Supporting Information for:

Protic Additives or Impurities Promote Imine Reduction with Pinacolborane

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General Considerations

Imines, pinacolborane, chiral additives, and solvents were dispensed in a 2001 issue IT Glovebox (H_2O levels typically 20-70 ppm). Reduction reactions were carried out in 4 dram oven dried scintillation vials equipped with magnetic stir bars and green Qorpak® PTFE lined caps. Substrates, reagents and solvents were loaded into vials inside the IT Glovebox. Reactions at ambient temperature were stirred within the glovebox. ^{1}H and ^{11}B NMR data were collected at 300K on a Bruker AV-500 NMR spectrometer. Standard NMR tubes and caps were used. ^{1}H NMR spectra are referenced to residual non-deuterated NMR solvent ($CHCl_3$ =7.26 ppm, CH_3CN = 1.94 ppm). HPLC data were acquired on a Varian Prostar instrument, equipped with detection at 254 nm, using a Astec Cellulose DMP column. A 99:1 hexanes/isopropanol solvent mixture was used as the eluent, with a flow rate of 0.5 mL/min. Configuration of the reduction product was determined by comparison with a known sample. Conversion ratios were measured by integration of the benzylic signals of the starting material and product, unless a signal was obscured, in which case the methyl group was instead integrated. A 0.10 M solution of ferrocene in $CDCl_3$ was also employed as an internal standard. 0.50 mL of this solution was dispensed to deliver 0.050 mmol of ferrocene to reactions. The ferrocene signal was integrated as 10, and molar quantities of substrates/products were accordingly calculated by multiplying obtained integral value by amount of ferrocene (0.050 mmol) divided by number of protons the signal represented (since ferrocene was normalized to 10).

Solvents

Acetonitrile was purchased from VWR in a 1L EMD Drisolv® bottle. This bottle was taken into the glovebox, and activated 3 Å molecular sieves were added. A 0.25 M solution of water in acetonitrile was prepared by adding water to dry acetonitrile outside of the glovebox.

Tetrahydrofuran was purchased from Fisher and was distilled from a purple solution of benzophenone/sodium ketyl, and stored in the glovebox over activated 3 Å molecular sieves were added. A 0.25 M solution of water in THF was prepared by adding water to dry THF outside of the glovebox.

Deuterochloroform(Cambridge Isotopes) was stored over activated 3 Å molecular sieves, but otherwise used as received.

Reagents

Chiral Additives were purchased from Oakwood Chemical (binol and sulfinimide) or Aldrich.

Imines were prepared according to literature procedures. Imine 10 was prepared by Mr. Toren Hynes.[1]

[1] A) M. R. Adams, C. H. Tien, B. S. N. Huchenski, M. J. Ferguson, A. W. H. Speed, *Angew. Chem., Int. Ed.,* 2017, **56**, 6268. B) M. R. Adams, C. H. Tien, R. McDonald, A. W. H. Speed, *Angew. Chem., Int. Ed.,* **56**, 16660. C) L. S. Bleicher, N. D. P. Cosford, A. Herbaut, J. S. McCallum, I. A. McDonald, *J. Org. Chem.* 1998, **63**, 1109. D) K. Vukics, T. Fodor, J. Fischer, I. Fellegvári, S. Lévai, *Org. Process Res. Dev.* 2002, **6**, 82.

Isopropyl alcohol and methanol were obtained from Dalhousie University chemical stockroom (Fisher Drums, absolute grade), and used as received.

Pinacolborane was purchased from Oakwood Chemical, stored at ambient temperature in the glovebox, and used as received.

Pinacolborane-*d* was prepared by Mr. Casper Macaulay from B_2Pin_2, using the procedure of Hartwig and Hall.[2]

***Tert*-Butyl alcohol** was purchased from Sigma and used as recieved.

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General Reduction Procedures:

All product amines are known compounds, and match previously reported spectra data.[1]

Control experiment

Compound **1** (50 mg, 0.186 mmol) was weighed into a 4-dram vial and equipped with a magnetic stir bar. Pinacol borane (0.02 mL, 0.278 mmol, 1.5 equiv.) was added to the vial and the mixture was left to stir for 3h. Deuterated chloroform (1 mL) was then added and the solution left to stir for 10 min. NMR spectra were acquired after a further 15 min and 1.5 h after chloroform-*d* addition with no observable reduction.

Deuterated methanol studies

Compound **1** (50 mg, 0.186 mmol) was weighed into a 4-dram vial. Pinacol borane was added to the vial followed by deuterated chloroform containing methanol-d_4 in the amounts detailed in the table below. The vial was then shaken and transferred to an NMR tube and NMR spectrum were acquired.

Entry	Pinacol Borane	Methanol-d_4	Chloroform-*d*	Starting material: product
1	0.04 mL (1.5 equiv.)	0.02 mL (2.6 equiv.)	1 mL	1:6.6
2	0.04 mL (1.5 equiv.)	0.04 mL (5.3 equiv.)	1 mL	1:10.9
3	0.04 mL (1.5 equiv.)	0.08 mL (10.6 equiv.)	1 mL	1:22.1
4	0.04 mL (1.5 equiv.)	0.04 mL (5.3 equiv.)	1 mL	1:12.6

Primary, secondary, and tertiary alcohol study

[2] C. S. Wei, C. A. Jiménez-Hoyos, M. F. Videa, J. F. Hartwig, M. B. Hall, *J. Am. Chem. Soc.* 2010, **132**, 3078.

Compound **1** (50 mg, 0.186 mmol) was weighed into a 4-dram vial. Pinacol borane (0.04 mL, 0.278 mmol, 1.5 equiv.) was added to the vial followed by a solution of the respective alcohol in deuterated chloroform (0.93 mL, 10 equiv., 2M in deuterated chloroform). The vial was then shaken and transferred to an NMR tube and NMR spectrum were acquired.

Entry	Alcohol	Starting material: product
1	Methanol	1:1.4
2	Isopropyl alcohol	1:6.4
3	*Tert*-butyl alcohol	1:12.6

Tert-butyl alcohol optimization study

Compound **1** (50 mg, 0.186 mmol) was weighed into a 4-dram vial. Pinacol borane was added to the vial and then dissolved in deuterated chloroform. Tert-butanol was added in amounts detailed in the table below. The vial was then shaken and transferred to an NMR tube and NMR spectrum were acquired.

Entry	Pinacol Borane	*tert*-butyl alcohol	Chloroform-*d*	Starting material: product
1	0.04 mL (1.5 equiv.)	26 µL (3 equiv.)	1 mL	1:3.1
2	0.04 mL (1.5 equiv.)	26 µL (3 equiv.)	0.5 mL	1:3.8
3	0.04 mL (1.5 equiv.)	53 µL (6 equiv.)	0.5 mL	1:6.6
4	0.05 mL (1.8 equiv.)	53 µL (6 equiv.)	0.5 mL	1:7.8
5	0.06 mL (2.2 equiv.)	53 µL (6 equiv.)	0.5 mL	1:12.6

General procedure for imine reduction:

A substrate (0.186 mmol) was weighed into a 4-dram vial. Pinacol borane (0.06 mL, 0.408 mmol, 2.2 equiv.) was added to the vial and then dissolved in deuterated chloroform (0.5 mL). *Tert*-butyl alcohol (53 µL, 0.556 mmol, 3 equiv.) was then added to the solution. The vial was then shaken and transferred to an NMR tube and NMR spectrum were acquired. Results of starting material to product integrations are detailed in the table below.

Compound #	Starting material: product
1	1:13.8
5	1: trace
6	1:0
7	1:0
8	1:0
9	1:0
10	1:0
11	1:13.1
12	1:3.7

13	1:4.2
14	1:8.5
15	1:8.6
16	1:3.7

Procedure for reduction with an internal standard:

An imine (0.1856 mmol) was weighed into a 1-dram vial. HBpin (0.06 mL, 0.4135 mmol, approx. 2.2 eq) was then added to the vial followed by ferrocene (0.50 mL of a 0.10M solution in $CDCl_3$). The contents were then shaken in the vial and then *tert*-butyl alcohol (53 µL, 0.5568 mmol, 3 eq) was added and the mixture was shaken for 5s. The solution was transferred to an NMR tube and NMR spectra were acquired after 10 min. Calculations and yields with internal standards are shown on the NMR spectra (page 16).

Chiral alcohol studies

Compound **1** (50 mg, 0.186 mmol) was weighed into a 4-dram vial. Pinacol borane (0.04 mL, 0.278 mmol, 1.5 equiv.) was added to the vial. A chiral alcohol (0.278 mmol, 1.5 equiv.) dissolved in deuterated chloroform (0.75 mL) was then added to the vial and shaken. Binol was added as a solid after chloroform addition due to incomplete solubility in the chloroform. The vial was then shaken and the contents were transferred to an NMR tube and NMR spectrum were acquired. Samples were subsequently acidified with 2N HCl, the organic layer was discarded, and the aqueous layer was made basic, then extracted with dichloromethane, dried over sodium sulfate, concentrated, and analyzed by HPLC.

Alcohol	Starting material: product	e.r. (% ee)
(1(S)-endo(-))-Borneol	1:8.4	racemic
(R)-(+)-1,1'-Bi-2-napthol	/	63.5:36.5 (27 %)
(1S,2R,5S)-(+)-Menthol	1:7.7	racemic
Cedrenol	1:5.9	racemic
(S)-(-)-*tert*-Butylsulfinamide	1:4.8	racemic

Deuteration study:

4-methoxyacetophenone-N-(4-methoxybenzyl)imine (50 mg, 0.186 mmol) was weighed into a 1-dram vial. DBpin (0.06 mL, 0.4135 mmol, approx. 2.2 eq) was then added to the vial followed by ferrocene (0.5mL, 0.1M in $CDCl_3$). The contents were then shaken in the vial and then *tert*-butyl alcohol (53 µL, 0.5568 mmol, 3 eq) was added and the mixture was shaken for 5s. The solution was transferred to an NMR tube and an NMR spectrum was acquired after 10 min. The solvent was then removed in vacuo. Ether (2 mL) was then added followed by HCl (1 mL, 2M in Et_2O). Solvent was removed in vacuo and the residue was washed with pentane (3 x 5mL) by decanting the pentane. NaOH (10 mL, 2M) was then added as well as DCM (10 mL) which was then

transferred to a separatory funnel. The aqueous layer was washed with DCM (3 x 7 mL) and the combined organic layers were dried with anh. sodium sulfate. Solvent was removed in vacuo and the resulting residue was purified by column chromatography with a gradient of 9:1 (hexanes : ether) to ether, resulting in the pure amine (38mg, 0.14 mmol, 75% yield).

^{1}H (500MHz, $CDCl_3$): δ 7.26 (m, 2H), 7.18 (m, 2H), 6.88 (m, 2H), 8.84 (m, 2H), 3.80 (s, 3H), 3.78 (s, 3H), 3.55 (ab q, 2H), 1.50 (s, 1H), 1.32 (s, 3H).

^{13}C{^{1}H} (500MHz, $CDCl_3$): δ 158.7, 137.8, 133.0, 129.4, 127.8, 113.9, 113.8, 56.3 (t), 55.4, 51.0, 24.5.

HRMS (APCI): [$M+H^+$ for $C_{17}H_{21}DNO_2$] calculated: 273.1708, found: 273.1715

200 mg scale reaction and product isolation:

4-methoxyacetophenone-N-(4-methoxybenzyl)imine (200 mg, 0.7426 mmol) was weighed into a 4-dram vial equipped with a magnetic stir bar. HBpin (0.24 mL, 1.633 mmol, approx. 2.2 eq) was then added to the vial followed by DCM (2 mL). The contents were then stirred in the vial and then *tert*-butyl alcohol (0.21 mL, 2.227 mmol, 3 eq) was added and the mixture was stirred for 30 min. An aliquot was taken, and an NMR spectrum was acquired. Solvent was then removed in vacuo and the residue was dissolved in ether (15 mL) and the amine was extracted with HCl (2 x 15 mL, 2M). The aqueous layer was then basified with NaOH (2M) and the product extracted with ether (3 x 20 mL). The organic extracts were then dried with anhydrous sodium sulfate and the filtered. Solvent was removed in vacuo and the resulting residue was purified by column chromatography with a gradient of 9:1 (hexanes : ether) to ether, resulting in the amine which was then purified again with the same acid base wash mentioned above to remove a small amount of remaining pinacol, which afforded the amine (153mg, 0.565 mmol, 77% yield).

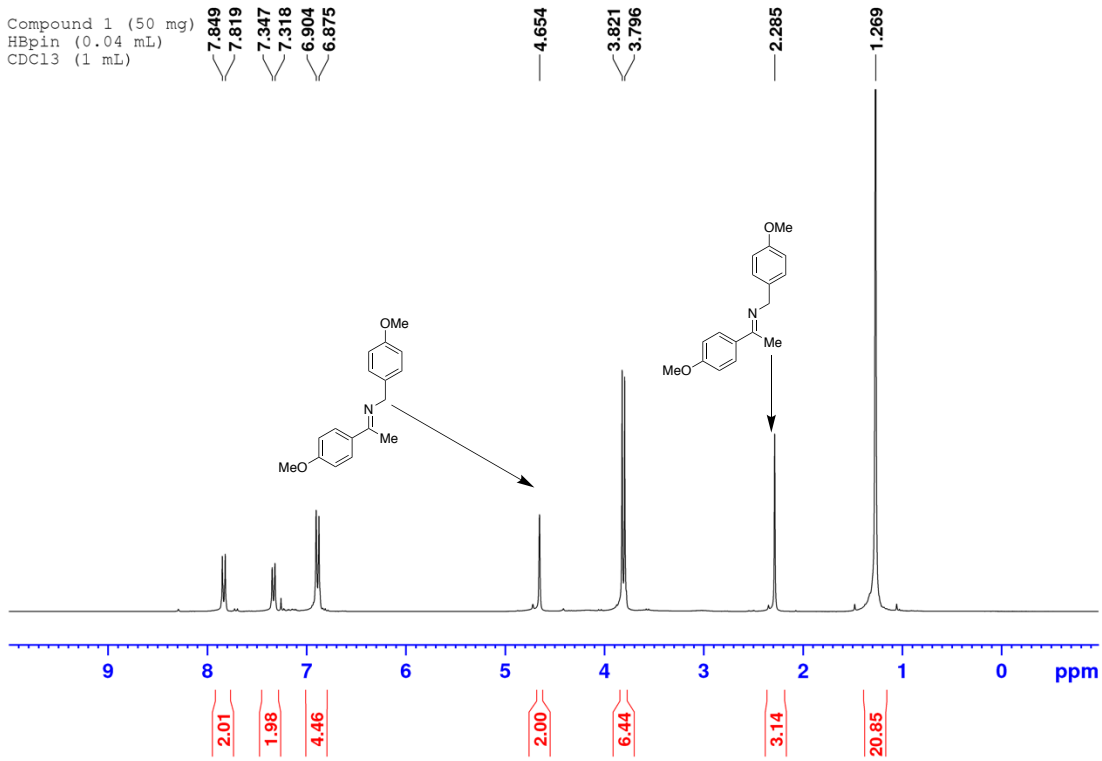
^{1}H (500MHz, $CDCl_3$): δ 7.26 (m, 2H), 7.18 (m, 2H), 6.88 (m, 2H), 8.84 (m, 2H), 3.80 (s, 3H), 3.79 (s, 3H), 3.75(q, J = 6.6 Hz, 1H), 3.55 (ab q, 2H), 1.50 (s, 1H), 1.33 (d, J = 6.6 Hz, 3H).

Procedure for autocatalytic reactions:

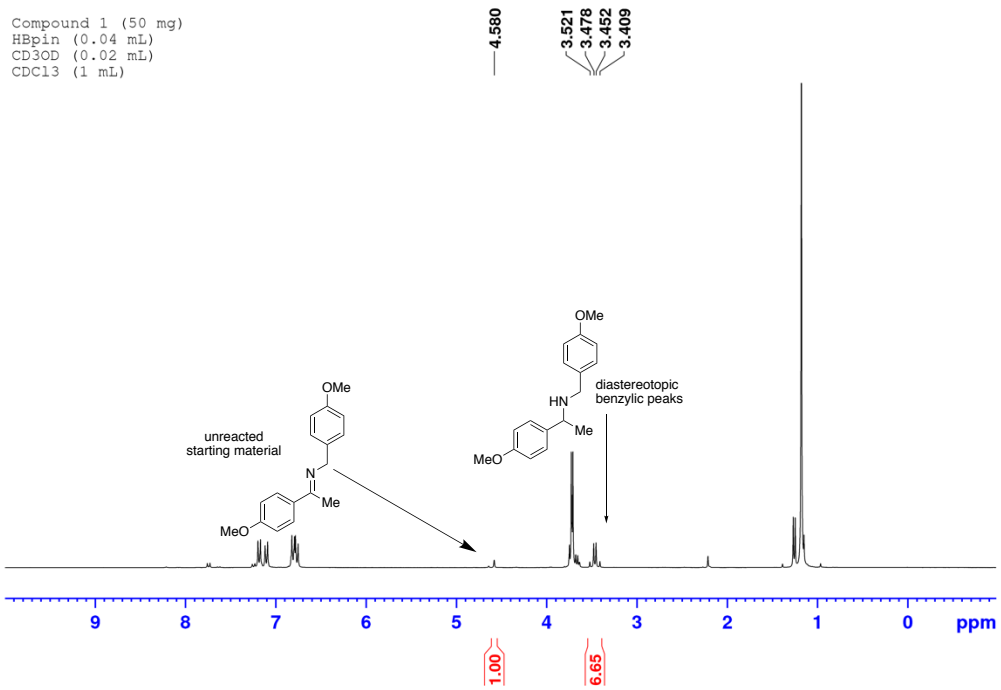
4-methoxyacetophenone-N-(4-methoxybenzyl)imine (50 mg, 0.1856 mmol) was weighed into a 1-dram vial. HBpin (0.06 mL, 0.4135 mmol, approx. 2.2 eq) was then added to the vial followed by ferrocene (0.5mL, 0.1M in $CDCl_3$). The contents were then shaken in the vial and then 4-methoxyacetophenone-N-(4-methoxybenzyl) amine (10 mg, 0.03712 mmol, 0.2 eq) was added as a solution in $CDCl_3$ (0.25 mL) and the mixture was shaken for 5s. The solution was transferred to an NMR tube after 10 min and NMR spectrum were acquired at 30 minutes and then at 24 hours.

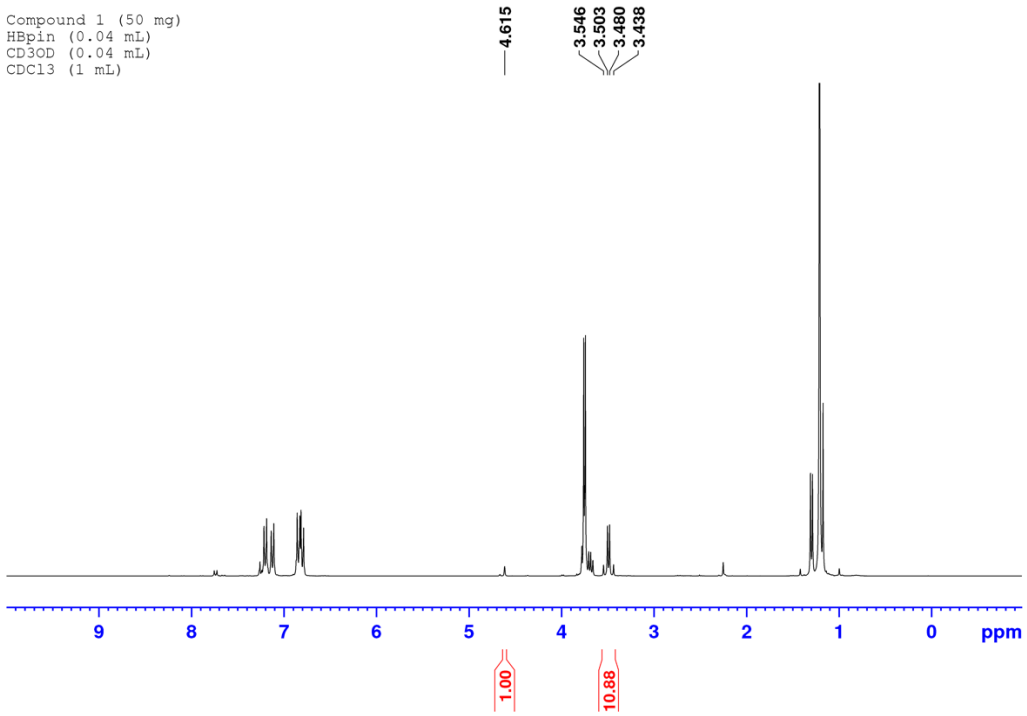
NMR spectra

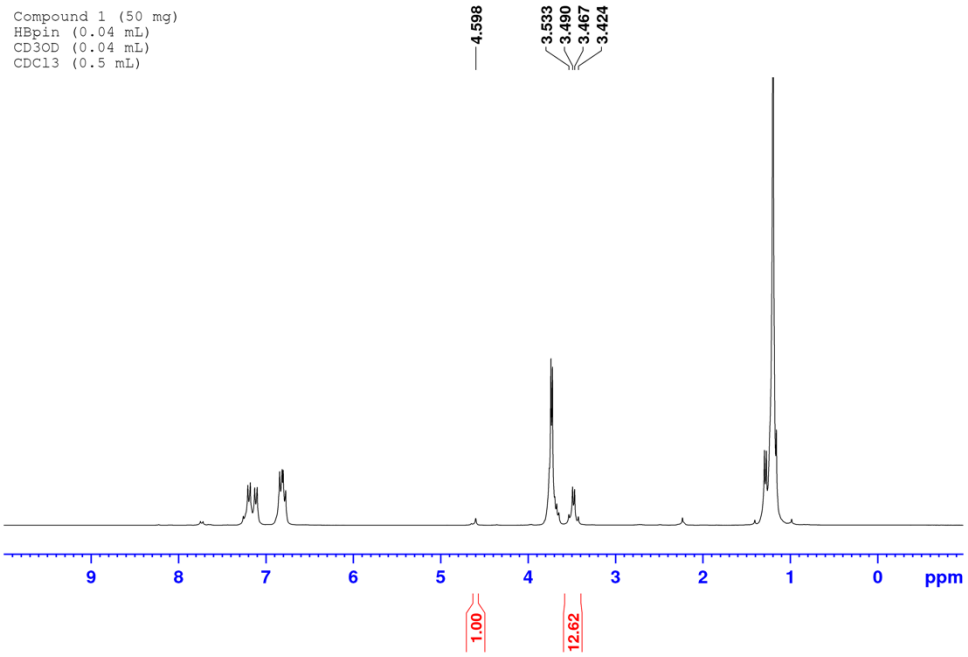
Control reaction. Mixture of imine **1** and HB(pin) in $CDCl_3$.

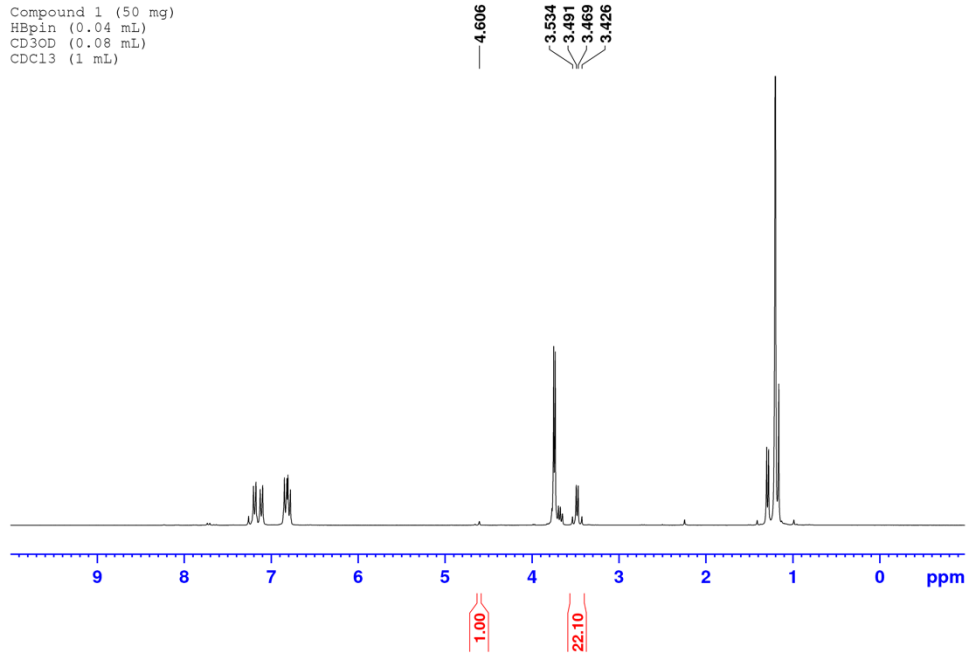


Studies with deuterated methanol addition



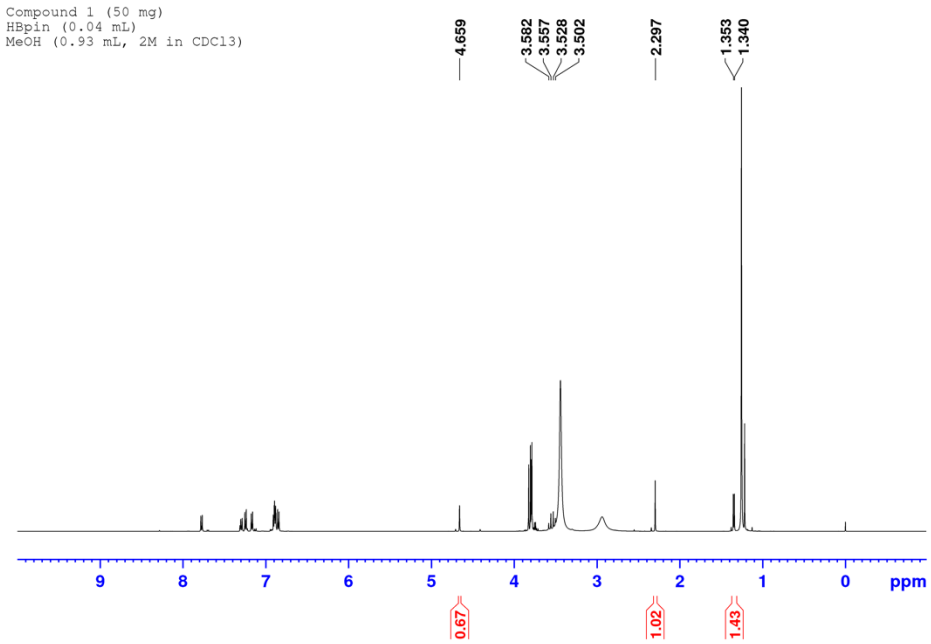



Compound 1 (50 mg)
HBpin (0.04 mL)
CD3OD (0.04 mL)
CDCl3 (0.5 mL)
4.598
3.533
3.490
3.467
3.424
9
8
7
6
5
4
3
2
1
0
ppm
1.00
12.62

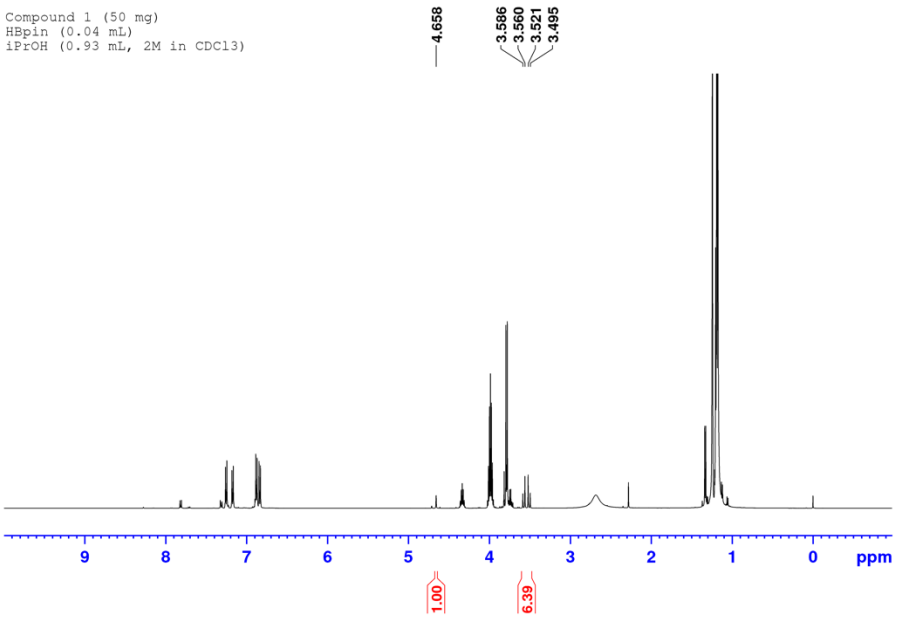

Compound 1 (50 mg)
HBpin (0.04 mL)
CD3OD (0.08 mL)
CDCl3 (1 mL)
4.606
3.534
3.491
3.469
3.426
9
8
7
6
5
4
3
2
1
0
ppm
1.00
22.10

Spectra from addition of methanol, isopropyl alcohol, and *tert*-butyl alcohol

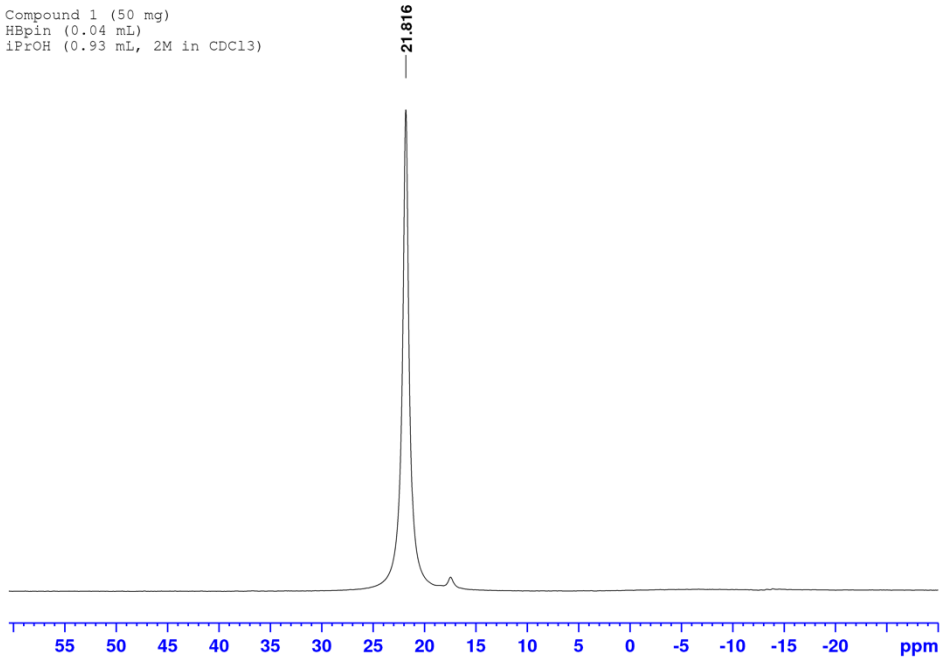
Methanol addition:



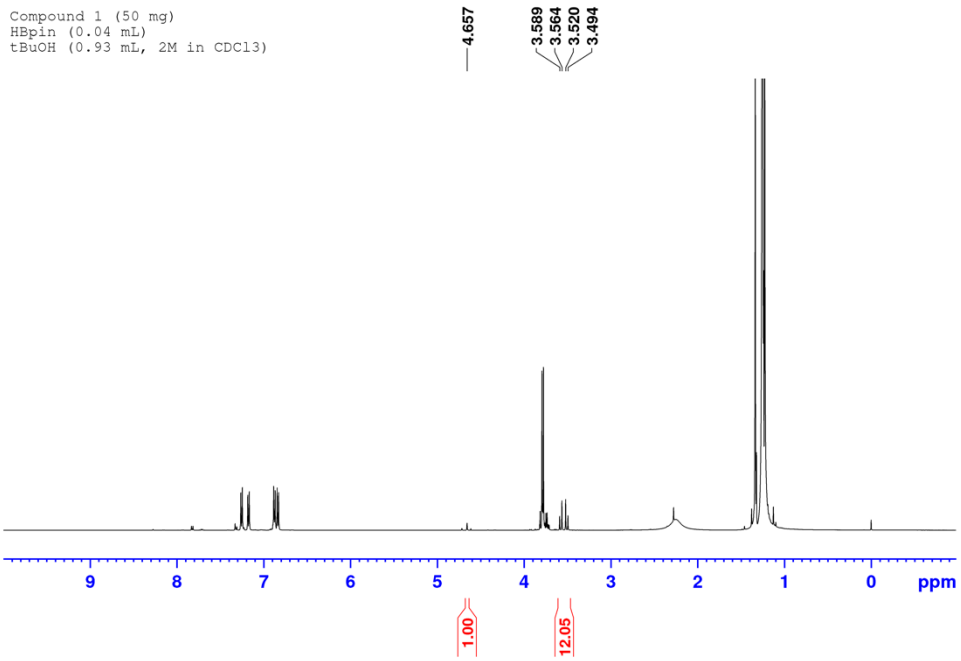
isopropyl alcohol addition:



^{11}B NMR Spectrum of isopropyl alcohol addition

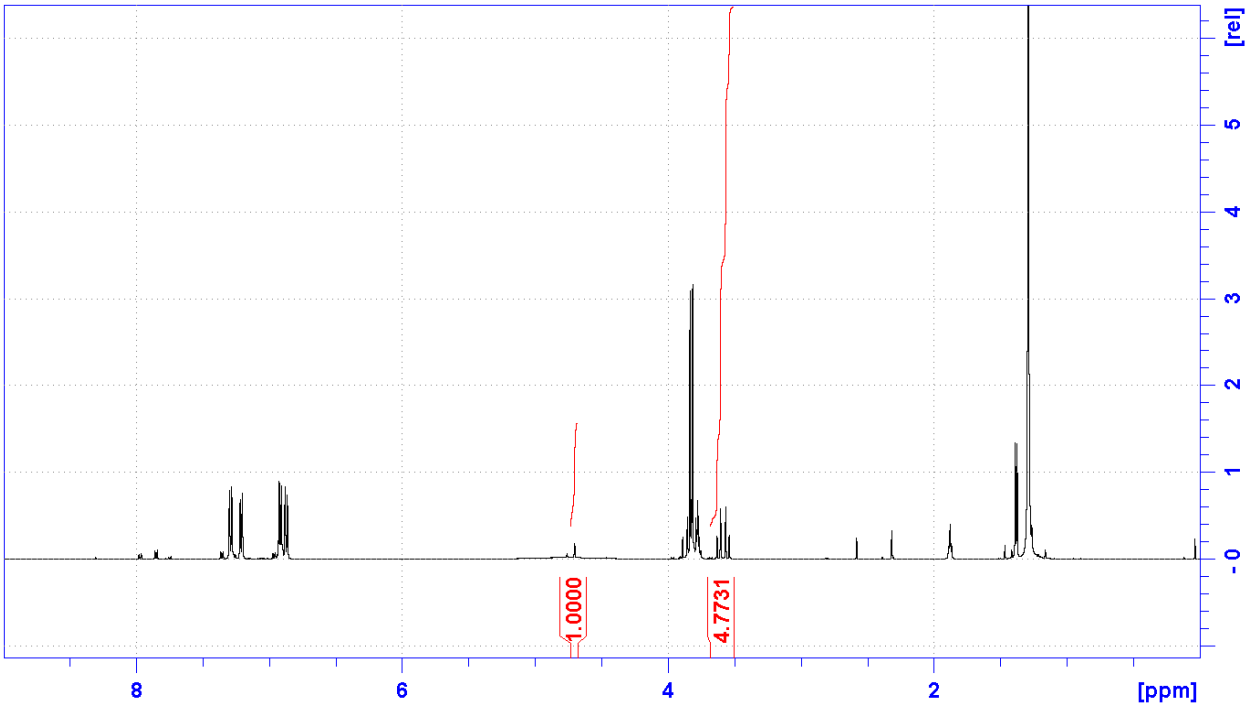


tert-butyl alcohol addition:

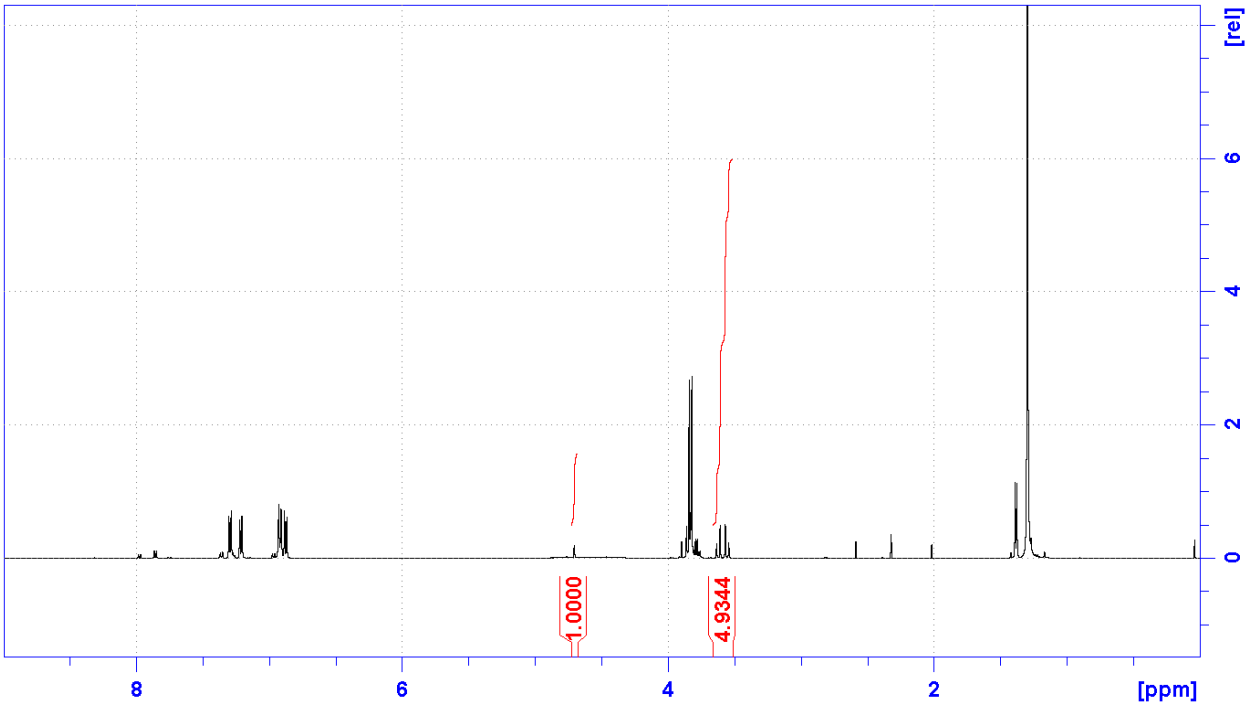


Spectra from addition of 0.25 M solutions of water in either THF or Acetonitrile to mixtures of imine 1 and pinacolborane

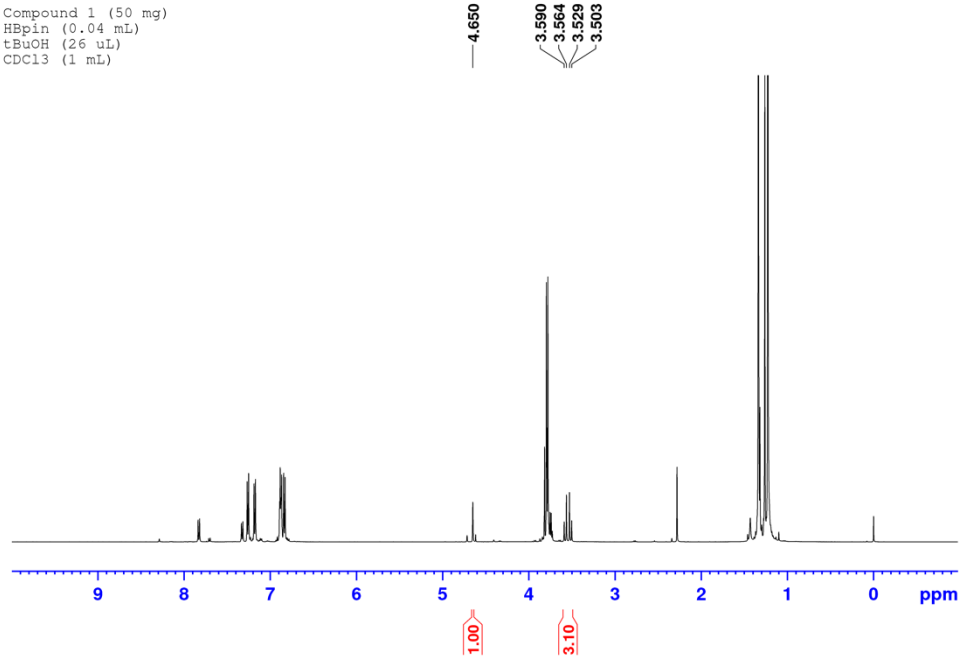
Water in THF:

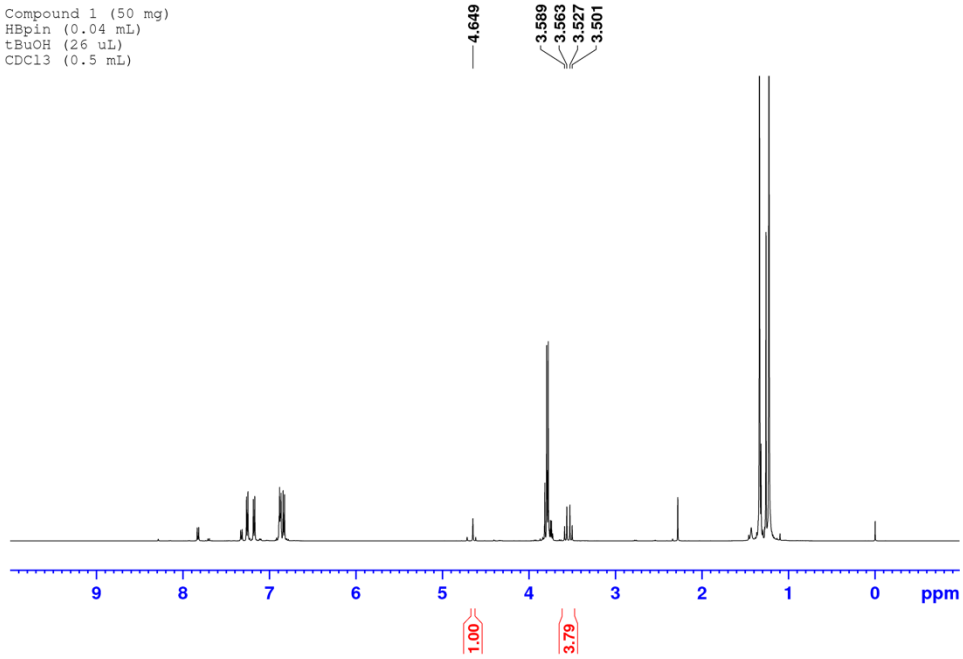


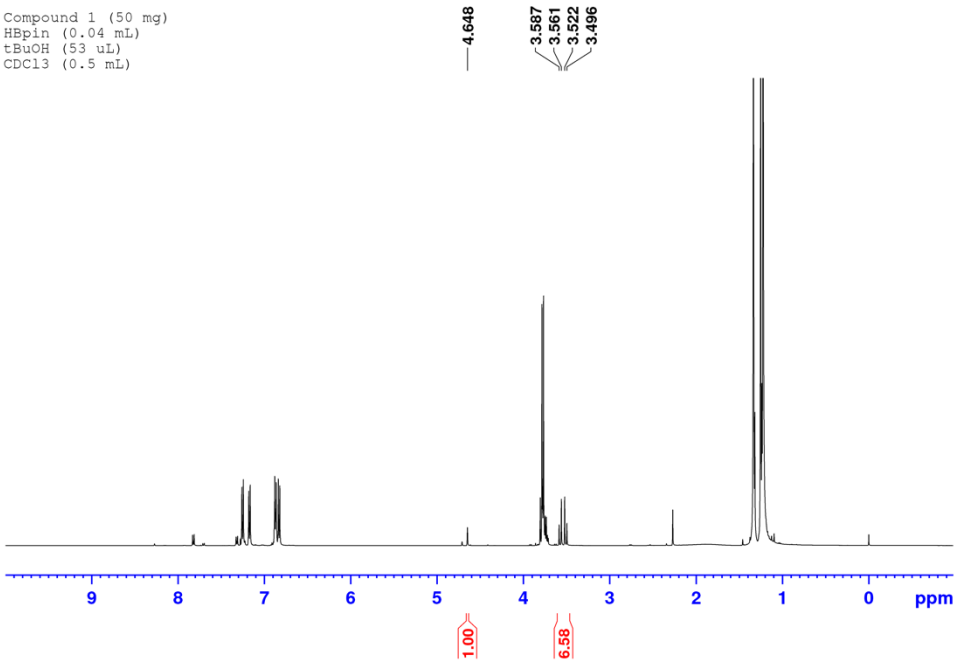
Water in acetonitrile:

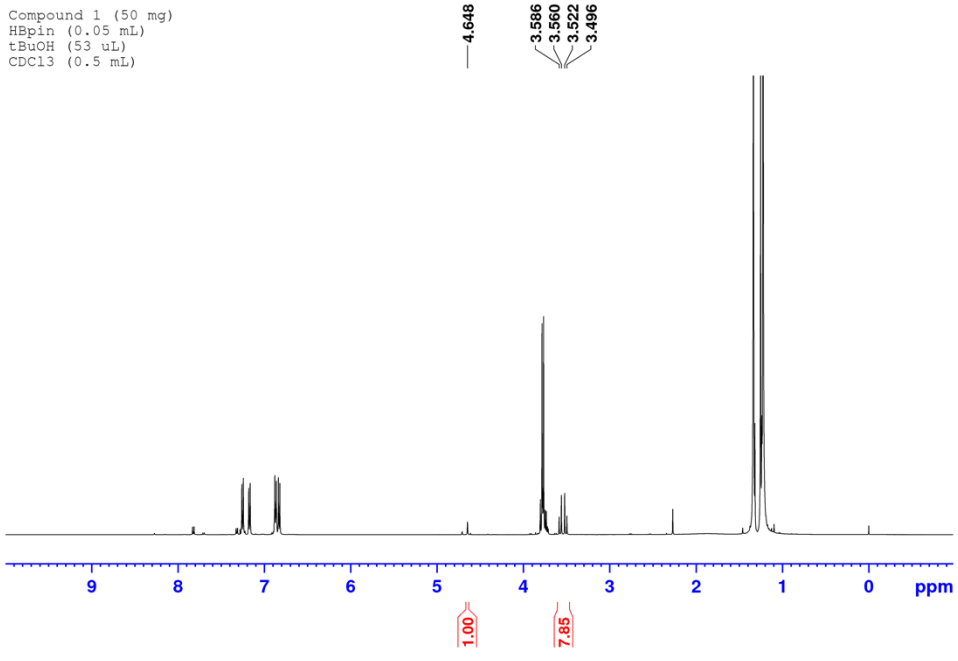


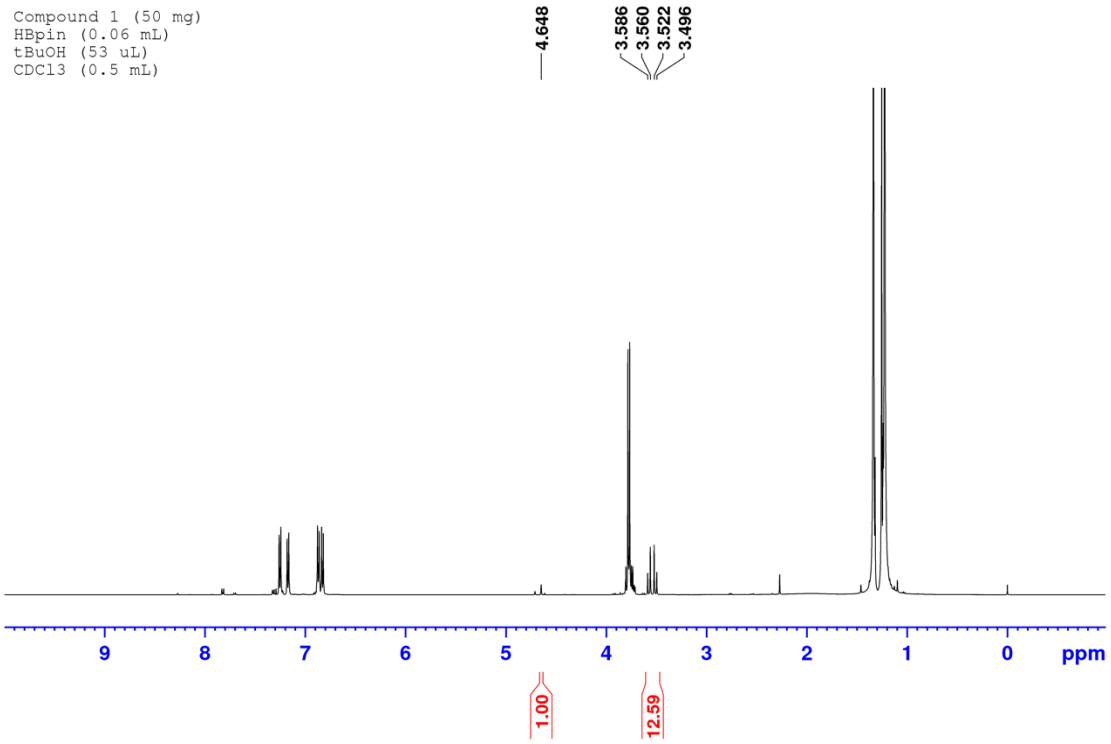
Spectra from varying ratios of *tert*-butanol and pinacolborane



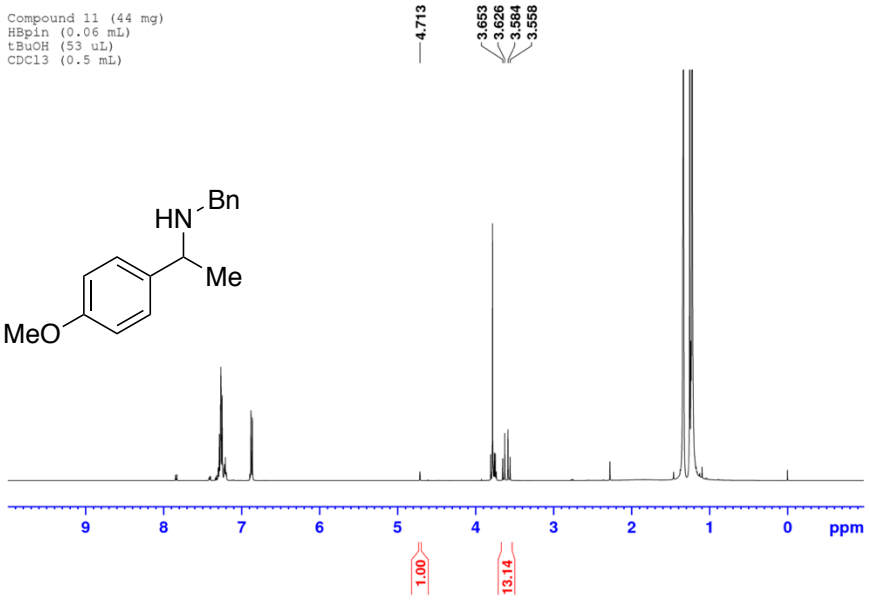



Compound 1 (50 mg)
HBpin (0.04 mL)
tBuOH (53 uL)
CDCl3 (0.5 mL)
4.648
3.587
3.561
3.522
3.496
9
8
7
6
5
4
3
2
1
0
ppm
1.00
6.58


Compound 1 (50 mg)
HBpin (0.05 mL)
tBuOH (53 uL)
CDCl3 (0.5 mL)
4.648
3.586
3.560
3.522
3.496
9
8
7
6
5
4
3
2
1
0
ppm
1.00
7.85

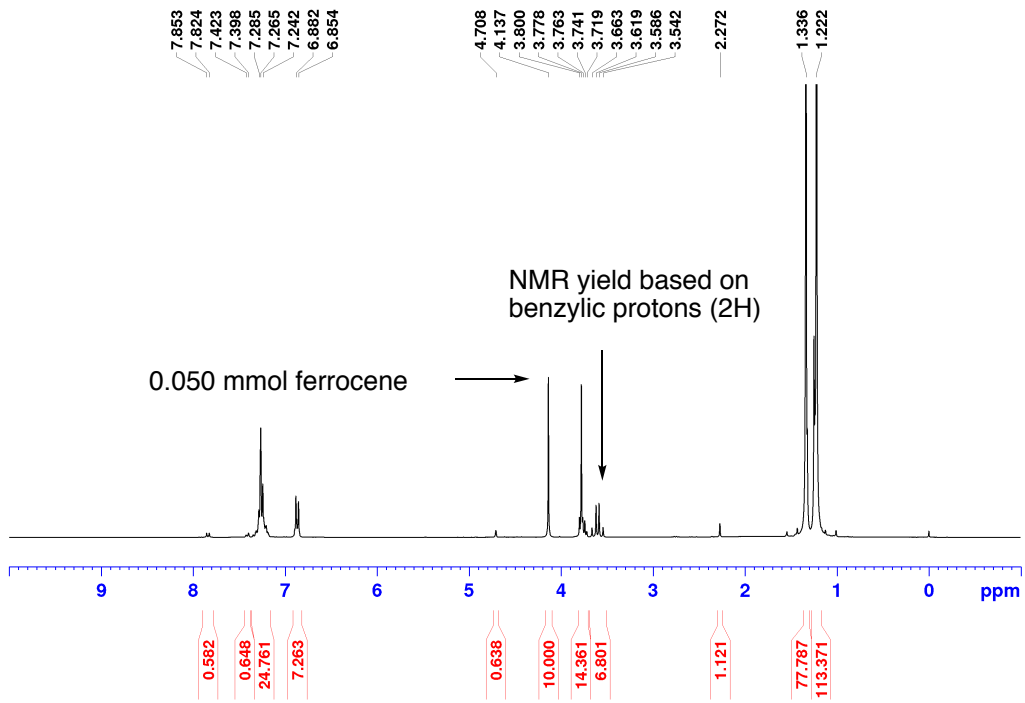

Compound 1 (50 mg)
HBpin (0.06 mL)
tBuOH (53 uL)
CDCl3 (0.5 mL)
4.648
3.586
3.560
3.522
3.496
9
8
7
6
5
4
3
2
1
0
ppm
1.00
12.59

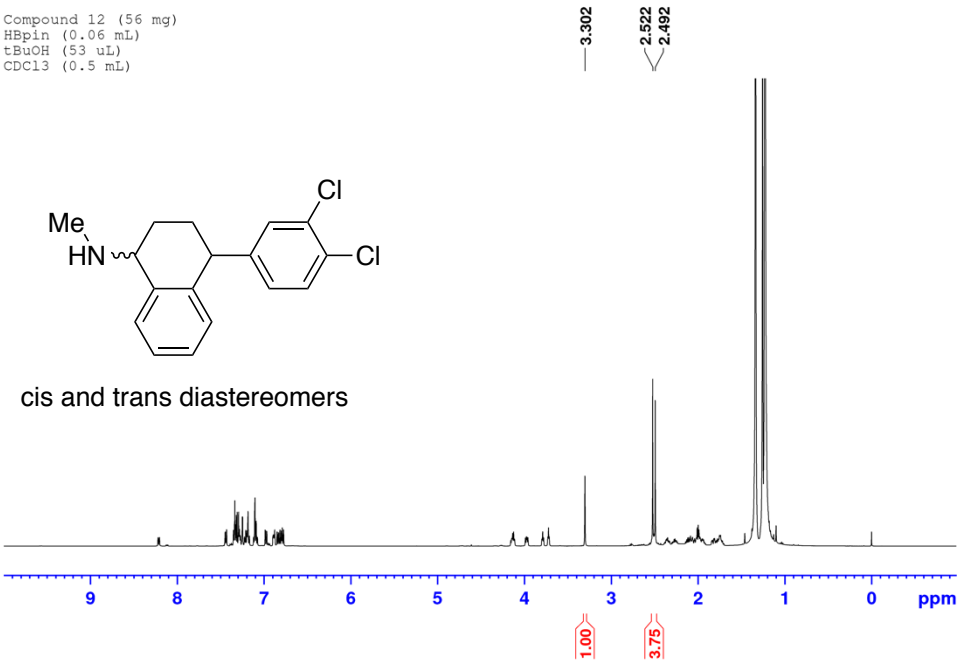
Reductions of imines shown in scheme 4 Without Internal standard:



Repeat with internal standard

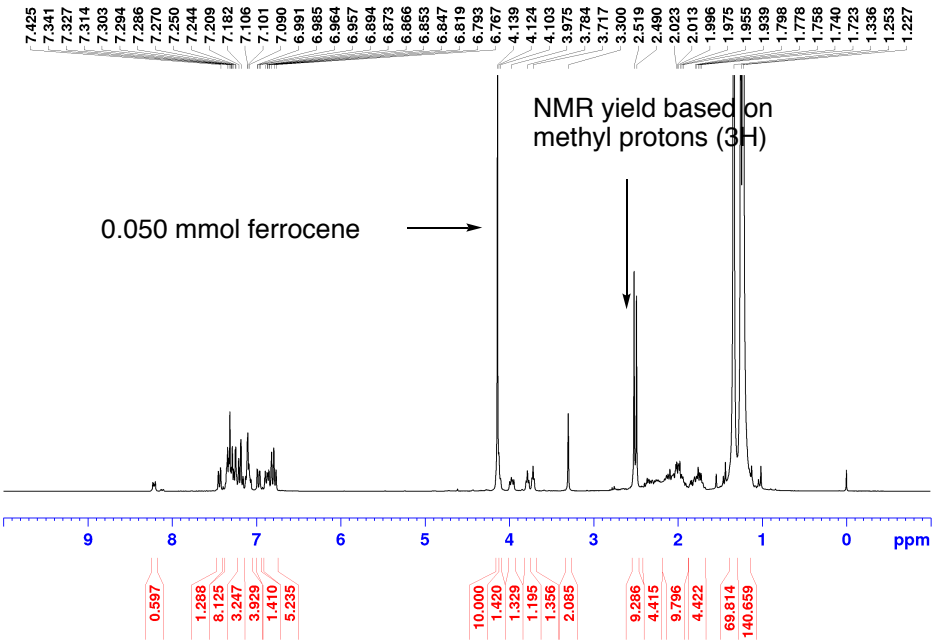
6.801*0.05/2 = 0.1700 mmol pdt 0.186 mmol sm added = 91% NMR Yield

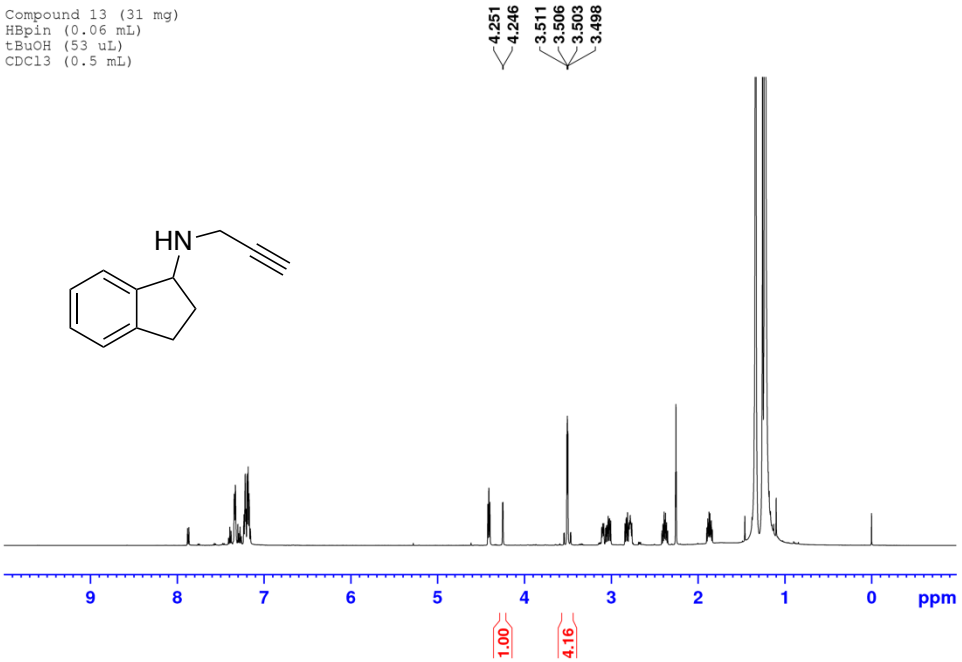




Repeat with internal standard:

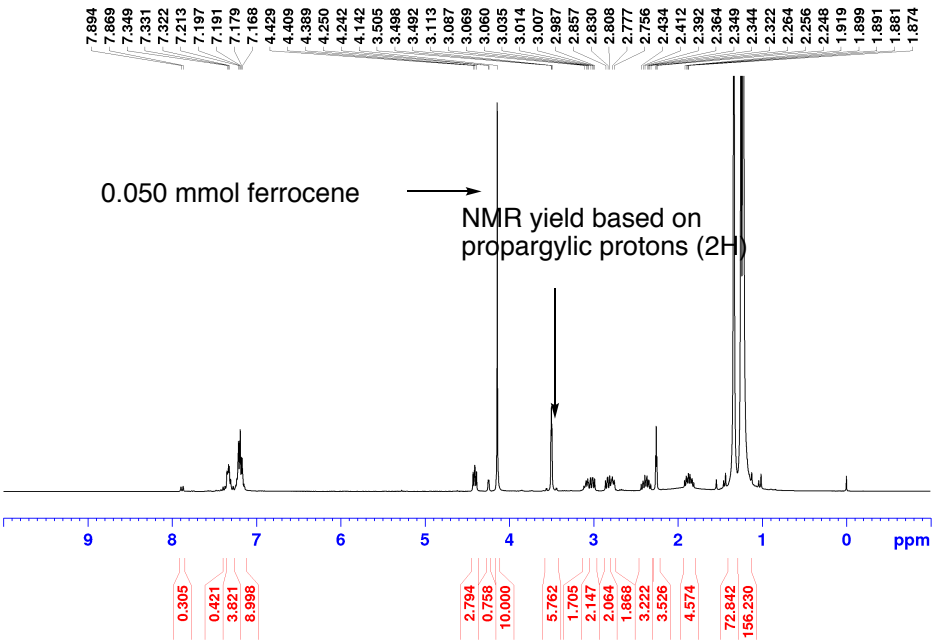
9.286*0.05/3= 0.1548 mmol products, 0.186 mmol added = 83% NMR yield

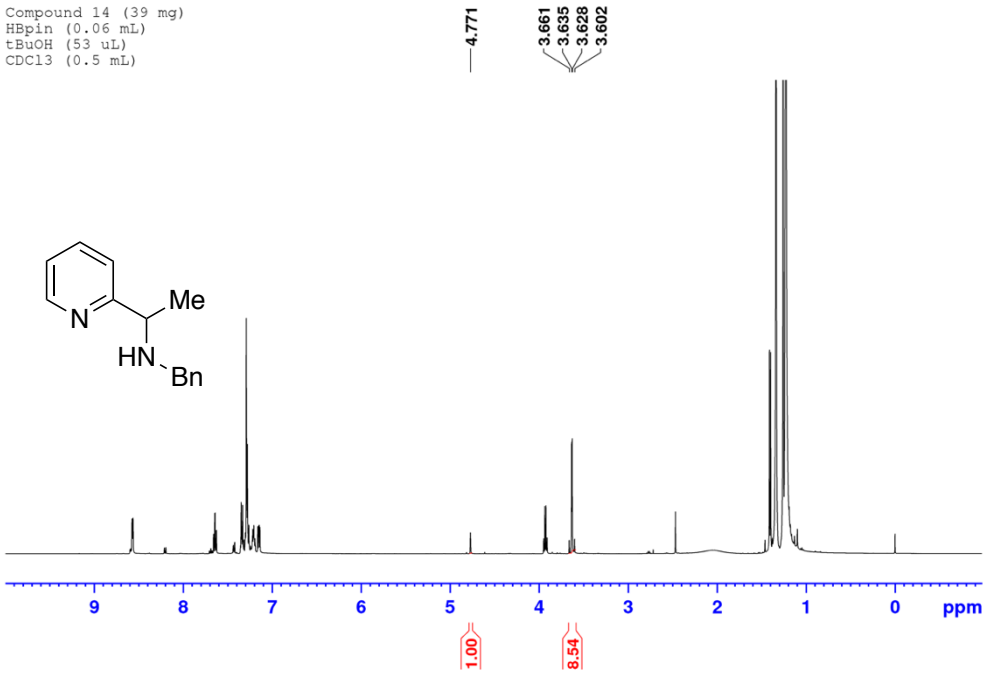




Repeat with Internal Standard

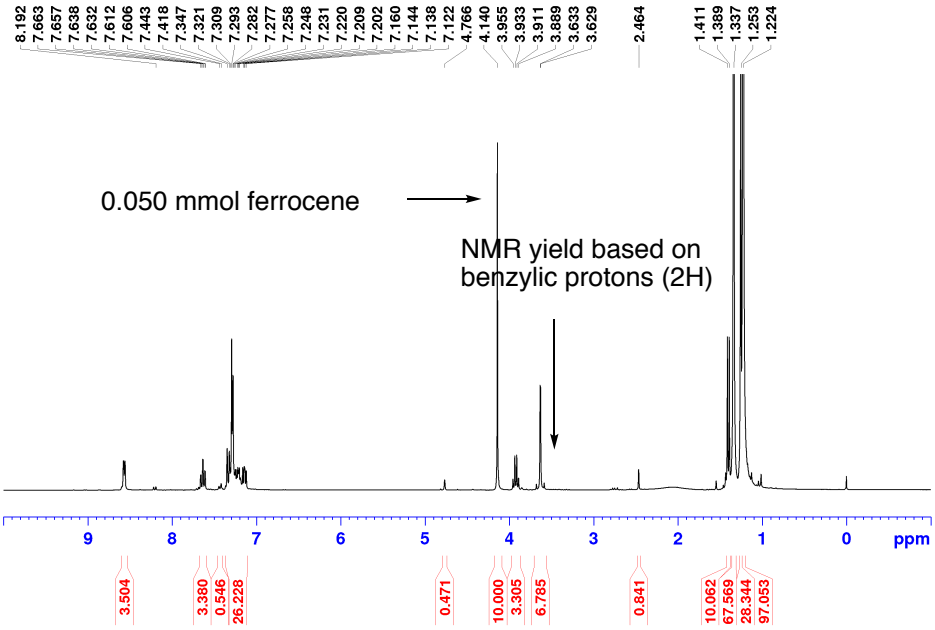
5.726*0.05/2 = 0.143 mmol product, 0.186 mmol sm added = 77% NMR yield

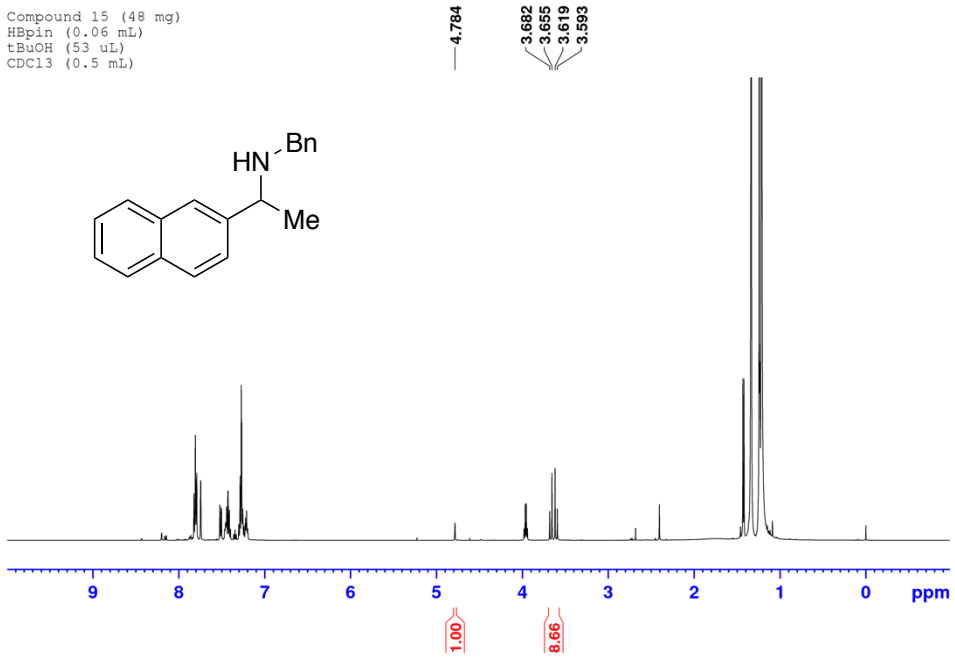




Repeat reaction with internal standard:

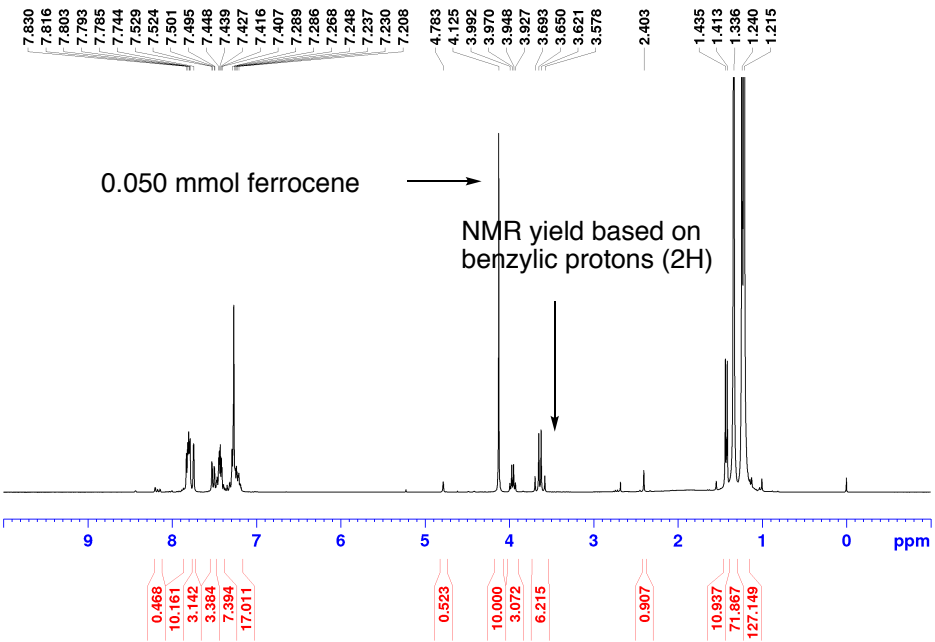
6.786*0.05/2 = 0.1697 mmol product, 0.186 mmol added = 92% NMR yield

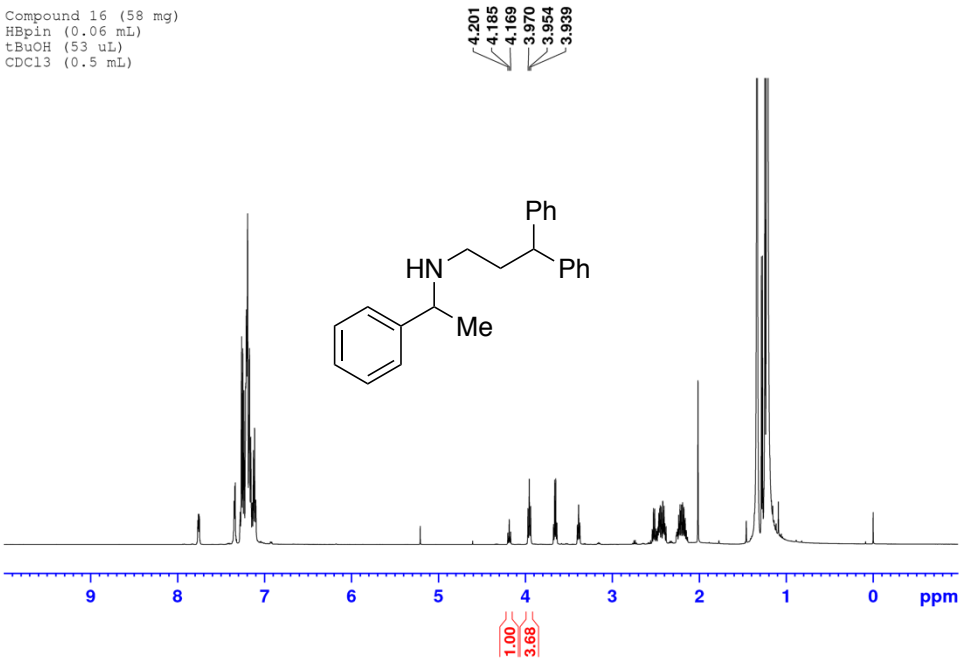




Repeat reaction with internal standard

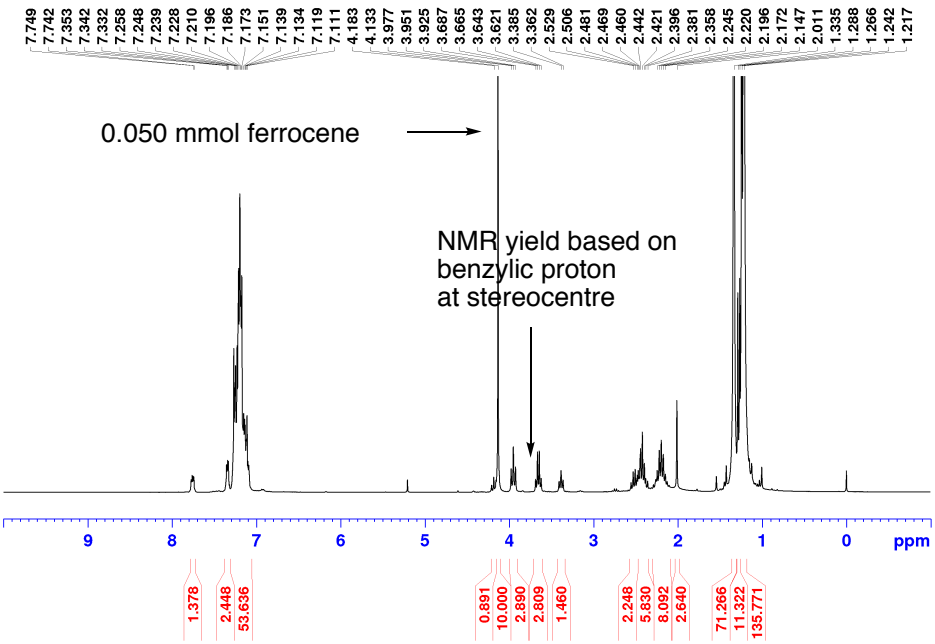
6.215*0.05/2=0.1554 mmol product, 0.186 mmol added = 84% NMR Yield



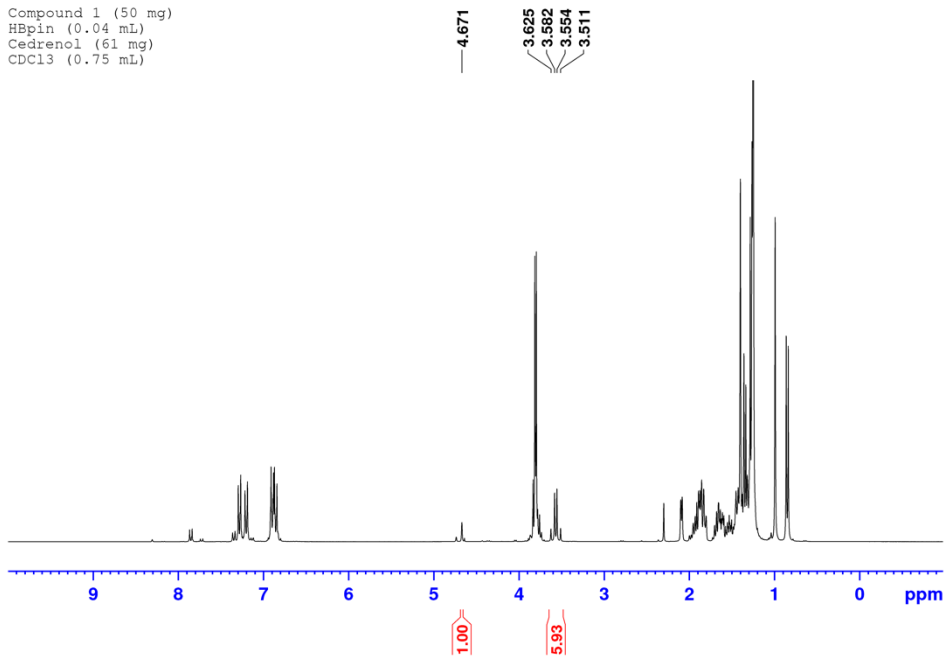


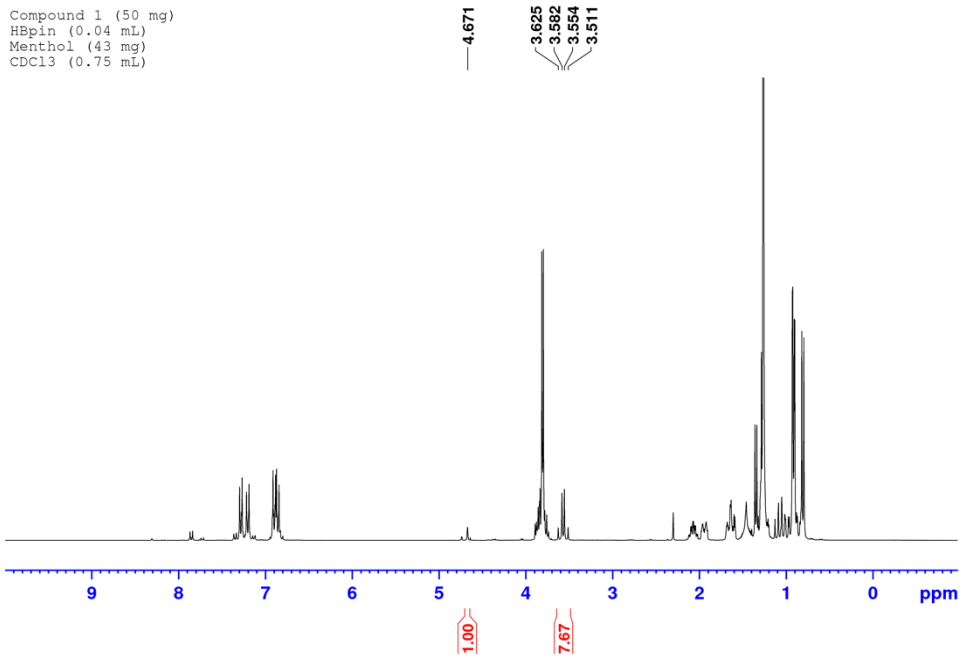
Repeat reaction with internal standard

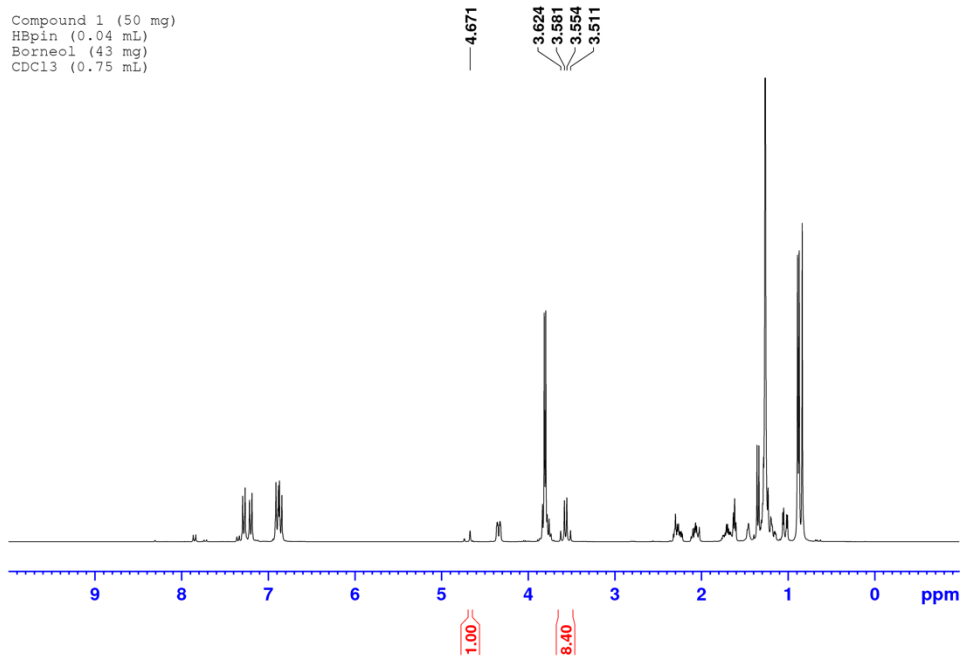
2.809*0.05=0.140 mmol product, 0.186 mmol added = 76% NMR Yield

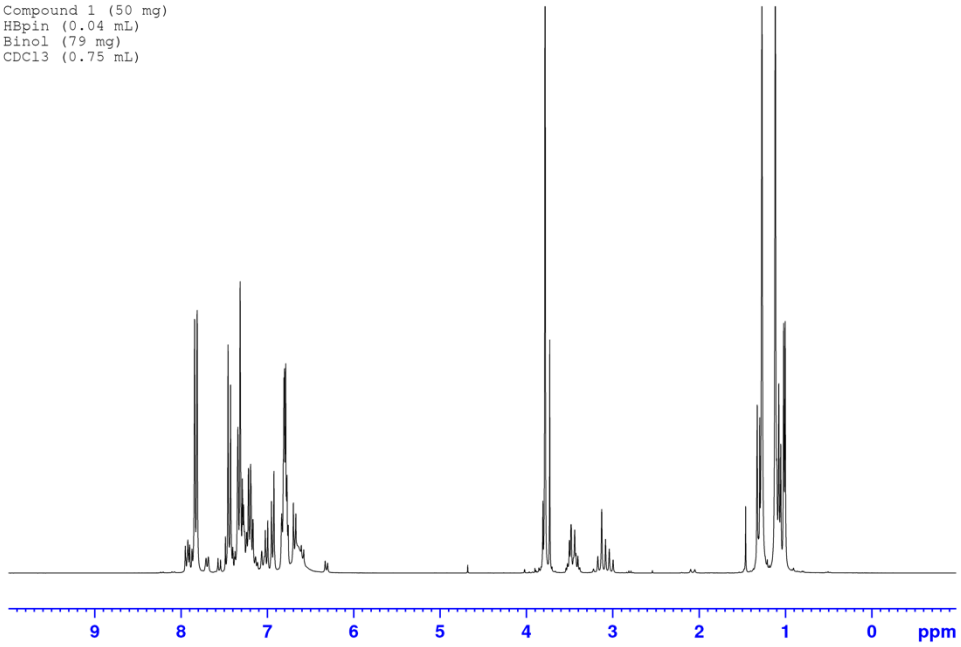


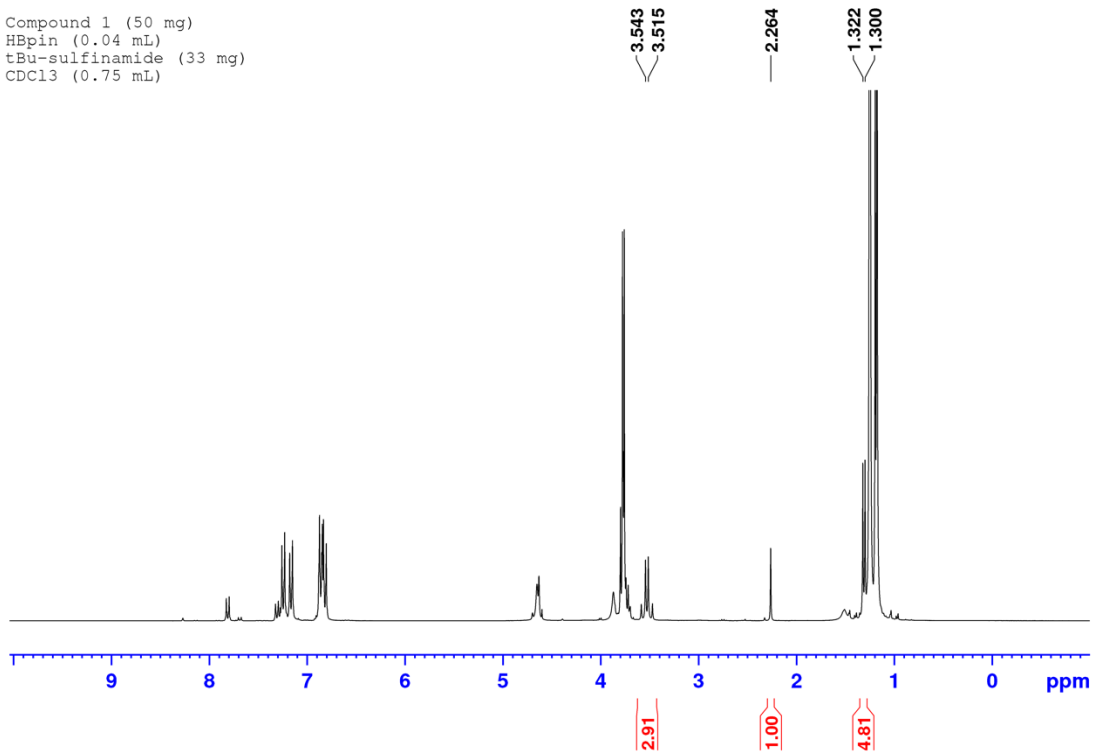
NMR spectra of additions of chiral additive additions



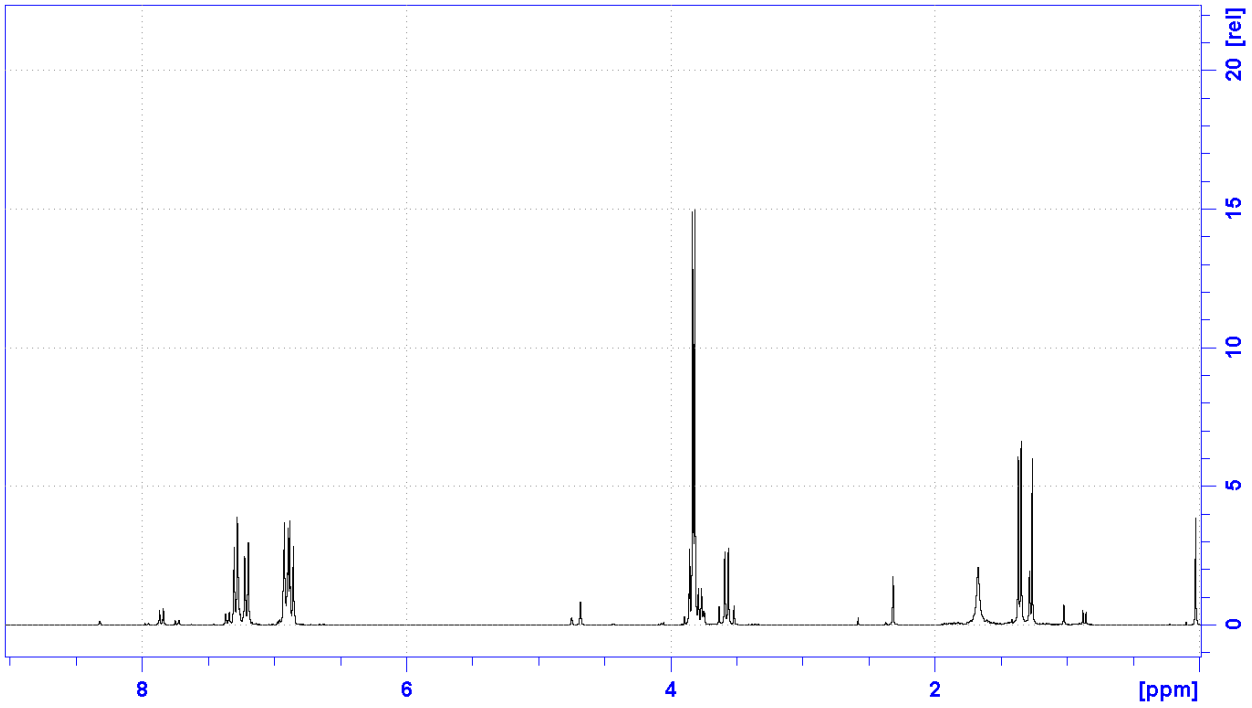



Compound 1 (50 mg)
HBpin (0.04 mL)
Borneol (43 mg)
CDCl3 (0.75 mL)
4.671
3.624
3.581
3.554
3.511
9
8
7
6
5
4
3
2
1
0
ppm
1.00
8.40


Compound 1 (50 mg)
HBpin (0.04 mL)
Binol (79 mg)
CDCl3 (0.75 mL)
9
8
7
6
5
4
3
2
1
0
ppm

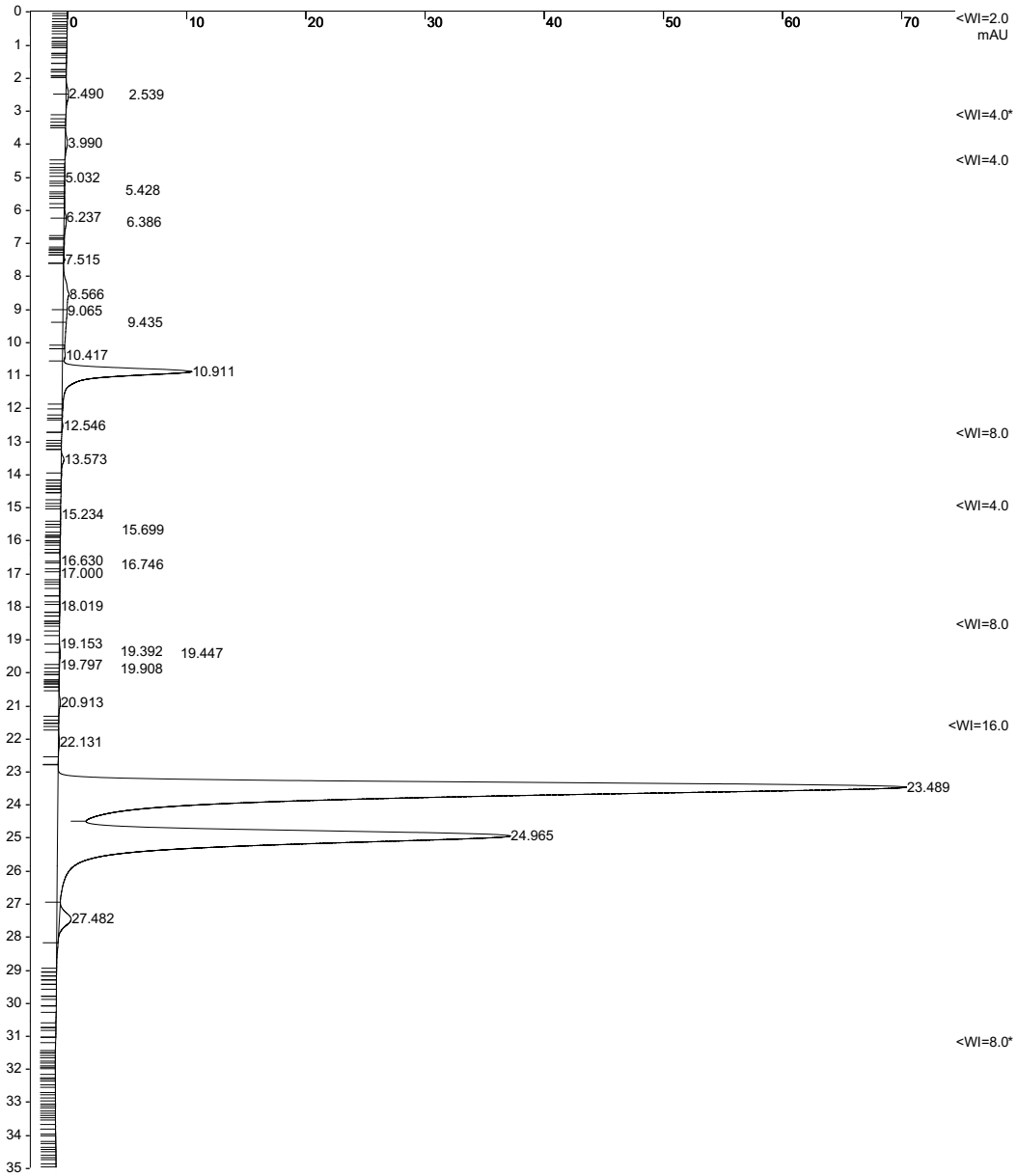


Binol reaction after acid/base workup



HPLC Trace from BINOL reaction

```
Title       :
Run File    : c:\star\data\brandon\matt adams\bh-xii-74b9-18-20185;31;25 pm.run
Method File : C:\star\estelle.mth
Sample ID   : bh-xii-74b

Injection Date: 9/18/2018 5:31 PM    Calculation Date: 9/18/2018 6:06 PM

Operator   : matt                    Detector Type: 0325
Workstation: DALHOUSI-GZ0GPÿ□HÚè-    Bus Address  : 44
Instrument : Analytical System       Sample Rate  : 20.00 Hz
Channel    : 1 = 254 nm              Run Time     : 35.000 min

** LC Workstation Version 6.41 ** 01907-64c0-ea4-04f1 **

Chart Speed =    0.59 cm/min   Attenuation = 3             Zero Offset = 4%
Start Time  =   0.000   min     End Time    = 35.000  min   Min / Tick  = 1.00
```



Print Date: Tue Sep 18 18:07:19 2018 Page 1 of 1

Title :
Run File : c:\star\data\brandon\matt adams\bh-xii-74b9-18-20185;31;25 pm.run
Method File : C:\star\estelle.mth
Sample ID : bh-xii-74b

Injection Date: 9/18/2018 5:31 PM Calculation Date: 9/18/2018 6:06 PM

Operator : matt Detector Type: 0325
Workstation: DALHOUSI-GZ0GPÿ□HÚè- Bus Address : 44
Instrument : Analytical System Sample Rate : 20.00 Hz
Channel : 1 = 254 nm Run Time : 35.000 min

** LC Workstation Version 6.41 ** 01907-64c0-ea4-04f1 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.0730	2.490	0.000	26347	PV	0.0	
2		0.0906	2.539	0.000	32693	VB	27.8	
3		0.1361	3.990	0.000	49086	BB	25.9	
4		0.0077	5.032	0.000	2775	VV	2.8	
5		0.0285	5.428	0.000	10279	VV	0.8	
6		0.0517	6.237	0.000	18664	VV	12.7	
7		0.1656	6.386	0.000	59746	VP	22.5	
8		0.0175	7.515	0.000	6306	TS	0.0	
9		0.5978	8.566	0.000	215623	PV	0.0	
10		0.2032	9.065	0.000	73295	VV	0.0	
11		0.2944	9.435	0.000	106196	VV	41.4	
12		0.1125	10.417	0.000	40562	VV	7.2	
13		5.1533	10.911	0.000	1858862	VB	14.0	
14		0.0198	12.546	0.000	7125	TS	0.0	
15		0.1234	13.573	0.000	44525	VB	14.3	
16		0.0162	15.234	0.000	5855	VV	11.3	
17		0.0113	15.699	0.000	4079	VB	3.2	
18		0.0074	16.630	0.000	2667	PV	7.9	
19		0.0100	16.746	0.000	3606	VV	21.3	
20		0.0158	17.000	0.000	5710	VP	1.3	
21		0.0069	18.019	0.000	2497	TF	0.0	
22		0.0090	19.153	0.000	3237	VV	8.7	
23		0.0273	19.392	0.000	9861	VV	15.1	
24		0.0443	19.447	0.000	15973	VV	27.6	
25		0.0069	19.797	0.000	2476	VV	6.3	
26		0.0232	19.908	0.000	8356	VV	0.0	
27		0.0822	20.913	0.000	29648	VP	15.3	
28		0.0553	22.131	0.000	19940	VP	20.8	
29		58.5499	23.489	0.000	21119578	PV	25.9	
30		33.3011	24.965	0.000	12012069	VP	26.1	
31		0.7580	27.482	0.000	273416	TS	0.0	
	Totals:	99.9999		0.000	36071052			

Total Unidentified Counts : 36071052 counts

Detected Peaks: 162 Rejected Peaks: 131 Identified Peaks: 0

Multiplier: 1 Divisor: 1 Unidentified Peak Factor: 0

Baseline Offset: 0 microAU LSB: 0.1 microAU

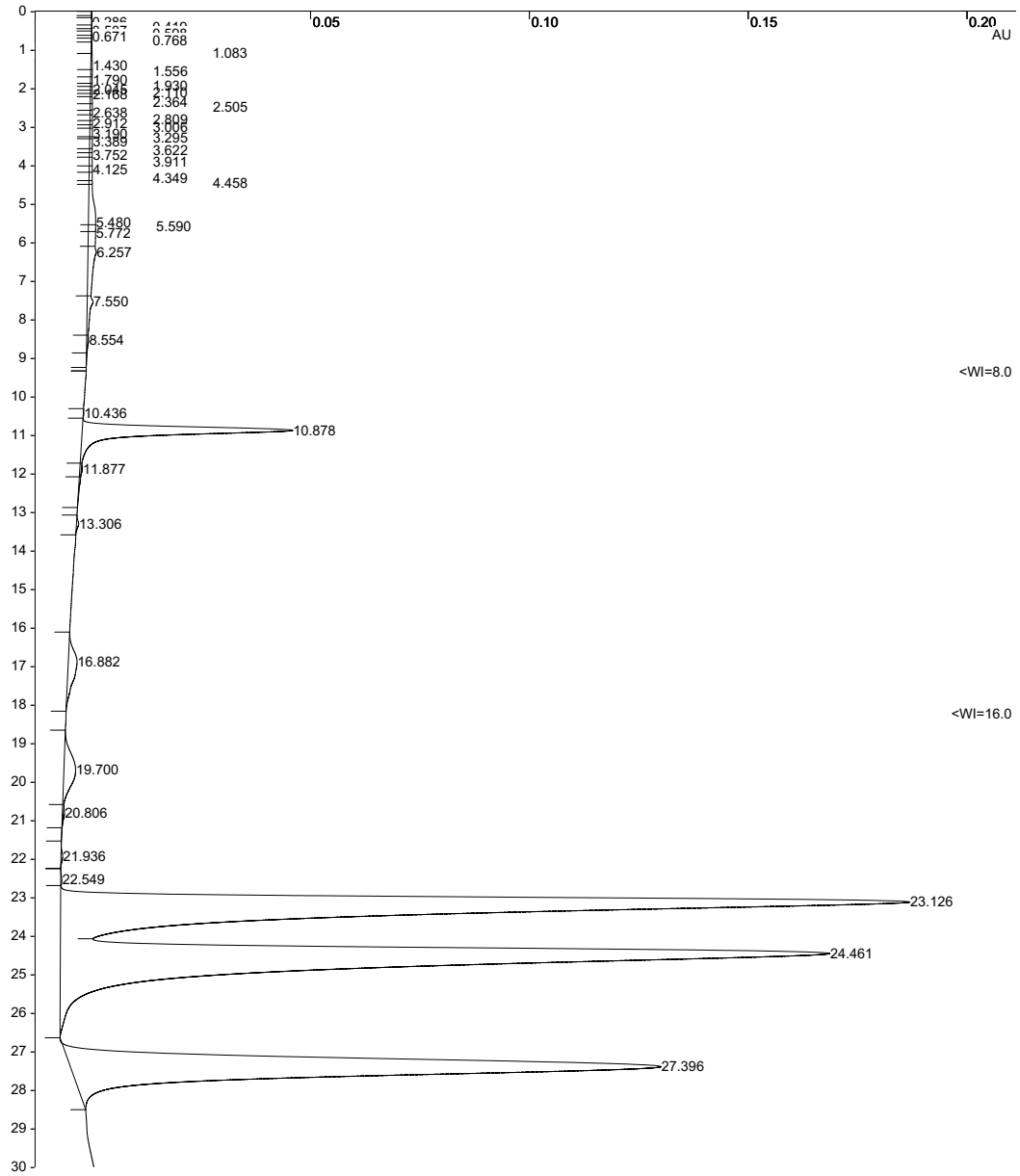
Noise (used): 0 microAU - monitored before this run

Vial: 2 Injection Number: 1 Partial Loopfill Volume: 10 ul

****

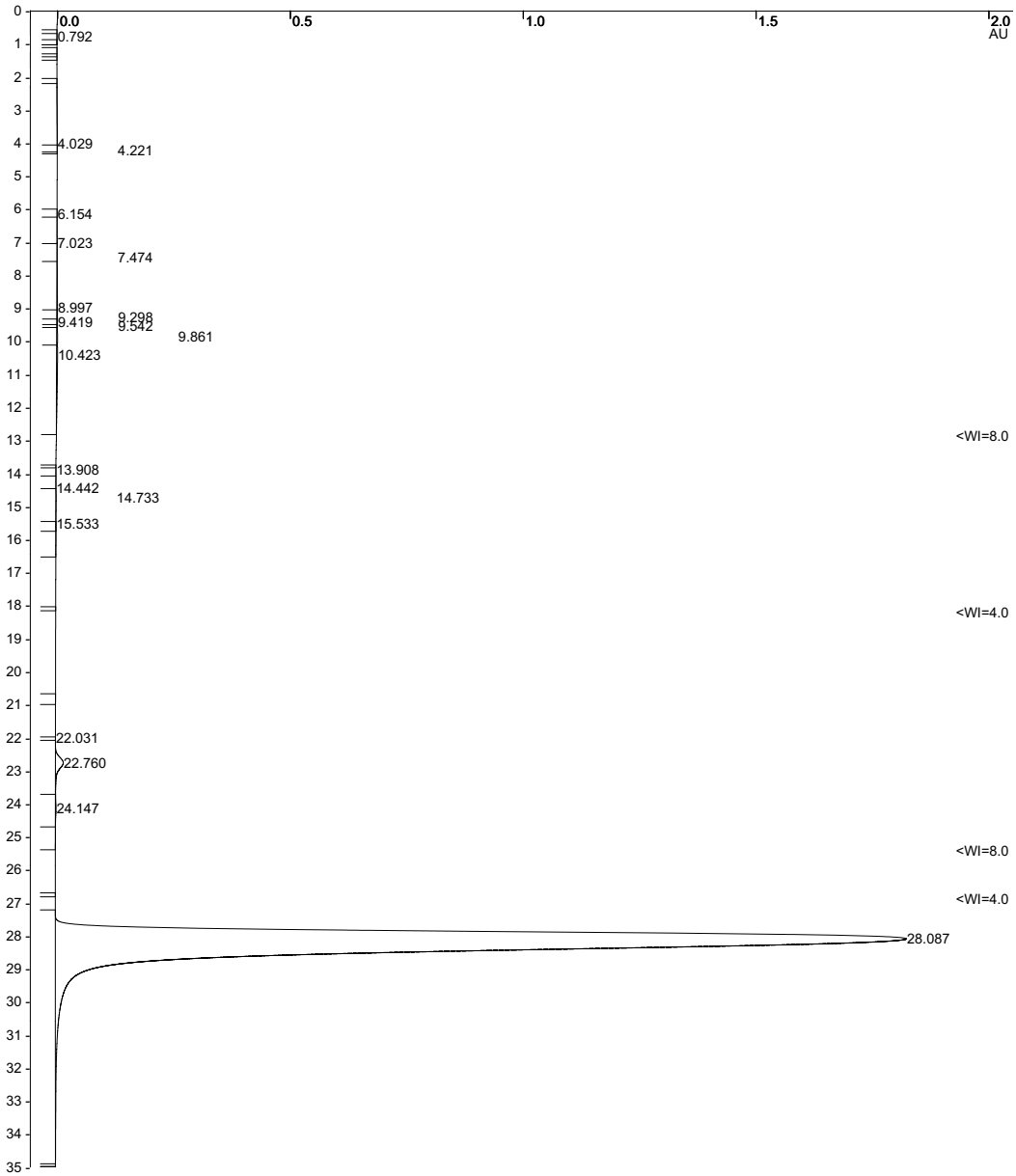
Racemate:

```
Title       :
Run File    : c:\star\data\brandon\matt adams\bh-xii-75d real9-18-20187;55;14 pm.run
Method File : C:\star\estelle.mth
Sample ID   : bh-xii-75d real

Injection Date: 9/18/2018 7:55 PM    Calculation Date: 9/18/2018 8:25 PM

Operator   : matt                    Detector Type: 0325
Workstation: DALHOUSI-GZ0GPÿ□HÚè-    Bus Address  : 44
Instrument : Analytical System       Sample Rate  : 20.00 Hz
Channel    : 1 = 254 nm              Run Time     : 30.000 min

** LC Workstation Version 6.41 ** 01907-64c0-ea4-04f1 **

Chart Speed =    0.69 cm/min   Attenuation = 9             Zero Offset = 5%
Start Time  =   0.000   min     End Time    = 30.000  min   Min / Tick  = 1.00
```

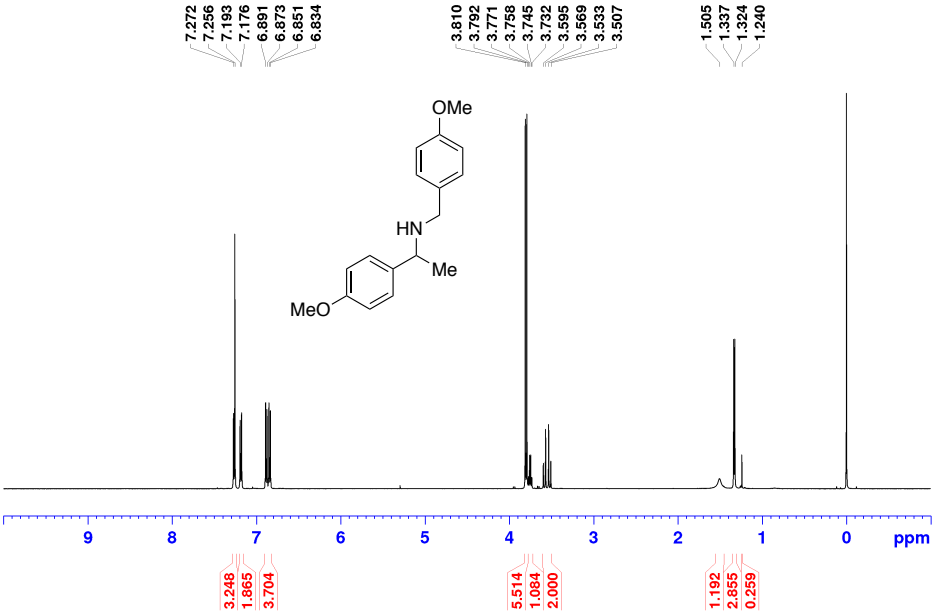


The peak at 27.396 arises from ketone impurity in the sample, arising from imine hydrolysis. A trace of injection of the ketone is shown below.

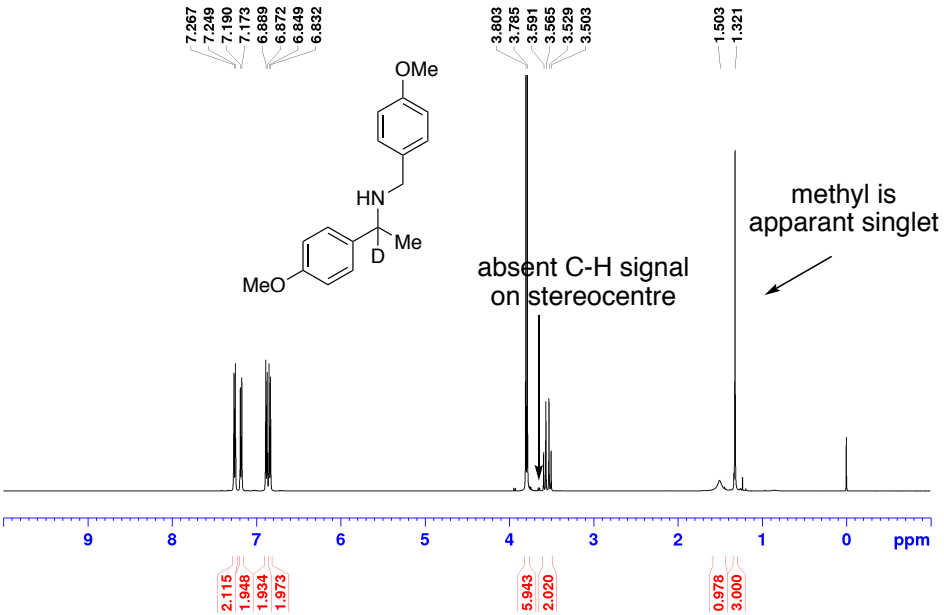
```
Title       :
Run File    : c:\star\data\brandon\matt adams\pmethoxy ketone9-19-20181;18;30 pm.run
Method File : C:\star\estelle.mth
Sample ID   : pmethoxy ketone

Injection Date: 9/19/2018 1:18 PM    Calculation Date: 9/19/2018 1:53 PM

Operator   : matt                   Detector Type: 0325
Workstation: DALHOUSI-GZ0GPÿ□HÚè-   Bus Address  : 44
Instrument : Analytical System      Sample Rate  : 20.00 Hz
Channel    : 1 = 254 nm             Run Time     : 35.000 min

** LC Workstation Version 6.41 ** 01907-64c0-ea4-04f1 **

Chart Speed =    0.59 cm/min   Attenuation = 86            Zero Offset = 2%
Start Time  =   0.000   min     End Time    = 35.000  min   Min / Tick  = 1.00
```

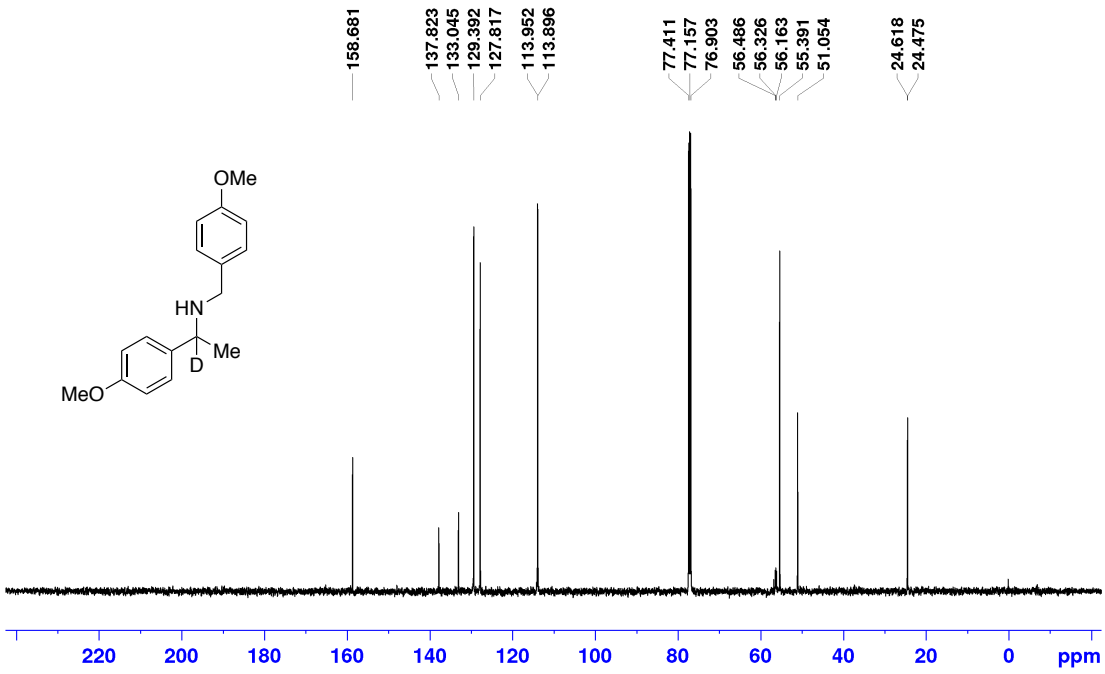


Spectral data of isolated product of 200 mg reaction:



Spectral data for deuterated reduction




158.681
137.823
133.045
129.392
127.817
113.952
113.896
77.411
77.157
76.903
56.486
56.326
56.163
55.391
51.054
24.618
24.475
OMe
HN
Me
D
MeO
220
200
180
160
140
120
100
80
60
40
20
0
ppm

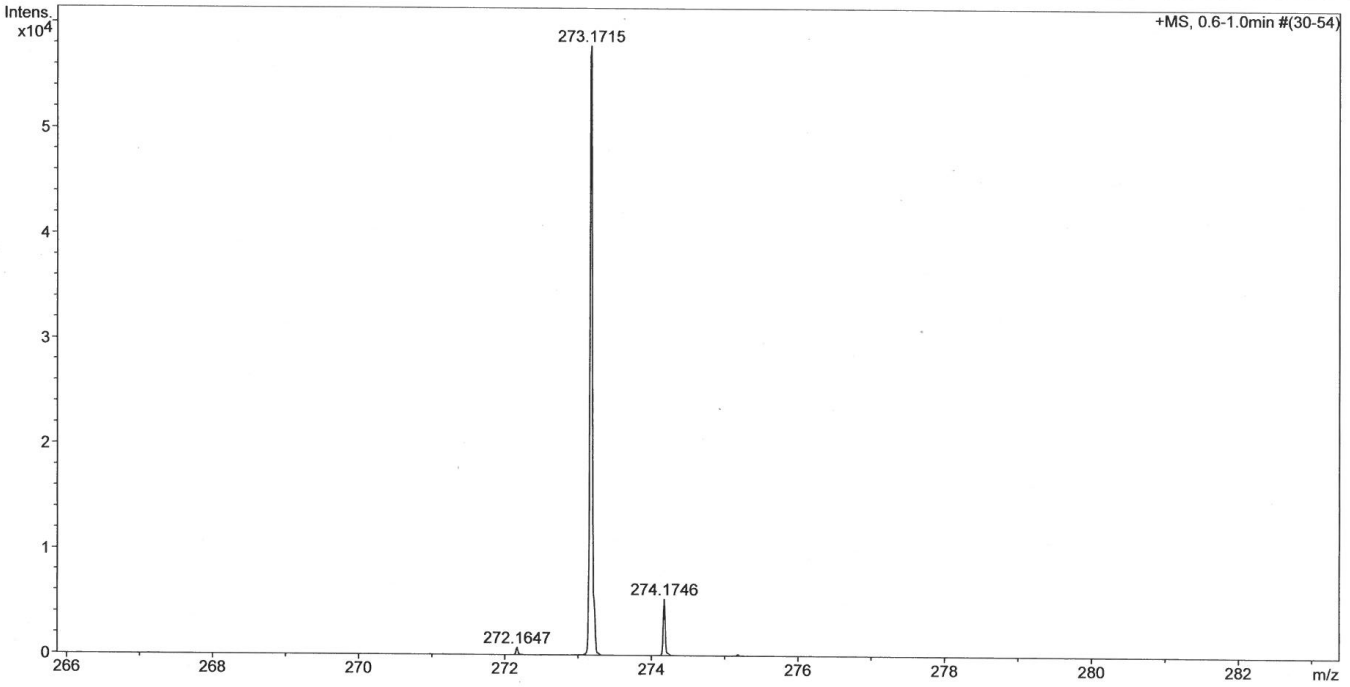
Analysis Info

Analysis Name	D:\Data\Xiao\Nov 01 2018000001.d	Acquisition Date	11/1/2018 9:32:17 AM
Method	Xiao 1.m		
Sample Name	BH-xiii-6	Operator	Administrator
Comment		Instrument	micrOTOF 57

Acquisition Parameter

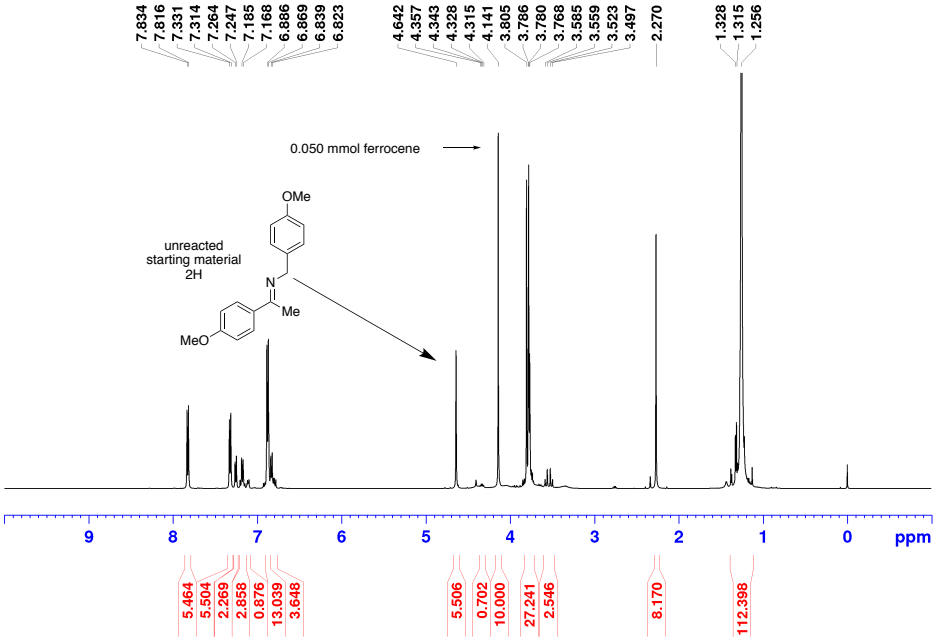
Source Type	ESI	Ion Polarity	Positive	Set Corrector Fill	45 V
Scan Range	n/a	Capillary Exit	100.0 V	Set Pulsar Pull	400 V
Scan Begin	50 m/z	Hexapole RF	120.0 V	Set Pulsar Push	400 V
Scan End	1500 m/z	Skimmer 1	50.0 V	Set Reflector	1300 V
		Hexapole 1	23.0 V	Set Flight Tube	9000 V
				Set Detector TOF	2200 V

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	rdb	N Rule	e^-
C 17 H 21 D 1 N 1 O 2	0.06	273.1708	-2.55	-2.34	7.50	ok	even



Spectral Data for Autocatalytic Reactions

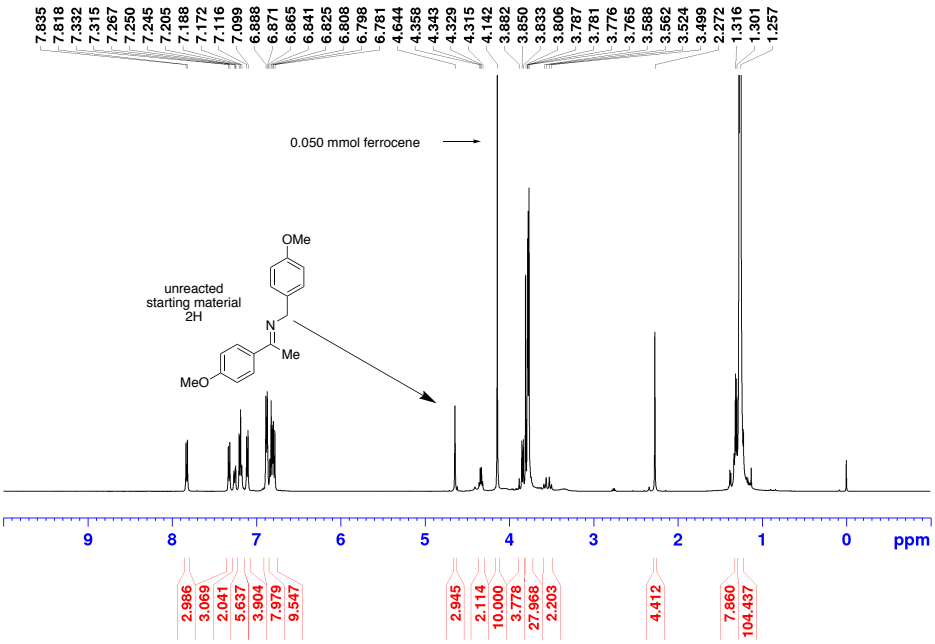
Spectrum for imine **1** after 30 minutes.



5.506*0.05/2 = 0.1376 mmol imine remain, 0.186 mmol imine were added

74% of imine remains, 26% was converted

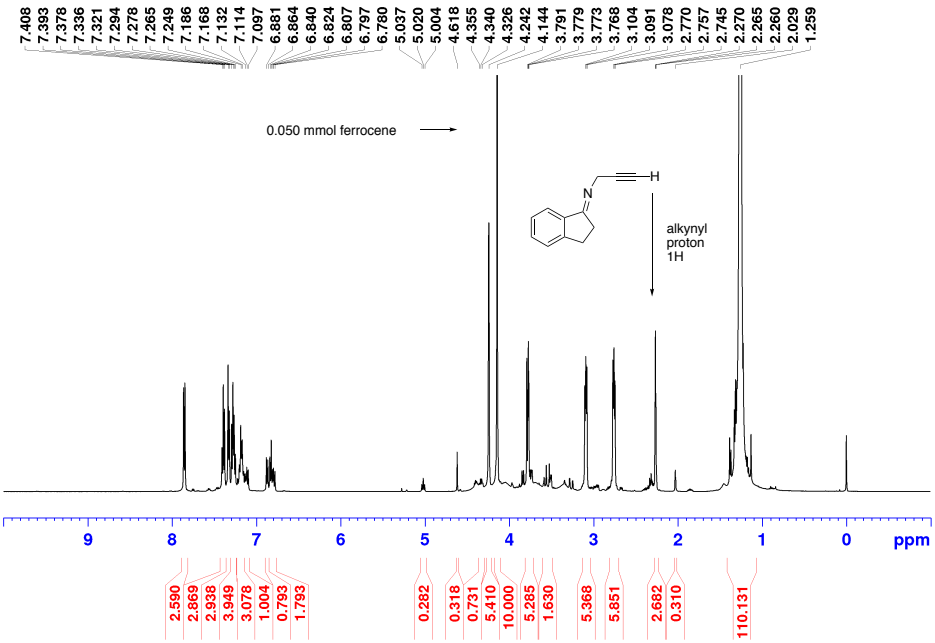
Spectrum for imine **1** after 24 hours



2.945*0.05/2= 0.0736 mmol imine remains, 0.186 mmol imine were added

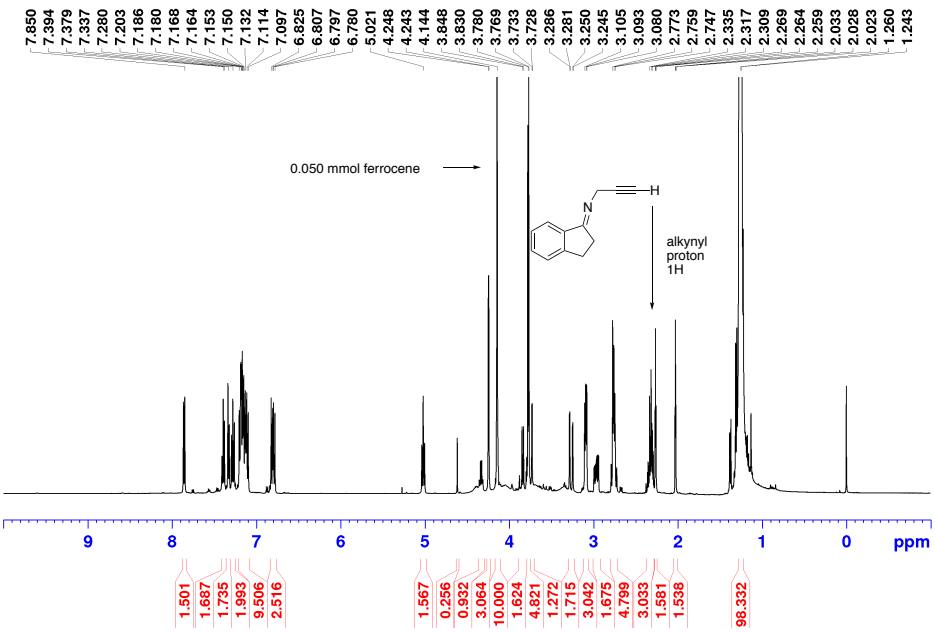
40% of imine remains, 60% was converted

Spectrum for imine **13** after 30 minutes



2.682*0.05 = 0.1341 mmol imine remain, 0.186 mmol imine were added

72% of imine remains, 28% was converted



1.581*0.05 = 0.07905 mmol imine remain, 0.186 mmol imine were added

42% of imine remains, 58% was converted