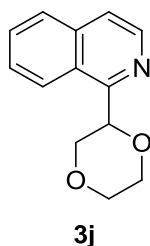


**2-(1,4-dioxan-2-yl)quinoline (3i):** The title compound was prepared according to the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (209 mg, 0.775 mmol), quinoline (50  $\mu\text{l}$ , 0.387 mmol) in 1,4-dioxane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 72 h and the product formation was monitored by TLC, purified by column chromatography as solid product (51 mg, 62% from heteroarene).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.93 (d,  $J = 4.5$  Hz, 1H), 8.15 (dd,  $J = 8.4, 0.5$  Hz, 1H), 8.01 (t,  $J = 7.5$  Hz, 1H), 7.75 – 7.69 (m, 1H), 7.62 – 7.56 (m, 2H), 5.38 (dd,  $J = 9.9, 2.4$  Hz, 1H), 4.13 (dt,  $J = 11.4, 2.9$  Hz, 1H), 4.07 (dt,  $J = 6.4, 4.1$  Hz, 2H), 3.92 – 3.87 (m, 1H), 3.84 – 3.79 (m, 1H), 3.51 – 3.44 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  150.4, 148.0, 143.6, 130.4, 129.2, 126.9, 125.3, 122.5, 118.3, 74.1, 71.9, 67.3,



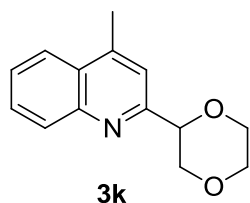
66.6; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{13}H_{14}NO_2$  216.1019 found 216.1016.

**1-(1,4-dioxan-2-yl)isoquinoline (3j):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (209 mg, 0.775 mmol), isoquinoline (50  $\mu$ l, 0.387 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml).



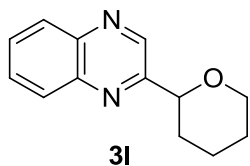
The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 72 h and the product formation was monitored by TLC, purified by column chromatography as solid product (55 mg, 66% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.43 (d,  $J = 5.6$  Hz, 1H), 8.22 (d,  $J = 8.3$  Hz, 1H), 7.73 (d,  $J = 8.0$  Hz, 1H), 7.62 – 7.48 (m, 3H), 5.41 – 5.34 (m, 1H), 4.13 – 3.93 (m, 4H), 3.86 – 3.74 (m, 2H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  155.9, 141.7, 136.3, 130.0, 127.4, 127.4, 126.4, 124.6, 121.0, 75.7, 70.2, 67.5, 66.4; GC-MS (EI)  $m/z$  (%) 216.4.4 (20.2,  $[M+H]^+$ ), 156.4 (100), 129.4 (33.7), 102.3 (15.7).

**1-(1,4-dioxan-2-yl)-4-methylisoquinoline (3k):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (188 mg, 0.699 mmol), 4-methylquinoline (50  $\mu$ l, 0.349 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 72 h and the product formation was monitored



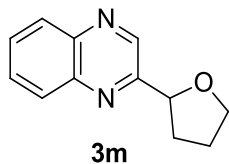
by TLC, purified by column chromatography as solid product (53 mg, 67% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.07 (d,  $J = 8.4$  Hz, 1H), 7.97 (d,  $J = 8.4$  Hz, 1H), 7.69 (t,  $J = 7.6$  Hz, 1H), 7.54 (dd,  $J = 8.2$ , 7.0 Hz, 1H), 7.45 (s, 1H), 4.89 (dd,  $J = 10.1$ , 2.9 Hz, 1H), 4.23 (dd,  $J = 11.6$ , 2.9 Hz, 1H), 4.06 – 3.95 (m, 2H), 3.87 – 3.75 (m, 2H), 3.63 (dd,  $J = 11.6$ , 10.2 Hz, 1H), 2.71 (s, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  157.8, 147.3, 145.2, 129.8, 129.3, 127.6, 126.2, 123.7, 119.1, 78.8, 71.1, 67.1, 66.4, 18.8; HRMS (TOF)  $m/z$   $[M + H]^+$  Calcd for  $C_{14}H_{16}NO_2$  230.1176 found 230.1172.

**2-(tetrahydro-2H-pyran-2-yl)quinoxaline (3l):** The title compound was prepared according



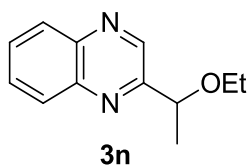
to the general procedure described above using  $K_2S_2O_8$  (207 mg, 0.768 mmol), quinoxaline (50 mg, 0.384 mmol) in tetrahydro-2H-pyran (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 36 h and the product formation was monitored by TLC, purified by column chromatography as solid product (52 mg, 64% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.04 (s, 1H), 8.09 (ddd,  $J = 9.7, 6.8, 3.9$  Hz, 2H), 7.78 – 7.71 (m, 2H), 4.74 – 4.67 (m, 1H), 4.25 (dd,  $J = 10.5, 2.8$  Hz, 1H), 3.71 (td,  $J = 11.5, 2.3$  Hz, 1H), 2.13 (d,  $J = 10.8$  Hz, 1H), 2.02 (d,  $J = 4.7$  Hz, 1H), 1.73 (ddd,  $J = 29.4, 14.1, 8.2$  Hz, 4H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  156.6, 144.9, 143.68, 142.98, 130.11, 129.47, 79.80, 68.97, 32.33, 25.64, 23.50; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{13}H_{15}N_2O_2$  215.1179 found 215.1188.

**2-(tetrahydrofuran-2-yl)quinoxaline (3m):** The title compound was prepared according to



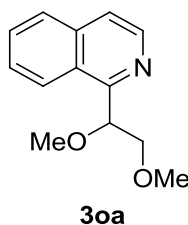
to the general procedure described above using  $K_2S_2O_8$  (207 mg, 0.768 mmol), quinoxaline (50 mg, 0.384 mmol) in tetrahydrofuran (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 36 h and the product formation was monitored by TLC, purified by column chromatography as solid product (47 mg, 62% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.04 (s, 3H), 8.15 – 8.09 (m, 7H), 7.76 – 7.69 (m, 6H), 5.23 (t,  $J = 7.0$  Hz, 3H), 4.24 – 4.00 (m, 7H), 2.53 (dt,  $J = 13.5, 7.0$  Hz, 4H), 2.22 – 1.99 (m, 11H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  157.6, 143.5, 141.8, 141.5, 130.0, 129.4, 129.0, 80.4, 69.4, 32.9, 25.9; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{12}H_{13}N_2O$  201.1022 found 201.1048.

**2-(1-ethoxyethyl)quinoxaline (3n):** The title compound was prepared according to the general procedure described above using  $K_2S_2O_8$  (207 mg, 0.768 mmol), quinoxaline (50 mg, 0.384 mmol) in ethoxyethane (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room



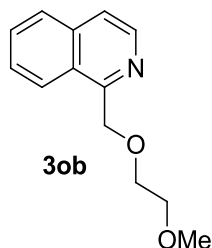
temperature for 24 h and the product formation was monitored by TLC, purified by column chromatography as solid product (40 mg, 52% from heteroarene).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.06 (s, 1H), 8.10 (ddd,  $J = 9.6, 6.8, 3.9$  Hz, 2H), 7.81 – 7.72 (m, 2H), 4.77 (q,  $J = 6.6$  Hz, 1H), 3.63 – 3.41 (m, 2H), 1.62 (d,  $J = 6.7$  Hz, 2H), 1.26 (t,  $J = 6.8$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  158.4, 143.6, 142.0, 141.5, 130.1, 129.5, 129.2, 129.0, 78.2, 65.0, 22.2, 15.4; HRMS (TOF)  $m/z$   $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{12}\text{H}_{15}\text{N}_2\text{O}$  203.1179 found 203.1192.

**1-(1,2-dimethoxyethyl)isoquinoline (3oa):** The title compound was prepared according to



the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (209 mg, 0.775 mmol), isoquinoline (50  $\mu\text{l}$ , 0.387 mmol) in 1,2-dimethoxyethane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 72 h and the product formation was monitored by TLC, purified by column chromatography as solid product (37 mg, 45% from heteroarene).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.62 (d,  $J = 8.6$  Hz, 1H), 8.57 (d,  $J = 5.6$  Hz, 1H), 7.88 (d,  $J = 8.2$  Hz, 1H), 7.75 – 7.70 (m, 1H), 7.67 – 7.62 (m, 2H), 5.31 (dd,  $J = 8.1, 3.7$  Hz, 1H), 4.09 (dd,  $J = 10.6, 8.1$  Hz, 1H), 3.77 (dd,  $J = 10.6, 3.7$  Hz, 1H), 3.46 (d,  $J = 1.0$  Hz, 3H), 3.42 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6, 141.8, 136.6, 130.1, 127.4, 127.3, 127.0, 125.1, 120.9, 83.6, 75.3, 59.3, 57.3; GC-MS (EI)  $m/z$  (%) 218.4 (100,  $[\text{M}+\text{H}]^+$ ), 186.4 (29.6).

**1-((2-methoxyethoxy)methyl)isoquinoline (3ob):** The title compound was prepared

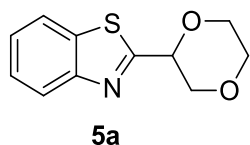


according to the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (209 mg, 0.775 mmol), isoquinoline (50  $\mu\text{l}$ , 0.387 mmol) in 1,2-dimethoxyethane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 72 h and the product formation was monitored by TLC, purified by column chromatography as solid product (16 mg, 20% from heteroarene).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 5.7$  Hz, 1H), 8.32 (d,  $J = 8.4$  Hz,



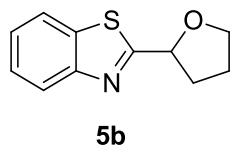
1H), 7.74 (d,  $J = 8.1$  Hz, 1H), 7.60 (t,  $J = 7.3$  Hz, 1H), 7.53 (t,  $J = 6.3$  Hz, 2H), 5.08 (s, 2H), 3.63 (dd,  $J = 5.5, 3.8$  Hz, 2H), 3.50 – 3.45 (m, 2H), 3.27 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.9, 140.0, 135.4, 129.3, 126.4, 126.0 (2C), 124.8, 120.3, 72.4, 70.7, 68.6, 57.9.

**2-(1,4-dioxan-2-yl)benzo[d]thiazole (5a):** The title compound was prepared according to the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (200 mg, 0.740



mmol), 4-benzo[d]thiazole (50  $\mu\text{l}$ , 0.370 mmol) in 1,4-dioxane (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 20 h and the product formation was monitored by TLC, purified by column chromatography as solid product (37 mg, 46% from heteroarene).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 (d,  $J = 8.2$  Hz, 1H), 7.98 – 7.94 (d,  $J = 8.3$  Hz, 1H), 7.55 – 7.50 (t,  $J = 7.5$  Hz, 1H), 7.44 (t,  $J = 7.6$  Hz, 1H), 5.11 (dd,  $J = 9.7, 3.1$  Hz, 1H), 4.35 (dd,  $J = 11.7, 3.0$  Hz, 1H), 4.10 – 3.98 (m, 2H), 3.89 (d,  $J = 11.0$  Hz, 1H), 3.85 – 3.78 (m, 1H), 3.75 (dd,  $J = 11.5, 9.8$  Hz, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  169.0, 153.0, 134.5 126.16 125.2, 123.1, 121.8, 75.4, 70.5, 67.0, 66.4; HRMS (TOF)  $m/z$   $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{11}\text{H}_{12}\text{NO}_2\text{S}$  222.0583 found 222.0590.

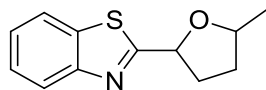
**2-(tetrahydrofuran-2-yl)benzo[d]thiazole (5b):** The title compound was prepared according to the general procedure described above using  $\text{K}_2\text{S}_2\text{O}_8$  (200 mg, 0.740



mmol), 4-benzo[d]thiazole (50  $\mu\text{l}$ , 0.370 mmol) in tetrahydrofuran (2 ml) and  $\text{H}_2\text{O}$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 36 h and the product formation was monitored by TLC, purified by column chromatography as solid product (46 mg, 61% from heteroarene).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 (d,  $J = 8.1$  Hz, 1H), 7.88 (d,  $J = 8.0$  Hz, 1H), 7.49 – 7.43 (m, 1H), 7.39 – 7.33 (m, 1H), 5.35 (dd,  $J = 7.8, 5.4$  Hz, 1H), 4.16 (dt,  $J = 8.1, 6.6$  Hz, 1H), 4.00 (dd,  $J = 15.2, 7.1$  Hz, 1H), 2.52 (dq,  $J = 12.6, 7.6$  Hz, 1H), 2.26 (qd,  $J = 12.6, 6.8$  Hz, 1H), 2.11 – 1.98 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$

176.3, 153.7, 134.8, 125.9, 124.7, 122.8, 121.7, 78.7, 69.4, 33.3, 25.7; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{11}H_{12}NOS$  206.0634 found 206.0636.

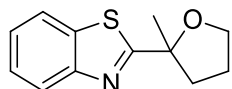
**2-(5-methyltetrahydrofuran-2-yl)benzo[d]thiazole (5ca):** The title compound was prepared



**5ca**

according to the general procedure described above using  $K_2S_2O_8$  (200mg, 0.740 mmol), 4-benzo[d]thiazole (50  $\mu$ l, 0.370 mmol) in 2-methyltetrahydrofuran (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 36 h and the product formation was monitored by TLC, purified by column chromatography as solid product (34 mg, 42% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.90 (d,  $J = 8.1$  Hz, 1H), 7.81 (d,  $J = 7.9$  Hz, 1H), 7.42 – 7.36 (m, 1H), 7.32 – 7.26 (m, 1H), 5.28 (dd,  $J = 8.3, 4.6$  Hz, 1H), 4.20 (dp,  $J = 8.6, 6.0$  Hz, 1H), 2.47 (dddd,  $J = 16.0, 12.8, 6.5, 3.0$  Hz, 1H), 2.21 (ddt,  $J = 10.4, 8.1, 5.1$  Hz, 1H), 2.08 – 1.99 (m, 1H), 1.59 (t,  $J = 6.0$  Hz, 1H), 1.38 – 1.34 (m, 3H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  177.3, 153.7, 134.7, 125.9, 124.7, 122.7, 121.73, 78.9, 77.7, 33.9, 32.6, 29.7, 21.1; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{12}H_{14}NOS$  220.0791 found 220.0808.

**2-(2-methyltetrahydrofuran-2-yl)benzo[d]thiazole (5cb):** The title compound was prepared



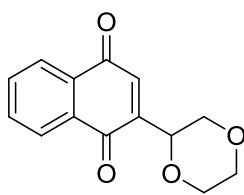
**5cb**

according to the general procedure described above using  $K_2S_2O_8$  (200 mg, 0.740 mmol), 4-benzo[d]thiazole (50  $\mu$ l, 0.370 mmol) in 2-methyltetrahydrofuran (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 36 h and the product formation was monitored by TLC, purified by column chromatography as solid product (17 mg, 22% from heteroarene).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.98 (d,  $J = 8.1$  Hz, 1H), 7.88 (d,  $J = 7.9$  Hz, 1H), 7.46 (t,  $J = 7.7$  Hz, 1H), 7.36 (t,  $J = 7.5$  Hz, 1H), 4.09 (t,  $J = 6.8$  Hz, 2H), 2.63 (ddd,  $J = 12.3, 7.6, 4.8$  Hz, 1H), 2.16 (dt,  $J = 12.3, 8.1$  Hz, 1H), 2.07 – 1.93 (m, 2H), 1.74 (s, 3H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  180.8,

154.0, 135.2, 125.8, 124.7, 122.7, 121.8, 85.0, 69.1, 39.2, 29.7, 27.9, 26.1; HRMS (TOF)  $m/z$   $[M+H]^+$  Calcd for  $C_{12}H_{14}NOS$  220.0791 found 220.0797.

**2-(1,4-dioxan-2-yl)naphthalene-1,4-dione (7a):** The title compound was prepared according

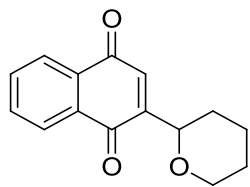
to the general procedure described above using  $K_2S_2O_8$  (170 mg, 0.632 mmol), naphthalene-1,4-dione (50 mg, 0.316 mmol) in 1,4-dioxane (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 18 h and the product formation was



monitored by TLC, purified by column chromatography as solid product (43 mg, 56% from naphthalene-1,4-dione).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.07 (dd,  $J = 8.5, 4.4$  Hz, 2H), 7.78 – 7.72 (m, 2H), 7.11 (s, 1H), 4.85 (d,  $J = 9.4$  Hz, 1H), 4.17 (dd,  $J = 11.1, 2.2$  Hz, 1H), 3.95 (qd,  $J = 11.7, 2.8$  Hz, 2H), 3.83 (d,  $J = 12.0$  Hz, 1H), 3.68 (td,  $J = 11.3, 3.7$  Hz, 1H), 3.29 – 3.22 (m, 1H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  184.8, 183.9, 147.3, 134.9, 134.7, 134.0, 133.8, 127.5, 126.5, 126.3, 71.9, 71.6, 67.0, 66.4; GC-MS (EI)  $m/z$  (%) 244.4 (19.6,  $[M]^+$ ), 200.4 (86.3), 172.3 (74.9), 102.3 (100), 76.2 (51.4).

**2-(tetrahydro-2H-pyran-2-yl)naphthalene-1,4-dione (7b):** The title compound was

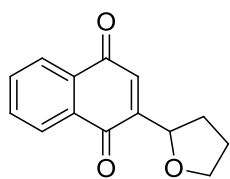
prepared according to the general procedure described above using  $K_2S_2O_8$  (170 mg, 0.632 mmol), naphthalene-1,4-dione (50 mg, 0.316 mmol) in tetrahydro-2H-pyran (2 ml) and  $H_2O$  (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 24



h and the product formation was monitored by TLC, purified by column chromatography as solid product (39 mg, 52% from naphthalene-1,4-dione).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.07 (dd,  $J = 8.8, 4.2$  Hz, 2H), 7.73 (dd,  $J = 5.3, 3.7$  Hz, 2H), 7.06 (d,  $J = 1.2$  Hz, 1H), 4.54 (d,  $J = 10.9$  Hz, 1H), 4.14 (t,  $J = 12.5$  Hz, 1H), 3.62 (td,  $J = 11.4, 2.8$  Hz, 1H), 2.05 (d,  $J = 14.1$  Hz, 1H), 1.92 (d,  $J = 10.2$  Hz, 1H), 1.75 – 1.59 (m, 4H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  185.3, 184.3, 151.9, 133.8, 133.6, 133.2, 132.2,

131.9, 126.4, 126.1, 73.4, 68.9, 33.0, 25.8, 23.7; GC-MS (EI) m/z (%) 243.4 (100, [M+H]<sup>+</sup>), 224.4 (21.4), 214.4 (35.3), 197.3 (44.8), 102.2 (77.2).

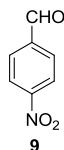
**2-(tetrahydrofuran-2-yl)naphthalene-1,4-dione (7c):** The title compound was prepared



**7c**

according to the general procedure described above using K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (170 mg, 0.632 mmol), naphthalene-1,4-dione (50 mg, 0.316 mmol) in tetrahydrofuran (2 ml) and H<sub>2</sub>O (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 32 h and the product formation was monitored by TLC, purified by column chromatography as solid product (34 mg, 48% from naphthalene-1,4-dione). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.12 – 8.01 (m, 2H), 7.78 – 7.69 (m, 2H), 7.04 (s, 1H), 5.04 (t, *J* = 7.1 Hz, 1H), 4.00 (dq, *J* = 12.0, 7.5 Hz, 2H), 2.52 (td, *J* = 14.3, 7.4 Hz, 1H), 1.98 (ddt, *J* = 19.0, 12.2, 6.1 Hz, 2H), 1.77 – 1.67 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 185.4, 185.1, 152.4, 133.9, 133.7, 132.3, 132.1, 132.1, 126.3, 126.2, 74.9, 68.9, 32.9, 25.8; HRMS (TOF) m/z [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>13</sub>O<sub>3</sub> 229.0859 found 229.0872.

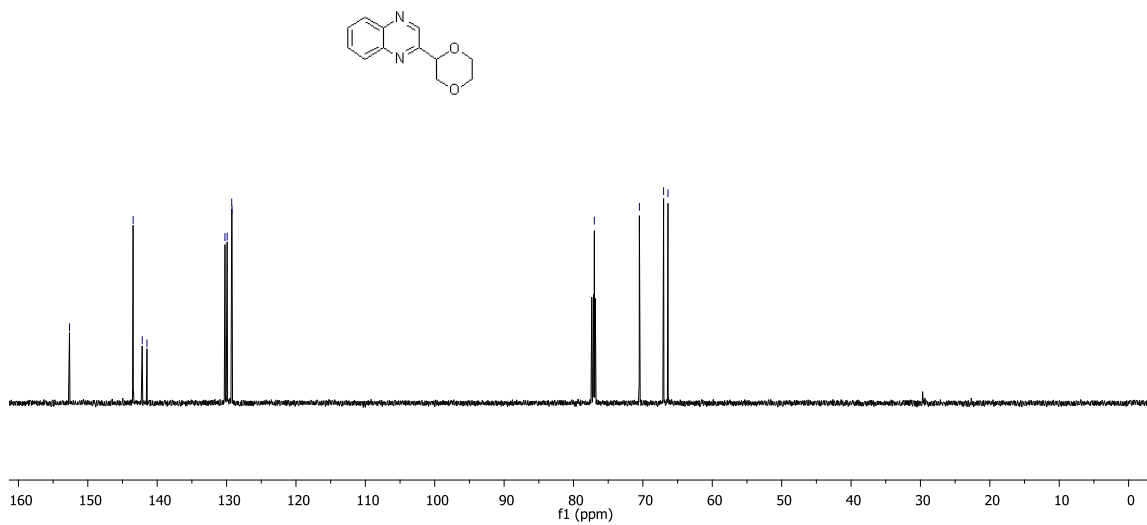
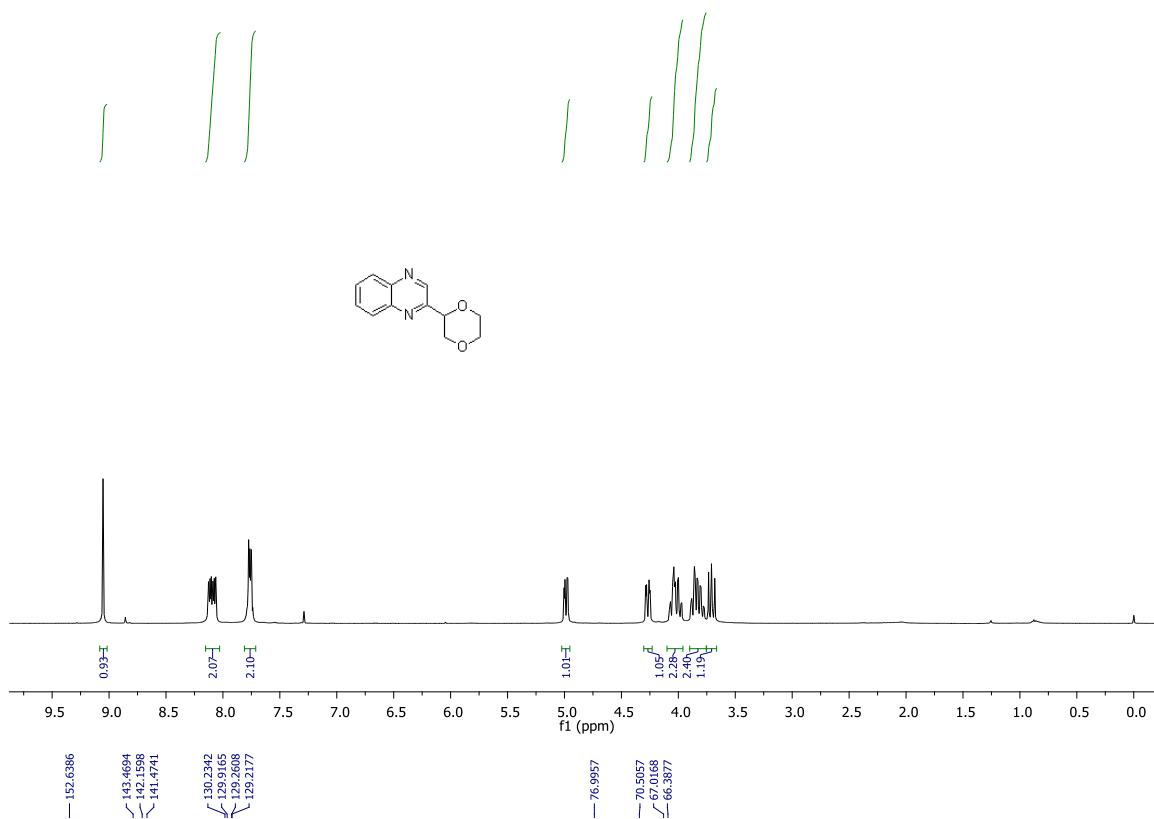
**4-nitrobenzaldehyde (9):** The title compound was prepared according to the general



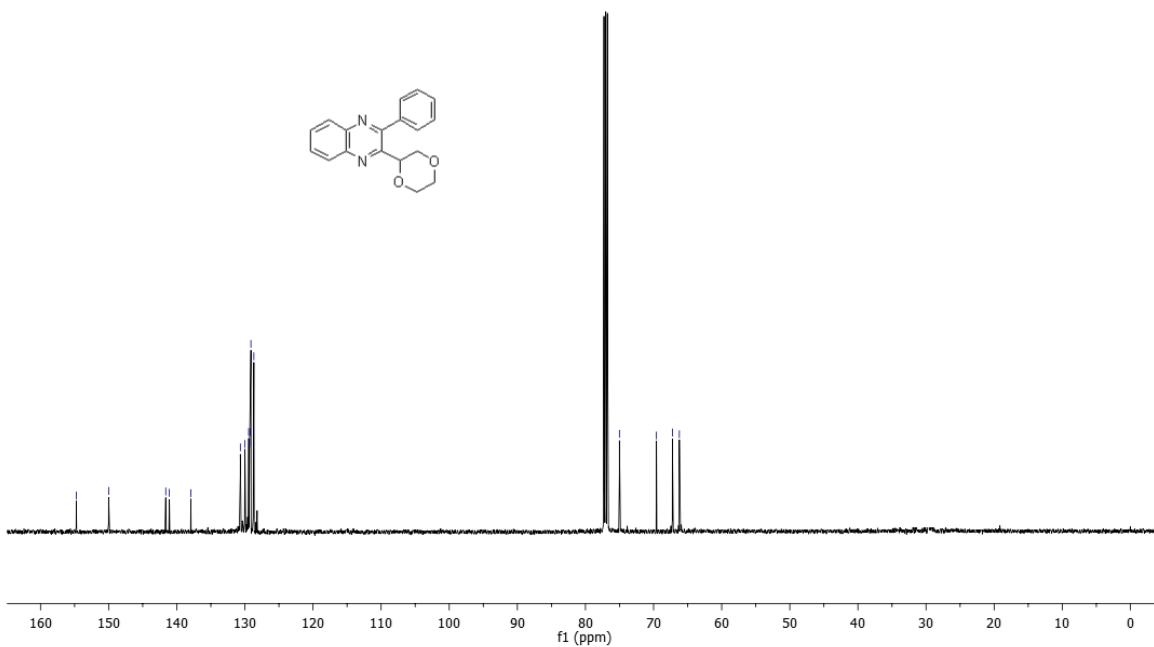
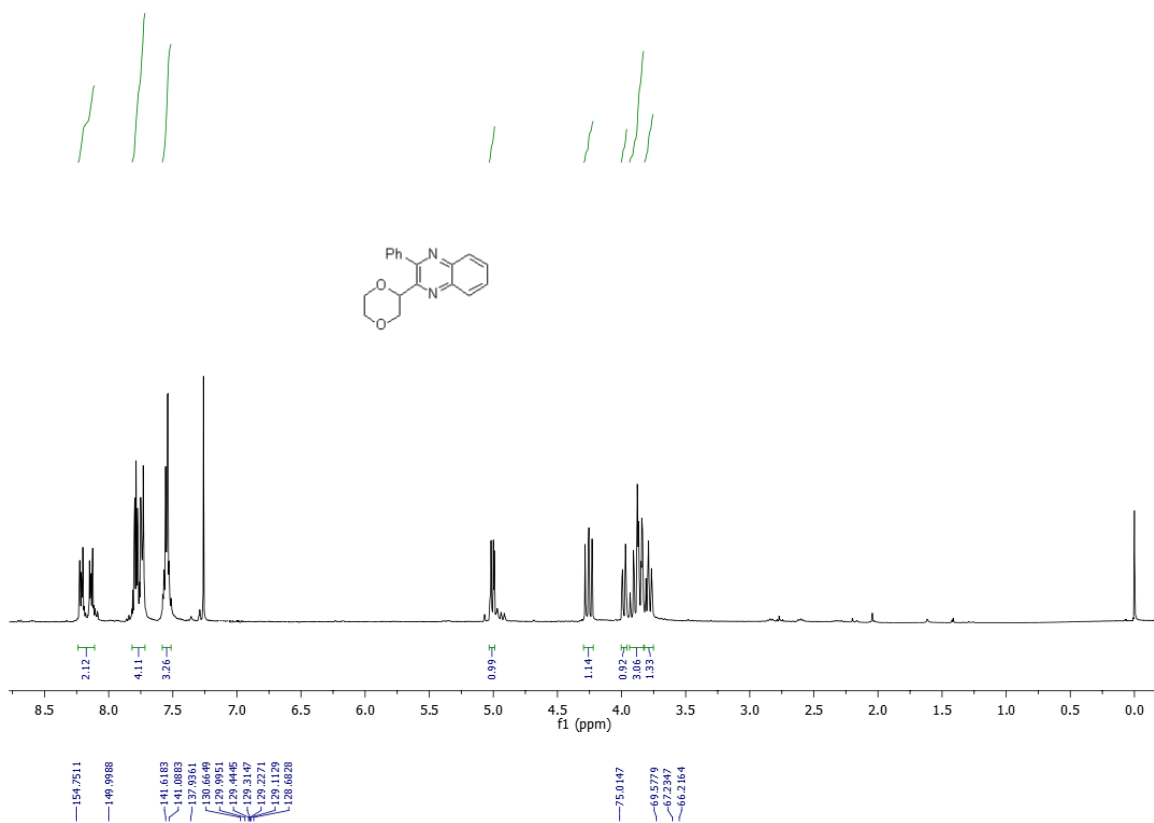
**9**

procedure described above using K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (236 mg, 0.876 mmol), N-(4-nitrobenzyl)aniline (100 mg, 0.438 mmol) in acetonitrile (2 ml) and H<sub>2</sub>O (2 ml). The mixture was irradiated with a house hold CFL bulb (27 W) at room temperature for 2 h and the product formation was monitored by TLC, purified by column chromatography as yellow solid product (56 mg, 85% ). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 10.17 (s, 1H), 8.44 – 8.38 (d, *J* = 8.2 Hz, 2H), 8.12 – 8.05 (d, *J* = 8.2 Hz, 2H).

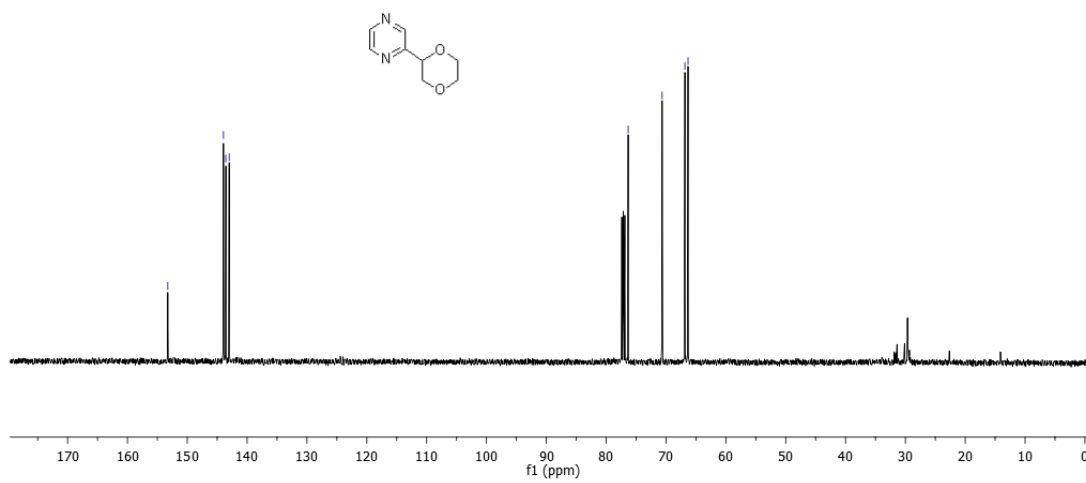
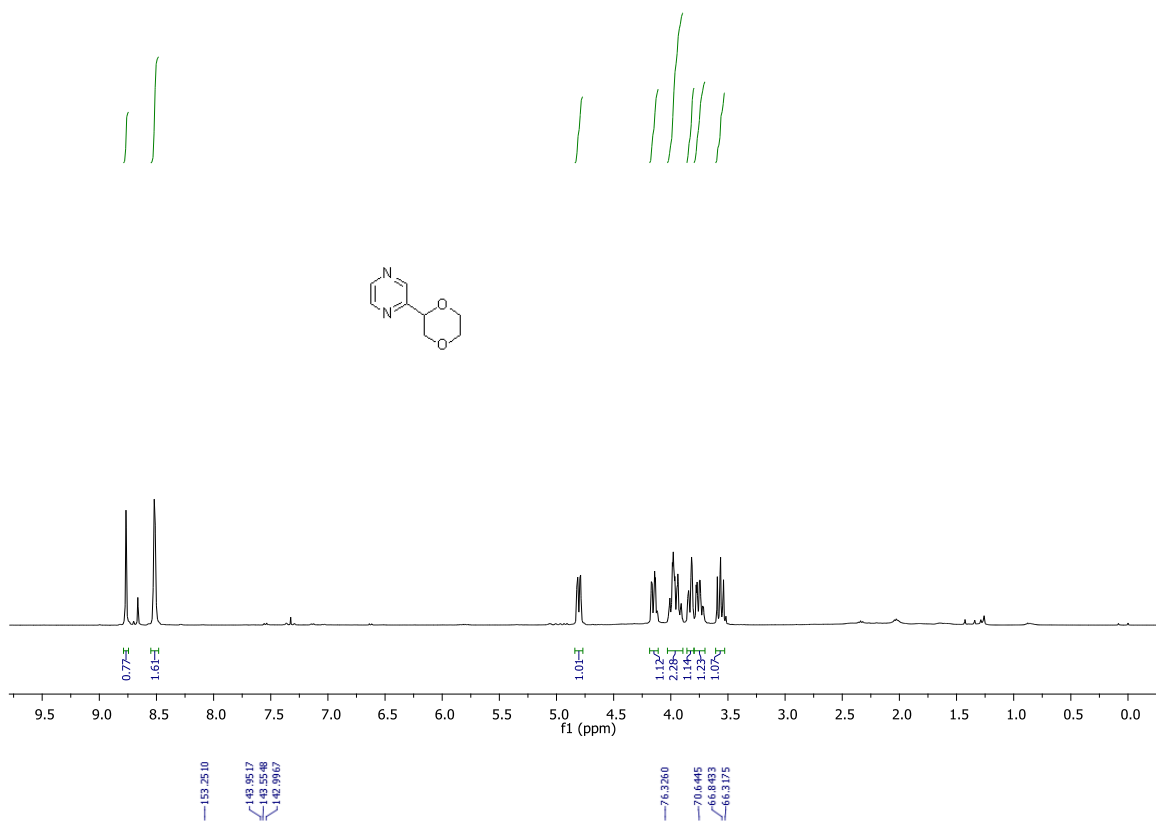
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3a



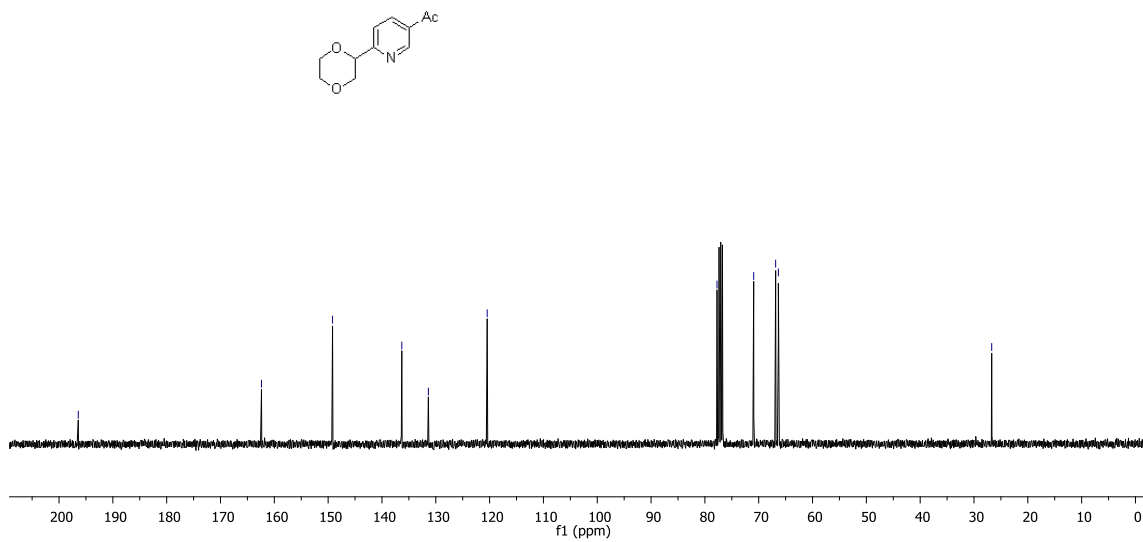
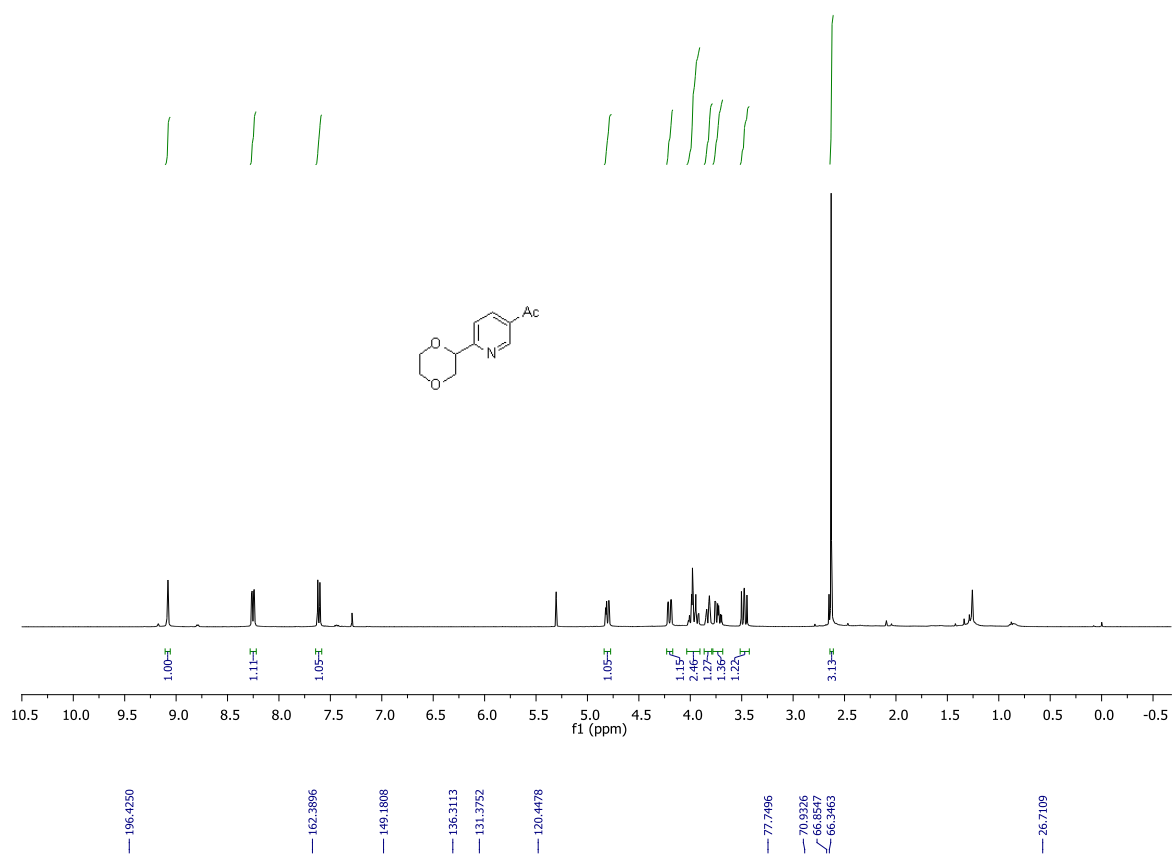
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3b



# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3c

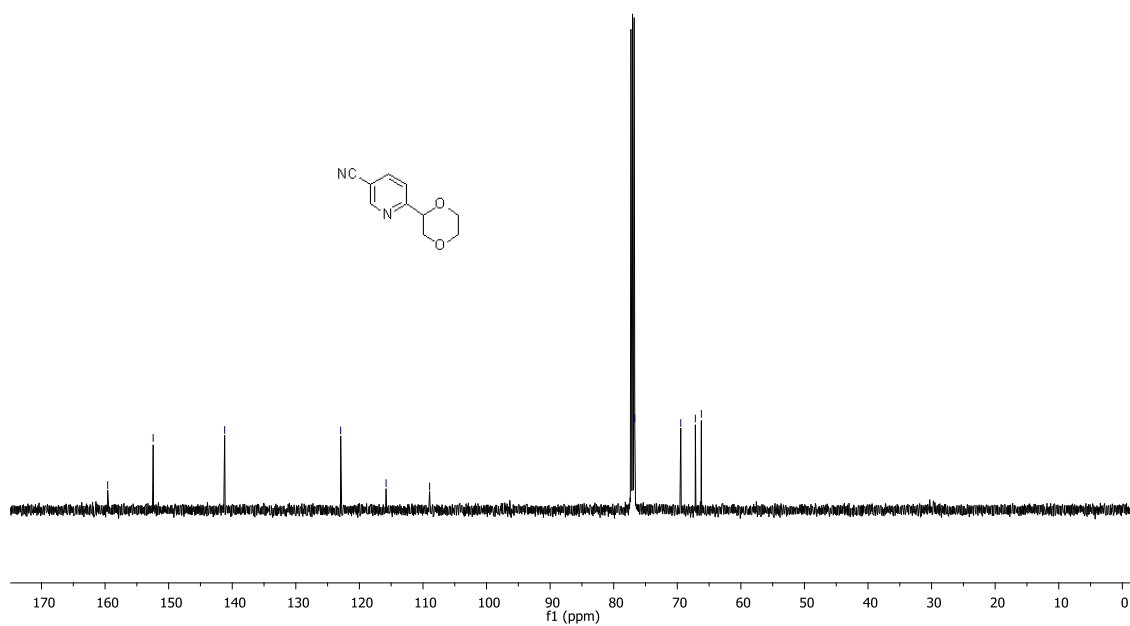
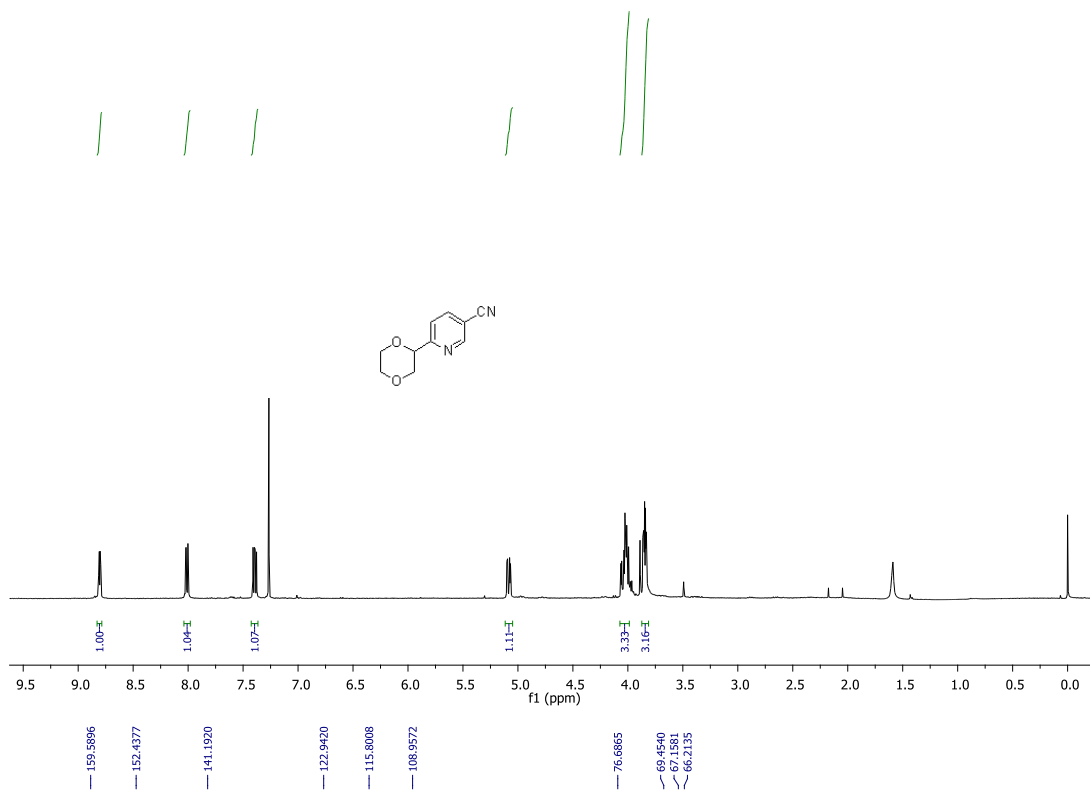


# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3d

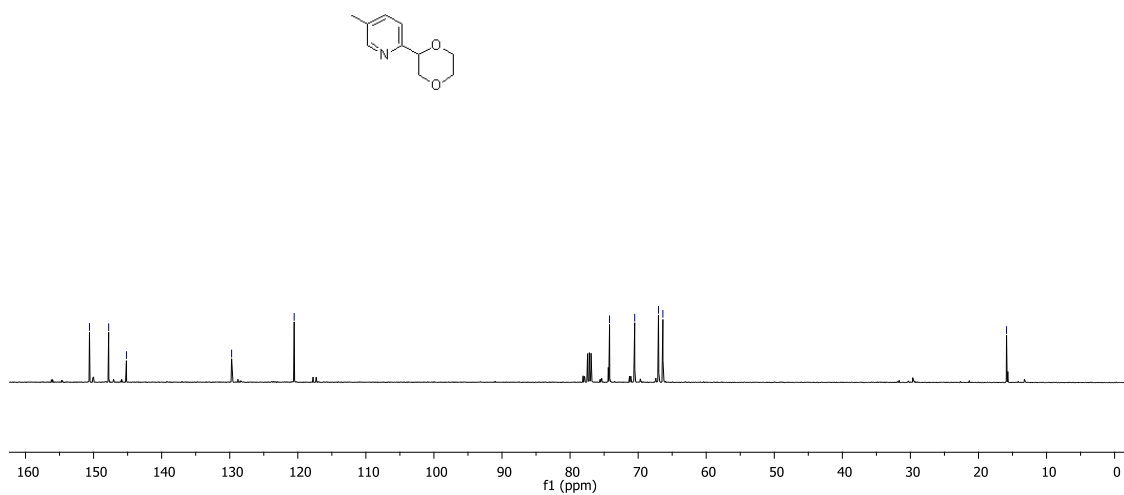
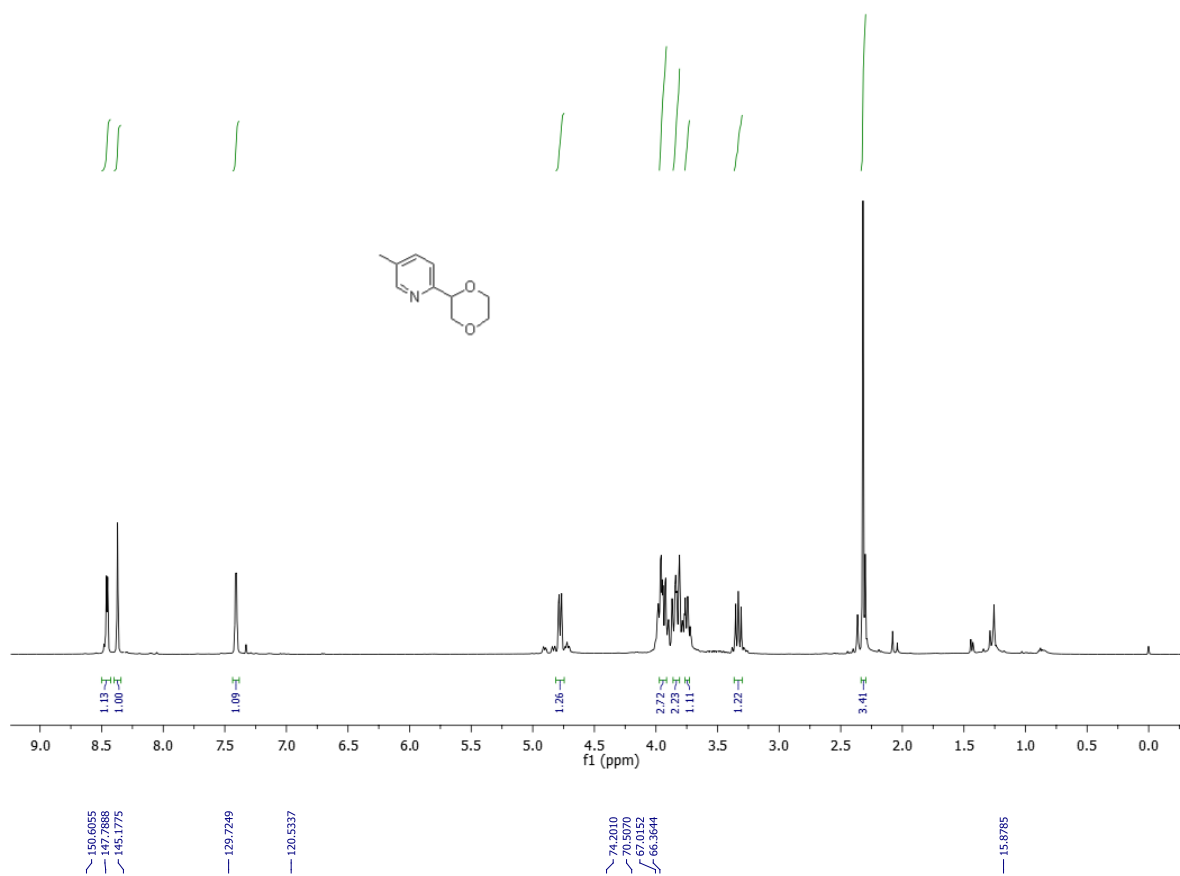




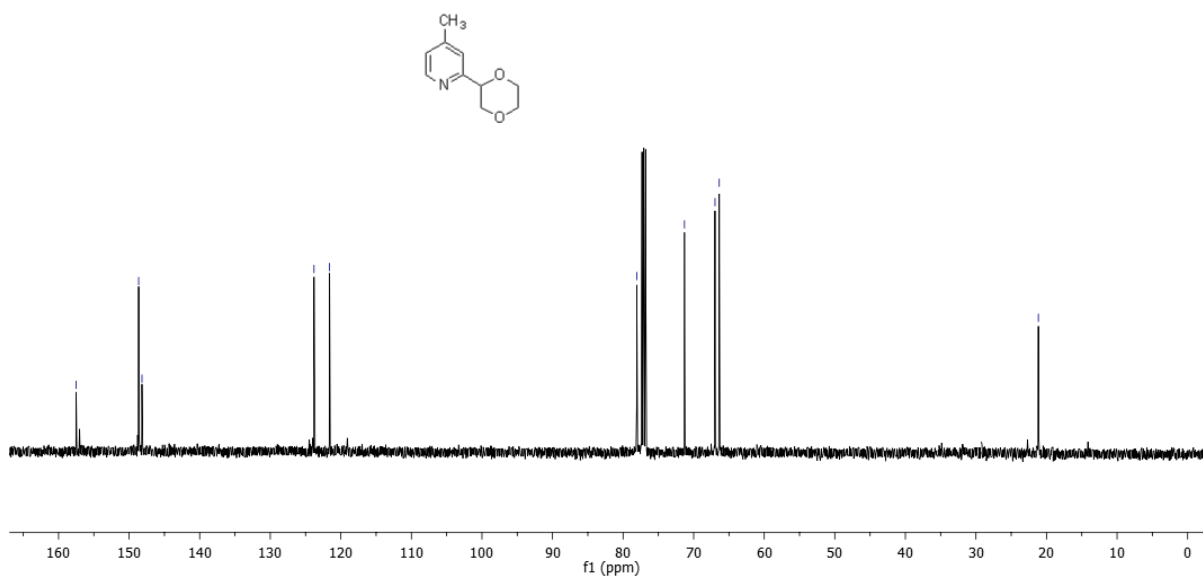
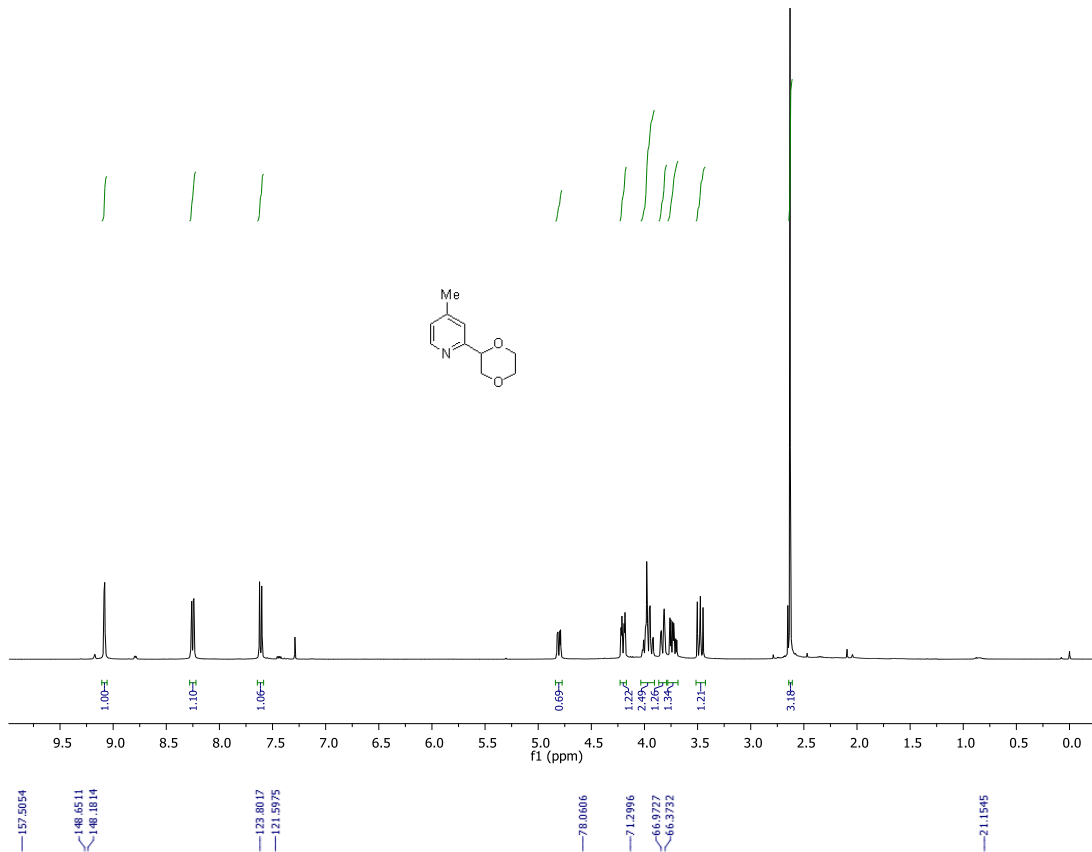
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3e



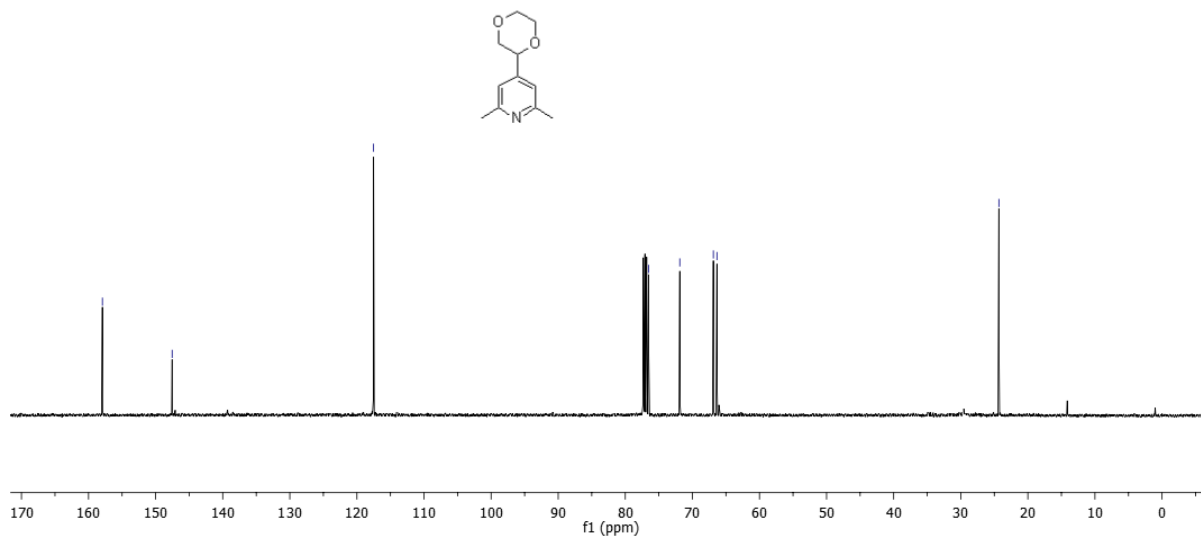
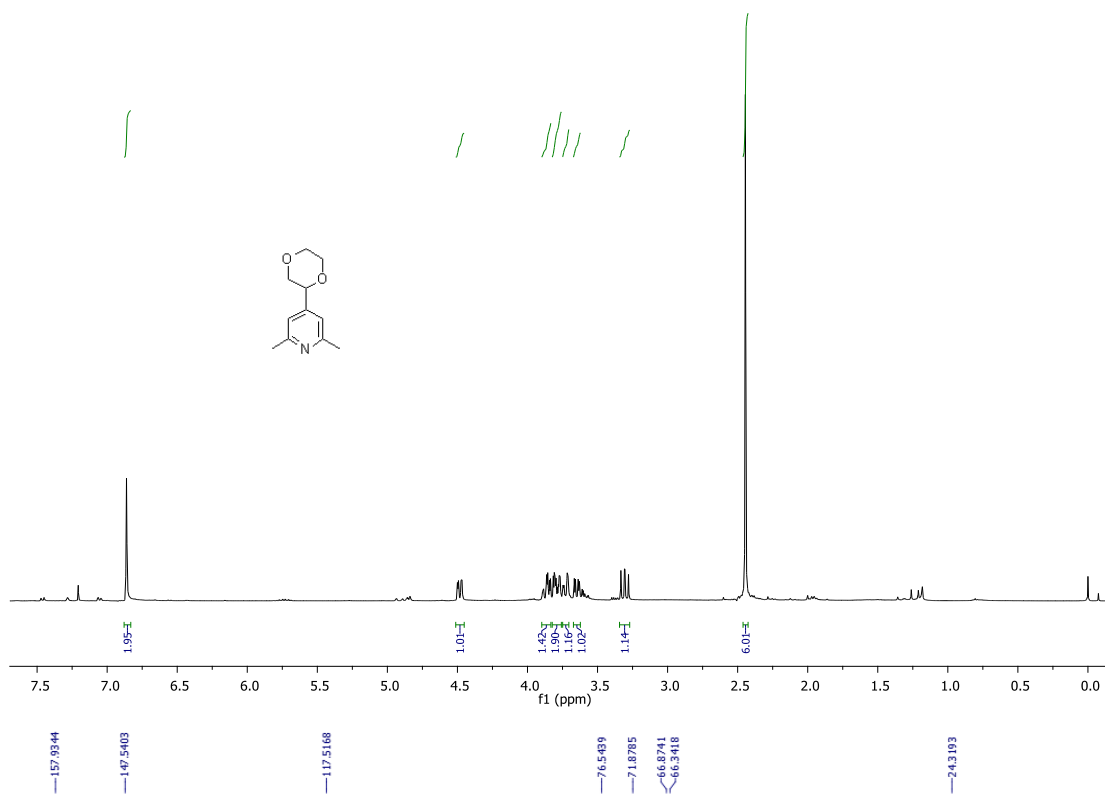
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3f



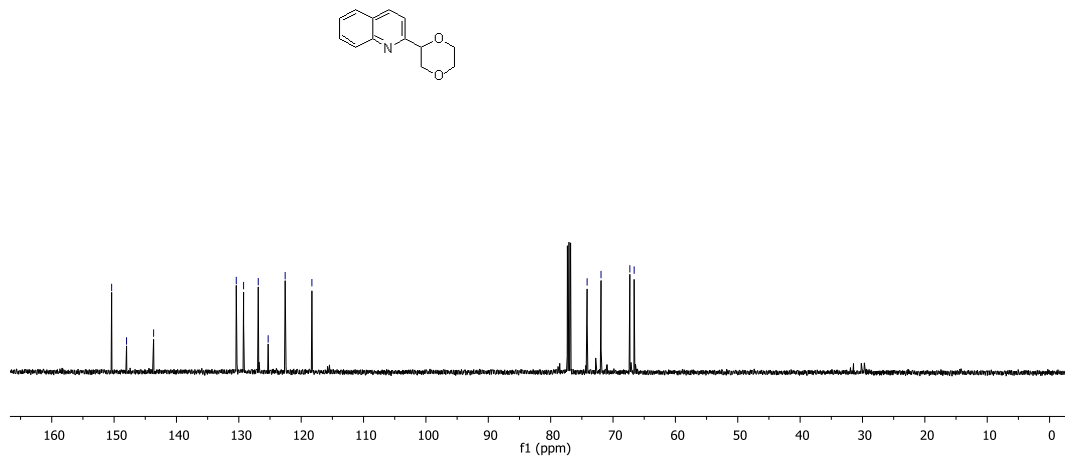
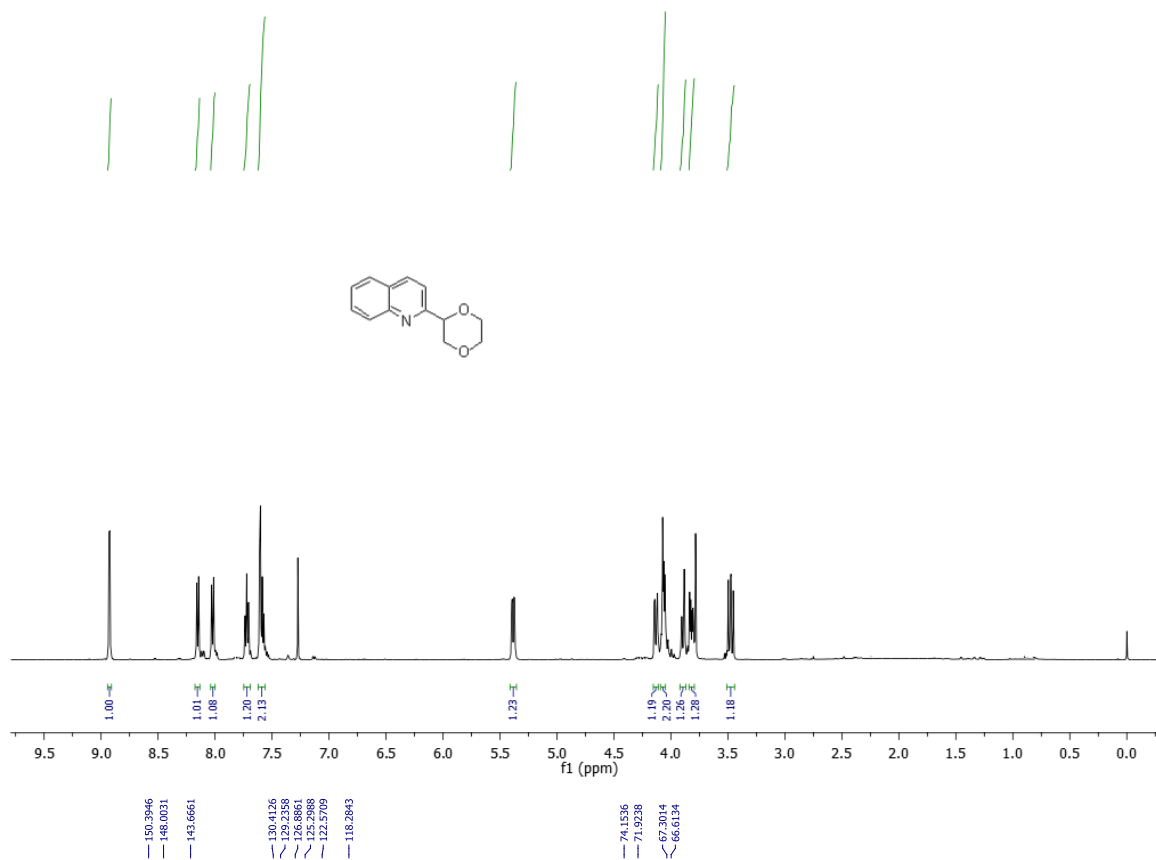
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3g



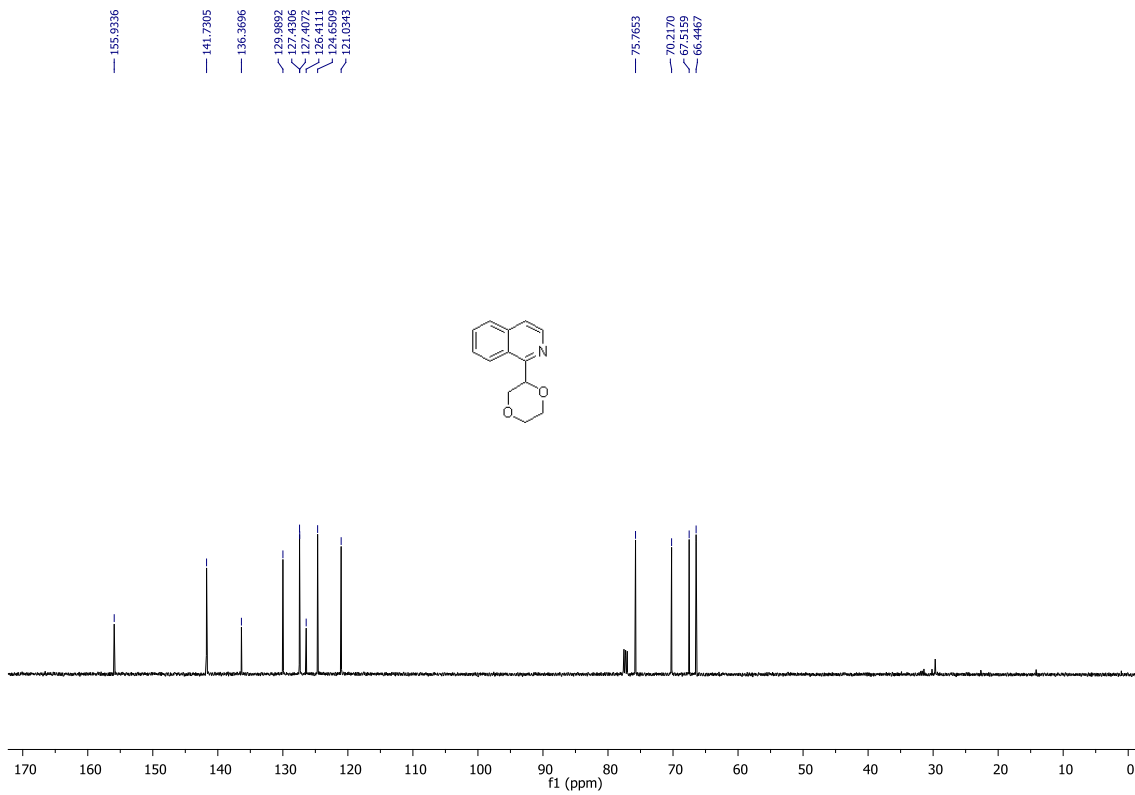
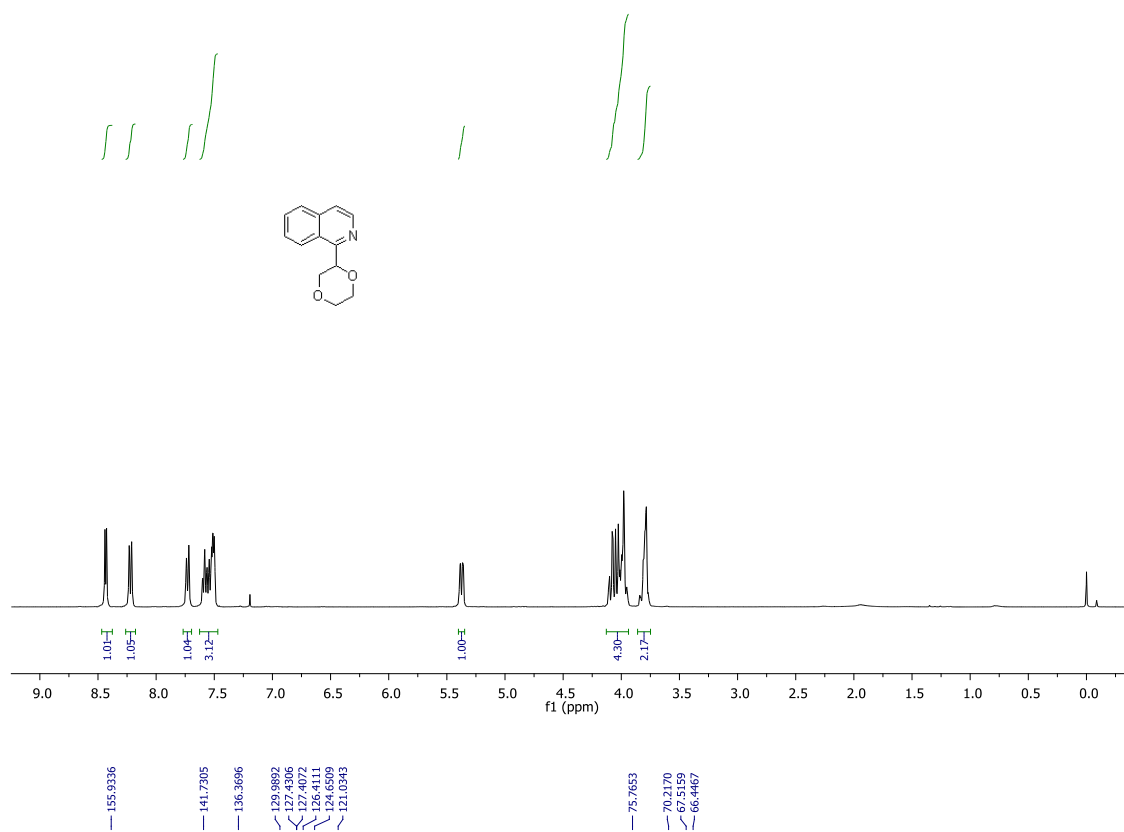
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3h



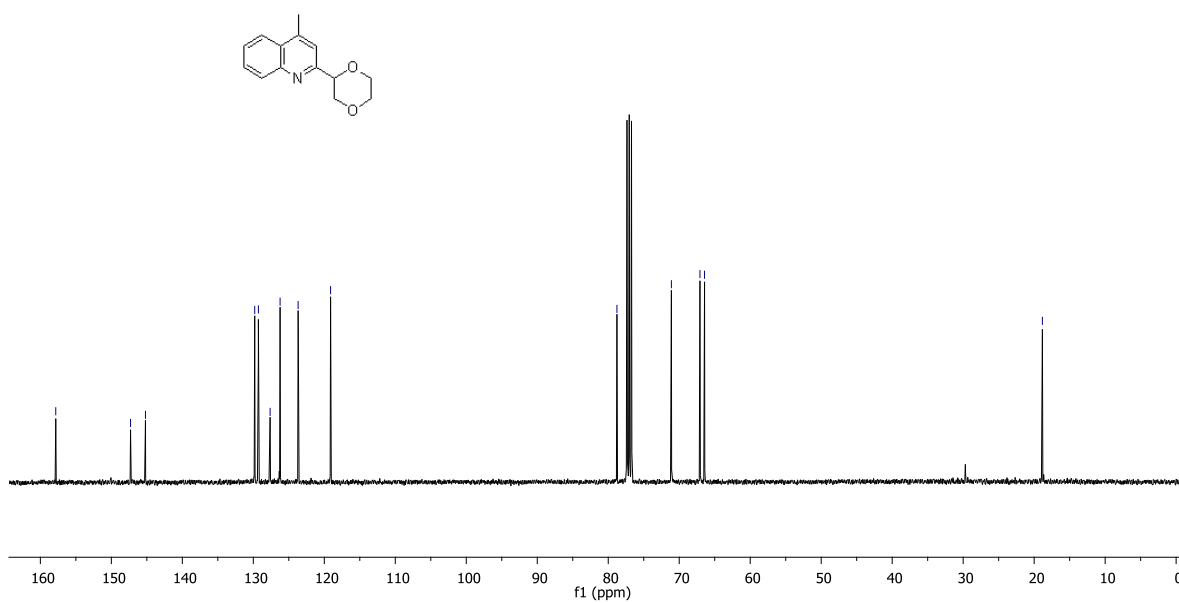
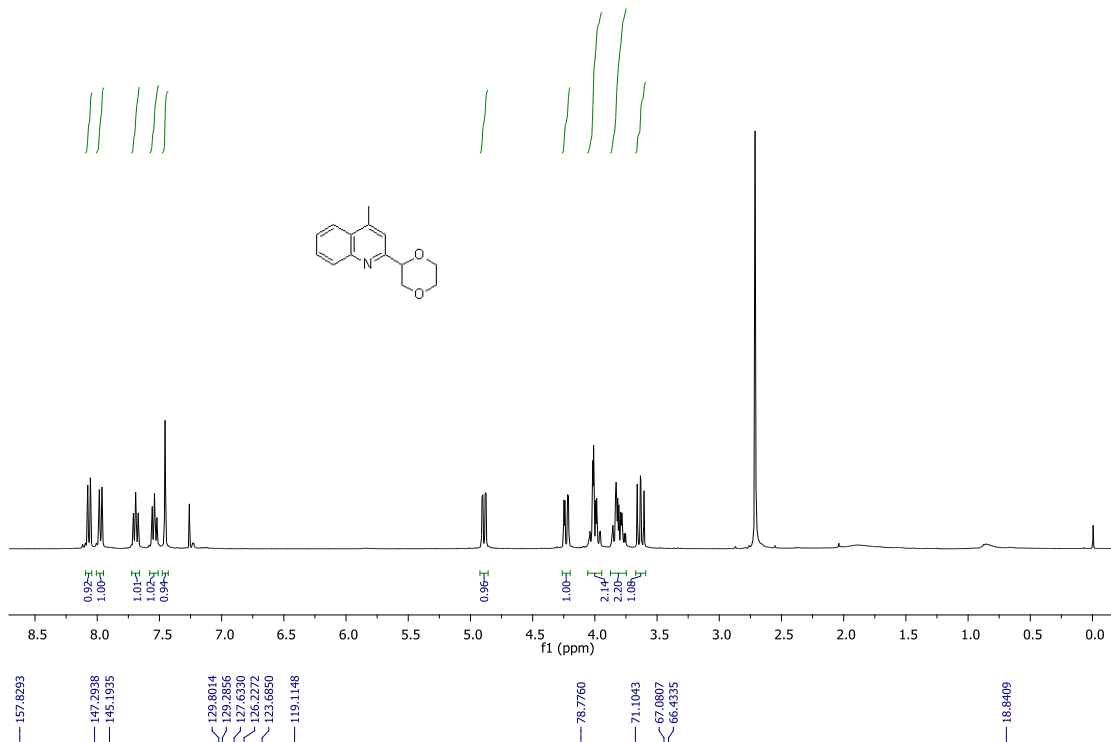
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3i



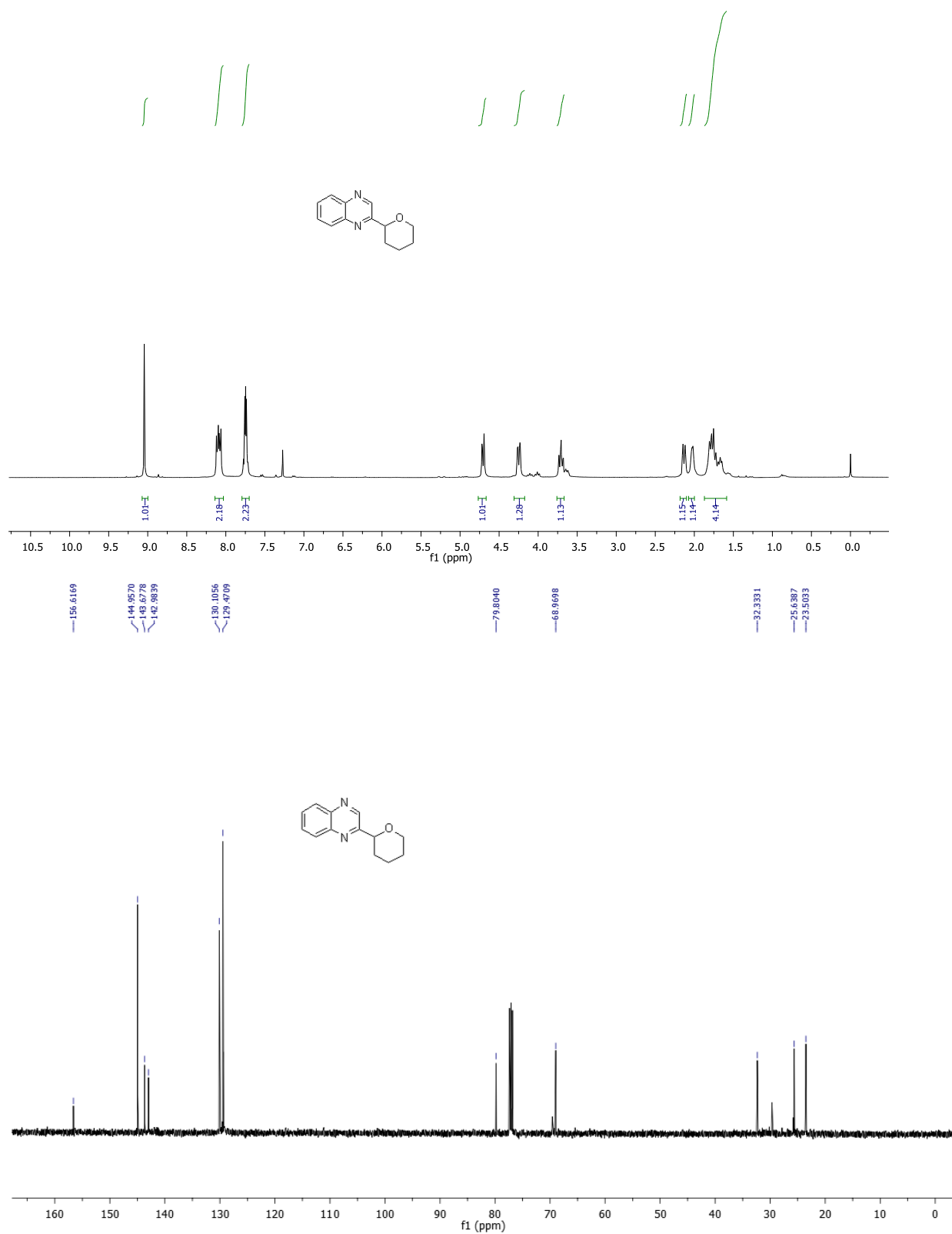
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3j



# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3k

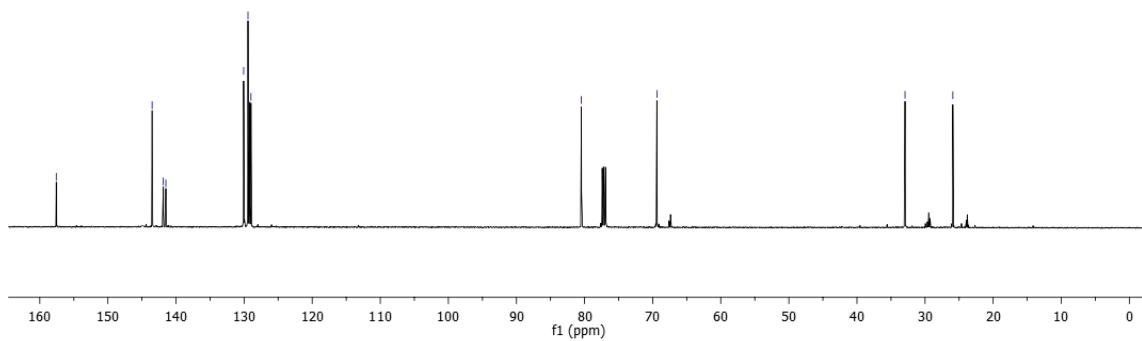
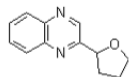
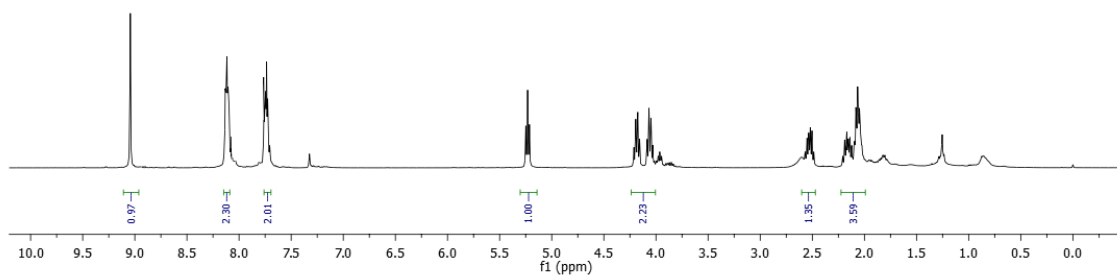
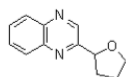
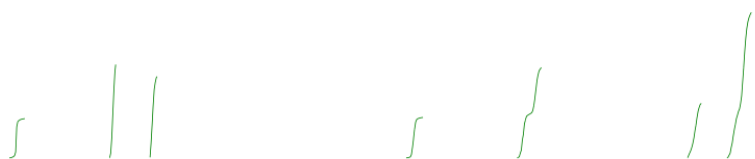


# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3l

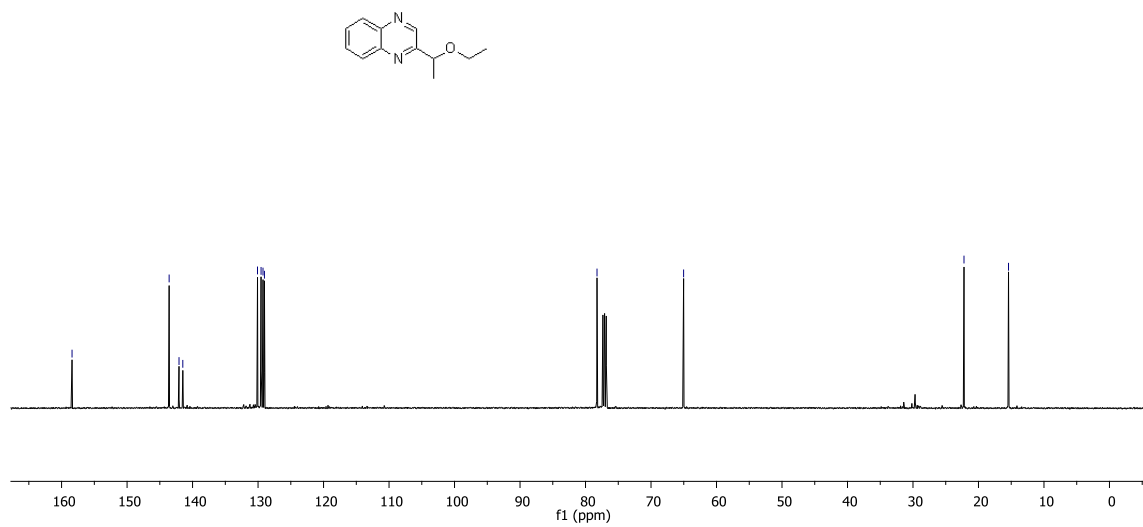
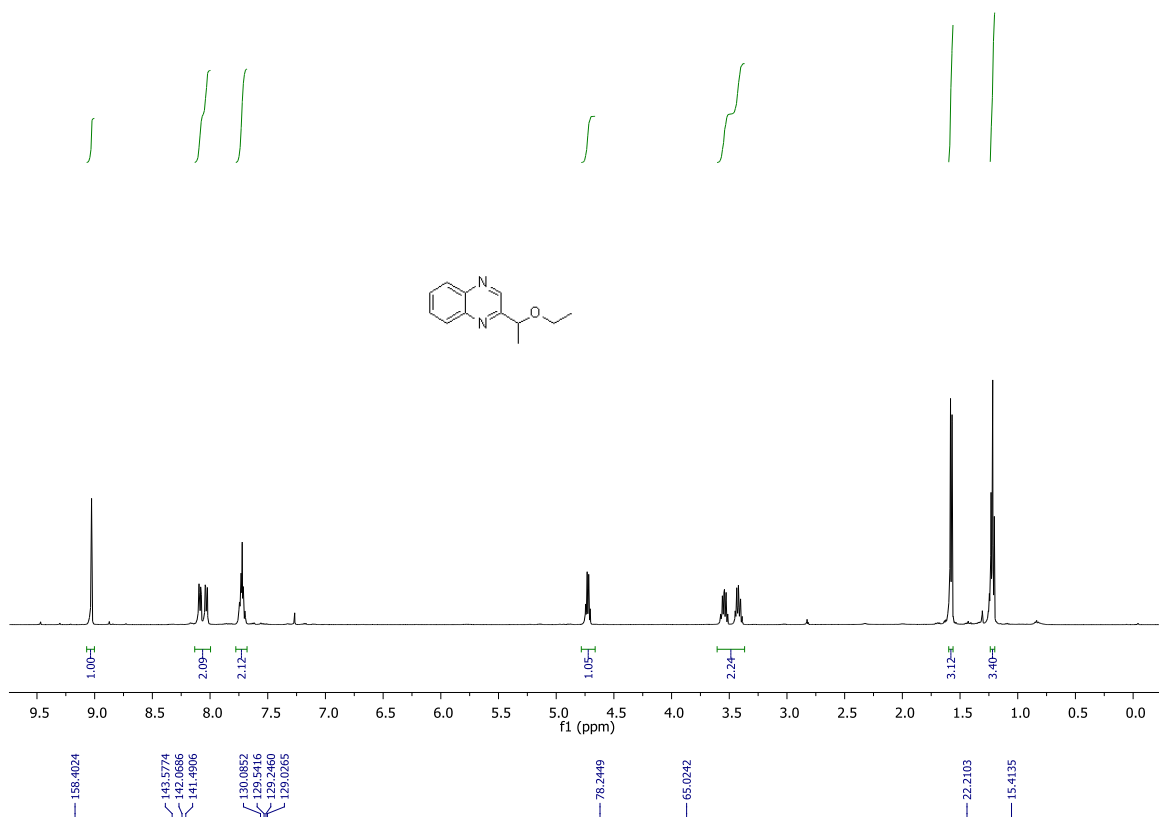




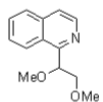
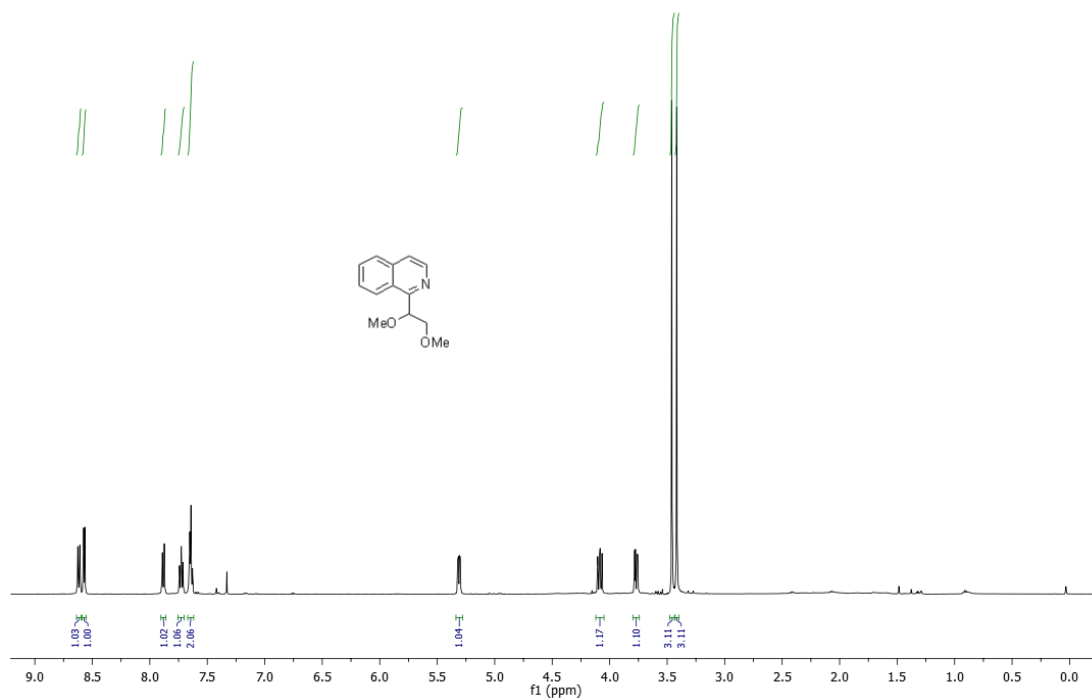
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3m



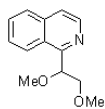
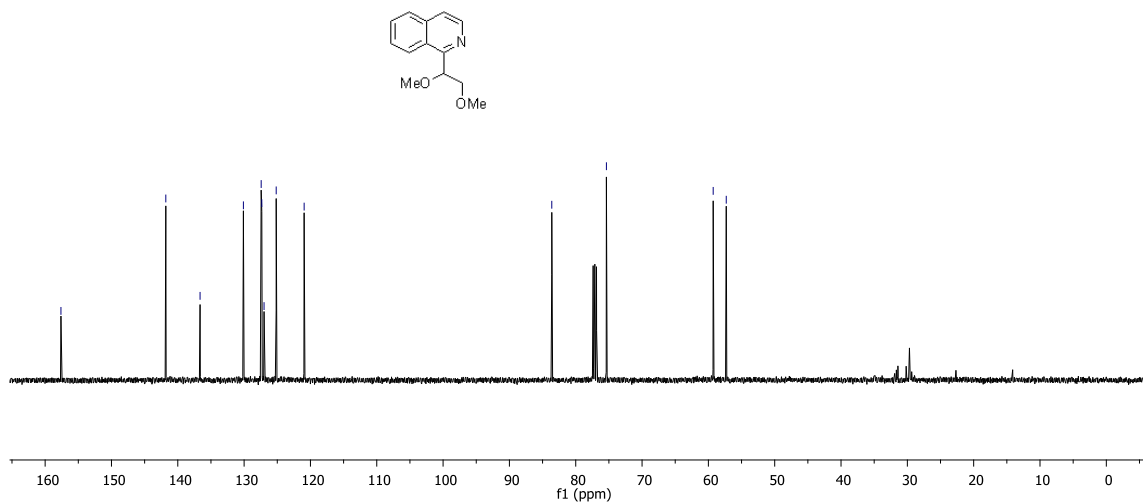
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3n



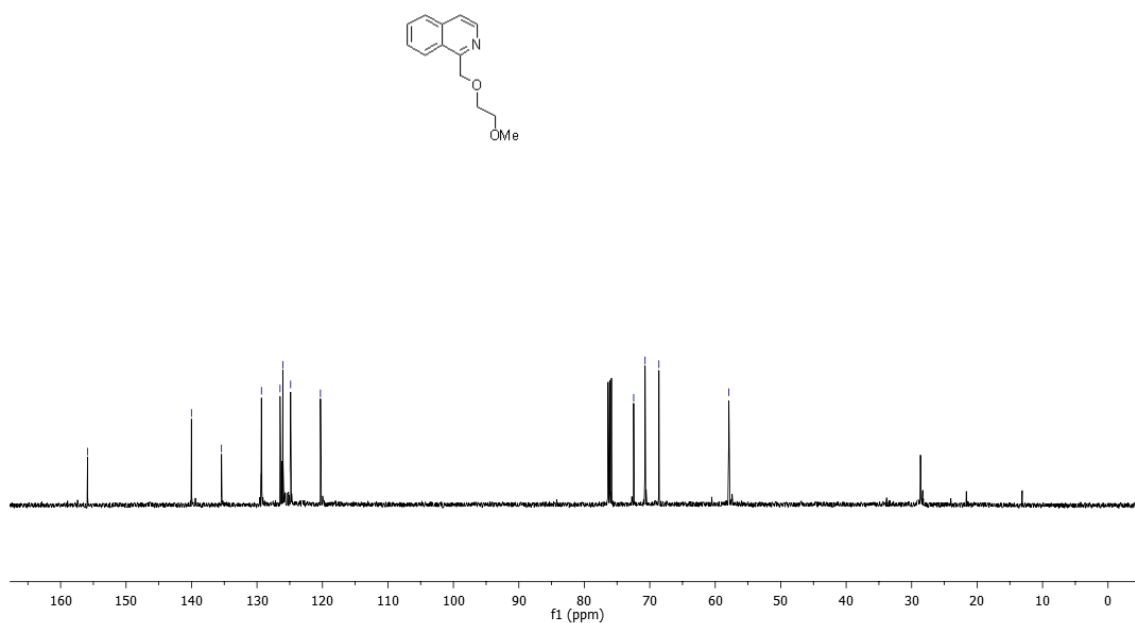
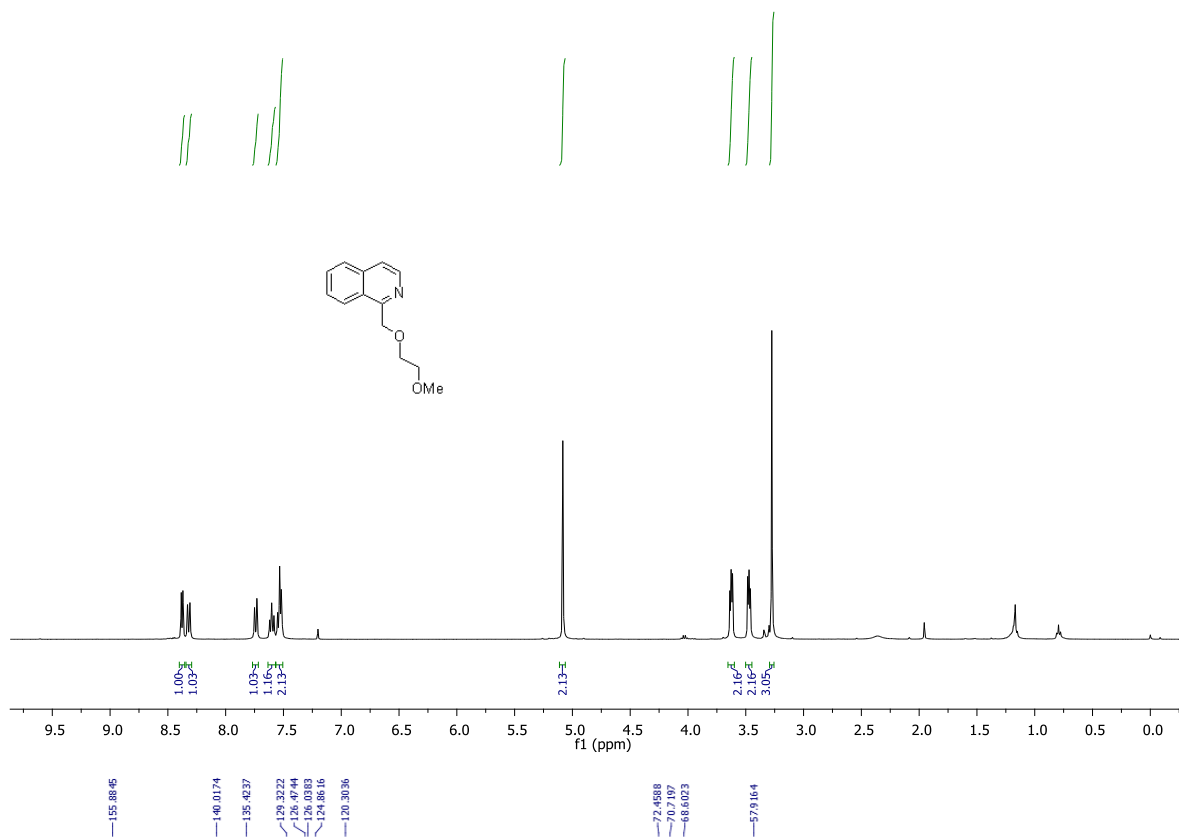
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 30a



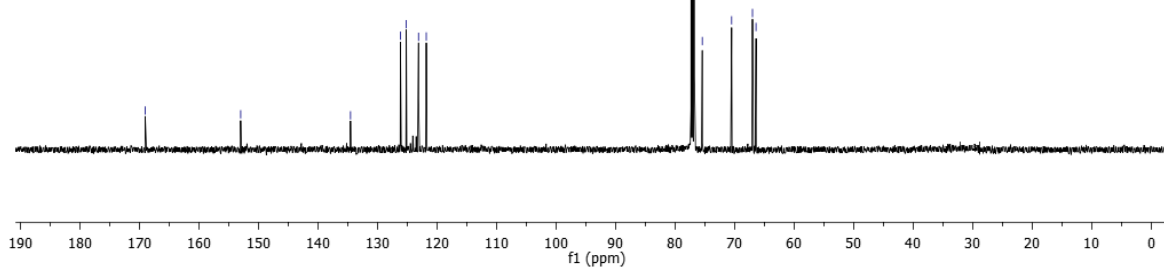
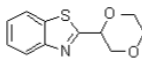
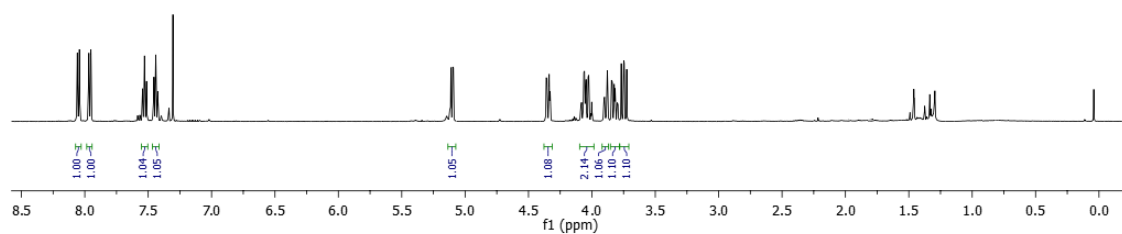
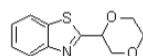
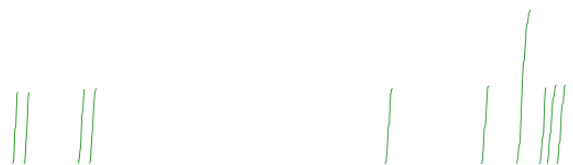
157.6176  
141.8067  
136.6411  
130.1067  
127.4280  
127.3924  
126.9931  
125.1610  
120.9489  
83.6112  
76.3745  
59.2753  
57.3059



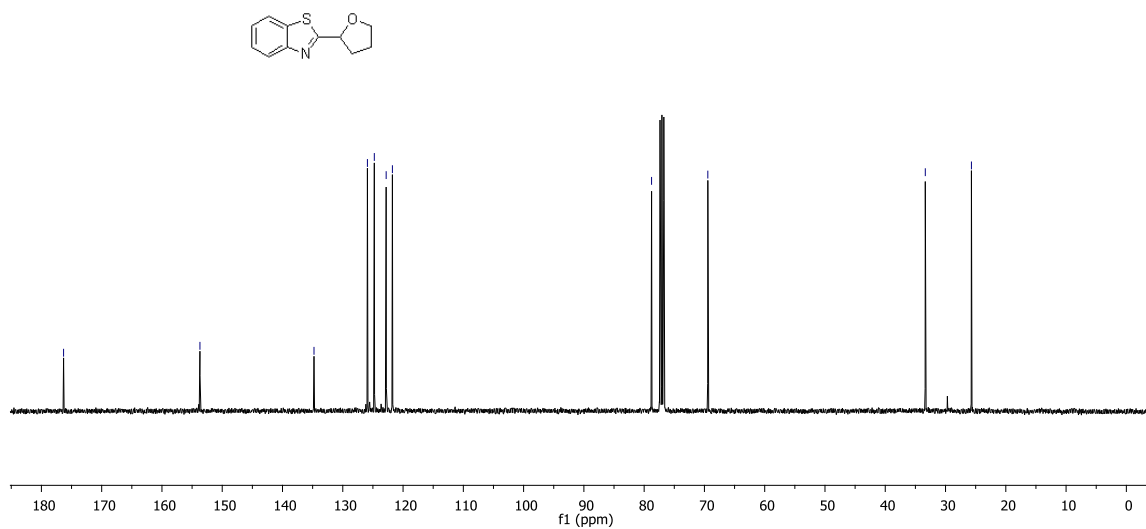
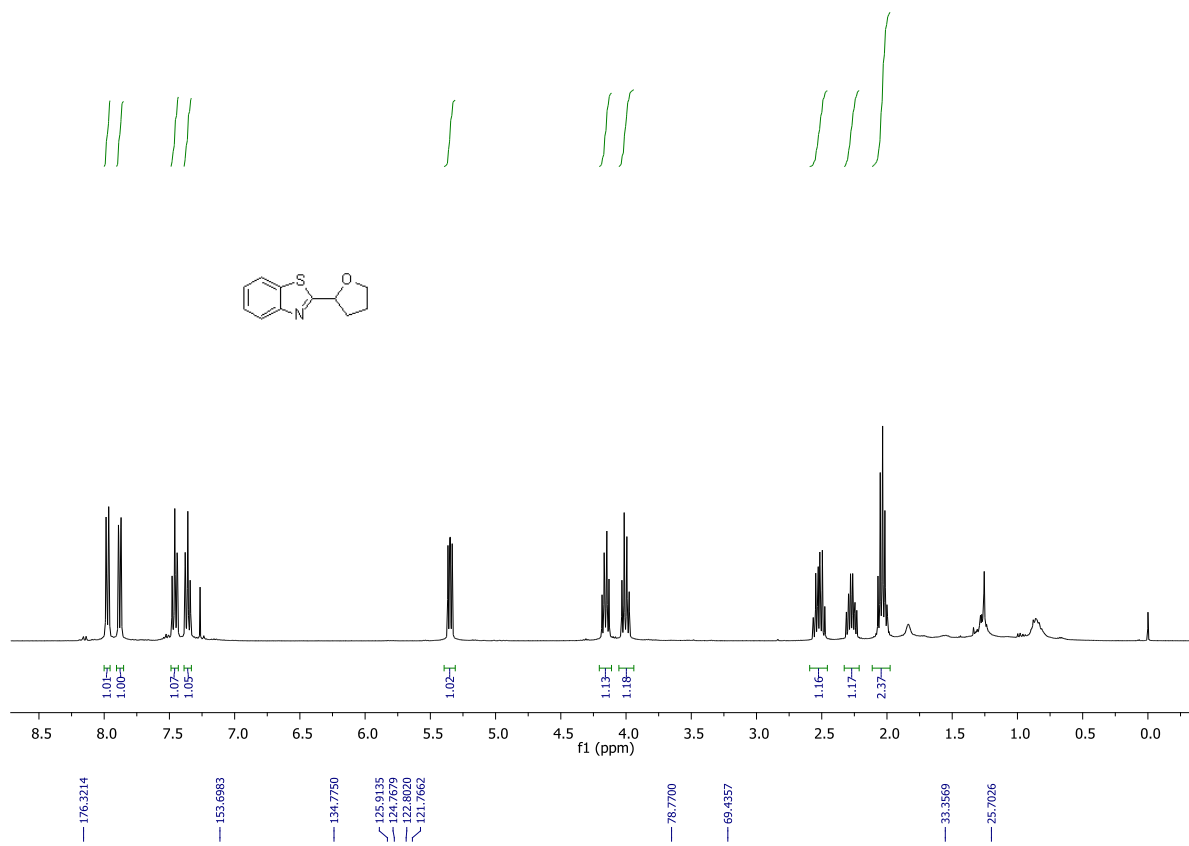
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 3ob



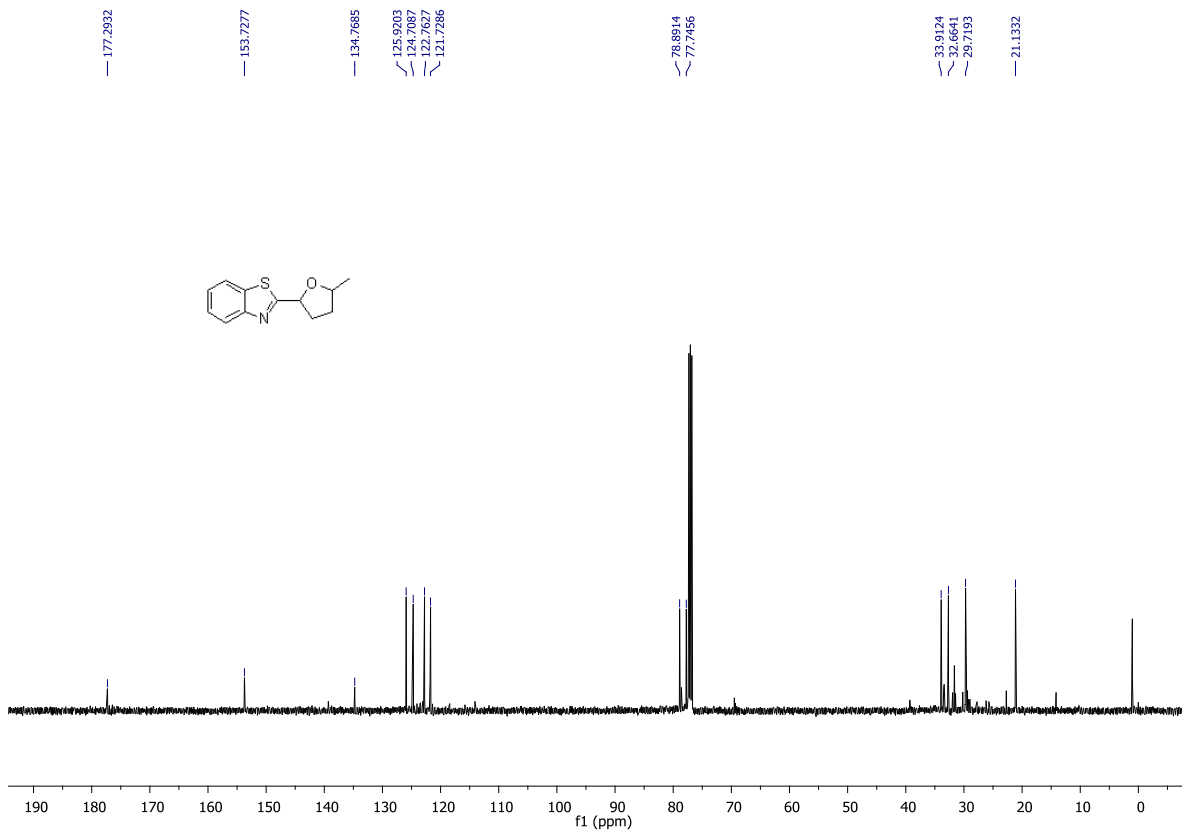
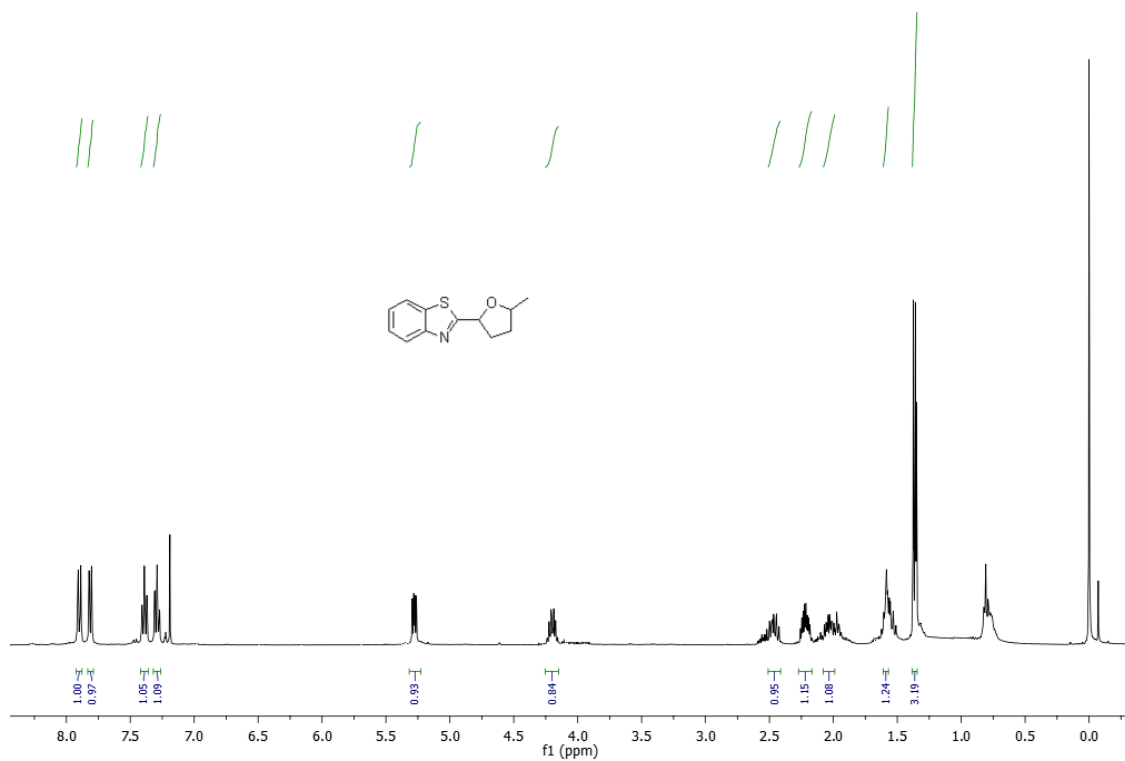
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 5a



# $^1\text{H}$ and $^{13}\text{C}$ NMR of 5b

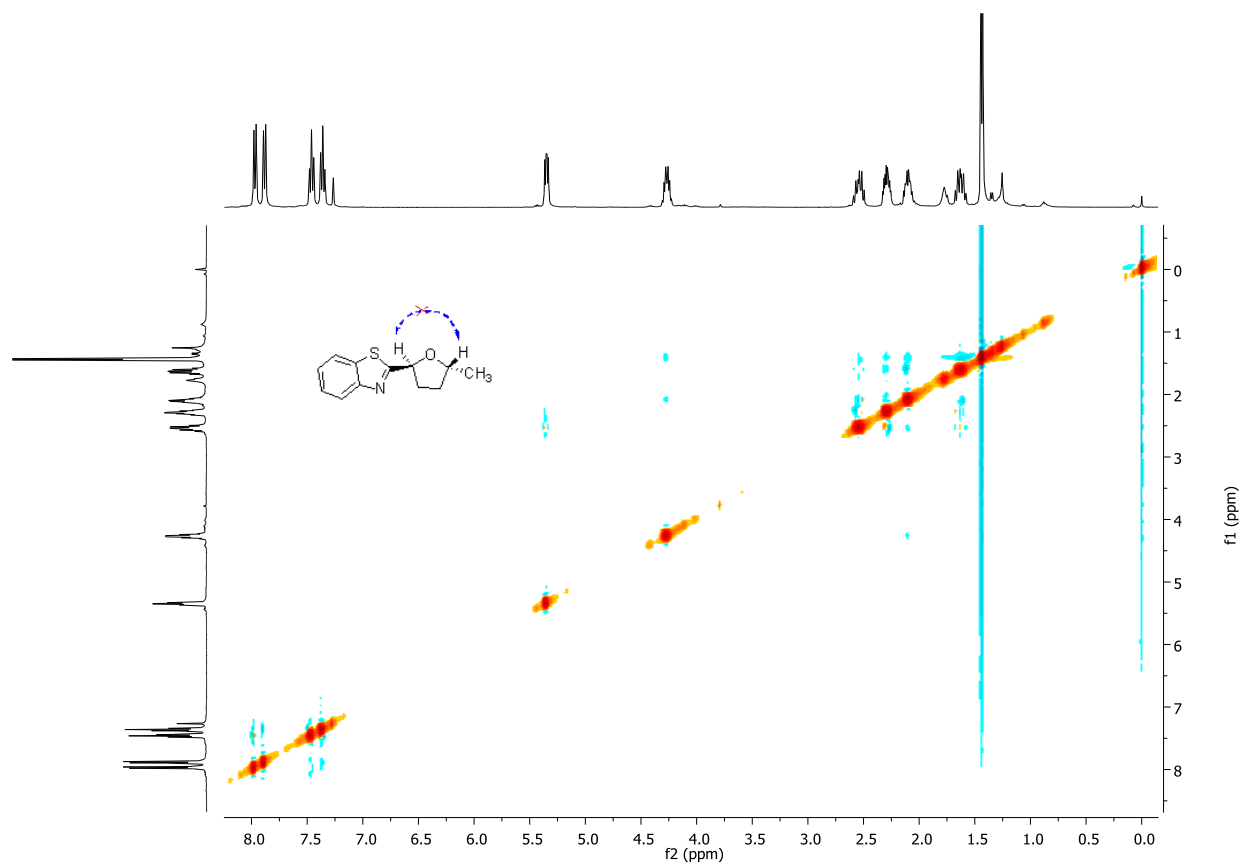


# $^1\text{H}$ NMR $^{13}\text{C}$ NMR of 5ca



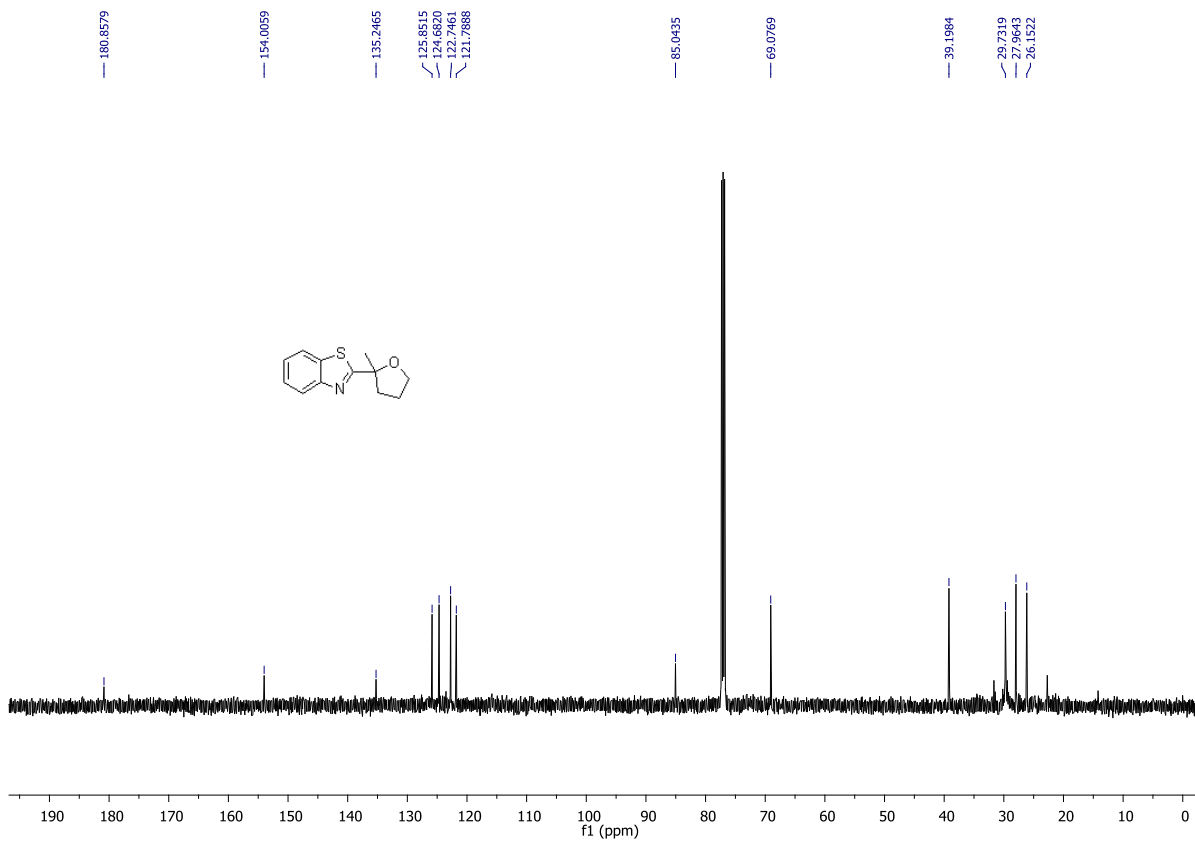
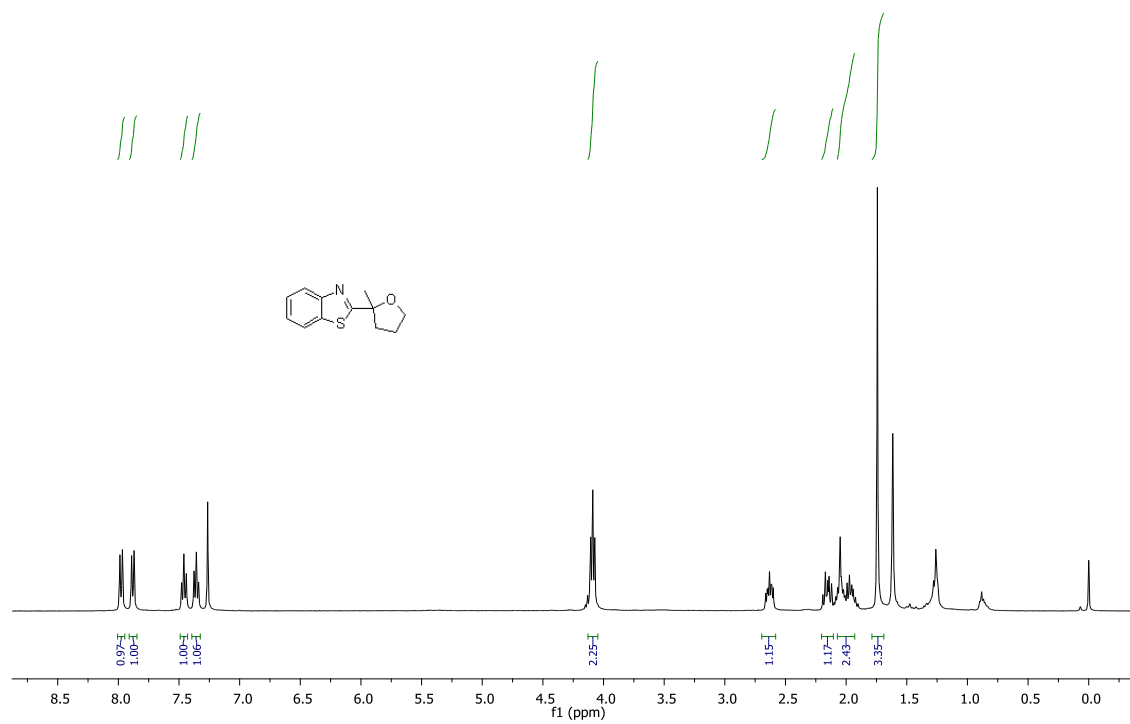
**NOESY NMR of 5ca;**

**No H-H correlation were observed between H<sub>2</sub>-H<sub>5</sub>**

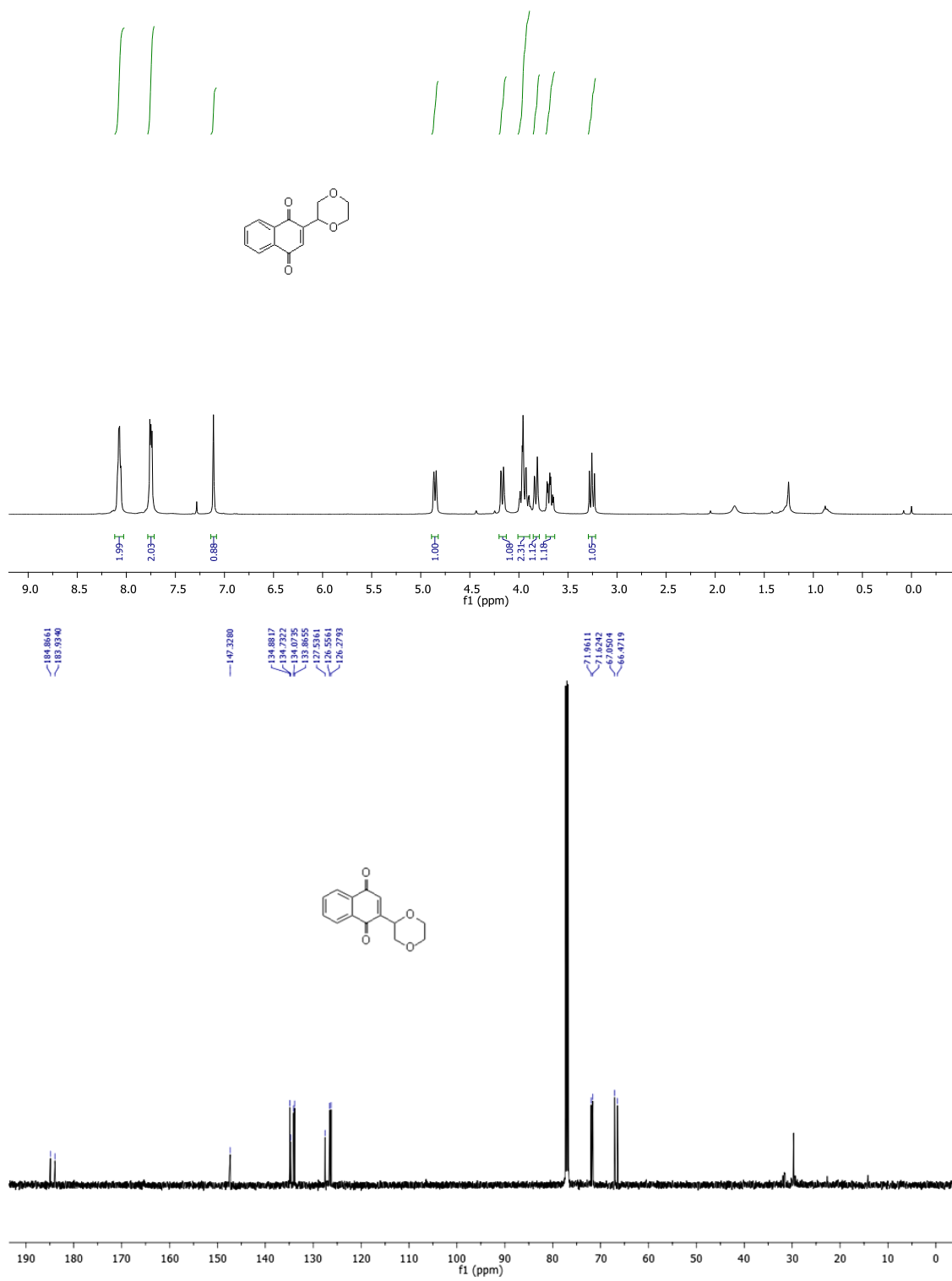




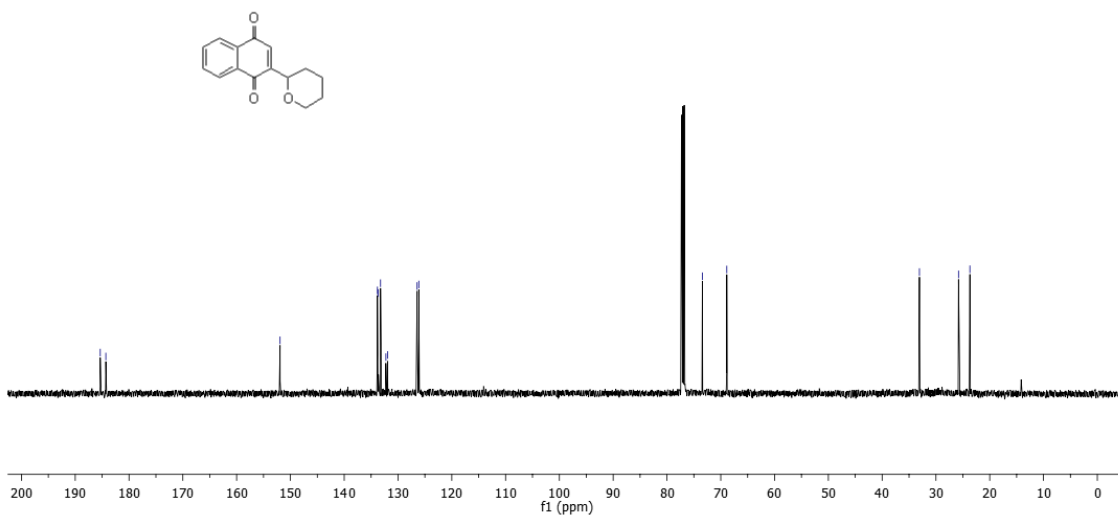
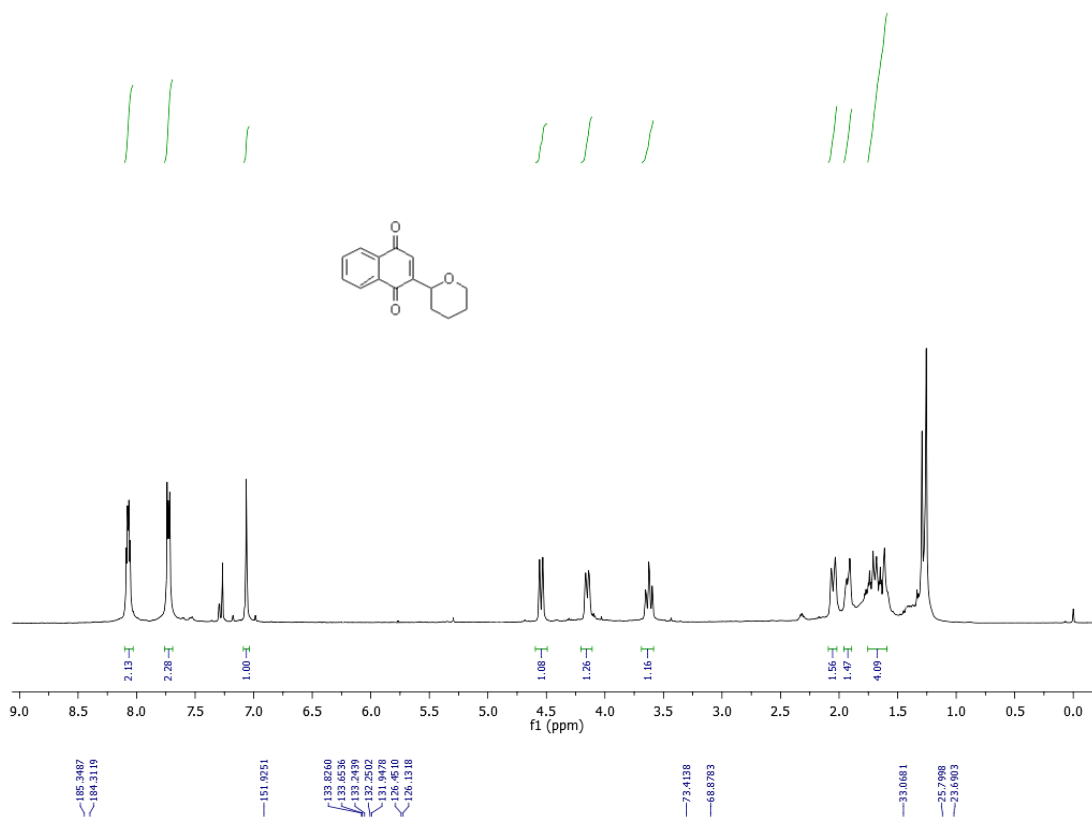
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 5cb



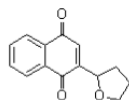
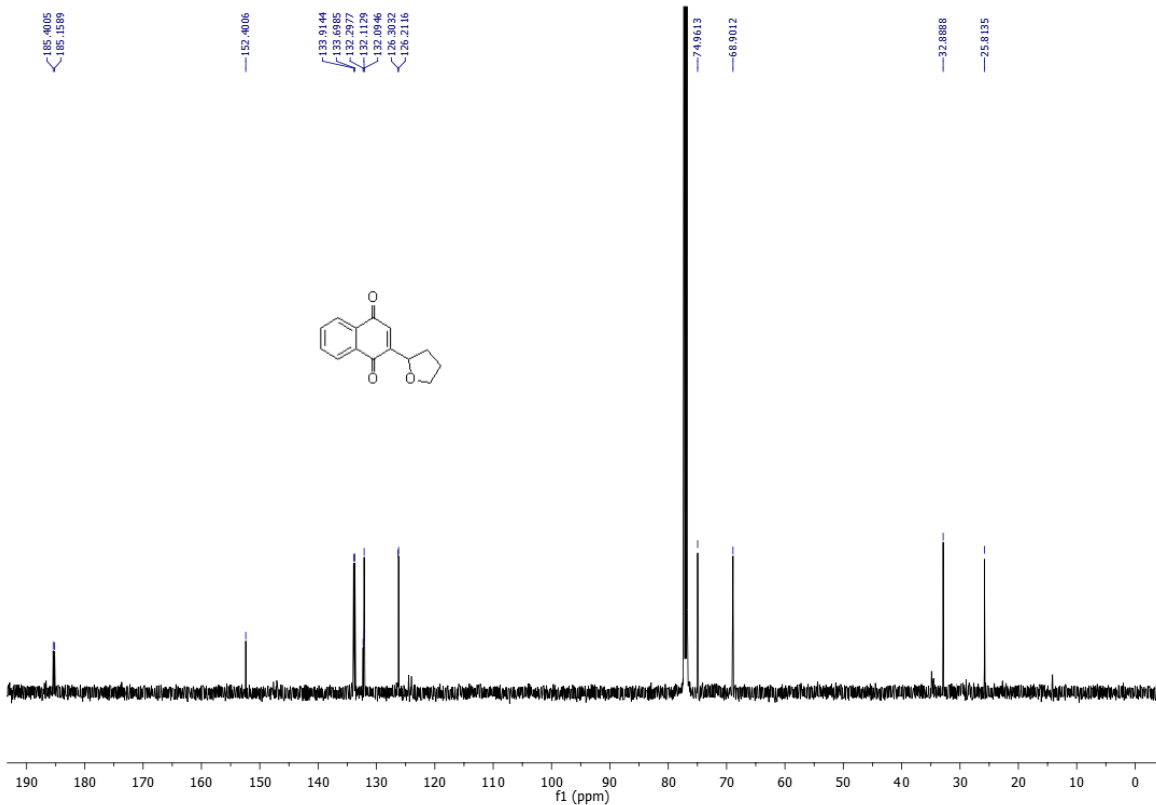
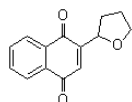
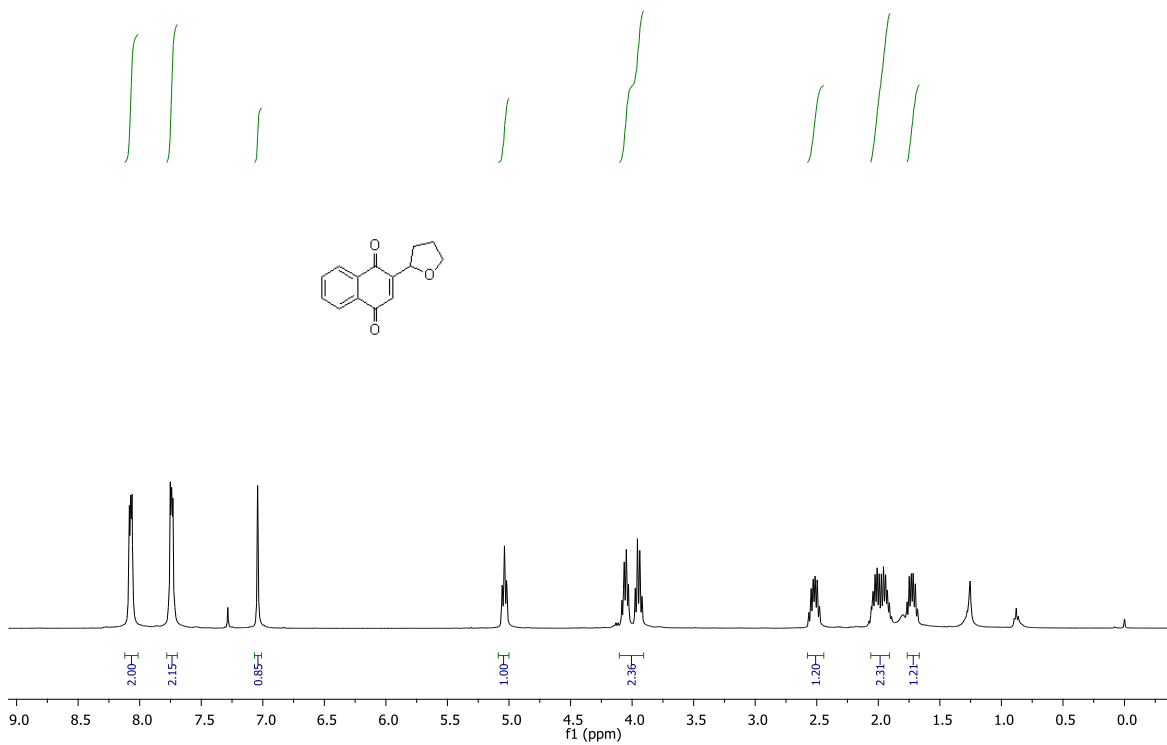
# $^1\text{H}$ and $^{13}\text{C}$ NMR of 7a



# $^1\text{H}$ and $^{13}\text{C}$ NMR of 7b



# $^1\text{H}$ NMR $^{13}\text{C}$ NMR of 7c



<sup>1</sup>H NMR of 9

