

Supporting Information

Magnesium-catalyzed hydroboration of organic carbonates, carbon dioxide and esters

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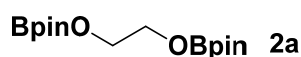
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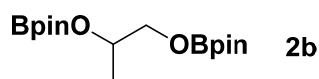
References

General Information. All reactions were performed under an atmosphere of nitrogen using glovebox technique. ^1H , $^{13}\text{C}\{^1\text{H}\}$, and $^{11}\text{B}\{^1\text{H}\}$ NMR spectra were recorded at 25 °C on Bruker Avance III 600 MHz or 400 MHz spectrometer in deuterated solvents and were referenced to the resonances of the solvent used. The following abbreviations are used; s: singlet, d: doublet, t: triplet, q: quartet, m: multiplet. All commercially available chemicals were purchased from Sigma-Aldrich or Alfa Aesar, and used without further purification. The compounds $[(^{\text{Ar}}\text{Nacnac})\text{Mg}]_2$ (Ar = Dipp, Mes, Xyl, DippXyl) and $[(^{\text{Xyl}}\text{Nacnac})\text{MgI}(\text{OEt}_2)]$ were prepared by literature procedures.^{S1-4}

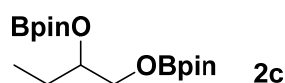
General Procedure for Hydroboration of Carbonates. Carbonates (0.40 mmol), HBpin (1.3 mmol) and catalyst **1** (1 mol%) were placed in a 10 ml Schlenk flask equipped with a magnetic stir bar inside the glove box. The reaction mixture was stirred for 6 h at 25 °C, and then subjected to NMR analysis. The progress of the reaction was monitored by ^1H NMR, ^{13}C NMR, and ^{11}B NMR, which indicated the completion of the reaction by the disappearance of the carbonate proton and appearance of new products and CH_3OBpin .



^1H NMR (CDCl_3): δ 3.86 (s, 4H, OCH_2), 1.17 (s, 24H, BOCMe_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3): δ 82.65 (BOCMe_2), 64.98 (OCH_2), 24.54 (BOCMe_2). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3): δ 22.22.

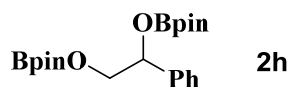


^1H NMR (CDCl_3): δ 4.22-4.17 (m, 1H, CH), 3.66 (d, $^3J_{\text{HH}} = 4.2$ Hz, 2H, OCH_2), 1.17 (s, 24H, BOCMe_2), 1.09 (d, $^3J_{\text{HH}} = 6.0$ Hz, 3H, Me). $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3): δ 82.66, 82.62 (BOCMe_2), 70.27 (OCH_2), 69.06 (CH), 24.55, 24.48 (BOCMe_2), 18.41 (Me). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3): δ 22.22.

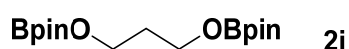


^1H NMR (CDCl_3): δ 3.99-3.80 (m, 1H, CH), 3.77-3.65 (m, 2H, OCH_2), 1.49-1.36 (m,

24.54, 24.48 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.21.



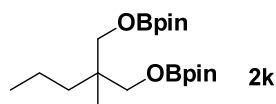
¹H NMR (CDCl₃): δ 7.28-7.04 (m, 4H, *Ar-H*), 5.22 (dd, *J*_{HH} = 8.0, 4.1 Hz, 1H, *CH*), 3.89-3.88 (m, 2H, *CH*₂), 1.06 (s, 24H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 128.09, 127.94, 127.69, 127.45 (*Ar-C*), 82.62, 82.50 (BOCMe₂), 77.07 (*CH*), 68.69(OCH₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.38.



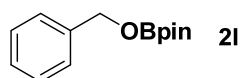
¹H NMR (CDCl₃): δ 3.85 (t, ³*J*_{HH} = 6.6 Hz, 4H, OCH₂), 1.78-1.75 (m, 2H, *CH*₂), 1.18 (s, 24H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.65 (BOCMe₂), 61.48 (OCH₂), 33.23 (*CH*₂), 24.68 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.22.



¹H NMR (CDCl₃): δ 3.82 (q, *J*_{HH} = 4.7, 4H, OCH₂), 1.17 (s, 24H, BOCMe₂), 1.14 (s, 6H, *Me*). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.62 (BOCMe₂), 70.32 (OCH₂), 36.70 (*C*(Me)₂), 24.60 (BOCMe₂), 20.87 (*Me*). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.35.

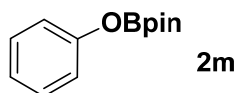


¹H NMR (CDCl₃): δ 3.58 (d, ³*J*_{HH} = 8.4 Hz, 4H, OCH₂), 1.91 (s, 4H, *CH*₂), 1.17-1.16 (24H, BOCMe₂, *CH*₂), 0.79 (t, ³*J*_{HH} = 6.6 Hz, 3H, *CH*₂Me), 0.75 (s, 3H, *CMe*). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.46 (BOCMe₂), 68.71 (OCH₂), 38.99 (*CMe*), 35.92 (*CCH*₂), 24.48 (BOCMe₂), 18.21 (*CMe*), 16.32 (*CH*₂Me), 14.83 (*CH*₂Me). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.24.



¹H NMR (CDCl₃): δ 7.26-7.15 (m, 5H, *Ar-H*), 4.84 (s, 2H, OCH₂), 1.16 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 139.14, 128.26, 127.20, 126.56 (*Ar-C*),

82.91 (BOCMe₂), 66.66 (OCH₂), 24.60 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.35.



¹H NMR (CDCl₃): δ 7.31-7.16 (m, 2H, Ar-H), 7.00-6.94 (m, 3H, Ar-H), 1.19 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 153.46, 129.24, 123.01, 119.48 (Ar-C), 83.43 (BOCMe₂), 24.58 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.23.



¹H NMR (CDCl₃): δ 3.84 (q, ³J_{HH} = 7.2 Hz, 2H, OCH₂), 1.17 (s, 12H, BOCMe₂), 1.15 (t, ³J_{HH} = 7.2 Hz, 3H, Me). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.44 (BOCMe₂), 60.49 (OCH₂), 24.50 (BOCMe₂), 17.11 (Me). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.07.

MeOBpin 2p

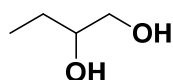
¹H NMR (CDCl₃): δ 3.46 (s, 3H, Me), 1.18 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.72 (BOCMe₂), 52.54 (Me), 24.53 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.24.



¹H NMR (CDCl₃): δ 5.89-5.80 (m, 1H, CH=CH₂), 5.22-5.02 (m, 2H, CH=CH₂), 5.05-5.02 (m, 2H, CH₂), 3.52 (s, 3H, OCH₃), 1.17 (s, 24H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 135.35 (CH=CH₂), 114.88 (CH=CH₂), 82.74, 82.67 (BOCMe₂), 76.75 (CH), 65.44 (OCH₂), 24.54, 24.48 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.23.

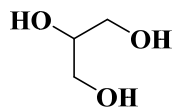
A Typical Procedure for the Synthesis of Boronate Esters and Their Hydrolysis to Alcohols

Catalyst **1** (1% mol), 4-ethyl-1,3-dioxolan-2-one (**2c**, 0.40 mmol) and HBpin (1.3 mmol) were placed in an oven-dried 10 ml Schlenk tube. The mixture was stirred at 25 °C for 6 h, and then subjected to NMR analysis. The crude product was purified by flash column chromatography on silica gel with acetate/hexane (1/4) as eluents to give the alcohol, 1, 2-butanediol (34 mg, 0.38 mmol, 94% yield).



^1H NMR (CDCl_3): δ 4.11 (s, 1H, CHOH), 4.01 (s, 1H, CH_2OH), 3.61 (d, $^3J_{\text{HH}} = 10.2$ Hz, 2H, CH_2OH), 3.43-3.40 (m, 1 H, CHOH), 1.48-1.43 (m, 2H, CH_2Me), 0.95 (t, $^3J_{\text{HH}} = 7.8$ Hz, 3H, CH_2Me). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 73.77 (CHOH), 66.31 (CH_2OH), 26.03 (CH_2Me), 9.90 (CH_2Me).

Catalyst **1** (1% mol), 4-(hydroxymethyl)-1,3-dioxolan-2-one (**2f**, 0.40 mmol) and HBpin (1.68 mmol) were placed in an oven-dried 10 ml Schlenk tube. The mixture was stirred at 25 °C for 6 h, and then subjected to NMR analysis. The crude product was hydrolyzed with 6 M HCl (10 ml). The aqueous phase was washed with diethyl ether (3 x 10 ml) and the combined organic phases were washed with water (10 ml). The combined organic phases were concentrated and the crude product was purified by flash chromatography (SiO_2 , $\text{DCM}:\text{MeOH}$ 9:1–2:1). The product was obtained as a colourless, viscous oil (92% yield).



^1H NMR (D_2O): δ 3.71 (ddd, $J_{\text{HH}} = 10.8$, 1 H, CHOH), 3.56 (dd, $J_{\text{HH}} = 11.4$, 2H, CH_2OH), 3.45 (d, $^3J_{\text{HH}} = 11.2$ Hz, 2 H, CH_2OH). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, D_2O): δ 72.03, 62.4.

Table S1. Optimization of Esters Hydroboration

$$\text{CH}_3\text{CO}_2\text{CH}_2\text{CH}_3 + 2 \text{HBpin} \xrightarrow[\text{rt}]{\text{Mg(I) 1}} 2 \text{CH}_3\text{CH}_2\text{OH} + \text{BpinOCH}_2\text{CH}_3$$

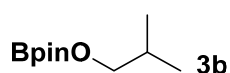
Entry	Cat	Cat. Loading (mol%)	Sol	Time (h)	Yield ^a (%)
1	-	-	C ₆ D ₆	1	0
2	-	-	C ₆ D ₆	15	0 ^b
3	1	5	C ₆ D ₆	<0.17	99
4	1	1	C ₆ D ₆	0.67	99
5	1	1	-	1	99
6	1	0.1	-	3	77
7	1	0.1	-	5	99

^aThe reaction was monitored by ¹H NMR spectroscopy. ^b70 °C.

General Procedure for Hydroboration of Esters. In a glove box, catalyst **1** (1 mol%) was added to a solution of esters (1.0 mmol) and HBpin (2.0 mmol) in a 10 ml Schlenk flask. The reaction mixture was stirred at 25 °C for 1 h, and then subjected to NMR analysis. The progress of the reaction was monitored by ¹H NMR, ¹³C NMR, and ¹¹B NMR, which indicated the completion of the reaction by the disappearance of the ester proton and appearance of new products.



¹H NMR (CDCl₃): δ 3.89 (q, ³J_{HH} = 7.2 Hz, 2H, MeCH₂OBpin), 1.23 (t, ³J_{HH} = 7.2 Hz, 3H, MeCH₂OBpin), 1.20 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 82.36 (BOCMe₂), 60.69 (MeCH₂OBpin), 24.74 (BOCMe₂), 17.52 (MeCH₂OBpin). ¹¹B{¹H} NMR (193 MHz, CDCl₃): δ 22.48.

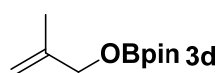


¹H NMR (C₆D₆): δ 3.69 (d, ³J_{HH} = 6.6 Hz, 2H, CH₂OBpin), 1.77 (d, ³J_{HH} = 6.6 Hz, CHMe₂), 1.07 (s, 12H, BOCMe₂), 0.83 (d, ³J_{HH} = 6.6 Hz, 6H, CHMe₂). ¹³C{¹H} NMR

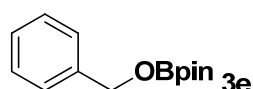
(151 MHz, C₆D₆): δ 82.36 (BOCMe₂), 71.52 (CH₂OBpin), 30.31 (CHMe₂), 24.75 (BOCMe₂), 18.95 (CHMe₂). ¹¹B{¹H} NMR (193 MHz, C₆D₆): δ 22.51.

Br-(CH₂)₅-OBpin 3c

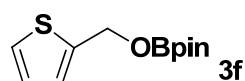
¹H NMR (C₆D₆): δ 3.84 (t, ³J_{HH} = 7.2 Hz, 2H, CH₂OBpin), 2.92 (t, ³J_{HH} = 7.2 Hz, 2H, (CH₂)₅), 1.46-1.37 (m, 6H, (CH₂)₅), 1.10-1.09 (m, 2H, (CH₂)₅), 1.07 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, C₆D₆): δ 82.35 (BOCMe₂), 64.84 (CH₂OBpin), 33.58, 32.99, 31.69, 28.02, 25.11 ((CH₂)₅), 24.96 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, C₆D₆): δ 22.50.



¹H NMR (C₆D₆): δ 5.12 (s, 1H, C=CH₂), 4.80-4.79 (m, C=CH₂), 4.30 (s, 2 H, CH₂OBpin), 1.55 (s, 3 H, MeC=CH₂), 1.05 (s, 12 H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, C₆D₆): δ 143.31 (C=CH₂), 110.14 (C=CH₂), 83.16 (BOCMe₂), 68.49 (CH₂OBpin), 24.95, 24.74 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, C₆D₆): δ 22.66.



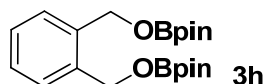
¹H NMR (C₆D₆): δ 7.30 (d, ³J_{HH} = 7.2 Hz, 2H, Ar-H), 7.13 (t, ³J_{HH} = 7.2 Hz, 2H, Ar-H), 7.05 (t, ³J_{HH} = 7.2 Hz, 1H, Ar-H), 4.94 (s, 2H, CH₂OBpin), 1.04 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (151 MHz, C₆D₆): δ 140.04, 130.02, 128.76, 128.58, 127.57, 127.04 (Ar-C), 82.75 (BOCMe₂), 66.94 (CH₂OBpin), 24.70 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, C₆D₆): δ 22.75.



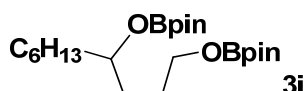
¹H NMR (C₆D₆): δ 6.91-6.85 (m, 1H, C₄H₃S), 6.82-6.81 (m, 1H, C₄H₃S), 6.68-6.66 (m, 1H, C₄H₃S), 5.00 (s, 1H, CH₂OBpin), 1.04 (s, 12H, BOCMe₂). ¹³C{¹H} NMR (175 MHz, C₆D₆): δ 142.80, 126.76, 126.08, 125.67 (C₄H₃S), 82.91 (BOCMe₂), 61.84 (CH₂OBpin), 24.70 (BOCMe₂). ¹¹B{¹H} NMR (193 MHz, C₆D₆): δ 22.51.



^1H NMR (C_6D_6): δ 4.45-4.42 (m, 1H, MeCH), 3.80 (d, $^3J_{\text{HH}} = 6.0$ Hz, 2H, CH_2OBpin), 1.10, 1.07 (s, 12H, BOCMe_2), 1.05-1.04 (m, 3H, MeCH). $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, C_6D_6): δ 82.59, 82.44 (BOCMe_2), 70.72 (MeCH), 69.56 (CH_2OBpin), 24.81, 24.78, 24.74, 24.67 (BOCMe_2), 18.59 (MeCH). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, C_6D_6): δ 22.24.



^1H NMR (C_6D_6): δ 7.51-7.49 (m, 2H, Ar-H), 7.10-7.07 (m, 2H, Ar-H), 5.06 (s, 4H, Ar- CH_2OBpin), 1.03 (s, 24H, BOCMe_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, C_6D_6): δ 137.31, 127.80, 127.74 (Ar-C), 82.73 (BOCMe_2), 64.52 (Ar- CH_2OBpin), 24.69 (BOCMe_2). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, C_6D_6): δ 22.51.

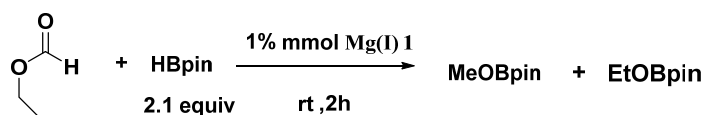


^1H NMR (C_6D_6): δ 4.22-4.18 (m, 1H, $\text{CHCH}_2\text{CH}_2\text{CH}_2$), 3.97-3.93 (m, 2H, $\text{CHCH}_2\text{CH}_2\text{CH}_2$), 1.80-1.66 (m, 2H, $\text{CHCH}_2\text{CH}_2\text{CH}_2$), 1.58-1.55 (m, 2H, $\text{CHCH}_2\text{CH}_2\text{CH}_2$), 1.36-1.33 (m, 2H, C_6H_{13}), 1.26-1.17 (m, 8H, C_6H_{13}), 1.07, 1.06 (s, 12H, BOCMe_2), 0.86 (t, $^3J_{\text{HH}} = 7.2$ Hz, 3 H, C_6H_{13}). $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, C_6D_6): δ 82.38, 82.22 (BOCMe_2), 65.02 ($\text{CHCH}_2\text{CH}_2\text{CH}_2$), 37.06 ($\text{CHCH}_2\text{CH}_2\text{CH}_2$), 33.12 ($\text{CHCH}_2\text{CH}_2\text{CH}_2$), 32.28 ($\text{CHCH}_2\text{CH}_2\text{CH}_2$), 29.62, 28.20, 25.94, 24.75 (C_6H_{13}), 24.70, 24.66 (BOCMe_2), 23.06, 14.36 (C_6H_{13}). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, C_6D_6): δ 22.29.

General Procedure for Hydroboration of CO_2

In a glove box, an oven-dried 10 ml Schlenk flask charged with mesitylene (0.50 mmol), HBpin (0.50 mmol) and catalyst **1** (5% mmol) was evacuated and refilled with CO_2 gas (1 atm) for three cycles, The resulting mixture was stirring at 100 °C for 15 h, and then subjected to NMR analysis.

Scheme S1: Mechanistic control experiments

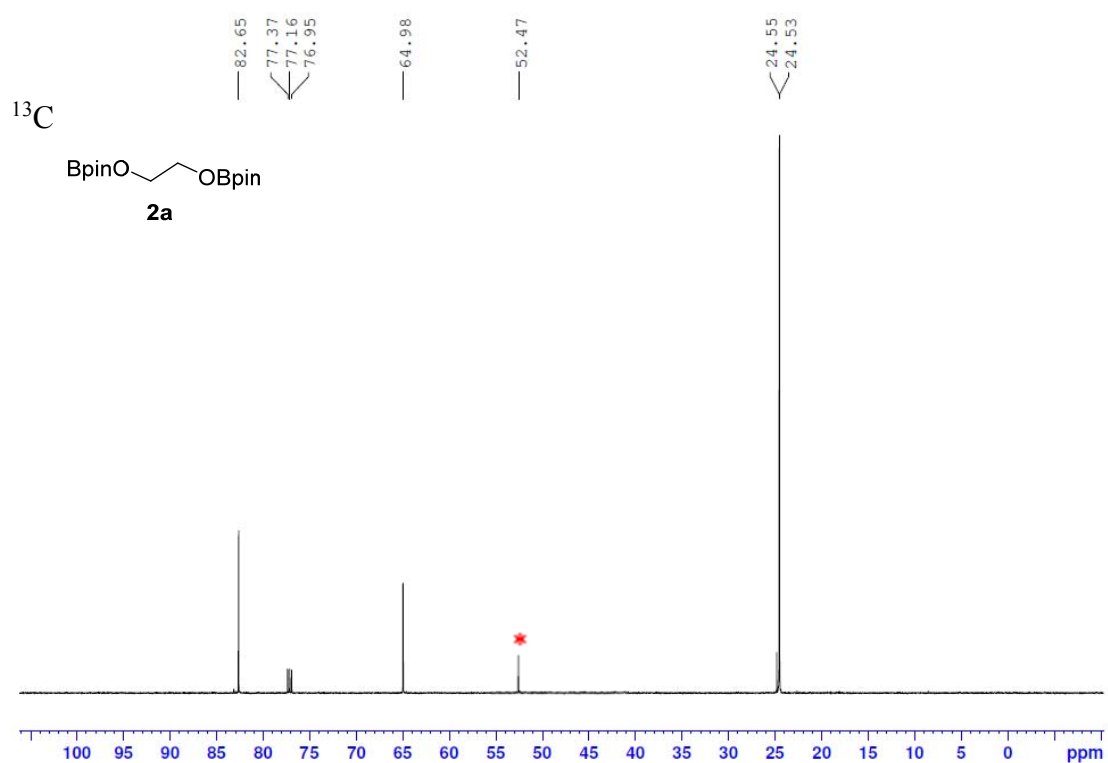
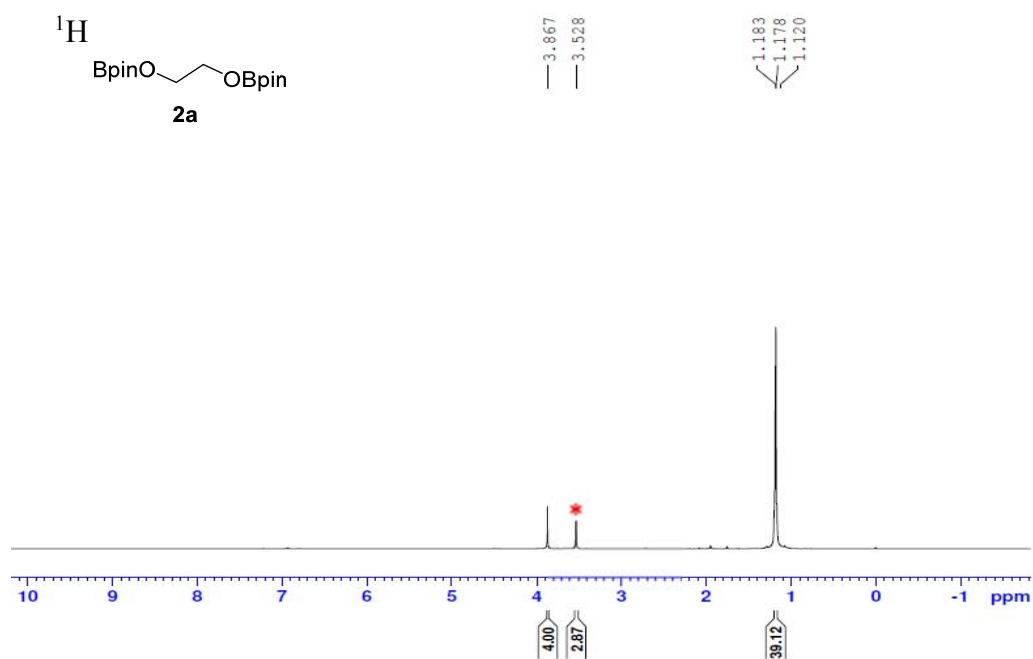


A: Mg(I) 1 (1% mmol): 99% conv.

B: No catalyst: trace

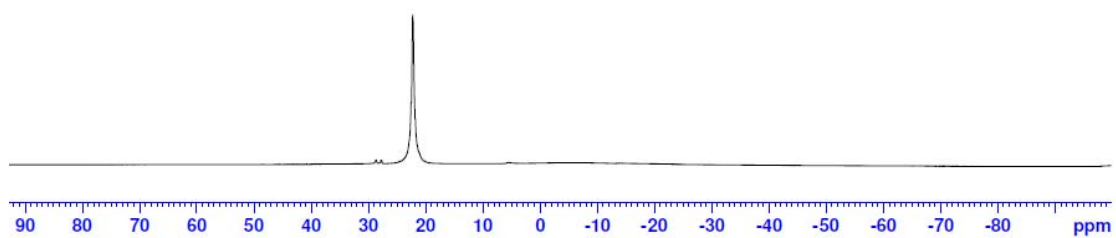
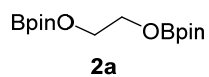
When a reaction of ethyl formate, postulated as an intermediate, was carried out with Mg(I)-HBpin catalytic system, the usually observed reaction products were present in the crude mixture (A). Without the catalyst **1**, however, the reduction does not proceed, as unreacted starting material and HBpin were still present in the reaction mixture, and no conversion was observed (B). These results suggest that the presence of active Mg(I) species is required for the reaction to take place in each catalytic cycle.

^1H , ^{13}C and ^{11}B NMR Spectra [MeOBpin (*), excess HBpin (∇)]

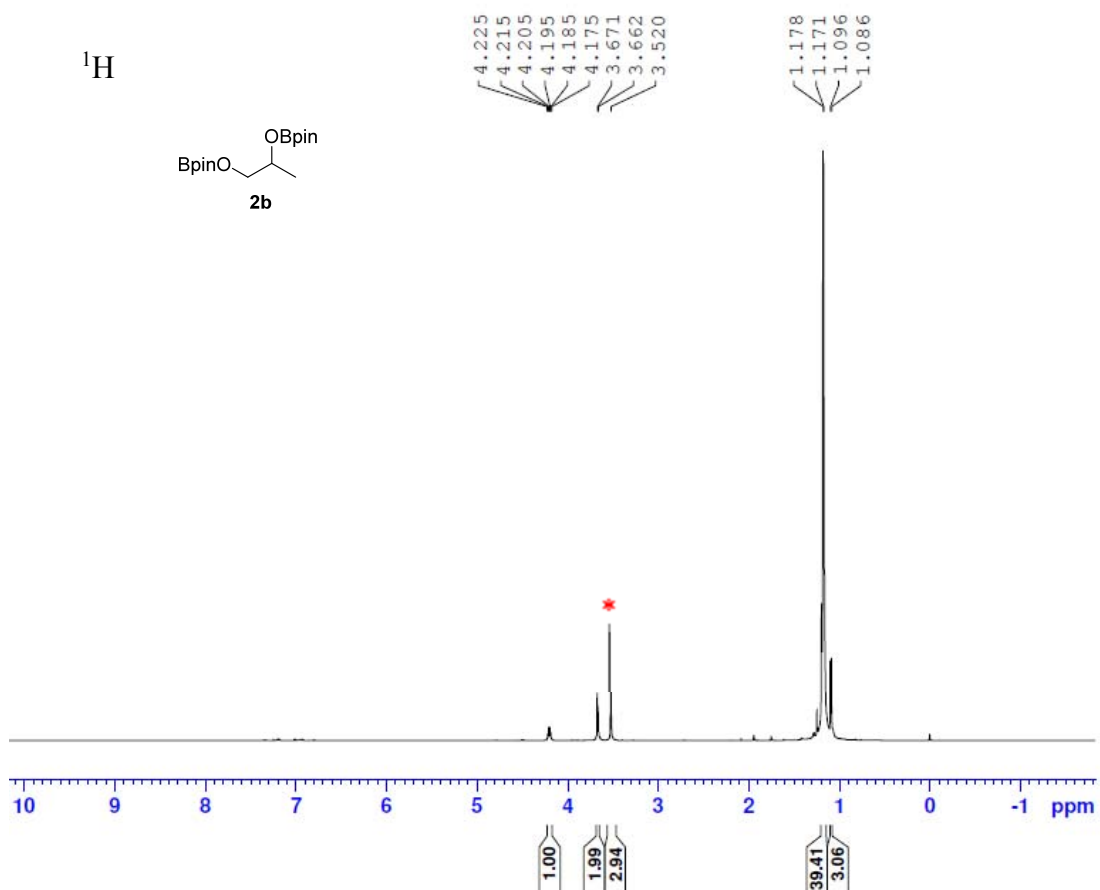
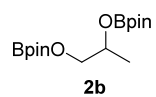


^{11}B

—22.24

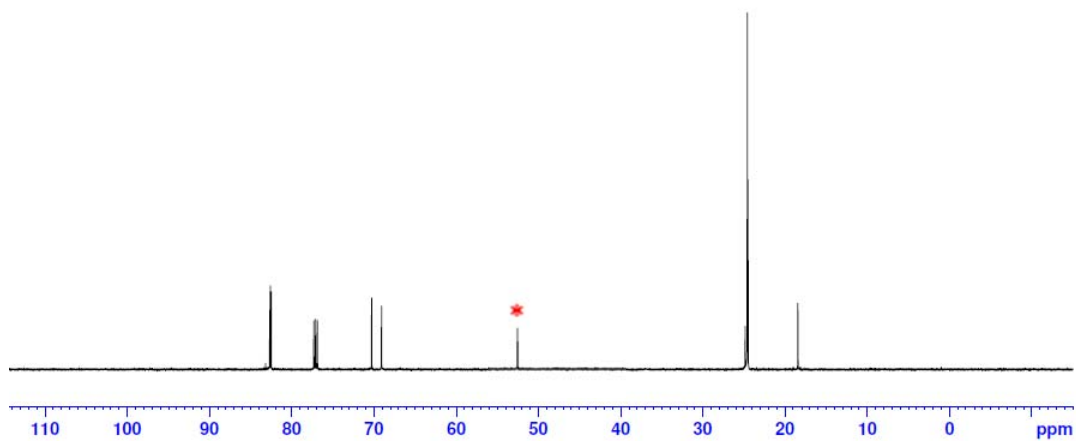
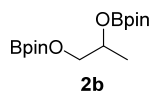


^1H

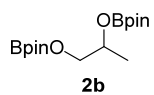


^{13}C

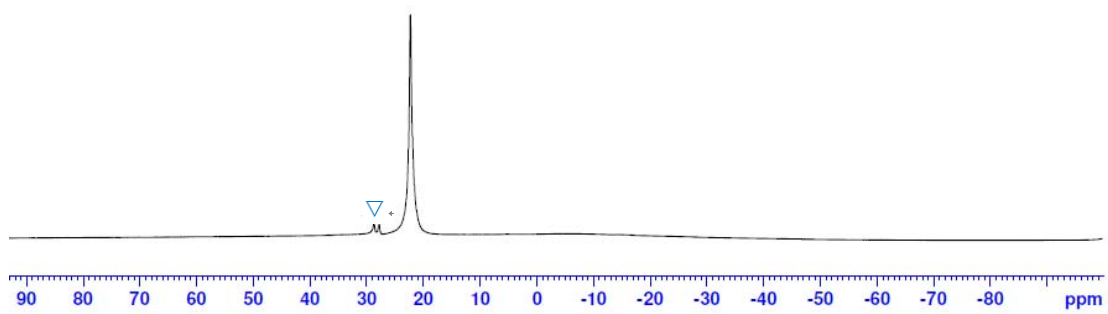
82.66
82.62
82.49
77.29
77.08
76.87
70.27
69.06
— 52.49
24.56
24.54
24.49
24.48
— 18.41

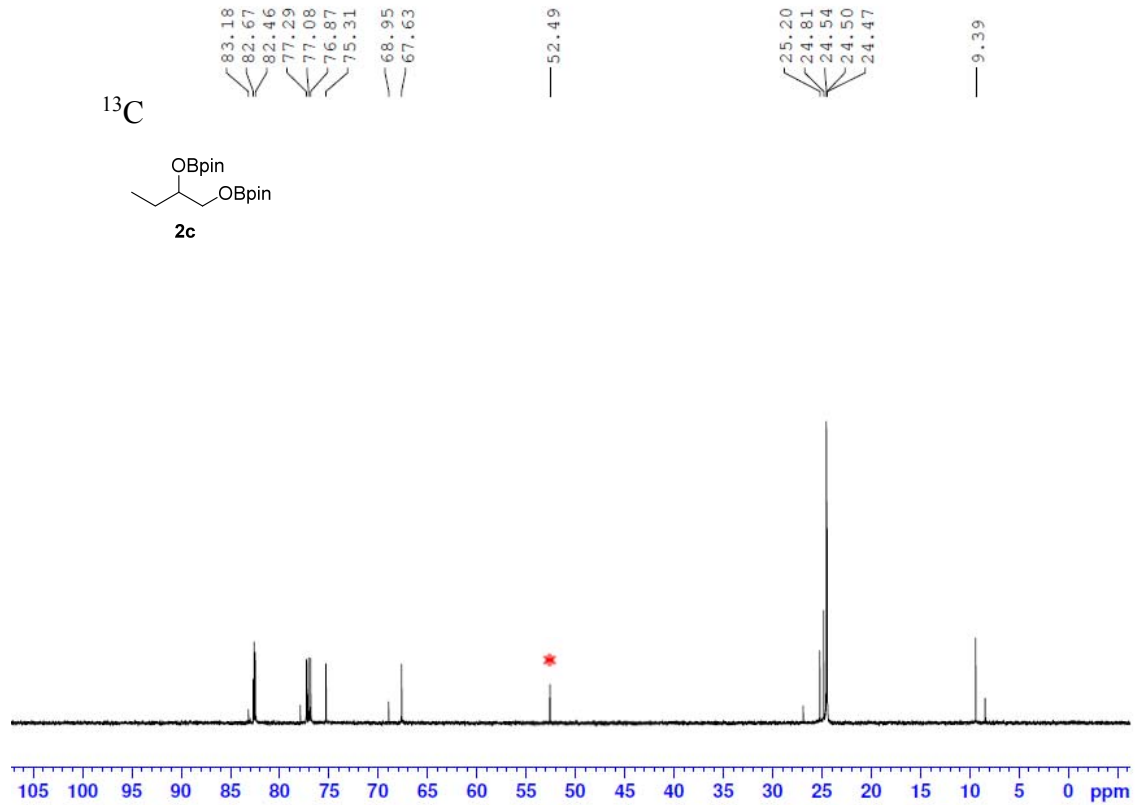
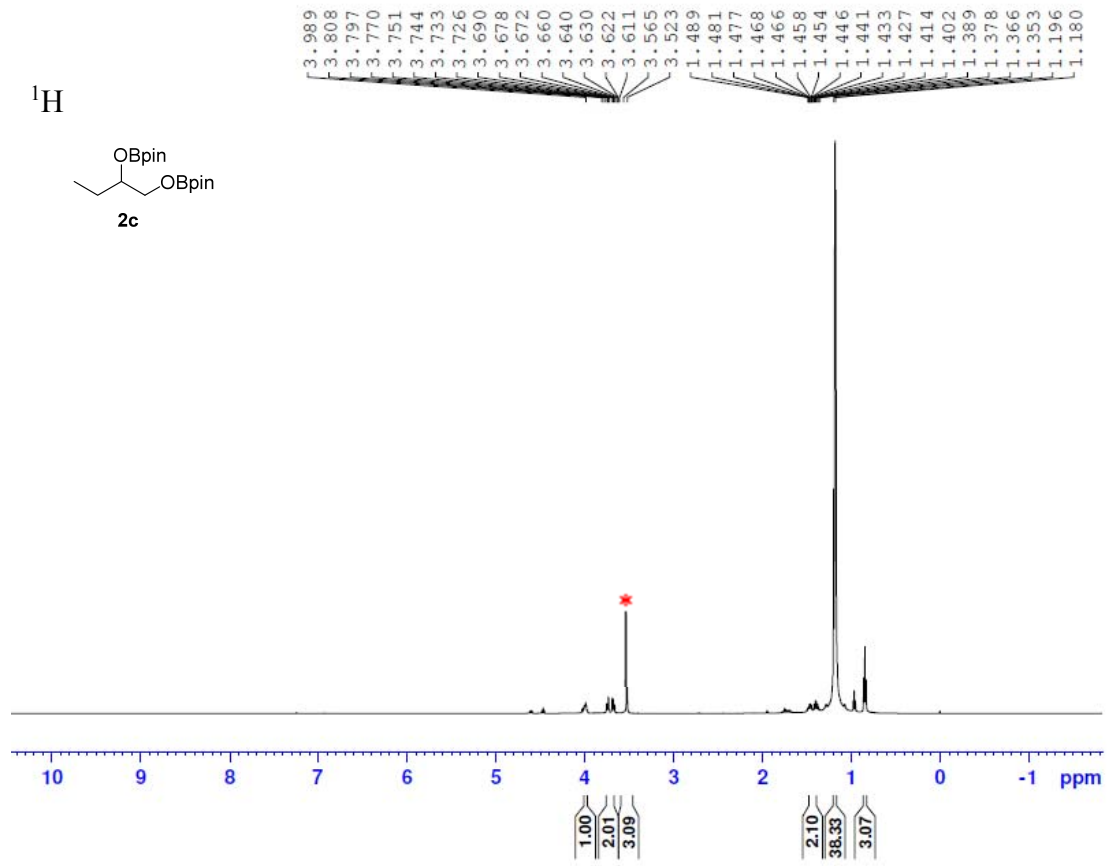


^{11}B

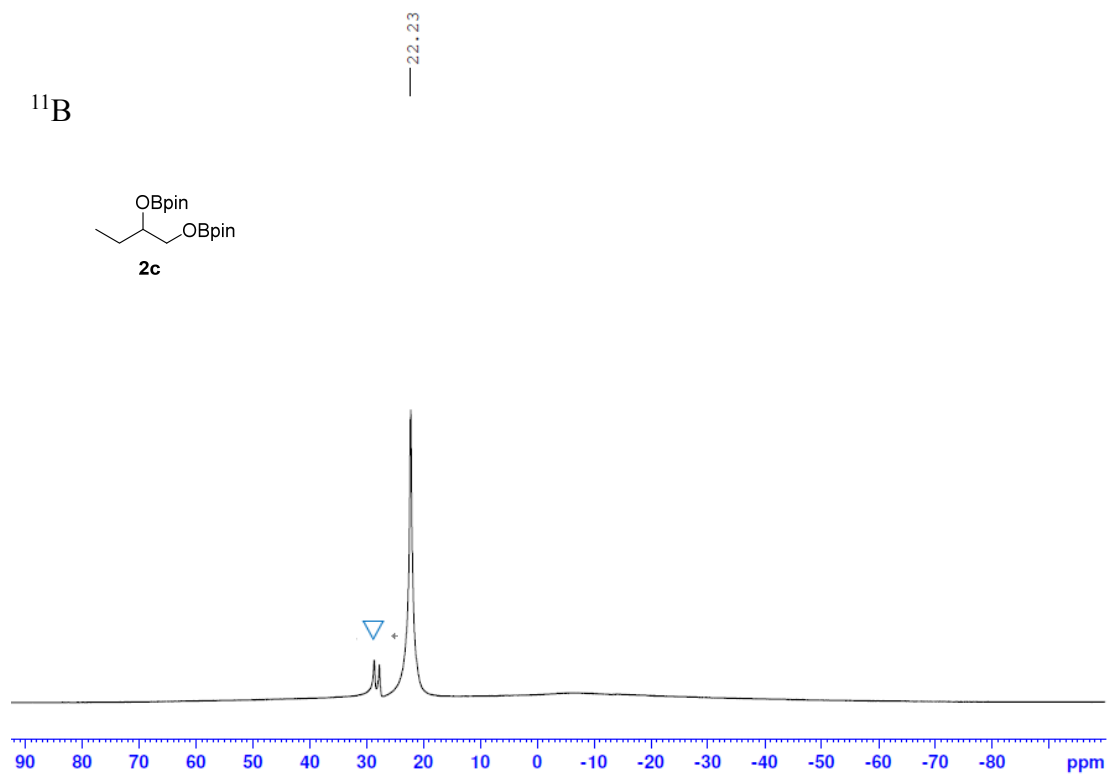
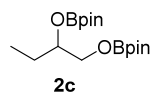


— 22.22

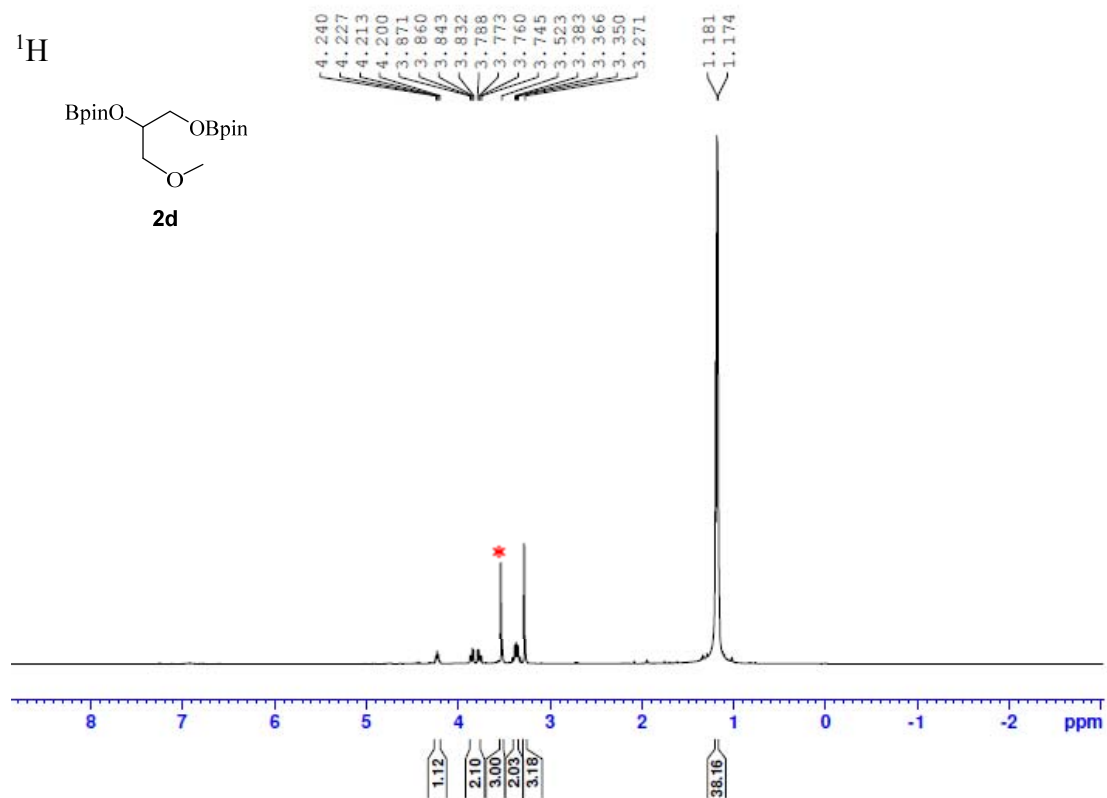
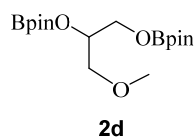




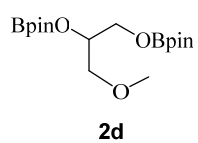
^{11}B

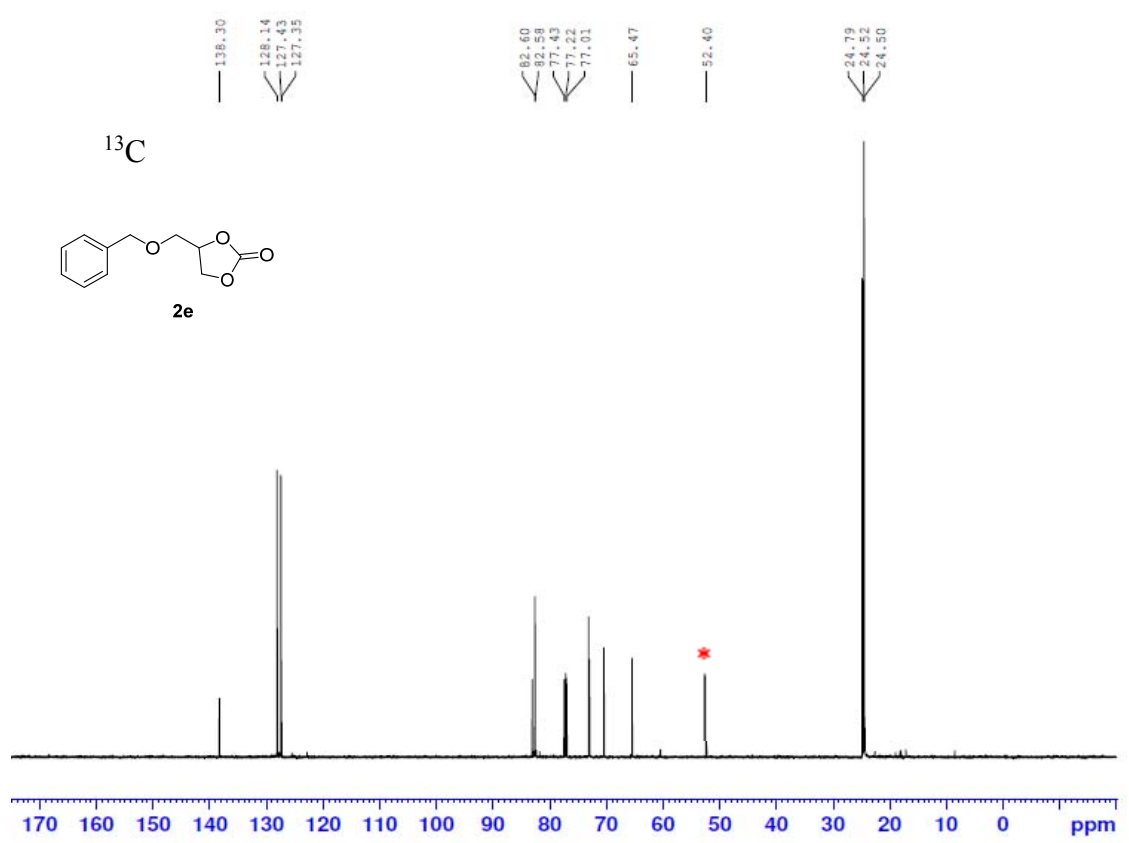
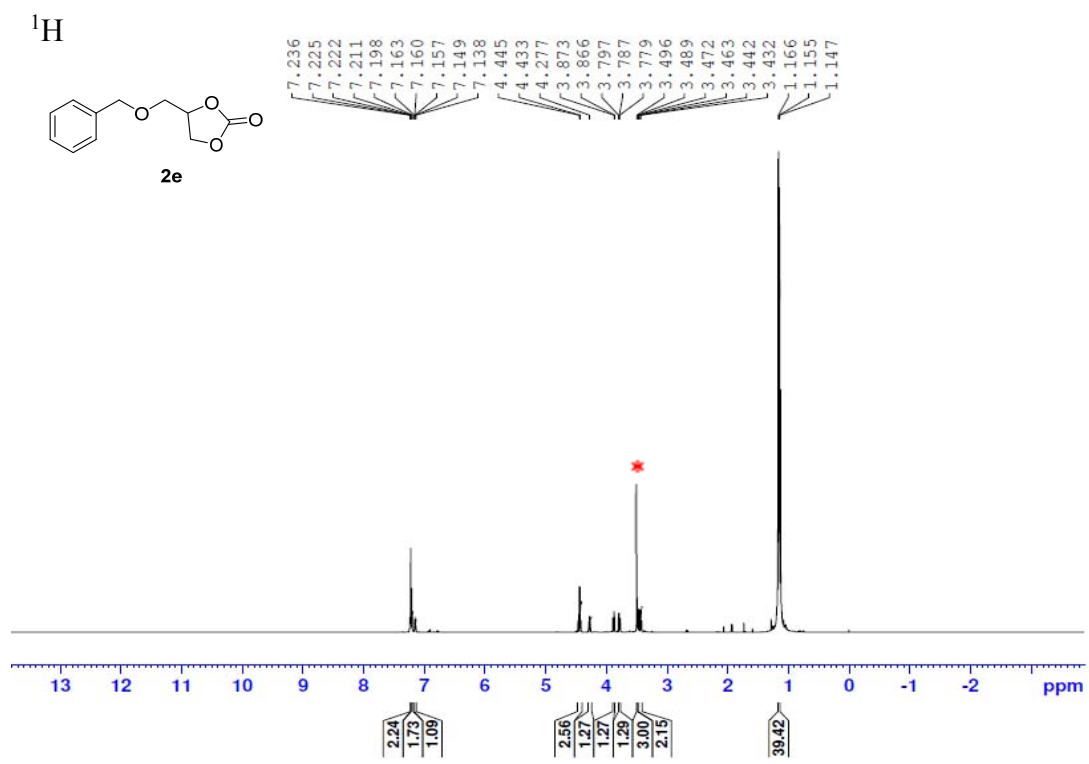


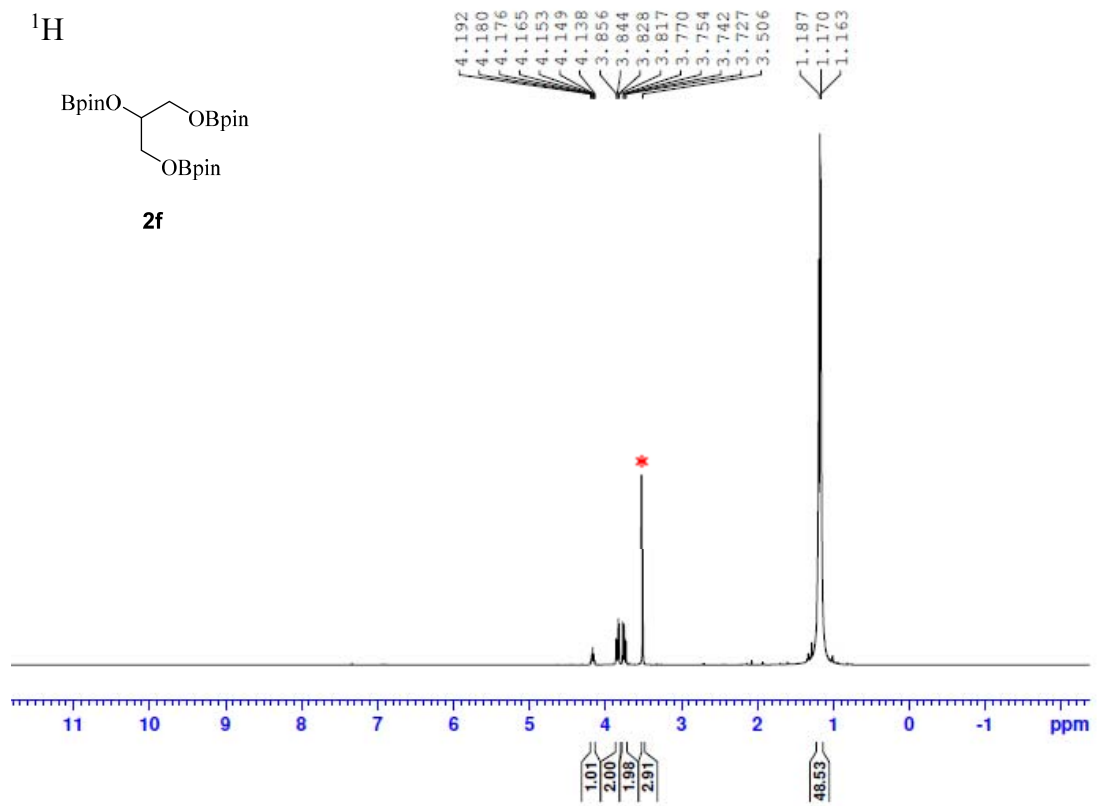
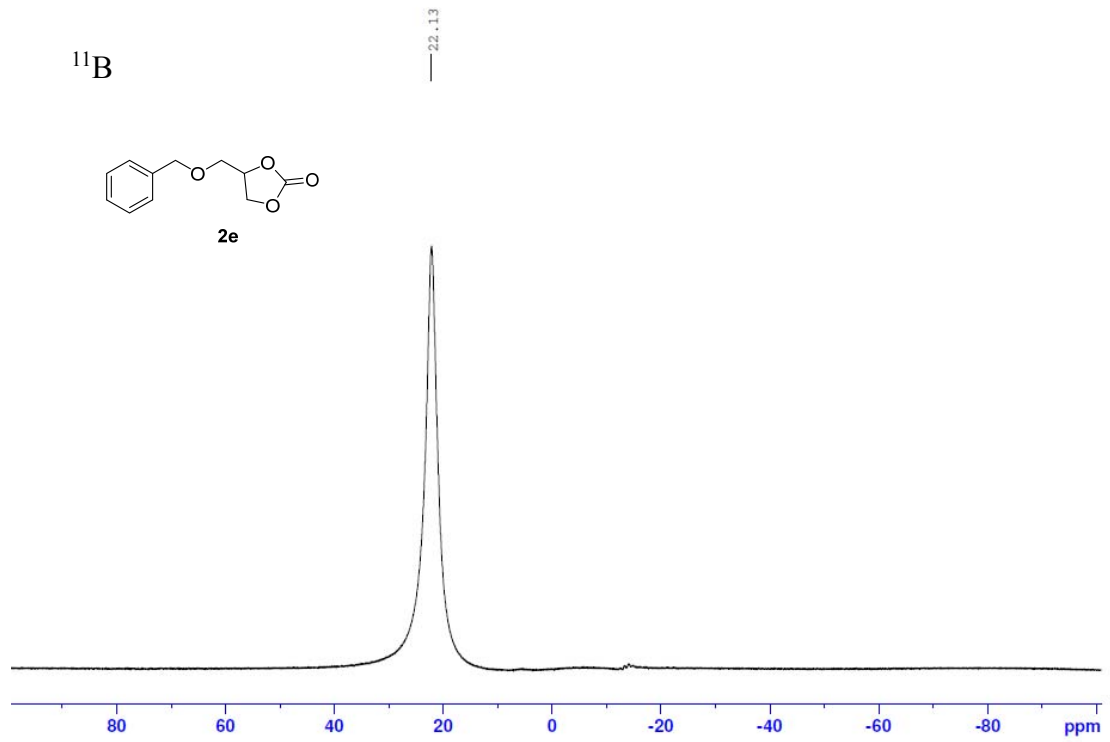
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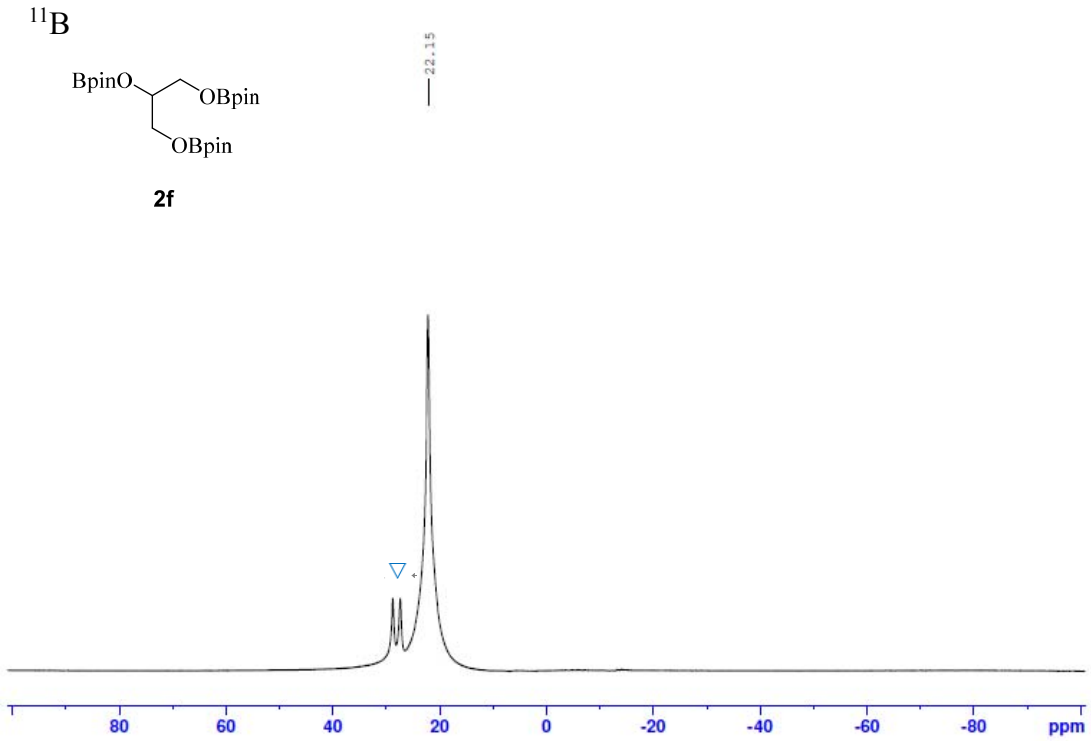
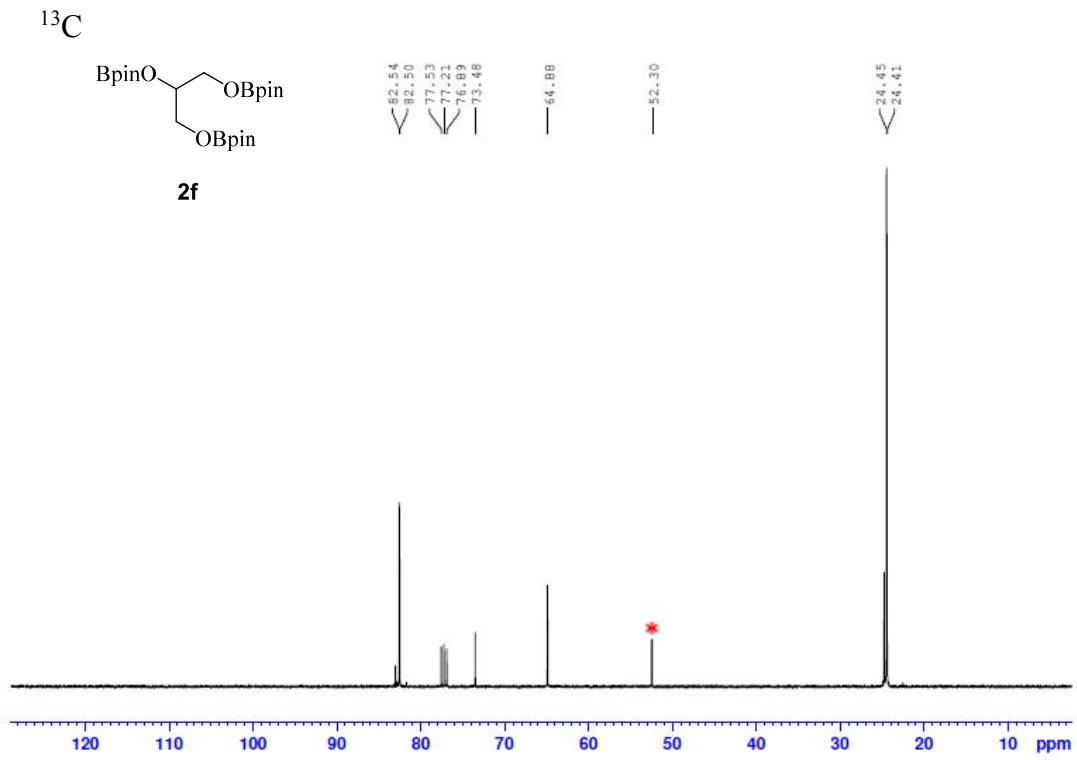


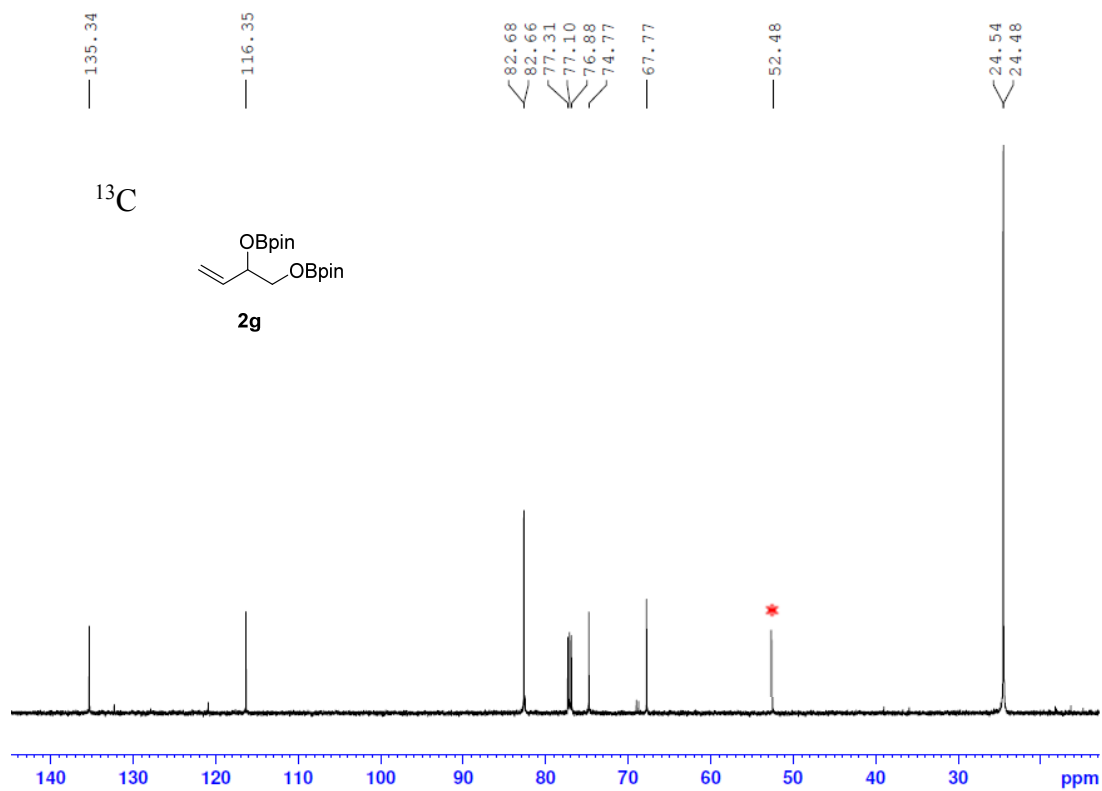
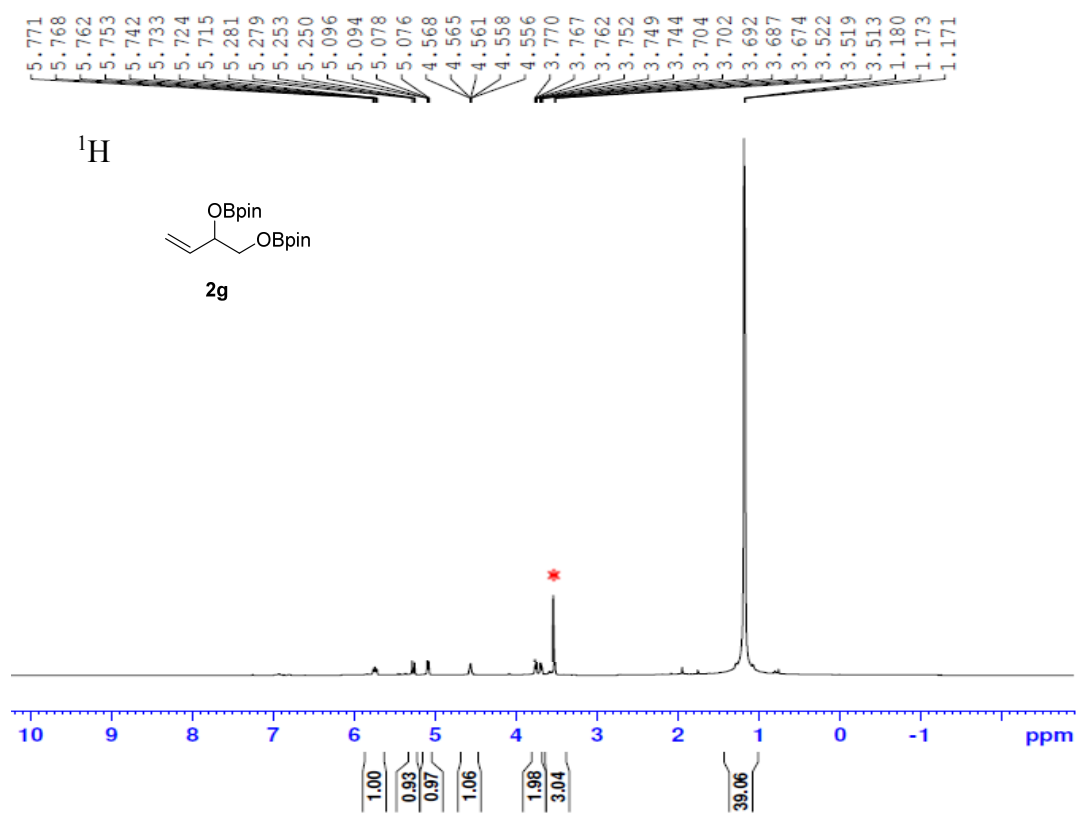
¹³C

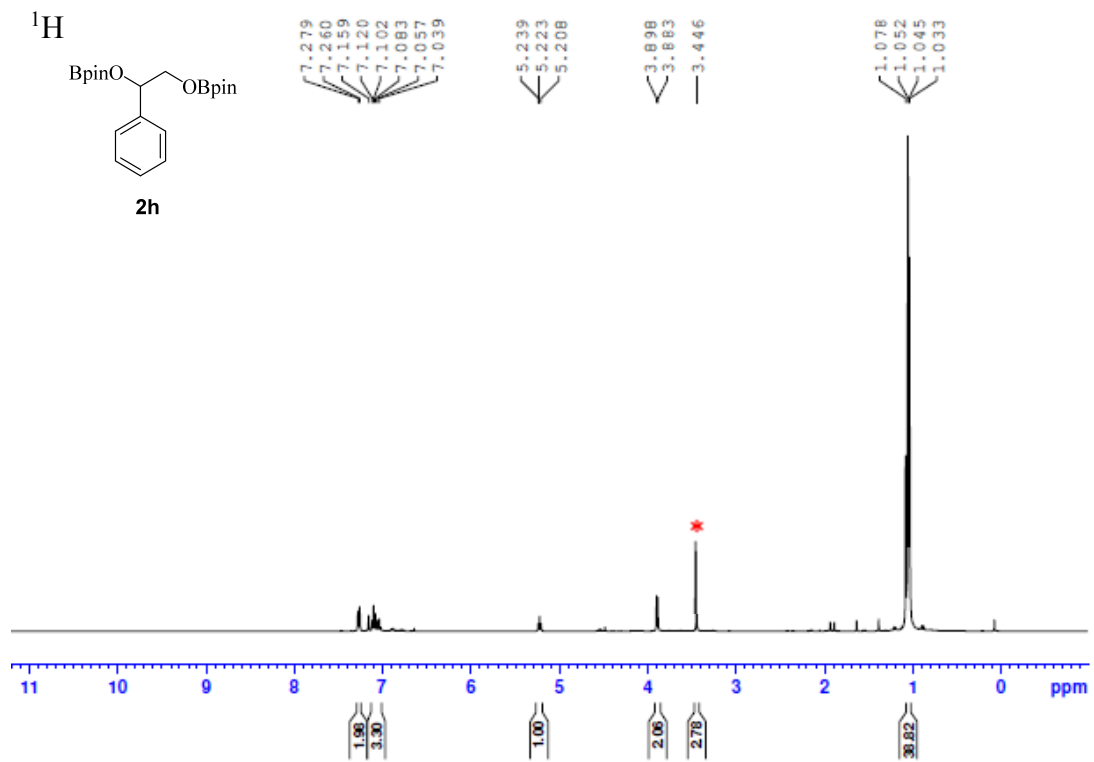
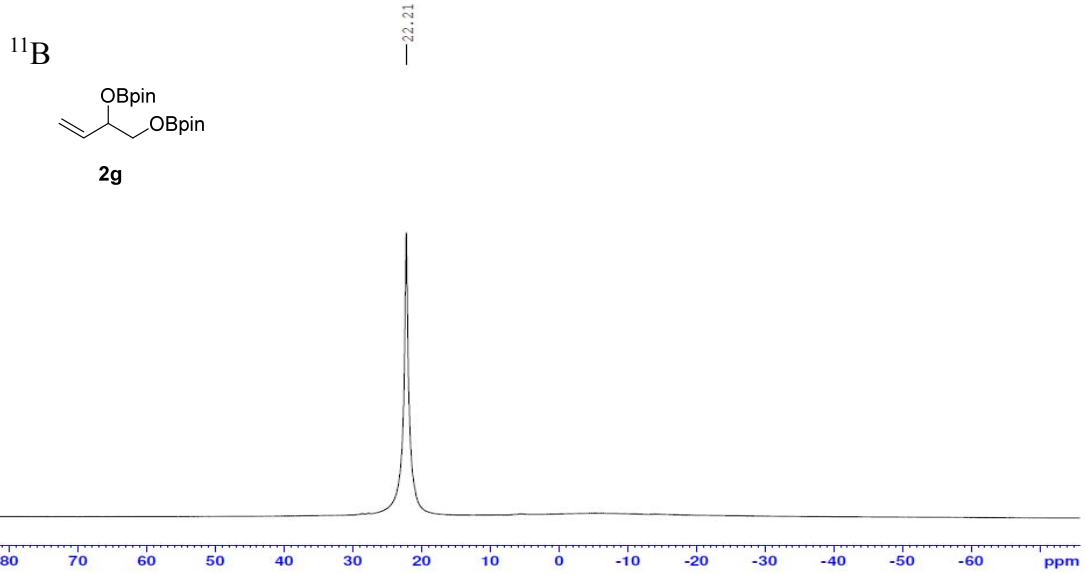




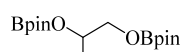




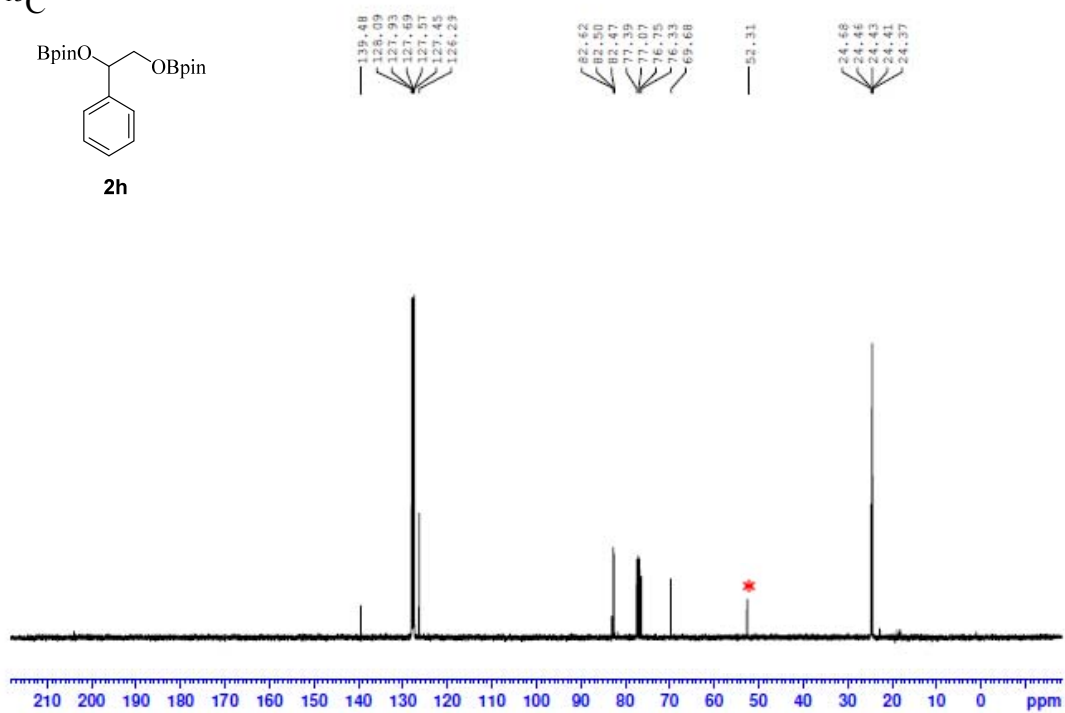




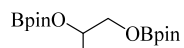
¹³C



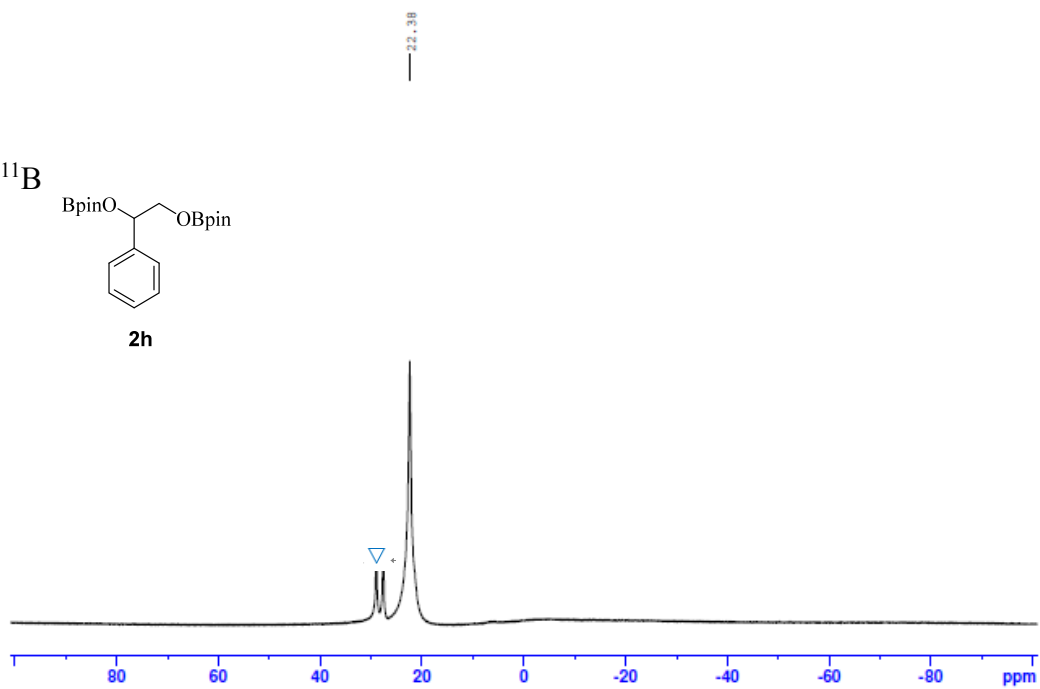
2h



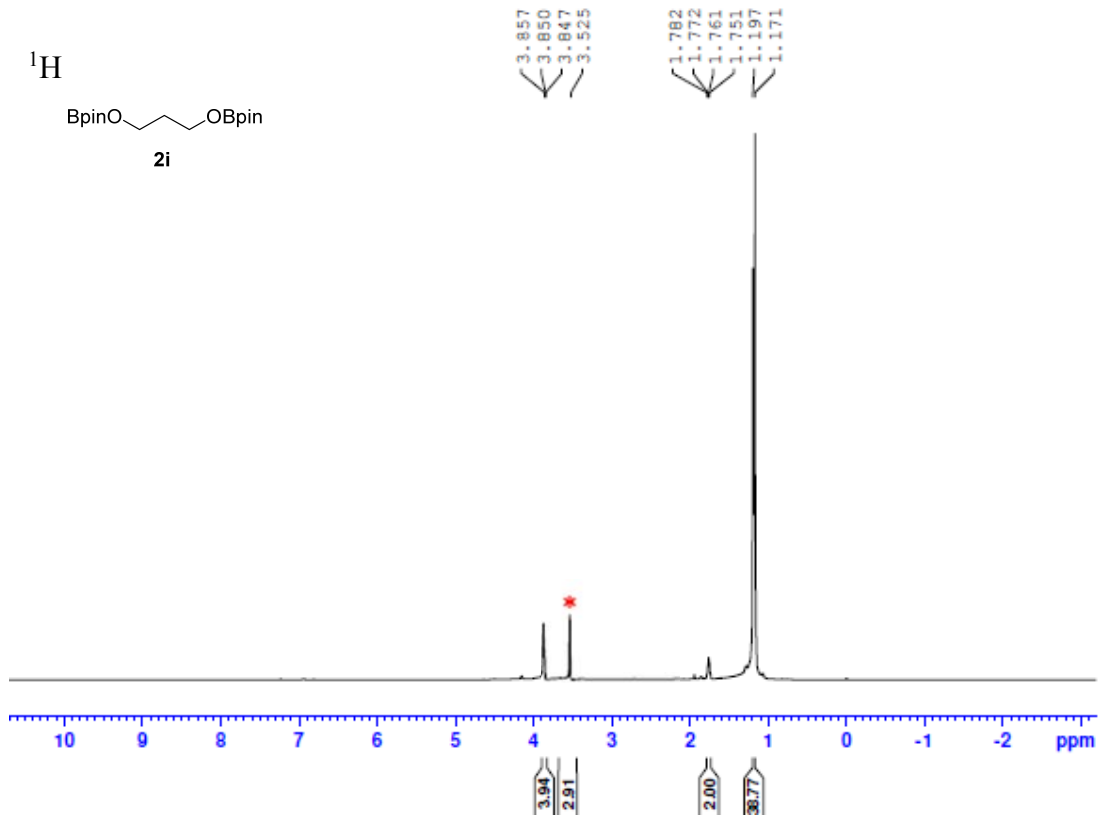
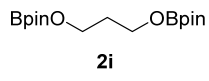
¹¹B



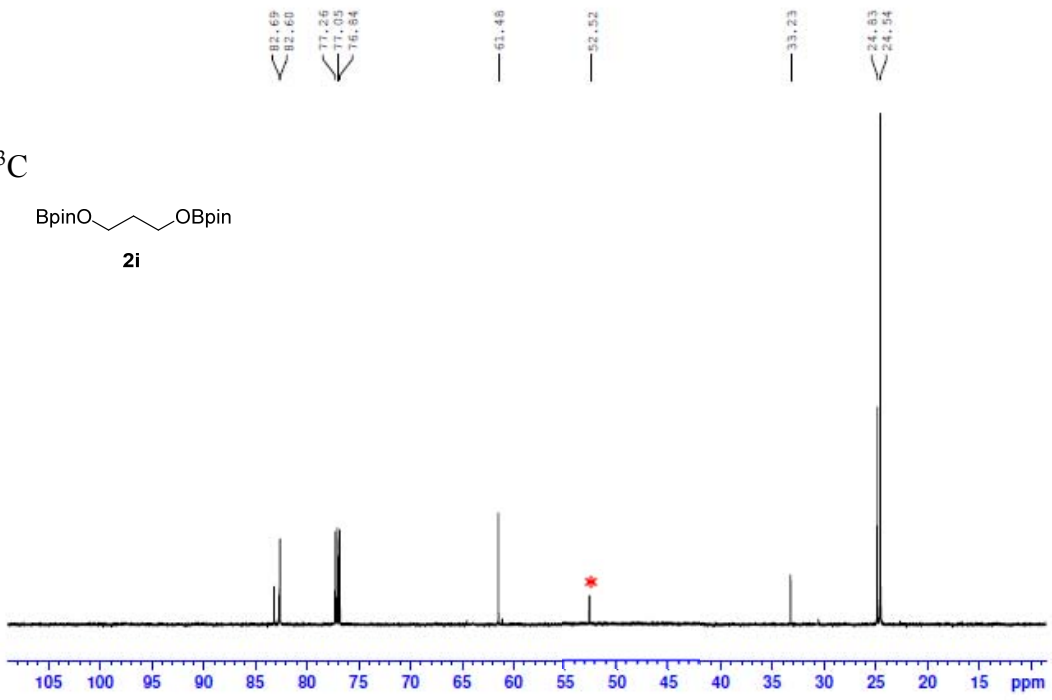
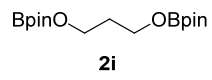
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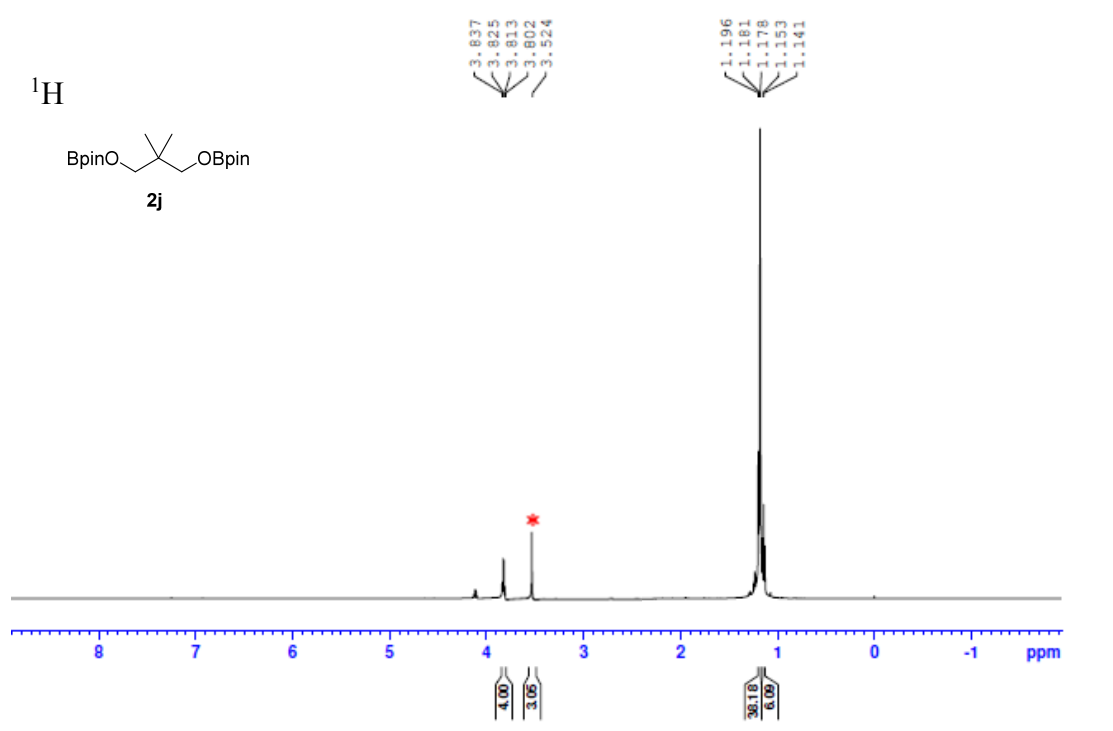
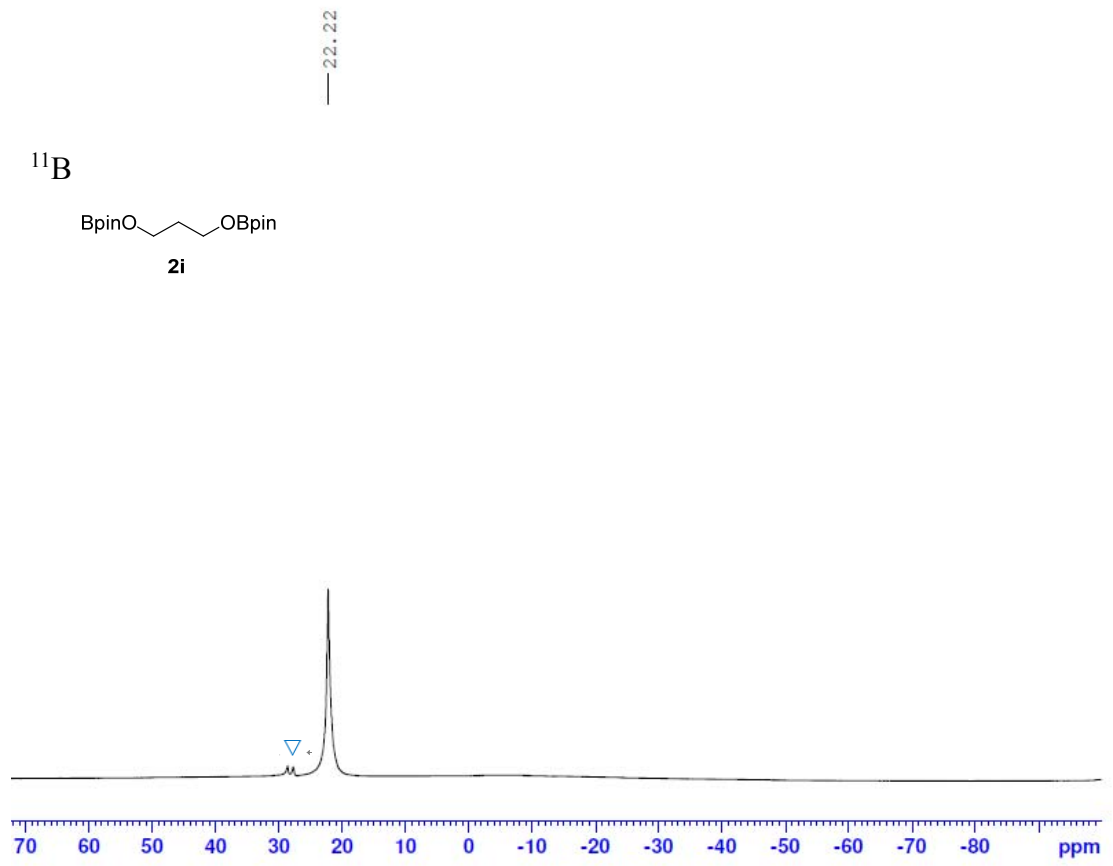


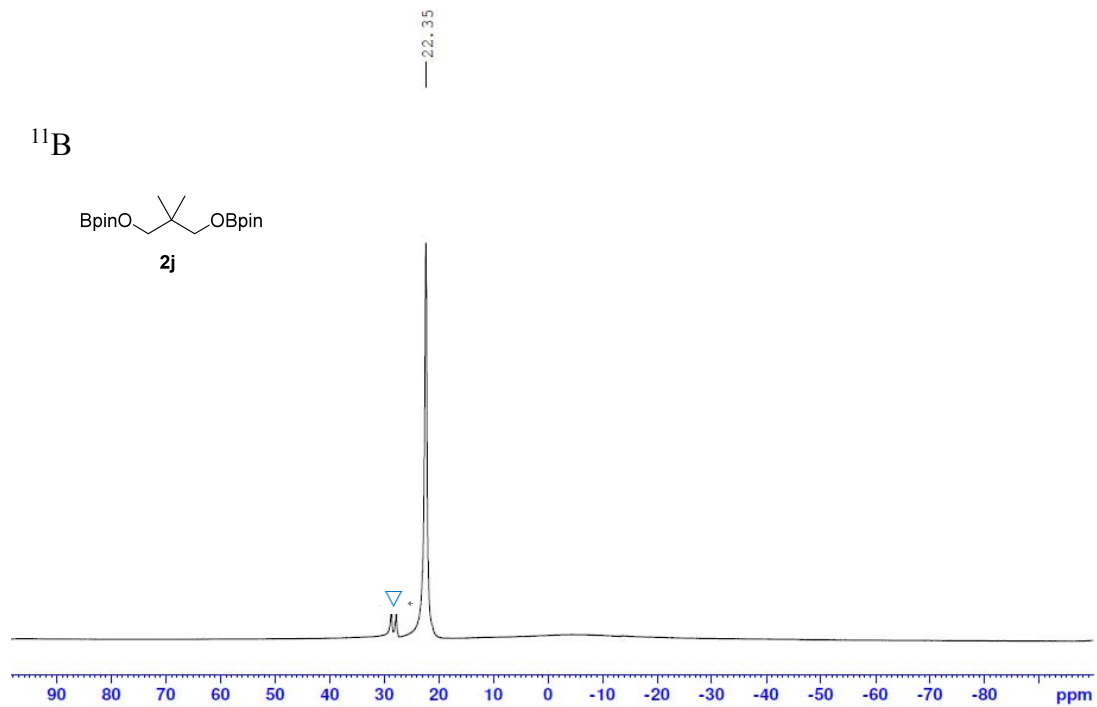
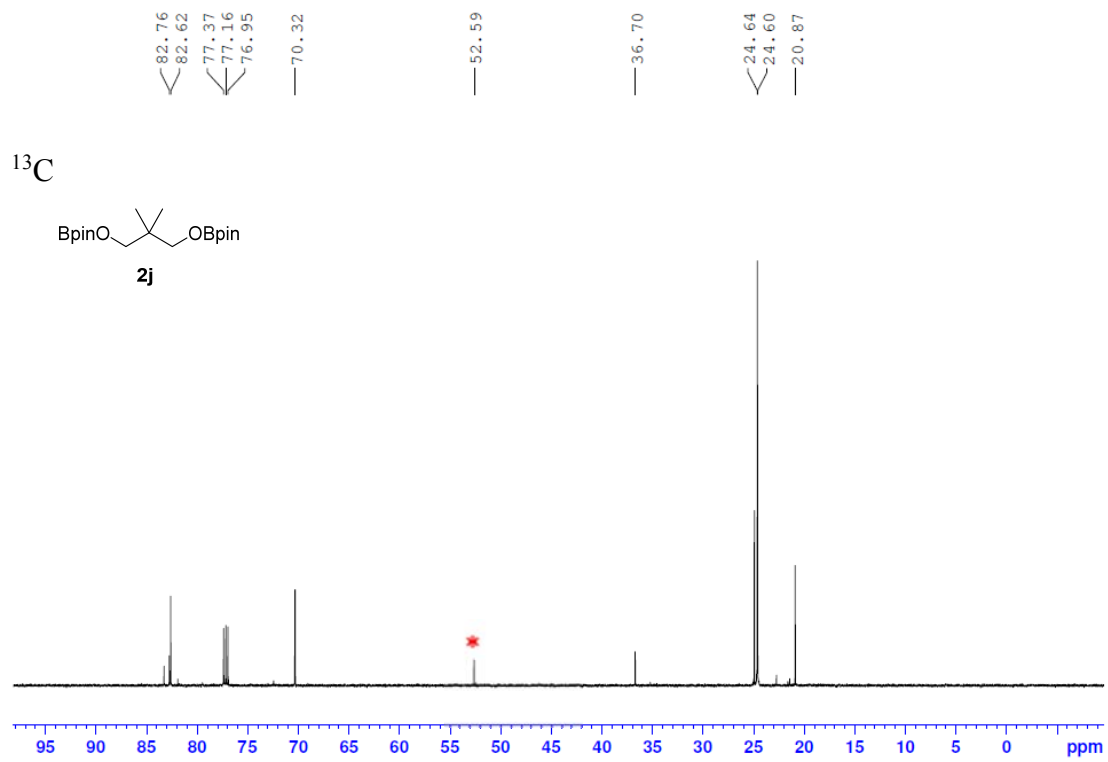
¹H

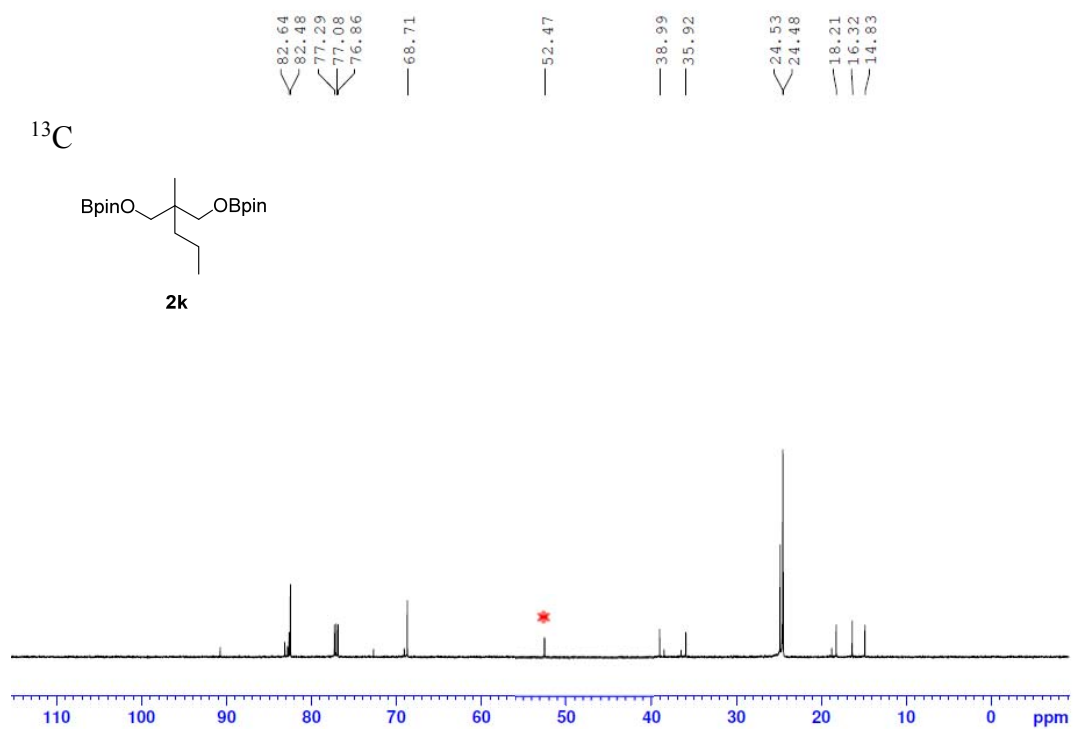
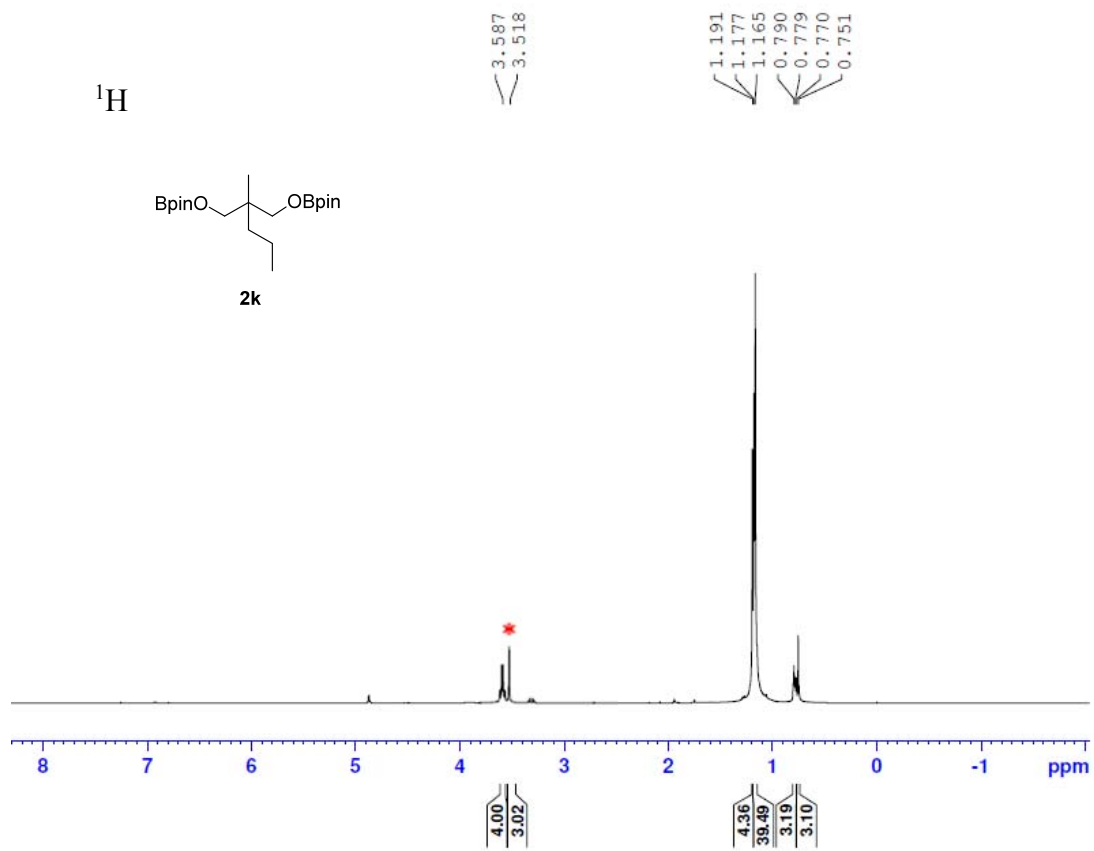


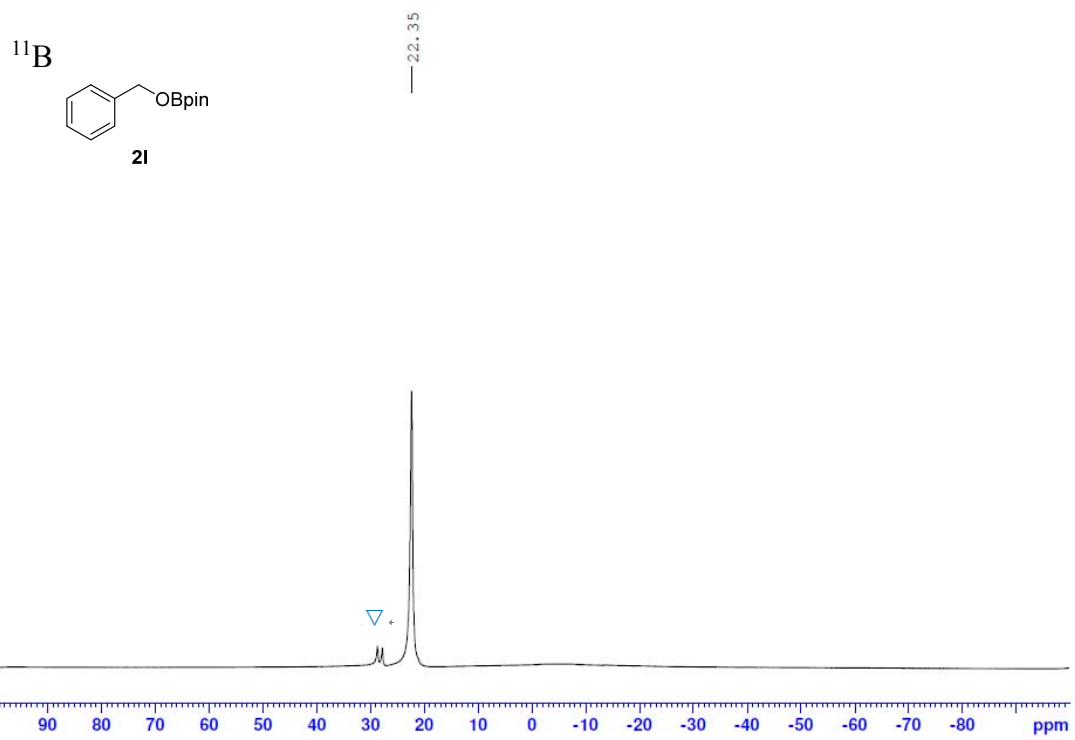
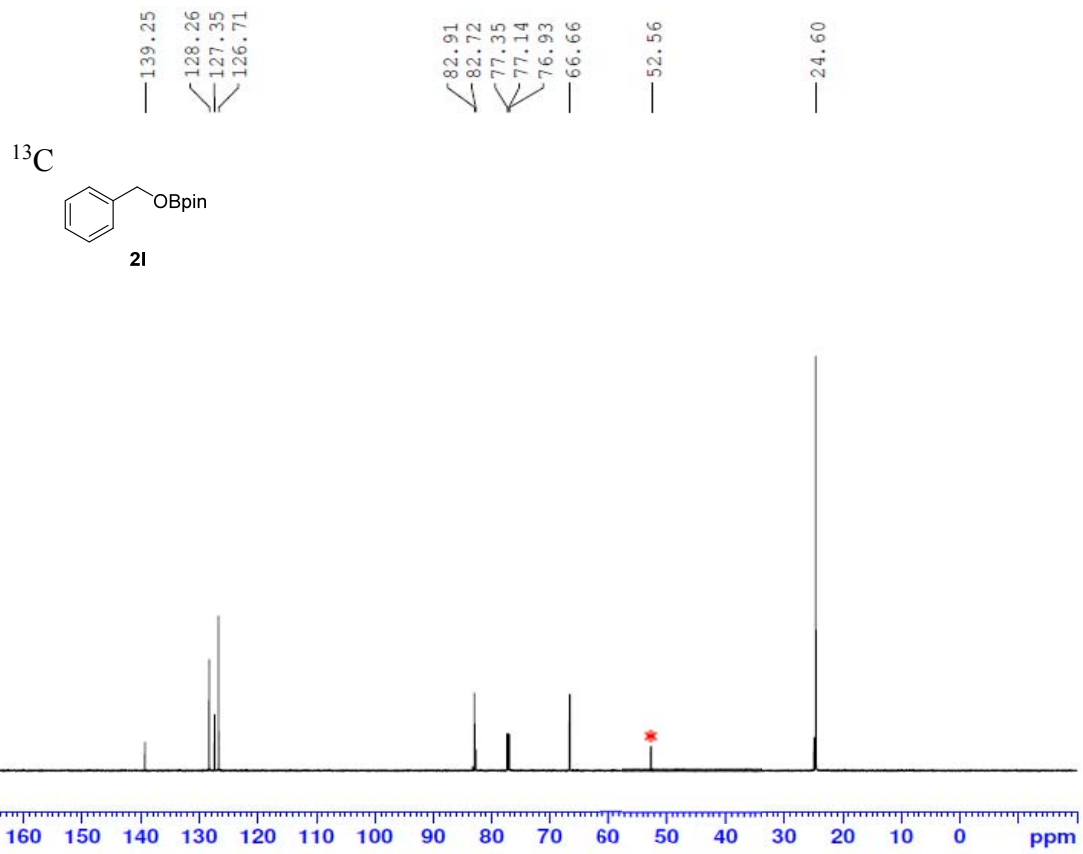
¹³C

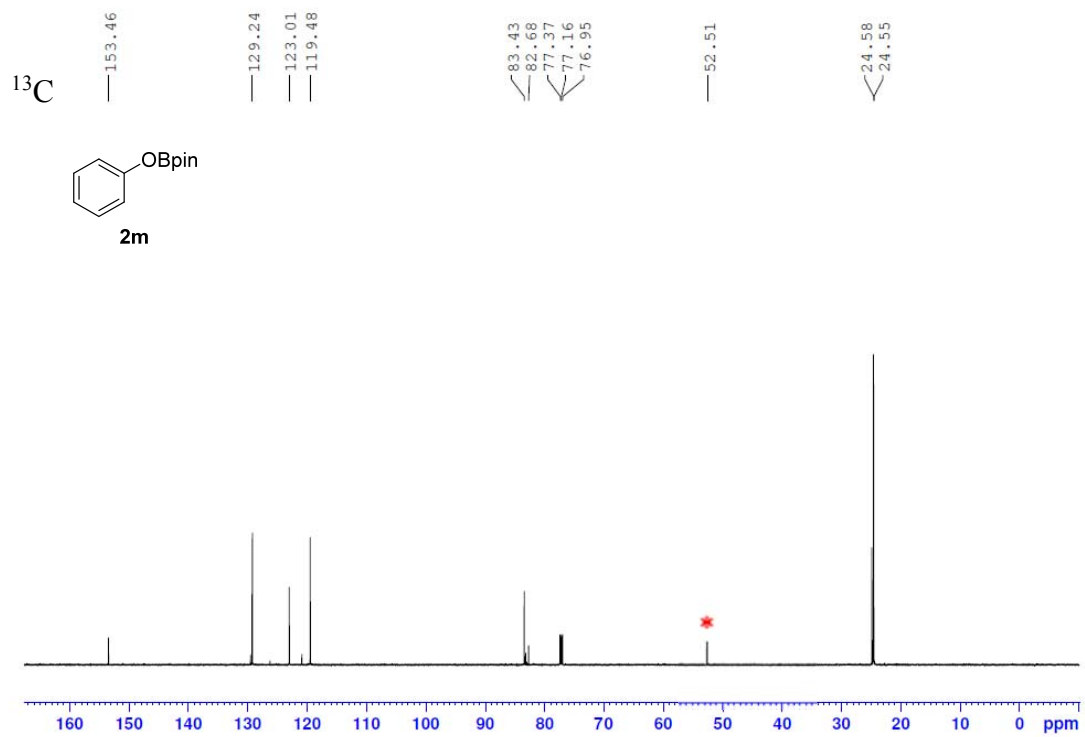
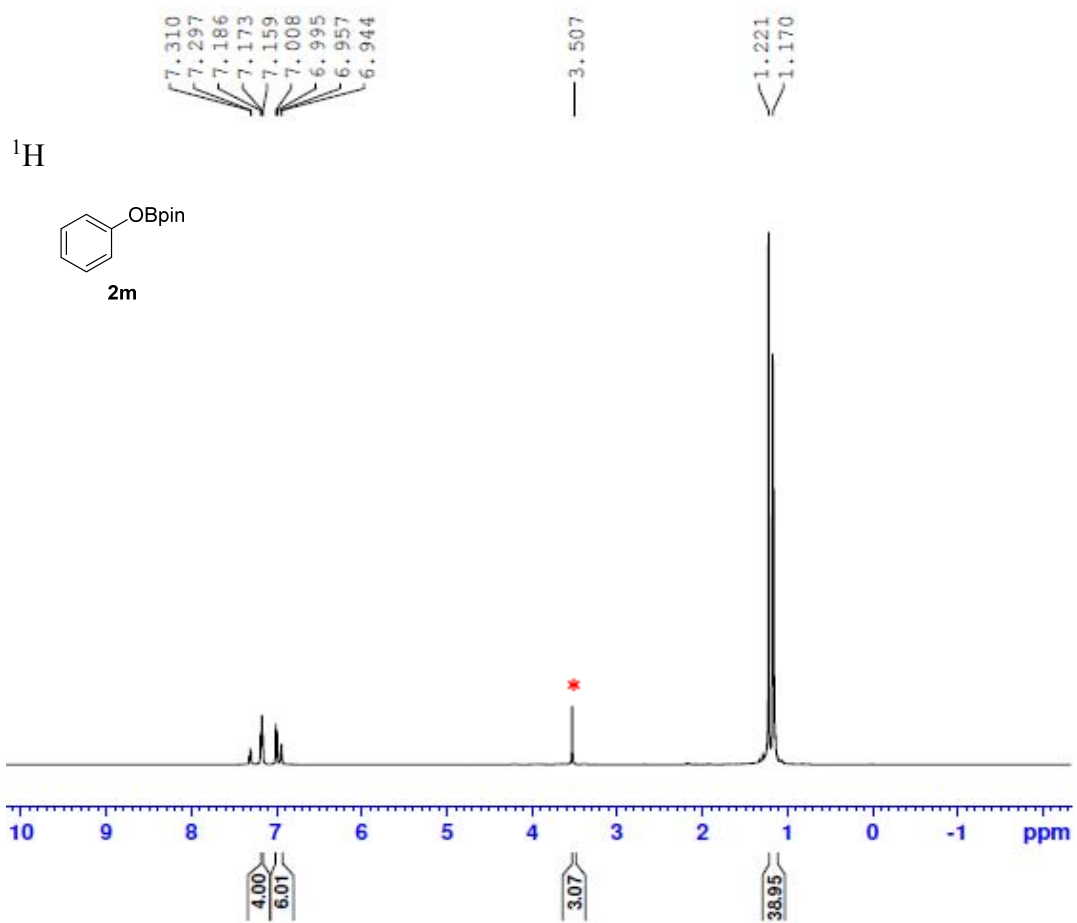


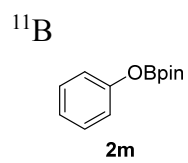




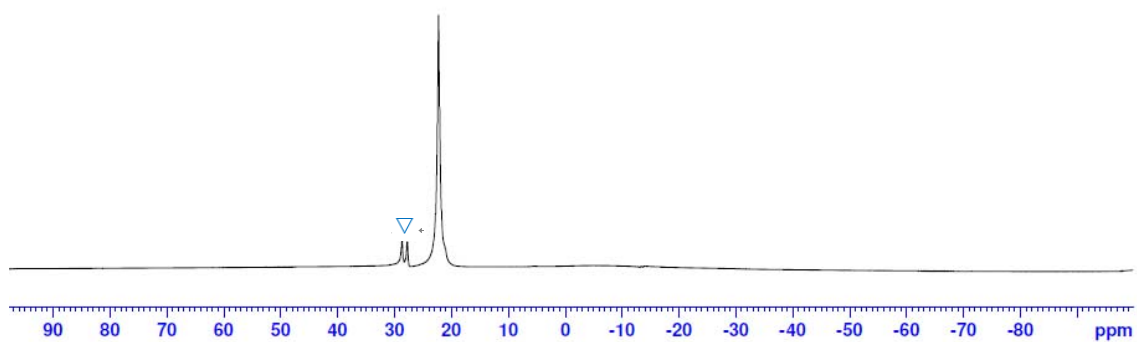




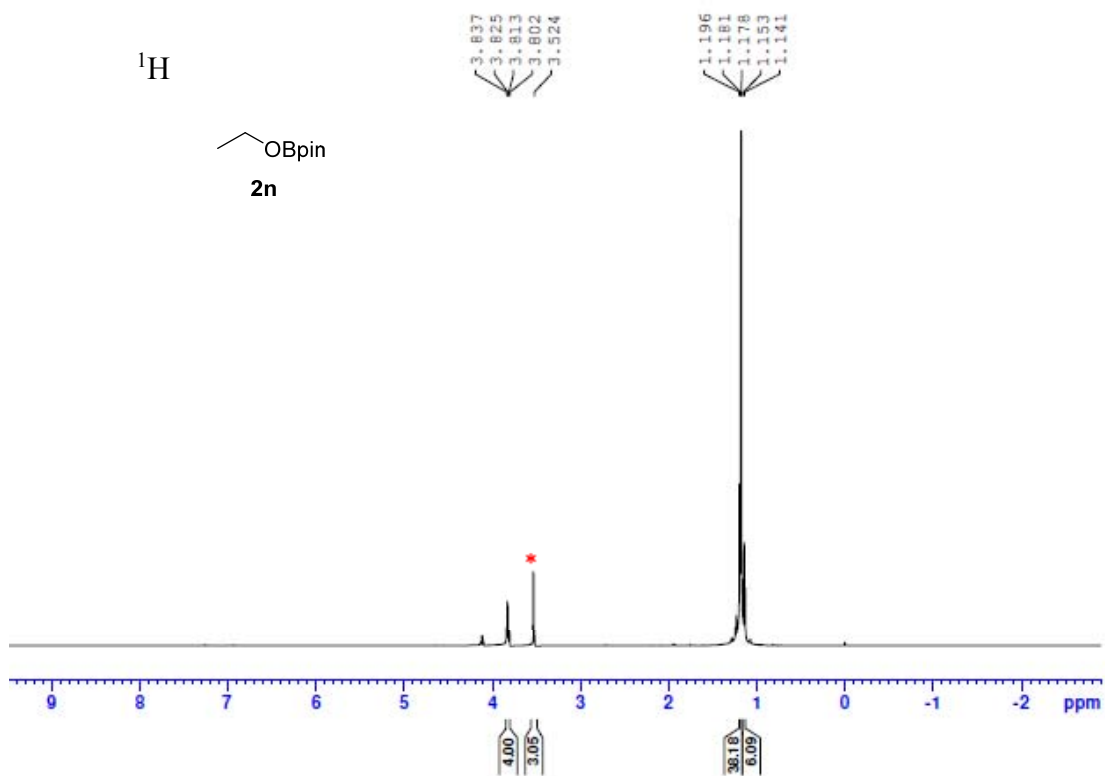
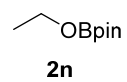


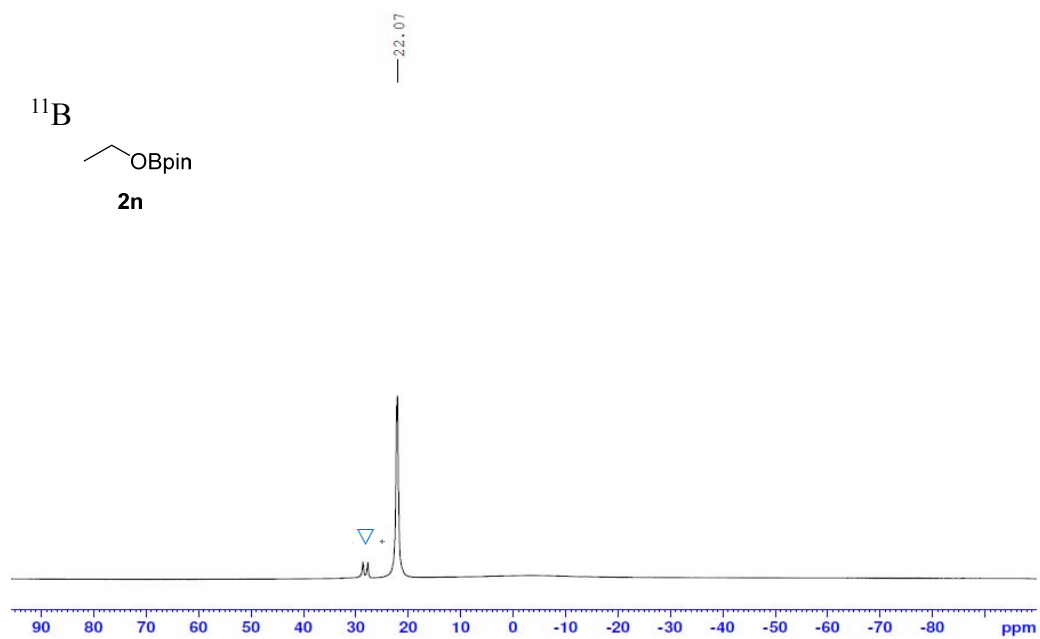
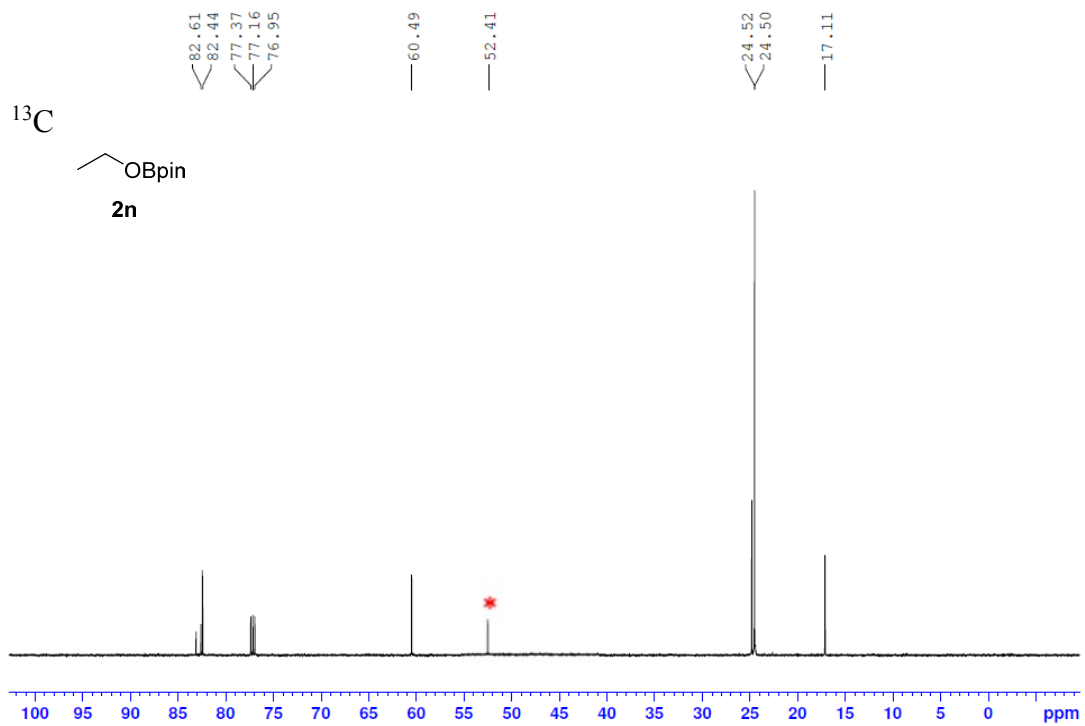


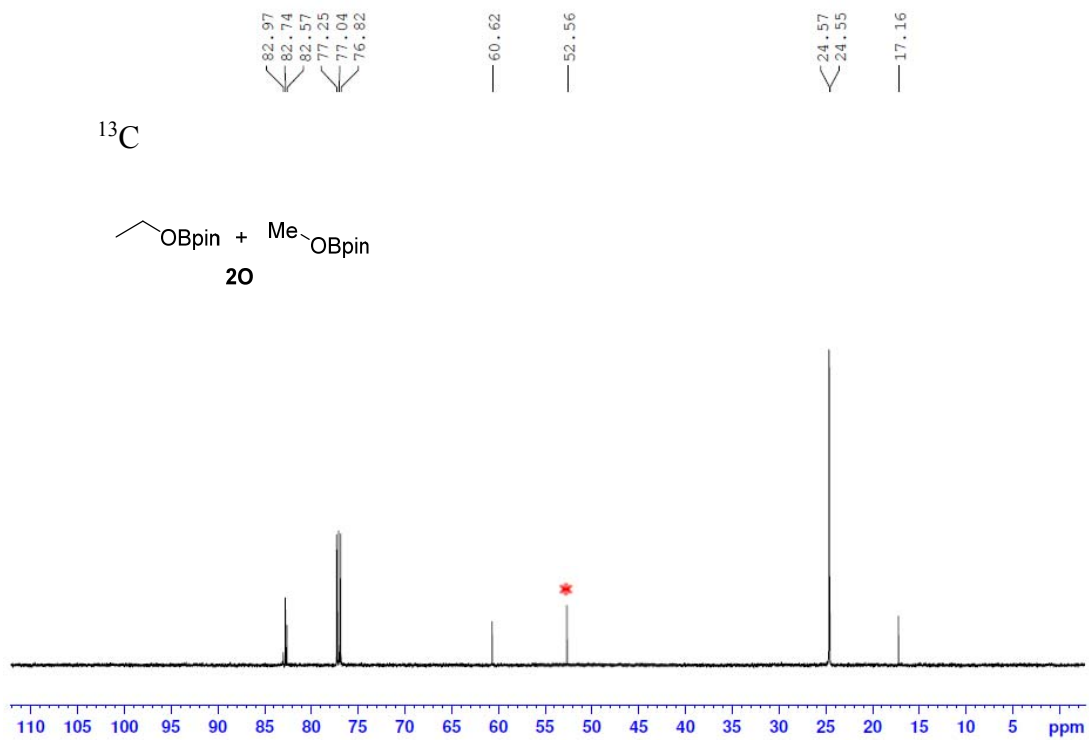
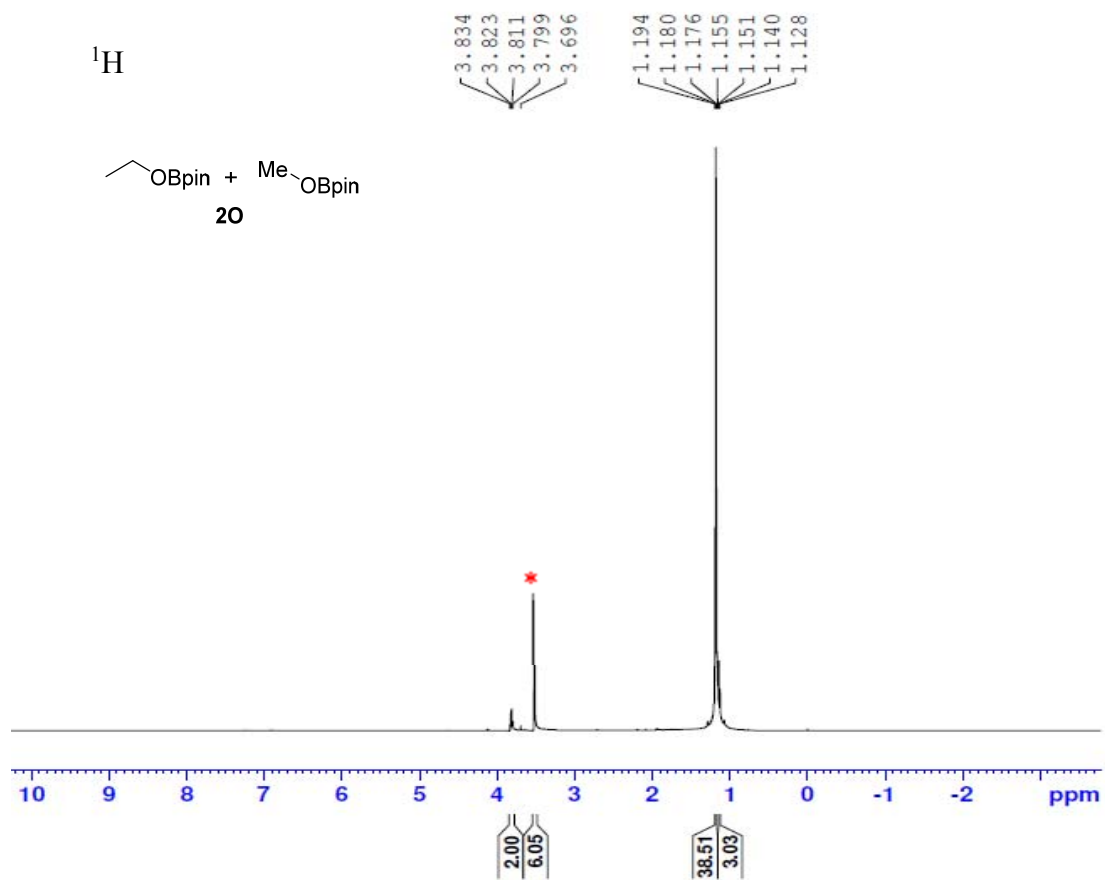
—22.23



^1H

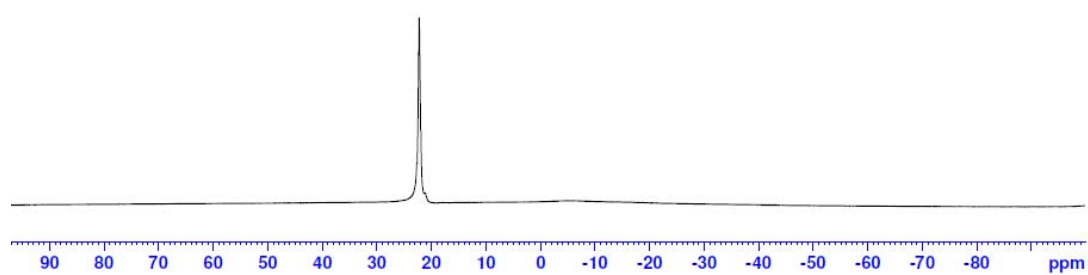
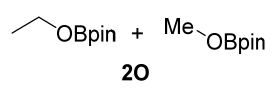




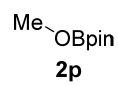


^{11}B

— 22.25

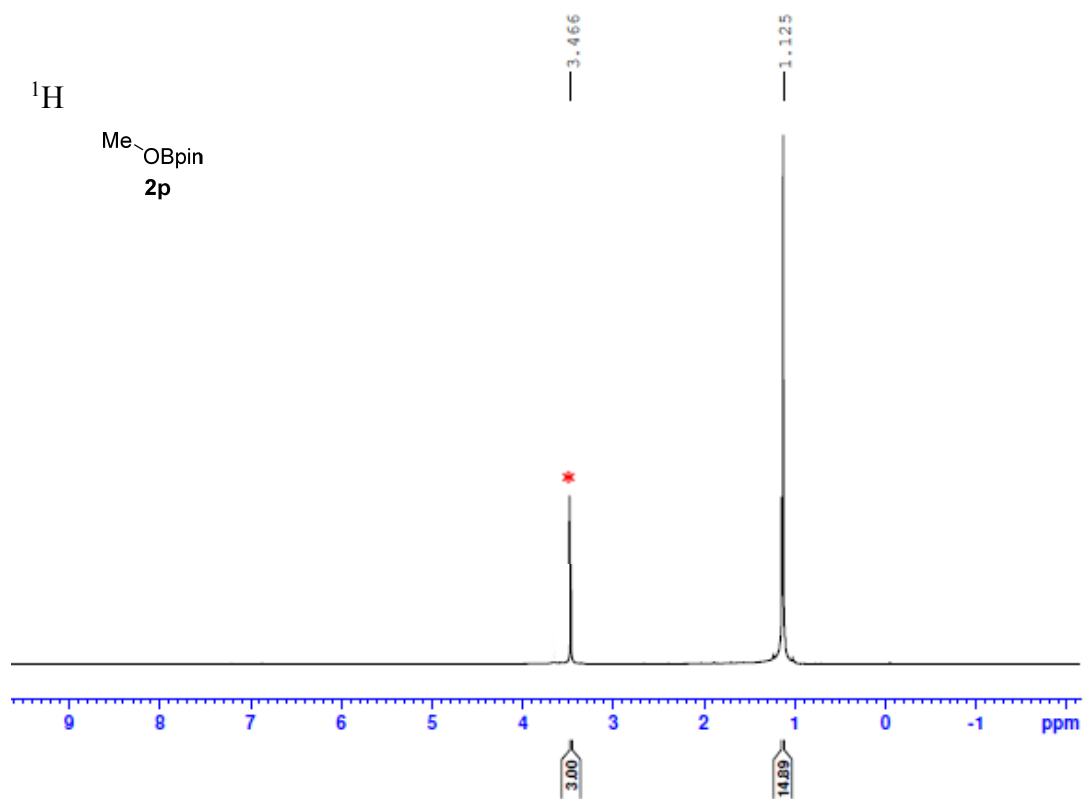


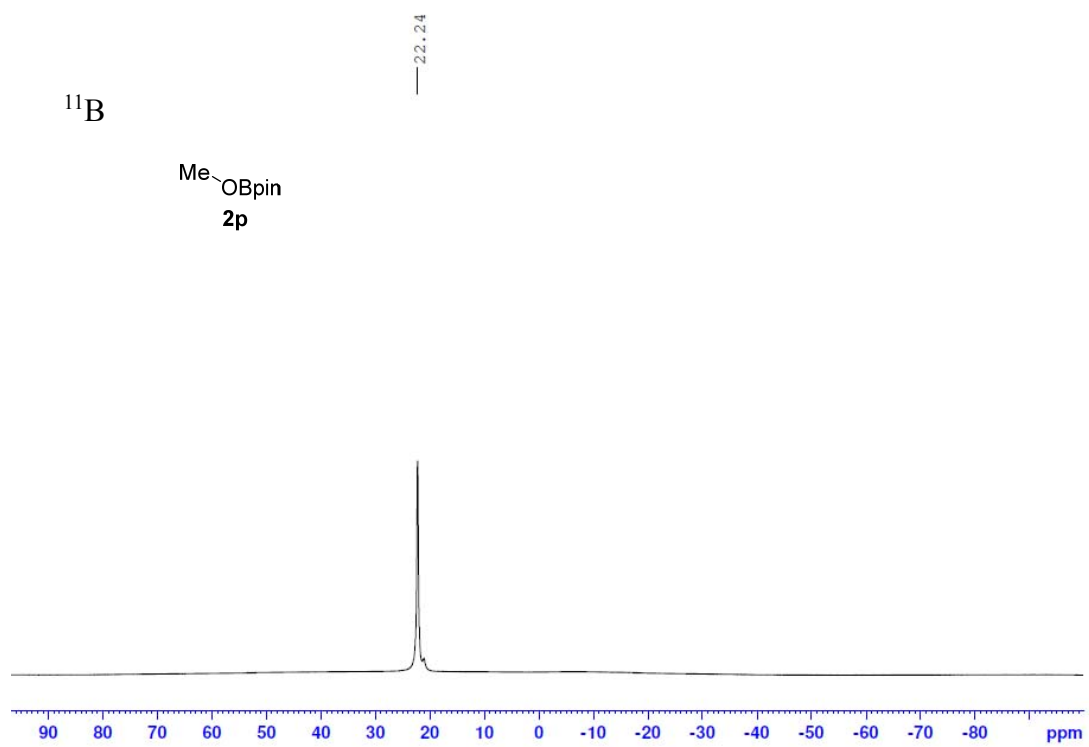
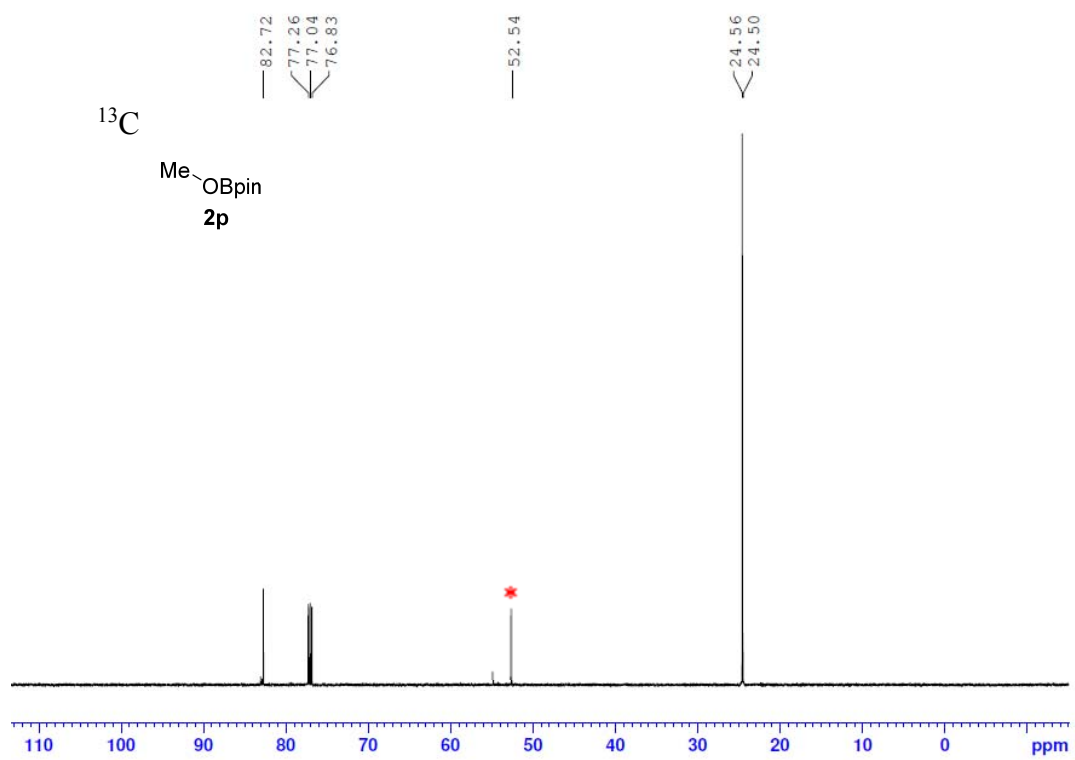
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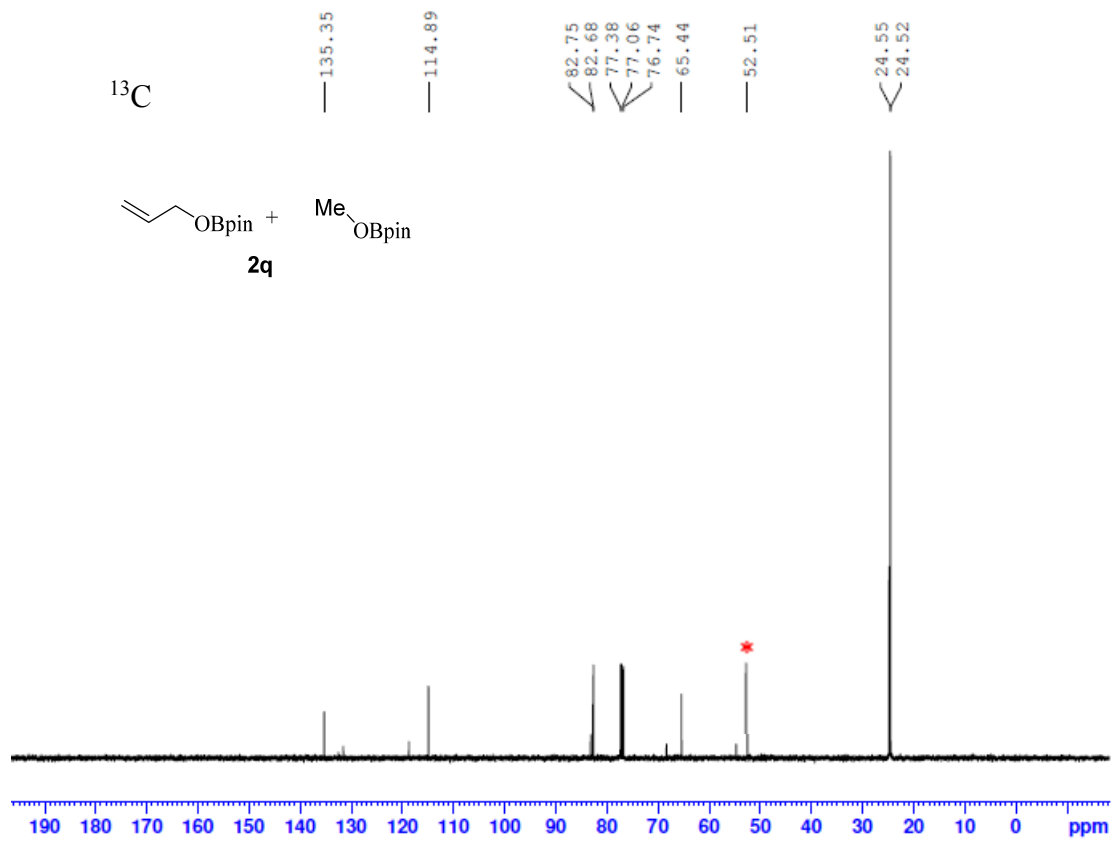
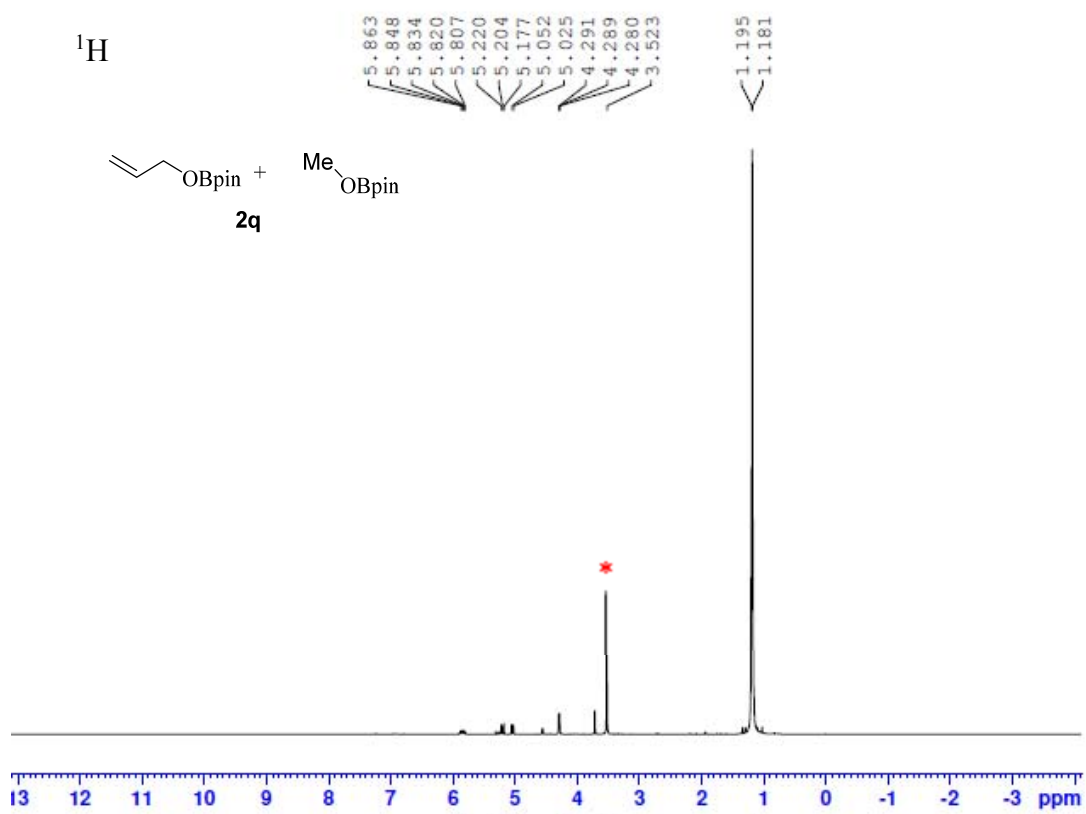


— 3.466

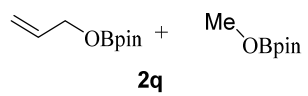
— 1.125



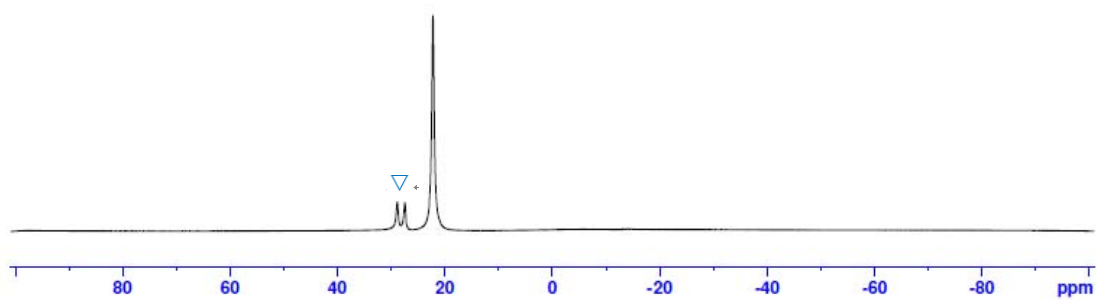




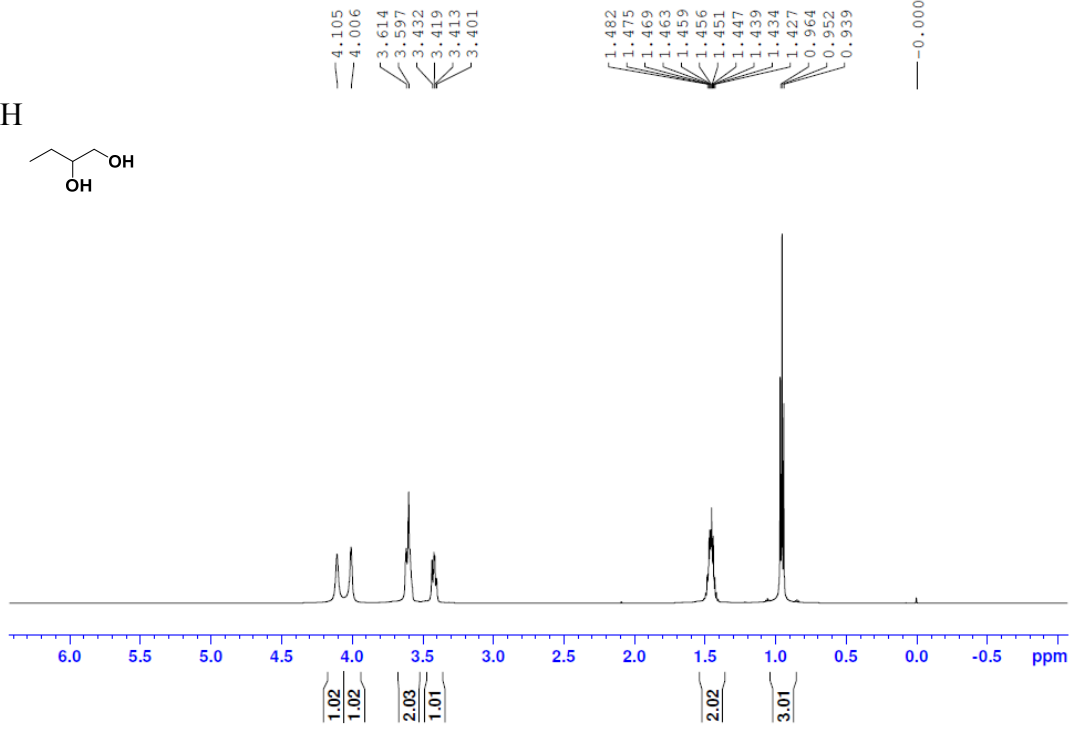
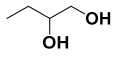
^{11}B



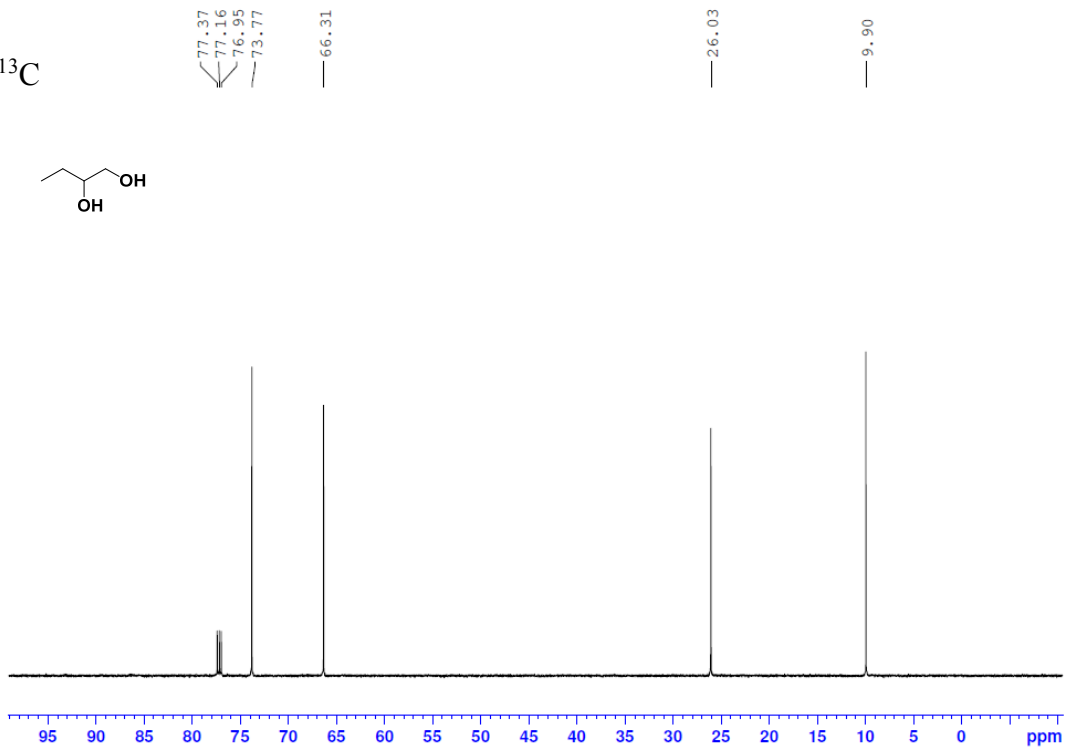
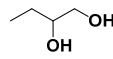
28.85
27.48
22.23



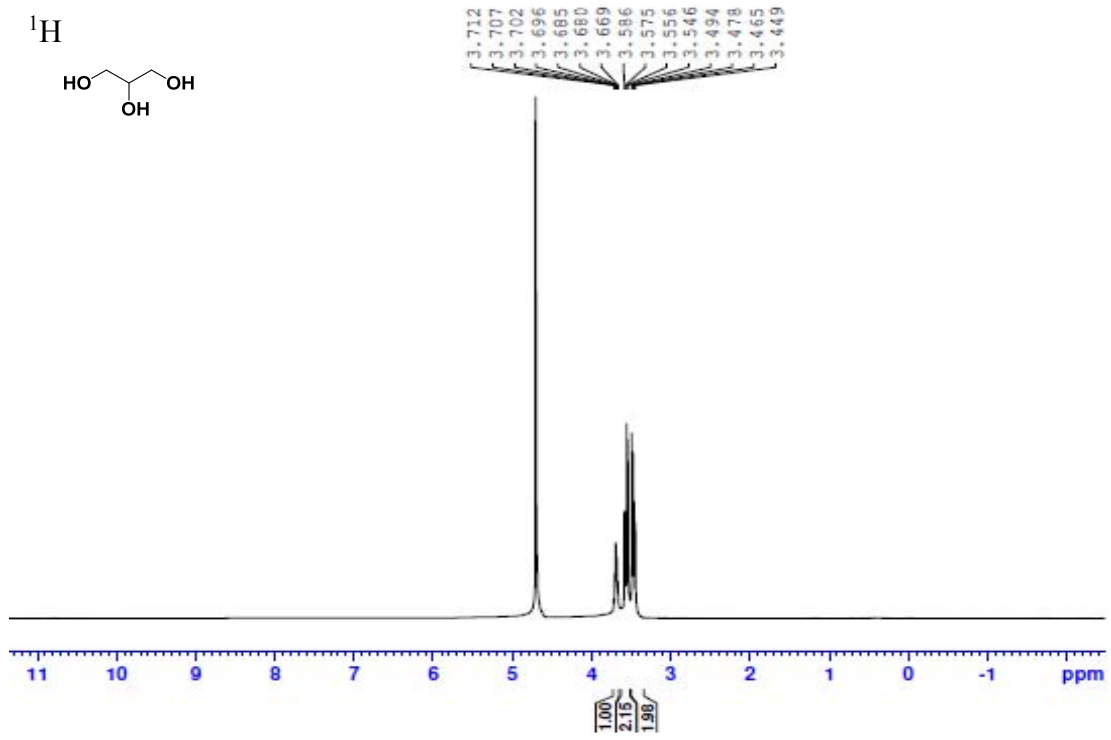
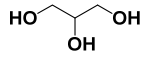
^1H



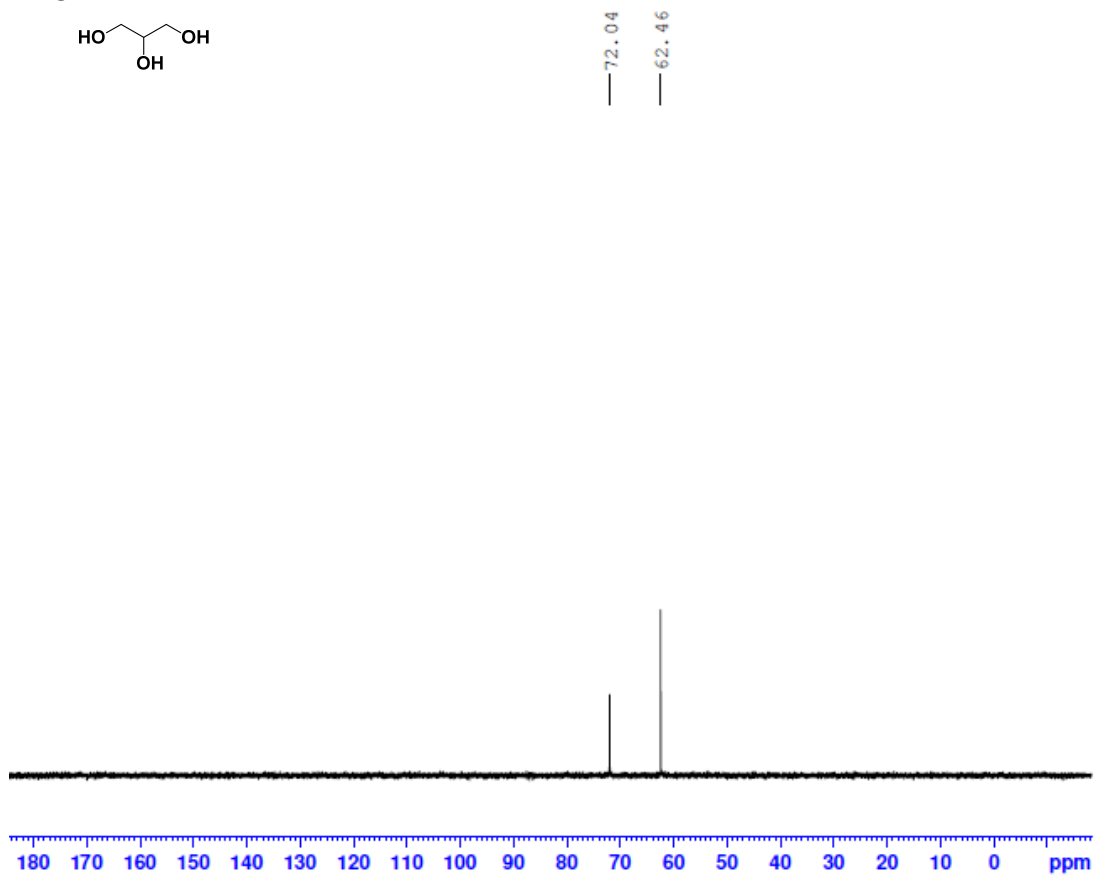
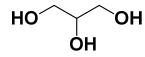
^{13}C

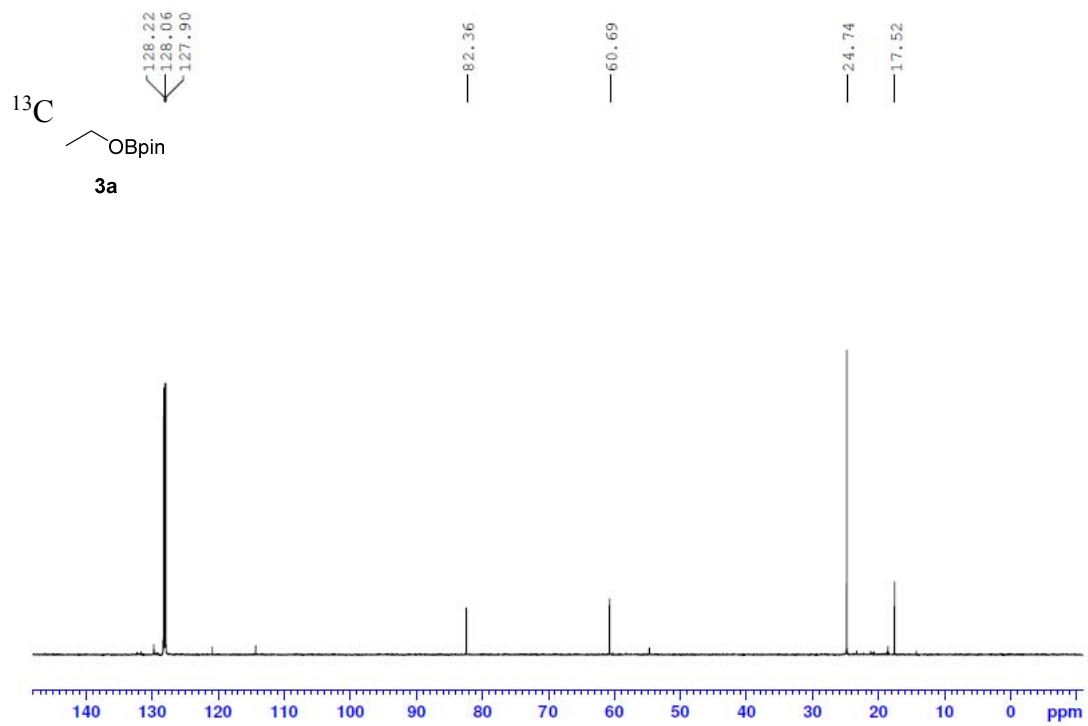
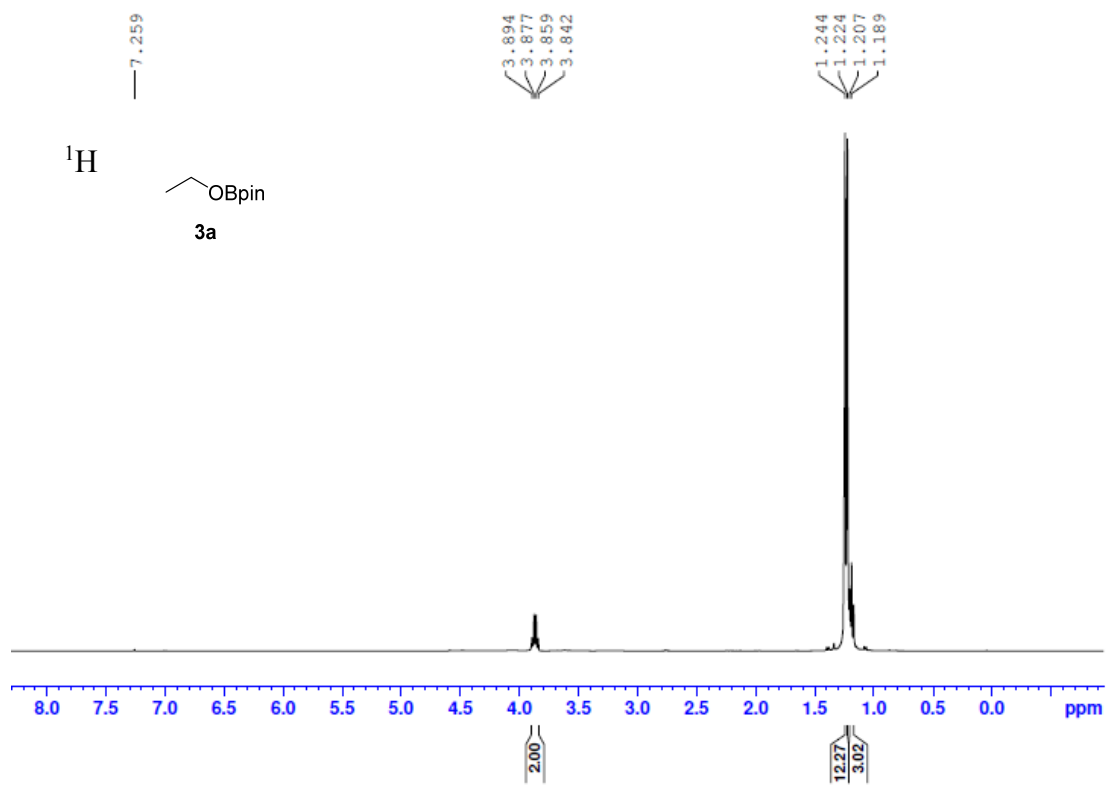


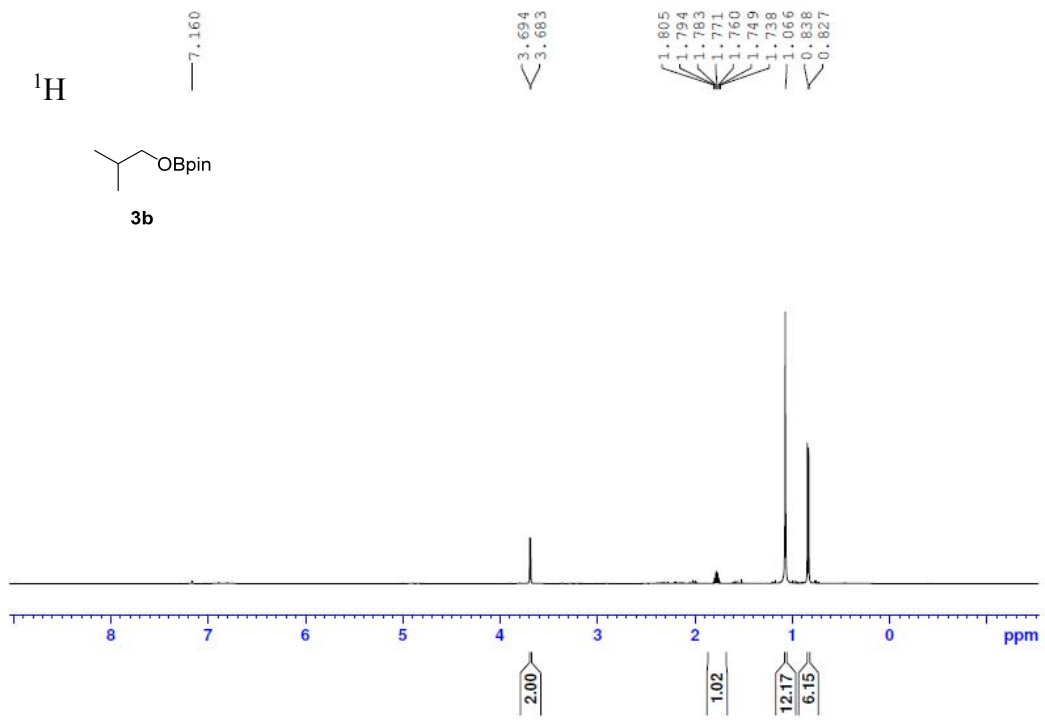
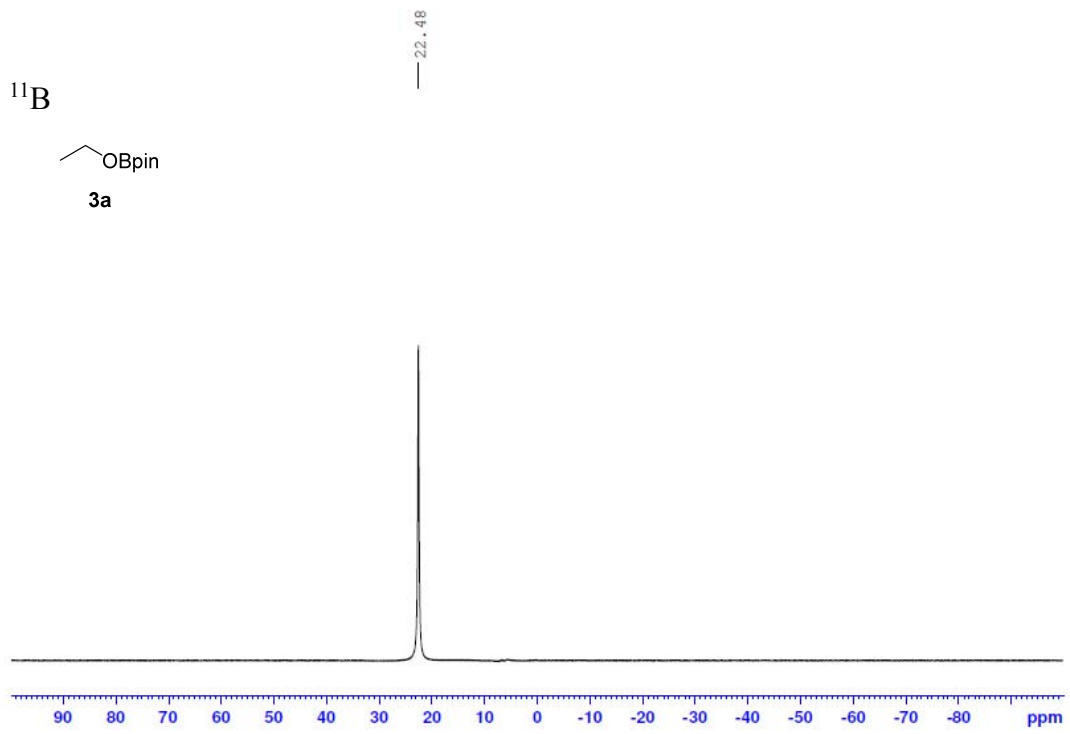
¹H

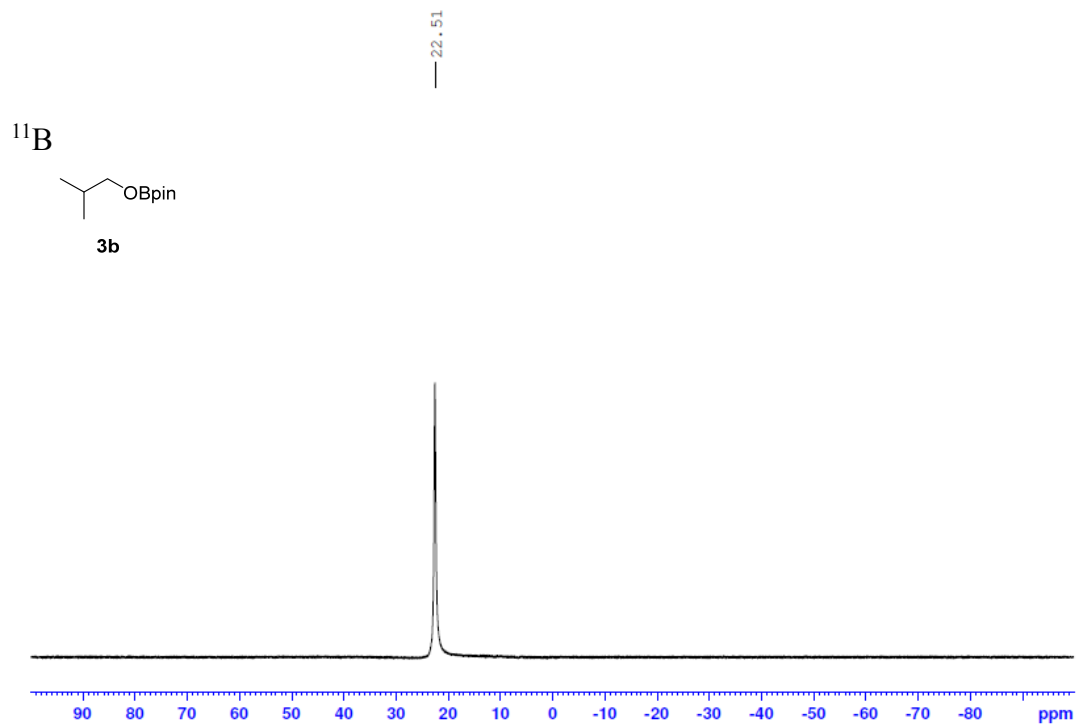
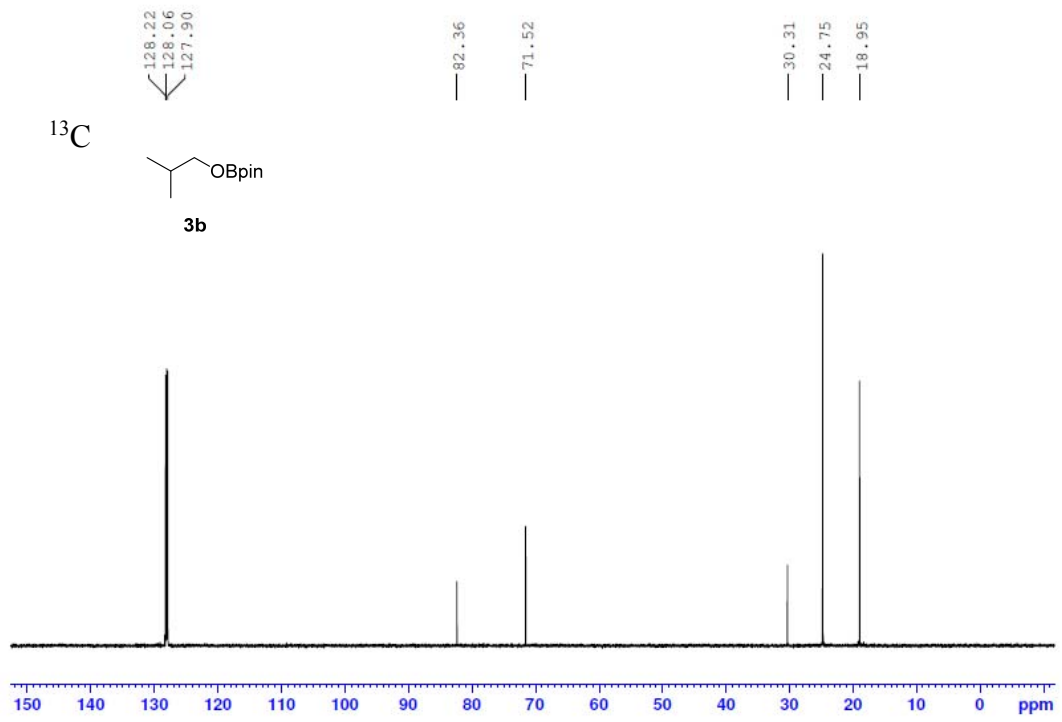


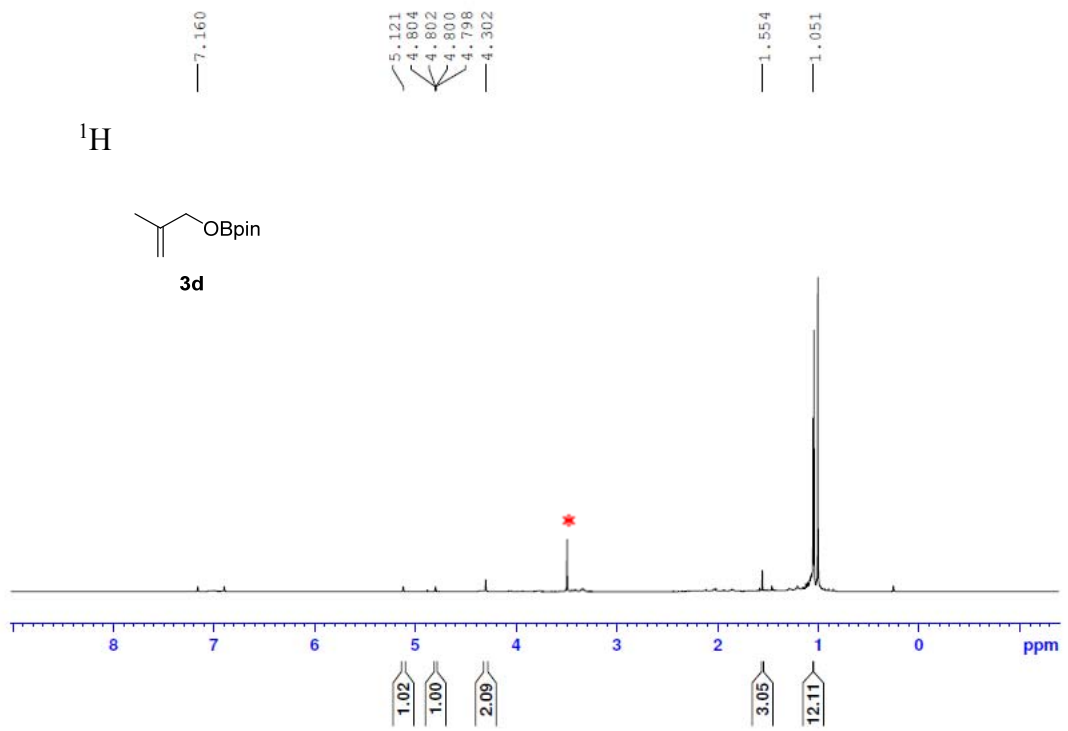
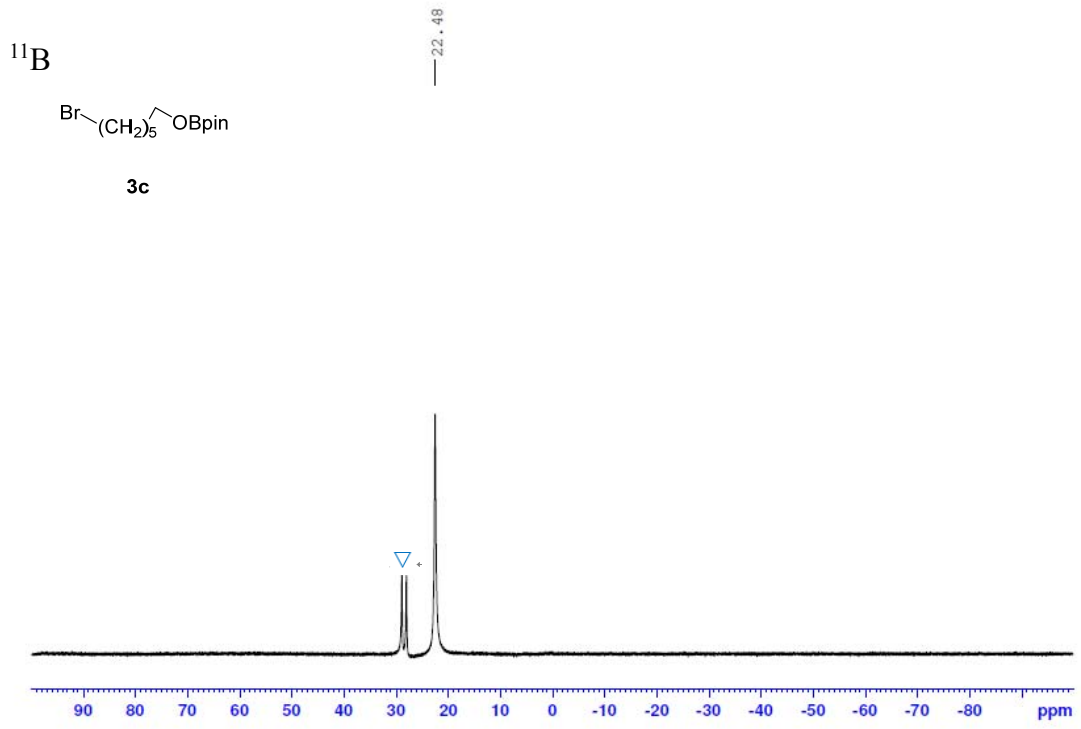
¹³C

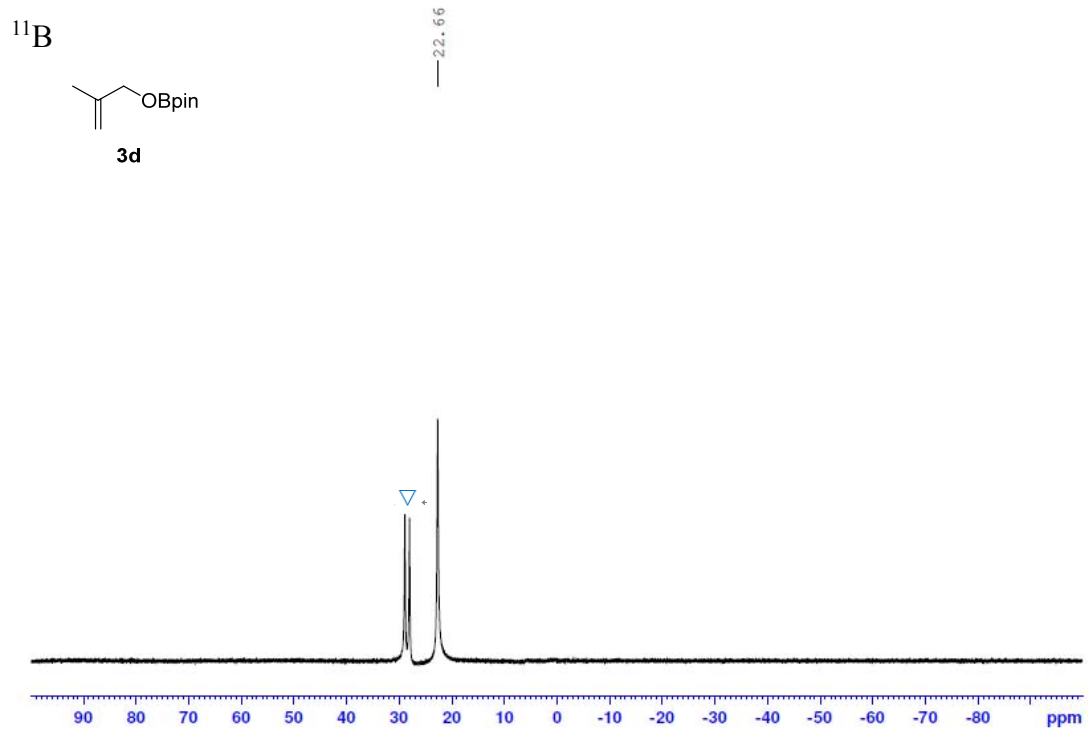
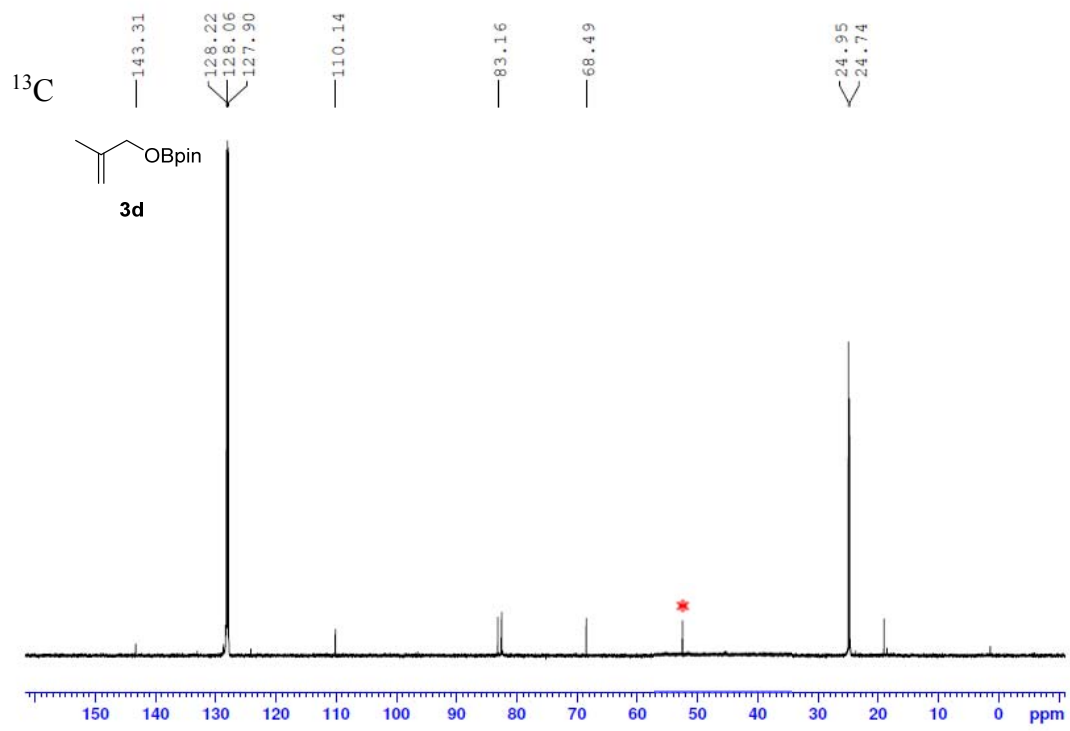


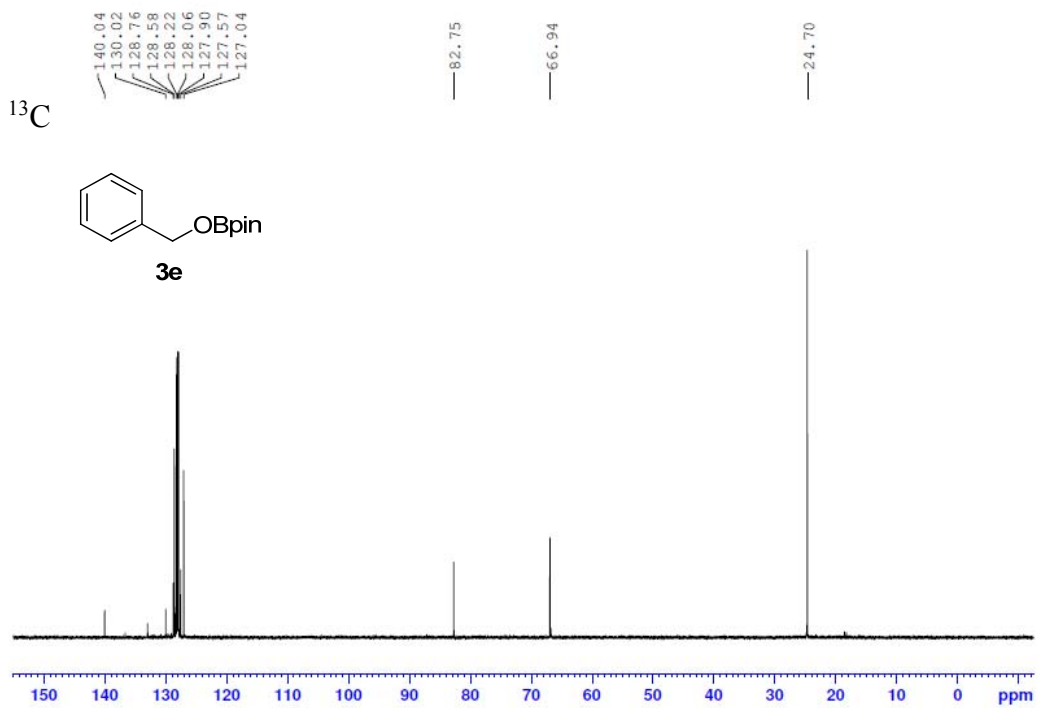
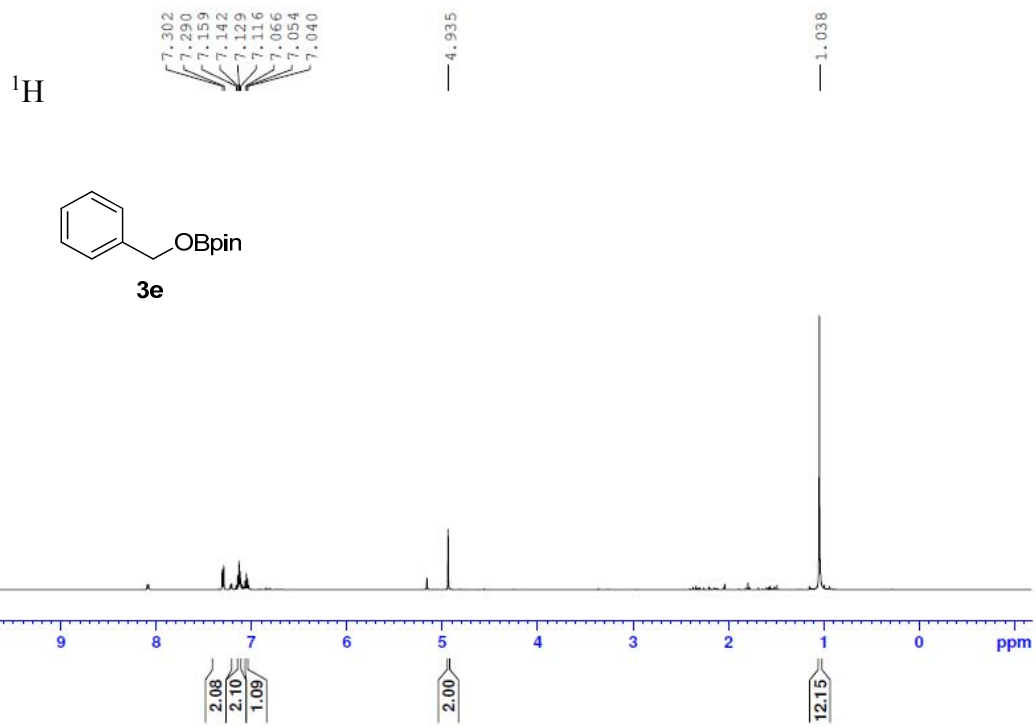


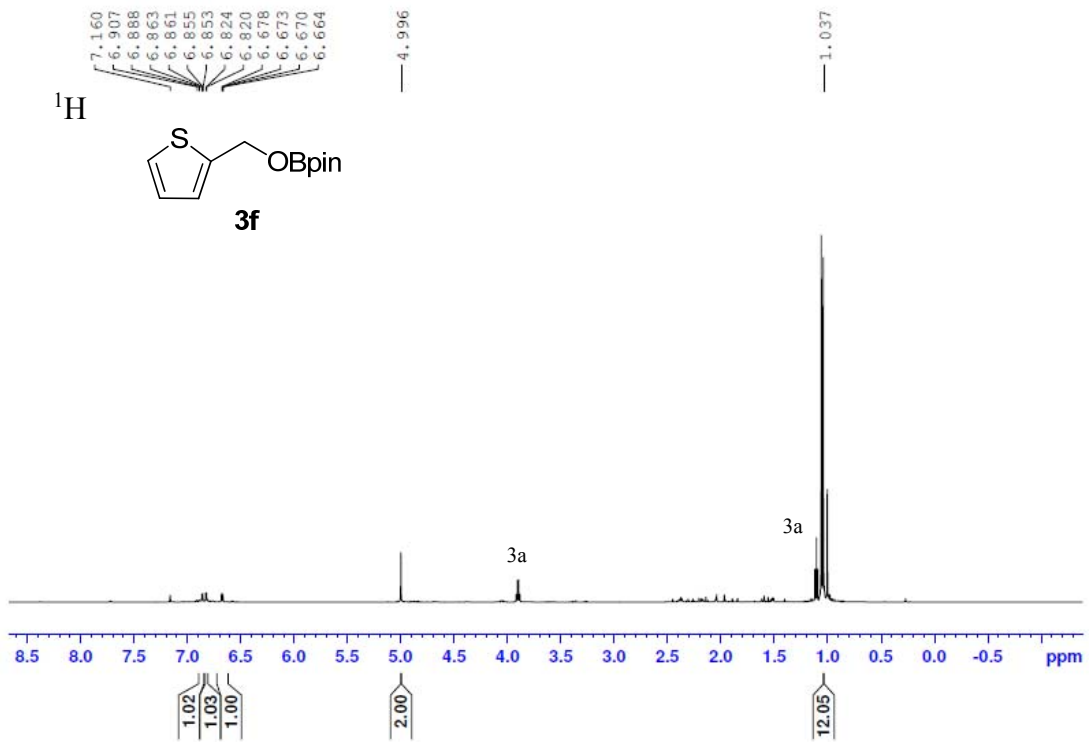
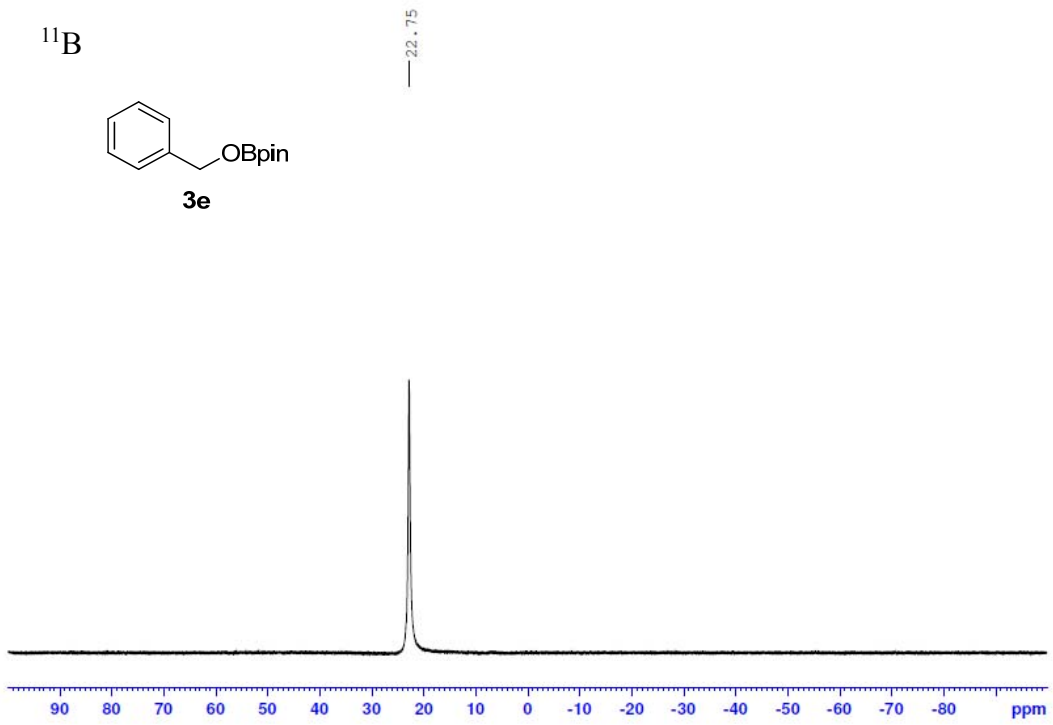


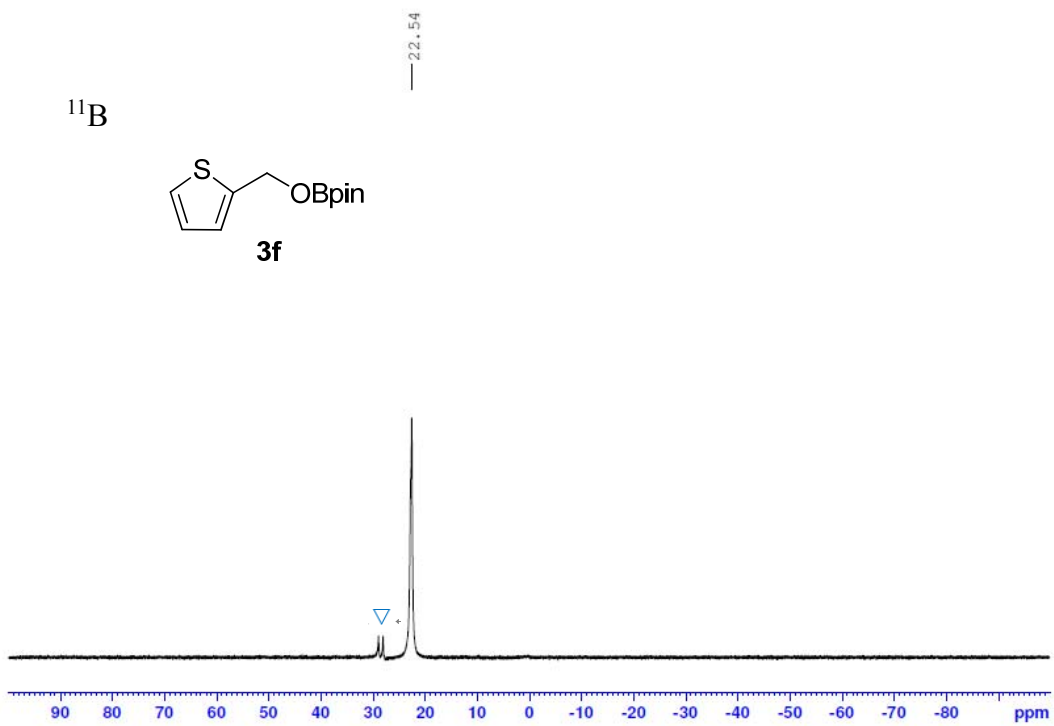
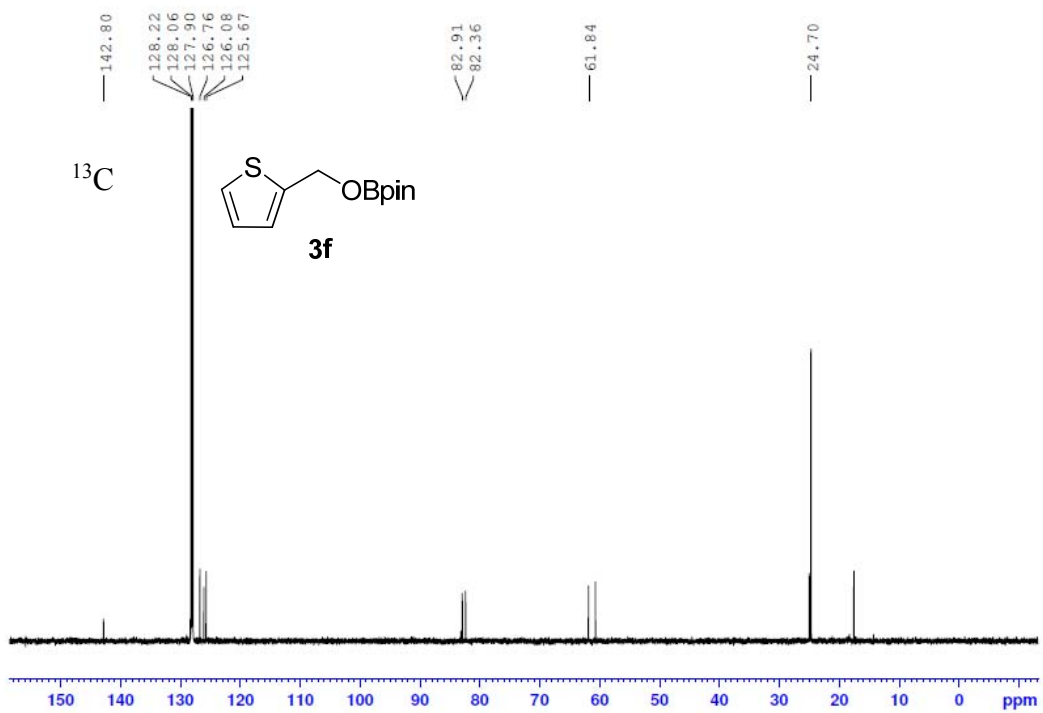


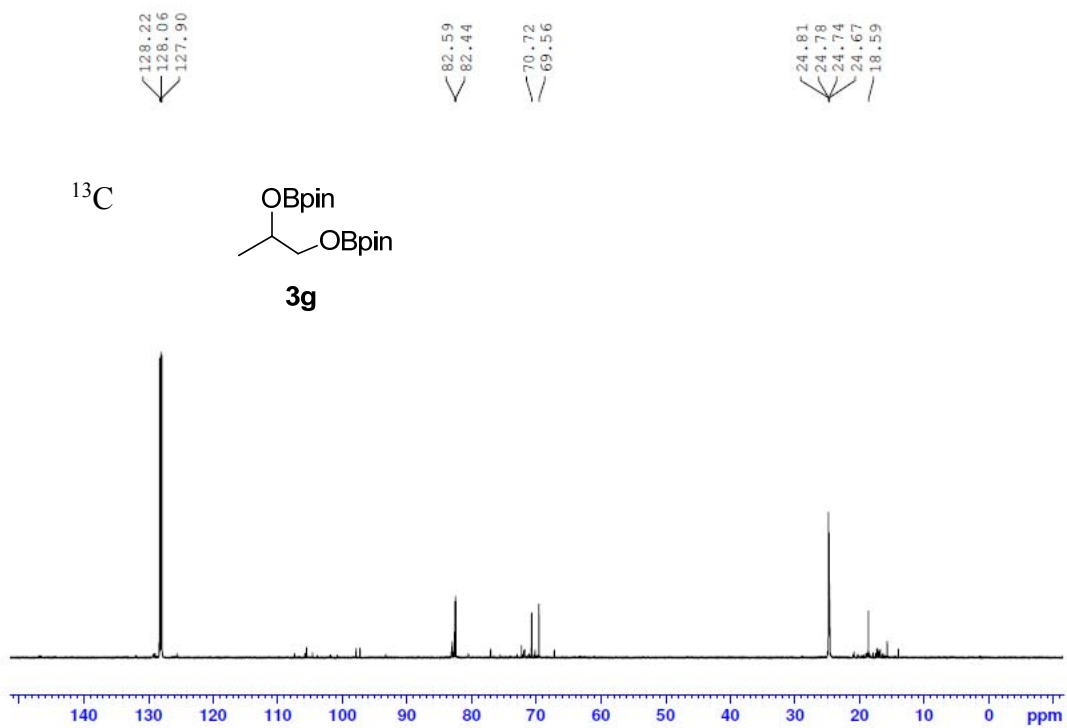
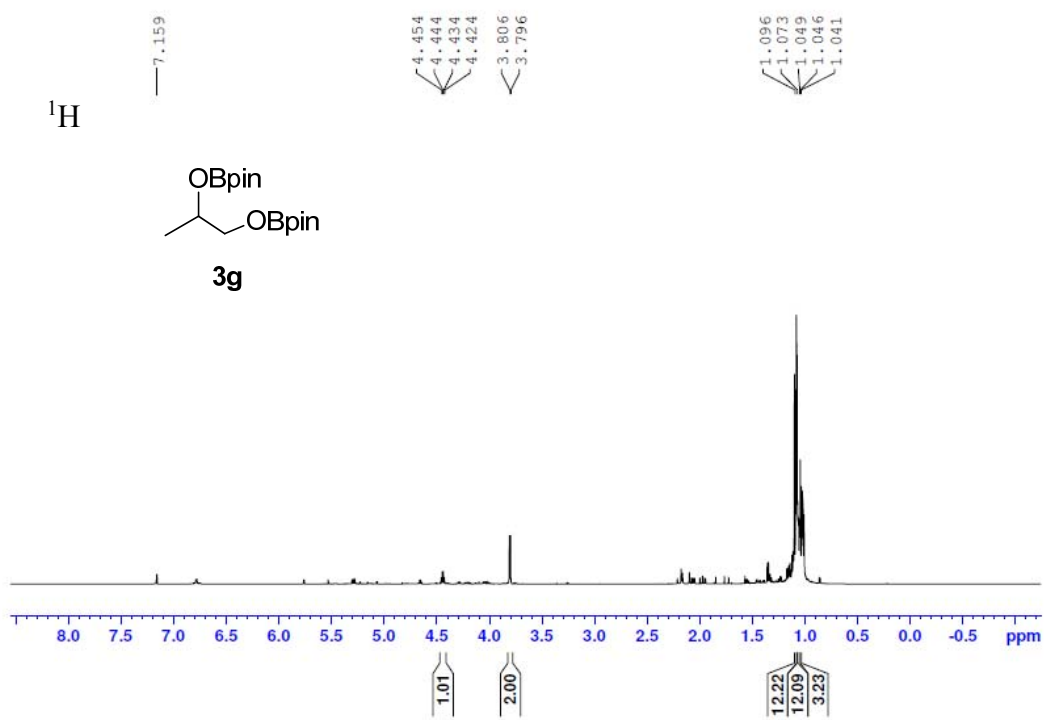


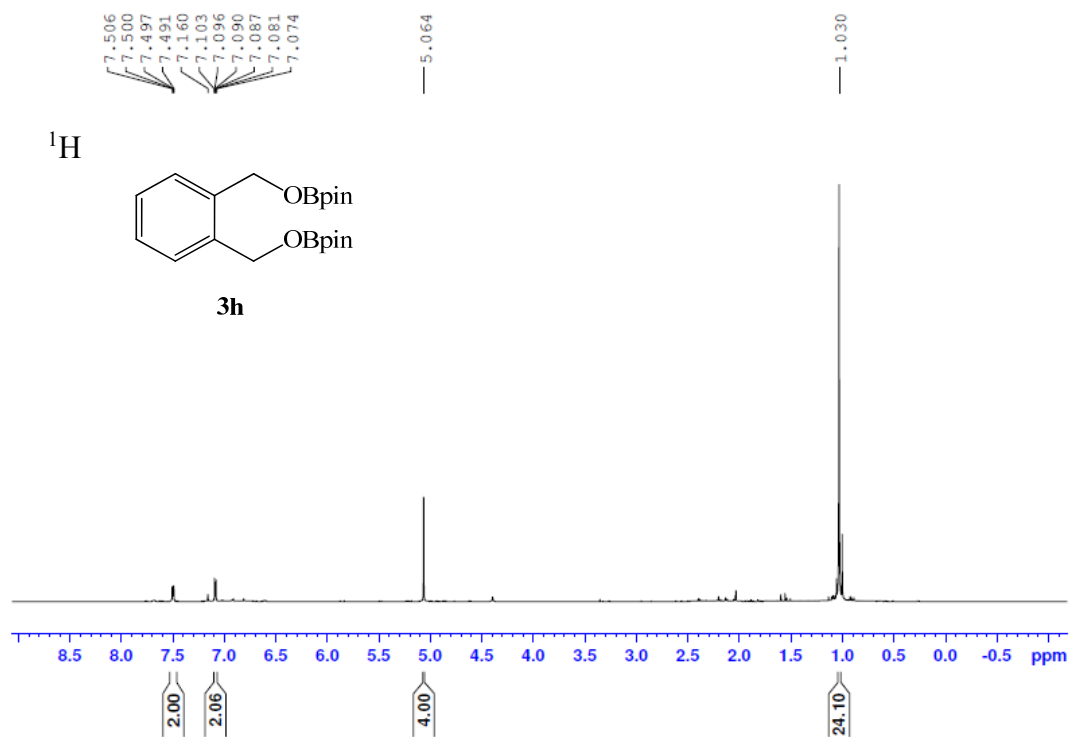
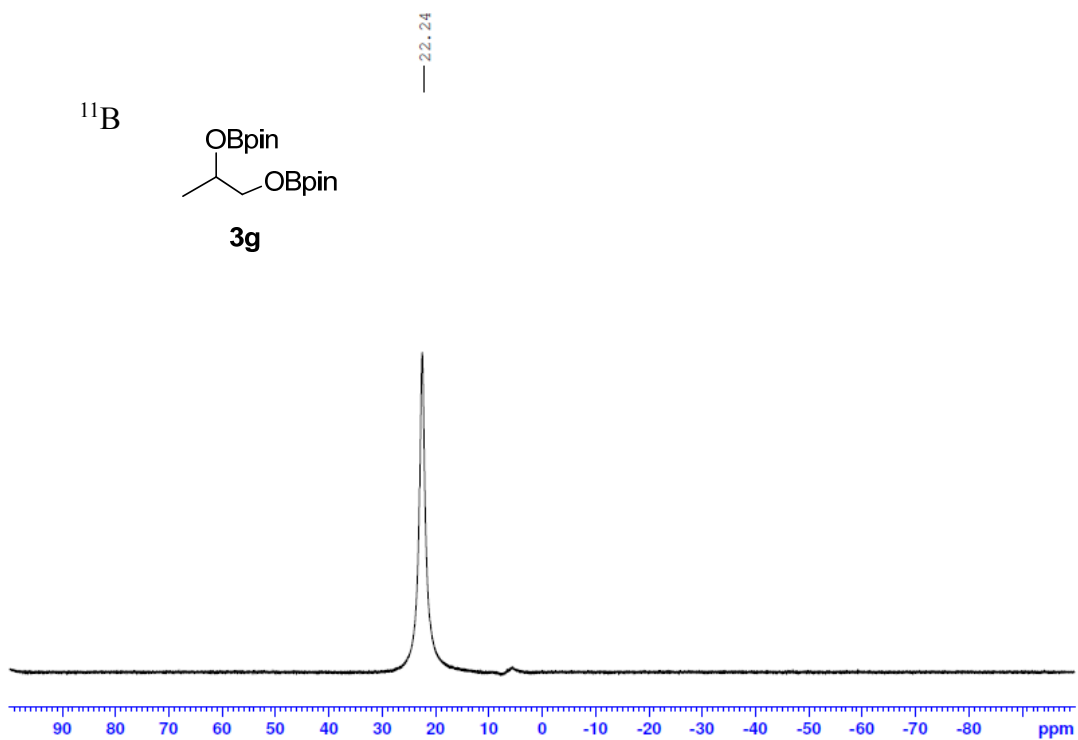


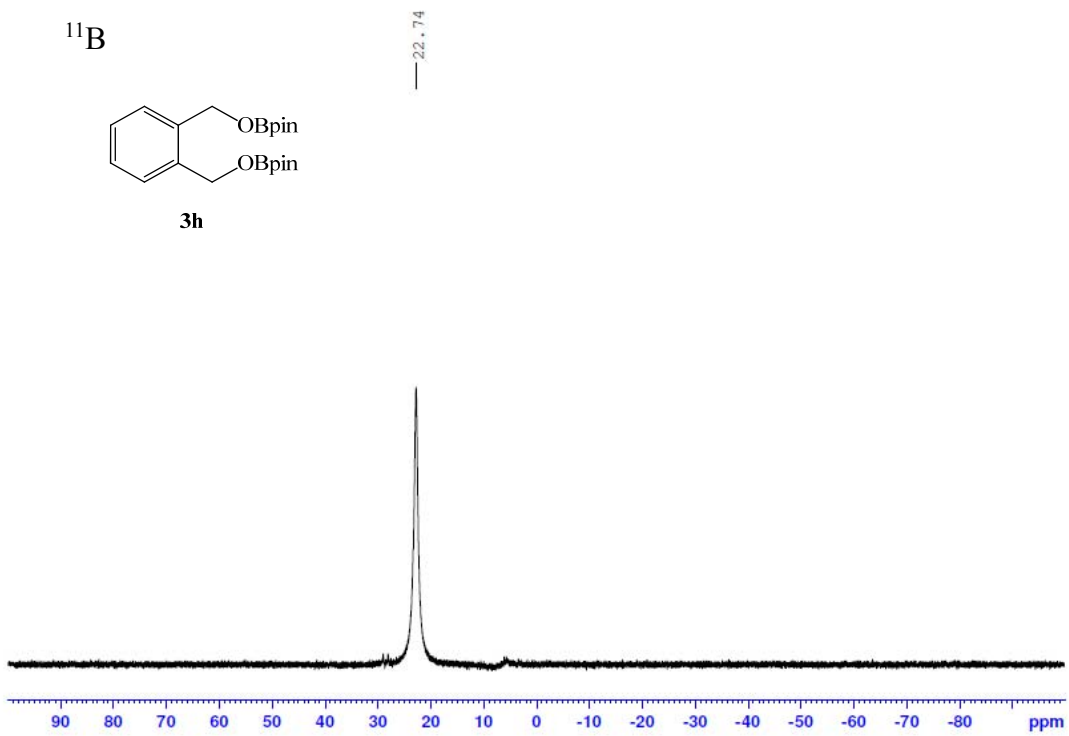
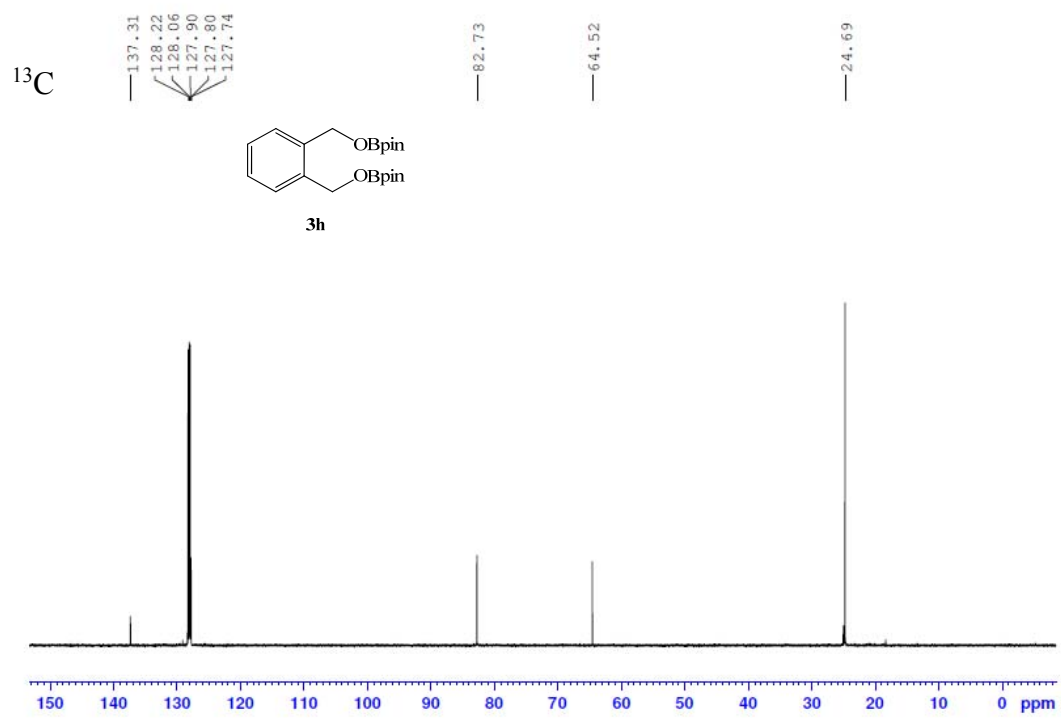


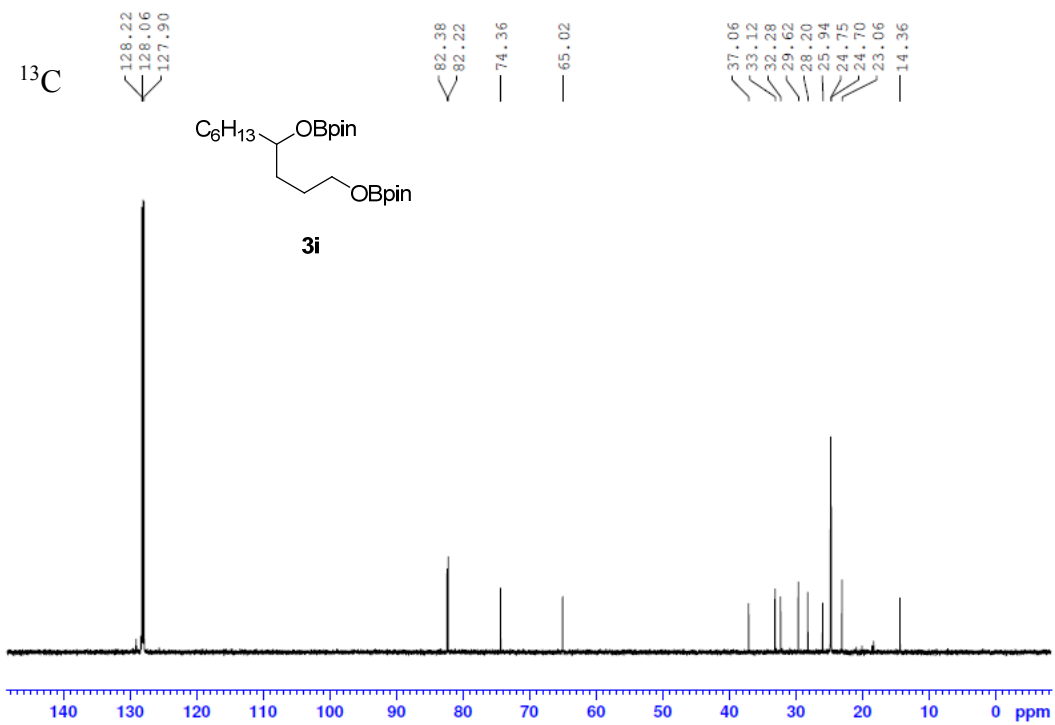
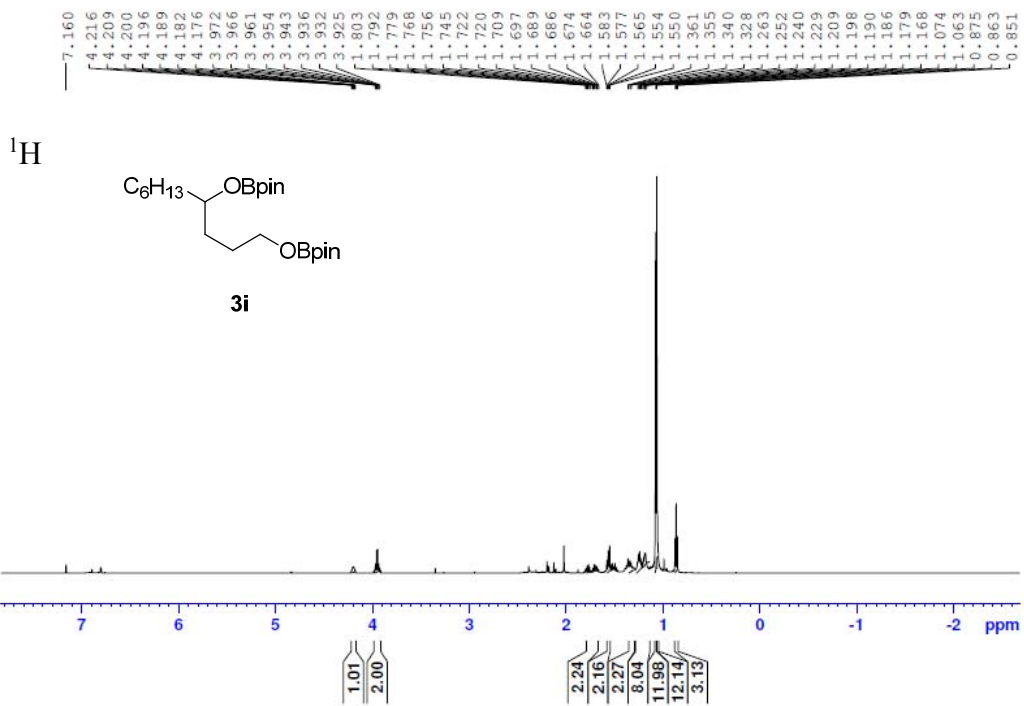


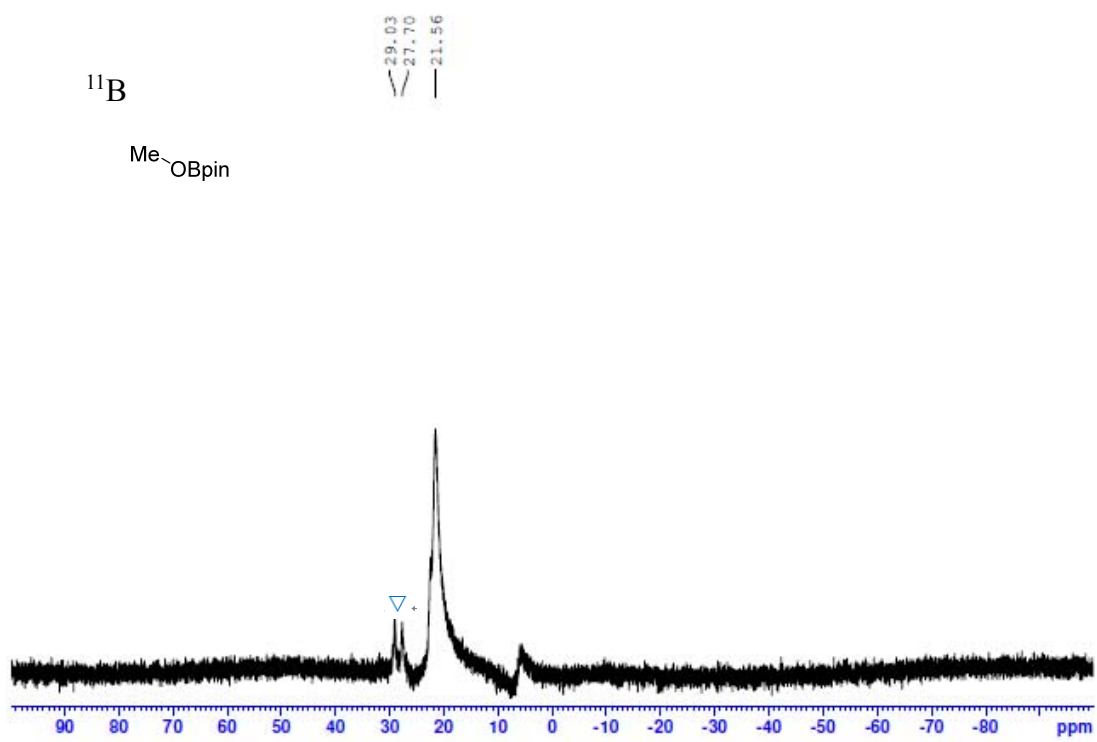
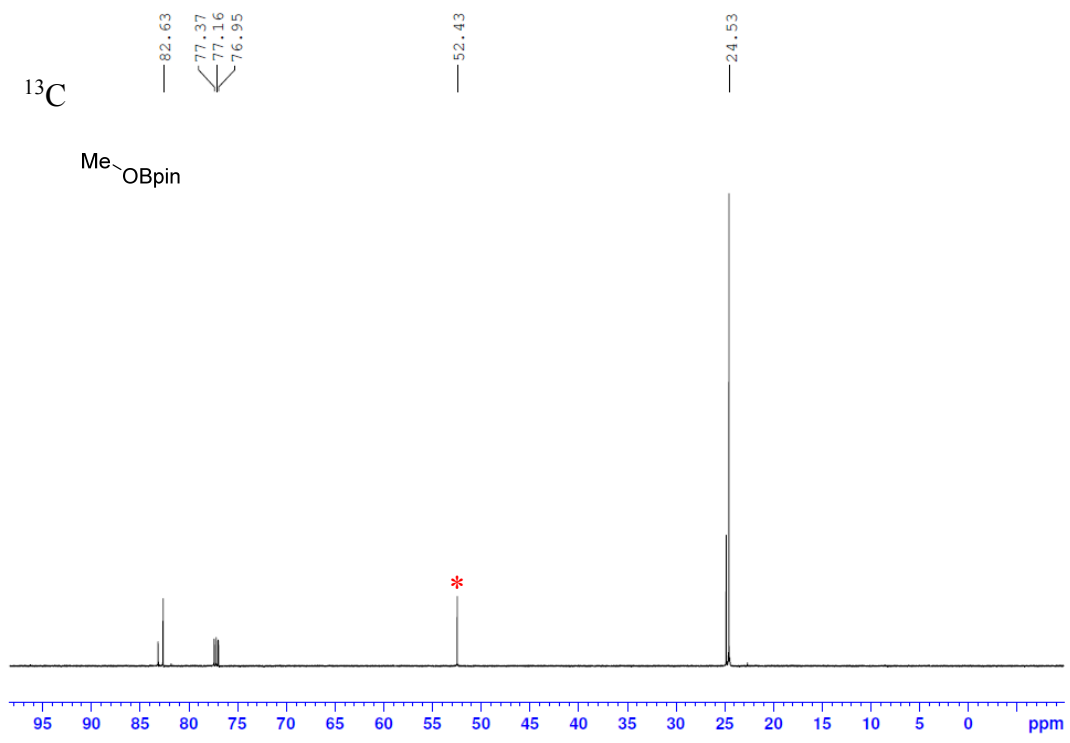


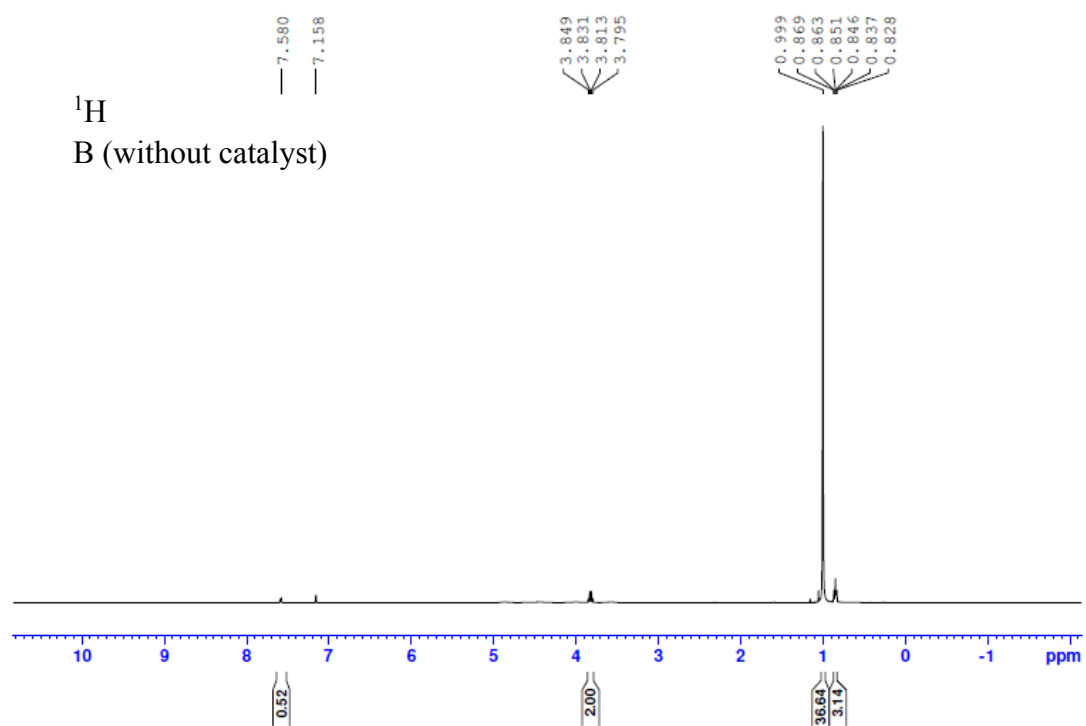
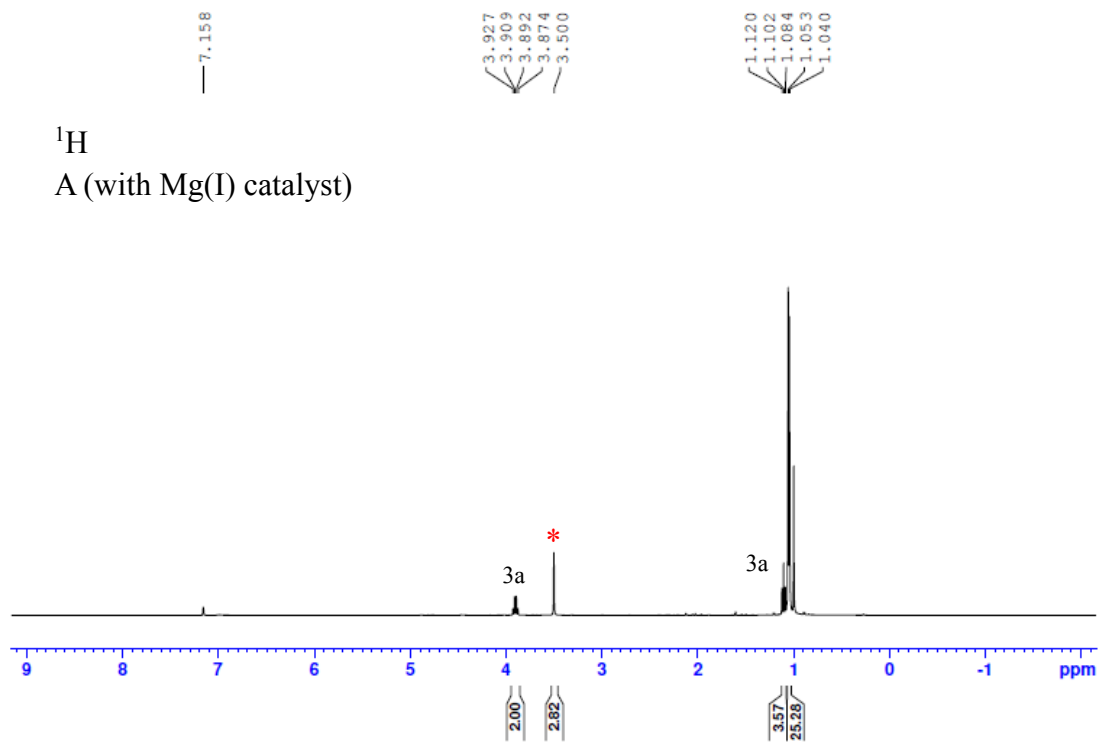












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