Supporting Information

Copper-catalyzed borylation of cycloalkylsilyl peroxides via radical C-C bond cleavage

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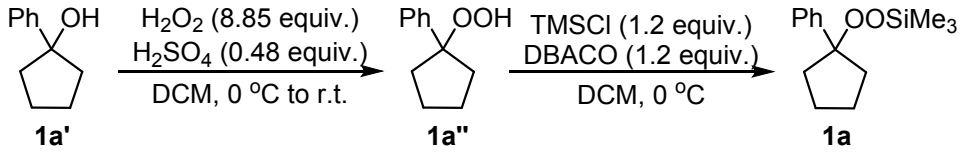
General Information

All reactions were conducted in oven-dried reaction tube under an atmosphere of nitrogen. Unless otherwise stated, all reagents were purchased from commercial sources and used without further purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance III-400 in solvents as indicated. Chemical shift are reported in ppm from TMS with the solvent resonance as internal standard ($CDCl_3$: $^{1}HNMR$: $\delta = 7.26$; ^{13}C NMR: $\delta = 77.0$). IR spectra were recorded on a Bruker Tensor 27 spectrometer and only major peaks are reported in cm^{-1}. HRMS were obtained on a Q-TOF micro spectrometer. Column chromatography was carried out on silica gel.

General Procedure for the Synthesis of Starting Peroxides

All of cycloalkylsilyl peroxides were prepared from the corresponding cycloalkyl alcohols according to the literature.[1] All of the NMR spectra of know compounds were in full accordance with the data in the literatures. The spectral data and the NMR spectra of all unknown starting materials were given.

1. General Procedure for the Synthesis of 1a–1l, 1p, 4a–4c: a Typical Procedure for the Synthesis of Alkylsilyl Peroxide 1a



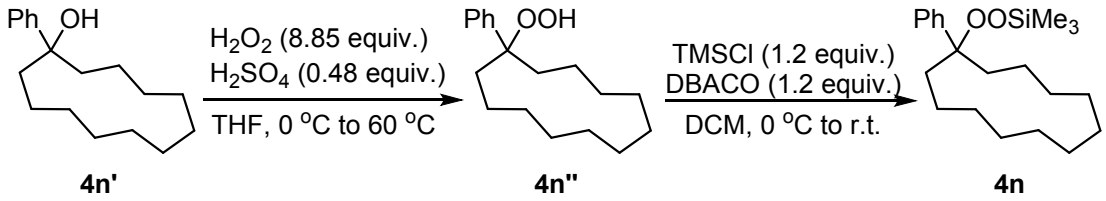
1a' **1a''** **1a**

To a solution of H_2O_2 (10 mL, 88.5 mmol, 30 wt% in H_2O) and conc. H_2SO_4 (0.25 mL, 4.8 mmol) was added a solution of 1-phenylcyclopentan-1-ol **1a'** (1.62 g, 10 mmol) in dichloromethane (2.0 mL) at 0 °C. The reaction mixture was stirred vigorously for 18 h at room temperature. After the reaction completed, the mixture was quenched with water and extracted with dichloromethane (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 15:1) affords corresponding alkyl hydroperoxide **1a''** as colorless oil.

An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with 1,4-diazabicyclo[2.2.2]octane (0.8 g, 7.2 mmol, 1.2 equiv). Then, the tube was evacuated and backfilled with nitrogen (three times). A solution of alkyl hydroperoxide **1a''** (1.07 g, 6 mmol) in 3 mL of dichloromethane at 0 °C was injected into the tube. Chlorotrimethylsilane (0.9 mL, 7.2 mmol, 1.2 equiv.) was added slowly under nitrogen atmosphere at 0 °C. The reaction mixture was stirred at room temperature for 5 h. The reaction was quenched with H_2O and the organic materials were extracted with dichloromethane (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum

ether/ethyl acetate 20:1) affords corresponding alkylsilyl peroxide **1a** as colorless oil.

2. General Procedure for the Synthesis of 1m–1o, 4d–4n, 6a-6d: a Typical Procedure for the Synthesis of Alkylsilyl Peroxide 4n



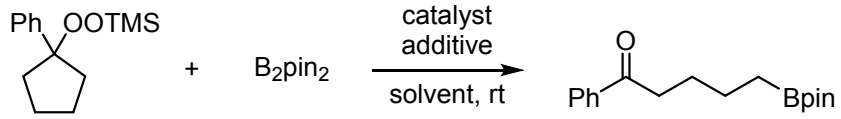
4n' **4n''** **4n**

To a solution of H_2O_2 (10 mL, 88.5 mmol, 30 wt% in H_2O) and conc. H_2SO_4 (0.25 mL, 4.8 mmol) was added a solution of 1-phenylcyclododecan-1-ol **4n'** (2.60 g, 10 mmol) in tetrahydrofuran (2.0 mL) at 0 °C. The reaction mixture was stirred vigorously for 12 h at 60 °C. After the reaction completed, the mixture was quenched with water and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 15:1) affords corresponding alkyl hydroperoxide **4n''** as colorless oil.

An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with 1,4-diazabicyclo[2.2.2]octane (1.08 g, 9.6 mmol, 1.2 equiv). Then, the tube was evacuated and backfilled with nitrogen (three times). A solution of alkyl hydroperoxide **4n''** (2.21 g, 8 mmol) in 4 mL of dichloromethane at 0 °C was injected into the tube. Chlorotrimethylsilane (1.2 mL, 9.6 mmol, 1.2 equiv.) was added slowly under nitrogen atmosphere at 0 °C. The reaction mixture was stirred at room temperature for 5 h. The reaction was quenched with H_2O and the organic materials were extracted with dichloromethane (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 20:1) affords corresponding alkylsilyl peroxide **4n** as colorless oil.

Optimization of Reaction Conditions

1. General Procedure for the Borylation of Cycloalkylsilyl Peroxide 1a with 2a

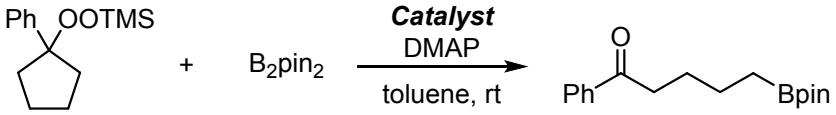


1a **2a** **3a**

An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with a certain amount of catalyst, B_2pin_2 and additive. Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkylsilyl peroxide **1a** (0.2 mmol, 1.0 equiv.) in solvent (2.0 mL) was injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 6 h. After the reaction completed, the mixture was quenched with water and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 10:1) affords the corresponding product **3a** as colorless oil.

Table S1 Optimization of the reaction conditions.

Catalysts[a]

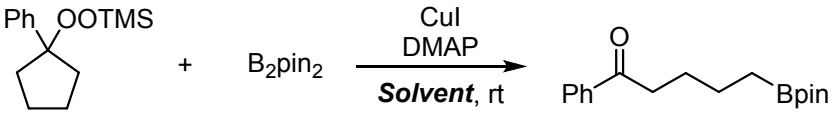


1a **2a** **3a**

Entry	Catalyst (mol%)	Additives (equiv.)	Solvents	Yield[b] (%)
1	**CuI (5)**	**DMAP (1.0)**	**toluene**	**61**
2	CuBr (5)	DMAP (1.0)	toluene	49
3	CuSCN (5)	DMAP (1.0)	toluene	36
4	$CuCl_2$ (5)	DMAP (1.0)	toluene	33
5	Cu (5)	DMAP (1.0)	toluene	trace
6	$CoCl_2$ (5)	DMAP (1.0)	toluene	0
7	$Pd(OAc)_2$ (5)	DMAP (1.0)	toluene	0
8	$NiBr_2$ (5)	DMAP (1.0)	toluene	0
9	$FeCl_2$ (5)	DMAP (1.0)	toluene	trace
10	$Fe(acac)_3$ (5)	DMAP (1.0)	toluene	trace

[a] Reaction conditions: 5 mol% catalyst, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMAP (0.2 mmol, 1.0 equiv.), toluene (2.0 mL), at room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

Solvents[a]

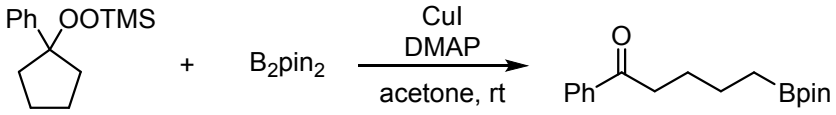


1a **2a** **3a**

Entry	Catalyst (mol%)	Additives (equiv.)	Solvents	Yield[b] (%)
1	CuI (5)	DMAP (1.0)	toluene	61
2	CuI (5)	DMAP (1.0)	THF	63
3	CuI (5)	DMAP (1.0)	DMF	41
4	CuI (5)	DMAP (1.0)	cyclohexane	9
5	CuI (5)	DMAP (1.0)	MTBE	48
6	CuI (5)	DMAP (1.0)	CH_2Cl_2	8
7	CuI (5)	DMAP (1.0)	CH_3CN	57
8	CuI (5)	DMAP (1.0)	$PhCF_3$	49
9	CuI (5)	DMAP (1.0)	HFIP	0
10	CuI (5)	DMAP (1.0)	DMSO	22
11	CuI (5)	DMAP (1.0)	CH_3NO_2	0
12	**CuI (5)**	**DMAP (1.0)**	**acetone**	**69**
13	CuI (5)	DMAP (1.0)	EtOAc	50

[a] Reaction conditions: 5 mol% catalyst, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMAP (0.2 mmol, 1.0 equiv.), solvent (2.0 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

The loading of the Catalyst[a]

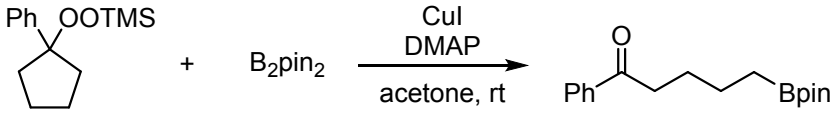


1a **2a** **3a**

Entry	Catalyst (mol%)	Additives (equiv.)	Solvents	Yield[b] (%)
1	CuI (2)	DMAP (1.0)	acetone	66
2	**CuI (5)**	**DMAP (1.0)**	**acetone**	**69**
3	CuI (10)	DMAP (1.0)	acetone	67

[a] Reaction conditions: 2-10 mol% catalyst, **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), DMAP (0.2 mmol, 1.0 equiv.), acetone (2.0 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

The ratio of 1a:2a[a]

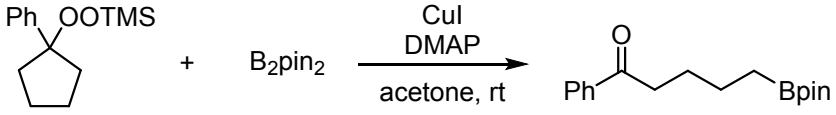


1a **2a** **3a**

Entry	**1a**:**2a**	Additives (equiv.)	Solvents	Yield[b] (%)
1	1:1	DMAP (1.0)	acetone	61
2	1:1.5	DMAP (1.0)	acetone	69
3	**1:2**	**DMAP (1.0)**	**acetone**	**74**
4	1:2.5	DMAP (1.0)	acetone	71

[a] Reaction conditions: 5 mol% of CuI, **1a** (0.2 mmol, 1 equiv.), **2a** (0.2-0.5 mmol, 1.0-2.5 equiv.), DMAP (0.2 mmol, 1.0 equiv.), acetone (2.0 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

The amount of the DMAP[a]

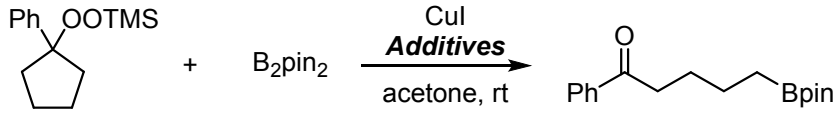


1a **2a** **3a**

Entry	Catalyst (mol%)	Additives (equiv.)	Solvents	Yield[b] (%)
1	CuI (5)	DMAP (1.0)	acetone	74
2	**CuI (5)**	**DMAP (1.5)**	**acetone**	**79**
3	CuI (5)	DMAP (2.0)	acetone	76

[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), 5 mol% of CuI, DMAP (0.2-0.4 mmol, 1.0-2.0 equiv.), acetone (2.0 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

Additives[a]



1a **2a** **3a**

Entry	Catalyst (mol%)	Additives (equiv.)	Solvents	Yield[b] (%)
1	**CuI (5)**	**DMAP (1.5)**	**acetone**	**79**
2	CuI (5)	4-cyanopyridine (1.5)	acetone	trace
3	CuI (5)	4-phenylpyridine (1.5)	acetone	trace
4	CuI (5)	4-pyrrolidinopyridine (1.5)	acetone	65

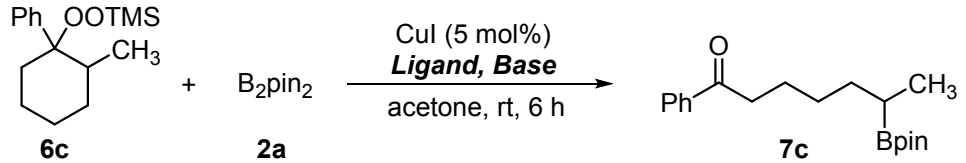
[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.4 mmol, 2.0 equiv.), 5 mol% of CuI, additive (0.3 mmol, 1.5 equiv.), acetone (2.0 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

2. General Procedure for the Borylation of Cycloalkylsilyl Peroxide 6c with 2b

An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with a certain amount of catalyst, ligand, B_2cat_2 and base. Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkylsilyl peroxide **6c** (0.2mmol, 1.0 equiv.) in solvent (2.0 mL) was injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred at room temperature for specified time. After that, pinacol (0.8 mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h. The mixture was then quenched with water and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 10:1) affords the corresponding product **7c** as colorless oil.

Table S2. Optimization of the reaction conditions

Ligands and Bases[a]

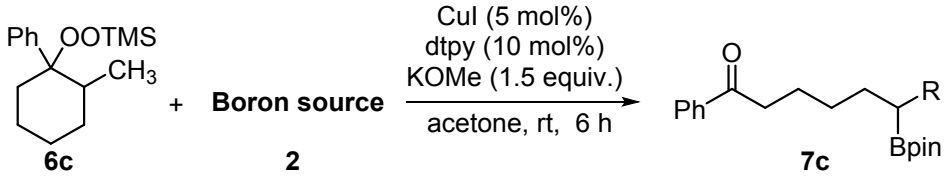


6c **2a** **7c**

Entry	Catalyst (mol%)	Ligands (mol%)	Bases (equiv.)	Solvents	Yield[b] (%)
1	CuI (5)	-	DMAP (1.5)	acetone	0
2	CuI (5)	-	KOMe (1.5)	acetone	0
3	CuI (5)	bipyridine (10)	KOMe (1.5)	acetone	trace
4	**CuI (5)**	**dtpy (10)**	**KOMe (1.5)**	**acetone**	**7**

[a] Reaction conditions: **6c** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), 5 mol% of CuI, ligand (0.02 mmol, 10 mol%), base (0.3 mmol, 1.5 equiv.), acetone (2 mL), room temperature, 6 h under nitrogen atmosphere. [b] Yield of isolated product.

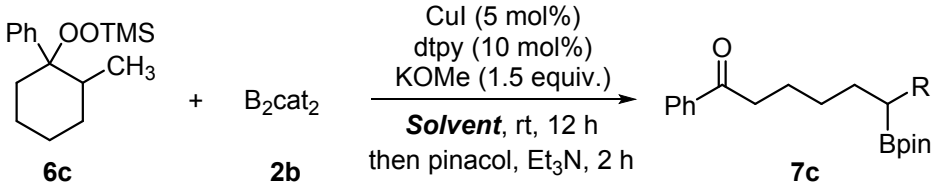
Boron source and time[a]



Entry	Boron source (equiv.)	Ligands (mol%)	Bases (equiv.)	Time (h)	Yield[b] (%)
4	B_2pin_2 (1.5)	dtpy (10)	KOMe (1.5)	6	7
2	$B_2(OH)_4$ (1.5)	dtpy (10)	KOMe (1.5)	6	trace[c]
3	B_2cat_2 (1.5)	dtpy (10)	KOMe (1.5)	6	26[c]
4	**B_2cat_2 (1.5)**	**dtpy (10)**	**KOMe (1.5)**	**12**	**32[c]**
5	B_2cat_2 (1.5)	dtpy (10)	KOMe (1.5)	24	32[c]

[a] Reaction conditions: **6c** (0.2 mmol, 1.0 equiv.), **2** (0.3 mmol, 1.5 equiv.), 5 mol% of CuI, acetone (2.0 mL), dtpy (10 mol%), KOMe (0.3 mmol, 1.5 equiv.), room temperature, 6 h under nitrogen atmosphere, unless otherwise noted. [b] Yield of isolated product. [c] The reaction mixture was stirred at room temperature for specified time. After that, pinacol (0.8 mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h.

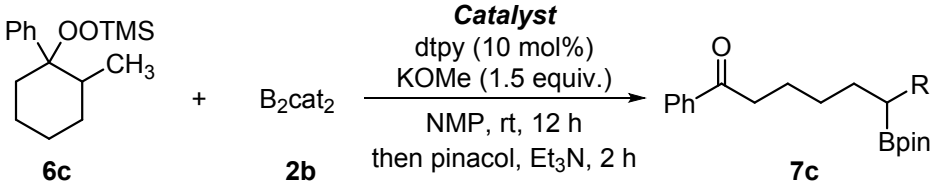
Solvents[a]



Entry	Catalyst (mol%)	Ligands (mol%)	Bases (equiv.)	Solvents	Yield[b] (%)
1	CuI (5)	dtpy (10)	KOMe (1.5)	acetone	32
2	CuI (5)	dtpy (10)	KOMe (1.5)	MTBE	trace
3	CuI (5)	dtpy (10)	KOMe (1.5)	THF	30
4	**CuI (5)**	**dtpy (10)**	**KOMe (1.5)**	**NMP**	**40**
5	CuI (5)	dtpy (10)	KOMe (1.5)	DMF	35

[a] Reaction conditions: **6c** (0.2 mmol, 1 equiv.), **2b** (0.3 mmol, 1.5 equiv.), 5 mol% CuI, dtpy (5.4 mg, 10 mol%), KOMe (1.5 equiv.), solvent (2.0 mL), room temperature, the reaction mixture was stirred at room temperature for 12 h. After that, pinacol (0.8 mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h. [b] Yield of isolated product.

Catalysts[a]

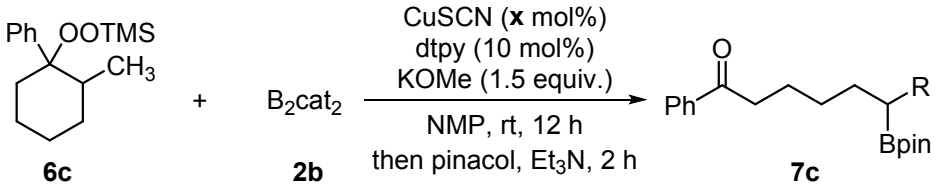


6c **2b** **7c**

Entry	Catalyst (mol%)	Ligands (mol%)	Bases (equiv.)	Solvents	Yield[b] (%)
1	CuCl (5)	dtpy (10)	KOMe (1.5)	NMP	37
2	CuBr (5)	dtpy (10)	KOMe (1.5)	NMP	42
3	CuI (5)	dtpy (10)	KOMe (1.5)	NMP	40
4	**CuSCN (5)**	**dtpy (10)**	**KOMe (1.5)**	**NMP**	**48**
5	$CuBr_2$ (5)	dtpy (10)	KOMe (1.5)	NMP	45

[a] Reaction conditions: 5 mol% catalyst, **6c** (0.2 mmol, 1.0 equiv.), **2b** (0.3 mmol, 1.5 equiv.), dtpy (10 mol%), KOMe (0.3 mmol, 1.5 equiv), NMP (2.0 mL), room temperature, the reaction mixture was stirred at room temperature for 12 h. After that, pinacol (0.8mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h. [b] Yield of isolated product.

The loading of Catalyst[a]

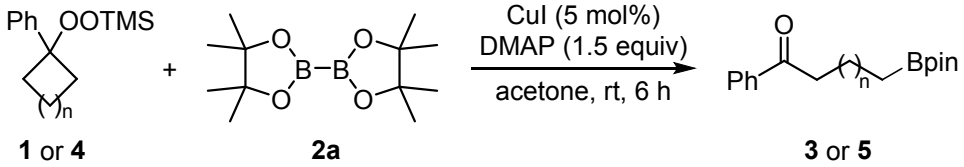


6c **2b** **7c**

Entry	Catalyst (mol%)	Ligands (mol%)	Bases (equiv.)	Solvents	Yield[b] (%)
1	CuSCN (5)	dtpy (10)	KOMe (1.5)	NMP	48
2	**CuSCN (10)**	**dtpy (10)**	**KOMe (1.5)**	**NMP**	**52**
3	CuSCN (15)	dtpy (10)	KOMe (1.5)	NMP	47

[a] Reaction conditions: 5-15 mol% CuSCN, **6c** (0.2 mmol, 1.0 equiv.), **2b** (0.3 mmol, 1.5 equiv.), dtpy (0.3 mmol, 1.5 equiv), KOMe (0.3 mmol, 1.5 equiv), NMP (2.0 mL), room temperature, the reaction mixture was stirred at room temperature for 12 h. After that, pinacol (0.8 mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h. [b] Yield of isolated product.

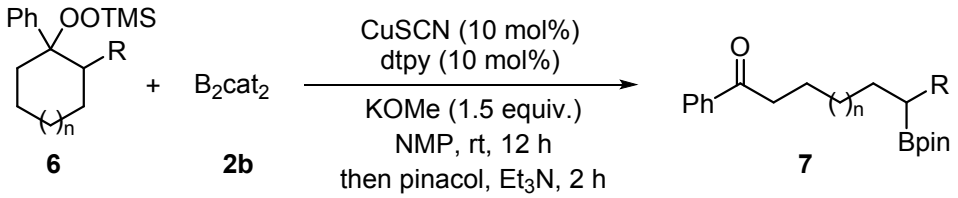
Representative Procedure for the Borylation of Cycloalkylsilyl Peroxides 1 or 4 with 2a



1 or **4** **2a** **3** or **5**

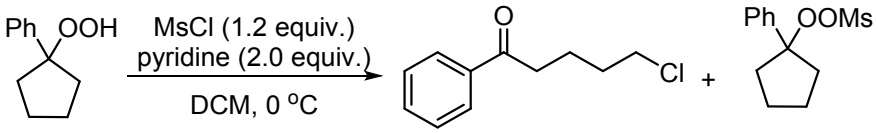
An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with CuI (1.9 mg, 5 mol%), B_2pin_2 (**2a**, 0.4mmol, 2.0 equiv.), DMAP (0.3 mmol, 1.5 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkylsilyl peroxides **1** or **4** (0.2 mmol, 1.0 equiv.) in acetone (2.0 mL) was injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 6 h. After the reaction completed, the mixture was quenched with water and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 10:1) affords the corresponding products **3** or **5** in yields list in Scheme 2 and Scheme 3.

Representative Procedure for the Borylation of 2-Substituted Cycloalkylsilyl Peroxides 6 with 2b



An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with CuSCN (2.4 mg, 10 mol%), dtby (5.4 mg, 10 mol%), B_2cat_2 (**2b**, 0.3 mmol, 1.5 equiv.), KOMe (0.3 mmol, 1.5 equiv.). Then, the tube was evacuated and backfilled with nitrogen (three times). Subsequently, a solution of cycloalkylsilyl peroxides **6** (0.2 mmol, 1.0 equiv.) in NMP (2.0 mL) was injected into the tube by syringe under nitrogen atmosphere. The reaction mixture was stirred at room temperature for 12 h. After that, pinacol (0.8 mmol, 4.0 equiv.) dissolved in Et_3N (0.7 mL) were added to the reaction mixture and stirred for 2 h. The mixture was then quenched with water and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 10:1) affords the corresponding products **7** in yields list in Scheme 3.

Investigation of the non-silicon-containing peroxide



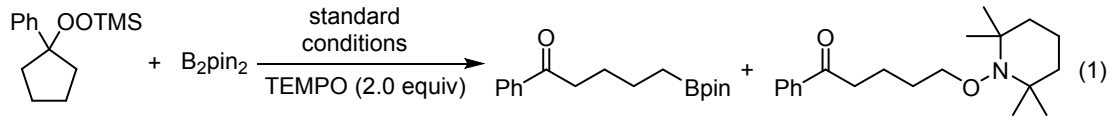
1a" **9a**, 35% **9b**, 0 %

A solution of alkyl hydroperoxide **1a"** (0.53g, 3 mmol) and pyridine (0.47g, 6 mmol, 2.0 equiv.) in 3 mL of dichloromethane at 0 °C was added methanesulfonyl chloride (0.41 g, 3.6 mmol, 1.2 equiv). The reaction was stirred for 1 h and quenched with 10% hydrochloric acid (10 mL). The mixture was extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by flash chromatography on silica gel (petroleum ether/ethyl acetate 10:1).

When alkyl hydroperoxide **1a"** conducted with methanesulfonyl chloride (MsCl), we failed to get the 1-phenylcyclopentyl methanesulfonoperoxoate **9b** but 5-chloro-1-phenylpentan-1-one **9a** obtained in 35% yield.

5-Chloro-1-phenylpentan-1-one (9a): (known compound)[3] Colorless oil (35%, 205.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.59-7.55 (m, 1H), 7.49-7.45 (m, 2H), 3.59 (t, *J* = 6.4 Hz, 2H), 3.02 (t, *J* = 8.0 Hz, 2H), 1.95-1.84 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.6, 136.8, 133.1, 128.6, 128.0, 44.7, 37.5, 32.0, 21.5 ppm.

Investigation of the Reaction Mechanism



1a **2a** **3a**, 0% **8a**, 75%

When 2.0 equiv of TEMPO was added to the reaction of **1a** with **2a** under the standard conditions, no desired product **3a** was observed and the corresponding TEMPO-adduct **8a** was obtained in 75% yield. This result indicates that a radical intermediate might be involved in this transformation.

1-Phenyl-5-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)pentan-1-one (8a): (known compound)[1b] Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98-7.96 (d, 2H, 7.2 Hz), 7.57-7.54 (m, 1H), 7.48-7.44 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 3.02 (t, *J* = 7.2 Hz, 2H), 1.88-1.80 (m, 2H), 1.66-1.59 (m, 2H), 1.44-1.29 (m, 6H), 1.15 (s, 6H), 1.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.3, 137.1, 132.9,128.5, 128.0, 59.6, 39.6, 38.6, 33.1, 28.4, 21.5, 20.1, 17.1 ppm.

1a **2a** **3a**, 26%

When 2.0 equiv of BHT was added to the reaction of **1a** with **2a** under the standard conditions, the reaction suppressed obviously and delivered the product **3a** in 26% yield. This result also supported a radical pathway for this transformation.

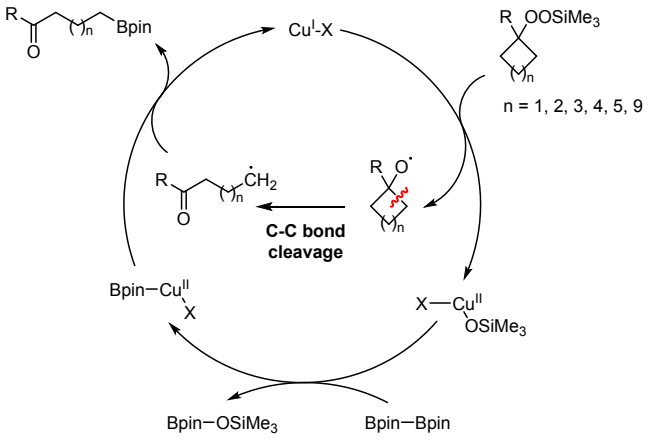
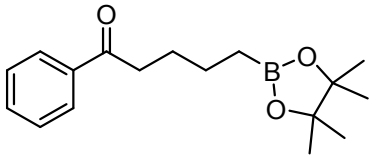
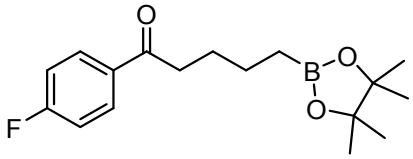


Figure 1. Proposed Mechanism

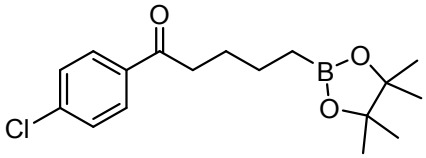
Characterization of Alkylboronic Esters 3



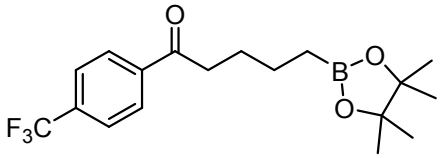
1-Phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3a): (known compound)[1c] Colorless oil (79%, 45.7 mg); R_f 0.32 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.55-7.52 (m, 1H), 7.46-7.42 (m, 2H), 2.96 (t, *J* = 7.6 Hz, 2H), 1.78-1.70 (m, 2H), 1.55-1.47 (m, 2H), 1.23 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 137.0, 132.8, 128.5, 128.0, 82.9, 38.5, 27.0, 24.8, 23.8 ppm.



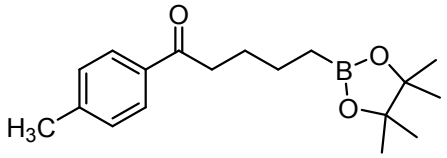
1-(4-Fluorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3b): (known compound)[1c] Colorless oil (87%, 53.1 mg); R_f 0.30 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00-7.95 (m, 2H), 7.14-7.08 (m, 2H), 2.93 (t, *J* = 8.0 Hz, 2H), 1.77-1.70 (m, 2H), 1.54-1.47 (m, 2H), 1.24 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =198.9, 165.6 (d, *J* = 252.8 Hz), 133.5 (d, *J* = 3.0 Hz), 130.6 (d, *J* = 9.3 Hz), 83.0, 38.4, 26.9, 24.8, 23.8 ppm.



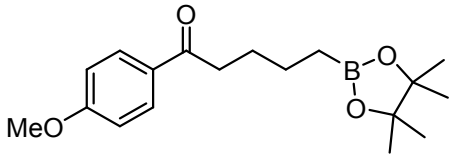
1-(4-Chlorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3c): Colorless oil (56%, 36.1 mg); R_f 0.28 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.90-7.86 (m, 2H), 7.43-7.40 (m, 2H), 2.92 (t, *J* = 7.6 Hz, 2H), 1.76-1.69 (m, 2H), 1.54-1.46 (m, 2H), 1.23 (s, 12H), 0.82 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =199.2, 139.2, 135.4, 129.5, 128.8, 83.0, 38.5, 26.8, 24.8, 23.8 ppm; IR (KBr): υ_{max} 2931, 1684, 1589, 1371, 1318, 1265, 1212, 1143, 1091, 968, 734, 703 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{25}BClO_3$ $[M+H]^+$ 323.1580, found 323.1575.



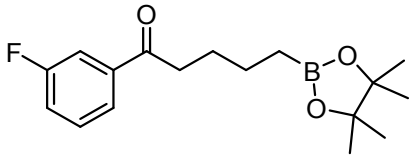
5-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(4-(trifluoromethyl)phenyl)pentan-1-one (3d): (known compound)[1c] Colorless oil (67%, 47.8 mg); R_f 0.22 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.79-1.72 (m, 2H), 1.56-1.48 (m, 2H), 1.24 (s, 12H), 0.84 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.4, 134.2 (d, *J* = 32.5 Hz), 128.4, 125.6 (q, *J* = 3.7 Hz), 123.6 (d, *J* = 271.1 Hz), 83.0, 38.8, 26.7, 24.8, 23.8 ppm.



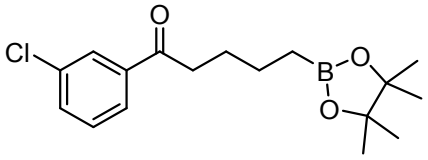
5-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(p-tolyl)pentan-1-one (3e): (known compound)[1c] Colorless oil (61%, 37.0 mg); R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.40 (s, 3H), 1.77-1.69 (m, 2H), 1.54-1.47 (m, 2H), 1.23 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.2, 143.5, 134.6, 129.2, 128.2, 82.9, 38.4, 27.1, 24.8, 23.9, 21.6 ppm.



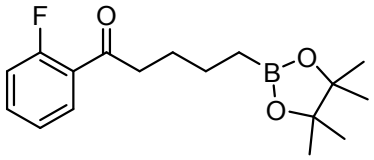
1-(4-Methoxyphenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3f): Colorless oil (66%, 42.0 mg); R_f 0.27 (EtOAc/petroleum ether = 1:8). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94-7.90 (m, 2H), 6.93-6.89 (m, 2H), 3.85 (s, 3H), 2.90 (t, *J* = 7.6 Hz, 2H), 1.76-1.68 (m, 2H), 1.54-1.46 (m, 2H), 1.23 (s, 12H), 0.82 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =199.1, 163.2, 130.3, 113.6, 82.9, 55.4, 38.2, 27.2, 24.8, 23.9 ppm; IR (KBr): υ_{max} 2930, 1675, 1600, 1370, 1256, 1143, 1027, 881, 803 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{28}BO_4$ $[M+H]^+$ 319.2075, found 319.2074.



1-(3-Fluorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3g): Colorless oil (71%, 43.6 mg); R_f 0.33 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72 (d, *J* = 7.6 Hz, 1H), 7.64-7.60 (m, 1H), 7.45-7.39 (m, 1H), 7.25-7.21 (m, 1H), 2.93 (t, *J* = 7.6 Hz, 2H), 1.77-1.70 (m, 2H), 1.54-1.47 (m, 2H), 1.23 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.1 (d, *J* = 1.9 Hz), 162.8 (d, *J* = 264.3 Hz), 139.2 (d, *J* = 1.6 Hz), 130.1 (d, *J* = 7.7 Hz), 123.8 (d, *J* = 2.8 Hz), 119.8 (d, *J* = 21.3 Hz), 114.8 (d, *J* = 21.9 Hz), 83.0, 38.6, 26.8, 24.8, 23.8 ppm; IR (KBr): υ_{max} 2928, 1688, 1588, 1442, 1318, 1267, 1143, 967, 790, 737, 684 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{25}BFO_3$ $[M+H]^+$ 307.1875, found 307.1872.

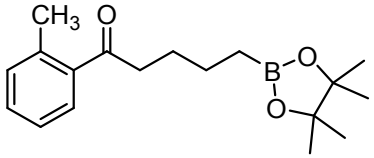


1-(3-Chlorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3h): Colorless oil (72%, 46.2 mg); R_f 0.28 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.91 (t, *J* = 1.6 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.52-7.49 (m, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 1.77-1.69 (m, 2H), 1.54-1.46 (m, 2H), 1.23 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.1, 138.6, 134.8, 132.7, 129.8, 128.2, 126.1, 83.0, 38.6, 26.7, 24.8, 23.8 ppm; IR (KBr): υ_{max} 2925, 1688, 1462, 1371, 1209, 1143, 967, 846, 680 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{24}BClNaO_3$ $[M+Na]^+$ 345.1399, found 345.1398.

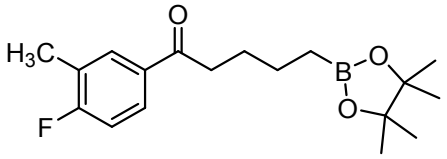


1-(2-Fluorophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3i): Colorless oil (68%, 41.5 mg); R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85-7.81 (m, 1H), 7.52-7.46 (m, 1H), 7.23-7.19 (m, 1H), 7.11 (ddd, *J* = 11.2, 8.4, 0.8 Hz, 1H), 2.98-2.94 (m, 2H), 1.75-1.68 (m, 2H), 1.53-1.45 (m, 2H), 1.23 (s, 12H), 0.82 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ

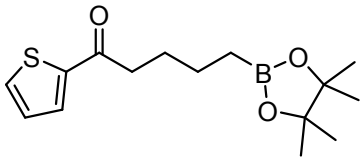
=198.9 (d, *J* = 4.0 Hz), 161.8 (d, *J* = 252.6 Hz), 134.2 (d, *J* = 9.0 Hz), 130.6 (d, *J* = 2.8 Hz), 125.9 (d, *J* = 13.1 Hz), 124.3 (d, *J* = 3.4 Hz), 116.6 (d, *J* = 23.9 Hz), 82.9, 43.5 (d, *J* = 6.8 Hz), 26.6, 24.8, 23.7 ppm; IR (KBr): υ_{max} 2931, 1685, 1609, 1452, 1371, 1268, 1107, 967, 762, 734, 703 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{24}BFNaO_3$ $[M+Na]^+$ 329.1695, found 329.1695.



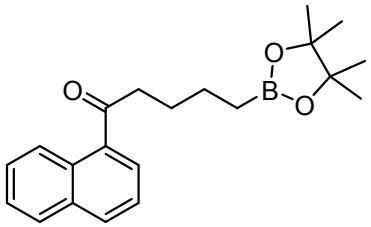
5-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(o-tolyl)pentan-1-one (3j): Colorless oil (46%, 28.1 mg); R_f 0.37 (EtOAc/petroleum ether = 1:10). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.61-7.59 (m, 1H), 7.36-7.32 (m, 1H), 7.24 (t, *J* = 7.2 Hz, 2H), 2.88 (t, *J* = 7.6 Hz, 2H), 2.48 (s, 3H), 1.74-1.66 (m, 2H), 1.53-1.45 (m, 2H), 1.23 (s, 12H), 0.82 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.9, 138.3, 137.8, 131.8, 130.9, 128.3, 125.6, 82.9, 41.6, 27.0, 24.8, 23.8, 21.2 ppm; IR (KBr): υ_{max} 2925, 1684, 1456, 1377, 1320, 1262, 1145, 1027, 967, 755 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{28}BO_3$ $[M+H]^+$ 303.2126, found 303.2115.



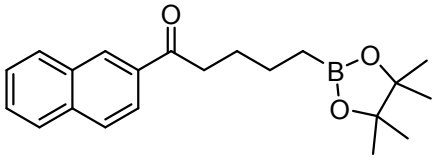
1-(4-Fluoro-3-methylphenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3k): Colorless oil (60%, 38.6 mg); R_f 0.30 (EtOAc/petroleum ether = 1:10). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.82-7.80 (m, 1H), 7.79-7.75 (m, 1H), 7.04 (t, *J* = 8.8 Hz, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.31 (d, *J* = 2.0 Hz, 3H), 1.76-1.68 (m, 2H), 1.54-1.46 (m, 2H), 1.23 (s, 12H), 0.82 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =199.2, 164.2 (d, *J* = 251.5 Hz), 133.2 (d, *J* = 3.5 Hz), 131.8 (d, *J* = 6.4 Hz), 127.9 (d, *J* = 9.0 Hz), 125.2 (d, *J* = 17.8 Hz), 115.1 (d, *J* = 23.0 Hz), 83.0, 38.4, 27.0, 24.8, 23.8, 14.5 (d, *J* = 3.5 Hz) ppm; IR (KBr): υ_{max} 2930, 1682, 1588, 1372, 1318, 1264, 1144, 967, 821, 734, 703 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{27}BFO_3$ $[M+H]^+$ 321.2032, found 321.2031.



5-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(thiophen-2-yl)pentan-1-one (3l): Colorless oil (70%, 41.4 mg); R_f 0.25 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.61 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.11 (dd, *J* = 4.8, 3.6 Hz, 1H), 2.89 (t, *J* = 7.6 Hz, 2H), 1.79-1.72 (m, 2H), 1.55-1.47 (m, 2H), 1.24 (s, 12H), 0.83 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.5, 144.5, 133.2, 131.6, 128.0, 83.0, 39.3, 27.4, 24.8, 23.8 ppm; IR (KBr): υ_{max} 2930, 1659, 1415, 1372, 1318, 1265, 1143, 967, 846, 732 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{24}BO_3S$ $[M+H]^+$ 295.1534, found 295.1530.

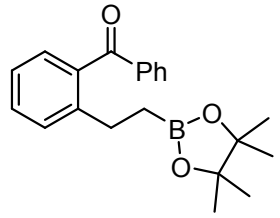


1-(Naphthalen-1-yl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3m): Colorless oil (62%, 42.3 mg); R_f 0.34 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.88-7.82 (m, 2H), 7.59-7.55 (m, 1H), 7.54-7.46 (m, 2H), 3.05 (t, *J* = 7.6 Hz, 2H), 1.83-1.76 (m, 2H), 1.58-1.50 (m, 2H), 1.23 (s, 12H), 0.84 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 205.0, 136.4, 133.9, 132.2, 130.1, 128.3, 127.7, 127.1, 126.3, 125.8, 124.3, 82.9, 42.2, 27.3, 24.8, 23.8 ppm; IR (KBr): υ_{max} 2924, 1681, 1508, 1461, 1372, 1318, 1214, 1143, 1093, 967, 846, 800, 776, 736 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{28}BO_3$ $[M+H]^+$ 339.2126, found 339.2119.

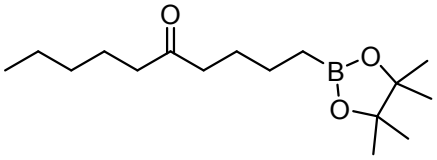


1-(Naphthalen-2-yl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentan-1-one (3n): Colorless oil (65%, 43.9 mg); R_f 0.33 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.46 (s, 1H), 8.04-8.01 (m, 1H), 7.97-7.95 (d, *J* = 8.0 Hz, 2H),

7.89-7.86 (m, 2H), 7.61-7.52 (m, 2H), 3.10 (t, *J* = 7.6 Hz, 2H), 1.85-1.77 (m, 2H), 1.60-1.52 (m, 2H), 1.24 (s, 12H), 0.87 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.4, 135.5, 134.4, 132.5, 129.6, 129.5, 128.3, 128.2, 127.7, 126.6, 124.0, 83.0, 38.6, 27.1, 24.8, 23.9 ppm; IR (KBr): υ_{max} 2929, 1679, 1467, 1371, 1317, 1266, 1213, 1143, 966, 847, 734, 702 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{28}BO_3$ $[M+H]^+$ 339.2126, found 339.2125.

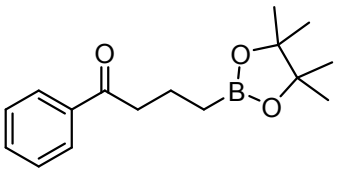


Phenyl(2-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)phenyl)methanone (3o): Colorless oil (35%, 23.5 mg); R_f 0.36 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.81-7.79 (m, 2H), 7.59-7.55 (m, 1H), 7.46-7.42 (m, 2H), 7.40-7.37 (m, 2H), 7.24-7.19 (m, 2H), 2.79 (t, *J* = 8.0 Hz, 2H), 1.18 (s, 12H), 1.06 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.7, 143.6, 138.3, 138.0, 133.0, 130.2, 130.1, 129.5, 128.4, 128.3, 125.0, 83.0, 27.3, 24.8 ppm; IR (KBr): υ_{max} 2926, 1663, 1372, 1264, 1143, 733, 702 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{26}BO_3$ $[M+H]^+$ 337.1970, found 337.1968.

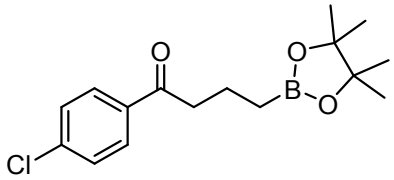


1-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)decan-5-one (3p): Colorless oil (54%, 30.0 mg); R_f 0.45 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.39-2.34 (m, 4H), 1.60-1.51 (m, 4H), 1.43-1.35 (m, 2H), 1.33-1.19 (m, 16H), 0.87 (t, *J* = 7.2 Hz, 3H), 0.77 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.6, 82.9, 42.7, 42.7, 31.4, 26.5, 24.8, 23.7, 23.5, 22.4, 13.9 ppm; IR (KBr): υ_{max} 2932, 1713, 1373, 1320, 1145, 968, 911, 847, 733, 648 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{31}BNaO_3$ $[M+Na]^+$ 305.2258, found 305.2257.

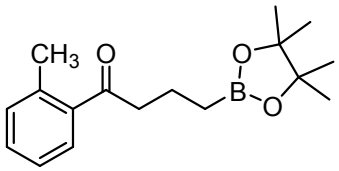
Characterization of Alkylboronic Esters 5



1-Phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butan-1-one (5a): (known compound)[2a] Colorless oil (72%, 39.6 mg); R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.98-7.96 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.43 (m, 2H), 2.98 (t, *J* = 7.6 Hz, 2H), 1.89-1.82 (m, 2H), 1.25 (s, 12H), 0.88 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 137.1, 132.8, 128.5, 128.1, 83.0, 40.9, 24.8, 19.2 ppm.

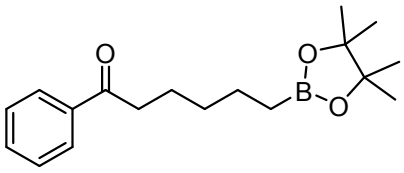


1-(4-Chlorophenyl)-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butan-1-one (5b): Colorless oil (65%, 40.2 mg); R_f 0.31 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.93-7.88 (m, 2H), 7.44-7.40 (m, 2H), 2.94 (t, *J* = 7.2 Hz, 2H), 1.88-1.80 (m, 2H), 1.25 (s, 12H), 0.88 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.3, 139.2, 135.3, 129.6, 128.8, 83.1, 40.9, 24.8, 19.2 ppm; IR (KBr): υ_{max} 2925, 1685, 1588, 1461, 1372, 1318, 1262, 1215, 1143, 1091, 1012, 968, 846, 798, 735 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{23}BClO_3$ $[M+H]^+$ 309.1423, found 309.1410.

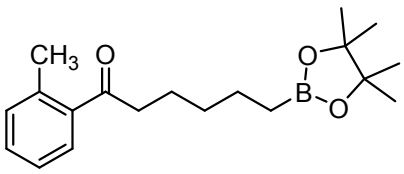


4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(o-tolyl)butan-1-one (5c): Colorless oil (78%, 45.1 mg); R_f 0.36 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.62 (d, *J* = 8.0 Hz, 1H), 7.36-7.32 (m, 1H), 7.23 (t, *J* = 7.6 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.47 (s, 3H), 1.85-1.78 (m, 2H), 1.23 (s, 12H), 0.86 (t, *J*

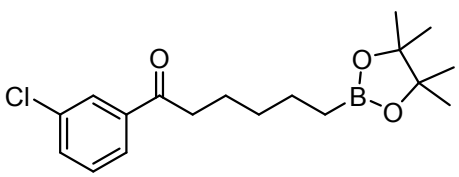
= 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.8, 138.3, 137.8, 131.8, 130.9, 128.3, 125.5, 83.0, 43.9, 24.8, 21.2, 19.1 ppm; IR (KBr): υ_{max} 2977, 1683, 1455, 1371, 1319, 1267, 1215, 1143, 968, 846, 733, 702 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{26}BO_3$ $[M+H]^+$ 289.1970, found 289.1963.



1-Phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one (5d): (known compound)[1c] Colorless oil (68%, 41.4 mg); R_f 0.33 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.93 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.43 (m, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 1.77-1.69 (m, 2H), 1.50-1.34 (m, 4H), 1.23 (s, 12H), 0.79 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 137.1, 132.8, 128.5, 128.0, 82.9, 38.5, 32.0, 24.8, 24.1, 23.8 ppm.

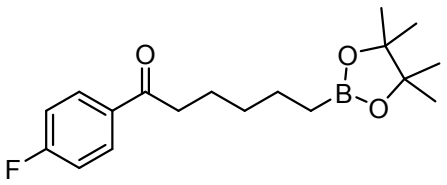


6-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(o-tolyl)hexan-1-one (5e): Colorless oil (43%, 27.2 mg); R_f 0.38 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.59 (d, *J* = 7.6 Hz, 1H), 7.37-7.33 (m, 1H), 7.23 (d, *J* = 7.2 Hz, 2H), 2.87 (t, *J* = 7.6 Hz, 2H), 2.47 (s, 3H), 1.73-1.65 (m, 2H), 1.48-1.41 (m, 2H), 1.39-1.32 (m, 2H), 1.23 (s, 12H), 0.78 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 205.0, 138.4, 137.7, 131.8, 130.9, 128.2, 125.6, 82.9, 41.6, 32.0, 24.8, 24.2, 23.8, 21.1 ppm; IR (KBr): υ_{max} 2925, 1685, 1456, 1373, 1318, 1263, 1144, 1018, 967, 802, 735, 703 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{29}BNaO_3$ $[M+Na]^+$ 339.2102, found 339.2097.



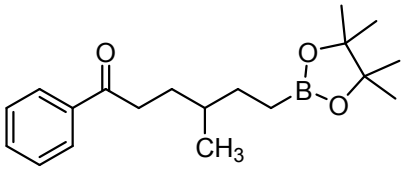
1-(3-Chlorophenyl)-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one

(5f): Colorless oil (56%, 37.7 mg); R_f 0.28 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.91 (t, *J* = 1.6 Hz, 1H), 7.83-7.80 (m, 1H), 7.52-7.50 (m, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 1.76-1.68 (m, 2H), 1.50-1.42 (m, 2H), 1.41-1.33 (m, 2H), 1.23 (s, 12H), 0.78 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.2, 138.6, 134.8, 132.7, 129.8, 128.2, 126.1, 82.9, 38.6, 31.9, 24.8, 23.9, 23.7 ppm; IR (KBr): υ_{max} 2927, 1689, 1571, 1415, 1370, 1317, 1249, 1204, 1143, 968, 846, 786, 712, 680 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{26}BClNaO_3$ $[M+Na]^+$ 359.1556, found 359.1548.



1-(4-Fluorophenyl)-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one

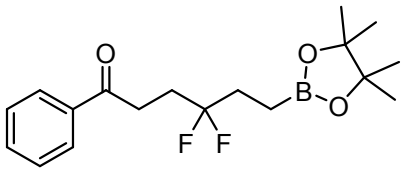
(5g): Colorless oil (65%, 41.6 mg); R_f 0.34 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00-7.96 (m, 2H), 7.14-7.09 (m, 2H), 2.92 (t, *J* = 8.0 Hz, 2H), 1.76-1.68 (m, 2H), 1.49-1.42 (m, 2H), 1.41-1.34 (m, 2H), 1.23 (s, 12H), 0.78 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =198.9, 165.6 (d, *J* = 252.4 Hz), 133.5 (d, *J* = 3.0 Hz), 130.6 (d, *J* = 9.4 Hz), 115.6 (d, *J* = 21.5 Hz), 82.9, 38.5, 32.0, 24.8, 24.1, 23.8 ppm; IR (KBr): υ_{max} 2931, 1684, 1598, 1506, 1371, 1318, 1265, 1232, 1143, 968, 845, 733, 703 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{26}BFNaO_3$ $[M+Na]^+$ 343.1851, found 343.1852.



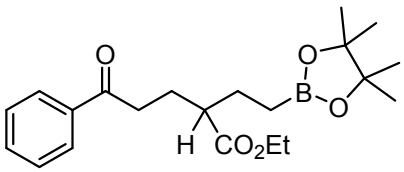
4-Methyl-1-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one

(5h): Colorless oil (50%, 31.8 mg); R_f 0.37 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.52 (m, 1H), 7.47-7.43 (m, 2H), 3.03-2.90 (m, 2H), 1.78-1.71 (m, 1H), 1.58-1.39 (m, 3H), 1.32-1.26 (m, 1H), 1.23 (s, 12H), 0.92 (d, *J* = 6.4 Hz, 3H), 0.86-0.70 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.8, 137.1, 132.8, 128.5, 128.0, 82.9, 36.4, 34.8, 31.0, 30.6, 24.8, 24.7, 19.1 ppm;

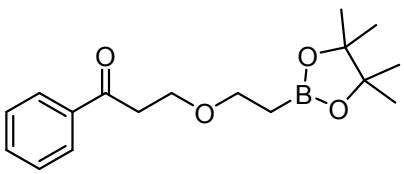
IR (KBr): υ_{max} 2925, 1686, 1598, 1449, 1371, 1319, 1271, 1210, 1145, 968, 847, 740, 691 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{29}BNaO_3$ $[M+Na]^+$ 339.2102, found 339.2098.



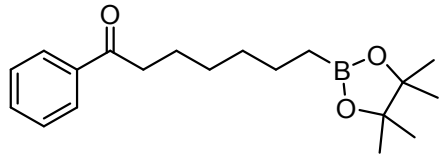
4,4-Difluoro-1-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one (5i): Colorless oil (52%, 35.2 mg); R_f 0.30 (EtOAc/petroleum ether = 1:10). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.99-7.97 (m, 2H), 7.59-7.55 (m, 1H), 7.49-7.45 (m, 2H), 3.24-3.20 (m, 2H), 2.36-2.23 (m, 2H), 2.07-1.95 (m, 2H), 1.25 (s, 12H), 0.96 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.4, 136.6, 133.2, 128.6, 128.0, 127.4, 125.0 (t, *J* = 239.7 Hz), 83.3, 31.5 (t, *J* = 25.9 Hz), 31.3 (t, *J* = 3.6 Hz), 30.4 (t, *J* = 25.2 Hz), 24.8 ppm; ^{19}F NMR (376 MHz, $CDCl_3$) δ -101.5 (s); IR (KBr): υ_{max} 2925, 1689, 1598, 1379, 1324, 1261, 1212, 1143, 1027, 968, 847, 798, 741 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{25}BF_2NaO_3$ $[M+Na]^+$ 361.1757, found 361.1754.



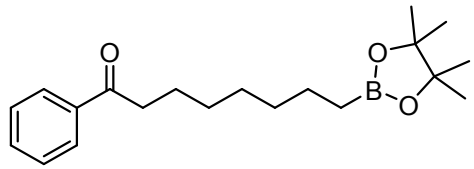
Ethyl 5-oxo-5-phenyl-2-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)-pentanoate (5j): Colorless oil (57%, 42.9 mg); R_f 0.31 (EtOAc/petroleum ether = 1:10). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.56-7.52 (m, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 4.19-4.07 (m, 2H), 3.04-2.90 (m, 2H), 2.46-2.39 (m, 1H), 2.04-1.91 (m, 2H), 1.82-1.72 (m, 1H), 1.70-1.61 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.23 (s, 12H), 0.81-0.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 199.5, 175.8, 136.8, 132.9, 128.5, 128.0, 83.1, 60.2, 46.9, 36.2, 26.8, 26.3, 24.8, 24.7, 14.3 ppm; IR (KBr): υ_{max} 2978, 1726, 1685, 1448, 1371, 1320, 1143, 967, 735, 690 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{31}BNaO_5$ $[M+Na]^+$ 397.2157, found 397.2152.



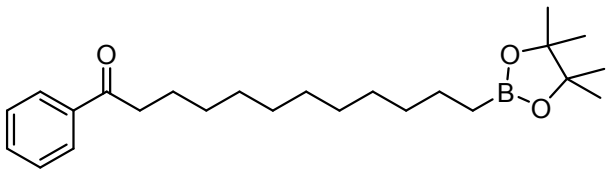
1-Phenyl-3-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethoxy)propan-1-one (5k): (known compound)[1c] Colorless oil (33%, 20.1 mg); R_f 0.25 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.57-7.54 (m, 1H), 7.47-7.44 (m, 2H), 3.85 (t, *J* = 6.8 Hz, 2H), 3.61 (t, *J* = 8.0 Hz, 2H), 3.25 (t, *J* = 6.8 Hz, 2H), 1.23 (s, 12H), 1.16 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.5, 137.0, 133.0, 128.5, 128.1, 83.1, 67.7, 65.7, 38.9, 24.8 ppm.



1-Phenyl-7-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptan-1-one (5l): (known compound)[1c] Colorless oil (51%, 32.3 mg); R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.52 (m, 1H), 7.47-7.43 (m, 2H), 2.95 (t, *J* = 7.2 Hz, 2H), 1.76-1.69 (m, 2H), 1.46-1.30 (m, 6H), 1.23 (s, 12H), 0.77 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 137.1, 132.8, 128.5, 128.0, 82.8, 38.6, 32.1, 29.1, 24.8, 24.3, 23.8 ppm.

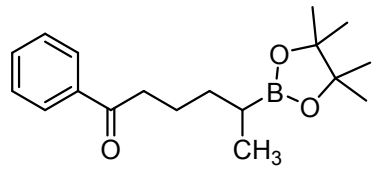


1-Phenyl-8-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)octan-1-one (5m): Colorless oil (53%, 35.1 mg); R_f 0.32 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.57-7.52 (m, 1H), 7.47-7.43 (m, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 1.76-1.68 (m, 2H), 1.44-1.28 (m, 8H), 1.23 (s, 12H), 0.76 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 137.1, 132.8, 128.5, 128.0, 82.8, 38.6, 32.2, 29.3, 29.2, 24.8, 24.4, 23.9 ppm; IR (KBr): υ_{max} 2924, 2854, 1685, 1598, 1448, 1371, 1318, 1144, 967, 846, 752, 690 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{31}BNaO_3$ $[M+Na]^+$ 353.2258, found 353.2259.

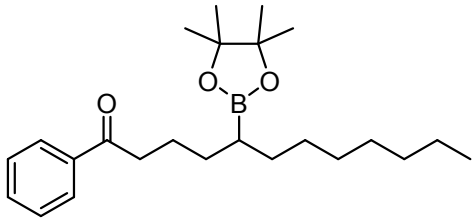


1-Phenyl-12-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)dodecan-1-one (5n): Colorless oil (61%, 47.1 mg); R_f 0.33 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.95 (m, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.48-7.44 (m, 2H), 2.96 (t, *J* = 7.6 Hz, 2H), 1.76-1.69 (m, 2H), 1.39-1.25 (m, 16H), 1.24 (s, 12H), 0.76 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 137.1, 132.8, 128.5, 128.0, 82.8, 38.6, 32.4, 29.6, 29.5, 29.5, 29.4, 29.3, 24.8, 24.4, 24.0 ppm; IR (KBr): υ_{max} 2923, 2852, 1687, 1598, 1448, 1371, 1317, 1144, 967, 846, 737, 690 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{39}BNaO_3$ $[M+Na]^+$ 409.2884, found 409.2884.

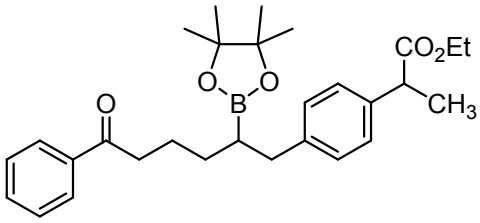
Characterization of Alkylboronic Esters 7



1-Phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one (7a): (known compound)[2b] Colorless oil (56%, 33.9 mg); R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.93 (m, 2H), 7.55-7.51 (m, 1H), 7.46-7.42 (m, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 1.83-1.67 (m, 2H), 1.59-1.50 (m, 1H), 1.42-1.33 (m, 1H), 1.22 (s, 12H), 1.09-0.97 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 137.0, 132.8, 128.5, 128.0, 82.8, 38.9, 32.9, 24.7, 24.6, 23.7, 15.4 ppm.

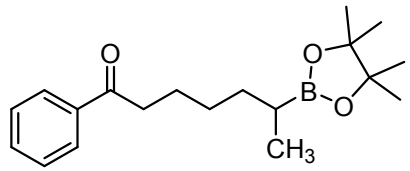


1-Phenyl-5-(4,4,5-trimethyl-1,3,2-dioxaborolan-2-yl)dodecan-1-one (7b): Colorless oil (43%, 33.2 mg); R_f 0.32 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.94 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.42 (m, 2H), 3.01-2.88 (m, 2H), 1.77-1.69 (m, 2H), 1.54-1.34 (m, 4H), 1.25 (br s, 10H), 1.23 (s, 12H), 1.05-0.97 (m, 1H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 137.0, 132.8, 128.5, 128.1, 82.9, 39.0, 31.8, 31.2, 31.1, 29.9, 29.2, 29.1, 24.8, 24.7, 24.1, 22.6, 14.1 ppm; IR (KBr): υ_{max} 2929, 1685, 1598, 1461, 1370, 1315, 1268, 1213, 1143, 967, 857, 735, 690 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{40}BO_3$ $[M+H]^+$ 387.3065, found 387.3066.



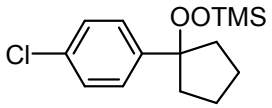
Ethyl 2-(4-(6-oxo-6-phenyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexyl) phenyl)propanoate (7c): Colorless oil (53%, 50.6 mg); R_f 0.30 (EtOAc/petroleum

ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95-7.93 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.43 (m, 2H), 7.18-7.13 (m, 4H), 4.16-4.02 (m, 2H), 3.65 (q, *J* = 7.2 Hz, 1H), 3.02-2.87 (m, 2H), 2.73-2.64 (m, 2H), 1.86-1.68 (m, 2H), 1.56-1.48 (m, 2H), 1.45 (dd, *J* = 7.2, 1.2 Hz, 3H), 1.43-1.36 (m, 1H), 1.19 (t, *J* = 7.2 Hz, 3H), 1.14 (s, 6H), 1.10 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.5, 174.7, 140.9, 138.0, 137.0, 132.8, 129.1, 128.5, 128.1, 127.1, 83.1, 60.6, 45.2, 38.9, 36.8, 30.9, 24.8, 24.6, 23.9, 18.6, 14.1 ppm; IR (KBr): υ_{max} 2928, 1732, 1684, 1449, 1372, 1321, 1260, 1211, 1143, 1090, 1023, 908, 799, 731, 690, 649 cm^{-1}; HRMS (ESI) calcd for $C_{29}H_{39}BNaO_5$ $[M+Na]^+$ 501.2783, found 501.2789.

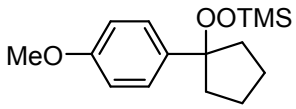


1-Phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptan-1-one (7d): Colorless oil (52%, 32.7 mg); R_f 0.33 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.96-7.93 (m, 2H), 7.56-7.52 (m, 1H), 7.46-7.43 (m, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 1.76-1.69 (m, 2H), 1.54-1.45 (m, 1H), 1.42-1.32 (m, 3H), 1.22 (s, 12H), 1.06-0.95 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 200.6, 137.1, 132.8, 128.5, 128.0, 82.8, 38.6, 32.9, 28.6, 24.7, 24.6, 24.5, 15.4 ppm; IR (KBr): υ_{max} 2925, 1685, 1461, 1371, 1314, 1261, 1143, 1014, 799, 735, 690 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{29}BNaO_3$ $[M+Na]^+$ 339.2102, found 339.2100.

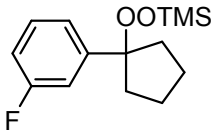
Characterization of New Alkylsilyl Peroxides 1



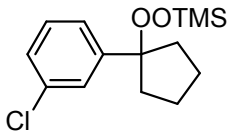
((1-(4-Chlorophenyl)cyclopentyl)peroxy)trimethylsilane (1c): Colorless oil; R_f 0.80 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.40-7.38 (m, 2H), 7.31-7.29 (m, 2H), 2.35-2.31 (m, 2H), 1.90-1.85 (m, 4H), 1.81-1.77 (m, 2H), 0.12 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =142.7, 132.6, 128.1, 127.8, 94.5, 36.6, 24.2, -1.3 ppm; IR (KBr): υ_{max} 2962, 1492, 1249, 1092, 892, 844, 825, 734 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{22}ClO_2Si$ $[M+H]^+$ 285.1072, found 285.1092.



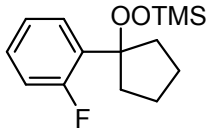
((1-(4-Methoxyphenyl)cyclopentyl)peroxy)trimethylsilane (1f): Colorless oil; R_f 0.35 (EtOAc/petroleum ether = 1:10). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 -7.37 (m, 2H), 6.87-6.85 (m, 2H), 3.81 (s, 3H), 2.36-2.30 (m, 2H), 1.93-1.82 (m, 4H), 1.79-1.74 (m, 2H), 0.08 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =158.5, 136.2, 128.0, 113.0, 94.6, 55.2, 36.3, 24.1, -1.3 ppm; IR (KBr): υ_{max} 2959, 1513, 1248, 906, 844, 732 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{24}NaO_3Si$ $[M+Na]^+$ 303.1387, found 303.1383.



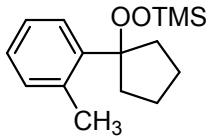
((1-(3-Fluorophenyl)cyclopentyl)peroxy)trimethylsilane (1g): Colorless oil; R_f 0.86 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32 -7.28 (m, 1H), 7.24-7.19 (m, 2H), 6.97-6.93 (m, 1H), 2.33 (br s, 2H), 1.91 (br s, 4H), 1.79 (br s, 2H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =162.7 (d, J = 242.7 Hz), 147.1 (d, J = 6.6 Hz), 129.1 (d, J = 8.1 Hz), 122.0 (d, J = 2.6 Hz), 113.8 (d, J = 22.0 Hz), 113.5 (d, J = 21.1 Hz), 94.7, 36.8, 24.3, -1.3 ppm; IR (KBr): υ_{max} 2961, 1615, 1590, 1438, 1249, 893, 840, 779, 741, 693 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{21}FKO_2Si$ $[M+K]^+$ 307.0926, found 307.0937.



((1-(3-Chlorophenyl)cyclopentyl)peroxy)trimethylsilane (1h): Colorless oil; R_f 0.82 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 (d, *J* = 1.6 Hz, 1H), 7.33-7.30 (m, 1H), 7.26-7.20 (m, 2H), 2.33-2.27 (m, 2H), 1.91-1.84 (m, 4H), 1.80-1.71 (m, 2H), 0.11 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =146.4, 133.7, 129.0, 127.0, 126.9, 124.7, 94.6, 36.7, 24.2, -1.3 ppm; IR (KBr): υ_{max} 2962, 1419, 1250, 905, 845, 782, 732, 694 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{21}ClNaO_2Si$ $[M+Na]^+$ 307.0892, found 307.0882.

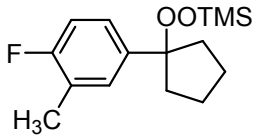


((1-(2-Fluorophenyl)cyclopentyl)peroxy)trimethylsilane (1i): Colorless oil; R_f 0.85 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57-7.53 (m, 1H), 7.25-7.20 (m, 1H), 7.11-7.07 (m, 1H), 6.99 (ddd, *J* = 12.0 Hz, 8.0 Hz, 1.2 Hz, 1H), 2.41-2.36 (m, 2H), 2.05-1.98 (m, 2H), 1.86-1.72 (m, 4H), 0.09 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =160.7 (d, *J* = 246.5 Hz), 131.0 (d, *J* = 11.7 Hz), 129.6 (d, *J* = 4.6 Hz), 128.6 (d, *J* = 8.5 Hz), 123.2 (d, *J* = 3.2 Hz), 115.8 (d, *J* = 23.2 Hz), 93.4 (d, *J* = 3.1 Hz), 36.1 (d, *J* = 3.4 Hz), 24.3, -1.4 ppm; IR (KBr): υ_{max} 2960, 1488, 1451, 1250, 1099, 906, 845, 755, 733, 648 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{21}FKO_2Si$ $[M+K]^+$ 307.0926, found 307.0952.

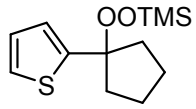


Trimethyl((1-(o-tolyl)cyclopentyl)peroxy)silane (1j): Colorless oil; R_f 0.90 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39 (d, *J* = 7.2 Hz, 1H), 7.16-7.12 (m, 3H), 2.50 (s, 3H), 2.45-2.39 (m, 2H), 2.04-1.97 (m, 2H), 1.87-1.77 (m, 2H), 1.73-1.62 (m, 2H), 0.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.4, 137.5, 131.8, 127.6, 127.1, 124.9, 95.7, 35.7, 24.1, 21.7, -1.2 ppm; IR (KBr): υ_{max} 2960, 1250, 1015, 905, 844, 726, 649 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{25}O_2Si$

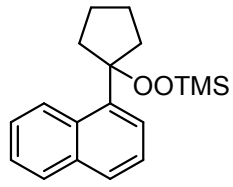
$[M+H]^+$ 265.1618, found 265.1633.



((1-(4-Fluoro-3-methylphenyl)cyclopentyl)peroxy)trimethylsilane (1k): Colorless oil; R_f 0.92 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.24-7.20 (m, 2H), 6.96-6.91 (m, 1H), 2.34-2.28 (m, 2H), 2.78 (s, 3H), 1.91-1.82 (m, 4H), 1.78-1.70 (m, 2H), 0.09 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =160.4 (d, *J* = 242.3 Hz), 139.5 (d, *J* = 3.5 Hz), 129.9 (d, *J* = 5.0 Hz), 125.6 (d, *J* = 7.8 Hz), 123.6 (d, *J* = 17.1 Hz), 114.1 (d, *J* = 22.0 Hz), 94.5, 36.5, 24.2, 14.7 (d, *J* = 3.3 Hz), -1.3 ppm; IR (KBr): υ_{max} 2960, 1503, 1250, 1186, 1119, 905, 844, 728, 649 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{24}FO_2Si$ $[M+H]^+$ 283.1524, found 283.1508.

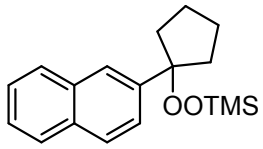


Trimethyl((1-(thiophen-2-yl)cyclopentyl)peroxy)silane (1l): Colorless oil; R_f 0.86 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.23-7.22 (m, 1H), 7.03-7.02 (m, 1H), 6.97-6.94 (m, 1H), 2.44-2.38 (m, 2H), 2.05-1.97 (m, 2H), 1.91-1.81 (m, 2H), 1.80-1.72 (m, 2H), 0.10 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =148.2, 126.0, 124.4, 124.3, 92.3, 37.4, 24.0, -1.4 ppm; IR (KBr): υ_{max} 2962, 1249, 906, 845, 734, 697 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_{21}O_2SSi$ $[M+H]^+$ 257.1026, found 257.1023.

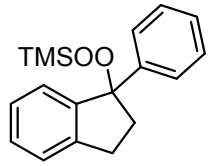


Trimethyl((1-(naphthalen-1-yl)cyclopentyl)peroxy)silane (1m): Colorless oil; R_f 0.87 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.55-8.53 (m, 1H), 7.86-7.82 (m, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 7.2 Hz, 1H), 7.48-7.43 (m, 2H), 7.40 (t, *J* = 7.6 Hz, 1H), 2.64-2.58 (m, 2H), 2.26-2.18 (m, 2H), 1.97-1.86 (m, 2H), 1.78-1.70 (m, 2H), -0.01 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =139.5, 134.5, 132.0, 128.7, 128.4, 127.1, 125.0, 124.8, 124.5, 95.8, 36.6, 24.3, -1.2 ppm; IR (KBr):

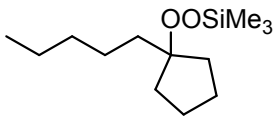
υ_{max} 2960, 1510, 1250, 904, 845, 798, 777, 728, 649 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{25}O_2Si$ $[M+H]^+$ 301.1618, found 301.1630.



Trimethyl((1-(naphthalen-2-yl)cyclopentyl)peroxy)silane (1n): Colorless oil; R_f 0.85 (EtOAc/petroleum ether = 1:20). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.90 (s, 1H), 7.86-7.80 (m, 3H), 7.63-7.60 (m, 1H), 7.49-7.44 (m, 2H), 2.45-2.40 (m, 2H), 1.98-1.88 (m, 2H), 1.86-1.78 (m, 2H), 0.11 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.7, 133.0, 132.5, 128.2, 127.4, 127.3, 125.7, 125.6, 125.4, 125.0, 95.1, 36.5, 24.3, -1.2 ppm; IR (KBr): υ_{max} 2961, 1249, 905, 844, 727, 649, 476 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{25}O_2Si$ $[M+H]^+$ 301.1618, found 301.1632.



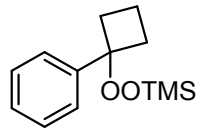
Trimethyl((1-phenyl-2,3-dihydro-1H-inden-1-yl)peroxy)silane (1o): Colorless oil; R_f 0.87 (EtOAc/petroleum ether = 1:20). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.48-7.46 (m, 2H), 7.39-7.33 (m, 4H), 7.31-7.28 (m, 1H), 7.26-7.19 (m, 2H), 3.21-3.13 (m, 1H), 2.99-2.92 (m, 1H), 2.68-2.62 (m, 1H), 2.40-2.32 (m, 1H), -0.03 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =145.9, 143.6, 143.5, 128.6, 127.8, 127.5, 126.9, 126.7, 125.8, 124.5, 96.8, 40.6, 30.4, -1.6 ppm; IR (KBr): υ_{max} 2960, 1446, 1249, 1021, 881, 842, 751, 736, 698, 606, 571 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{23}O_2Si$ $[M+H]^+$ 299.1462, found 299.1444.



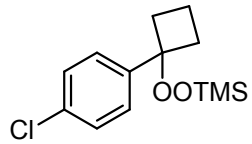
Trimethyl((1-pentylcyclopentyl)peroxy)silane (1p): Colorless oil; R_f 0.92 (EtOAc/petroleum ether = 1:20). ^1H NMR (400 MHz, $CDCl_3$): δ = 1.88-1.82 (m, 2H), 1.72-1.63 (m, 4H), 1.57-1.49 (m, 2H), 1.47-1.40 (m, 2H), 1.37-1.23 (m, 6H), 0.89 (t, *J* = 7.2 Hz, 3H), 0.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =94.5, 36.7, 34.8, 32.4, 24.8, 24.1, 22.7, 14.1, -1.1 ppm; IR (KBr): υ_{max} 2957, 1249, 893, 841, 742 cm^{-1};

HRMS (ESI) calcd for $C_{13}H_{28}NaO_2Si$ $[M+Na]^+$ 267.1751, found 267.1734.

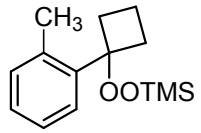
Characterization of New Alkylsilyl Peroxides 4



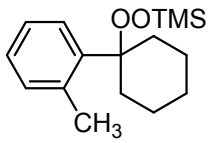
Trimethyl((1-phenylcyclobutyl)peroxy)silane (4a): Colorless oil; R_f 0.92 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44-7.42 (m, 2H), 7.38-7.34 (m, 2H), 7.31-7.26 (m, 1H), 2.66-2.58 (m, 2H), 2.48-2.41 (m, 2H), 2.10-1.99 (m, 1H), 1.77-1.66 (m, 1H), 0.07 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =142.6, 127.9, 127.4, 126.8, 87.7, 31.3, 13.8, -1.3 ppm; IR (KBr): υ_{max} 2956, 1447, 1304, 1250, 1023, 845, 758, 697 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{20}NaO_2Si$ $[M+Na]^+$ 259.1125, found 259.1117.



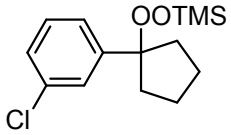
((1-(4-Chlorophenyl)cyclobutyl)peroxy)trimethylsilane (4b): Colorless oil; R_f 0.86 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36-7.30 (m, 4H), 2.61-2.53 (m, 2H), 2.43-2.36 (m, 2H), 2.10-1.99 (m, 1H), 1.77-1.67 (m, 1H), 0.08 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.3, 133.2, 128.2, 128.0, 87.1, 31.4, 13.9, -1.3 ppm; IR (KBr): υ_{max} 2956, 1492, 1250, 1092, 1014, 845, 750 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{19}ClNaO_2Si$ $[M+Na]^+$ 293.0735, found 293.0723.



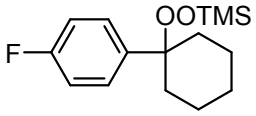
Trimethyl((1-(o-tolyl)cyclobutyl)peroxy)silane (4c): Colorless oil; R_f 0.95 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.28-7.24 (m, 1H), 7.21-7.11 (m, 3H), 2.68-2.52 (m, 4H), 2.34 (s, 3H), 2.23-2.12 (m, 1H), 1.81-1.72 (m, 1H), 0.00 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =139.9, 137.4, 131.1, 128.0, 127.7, 124.9, 89.6, 31.6, 20.0, 15.5, -1.4 ppm; IR (KBr): υ_{max} 2955, 1297, 1250, 906, 845, 731, 649 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{23}O_2Si$ $[M+H]^+$ 251.1462, found 251.1455.



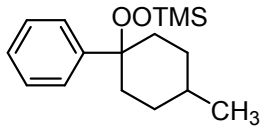
Trimethyl((1-(o-tolyl)cyclohexyl)peroxy)silane (4e): Colorless oil; R_f 0.92 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.40-7.37 (m, 1H), 7.17-7.12 (m, 3H), 2.59 (s, 3H), 2.33-2.30 (m, 2H), 1.86-1.70 (m, 5H), 1.59-1.54 (m, 2H), 1.36-1.24 (m, 1H), 0.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =143.2, 136.7, 132.4, 126.8, 126.7, 125.3, 85.4, 33.7, 25.7, 22.1, 21.9, -0.8 ppm; IR (KBr): υ_{max} 2933, 1448, 1249, 1015, 883, 842, 751, 723 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{27}O_2Si$ $[M+H]^+$ 279.1775, found 279.1774.



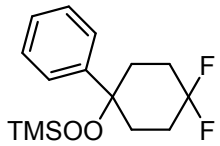
((1-(3-Chlorophenyl)cyclohexyl)peroxy)trimethylsilane (4f): Colorless oil; R_f 0.87 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 (t, *J* = 1.6 Hz, 1H), 7.32-7.29 (m, 1H), 7.27-7.23 (m, 1H), 7.22-7.19 (m, 1H), 2.11-2.06 (m, 2H), 1.78-1.63 (m, 5H), 1.59-1.51 (m, 2H), 1.34-1.26 (m, 1H), 0.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =148.1, 133.8, 129.1, 126.8, 126.4, 124.1, 83.7, 34.4, 25.5, 22.0, -1.2 ppm; IR (KBr): υ_{max} 2938, 1250, 906, 846, 783, 732, 693, 649 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{23}ClKO_2Si$ $[M+K]^+$ 337.0787, found 337.0752.



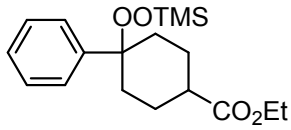
((1-(4-Fluorophenyl)cyclohexyl)peroxy)trimethylsilane (4g): Colorless oil; R_f 0.90 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43-7.38 (m, 2H), 7.03-6.97 (m, 2H), 2.10 (d, *J* = 12.8 Hz, 2H), 1.81-1.74 (m, 2H), 1.73-1.64 (m, 3H), 1.54-1.48 (m, 2H), 1.35-1.26 (m, 1H), 0.15 (s, 9); ^{13}C NMR (100 MHz, $CDCl_3$): δ =161.7 (d, *J* = 243.3 Hz), 141.3, 127.7 (d, *J* = 7.8 Hz), 114.5 (d, *J* = 20.9 Hz), 83.6, 34.5, 25.6, 22.1, -1.1 ppm; IR (KBr): υ_{max} 2937, 1605, 1510, 1447, 1250, 1232, 1161, 1013, 883, 845, 735 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{23}FNaO_2Si$ $[M+Na]^+$ 305.1344, found 305.1351.



Trimethyl((4-methyl-1-phenylcyclohexyl)peroxy)silane (4h): Colorless oil; R_f 0.92 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52-7.49 (m, 2H), 7.37-7.32 (m, 2H), 7.30-7.27 (m, 1H), 2.41-2.36 (m, 2H), 1.94-1.87 (m, 2H), 1.77-1.71 (m, 2H), 1.66-1.56 (m, 1H), 1.06-0.97 (m, 2H), 0.86-0.84 (d, *J* = 6.8 Hz, 3H), 0.02 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =127.9, 127.6, 127.3, 84.8, 32.1, 31.2, 31.0, 20.8, -1.2 ppm; IR (KBr): υ_{max} 2954, 1454, 1250, 906, 872, 845, 732 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{27}O_2Si$ $[M+H]^+$ 279.1775, found 279.1758.

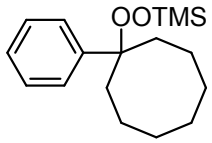


((4,4-difluoro-1-phenylcyclohexyl)peroxy)trimethylsilane (4i): Colorless oil; R_f 0.89 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44-7.42 (m, 2H), 7.37-7.33 (m, 2H), 7.30-7.27 (m, 1H), 2.32-2.28 (m, 2H), 2.22-2.14 (m, 1H), 2.12-1.97 (m, 5H), 0.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =143.8, 128.0, 127.3, 125.6, 123.3 (dd, *J* = 240.6, 236.2 Hz), 82.4, 30.8 (d, *J* = 9.4 Hz), 29.9 (dd, *J* = 25.4, 24.0 Hz), -1.3 ppm; ^{19}F NMR (376 MHz, $CDCl_3$) δ = -92.4 (d, *J* = -235.0 Hz), -102.7 (d, *J* = -235.0 Hz).; IR (KBr): υ_{max} 2959, 1376, 1252, 1220, 1108, 989, 973, 908, 874, 846, 698 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{23}F_2O_2Si$ $[M+H]^+$ 301.1430, found 301.1411.

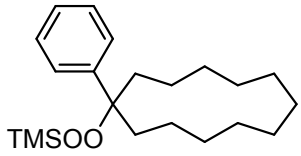


Ethyl 4-phenyl-4-((trimethylsilyl)peroxy)cyclohexane-1-carboxylate (4j): Colorless oil; R_f 0.93 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44-7.42 (m, 2H), 7.35-7.32 (m, 2H), 7.27-7.23 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.39-2.32 (m, 1H), 2.31 -2.27 (m, 2H), 1.98-1.84 (m, 2H), 1.78-1.70 (m, 2H), 1.28 (t, *J* = 7.2 Hz, 3H), 0.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =175.8, 145.6, 127.9, 126.8, 125.5, 82.7, 60.2, 42.4, 33.5, 24.4, 14.2, -1.2 ppm; IR (KBr): υ_{max} 2959, 1726,

1446, 1251, 1181, 1042, 907, 880, 845, 730, 697 cm^{-1}; HRMS (ESI) calcd for $C_{18}H_{28}NaO_4Si$ $[M+Na]^+$ 359.1649, found 359.1640.

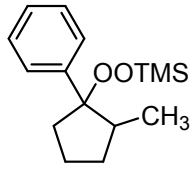


Trimethyl((1-phenylcyclooctyl)peroxy)silane (4m): Colorless oil; R_f 0.91 (EtOAc/petroleum ether = 1:20). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.45 (d, *J* = 8.4 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.26-7.22 (m, 1H), 2.26-2.19 (m, 2H), 1.95-1.89 (m, 2H), 1.68 (br s, 5H), 1.53-1.47 (m, 5H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =145.2, 127.7, 126.6, 126.5, 88.3, 31.9, 28.4, 25.0, 22.1, -1.1 ppm; IR (KBr): υ_{max} 2924, 1446, 1249, 905, 846, 731, 698, 649 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{29}O_2Si$ $[M+Na]^+$ 293.1931, found 293.1925.

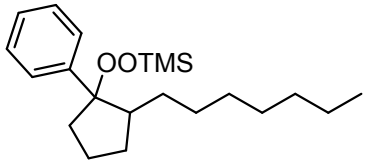


Trimethyl((1-phenylcyclododecyl)peroxy)silane (4n): Colorless oil; R_f 0.88 (EtOAc/petroleum ether = 1:20). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.40 (d, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 8.0 Hz, 2H), 7.25-7.22 (m, 1H), 2.08-2.01 (m, 2H), 1.68-1.62 (m, 2H), 1.44-1.16 (m, 18H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =145.3, 127.5, 126.5, 126.2, 87.7, 30.6, 26.3, 26.2, 22.3, 22.0, 19.4, -1.1 ppm; IR (KBr): υ_{max} 2931, 1470, 1446, 1249, 894, 866, 845, 732, 697 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{37}O_2Si$ $[M+H]^+$ 349.2557, found 349.2554.

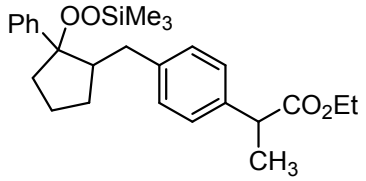
Characterization of New Alkylsilyl Peroxides 6



Trimethyl((2-methyl-1-phenylcyclopentyl)peroxy)silane (6a): Colorless oil; R_f 0.93 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44-7.42 (m, 2H), 7.38-7.34 (m, 2H), 7.30-7.28 (m, 1H), 2.55-2.49 (m, 1H), 2.28-2.22 (m, 2H), 2.19-2.10 (m, 1H), 1.90-1.84 (m, 2H), 1.44-1.36 (m, 1H), 0.59 (d, *J* = 7.2 Hz, 3H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.6, 127.5, 127.4, 126.8, 97.7, 41.9, 32.6, 30.8, 21.5, 18.2, -1.2 ppm; IR (KBr): υ_{max} 2941, 2862, 1447, 1249, 1213, 904, 874, 755, 735, 697 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{25}O_2Si$ $[M+H]^+$ 265.1618, found 265.1612.

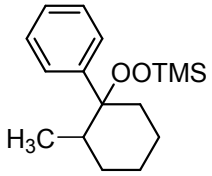


((2-Heptyl-1-phenylcyclopentyl)peroxy)trimethylsilane (6b): Colorless oil; R_f 0.88 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39-7.36 (m, 2H), 7.34-7.30 (m, 2H), 7.26-7.22 (m, 1H), 2.51-2.45 (m, 1H), 2.17-2.09 (m, 1H), 2.07-1.95 (m, 2H), 1.90-1.76 (m, 2H), 1.46-1.38 (m, 1H), 1.27-1.05 (m, 11H), 0.85 (t, J = 6.8 Hz, 3H), 0.67-0.58 (m, 1H), 0.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.9, 127.4, 127.3, 126.6, 97.5, 47.6, 32.2, 31.9, 31.8, 30.1, 29.5, 29.2, 28.0, 22.6, 21.8, 14.1 -1.2 ppm; IR (KBr): υ_{max} 2956, 2926, 1448, 1249, 1213, 907, 885, 845, 734, 698, 650 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{37}O_2Si$ $[M+H]^+$ 349.2557, found 349.2542.



Ethyl 2-(4-((2-phenyl-2-((trimethylsilyl)peroxy)cyclopentyl)methyl)phenyl) propanoate (6c): Colorless oil; R_f 0.80 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48-7.46 (m, 2H), 7.38-7.34 (m, 2H), 7.29-7.25 (m, 1H),

7.16 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 8.0 Hz, 2H), 4.18-4.05 (m, 1H), 3.66 (q, J = 7.2 Hz, 1H), 2.57-2.51 (m, 1H), 2.44-2.39 (m, 1H), 2.33-2.22 (m, 2H), 1.92-1.78 (m, 4H), 1.52-1.42 (m, 4H), 1.21 (t, J = 7.2 Hz, 3H), 0.14 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =174.7, 141.5, 139.9, 137.9, 129.0, 127.6, 127.4, 127.1, 126.9, 97.1, 60.6, 49.1, 49.1, 45.1, 37.7, 32.1, 29.5, 21.5, 18.5, 14.1, -1.2 ppm; IR (KBr): υ_{max} 2928, 1732, 1684, 1449, 1372, 1321, 1260, 1211, 1143, 1090, 1023, 908, 799, 731, 690, 649 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{37}O_2Si$ $[M+H]^+$ 463.2275, found 463.2279.



Trimethyl((2-methyl-1-phenylcyclohexyl)peroxy)silane (6d): Colorless oil; R_f 0.93 (EtOAc/petroleum ether = 1:20). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38-7.35 (m, 2H), 7.33-7.29 (m, 2H), 7.25-7.21 (m, 1H), 2.34-2.30 (m, 1H), 2.05-1.93 (m, 3H), 1.81-1.69 (m, 1H), 1.65-1.61 (m, 1H), 1.52 -1.44 (m, 2H), 1.36-1.33 (m, 1H), 0.64 (d, J = 9.2 Hz, 3H), 0.14 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =145.1, 127.5, 126.5, 126.4, 86.6, 37.2, 28.7, 25.3, 21.5, 19.7, 15.8, -1.1 ppm; IR (KBr): υ_{max} 2941, 2862, 1447, 1249, 1213, 904, 874, 755, 735, 697 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{27}O_2Si$ $[M+H]^+$ 279.1775, found 279.1774.

References

(1) (a) R. Sakamoto, S. Sakurai and K. Maruoka, *Chem.-Eur. J.*, 2017, **23**, 9030; (b) R. Sakamoto, T. Kato, S. Sakurai and K. Maruoka, *Org. Lett.*, 2018, **20**, 1400; (c) T. Seihara, S. Sakurai, T. Kato, R. Sakamoto and K. Maruoka, *Org. Lett.*, 2019, **21**, 2477; (d) P. C. Too, Y. L. Tnay and S. Chiba, *Beilstein J. Org. Chem.*, 2013, **9**, 1217.

(2) (a) Y. Sumida, H. Yorimitsu and K. Oshima, *J. Org. Chem.*, 2009, **74**, 3196; (b) J. R. Smith, B. S. L. Collins, M. J. Hesse, M. A. Graham, E. L. Myers and V. K. Aggarwal, *J. Am. Chem. Soc.*, 2017, **139**, 9148.

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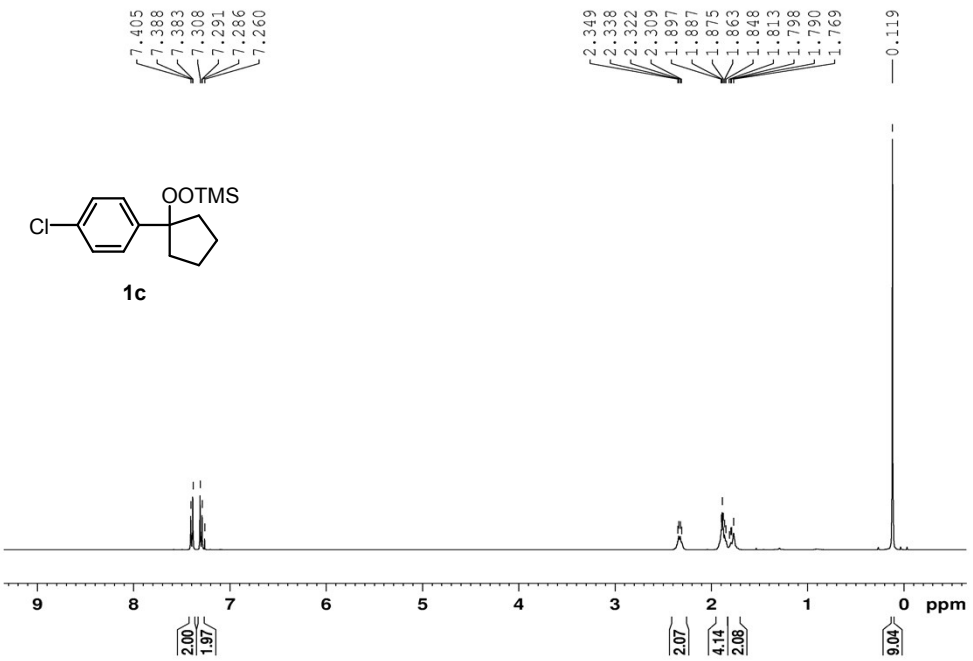
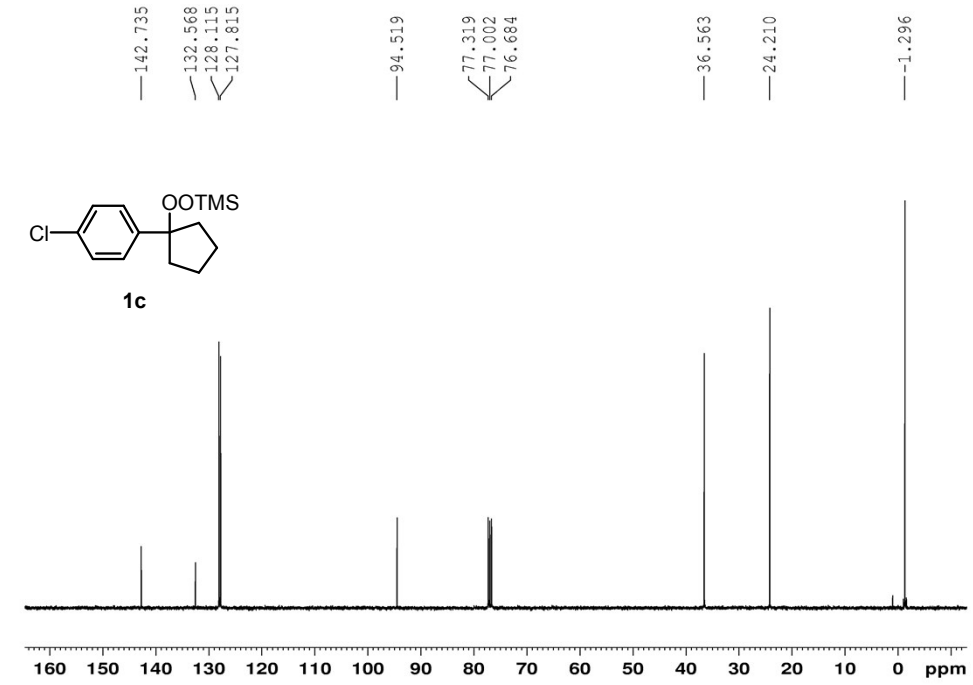
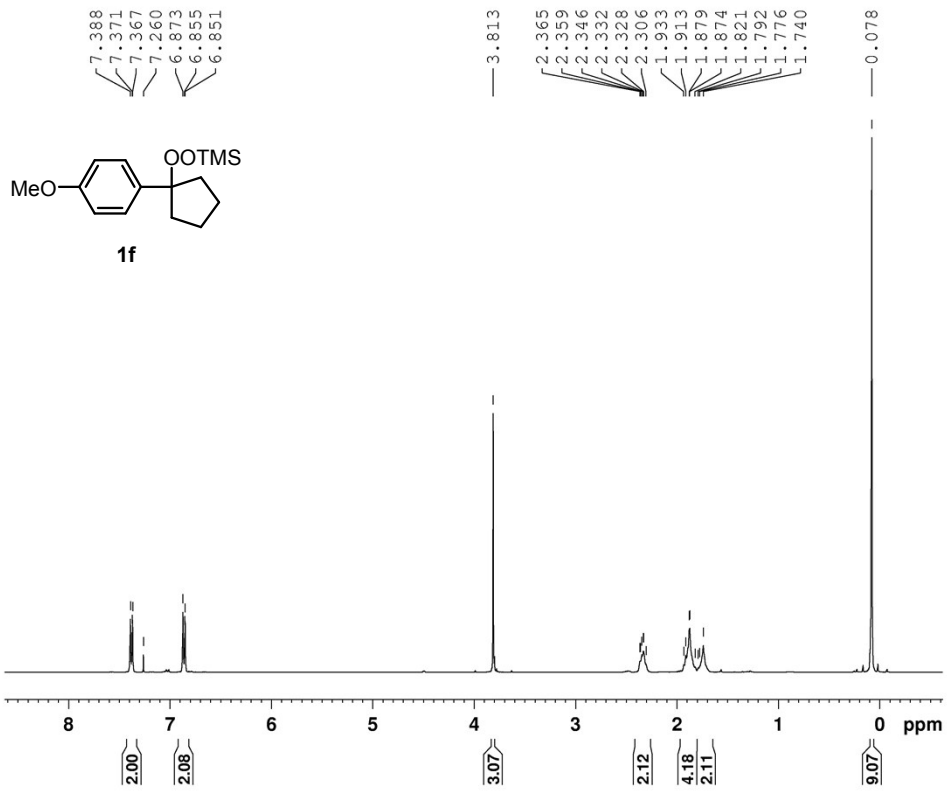
^{1}H NMR and ^{13}C NMR Spectra of the Starting Materials **1**, **4**, and **6**



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MeO
1f
8
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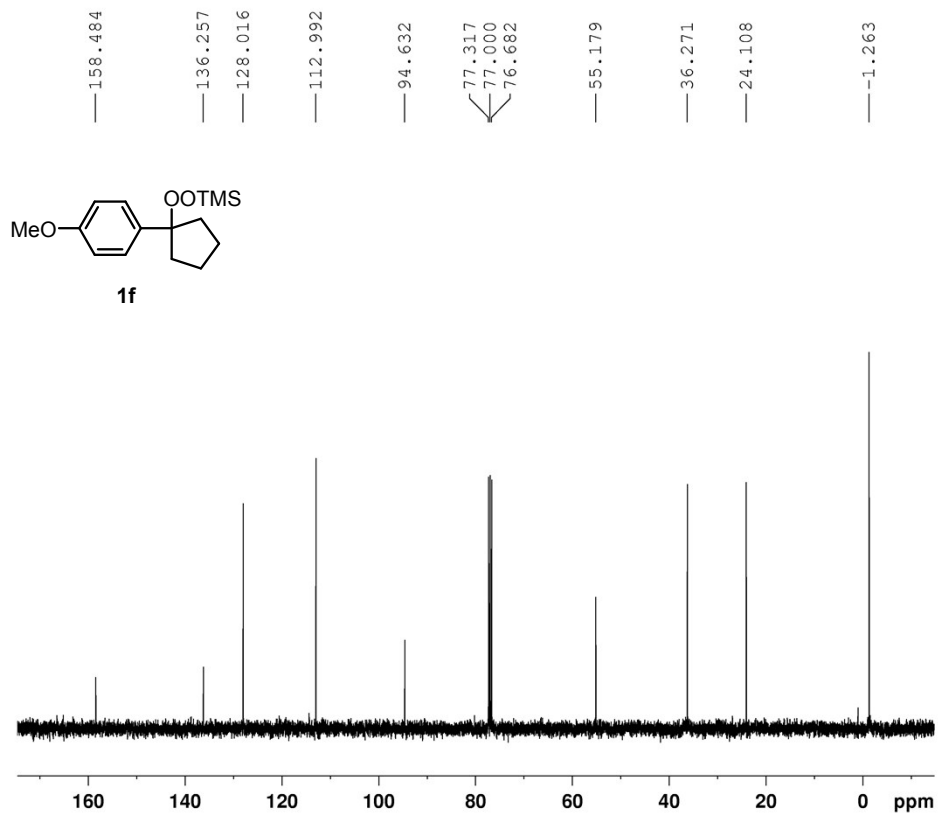
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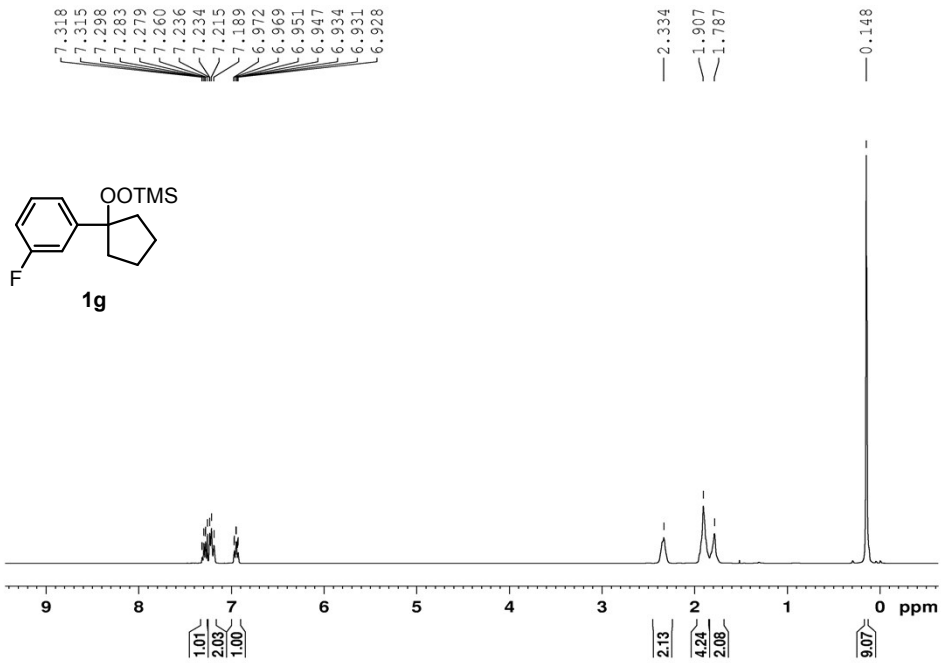
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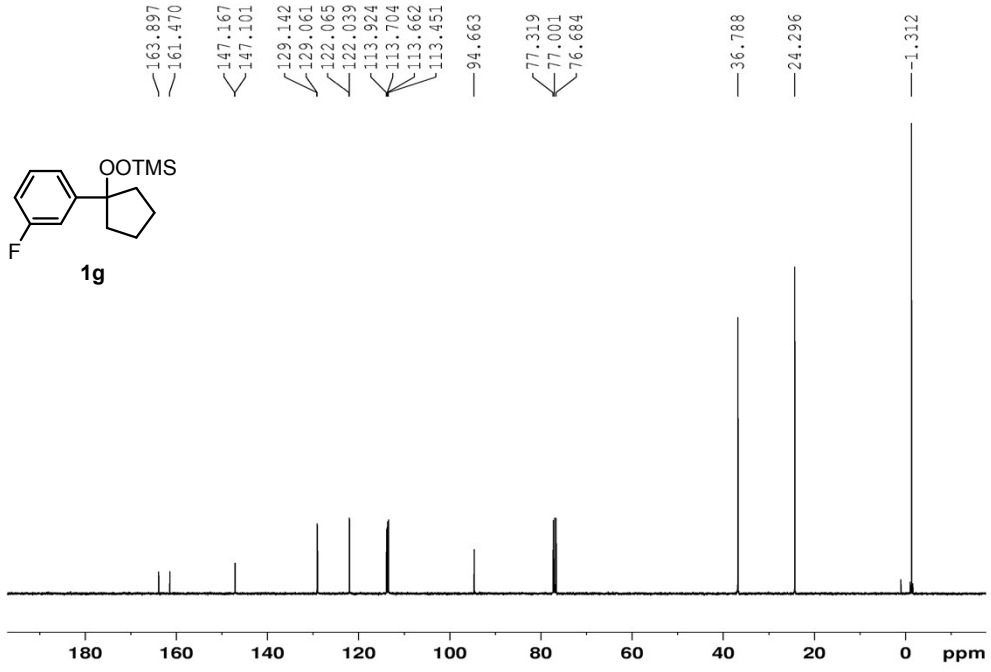
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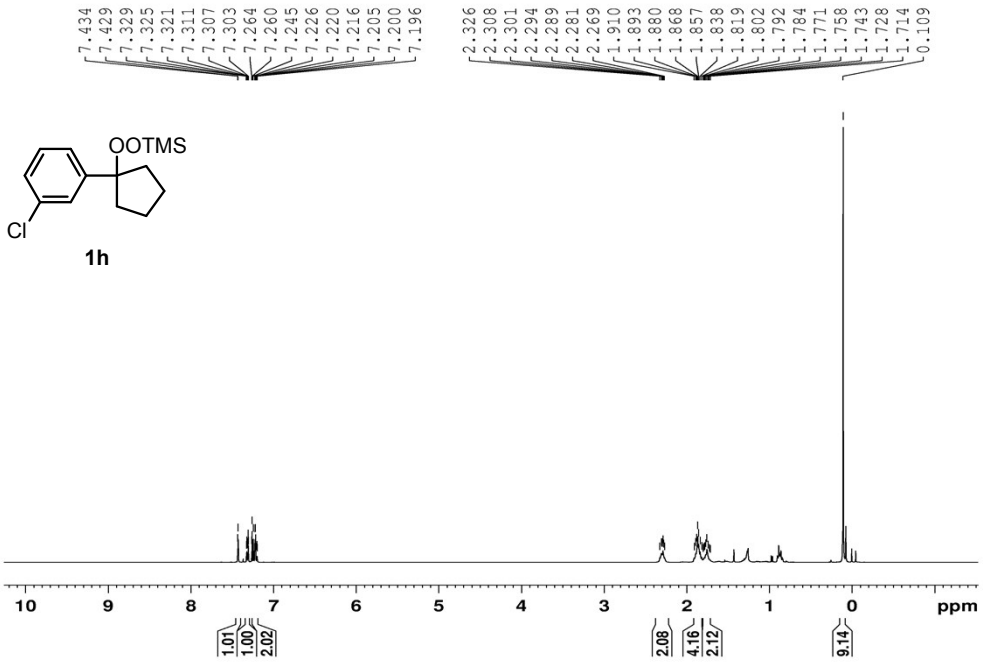
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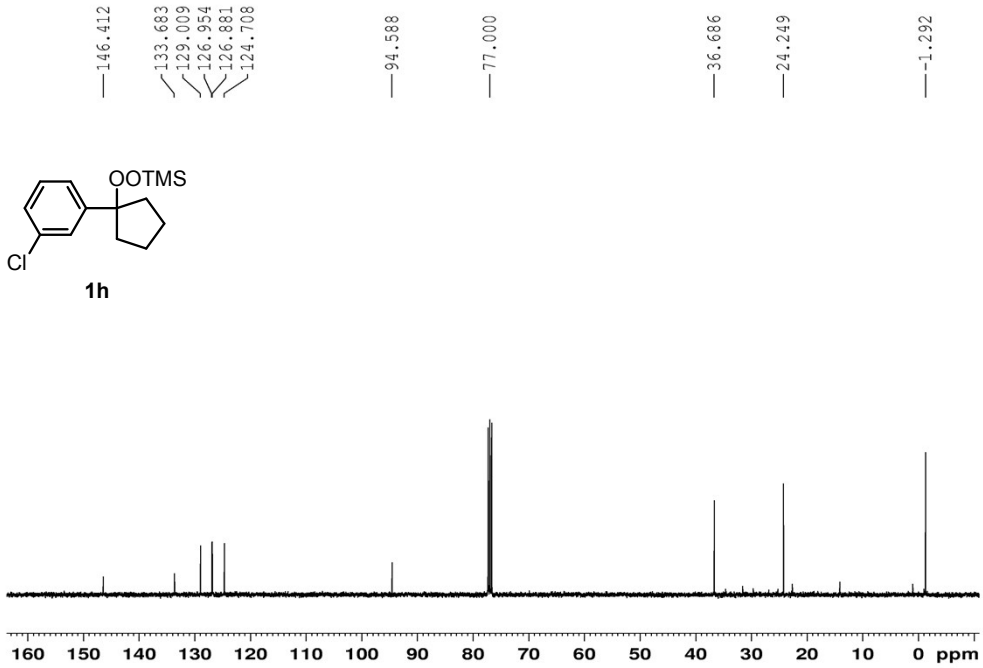
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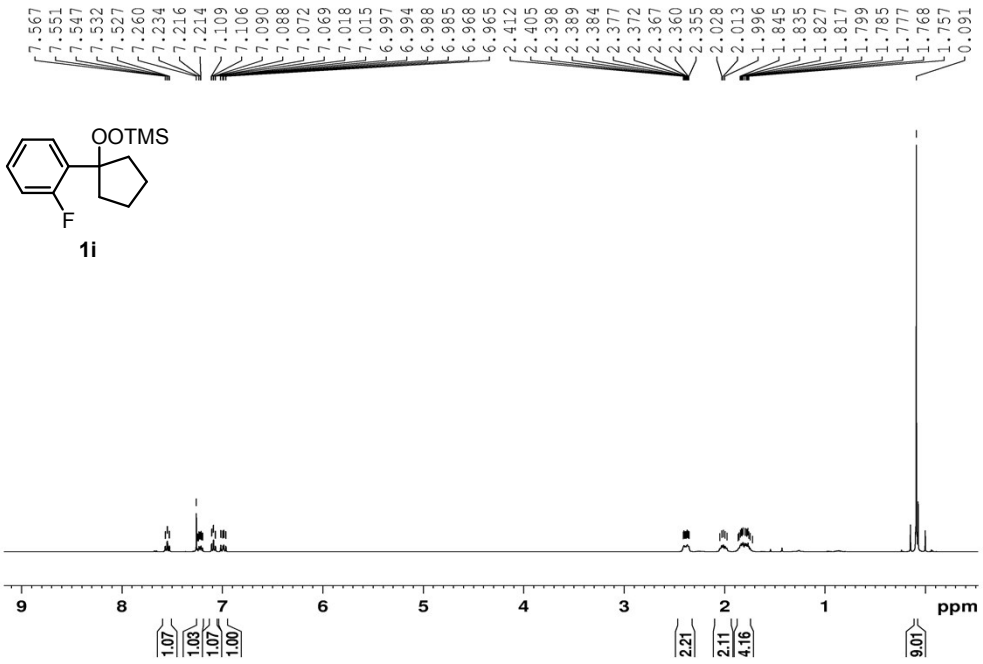
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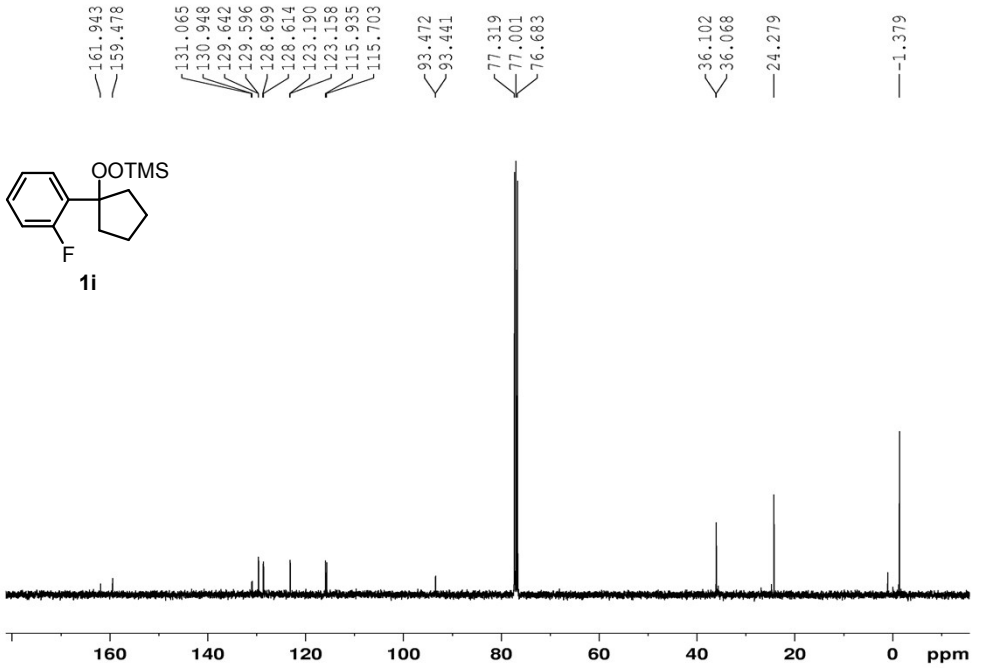
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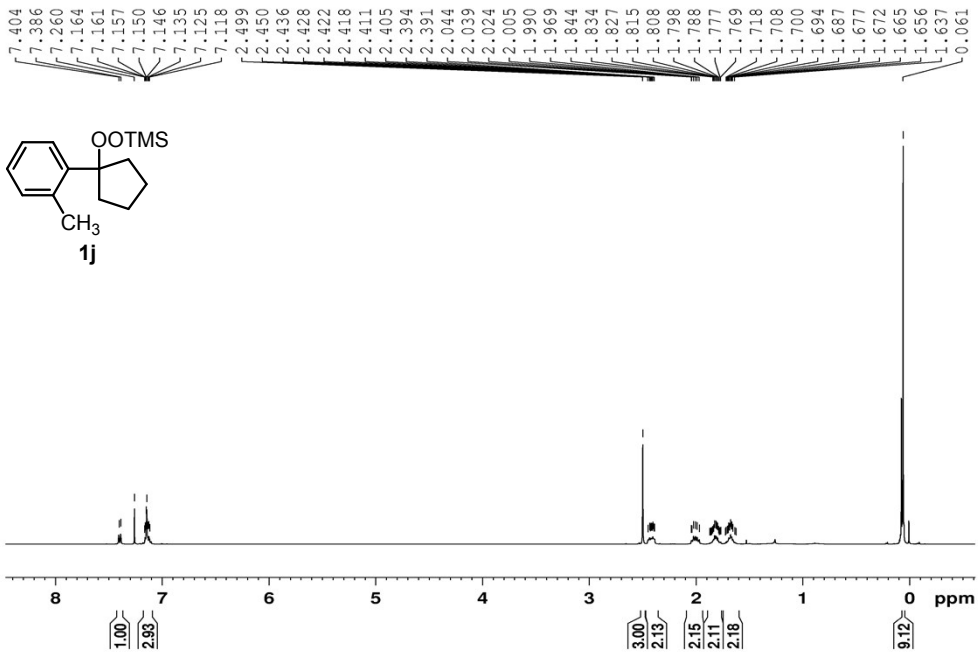
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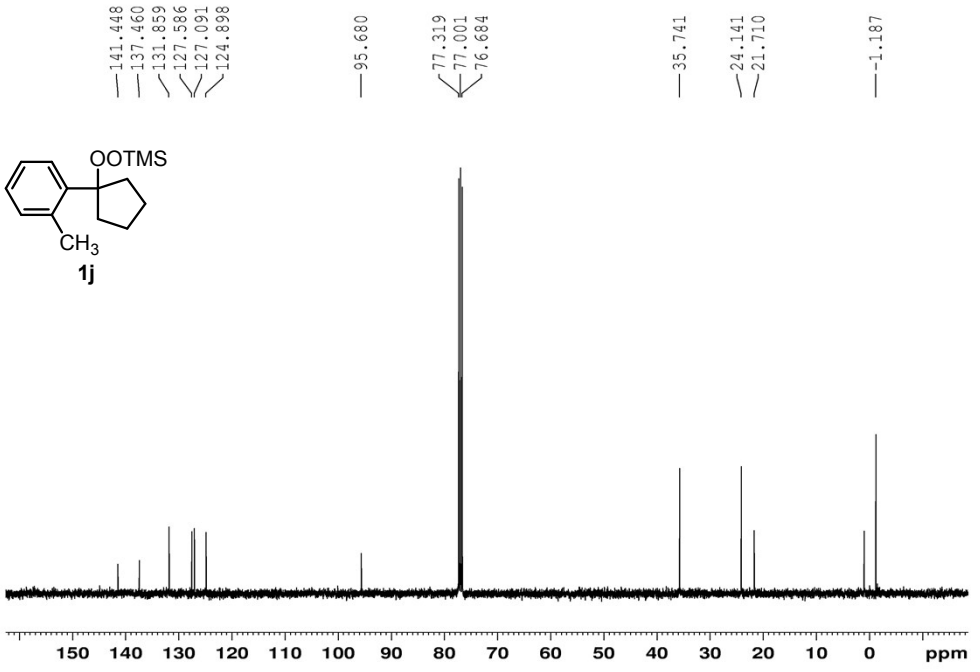
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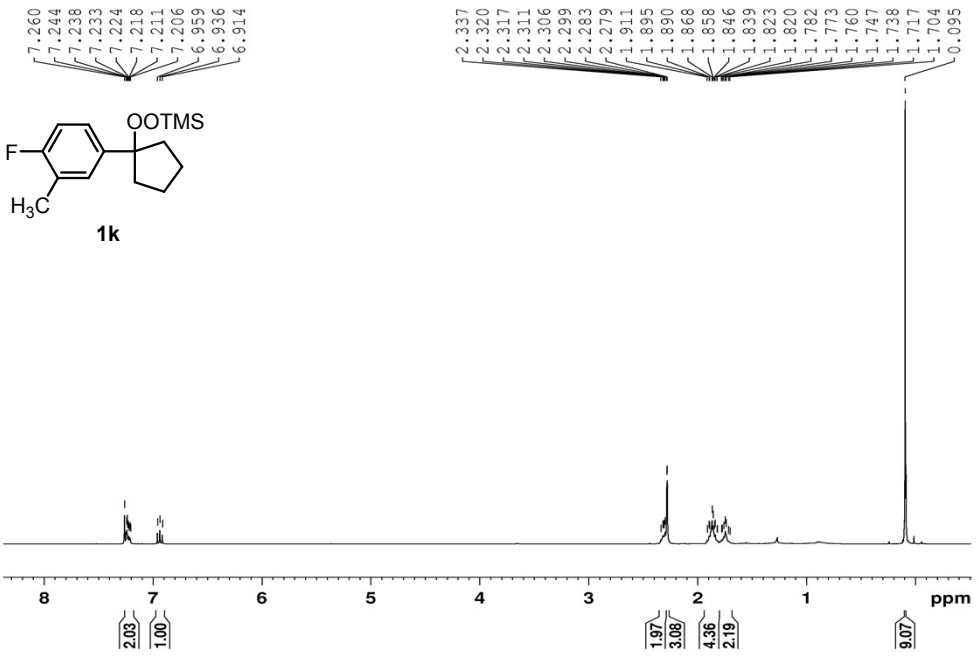
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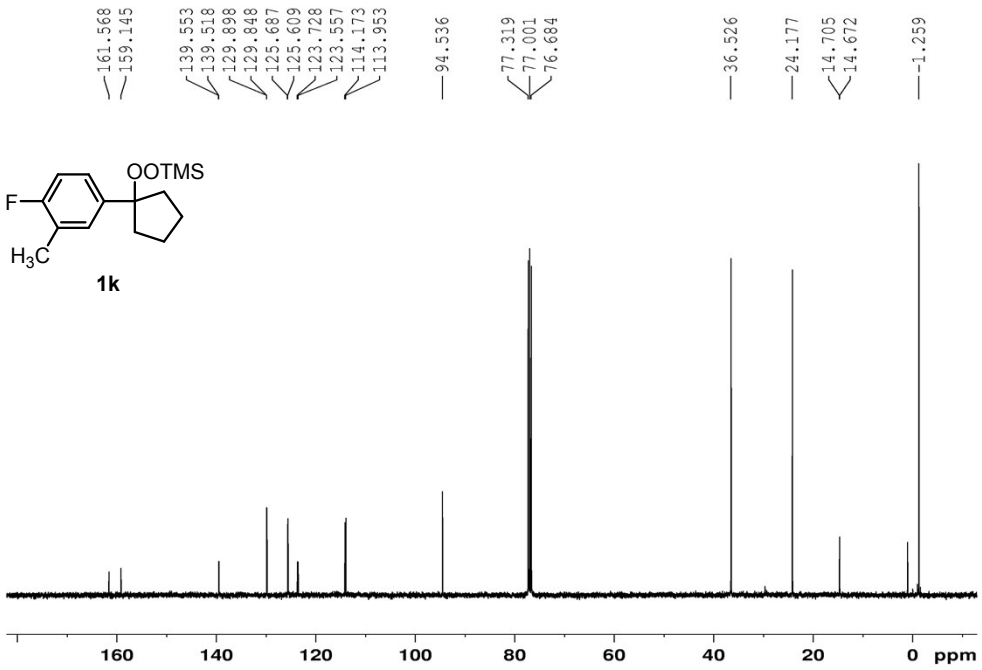
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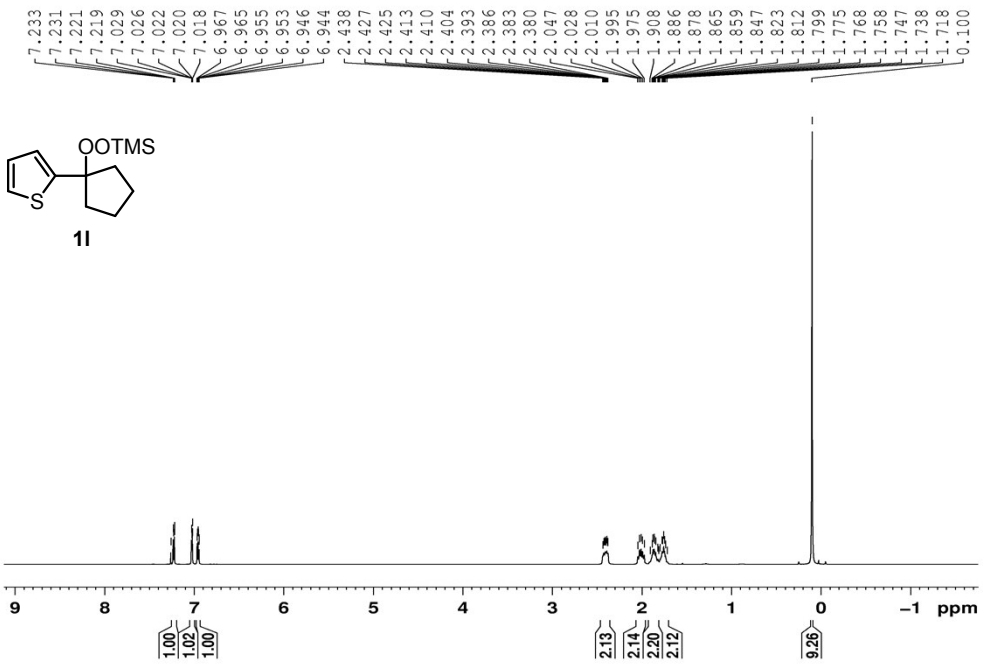
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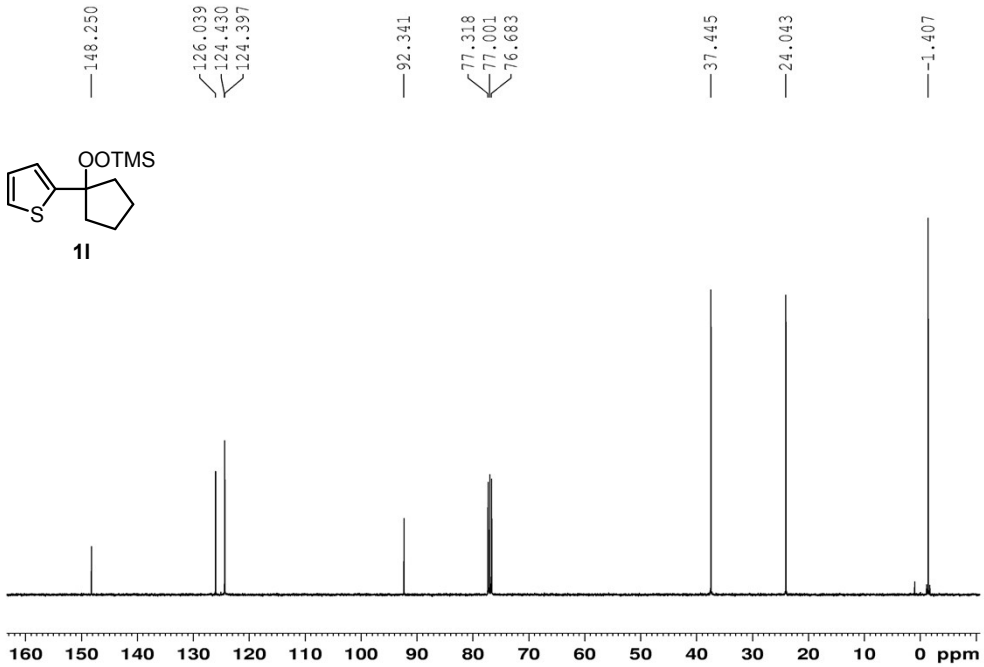
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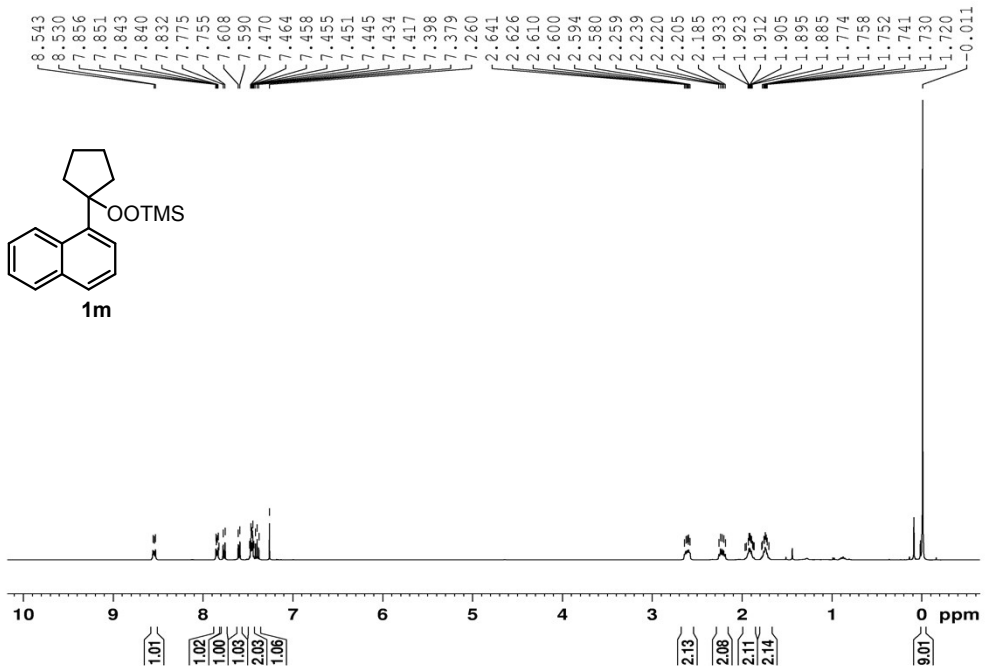
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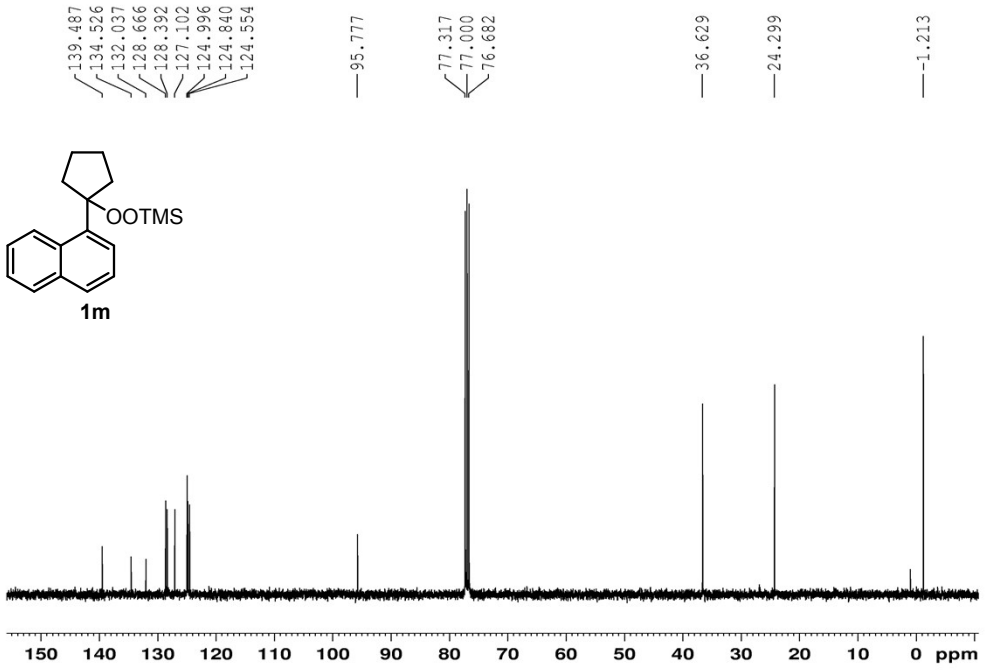
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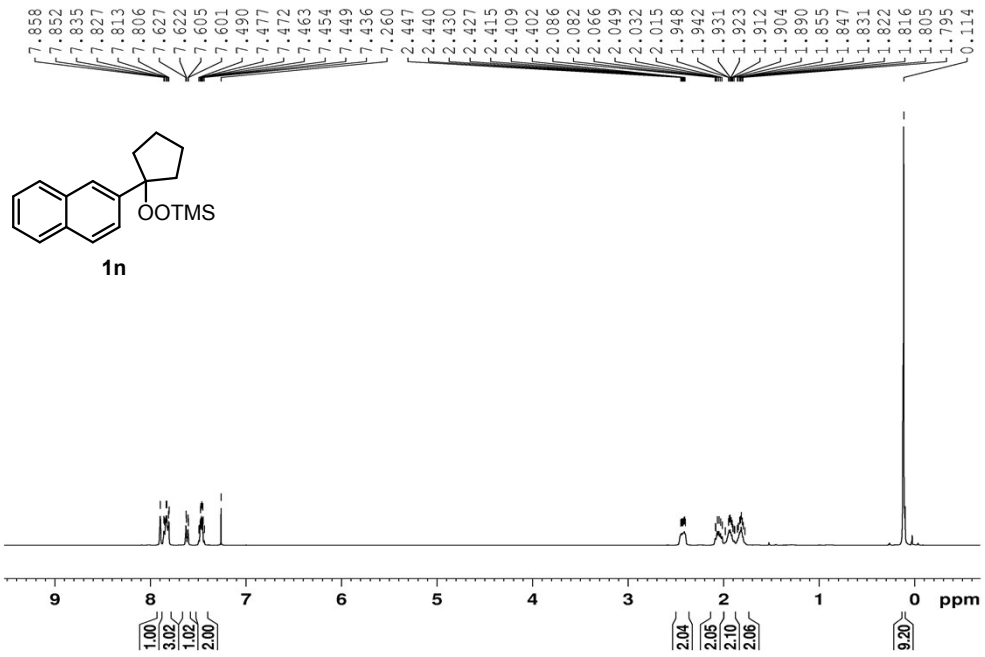
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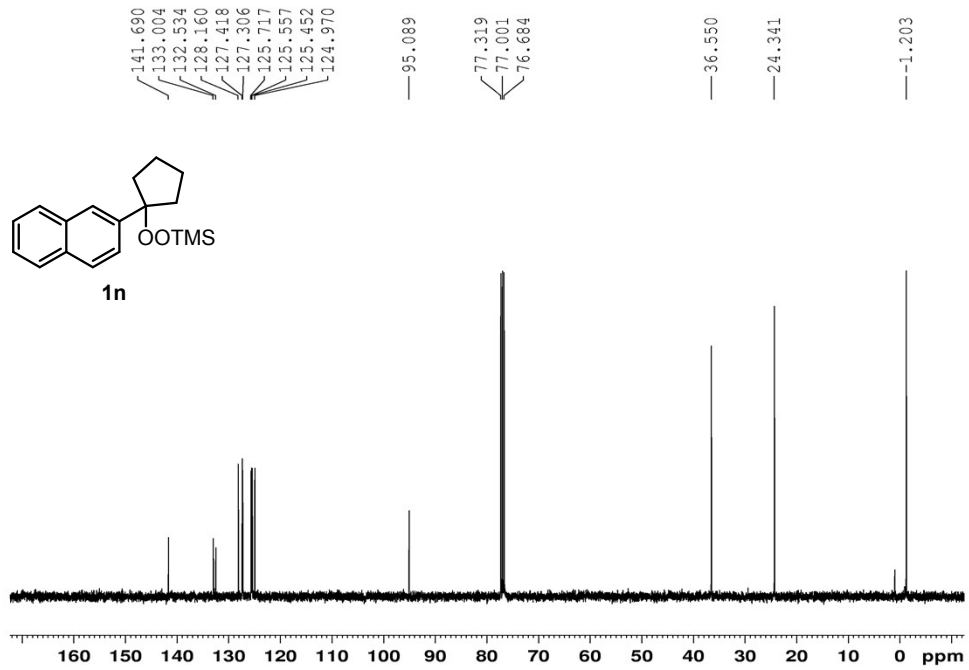
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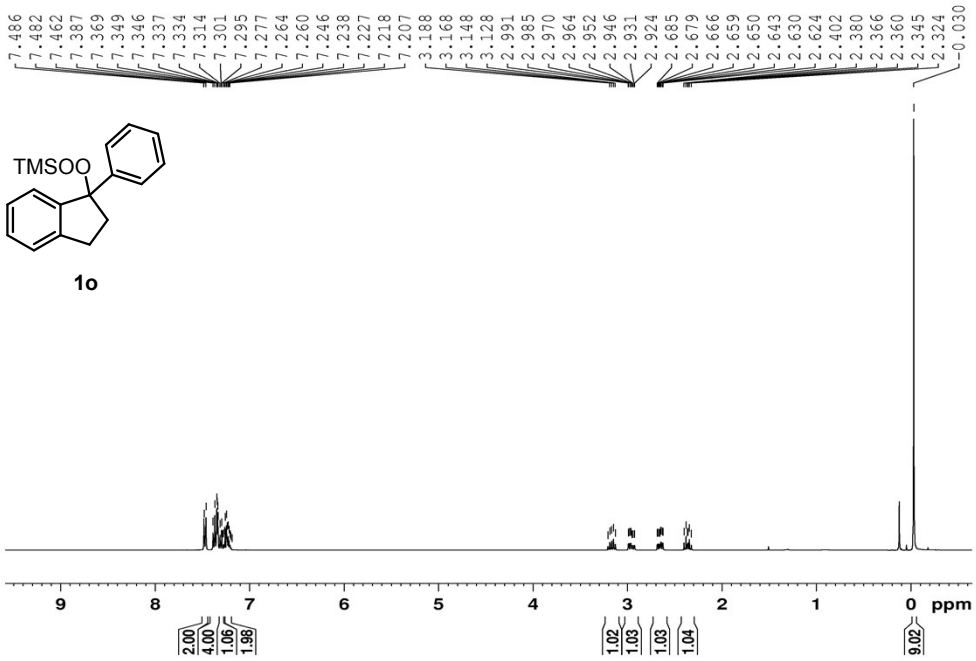
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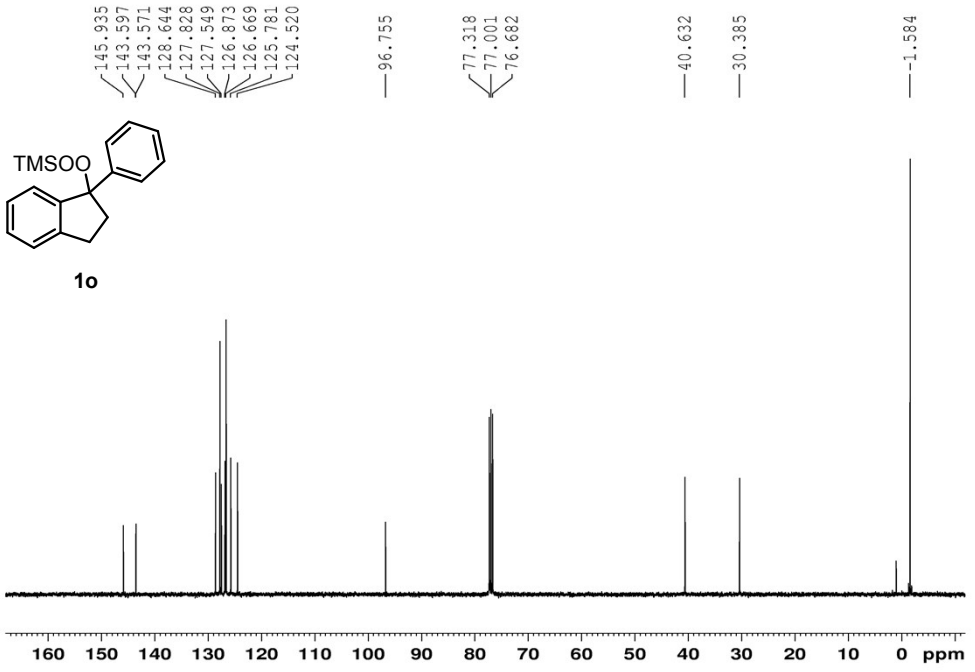
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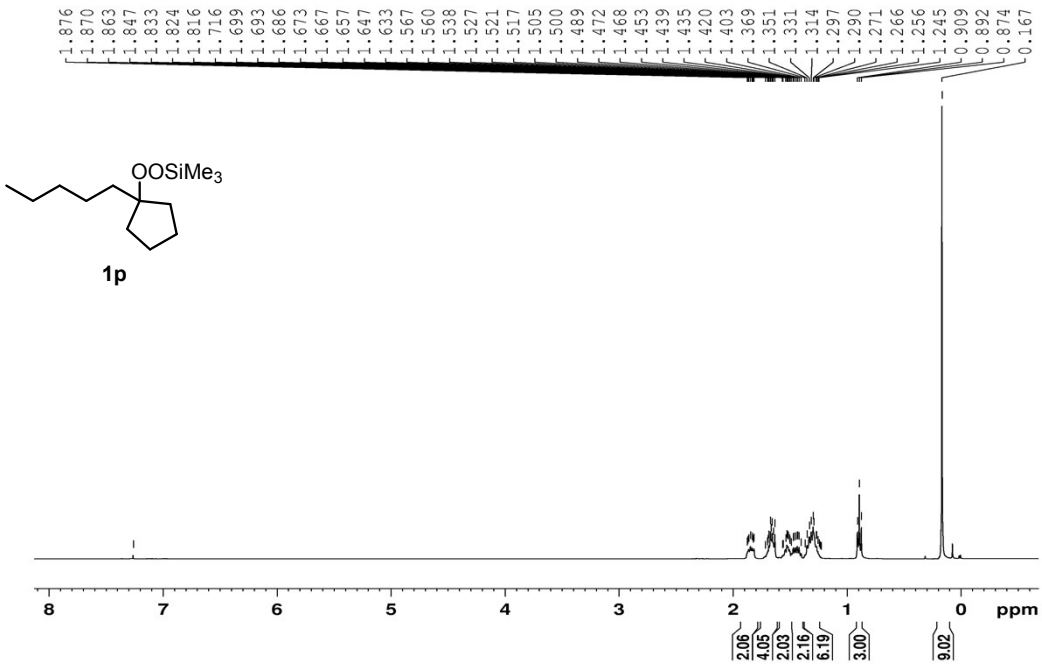
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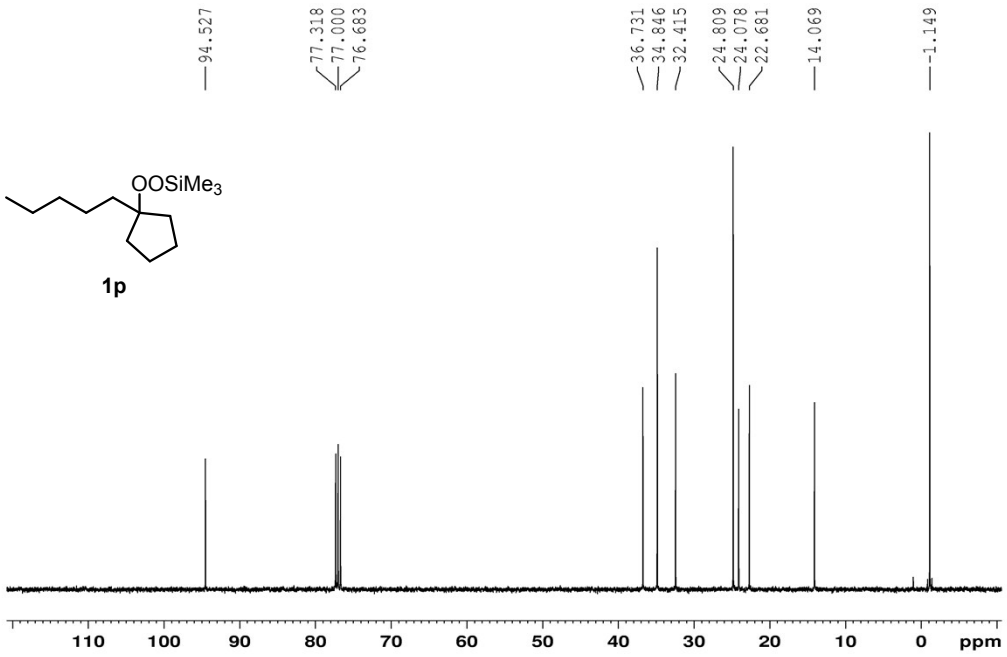
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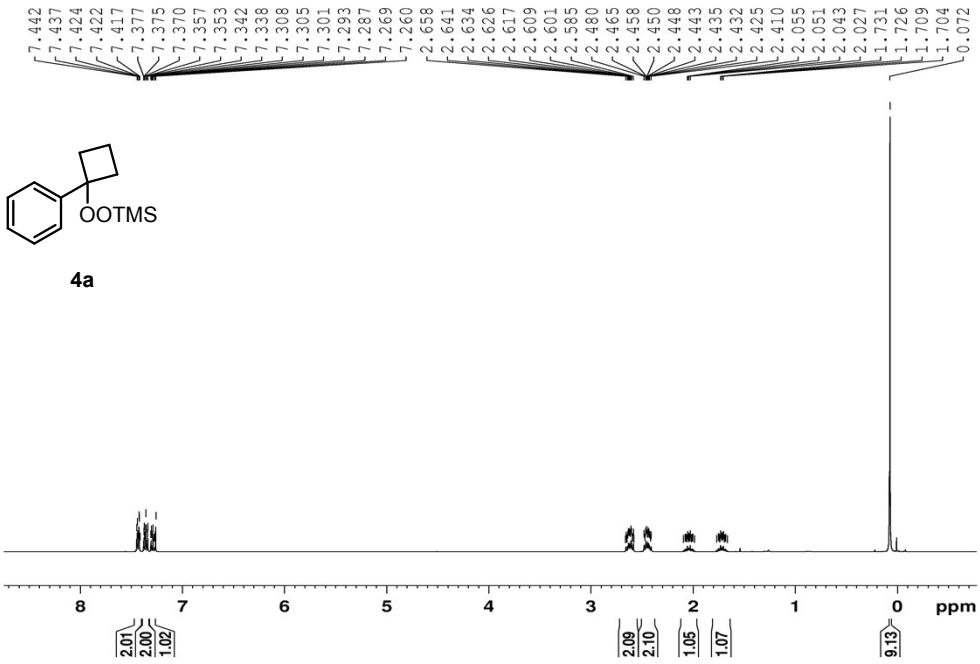
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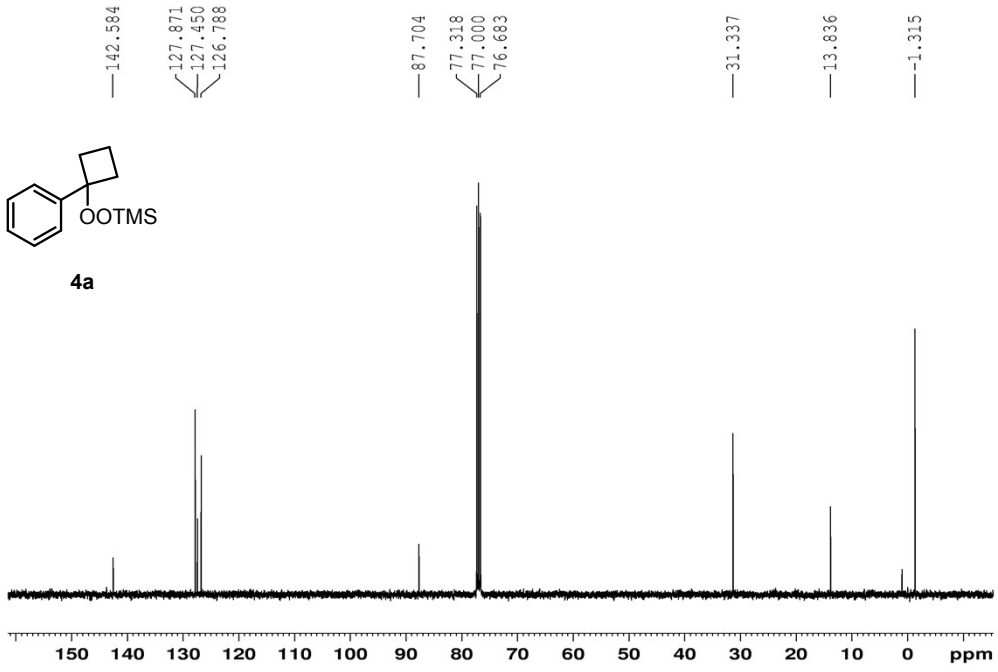
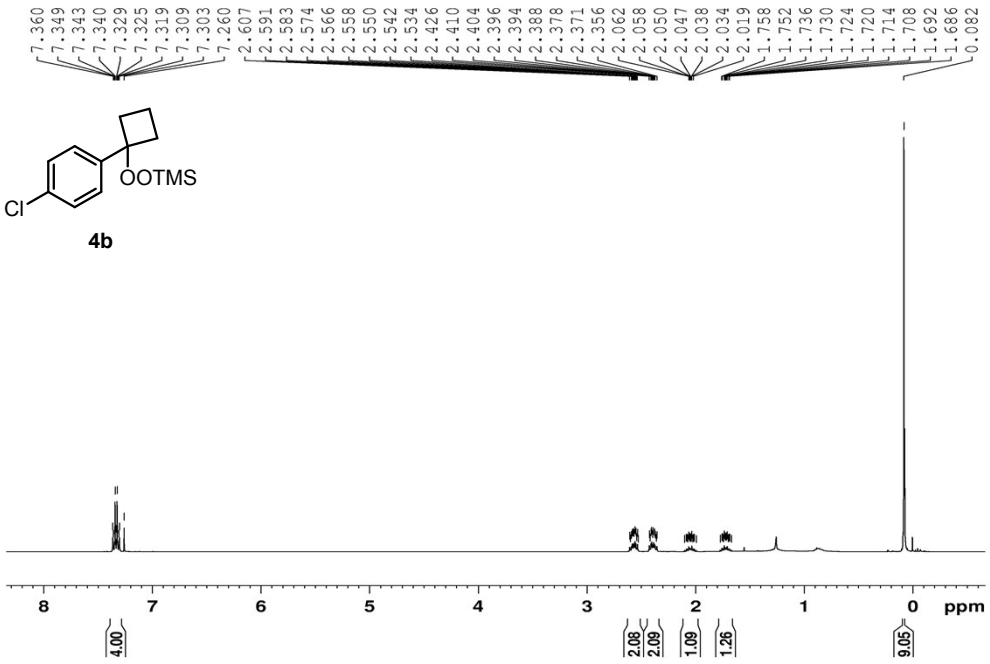
7.370
7.357
7.353
7.342
7.338
7.308
7.305
7.301
7.293
7.287
7.269
7.260
2.658
2.641
2.634
2.626
2.617
2.609
2.601
2.585
2.480
2.465
2.458
2.450
2.448
2.443
2.435
2.432
2.425
2.410
2.055
2.051
2.043
2.027
1.731
1.726
1.709
1.704
0.072
OOTMS
8
7
6
5
4
3
2
1
0
ppm
2.01
2.00
1.02
2.09
2.10
1.05
1.07
9.13

4a


142.584
127.871
127.450
126.788
87.704
77.318
77.000
76.683
31.337
13.836
-1.315
OOTMS
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4a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7.340
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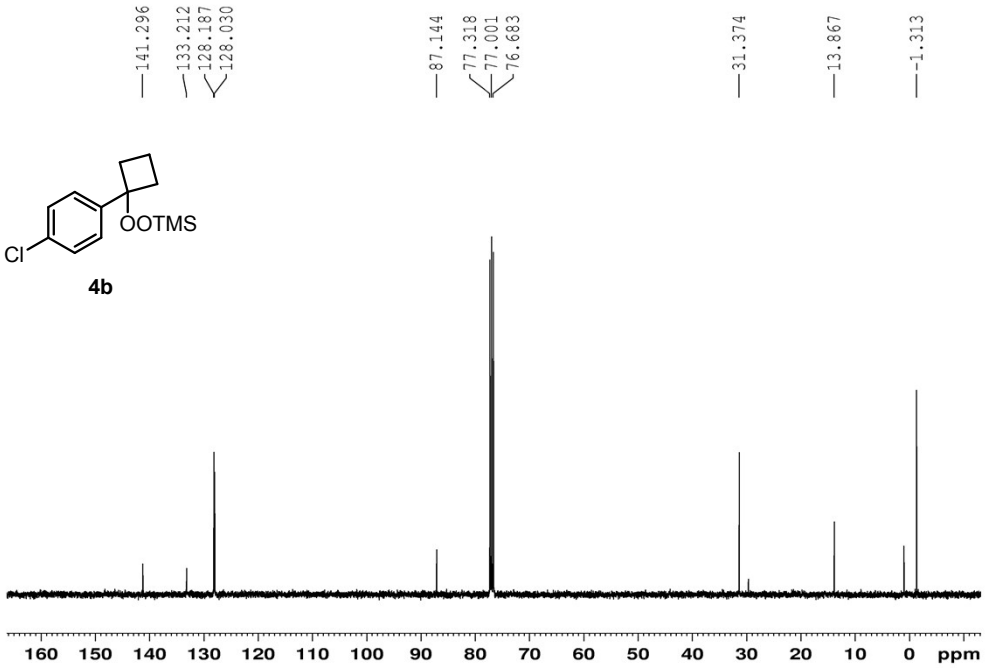
7.325
7.319
7.309
7.303
7.260
2.607
2.591
2.583
2.574
2.566
2.558
2.550
2.542
2.534
2.426
2.410
2.404
2.396
2.394
2.388
2.378
2.371
2.356
2.062
2.058
2.050
2.047
2.038
2.034
2.019
1.758
1.752
1.736
1.730
1.724
1.720
1.714
1.708
1.692
1.686
0.082
OOTMS
Cl
8
7
6
5
4
3
2
1
0
ppm
4.00
2.08
2.09
1.09
1.26
9.05

4b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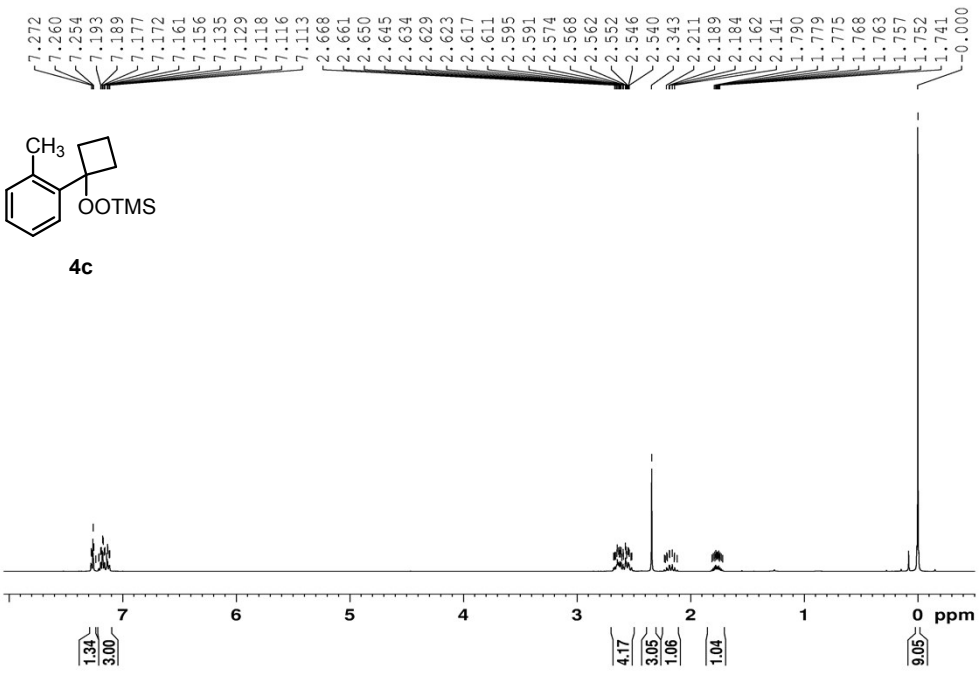
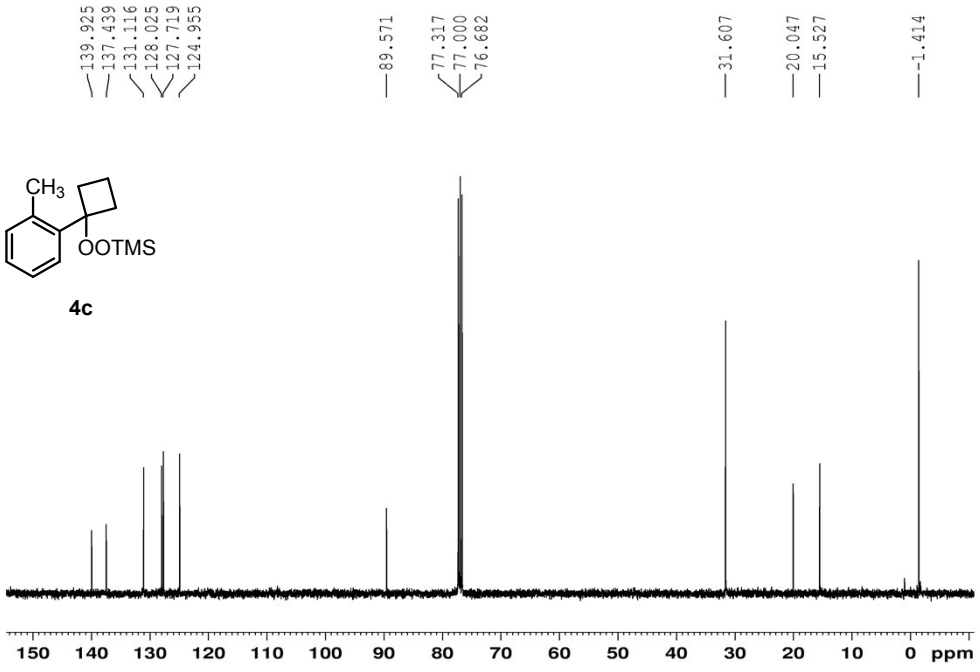
133.212
128.187
128.030
87.144
77.318
77.001
76.683
31.374
13.867
-1.313
OOTMS
Cl
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4b


7.272
7.260
7.254
7.193
7.189
7.177
7.172
7.161
7.156
7.135
7.129
7.118
7.116
7.113
2.668
2.661
2.650
2.645
2.634
2.629
2.623
2.617
2.611
2.595
2.591
2.574
2.568
2.562
2.552
2.546
2.540
2.343
2.211
2.189
2.184
2.162
2.141
1.790
1.779
1.775
1.768
1.763
1.757
1.752
1.741
-0.000
CH3
OOTMS
4c
7
6
5
4
3
2
1
0 ppm
1.34
3.00
4.17
3.05
1.06
1.04
9.05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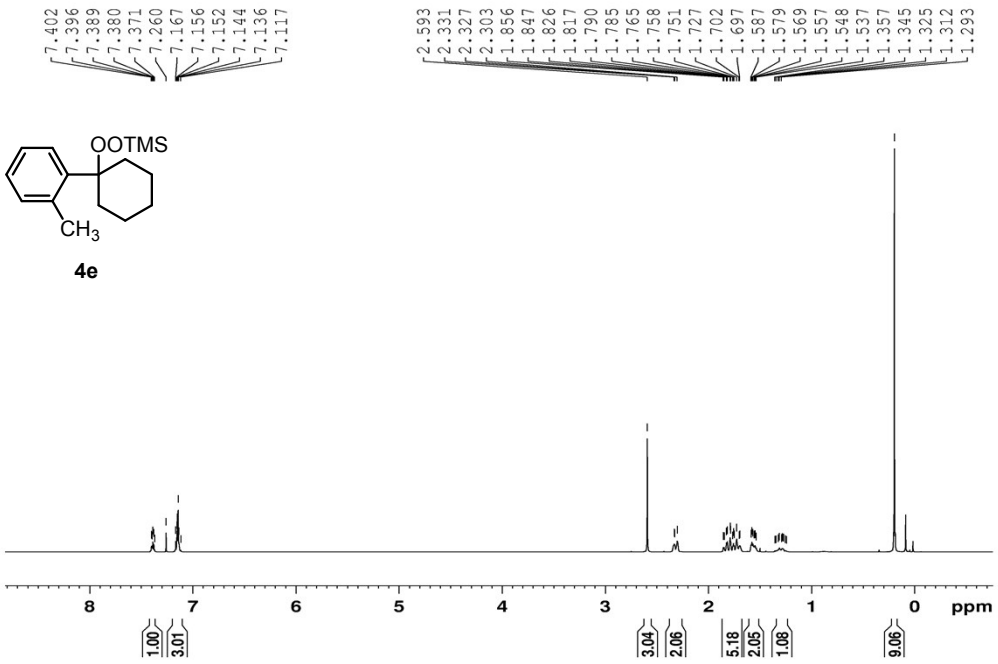
137.439
131.116
128.025
127.719
124.955
89.571
77.317
77.000
76.682
31.607
20.047
15.527
-1.414
CH3
OOTMS
4c
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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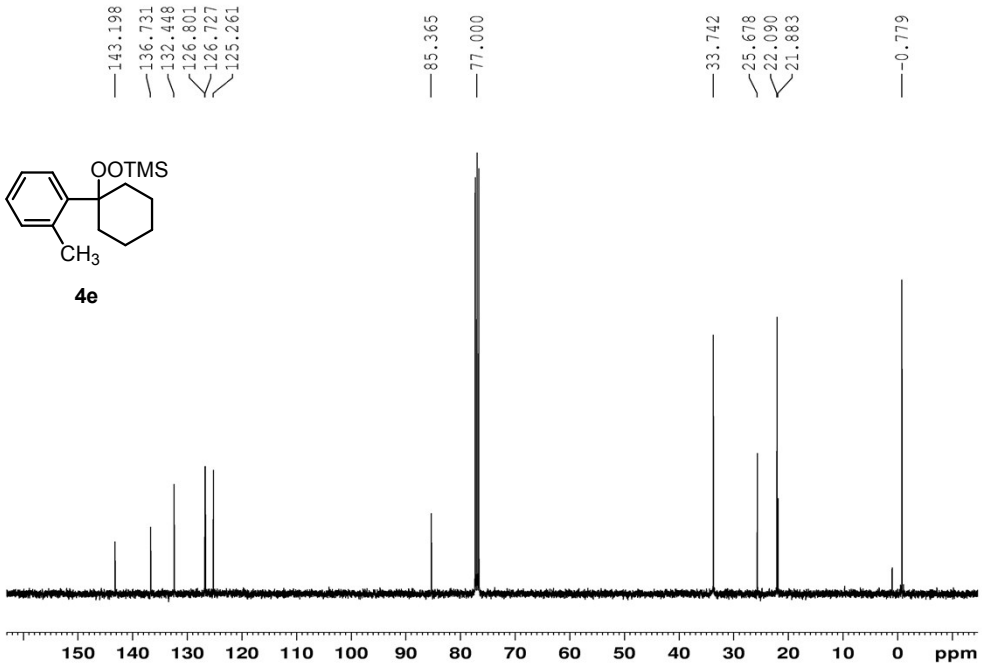
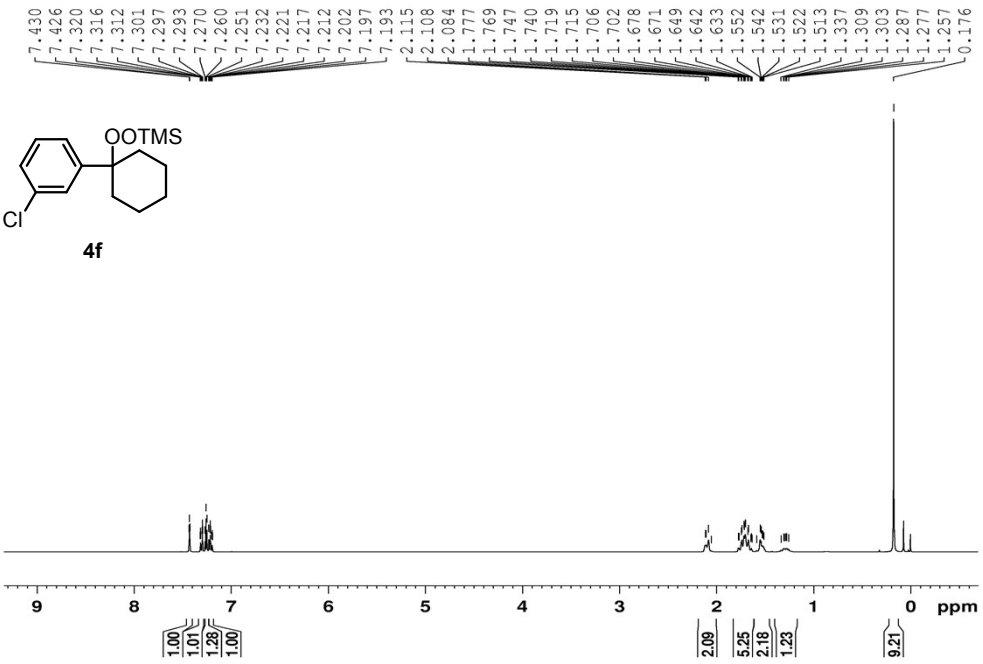
7.396
7.389
7.380
7.371
7.260
7.167
7.156
7.152
7.144
7.136
7.117
2.593
2.331
2.327
2.303
1.856
1.847
1.826
1.817
1.790
1.785
1.765
1.758
1.751
1.727
1.702
1.697
1.587
1.579
1.569
1.557
1.548
1.537
1.357
1.345
1.325
1.312
1.293
OOTMS
CH3
8
7
6
5
4
3
2
1
0
ppm
1.00
3.01
3.04
2.06
5.18
2.05
1.08
9.06

4e


143.198
136.731
132.448
126.801
126.727
125.261
85.365
77.000
33.742
25.678
22.090
21.883
-0.779
OOTMS
CH3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4e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7.426
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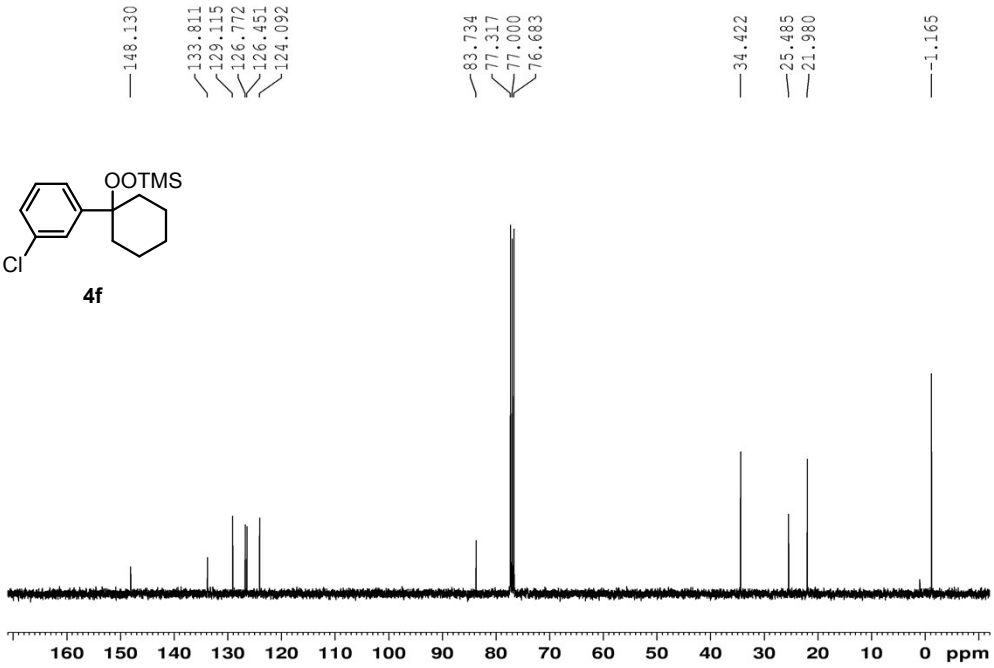
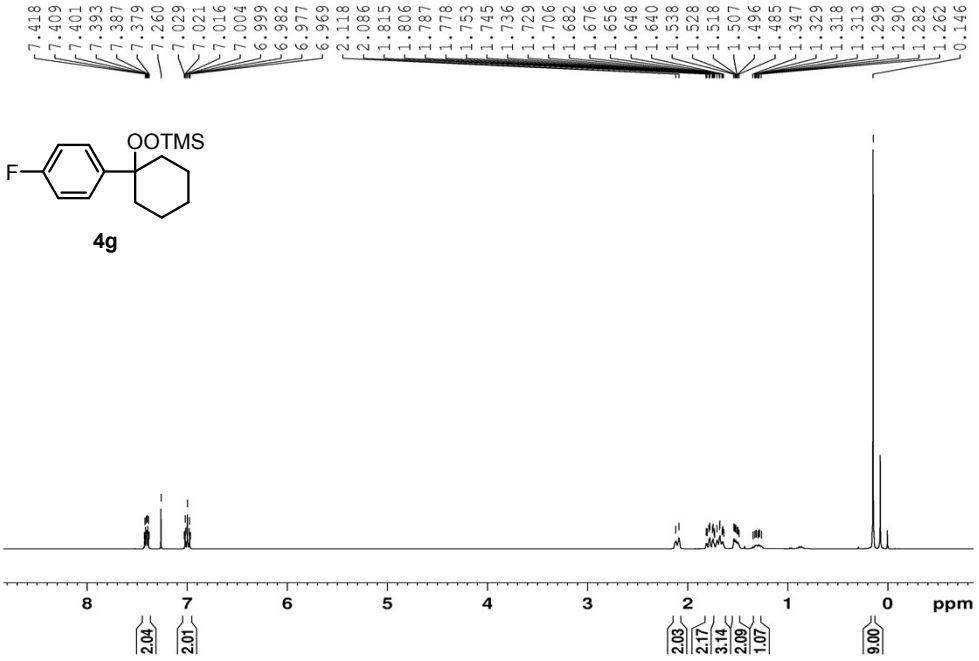
7.316
7.312
7.301
7.297
7.293
7.270
7.260
7.251
7.232
7.221
7.217
7.212
7.202
7.197
7.193
2.115
2.108
2.084
1.777
1.769
1.747
1.740
1.719
1.715
1.706
1.702
1.678
1.671
1.649
1.642
1.633
1.552
1.542
1.531
1.522
1.513
1.337
1.309
1.303
1.287
1.277
1.257
0.176
OOTMS
Cl
4f
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
1.28
1.00
2.09
5.25
2.18
1.23
9.21


148.130
133.811
129.115
126.772
126.451
124.092
83.734
77.317
77.000
76.683
34.422
25.485
21.980
-1.165
OOTMS
Cl
4f
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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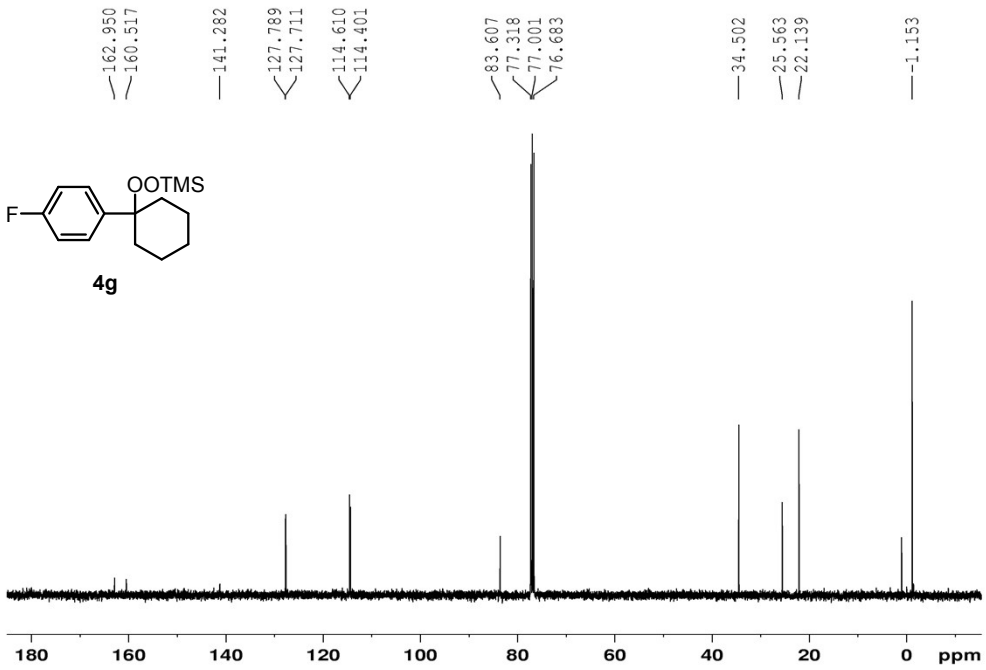
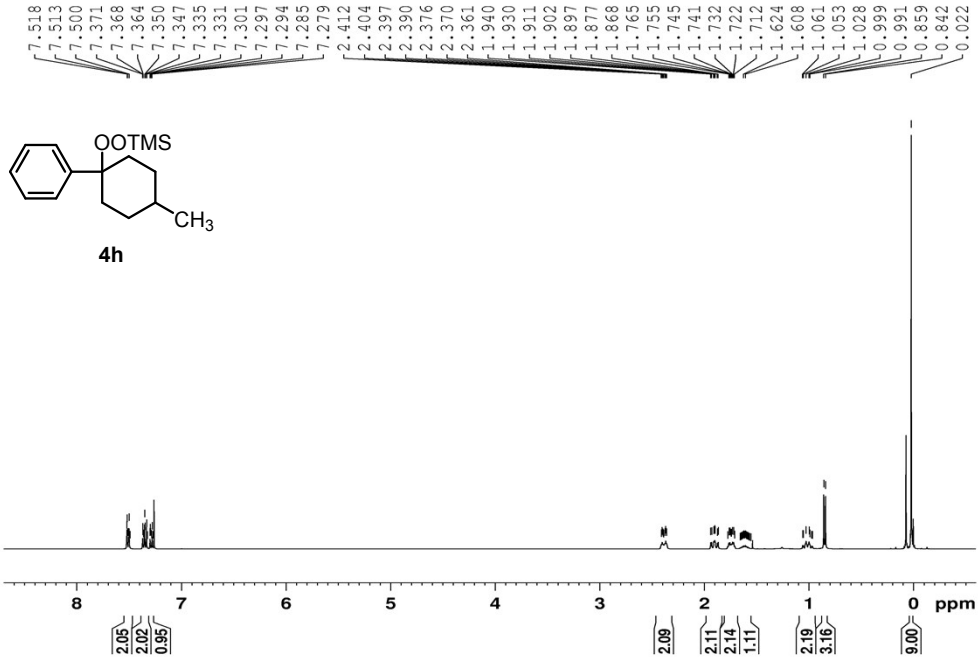
7.409
7.401
7.393
7.387
7.379
7.260
7.029
7.021
7.016
7.004
6.999
6.982
6.977
6.969
2.118
2.086
1.815
1.806
1.787
1.778
1.753
1.745
1.736
1.729
1.706
1.682
1.676
1.656
1.648
1.640
1.538
1.528
1.518
1.507
1.496
1.485
1.347
1.329
1.318
1.313
1.299
1.290
1.282
1.262
0.146
OOTMS
F
4g
8
7
6
5
4
3
2
1
0
ppm
2.04
2.01
2.03
2.17
3.14
2.09
1.07
9.00


162.950
160.517
141.282
127.789
127.711
114.610
114.401
83.607
77.318
77.001
76.683
34.502
25.563
22.139
-1.153
OOTMS
F
4g
180
160
140
120
100
80
60
40
20
0
ppm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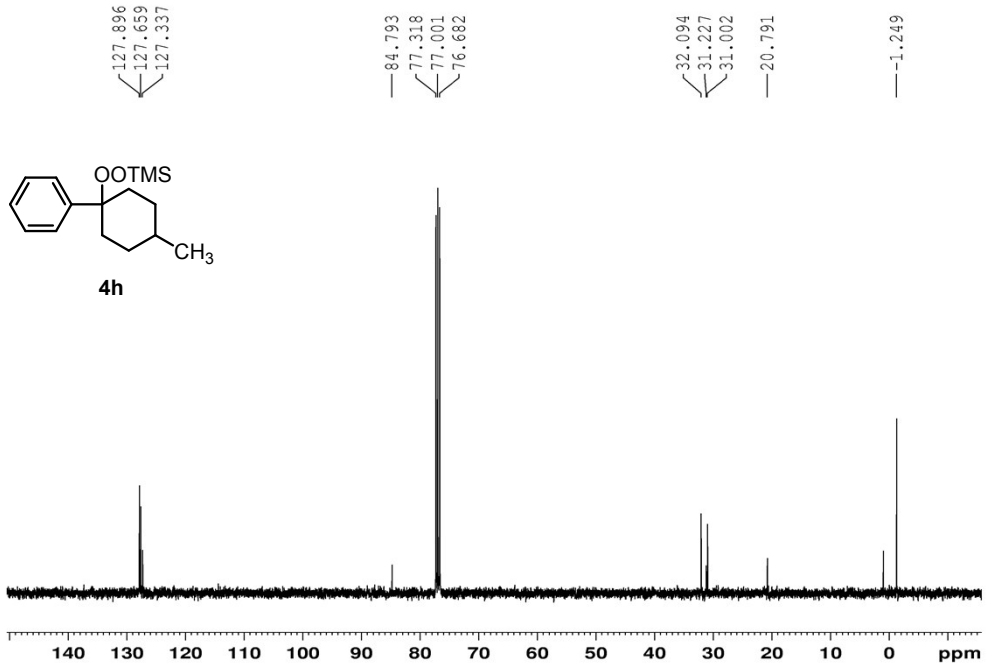
7.513
7.500
7.371
7.368
7.364
7.350
7.347
7.335
7.331
7.301
7.297
7.294
7.285
7.279
2.412
2.404
2.397
2.390
2.376
2.370
2.361
1.940
1.930
1.911
1.902
1.897
1.877
1.868
1.765
1.755
1.745
1.741
1.732
1.722
1.712
1.624
1.608
1.061
1.053
1.028
0.999
0.991
0.859
0.842
0.022
OOTMS
CH3
8
7
6
5
4
3
2
1
0
ppm
2.05
2.02
0.95
2.09
2.11
2.14
1.11
2.19
3.16
9.00

4h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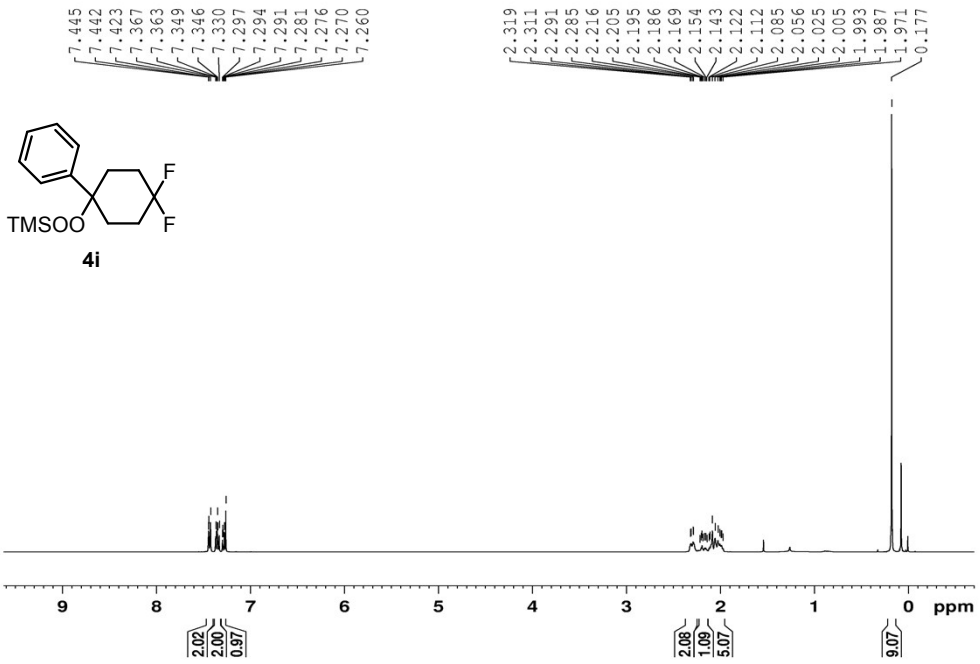
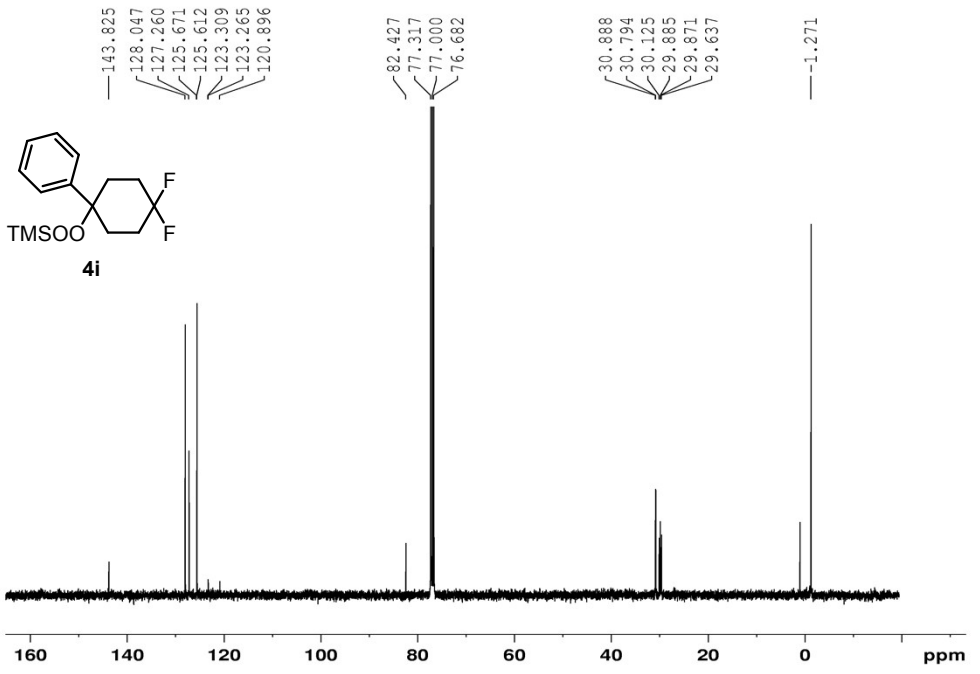
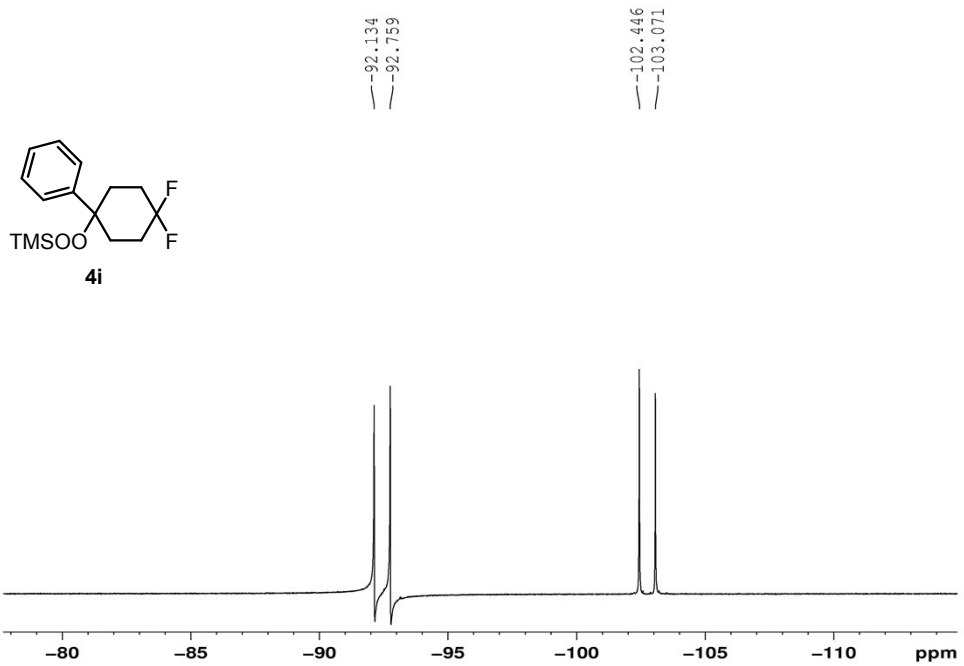
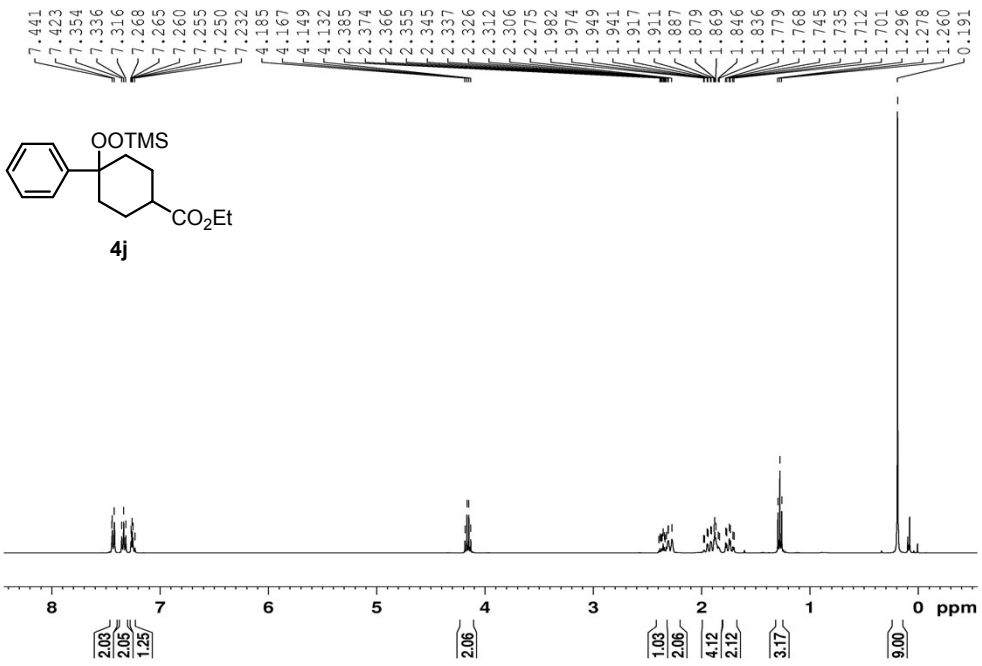
127.659
127.337
84.793
77.318
77.001
76.682
32.094
31.227
31.002
20.791
-1.249
OOTMS
CH3
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

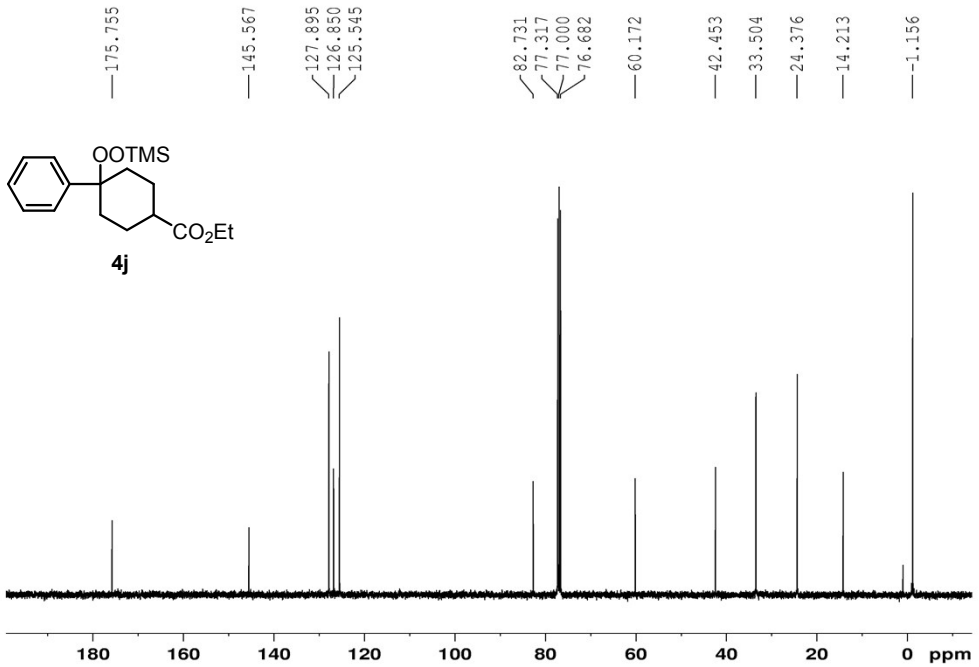
4h


7.445
7.442
7.423
7.367
7.363
7.349
7.346
7.330
7.297
7.294
7.291
7.281
7.276
7.270
7.260
2.319
2.311
2.291
2.285
2.216
2.205
2.195
2.186
2.169
2.154
2.143
2.122
2.112
2.085
2.056
2.025
2.005
1.993
1.987
1.971
0.177
F
F
TMSOO
4i
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.00
0.97
2.08
1.00
5.07
9.07


143.825
128.047
127.260
125.671
125.612
123.309
123.265
120.896
82.427
77.317
77.000
76.682
30.888
30.794
30.125
29.885
29.871
29.637
1.271
F
F
TMSOO
4i
160
140
120
100
80
60
40
20
0
ppm


-92.134
-92.759
-102.446
-103.071
F
F
TMSOO
4i
−80
−85
−90
−95
−100
−105
−110
ppm


7.441
7.423
7.354
7.336
7.316
7.268
7.265
7.260
7.255
7.250
7.232
4.185
4.167
4.149
4.132
2.385
2.374
2.366
2.355
2.345
2.337
2.326
2.312
2.306
2.275
1.982
1.974
1.949
1.941
1.917
1.911
1.887
1.879
1.869
1.846
1.836
1.779
1.768
1.745
1.735
1.712
1.701
1.296
1.278
1.260
0.191
OOTMS
CO2Et
4j
8
7
6
5
4
3
2
1
0 ppm
2.03
2.05
1.25
2.06
1.03
2.06
4.12
2.12
3.17
9.00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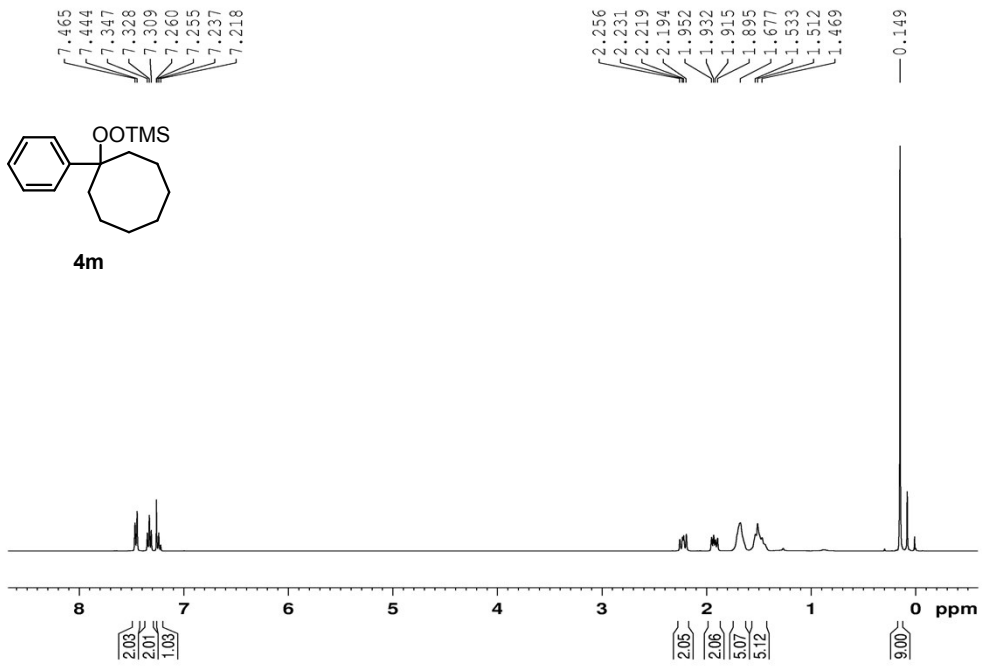
126.850
125.545
82.731
77.317
77.000
76.682
60.172
42.453
33.504
24.376
14.213
-1.156
OOTMS
CO2Et
4j
180
160
140
120
100
80
60
40
20
0 ppm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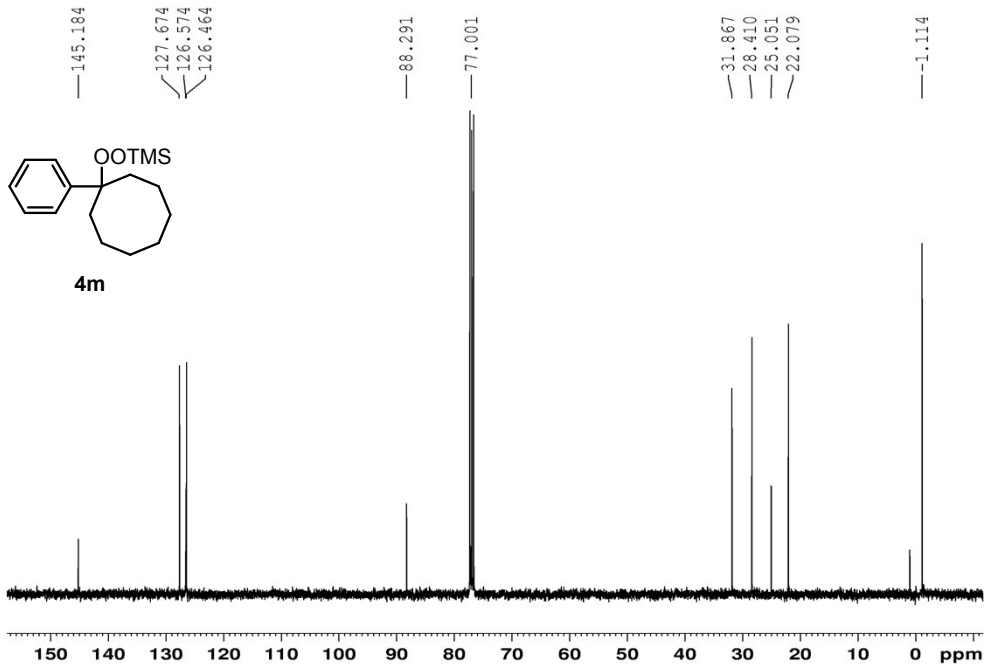
7.444
7.347
7.328
7.309
7.260
7.255
7.237
7.218
2.256
2.231
2.219
2.194
1.952
1.932
1.915
1.895
1.677
1.533
1.512
1.469
0.149
OOTMS
4m
8
7
6
5
4
3
2
1
0 ppm
2.03
2.01
1.03
2.05
2.06
5.07
5.12
9.00


145.184
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28.410
25.051
22.079
1.114
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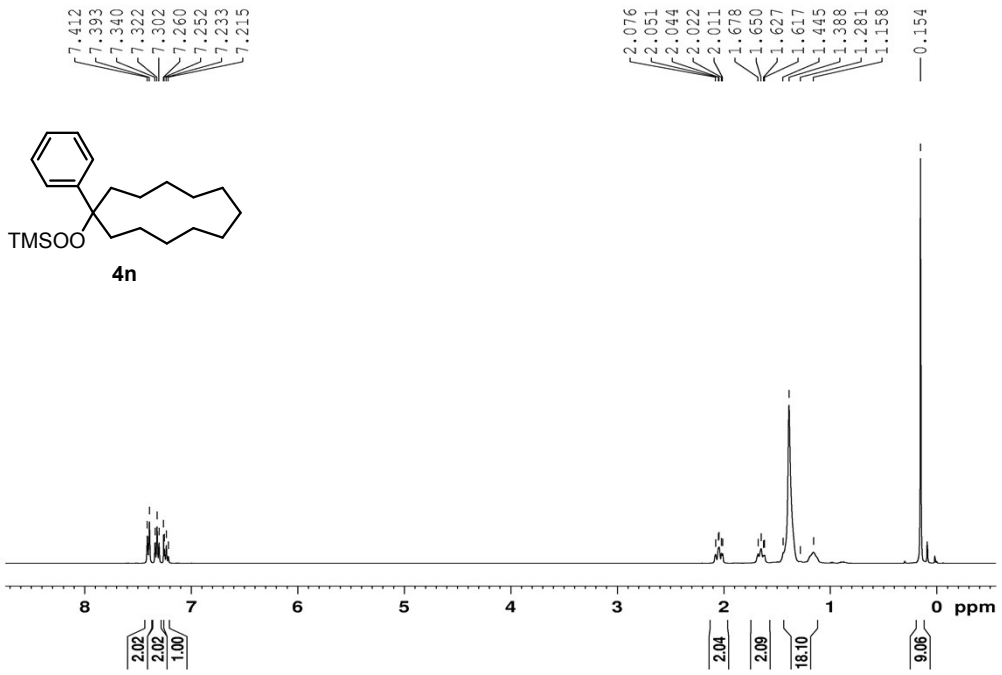
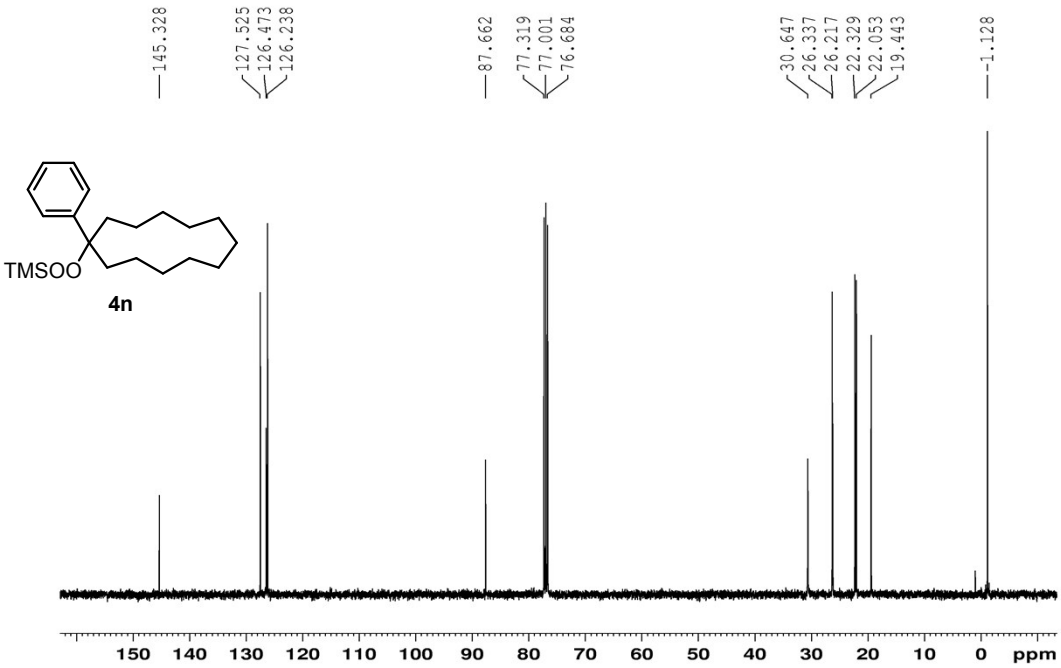
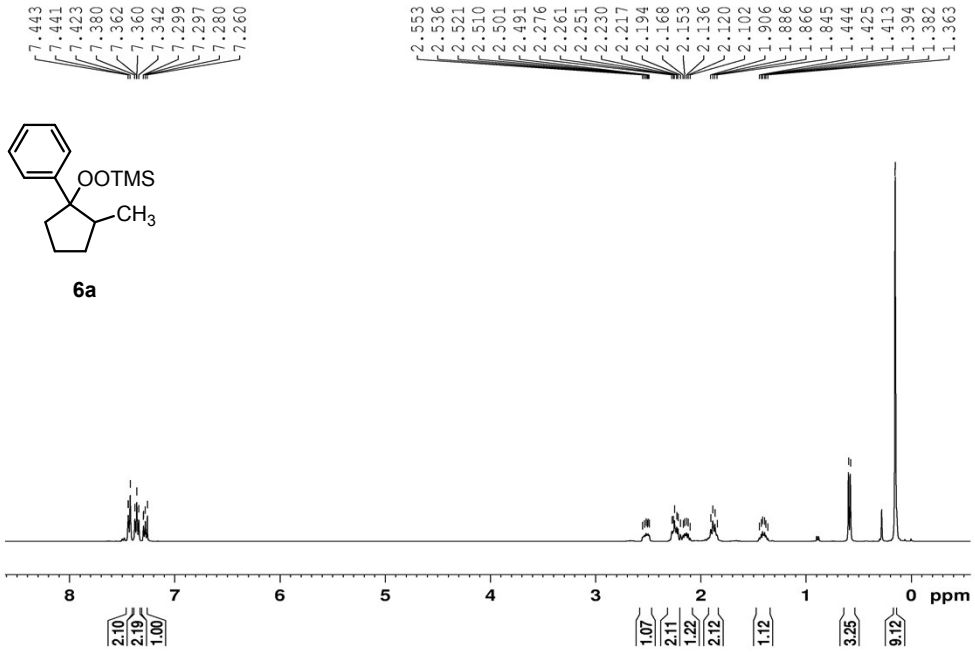
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4m

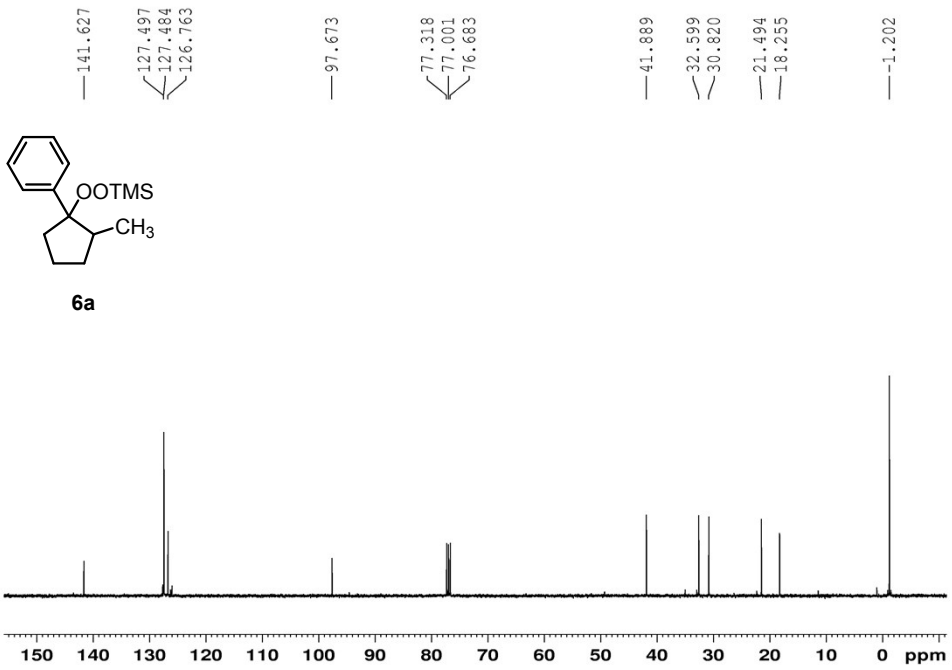

7.412
7.393
7.340
7.322
7.302
7.260
7.252
7.233
7.215
2.076
2.051
2.044
2.022
2.011
1.678
1.650
1.627
1.617
1.445
1.388
1.281
1.158
0.154
TMSOO
8 7 6 5 4 3 2 1 0 ppm
2.02
2.02
1.00
2.04
2.09
18.10
9.06

4n


145.328
127.525
126.473
126.238
87.662
77.319
77.001
76.684
30.647
26.337
26.217
22.329
22.053
19.443
-1.128
TMSOO
4n
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.443
7.441
7.423
7.380
7.362
7.360
7.342
7.299
7.297
7.280
7.260
2.553
2.536
2.521
2.510
2.501
2.491
2.276
2.261
2.251
2.230
2.217
2.194
2.168
2.153
2.136
2.120
2.102
1.906
1.886
1.866
1.845
1.444
1.425
1.413
1.394
1.382
1.363
OOTMS
CH3
8
7
6
5
4
3
2
1
0 ppm
2.10
2.19
1.00
1.07
2.11
1.22
2.12
1.12
3.25
9.12

6a


141.627
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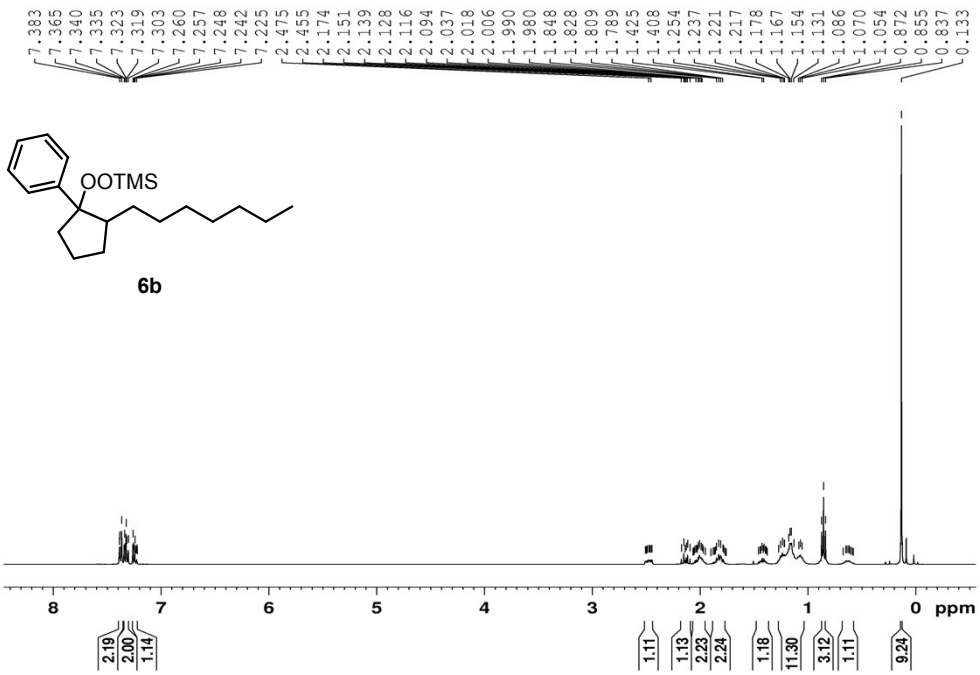
127.484
126.763
97.673
77.318
77.001
76.683
41.889
32.599
30.820
21.494
18.255
-1.202
OOTMS
CH3
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

6a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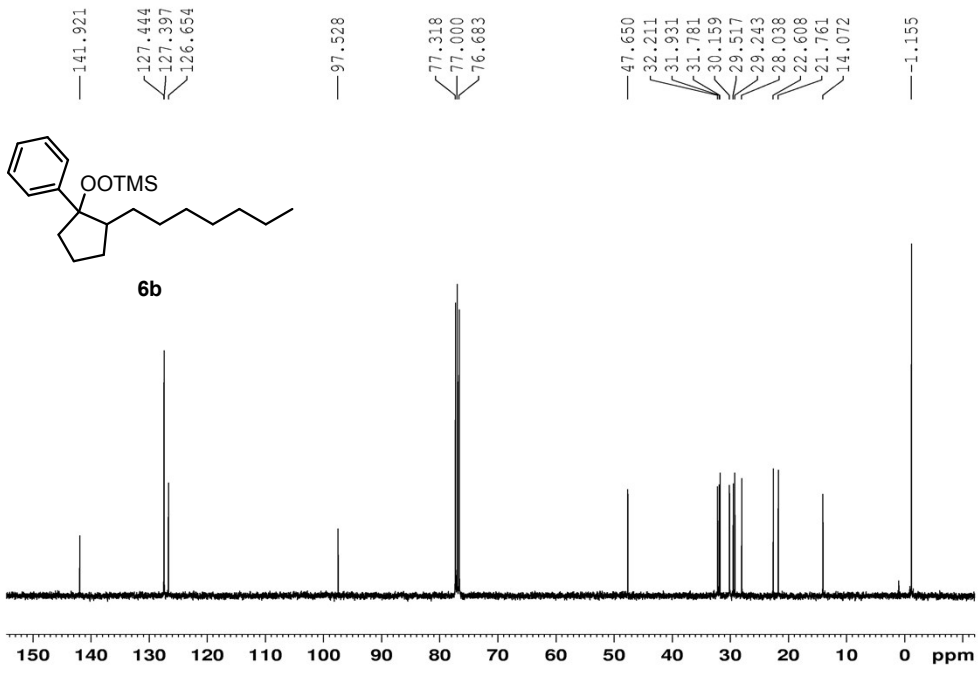
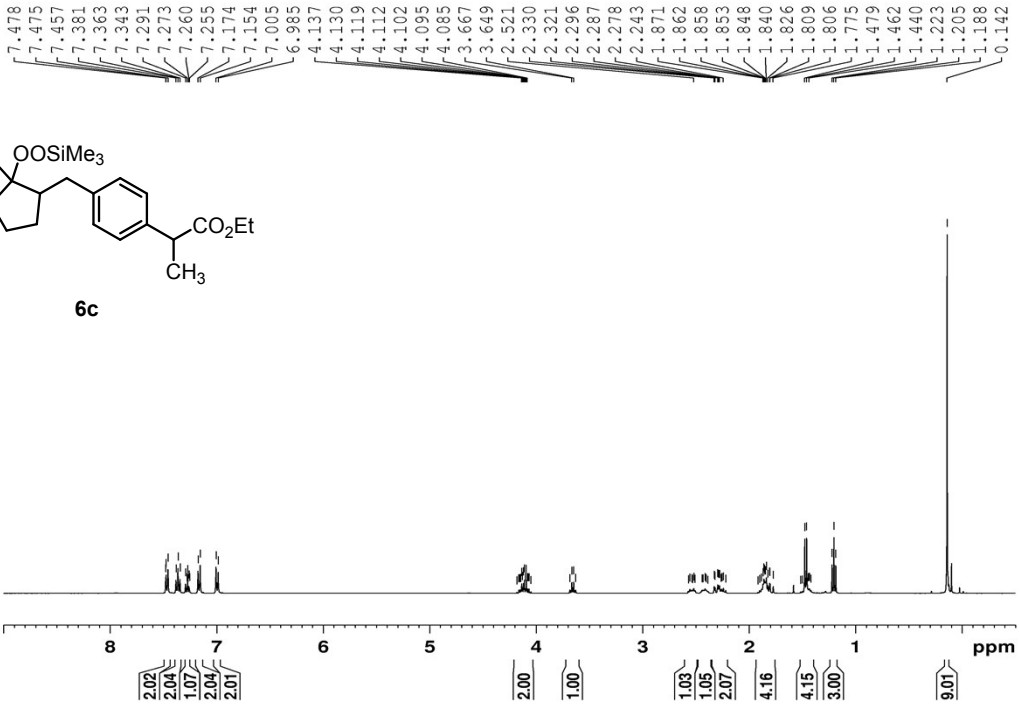
7.365
7.340
7.335
7.323
7.319
7.303
7.260
7.257
7.248
7.242
7.225
2.475
2.455
2.174
2.151
2.139
2.128
2.116
2.094
2.037
2.018
2.006
1.990
1.980
1.848
1.828
1.809
1.789
1.425
1.408
1.254
1.237
1.221
1.217
1.178
1.167
1.154
1.131
1.086
1.070
1.054
0.872
0.855
0.837
0.133
OOTMS
8
7
6
5
4
3
2
1
0 ppm
2.19
2.00
1.14
1.11
1.13
2.23
2.24
1.18
11.30
3.12
1.11
9.24

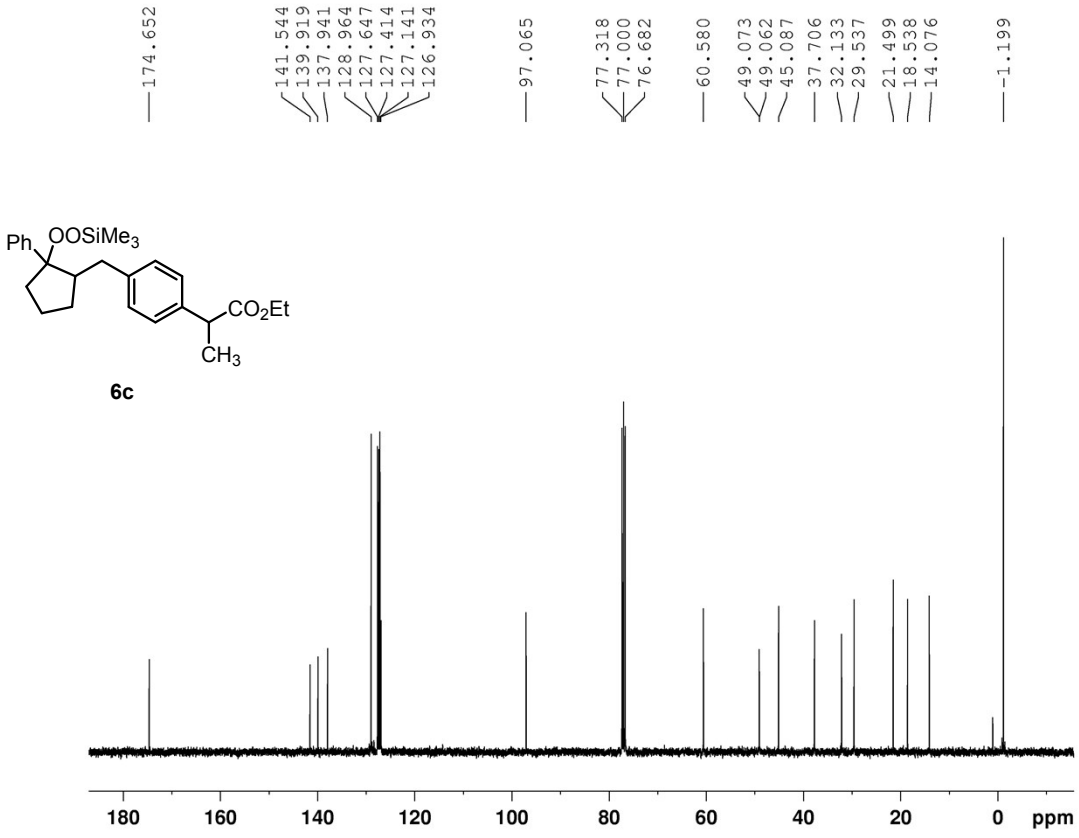
6b


141.921
127.444
127.397
126.654
97.528
77.318
77.000
76.683
47.650
32.211
31.931
31.781
30.159
29.517
29.243
28.038
22.608
21.761
14.072
-1.155
OOTMS
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

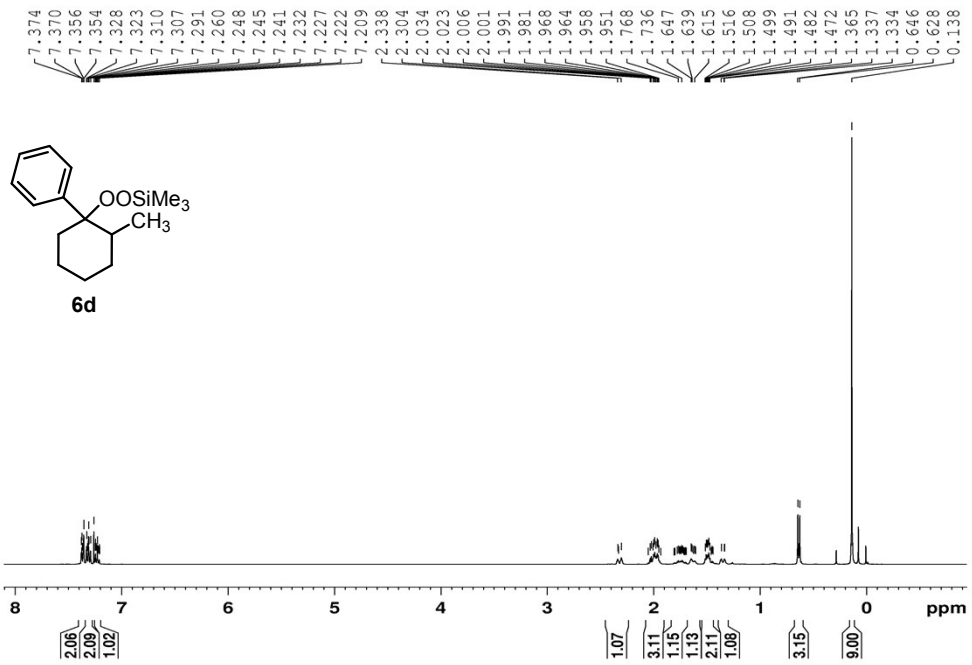
6b

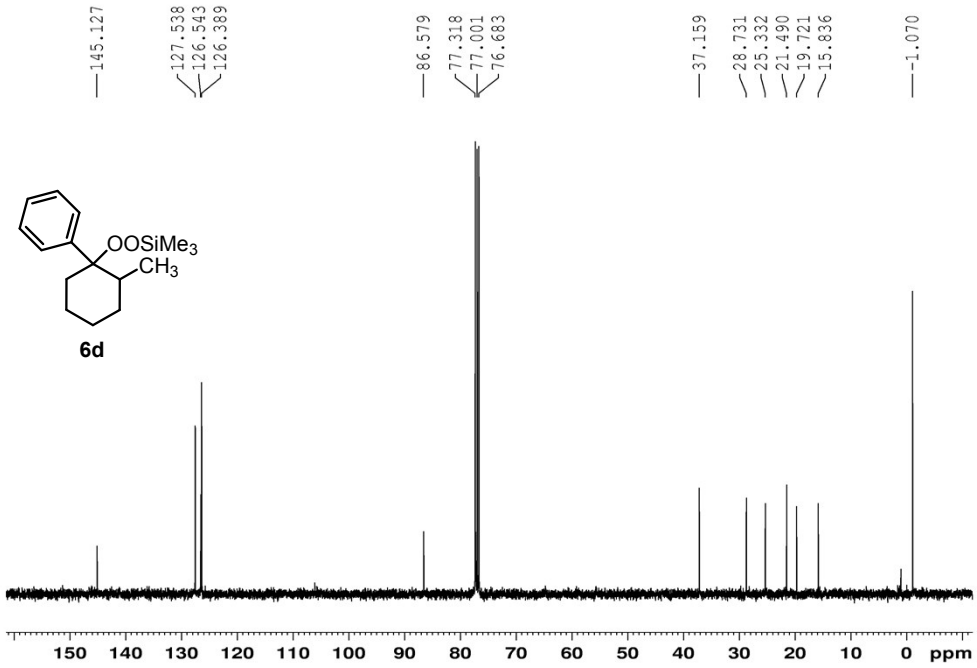

7.478
7.475
7.457
7.381
7.363
7.343
7.291
7.273
7.260
7.255
7.174
7.154
7.005
6.985
4.137
4.130
4.119
4.112
4.102
4.095
4.085
3.667
3.649
2.521
2.330
2.321
2.296
2.287
2.278
2.243
1.871
1.862
1.858
1.853
1.848
1.840
1.826
1.809
1.806
1.775
1.479
1.462
1.440
1.223
1.205
1.188
0.142
Ph
OOSiMe3
CO2Et
CH3
8
7
6
5
4
3
2
1
ppm
2.02
2.04
1.07
2.04
2.01
2.00
1.00
1.03
1.05
2.07
4.16
4.15
3.00
9.01

6c

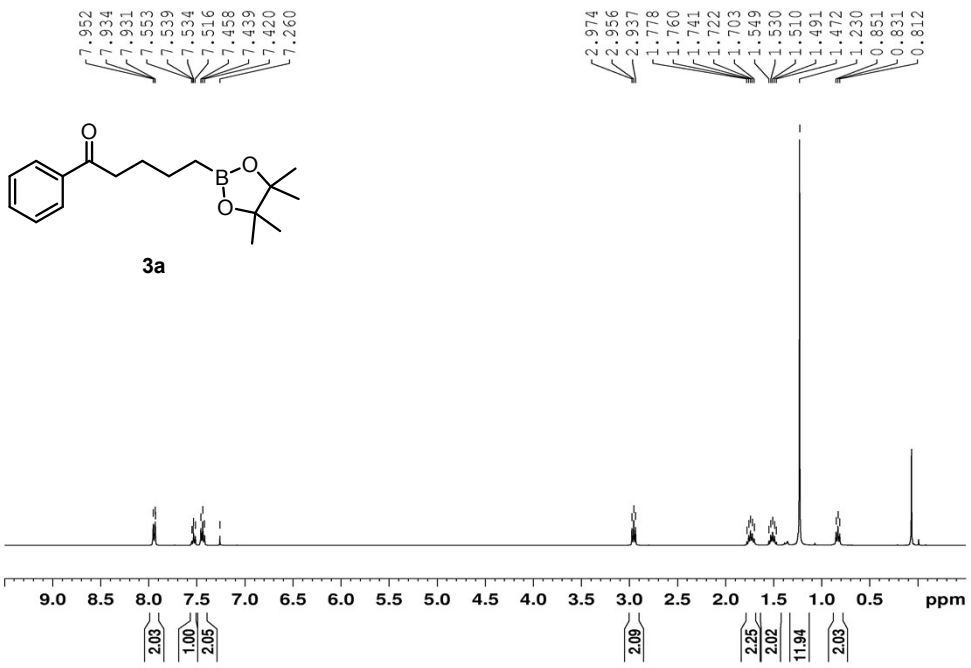

174.652
141.544
139.919
137.941
128.964
127.647
127.414
127.141
126.934
97.065
77.318
77.000
76.682
60.580
49.073
49.062
45.087
37.706
32.133
29.537
21.499
18.538
14.076
-1.199
Ph
OOSiMe3
CO2Et
CH3
180
160
140
120
100
80
60
40
20
0
ppm

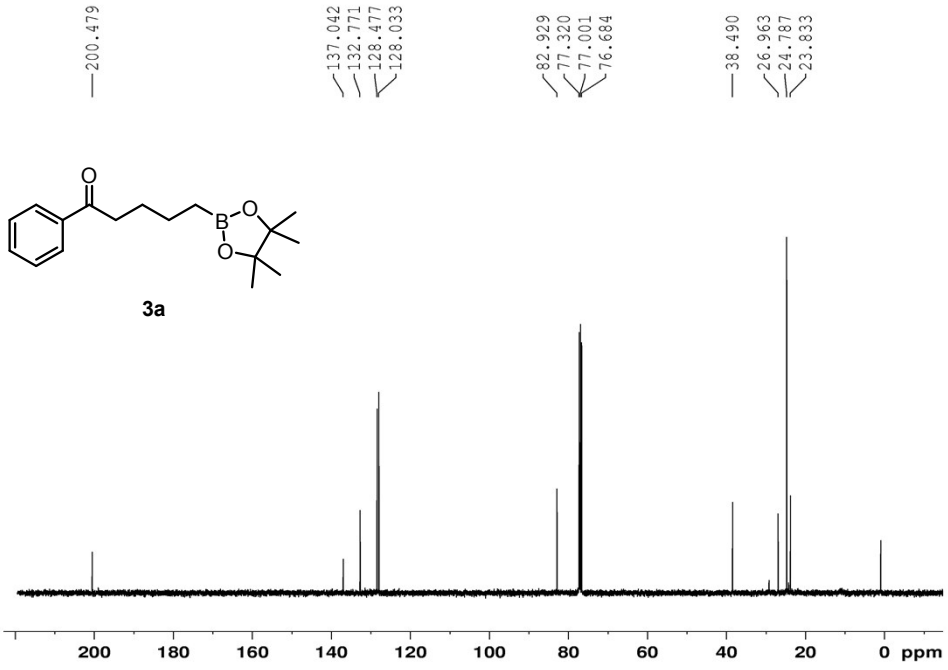
6c

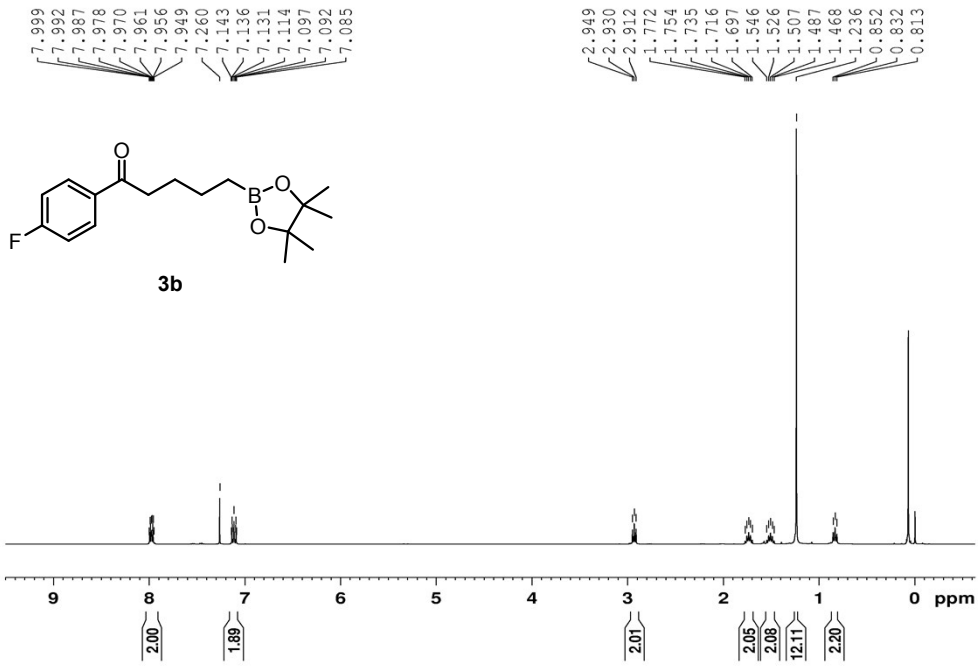

7.374
7.370
7.356
7.354
7.328
7.323
7.310
7.307
7.291
7.260
7.248
7.245
7.241
7.232
7.227
7.222
7.209
2.338
2.304
2.034
2.023
2.006
2.001
1.991
1.981
1.968
1.964
1.958
1.951
1.768
1.736
1.647
1.639
1.615
1.516
1.508
1.499
1.491
1.482
1.472
1.365
1.337
1.334
0.646
0.628
0.138
OOSiMe3
CH3
6d
8
7
6
5
4
3
2
1
0
ppm
2.06
2.09
1.02
1.07
3.11
1.15
1.13
2.11
1.08
3.15
9.00

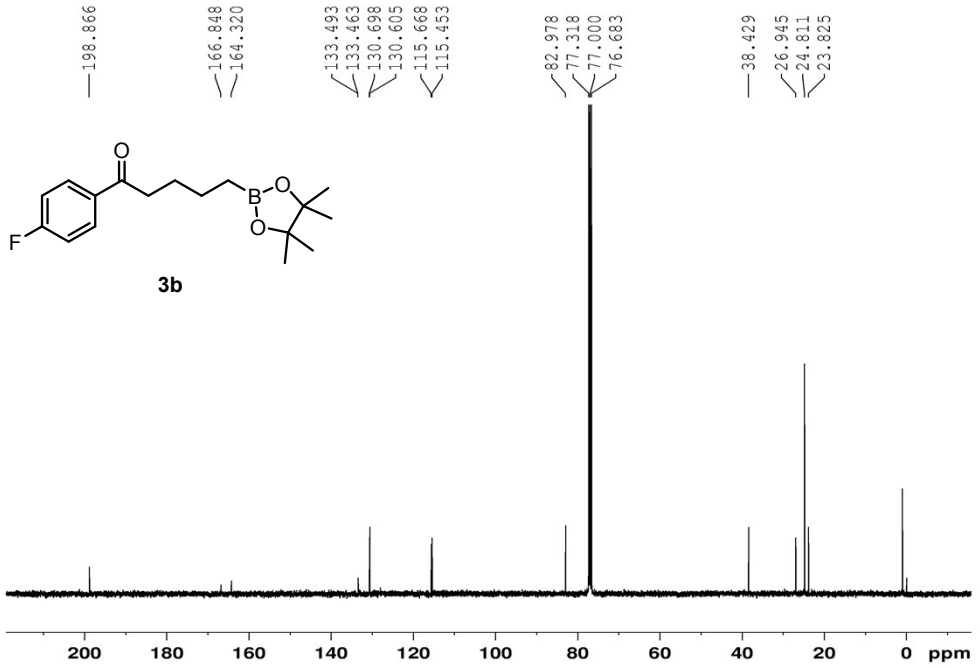

145.127
127.538
126.543
126.389
86.579
77.318
77.001
76.683
37.159
28.731
25.332
21.490
19.721
15.836
-1.070
OOSiMe3
CH3
6d
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

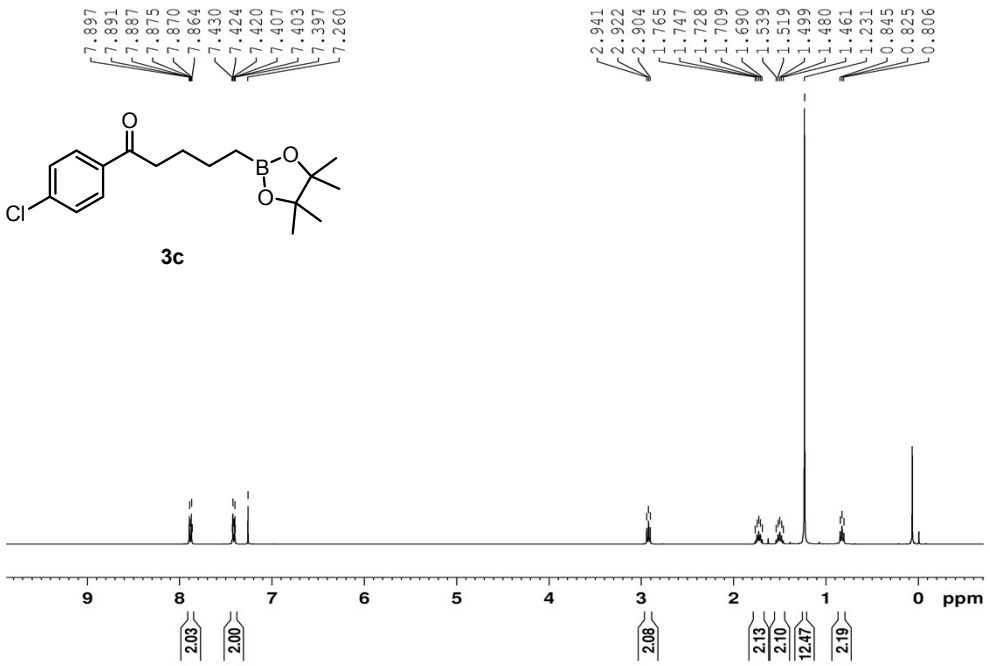
^{1}H NMR and ^{13}C NMR Spectra of the Products **3**, **5** and **7**

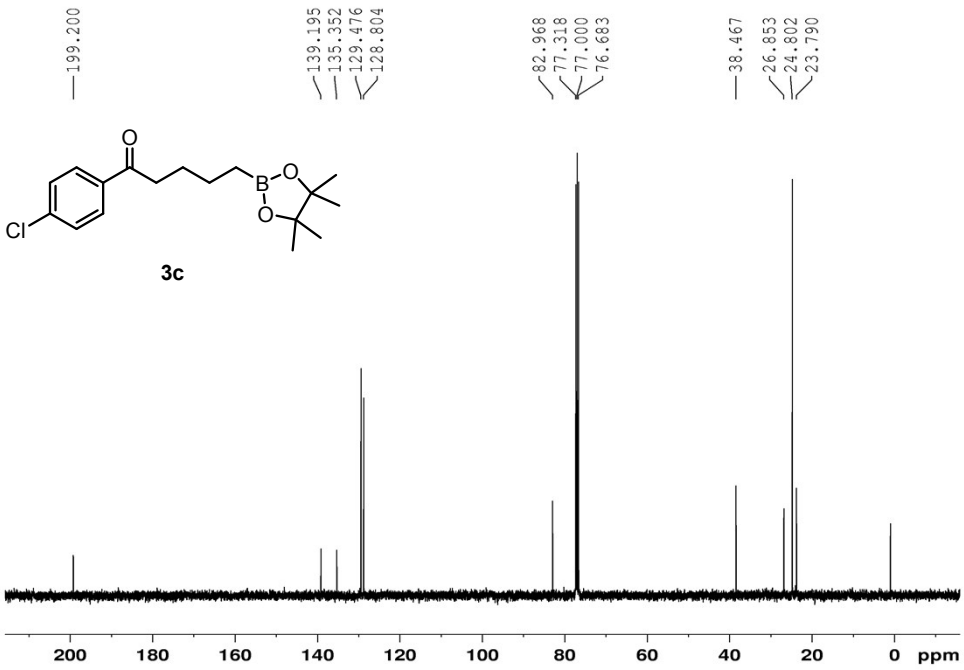


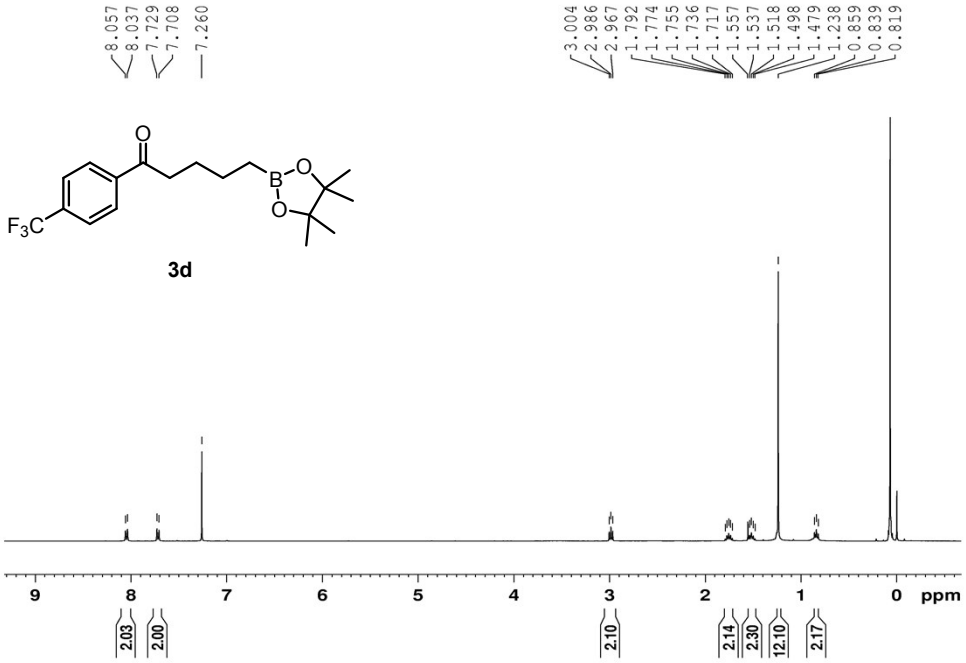



7.999
7.992
7.987
7.978
7.970
7.961
7.956
7.949
7.260
7.143
7.136
7.131
7.114
7.097
7.092
7.085
2.949
2.930
2.912
1.772
1.754
1.735
1.716
1.697
1.546
1.526
1.507
1.487
1.468
1.236
0.852
0.832
0.813
O
B
O
O
F
3b
9
8
7
6
5
4
3
2
1
0 ppm
2.00
1.89
2.01
2.05
2.08
12.11
2.20

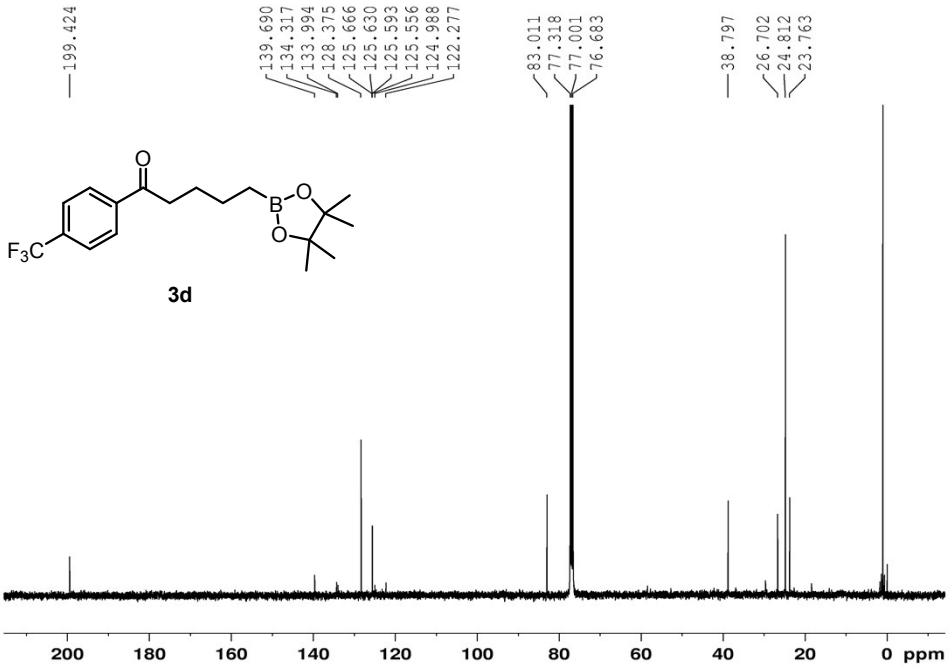

198.866
166.848
164.320
133.493
133.463
130.698
130.605
115.668
115.453
82.978
77.318
77.000
76.683
38.429
26.945
24.811
23.825
O
B
O
O
F
3b
200
180
160
140
120
100
80
60
40
20
0 ppm


7.897
7.891
7.887
7.875
7.870
7.864
7.430
7.424
7.420
7.407
7.403
7.397
7.260
2.941
2.922
2.904
1.765
1.747
1.728
1.709
1.690
1.539
1.519
1.499
1.480
1.461
1.231
0.845
0.825
0.806
O
B
O
O
Cl
3c
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.00
2.08
2.13
2.10
12.47
2.19


199.200
139.195
135.352
129.476
128.804
82.968
77.318
77.000
76.683
38.467
26.853
24.802
23.790
O
B
O
O
Cl
3c
200
180
160
140
120
100
80
60
40
20
0
ppm


8.057
8.037
7.729
7.708
7.260
3.004
2.986
2.967
1.792
1.774
1.755
1.736
1.717
1.557
1.537
1.518
1.498
1.479
1.238
0.859
0.839
0.819
O
B
O
O
F3C
9
8
7
6
5
4
3
2
1
0 ppm
2.03
2.00
2.10
2.14
2.30
12.10
2.17

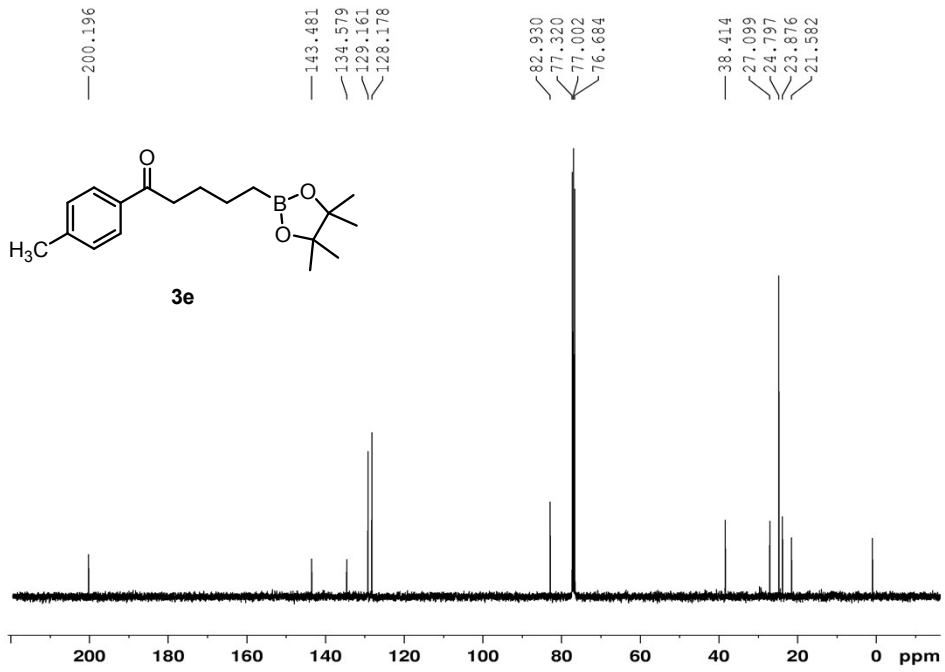
3d


199.424
139.690
134.317
133.994
128.375
125.666
125.630
125.593
125.556
124.988
122.277
83.011
77.318
77.001
76.683
38.797
26.702
24.812
23.763
O
B
O
O
F3C
200
180
160
140
120
100
80
60
40
20
0 ppm

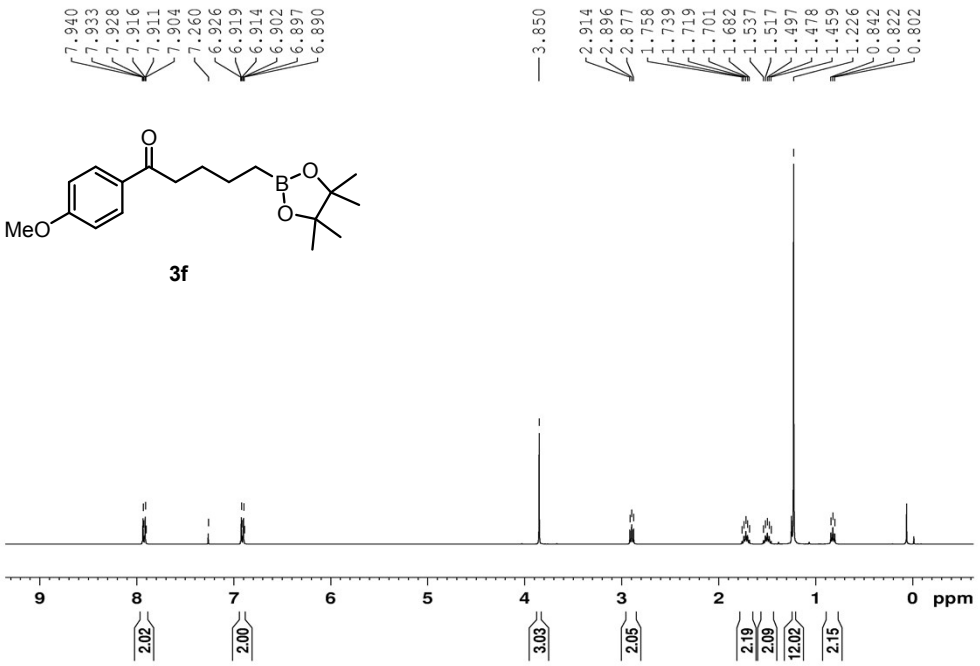
3d

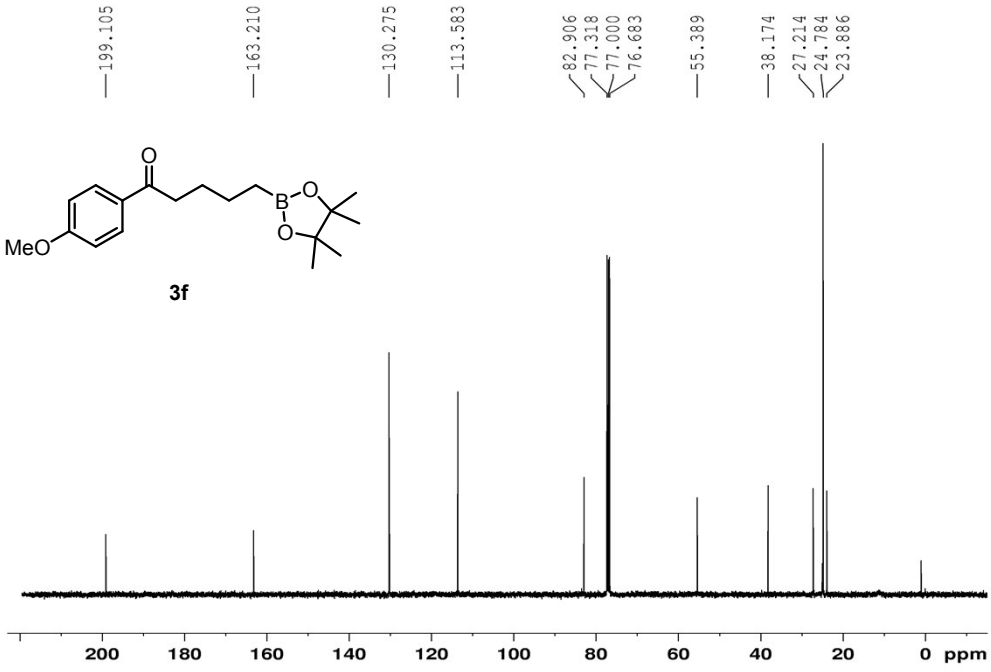

7.856
7.835
7.260
7.247
7.227
2.947
2.928
2.909
2.398
1.768
1.749
1.730
1.711
1.692
1.544
1.524
1.505
1.485
1.466
1.233
0.849
0.829
0.810
O
B
O
O
H3C
8
7
6
5
4
3
2
1
0 ppm
2.00
2.04
2.06
3.06
2.09
2.01
12.11
2.17

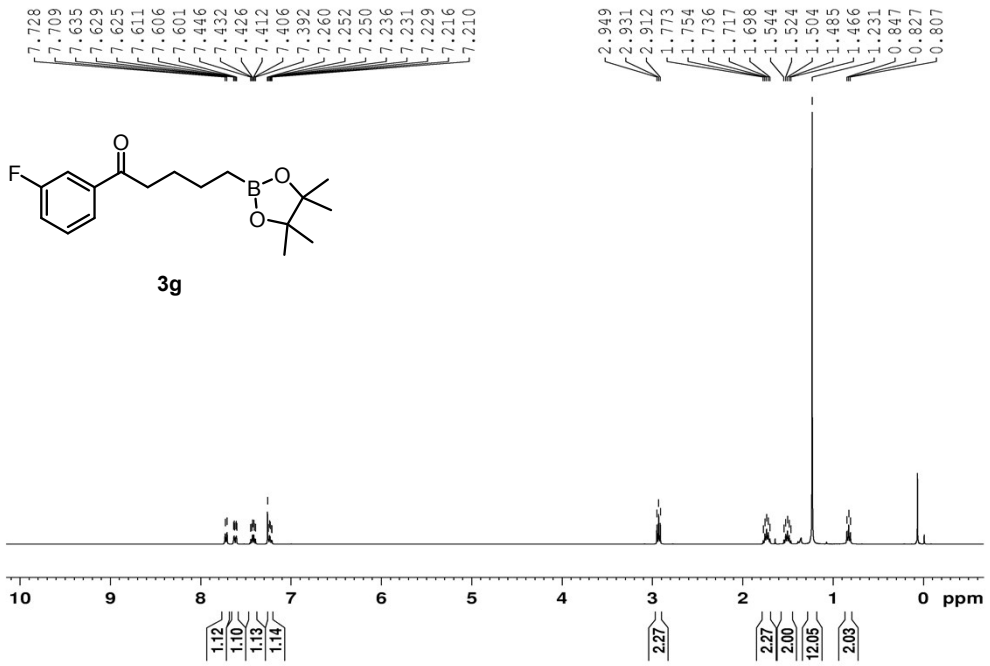
3e


200.196
143.481
134.579
129.161
128.178
82.930
77.320
77.002
76.684
38.414
27.099
24.797
23.876
21.582
O
B
O
O
H3C
200
180
160
140
120
100
80
60
40
20
0 ppm

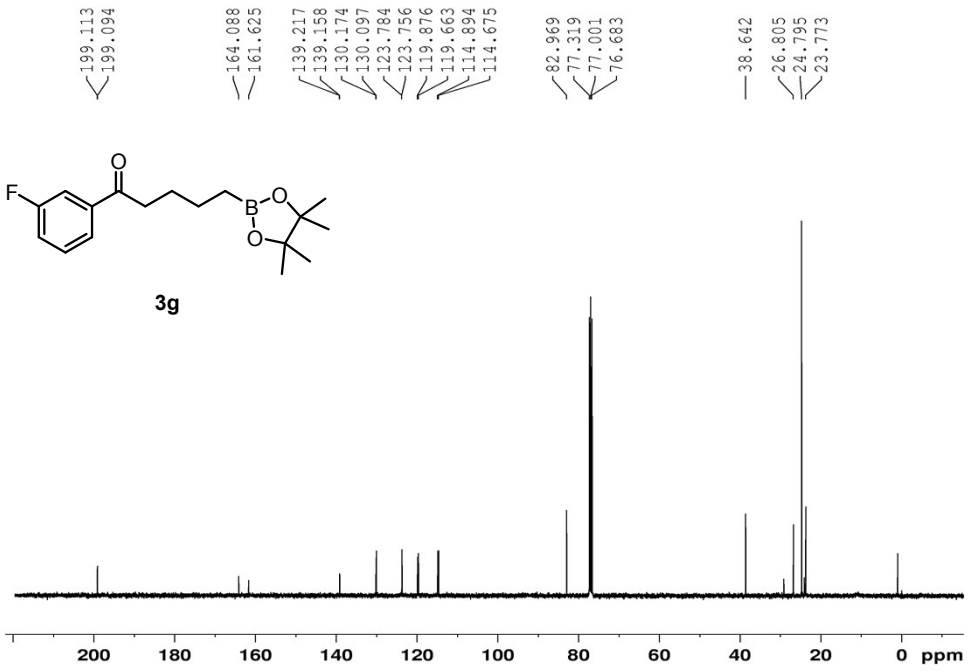
3e


7.940
7.933
7.928
7.916
7.911
7.904
7.260
6.926
6.919
6.914
6.902
6.897
6.890
3.850
2.914
2.896
2.877
1.758
1.739
1.719
1.701
1.682
1.537
1.517
1.497
1.478
1.459
1.226
0.842
0.822
0.802
O
B
O
O
MeO
3f
9
8
7
6
5
4
3
2
1
0 ppm
2.02
2.00
3.03
2.05
2.19
2.09
12.02
2.15

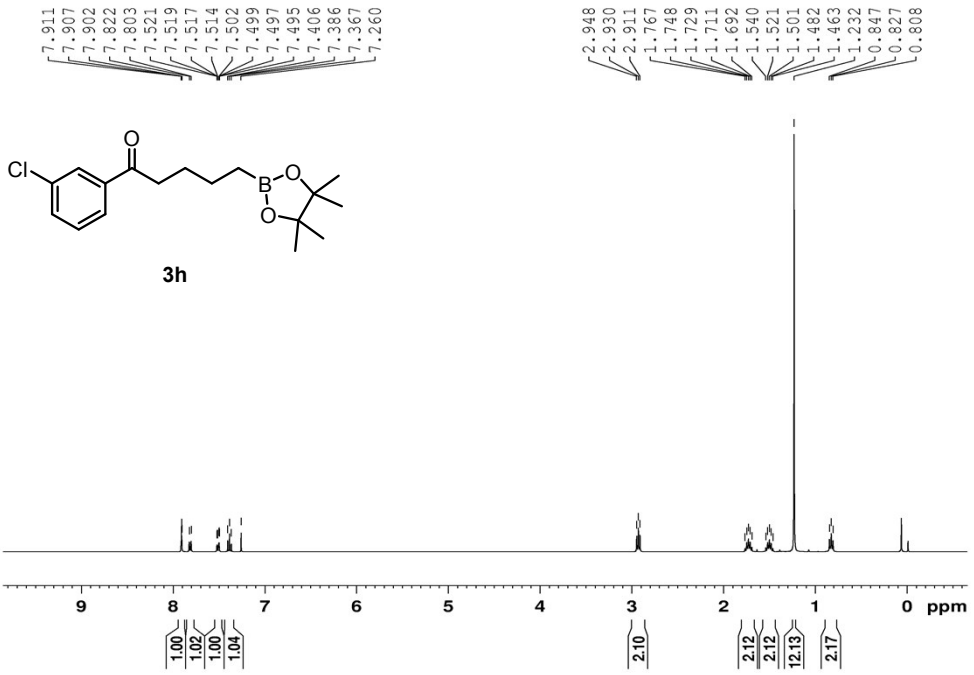

199.105
163.210
130.275
113.583
82.906
77.318
77.000
76.683
55.389
38.174
27.214
24.784
23.886
O
B
O
O
MeO
3f
200
180
160
140
120
100
80
60
40
20
0 ppm


7.728
7.709
7.635
7.629
7.625
7.611
7.606
7.601
7.446
7.432
7.426
7.412
7.406
7.392
7.260
7.252
7.250
7.236
7.231
7.229
7.216
7.210
2.949
2.931
2.912
1.773
1.754
1.736
1.717
1.698
1.544
1.524
1.504
1.485
1.466
1.231
0.847
0.827
0.807
O
F
B
O
O
10
9
8
7
6
5
4
3
2
1
0 ppm
1.12
1.10
1.13
1.14
2.27
2.27
2.00
12.05
2.03

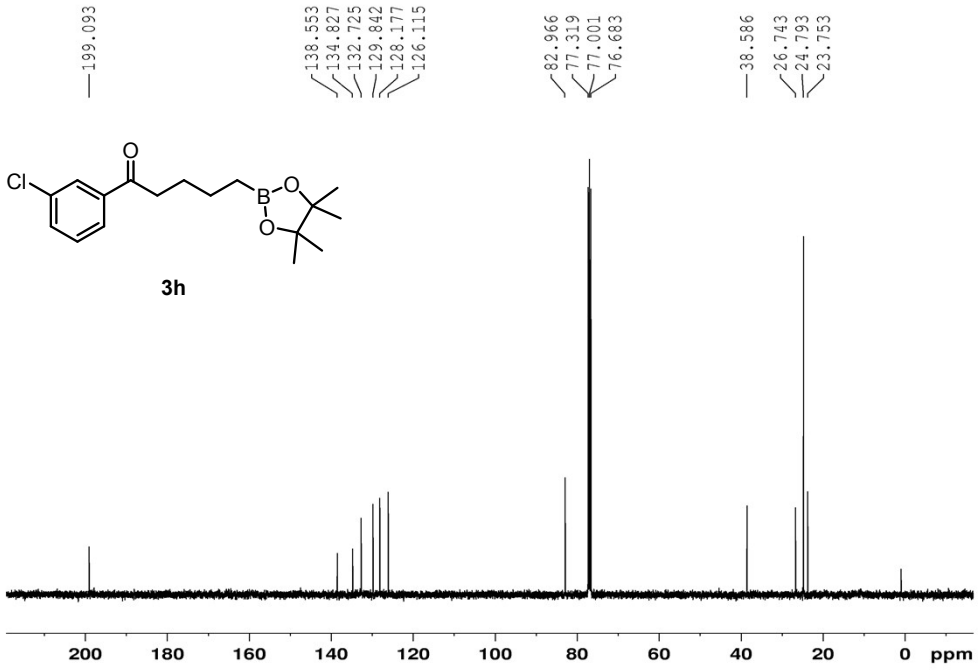
3g


199.113
199.094
164.088
161.625
139.217
139.158
130.174
130.097
123.784
123.756
119.876
119.663
114.894
114.675
82.969
77.319
77.001
76.683
38.642
26.805
24.795
23.773
O
F
B
O
O
200
180
160
140
120
100
80
60
40
20
0 ppm

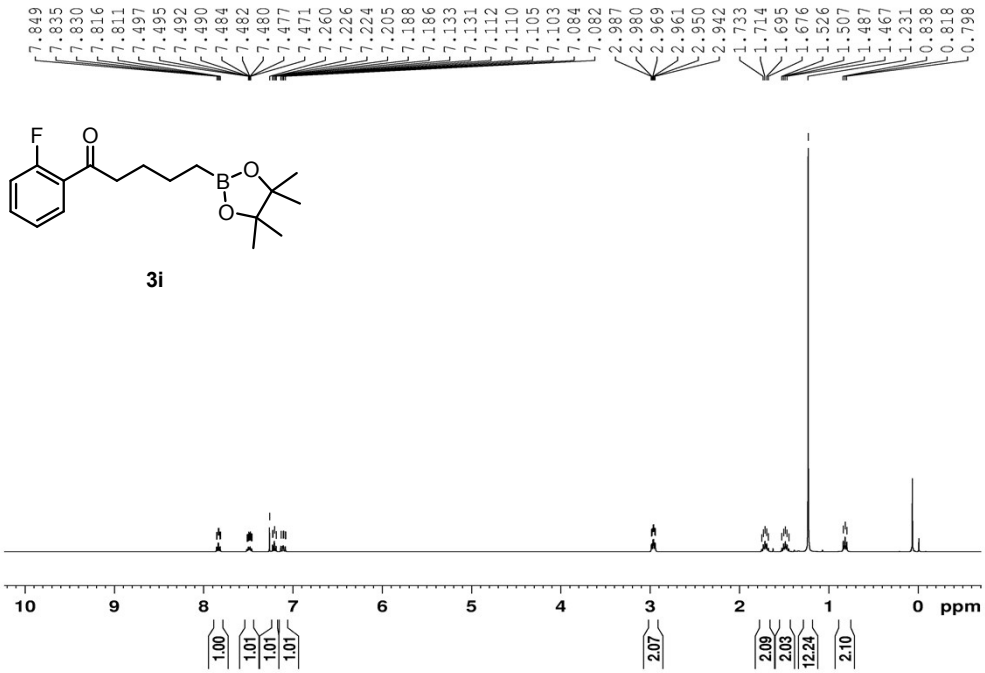
3g


7.911
7.907
7.902
7.822
7.803
7.521
7.519
7.517
7.514
7.502
7.499
7.497
7.495
7.406
7.386
7.367
7.260
2.948
2.930
2.911
1.767
1.748
1.729
1.711
1.692
1.540
1.521
1.501
1.482
1.463
1.232
0.847
0.827
0.808
Cl
O
B
O
O
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.02
1.00
1.04
2.10
2.12
2.12
12.13
2.17

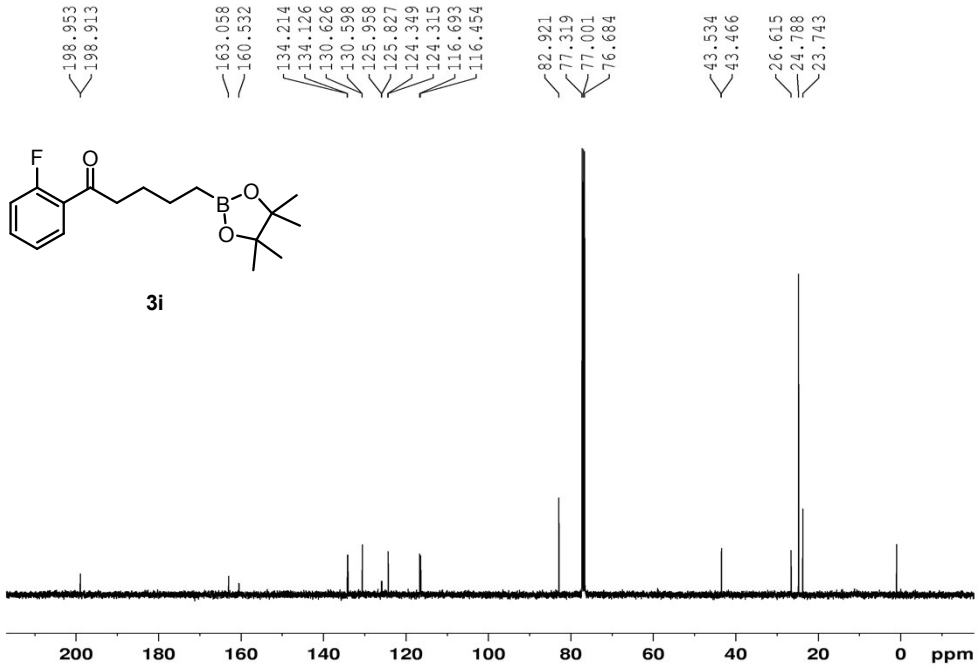
3h


199.093
138.553
134.827
132.725
129.842
128.177
126.115
82.966
77.319
77.001
76.683
38.586
26.743
24.793
23.753
Cl
O
B
O
O
200
180
160
140
120
100
80
60
40
20
0 ppm

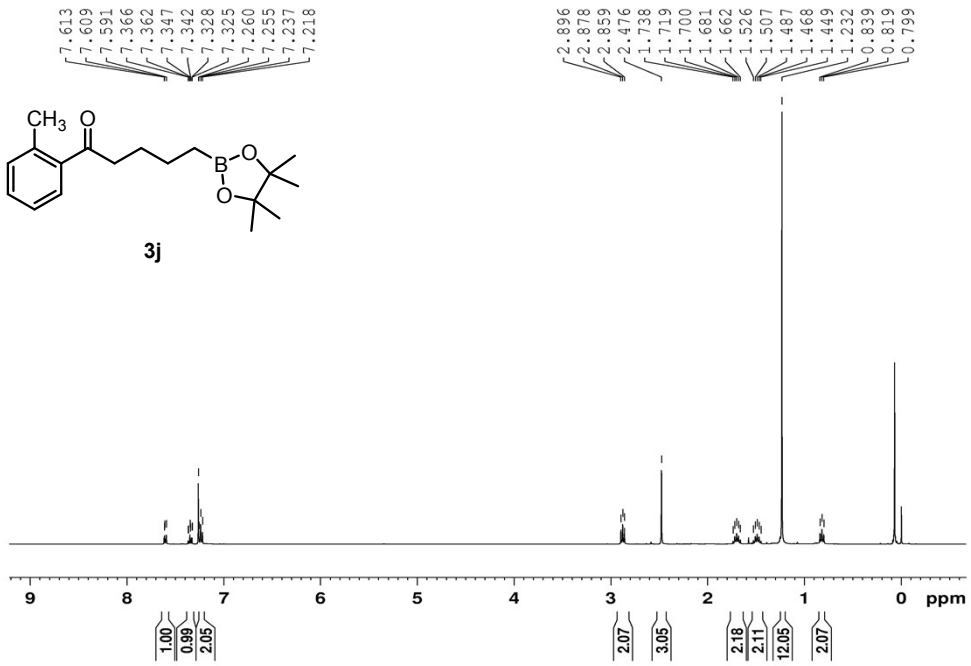
3h

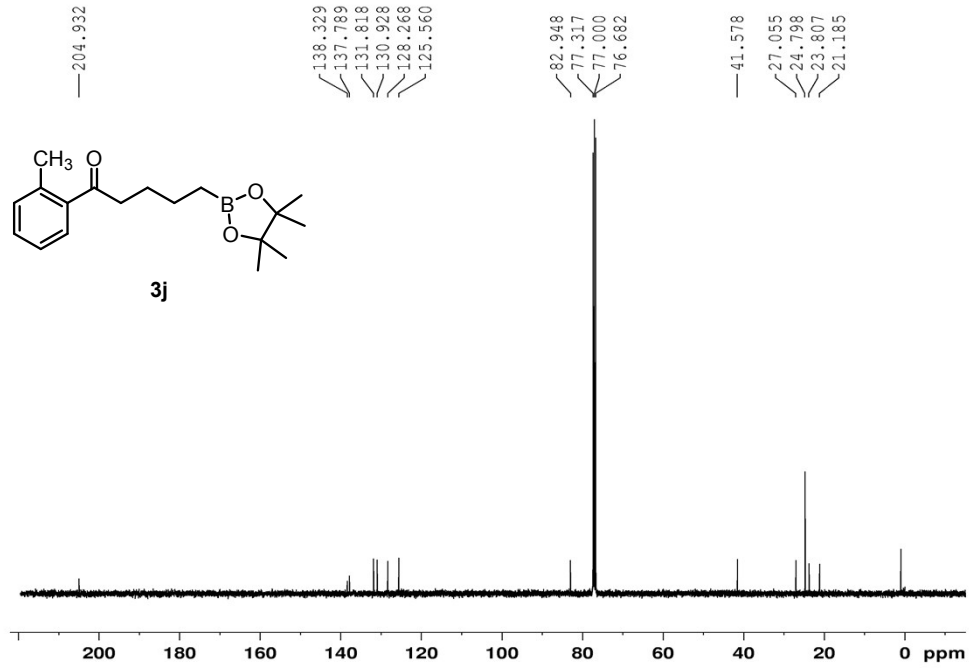

7.849
7.835
7.830
7.816
7.811
7.497
7.495
7.492
7.490
7.484
7.482
7.480
7.477
7.471
7.260
7.226
7.224
7.205
7.188
7.186
7.133
7.131
7.112
7.110
7.105
7.103
7.084
7.082
2.987
2.980
2.969
2.961
2.950
2.942
1.733
1.714
1.695
1.676
1.526
1.507
1.487
1.467
1.231
0.838
0.818
0.798
F
O
B
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
1.01
1.01
2.07
2.09
2.03
12.24
2.10

3i


198.953
198.913
163.058
160.532
134.214
134.126
130.626
130.598
125.958
125.827
124.349
124.315
116.693
116.454
82.921
77.319
77.001
76.684
43.534
43.466
26.615
24.788
23.743
F
O
B
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm

3i


7.613
7.609
7.591
7.366
7.362
7.347
7.342
7.328
7.325
7.260
7.255
7.237
7.218
2.896
2.878
2.859
2.476
1.738
1.719
1.700
1.681
1.662
1.526
1.507
1.487
1.468
1.449
1.232
0.839
0.819
0.799
CH3
O
B
O
O
3j
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.99
2.05
2.07
3.05
2.18
2.11
12.05
2.07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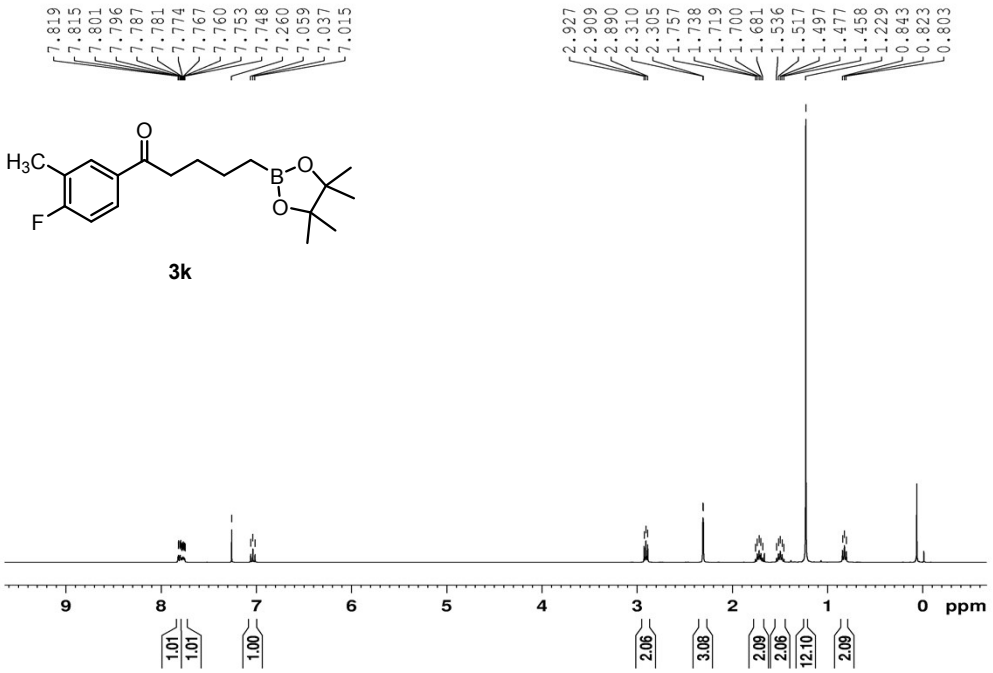
137.789
131.818
130.928
128.268
125.560
82.948
77.317
77.000
76.682
41.578
27.055
24.798
23.807
21.185
CH3
O
B
O
O
3j
200
180
160
140
120
100
80
60
40
20
0
ppm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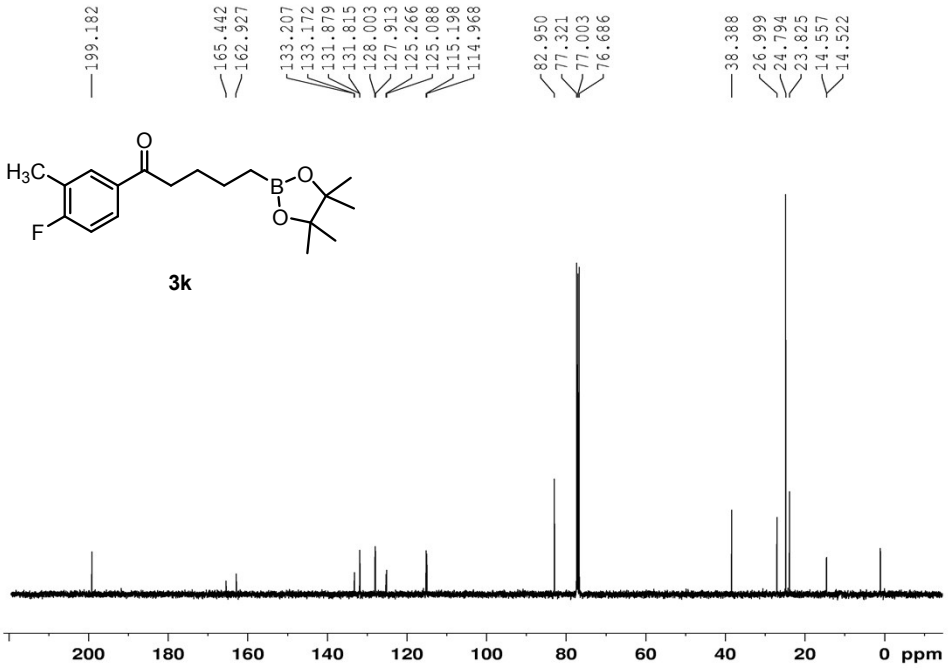
7.815
7.801
7.796
7.787
7.781
7.774
7.767
7.760
7.753
7.748
7.260
7.059
7.037
7.015
2.927
2.909
2.890
2.310
2.305
1.757
1.738
1.719
1.700
1.681
1.536
1.517
1.497
1.477
1.458
1.229
0.843
0.823
0.803
O
H3C
B
O
O
F
9
8
7
6
5
4
3
2
1
0 ppm
1.01
1.01
1.00
2.06
3.08
2.09
2.06
12.10
2.09

3k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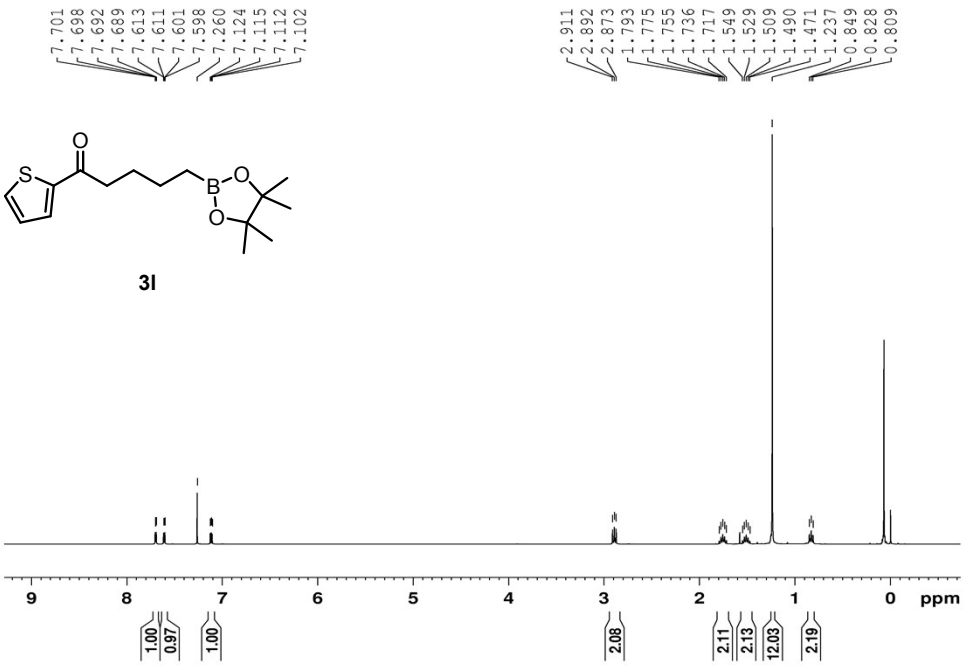
162.927
133.207
133.172
131.879
131.815
128.003
127.913
125.266
125.088
115.198
114.968
82.950
77.321
77.003
76.686
38.388
26.999
24.794
23.825
14.557
14.522
O
H3C
B
O
O
F
200
180
160
140
120
100
80
60
40
20
0 ppm

3k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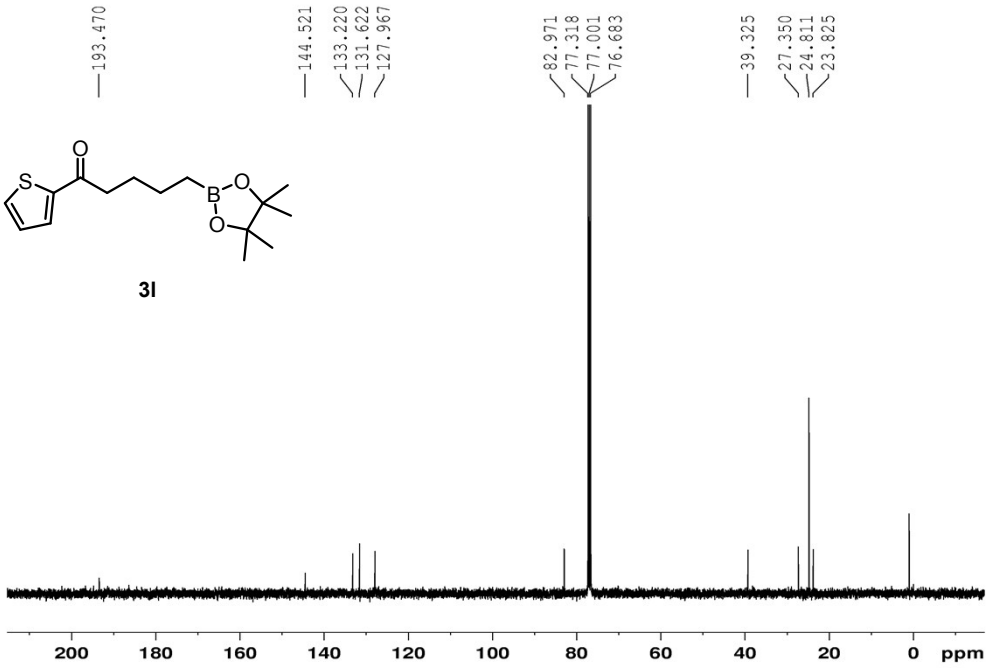
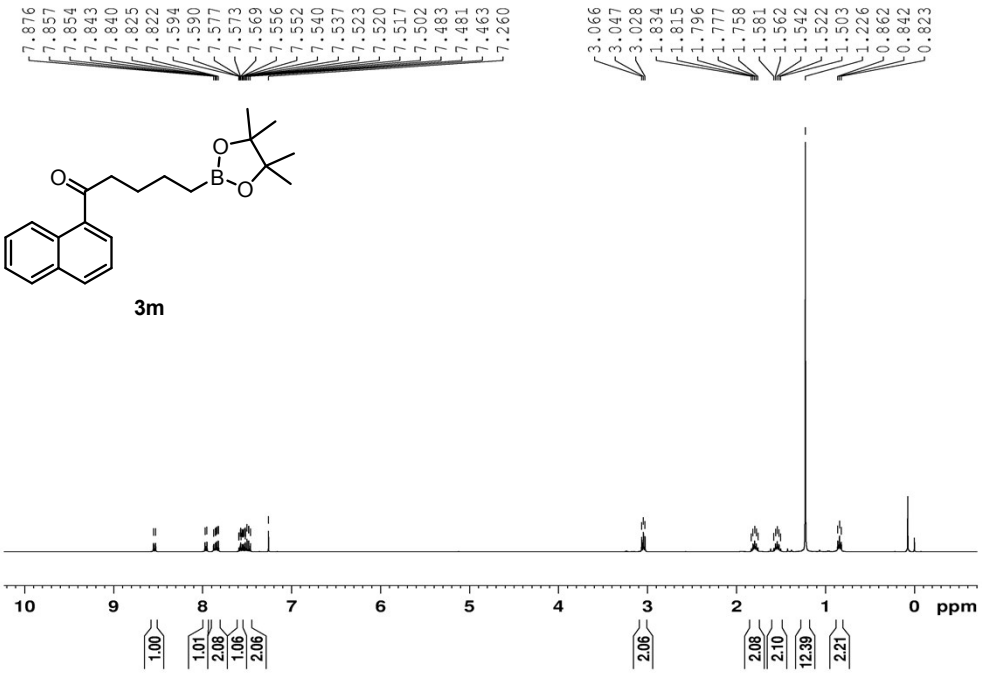
7.692
7.689
7.613
7.611
7.601
7.598
7.260
7.124
7.115
7.112
7.102
2.911
2.892
2.873
1.793
1.775
1.755
1.736
1.717
1.549
1.529
1.509
1.490
1.471
1.237
0.849
0.828
0.809
O
S
B
O
O
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.97
1.00
2.08
2.11
2.13
12.03
2.19

3l


193.470
144.521
133.220
131.622
127.967
82.971
77.318
77.001
76.683
39.325
27.350
24.811
23.825
O
S
B
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm

3l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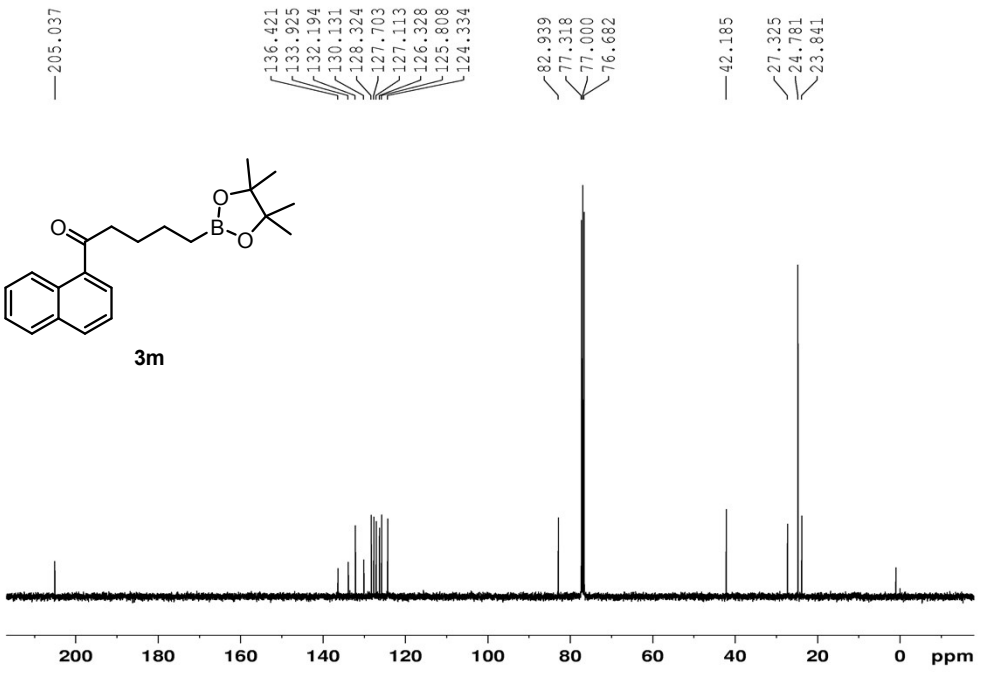
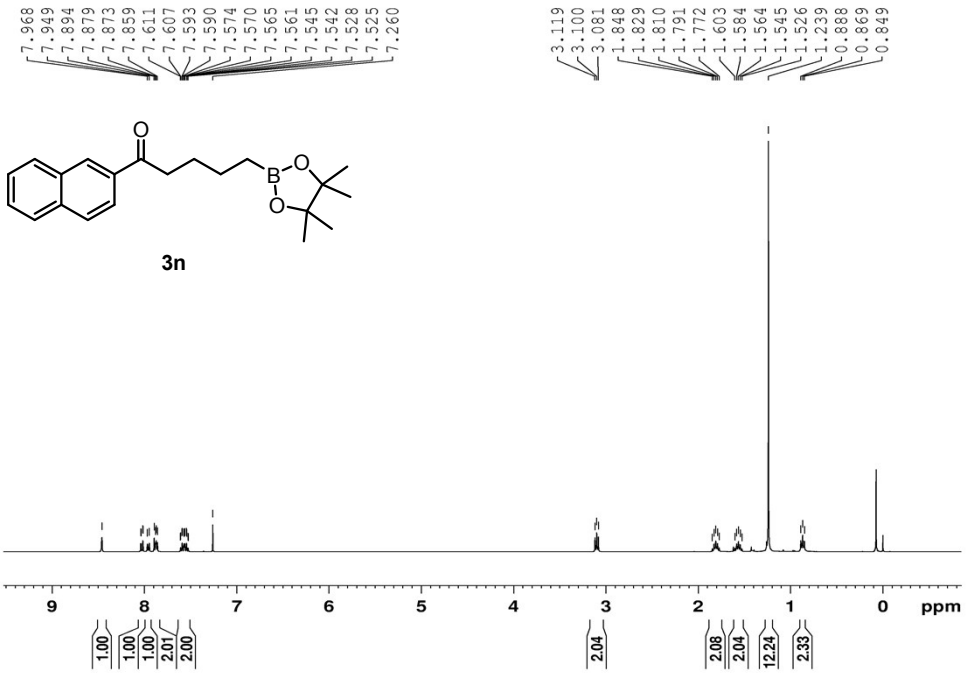
7.857
7.854
7.843
7.840
7.825
7.822
7.594
7.590
7.577
7.573
7.569
7.556
7.552
7.540
7.537
7.523
7.520
7.517
7.502
7.483
7.481
7.463
7.260
3.066
3.047
3.028
1.834
1.815
1.796
1.777
1.758
1.581
1.562
1.542
1.522
1.503
1.226
0.862
0.842
0.823
O
O
B
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
2.08
1.06
2.06
2.06
2.08
2.10
12.39
2.21

3m


205.037
136.421
133.925
132.194
130.131
128.324
127.703
127.113
126.328
125.808
124.334
82.939
77.318
77.000
76.682
42.185
27.325
24.781
23.841
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0
ppm

3m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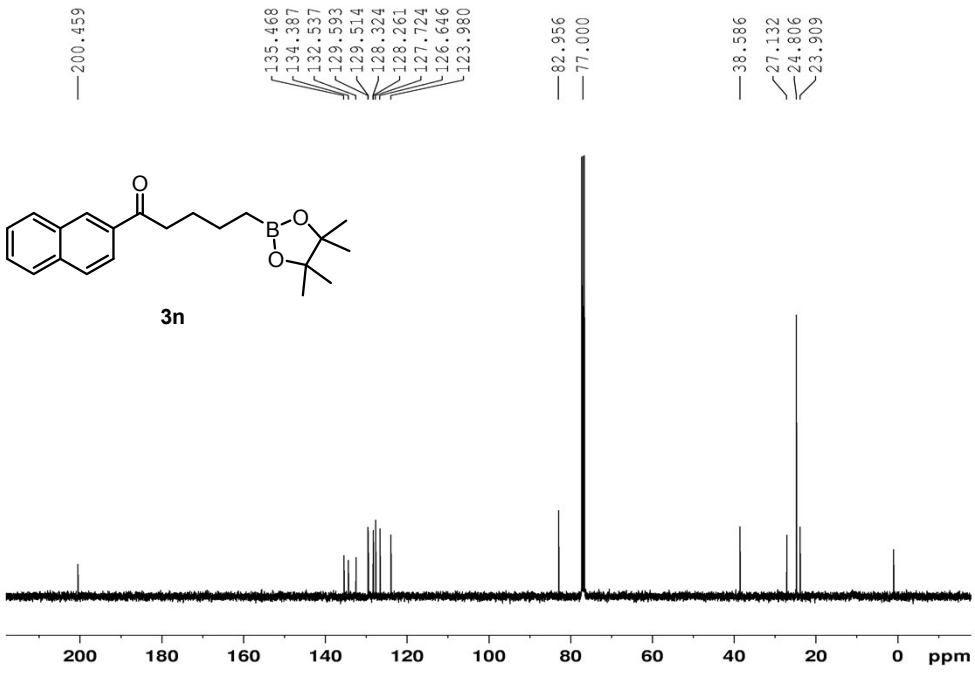
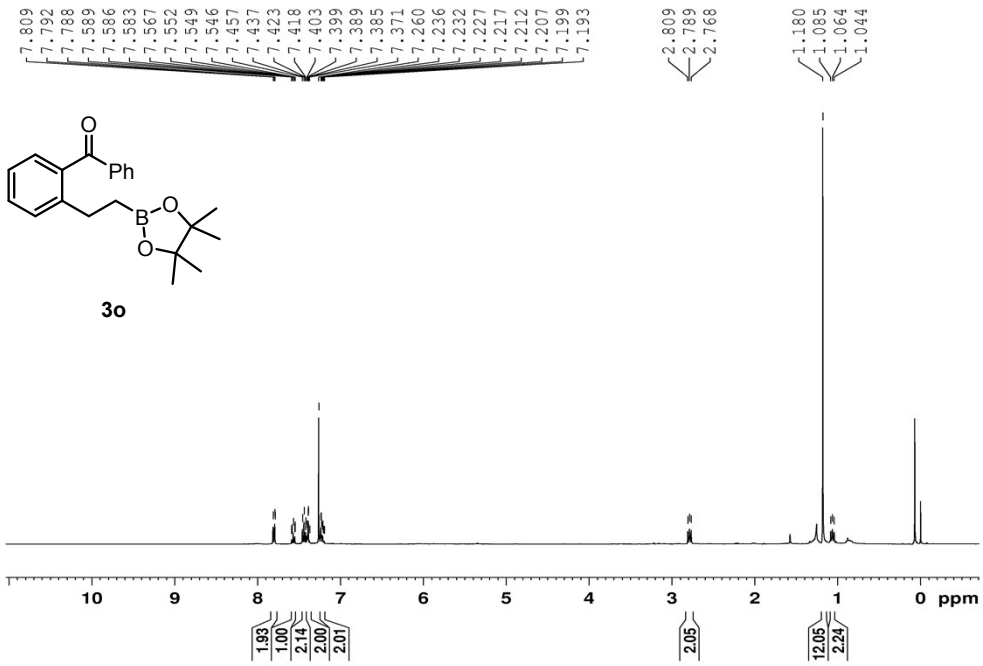
7.894
7.879
7.873
7.859
7.611
7.607
7.593
7.590
7.574
7.570
7.565
7.561
7.545
7.542
7.528
7.525
7.260
3.119
3.100
3.081
1.848
1.829
1.810
1.791
1.772
1.603
1.584
1.564
1.545
1.526
1.239
0.888
0.869
0.849
O
B
O
O
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.00
2.01
2.00
2.04
2.08
2.04
12.24
2.33

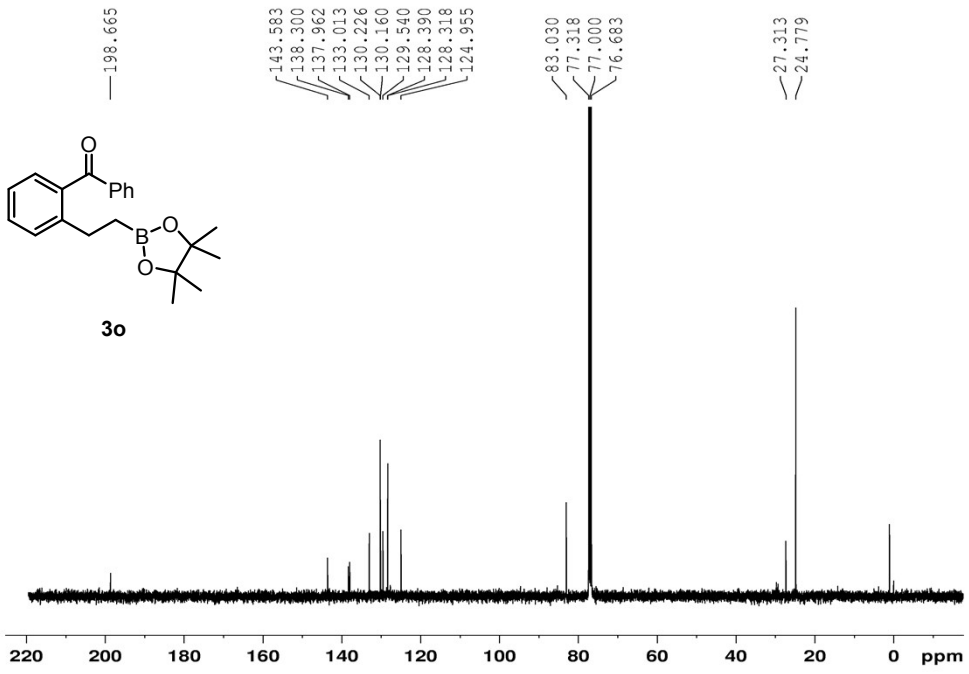
3n


200.459
135.468
134.387
132.537
129.593
129.514
128.324
128.261
127.724
126.646
123.980
82.956
77.000
38.586
27.132
24.806
23.909
O
B
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm

3n


7.809
7.792
7.788
7.589
7.586
7.583
7.567
7.552
7.549
7.546
7.457
7.437
7.423
7.418
7.403
7.399
7.389
7.385
7.371
7.260
7.236
7.232
7.227
7.217
7.212
7.207
7.199
7.193
2.809
2.789
2.768
1.180
1.085
1.064
1.044
O
Ph
B
O
O
10
9
8
7
6
5
4
3
2
1
0 ppm
1.93
1.00
2.14
2.00
2.01
2.05
12.05
2.24

3o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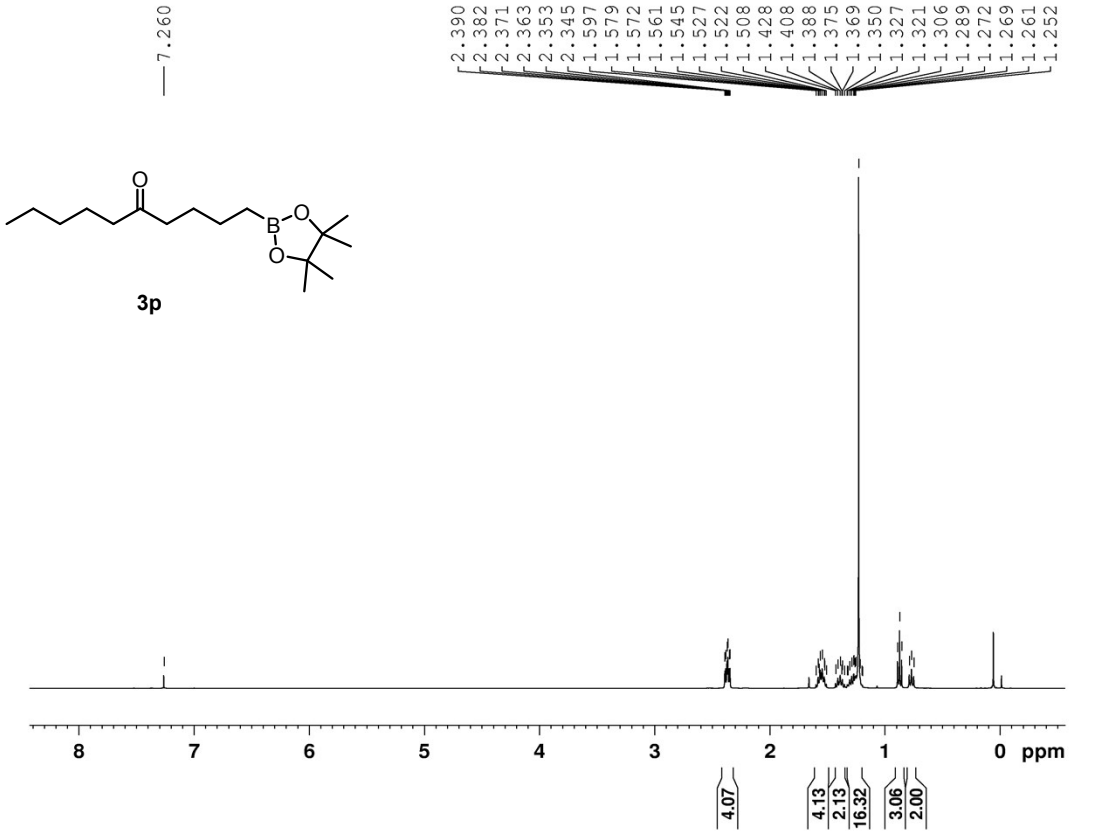
137.962
133.013
130.226
130.160
129.540
128.390
128.318
124.955
83.030
77.318
77.000
76.683
27.313
24.779
O
Ph
B
O
O
220
200
180
160
140
120
100
80
60
40
20
0 ppm

3o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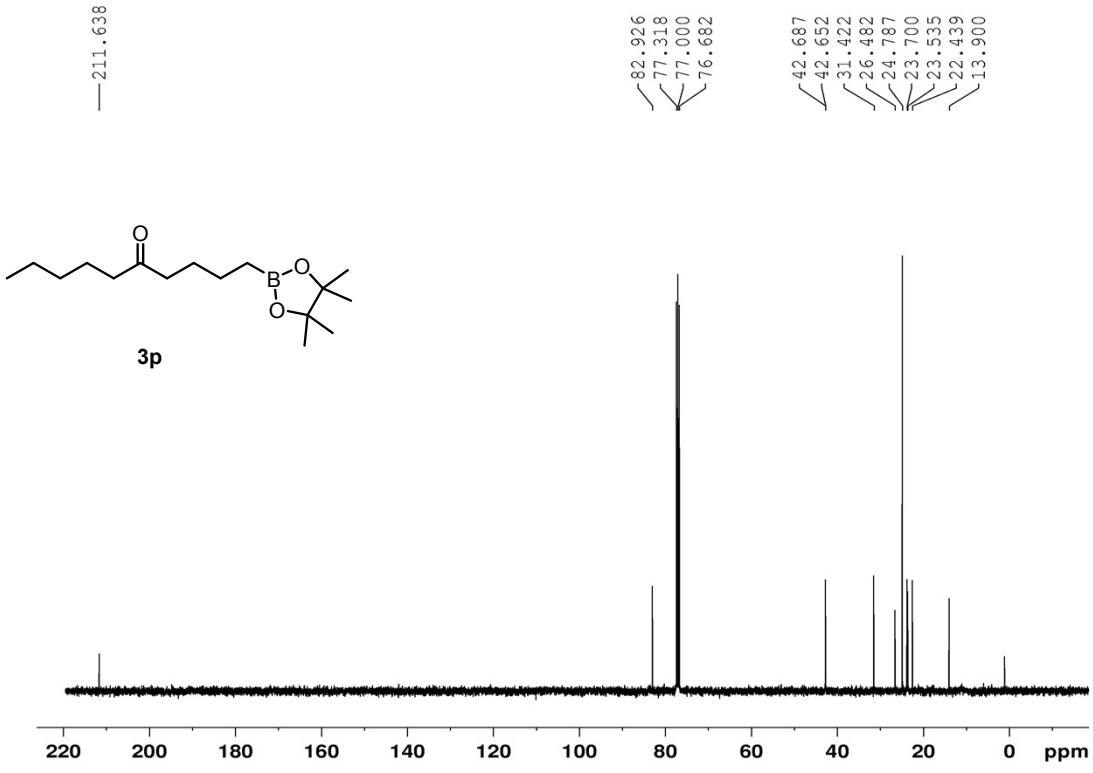
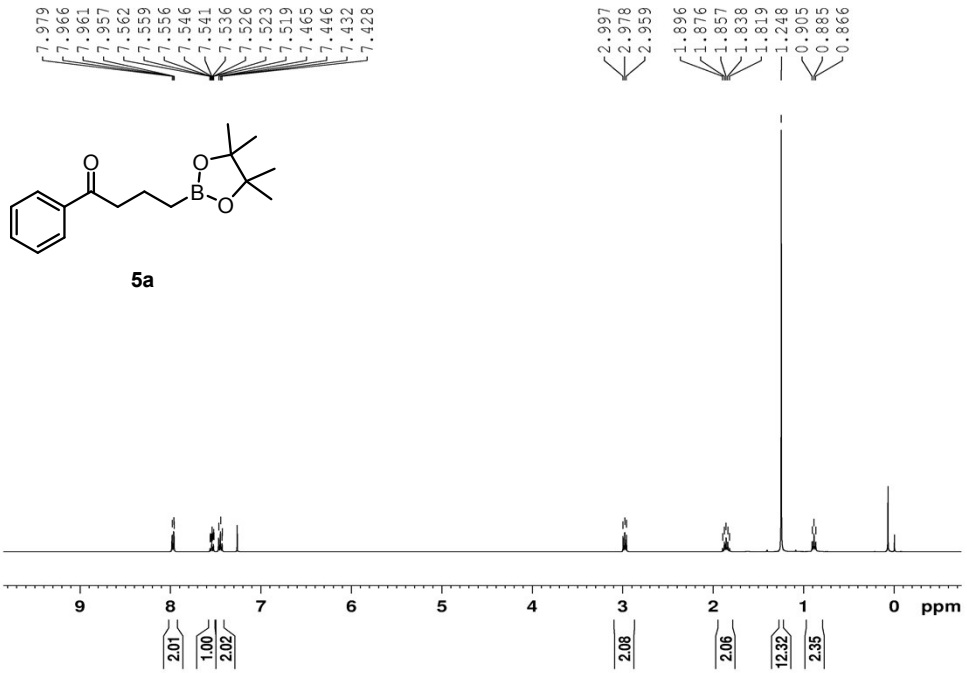
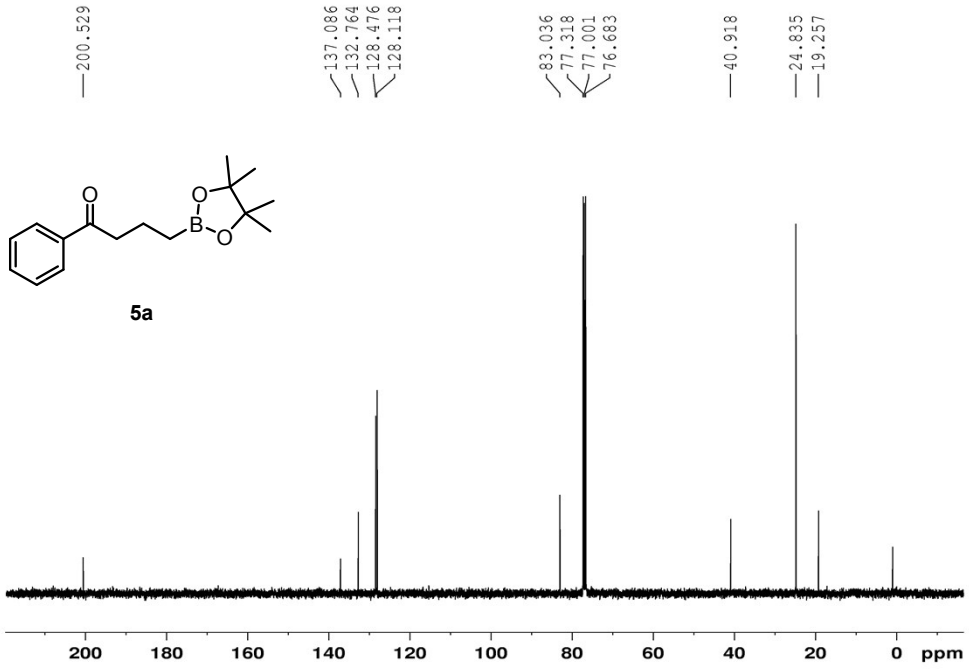
2.390
2.382
2.371
2.363
2.353
2.345
1.597
1.579
1.572
1.561
1.545
1.527
1.522
1.508
1.428
1.408
1.388
1.375
1.369
1.350
1.327
1.321
1.306
1.289
1.272
1.269
1.261
1.252
O
B
O
O
3p
8
7
6
5
4
3
2
1
0 ppm
4.07
4.13
2.13
16.32
3.06
2.00

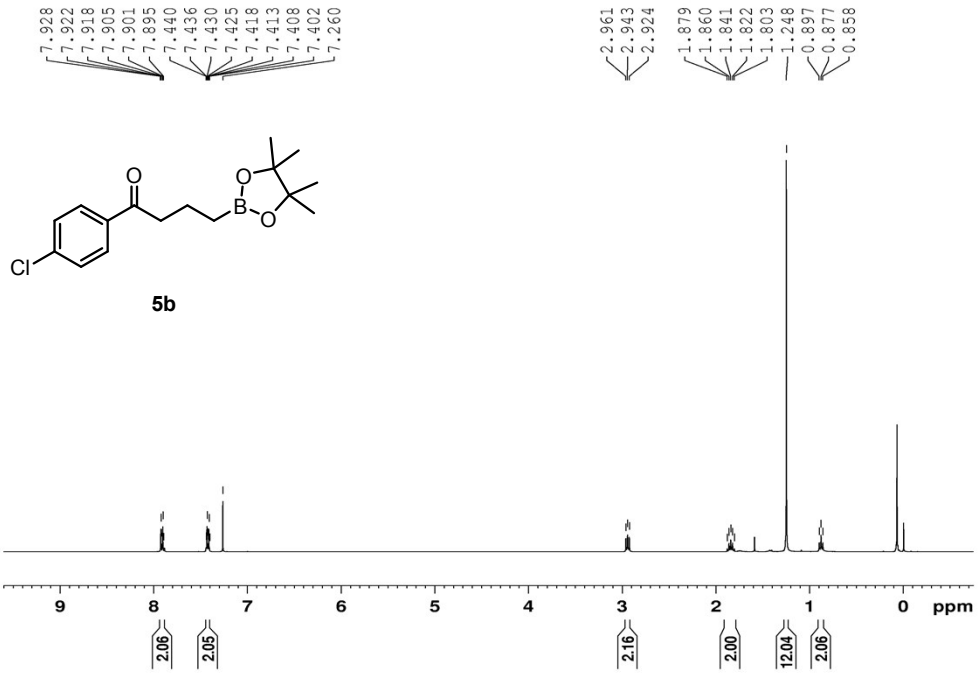

211.638
82.926
77.318
77.000
76.682
42.687
42.652
31.422
26.482
24.787
23.700
23.535
22.439
13.900
O
B
O
O
3p
220
200
180
160
140
120
100
80
60
40
20
0 ppm


7.979
7.966
7.961
7.957
7.562
7.559
7.556
7.546
7.541
7.536
7.526
7.523
7.519
7.465
7.446
7.432
7.428
2.997
2.978
2.959
1.896
1.876
1.857
1.838
1.819
1.248
0.905
0.885
0.866
O
O
B
O
9
8
7
6
5
4
3
2
1
0
ppm
2.01
1.00
2.02
2.08
2.06
12.32
2.35

5a


200.529
137.086
132.764
128.476
128.118
83.036
77.318
77.001
76.683
40.918
24.835
19.257
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0
ppm

5a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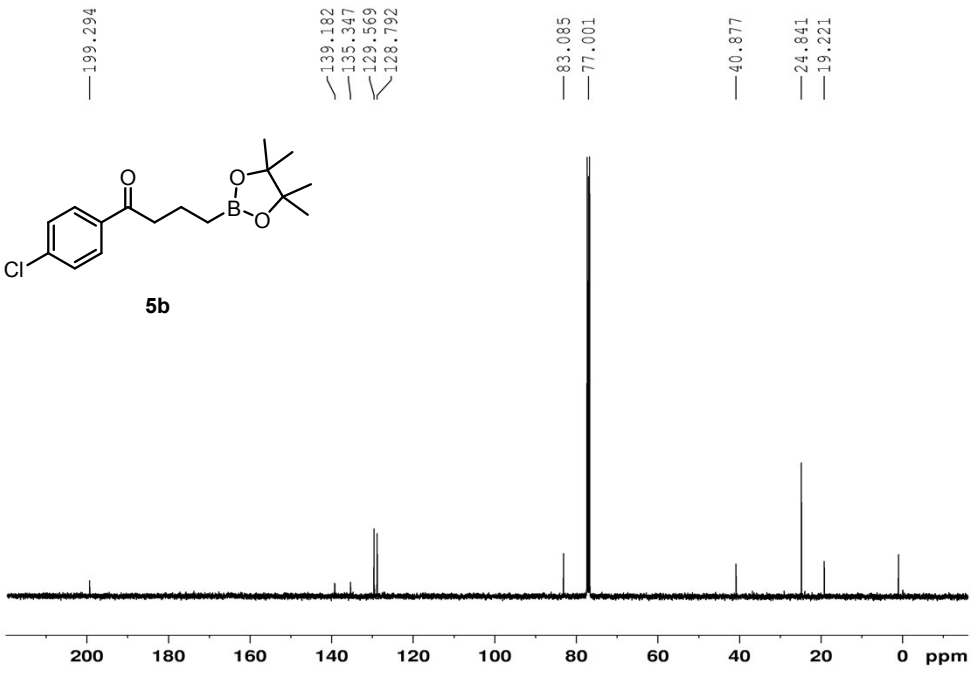
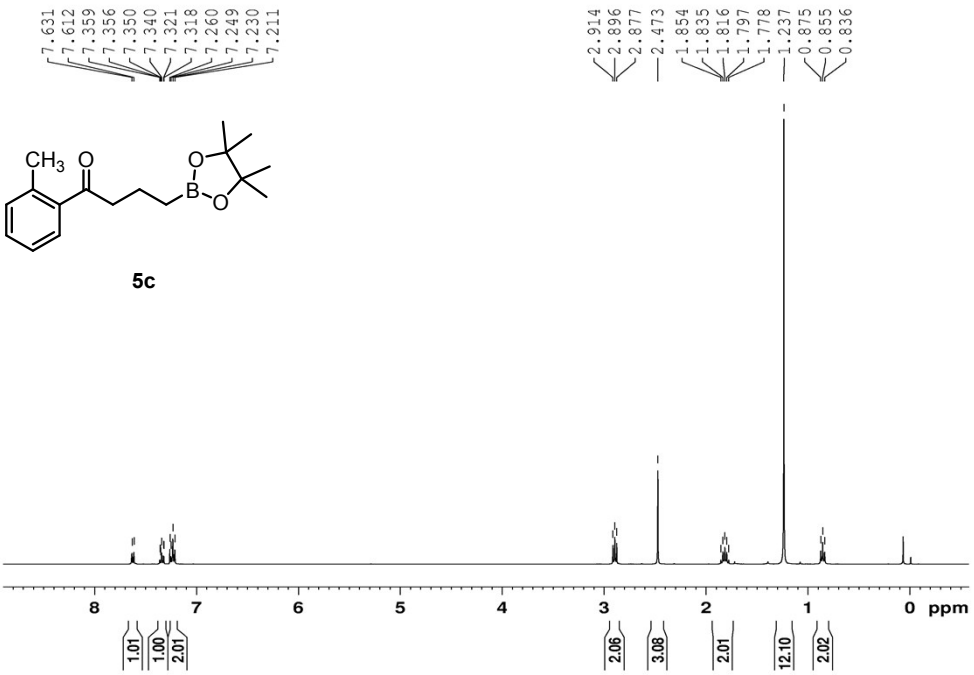
7.922
7.918
7.905
7.901
7.895
7.440
7.436
7.430
7.425
7.418
7.413
7.408
7.402
7.260
2.961
2.943
2.924
1.879
1.860
1.841
1.822
1.803
1.248
0.897
0.877
0.858
O
O
B
O
Cl
9
8
7
6
5
4
3
2
1
0
ppm
2.06
2.05
2.16
2.00
12.04
2.06

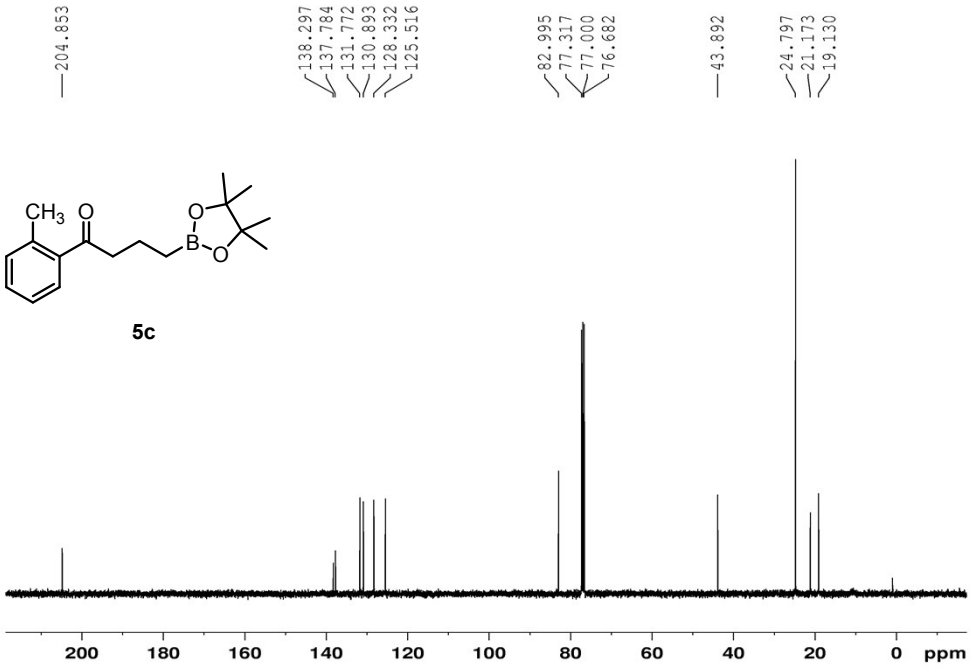
5b


199.294
139.182
135.347
129.569
128.792
83.085
77.001
40.877
24.841
19.221
O
O
B
O
Cl
200
180
160
140
120
100
80
60
40
20
0
ppm

5b


7.631
7.612
7.359
7.356
7.350
7.340
7.321
7.318
7.260
7.249
7.230
7.211
2.914
2.896
2.877
2.473
1.854
1.835
1.816
1.797
1.778
1.237
0.875
0.855
0.836
CH3
O
O
B
O
8
7
6
5
4
3
2
1
0 ppm
1.01
1.00
2.01
2.06
3.08
2.01
12.10
2.02

5c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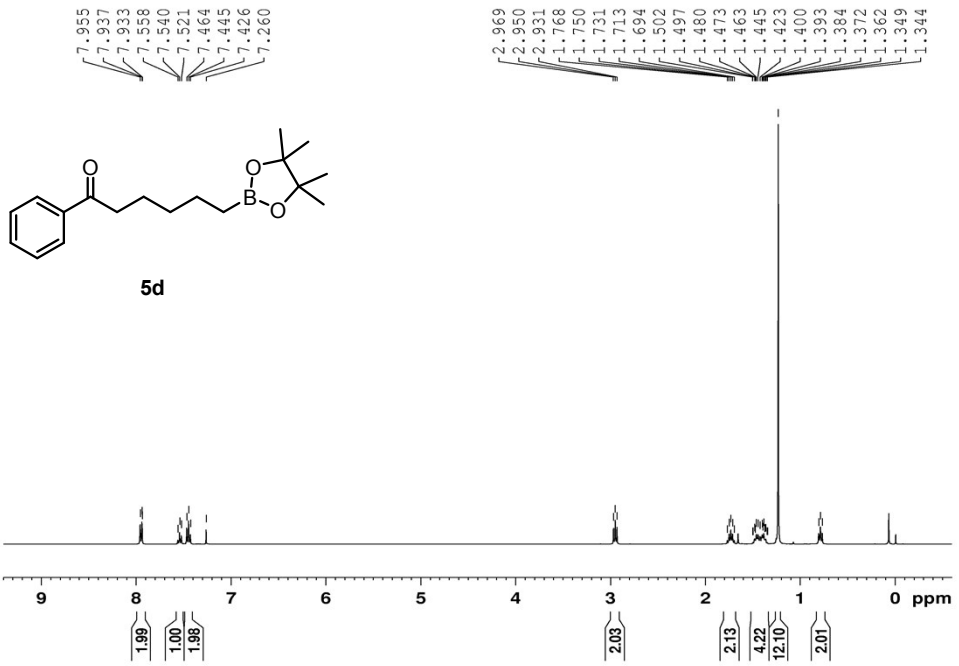
131.772
130.893
128.332
125.516
82.995
77.317
77.000
76.682
43.892
24.797
21.173
19.130
CH3
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0 ppm

5c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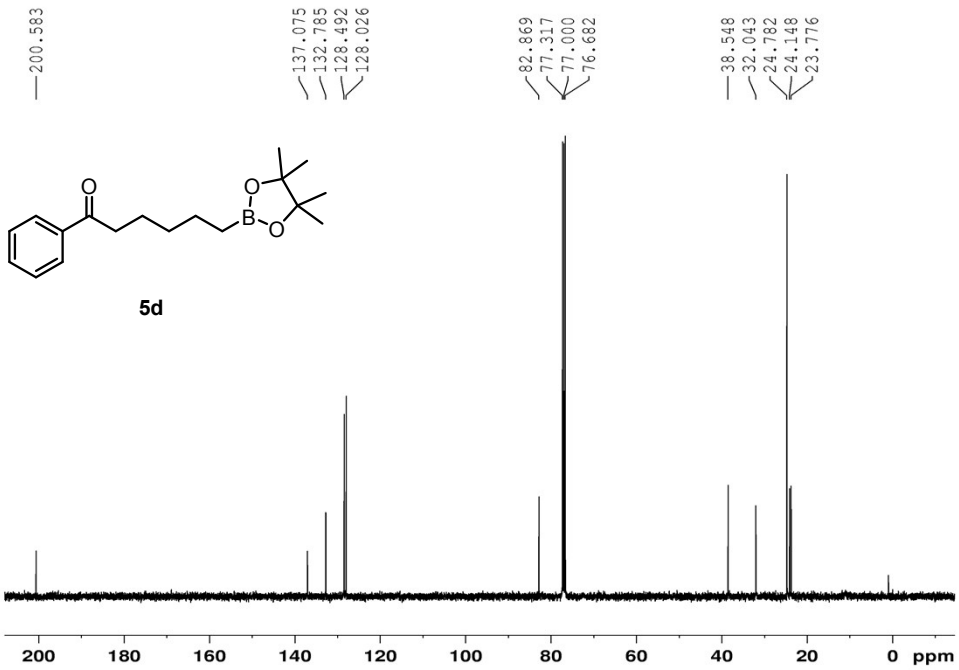
7.937
7.933
7.558
7.540
7.521
7.464
7.445
7.426
7.260
2.969
2.950
2.931
1.768
1.750
1.731
1.713
1.694
1.502
1.497
1.480
1.473
1.463
1.445
1.423
1.400
1.393
1.384
1.372
1.362
1.349
1.344
O
O
B
O
9
8
7
6
5
4
3
2
1
0 ppm
1.99
1.00
1.98
2.03
2.13
4.22
12.10
2.01

5d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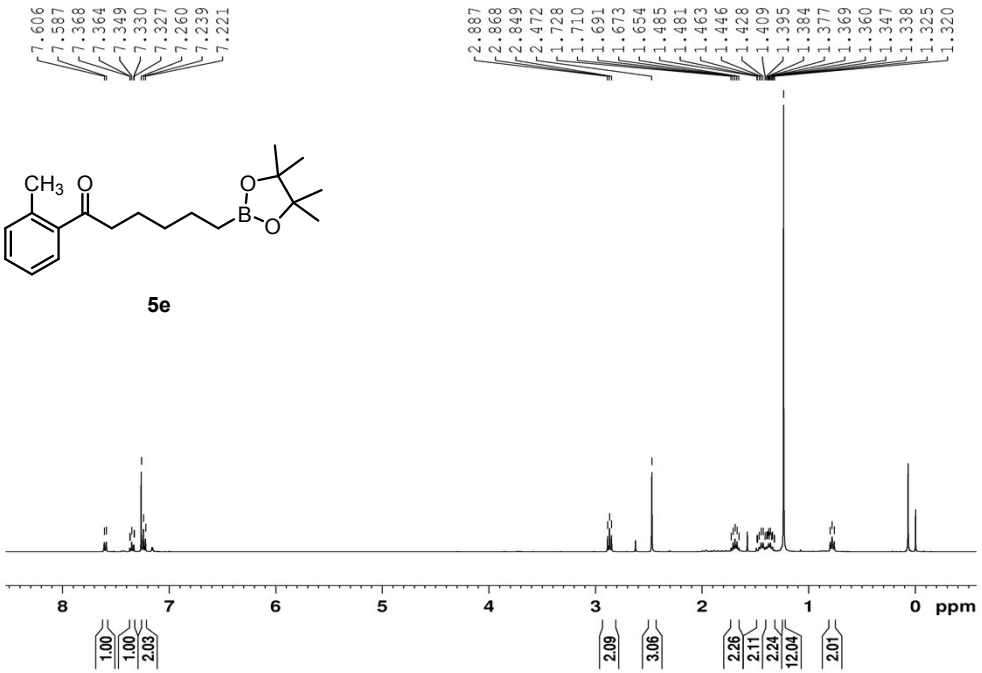
132.785
128.492
128.026
82.869
77.317
77.000
76.682
38.548
32.043
24.782
24.148
23.776
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0 ppm

5d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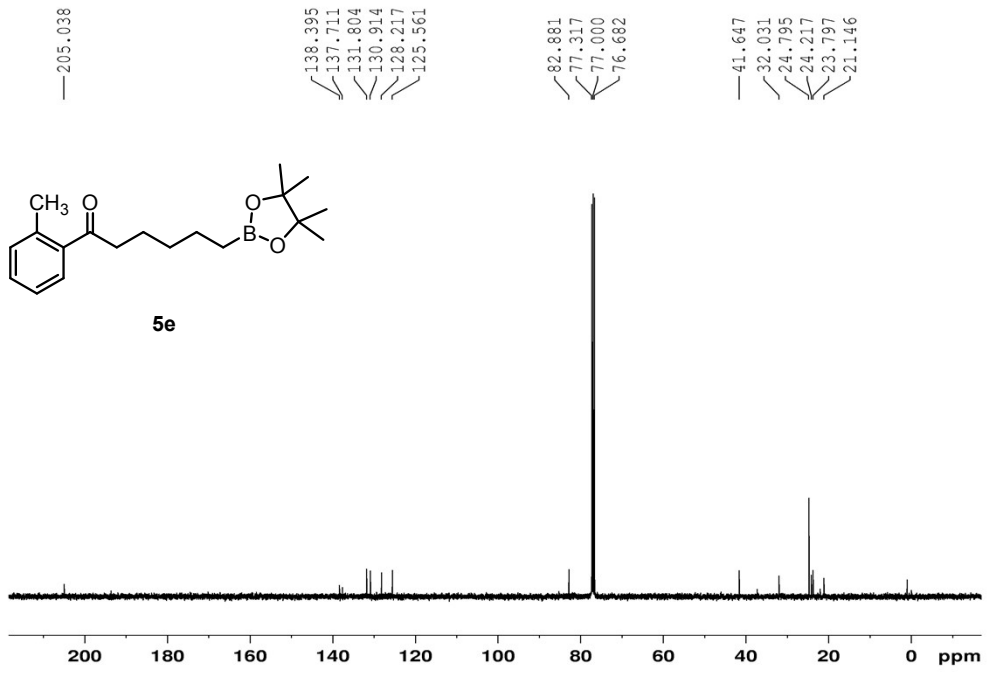
7.587
7.368
7.364
7.349
7.330
7.327
7.260
7.239
7.221
2.887
2.868
2.849
2.472
1.728
1.710
1.691
1.673
1.654
1.485
1.481
1.463
1.446
1.428
1.409
1.395
1.384
1.377
1.369
1.360
1.347
1.338
1.325
1.320
CH3
O
O
B
O
8
7
6
5
4
3
2
1
0 ppm
1.00
1.00
2.03
2.09
3.06
2.26
2.11
2.24
12.04
2.01

5e


205.038
138.395
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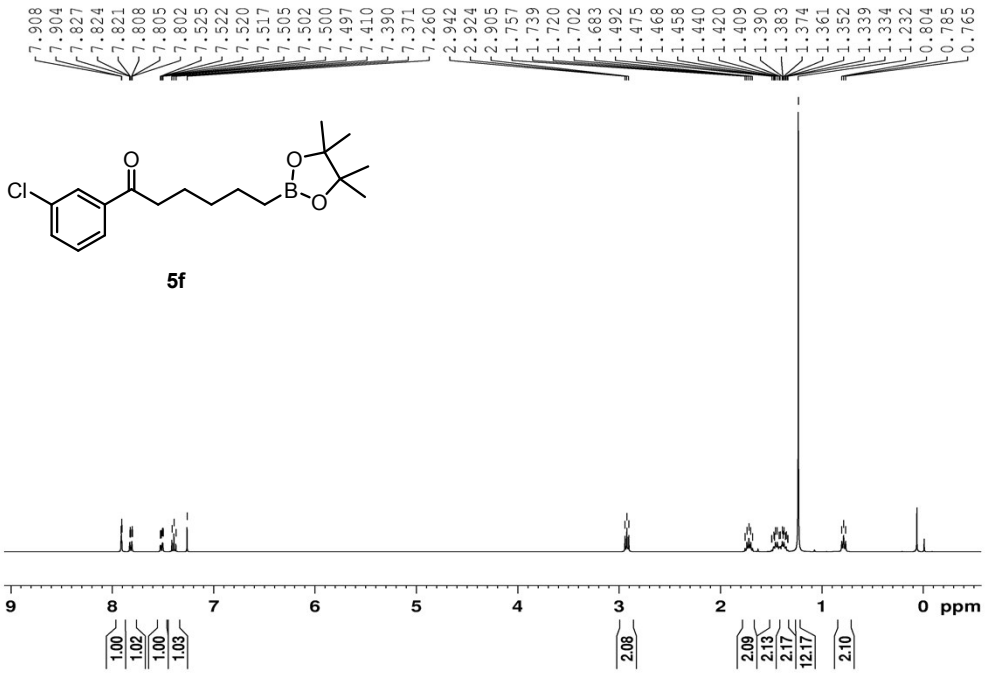
130.914
128.217
125.561
82.881
77.317
77.000
76.682
41.647
32.031
24.795
24.217
23.797
21.146
CH3
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0 ppm

5e


7.908
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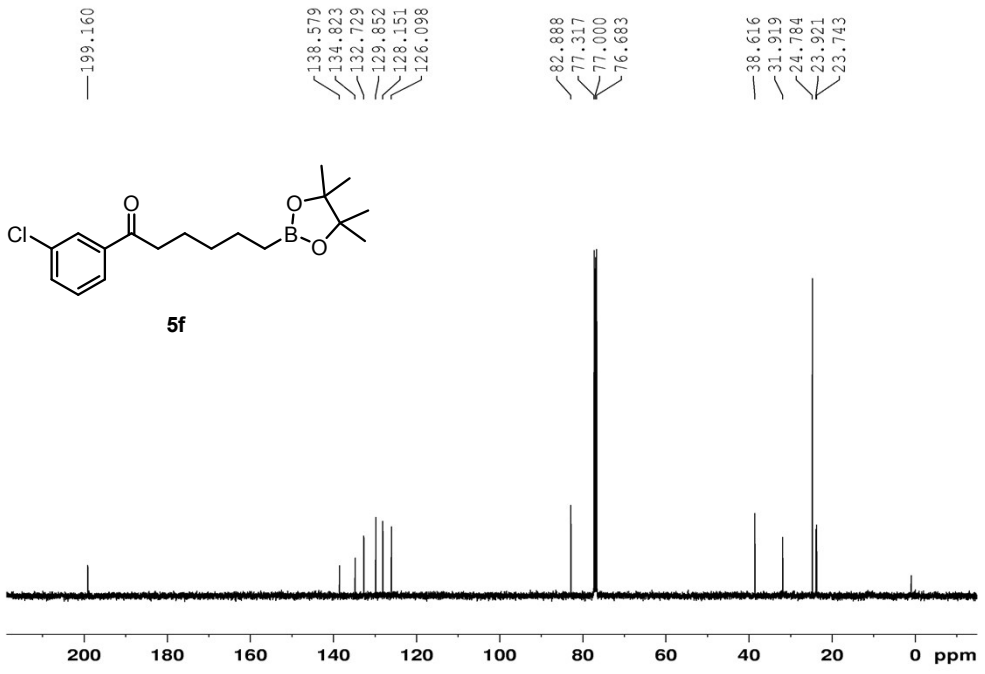
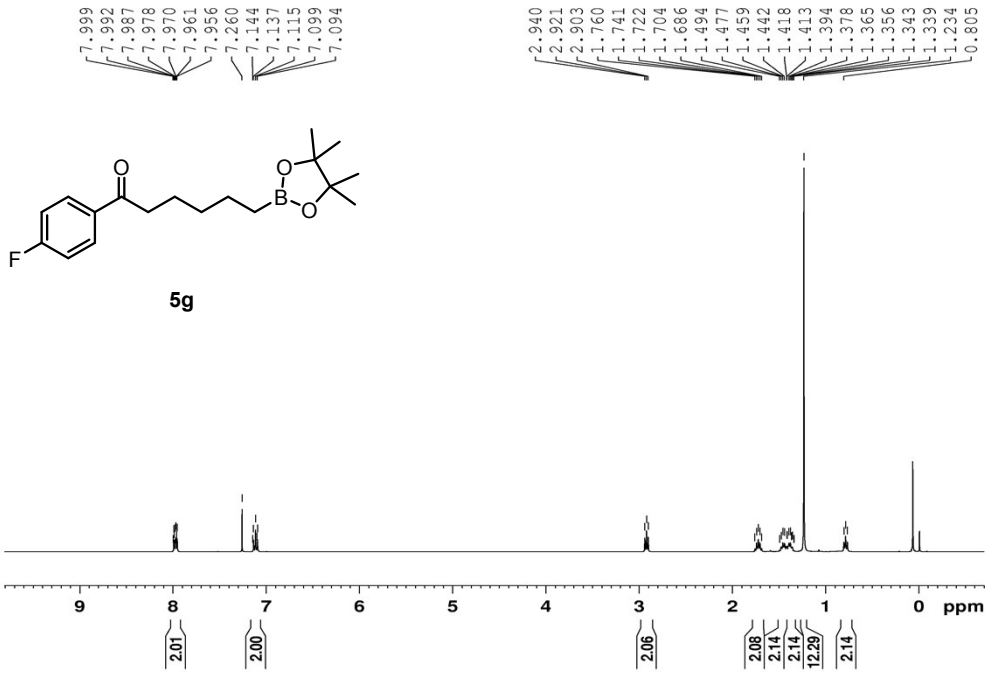
7.827
7.824
7.821
7.808
7.805
7.802
7.525
7.522
7.520
7.517
7.505
7.502
7.500
7.497
7.410
7.390
7.371
7.260
2.942
2.924
2.905
1.757
1.739
1.720
1.702
1.683
1.492
1.475
1.468
1.458
1.440
1.420
1.409
1.390
1.383
1.374
1.361
1.352
1.339
1.334
1.232
0.804
0.785
0.765
O
O
B
O
Cl
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.02
1.00
1.03
2.08
2.09
2.13
2.17
12.17
2.10

5f


199.160
138.579
134.823
132.729
129.852
128.151
126.098
82.888
77.317
77.000
76.683
38.616
31.919
24.784
23.921
23.743
O
O
B
O
Cl
200
180
160
140
120
100
80
60
40
20
0 ppm

5f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7.956
7.260
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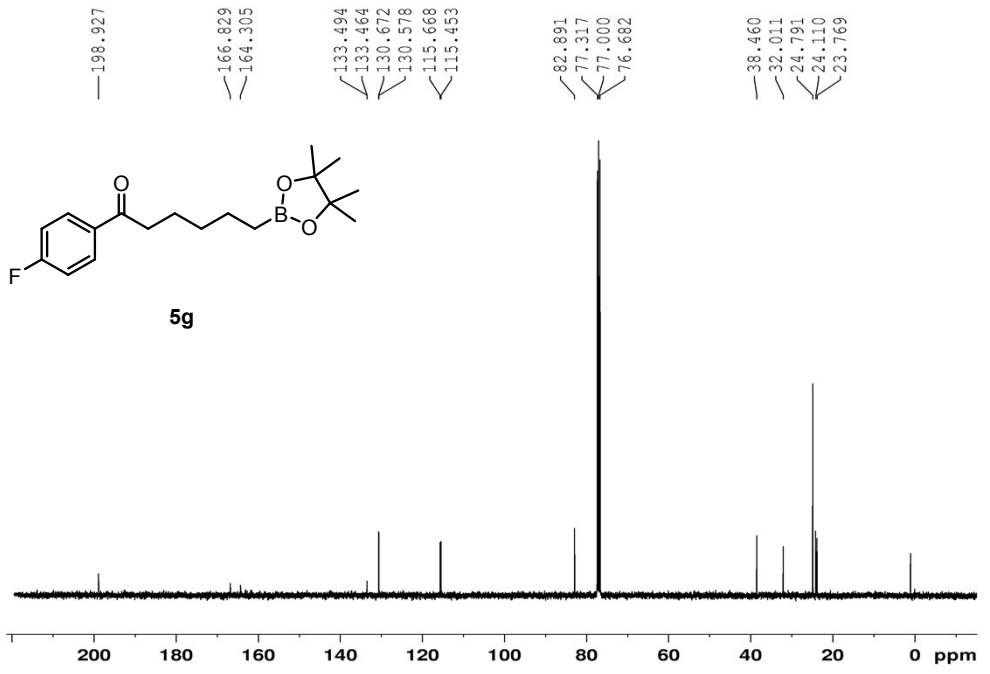
7.137
7.115
7.099
7.094
2.940
2.921
2.903
1.760
1.741
1.722
1.704
1.686
1.494
1.477
1.459
1.442
1.418
1.413
1.394
1.378
1.365
1.356
1.343
1.339
1.234
0.805
O
O
B
O
F
5g
9
8
7
6
5
4
3
2
1
0 ppm
2.01
2.00
2.06
2.08
2.14
2.14
12.29
2.14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133.464
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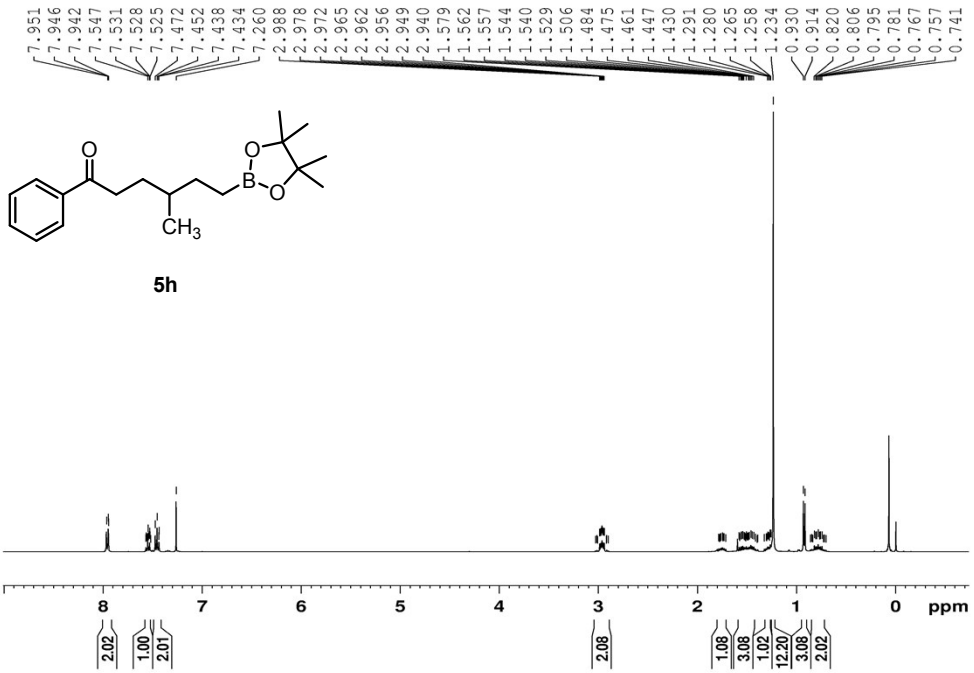
130.578
115.668
115.453
82.891
77.317
77.000
76.682
38.460
32.011
24.791
24.110
23.769
O
O
B
O
F
5g
200
180
160
140
120
100
80
60
40
20
0 ppm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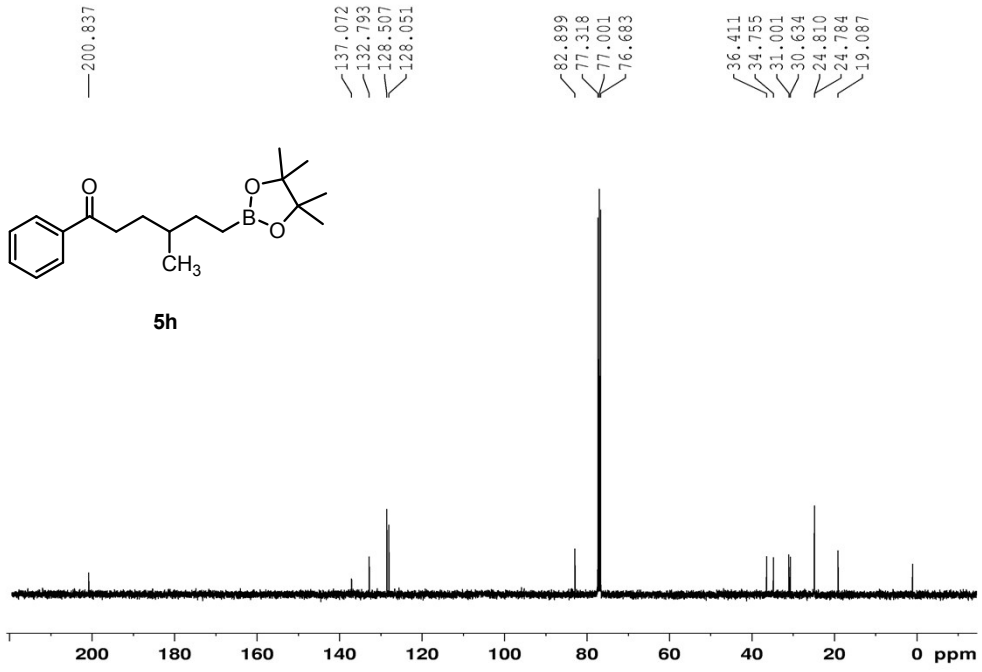
7.547
7.531
7.528
7.525
7.472
7.452
7.438
7.434
7.260
2.988
2.978
2.972
2.965
2.962
2.956
2.949
2.940
1.579
1.562
1.557
1.544
1.540
1.529
1.506
1.484
1.475
1.461
1.447
1.430
1.291
1.280
1.265
1.258
1.234
0.930
0.914
0.820
0.806
0.795
0.781
0.767
0.757
0.741
O
O
B
O
CH3
8
7
6
5
4
3
2
1
0
ppm
2.02
1.00
2.01
2.08
1.08
3.08
1.02
12.20
3.08
2.02

5h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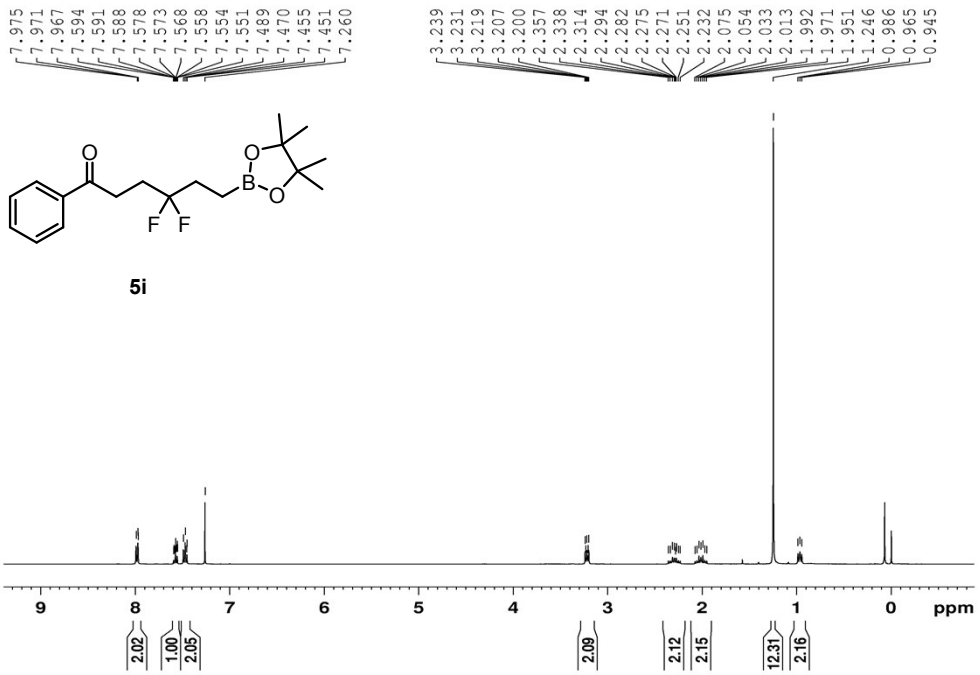
77.318
77.001
76.683
36.411
34.755
31.001
30.634
24.810
24.784
19.087
O
O
B
O
CH3
200
180
160
140
120
100
80
60
40
20
0 ppm

5h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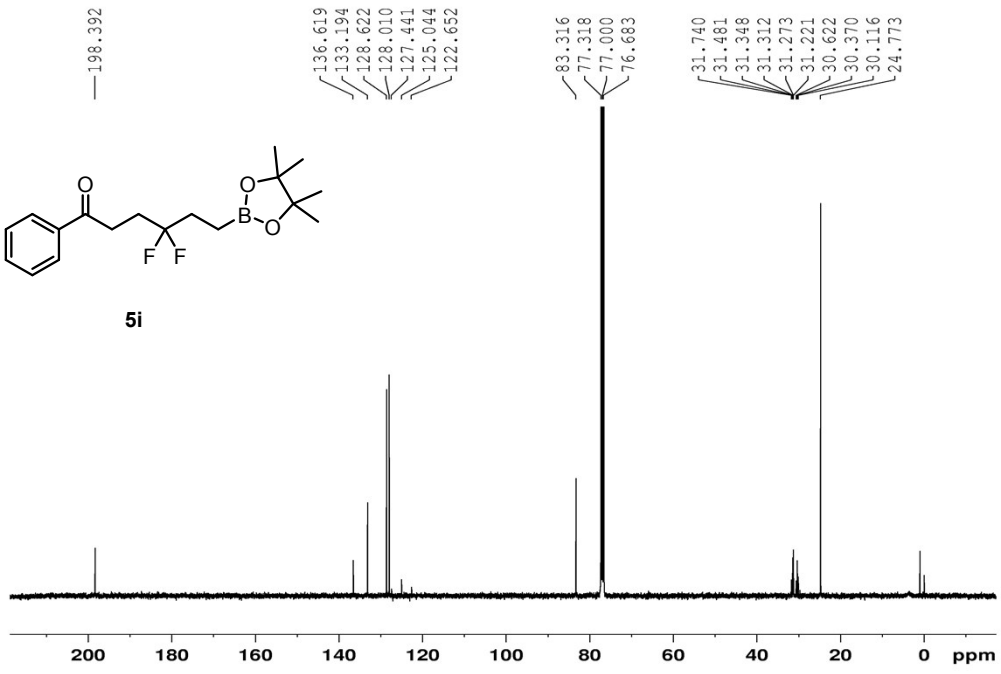
7.594
7.591
7.588
7.578
7.573
7.568
7.558
7.554
7.551
7.489
7.470
7.455
7.451
7.260
3.239
3.231
3.219
3.207
3.200
2.357
2.338
2.314
2.294
2.282
2.275
2.271
2.251
2.232
2.075
2.054
2.033
2.013
1.992
1.971
1.951
1.246
0.986
0.965
0.945
O
O
B
O
F F
9
8
7
6
5
4
3
2
1
0
ppm
2.02
1.00
2.05
2.09
2.12
2.15
12.31
2.16

5i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136.619
133.194
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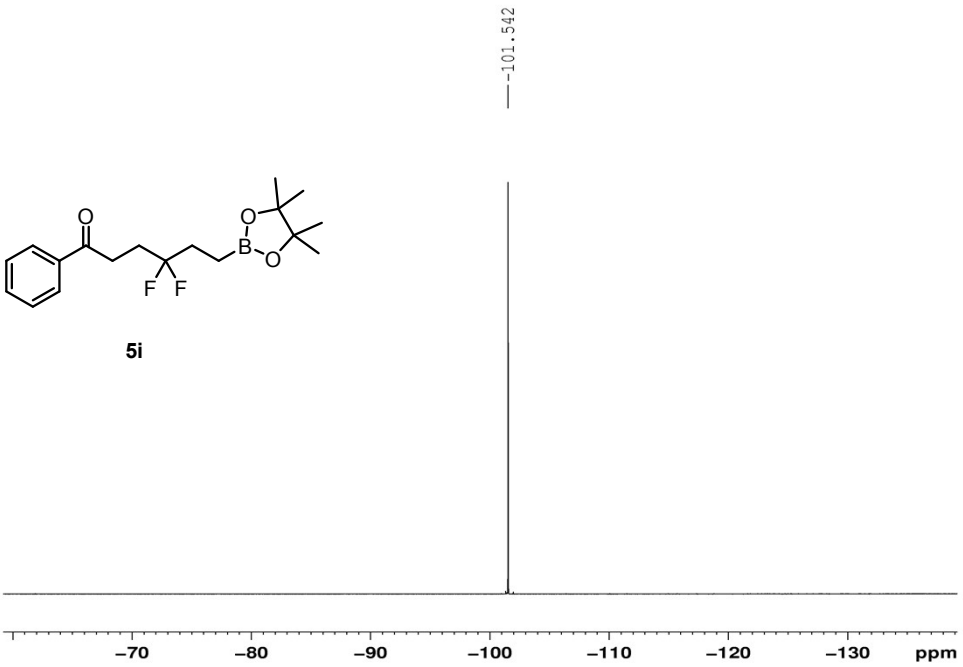
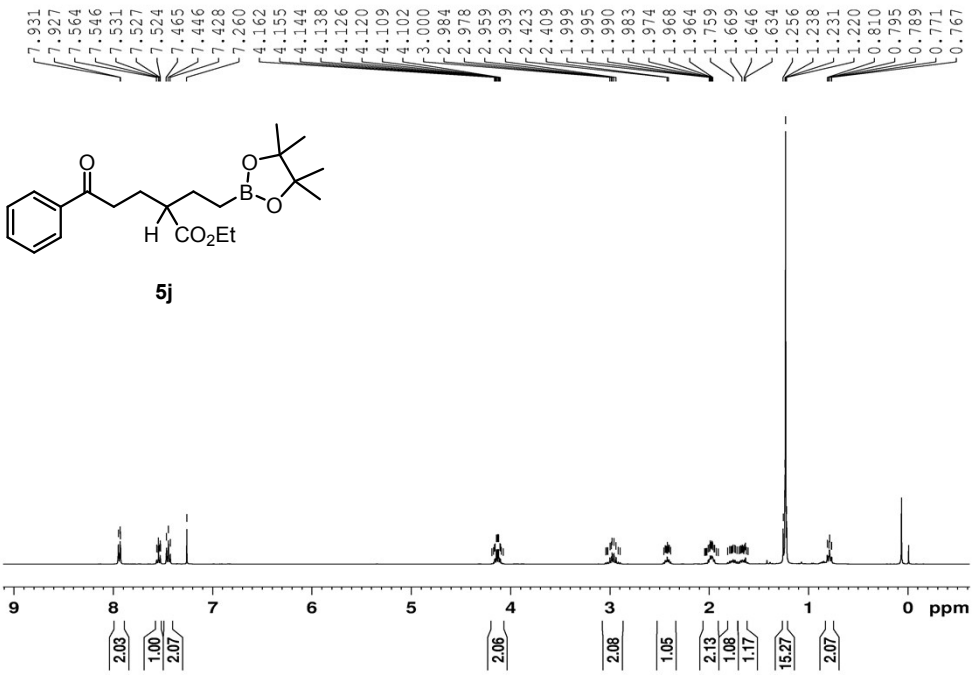
128.010
127.441
125.044
122.652
83.316
77.318
77.000
76.683
31.740
31.481
31.348
31.312
31.273
31.221
30.622
30.370
30.116
24.773
O
O
B
O
F F
200
180
160
140
120
100
80
60
40
20
0
ppm

5i


-101.542
O
O
B
O
F F
5i
−70
−80
−90
−100
−110
−120
−130
ppm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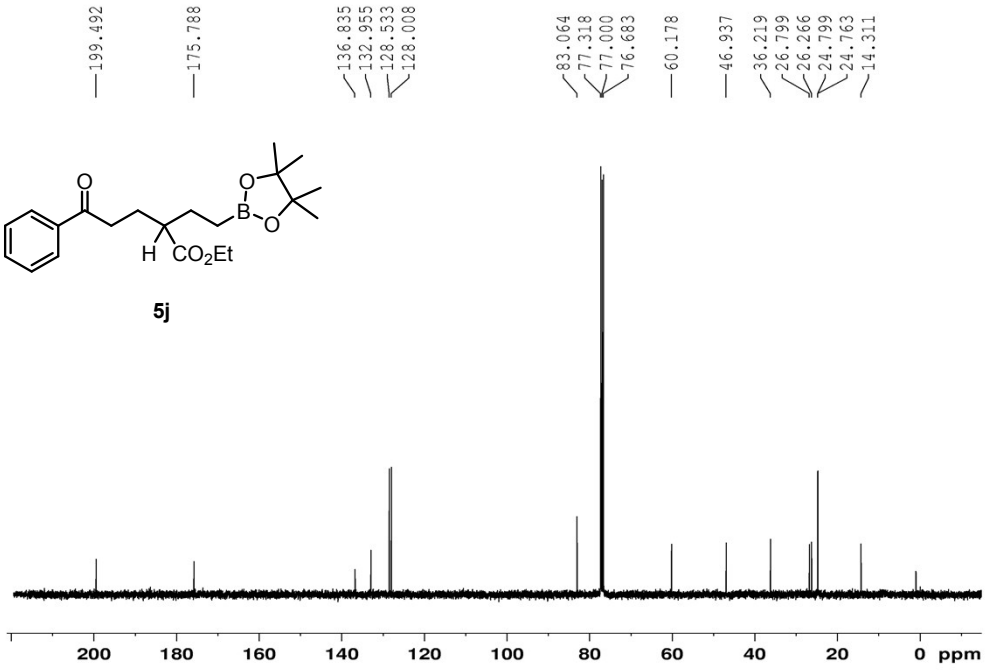
7.927
7.564
7.546
7.531
7.527
7.524
7.465
7.446
7.428
7.260
4.162
4.155
4.144
4.138
4.126
4.120
4.109
4.102
3.000
2.984
2.978
2.959
2.939
2.423
2.409
1.999
1.995
1.990
1.983
1.974
1.968
1.964
1.759
1.669
1.646
1.634
1.256
1.238
1.231
1.220
0.810
0.795
0.789
0.771
0.767
O
O
B
O
H
CO2Et
9
8
7
6
5
4
3
2
1
0 ppm
2.03
1.00
2.07
2.06
2.08
1.05
2.13
1.08
1.17
15.27
2.07

5j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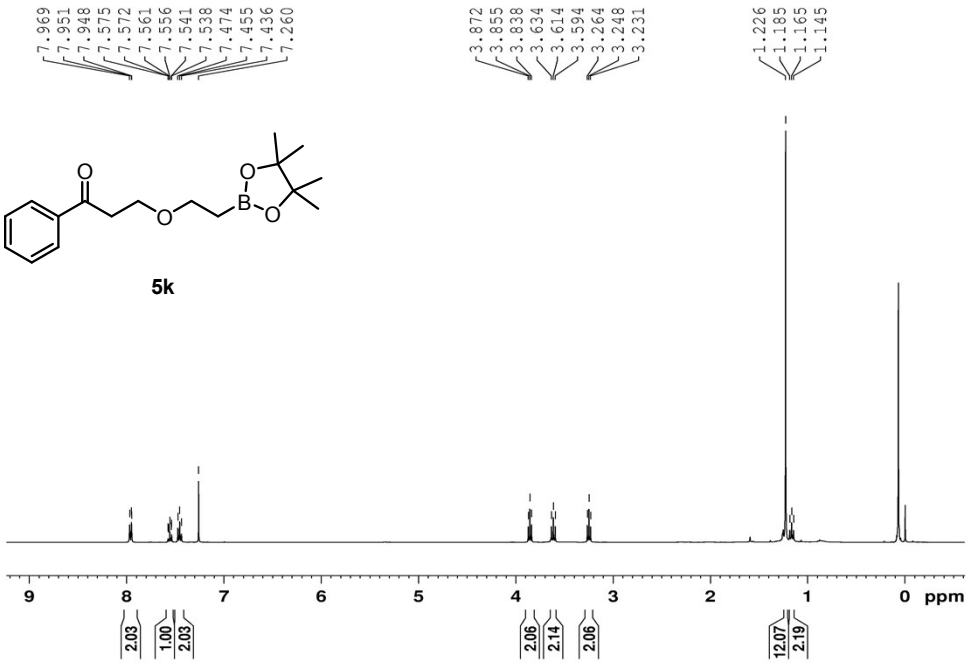
175.788
136.835
132.955
128.533
128.008
83.064
77.318
77.000
76.683
60.178
46.937
36.219
26.799
26.266
24.799
24.763
14.311
O
O
B
O
H
CO2Et
200
180
160
140
120
100
80
60
40
20
0 ppm

5j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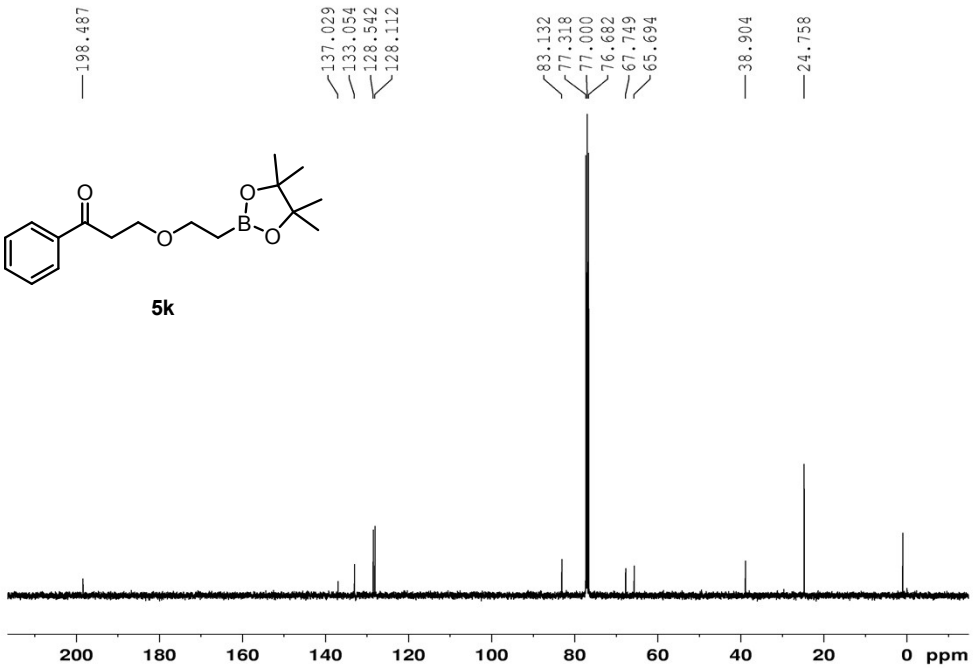
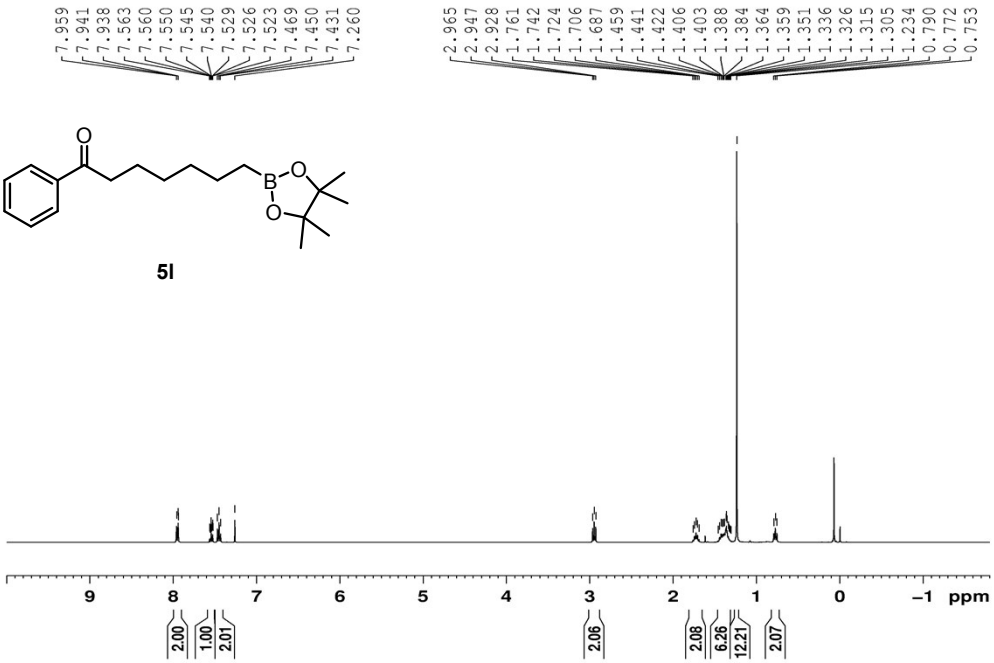
7.951
7.948
7.575
7.572
7.561
7.556
7.541
7.538
7.474
7.455
7.436
7.260
3.872
3.855
3.838
3.634
3.614
3.594
3.264
3.248
3.231
1.226
1.185
1.165
1.145
O
O
B
O
O
9
8
7
6
5
4
3
2
1
0 ppm
2.03
1.00
2.03
2.06
2.14
2.06
12.07
2.19

5k


198.487
137.029
133.054
128.542
128.112
83.132
77.318
77.000
76.682
67.749
65.694
38.904
24.758
O
O
B
O
O
200
180
160
140
120
100
80
60
40
20
0 ppm

5k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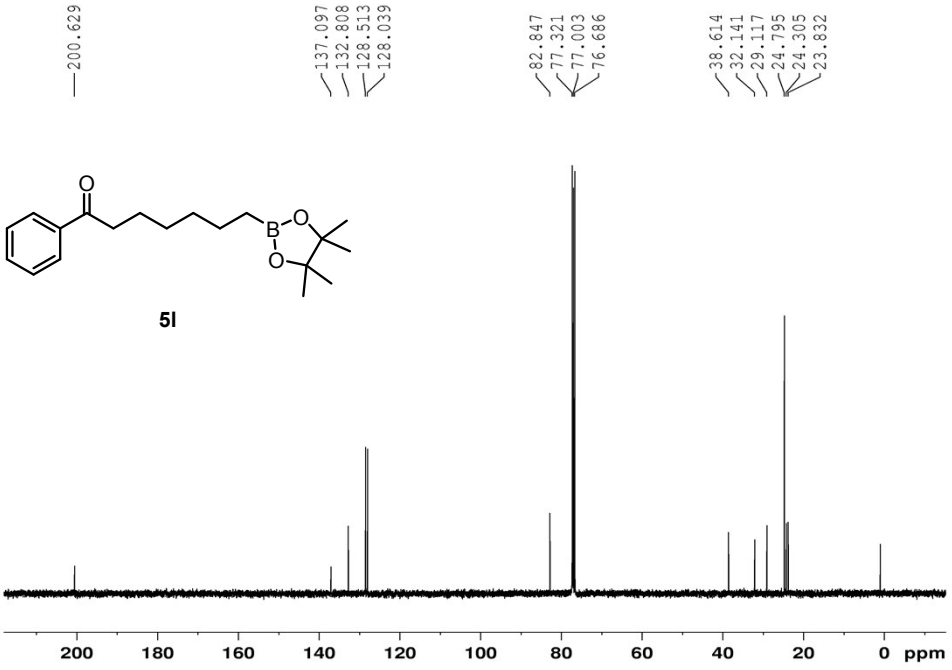
7.941
7.938
7.563
7.560
7.550
7.545
7.540
7.529
7.526
7.523
7.469
7.450
7.431
7.260
2.965
2.947
2.928
1.761
1.742
1.724
1.706
1.687
1.459
1.441
1.422
1.406
1.403
1.388
1.384
1.364
1.359
1.351
1.336
1.326
1.315
1.305
1.234
0.790
0.772
0.753
O
B
O
O
5l
9
8
7
6
5
4
3
2
1
0
−1 ppm
2.00
1.00
2.01
2.06
2.08
6.26
12.21
2.07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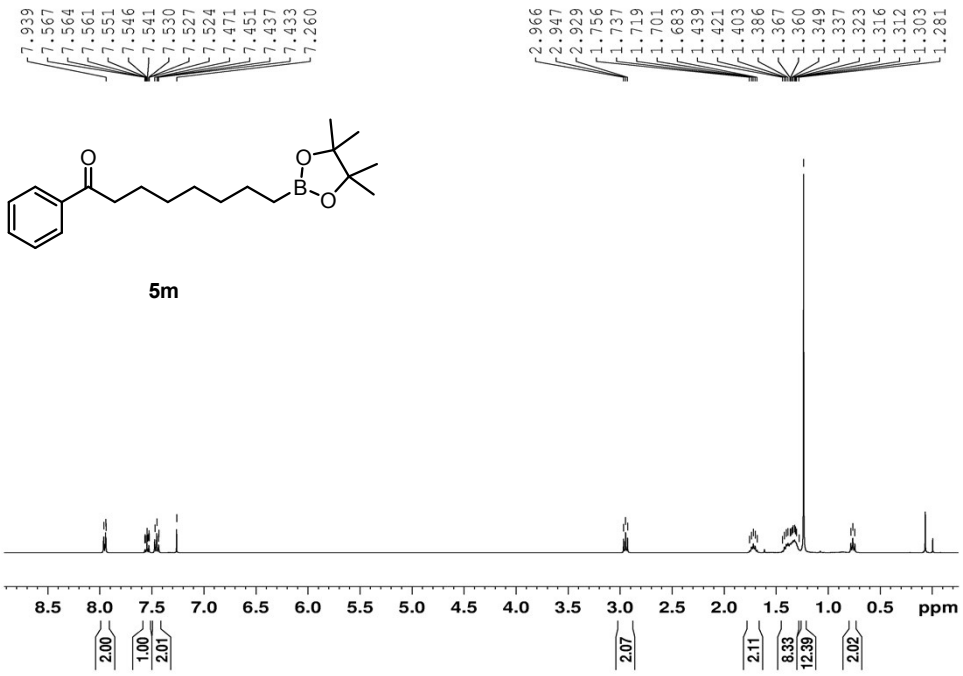
128.039
82.847
77.321
77.003
76.686
38.614
32.141
29.117
24.795
24.305
23.832
O
B
O
O
5l
200
180
160
140
120
100
80
60
40
20
0 ppm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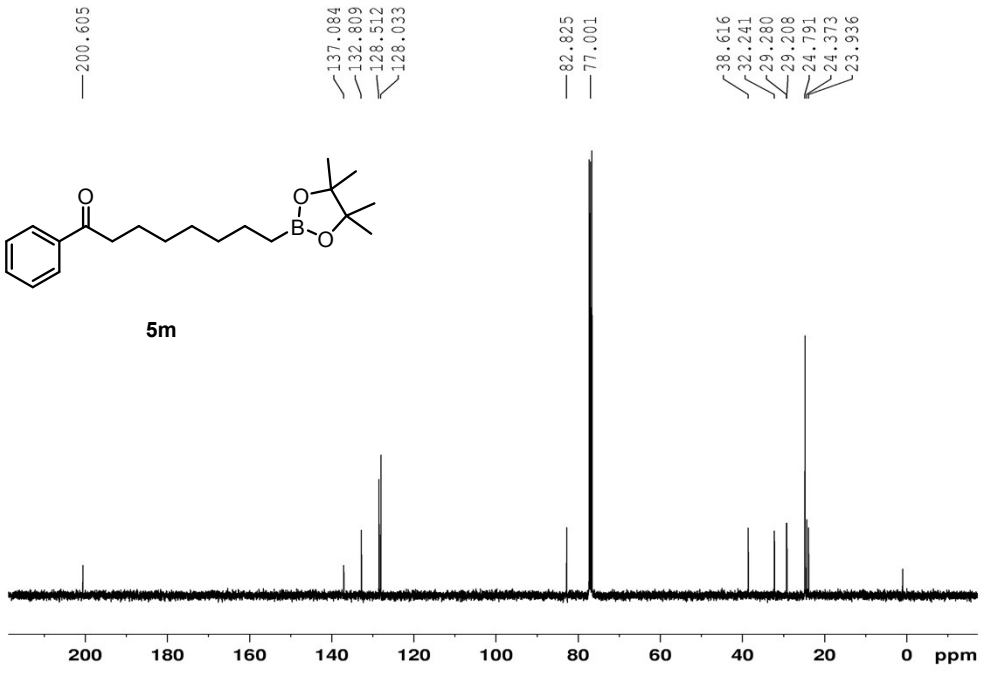
7.567
7.564
7.561
7.551
7.546
7.541
7.530
7.527
7.524
7.471
7.451
7.437
7.433
7.260
2.966
2.947
2.929
1.756
1.737
1.719
1.701
1.683
1.439
1.421
1.403
1.386
1.367
1.360
1.349
1.337
1.323
1.316
1.312
1.303
1.281
O
O
B
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
1.00
2.01
2.07
2.11
8.33
12.39
2.02

5m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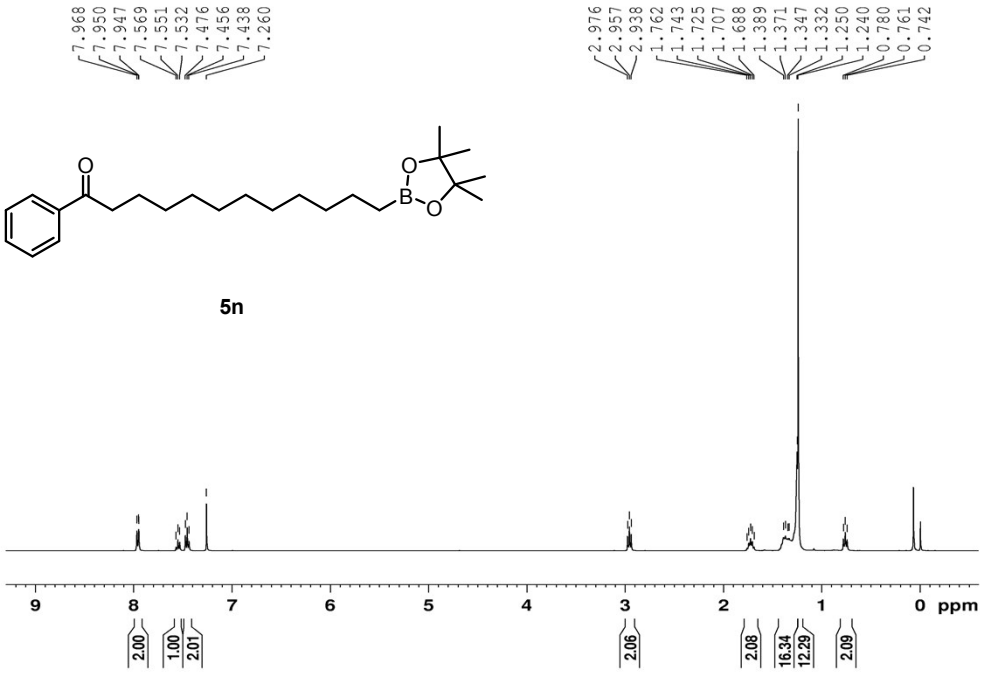
137.084
132.809
128.512
128.033
82.825
77.001
38.616
32.241
29.280
29.208
24.791
24.373
23.936
O
O
B
O
200 180 160 140 120 100 80 60 40 20 0 ppm

5m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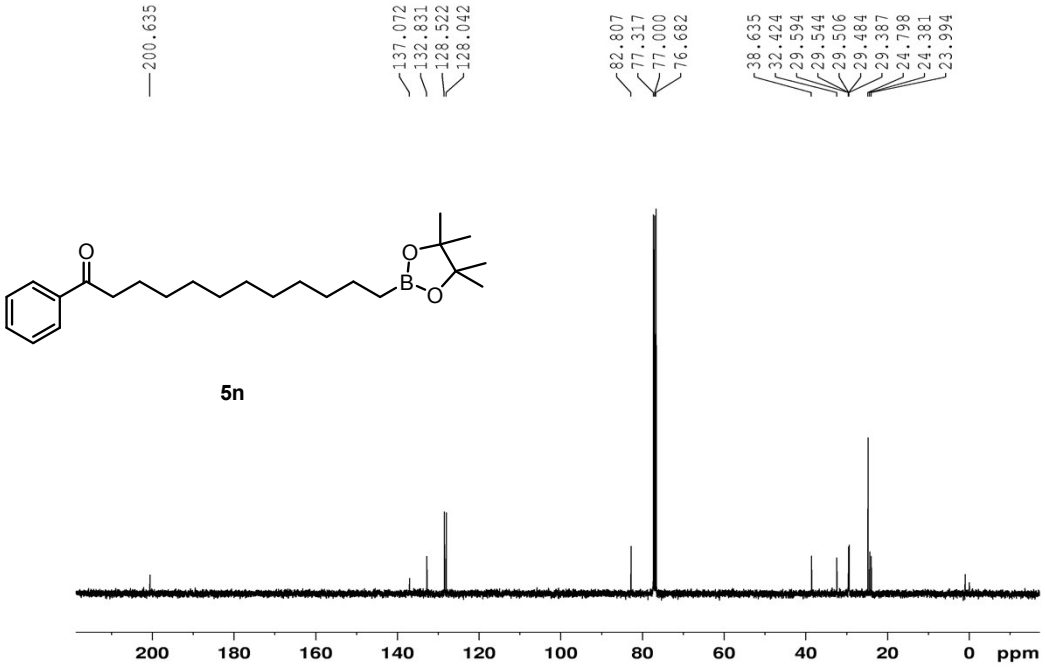
7.569
7.551
7.532
7.476
7.456
7.438
7.260
2.976
2.957
2.938
1.762
1.743
1.725
1.707
1.688
1.389
1.371
1.347
1.332
1.250
1.240
0.780
0.761
0.742
O
O
B
O
9
8
7
6
5
4
3
2
1
0 ppm
2.00
1.00
2.01
2.06
2.08
16.34
12.29
2.09

5n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128.042
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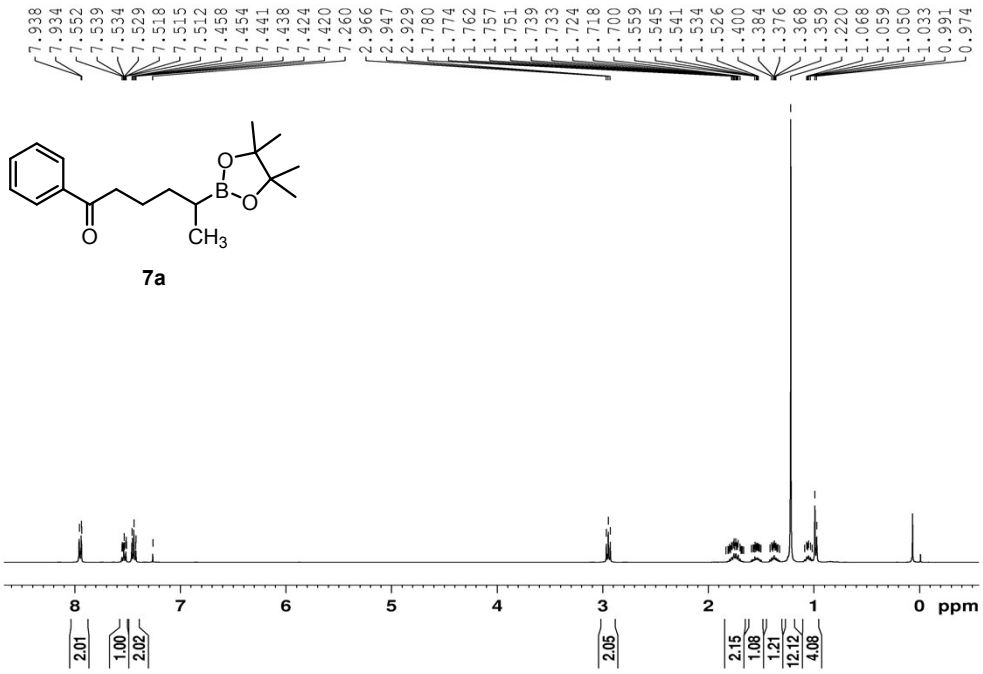
77.317
77.000
76.682
38.635
32.424
29.594
29.544
29.506
29.484
29.387
24.798
24.381
23.994
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0 ppm

5n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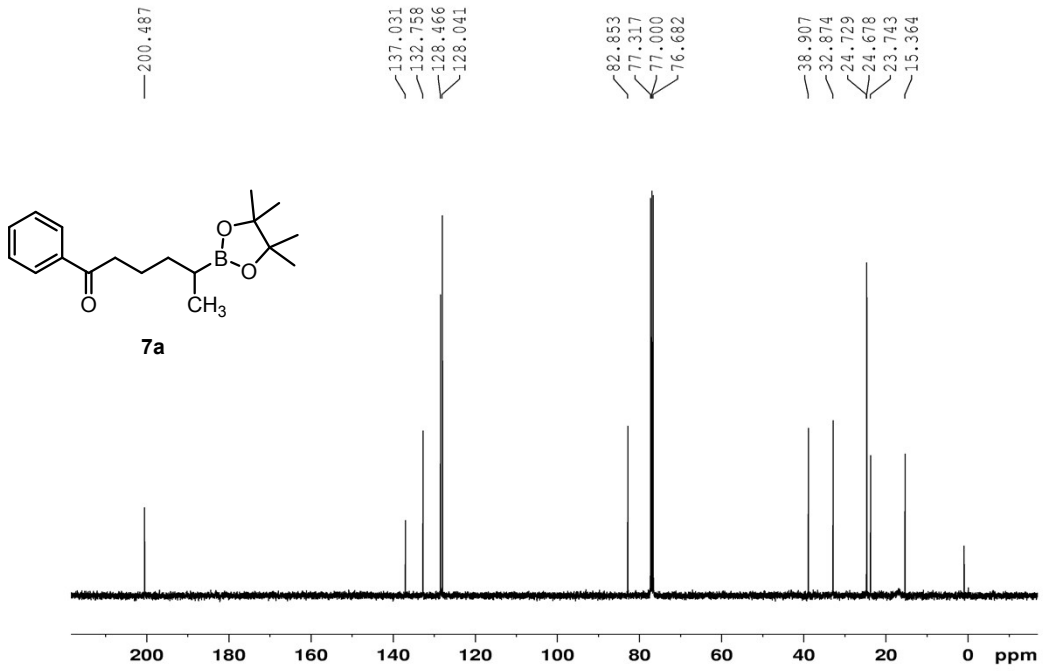
7.552
7.539
7.534
7.529
7.518
7.515
7.512
7.458
7.454
7.441
7.438
7.424
7.420
7.260
2.966
2.947
2.929
1.780
1.774
1.762
1.757
1.751
1.739
1.733
1.724
1.718
1.700
1.559
1.545
1.541
1.534
1.526
1.400
1.384
1.376
1.368
1.359
1.220
1.068
1.059
1.050
1.033
0.991
0.974
O
B
O
CH3
8
7
6
5
4
3
2
1
0 ppm
2.01
1.00
2.02
2.05
2.15
1.08
1.21
12.12
4.08

7a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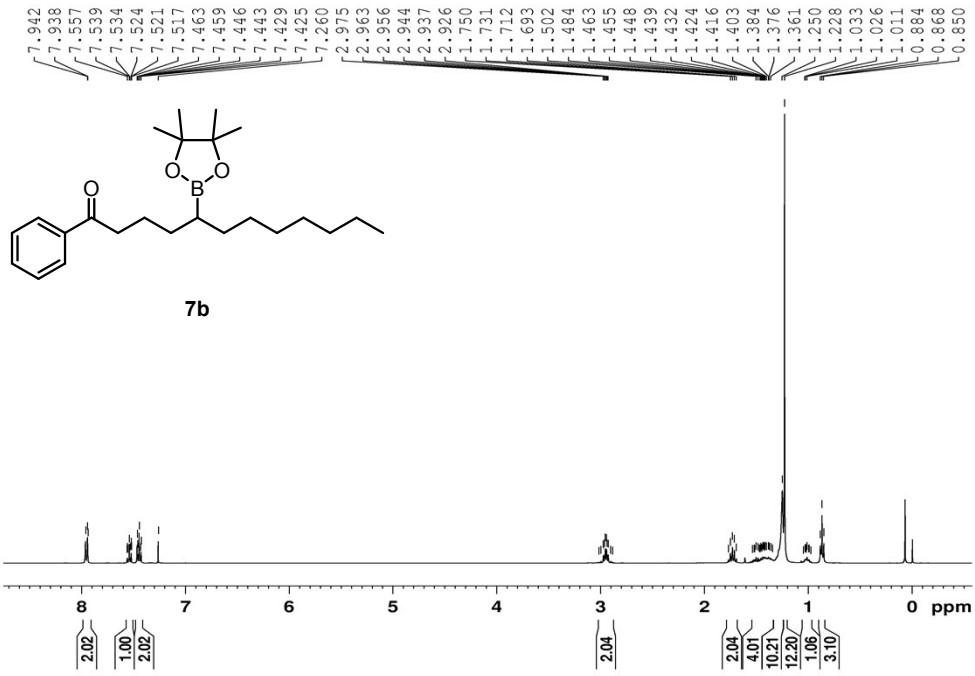
128.041
82.853
77.317
77.000
76.682
38.907
32.874
24.729
24.678
23.743
15.364
O
B
O
CH3
200
180
160
140
120
100
80
60
40
20
0 ppm

7a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7.524
7.521
7.517
7.463
7.459
7.446
7.443
7.429
7.425
7.260
2.975
2.963
2.956
2.944
2.937
2.926
1.750
1.731
1.712
1.693
1.502
1.484
1.463
1.455
1.448
1.439
1.432
1.424
1.416
1.403
1.384
1.376
1.361
1.250
1.228
1.033
1.026
1.011
0.884
0.868
0.850
O
O
B
O
8
7
6
5
4
3
2
1
0 ppm
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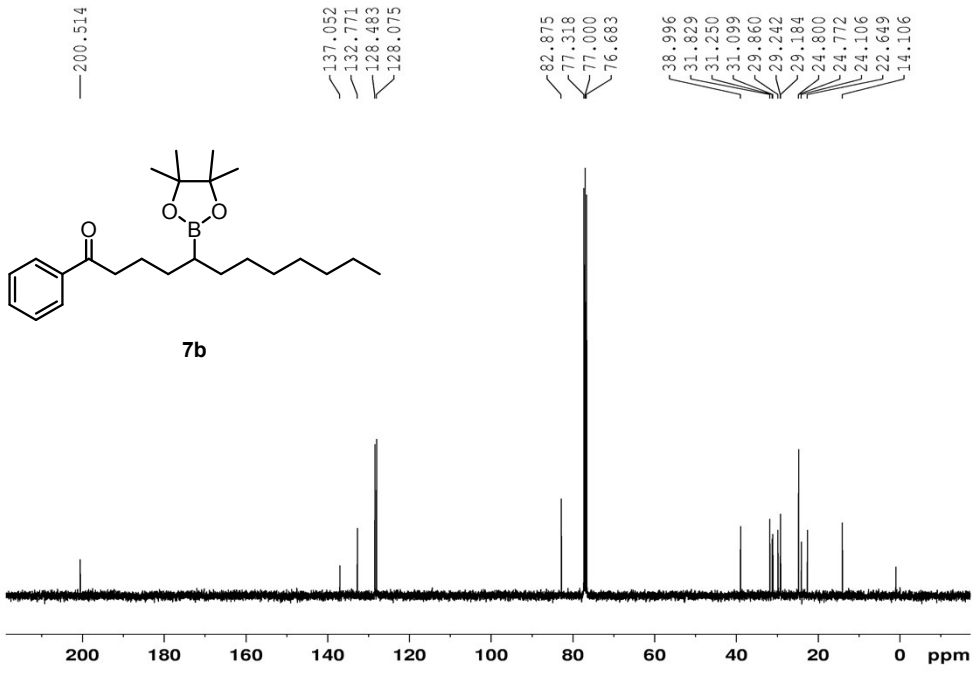
1.00
2.02
2.04
2.04
4.01
10.21
12.20
1.06
3.10

7b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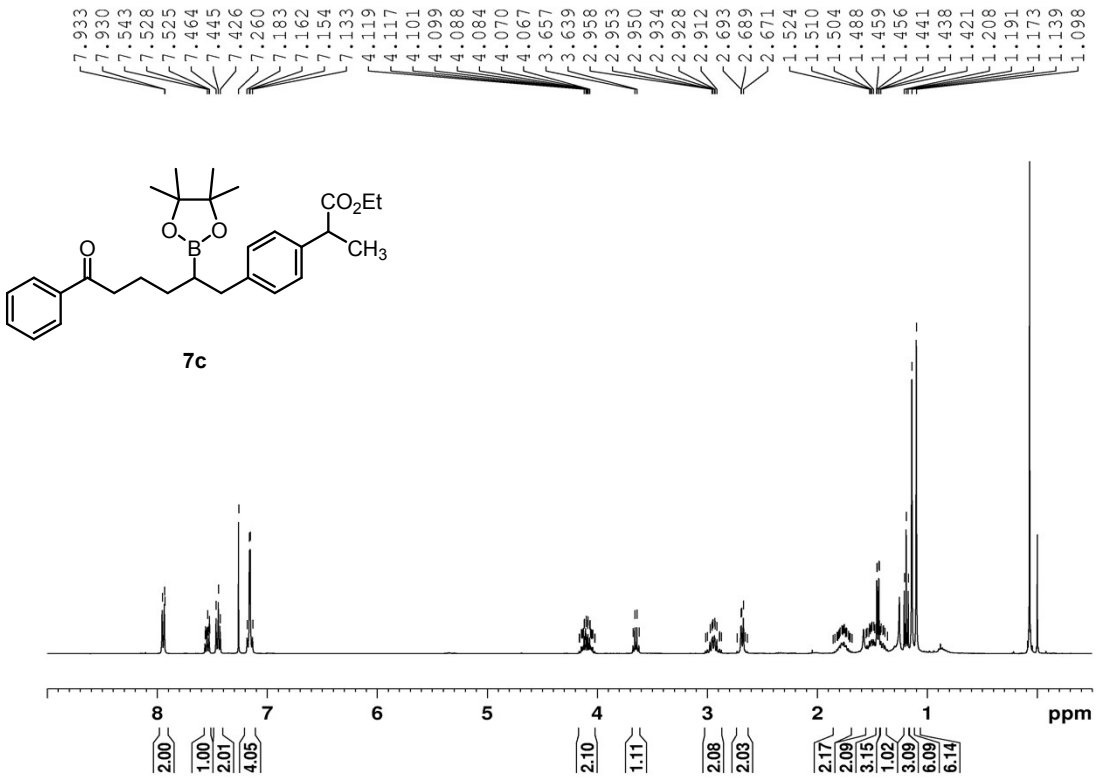
128.075
82.875
77.318
77.000
76.683
38.996
31.829
31.250
31.099
29.860
29.242
29.184
24.800
24.772
24.106
22.649
14.106
O
O
B
O
200
180
160
140
120
100
80
60
40
20
0 ppm

7b


7.933
7.930
7.543
7.528
7.525
7.464
7.445
7.426
7.260
7.183
7.162
7.154
7.133
4.119
4.117
4.101
4.099
4.088
4.084
4.070
4.067
3.657
3.639
2.958
2.953
2.950
2.934
2.928
2.912
2.693
2.689
2.671
1.524
1.510
1.504
1.488
1.459
1.456
1.441
1.438
1.421
1.208
1.191
1.173
1.139
1.098
CO2Et
CH3
O
B
O
O
7c
8
7
6
5
4
3
2
1
ppm
2.00
1.00
2.01
4.05
2.10
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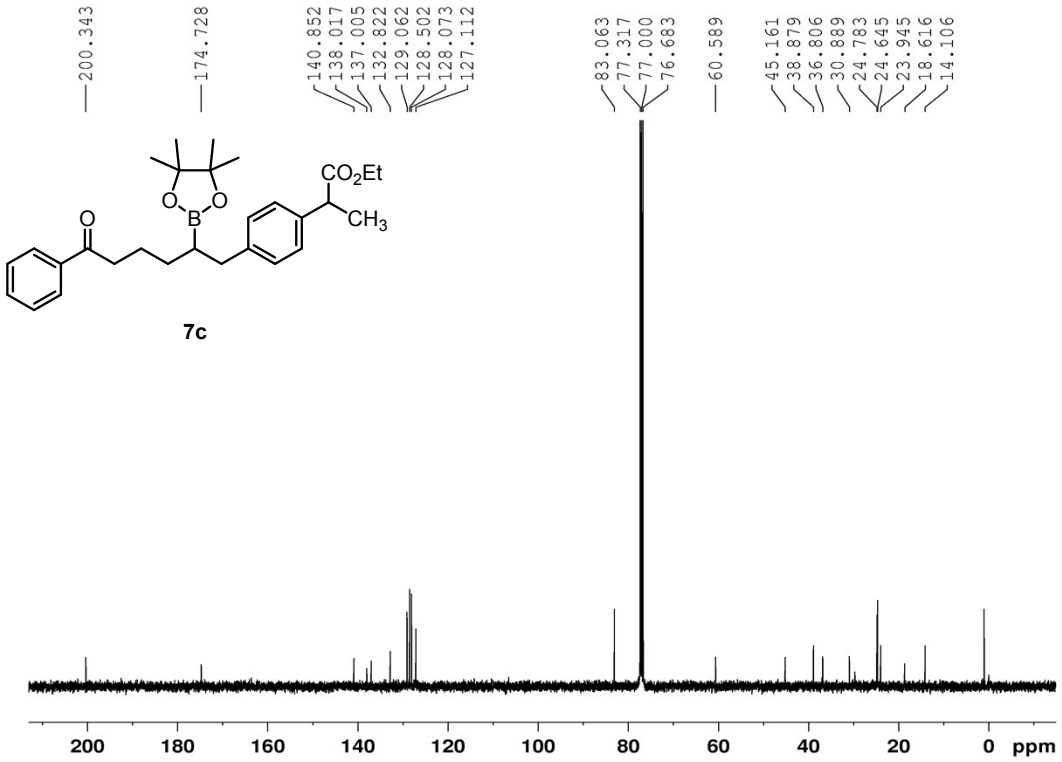
2.08
2.03
2.17
2.09
3.15
1.02
3.09
6.09
6.14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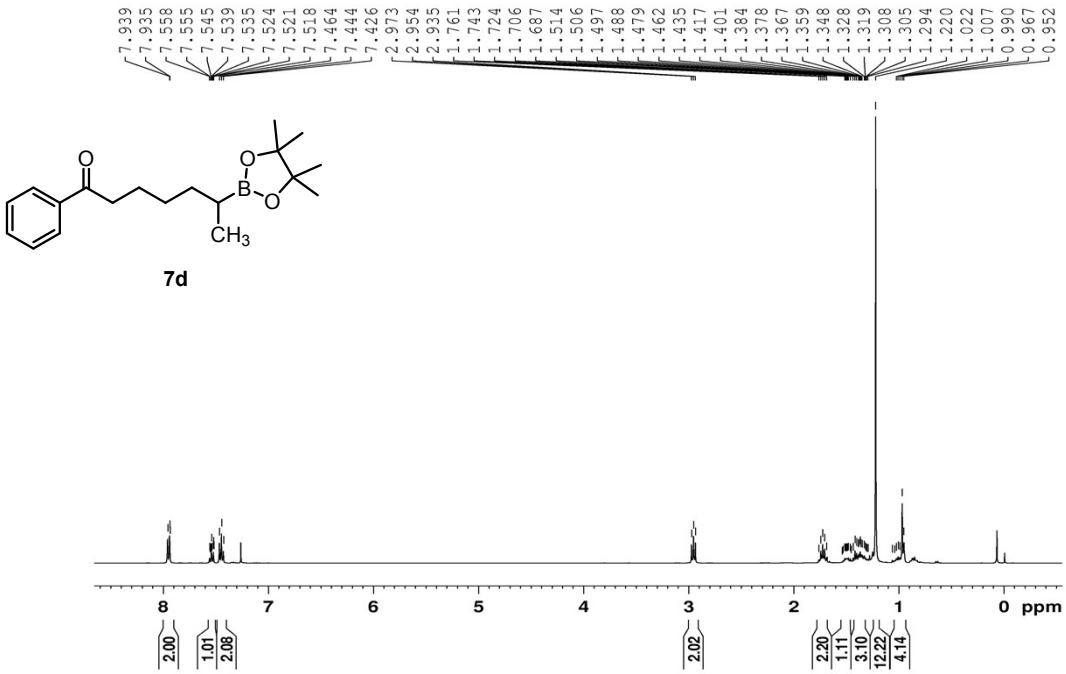
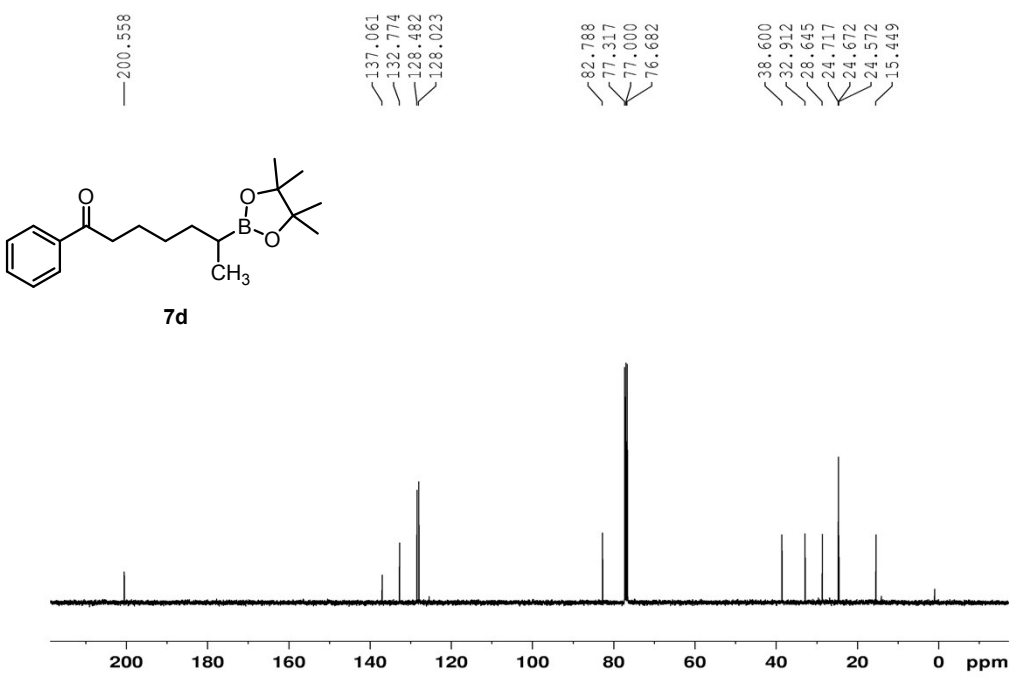
174.728
140.852
138.017
137.005
132.822
129.062
128.502
128.073
127.112
83.063
77.317
77.000
76.683
60.589
45.161
38.879
36.806
30.889
24.783
24.645
23.945
18.616
14.106
CO2Et
CH3
O
B
O
O
7c
200
180
160
140
120
100
80
60
40
20
0
ppm


7.939
7.935
7.558
7.555
7.545
7.539
7.535
7.524
7.521
7.518
7.464
7.444
7.426
2.973
2.954
2.935
1.761
1.743
1.724
1.706
1.687
1.514
1.506
1.497
1.488
1.479
1.462
1.435
1.417
1.401
1.384
1.378
1.367
1.359
1.348
1.328
1.319
1.308
1.305
1.294
1.220
1.022
1.007
0.990
0.967
0.952
O
O
B
O
CH3
8
7
6
5
4
3
2
1
0 ppm
2.00
1.01
2.08
2.02
2.20
1.11
3.10
12.22
4.14

7d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137.061
132.774
128.482
128.023
82.788
77.317
77.000
76.682
38.600
32.912
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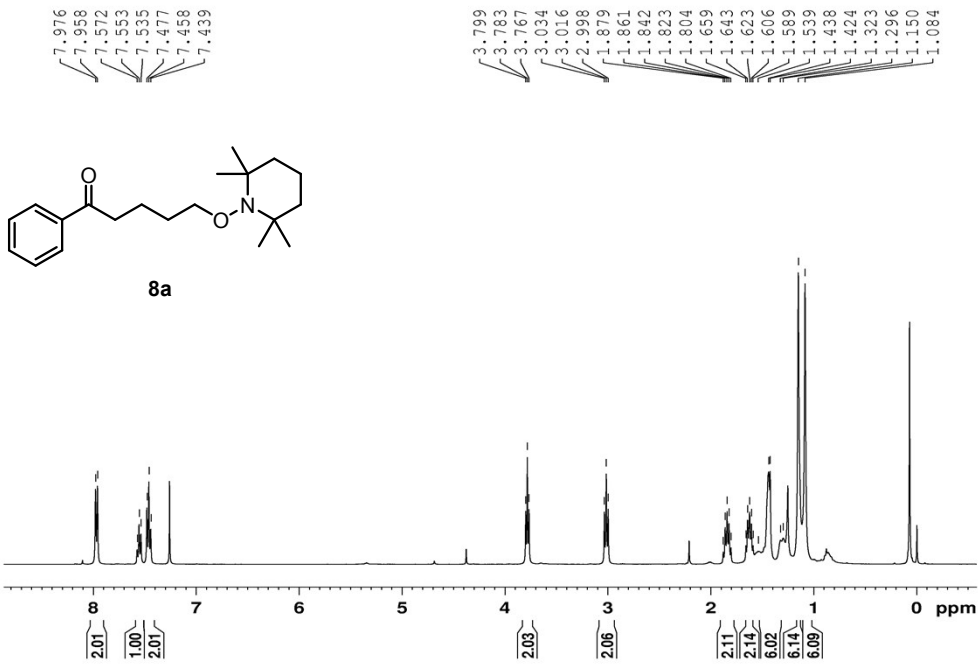
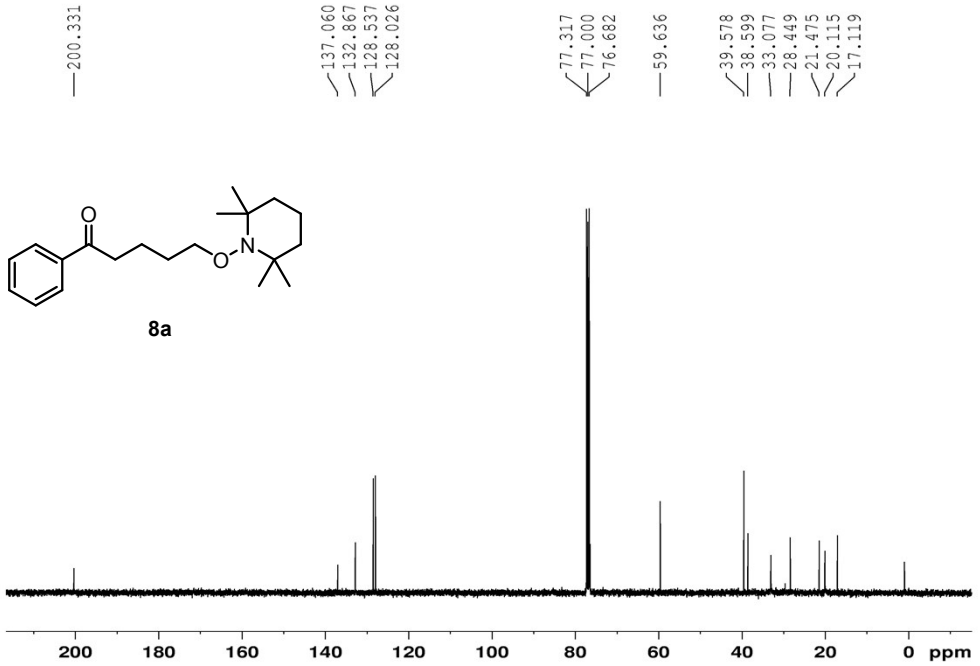
24.717
24.672
24.572
15.449
O
O
B
O
CH3
200
180
160
140
120
100
80
60
40
20
0 ppm

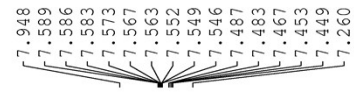
7d

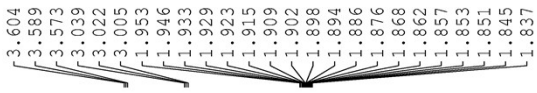
^{1}H NMR and ^{13}C NMR Spectra of the Radical Adduct **8a**

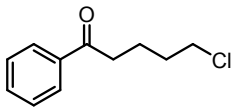




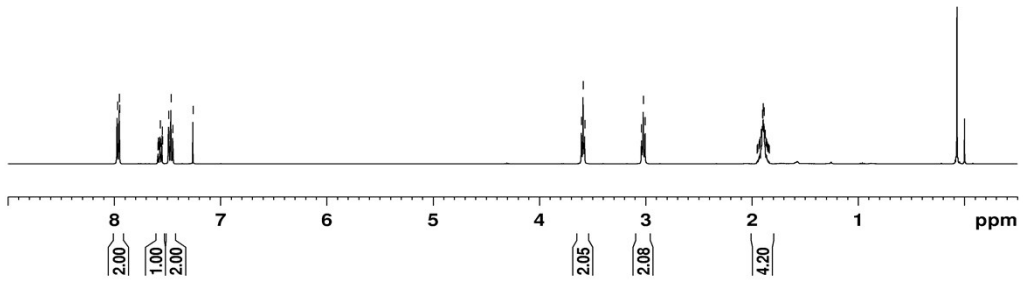
^{1}H NMR and ^{13}C NMR Spectra of **9a**

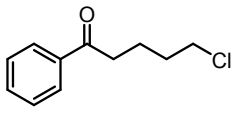






9a





9a