Supporting Information

Highly Selective Bis(imino)pyridine Iron-Catalyzed Alkene Hydroboration.

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Table of Contents:

1.) General Considerations

All reactions were carried out in an MBraun inert atmosphere (nitrogen) dry box unless otherwise stated. All glassware were stored in the oven before use. The solvents used in the dry box were dried and deoxygenated using literature procedures.[1] Deuterated solvents (Cambridge Isotope Laboratories), HBPin (Aldrich), and sodium triethylborohydride (1.0M in toluene, Aldrich) were used without further purification.

The olefins, 4-methyl-1-pentene (Aldrich), 1-octene (TCI America), *cis*-4-octene (Alfa Aesar), *tert*-butylethylene (Aldrich), cyclooctene (Aldrich), allyltrimethylsilane (Alfa Aesar), *N*,*N*-dimethylallylamine (TCI America), 4-hexen-3-one (Aldrich) , styrene (Alfa Aesar), α-methyl styrene (Aldrich), and *cis*-β-methyl styrene (Aldrich) were dried on CaH_2 and distilled under reduced pressure before use. Cyclohexene (Acros Organics) was dried on $LiAlH_4$ and distilled under reduced pressure before use.

^{1}H NMR spectra were recorded on Bruker 300 and 500 spectrophotometers operating at 300 MHz, and 500 MHz, respectively. ^{13}C NMR spectra were recorded on a Bruker 500 spectrometer operating at 125 MHz. All ^{1}H and ^{13}C NMR chemical shifts are reported relative to $SiMe_4$ using the ^{1}H (residual) and ^{13}C chemical shifts of the solvent as a secondary standard. The NMR spectra of all the hydroboration products were taken using $CDCl_3$ as the solvent unless otherwise specified. Carbons that are directly attached to boron atoms were not observed due to quadrupolar relaxation.[2] ^{1}H NMR spectra of side products were not assigned because their NMR resonances overlap with that of the major product. Only their ^{13}C NMR spectra were assigned.

GC analyses were performed using a Shimadzu GC-2010 gas chromatograph equipped with a Shimadzu AOC-20s autosampler and a Shimadzu SHRXI-5MS capillary column (15m x 250μm). The instrument was set to an injection volume of 1 μL, an inlet split ratio of 20:1, and inlet and detector temperatures of 250 °C and 275 °C, respectively. UHP-grade helium was used as carrier gas with a flow rate of 1.82 mL/min. The temperature program used for all the analyses is as follows: 60 °C, 1 min; 15 ^{o}C/min to 250 °C, 2 min.

2.) Preparation of Catalysts

(^{Mes}PDI)FeCl$_2$[3], (^{iPr}PDI)FeCl$_2$[3], [(^{Mes}PDI)Fe(N$_2$)]$_2$(μ_2-N$_2$)[4], (^{iPr}PDI)Fe(N$_2$)$_2$[5], (^{iPr}CNC)Fe(N$_2$)$_2$[6], (4-^{t}Bu-^{iPr}PDI)Fe(N$_2$)$_2$[7] and (^{Mes}PDI)CoCH$_3$[8] were prepared according to literature procedures.

3.) General Procedure for the Hydroboration of 4-methyl-1-pentene (in solvent)

Using isolated pre-catalysts:

A scintillation vial was charged with 0.014 g (0.025 mmol, 0.005 equiv) of (^{iPr}PDI)Fe(N$_2$)$_2$, 2 mL of THF, 0.084 g (1.000 mmol, 2 equiv) of 4-methyl-1-pentene and 0.064 g (0.500 mmol, 1 equiv) of HBPin (See **Table S1**). The vial was then capped and stirred for 60 minutes. The reaction was quenched by exposing the mixture to air. The solvent was evaporated in vacuo and the residue was purified through a plug of silica gel with pentane. Colorless oil was obtained after evaporation of pentane in vacuo. The isolated compound was analyzed by ^{1}H and ^{13}C NMR spectroscopy.

In-situ method:

The hydroboration reaction was carried out according to a previously reported procedure.[9] A scintillation vial was charged with 0.015 g (0.025 mmol, 0.005 equiv) of (^{iPr}PDI)FeCl$_2$, 2 mL of THF, 0.084 g (1.000 mmol, 2 equiv) of 4-methyl-1-pentene and 0.064 g (0.500 mmol, 1 equiv) of HBPin (See **Table S1**). The mixture was stirred for 1 minute then 0.433 g of a 1.0 M solution (in toluene) of NaBEt$_3$H (0.064 g, 0.015 equiv) was added. The vial was then capped and stirred for 60 minutes. The reaction was quenched by exposing the mixture to air. The solvent was evaporated in vacuo and the residue was purified through a plug of silica gel eluting with pentane. Colorless oil was obtained after evaporation of pentane in vacuo. The isolated compound was analyzed by ^{1}H and ^{13}C NMR spectroscopy.

Table S1. Catalytic Hydroboration of 4-methyl-1-pentene (in solvent).[a,b]

Entry[c]	Catalyst	Mass of Catalyst	Solvent
1	(^{iPr}PDI)Fe(N$_2$)$_2$	0.014 g	THF
2			C_6H_6
3	[(^{Mes}PDI)Fe(N$_2$)]$_2$(μ_2-N$_2$)	0.012 g	THF
4			C_6H_6

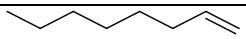
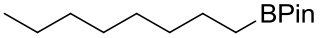
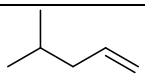
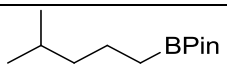
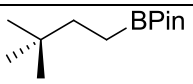
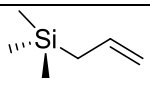
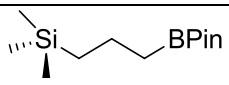
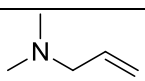
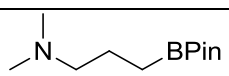
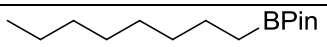
5	(4-^{t}Bu-^{iPr}PDI)Fe(N_2)$_2$	0.016 g	THF
6	(^{iPr}CNC)Fe(N_2)$_2$	0.016 g	THF
7			C_6H_6
9	(^{Mes}PDI)$FeCl_2$/ $NaHBEt_3$	0.013 g	THF

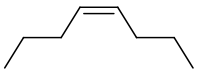
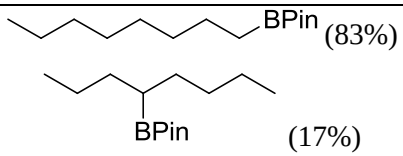
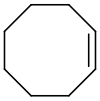
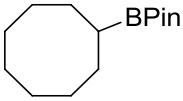
[a] Entries 1-7: catalyst (0.025 mmol, 0.005 equiv), 0.084 g of 4-methyl-1-pentene (1.000 mmol, 2 equiv), 0.064 g of HBPin (0.500 mmol, 1 equiv) in solvent (2 mL) at 25°C. [b]Entry 9: catalyst (0.025 mmol, 0.005 equiv), 0.084 g of 4-methyl-1-pentene (1.000 mmol, 2 equiv), 0.064 g of HBPin (0.500 mmol, 1 equiv), $NaBEt_3H$ (0.0075 mmol, 0.15 equiv) in solvent (2 mL) at 25°C. [c]Entry numbers correspond to those described in the main paper.

4.) General Procedure for the Hydroboration of Liquid Olefins (Neat)

A scintillation vial was charged with 0.100 g of olefin, a stoichiometric amount of HBPin and then with 1 mol% of the catalyst (See **Table S2** for the amounts in mmol). The vial was then capped and stirred until the reaction was complete. The reaction was monitored by the analysis of an aliquot of the mixture by GC-FID. The reaction was quenched by exposing the mixture to air. The mixture was dissolved in $CDCl_3$, passed through a plug of silica gel in a Pasteur pipette and then analyzed by NMR spectroscopy without any further purification.

Table S2. Catalytic Hydroboration of Liquid Olefins (Neat).

Olefin	**Catalyst**	**Product(s)**	**mmol of Substrate**	**mmol of HBPin**	**mmol of Catalyst**
	1 2		0.891	0.891	0.009
	1 2		1.188	1.118	0.012
	1 2		1.188	1.188	0.019
	1 2		0.875	0.875	0.009
	1 2		1.174	1.174	0.012
	1		0.891	0.891	0.009

	2				
	1		0.907	0.907	0.009
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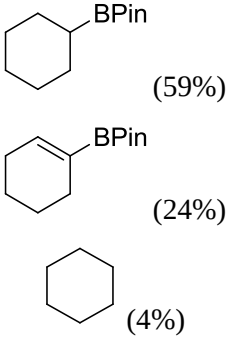
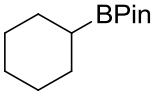
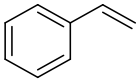
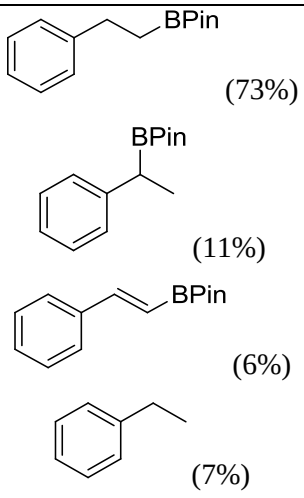
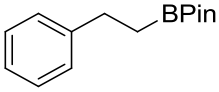
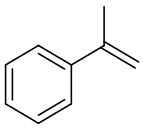
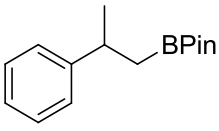
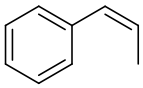
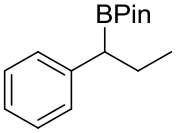
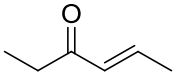
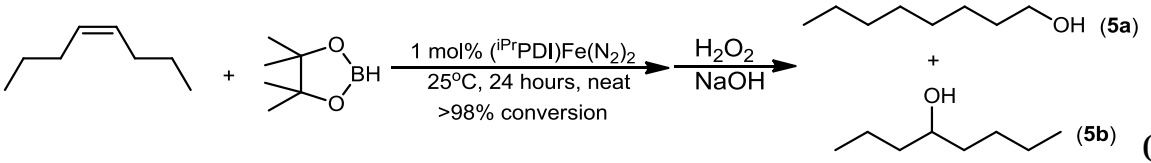
	1		1.217	1.217	0.012
	2				
	1		0.960	0.960	0.010
	2				
	1		0.846	0.846	0.008
	2				

	1 2		0.846	0.846	0.008
	1	No productive hydroboration	1.019	1.019	0.010

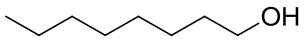
Conditions: HBPin (1 equiv), olefin (1 equiv) and catalyst (0.001 equiv), neat at 25°C. **Catalysts:** 1: $[(^{Mes}PDI)Fe(N_2)]_2(\mu_2\text{-}N_2)$, 2: $(^{iPr}PDI)Fe(N_2)_2$.

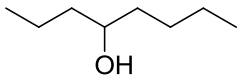
5.) Procedure for Hydroboration-Oxidation

The reaction of *cis*-4-octene with HBPin using $(^{iPr}PDI)Fe(N_2)_2$ as the catalyst yielded 2 products in a 5:1 ratio, with 1-octylboronate (**8a**) as the predominant product. To confirm the identity of the second hydroboration product, oxidation with hydrogen peroxide was done according to a previously reported procedure (**Eq S1**).[10] The oxidized mixture contained 1-octanol (**5a**) and 4-octanol (**5b**) (determined by ^{13}C NMR). The second hydroboration product in the reaction of *cis*-4-octene with HBPin was then identified to be the 4-octylboronate (**8f**).



(Eq. S1)

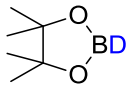
 **(5a)**: ^{13}C NMR (126 MHz, $CDCl_3$) δ = 62.80, 32.69, 31.85, 29.45, 29.32, 25.79, 22.69, 14.13.

 **(5b)**: ^{13}C NMR (126 MHz, $CDCl_3$) δ = 71.69, 39.55, 37.09, 27.85, 22.80, 18.84, 14.16, 14.12.

6.) Preparation of DBPin

In an inert atmosphere glove box, a thick-walled glass vessel was charged with 2.00 g (15.6 mmol, 1 equiv) of HBPin and 0.018 g (0.04 mmol, 0.0025 equiv) of $(^{Mes}PDI)CoCH_3$. The glass vessel was sealed, removed from the glove box and then attached to a vacuum line. The mixture was frozen in liquid nitrogen, degassed and then purged with 4 atmospheres of D_2(g). The mixture was stirred at 23°C for 24 hours. The product was isolated from the cobalt catalyst via an air-free vacuum distillation. The extent of

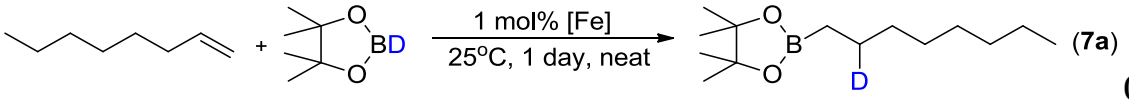
^{2}H incorporation was determined to be 83% by the integration of the ^{2}H resonances against a known amount of an internal standard (C_6D_6).



(6a): ^{2}H NMR (77 MHz, C_6H_6): δ 4.45(broad)

7.) Deuterium Labeling Experiments

The reaction of 1-octene with DBPin (**6a**) using either [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) or (^{iPr}PDI)Fe(N_2)$_2$ (**Eq. S2**) yielded a product with deuterium exclusively in the 2-position of the hydrocarbyl chain. This result was established by ^{2}H NMR (**Figure S1**) and quantitative ^{13}C NMR spectroscopy (triplet at the C2 position, **Figure S2** and **Figure S3**). Benzene-d_6 was for the analysis of ^{2}H NMR spectra since the peaks for the product are well-separated in this solvent.



(Eq. S2)

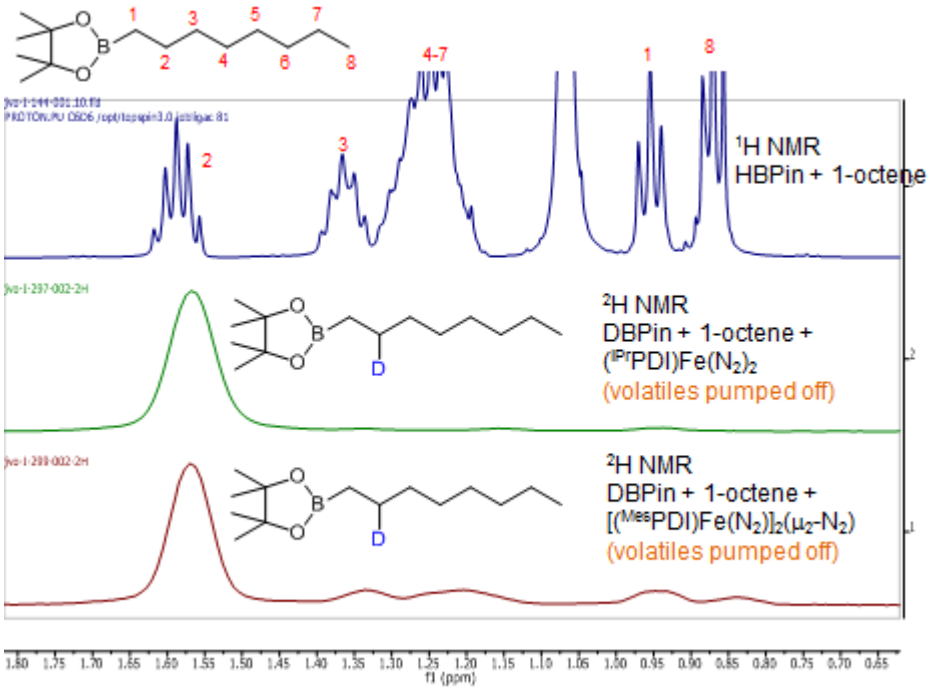


Figure S1. ^{2}H NMR spectra of **7a** using [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) and (^{iPr}PDI)Fe(N_2)$_2$ as catalysts.

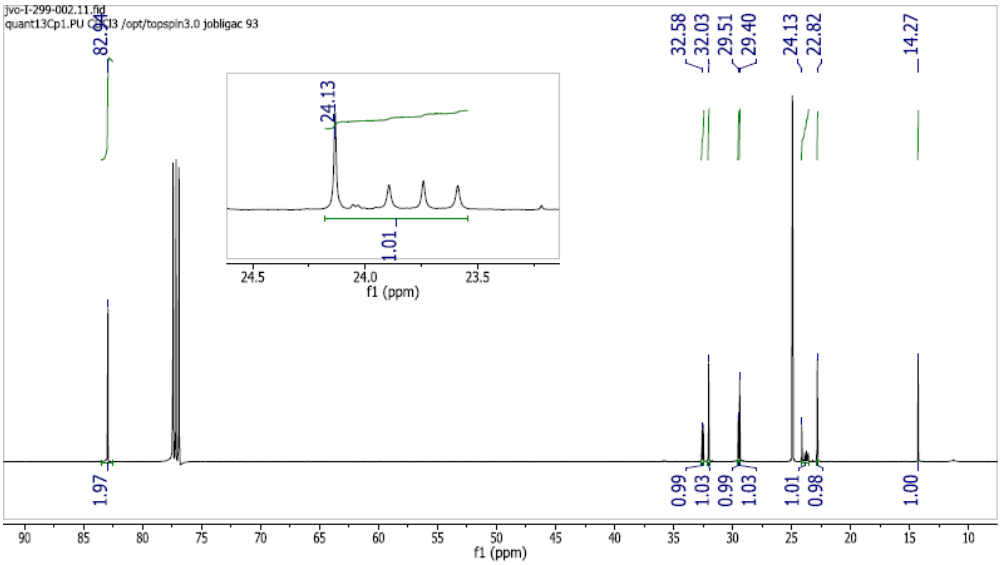


Figure S2. Quantitative ^{13}C NMR spectrum of **7a** using [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) as the catalyst.

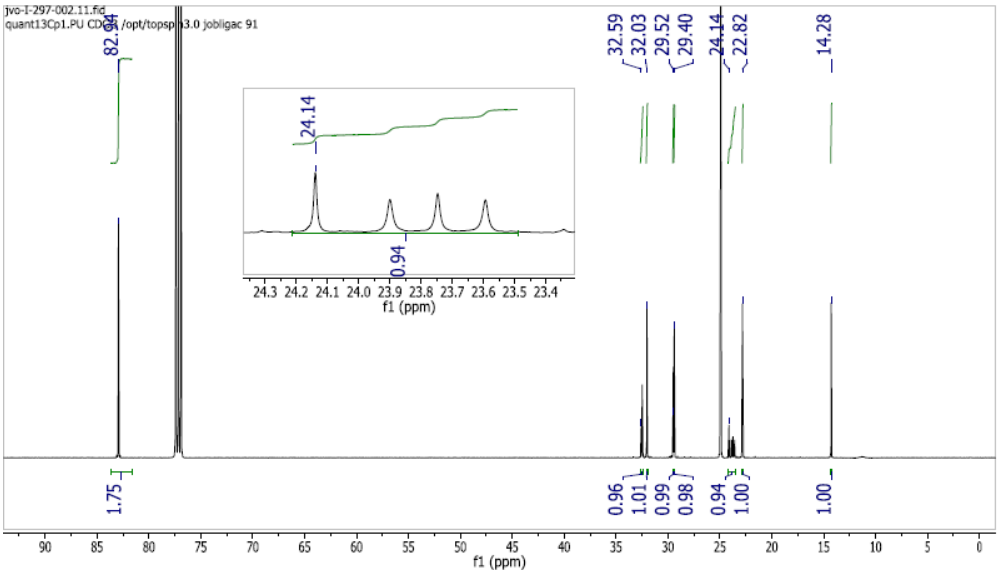
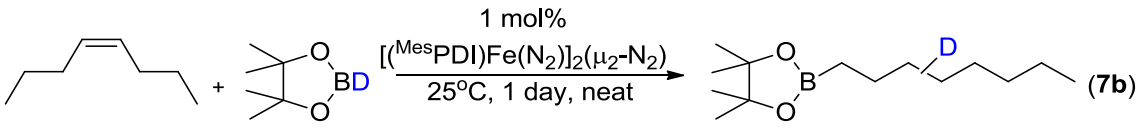


Figure S3. Quantitative ^{13}C NMR spectrum of **7a** using (^{iPr}PDI)Fe(N_2)$_2$ as the catalyst.

The reaction of *cis*-4-octene with DBPin (**6a**) using [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) yielded a product with deuterium at every position of the hydrocarbyl chain. The majority of the deuterium was located on the C2-C5 carbons. This result was established by ^{2}H NMR spectroscopy (**Figure S4**).



(Eq. S3)

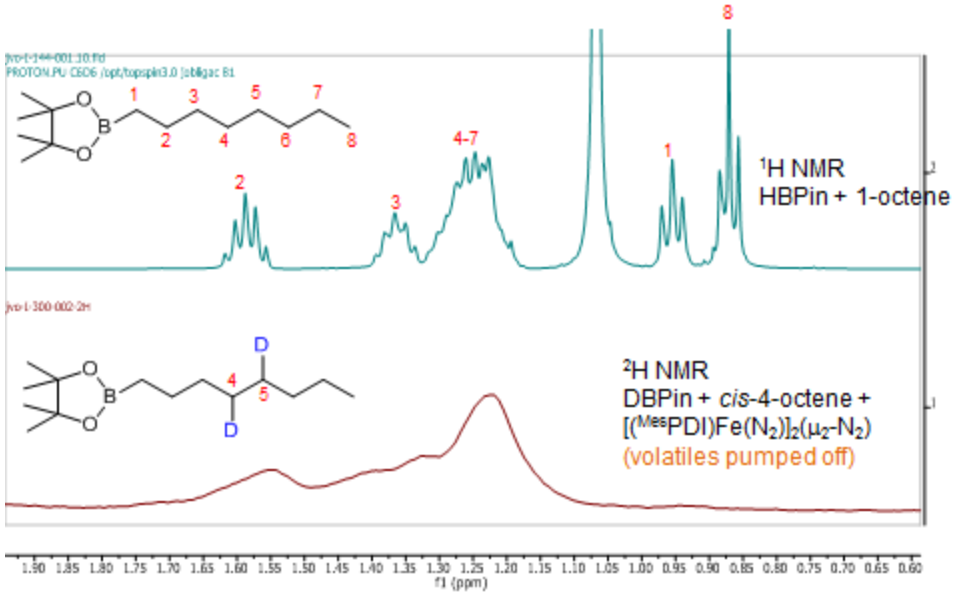
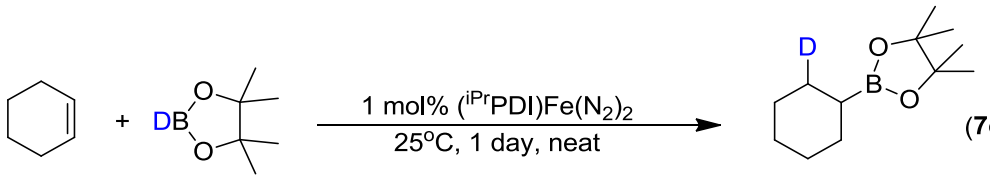


Figure S4. ^{2}H NMR spectrum **7b**.

The reaction of cyclohexene with DBPin (**6a**) using (^{iPr}PDI)Fe(N_2)$_2$ yielded a product with deuterium exclusively in the 2-position of the hydrocarbyl chain (**7c**). This result was established by quantitative ^{13}C NMR spectroscopy (triplet at the C2 position, **Figure S5**).



(Eq. S4)

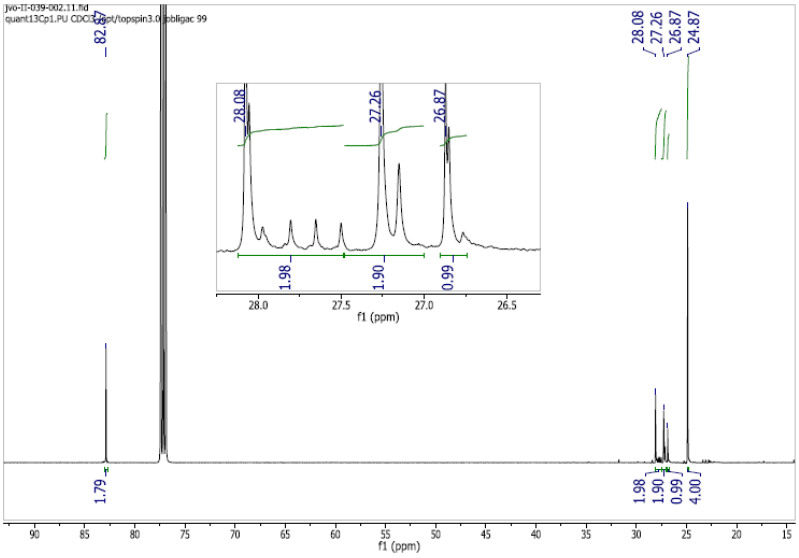
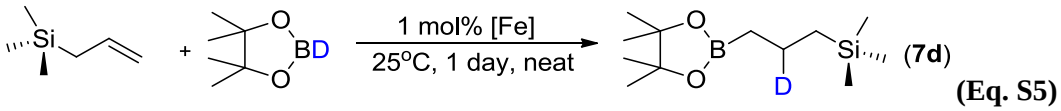


Figure S5. Quantitative ^{13}C NMR spectrum of **7c**.

The reaction of allyltrimethylsilane with DBPin using either [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) or (^{iPr}PDI)Fe(N_2)$_2$ (**Eq. S5**) yielded a product with deuterium exclusively in the 2-position of the hydrocarbyl chain (**7d**). This result was established by ^{2}H NMR (**Figure S6**) and quantitative ^{13}C NMR spectroscopy (triplet at the C2 position, **Figure S7** and **Figure S8**).



(Eq. S5)

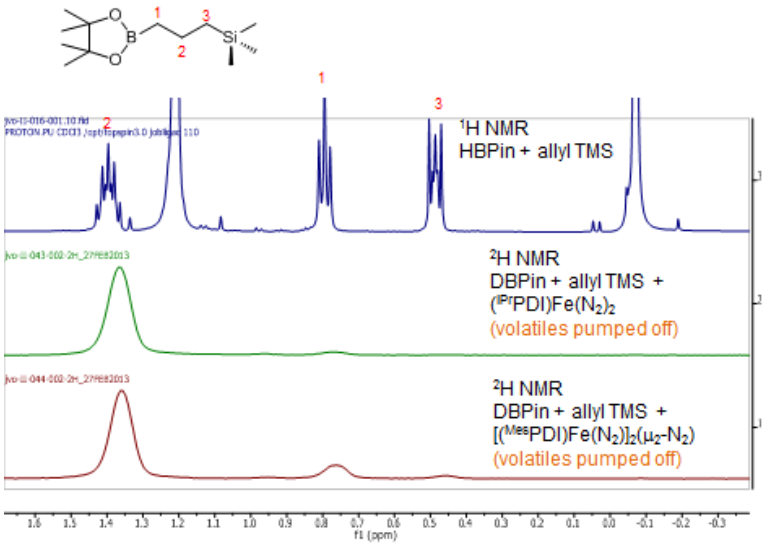


Figure S6. ^{2}H NMR spectra of **7d** using [(^{Mes}PDI)Fe(N_2)]$_2$(μ_2-N_2) and (^{iPr}PDI)Fe(N_2)$_2$ as catalysts.

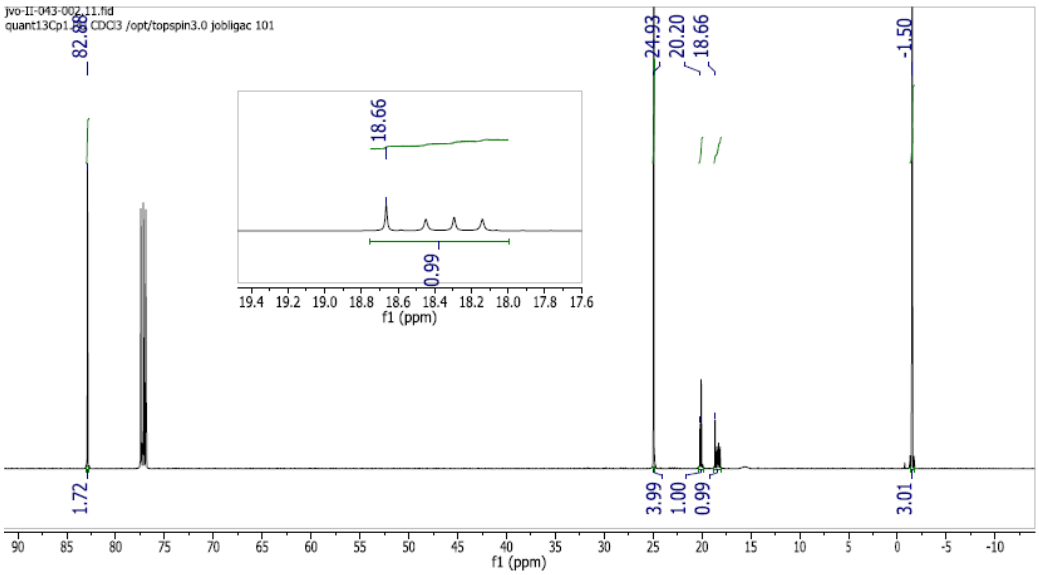


Figure S7. Quantitative ^{13}C NMR spectrum of **7d** using $[(^{Mes}PDI)Fe(N_2)]_2(\mu_2\text{-}N_2)$ as the catalyst.

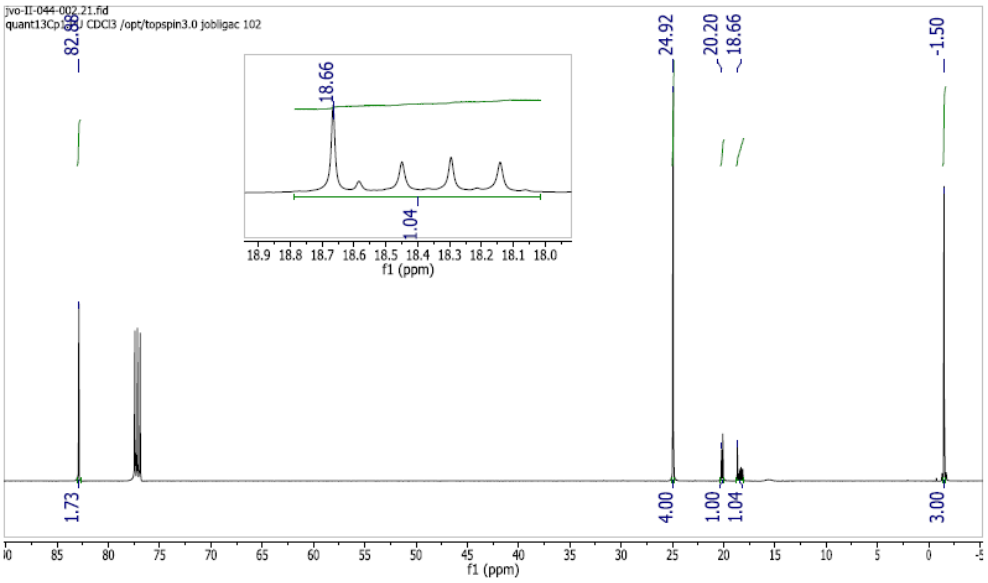
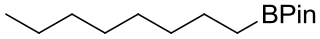
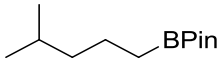


Figure S8. Quantitative ^{13}C NMR spectrum of **7d** using $(^{iPr}PDI)Fe(N_2)_2$ as the catalyst.

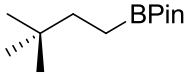
8.) NMR Resonances for Hydroboration Products



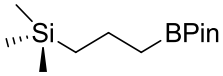
(8a): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.43 – 1.33 (m, 2H), 1.34 – 1.01 (m, 22H), 0.84 (t, 3H), 0.73 (t, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.87, 32.54, 31.99, 29.48, 29.36, 24.87, 24.09, 22.78, 14.22.



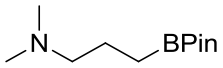
(8b): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.50 – 1.40 (m, 1H), 1.37 – 1.28 (m, 2H), 1.16 (s, 12H), 1.11 – 1.06 (m, 2H), 0.78 (d, *J*=6.9, 6H), 0.66 (t, *J*=7.9, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.78, 42.00, 27.84, 24.84, 22.69, 21.84.



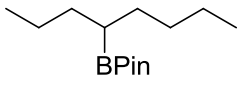
(8c): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.21 (t, 2H), 1.16 (s, 12H), 0.76 (s, 9H), 0.62 (t, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.82, 37.75, 30.84, 28.89, 24.85.



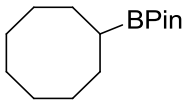
(8d): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.45 – 1.35 (m, 2H), 1.21 (s, 12H), 0.79 (t, 2H), 0.49 (t, 2H), -0.07 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.89, 24.93, 20.21, 18.67, -1.50.



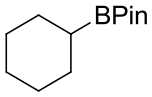
(8e): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 2.22 – 2.12 (t, 2H), 2.09 (s, 6H), 1.51 – 1.38 (m, 2H), 1.08 (s, 12H), 0.58 (t, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.21, 62.09, 45.31, 24.88, 21.63.



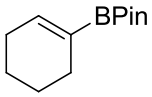
(8f): ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.80, 33.87, 31.66, 31.21, 24.87, 23.08, 22.52, 14.55, 14.20.



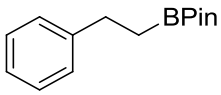
(8g): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.73 – 1.64 (m, 2H), 1.60 (q, *J*=8.9, 8.4, 2H), 1.54 – 1.41 (m, 10H), 1.19 (s, 12H), 1.10 – 1.05 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.79, 27.59, 27.04, 26.89, 26.68, 24.78.



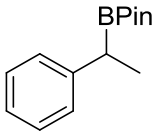
(8h): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.68 – 1.50 (m, 5H), 1.36 – 1.24 (m, 5H), 1.21 (s, 12H), 1.02 – 0.91 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 82.84, 28.06, 27.24, 26.86, 24.85.



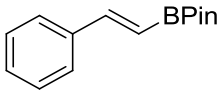
(8i): ^{13}C NMR (126 MHz, $CDCl_3$) δ = 143.15, 83.12, 26.73, 26.21, 24.93, 22.64, 22.27.



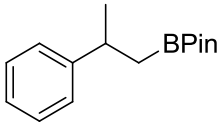
(8j): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.32 – 7.23 (m, 4H), 7.21 – 7.16 (m, 1H), 2.79 (t, 2H), 1.25 (s, 12H), 1.19 (t, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 144.41, 128.23, 128.04, 125.55, 83.11, 30.01, 24.86.



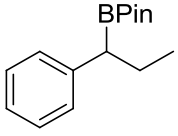
(8k): ^{13}C NMR (126 MHz, $CDCl_3$) δ = 144.70, 136.67, 128.09, 126.84, 82.99, 24.38, 16.92.



(8l): ^{13}C NMR (126 MHz, $CDCl_3$) δ = 149.37, 137.22, 128.70, 127.55, 124.88, 83.06, 24.42.



(8m): ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.30 – 7.23 (m, 4H), 7.19 – 7.13 (m, 1H), 3.05 (h, *J*=7.2, 1H), 1.28 (d, 3H), 1.17 (broad s, 14H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 149.25, 128.26, 126.69, 125.77, 83.07, 35.90, 25.04, 24.85, 24.77.



(8n): ^{1}H NMR (501 MHz, $CDCl_3$) δ = 7.40 – 7.16 (m, 8H), 2.27 (t, 1H), 1.98 – 1.87 (m, 1H), 1.82 – 1.68 (m, 1H), 1.26 (s, 6H), 1.24 (s, 6H), 0.96 (t, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ = 149.25, 128.26, 126.69, 125.77, 83.07, 35.90, 25.04, 24.85, 24.77.

9.) NMR Spectra for Hydroboration Products

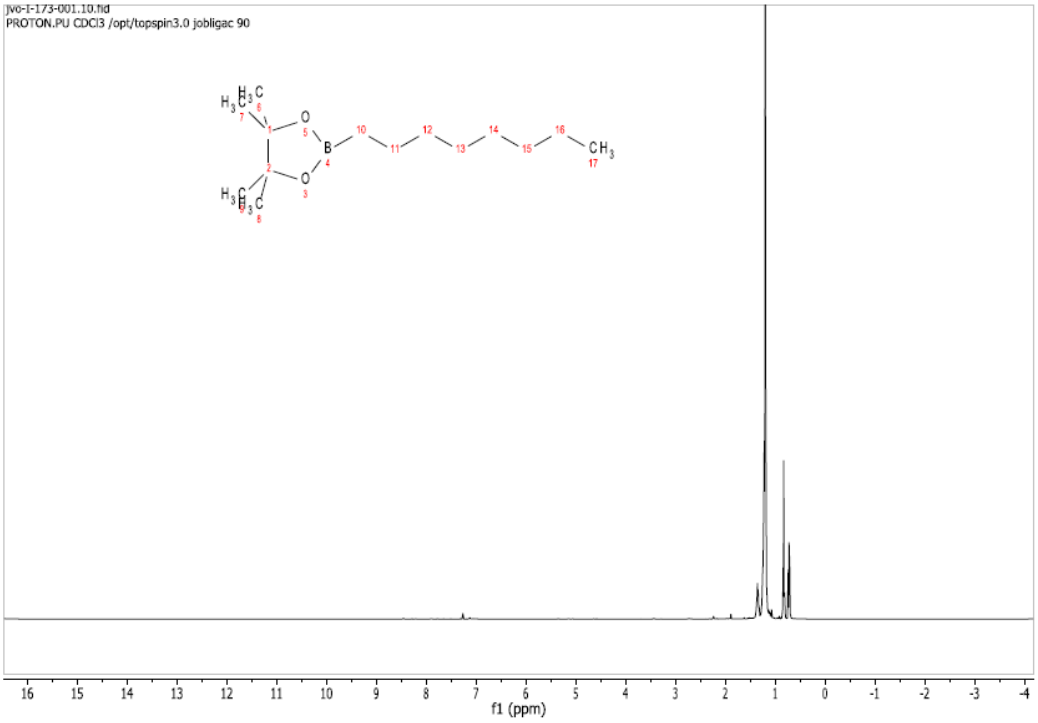


Figure S9. ^{1}H NMR spectrum of **8a** in $CDCl_3$ (500 MHz).

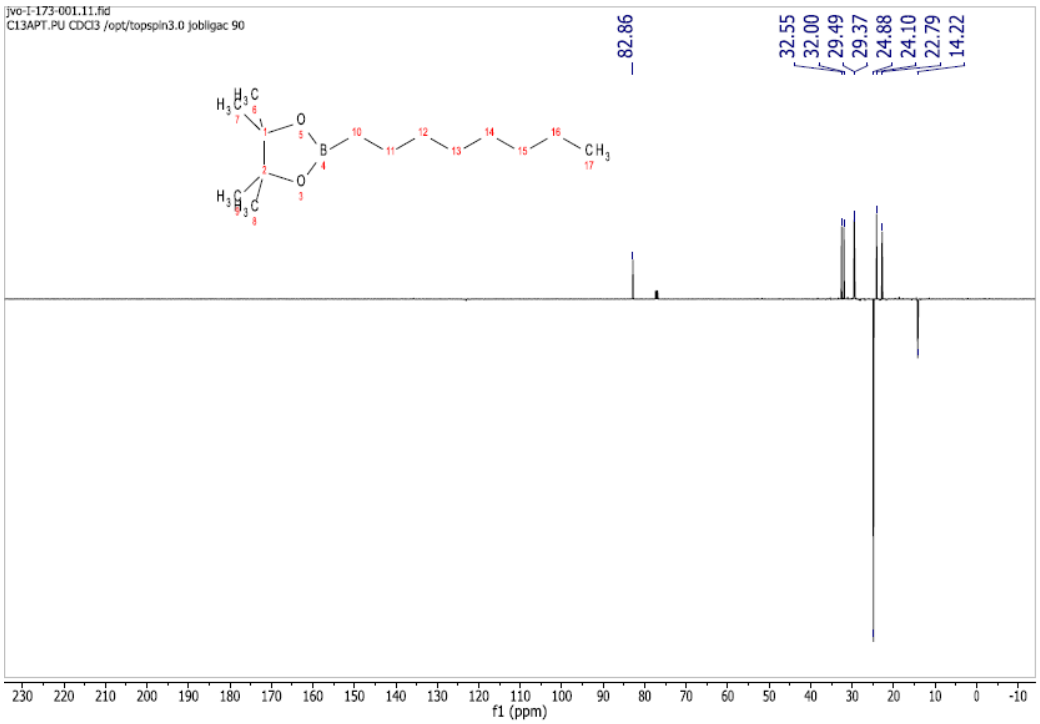


Figure S10. ^{13}C(APT) NMR spectrum of **8a** in $CDCl_3$ (126 MHz).

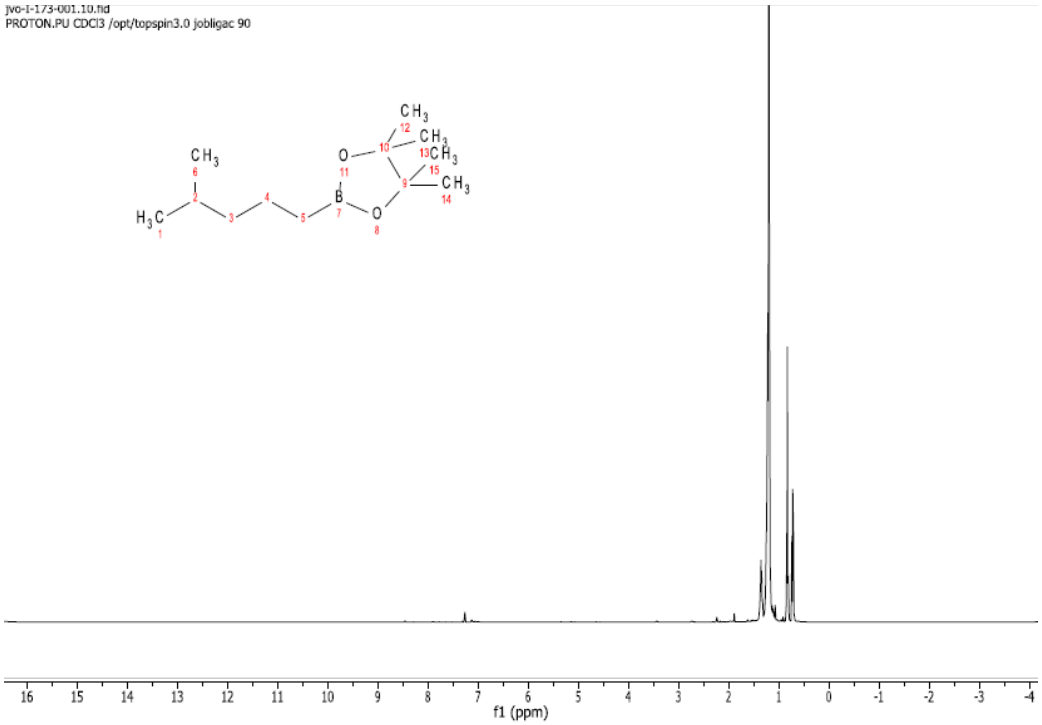


Figure S11. ^{1}H NMR spectrum of **8b** in $CDCl_3$ (500 MHz).

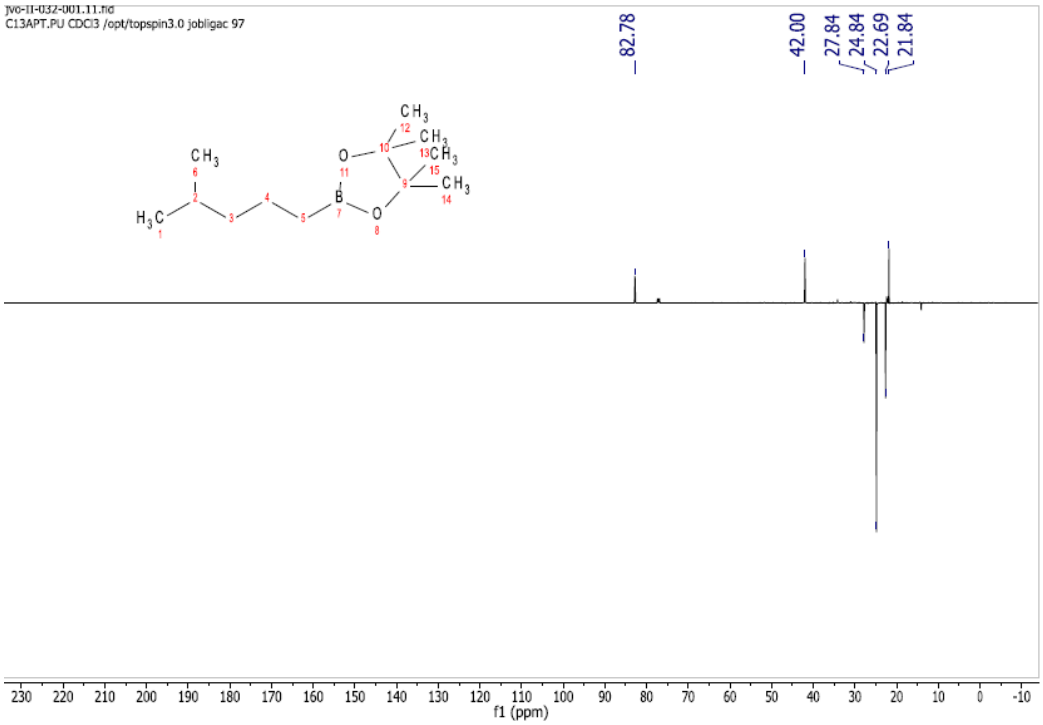


Figure S12. ^{13}C(APT) NMR spectrum of **8b** in $CDCl_3$ (126 MHz).

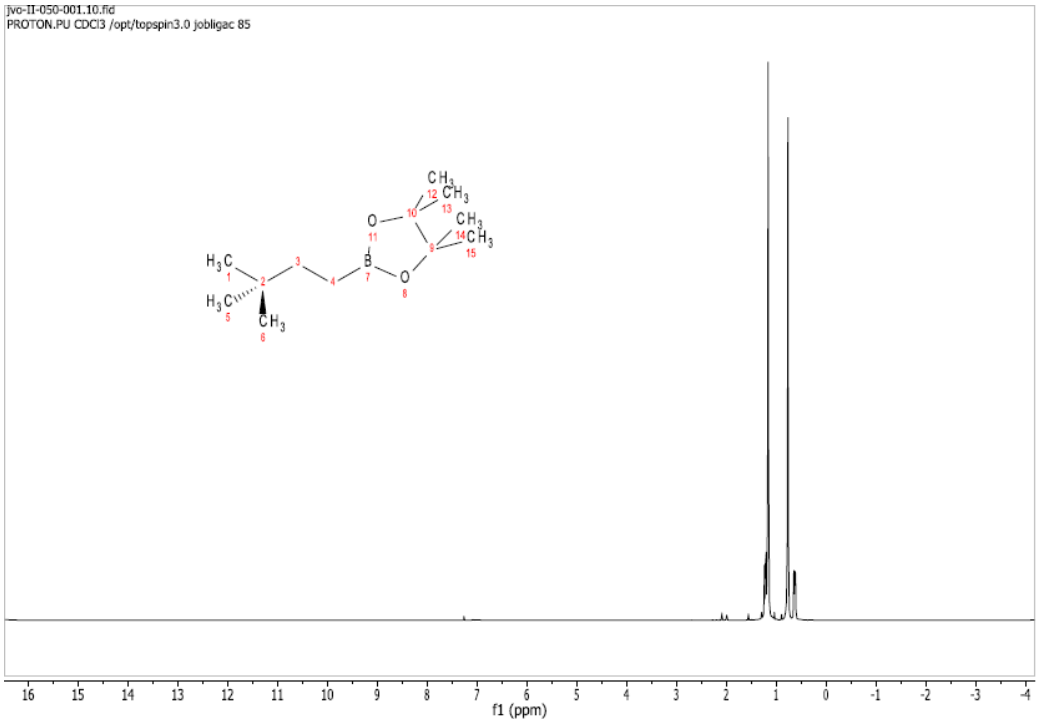


Figure S13. ^{1}H NMR spectrum of **8c** in $CDCl_3$ (500 MHz).

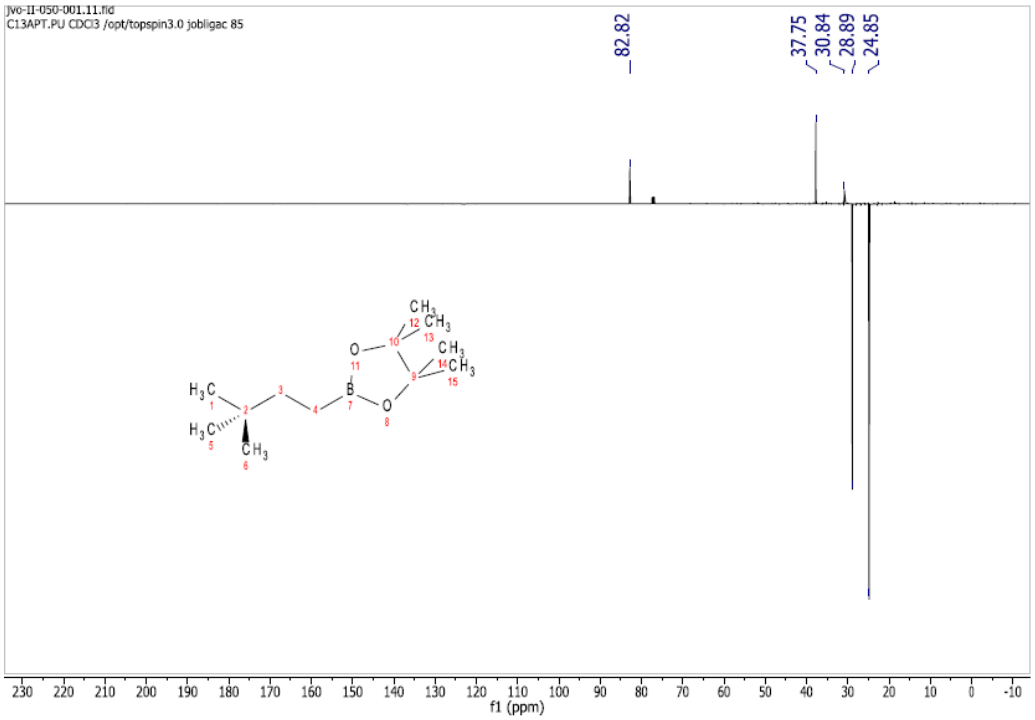


Figure S14. ^{13}C(APT) NMR spectrum of **8c** in $CDCl_3$ (126 MHz).

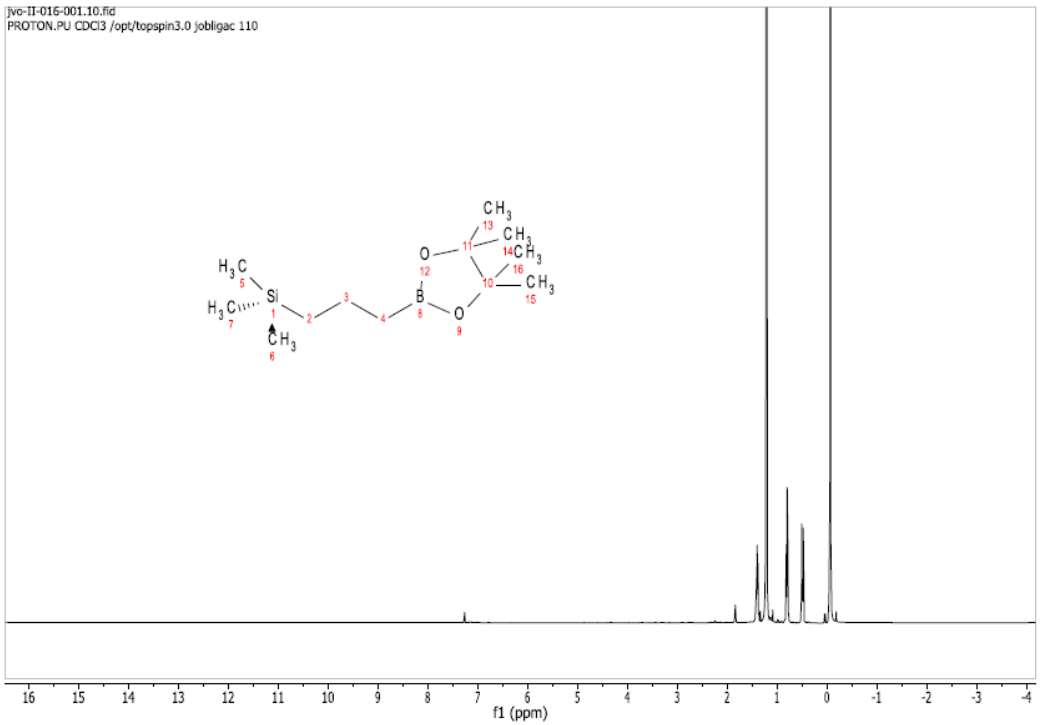


Figure S15. ^{1}H NMR spectrum of **8d** in $CDCl_3$ (500 MHz).

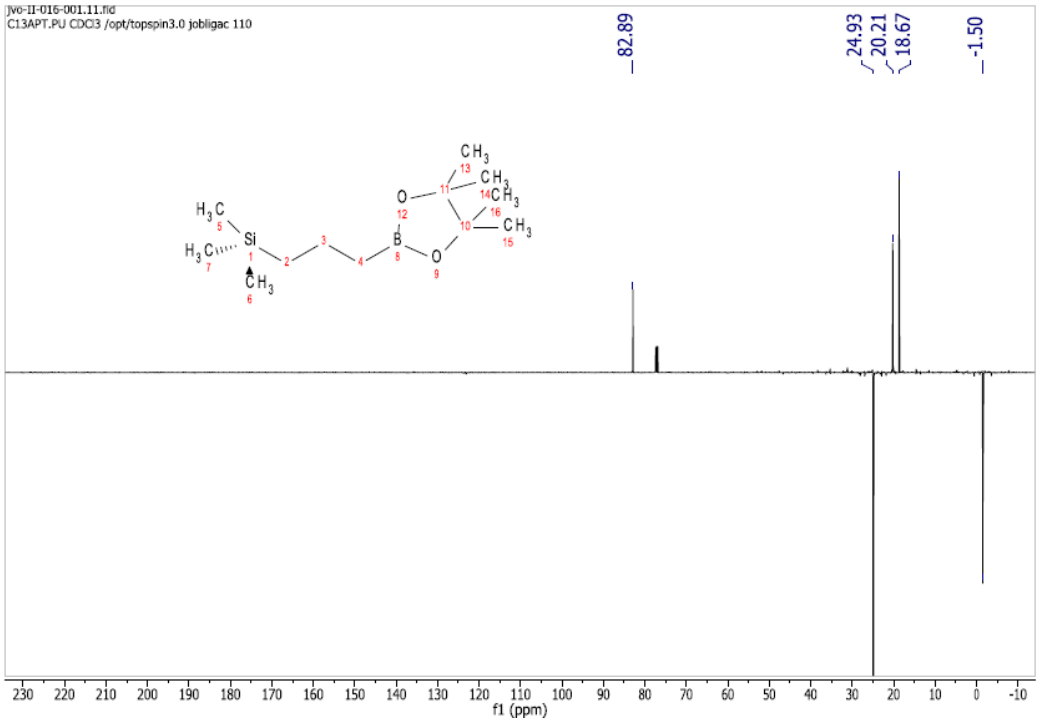


Figure S16. ^{13}C(APT) NMR spectrum of **8d** in $CDCl_3$ (126 MHz).

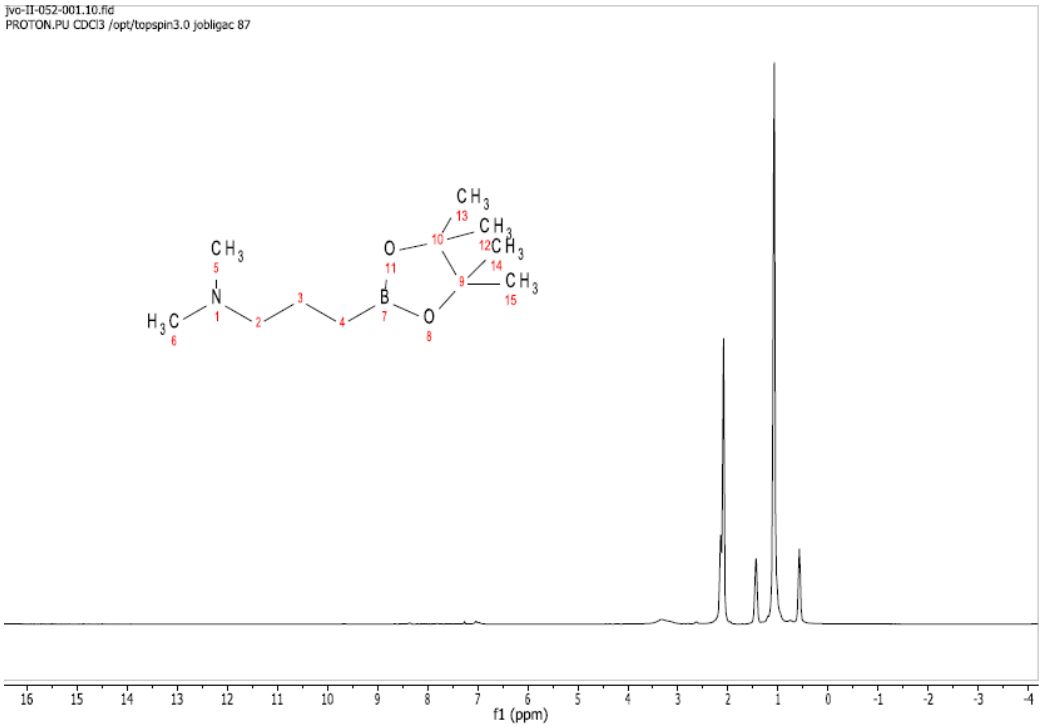


Figure S17. ^{1}H NMR spectrum of **8e** in $CDCl_3$ (500 MHz).

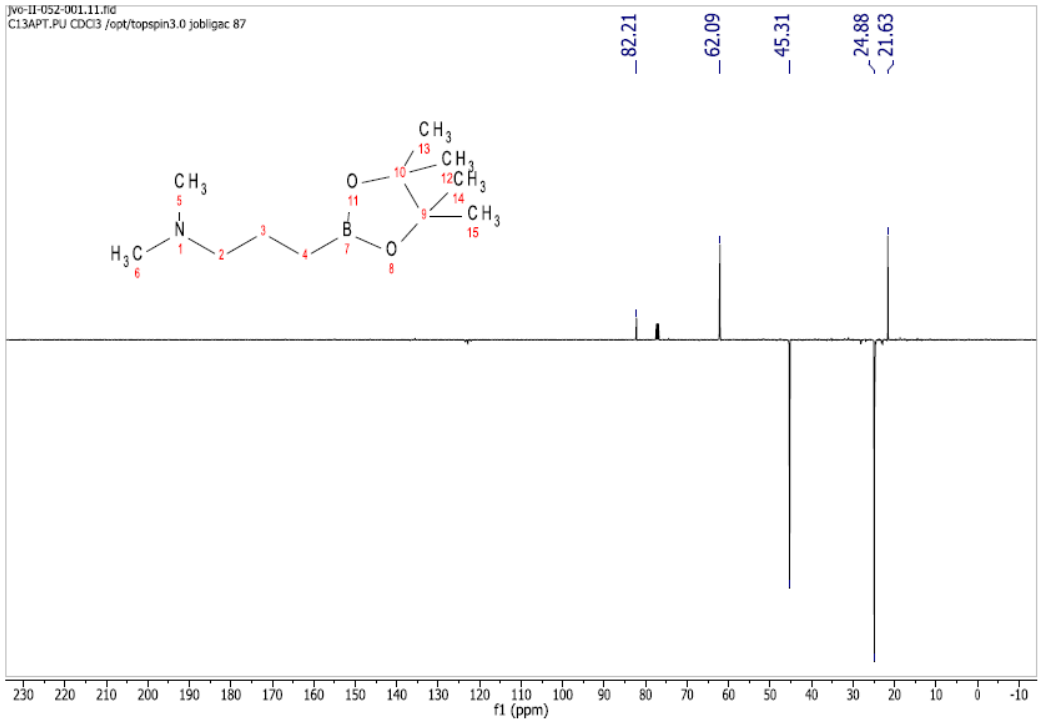


Figure S18. ^{13}C(APT) NMR spectrum of **8e** in $CDCl_3$ (126 MHz).

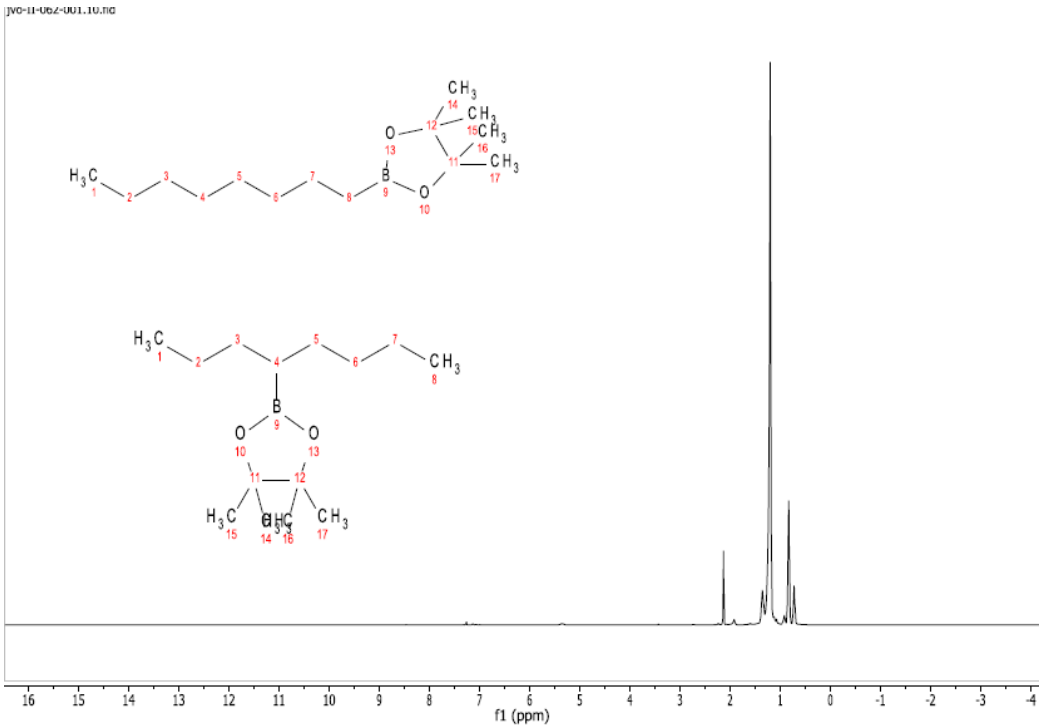


Figure S19. ^{1}H NMR spectrum of **8a** and **8f** in $CDCl_3$ (500 MHz).

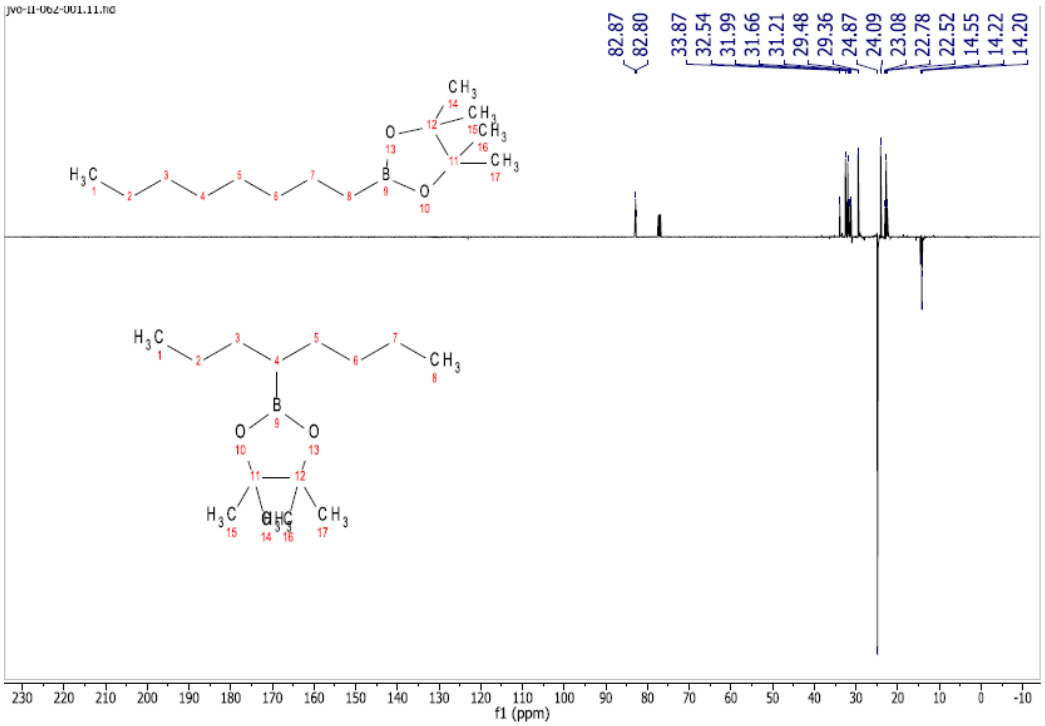


Figure S20. ^{13}C(APT) NMR spectrum of **8a** and **8f** in $CDCl_3$ (126 MHz).

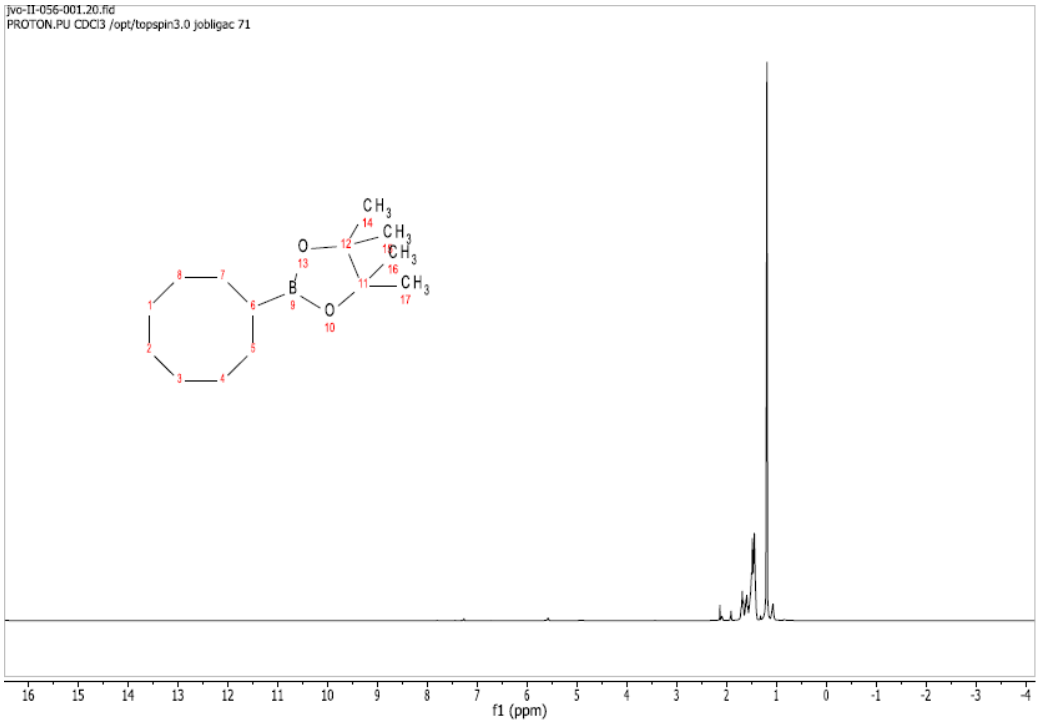


Figure S21. ^{1}H NMR spectrum of **8g** in $CDCl_3$ (500 MHz).

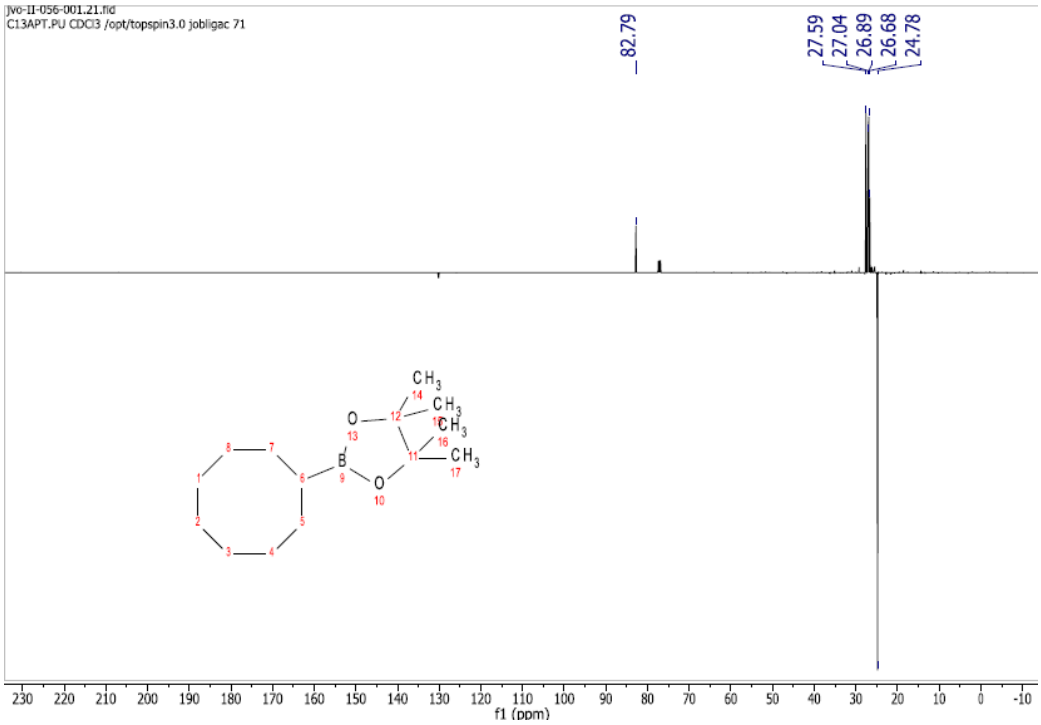


Figure S22. ^{13}C(APT) NMR spectrum of **8g** in $CDCl_3$ (126 MHz).

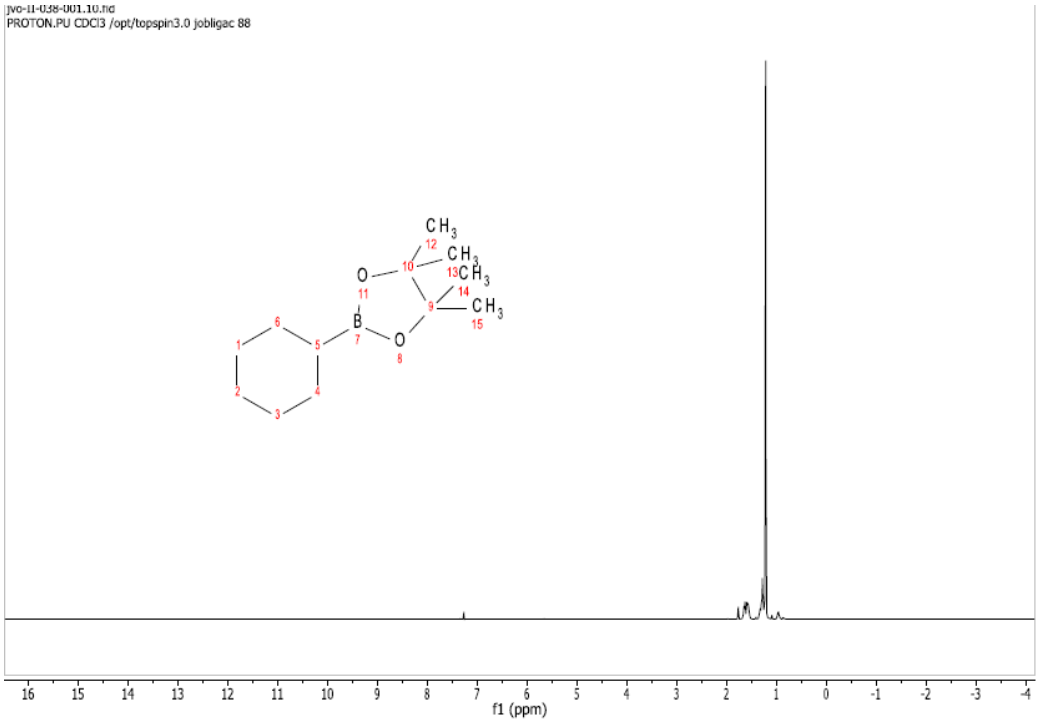


Figure S23. ^{1}H NMR spectrum of **8h** in $CDCl_3$ (500 MHz).

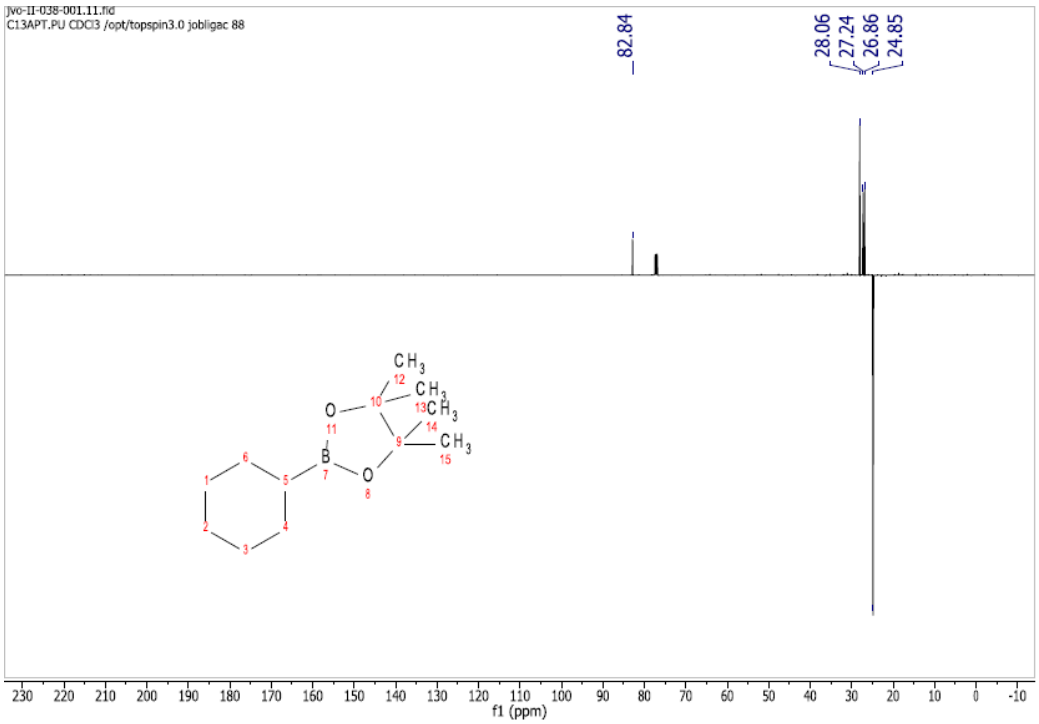


Figure S24. ^{13}C(APT) NMR spectrum of **8h** in $CDCl_3$ (126 MHz).

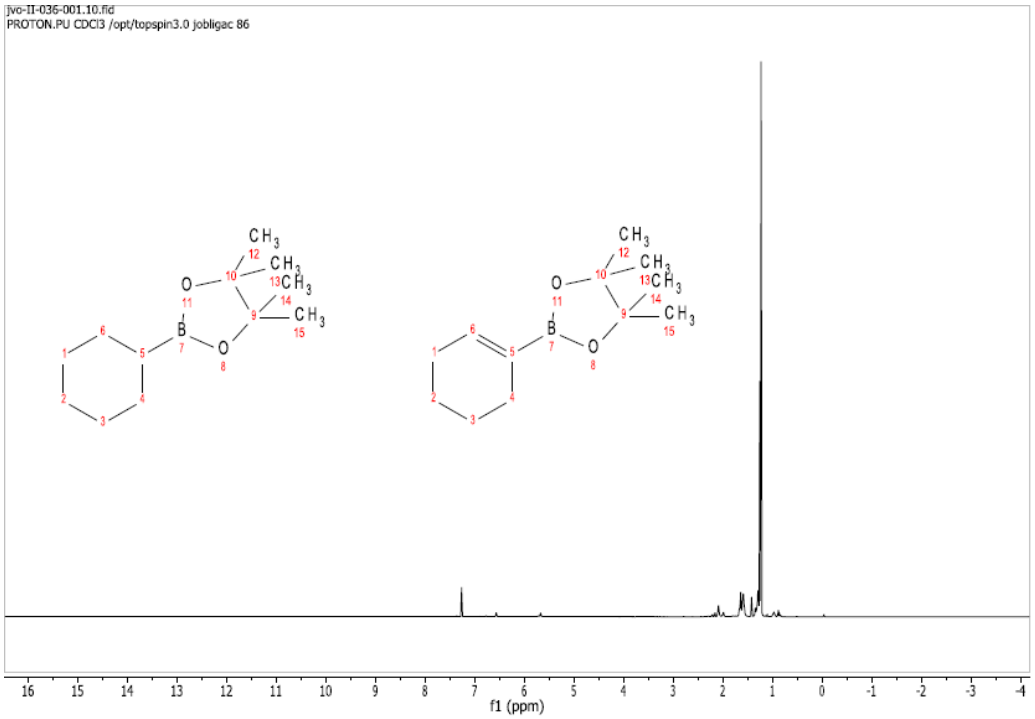


Figure S25. ^{1}H NMR spectrum of **8h** and **8i** in $CDCl_3$ (500 MHz).

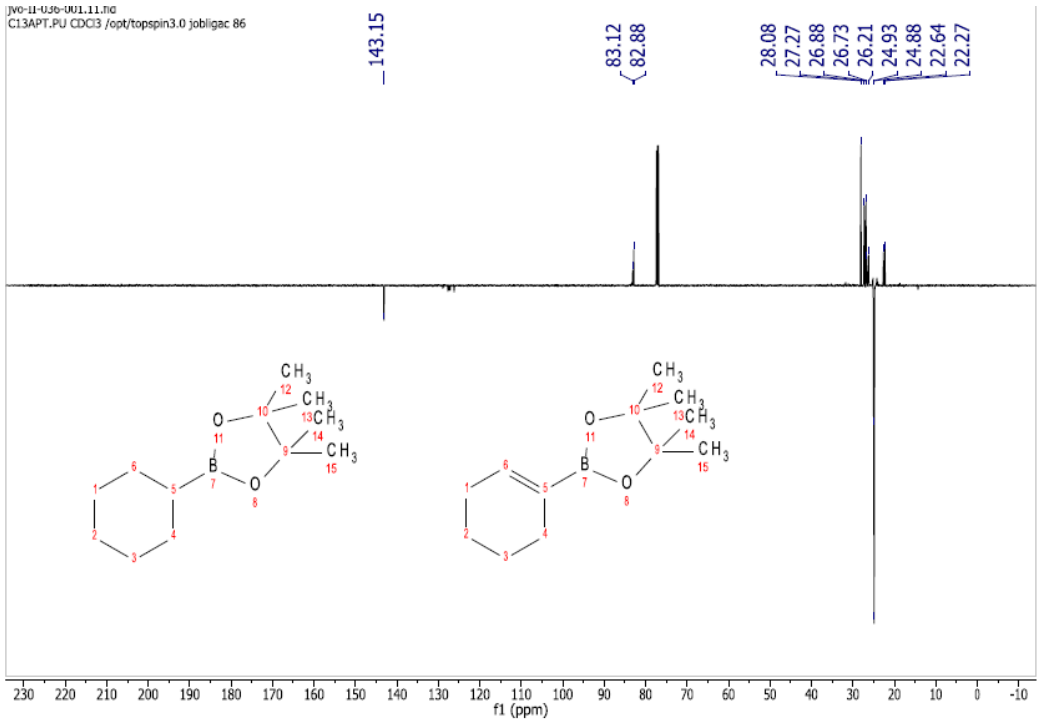


Figure S26. ^{13}C(APT) NMR spectrum of **8h** and **8i** in $CDCl_3$ (126 MHz).

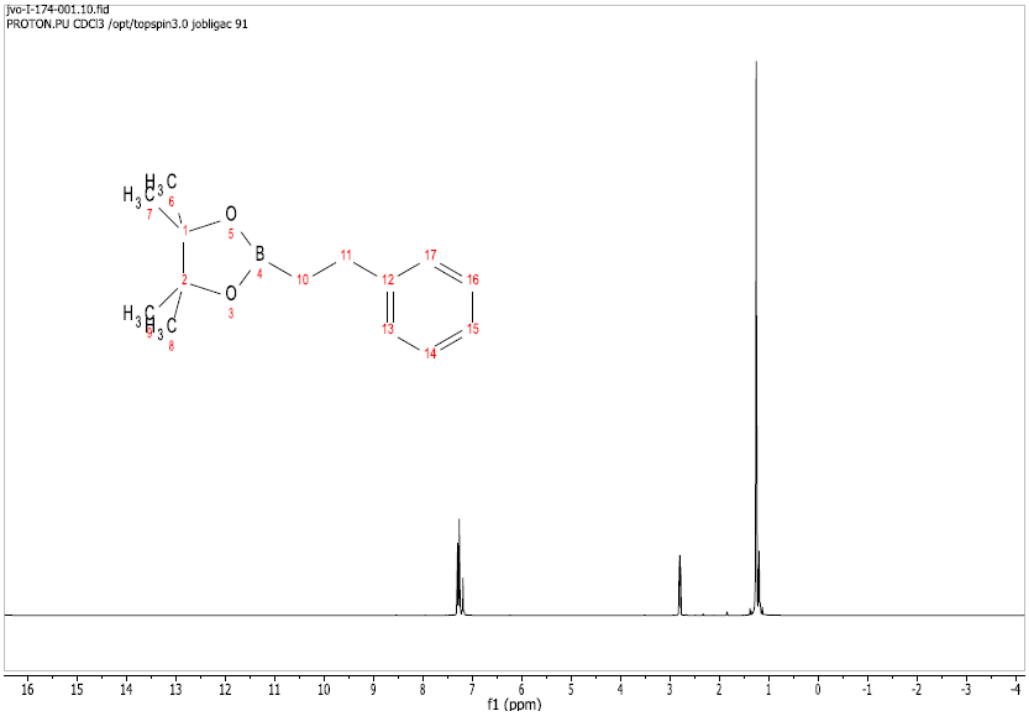


Figure S27. ^{1}H NMR spectrum of **8j** in $CDCl_3$ (500 MHz).

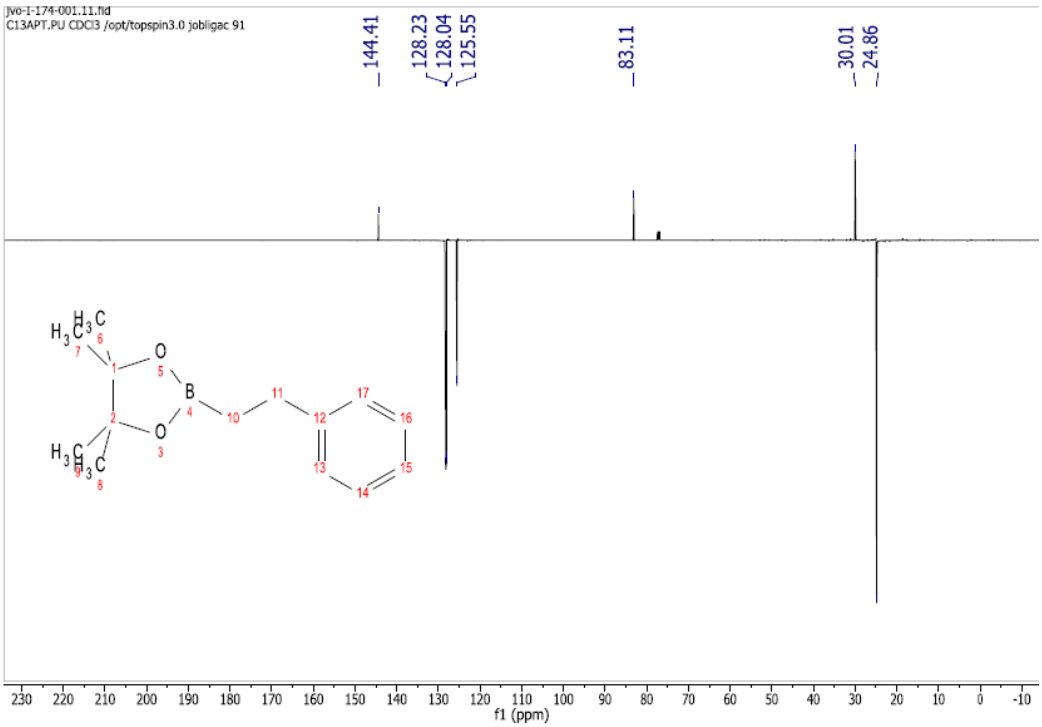


Figure S28. ^{13}C(APT) NMR spectrum of **8j** in $CDCl_3$ (126 MHz).

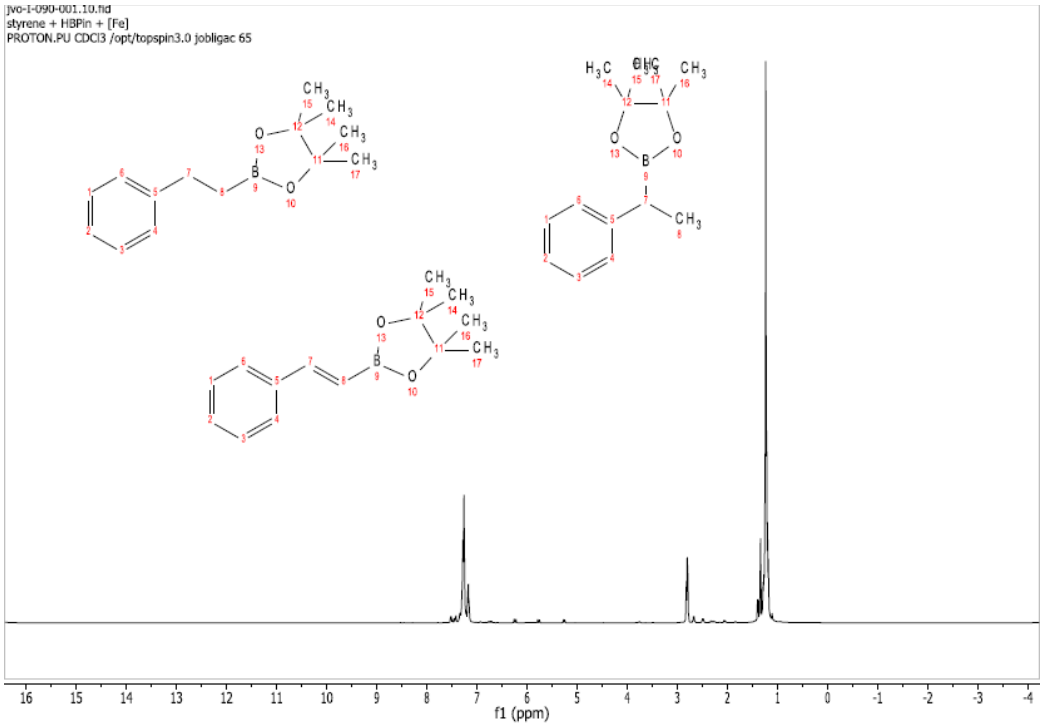


Figure S29. ^{1}H NMR spectrum of **8j**, **8k** and **8l** in $CDCl_3$ (500 MHz).

Figure S30. ^{13}C(APT) NMR spectrum of **8j**, **8k** and **8l** in $CDCl_3$ (126 MHz).

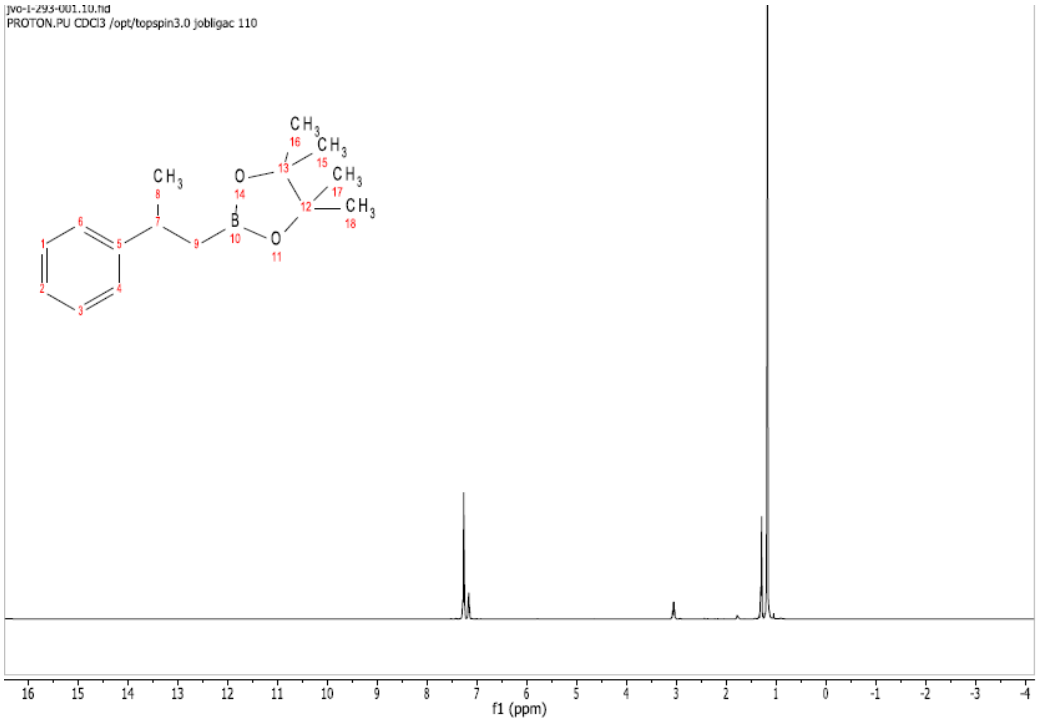


Figure S31. ^{1}H NMR spectrum of **8m** in $CDCl_3$ (500 MHz).

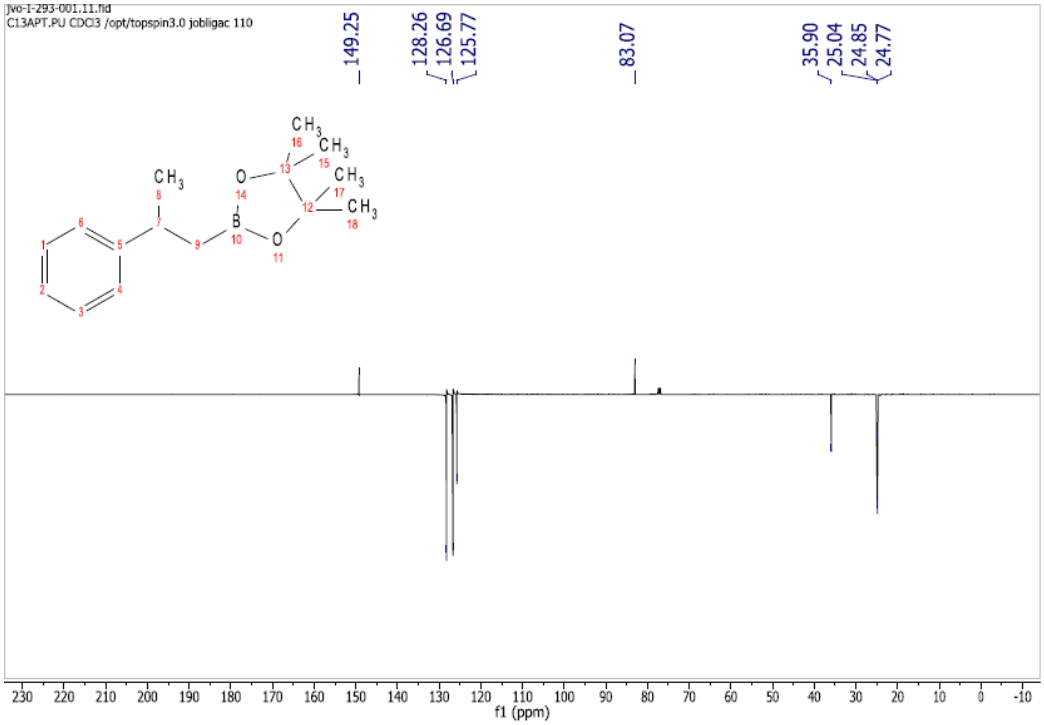


Figure S32. ^{13}C(APT) NMR spectrum of **8m** in $CDCl_3$ (126 MHz).

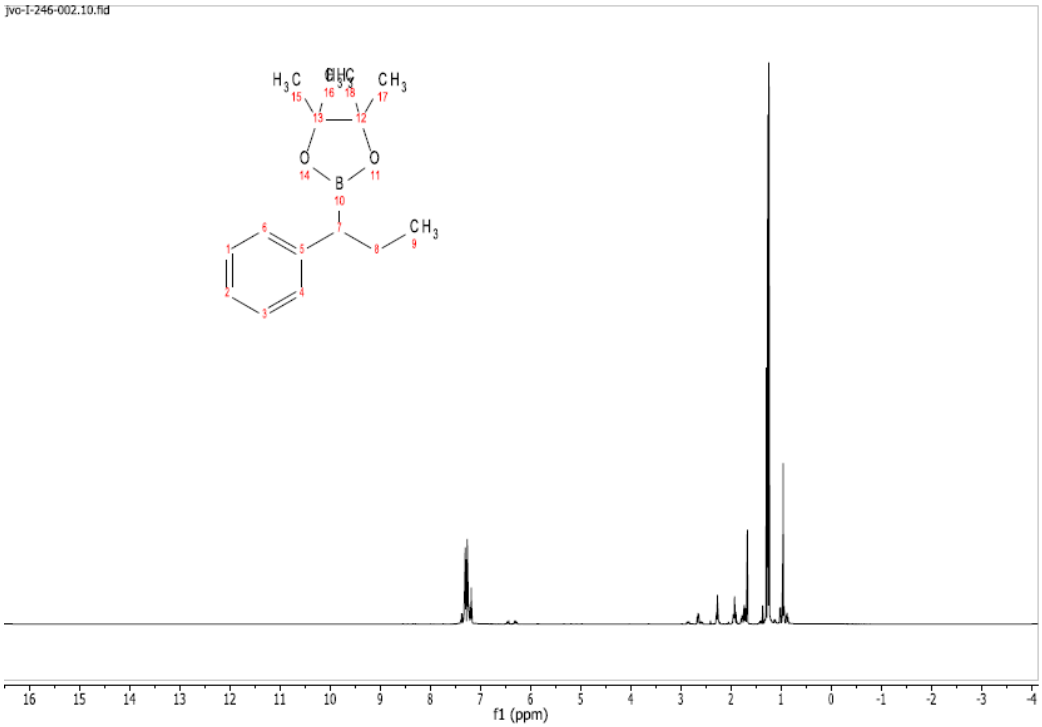


Figure S33. ^{1}H NMR spectrum of **8n** in $CDCl_3$ (500 MHz).

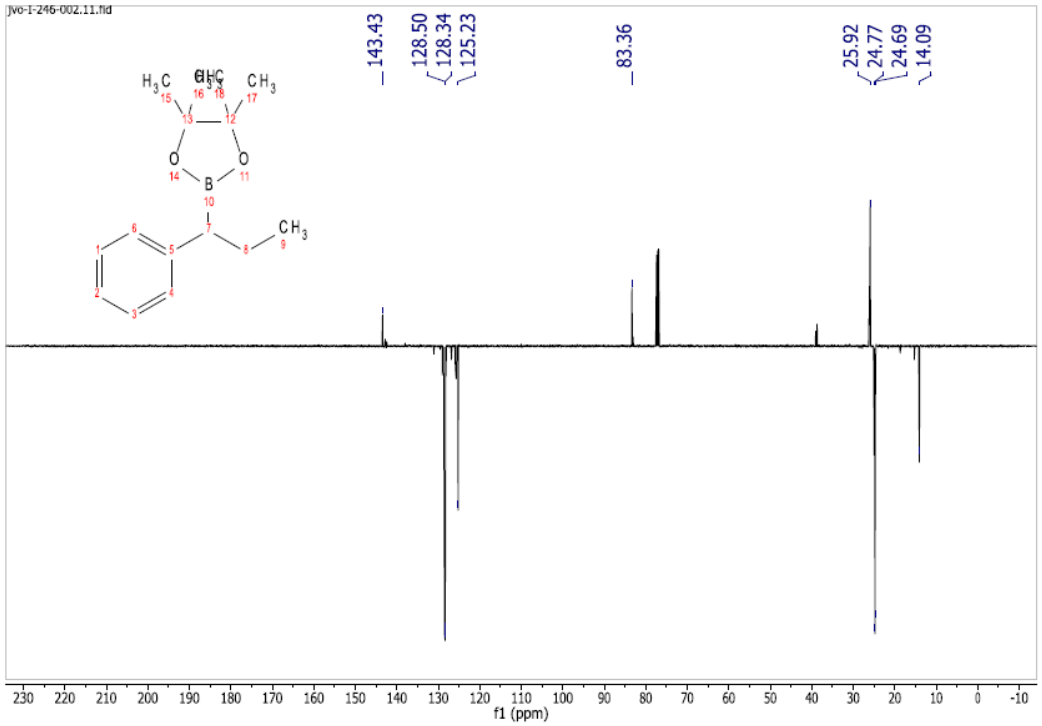


Figure S34. ^{13}C(APT) NMR spectrum of **8n** in $CDCl_3$ (126 MHz).

10.) References

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