Supplementary Information (SI) for Catalysis Science & Technology.

Supporting Information

Oxidation of α-Hydroxyl Acetophenone Promoted by Sulfone-based Iron Site on Hyper Cross-linked Polymers

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Content

1. Materials and General Methods

Unless specially indicated, all chemical reagents were purchased from commercial sources and were used as received without further purification. Fourier transform infrared spectroscopy (FT-IR) spectra were recorded on a Bruker INVENIO-R FT-IR spectrophotometer. Scanning electron microscopy (SEM) and was carried out on an VEGA3 TESCAN at 15 kV. N_2 sorption curves were obtained by Micromeritics ASAP 2420-4MP Plus automated sorption analyzer under 77 K. Thermogravimetric analysis (TGA) was performed using Pyris1 TGA in N_2 atmosphere by heating from room temperature to 600 ℃. The X-ray photoelectron spectra (XPS) were obtained with the Thermo Scientific K-Alpha. ICP-OES results were obtained with the iCAP7200plus. EPR results were obtained with the EMXmicro-6/1/P/L.LC-MS results were obtained with the Orbitrap LC/MS (Q Exactive). ^{1}H and ^{13}C NMR spectra of organic compounds were recorded on Bruker 400. Chemical shifts are expressed in ppm relative to Me_4Si in $CDCl_3$ or DMSO-d_6.

2. Experimental Section

2.1 Synthesis of monomers

The synthesis of monomer **DDS** is described as a representative example. Anhydrous sodium acetate (110 mmol) and dapsone (10 mmol) were added to a 100 mL round-bottom flask containing 40 mL of glacial acetic acid. Subsequently, 2,5-dimethoxytetrahydrofuran (20 mmol) dissolved in 10 mL of glacial acetic acid was added to the reaction mixture. The reaction was refluxed at 130 °C under air for 10 h. After cooling to room temperature, the reaction mixture was neutralized with saturated aqueous $NaHCO_3$ solution. The mixture was extracted with dichloromethane (DCM, 30 mL × 3), and the combined organic phases were dried over anhydrous sodium sulfate. After removal of the solvent under reduced pressure, the crude product was purified by column chromatography (eluent: dichloromethane/ethyl acetate = 2:1, v/v) to afford monomer DDS in 76% yield. Monomer **DDM** was synthesized in an analogous manner in 70% yield.

2.2 Synthesis of functional polymers

The synthesis of PDDS-PXC is described as a representative example. $FeCl_3$ (9 mmol), monomer DDS (3 mmol), and 1,2-dichloroethane (DCE, 20 mL) were sequentially added to a 100 mL round-bottom flask equipped with a magnetic stir bar, and the mixture was heated with stirring in an oil bath at 90 °C. *p*-Xylylene dichloride (PXC, 9 mmol) dissolved in 15 mL of DCE was rapidly added to the reaction mixture via a plastic dropper. After the addition was complete, the reaction was refluxed under air for 24 h. Upon cooling to room temperature, the resulting solid was collected by vacuum filtration. The solid was then transferred to a 100 mL round-bottom flask containing 20 mL of 2 M hydrochloric acid and stirred at 65 °C for 4 h. After cooling to room temperature, the solid was isolated by vacuum filtration and washed thoroughly with deionized water. The solid was subsequently subjected to Soxhlet extraction with ethanol at 115 °C for 24 h. The obtained solid was dried under vacuum at 70 °C for 10 h to afford PDDS-PXC. The remaining materials, PDDM-PXC, PDDS-FDA, and

PDDS-FDA-TfOH, were synthesized following the same procedure.

2.3 Synthesis of starting materials

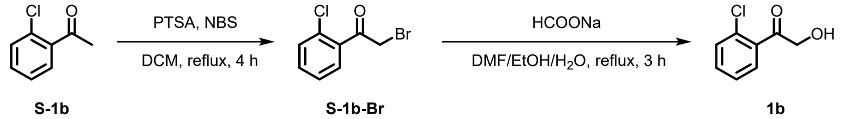


Figure S1 Method A for synthesis of starting materials.

Synthesis of substrates 1b–1g, 1n, and C2: The synthesis of **1b** is described as a representative example, and the synthetic route is illustrated in Figure S1. 2-Chloroacetophenone (**S-1b**, 10 mmol) was added to a round-bottom flask containing PTSA (1 mmol), *N*-bromo succinimide (NBS, 12 mmol), and DCM (20 mL). The reaction mixture was stirred under reflux at 60 °C for 4 h. After completion, the reaction was quenched with an excess of water and extracted with ethyl acetate (30 mL × 3). The combined organic phases were dried over anhydrous magnesium sulfate. After removal of the solvent under reduced pressure, the crude product was purified by column chromatography (eluent: petroleum ether/ethyl acetate = 10:1, v/v) to afford the brominated product **S-1b-Br**. Anhydrous sodium formate (15 mmol) was added to a round-bottom flask containing DMF/EtOH/H_2O (3 mL/9 mL/3 mL), followed by the addition of **S-1b-Br** (5 mmol). The reaction mixture was refluxed at 90 °C for 3 h. After cooling to room temperature, an excess of water was added, and the mixture was extracted with DCM (20 mL × 3). The combined organic phases were dried over anhydrous magnesium sulfate, concentrated under reduced pressure, and purified by column chromatography (eluent: petroleum ether/ethyl acetate = 10:1, v/v) to afford the target substrate **1b**. Substrates **1c**–**1g** and **1j** were synthesized following the same procedure.

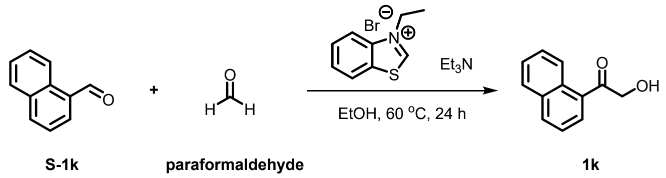


Figure S2 Method B for synthesis of starting materials.

Synthesis of substrates 1k–1m and 1s–1u: The synthesis of 1k is described as a representative example, and the synthetic route is illustrated in Figure S1. To a 25 mL round-bottom flask equipped with a magnetic stir bar were sequentially added paraformaldehyde (10 mmol), 3-ethylbenzothiazolium bromide (1 mmol), EtOH (10 mL), and triethylamine (Et_3N, 1 mmol). After thorough mixing, 1-naphthaldehyde (**S-1k**, 10 mmol) was added to the mixture. The reaction was carried out at 60 °C for 24 h, and the mixture was then cooled to room temperature. The solvent was removed by rotary evaporation, and the residue was dissolved in ethyl acetate (80 mL). The resulting solution was washed sequentially with deionized water (80 mL × 2) and saturated aqueous sodium chloride solution (80 mL × 2), and the organic phase was dried over anhydrous sodium sulfate. After concentration under reduced pressure, the crude product was purified by column chromatography (eluent: petroleum ether/ethyl acetate = 10:1, v/v) to afford the target substrate **1k**. Substrates **1l**–**1m** and **1s**–**1u** were synthesized following the same procedure.

2.4 Typical procedure of oxidation reaction

The synthesis of compound **2a** is described as a representative example. To a V-shaped reaction tube equipped with a triangular magnetic stir bar were added substrate **1a** (0.3 mmol), PDDS-PXC (15 mg), PTSA (0.15 mmol), and ^{n}BuOH (2 mL) as the solvent. The reaction was carried out at 80 °C under air atmosphere for 6 h. Upon completion as monitored by TLC, the mixture was cooled to room temperature, and the organic layer was collected by centrifugation. Purification by preparative thin-layer chromatography (PTLC, 20 cm × 20 cm; eluent: petroleum ether/ethyl acetate = 40:1, v/v) afforded the target product **2a** in 70% yield.

2.5 Characterization of HCPs

Table S1 Elements Analysis results.

Sample	C (%)	H (%)	N (%)	S (%)
PDDS-PXC	70.4	4.6	5.5	6.4
PDDM-PXC	78.5	5.5	5.2	0

Table S2 S_{BET} and Fe mass fraction of different materials.

Sample	S_{BET} (m^2/g)	Fe (wt%)
PDDS-PXC	103.5	0.45
PDDM-PXC	233.7	0.095
PDDS-PXC-used	30.2	0.02
PDDS-FDA-TfOH	94.2	0

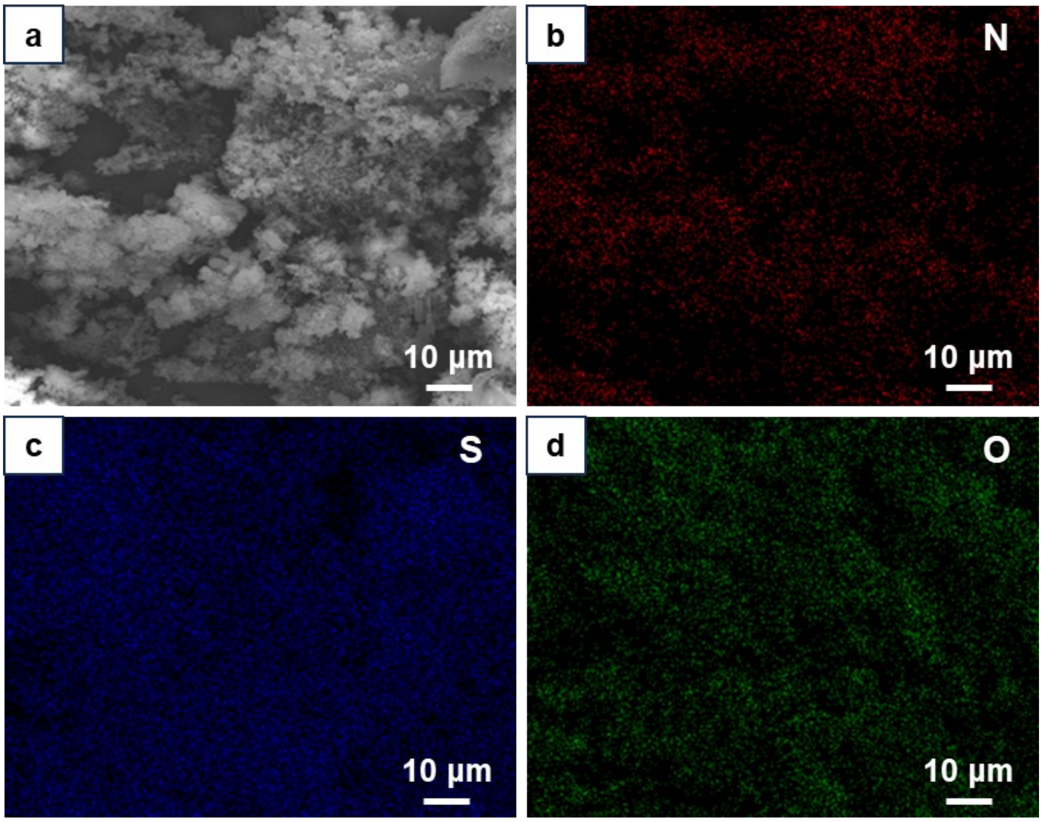


Figure S3 (a) SEM image of PDDS-PXC; (b–d) elemental mapping of PDDS-PXC for N (b), S (c), and O (d).

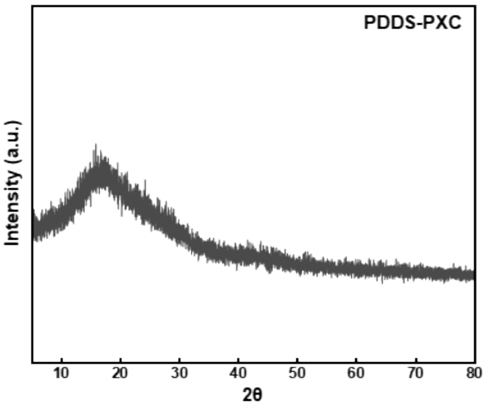
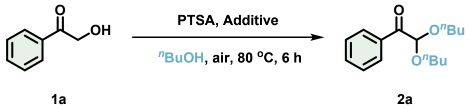


Figure S4 XRD patterns of PDDS-PXC.

2.6 Hot filtration reaction

Table S3 Verification of the heterogeneity.

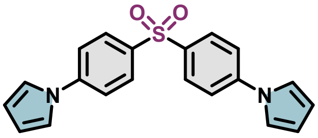


Entry [a]	Variations from standard conditions	Yield of **2a** [b]
1	none	70
2	1 h	30
3	Remove PDDS-PXC after 1 h, then reaction for 5 h	30

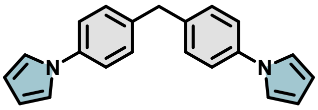
[a] Standard conditions: **1a** (0.3 mmol), PTSA (50 mol%), PDDS-PXC (15.0 mg), ^{n}BuOH (2.0 mL), air, 80 ℃, 6 h. [b] Isolated yield.

2.7. NMR data for some products

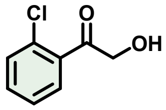
For previously reported compounds, ^{1}H NMR spectroscopic data are provided and compared with the literature values.



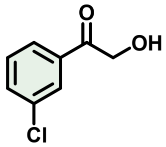
1,1'-(Sulfonyl bis(4,1-phenylene)) bis(1*H*-pyrrole) (DDS): white solid, yield 76%, ^{1}H NMR (400 MHz, DMSO) δ 8.04 (d, *J* = 8.7 Hz, 4H), 7.85 (d, *J* = 8.5 Hz, 4H), 7.52 (t, *J* = 2.3 Hz, 4H), 6.33 (t, *J* = 2.2 Hz, 4H). ^{13}C NMR (101 MHz, DMSO) δ 143.9, 137.5, 129.7, 120.0, 119.7, 112.3. IR: 1597.0, 1510.2, 1307.7, 1153.4, 1126.4, 1105.1, 1064.6, 1014.5, 920.0, 840.9, 827.4. HRMS (TOF, ESI): m/z calcd for $C_{20}H_{17}N_2O_2S^+$, $[M+H]^+$ = 349.1006, found 349.1006.



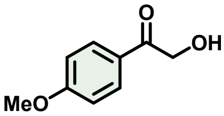
Bis(4-(1*H*-pyrrol-1-yl) phenyl) methane[1] (DDM): white solid, yield 80%, ^{1}H NMR (400 MHz, DMSO) δ 7.49 (d, *J* = 8.2 Hz, 4H), 7.36–7.29 (m, 8H), 6.24 (t, *J* = 2.1 Hz, 4H), 3.98 (s, 2H). ^{13}C NMR (101 MHz, DMSO) δ 138.8, 138.6, 130.3, 120.0, 119.4, 110.7, 40.0.



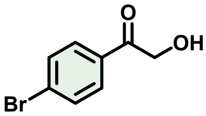
1-(2-Chlorophenyl)-2-hydroxyethan-1-one[2] **(1b):** yellow white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.7 Hz, 1H), 7.51–7.46 (m, 2H), 7.42–7.36 (m, 1H), 4.84 (d, *J* = 4.1 Hz, 2H), 3.46 (t, *J* = 4.9 Hz, 1H).



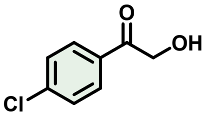
1-(3-Chlorophenyl)-2-hydroxyethan-1-one[2] **(1c):** white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 7.91 (t, *J* = 1.9 Hz, 1H), 7.80 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.61 (ddd, *J* = 8.0, 2.2, 1.1 Hz, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 4.87 (s, 2H), 3.42 (s, 1H).



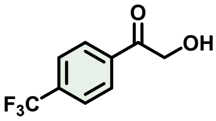
2-Hydroxy-1-(4-methoxyphenyl) ethan-1-one[2] **(1d):** yellow white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 7.94–7.88 (m, 2H), 7.02–6.93 (m, 2H), 4.83 (d, *J* = 4.6 Hz, 2H), 3.89 (s, 3H), 3.58 (t, *J* = 4.6 Hz, 1H).



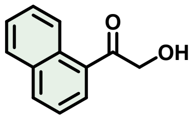
1-(4-Bromophenyl)-2-hydroxy ethan-1-one[2] **(1e):** white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.6 Hz, 2H), 7.66 (d, *J* = 8.6 Hz, 2H), 4.85 (d, *J* = 4.7 Hz, 2H), 3.45 (t, *J* = 4.8 Hz, 1H).



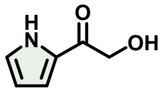
1-(4-Chlorophenyl)-2-hydroxyethan-1-one[2] **(1f):** white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 4.86 (d, *J* = 4.4 Hz, 2H), 3.46 (t, *J* = 4.7 Hz, 1H).



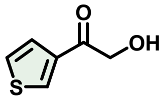
2-Hydroxy-1-(4-(trifluoromethyl) phenyl) ethan-1-one[2] **(1g):** white solid, ^1H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.1 Hz, 2H), 7.79 (d, *J* = 8.2 Hz, 2H), 4.92 (s, 2H), 3.42 (s, 1H).



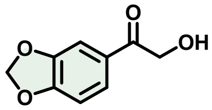
2-Hydroxy-1-(naphthalen-1-yl) ethan-1-one[3] **(1k):** white solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.88 (d, J = 8.6 Hz, 1H), 8.08 (d, J = 8.1 Hz, 1H), 7.92–7.87 (m, 2H), 7.66 (ddd, J = 8.5, 5.6, 1.5 Hz, 2H), 7.61–7.56 (m, 1H), 7.53 (t, J = 7.7 Hz, 1H), 4.92 (s, 2H), 3.71 (s, 1H).



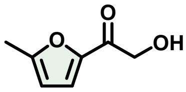
2-Hydroxy-1-(1*H*-pyrrol-2-yl) ethan-1-one[3] **(1l):** yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.48 (d, J = 1.2 Hz, 1H), 7.11 (dt, J = 5.8, 2.1 Hz, 1H), 6.26 (dd, J = 4.0, 2.5 Hz, 1H), 5.46 (d, J = 8.2 Hz, 2H), 5.30 (t, J = 8.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.38, 131.89, 131.48, 126.74, 110.02, 72.62. HRMS (TOF, ESI): m/z calcd for $C_6H_8NO_2^+$, $[M+H]^+$ = 126.0550, found 126.0552.



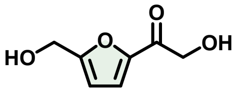
2-Hydroxy-1-(thiophen-3-yl) ethan-1-one[3] **(1m):** yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 3.8 Hz, 1H), 7.56 (d, J = 5.1 Hz, 1H), 7.40 (dd, J = 5.2, 2.9 Hz, 1H), 4.77 (d, J = 3.5 Hz, 2H), 3.43 (t, J = 4.8 Hz, 1H).



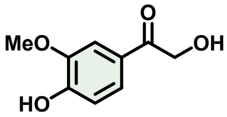
1-(1,3-Benzodioxol-5-yl)-2-hydroxyethanone[2] **(1n):** white solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, J = 8.2, 1.8 Hz, 1H), 7.41 (d, J = 1.8 Hz, 1H), 6.89 (d, J = 8.1 Hz, 1H), 6.08 (s, 2H), 4.82–4.77 (m, 2H), 3.54 (t, J = 4.6 Hz, 1H).



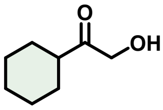
2-Hydroxy-1-(5-methyl-2-furanyl) ethanone[3] **(1s):** brown solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, J = 3.5 Hz, 1H), 6.22 (d, J = 3.5 Hz, 1H), 4.69 (d, J = 3.6 Hz, 2H), 3.36 (t, J = 4.9 Hz, 1H), 2.42 (s, 3H).



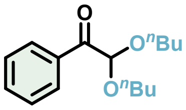
2-Hydroxy-1-[5-(hydroxymethyl)-2-furanyl] ethanone[3] (1t): orange solid, ^{1}H NMR (400 MHz, MeOD) δ 7.34 (d, *J* = 3.7 Hz, 1H), 6.53 (d, *J* = 3.7 Hz, 1H), 4.68 (s, 2H), 4.59 (s, 2H).



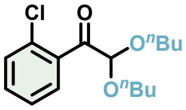
2-Hydroxy-1-(4-hydroxy-3-methoxyphenyl) ethanone[3] (1u): brownish solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.5 Hz, 2H), 6.98 (dd, *J* = 7.6, 1.1 Hz, 1H), 6.16 (s, 1H), 4.41 (s, 2H), 3.97 (s, 3H).



1-Cyclohexyl-2-hydroxyethanone[2] (C2): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.30 (d, *J* = 2.8 Hz, 2H), 3.22 (d, *J* = 4.8 Hz, 1H), 2.39 (tt, *J* = 11.6, 3.4 Hz, 1H), 1.87–1.77 (m, 4H), 1.73–1.63 (m, 1H), 1.48–1.15 (m, 5H).

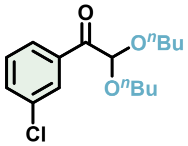


2,2-Dibutoxy-1-phenylethan-1-one (2a): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 5.23 (d, *J* = 1.4 Hz, 1H), 3.70 (dt, *J* = 9.5, 6.7 Hz, 2H), 3.57 (dt, *J* = 9.5, 6.6 Hz, 2H), 1.67–1.54 (m, 4H), 1.36 (h, *J* = 7.4 Hz, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.2, 133.8, 133.4, 129.8, 128.3, 103.1, 77.4, 77.0, 76.7, 67.6, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{25}O_3^+$, $[M+H]^+$ = 265.1799, found 265.1803.

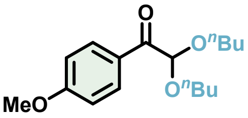


2,2-Dibutoxy-1-(2-chlorophenyl) ethan-1-one (2b): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.45–7.34 (m, 2H), 7.31 (ddd, *J* = 7.6, 6.6, 2.0 Hz, 1H), 5.26 (s, 1H), 3.68 (dt, *J* = 9.4, 6.6 Hz, 2H), 3.58 (dt, *J* = 9.4, 6.6 Hz, 2H), 1.63–1.51 (m, 4H), 1.33 (h, *J* = 7.4 Hz, 4H), 0.88 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz,

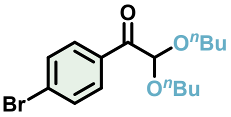
$CDCl_3$) δ 197.2, 136.4, 131.9, 131.8, 130.3, 130.0, 126.4, 102.4, 67.7, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{24}ClO_3^+$, $[M+H]^+$ = 299.1409, found 299.1409.



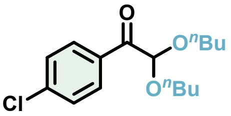
2,2-Dibutoxy-1-(3-chlorophenyl) ethan-1-one (2c): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (t, *J* = 1.8 Hz, 1H), 8.05 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.53 (ddd, *J* = 7.9, 2.2, 1.1 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 5.14 (s, 1H), 3.71 (dt, *J* = 9.4, 6.6 Hz, 2H), 3.56 (dt, *J* = 9.5, 6.5 Hz, 2H), 1.66–1.54 (m, 4H), 1.44–1.29 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.0, 135.2, 134.5, 133.3, 129.9, 129.6, 128.0, 103.5, 67.9, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{24}ClO_3^+$, $[M+H]^+$ = 299.1409, found 299.1409.



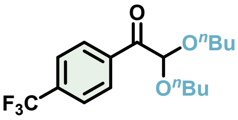
2,2-Dibutoxy-1-(4-methoxyphenyl) ethan-1-one (2d): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 5.18 (s, 1H), 3.87 (s, 3H), 3.69 (dt, *J* = 9.4, 6.7 Hz, 2H), 3.56 (dt, *J* = 9.3, 6.5 Hz, 2H), 1.59 (p, *J* = 6.7 Hz, 4H), 1.36 (h, *J* = 7.4 Hz, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.8, 163.7, 132.2, 126.7, 113.5, 103.4, 77.4, 77.1, 76.7, 67.5, 55.4, 31.7, 19.3, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{17}H_{27}O_4^+$, $[M+H]^+$ = 295.1904, found 295.1904.



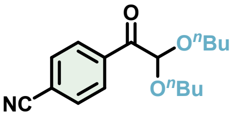
1-(4-Bromophenyl)-2,2-dibutoxyethan-1-one (2e): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.7 Hz, 2H), 7.59 (d, *J* = 8.5 Hz, 2H), 5.11 (s, 1H), 3.71 (dt, *J* = 9.4, 6.7 Hz, 2H), 3.55 (dt, *J* = 9.4, 6.5 Hz, 2H), 1.63–1.53 (m, 4H), 1.41–1.30 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.3, 132.3, 131.6, 131.5, 128.7, 103.9, 68.0, 31.7, 19.2, 13.7. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{24}BrO_3^+$, $[M+H]^+$ = 343.0904., found 343.0905.



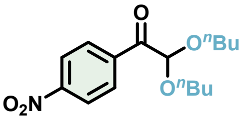
2,2-Dibutoxy-1-(4-chlorophenyl) ethan-1-one (2f): yellow oil, ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.11 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.4 Hz, 2H), 5.12 (s, 1H), 3.70 (dt, J = 9.4, 6.7 Hz, 2H), 3.54 (dt, J = 9.5, 6.5 Hz, 2H), 1.64–1.51 (m, 4H), 1.34 (h, J = 7.4 Hz, 4H), 0.88 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 193.2, 139.9, 131.9, 131.4, 128.6, 103.8, 77.3, 77.0, 76.7, 68.0, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{24}ClO_3^+$, $[M+H]^+$ = 299.1409, found 299.1409.



2,2-Dibutoxy-1-(4-(trifluoromethyl) phenyl) ethan-1-one (2g): yellow oil, ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.29 (d, J = 8.1 Hz, 2H), 7.71 (d, J = 8.2 Hz, 2H), 5.13 (s, 1H), 3.74 (dt, J = 9.4, 6.6 Hz, 2H), 3.57 (dt, J = 9.4, 6.5 Hz, 2H), 1.66–1.54 (m, 4H), 1.43–1.29 (m, 4H), 0.89 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 193.3, 136.3, 134.5 (q, J = 32.7 Hz), 130.3, 125.2 (q, J = 3.8 Hz), 123.7 (q, J = 273.7 Hz), 104.1, 68.2, 31.7, 19.2, 13.7. ^{19}F NMR (376 MHz, CDCl$_3$) δ -63.2. HRMS (TOF, ESI): m/z calcd for $C_{17}H_{24}F_3O_3^+$, $[M+H]^+$ = 333.1673, found 333.1676.

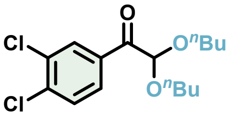


4-(2,2-Dibutoxyacetyl) benzonitrile (2h): yellow oil, ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.26 (d, J = 8.2 Hz, 2H), 7.74 (d, J = 8.3 Hz, 2H), 5.08 (s, 1H), 3.74 (dt, J = 9.3, 6.6 Hz, 2H), 3.55 (dt, J = 9.4, 6.5 Hz, 2H), 1.65–1.53 (m, 4H), 1.35 (h, J = 7.4 Hz, 4H), 0.89 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 193.0, 136.6, 132.0, 130.4, 118.1, 116.4, 104.3, 68.5, 31.7, 19.2, 13.7. HRMS (TOF, ESI): m/z calcd for $C_{17}H_{24}NO_3^+$, $[M+H]^+$ = 290.1751, found 290.1751.

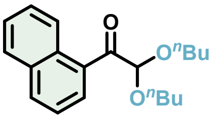


2,2-Dibutoxy-1-(4-nitrophenyl) ethan-1-one (2i): yellow oil, ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.38–8.25 (m, 4H), 5.09 (s, 1H), 3.76 (dt, J = 9.4, 6.6 Hz, 2H), 3.57 (dt, J = 9.4, 6.5 Hz, 2H), 1.65–1.54 (m, 4H), 1.42–1.27 (m, 4H), 0.89 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 192.8, 150.4, 138.1, 131.1, 123.4, 104.5, 68.6, 31.7, 19.2, 13.7. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{24}NO_5^+$, $[M+H]^+$ = 310.1649, found

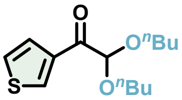
310.1647.



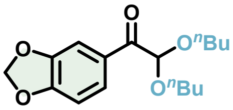
2,2-Dibutoxy-1-(3,4-dichlorophenyl) ethan-1-one (2j): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, *J* = 1.9 Hz, 1H), 8.02 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 1H), 5.07 (s, 1H), 3.72 (dt, *J* = 9.3, 6.6 Hz, 2H), 3.55 (dt, *J* = 9.4, 6.5 Hz, 2H), 1.65–1.54 (m, 4H), 1.41–1.29 (m, 4H), 0.90 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.2, 137.9, 133.0, 132.9, 132.0, 130.4, 129.1, 104.1, 68.2, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{23}Cl_2O_3^+$, $[M+H]^+$ = 333.1019, found 333.1019.



2,2-Dibutoxy-1-(naphthalen-1-yl) ethan-1-one (2k): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75–8.68 (m, 1H), 8.31 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.86 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.59 (ddd, *J* = 8.6, 6.8, 1.5 Hz, 1H), 7.55–7.46 (m, 2H), 5.31 (s, 1H), 3.73 (dt, *J* = 9.4, 6.6 Hz, 2H), 3.61 (dt, *J* = 9.4, 6.5 Hz, 2H), 1.66–1.53 (m, 4H), 1.42–1.27 (m, 4H), 0.86 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 133.9, 133.3, 131.6, 131.0, 130.1, 128.6, 128.1, 126.3, 125.6, 124.2, 103.5, 77.4, 77.1, 76.8, 67.6, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{20}H_{27}O_3^+$, $[M+H]^+$ = 315.1955, found 315.1958.

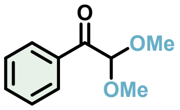


2,2-Dibutoxy-1-(thiophen-3-yl) ethan-1-one (2m): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.66 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.28 (dd, *J* = 5.0, 2.8 Hz, 1H), 5.04 (s, 1H), 3.69 (dt, *J* = 9.4, 6.7 Hz, 2H), 3.56 (dt, *J* = 9.4, 6.5 Hz, 2H), 1.66–1.53 (m, 4H), 1.44–1.30 (m, 4H), 0.90 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.1, 138.0, 135.1, 128.0, 125.4, 103.6, 67.6, 31.7, 19.3, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{14}H_{23}O_3S^+$, $[M+H]^+$ = 271.1363, found 271.1363.

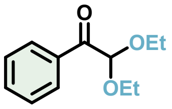


1-(1,3-Benzodioxol-5-yl)-2,2-dibutoxy ethanone (2n): yellow oil, ^{1}H NMR (400

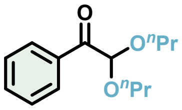
MHz, $CDCl_3$) δ 7.84 (dd, $J = 8.3, 1.7$ Hz, 1H), 7.62 (d, $J = 1.7$ Hz, 1H), 6.85 (d, $J = 8.2$ Hz, 1H), 6.04 (s, 2H), 5.14 (s, 1H), 3.69 (dt, $J = 9.4, 6.7$ Hz, 2H), 3.55 (dt, $J = 9.4, 6.6$ Hz, 2H), 1.65–1.52 (m, 4H), 1.37 (dt, $J = 14.9, 7.4$ Hz, 4H), 0.89 (t, $J = 7.4$ Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.4, 152.0, 147.7, 128.3, 126.6, 109.5, 107.9, 103.5, 101.7, 67.7, 31.7, 19.2, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{17}H_{25}O_5^+$, $[M+H]^+$ = 309.1697, found 309.1697.



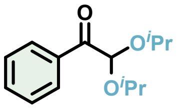
2,2-Dimethoxy-1-phenylethan-1-one[4] (2o): yellow oil, ^1H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, $J = 8.2, 1.5$ Hz, 2H), 7.63–7.54 (m, 1H), 7.47 (t, $J = 7.7$ Hz, 2H), 5.24 (s, 1H), 3.48 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.5, 133.8, 133.7, 129.5, 128.5, 103.3, 54.6.



2,2-Diethoxy-1-phenylethan-1-one[5] (2p): yellow oil, ^1H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, $J = 7.2$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 5.29 (s, 1H), 3.76 (dq, $J = 9.7, 7.1$ Hz, 2H), 3.66 (dq, $J = 9.7, 7.1$ Hz, 2H), 1.25 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.1, 133.8, 133.5, 129.8, 128.4, 102.4, 63.2, 15.2.

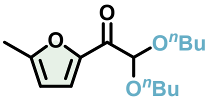


1-Phenyl-2,2-dipropoxyethan-1-one[6] (2q): yellow oil, ^1H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, $J = 7.6$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 5.24 (s, 1H), 3.72–3.61 (m, 2H), 3.60–3.49 (m, 2H), 1.68–1.59 (m, 4H), 0.91 (t, $J = 7.4$ Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.2, 133.8, 133.4, 129.8, 128.3, 103.1, 69.5, 22.9, 10.6.

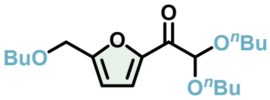


2,2-Diisopropoxy-1-phenylethan-1-one (2r): yellow oil, ^1H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, $J = 7.7$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 2H), 5.19 (s, 1H), 3.95 (hept, $J = 6.3$ Hz, 2H), 1.26 (d, $J = 6.1$ Hz, 6H), 1.15 (d, $J = 6.2$ Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.1, 133.5, 133.2, 130.3, 128.1, 102.0, 70.4, 23.0, 22.3.

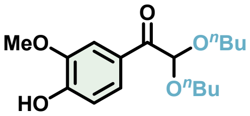
HRMS (TOF, ESI): m/z calcd for $C_{14}H_{21}O_3^+$, $[M+H]^+ = 237.1486$, found 237.1487.



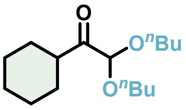
2,2-Dibutoxy-1-(5-methylfuran-2-yl) ethan-1-one (2s): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 3.5 Hz, 1H), 6.18 (dd, *J* = 3.5, 1.0 Hz, 1H), 5.07 (s, 1H), 3.68 (dt, *J* = 9.5, 6.7 Hz, 2H), 3.57 (dt, *J* = 9.5, 6.6 Hz, 2H), 2.41 (s, 3H), 1.67–1.53 (m, 4H), 1.43–1.31 (m, 4H), 0.91 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.5, 158.9, 148.9, 123.4, 109.1, 102.0, 67.3, 31.7, 19.3, 14.1, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{15}H_{25}O_4^+$, $[M+H]^+ = 269.1748$, found 269.1748.



2,2-Dibutoxy-1-(5-(butoxy methyl) furan-2-yl) ethan-1-one (2t): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 3.5 Hz, 1H), 6.47 (d, *J* = 3.5 Hz, 1H), 5.08 (s, 1H), 4.52 (s, 2H), 3.69 (dt, *J* = 9.4, 6.7 Hz, 2H), 3.57 (dt, *J* = 9.4, 6.6 Hz, 2H), 3.52 (t, *J* = 6.6 Hz, 2H), 1.65–1.54 (m, 6H), 1.38 (h, *J* = 7.4 Hz, 6H), 0.91 (t, *J* = 7.4 Hz, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 183.0, 158.2, 149.5, 122.4, 110.5, 102.0, 71.0, 67.5, 65.1, 31.7, 31.7, 19.3, 19.2, 13.9, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{19}H_{33}O_5^+$, $[M+H]^+ = 341.2323$, found 341.2325.



2,2-Dibutoxy-1-(4-hydroxy-3-methoxy phenyl) ethan-1-one (2u): yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.68 (d, *J* = 1.9 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 6.14 (s, 1H), 5.20 (s, 1H), 3.95 (s, 3H), 3.69 (dt, *J* = 9.5, 6.7 Hz, 2H), 3.56 (dt, *J* = 9.5, 6.5 Hz, 2H), 1.67–1.56 (m, 4H), 1.44–1.30 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.7, 150.7, 146.3, 126.5, 125.6, 113.8, 111.5, 103.2, 77.4, 77.0, 76.7, 67.5, 56.0, 31.7, 19.3, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{17}H_{25}O_5^-$, $[M-H]^- = 309.1707$, found 309.1707.



2,2-Dibutoxy-1-cyclohexylethan-1-one (2v): colorless oil, ^{1}H NMR (400 MHz,

$CDCl_3$) δ 4.62 (s, 1H), 3.61 (dt, *J* = 9.5, 6.6 Hz, 2H), 3.47 (dt, *J* = 9.4, 6.6 Hz, 2H), 2.81 (tt, *J* = 11.1, 3.3 Hz, 1H), 1.85–1.73 (m, 4H), 1.70–1.58 (m, 4H), 1.46–1.34 (m, 5H), 1.34–1.23 (m, 5H), 0.93 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.8, 102.6, 77.3, 77.0, 76.7, 67.5, 58.5, 45.4, 31.7, 28.5, 25.8, 25.6, 19.3, 18.5, 13.8. HRMS (TOF, ESI): m/z calcd for $C_{16}H_{31}O_3^+$, $[M+H]^+$ = 271.2268, found 271.2268.

3. References

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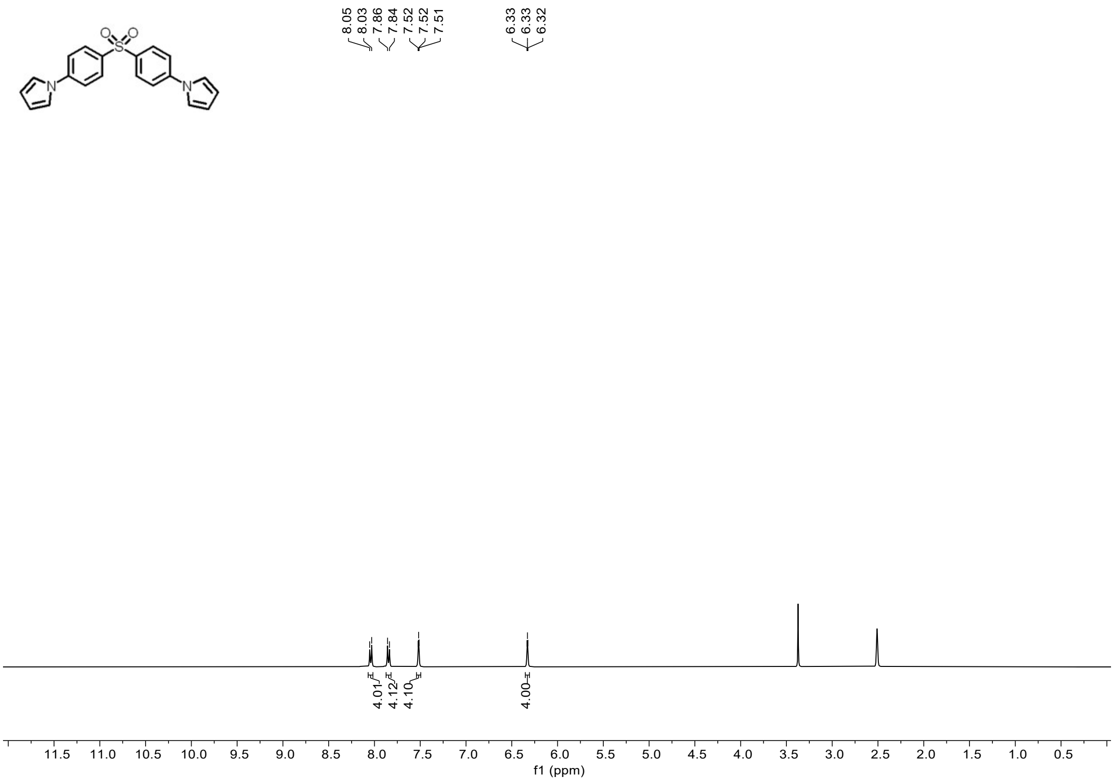
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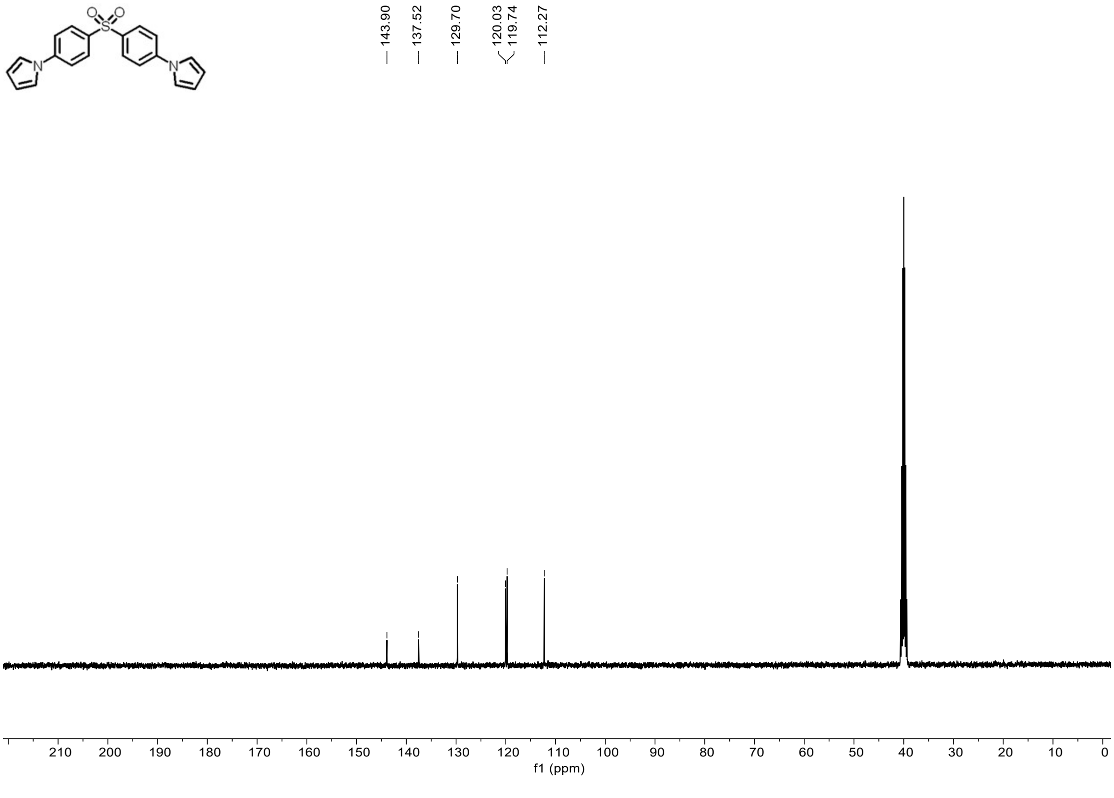
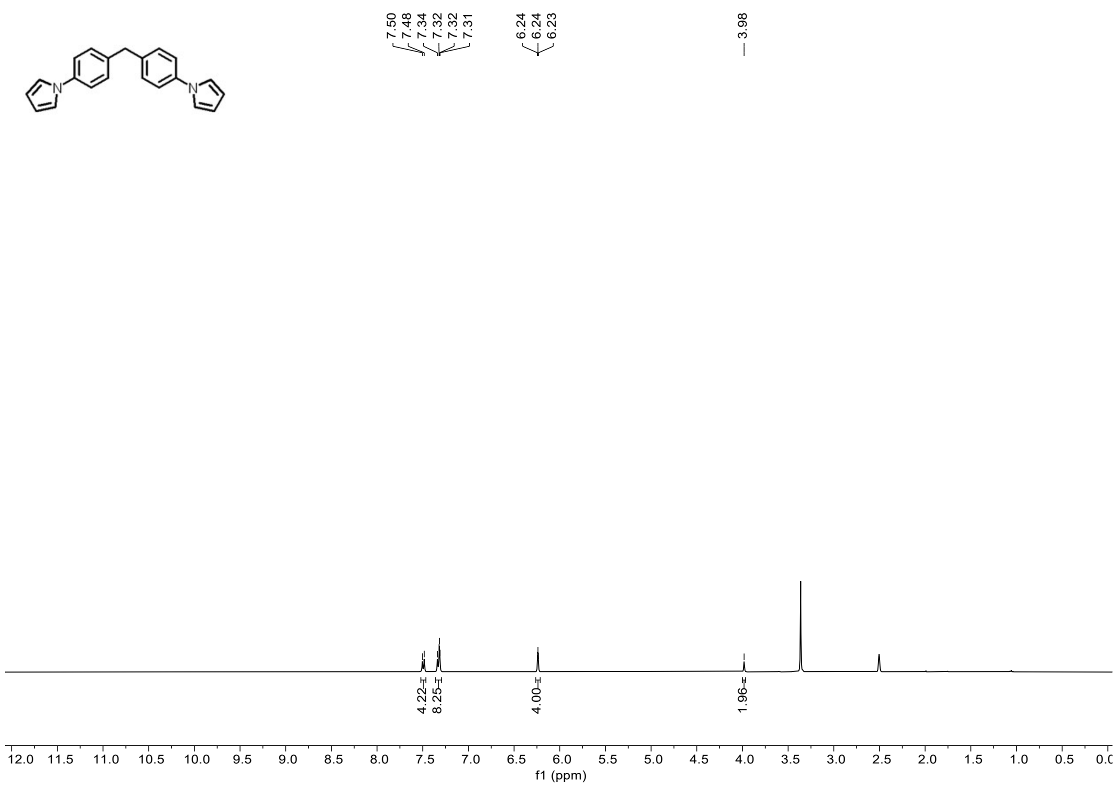
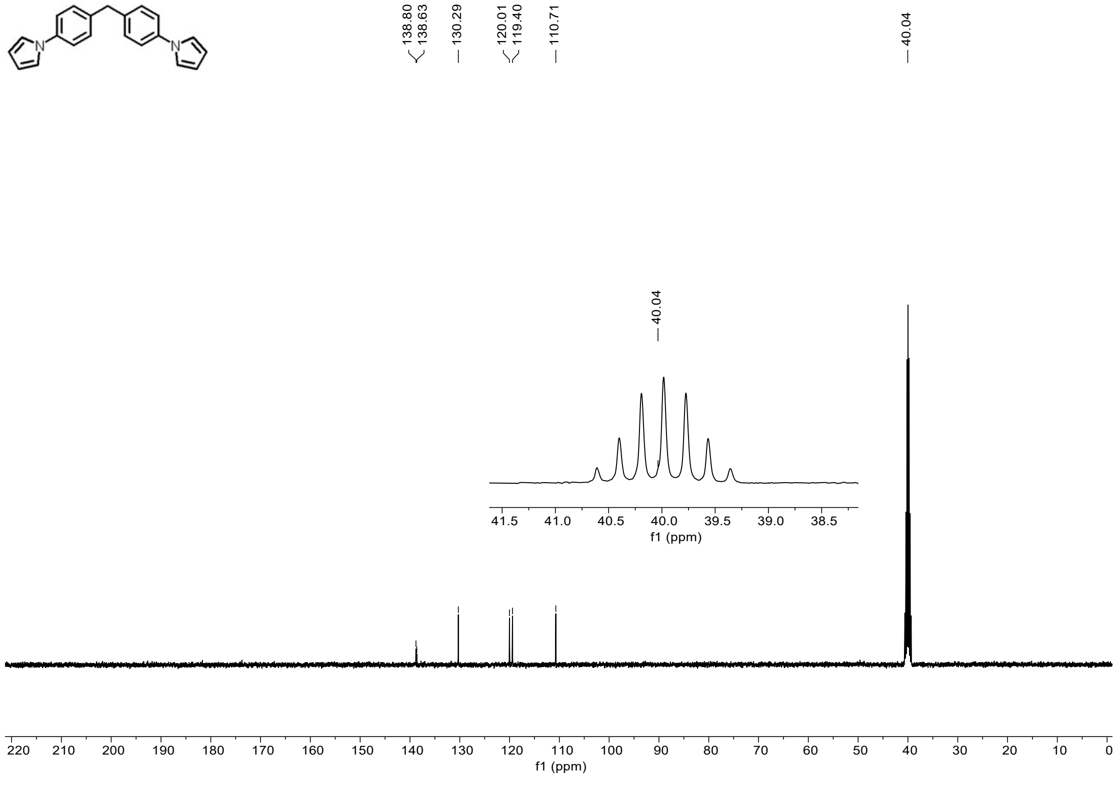
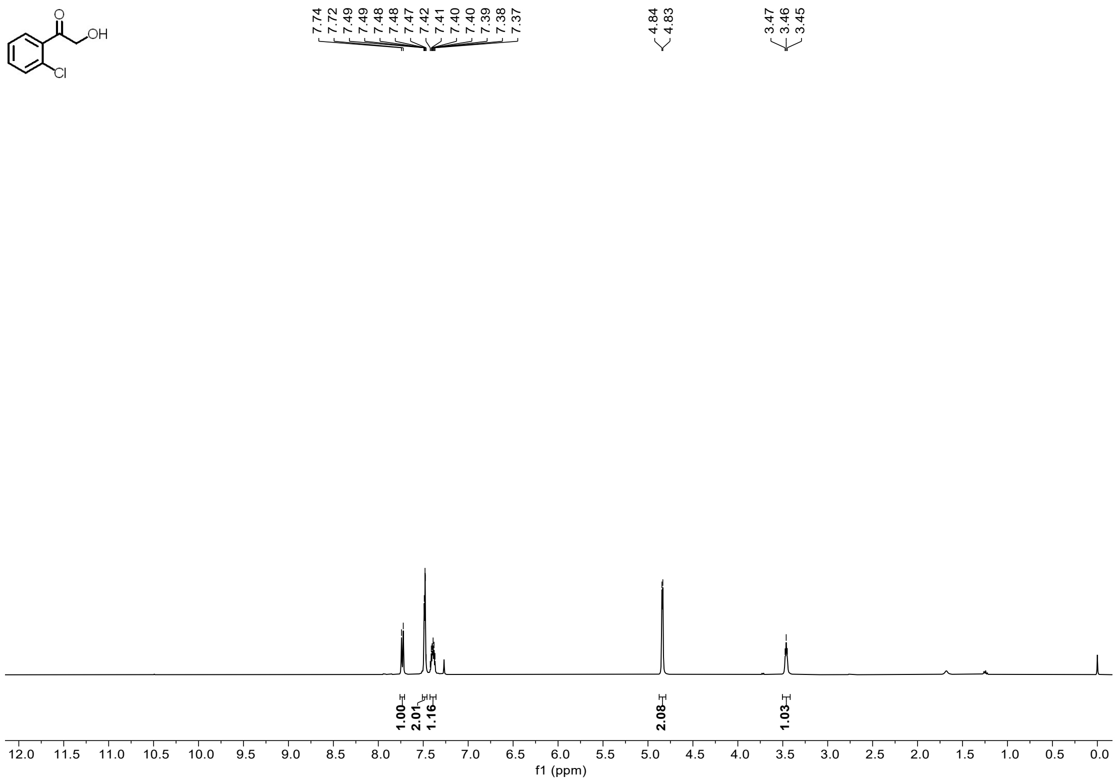
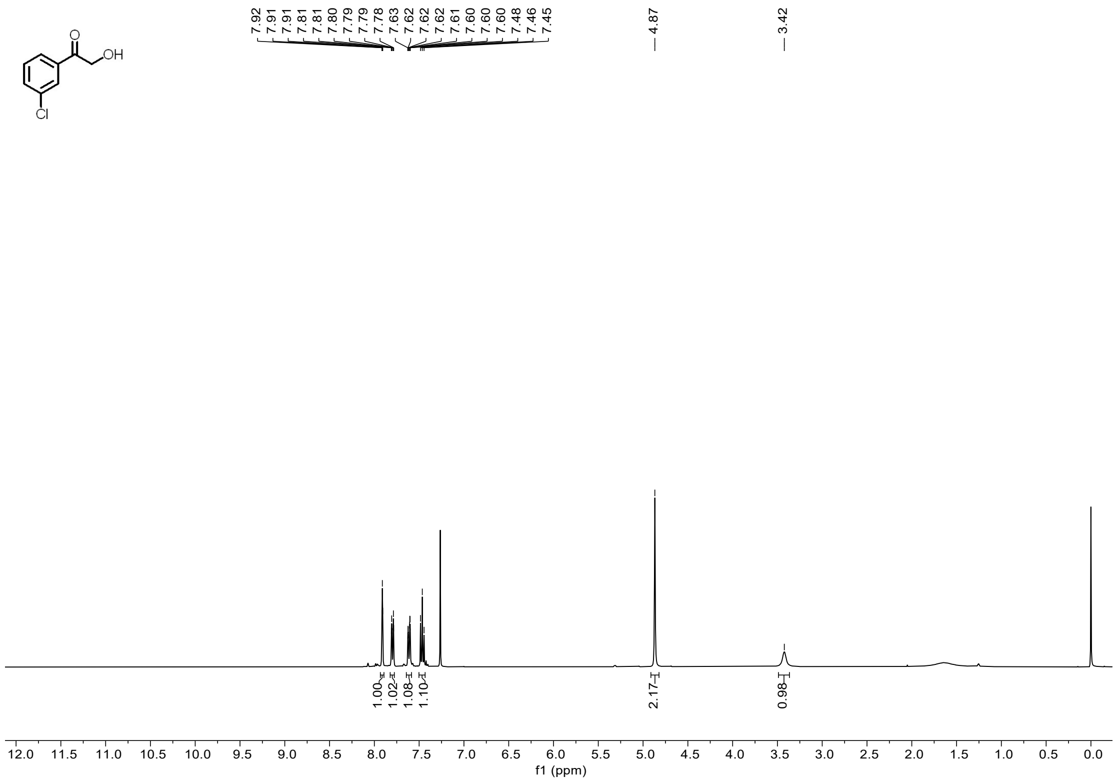
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

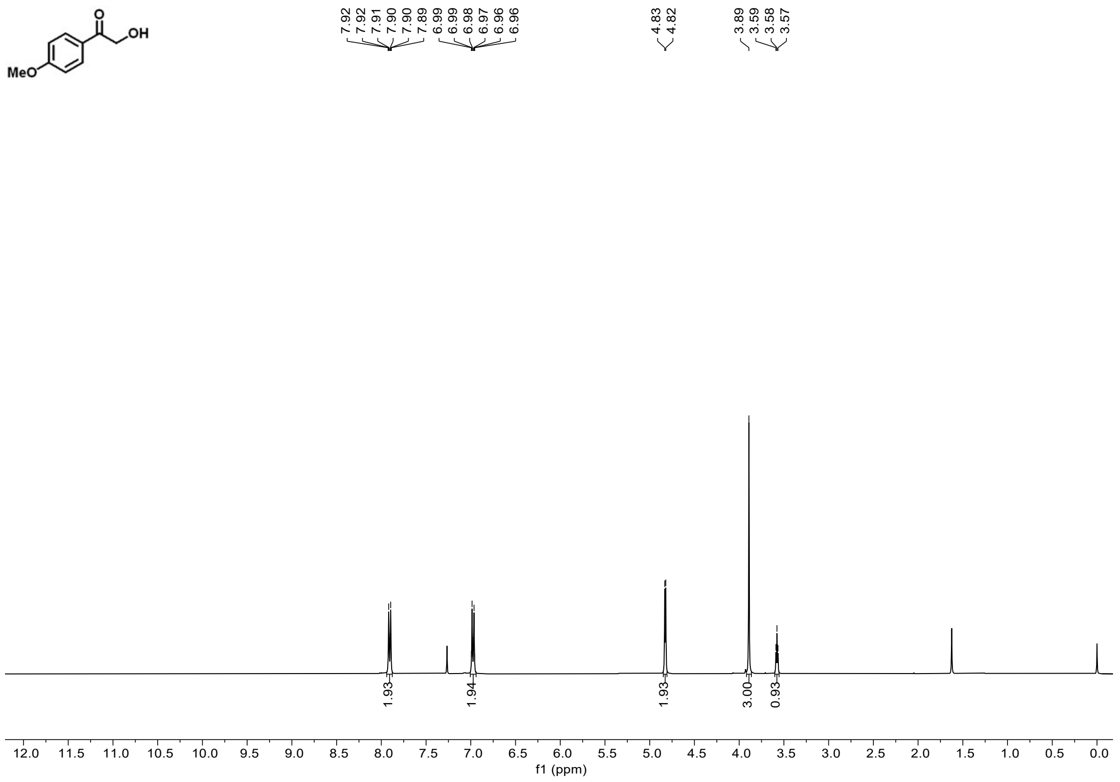

O
S
O
N
N
143.90
137.52
129.70
120.03
119.74
112.27
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

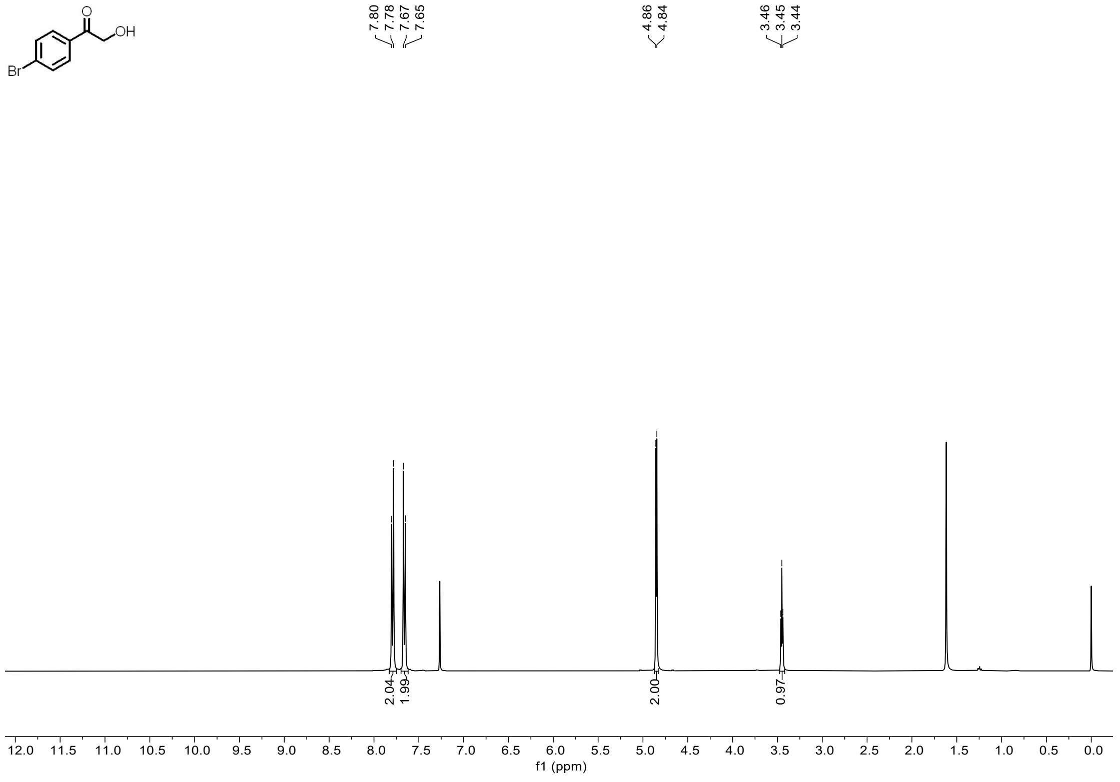

7.50
7.48
7.34
7.32
7.32
7.31
6.24
6.24
6.23
3.98
4.22
8.25
4.00
1.96
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
N
N

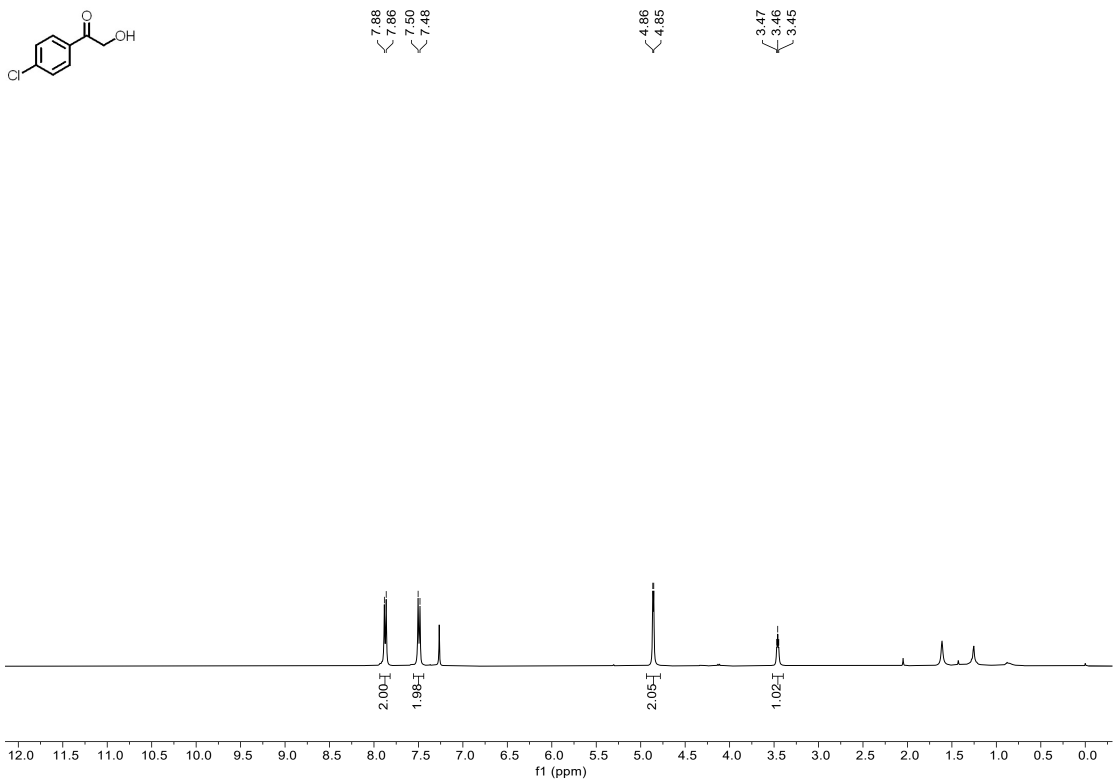

138.80
138.63
130.29
120.01
119.40
110.71
40.04
40.04
41.5
41.0
40.5
40.0
39.5
39.0
38.5
f1 (ppm)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
N
N

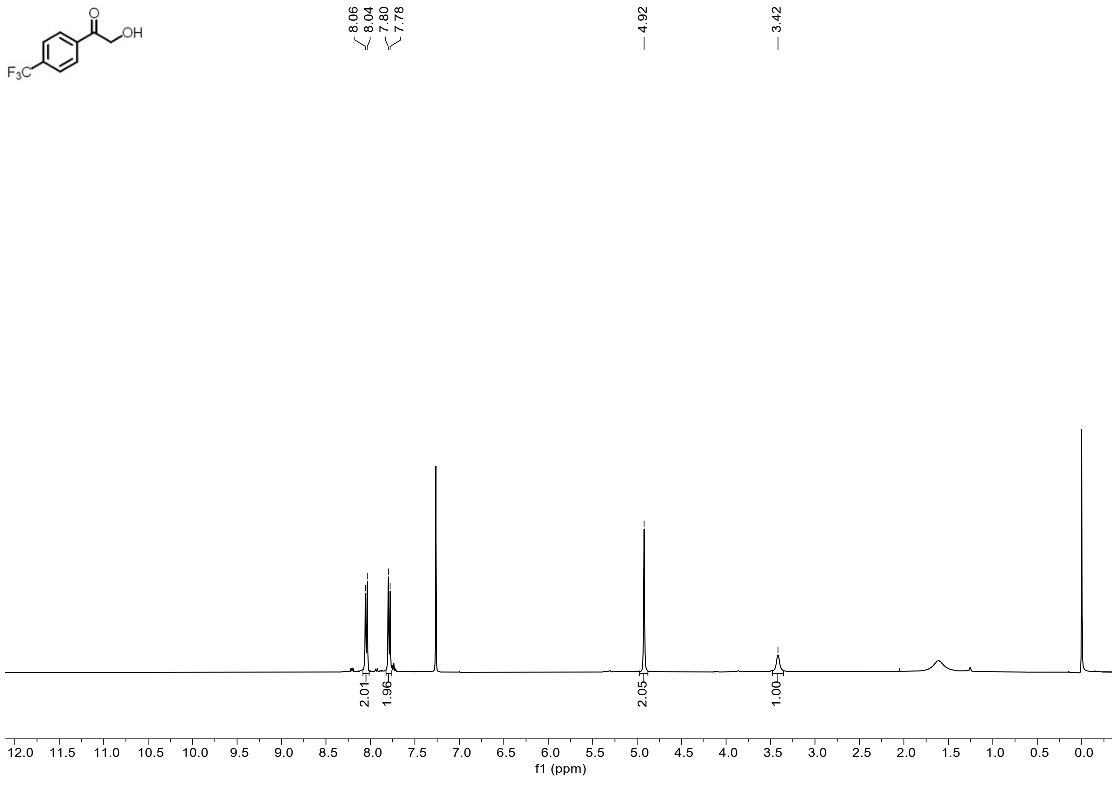

OH
Cl
7.74
7.72
7.49
7.48
7.48
7.47
7.42
7.41
7.40
7.40
7.39
7.38
7.37
4.84
4.83
3.47
3.46
3.45
1.00
2.01
1.16
2.08
1.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

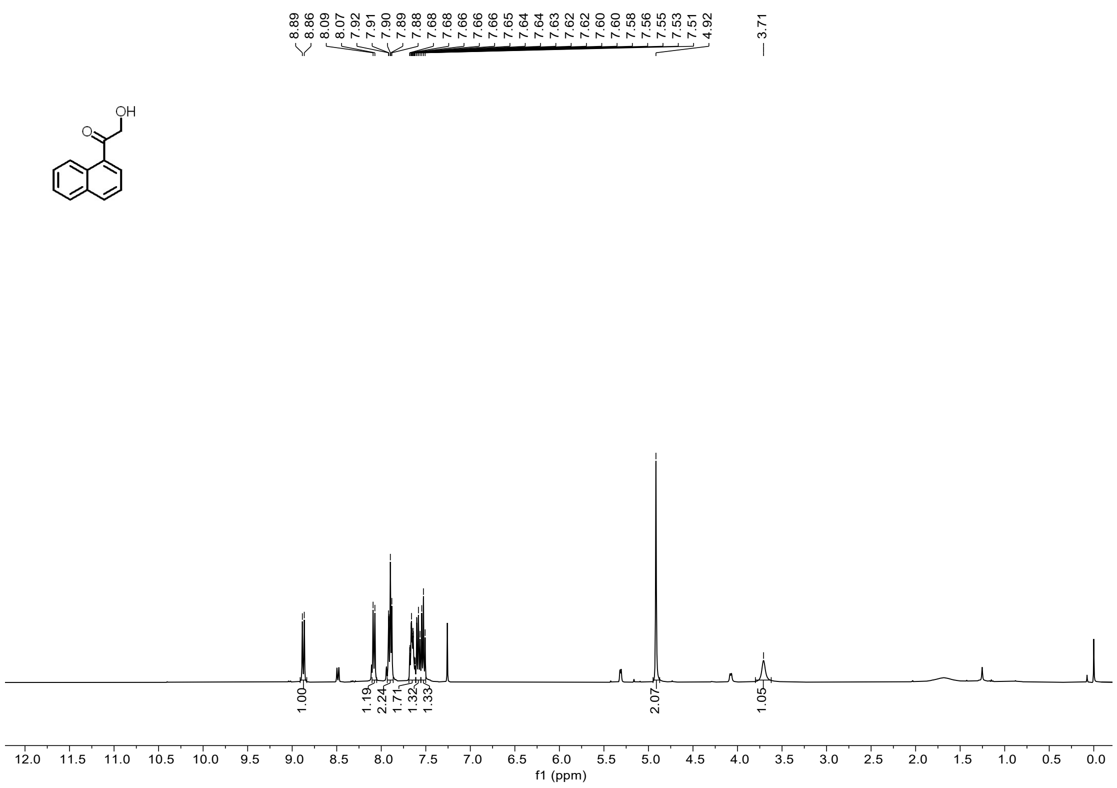

OH
Cl
7.92
7.91
7.91
7.81
7.80
7.79
7.78
7.63
7.62
7.62
7.62
7.61
7.60
7.60
7.48
7.46
7.45
4.87
3.42
1.00
1.02
1.08
1.10
2.17
0.98
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

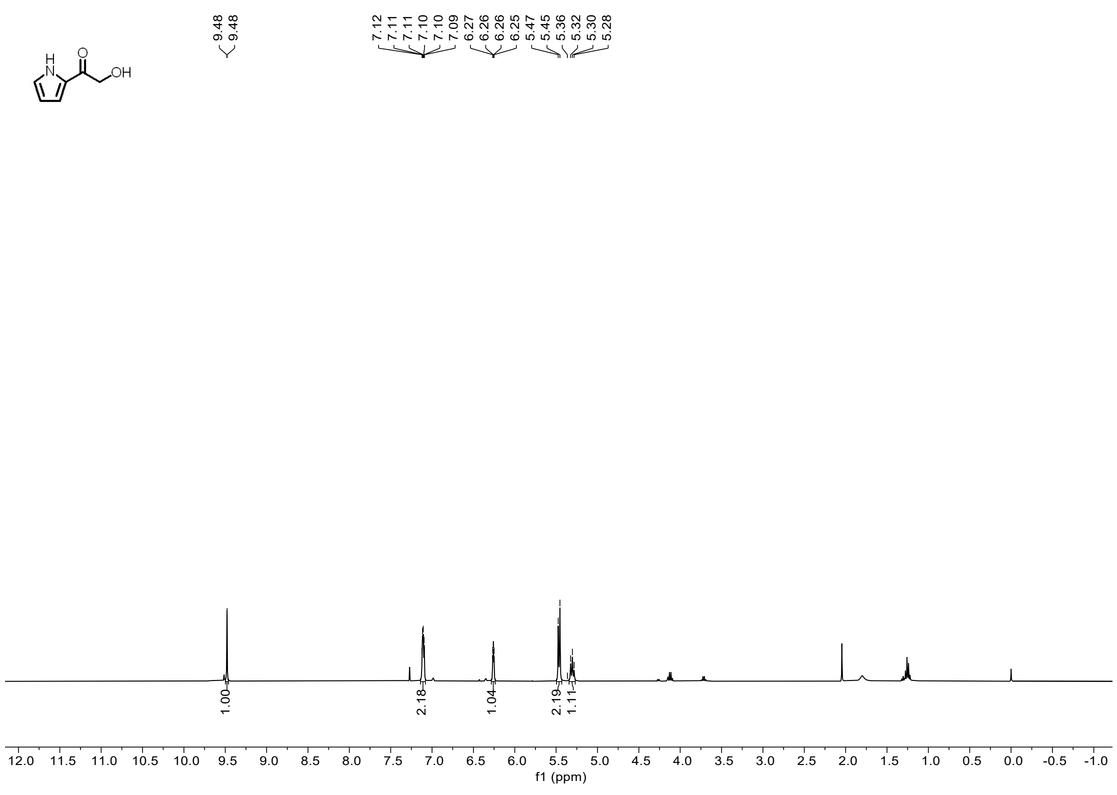

O
OH
MeO
7.92
7.92
7.91
7.90
7.90
7.89
6.99
6.99
6.98
6.97
6.96
6.96
4.83
4.82
3.89
3.59
3.58
3.57
1.93
1.94
1.93
3.00
0.93
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

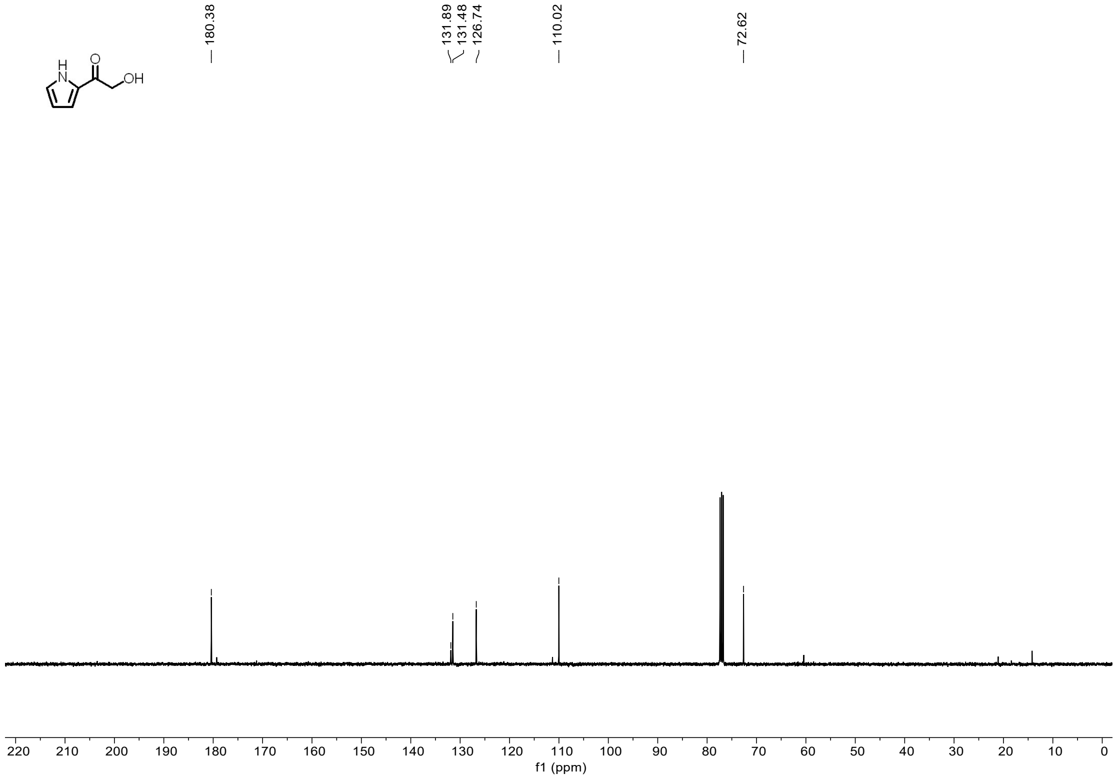

O
OH
Br
7.80
7.78
7.67
7.65
4.86
4.84
3.46
3.45
3.44
2.04
1.99
2.00
0.97
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

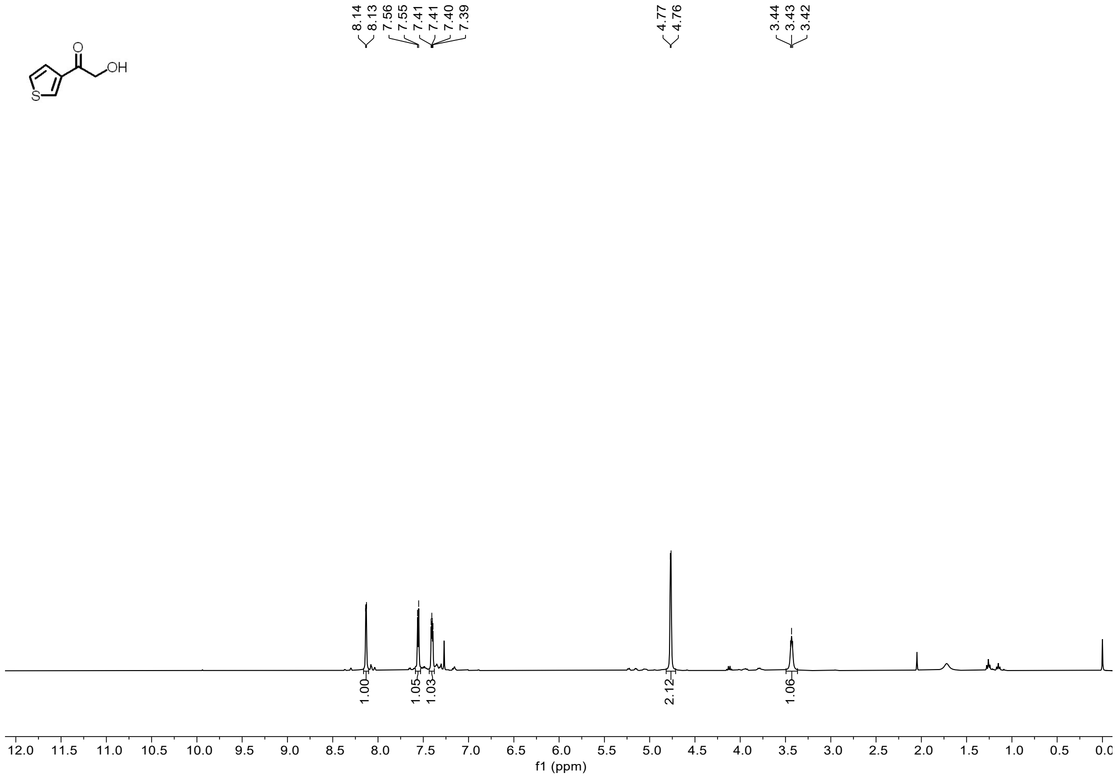

O
OH
Cl
7.88
7.86
7.50
7.48
4.86
4.85
3.47
3.46
3.45
2.00
1.98
2.05
1.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

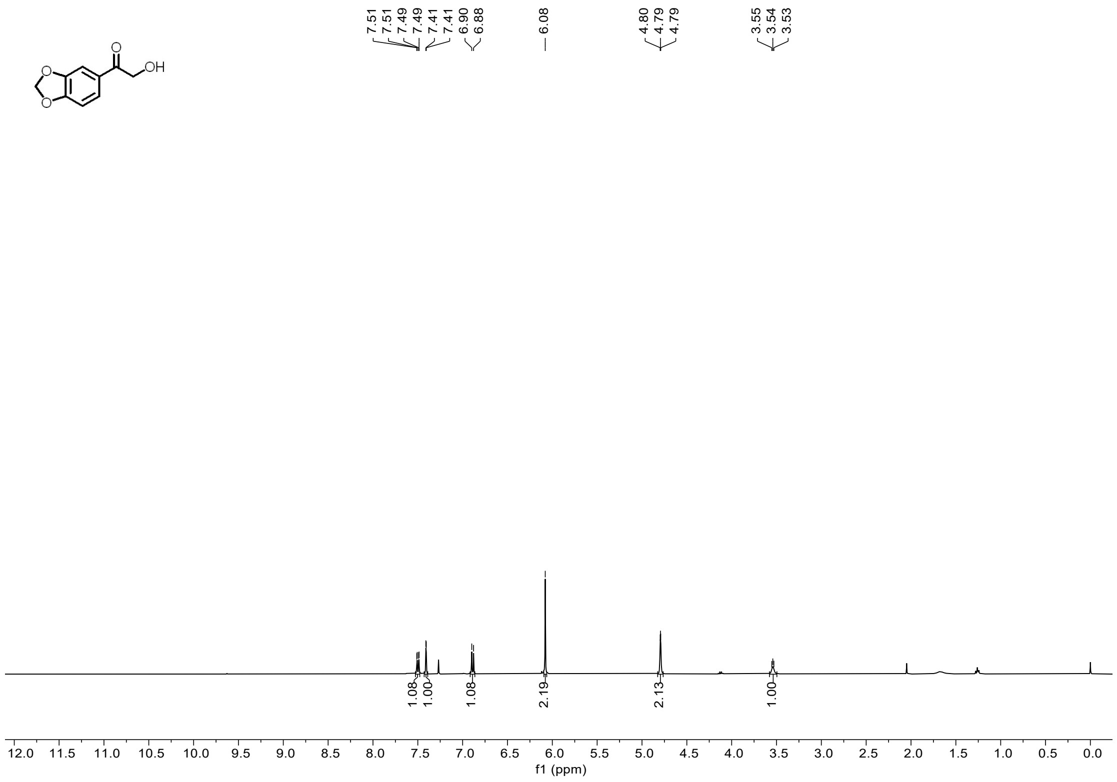

O
OH
F3C
8.06
8.04
7.80
7.78
4.92
3.42
2.01
1.96
2.05
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

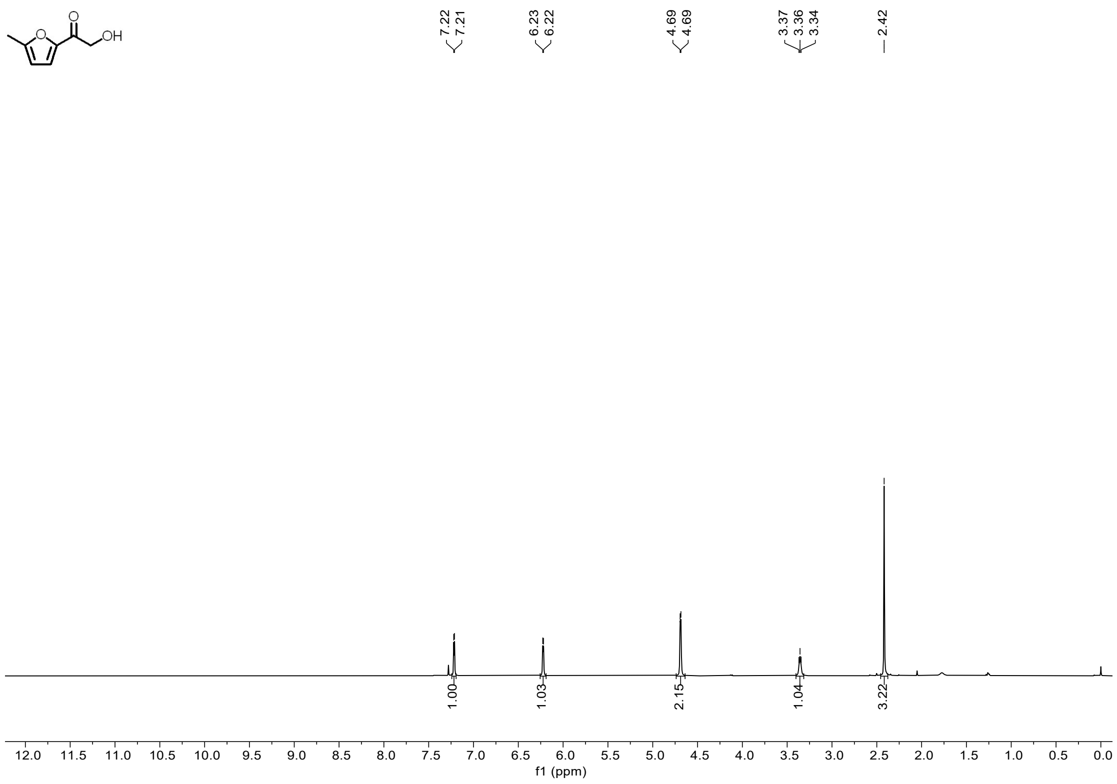

OH
O
8.89
8.86
8.09
8.07
7.92
7.91
7.90
7.89
7.88
7.68
7.66
7.66
7.65
7.64
7.63
7.62
7.62
7.60
7.60
7.58
7.56
7.55
7.53
7.51
4.92
3.71
1.00
1.19
2.24
1.71
1.32
1.33
2.07
1.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

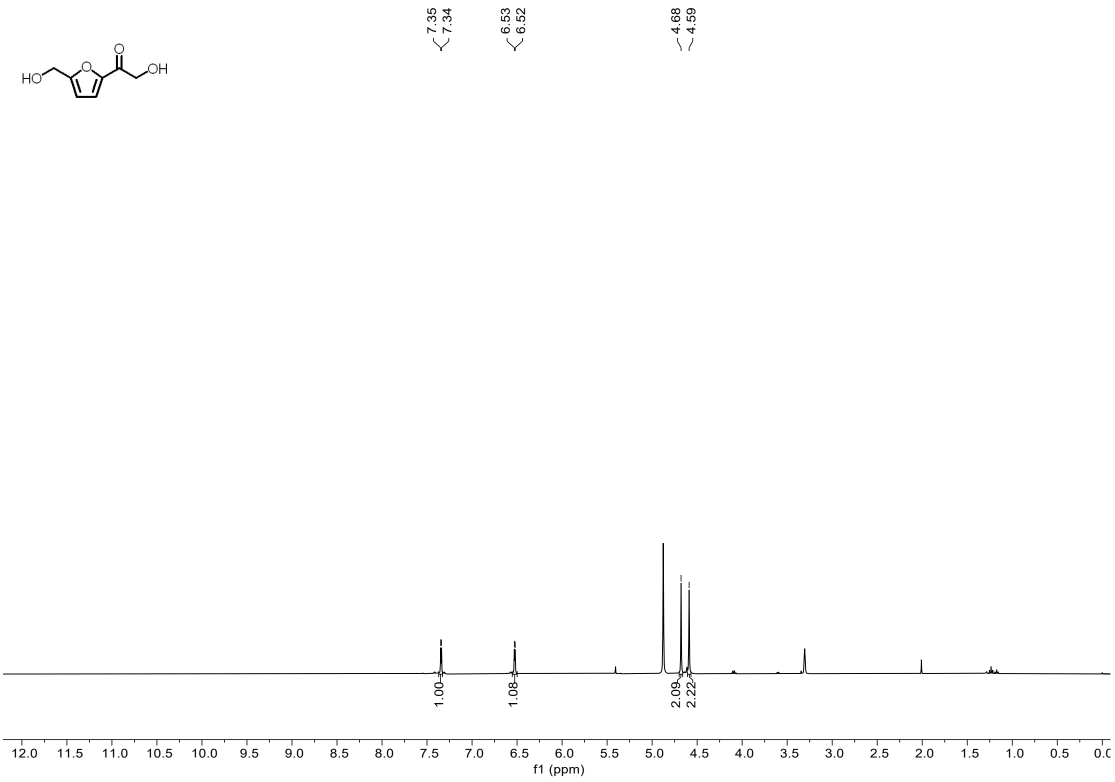

H
N
O
OH
9.48
9.48
7.12
7.11
7.11
7.10
7.09
6.27
6.26
6.26
6.25
5.47
5.45
5.36
5.32
5.30
5.28
1.00
2.18
1.04
2.19
1.11
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

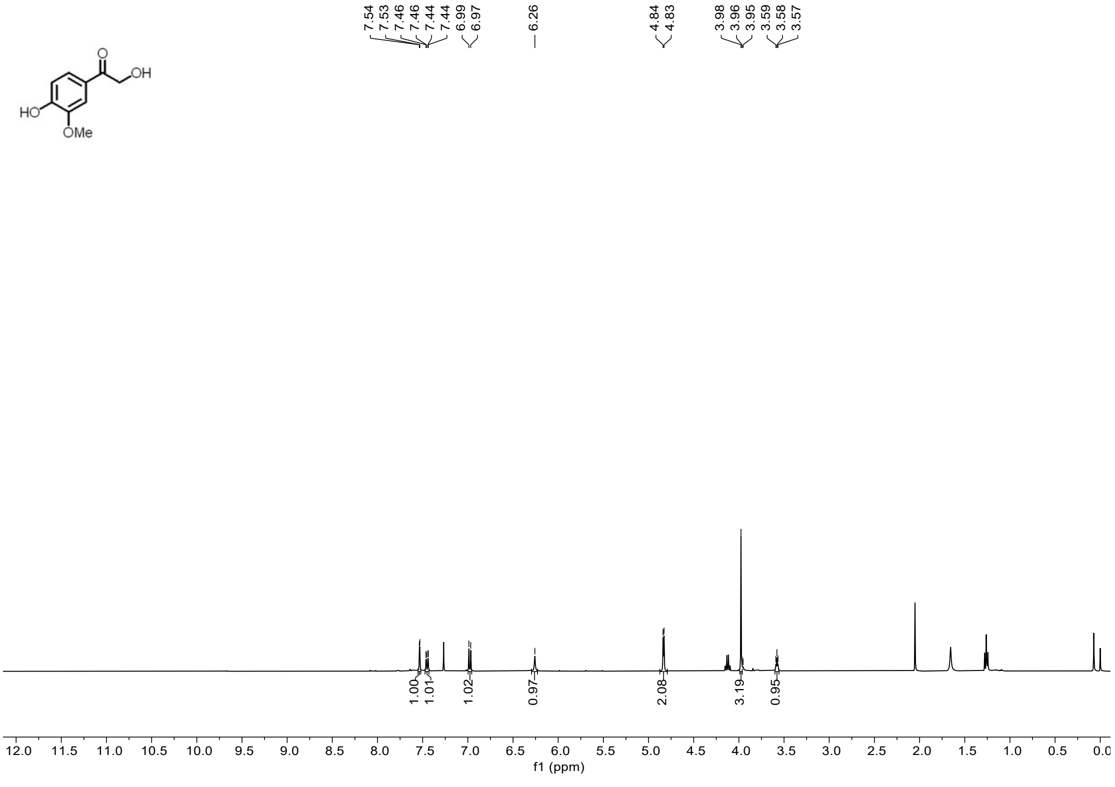

H
N
O
OH
180.38
131.89
131.48
126.74
110.02
72.62
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

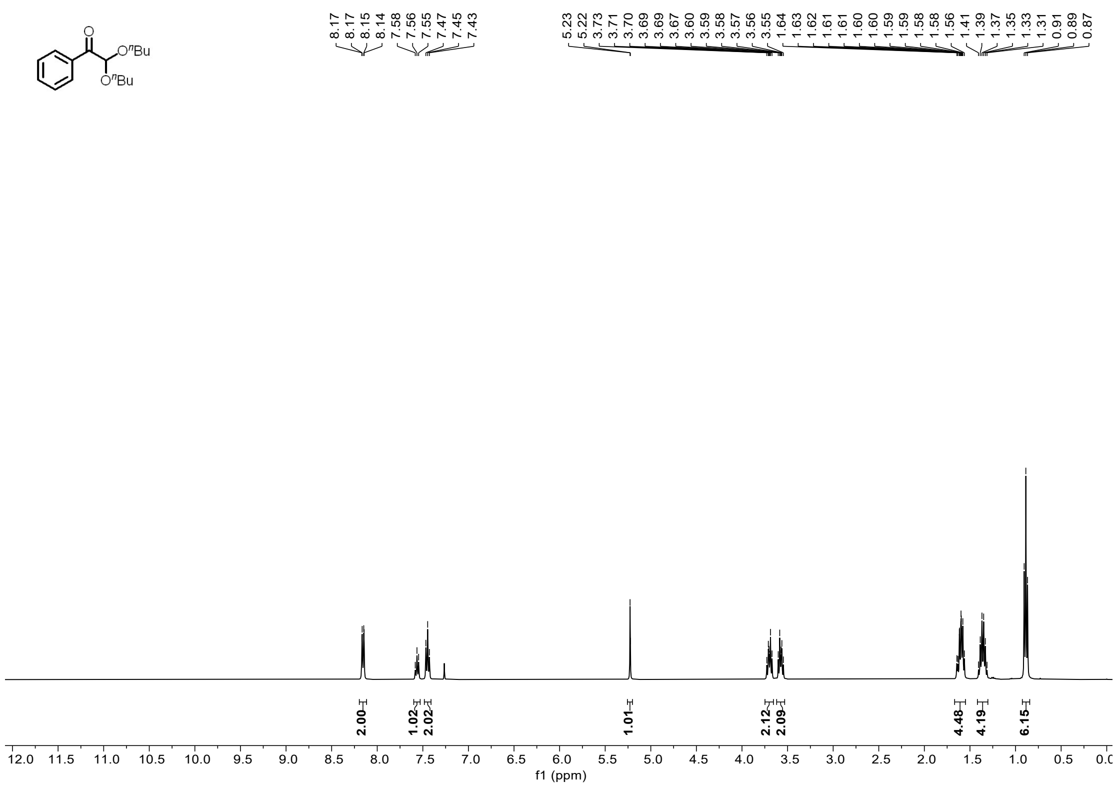

O
OH
S
8.14
8.13
7.56
7.55
7.41
7.41
7.40
7.39
4.77
4.76
3.44
3.43
3.42
1.00
1.05
1.03
2.12
1.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

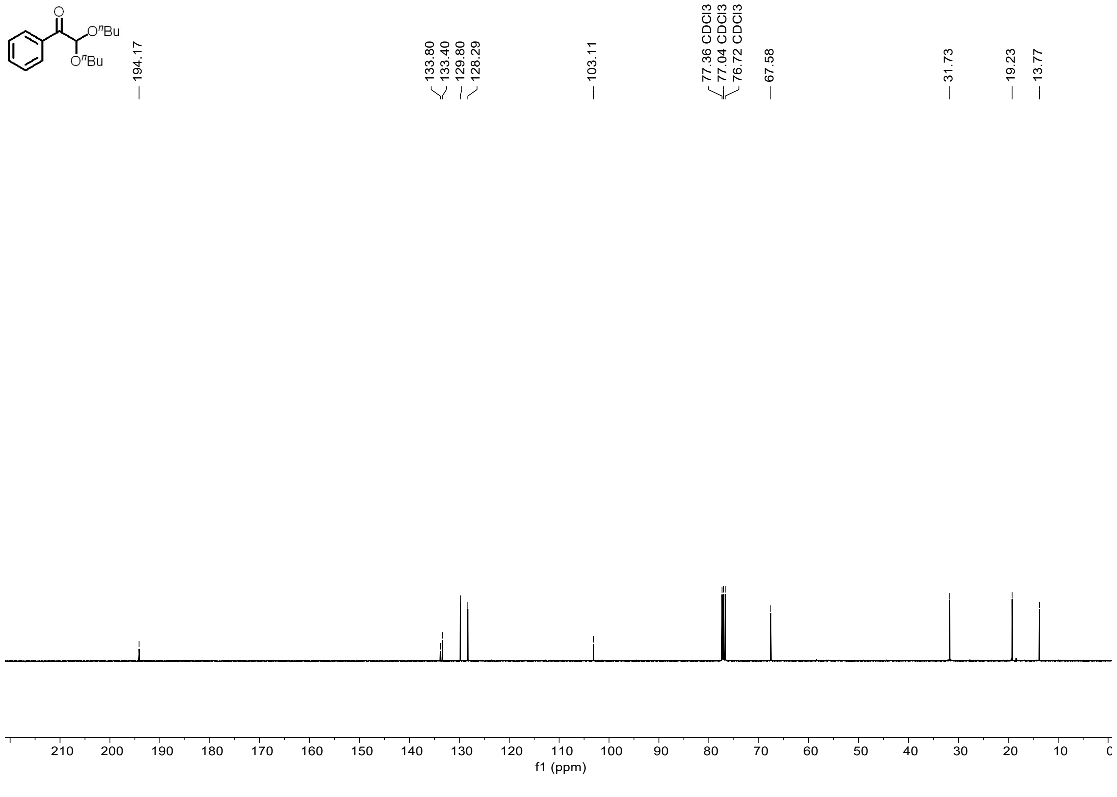

O
O
O
OH
7.51
7.51
7.49
7.49
7.41
7.41
6.90
6.88
6.08
4.80
4.79
4.79
3.55
3.54
3.53
1.08
1.00
1.08
2.19
2.13
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

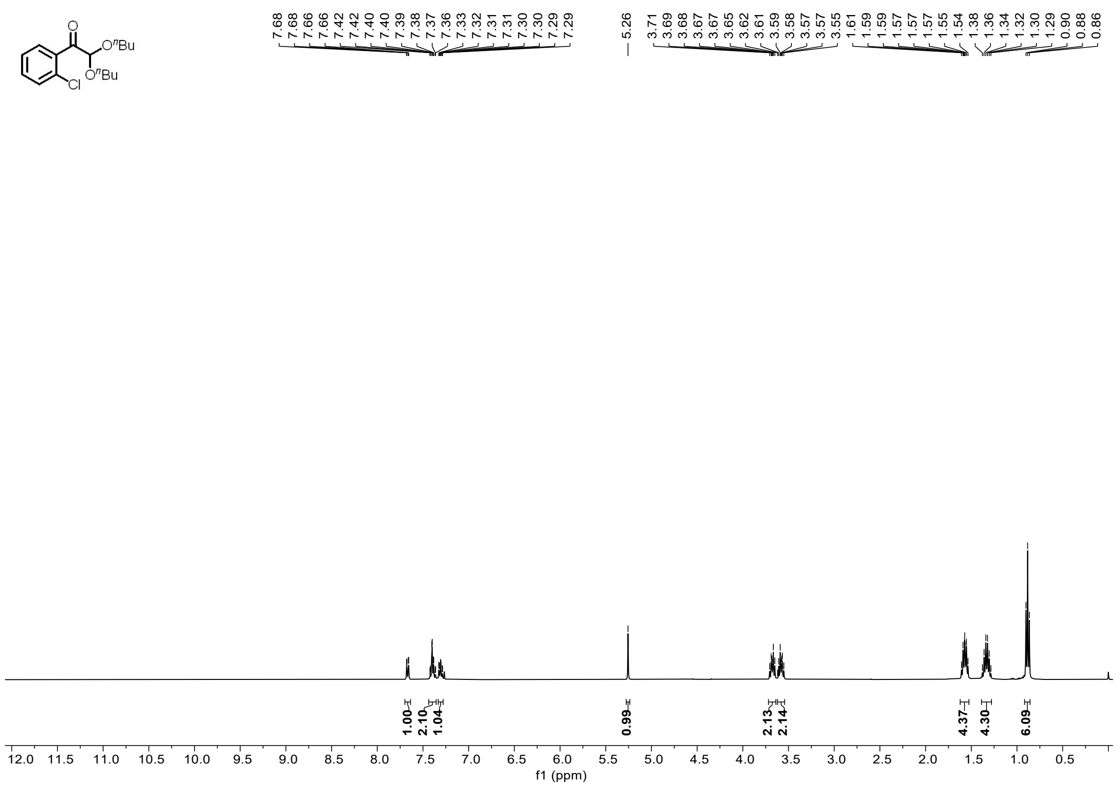

O
O
OH
7.22
7.21
6.23
6.22
4.69
4.69
3.37
3.36
3.34
2.42
1.00
1.03
2.15
1.04
3.22
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

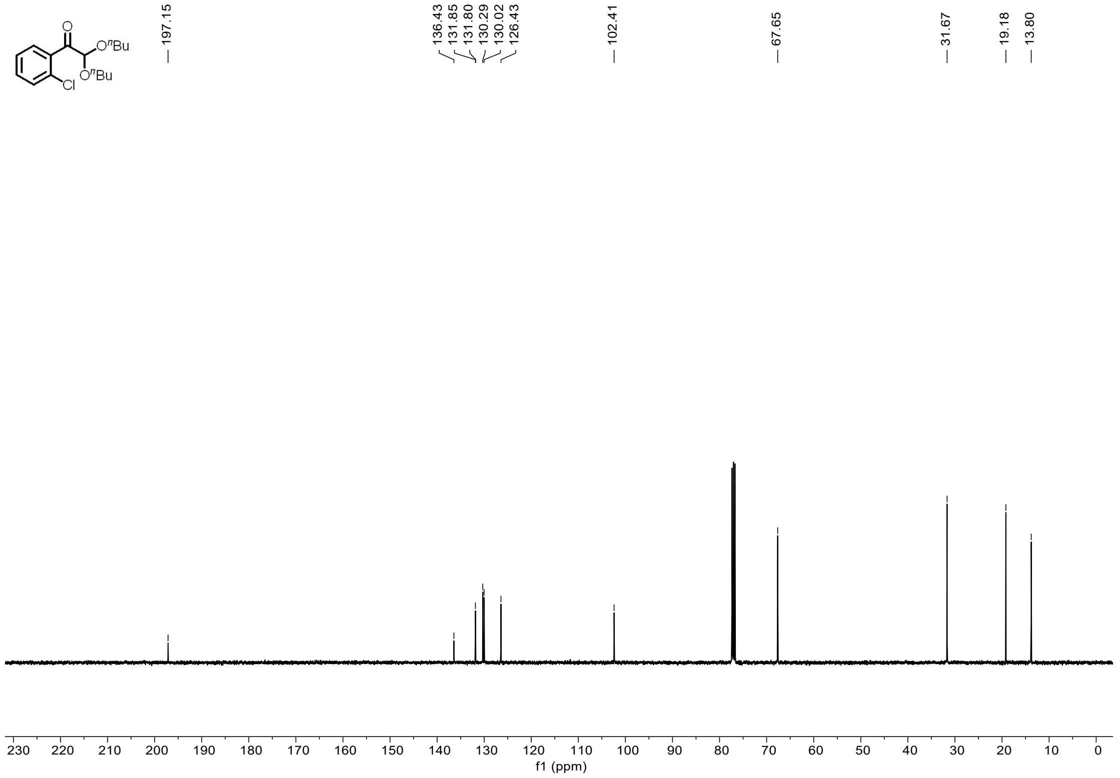

HO
O
O
OH
7.35
7.34
6.53
6.52
4.68
4.59
1.00
1.08
2.09
2.22
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

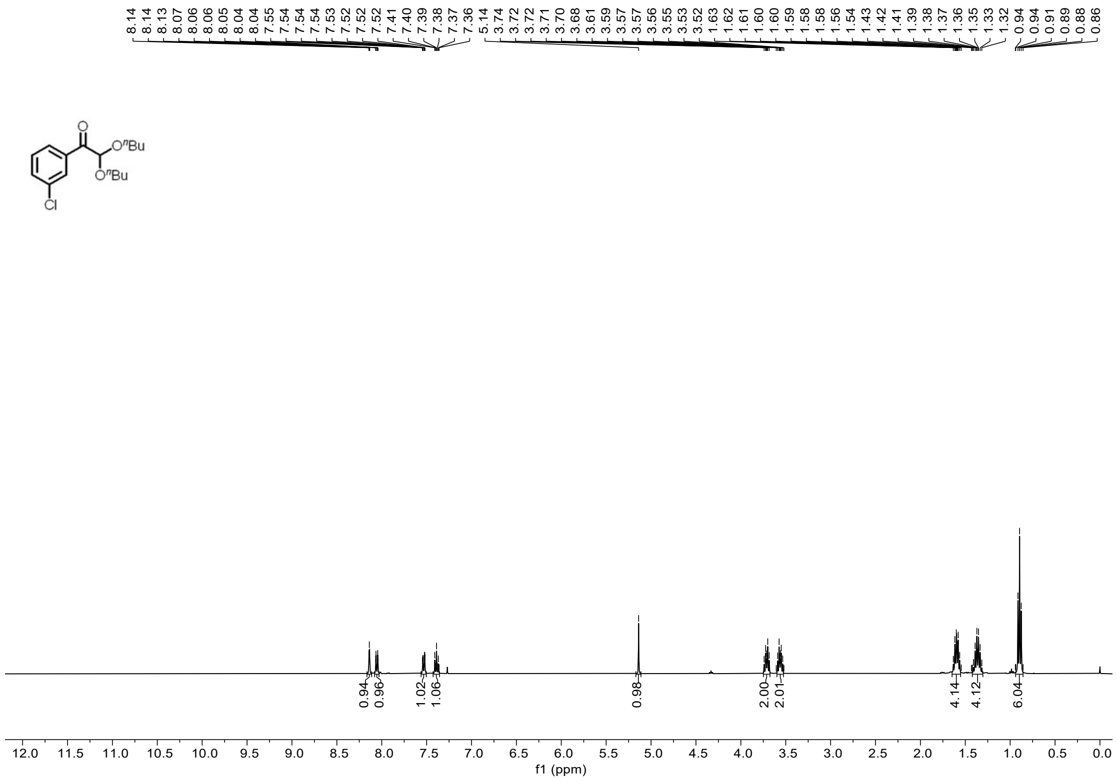

O
OH
HO
OMe
7.54
7.53
7.46
7.46
7.44
7.44
6.99
6.97
6.26
4.84
4.83
3.98
3.96
3.95
3.59
3.58
3.57
1.00
1.01
1.02
0.97
2.08
3.19
0.95
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

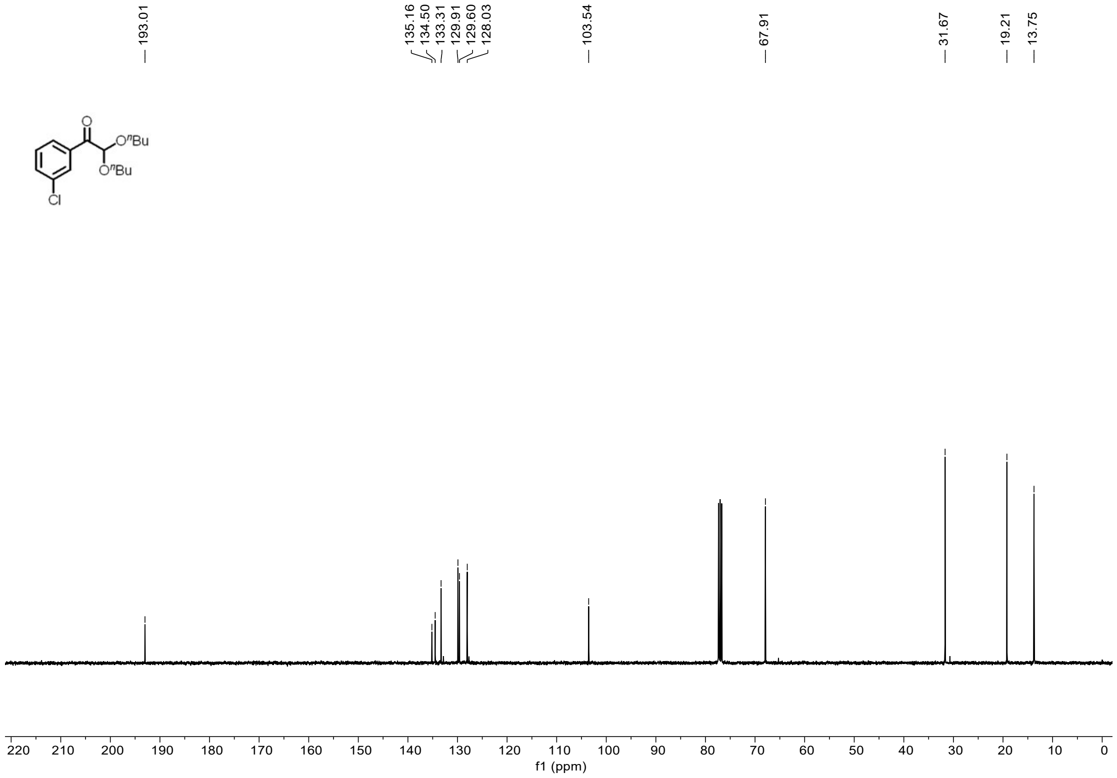

O
O^{n}Bu
O^{n}Bu
8.17
8.17
8.15
8.14
7.58
7.56
7.55
7.47
7.45
7.43
5.23
5.22
3.73
3.71
3.70
3.69
3.69
3.67
3.60
3.59
3.58
3.57
3.56
3.55
1.64
1.63
1.62
1.61
1.61
1.60
1.60
1.59
1.59
1.58
1.58
1.56
1.41
1.39
1.37
1.35
1.33
1.31
0.91
0.89
0.87
2.00
1.02
2.02
1.01
2.12
2.09
4.48
4.19
6.15
12.0 11.5 11.0 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
f1 (ppm)

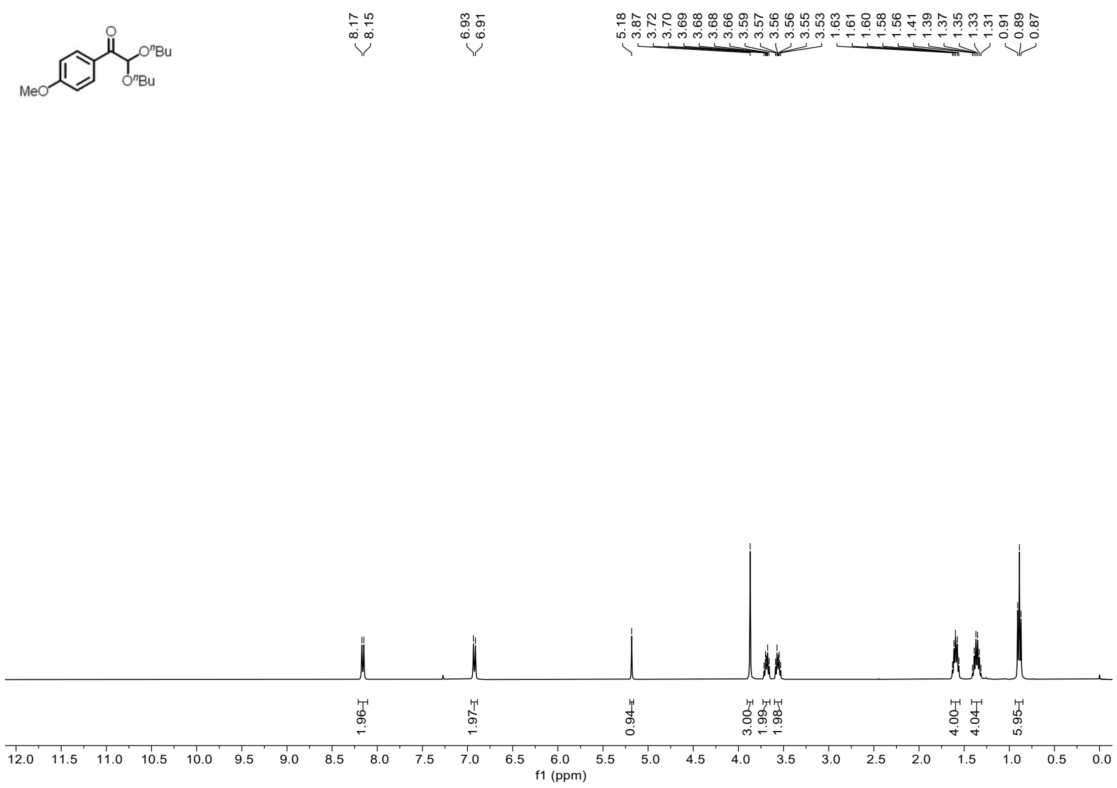

O
O^{n}Bu
O^{n}Bu
194.17
133.80
133.40
129.80
128.29
103.11
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
67.58
31.73
19.23
13.77
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

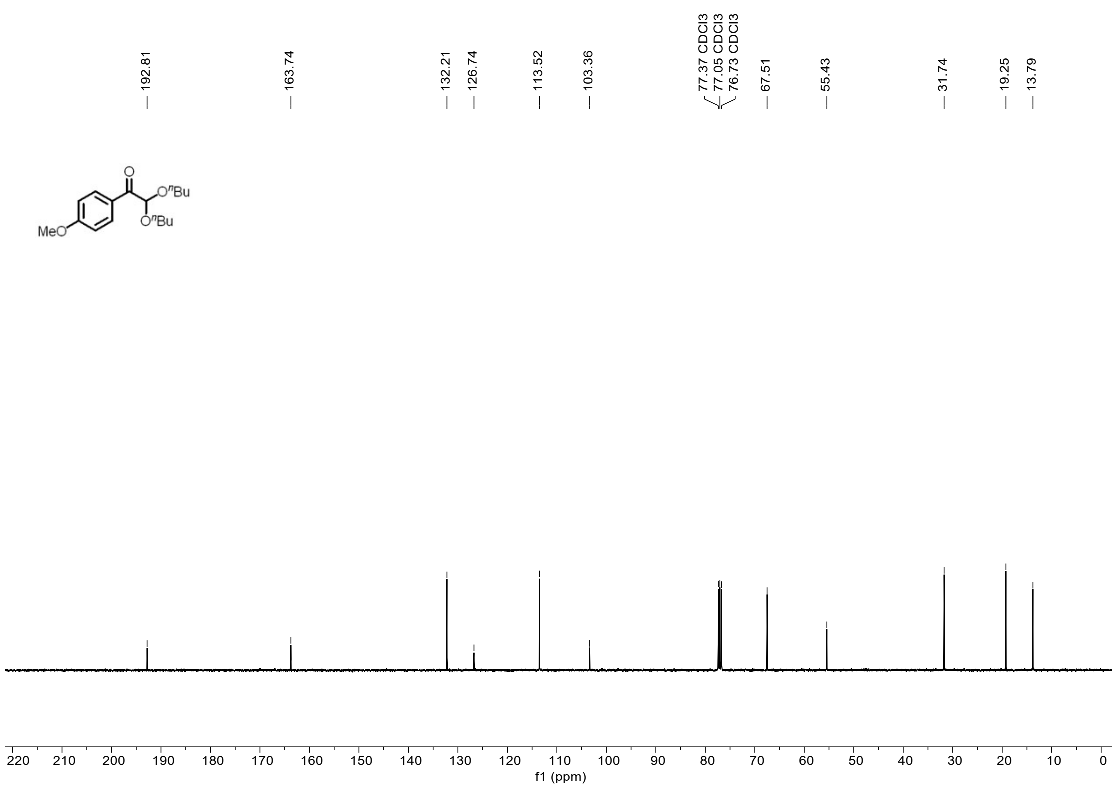

O
O^{n}Bu
O^{n}Bu
Cl
7.68
7.68
7.66
7.66
7.42
7.40
7.39
7.38
7.37
7.36
7.33
7.32
7.31
7.31
7.30
7.29
7.29
5.26
3.71
3.69
3.68
3.67
3.65
3.62
3.61
3.59
3.58
3.57
3.55
1.61
1.59
1.57
1.57
1.55
1.54
1.38
1.36
1.34
1.32
1.30
1.29
0.90
0.88
0.86
1.00
2.10
1.04
0.99
2.13
2.14
4.37
4.30
6.09
12.0
11.5
11.0
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
f1 (ppm)

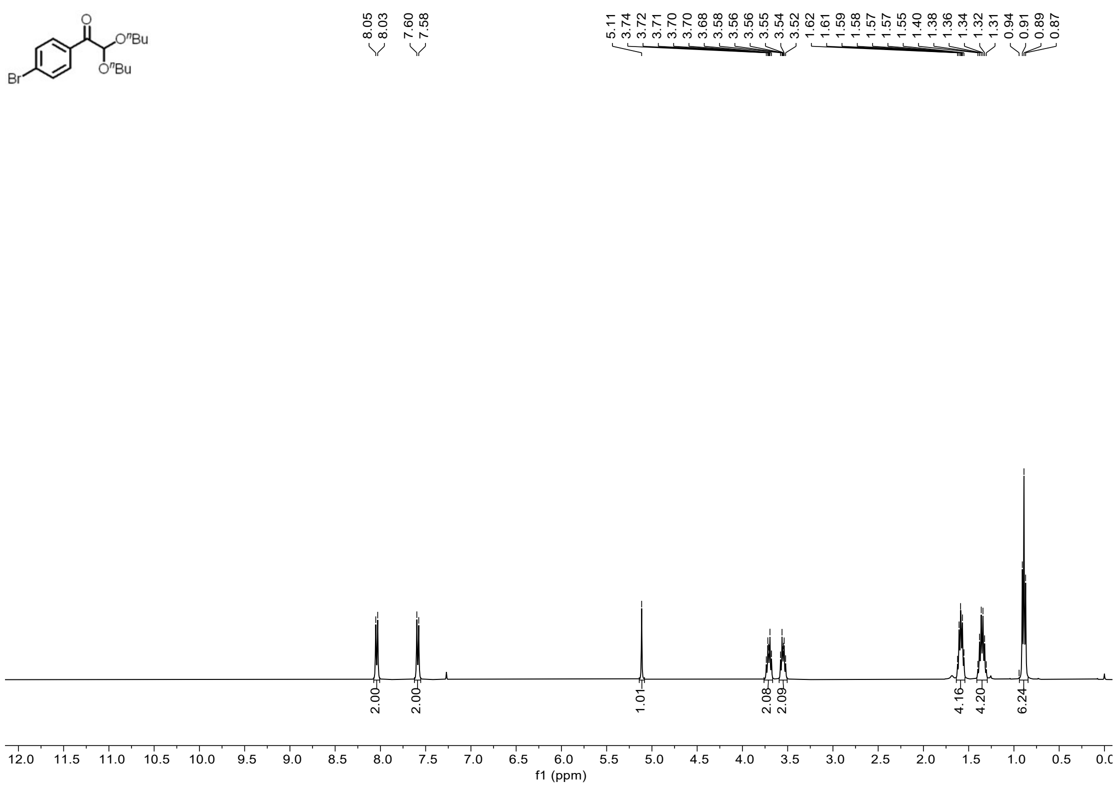

O
O^{n}Bu
O^{n}Bu
Cl
197.15
136.43
131.85
131.80
130.29
130.02
126.43
102.41
67.65
31.67
19.18
13.80
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

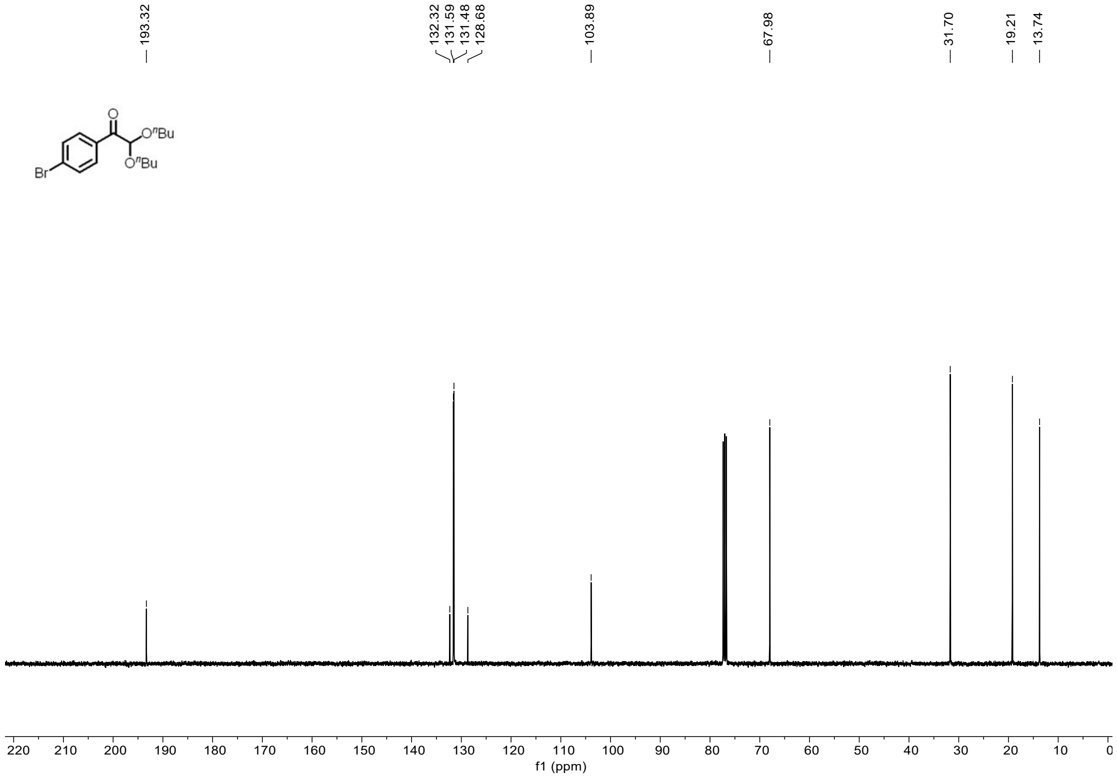

8.14
8.14
8.13
8.07
8.06
8.05
8.04
7.55
7.54
7.54
7.53
7.52
7.52
7.52
7.41
7.40
7.39
7.38
7.37
7.36
5.14
3.74
3.72
3.71
3.70
3.68
3.61
3.59
3.57
3.57
3.56
3.55
3.53
3.52
1.63
1.62
1.61
1.60
1.60
1.59
1.58
1.58
1.56
1.54
1.43
1.42
1.41
1.39
1.38
1.37
1.36
1.35
1.33
1.32
0.94
0.94
0.91
0.89
0.88
0.86
O
O^{n}Bu
O^{n}Bu
Cl
0.94
0.96
1.02
1.06
0.98
2.00
2.01
4.14
4.12
6.04
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
f1 (ppm)

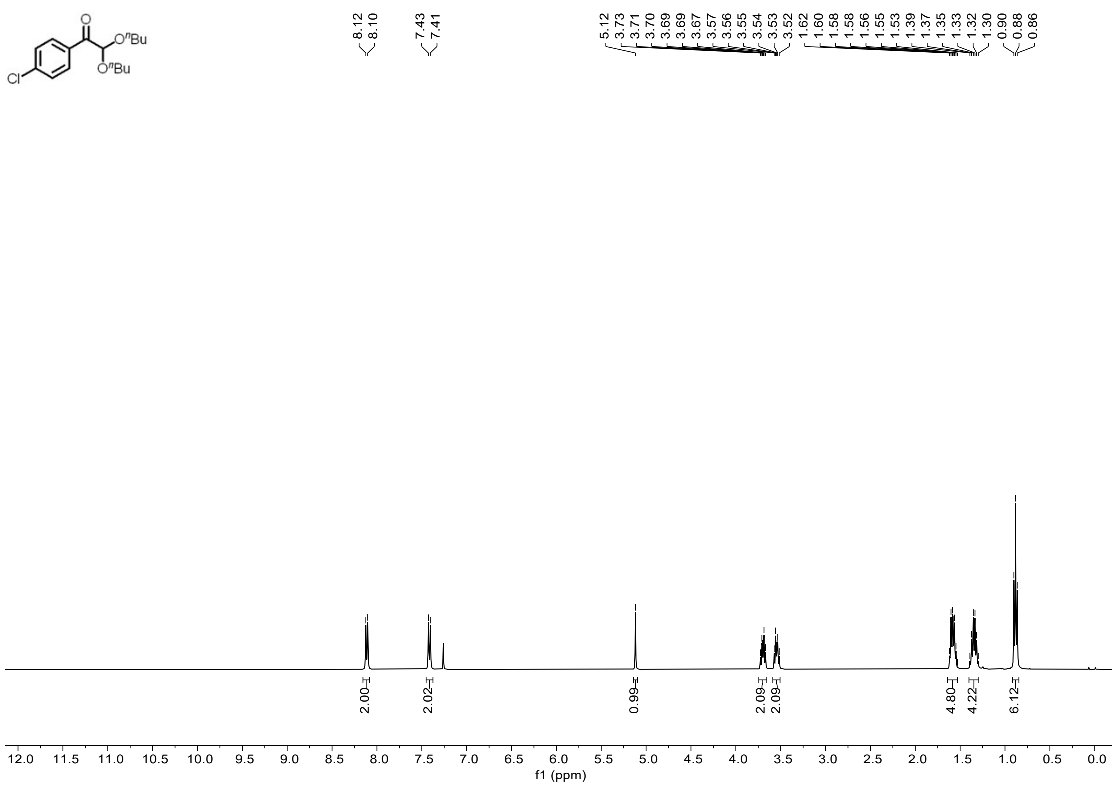

193.01
135.16
134.50
133.31
129.91
129.60
128.03
103.54
67.91
31.67
19.21
13.75
O
O^{n}Bu
O^{n}Bu
Cl
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

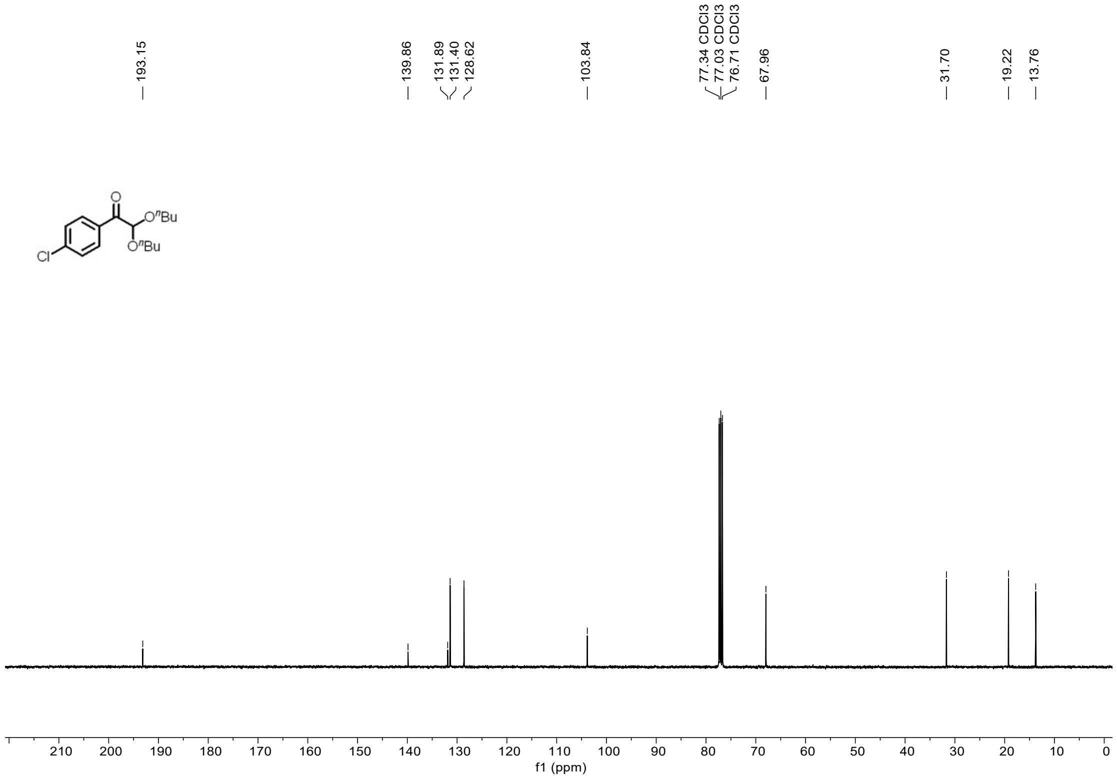

O
O^{n}Bu
O^{n}Bu
MeO
8.17
8.15
6.93
6.91
5.18
3.87
3.72
3.70
3.69
3.68
3.68
3.66
3.59
3.57
3.56
3.56
3.55
3.53
1.63
1.61
1.60
1.58
1.56
1.41
1.39
1.37
1.35
1.33
1.31
0.91
0.89
0.87
1.96
1.97
0.94
3.00
1.99
1.98
4.00
4.04
5.95
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


192.81
163.74
132.21
126.74
113.52
103.36
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
67.51
55.43
31.74
19.25
13.79
O
O^{n}Bu
O^{n}Bu
MeO
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O^{n}Bu
O^{n}Bu
Br
8.05
8.03
7.60
7.58
5.11
3.74
3.72
3.71
3.70
3.70
3.68
3.58
3.56
3.56
3.55
3.54
3.52
1.62
1.61
1.59
1.58
1.57
1.57
1.55
1.40
1.38
1.36
1.34
1.32
1.31
0.94
0.91
0.89
0.87
2.00
2.00
1.01
2.08
2.09
4.16
4.20
6.24
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
f1 (ppm)


193.32
132.32
131.59
131.48
128.68
103.89
67.98
31.70
19.21
13.74
O
O^{n}Bu
O^{n}Bu
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
O^{n}Bu
O^{n}Bu
Cl
8.12
8.10
7.43
7.41
5.12
3.73
3.71
3.70
3.69
3.69
3.67
3.57
3.56
3.55
3.54
3.53
3.52
1.62
1.60
1.58
1.58
1.56
1.55
1.53
1.39
1.37
1.35
1.33
1.32
1.30
0.90
0.88
0.86
2.00
2.02
0.99
2.09
2.09
4.80
4.22
6.12
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
f1 (ppm)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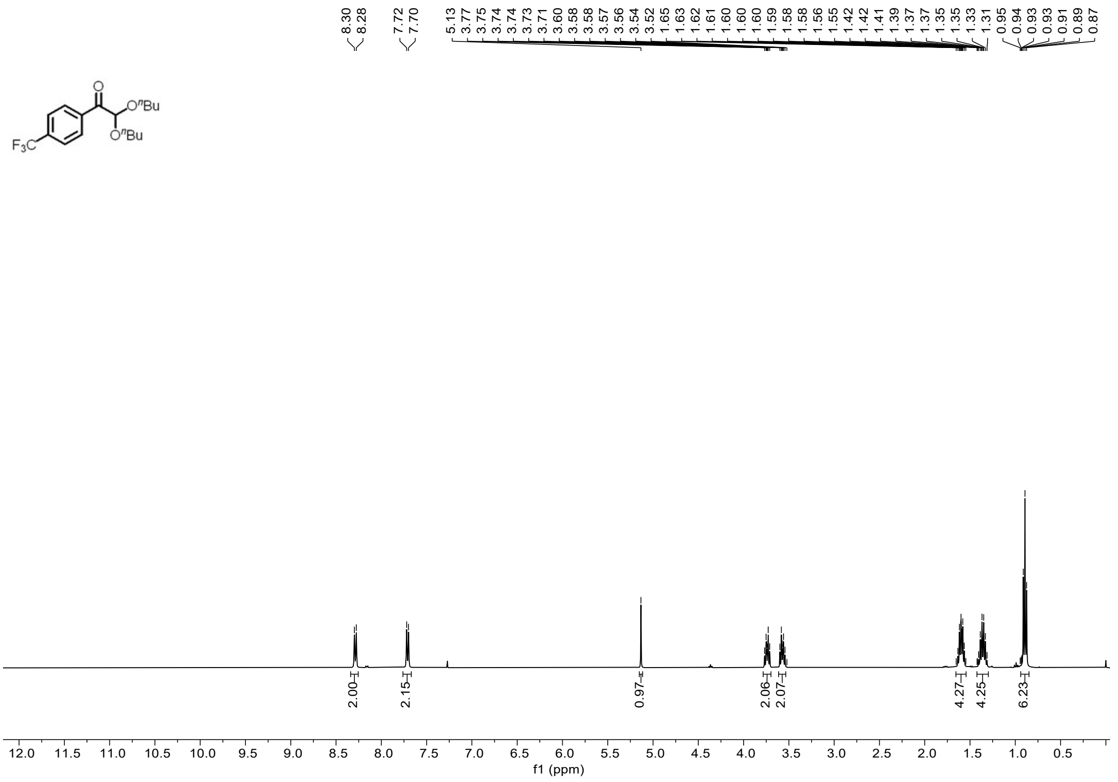
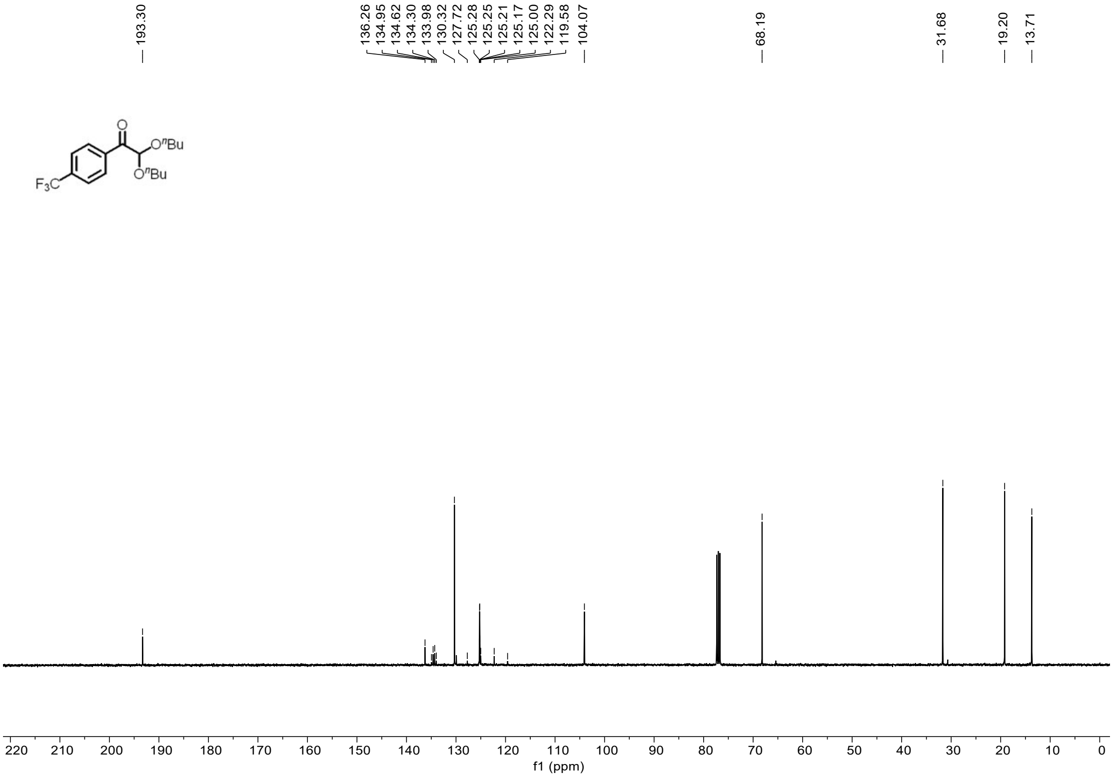
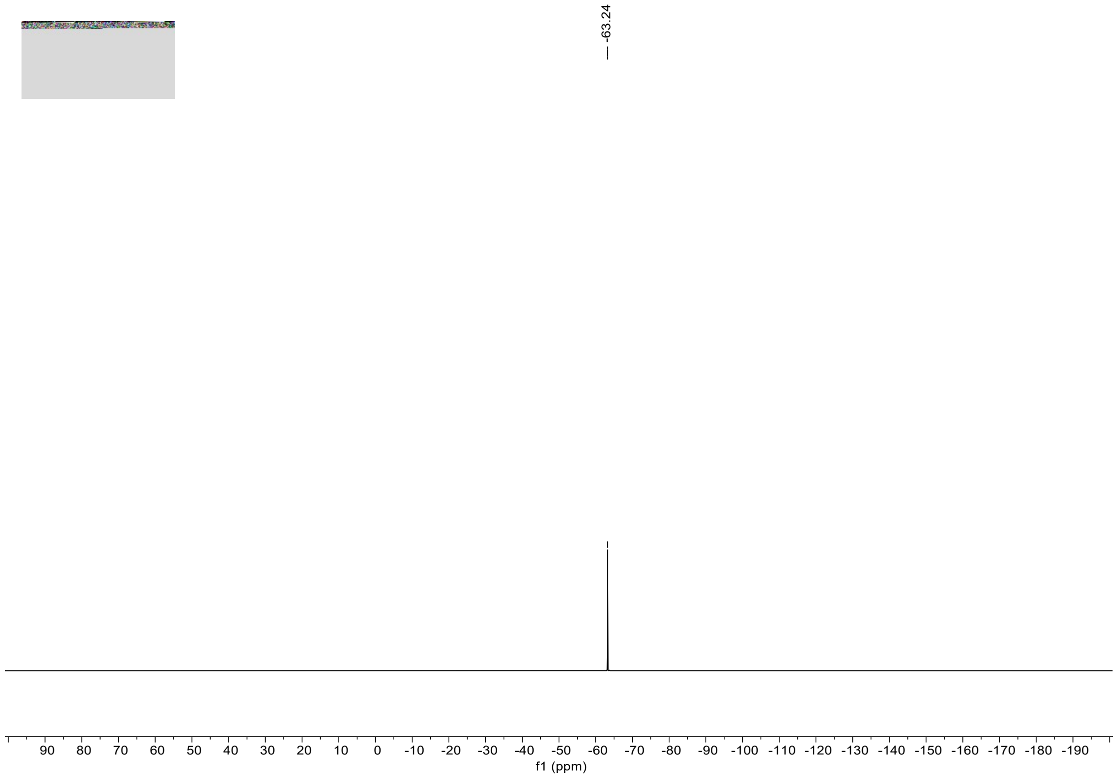
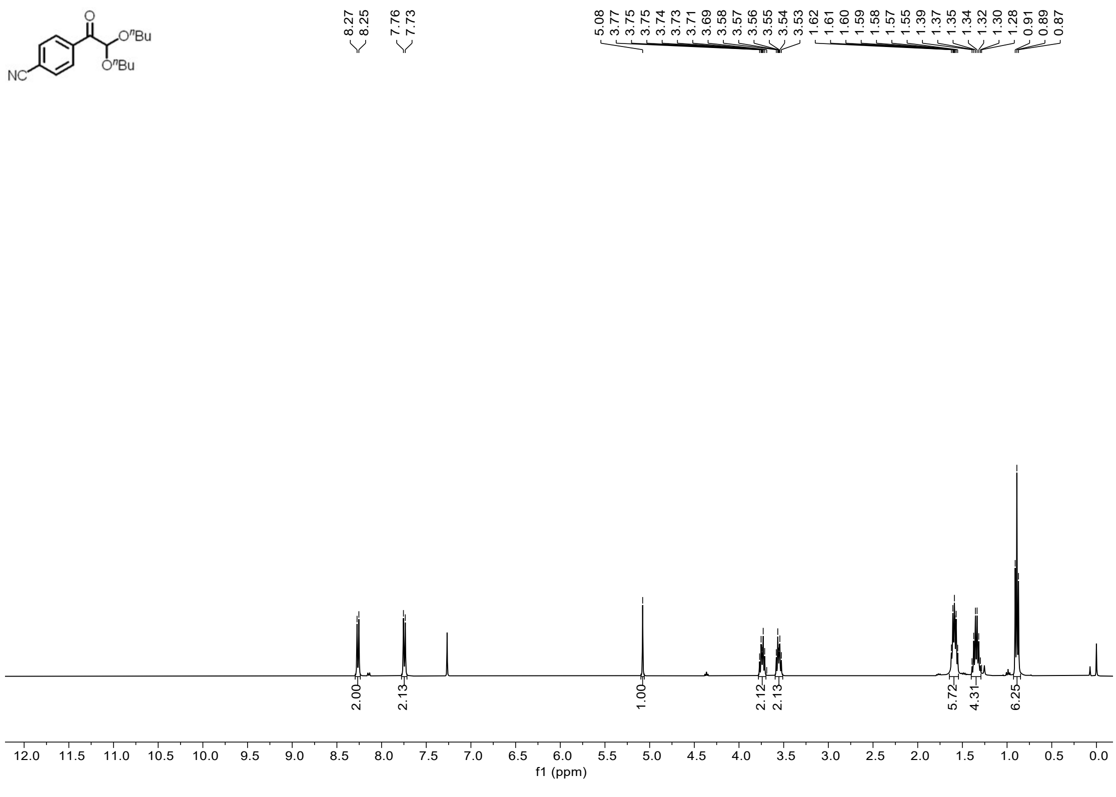
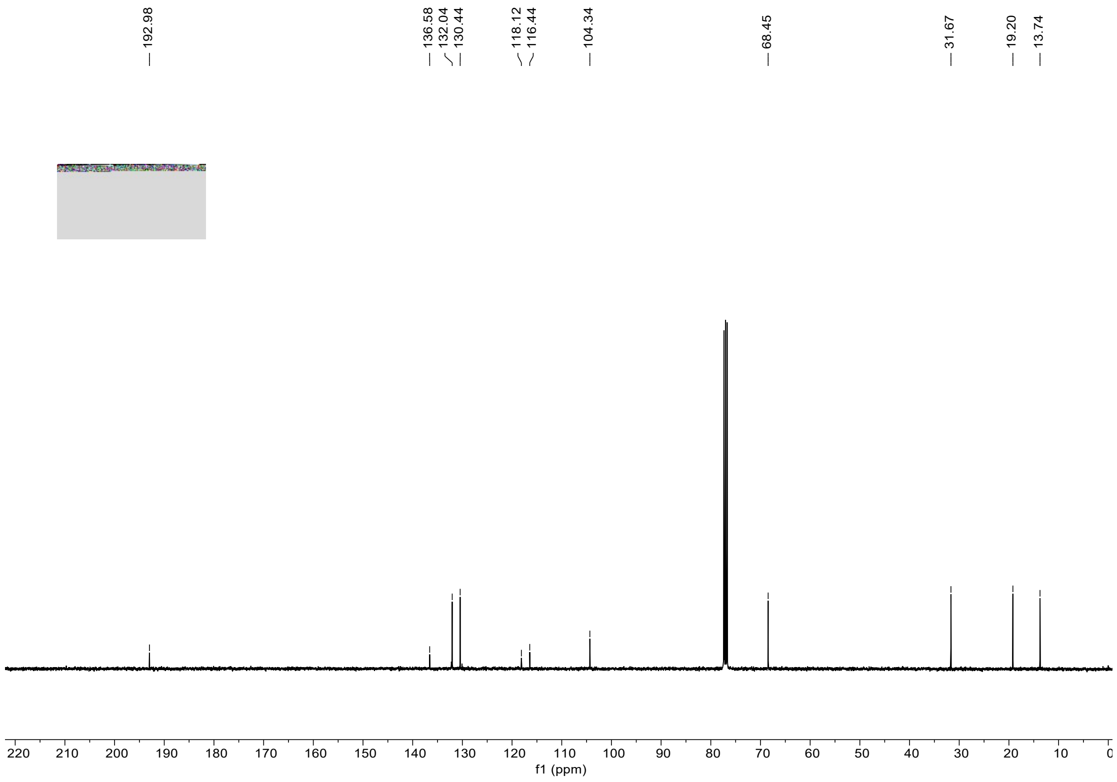
139.86
131.89
131.40
128.62
103.84
77.34 CDCl3
77.03 CDCl3
76.71 CDCl3
67.96
31.70
19.22
13.76
O
O^{n}Bu
O^{n}Bu
Cl
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

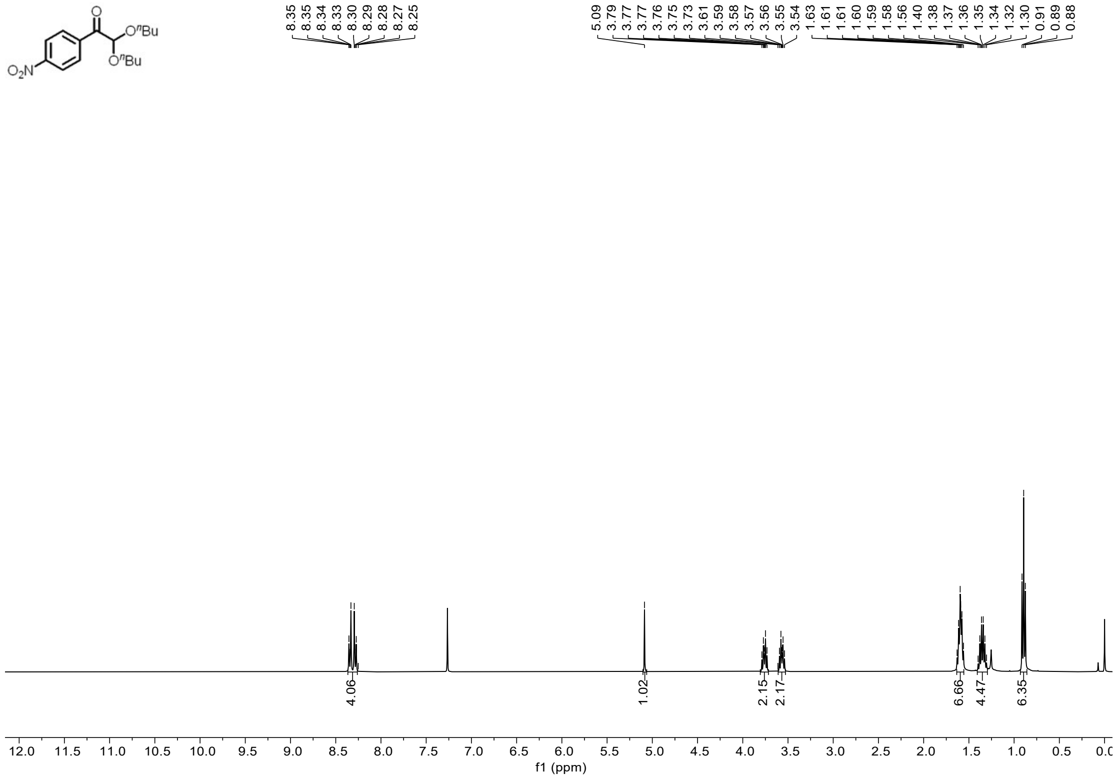

8.30
8.28
7.72
7.70
5.13
3.77
3.75
3.74
3.74
3.73
3.71
3.60
3.58
3.58
3.57
3.56
3.54
3.52
1.65
1.63
1.62
1.61
1.60
1.60
1.59
1.58
1.58
1.56
1.55
1.42
1.42
1.41
1.39
1.37
1.37
1.35
1.35
1.33
1.31
0.95
0.94
0.93
0.93
0.91
0.89
0.87
O
O^{n}Bu
O^{n}Bu
F$_3$C
2.00
2.15
0.97
2.06
2.07
4.27
4.25
6.23
12.0
11.5
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
f1 (ppm)

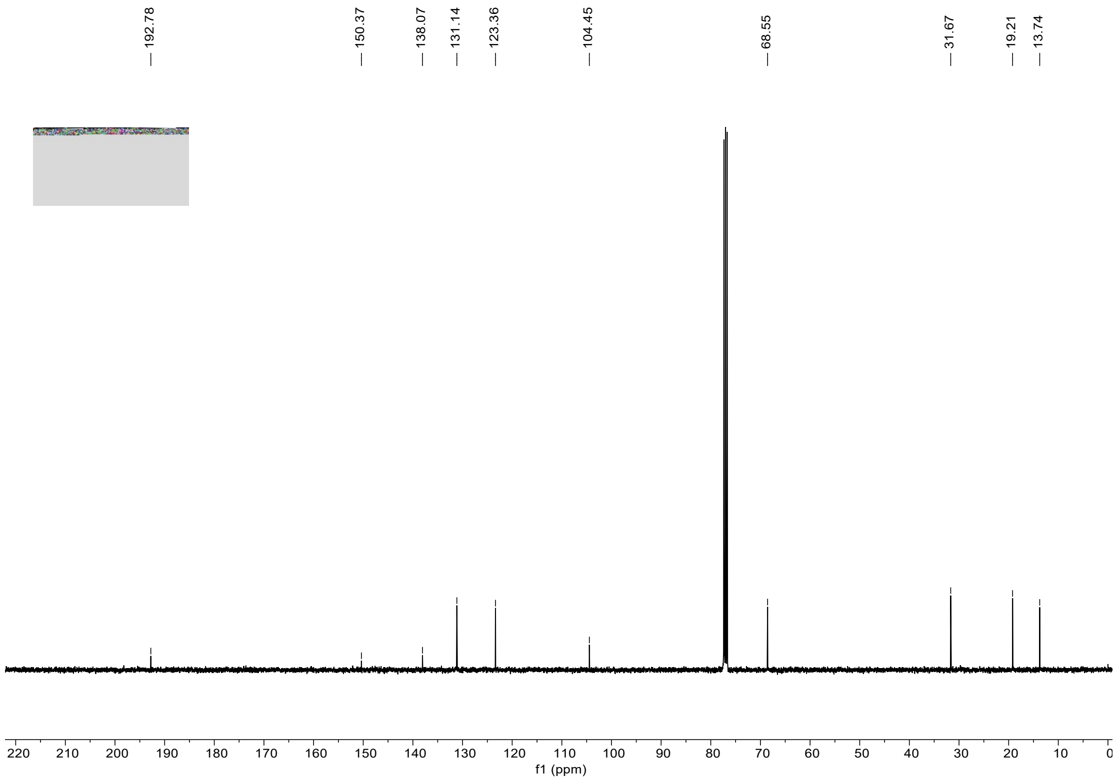

193.30
136.26
134.95
134.62
134.30
133.98
130.32
127.72
125.28
125.25
125.21
125.17
125.00
122.29
119.58
104.07
68.19
31.68
19.20
13.71
O
O^{n}Bu
O^{n}Bu
F$_3$C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

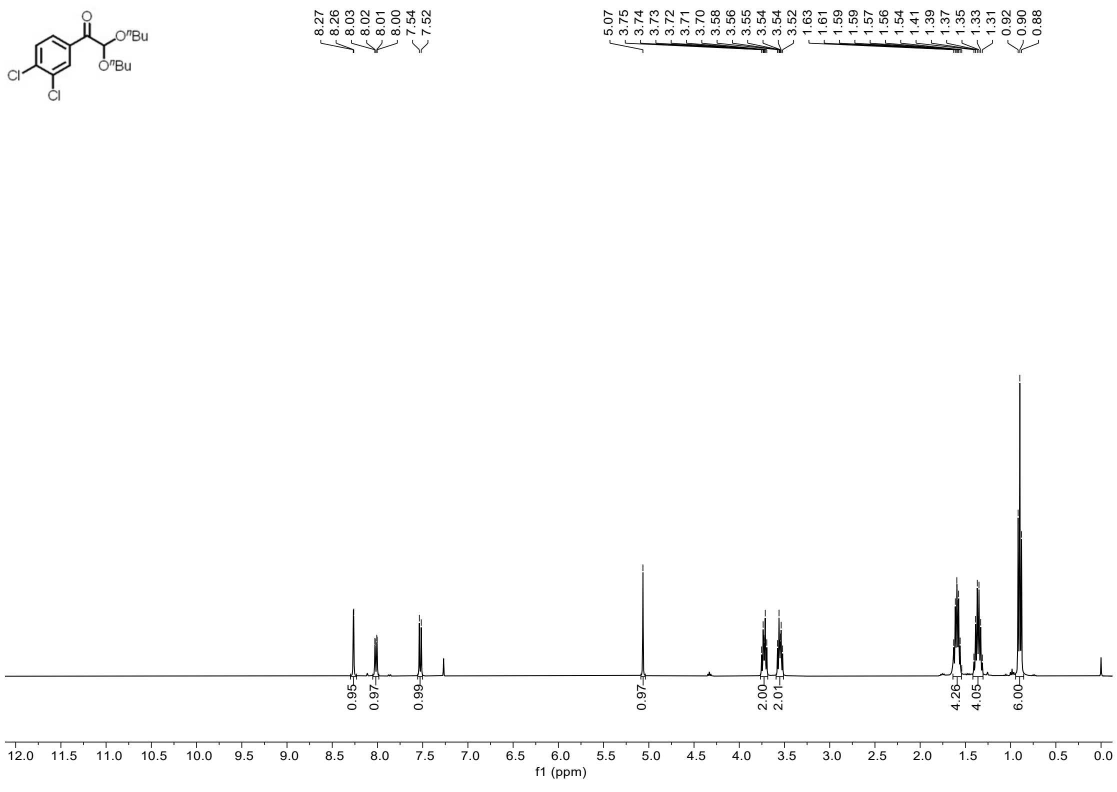

-63.24
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
f1 (ppm)

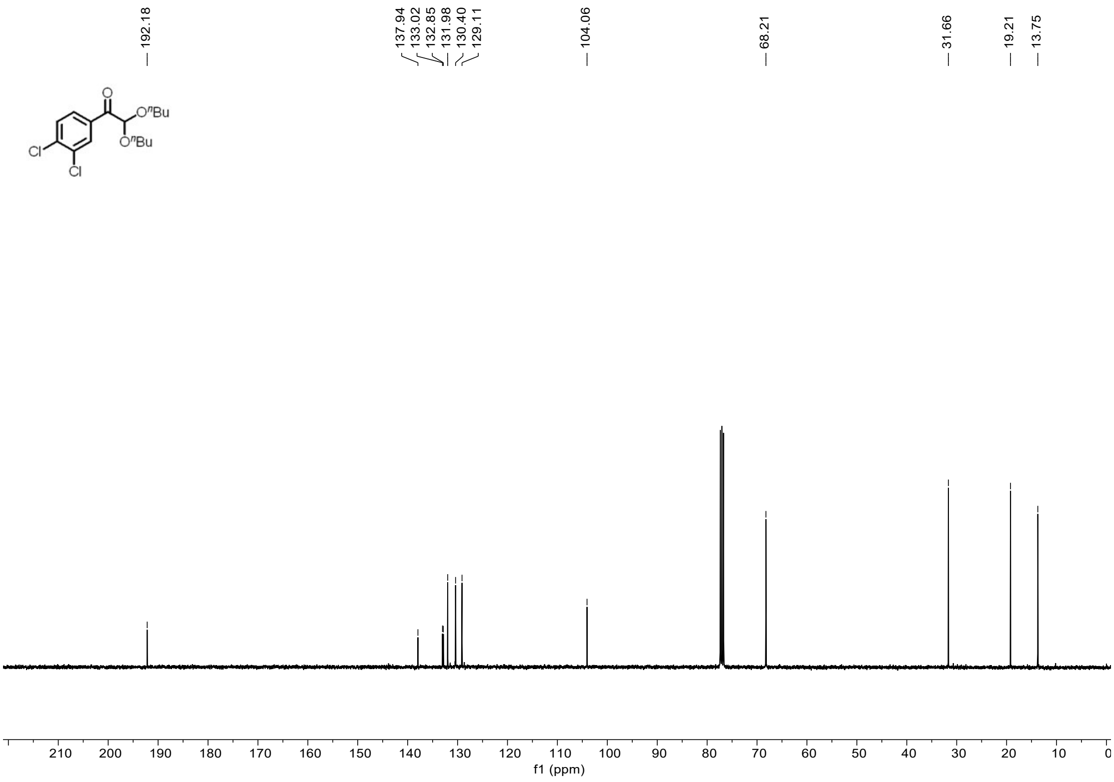

O
O^{n}Bu
O^{n}Bu
NC
8.27
8.25
7.76
7.73
5.08
3.77
3.75
3.75
3.74
3.73
3.71
3.69
3.58
3.57
3.56
3.55
3.54
3.53
1.62
1.61
1.60
1.59
1.58
1.57
1.55
1.39
1.37
1.35
1.34
1.32
1.30
1.28
0.91
0.89
0.87
2.00
2.13
1.00
2.12
2.13
5.72
4.31
6.25
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

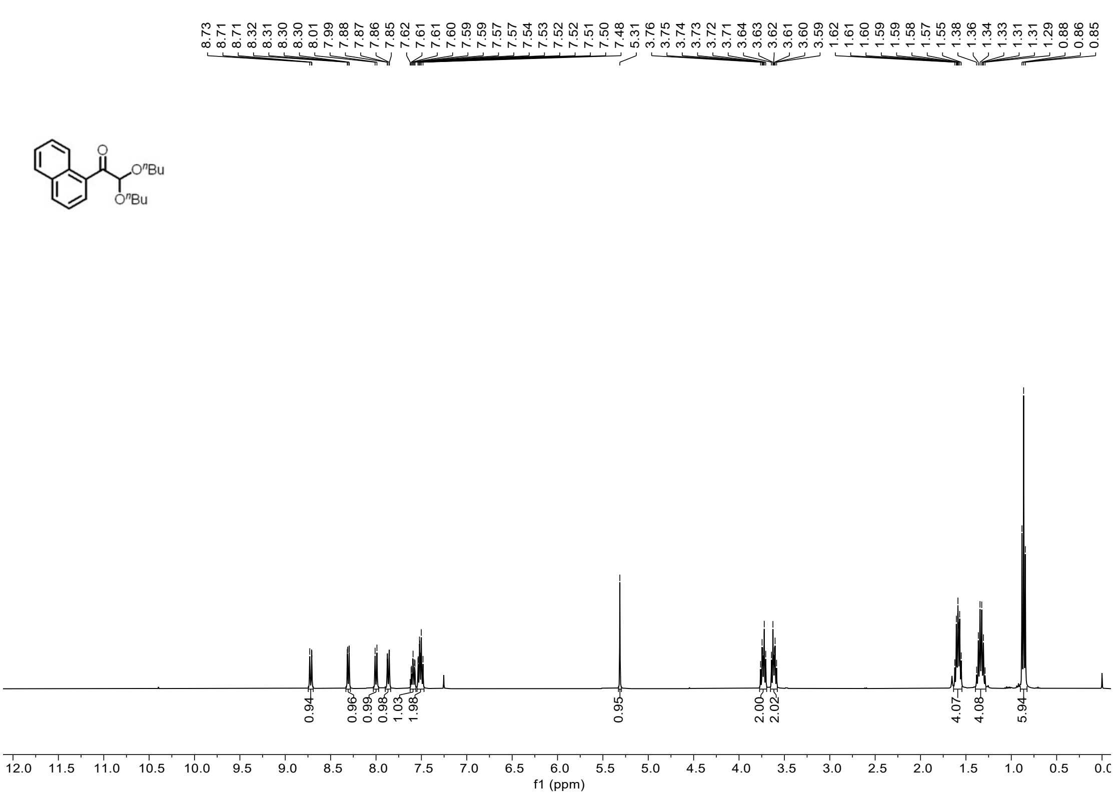

192.98
136.58
132.04
130.44
118.12
116.44
104.34
68.45
31.67
19.20
13.74
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

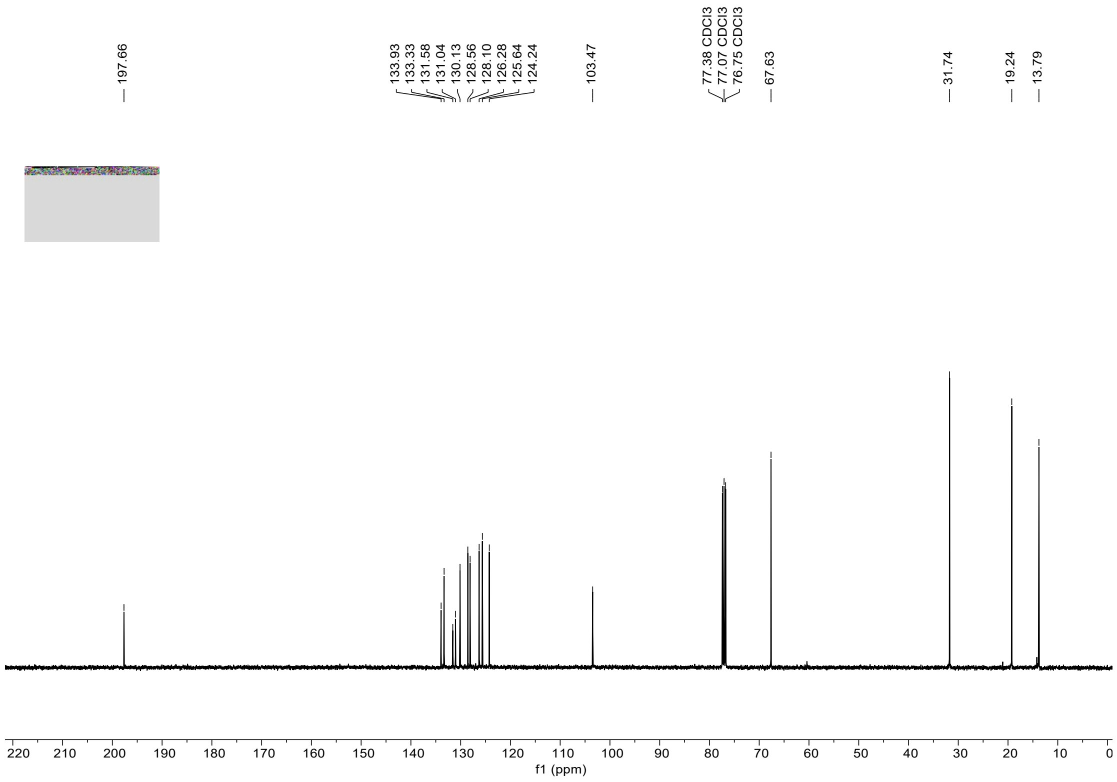

O
O^{n}Bu
O^{n}Bu
O$_2$N
8.35
8.35
8.34
8.33
8.30
8.29
8.28
8.27
8.25
5.09
3.79
3.77
3.77
3.76
3.75
3.73
3.61
3.59
3.58
3.57
3.56
3.55
3.54
1.63
1.61
1.61
1.60
1.59
1.58
1.56
1.40
1.38
1.37
1.36
1.35
1.34
1.32
1.30
0.91
0.89
0.88
4.06
1.02
2.15
2.17
6.66
4.47
6.35
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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
f1 (ppm)

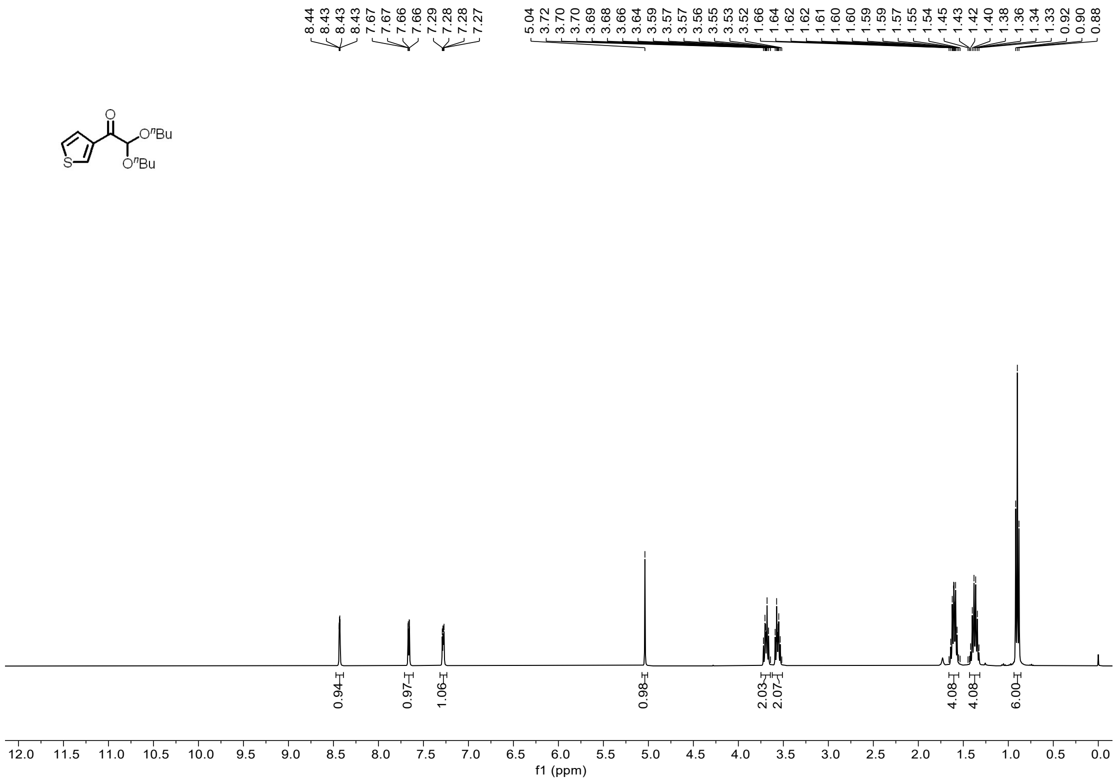

192.78
150.37
138.07
131.14
123.36
104.45
68.55
31.67
19.21
13.74
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

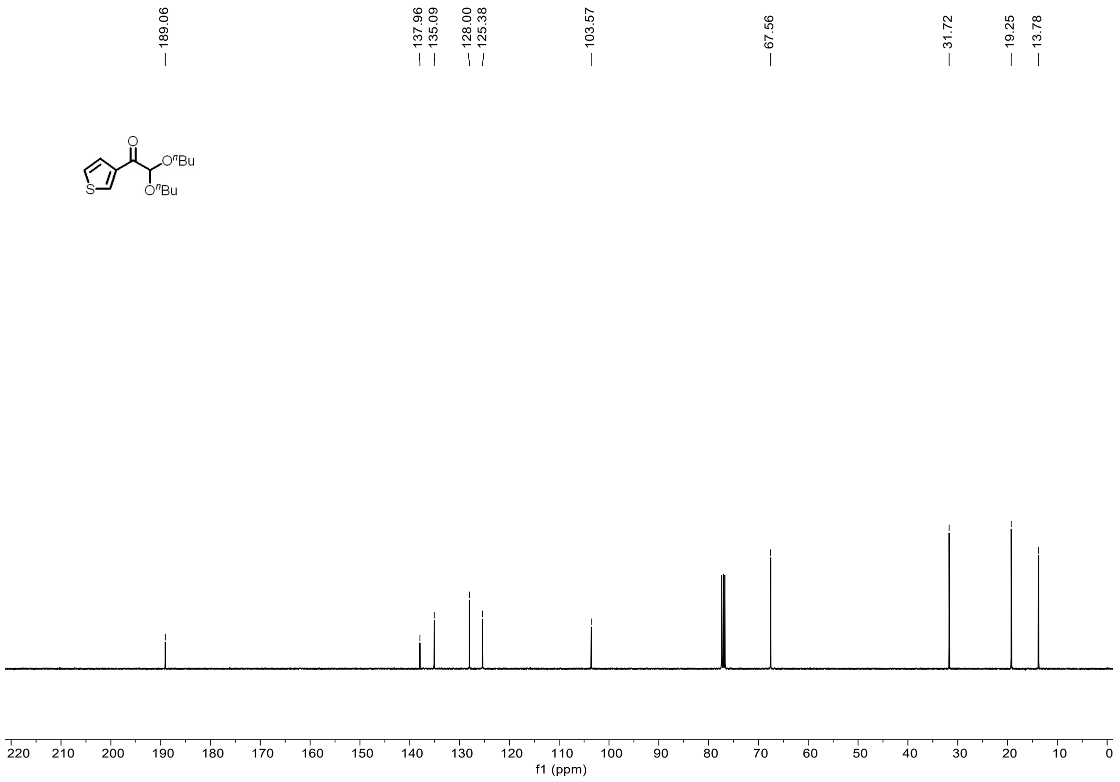

O
O^nBu
O^nBu
Cl
Cl
8.27
8.26
8.03
8.02
8.01
8.00
7.54
7.52
5.07
3.75
3.74
3.73
3.72
3.71
3.70
3.58
3.56
3.55
3.54
3.54
3.52
1.63
1.61
1.59
1.57
1.56
1.54
1.41
1.39
1.37
1.35
1.33
1.31
0.92
0.90
0.88
0.95
0.97
0.99
0.97
2.00
2.01
4.26
4.05
6.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

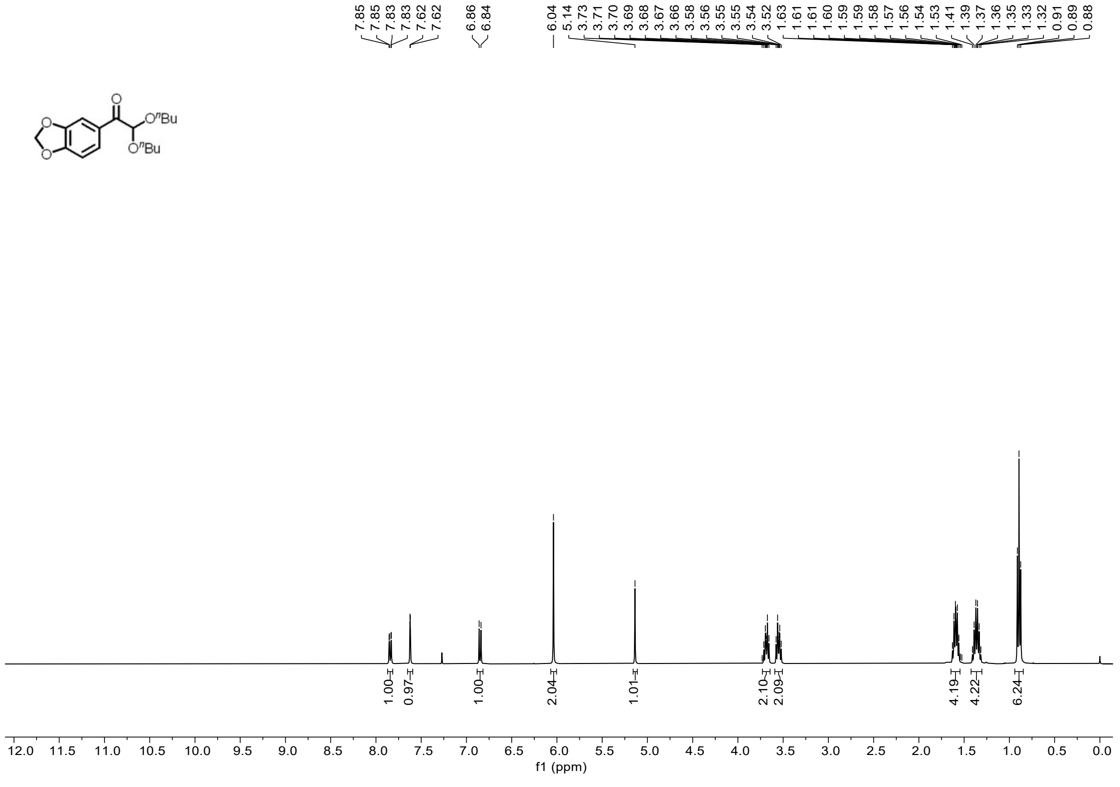

192.18
137.94
133.02
132.85
131.98
130.40
129.11
104.06
68.21
31.66
19.21
13.75
O
O^{n}Bu
O^{n}Bu
Cl
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

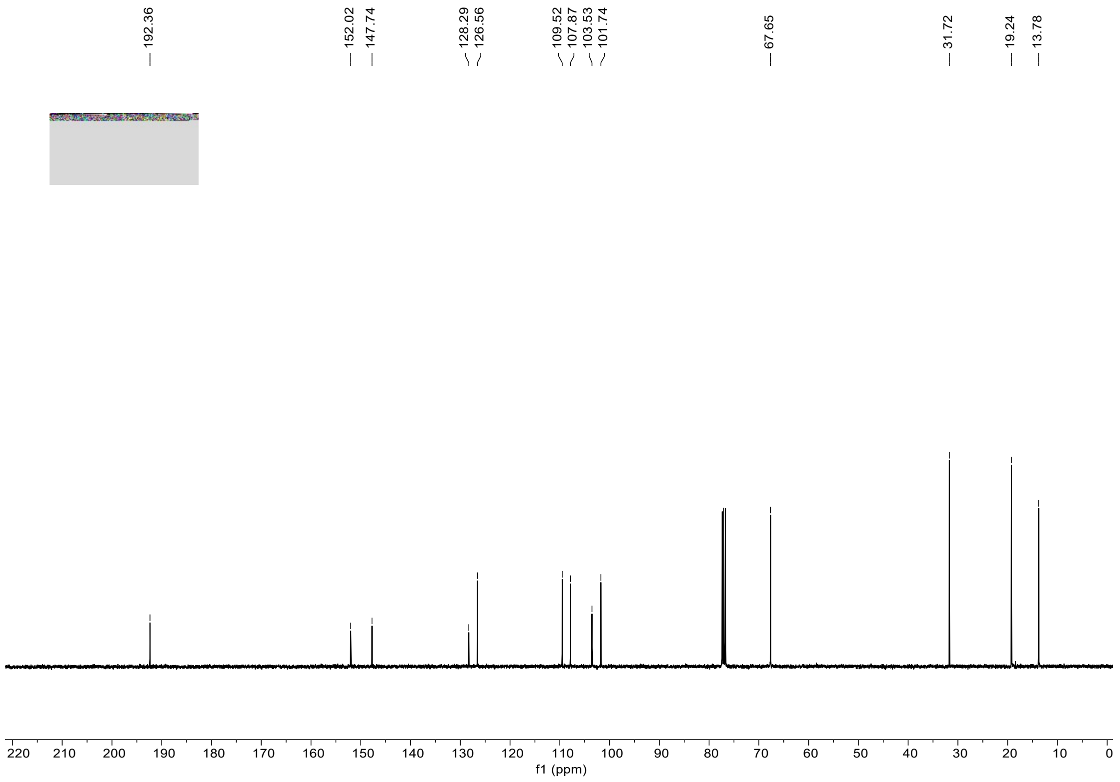

8.73
8.71
8.71
8.32
8.31
8.30
8.30
8.01
7.99
7.88
7.87
7.86
7.85
7.62
7.61
7.61
7.60
7.59
7.59
7.57
7.54
7.53
7.52
7.52
7.51
7.50
7.48
5.31
3.76
3.75
3.74
3.73
3.72
3.71
3.64
3.63
3.62
3.61
3.60
3.59
1.62
1.61
1.60
1.59
1.58
1.57
1.55
1.38
1.36
1.34
1.33
1.31
1.29
0.88
0.86
0.85
O
O^{n}Bu
O^{n}Bu
0.94
0.96
0.99
0.98
1.03
1.98
0.95
2.00
2.02
4.07
4.08
5.94
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
f1 (ppm)

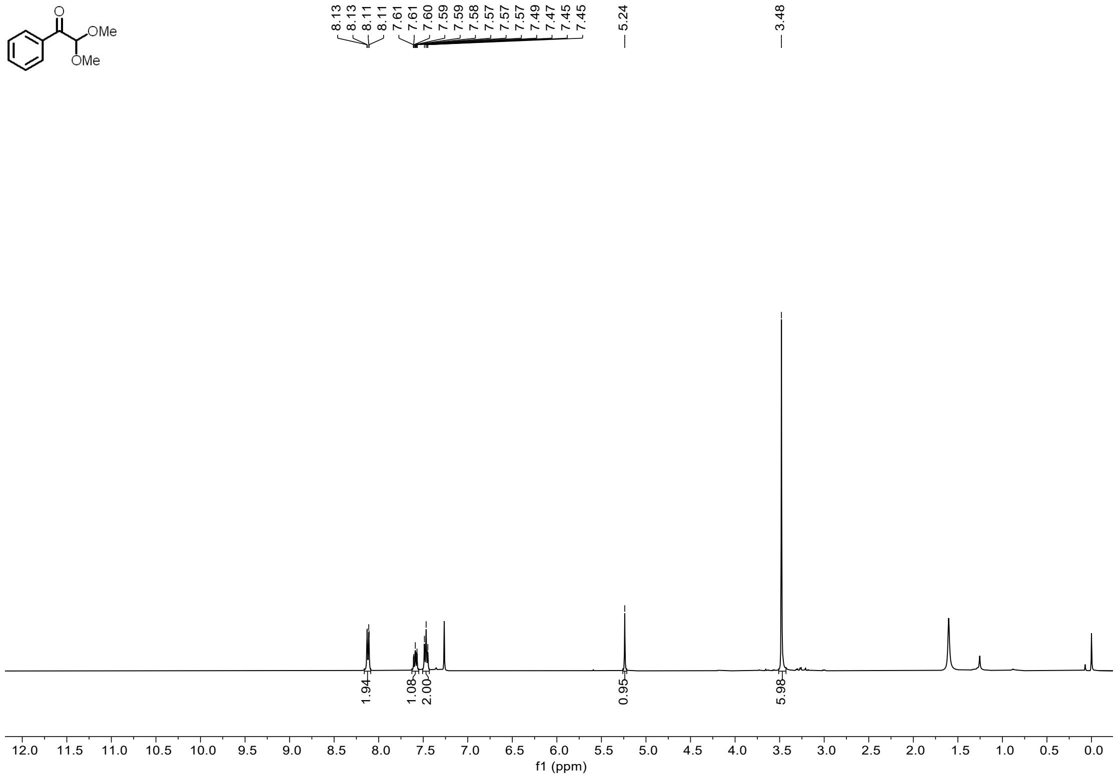

197.66
133.93
133.33
131.58
131.04
130.13
128.56
128.10
126.28
125.64
124.24
103.47
77.38 CDCl3
77.07 CDCl3
76.75 CDCl3
67.63
31.74
19.24
13.79
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

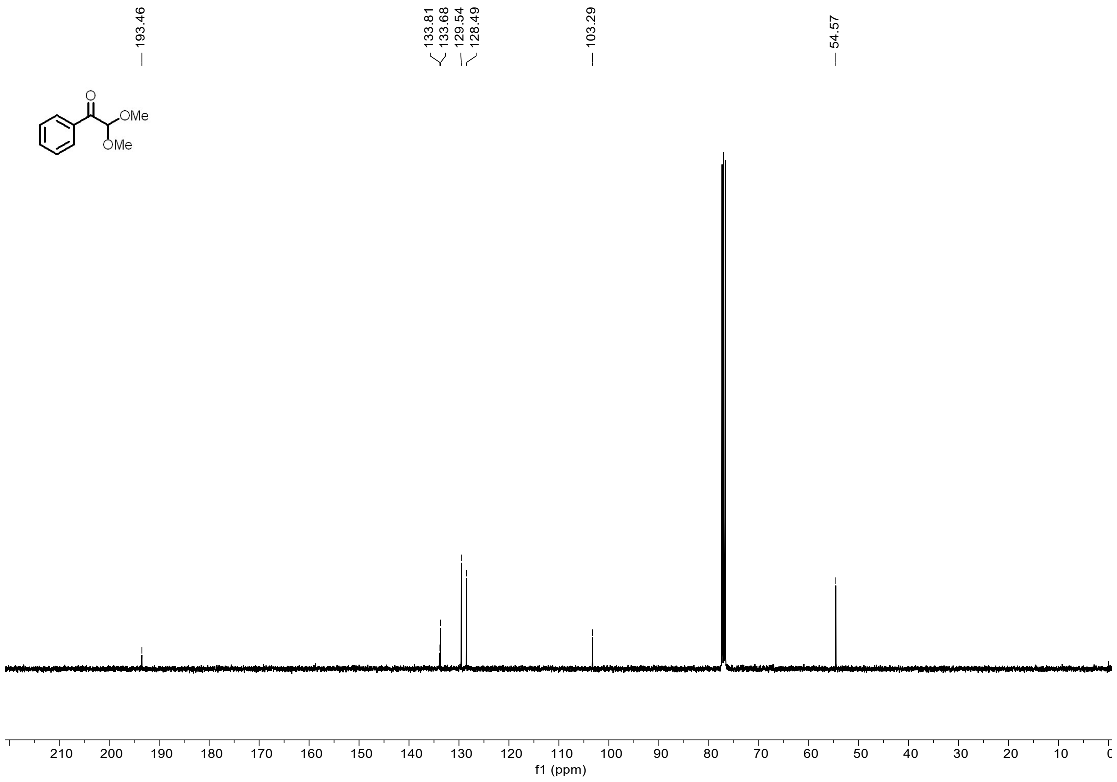

8.44
8.43
8.43
8.43
7.67
7.67
7.66
7.66
7.29
7.28
7.28
7.27
5.04
3.72
3.70
3.69
3.68
3.66
3.64
3.59
3.57
3.56
3.55
3.53
3.52
1.66
1.64
1.62
1.62
1.61
1.60
1.60
1.59
1.57
1.55
1.54
1.45
1.43
1.42
1.40
1.38
1.36
1.34
1.33
0.92
0.90
0.88
O
S
O^{n}Bu
O^{n}Bu
0.94
0.97
1.06
0.98
2.03
2.07
4.08
4.08
6.00
12.0 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
f1 (ppm)

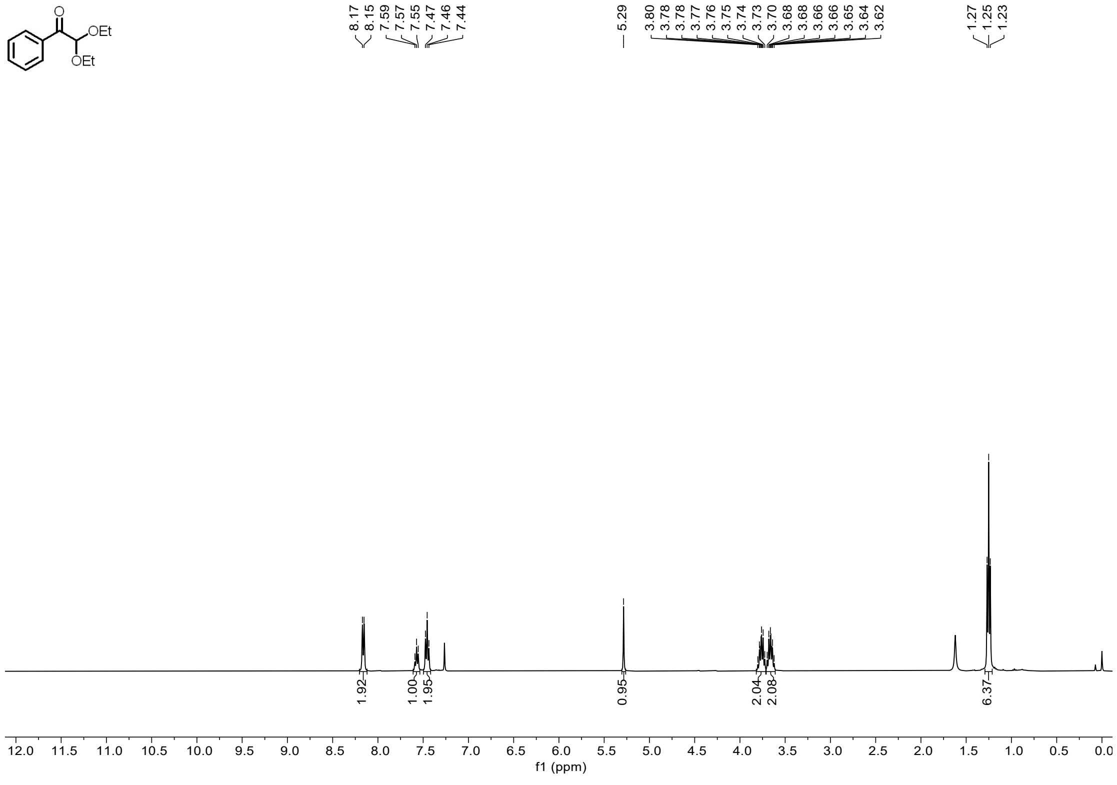

189.06
137.96
135.09
128.00
125.38
103.57
67.56
31.72
19.25
13.78
O
O^{n}Bu
O^{n}Bu
S
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

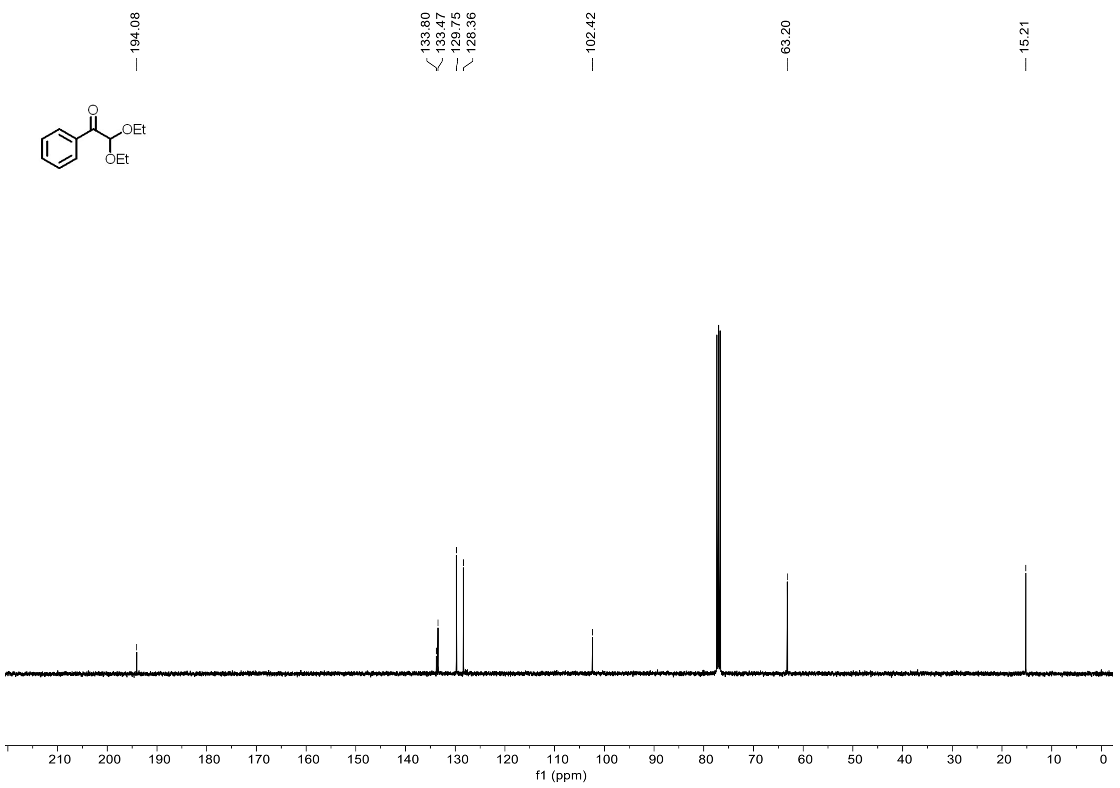

7.85
7.85
7.83
7.83
7.62
7.62
6.86
6.84
6.04
5.14
3.73
3.71
3.70
3.69
3.68
3.67
3.66
3.58
3.56
3.55
3.54
3.52
1.63
1.61
1.60
1.59
1.58
1.57
1.56
1.54
1.53
1.41
1.39
1.37
1.36
1.35
1.33
1.32
0.91
0.89
0.88
O
O
O
O^{n}Bu
O^{n}Bu
1.00
0.97
1.00
2.04
1.01
2.10
2.09
4.19
4.22
6.24
12.0
11.5
11.0
10.5
10.0
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
f1 (ppm)

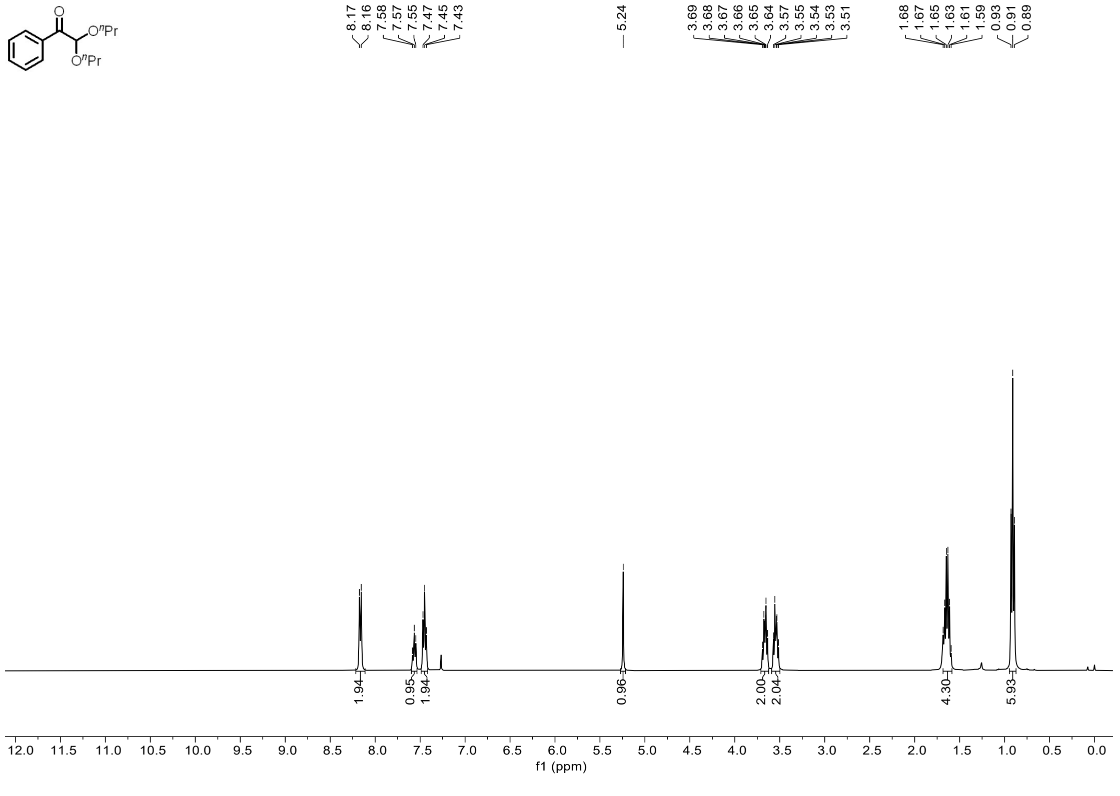

192.36
152.02
147.74
128.29
126.56
109.52
107.87
103.53
101.74
67.65
31.72
19.24
13.78
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

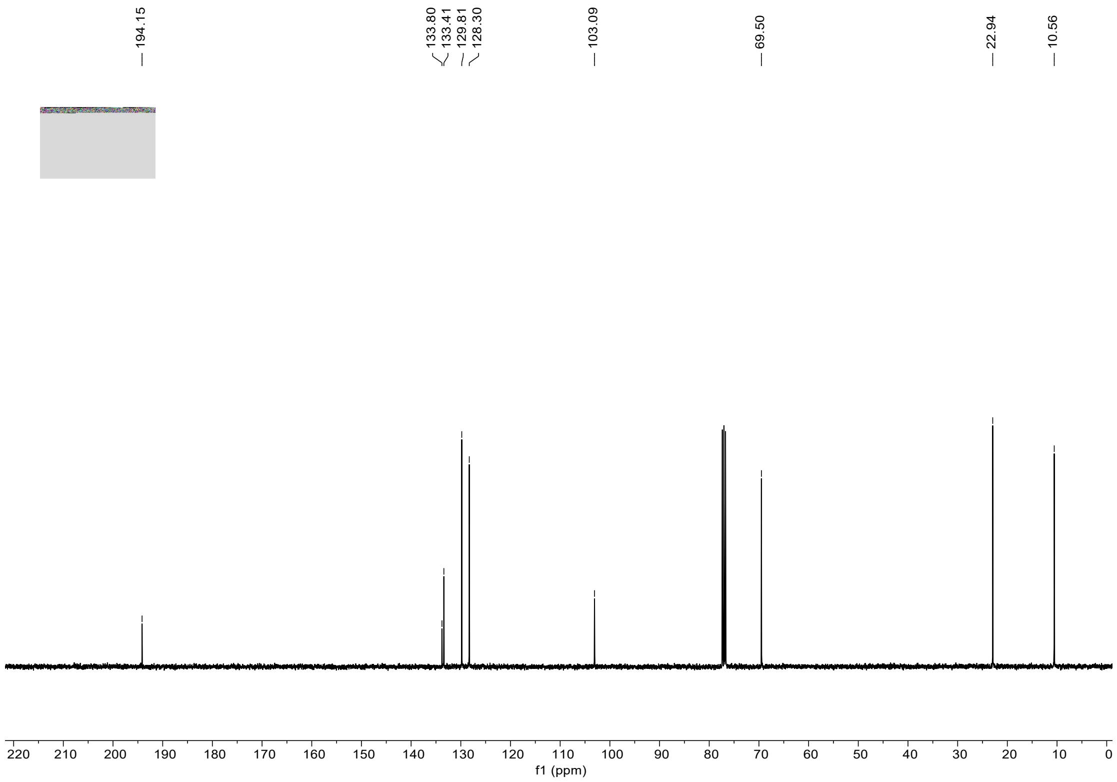

O
OMe
OMe
8.13
8.13
8.11
8.11
7.61
7.60
7.59
7.58
7.57
7.57
7.57
7.49
7.47
7.45
7.45
5.24
3.48
1.94
1.08
2.00
0.95
5.98
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

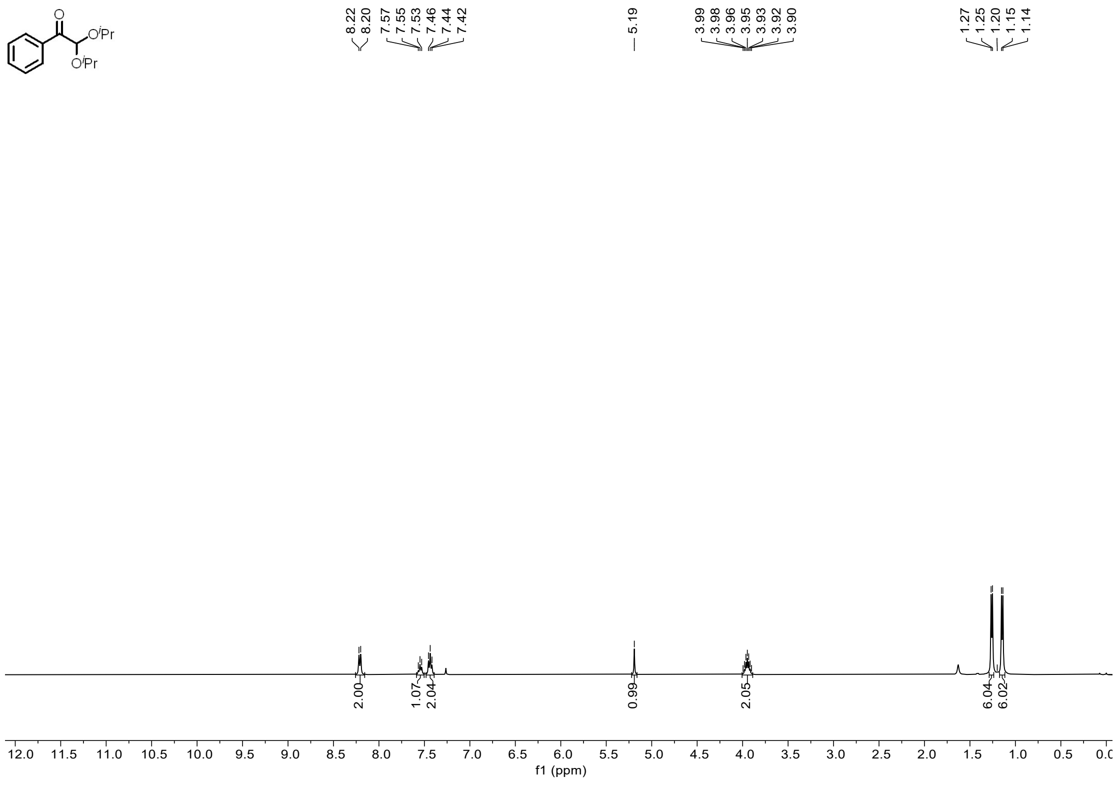

193.46
133.81
133.68
129.54
128.49
103.29
54.57
O
OMe
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

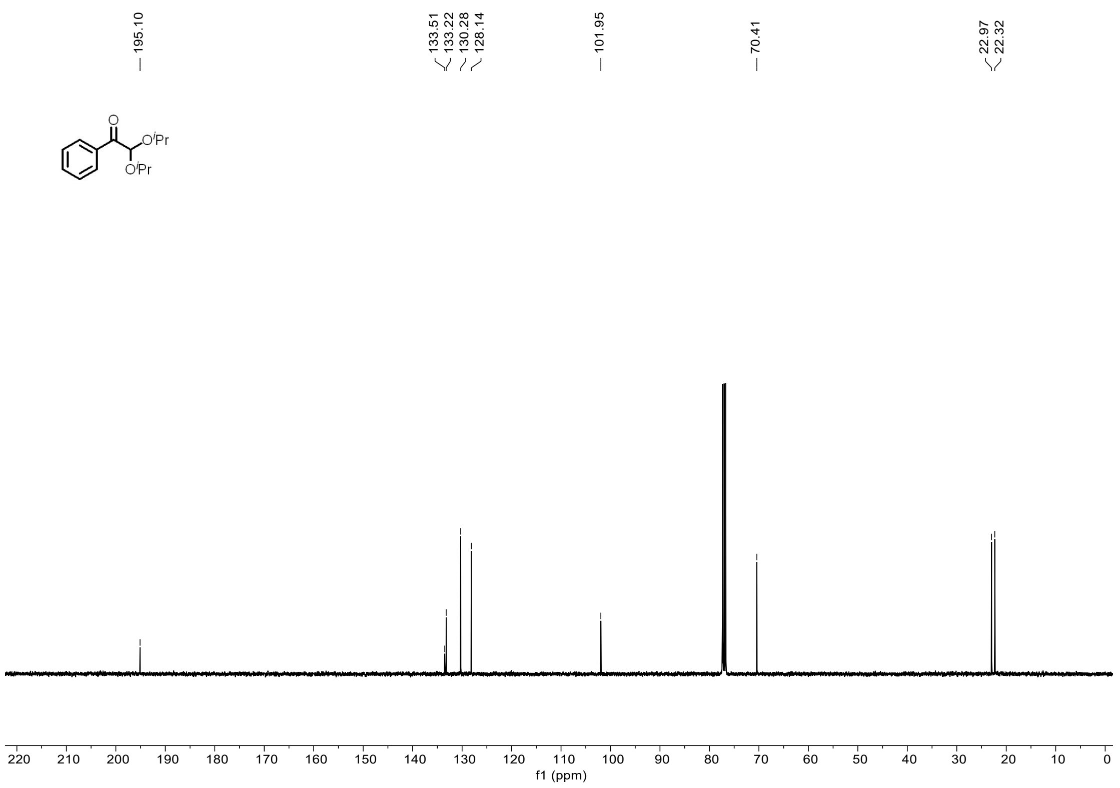

8.17
8.15
7.59
7.57
7.55
7.47
7.46
7.44
5.29
3.80
3.78
3.78
3.77
3.76
3.75
3.74
3.73
3.70
3.68
3.66
3.66
3.65
3.64
3.62
1.27
1.25
1.23
O
OEt
OEt
1.92
1.00
1.95
0.95
2.04
2.08
6.37
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

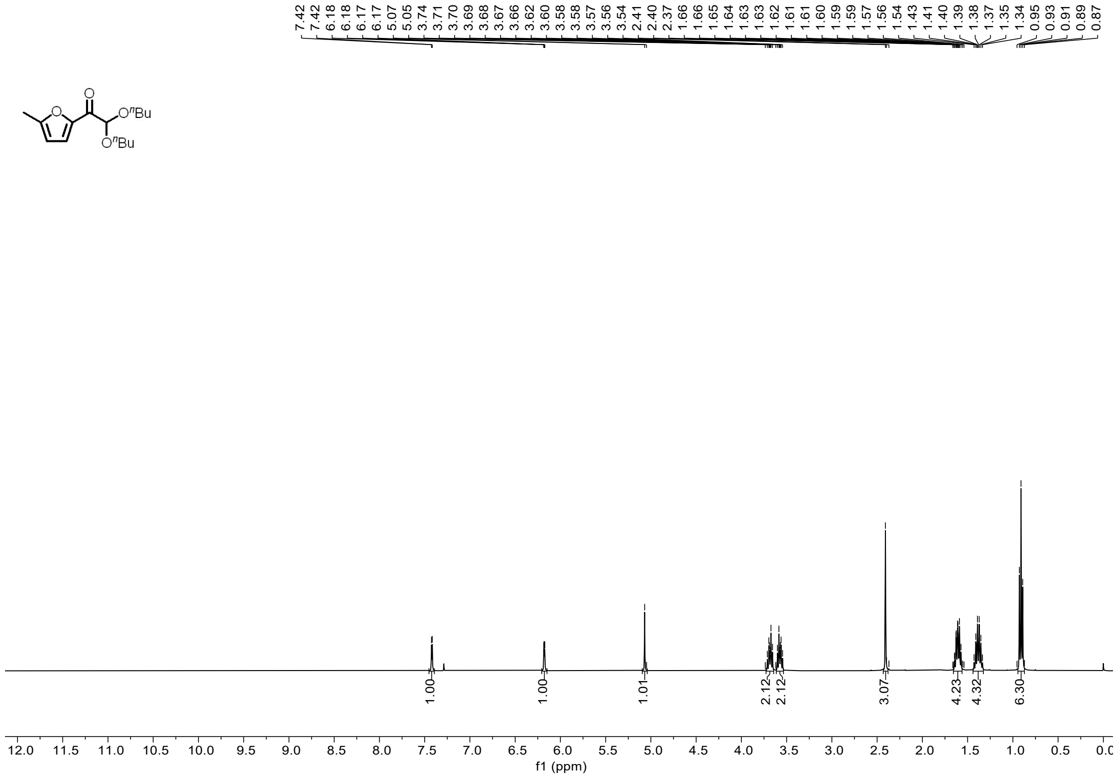

194.08
133.80
133.47
129.75
128.36
102.42
63.20
15.21
O
OEt
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

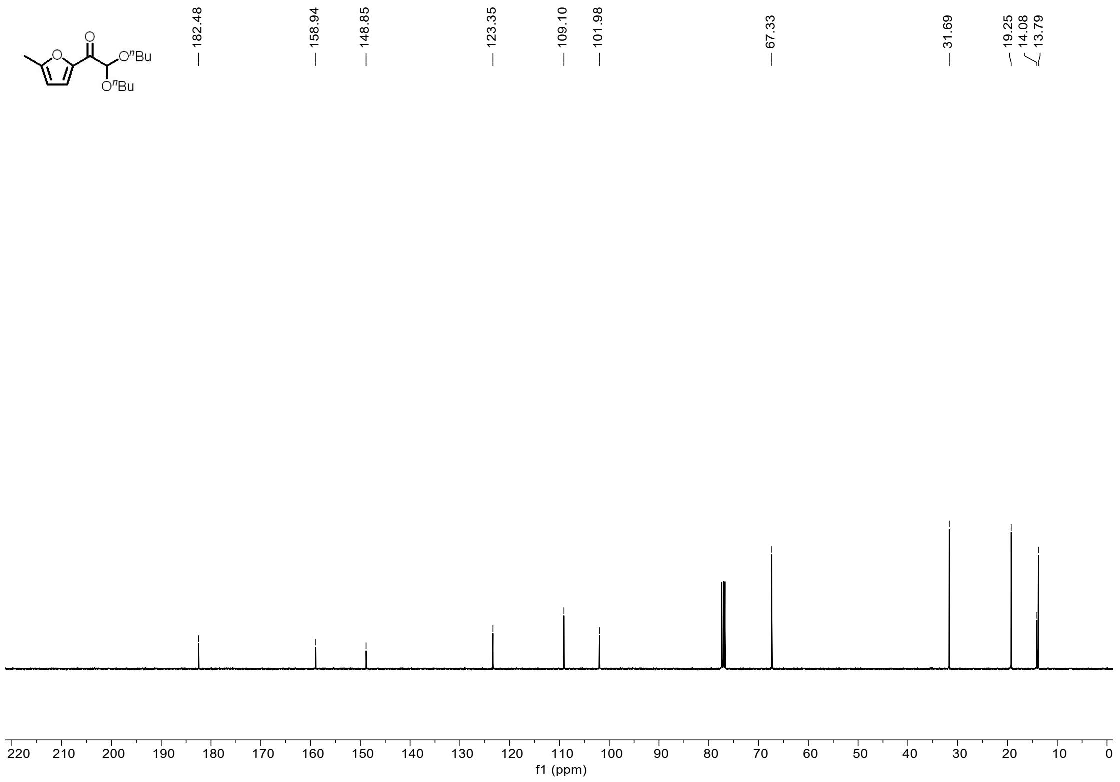

O
O^{n}Pr
O^{n}Pr
8.17
8.16
7.58
7.57
7.55
7.47
7.45
7.43
5.24
3.69
3.68
3.67
3.66
3.65
3.64
3.57
3.55
3.54
3.53
3.51
1.68
1.67
1.65
1.63
1.61
1.59
0.93
0.91
0.89
1.94
0.95
1.94
0.96
2.00
2.04
4.30
5.93
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

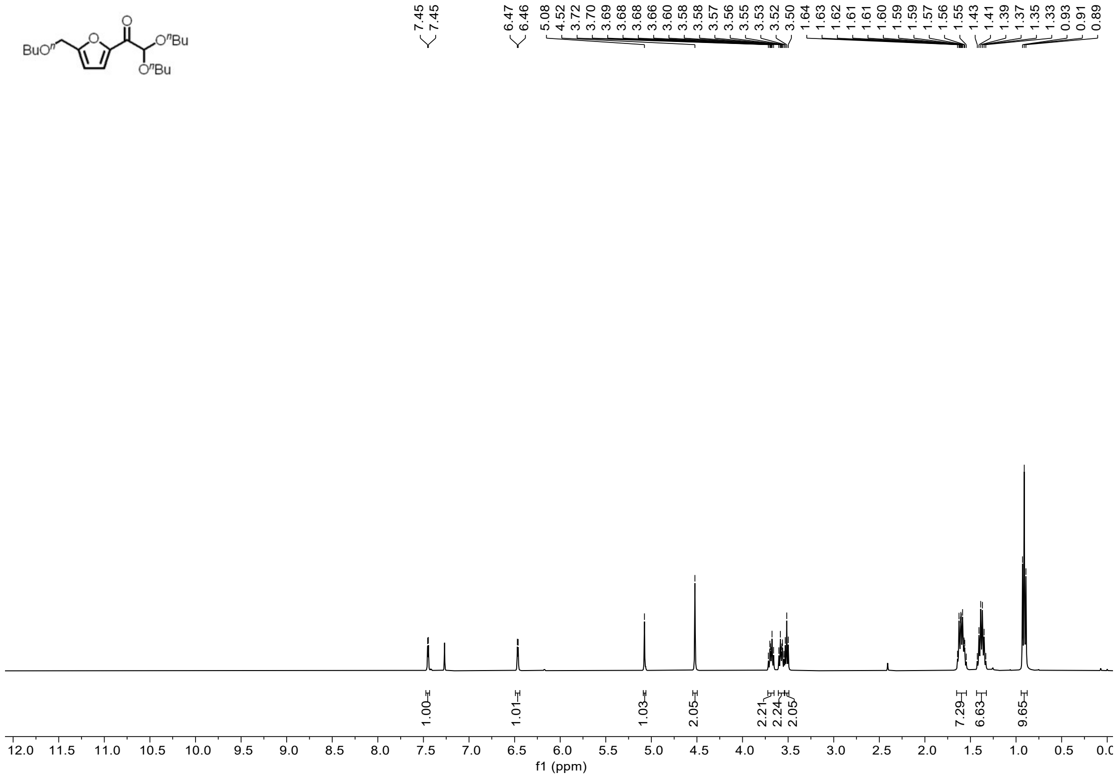

194.15
133.80
133.41
129.81
128.30
103.09
69.50
22.94
10.56
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

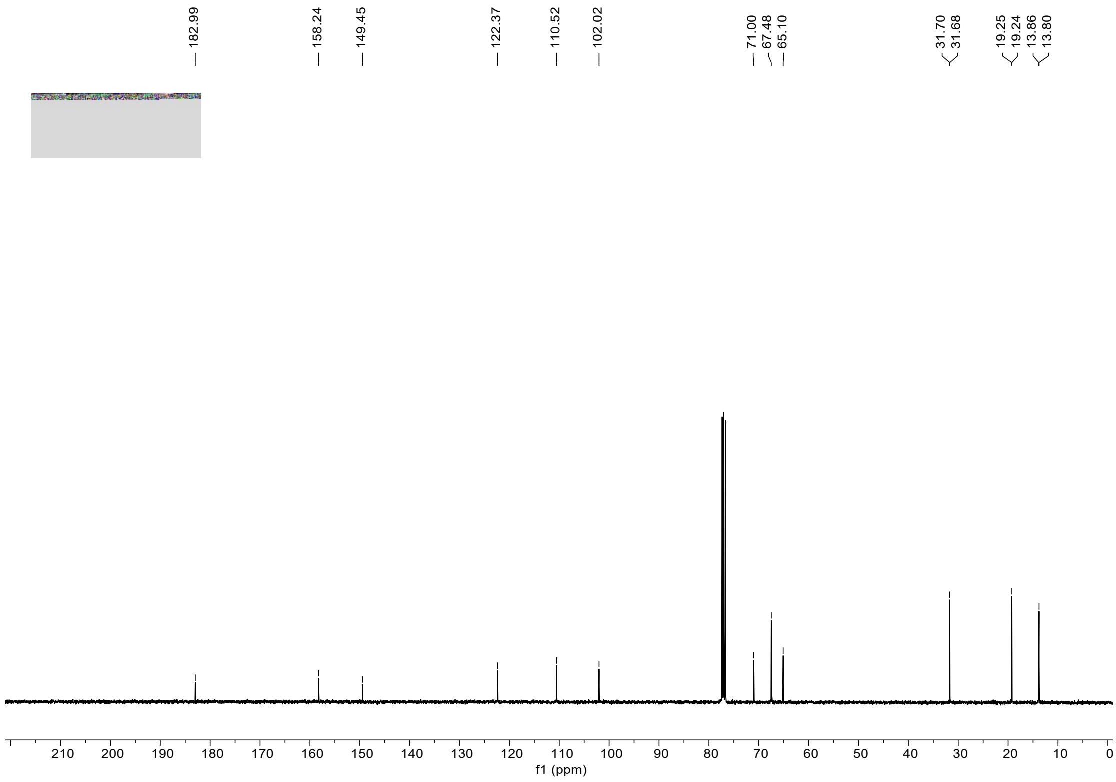

O
O^{i}Pr
O^{i}Pr
8.22
8.20
7.57
7.55
7.53
7.46
7.44
7.42
5.19
3.99
3.98
3.96
3.95
3.93
3.92
3.90
1.27
1.25
1.20
1.15
1.14
2.00
1.07
2.04
0.99
2.05
6.04
6.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

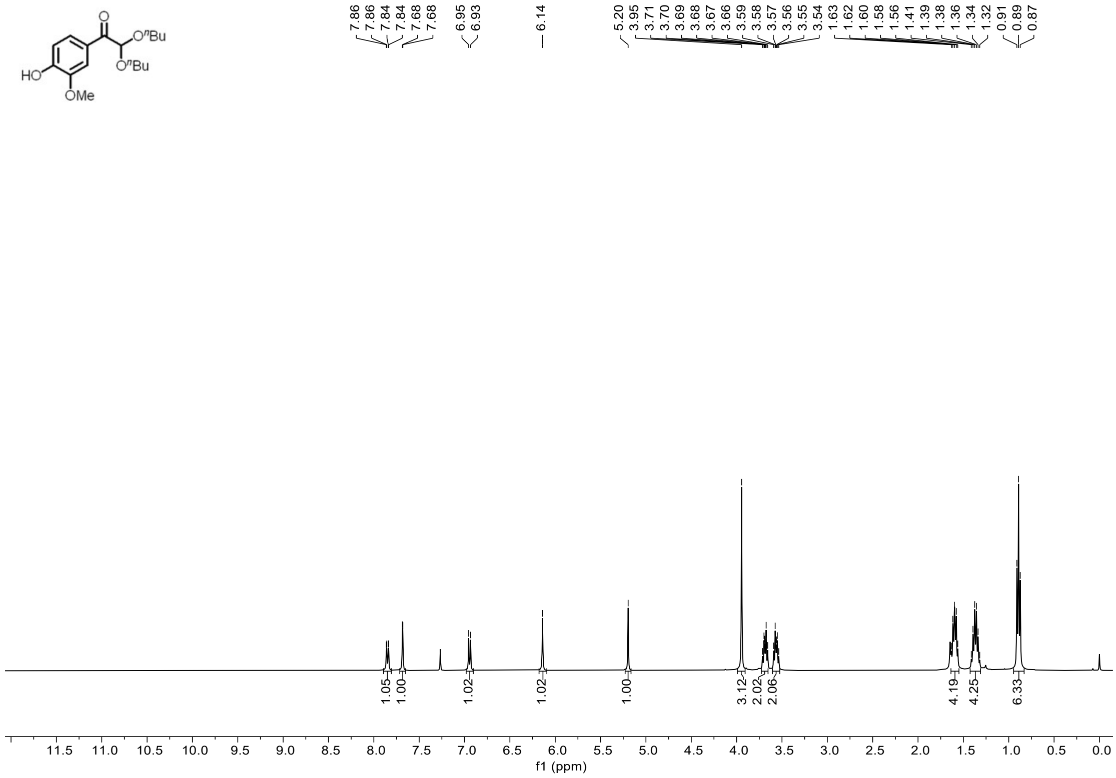

195.10
133.51
133.22
130.28
128.14
101.95
70.41
22.97
22.32
O
O^{i}Pr
O^{i}Pr
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

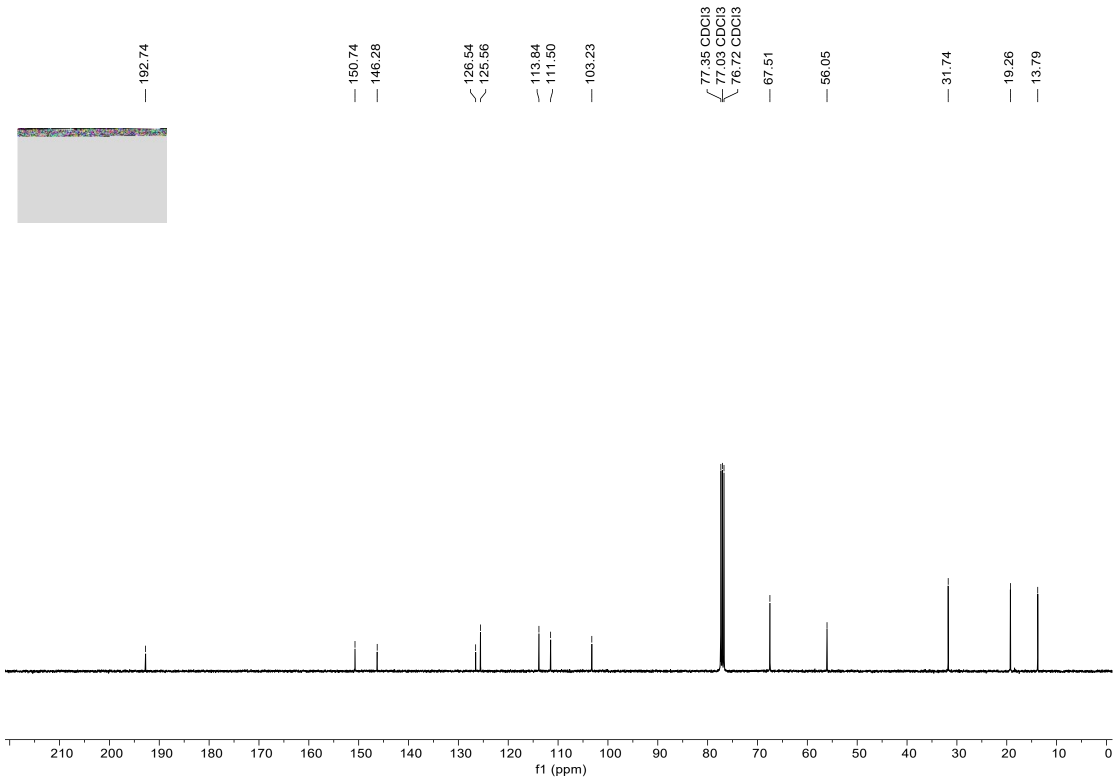

7.42
7.42
6.18
6.18
6.17
6.17
5.07
5.05
3.74
3.71
3.70
3.69
3.68
3.67
3.66
3.62
3.60
3.58
3.58
3.57
3.56
3.54
2.41
2.40
2.37
1.66
1.66
1.65
1.64
1.63
1.63
1.62
1.61
1.61
1.60
1.59
1.59
1.57
1.56
1.54
1.43
1.41
1.40
1.39
1.38
1.37
1.35
1.34
0.95
0.93
0.91
0.89
0.87
O
O
O^{n}Bu
O^{n}Bu
1.00
1.00
1.01
2.12
2.12
3.07
4.23
4.32
6.30
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
f1 (ppm)

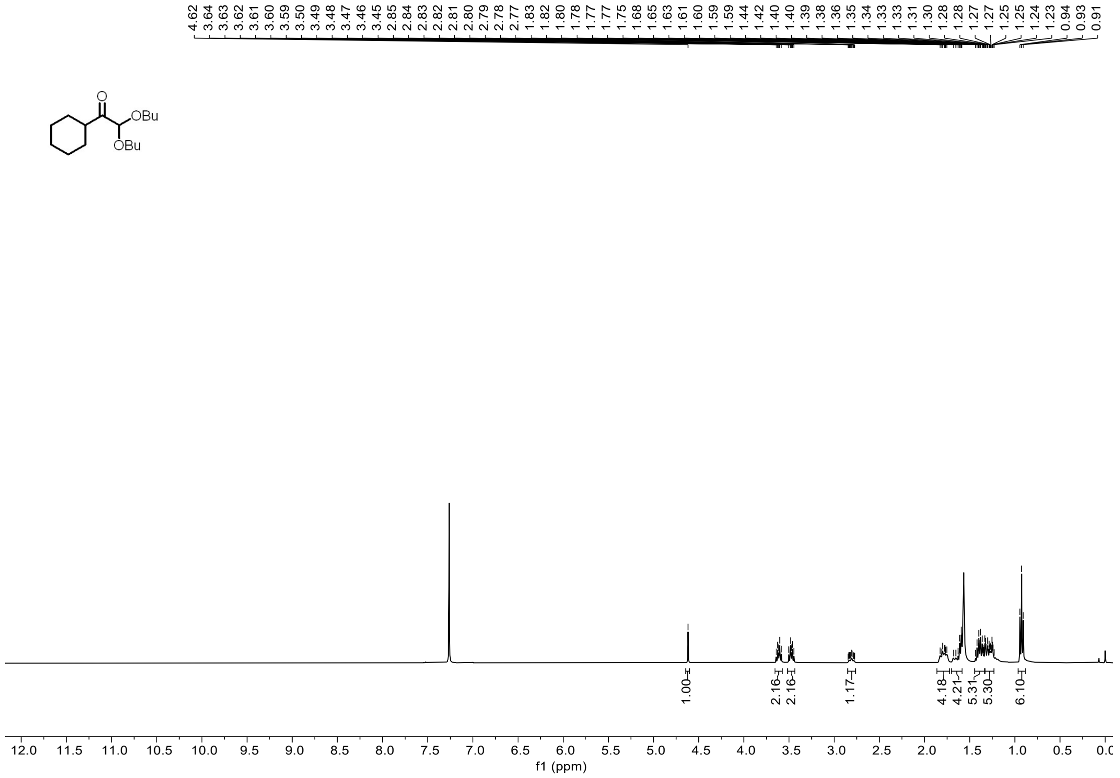

O
O^{n}Bu
O^{n}Bu
182.48
158.94
148.85
123.35
109.10
101.98
67.33
31.69
19.25
14.08
13.79
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

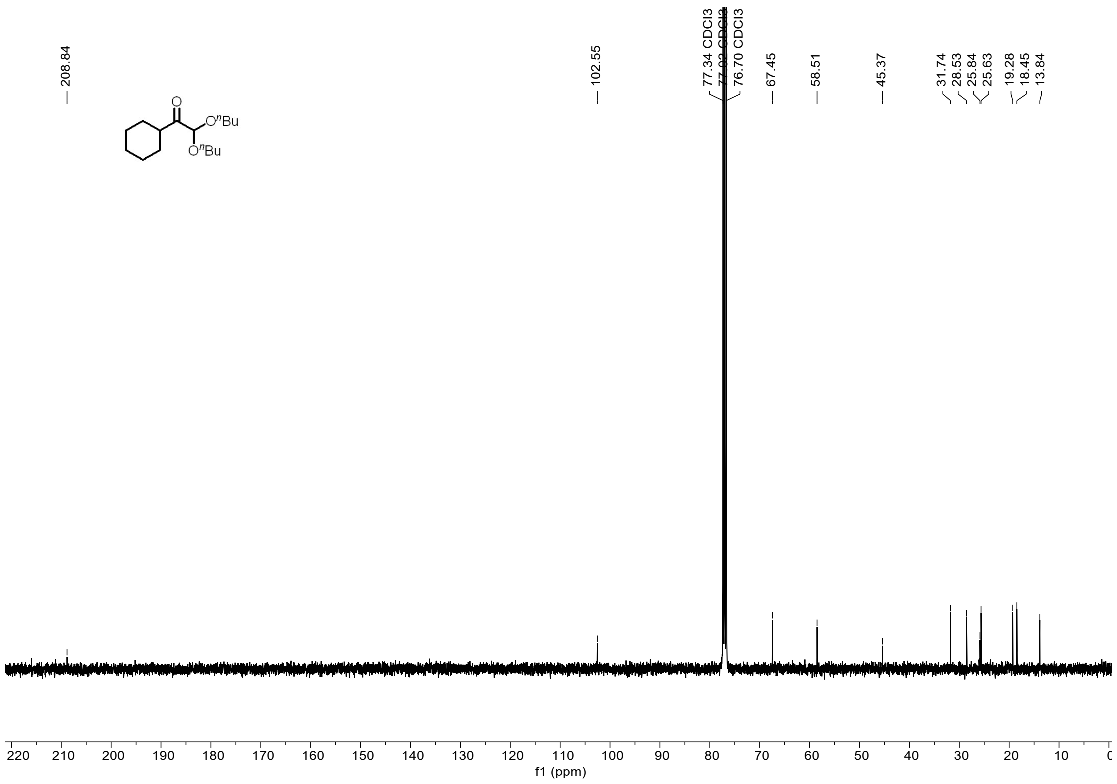

BuO^n
O
O^{n}Bu
O^{n}Bu
7.45
7.45
6.47
6.46
5.08
4.52
3.72
3.70
3.69
3.68
3.66
3.60
3.58
3.58
3.57
3.56
3.55
3.53
3.52
3.50
1.64
1.63
1.62
1.61
1.61
1.60
1.59
1.59
1.57
1.56
1.55
1.43
1.41
1.39
1.37
1.35
1.33
0.93
0.91
0.89
1.00
1.01
1.03
2.05
2.21
2.24
2.05
7.29
6.63
9.65
12.0 11.5 11.0 10.5 10.0 9.5 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
f1 (ppm)


182.99
158.24
149.45
122.37
110.52
102.02
71.00
67.48
65.10
31.70
31.68
19.25
19.24
13.86
13.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
O^{n}Bu
O^{n}Bu
HO
OMe
7.86
7.86
7.84
7.84
7.68
7.68
6.95
6.93
6.14
5.20
3.95
3.71
3.70
3.69
3.68
3.67
3.66
3.59
3.58
3.57
3.56
3.55
3.54
1.63
1.62
1.60
1.58
1.56
1.41
1.39
1.38
1.36
1.34
1.32
0.91
0.89
0.87
1.05
1.00
1.02
1.02
1.00
3.12
2.02
2.06
4.19
4.25
6.33
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
f1 (ppm)


192.74
150.74
146.28
126.54
125.56
113.84
111.50
103.23
77.35 CDCl3
77.03 CDCl3
76.72 CDCl3
67.51
56.05
31.74
19.26
13.79
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


4.62
3.64
3.63
3.62
3.61
3.60
3.59
3.50
3.49
3.48
3.47
3.46
3.45
2.85
2.84
2.83
2.82
2.81
2.80
2.79
2.78
2.77
1.83
1.82
1.80
1.78
1.77
1.77
1.75
1.68
1.65
1.63
1.61
1.60
1.59
1.59
1.44
1.42
1.40
1.40
1.39
1.38
1.36
1.35
1.34
1.33
1.33
1.31
1.30
1.28
1.28
1.27
1.27
1.25
1.24
1.23
0.94
0.93
0.91
O
OBu
OBu
1.00
2.16
2.16
1.17
4.18
4.21
5.31
5.30
6.10
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
f1 (ppm)


208.84
102.55
77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
67.45
58.51
45.37
31.74
28.53
25.84
25.63
19.28
18.45
13.84
O
O^{n}Bu
O^{n}Bu
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)