High-Throughput Optimization of the C–H Arylation of Oxetanes via Ni/Aldehyde Photocatalysis

Justin B. Diccianni*, Bo Hao‡, Wei Liu‡, Iulia I. Strambeanu

Chemical Capabilities, Analytical and Purification, Global Discovery Chemistry, Therapeutics Discovery, Janssen Research & Development, LLC, Welsh & McKean Roads, Spring House, Pennsylvania 19477, United States

Supporting Information

I. General Experimental Information

General Information. All reagents and starting materials were purchased and used as received without further purification. Solvents were used from sealed anhydrous bottles. Catalysts were purchased from commercial sources and stored inside of an MBraun glovebox (O_2 <0.1 ppm and H_2O <0.1 ppm) High-throughput experiments were set up inside of the glovebox, in aluminum 24- or 96-position reactor blocks (Analytical Sales & Services, SKU 24253 and SKU 96973) filled with 1 mL borosilicate vials (Analytical Sales & Services, 8 x 30 mm, flat bottom, SKU 84001) containing 4.80 mm parylene coated magnetic stir bars (Analytical Sales & Services, SKU 13258) and sealed with a film of PFA supported by a rubber mat and an aluminum backed screw-down plate cap, while stirring and heating were achieved by tumble stirring (V&P Scientific Magnetic Tumble Stirrer, 500 rpm) and heating on a temperature-controlled heating block. Liquid dispensing was achieved using standard single channel and multi-channel pipettes without additional calibration. Slurry transfer was achieved in parallel using a multi-channel pipette and clipped pipette tips to permit transfer of heterogenous mixtures. Scale-up reactions were performed in standard lab glassware or disposable borosilicate vials with stirring. Purified compounds were obtained in >95% purity, as assessed by UPLC-MS and/or ^{1}H NMR, unless otherwise indicated. Analytical thin layer chromatography (TLC) was performed using 250 μm Silica Gel 60 F254 pre-coated plates. Chemical shifts (δ) are reported in parts per million (ppm) downfield relative to tetramethylsilane (TMS, 0.0 ppm), $CDCl_3$ (7.26 ppm for ^{1}H NMR, 77.23 ppm for ^{13}C NMR) or $(CD_3)_2SO$ (2.50 ppm for ^{1}H NMR, 39.58 ppm for ^{13}C NMR). Ratios of diastereomers were determined by ^{1}H NMR analysis of the crude reaction mixtures before purification. In reactions with substituted oxetanes, the relative stereochemistry of the major diastereomers was determined by analysis of coupling constants. Coupling constants (J) are reported in Hz. Multiplicities are reported using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; b, broad.

Instrumentation. Irradiation of all reactions was performed with Analytical Sales and Services Lumidox II equipment – 24- and 96-position LED arrays (lens mat with active cooling, λ=375 nm, optical power = stage 1). Analytical samples were performed on a Waters Acquity I-Class Ultra-High Pressure Liquid Chromatography-Mass Spec with a PDA detector and SQD2 mass-detection (ESI-pos/neg). Charged-Aerosol Detection was performed using a Waters Acquity I-Class UPLC-MS equipped with PDA and SQD2 detectors (ESI positive ionization mode); column: Waters CSH C18, 2.1mm x 50 mm (Waters # 186006101) with Waters ACQUITY UPLC Col. In-Line Filter Kit (Waters #205000343); Mobile Phase A: 0.1 % trifluoroacetic acid in MilliQ water, Mobile Phase B: 0.1% trifluoroacetic acid in Optima grade Acetonitrile. Corona Aerosol Detection (CAD) analysis was conducted with Thermo Corona™ Veo™ RS Charged Aerosol Detector. The flow rate was 2 mL/min using a gradient from 5-100% Acetonitrile over 2 minutes. The total run time for each injection was 2.3 minutes. Flash column chromatography was performed using an Isco with RediSep silica columns. Proton nuclear magnetic resonance (1H NMR) spectra were recorded using a Bruker AVANCE III HD NanoBay 400 NMR spectrometer. Carbon-13 nuclear magnetic resonance (13C NMR) spectra were recorded using a Bruker AVANCE III HD NanoBay 400 NMR spectrometer at 101 MHz. Reverse phase purification was performed using a Waters mass-triggered liquid chromatography using Waters XSelect CSH (C18, 5u, 19x100 mm) or Waters XBridge BEH (C18, 5u, 19x100 mm) reversed-phase columns and appropriate linear gradients of increasing concentration of acetonitrile in water, 0.1% TFA or FA. Fractions

containing the desired product were combined and concentrated by centrifuge evaporation. High-throughput NMR data were collected using a Bruker AVANCE III HD solution-state NMR spectrometer equipped with a room temperature 1 mm TXI Microprobe at 400 MHz proton frequency at 298 K. The pulse sequence used was a one-dimensional (1D) proton experiment with water presaturation (zgpr). The acquisition time was 2.04 s using 32768 points for 20 ppm sweep width. High resolution mass spectra (HRMS) are reported as m/z (relative ratio). An Agilent 1260 Infinity Series was used for ESI-TOF analysis (Agilent model 6230). Accurate m/z are reported for the molecular ion [M+H]+ unless stated otherwise.

II. General Procedures

General Procedure A. General procedure for oxetane arylation via HTE

In a nitrogen-filled glovebox, a 96-well plate of 1 mL vials was charged with base (20 μmol, 2 equiv) and Ni catalyst (1 μmol, 0.1 equiv) as solids. A solution of aldehyde (5 μmol, 0.5 equiv) or other HAT reagent was added to the wells in the chosen solvent. A solution of aryl halide (10 μmol, 1 equiv) and oxetane (6.5 μmol, 10 equiv) in the chosen solvent was added to the wells. The plate was sealed, removed from the glovebox, and irradiated with 375 nm light on a 96 well plate Lumidox® II at power level 1 (50 mW) for 20 h. A solution of Ph_3N (0.5 mL, 0.01 M) internal standard in MeCN was added to each well and the plate was centrifuged to settle out the solids. Aliquots of 25 mL were taken from the supernatants and diluted with 0.75 mL MeCN. Analysis was performed by UPLC-MS and yields were determined by calibration against an authentic product standard.

General Procedure B. General procedure for preparative scale oxetane arylation

A 1 dram vial was charged with aryl halide (0.2 mmol, 1 equiv) and brought into a nitrogen-filled glovebox. KF (23.2 mg, 0.4 mmol, 2 equiv) was added to the vial. A solution of (dtbbpy)$NiBr_2$ (11.2 mg, 0.02 mmol, 10 mol %) and *p*-cyanobenzaldehyde (13.2 mg, 0.1 mmol, 0.5 equiv) in 2 mL of 3:1 TFT:acetone was added to the vial. Oxetane (130 mL, 2 mmol, 10 equiv) was added then the vial was capped and removed from the glovebox. The reaction mixture was irradiated with 375 nm light on a 24 well plate Lumidox® II at power level 1 (60 mW) for 20 h while being cooled with two fans. The reaction mixture was filtered, and volatiles were removed from the filtrate under vacuum. The residue was purified by chromatography (silica, EtOAc/heptane).

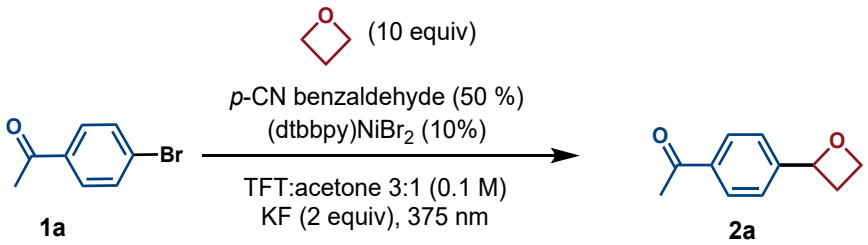
General Procedure C. General procedure for parallel oxetane arylation

In a nitrogen-filled glovebox, to 1-dram vials containing the appropriate aryl halide substrate (0.1 mmol) was added (dtbbpy)NiBr2 4H2O (5.59 mg, 0.01 mmol, 0.1 eq.), 4-cyanobenzaldehyde (6.6 mg, 0.05 mmol, 0.5 eq.), potassium fluoride (11.6 mg, 0.2 mmol, 2 eq.), trifluorotoluene (0.75 mL) and acetone (0.25 mL), followed by oxetane (65 uL, 1 mmol, 10 eq.). The 24-well plate was then sealed, stirred, and irradiated with LED arrays (24 well plate Lumidox® II at power level 1, 60 mW) overnight. Afterwards, the solvent was reduced to ~0.1 mL by Genevac. To each vial was added 0.2 mL of 1,3,5-trimethoxybenzene in d6-DMSO (0.1 M) as the internal standard. After stirring for 10 mins, a 0.1 mL aliquot was taken and filtered into a 96-well plate, followed by 0.2 mL d6-DMSO rinse. The plate was sealed and analyzed by HT-NMR of the crude reaction mixtures.

III. Control reactions

Experiments were set up according to General Procedure A with the modifications indicated in the table. Yields were determined by UPLC calibrated against an authentic product sample.

Table S1. Control reactions



	Deviation from standard conditions	UPLC Yield (%)
1	None	95
2	No ligand	10
3	No Ni	0
4	No aldehyde	0
5	No base	50
6	1 equiv TEMPO added	13
7	No light	0
8	445 nm	0
9	405 nm	23
10	395 nm	82
11	365 nm	42

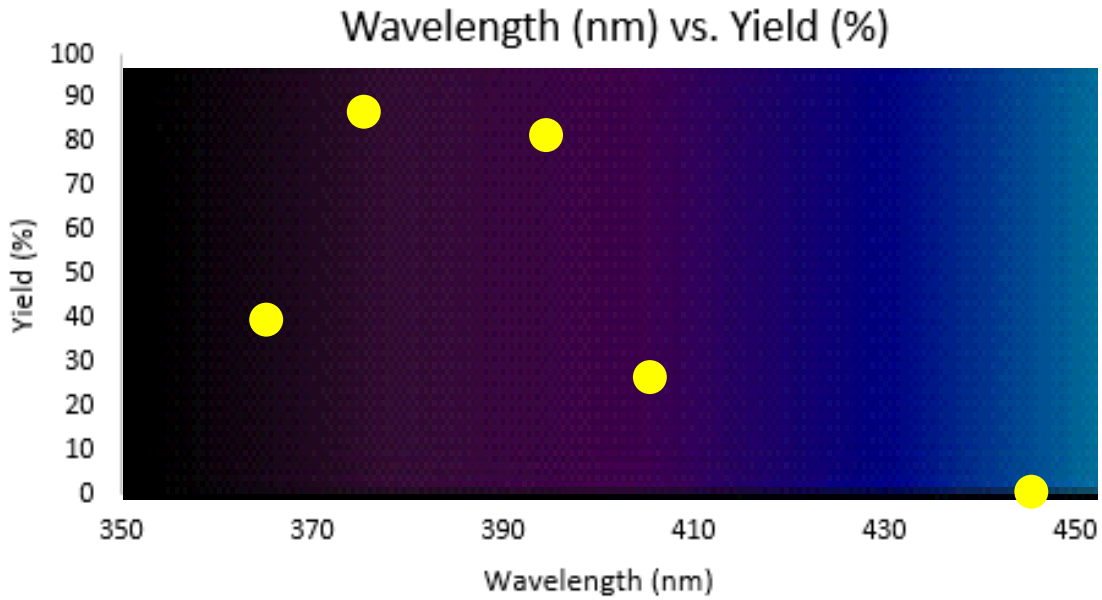


Figure S1. Yield of model reaction vs. wavelength of light employed.

IV. Additional substrates

Compounds were synthesized according to General Procedure A using the substrates indicated.

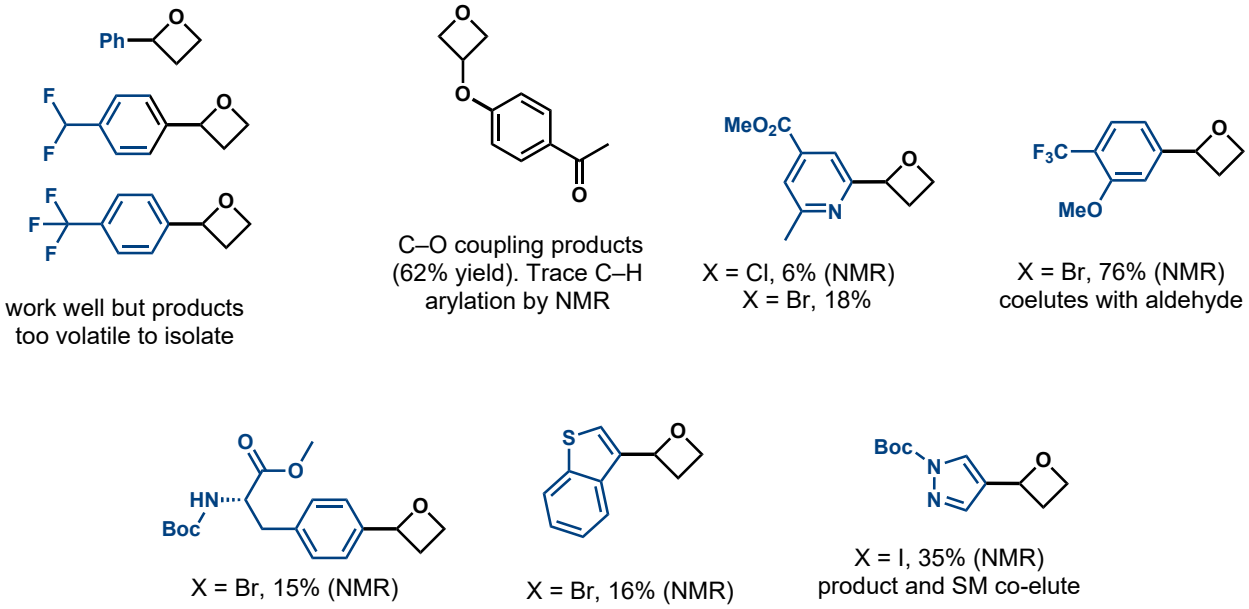


Figure S2. Additional substrates that were not isolated in pure form.

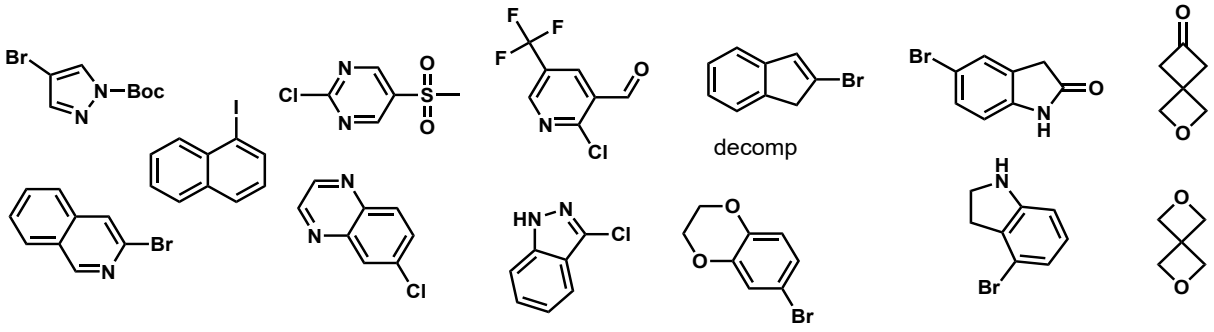


Figure S3. Substrates that did not couple under the standard reaction conditions.

V. Reaction Kinetics

Parallel experiments were set up according to General Procedure A and quenched at the indicated time points. The reactions were analyzed by UPLC and calibrated with authentic samples of product and starting material. Reactions were conducted in duplicate.

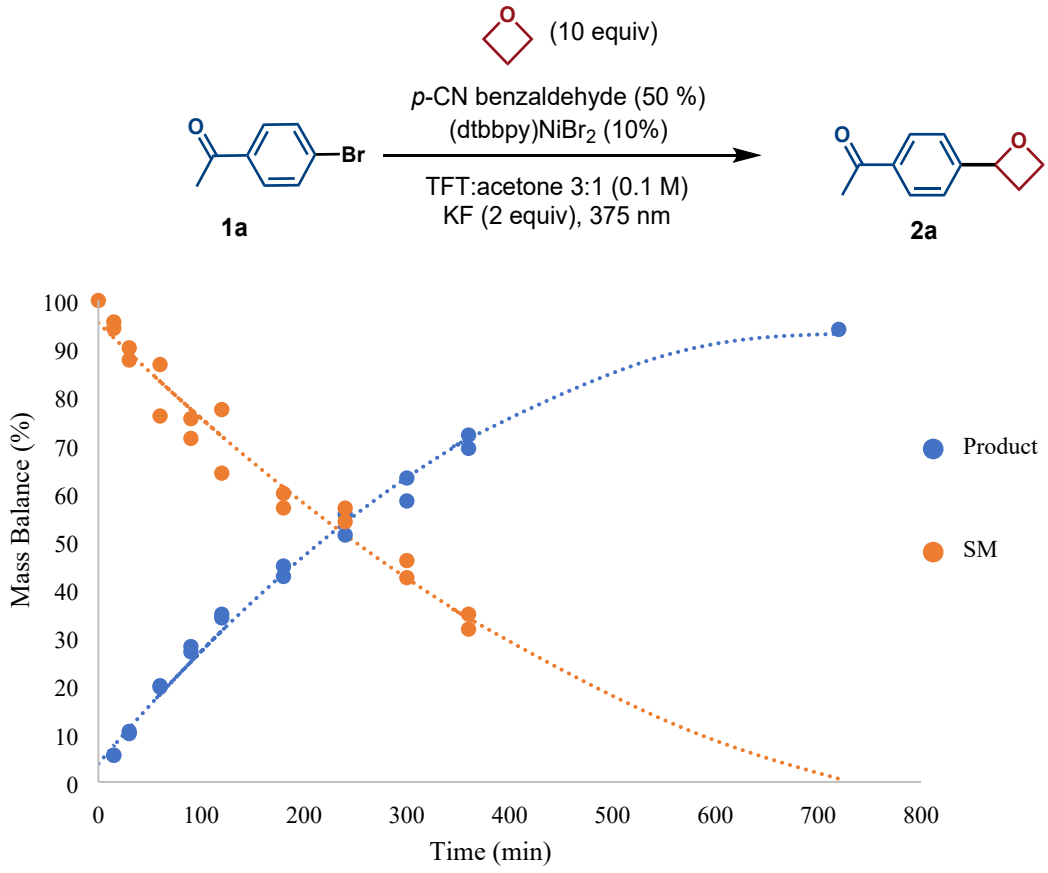
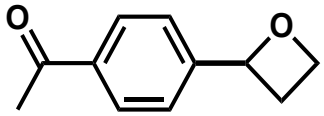


Figure S4. Reaction kinetics measured by UPLC analysis in duplicate.

VI. Compound Characterization

1-(4-(oxetan-2-yl)phenyl)ethan-1-one (2a)

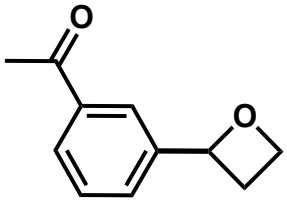


2a

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.3 mmol). A colorless oil (46 mg, 88%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 5.87 (t, *J* = 7.6 Hz, 1H), 4.86 (td, *J* = 8.0, 5.9 Hz, 1H), 4.69 (dt, *J* = 9.2, 5.9 Hz, 1H), 3.09 (dtd, *J* = 11.0, 8.1, 5.8 Hz, 1H), 2.70 – 2.55 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 148.9, 136.5, 128.7, 125.1, 82.2, 68.5, 30.6, 26.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{11}H_{13}O_2^+$ 177.0916; found 177.0920.

1-(3-(oxetan-2-yl)phenyl)ethan-1-one (2b)

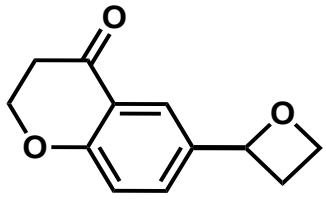


2b

Synthesized following General Procedure B starting with 3-bromoacetophenone (0.2 mmol). A colorless oil (23 mg, 65%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (t, *J* = 1.9 Hz, 1H), 7.90 (dt, *J* = 7.8, 1.5 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 5.87 (t, *J* = 7.5 Hz, 1H), 4.87 (td, *J* = 8.1, 5.9 Hz, 1H), 4.69 (dt, *J* = 9.2, 5.8 Hz, 1H), 3.08 (dtd, *J* = 11.0, 8.0, 5.7 Hz, 1H), 2.73 – 2.59 (m, 1H), 2.63 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 144.3, 137.4, 129.9, 128.9, 127.8, 124.9, 82.3, 68.4, 30.7, 26.8. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{11}H_{13}O_2^+$ 177.0916; found 177.0918.

6-(oxetan-2-yl)chroman-4-one (2c)

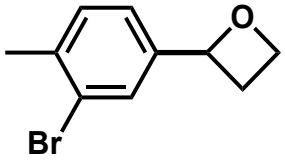


2c

Synthesized following General Procedure B starting with 6-bromochroman-4-one (0.2 mmol). A colorless oil (16 mg, 38%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 2.3 Hz, 1H), 7.66 (dd, *J* = 8.6, 2.3 Hz, 1H), 7.04 (d, *J* = 8.6 Hz, 1H), 5.79 (t, *J* = 7.5 Hz, 1H), 4.84 (td, *J* = 8.1, 5.9 Hz, 1H), 4.67 (dt, *J* = 9.2, 5.7 Hz, 1H), 4.57 (dd, *J* = 6.9, 6.0 Hz, 2H), 3.02 (dtd, *J* = 11.1, 8.0, 5.6 Hz, 1H), 2.85 (dd, *J* = 7.0, 5.9 Hz, 2H), 2.68 (dddd, *J* = 11.1, 9.3, 7.9, 7.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.7, 161.6, 136.7, 133.5, 124.2, 121.0, 118.3, 82.2, 68.1, 67.1, 37.8, 30.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{12}H_{13}O_3^+$ 205.0865; found 205.0868.

2-(3-bromo-4-methylphenyl)oxetane (2e)

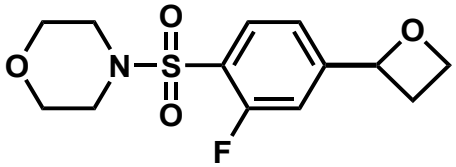


2e

Synthesized following General Procedure B starting with 2-bromo-4-iodo-1-methylbenzene (0.2 mmol). A colorless oil (18 mg, 39%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.25 - 7.22 (m, 2H), 5.74 (t, *J* = 7.5 Hz, 1H), 4.81 (td, *J* = 8.0, 5.9 Hz, 1H), 4.65 (dt, *J* = 9.2, 5.8 Hz, 1H), 3.01 (dtd, *J* = 11.0, 8.0, 5.7 Hz, 1H), 2.62 (dddd, *J* = 11.1, 9.2, 7.8, 7.1 Hz, 1H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.1, 137.3, 130.9, 129.2, 125.1, 124.1, 82.00, 68.3, 30.8, 22.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_{12}BrO^+$ 227.0072; found 227.0065.

4-((2-fluoro-4-(oxetan-2-yl)phenyl)sulfonyl)morpholine (2f)

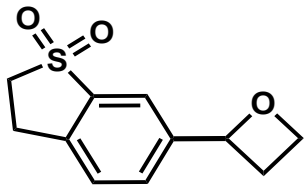


2f

Synthesized following General Procedure B starting with 4-((4-bromo-2-fluorophenyl)sulfonyl)morpholine (0.3 mmol). A colorless oil (48 mg, 53%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 8.1, 6.9 Hz, 1H), 7.34 (dd, *J* = 10.8, 1.6 Hz, 1H), 7.30 – 7.27 (m, 1H), 5.84 (t, *J* = 7.6 Hz, 1H), 4.87 (td, *J* = 7.9, 6.0 Hz, 1H), 4.68 (dt, *J* = 9.2, 5.9 Hz, 1H), 3.79 – 3.72 (m, 4H), 3.23 – 3.06 (m, 5H), 2.60 (ddt, *J* = 11.2, 9.2, 7.3 Hz, 1H).^{19}F NMR (377 MHz, $CDCl_3$) δ -106.58.^{13}C NMR (101 MHz, $CDCl_3$) δ 159.4 (d, *J* = 257.6 Hz), 152.5 (d, *J* = 7.4 Hz), 131.7, 123.4 (d, *J* = 15.1 Hz), 120.6 (d, *J* = 3.4 Hz), 113.8 (d, *J* = 23.1 Hz), 81.1 (d, *J* = 1.5 Hz), 68.6, 66.4, 45.8 (d, *J* = 2.0 Hz), 30.4. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{13}H_{17}FNO_4S^+$ 302.0862; found 302.0861.

6-(oxetan-2-yl)-2,3-dihydrobenzo[b]thiophene 1,1-dioxide (2g)

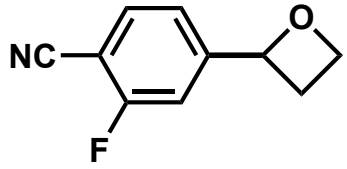


2g

Synthesized following General Procedure B starting with 6-bromo-2,3-dihydrobenzo[b]thiophene 1,1-dioxide (0.2 mmol). A colorless oil (18 mg, 41%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 1.6, 0.7 Hz, 1H), 7.68 - 7.61 (m, 1H), 7.40 (dt, J = 8.0, 0.9 Hz, 1H), 5.86 (t, J = 7.5 Hz, 1H), 4.85 (td, J = 8.0, 6.0 Hz, 1H), 4.67 (dt, J = 9.2, 5.9 Hz, 1H), 3.52 (ddd, J = 7.3, 6.3, 0.8 Hz, 2H), 3.39 (t, J = 6.9 Hz, 2H), 3.09 (dtd, J = 11.1, 8.1, 5.8 Hz, 1H), 2.60 (dddd, J = 11.2, 9.2, 7.8, 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.4, 139.2, 136.5, 130.4, 127.4, 118.1, 81.8, 68.4, 50.9, 30.7, 25.2. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{11}H_{13}O_3S^+$ 225.0585; found 275.0587.

2-fluoro-4-(oxetan-2-yl)benzonitrile (2h)

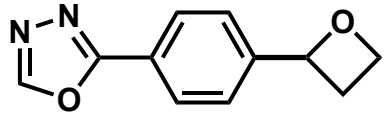


2h

Synthesized following General Procedure B starting with 4-bromo-2-fluorobenzonitrile (0.2 mmol). A colorless oil (24 mg, 68%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.0, 6.4 Hz, 1H), 7.36 – 7.28 (m, 1H), 7.28 – 7.22 (m, 1H), 5.82 (t, *J* = 8.1 Hz, 1H), 4.86 (ddd, *J* = 8.3, 7.6, 6.0 Hz, 1H), 4.67 (dt, *J* = 9.2, 5.9 Hz, 1H), 3.13 (dtd, *J* = 11.1, 8.2, 5.9 Hz, 1H), 2.57 (dddd, *J* = 11.1, 9.2, 7.6, 7.0 Hz, 1H). ^{19}F NMR (377 MHz, $CDCl_3$) δ -105.85. ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5 (d, *J* = 260.6 Hz), 152.5 (d, *J* = 7.2 Hz), 133.8, 121.0 (d, *J* = 3.4 Hz), 114.0, 112.8 (d, *J* = 20.5 Hz), 100.3 (d, *J* = 15.8 Hz), 81.1 (d, *J* = 1.7 Hz), 68.6, 30.4. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_9FNO^+$ 178.0668; found 178.0669.

2-(4-(oxetan-2-yl)phenyl)-1,3,4-oxadiazole (2i)

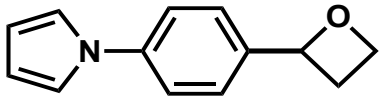


2i

Synthesized following General Procedure B starting with 2-(4-bromophenyl)-1,3,4-oxadiazole (0.2 mmol). A colorless oil (26 mg, 64%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.09 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 5.86 (t, *J* = 7.6 Hz, 1H), 4.86 (td, *J* = 8.0, 5.9 Hz, 1H), 4.68 (dt, *J* = 9.2, 5.8 Hz, 1H), 3.09 (dtd, *J* = 11.1, 8.1, 5.8 Hz, 1H), 2.70 – 2.57 (m, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 152.6, 147.9, 127.4, 125.7, 122.8, 82.1, 68.5, 30.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{11}H_{11}N_2O_2^+$ 203.0821; found 203.0820.

1-(4-(oxetan-2-yl)phenyl)-1H-pyrrole (2j)

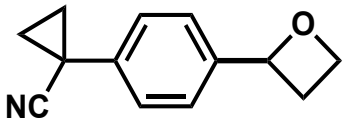


2j

Synthesized following General Procedure B starting with 1-(4-bromophenyl)-1H-pyrrole (0.2 mmol). A colorless oil (26 mg, 65%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.46 (m, 2H), 7.45 – 7.37 (m, 2H), 7.10 (t, *J* = 2.2 Hz, 2H), 6.35 (t, *J* = 2.2 Hz, 2H), 5.83 (t, *J* = 7.5 Hz, 1H), 4.85 (td, *J* = 8.0, 5.9 Hz, 1H), 4.67 (dt, *J* = 9.2, 5.8 Hz, 1H), 3.05 (dtd, *J* = 11.0, 8.0, 5.6 Hz, 1H), 2.69 (dddd, *J* = 11.0, 9.2, 7.9, 7.2 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 140.9, 140.3, 126.6, 120.5, 119.3, 110.5, 82.4, 68.2, 30.8. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{13}H_{14}NO^+$ 200.1075; found 200.1077.

1-(4-(oxetan-2-yl)phenyl)cyclopropane-1-carbonitrile (2k)

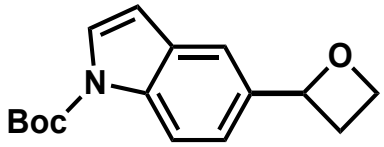


2k

Synthesized following General Procedure B starting with 1-(4-bromophenyl)cyclopropane-1-carbonitrile (0.2 mmol). A colorless oil (27 mg, 68%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 5.79 (t, *J* = 7.5 Hz, 1H), 4.83 (td, *J* = 8.0, 5.8 Hz, 1H), 4.65 (dt, *J* = 9.3, 5.8 Hz, 1H), 3.03 (dtd, *J* = 11.1, 8.0, 5.6 Hz, 1H), 2.69 – 2.55 (m, 1H), 1.73 (q, *J* = 5.0 Hz, 2H), 1.41 (q, *J* = 5.0 Hz, 2H).^{13}C NMR (101 MHz, $CDCl_3$) δ 143.2, 135.6, 126.0, 125.8, 122.6, 82.3, 68.3, 30.7, 18.2, 13.7. HRMS m/z: $[M+Na]^+$ calcd. for $C_{13}H_{13}NONa$, 222.0895; found 222.0903.

***tert*-butyl-5-(oxetan-2-yl)-1H-indole-1-carboxylate (2l)**

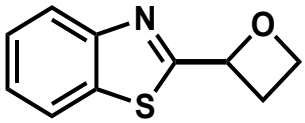


2l

Synthesized following General Procedure B starting with *tert*-butyl 5-bromo-1*H*-indole-1-carboxylate (0.2 mmol). A colorless oil (28 mg, 51%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.6 Hz, 1H), 7.66 (dd, *J* = 1.7, 0.8 Hz, 1H), 7.60 (d, *J* = 3.7 Hz, 1H), 7.39 (dd, *J* = 8.6, 1.7 Hz, 1H), 6.57 (dd, *J* = 3.7, 0.8 Hz, 1H), 5.91 (t, *J* = 7.5 Hz, 1H), 4.85 (td, *J* = 8.0, 5.8 Hz, 1H), 4.69 (dt, *J* = 9.2, 5.7 Hz, 1H), 3.05 (dtd, *J* = 11.0, 8.0, 5.6 Hz, 1H), 2.71 (dddd, *J* = 11.0, 9.2, 7.9, 7.2 Hz, 1H), 1.67 (s, 9H).^{13}C NMR (101 MHz, $CDCl_3$) δ 149.7, 138.1, 134.9, 130.7, 126.4, 121.8, 117.8, 115.2, 107.4, 83.7, 83.3, 68.1, 31.2, 28.2. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{16}H_{20}NO_3^+$ 274.1443; found 274.1442.

2-(oxetan-2-yl)benzo[*d*]thiazole (2m)

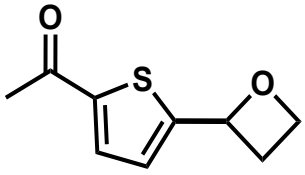


2m

Synthesized following General Procedure B starting with 2-bromobenzo[*d*]thiazole (0.2 mmol). A colorless oil (23 mg, 59%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (ddd, *J* = 8.2, 1.3, 0.7 Hz, 1H), 7.88 (ddd, *J* = 7.9, 1.3, 0.6 Hz, 1H), 7.43 (ddd, *J* = 8.3, 7.2, 1.3 Hz, 1H), 7.34 (ddd, *J* = 8.3, 7.2, 1.2 Hz, 1H), 6.04 (dd, *J* = 8.3, 6.5 Hz, 1H), 4.89 – 4.74 (m, 2H), 3.19 (dtd, *J* = 11.5, 8.4, 6.4 Hz, 1H), 2.82 (dddd, *J* = 11.4, 9.0, 7.3, 6.5 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 175.2, 153.5, 134.9, 126.2, 125.1, 123.1, 122.0, 80.4, 69.9, 29.4. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_{10}NOS^+$ 192.0483; found 192.0488.

1-(5-(oxetan-2-yl)thiophen-2-yl)ethan-1-one (2n)

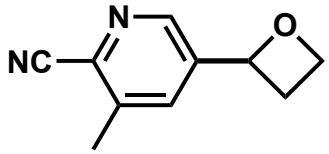


2n

Synthesized following General Procedure B starting with 1-(5-bromothiophen-2-yl)ethan-1-one (0.2 mmol). A colorless oil (16 mg, 44%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 3.8 Hz, 1H), 7.04 (dd, *J* = 3.8, 0.8 Hz, 1H), 5.95 (t, *J* = 7.3 Hz, 1H), 4.79 (td, *J* = 8.0, 6.0 Hz, 1H), 4.70 (dt, *J* = 9.1, 5.9 Hz, 1H), 3.12 (dtd, *J* = 11.3, 8.1, 6.0 Hz, 1H), 2.76 (dddd, *J* = 11.3, 9.1, 7.7, 6.9 Hz, 1H), 2.55 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 190.7, 156.4, 143.9, 132.4, 124.65, 78.5, 68.3, 31.5, 26.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_9H_{10}O_2S^+$ 183.0480; found 183.0475.

3-methyl-5-(oxetan-2-yl)picolinonitrile (2o)

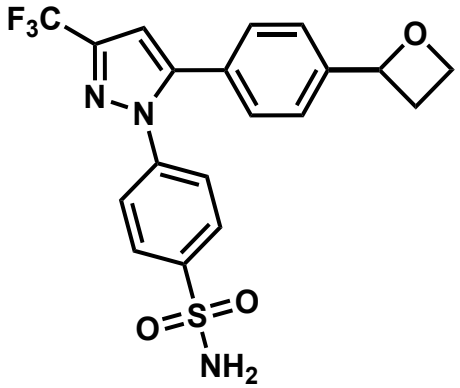


2o

Synthesized following General Procedure B starting with 5-bromo-3-methylpicolinonitrile (0.2 mmol). A colorless oil (25 mg, 72%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 2.0 Hz, 1H), 7.78 (d, *J* = 2.0 Hz, 1H), 5.87 (t, *J* = 7.6 Hz, 1H), 4.89 (td, *J* = 8.0, 6.0 Hz, 1H), 4.69 (dt, *J* = 9.2, 5.9 Hz, 1H), 3.14 (dtd, *J* = 11.2, 8.2, 5.9 Hz, 1H), 2.69 – 2.56 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.6, 142.5, 138.5, 134.6, 133.1, 116.4, 79.7, 68.8, 30.4, 18.7. HRMS m/z: $[M+H]^+$ calcd. for $C_{10}H_{10}N_2O$, 175.0871; found 175.0869.

4-(5-(4-(oxetan-2-yl)phenyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (2p)



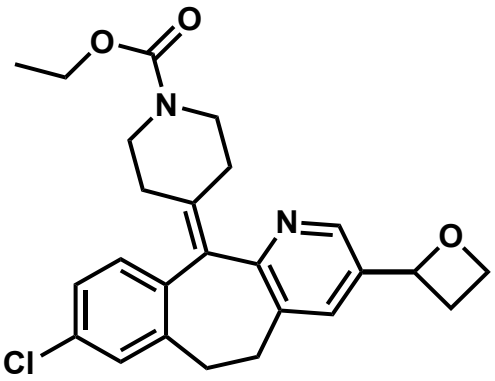
2p

Synthesized following General Procedure B starting with 4-(5-(4-bromophenyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)benzenesulfonamide (0.1 mmol). A colorless oil (27 mg, 63%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.81 (m, 2H), 7.55 – 7.40 (m, 4H), 7.25 (d, *J* = 6.4 Hz, 1H), 6.78 (s, 1H), 5.82 (t, *J* = 7.5 Hz, 1H), 5.08 (s, 2H), 4.85 (td, *J* = 8.0, 5.9 Hz, 1H), 4.66 (dt, *J* = 9.3, 5.8 Hz, 1H), 3.06 (dtd, *J* = 11.1, 8.1, 5.7 Hz, 1H), 2.73 – 2.58 (m, 1H). ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.44. ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.1, 144.9, 144.2 (q, *J* = 38.6 Hz), 142.4, 141.5, 129.0,

128.0, 127.6, 126.0, 125.6, 121.0 (q, *J* = 268 Hz), 106.7, 82.3, 68.4, 30.6. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{19}H_{17}F_3N_3O_3S^+$ 424.0943; found 424.0940.

Ethyl 4-(8-chloro-3-(oxetan-2-yl)-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate (2q)

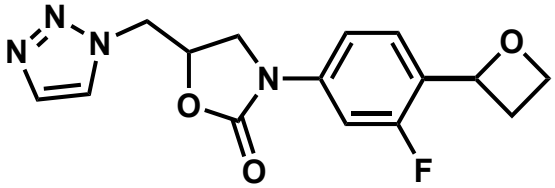


2q

Synthesized following General Procedure B starting with ethyl 4-(3-bromo-8-chloro-5,6-dihydro-11*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridin-11-ylidene)piperidine-1-carboxylate (0.2 mmol). A colorless oil (18 mg, 21%) was obtained after purification by reverse phase preparative HPLC. The product was isolated as the TFA salt.

^{1}H NMR (400 MHz, DMSO) δ 8.46 (dd, *J* = 9.8, 2.0 Hz, 1H), 7.90 (d, *J* = 8.1 Hz, 1H), 7.35 (dd, *J* = 4.0, 2.3 Hz, 1H), 7.25 (dd, *J* = 8.1, 2.3 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 1H), 5.77 (td, *J* = 7.5, 1.8 Hz, 1H), 4.69 (td, *J* = 8.0, 5.7 Hz, 1H), 4.58 (dtd, *J* = 9.2, 5.7, 1.4 Hz, 1H), 4.04 (q, *J* = 7.1 Hz, 2H), 3.70 – 3.65 (m, 5H), 3.43 – 3.29 (m, 2H), 3.20 – 3.10 (m, 2H), 3.05 – 2.80 (m, 2H), 2.70 – 2.55 (m, 1H), 2.38 – 2.13 (m, 2H), 1.17 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 158.9, 158.6, 158.2, 155.0, 142.7, 140.7, 140.7, 137.9, 132.3, 131.1, 129.5, 126.3, 79.8, 79.7, 68.1, 66.1, 61.7, 61.2, 44.7, 44.7, 31.1, 31.0, 30.8, 30.0, 15.1. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{25}H_{28}ClN_2O_3^+$ 439.1788; found 439.1789.

5-((1H-1,2,3-triazol-1-yl)methyl)-3-(3-fluoro-4-((S)-oxetan-2-yl)phenyl)oxazolidin-2-one (2r)

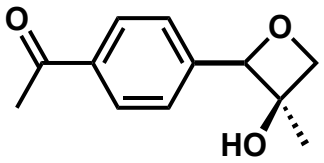


2r

Synthesized following General Procedure B starting with 5-((1*H*-1,2,3-triazol-1-yl)methyl)-3-(3-fluoro-4-iodophenyl)oxazolidin-2-one (0.1 mmol). A colorless oil (25 mg, 79%) was obtained after purification by silica gel chromatography. The compound is obtained as a 1:1 mixture of diastereomers that was not separable by chromatography. Diastereomers could not be unambiguously assigned by NMR but have been explicitly picked where possible.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.68 (s, 1H), 7.54 (t, *J* = 8.3 Hz, 1H), 7.27 (ddd, *J* = 18.0, 12.2, 2.3 Hz, 1H), 7.03 (ddd, *J* = 19.4, 8.5, 2.2 Hz, 1H), 5.91 (t, *J* = 7.5 Hz, 1H), 5.01 (ddd, *J* = 10.7, 8.9, 4.5 Hz, 1H), 4.80 – 4.66 (m, 3H), 4.59 (dt, *J* = 9.2, 5.9 Hz, 1H), 4.11 (td, *J* = 9.1, 1.6 Hz, 1H), 3.87 (ddd, *J* = 9.5, 6.2, 1.5 Hz, 1H), 3.00 (dtd, *J* = 11.2, 8.1, 5.9 Hz, 1H), 2.55 (ddd, *J* = 17.7, 10.6, 7.8 Hz, 1H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -116.76 and -116.78 (pair of diastereomers). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.25 (d, *J* = 247.5), 153.2, 138.3 – 137.7 (m), 134.6, 127.6 (d, *J* = 2.9 Hz) and 127.5 (d, *J* = 2.9 Hz) (pair of diastereomers), 126.9 (d, *J* = 14.1 Hz), 125.1, 113.50 (d, *J* = 10.4) and 113.47 (d, *J* = 10.4) (pair of diastereomers), 106.0 (d, *J* = 26.8) and 105.9 (d, *J* = 26.8) (pair of diastereomers), 70.5, 68.8, 52.0, 47.3, 47.2, 29.9. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{15}H_{16}FN_4O_3^+$ 319.1206; found 319.1208.

1-(4-(3-hydroxy-3-methyloxetan-2-yl)phenyl)ethan-1-one (3a)

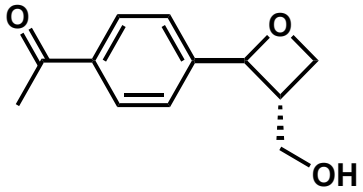


3a

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 3-methyloxetan-3-ol. A colorless oil (16 mg, 39%, 10:1 d.r.) was obtained after purification by silica gel chromatography. The minor diastereomer could not fully be separated by chromatography and small peaks for the minor diastereomer remain in the NMR spectra.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 - 8.00 (m, 2H), 7.51 - 7.45 (m, 2H), 5.60 (s, 1H), 4.69 (d, J = 6.9 Hz, 1H), 4.58 (d, J = 6.9 Hz, 1H), 2.62 (s, 3H), 1.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 142.6, 137.0, 128.7, 126.0, 92.8, 83.7, 74.2, 26.7, 25.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{12}H_{15}O_3^+$ 207.1021; found 207.1017.

1-(4-(3-(hydroxymethyl)oxetan-2-yl)phenyl)ethan-1-one (3b)

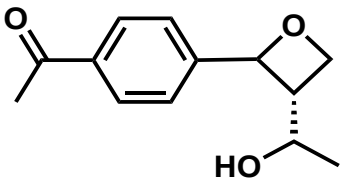


3b

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with oxetan-3-ylmethanol. A colorless oil (20 mg, 48%, 10:1 d.r.) was obtained after purification by silica gel chromatography. The minor diastereomer could not fully be separated by chromatography and small peaks for the minor diastereomer remain in the NMR spectra.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 5.65 (d, *J* = 6.1 Hz, 1H), 4.75 (dd, *J* = 8.4, 6.2 Hz, 1H), 4.60 (t, *J* = 6.5 Hz, 1H), 3.99 (q, *J* = 10.7, 9.6 Hz, 2H), 3.03 (dp, *J* = 8.2, 6.5 Hz, 1H), 2.61 (s, 3H), 1.74 (br s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 148.1, 136.5, 128.7, 125.0, 84.9, 70.4, 63.5, 45.7, 26.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{12}H_{15}O_3^+$ 207.1021; found 207.1017.

1-(4-(3-(1-hydroxyethyl)oxetan-2-yl)phenyl)ethan-1-one (3c)

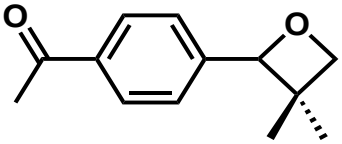


3c

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 1-(oxetan-3-yl)ethan-1-ol. A colorless oil (11 mg, 26%, 15:1 d.r.) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.56 (d, *J* = 8.2 Hz, 2H), 5.75 (d, *J* = 6.1 Hz, 1H), 4.71 (dd, *J* = 8.4, 6.2 Hz, 1H), 4.53 (t, *J* = 6.5 Hz, 1H), 4.27-4.22 (m, 1H), 2.78 (m, 1H), 2.61 (s, 3H), 1.19 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 144.8, 138.8, 128.9, 125.4, 85.2, 70.3, 69.5, 51.1, 26.9, 21.5. HRMS m/z: $[M+H]^+$ calcd. for $C_{13}H_{16}O_3$, 221.1178; found 221.1175.

1-(4-(3-hydroxy-3-methyloxetan-2-yl)phenyl)ethan-1-one (3d)

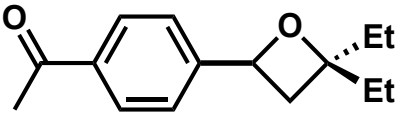


3d

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 3,3-dimethyloxetane. A colorless oil (18 mg, 44%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 - 7.92 (m, 2H), 7.41 - 7.33 (m, 2H), 5.54 (s, 1H), 4.55 (d, *J* = 5.4 Hz, 1H), 4.27 (d, *J* = 5.4 Hz, 1H), 2.61 (s, 3H), 1.43 (s, 3H), 0.78 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 145.9, 136.4, 128.5, 125.3, 91.3, 81.5, 40.9, 27.1, 26.8, 22.6. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{13}H_{17}O_2^+$ 205.1229; found 205.1226.

1-(4-(4,4-diethyloxetan-2-yl)phenyl)ethan-1-one (3e)

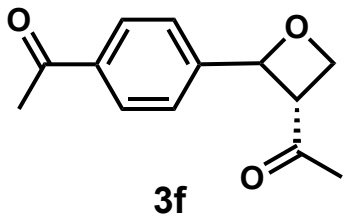


3e

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 2,2-diethyloxetane. A colorless oil (26 mg, 55%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.92 (m, 2H), 7.51 – 7.43 (m, 2H), 5.67 – 5.59 (m, 1H), 2.76 (dd, *J* = 10.9, 8.3 Hz, 1H), 2.60 (s, 3H), 2.34 – 2.20 (m, 1H), 1.98 – 1.59 (m, 4H), 1.03 (t, *J* = 7.5 Hz, 3H), 0.83 (dt, *J* = 10.2, 7.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 149.9, 136.1, 128.6, 124.9, 85.8, 74.6, 38.1, 32.6, 30.7, 26.6, 7.6, 7.3. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{15}H_{21}O_2^+$ 233.1542; found 233.1535.

1-(4-(3-acetyloxetan-2-yl)phenyl)ethan-1-one (3f)

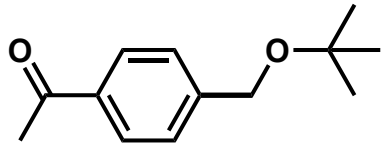


3f

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 1-(oxetan-3-yl)ethan-1-one. A colorless oil (6 mg, 14%, >20:1 d.r.) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.54 (d, *J* = 8.2 Hz, 2H), 5.89 (d, *J* = 6.1 Hz, 1H), 4.88 (t, *J* = 6.5 Hz, 1H), 4.79 (dd, *J* = 8.4, 6.2 Hz, 1H), 3.76 (m, 1H), 2.62 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.4, 197.6, 146.8, 137.1, 128.9, 125.2, 82.9, 68.8, 54.6, 28.4, 26.7. HRMS m/z: $[M+H]^+$ calcd. for $C_{13}H_{14}O_3$, 219.1021; found 219.1019.

1-(4-(*tert*-butoxymethyl)phenyl)ethan-1-one(3i)

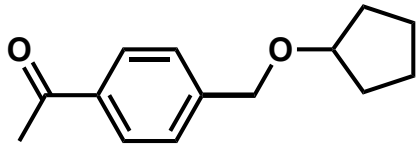


3i

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with methyl tert-butyl ether. A colorless oil (15 mg, 36%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.44 (dp, *J* = 7.6, 0.9 Hz, 2H), 4.51 (s, 2H), 2.59 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 145.8, 136.2, 128.5, 127.2, 73.9, 63.7, 27.8, 26.8. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{13}H_{19}O_2^+$ 207.1385; found 207.1384.

1-(4-((cyclopentyloxy)methyl)phenyl)ethan-1-one (3j)

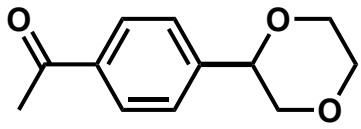


3j

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with cyclopentyl methyl ether. A colorless oil (12 mg, 27%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.43 (dp, *J* = 7.6, 0.9 Hz, 2H), 4.53 (s, 2H), 4.06 – 3.96 (m, 1H), 2.60 (s, 3H), 1.82 – 1.67 (m, 5H), 1.60 – 1.52 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 144.8, 136.2, 128.5, 127.3, 81.4, 70.1, 32.3, 26.7, 23.6. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{14}H_{19}O_2^+$ 219.1385; found 219.1386.

1-(4-(1,4-dioxan-2-yl)phenyl)ethan-1-one (3l)

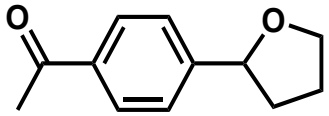


3l

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 1,4-dioxane. (ttbterpy)$NiBr_2$ was used in place of (dtbbpy)$NiBr_2$. One equivalent (100%) *p*-cyanobenzaldehyde was used. K_2HPO_4 was used instead of KF. A colorless oil (25 mg, 61%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.45 (dp, *J* = 7.6, 0.9 Hz, 2H), 4.69 (dd, *J* = 6.9, 1.0 Hz 1H), 3.99 – 3.39 (m, 5H), 3.42 (dd, *J* = 9.2, 7.8 Hz, 1H),.2.60 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 143.5, 136.8, 128.5, 126.3, 77.4, 72.3, 67.0, 66.4, 26.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{12}H_{15}O_3^+$ 207.1021; found 207.1015.

1-(4-(tetrahydrofuran-2-yl)phenyl)ethan-1-one (3m)

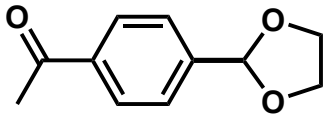


3m

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.3 mmol) and replacing oxetane with tetrahydrofuran. One equivalent (100%) *p*-cyanobenzaldehyde was used. K_2HPO_4 was used instead of KF. A colorless oil (42 mg, 73%) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 2H), 4.95 (t, *J* = 7.2 Hz, 1H), 4.11 (dt, *J* = 8.4, 6.8 Hz, 1H), 3.97 (dt, *J* = 8.3, 6.9 Hz, 1H), 2.60 (s, 3H), 2.37 (dq, *J* = 12.5, 6.7 Hz, 1H), 2.09 – 1.92 (m, 2H), 1.85 – 1.70 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 149.2, 136.1, 128.5, 125.6, 80.1, 77.4, 77.0, 76.7, 68.9, 34.7, 26.6, 26.0. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{12}H_{15}O_2^+$ 191.1072; found 191.1069.

1-(4-(1,3-dioxolan-2-yl)phenyl)ethan-1-one (3n)



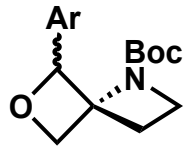
3n

Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with 1,3-dioxolane. A colorless oil (20 mg, 51%, 3:1 r.r.) was obtained after purification by silica gel chromatography.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 - 7.98 (m, 2H), 7.63 - 7.54 (m, 2H), 5.89 (s, 1H), 4.20 - 4.03 (m, 4H), 2.63 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 128.4, 126.7, 103.0, 96.2, 71.8, 65.4, 26.7. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{11}H_{13}O_3^+$ 193.0865; found 193.0863.

***tert*-butyl 5-(4-acetylphenyl)-6-oxa-1-azaspiro[3.3]heptane-1-carboxylate (3o-1)** and **tert-butyl 2-(4-acetylphenyl)-6-oxa-1-azaspiro[3.3]heptane-1-carboxylate (3o-2)**

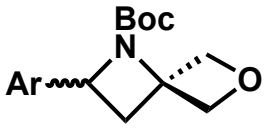
Synthesized following General Procedure B starting with 4-bromoacetophenone (0.2 mmol) and replacing oxetane with *tert*-butyl 6-oxa-1-azaspiro[3.3]heptane-1-carboxylate (5 equiv). Purified by silica gel chromatography.



3o-1, 1.5:1 d.r.

A colorless oil (10mg, 16%, 1.5:1 d.r.) was obtained.

^{1}H NMR – major isomer – (400 MHz, $CDCl_3$, 25 °C) δ 8.04 – 7.98 (m, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 6.22 – 6.17 (m, 1H), 5.35 – 5.19 (m, 1H), 4.63 – 4.53 (m, 1H), 3.78 – 3.64 (m, 1H), 3.61 – 3.51 (m, 1H), 2.63 (s, 3H), 2.00 (ddd, *J* = 12.1, 8.8, 5.6 Hz, 1H), 1.89 – 1.73 (m, 1H), 1.57 (s, 9H). A satisfactory ^{13}C NMR spectrum could not be obtained at 25 °C due to broadening from the presence of rotamers. The ^{13}C NMR spectrum was better resolved at 0 °C, but some broadening as well as additional peaks were still observed. ^{13}C NMR (101 MHz, $CDCl_3$, 0 °C) δ 197.2, 154.3, 144.1, 136.7, 128.9, 125.6, 89.9, 88.7, 80.9, 79.4, 71.3, 67.2, 44.0, 28.7 (br), 26.9. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{18}H_{24}NO_4^+$ 318.1705; found 318.1699.



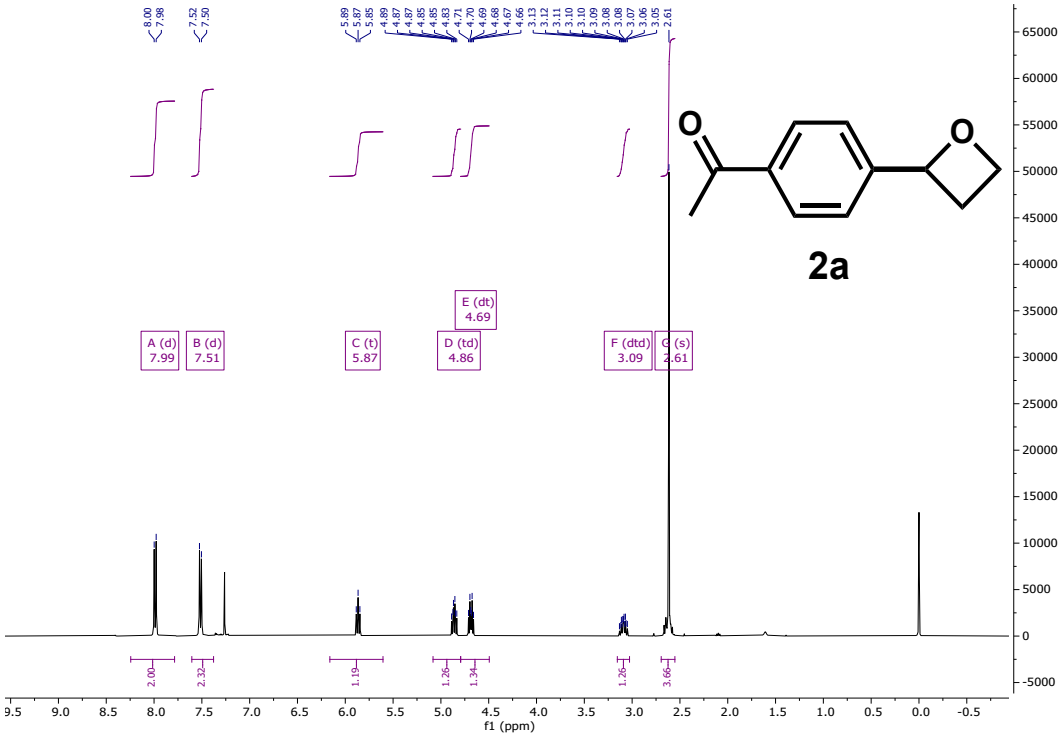
3o-2

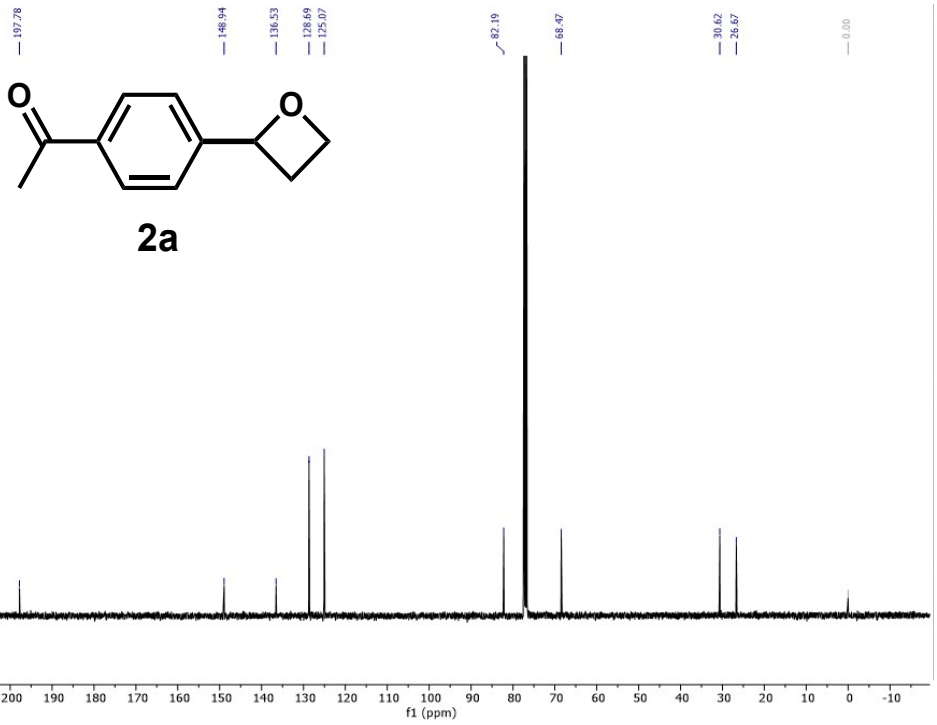
A colorless oil (30mg, 47%) was obtained.

^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.98 – 7.88 (m, 2H), 7.40 – 7.28 (m, 2H), 5.60 – 5.18 (br m, 2H), 5.08 (br t, *J* = 7.1 Hz, 1H), 4.68 (t, *J* = 6.0 Hz, 2H), 2.93 (dd, *J* = 12.0, 8.5 Hz, 1H), 2.59 (s, 3H), 2.32 (dd, *J* = 12.0, 5.8 Hz, 1H), 1.68 – 1.37 (br m, 9H). A satisfactory ^{13}C NMR spectrum could not be obtained at 25 °C due to broadening from the presence of rotamers. The ^{13}C NMR spectrum was better resolved at 0 °C, some broadening as well as additional peaks were still observed. ^{13}C NMR (101 MHz, $CDCl_3$, 0 °C) δ 198.0, 154.5, 146.5, 136.4, 129.0, 125.7,

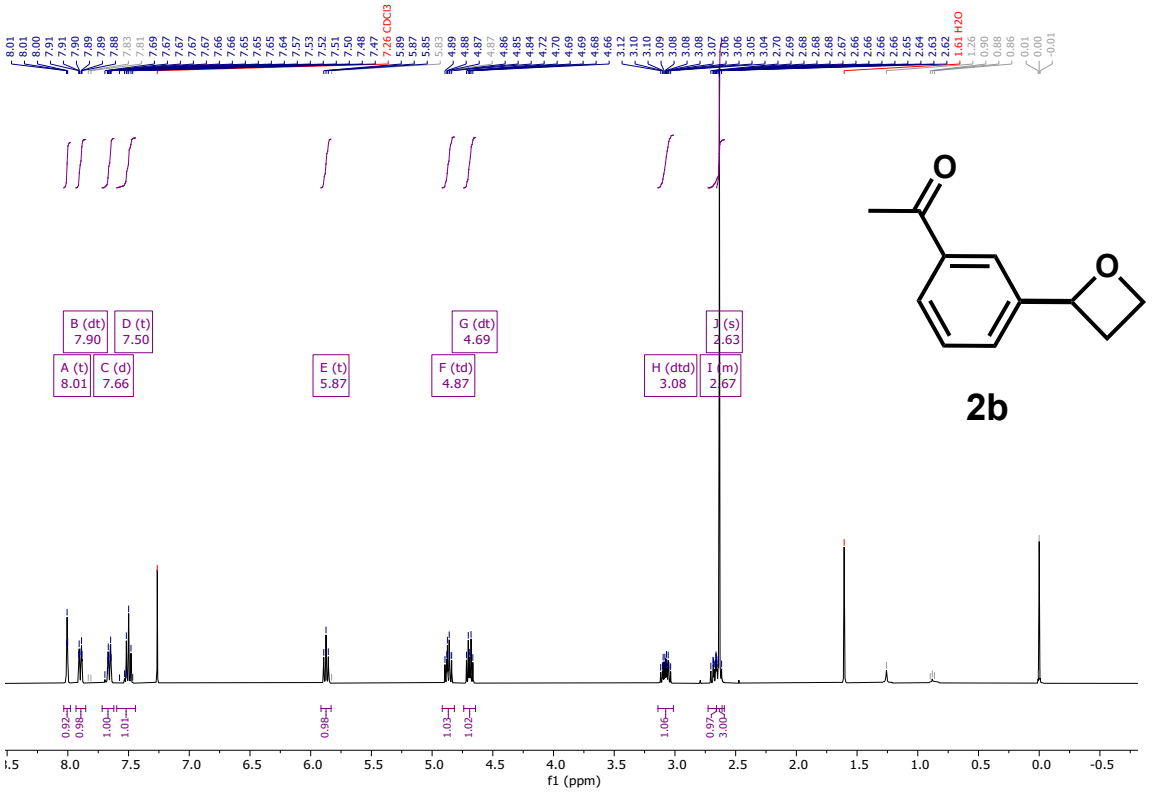
82.6, 81.6, 80.1, 65.3, 58.9, 38.3, 28.6 (br), 26.9. HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{18}H_{24}NO_4^+$ 318.1705; found 318.1701.

VII. Pictures of NMR Spectra

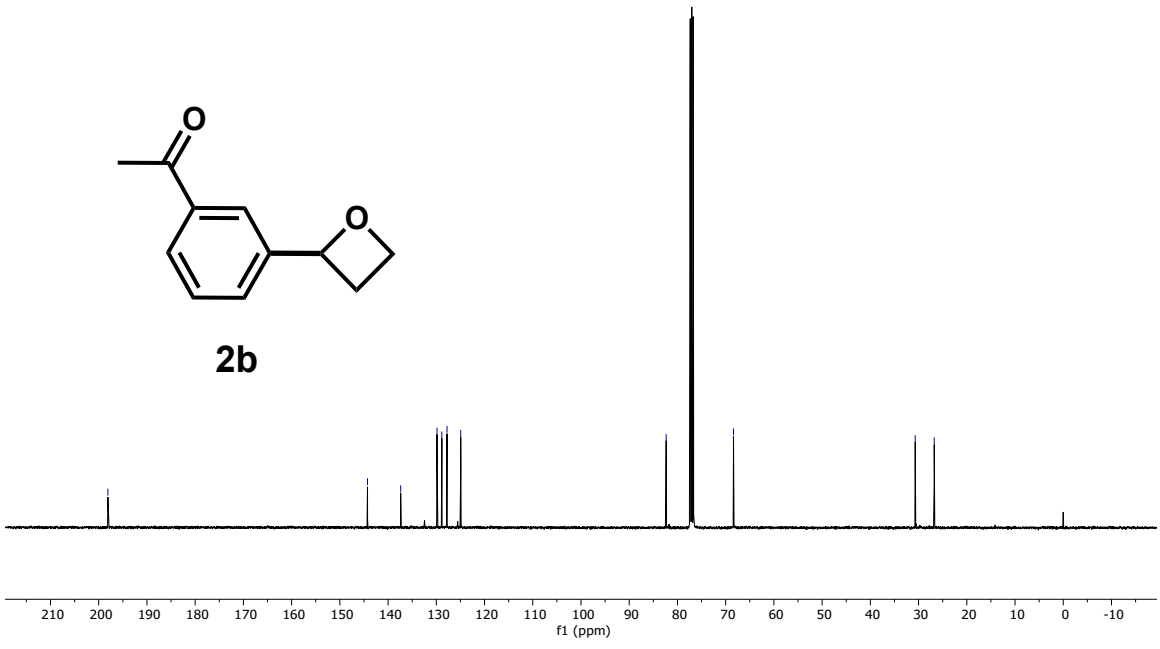




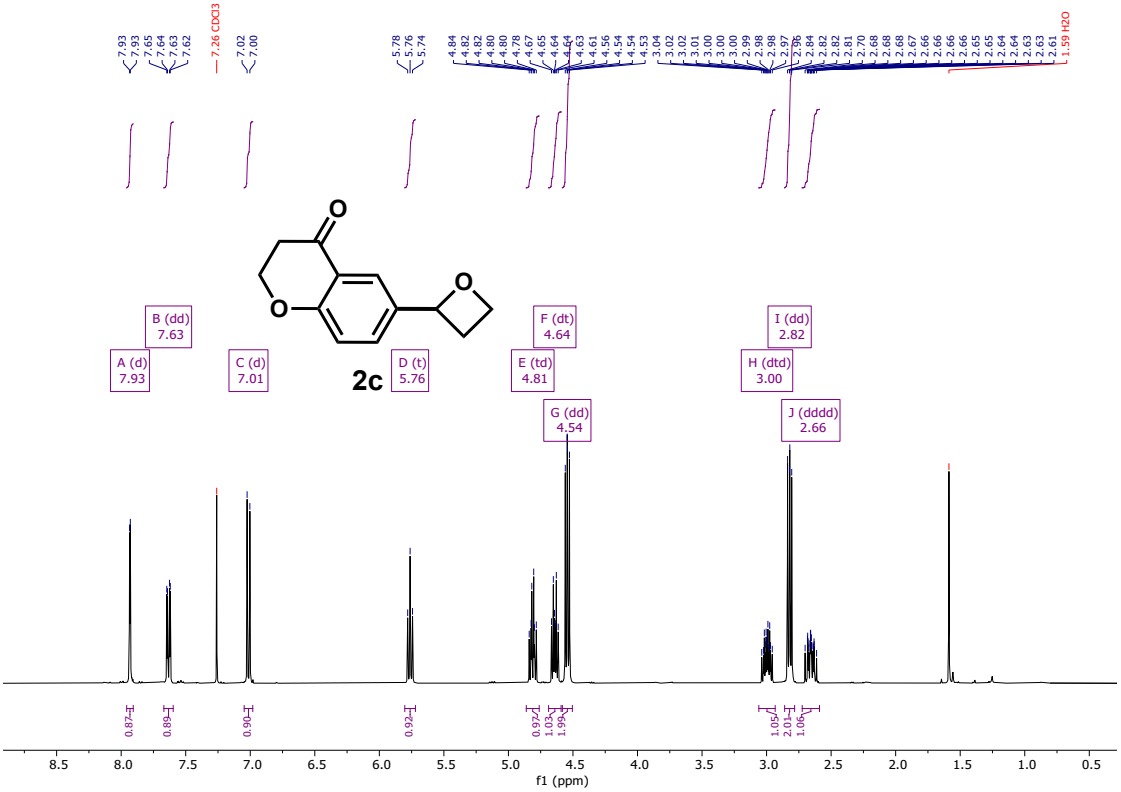
2a

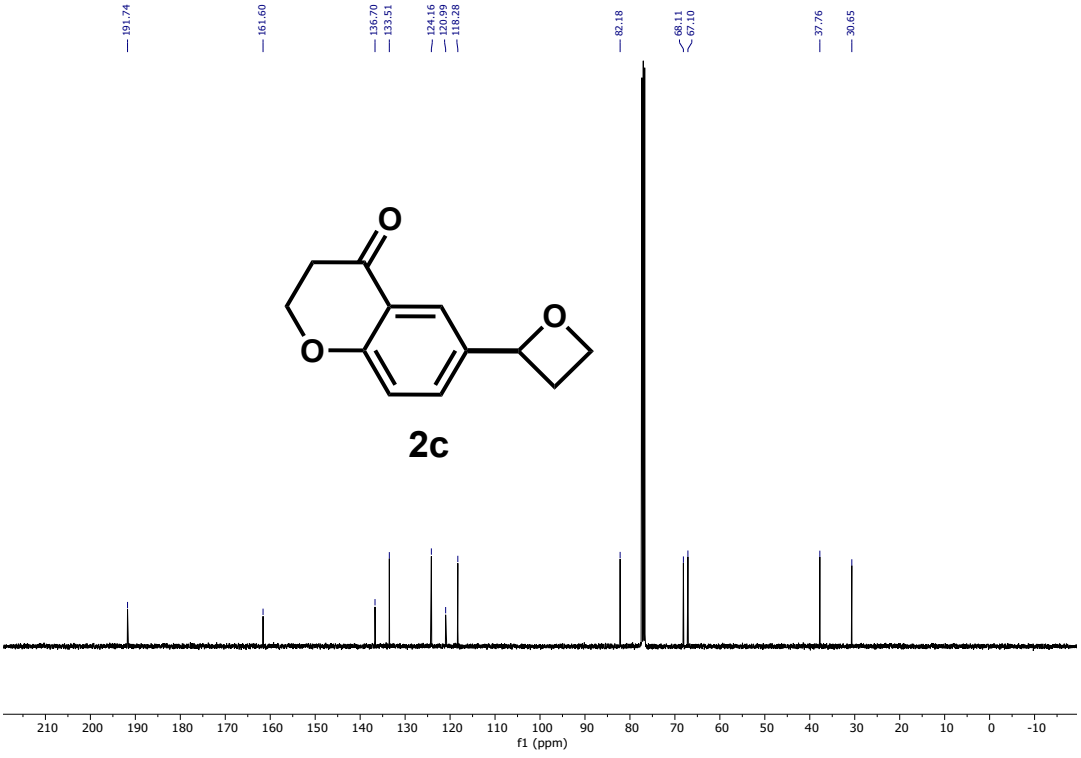

B (dt) 7.90
D (t) 7.50
A (t) 8.01
C (d) 7.66
E (t) 5.87
G (dt) 4.69
F (td) 4.87
H (dtd) 3.08
I (m) 2.67
J (s) 2.63
f1 (ppm)

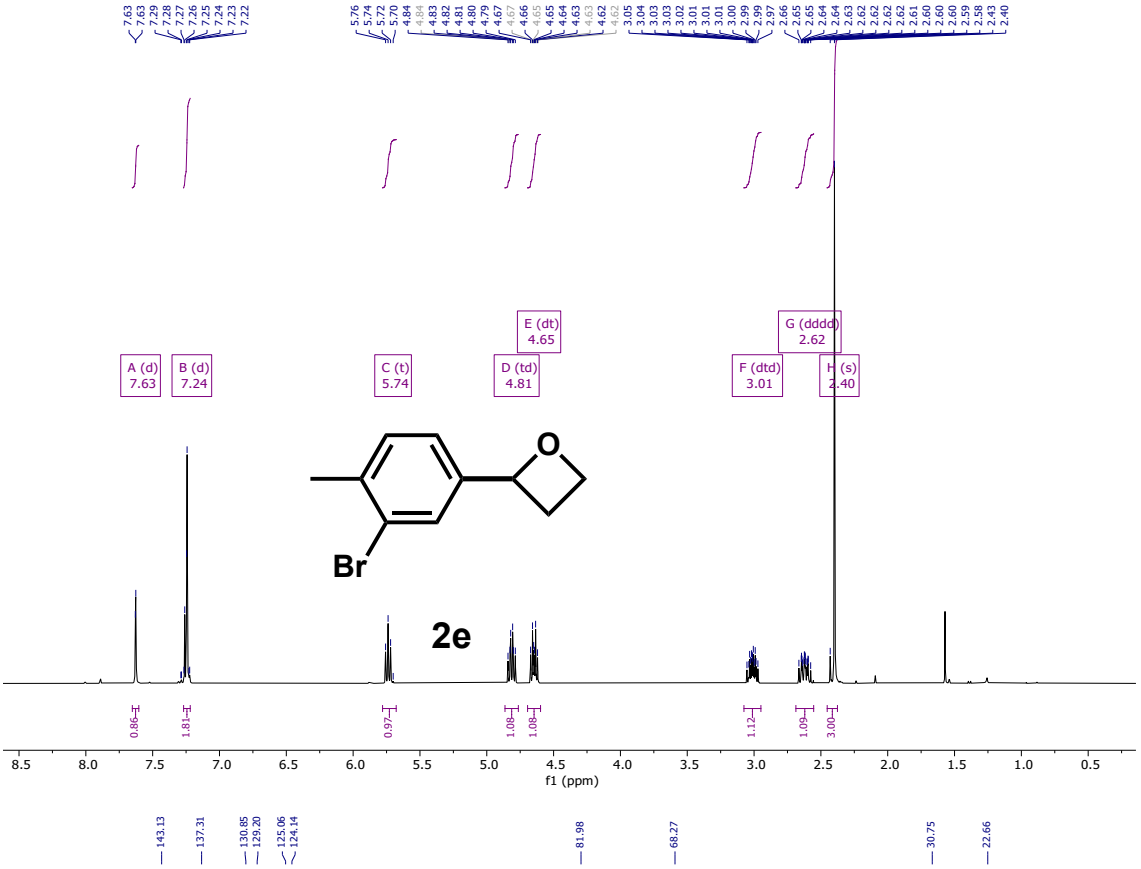
2b

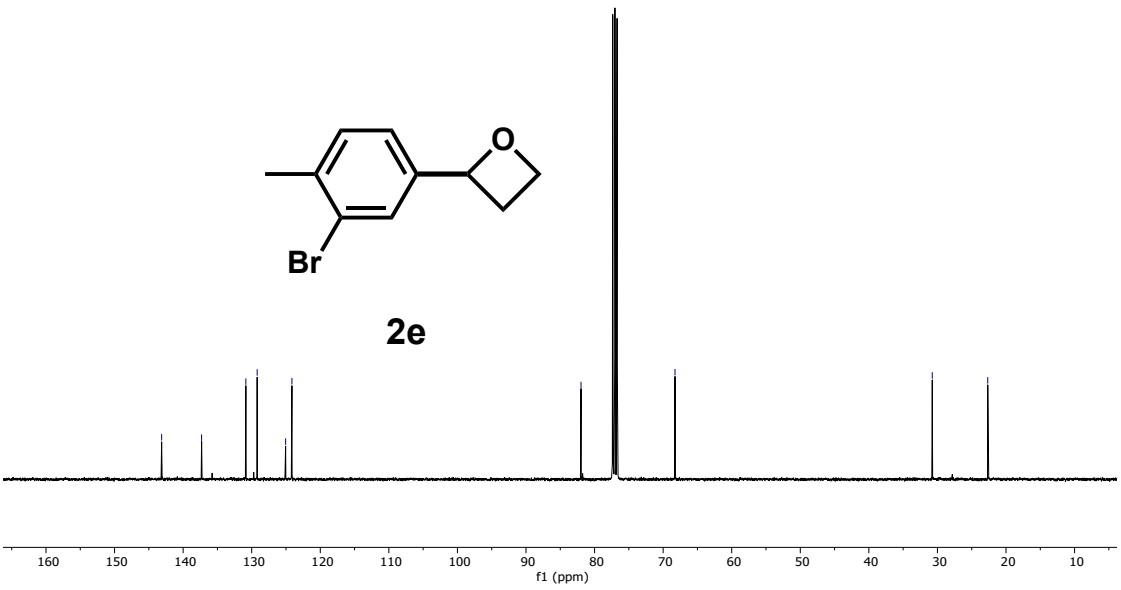

198.10
144.27
137.39
129.86
128.85
127.78
124.94
82.34
68.38
30.69
26.75
f1 (ppm)

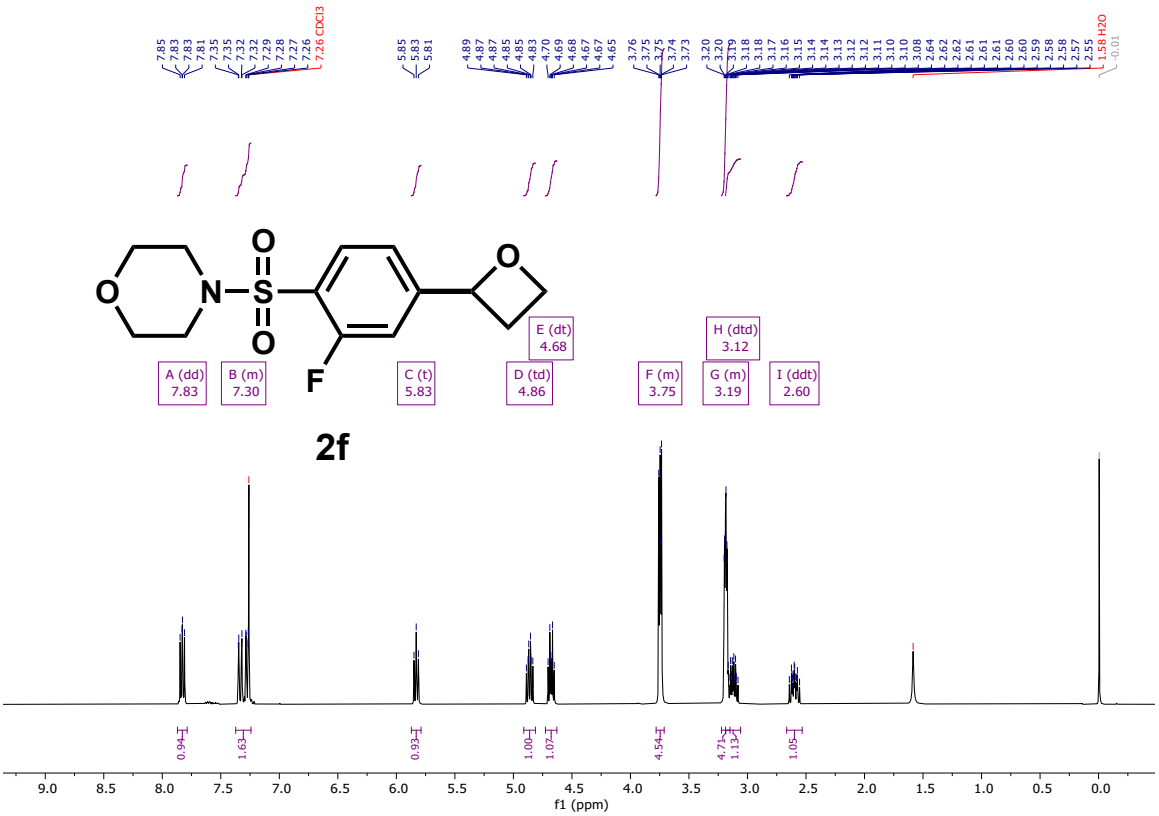
2b


A (d) 7.93
B (dd) 7.63
C (d) 7.01
D (t) 5.76
E (td) 4.81
F (dt) 4.64
G (dd) 4.54
H (dtd) 3.00
I (dd) 2.82
J (dddd) 2.66
2c
f1 (ppm)

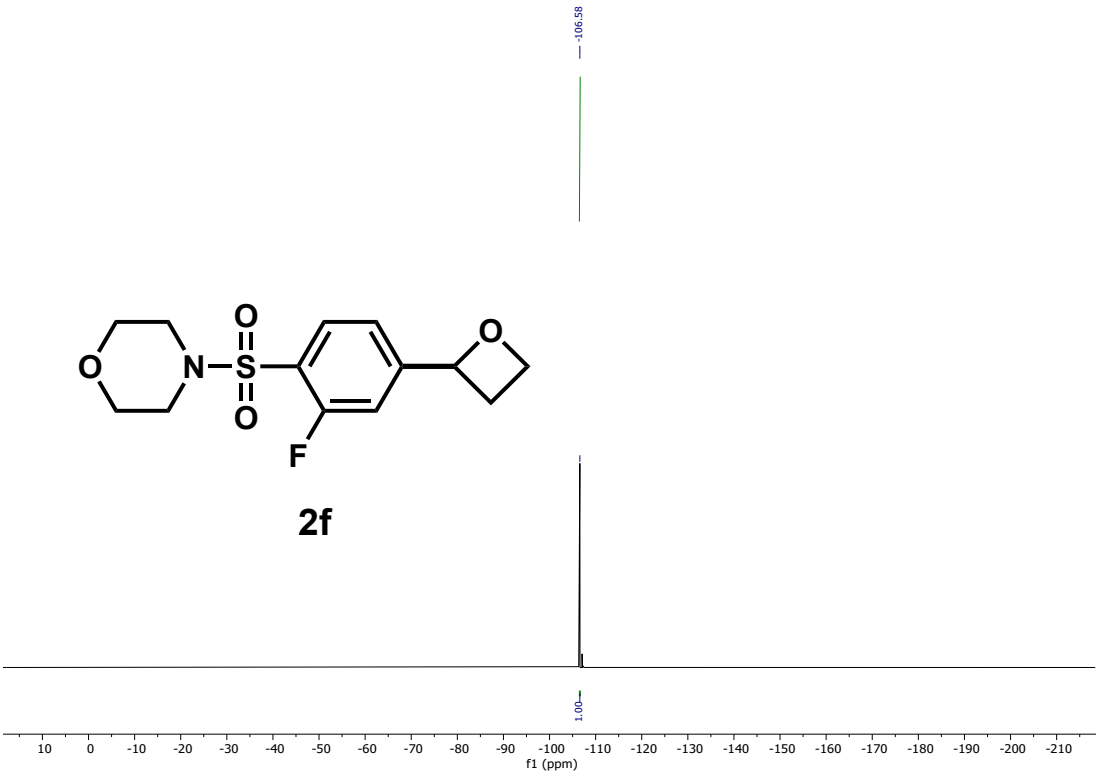

2c
f1 (ppm)


A (d) 7.63
B (d) 7.24
C (t) 5.74
D (td) 4.81
E (dt) 4.65
F (dtd) 3.01
G (dddd) 2.62
H (s) 2.40
Br
2e
f1 (ppm)

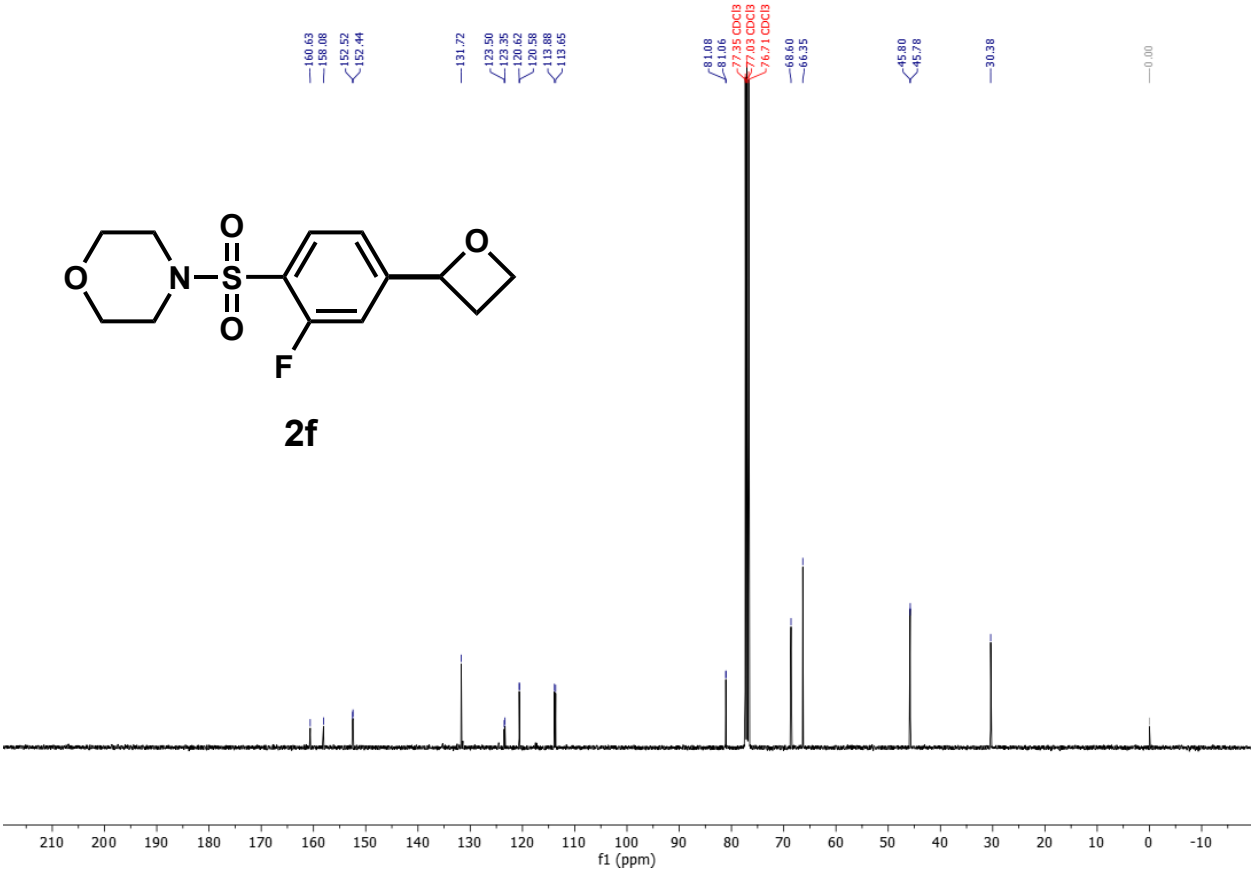

Br
2e
f1 (ppm)


A (dd) 7.83
B (m) 7.30
C (t) 5.83
D (td) 4.86
E (dt) 4.68
F (m) 3.75
G (m) 3.19
H (dtd) 3.12
I (ddt) 2.60
f1 (ppm)

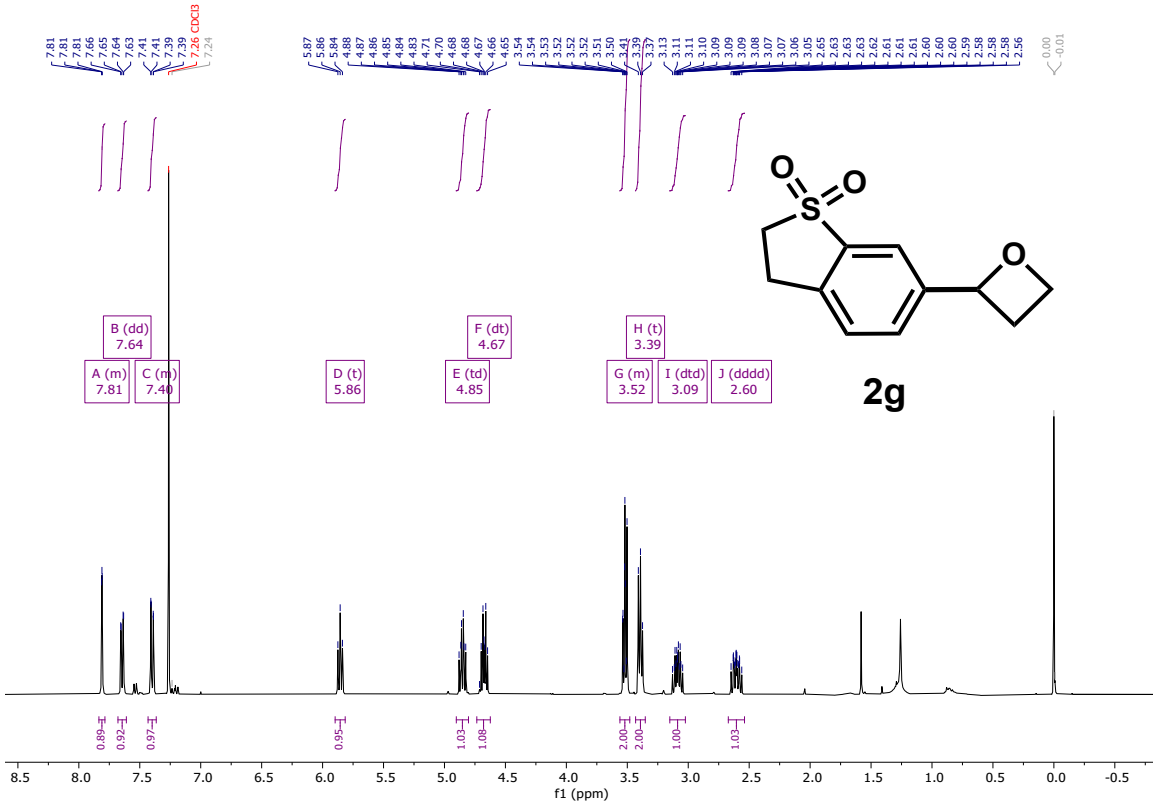
2f

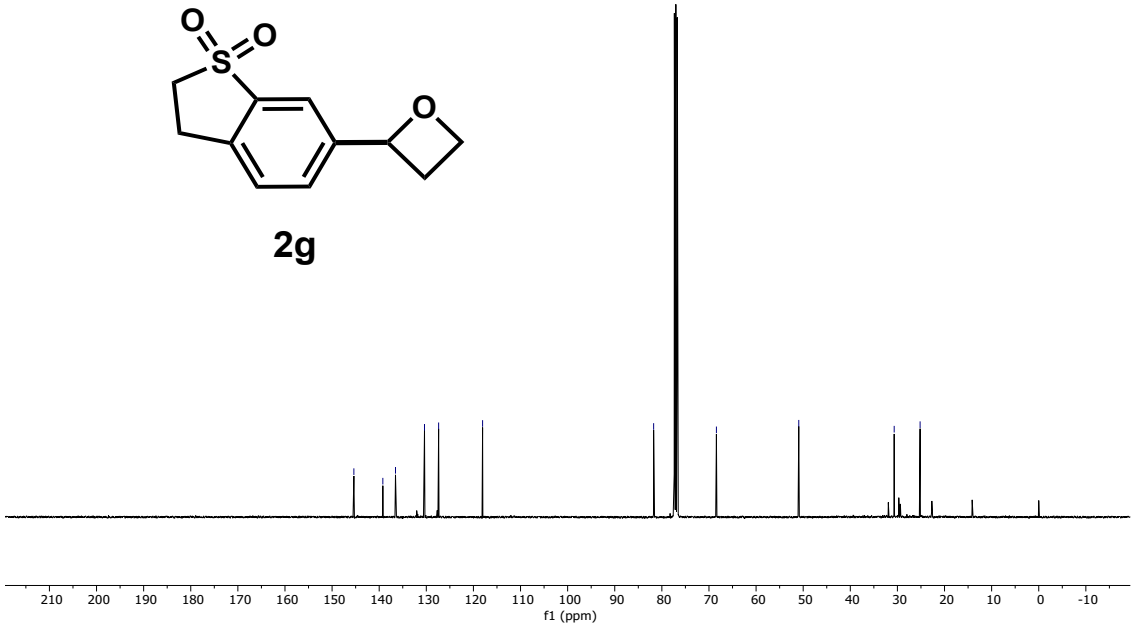

-106.58
f1 (ppm)

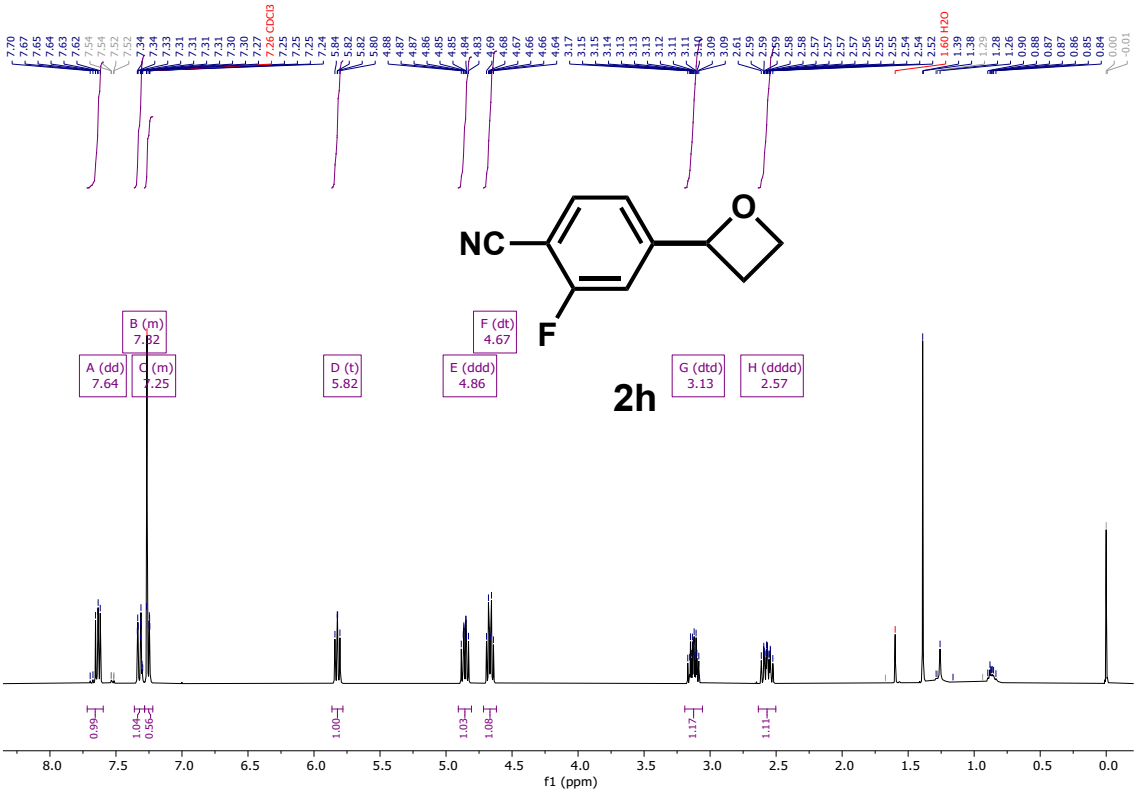
2f

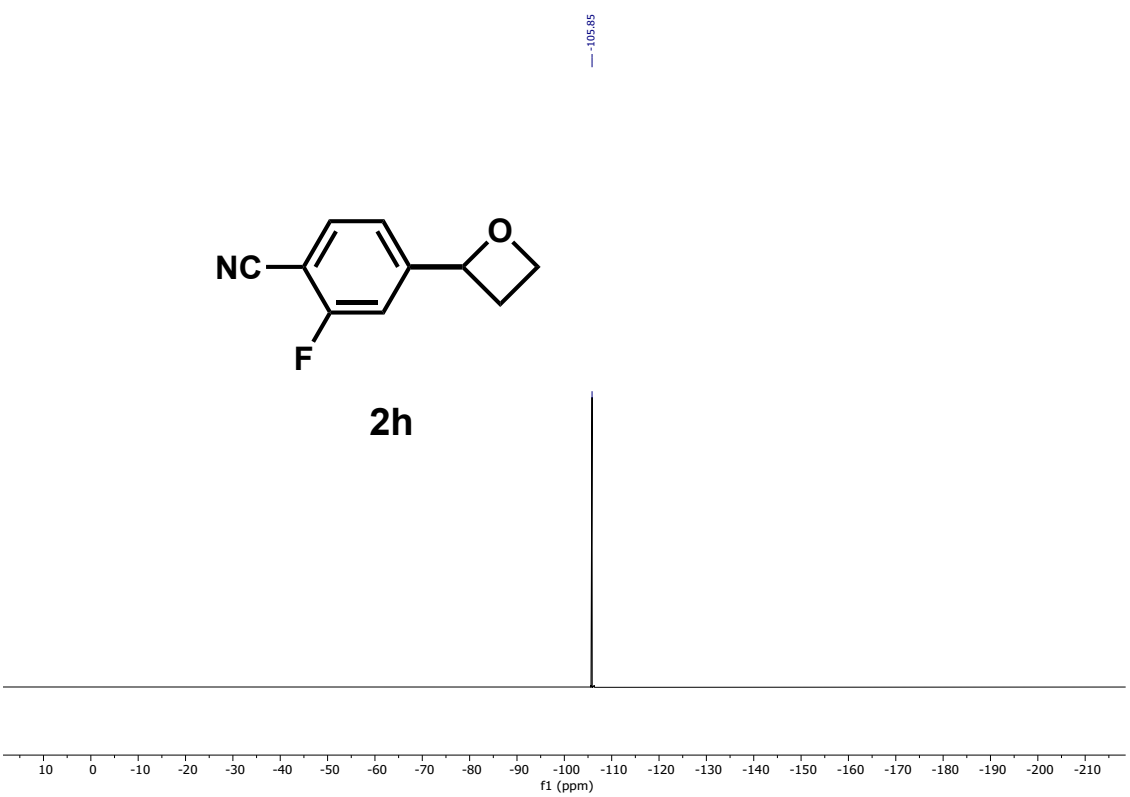

160.63
158.08
152.52
152.44
131.72
123.50
123.35
120.62
120.58
113.88
113.65
81.08
81.06
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
68.60
66.35
45.80
45.78
30.38
0.00
O
N
S
O
O
O
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

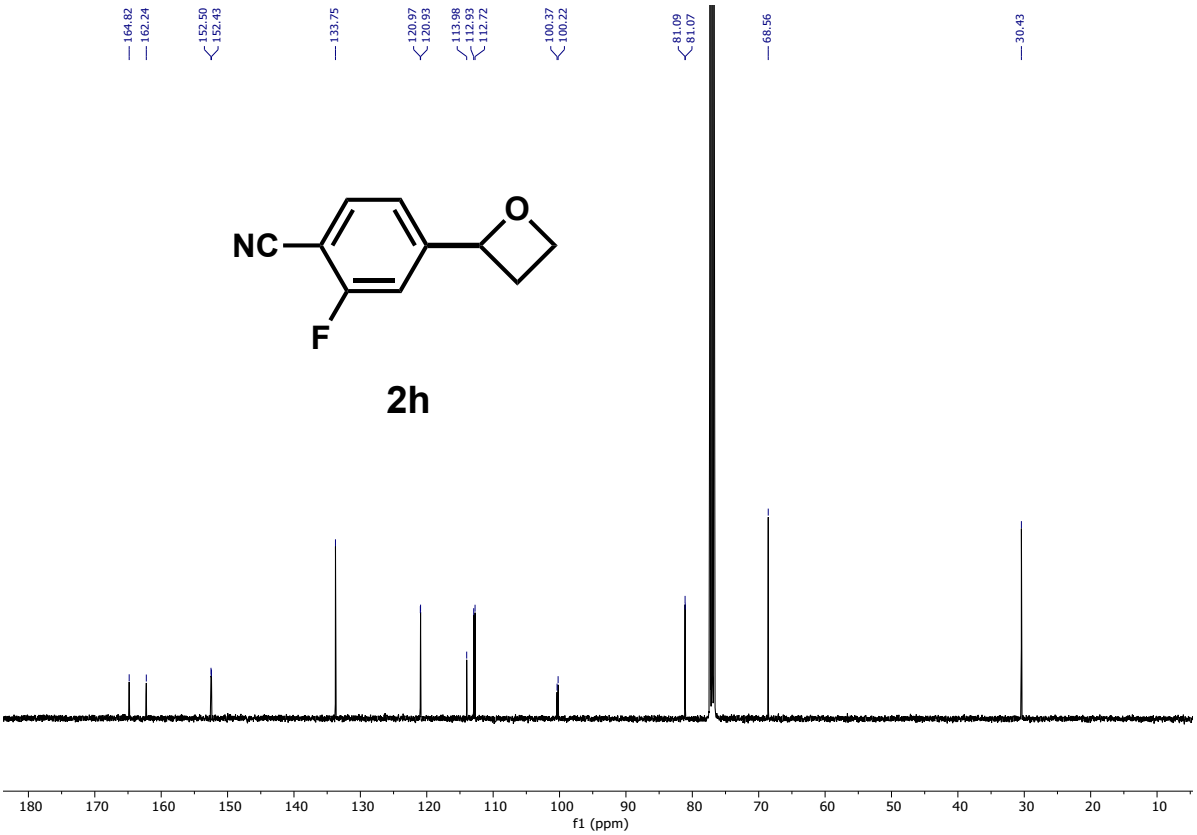
2f

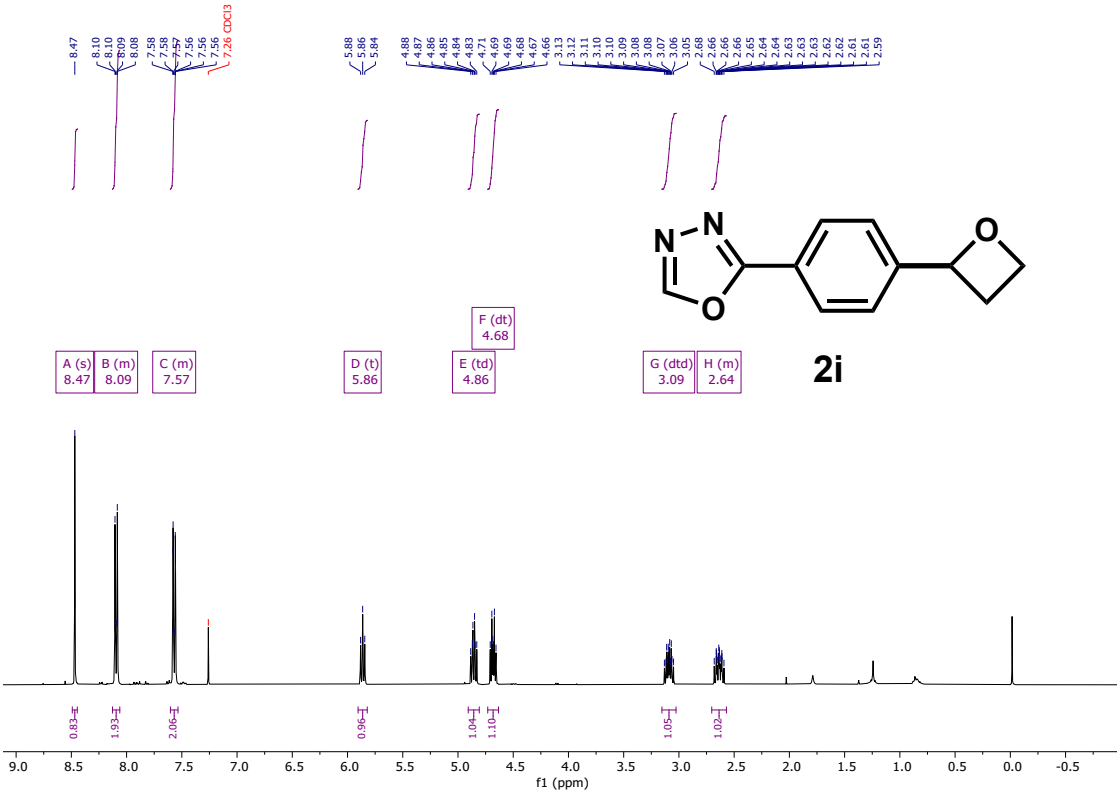

B (dd)
7.64
F (dt)
4.67
H (t)
3.39
A (m)
7.81
C (m)
7.40
D (t)
5.86
E (td)
4.85
G (m)
3.52
I (dtd)
3.09
J (dddd)
2.60
2g
f1 (ppm)

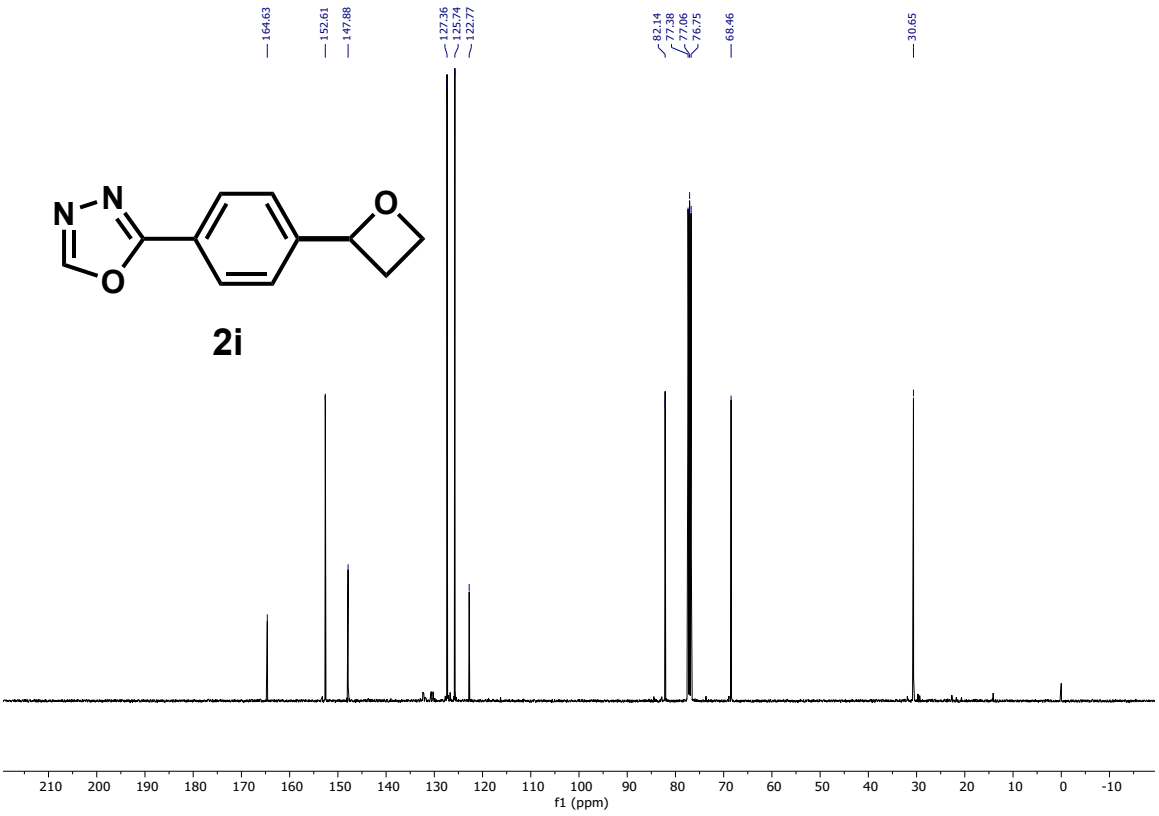

145.38
139.23
136.54
130.38
127.38
118.05
81.75
68.42
50.94
30.68
25.18
2g
f1 (ppm)

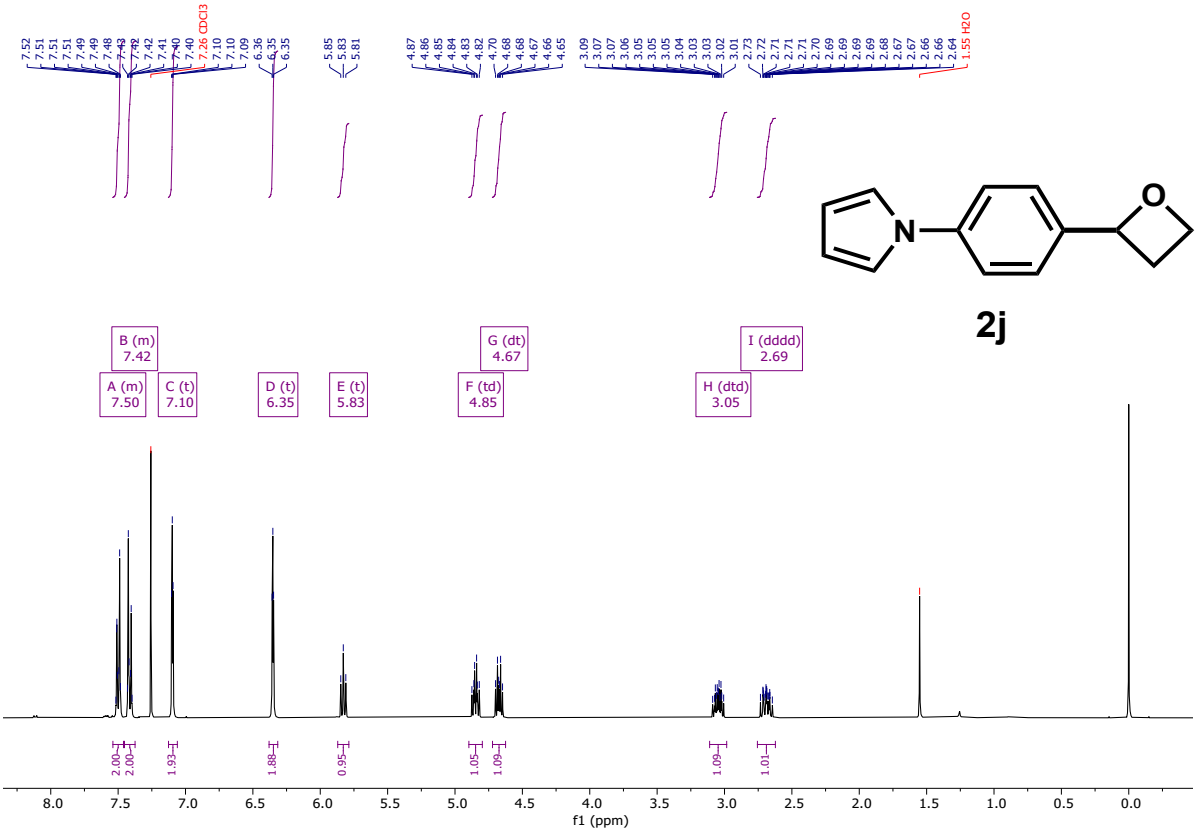

NC
O
F
B (m) 7.82
F (dt) 4.67
A (dd) 7.64
C (m) 2.25
D (t) 5.82
E (ddd) 4.86
G (dtd) 3.13
H (dddd) 2.57
2h
f1 (ppm)

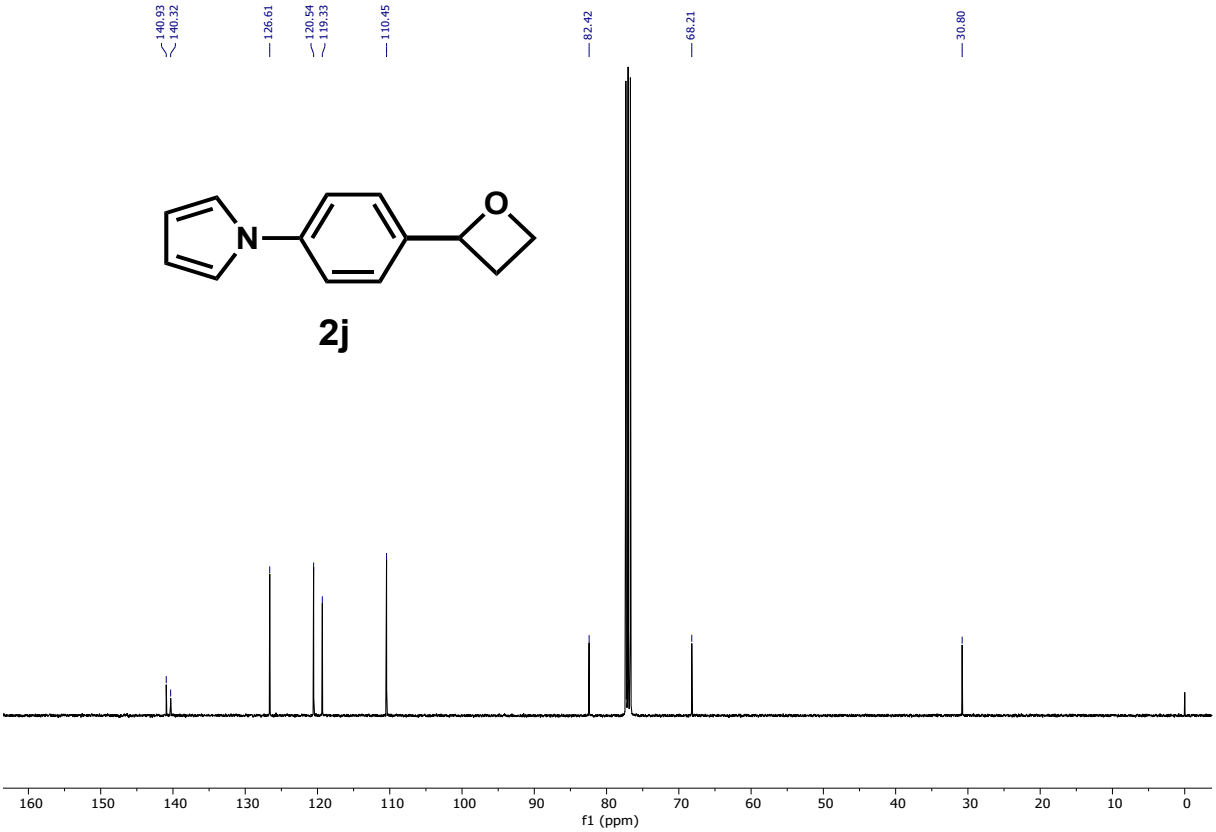

-105.85
NC
O
F
2h
f1 (ppm)

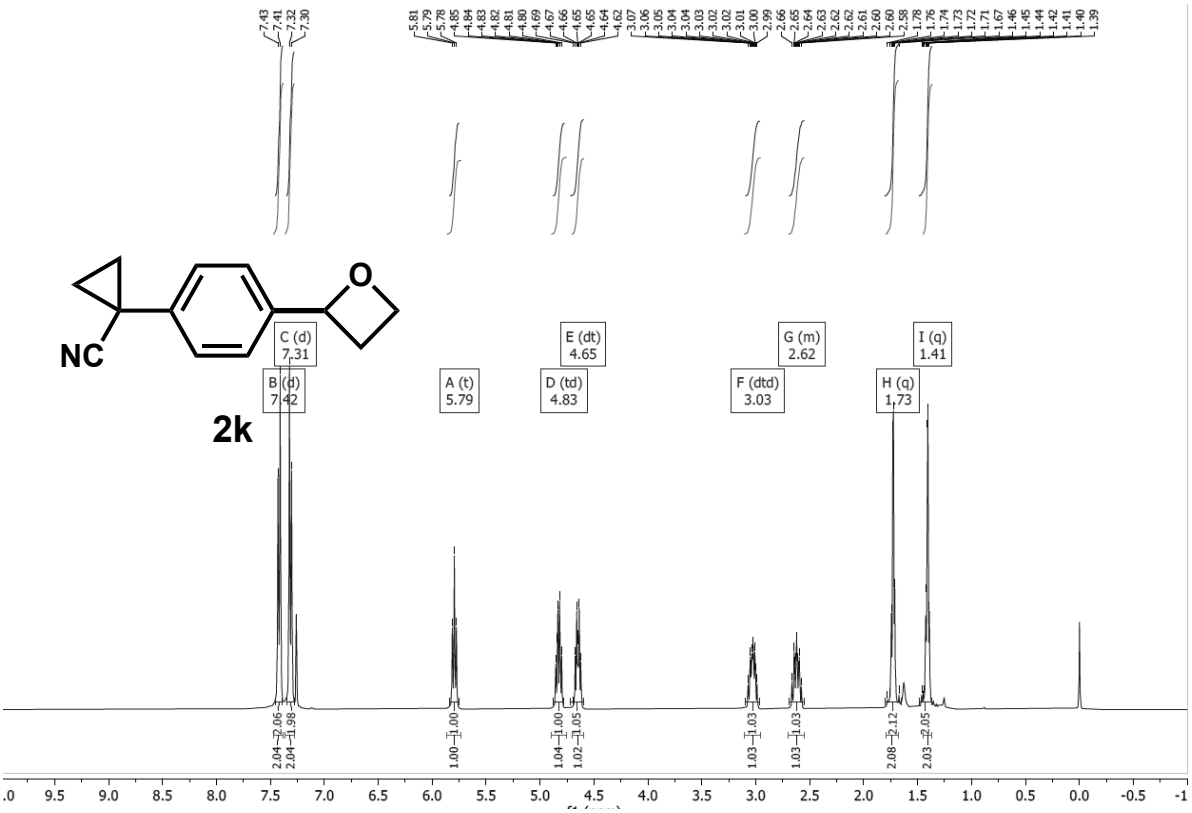

164.82
162.24
152.50
152.43
133.75
120.97
120.93
113.98
112.93
112.72
100.37
100.22
81.09
81.07
68.56
30.43
NC
F
O
2h
f1 (ppm)

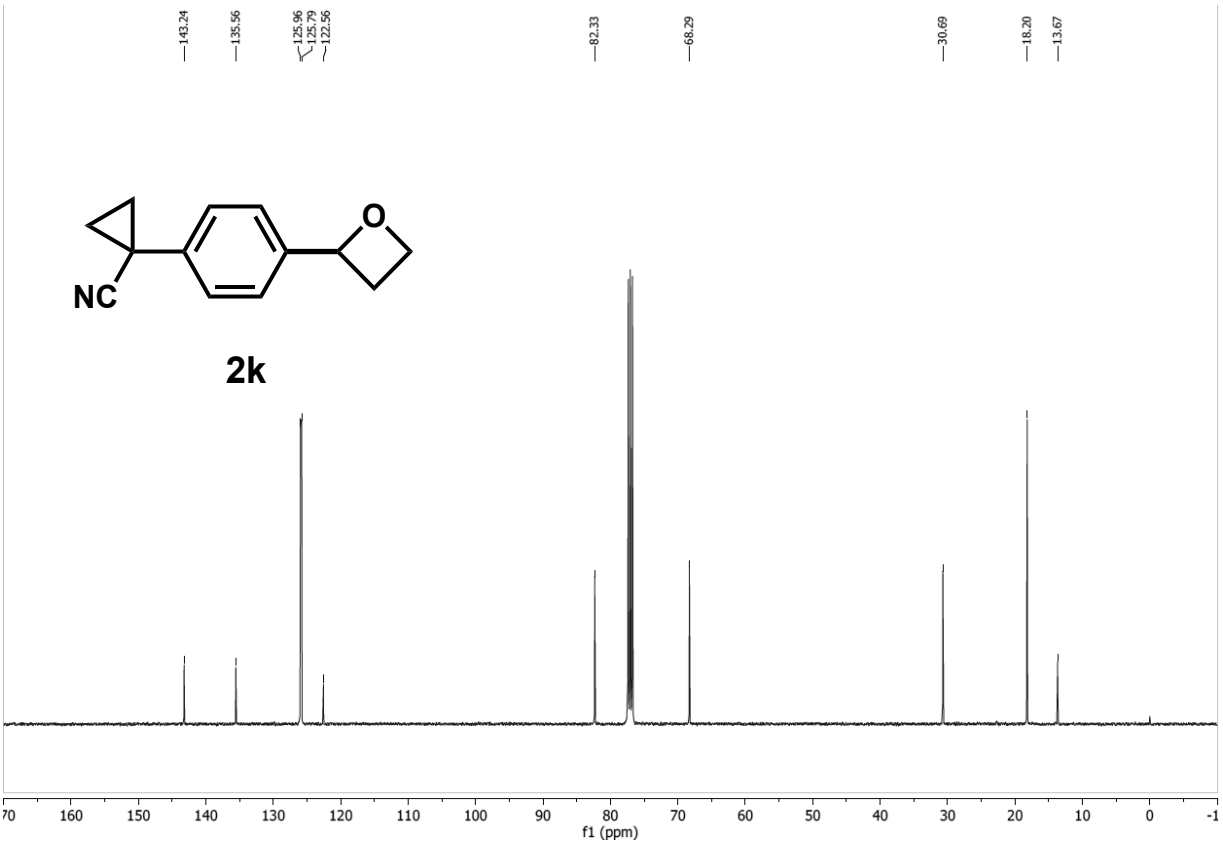

8.47
8.10
8.10
8.09
8.08
7.58
7.58
7.57
7.56
7.56
7.56
7.26 CDCl3
5.88
5.86
5.84
4.88
4.87
4.86
4.85
4.84
4.83
4.71
4.69
4.69
4.68
4.67
4.66
3.13
3.12
3.11
3.10
3.10
3.09
3.08
3.08
3.07
3.06
3.05
2.68
2.66
2.66
2.66
2.65
2.64
2.64
2.63
2.63
2.63
2.62
2.62
2.61
2.61
2.59
N
N
O
O
A (s) 8.47
B (m) 8.09
C (m) 7.57
D (t) 5.86
E (td) 4.86
F (dt) 4.68
G (dtd) 3.09
H (m) 2.64
2i
0.83
1.93
2.06
0.96
1.04
1.10
1.05
1.02
f1 (ppm)

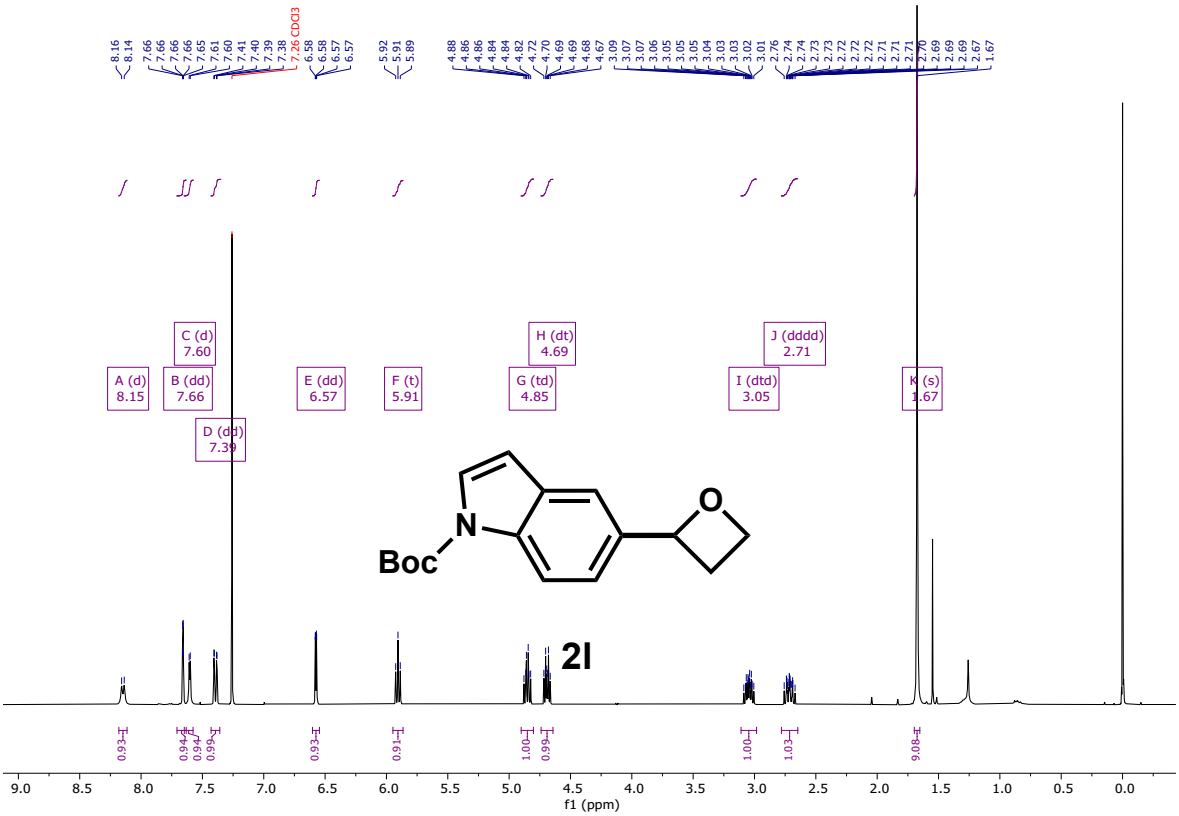

164.63
152.61
147.88
127.36
125.74
122.77
82.14
77.38
77.06
76.75
68.46
30.65
N
N
O
O
2i
f1 (ppm)

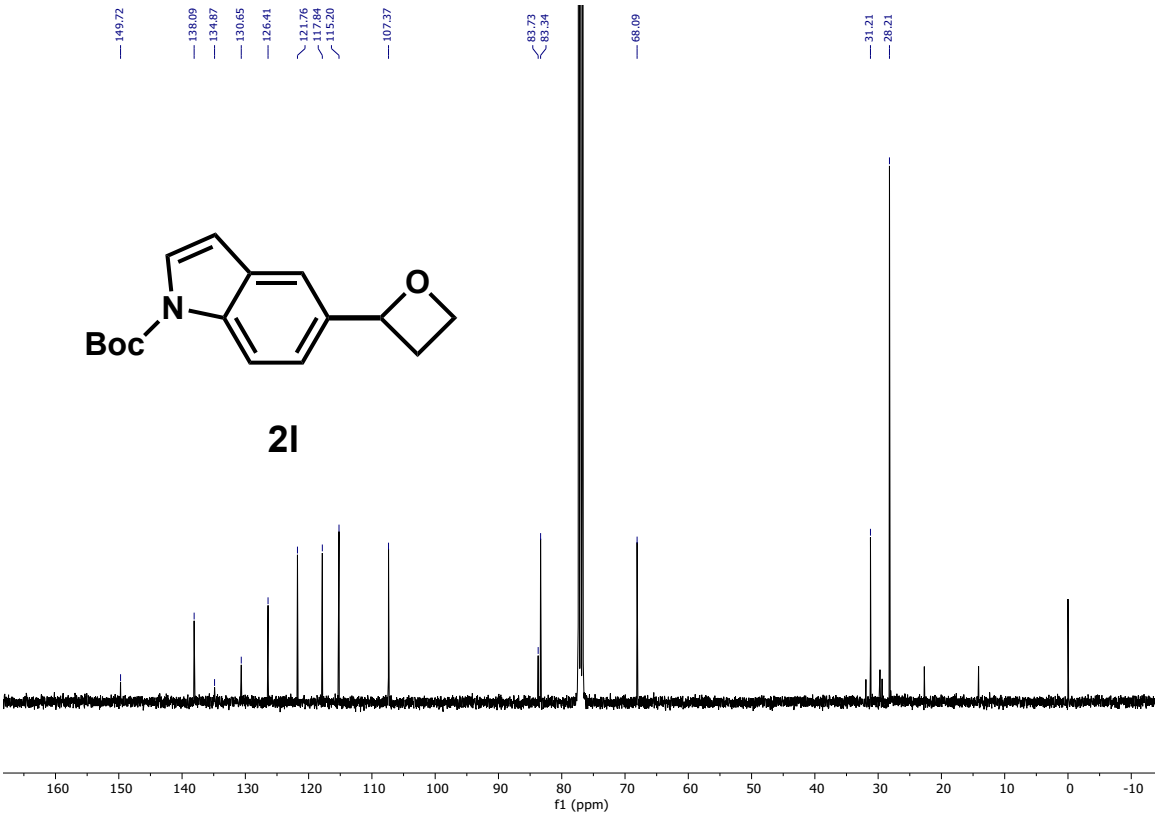

7.26 CDCl3
1.55 H2O
N
O
2j
B (m) 7.42
A (m) 7.50
C (t) 7.10
D (t) 6.35
E (t) 5.83
G (dt) 4.67
F (td) 4.85
H (dtd) 3.05
I (dddd) 2.69
f1 (ppm)


140.93
140.32
126.61
120.54
119.33
110.45
82.42
68.21
30.80
N
O
2j
f1 (ppm)

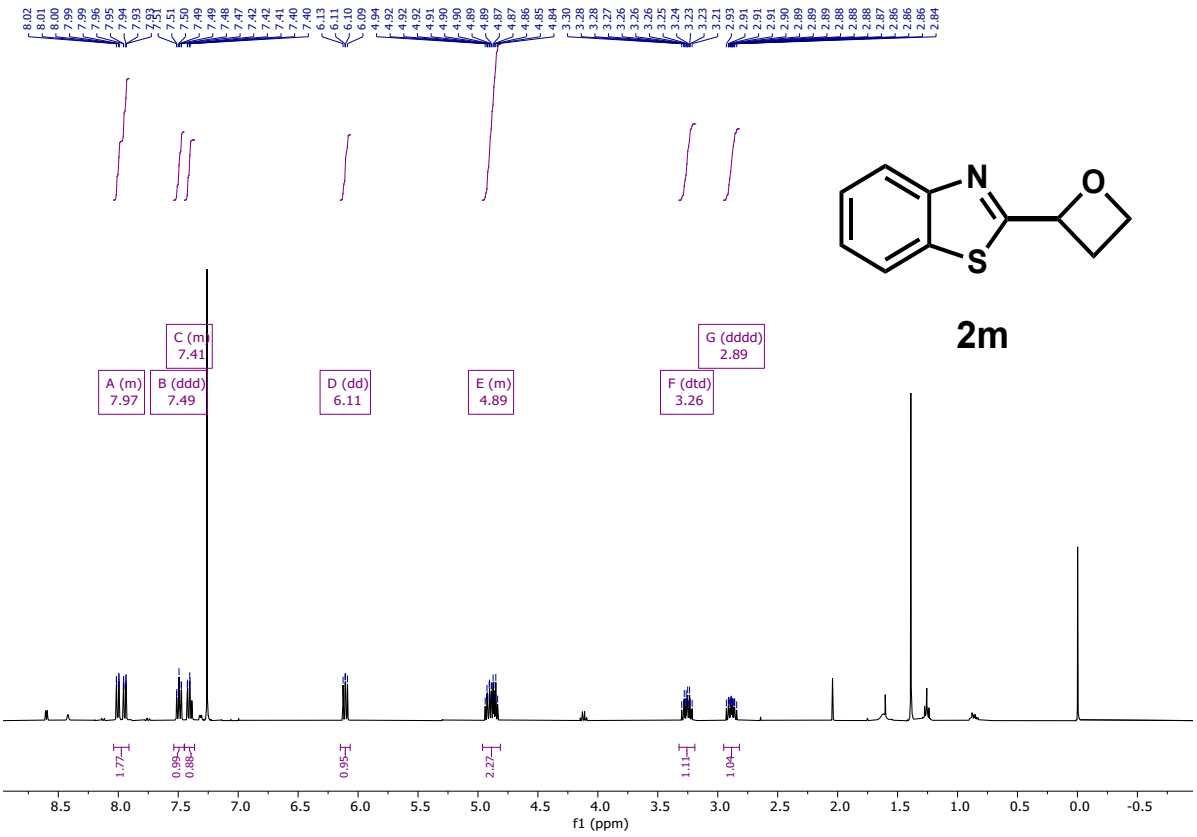

NC
O
2k
C (d) 7,31
B (d) 7.42
A (t) 5.79
E (dt) 4.65
D (td) 4.83
G (m) 2.62
F (dtd) 3.03
I (q) 1.41
H (q) 1,73


143.24
135.56
125.96
125.79
122.56
82.33
68.29
30.69
18.20
13.67
NC
O
2k
f1 (ppm)


A (d) 8.15
B (dd) 7.66
C (d) 7.60
D (dd) 7.39
E (dd) 6.57
F (t) 5.91
G (td) 4.85
H (dt) 4.69
I (dtd) 3.05
J (dddd) 2.71
K (s) 1.67
Boc
N
O
2l
f1 (ppm)

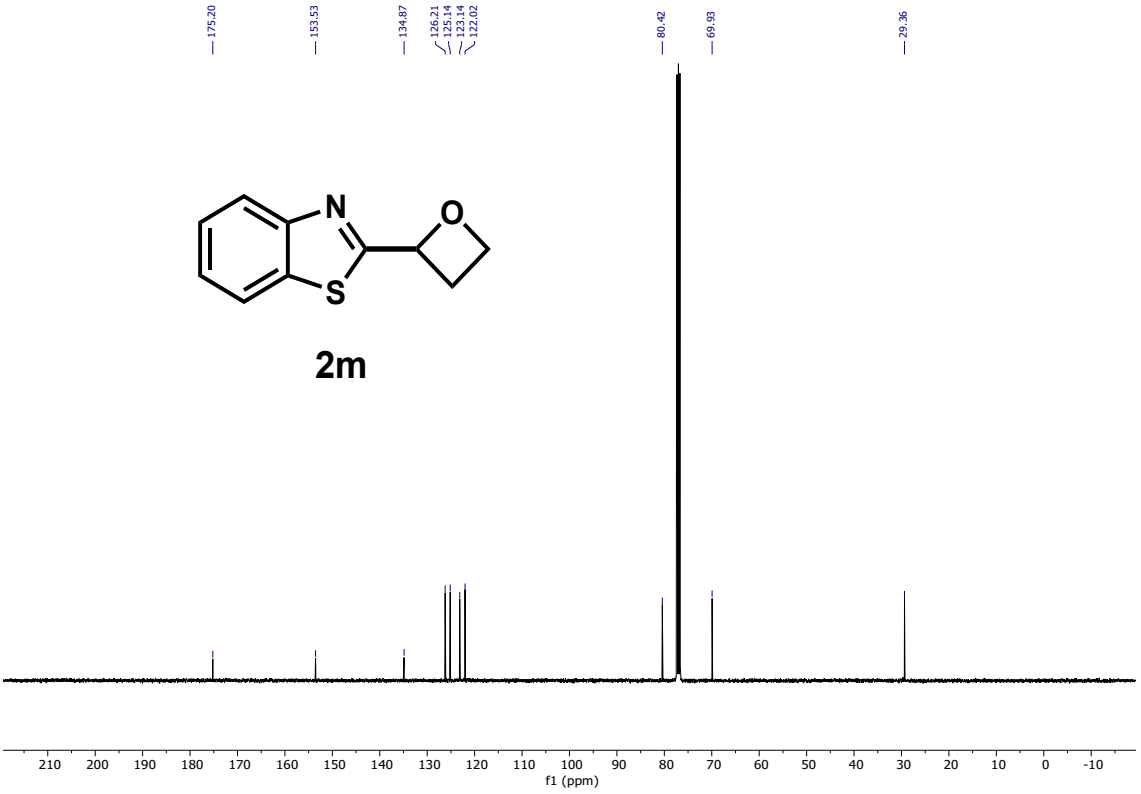

149.72
138.09
134.87
130.65
126.41
121.76
117.84
115.20
107.37
83.73
83.34
68.09
31.21
28.21
Boc
N
O
f1 (ppm)

2l

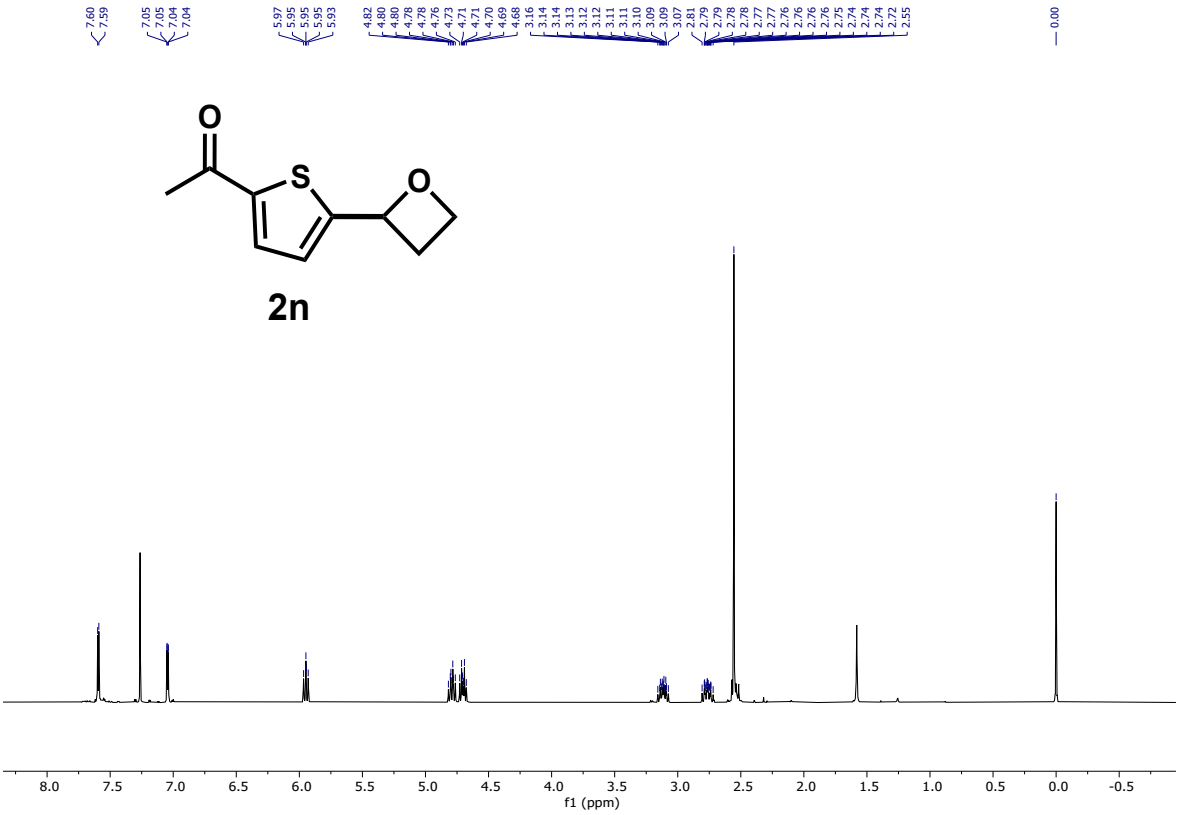

A (m) 7.97
B (ddd) 7.49
C (m) 7.41
D (dd) 6.11
E (m) 4.89
F (dtd) 3.26
G (dddd) 2.89
1.77
0.99
0.88
0.95
2.27
1.11
1.04
f1 (ppm)

N
O
S

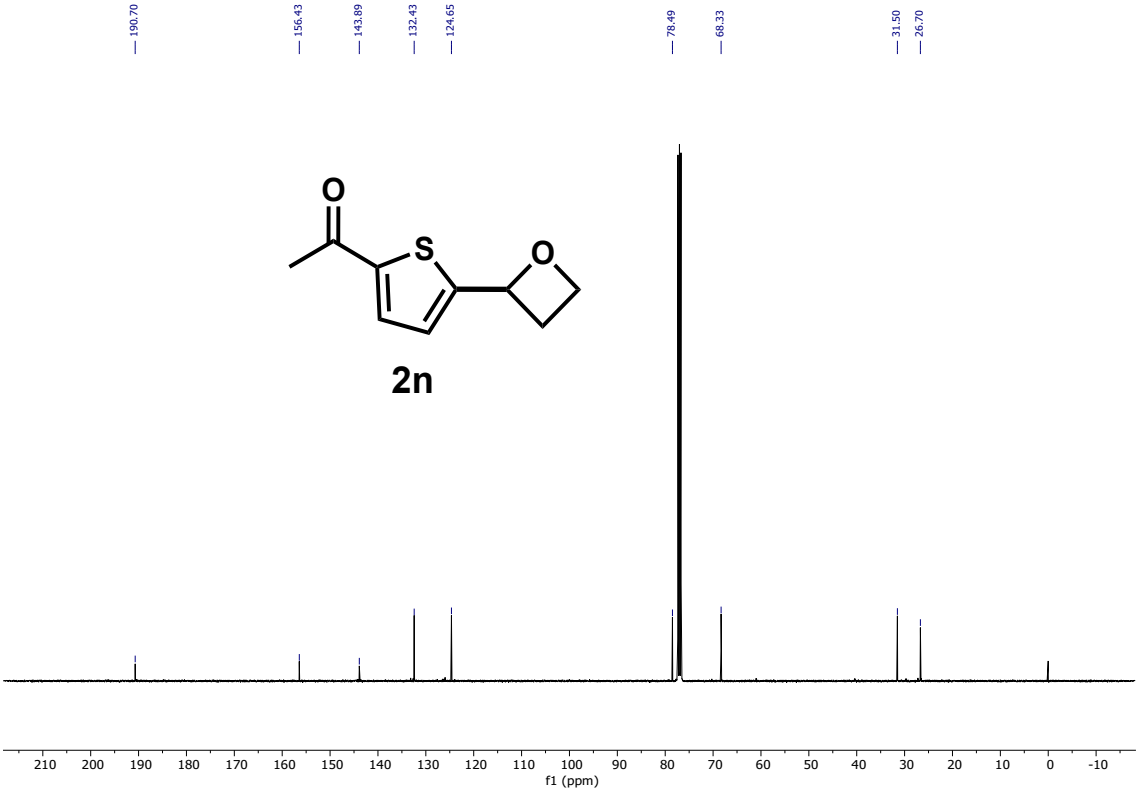
2m


175.20
153.53
134.87
126.21
125.14
123.14
122.02
80.42
69.93
29.36
N
O
S
f1 (ppm)

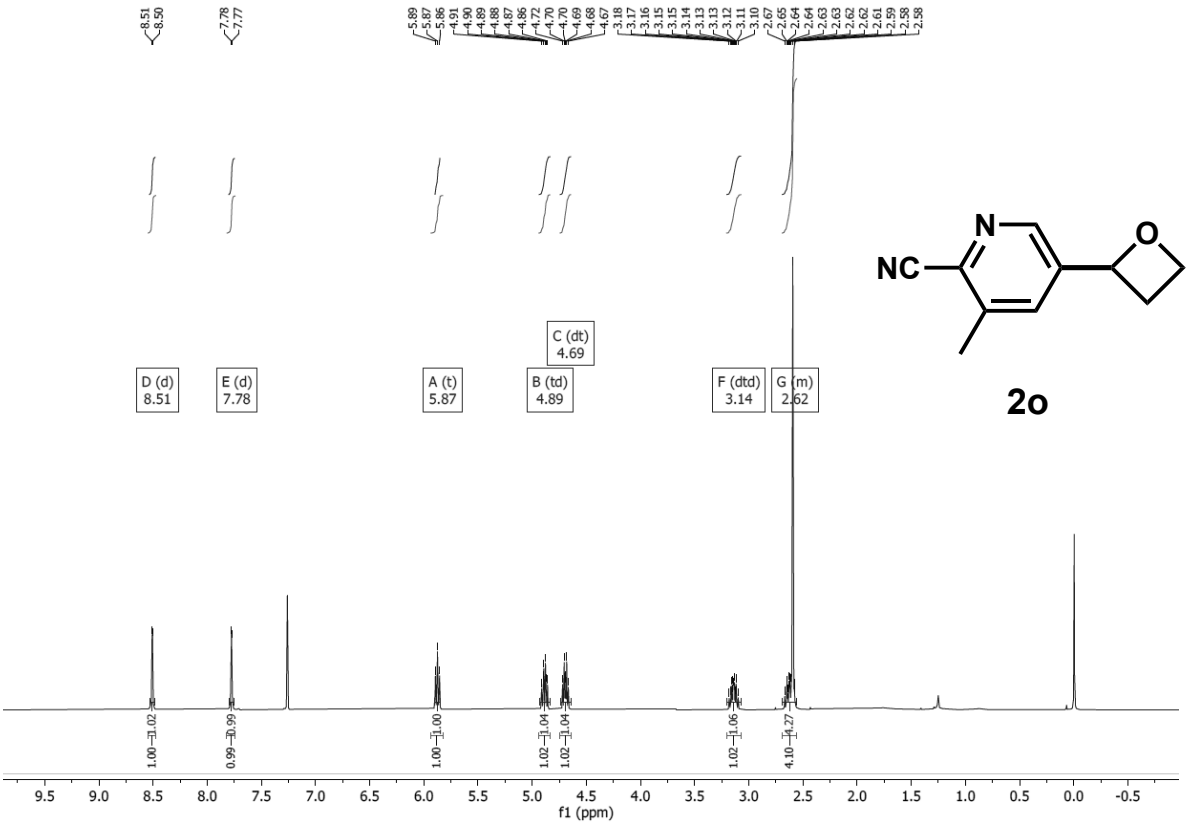
2m


7.60
7.59
7.05
7.05
7.04
7.04
5.97
5.95
5.95
5.95
5.93
4.82
4.80
4.80
4.78
4.78
4.76
4.73
4.71
4.71
4.70
4.69
4.68
3.16
3.14
3.14
3.13
3.12
3.12
3.11
3.11
3.10
3.09
3.09
3.07
2.81
2.79
2.79
2.78
2.78
2.77
2.77
2.76
2.76
2.76
2.76
2.75
2.74
2.74
2.74
2.72
2.55
0.00
O
S
O
f1 (ppm)

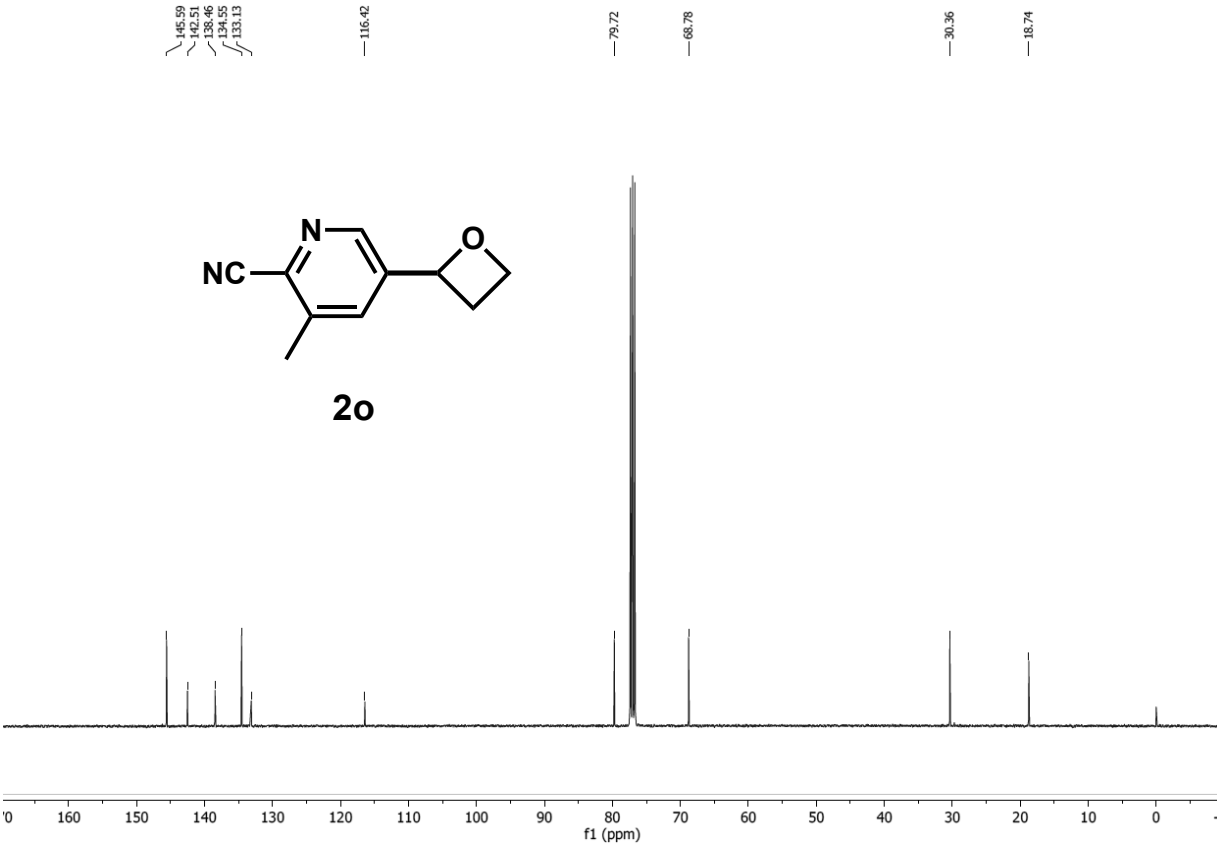
2n

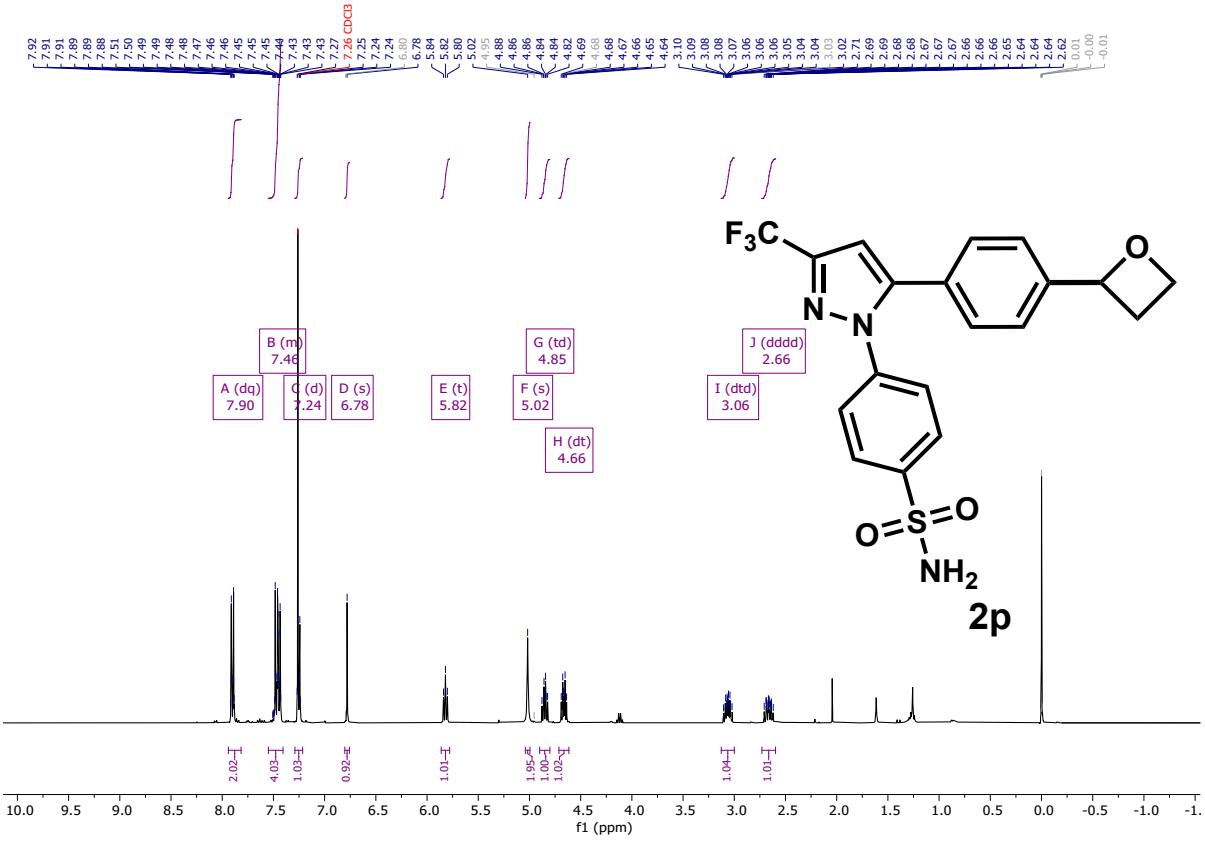

190.70
156.43
143.89
132.43
124.65
78.49
68.33
31.50
26.70
O
S
O
f1 (ppm)

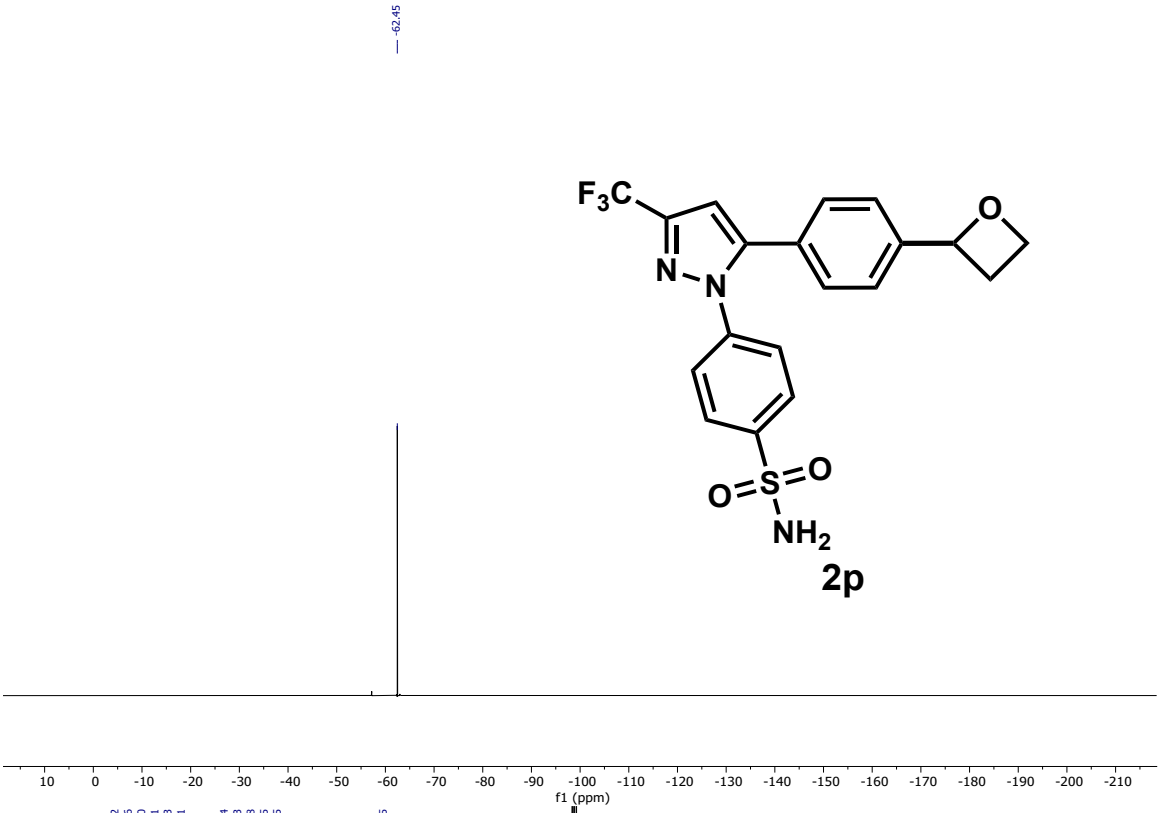
2n

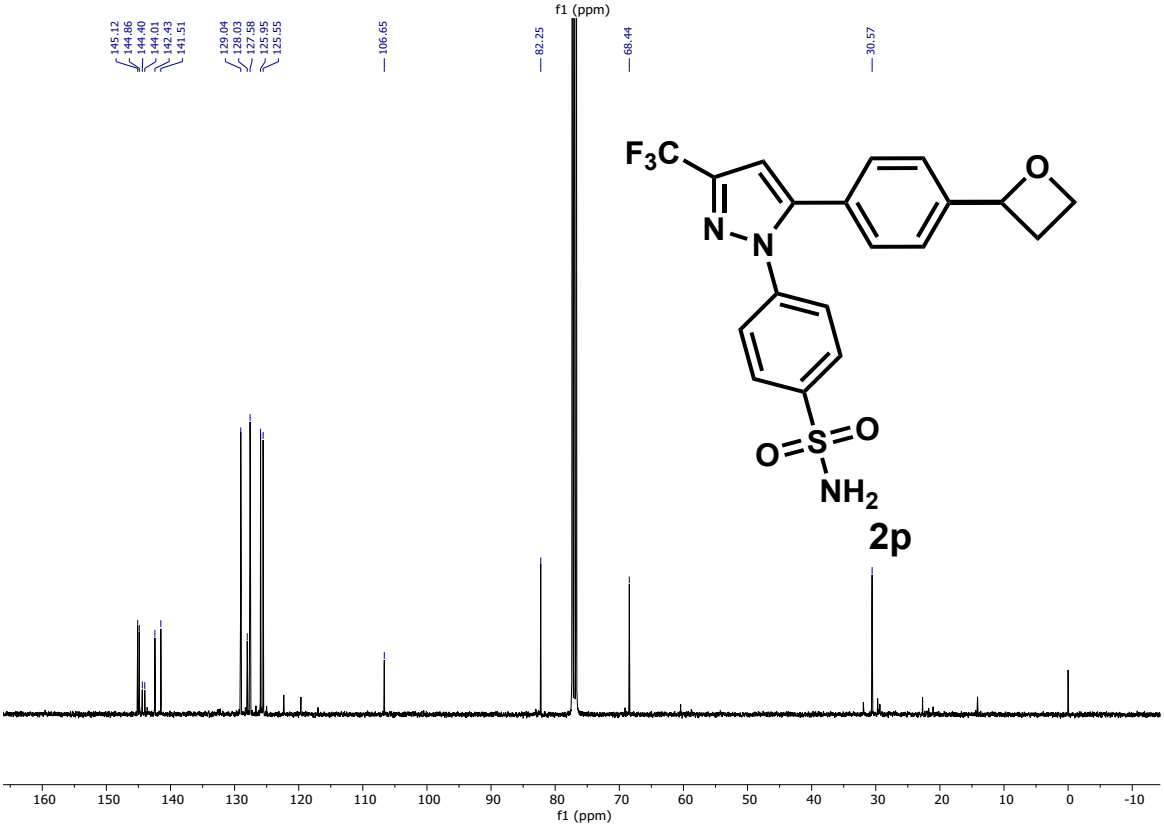

8.51
8.50
7.78
7.77
N
NC
O
D (d)
8.51
E (d)
7.78
A (t)
5.87
C (dt)
4.69
B (td)
4.89
F (dtd)
3.14
G (m)
2.62
f1 (ppm)

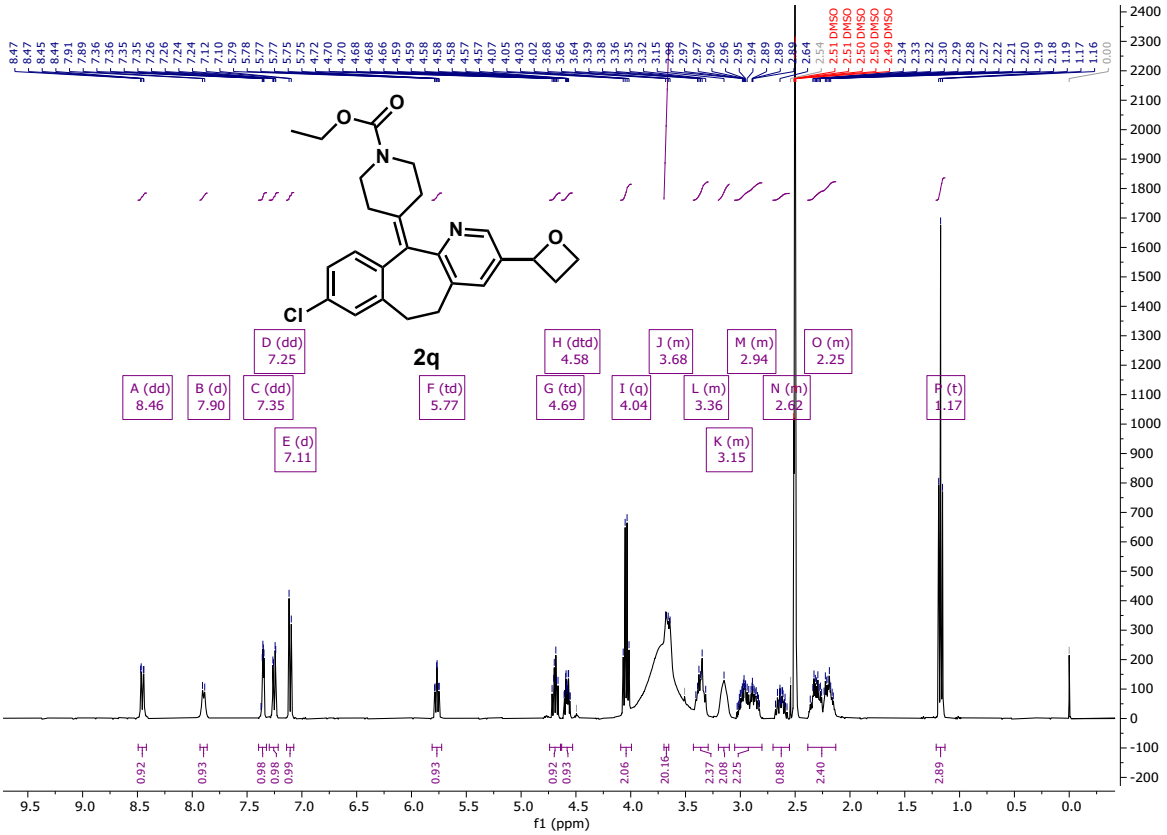
2o

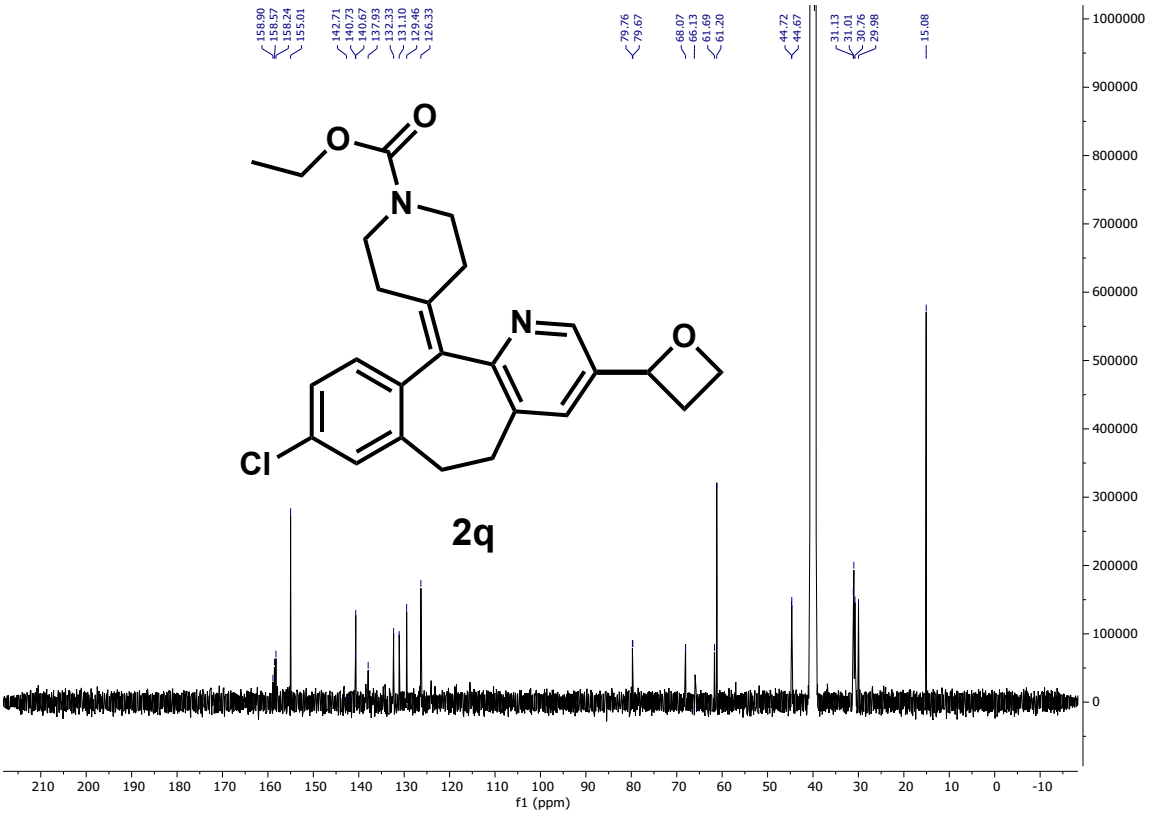

145.59
142.51
138.46
134.55
133.13
116.42
79.72
68.78
30.36
18.74
NC
N
O
2o
f1 (ppm)

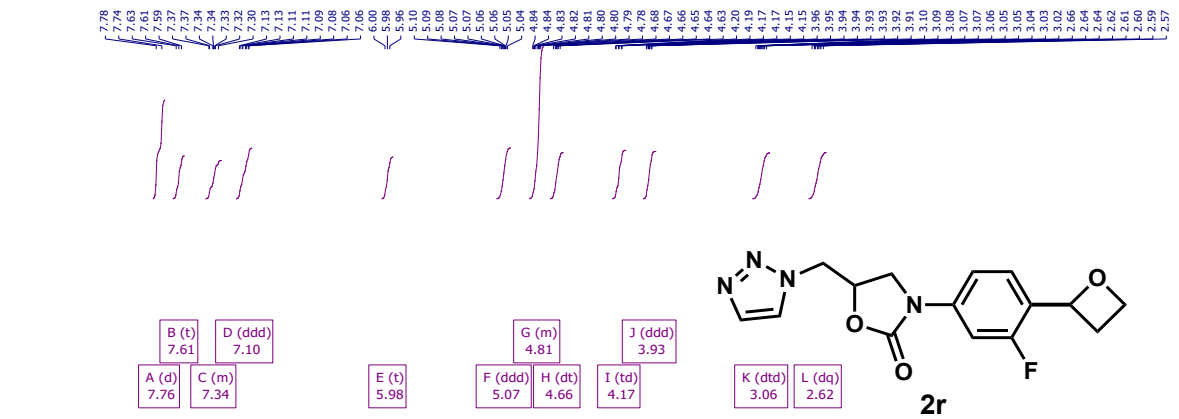

CDCl3
A (dq) 7.90
B (m) 7.46
C (d) 7.24
D (s) 6.78
E (t) 5.82
F (s) 5.02
G (td) 4.85
H (dt) 4.66
I (dtd) 3.06
J (dddd) 2.66
F3C
N
O
O=S=O
NH2
2p
f1 (ppm)

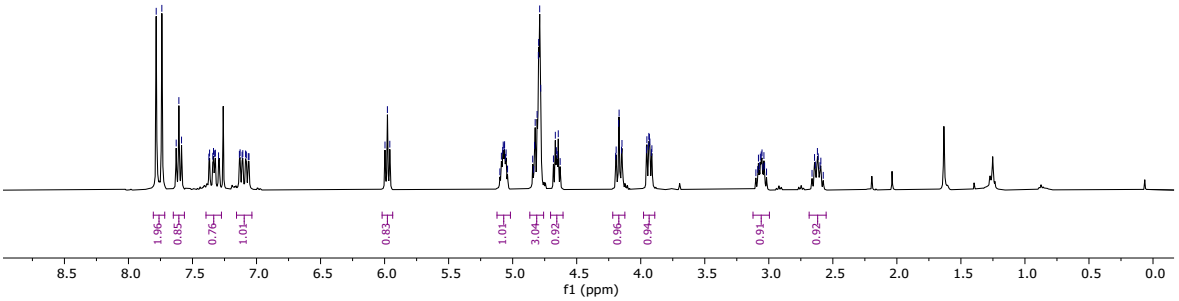

-62.45
F3C
N
N
O
O
S
O
NH2
2p
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

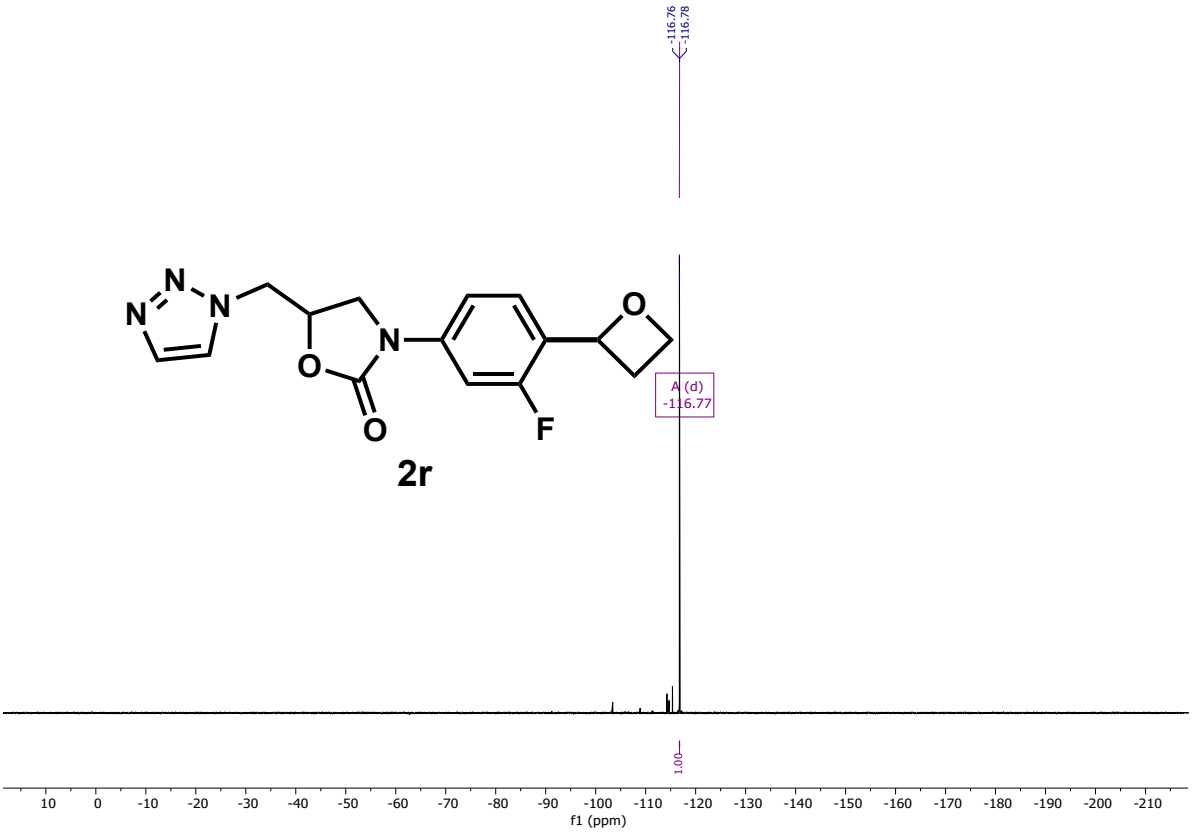

145.12
144.86
144.40
144.01
142.43
141.51
129.04
128.03
127.58
125.95
125.55
106.65
82.25
68.44
30.57
F3C
N
N
O
O
S
O
NH2
2p
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

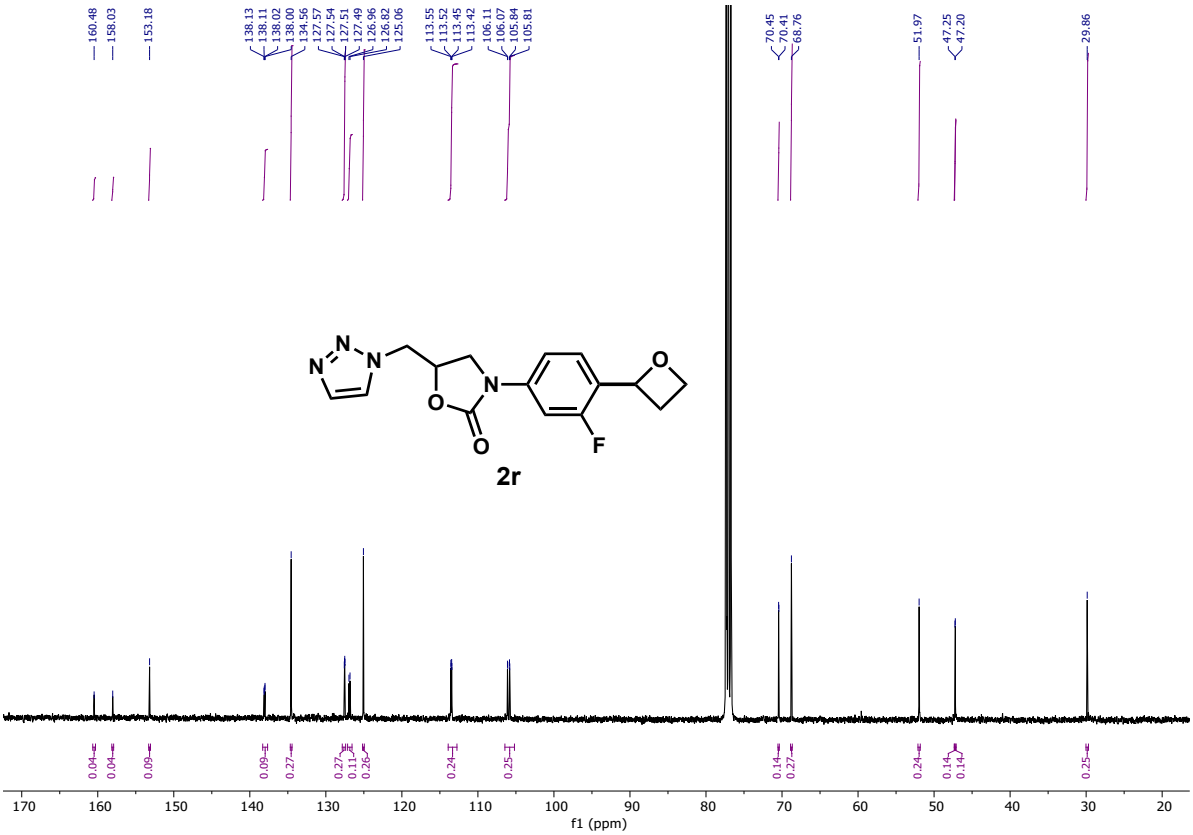

2q
A (dd) 8.46
B (d) 7.90
C (dd) 7.35
D (dd) 7.25
E (d) 7.11
F (td) 5.77
G (td) 4.69
H (dtd) 4.58
I (q) 4.04
J (m) 3.68
K (m) 3.15
L (m) 3.36
M (m) 2.94
N (m) 2.62
O (m) 2.25
P (t) 1.17
f1 (ppm)

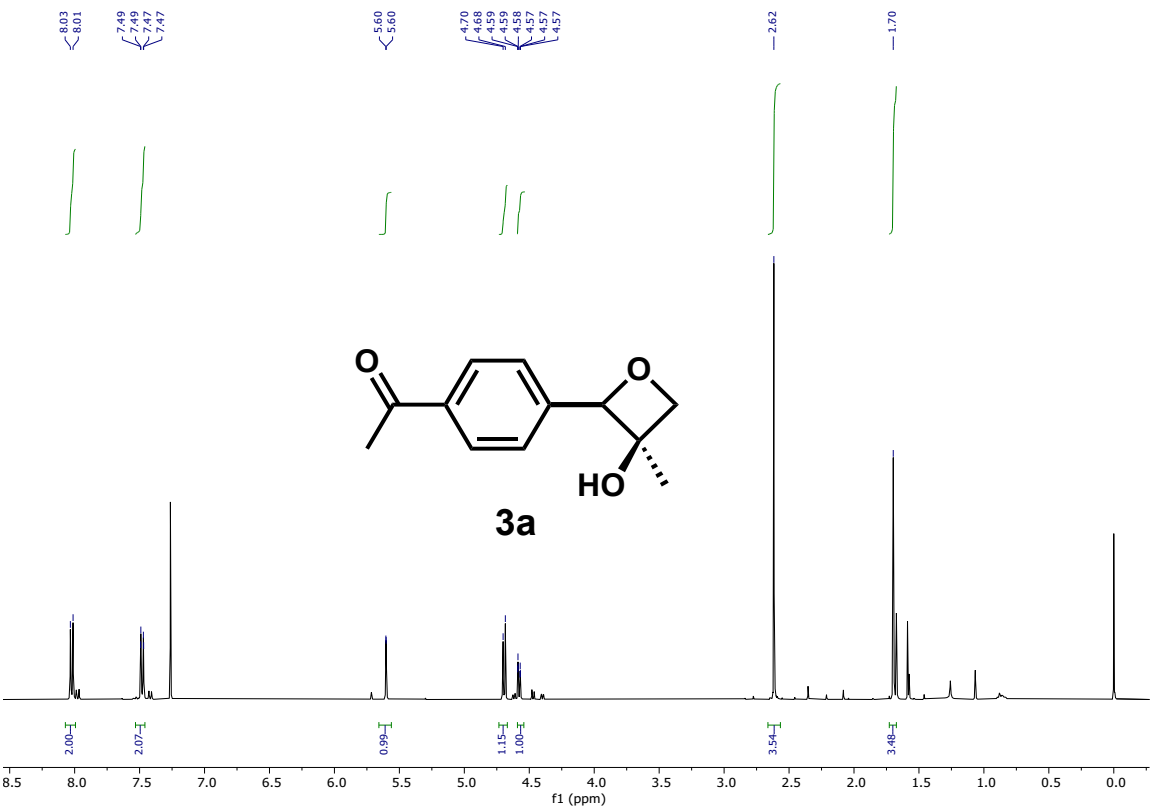

2q
f1 (ppm)


B (t) 7.61
D (ddd) 7.10
G (m) 4.81
J (ddd) 3.93
A (d) 7.76
C (m) 7.34
E (t) 5.98
F (ddd) 5.07
H (dt) 4.66
I (td) 4.17
K (dtd) 3.06
L (dq) 2.62
2r

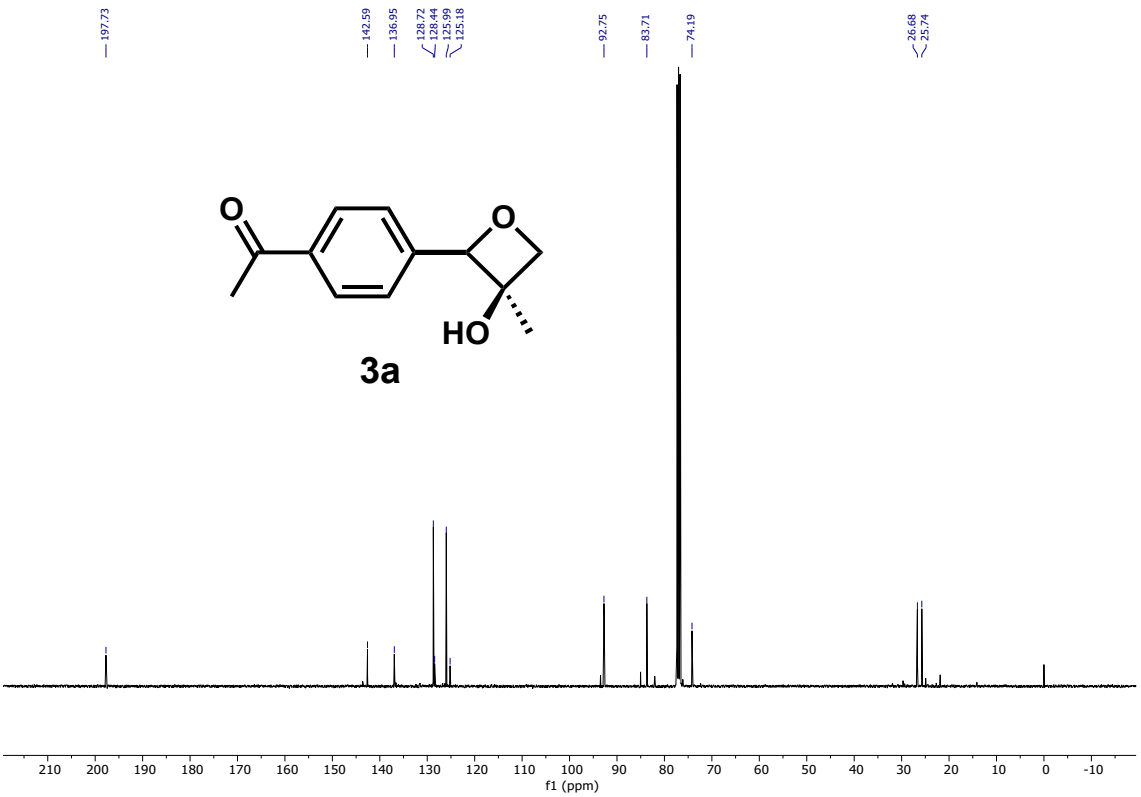

f1 (ppm)


-116.76
-116.78
A (d) -116.77
2r
f1 (ppm)

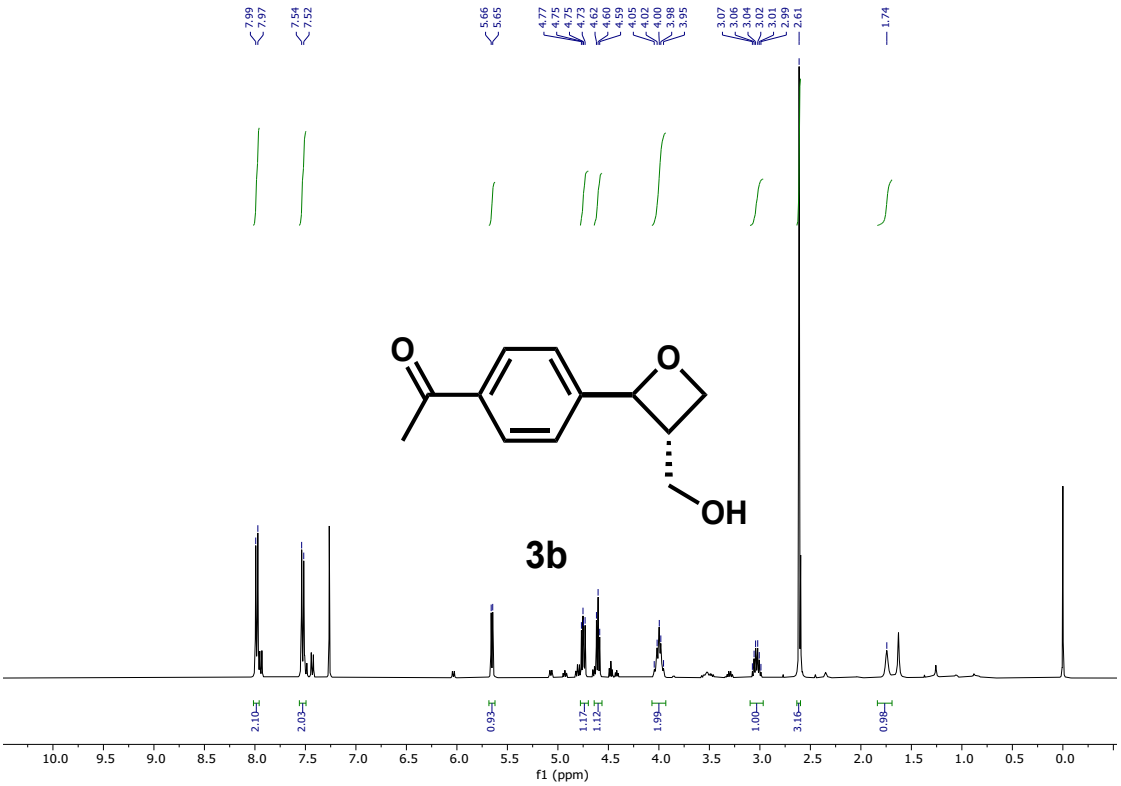


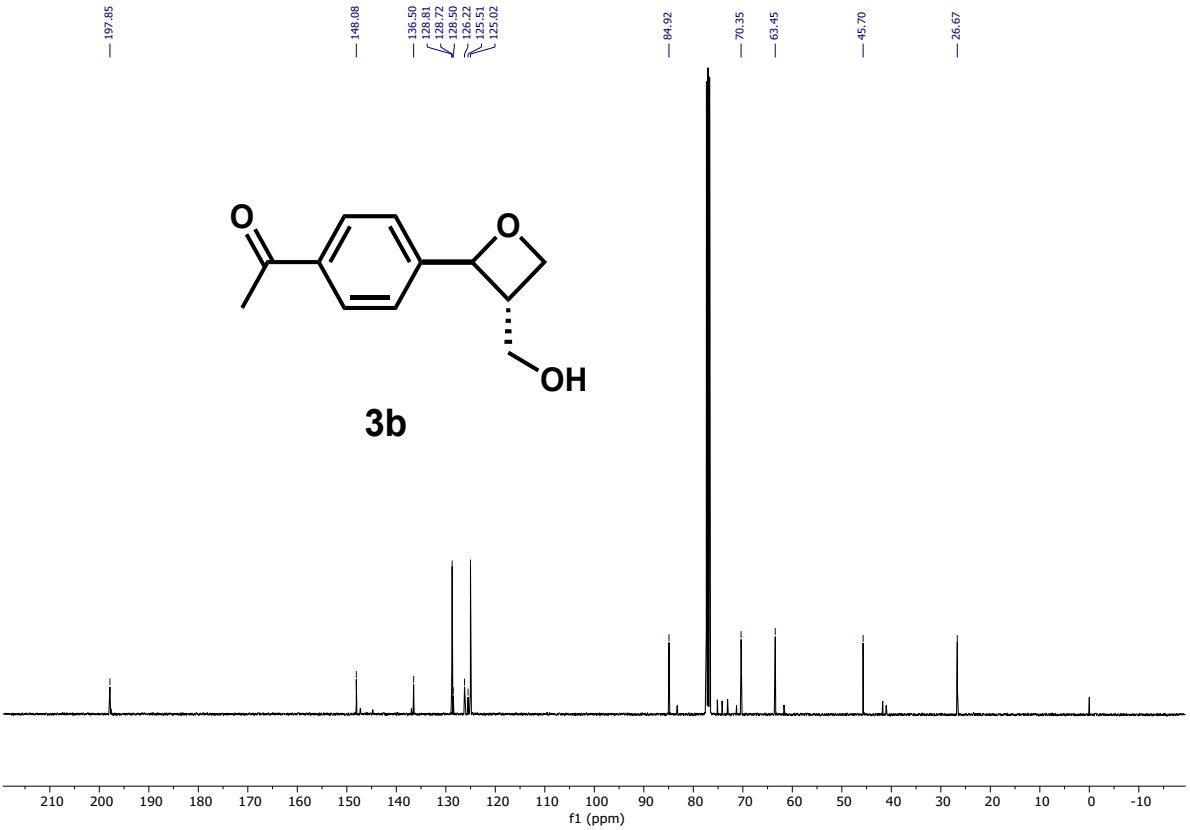


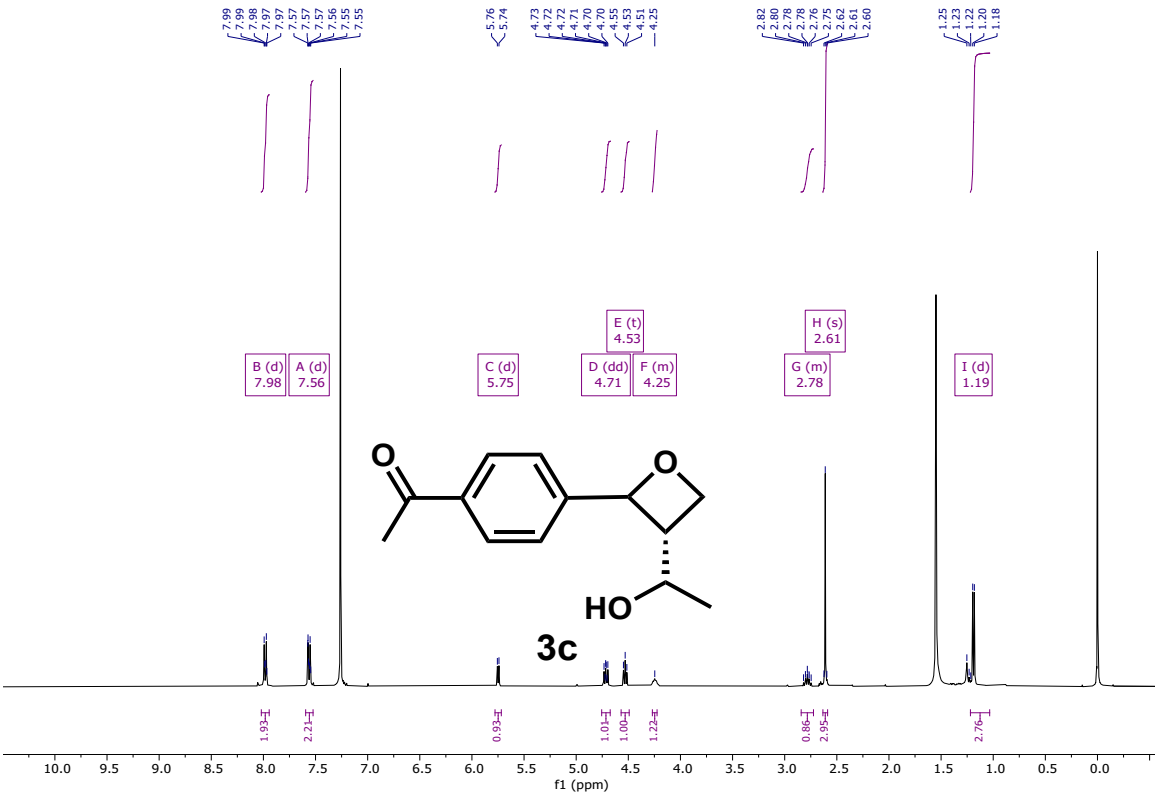
The minor diastereomer was not fully separable by chromatography.

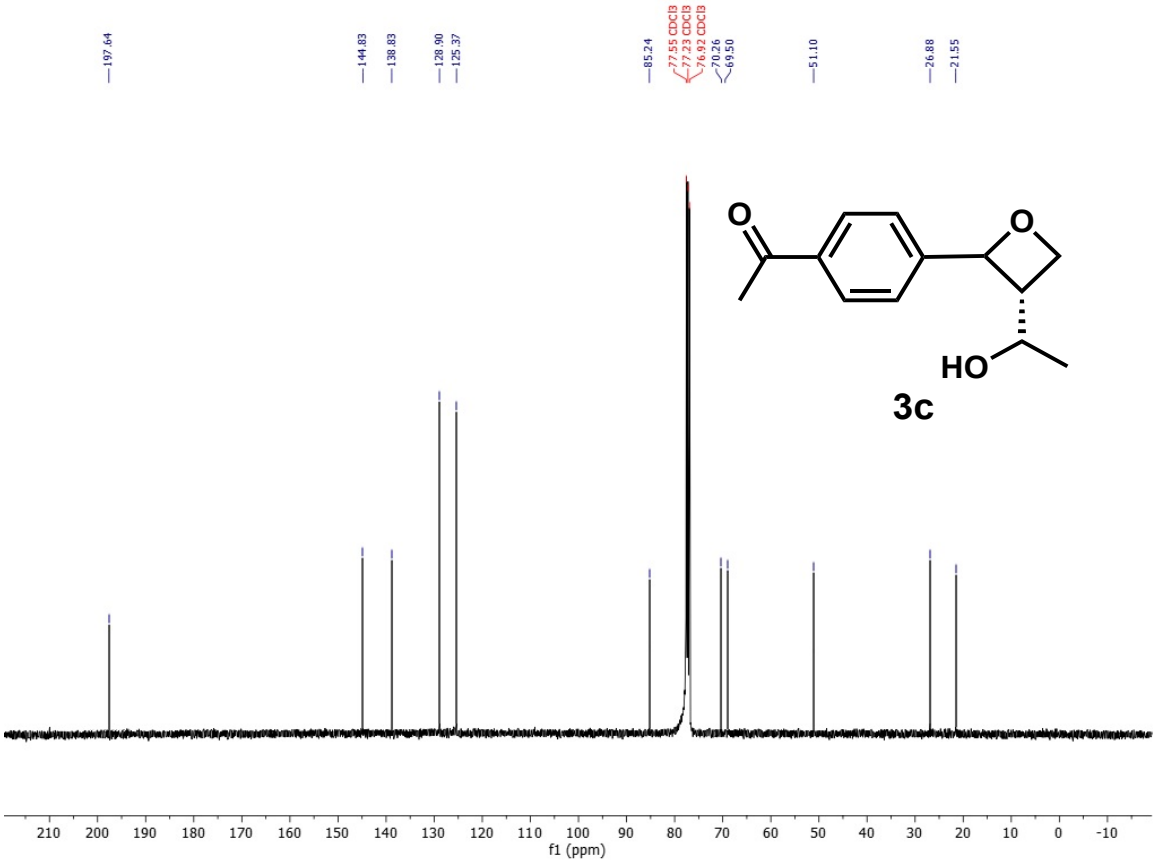


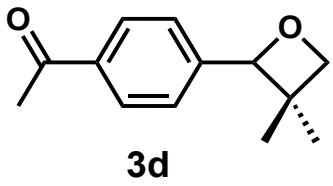
The minor diastereomer was not fully separable by chromatography.



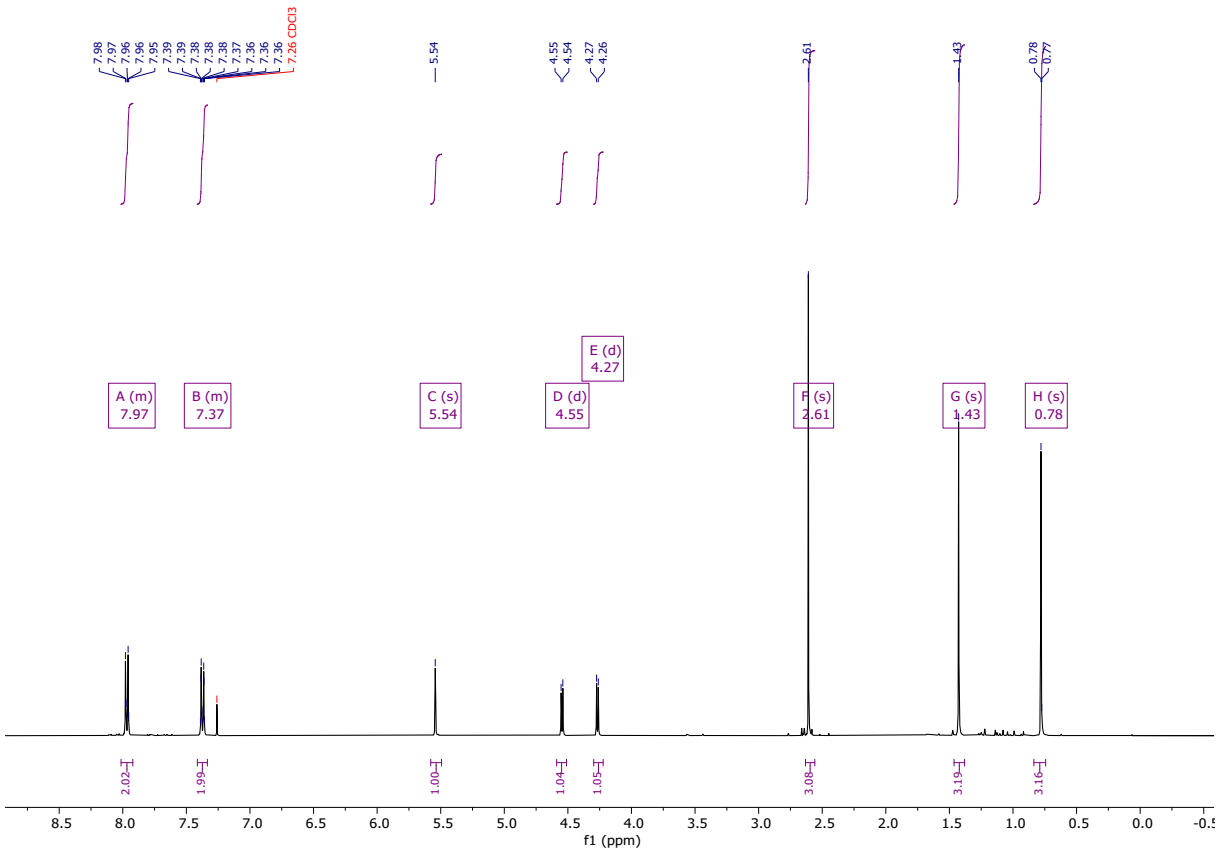

197.85
148.08
136.50
128.81
128.72
128.50
126.22
125.51
125.02
84.92
70.35
63.45
45.70
26.67
O
O
OH
3b
f1 (ppm)

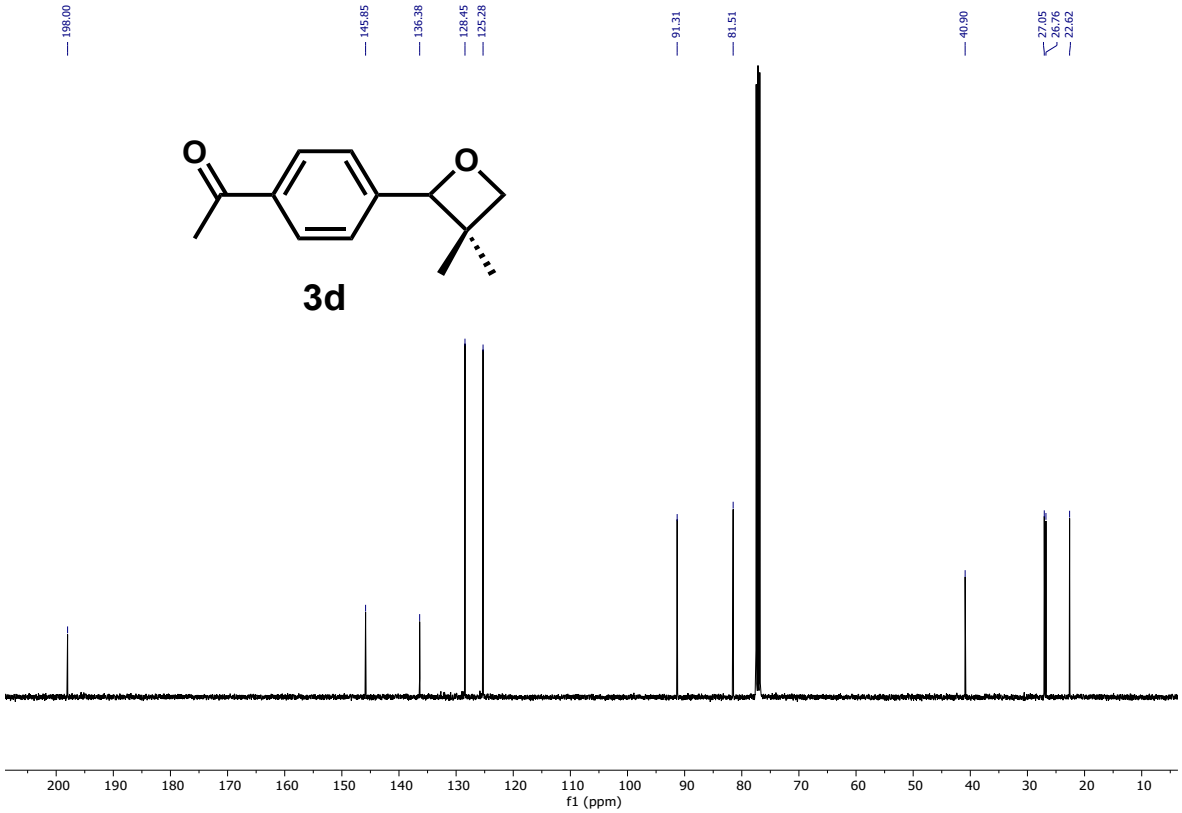

B (d) 7.98
A (d) 7.56
C (d) 5.75
E (t) 4.53
D (dd) 4.71
F (m) 4.25
H (s) 2.61
G (m) 2.78
I (d) 1.19
O
O
HO
3c
f1 (ppm)

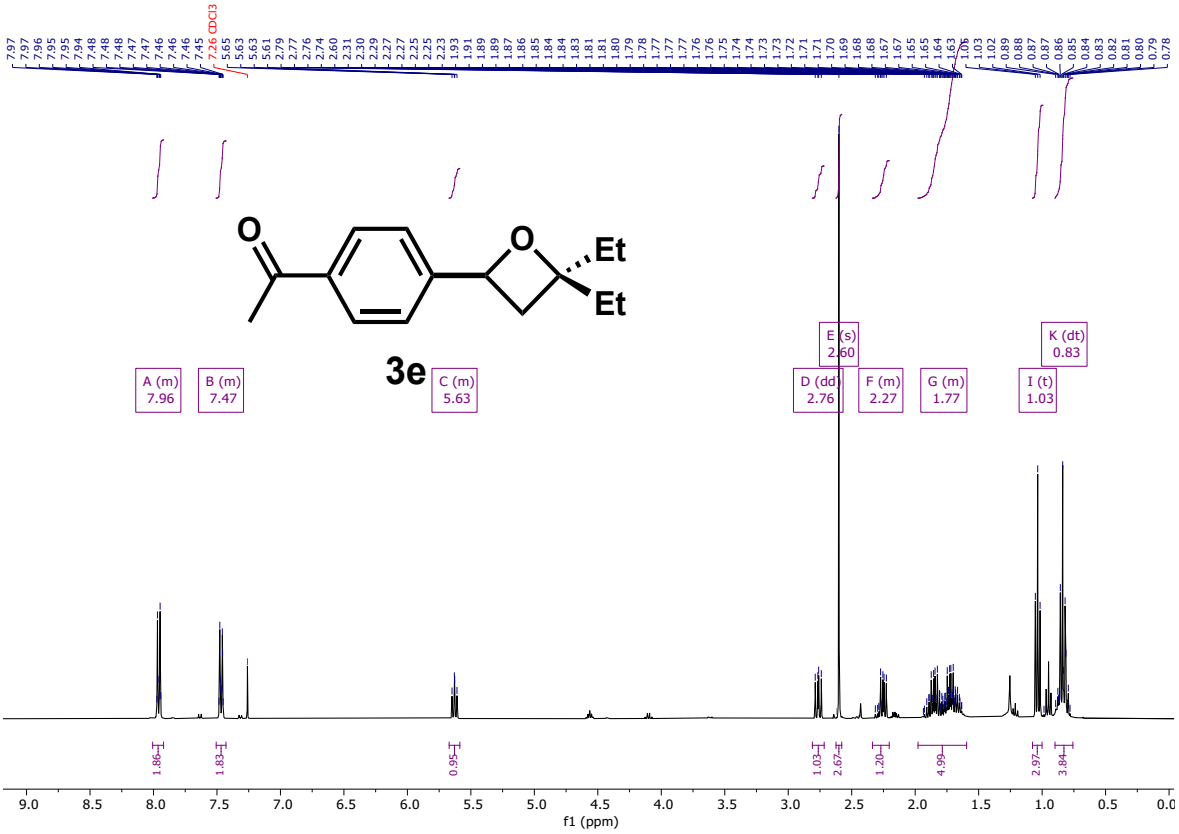

197.64
144.83
138.83
128.90
125.37
85.24
77.55 CDCl3
77.23 CDCl3
76.92 CDCl3
70.26
69.50
51.10
26.88
21.55
O
O
HO
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

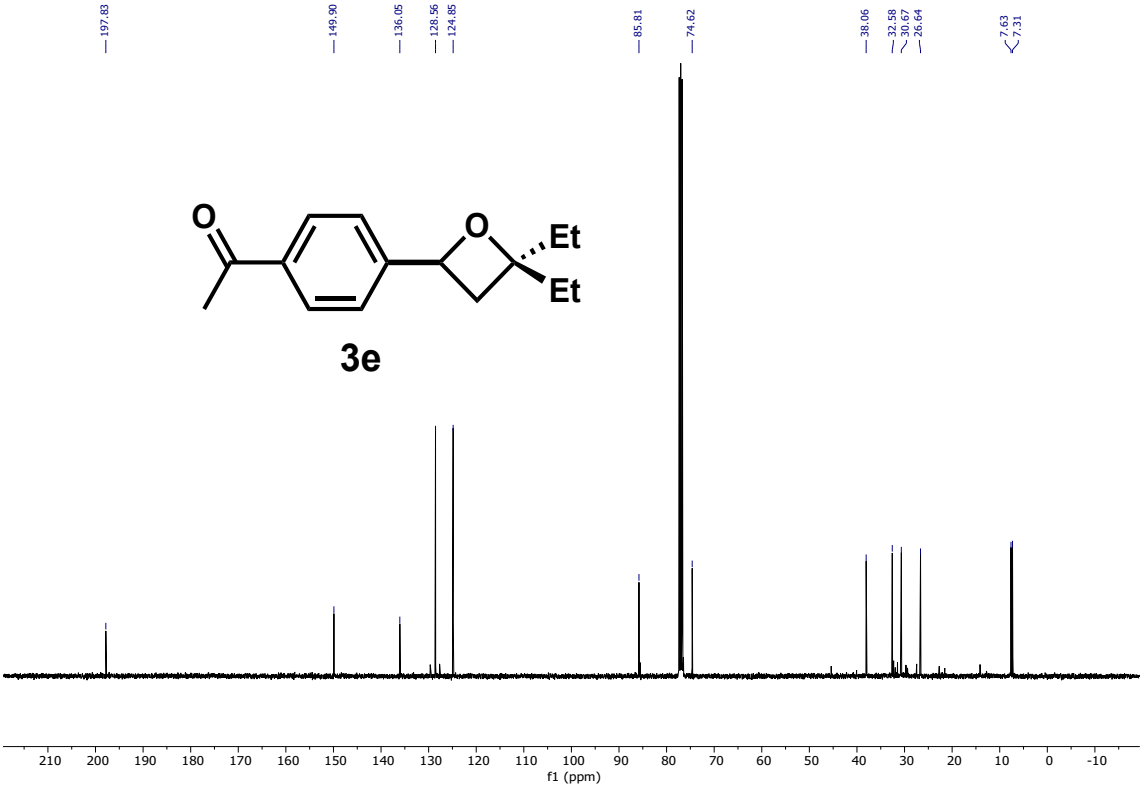

O
O

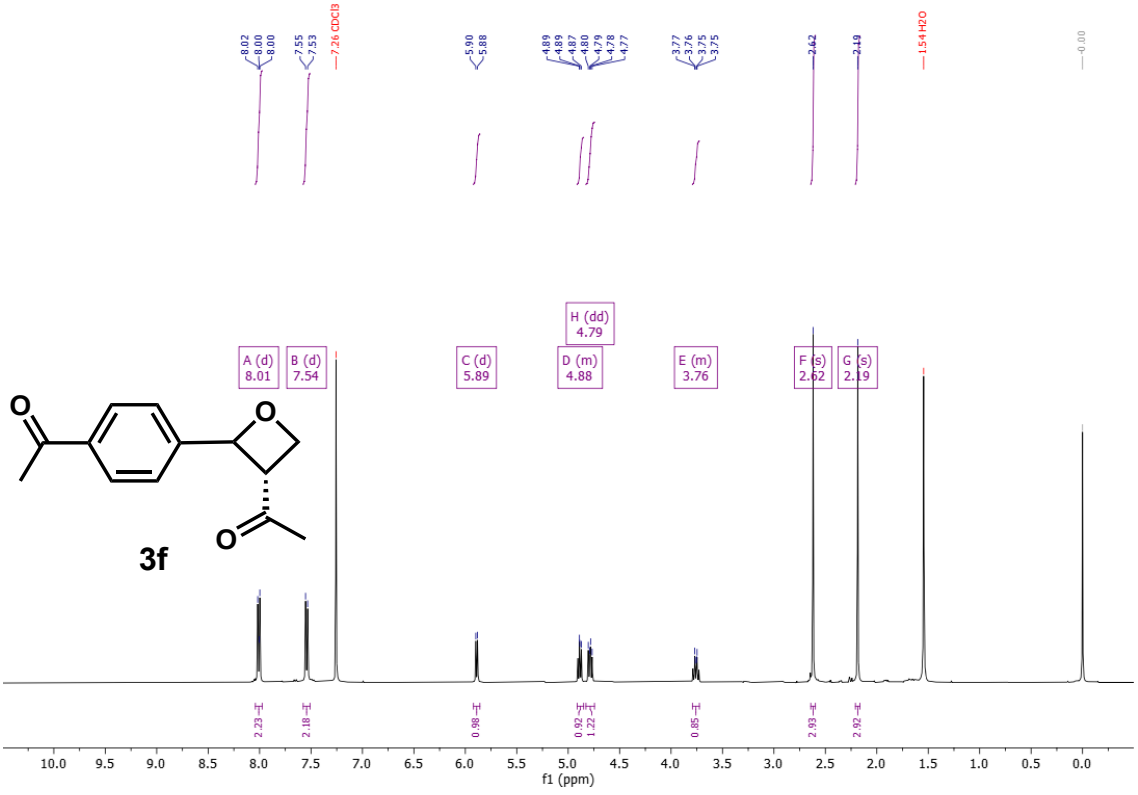
3d

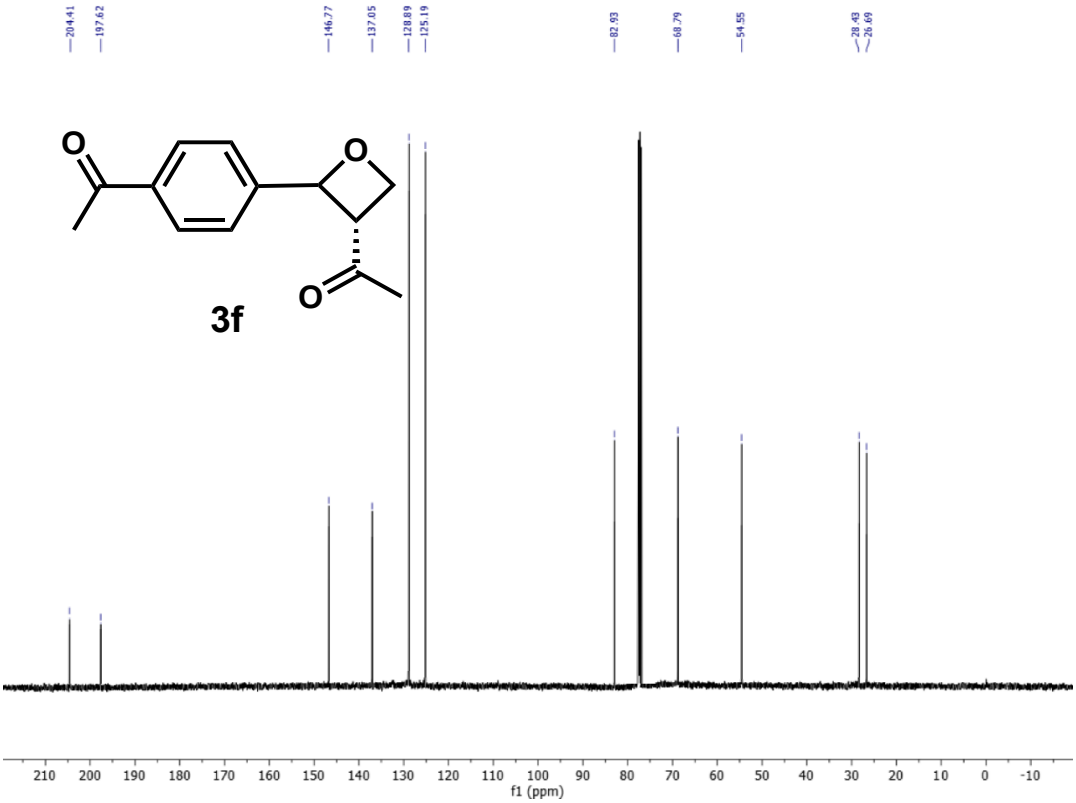

7.98
7.97
7.96
7.96
7.95
7.39
7.38
7.38
7.38
7.37
7.36
7.36
7.36
7.26 CDCl3
5.54
4.55
4.54
4.27
4.26
2.61
1.43
0.78
0.77
A (m) 7.97
B (m) 7.37
C (s) 5.54
D (d) 4.55
E (d) 4.27
F (s) 2.61
G (s) 1.43
H (s) 0.78
2.02
1.99
1.00
1.04
1.05
3.08
3.19
3.16
f1 (ppm)

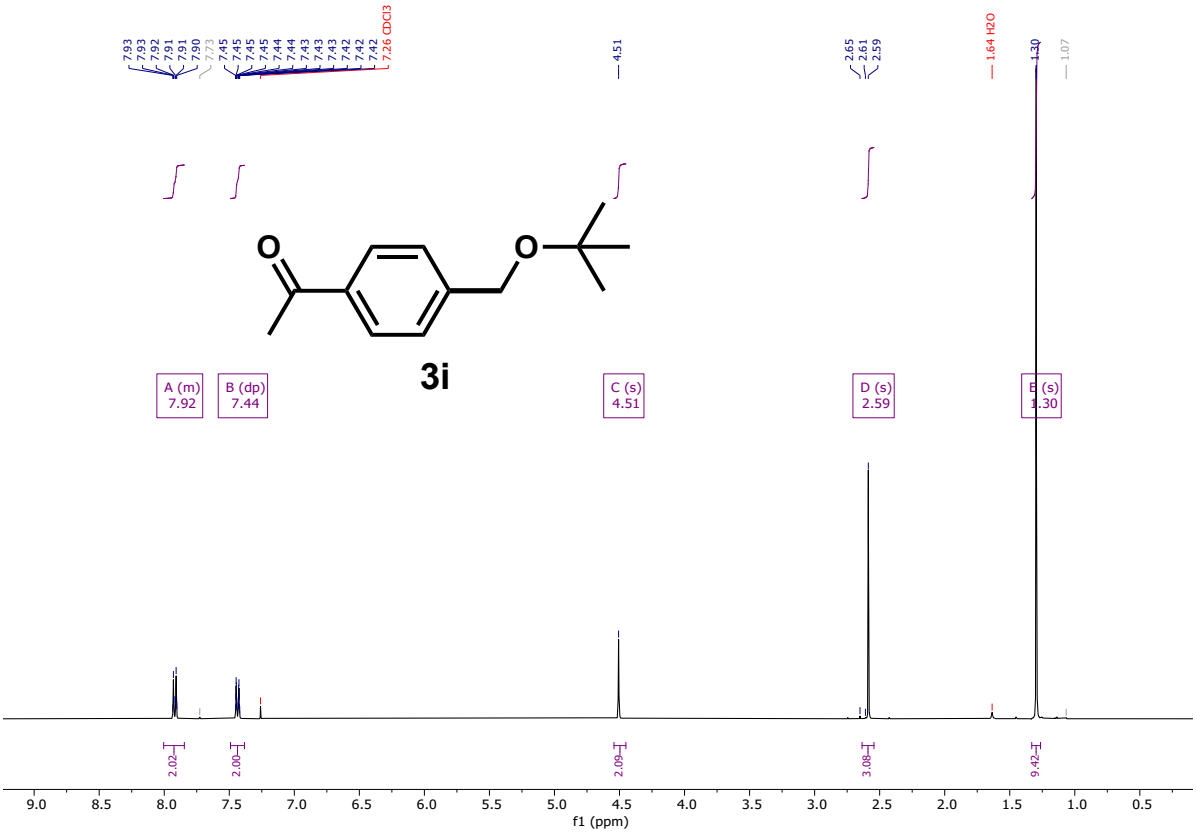

198.00
145.85
136.38
128.45
125.28
91.31
81.51
40.90
27.05
26.76
22.62
O
O
3d
f1 (ppm)

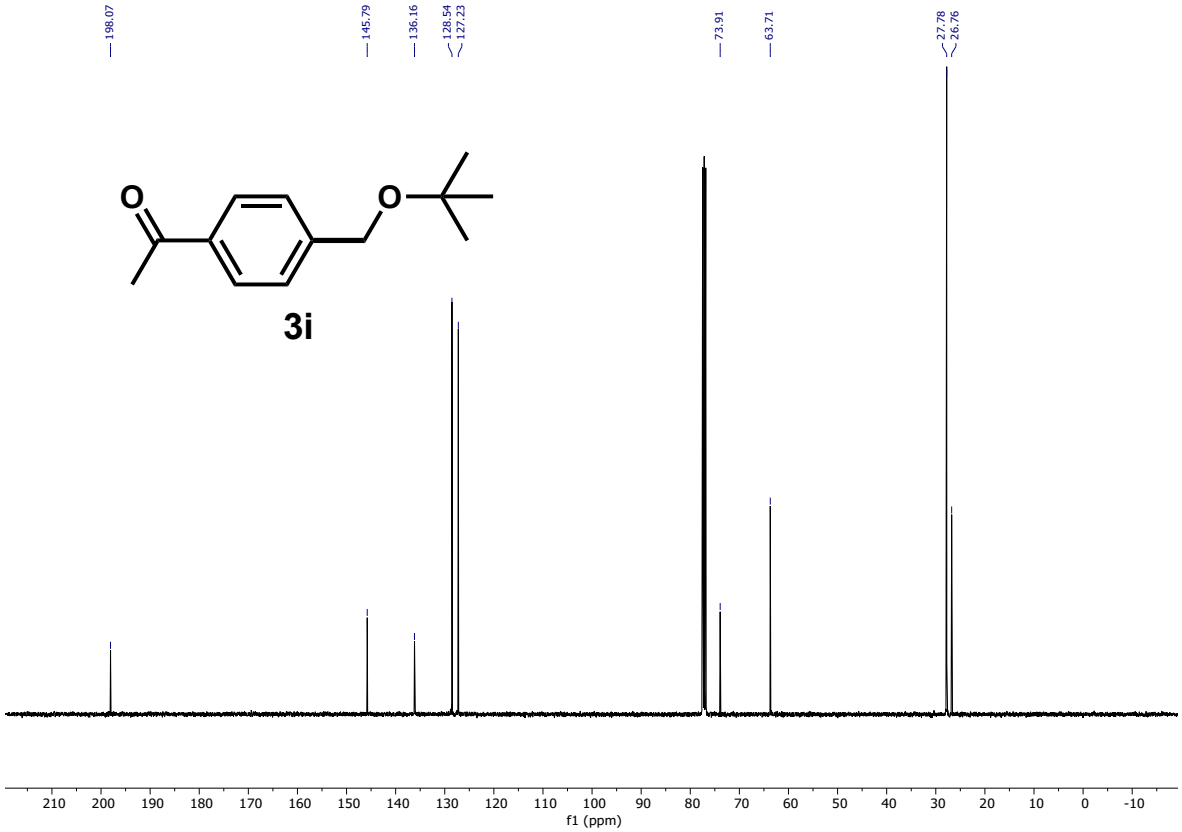

O
O
Et
Et
3e
A (m) 7.96
B (m) 7.47
C (m) 5.63
D (dd) 2.76
E (s) 2.60
F (m) 2.27
G (m) 1.77
I (t) 1.03
K (dt) 0.83
1.86
1.83
0.95
1.03
2.67
1.20
4.99
2.97
3.84
f1 (ppm)

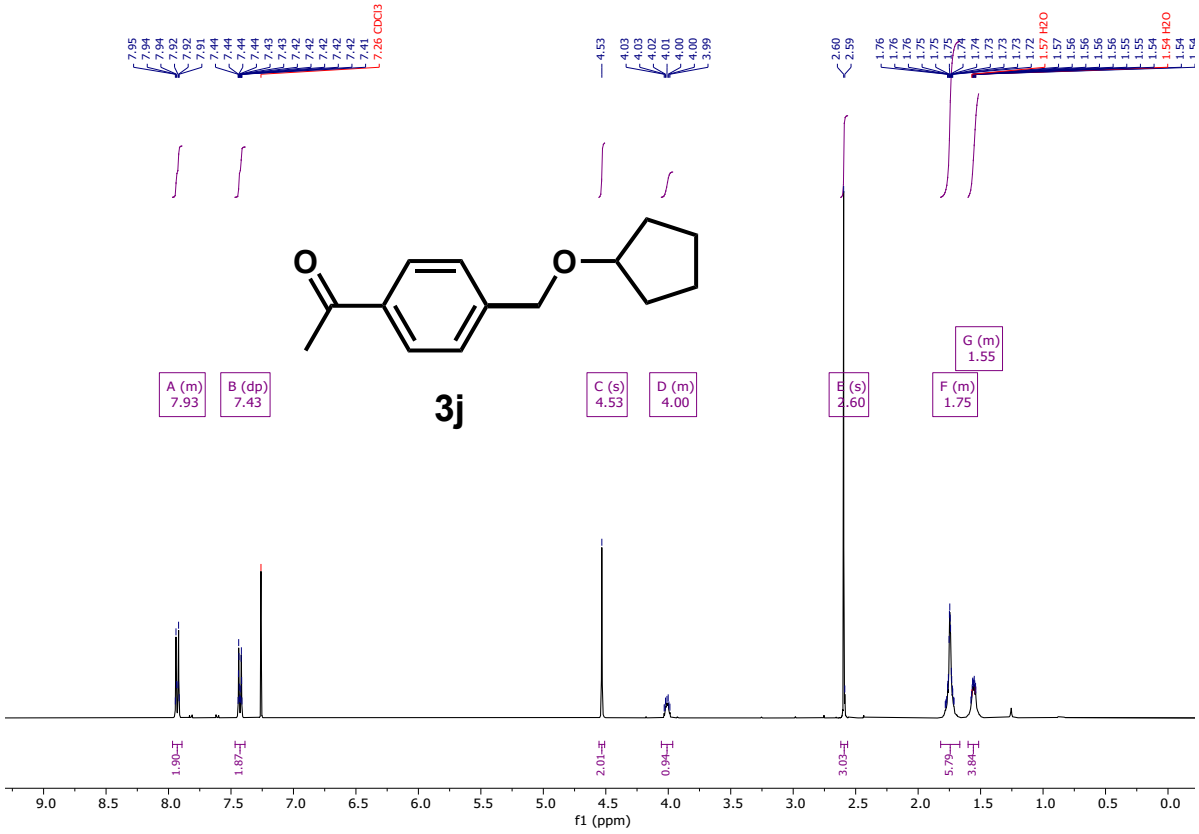

197.83
149.90
136.05
128.56
124.85
85.81
74.62
38.06
32.58
30.67
26.64
7.63
7.31
O
O
Et
Et
3e
f1 (ppm)

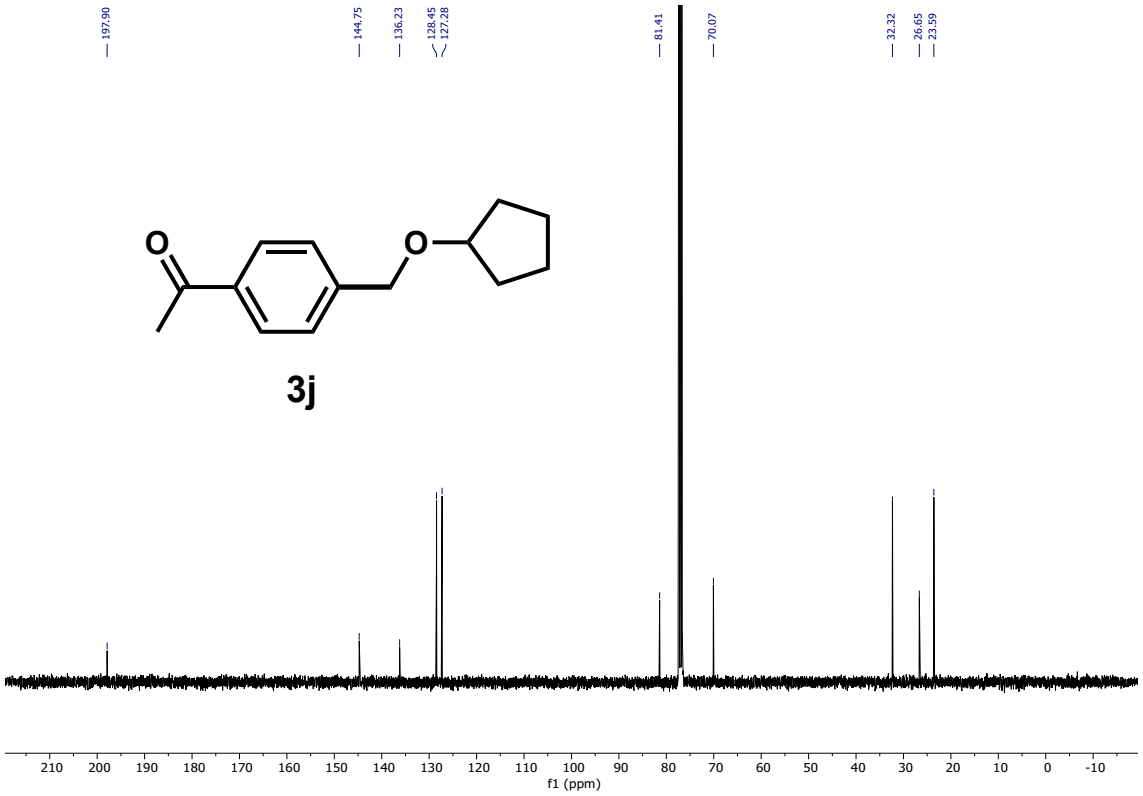

8.02
8.00
8.00
7.55
7.53
7.26 CDCl3
5.90
5.88
4.89
4.89
4.87
4.80
4.79
4.78
4.77
3.77
3.76
3.75
3.75
2.62
2.19
1.54 H2O
0.00
A (d) 8.01
B (d) 7.54
C (d) 5.89
H (dd) 4.79
D (m) 4.88
E (m) 3.76
F (s) 2.62
G (s) 2.19
O
O
O
3f
2.23
2.18
0.98
0.92
1.22
0.85
2.93
2.92
f1 (ppm)

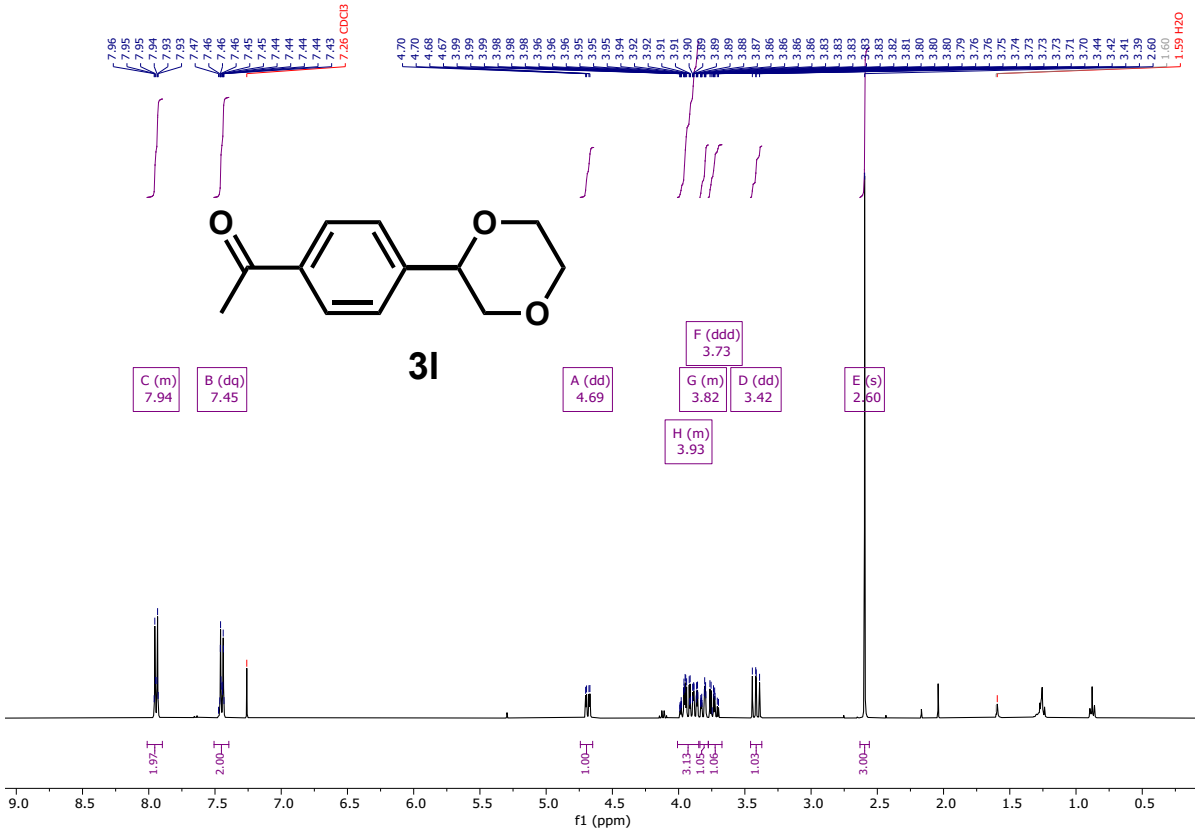

204.41
197.62
146.77
137.05
128.89
125.19
82.93
68.79
54.55
28.43
26.69
3f
f1 (ppm)

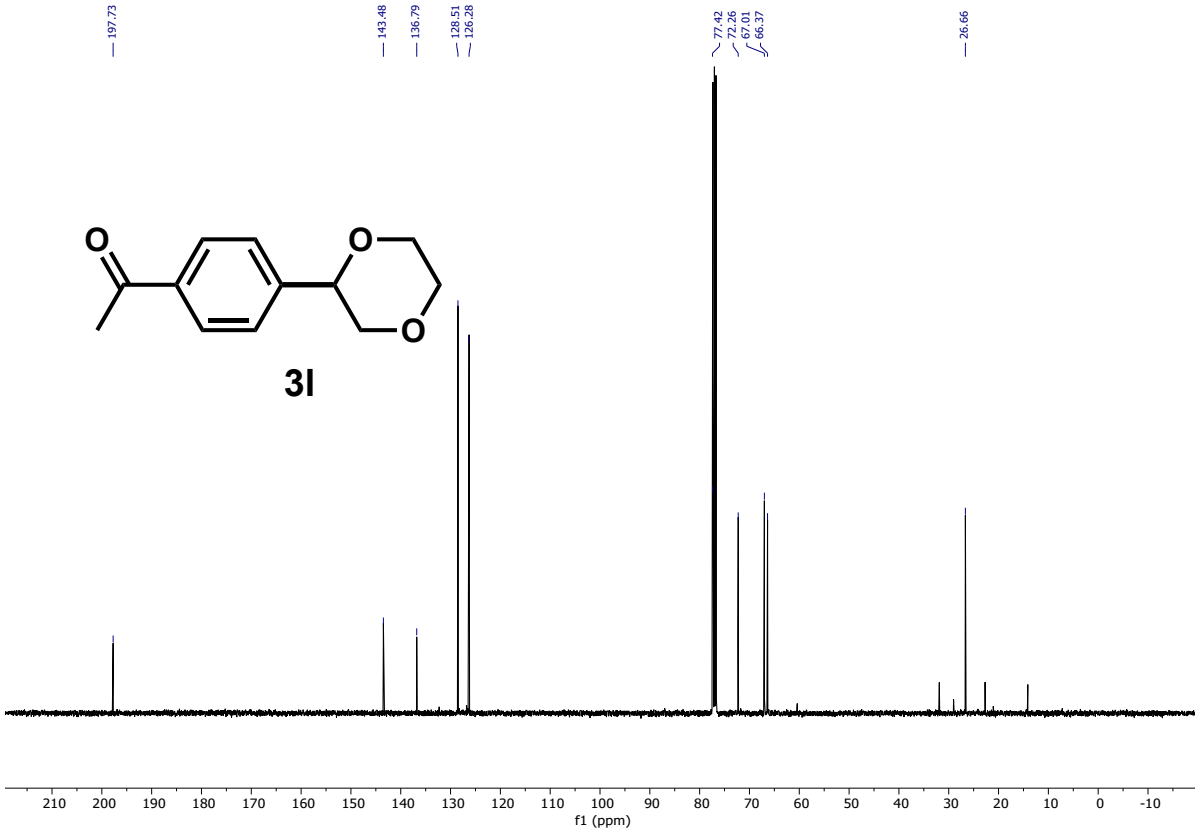

7.93
7.92
7.91
7.90
7.45
7.44
7.43
7.42
7.26 CDCl3
4.51
2.65
2.61
2.59
1.64 H2O
1.30
1.07
3i
A (m) 7.92
B (dp) 7.44
C (s) 4.51
D (s) 2.59
E (s) 1.30
2.02
2.00
2.09
3.08
9.42
f1 (ppm)

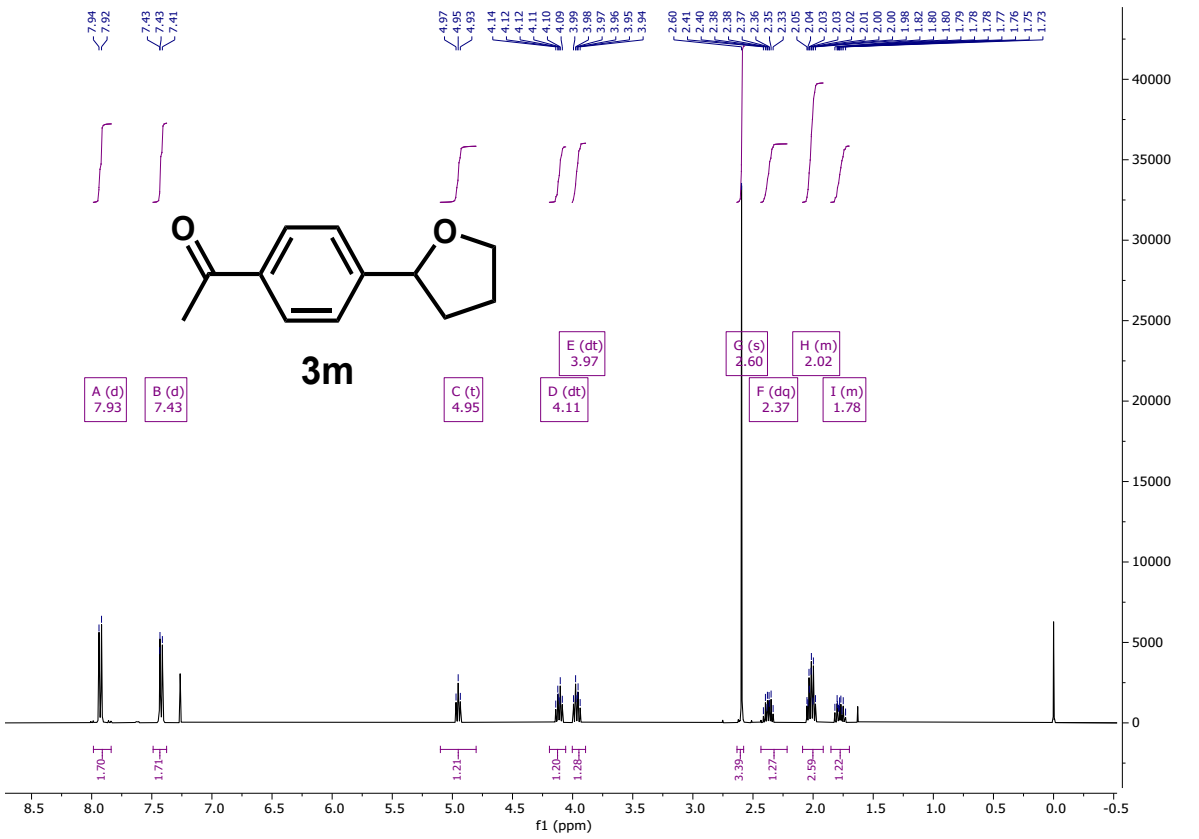

198.07
145.79
136.16
128.54
127.23
73.91
63.71
27.78
26.76
O
O
3i
f1 (ppm)


7.95
7.94
7.94
7.92
7.92
7.91
7.44
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.42
7.42
7.42
7.41
7.26 CDCl3
4.53
4.03
4.03
4.02
4.01
4.00
4.00
3.99
2.60
2.59
1.76
1.57 H2O
1.54 H2O
O
O
3j
A (m) 7.93
B (dp) 7.43
C (s) 4.53
D (m) 4.00
E (s) 2.60
F (m) 1.75
G (m) 1.55
1.90
1.87
2.01
0.94
3.03
5.79
3.84
f1 (ppm)


197.90
144.75
136.23
128.45
127.28
81.41
70.07
32.32
26.65
23.59
O
O
3j
f1 (ppm)


7.26 CDCl3
1.59 H2O
O
O
O
3l
C (m) 7.94
B (dq) 7.45
A (dd) 4.69
F (ddd) 3.73
G (m) 3.82
D (dd) 3.42
H (m) 3.93
E (s) 2.60
1.97
2.00
1.00
3.13
1.05
1.06
1.03
3.00
f1 (ppm)


197.73
143.48
136.79
128.51
126.28
77.42
72.26
67.01
66.37
26.66
3l
f1 (ppm)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7.41
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4.14
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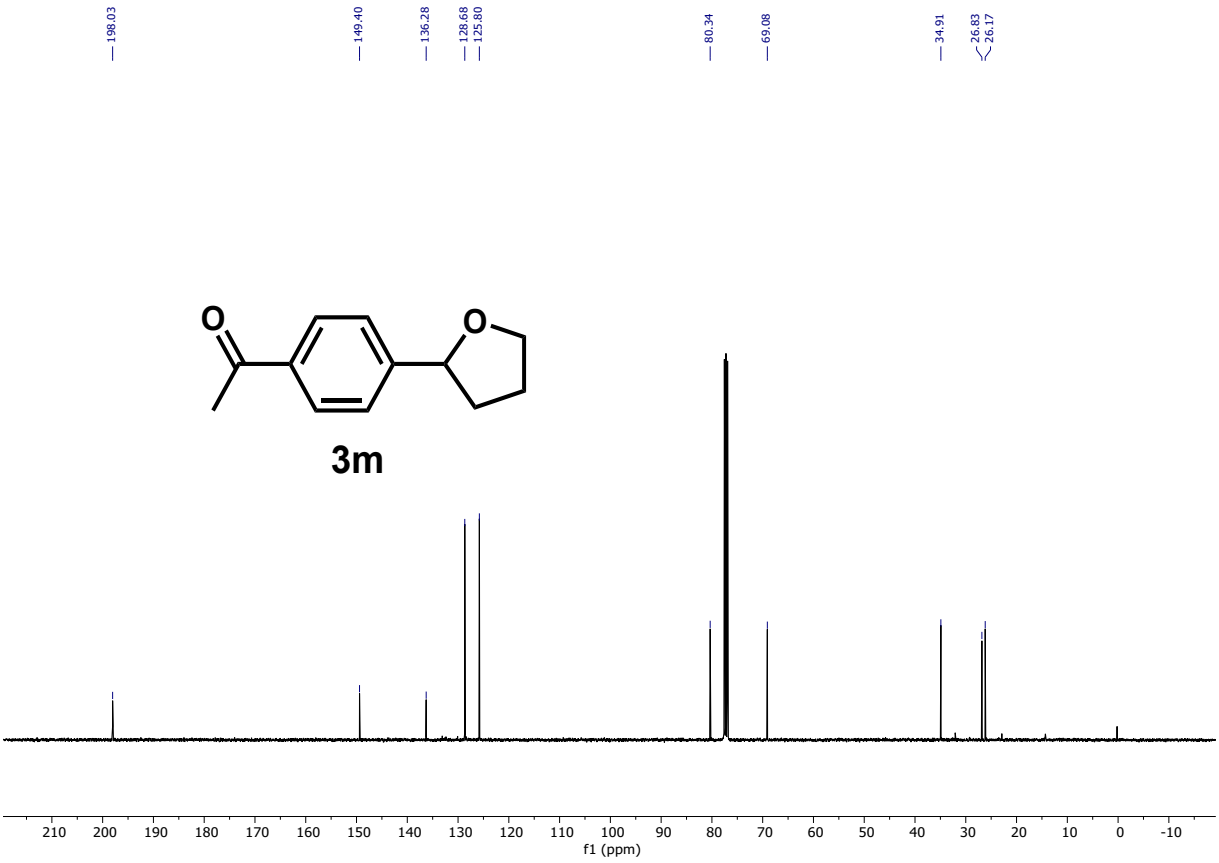
4.11
4.10
4.09
3.99
3.98
3.97
3.96
3.95
3.94
2.60
2.41
2.40
2.38
2.38
2.37
2.36
2.35
2.33
2.05
2.04
2.03
2.03
2.02
2.01
2.00
2.00
1.98
1.92
1.80
1.80
1.79
1.78
1.78
1.77
1.76
1.75
1.73
3m
A (d) 7.93
B (d) 7.43
C (t) 4.95
D (dt) 4.11
E (dt) 3.97
F (dq) 2.37
G (s) 2.60
H (m) 2.02
I (m) 1.78
1.70
1.71
1.21
1.20
1.28
3.39
1.27
2.59
1.22
f1 (ppm)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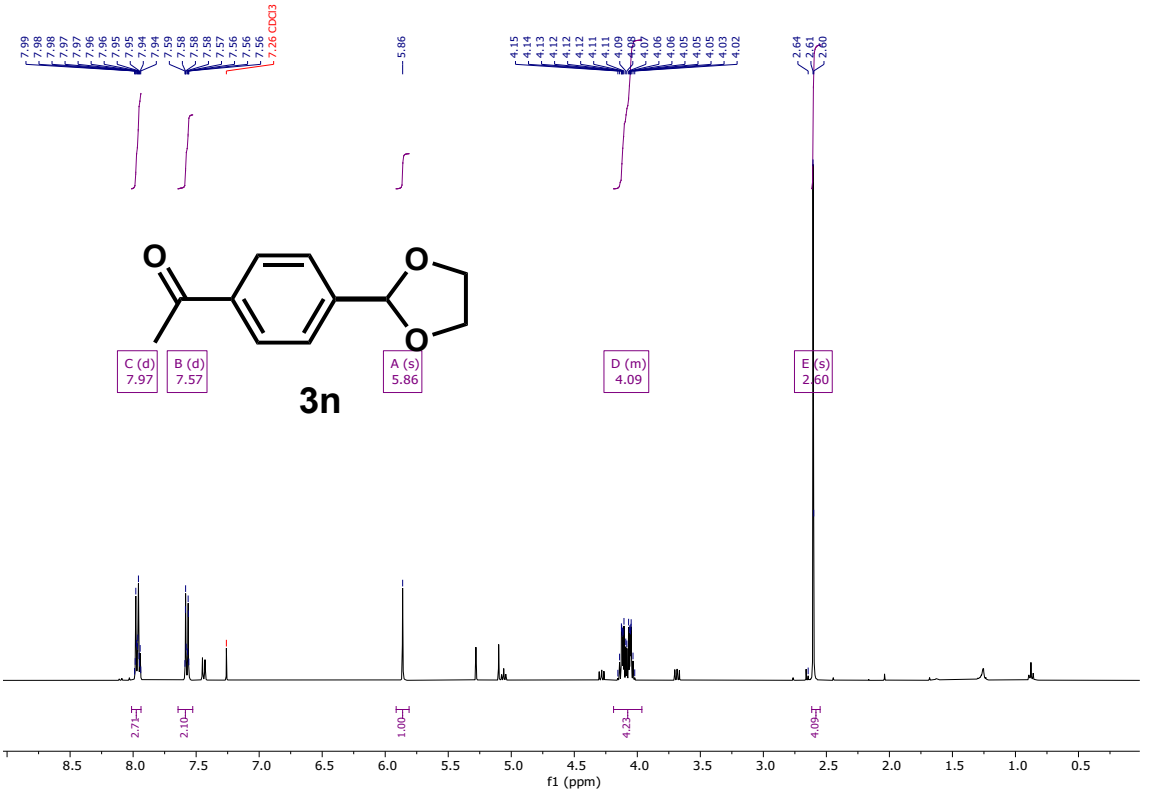
34.91
26.83
26.17
O
O
3m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.97
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7.95
7.95
7.94
7.94
7.59
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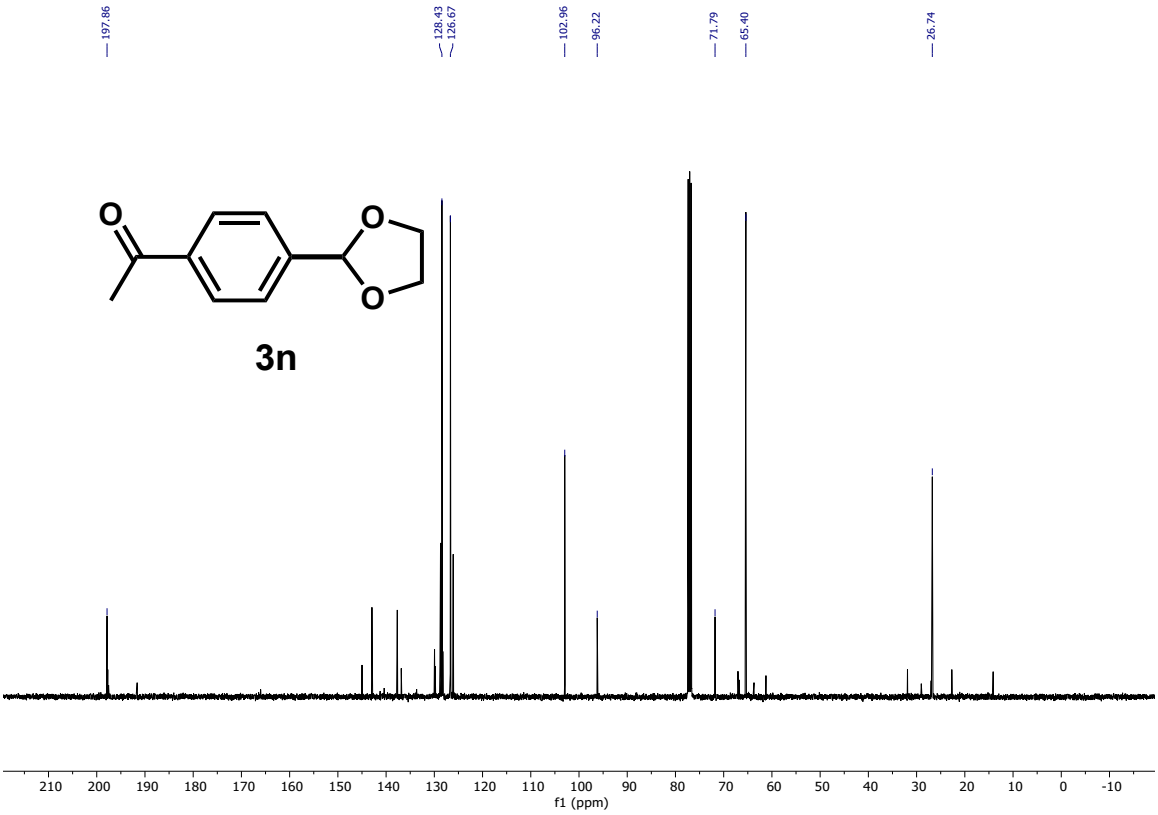
7.58
7.58
7.57
7.56
7.56
7.56
7.26 CDCl3
5.86
4.15
4.14
4.13
4.12
4.12
4.12
4.11
4.11
4.09
4.08
4.07
4.06
4.06
4.06
4.05
4.05
4.05
4.03
4.02
2.64
2.61
2.60
O
O
O
C (d) 7.97
B (d) 7.57
A (s) 5.86
D (m) 4.09
E (s) 2.60
3n
2.71
2.10
1.00
4.23
4.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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128.43
126.67
102.96
96.22
71.79
65.40
26.74
O
O
O
3n
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200
190
180
170
160
150
140
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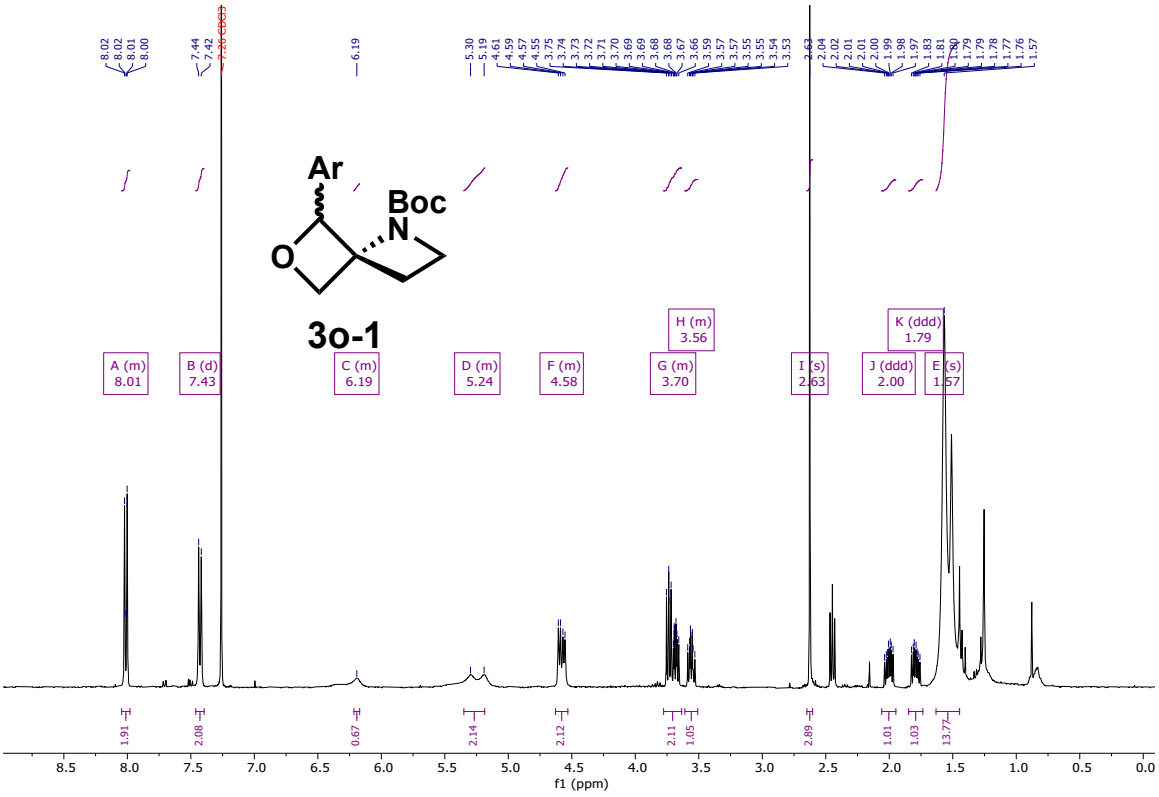
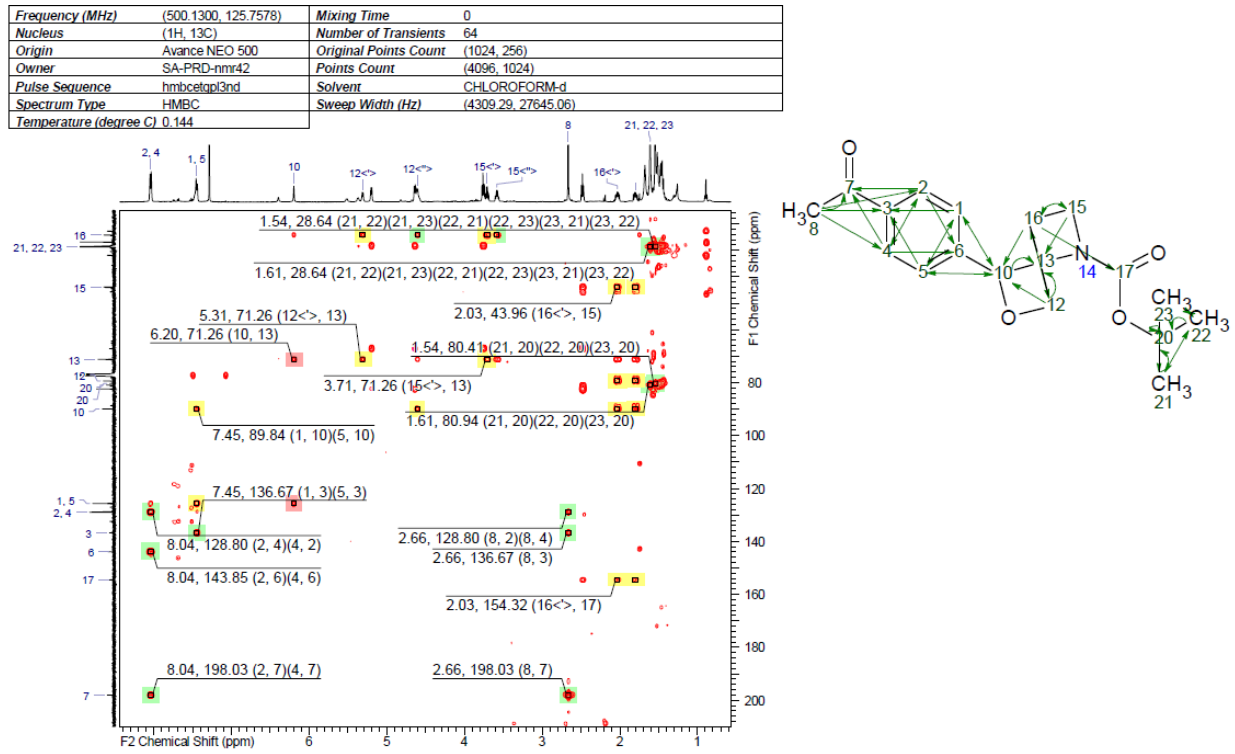
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

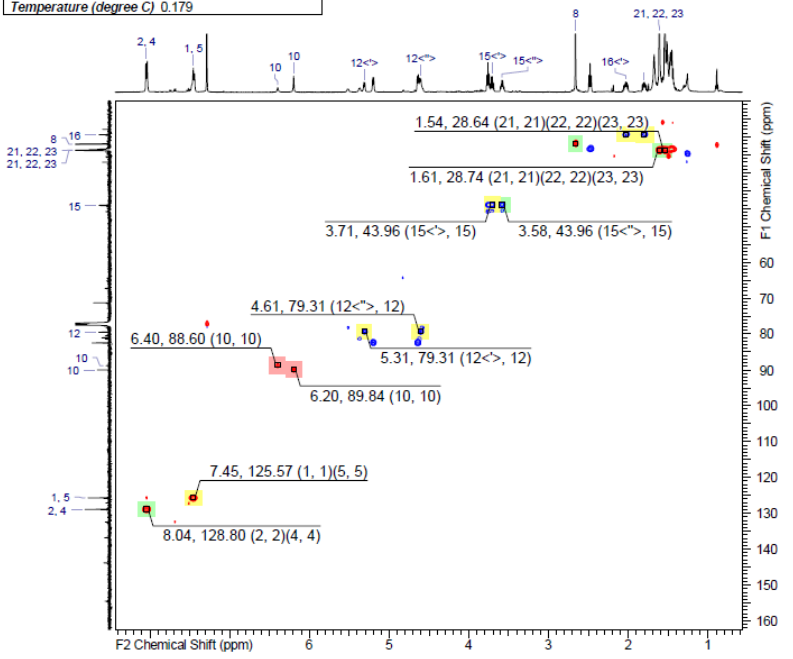


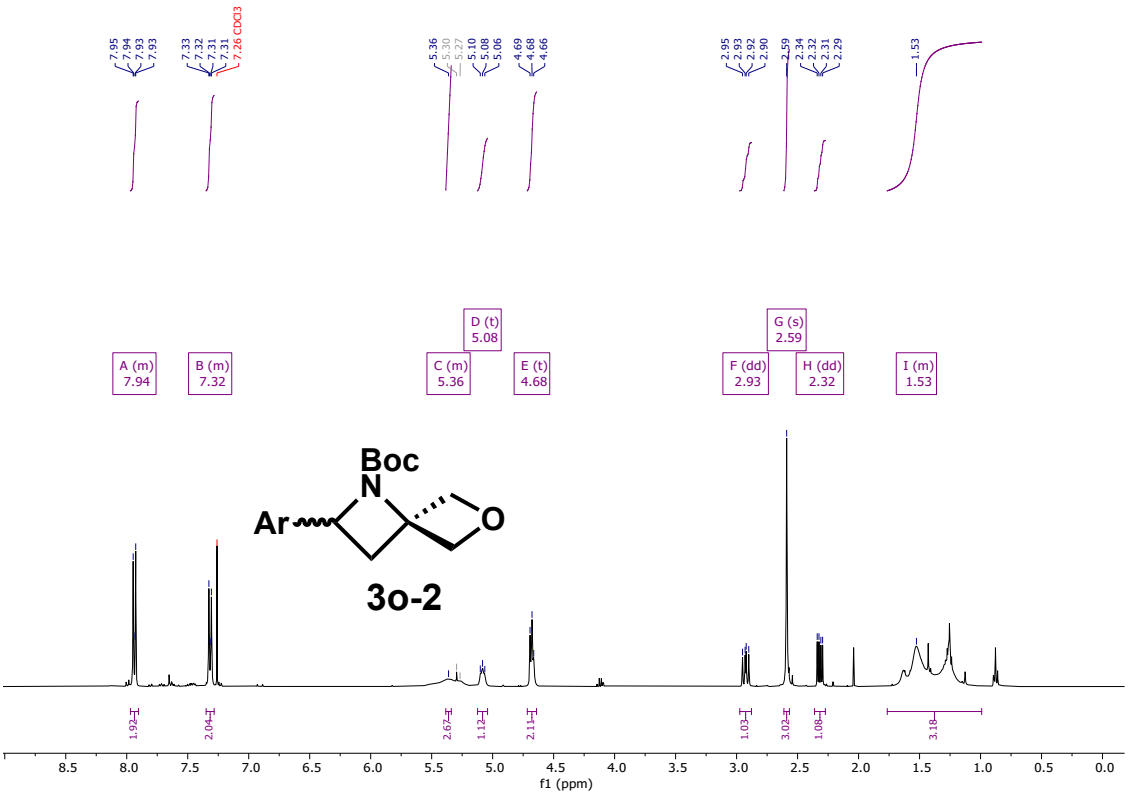
HMBC for **3o-1** ($CDCl_3$, 0 °C)



HSQC-DEPT for **3o-1** ($CDCl_3$, 0 °C)

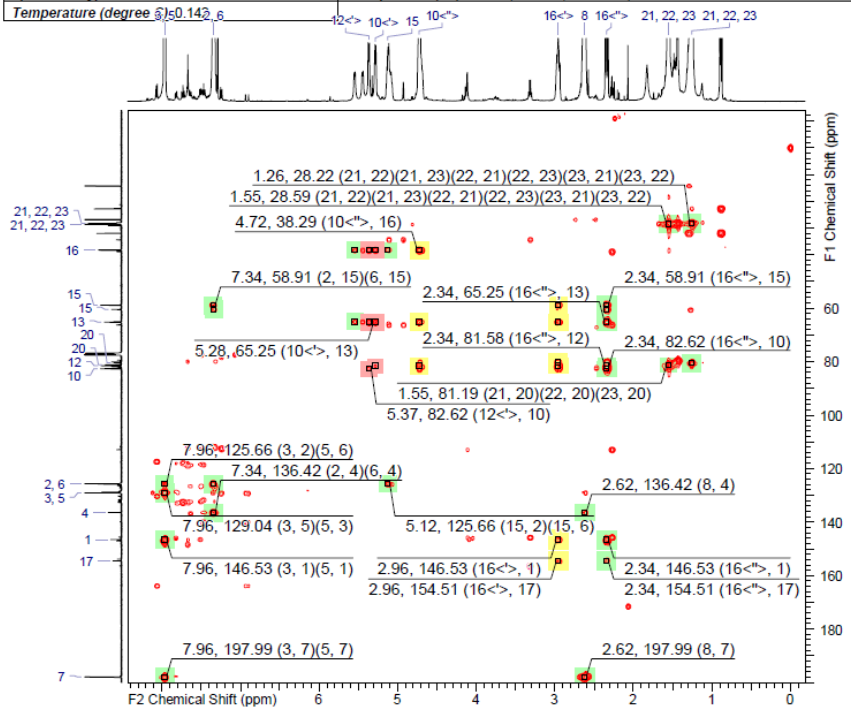
Frequency (MHz)	(500.1300, 125.7578)	*Mixing Time*	0
Nucleus	(1H, 13C)	*Number of Transients*	16
Origin	Avance NEO 500	*Original Points Count*	(1024, 256)
Owner	SA-PRD-nmr42	*Points Count*	(1024, 1024)
Pulse Sequence	hsqcedetgpsisp2.3	*Solvent*	CHLOROFORM-d
Spectrum Type	HSQC-DEPT	*Sweep Width (Hz)*	(4306.14, 20732.00)
Temperature (degree C)	0.179		

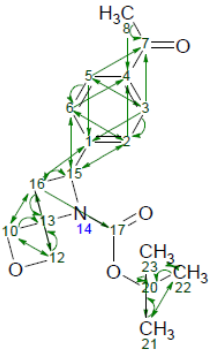




HMBC of **3o-2** ($CDCl_3$, 0 °C)

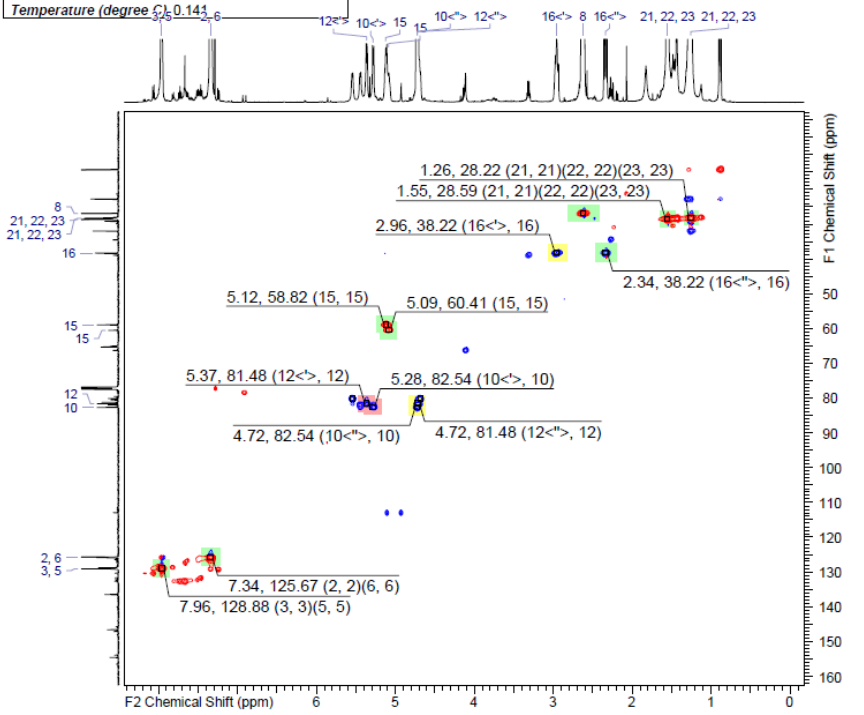
Frequency (MHz)	(500.1300, 125.7578)	*Mixing Time*	0
Nucleus	(1H, 13C)	*Number of Transients*	40
Origin	Avance NEO 500	*Original Points Count*	(1024, 256)
Owner	SA-PRD-nmr42	*Points Count*	(4096, 1024)
Pulse Sequence	hmbcetgpl3nd	*Solvent*	CHLOROFORM-d
Spectrum Type	HMBC	*Sweep Width (Hz)*	(4309.29, 27645.06)
Temperature (degree C)	0.14		

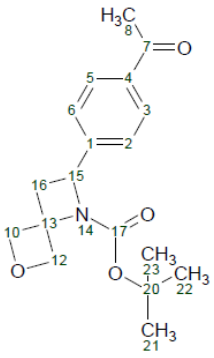




HSQC-DEPT of **3o-2** ($CDCl_3$, 0 °C)

Frequency (MHz)	(500.1300, 125.7578)	*Mixing Time*	0
Nucleus	(1H, 13C)	*Number of Transients*	8
Origin	Avance NEO 500	*Original Points Count*	(1024, 256)
Owner	SA-PRD-nmr42	*Points Count*	(1024, 1024)
Pulse Sequence	hsqcedetgpsisp2.3	*Solvent*	CHLOROFORM-d
Spectrum Type	HSQC-DEPT	*Sweep Width (Hz)*	(4306.14, 20732.00)
Temperature (degree C)	0.141		





VIII. HTE Data

Table S2. Full HTE data set. Reactions set up as described in General Procedure A.

Photocatalyst (PC)	PC %	Ni Catalyst	Ni %	Base (2 equiv)	Oxetane Equivs	Solvent (0.1 M)	Additive	Additive %	Yield (%)
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			4.7
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			37.0
benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			6.0
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			0.0
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			4.6
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			3.7
benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
p-anisaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			77.7
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			51.8
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			54.4
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			63.6
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			30.9
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			4.9
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			7.1
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			87.9
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			46.7
benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			7.0

benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			7.8
benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			21.1
benzophenone	100	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			20.3
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			36.9
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			35.3
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			2.8
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			13.1
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			35.0
benzophenone	100	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			36.8
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			2.8
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			0.0
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
none	0	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			0.0
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			3.5
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			0.0
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			0.0
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
none	0	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
Eosin Y	100	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			0.0
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			42.7
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			3.1
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			16.0
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			2.2
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			16.9
TBADT	10	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			54.3
TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			4.4
TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	DCM			0.0

TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			0.0
TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
TBADT	10	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	DMSO			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	DCM			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	DMF			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	acetone			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			13.9
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	DMSO			75.3
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(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	DMF			8.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	acetone			6.4
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			70.0
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			45.7
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			51.4
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			39.8
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			48.5
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			54.4
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			6.7
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			36.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			54.8
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			35.5
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			42.0
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			34.1
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			7.7
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			74.9
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			54.6
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	TFT			45.6
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			58.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			65.6
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			3.5

m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			26.2
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			67.5
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			25.6
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			50.2
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			13.5
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	TFT			16.2
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	TFT			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			40.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			42.2
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			32.4
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			44.8
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			45.0
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			44.2
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	MeCN			13.1
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			13.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			16.2
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0

m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			2.4
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			4.4
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			1.9
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			5.2
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Cs2CO3	10	MeCN			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			9.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			1.7
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			5.8
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			3.6
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Quinuclidine	10	MeCN			0.0
(Ir[dF(CF3)ppy]2(dtbpy))PF6	10	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
p-CF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
p-NO2-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
m-CHO-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
3,5-diCF3-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
3-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
4-py-carboxaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
pentafluorobenzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			2.9
p-Ac-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
2-naphthaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
o-Me-benzaldehyde	100	(dtbbpy)NiBr2	10	Barton's base	10	MeCN			0.0
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4	5	TFT			9.7
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4	5	TFT			35.4

p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	5	TFT			40.8
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4	5	TFT			21.5
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4	5	TFT			59.7
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K3PO4	5	TFT			24.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K3PO4	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K3PO4	5	TFT			45.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	5	TFT			55.1
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K3PO4	5	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	5	TFT			37.6
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			21.4
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4	10	TFT			42.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4	10	TFT			54.2
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4	10	TFT			19.9
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4	10	TFT			83.3
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K3PO4	10	TFT			41.2
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K3PO4	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT			42.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT			67.6
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K3PO4	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT			93.4
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4	5	TFT			24.8
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4	5	TFT			35.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4	5	TFT			18.7
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4	5	TFT			6.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4	5	TFT			64.2
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K3PO4	5	TFT			42.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K3PO4	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K3PO4	5	TFT			40.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K3PO4	5	TFT			34.4

p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K3PO4	5	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K3PO4	5	TFT			68.8
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4	10	TFT			49.7
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4	10	TFT			76.3
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K3PO4	10	TFT			47.5
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K3PO4	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K3PO4	10	TFT			52.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K3PO4	10	TFT			41.9
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K3PO4	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K3PO4	10	TFT			87.0
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4 (slurry)	5	TFT			16.8
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4 (slurry)	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4 (slurry)	5	TFT			6.1
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4 (slurry)	5	TFT			46.4
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4 (slurry)	5	TFT			11.9
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4 (slurry)	5	TFT			51.1
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4 (lab ram)	5	TFT			16.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4 (lab ram)	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4 (lab ram)	5	TFT			51.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4 (lab ram)	5	TFT			0.0
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4 (lab ram)	5	TFT			27.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4 (lab ram)	5	TFT			49.4
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4 (slurry)	10	TFT			20.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4 (slurry)	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4 (slurry)	10	TFT			0.0

p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4 (slurry)	10	TFT			52.9
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4 (slurry)	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4 (slurry)	10	TFT			95.3
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	10	K2HPO4 (lab ram)	10	TFT			31.6
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	10	K2HPO4 (lab ram)	10	TFT			5.3
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	10	K2HPO4 (lab ram)	10	TFT			61.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K2HPO4 (lab ram)	10	TFT			55.3
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	10	K2HPO4 (lab ram)	10	TFT			25.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K2HPO4 (lab ram)	10	TFT			77.7
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4 (slurry)	5	TFT			20.7
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4 (slurry)	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4 (slurry)	5	TFT			27.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4 (slurry)	5	TFT			0.0
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4 (slurry)	5	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4 (slurry)	5	TFT			52.4
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4 (lab ram)	5	TFT			23.8
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4 (lab ram)	5	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4 (lab ram)	5	TFT			63.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4 (lab ram)	5	TFT			22.8
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4 (lab ram)	5	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4 (lab ram)	5	TFT			57.9
p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4 (slurry)	10	TFT			0.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4 (slurry)	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4 (slurry)	10	TFT			40.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4 (slurry)	10	TFT			54.6
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4 (slurry)	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4 (slurry)	10	TFT			62.3

p-CN-benzaldehyde	50	(dtbbpy)NiBr2	20	K2HPO4 (lab ram)	10	TFT			40.0
p-CN-benzaldehyde	100	Ni(acac)2 + ttbterpy	20	K2HPO4 (lab ram)	10	TFT			0.0
p-CN-benzaldehyde	50	NiBr2(DME) + ttbterpy	20	K2HPO4 (lab ram)	10	TFT			42.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	K2HPO4 (lab ram)	10	TFT			44.8
p-CN-benzaldehyde	50	Ni(acac)2 + ttbterpy	20	K2HPO4 (lab ram)	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	20	K2HPO4 (lab ram)	10	TFT			70.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DABCO	10	TFT			73.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	Quinuclidine	10	TFT			21.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NMI	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	TBD	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NaBHT	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	CsF	10	TFT			75.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	KOPiv	10	TFT			5.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DIPEA	10	TFT			37.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	P2Et	10	TFT			3.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DBU	10	TFT			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	2,6-lutidine	10	TFT			24.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	iPr2NH	10	TFT			71.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DABCO	10	MeCN			5.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	Quinuclidine	10	MeCN			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NMI	10	MeCN			14.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	TBD	10	MeCN			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NaBHT	10	MeCN			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	CsF	10	MeCN			26.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	KOPiv	10	MeCN			1.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DIPEA	10	MeCN			4.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	P2Et	10	MeCN			3.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DBU	10	MeCN			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	2,6-lutidine	10	MeCN			17.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	iPr2NH	10	MeCN			28.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DABCO	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	Quinuclidine	10	acetone			20.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NMI	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	TBD	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NaBHT	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	CsF	10	acetone			42.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	KOPiv	10	acetone			3.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DIPEA	10	acetone			22.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	P2Et	10	acetone			0.0

p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DBU	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	2,6-lutidine	10	acetone			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	iPr2NH	10	acetone			61.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DABCO	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	Quinuclidine	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NMI	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	TBD	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	NaBHT	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	CsF	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	KOPiv	10	t-amyl OH			3.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DIPEA	10	t-amyl OH			6.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	P2Et	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	DBU	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	2,6-lutidine	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	20	iPr2NH	10	t-amyl OH			34.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DABCO	10	TFT			6.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	Quinuclidine	10	TFT			63.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NMI	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	TBD	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NaBHT	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	CsF	10	TFT			88.9
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	KOPiv	10	TFT			51.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DIPEA	10	TFT			50.7
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	P2Et	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DBU	10	TFT			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	2,6-lutidine	10	TFT			16.9
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	iPr2NH	10	TFT			64.2
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DABCO	10	MeCN			4.8
p-CN-benzaldehyde	100	NiBr2(DME) +	10	Quinuclidine	10	MeCN			5.4

		ttbterpy							
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NMI	10	MeCN			11.6
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	TBD	10	MeCN			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NaBHT	10	MeCN			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	CsF	10	MeCN			17.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	KOPiv	10	MeCN			15.6
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DIPEA	10	MeCN			30.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	P2Et	10	MeCN			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DBU	10	MeCN			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	2,6-lutidine	10	MeCN			4.7
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	iPr2NH	10	MeCN			14.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DABCO	10	acetone			25.6
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	Quinuclidine	10	acetone			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NMI	10	acetone			12.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	TBD	10	acetone			1.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NaBHT	10	acetone			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	CsF	10	acetone			12.5
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	KOPiv	10	acetone			21.2
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DIPEA	10	acetone			65.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	P2Et	10	acetone			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DBU	10	acetone			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	2,6-lutidine	10	acetone			5.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	iPr2NH	10	acetone			16.2
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DABCO	10	t-amyl OH			35.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	Quinuclidine	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	NMI	10	t-amyl OH			14.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	TBD	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	NiBr2(DME) +	10	NaBHT	10	t-amyl			0.0

		ttbterpy				OH			
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	CsF	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	KOPiv	10	t-amyl OH			4.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DIPEA	10	t-amyl OH			21.7
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	P2Et	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	DBU	10	t-amyl OH			0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	2,6-lutidine	10	t-amyl OH			11.7
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	iPr2NH	10	t-amyl OH			24.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCE	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	acetone	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	t-amyl OH	50	60.5
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFE	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMSO	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCM	50	29.6
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMF	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	MeCN	50	88.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFT	50	100.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMA	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	2,6-lutidine	50	3.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	water	50	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCE	25	51.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	acetone	25	100.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	t-amyl OH	25	77.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFE	25	22.2
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMSO	25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCM	25	24.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMF	25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) +	10	K3PO4	10	TFT	MeCN	25	76.9

		ttbterpy							
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFT	25	85.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMA	25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	2,6-lutidine	25	3.6
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	water	25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCE	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	acetone	12.5	100.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	t-amyl OH	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFE	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMSO	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCM	12.5	40.1
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMF	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	MeCN	12.5	1.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFT	12.5	100.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMA	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	2,6-lutidine	12.5	11.7
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	water	12.5	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCE	6.25	5.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	acetone	6.25	98.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	t-amyl OH	6.25	83.8
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFE	6.25	1.5
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMSO	6.25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DCM	6.25	52.4
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMF	6.25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	MeCN	6.25	53.3
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	TFT	6.25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	DMA	6.25	0.0
p-CN-benzaldehyde	100	NiBr2(DME) +	10	K3PO4	10	TFT	2,6-	6.25	0.0

		ttbterpy					lutidine		
p-CN-benzaldehyde	100	NiBr2(DME) + ttbterpy	10	K3PO4	10	TFT	water	6.25	1.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCE	50	68.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	acetone	50	2.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	t-amyl OH	50	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFE	50	7.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMSO	50	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCM	50	66.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMF	50	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	MeCN	50	42.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFT	50	75.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMA	50	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	2,6-lutidine	50	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	water	50	2.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCE	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	acetone	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	t-amyl OH	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFE	25	5.7
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMSO	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCM	25	57.5
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMF	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	MeCN	25	46.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFT	25	61.1
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMA	25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	2,6-lutidine	25	15.9
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	water	25	2.2
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCE	12.5	25.6
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	acetone	12.5	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	t-amyl OH	12.5	39.1
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFE	12.5	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMSO	12.5	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCM	12.5	63.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMF	12.5	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	MeCN	12.5	39.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFT	12.5	59.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMA	12.5	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	2,6-lutidine	12.5	31.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	water	12.5	3.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCE	6.25	72.2

p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	acetone	6.25	7.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	t-amyl OH	6.25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFE	6.25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMSO	6.25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DCM	6.25	74.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMF	6.25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	MeCN	6.25	8.8
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	TFT	6.25	9.4
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	DMA	6.25	0.0
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	2,6-lutidine	6.25	42.3
p-CN-benzaldehyde	100	(dtbbpy)NiBr2	10	K3PO4	10	TFT	water	6.25	3.6
4-CN-benzaldehyde	50	dtbbpy + NiBr2(DME)	10	K2HPO4	10	TFT			35.2
4-CN-benzaldehyde	50	diOMe bpy + NiBr2(DME)	10	K2HPO4	10	TFT			25.4
4-CN-benzaldehyde	50	diMe bpy + NiBr2(DME)	10	K2HPO4	10	TFT			27.1
4-CN-benzaldehyde	50	diMe Phen + NiBr2(DME)	10	K2HPO4	10	TFT			22.7
4-CN-benzaldehyde	50	diOMe phen + NiBr2(DME)	10	K2HPO4	10	TFT			29.0
4-CN-benzaldehyde	50	bphen + NiBr2(DME)	10	K2HPO4	10	TFT			36.1
4-CN-benzaldehyde	50	ttbterpy + NiBr2(DME)	10	K2HPO4	10	TFT			100.0
4-CN-benzaldehyde	50	BPyCamCN + NiBr2(DME)	10	K2HPO4	10	TFT			28.0
4-CN-benzaldehyde	50	Me bpp + NiBr2(DME)	10	K2HPO4	10	TFT			9.8
4-CN-benzaldehyde	50	PyCamCN + NiBr2(DME)	10	K2HPO4	10	TFT			25.1
4-CN-benzaldehyde	50	BnBiox + NiBr2(DME)	10	K2HPO4	10	TFT			21.5
4-CN-benzaldehyde	50	IndenoBOX + NiBr2(DME)	10	K2HPO4	10	TFT			21.1
4-CN-benzaldehyde	50	dtbbpy + Ni(cod)(DQ)	10	K2HPO4	10	TFT			59.9
4-CN-benzaldehyde	50	diOMe bpy + Ni(cod)(DQ)	10	K2HPO4	10	TFT			30.1
4-CN-benzaldehyde	50	diMe bpy + Ni(cod)(DQ)	10	K2HPO4	10	TFT			24.0
4-CN-benzaldehyde	50	diMe Phen + Ni(cod)(DQ)	10	K2HPO4	10	TFT			21.2
4-CN-benzaldehyde	50	diOMe phen + Ni(cod)(DQ)	10	K2HPO4	10	TFT			31.4
4-CN-benzaldehyde	50	bphen + Ni(cod)(DQ)	10	K2HPO4	10	TFT			24.1
4-CN-benzaldehyde	50	ttbterpy + Ni(cod)(DQ)	10	K2HPO4	10	TFT			0.0
4-CN-benzaldehyde	50	BPyCamCN +	10	K2HPO4	10	TFT			10.4

		Ni(cod)(DQ)							
4-CN-benzaldehyde	50	Me bpp + Ni(cod)(DQ)	10	K2HPO4	10	TFT			22.4
4-CN-benzaldehyde	50	PyCamCN + Ni(cod)(DQ)	10	K2HPO4	10	TFT			10.1
4-CN-benzaldehyde	50	BnBiox + Ni(cod)(DQ)	10	K2HPO4	10	TFT			16.7
4-CN-benzaldehyde	50	IndenoBOX + Ni(cod)(DQ)	10	K2HPO4	10	TFT			10.3
4-CN-benzaldehyde	50	dtbbpy + Ni(acac)2	10	K2HPO4	10	TFT			40.3
4-CN-benzaldehyde	50	diOMe bpy + Ni(acac)2	10	K2HPO4	10	TFT			29.1
4-CN-benzaldehyde	50	diMe bpy + Ni(acac)2	10	K2HPO4	10	TFT			27.3
4-CN-benzaldehyde	50	diMe Phen + Ni(acac)2	10	K2HPO4	10	TFT			75.7
4-CN-benzaldehyde	50	diOMe phen + Ni(acac)2	10	K2HPO4	10	TFT			57.9
4-CN-benzaldehyde	50	bphen + Ni(acac)2	10	K2HPO4	10	TFT			77.9
4-CN-benzaldehyde	50	ttbterpy + Ni(acac)2	10	K2HPO4	10	TFT			90.0
4-CN-benzaldehyde	50	BPyCamCN + Ni(acac)2	10	K2HPO4	10	TFT			14.7
4-CN-benzaldehyde	50	Me bpp + Ni(acac)2	10	K2HPO4	10	TFT			35.7
4-CN-benzaldehyde	50	PyCamCN + Ni(acac)2	10	K2HPO4	10	TFT			16.3
4-CN-benzaldehyde	50	BnBiox + Ni(acac)2	10	K2HPO4	10	TFT			22.7
4-CN-benzaldehyde	50	IndenoBOX + Ni(acac)2	10	K2HPO4	10	TFT			12.8
4-CN-benzaldehyde	50	dtbbpy + NiCl2-6H2O	10	K2HPO4	10	TFT			38.5
4-CN-benzaldehyde	50	diOMe bpy + NiCl2-6H2O	10	K2HPO4	10	TFT			8.7
4-CN-benzaldehyde	50	diMe bpy + NiCl2-6H2O	10	K2HPO4	10	TFT			20.8
4-CN-benzaldehyde	50	diMe Phen + NiCl2-6H2O	10	K2HPO4	10	TFT			0.0
4-CN-benzaldehyde	50	diOMe phen + NiCl2-6H2O	10	K2HPO4	10	TFT			5.4
4-CN-benzaldehyde	50	bphen + NiCl2-6H2O	10	K2HPO4	10	TFT			20.9
4-CN-benzaldehyde	50	ttbterpy + NiCl2-6H2O	10	K2HPO4	10	TFT			85.8
4-CN-benzaldehyde	50	BPyCamCN + NiCl2-6H2O	10	K2HPO4	10	TFT			14.1
4-CN-benzaldehyde	50	Me bpp + NiCl2-6H2O	10	K2HPO4	10	TFT			8.3
4-CN-benzaldehyde	50	PyCamCN + NiCl2-6H2O	10	K2HPO4	10	TFT			0.0
4-CN-benzaldehyde	50	BnBiox +	10	K2HPO4	10	TFT			20.2

		NiCl2-6H2O							
4-CN-benzaldehyde	50	IndenoBOX + NiCl2-6H2O	10	K2HPO4	10	TFT			18.0
pentafluorobenzaldehyde	50	dtbbpy + NiBr2(DME)	10	Cs2CO3	10	TFT			46.6
pentafluorobenzaldehyde	50	diOMe bpy + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	diMe bpy + NiBr2(DME)	10	Cs2CO3	10	TFT			6.0
pentafluorobenzaldehyde	50	diMe Phen + NiBr2(DME)	10	Cs2CO3	10	TFT			5.1
pentafluorobenzaldehyde	50	diOMe phen + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	bphen + NiBr2(DME)	10	Cs2CO3	10	TFT			27.2
pentafluorobenzaldehyde	50	ttbterpy + NiBr2(DME)	10	Cs2CO3	10	TFT			55.7
pentafluorobenzaldehyde	50	BPyCamCN + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	Me bpp + NiBr2(DME)	10	Cs2CO3	10	TFT			8.2
pentafluorobenzaldehyde	50	PyCamCN + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	BnBiox + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	IndenoBOX + NiBr2(DME)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	dtbbpy + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			49.8
pentafluorobenzaldehyde	50	diOMe bpy + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			28.5
pentafluorobenzaldehyde	50	diMe bpy + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			25.1
pentafluorobenzaldehyde	50	diMe Phen + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			26.2
pentafluorobenzaldehyde	50	diOMe phen + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			15.8
pentafluorobenzaldehyde	50	bphen + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			49.7
pentafluorobenzaldehyde	50	ttbterpy + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			51.9
pentafluorobenzaldehyde	50	BPyCamCN + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	Me bpp + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			15.3
pentafluorobenzaldehyde	50	PyCamCN + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			5.3
pentafluorobenzaldehyde	50	BnBiox + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	IndenoBOX + Ni(cod)(DQ)	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	dtbbpy + Ni(acac)2	10	Cs2CO3	10	TFT			52.3
pentafluorobenzaldehyde	50	diOMe bpy +	10	Cs2CO3	10	TFT			48.1

		Ni(acac)2							
pentafluorobenzaldehyde	50	diMe bpy + Ni(acac)2	10	Cs2CO3	10	TFT			43.4
pentafluorobenzaldehyde	50	diMe Phen + Ni(acac)2	10	Cs2CO3	10	TFT			36.8
pentafluorobenzaldehyde	50	diOMe phen + Ni(acac)2	10	Cs2CO3	10	TFT			31.9
pentafluorobenzaldehyde	50	bphen + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	ttbterpy + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	BPyCamCN + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	Me bpp + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	PyCamCN + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	BnBiox + Ni(acac)2	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	IndenoBOX + Ni(acac)2	10	Cs2CO3	10	TFT			8.1
pentafluorobenzaldehyde	50	dtbbpy + NiCl2-6H2O	10	Cs2CO3	10	TFT			62.3
pentafluorobenzaldehyde	50	diOMe bpy + NiCl2-6H2O	10	Cs2CO3	10	TFT			14.1
pentafluorobenzaldehyde	50	diMe bpy + NiCl2-6H2O	10	Cs2CO3	10	TFT			16.4
pentafluorobenzaldehyde	50	diMe Phen + NiCl2-6H2O	10	Cs2CO3	10	TFT			34.4
pentafluorobenzaldehyde	50	diOMe phen + NiCl2-6H2O	10	Cs2CO3	10	TFT			21.2
pentafluorobenzaldehyde	50	bphen + NiCl2-6H2O	10	Cs2CO3	10	TFT			39.7
pentafluorobenzaldehyde	50	ttbterpy + NiCl2-6H2O	10	Cs2CO3	10	TFT			21.0
pentafluorobenzaldehyde	50	BPyCamCN + NiCl2-6H2O	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	Me bpp + NiCl2-6H2O	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	PyCamCN + NiCl2-6H2O	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	BnBiox + NiCl2-6H2O	10	Cs2CO3	10	TFT			0.0
pentafluorobenzaldehyde	50	IndenoBOX + NiCl2-6H2O	10	Cs2CO3	10	TFT			0.0

IX. Mass Table for Parallel Oxetane Arylation

Row	Column	Expected	Observed	Ion
A	1	306.1	306.2	$[M\text{-}tBu+H]^+$
A	2	249.1	249.1	$[M+H]^+$
A	3	-	N/A	-
A	4	261.2	261.3	$[M+H]^+$
A	5		N/A	-
A	6	252.1	252.1	$[M+H]^+$
B	1	-	N/A	-
B	2	233.1	233.1	$[M+H]^+$
B	3	-	N/A	-
B	4	-	N/A	-
B	5	263.1	263.0	$[M+H]^+$
B	6	327.1	327.3	$[M+H]^+$
C	1	247.1	247.1	$[M+H]^+$
C	2	243.1	243.1	$[M+H]^+$
C	3	150.1	150.0	$[M+H]^+$
C	4	217.1	217.2	$[M+H]^+$
C	5	295.1	295.2	$[M+H]^+$
C	6	391.1	391.2	$[M+Na]^+$
D	1	224.1	224.1	$[M\text{-}tBu+H]^+$
D	2	210.1	210.1	$[M\text{-}tBu+H]^+$
D	3	-	N/A	-
D	4	190.1	190.1	$[M+H]^+$
D	5	244.1	244.1	$[M+H]^+$
D	6	312.1	312.1	$[M+H]^+$