Supporting Information

Electrophilic Aromatic Substitution Reactions of 1,2-Dihydro-1,2-Azaborines

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Experimental Section

General Procedures. Manipulations of air-sensitive compounds were performed under nitrogen or argon atmosphere using standard Schlenk techniques or in a nitrogen-filled drybox. Pentane and hexanes were dried and deoxygenated by distillation from sodium/benzophenone ketyl. CH₂Cl₂ was dried and deoxygenated by distillation from CaH₂. Br₂ (Aldrich), anhydrous DMF (Aldrich), anhydrous CH₃CN (Acros), CuCN (Aldrich), ICl (Aldrich), *N,N*-dimethylmethyleniminium chloride (Fluka) were used without further purification. ¹H, ¹³C and ¹¹B NMR spectra were recorded on a Varian Inova 400 or 500 NMR spectrometer at ambient temperature. Chemical shifts are reported in parts per million (δ). Proton and carbon chemical shifts are relative to respective solvent internal standards shown below: benzene-*d*₆ δ 7.16(¹H), 128.39(¹³C); CD₂Cl₂ δ 5.32(¹H), 54.00(¹³C). The coupling constants (*J*) are reported in herz. The following abbreviations are used to describe peak patterns: br = broad, s = singlet, d =

doublet, t = triplet, q = quartet and m = multiplet. All ^{13}C and ^{11}B spectra were determined with complete proton decoupling. GC-Mass spectra were recorded on a Finnigan Trace GC/MS spectrometer. High-resolution mass spectra were recorded on a VG-250S spectrometer with an electron-impact at 70 eV. Elemental analyses were conducted on a Perkin-Elmer 240 CHN analyzer by the Analytical Service Department of the Chemistry Department at the University of Michigan, Ann Arbor.

Deuterations

1. 1-Ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (25mg, 0.14mmol) was dissolved in 1:3 w/w mixtures of $\text{CF}_3\text{COOD}/\text{CD}_3\text{COOD}$ (1.0g) at room temperature. In the ^1H NMR spectra, the integral of signal at $\delta = 6.72$ decreased slowly indicating the D-H exchange occurred at C(3)H position. On standing at room temperature for 24 hours colorless crystals formed. These were identified as phenylboronic anhydride by X-ray crystallography.⁹ Under the same condition, the NMR samples of furan (10mg, 0.15mmol) in 1:3 w/w mixtures of $\text{CF}_3\text{COOD}/\text{CD}_3\text{COOD}$ (1.0g) and thiophene (12mg, 0.14mmol) in 1:3 w/w mixtures of $\text{CF}_3\text{COOD}/\text{CD}_3\text{COOD}$ (1.0g) were prepared. On standing at room temperature for 48 hours, both of the ^1H NMR spectra remained unchanged suggesting no D-H exchange occurred.
2. 1-Ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (25mg, 0.14mmol) was dissolved in CD_3COOD (1mL) at room temperature. After leaving at room temperature for 7

days, the ^1H NMR spectra remained unchanged indicating no D-H exchange happened. Under the same condition, 1-methylindole (20mg, 0.15mmol) was dissolved in CD_3COOD (1ml). The proton signal of C(3)H disappeared completely within 30mins indicating rapid D-H exchange happened at the C(3)H position in 1-methylindole.

3. 2-Methylfuran (30mg, 0.37mmol) and 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (56mg, 0.31mmol) were dissolved in 1:3 w/w mixtures of $\text{CF}_3\text{COOD}/\text{CD}_3\text{COOD}$ (4.6g) at room temperature. After stirring at room temperature for 40 mins, the mixture was cooled to $0\text{ }^\circ\text{C}$ followed by quenching the reaction with 1M NaOH (50ml). The solution was extracted with pentane ($3 \times 10\text{mL}$). The organic solution was combined and dried over sodium sulfate. This solution was analyzed by GC-MS. The percentages of deuterium incorporation in 2-methylfuran (30% D-incorporation) and 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (42% D-incorporation) were determined by following the procedure described by Cooks¹⁴. The relative rates of deuterium incorporation were 1: 1.4 for 2-methylfuran and 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine.

(14) Lambert, J. B.; Shurvell, H. B.; Lightner, D. A. Cooks, R. G. In *Organic Structural Spectroscopy*; Challice, J., Ed.; Prentice-Hall: Upper Saddle River, 1998; pp 447-448.

3-Bromo-1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (2). Br₂ (208mg, 1.30mmol) in 6mL CH₂Cl₂ was added slowly to a solution of 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (224mg, 1.22mmol) in 6mL CH₂Cl₂ at 0 °C. The resulting solution was stirred at 0 °C for 10 mins and at room temperature for 30 mins. After removal all the volatiles in vacuo, the residue was extracted with 3 × 5mL pentane. Pentane was removed in vacuo to yield the product as a pale yellow oil which slowly crystallized on standing(292mg, 91%), mp = 22 °C. ¹H NMR (500 MHz, C₆D₆): δ 0.70 (t, *J* = 7.3 Hz, 3H, CH₃); 3.16 (q, *J* = 7.3 Hz, 2H, NCH₂); 5.86 (t, *J* = 7.0 Hz, 1H, C(5)H); 6.53 (d, *J* = 7.0 Hz, 1H, C(6)H); 7.22 (t, 1H, *J* = 7.4 Hz, ArH); 7.30 (t, 2H, *J* = 7.4, ArH); 7.44 (d, 2H, *J* = 7.4 Hz, ArH); 7.86 (d, 1H, *J* = 7.0 Hz, C(4)H). ¹³C NMR (100.6 MHz, CD₂Cl₂): δ 18.5, 50.2, 111.8, 128.1, 128.2, 132.8, 138.5, 144.8. Signal for C_{isop} and C(3) not observed. ¹¹B NMR (160.4 MHz, CD₂Cl₂): δ 35.3. HRMS (EI, *m/z*): calcd for C₁₂H₁₃¹¹BNBr (M⁺), 261.0324; found, 261.0330. Anal Calcd for C₁₂H₁₃BNBr: C, 55.02; H, 5.00; N, 5.35. Found: C, 54.77; H, 5.15; N, 4.98.

3-Cyano-1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (3). CuCN (185mg, 2.07mmol) was added to a solution of **2** (180mg, 0.69mmol) in 10mL anhydrous DMF at room temperature. The resulting solution was heated at 130 °C for 16 hours. After removal of

the solvent in vacuo, the residue was extracted with $4 \times 5\text{mL CH}_2\text{Cl}_2$. The CH_2Cl_2 was removed in vacuo and the residue was purified by column chromatography on silica gel (40% ethyl acetate in hexanes elution) to give a pure sample of the product (86mg, 60%) as a yellow oil. IR (liquid film): 2207 cm^{-1} . ^1H NMR (500 MHz, C_6D_6): δ 0.59 (t, $J = 7.3$ Hz, 3H, CH_3); 3.10 (q, $J = 7.3$ Hz, 2H, NCH_2); 5.78 (t, $J = 6.8$ Hz, 1H, C(5)H); 6.47 (d, $J = 6.8$ Hz, 1H, C(6)H); 7.21 (t, 1H, $J = 7.8$ Hz, ArH); 7.28 (t, 2H, $J = 7.8$, ArH); 7.44 (d, 2H, $J = 7.8$ Hz, ArH); 7.58 (d, 1H, $J = 6.8$ Hz, C(4)H). ^{13}C NMR (125.7 MHz, C_6D_6): δ 18.0, 49.3, 111.5, 128.7, 129.0, 133.1, 142.9, 150.3. Signal for C_{isop} and C(3) not observed. ^{11}B NMR (160.4 MHz, $\text{DMF-}d_7$): δ 36.9. HRMS (EI, m/z): calcd for $\text{C}_{13}\text{H}_{13}^{11}\text{BN}_2$ (M^+), 208.1172; found, 208.1171. Anal Calcd for $\text{C}_{13}\text{H}_{13}\text{BN}_2$: C, 75.04; H, 6.30; N, 13.46. Found: C, 74.88; H, 6.29; N, 13.44.

1-Ethyl-1,2-dihydro-3-hydroxy-2-phenyl-1,2-azaborine (4). 1-Ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (332mg, 1.81mmol) in 4mL CH_2Cl_2 was added slowly to a solution of ICl (885mg, 5.45 mmol) in 6mL CH_2Cl_2 at 0°C . The resulting solution was stirred at 0°C for 2 hours and at room temperature for 20 hours. After filtration, the residue was washed with ethyl ether ($2 \times 10\text{mL}$). The organic solutions were combined, washed with 20% $\text{Na}_2\text{S}_2\text{O}_3$ aqueous solution ($2 \times 30\text{mL}$) and dried over sodium sulfate. After removal of the volatiles in vacuo, the residue was extracted with pentane ($3 \times 10\text{mL}$). The pentane

solution was concentrated . Cooling the solution to -78 °C resulted in the formation of a dark red precipitate. After removal the precipitate by filtration, the pentane solution was allowed to warm to room temperature. Pentane was removed in vacuo to yield the product as a pale yellow oil (166mg, 46%). IR (liquid film): 3634 cm^{-1} . ^1H NMR (500 MHz, C_6D_6): δ 1.06 (t, J = 7.3 Hz, 3H, CH_3); 3.44 (q, J = 7.3 Hz, 2H, NCH_2); 4.06 (s, 1H, OH); 5.87 (t, J = 6.6 Hz, 1H, C(5)H); 6.54 (d, J = 6.6 Hz, 1H, C(6)H); 7.06 (t, 1H, J = 7.6 Hz, ArH); 7.18 (t, 2H, J = 7.6, ArH); 7.29 (d, 2H, J = 7.6 Hz, ArH); 7.38 (d, 1H, J = 6.6 Hz, C(4)H). ^{13}C NMR (125.7 MHz, CD_2Cl_2): δ 17.7, 45.3, 105.9, 126.5, 127.8, 129.6, 138.4, 142.2. Signal for C_{ispo} and C(3) not observed. ^{11}B NMR (160.4 MHz, CD_2Cl_2): δ 28.8. HRMS (EI, m/z): calcd for $\text{C}_{12}\text{H}_{14}^{11}\text{BNO}$ (M^+), 199.1168; found, 199.1170. Anal Calcd for $\text{C}_{12}\text{H}_{14}\text{BNO}$: C, 72.41; H, 7.09; N, 7.04. Found: C, 72.15; H, 7.25; N, 6.84.

5-Acetyl-1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (5). Acetic anhydride (644mg, 6.31mmol) and 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (200mg, 1.09mmol) were dissolved in 5mL CH_2Cl_2 at room temperature. To this was added a solution of SnCl_4 (200mg, 0.77mmol) in 3mL CH_2Cl_2 . The mixture was stirred at room temperature for 24 hours. After quenching the reaction by the addition of 5% NaHCO_3 aqueous solution (20mL), the mixture was extracted with ethyl ether ($3 \times 10\text{mL}$). The combined organic layer was dried over sodium sulfate. After removal of all the volatiles in vacuo, the residue was extracted with pentane ($3 \times 10\text{mL}$). The solution was concentrated and the

product was precipitated at -78 °C. The product was obtained after repeated precipitation at -78 °C in pentane/ether as a pale yellow powder (25mg, 10%). IR (cyclohexane film): 1681 cm⁻¹. ¹H NMR (500 MHz, C₆D₆): δ 0.74 (t, *J* = 7.3 Hz, 3H, CH₃); 2.12 (s, 3H, COCH₃); 3.31 (q, *J* = 7.3 Hz, 2H, NCH₂); 6.95 (d, *J* = 11.2 Hz, 1H, C(3)H); 7.25 (t, 1H, *J* = 7.3 Hz, ArH); 7.30 (t, 2H, *J* = 7.3, ArH); 7.49 (d, 2H, *J* = 7.3 Hz, ArH); 7.92 (s, 1H, C(6)H); 8.16 (d, 1H, *J* = 11.2 Hz, C(4)H). ¹³C NMR (100.6 MHz, CD₂Cl₂): δ 18.7, 26.7, 49.7, 128.3, 128.5, 133.1, 141.9, 144.8. Signal for C_{ispo}, C(3) and C(5) not observed. ¹¹B NMR (160.4 MHz, C₆D₆): δ 37.7. HRMS (EI, *m/z*): calcd for C₁₄H₁₆¹¹BNO (M⁺), 225.1325; found, 225.1330.

1-Ethyl-1,2-dihydro-5-dimethylaminomethyl-2-phenyl-1,2-azaborine (6). *N,N*-Dimethylmethyleniminium chloride (154mg, 1.65mmol) was mixed with 1-ethyl-1,2-dihydro-2-phenyl-1,2-azaborine (273mg, 1.49mmol) in 10 mL CH₃CN at room temperature. After refluxing for 2.5 hours, the mixture was worked up with 50 mL NaHCO₃ aqueous solution (5%) and extracted with ethyl ether (3 × 30mL). The combined organic layer was washed with brine and dried over sodium sulfate. After removal the solvent in vacuo, the residue was extracted with pentane (3 × 10mL). The solution was concentrated. Cooling the solution to -78 °C resulted in the formation of a precipitate. After removal the precipitate by filtration, the pentane solution was allowed to warm to room temperature. Pentane was removed in vacuo to yield the product as a pale yellow oil (216mg, 60%). ¹H NMR (500 MHz, C₆D₆): δ 0.93 (t, *J* = 7.3 Hz, 3H,

CH₃); 2.14 (s, 6H, N(CH₃)₂); 3.15 (s, 2H, CH₂NMe₂); 3.49 (q, $J = 7.3$ Hz, 2H, NCH₂); 6.96 (s, 1H, C(6)H); 7.11 (d, 1H, $J = 11.0$ Hz, C(3)H); 7.25 (t, 1H, $J = 7.3$ Hz, ArH); 7.33 (t, 2H, $J = 7.3$, ArH); 7.61 (d, 2H, $J = 7.3$ Hz, ArH); 7.81 (d, 1H, $J = 11.0$ Hz, C(4)H). ¹³C NMR (125.7 MHz, CD₂Cl₂): δ 18.8, 45.4, 48.7, 62.9, 127.8, 128.1, 131.5 (br), 133.3, 138.5, 145.5. Signal for C_{ispo}, and C(5) not observed. ¹¹B NMR (160.4 MHz, CD₂Cl₂): δ 35.1. HRMS (EI, m/z): calcd for C₁₅H₂₁¹¹BN₂ (M⁺), 240.1798; found, 240.1806. Anal Calcd for C₁₅H₂₁BN₂: C, 75.02; H, 8.81; N, 11.66. Found: C, 75.27; H, 8.85; N, 11.33.

N-Et-B-Ph-C-Br-pure-Benzene

expl Proton

SAMPLE SPECIAL
 date Jan 4 2006 temp not used
 solvent C6D6 gain not used
 file /export/home/~ spin 20
 Inova.500/wuqian/v~ hst 0.008
 nmrsys/data/N-Et-B~ pw90 5.500
 -Ph-C-Br-pure-Benz~ alfa 6.600

ACQUISITION
 sw 8997.9 in n
 at 3.000 dp nw
 np 53986 hs y
 fb 5000 nn
 bs 8 lb 0.20
 ss 2 fn not used
 d1 0.500
 nt 10000
 ct 64

PROCESSING
 sp -10.4
 wp 4510.1
 rf1 4564.6
 rfp 3579.3
 rp 159.1
 lp -4.8

TRANSMITTER
 tn H1
 sfrq 499.908
 tof 999.8
 tpwr 56
 pw 2.750
 wc 250
 sc 0
 vs 15
 th 6
 ai cdc ph
 dn C13
 dof 1285.0
 dm nnn
 dmm p
 dpwr 41
 dmf 32258

INDEX	FREQUENCY	PPM	HEIGHT
1	3935.088	7.872	7.3
2	3927.948	7.857	7.3
3	3723.919	7.449	11.8
4	3722.546	7.447	13.8
5	3715.956	7.433	16.4
6	3655.544	7.312	10.3
7	3648.129	7.298	21.0
8	3640.715	7.283	11.7
9	3616.001	7.233	6.7
10	3608.587	7.219	9.2
11	3579.205	7.160	7.5
12	3267.807	6.537	10.7
13	3260.942	6.523	11.0
14	2934.166	5.869	9.0
15	2927.026	5.855	14.6
16	2920.161	5.841	8.1
17	1593.287	3.187	9.2
18	1586.148	3.173	29.1
19	1579.008	3.159	29.2
20	1571.594	3.144	9.6
21	355.384	0.711	31.7
22	347.970	0.696	63.0
23	340.831	0.682	31.2

N-Et-B-Ph-C-Br-pure-CD2C12-C13

exp1 Carbon

SAMPLE
date Jul 25 2006 temp not used
solvent CD2C12 gain 30
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Inova.400/wuqian/v~ hst 0.008
nmrsys/data/N-Et-B~ pw90 15.700
-Ph-C-Br-pure-CD2C~ alfa 10.000
12-C13.fid

ACQUISITION
sw 20115.7 in n
at 1.300 dp y
np 52320 hs nn
fb 11000
bs 32 lb 1.00
ss 2 fn not used
dl 0.100
nt 100000 sp
ct 4704 wp 20115.7

TRANSMITTER
tn C13 rfl 6372.9
sfrq 100.581 rfp 5430.9
tof -480.0 rp -105.7
tpwr 51 lp -146.6

DECOUPLER
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dof 0 sc 0
dm yyy vs 20000
dmm w th 4
dpwr 38
dmf 9600

PROCESSING
DISPLAY -942.0
PLOT 250

FLAGS
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in n
dp y
hs nn
lb 1.00
fn not used
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rfl 6372.9
rfp 5430.9
rp -105.7
lp -146.6
wc 250
sc 0
vs 20000
th 4
ai cdc ph

INDEX	FREQUENCY	PPM	HEIGHT
1	14563.234	144.805	23.3
2	13926.619	138.475	14.6
3	13356.920	132.810	38.7
4	12889.128	128.159	23.4
5	12882.989	128.098	36.6
6	11241.420	111.776	14.6
7	5485.493	54.543	7.4
8	5458.481	54.275	14.5
9	5430.856	54.000	21.9
10	5403.844	53.731	15.4
11	5376.832	53.463	7.6
12	5049.010	50.203	15.5
13	1863.482	18.529	20.3

N-Et-B-Ph-C-CN-pure-C6D6

exp1 Proton

SAMPLE SPECIAL
date Apr 13 2006 temp not used
solvent C6D6 gain not used
file /export/home/~ spin 20
Inova.500/wuqian/v~ hst 0.008
nmrsys/data/N-Et-B~ pw90 6.300
-Ph-C-CN-pure-C6D6~ alfa 6.600

ACQUISITION il n
sw 7998.4 in nw
at 3.000 dp y
np 47990 hs nn

PROCESSING
fb 4000
bs 8 lb 0.20
ss 2 fn not used

DISPLAY
d1 0.500
nt 10000 sp -6.6
ct 80 wp 4505.1

TRANSMITTER H1
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tof 499.9 rfp 3579.3
tpwr 56 rp 167.8
pw 3.150 lp 2.4

PLOT
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dm nnn vs 25
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dpwr 44 ai cdc ph
dmf 15385

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2	3794.527	7.591	4.2
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4	3787.448	7.576	4.1
5	3726.667	7.455	8.6
6	3725.203	7.452	10.2
7	3718.368	7.438	11.4
8	3717.392	7.436	8.8
9	3645.871	7.293	4.9
10	3644.894	7.291	7.0
11	3637.815	7.277	13.5
12	3631.469	7.264	4.4
13	3630.248	7.262	7.5
14	3610.964	7.223	5.1
15	3603.397	7.208	6.2
16	3579.231	7.160	19.0
17	3240.910	6.483	6.7
18	3239.689	6.481	6.7
19	3234.075	6.469	6.9
20	3233.099	6.467	6.8
21	2896.486	5.794	6.9
22	2889.651	5.780	12.0
23	2882.816	5.767	6.2
24	1560.775	3.122	7.2
25	1553.452	3.107	22.1
26	1546.129	3.093	21.9
27	1539.050	3.079	7.3
28	301.711	0.604	25.0
29	294.388	0.589	51.2
30	287.065	0.574	23.9

N-Et-B-Ph-C-CN-AfterTLC-C6D6-2days-C13

expl Carbon

SAMPLE SPECIAL
 date Apr 8 2006 temp not used
 solvent C6D6 gain 50
 file /export/home/~ spin 20
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 -Ph-C-CN-AfterTLC-~ alfa 10.000
 C6D6-2days-C13.fid

ACQUISITION il n
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 at 1.300 dp y
 np 65392 hs nn
 fb 14000
 bs 16 lb
 dl 0.100 fn not used
 nt 100000
 ct 7712

TRANSMITTER sp
 tn C13 wp 25141.4
 sfrq 125.712 rfl 17306.7
 tof -599.9 rfp 16138.7
 tpwr 59 rp 148.6
 pw 6.250 lp -165.4

DECOUPLER wc 250
 dn H1 sc 0
 dof 500.0 vs 2500
 dm yyy th 5
 dmm w ai cdc ph
 dpwr 31
 dmf 12200

FLAGS

PROCESSING

not used

DISPLAY

PLOT

cdc ph

INDEX	FREQUENCY	PPM	HEIGHT
1	18895.577	150.322	7.5
2	17968.704	142.948	5.0
3	16730.316	133.096	15.7
4	16220.076	129.037	9.5
5	16174.807	128.677	8.9
6	16162.530	128.579	100.2
7	16138.744	128.390	100.8
8	16114.192	128.195	105.6
9	14018.754	111.525	6.1
10	6190.976	49.252	7.9
11	2264.811	18.017	8.6

N-Et-B-PhC-OH-C6D6-pure

exp1 Proton

SAMPLE
date Jul 31 2006 temp not used
solvent C6D6 gain not used
file /export/home/~ spin 20
Inova.500/wuqian/v~ hst 0.008
nmrsys/data/N-Et-B~ pw90 5.900
-PhC-OH-C6D6-pure~ alfa 6.600

ACQUISITION
sw 7998.4 in n
at 3.000 dp nw
np 47990 hs y
fb 4000 nn

PROCESSING
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dl 0.500
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ct 72 wp -11.6

TRANSMITTER
tn H1 rfp 3579.3
sfrq 499.907 rp 150.2
tof 499.9 lp 5.6

PLOT
pw 2.950 wc 250
dn C13 sc 0
dof 499.9 vs 20
dm nnn th 5
dmm c
dpwr 41
dmf 32258

DECOUPLER
ai cdc ph

INDEX	FREQUENCY	PPM	HEIGHT
1	3696.518	7.394	5.8
2	3695.542	7.392	6.0
3	3690.171	7.382	6.1
4	3688.951	7.379	5.9
5	3648.919	7.299	16.3
6	3647.698	7.297	19.8
7	3645.745	7.293	6.1
8	3642.572	7.287	7.3
9	3641.596	7.285	12.7
10	3640.619	7.283	22.1
11	3639.643	7.281	20.6
12	3596.681	7.195	14.3
13	3594.729	7.191	5.2
14	3590.579	7.183	6.6
15	3589.114	7.180	23.9
16	3587.650	7.177	8.5
17	3583.012	7.167	6.6
18	3581.303	7.164	15.5
19	3579.594	7.161	6.5
20	3578.862	7.159	6.0
21	3539.074	7.080	8.4
22	3531.507	7.064	11.4
23	3524.184	7.050	5.1
24	3271.541	6.544	6.9
25	3270.321	6.542	6.9
26	3264.706	6.531	7.2
27	3263.730	6.529	7.0
28	2943.228	5.888	13.7
29	2936.637	5.874	24.0
30	2929.802	5.861	12.7
31	2029.808	4.060	29.4
32	1731.519	3.464	10.9
33	1724.196	3.449	31.9
34	1717.117	3.435	33.3
35	1709.794	3.420	11.0
36	535.433	1.071	38.4
37	528.354	1.057	79.3
38	521.031	1.042	38.9

N-Et-B-PhC-OH-CD2C12-pure-C13

exp1 Carbon

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SAMPLE          SPECIAL
date Jul 31 2006 temp not used
solvent CD2C12 gain 50
file /export/home/~ spin 20
Inova.500/wuqian/v~ hst 0.008
nmr sys/data/N-Et-B~ pw90 14.500
-PhC-OH-CD2C12-pur~ alfa 10.000
e-C13.fid
ACQUISITION il n
sw 25141.4 in n
at 1.300 dp y
np 65392 hs nn
fb 14000
bs 16 lb 1.00
dl 0.100 fn not used
nt 100000 DISPLAY
ct 2368 sp -1182.6
TRANSMITTER wp 25141.4
tn C13 rfl 7970.5
sfrq 125.713 rfp 6787.9
tof -599.9 rp 58.2
tpwr 57 lp -170.0
pw 7.250 PLOT
DECOUPLER wc 250
dn H1 sc 0
dof 500.0 vs 700
dm yyy th 4
dmm w ai cdc ph
dpwr 31
dmf 11500

```

INDEX	FREQUENCY	PPM	HEIGHT
1	17873.507	142.190	13.3
2	17399.328	138.418	9.2
3	16292.145	129.610	23.9
4	16068.100	127.828	23.6
5	15898.531	126.479	15.0
6	13312.802	105.908	6.0
7	6787.864	54.000	4.6
8	5699.096	45.338	10.8
9	2218.720	17.651	12.6

N-Et-B-Ph-C-COMe-pure-C6D6

expt1 Proton

SAMPLE
date Jun 13 2006 temp not used
solvent C6D6 gain not used
file /export/home/~ spin 20
Inova.500/wuqian/v hst 0.008
nmr/sys/data/N-Et-B- pw90 6.300
-Ph-C-COMe-pure-C6- alfa 6.600
D6.fid

ACQUISITION
sw 7998.4 il n
at 3.000 in nw
np 47990 dp y
fb 4000 hs nn
bs 8 lb 0.20
ss 2 fn not used
d1 0.500
nt 10000
ct 80

TRANSMITTER
tn H1
sfrq 499.907
tof 499.9
tpwr 56
pw 3.150 wc 250
DECOUPLER C13 sc 0
dn C13 vs 20
dof 499.9 th 3
dm nnn ai cdc ph
dmm c
dpwr 44
dmf 15385

SPECIAL
FLAGS
PROCESSING
DISPLAY
PLOT

INDEX	FREQUENCY	PPM	HEIGHT
1	4084.273	8.170	3.7
2	4082.564	8.167	3.9
3	4072.800	8.147	3.8
4	4071.336	8.144	3.9
5	3959.294	7.920	8.2
6	3957.829	7.917	8.1
7	3749.857	7.501	7.2
8	3748.392	7.498	8.9
9	3741.557	7.485	9.8
10	3740.337	7.482	7.4
11	3658.808	7.319	3.9
12	3657.587	7.317	6.1
13	3650.753	7.303	10.8
14	3649.288	7.300	6.3
15	3643.185	7.288	6.8
16	3629.028	7.259	4.3
17	3621.705	7.245	4.9
18	3579.231	7.160	22.9
19	3483.545	6.968	8.3
20	3472.316	6.946	7.8
21	1664.761	3.330	4.8
22	1657.438	3.316	15.0
23	1650.115	3.301	15.0
24	1643.036	3.287	4.9
25	1062.080	2.125	75.0
26	377.626	0.755	16.2
27	370.303	0.741	33.4
28	363.224	0.727	15.8

N-Et-B-Ph-C-COMe-pure-CD2C12-C13-2days

exp1 Carbon

SAMPLE SPECIAL
date Jun 15 2006 temp not used
solvent CD2C12 gain 30
file /export/home/~ spin 20
Inova.400/wuqian/v~ hst 0.008
nmrsys/data/N-Et-B~ pw90 15.700
-Ph-C-COMe-pure-CD~ alfa 10.000
2C12-C13-2days.fid
ACQUISITION
sw 20115.7 in n
at 1.300 dp y
np 52320 hs nn
fb 11000
bs 32 lb PROCESSING 1.00
ss 2 fn not used
d1 0.100
nt 100000 sp DISPLAY -942.7
ct 26496 wp 20115.7
rfl 6373.5
tn C13 rfp 5430.9
sfrq 100.581 rp 80.6
tof -480.0 lp -106.8
tpwr 51
pw 7.850 wc PLOT 250
DECOUPLER H1 sc 0
dn dof 0 vs 122946
dm yyy ai cdc ph 4
dmm w
dpwr 38
dmf 9600

INDEX	FREQUENCY	PPM	HEIGHT
1	14563.234	144.805	6.9
2	14267.334	141.863	8.4
3	13387.001	133.109	16.2
4	12924.120	128.507	10.8
5	12903.248	128.299	14.6
6	5485.493	54.543	47.7
7	5457.867	54.269	99.0
8	5430.856	54.000	151.2
9	5403.844	53.731	98.0
10	5376.218	53.457	49.6
11	4994.987	49.666	6.8
12	2680.583	26.654	5.2
13	1878.830	18.682	8.3

N-Et-B-Ph-C-CH2NMe2-C6D6-pure

expl Proton

SAMPLE SPECIAL
date Jul 24 2006 temp not used
solvent C6D6 gain not used
file /export/home/~ spin 20
Inova.500/wuqian/v~ hst 0.008
nmrsys/data/N-Et-B- pw90 5.900
-Ph-C-CH2NMe2-C6D6~ alfa 6.600
-pure.fid

ACQUISITION
sw 7998.4 il n
at 3.000 in nw
np 47990 dp y
fb 4000 hs nn

PROCESSING
bs 8 lb 0.20
ss 2 fn not used
d1 0.500
nt 100000 sp
ct 80 wp 4498.5
rf1 4565.2

TRANSMITTER
tn H1 rfp 3579.3
sfrq 499.907 rp 145.4
tof 499.9 lp 4.8

PLOT
tpwr 57
pw 2.950 wc 250
sc 0

DECOUPLER
dn C13 vs 14
dof 499.9 th 3
dm nnn ai cdc ph
dmm c
dpwr 41
dmf 32258

INDEX	FREQUENCY	PPM	HEIGHT
1	3910.718	7.823	3.7
2	3909.254	7.820	3.6
3	3899.734	7.801	3.8
4	3898.025	7.798	3.7
5	3810.149	7.622	8.5
6	3808.685	7.619	10.7
7	3801.850	7.605	10.3
8	3800.630	7.603	8.1
9	3670.281	7.342	4.5
10	3669.304	7.340	7.1
11	3663.446	7.328	3.9
12	3662.225	7.326	13.2
13	3655.879	7.313	3.9
14	3654.902	7.311	7.1
15	3630.736	7.263	4.9
16	3623.169	7.248	5.6
17	3579.231	7.160	7.2
18	3577.279	7.156	4.4
19	3561.412	7.124	8.6
20	3550.428	7.102	8.1
21	3477.930	6.957	7.7
22	3477.198	6.956	7.7
23	1756.542	3.514	5.3
24	1749.219	3.499	16.0
25	1742.140	3.485	16.7
26	1734.818	3.470	5.3
27	1576.153	3.153	30.7
28	1070.135	2.141	151.2
29	691.781	1.384	4.4
30	471.116	0.942	18.9
31	464.037	0.928	41.2
32	456.714	0.914	19.1

N-Et-B-Ph-C-CH₂NMe₂-CD₂C12-C13

exp1 Carbon

```

SAMPLE              SPECIAL
date  Jul 23 2006   temp    not used
solvent  CD2C12     gain      50
file  /export/home/~ spin      20
Inova.500/wuqlan/v~ hst      0.008
nmrsys/data/N-Et-B~ pw90     15.000
-Ph-C-CH2NMe2-CD2C~ alfa     10.000
12-C13.fid
ACQUISITION         il      n
sw      25141.4      in      n
at      1.300      dp      y
np      65392      hs      nn
fb      14000
ls      16
d1      0.100      fn      not used
nt      100000
ct      6080
TRANSMITTER
tn      C13
sfrq    125.713      rfp      6787.9
tof     -600.0      rp      71.3
tpwr     57      lp      -182.4
pw      7.500
DECOUPLER
dn      H1
dof     500.0
dm      yyy
dmm      w
dpwr     31
dmf     12200
PROCESSING
1b      1.00
fn      not used
DISPLAY
sp      -1184.2
wp      25141.4
rfl      7972.0
rfp      6787.9
rp      71.3
lp      -182.4
PLOT
wc      250
sc      0
vs      3500
th      5
ai      cdc ph

```

INDEX	FREQUENCY	PPM	HEIGHT
1	18287.070	145.480	18.4
2	17411.605	138.516	5.8
3	16760.953	133.340	33.3
4	16101.860	128.096	28.9
5	16060.427	127.767	20.5
6	7901.953	62.863	13.1
7	6842.341	54.433	7.1
8	6815.486	54.220	14.9
9	6787.864	54.000	20.9
10	6761.010	53.786	15.4
11	6734.155	53.573	7.2
12	6124.936	48.726	16.8
13	5701.398	45.357	21.5
14	2367.572	18.835	19.3