Supporting Information

Aminoboranes as "Compatible" Iminium Ion Generators in Aminative C-C Bond Formations

Michinori Suginome,* Lars Uehlin, and Masahiro Murakami*

Department of Synthetic Chemistry and Biological Chemistry, Graduate School of Engineering, Kyoto University, Katsura, Nishikyo-ku, Kyoto 615-8510, Japan

Contents

General

All reactions were performed in a drybox or using Schlenk technique under a nitrogen atmosphere with magnetic stirring. ^{1}H NMR spectra were recorded on a Varian Mercury-400 (400 MHz) or Varian GEMINI-2000 (300 MHz) spectrometer using $CDCl_3$ or C_6D_6 as a solvent. ^{13}C NMR spectra were recorded on a Varian GEMINI-2000 spectrometer at 75 MHz with $CDCl_3$ as solvent. Chemical shifts of the ^{13}C NMR spectra were recorded relative to $CDCl_3$ (77.0 ppm). ^{11}B NMR spectra were recorded on a Varian GEMINI-2000 spectrometer at 1288 MHz with C_6D_6 as solvent. Chemical shifts of the ^{11}B NMR spectra were recorded relative to BF_3•Et_2O (0 ppm). High resolution mass (FAB) spectra were recorded on a JEOL JMS-700 spectrometer.

Anhydrous solvents were purchased from Kanto Chemical Co., aldehydes and ketones dried over CaH_2 prior to distillation. Bis(diamino)chloroboranes were synthesized

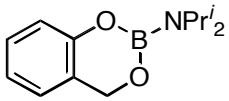
according to the literature.[1]

Preparation of amino boranes

***N,N-bis*(diethylamino)(isopropoxy)borane (2)**

n-Butyllithium (1.6 M of in hexane, 84 mmol) was added at 0 °C to diisopropylamine (12 mL, 84 mmol) in THF (100 mL). After stirring for 15 min, the reaction was cooled to –78 °C and isopropanol (6.4 mL, 84 mmol) was added. Stirring was continued for 30 min, then bis(diethylamino)chloroborane (12 g, 84 mmol) was added and the mixture allowed to warm up to room temperature. The solvent was removed in vacuo, the residue extracted with pentane (100 mL) and the desired compound purified by distillation (30-35 °C, 0.5 mbar). Yield: 15.3 g (83 %) colorless oil. ^{1}H NMR (C_6D_6) δ 0.97 (t, J = 7.6 Hz, 12H), 1.14 (d, J = 8.4 Hz, 6H), 2.90 (q, J = 7.6 Hz, 8H), 4.08 (h, J = 8.0 Hz, 1H); ^{13}C NMR (C_6D_6) δ 15.1 (4C), 25.3 (2C), 40.3 (4C), 65.3. ^{11}B NMR (C_6D_6) δ 24.89.

***N,N*-diisopropyl-4H-benzo[d][1,3,2]dioxaborinin-2-amine (6)**



Saligenin (1.45 g, 11.7 mmol) was dried by azeotropic distillation of toluene (15 ml). Cyclohexane (20 mL) was added, the mixture cooled in an ice bath and chlorobis(diisopropylamino)borane (2.88g, 11.7 mmol) was added. The mixture was allowed to warm up to room temperature and subsequently heated to 120 °C overnight. Removal of the formed lithium chloride by filtration followed by evaporation of the solvent in vacuo and Kugelrohr distillation of the remaining oil (90°C, 1 mbar) yielded 2.0 g of colorless product (73 %). ^{1}H NMR (C_6D_6) δ 1.16 (d, J = 8.8 Hz, 6H), 3.58 (h, J = 8.0 Hz, 2H), 4.60 (s, 2H), 6.47 (dd, J = 8.8 Hz, J = 2.0 Hz, 1H), 6.68 (dt, J = 8.8 Hz, J = 2.4 Hz, 1H), 6.90 (m, 2H) 7.77 (dd, J = 7.2 Hz, J = 1.2 Hz, 2H); ^{13}C NMR (C_6D_6)δ 23.1 (4C), 44.1 *(2C)*, 62.6, 117.5, 121.6, 123.8, 124.7, 128.4, 15.2; ^{11}B NMR (C_6D_6) δ 19.84.

Three-Component Mannich Reactions Using Aldehyde, Silyl Ketene Acetal, and Amino Boranes

General procedure A Including Extractive Workup with Acid-Treatment

---

[1]Chavant, P. Y.; Vaultier M.; *J. Organomet. Chem.* **1993**; 455; 37-46; Gerrard, W.; Lappert, M. F.; Pearce, C.A. *J.Chem Soc.* **1957**; 381-386

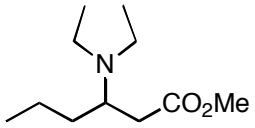
To a solution of aminoborane (**1**—**6**, 0.125 mmol) in *N*-methylpyrrolidinone (0.25 mL) were added 2-piperidinone (10% w/w solution in NMP, 25 mg, 0.025 mmol), aldehyde (0.25 mmol), and silyl ketene acetal (0.125 mmol) at room temperature with stirring. The mixture was stirred at room temperature for 2 h. To the reaction mixture were added ice water and *tert*-butyl methyl ether (15 mL) with stirring. Basic material was extracted from the organic phase three times with 5 ml 0.5 N hydrochloric acid. The combined acid layers were kept at 0 °C, washed with 10 ml *tert*-butyl methyl ether and the pH brought to 8 by addition of conc. ammonia solution. Organic materials were extracted with *tert*-butyl methyl ether three times and the combined organic layer was washed with 10 ml water. Evaporation left the crude products that showed purities of > 90% and were purified by column chromatography on silica gel (eluent: ethyl acetate/hexane). The reaction scale could be increased at least to a 4.4 mmol scale for the reaction shown as entry 5 in Table 2.

General procedure B: Non-Acidic Workup

Reactions were performed according to the procedure same as the general procedure A shown above. The reaction mixture was then diluted with ether (15 mL) and washed with ice-water three times. The organic layer was dried over K_2CO_3 and evaporated under vacuum. The crude material was purified by chromatography on silica gel using ethyl acetate/hexane mixture as an eluent.

The product shown as entries 1-6 in Table 1 and entry 1 in Table3 has been reported in the literature. (e.g., Saidi et al. *J. Chem. Soc. Perkin Trans. 1* **1997**, 1983.) CAS registry No.: 193820-05-2.

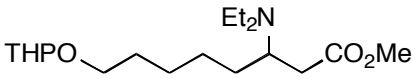
Methyl 3-(diethylamino)-hexanoate (Table 2, entry 1)



General Procedure A. Yield (0.125 mmol scale): 24 mg (96%). ^{1}H NMR ($CDCl_3$) δ 0,86 (t, J = 6.9 Hz, 3H), 0.96 (t, J = 6.9 Hz, 6H), 1.12-1.50 (m, 4H), 2.14-2.21 (m, 1H), 2.25-2.48 (m, 5H), 3.10 (quintet, J = 6.9 Hz, 1H), 3.62 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 14.1, 14.6, 20.1,

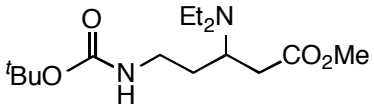
33.3, 36.0, 43.3, 51.4, 56.6, 173.7; IR (neat) 2965, 1740 cm^{-1}. Anal. Calcd. for $C_{11}H_{23}NO_2$: C, 65.63; H, 11.52; N, 6.96. Found: C, 65.54; H, 11.34; N, 6.76.

Methyl 3-(diethylamino)-8-(tetrahydro-2H-pyran-2-yloxy)octanoate (Table 2, entry 2; Table 3, entry 7)



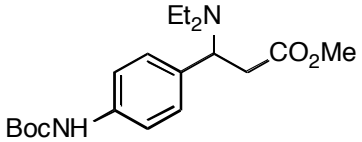
General Procedure B. Yield (0.125 mmol scale): 40 mg (Table 2, 98%) and 30 mg (Table 3, 73%). ^{1}H NMR ($CDCl_3$) δ 0.96 (t, J = 6.9 Hz, 6H), 1.19-1.82 (m, 9H), 2.15 (dd, J = 14.1 Hz, J = 6.9 Hz, 2H), 2.26-2.46 (m, 5H), 3.08 (quin, J = 6.9 Hz, 1H), 3.31-3.38 (m, 1H), 3.43-3.50 (m, 1H), 3.62 (s, 3H), 3.65-3.73 (m, 1H), 3.80-3.87 (m, 1H), 4.54 (brs, 1H); ^{13}C NMR ($CDCl_3$) δ 14.9 (2C), 19.9, 25.7, 26.6, 27.1, 30.0, 31.0, 31.3, 36.2, 43.5 (2C), 51.6, 57.1, 62.5, 67.8, 99.0, 173.9; IR (neat) 2939, 1740 cm^{-1}. Anal. Calcd. for $C_{18}H_{35}NO_4$: C, 65.62; H, 10.71; N, 4,25. Found: C, 65.51; H, 10.39; N, 3.76.

Methyl 5-(tert-butoxycarbonylamino)-3-(diethylamino)pentanoate (Table 2, entry 3)



General Procedure B. Yield (0.125 mmol scale): 24 mg (64%). ^{1}H NMR ($CDCl_3$) δ 1.03 (t, J = 7.2 Hz, 6H), 1.40 (s, 9H), 1.44-1.50 (m, 2H), 1.60-1.69 (m, 2H), 2.12 (dd, J = 17.7 Hz, J = 9.7 Hz, 1H), 2.20-2.31 (m, 2H), 2.48-2.60 (m, 3H), 3.01-3.10 (m, 1H), 3.12-3.23 (m, 1H), 3.26-3.39 (m, 1H), 3.64 (s, 3H), 5.76, (s (br), 1H); ^{13}C NMR ($CDCl_3$) δ 14.4 (2C), 28.7 (3C), 31.2, 34.3, 39.8, 43.4 (2C), 51.9, 56.8, 156.2, 173.5; IR (neat) 3368, 2974, 1720 cm^{-1}. Anal. Calcd. for $C_{15}H_{30}N_2O_4$: C, 59.57; H, 10.00; N, 9.26. Found: C, 59.27; H, 9.75; N, 9.21.

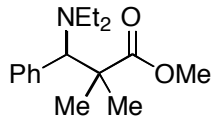
Methyl 3-(4-((tert-butoxycarbonyl)amino)phenyl)-3-(diethylamino)propanoate (Table 2, entry 4; Table 3, entry 8)



General Procedure B. Yield (0.125 mmol scale): 41 mg (Table 2, 64%) and 43 mg (Table 3, 98%). ^{1}H NMR ($CDCl_3$) δ 0.98 (t, J = 6.9 Hz, 6H), 1.49 (s, 9H), 2.20-2.31 (m, 2H), 2.46-2.53 (m, 2H), 2.63 (dd, J = 14.4 Hz, J = 8.1 Hz, 1H), 2.90 (dd, J = 14.4 Hz, J = 7.2 Hz, 1H),

4.23 (t, J = 7.5 Hz, 1H), 6.45 (s (br), 1H), 7.17 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H; ^{13}C NMR ($CDCl_3$) δ 13.6 (2C), 28.6 (3C), 37.7, 43.4 (2C), 51.8, 60.0, 118.3 (2C), 129.0 (2C), 134.6, 137.5, 152.9, 172.7; IR (neat) 3343, 2974, 1732 cm^{-1}. HRMS (FAB) Calcd. for $C_{19}H_{30}N_2O_4$•H^+ (MH^+): 351.2278. Found: 351.2283.

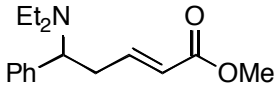
Methyl 3-(diethylamino)-2,2-dimethyl-3-phenylpropanoate (Table 2, entry 5)



General Procedure A. Yield (4.4 mmol scale): 0.93 g (81%); (0.125 mmol scale): 29 mg (88%). ^{1}H NMR ($CDCl_3$) δ 0.94 (t, J = 7.2 Hz, 6H), 1.04 (s, 3H), 1.32 (s, 3H), 2.34 (dq, J = 13.2 Hz, J = 6.6 Hz, 2H), 2.64 (dq, J = 13.2 Hz, J = 4.2 Hz, 2H), 3.60 (s, 3H), 4.02 (s, 1H), 7.23-7.30 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 13.2 (2C), 21.9, 25.8, 44.9 (2C), 47.5, 51.6, 70.9, 126.9, 127.5 (2C), 130.4 (2C), 138.1, 178.4; IR (neat) 2970, 1740 cm^{-1}. HRMS for $C_{16}H_{25}O_2N$•H^+: Calcd.:264.1964. Found: 264.1964.

The product shown as entry 6 in Table 2 has been reported in the literature. See for example, Suginome, Lars, Murakami *Org. Lett*. **2004**, *6*, 1167. CAS registry No.: 83188-04-9

Methyl (*E*)-5-(diethylamino)-5-phenylpent-2-enoate (eq 2)



General Procedure A. Yield (0.125 mmol scale): 32 mg (98%). ^{1}H NMR ($CDCl_3$) δ 0.99 (t, J = 6.9 Hz, 6H), 2.34 (dq, J = 12.9 Hz, J = 6.9 Hz, 2H), 2.54-2.66 (m, 2H), 3.45 (m, 3H), 2.72-2.83 (m, 1H), 3.66 (s, 3H), 3.80 (dd, J= 11.2 Hz, J= 6.0 Hz, 1H), 5.77 (dt, J= 15.6 Hz, J = 1.5 Hz, 1H), 6.87 (dt, J = 15.6 Hz, J = 4.2 Hz, 1H), 7.24-7.32 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 13.1 (2C), 5.6, 43.3 (2C), 51.6, 63.6, 122.3, 127.3, 128.3 (2C), 128.6 (2C), 140.5, 147.5, 167.0; IR (neat) 2971, 1725, 1655 cm^{-1}. Anal. Calcd. for $C_{16}H_{23}NO_2$: C, 73.53; H, 8.87; N, 5.36. Found: C, 73.41; H, 8.73; N, 5.41.

Three-Component Mannich Reactions Using Aldehyde, *sec*-Amine, Silyl Ketene Acetal with Amino Boranes

General procedure A Including Extractive Workup with Acid-Treatment

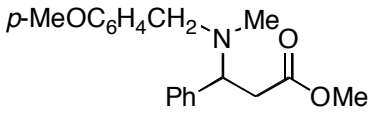
To a solution of diisopyopylaminoborane (**11** or **12**, 0.125 mmol) in NMP (0.25 ml)were

added 2-piperidinone (0.025 mmol), aldehyde (0.19 mmol), and *sec*-amine (0.125 mmol). To the mixture was added the nucleophile (0.13 mmol); the mixture was stirred at room temperature for 1–3 h. To the reaction mixture were added ice water and *tert*-butyl methyl ether (15 mL) with stirring. Basic material was extracted from the organic phase three times with 5 ml 0.5 N hydrochloric acid. The combined acid layers were kept at 0 °C, washed with 10 ml *tert*-butyl methyl ether and the pH brought to 8 by addition of conc. ammonia solution. Organic materials were extracted with *tert*-butyl methyl ether three times and the combined organic layer was washed with 10 ml water. Evaporation left the crude products that showed purities of > 90% and were purified by column chromatography on silica gel (eluent: ethyl acetate/hexane).

General procedure B: Non-Acidic Workup

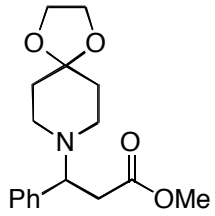
Reactions were performed according to the procedure same as the general procedure A shown above. The reaction mixture was then diluted with ether (15 mL) and washed with ice-water three times. The organic layer was dried over K_2CO_3 and evaporated under vacuum. The crude material was purified by chromatography on silica gel using ethyl acetate/hexane mixture as an eluent.

Methyl 3-[(4-methoxybenzyl)(methyl)amino]-3-phenylpropanoate (Table 3, entry 2)



General Procedure A. Yield (0.125 mmol scale): 28 mg (71%). ^{1}H NMR ($CDCl_3$) δ 2.07 (σ, 3H), 2.72 (dd, J = 14.7 Hz, J = 7.2 Hz, 1H), 3.06 (dd, J = 14.7 Hz, J = 8.1 Hz, 21H), 3.24 (d, J = 13.2 Hz, 1H), 3.47 (d, J = 13.2 Hz, 1H), 3.63 (s, 3H), 3.78 (s, 3H), 4.42 (t, J= 7.5 Hz, 1H), 6.82 (dd, J = 6.3 Hz, J = 1.8 Hz, 2H), 7.17 (d, J = 8.4 Hz, 2H), 7.25-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 37.6, 37.8, 51.9, 55.5, 58.0, 64.4, 113.8 (2C), 127.6, 128,3 (2C), 128,6 (2C), 130.0 (2C), 131.7, 138.4, 158.8, 172.6; IR (neat) 2951, 1740, 1512, 1246 cm^{-1}. Anal. Calcd. for $C_{19}H_{23}NO_3$: C, 72.82; H, 7.40; N, 4.47. Found: C, 72.76; H, 7.47; N, 4.53.

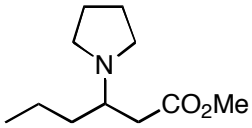
Methyl 3-(1,4-Dioxa-8-azaspiro[4,5]dec-8-yl)-3-phenylpropanoate (Table 3, entry 3)



General Procedure B. Yield (0.125 mmol scale): 32 mg (84%). ^{1}H NMR ($CDCl_3$) δ 1.66 (t, J = 5.4 Hz, 4H), 2.40-2.47 (m, 2H), 2.48-2.58 (m, 2H), 2.68 (dd, J = 19.6, 10.0 Hz, 1H), 2.98 (dd, J = 19.6, 10.0 Hz, 1H), 3.59 (s, 3H), 3.86 (s, 4H), 4.06 (t, J = 7.8 Hz, 1H), 7.20-7.33 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 35.5 (2C), 38.3, 47.9 (2C), 51.8, 64.4 (2C), 65.7, 107.4, 127.6, 128.4 (4C), 138.8, 172.5; IR (neat) 2953, 1740 cm^{-1}. Anal. Calcd. for $C_{17}H_{23}NO_4$: C, 66.86; H, 7.59; N, 4.59. Found: C, 66.66; H, 7.66; N, 4.51.

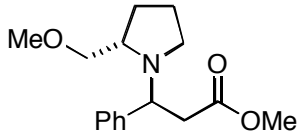
The product shown as entry 4 in Table 3 has been reported in the literature. See for example, Pacheco et al. *Bull. Chim. Soc. Fr.* **1962**, 1379. CAS registry No.: 7032-65-7.

Methyl 3-(pyrrolidin-1-yl)hexanoate (Table 3, entry 5)



General Procedure A. Yield (0.125 mmol scale): 23 mg (94%). ^{1}H NMR ($CDCl_3$) δ 0.87 (dt, J = 6.9 Hz, 3H), 1.28-1.52 (m, 4H), 1.67-1.74 (m, 4H), 2.32 (dd, J = 14.7 Hz, J = 6.9 Hz, 1H), 2.49-2.56 (m, 4H), 3.64 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 14.5, 19.2, 23,7 (2C), 35.4, 36.7, 49.8 (2C), 51.7, 59.0, 173.7; IR (neat) 2959, 1740, 1458, 1437 cm^{-1}. Anal. Calcd. for $C_{11}H_{21}NO_2$: C, 66.29; H, 10.62; N, 7.03. Found: C, 66.29; H, 10.37; N, 7.00.

Methyl 3-((*S*)-2-methoxymethylpyrolidino)-3-phenylpropanoate (Table 3, entry 6)

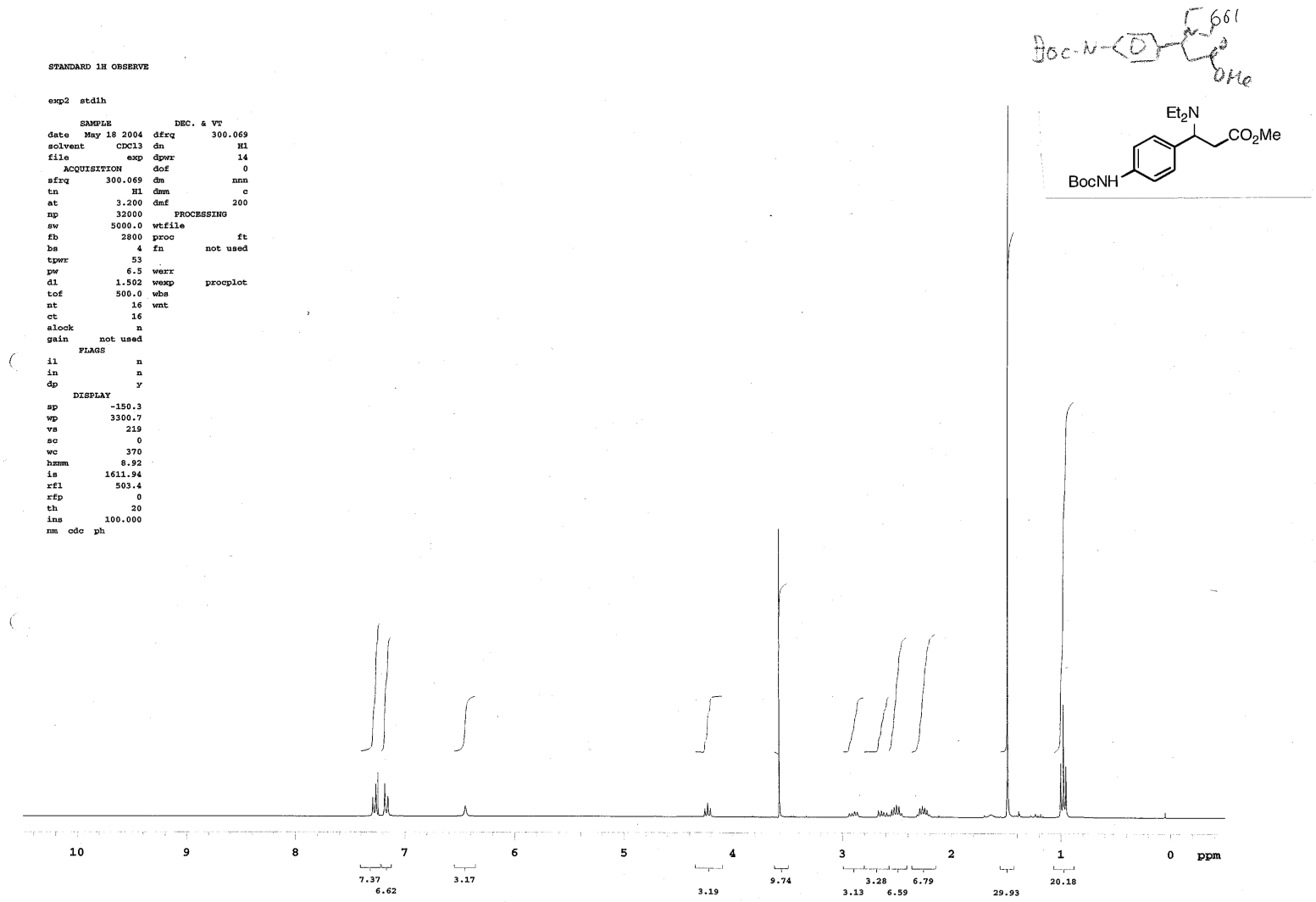


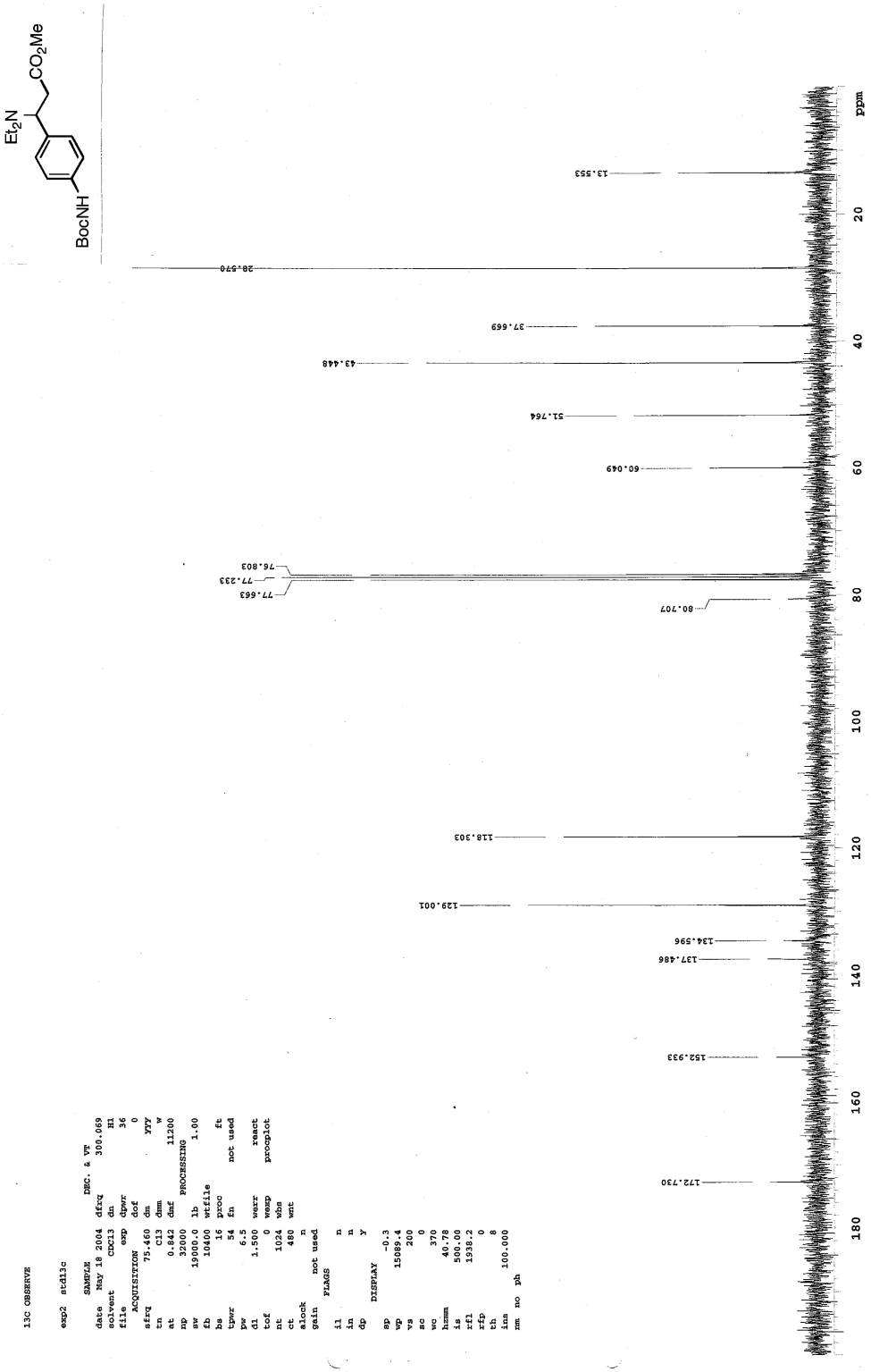
General Procedure A. Yield (0.40 mmol scale): 105 mg (88%). $[\alpha]_D^{25}$ = –42.2 (c 0.832, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 0.60-0.80 (m, 4H), 2.45-2.55 (m, 1H), 2.75-3.02 (m, 6H), 3.15 (s, 3H), 3.51 (s, 3H), 4.20 (dd, J = 9.0, 5.4 Hz, 1H), 7.19-7.36 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 23.6, 28.6, 37.6, 50.8, 51.5, 58.8, 59.7, 63.1, 76.1, 127.3, 128.1, 128.2, 141.5, 172.3; IR (neat) 2951, 1735 cm^{-1}. HRMS Calcd. for $C_{16}H_{23}NO_3 \bullet H^+$ (MH^+): 278.1751.

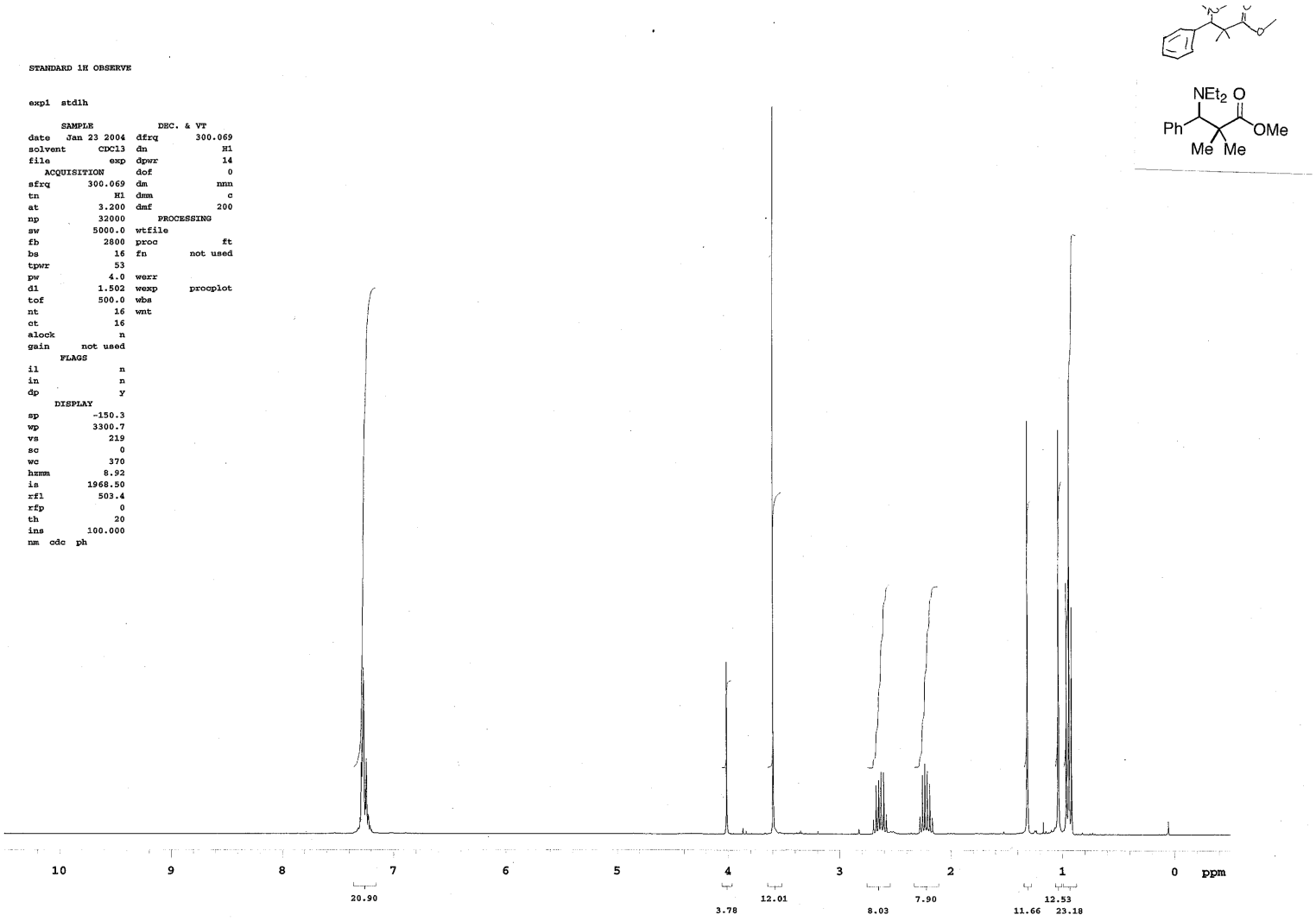
Found: 278.1759.

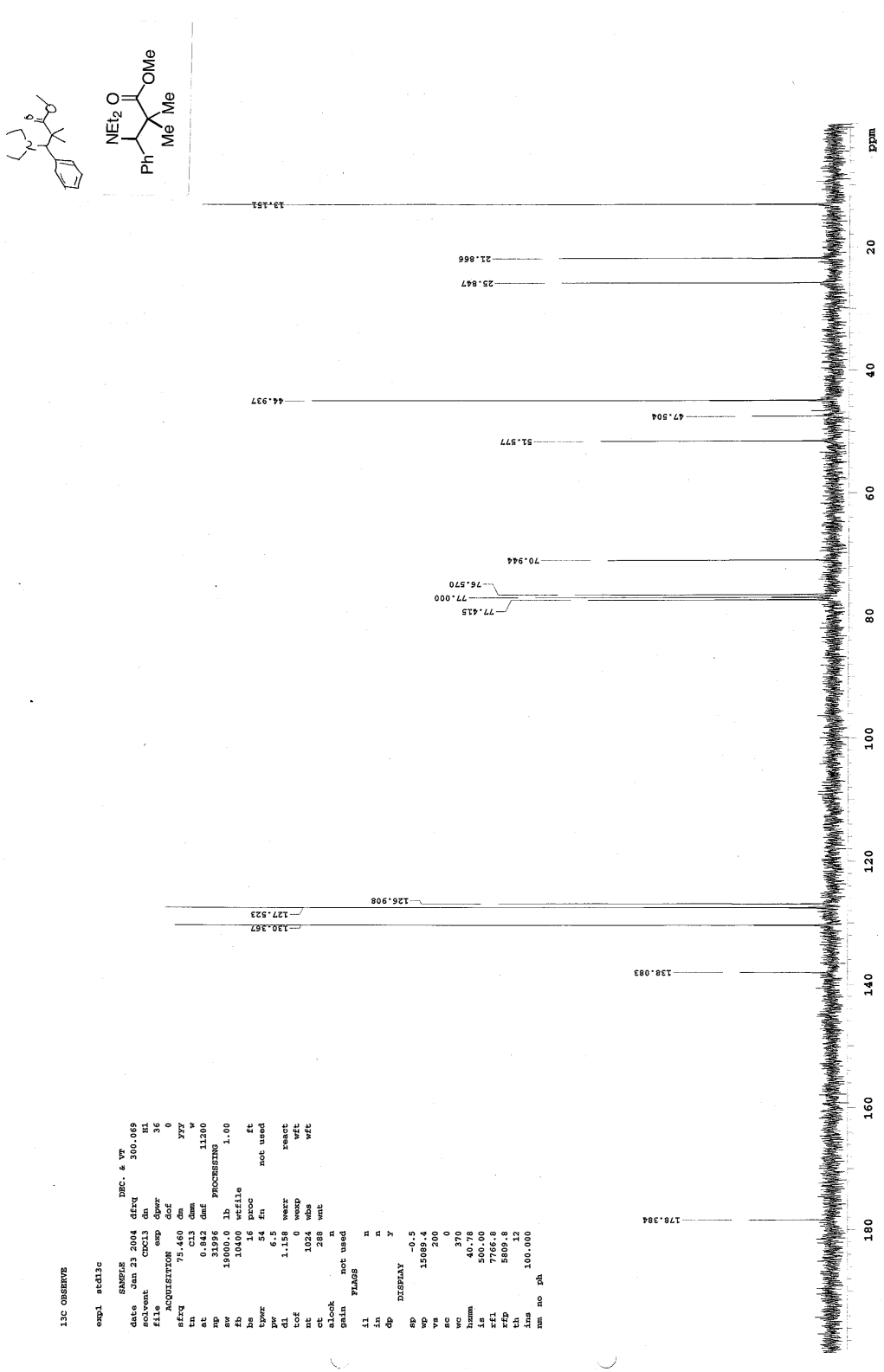
Reaction of Aminoborane 5 and Benzaldehyde in DMF-d_7.

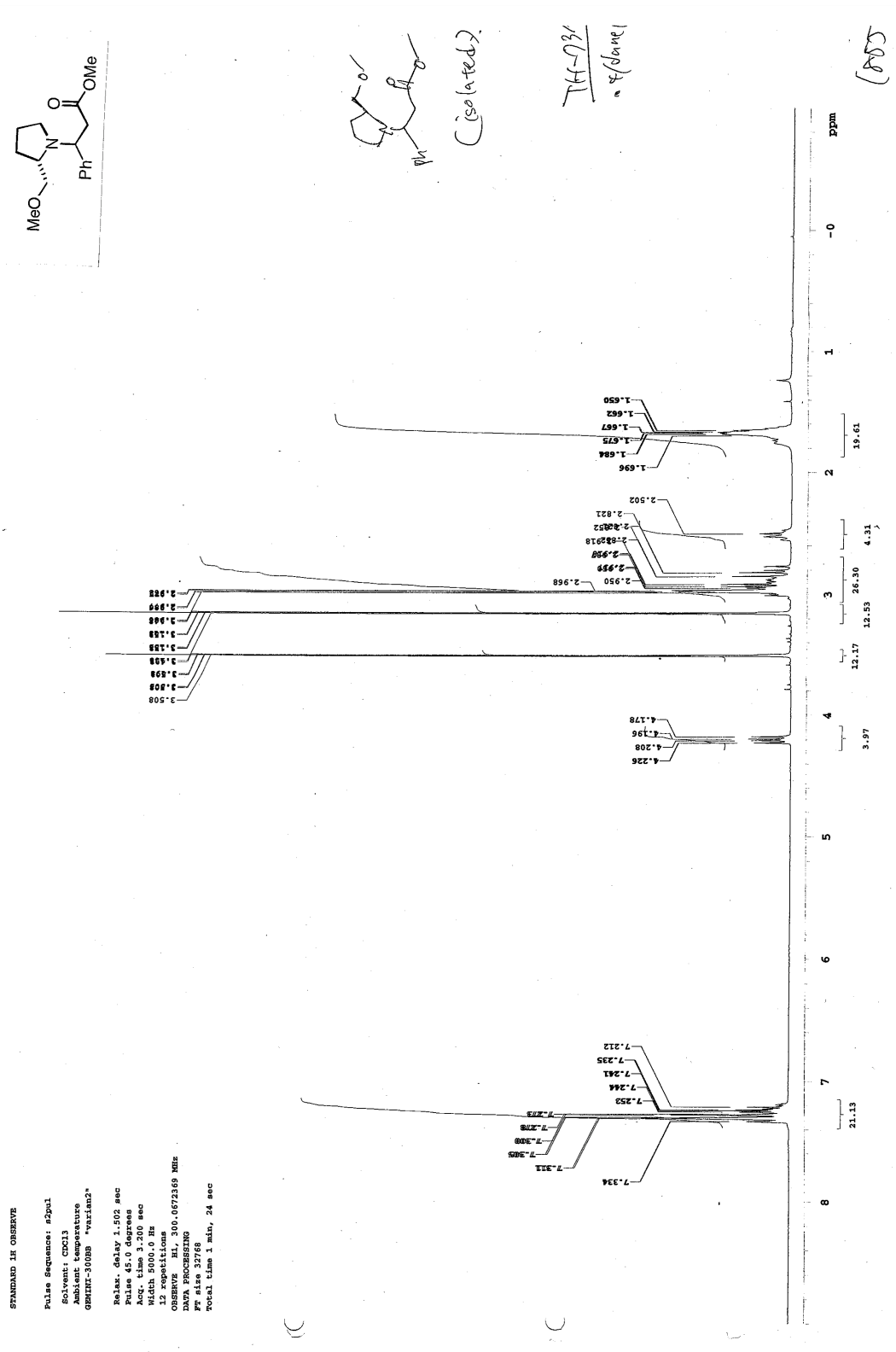
Aminoborane **5** (24 mg, 0.125 mmol) and benzaldehyde (13 mg, 0.125 mmol) were dissolved in freshly distilled DMF-d_7 (0.70 mL) at room temperature. The mixture was subjected to ^{1}H NMR analyses after 10 min (55% conv.), 3 h (64% conv.), and 14 h (64% conv.). The ^{1}H and ^{13}C NMR charts (after 14 h) are shown at the end of the Supporting Information.

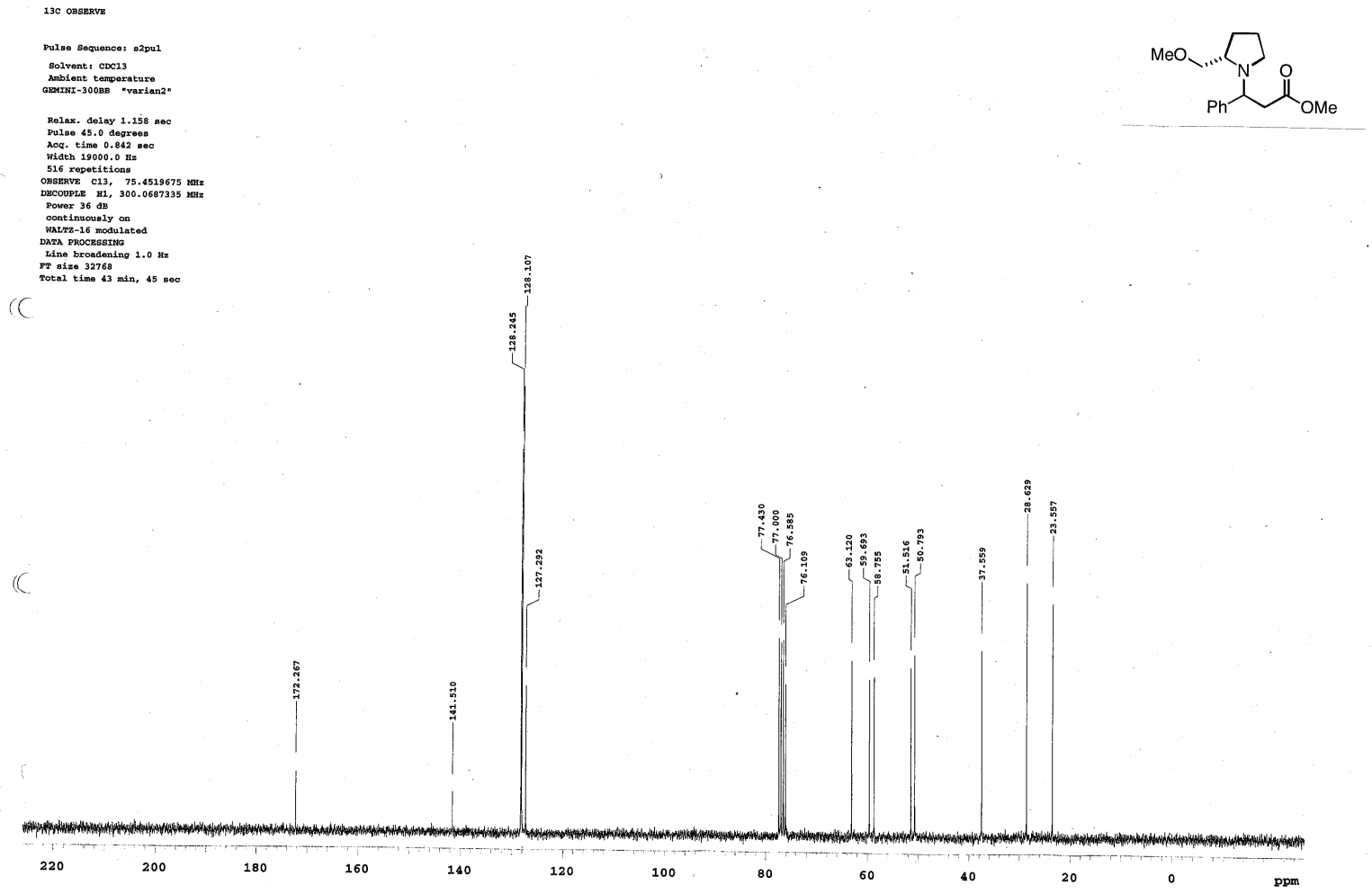

STANDARD 1H OBSERVE
exp2 stdih
SAMPLE
date May 18 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 300.069
tn H1
at 3.200
np 32000
sw 5000.0
fb 2800
bs 4
tpwr 53
pw 6.5
dl 1.502
tof 500.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -150.3
wp 3300.7
vs 219
sc 0
wc 370
hzmm 8.92
is 1611.94
rfl 503.4
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.069
dn H1
dpwr 14
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp procplot
wbs
wnt
Et2N
CO2Me
BocNH
10
9
8
7
6
5
4
3
2
1
0
ppm
7.37
6.62
3.17
3.19
9.74
3.13
3.28
6.59
6.79
29.93
20.18

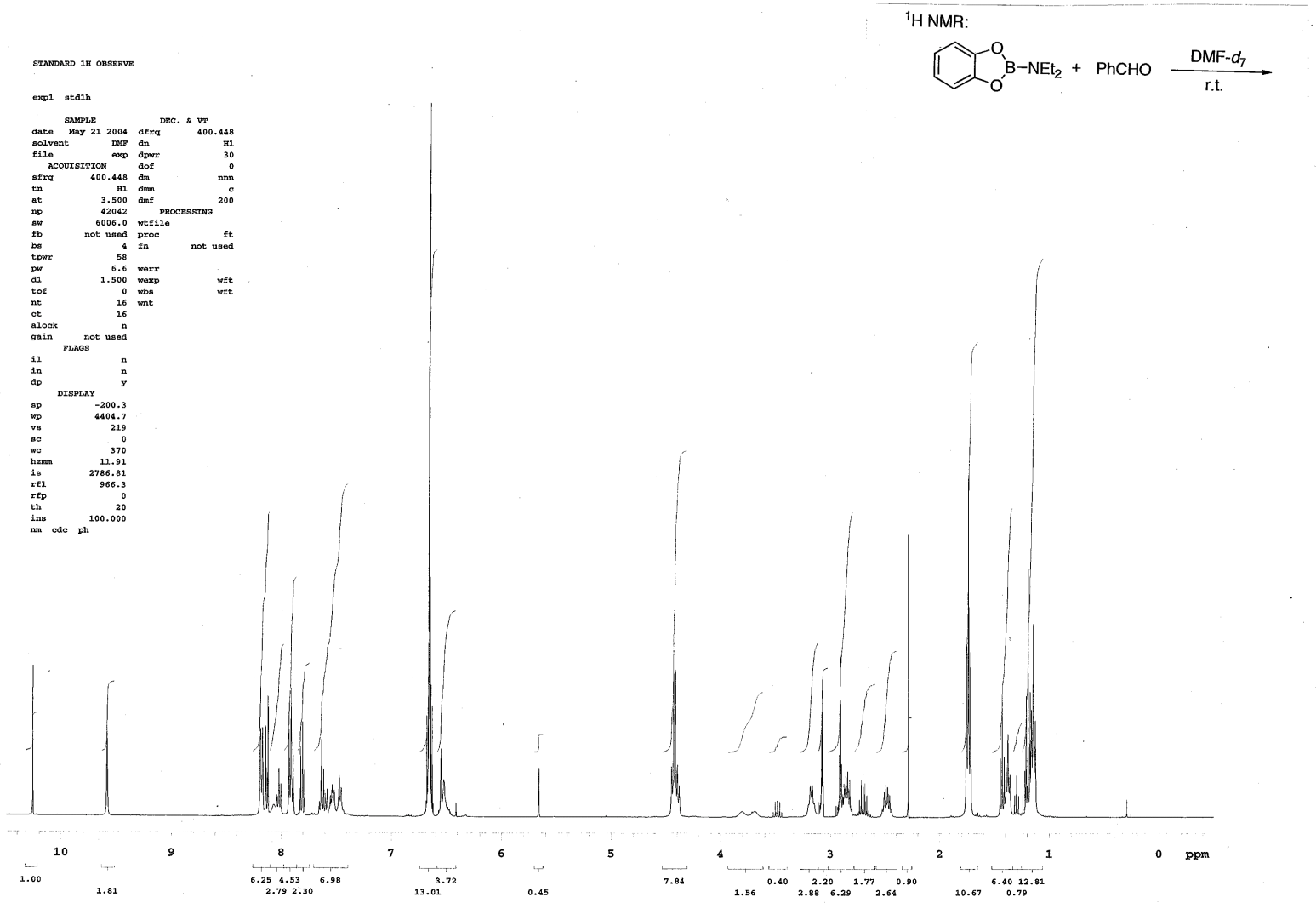

Et2N
CO2Me
BocNH
13C OBSERVE
exp2 std13c
SAMPLE
date May 18 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 0.842
np 32000
sw 19000.0
fb 10400
bs 16
tpwr 54
pw 6.5
d1 1.500
tof 0
nt 1024
ct 480
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.3
wp 15089.4
vs 200
sc 0
wc 370
hzmm 40.78
is 500.00
rfl 1938.2
rfp 0
th 8
lin 100.000
nm no ph
DEC. & VT
dfrq 300.069
dn H1
dpwr 36
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
1b 1.00
wtfile
proc ft
fn not used
werr react
wdw procplot
wbs
wnt
13.553
28.570
37.669
43.448
51.764
60.049
76.803
77.233
77.663
80.707
118.303
129.001
134.596
137.486
152.933
172.730
ppm


STANDARD 1H OBSERVE
exp1 stdlh
SAMPLE
date Jan 23 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 300.069
tn H1
at 3.200
np 32000
sw 5000.0
fb 2800
bs 16
tpwr 53
pw 4.0
d1 1.502
tof 500.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -150.3
wp 3300.7
vs 219
sc 0
wc 370
hzmm 8.92
is 1968.50
rfl 503.4
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 300.069
dn H1
dpwr 14
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp procplot
wba
wmt
NEt2 O
Ph
OMe
Me Me
10
9
8
7
6
5
4
3
2
1
0
ppm
20.90
3.78
12.01
8.03
7.90
11.66
12.53
23.18


13C OBSERVE
expl stdl3c
SAMPLE
date Jan 23 2004
solvent CDCl3
file
ACQUISITION
sfrq 75.460
tn C13
at 0.842
np 31996
sw 19000.0
fb 10400
bs 16
tpwr 54
pw 6.5
pl1 1.158
tof 0
nt 1024
ct 288
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.5
wp 15089.4
vs 200
sc 0
wc 370
hzmm 40.78
is 500.00
rfl 7766.8
rfp 5809.8
th 12
lvl 100.000
nm no ph
DEC. & VT
dfrq 300.069
dn H1
dpwr 36
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
1b 1.00
wtfile
proc ft
fn not used
werr rsat
wexp wft
wbs wft
wnt
NEt2
O
OMe
Ph
Me
Me
178.384
138.083
130.367
127.523
126.908
77.415
77.000
76.570
70.944
51.577
47.504
44.937
25.847
21.866
13.151
ppm
180
160
140
120
100
80
60
40
20


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.300 sec
Width 5000.0 Hz
12 repetitions
OBSERVE H1, 300.0672369 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
MeO
Ph
OMe
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
516 repetitions
OBSERVE C13, 75.4519675 MHz
DECOUPLE H1, 300.0687335 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 43 min, 45 sec
MeO
N
O
Ph
OMe
172.267
141.510
128.245
128.107
127.292
77.430
77.000
76.585
76.109
63.120
59.693
58.755
51.516
50.793
37.559
28.629
23.557
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date May 21 2004
solvent DMF
file exp
ACQUISITION
sfrq 400.448
tn H1
at 3.500
np 42042
sw 6006.0
fb not used
bs 4
tpwr 58
pw 6.6
d1 1.500
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4404.7
vs 219
sc 0
wc 370
hzmm 11.91
is 2786.81
rfl 966.3
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 400.448
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
wexr
wexp wft
wbs wft
wnt
1H NMR:
B–NEt2
+ PhCHO
DMF-d7
r.t.
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.81
6.25 4.53 6.98
2.79 2.30
3.72
13.01
0.45
7.84
0.40
1.56
2.20 1.77 0.90
2.88 6.29 2.64
6.40 12.81
10.67 0.79

Mon May 24 14:22:01 2004
PEAK 52
MXINT 77.6265945
RESOL 0.7926643 HZ
RESOL 0.0078863 PPM
EXREF 30.1000004 PPM
OBS -30438.89 HZ
ABOBS 100535.5000000 KHZ
NGAIN 1
COMNT

NO.	PPM	INT(%)	FREQU(HZ)	POSITION	BAR GRAPH
1	193.59226	19.21092	19460.913	4750	+++
2	172.57025	4.01611	17347.670	7416	
3	162.98970	37.56260	16384.583	8631	+++++++
4	162.89508	3.96657	16375.071	8643	
5	162.69795	38.75281	16355.254	8668	+++++++
6	162.40620	38.51586	16325.926	8705	+++++++
7	153.81919	2.78271	15462.714	9794	
8	153.07798	34.02372	15388.204	9888	++++++
9	139.10536	4.43208	13983.603	11660	
10	137.40215	3.31138	13812.387	11876	
11	135.99858	9.74168	13671.293	12054	+
12	135.17851	18.36908	13588.856	12158	+++
13	132.98642	22.53567	13368.495	12436	++++
14	130.34487	22.04331	13102.953	12771	++++
15	130.13197	30.79151	13081.551	12798	++++++
16	129.84022	37.03117	13052.222	12835	+++++++
17	129.25671	14.28017	12993.565	12909	++
18	129.20151	9.07663	12988.016	12916	+
19	128.70474	10.25448	12938.078	12979	++
20	128.41299	3.16941	12908.750	13016	
21	128.33414	13.24935	12900.823	13026	++
22	128.28683	16.74634	12896.067	13032	+++
23	128.20009	6.59509	12887.348	13043	+
24	127.80583	3.34294	12847.715	13093	
25	117.76794	100.00000	11838.653	14366	++++++++++++++++++
26	108.17162	29.92878	10873.980	15583	+++++
27	80.78623	2.96333	8121.057	19056	
28	77.97909	2.99737	7838.868	19412	
29	43.22890	2.93089	4345.567	23819	
30	42.92138	3.71349	4314.683	23858	
31	42.40884	9.42002	4263.160	23923	+
32	42.03035	10.44497	4225.112	23971	++
33	35.84044	4.58389	3602.870	24756	
34	35.63543	13.76119	3582.261	24782	++
35	35.42253	27.35017	3560.859	24809	+++++
36	35.21751	32.21557	3540.250	24835	++++++
37	35.00461	26.44776	3518.848	24862	+++++
38	34.79959	13.70075	3498.238	24888	++
39	34.59458	4.51729	3477.629	24914	
40	30.73082	5.63362	3089.224	25404	+
41	30.67562	3.11917	3083.675	25411	
42	30.51792	16.75507	3067.822	25431	+++
43	30.31290	33.52541	3047.212	25457	++++++
44	30.10000	38.73856	3025.810	25484	+++++++
45	29.89498	33.54642	3005.201	25510	++++++
46	29.68208	16.59271	2983.799	25537	+++
47	29.47707	5.73689	2963.190	25563	+
48	13.20196	4.94059	1327.131	27627	
49	12.51595	7.65861	1258.169	27714	+
50	12.48441	11.96421	1254.998	27718	++
51	12.05860	14.03343	1212.194	27772	++
52	12.02706	6.55259	1209.024	27776	+