Electronic Supplementary Information (ESI)

Regioselective Desymmetrization of Diaryltetrahydrofurans via Directed *ortho*-Lithiation: An Unexpected Help from Green Chemistry

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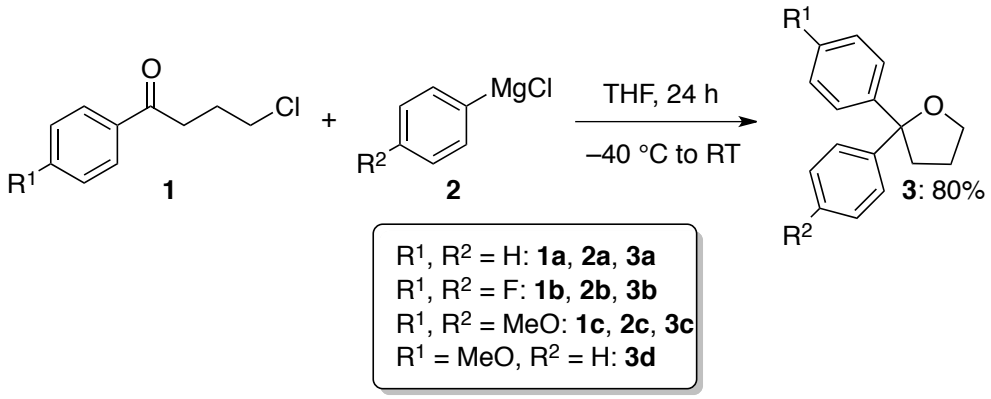
1. General Methods

Tetrahydrofuran (THF), diethyl ether (Et_2O), and toluene were freshly distilled under a nitrogen atmosphere: THF and Et_2O over sodium/benzophenone ketyl, toluene over calcium hydride. Anhydrous cyclopentyl methyl ether (CPME) was used as purchased. Eutectic mixtures of solvents [choline chloride (ChCl)/glycerol (Gly) 1/2; ChCl/urea 1/2; ChCl/water 1/2 (molar ratios)] were prepared by gently heating under stirring at 70 °C for 5 min the corresponding individual components until a clear solution was obtained. For the ^{1}H and ^{13}C NMR spectra (^{1}H NMR 400, 500 or 600 MHz; ^{13}C NMR 100, 125 or 150 MHz), $CDCl_3$ was used as the solvent. GC-MS spectrometry analyses were performed on a gas chromatograph (dimethylsilicon capillary column, 30 m, 0.25 mm i.d.) equipped with a mass selective detector operating at 70 eV (EI). Elemental analyses were performed by using a Carlo Erba CHNS-O EA1108-Elemental Analyzer. Melting points (mp) were determined using an electrothermal melting point apparatus and are uncorrected. Analytical thin layer chromatography (TLC) was carried out on precoated 0.25 mm thick plates of Kieselgel 60 F254; visualization was accomplished by UV light (254 nm) or by spraying with a solution of 5 % (w/v) ammonium molybdate and 0.2 % (w/v) cerium(III) sulfate in 100 ml 17.6 % (w/v) aq. sulphuric acid and heating to 473 K for some time until blue spots appear. All reactions involving air-sensitive reagents were performed under nitrogen in oven-dried glassware using syringe-septum cap technique. Lithiation-electrophilic trapping reactions were performed in an acetone/dry ice bath (–78 °C), or in a salt ice water bath (−20 °C), or in an ice water bath (0 °C). Spectroscopic data of compounds **3a**[1] and **5a**[2] have been reported.

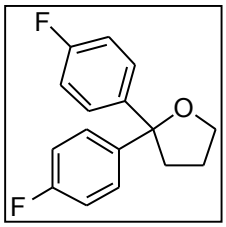
[1] E. D. Butova, A. V. Barabash, A. A. Petrova, C. M. Kleiner, P. R. Schreiner and A. A. Fokin, *J. Org. Chem.* 2010, **75**, 6229.
[2] X.-J. Wei, D.-T. Yang, L. Wang, T. Song, L.-Z. Wu and Q. Liu, *Org. Lett.* 2013, **15**, 6054.

2. Experimental procedures and characterization data

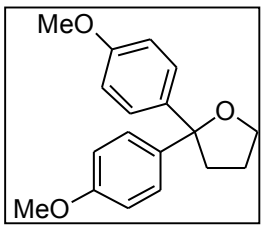
2.1 Preparation of diaryltetrahydrofurans 3a–d. General Procedure.



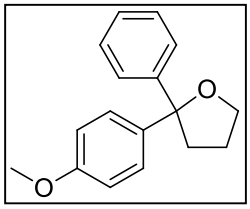
To a THF solution (60 mL) of the commercially available Grignard reagent **2** (**2a**, or **2b**, or **2c**) (30 mmol, 0.5–2 M in Et_2O or THF), previously cooled to –40 °C, a THF solution (10 mL) of the ketone **1** (**1a**, or **1b**, or **1c**) (10 mmol), was added dropwise. The reaction mixture was allowed to warm to room temperature and after 24 h was extracted with Et_2O (3 × 40 mL). The combined organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The crude product was purified by flash-chromatography (silica gel, hexane/AcOEt 95:5, Et_3N 2%) to give diaryltetrahydrofuran **3** (**3a**, or **3b**, or **3c**, or **3d**).



2,2-Bis(4-fluorophenyl)tetrahydrofuran (3b): colourless oil, 80%. ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.93−2.00 (m, 2 H), 2.50−2.54 (m, 2H), 4.03−4.06 (m, 2 H), 6.96−7.01 (m, 4 H), 7.36−7.40 (m, 4 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.4, 38.8, 67.4, 87.2, 114.9 (d, $^2J_{C\text{-}F}$ = 21.2 Hz), 127.4 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 141.9 (d, $^4J_{C\text{-}F}$ = 3.3 Hz), 161.6 (d, $^1J_{C\text{-}F}$ = 240 Hz); FT-IR (film, cm^{-1}) 3055, 2959, 1598, 1506, 1266, 743; GC-MS (70eV), m/z (%): 260 (3), 219 (100), 123 (60). Anal. Calcd. for $C_{16}H_{14}F_2O$: C, 73.83; H, 5.42; Found: C, 74.02; H, 5.55.

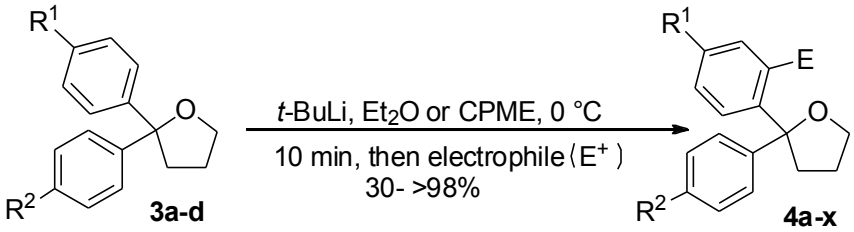


2,2-Bis(4-methoxyphenyl)tetrahydrofuran (3c): colourless oil, 80%, ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.92−1.97 (m, 2 H), 2.48−2.51 (m, 2 H), 3.77 (s, 6 H), 4.01−4.03 (m, 2 H), 6.82−6.83 (m, 4 H), 7.31−7.32 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 25.5, 38.7, 55.2, 67.2, 86.0, 87.4, 110.0, 113.4, 127.0, 138.7, 158.2; FT-IR (film, cm^{-1}) 2954, 1609, 1509, 1246, 1174, 1034, 828, 736; GC-MS (70eV), m/z (%): 284 (M^+, 51), 269 (1), 253 (27), 223 (6), 177 (100), 135 (55). Anal. Calcd. for $C_{18}H_{20}O_3$: C, 76.03; H, 7.09; Found: C, 76.24; H, 7.22.



2-(4-Methoxyphenyl)-2-phenyltetrahydrofuran (3d): colourless oil, 80%, ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.91−1.98 (m, 2 H), 2.50−2.57 (m, 2 H), 3.76 (s, 3 H), 4.02−4.06 (m, 2 H), 6.81−6.84 (m, 2 H), 7.16−7.20 (m, 1 H), 7.27−7.36 (m, 4 H), 7.41−7.42 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.5, 38.7, 55.5, 67.3, 87.8, 113.5, 125.8, 126.6, 127.1, 128.1, 138.5, 146.7, 158.4; FT-IR (film, cm^{-1}) 3059, 2953, 2876, 1610, 1509, 1250, 1055, 829; GC-MS (70eV), m/z (%): 254 (M^+, 25), 223 (10), 177 (100), 135 (50). Anal. Calcd. for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13; Found: C, 80.51; H, 7.42.

2.2 Directed *ortho*-lithiation/functionalization of diaryltetrahydrofuran derivatives 3a–d in Et_2O or cyclopentyl methyl ether (CPME). General Procedure.



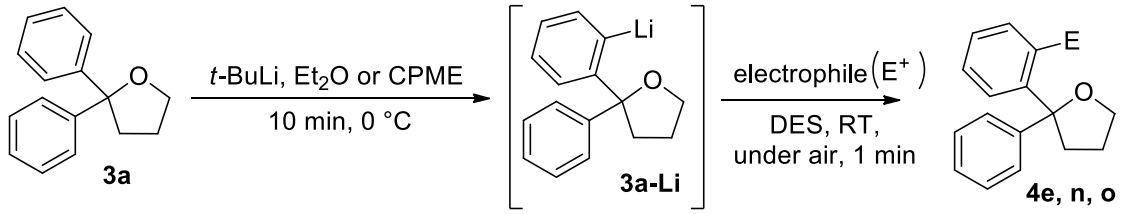
An Et_2O (or CPME) solution of **3** (1 mmol in 2 mL of solvent) was cooled to 0 °C and treated with *t*-BuLi (1.9 mmol, 1.12 mL of a solution 1.7 M in pentane) under N_2. The color of the mixture became dark orange. After stirring for 10 min, the electrophile (2.0 mmol),[3] as pure liquid or as a solution in 1 mL of the solvent if solid, was added all at once and the mixture was stirred for an additional 30 min at 0 °C. After this time, 5 mL of water was added and the reaction mixture was allowed to warm to room temperature and finally extracted with Et_2O (3 × 10 mL). Once CPME was used as the solvent, the organic phase was simply diluted with water and separated without further extraction. This is because of the high hydrophobicity and limited miscibility of CPME with water.[4] The (combined) organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The crude was purified by flash-chromatography (silica gel; hexane/AcOEt 8:2 ÷ 95:5) to give products **4a–x**.

In the case of acetone as the electrophile, once an Et_2O solution (2 mL) of the *ortho*-lithiated intermediate **3a–Li** (1 mmol), prepared as above described, was spread out by a syringe over an acetone-water mixture (6 mmol each) or over acetone alone (6 mmol) (neat conditions) at room temperature, the desired adduct **4n** could be isolated in 30% yield in either case (see also Table 2 in the main text).

[3] 3 mmol in the case of acetaldehyde.

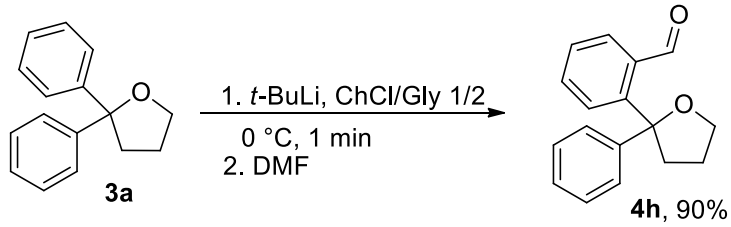
[4] (*a*) K. Watanabe, N. Yamagiwa and Y. Torisawa, *Org. Process Res. Dev.*, 2007, **11**, 251; (*b*) A. Kadam, M. Nguyen, M. Kopach, P. Richardson, F. Gallou, Z.-K. Wan and W. Zhang, *Green Chem.* 2013, **15**, 1880.

2.3 Directed *ortho*-lithiation of diphenyltetrahydrofuran 3a in Et_2O or cyclopentyl methyl ether (CPME) followed by trapping with electrophiles in Deep Eutectic Solvents (DESs). General Procedure.



An Et_2O (or CPME) solution of **3a** (1 mmol in 2 mL of solvent) was cooled to 0 °C and treated with *t*-BuLi (1.9 mmol, 1.12 mL of a solution 1.7 M in pentane) under N_2. After stirring for 10 min, the solution of the corresponding anion was transferred by a syringe, at room temperature and under air, to a round-bottomed flask containing a stirring solution of the electrophile in the selected DES [ChCl/Gly 1/2, ChCl/urea 1/2; ChCl/water 1/2 (molar ratios)) (2 g) (see also Table 3 in the main text), prepared as reported in the General Methods. After 1 min, the reaction mixture so obtained was diluted with 10 mL of water and extracted with Et_2O (3 × 10 mL). The combined organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The crude was purified by flash-chromatography (silica gel; hexane/AcOEt 8:2 ÷ 95:5) to give product **4e**, or **4n**, or **4o**.

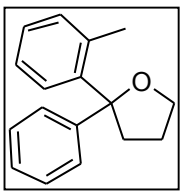
2.4 Directed *ortho*-lithiation of diphenyltetrahydrofuran 3a in the eutectic solvent ChCl/Gly 1/2 followed by quenching with DMF.



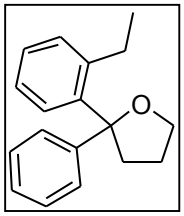
A CPME solution of the substrate **3a** (1 mmol in 2 mL of solvent) was added to the DES ChCl/Gly 1/2 mixture (molar ratio) (2 g).[5] The corresponding clean solution so obtained was cooled to 0 °C and was treated with *t*-BuLi (1.9 mmol, 1.12 mL of a solution 1.7 M in pentane) under air. After stirring for 1 min, the reaction mixture was quenched with neat DMF (2 mmol), diluted with 10 ml of water, and finally extracted with Et_2O (3 × 10 mL). The combined organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The crude was purified by flash-chromatography (silica gel; hexane/AcOEt 8:2) to give the compound **4h** in 90% yield.

[5] A preliminary solubilization of the substrate **3a** in CPME was necessary as it tends to precipitate from the DES mixture once the latter was cooled to 0 °C.

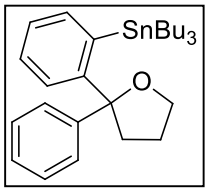
2.5 Characterization data of compounds 4a–x



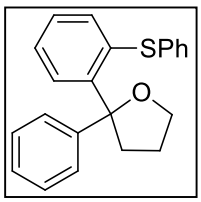
2-Phenyl-2-(*o*-tolyl)-tetrahydrofuran (4a): colourless oil; >98% (Et_2O and CPME). ^{1}H NMR (500 MHz; $CDCl_3$): δ 2.01−2.08 ppm (m, 2 H), 2.11 (s, 3 H), 2.55−2.65 (m, 2 H), 3.96−4.00 (m, 1 H), 4.13−4.18 (m, 1 H), 7.11−7.13 (m, 1 H), 7.20−7.34 (m, 7 H), 7.70−7.71 (m, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 23.2, 26.1, 38.9, 67.8, 88.4, 125.7, 126.4, 127.1, 127.2, 127.8, 128.7, 141.2, 142.9, 146.5; FT-IR (film, cm^{-1}) 3059, 2972, 2874, 1488, 1447, 1050, 753 cm^{-1}; GC-MS (70eV), m/z (%): 238 (M^+, 10), 223 (10), 195 (70), 179 (87), 161 (88), 147 (100), 105 (38). Anal. Calcd. for $C_{17}H_{18}O$: C, 85.67; H, 7.61; Found: C, 85.85; H, 7.72.



2-(2-Ethylphenyl)-2-phenyltetrahydrofuran (4b): colourless oil; 80% (CPME). ^{1}H NMR (500 MHz; $CDCl_3$): δ 0.84 (t, J = 7.4 Hz, 3 H), 2.00–2.08 (m, 2 H), 2.48–2.64 (m, 4 H), 3.97–4.02 (m, 1 H), 4.17–4.21 (m, 1 H), 7.21–7.50 (m, 6 H), 7.63–7.65 (m, 1 H), 7.69–7.71 (m, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.2, 25.8, 26.2, 39.0, 67.9, 88.5, 125.0, 125.8, 126.1, 126.5, 127.9, 128.8, 130.4, 142.9, 143.1, 146.6; FT-IR (film, cm^{-1}) 3055, 2979, 1481, 1265, 1049, 738; ESI-MS: 275 [M^+ + 23 (Na)]. Anal. Calcd. for $C_{18}H_{20}O$: C, 85.67; H, 7.99; Found: C, 85.85; H, 8.11.

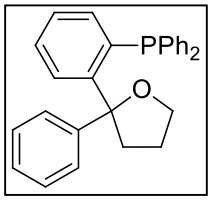


2-(2-Tributylstannylphenyl)-2-phenyltetrahydrofuran (4c): colourless oil, 90% (Et_2O). ^{1}H NMR (500 MHz; $CDCl_3$): δ 0.94 (t, J = 7.0 Hz, 9 H), 1.33−1.57 (m, 18 H), 2.03−2.07 (m, 2 H), 2.30−2.36 (m, 1 H), 2.75−2.80 (m, 1 H), 3.91−3.95 (m, 1 H), 4.02−4.06 (m, 1 H), 7.24−7.34 (m, 6 H), 7.40−7.42 (m, 2 H), 7.60−7.62 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 11.9, 13.7, 25.0, 27.6, 29.2, 39.9, 66.9, 89.2, 125.8, 126.6, 126.8, 127.4, 127.5, 127.9, 137.4, 138.2, 146.0, 151.5; FT-IR (film, cm^{-1}) 2955, 2921, 1463, 1050, 699 cm^{-1}; GC-MS (70eV), m/z (%): 457 (M−57, 100), 343 (13), 311 (16), 193 (20). Anal. Calcd. for $C_{28}H_{42}OSn$: C, 65.51; H, 8.25; Found: C, 65.76; H, 8.40.

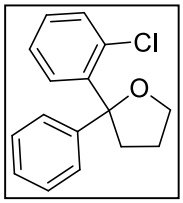


2-(2-Phenylthiophenyl)-2-phenyltetrahydrofuran (4d): colourless oil, 85% (Et_2O). ^{1}H NMR (600 MHz; $CDCl_3$): δ 2.05−2.20 (m, 2 H), 2.70−2.76 (m, 1 H), 3.09−3.15 (m, 1 H), 4.09−4.16 (m, 2 H), 7.11−7.13 (m, 2 H), 7.20−7.28 (m, 6 H), 7.32−7.36 (m, 3 H), 7.48−7.50 (m, 2 H), 7.92−7.94 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.4, 37.1, 67.3, 88.3, 126.5, 126.6, 126.7, 126.9, 127.7, 128.8, 130.9, 134.1, 134.3, 137.2, 144.4, 146.6; FT-IR (film, cm^{-1}) 3057, 2978, 1265, 738; GC-MS (70eV), m/z (%): 332 (M^+,

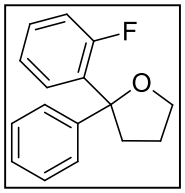
100), 255 (44), 213 (35), 147 (40), 105 (32). Anal. Calcd. for $C_{22}H_{20}OS$: C, 79.48; H, 6.06; Found: C, 79.69; H, 6.16.



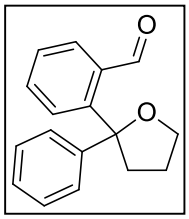
(2-(2-Phenyltetrahydrofuran-2-yl)phenyl)diphenylphosphine (4e): colourless oil. 60% (Et_2O), 85% (CPME), 75% (ChCl/urea 1/2). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.72−1.81 (m, 1 H), 2.02−2.09 (m, 1 H), 2.62−2.68 (m, 1 H), 2.90−2.94 (m, 1 H), 3.39−3.44 (m, 1 H), 4.13−4.19 (m, 1 H), 7.12−7.65 (m, 17 H), 7.85−7.90 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 39.3, 66.2, 89.1, 125.9, 126.9, 127.0, 127.8, 127.9, 128.0, 128.1, 128.5, 129.5, 130.4, 130.5, 130.6, 131.2, 131.6, 132.0, 132.1, 136.0, 136.2, 136.5, 136.8, 137.4, 144.8, 152.0; FT-IR (film, cm^{-1}) 3042, 3058, 2979, 2220, 1438, 1221, 1188, 1131, 1037, 909, 733, 697, 545; GC-MS (70eV), m/z (%): 408 (M^+, 38), 380 (64), 351 (50), 303 (100), 289 (27), 183 (39). 105 (9). Anal. Calcd. for $C_{28}H_{25}OP$: C, 82.33; H, 6.17; Found: C, 82.53; H, 6.40.



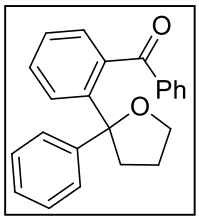
2-(2-Chlorophenyl)-2-phenyltetrahydrofuran (4f): white waxy solid, 90% (Et_2O). ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.95−2.03 (m, 1 H), 2.07−2.14 (m, 1 H), 2.55−2.60 (m, 1 H), 3.03−3.07 (m, 1 H), 3.96−4.05 (m, 2 H), 7.20−7.23 (m, 2 H), 7.27−7.33 (m, 6 H), 7.92−7.93 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 26.6, 36.3, 67.6, 87.6, 126.5, 126.8, 127.1, 127.6, 127.9, 128.4, 131.0, 132.0, 143.6, 143.8; FT-IR (film, cm^{-1}) 2918, 1492, 1459, 758, 697; GC-MS (70eV), m/z (%): 258 (M^+, 22), 223 (28), 181 (80), 147 (100), 105 (57). Anal. Calcd. for $C_{16}H_{15}ClO$: C, 74.27; H, 5.84; Found: C, 74.59; H, 6.02.



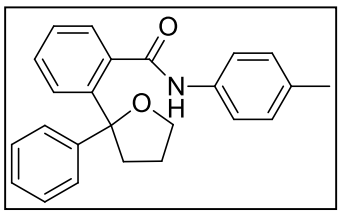
2-(2-Fluorophenyl)-2-phenyltetrahydrofuran (4g): colourless oil, 85% (Et_2O). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.92−2.04 (m, 2 H), 2.61−2.65 (m, 1 H), 2.77−2.82 (m, 1H), 4.02−4.08 (m, 2 H), 6.95−6.98 (m, 1 H), 7.12−7.15 (m, 1 H), 7.20−7.23 (m, 2 H), 7.29−7.32 (m, 2 H), 7.48−7.49 (m, 2 H), 7.70−7.73 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 25.6, 37.2 (d, $^4J_{C\text{-}F}$ = 3.7 Hz), 67.2, 85.7 (d, $^3J_{C\text{-}F}$ = 2.5 Hz), 116.0 (d, $^2J_{C\text{-}F}$ = 18.7 Hz), 123.8 (d, $^4J_{C\text{-}F}$ = 2.5 Hz), 125.9, 126.9, 127.5 (d, $^3J_{C\text{-}F}$ = 3.7 Hz), 128.0, 128.7 (d, $^3J_{C\text{-}F}$ = 7.5 Hz), 133.5 (d, $^2J_{C\text{-}F}$ = 10 Hz), 144.9, 159. 3 (d, $^1J_{C\text{-}F}$ = 203.7 Hz); FT-IR (film, cm^{-1}) 2978, 2886, 2251, 1483, 1449, 1214, 1052, 910, 735; GC-MS (70eV), m/z (%): 242 (M+, 45), 223 (1), 165 (100), 147 (45), 123 (65), 105 (55). Anal. Calcd. for $C_{16}H_{15}FO$: C, 79.32; H, 6.24; Found: C, 79.44; H, 6.49.



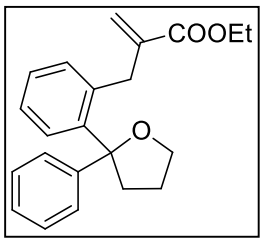
2-(2-Phenyltetrahydrofuran-2-yl)benzaldehyde (4h): colourless oil 90% (Et_2O), 90% (ChCl/Gly 1/2). ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.98−2.02 (m, 2 H), 2.51−2.53 (m, 1 H), 2.69−2.73 (m, 1 H), 3.96−3.98 (m, 1 H), 4.22−4.23 (m, 1 H), 7.22−7.42 (m, 6 H), 7.58−7.60 (m, 1 H), 7.67−7.68 (m, 1 H), 7.86−7.87 (m, 1 H), 10.43 (s, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.2, 40.5, 68.2, 88.4, 125.4, 126.3, 127.0, 127.7, 128.3, 128.7, 132.4, 135.0, 146.3, 147.1, 193.7; FT-IR (film, cm^{-1}) 3054, 2985, 2305, 1691, 1265, 740, 705; GC-MS (70eV), m/z (%): 252 (M^+, 57), 234 (50), 207 (100), 105 (58). Anal. Calcd. for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39; Found: C, 81.15; H, 6.56.



[2-(2-Phenyltetrahydrofuran-2-yl)phenyl]phenylmethanone (4i): colourless oil, 90% (Et_2O). ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.79−1.94 (m, 2 H), 2.52−2.56 (m, 1 H), 2.66−2.71 (m, 1 H), 3.58−3.68 (m, 2 H), 7.16−7.24 (m, 5 H), 7.30−7.39 (m, 6 H), 7.49−7.52 (m, 1 H), 7.72−7.73 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.8, 40.0, 67.1, 88.4, 126.2, 126.7, 127.0, 127.3, 127.9, 128.4, 129.3, 132.1, 137.7, 138.3, 145.1, 145.3, 197.6; FT-IR (film, cm^{-1}) 3055, 2987, 2306, 1266, 740; GC-MS (70eV), m/z (%): 328 (M^+, 33), 270 (50), 251 (50), 209 (94), 147 (100), 105 (57). Anal. Calcd. for $C_{23}H_{20}O_2$: C, 84.12; H, 6.14; Found: C, 84.43; H, 6.21.

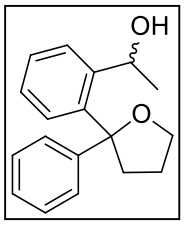


2-(2-Phenyltetrahydrofuran-2-yl)-*N*-(*p*-tolyl)benzamide (4j): colourless oil, 70% (Et_2O). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.96−2.02 (m, 2 H), 2.31 (s, 3 H), 2.43−2.49 (m, 1 H), 2.75−2.80 (m, 1 H), 3.97−4.02 (m, 1H), 4.05−4.09 (m, 1 H), 7.06−7.08 (m, 2 H), 7.13−7.15 (m, 2 H), 7.19−7.25 (m, 2 H), 7.30−7.37 (m, 4 H), 7.43−7.46 (m, 1 H), 7.51−7.53 (m, 1 H), 7.60−7.61 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 20.9, 25.9, 39.7, 68.1, 88.2, 119.6, 126.0, 126.9, 127.1, 127.6, 128.3, 129.2, 129.8, 133.4, 135.6, 145.6; FT-IR (film, cm^{-1}) 3410, 3306, 3054, 2305, 1670, 1515, 1318, 1265, 1048, 740; GC-MS (70eV), m/z (%): 357 (M^+, 6), 251 (100), 209 (28). Anal. Calcd. for $C_{24}H_{23}NO_2$: C, 80.64; H, 6.49; Found: C, 80.88; H, 6.62.

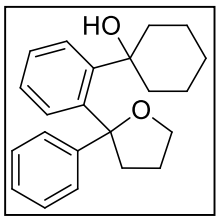


Ethyl 2-(2-(2-Phenyltetrahydrofuran-2-yl)benzyl) acrylate (4k): colourless oil, 70% (Et_2O). ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.20 (t, J = 7.0 Hz 3 H), 1.91−1.98 (m, 2 H), 2.49−2.61 (m, 2 H), 3.49−3.61 (m, 2 H), 3.87−3.93 (m, 1 H), 4.04−4.14 (m, 3 H), 4.79 (s, 1 H), 5.92 (s, 1 H), 7.00−7.03 (m, 1 H), 7.10−7.14 (m, 1 H), 7.17−7.27 (m, 6 H), 7.68−7.70 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 14.1, 25.5, 35.0, 38.5, 60.4, 67.7, 88.3, 125.3, 125.8, 126.1, 126.4, 127.2, 127.8,

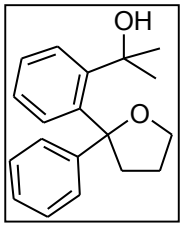
131.7, 137.1, 140.1, 143.8, 145.7, 167.2; FT-IR (film, cm^{-1}) 3048, 2982, 2878, 1715, 1447, 1266, 1048, 738; GC-MS (70eV), m/z (%): 336 (M^+, 19), 277 (40), 234 (21), 209 (33), 147 (100), 105 (79). Anal. Calcd. for $C_{22}H_{24}O_3$: C, 78.54; H, 7.19; Found: C, 78.77; H, 7.32.



1-(2-(2-Phenyltetrahydrofuran-2-yl)phenyl)ethanol (4l): colourless oil, 70% overall yield (Et_2O), (separable mixture of diastereomers, dr 60/40). Minor diastereomer. ^1H NMR (600 MHz; $CDCl_3$): δ 1.29 (d, J = 6.5 Hz, 3 H), 1.99−2.03 (m, 2 H), 2.42−2.47 (m, 1 H), 2.69−2.74 (m, 1 H), 3.94−3.98 (m, 1 H), 4.18−4.21 (m, 1 H), 5.22 (q, J = 6.5 Hz, 1 H), 7.19−7.21 (m, 1 H), 7.28−7.32 (m, 5 H), 7.36−7.38 (m, 1 H), 7.56−7.58 (m, 1 H), 7.60−7.62 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 24.1, 25.3, 39.5, 66.2, 68.0, 88.4, 125.7_2, 125.7_5, 126.6, 126.7, 128.1_2, 128.1_4, 141.6, 145.3, 147.1; FT-IR (film, cm^{-1}) 3435, 3056, 2976, 1445, 1053, 700; GC-MS (70eV), m/z (%): 268 (M^+, 1), 250 (100), 209 (66), 190 (81), 147 (81), 105 (60). Anal. Calcd. for $C_{18}H_{20}O_2$: C, 80.56; H, 7.51; Found: C, 80.74; H, 7.69. Major diasteromer. ^1H NMR (600 MHz; $CDCl_3$): δ 1.15 (d, J = 6.5 Hz, 3 H), 1.96−2.05 (m, 2 H), 2.42−2.46 (m, 1 H), 2.70−2.74 (m, 1 H), 4.03−4.07 (m, 1 H), 4.31−4.35 (m, 1 H), 5.06 (q, J = 6.5 Hz, 1 H), 7.19−7.21 (m, 1 H), 7.27−7.38 (m, 6 H), 7.56−7.58 (m, 1 H), 7.60−7.62 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.9, 24.7, 40.5, 64.8, 68.8, 89.5, 125.3, 126.2, 126.5, 127.1, 128.0, 128.1, 142.6, 143.9, 146.9; FT-IR (film, cm^{-1}) 3436, 3059, 2976, 1447, 1050, 701; GC-MS (70eV), m/z (%): 268 (M^+, 1), 250 (100), 209 (66), 190 (81), 147 (81), 105 (60). Anal. Calcd. for $C_{18}H_{20}O_2$: C, 80.56; H, 7.51; Found: C, 80.79; H, 7.64.

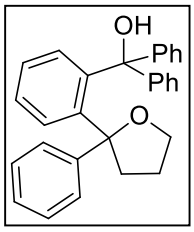


1-[2-(2-Phenyltetrahydrofuran2-yl)phenyl]cyclohexanol (4m): colourless oil, 70% (Et_2O). ^1H NMR (500 MHz; $CDCl_3$): δ 1.32−1.41 (m, 2 H), 1.48−1.62 (m, 3 H), 1.75−2.08 (m, 8 H), 2.34−2.40 (m, 1 H), 3.60−3.62 (m, 2H), 6.96−6.99 (m, 1 H), 7.07−7.09 (m, 1 H), 7.18−7.38 (m, 5 H), 7.60−7.61 (m, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 22.5, 25.4, 37.9, 38.8, 63.1, 67.4, 89.6, 121.0, 122.3, 125.1, 126.6, 127.6, 128.1, 143.5, 146.3_7, 146.3_8; FT-IR (film, cm^{-1}) 3048, 2981, 2310, 1417, 1265, 748; GC-MS (70eV), m/z (%): 322 (M^+, 3), 263 (100), 147 (10), 105 (12). Anal. Calcd. for $C_{22}H_{26}O_2$: C, 81.95; H, 8.13; Found: C, 82.18; H, 8.18.

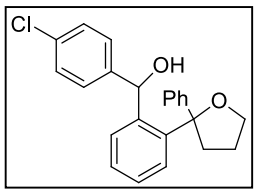


2-(2-(2-Phenyltetrahydrofuran-2-yl)phenyl)propan-2-ol (4n): colourless oil; 0% (Et_2O), 30% (CPME), 40% (ChCl/Gly 1/2). ^1H NMR (600 MHz; $CDCl_3$): δ 1.37 (s, 3 H), 1.53 (s, 3 H), 1.89-2.02 (m, 2 H), 2.51-2.63 (m, 2 H), 3.98-4.03 (m, 1 H), 4.17-4.21 (m, 1 H), 6.08 (br s, exchanges with D_2O, 1 H), 7.10-7.21 (m, 7 H), 7.30-7.33

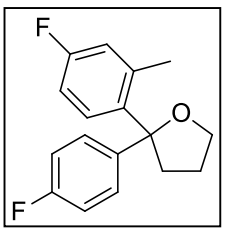
(m, 1 H), 7.39-7.41 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 28.0, 29.9, 30.0, 63.0, 85.3, 89.7, 120.8, 122.3, 125.3, 126.7, 127.6, 127.9, 128.1, 143.1, 146.1, 146.5; FT-IR (film, cm^{-1}) 3392, 3058, 2969, 1454, 1054, 758; GC-MS (70eV), m/z (%): 282 (M^+, 4), 264 (100), 190 (79), 147 (81), 105 (60). Anal. Calcd. for $C_{19}H_{22}O_2$: C, 80.82; H, 7.85; Found: C, 80.99; H, 7.88.



(2-(2-Phenyltetrahydrofuran-2-yl)phenyl)diphenylmethanol (4o): white solid, mp: 70–71 °C (Et_2O), 40% (Et_2O), 90% (CPME), 33% (ChCl/H_2O 1/2), 75% (ChCl/urea 1/2 and ChCl/Gly 1/2). ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.50−1.58 (m, 2 H), 1.73−1.84 (m, 1 H), 2.32−2.39 (m, 1 H), 2.60−2.73 (m, 2 H), 3.52−3.57 (m, 1 H), 6.65−6.68 (m, 1 H), 6.96−7.00 (m, 1 H), 7.03−7.06 (m, 2 H), 7.15−7.36 (m, 15H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.8, 41.4, 67.5, 83.3, 90.3, 125.4, 126.1, 126.3, 126.4, 126.6, 126.8, 127.3, 127.4, 127.6, 128.1, 128.6, 130.9, 132.6, 143.2, 144.7, 146.6, 149.3, 149.8; FT-IR (film, cm^{-1}) 3054, 1265, 740, 705; GC-MS (70eV), m/z (%): 406 (M^+, 1), 388 (8), 347 (53), 329 (97), 312 (34), 224 (31), 178 (67), 105 (100). Anal. Calcd. for $C_{29}H_{26}O_2$: C, 85.68; H, 6.45; Found: C, 85.91; H, 6.68.

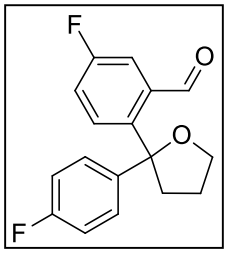


(4-Chlorophenyl)-(2-(2-phenyltetrahydrofuran-2-yl)phenyl) methanol (4p): (inseparable mixture of diastereomers, dr: 3/1), colourless oil, 40% overall yield (Et_2O), 90% overall yield (CPME). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.99−2.06 (m, 2 H major + 2 H minor), 2.39−2.44 (m, 1 H major + 1 H minor), 2.78−2.84 (m, 1 H major + 1 H minor), 3.95−4.00 (m, 1 H minor), 4.06−4.10 (m, 1 H major), 4.18−4.22 (m, 1 H minor), 4.36−4.40 (m, 1 H major), 6.04 (s, 1 H major), 6.40 (s, 1 H minor), 6.73−6.75 (m, 2 H major), 6.83−6.85 (m, 2 H minor), 7.12−7.34 (m, 10 H major + 10 H minor), 7.60−7.62 (m, 1 H major + 1 H minor); ^{13}C NMR (125 MHz, $CDCl_3$): δ 24.7, 25.1, 39.5, 40.3, 68.2, 69.2, 70.3, 88.6, 89.9, 125.4, 125.5, 125.9, 126.2, 126.7, 127.1, 127.4, 127.6, 127.8, 128.1, 128.2_1, 128.2_3, 128.3, 130.6, 130.7, 132.0, 141.3, 142.6, 142.7, 143.2, 143.4, 143.6, 147.3, 147.5; FT-IR (film, cm^{-1}) 3584, 3058; 2979; 1491, 1090, 911; GC-MS (70eV), m/z (%): 364 (M^+, 10), 346, (9), 329 (85), 305 (96), 270 (100), 240 (58), 207 (46), 165 (70), 105 (58). Anal. Calcd. for $C_{23}H_{21}ClO_2$: C, 75.71; H, 5.80; Found: C, 75.84; H, 5.91.

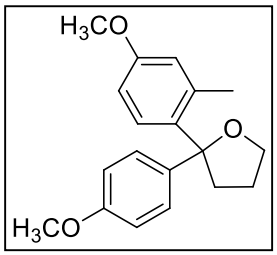


2-(4-Fluoro-2-methylphenyl)-2-(4-fluorophenyl)tetrahydrofuran (4q): colourless oil, 80% (Et_2O). ^{1}H NMR (600 MHz; $CDCl_3$): δ 1.96−2.08 (m, 2 H), 2.05 (s, 3 H), 2.48−2.53 (m, 2 H), 3.89−3.95 (m, 1 H), 4.08−4.13 (m, 1 H), 6.79−6.82 (m, 1 H), 6.87−6.98 (m, 3 H), 7.19−7.25 (m, 2 H), 7.58−7.62 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4, 25.8, 38.7, 67.7, 87.6, 111.5 (d, $^2J_{C\text{-}F}$ =

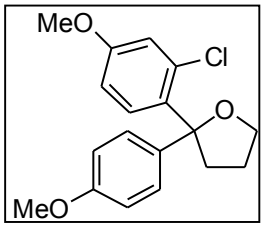
20.1 Hz), 114.7 (d, $^{2}J_{C\text{-}F}$ = 20.1 Hz), 118.7 (d, $^{2}J_{C\text{-}F}$ = 20.1 Hz), 127.3 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 127.7 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 139.2(d, $^{4}J_{C\text{-}F}$ = 5.0 Hz), 139.3 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 141.3 (d, $^{4}J_{C\text{-}F}$ = 5.0 Hz), 161.1 (d, $^{1}J_{C\text{-}F}$ = 240 Hz), 161.3 (d, $^{1}J_{C\text{-}F}$ = 240 Hz); FT-IR (film, cm^{-1}) 3054, 2983, 2306, 1507, 1265, 1158, 1048, 748; GC-MS (70eV), m/z (%): 274 (M^{+}, 22), 259 (8), 231 (100), 215 (58), 179 (84), 165 (88), 137 (43), 123 (49). Anal. Calcd. for $C_{17}H_{16}F_2O$: C, 74.44; H, 5.88; Found: C, 74.74; H, 5.95.



(5-Fluoro-2-(2-(4-fluorophenyl)tetrahydrofuran-2-yl)benzaldehyde (4r): colourless oil, 70% (Et_2O). ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.95−2.05 (m, 2 H), 2.42−2.48 (m, 1 H), 2.63−2.70 (m, 1 H), 3.91-3.97 (m, 1 H), 4.17−4.23 (m, 1 H), 6.94−7.00 (m, 2 H), 7.23−7.29 (m, 3 H), 7.52−7.55 (m, 1 H), 7.59−7.63 (m, 1 H), 10.3 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 25.2, 40.7, 68.3, 87.7, 115.2 (d, $^{2}J_{C\text{-}F}$ = 20.1 Hz), 115.5 (d, $^{2}J_{C\text{-}F}$ = 20.1 Hz), 119.0 (d, $^{2}J_{C\text{-}F}$ = 20.0 Hz), 127.1 (d, $^{3}J_{C\text{-}F}$ = 8.5 Hz), 128.2, (d, $^{3}J_{C\text{-}F}$ = 8.5 Hz), 137.0 (d, $^{4}J_{C\text{-}F}$ = 5.0 Hz), 142.1 (d, $^{4}J_{C\text{-}F}$ = 5.0 Hz), 142.8 (d, $^{3}J_{C\text{-}F}$ = 7.5 Hz), 161.8 (d, $^{1}J_{C\text{-}F}$ = 240 Hz), 162.0 (d, $^{1}J_{C\text{-}F}$ = 240 Hz), 191.9; FT-IR (film, cm^{-1}) 3048, 2928, 1689, 1456, 1265, 740; GC-MS (70eV), m/z (%): 288 (M^{+}, 44), 270 (59), 243 (100), 229 (46), 201 (62), 123 (80). Anal. Calcd. for $C_{17}H_{14}F_2O_2$: C, 70.83; H, 4.89; Found: C, 71.12; H, 4.95.

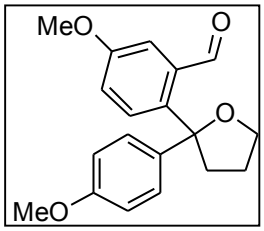


2-(4-Methoxy-2-methylphenyl)-2-(4-methoxyphenyl)tetrahydrofuran (4s): colourless oil, 70% (Et_2O). ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.95−2.02 (m, 2 H), 2.05 (s, 3 H), 2.47−2.51 (m, 2 H), 3.75 (s, 3 H), 3.77 (s, 3 H), 3.87−3.93 (m, 1 H), 4.05−4.10 (m, 1 H), 6.64−6.78 (m, 4 H), 7.15−7.17 (m, 2 H), 7.54−7.56 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.6, 36.5, 55.1, 55.4, 67.2, 86.3, 87.0, 112.0, 113.1, 116.4, 127.9, 128.2, 132.3, 136.1, 136.2, 158.4, 158.9; FT-IR (film, cm^{-1}) 2953, 2835, 1607, 1508, 1247, 1043, 829; GC-MS (70eV), m/z (%): 298 (M^{+}, 60), 283 (12), 267 (20), 255 (42), 225 (29), 191 (90), 177 (100). Anal. Calcd. for $C_{19}H_{20}O_4$: C, 73.06; H, 6.45; Found: C, 73.31; H, 6.66.

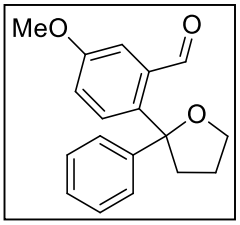


2-(2-Chloro-4-methoxyphenyl)-2-(4-methoxyphenyl)tetrahydrofuran (4t): colourless oil, 85% (Et_2O). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.94−2.015 (m, 2 H), 2.55−2.60 (m, 1 H), 2.94−2.99 (m, 1 H), 3.80 (s, 3 H), 3.82 (s, 3 H) 3.95−4.07 (m, 2 H), 6.82−6.90 (m, 4 H), 7.25−7.27 (m, 2 H), 7.81−7.83 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.6, 36.5, 55.1, 55.4, 67.2, 86.3, 87.0, 112.0, 113.1, 116.4, 127.9, 128.2, 132.3, 136.1, 136.2, 158.4, 158.9; FT-IR (film, cm^{-1}) 2954, 1609, 1509, 1246, 1174,

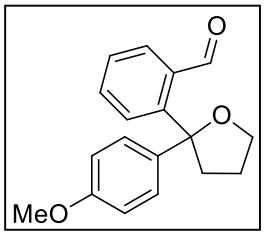
1034, 828, 736; GC-MS (70eV), m/z (%): 284 (M+, 51), 269 (1), 253 (27), 223 (6), 177 (100), 135 (55). Anal. Calcd. for $C_{18}H_{19}ClO_3$: C, 67.82; H, 6.01; Found: C, 67.87; H, 5.95.



5-Methoxy-2-(2-(4-methoxyphenyl)tetrahydrofuran-2-yl)benzaldehyde (4u): colourless oil, 60% (Et_2O). ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.90−1.99 (m, 2 H), 2.37−2.44 (m, 1 H), 2.57−2.64 (m, 1 H), 3.73 (s, 3 H), 3.81 (s, 3 H), 3.86−3.92 (m, 1 H), 4.12−4.17 (m, 1 H), 6.76−6.78 (m, 2 H), 7.04−7.07 (m, 1 H), 7.17−7.19 (m, 2 H), 7.34−7.35 (m, 1 H), 7.50−7.52 (m, 1 H), 10.33 (s, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 25.3, 40.6, 55.2, 55.5, 68.0, 87.8, 112.2, 113.6, 119.0, 126.7, 127.7, 136.0, 139.0, 140.1, 158.4, 158.8, 193.4; FT-IR (film, cm^{-1}) 3521, 2954, 1683, 1606, 1176, 1034, 826, 732; GC-MS (70eV), m/z (%): 312 (M^+, 100), 282 (22), 267 (60), 255 (68), 135(32). Anal. Calcd. for $C_{19}H_{20}O_4$: C, 73.06; H, 6.45; Found: C, 73.30; H, 6.56.

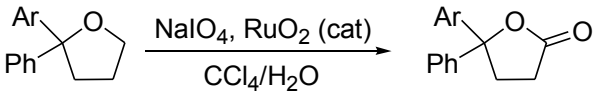


5-Methoxy-2-(2-phenyltetrahydrofuran-2-yl)benzaldehyde (4v): colourless oil, 55% (Et_2O), 57% (CPME). ^{1}H NMR (400 MHz; $CDCl_3$): δ 1.91−1.99 (m, 2 H), 2.38−2.45 (m, 1 H), 2.61−2.68 (m, 1 H), 3.81 (s, 3 H), 3.89−3.94 (m, 1 H), 4.15−4.20 (m, 1 H), 7.07 (dd, J = 8.6, 2.9 Hz, 1H), 7.13−7.30 (m, 5 H), 7.35 (d, J = 2.9 Hz, 1 H), 7.53 (d, J = 8.6 Hz, 1 H), 10.35 (s, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 25.3, 40.7, 55.5, 68.2, 88.2, 112.3, 119.0, 125.4, 126.9, 127.8, 128.3, 136.2, 139.9, 146.9, 158.9, 193.5; FT-IR (film, cm^{-1}) 3059, 2926, 2880, 1683, 1603, 1245, 763, 701; GC-MS (70eV), m/z (%): 282 (M^+, 35), 264 (15), 237 (40), 223 (100), Anal. Calcd. for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43; Found: C, 76.79; H, 6.59.



2-[2-(4-Methoxyphenyl)tetrahydrofuran-2-yl]benzaldehyde (4x): colourless oil, 30% (Et_2O), 33% (CPME). ^{1}H NMR (500 MHz; $CDCl_3$): δ 1.94−2.08 (m, 2 H), 2.50−2.55 (m, 1 H), 2.64−2.70 (m, 1 H), 3.79 (s, 3 H), 3.93−3.97 (m, 1 H), 4.18−4.22 (m, 1 H), 6.82−6.84 (m, 2 H), 7.24−7.26 (m, 2 H), 7.40−7.43 (m, 1 H), 7.57−7.60 (m, 1 H), 7.65−7.67 (m, 1 H), 7.85−7.87 (m, 1 H), 10.40 (s, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 25.4, 40.5, 55.5, 68.1, 88.1, 113.7, 126.3, 126.8, 127.6, 128.8, 132.4, 135.0, 138.5, 147.5, 158.6, 193.7; FT-IR (film, cm^{-1}) 3054, 2985, 1687, 1509, 1265, 738, 704; GC-MS (70eV), m/z (%): 282 (M^+, 70), 237 (100), 223 (75), 177 (80), 152 (25), 135 (40). Anal. Calcd. for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43; Found: C, 76.79; H, 6.59.

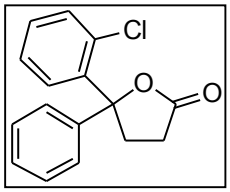
2.6 Preparation of γ-butyrolactones 5a,f. Typical procedure.



3a (Ar = Ph)
4f (Ar = 2-ClC_6H_4)

5a (Ar = Ph)
5f (Ar = 2-ClC_6H_4)

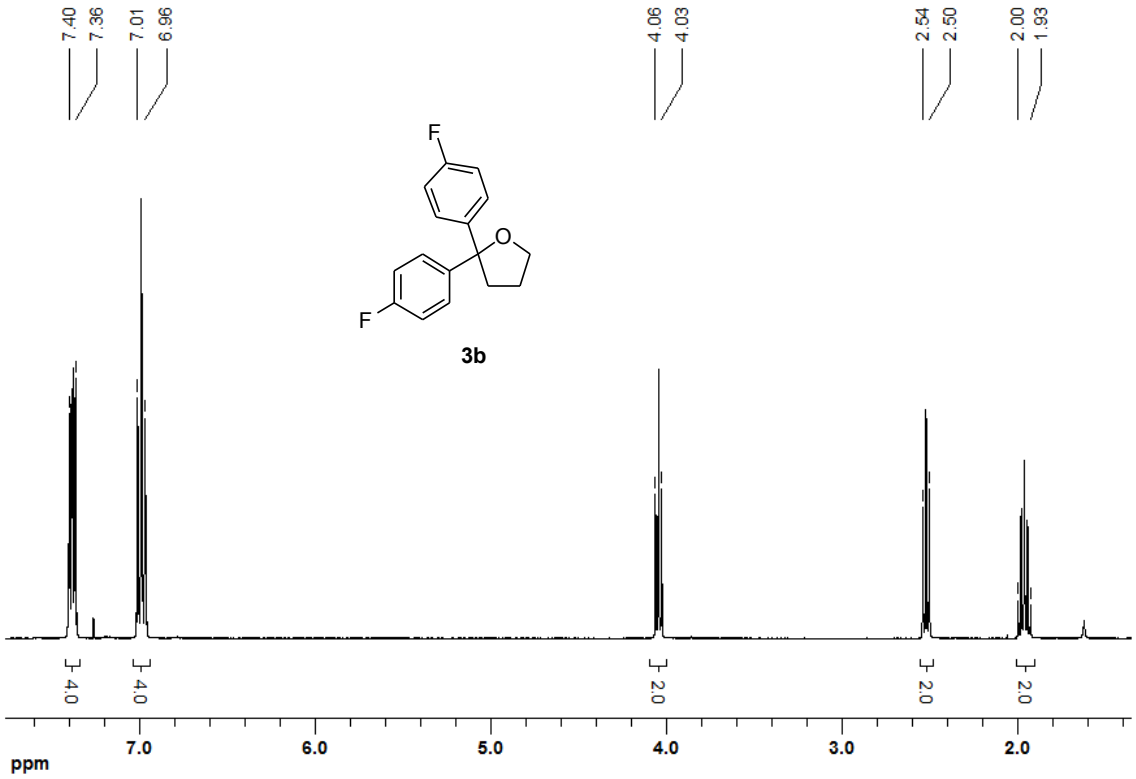
A suspension of $RuO_2 \cdot H_2O$ (21 mg, 0.16 mmol) and $NaIO_4$ (1.04 g, 4.88 mmol) in H_2O (5 ml) was added to a solution of **3a** or **4f** (1.0 mmol) in CCl_4 (5 ml) at room temperature. After stirring for 24 h, additional water was added (5 mL), and the resulting mixture was extracted with CH_2Cl_2 (3 × 5 ml). The combined organic phases were dried over Na_2SO_4, passed through a pad of celite, and concentrated *in vacuo*. The crude product was purified by flash-chromatography (silica gel; hexane/AcOEt 9/1) to afford the lactone **5a** or **5f**, each in 70% yield.



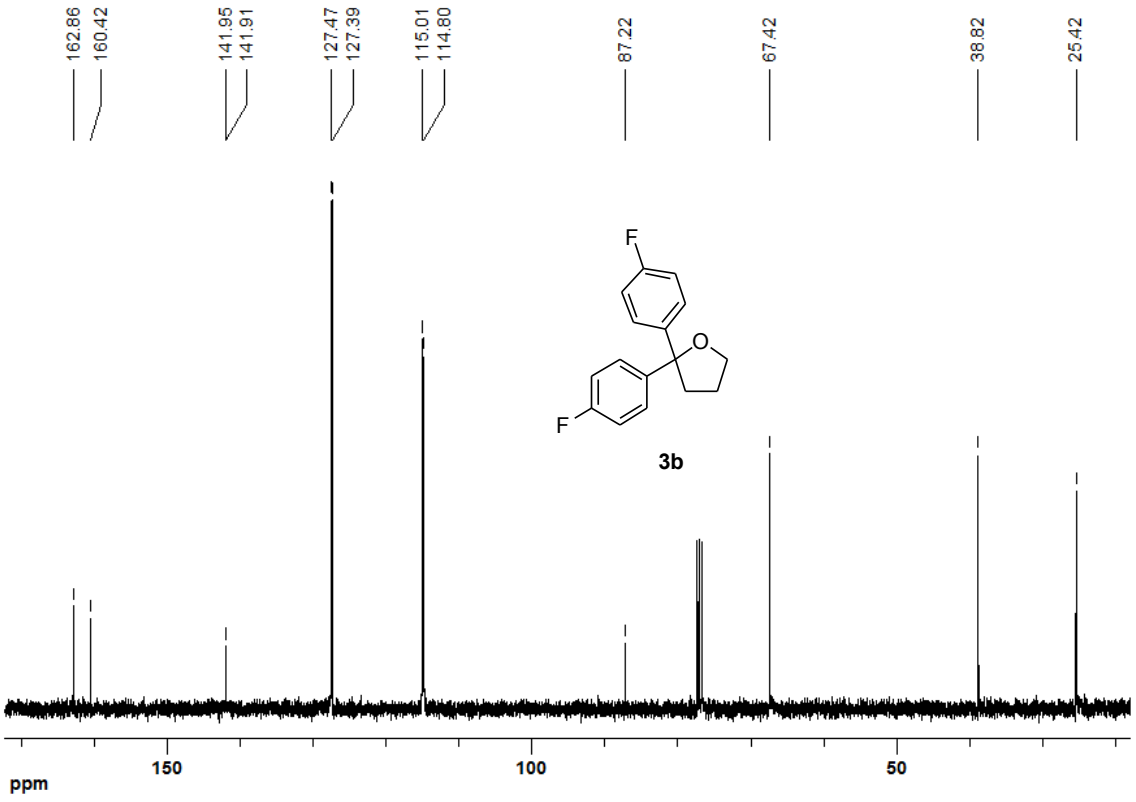
5-(2-Chlorophenyl)-5-phenyldihydrofuran-2(3H)-one (5f): colourless oil, 80%. ^{1}H NMR (500 MHz; $CDCl_3$): δ 2.60–2.76 (m, 2 H), 2.91–2.98 (m, 1 H), 3.41–3.47 (m, 1 H), 7.26–7.41 (m, 7 H), 7.83–7.84 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 29.2, 32.4, 89.4, 126.9, 127.0, 127.1, 128.3, 128.4, 129.5, 131.5, 131.6, 140.1, 140.7, 175.6; FT-IR (film, cm^{-1}) 2986, 1741, 1374, 1241; ESI-MS: 295 [M^+ + 23 (Na)]. Anal. Calcd. for $C_{16}H_{13}ClO_2$: C, 70.46; H, 4.80; Found: C, 70.70; H, 4.99.

3. ^{1}H and ^{13}C NMR spectra of compounds 3b–d, 4a–x, 5f

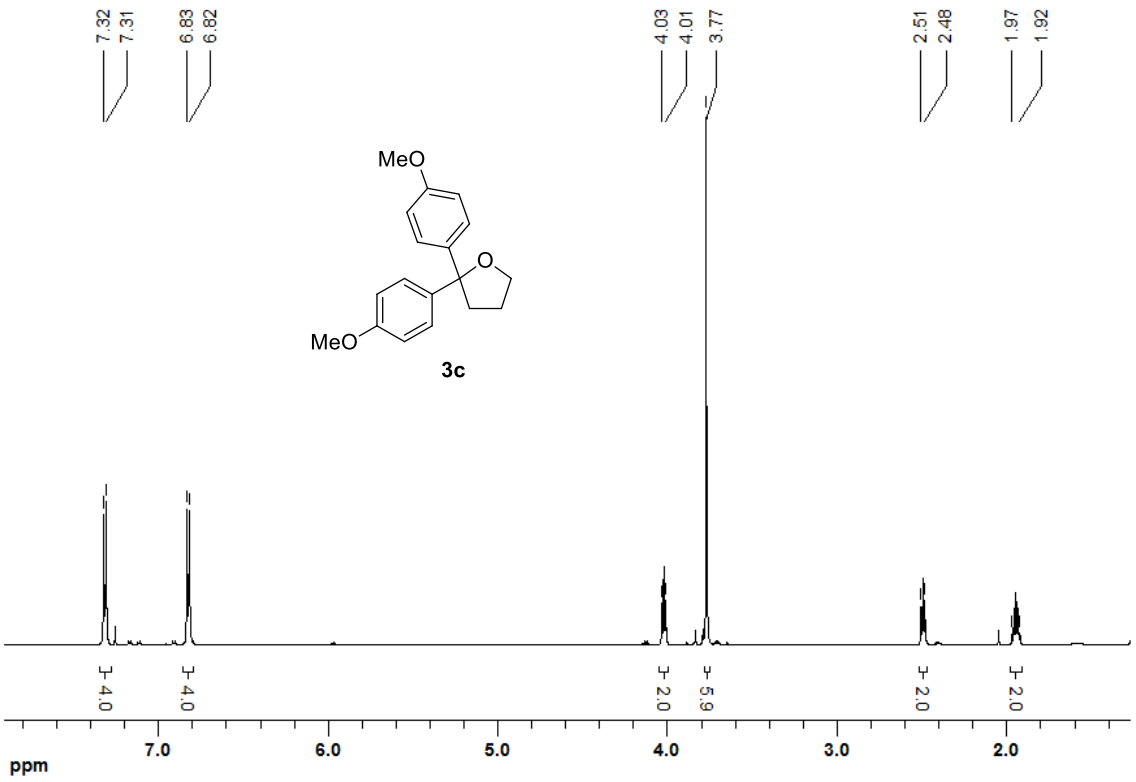
^{1}H NMR, 400 MHz, $CDCl_3$



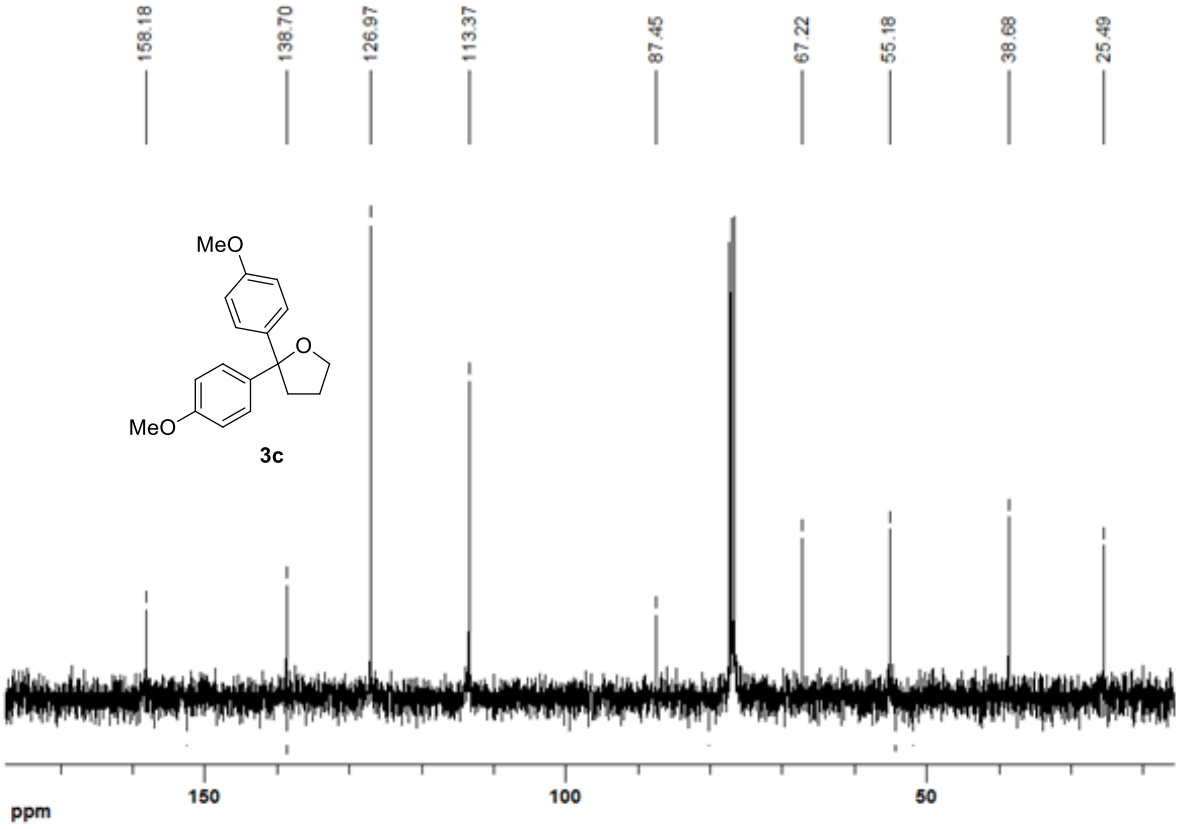
^{13}C NMR, 150 MHz, $CDCl_3$



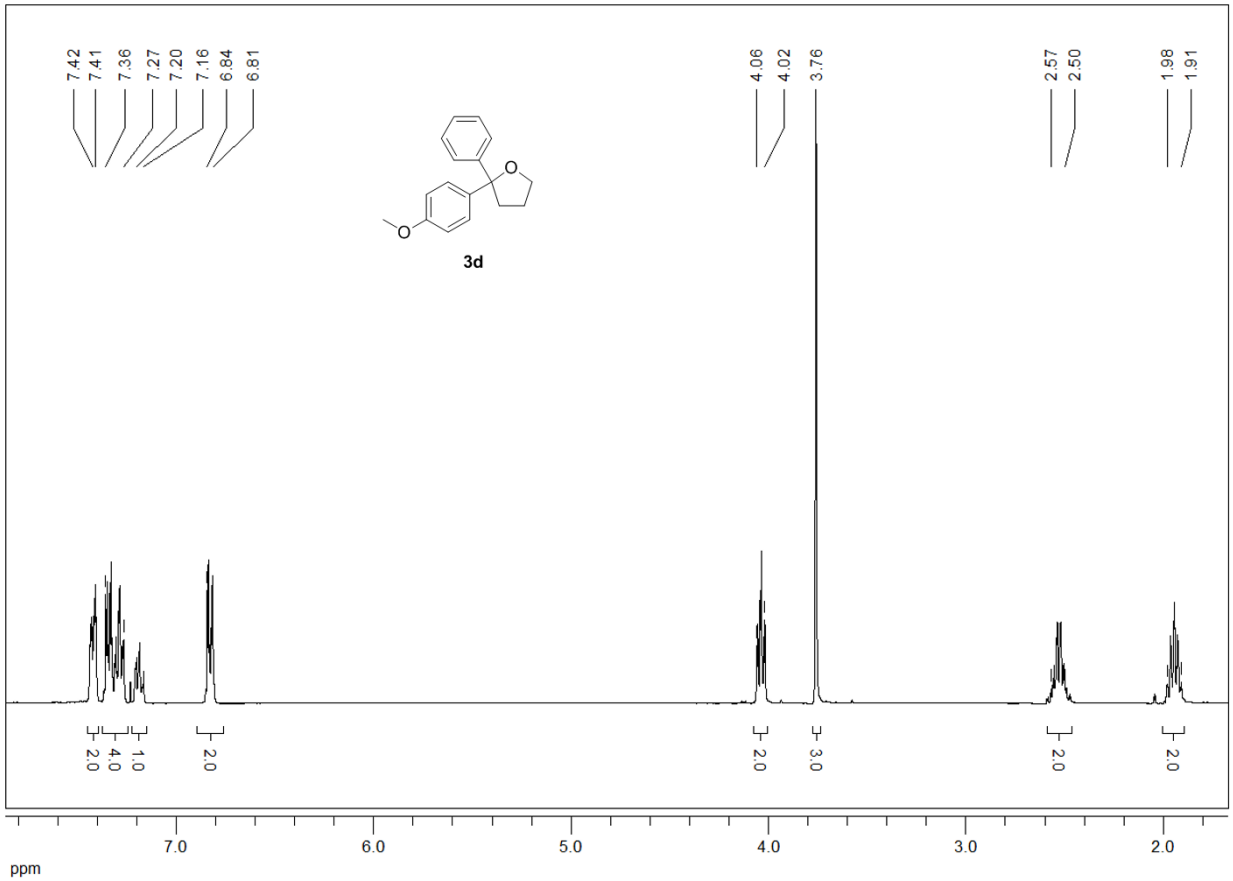
^{1}H NMR, 600 MHz, $CDCl_3$



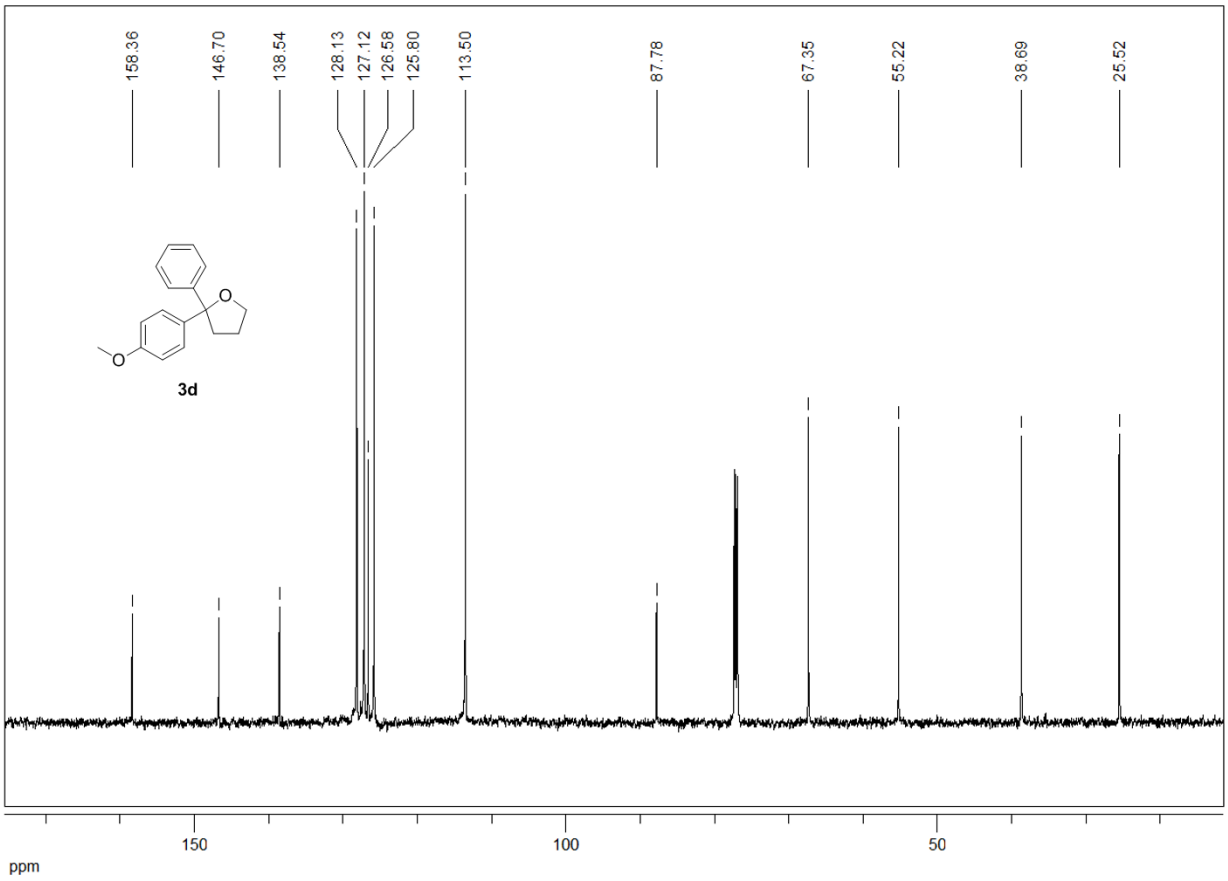
^{13}C NMR, 100 MHz, $CDCl_3$



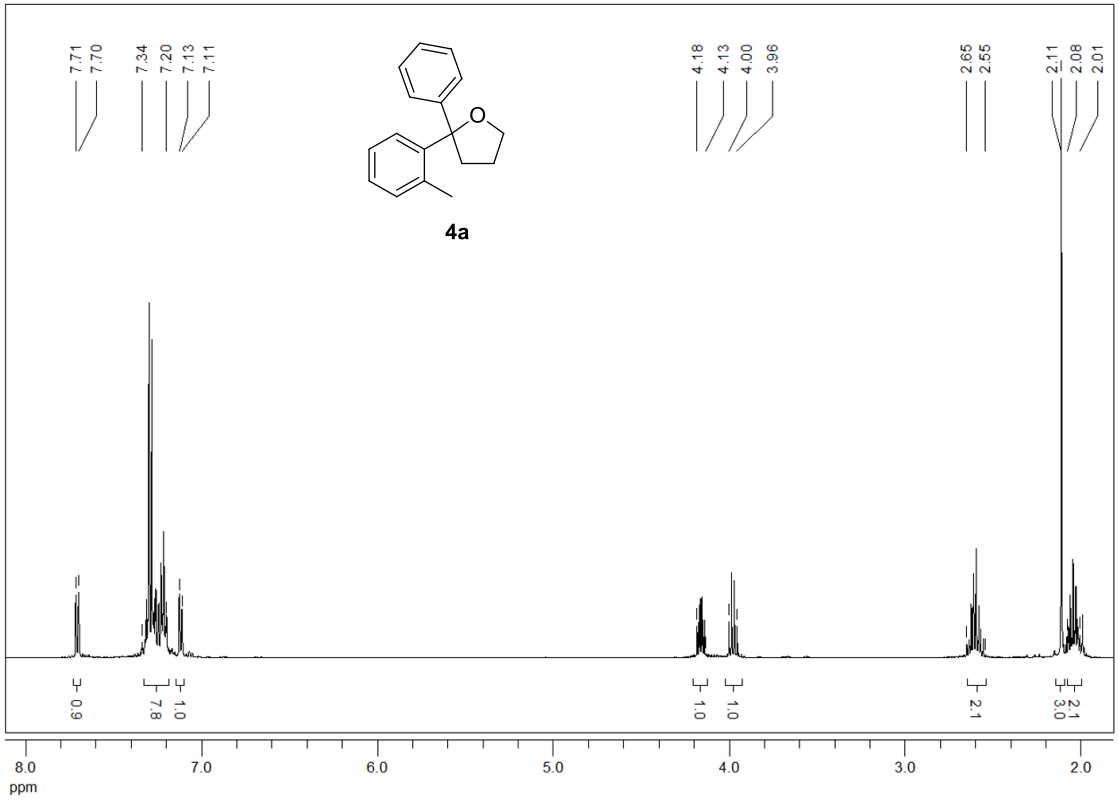
^{1}H NMR, 400 MHz, $CDCl_3$



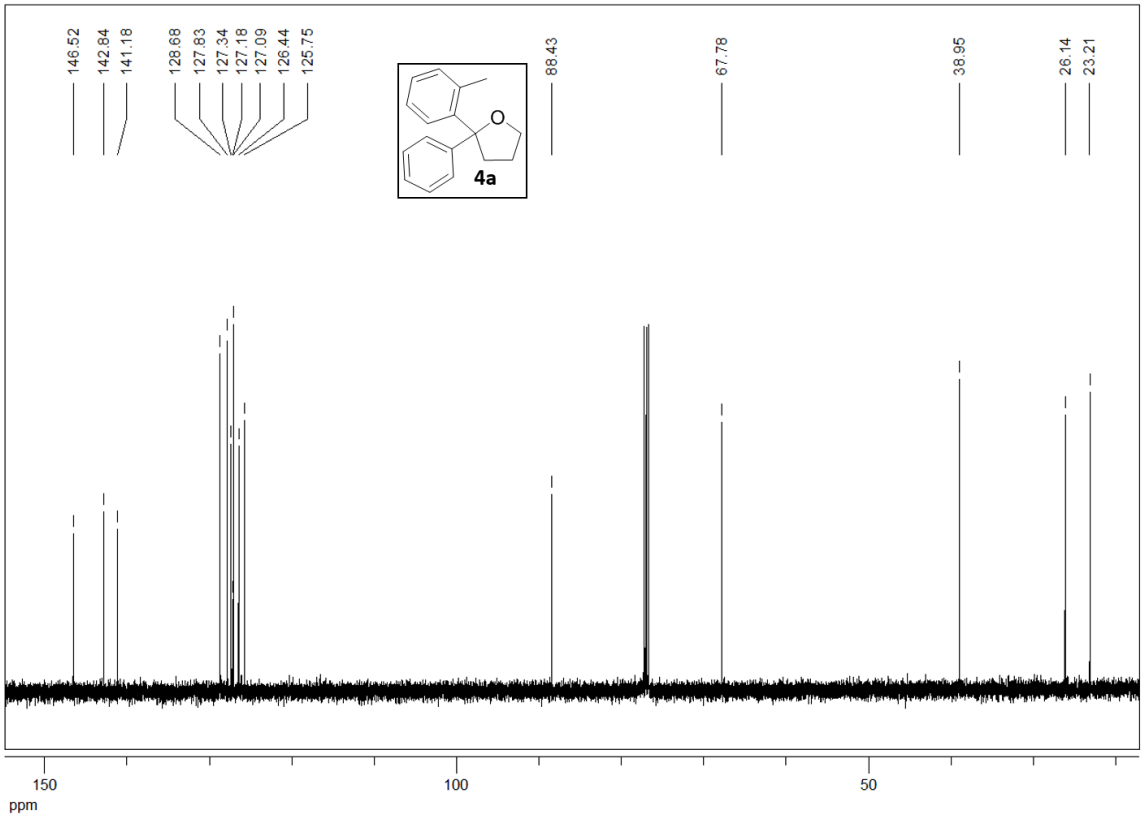
^{13}C NMR, 100 MHz, $CDCl_3$



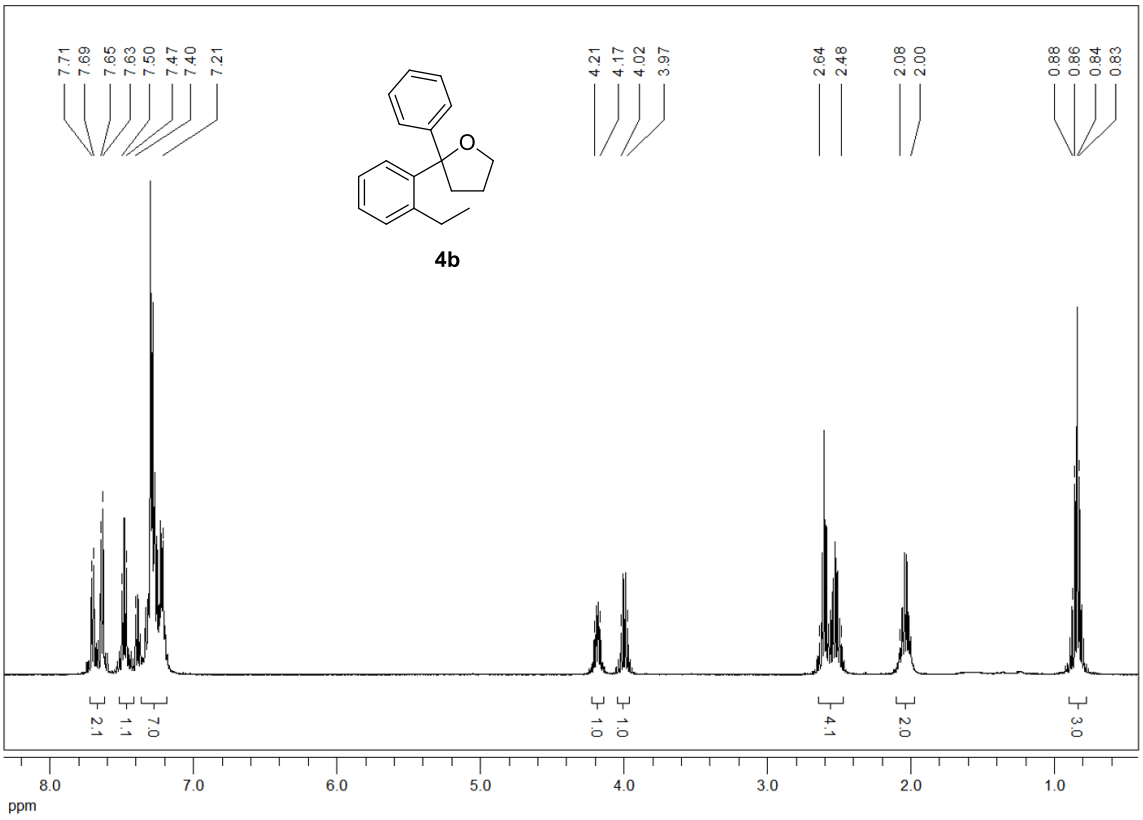
^{1}H NMR, 500 MHz, $CDCl_3$



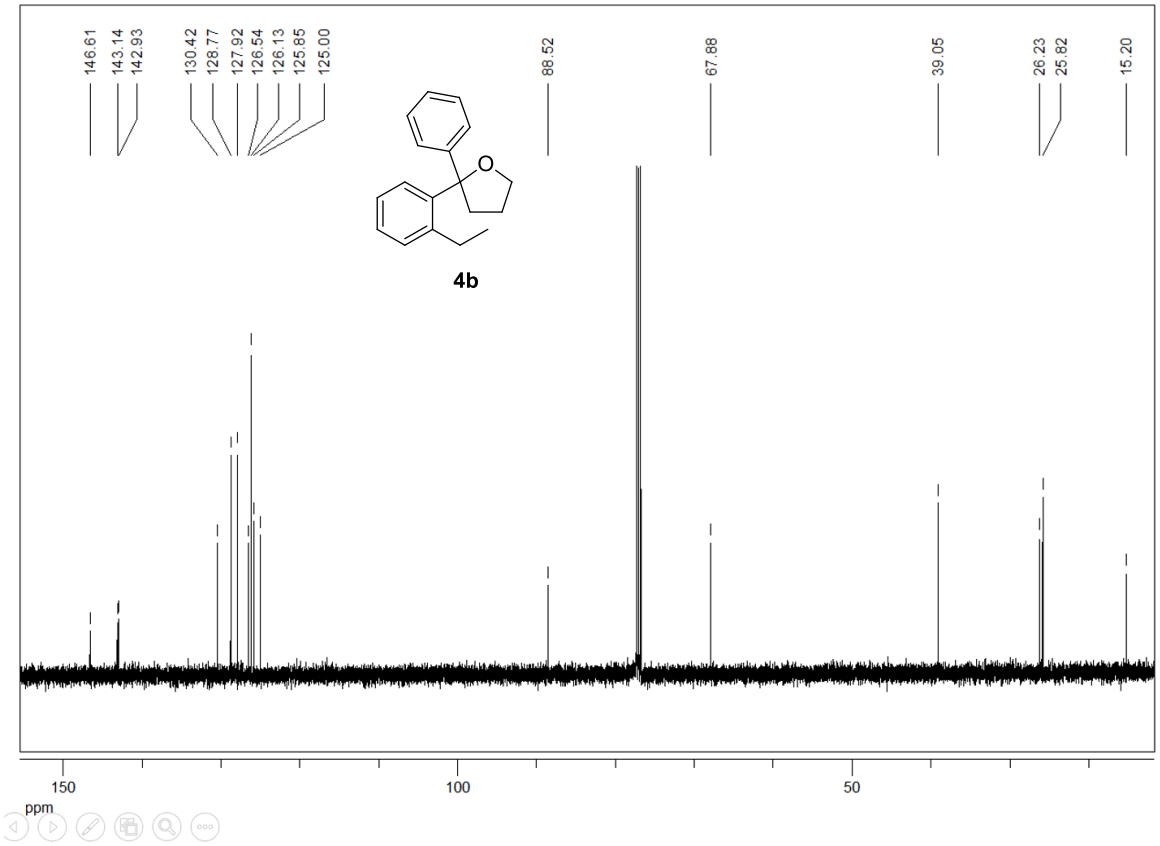
^{13}C NMR, 125 MHz, $CDCl_3$



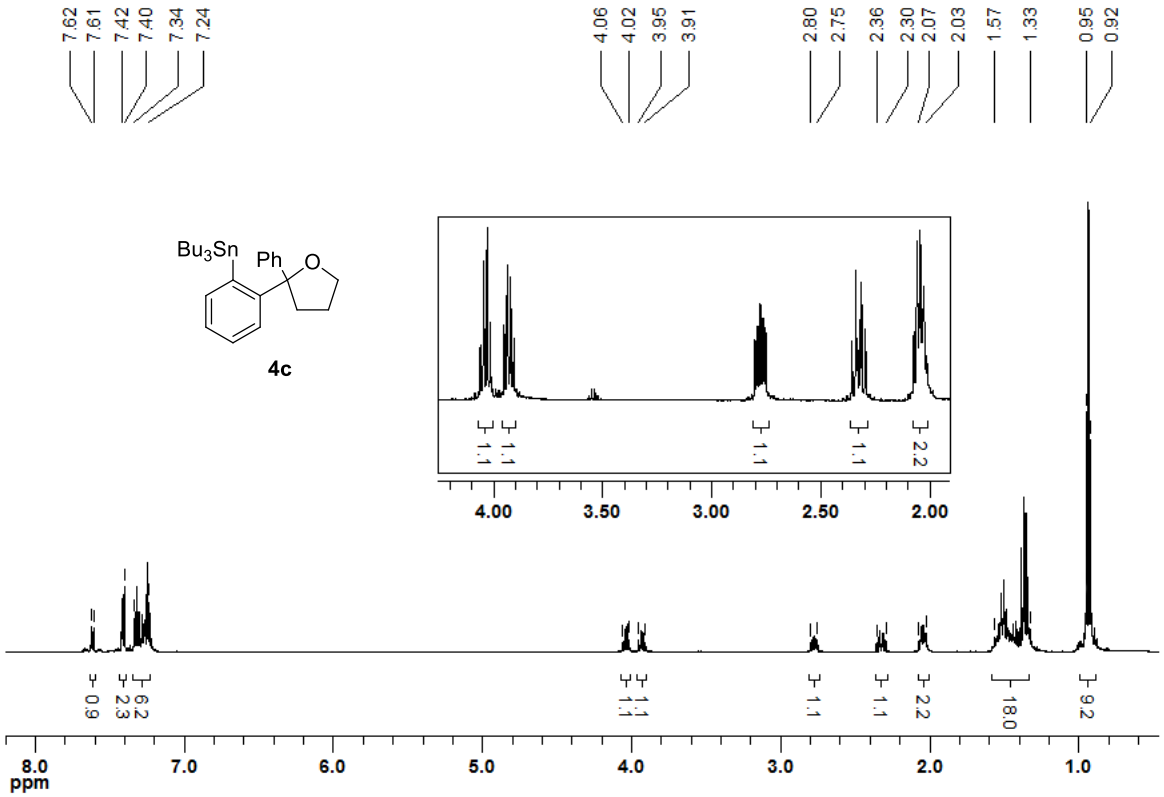
^{1}H NMR, 500 MHz, $CDCl_3$



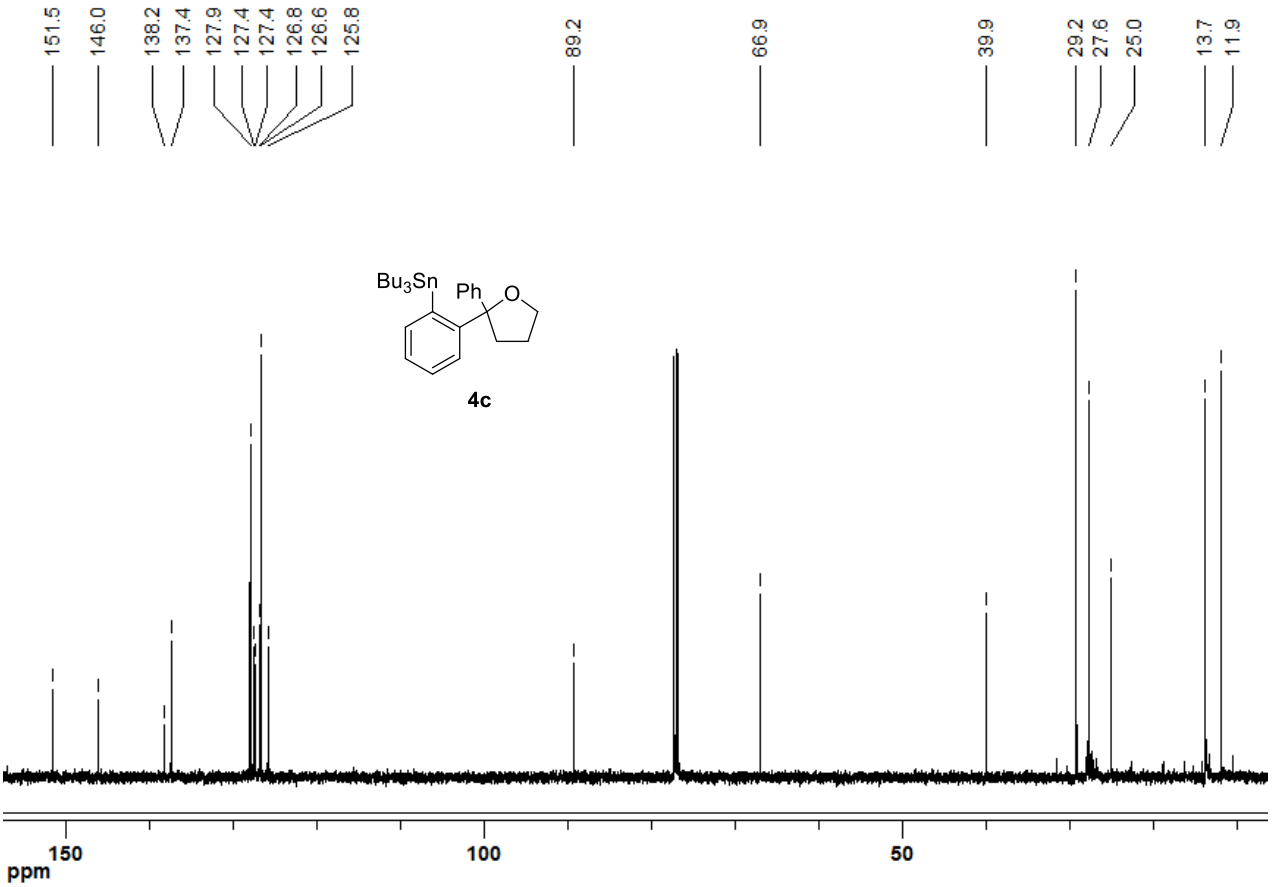
^{13}C NMR, 125 MHz, $CDCl_3$



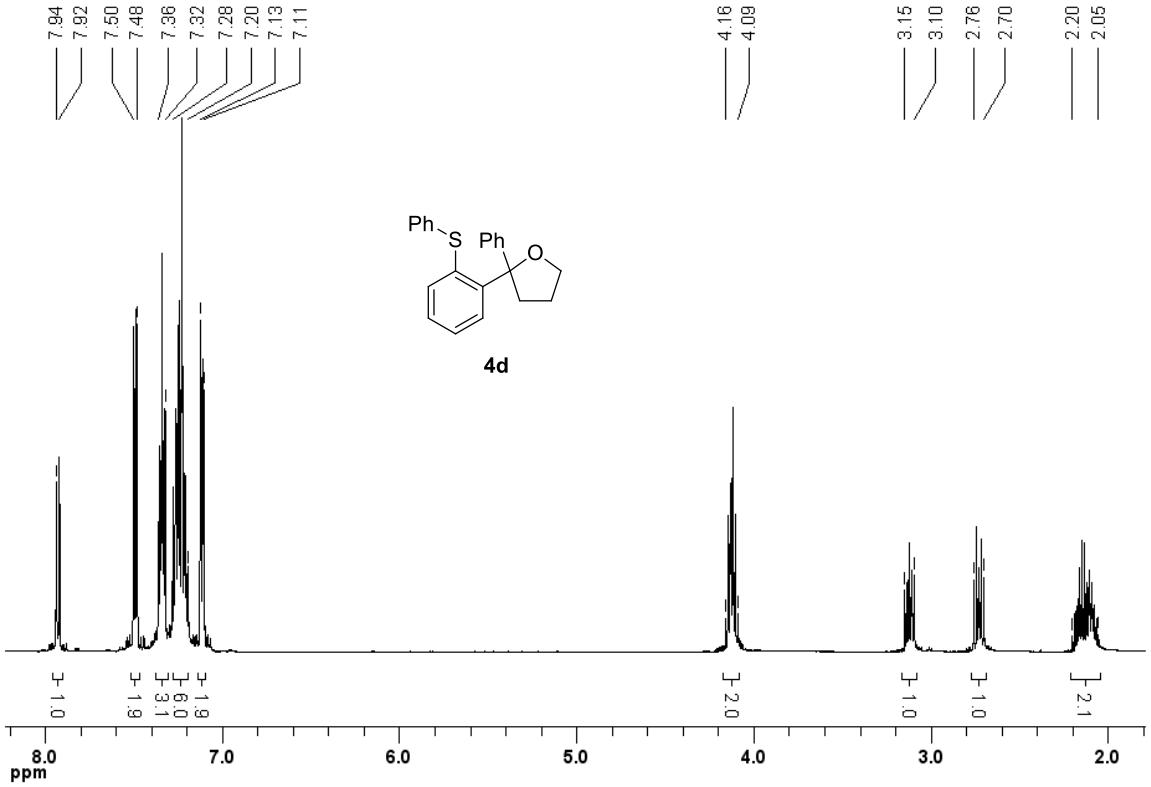
^{1}H NMR, 500 MHz, $CDCl_3$



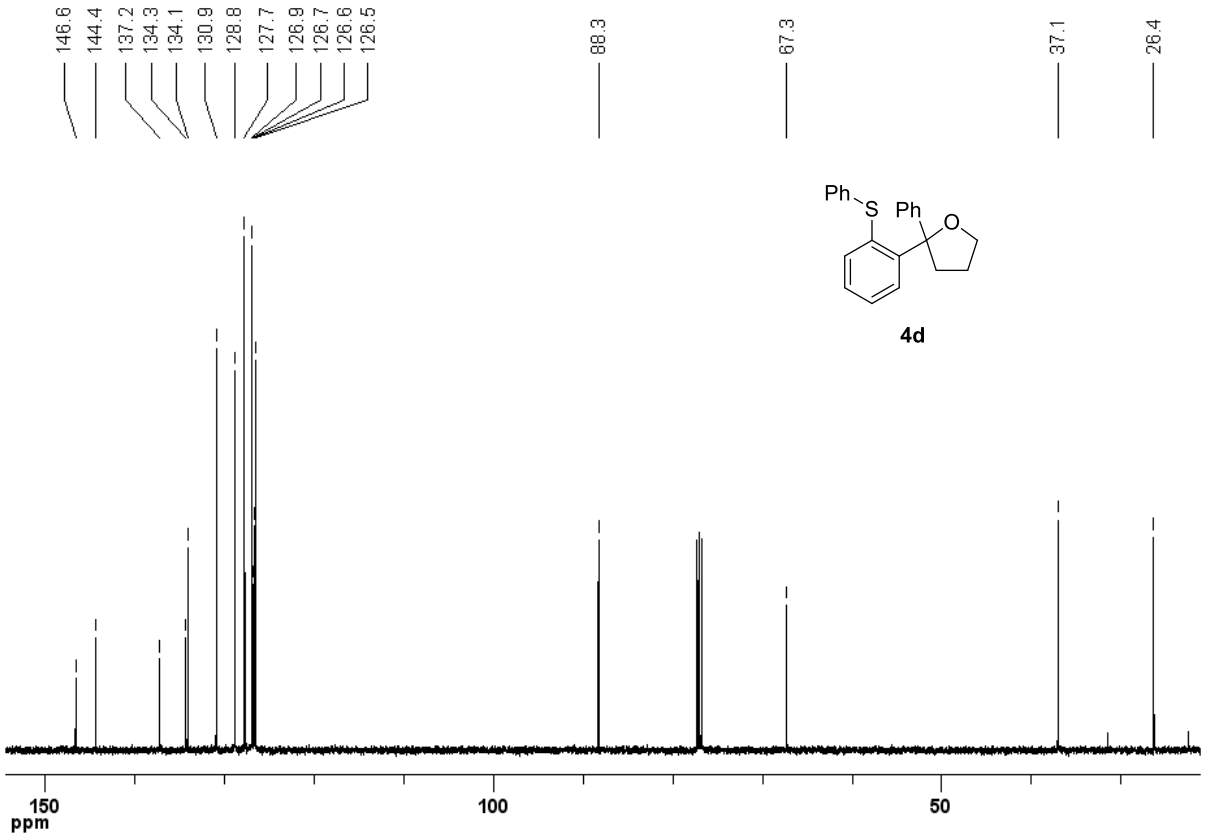
^{13}C NMR, 125 MHz, $CDCl_3$



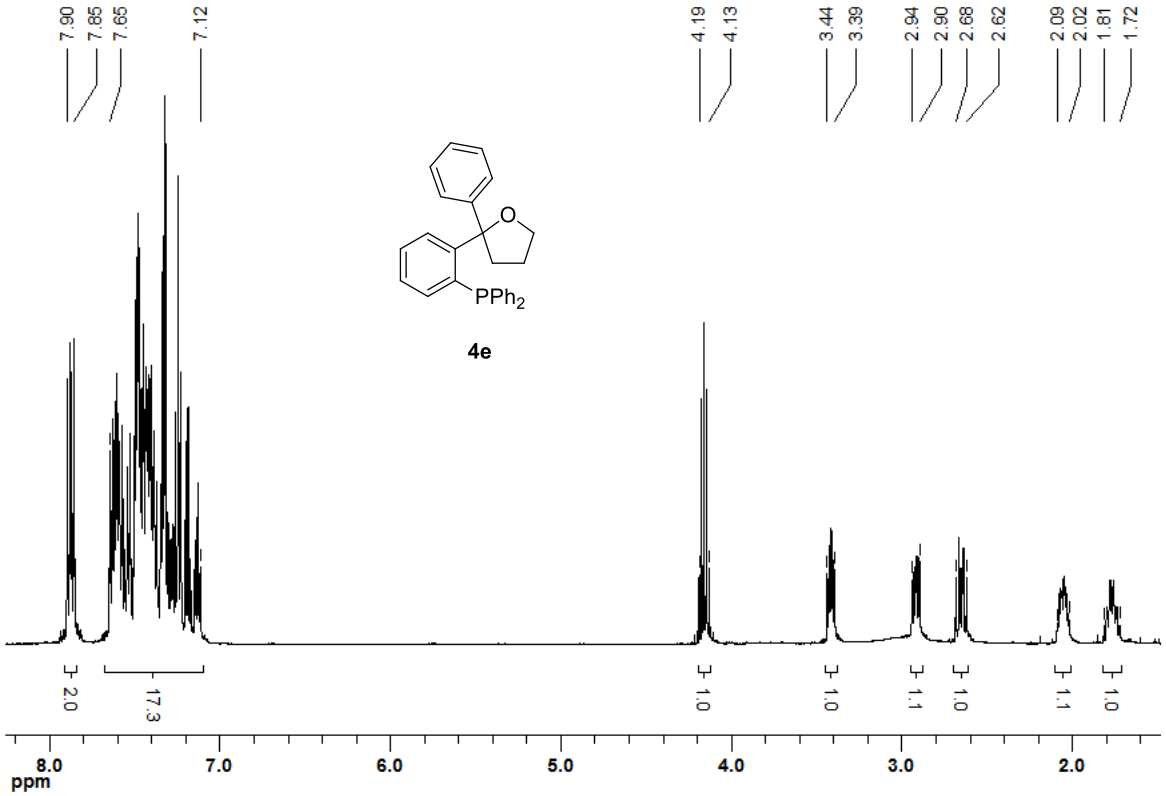
^{1}H NMR, 500 MHz, $CDCl_3$



^{13}C NMR, 125 MHz, $CDCl_3$

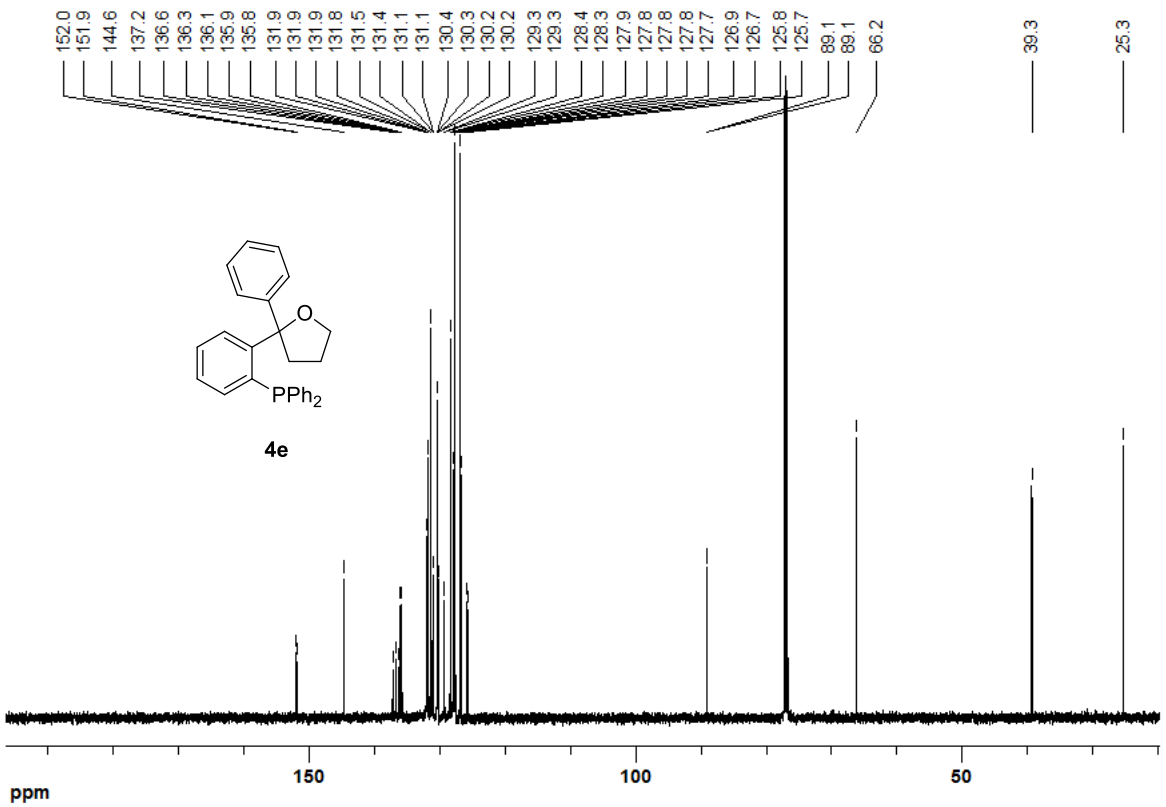


^{1}H NMR, 500 MHz, $CDCl_3$



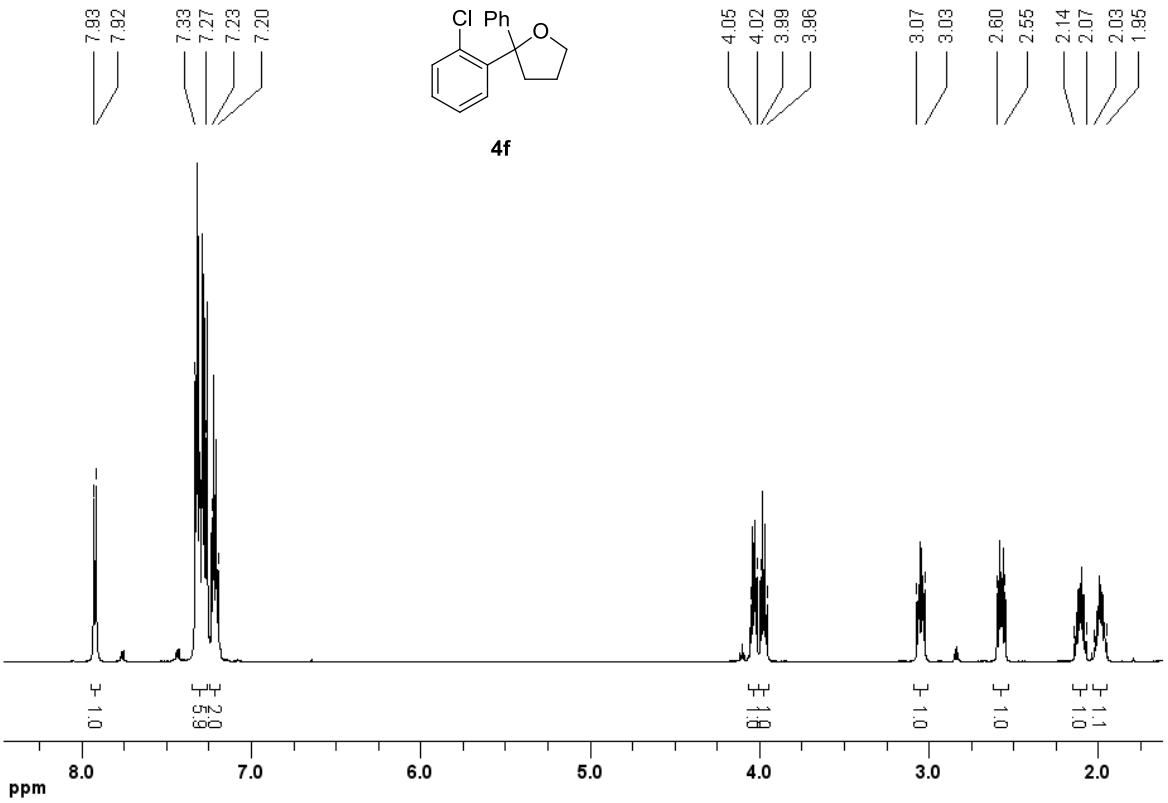
4e

^{13}C NMR, 125 MHz, $CDCl_3$

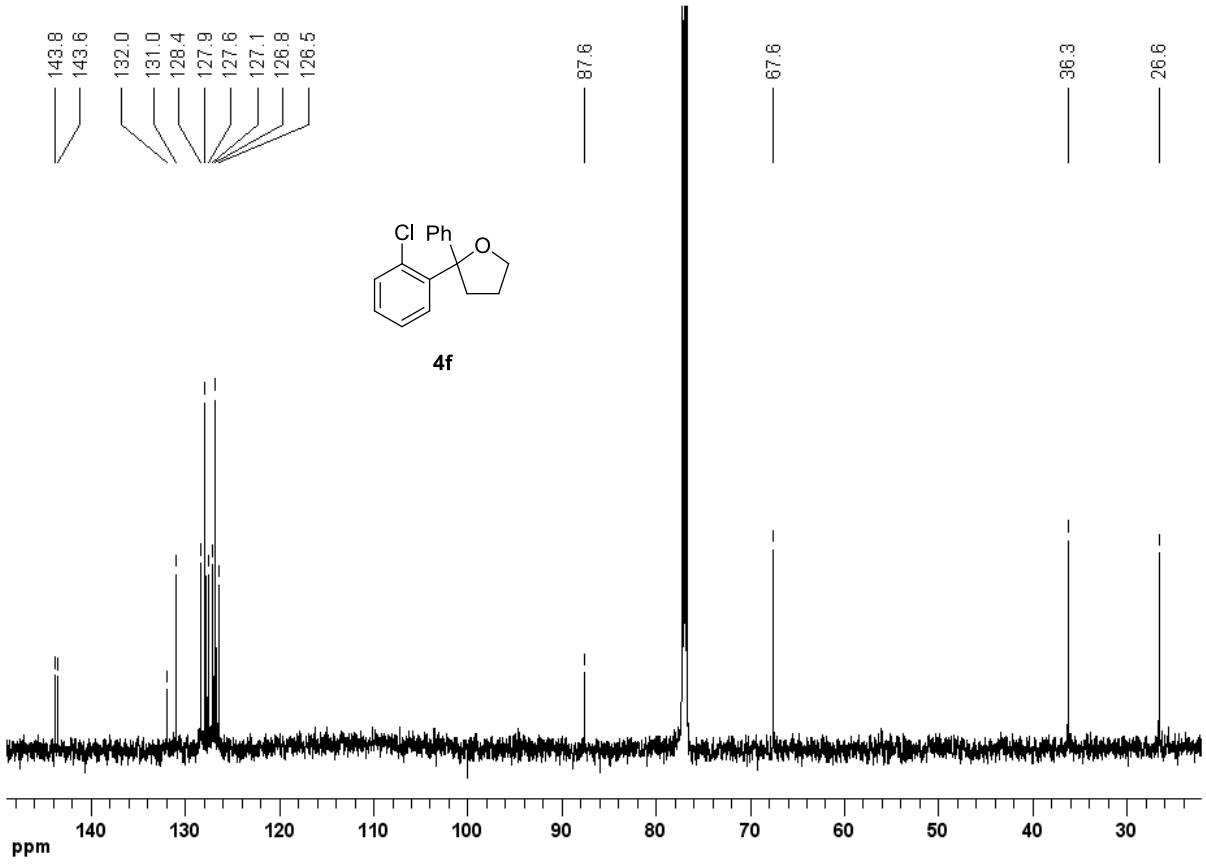


4e

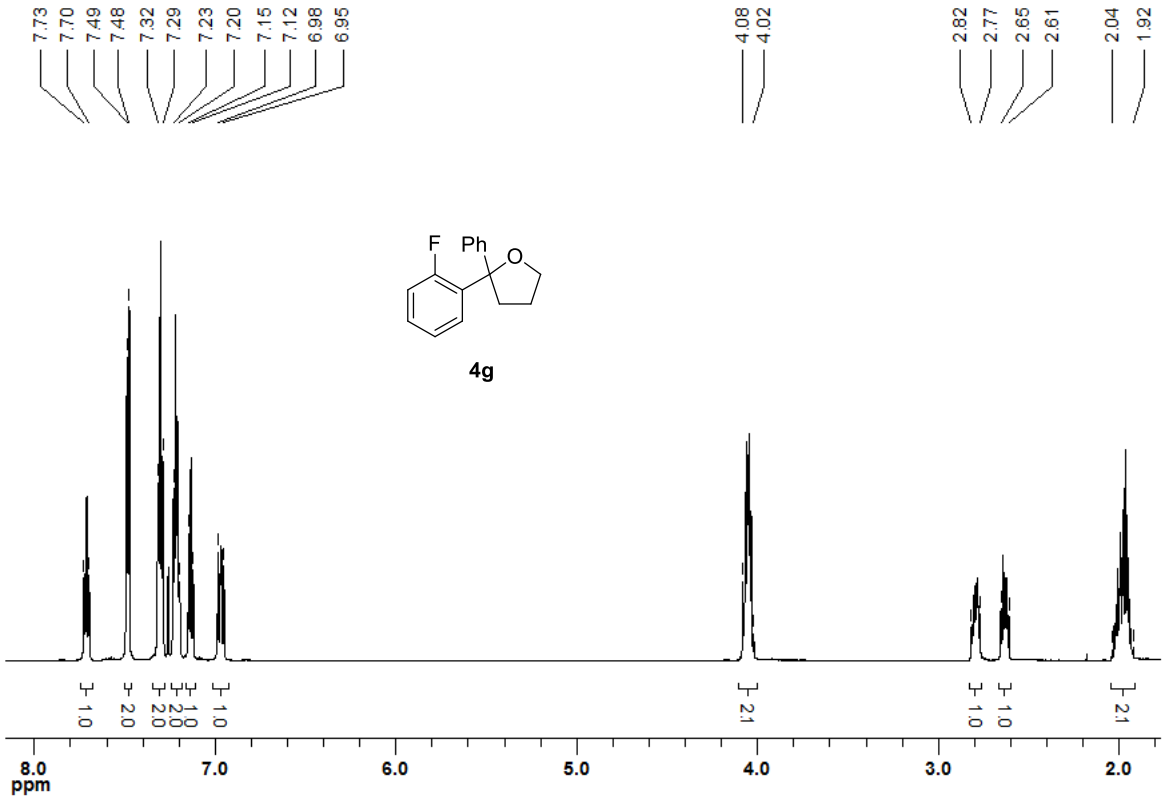
^{1}H NMR, 600 MHz, $CDCl_3$



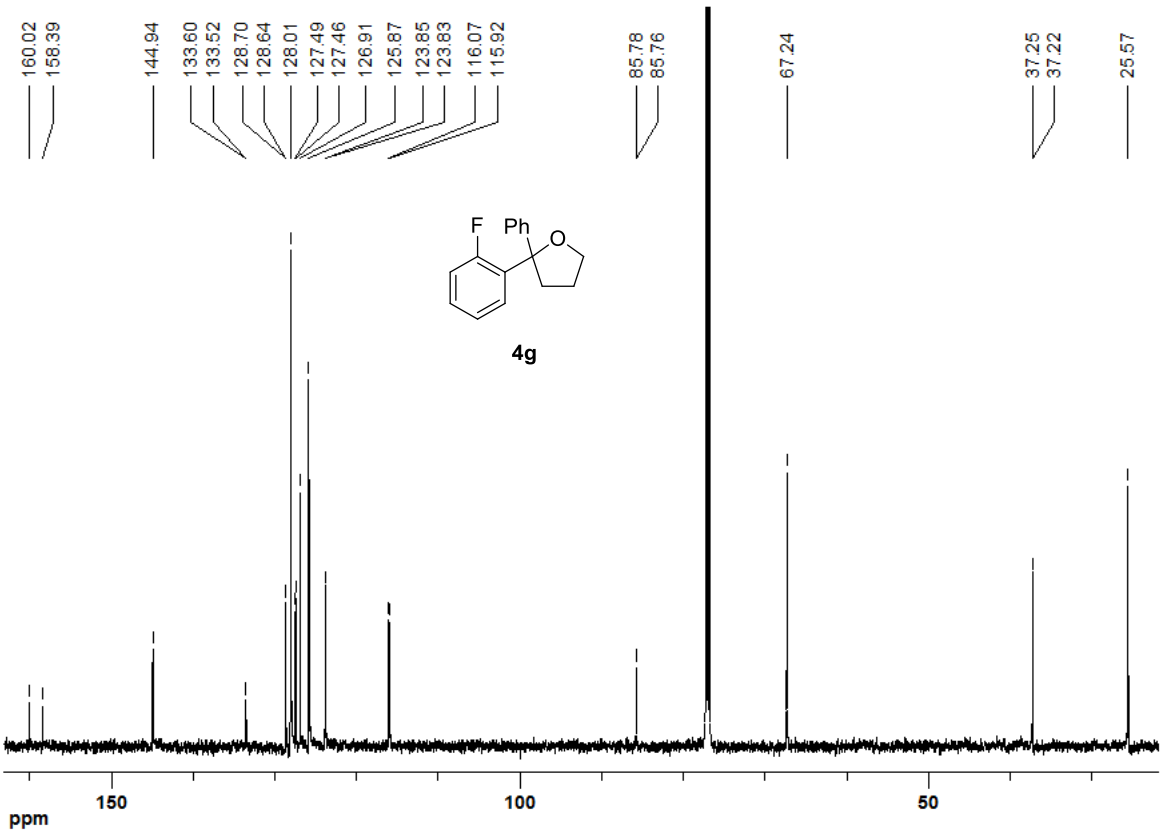
^{13}C NMR, 150 MHz, $CDCl_3$



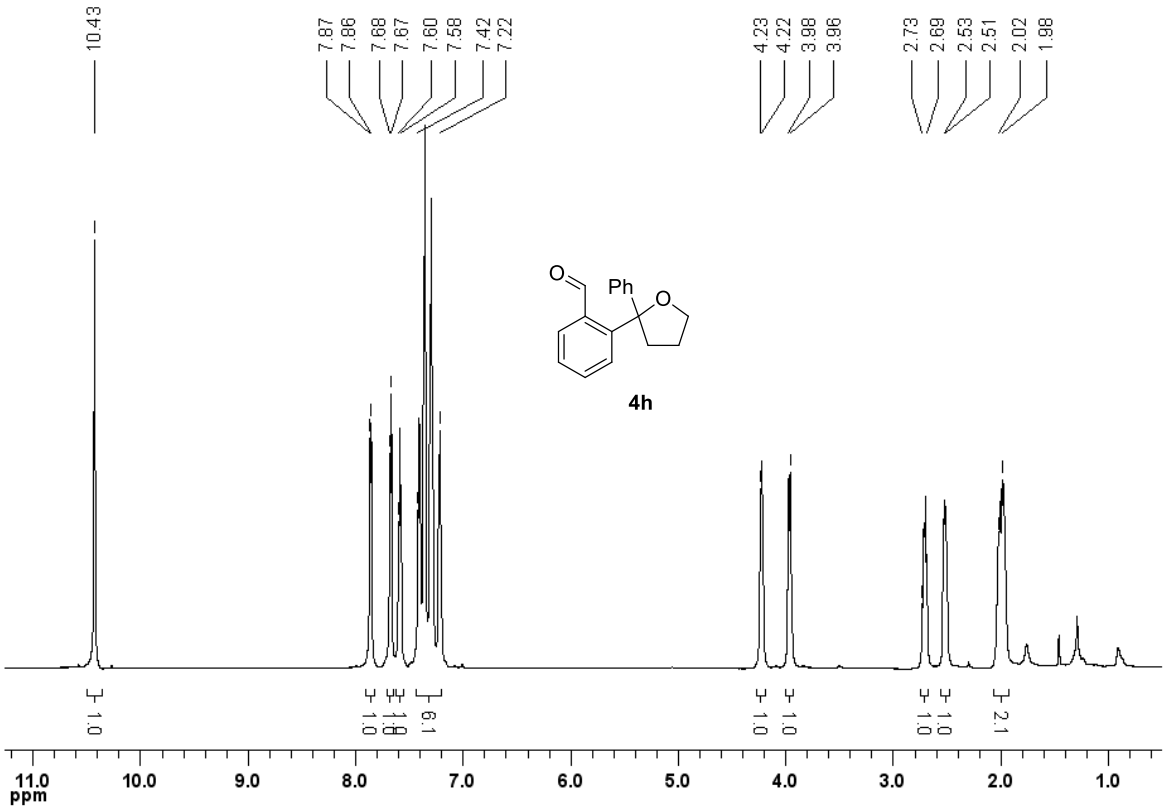
^{1}H NMR, 500 MHz, $CDCl_3$



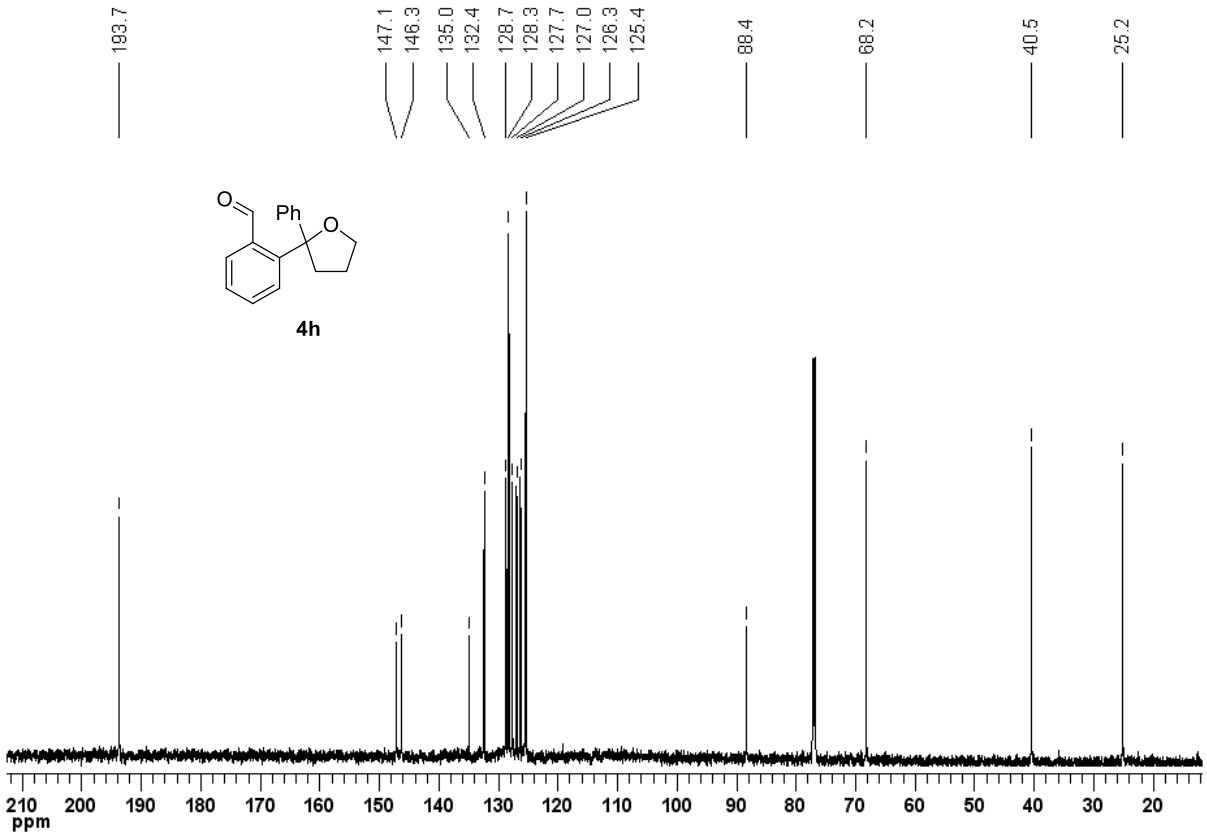
^{13}C NMR, 125 MHz, $CDCl_3$



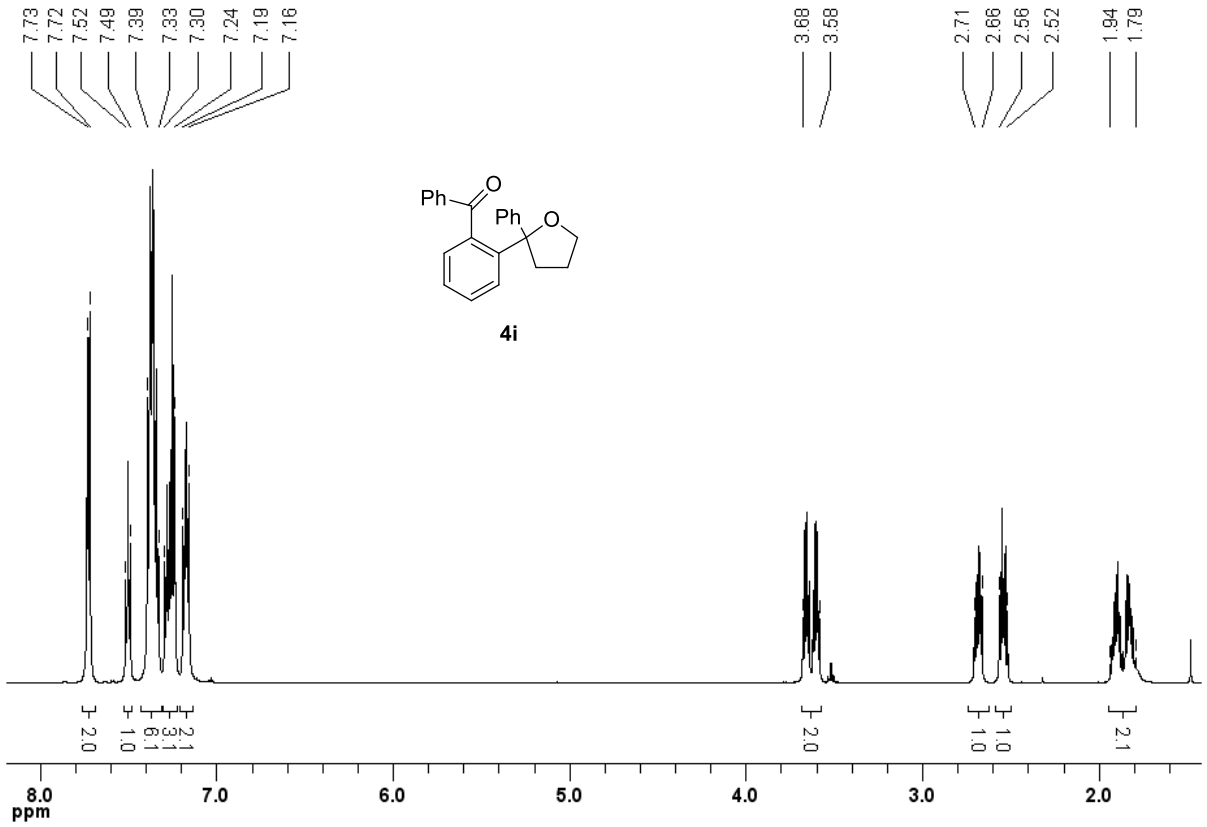
^{1}H NMR, 600 MHz, $CDCl_3$



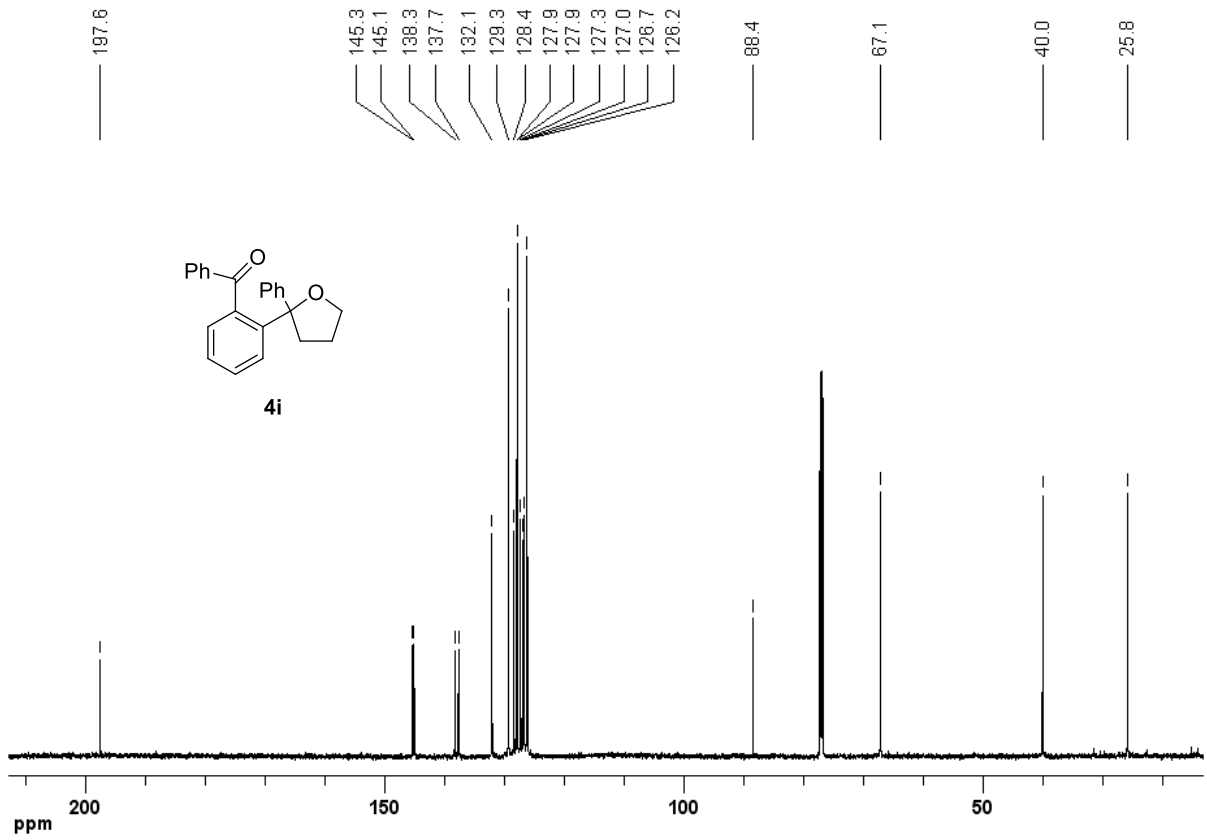
^{13}C NMR, 150 MHz, $CDCl_3$



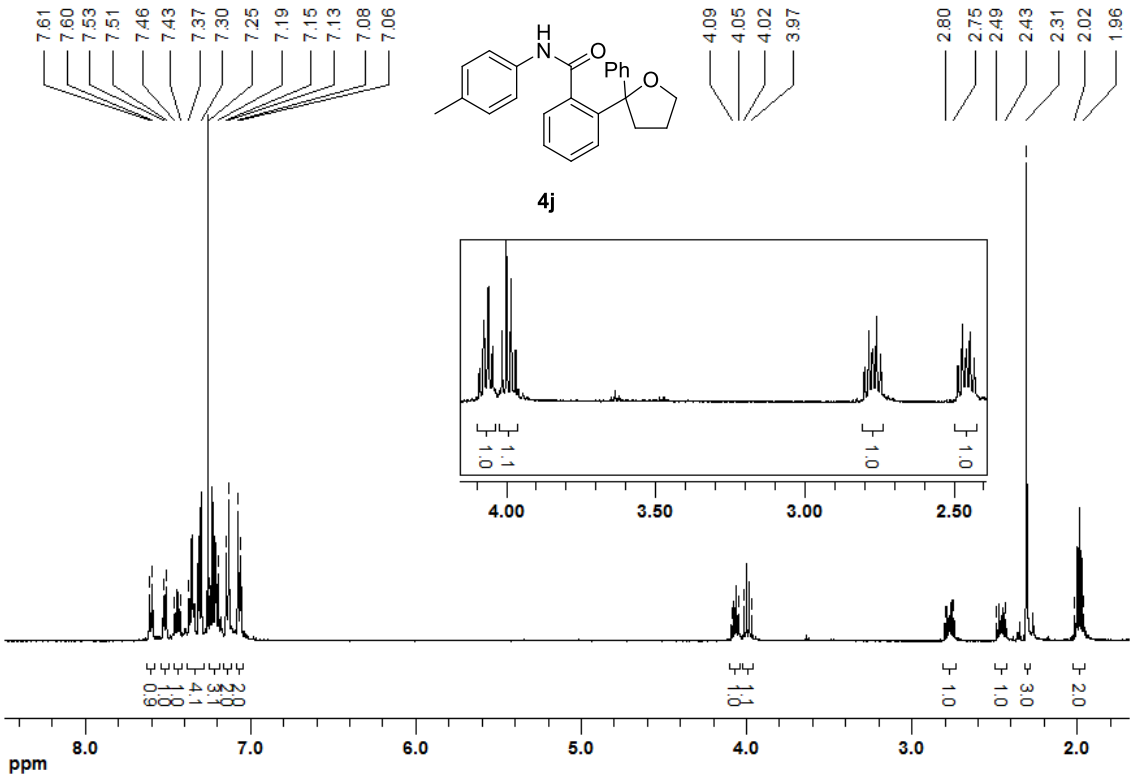
^{1}H NMR, 600 MHz, $CDCl_3$



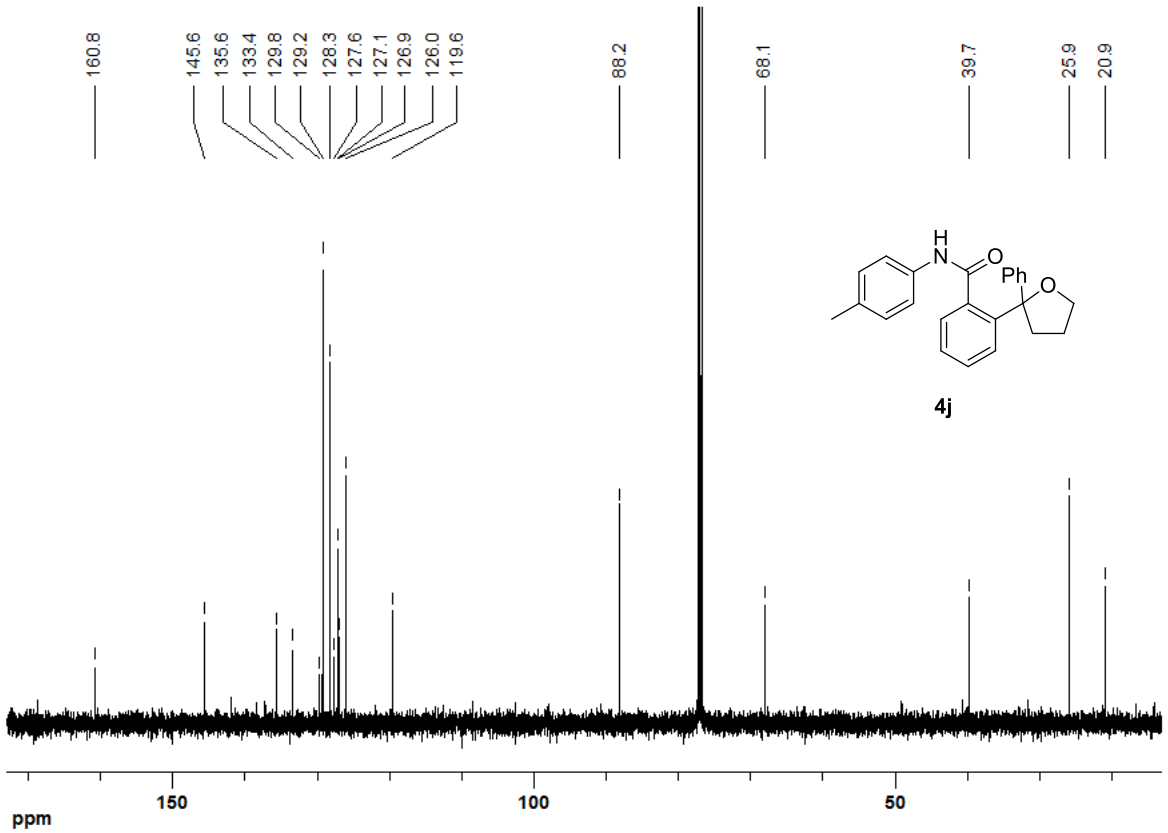
^{13}C NMR, 150 MHz, $CDCl_3$



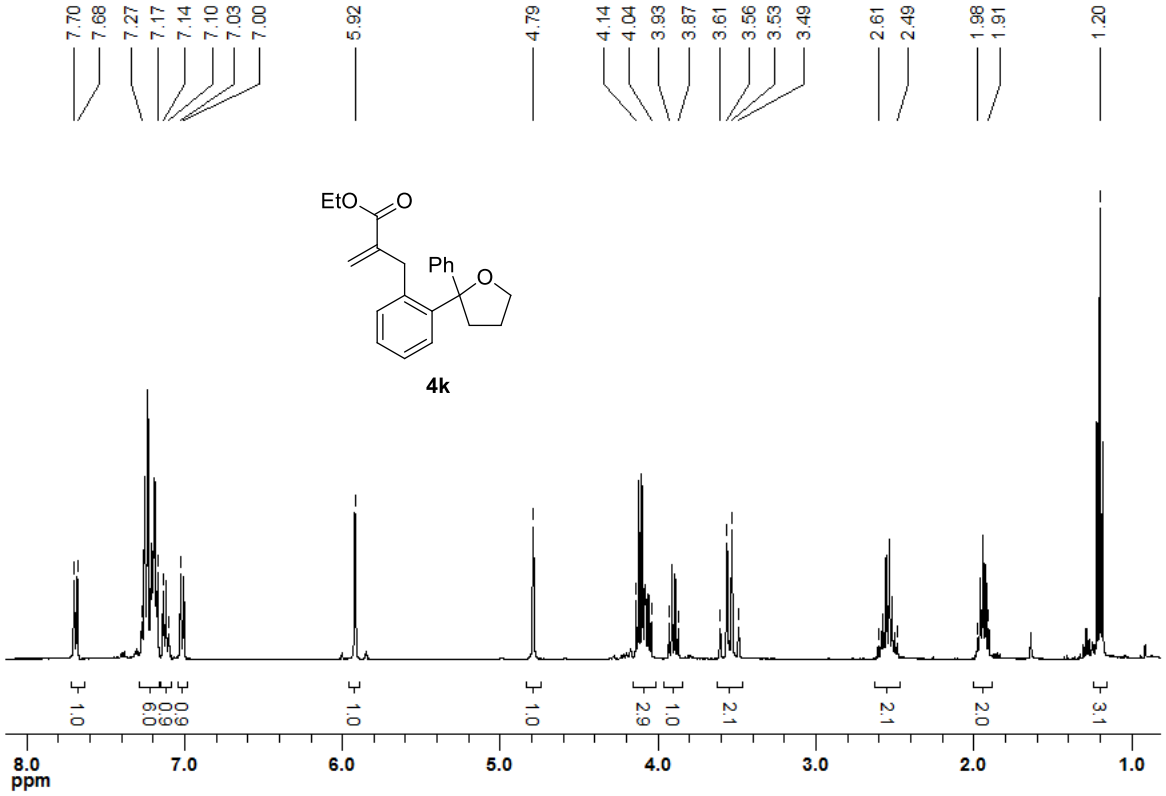
^{1}H NMR, 500 MHz, $CDCl_3$



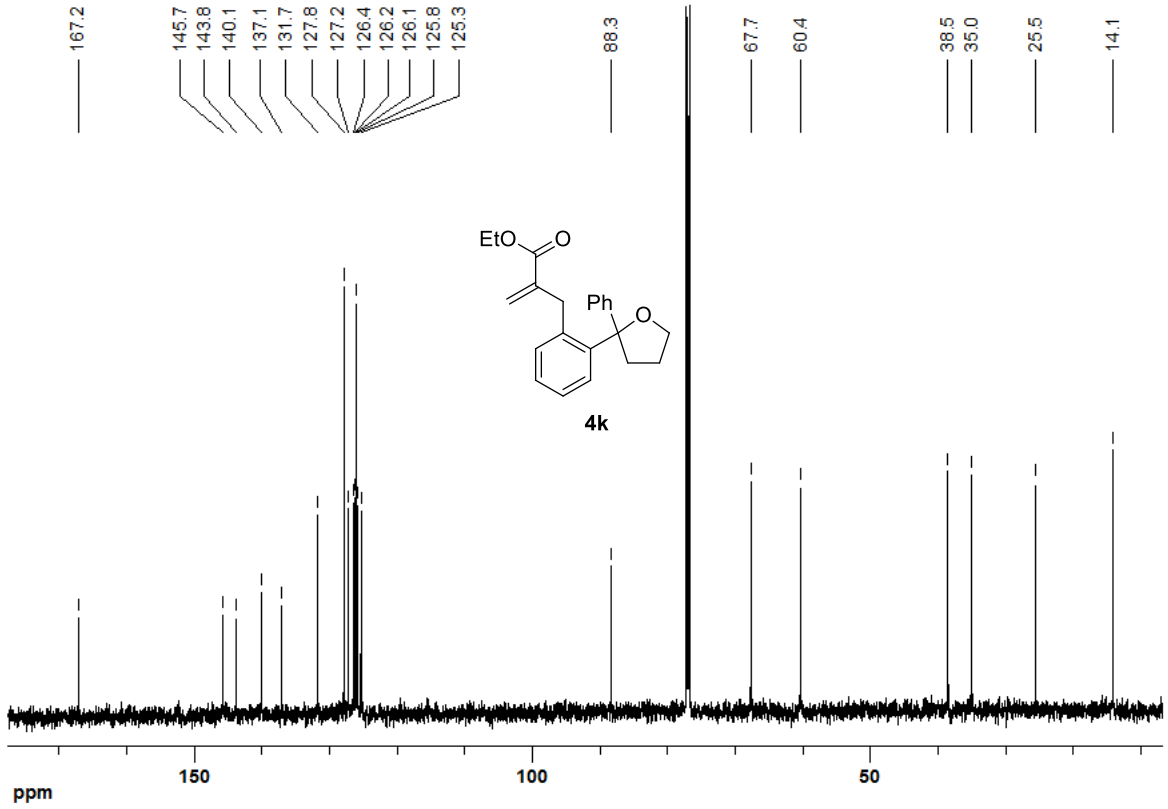
^{13}C NMR, 125 MHz, $CDCl_3$



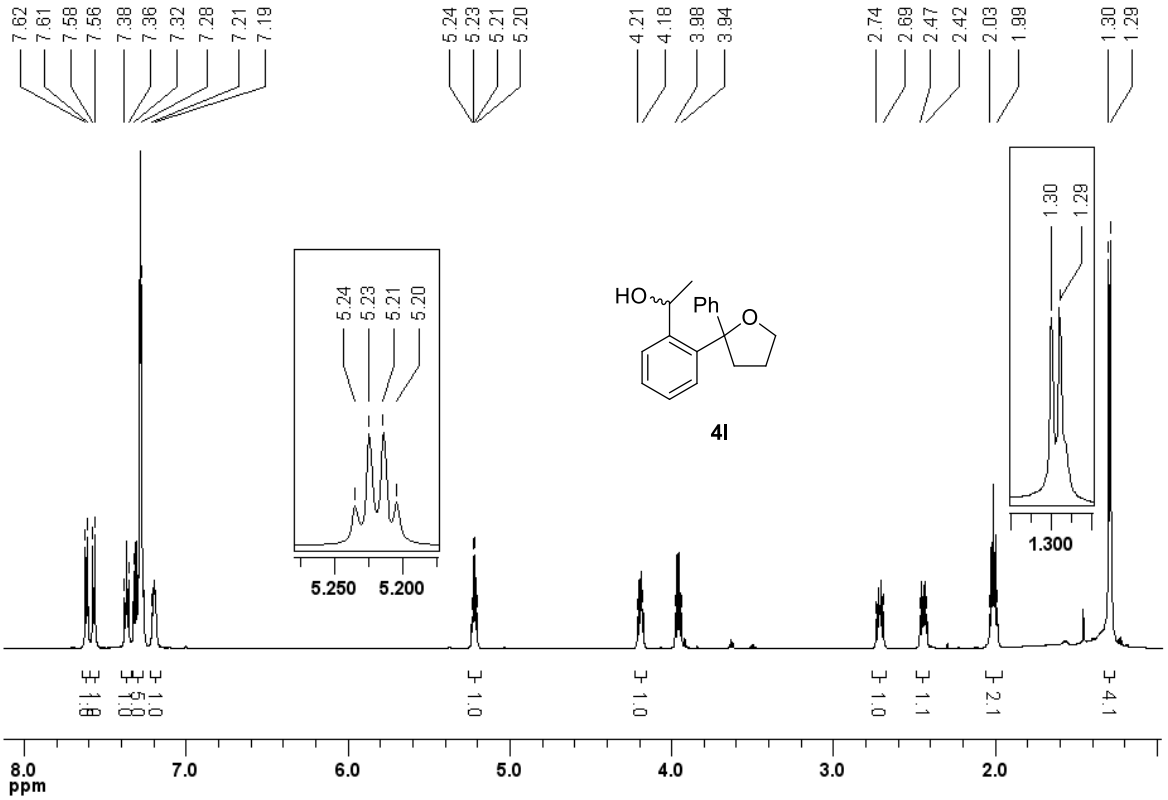
^{1}H NMR, 400 MHz, $CDCl_3$



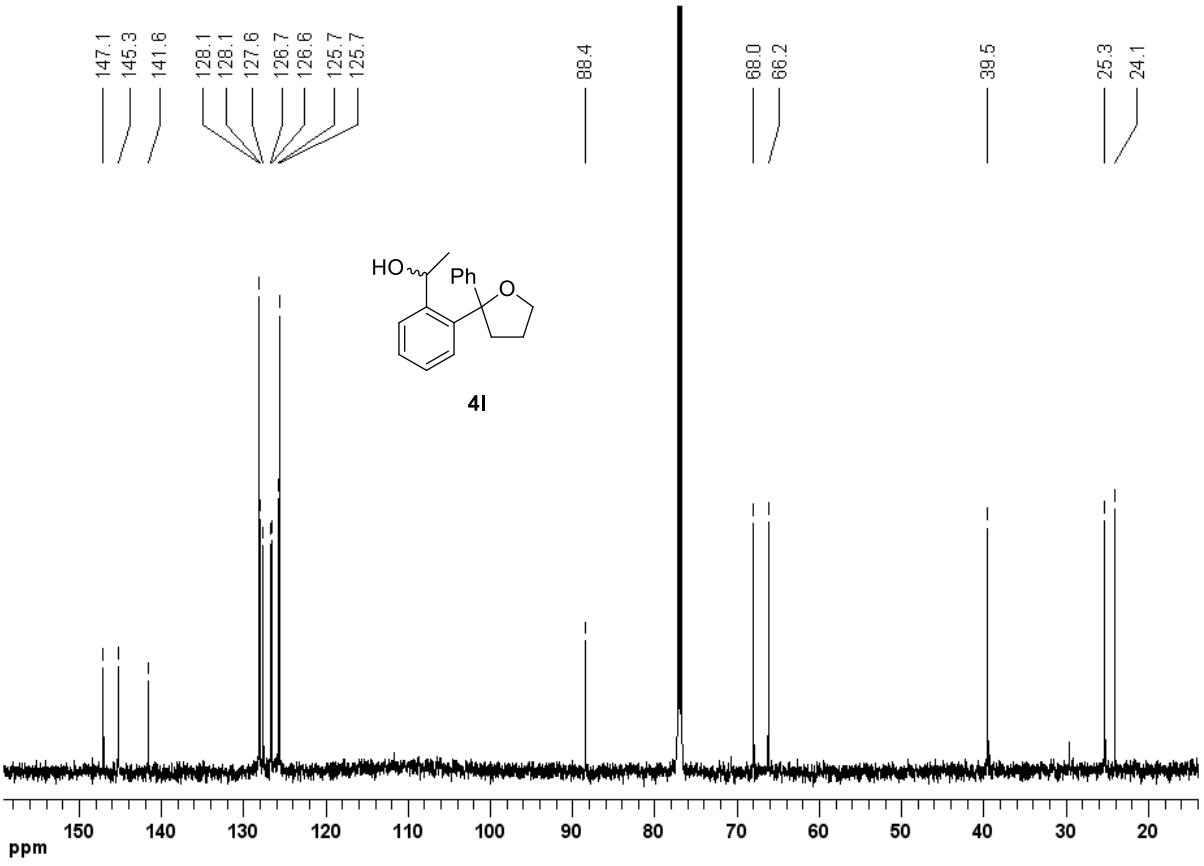
^{13}C NMR, 150 MHz, $CDCl_3$



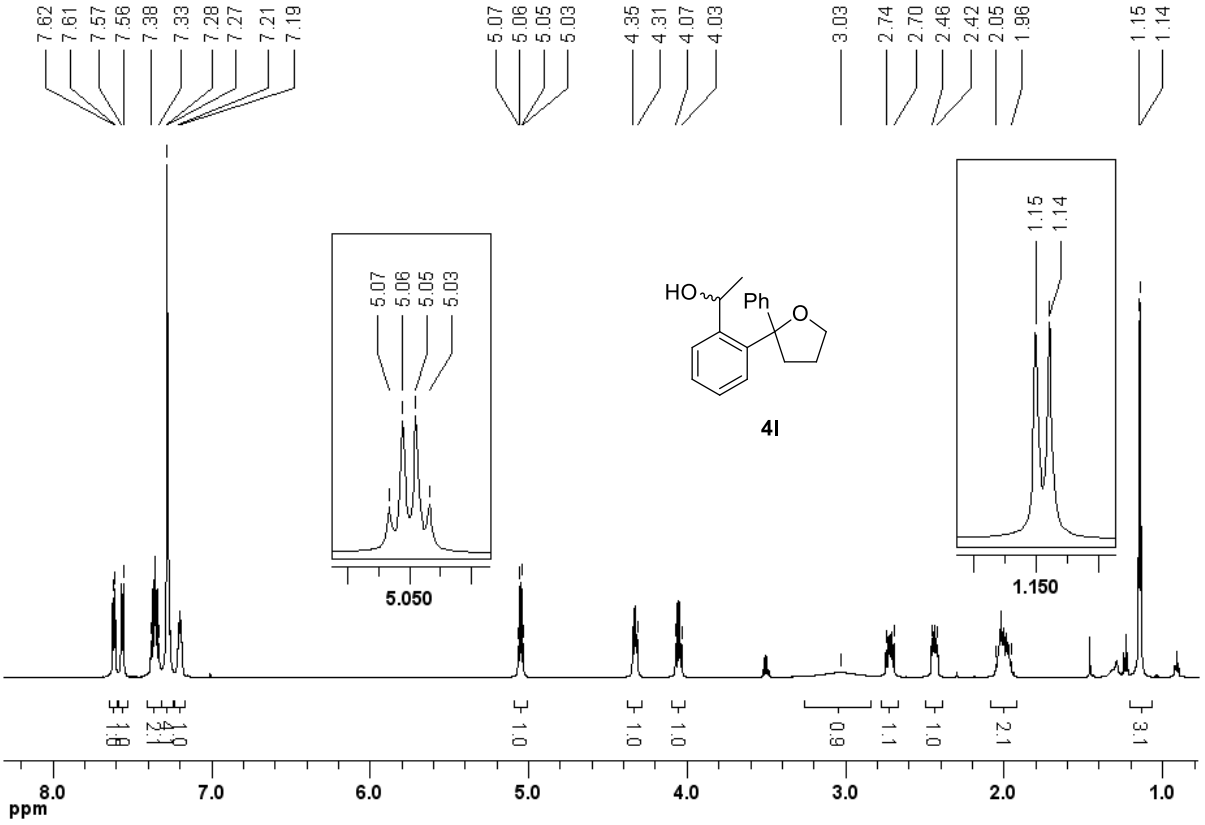
^{1}H NMR, 600 MHz, $CDCl_3$ (minor diastereomer)



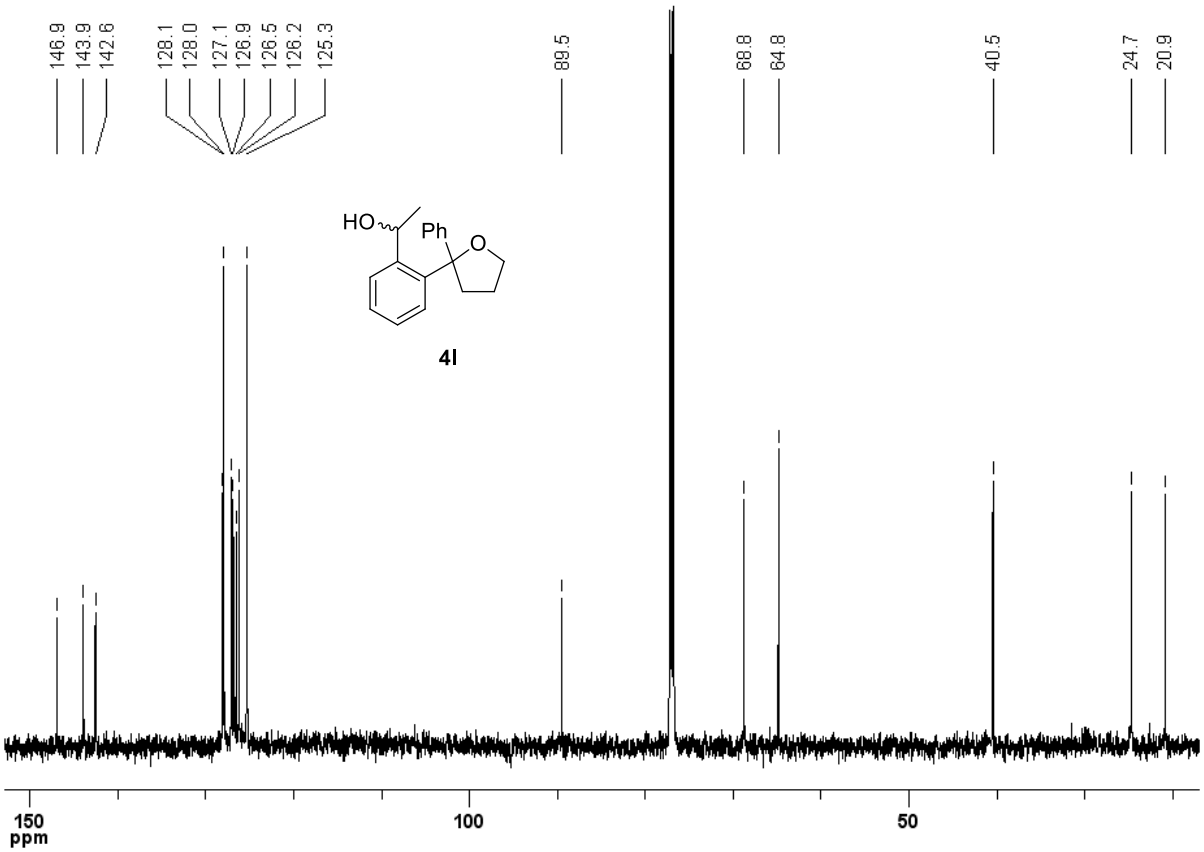
^{13}C NMR, 150 MHz, $CDCl_3$ (minor diastereomer)



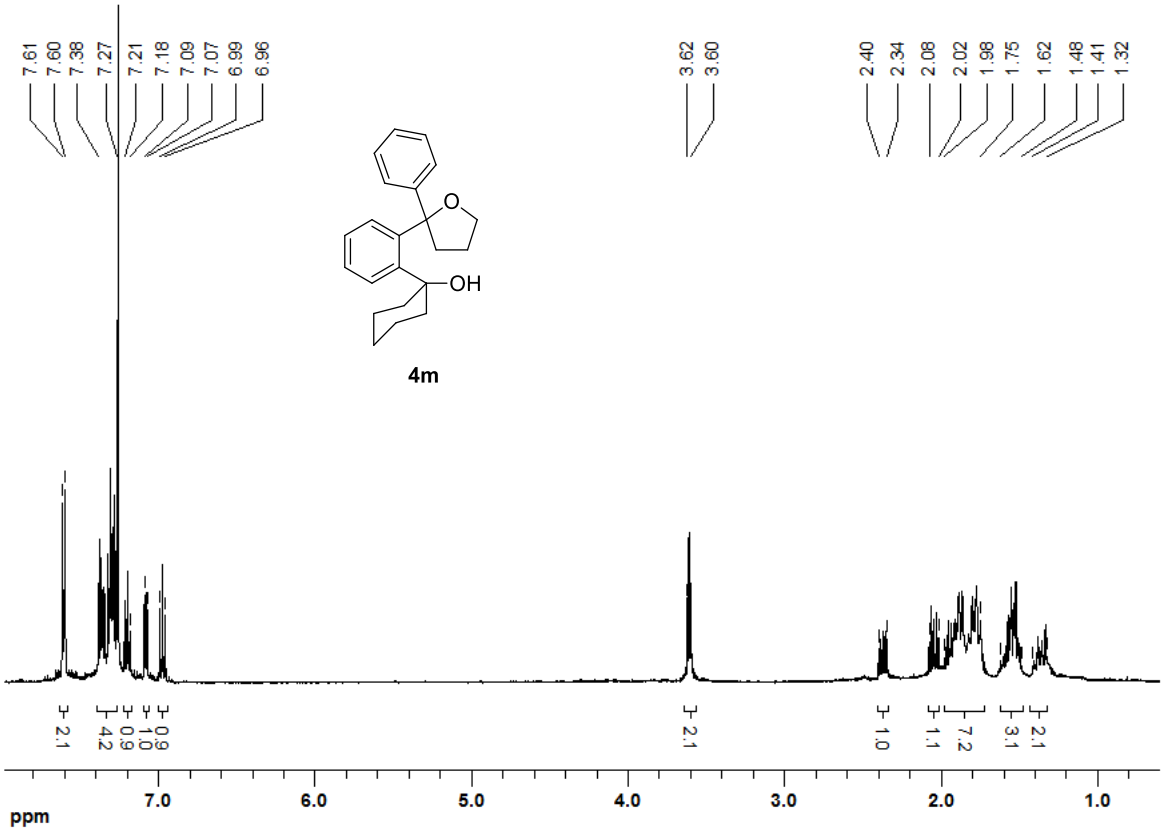
^{1}H NMR, 600 MHz, $CDCl_3$ (major diastereomer)



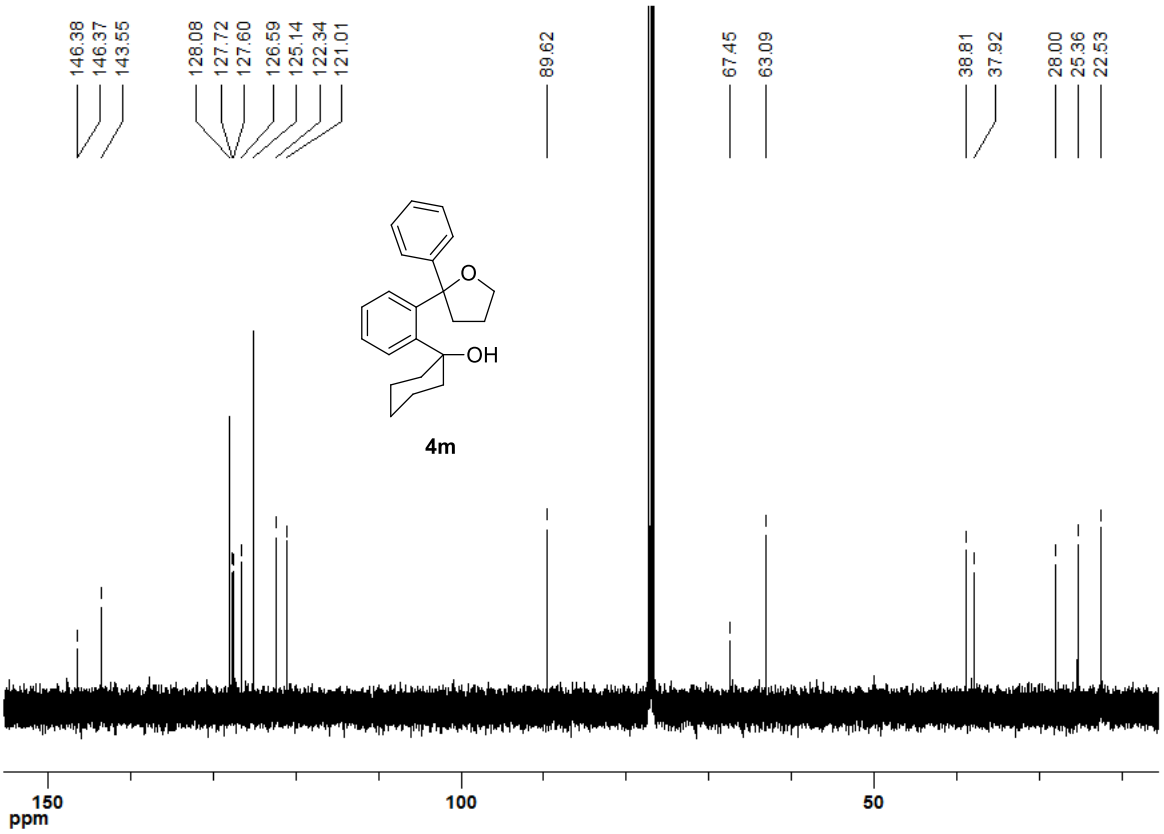
^{13}C NMR, 150 MHz, $CDCl_3$ (major diastereomer)



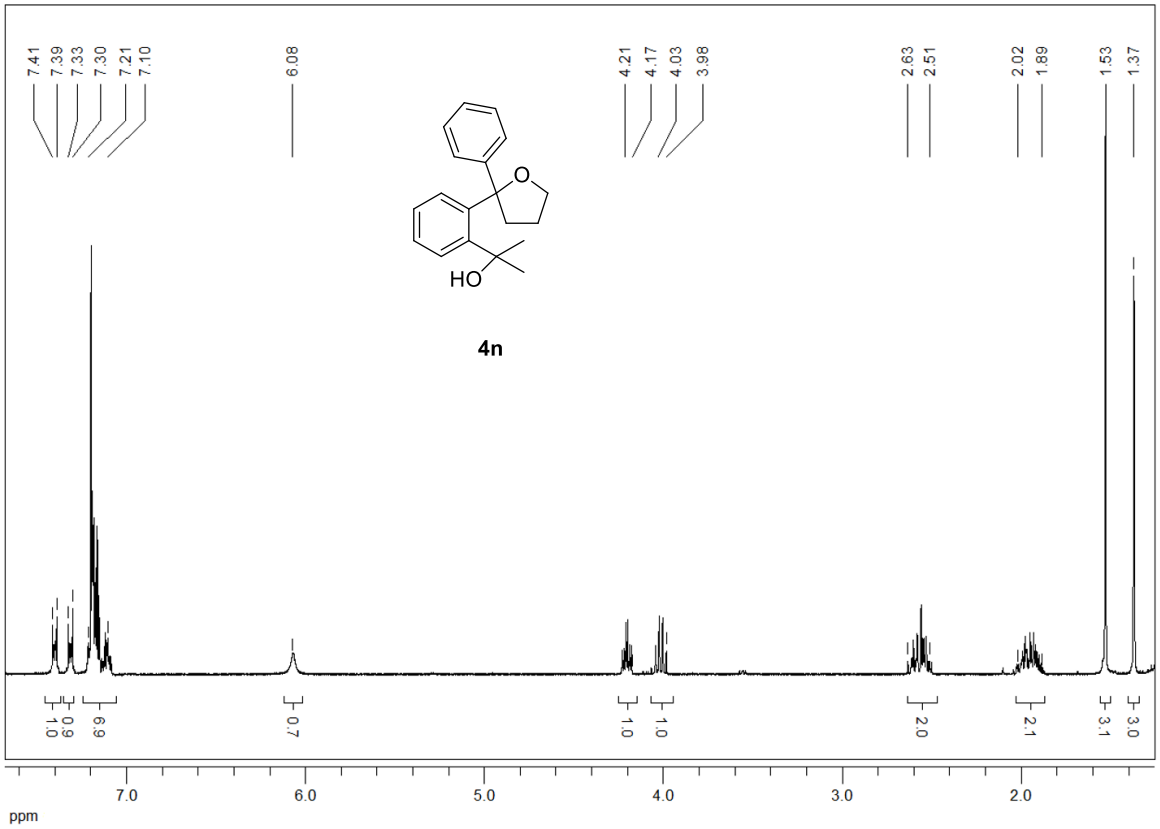
^{1}H NMR, 500 MHz, $CDCl_3$



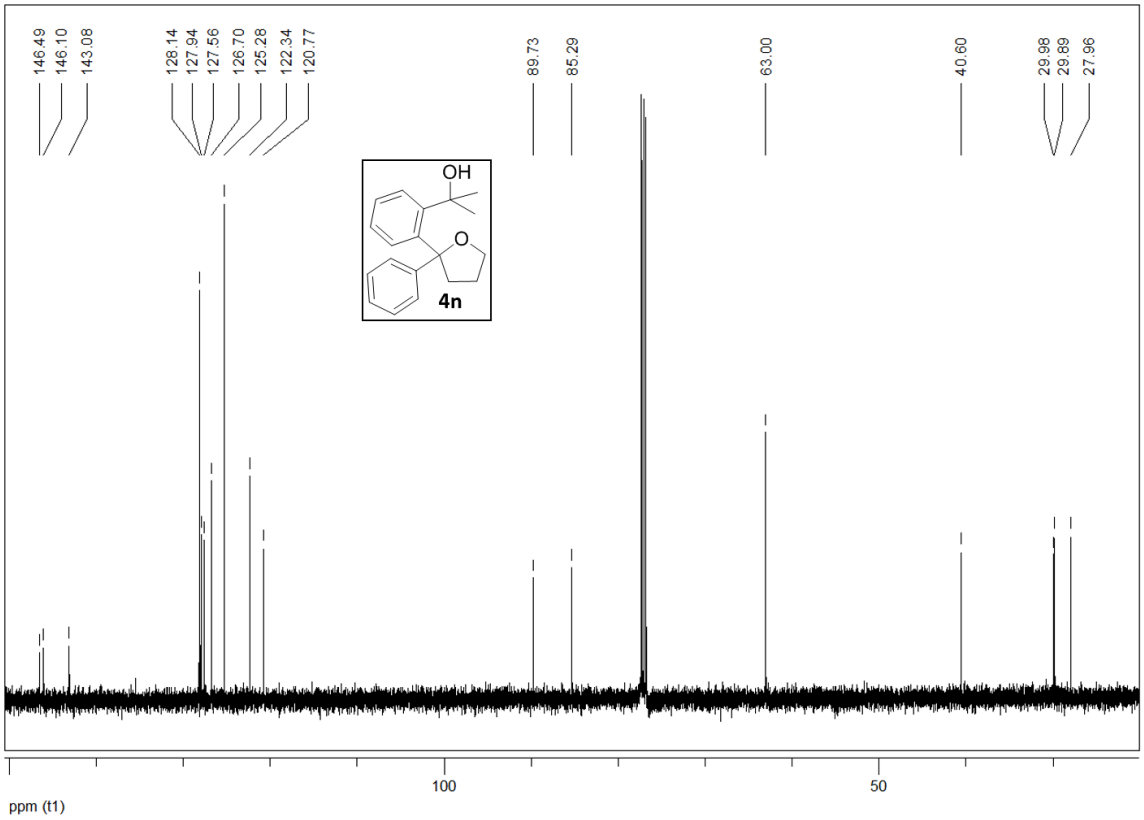
^{13}C NMR, 125 MHz, $CDCl_3$



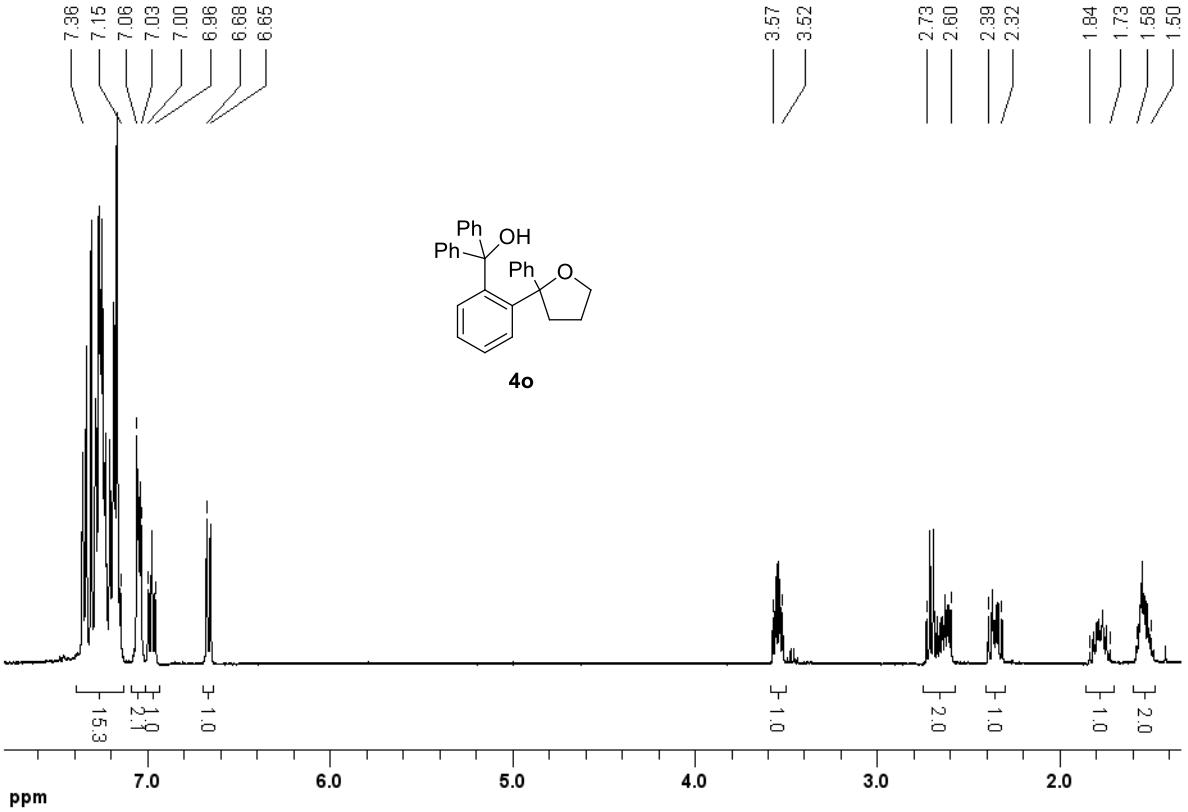
^{1}H NMR, 500 MHz, $CDCl_3$



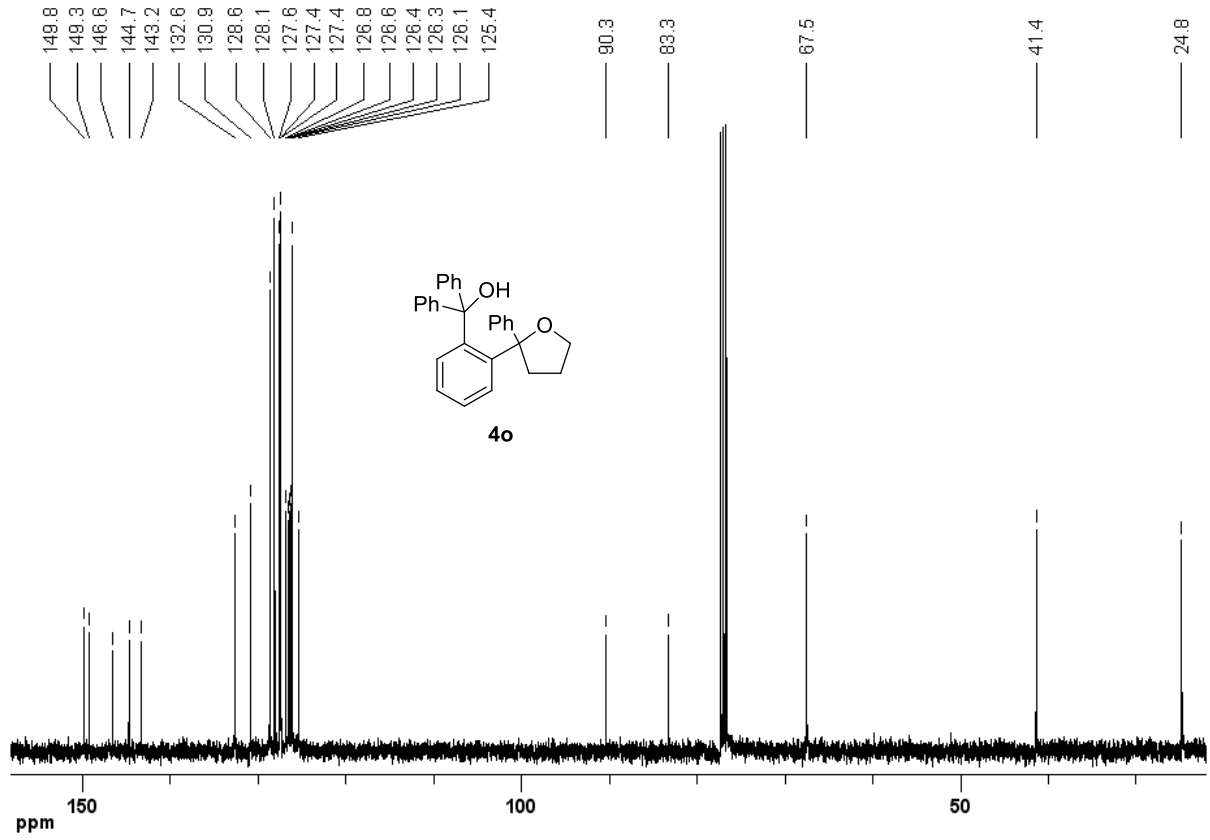
^{13}C NMR, 125 MHz, $CDCl_3$



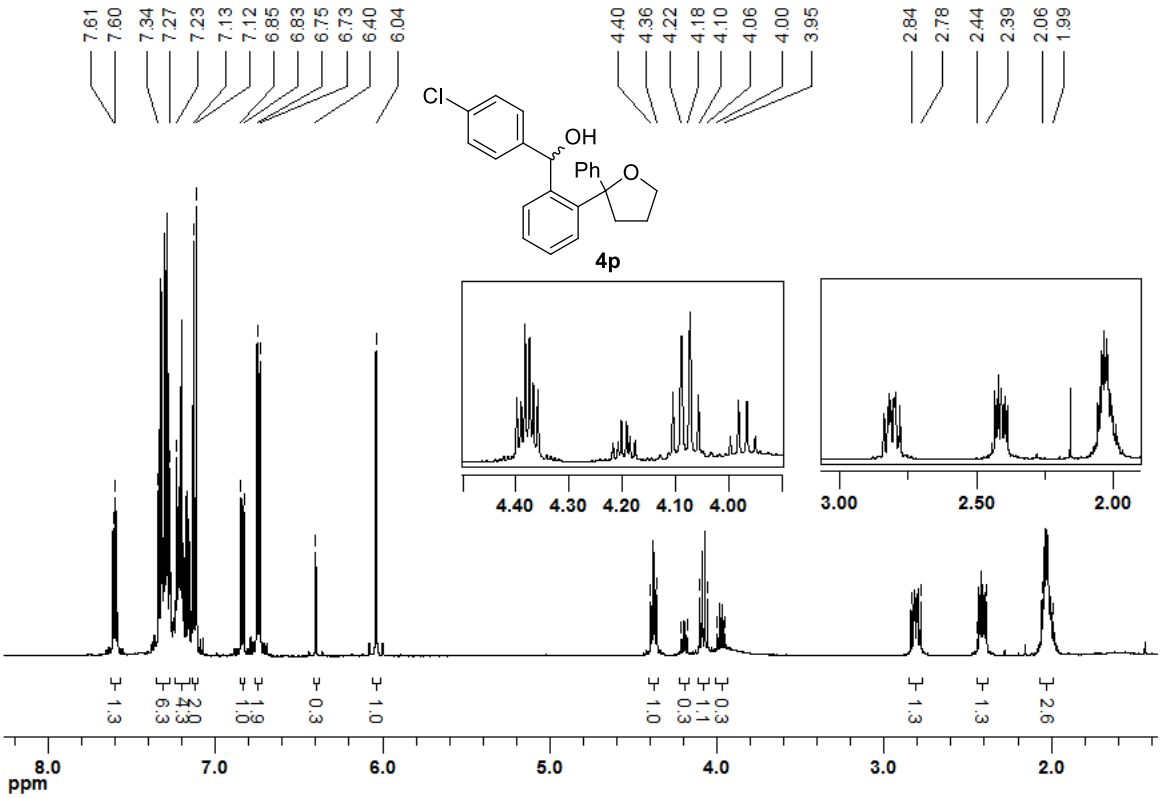
^{1}H NMR, 600 MHz, $CDCl_3$



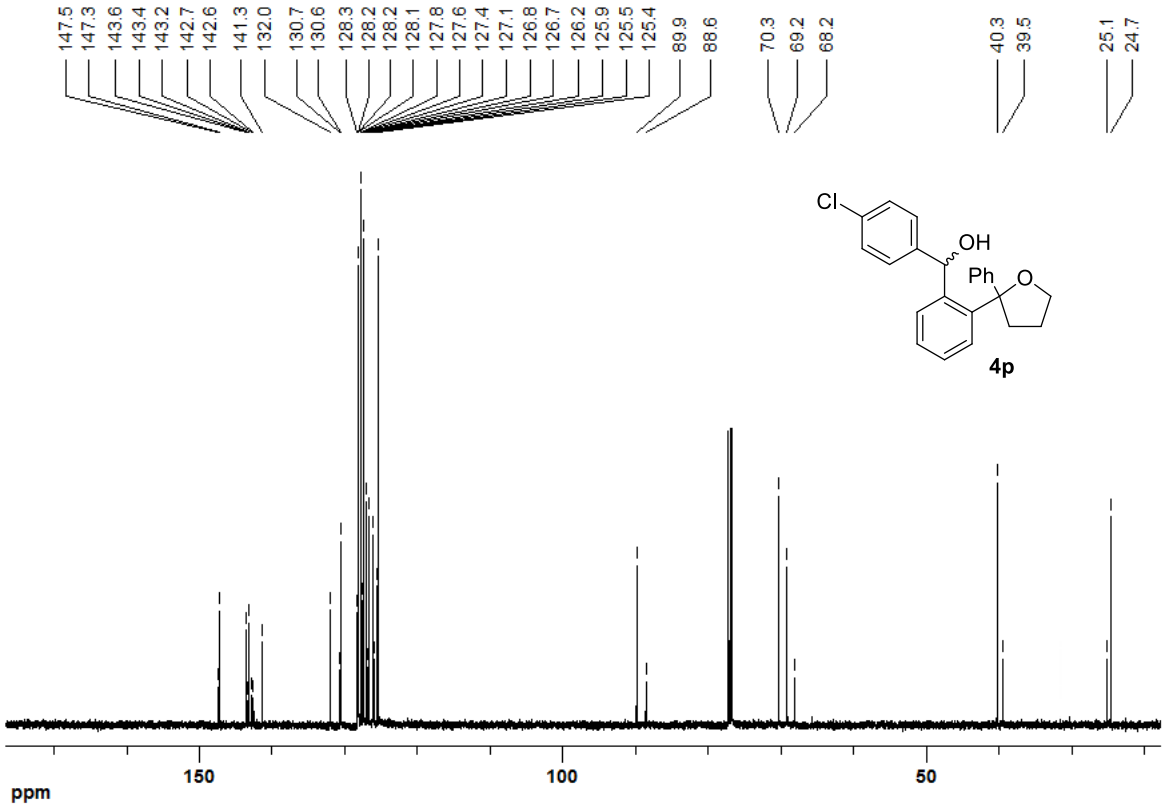
^{13}C NMR, 150 MHz, $CDCl_3$



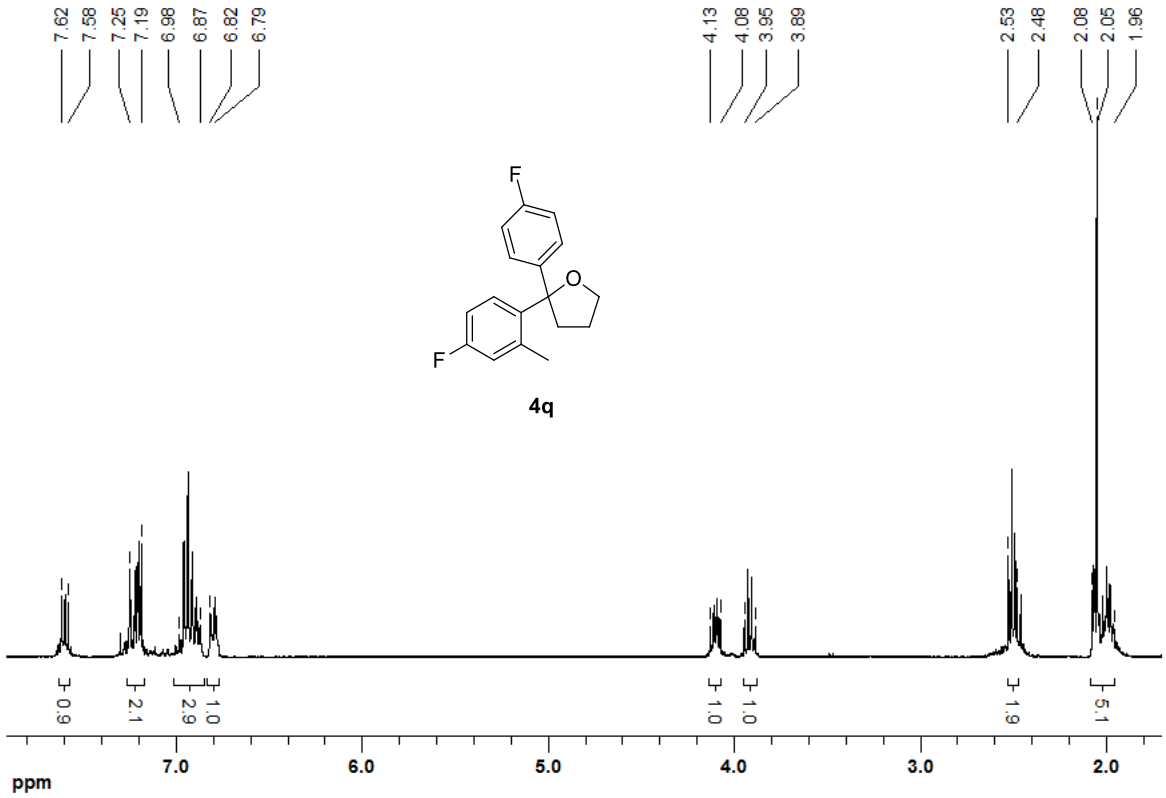
^{1}H NMR, 500 MHz, $CDCl_3$



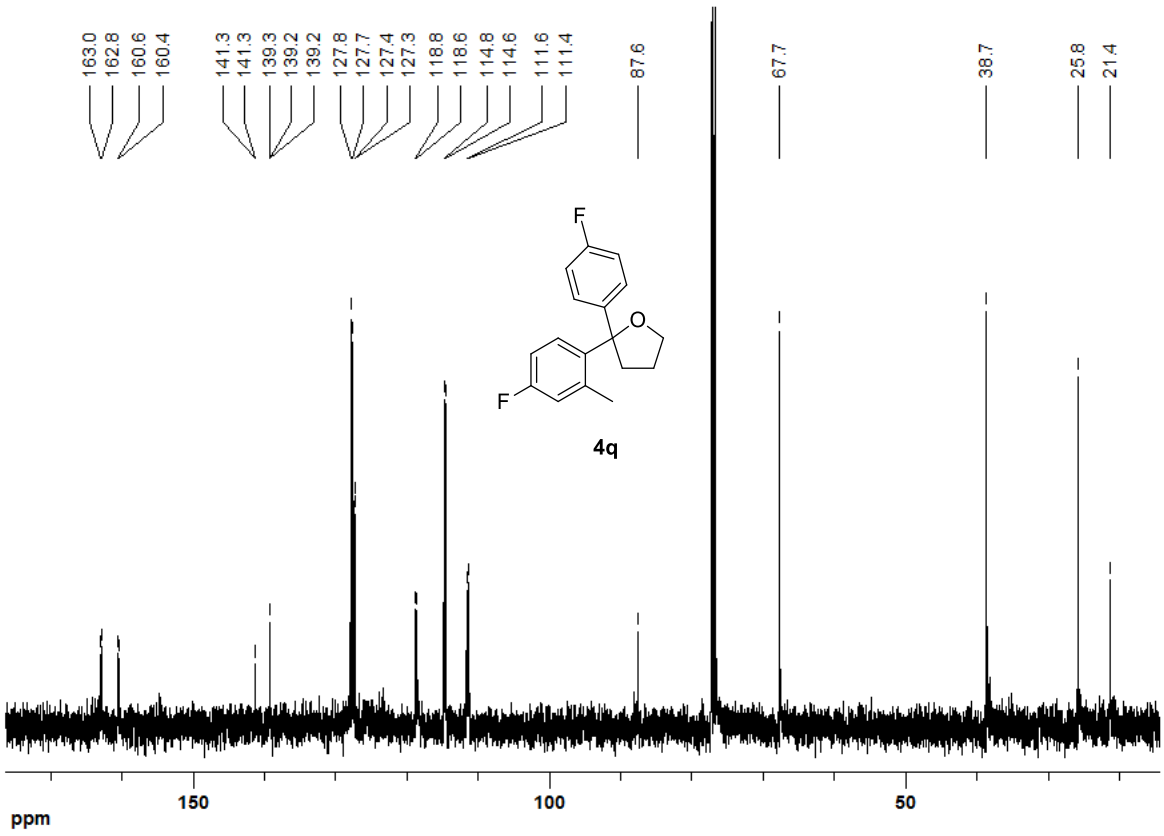
^{13}C NMR, 125 MHz, $CDCl_3$



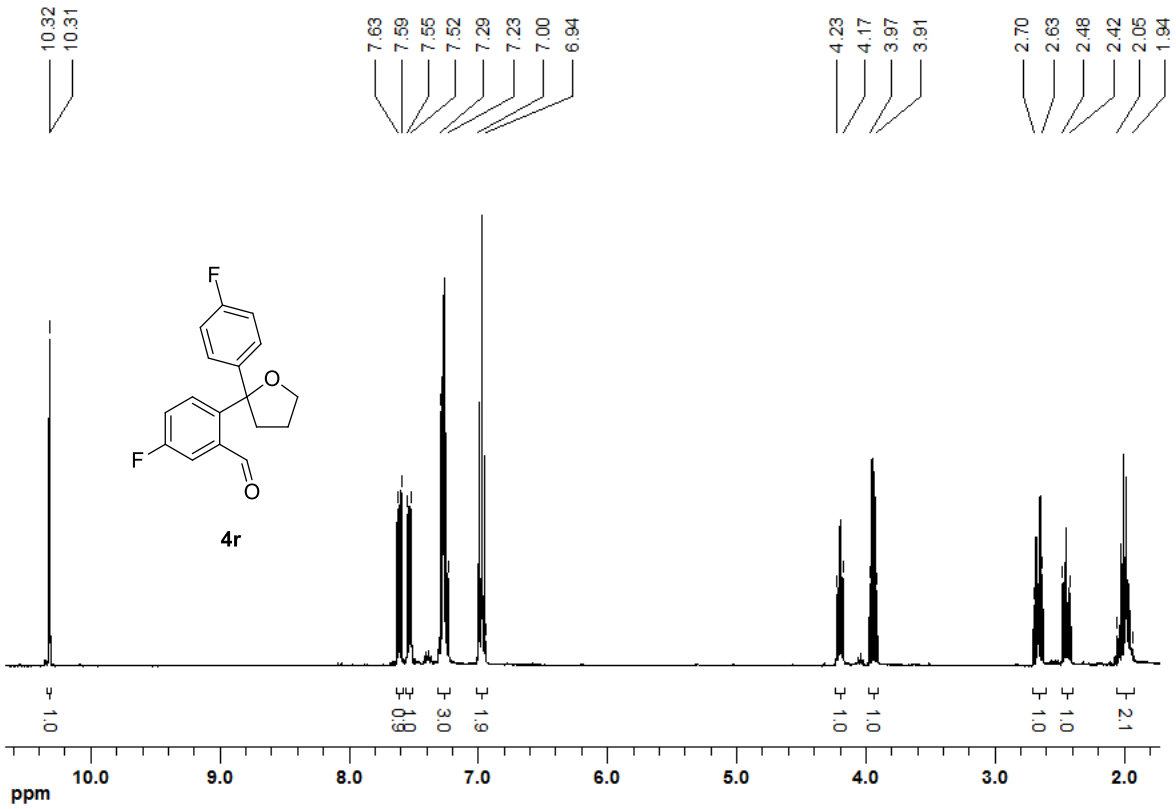
^{1}H NMR, 400 MHz, $CDCl_3$



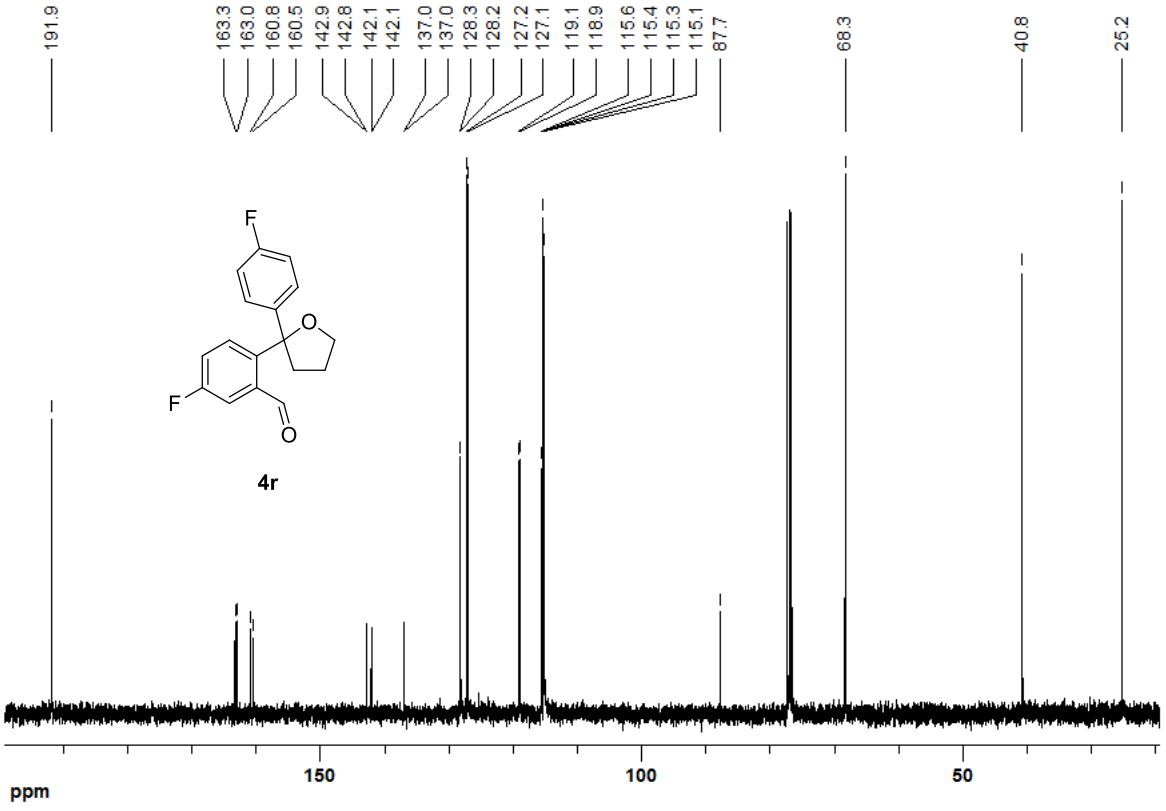
^{13}C NMR, 100 MHz, $CDCl_3$



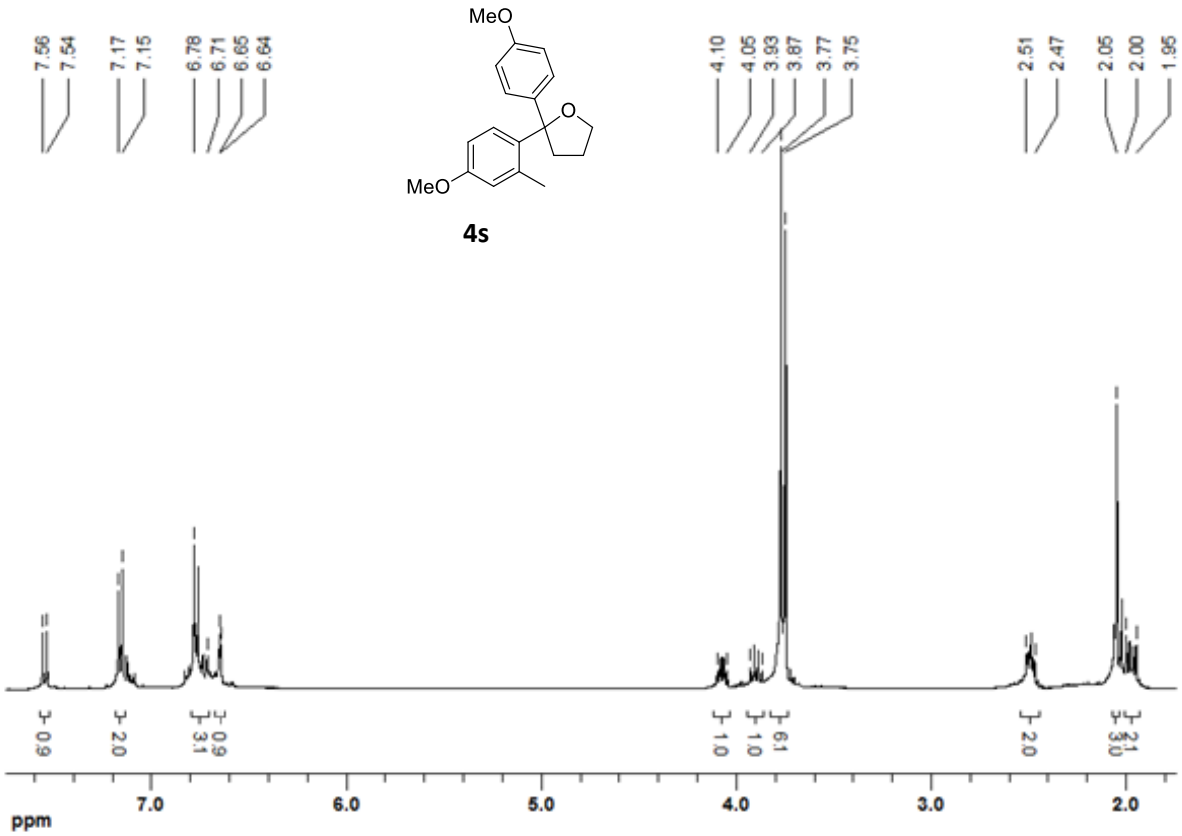
^{1}H NMR, 400 MHz, $CDCl_3$



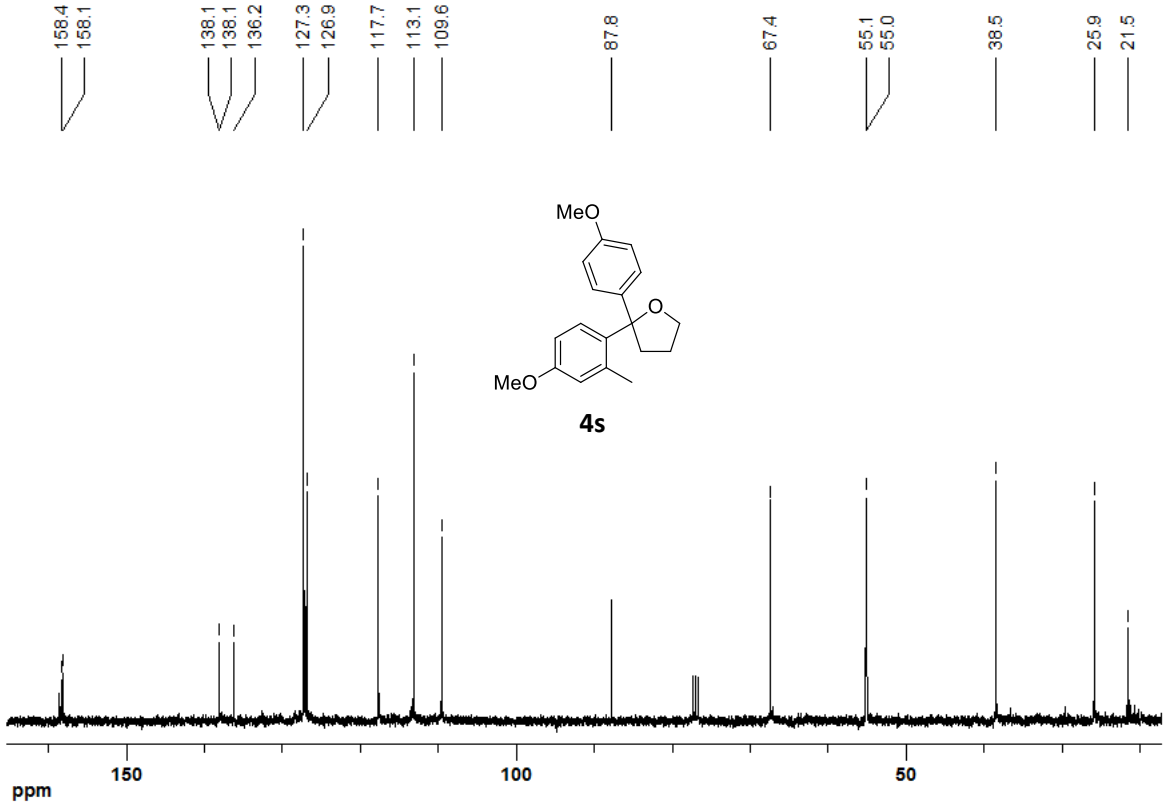
^{13}C NMR, 100 MHz, $CDCl_3$



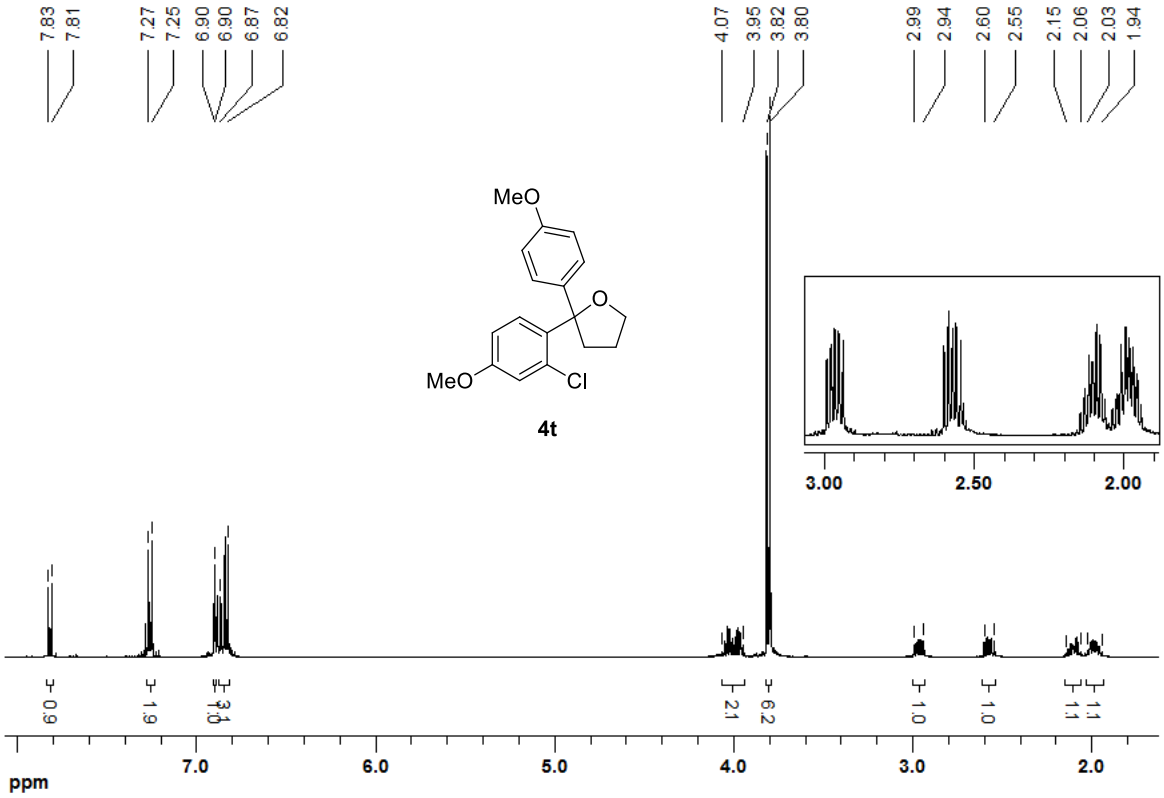
^{1}H NMR, 400 MHz, $CDCl_3$



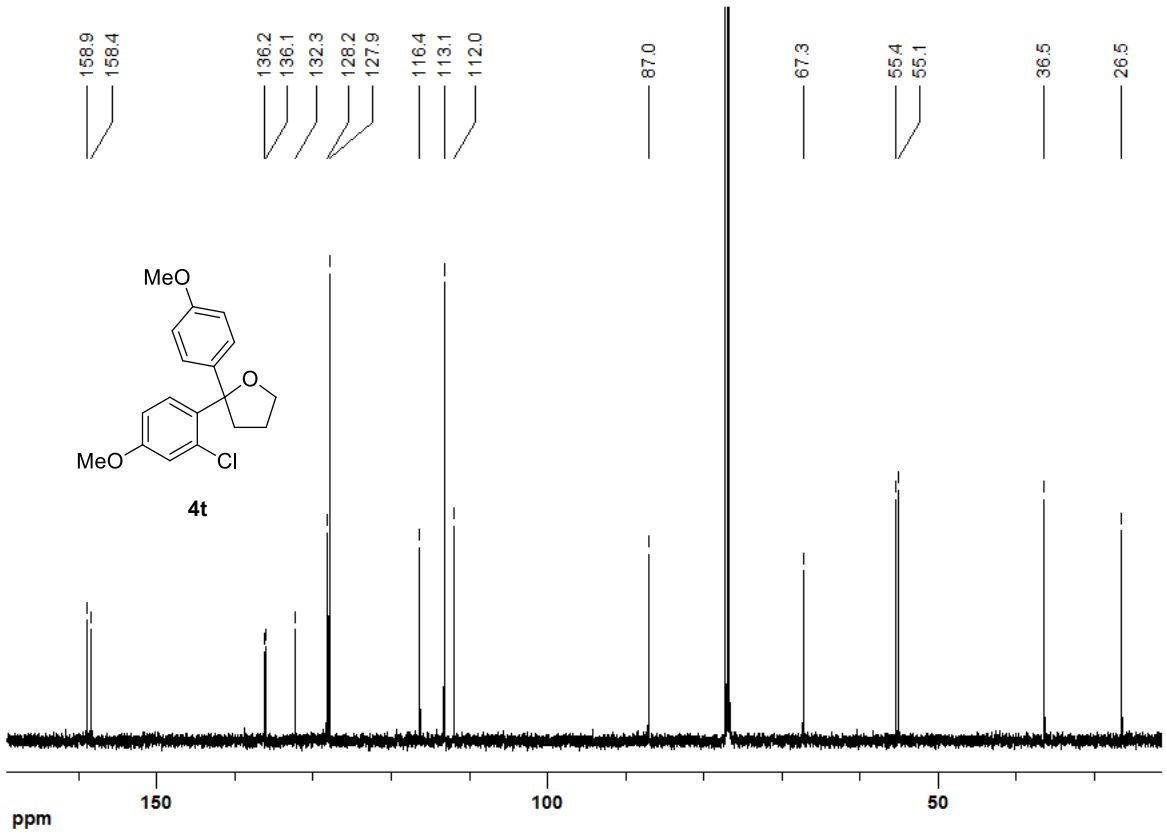
^{13}C NMR, 100 MHz, $CDCl_3$



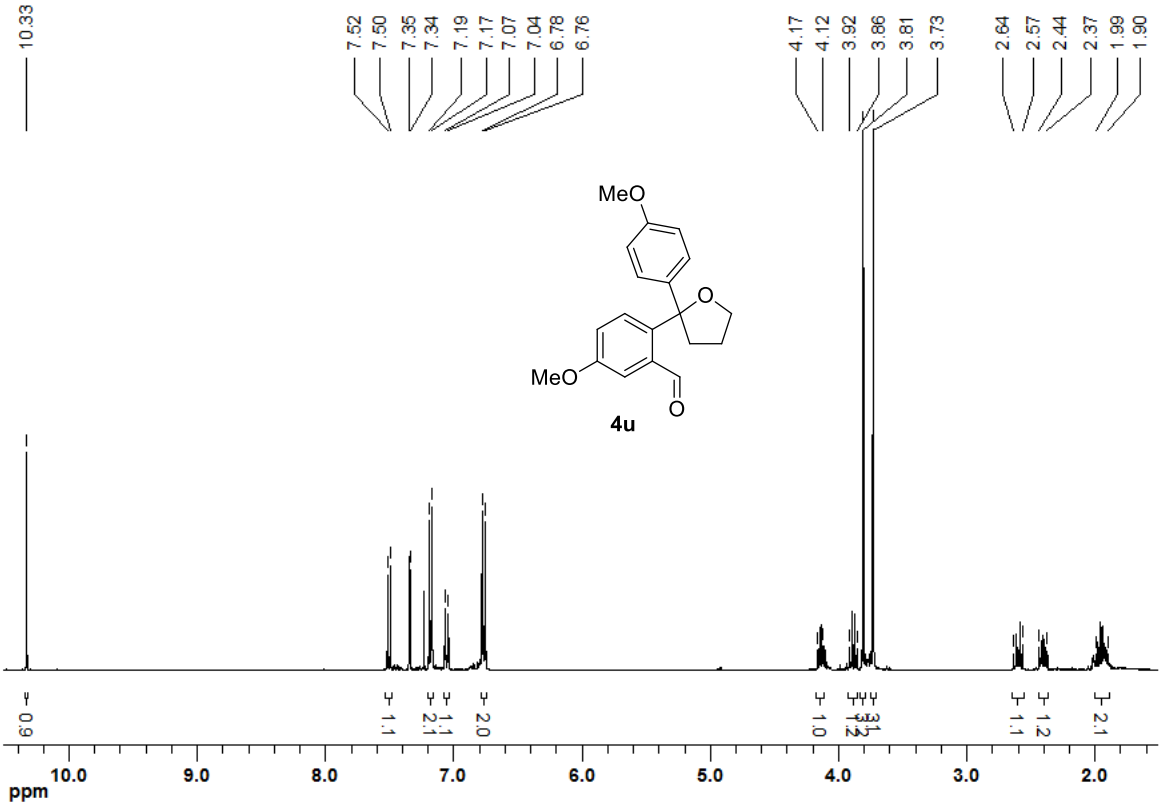
^{1}H NMR, 500 MHz, $CDCl_3$



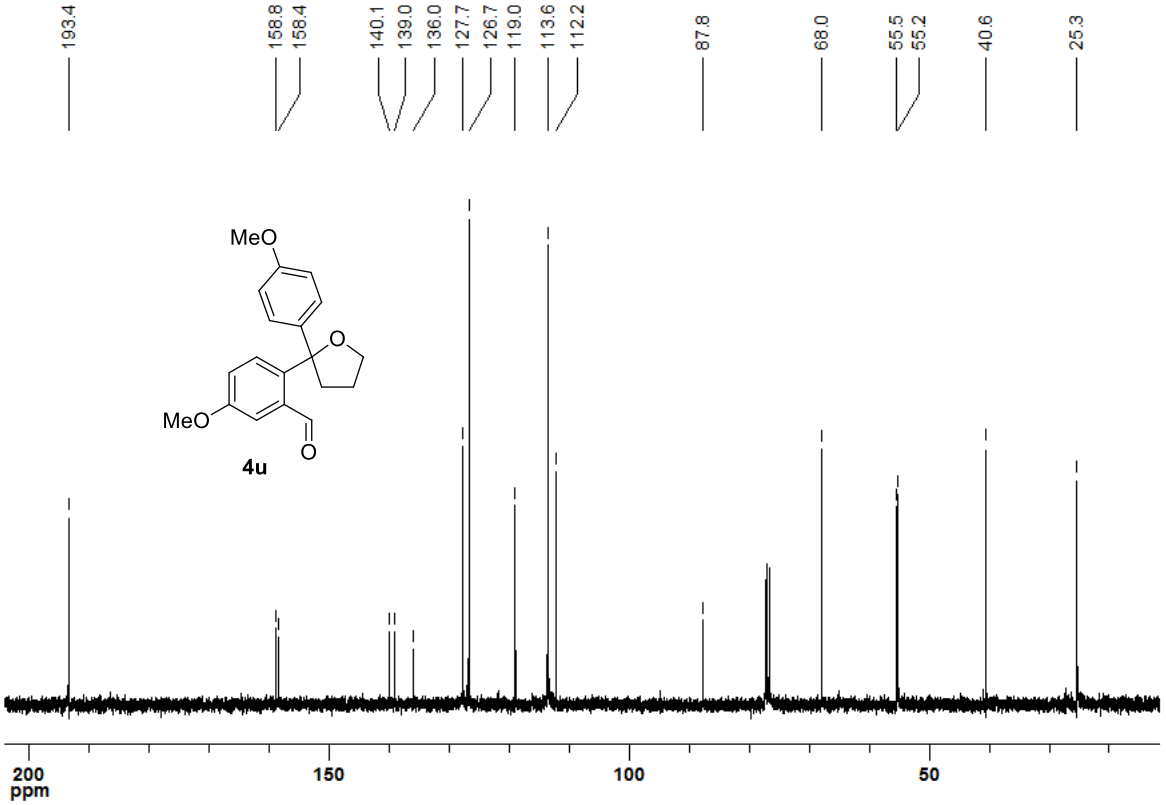
^{13}C NMR, 125 MHz, $CDCl_3$



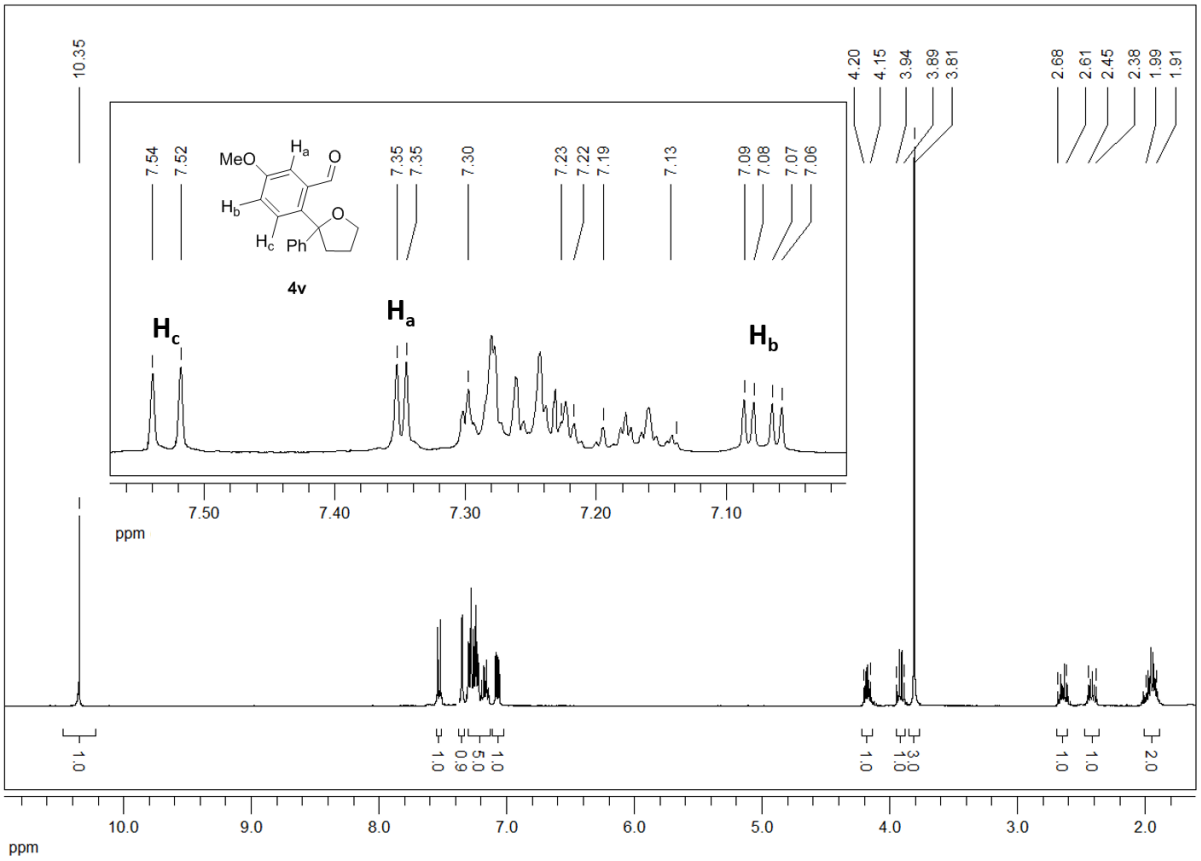
^{1}H NMR, 400 MHz, $CDCl_3$



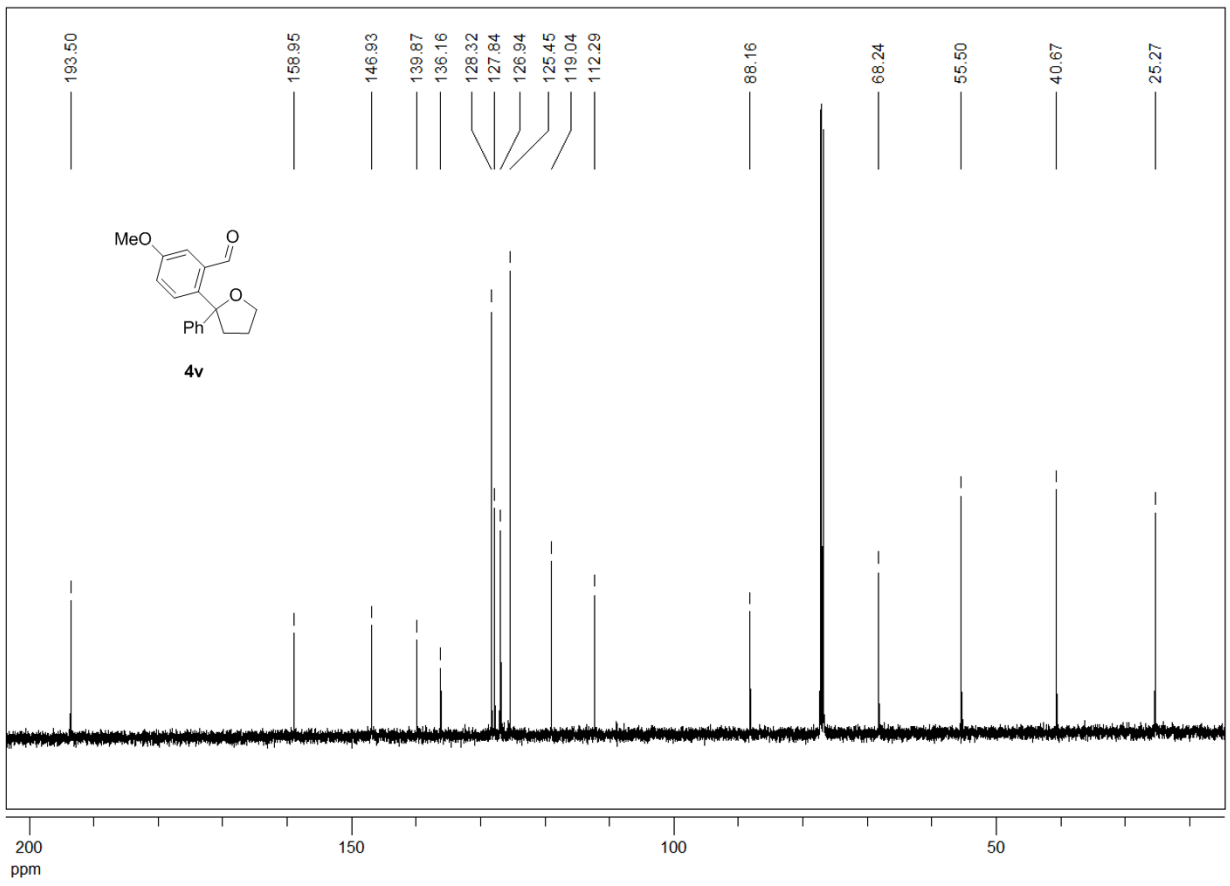
^{13}C NMR, 150 MHz, $CDCl_3$



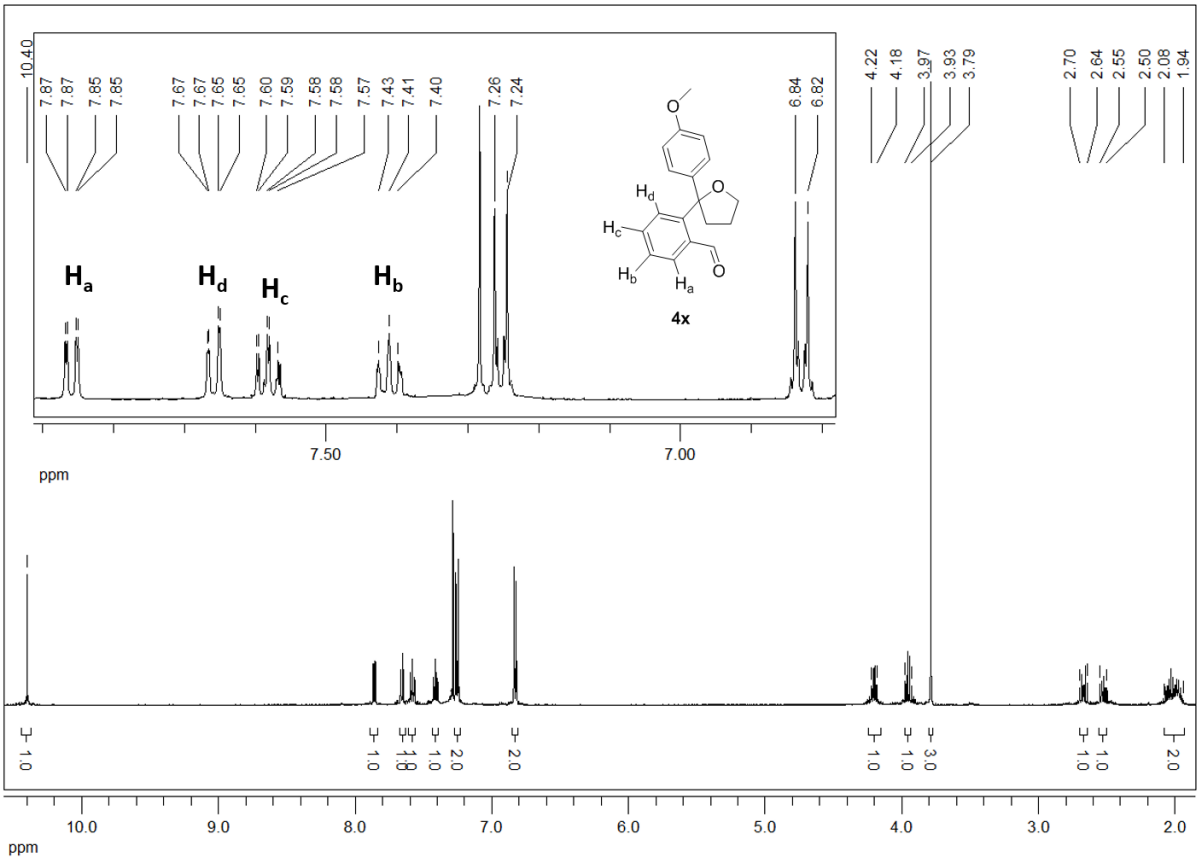
^{1}H NMR, 400 MHz, $CDCl_3$



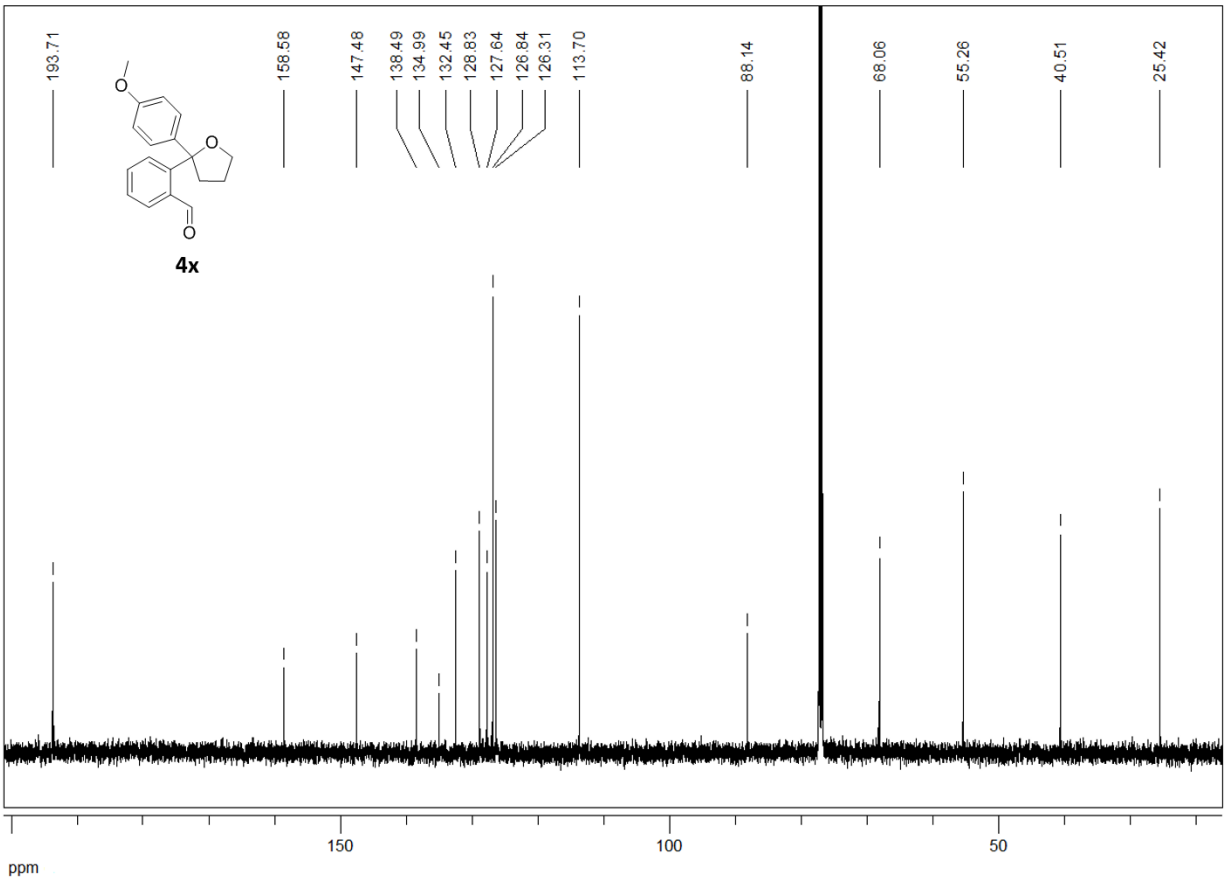
^{13}C NMR, 125 MHz, $CDCl_3$



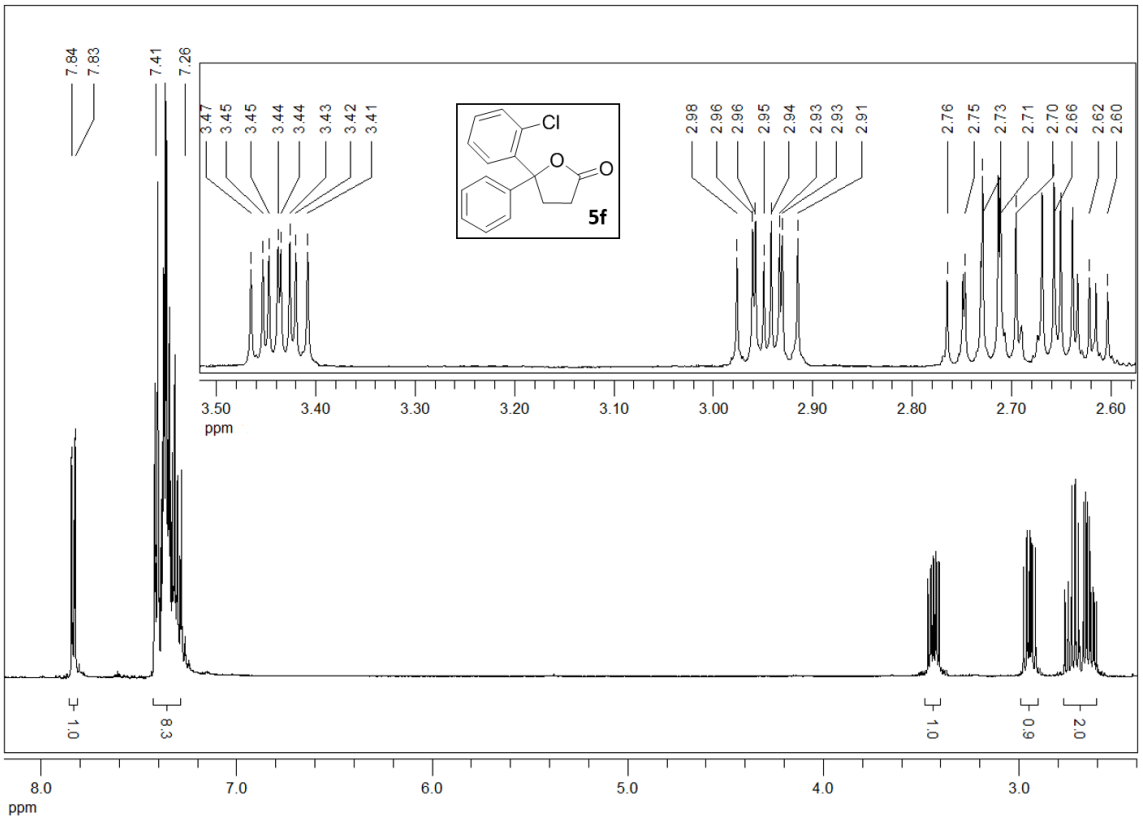
^{1}H NMR, 500 MHz, $CDCl_3$



^{13}C NMR, 125 MHz, $CDCl_3$



^{1}H NMR, 500 MHz, $CDCl_3$



^{13}C NMR, 125 MHz, $CDCl_3$