A Boron-Substituted Analogue of the Shvo Hydrogenation Catalyst: Catalytic Hydroboration of Aldehydes, Imines, and Ketones

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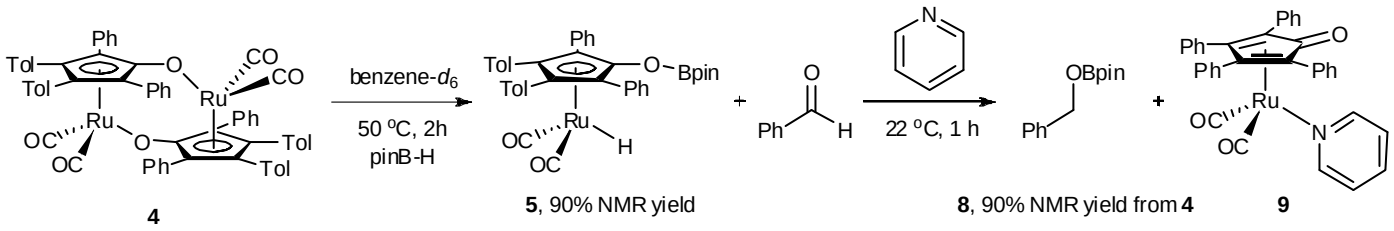
Supporting Information

Contents:

General Procedures. Melting points were obtained using an Electrothermal melting point apparatus and are reported uncorrected. ^{1}H, $^{13}C\{^{1}H\}$, and ^{11}B NMR spectra were obtained on a Varian Unity 500, Inova 500, or a Mercury 300 spectrometer. ^{11}B NMR resonances are reported on the δ scale with absolute referencing. The ^{1}H NMR data are reported as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, sext = sextet, and m = multiplet), coupling constants (Hz), and integration. The ^{13}C NMR chemical shifts are reported in ppm from tetramethylsilane on the δ scale, using tetramethylsilane as an internal standard. IR spectra were obtained on a Mattson Polaris or Mattson Genesis FT-IR spectrometer. ^{1}H NMR yields and product ratios determined by ^{1}H NMR spectroscopy were acquired using a 10 second relaxation delay. Microanalyses were performed by Desert Analytics, Tucson, AZ. Analytical thin-layer chromatography was performed on Whatman 0.50 mm PK6F silica gel 60 Å plates. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on EM Reagents silica gel (SiO_2) 60 (70–230) mesh.

All air-sensitive materials were manipulated under dry nitrogen in an Innovative Technologies or MBraun glovebox, or by standard high vacuum and Schlenk line techniques. Toluene and methylene chloride were dried in a solvent purification system by passing through an activated alumina column and an oxygen scavenging column under nitrogen, followed by deoxygenation (three cycles of freeze, pump, thaw). Pinacolborane was purchased from Aldrich and vacuum transferred prior to storage at –30 °C in the glovebox. [2,5-Ph_2-3,4-Tol_2(η^4-C_4CO)]$Ru(CO)_2]_2$ (**4**) was synthesized according to the literature procedure.[1] Imines **12** and *N*-benzylideneaniline were synthesized by known methods[2] and the spectral data matched the known literature values. 4′-nitroacetophenone, and all solid aldehydes were pumped into the glovebox and used without purification. All liquid aldehydes were distilled from a drying agent, degassed, and stored in the glovebox.

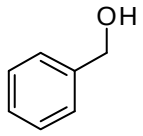
I. Stoichiometric Hydroboration of Benzaldehyde



Stoichiometric Hydroboration of Benzaldehyde with 5. To a resealable NMR tube containing {[2,5-Ph_2-3,4-Tol_2(η^4-C_4CO)Ru(CO)$_2$}$_2$ (**4**) (**0.500 g**, 0.028 mmol) was added benzene-d_6 (0.44 mL) and pinacolborane (0.062 mL, 0.031 mmol, 0.50 M solution in benzene-d_6). After 2 hours at 50 °C, a ^{1}H NMR spectra showed a 90% yield (0.028 mmol) of [2,5-Ph_2-3,4-Tol_2(η^4-C_4OBpin)]Ru(CO)$_2$H (**5**) as compared to a $PhSiMe_3$ internal standard. To the in situ prepared solution of **5** (0.028 mmol) in 0.60 mL of benzene-d_6 was added benzaldehyde (0.004 mL, 0.039 mmol) and pyridine (0.0035 mL, 0.043 mmol). After 1 hour at 22 °C, a ^{1}H NMR of the reaction mixture showed a 90% NMR yield of **8** relative to an internal $PhSiMe_3$ standard. ^{1}H NMR (C_6D_6, 300 MHz) characteristic peaks: δ 4.97 (s, 2H), 1.04 (s, 12H).

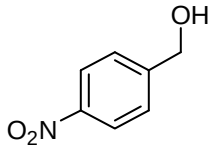
II. Catalytic Hydroboration of Carbonyls and Imines

General Procedure for the Catalytic Hydroboration of Aldehydes, Imines, and 4′-Nitroacetophenone



Benzyl alcohol[3]

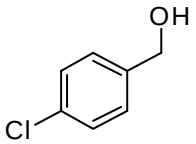
To a resealable glass tube containing {[2,5-Ph_2-3,4-Tol_2(η^4-C_4CO)Ru$(CO)_2$}$_2$ (**4**) (0.068 g, 0.060 mmol), 15 mL of toluene, and pinacolborane (0.700 mL, 4.50 mmol) was added benzaldehyde (0.305 mL, 3.00 mmol). After 26 h at 50 °C the reaction mixture was stirred over silica gel for 3 hours, filtered through a silica plug with ethyl acetate (~500 mL) and concentrated *en vacuo* to an orange/brown oil. Purification by bulb-to-bulb distillation (70–110 °C, 5 mm Hg) and column chromatography (2:98 to 3:97 ethyl acetate:CH_2Cl_2) provided benzyl alcohol as a colorless oil (0.254 g, 78%): ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.37 (m, 4H), 7.32 (m, 1H), 4.69 (s, 2H), 1.98 (s, 1H); ^{13}C-NMR ($CDCl_3$, 125 MHz) δ 141.1, 128.7, 127.8, 127.2, 65.3.**;** IR (thin film) 3335, 3064, 3031, 2931, 2873, 1496, 1454, 1020, 734, 698 cm^{-1}.



4-nitrobenzyl alcohol[3]

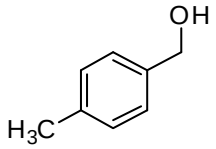
The general procedure was followed at 50 °C for 1 hour. Purification by column chromatography (5:95 ethyl acetate:CH_2Cl_2) gave 4-nitrobenzyl alcohol (0.402 g, 88%) as a pale brown solid: mp 91.1–92.3 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.23 (d, J = 8.5, 2H), 7.54 (d, J = 9.0, 2H), 4.85 (d,

J = 5.0, 2H), 1.93 (t, *J* = 5.0, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 148.3, 147.5, 127.2, 124.0, 64.2; IR (thin film) 3522, 3112, 3081, 2922, 2868, 1602, 1508, 1344, 1057 cm^{-1}.



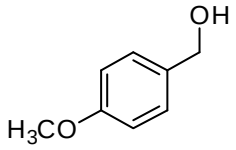
4-Chlorophenylmethanol[3]

The general procedure was followed using a 50 °C reaction temperature for 40 hours. Purification by column chromatography (2:98 ethyl acetate:CH_2Cl_2 to 5:95 ethyl acetate:CH_2Cl_2), followed by recrystallization with Hexanes to provide 4-chlorobenzyl alcohol (0.267 g, 62 %) as a white solid: mp 70.3–71.8 °C; ^{1}H-NMR ($CDCl_3$, 500 MHz) δ 7.32 (m, 4H), 4.68 (s, 2H), 1.69 (br s, 1H); ^{13}C-NMR ($CDCl_3$, 125 MHz) δ 139.2, 133.4, 128.7, 128.3, 64.5; IR (thin film) 3335, 3089, 3064, 3031, 2931, 2873, 1532, 1496, 1454, 1020, 734, 698 cm^{-1}.



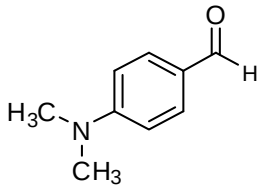
4-methylbenzyl alcohol[4]

The general procedure was followed using a 70 °C reaction temperature for 17 hours. Purification by column chromatography (4:96 ethyl acetate:CH_2Cl_2) gave 4-methylbenzyl alcohol (0.266 g, 72%) as a white solid: mp 60.2–61.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.26 (d, *J* = 7.5, 2H), 7.17 (d, *J* = 7.5, 2H), 4.63 (s, 2H), 2.35 (s, 3H), 1.73 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 138.1, 137.6, 129.4, 127.3, 65.5, 21.3; IR (thin film) 3347, 3055, 3025, 2950, 2921, 1517 cm^{-1}.



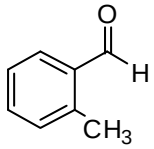
4-methoxybenzyl alcohol[3]

The general procedure was followed at 70 °C for 23 hours. Purification by column chromatography (6:94 ethyl acetate:CH_2Cl_2) gave 4-methoxybenzyl alcohol (0.377 g, 91%) as a yellow oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.28 (d, *J* = 8.5, 2H), 6.88 (d, *J* = 9.0, 2H), 4.61 (s, 2H), 3.81 (s, 3H), 1.72 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 159.4, 133.3, 128.8, 114.1, 65.2, 55.5; IR (thin film) 3601, 3447, 3054, 2987, 2880, 2838, 1612, 1514 cm^{-1}.



4-(*N*,*N*-Dimethylamino)benzyl alcohol[3]

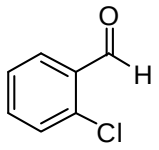
The general procedure was followed at 50 °C for 20 hours. Purification by column chromatography (12:88 ethyl acetate:CH_2Cl_2) and bulb-to-bulb distillation (80–95 °C, 20 mm Hg) gave 4-(N,N-dimethylamino)benzyl alcohol (0.274 g, 60%) as a colorless oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.23 (d, *J* = 7.3, 2H), 6.72 (d, *J* = 6.8, 2H), 4.55 (s, 2H), 2.91 (s, 6H), 1.69 (br s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 150.4, 128.9, 128.6, 112.7, 65.3, 40.7; IR (thin film) 3402, 2981, 2917, 1614, 1524 cm^{-1}.



2-Methylbenzyl alcohol[5]

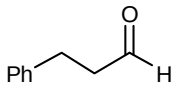
The general procedure was followed at 50 °C for 23 hours. Purification by column chromatography (20:80 ethyl acetate:CH_2Cl_2) and bulb-to-bulb distillation (75 °C, 10 mm Hg)

gave 2-methylbenzyl alcohol (0.490 g, 80%) as a white solid: mp 37.5–38.3 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.36 (m, 1H), 7.20 (m, 3H), 4.71 (d, *J* = 5.9, 2H), 2.37 (s, 3H), 1.51 (t, *J* = 5.9, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 138.9, 136.3, 130.6, 128.0, 127.8, 126.3, 63.8, 18.9; IR (thin film) 3415, 2924 cm^{-1}.



2-Chlorobenzyl alcohol[3]

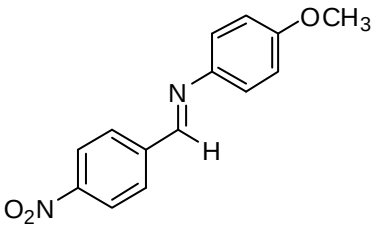
The general procedure was followed at 50 °C for 23 hours. Purification by column chromatography (2:98 ethyl acetate:CH_2Cl_2) and recrystallization from hexanes gave 2-chlorobenzyl alcohol (0.509 g, 71%) as a white solid: mp 70.1–71.7 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.49 (dd, *J* = 7.3, 1.5, 1H), 7.37 (dd, *J* = 7.8, 1.5, 1H), 7.27 (m, 2H), 4.79 (d, *J* = 6.4, 2H), 1.93 (t, *J* = 6.3, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 138.3, 132.9, 129.6, 129.0, 128.9, 127.2, 63.0; IR (thin film) 3428, 3054, 2986, 2305, 1421, 1265 cm^{-1}.



3-Phenyl-1-propanol[3]

The general procedure was followed at 50 °C for 6 hours. The crude reaction mixture was stirred with silica gel for one hour at room temperature, filtered through a silica plug with ethyl acetate (50 mL), and purified by bulb-to-bulb distillation (110 °C, 10 mm Hg) followed by purification by column chromatography (10:90 ethyl acetate:CH_2Cl_2) to give 3-phenyl-1-propanol (0.237 g, 58%) as a colorless oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.29 (t, *J* = 7.8, 2H), 7.21 (d, *J* = 7.8, 3H), 3.69 (d, *J* = 4.9, 2H), 2.72 (t, *J* = 7.6, 2H), 1.91 (tt, *J* = 7.8, 6.4, 2H), 1.31 (s, 1H); ^{13}C NMR

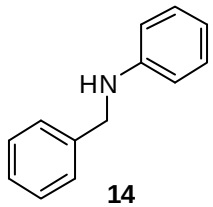
($CDCl_3$, 125 MHz) δ 142.0, 128.63, 128.61, 126.1, 62.5, 34.4, 32.3; IR (thin film) 3436, 3054, 2986, 2943, 1421, 1265 cm^{-1}.



13

***N*-(4′-methoxyphenyl)-*N*-(4-nitrobenzyl)imine (13)**

To a round bottom flask containing ethanol (20 mL) was added *p*-nitrobenzaldehyde (1.51 g, 10.0 mmol). A solution of *p*-anisidine (1.23 g, 10.0 mmol in ~3 mL ethanol) was added slowly over 5 minutes. After stirring for 22 hours, the solvent was removed en vacuo to provide a yellow solid. Recrystallization from ethanol gave **13** as a yellow solid: mp 134.6 – 135.8 ºC; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.58 (s, 1H), 8.32 (d, *J* = 8.5, 2H), 8.06 (d, *J* = 8.5, 2H), 7.31 (d, *J* = 9.0, 2H), 6.96 (d, *J* = 9.0, 2H), 3.86 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz); δ 159.5, 155.0, 149.2, 143.8, 142.2, 129.3, 124.2, 122.8, 114.8, 55.8; IR (thin film) 2952, 2893, 2836, 1643, 1586, 1508, 1344, 1247 cm^{-1}. LRMS (CI): *m*/*z* 257 ($M+H^+$).

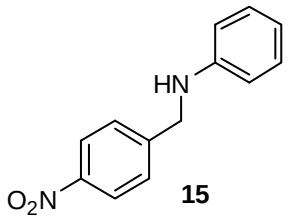


14

***N*-benzylaniline (14)**[6]

The general procedure was followed at 70 °C for 5 days. Purification by column chromatography (30:60 CH_2Cl_2:hexanes) gave **14** (0.454 g, 82%) as a brown oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.35 (m, 4H), 7.27 (m, 1H), 7.17 (m, 2H), 6.71 (t, *J* = 7.5, 1H), 6.63 (d, *J* = 8.0, 2H), 4.32 (s,

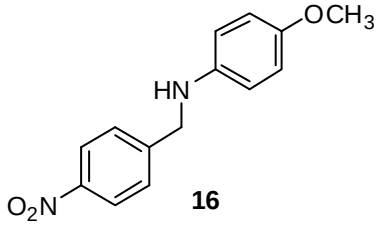
2H), 4.02 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 148.2, 139.4, 129.3, 128.6, 127.5, 127.2, 117.6, 112.8, 48.3; IR (thin film) 3419, 3051, 3026, 2849, 1602, 1507 cm^{-1}.



15

***N*-(4-nitrobenzyl)aniline (15)**[2]

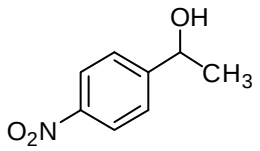
The general procedure was followed at 70 °C for 22 hours. Purification by column chromatography (45:55 CH_2Cl_2:hexanes) gave **15** (0.592 g, 86%) as a yellow oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.16 (d, *J* = 9.0, 2H), 7.51 (d, *J* = 8.5, 2H), 7.16 (m, 2H), 6.74 (t, *J* = 7.0, 1H), 6.57 (d, *J* = 7.5, 2H), 4.46 (s, 2H), 4.23 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 147.7, 147.5, 147.3, 129.5, 127.9, 124.1, 118.3, 113.1, 47.8; IR (thin film) 3441, 3054, 2986, 1603, 1523, 1347, 1265 cm^{-1}.



16

***N*-(4′-methoxyphenyl)-*N*-(4-nitrobenzyl)amine (16)**[7]

The general procedure was followed at 70 °C for 17 hours. Purification by column chromatography (65:35 CH_2Cl_2:hexanes) gave **16** (0.575 g, 74%) as a yellow-orange solid: mp 96.9-98.0° C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.19 (d, *J* = 8.5, 2H), 7.54 (d, *J* = 9.0, 2H), 6.77 (d, *J* = 9.5, 2H), 6.54 (d, *J* = 9.0, 2H), 4.43 (s, 2H), 3.73 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 152.8, 148.0, 147.4, 141.7, 128.0, 124.1, 115.2, 114.4, 56.0, 48.7; IR (thin film) 3445, 3055, 2987, 1605, 1515, 1422, 1347, 1265 cm^{-1}.



18

1-(4-nitrophenyl)-ethanol (18)[8]

The general procedure was followed at 70 °C for 4.5 days. Purification by column chromatography (1.5:98.5 ethyl acetate:CH_2Cl_2) gave **18** (0.396 g, 79%) as a yellow oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.21 (d, *J* = 9.0, 2H), 7.55 (d, *J* = 8.0, 2H), 5.03 (q, *J* = 6.5, 1H), 2.14 (s, 1H), 1.52 (d, *J* = 6.5, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz); δ 153.1, 147.2, 126.1, 123.8, 69.5, 25.5; IR (thin film) 3598, 3453, 3055, 2984, 2932, 1605, 1523, 1347, 1265 cm^{-1}.

III. Details of the Competition Experiments

General Procedure for Competition Reactions. To a resealable NMR tube containing {[2,5-Ph_2-3,4-Tol_2(η^4-C_4CO]Ru$(CO)_2$}$_2$ (Ru-dimer) **5** (0.004g, 0.004 mmol) was added 0.32 mL of a 0.50 M solution of phenyltrimethylsilane in benzene-d_6, the aldehydes (0.450 mmol of each), and 0.18 mL of a 0.5 M solution of pinacolborane in benzene-d_6 (0.090 mmol). After 2 hours at 50 °C, a ^{1}H-NMR (C_6D_6, 500 MHz) of the reaction was taken to determine the NMR yield relative to the internal phenyltrimethylsilane standard.

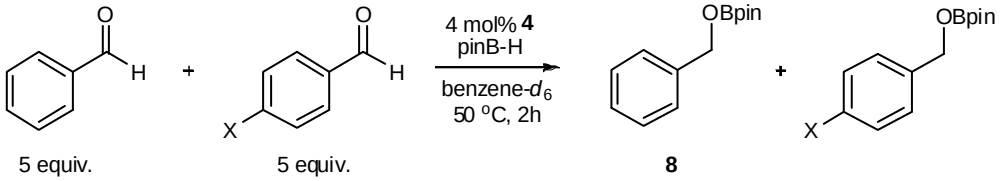
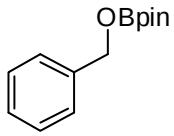


Table *S*-1. ^{1}H NMR Yields Resulting from the Competition Reactions

Substituent (X)	% NMR Yield[a] (X)	% NMR Yield (H)	Total % Yield	k_X/k_H
-OCH_3	26	65	91	0.40
-CH_3	30	42	72	0.71
-Cl	56	38	94	1.47
-NO_2[b]	81	18	99	4.52

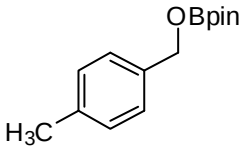
[a] Product ratios determined by ^{1}H NMR with phenyltrimethylsilane as the internal standard and a ten second relaxation delay. [b] Reaction run at 22 °C for two hours.

The identity of each product was confirmed by an independent reaction involving individual aldehydes, monitored by ^{1}H NMR spectroscopy, which was ultimately isolated and characterized after hydrolysis to form the benzyl alcohol product. The following characteristic resonances in the ^{1}H NMR spectra of independent reactions were used to verify the identity of each product in the competition experiments:



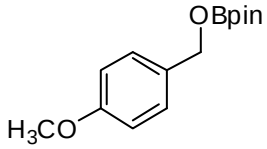
4,4,5,5-tetramethyl-2-benzyloxy-1,3,2-dioxaborolane

^{1}H NMR (C_6D_6, 300 MHz) δ 4.97 (s, 2H), 1.04 (s, 12H).



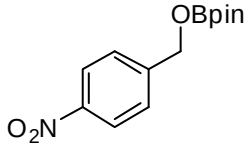
4,4,5,5-tetramethyl-2-(4-methylbenzyloxy)-1,3,2-dioxaborolane

^{1}H NMR (C_6D_6, 500 MHz) δ 4.93 (s, 2H), 2.10 (s, 3H).



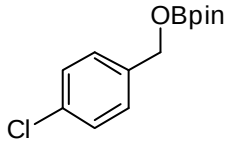
2-(4-methoxybenzyloxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane

^{1}H NMR (C_6D_6, 500 MHz) δ 4.91 (s, 2H), 3.35 (s, 3H).



4,4,5,5-tetramethyl-2-(4-nitrobenzyloxy)-1,3,2-dioxaborolane

^{1}H NMR (C_6D_6, 500 MHz) δ 4.73 (s, 2H).



2-(4-chlorobenzyloxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane

^{1}H NMR (C_6D_6, 500 MHz) δ 4.77 (s, 2H).

IV. **References**

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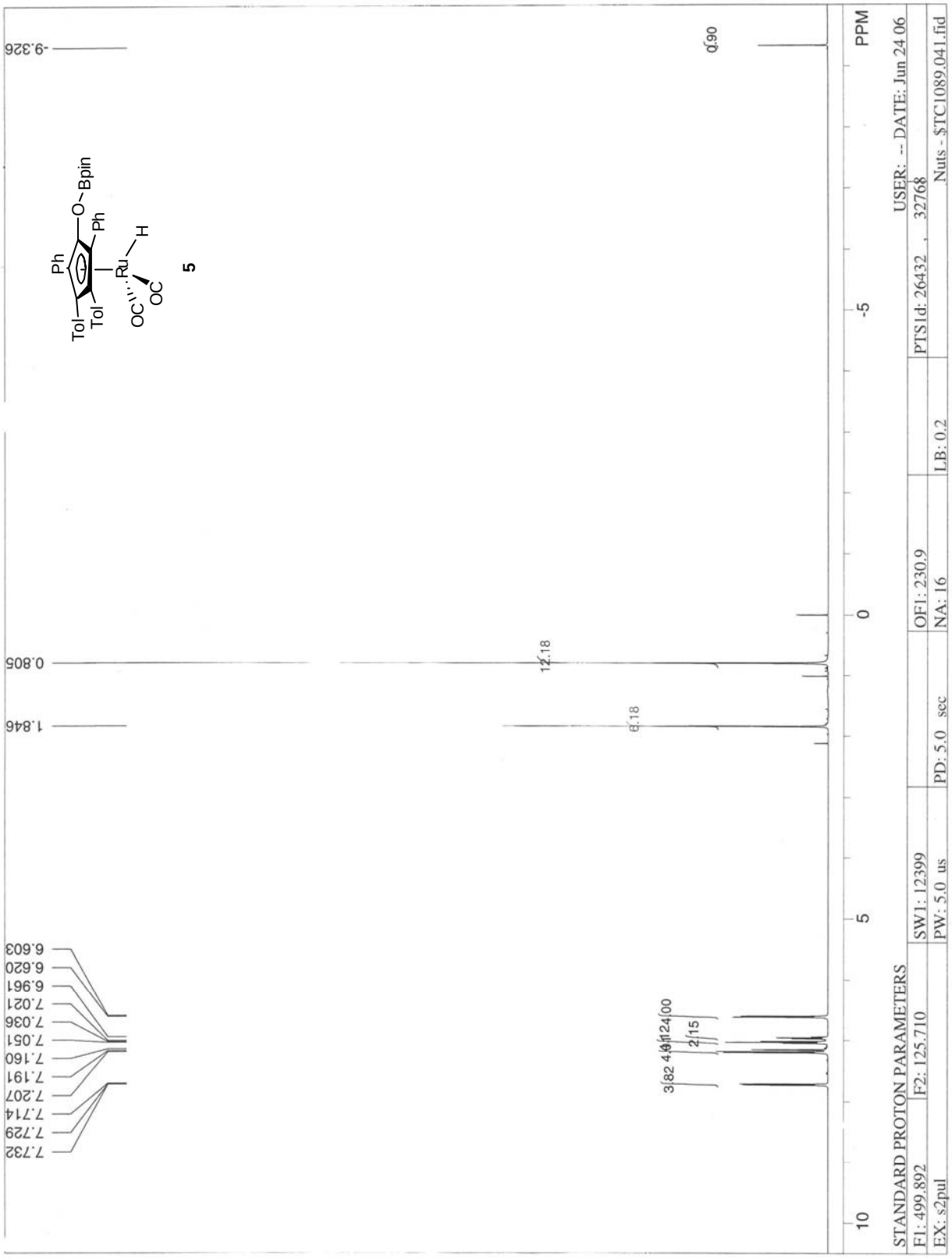
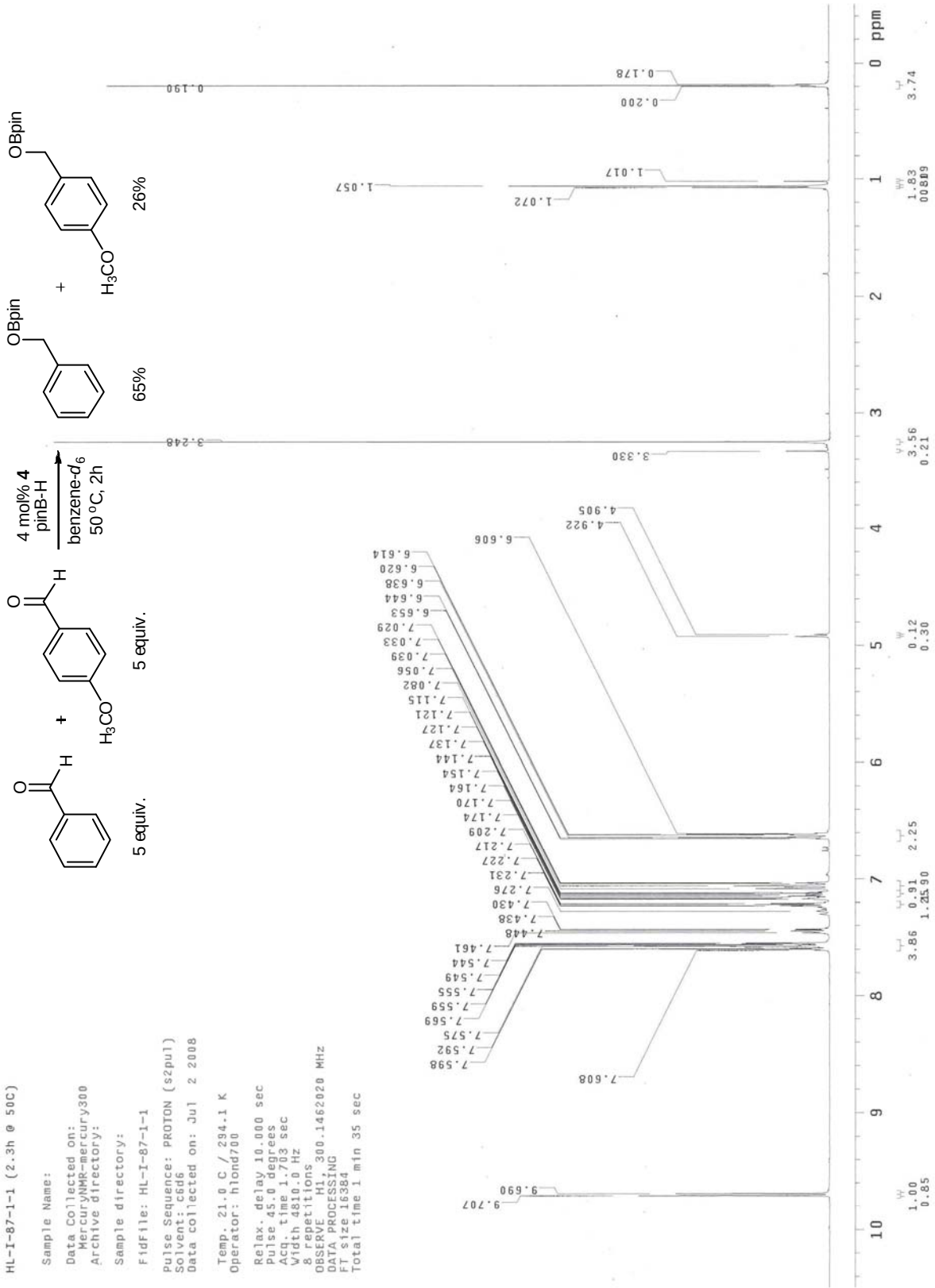
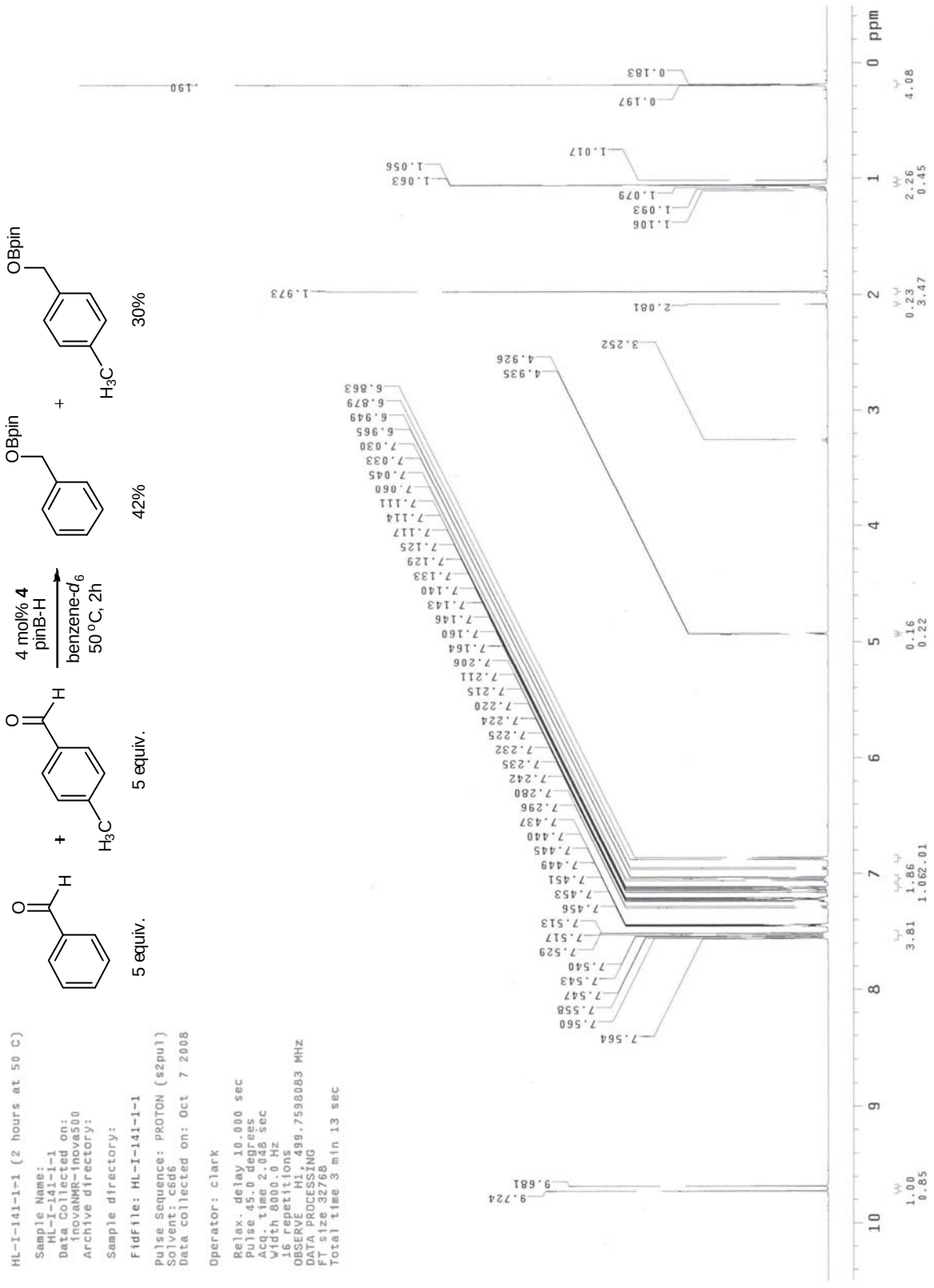
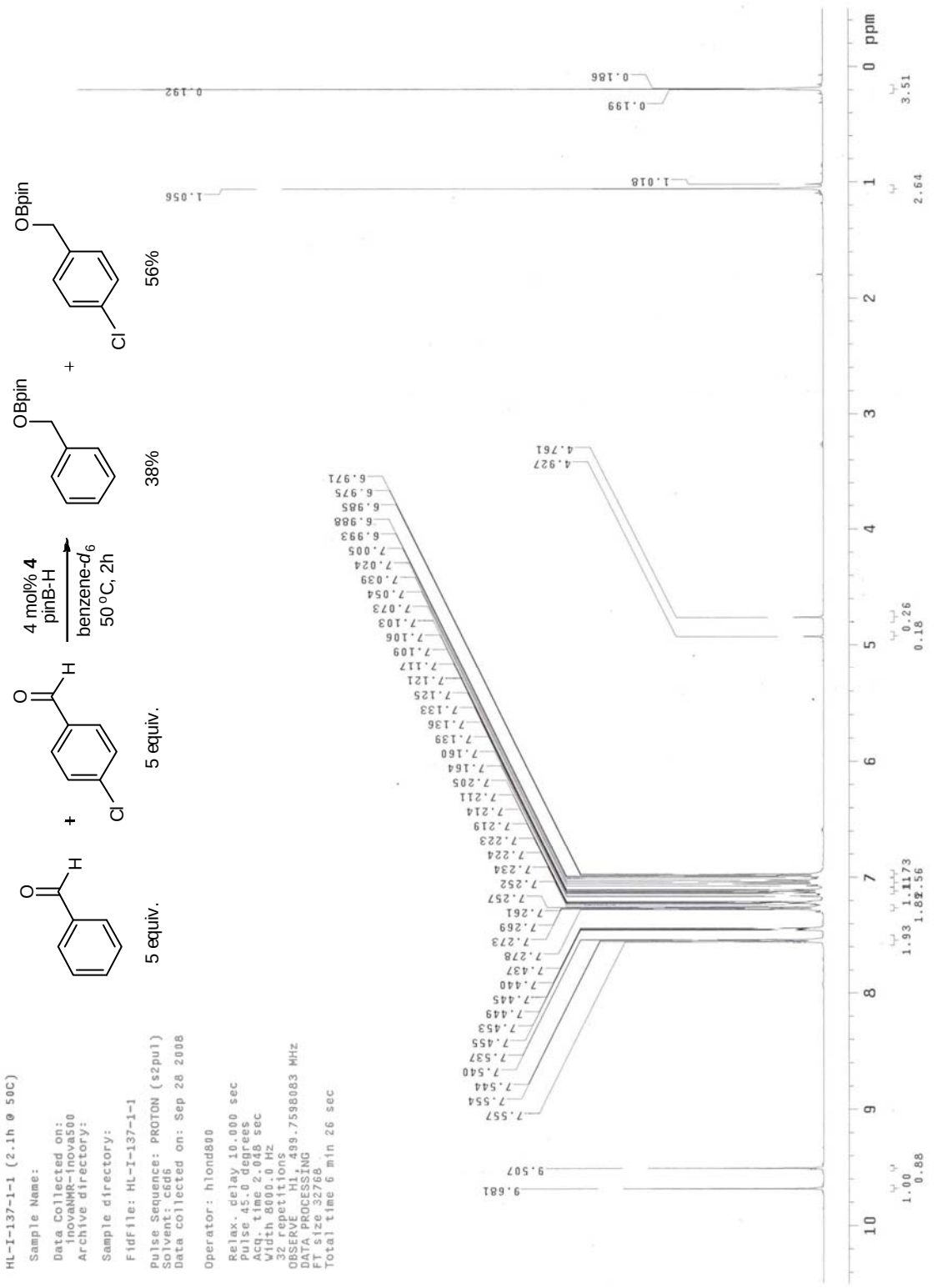
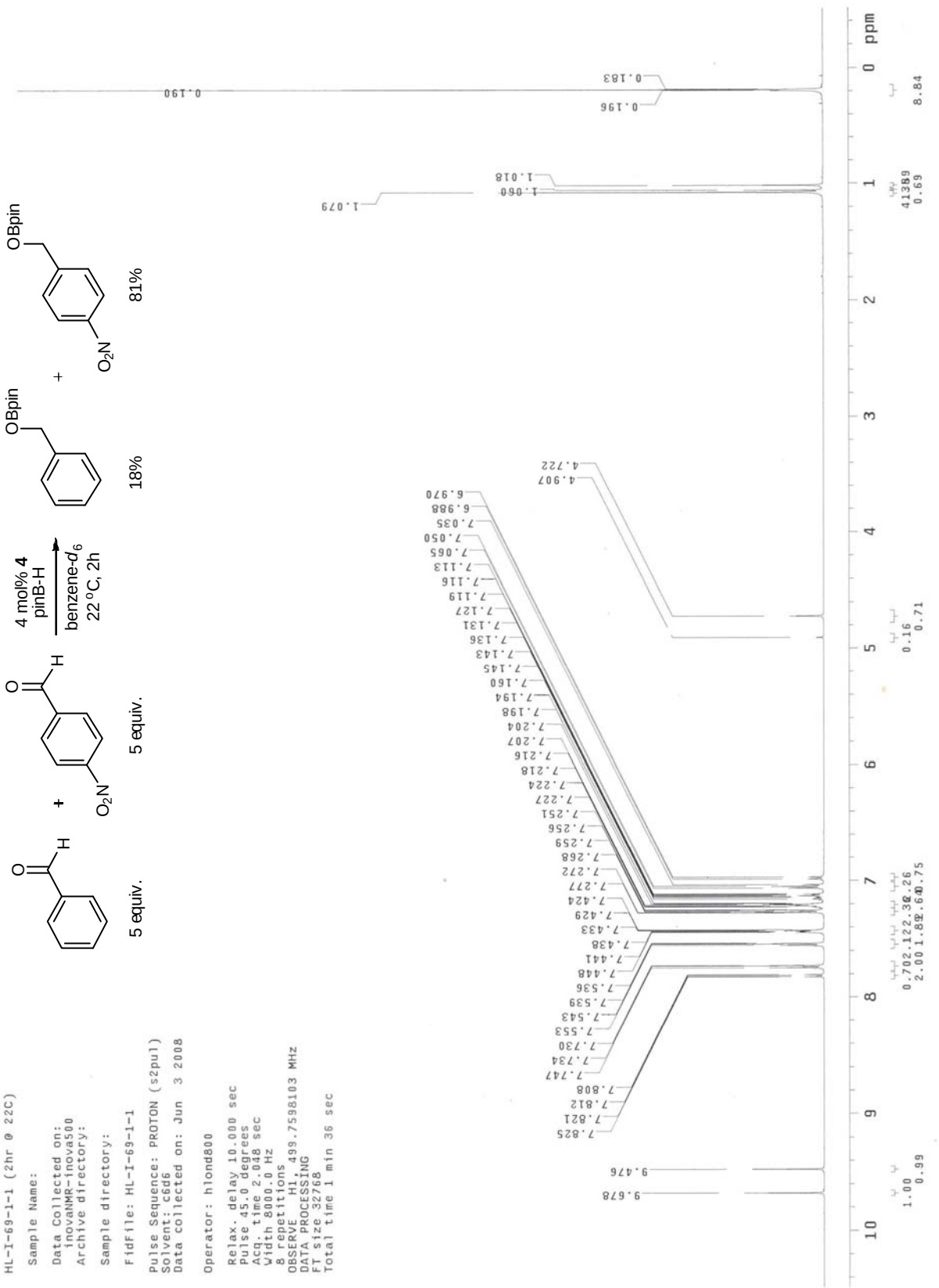

-9.326
0.90
1.846
0.805
12.18
6.18
3.82
4.00
2.15
7.732
7.729
7.714
7.207
7.191
7.160
7.051
7.036
7.021
6.961
6.620
6.603
10
5
0
-5
PPM
STANDARD PROTON PARAMETERS
F1: 499.892
F2: 125.710
SW1: 12399
OF1: 230.9
PTS1d: 26432
32768
EX: s2pul
PW: 5.0 us
PD: 5.0 sec
NA: 16
LB: 0.2
Nuts - $TC1089.041.fid
USER: -- DATE: Jun 24 06
Ph
Tol
Tol
Ru
H
OC
OC
O
Bpin
Ph
5


HL-I-87-1-1 (2.3h @ 50C)
Sample Name:
Data Collected on:
MercuryNMR-mercury300
Archive directory:
Sample directory:
FidFile: HL-I-87-1-1
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jul 2 2008
Temp. 21.0 C / 294.1 K
Operator: hlond700
Relax. delay 10.000 sec
Pulse 45.0 degrees
Acq. time 1.703 sec
Width 4810.0 Hz
8 repetitions
OBSERVE H1, 300.1462020 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 35 sec
5 equiv.
5 equiv.
4 mol% 4
pinB-H
benzene-d6
50 °C, 2h
65%
26%
OBpin
OBpin
H3CO
ppm


OBpin
H3C
30%
OBpin
42%
4 mol% 4
pinB-H
benzene-d6
50 °C, 2h
H3C
5 equiv.
5 equiv.
5 equiv.
HL-I-141-1-1 (2 hours at 50 C)
Sample Name:
HL-I-141-1-1
Data Collected on:
inovaNMR-inova500
Archive directory:
Sample directory:
FidFile: HL-I-141-1-1
Pulse Sequence: PROTON (s2pul)
Solvent: C6D6
Data collected on: Oct 7 2008
Operator: clark
Relax. delay 10.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1 499.7598083 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 13 sec
ppm


5 equiv.
5 equiv.
4 mol% 4
pinB-H
benzene-d_6
50 °C, 2h
38%
56%
OBpin
OBpin
Cl
HL-I-137-1-1 (2.1h @ 50C)
Sample Name:
Data Collected on:
inovaNMR-inova500
Archive directory:
Sample directory:
FidFile: HL-I-137-1-1
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Sep 28 2008
Operator: hlond800
Relax. delay 10.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7598083 MHz
DATA PROCESSING
FT size 32768
Total time 6 min 26 sec
ppm


5 equiv.
5 equiv.
4 mol% 4
pinB-H
benzene-d_6
22 °C, 2h
18%
81%
OBpin
OBpin
O_2N
O_2N
ppm