

Supporting Information

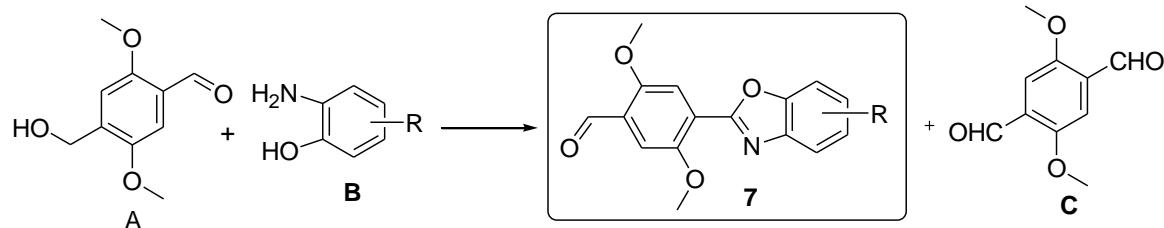
For

A Versatile Synthesis of Bis[2-(2'-hydroxylphenyl)benzoxazole] Derivatives as Zinc sensors

Junfeng Wang and Yi Pang*

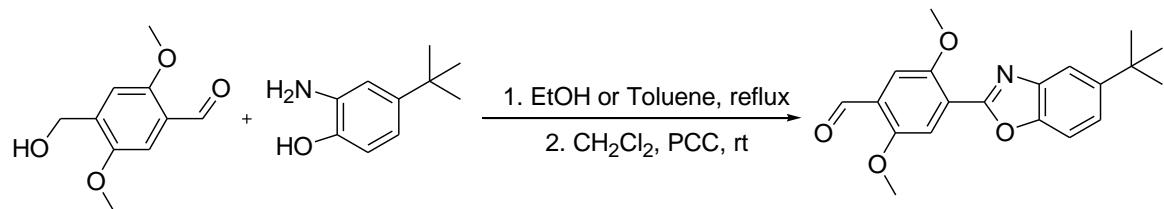
Department of Chemistry, Department of Chemistry & Maurice Morton Institute of Polymer Science
The University of Akron, Akron, Ohio 44325 U.S.A.

General procedure A



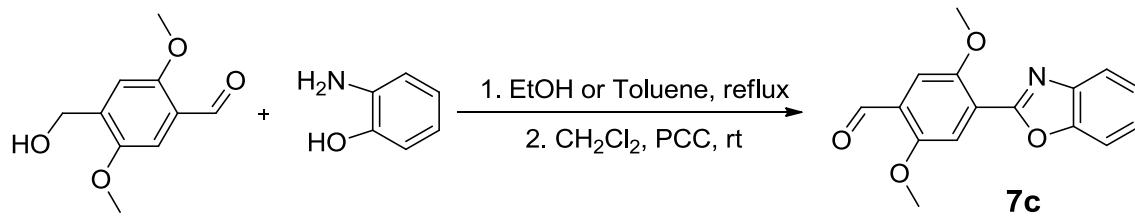
Intermediates **A** (600 mg, 3.06 mmol) and **B** (3.2 mmol) were dissolved in EtOH (50 mL), and the mixture was refluxed overnight. The crude product mixture was concentrated under vacuum and dried in a vacuum oven overnight. The resulting mixture was then redissolved in 80 mL anhydrous DCM (Solution I). PCC (1.5 g) and silica gel (4.5 g) were mixed in 80 mL DCM and stirred at room temperature for one hour (Solution II). The PCC solution II was added to the solution I and the mixture was stirred overnight and then filtered through a short pad of silica and washed by EtOAc. The organic phase was collected, concentrated in vacuum, and purified on a silica gel column to give **7** in 50-65% yield as yellow solid.

Synthesis of 7b



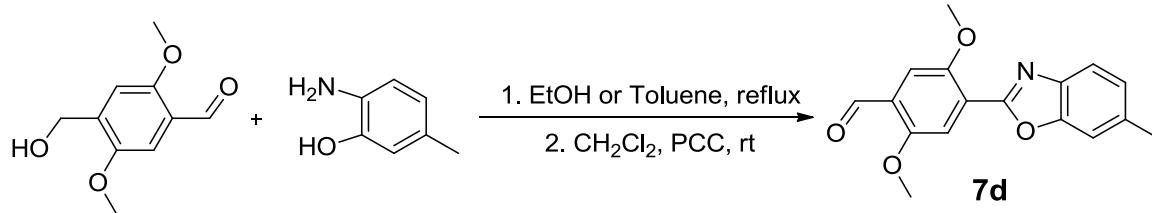
Compound **7b** was made according to the general procedure A. ^1H NMR (300 MHz, CDCl_3): 10.50 (1H, s), 7.87 (1H, d, J = 1.2 Hz), 7.80 (1H, s), 7.53 (1H, d, J = 8.4 Hz), 7.52 (1H, s), 7.45 (1H, dd, J = 1.2 Hz, J = 8.4 Hz), 4.01 (6H, s), 1.39 (9H, s). ^{13}C NMR (75 MHz, CDCl_3): 189.2, 160.8, 155.9, 152.7, 148.9, 148.5, 142.0, 126.9, 123.8, 122.8, 117.2, 114.7, 111.3, 110.1, 57.0, 56.6, 35.2, 31.9.

Synthesis of **7c**



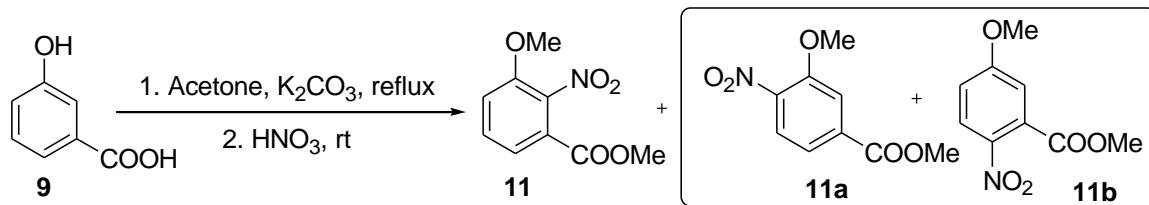
Compound **7c** was made according to the general procedure A. ^1H NMR (300 MHz, CDCl_3): 10.52 (1H, s), 7.87-7.83 (1H, m), 7.83 (1H, s), 7.65-7.62 (1H, m), 7.54 (1H, s), 7.41-7.37 (2H, m), 4.03 (6H, s). ^{13}C NMR (75 MHz, CDCl_3): 188.9, 160.5, 155.6, 152.5, 150.6, 141.8, 126.9, 125.7, 124.7, 122.3, 120.5, 114.6, 111.0, 110.7, 56.8, 56.3.

Synthesis of **7d**



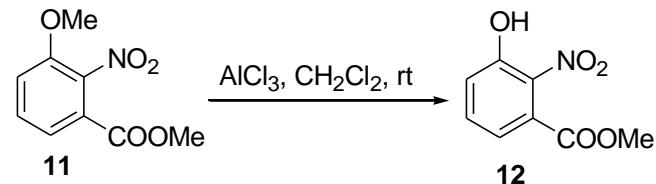
Compound **7d** was made according to the general procedure A. ^1H NMR (300 MHz, CDCl_3): 10.46 (1H, s), 7.74 (1H, s), 7.67 (1H, d, J = 8.4 Hz), 7.47 (1H, s), 7.38 (1H, s), 7.15 (1H, d, J = 8.4 Hz), 3.98 (6H, s), 2.48 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 189.0, 160.0, 155.7, 152.5, 151.0, 139.9, 136.4, 126.8, 126.2, 122.6, 119.9, 114.5, 111.1, 110.9, 56.9, 56.5, 22.0.

Synthesis of **11**



3-Hydroxybenzoic acid (Acros) **9** (1.38 g) was dissolved in 40 mL acetone, then K_2CO_3 (3.0 g) and dimethyl sulfate (2.7g) were added in one portion. The mixture was heated to reflux for 1-2 hours until **9** was completely consumed (monitored by TLC). Then the reaction mixture was cooled down to room temperature. The reaction mixture was passing through a short pad of silica gel to remove K_2CO_3 , and 200 mL acetone was used subsequently to wash the residue. The clear filtration was condensed under vacuum to give a syrup-like residue, which was dissolved in 15 mL HNO_3 (68-70% in aqueous as received) at room temperature. The solution was well stirred overnight until the starting material was consumed (monitored by TLC). Compound **11** was precipitated out from the solution (Nitrates **11a** and **11b** were still in the solution and were recovered by extraction with DCM). Simple filtration will afford **11**, which was recrystallized from MeOH or EtOAc/Hexane to give large colorless square crystals as pure product in about 50-60% yield. ^1H NMR (300 MHz, CDCl_3): 7.58 (1H, dd, $J = 1.2$ Hz, $J = 7.2$ Hz), 7.47 (1H, t, $J = 8.1$ Hz), 7.24 (1H, dd, $J = 1.2$ Hz, $J = 8.1$ Hz), 3.90 (3H, s), 3.87 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 163.4, 150.9, 130.8, 123.6, 122.1, 117.1, 56.8, 52.9.

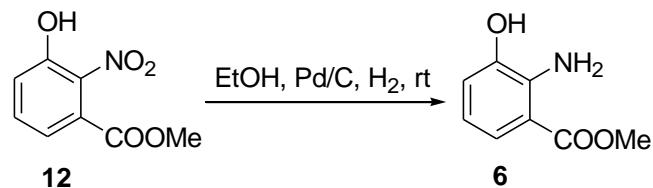
Synthesis of **12**



Compound **11** (2.11 g) was dissolved in 35 mL dichloromethane, then AlCl_3 (1.9g) was added in one portion. The solution was stirred at room temperature overnight. After the completion of the reaction, water was added to stop the reaction and the product was extracted by DCM. The organic layer was passing through a short pad of silica and washed with EtOAc to afford **12** in 98% yield as light yellow solid. ^1H NMR (300 MHz, CDCl_3): 10.13 (1H, s), 7.57 (1H, tri, $J = 7.5$ Hz), 7.24 (1H, dd, $J = 1.5$ Hz, $J = 7.5$ Hz),

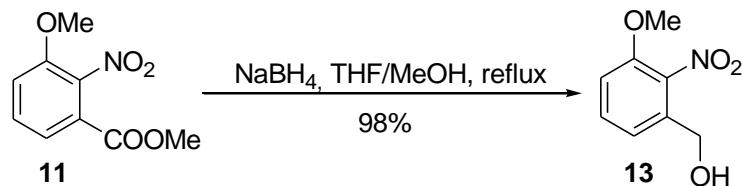
7.07 (1H, dd, J = 1.5 Hz, J = 7.5 Hz), 3.93 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 166.8, 154.7, 136.2, 131.2, 122.1, 120.9, 53.6.

Synthesis of 6



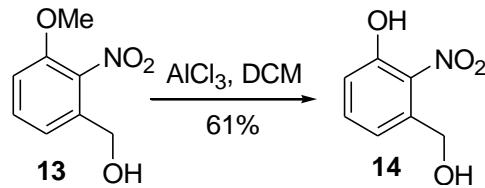
To a solution of **12** (600 mg) in EtOH (25 mL) was added Pd/C (40 mg). The reaction mixture in the flask was stirred under H_2 at room temperature overnight. The resulting product mixture was filtered to afford **6** in quantitative yield as white solid. ^1H NMR (300 MHz, CDCl_3): 7.46 (1H, d, J = 8.1 Hz), 6.82 (1H, d, J = 7.8 Hz), 6.50 (1H, tri, J = 7.8 Hz), 6.05 (4H, br), 3.87 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 169.3, 143.8, 140.5, 123.2, 118.4, 115.8, 111.8, 51.9.

Synthesis of 13

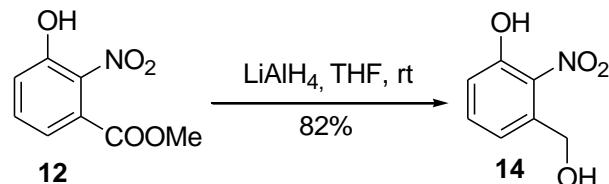


To a solution of **11** (300 mg) in dry THF (25 mL) was added NaBH_4 (250 mg), the reaction mixture was heated to reflux for 4 hours. Then a small amount of methanol was added in portions and the reaction was monitored by TLC carefully until **11** disappeared completely. Then the reaction was cooled down by icy water bath and quenched by addition of water. The solvent was removed under vacuum. The residue was dissolved by adding water and DCM. The water was washed by DCM three times. The organic phase was purified by silica gel chromatography to give **13** in 98% yields as white solid. ^1H NMR (300 MHz, CDCl_3): 7.74 (1H, t, J = 8.1 Hz), 7.07 (1H, d, J = 7.5 Hz), 6.97 (1H, d, J = 8.4 Hz), 4.58 (2H, s), 3.86 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 151.2, 134.4, 131.8, 120.5, 112.3, 60.9, 56.6.

Synthesis of 14

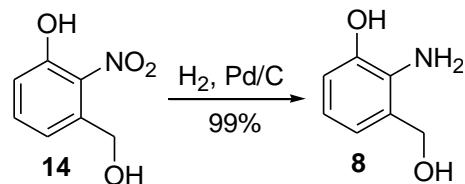


Method-I: Compound **14** was synthesized from **13**, using the method for the preparation of **12**.



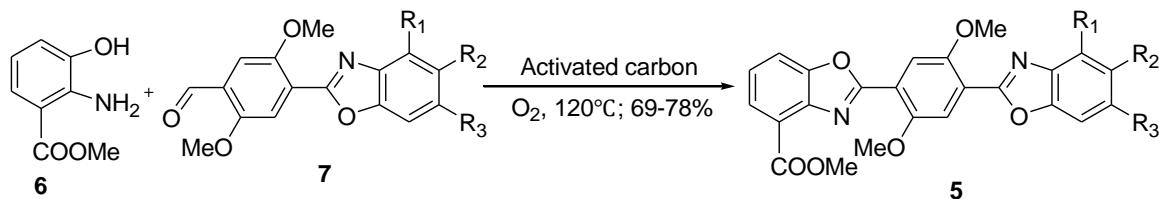
Method-II: To a solution of **12** (300 mg) in dry THF (25 mL) was added LiAlH₄ (250 mg), the reaction mixture was stirred at room temperature for 2 hours. Upon completion of the reaction, the reaction was quenched by addition of Na₂SO₄ solution. The resulting mixture was filtered and then crude product was concentrated and purified on a silica gel column to give **12** in 82-90% yields. ¹H NMR (300 MHz, CDCl₃): 10.58 (1H, s), 7.54 (1H, t, *J* = 8.1 Hz), 7.25 (1H, d, *J* = 6.3 Hz), 7.12 (1H, d, *J* = 8.4 Hz), 4.97 (2H, s). ¹³C NMR (75 MHz, CDCl₃): 155.9, 139.5, 136.3, 120.8, 119.4, 63.4.

Synthesis of **8**

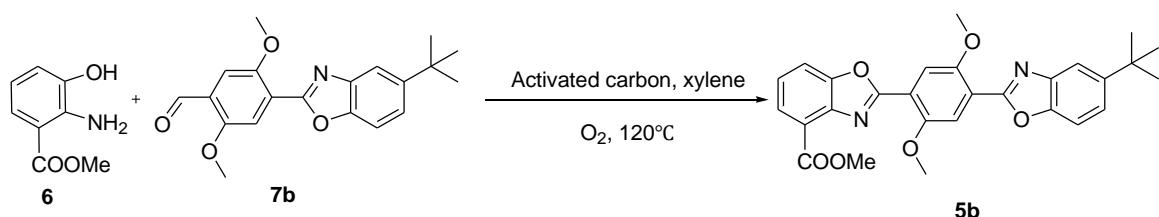


To a solution of **14** (500 mg) in EtOH (35 mL) was added Pd/C (35 mg). The reaction mixture in the flask was stirred under H₂ at room temperature overnight. The resulting product mixture was filtered to afford **8** in quantitative yield as white solid. ¹H NMR (300 MHz, CDCl₃): 6.58 (1H, d, *J* = 7.8 Hz), 6.53 (1H, d, *J* = 7.2 Hz), 6.46 (1H, t, *J* = 7.5 Hz), 4.48 (2H, s), 3.94 (2H, br). ¹³C NMR (75 MHz, CDCl₃): 144.9, 133.7, 126.6, 120.4, 118.4, 114.4, 62.9.

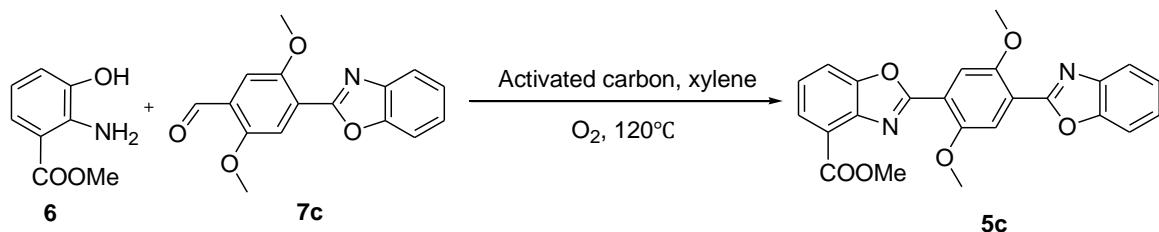
General procedure B



Synthesis of 5b

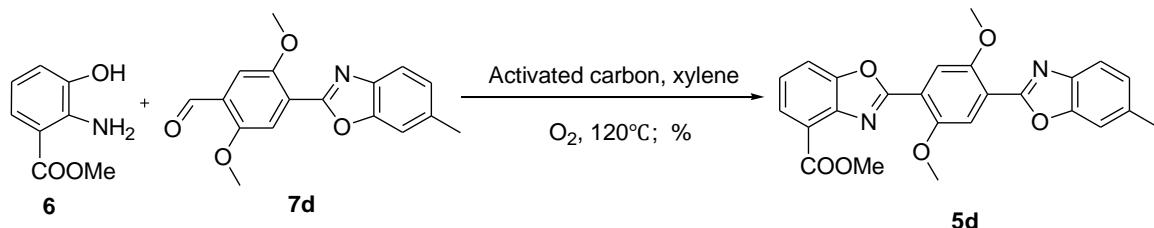


Synthesis of 5c



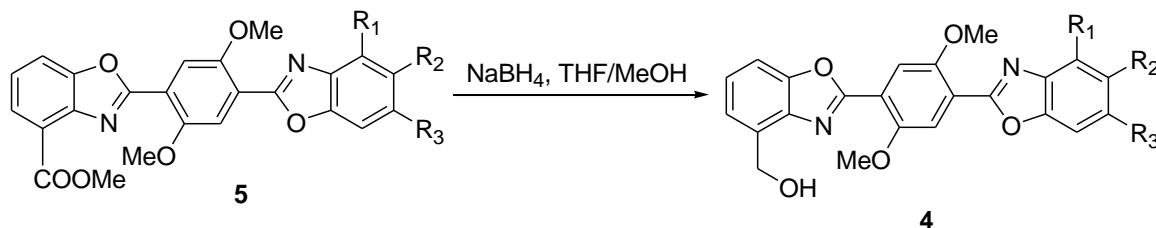
Compound **5c** was made according to the general procedure **B**. ¹H NMR (300 MHz, CDCl₃): 8.04 (1H, d, *J* = 7.8 Hz), 7.98 (1H, s), 7.88 (1H, s), 7.86-7.83 (1H, m), 7.62 (1H, m), 7.45 (1H, d, *J* = 8.1 Hz), 7.40-7.35 (2H, m), 4.11 (3H, s), 4.10 (3H, s), 4.06 (3H, s). ¹³C NMR (75 MHz, CDCl₃): 165.7, 162.7, 160.5, 152.7, 152.3, 153.3, 151.3, 150.5, 141.9, 141.2, 127.2, 125.5, 124.7, 124.6, 122.2, 120.4, 119.9, 118.8, 115.4, 115.0, 114.8, 110.7, 57.1, 56.9, 52.5.

Synthesis of **5d**



Compound **5d** was made according to the general procedure **B**. ¹H NMR (300 MHz, CDCl₃): 8.07 (1H, d, *J* = 7.8 Hz), 7.99 (1H, s), 7.89 (1H, s), 7.84 (1H, d, *J* = 8.1 Hz), 7.72 (1H, d, *J* = 8.4 Hz), 7.48-7.43 (2H, m), 7.20 (1H, d, *J* = 8.1 Hz), 4.13 (3H, s), 4.12 (3H, s), 4.08 (3H, s), 2.53 (3H, s). ¹³C NMR (75 MHz, CDCl₃): 166.2, 163.2, 160.5, 153.1, 152.6, 151.8, 151.3, 141.7, 140.3, 136.6, 127.6, 126.4, 125.2, 122.6, 120.6, 120.2, 118.9, 115.9, 115.5, 115.2, 111.3, 57.5, 57.4, 52.9, 22.3.

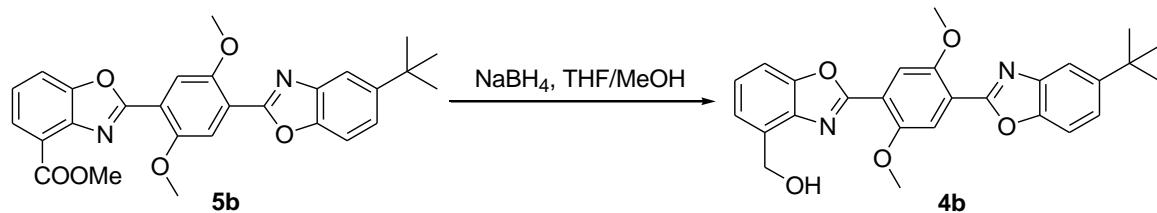
General procedure C



To a solution of **5** (300 mg) in dry THF (25 mL) was added NaBH₄ (400 mg), the reaction mixture was heated to reflux for 3 hours. Then small amount of methanol was added quickly and the reaction was monitored by TLC carefully until **5** disappeared completely. The reaction was cooled down by icy water bath and quenched by addition of water. The solvent was removed under vacuum. The residue was dissolved by adding water and DCM. The aqueous layer was washed by DCM three times. The organic phase

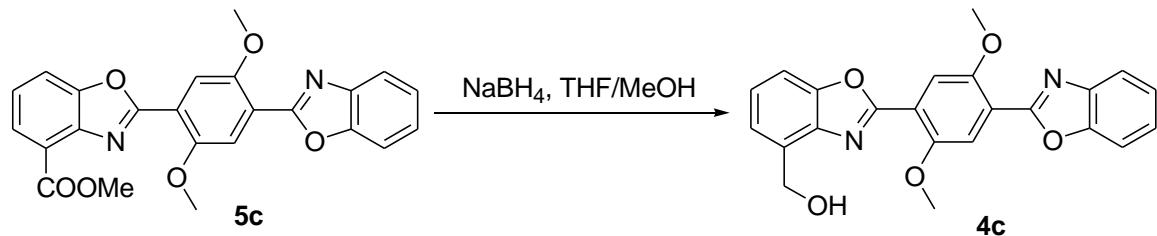
was purified by silica gel chromatography to give **4** in 82-88% yields as light yellow solid.

Synthesis of **4b**



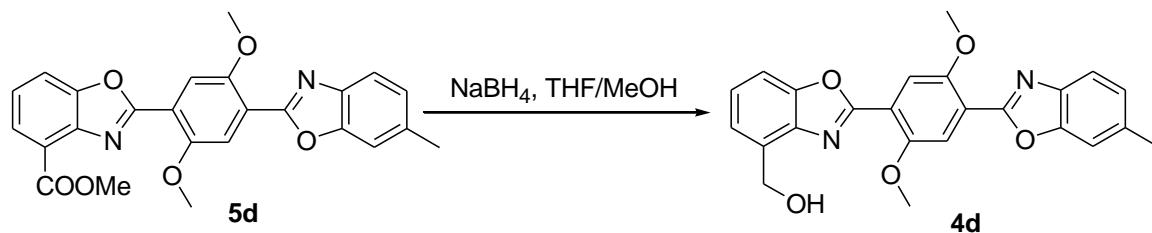
Compound **4b** was made according to the general procedure **C**. ¹H NMR (300 MHz, CDCl₃): 7.87 (1H, s), 7.82 (2H, s), 7.54-7.50 (2H, m), 7.43 (1H, dd, *J* = 1.5 Hz, *J* = 8.7 Hz), 7.34-7.30 (2H, m), 5.16 (2H, d, *J* = 6.6 Hz), 5.13 (2H, d, *J* = 6.0 Hz), 4.08 (3H, s), 4.06 (3H, s), 3.67 (1H, br), 1.39 (9H, s). ¹³C NMR (75 MHz, CDCl₃): 160.7, 160.5, 152.2, 150.5, 148.5, 148.2, 141.8, 139.8, 133.1, 125.3, 123.3, 122.7, 119.6, 118.9, 116.9, 114.9, 114.8, 109.7, 109.7, 61.9, 56.9, 56.9, 34.9, 31.8.

Synthesis of **4c**



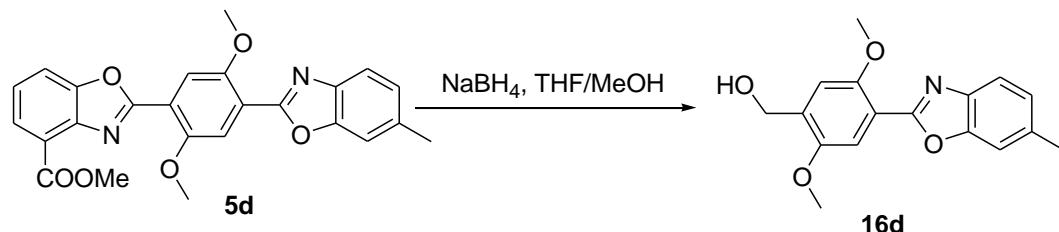
Compound **4c** was made according to the general procedure **C**. ¹H NMR (300 MHz, DMSO-*d*₆): 7.88-7.81 (4H, m), 7.68 (1H, d, *J* = 7.5 Hz), 7.50-7.42 (4H, m), 5.34 (1H, tri, *J* = 5.7 Hz), 4.95 (2H, d, *J* = 5.7 Hz), 4.01 (3H, s), 4.00 (3H, s). ¹³C NMR (75 MHz, DMSO-*d*₆): 161.3, 159.9, 151.7, 151.6, 150.2, 149.9, 141.1, 138.4, 134.6, 125.7, 125.5, 124.9, 122.4, 119.9, 119.1, 118.8, 115.1, 114.9, 110.9, 109.0, 58.4, 56.7, 56.7.

Synthesis of **4d**



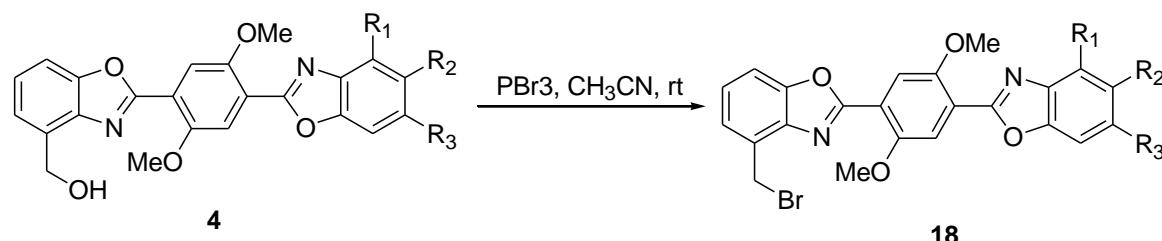
Compound **4d** was made according to the general procedure C. ^1H NMR (300 MHz, CDCl_3): 7.88 (2H, s), 7.72 (1H, d, $J = 8.1$ Hz), 7.56 (1H, dd, $J = 1.2$ Hz, $J = 7.8$ Hz), 7.44 (1H, s), 7.39-7.31 (2H, m), 7.20 (1H, d, $J = 7.8$ Hz), 5.16 (2H, d, $J = 6.6$ Hz), 4.11 (3H, s), 4.09 (3H, s), 2.53 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 161.1, 152.8, 150.6, 139.7, 135.4, 134.3, 125.6, 119.3, 114.8, 112.7, 111.9, 110.6, 60.9, 56.7, 55.8, 21.8.

Byproduct **16d**



Compound **16d** was separated in the synthesis of **4d** when MeOH was used as the solvent. ^1H NMR (300 MHz, CDCl_3): 7.64 (1H, d, $J = 8.1$ Hz), 7.55 (1H, s), 7.37 (1H, s), 7.14 (1H, d, $J = 8.1$ Hz), 7.10 (1H, s), 4.73 (2H, s), 3.92 (3H, s), 3.87 (3H, s), 2.48 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 161.8, 153.4, 151.3, 140.4, 136.0, 134.9, 126.3, 119.9, 115.5, 113.3, 112.6, 111.3, 61.6, 57.4, 56.5, 22.4.

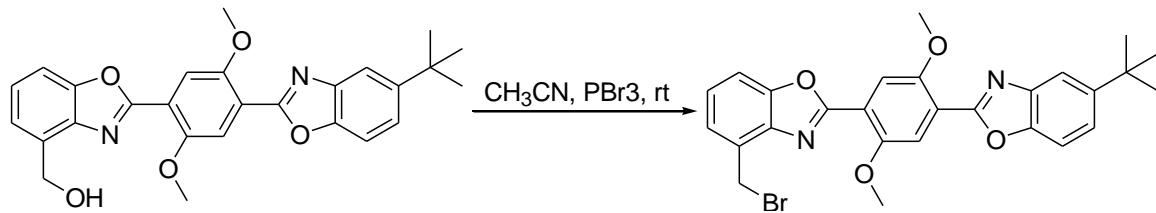
General procedure D



To a solution of **4** (1.0 mmol) in acetonitrile (15 mL) was added PBr_3 (2 mmol), the reaction mixture was stirred at room temperature overnight. The reaction was quenched

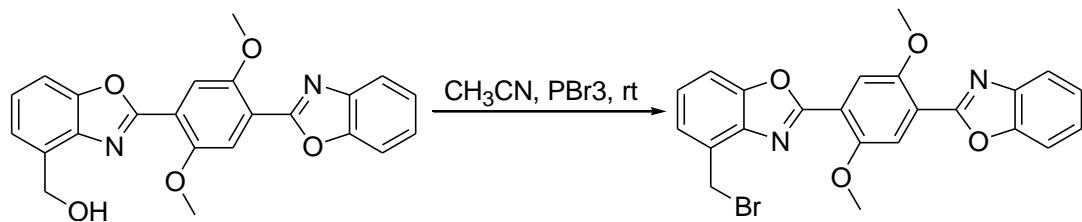
by addition of water and the resulting reaction mixture was concentrated. The residue was dissolved by adding water and DCM. The aqueous layer was washed by DCM three times. The organic phase was purified by silica gel chromatography to give **18** in up to 99% yield as light yellow solid.

Synthesis of **18b**



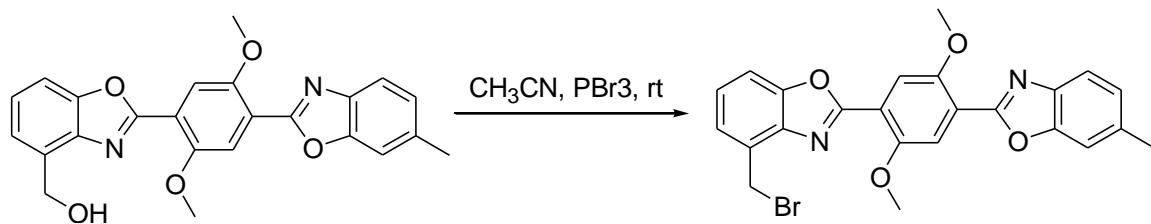
Compound **18b** was made according to the general procedure **D**. ¹H NMR (300 MHz, CDCl₃): 7.91 (1H, s), 7.89 (2H, s), 7.57 (1H, d, *J* = 8.4 Hz), 7.55 (1H, d, *J* = 8.7 Hz), 7.45 (2H, d, *J* = 8.7 Hz), 7.36 (1H, t, *J* = 7.8 Hz), 4.99 (2H, s), 4.13 (3H, s), 4.09 (3H, s), 1.41 (9H, s). ¹³C NMR (75 MHz, CDCl₃): 161.5, 160.9, 152.6, 152.6, 151.0, 148.8, 148.4, 142.1, 140.7, 130.1, 125.7, 125.6, 123.5, 120.0, 119.5, 117.1, 115.5, 115.2, 111.0, 109.9, 57.3, 57.3, 35.2, 32.0, 27.9.

Synthesis of **18c**



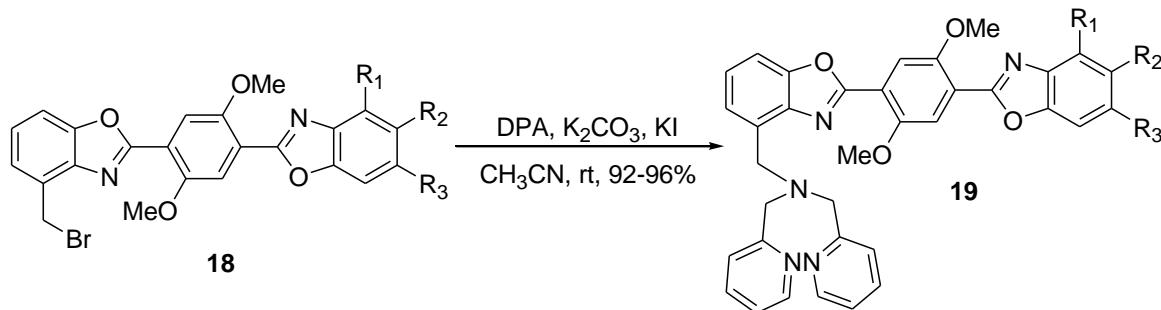
Compound **18c** was made according to the general procedure **D**. ¹H NMR (300 MHz, CDCl₃): 7.93 (1H, s), 7.91 (1H, s), 7.88-7.84 (1H, m), 7.66-7.63 (1H, m), 7.58 (1H, dd, *J* = 0.9 Hz, *J* = 8.1 Hz), 7.45 (1H, d, *J* = 6.9 Hz), 7.44-7.34 (3H, m), 4.99 (2H, s), 4.14 (3H, s), 4.10 (3H, s). ¹³C NMR (75 MHz, CDCl₃): 161.2, 160.6, 152.4, 150.8, 150.5, 141.9, 140.4, 129.9, 125.5, 125.5, 125.4, 124.6, 120.4, 119.6, 119.5, 115.3, 115.1, 110.8, 110.7, 57.1, 57.1, 27.7.

Synthesis of **18d**



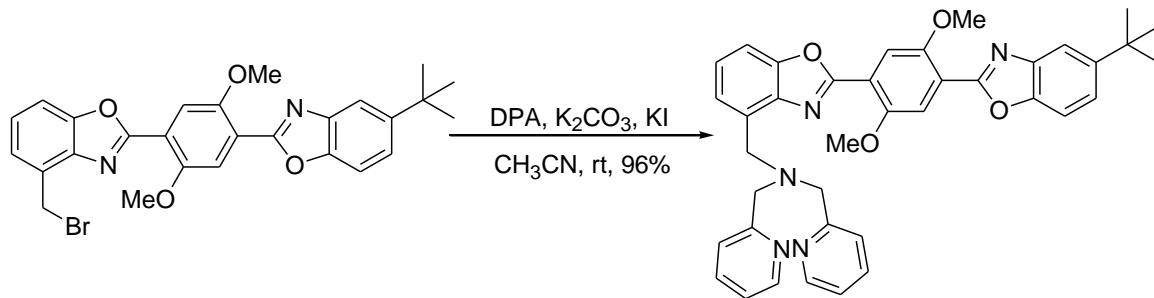
Compound **18d** was made according to the general procedure **D**. ¹H NMR (300 MHz, CDCl₃): 7.91 (1H, s), 7.88 (1H, s), 7.72 (1H, d, *J* = 8.1 Hz), 7.57 (1H, d, *J* = 7.8 Hz), 7.45 (1H, d, *J* = 6.6 Hz), 7.44 (1H, s), 7.36 (1H, t, *J* = 7.8 Hz), 7.19 (1H, d, *J* = 8.1 Hz), 4.99 (2H, s), 4.12 (3H, s), 4.09 (3H, s), 2.53 (3H, s). ¹³C NMR (75 MHz, CDCl₃): 161.2, 160.1, 152.4, 152.3, 150.8, 150.7, 140.4, 139.8, 136.0, 129.8, 125.9, 125.5, 125.4, 119.8, 119.2, 115.2, 115.0, 110.8, 110.8, 57.1, 57.0, 27.7, 21.9.

General procedure E



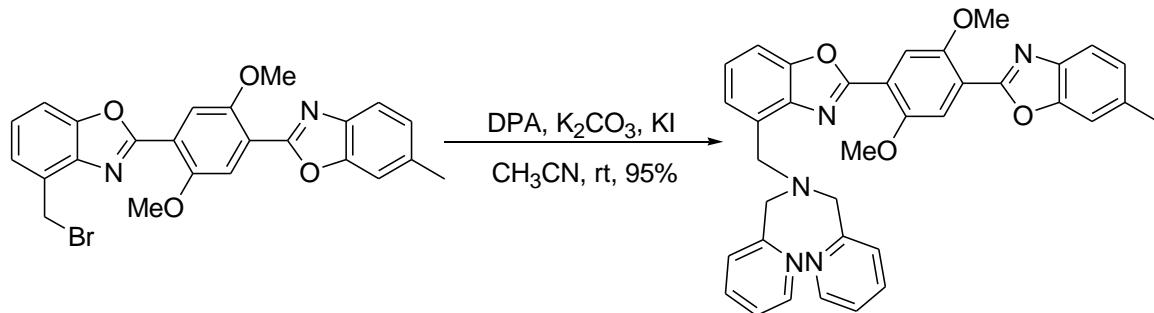
To a solution of **18** (1.0 mmol) in acetonitrile (15 mL) was added DPA (1.1 mmol), K₂CO₃ (2.0 mmol), and catalytic amount of KI. The reaction mixture was stirred at room temperature for 2 hours. The reaction mixture was concentrated and the residue was dissolved by adding water and DCM. The water was washed by DCM three times. The organic phase was purified by silica gel chromatography to give **19** in 92-96% yields as light yellow solid.

Synthesis of **19b**



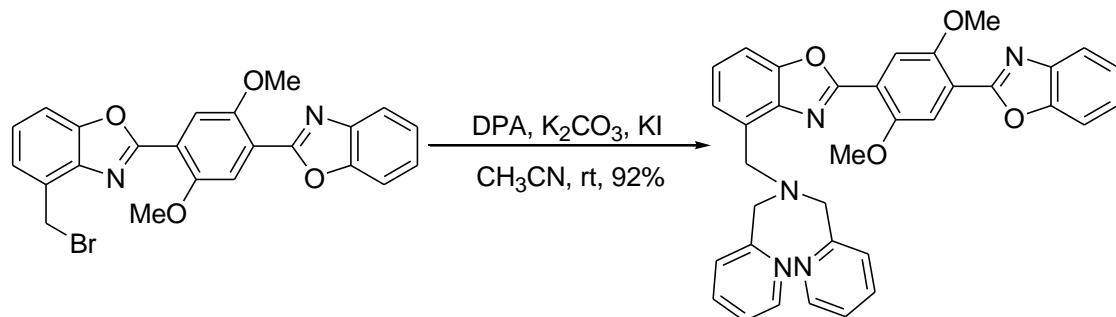
Compound **19b** was made according to the general procedure **E**. ¹H NMR (300 MHz, CDCl₃): 8.52 (2H, d, *J* = 6.4 Hz), 7.82 (3H, d, *J* = 6.6 Hz), 7.71 (1H, d, *J* = 8.1 Hz), 7.57 (2H, td, *J* = 1.5 Hz, *J* = 7.5 Hz), 7.51 (1H, d, *J* = 7.5 Hz), 7.45 (1H, d, *J* = 7.8 Hz), 7.38 (1H, dd, *J* = 1.8 Hz, *J* = 8.7 Hz), 7.30 (1H, t, *J* = 7.8 Hz), 7.07-7.02 (2H, m), 4.19 (2H, s), 4.04 (3H, s), 4.01 (3H, s), 3.89 (4H, s), 1.33 (9H, s). ¹³C NMR (75 MHz, CDCl₃): 160.7, 160.3, 152.6, 152.6, 150.9, 149.2, 148.8, 148.4, 142.1, 141.4, 136.6, 131.6, 125.4, 124.8, 123.5, 122.9, 122.1, 119.9, 119.8, 117.1, 115.5, 115.2, 109.9, 109.5, 60.6, 57.3, 57.3, 53.7, 35.2, 32.0, 29.9, 29.6.

Synthesis of **19c**



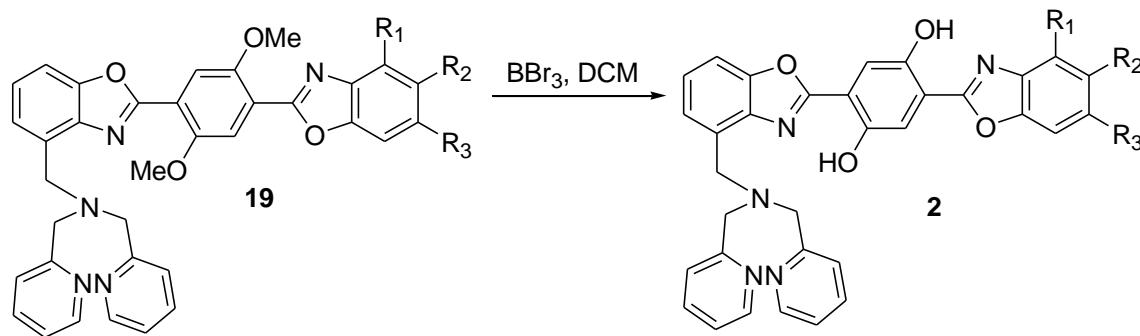
Compound **19c** was made according to the general procedure **E**. ¹H NMR (300 MHz, CDCl₃): 8.51 (2H, d, *J* = 4.2 Hz), 7.91 (1H, s), 7.87 (1H, s), 7.76 (2H, d, *J* = 7.8 Hz), 7.71 (1H, d, *J* = 8.1 Hz), 7.64 (2H, td, *J* = 1.5 Hz, *J* = 7.5 Hz), 7.575 (1H, d, *J* = 7.5 Hz), 7.51 (1H, d, *J* = 7.8 Hz), 7.44 (1H, s), 7.36 (1H, t, *J* = 7.8 Hz), 7.19 (1H, d, *J* = 8.1 Hz), 7.14-7.09 (2H, m), 4.26 (2H, s), 4.10 (3H, s), 4.08 (3H, s), 3.95 (4H, s), 2.52 (3H, s). ¹³C NMR (75 MHz, CDCl₃): 160.5, 160.1, 159.9, 152.3, 152.3, 150.8, 150.7, 148.9, 141.1, 139.8, 136.4, 136.0, 131.4, 125.9, 125.2, 124.6, 122.8, 121.9, 119.7, 119.5, 119.5, 115.2, 114.9, 110.8, 109.2, 60.3, 57.1, 57.0, 53.6, 21.9. TOF-MS ES⁺(m/z): [M+H]⁺ calcd for C₃₆H₃₂N₅O₄, 598.67; found, 598.96.

Synthesis of 19d



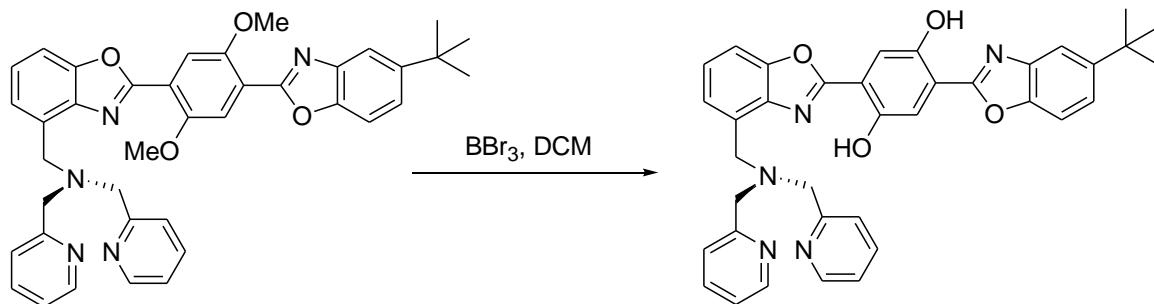
Compound **19d** was made according to the general procedure **E**. ^1H NMR (300 MHz, CDCl_3): 8.52 (2H, d, $J = 4.5$ Hz), 7.92 (1H, s), 7.89 (1H, s), 7.89-7.48 (1H, m), 7.78 (2H, d, $J = 7.8$ Hz), 7.67-7.62 (3H, m), 7.58 (1H, d, $J = 8.1$ Hz), 7.52 (1H, d, $J = 8.1$ Hz), 7.42-7.34 (4H, m), 7.15-7.10 (2H, m), 4.26 (2H, s), 4.12 (3H, s), 4.08 (3H, s), 3.96 (4H, s). HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{35}\text{H}_{30}\text{N}_5\text{O}_4$, 584.2298; found, 584.2316.

General procedure F



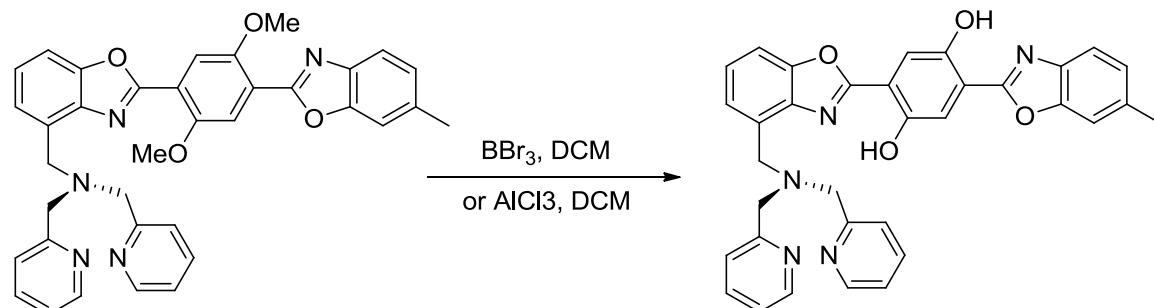
Compound **19** (0.5 mmol) was dissolved in anhydrous DCM, and the solution was cooled to -78°C . Then BBr_3 (1.2 mmol) was added slowly, and the resulting mixture was warmed to room temperature and stirred overnight. The reaction mixture was quenched by addition of water, and DCM layer was separated on a silica gel column in 72-78% yield.

Synthesis of 2b



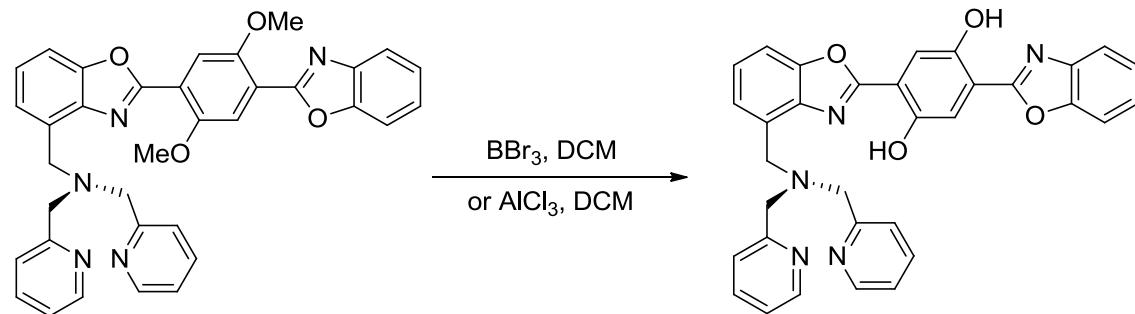
Compound **2b** was made according to the general procedure **F**. ^1H NMR (300 MHz, CDCl_3): 11.09 (1H, s), 11.01 (1H, s), 8.53 (2H, t, J = 1.2 Hz), 7.80 (3H, m), 7.72-7.62 (4H, m), 7.58-7.48 (4H, m), 7.40 (1H, t, J = 7.8 Hz), 7.15 (2H, m), 4.15 (2H, s), 3.91 (4H, d, J = 1.5 Hz), 1.42 (9H, tri, J = 1.2 Hz). ^{13}C NMR (75 MHz, CDCl_3): 161.9, 161.6, 159.6, 151.1, 150.9, 149.3, 149.0, 147.4, 140.0, 139.5, 136.6, 130.6, 126.1, 125.8, 123.9, 122.8, 122.0, 116.1, 114.9, 114.5, 110.0, 109.6, 60.4, 53.7, 35.1, 31.8.

Synthesis of **2c**

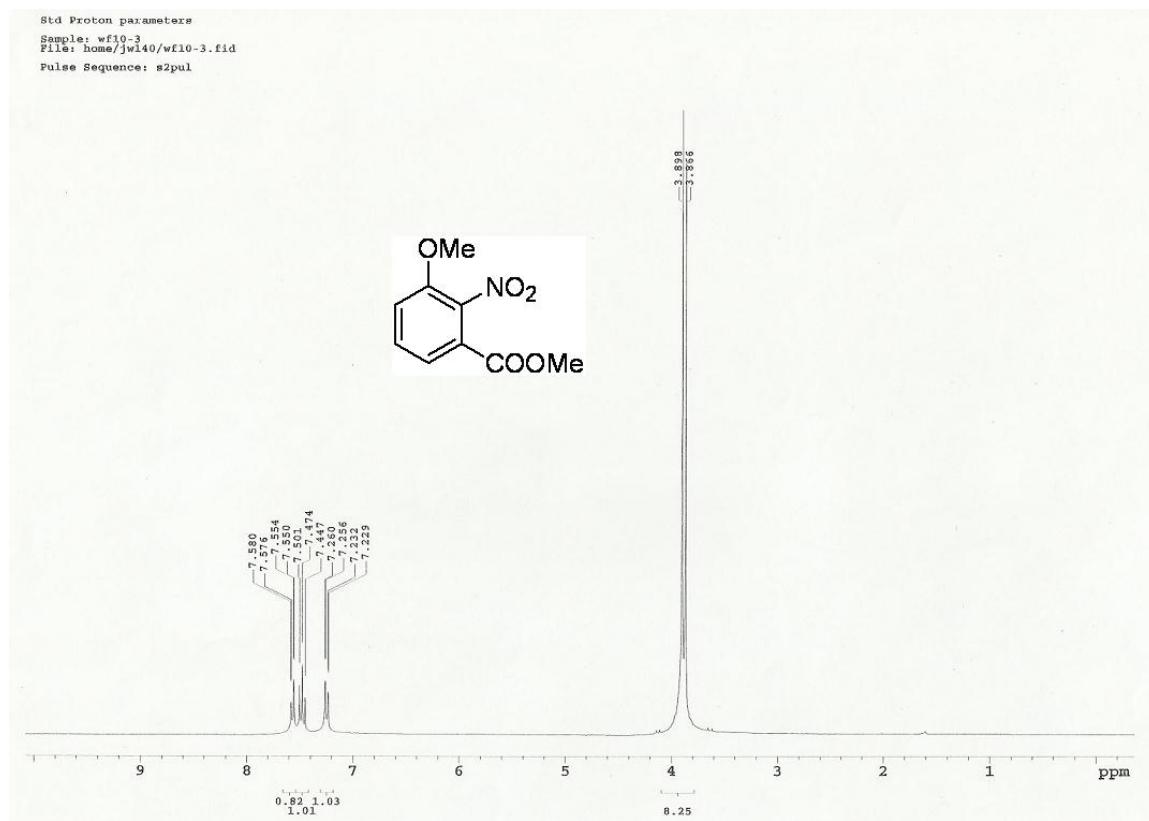


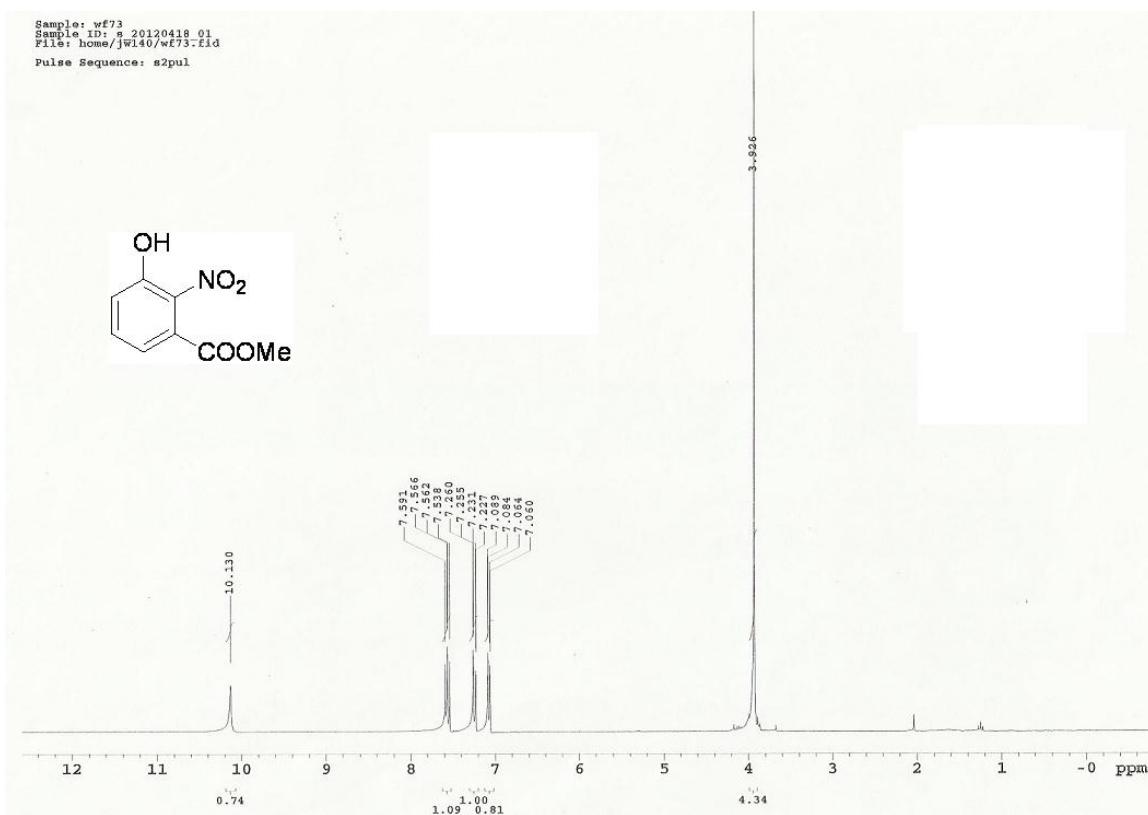
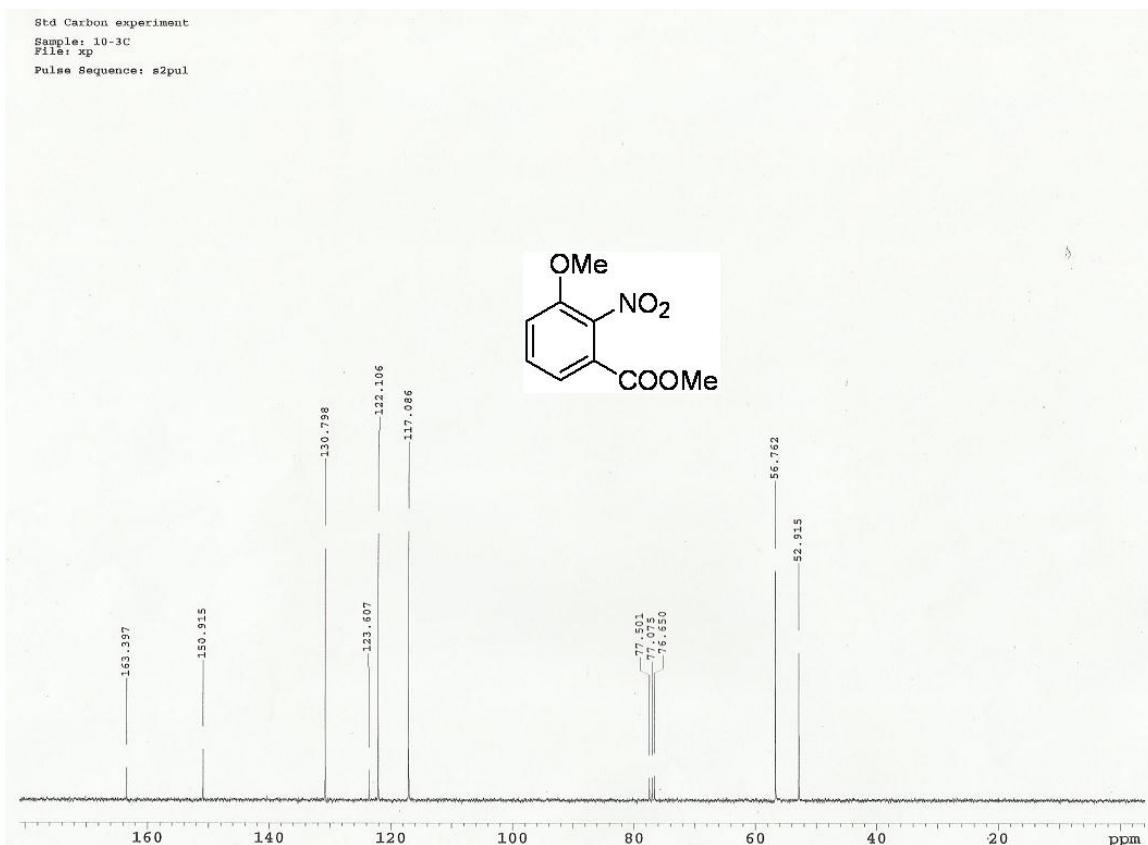
Compound **2c** was made according to the general procedure **F**. ^1H NMR (300 MHz, CDCl_3): 11.00 (1H, s), 10.97 (1H, s), 8.53 (2H, t, J = 4.8 Hz), 7.74-7.64 (3H, m), 7.63-7.57 (1H, m), 7.53-7.49 (2H, m), 7.42 (1H, s), 7.35 (1H, tri, J = 7.8 Hz), 7.19-7.13 (2H, m), 4.12 (2H, s), 3.90 (4H, s), 2.51 (3H, s). ^{13}C NMR (75 MHz, CDCl_3): 161.5, 161.3, 159.6, 150.9, 150.8, 149.6, 149.2, 149.0, 139.4, 137.8, 136.7, 136.5, 130.5, 126.5, 126.1, 125.7, 122.8, 121.9, 118.9, 114.8, 114.4, 114.4, 114.3, 110.9, 109.5, 60.3, 53.6, 21.8. HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{34}\text{H}_{28}\text{N}_5\text{O}_4$, 570.2141; found, 570.2169.

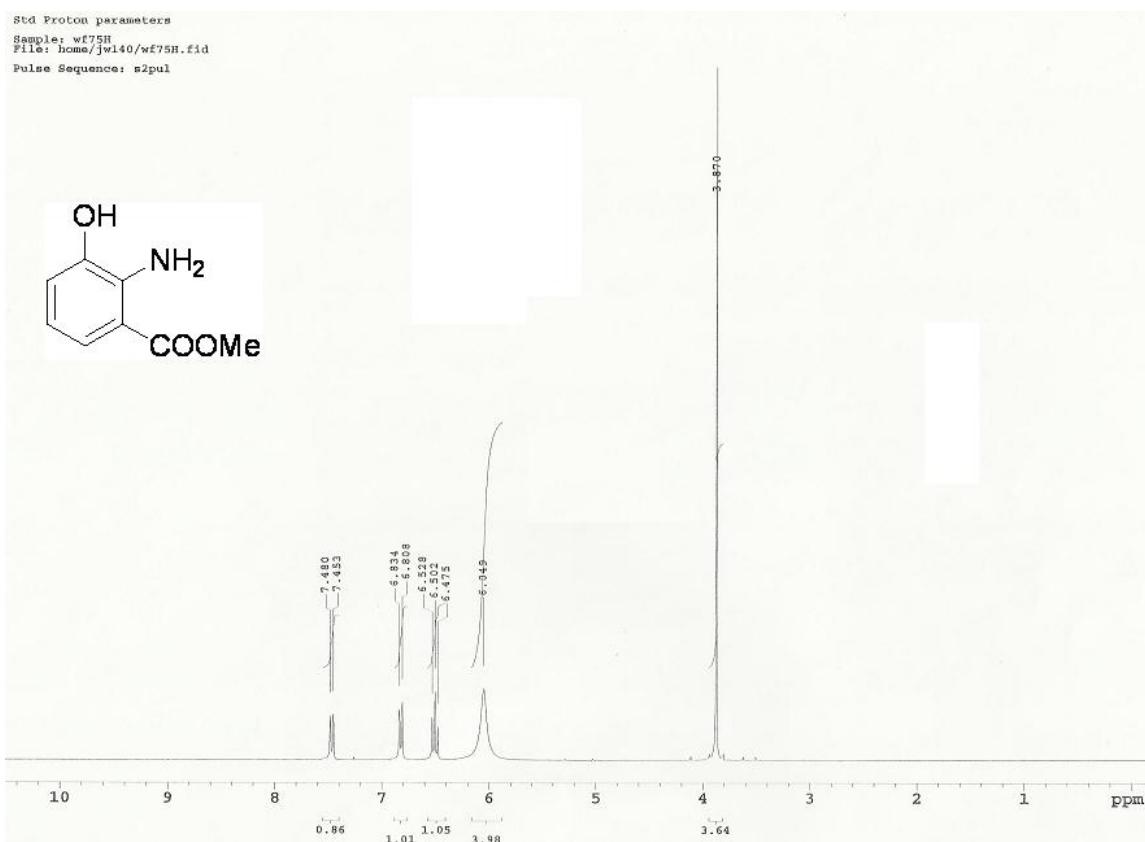
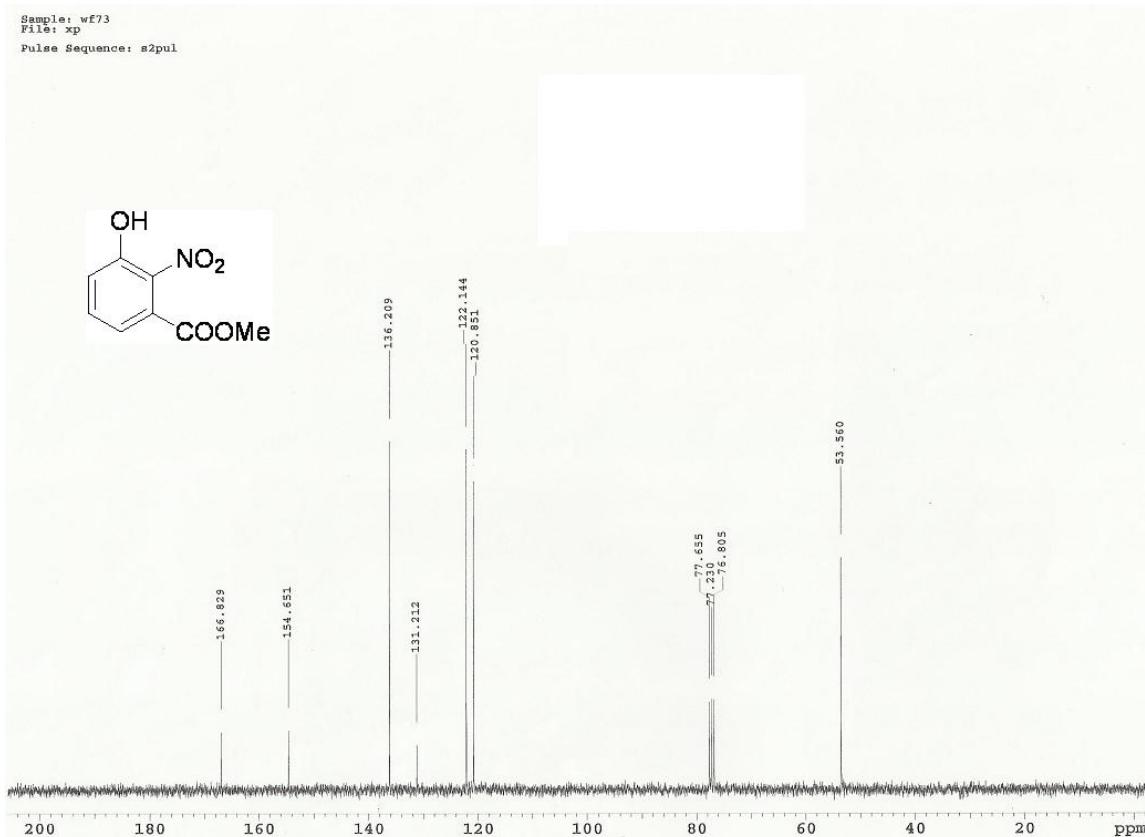
Synthesis of **2d**

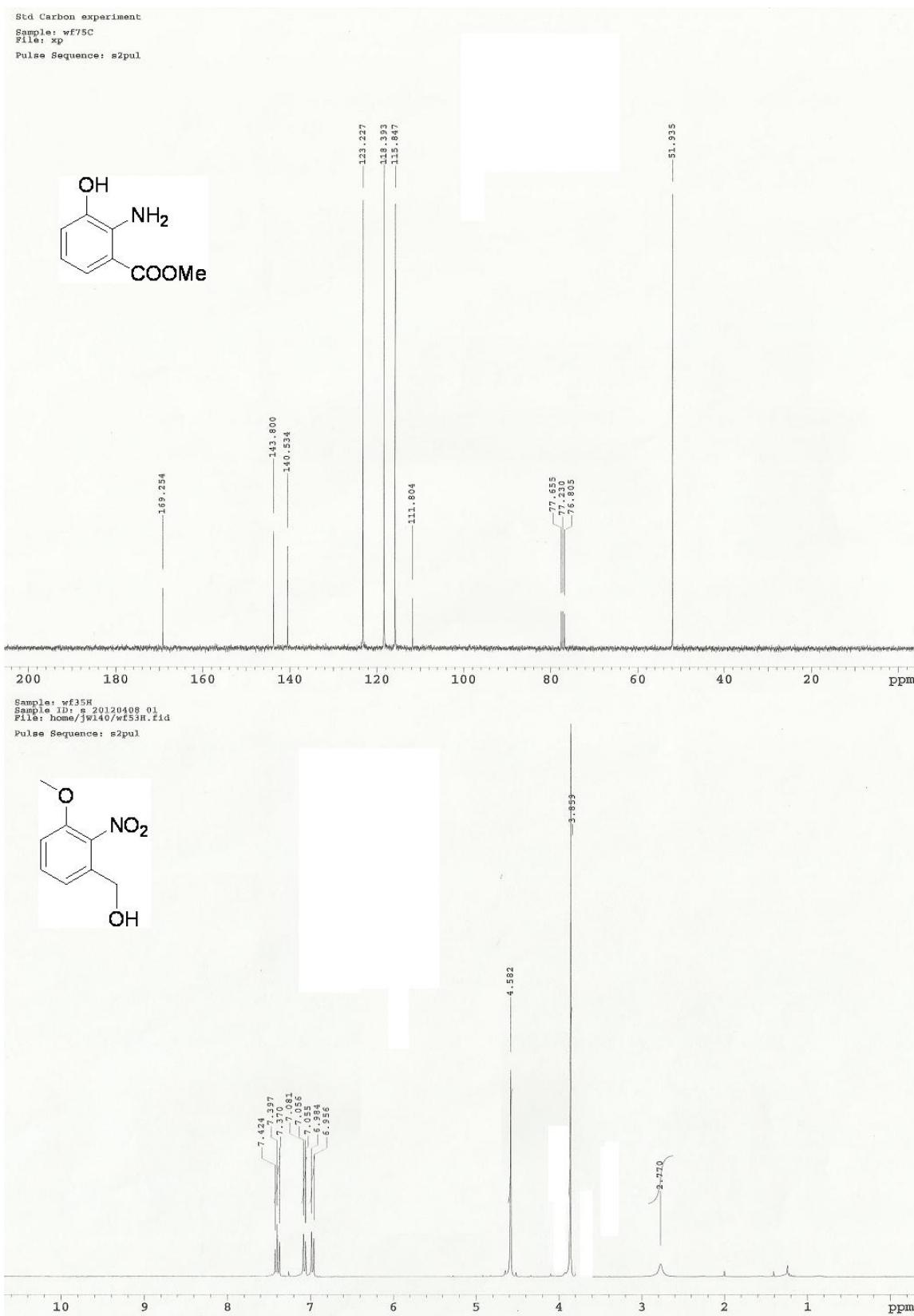


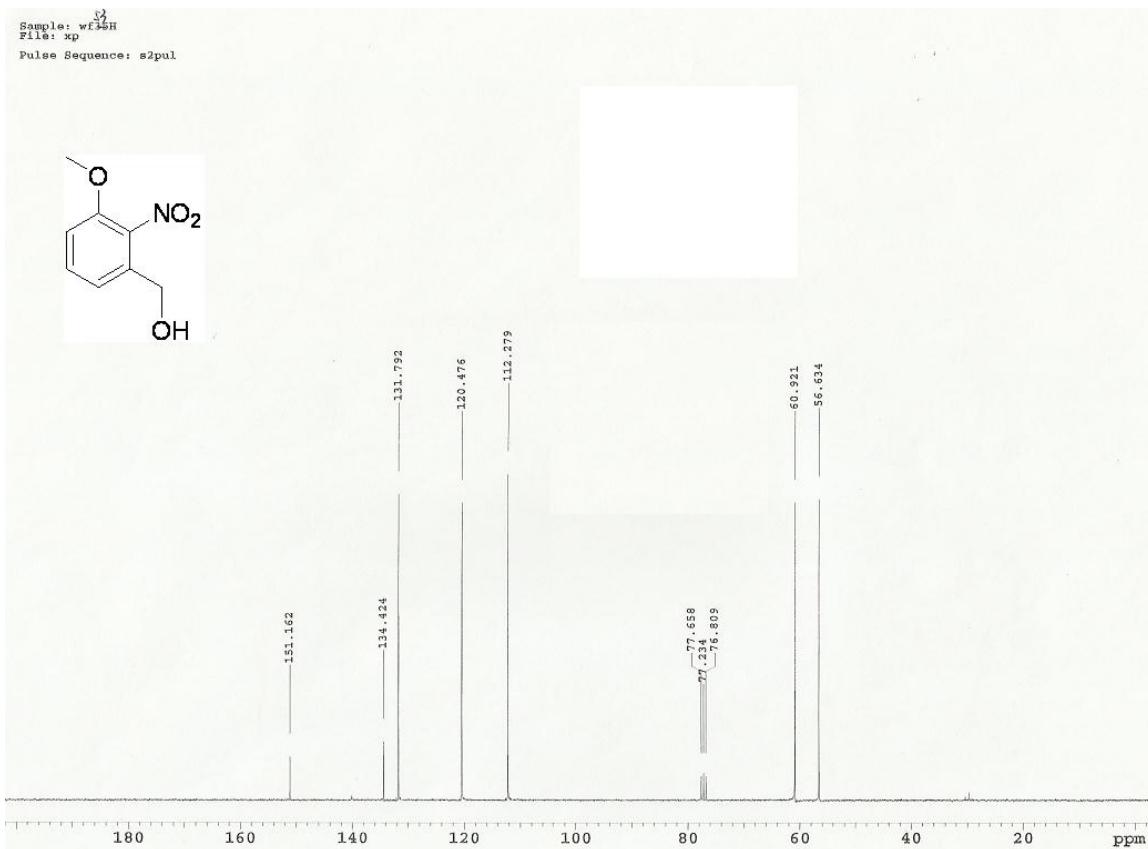
Compound **2d** was made according to the general procedure **F**. ^1H NMR (300 MHz, CDCl_3): 11.03 (2H, s), 8.53 (2H, d, $J = 4.2$ Hz), 7.81 (1H, s), 7.78 (1H, s), 7.76 (1H, m), 7.72-7.63 (6H, m), 7.54 (2H, m), 7.46-7.36 (4H, m), 7.15 (2H, m), 4.15 (2H, s), 3.91 (4H, s). ^{13}C NMR (75 MHz, CDCl_3): 161.8, 161.4, 159.6, 150.9, 149.3, 149.2, 149.0, 139.9, 139.4, 136.6, 130.5, 126.1, 125.9, 125.8, 125.2, 122.8, 122.0, 119.5, 114.6, 114.6, 110.8, 109.5, 60.3, 53.6. HRMS (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{33}\text{H}_{26}\text{N}_5\text{O}_4$, 556.1985; found, 556.1993.

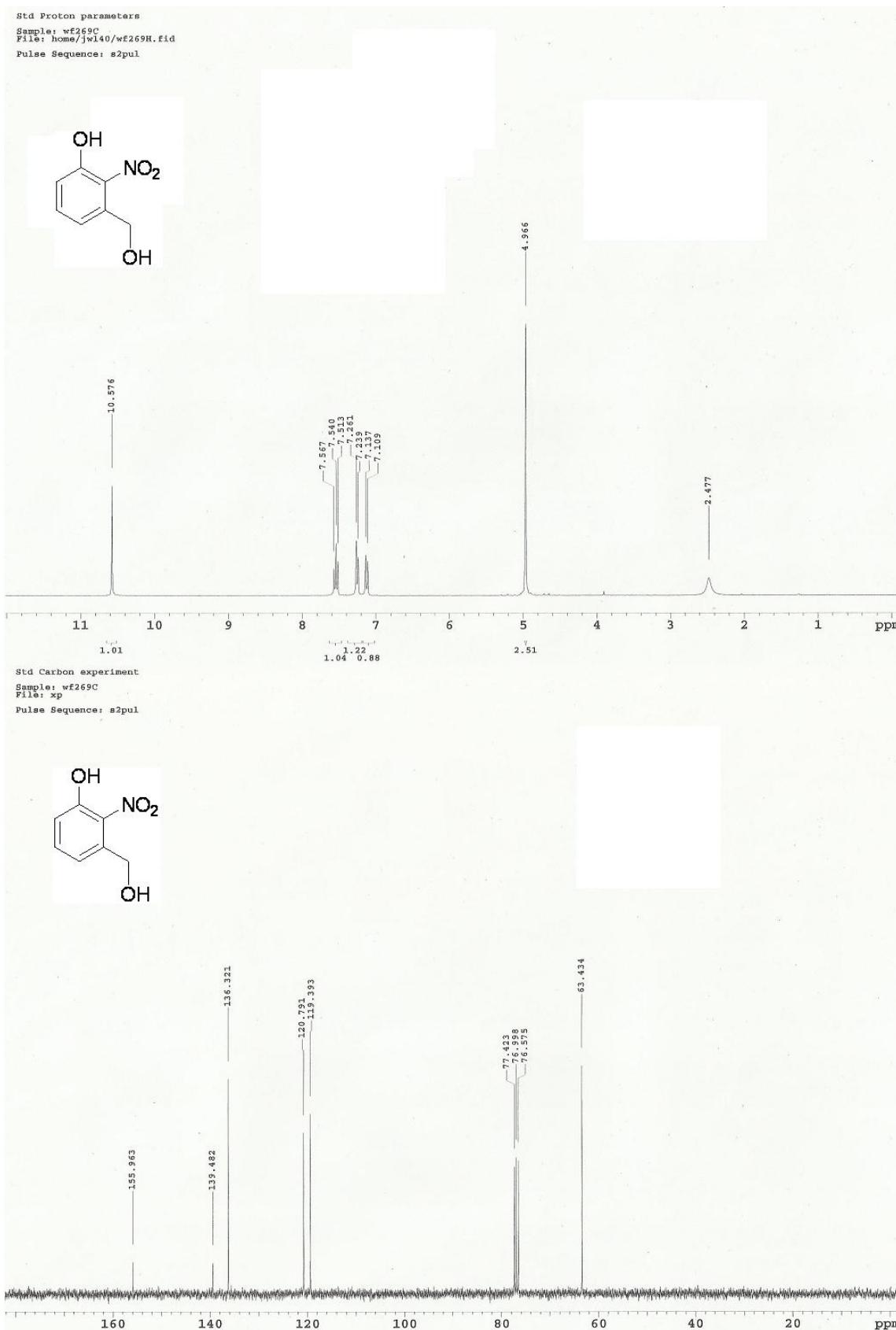


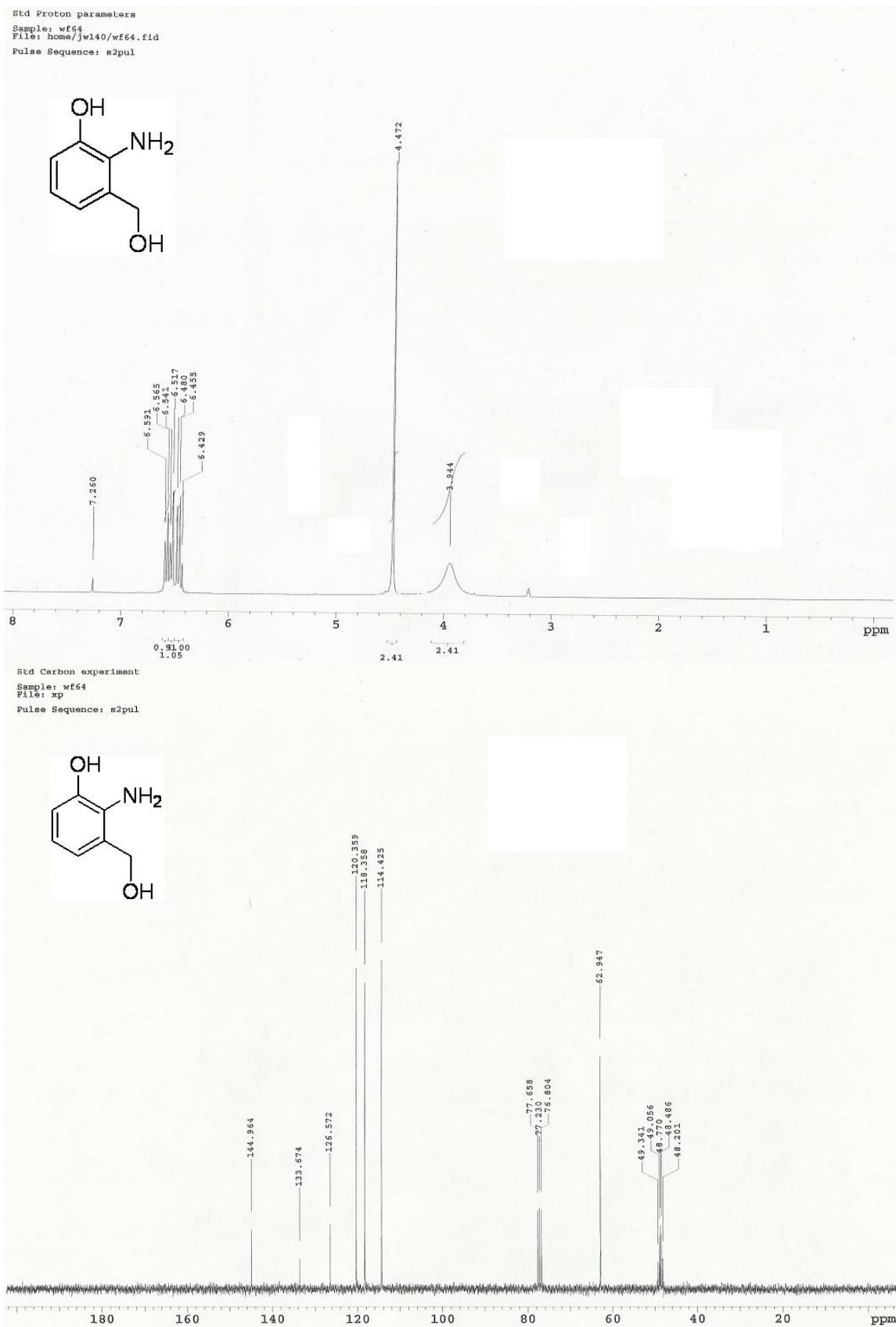


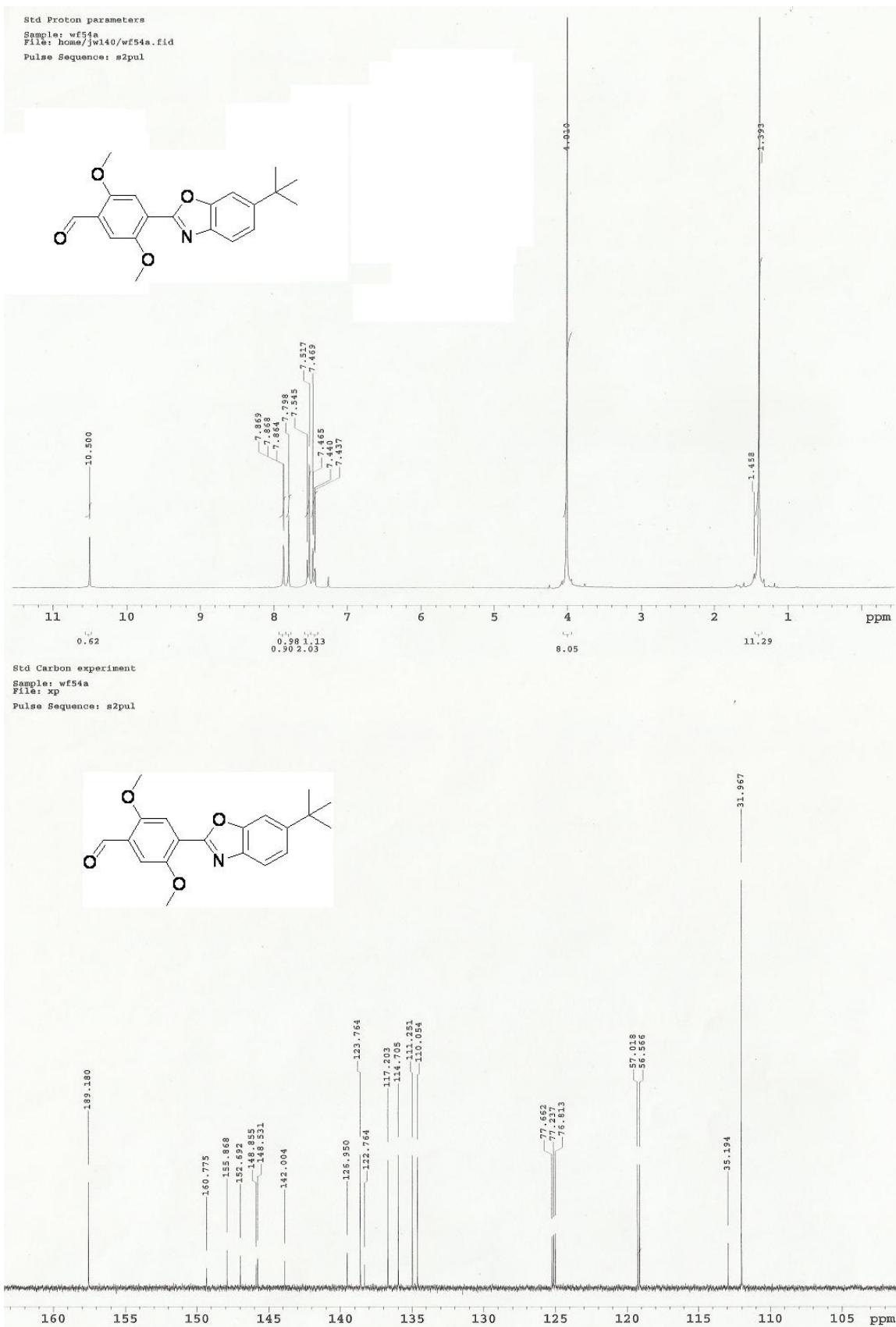


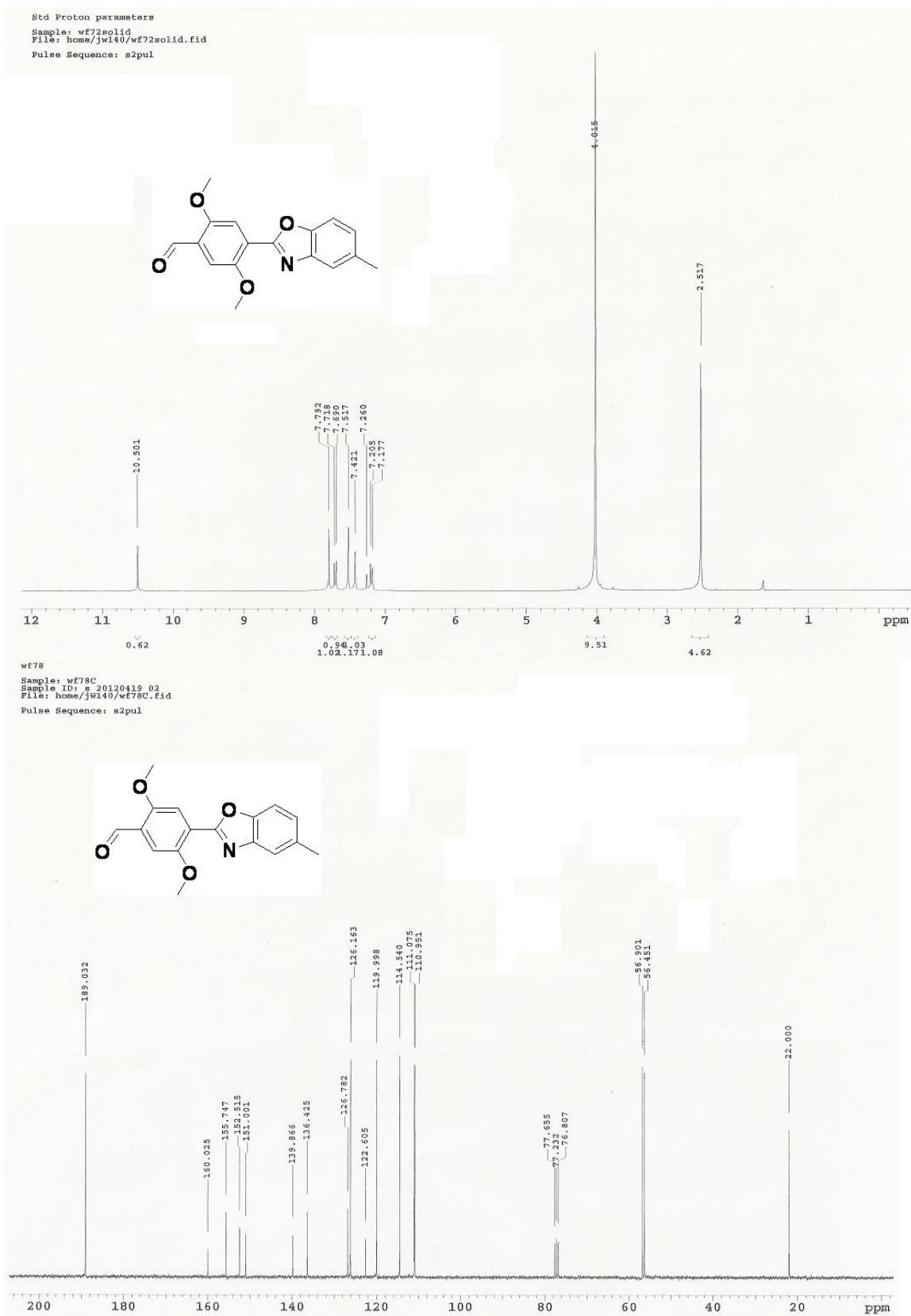


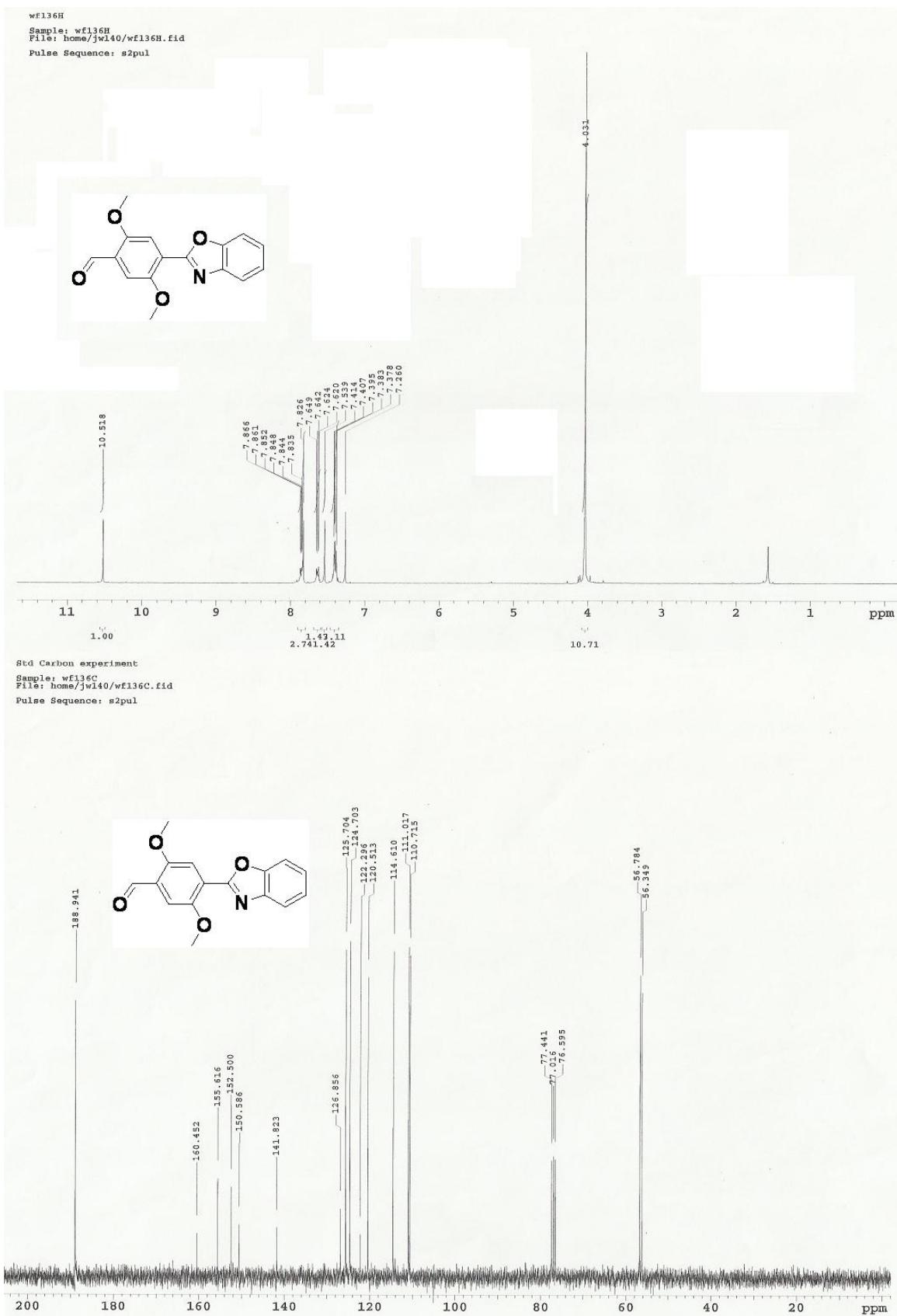




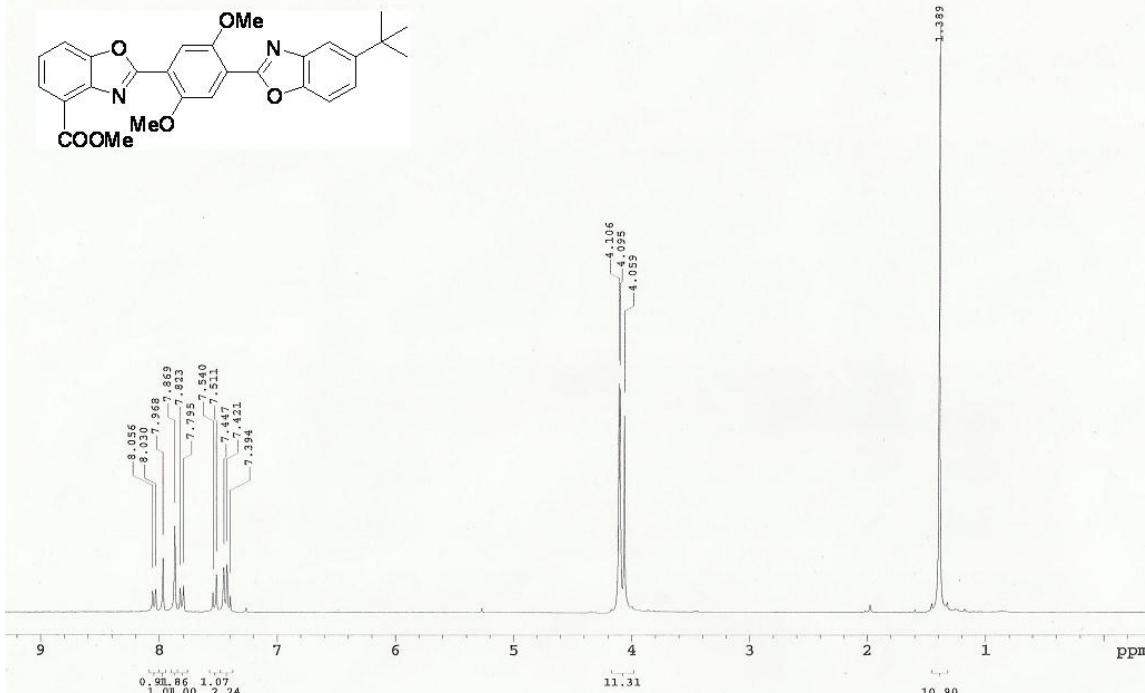
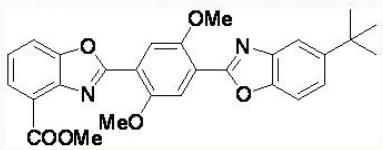




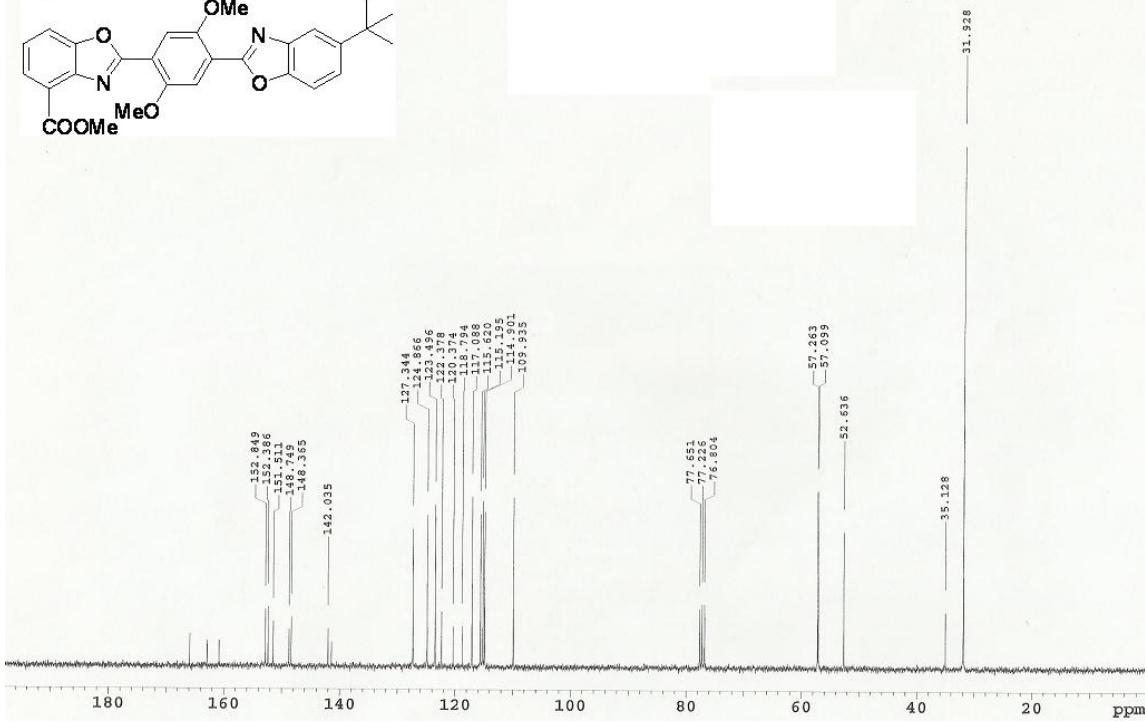
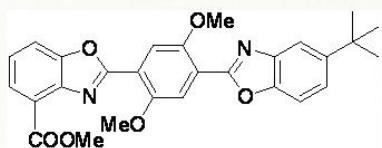




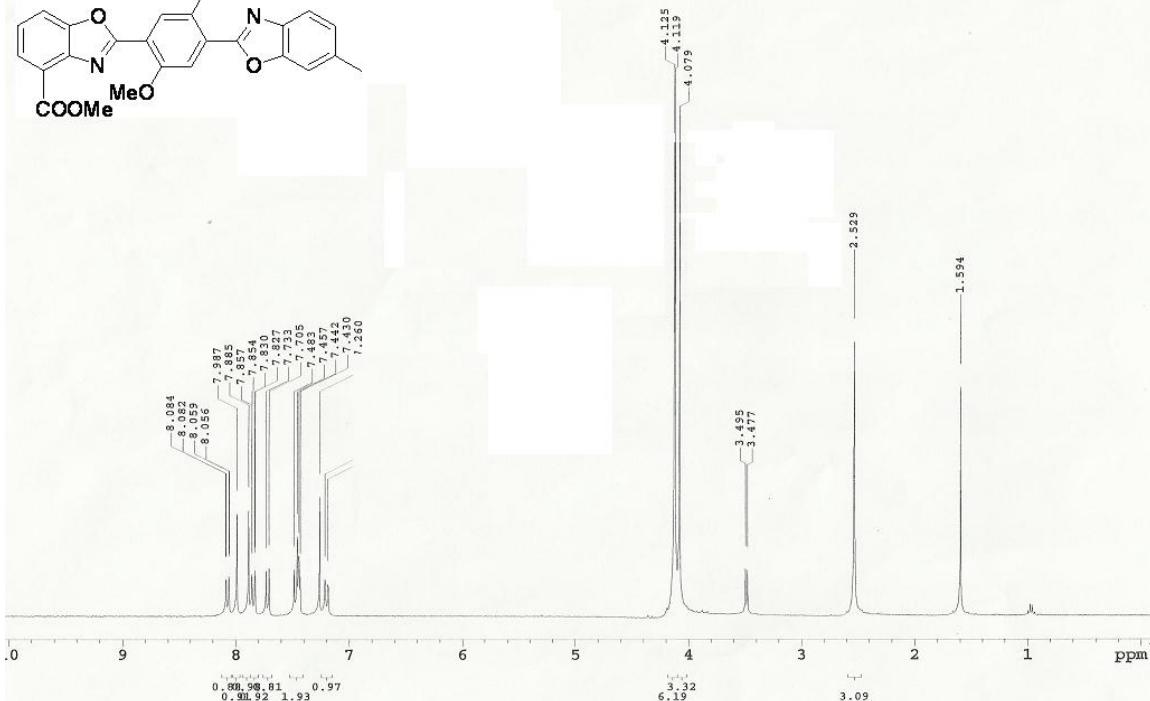
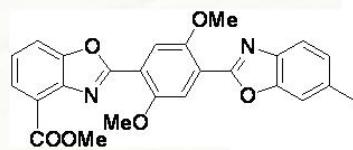
Std Proton parameters
Sample: wf47a
File: home/jwl40/wf47a.fid
Pulse Sequence: s2pul



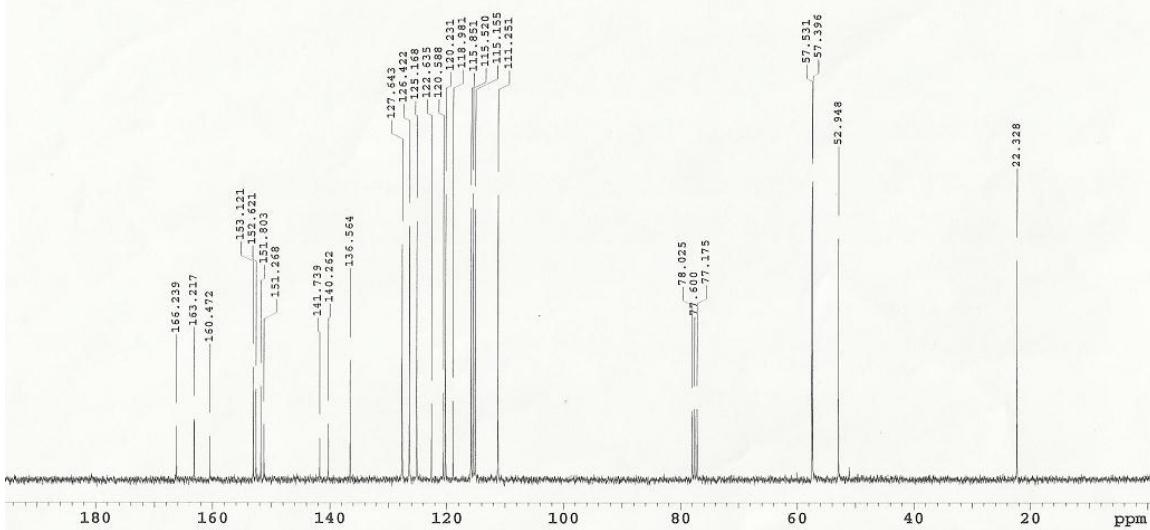
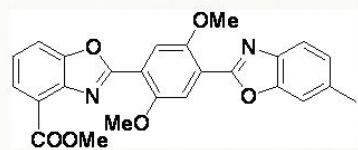
1.0M
Std Carbon experiment
Sample: wf47a
File: xp
Pulse Sequence: s2pul

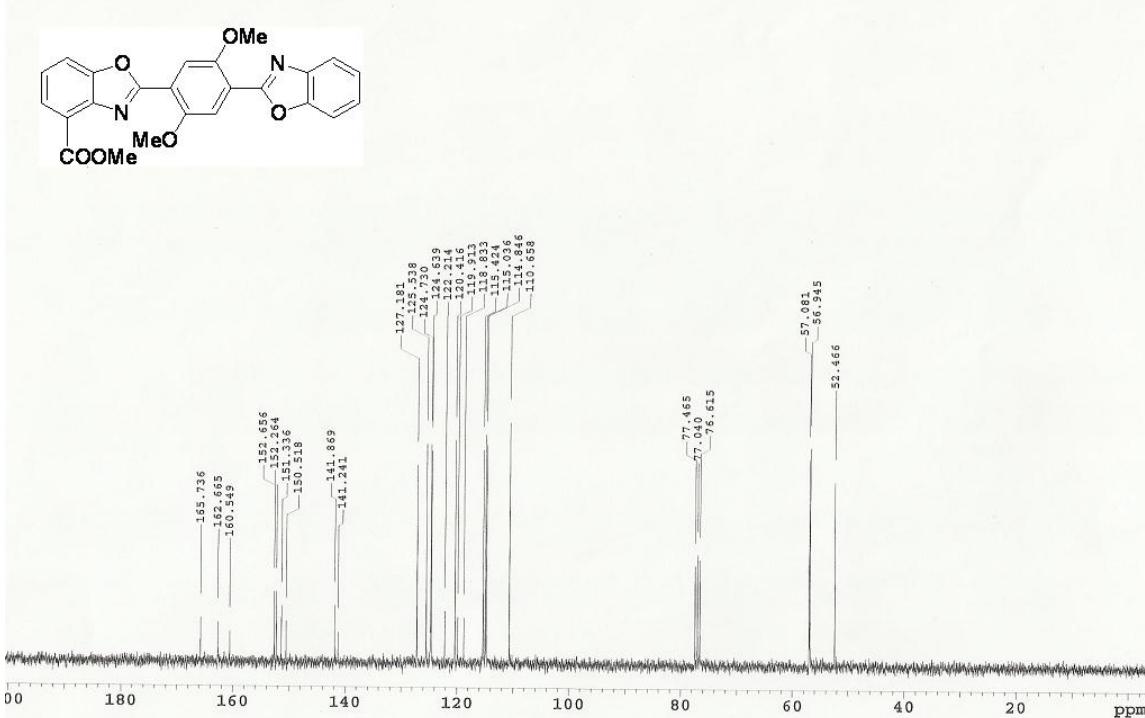
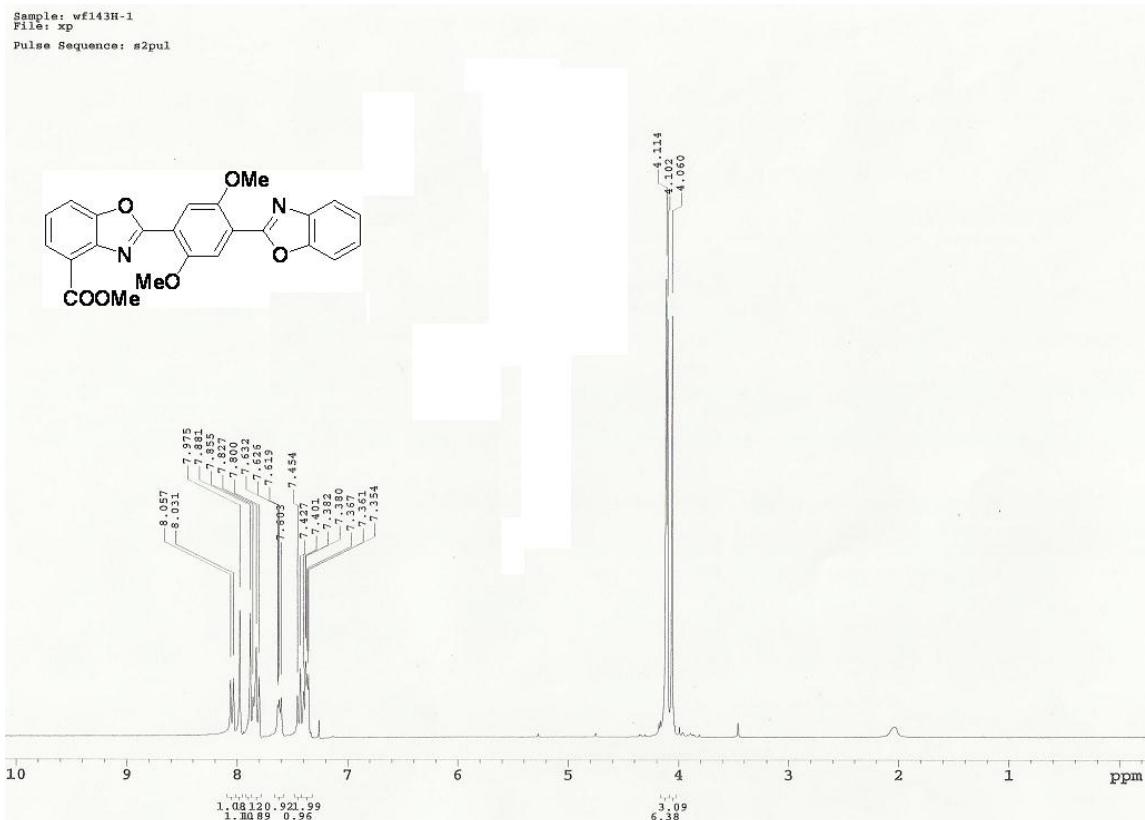


126
Sample: wf126
File: xp
Pulse Sequence: s2pul

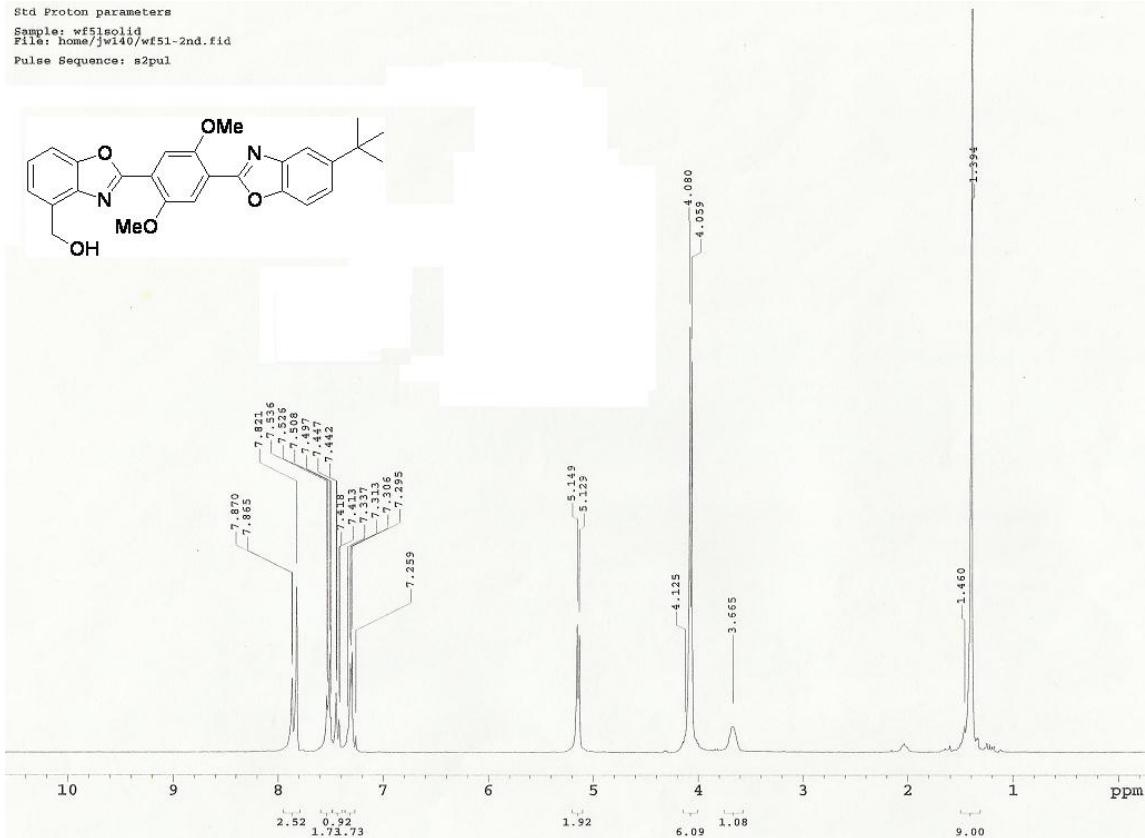
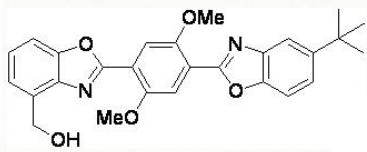


126C
Sample: wf126C
File: xp
Pulse Sequence: s2pul

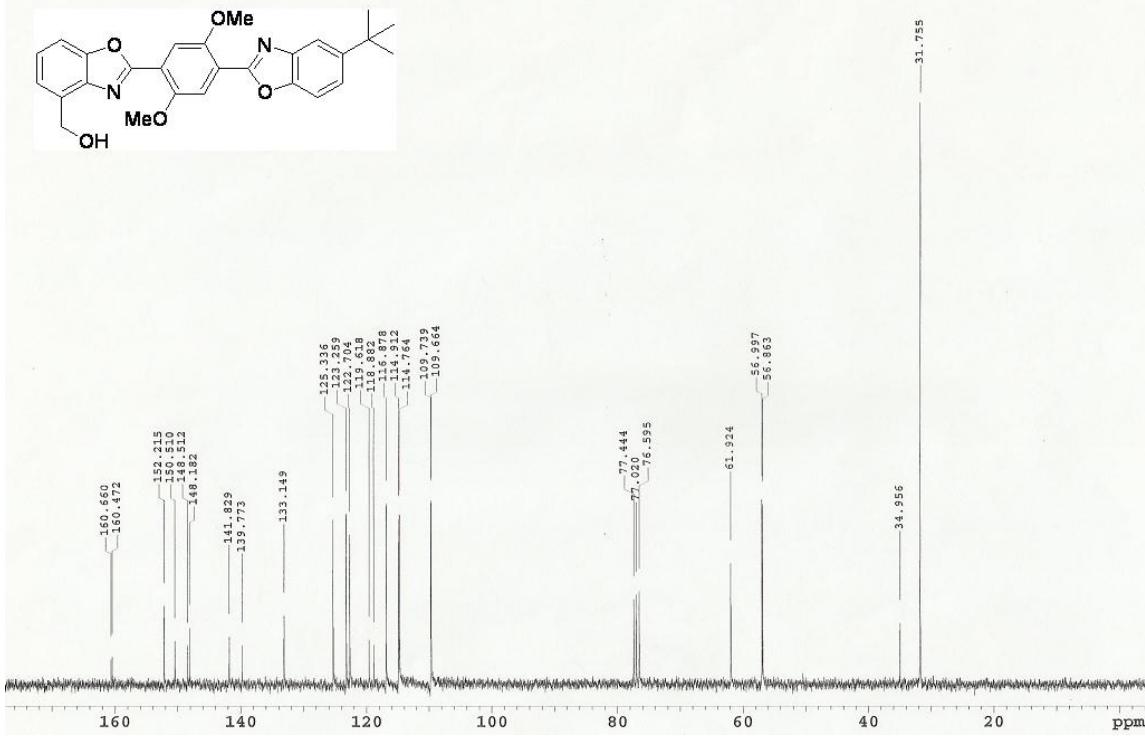
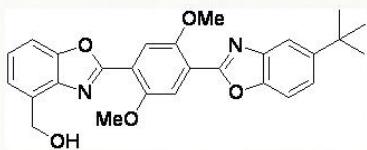


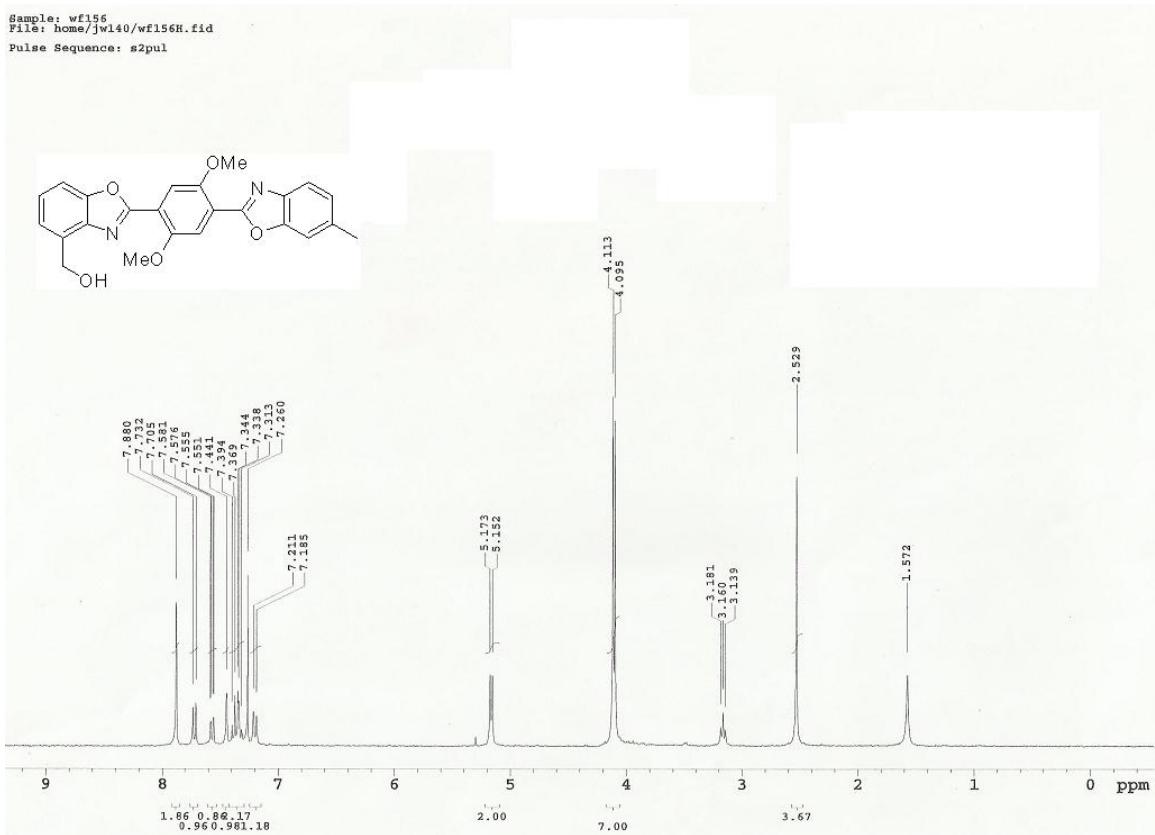


```
Std Proton parameters
Sample: wf51solid
File: home/jwi40/wf51-2nd.fil
Pulse Sequence: s2pul
```

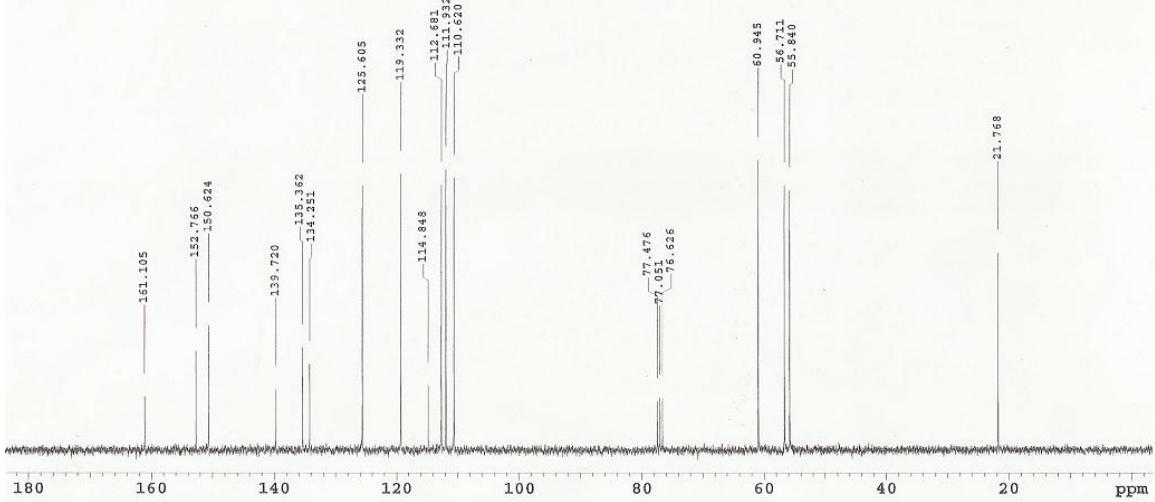
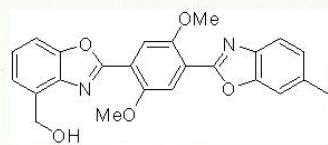


Std Carbon experiment
Sample: wf51solid
File: home/jwl40/wf51-2nd-C.fid
Pulse Sequence: s2pul

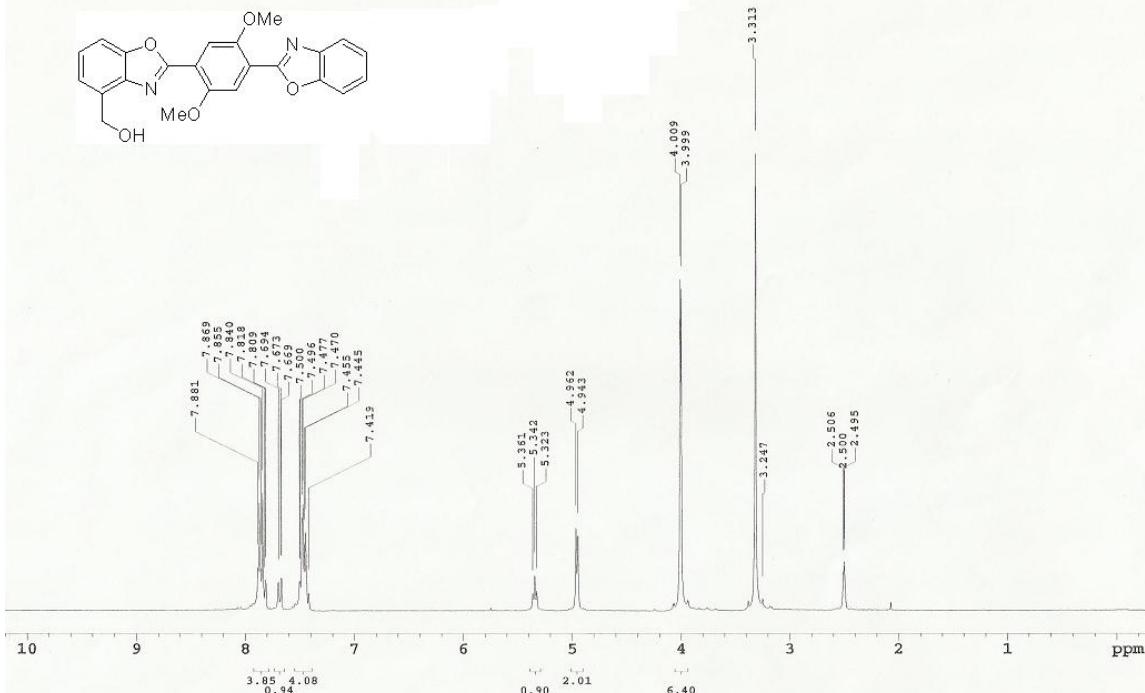




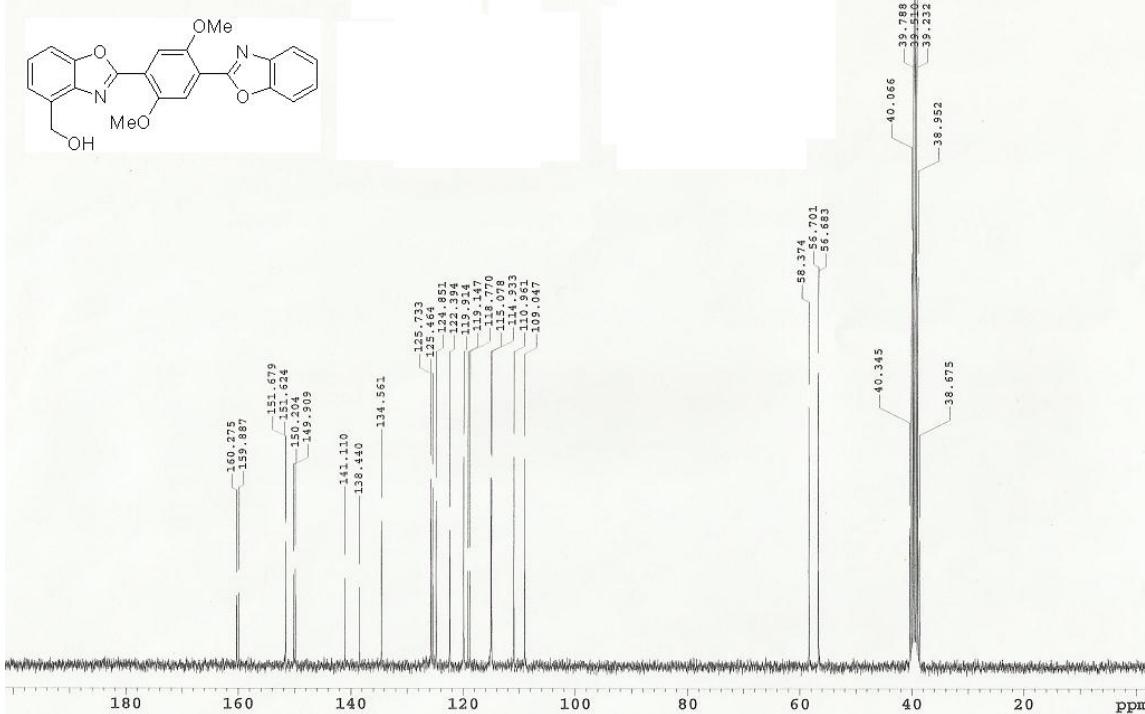
Sample: wf156-2
Sample ID: s_20120811_01
File: home/jwl40/wf156C-2-2.fid
Pulse Sequence: s2pul



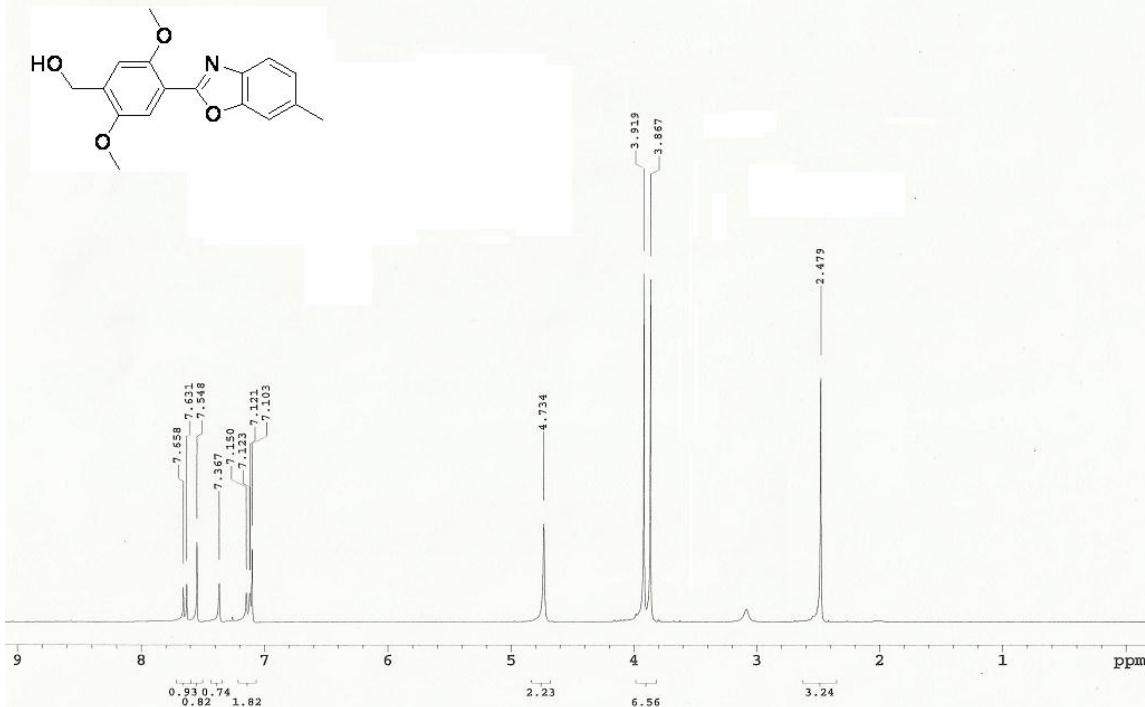
Std Proton parameters
Sample: wf144H2 **DMSO**
File: xp
Pulse Sequence: s2pul



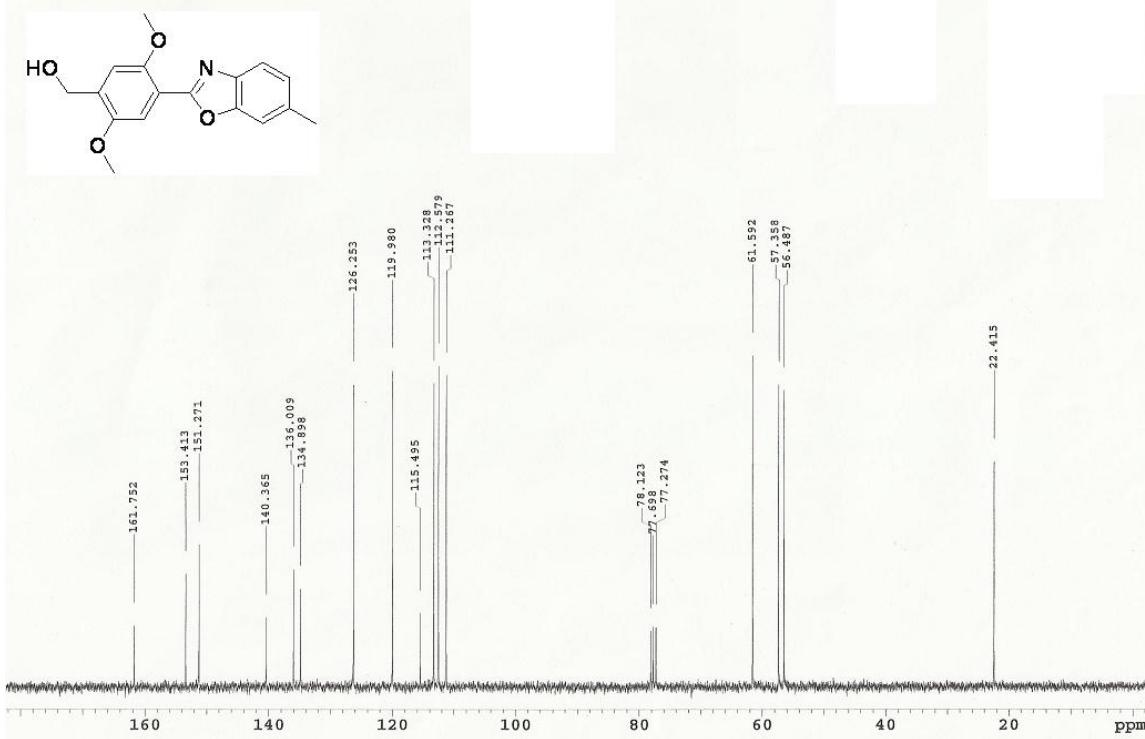
DMSO
Sample: wf144C-2
File: xp
Pulse Sequence: s2pul



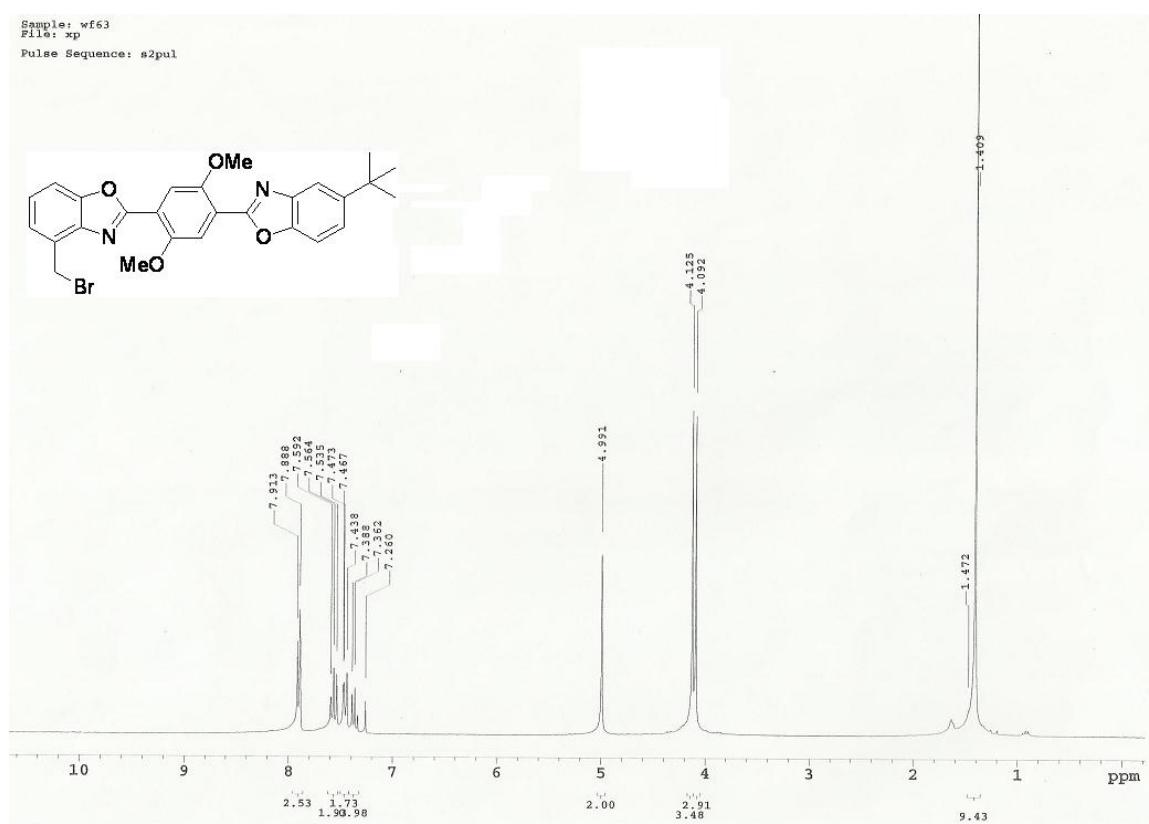
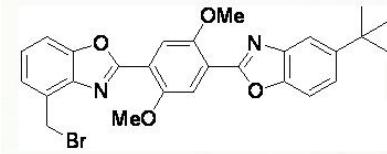
Sample: wf156-2
Sample ID: g_20120811_01
File: home/jwl40/wf156-2-2.fid
Pulse Sequence: s2pul



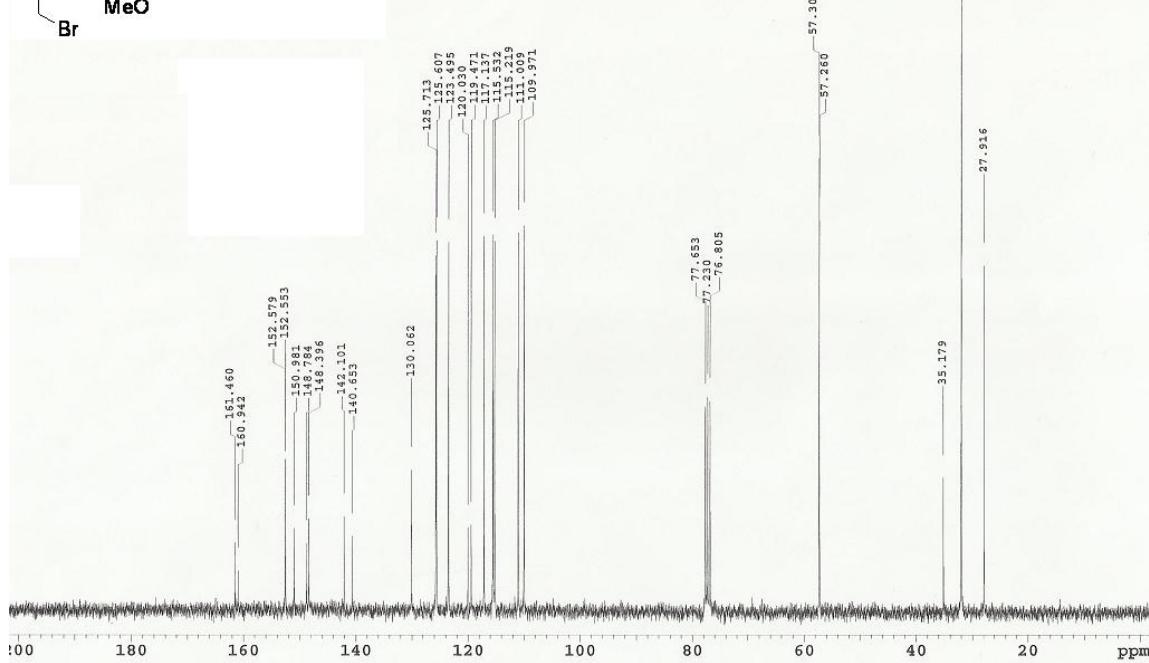
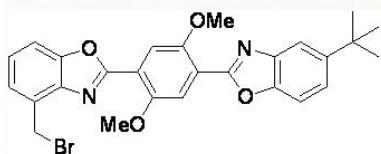
Sample: wf156-2
File: xp
Pulse Sequence: s2pul



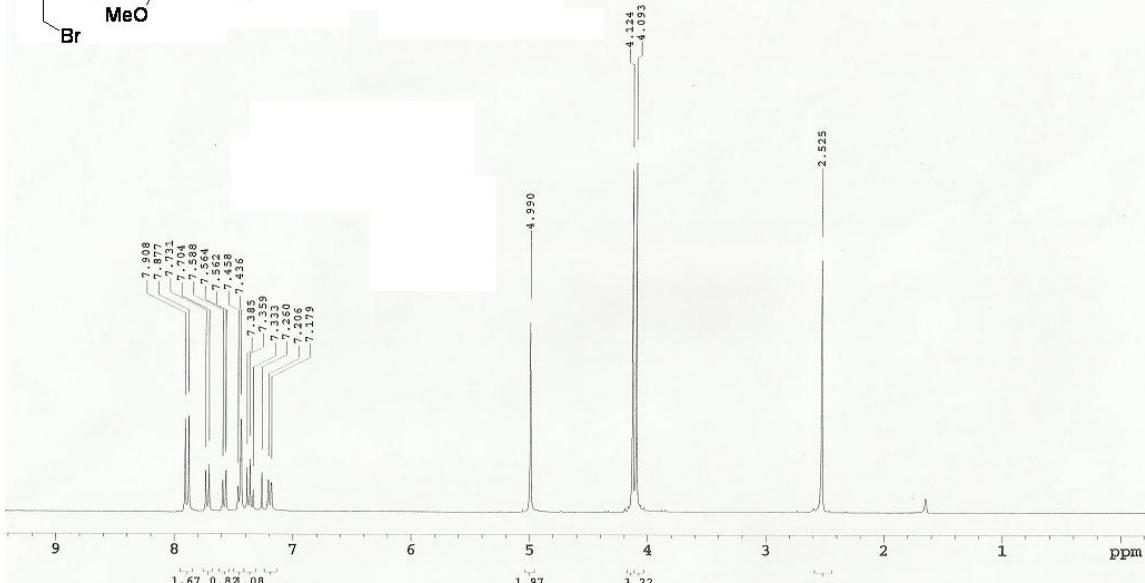
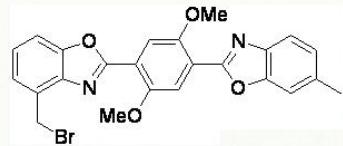
Sample: wf63
File: xp
Pulse Sequence: s2pul



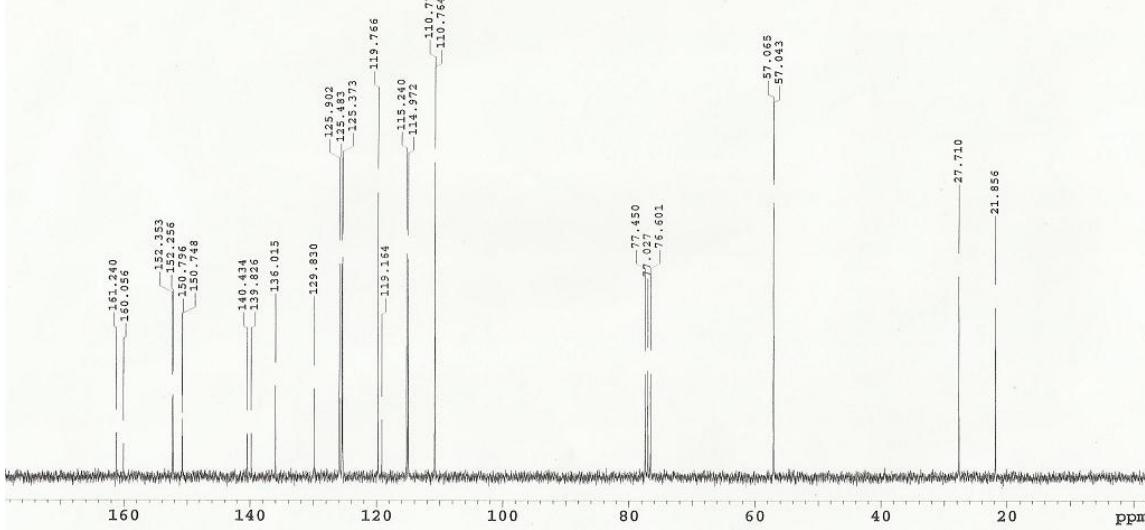
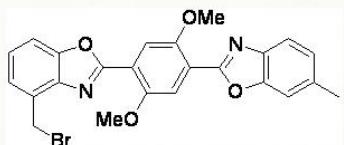
wf63
Sample: wf63H
File: xp
Pulse Sequence: s2pul



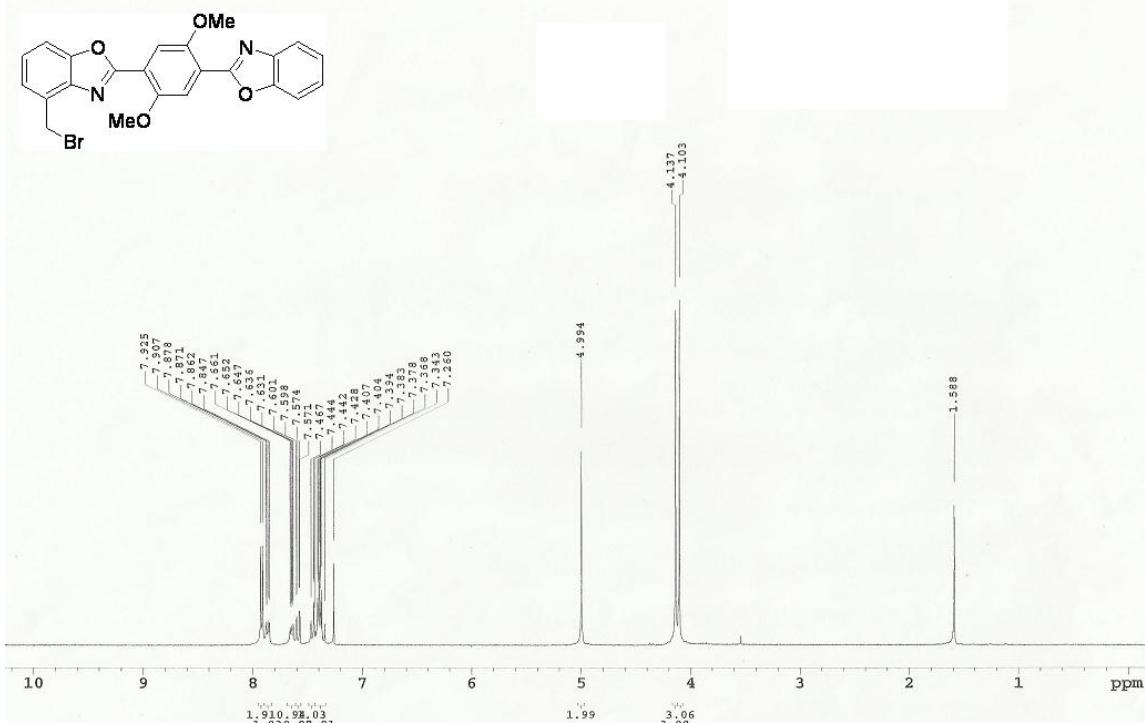
Std Proton parameters
Sample: wf157H
File: home/jwl40/wf157H.fid
Pulse Sequence: s2pul



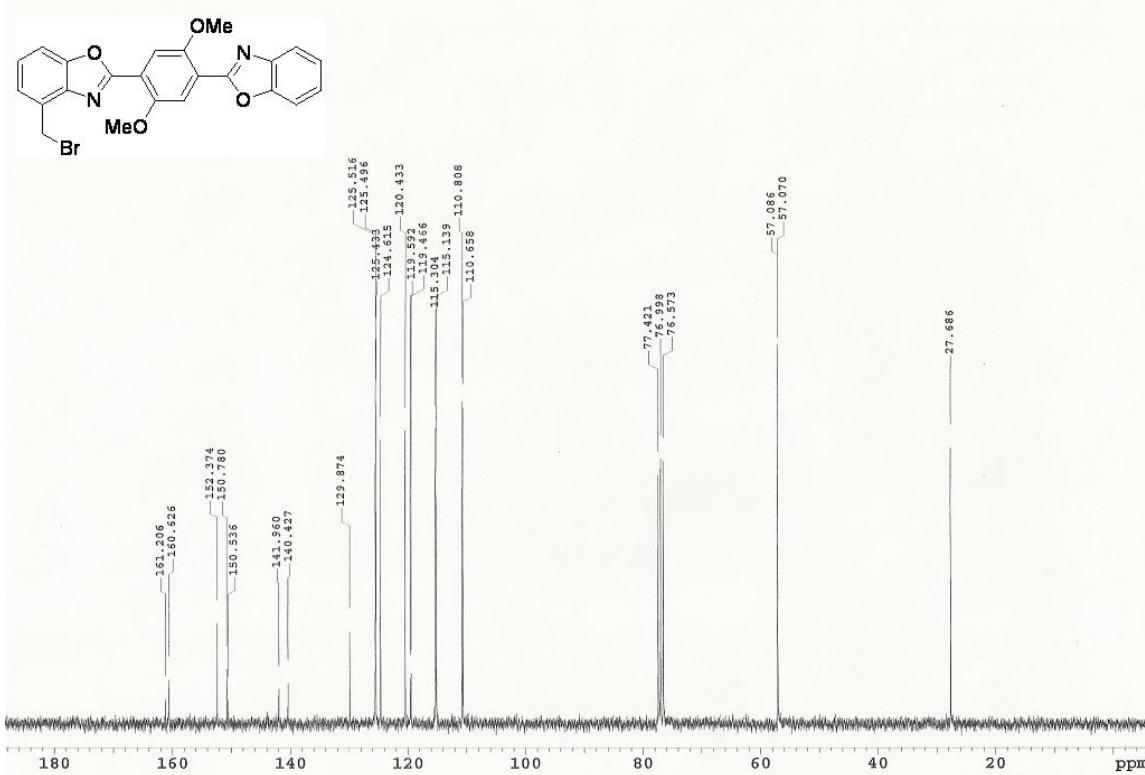
Sample: wf157C
File: xp
Pulse Sequence: s2pul

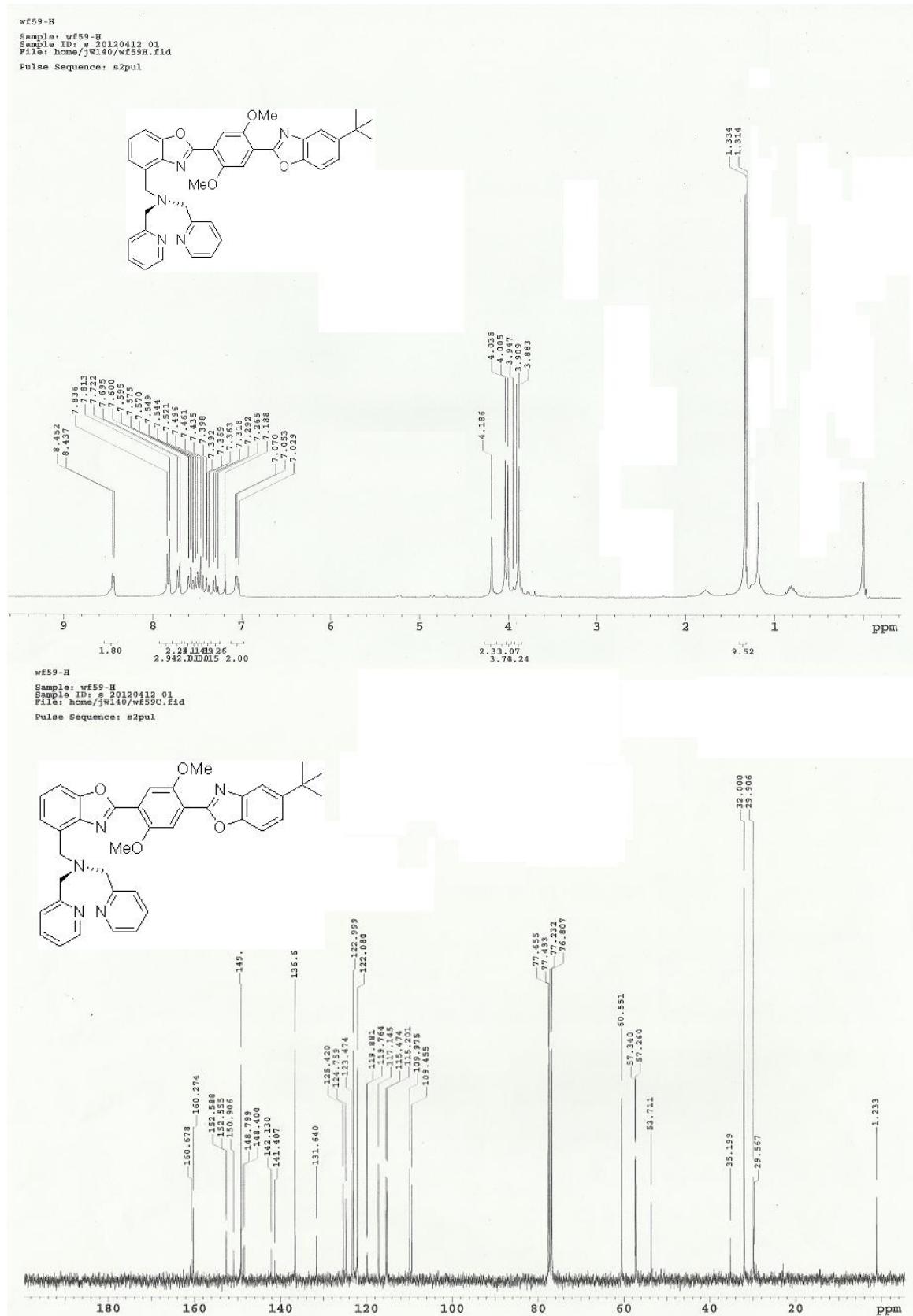


Sample: wf145
Sample ID: s_20120809_01
File: home/jw140/wf145H-1.fid
Pulse Sequence: s2pul

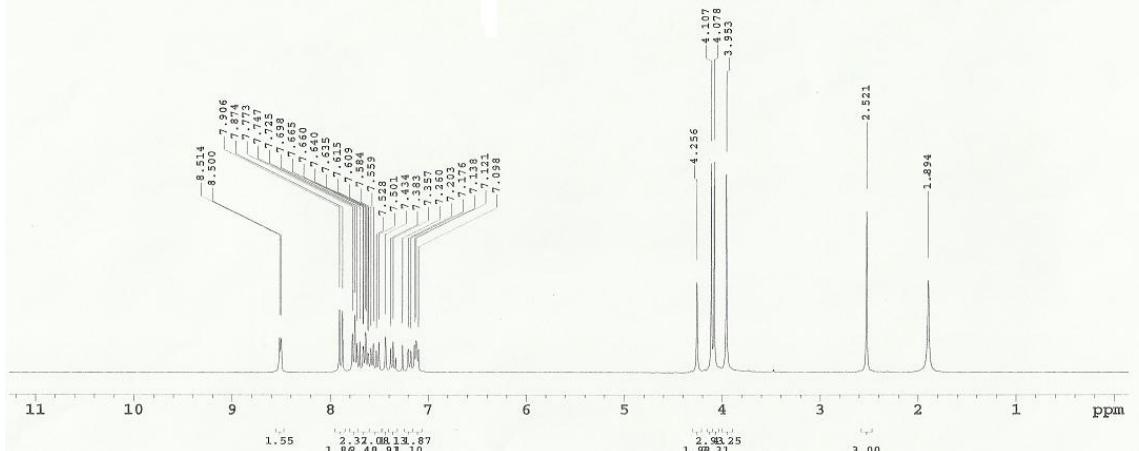
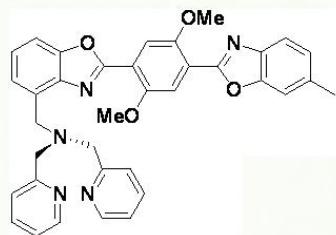


Sample: wf145C
File: xp
Pulse Sequence: s2pul





Sample: wf201H
Sample ID: s 20120913 02
File: home/jw140/wf201H.fid
Pulse Sequence: s2pul



Std Carbon experiment
Sample: wf201C
File: xp
Pulse Sequence: s2pul

