

## SUPPORTING INFORMATION

### Formal Total Synthesis of (-)-Balanol: Concise Approach to the Hexahydroazepine Segment Based on RCM

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**Instrumentation and Spectra Formats:** NMR: Spectra were recorded on a Bruker AC 200 or DMX 300 spectrometer in CDCl<sub>3</sub> or CD<sub>2</sub>Cl<sub>2</sub> as the solvents; chemical shifts ( $\delta$ ) are given in ppm relative to TMS, coupling constants ( $J$ ) in Hz. IR: Nicolet FT-7199, wavenumbers in cm<sup>-1</sup>. MS (EI): Finnigan MAT 8200 (70 eV). HRMS: Finnigan MAT SSQ 7000 (70 eV).

**Table 1. Characteristic Infrared Absorptions of New Compounds (cm<sup>-1</sup>)**

<b>Product</b>	<b>IR</b>
<b>6</b>	3064, 3031, 2990, 2924, 2863, 1644, 1606, 1586, 1497, 1453, 1086, 1070, 1028, 993, 935, 738, 699
<b>7</b>	3315, 3076, 3030, 2979, 2862, 1643, 1607, 1586, 1497, 1454, 1070, 995, 924, 736, 699
<b>8</b>	3452, 3080, 3030, 3006, 2978, 2930, 2869, 1694, 1646, 1496, 1367, 1249, 1070, 995, 926, 737, 699
<b>9</b>	3453, 3088, 3064, 3030, 2975, 2929, 2871, 1694, 1607, 1496, 1392, 1366, 1247, 1114, 737, 698
<b>10</b>	3066, 3030, 3005, 2978, 2929, 2868, 2121, 1697, 1643, 1591, 1456, 1367, 1249, 1172, 1153, 1097, 993, 931, 736, 698
<b>11</b>	3065, 3031, 2976, 2927, 2857, 2108, 1699, 1657, 1496, 1456, 1393, 1366, 1248, 1168, 1135, 1095, 737, 698
<b>13</b>	3427, 2929, 2875, 1665, 1606, 1543, 1504, 1454, 1420, 1367, 1251, 1176, 845, 768, 739, 697
<b>14</b>	3981, 2927, 2827, 1773, 1723, 1649, 1497, 1455, 1369, 1294, 1256, 1155, 1089, 1028, 997, 954, 931, 851, 740, 699
<b>15</b>	3087, 3066, 3030, 3005, 2976, 2933, 2881, 1701, 1497, 1477, 1455, 1367, 1257, 1171, 1105, 1073, 991, 929, 910, 736, 698

**Table 2. MS Data (EI) of New Compounds**

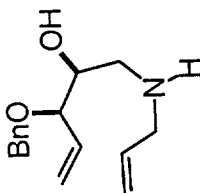
<b>Product</b>	<b><i>m/z</i> (rel. intensity)</b>
<b>6</b>	190 ([M <sup>+</sup> ], < 1), 147 (2), 107 (25), 91 (100), 84 (9), 65 (10), 55 (6)
<b>7</b>	247 ([M <sup>+</sup> ], < 1), 156 (14), 100 (18), 91 (37), 70 (100), 41 (26)
<b>8</b>	347 ([M <sup>+</sup> ], < 1), 274 (2), 200 (15), 167 (3), 148 (7), 144 (100), 100 (30), 91 (93), 70 (47), 57 (75), 41 (30)
<b>9</b>	319 ([M <sup>+</sup> ], < 1), 263 (6), 211 (4), 172 (9), 160 (9), 155 (12), 128 (4), 120 (3), 110 (7), 91 (100), 57 (39)
<b>10</b>	372 ([M <sup>+</sup> ], < 1), 299 (1), 225 (1), 197 (1), 170 (1), 147 (5), 114 (16), 91 (91), 70 (60), 57 (100), 41 (24)
<b>11</b>	344 ([M <sup>+</sup> ], < 1), 260 (1), 225 (2), 186 (1), 180 (3), 169 (14), 160 (6), 125 (11), 91 (100), 57 (57)
<b>13</b>	441 ([M <sup>+</sup> ], < 1), 253 (27), 228 (27), 211 (56), 170 (4), 157 (36), 121 (8), 113 (9), 91 (100), 57 (35)
<b>14</b>	358 ([M <sup>+</sup> ], < 1), 252 (4), 246 (4), 152 (8), 95 (14), 91 (100), 57 (33)
<b>15</b>	347 ([M <sup>+</sup> ], < 1), 318 (5), 262 (16), 218 (12), 200 (5), 144 (12), 100 (37), 91 (100), 70 (7), 57 (70)

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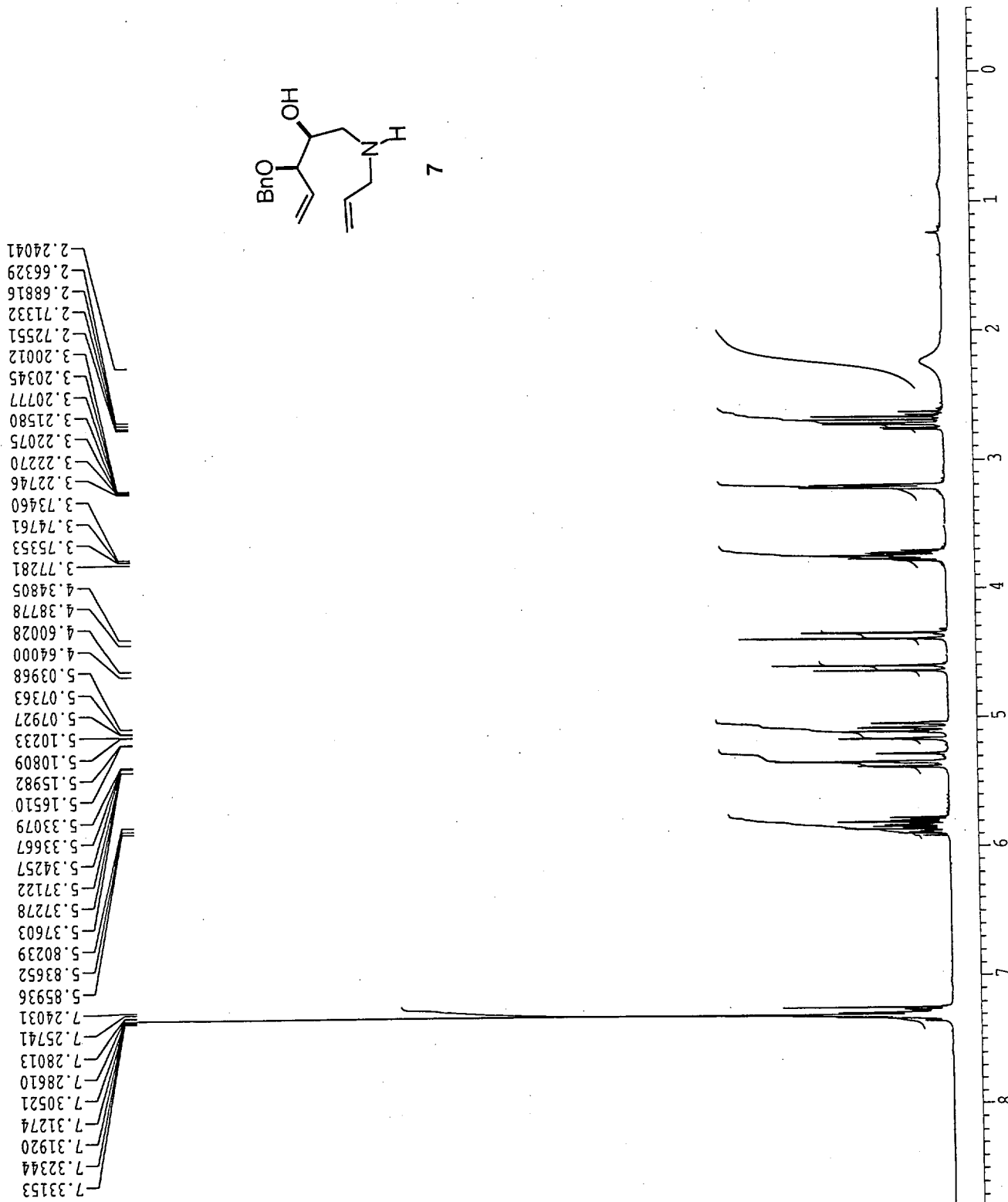
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7



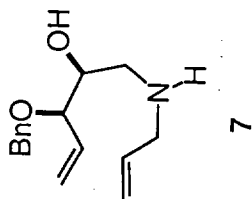
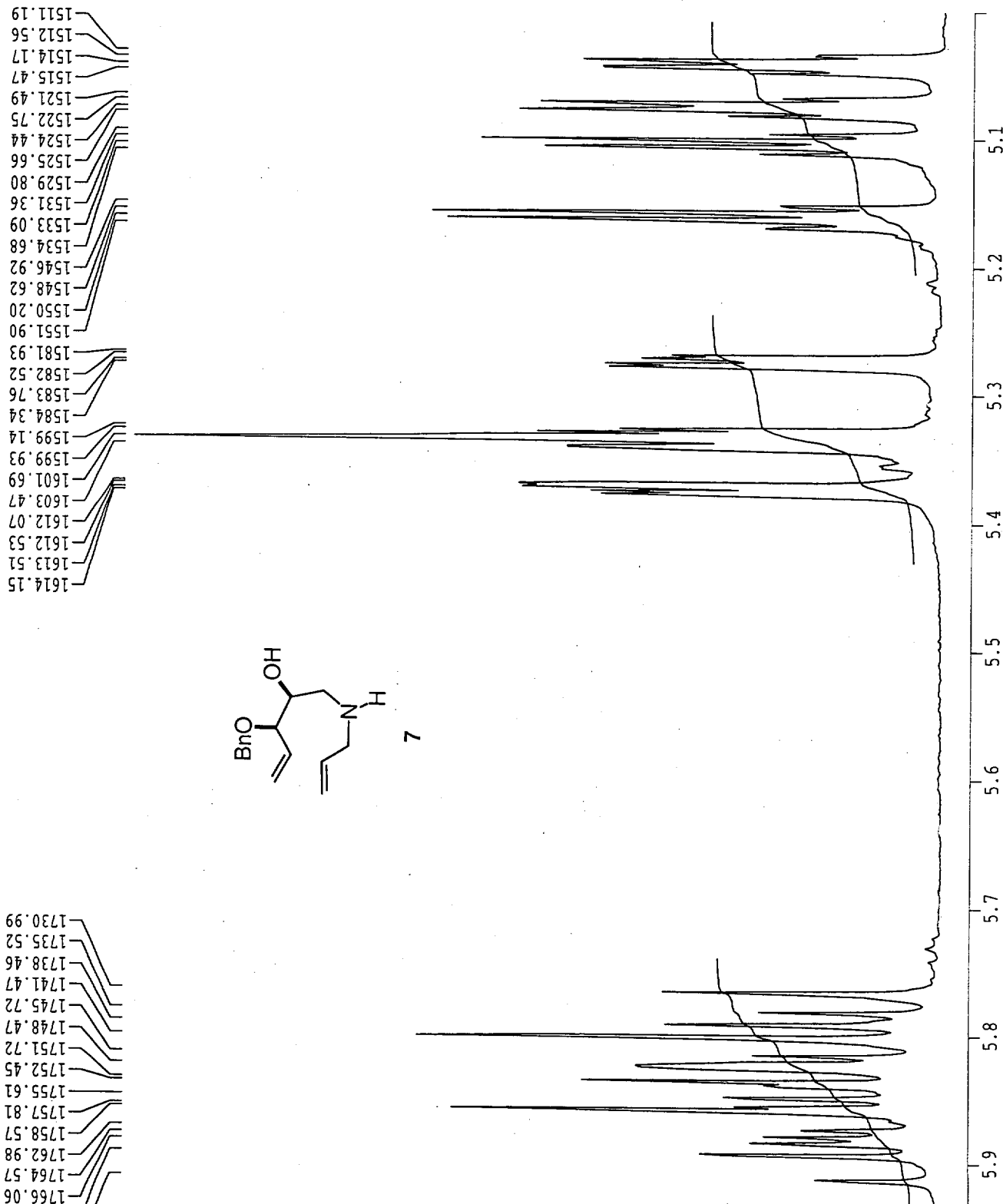
THL-TA-087-01

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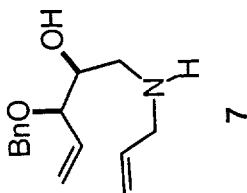


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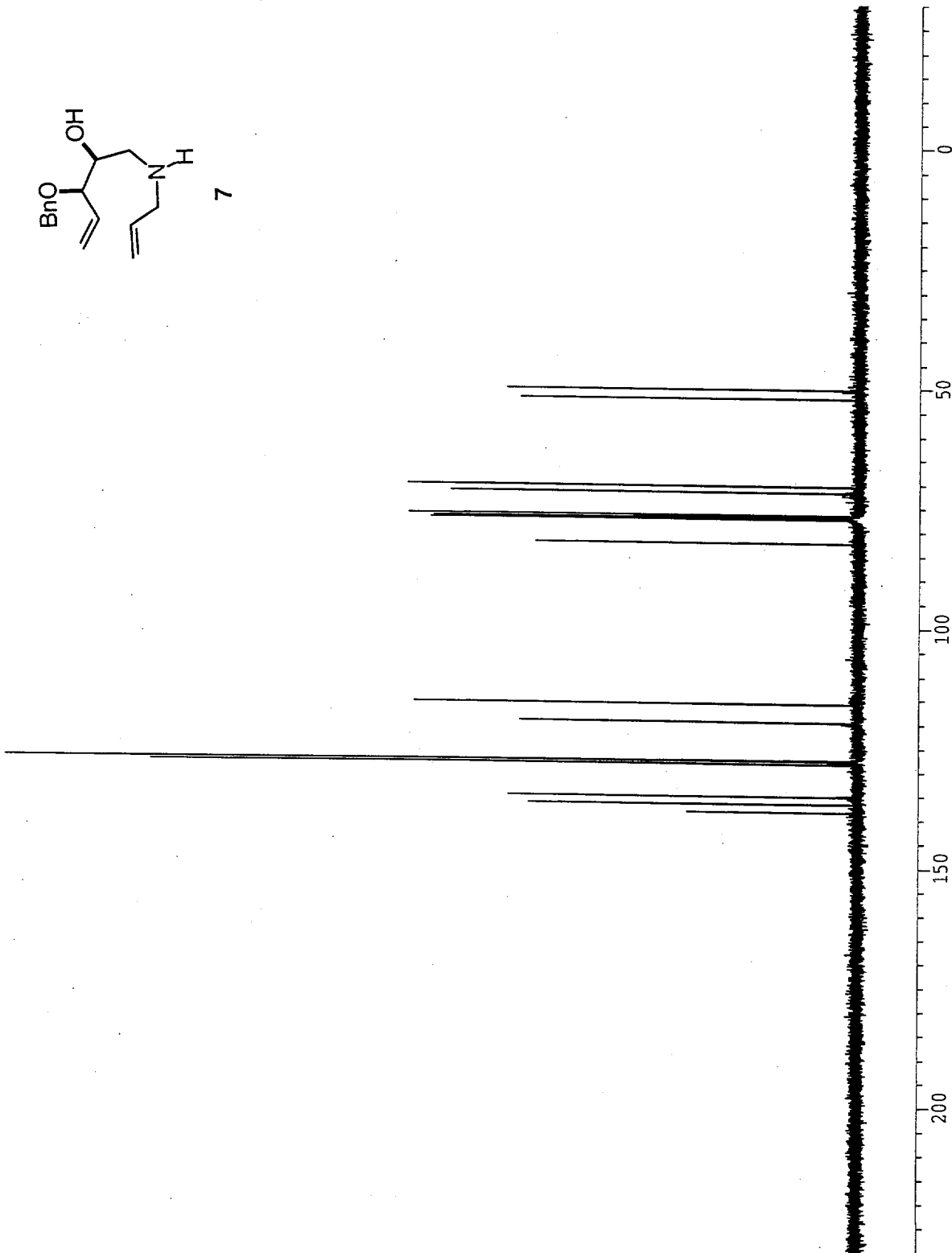
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 F1 18866.94 Hz  
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 F2 -2264.03 Hz  
 PPMCM 12.72727 ppm/cm  
 HZCM 960.49866 Hz/cm



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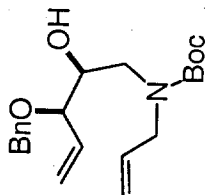
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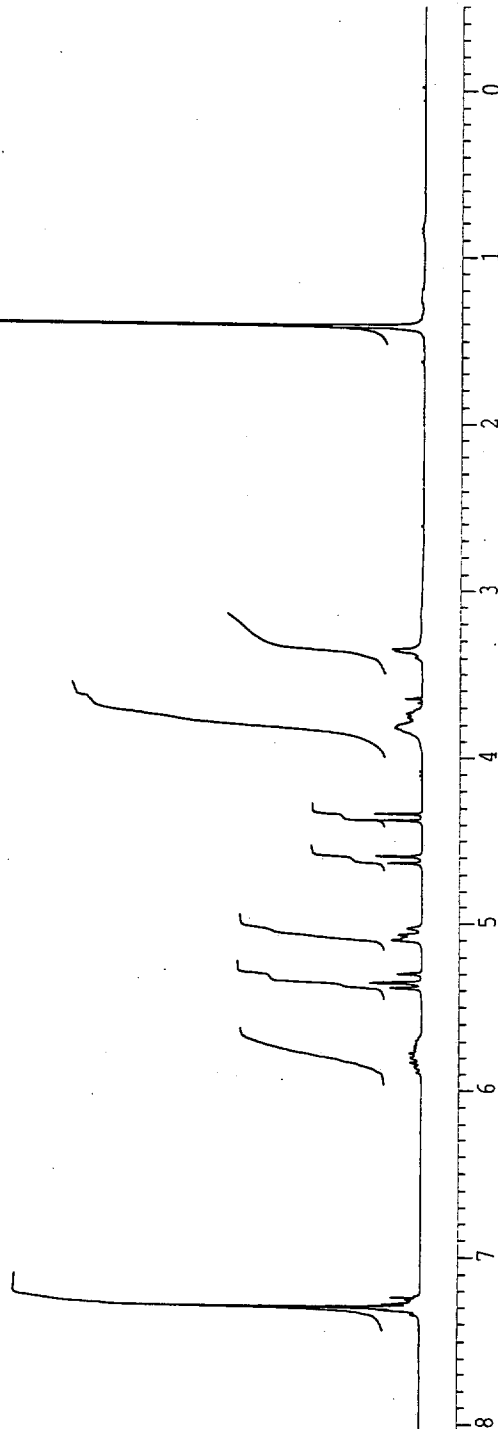
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 F1 2851.24 Hz  
 F2P -0.500 ppm  
 F2 -150.07 Hz  
 PPMCH 0.45455 ppm/cm  
 HZCM 136.42273 Hz/cm

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8 (rotamers)

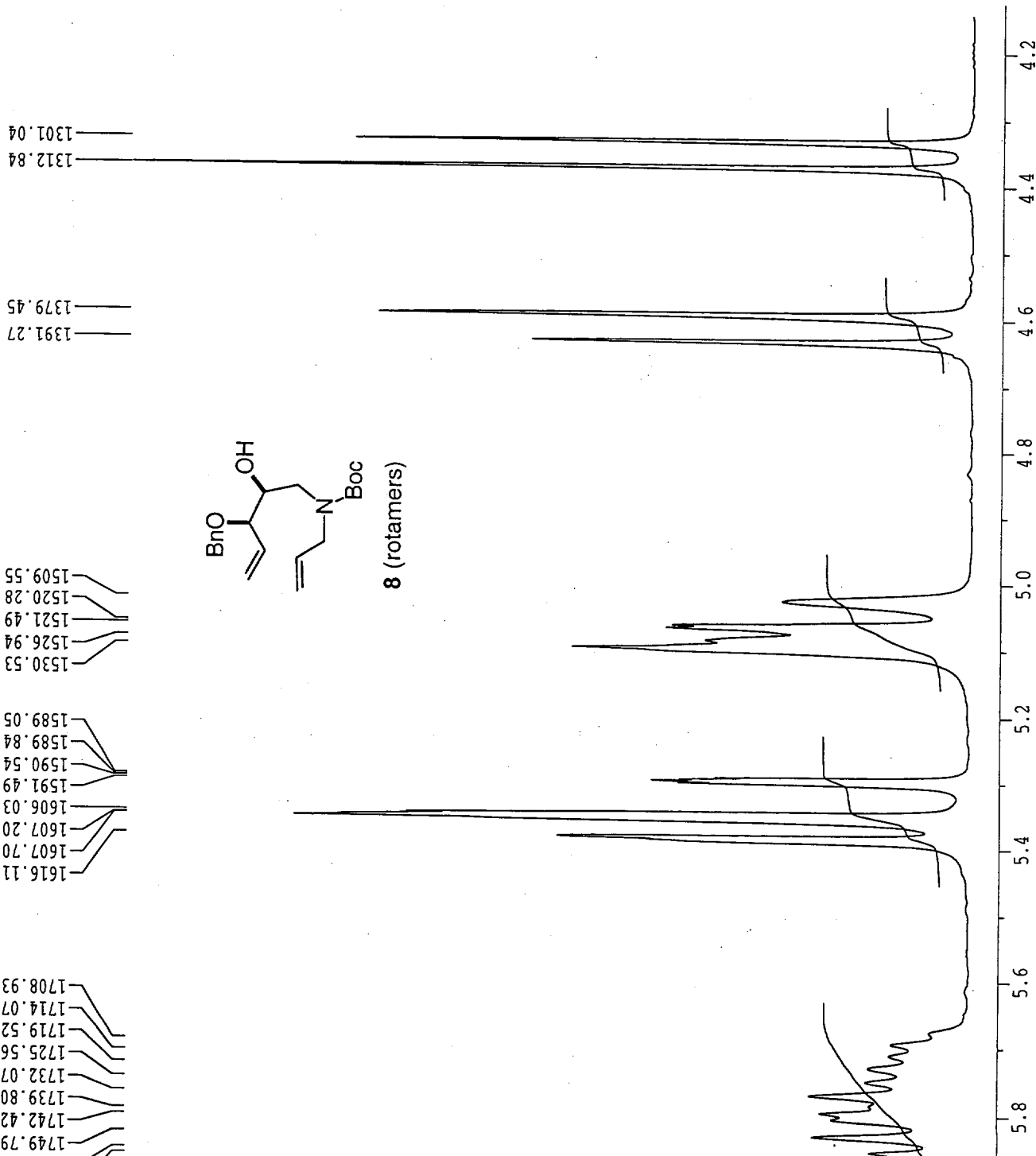


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THL-TA-090-02

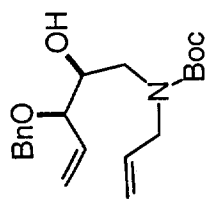
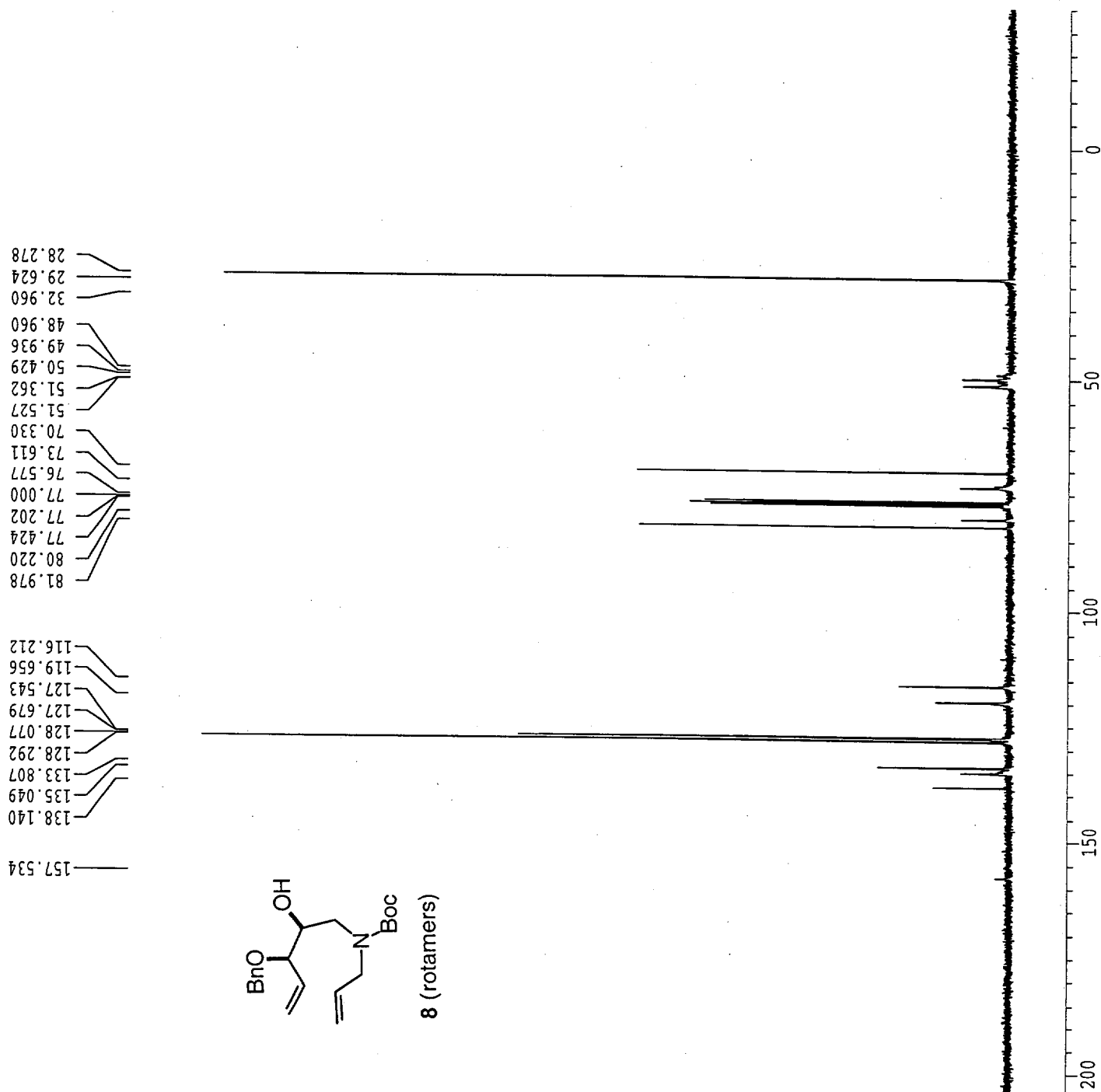


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 FIDRES 0.323966 Hz  
 AQ 1.5434228 sec  
 RG 16384  
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 DE 4.50 usec  
 TE 300.0 K  
 D11 0.03000000 sec  
 CEDPRG waltz16  
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1D NMR plot parameters  
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 F1 18866.94 Hz  
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 F2 -2264.03 Hz  
 PPMCH 12.72727 ppm/cm  
 HZCM 960.49872 Hz/cm



8 (rotamers)

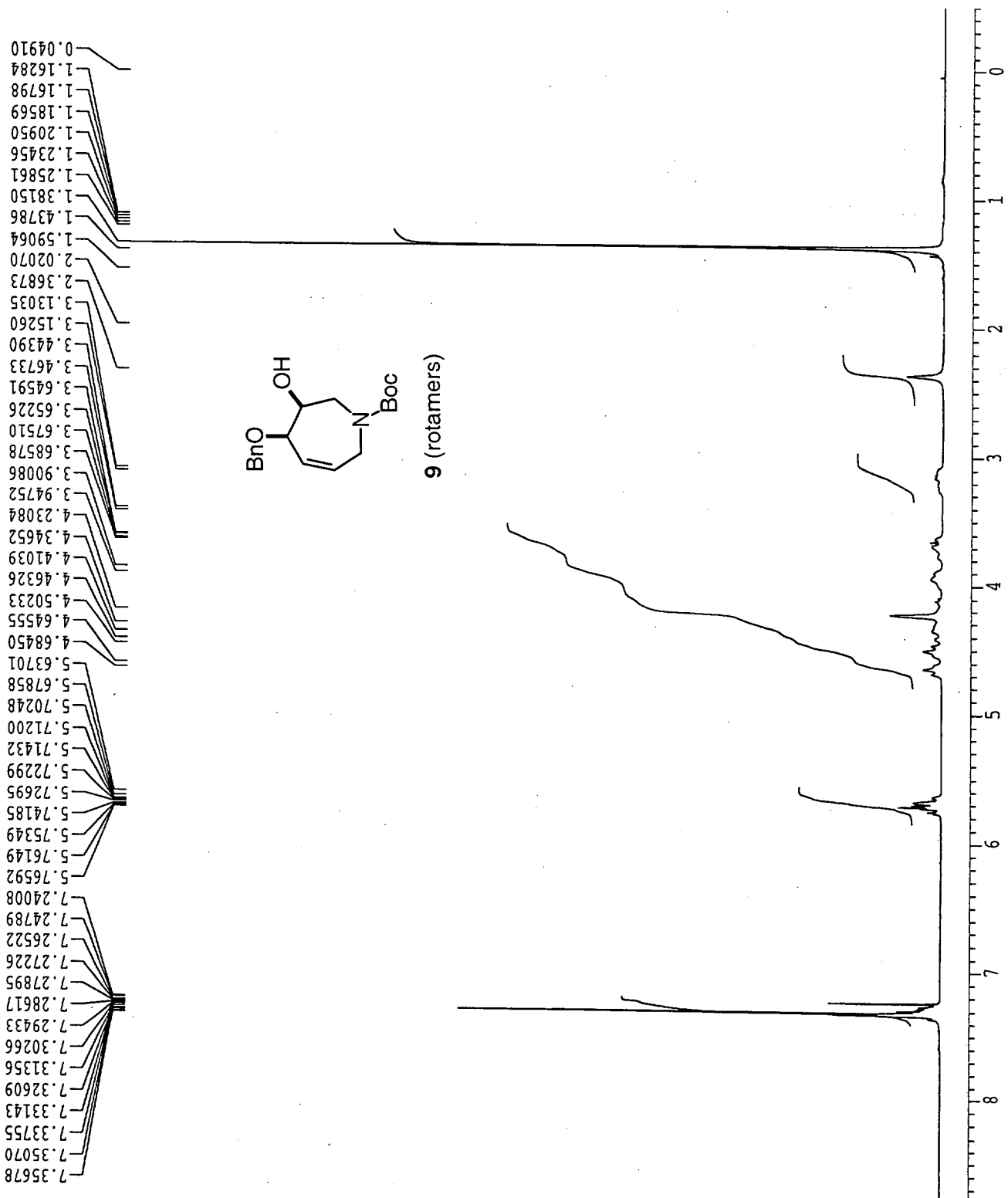
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 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 203.2  
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1D NMR plot parameters  
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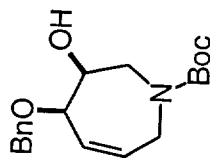
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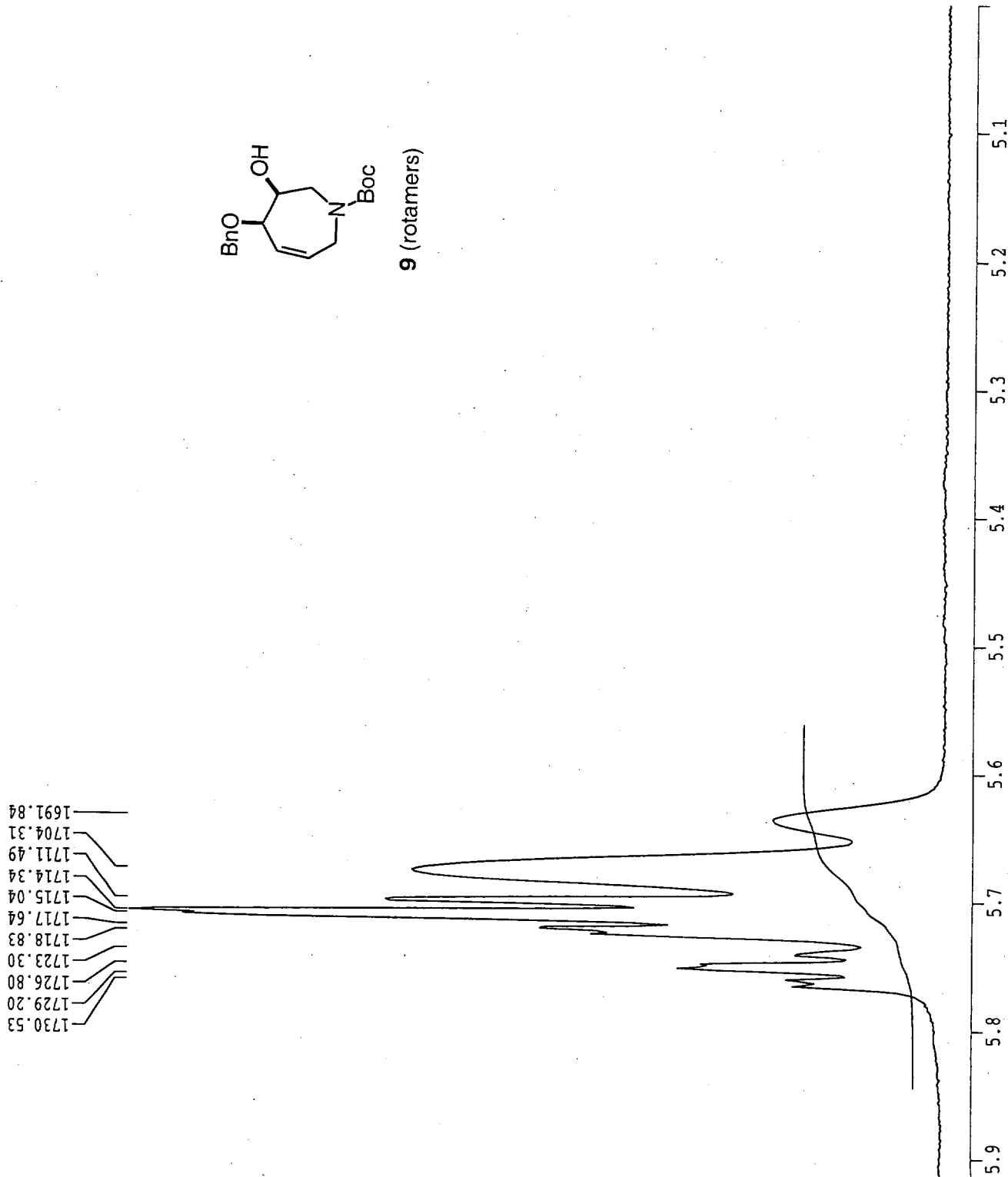
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1D NMR plot parameters  
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 F1 1800.78 Hz  
 F2P 5.000 ppm  
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 PPMCM 0.04545 ppm/cm  
 HZCM 13.64227 Hz/cm



9 (rotamers)



Current Data Parameters  
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 DS 0  
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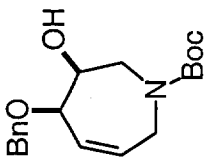
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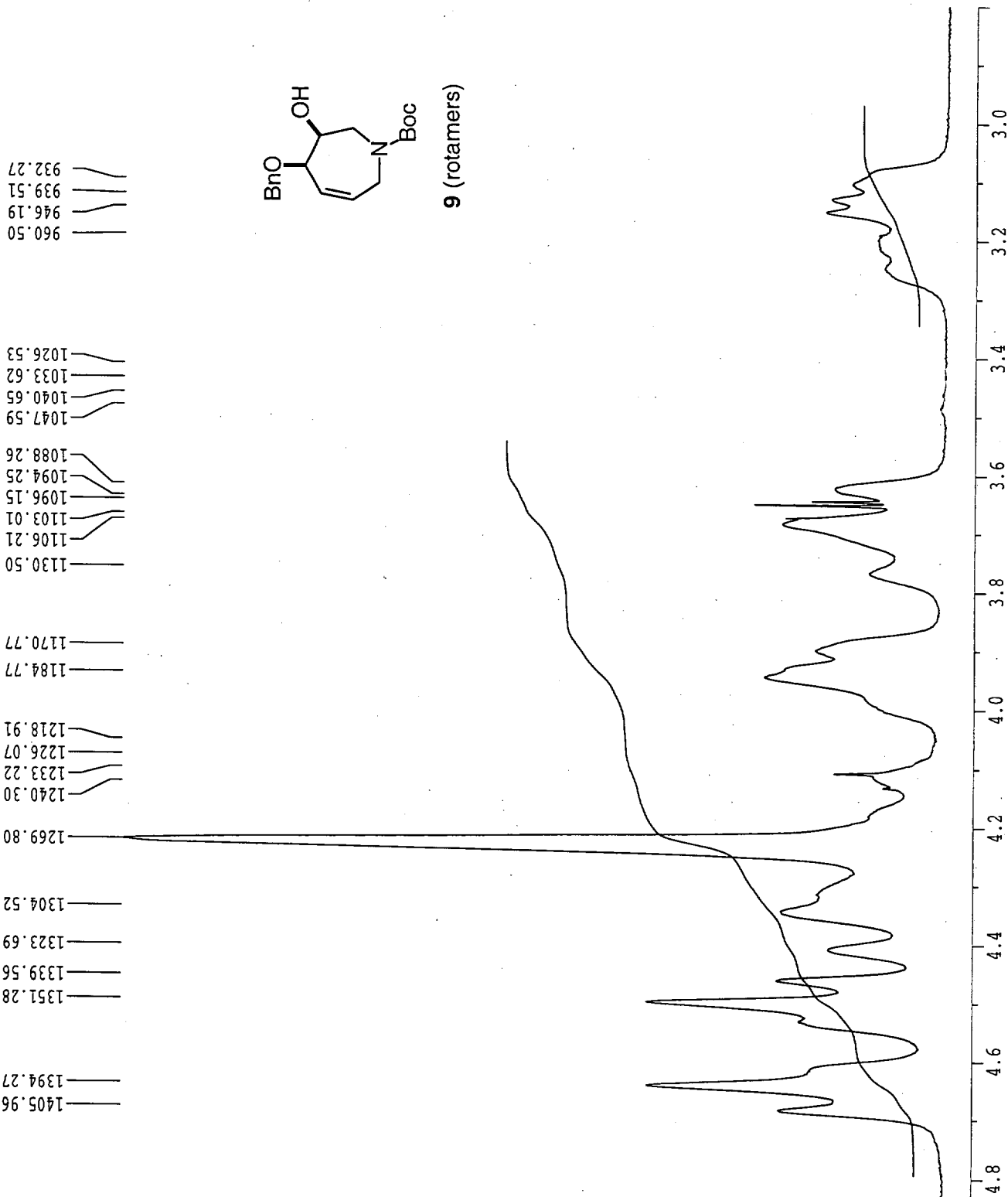
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9 (rotamers)



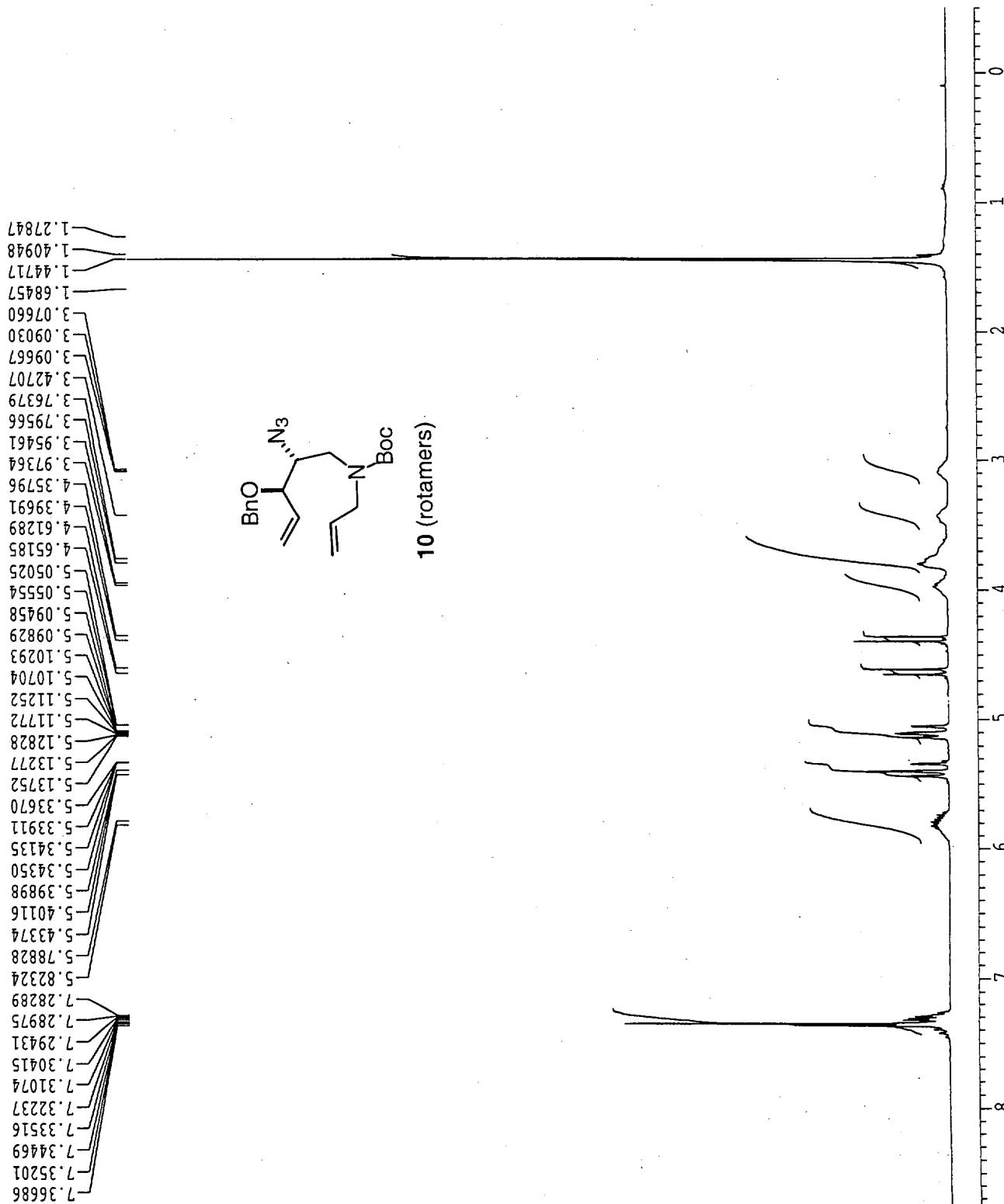
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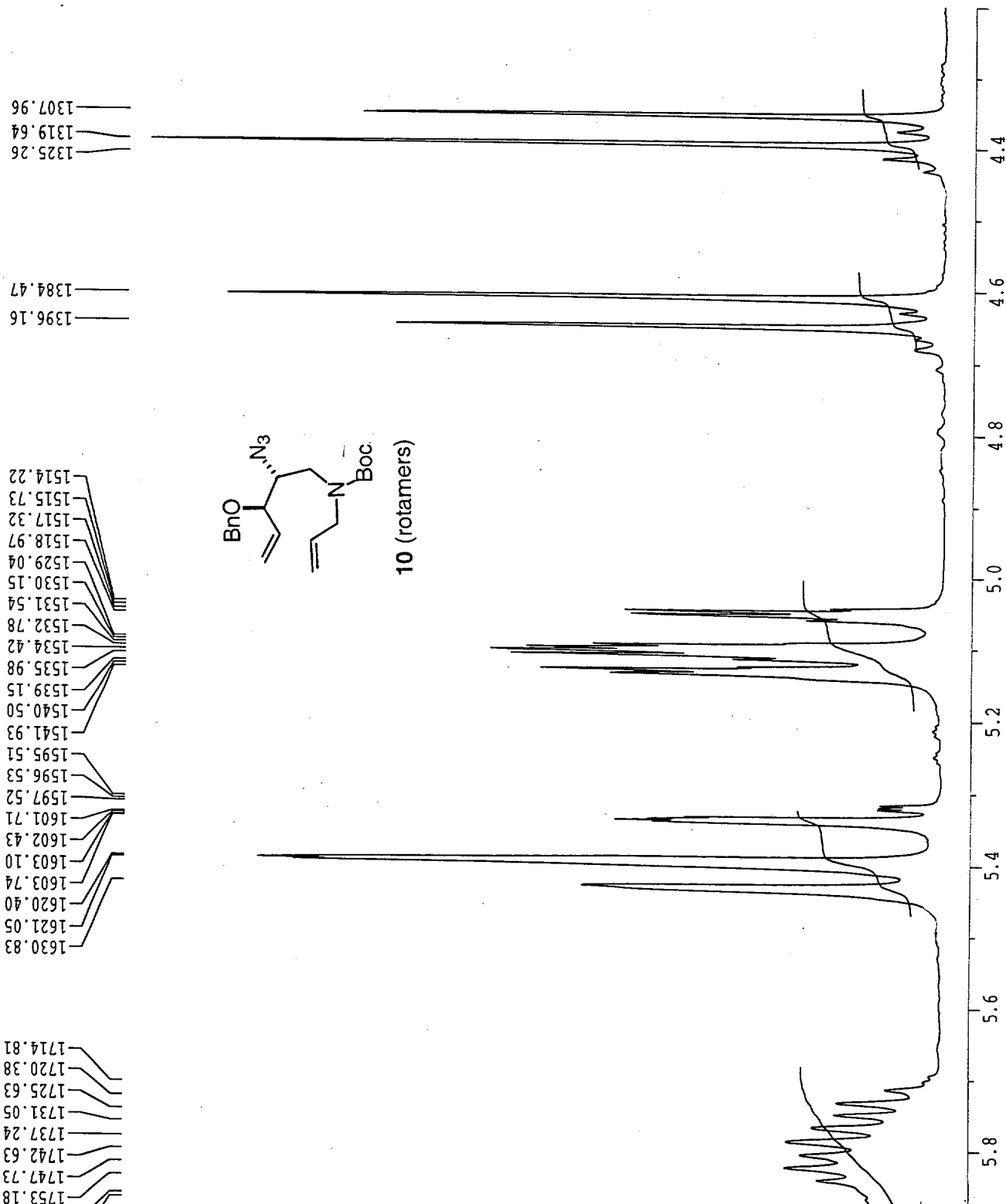
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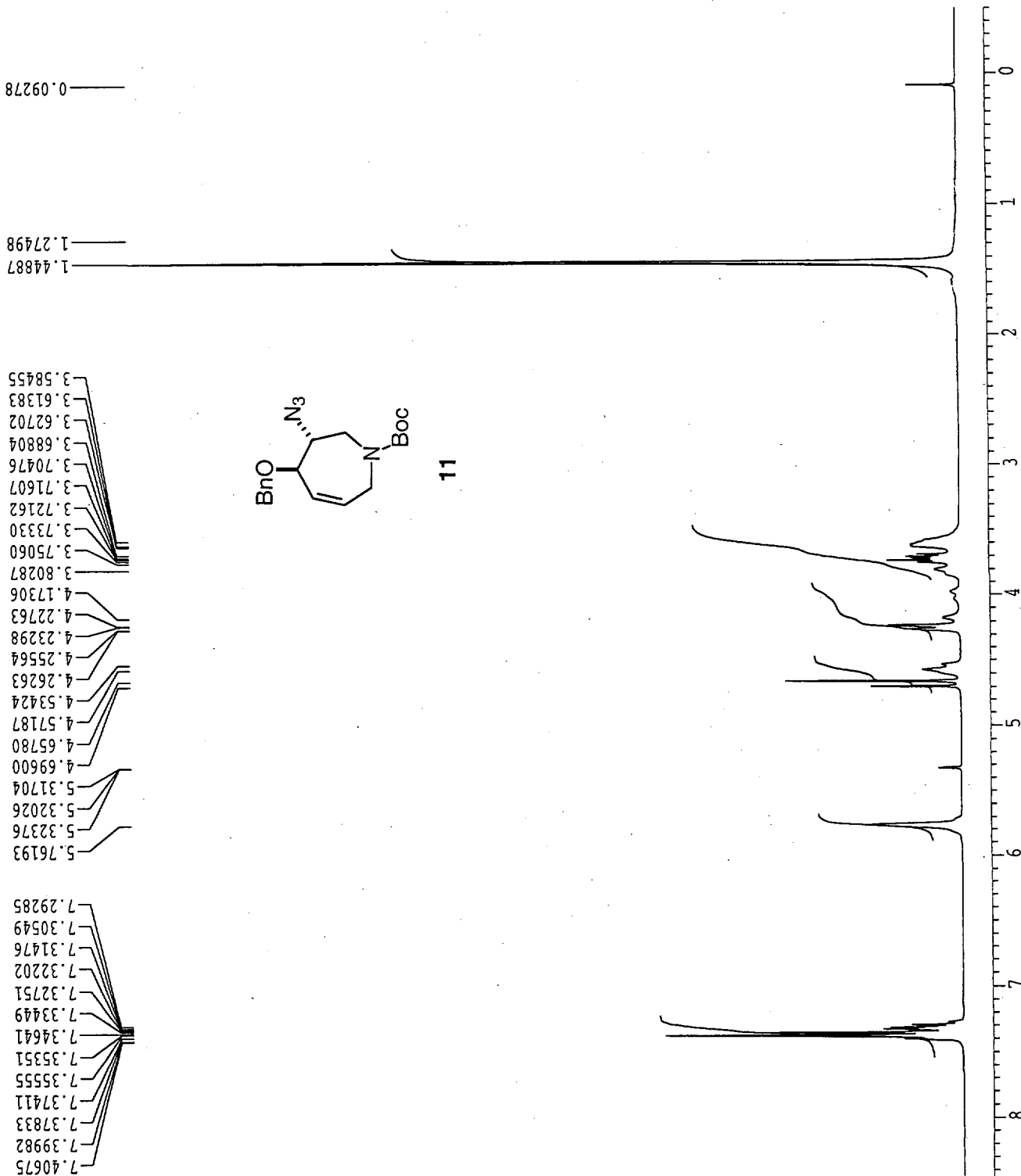
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 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 203.2  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.00000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SF01 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300103 MHz  
 SR 10.31 Hz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.00 cm  
 FIP 9.500 ppm  
 F1 2851.24 Hz  
 F2P -0.500 ppm  
 F2 -150.07 Hz  
 PPMCM 0.45455 ppm/cm  
 HZCM 136.42273 Hz/cm



THL-TB-015-02

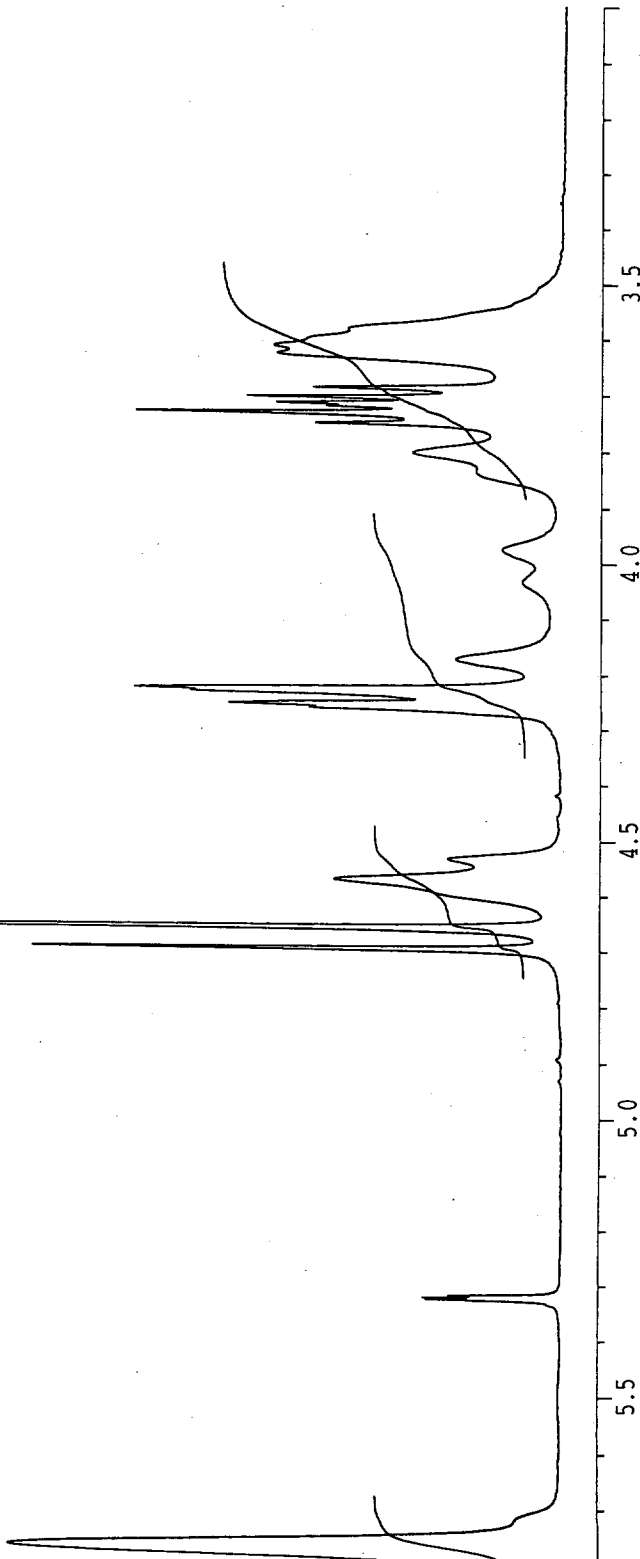
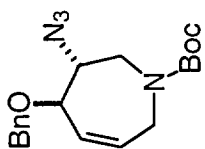
Current Data Parameters  
 NAME ag20032  
 EXPNO 10  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990821  
 Time 14.52  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zg30  
 TD 32768  
 SOLVENT CD2Cl2  
 NS 32  
 DS 0  
 SWH 6172.839 Hz  
 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 203.2  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.0000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SFO1 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300103 MHz  
 SR 10.31 Hz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 6.000 ppm  
 F1 1800.78 Hz  
 F2P 3.000 ppm  
 F2 900.39 Hz  
 PPMCM 0.13636 ppm/cm  
 HZCM 40.92682 Hz/cm

1729.33  
 1597.82  
 1596.77  
 1595.80  
 1409.41  
 1397.94  
 1372.15  
 1360.86  
 1279.34  
 1277.25  
 1270.44  
 1268.84  
 1252.46  
 1210.43  
 1193.12  
 1141.36  
 1125.67  
 1120.48  
 1116.97  
 1115.30  
 1111.91  
 1106.89  
 1088.58  
 1084.62  
 1075.83



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Current Data Parameters  
 NAME se01055  
 EXPNO 10  
 PROCNO 1  
 DU mpi  
 USER othiel

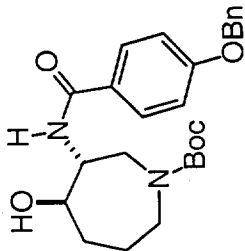
F2 - Acquisition Parameters  
 Date\_ 990903  
 Time 9.51  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zg30  
 TD 32768  
 SOLVENT CD2Cl2  
 NS 32  
 DS 0  
 SWH 6172.839 Hz  
 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 101.6  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.0000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SFO1 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300103 MHz  
 SR 10.33 Hz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

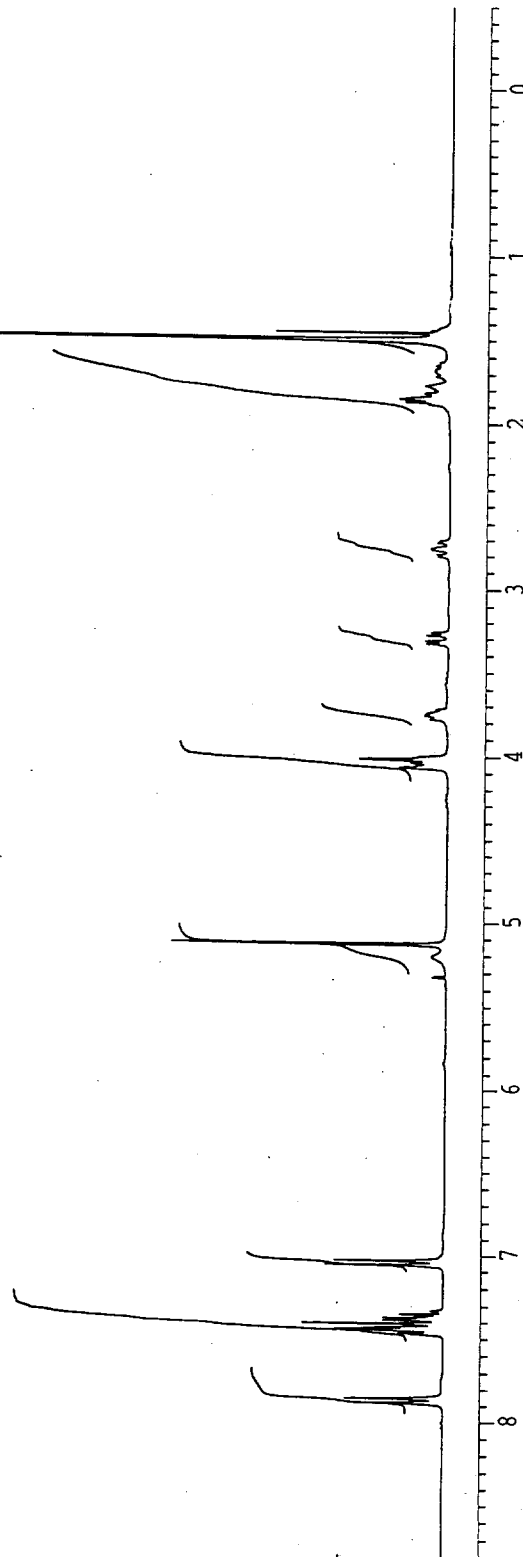
ID NMR plot parameters  
 CX 22.00 cm  
 FIP 9.500 ppm  
 F1 2851.24 Hz  
 F2P -0.500 ppm  
 F2 -150.07 Hz  
 PPMCM 0.45455 ppm/cm  
 HZCM 136.42273 Hz/cm

1.44340  
 1.45684  
 1.49852  
 1.77328  
 1.81621  
 1.82078  
 1.85231  
 1.87203  
 1.88616  
 2.75322  
 2.76029  
 3.26018  
 3.27744  
 3.31174  
 3.32879  
 3.73998  
 3.75045  
 3.75690  
 3.77824  
 3.99972  
 4.01421  
 4.03331  
 4.05236  
 4.06381  
 5.12711

7.02884  
 7.05840  
 7.34740  
 7.36390  
 7.36968  
 7.37579  
 7.38331  
 7.38738  
 7.40034  
 7.40651  
 7.41335  
 7.42105  
 7.42880  
 7.43388  
 7.44175  
 7.46077  
 7.46790  
 7.47383  
 7.85126  
 7.88074



13 (rotamers)



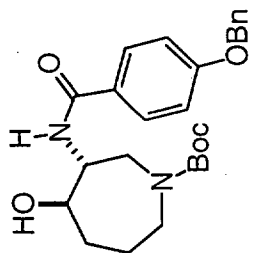
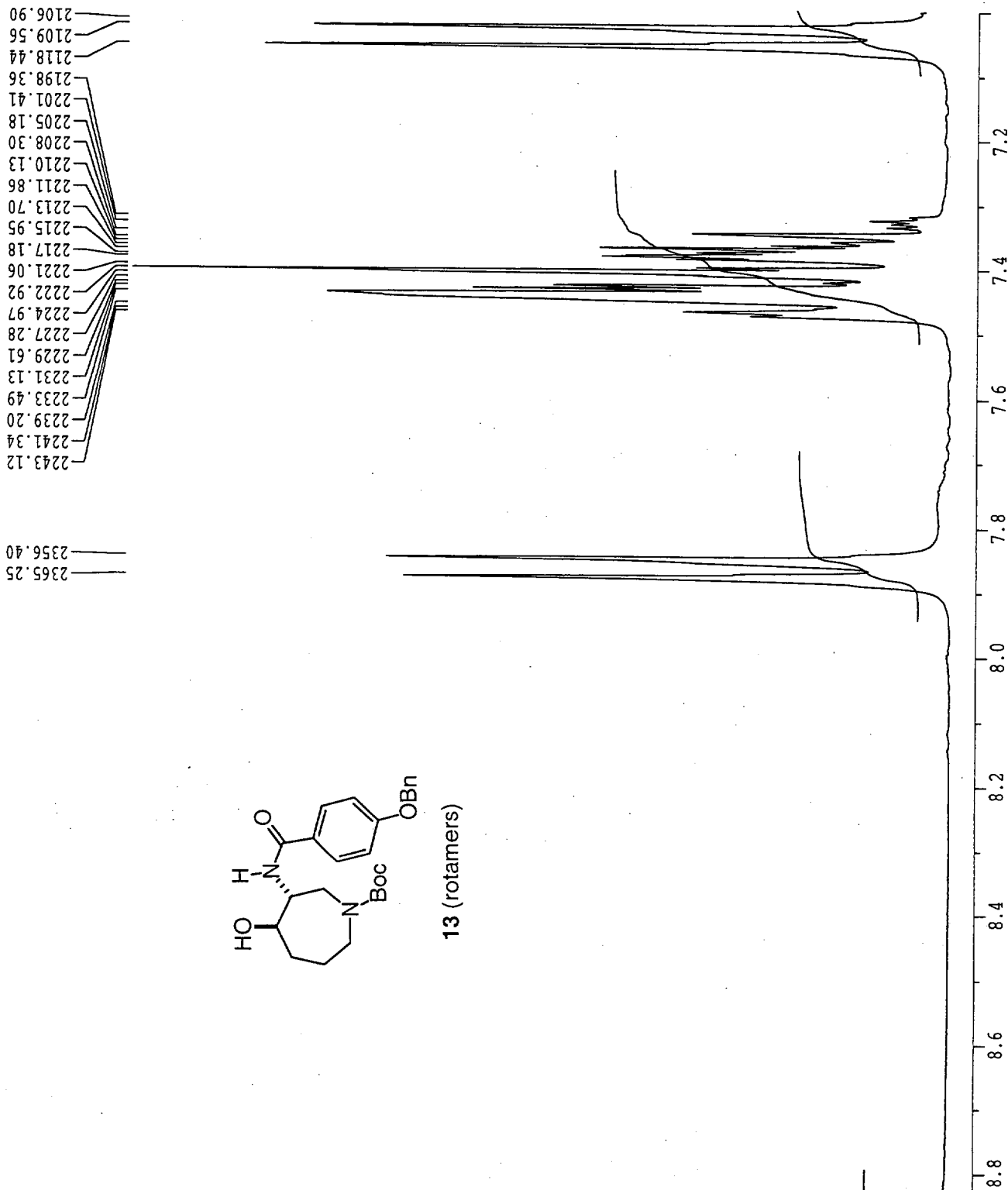
THL-TB-021-02

Current Data Parameters  
 NAME se01055  
 EXPNO 10  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990903  
 Time 9.51  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zg30  
 TD 32768  
 SOLVENT CD2Cl2  
 NS 32  
 DS 0  
 SWH 6172.839 Hz  
 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 101.6  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.00000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SFO1 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300103 MHz  
 SR 10.33 Hz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 9.000 ppm  
 F1 2701.17 Hz  
 F2P 7.000 ppm  
 F2 2100.91 Hz  
 PPMCM 0.09091 ppm/cm  
 HZCM 27.28455 Hz/cm

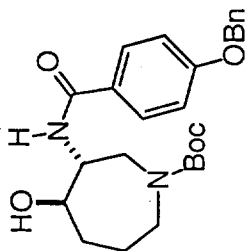


13 (rotamers)

THL-TB-021-02

168.384  
161.918  
161.893  
157.659  
136.969  
136.944  
129.340  
128.927  
128.906  
128.459  
128.436  
127.938  
127.914  
126.469  
114.918  
114.894

80.913  
79.802  
70.440  
70.415  
61.091  
54.524  
54.162  
53.802  
53.441  
53.081  
50.655  
50.092  
33.065  
28.472  
27.414

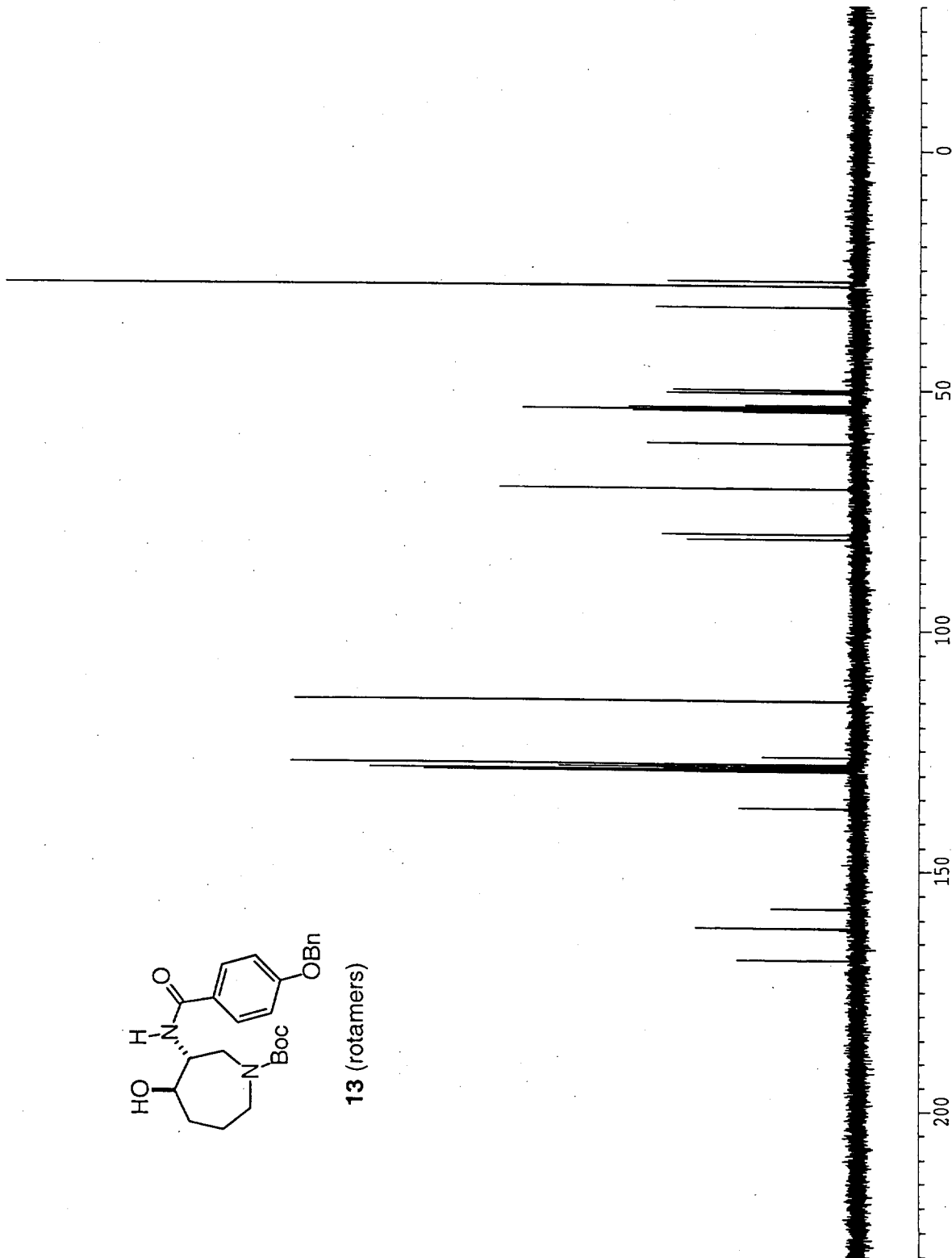


13 (rotamers)

F2 - Acquisition Parameters  
Date\_ 990903  
Time 4.03  
INSTRUM dpx300  
PROBHD 5 mm QNP  
PULPROG zgpg30  
TD 65536  
SOLVENT CD2Cl2  
NS 1200  
DS 2  
SWH 21231.422 Hz  
FIDRES 0.323966 Hz  
AQ 1.5434228 sec  
RG 16384  
DW 23.550 usec  
DE 4.50 usec  
TE 300.0 K  
D11 0.0300000 sec  
CPDPRG waltz16  
P31 80.00 usec  
S2 83 dB  
HL1 90 dB  
D1 0.0300000 sec  
P1 6.60 usec  
DE 4.50 usec  
SF01 75.4760670 MHz  
NUCLEUS off

F2 - Processing parameters  
SI 32768  
SF 75.4679482 MHz  
SR 229.18 Hz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 22.00 cm  
F1P 250.000 ppm  
F1 18866.99 Hz  
F2P -30.000 ppm  
F2 -2264.04 Hz  
PPMCM 12.72727 ppm/cm  
HZCM 960.50116 Hz/cm



THL-TB-021-02

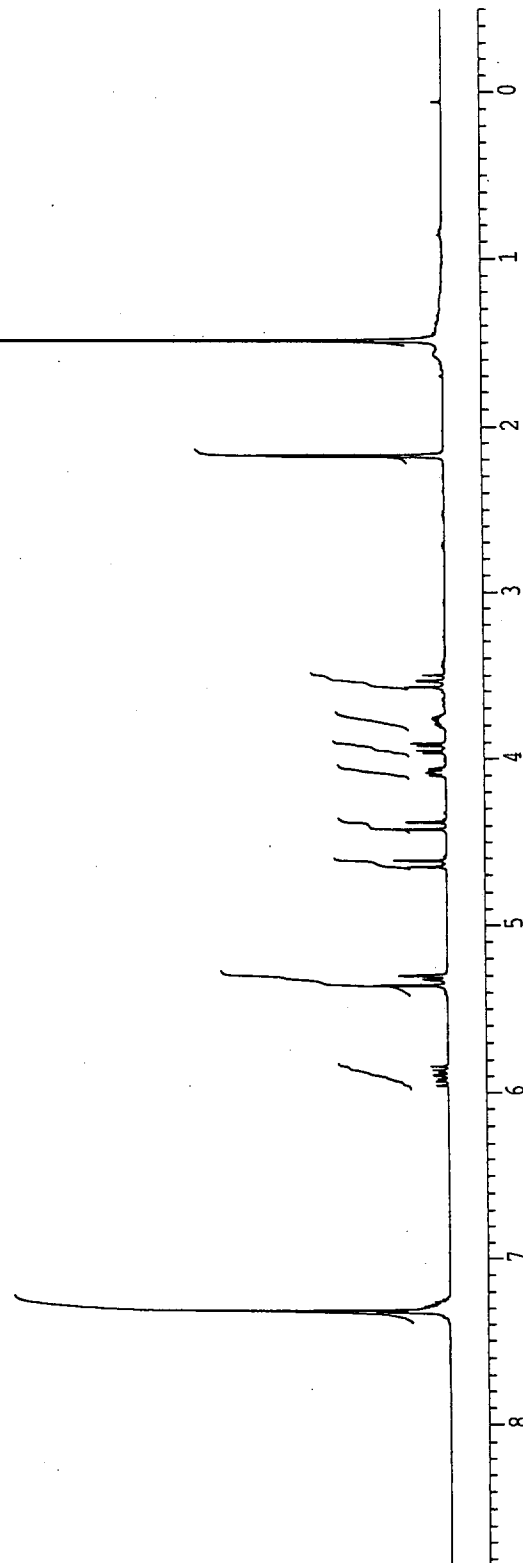
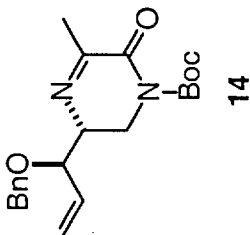
Current Data Parameters  
 NAME j130043  
 EXPNO 10  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990731  
 Time 12.31  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zg30  
 TD 32768  
 SOLVENT CD2Cl2  
 NS 32  
 DS 0  
 SMH 6172.839 Hz  
 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 362  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.0000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SF01 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300171 MHz  
 SR 17.10 Hz  
 EM  
 WDW 0  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.00 cm  
 FIP 9.500 ppm  
 F1 2851.24 Hz  
 F2P -0.500 ppm  
 F2 -150.07 Hz  
 PPMCM 0.45455 ppm/cm  
 HZCM 136.42273 Hz/cm

7.34202  
7.32873  
7.32456  
7.31180  
7.30318  
7.28890  
7.28080  
7.27418  
7.26054  
5.89808  
5.89039  
5.86044  
5.83593  
5.3548  
5.32478  
5.32205  
5.31993  
5.30129  
5.29792  
5.29510  
4.65433  
4.61465  
4.41882  
4.37930  
4.10028  
4.08473  
4.08120  
4.07597  
4.06024  
3.96356  
3.94921  
3.91996  
3.90545  
3.76072  
3.75316  
3.57429  
3.54003  
3.53066  
3.49631  
2.18091  
2.17336  
1.56892  
1.52867  
1.48892  
1.46891  
1.44573  
1.42727  
1.39973  
1.38109  
1.29183  
1.24284  
1.23401  
1.21119  
0.06075



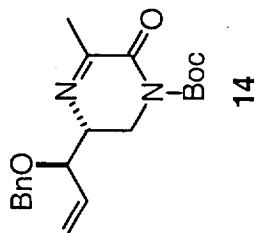
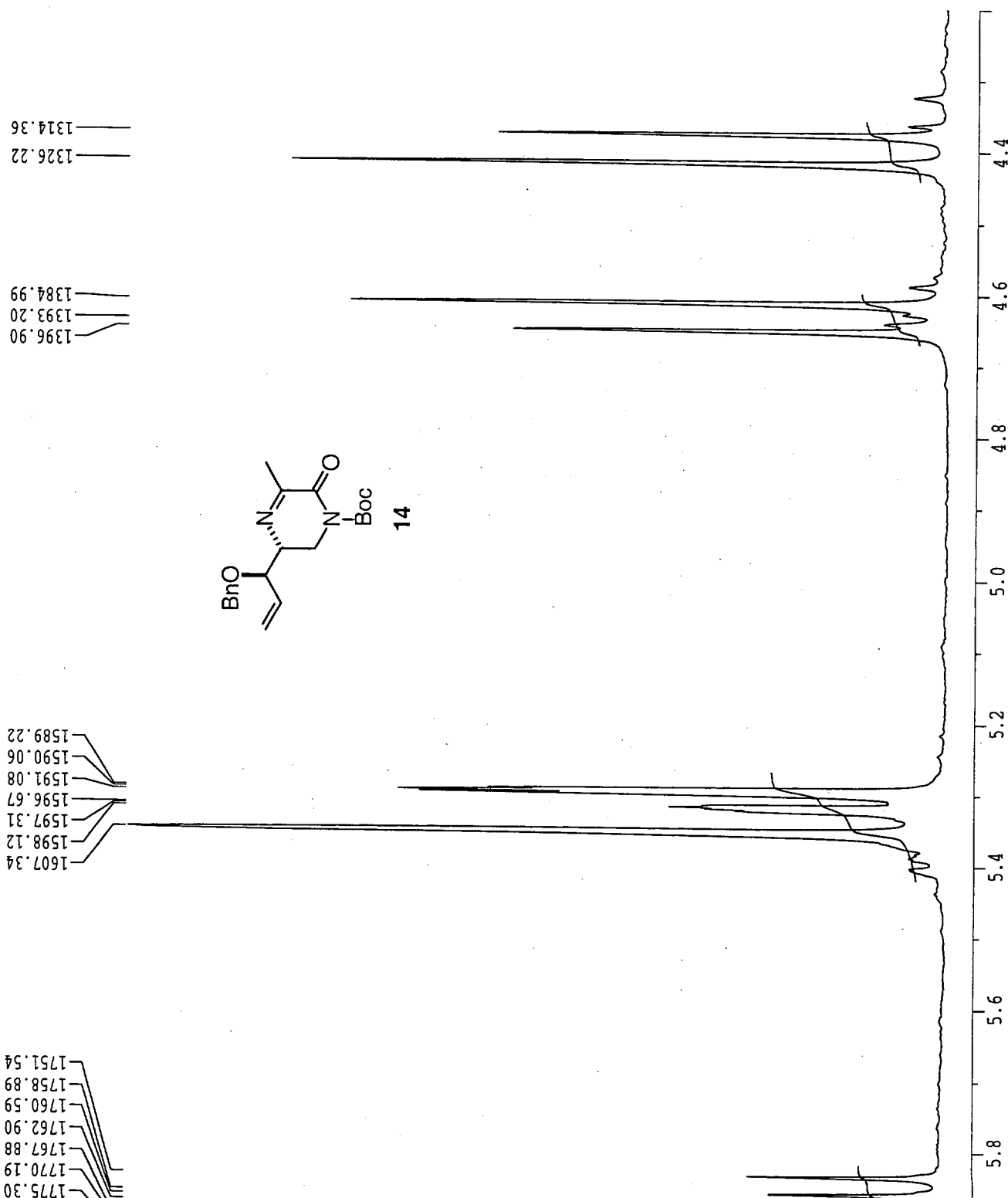
THL-TA-194-02

Current Data Parameters  
 NAME j130043  
 EXPNO 10  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990731  
 Time 12.31  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zg30  
 TD 32768  
 SOLVENT CD2Cl2  
 NS 32  
 DS 0  
 SMH 6172.839 Hz  
 FIDRES 0.188380 Hz  
 AQ 2.6542580 sec  
 RG 362  
 DW 81.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 HL1 90 dB  
 D1 2.0000000 sec  
 P1 7.30 usec  
 DE 4.50 usec  
 SFO1 300.1318534 MHz  
 NUCLEUS off

F2 - Processing parameters  
 SI 16384  
 SF 300.1300171 MHz  
 SR 17.10 Hz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 2.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 6.000 ppm  
 F1 1800.78 Hz  
 F2P 4.200 ppm  
 F2 1260.55 Hz  
 PPMCM 0.08182 ppm/cm  
 HZCM 24.55609 Hz/cm



THL-TA-194-02

Current Data Parameters  
 NAME j130043  
 EXPNO 11  
 PROCNO 1  
 DU mpi  
 USER othiel

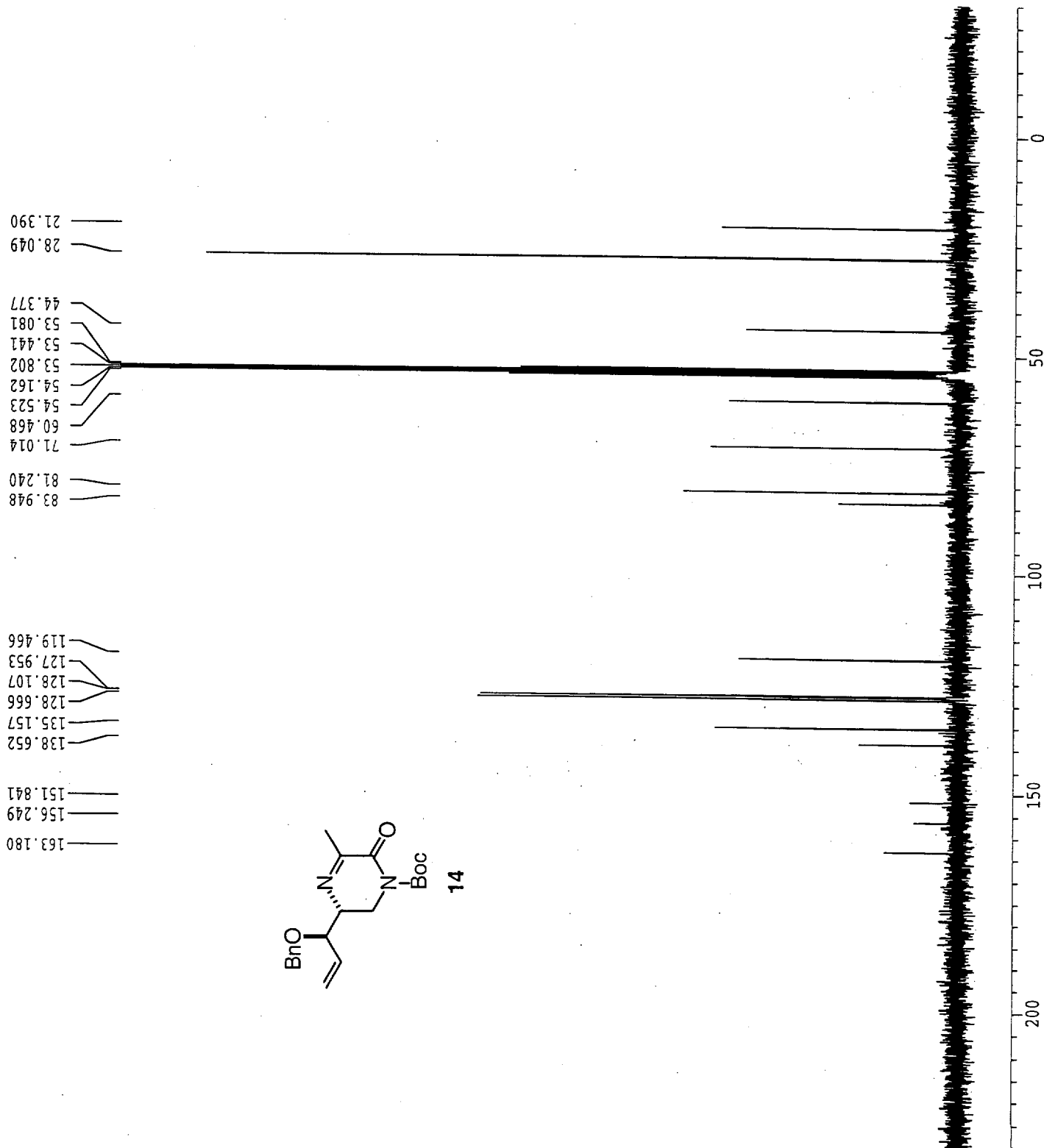
F2 - Acquisition Parameters

Date\_ 990731  
 Time 13.04  
 INSTRUM dpx300  
 PROBHD 5 mm QNP  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CD2Cl2  
 NS 1200  
 DS 2  
 SWH 21231.422 Hz  
 FIDRES 0.323966 Hz  
 AQ 1.5434228 sec  
 RG 16384  
 DW 23.550 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D11 0.03000000 sec  
 CPDPRG waltz16  
 P31 80.00 usec  
 S2 83 dB  
 HL1 90 dB  
 D1 0.03000000 sec  
 P1 6.60 usec  
 DE 4.50 usec  
 SF01 75.4760670 MHz  
 NUCLEUS off

F2 - Processing parameters

SI 32768  
 SF 75.4677196 MHz  
 SR 0.59 Hz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

ID NMR plot parameters  
 CX 22.00 cm  
 F1P 250.000 ppm  
 F1 18866.93 Hz  
 F2P -30.000 ppm  
 F2 -2264.03 Hz  
 PPMCM 12.72727 ppm/cm  
 HZCM 960.49823 Hz/cm

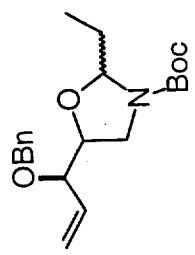
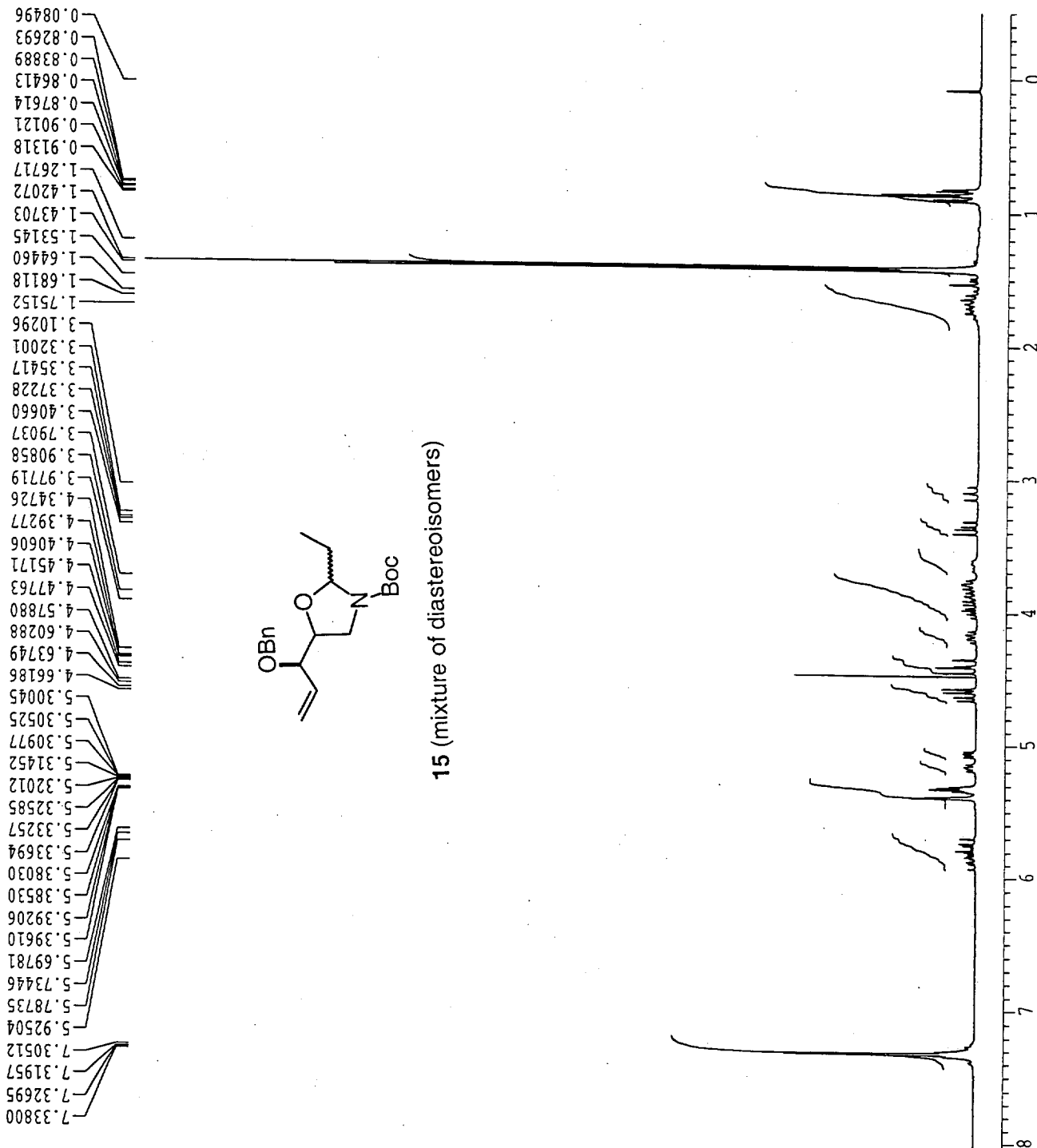


Current Data Parameters  
 NAME MR180F  
 EXPNO 113  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990318  
 Time 21.22  
 INSTRUM ac200  
 PROBHD X59.AU  
 PULPROG 32768  
 TD unknown  
 SOLVENT unknown  
 NS 150  
 DS 0  
 SWH 4032.258 Hz  
 FIDRES 0.123055 Hz  
 AQ 4.0632820 sec  
 RG 64  
 DW 124.000 usec  
 DE 155.00 usec  
 TE 300.0 K  
 F1 10.10 usec  
 HL1 83 dB  
 D1 1.00000000 sec  
 DE 155.00 usec  
 SFO1 200.1336240 MHz  
 NUCLEUS unknown

F2 - Processing parameters  
 SI 16384  
 SF 200.1327279 MHz  
 SR 2727.88 Hz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 4.00

1D NMR plot parameters  
 CX 22.00 cm  
 F1P 9.500 ppm  
 F1 1901.26 Hz  
 F2P -0.500 ppm  
 F2 -100.07 Hz  
 PPMCM 0.45455 ppm/cm  
 HZCM 90.96942 Hz/cm



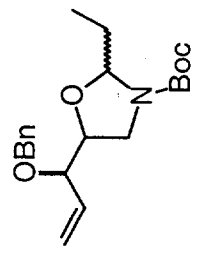
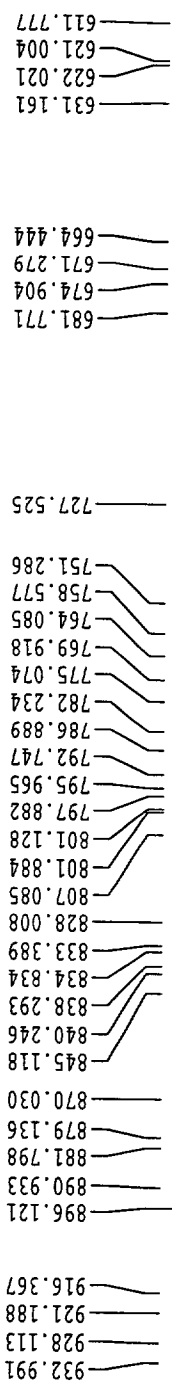
THL-TA-103-03

Current Data Parameters  
 NAME MRI80F  
 EXPNO 113  
 PROCNO 1  
 DU mpi  
 USER othiel

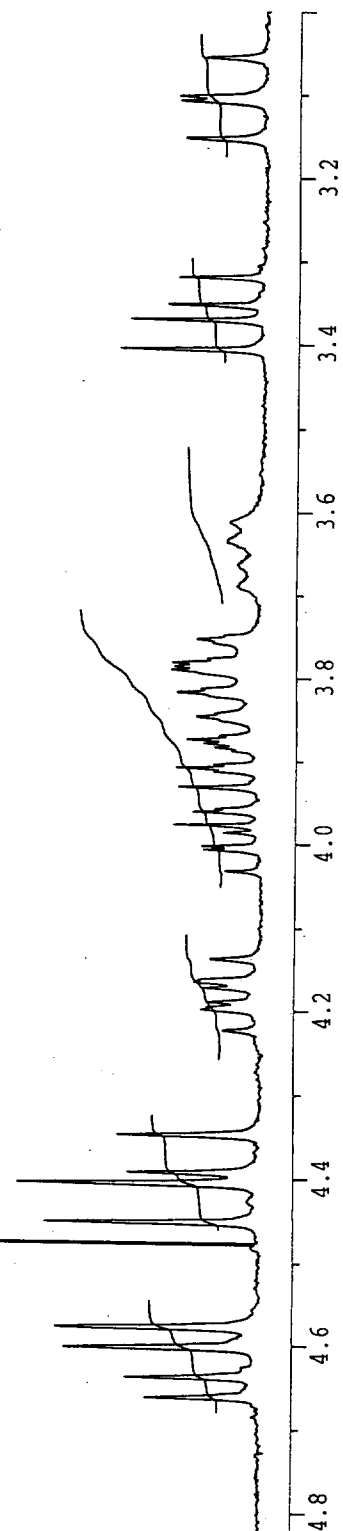
F2 - Acquisition Parameters  
 Date\_ 990318  
 Time 21.22  
 INSTRUM ac200  
 PROBHD X59.AU  
 PULPROG 32768  
 TD unknown  
 SOLVENT unknown  
 NS 150  
 DS 0  
 SWH 4032.258 Hz  
 FIDRES 0.123055 Hz  
 AQ 4.0632820 sec  
 RG 64  
 DW 124.000 usec  
 DE 155.00 usec  
 TE 300.0 K  
 P1 10.10 usec  
 HL1 83 dB  
 DE 1.00000000 sec  
 DE 155.00 usec  
 SFO1 200.1336240 MHz  
 NUCLEUS unknown

F2 - Processing parameters  
 SI 16384  
 SF 200.1327279 MHz  
 SR 2727.88 Hz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 4.00

ID NMR plot parameters  
 CX 22.00 cm  
 F1P 5.000 ppm  
 F1 1000.66 Hz  
 F2P 3.000 ppm  
 F2 600.40 Hz  
 PPMCM 0.09091 ppm/cm  
 HZCM 18.19388 Hz/cm



15 (mixture of diastereoisomers)





Current Data Parameters  
 NAME MR180F  
 EXPNO 113  
 PROCNO 1  
 DU mpi  
 USER othiel

F2 - Acquisition Parameters  
 Date\_ 990318  
 Time 21.22  
 INSTRUM ac200

PROBHD X59.AU  
 PULPROG 32768  
 TD unknown  
 SOLVENT unknown  
 NS 150  
 DS 0  
 SWH 4032.258 Hz  
 FIDRES 0.123055 Hz  
 AQ 4.0632820 sec  
 RG 64  
 DW 124.000 usec  
 DE 155.00 usec  
 TE 300.0 K  
 P1 10.10 usec  
 H1 83 dB  
 D1 1.0000000 sec  
 DE 155.00 usec  
 SFO1 200.1336240 MHz  
 NUCLEUS unknown

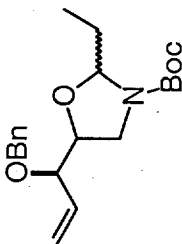
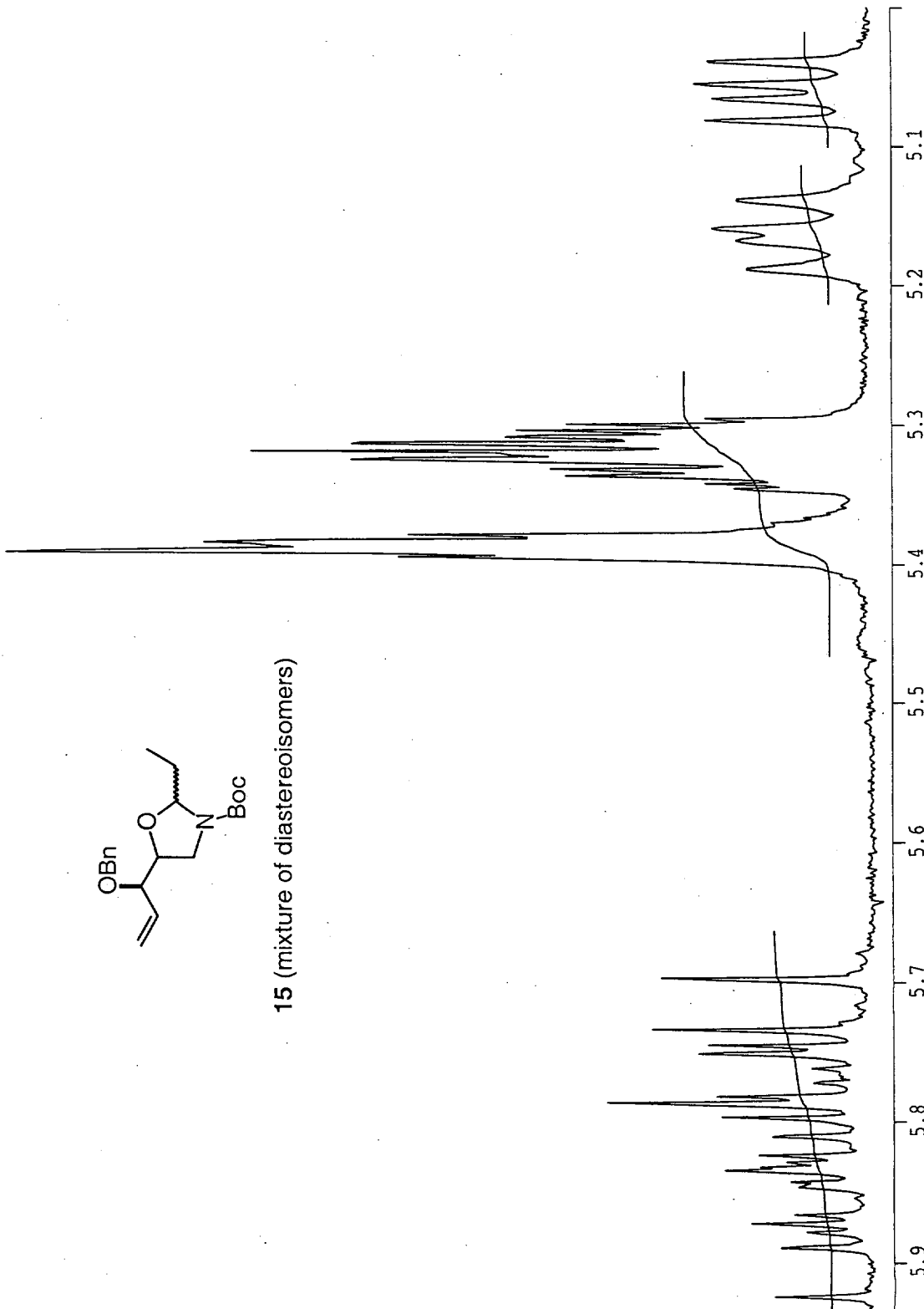
F2 - Processing parameters  
 SI 16384  
 SF 200.1327279 MHz  
 SR 2727.88 Hz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 4.00

1D NMR plot parameters  
 CX 22.00 cm  
 FIP 6.000 ppm  
 F1 1200.80 Hz  
 F2P 5.000 ppm  
 F2 1000.66 Hz  
 PPMCM 0.04545 ppm/cm  
 HZCM 9.09694 Hz/cm

1079.94  
 1079.13  
 1077.77  
 1076.77  
 1069.14  
 1068.10  
 1067.22  
 1065.88  
 1064.73  
 1063.61  
 1062.66  
 1061.76  
 1060.79

1038.51  
 1032.73  
 1028.66

1017.20  
 1014.06  
 1011.85  
 1008.70



15 (mixture of diastereoisomers)

1185.79  
 1178.61  
 1175.23  
 1174.01  
 1169.35  
 1167.83  
 1165.65  
 1162.83  
 1160.23  
 1158.24  
 1157.24  
 1151.02  
 1149.88  
 1147.65  
 1140.32

THL-TA-103-03