Supporting Information

Ni-catalyzed Electrochemical Cross-Electrophile Coupling Paired with Oxygen Evolution Reaction

Contents

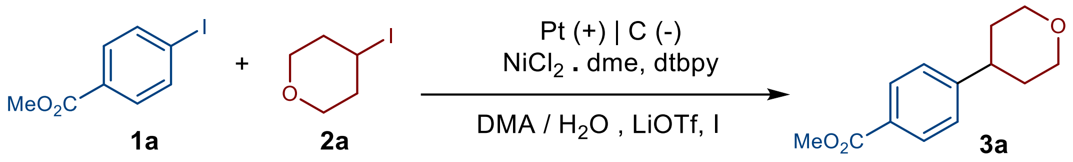
1. General Information

Nuclear Magnetic Resonance spectra were recorded on a Bruker Avance 400 MHz or 600MHz instruments at ambient temperature. All ^{1}H NMR spectra were measured in part per million (ppm) relative to the signals of tetramethylsilane (TMS, 0.00 ppm) added into the deuterated chloroform ($CDCl_3$, 7.26 ppm) unless otherwise stated. Data for ^{1}H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets), coupling constants, and integration. All ^{13}C NMR spectra were reported in ppm relative to tetramethylsilane (0.00 ppm) unless otherwise stated, and were obtained with complete ^{1}H decoupling. All GC analyses were performed on a Perkin-Elmer Clarus 400 GC system with an FID detector. Chiral HPLC analysis was performed with a Shimadzu HPLC instrument (LC-2030 Plus). High-resolution mass spectra were obtained with an AB Triple 5600 mass spectrometer by ESI on a TOF mass analyzer.

Unless otherwise noted, all chemicals used in the preparations of starting materials and in the nickel catalyzed electroreductive carbonylative reactions were commercially available and were used as received without further purifications or prepared according to previous work. Solvents transferred to the glove box without exposure to air. Anhydrous dimethylacetamide (DMA) (99.9% purity) was purchased from China National Pharmaceutical Group Corporation.

2. Detailed Screening of the Reaction Conditions

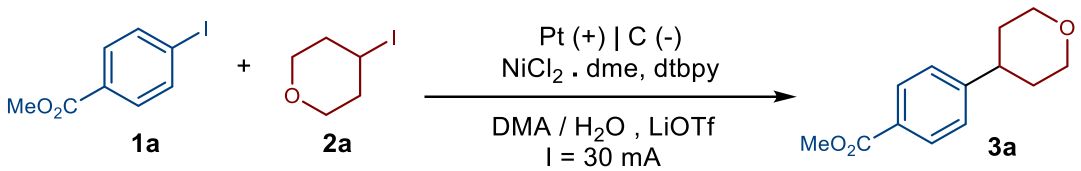
Table S1 Electric Current Screening



Entry	Current	Yield
1	15 mA	39%
2	20 mA	45%
3	25 mA	52%
4	30 mA	66%
5	40 mA	62%
6	50 mA	60%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 1.5 mmol. H_2O, 0.2 M lithium triflate, room temperature, 4 h.

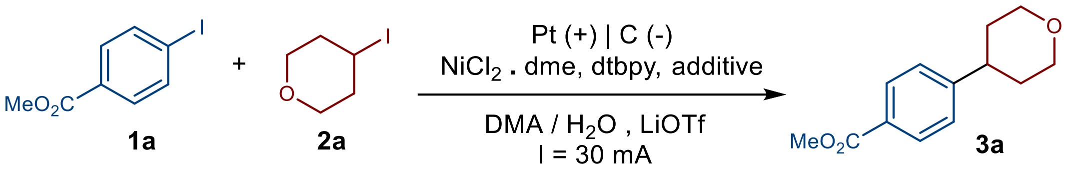
Table S2 Water Addition Screening



Entry	Water Equivalent	Yield
1	none	37%
2	3 eq.	66%
3	10 eq.	78%
4	30 eq.	86%
5	1 mL	52%
6	DMA (Water≤50 ppm)	3%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 0.2 M lithium triflate, room temperature, 30 mA, 4 h.

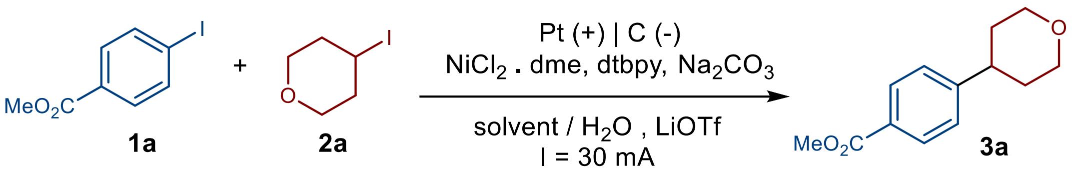
Table S3 Additives Screening



Entry	Additive	Yield
1	Na_2CO_3	>99%（95%）
2	Cs_2CO_3	88%
3	NaOH	74%
4	HCl	<50%
5	NaI	<20%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M lithium triflate, 20 mol% addictive, room tempreture, 30 mA, 4 h

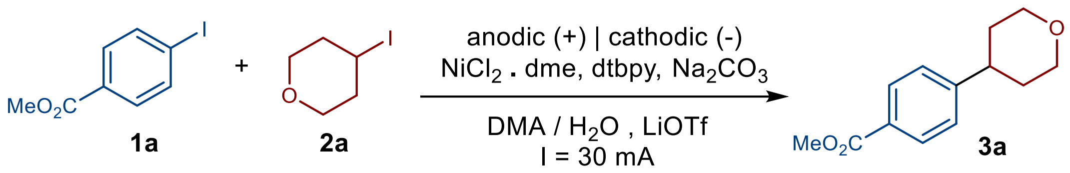
Table S4 Solvent Screening



Entry	Slovent	Yield
1	DMA	>99%（95%）
2	DMF	53%
3	NMP	35%
4	CH_3OH	n.d.

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL solvent, 30 eq H_2O, 0.2 M lithium triflate, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h.

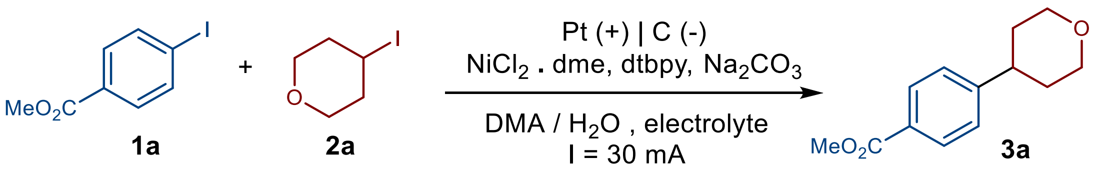
Table S5 Electrode Screening



Entry	electrode	Yield
1	Pt (+)|C felt (-)	>99%（95%）
2	Au (+)|C felt (-)	53%
3	C felt (+)|C felt (-)	38%
4	C plate (+)|C felt (-)	<30%
5	IrO_2 (+)|C felt (-)	trace
6	Pt (+)|C plate (-)	47%
7	Pt (+)|Ni foam (-)	36%
8	Pt (+)|Zn foam (-)	<30%
9	Pt (+)|Fe foam (-)	<30%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M lithium triflate, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h

Table S6 Electrode Screening

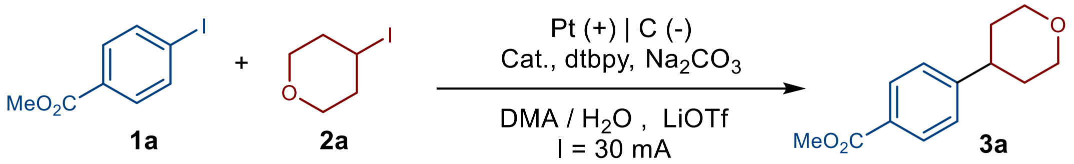


Entry	electrolyte	Yield
1	LiOTf	>99%（95%）
2	^{n}BuNClO$_4$	78%
3	^{n}BuNPF$_6$	65%
4	^{n}BuNBr	13%
5	^{n}BuNOAc	trace
5	^{n}BuNI	trace

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-

2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M electrolyte, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h.

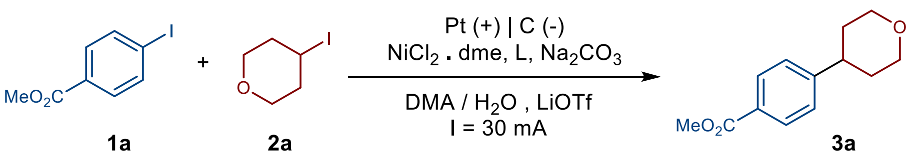
Table S7 Catalysts Screening

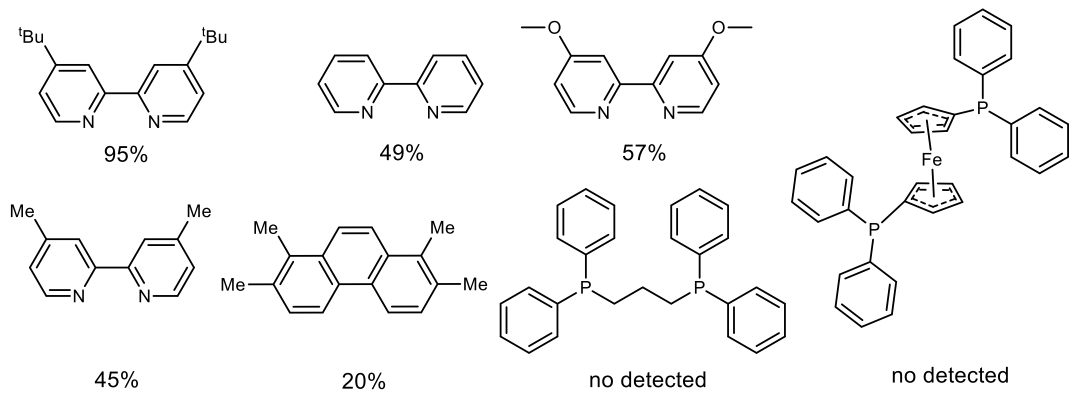


Entry	Cat.	Yield
1	$NiCl_2$·dme	（95%）>99%
2	$NiBr_2$·dme	78%
3	$NiBr_2$	57%
4	$Ni(OAc)_2$·$4H_2O$	60%
5	NiI_2	66%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% Cat., 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M electrolyte, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h.

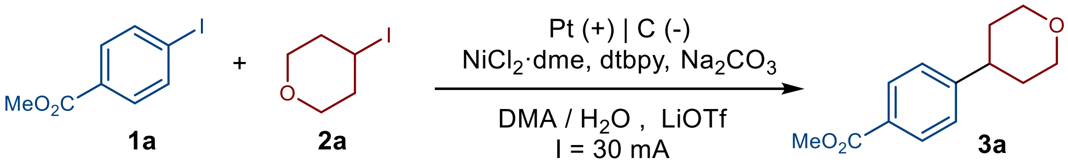
Table S8 Ligand Screening





Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M electrolyte, 20 mol% addictive, room tempreture, 30 mA, 4 h.

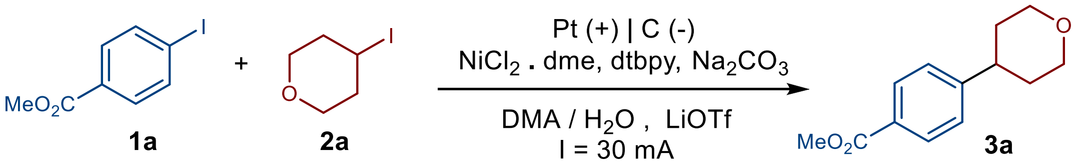
Table S9 Reaction time Screening



Entry	Time	Yield
1	1 h	36%
2	2 h	69%
3	3 h	86%
4	4 h	（95%）>99%
5	5 h	96%
6	6 h	96%

Reaction condition: 0.5 mmol **1a**, 0.6 mmol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M electrolyte, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h.

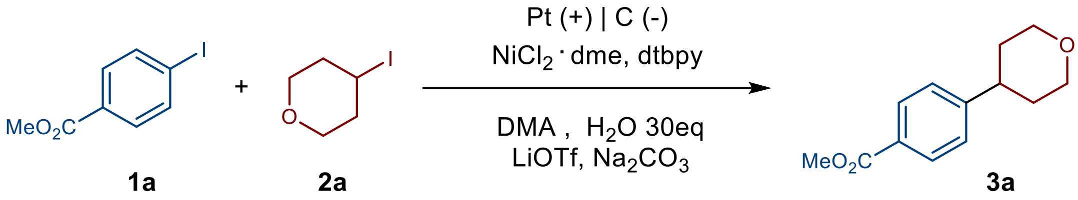
Table S10 Control Experients



Entry	Condition	Yield
1	None	95%
2	No $NiCl_2$·dme	No detection
3	No dtbpy	No detection
4	No current	No detection
5	No H_2O	37%

Reaction condition: 0.5 mmol **1a**, 0.6 mol **2a**, 10 mol% $NiCl_2$·dme, 15 mol% 4,4'-di-tert-butyl-2,2'bipyridine, 6 mL DMA, 30 eq. H_2O, 0.2 M LiOTf, 20 mol% Na_2CO_3, room tempreture, 30 mA, 4 h.

3. General Procedure for Nickel-Catalyzed Electrochemical Reductive Coupling with H_2O as Sacrificial Reductant in the undivided cell



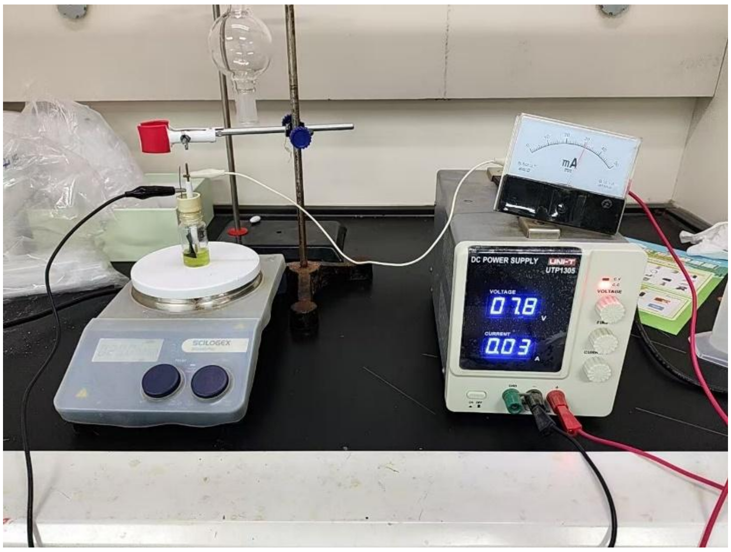


Figure S1. Reaction set-up of undivided cell

An oven-dried 20 mL re-sealable screw-cap test tube equipped with a Teflon-coated magnetic stir bar was sequentially charged with dtbpy (15 mol%), $NiCl_2$·dme (10 mol%), LiOTf (0.2 M), Na_2CO_3 (0.1 mmol), **1a** (0.5 mmol) in the glove. Then DMA (6 mL), **2a** (0.6 mmol), 270μL pure water were added into the tube in turn. All these procedures were conducted in the glovebox. Screw the vial cap with electrode (Pt anode (10 mm/10 mm/0.5 mm), graphite felt cathode (10 mm/10 mm)) onto the vial to finger tight and adapt the electrochemical cell. Then removed from the glove box. The reaction mixture was stirred and electrolyzed at a constant current (30 mA) at room temperature for 4 h. Then the reaction was quenched with H_2O, the aqueous layer was extracted with EA. The organic layers were combined, and washed with brine. Dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

4. General Procedure for Nickel-Catalyzed Electrochemical Reductive Coupling with H_2O as Sacrificial Reductant in the divided cell

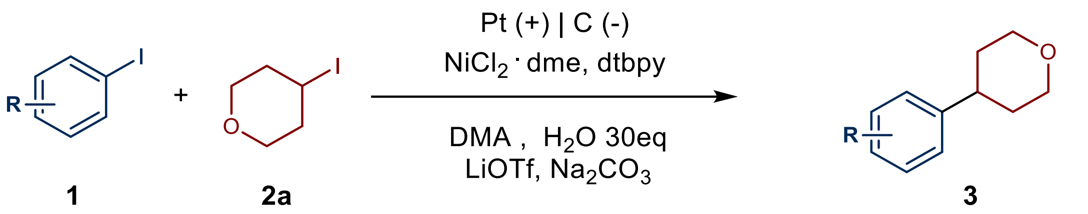


Figure S2. Reaction set-up of divided cell

A divided H-type electrolytic cell was assembled employing an anion exchange membrane as the separator, ensuring airtightness at all interfaces. The anode compartment was charged with an aqueous NaOH solution (0.2 M, 10 mL) and a platinum electrode. The cathode compartment was sequentially charged in a glovebox with dtbpy (15 mol%), $NiCl_2$·dme (10 mol%), Na_2CO_3 (20 mol%) and LiOTf (0.2 M), DMAc (10 mL), aryl-I (0.5 mmol), and alkyl iodides **2a** (0.6 mmol) were then added. A carbon felt electrode was installed as the cathode. The assembled cell was removed from the glovebox. The reaction mixture was stirred and electrolyzed at a constant current (30 mA) at room temperature for 4 h. Upon completion, the reaction was quenched with H_2O and the aqueous layer was extracted with ethyl acetate (EA). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography using petroleum ether/EA (5:1, v/v) as the eluent.

5. Procedure for Scale-up Reaction

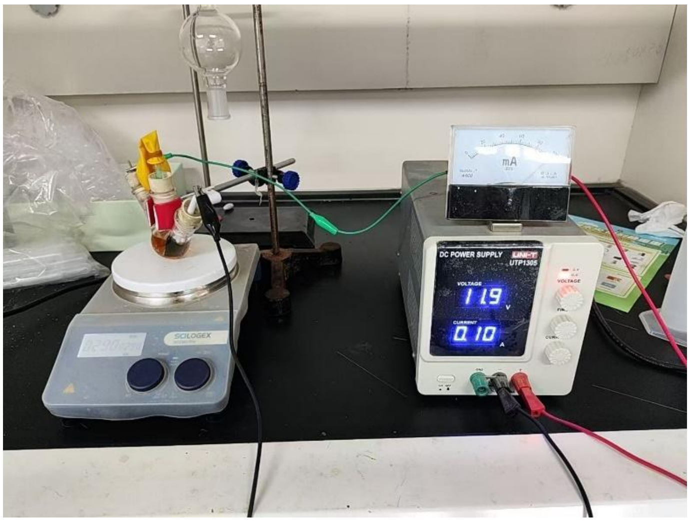


Figure S3. Scale-up reaction set-up

An oven-dried 25 mL three-necked flask equipped with a Teflon-coated magnetic stir bar was sequentially charged with dtbpy (15 mol%), $NiCl_2$·dme (10 mol%), LiOTf (0.2 M) Na_2CO_3 (2 mmol), **1a** (10 mmol) in the glove. Then DMA (24 mL), **2a** (12 mmol), $ClCO_2Pr$ (15 mmol.), 1mL pure water were added into the tube in turn. All these procedures were conducted in the glovebox. Screw the vial cap with electrode onto the vial to finger tight and adapt the electrochemical cell. Then removed from the glove box. The reaction mixture was stirred and electrolyzed at a constant current (100 mA) at room temperature for overnight. Then the reaction was quenched with H_2O, the aqueous layer was extracted with EA. The organic layers were combined, and washed with brine. Dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

6. Control Experiments

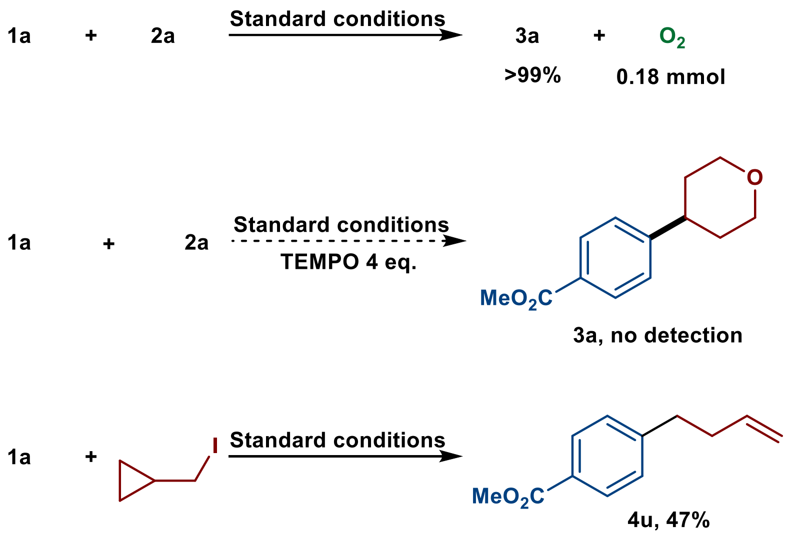


Figure S4. Control experiment

7. Cyclic Voltammetry Experiments

Cyclic voltammograms in solvent (15 mL) by using glassy carbon as the working electrode, Pt wire as the counter electrode and Ag/AgCl as the reference electrode under N_2 at room temperature. DMA (15 mL) containing 0.1 M $^nBu_4PF_6$ was poured into the electro chemical cell in all experiments. The scan rate was 100 mV s^{-1}.

1) Reduction Potential of Substrates

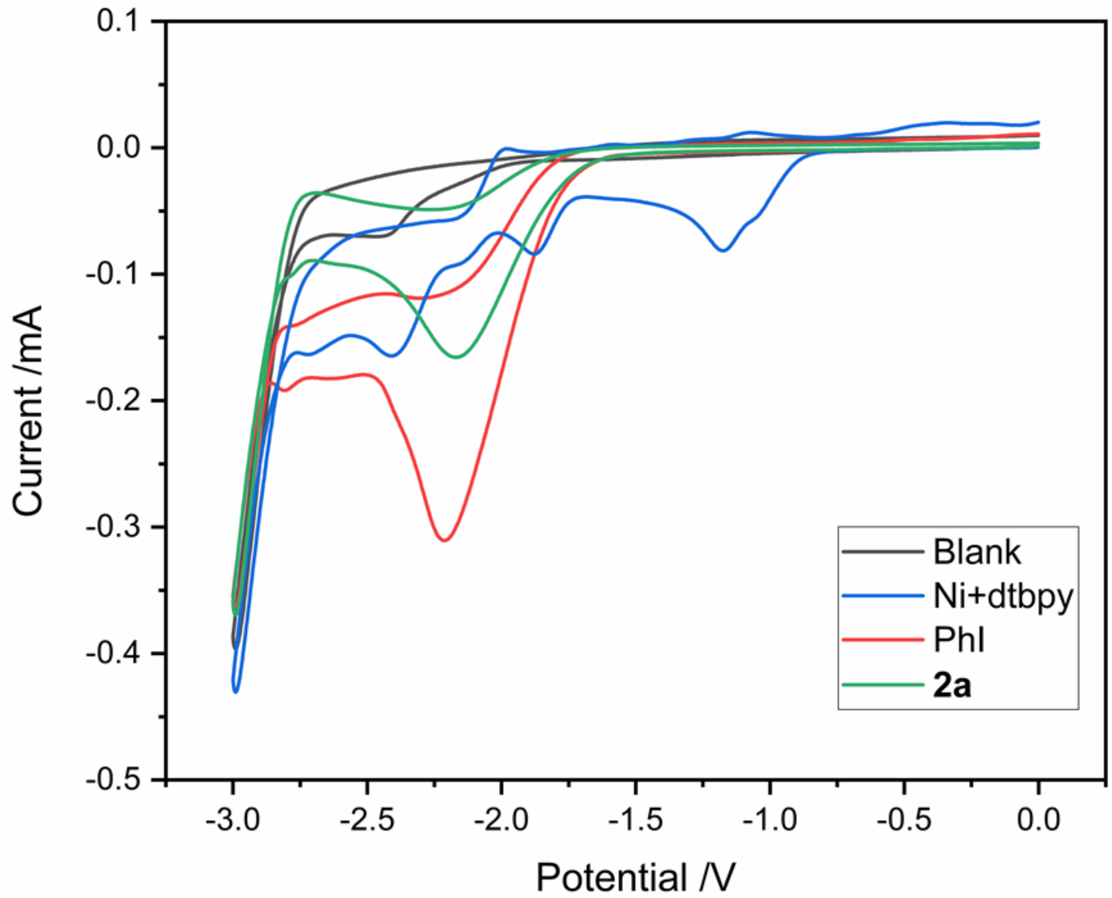


Figure S5: Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs^{-1} in: (Gary) DMAc (15 mL), $^nBu_4PF_6$ (0.1 M); (Blue) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^nBu_4PF_6$ (0.1 M); (Red) PhI (0.1 mmol), DMAc (15 mL), $^nBu_4PF_6$ (1 mol); (Green) **2a** (0.1 mmol), DMA (10 mL), $^nBu_4PF_6$ (1 mol).

2) Interaction between [Ni]/L and PhI

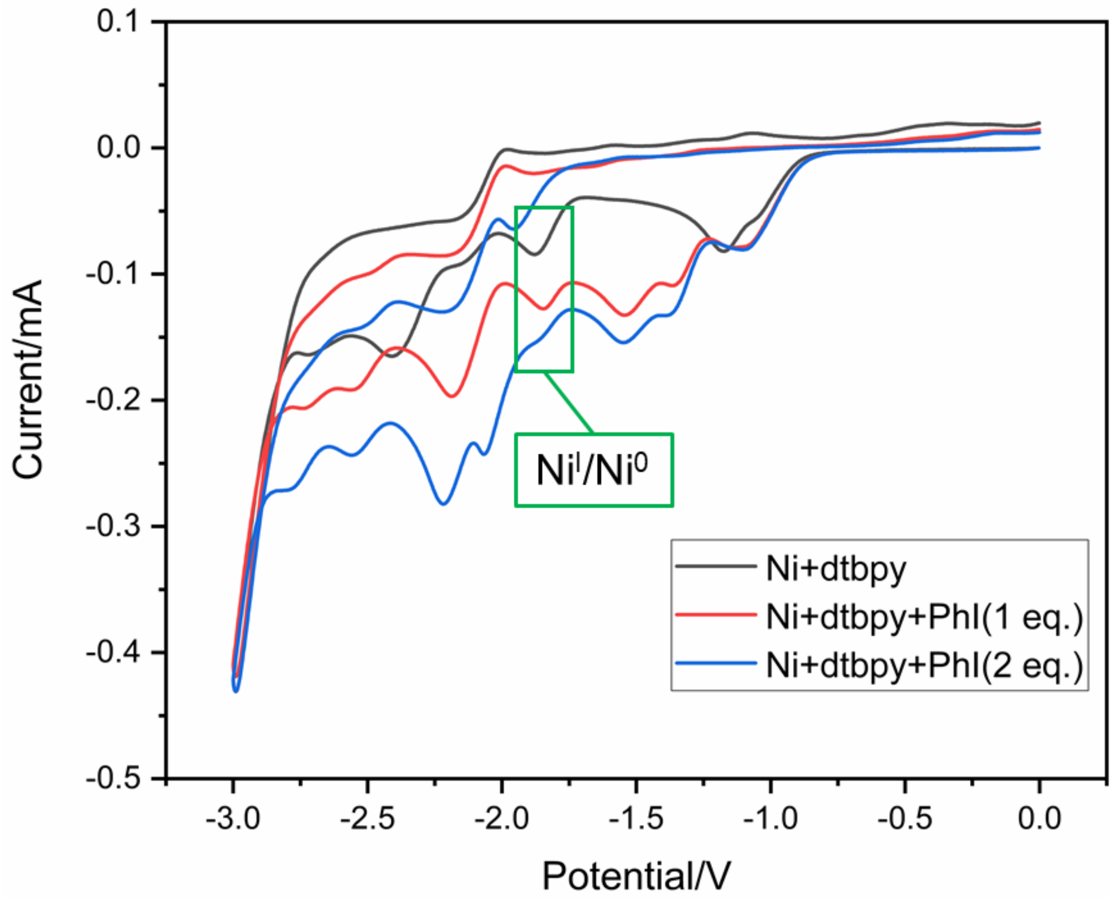


Figure S6: Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs^{-1} in: (Gray) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (0.1 M); (Red) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (1 mol), PhI (0.1 mmol); (Blue) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (1 mol), PhI (0.2 mmol)

3) Interaction between [Ni]/L and 2a

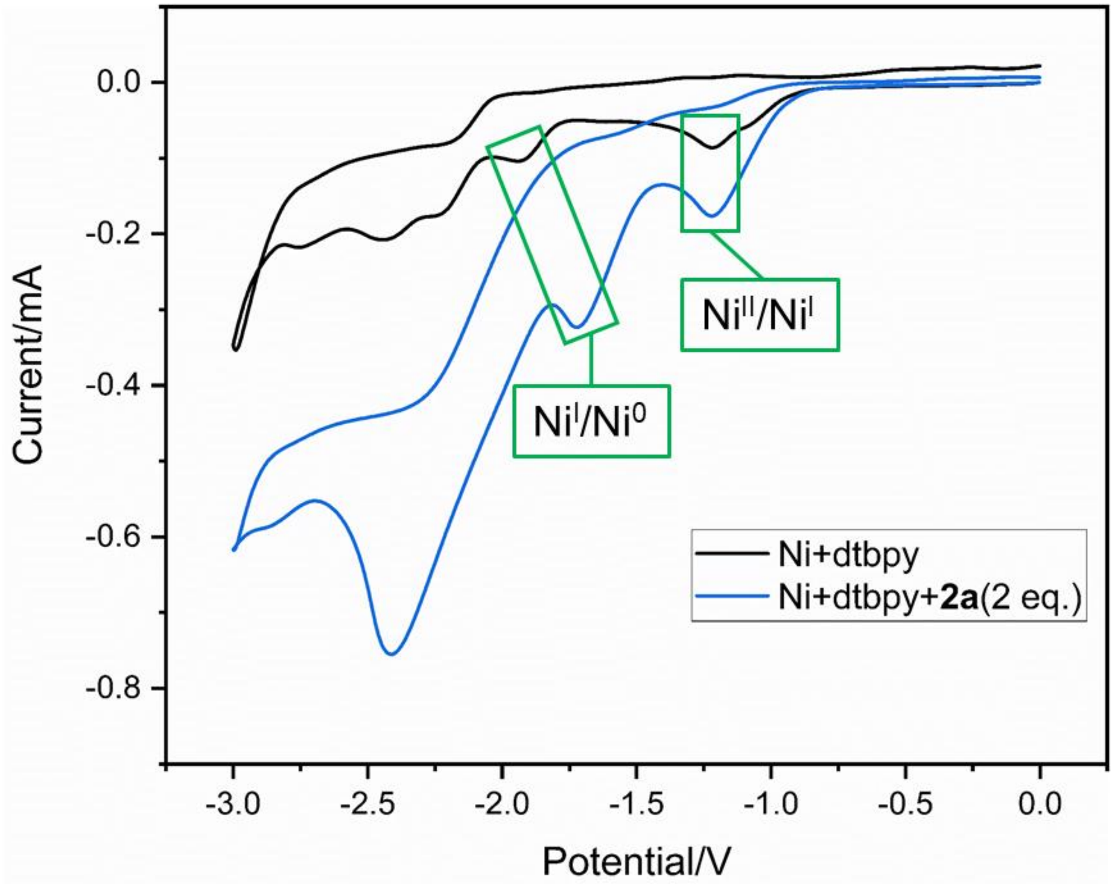
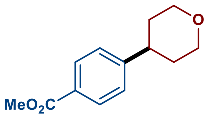


Figure S7: Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs^{-1} in: (Gray) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (0.1 M); (Red) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (1 mol), **2a** (0.1 mmol); (Blue) $NiCl_2$·dme (0.05 mmol)/dtbpy (0.075 mmol), DMAc (15 mL), $^{n}Bu_4PF_6$ (1 mol), **2a** (0.2 mmol)

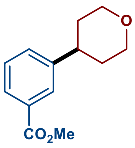
8. Analytical Data of Products



methyl 4-(tetrahydro-2H-pyran-4-yl)benzoate[1]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.91 (d, J = 8.3 Hz, 2H), 7.21 (d, J = 8.1 Hz, 2H), 4.02 – 3.98 (m, 2H), 3.82 (s, 3H), 3.45 (td, J = 11.5, 2.8 Hz, 2H), 2.77-2.70 (m, 1H), 1.80 – 1.67 (m, 4H).

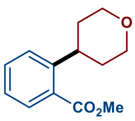
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.98, 151.09, 129.90, 128.30, 126.80, 68.20, 52.00, 41.64, 33.58.



methyl 3-(tetrahydro-2H-pyran-4-yl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.86 (m, 2H), 7.41 – 7.36 (m, 2H), 4.09 – 4.04 (m, 2H), 3.90 (d, *J* = 1.3 Hz, 3H), 3.45 (td, *J* = 11.5, 2.6 Hz, 2H), 2.82 – 2.75 (m, 1H), 1.84 – 1.73 (m, 4H).

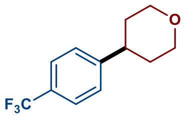
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.01, 146.02, 131.24, 130.24, 128.46, 127.85, 127.48, 68.13, 51.96, 41.25, 33.66.



methyl 2-(tetrahydro-2H-pyran-4-yl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.51 (td, *J* = 7.5, 1.3 Hz, 1H), 7.44 – 7.42 (m, 1H), 7.29 – 7.27 (m, 1H), 4.10 (dd, *J* = 11.4, 4.3 Hz, 2H), 3.93 (s, 3H), 3.70 – 3.64 (m, 1H), 3.60 (td, *J* = 11.7, 2.2 Hz, 2H), 1.87 – 1.78 (m, 4H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.44, 146.78, 132.04, 130.25, 129.71, 126.93, 125.91, 68.54, 52.04, 37.44, 33.90.

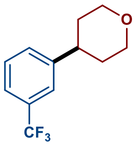


4-(4-(trifluoromethyl)phenyl)tetrahydro-2H-pyran[2]**:** The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.12 – 4.08 (m, 2H), 3.54 (td, *J* = 11.5, 2.8 Hz, 2H), 2.87 – 2.78 (m, 1H), 1.89-1.75 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 149.74, 128.62 (q, *J* = 32.45 Hz), 127.07, 125.43 (q, *J* = 3.80 Hz), 124.23 (q, *J* = 272.90 Hz), 68.14, 41.46, 33.60.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.36.

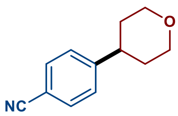


4-(3-(trifluoromethyl)phenyl)tetrahydro-2H-pyran: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.40 (m, 4H), 4.12 – 4.08 (m, 2H), 3.57 – 3.52 (m, 2H), 2.87 – 2.80 (m, 1H), 1.89-1.75 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 146.65, 130.79 (q, *J* = 32.22 Hz), 130.10, 128.95, 125.50, 123.57, 123.21, 68.17, 41.42, 33.71.

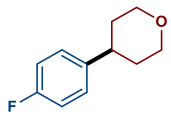
^{19}F NMR (377 MHz, $CDCl_3$) δ -62.56.



4-(tetrahydro-2H-pyran-4-yl)benzonitrile[3]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 4.02 (dd, *J* = 10.4, 3.0 Hz, 2H), 3.46 (td, *J* = 11.3, 3.5 Hz, 2H), 2.81 -2.73 (m, 1H), 1.78-1.69 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 151.18, 132.42, 127.64, 118.96, 110.19, 68.04, 41.71, 33.39.

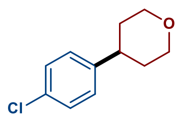


4-(4-fluorophenyl)tetrahydro-2H-pyran: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.20 (m, 2H), 7.02 (d, *J* = 8.7 Hz, 2H), 4.11 (d, *J* = 10.8 Hz, 2H), 3.55 (td, *J* = 11.3, 3.5 Hz, 2H), 2.81 – 2.73 (m, 1H), 1.84 – 1.78 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.38 (d, *J* = 245.03 Hz), 141.53, 128.04 (d, *J* = 7.88 Hz), 115.21 (d, *J* = 21.11 Hz), 68.32, 40.85, 34.10.

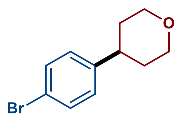
^{19}F NMR (377 MHz, $CDCl_3$) δ -117.07.



4-(4-chlorophenyl)tetrahydro-2H-pyran[3]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.2 Hz, 2H), 4.11 (d, *J* = 10.6 Hz, 2H), 3.55 (t, *J* = 11.4 Hz, 2H), 2.81 – 2.73 (m, 1H), 1.79 (tt, *J* = 11.1, 5.2 Hz, 4H).

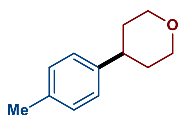
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.21, 131.83, 128.53, 128.01, 68.17, 40.91, 33.79.



4-(4-bromophenyl)tetrahydro-2H-pyran[3]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 3.98 (dd, *J* = 10.5, 3.6 Hz, 2H), 3.43 (td, *J* = 11.2, 3.5 Hz, 2H), 2.63 (m, 1H), 1.74-1.64 (m, 4H).

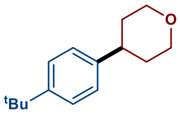
^{13}C NMR (101 MHz, CDCl3) δ 143.75, 130.51, 127.46, 118.90, 67.18, 39.99, 32.74.



4-(p-tolyl)tetrahydro-2H-pyran[2]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.17 – 7.14 (m, 4H), 4.11-4.08 (m, 2H), 3.55 (td, *J* = 11.7, 2.3 Hz, 2H), 2.77 – 2.71 (m, 1H), 2.35 (s, 3H), 1.86 – 1.76 (m, 4H).

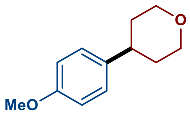
^{13}C NMR (151 MHz, $CDCl_3$) δ 142.93, 135.82, 129.19, 126.60, 68.45, 41.15, 34.05, 20.97.



4-(4-(tert-butyl)phenyl)tetrahydro-2H-pyran[4]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 4.12-4.10 (m, 2H), 3.56 (td, *J* = 11.6, 2.5 Hz, 2H), 2.78-2.74 (m, 1H), 1.87 – 1.78 (m, 4H), 1.35 (d, *J* = 0.8 Hz, 9H).

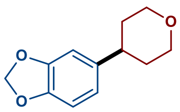
^{13}C NMR (151 MHz, $CDCl_3$) δ 149.05, 142.80, 126.33, 125.35, 68.44, 40.97, 34.36, 33.95, 31.37.



4-(4-methoxyphenyl)tetrahydro-2H-pyran[3]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.07 (d, J = 8.3 Hz, 2H), 6.78 (d, J = 8.3 Hz, 2H), 4.01 – 3.97 (m, 2H), 3.71 (s, 3H), 3.44 (td, J = 11.2, 3.3 Hz, 2H), 2.62 (m, 1H), 1.76-1.66 (m, 4H).

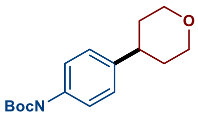
^{13}C NMR (101 MHz, $CDCl_3$) δ 158.05, 138.13, 127.61, 113.89, 68.46, 55.27, 40.72, 34.23.



5-(tetrahydro-2H-pyran-4-yl)benzo[d][1,3]dioxole[5]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 6.78 – 6.70 (m, 2H), 6.69 (dt, *J* = 8.0, 1.1 Hz, 1H), 5.95 (s, 2H), 4.10 – 4.07 (m, 2H), 3.52 (td, *J* = 11.4, 3.1 Hz, 2H), 2.70 - 2.69 (m, 1H), 1.80 – 1.72 (m, 4H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 147.63, 145.81, 139.98, 119.45, 108.18, 107.18, 100.79, 68.33, 41.32, 34.22.

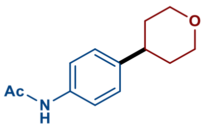


tert-butyl (4-(tetrahydro-2H-pyran-4-yl)phenyl)carbamate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 2H), 6.83 (s, 1H), 4.12-4.08 (m, 2H), 3.54 (td, *J* = 11.3, 3.0 Hz, 2H), 2.75 – 2.67 (m, 1H), 1.85 – 1.73 (m, 4H), 1.54 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 152.89, 140.36, 136.56, 126.97, 118.78, 80.12, 68.21, 40.70, 33.87, 28.23.

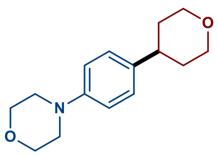
HRMS (ESI): m/z: $[M + Na]^+$ target$C_{16}H_{23}NNaO_3^+$ 300.1570; found300.1564.



N-(4-(tetrahydro-2H-pyran-4-yl)phenyl)acetamide: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 4.09 – 4.05 (m, 2H), 3.52 (td, *J* = 11.3, 3.3 Hz, 2H), 2.76 – 2.68 (m, 1H), 2.16 (s, 3H), 1.84 – 1.74 (m, 4H).

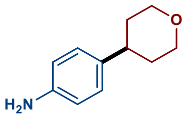
^{13}C NMR (101 MHz, $CDCl_3$) δ 168.27, 141.19, 136.00, 127.19, 120.22, 68.34, 40.96, 33.93, 24.50.



4-(4-(tetrahydro-2H-pyran-4-yl)phenyl)morpholine: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15 (d, *J* = 8.3 Hz, 2H), 6.88 (d, *J* = 8.3 Hz, 2H), 4.13 – 4.02 (m, 2H), 3.86 (t, *J* = 4.7 Hz, 4H), 3.52 (td, *J* = 11.2, 3.3 Hz, 2H), 3.14 (t, *J* = 4.8 Hz, 4H), 2.73 - 2.66 (m, 1H), 1.84-1.72 (m, *J* = 11.6, 5.7 Hz, 4H).

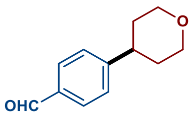
^{13}C NMR (101 MHz, $CDCl_3$) δ 149.69, 137.52, 127.34, 115.85, 68.41, 66.92, 49.54, 40.59, 34.06.



4-(tetrahydro-2H-pyran-4-yl)aniline: The product was purified by silica gel column chromatography, using petroleum ether/EA = 10/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.02 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.0 Hz, 2H), 4.09 – 4.04 (m, 2H), 3.55-3.47 (m, 2H), 2.69 – 2.60 (m, 1H), 1.82 – 1.70 (m, 4H).

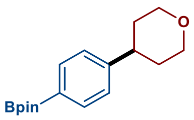
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.59, 136.04, 127.39, 115.20, 68.40, 40.58, 34.16.



4-(tetrahydro-2H-pyran-4-yl)benzaldehyde[6]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.98 (s, 1H), 7.84 (d, *J* = 8.o Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 4.13 – 4.08 (m, 2H), 3.58-3.51 (m, 2H), 2.90 – 2.82 (m, 1H), 1.82 – 1.76 (m, 4H).

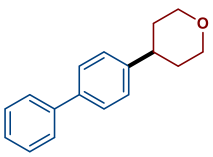
^{13}C NMR (101 MHz, $CDCl_3$) δ 191.81, 152.80, 134.81, 130.03, 127.37, 68.03, 41.75, 33.39.



4,4,5,5-tetramethyl-2-(4-(tetrahydro-2H-pyran-4-yl)phenyl)-1,3,2-dioxaborolane[3]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 7.6 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 4.07 (dd, *J* = 10.9, 3.9 Hz, 2H), 3.52 (td, *J* = 11.6, 2.5 Hz, 2H), 2.80 -2.72 (m, 1H), 1.88 – 1.74 (m, 4H), 1.33 (s, 12H).

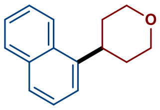
^{13}C NMR (101 MHz, $CDCl_3$) δ 149.14, 135.11, 126.23, 83.69, 68.36, 41.79, 33.74, 24.87.



4-([1,1'-biphenyl]-4-yl)tetrahydro-2H-pyran: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.52 (m, 4H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.29 (dd, *J* = 17.2, 7.7 Hz, 3H), 4.08 (dd, J = 10.9, 3.6 Hz, 2H), 3.55 – 3.48 (m, 2H), 2.80 – 2.74 (m, 1H), 1.89 – 1.75 (m, 4H).

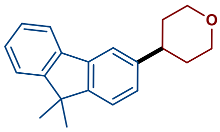
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.86, 140.85, 139.17, 128.65, 127.15, 127.07, 127.02, 126.91, 68.29, 41.13, 33.85.



4-(naphthalen-1-yl)tetrahydro-2H-pyran: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.20 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 7.9 Hz, 1H), 7.81 (d, J = 8.1 Hz, 1H), 7.63 – 7.47 (m, 4H), 4.23 (dd, J = 11.0, 3.5 Hz, 2H), 3.80 – 3.74 (td, J = 11.5, 2.6 Hz, 2H), 3.66 (tt, J = 11.5, 4.1 Hz, 1H), 2.10 – 1.97 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.41, 133.89, 131.03, 129.02, 126.69, 125.78, 125.60, 125.31, 122.68, 122.44, 68.59, 33.79, 33.67.

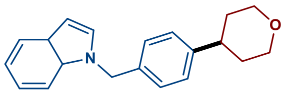


4-(9,9-dimethyl-9H-fluoren-3-yl)tetrahydro-2H-pyran: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.75 (dd, J = 12.6, 7.3 Hz, 2H), 7.49 (d, J = 7.5 Hz, 1H), 7.41 – 7.34 (m, 3H), 7.27 (d, J = 8.9 Hz, 1H), 4.19 (dd, J = 11.0, 3.7 Hz, 2H), 3.63 (td, J = 11.6, 2.3 Hz, 2H), 2.91 (tt, J = 11.6, 4.1 Hz, 1H), 1.99 – 1.88 (m, 4H), 1.56 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.88, 153.55, 145.20, 139.01, 137.47, 126.84, 125.44, 122.45, 120.84, 119.88, 119.70, 68.40, 46.71, 41.86, 34.19, 27.19.

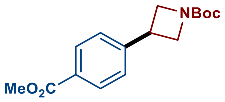
HRMS (ESI)： m/z: [M + e]$^-$ Calcd for $C_{20}H_{22}Oe^-$ 278.1665； Found278.1670.



1-(4-(tetrahydro-2H-pyran-4-yl)benzyl)-3a,7a-dihydro-1H-indole: The product was purified by silica gel column chromatography, using petroleum ether/EA = 20/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.15 (m, 9H), 6.63 (d, *J* = 3.1 Hz, 1H), 5.35 (s, 2H), 4.14 (dd, *J* = 10.3, 3.6 Hz, 2H), 3.58 (td, *J* = 11.5, 2.7 Hz, 2H), 2.79 (ddt, *J* = 11.4, 7.1, 4.3 Hz, 1H), 1.81 (dt, *J* = 15.0, 7.6 Hz, 4H).

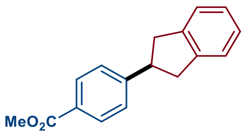
^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.19, 136.20, 135.47, 131.95, 128.63, 128.12, 127.03, 126.91, 121.57, 120.90, 119.42, 109.60, 101.57, 68.25, 49.67, 41.10, 33.80.



tert-butyl 3-(4-(methoxycarbonyl)phenyl)azetidine-1-carboxylate[7]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 4.35 (t, *J* = 8.6 Hz, 2H), 3.97 (dd, *J* = 8.5, 6.0 Hz, 2H), 3.90 (s, 3H), 3.81-3.74 (m, 1H), 1.46 (s, 9H).

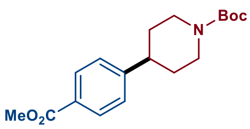
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.62, 156.18, 147.32, 129.91, 128.76, 126.67, 79.57, 56.13, 51.94, 33.32, 28.26.



methyl 4-(2,3-dihydro-1H-inden-2-yl)benzoate[8]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.33 – 7.26 (m, 2H), 7.27 (dd, *J* = 5.6, 3.2 Hz, 2H), 3.97 (s, 3H), 3.79 (q, *J* = 8.4 Hz, 1H), 3.44 (dd, *J* = 15.6, 8.2 Hz, 2H), 3.15 (dd, *J* = 15.6, 8.6 Hz, 2H).

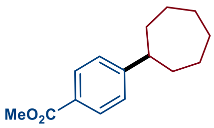
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.91, 150.88, 142.40, 129.72, 128.06, 126.96, 126.51, 124.25, 51.88, 45.23, 40.61.



tert-butyl 4-(4-(methoxycarbonyl)phenyl)piperidine-1-carboxylate[8]**:** The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 4.18 (d, *J* = 13.0 Hz, 2H), 3.81 (d, *J* = 0.9 Hz, 3H), 2.76 – 2.59 (m, 3H), 1.76 – 1.72 (m, 2H), 1.55 (tt, *J* = 12.6, 6.3 Hz, 2H), 1.40 (d, *J* = 1.0 Hz, 9H).

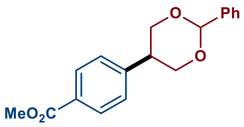
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 166.84, 154.66, 150.93, 129.76, 128.21, 126.71, 79.40, 51.87, 44.13, 42.67, 32.76, 28.36.



methyl 4-cycloheptylbenzoate[8]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.3 Hz, 2H), 3.92 (s, 3H), 2.74 (tt, *J* = 10.5, 3.6 Hz, 1H), 1.94 – 1.58 (m, 12H).

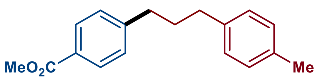
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 167.10, 155.28, 137.65, 129.68, 126.63, 51.83, 47.00, 36.43, 27.81, 27.15.



methyl 4-(2-phenyl-1,3-dioxan-5-yl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}**H NMR** (600 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.1 Hz, 2H), 7.48 – 7.47 (m, 2H), 7.40 – 7.36 (m, 5H), 5.99 (s, 1H), 4.53 – 4.52 (m, 1H), 4.22 (dd, *J* = 8.2, 6.1 Hz, 1H), 3.93 (d, *J* = 0.7 Hz, 3H), 3.78-3.75 (m, 1H), 3.17 (dd, *J* = 13.9, 6.8 Hz, 1H), 2.97 (dd, *J* = 14.0, 6.2 Hz, 1H).

^{13}**C NMR** (151 MHz, $CDCl_3$) δ 166.97, 142.70, 138.11, 129.87, 129.31, 129.17, 128.65, 128.37, 126.33, 103.52, 70.08, 52.06, 39.46.

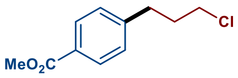


methyl 4-(3-(p-tolyl)propyl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.1 Hz, 2H), 7.27 (dd, *J* = 8.0, 5.4 Hz, 4H), 7.13 (d, *J*

= 7.8 Hz, 2H), 3.94 (s, 3H), 2.87 (dt, J = 29.0, 7.4 Hz, 4H), 2.36 (s, 3H), 1.99 (q, J = 7.4 Hz, 2H).

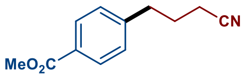
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.02, 146.87, 136.18, 132.32, 130.15, 129.69, 129.64, 128.47, 127.93, 51.93, 34.52, 33.62, 30.27, 20.95.



methyl 4-(3-chloropropyl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 80/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 8.00 (m, 2H), 7.29 (d, J = 8.1 Hz, 2H), 3.93 (s, 3H), 3.55 (t, J = 6.4 Hz, 2H), 2.86 (t, J = 7.5 Hz, 2H), 2.12 (p, J = 6.6 Hz, 2H).

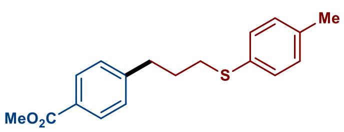
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.95, 146.12, 129.78, 128.52, 128.15, 51.96, 43.92, 33.55, 32.71.



methyl 4-(4-cyanobutyl)benzoate[9]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (d, J = 7.8 Hz, 2H), 7.25 (d, J = 8.4 Hz, 2H), 3.92 (s, 3H), 2.73 (t, J = 7.5 Hz, 2H), 2.37 (t, J = 7.0 Hz, 2H), 1.84 - 1.68 (m, 2H).

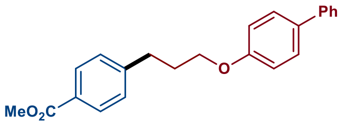
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.91, 146.61, 129.73, 128.29, 128.05, 119.37, 51.91, 34.91, 29.79, 24.73, 16.94.



methyl 4-(3-(p-tolylthio)propyl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (d, J = 8.3 Hz, 2H), 7.64 (d, J = 8.1 Hz, 2H), 7.48 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 4.58 (s, 2H), 3.93 (s, 3H), 3.53 (t, J = 6.2 Hz, 2H), 2.84 – 2.80 (m, 2H), 2.00 (dt, J = 13.8, 6.3 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.11, 147.37, 130.21, 129.73, 128.50, 127.93, 127.51, 125.33, 125.29, 72.12, 69.60, 51.96, 32.41, 30.95.

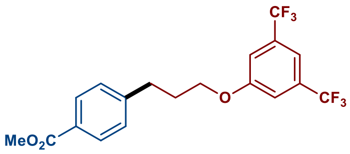


methyl 4-(3-([1,1'-biphenyl]-4-yloxy)propyl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.2 Hz, 2H), 7.57 (dd, *J* = 20.1, 7.9 Hz, 4H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 3H), 7.01 – 6.98 (m, 2H), 4.03 (t, *J* = 6.2 Hz, 2H), 3.94 (s, 3H), 2.94 – 2.91 (m, 2H), 2.17 (dt, *J* = 13.6, 6.3 Hz, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.08, 158.45, 147.03, 140.78, 133.79, 129.78, 128.69, 128.56, 128.13, 128.00, 126.69, 126.63, 114.76, 66.70, 51.97, 32.22, 30.50.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{22}NaO_3^+$ 369.1461; Found 369.1457.

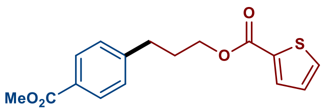


methyl 4-(3-(3,5-bis(trifluoromethyl)phenoxy)propyl)benzoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 7.9 Hz, 2H), 7.39 (s, 1H), 7.24 – 7.17 (m, 4H), 3.97 (t, *J* = 6.1 Hz, 2H), 3.84 (s, 3H), 2.82 (t, *J* = 7.6 Hz, 2H), 2.20 – 2.03 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.95, 159.43, 146.41, 133.27, 132.94, 132.61, 132.27, 129.86, 128.45, 128.23, 124.51, 121.80, 114.73, 114.23, 67.49, 51.94, 32.03, 30.14.

^{19}F NMR (377 MHz, Chloroform-*d*) δ -63.07.

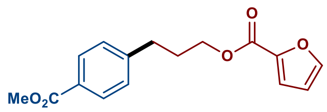


3-(4-(methoxycarbonyl)phenyl)propyl thiophene-2-carboxylate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 3.8 Hz, 1H), 7.58 (d, *J* = 4.8 Hz, 1H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.14 – 7.11 (m, 1H), 4.34 (t, *J* = 6.4 Hz, 2H), 3.92 (s, 3H), 2.87 – 2.83 (m, 2H), 2.12 (dt, *J* = 13.5, 6.6 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.94, 162.06, 146.53, 133.67, 133.33, 132.30, 129.75, 128.39, 128.02, 127.68, 64.05, 51.91, 32.18, 29.86.

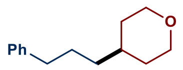
HRMS (ESI): m/z: [M + Li]$^+$ Calcd for $C_{16}H_{16}LiO_4S^+$ 311.0924; Found 311.0871.



3-(4-(methoxycarbonyl)phenyl)propyl furan-2-carboxylate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.59 – 7.58 (m, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 3.6 Hz, 1H), 6.51 (dd, *J* = 3.6, 1.8 Hz, 1H), 4.32 (t, *J* = 6.5 Hz, 2H), 3.90 (s, 3H), 2.81 (t, *J* = 7.7 Hz, 2H), 2.10 (dt, *J* = 13.7, 6.6 Hz, 2H).

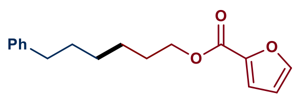
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.86, 158.52, 146.44, 146.21, 144.50, 129.68, 128.31, 127.96, 117.77, 111.70, 63.84, 51.84, 32.07, 29.75.



4-(3-phenylpropyl)tetrahydro-2H-pyran[10]**:** The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.24 (m,, 2H), 7.19 – 7.15 (m, 3H), 3.93 (dd, *J* = 11.6, 4.4 Hz, 2H), 3.38 – 3.31 (m, 2H), 2.59 (t, *J* = 7.8 Hz, 2H), 1.67 – 1.55 (m, 4H), 1.52 – 1.42 (m ,1H), 1.32 – 1.23 (m, 4H).

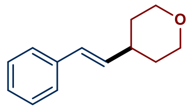
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.55, 128.30, 128.22, 125.63, 68.10, 36.52, 36.06, 34.88, 33.13, 28.25.



6-phenylhexyl furan-2-carboxylate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 1.7 Hz, 1H), 7.23 – 7.15 (m, 2H), 7.08 (d, *J* = 9.6 Hz, 4H), 6.41 (dd, *J* = 3.5, 1.8 Hz, 1H), 4.21 (t, *J* = 6.7 Hz, 2H), 2.53 (t, *J* = 7.7 Hz, 2H), 1.66 (p, *J* = 6.9 Hz, 2H), 1.56 (p, *J* = 7.5 Hz, 2H), 1.34 (tdd, *J* = 15.4, 5.9, 2.9 Hz, 4H).

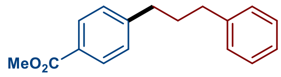
^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.86, 146.20, 144.91, 142.62, 128.40, 128.27, 125.66, 117.72, 111.79, 65.03, 35.85, 31.33, 28.87, 28.64, 25.80.



(E)-4-styryltetrahydro-2H-pyran[11]**:** The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.11 (m, 5H), 6.31 (d, *J* = 8.0 Hz, 1H), 6.09 (dd, *J* = 15.6, 6.4 Hz, 1H), 3.96 – 3.91 (m, 2H), 3.42 – 3.36 (m, 2H), 2.36 – 2.26 (m, 1H), 1.66 - 1.60 (m, 2H), 1.55-1.45 (m, 4H).

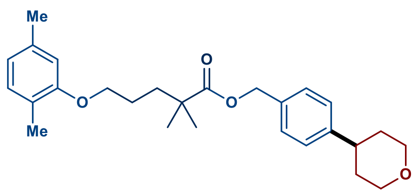
^{13}C NMR (101 MHz, $CDCl_3$) δ 137.51, 134.57, 128.50, 128.24, 127.06, 126.00, 67.71, 38.35, 32.61.



methyl 4-(3-phenylpropyl)benzoate[12]: The product was purified by silica gel column chromatography, using petroleum ether/EA = 30/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 8.2 Hz, 2H), 7.36 – 7.22 (m, 5H), 7.23 (d, *J* = 8.1 Hz, 2H), 3.95 (s, 3H), 2.73 (dt, *J* = 18.7, 7.7 Hz, 4H), 2.03 (p, *J* = 7.7 Hz, 2H).

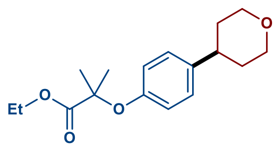
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.07, 147.76, 141.83, 129.62, 128.40, 128.35, 128.30, 127.74, 125.80, 51.89, 35.35, 35.29, 32.52.



4-(tetrahydro-2H-pyran-4-yl)benzyl 5-(2,5-dimethylphenoxy)-2-methylpentanoate: The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 7.9 Hz, 2H), 7.26 (d, *J* = 7.9 Hz, 2H), 7.06 (d, *J* = 7.4 Hz, 1H), 6.72 (d, *J* = 7.5 Hz, 1H), 6.65 (s, 1H), 5.16 (s, 2H), 4.17 – 4.12 (m, 2H), 3.94 (t, *J* = 5.1 Hz, 2H), 3.58 (td, *J* = 11.5, 2.7 Hz, 2H), 2.79 (td, *J* = 11.5, 5.7 Hz, 1H), 2.37 (s, 3H), 2.22 (s, 3H), 1.85 – 1.79 (m, 8H), 1.31 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 177.49, 156.85, 145.65, 136.31, 134.25, 130.19, 128.03, 126.78, 123.45, 120.59, 111.82, 68.24, 67.79, 65.86, 42.07, 41.21, 37.03, 33.80, 29.62, 25.07, 21.33, 15.69.

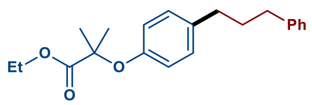


ethyl 2-methyl-2-(4-(tetrahydro-2H-pyran-4-yl)phenoxy)propanoate: White solid (59% yield.). The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.10 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 4.25 (q, *J* = 7.1 Hz, 2H), 4.10 – 4.06 (m, 2H), 3.56 – 3.49 (m, 2H), 2.70 (tt, *J* = 10.5, 5.5 Hz, 1H), 1.80 – 1.74 (m, 4H), 1.60 (s, 6H), 1.28 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 174.33, 153.75, 139.60, 127.25, 119.28, 68.40, 61.34, 40.73, 34.10, 28.90, 25.38, 14.07.

m/z: $[M + H]^+$ Calcd for $C_{17}H_{25}O_4^+$ 293.1747; Found 293.1729.

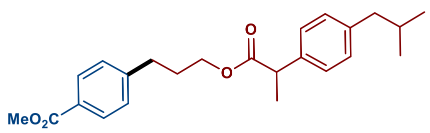


ethyl 2-methyl-2-(4-(3-phenylpropyl)phenoxy)propanoate: Yellow oil (81% yield.). The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.29 (m, 2H), 7.21 (dd, *J* = 7.6, 5.5 Hz, 3H), 7.08 (d, *J* = 8.3 Hz, 2H), 6.81 (d, *J* = 8.3 Hz, 2H), 4.30-4.25 (m, 2H), 2.64 (dt, *J* = 18.0, 7.7 Hz, 4H), 2.00-1.92 (m, 2H), 1.61 (d, *J* = 1.0 Hz, 6H), 1.31 – 1.27 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 174.44, 153.40, 142.33, 136.06, 128.99, 128.43, 128.29, 125.72, 119.32, 79.10, 61.35, 35.41, 34.59, 33.05, 25.38, 14.10.

m/z: $[M + Na]^+$ Calcd for $C_{21}H_{26}NaO_3^+$ 349.1774; Found 349.1770.

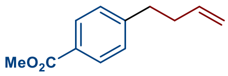


methyl 4-(3-((2-(4-isobutylphenyl)propanoyl)oxy)propyl)benzoate: White solid (81% yield.). The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.2 Hz, 2H), 7.28 – 7.24 (m, 2H), 7.14 – 7.12 (m, 4H), 4.13 – 4.03 (m, 2H), 3.92 (s, 3H), 3.72 (q, *J* = 7.2 Hz, 1H), 2.60 (t, *J* = 7.7 Hz, 2H), 2.47 (d, *J* = 7.2 Hz, 2H), 1.97 – 1.81 (m, 3H), 1.52 (d, *J* = 7.2 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 174.63, 167.03, 146.64, 140.59, 137.81, 129.71, 129.32, 128.41, 127.97, 127.16, 63.47, 51.95, 45.16, 45.00, 31.95, 30.16, 29.82, 22.33, 18.25.

m/z: [M + Na]$^{+}$ Calcd for $C_{24}H_{30}NaO_4^{+}$ 405.2036； Found 405.2059.

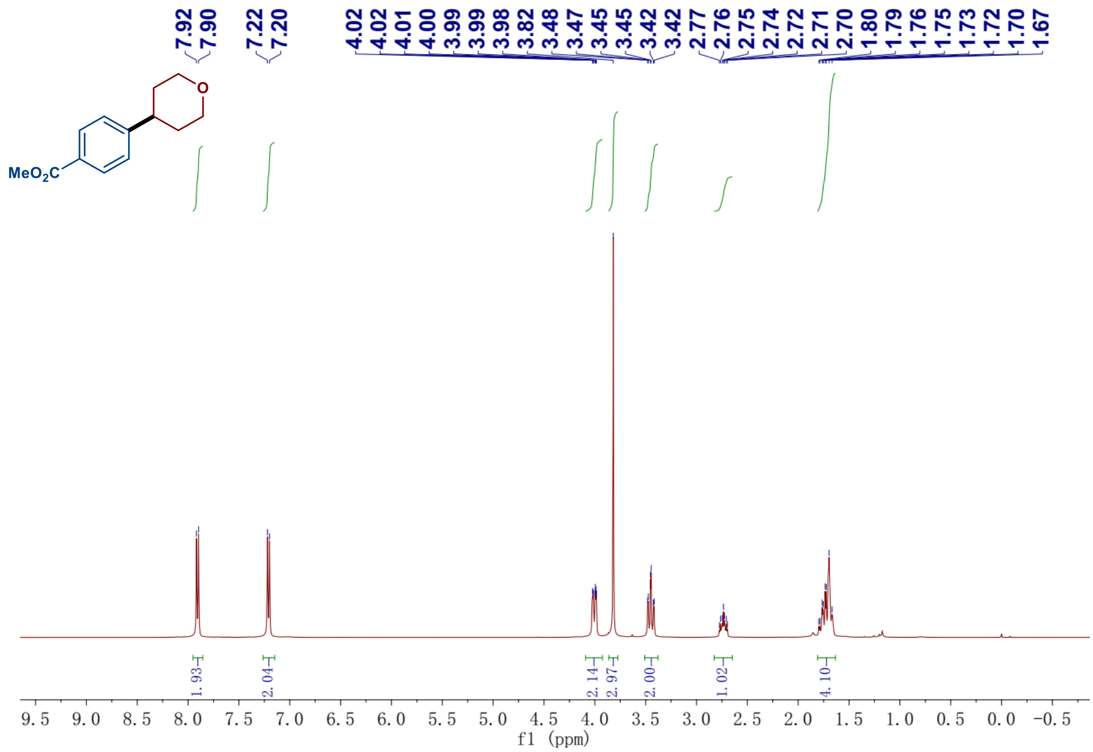


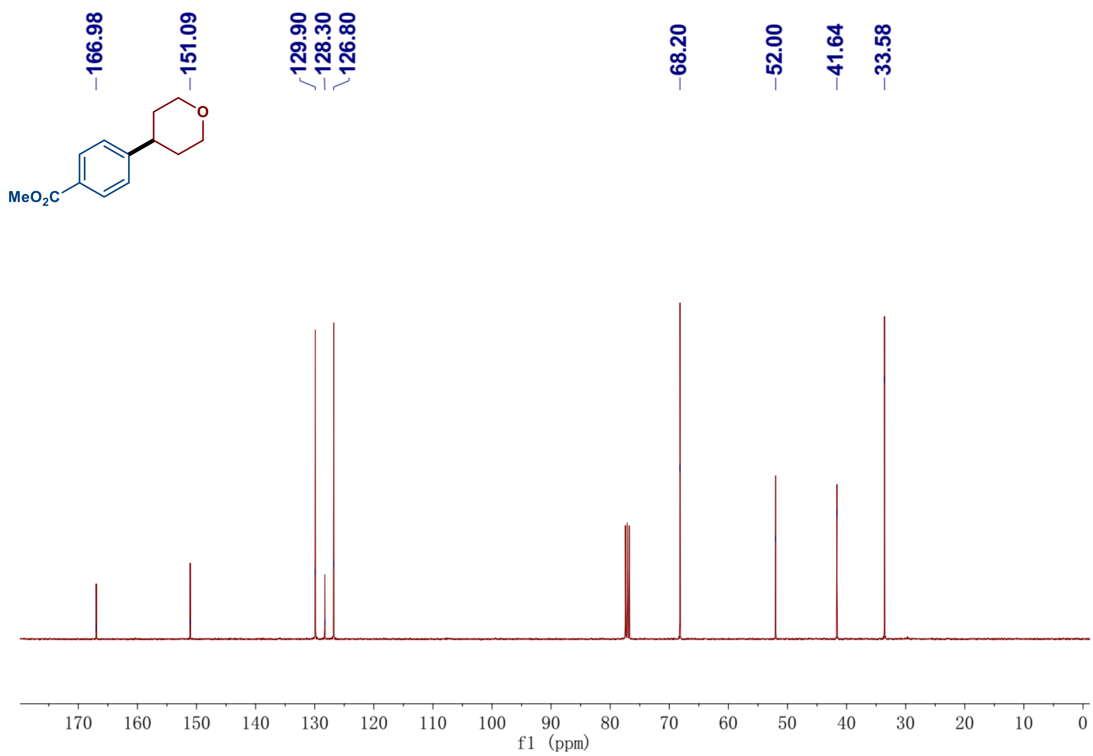
methyl 4-(but-3-en-1-yl)benzoate[8]: Yellow oil (81% yield.). The product was purified by silica gel column chromatography, using petroleum ether/EA = 60/1 (v/v) as an eluent.

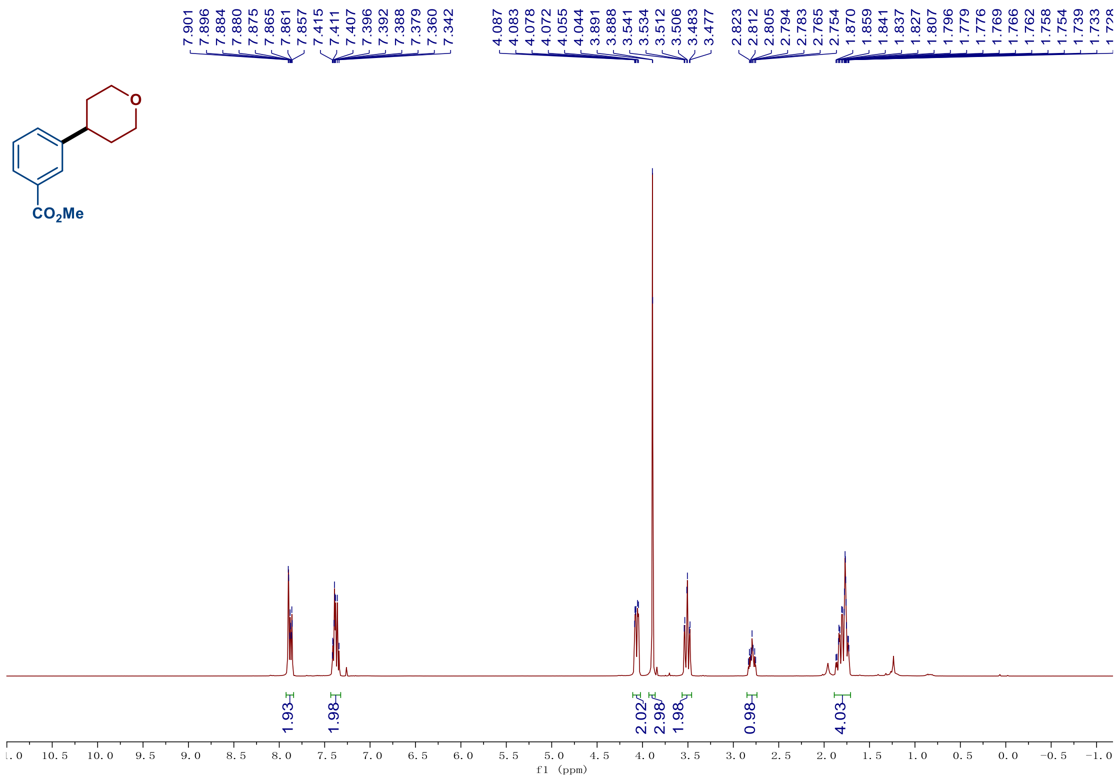
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 7.6 Hz, 2H), 7.16 (d, *J* = 7.6 Hz, 2H), 5.80 - 5.67 (m, 1H), 4.97 -4.88 (m, 2H), 3.81 (s, 3H), 2.68 (t, *J* = 7.6 Hz, 2H), 2.30 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.07, 147.27, 137.40, 129.60, 128.42, 127.80, 115.29, 51.89, 35.30, 34.99.

9. NMR Spectra of Products



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4.072
4.055
4.044
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3.512
3.506
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1.827
1.807
1.796
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1.754
1.739
1.733
O
CO2Me
1.93
1.98
2.02
2.98
1.98
0.98
4.03
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
2.0
1.5
1.0
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0.0
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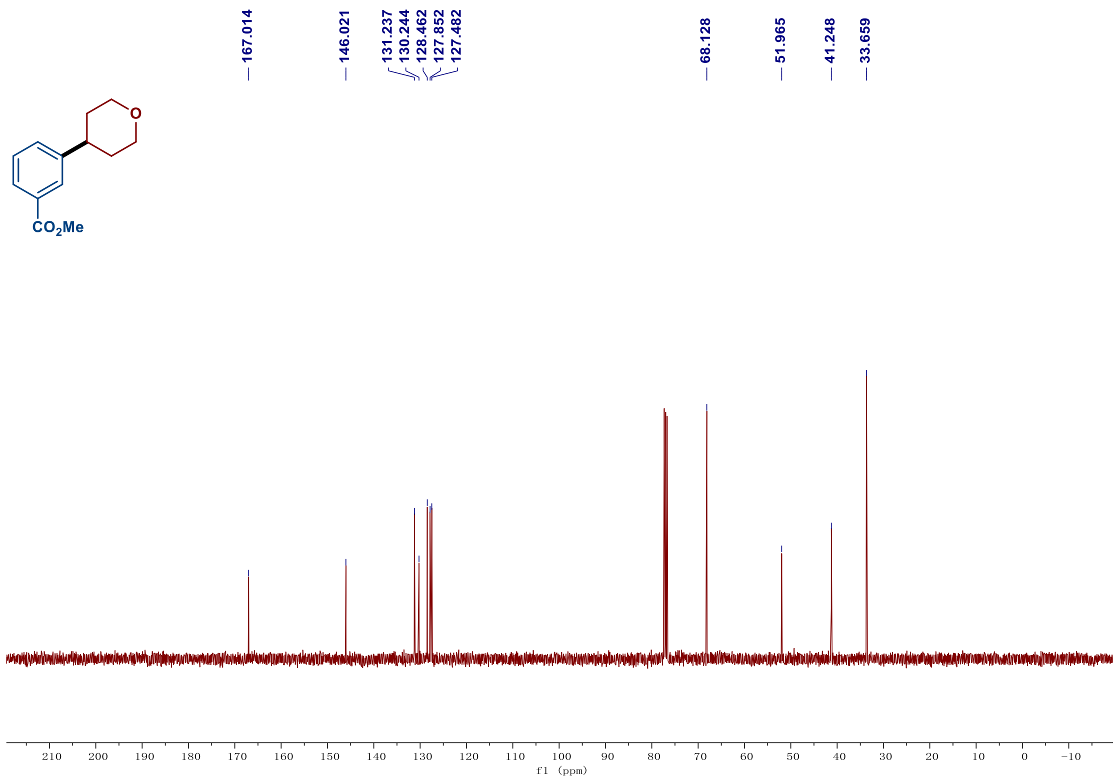
-1.0
f1 (ppm)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CO2Me
210
200
190
180
170
160
150
140
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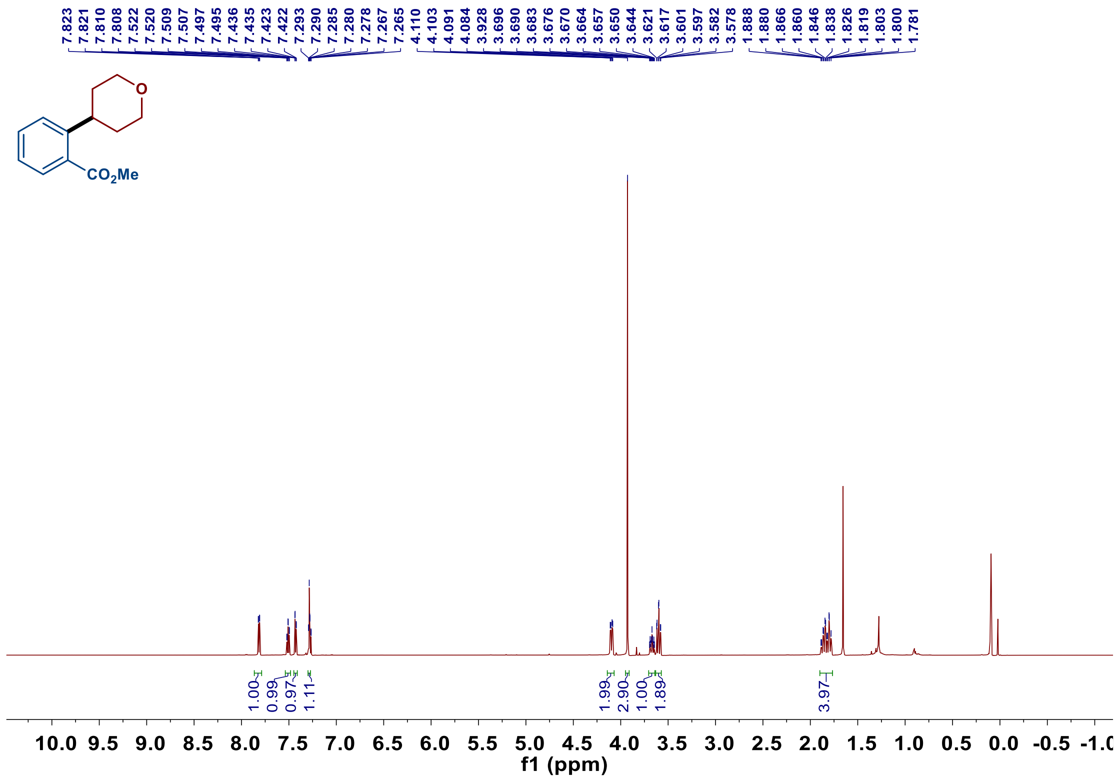
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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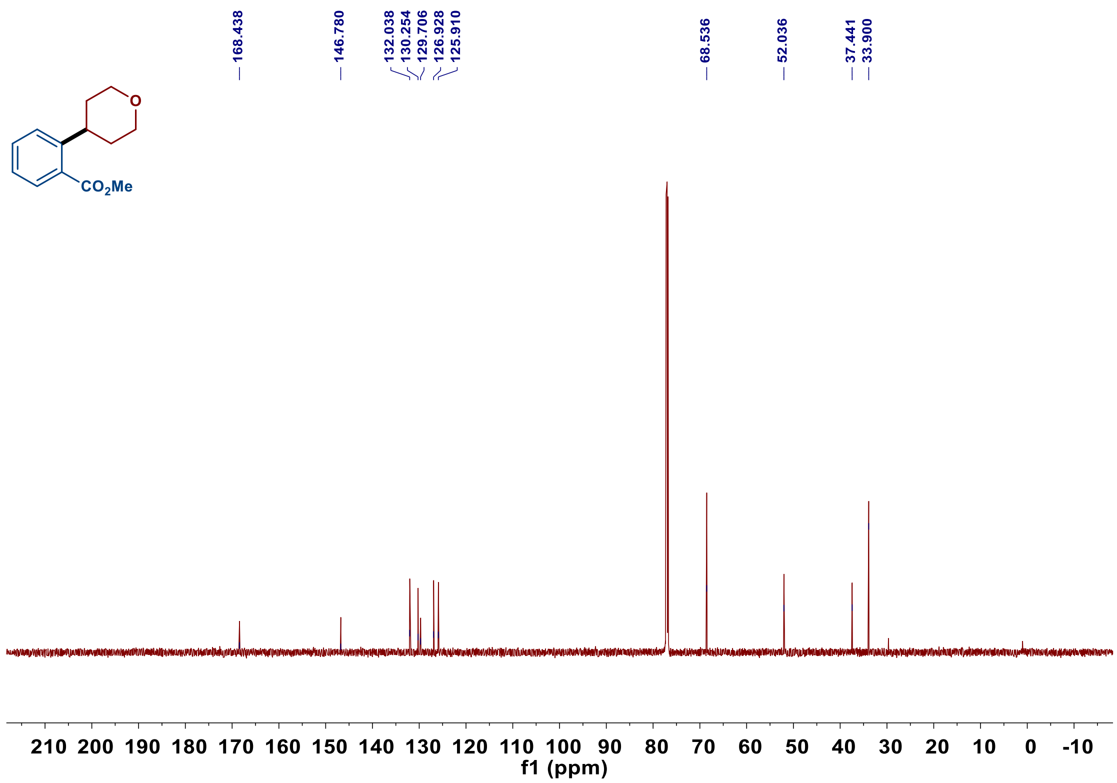
O
CO2Me
1.00 0.99 0.97 1.11
1.99 2.90 1.00 1.89
3.97
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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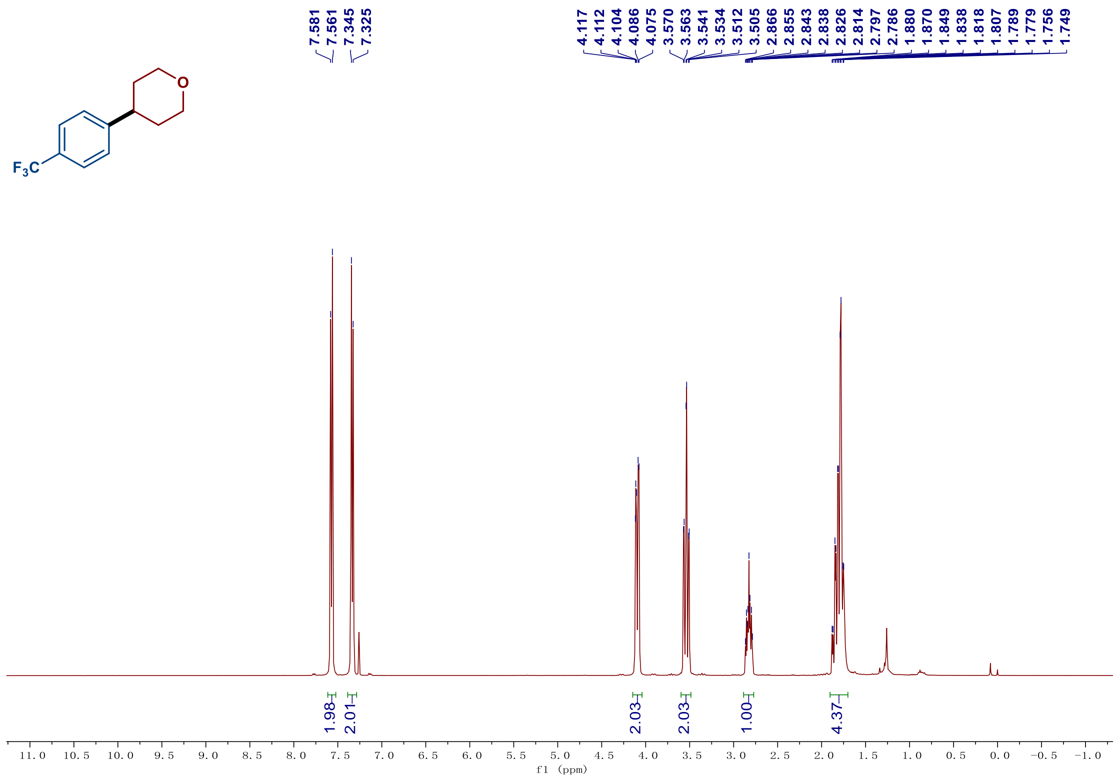
37.441 33.900
O
CO2Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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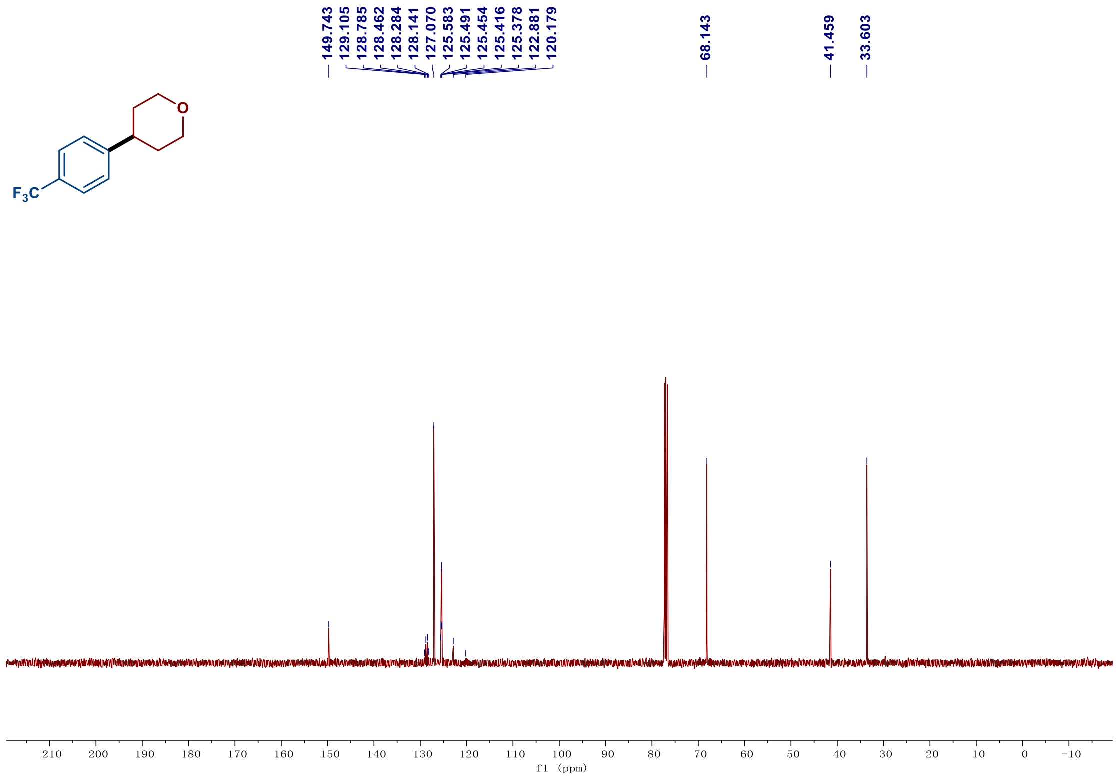
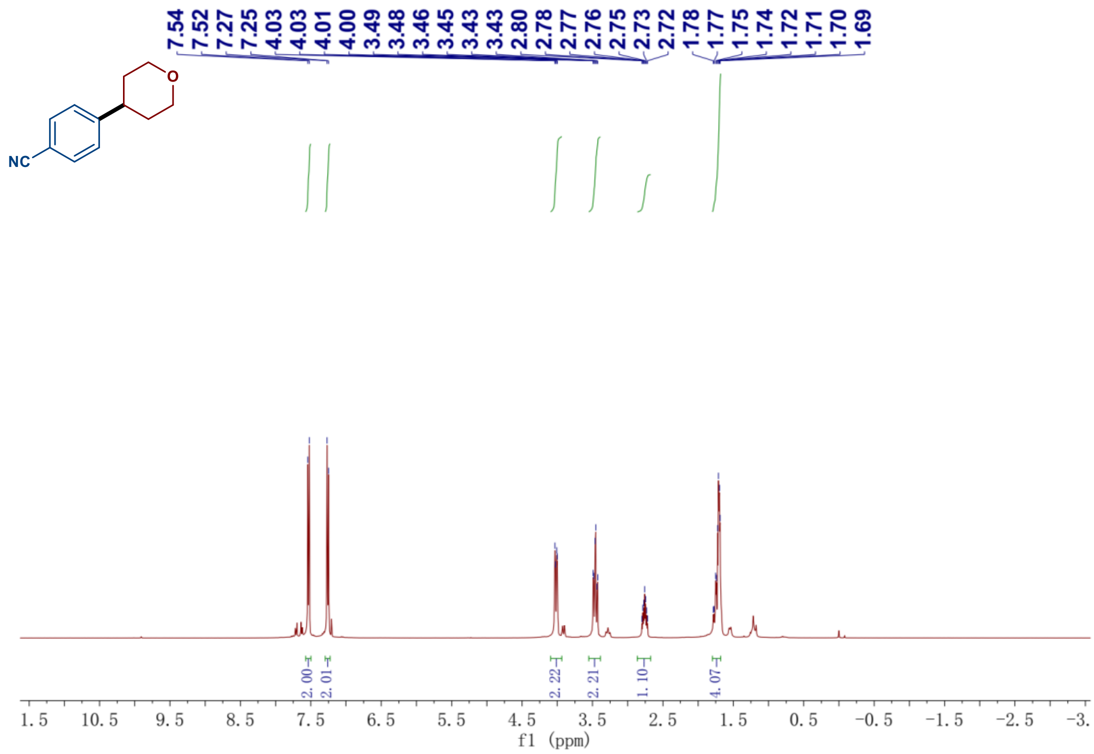
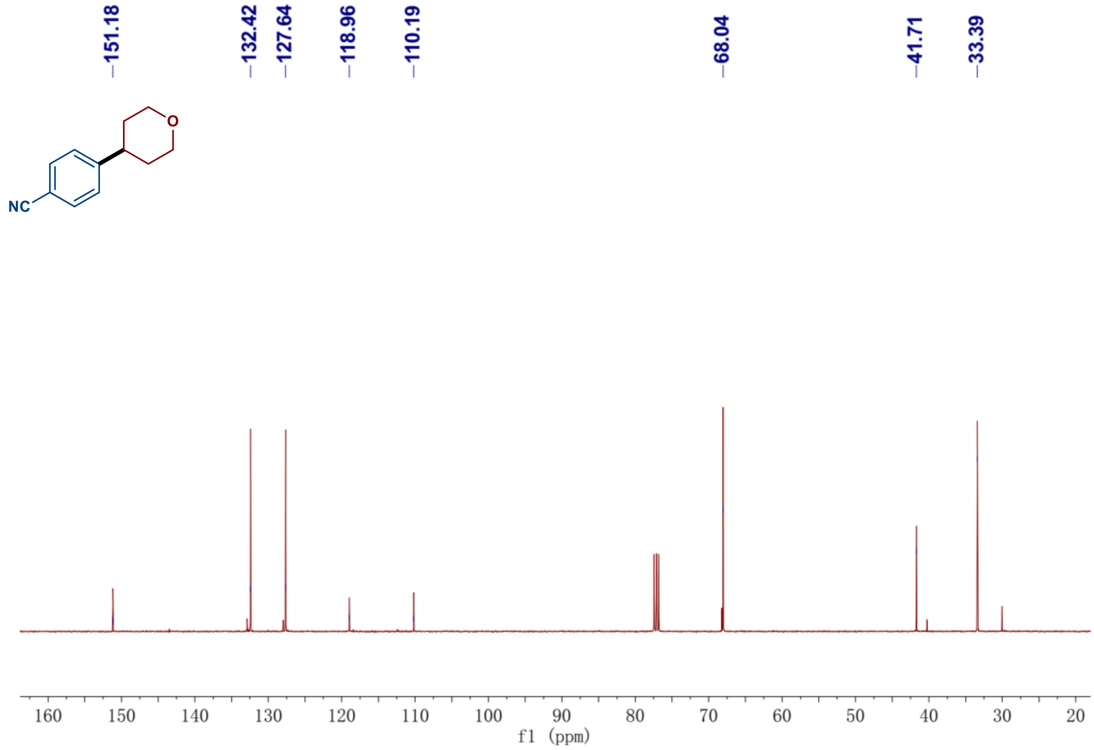
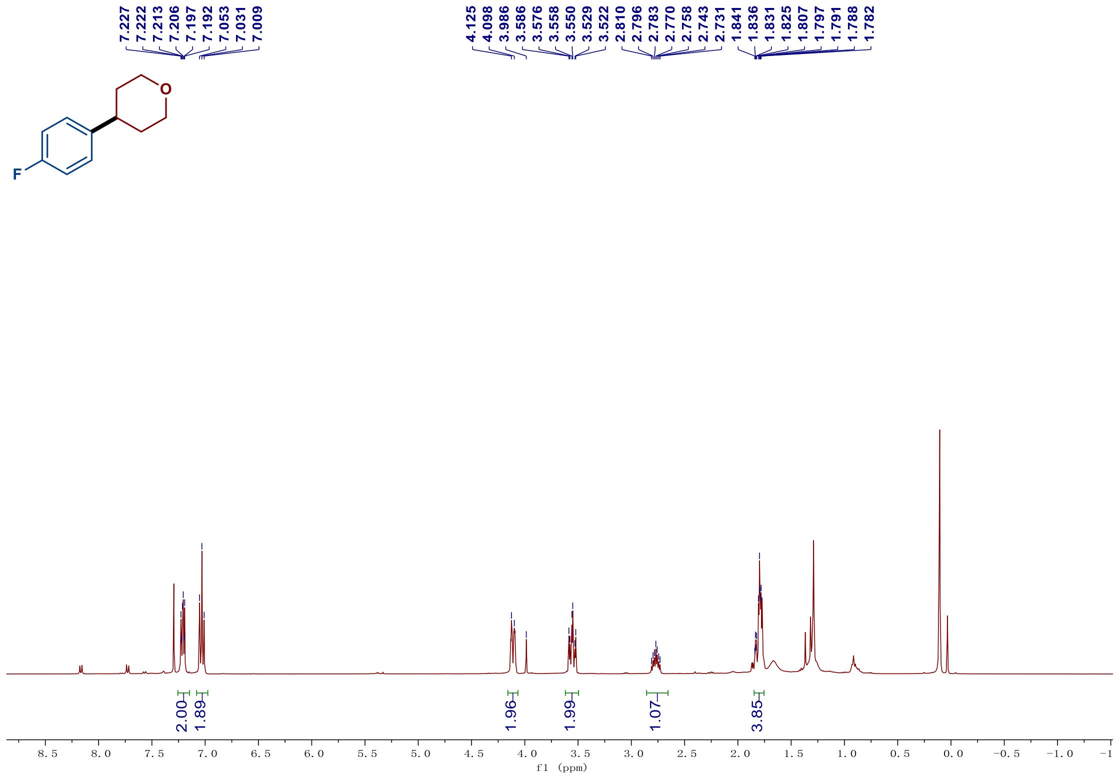
3.563
3.541
3.534
3.512
3.505
2.866
2.855
2.843
2.838
2.826
2.814
2.797
2.786
1.880
1.870
1.849
1.838
1.818
1.807
1.789
1.779
1.756
1.749
F3C
O
1.98
2.01
2.03
2.03
1.00
4.37
f1 (ppm)


149.743
129.105
128.785
128.462
128.284
128.141
127.070
125.583
125.491
125.454
125.416
125.378
122.881
120.179
68.143
41.459
33.603
F3C
O
f1 (ppm)

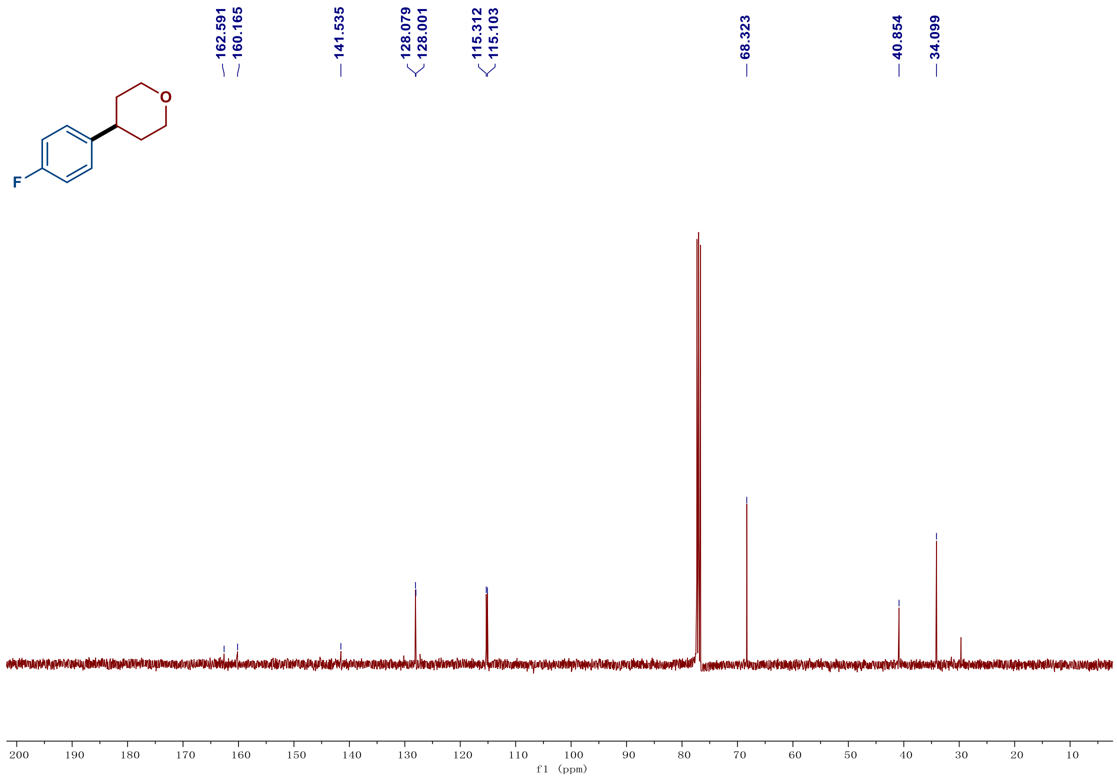


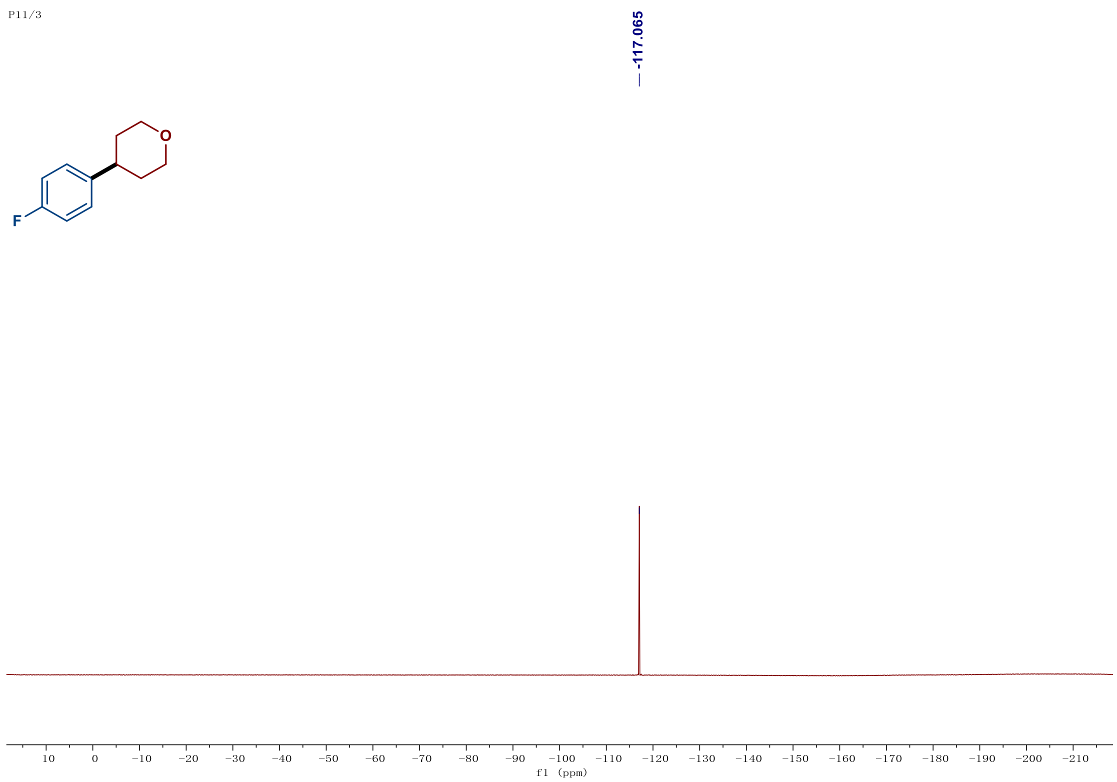


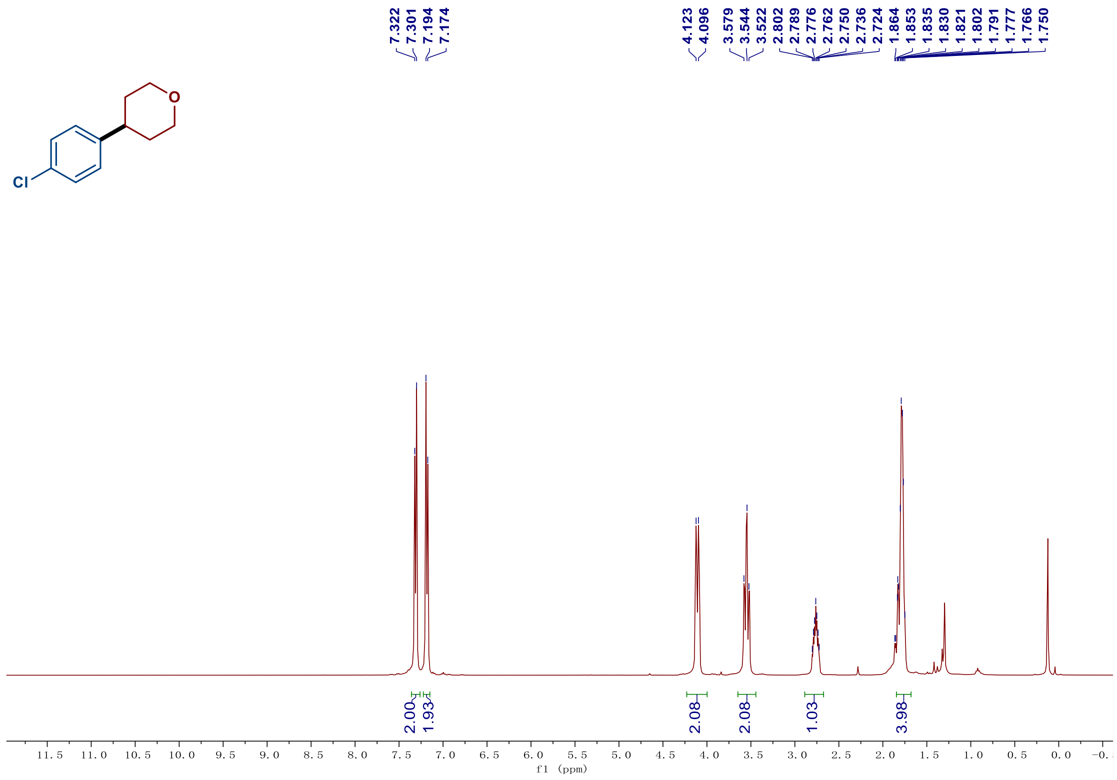
151.18
132.42
127.64
118.96
110.19
68.04
41.71
33.39
O
NC
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

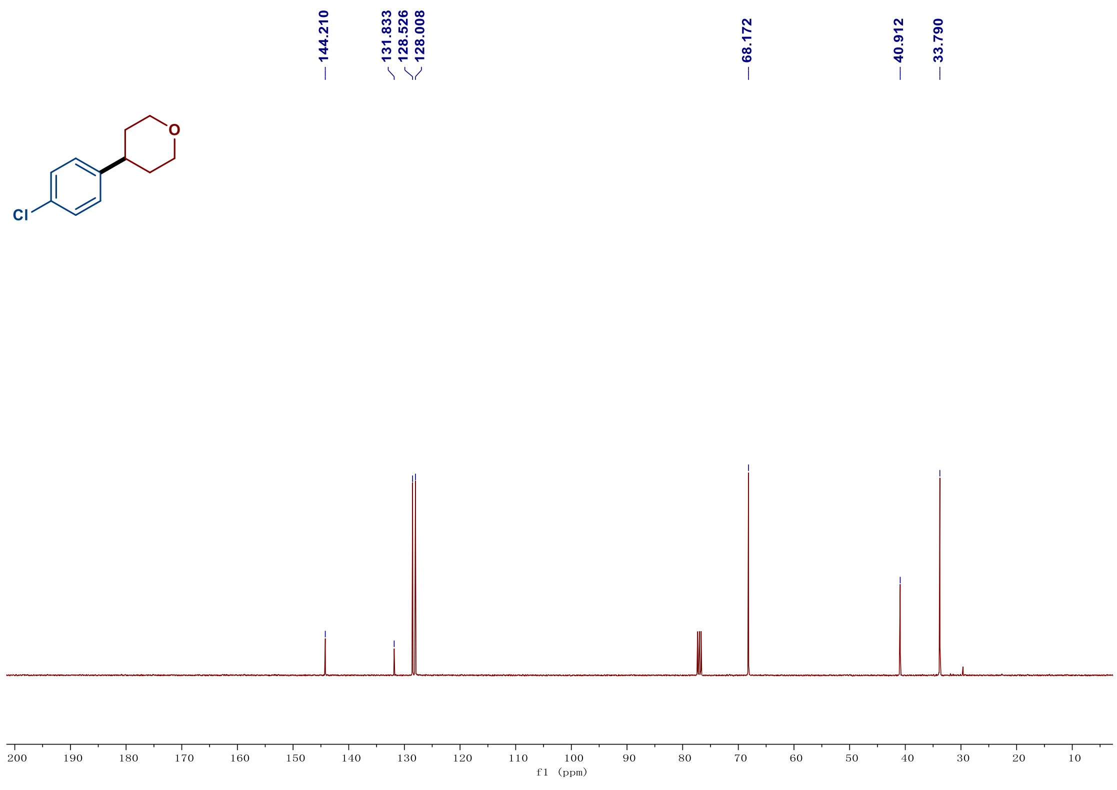


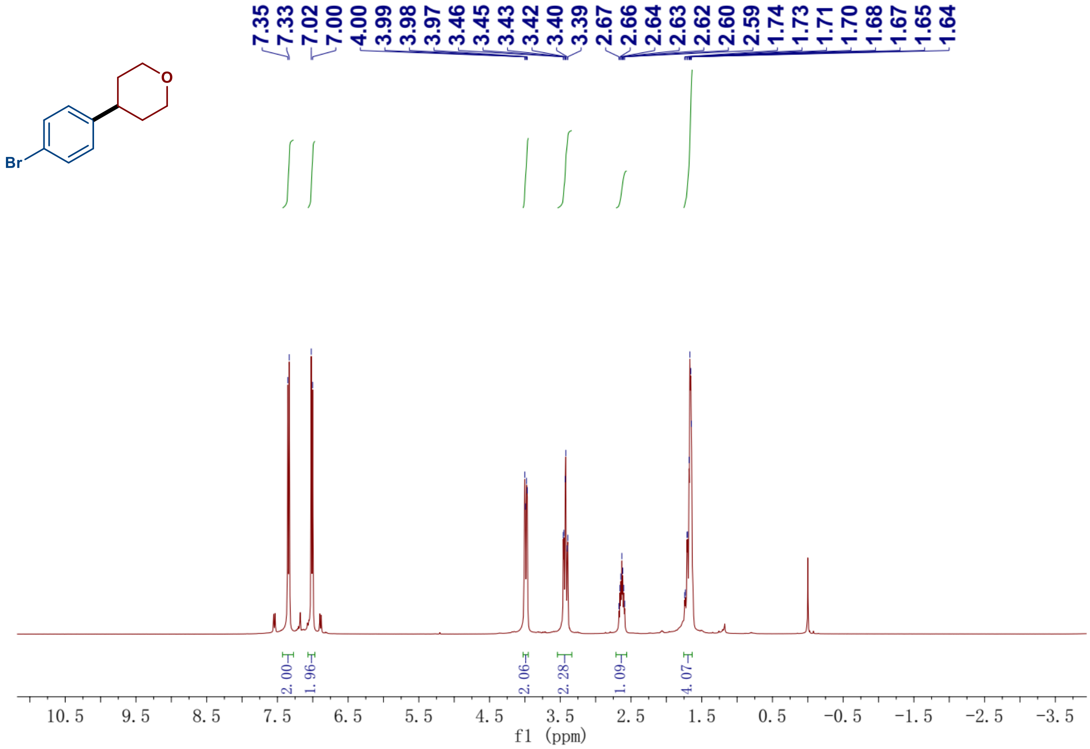
7.227 7.222 7.213 7.206 7.197 7.192 7.053 7.031 7.009
4.125 4.098 3.986 3.586 3.576 3.558 3.550 3.529 3.522 2.810 2.796 2.783 2.770 2.758 2.743 2.731 1.841 1.836 1.831 1.825 1.807 1.797 1.791 1.788 1.782
O
F
2.00
1.89
1.96
1.99
1.07
3.85
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)

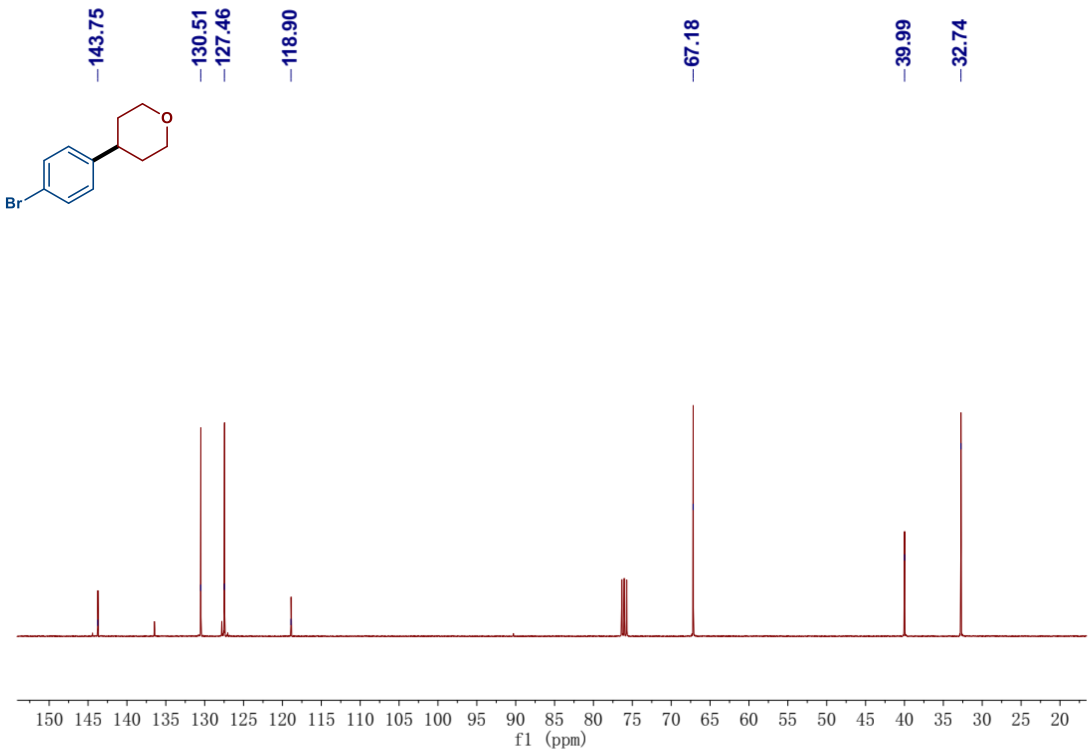

162.591
160.165
141.535
128.079
128.001
115.312
115.103
68.323
40.854
34.099
F
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

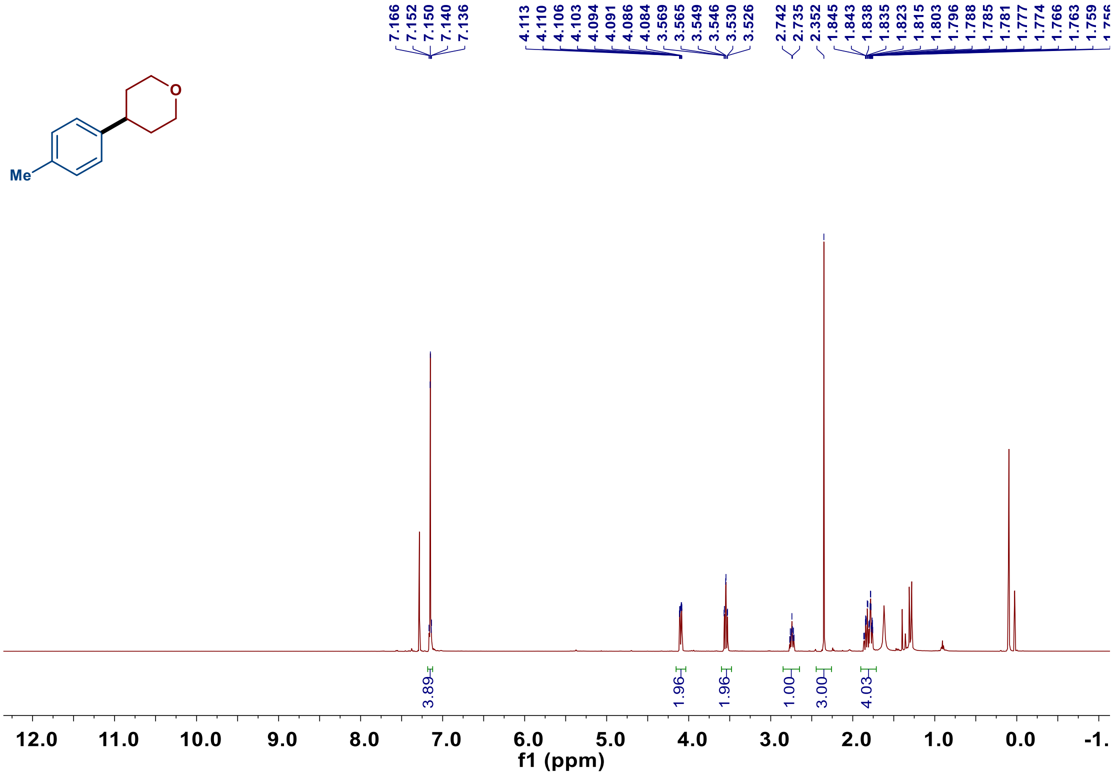

P11/3
-117.065
F
O
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

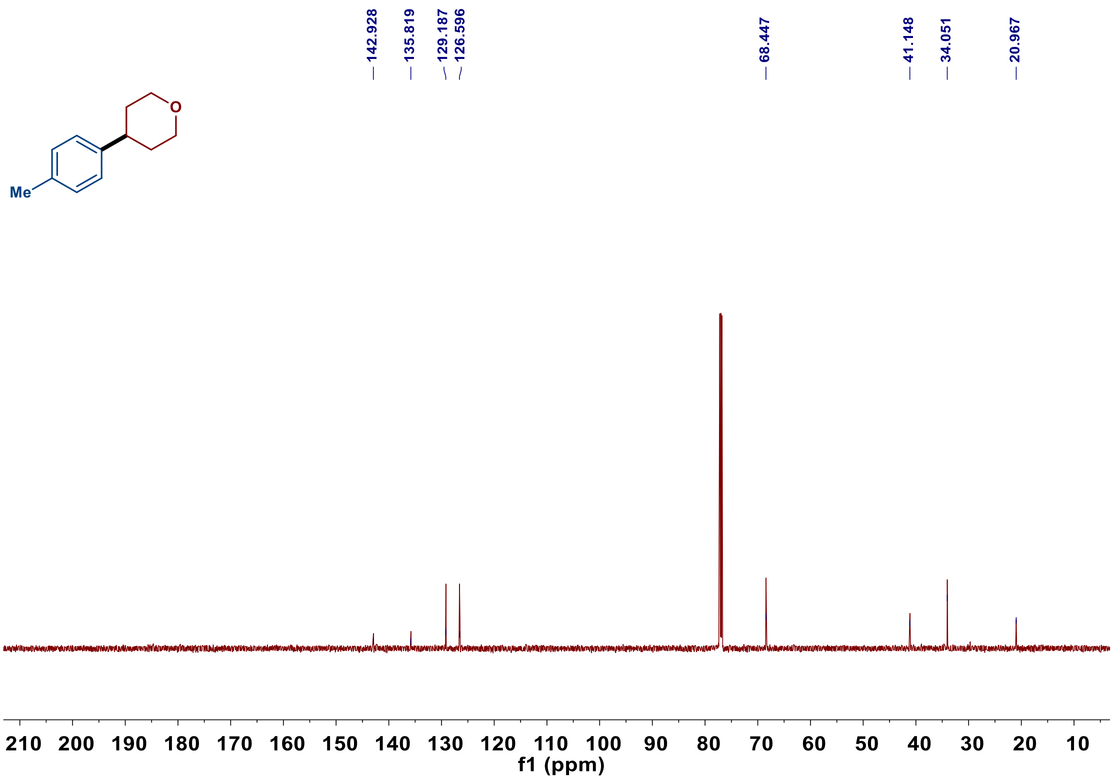


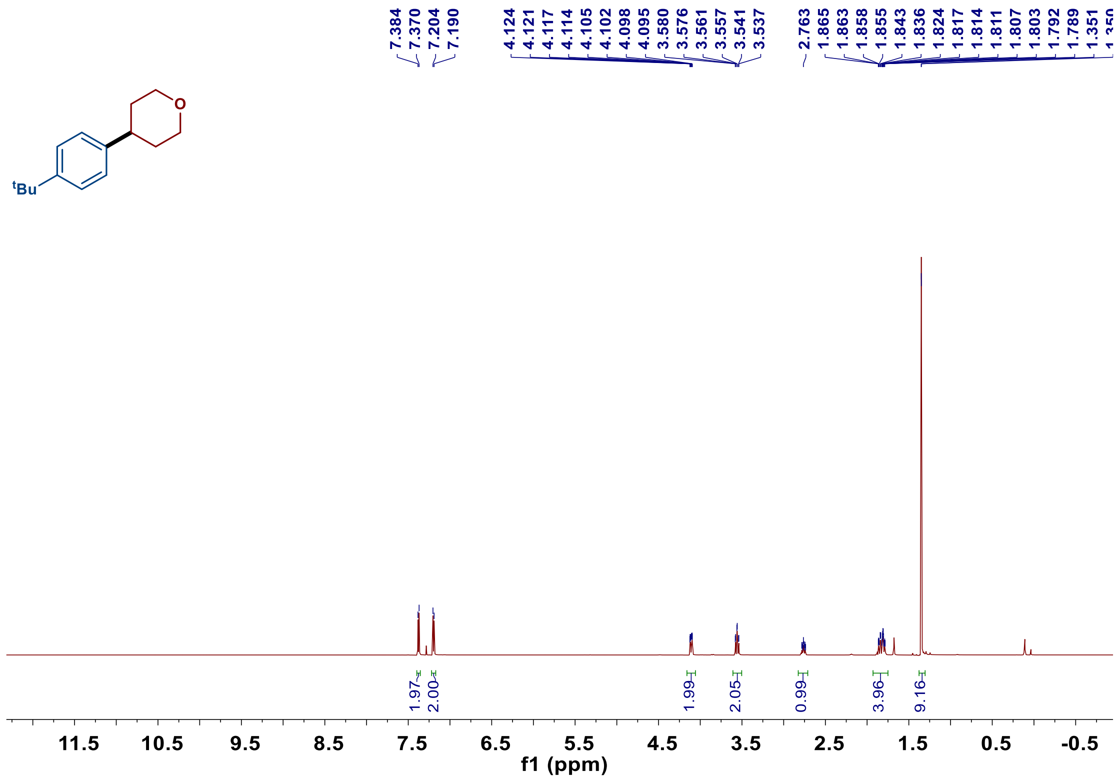


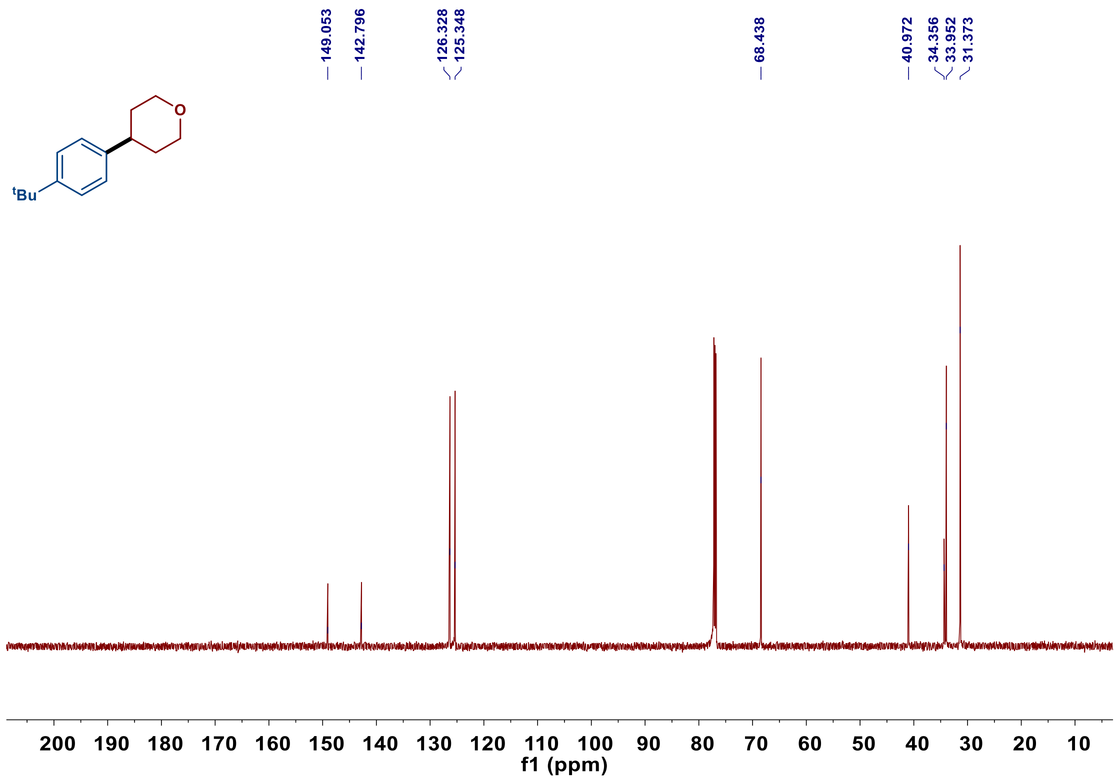

7.35
7.33
7.02
7.00
4.00
3.99
3.98
3.97
3.46
3.45
3.43
3.42
3.40
3.39
2.67
2.66
2.64
2.63
2.62
2.60
2.59
1.74
1.73
1.71
1.70
1.68
1.67
1.65
1.64
O
Br
2.00
1.96
2.06
2.28
1.09
4.07
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5 -2.5 -3.5
f1 (ppm)

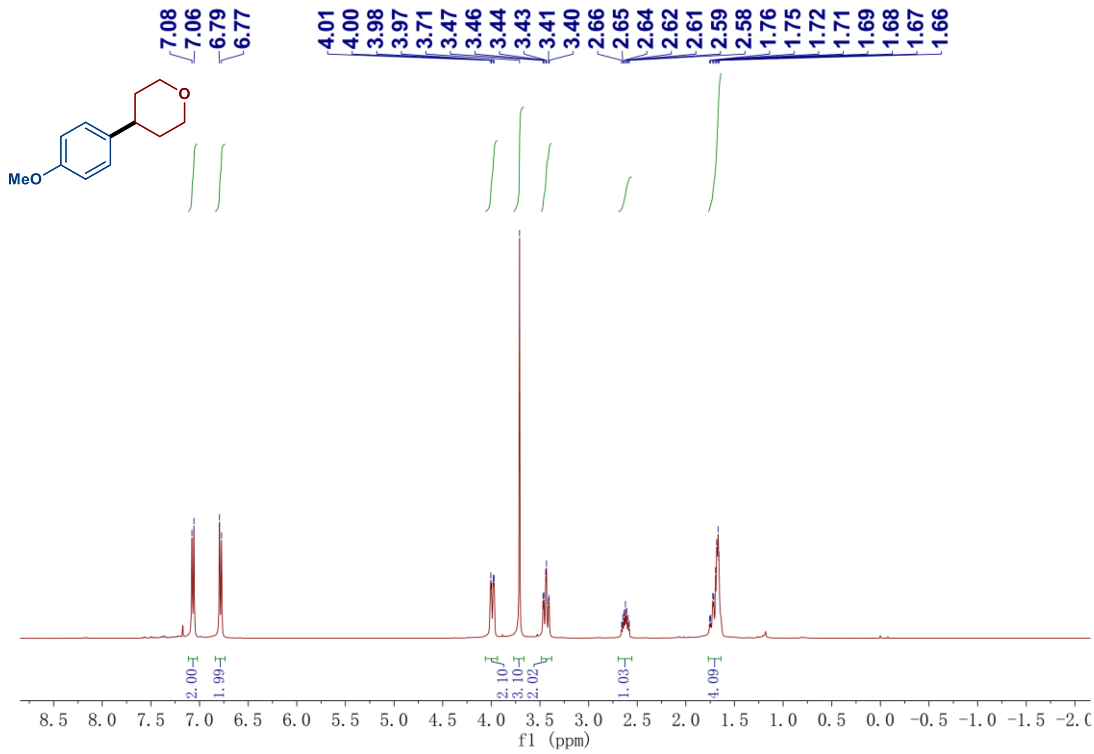

143.75
130.51
127.46
118.90
67.18
39.99
32.74
O
Br
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20
f1 (ppm)

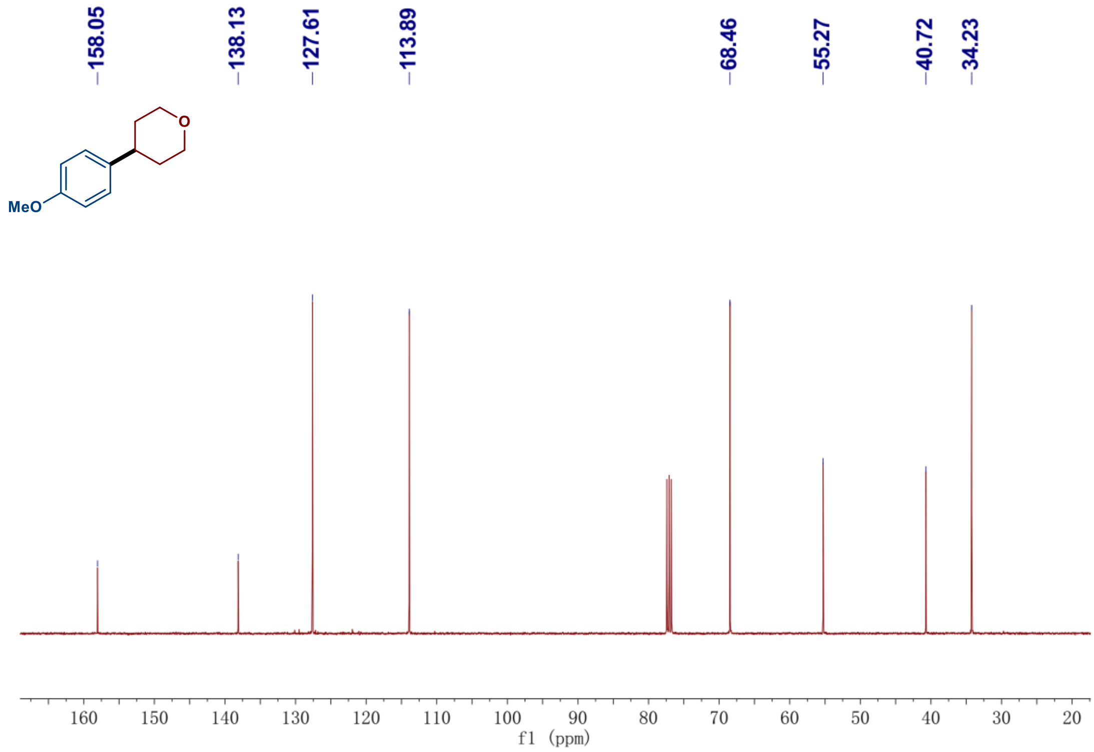

7.166
7.152
7.150
7.140
7.136
4.113
4.110
4.106
4.103
4.094
4.091
4.086
4.084
3.569
3.565
3.549
3.546
3.530
3.526
2.742
2.735
2.352
1.845
1.843
1.838
1.835
1.823
1.815
1.803
1.796
1.788
1.785
1.781
1.777
1.774
1.766
1.763
1.759
O
Me
3.89
1.96
1.96
1.00
3.00
4.03
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.
f1 (ppm)

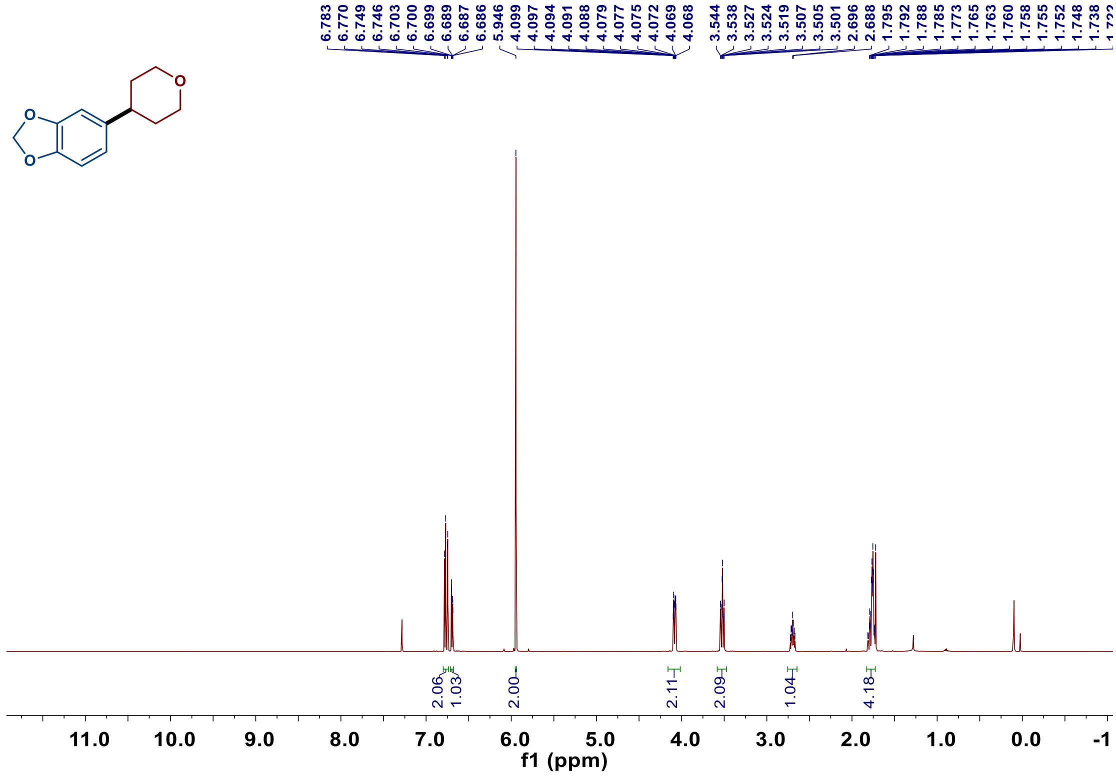

142.928
135.819
129.187
126.596
68.447
41.148
34.051
20.967
O
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


tBu
O
7.384
7.370
7.204
7.190
4.124
4.121
4.117
4.114
4.105
4.102
4.098
4.095
3.580
3.576
3.561
3.557
3.541
3.537
2.763
1.865
1.863
1.858
1.855
1.843
1.836
1.824
1.817
1.814
1.811
1.807
1.803
1.792
1.789
1.351
1.97
2.00
1.99
2.05
0.99
3.96
9.16
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

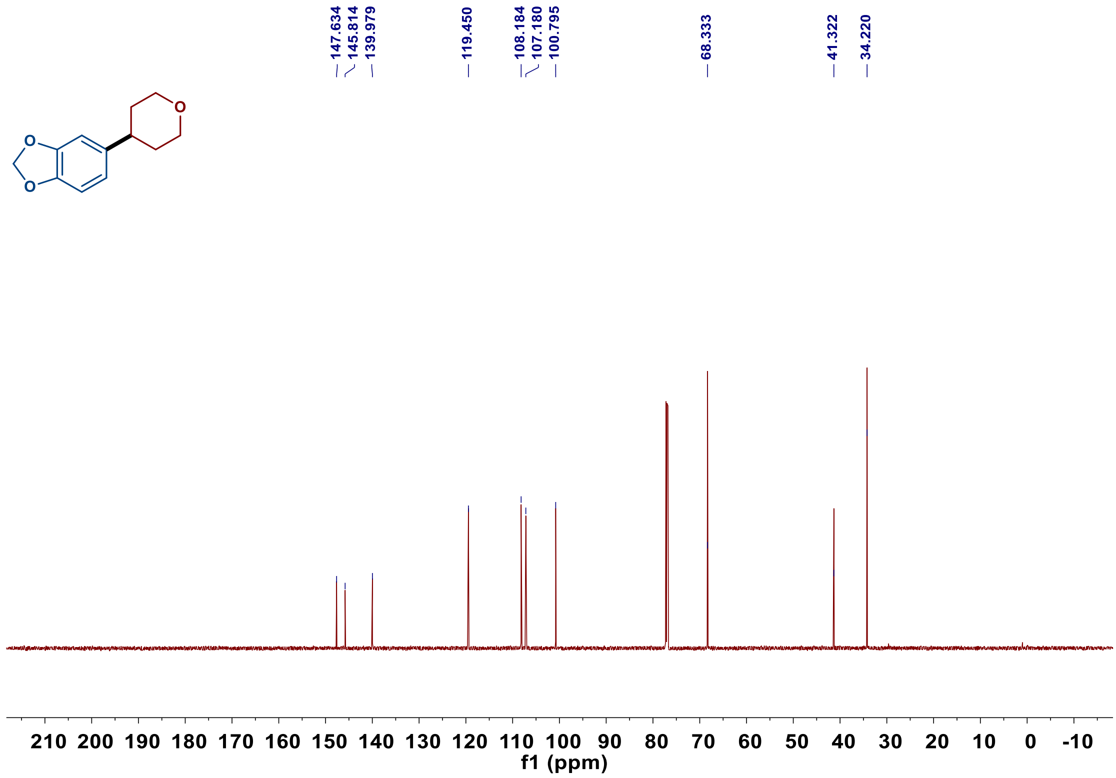

tBu
O
149.053
142.796
126.328
125.348
68.438
40.972
34.356
33.952
31.373
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.08
7.06
6.79
6.77
4.01
4.00
3.98
3.97
3.71
3.47
3.46
3.44
3.43
3.41
3.40
2.66
2.65
2.64
2.62
2.61
2.59
2.58
1.76
1.75
1.72
1.71
1.69
1.68
1.67
1.66
MeO
O
2.00
1.99
2.10
3.10
2.02
1.03
4.09
f1 (ppm)

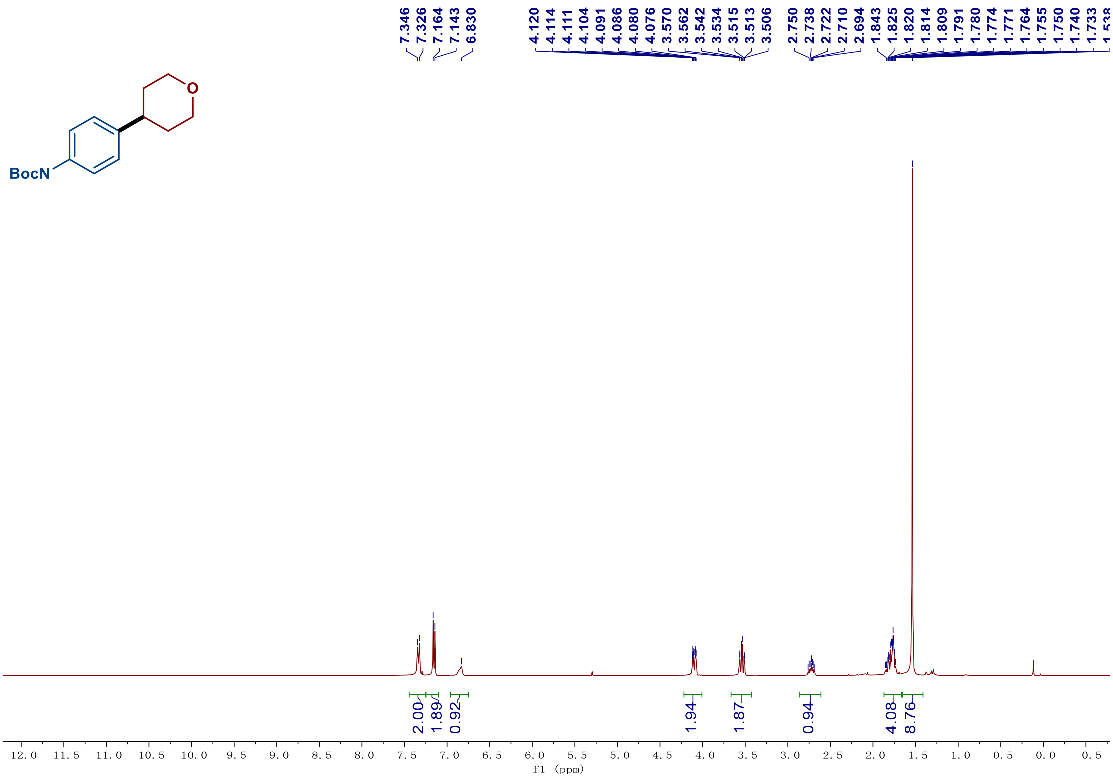

158.05
138.13
127.61
113.89
68.46
55.27
40.72
34.23
MeO
O
f1 (ppm)



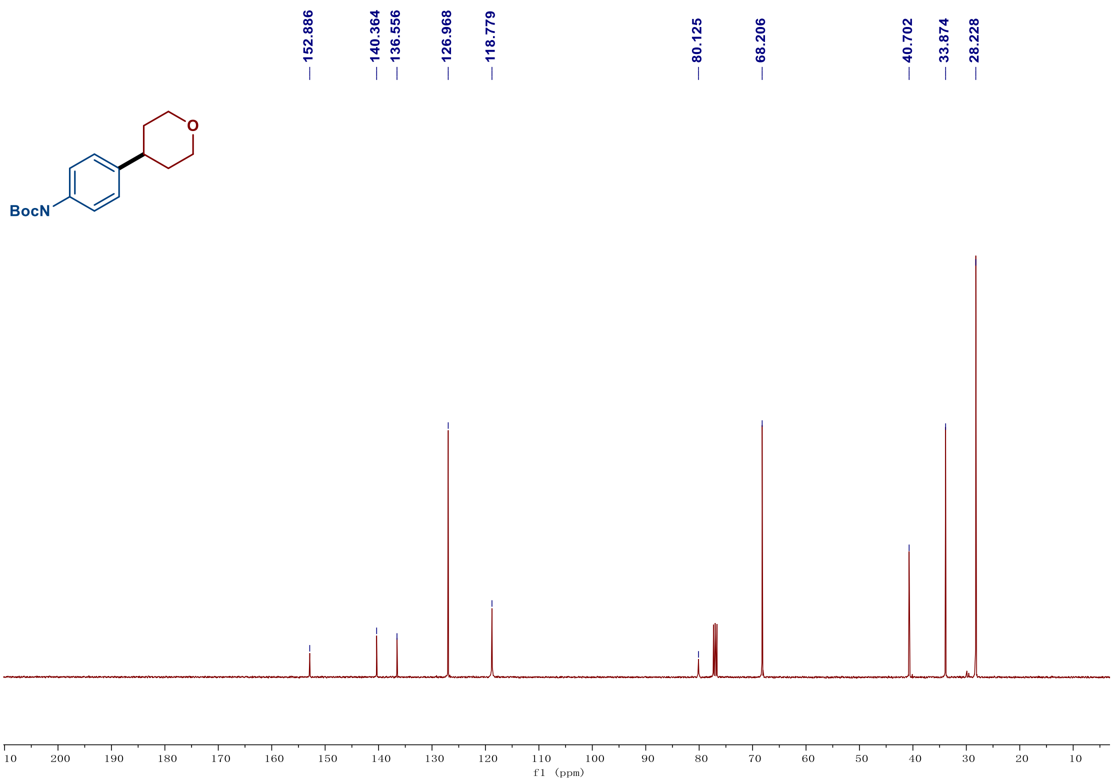
6.783
6.770
6.749
6.746
6.703
6.700
6.699
6.689
6.687
6.686
5.946
4.099
4.097
4.094
4.091
4.088
4.079
4.077
4.075
4.072
4.069
4.068
3.544
3.538
3.527
3.524
3.519
3.507
3.505
3.501
2.696
2.688
1.795
1.792
1.788
1.785
1.773
1.765
1.763
1.760
1.758
1.755
1.752
1.748
1.738
O
O
O
2.06
1.03
2.00
2.11
2.09
1.04
4.18
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1
f1 (ppm)



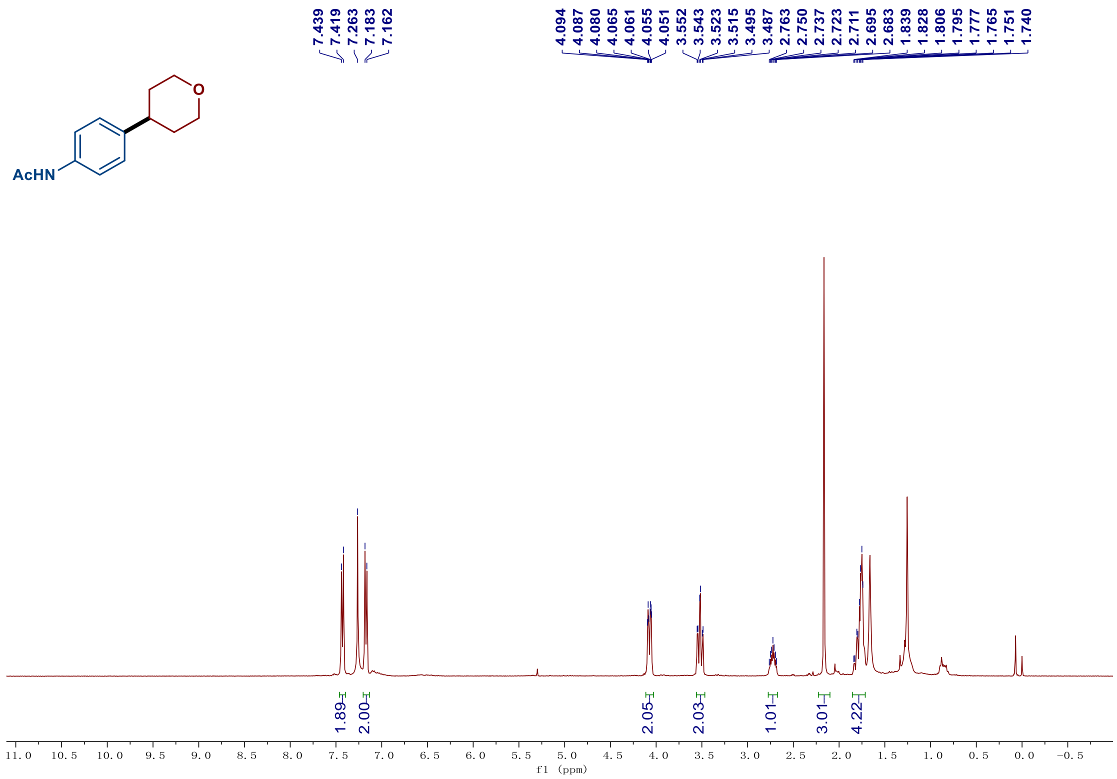
147.634
145.814
139.979
119.450
108.184
107.180
100.795
68.333
41.322
34.220
O
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

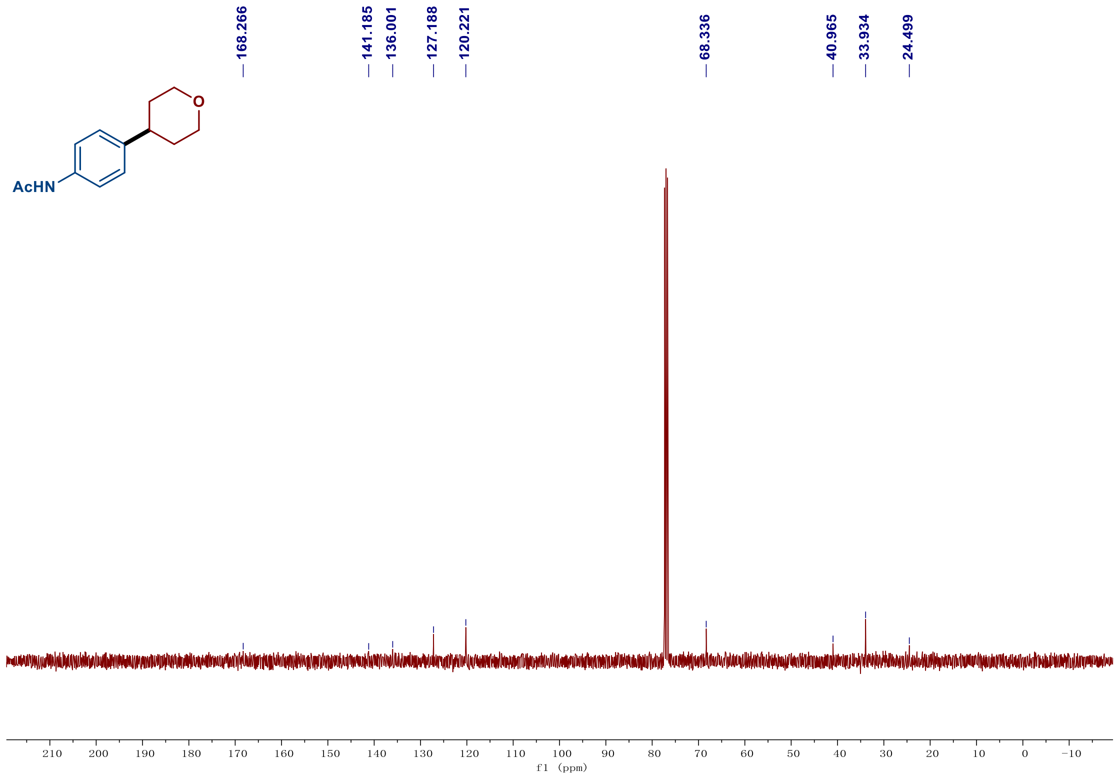


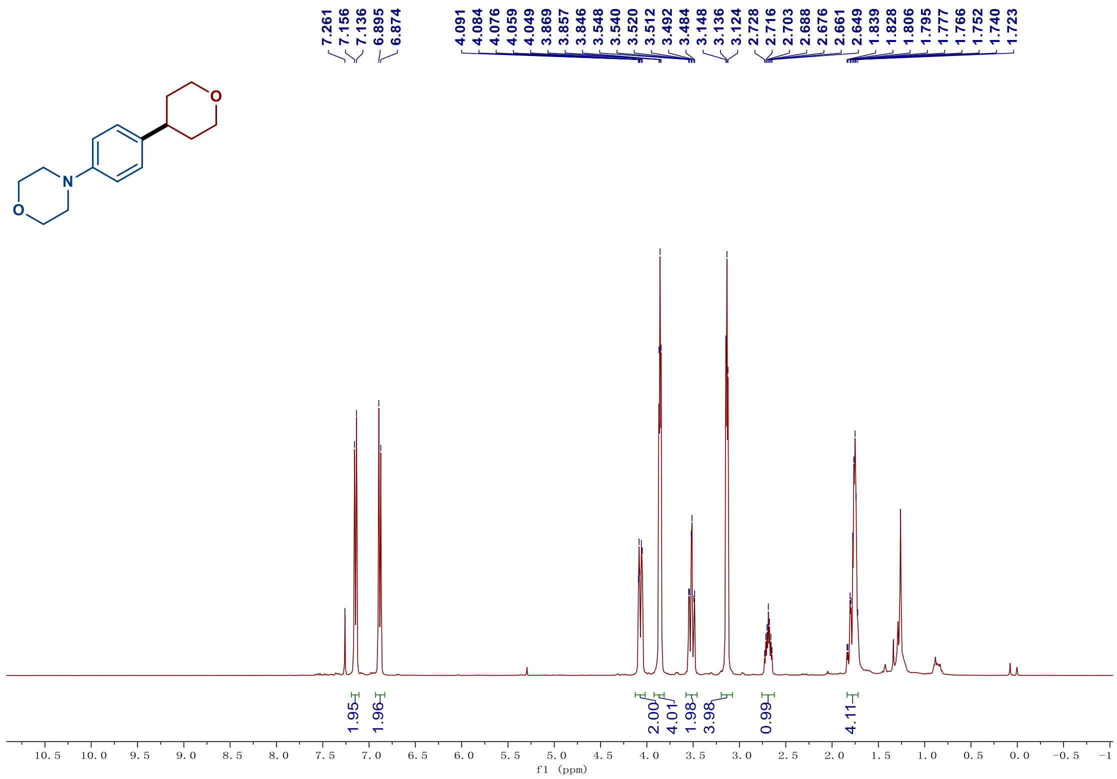
BocN
O
7.346
7.326
7.164
7.143
6.830
4.120
4.114
4.111
4.104
4.091
4.086
4.080
4.076
3.570
3.562
3.542
3.534
3.515
3.513
3.506
2.750
2.738
2.722
2.710
2.694
1.843
1.825
1.820
1.814
1.809
1.791
1.780
1.774
1.771
1.764
1.755
1.750
1.740
1.733
2.00
1.89
0.92
1.94
1.87
0.94
4.08
8.76
f1 (ppm)

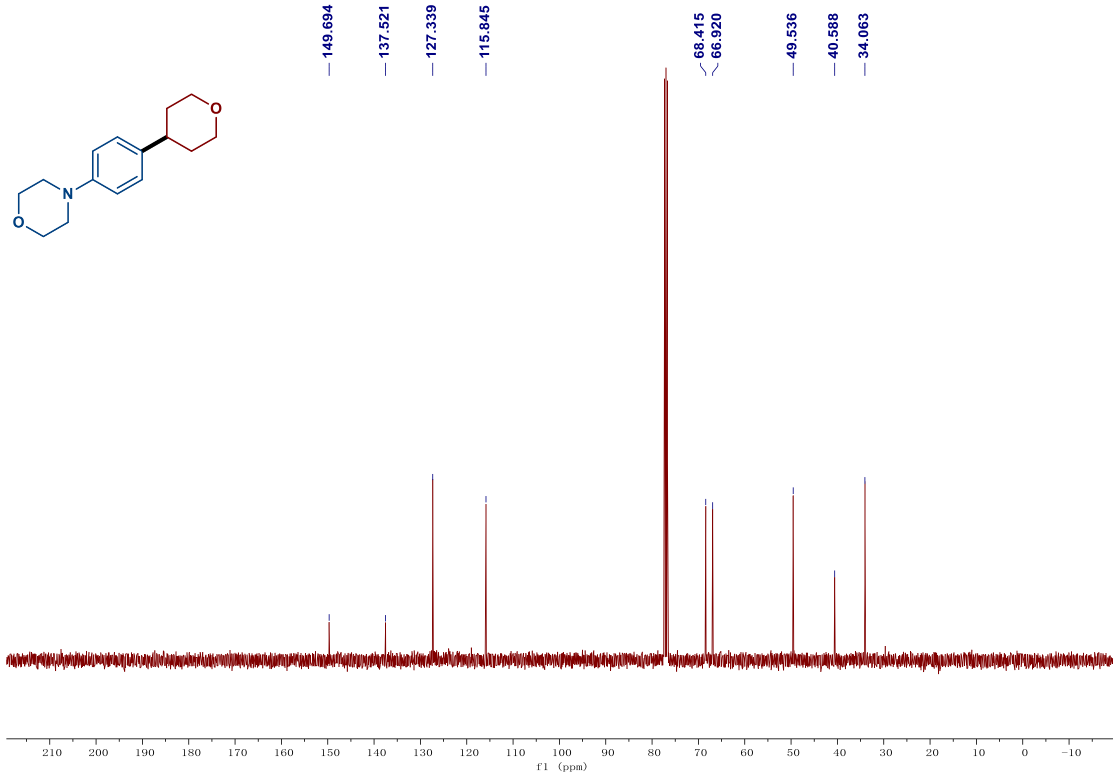


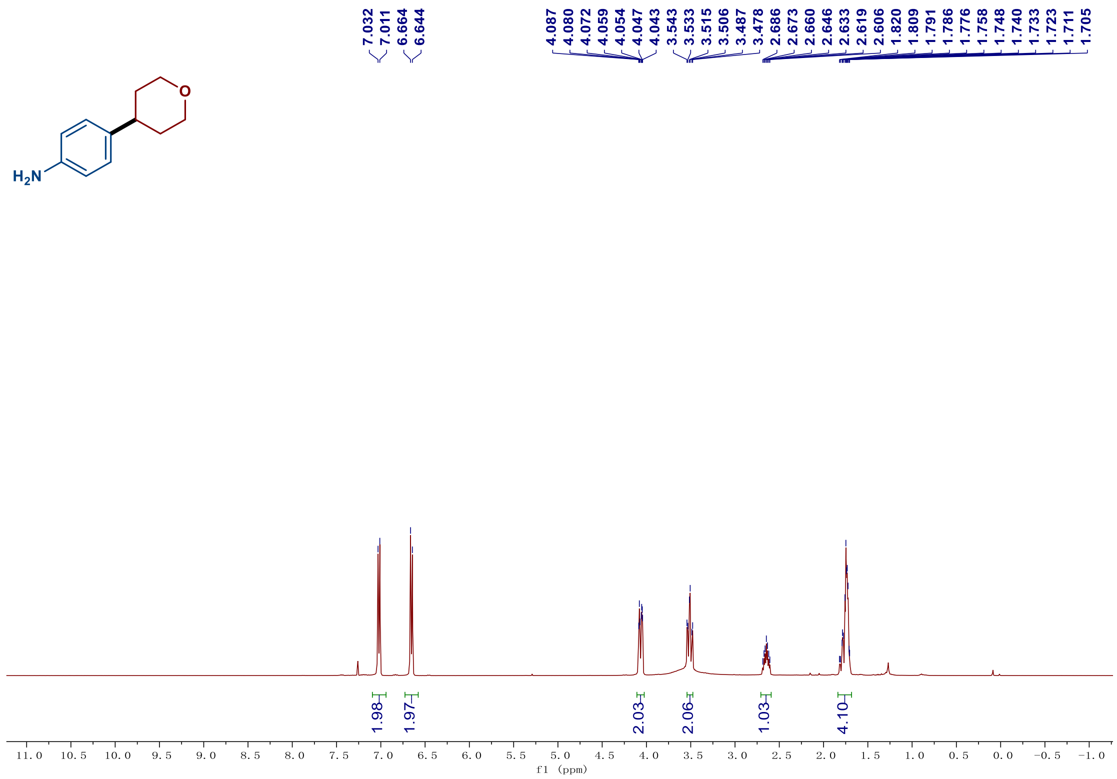
BocN
O
152.886
140.364
136.556
126.968
118.779
80.125
68.206
40.702
33.874
28.228
f1 (ppm)

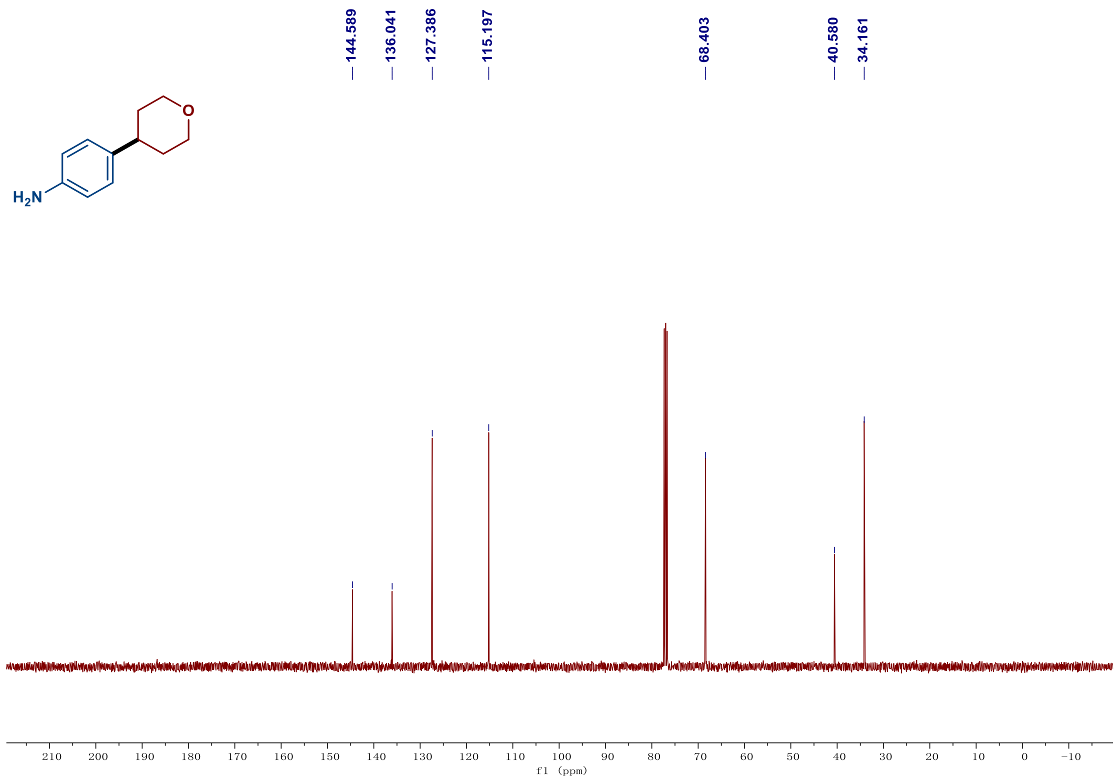

7.439
7.419
7.263
7.183
7.162
4.094
4.087
4.080
4.065
4.061
4.055
4.051
3.552
3.543
3.523
3.515
3.495
3.487
2.763
2.750
2.737
2.723
2.711
2.695
2.683
1.839
1.828
1.806
1.795
1.777
1.765
1.751
1.740
AcHN
O
1.89
2.00
2.05
2.03
1.01
3.01
4.22
f1 (ppm)

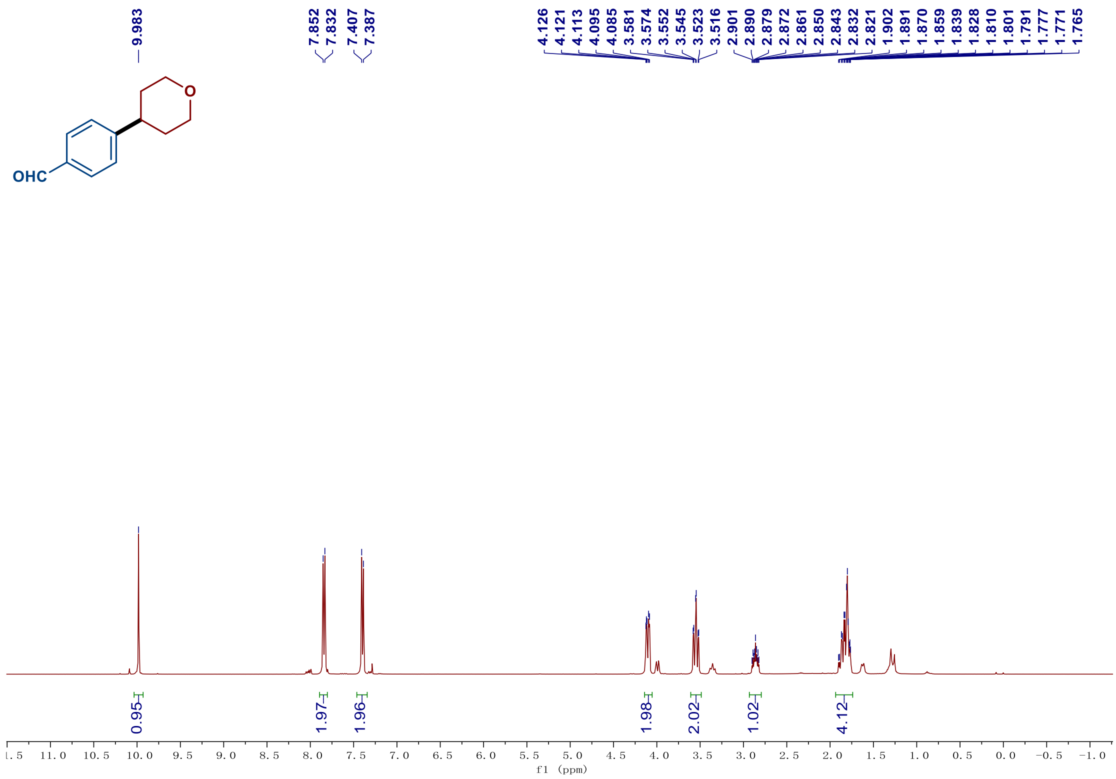

168.266
141.185
136.001
127.188
120.221
68.336
40.965
33.934
24.499
AcHN
O
f1 (ppm)

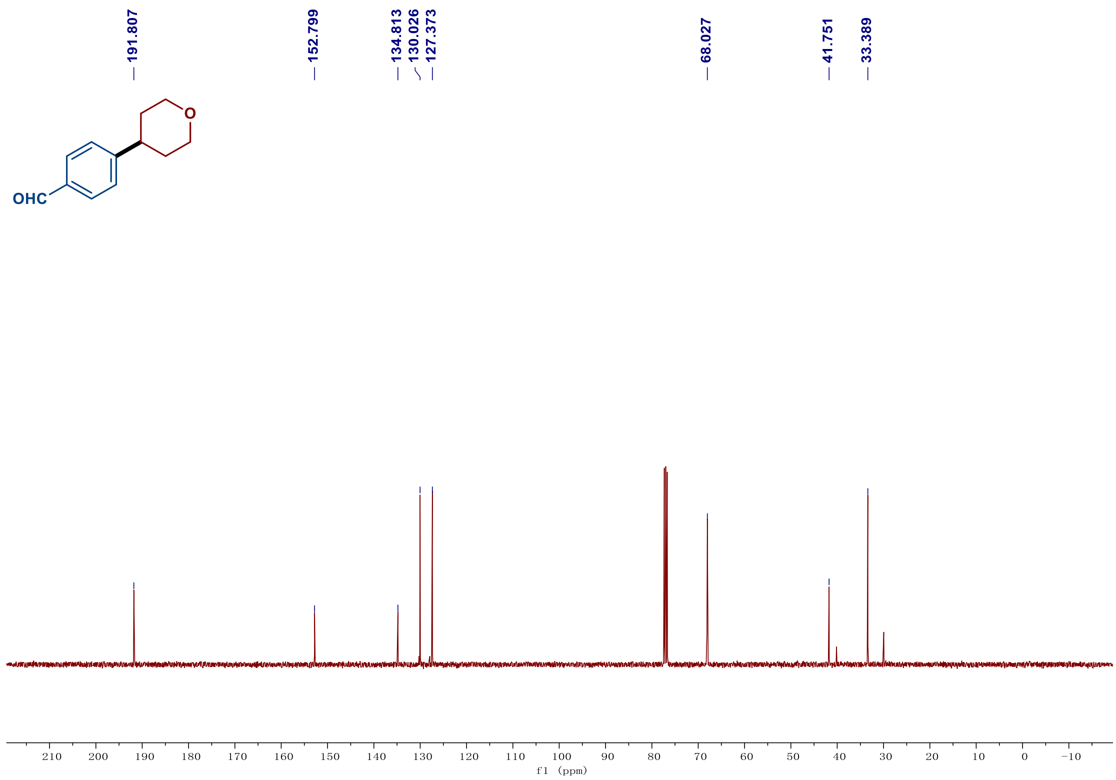

7.261
7.156
7.136
6.895
6.874
4.091
4.084
4.076
4.059
4.049
3.869
3.857
3.846
3.548
3.540
3.520
3.512
3.492
3.484
3.148
3.136
3.124
2.728
2.716
2.703
2.688
2.676
2.661
2.649
1.839
1.828
1.806
1.795
1.777
1.766
1.752
1.740
1.723
O
N
O
1.95
1.96
2.00
4.01
1.98
3.98
0.99
4.11
f1 (ppm)

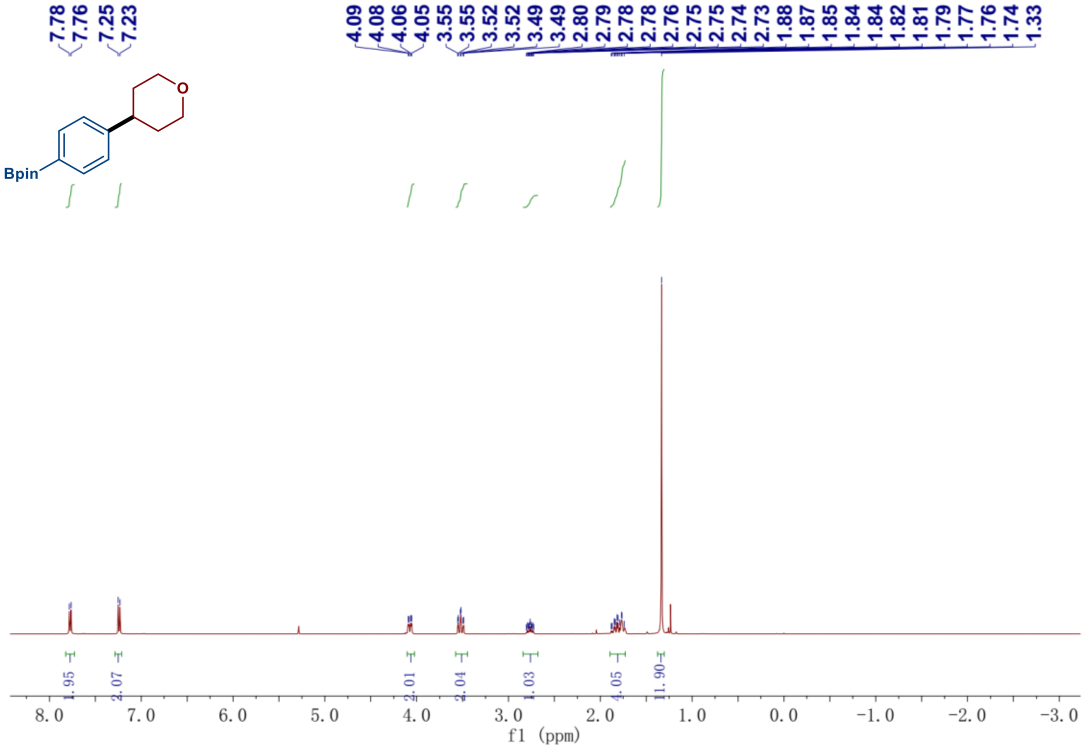

149.694
137.521
127.339
115.845
68.415
66.920
49.536
40.588
34.063
O
N
O
f1 (ppm)

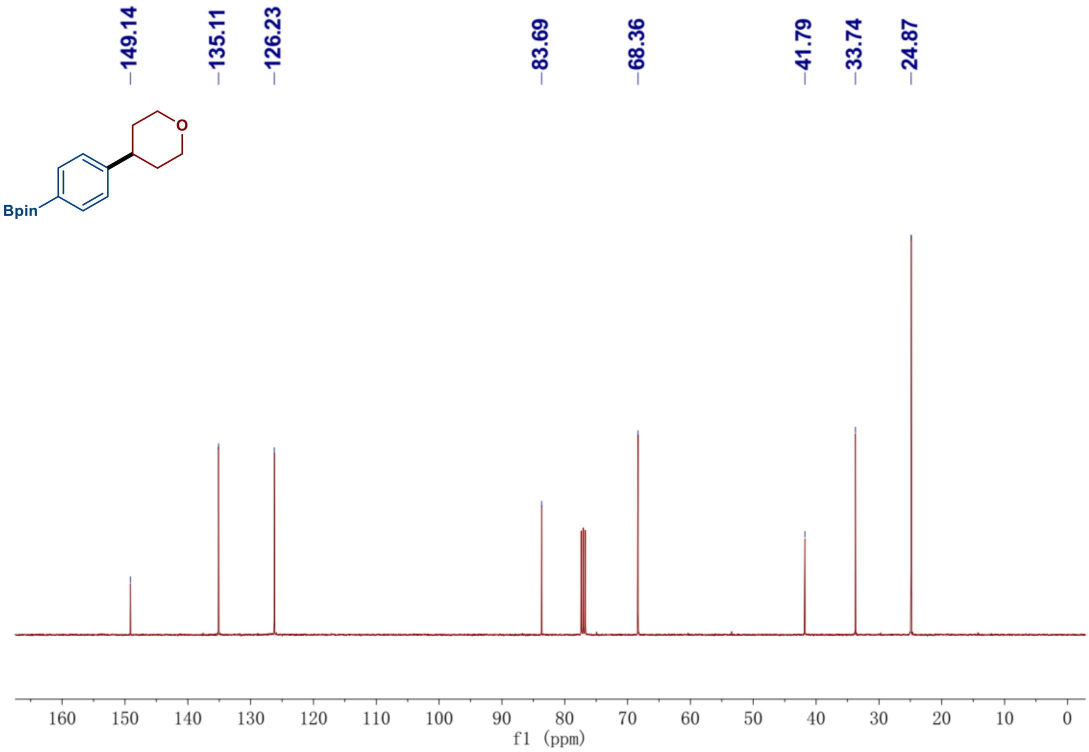

H2N
O
7.032
7.011
6.664
6.644
4.087
4.080
4.072
4.059
4.054
4.047
4.043
3.543
3.533
3.515
3.506
3.487
3.478
2.686
2.673
2.660
2.646
2.633
2.619
2.606
1.820
1.809
1.791
1.786
1.776
1.758
1.748
1.740
1.733
1.723
1.711
1.705
1.98
1.97
2.03
2.06
1.03
4.10
f1 (ppm)

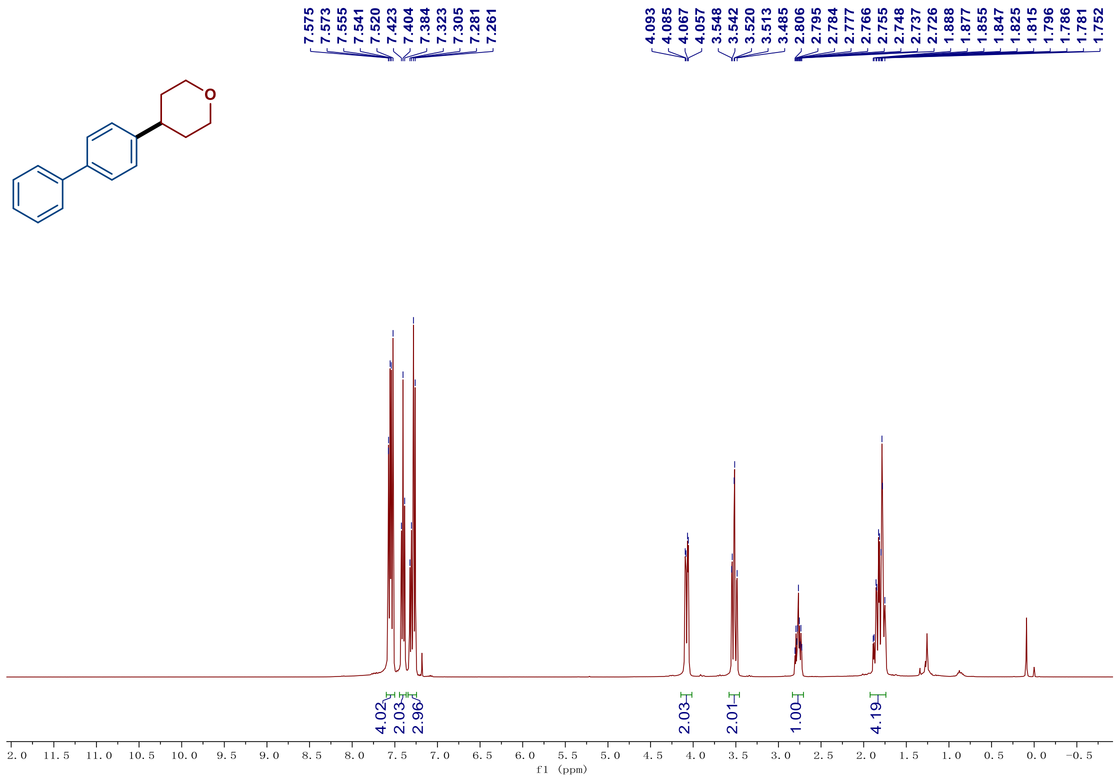

H2N
O
144.589
136.041
127.386
115.197
68.403
40.580
34.161
f1 (ppm)

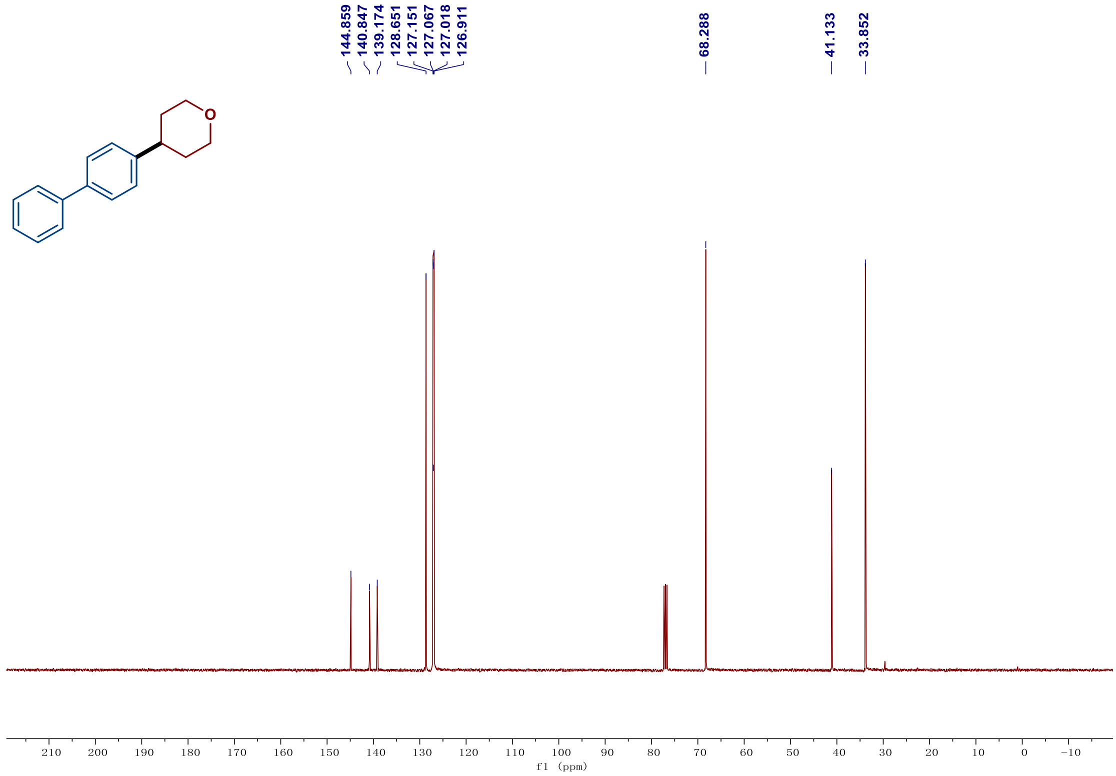

9.983
7.852
7.832
7.407
7.387
4.126
4.121
4.113
4.095
4.085
3.581
3.574
3.552
3.545
3.523
3.516
2.901
2.890
2.879
2.872
2.861
2.850
2.843
2.832
2.821
1.902
1.891
1.870
1.859
1.839
1.828
1.810
1.801
1.791
1.777
1.771
1.765
OHC
O
0.95
1.97
1.96
1.98
2.02
1.02
4.12
f1 (ppm)

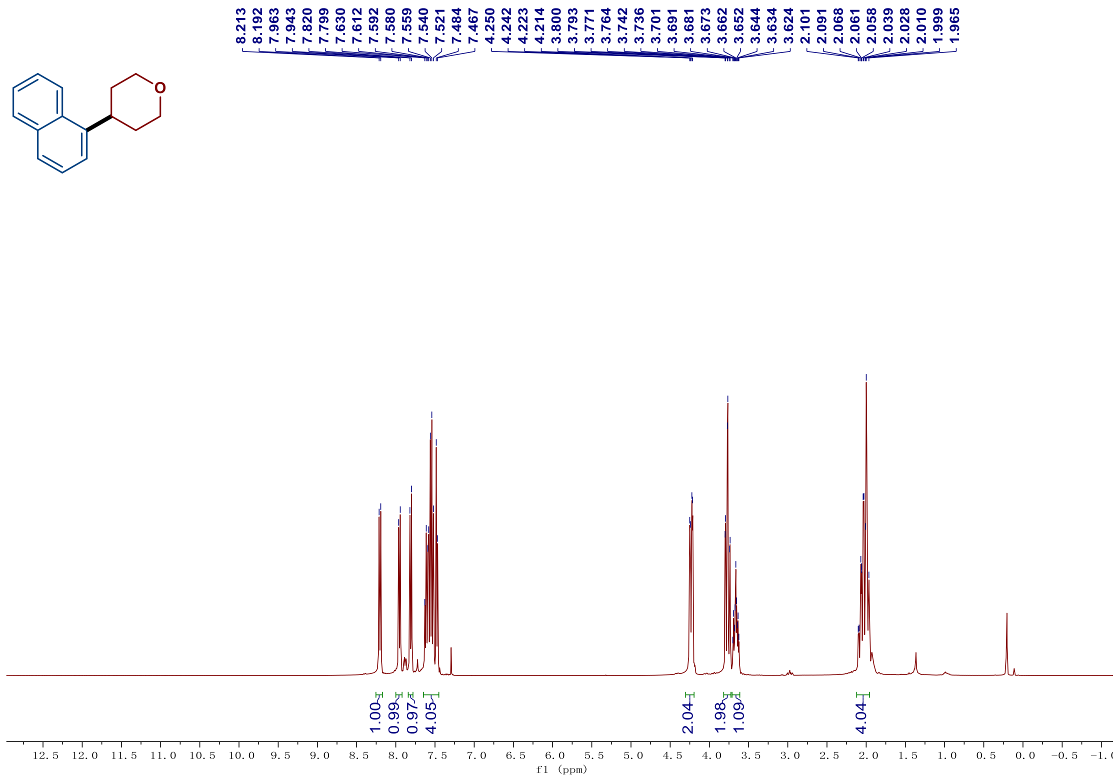

191.807
152.799
134.813
130.026
127.373
68.027
41.751
33.389
OHC
O
f1 (ppm)

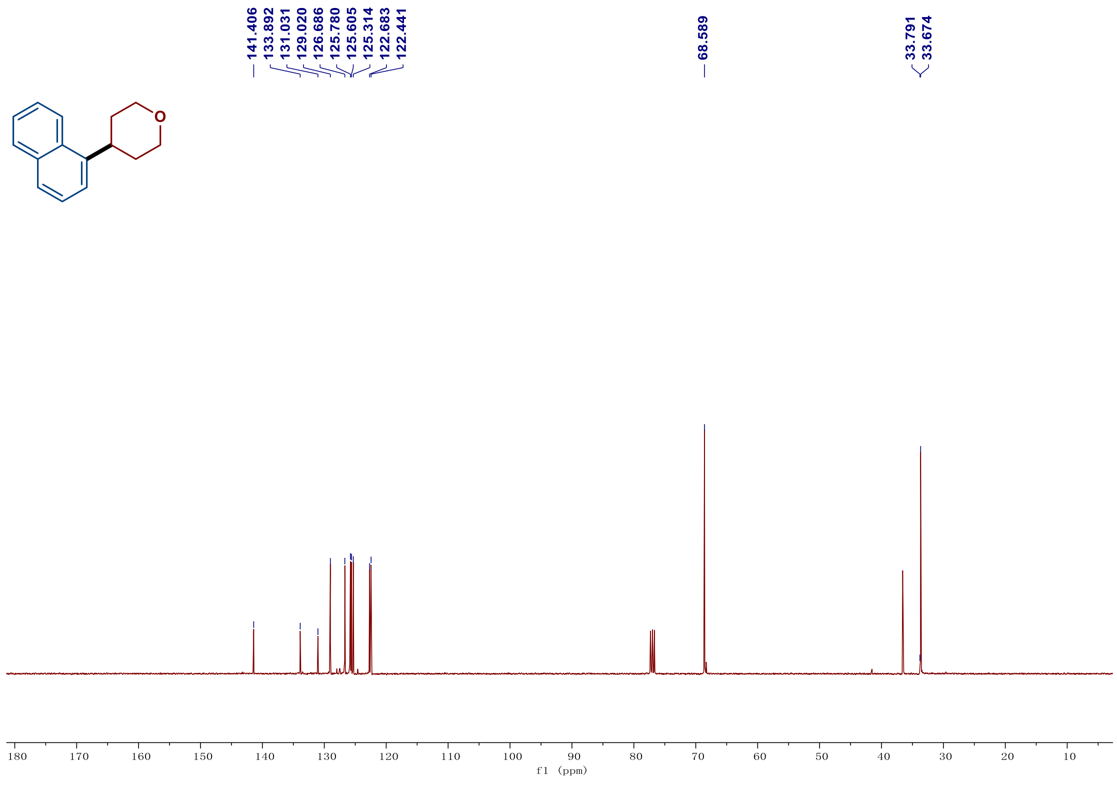

7.78
7.76
7.25
7.23
4.09
4.08
4.06
4.05
3.55
3.55
3.52
3.52
3.49
3.49
2.80
2.79
2.78
2.78
2.76
2.75
2.75
2.74
2.73
1.88
1.87
1.85
1.84
1.84
1.82
1.81
1.79
1.77
1.76
1.74
1.33
Bpin
O
1.95
2.07
2.01
2.04
1.03
4.05
11.90
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2.0 -3.0
f1 (ppm)

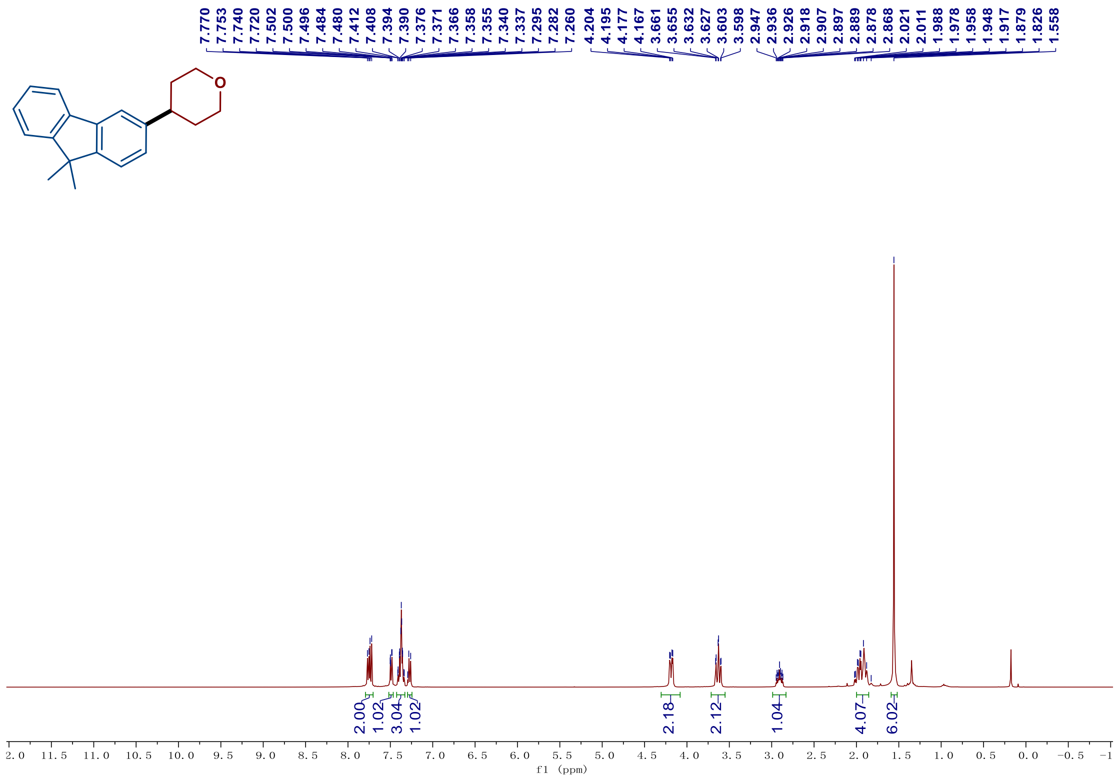

149.14
135.11
126.23
83.69
68.36
41.79
33.74
24.87
Bpin
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

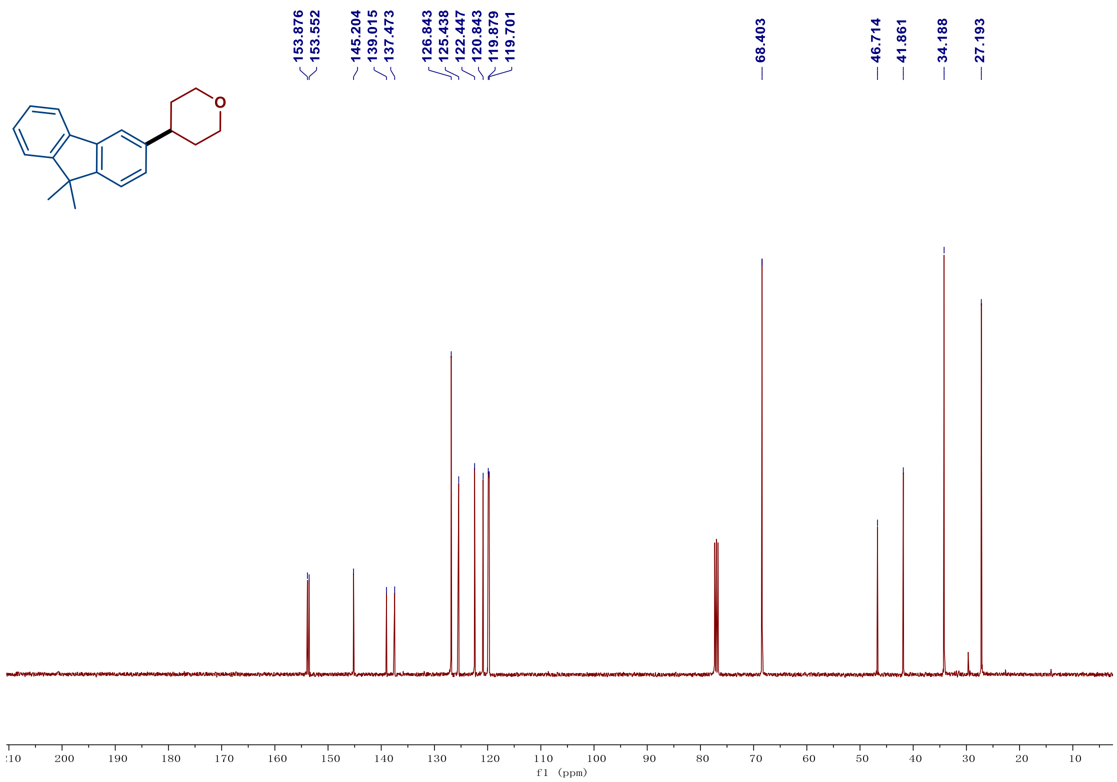

7.575 7.573 7.555 7.541 7.520 7.423 7.404 7.384 7.323 7.305 7.281 7.261
4.093 4.085 4.067 4.057 3.548 3.542 3.520 3.513 3.485 2.806 2.795 2.784 2.777 2.766 2.755 2.748 2.737 2.726 1.888 1.877 1.855 1.847 1.825 1.815 1.796 1.786 1.781 1.752
O
4.02 2.03 2.96 2.03 2.01 1.00 4.19
f1 (ppm)

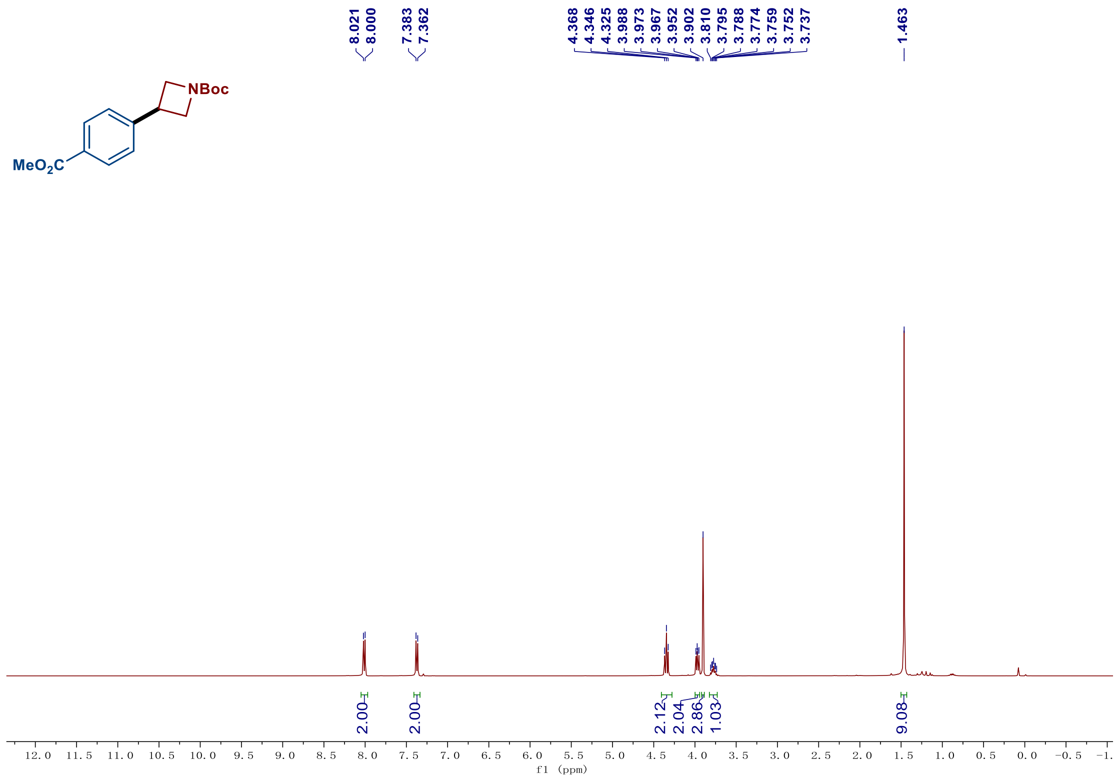

144.859 140.847 139.174 128.651 127.151 127.067 127.018 126.911
68.288
41.133
33.852
O
f1 (ppm)


8.213
8.192
7.963
7.943
7.820
7.799
7.630
7.612
7.592
7.580
7.559
7.540
7.521
7.484
7.467
4.250
4.242
4.223
4.214
3.800
3.793
3.771
3.764
3.742
3.736
3.701
3.691
3.681
3.673
3.662
3.652
3.644
3.634
3.624
2.101
2.091
2.068
2.061
2.058
2.039
2.028
2.010
1.999
1.965
O
1.00
0.99
0.97
4.05
2.04
1.98
1.09
4.04
f1 (ppm)

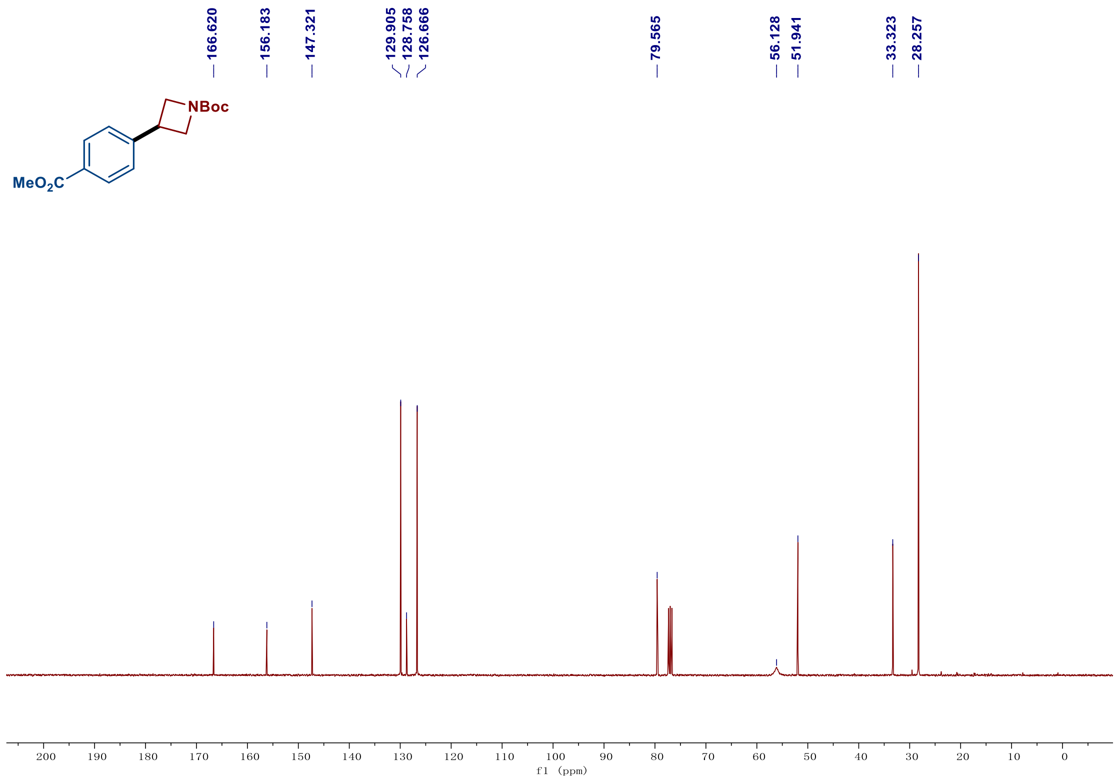

141.406
133.892
131.031
129.020
126.686
125.780
125.605
125.314
122.683
122.441
68.589
33.791
33.674
O
f1 (ppm)



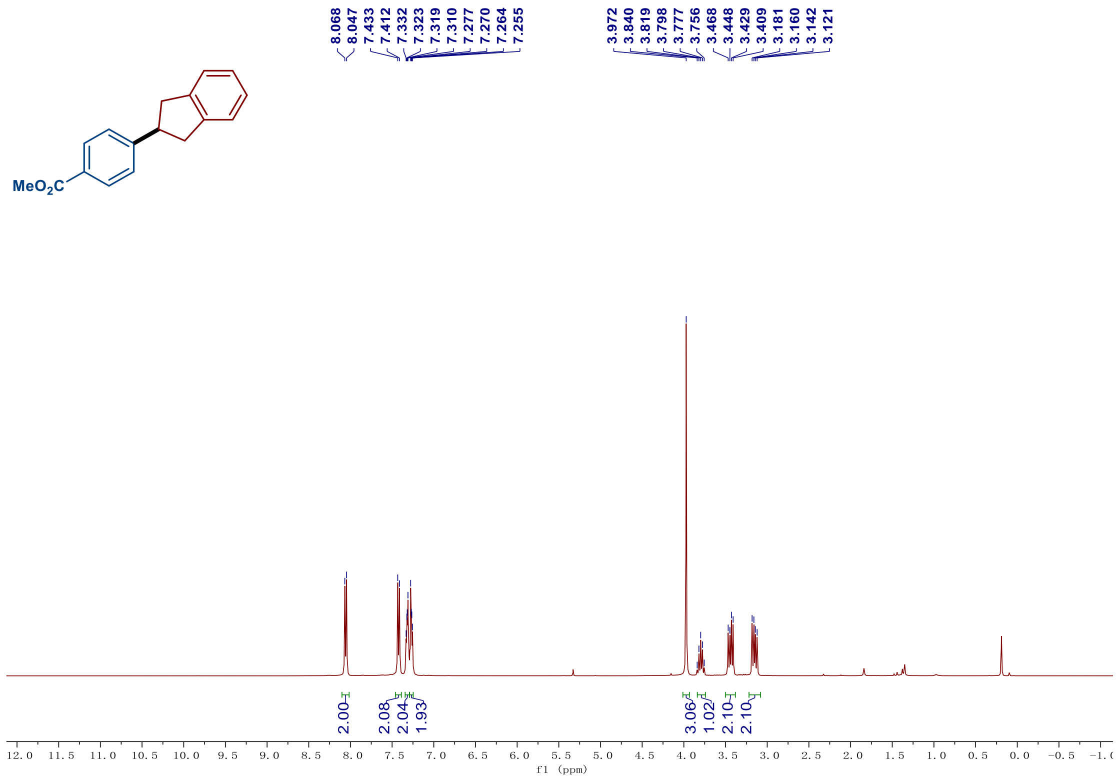


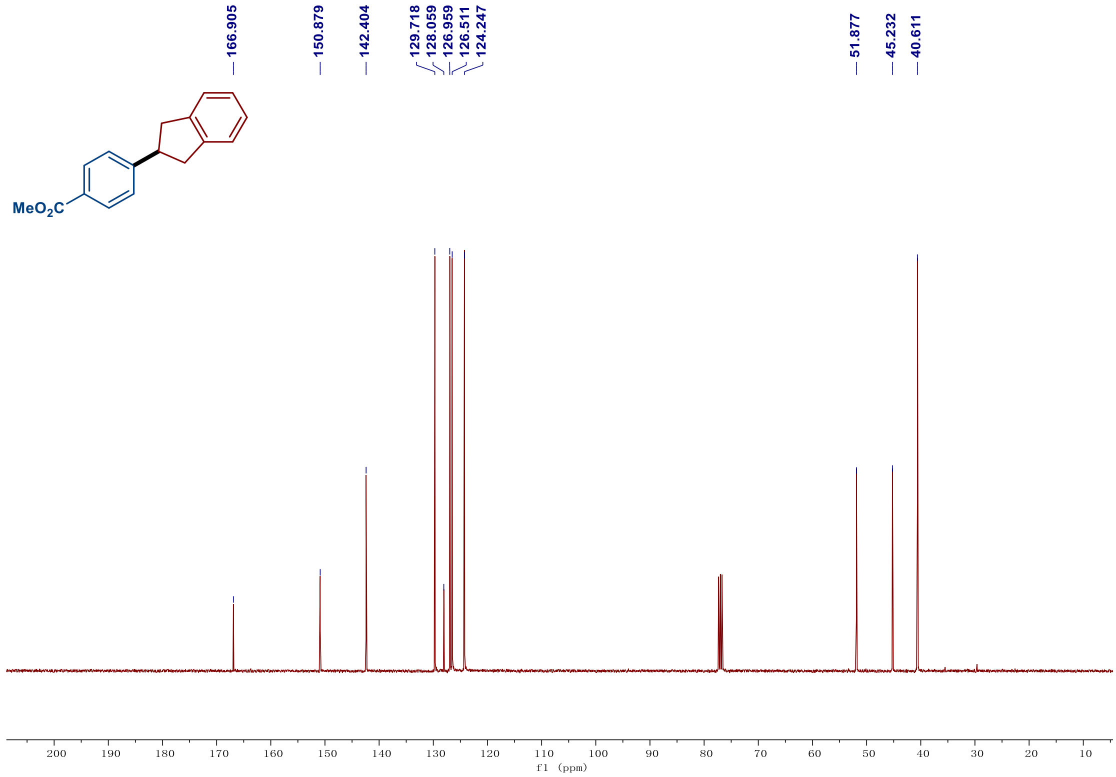


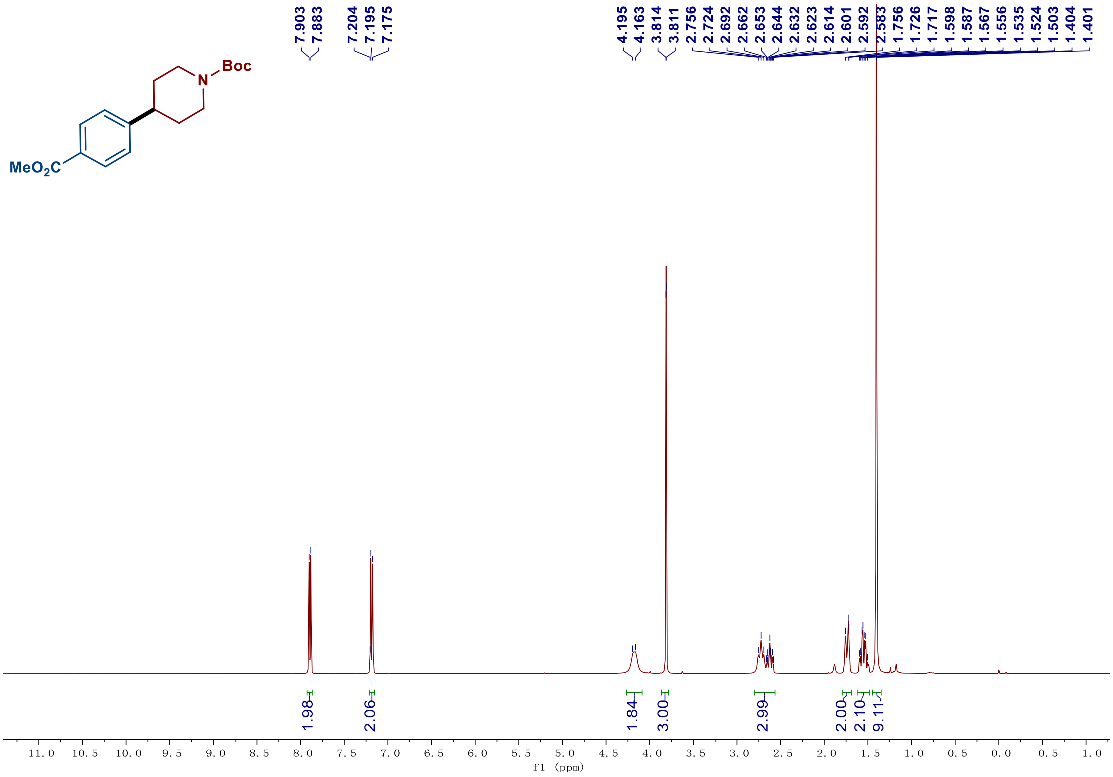
NBoc
MeO2C
8.021
8.000
7.383
7.362
4.368
4.346
4.325
3.988
3.973
3.967
3.952
3.902
3.810
3.795
3.788
3.774
3.759
3.752
3.737
1.463
2.00
2.00
2.12
2.04
2.86
1.03
9.08
f1 (ppm)

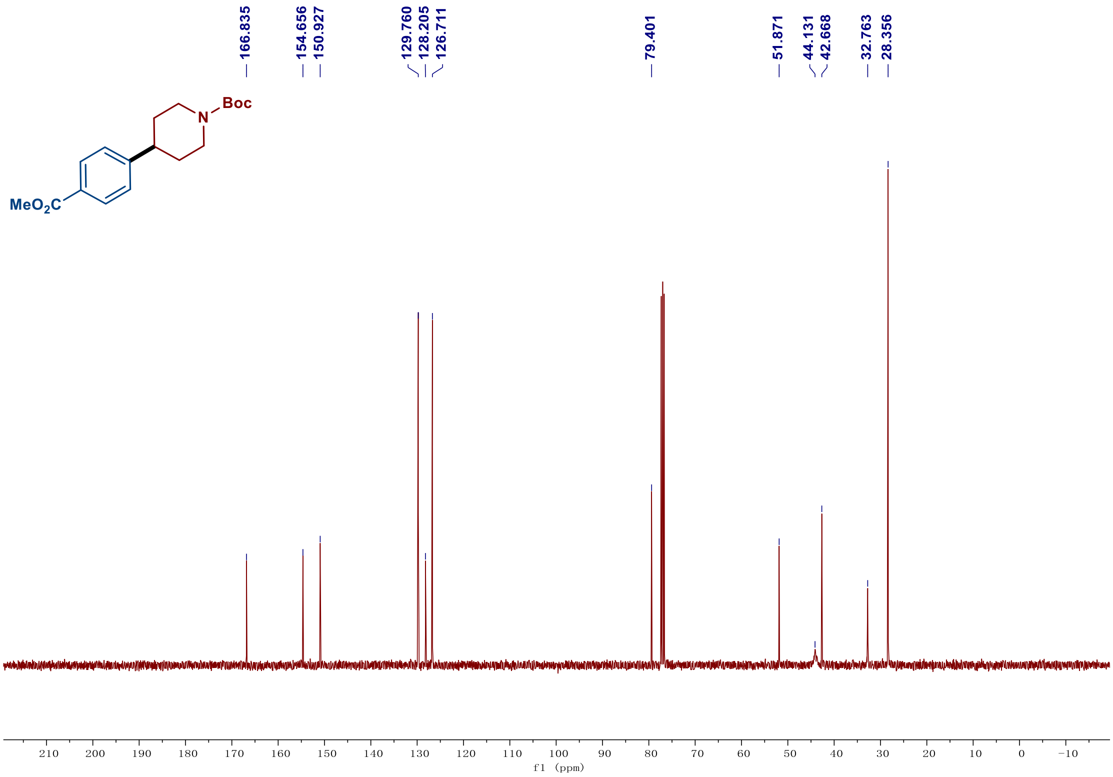


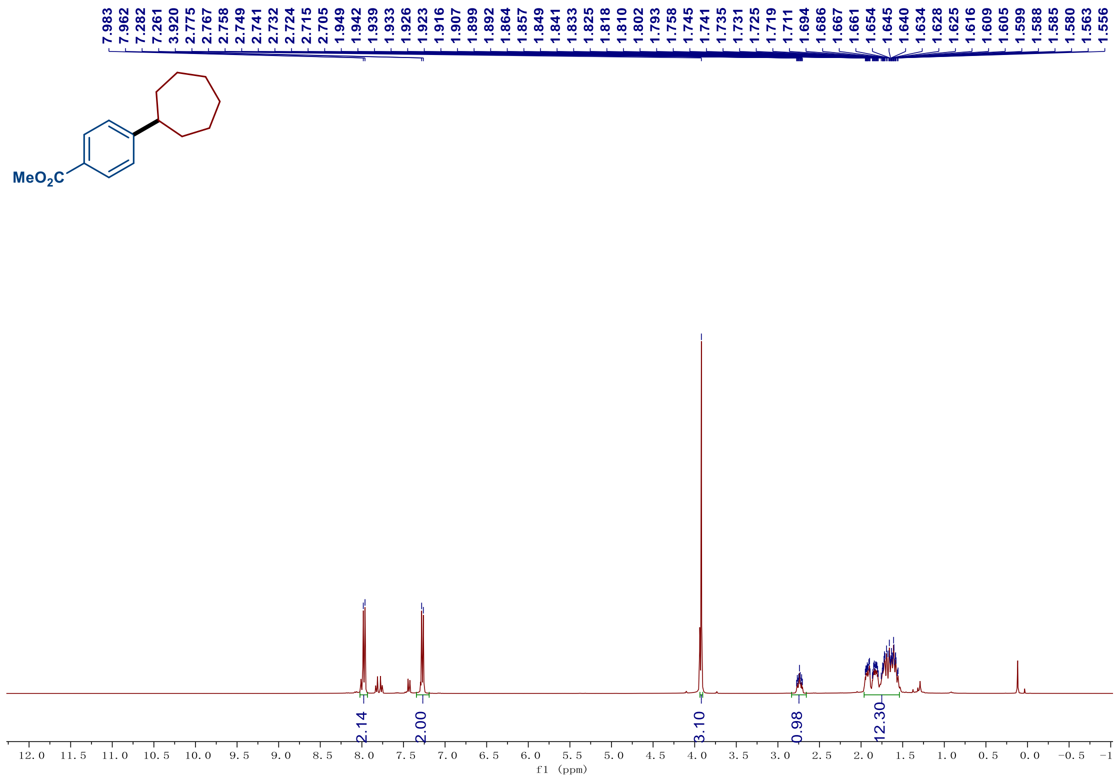
NBoc
MeO2C
166.620
156.183
147.321
129.905
128.758
126.666
79.565
56.128
51.941
33.323
28.257
f1 (ppm)


8.068
8.047
7.433
7.412
7.332
7.323
7.319
7.310
7.277
7.270
7.264
7.255
3.972
3.840
3.819
3.798
3.777
3.756
3.468
3.448
3.429
3.409
3.181
3.160
3.142
3.121
MeO2C
2.00
2.08
2.04
1.93
3.06
1.02
2.10
2.10
f1 (ppm)

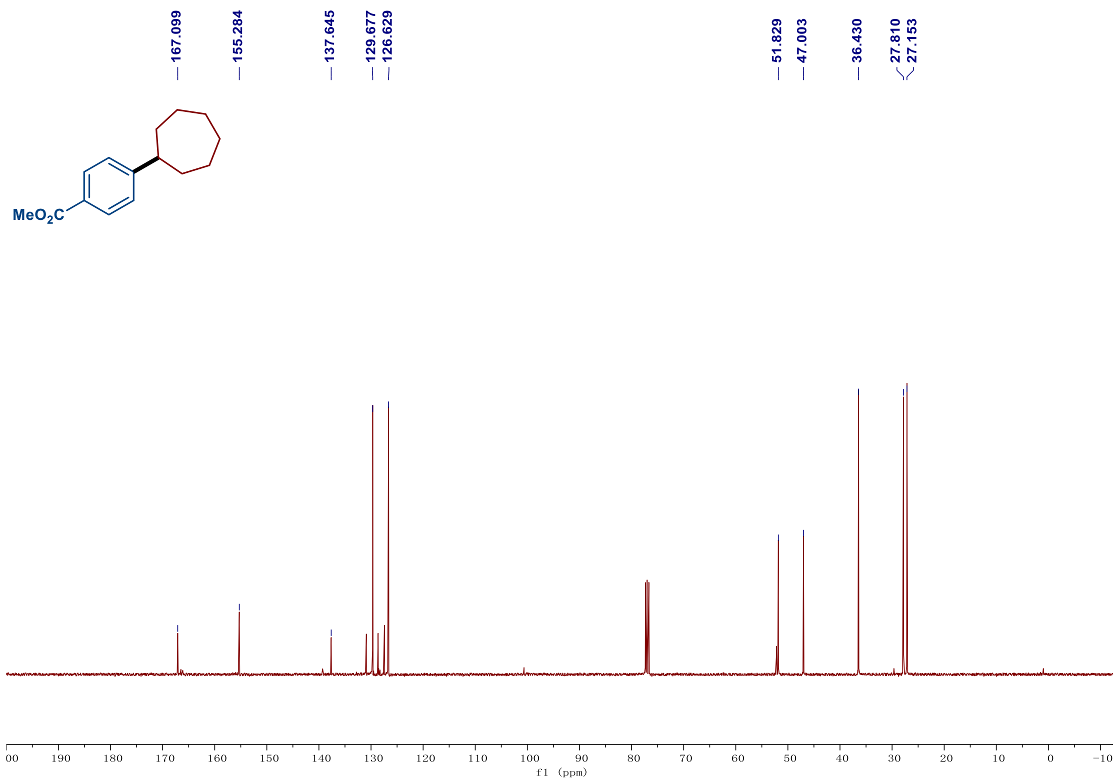

166.905
150.879
142.404
129.718
128.059
126.959
126.511
124.247
51.877
45.232
40.611
MeO2C
f1 (ppm)


Boc
N
MeO2C
7.903
7.883
7.204
7.195
7.175
4.195
4.163
3.814
3.811
2.756
2.724
2.692
2.662
2.653
2.644
2.632
2.623
2.614
2.601
2.592
2.583
1.756
1.726
1.717
1.598
1.587
1.567
1.556
1.535
1.524
1.503
1.404
1.401
1.98
2.06
1.84
3.00
2.99
2.00
2.10
9.11
f1 (ppm)

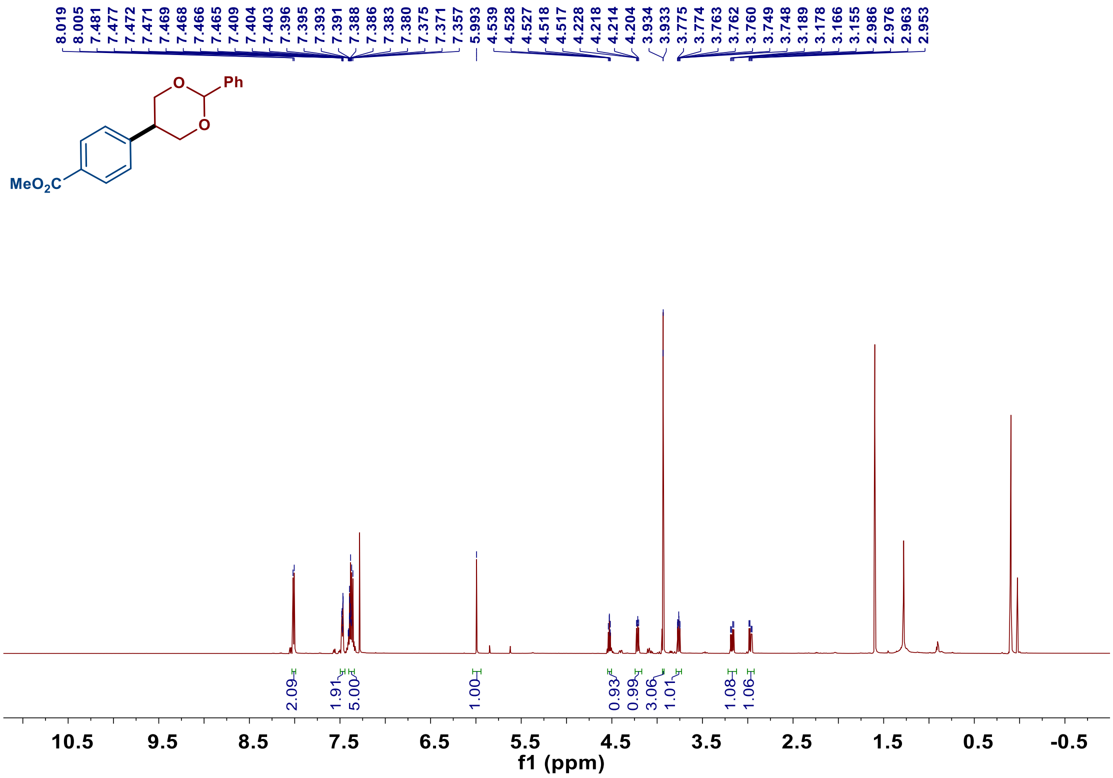

Boc
N
MeO2C
166.835
154.656
150.927
129.760
128.205
126.711
79.401
51.871
44.131
42.668
32.763
28.356
f1 (ppm)

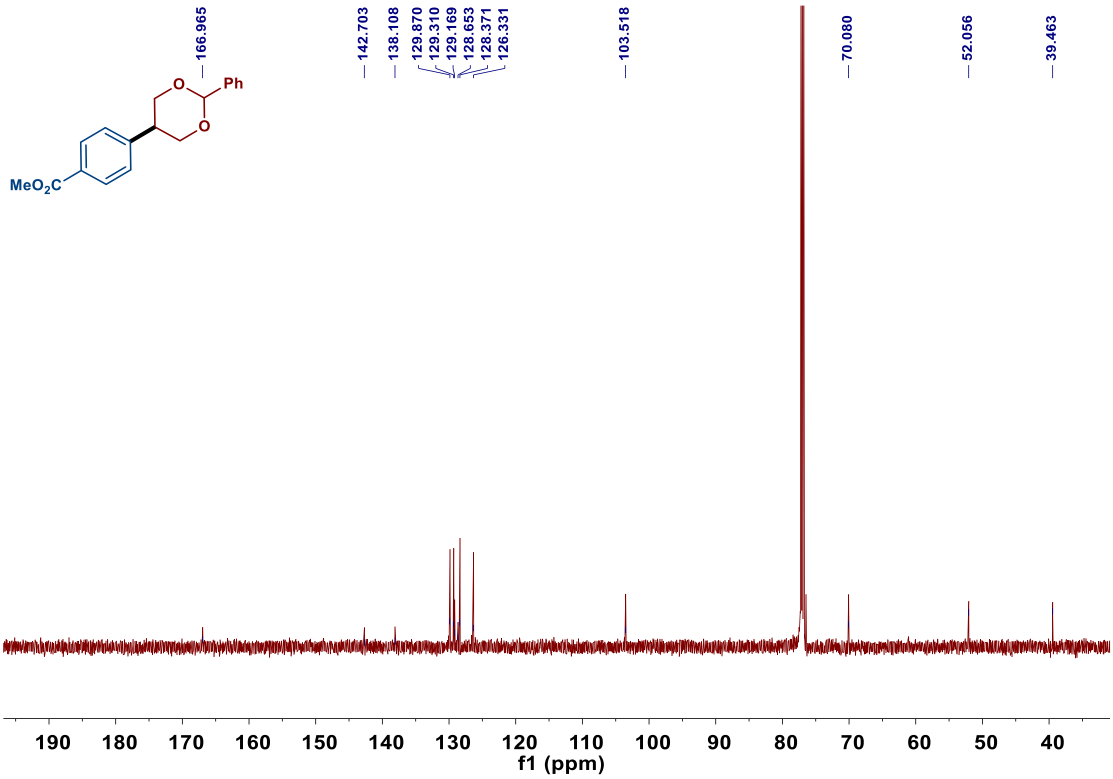


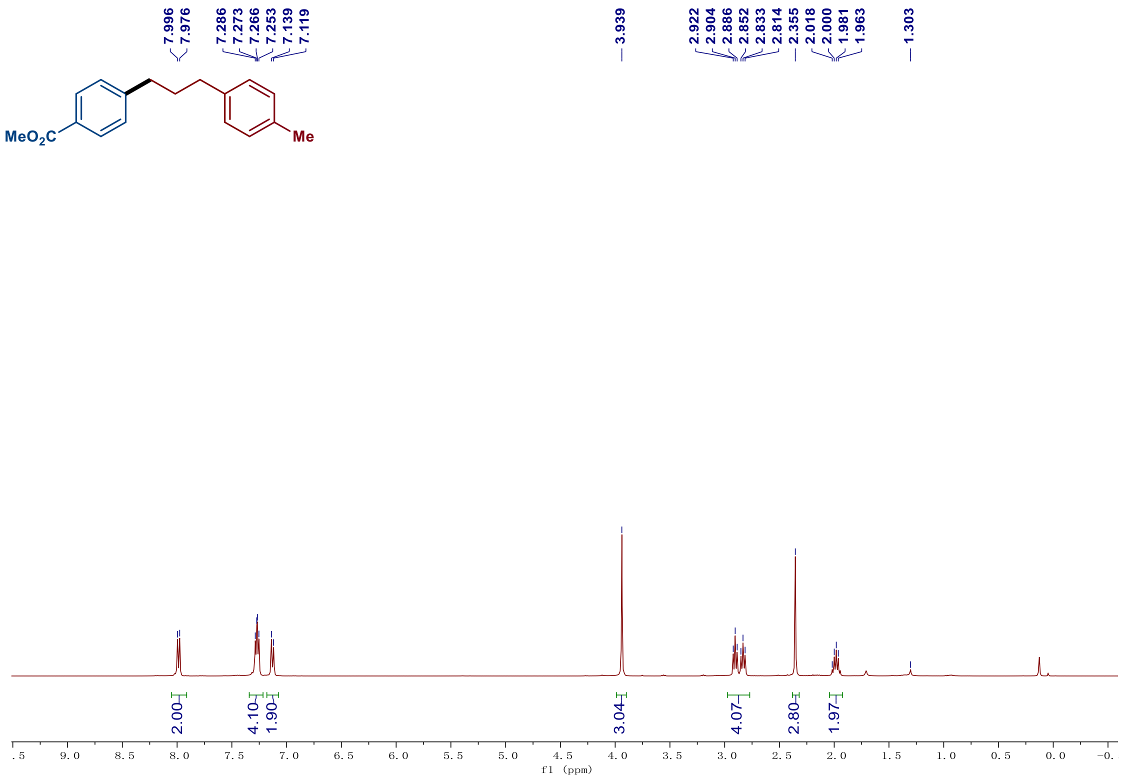
7.983
7.962
7.282
7.261
3.920
2.775
2.767
2.758
2.749
2.741
2.732
2.724
2.715
2.705
1.949
1.942
1.939
1.933
1.926
1.923
1.916
1.907
1.899
1.892
1.864
1.857
1.849
1.841
1.833
1.825
1.818
1.810
1.802
1.793
1.758
1.745
1.741
1.735
1.731
1.725
1.719
1.711
1.694
1.686
1.667
1.661
1.654
1.645
1.640
1.634
1.628
1.625
1.616
1.609
1.605
1.599
1.588
1.580
1.563
1.556
MeO2C
2.14
2.00
3.10
0.98
12.30
f1 (ppm)

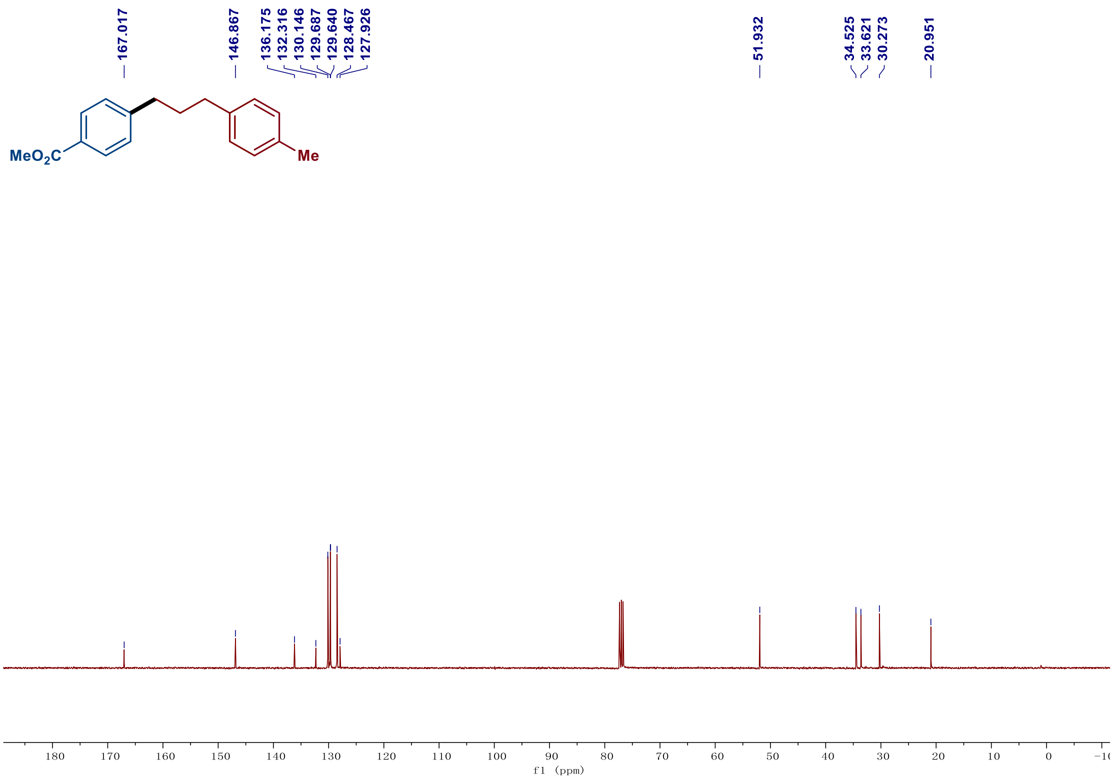


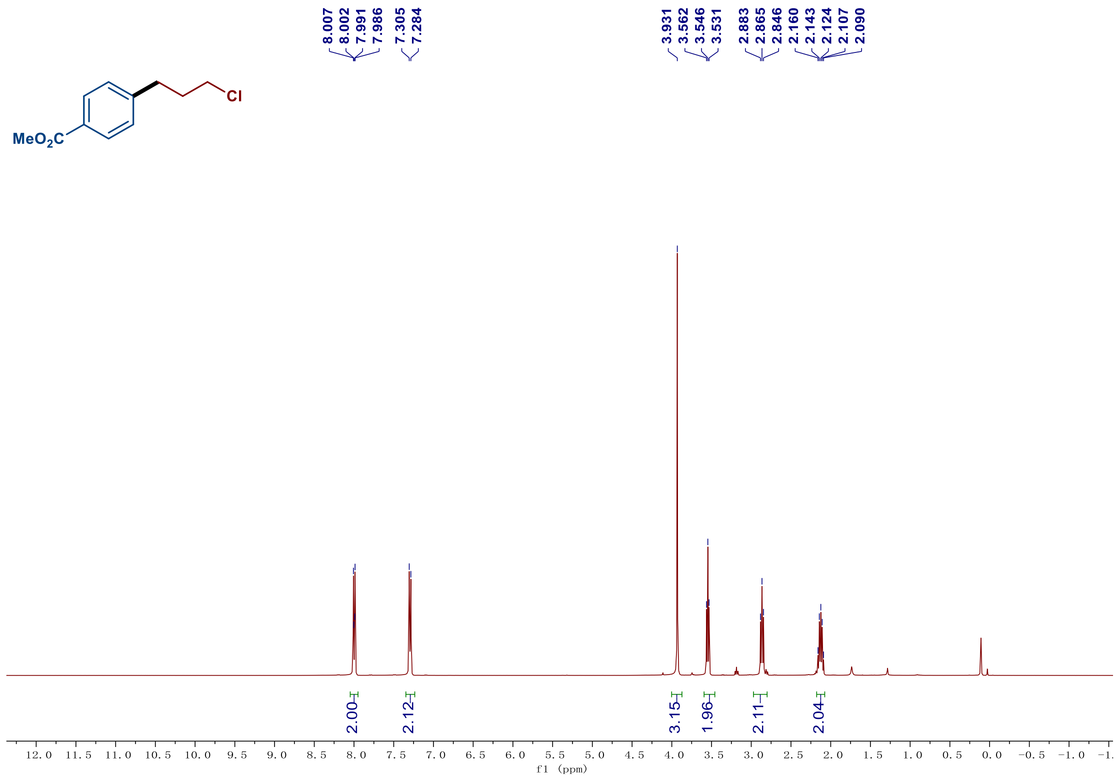
167.099
155.284
137.645
129.677
126.629
51.829
47.003
36.430
27.810
27.153
MeO2C
f1 (ppm)


8.019
8.005
7.481
7.477
7.472
7.471
7.469
7.468
7.466
7.465
7.409
7.404
7.403
7.396
7.395
7.393
7.391
7.388
7.386
7.383
7.380
7.375
7.371
7.357
5.993
4.539
4.528
4.527
4.518
4.517
4.228
4.218
4.214
4.204
3.934
3.933
3.775
3.774
3.763
3.762
3.760
3.749
3.748
3.189
3.178
3.166
3.155
2.986
2.976
2.963
2.953
Ph
O
O
MeO2C
2.09
1.91
5.00
1.00
0.93
0.99
3.06
1.01
1.08
1.06
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

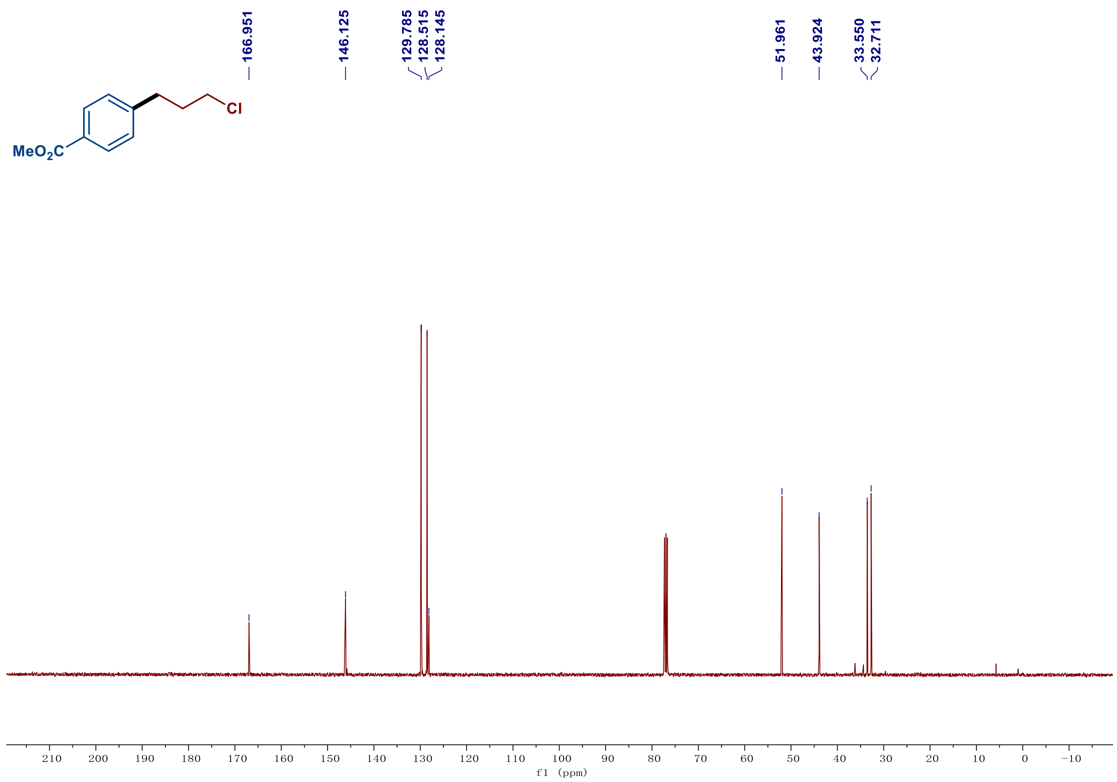

166.965
142.703
138.108
129.870
129.310
129.169
128.653
128.371
126.331
103.518
70.080
52.056
39.463
Ph
O
O
MeO2C
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
f1 (ppm)


7.996
7.976
7.286
7.273
7.266
7.253
7.139
7.119
3.939
2.922
2.904
2.886
2.852
2.833
2.814
2.355
2.018
2.000
1.981
1.963
1.303
MeO2C
Me
2.00
4.10
1.90
3.04
4.07
2.80
1.97
f1 (ppm)

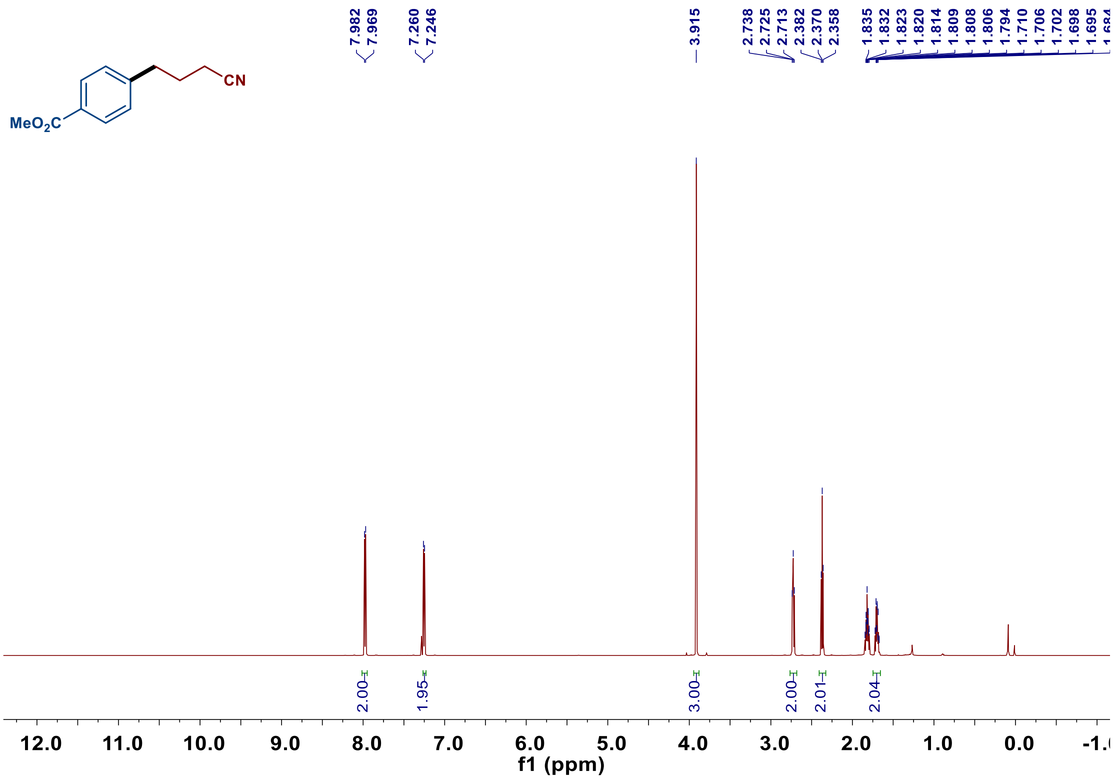

167.017
146.867
136.175
132.316
130.146
129.687
129.640
128.467
127.926
51.932
34.525
33.621
30.273
20.951
MeO2C
Me
f1 (ppm)

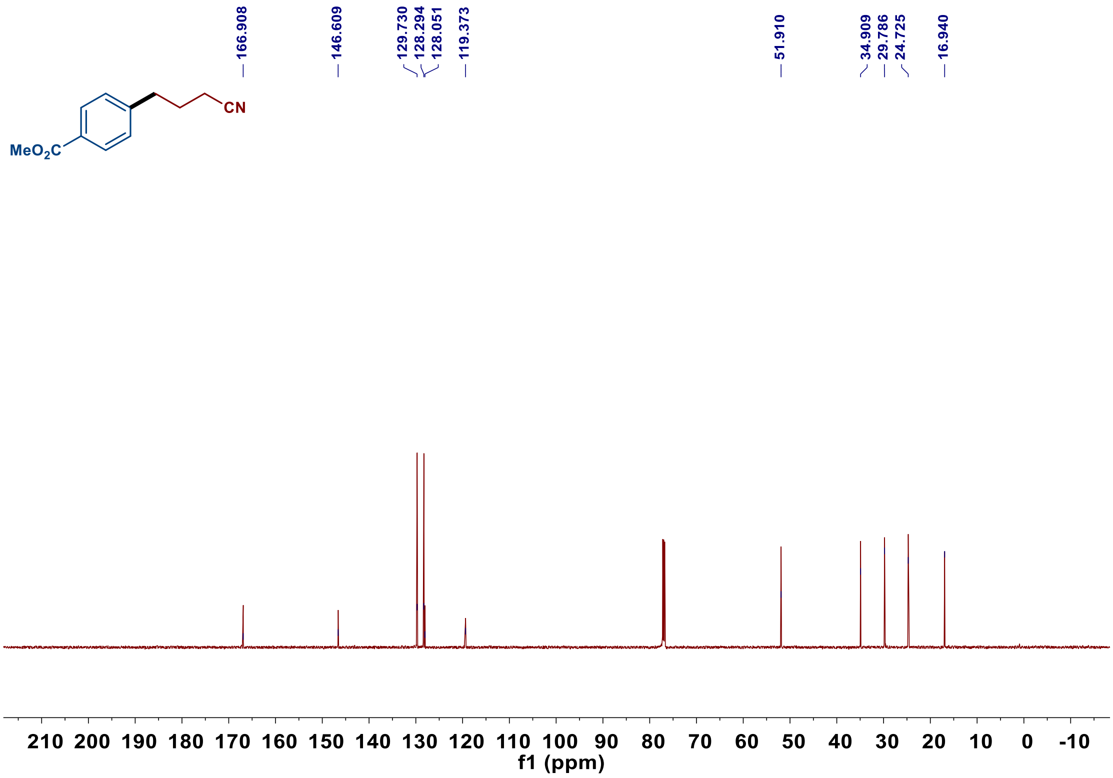


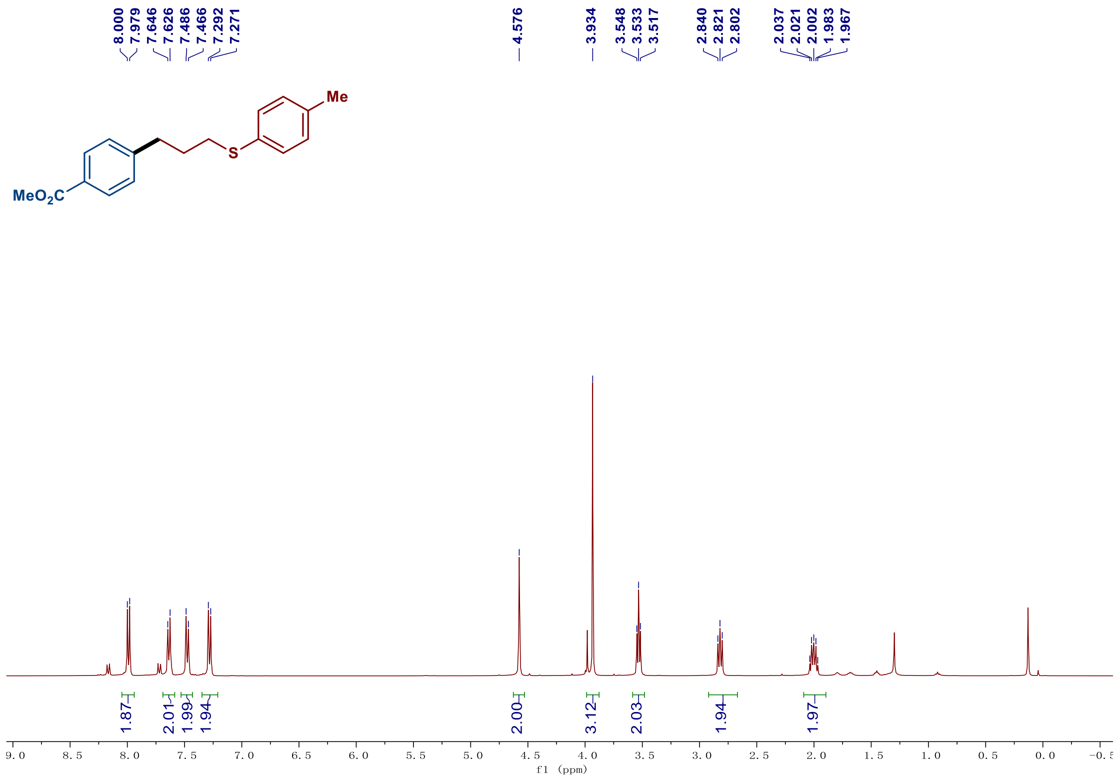
MeO2C
Cl
8.007
8.002
7.991
7.986
7.305
7.284
3.931
3.562
3.546
3.531
2.883
2.865
2.846
2.160
2.143
2.124
2.107
2.090
2.00
2.12
3.15
1.96
2.11
2.04
f1 (ppm)

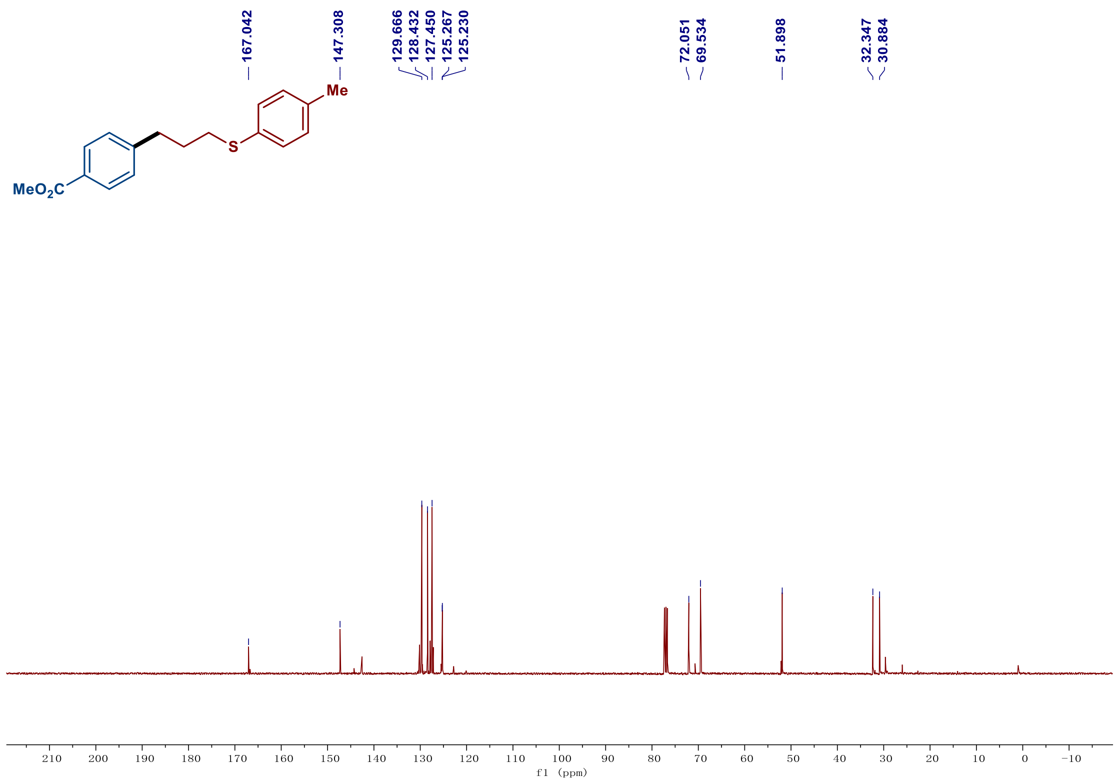


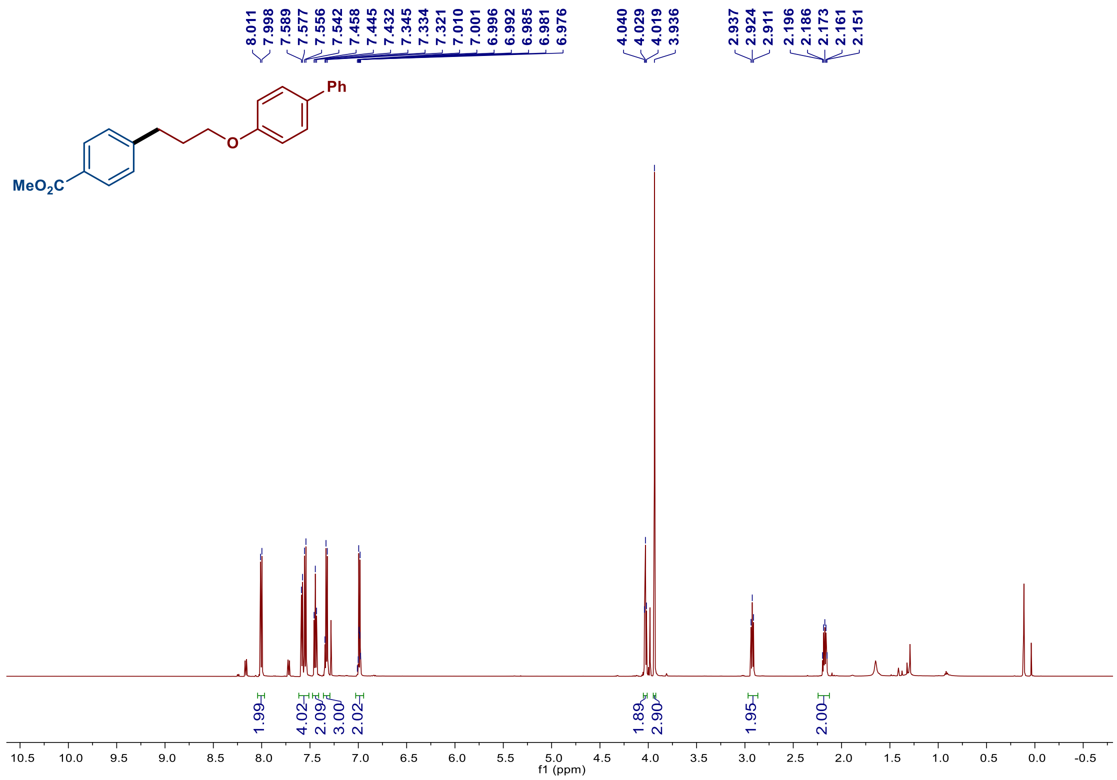
MeO2C
Cl
166.951
146.125
129.785
128.515
128.145
51.961
43.924
33.550
32.711
f1 (ppm)

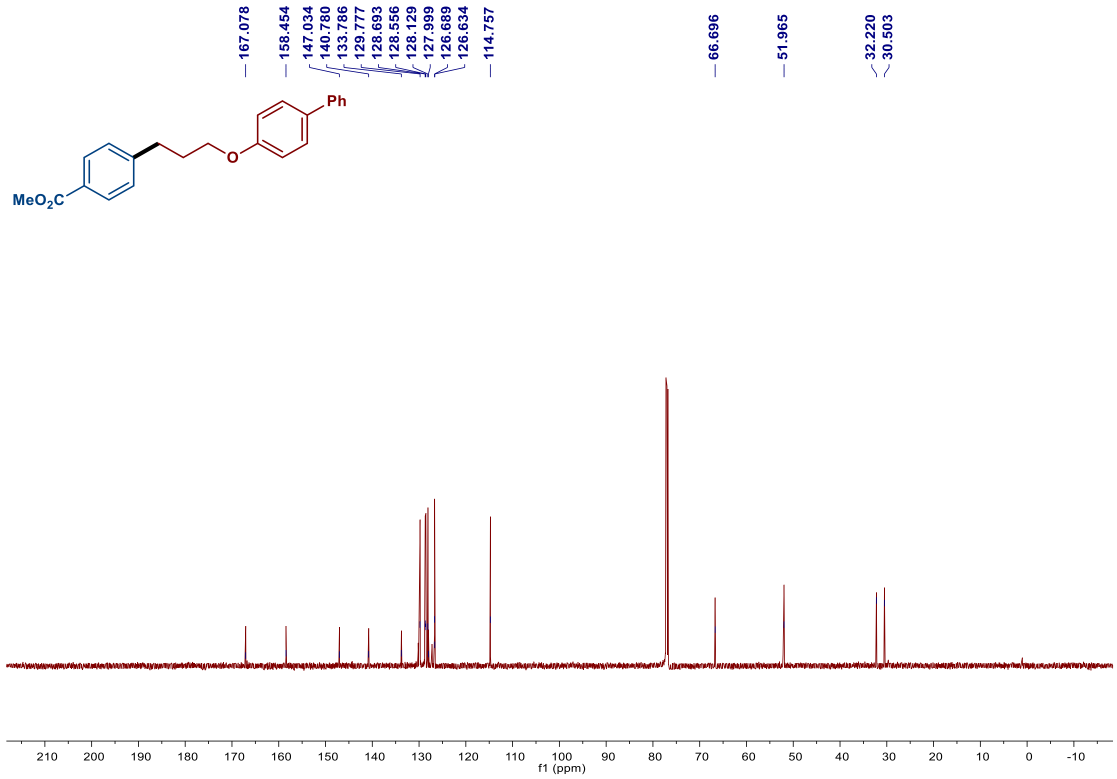

MeO2C
CN
7.982
7.969
7.260
7.246
3.915
2.738
2.725
2.713
2.382
2.370
2.358
1.835
1.832
1.823
1.820
1.814
1.809
1.808
1.806
1.794
1.710
1.706
1.702
1.698
1.695
2.00
1.95
3.00
2.00
2.01
2.04
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

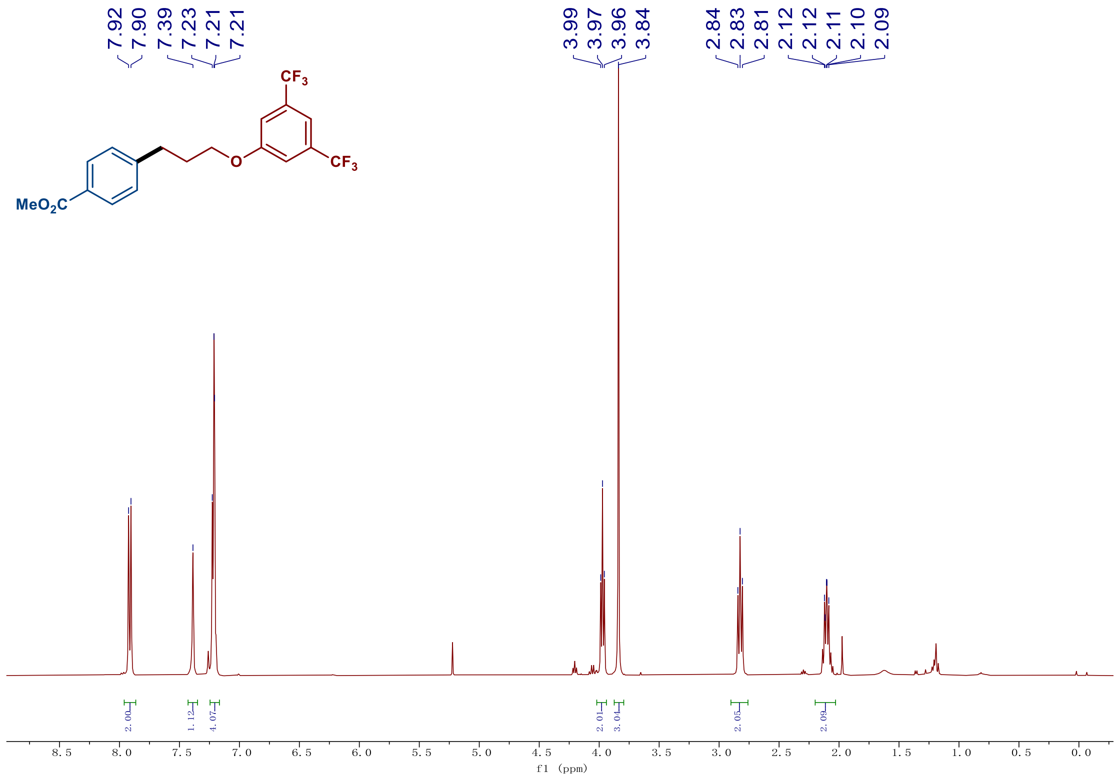

MeO2C
CN
166.908
146.609
129.730
128.294
128.051
119.373
51.910
34.909
29.786
24.725
16.940
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

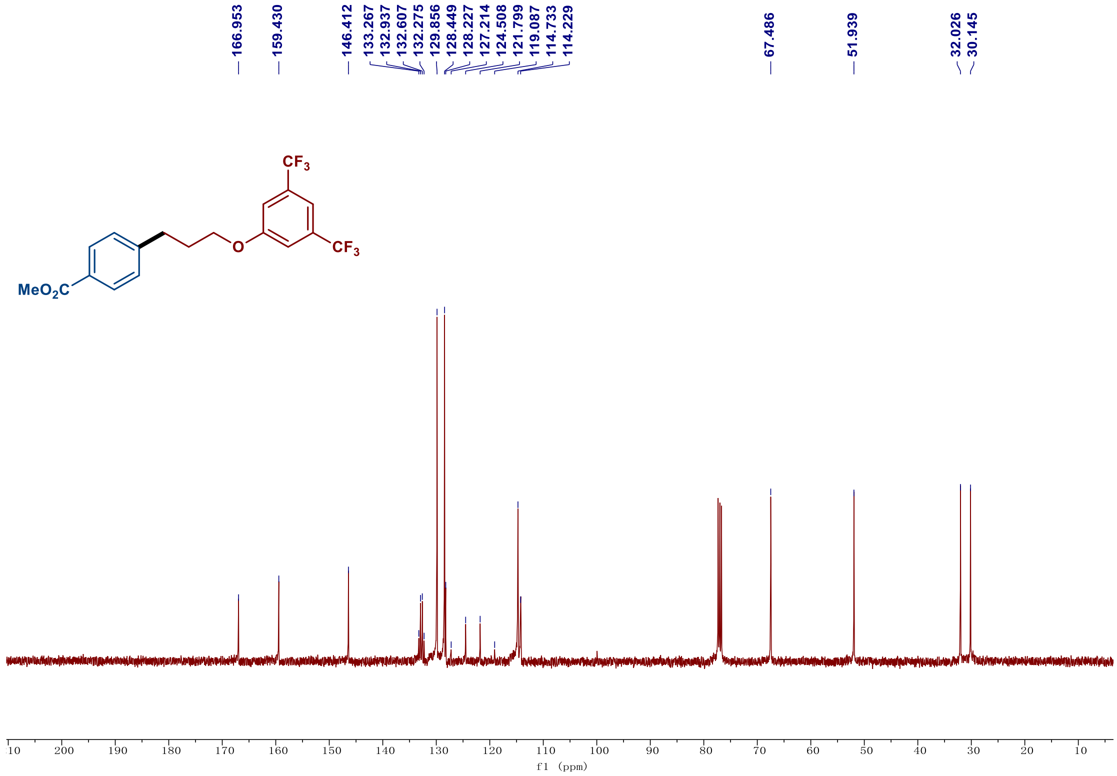

8.000
7.979
7.646
7.626
7.486
7.466
7.292
7.271
4.576
3.934
3.548
3.533
3.517
2.840
2.821
2.802
2.037
2.021
2.002
1.983
1.967
Me
S
MeO2C
1.87
2.01
1.99
1.94
2.00
3.12
2.03
1.94
1.97
f1 (ppm)

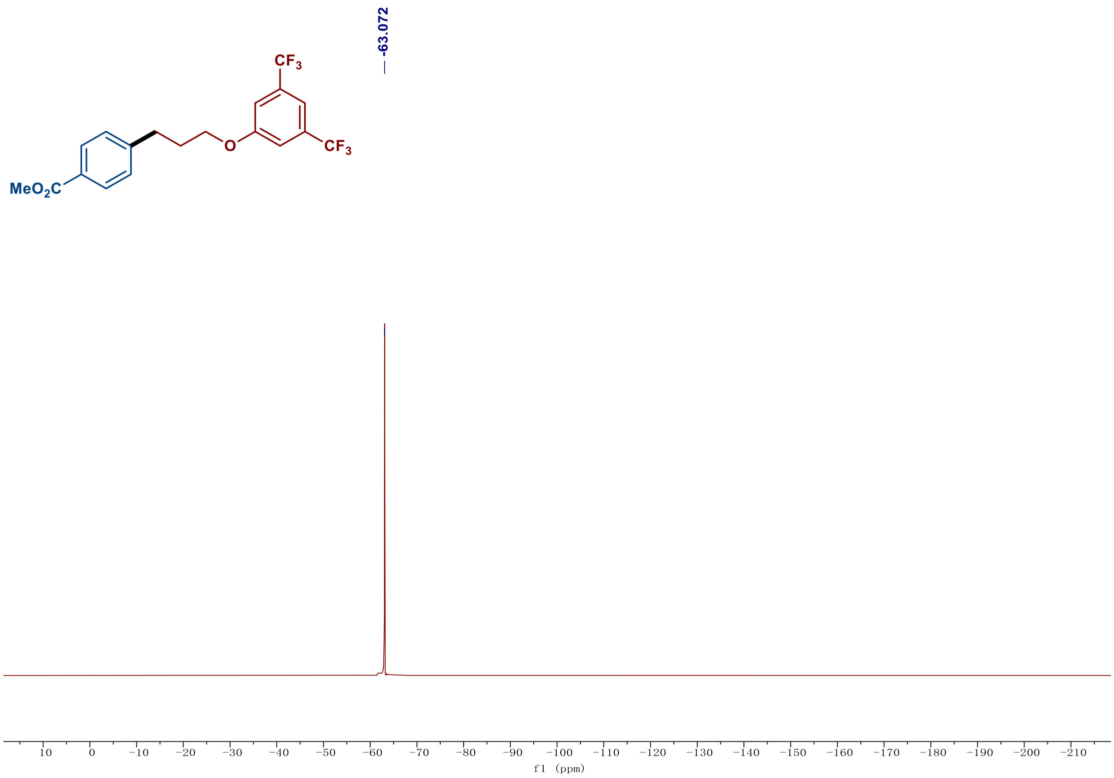

167.042
147.308
129.666
128.432
127.450
125.267
125.230
72.051
69.534
51.898
32.347
30.884
Me
S
MeO2C
f1 (ppm)


8.011
7.998
7.589
7.577
7.556
7.542
7.458
7.445
7.432
7.345
7.334
7.321
7.010
7.001
6.996
6.992
6.985
6.981
6.976
4.040
4.029
4.019
3.936
2.937
2.924
2.911
2.196
2.186
2.173
2.161
2.151
Ph
O
MeO2C
1.99
4.02
2.09
3.00
2.02
1.89
2.90
1.95
2.00
f1 (ppm)

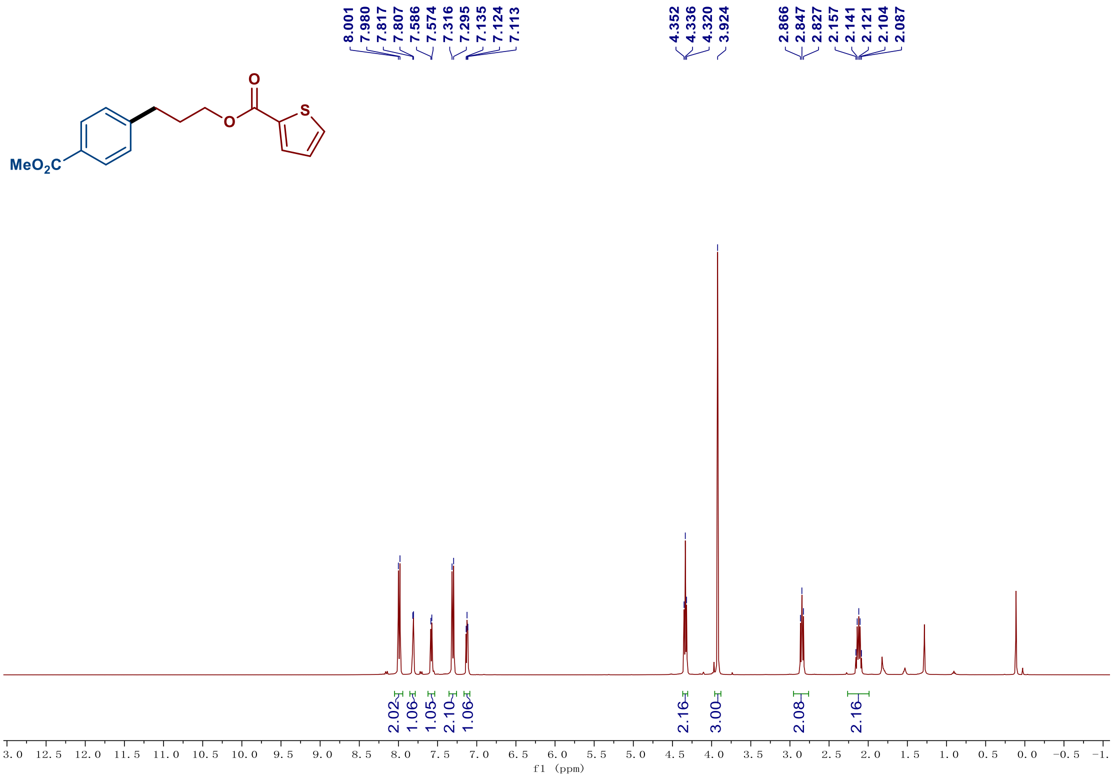

167.078
158.454
147.034
140.780
133.786
129.777
128.693
128.556
128.129
127.999
126.689
126.634
114.757
66.696
51.965
32.220
30.503
Ph
O
MeO2C
f1 (ppm)


7.92
7.90
7.39
7.23
7.21
7.21
3.99
3.97
3.96
3.84
2.84
2.83
2.81
2.12
2.12
2.11
2.10
2.09
CF3
CF3
O
MeO2C
2.00
1.12
4.07
2.01
3.04
2.05
2.09
f1 (ppm)

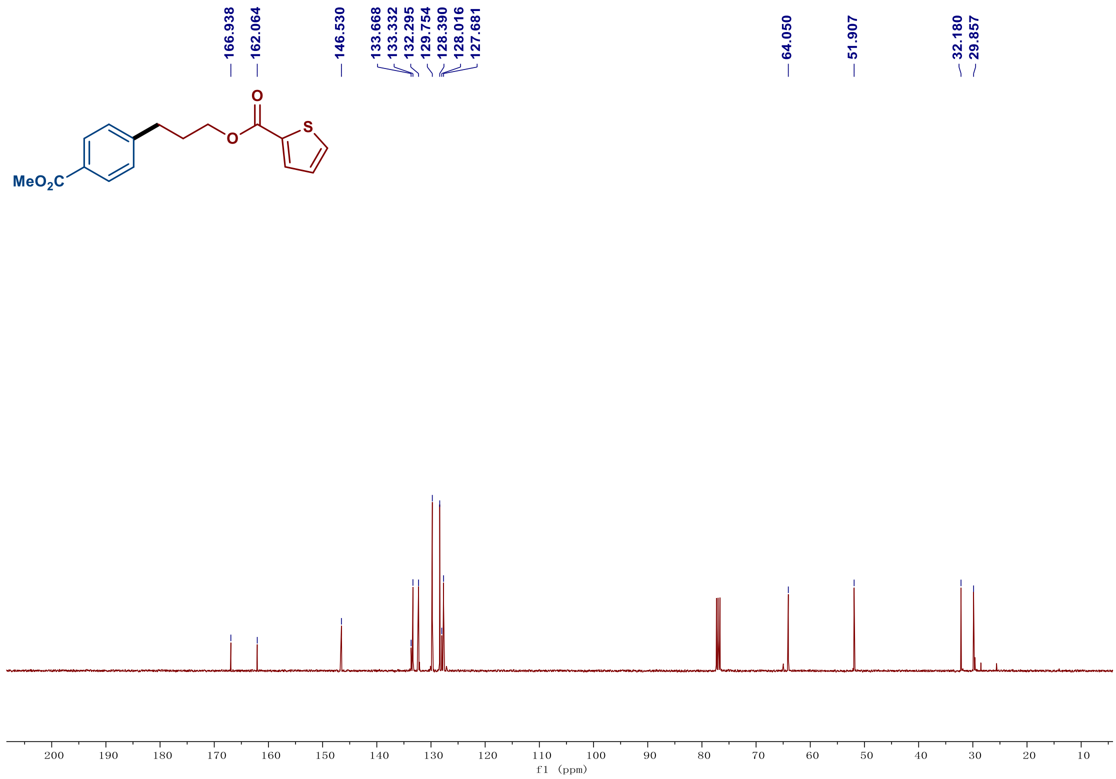

166.953
159.430
146.412
133.267
132.937
132.607
132.275
129.856
128.449
128.227
127.214
124.508
121.799
119.087
114.733
114.229
67.486
51.939
32.026
30.145
CF3
CF3
O
MeO2C
f1 (ppm)

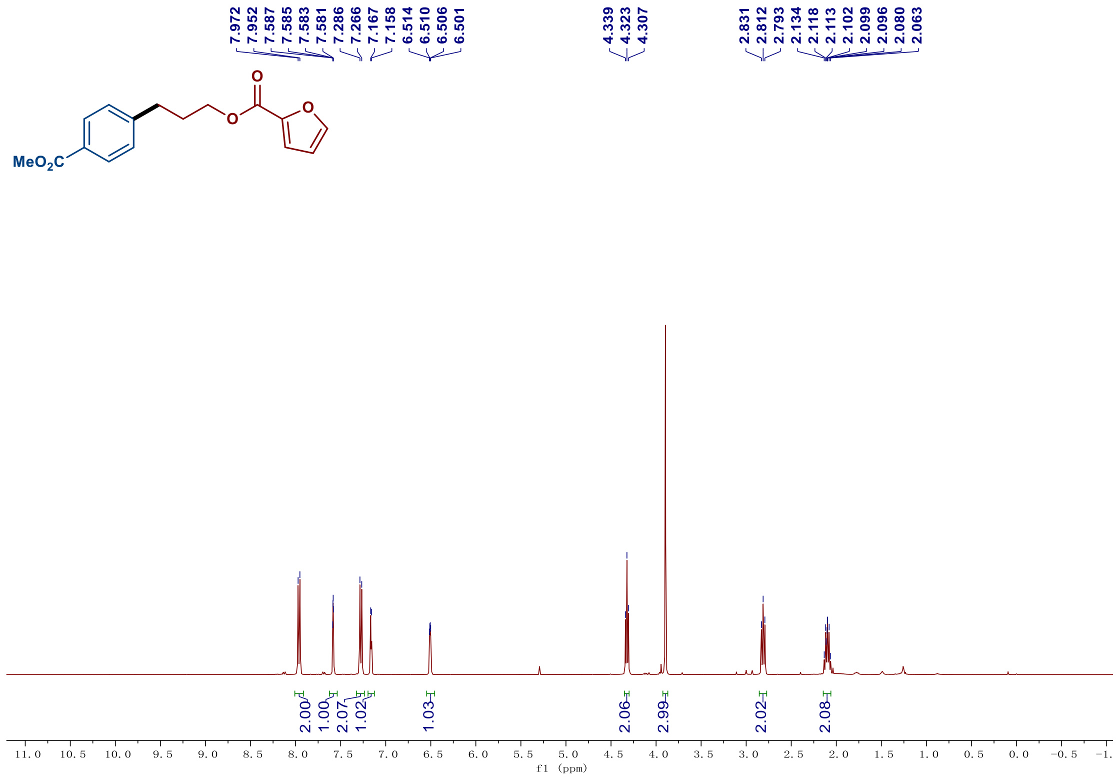


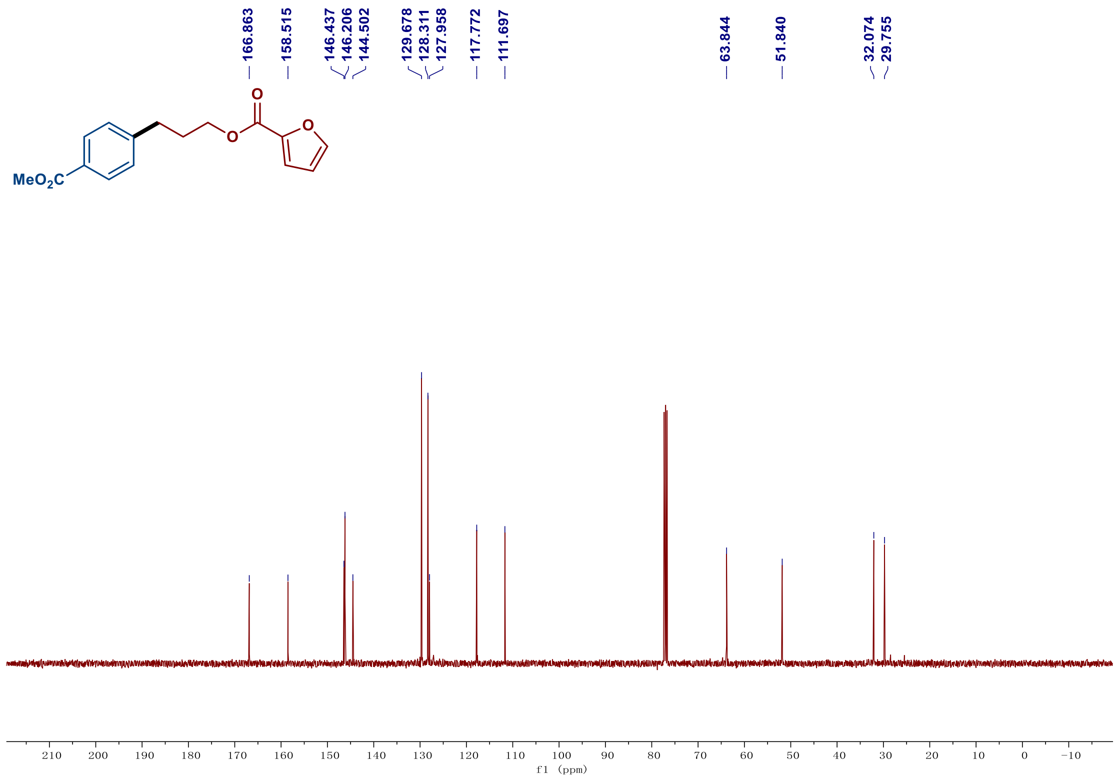
-63.072
CF3
CF3
O
MeO2C
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

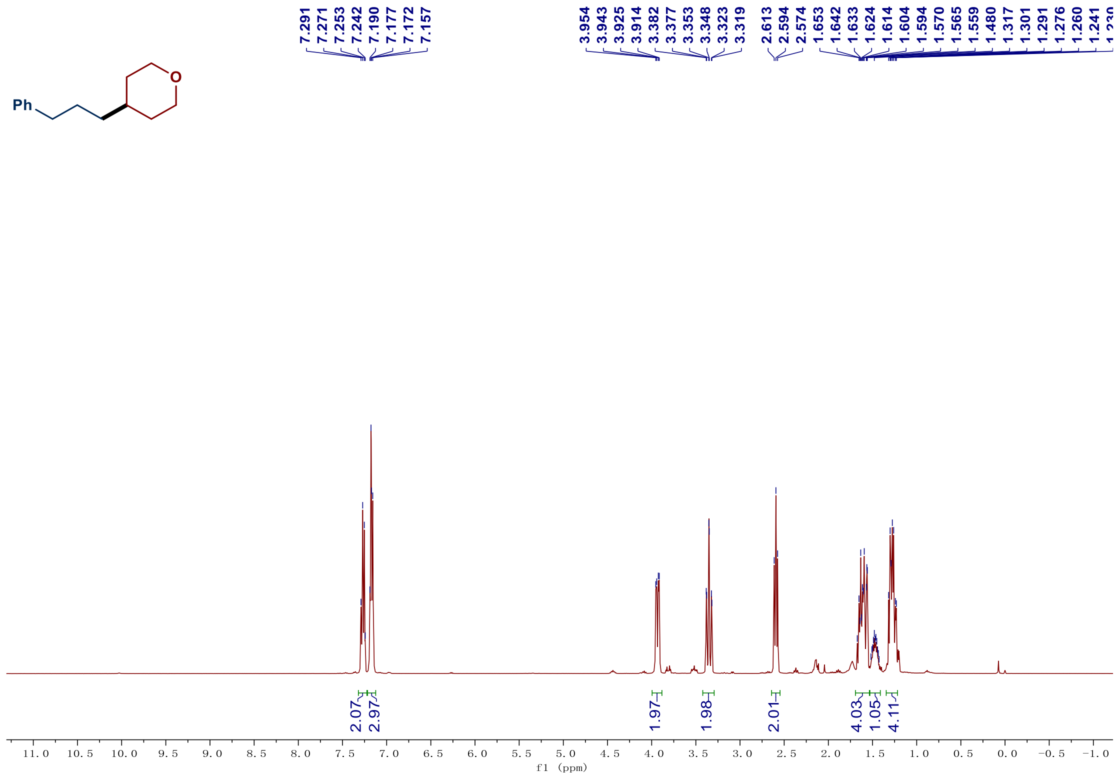


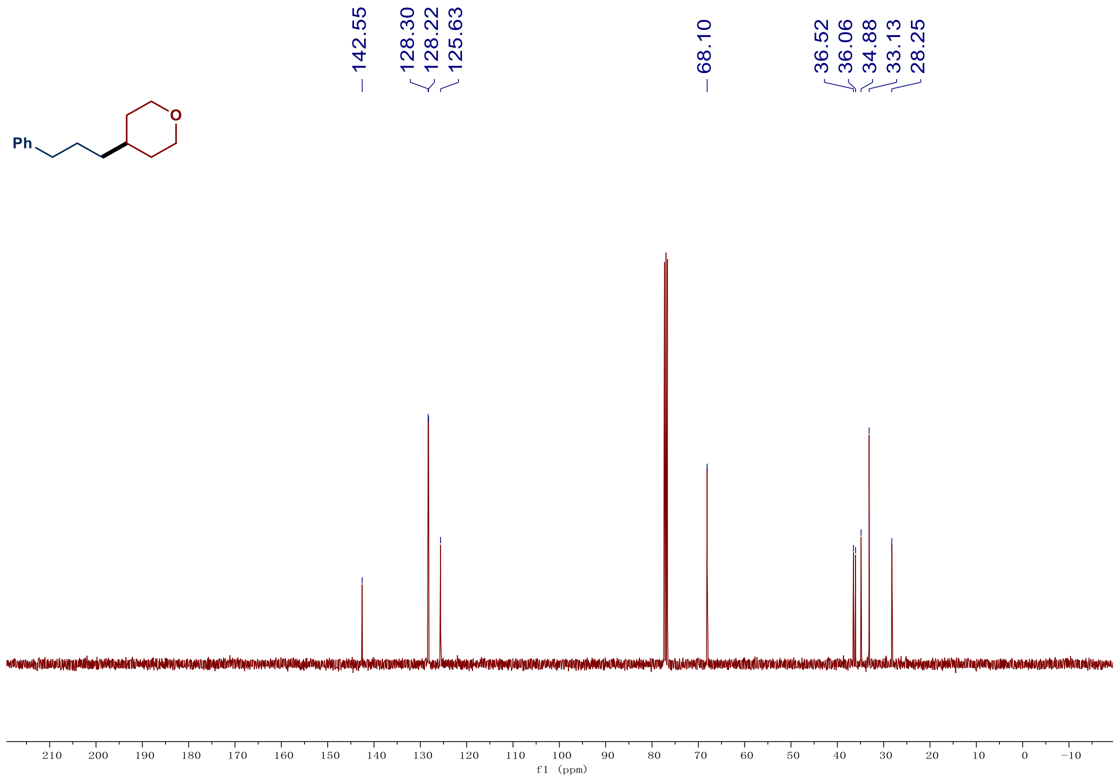
8.001
7.980
7.817
7.807
7.586
7.574
7.316
7.295
7.135
7.124
7.113
4.352
4.336
4.320
3.924
2.866
2.847
2.827
2.157
2.141
2.121
2.104
2.087
O
S
O
MeO2C
2.02
1.06
1.05
2.10
1.06
2.16
3.00
2.08
2.16
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)


166.938
162.064
146.530
133.668
133.332
132.295
129.754
128.390
128.016
127.681
64.050
51.907
32.180
29.857
O
S
O
MeO2C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

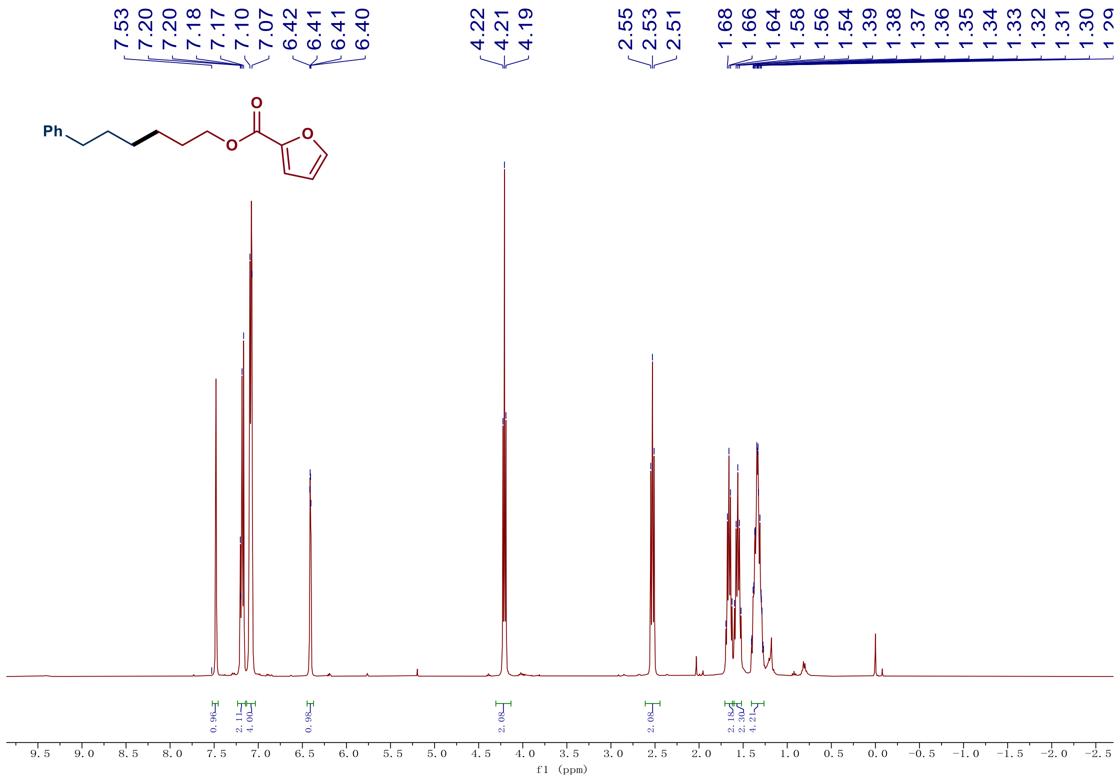

7.972
7.952
7.587
7.585
7.583
7.581
7.286
7.266
7.167
7.158
6.514
6.510
6.506
6.501
4.339
4.323
4.307
2.831
2.812
2.793
2.134
2.118
2.113
2.102
2.099
2.096
2.080
2.063
O
O
O
MeO2C
2.00
1.00
2.07
1.02
1.03
2.06
2.99
2.02
2.08
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)


MeO2C
O
O
O
166.863
158.515
146.437
146.206
144.502
129.678
128.311
127.958
117.772
111.697
63.844
51.840
32.074
29.755
f1 (ppm)

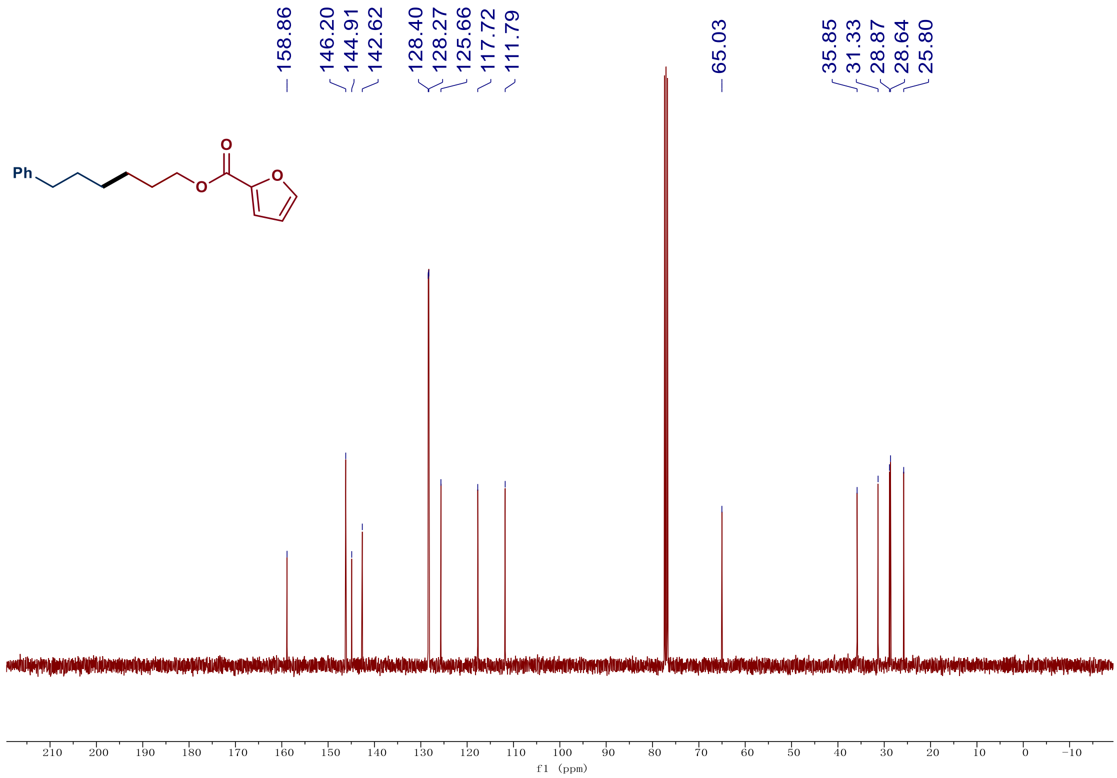

Ph
O
7.291
7.271
7.253
7.242
7.190
7.177
7.172
7.157
3.954
3.943
3.925
3.914
3.382
3.377
3.353
3.348
3.323
3.319
2.613
2.594
2.574
1.653
1.642
1.633
1.624
1.614
1.604
1.594
1.570
1.565
1.559
1.480
1.317
1.301
1.291
1.276
1.260
1.241
2.07
2.97
1.97
1.98
2.01
4.03
1.05
4.11
f1 (ppm)

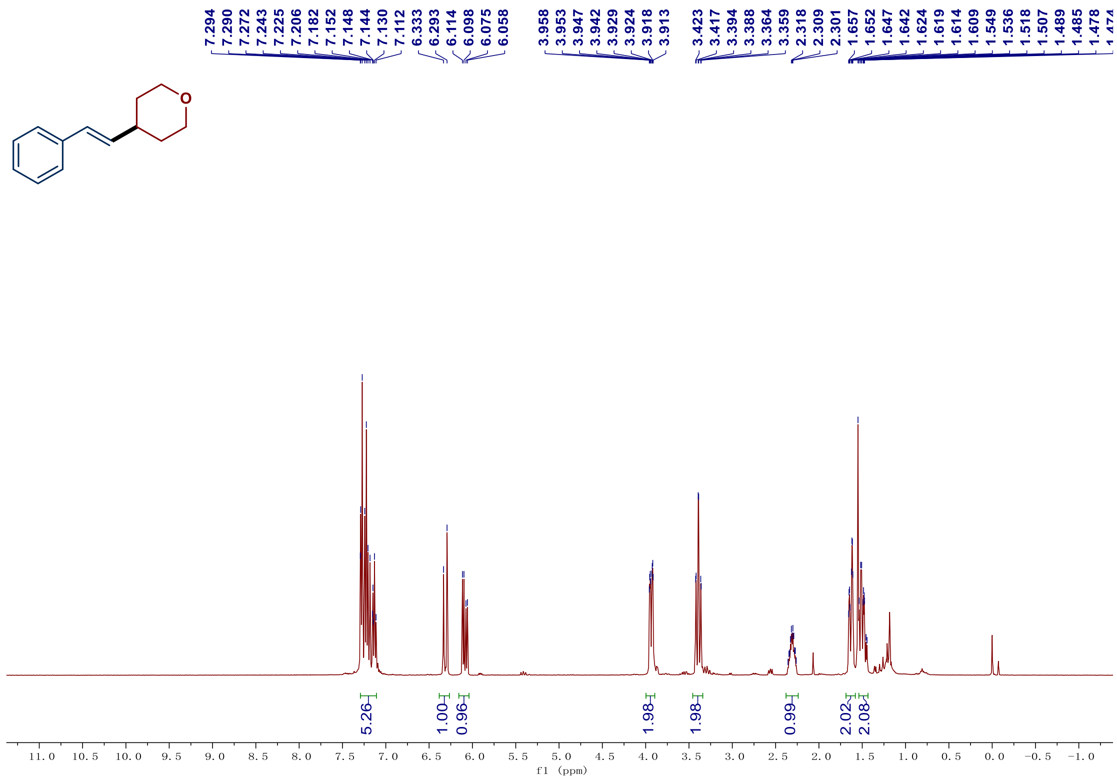


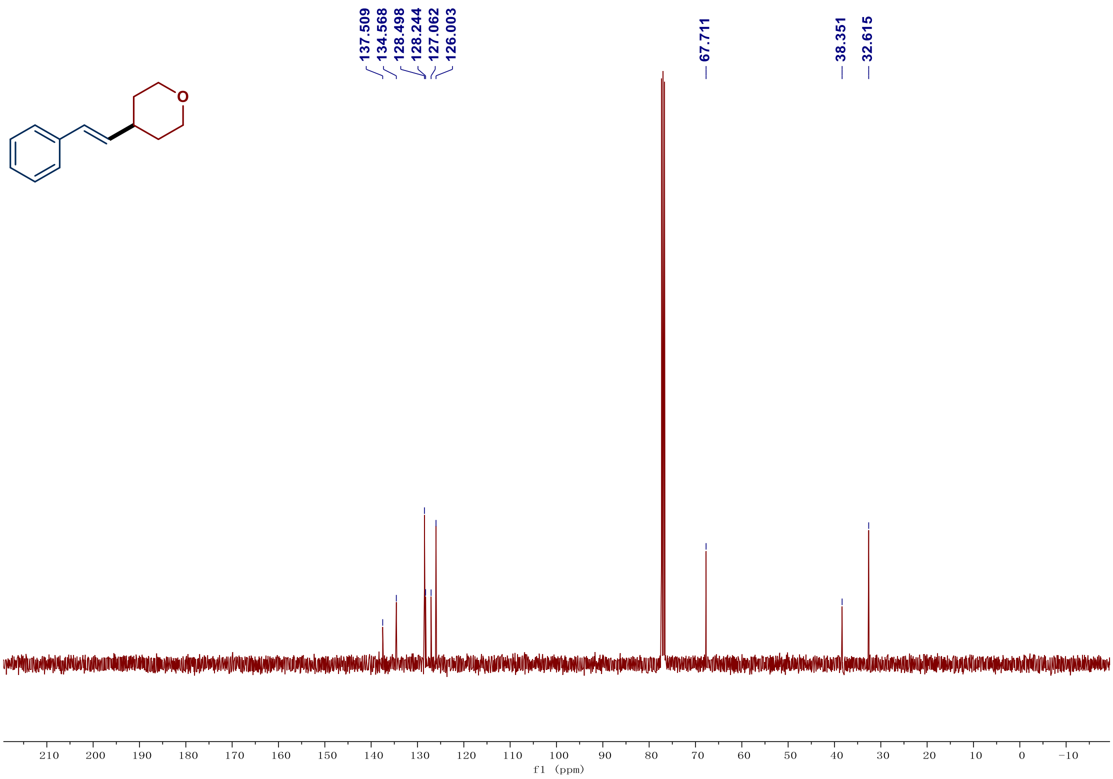
142.55
128.30
128.22
125.63
68.10
36.52
36.06
34.88
33.13
28.25
Ph
O
f1 (ppm)

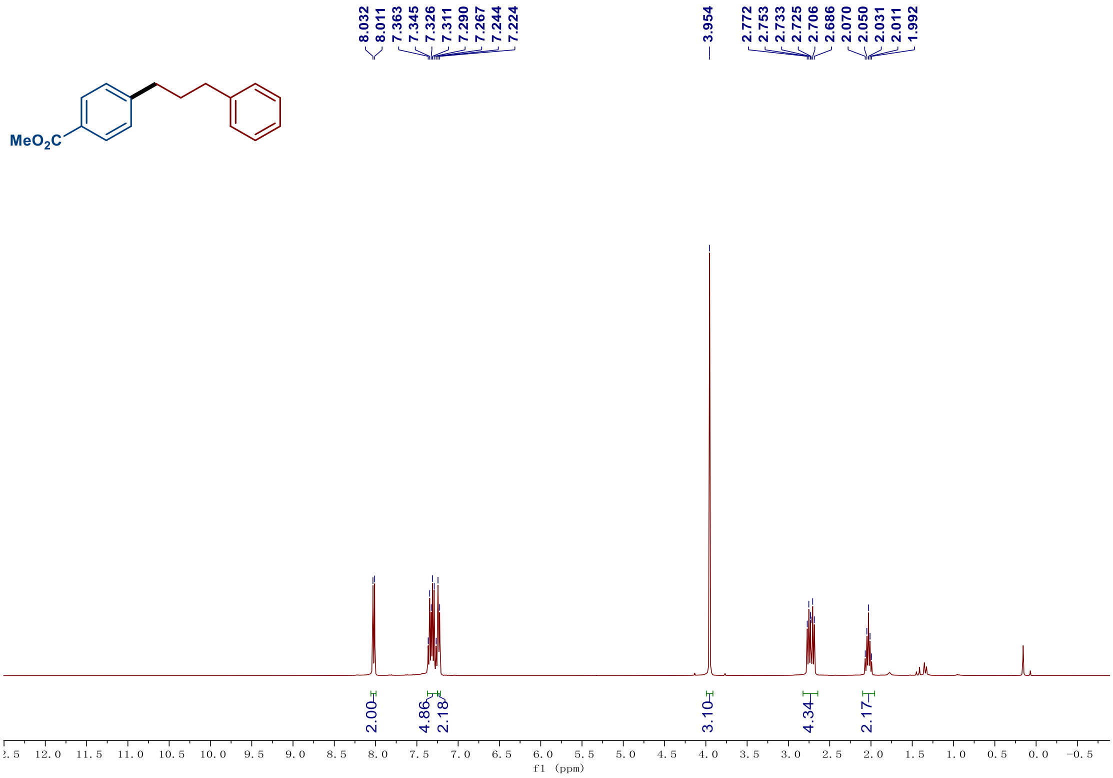


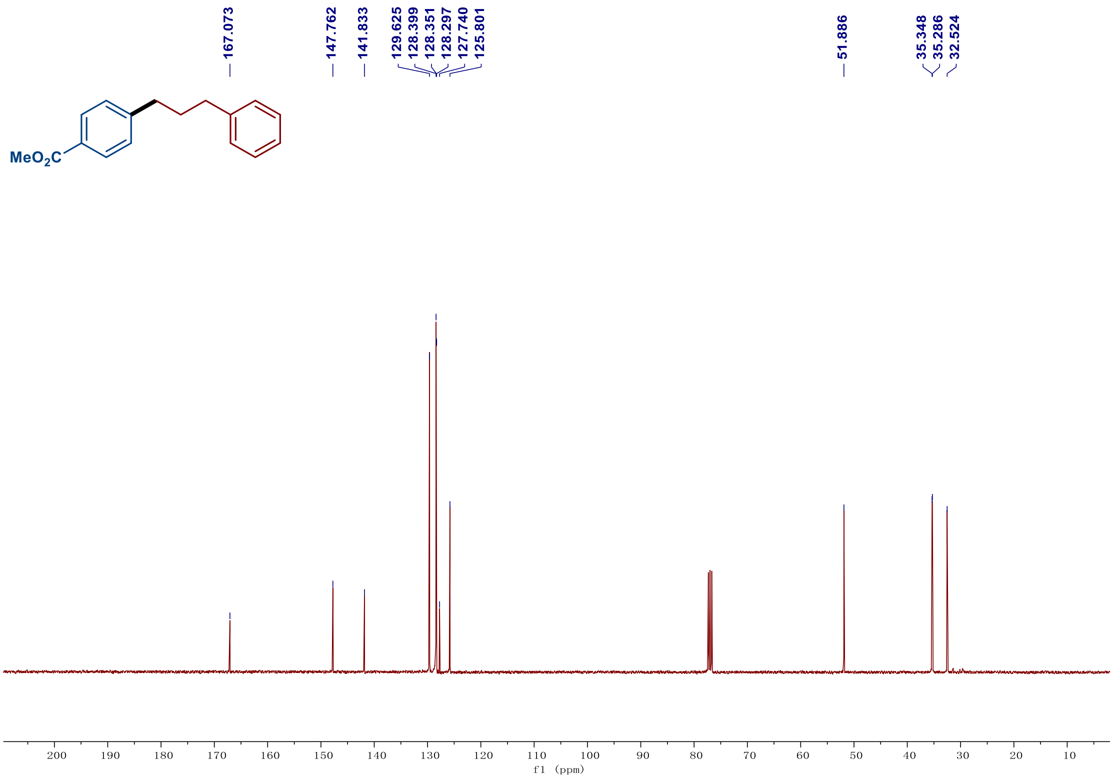
7.53
7.20
7.20
7.18
7.17
7.10
7.07
6.42
6.41
6.41
6.40
4.22
4.21
4.19
2.55
2.53
2.51
1.68
1.66
1.64
1.58
1.56
1.54
1.39
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
Ph
O
O
O
0.96
2.11
4.00
0.98
2.08
2.08
2.18
2.30
4.21
f1 (ppm)


158.86
146.20
144.91
142.62
128.40
128.27
125.66
117.72
111.79
65.03
35.85
31.33
28.87
28.64
25.80
Ph
O
O
O
f1 (ppm)

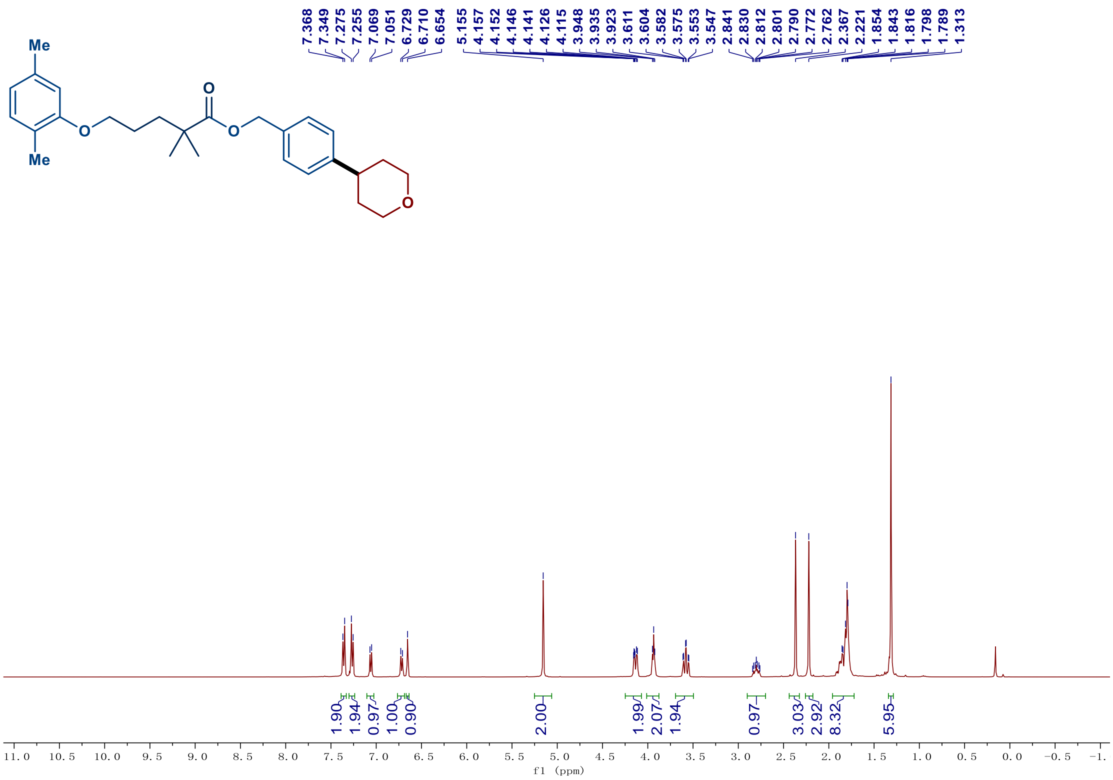

7.294
7.290
7.272
7.243
7.225
7.206
7.182
7.152
7.148
7.144
7.130
7.112
6.333
6.293
6.114
6.098
6.075
6.058
3.958
3.953
3.947
3.942
3.929
3.924
3.918
3.913
3.423
3.417
3.394
3.388
3.364
3.359
2.318
2.309
2.301
1.657
1.652
1.647
1.642
1.624
1.619
1.614
1.609
1.549
1.536
1.518
1.507
1.489
1.485
1.478
O
5.26
1.00
0.96
1.98
1.98
0.99
2.02
2.08
f1 (ppm)


137.509
134.568
128.498
128.244
127.062
126.003
67.711
38.351
32.615
O
f1 (ppm)

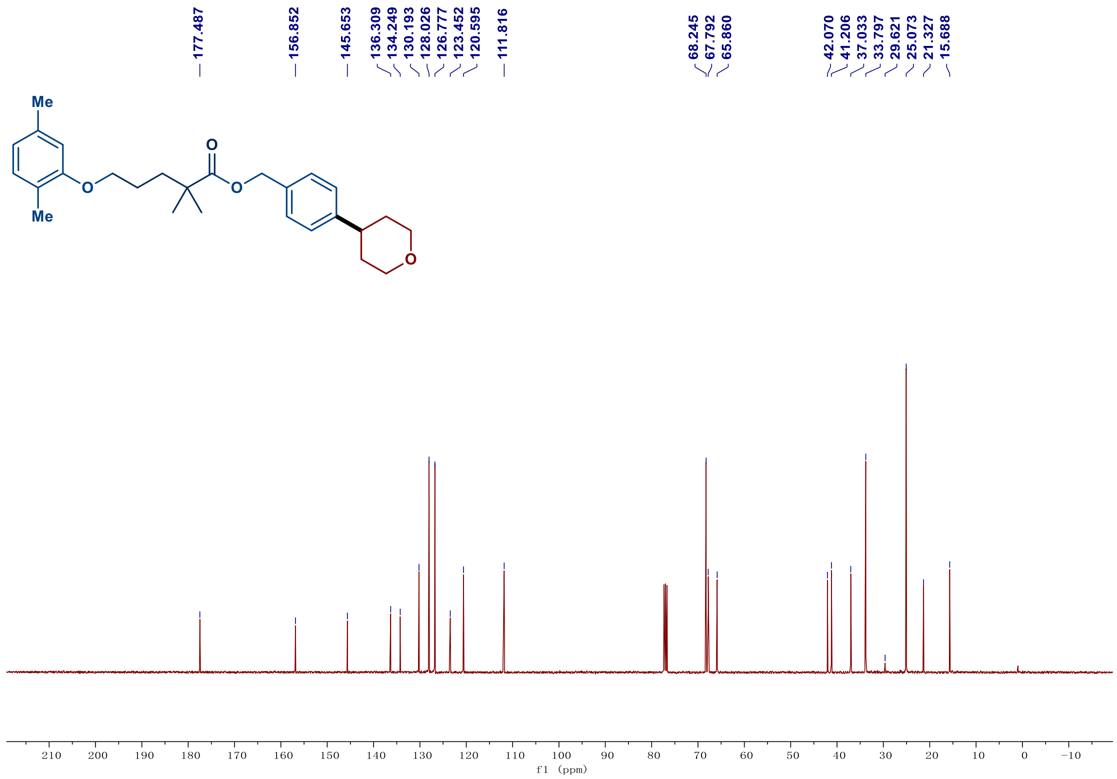

8.032
8.011
7.363
7.345
7.326
7.311
7.290
7.267
7.244
7.224
3.954
2.772
2.753
2.733
2.725
2.706
2.686
2.070
2.050
2.031
2.011
1.992
MeO$_2$C
2.00
4.86
2.18
3.10
4.34
2.17
f1 (ppm)

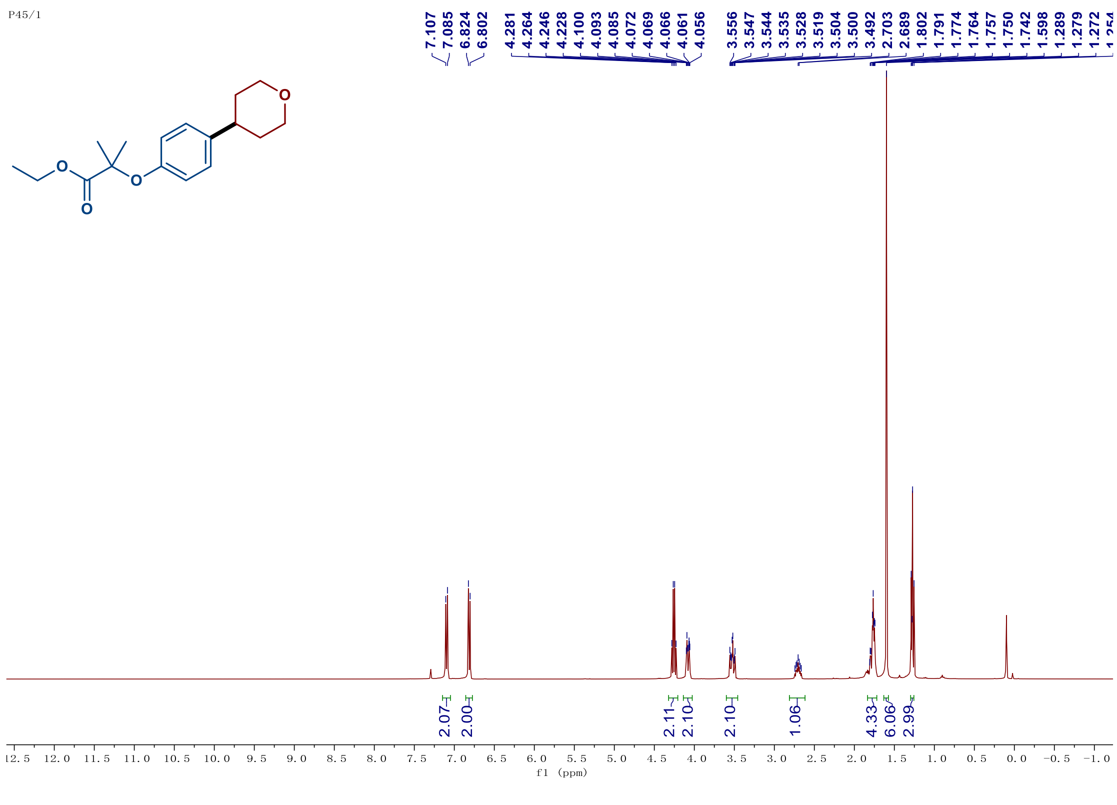


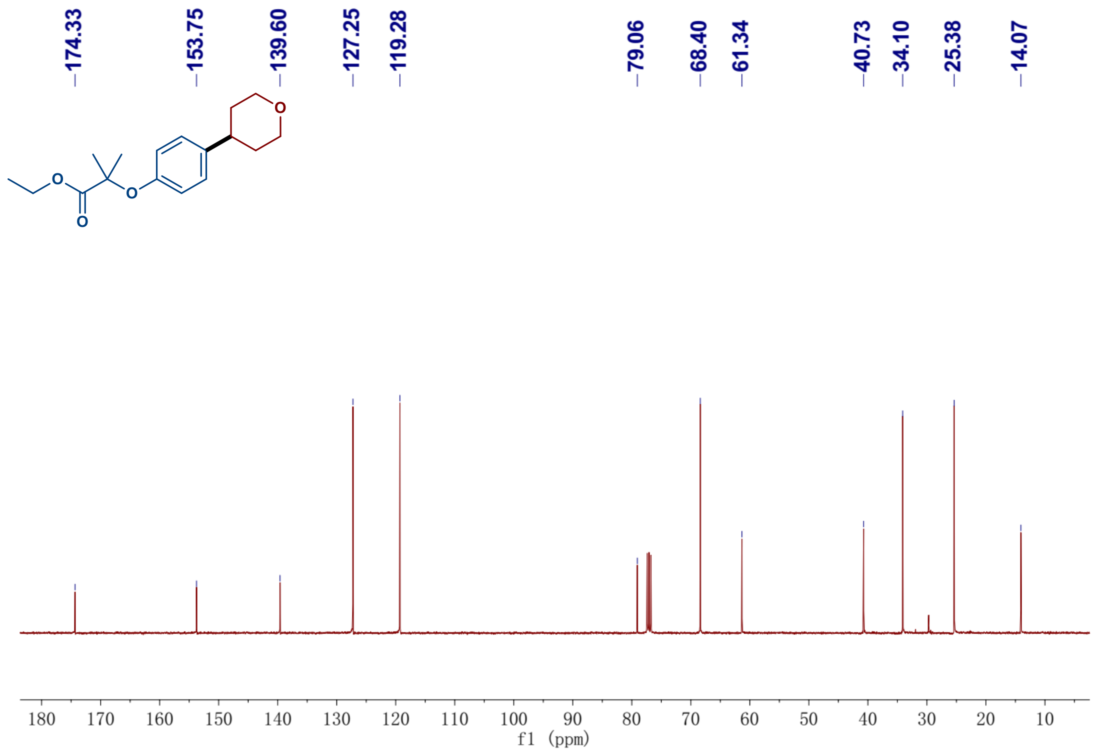
MeO2C
167.073
147.762
141.833
129.625
128.399
128.351
128.297
127.740
125.801
51.886
35.348
35.286
32.524
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



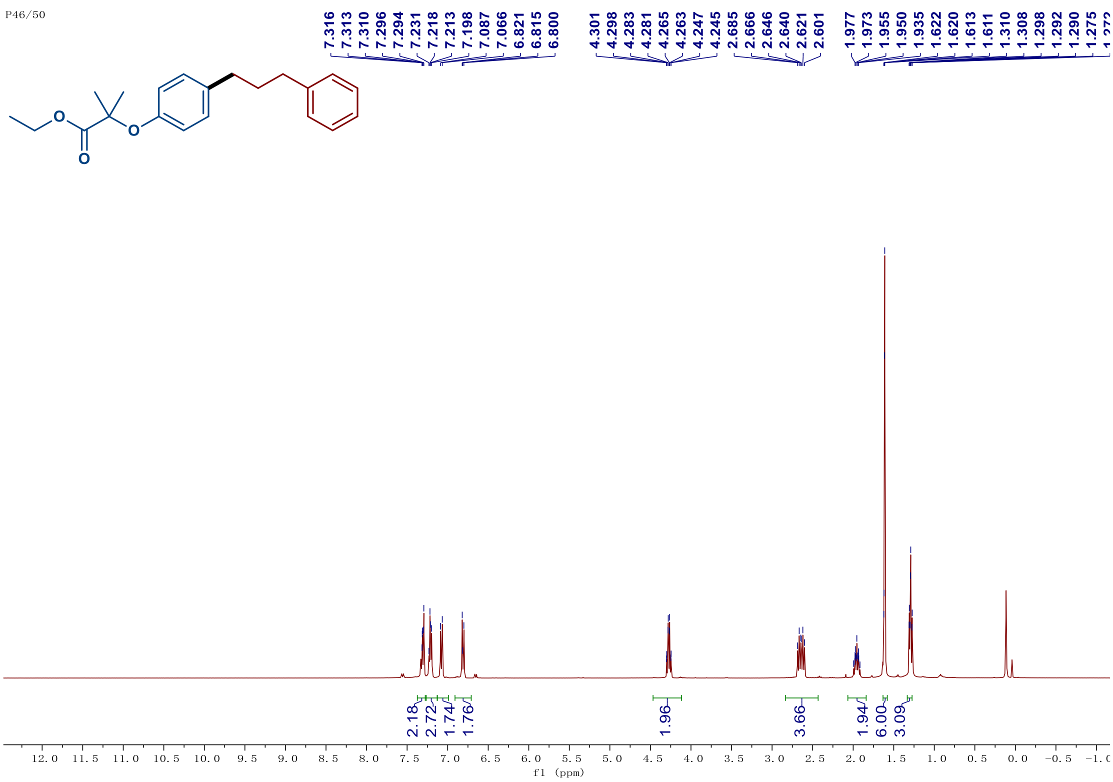
Me
O
Me
O
O
O
7.368
7.349
7.275
7.255
7.069
7.051
6.729
6.710
6.654
5.155
4.157
4.152
4.146
4.141
4.126
4.115
3.948
3.935
3.923
3.611
3.604
3.582
3.575
3.553
3.547
2.841
2.830
2.812
2.801
2.790
2.772
2.762
2.367
2.221
1.854
1.843
1.816
1.798
1.789
1.313
1.90
1.94
0.97
1.00
0.90
2.00
1.99
2.07
1.94
0.97
3.03
2.92
8.32
5.95
11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5 -1.
f1 (ppm)


177.487
156.852
145.653
136.309
134.249
130.193
128.026
126.777
123.452
120.595
111.816
68.245
67.792
65.860
42.070
41.206
37.033
33.797
29.621
25.073
21.327
15.688
Me
O
O
O
Me
O
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)

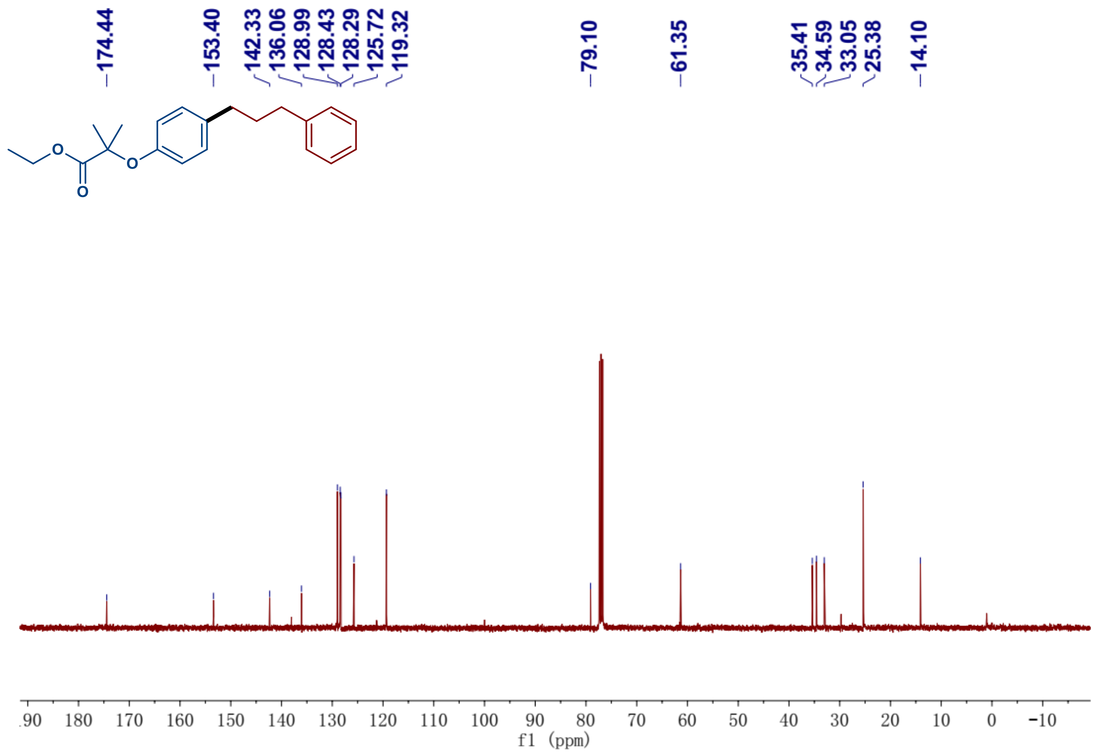

P45/1
7.107
7.085
6.824
6.802
4.281
4.264
4.246
4.228
4.100
4.093
4.085
4.072
4.069
4.066
4.061
4.056
3.556
3.547
3.544
3.535
3.528
3.519
3.504
3.500
3.492
2.703
2.689
1.802
1.791
1.774
1.764
1.757
1.750
1.742
1.598
1.289
1.279
1.272
O
O
O
O
2.07
2.00
2.11
2.10
2.10
1.06
4.33
6.06
2.99
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5 -1.0
f1 (ppm)

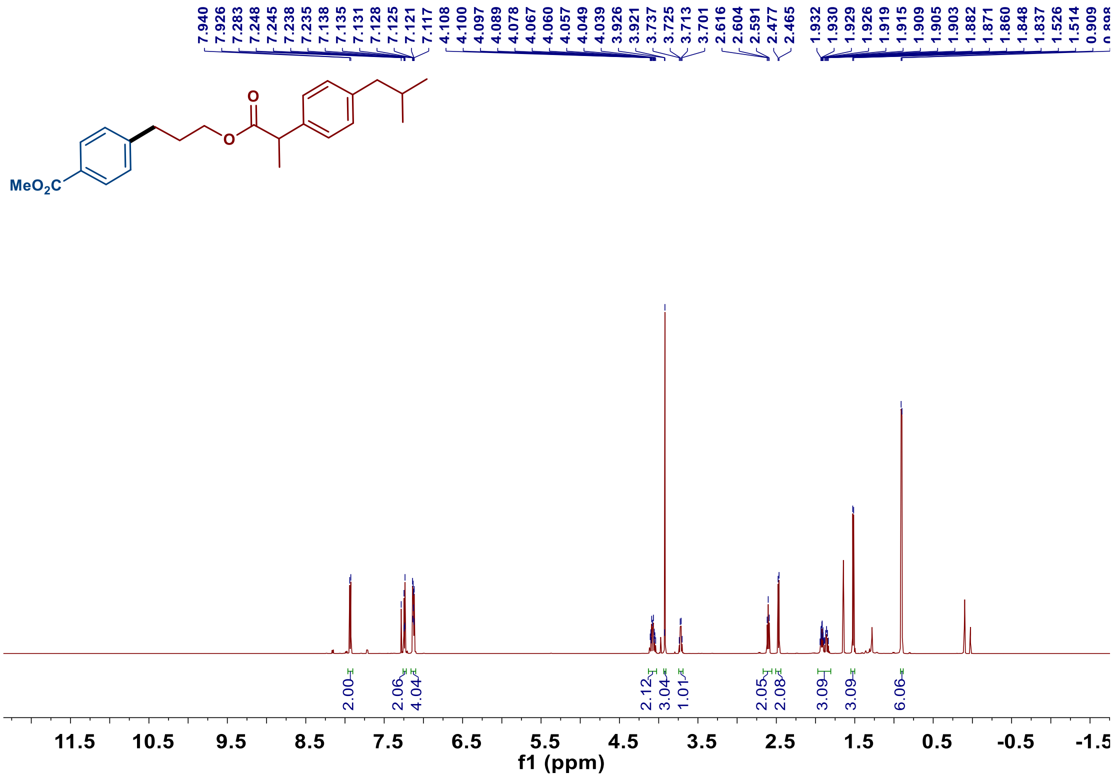


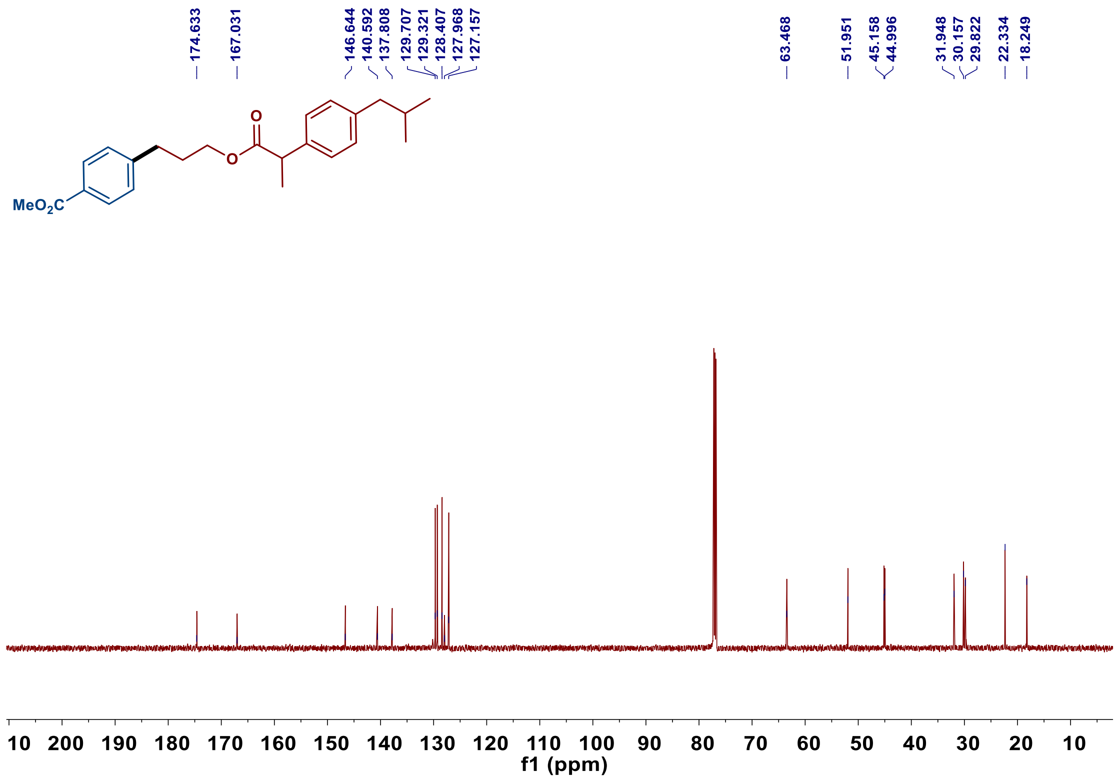
174.33
153.75
139.60
127.25
119.28
79.06
68.40
61.34
40.73
34.10
25.38
14.07
O
O
O
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

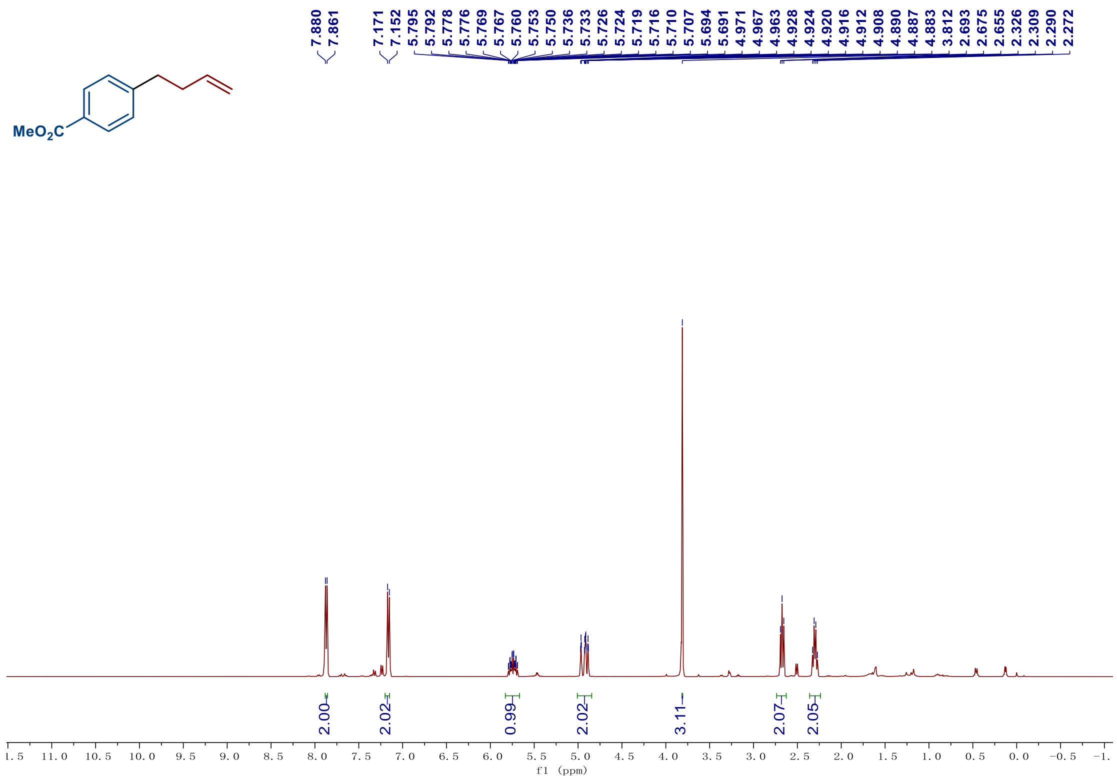


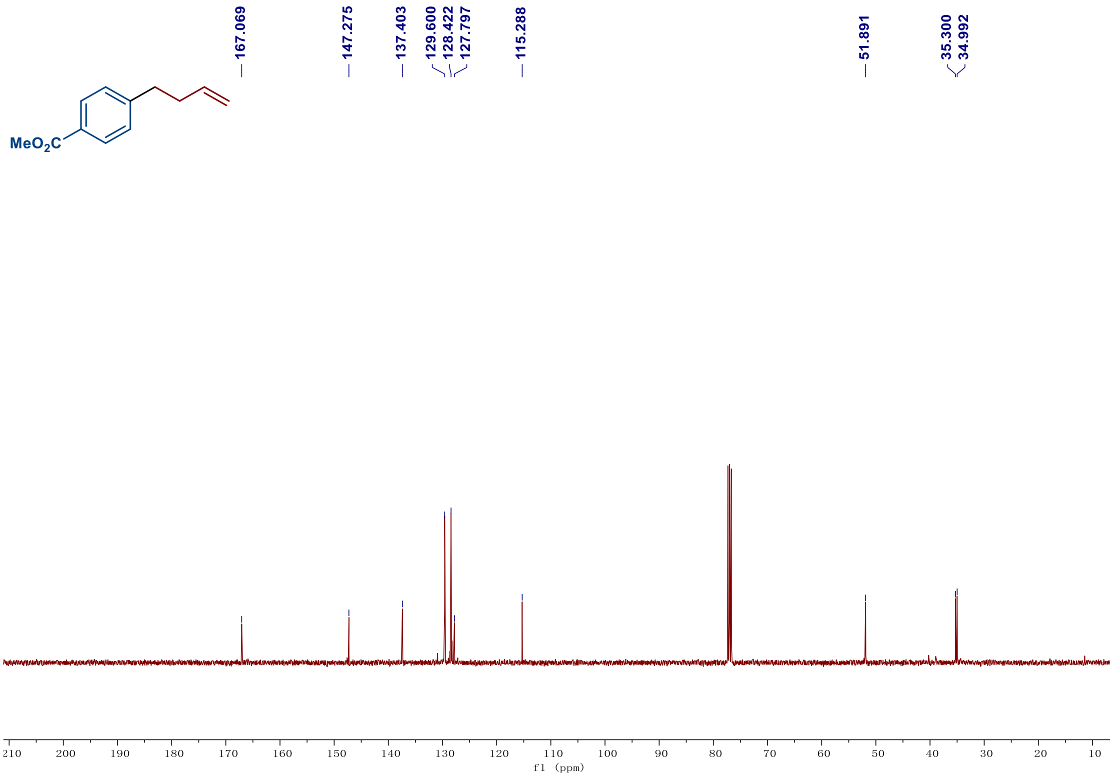
P46/50
7.316
7.313
7.310
7.296
7.294
7.231
7.218
7.213
7.198
7.087
7.066
6.821
6.815
6.800
4.301
4.298
4.283
4.281
4.265
4.263
4.247
4.245
2.685
2.666
2.646
2.640
2.621
2.601
1.977
1.973
1.955
1.950
1.935
1.622
1.620
1.613
1.611
1.310
1.308
1.298
1.292
1.290
1.275
O
O
O
2.18
2.72
1.74
1.76
1.96
3.66
1.94
6.00
3.09
12.0 11.5 11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5
f1 (ppm)


174.44
153.40
142.33
136.06
128.99
128.43
128.29
125.72
119.32
79.10
61.35
35.41
34.59
33.05
25.38
14.10
O
O
O
f1 (ppm)


7.940
7.926
7.283
7.248
7.245
7.238
7.235
7.138
7.135
7.131
7.128
7.125
7.121
7.117
4.108
4.100
4.097
4.089
4.078
4.067
4.060
4.057
4.049
4.039
3.926
3.921
3.737
3.725
3.713
3.701
2.616
2.604
2.591
2.477
2.465
1.932
1.930
1.929
1.926
1.919
1.915
1.909
1.905
1.903
1.882
1.871
1.860
1.848
1.837
1.526
1.514
0.909
MeO2C
O
O
2.00
2.06
4.04
2.12
3.04
1.01
2.05
2.08
3.09
3.09
6.06
f1 (ppm)


174.633
167.031
146.644
140.592
137.808
129.707
129.321
128.407
127.968
127.157
63.468
51.951
45.158
44.996
31.948
30.157
29.822
22.334
18.249
MeO$_2$C
O
O
f1 (ppm)


7.880
7.861
7.171
7.152
5.795
5.792
5.778
5.776
5.769
5.767
5.760
5.753
5.750
5.736
5.733
5.726
5.724
5.719
5.716
5.710
5.707
5.694
5.691
4.971
4.967
4.963
4.928
4.924
4.920
4.916
4.912
4.908
4.890
4.887
4.883
3.812
2.693
2.675
2.655
2.326
2.309
2.290
2.272
MeO$_2$C
2.00
2.02
0.99
2.02
3.11
2.07
2.05
f1 (ppm)


167.069
147.275
137.403
129.600
128.422
127.797
115.288
51.891
35.300
34.992
MeO2C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

10.References

[1] Campbell P S, Jamieson C, Simpson I, et al. Chemical Communications, 2018, 54(1): 46-49.

[2] Knauber T, Chandrasekaran R, Tucker J W, et al. Organic Letters, 2017, 19(24): 6566-6569.

[3] Watanabe E, Chen Y, May O, et al. Chemistry-a European Journal, 2020, 26(1): 186-191.

[4] Chen H, Hu L, Ji W, et al. Acs Catalysis, 2018, 8(11): 10479-10485.

[5] Hofmayer M S, Hammann J M, Haas D, et al. Organic Letters, 2016, 18(24): 6456-6459.

[6] Martin-Montero R, Reddy Yatham V, Yin H, et al. Organic Letters, 2019, 21(8): 2947-2951.

[7] Abdiaj I, Huck L, Mateo J M, et al. Angewandte Chemie-International Edition, 2018, 57(40): 13231-13236.

[8] Zhang X, Macmillan D W C. Journal of the American Chemical Society, 2016, 138(42): 13862-13865.

[9] Suga T, Shimazu S, Ukaji Y. Organic Letters, 2018, 20(17): 5389-5392.

[10] Toriyama F, Cornella J, Wimmer L, et al. Journal of the American Chemical Society, 2016, 138(35): 11132-11135.

[11] Cao H, Jiang H, Feng H, et al. Journal of the American Chemical Society, 2018, 140(47): 16360-16367.

[12] Peng L, Li Y, Li Y, et al. Acs Catalysis, 2018, 8(1): 310-313.