# Mild Conditions for Pd-Catalyzed Carboamination of Terminal-, α-Disubstituted-, and β-Disubstituted γ-Aminoalkenes. Scope, Limitations, and Mechanism of Pyrrolidine Formation.

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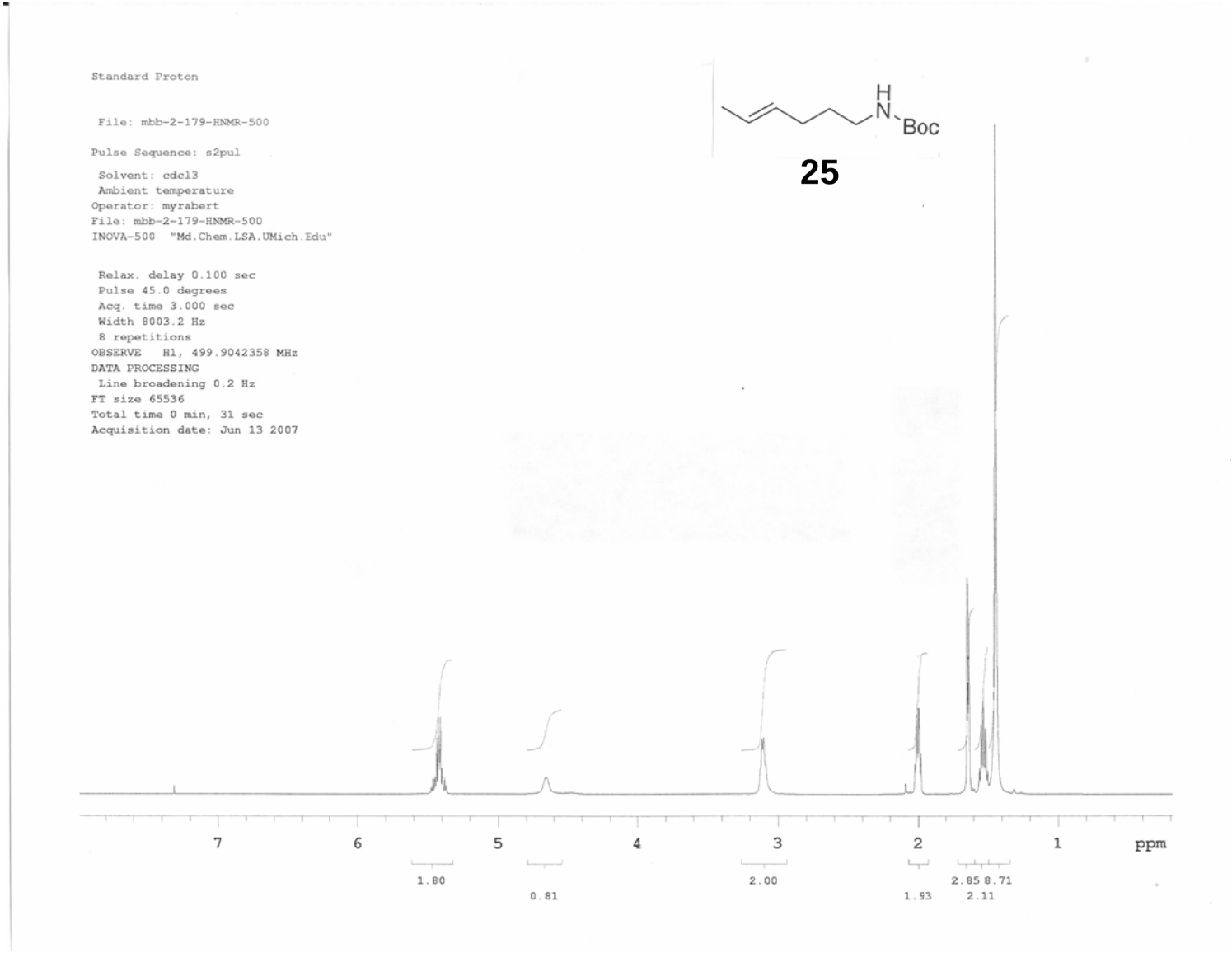
## Supporting Information

Experimental procedures and characterization data for new compounds in Tables 1–5 and equations 2–12 (146 pages).

## Table of Contents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Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-2-179-HNMR-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042358 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jun 13 2007
H
N
Boc
25
7
6
5
4
3
2
1
ppm
1.80
0.81
2.00
1.93
2.85
8.71
2.11

Standard Carbon

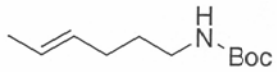
File: mbb-2-179-C13-500

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-2-179-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
224 repetitions
OBSERVE C13, 125.7010313 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 min, 18 sec
Acquisition date: Jun 13 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19594.655 | 155.883 | 3.1 |
| 2 | 16370.286 | 130.232 | 35.7 |
| 3 | 15767.760 | 125.439 | 33.0 |
| 4 | 9906.820 | 78.813 | 3.7 |
| 5 | 9712.115 | 77.264 | 9.3 |
| 6 | 9679.894 | 77.007 | 9.6 |
| 7 | 9648.134 | 76.755 | 9.4 |
| 8 | 5032.753 | 40.037 | 13.7 |
| 9 | 3736.101 | 29.722 | 36.1 |
| 10 | 3559.347 | 28.316 | 148.5 |
| 11 | 2309.646 | 18.374 | 3.3 |
| 12 | 2232.316 | 17.759 | 47.9 |

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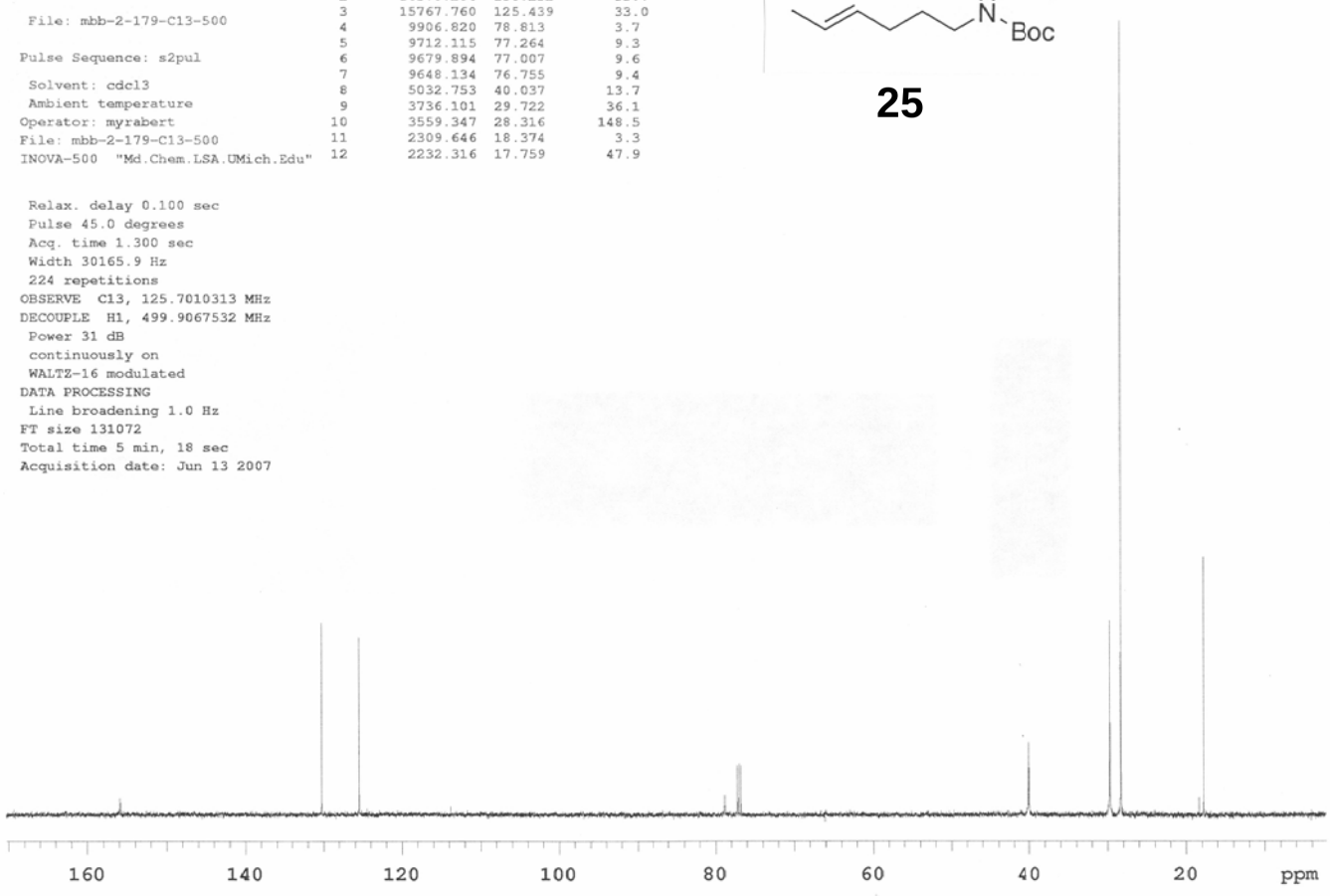


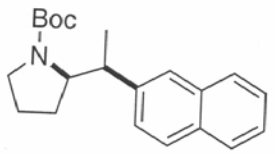
Standard Proton

File: mbb-9-63-co-iso-HNMR

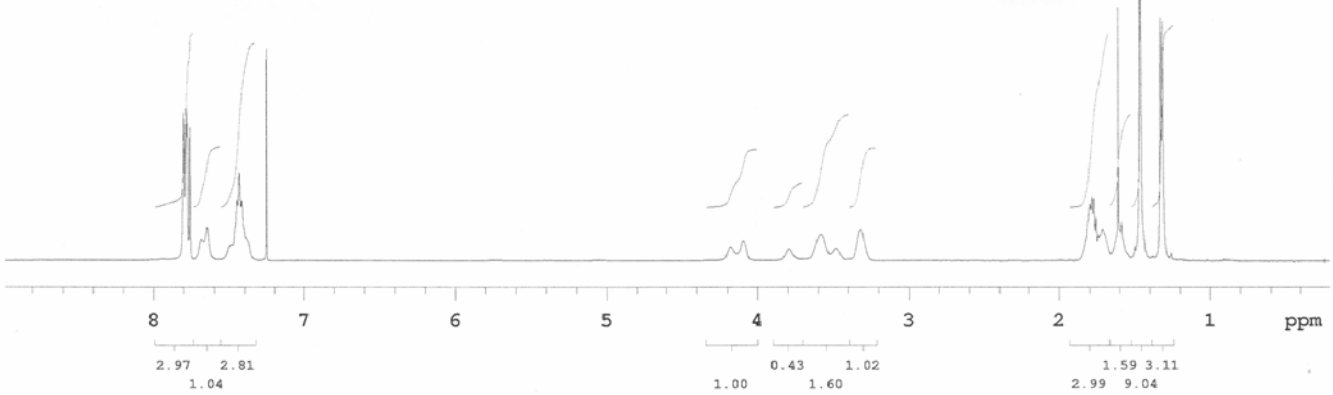
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-63-co-iso-HNMR
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
16 repetitions
OBSERVE H1, 499.9042639 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Acquisition date: Jun 18 2007




**26**



Standard Carbon

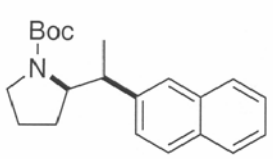
File: mbb-9-63-C13-500

Pulse Sequence: s2pul

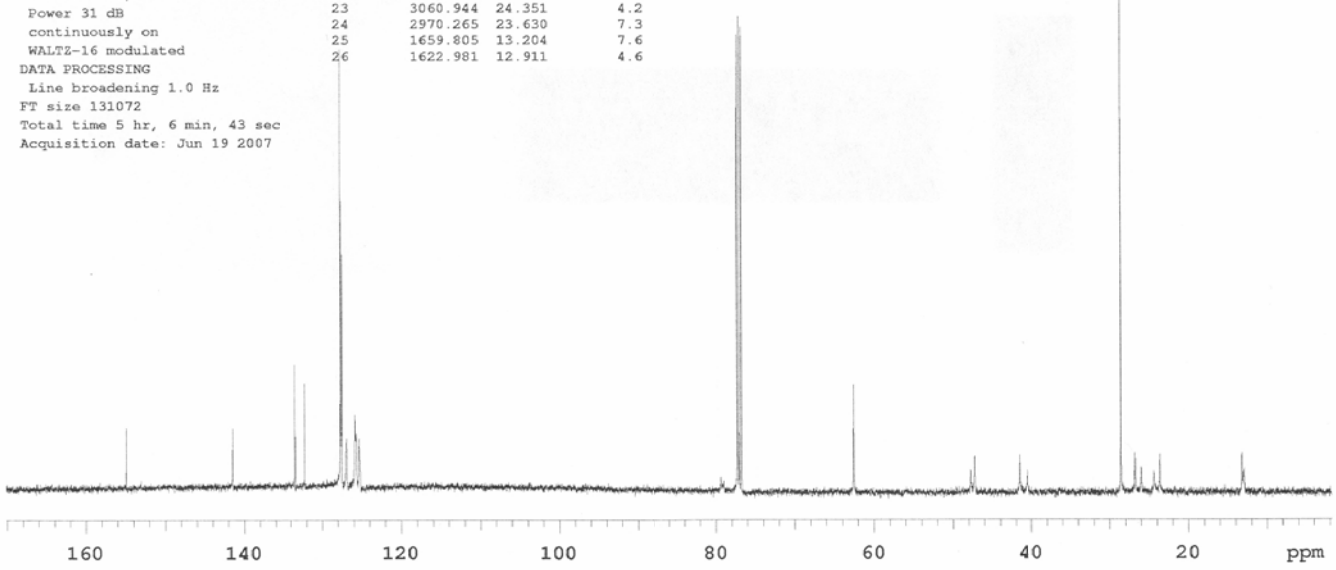
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-63-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
13056 repetitions
OBSERVE C13, 125.7010266 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 hr, 6 min, 43 sec
Acquisition date: Jun 19 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19469.550 | 154.888 | 12.3 |
| 2 | 19230.197 | 152.984 | 2.5 |
| 3 | 17784.409 | 141.482 | 12.2 |
| 4 | 16774.521 | 133.448 | 24.0 |
| 5 | 16619.402 | 132.214 | 20.5 |
| 6 | 16046.334 | 127.655 | 82.6 |
| 7 | 16027.922 | 127.508 | 54.2 |
| 8 | 15952.894 | 126.911 | 10.4 |
| 9 | 15821.710 | 125.868 | 14.7 |
| 10 | 15752.206 | 125.315 | 10.3 |
| 11 | 9974.579 | 79.352 | 3.1 |
| 12 | 9711.750 | 77.261 | 85.2 |
| 13 | 9679.990 | 77.008 | 88.7 |
| 14 | 9648.230 | 76.755 | 86.7 |
| 15 | 7865.046 | 62.569 | 20.2 |
| 16 | 5995.326 | 47.695 | 4.4 |
| 17 | 5930.425 | 47.179 | 7.0 |
| 18 | 5216.967 | 41.503 | 7.1 |
| 19 | 5093.147 | 40.518 | 4.3 |
| 20 | 3585.220 | 28.522 | 159.8 |
| 21 | 3364.278 | 26.764 | 7.5 |
| 22 | 3261.632 | 25.948 | 4.8 |
| 23 | 3060.944 | 24.351 | 4.2 |
| 24 | 2970.265 | 23.630 | 7.3 |
| 25 | 1659.805 | 13.204 | 7.6 |
| 26 | 1622.981 | 12.911 | 4.6 |




**26**



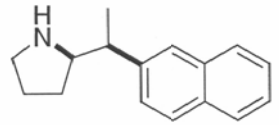
Standard Proton

File: mbb-9-74-HNMR

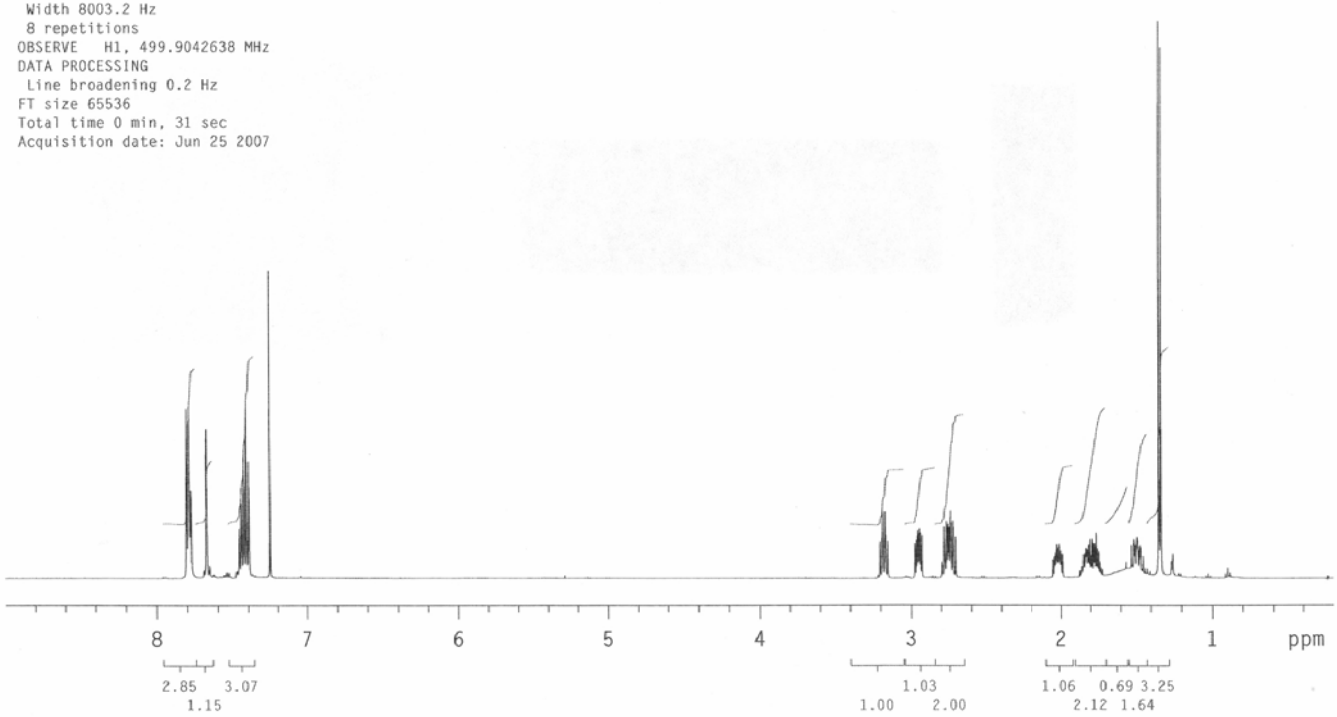
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-74-HNMR
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042638 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jun 25 2007




**26a**



Standard Carbon

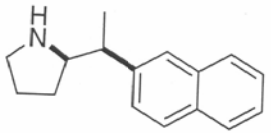
File: mbb-9-74-C13-500

Pulse Sequence: s2pul

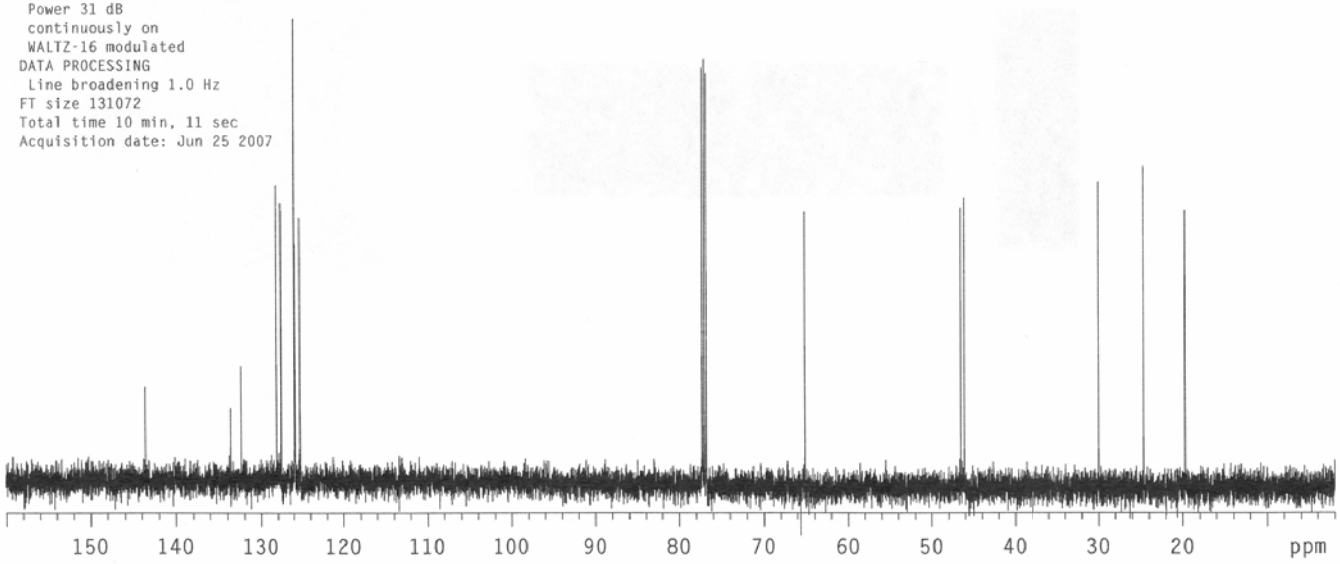
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-74-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

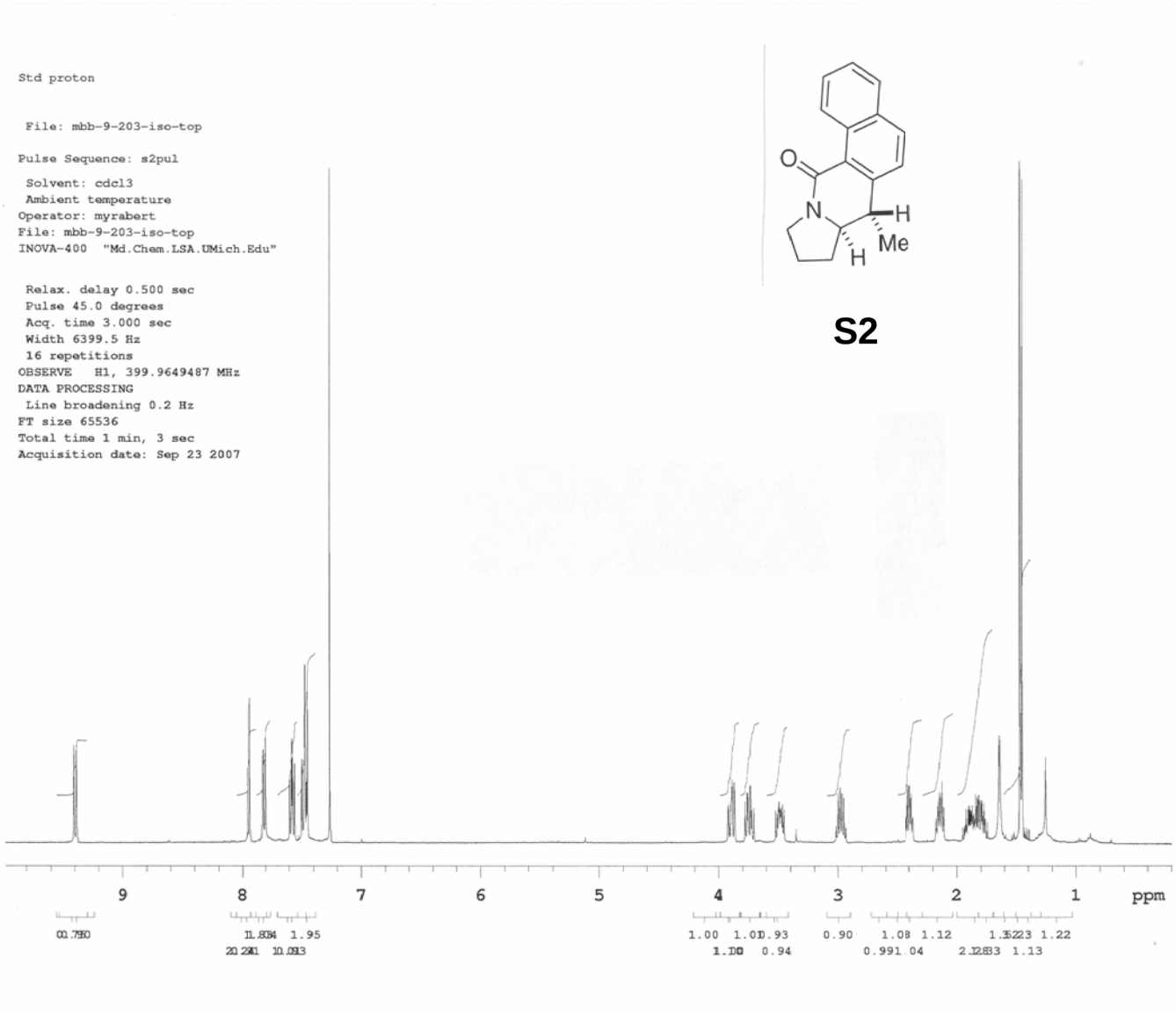
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
432 repetitions
OBSERVE C13, 125.7010264 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 10 min, 11 sec
Acquisition date: Jun 25 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 18043.686 | 143.544 | 18.2 |
| 2 | 16791.222 | 133.581 | 14.4 |
| 3 | 16635.642 | 132.343 | 22.0 |
| 4 | 16103.541 | 128.110 | 55.6 |
| 5 | 16040.481 | 127.608 | 52.3 |
| 6 | 16034.497 | 127.561 | 50.9 |
| 7 | 15823.221 | 125.880 | 86.6 |
| 8 | 15809.873 | 125.774 | 44.7 |
| 9 | 15739.447 | 125.213 | 49.6 |
| 10 | 9711.881 | 77.262 | 77.5 |
| 11 | 9679.660 | 77.005 | 79.1 |
| 12 | 9647.900 | 76.753 | 76.4 |
| 13 | 8255.046 | 65.672 | -9.2 |
| 14 | 8185.081 | 65.115 | 50.7 |
| 15 | 5848.162 | 46.524 | 51.3 |
| 16 | 5791.546 | 46.074 | 53.2 |
| 17 | 3768.548 | 29.980 | 56.2 |
| 18 | 3094.675 | 24.619 | 59.2 |
| 19 | 2476.959 | 19.705 | 51.0 |




**26a**

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Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-203-iso-top
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649487 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Sep 23 2007
O
N
H
H
Me
S2
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
0.93
0.90
1.08
1.12
1.22
0.94
0.99
1.04
1.13

Std Carbon

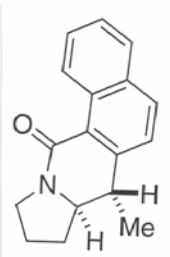
File: mbb-9-203-iso-top-C13-CDCl3-400

Pulse Sequence: s2pul

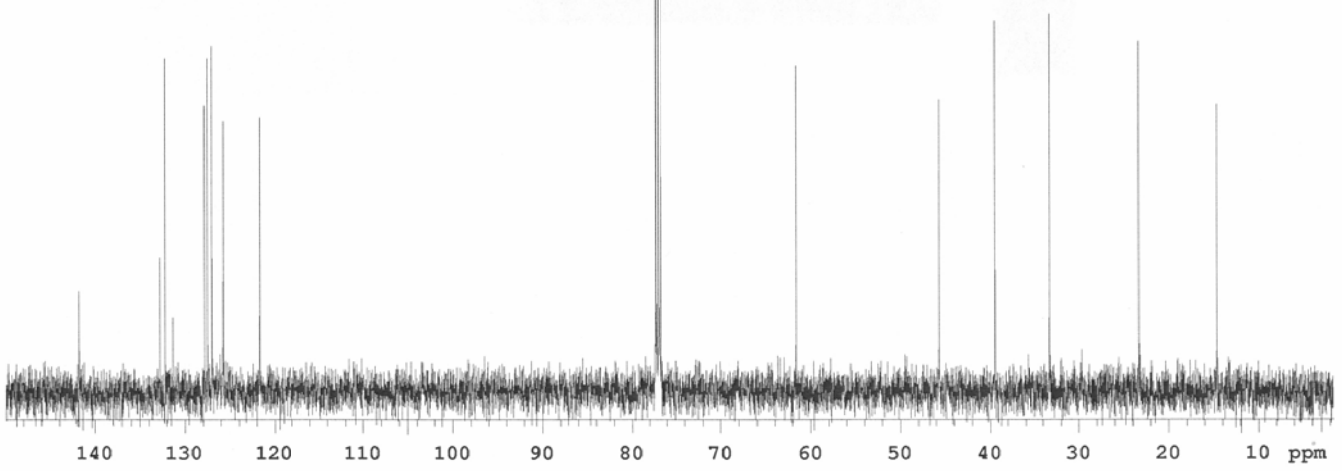
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-203-iso-top-C13-CDCl3-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

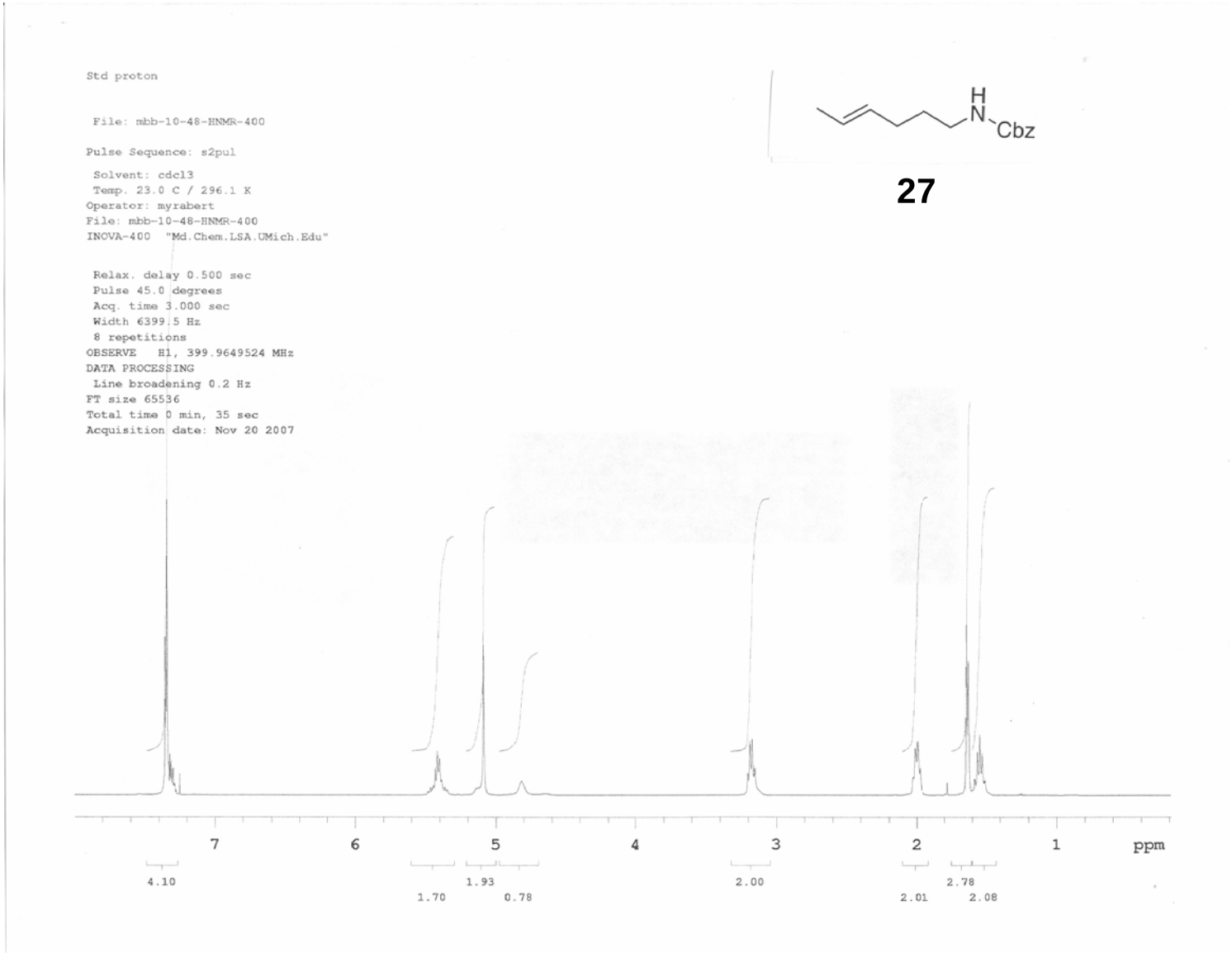
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
560 repetitions
OBSERVE C13, 100.5712668 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 13 min, 9 sec
Acquisition date: Sep 23 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 14260.219 | 141.792 | 19.1 |
| 2 | 13356.294 | 132.804 | 25.4 |
| 3 | 13295.149 | 132.196 | 62.2 |
| 4 | 13207.482 | 131.325 | 14.2 |
| 5 | 12854.605 | 127.816 | 53.4 |
| 6 | 12823.664 | 127.508 | 62.1 |
| 7 | 12770.622 | 126.981 | 64.4 |
| 8 | 12642.437 | 125.706 | 50.6 |
| 9 | 12232.835 | 121.633 | 51.2 |
| 10 | 7776.568 | 77.324 | 190.9 |
| 11 | 7744.891 | 77.009 | 188.1 |
| 12 | 7712.476 | 76.687 | 194.5 |
| 13 | 6195.622 | 61.604 | 60.8 |
| 14 | 4590.364 | 45.643 | 54.5 |
| 15 | 3964.910 | 39.424 | 69.2 |
| 16 | 3346.087 | 33.271 | 70.4 |
| 17 | 2341.235 | 23.279 | 65.3 |
| 18 | 1467.515 | 14.592 | 53.7 |




**S2**

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Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-48-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649524 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Nov 20 2007
H
N
Cbz
27
7
6
5
4
3
2
1
ppm
4.10
1.70
1.93
0.78
2.00
2.01
2.78
2.08

Std Carbon

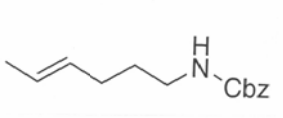
File: mbb-10-48-C13-400

Pulse Sequence: s2pul

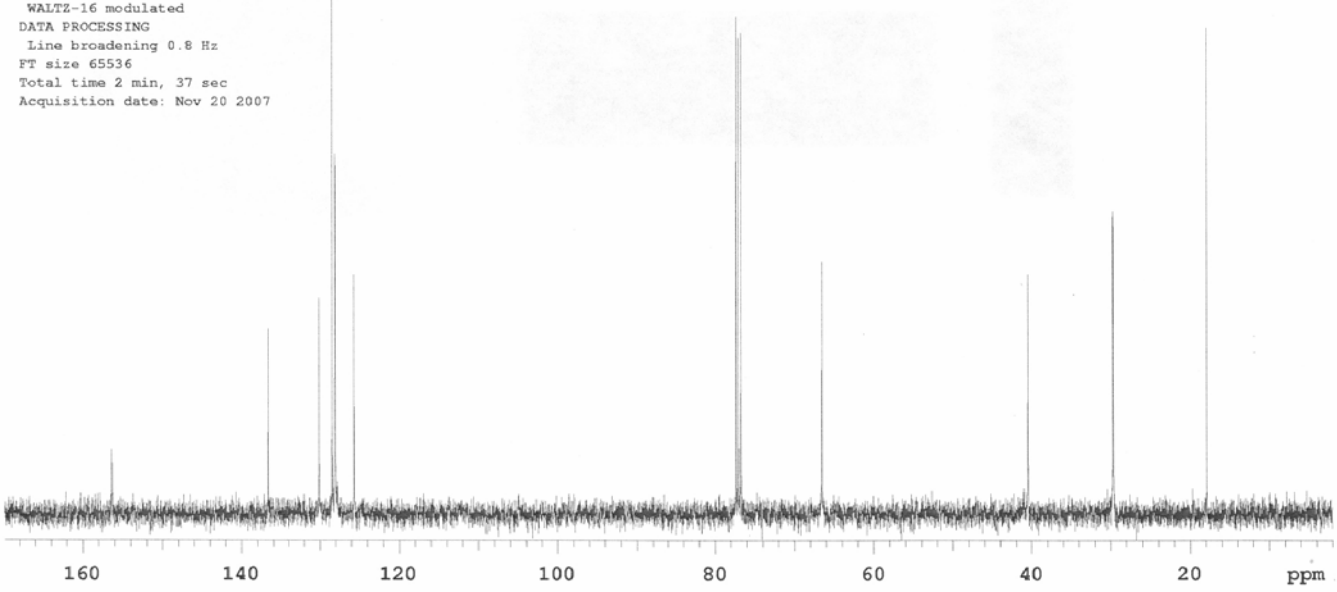
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-48-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

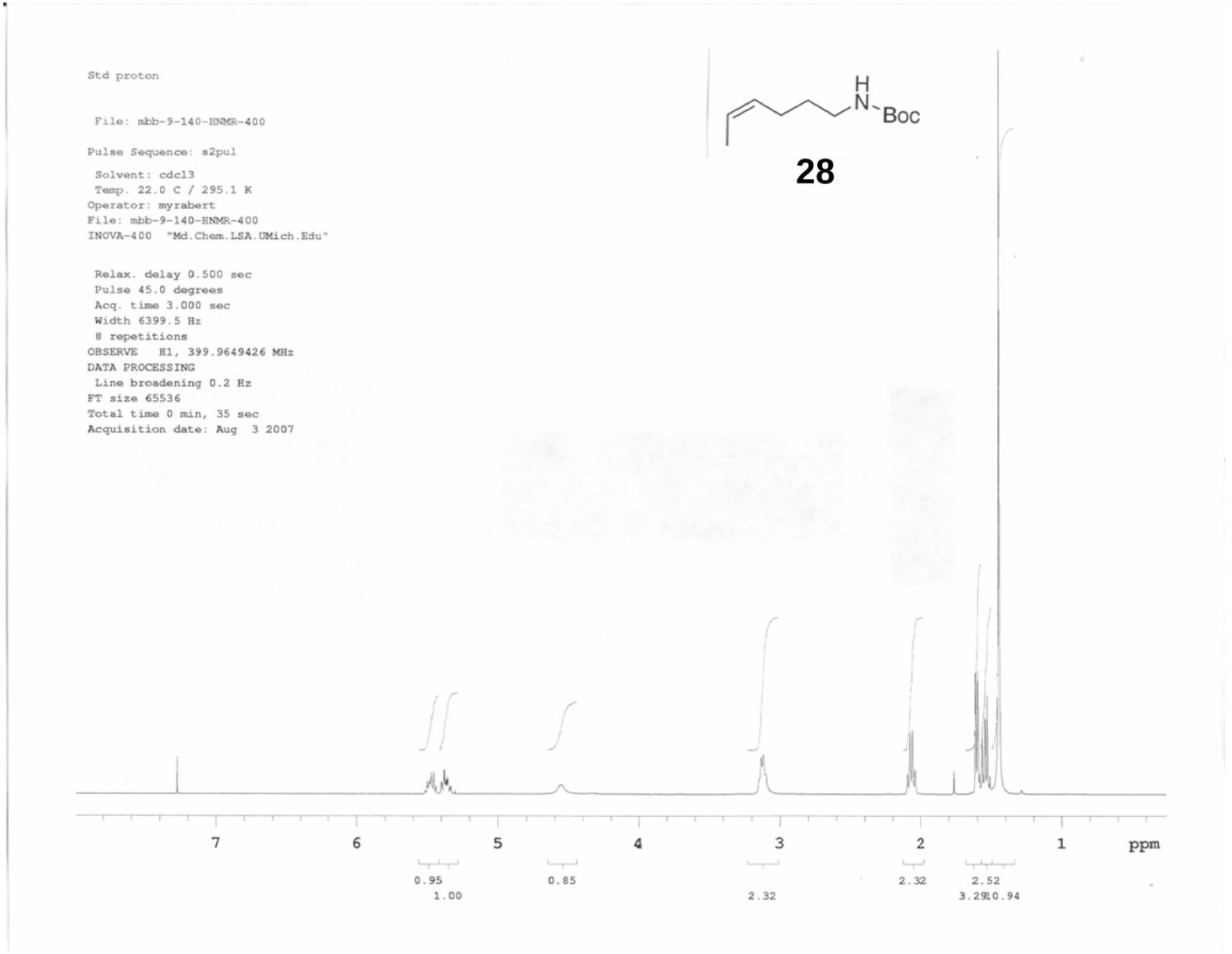
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
112 repetitions
OBSERVE C13, 100.5712728 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 2 min, 37 sec
Acquisition date: Nov 20 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15720.275 | 156.310 | 12.1 |
| 2 | 13738.566 | 136.605 | 34.6 |
| 3 | 13082.908 | 130.086 | 40.3 |
| 4 | 12917.152 | 128.438 | 151.7 |
| 5 | 12878.107 | 128.050 | 66.8 |
| 6 | 12873.687 | 128.006 | 64.7 |
| 7 | 12640.154 | 125.684 | 44.5 |
| 8 | 7776.496 | 77.323 | 92.4 |
| 9 | 7744.818 | 77.008 | 88.5 |
| 10 | 7712.403 | 76.686 | 89.4 |
| 11 | 6687.661 | 66.497 | 47.0 |
| 12 | 4075.342 | 40.522 | 44.6 |
| 13 | 2982.823 | 29.659 | 56.2 |
| 14 | 2978.403 | 29.615 | 53.9 |
| 15 | 1793.798 | 17.836 | 90.4 |




**27**




Std proton
File: mbb-9-140-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: myrabert
File: mbb-9-140-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649426 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Aug 3 2007
H
N
Boc
28
7
6
5
4
3
2
1
ppm
0.95
1.00
0.85
2.32
2.32
2.52
3.29
10.94

Std Carbon

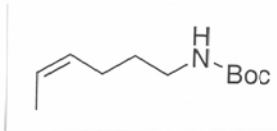
File: mbb-9-140-C13-400

Pulse Sequence: s2pul

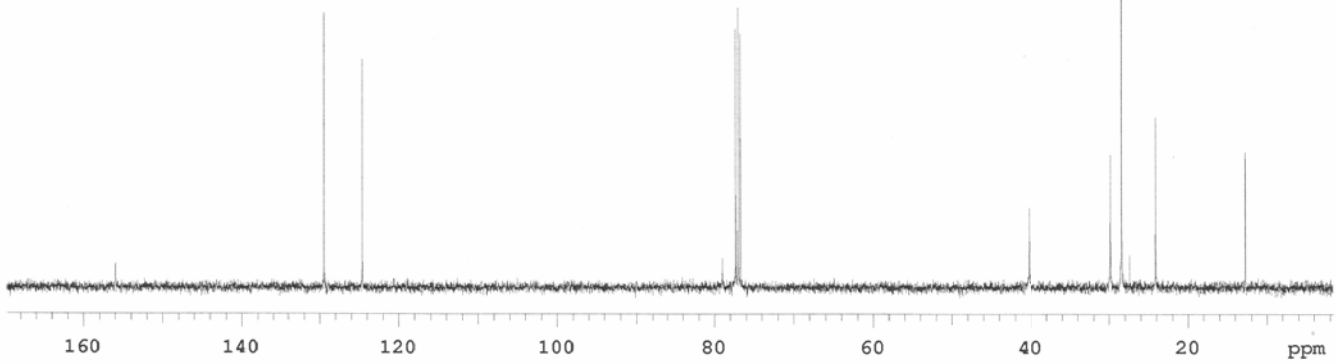
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: myrabert
File: mbb-9-140-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

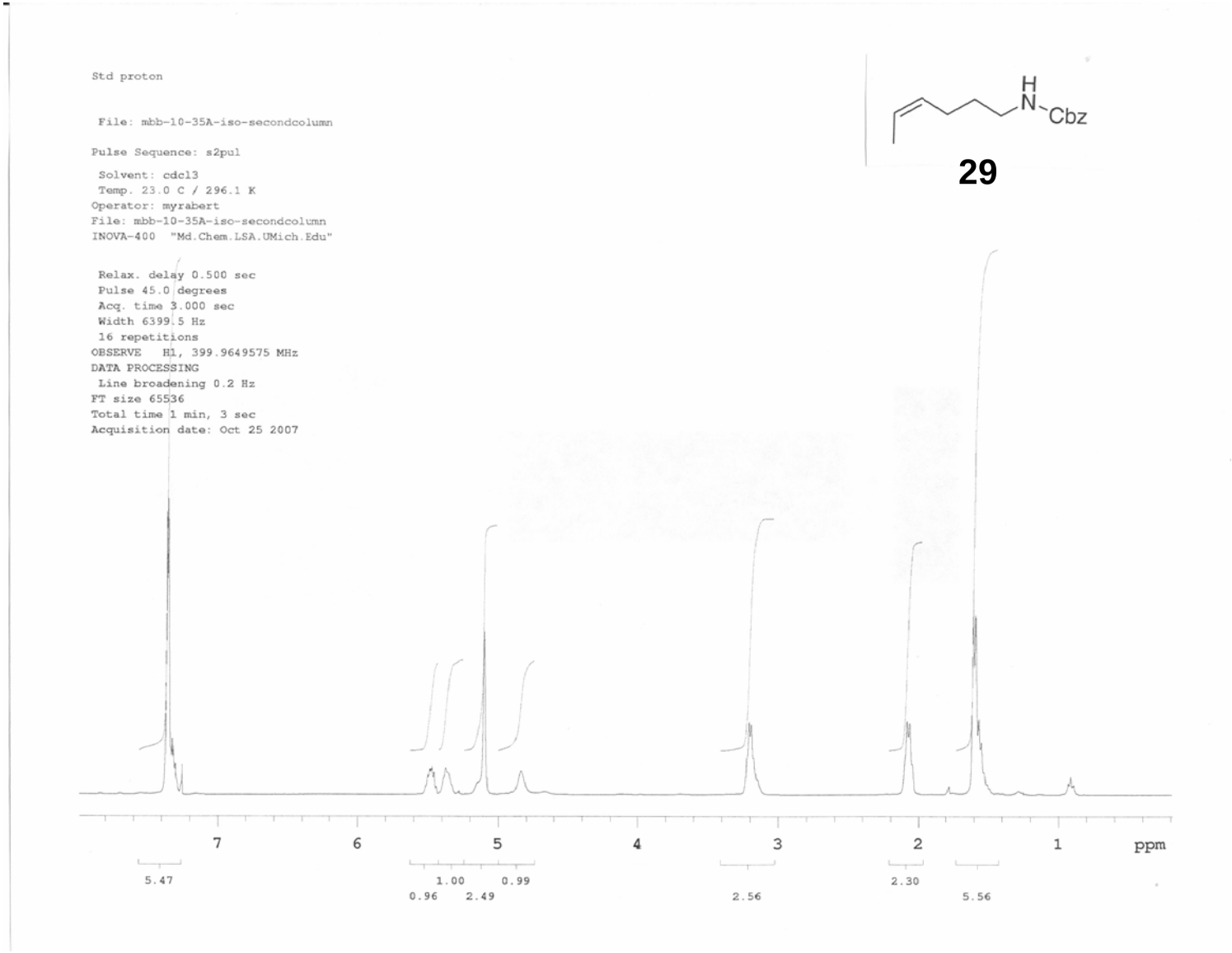
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
400 repetitions
OBSERVE C13, 100.5712669 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 23 sec
Acquisition date: Aug 3 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15681.967 | 155.929 | 4.4 |
| 2 | 13019.552 | 129.456 | 51.0 |
| 3 | 12531.123 | 124.599 | 42.5 |
| 4 | 7944.462 | 78.993 | 5.6 |
| 5 | 7776.496 | 77.323 | 48.2 |
| 6 | 7764.709 | 77.206 | 4.1 |
| 7 | 7744.818 | 77.008 | 52.1 |
| 8 | 7713.140 | 76.693 | 47.2 |
| 9 | 4044.400 | 40.214 | 15.0 |
| 10 | 2996.820 | 29.798 | 25.0 |
| 11 | 2855.375 | 28.392 | 214.6 |
| 12 | 2753.711 | 27.381 | 6.2 |
| 13 | 2422.935 | 24.092 | 31.9 |
| 14 | 1278.112 | 12.709 | 25.5 |




**28**




Std proton
File: mbb-10-35A-iso-secondcolumn
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-35A-iso-secondcolumn
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649575 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Oct 25 2007
H
N
Cbz
29
7
6
5
4
3
2
1
ppm
5.47
0.96
1.00
2.49
0.99
2.56
2.30
5.56

Std Carbon

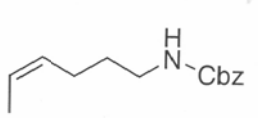
File: mbb-10-35A-C13-400

Pulse Sequence: s2pul

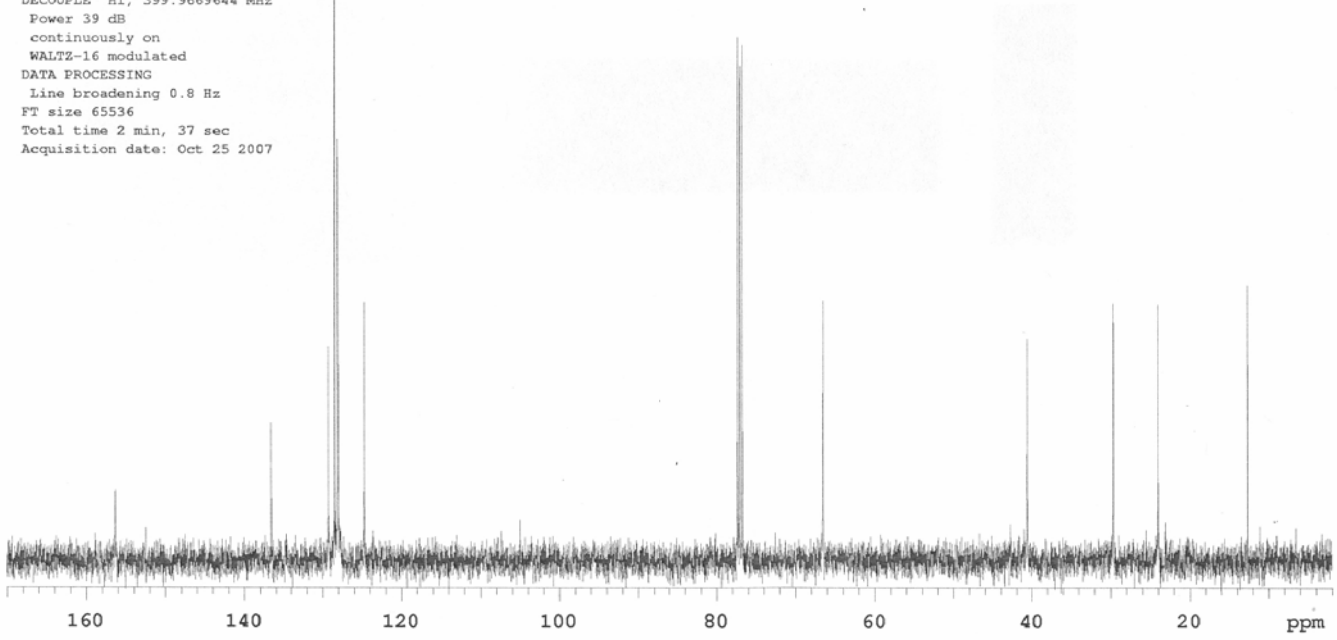
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-35A-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

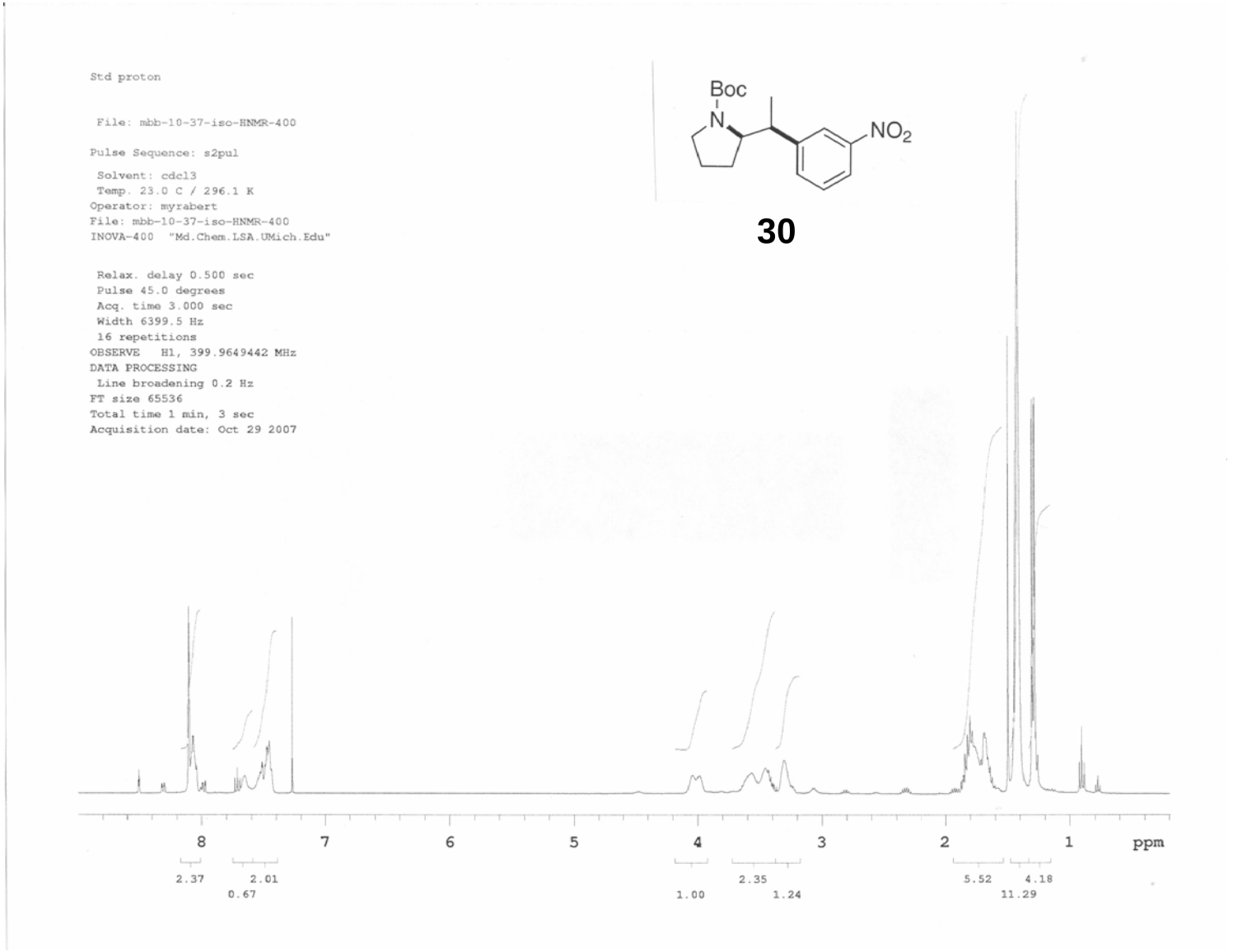
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
112 repetitions
OBSERVE C13, 100.5712746 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 2 min, 37 sec
Acquisition date: Oct 25 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15721.400 | 156.321 | 13.4 |
| 2 | 13738.218 | 136.602 | 26.0 |
| 3 | 13001.523 | 129.277 | 40.1 |
| 4 | 12917.540 | 128.442 | 159.6 |
| 5 | 12878.495 | 128.053 | 78.5 |
| 6 | 12874.812 | 128.017 | 75.1 |
| 7 | 12541.826 | 124.706 | 48.1 |
| 8 | 7776.885 | 77.327 | 97.6 |
| 9 | 7744.470 | 77.005 | 92.2 |
| 10 | 7712.792 | 76.690 | 96.2 |
| 11 | 6688.787 | 66.508 | 48.6 |
| 12 | 4089.728 | 40.665 | 41.6 |
| 13 | 2980.265 | 29.633 | 48.1 |
| 14 | 2414.484 | 24.008 | 47.9 |
| 15 | 1277.027 | 12.698 | 51.4 |




**29**




Std proton
File: mbb-10-37-iso-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-37-iso-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649442 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Oct 29 2007
Boc
N
NO2
30
8
7
6
5
4
3
2
1
ppm
2.37
2.01
0.67
1.00
2.35
1.24
5.52
4.18
11.29

Std Carbon

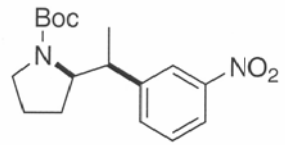
File: mbb-10-37-C13-400

Pulse Sequence: s2pul

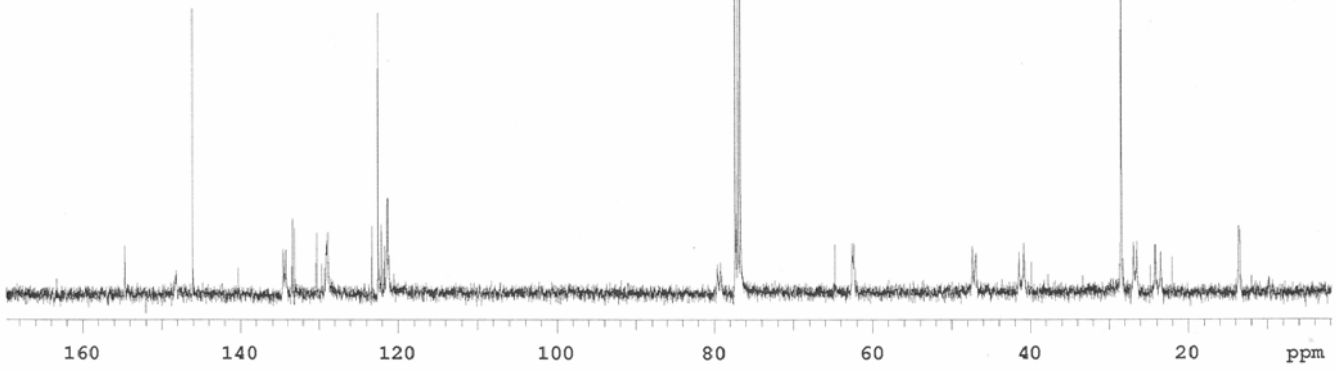
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-37-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
3888 repetitions
OBSERVE C13, 100.5712676 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 31 min, 19 sec
Acquisition date: Oct 29 2007

| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|---|---|---|---|
| 1 | 15556.003 | 154.676 | 9.040 | | 2704.364 | 26.890 | 9.6 |
| 2 | 15285.636 | 151.988 | -3.841 | | 2659.426 | 26.443 | 9.8 |
| 3 | 14896.661 | 148.120 | 4.342 | | 2485.566 | 24.714 | 5.3 |
| 4 | 14691.860 | 146.084 | 53.043 | | 2428.840 | 24.150 | 9.2 |
| 5 | 14683.757 | 146.003 | 4.544 | | 2357.381 | 23.440 | 7.8 |
| 6 | 14111.345 | 140.312 | 4.945 | | 2210.779 | 21.982 | 6.9 |
| 7 | 13539.670 | 134.628 | 8.346 | | 1362.106 | 13.544 | 12.7 |
| 8 | 13502.835 | 134.261 | 8.2 | | | | |
| 9 | 13415.905 | 133.397 | 5.1 | | | | |
| 10 | 13404.118 | 133.280 | 14.1 | | | | |
| 11 | 13379.807 | 133.038 | 12.3 | | | | |
| 12 | 13102.073 | 130.277 | 11.3 | | | | |
| 13 | 13036.507 | 129.625 | 5.6 | | | | |
| 14 | 12995.989 | 129.222 | 4.8 | | | | |
| 15 | 12959.154 | 128.855 | 11.5 | | | | |
| 16 | 12397.793 | 123.274 | 12.6 | | | | |
| 17 | 12323.387 | 122.534 | 52.1 | | | | |
| 18 | 12282.132 | 122.124 | 12.9 | | | | |
| 19 | 12277.712 | 122.080 | 11.7 | | | | |
| 20 | 12249.717 | 121.801 | 4.6 | | | | |
| 21 | 12237.194 | 121.677 | 8.9 | | | | |
| 22 | 12202.569 | 121.333 | 17.9 | | | | |
| 23 | 12181.941 | 121.127 | 7.2 | | | | |
| 24 | 8004.146 | 79.587 | 5.5 | | | | |
| 25 | 7964.364 | 79.191 | 5.8 | | | | |
| 26 | 7776.507 | 77.323 | 233.1 | | | | |
| 27 | 7765.457 | 77.213 | 12.5 | | | | |
| 28 | 7744.829 | 77.008 | 238.4 | | | | |
| 29 | 7712.415 | 76.686 | 229.4 | | | | |
| 30 | 6513.812 | 64.768 | 9.2 | | | | |
| 31 | 6266.283 | 62.307 | 9.5 | | | | |
| 32 | 4759.742 | 47.327 | 8.9 | | | | |
| 33 | 4717.014 | 46.902 | 7.6 | | | | |
| 34 | 4173.333 | 41.496 | 7.8 | | | | |
| 35 | 4109.978 | 40.866 | 9.5 | | | | |
| 36 | 4000.210 | 39.775 | 5.9 | | | | |
| 37 | 2864.964 | 28.487 | 49.1 | | | | |
| 38 | 2857.597 | 28.414 | 269.3 | | | | |
| 39 | 2835.496 | 28.194 | 6.5 | | | | |




**30**



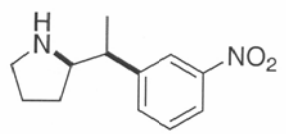
Std proton

File: mbb-10-38-cr

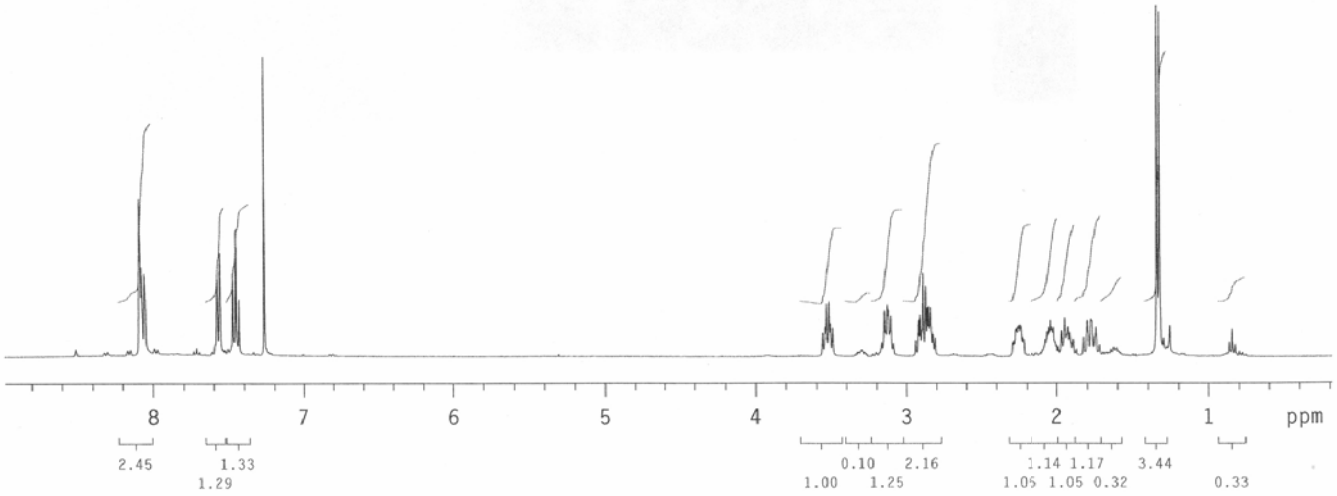
Pulse Sequence: s2pul

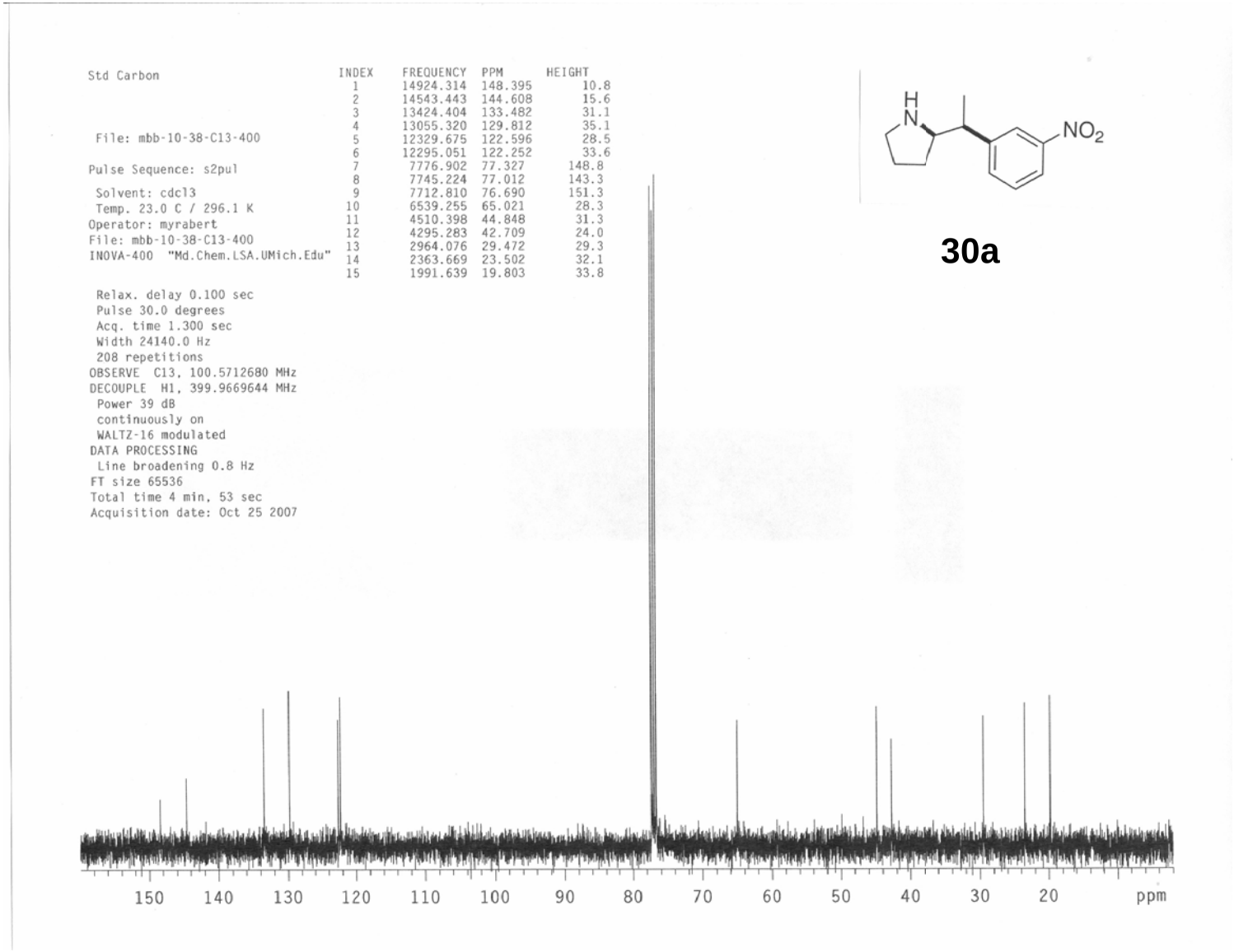
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-38-cr
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649515 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Oct 25 2007

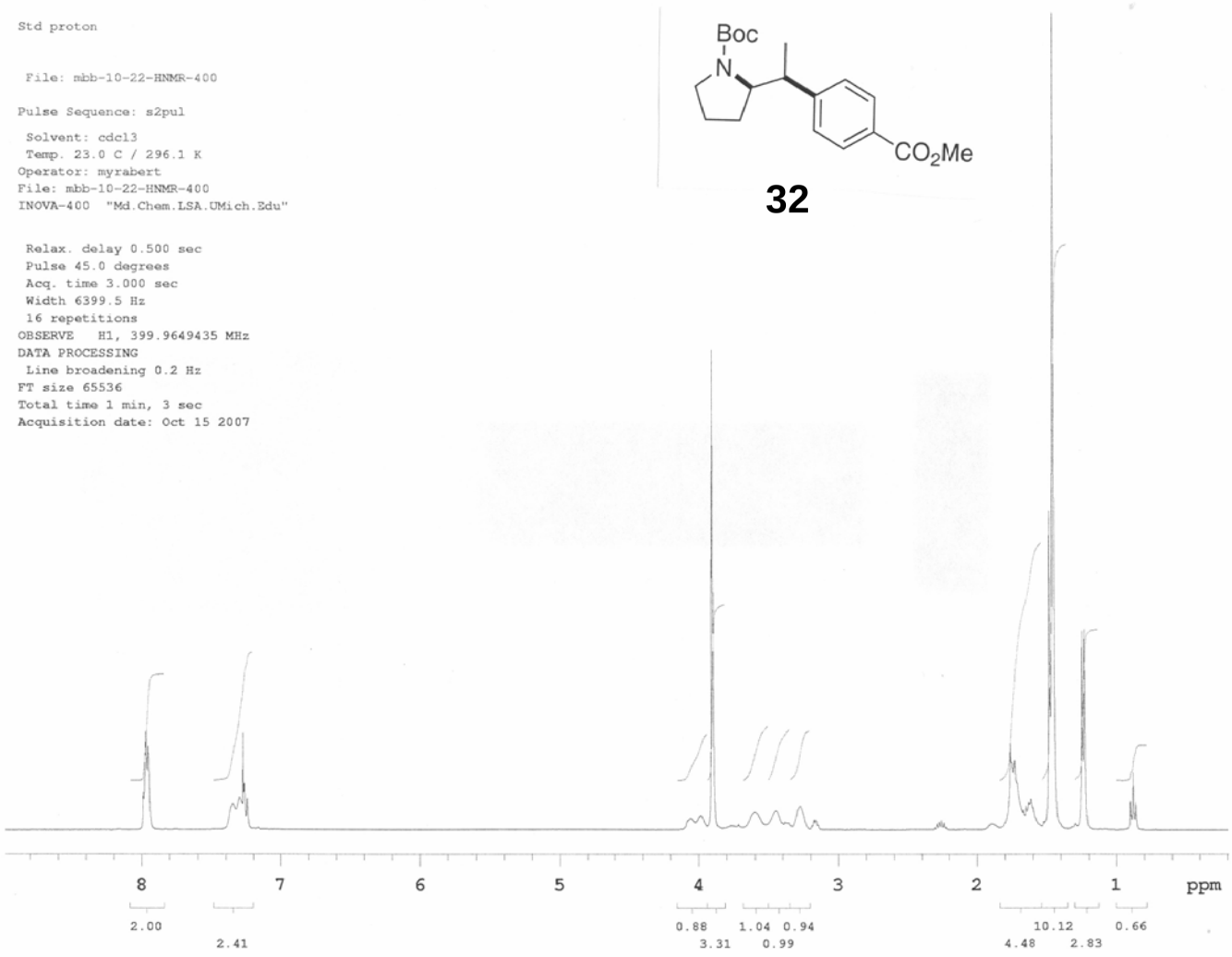



**30a**




Std Carbon
File: mbb-10-38-C13-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-38-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
208 repetitions
OBSERVE C13, 100.5712680 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 4 min, 53 sec
Acquisition date: Oct 25 2007
INDEX FREQUENCY PPM HEIGHT
1 14924.314 148.395 10.8
2 14543.443 144.608 15.6
3 13424.404 133.482 31.1
4 13055.320 129.812 35.1
5 12329.675 122.596 28.5
6 12295.051 122.252 33.6
7 7776.902 77.327 148.8
8 7745.224 77.012 143.3
9 7712.810 76.690 151.3
10 6539.255 65.021 28.3
11 4510.398 44.848 31.3
12 4295.283 42.709 24.0
13 2964.076 29.472 29.3
14 2363.669 23.502 32.1
15 1991.639 19.803 33.8
H
N
NO2
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

**30a**


Std proton
File: mbb-10-22-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-22-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649435 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Oct 15 2007
Boc
N
CO2Me
32
8
7
6
5
4
3
2
1
ppm
2.00
2.41
0.88
3.31
1.04
0.99
0.94
10.12
4.48
2.83
0.66

Std Carbon

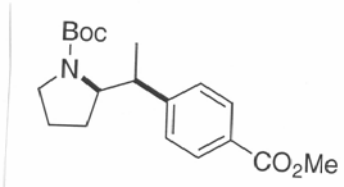
File: mbb-10-22-C13-400

Pulse Sequence: s2pul

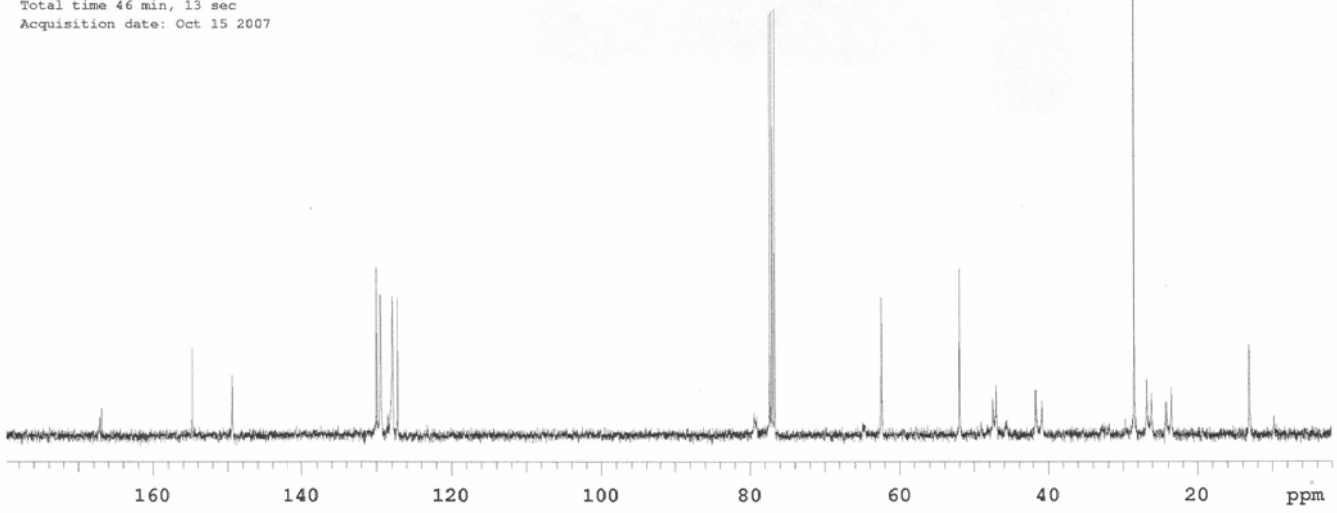
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-22-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
1968 repetitions
OBSERVE C13, 100.5712685 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 46 min, 13 sec
Acquisition date: Oct 15 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16802.346 | 167.069 | 3.4 |
| 2 | 16781.718 | 166.864 | 5.1 |
| 3 | 15564.699 | 154.763 | 16.4 |
| 4 | 15023.228 | 149.379 | 11.3 |
| 5 | 13070.250 | 129.960 | 31.5 |
| 6 | 13021.629 | 129.477 | 26.5 |
| 7 | 12918.491 | 128.451 | 4.0 |
| 8 | 12858.082 | 127.850 | 26.3 |
| 9 | 12785.886 | 127.133 | 25.9 |
| 10 | 7987.057 | 79.417 | 4.3 |
| 11 | 7776.362 | 77.322 | 78.3 |
| 12 | 7764.575 | 77.205 | 5.6 |
| 13 | 7743.948 | 77.000 | 78.9 |
| 14 | 7712.270 | 76.685 | 79.4 |
| 15 | 6278.662 | 62.430 | 25.9 |
| 16 | 5230.345 | 52.006 | 17.6 |
| 17 | 5224.452 | 51.948 | 31.2 |
| 18 | 4780.225 | 47.531 | 7.0 |
| 19 | 4733.813 | 47.069 | 9.4 |
| 20 | 4185.712 | 41.619 | 8.6 |
| 21 | 4102.466 | 40.792 | 6.6 |
| 22 | 2863.345 | 28.471 | 337.2 |
| 23 | 2696.116 | 26.808 | 10.5 |
| 24 | 2626.130 | 26.112 | 7.8 |
| 25 | 2433.852 | 24.200 | 6.4 |
| 26 | 2363.866 | 23.504 | 9.1 |
| 27 | 1316.286 | 13.088 | 17.1 |
| 28 | 978.880 | 9.733 | 3.8 |




**32**



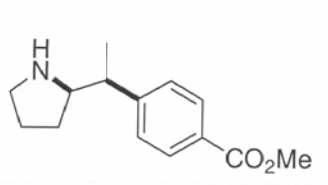
Std proton

File: mbb-10-21-cr

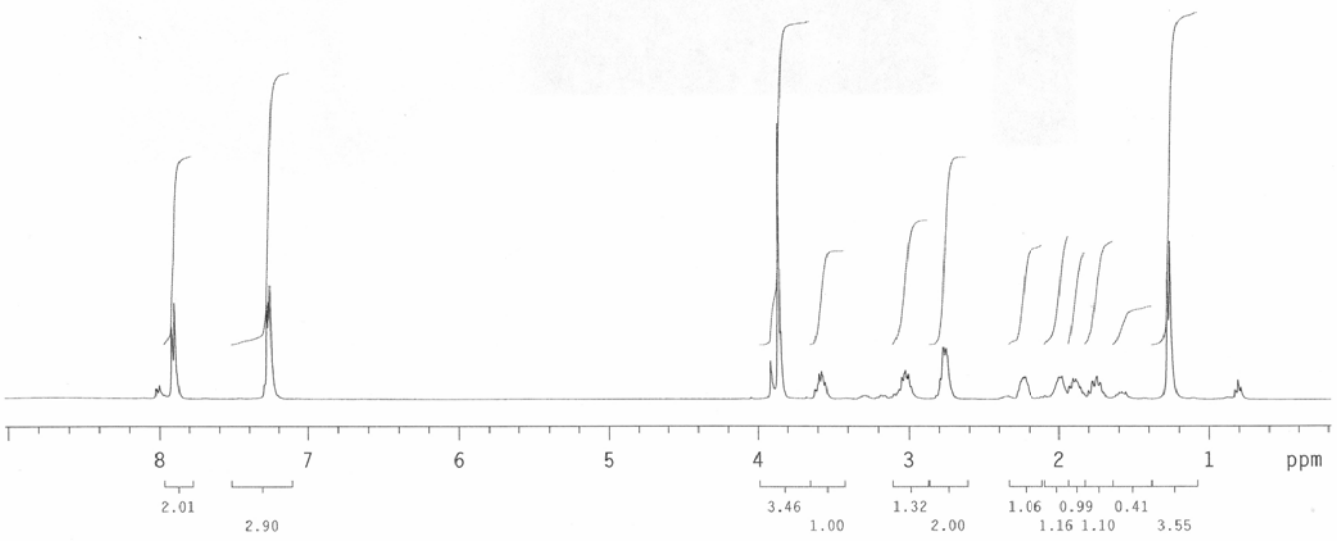
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-21-cr
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Oct 13 2007




**32a**



Std Carbon

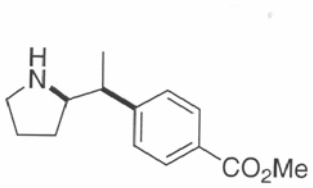
File: mbb-10-21-cr-C13-400

Pulse Sequence: s2pul

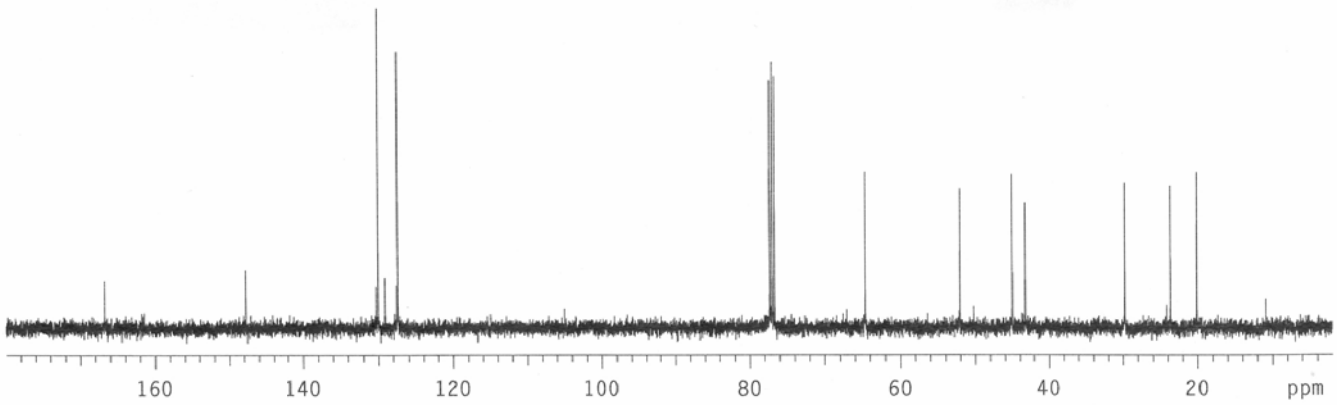
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-21-cr-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

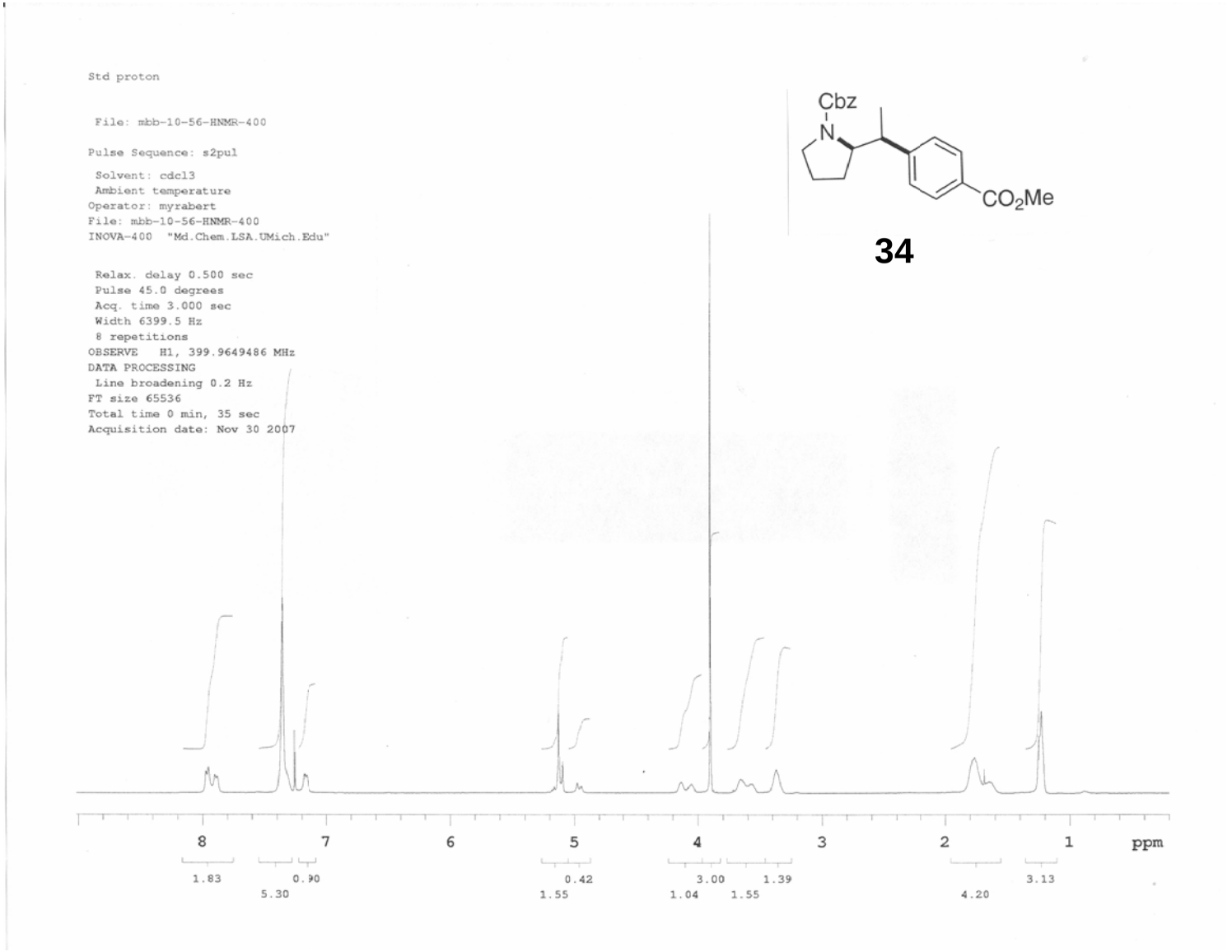
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
352 repetitions
OBSERVE C13, 100.5712666 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 8 min, 16 sec
Acquisition date: Oct 13 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16768.129 | 166.729 | 8.6 |
| 2 | 14860.826 | 147.764 | 10.7 |
| 3 | 13105.283 | 130.308 | 7.7 |
| 4 | 13078.025 | 130.037 | 59.0 |
| 5 | 12985.201 | 129.114 | 9.3 |
| 6 | 12827.549 | 127.547 | 7.8 |
| 7 | 12816.498 | 127.437 | 51.0 |
| 8 | 7776.033 | 77.319 | 45.7 |
| 9 | 7744.355 | 77.004 | 49.1 |
| 10 | 7712.677 | 76.689 | 46.5 |
| 11 | 6502.288 | 64.654 | 28.8 |
| 12 | 5235.910 | 52.062 | 25.7 |
| 13 | 4527.946 | 45.022 | 28.3 |
| 14 | 4353.349 | 43.286 | 23.0 |
| 15 | 2991.201 | 29.742 | 26.7 |
| 16 | 2377.534 | 23.640 | 26.1 |
| 17 | 2019.501 | 20.080 | 28.7 |




**32a**




Std proton
File: mbb-10-56-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-56-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649486 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Nov 30 2007
Cbz
N
CO2Me
34
8
7
6
5
4
3
2
1
ppm
1.83
5.30
0.90
0.42
1.55
3.00
1.04
1.39
1.55
4.20
3.13

Std Carbon

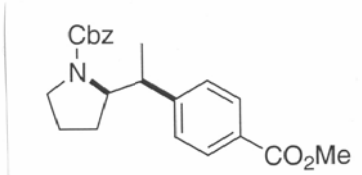
File: mbb-10-56-C13-400

Pulse Sequence: s2pul

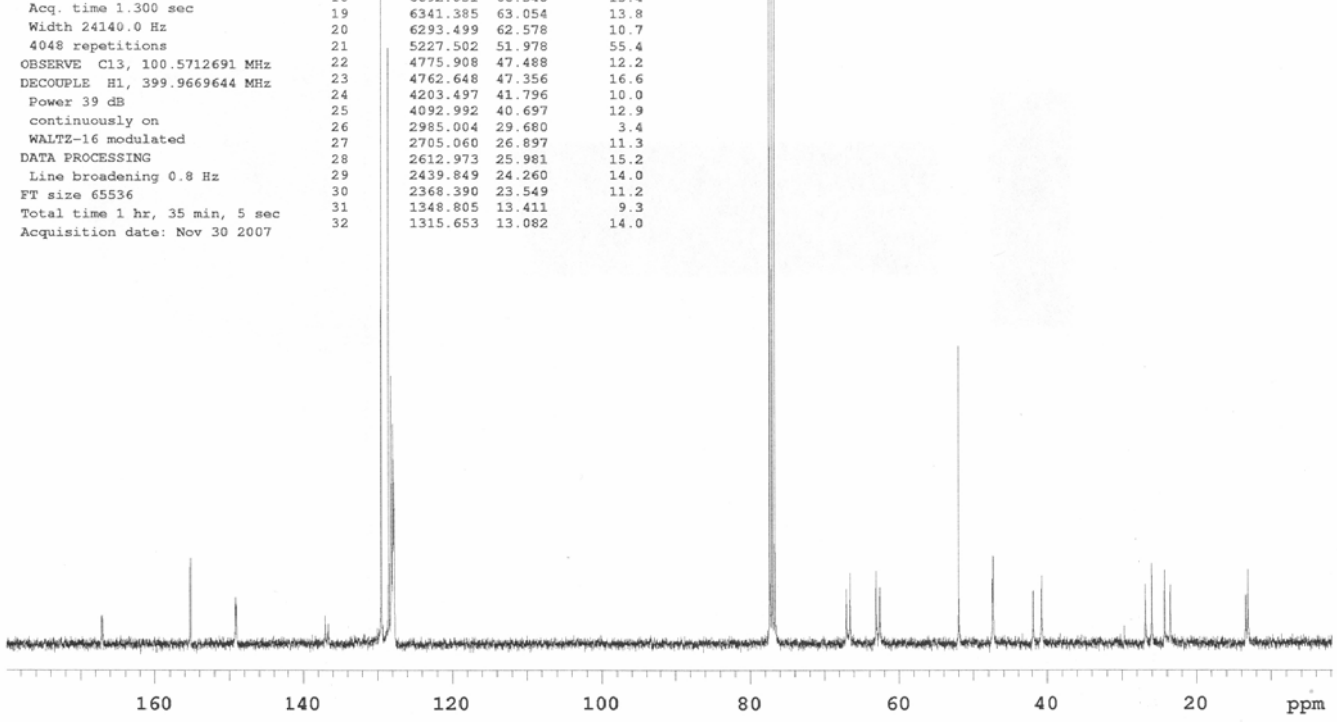
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-56-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

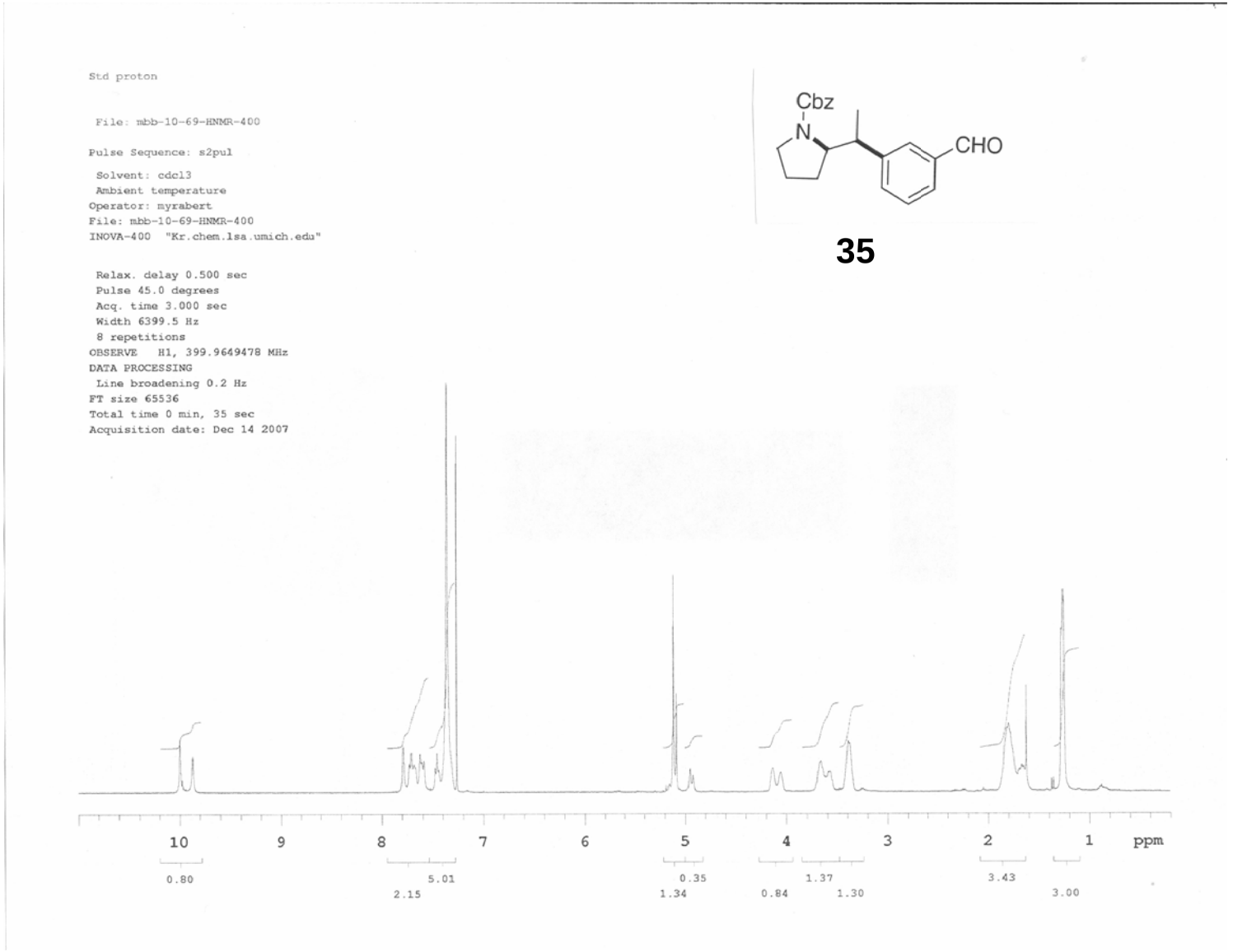
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
4048 repetitions
OBSERVE C13, 100.5712691 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 35 min, 5 sec
Acquisition date: Nov 30 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16803.186 | 167.077 | 5.4 |
| 2 | 15607.531 | 155.189 | 16.2 |
| 3 | 15001.231 | 149.160 | 8.8 |
| 4 | 13776.108 | 136.979 | 5.3 |
| 5 | 13733.379 | 136.554 | 3.7 |
| 6 | 13075.511 | 130.012 | 3.0 |
| 7 | 13018.785 | 129.448 | 168.0 |
| 8 | 12993.738 | 129.199 | 3.5 |
| 9 | 12919.332 | 128.459 | 110.8 |
| 10 | 12886.917 | 128.137 | 49.9 |
| 11 | 12863.343 | 127.903 | 41.0 |
| 12 | 12847.872 | 127.749 | 30.2 |
| 13 | 7777.203 | 77.330 | 146.4 |
| 14 | 7765.415 | 77.213 | 11.4 |
| 15 | 7744.788 | 77.008 | 156.8 |
| 16 | 7713.110 | 76.693 | 154.5 |
| 17 | 6745.830 | 67.075 | 10.3 |
| 18 | 6692.051 | 66.540 | 13.4 |
| 19 | 6341.385 | 63.054 | 13.8 |
| 20 | 6293.499 | 62.578 | 10.7 |
| 21 | 5227.502 | 51.978 | 55.4 |
| 22 | 4775.908 | 47.488 | 12.2 |
| 23 | 4762.648 | 47.356 | 16.6 |
| 24 | 4203.497 | 41.796 | 10.0 |
| 25 | 4092.992 | 40.697 | 12.9 |
| 26 | 2985.004 | 29.680 | 3.4 |
| 27 | 2705.060 | 26.897 | 11.3 |
| 28 | 2612.973 | 25.981 | 15.2 |
| 29 | 2439.849 | 24.260 | 14.0 |
| 30 | 2368.390 | 23.549 | 11.2 |
| 31 | 1348.805 | 13.411 | 9.3 |
| 32 | 1315.653 | 13.082 | 14.0 |




**34**




Std proton
File: mbb-10-69-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-69-HNMR-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649478 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Dec 14 2007
Cbz
N
CHO
35
10
9
8
7
6
5
4
3
2
1
ppm
0.80
2.15
5.01
1.34
0.35
0.84
1.37
1.30
3.43
3.00

Std Carbon

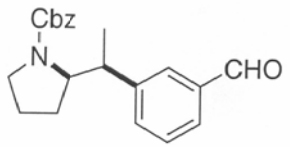
File: mbb-10-69-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-69-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
6688 repetitions
OBSERVE C13, 100.5712678 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 2 hr, 37 min, 5 sec
Acquisition date: Dec 14 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19369.700 | 192.597 | 16.2 |
| 2 | 19354.230 | 192.443 | 14.6 |
| 3 | 15606.664 | 155.180 | 29.4 |
| 4 | 14567.187 | 144.844 | 12.9 |
| 5 | 13712.622 | 136.347 | 61.2 |
| 6 | 13524.028 | 134.472 | 16.2 |
| 7 | 13490.140 | 134.135 | 12.2 |
| 8 | 12955.299 | 128.817 | 55.0 |
| 9 | 12922.148 | 128.487 | 50.5 |
| 10 | 12883.103 | 128.099 | 32.7 |
| 11 | 12865.423 | 127.923 | 109.0 |
| 12 | 12844.059 | 127.711 | 26.5 |
| 13 | 7777.072 | 77.329 | 362.8 |
| 14 | 7765.285 | 77.212 | 21.5 |
| 15 | 7744.658 | 77.007 | 387.3 |
| 16 | 7712.980 | 76.692 | 381.8 |
| 17 | 6747.910 | 67.096 | 12.4 |
| 18 | 6694.868 | 66.568 | 16.2 |
| 19 | 6344.938 | 63.089 | 17.9 |
| 20 | 6305.893 | 62.701 | 11.5 |
| 21 | 4771.358 | 47.443 | 17.3 |
| 22 | 4761.781 | 47.347 | 19.9 |
| 23 | 4177.582 | 41.539 | 11.4 |
| 24 | 4062.658 | 40.396 | 16.3 |
| 25 | 2708.613 | 26.932 | 12.4 |
| 26 | 2612.106 | 25.973 | 19.6 |
| 27 | 2441.193 | 24.273 | 18.8 |
| 28 | 2367.523 | 23.541 | 12.1 |
| 29 | 1368.565 | 13.608 | 13.5 |
| 30 | 1329.520 | 13.220 | 14.3 |




**35**

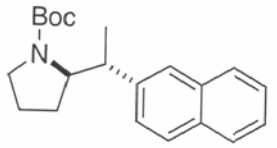
Std proton

File: mbb-9-201-HNMR-400

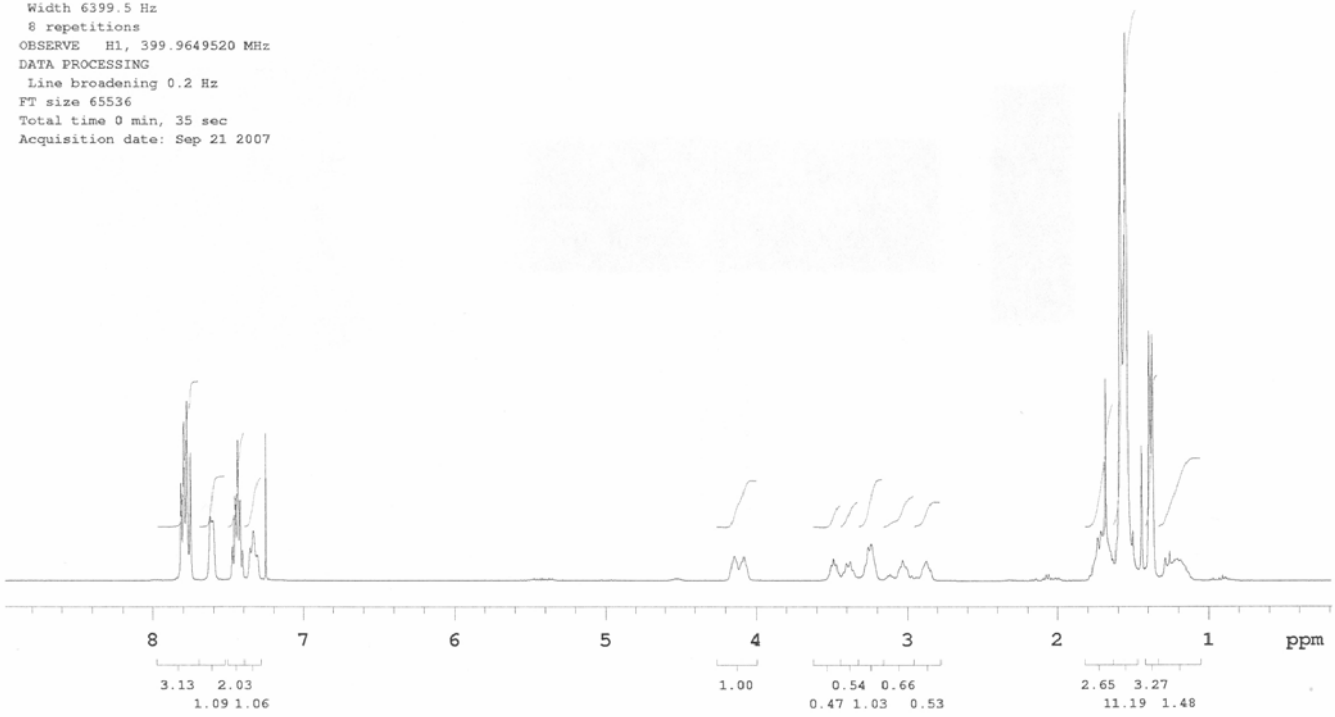
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-9-201-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649520 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Sep 21 2007




**36**



Std Carbon

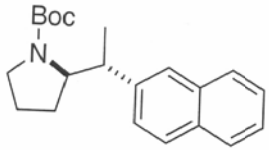
File: mbb-9-201-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-9-201-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
4016 repetitions
OBSERVE C13, 100.5712684 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 34 min, 19 sec
Acquisition date: Sep 21 2007

| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|---|---|---|---|
| 1 | 15605.319 | 155.167 | 5.940 | | 2277.038 | 22.641 | 21.7 |
| 2 | 14203.389 | 141.227 | 15.041 | | 1906.481 | 18.957 | 4.0 |
| 3 | 13412.179 | 133.360 | 48.742 | | 1851.966 | 18.414 | 21.3 |
| 4 | 13301.675 | 132.261 | 41.943 | | 1794.503 | 17.843 | 23.8 |
| 5 | 13020.257 | 129.463 | 4.6 | | | | |
| 6 | 12848.607 | 127.756 | 24.8 | | | | |
| 7 | 12835.347 | 127.624 | 176.3 | | | | |
| 8 | 12827.243 | 127.544 | 84.3 | | | | |
| 9 | 12816.930 | 127.441 | 29.6 | | | | |
| 10 | 12780.095 | 127.075 | 24.4 | | | | |
| 11 | 12742.523 | 126.701 | 21.3 | | | | |
| 12 | 12730.736 | 126.584 | 24.7 | | | | |
| 13 | 12702.005 | 126.299 | 20.4 | | | | |
| 14 | 12660.750 | 125.888 | 25.7 | | | | |
| 15 | 12648.963 | 125.771 | 27.6 | | | | |
| 16 | 12606.235 | 125.346 | 26.9 | | | | |
| 17 | 12595.921 | 125.244 | 28.8 | | | | |
| 18 | 7984.949 | 79.396 | 8.6 | | | | |
| 19 | 7954.744 | 79.096 | 5.8 | | | | |
| 20 | 7943.694 | 78.986 | 10.3 | | | | |
| 21 | 7777.201 | 77.330 | 133.2 | | | | |
| 22 | 7765.414 | 77.213 | 10.4 | | | | |
| 23 | 7744.787 | 77.008 | 147.8 | | | | |
| 24 | 7713.109 | 76.693 | 144.1 | | | | |
| 25 | 6291.288 | 62.556 | 28.0 | | | | |
| 26 | 6281.711 | 62.460 | 30.0 | | | | |
| 27 | 4697.081 | 46.704 | 26.0 | | | | |
| 28 | 4640.355 | 46.140 | 21.6 | | | | |
| 29 | 4280.848 | 42.565 | 21.5 | | | | |
| 30 | 4149.716 | 41.261 | 21.8 | | | | |
| 31 | 2997.526 | 29.805 | 4.9 | | | | |
| 32 | 2985.739 | 29.688 | 4.7 | | | | |
| 33 | 2878.918 | 28.626 | 547.0 | | | | |
| 34 | 2856.817 | 28.406 | 29.0 | | | | |
| 35 | 2839.873 | 28.237 | 8.2 | | | | |
| 36 | 2775.044 | 27.593 | 23.5 | | | | |
| 37 | 2660.856 | 26.457 | 27.6 | | | | |
| 38 | 2568.033 | 25.534 | 4.4 | | | | |
| 39 | 2360.285 | 23.469 | 25.4 | | | | |




**36**

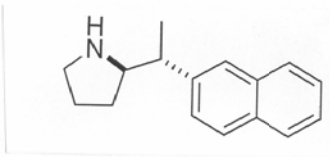
STANDARD PROTON PARAMETERS

File: mbb-9-204-HNMR-400

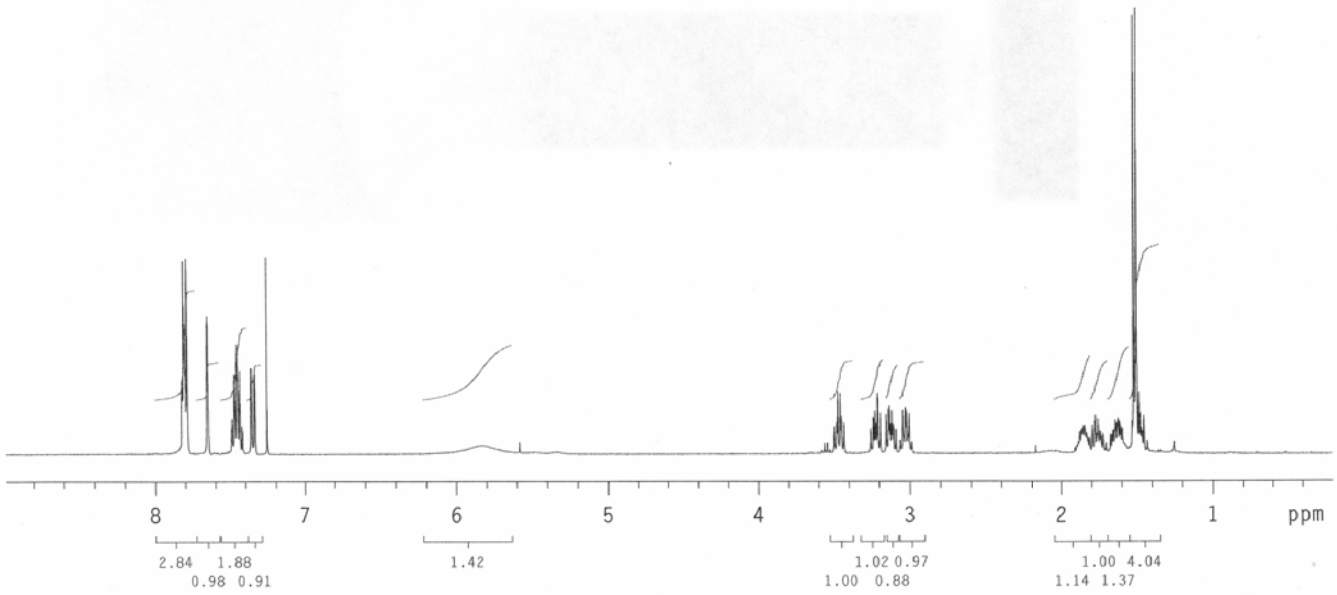
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-204-HNMR-400
VNMRS-401 "Md.Chem.LSA.UMich.Edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.5297577 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec
Acquisition date: May 20 2008




**36a**



STANDARD CARBON PARAMETERS

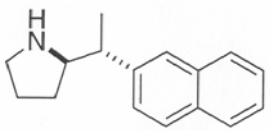
File: mbb-9-204-C13-400

Pulse Sequence: s2pul

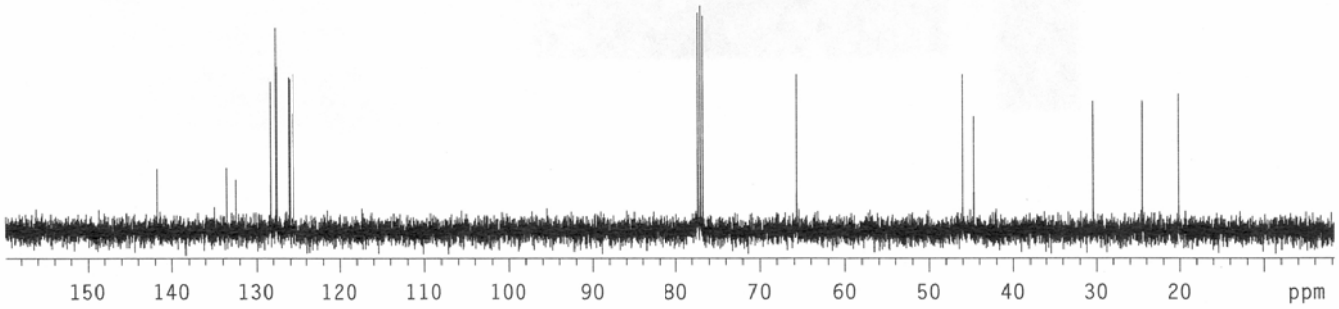
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-204-C13-400
VNMRS-401 "Md.Chem.LSA.UMich.Edu"

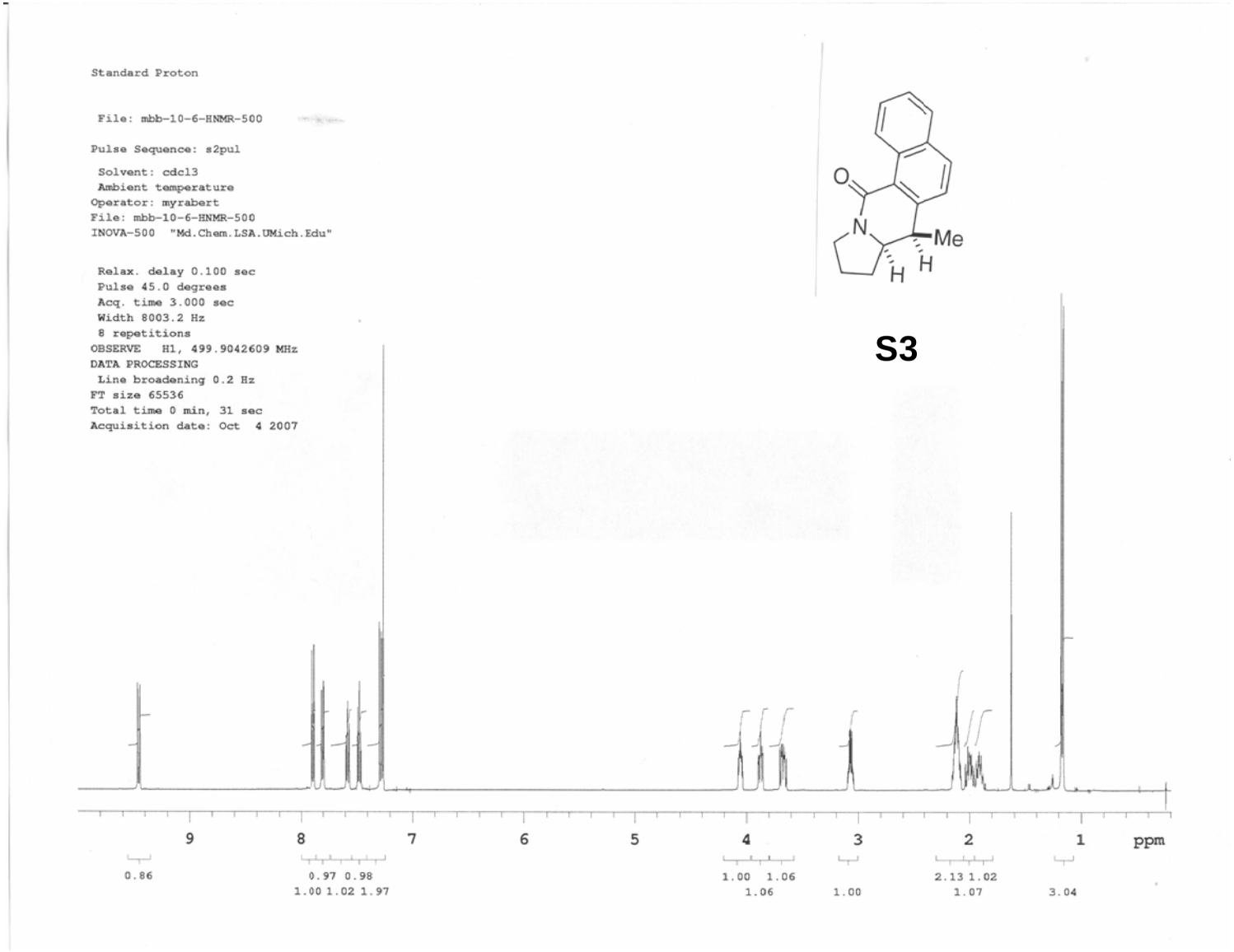
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
80 repetitions
OBSERVE C13, 100.7132879 MHz
DECOUPLE H1, 400.5317616 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min, 3 sec
Acquisition date: May 20 2008

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 14273.454 | 141.724 | 11.5 |
| 2 | 13446.677 | 133.514 | 11.7 |
| 3 | 13338.464 | 132.440 | 9.5 |
| 4 | 12921.183 | 128.297 | 27.7 |
| 5 | 12855.010 | 127.640 | 37.7 |
| 6 | 12851.117 | 127.601 | 30.5 |
| 7 | 12700.865 | 126.109 | 28.5 |
| 8 | 12689.187 | 125.993 | 28.2 |
| 9 | 12650.262 | 125.607 | 21.7 |
| 10 | 12644.034 | 125.545 | 29.1 |
| 11 | 7787.691 | 77.325 | 40.5 |
| 12 | 7755.772 | 77.008 | 41.8 |
| 13 | 7723.853 | 76.691 | 39.9 |
| 14 | 6614.477 | 65.676 | 29.1 |
| 15 | 4633.948 | 46.011 | 29.0 |
| 16 | 4499.266 | 44.674 | 21.3 |
| 17 | 3063.694 | 30.420 | 24.1 |
| 18 | 2469.691 | 24.522 | 24.2 |
| 19 | 2029.055 | 20.147 | 25.4 |




**36a**




Standard Proton
File: mbb-10-6-HNMR-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-6-HNMR-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042609 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Oct 4 2007
O
N
Me
H
H
S3
9
8
7
6
5
4
3
2
1
ppm
0.86
0.97 0.98
1.00 1.02 1.97
1.00 1.06
1.06
1.00
2.13 1.02
1.07
3.04

Std Carbon

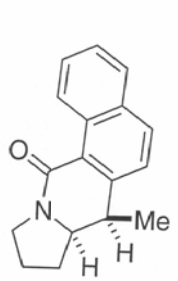
File: mbb-10-6-C13-400

Pulse Sequence: s2pul

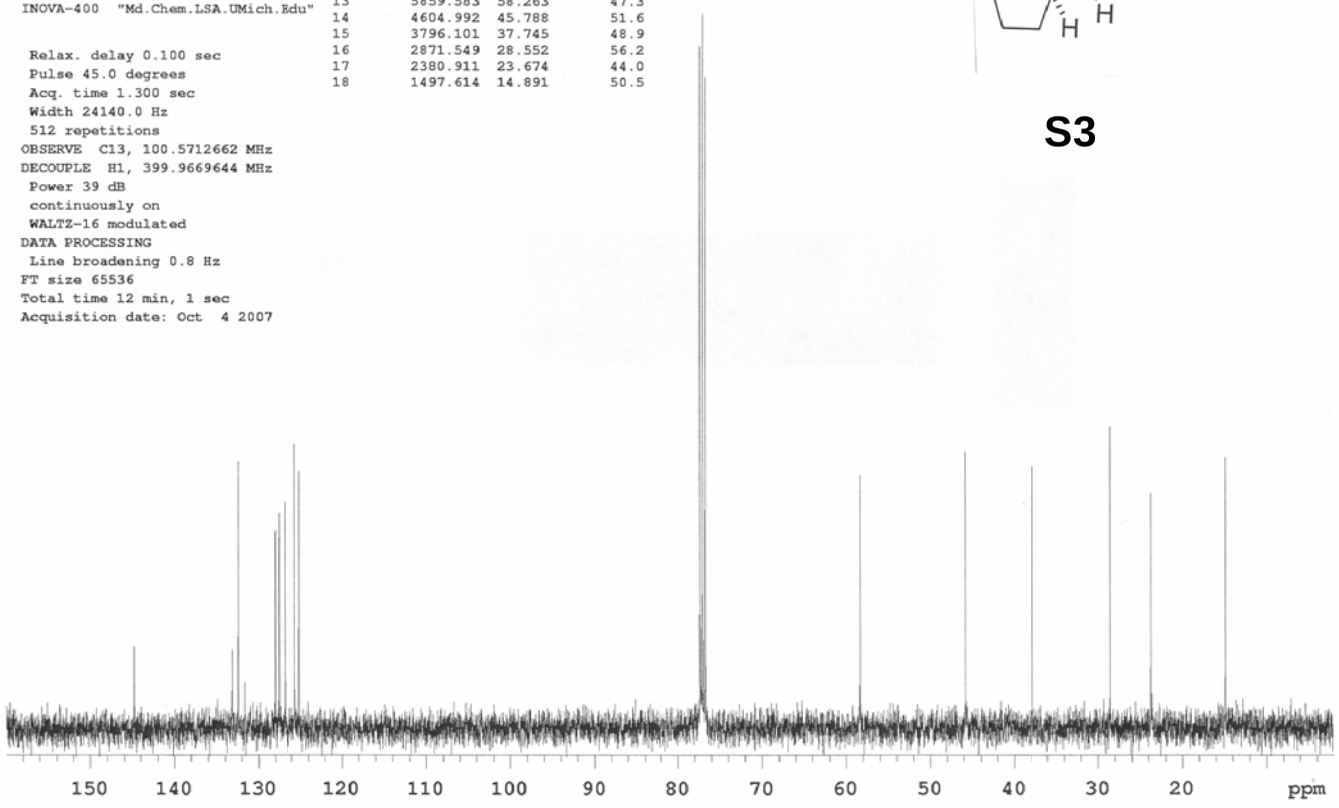
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-6-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
512 repetitions
OBSERVE C13, 100.5712662 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 12 min, 1 sec
Acquisition date: Oct 4 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 14562.158 | 144.794 | 15.4 |
| 2 | 13390.076 | 133.140 | 14.9 |
| 3 | 13320.090 | 132.444 | 49.7 |
| 4 | 13236.844 | 131.617 | 8.7 |
| 5 | 12872.917 | 127.998 | 37.1 |
| 6 | 12825.032 | 127.522 | 40.2 |
| 7 | 12756.519 | 126.841 | 42.4 |
| 8 | 12637.911 | 125.661 | 52.9 |
| 9 | 12582.659 | 125.112 | 47.9 |
| 10 | 7776.463 | 77.323 | 126.9 |
| 11 | 7744.785 | 77.008 | 132.9 |
| 12 | 7712.370 | 76.686 | 121.2 |
| 13 | 5859.583 | 58.263 | 47.3 |
| 14 | 4604.992 | 45.788 | 51.6 |
| 15 | 3796.101 | 37.745 | 48.9 |
| 16 | 2871.549 | 28.552 | 56.2 |
| 17 | 2380.911 | 23.674 | 44.0 |
| 18 | 1497.614 | 14.891 | 50.5 |




**S3**



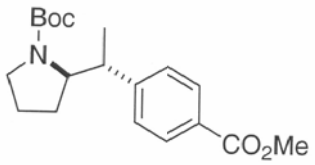
Std proton

File: mbb-10-15-HNMR-400

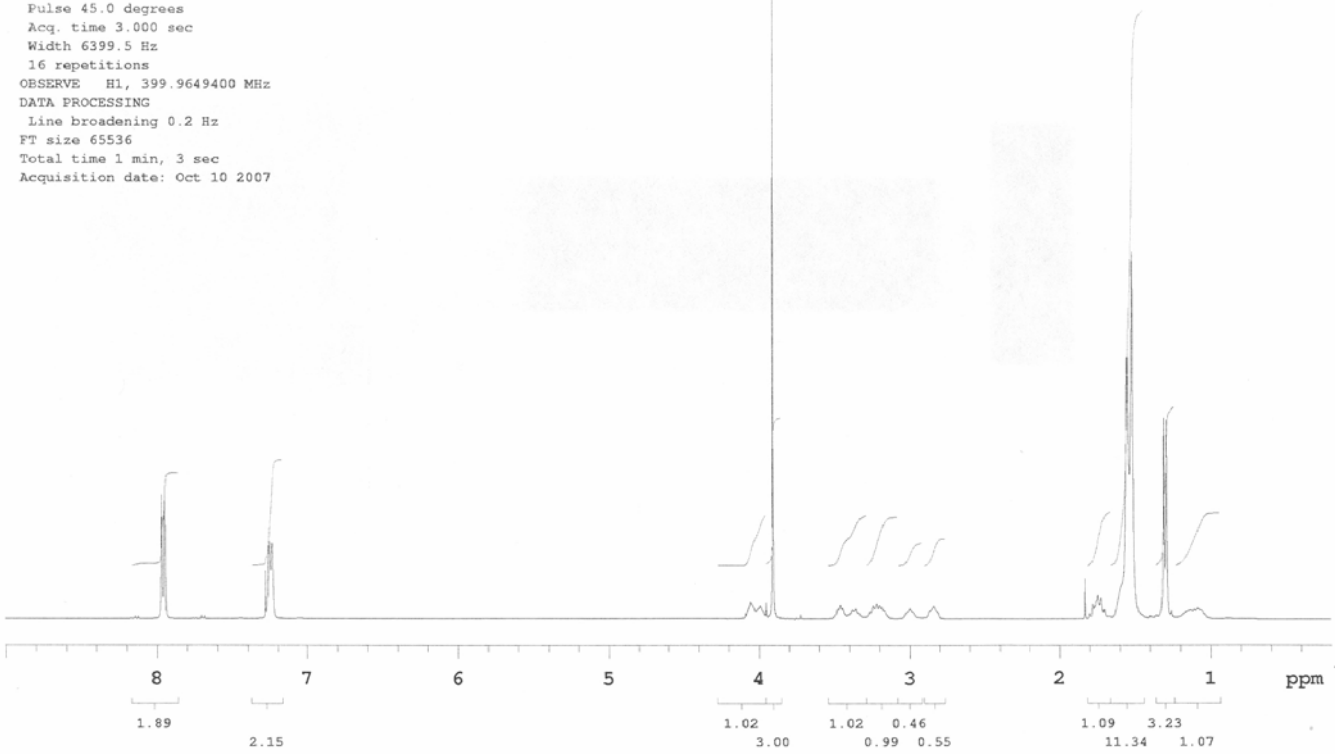
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-15-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
16 repetitions
OBSERVE H1, 399.9649400 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 3 sec
Acquisition date: Oct 10 2007




**37**



Std Carbon

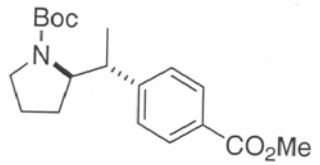
File: mbb-10-15-C13-400

Pulse Sequence: s2pul

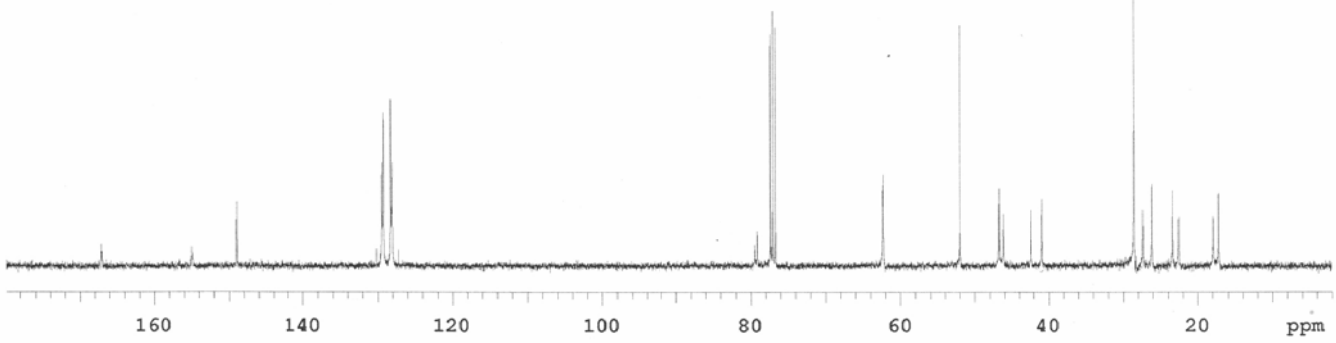
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-15-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

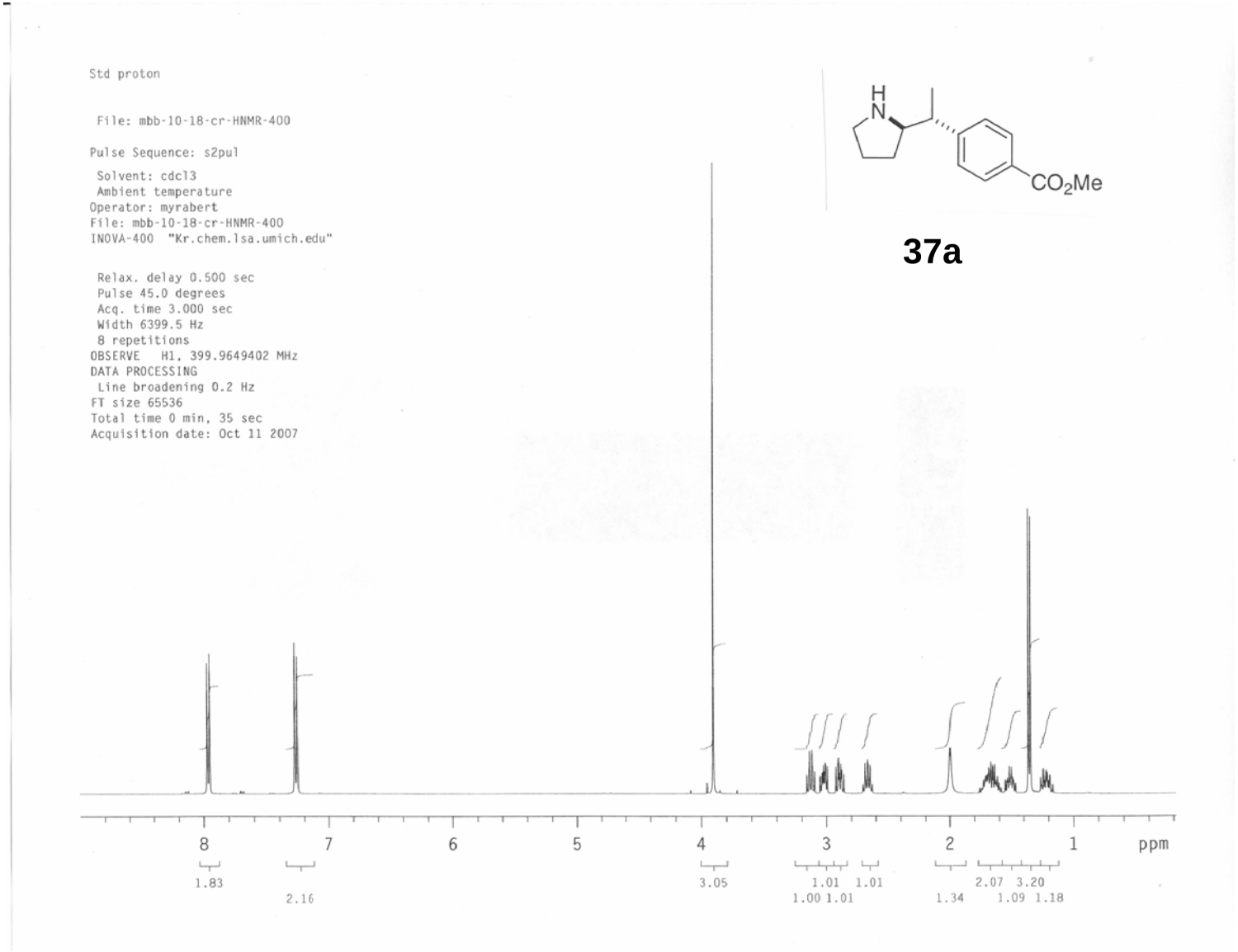
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
1136 repetitions
OBSERVE C13, 100.5712691 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 26 min, 41 sec
Acquisition date: Oct 10 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16808.356 | 167.129 | 4.1 |
| 2 | 15590.599 | 155.020 | 3.6 |
| 3 | 14982.826 | 148.977 | 12.1 |
| 4 | 13089.521 | 130.152 | 3.2 |
| 5 | 13018.798 | 129.448 | 19.4 |
| 6 | 12995.961 | 129.221 | 28.7 |
| 7 | 12904.611 | 128.313 | 31.3 |
| 8 | 12878.826 | 128.057 | 19.4 |
| 9 | 12791.896 | 127.192 | 3.0 |
| 10 | 7992.330 | 79.469 | 3.9 |
| 11 | 7954.022 | 79.088 | 6.4 |
| 12 | 7776.479 | 77.323 | 43.3 |
| 13 | 7765.428 | 77.213 | 3.7 |
| 14 | 7744.801 | 77.008 | 47.5 |
| 15 | 7713.123 | 76.693 | 44.4 |
| 16 | 6274.358 | 62.387 | 14.1 |
| 17 | 6264.781 | 62.292 | 17.2 |
| 18 | 5225.305 | 51.956 | 44.9 |
| 19 | 4699.305 | 46.726 | 14.5 |
| 20 | 4643.316 | 46.169 | 9.7 |
| 21 | 4260.972 | 42.368 | 10.5 |
| 22 | 4110.686 | 40.873 | 12.6 |
| 23 | 2870.829 | 28.545 | 193.7 |
| 24 | 2748.537 | 27.329 | 10.6 |
| 25 | 2624.773 | 26.099 | 15.4 |
| 26 | 2349.986 | 23.366 | 14.2 |
| 27 | 2266.002 | 22.531 | 9.3 |
| 28 | 1801.148 | 17.909 | 9.4 |
| 29 | 1732.635 | 17.228 | 13.7 |




**37**




Std proton
File: mbb-10-18-cr-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-18-cr-HNMR-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649402 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Oct 11 2007
H
N
CO2Me
37a
8
7
6
5
4
3
2
1
ppm
1.83
2.16
3.05
1.01
1.01
1.00 1.01
2.07
3.20
1.34
1.09 1.18

Std Carbon

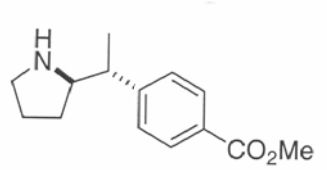
File: mbb-10-18-cr-C13-400

Pulse Sequence: s2pul

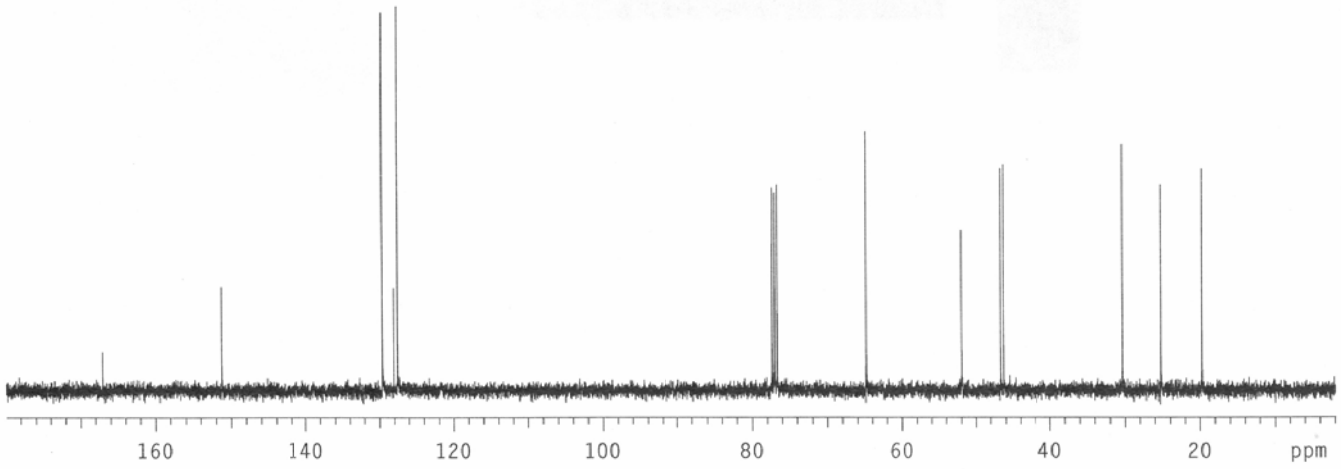
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-18-cr-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"

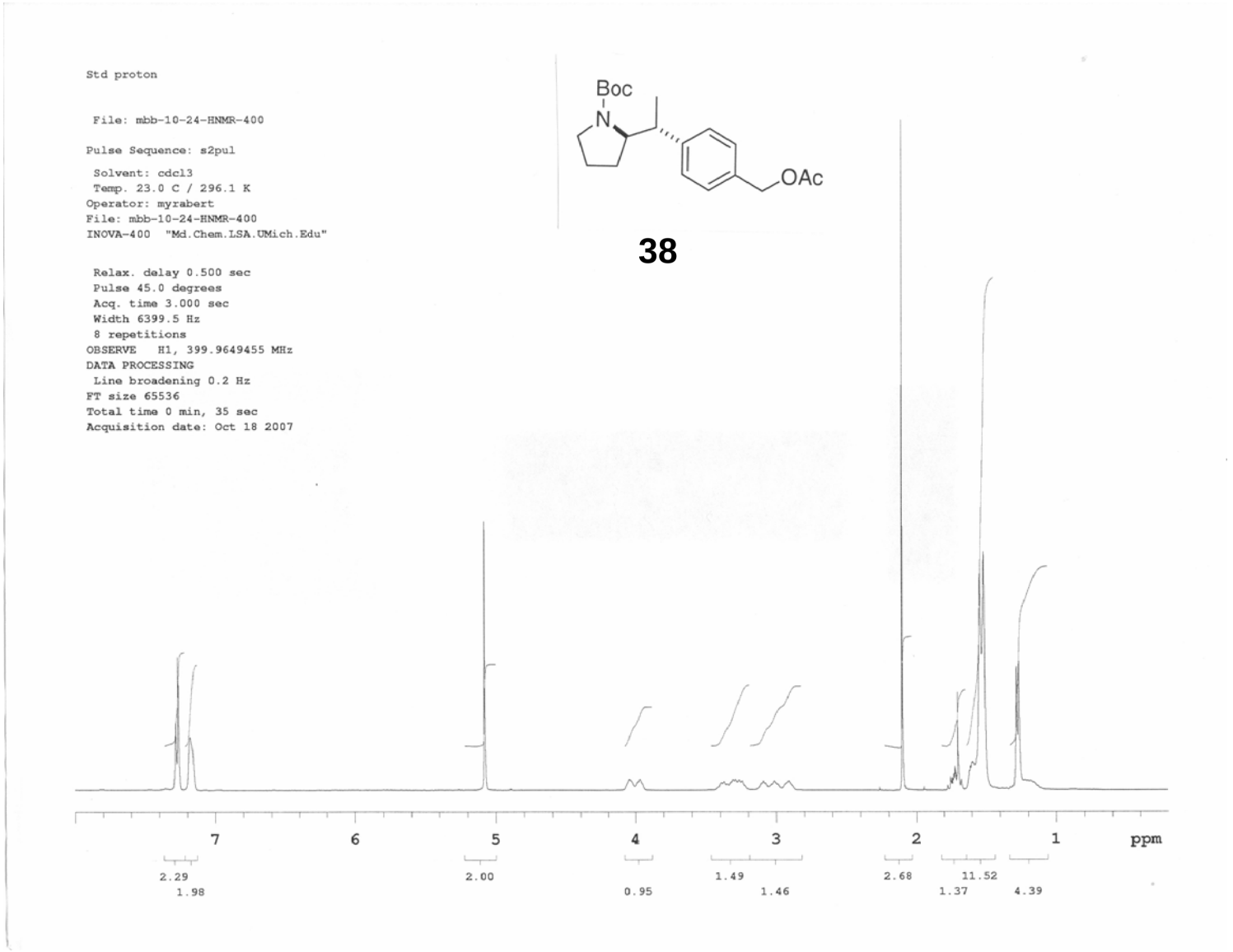
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
64 repetitions
OBSERVE C13, 100.5712703 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 min, 30 sec
Acquisition date: Oct 11 2007

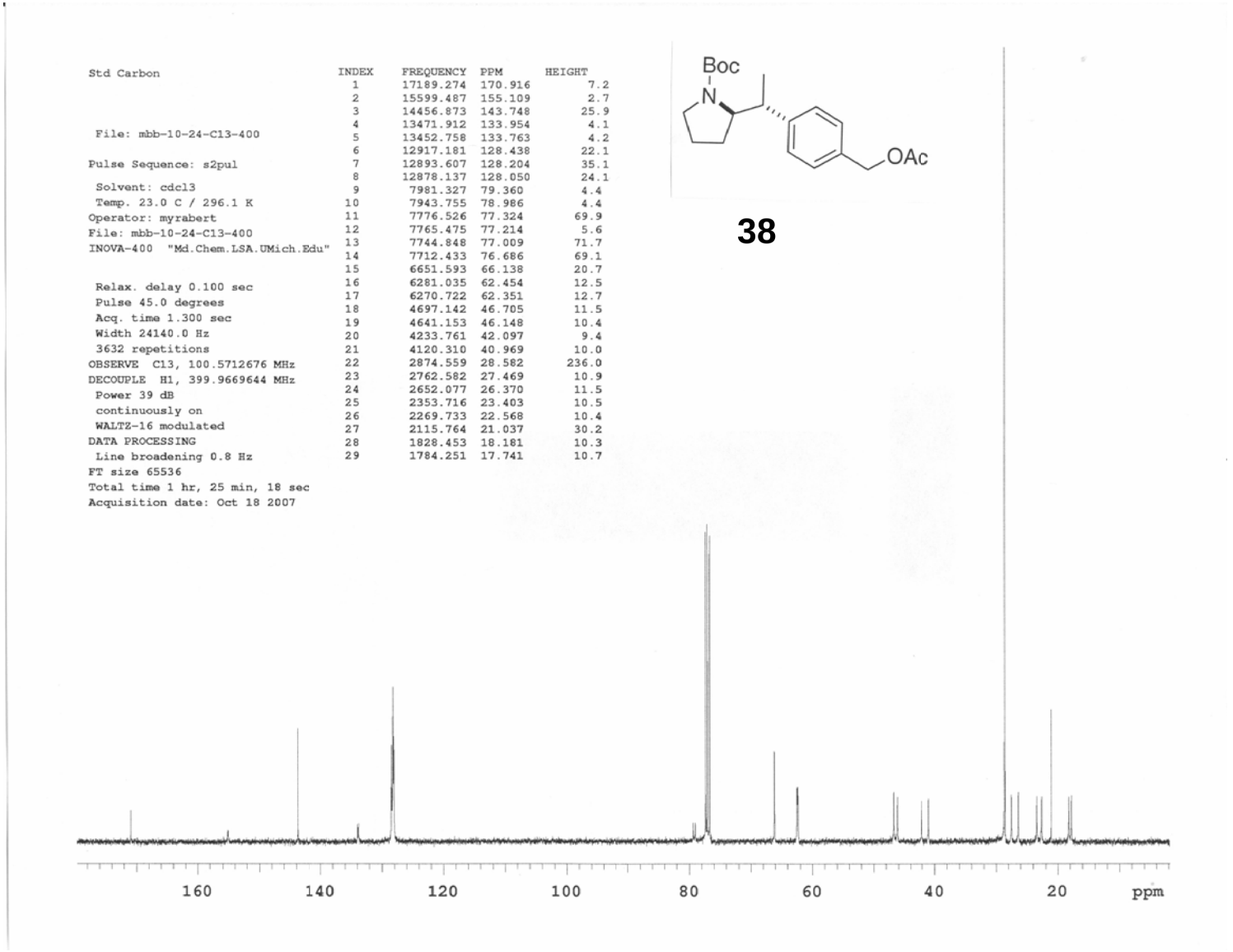
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16799.040 | 167.036 | 7.0 |
| 2 | 15207.043 | 151.207 | 19.1 |
| 3 | 13036.740 | 129.627 | 69.9 |
| 4 | 12881.298 | 128.081 | 18.9 |
| 5 | 12827.519 | 127.547 | 71.0 |
| 6 | 7776.004 | 77.318 | 37.4 |
| 7 | 7744.326 | 77.003 | 36.5 |
| 8 | 7711.911 | 76.681 | 37.9 |
| 9 | 6519.202 | 64.822 | 47.8 |
| 10 | 5223.356 | 51.937 | 29.5 |
| 11 | 4692.199 | 46.655 | 41.0 |
| 12 | 4650.208 | 46.238 | 41.6 |
| 13 | 3048.634 | 30.313 | 45.4 |
| 14 | 2537.367 | 25.230 | 38.0 |
| 15 | 1980.426 | 19.692 | 41.0 |




**37a**




Std proton
File: mbb-10-24-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-24-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649455 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Oct 18 2007
Boc
N
OAc
38
7
6
5
4
3
2
1
ppm
2.29
1.98
2.00
0.95
1.49
1.46
2.68
11.52
1.37
4.39


Std Carbon
File: mbb-10-24-C13-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-24-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
3632 repetitions
OBSERVE C13, 100.5712676 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 25 min, 18 sec
Acquisition date: Oct 18 2007
INDEX FREQUENCY PPM HEIGHT
1 17189.274 170.916 7.2
2 15599.487 155.109 2.7
3 14456.873 143.748 25.9
4 13471.912 133.954 4.1
5 13452.758 133.763 4.2
6 12917.181 128.438 22.1
7 12893.607 128.204 35.1
8 12878.137 128.050 24.1
9 7981.327 79.360 4.4
10 7943.755 78.986 4.4
11 7776.526 77.324 69.9
12 7765.475 77.214 5.6
13 7744.848 77.009 71.7
14 7712.433 76.686 69.1
15 6651.593 66.138 20.7
16 6281.035 62.454 12.5
17 6270.722 62.351 12.7
18 4697.142 46.705 11.5
19 4641.153 46.148 10.4
20 4233.761 42.097 9.4
21 4120.310 40.969 10.0
22 2874.559 28.582 236.0
23 2762.582 27.469 10.9
24 2652.077 26.370 11.5
25 2353.716 23.403 10.5
26 2269.733 22.568 10.4
27 2115.764 21.037 30.2
28 1828.453 18.181 10.3
29 1784.251 17.741 10.7
Boc
N
OAc
38
160
140
120
100
80
60
40
20
ppm

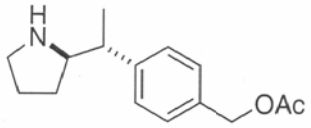
Standard Proton

File: mbb-10-31-HNMR-cr-500

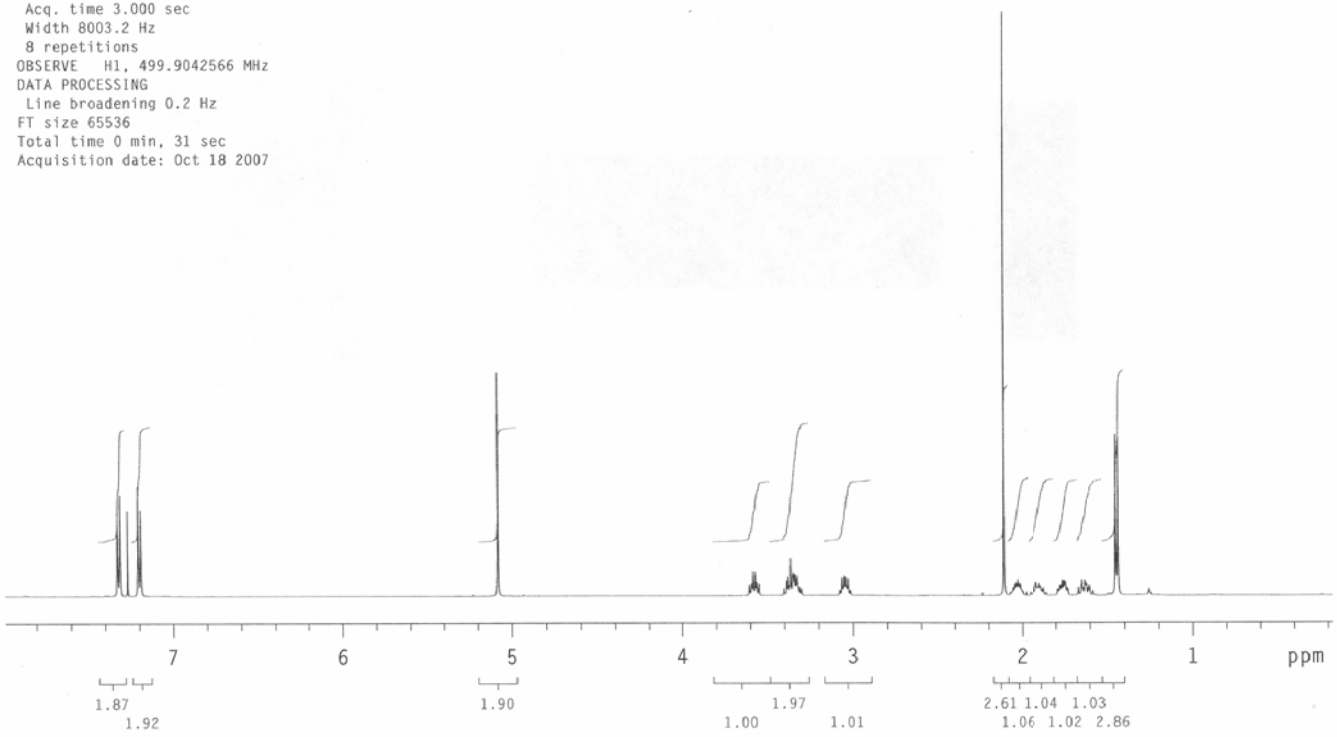
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-31-HNMR-cr-500
INOVA-500 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042566 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Oct 18 2007




**38a**



Standard Carbon

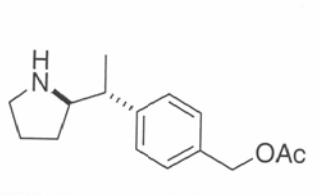
File: mbb-10-31-C13-500

Pulse Sequence: s2pul

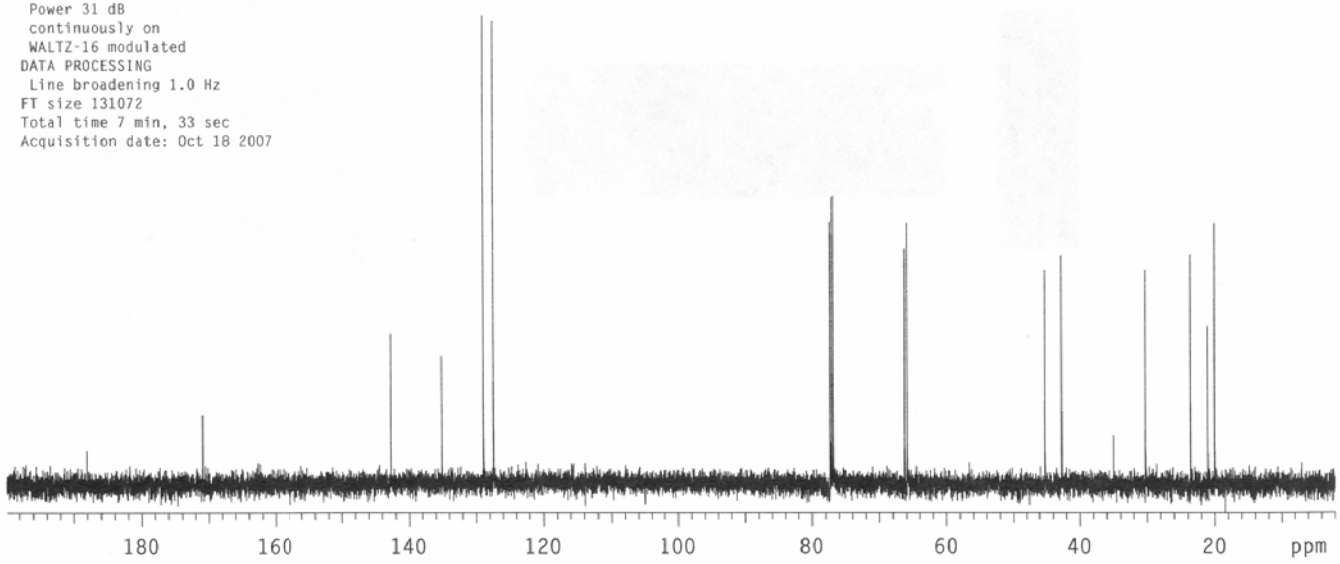
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-31-C13-500
INOVA-500 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
320 repetitions
OBSERVE C13, 125.7010246 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 min, 33 sec
Acquisition date: Oct 18 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 23634.437 | 188.021 | 6.4 |
| 2 | 21480.255 | 170.884 | 13.0 |
| 3 | 17933.220 | 142.666 | 28.1 |
| 4 | 16980.408 | 135.086 | 23.9 |
| 5 | 16196.065 | 128.846 | 87.2 |
| 6 | 16010.566 | 127.370 | 86.2 |
| 7 | 9714.647 | 77.284 | 48.7 |
| 8 | 9682.426 | 77.027 | 53.3 |
| 9 | 9650.666 | 76.775 | 53.6 |
| 10 | 8321.794 | 66.203 | 43.8 |
| 11 | 8279.446 | 65.866 | 48.5 |
| 12 | 5679.699 | 45.184 | 39.8 |
| 13 | 5365.777 | 42.687 | 42.5 |
| 14 | 4398.697 | 34.993 | 9.2 |
| 15 | 3803.535 | 30.259 | 39.7 |
| 16 | 2963.036 | 23.572 | 42.6 |
| 17 | 2639.908 | 21.001 | 29.4 |
| 18 | 2514.708 | 20.005 | 48.5 |




**38a**



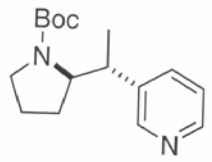
Std proton

File: mbb-10-72-HNMR-400

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-72-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649445 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Dec 17 2007




**39**

Std Carbon

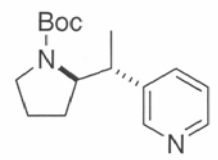
File: mbb-10-72-C13-400

Pulse Sequence: s2pul

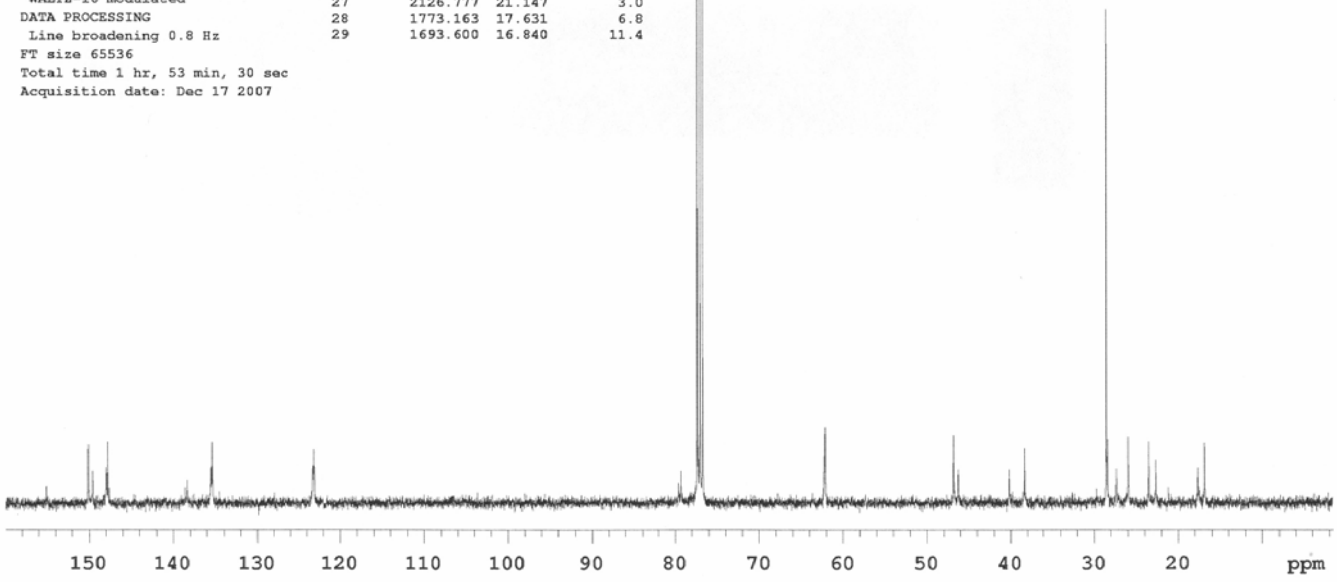
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-72-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

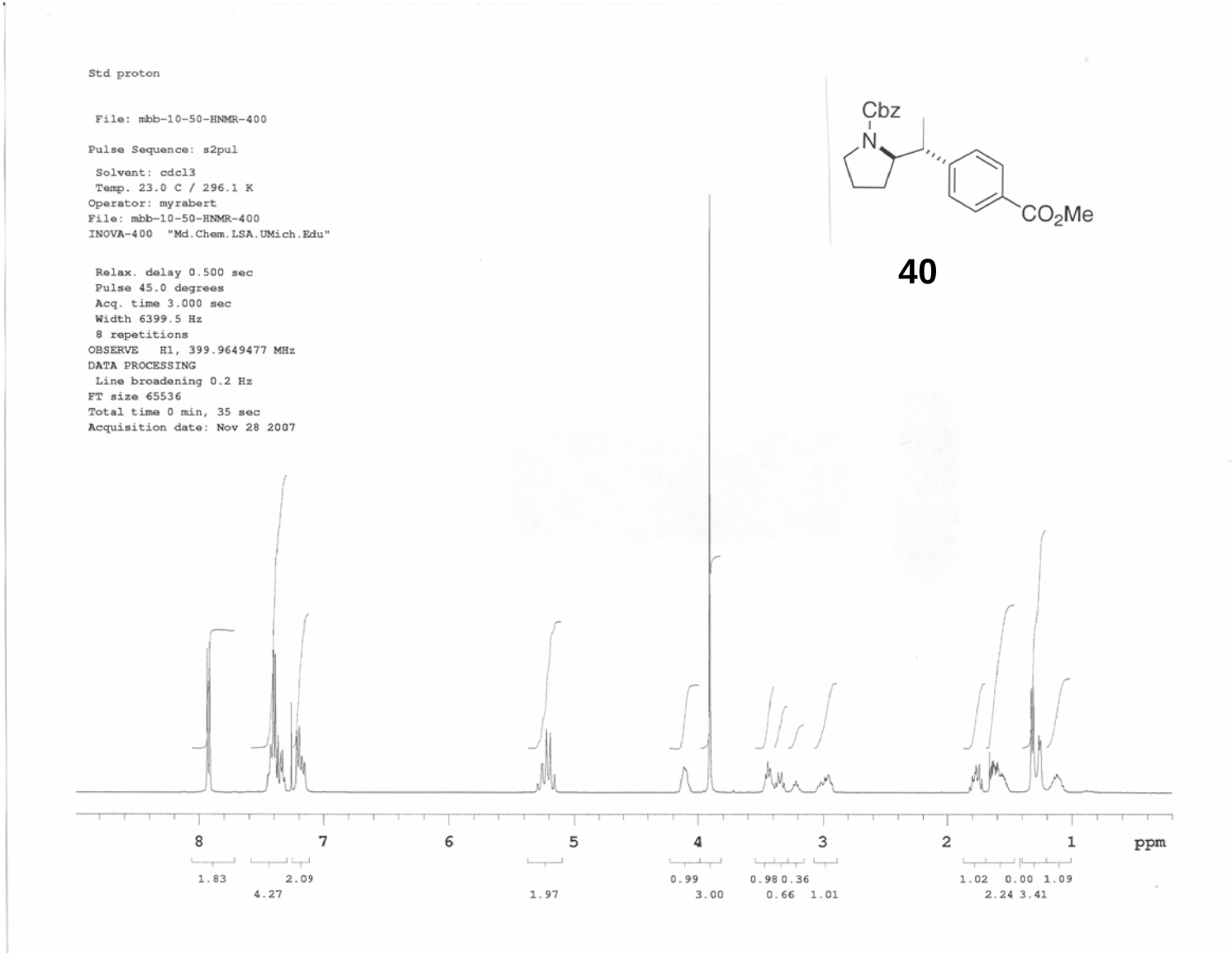
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
4832 repetitions
OBSERVE C13, 100.5712669 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 53 min, 30 sec
Acquisition date: Dec 17 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15596.503 | 155.079 | 3.3 |
| 2 | 15094.077 | 150.083 | 11.1 |
| 3 | 15042.508 | 149.571 | 6.1 |
| 4 | 14880.436 | 147.959 | 6.8 |
| 5 | 14859.808 | 147.754 | 11.6 |
| 6 | 13910.945 | 138.319 | 4.4 |
| 7 | 13626.581 | 135.492 | 6.7 |
| 8 | 13613.321 | 135.360 | 11.5 |
| 9 | 12380.830 | 123.105 | 10.2 |
| 10 | 8004.864 | 79.594 | 3.8 |
| 11 | 7973.186 | 79.279 | 6.1 |
| 12 | 7777.225 | 77.330 | 154.3 |
| 13 | 7765.438 | 77.213 | 10.6 |
| 14 | 7744.811 | 77.008 | 162.0 |
| 15 | 7713.133 | 76.693 | 160.9 |
| 16 | 6246.374 | 62.109 | 14.4 |
| 17 | 4705.945 | 46.792 | 12.8 |
| 18 | 4651.430 | 46.250 | 6.4 |
| 19 | 4035.553 | 40.126 | 6.5 |
| 20 | 3855.799 | 38.339 | 10.4 |
| 21 | 2871.575 | 28.553 | 92.0 |
| 22 | 2854.631 | 28.384 | 12.1 |
| 23 | 2749.284 | 27.337 | 6.6 |
| 24 | 2607.838 | 25.930 | 12.6 |
| 25 | 2359.572 | 23.462 | 11.6 |
| 26 | 2277.799 | 22.649 | 8.2 |
| 27 | 2126.777 | 21.147 | 3.0 |
| 28 | 1773.163 | 17.631 | 6.8 |
| 29 | 1693.600 | 16.840 | 11.4 |




**39**




Std proton
File: mbb-10-50-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-50-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649477 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Nov 28 2007
Cbz
N
CO2Me
40
8
7
6
5
4
3
2
1
ppm
1.83
2.09
4.27
1.97
0.99
3.00
0.98
0.36
0.66
1.01
1.02
0.00
1.09
2.24
3.41

Std Carbon

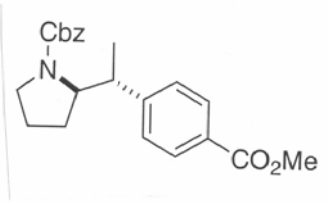
File: mbb-10-50-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-50-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
3680 repetitions
OBSERVE C13, 100.5712684 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 26 min, 26 sec
Acquisition date: Nov 28 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16806.146 | 167.107 | 7.4 |
| 2 | 15623.014 | 155.343 | 11.9 |
| 3 | 14948.938 | 148.640 | 12.2 |
| 4 | 14939.361 | 148.545 | 6.2 |
| 5 | 13793.064 | 137.147 | 7.0 |
| 6 | 13759.913 | 136.818 | 3.5 |
| 7 | 13017.325 | 129.434 | 21.5 |
| 8 | 13005.538 | 129.317 | 44.5 |
| 9 | 12920.081 | 128.467 | 44.8 |
| 10 | 12903.137 | 128.298 | 50.1 |
| 11 | 12885.456 | 128.123 | 24.9 |
| 12 | 12879.563 | 128.064 | 25.7 |
| 13 | 12864.829 | 127.918 | 25.0 |
| 14 | 12852.305 | 127.793 | 35.4 |
| 15 | 7776.479 | 77.323 | 152.7 |
| 16 | 7764.692 | 77.206 | 10.5 |
| 17 | 7744.801 | 77.008 | 147.4 |
| 18 | 7712.386 | 76.686 | 152.5 |
| 19 | 6738.476 | 67.002 | 10.9 |
| 20 | 6699.431 | 66.614 | 23.2 |
| 21 | 6331.820 | 62.959 | 22.5 |
| 22 | 6276.568 | 62.409 | 9.9 |
| 23 | 5229.725 | 52.000 | 53.0 |
| 24 | 4708.882 | 46.821 | 11.7 |
| 25 | 4682.361 | 46.558 | 22.5 |
| 26 | 4238.134 | 42.141 | 9.9 |
| 27 | 4112.159 | 40.888 | 21.4 |
| 28 | 2984.280 | 29.673 | 3.1 |
| 29 | 2740.434 | 27.249 | 11.3 |
| 30 | 2627.719 | 26.128 | 23.8 |
| 31 | 2354.406 | 23.410 | 22.9 |
| 32 | 2271.159 | 22.583 | 10.2 |
| 33 | 1776.837 | 17.667 | 9.5 |
| 34 | 1745.159 | 17.352 | 21.4 |

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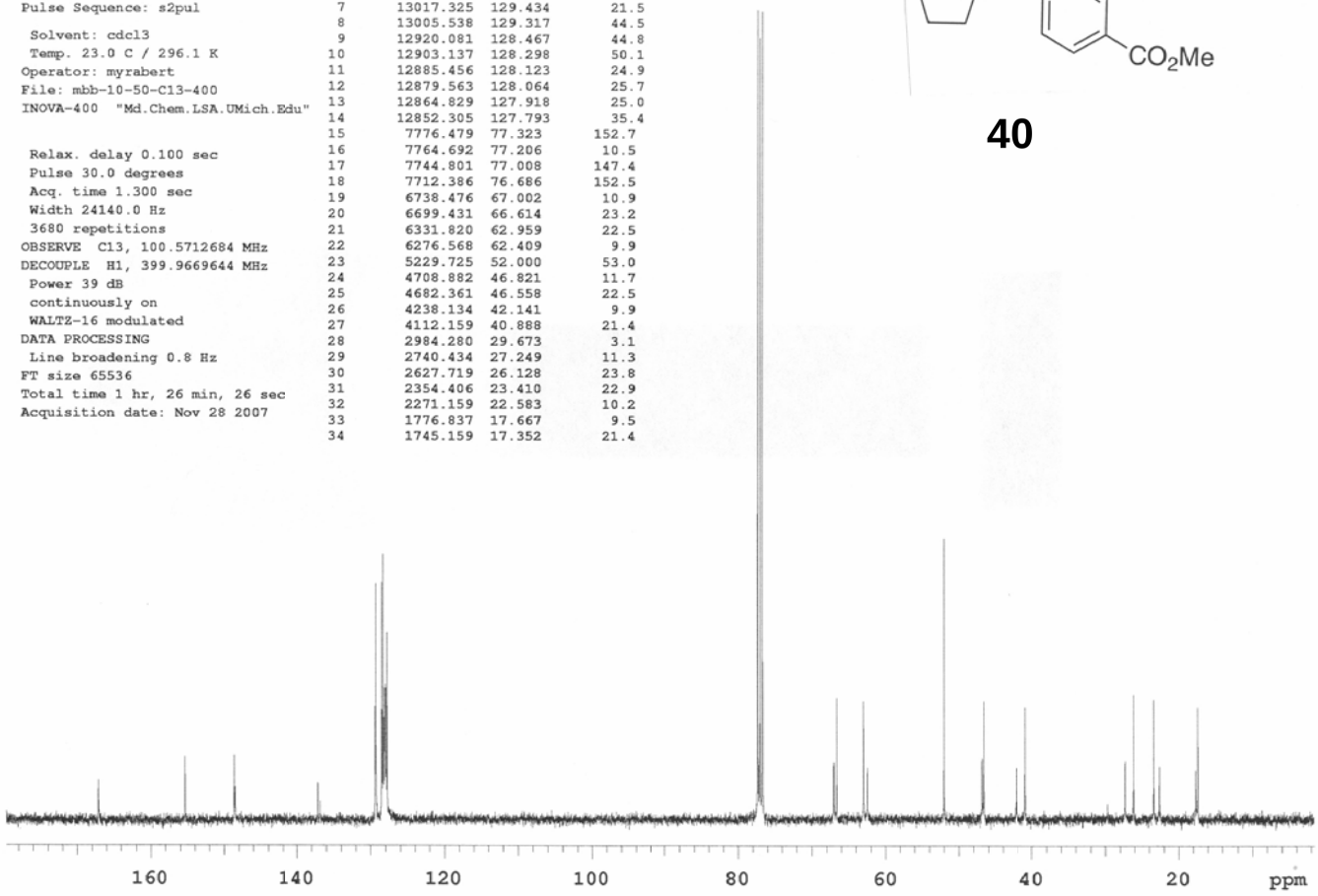
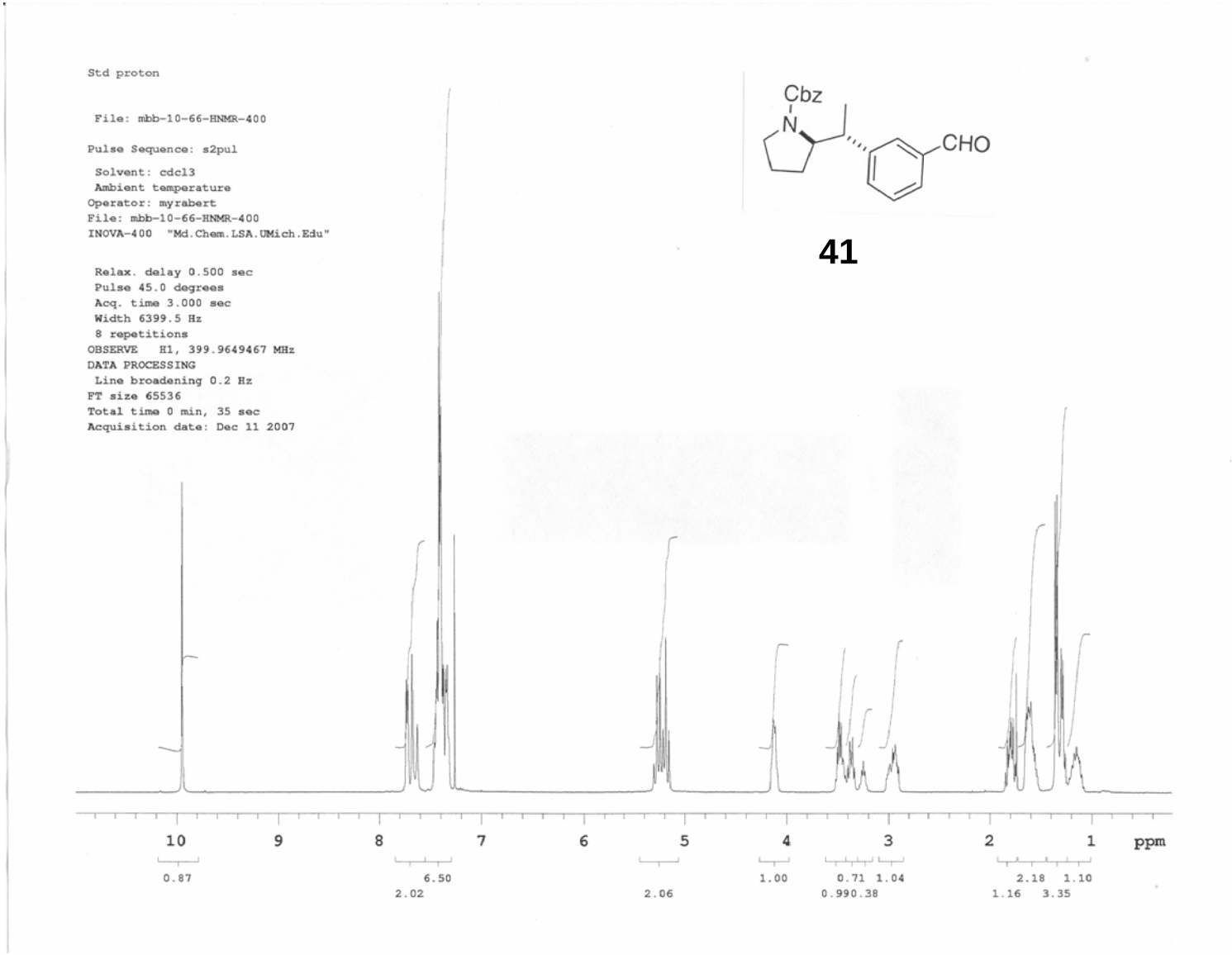



Std proton
File: mbb-10-66-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-66-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649467 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Dec 11 2007
Cbz
N
CHO
41
10
9
8
7
6
5
4
3
2
1
ppm
0.87
6.50
2.02
2.06
1.00
0.71
1.04
0.99
0.38
2.18
1.10
1.16
3.35

Std Carbon

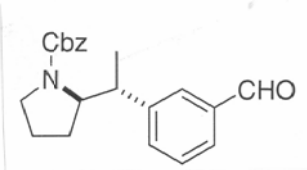
File: mbb-10-66-C13-400

Pulse Sequence: s2pul

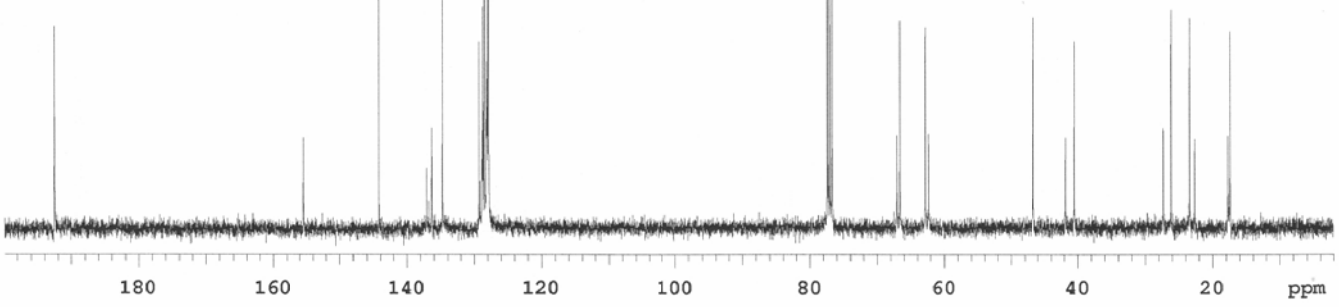
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-66-C13-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"

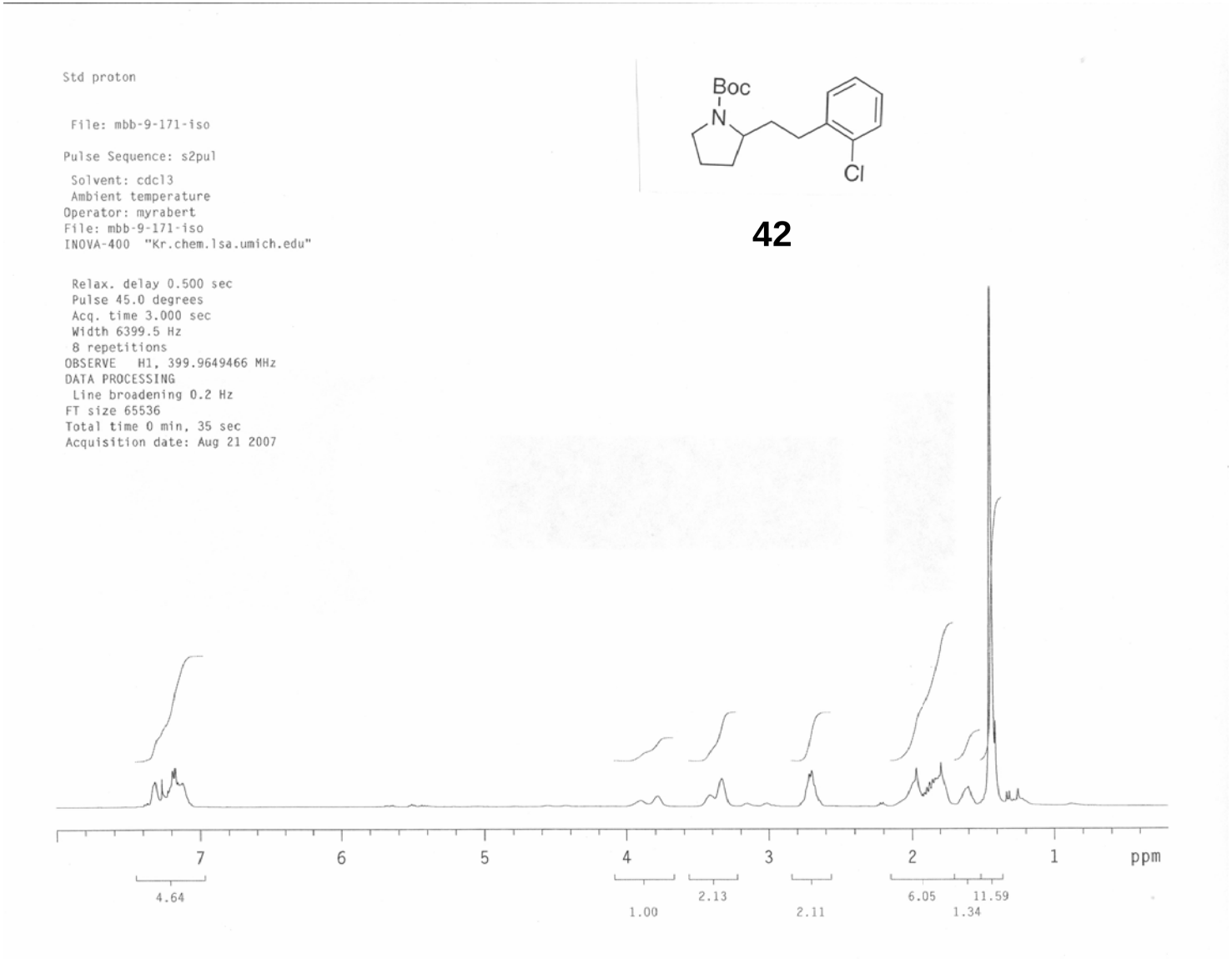
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
1696 repetitions
OBSERVE C13, 100.5712706 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 39 min, 50 sec
Acquisition date: Dec 11 2007

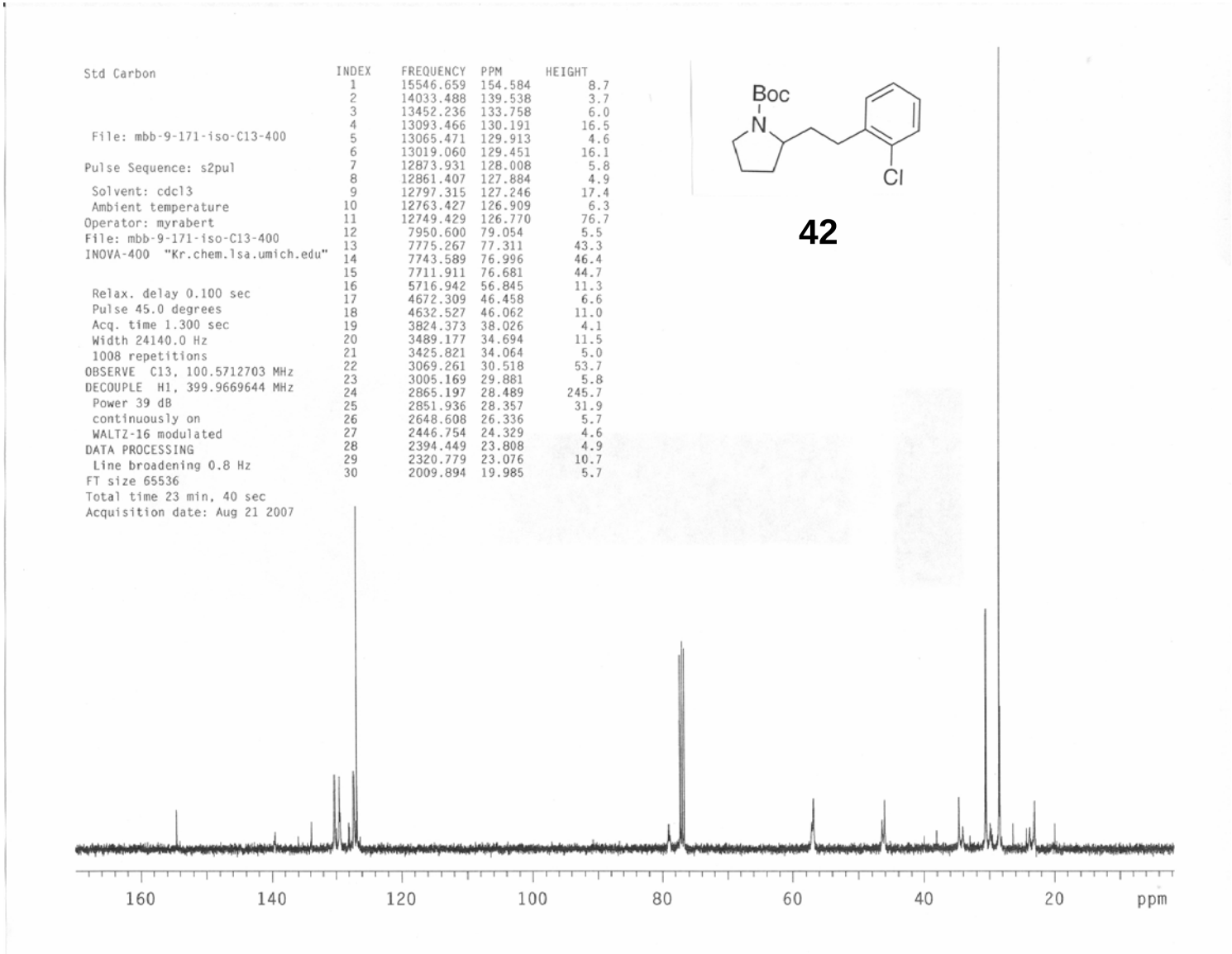
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19365.396 | 192.554 | 37.6 |
| 2 | 15626.670 | 155.379 | 16.9 |
| 3 | 14502.474 | 144.201 | 78.0 |
| 4 | 13787.880 | 137.096 | 11.3 |
| 5 | 13704.634 | 136.268 | 18.8 |
| 6 | 13546.245 | 134.693 | 133.0 |
| 7 | 12997.407 | 129.236 | 34.8 |
| 8 | 12959.836 | 128.862 | 18.3 |
| 9 | 12948.785 | 128.752 | 41.2 |
| 10 | 12940.682 | 128.672 | 18.4 |
| 11 | 12920.791 | 128.474 | 71.7 |
| 12 | 12887.640 | 128.144 | 31.1 |
| 13 | 12880.273 | 128.071 | 24.3 |
| 14 | 12866.275 | 127.932 | 74.5 |
| 15 | 12857.435 | 127.844 | 63.9 |
| 16 | 7776.452 | 77.323 | 166.8 |
| 17 | 7765.401 | 77.213 | 12.0 |
| 18 | 7744.774 | 77.008 | 176.0 |
| 19 | 7712.359 | 76.686 | 160.0 |
| 20 | 6740.659 | 67.024 | 17.5 |
| 21 | 6700.877 | 66.628 | 38.7 |
| 22 | 6318.533 | 62.826 | 37.4 |
| 23 | 6265.491 | 62.299 | 17.7 |
| 24 | 4710.328 | 46.836 | 17.7 |
| 25 | 4685.281 | 46.587 | 39.3 |
| 26 | 4204.956 | 41.811 | 17.0 |
| 27 | 4076.771 | 40.536 | 34.9 |
| 28 | 2741.880 | 27.263 | 18.8 |
| 29 | 2626.219 | 26.113 | 40.7 |
| 30 | 2356.589 | 23.432 | 39.2 |
| 31 | 2274.816 | 22.619 | 16.7 |
| 32 | 1779.020 | 17.689 | 17.3 |
| 33 | 1745.869 | 17.360 | 36.7 |




**41**




Std proton
File: mbb-9-171-iso
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-171-iso
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649466 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Aug 21 2007
Boc
N
Cl
42
7
6
5
4
3
2
1
ppm
4.64
1.00
2.13
2.11
6.05
1.34
11.59


Std Carbon
File: mbb-9-171-iso-C13-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-171-iso-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
1008 repetitions
OBSERVE C13, 100.5712703 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 23 min, 40 sec
Acquisition date: Aug 21 2007
INDEX FREQUENCY PPM HEIGHT
1 15546.659 154.584 8.7
2 14033.488 139.538 3.7
3 13452.236 133.758 6.0
4 13093.466 130.191 16.5
5 13065.471 129.913 4.6
6 13019.060 129.451 16.1
7 12873.931 128.008 5.8
8 12861.407 127.884 4.9
9 12797.315 127.246 17.4
10 12763.427 126.909 6.3
11 12749.429 126.770 76.7
12 7950.600 79.054 5.5
13 7775.267 77.311 43.3
14 7743.589 76.996 46.4
15 7711.911 76.681 44.7
16 5716.942 56.845 11.3
17 4672.309 46.458 6.6
18 4632.527 46.062 11.0
19 3824.373 38.026 4.1
20 3489.177 34.694 11.5
21 3425.821 34.064 5.0
22 3069.261 30.518 53.7
23 3005.169 29.881 5.8
24 2865.197 28.489 245.7
25 2851.936 28.357 31.9
26 2648.608 26.336 5.7
27 2446.754 24.329 4.6
28 2394.449 23.808 4.9
29 2320.779 23.076 10.7
30 2009.894 19.985 5.7
Boc
N
Cl
42
160
140
120
100
80
60
40
20
ppm

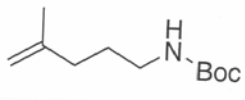
Standard Proton

File: mbb-9-66-HNMR-iso-500

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-66-HNMR-iso-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042149 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jun 21 2007

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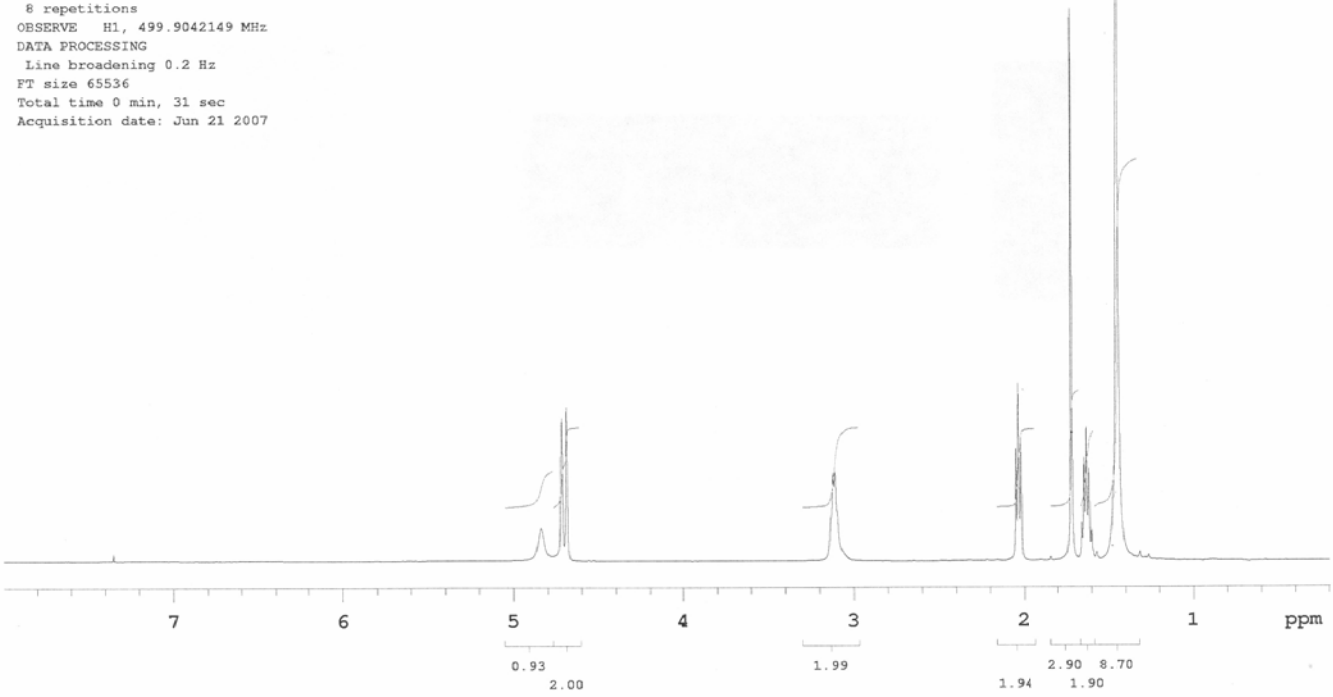


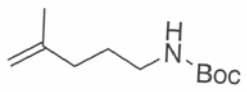
Standard Carbon

File: mbb-9-66-C13-500

Pulse Sequence: s2pul

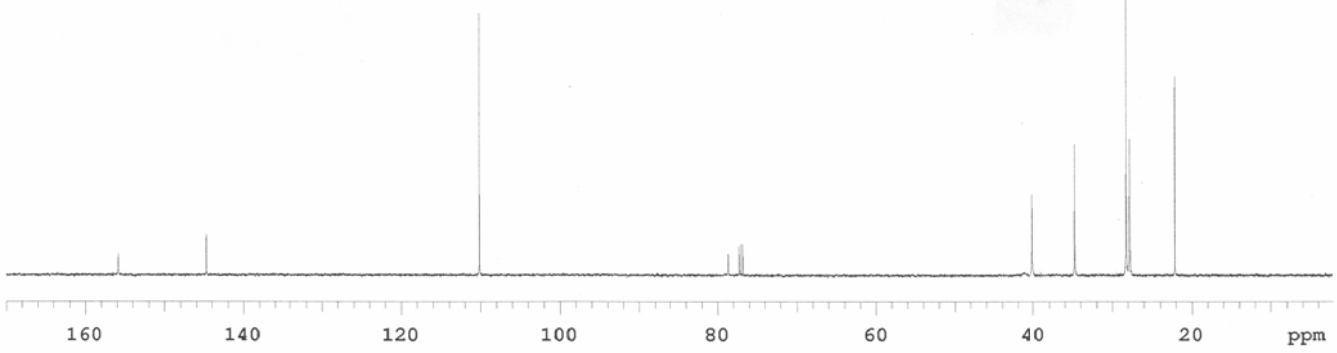
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-66-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

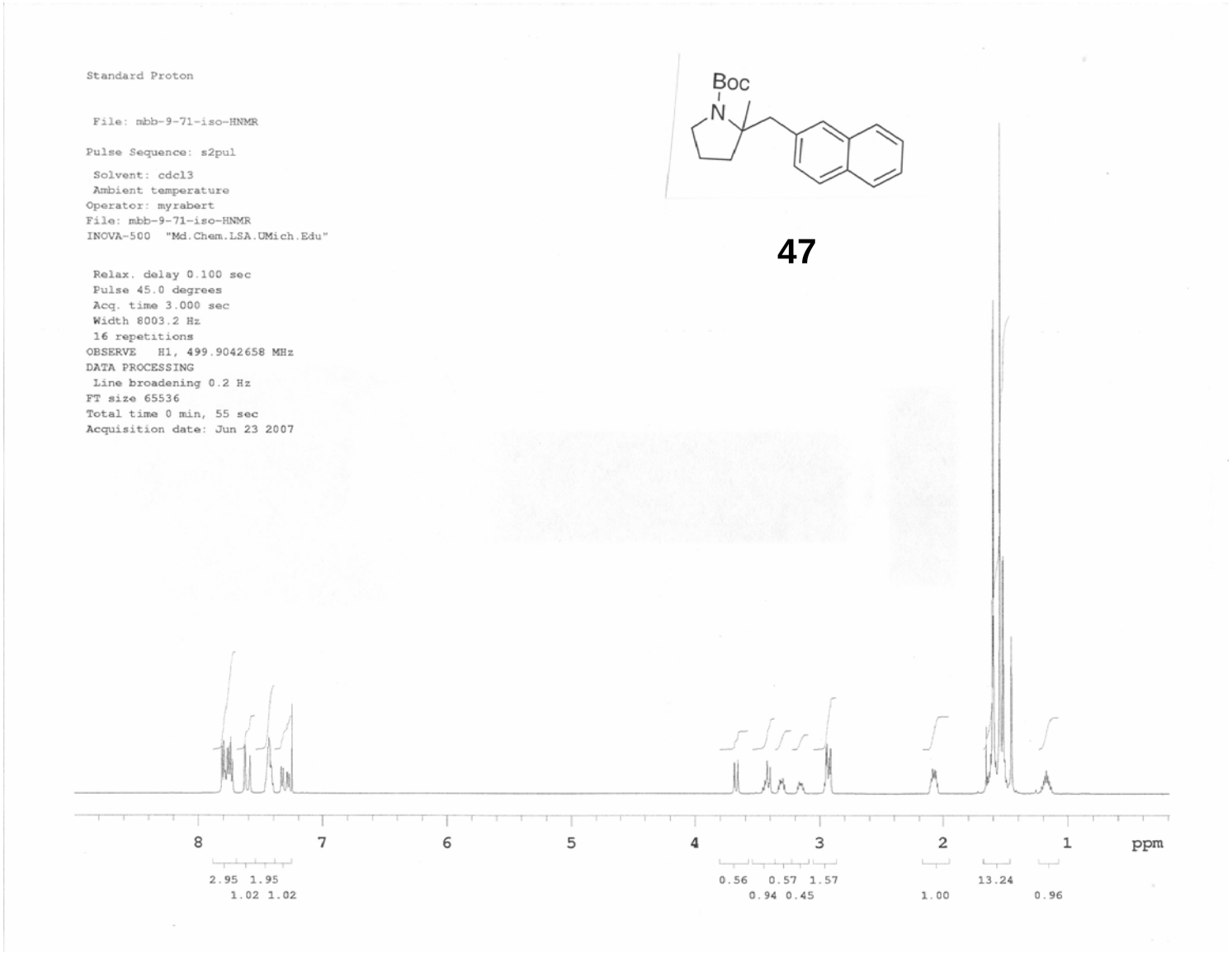
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19586.757 | 155.820 | 4.0 |
| 2 | 18191.602 | 144.721 | 7.5 |
| 3 | 13842.272 | 110.121 | 48.4 |
| 4 | 9884.653 | 78.636 | 4.1 |
| 5 | 9712.042 | 77.263 | 5.4 |
| 6 | 9679.821 | 77.007 | 5.9 |
| 7 | 9648.061 | 76.754 | 5.8 |
| 8 | 5039.585 | 40.092 | 15.1 |
| 9 | 4370.775 | 34.771 | 24.3 |
| 10 | 3546.847 | 28.217 | 148.5 |
| 11 | 3490.231 | 27.766 | 25.2 |
| 12 | 2776.773 | 22.090 | 36.8 |




**46**

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
240 repetitions
OBSERVE C13, 125.7010382 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 min, 41 sec
Acquisition date: Jun 21 2007




Standard Proton
File: mbb-9-71-iso-HNMR
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-71-iso-HNMR
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
16 repetitions
OBSERVE H1, 499.9042658 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Acquisition date: Jun 23 2007
Boc
N
47
8
7
6
5
4
3
2
1
ppm
2.95 1.95
1.02 1.02
0.56 0.57 1.57
0.94 0.45
1.00
13.24
0.96

Standard Carbon

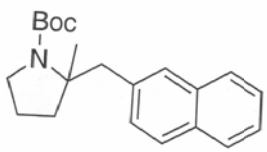
File: mbb-9-71-C13-500

Pulse Sequence: s2pul

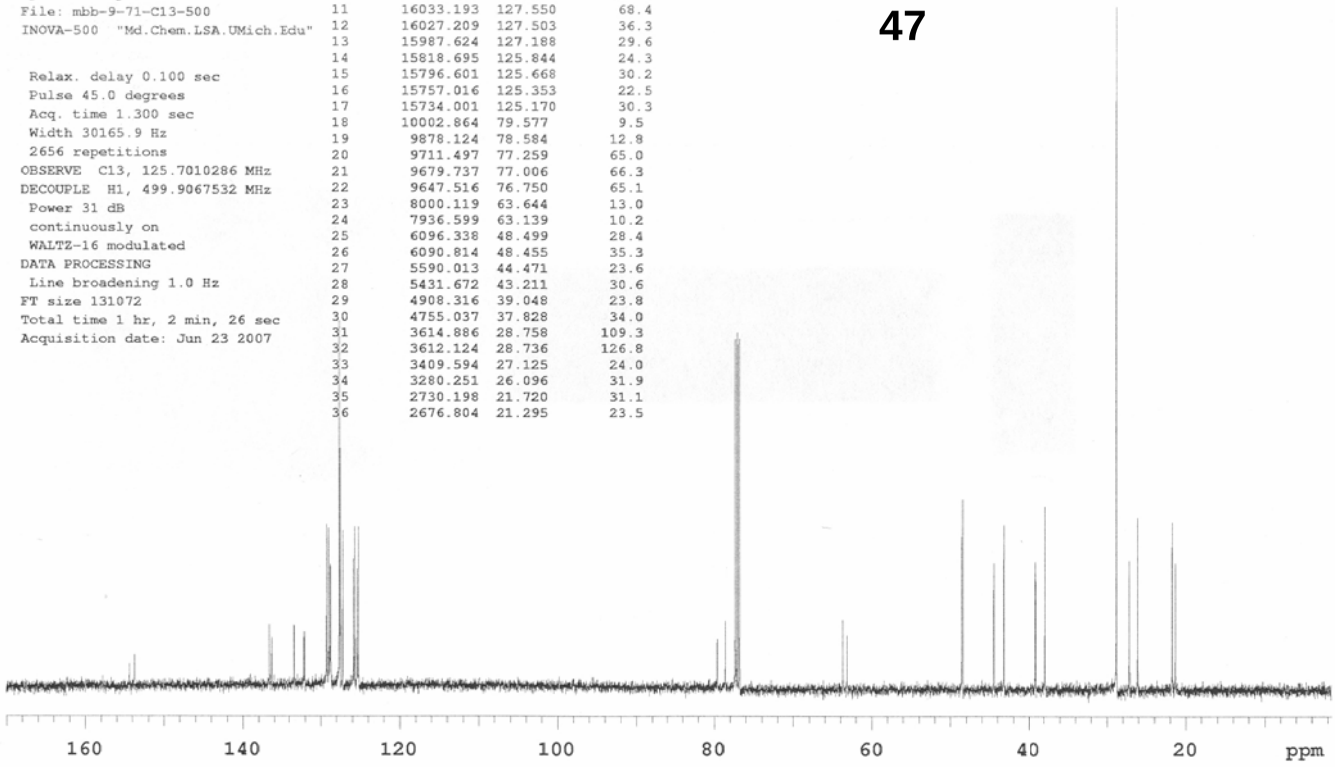
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-71-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

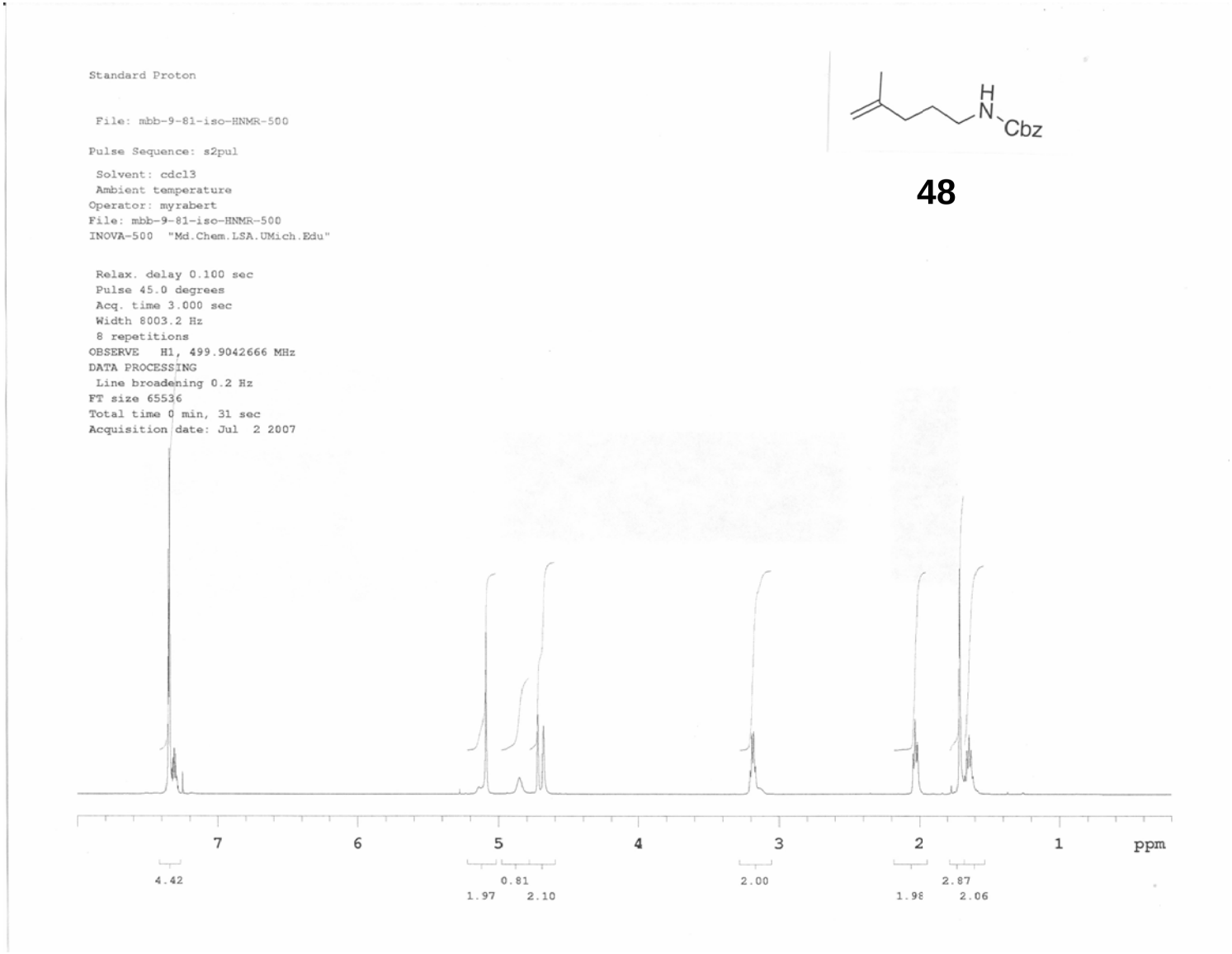
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
2656 repetitions
OBSERVE C13, 125.7010286 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 2 min, 26 sec
Acquisition date: Jun 23 2007

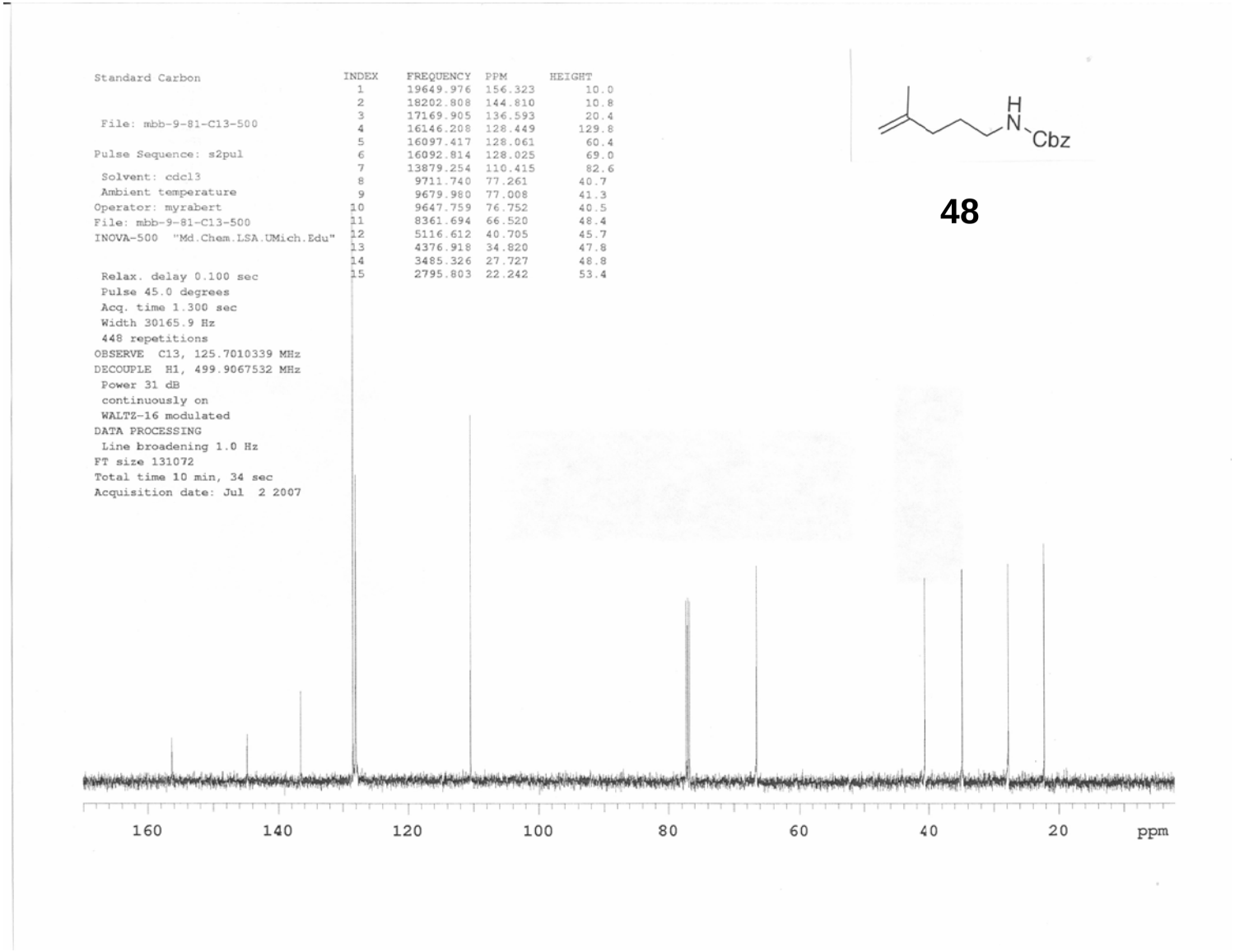
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19408.538 | 154.402 | 4.9 |
| 2 | 19327.526 | 153.758 | 6.6 |
| 3 | 17169.662 | 136.591 | 12.2 |
| 4 | 17137.441 | 136.335 | 9.7 |
| 5 | 16765.523 | 133.376 | 12.1 |
| 6 | 16597.975 | 132.043 | 10.8 |
| 7 | 16238.945 | 129.187 | 30.7 |
| 8 | 16208.105 | 128.942 | 29.9 |
| 9 | 16187.852 | 128.781 | 23.3 |
| 10 | 16178.646 | 128.707 | 22.9 |
| 11 | 16033.193 | 127.550 | 68.4 |
| 12 | 16027.209 | 127.503 | 36.3 |
| 13 | 15987.624 | 127.188 | 29.6 |
| 14 | 15818.695 | 125.844 | 24.3 |
| 15 | 15796.601 | 125.668 | 30.2 |
| 16 | 15757.016 | 125.353 | 22.5 |
| 17 | 15734.001 | 125.170 | 30.3 |
| 18 | 10002.864 | 79.577 | 9.5 |
| 19 | 9878.124 | 78.584 | 12.8 |
| 20 | 9711.497 | 77.259 | 65.0 |
| 21 | 9679.737 | 77.006 | 66.3 |
| 22 | 9647.516 | 76.750 | 65.1 |
| 23 | 8000.119 | 63.644 | 13.0 |
| 24 | 7936.599 | 63.139 | 10.2 |
| 25 | 6096.338 | 48.499 | 28.4 |
| 26 | 6090.814 | 48.455 | 35.3 |
| 27 | 5590.013 | 44.471 | 23.6 |
| 28 | 5431.672 | 43.211 | 30.6 |
| 29 | 4908.316 | 39.048 | 23.8 |
| 30 | 4755.037 | 37.828 | 34.0 |
| 31 | 3614.886 | 28.758 | 109.3 |
| 32 | 3612.124 | 28.736 | 126.8 |
| 33 | 3409.594 | 27.125 | 24.0 |
| 34 | 3280.251 | 26.096 | 31.9 |
| 35 | 2730.198 | 21.720 | 31.1 |
| 36 | 2676.804 | 21.295 | 23.5 |

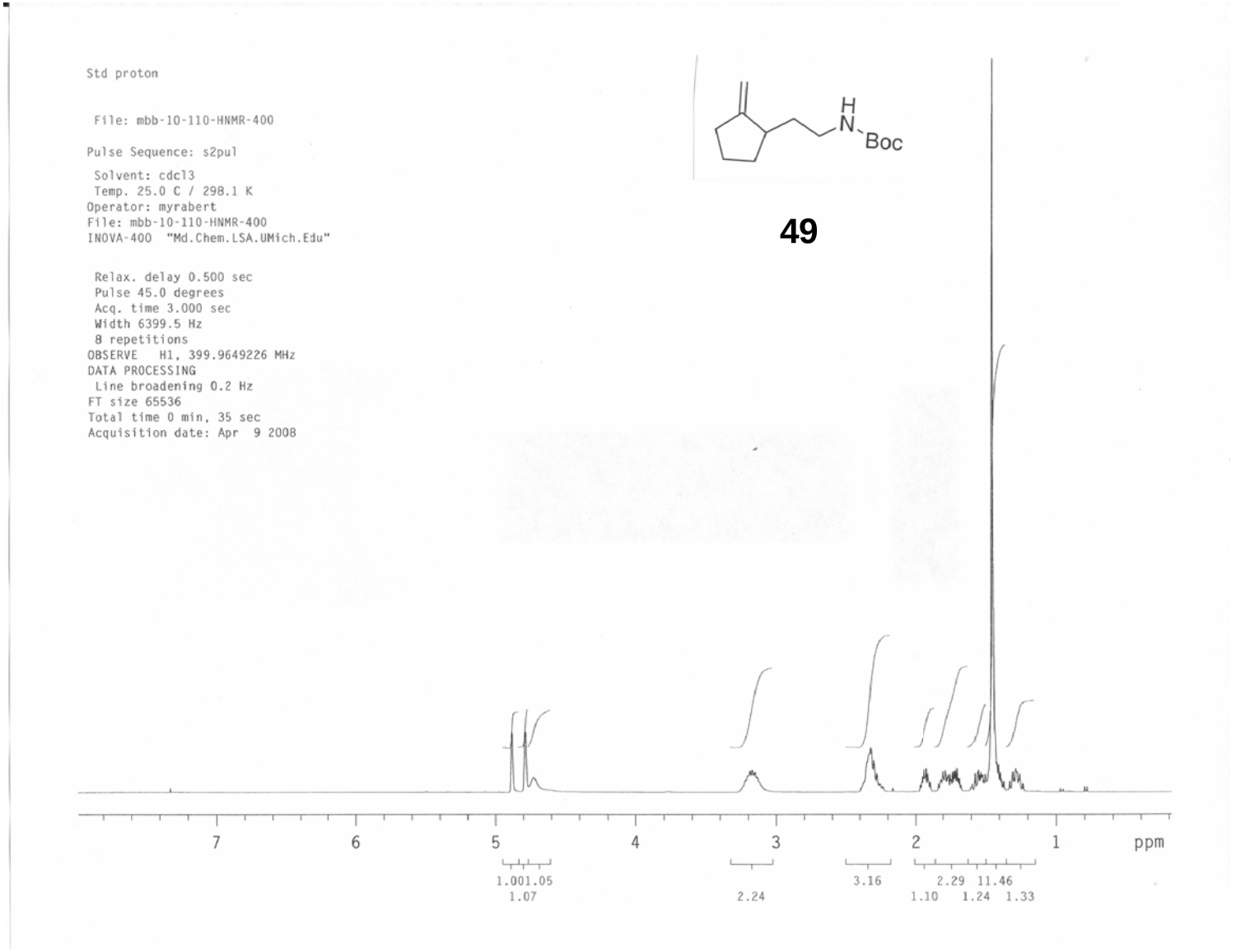


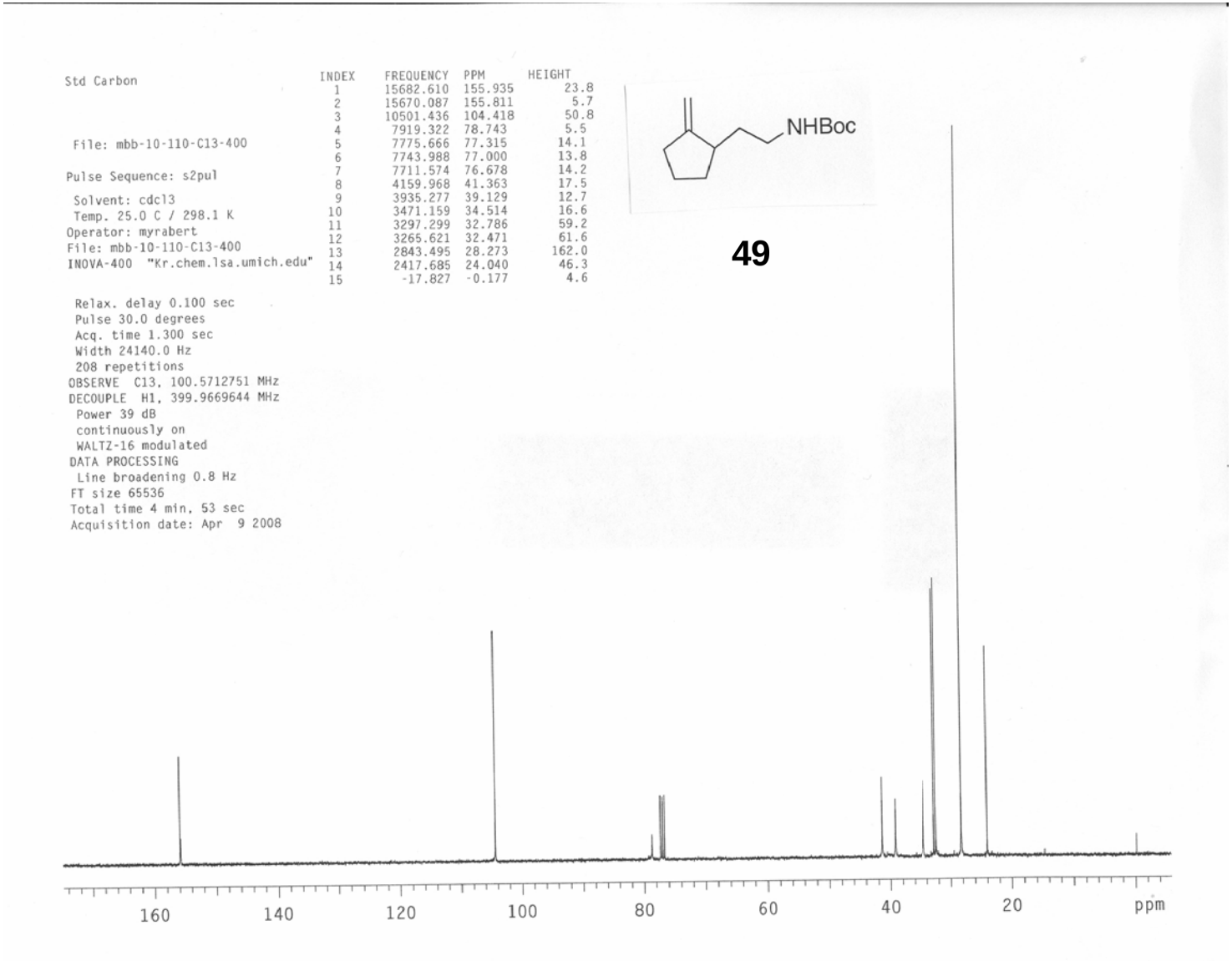

**47**

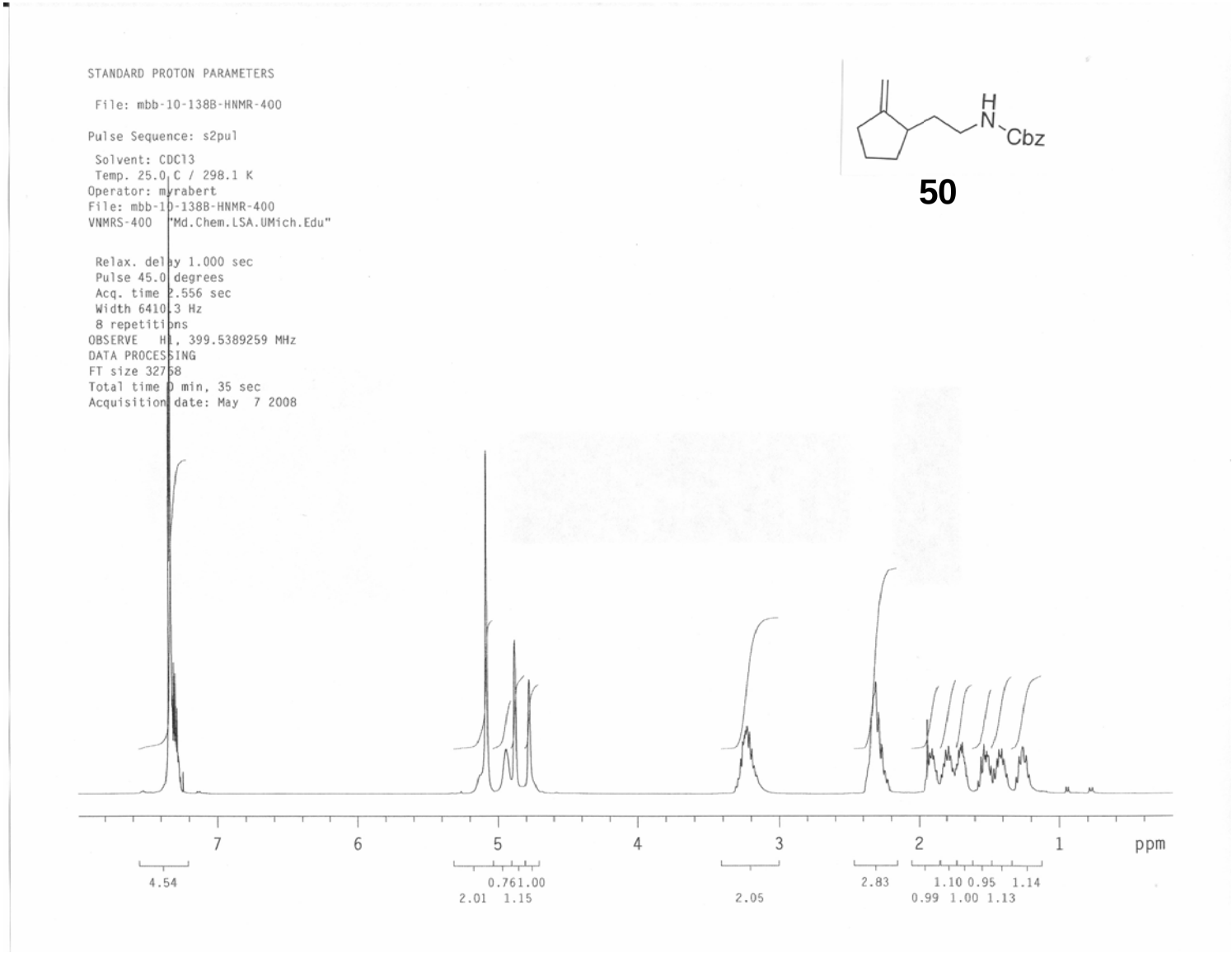


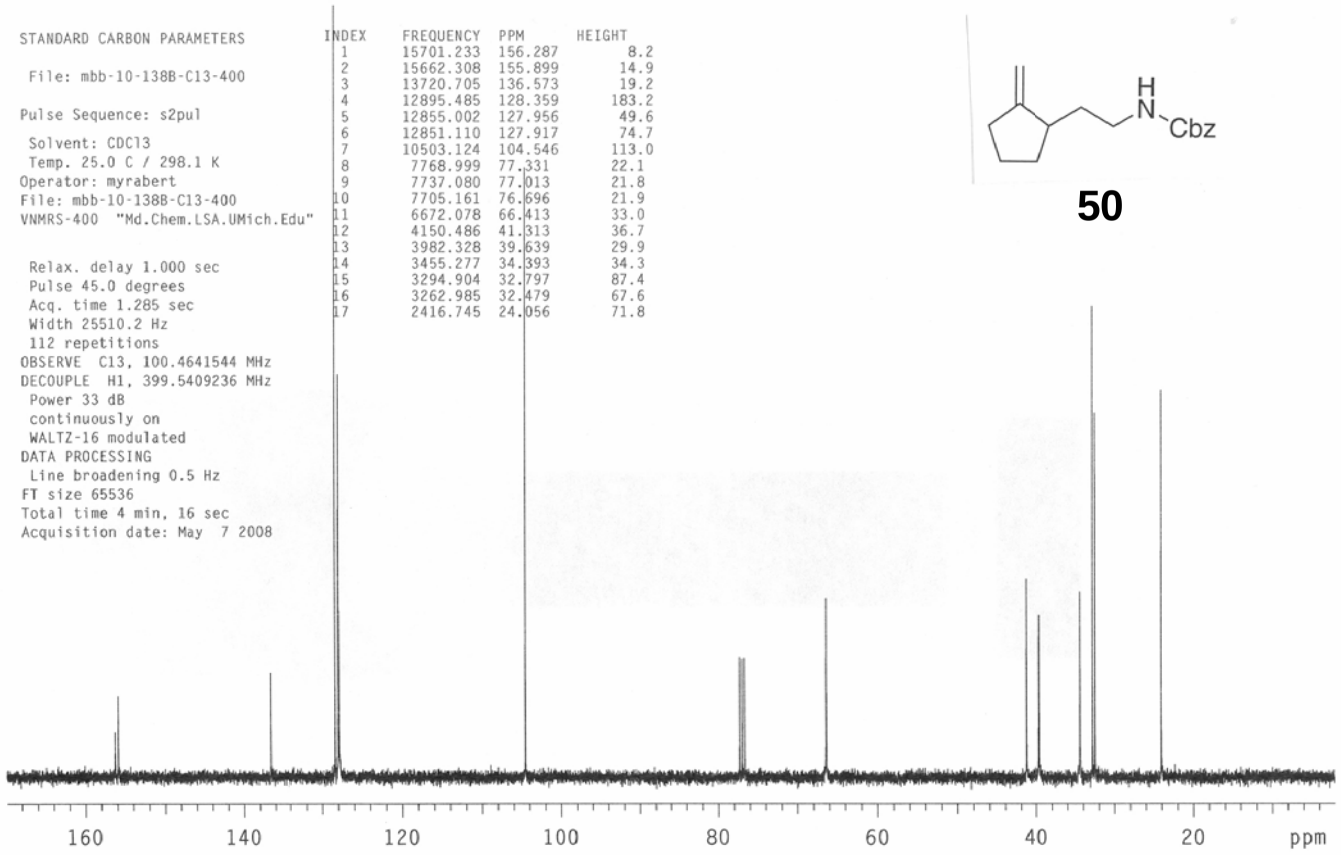

Standard Proton
File: mbb-9-81-iso-HNMR-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-81-iso-HNMR-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042666 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jul 2 2007
H
N
Cbz
48
7
6
5
4
3
2
1
ppm
4.42
0.81
1.97
2.10
2.00
1.98
2.87
2.06


Standard Carbon
File: mbb-9-81-C13-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-81-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
448 repetitions
OBSERVE C13, 125.7010339 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 10 min, 34 sec
Acquisition date: Jul 2 2007
INDEX FREQUENCY PPM HEIGHT
1 19649.976 156.323 10.0
2 18202.808 144.810 10.8
3 17169.905 136.593 20.4
4 16146.208 128.449 129.8
5 16097.417 128.061 60.4
6 16092.814 128.025 69.0
7 13879.254 110.415 82.6
8 9711.740 77.261 40.7
9 9679.980 77.008 41.3
10 9647.759 76.752 40.5
11 8361.694 66.520 48.4
12 5116.612 40.705 45.7
13 4376.918 34.820 47.8
14 3485.326 27.727 48.8
15 2795.803 22.242 53.4
H
N
Cbz
48
160
140
120
100
80
60
40
20
ppm

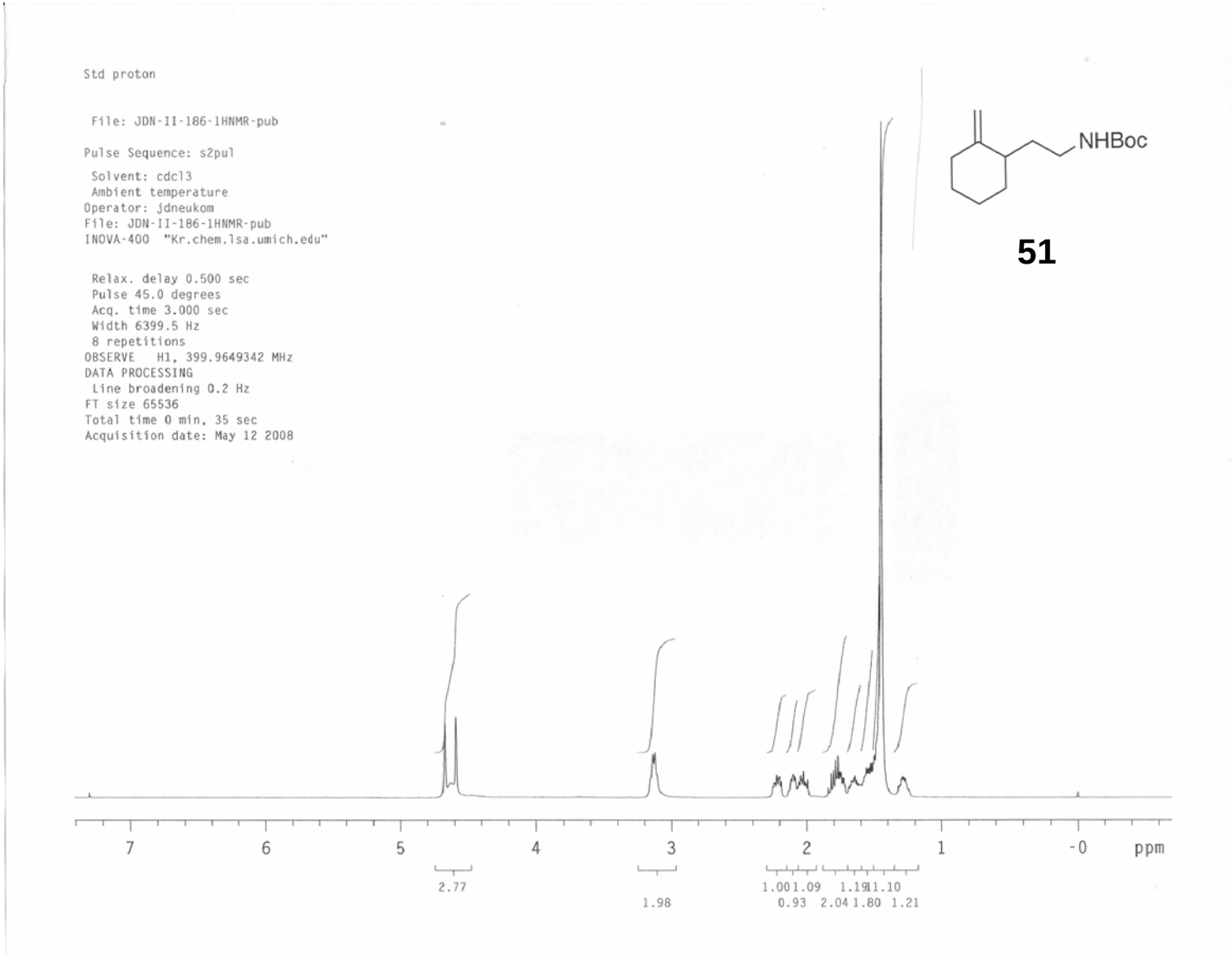

Std proton
File: mbb-10-110-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: myrabert
File: mbb-10-110-HNMR-400
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649226 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Apr 9 2008
H
N
Boc
49
7
6
5
4
3
2
1
ppm
1.00 1.05
1.07
2.24
3.16
2.29 11.46
1.10 1.24 1.33

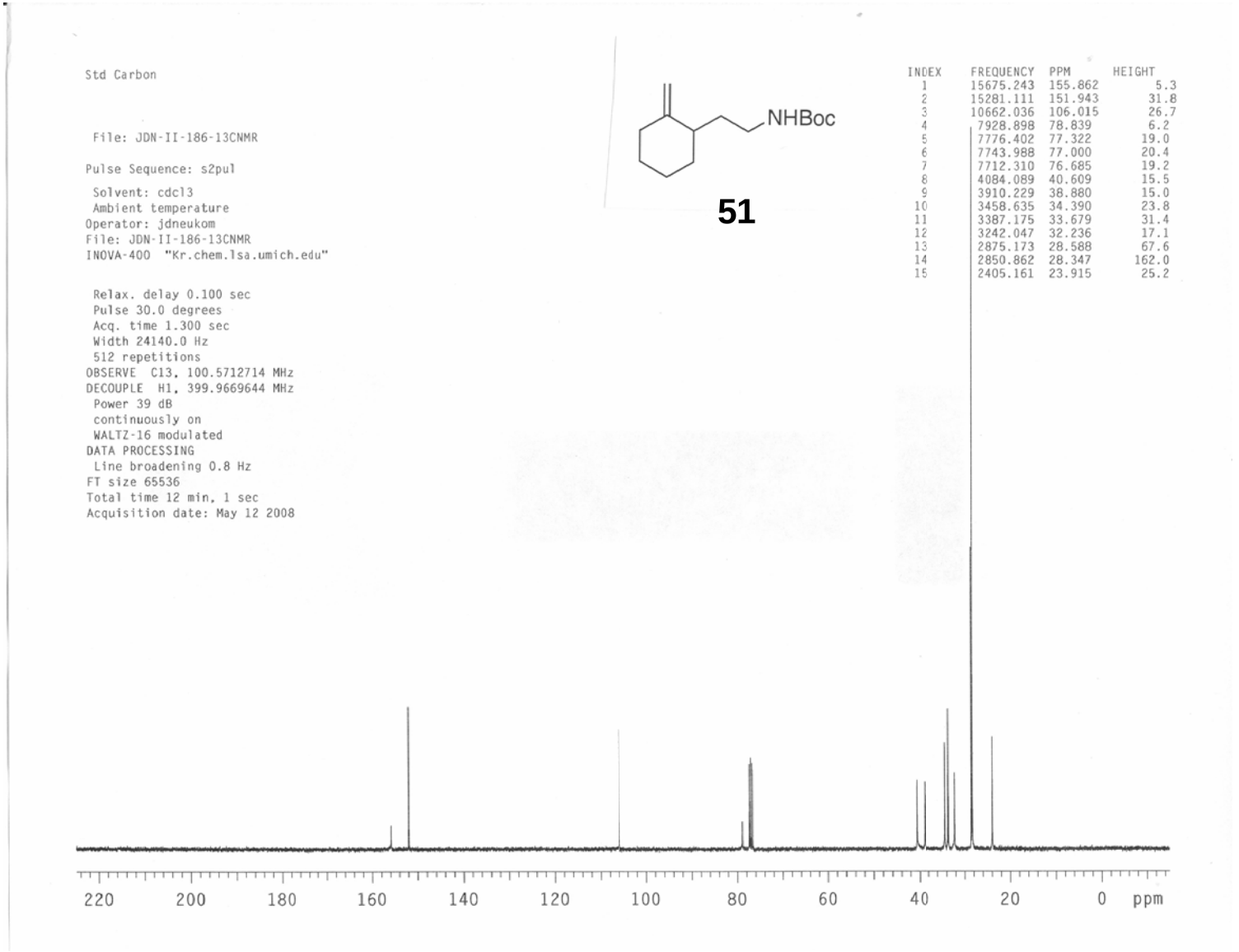

Std Carbon
File: mbb-10-110-C13-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: myrabert
File: mbb-10-110-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
208 repetitions
OBSERVE C13, 100.5712751 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 4 min, 53 sec
Acquisition date: Apr 9 2008
INDEX FREQUENCY PPM HEIGHT
1 15682.610 155.935 23.8
2 15670.087 155.811 5.7
3 10501.436 104.418 50.8
4 7919.322 78.743 5.5
5 7775.666 77.315 14.1
6 7743.988 77.000 13.8
7 7711.574 76.678 14.2
8 4159.968 41.363 17.5
9 3935.277 39.129 12.7
10 3471.159 34.514 16.6
11 3297.299 32.786 59.2
12 3265.621 32.471 61.6
13 2843.495 28.273 162.0
14 2417.685 24.040 46.3
15 -17.827 -0.177 4.6
NHBoc
49
160
140
120
100
80
60
40
20
ppm

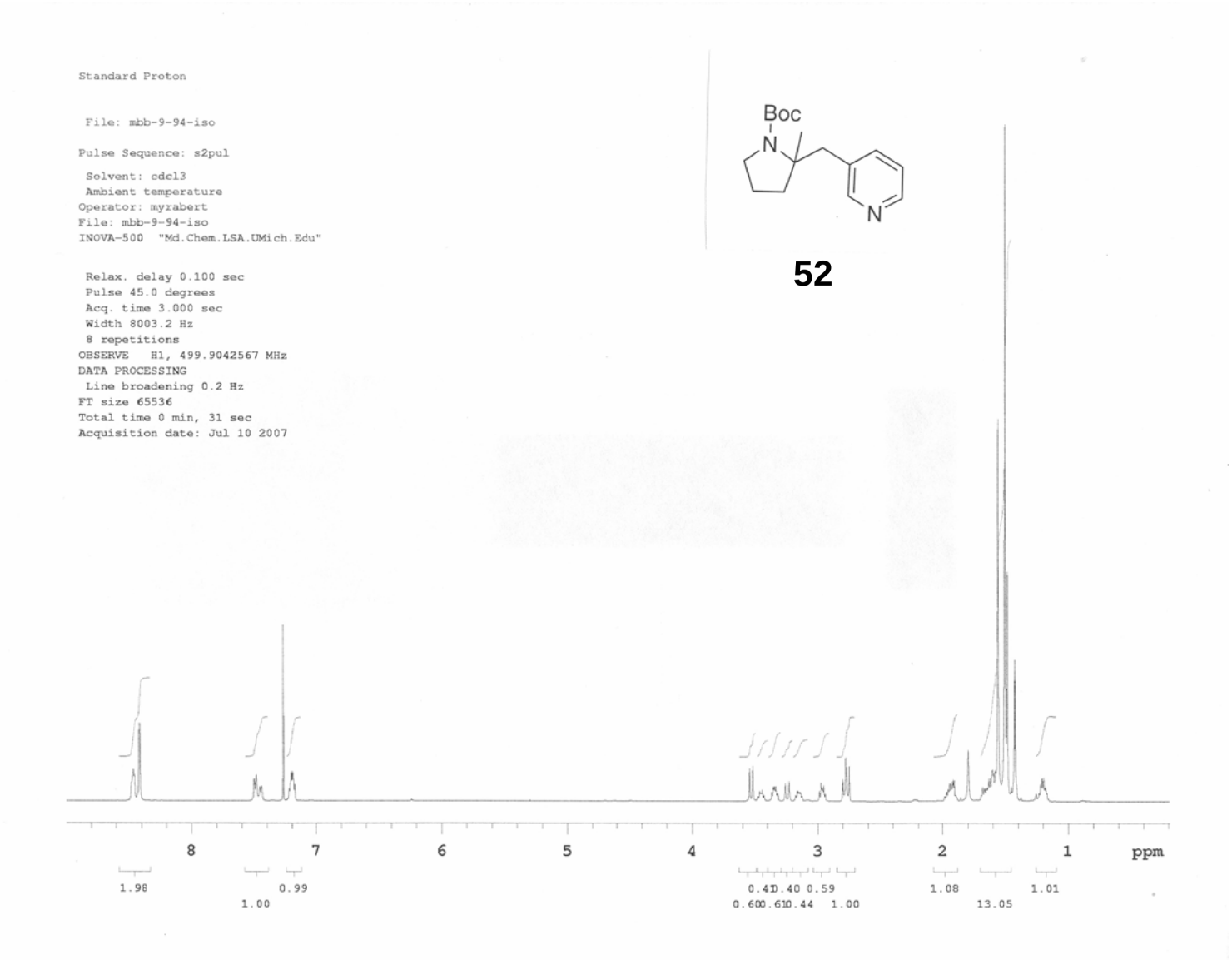

STANDARD PROTON PARAMETERS
File: mbb-10-138B-HNMR-400
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: myrabert
File: mbb-10-138B-HNMR-400
VNMRS-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.5389259 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec
Acquisition date: May 7 2008
H
N
Cbz
50
7
6
5
4
3
2
1
ppm
4.54
0.76 1.00
2.01 1.15
2.05
2.83
1.10 0.95 1.14
0.99 1.00 1.13


STANDARD CARBON PARAMETERS
File: mbb-10-138B-C13-400
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: myrabert
File: mbb-10-138B-C13-400
VNMRS-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
112 repetitions
OBSERVE C13, 100.4641544 MHz
DECOUPLE H1, 399.5409236 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 4 min, 16 sec
Acquisition date: May 7 2008
INDEX FREQUENCY PPM HEIGHT
1 15701.233 156.287 8.2
2 15662.308 155.899 14.9
3 13720.705 136.573 19.2
4 12895.485 128.359 183.2
5 12855.002 127.956 49.6
6 12851.110 127.917 74.7
7 10503.124 104.546 113.0
8 7768.999 77.331 22.1
9 7737.080 77.013 21.8
10 7705.161 76.696 21.9
11 6672.078 66.413 33.0
12 4150.486 41.313 36.7
13 3982.328 39.639 29.9
14 3455.277 34.393 34.3
15 3294.904 32.797 87.4
16 3262.985 32.479 67.6
17 2416.745 24.056 71.8
H
N
Cbz
160
140
120
100
80
60
40
20
ppm

**50**


Std proton
File: JDN-II-186-1HNMR-pub
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-186-1HNMR-pub
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649342 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: May 12 2008
NHBoc
51
7
6
5
4
3
2
1
-0
ppm
2.77
1.98
1.00 1.09 1.19 1.10
0.93 2.04 1.80 1.21


Std Carbon
File: JDN-II-186-13CNMR
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-186-13CNMR
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
512 repetitions
OBSERVE C13, 100.5712714 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 12 min, 1 sec
Acquisition date: May 12 2008
NHBoc
51
INDEX FREQUENCY PPM HEIGHT
1 15675.243 155.862 5.3
2 15281.111 151.943 31.8
3 10662.036 106.015 26.7
4 7928.898 78.839 6.2
5 7776.402 77.322 19.0
6 7743.988 77.000 20.4
7 7712.310 76.685 19.2
8 4084.089 40.609 15.5
9 3910.229 38.880 15.0
10 3458.635 34.390 23.8
11 3387.175 33.679 31.4
12 3242.047 32.236 17.1
13 2875.173 28.588 67.6
14 2850.862 28.347 162.0
15 2405.161 23.915 25.2
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Standard Proton
File: mbb-9-94-iso
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-94-iso
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042567 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jul 10 2007
Boc
N
N
52
8
7
6
5
4
3
2
1
ppm
1.98
1.00
0.99
0.41
0.60
0.40
0.61
0.59
0.44
1.00
1.08
13.05
1.01

Standard Carbon

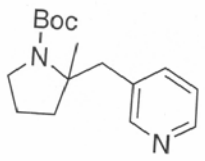
File: mbb-9-94-C13-500

Pulse Sequence: s2pul

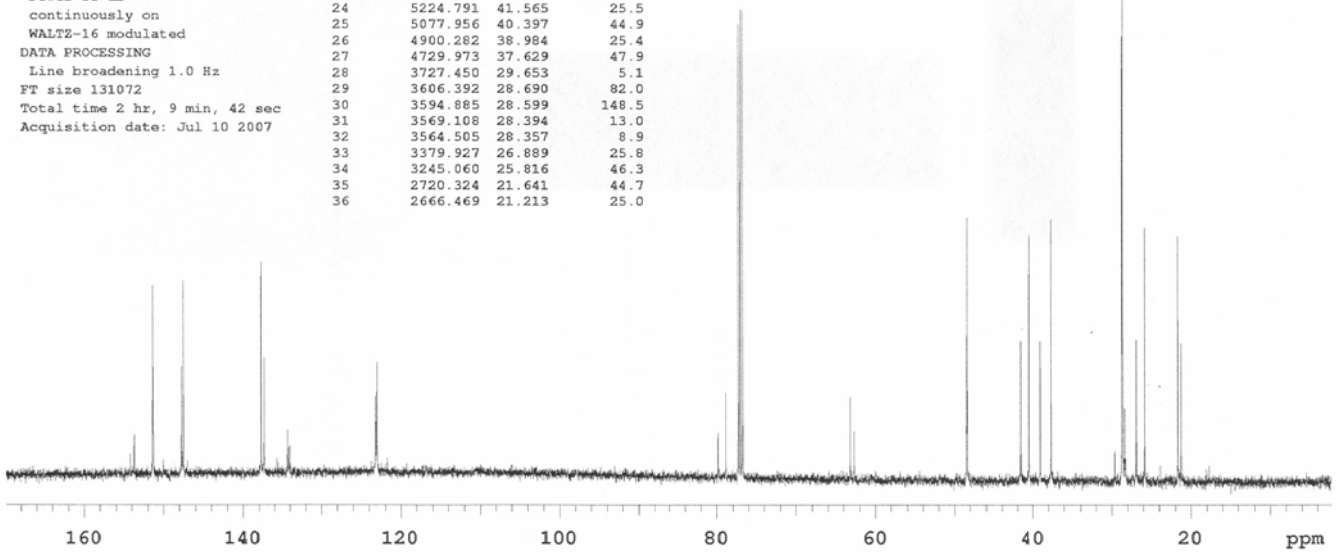
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-94-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

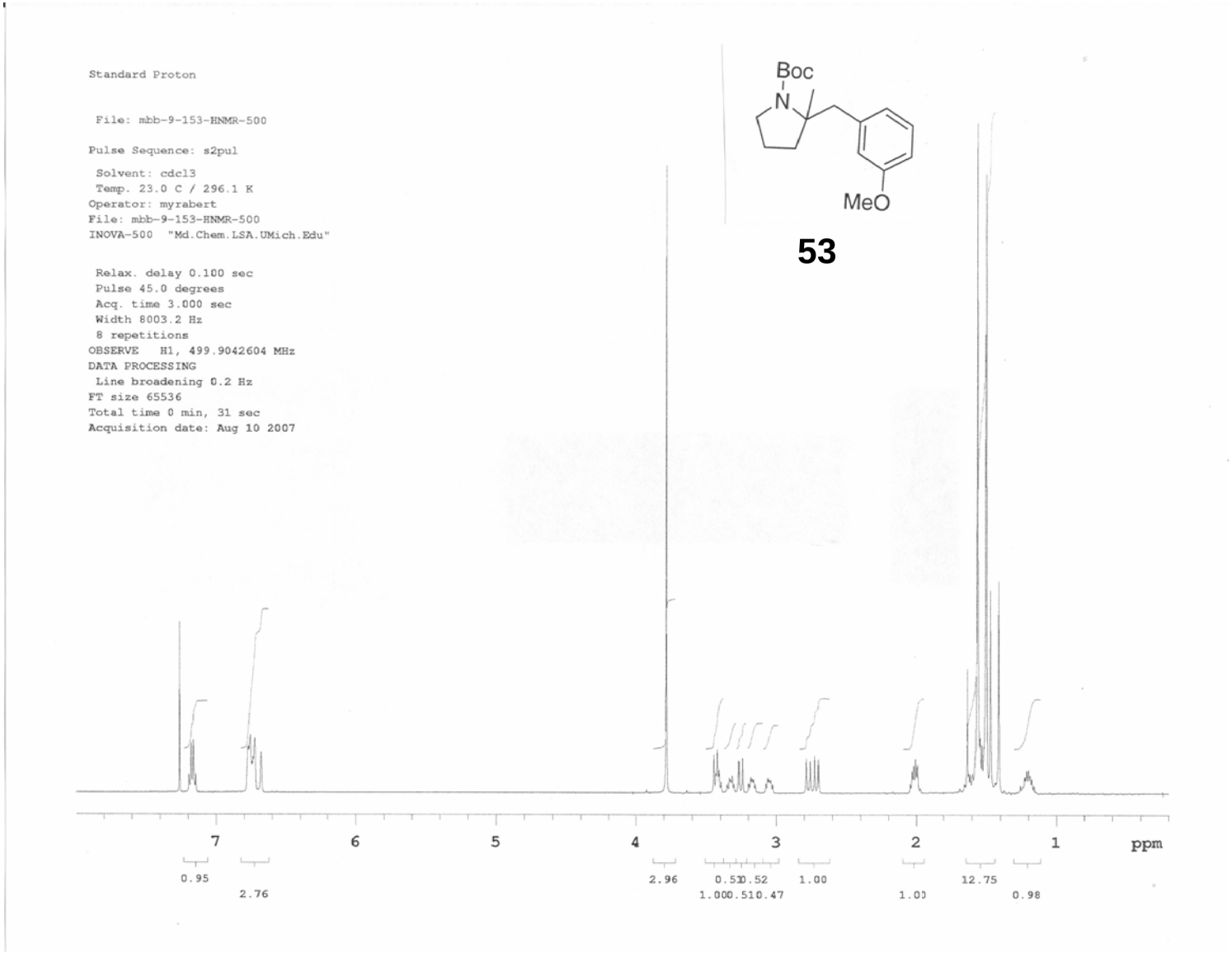
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
5520 repetitions
OBSERVE C13, 125.7010275 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 9 min, 42 sec
Acquisition date: Jul 10 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 19380.712 | 154.181 | 4.6 |
| 2 | 19320.413 | 153.701 | 8.2 |
| 3 | 19026.745 | 151.365 | 35.7 |
| 4 | 19016.158 | 151.281 | 20.6 |
| 5 | 18575.195 | 147.773 | 20.7 |
| 6 | 18543.895 | 147.524 | 36.5 |
| 7 | 17312.605 | 137.728 | 40.0 |
| 8 | 17260.132 | 137.311 | 22.3 |
| 9 | 17057.141 | 135.696 | 3.7 |
| 10 | 16874.864 | 134.246 | 9.1 |
| 11 | 16847.707 | 134.030 | 6.1 |
| 12 | 15485.693 | 123.195 | 15.3 |
| 13 | 15460.377 | 122.993 | 21.7 |
| 14 | 15298.813 | 121.708 | 3.8 |
| 15 | 10030.273 | 79.795 | 8.5 |
| 16 | 9912.438 | 78.857 | 15.9 |
| 17 | 9711.749 | 77.261 | 84.2 |
| 18 | 9679.989 | 77.008 | 87.0 |
| 19 | 9647.768 | 76.752 | 86.8 |
| 20 | 7940.073 | 63.166 | 15.2 |
| 21 | 7875.631 | 62.654 | 8.8 |
| 22 | 6091.066 | 48.457 | 26.4 |
| 23 | 6080.480 | 48.373 | 48.2 |
| 24 | 5224.791 | 41.565 | 25.5 |
| 25 | 5077.956 | 40.397 | 44.9 |
| 26 | 4900.282 | 38.984 | 25.4 |
| 27 | 4729.973 | 37.629 | 47.9 |
| 28 | 3727.450 | 29.653 | 5.1 |
| 29 | 3606.392 | 28.690 | 82.0 |
| 30 | 3594.885 | 28.599 | 148.5 |
| 31 | 3569.108 | 28.394 | 13.0 |
| 32 | 3564.505 | 28.357 | 8.9 |
| 33 | 3379.927 | 26.889 | 25.8 |
| 34 | 3245.060 | 25.816 | 46.3 |
| 35 | 2720.324 | 21.641 | 44.7 |
| 36 | 2666.469 | 21.213 | 25.0 |




**52**




Standard Proton
File: mbb-9-153-HNMR-500
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-9-153-HNMR-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042604 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Aug 10 2007
Boc
N
MeO
53
7
6
5
4
3
2
1
ppm
0.95
2.76
2.96
1.00
0.98
12.75

Standard Carbon

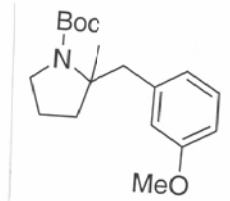
File: mbb-9-153-C13-500

Pulse Sequence: s2pul

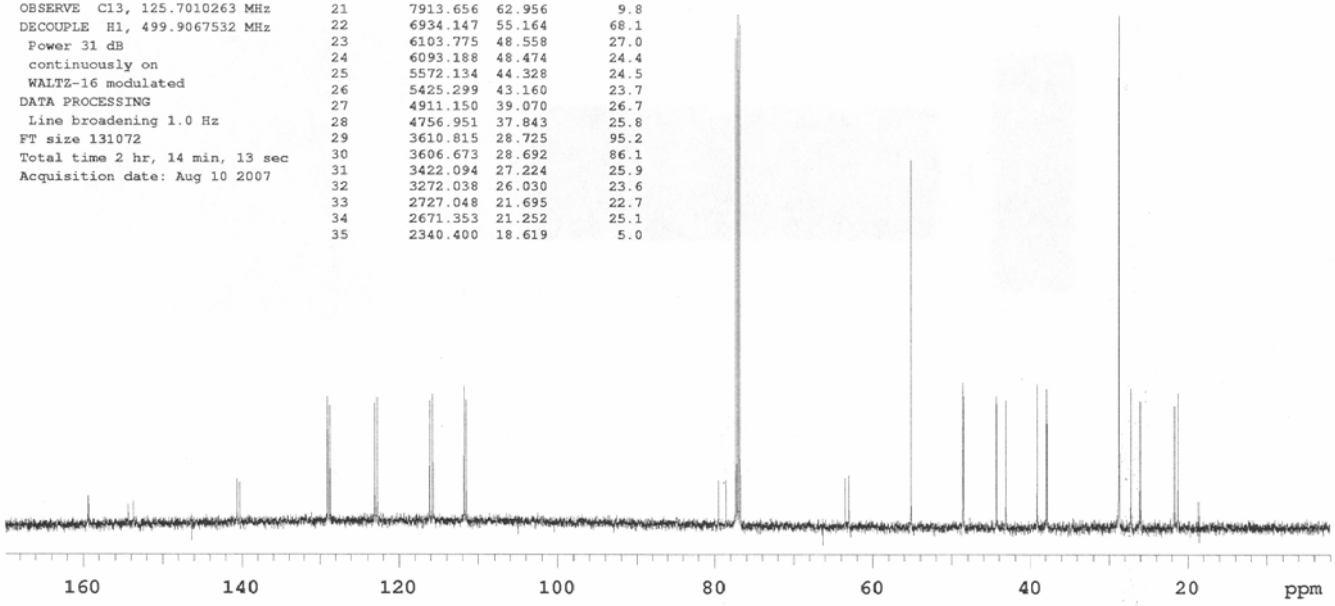
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-9-153-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

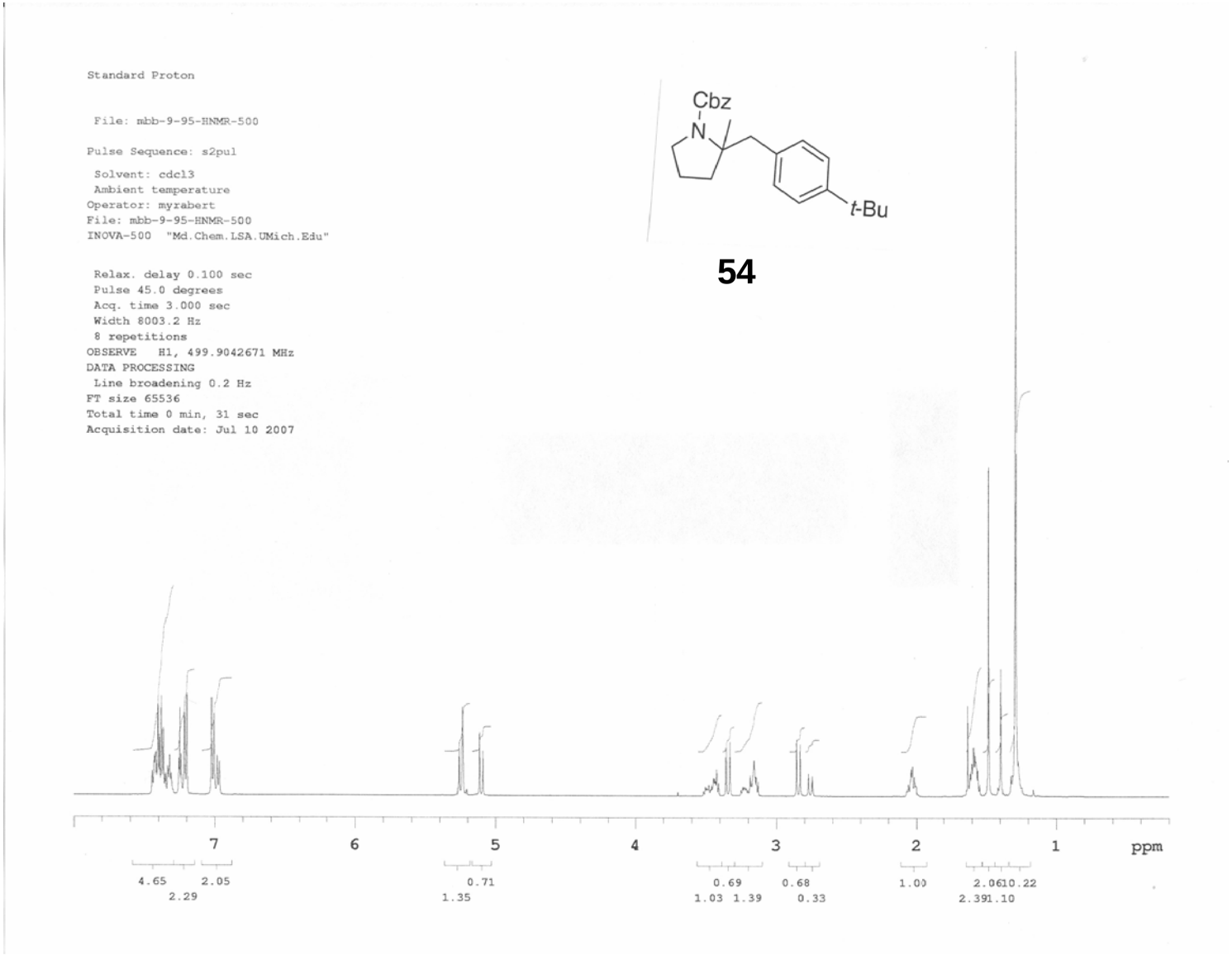
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
5712 repetitions
OBSERVE C13, 125.7010263 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 14 min, 13 sec
Acquisition date: Aug 10 2007

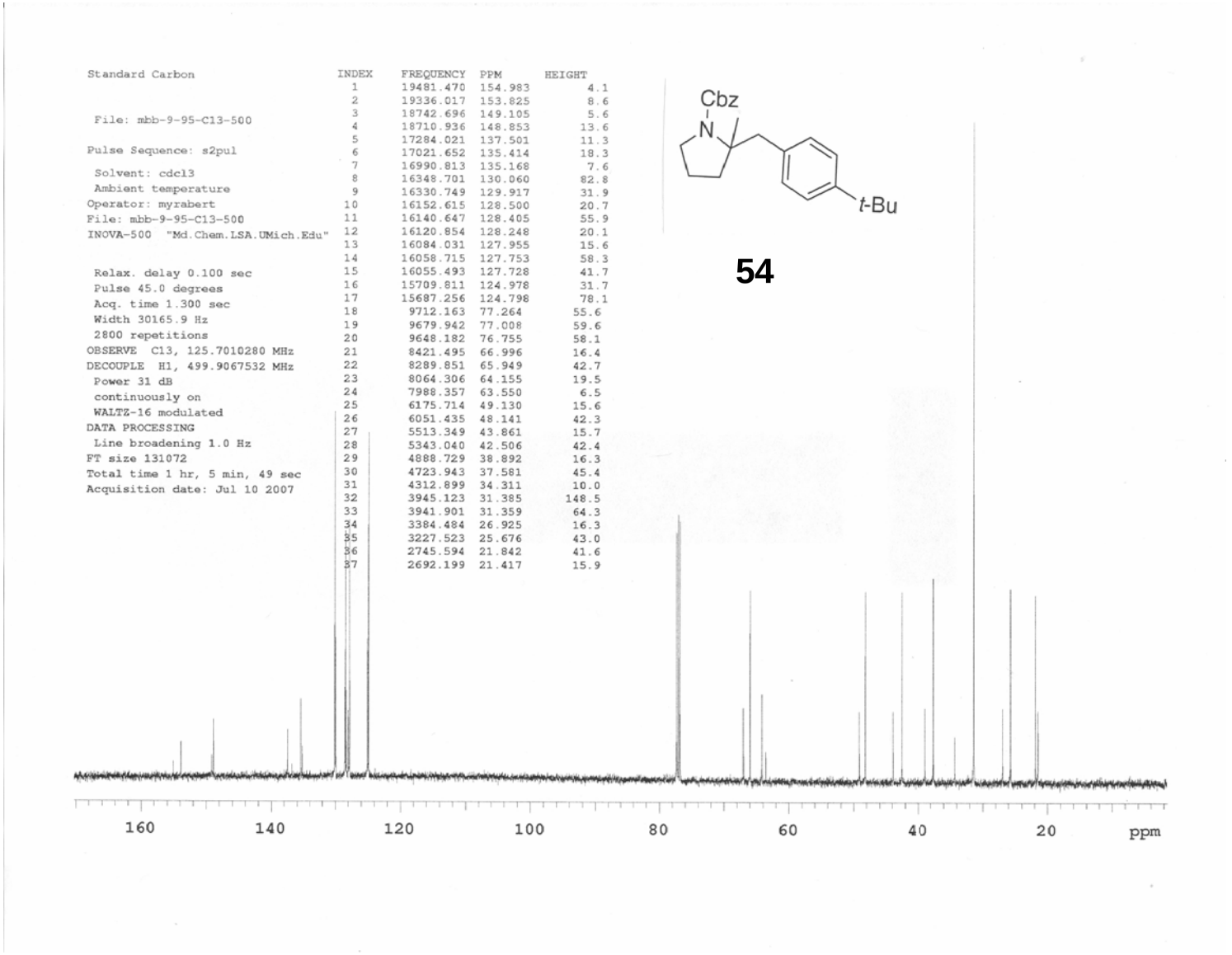
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 20033.691 | 159.376 | 5.9 |
| 2 | 20021.724 | 159.281 | 6.0 |
| 3 | 19399.404 | 154.330 | 4.4 |
| 4 | 19316.091 | 153.667 | 5.0 |
| 5 | 17668.694 | 140.561 | 9.2 |
| 6 | 17632.791 | 140.276 | 8.6 |
| 7 | 16214.161 | 128.990 | 24.4 |
| 8 | 16179.178 | 128.712 | 22.8 |
| 9 | 15465.260 | 123.032 | 23.2 |
| 10 | 15428.897 | 122.743 | 24.2 |
| 11 | 14595.302 | 116.111 | 23.7 |
| 12 | 14547.432 | 115.730 | 24.8 |
| 13 | 14051.233 | 111.783 | 26.4 |
| 14 | 14018.552 | 111.523 | 23.8 |
| 15 | 9991.429 | 79.486 | 8.9 |
| 16 | 9876.815 | 78.574 | 9.0 |
| 17 | 9711.569 | 77.259 | 90.8 |
| 18 | 9679.809 | 77.007 | 95.2 |
| 19 | 9648.049 | 76.754 | 93.4 |
| 20 | 7979.938 | 63.483 | 9.3 |
| 21 | 7913.656 | 62.956 | 9.8 |
| 22 | 6934.147 | 55.164 | 68.1 |
| 23 | 6103.775 | 48.558 | 27.0 |
| 24 | 6093.188 | 48.474 | 24.4 |
| 25 | 5572.134 | 44.328 | 24.5 |
| 26 | 5425.299 | 43.160 | 23.7 |
| 27 | 4911.150 | 39.070 | 26.7 |
| 28 | 4756.951 | 37.843 | 25.8 |
| 29 | 3610.815 | 28.725 | 95.2 |
| 30 | 3606.673 | 28.692 | 86.1 |
| 31 | 3422.094 | 27.224 | 25.9 |
| 32 | 3272.038 | 26.030 | 23.6 |
| 33 | 2727.048 | 21.695 | 22.7 |
| 34 | 2671.353 | 21.252 | 25.1 |
| 35 | 2340.400 | 18.619 | 5.0 |

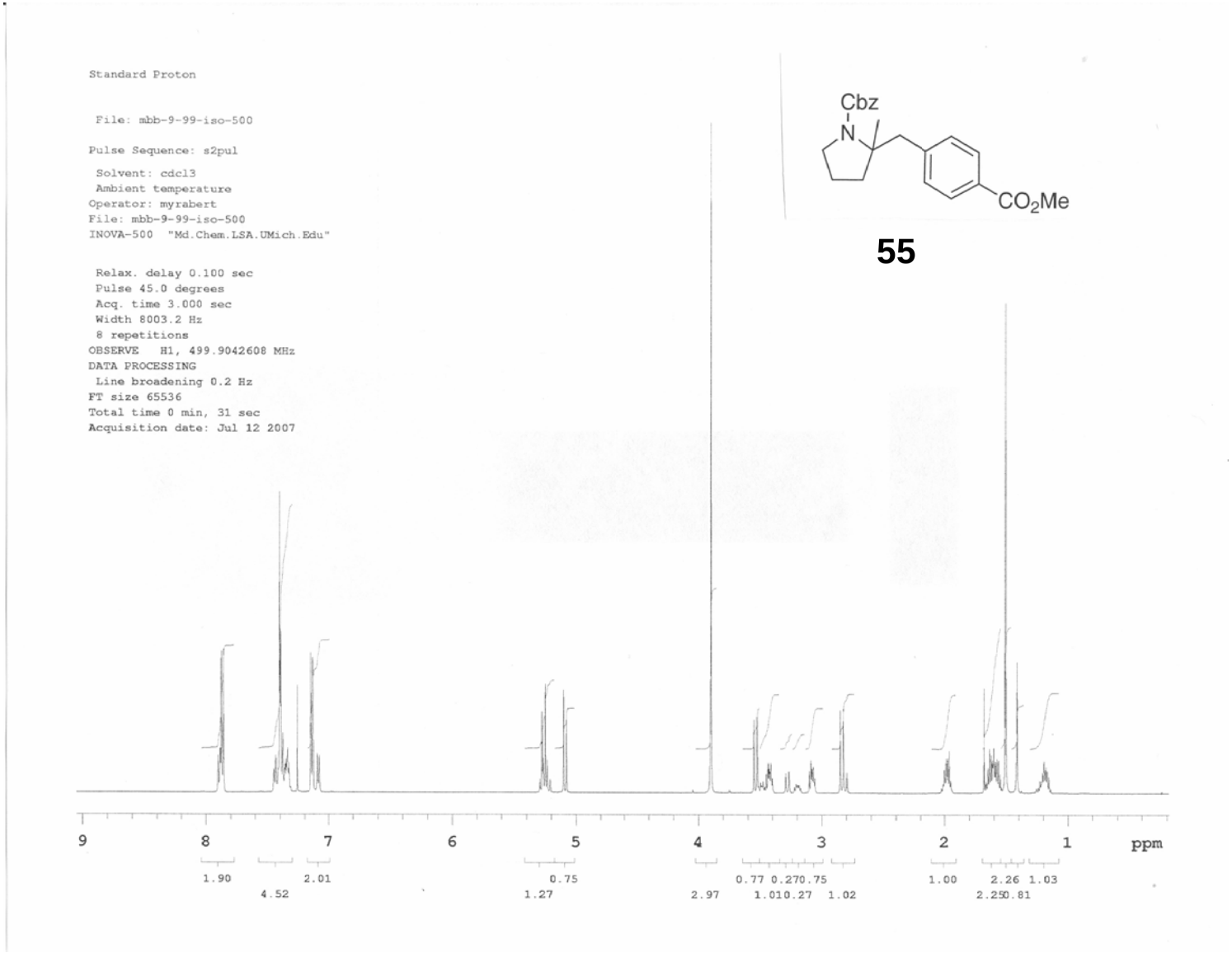



**53**




Standard Proton
File: mbb-9-95-HNMR-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-95-HNMR-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042671 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jul 10 2007
Cbz
N
t-Bu
54
7
6
5
4
3
2
1
ppm
4.65
2.05
2.29
0.71
1.35
0.69
0.68
1.03
1.39
0.33
1.00
2.06
10.22
2.39
1.10


Standard Carbon
File: mbb-9-95-C13-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-95-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
2800 repetitions
OBSERVE C13, 125.7010280 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 5 min, 49 sec
Acquisition date: Jul 10 2007
INDEX FREQUENCY PPM HEIGHT
1 19481.470 154.983 4.1
2 19336.017 153.825 8.6
3 18742.696 149.105 5.6
4 18710.936 148.853 13.6
5 17284.021 137.501 11.3
6 17021.652 135.414 18.3
7 16990.813 135.168 7.6
8 16348.701 130.060 82.8
9 16330.749 129.917 31.9
10 16152.615 128.500 20.7
11 16140.647 128.405 55.9
12 16120.854 128.248 20.1
13 16084.031 127.955 15.6
14 16058.715 127.753 58.3
15 16055.493 127.728 41.7
16 15709.811 124.978 31.7
17 15687.256 124.798 78.1
18 9712.163 77.264 55.6
19 9679.942 77.008 59.6
20 9648.182 76.755 58.1
21 8421.495 66.996 16.4
22 8289.851 65.949 42.7
23 8064.306 64.155 19.5
24 7988.357 63.550 6.5
25 6175.714 49.130 15.6
26 6051.435 48.141 42.3
27 5513.349 43.861 15.7
28 5343.040 42.506 42.4
29 4888.729 38.892 16.3
30 4723.943 37.581 45.4
31 4312.899 34.311 10.0
32 3945.123 31.385 148.5
33 3941.901 31.359 64.3
34 3384.484 26.925 16.3
35 3227.523 25.676 43.0
36 2745.594 21.842 41.6
37 2692.199 21.417 15.9
Cbz
N
t-Bu
54
160
140
120
100
80
60
40
20
ppm


Standard Proton
File: mbb-9-99-iso-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-99-iso-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042608 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jul 12 2007
Cbz
N
CO2Me
55
9
8
7
6
5
4
3
2
1
ppm
1.90
4.52
2.01
0.75
1.27
2.97
0.77 0.27 0.75
1.01 0.27
1.02
1.00
2.26 1.03
2.25 0.81

Standard Carbon

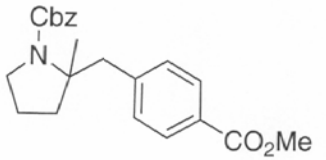
File: mbb-9-99-C13-500

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-99-C13-500
INOVA-500 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
912 repetitions
OBSERVE C13, 125.7010292 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 21 min, 28 sec
Acquisition date: Jul 13 2007

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 21010.769 | 167.149 | 9.7 |
| 2 | 19340.817 | 153.864 | 10.2 |
| 3 | 18115.971 | 144.120 | 19.6 |
| 4 | 18074.545 | 143.790 | 4.3 |
| 5 | 17258.441 | 137.298 | 10.7 |
| 6 | 16390.785 | 130.395 | 90.2 |
| 7 | 16370.532 | 130.234 | 24.3 |
| 8 | 16259.601 | 129.351 | 25.5 |
| 9 | 16245.331 | 129.238 | 86.0 |
| 10 | 16158.796 | 128.549 | 17.2 |
| 11 | 16146.368 | 128.451 | 63.7 |
| 12 | 16139.924 | 128.399 | 16.5 |
| 13 | 16124.274 | 128.275 | 5.0 |
| 14 | 16104.021 | 128.114 | 11.5 |
| 15 | 16099.418 | 128.077 | 15.2 |
| 16 | 16071.800 | 127.857 | 43.2 |
| 17 | 16062.134 | 127.780 | 56.8 |
| 18 | 9711.900 | 77.262 | 52.1 |
| 19 | 9679.679 | 77.006 | 54.7 |
| 20 | 9647.919 | 76.753 | 55.7 |
| 21 | 8434.581 | 67.100 | 10.8 |
| 22 | 8307.539 | 66.090 | 46.3 |
| 23 | 8047.012 | 64.017 | 21.8 |
| 24 | 7967.381 | 63.384 | 4.7 |
| 25 | 6533.561 | 51.977 | 37.3 |
| 26 | 6169.928 | 49.084 | 11.8 |
| 27 | 6046.569 | 48.103 | 44.7 |
| 28 | 5584.892 | 44.430 | 11.5 |
| 29 | 5408.599 | 43.027 | 44.2 |
| 30 | 4898.592 | 38.970 | 12.3 |
| 31 | 4722.299 | 37.568 | 46.5 |
| 32 | 3413.219 | 27.153 | 12.5 |
| 33 | 3267.306 | 25.993 | 46.2 |
| 34 | 2741.188 | 21.807 | 45.9 |
| 35 | 2688.714 | 21.390 | 11.3 |
| 36 | 2379.856 | 18.933 | -4.9 |

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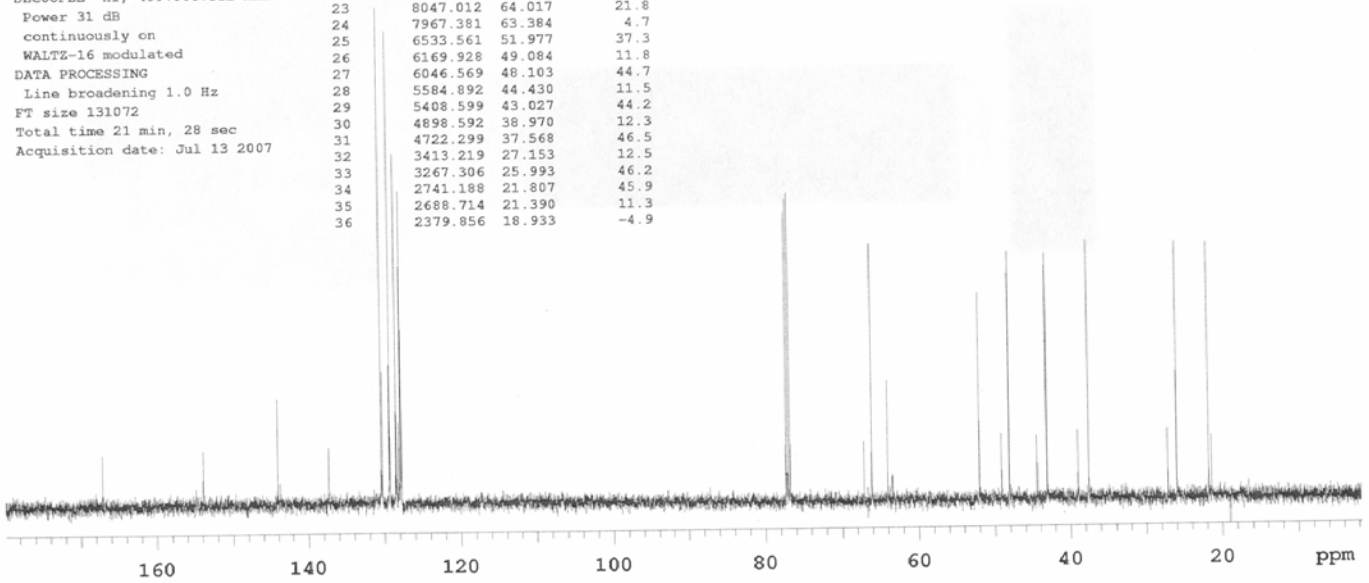
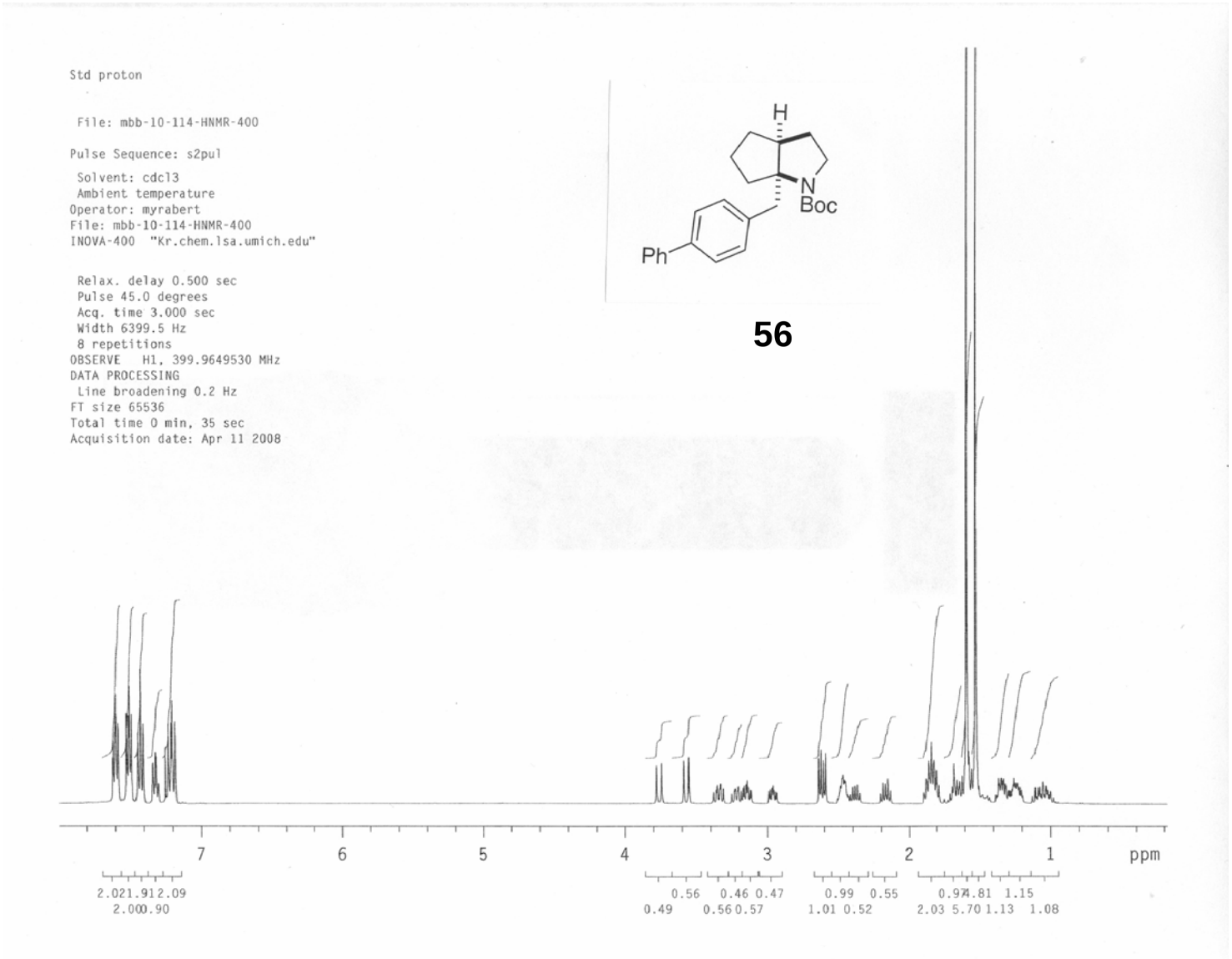



Std proton
File: mbb-10-114-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-114-HNMR-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649530 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Apr 11 2008
H
N
Boc
Ph
56
7
6
5
4
3
2
1
ppm
2.02 1.91 2.09
2.00 0.90
0.56 0.46 0.47
0.49 0.56 0.57
0.99 0.55
1.01 0.52
0.97 4.81 1.15
2.03 5.70 1.13 1.08

Std Carbon

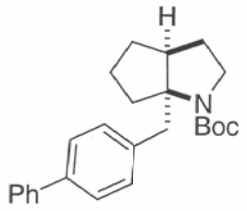
File: mbb-10-114-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-114-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
464 repetitions
OBSERVE C13, 100.5712693 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 10 min, 53 sec
Acquisition date: Apr 11 2008

| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|---|---|---|---|
| 1 | 15502.768 | 154.147 | 6.740 | | 2555.358 | 25.408 | 14.3 |
| 2 | 15457.093 | 153.693 | 5.2 | | | | |
| 3 | 14174.507 | 140.940 | 5.0 | | | | |
| 4 | 14161.247 | 140.808 | 6.6 | | | | |
| 5 | 13968.969 | 138.896 | 6.6 | | | | |
| 6 | 13945.395 | 138.662 | 8.2 | | | | |
| 7 | 13917.401 | 138.383 | 6.7 | | | | |
| 8 | 13870.989 | 137.922 | 10.4 | | | | |
| 9 | 13134.294 | 130.597 | 29.3 | | | | |
| 10 | 13122.507 | 130.480 | 30.4 | | | | |
| 11 | 12944.964 | 128.714 | 34.1 | | | | |
| 12 | 12942.017 | 128.685 | 35.4 | | | | |
| 13 | 12779.944 | 127.074 | 18.7 | | | | |
| 14 | 12773.314 | 127.008 | 14.9 | | | | |
| 15 | 12760.790 | 126.883 | 72.2 | | | | |
| 16 | 12743.109 | 126.707 | 30.9 | | | | |
| 17 | 12720.272 | 126.480 | 25.0 | | | | |
| 18 | 7990.692 | 79.453 | 9.7 | | | | |
| 19 | 7911.129 | 78.662 | 12.0 | | | | |
| 20 | 7777.050 | 77.329 | 43.9 | | | | |
| 21 | 7744.636 | 77.006 | 44.8 | | | | |
| 22 | 7712.958 | 76.691 | 45.0 | | | | |
| 23 | 7511.104 | 74.684 | 8.1 | | | | |
| 24 | 7448.485 | 74.062 | 13.1 | | | | |
| 25 | 4927.515 | 48.995 | 14.9 | | | | |
| 26 | 4800.804 | 47.735 | 16.0 | | | | |
| 27 | 4797.857 | 47.706 | 15.2 | | | | |
| 28 | 4783.860 | 47.567 | 14.9 | | | | |
| 29 | 4326.372 | 43.018 | 16.5 | | | | |
| 30 | 4201.134 | 41.773 | 13.7 | | | | |
| 31 | 4094.314 | 40.711 | 17.2 | | | | |
| 32 | 3944.028 | 39.216 | 13.9 | | | | |
| 33 | 3265.532 | 32.470 | 16.2 | | | | |
| 34 | 3244.905 | 32.265 | 19.2 | | | | |
| 35 | 2895.711 | 28.793 | 59.3 | | | | |
| 36 | 2884.661 | 28.683 | 47.1 | | | | |
| 37 | 2841.196 | 28.251 | 13.8 | | | | |
| 38 | 2747.636 | 27.320 | 17.3 | | | | |
| 39 | 2574.512 | 25.599 | 20.0 | | | | |

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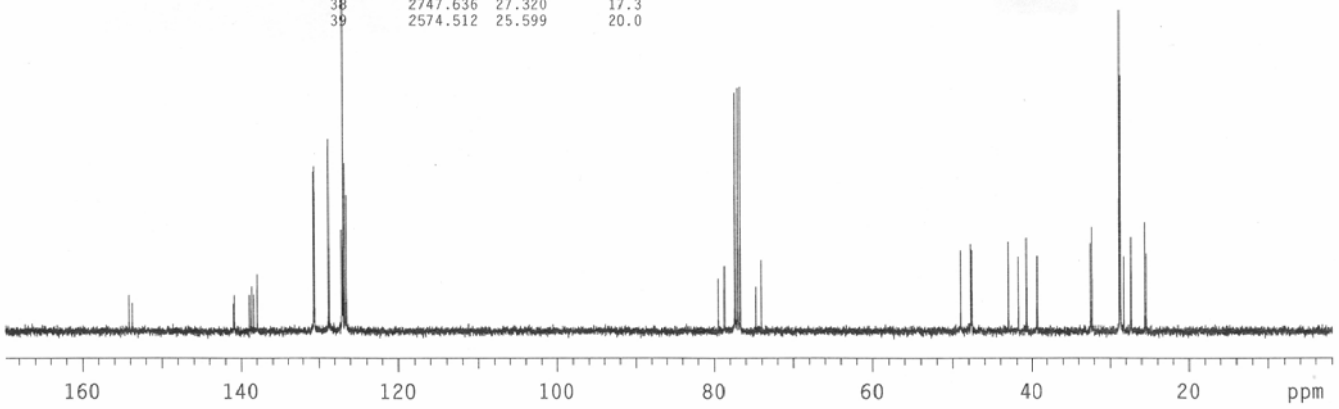
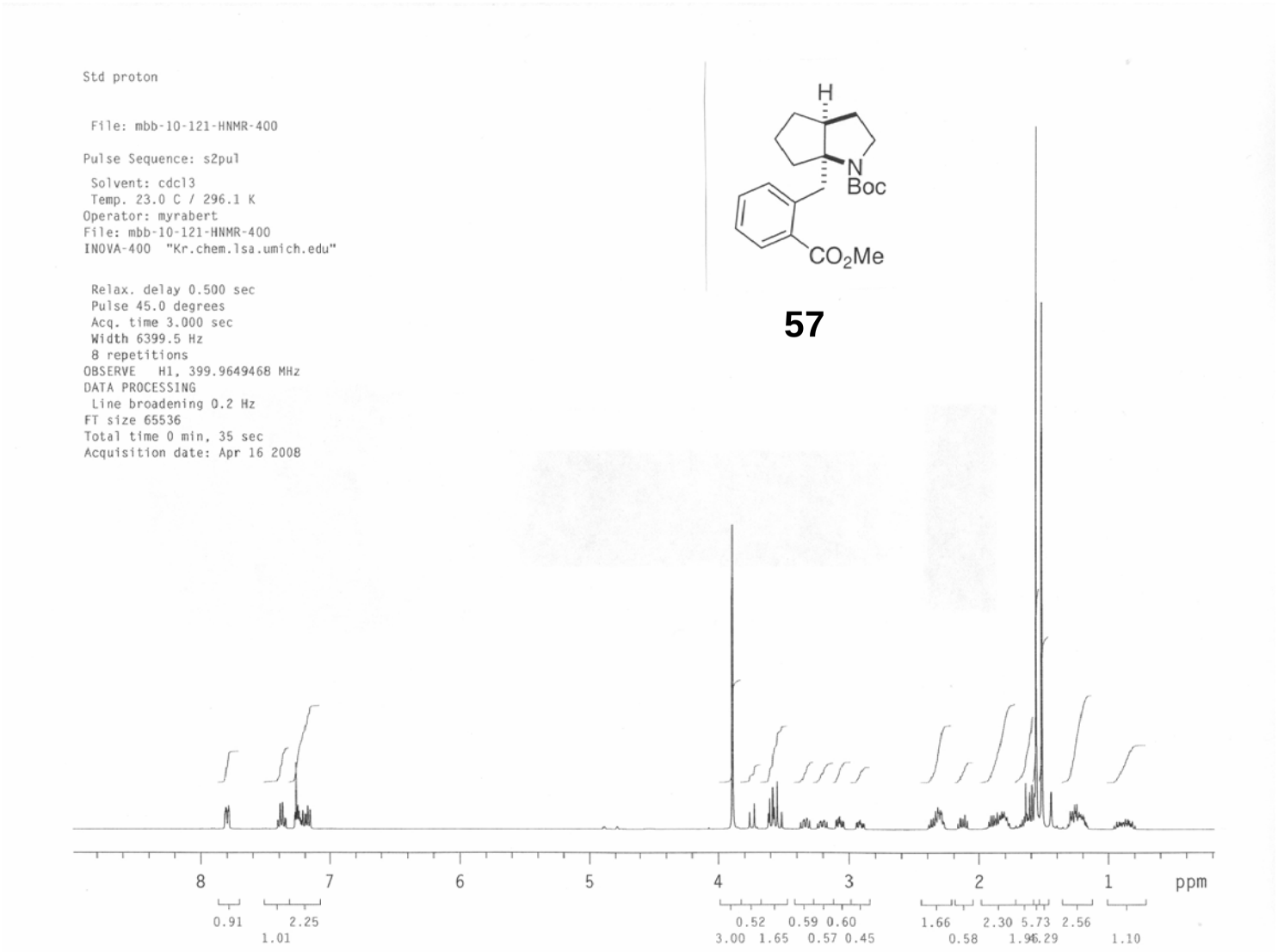



Std proton
File: mbb-10-121-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-121-HNMR-400
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649468 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Apr 16 2008
H
N
Boc
CO2Me
57
8
7
6
5
4
3
2
1
ppm
0.91
2.25
1.01
0.52
0.59
0.60
3.00
1.65
0.57
0.45
1.66
2.30
5.73
2.56
0.58
1.96
6.29
1.10

Std Carbon

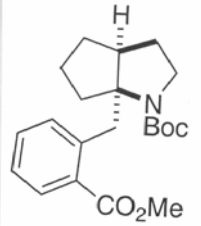
File: mbb-10-121-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 23.0 C / 296.1 K
Operator: myrabert
File: mbb-10-121-C13-400
INOVA-400 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
2624 repetitions
OBSERVE C13, 100.5712663 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 1 min, 38 sec
Acquisition date: Apr 16 2008

| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|---|---|---|---|
| 1 | 16989.469 | 168.930 | 6.040 | | 2570.144 | 25.555 | 30.0 |
| 2 | 16973.999 | 168.776 | 7.541 | | 2550.990 | 25.365 | 23.2 |
| 3 | 15510.186 | 154.221 | 7.4 | | | | |
| 4 | 15457.881 | 153.701 | 5.3 | | | | |
| 5 | 14121.517 | 140.413 | 8.0 | | | | |
| 6 | 14077.315 | 139.974 | 13.0 | | | | |
| 7 | 13338.410 | 132.626 | 18.6 | | | | |
| 8 | 13303.049 | 132.275 | 26.1 | | | | |
| 9 | 13205.805 | 131.308 | 21.5 | | | | |
| 10 | 13194.755 | 131.198 | 9.4 | | | | |
| 11 | 13183.705 | 131.088 | 19.6 | | | | |
| 12 | 13084.251 | 130.099 | 31.9 | | | | |
| 13 | 12676.122 | 126.041 | 26.1 | | | | |
| 14 | 12658.441 | 125.865 | 21.0 | | | | |
| 15 | 7997.374 | 79.519 | 19.1 | | | | |
| 16 | 7915.601 | 78.706 | 10.2 | | | | |
| 17 | 7776.365 | 77.322 | 108.7 | | | | |
| 18 | 7744.688 | 77.007 | 118.4 | | | | |
| 19 | 7713.010 | 76.692 | 113.3 | | | | |
| 20 | 7571.564 | 75.286 | 12.3 | | | | |
| 21 | 7501.578 | 74.590 | 17.2 | | | | |
| 22 | 5237.715 | 52.080 | 19.5 | | | | |
| 23 | 5233.295 | 52.036 | 12.8 | | | | |
| 24 | 4842.847 | 48.153 | 27.6 | | | | |
| 25 | 4786.122 | 47.589 | 20.4 | | | | |
| 26 | 4777.281 | 47.501 | 23.9 | | | | |
| 27 | 4708.769 | 46.820 | 20.7 | | | | |
| 28 | 4142.987 | 41.195 | 24.5 | | | | |
| 29 | 3965.444 | 39.429 | 19.1 | | | | |
| 30 | 3855.676 | 38.338 | 25.3 | | | | |
| 31 | 3748.119 | 37.268 | 19.5 | | | | |
| 32 | 3280.318 | 32.617 | 4.5 | | | | |
| 33 | 3240.536 | 32.221 | 21.9 | | | | |
| 34 | 3227.276 | 32.089 | 27.0 | | | | |
| 35 | 2891.343 | 28.749 | 92.6 | | | | |
| 36 | 2885.449 | 28.691 | 66.2 | | | | |
| 37 | 2858.192 | 28.420 | 10.4 | | | | |
| 38 | 2840.511 | 28.244 | 20.1 | | | | |
| 39 | 2757.264 | 27.416 | 25.5 | | | | |

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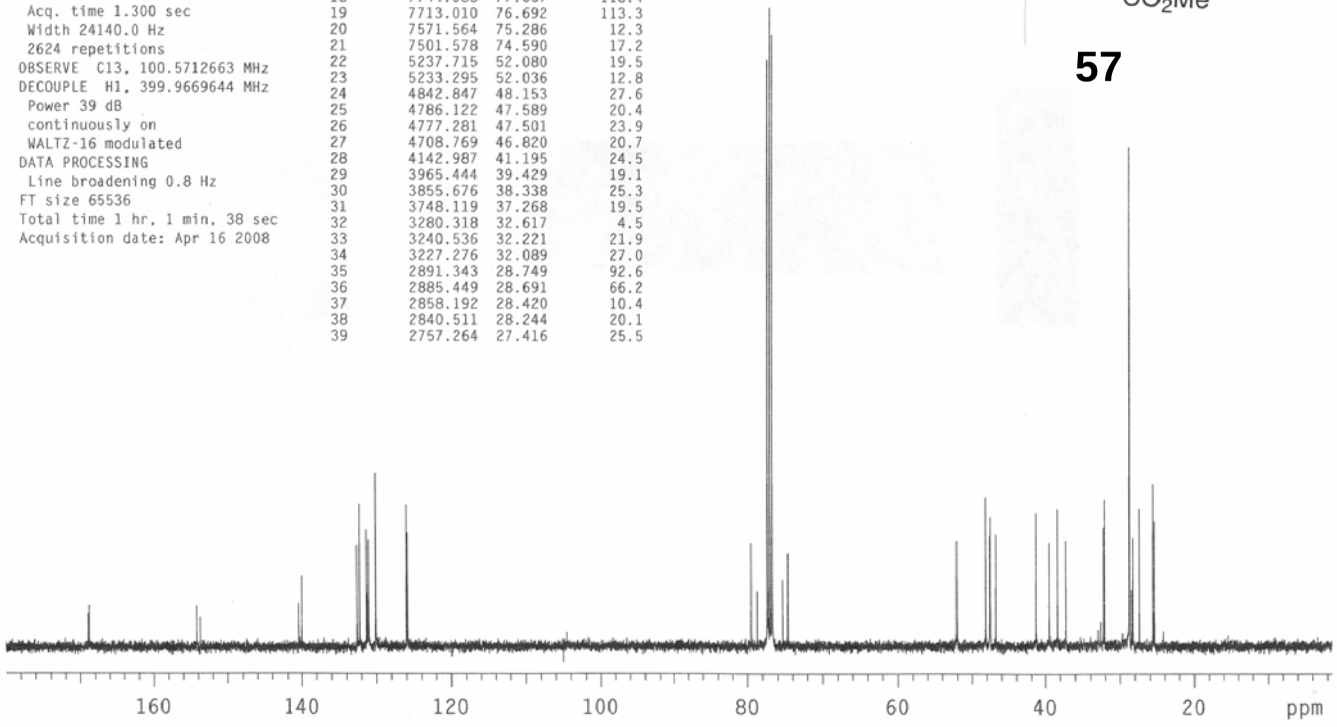
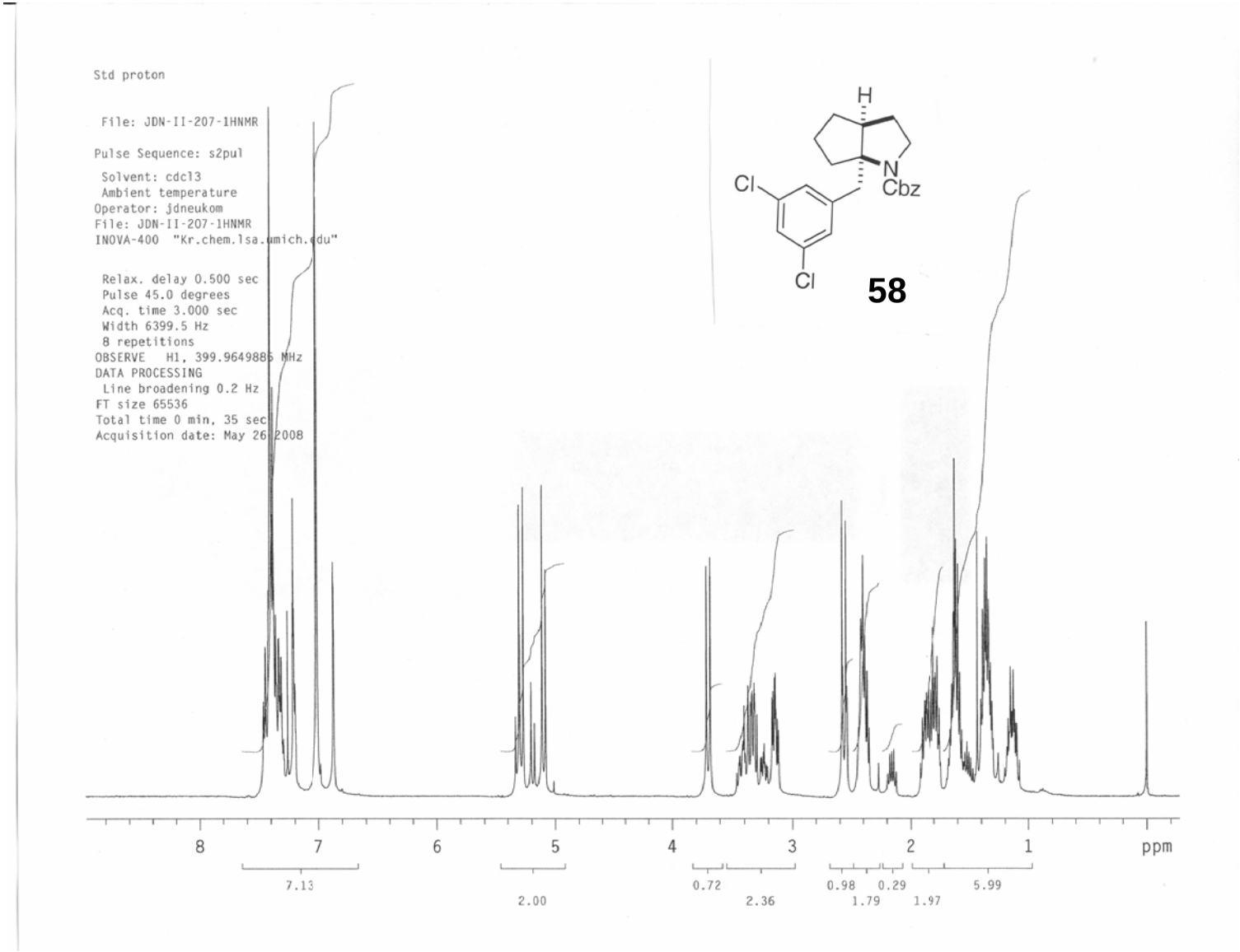



Std proton
File: JDN-II-207-1HNMR
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-207-1HNMR
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649886 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: May 26 2008
H
N
Cbz
Cl
Cl
58
8
7
6
5
4
3
2
1
ppm
7.13
2.00
0.72
2.36
0.98
1.79
0.29
1.97
5.99

Std Carbon

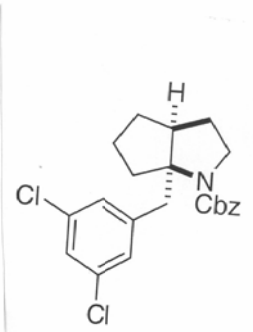
File: JDN-II-207-13CNMR

Pulse Sequence: s2pul

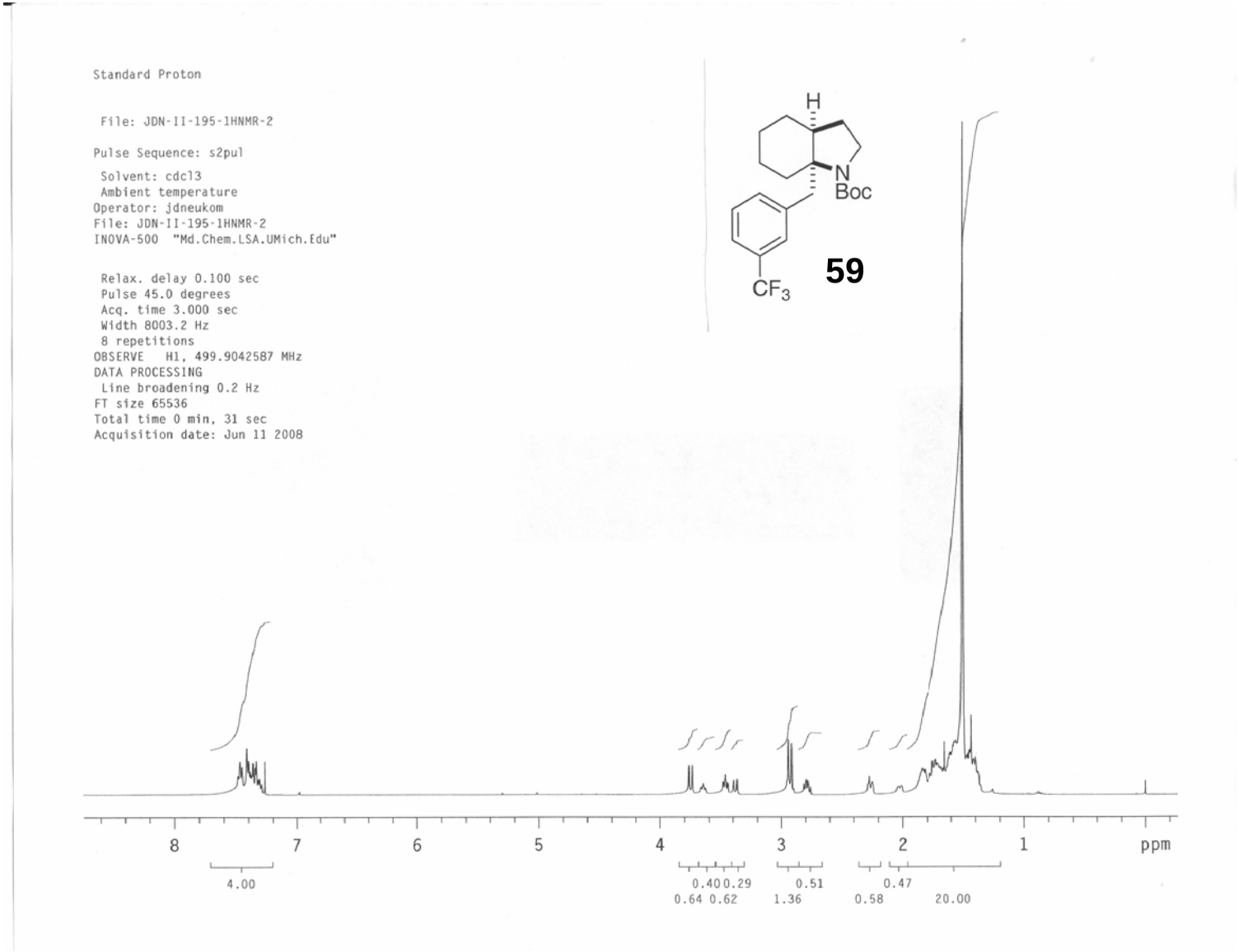
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-207-13CNMR
INOVA-400 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
256 repetitions
OBSERVE C13, 100.5712788 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 6 min, 0 sec
Acquisition date: May 26 2008

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15484.440 | 153.965 | 17.3 |
| 2 | 14310.885 | 142.296 | 33.3 |
| 3 | 14264.473 | 141.834 | 13.4 |
| 4 | 13789.305 | 137.110 | 21.0 |
| 5 | 13515.255 | 134.385 | 43.3 |
| 6 | 12936.949 | 128.635 | 39.5 |
| 7 | 12925.162 | 128.517 | 110.5 |
| 8 | 12920.742 | 128.473 | 141.9 |
| 9 | 12912.639 | 128.393 | 56.5 |
| 10 | 12914.112 | 128.408 | 64.6 |
| 11 | 12892.011 | 128.188 | 26.5 |
| 12 | 12856.650 | 127.836 | 56.7 |
| 13 | 12840.442 | 127.675 | 85.3 |
| 14 | 12729.202 | 126.569 | 20.0 |
| 15 | 12712.258 | 126.400 | 59.1 |
| 16 | 7775.666 | 77.315 | 161.7 |
| 17 | 7743.988 | 77.000 | 160.3 |
| 18 | 7711.574 | 76.678 | 162.0 |
| 19 | 7539.924 | 74.971 | 31.5 |
| 20 | 7450.047 | 74.077 | 12.9 |
| 21 | 6753.871 | 67.155 | 23.2 |
| 22 | 6676.518 | 66.386 | 57.8 |
| 23 | 4932.761 | 49.047 | 25.2 |
| 24 | 4845.095 | 48.176 | 22.1 |
| 25 | 4779.529 | 47.524 | 71.4 |
| 26 | 4774.372 | 47.473 | 64.8 |
| 27 | 4316.885 | 42.924 | 21.3 |
| 28 | 4164.389 | 41.407 | 60.6 |
| 29 | 4063.462 | 40.404 | 21.2 |
| 30 | 3938.224 | 39.159 | 73.5 |
| 31 | 3261.938 | 32.434 | 69.7 |
| 32 | 3231.733 | 32.134 | 23.7 |
| 33 | 2870.016 | 28.537 | 59.7 |
| 34 | 2780.876 | 27.651 | 20.4 |
| 35 | 2560.605 | 25.461 | 21.9 |
| 36 | 2541.450 | 25.270 | 64.6 |




**58**


Standard Proton
File: JDN-II-195-1HNMR-2
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-195-1HNMR-2
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042587 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jun 11 2008
H
N
Boc
CF3
59
8
7
6
5
4
3
2
1
ppm
4.00
0.40 0.29
0.64 0.62
0.51
1.36
0.47
0.58
20.00

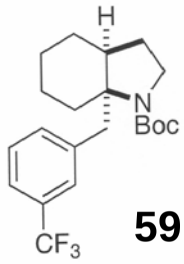
Std Carbon

File: mnt/i400/jdneukom/vnmrsys/data/JDN-II-195-13CNMR.fid

Pulse Sequence: s2pul

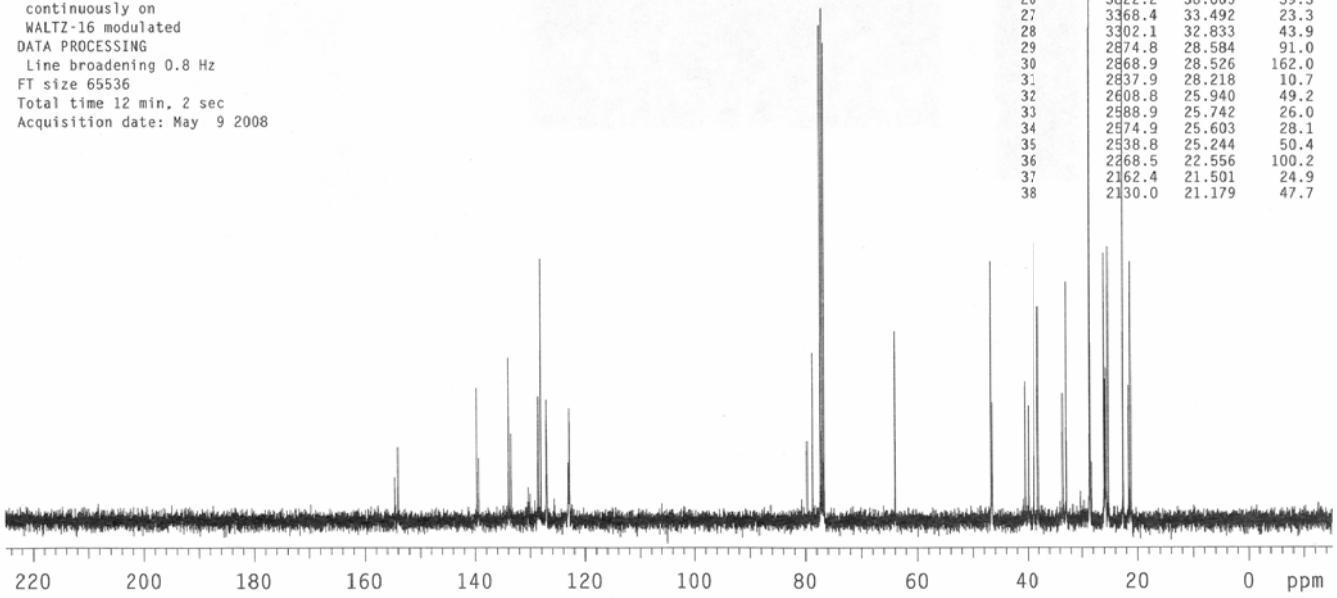
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-195-13CNMR
INOVA-400 "Dy.Chem.LSA.UMich.Edu"

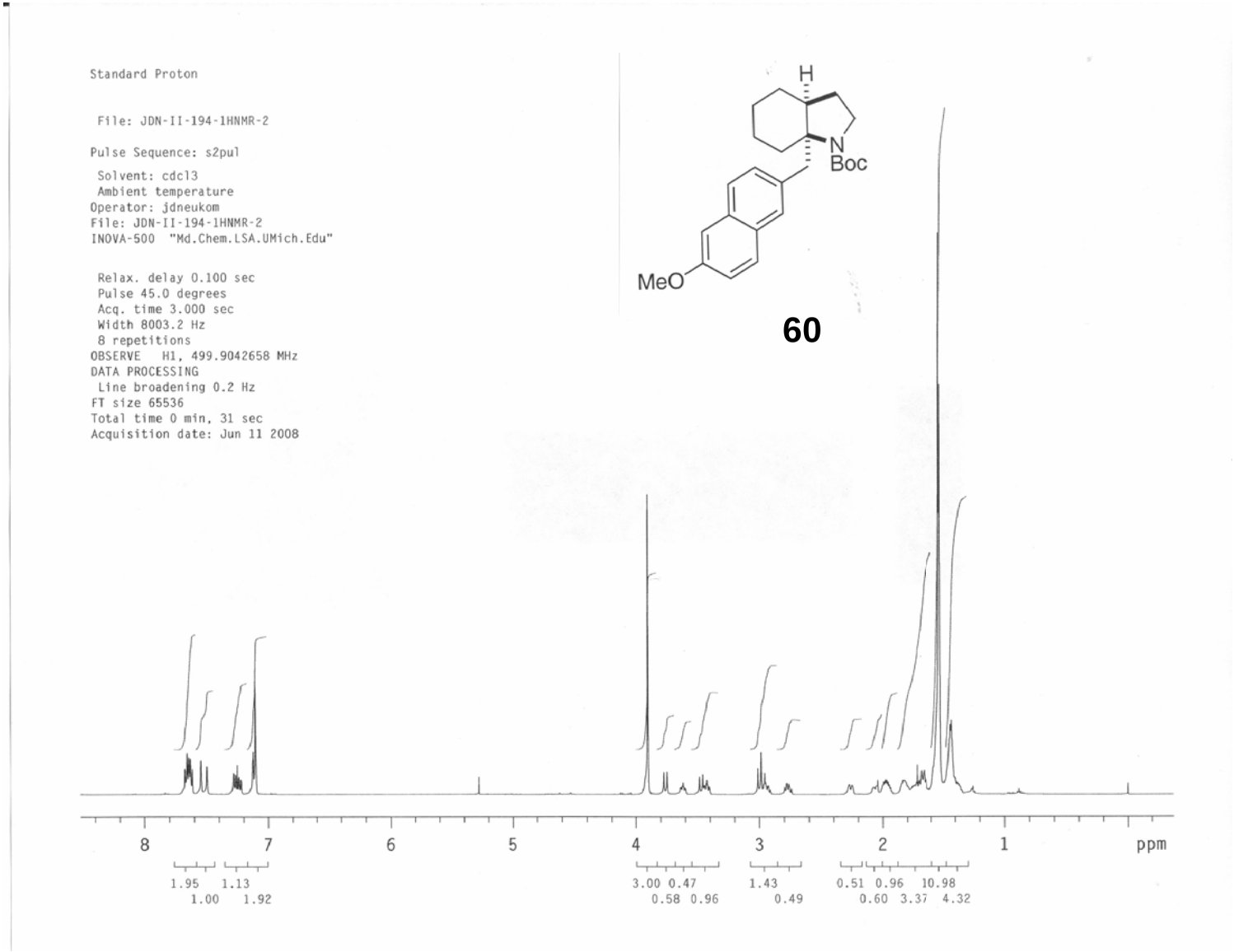
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
512 repetitions
OBSERVE C13, 100.5712703 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 12 min, 2 sec
Acquisition date: May 9 2008




**59**

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15542.2 | 154.540 | 8.0 |
| 2 | 15487.7 | 153.997 | 13.5 |
| 3 | 14046.0 | 139.662 | 24.4 |
| 4 | 14009.9 | 139.303 | 11.5 |
| 5 | 13464.8 | 133.883 | 30.0 |
| 6 | 13422.0 | 133.458 | 16.0 |
| 7 | 13111.1 | 130.367 | 6.2 |
| 8 | 12923.3 | 128.499 | 22.9 |
| 9 | 12878.4 | 128.052 | 48.2 |
| 10 | 12773.7 | 127.012 | 18.5 |
| 11 | 12769.3 | 126.968 | 22.2 |
| 12 | 12373.7 | 123.034 | 10.7 |
| 13 | 12352.4 | 122.822 | 20.7 |
| 14 | 12348.7 | 122.785 | 18.1 |
| 15 | 8011.7 | 79.662 | 14.6 |
| 16 | 7912.3 | 78.673 | 30.8 |
| 17 | 7773.1 | 77.289 | 91.5 |
| 18 | 7741.4 | 76.974 | 94.6 |
| 19 | 7709.0 | 76.652 | 88.2 |
| 20 | 6433.7 | 63.972 | 34.9 |
| 21 | 4686.3 | 46.597 | 47.7 |
| 22 | 4675.3 | 46.487 | 21.7 |
| 23 | 4064.5 | 40.414 | 25.5 |
| 24 | 4007.1 | 39.843 | 21.1 |
| 25 | 3898.8 | 38.766 | 51.0 |
| 26 | 3822.2 | 38.005 | 39.3 |
| 27 | 3368.4 | 33.492 | 23.3 |
| 28 | 3302.1 | 32.833 | 43.9 |
| 29 | 2874.8 | 28.584 | 91.0 |
| 30 | 2868.9 | 28.526 | 162.0 |
| 31 | 2837.9 | 28.218 | 10.7 |
| 32 | 2608.8 | 25.940 | 49.2 |
| 33 | 2588.9 | 25.742 | 26.0 |
| 34 | 2574.9 | 25.603 | 28.1 |
| 35 | 2538.8 | 25.244 | 50.4 |
| 36 | 2268.5 | 22.556 | 100.2 |
| 37 | 2162.4 | 21.501 | 24.9 |
| 38 | 2130.0 | 21.179 | 47.7 |




Standard Proton
File: JDN-II-194-1HNMR-2
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-194-1HNMR-2
INOVA-500 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042658 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jun 11 2008
H
N
Boc
MeO
60
8
7
6
5
4
3
2
1
ppm
1.95 1.13
1.00 1.92
3.00 0.47
0.58 0.96
1.43
0.49
0.51 0.96 10.98
0.60 3.37 4.32

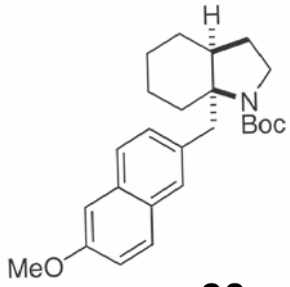
Std Carbon

File: mnt/i400/jdneukom/vnmrsys/data/JDN-II-194-13CNMR.fid

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-II-194-13CNMR
INOVA-400 "Dy.Chem.LSA.UMich.Ecu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
512 repetitions
OBSERVE C13, 100.5712703 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 12 min, 2 sec
Acquisition date: May 8 2008

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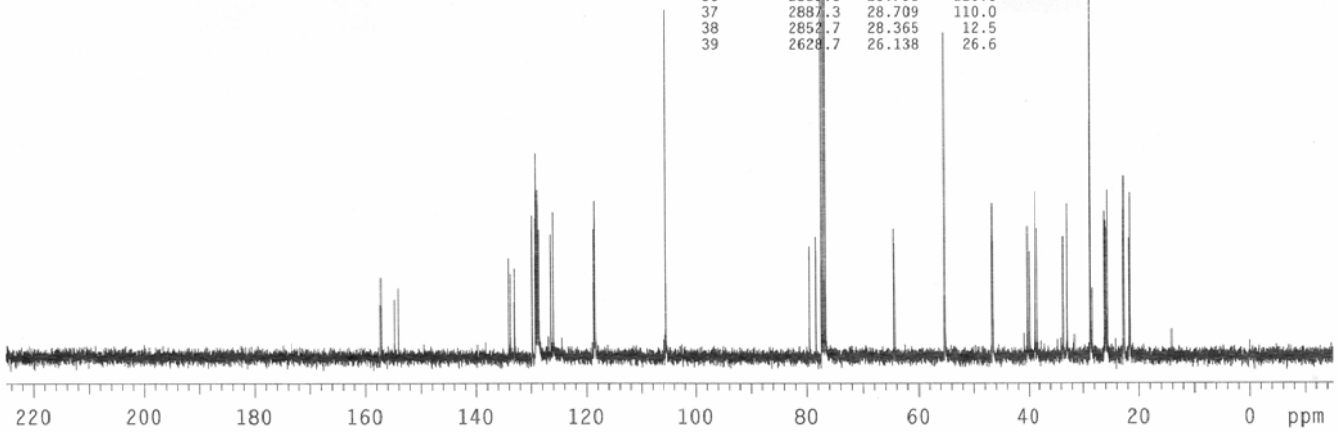
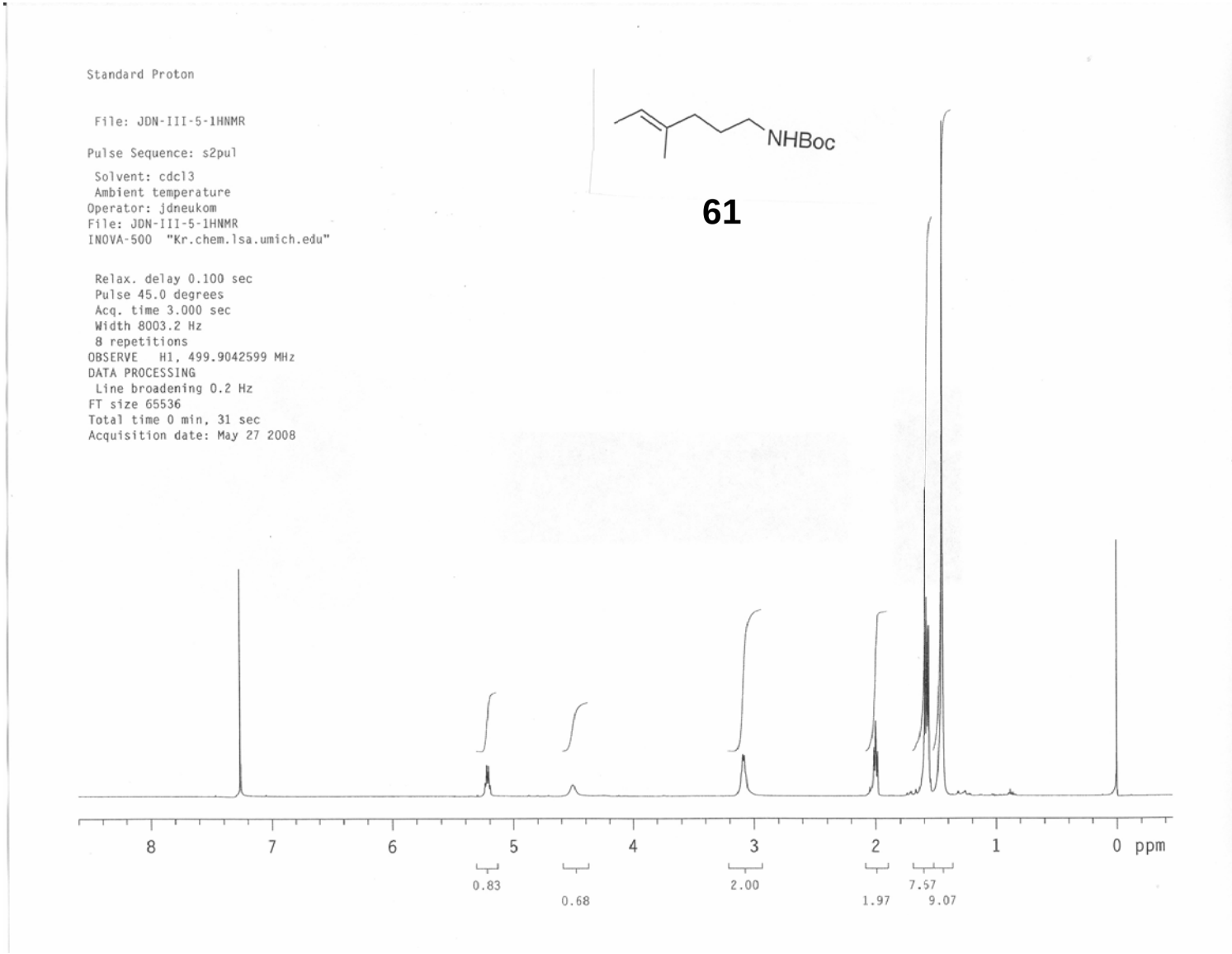
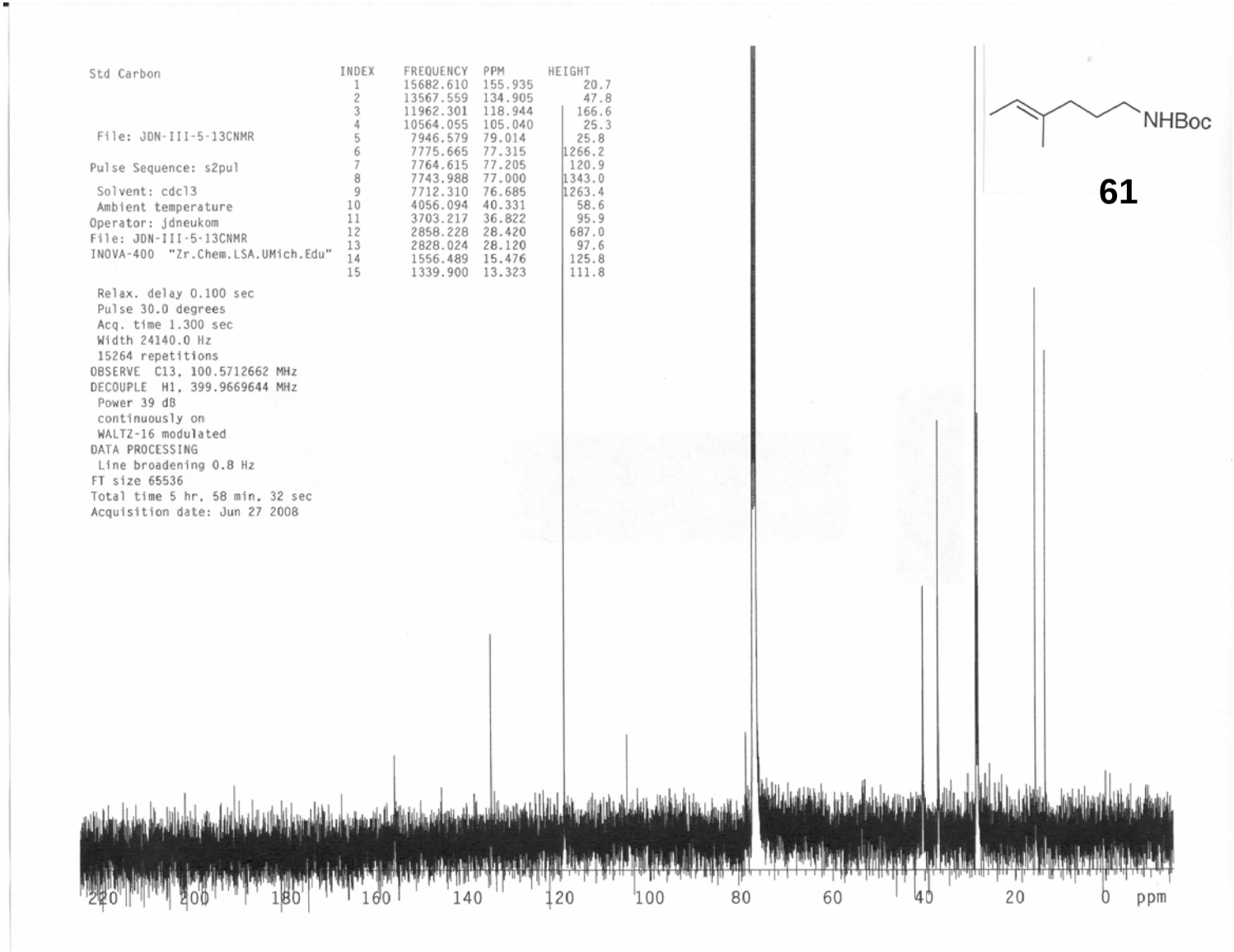
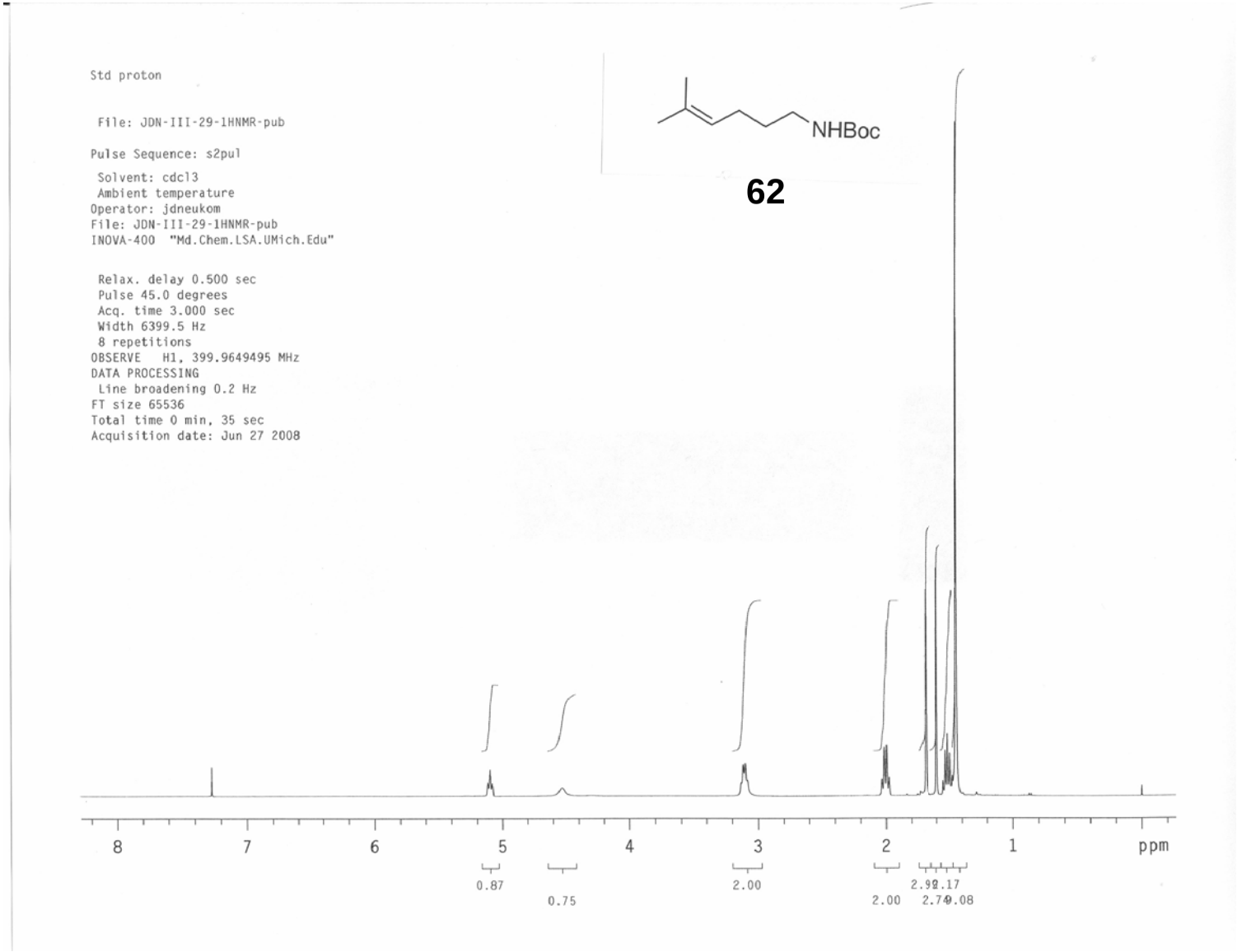
| INDEX | FREQUENCY | PPM | HEIGHT | INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|---|---|---|---|
| 1 | 15816.3 | 157.264 | 9.5 | 40 | 2605.1 | 25.903 | 24.9 |
| 2 | 15803.8 | 157.140 | 14.5 | 41 | 2594.1 | 25.794 | 23.6 |
| 3 | 15565.1 | 154.767 | 10.5 | 42 | 2569.8 | 25.552 | 30.5 |
| 4 | 15489.9 | 154.019 | 12.5 | 43 | 2281.0 | 22.680 | 27.1 |
| 5 | 13478.8 | 134.022 | 18.0 | 44 | 2276.6 | 22.636 | 33.1 |
| 6 | 13445.6 | 133.692 | 15.1 | 45 | 2179.3 | 21.670 | 21.7 |
| 7 | 13385.2 | 133.092 | 10.0 | 46 | 2161.7 | 21.494 | 30.0 |
| 8 | 13376.4 | 133.004 | 16.1 | | | | |
| 9 | 13050.7 | 129.766 | 25.8 | | | | |
| 10 | 12988.9 | 129.151 | 25.3 | | | | |
| 11 | 12977.1 | 129.034 | 37.2 | | | | |
| 12 | 12956.4 | 128.828 | 20.7 | | | | |
| 13 | 12952.8 | 128.792 | 30.5 | | | | |
| 14 | 12931.4 | 128.579 | 23.2 | | | | |
| 15 | 12715.5 | 126.433 | 22.4 | | | | |
| 16 | 12668.4 | 125.964 | 26.4 | | | | |
| 17 | 11929.5 | 118.617 | 23.3 | | | | |
| 18 | 11910.3 | 118.427 | 28.5 | | | | |
| 19 | 10602.7 | 105.425 | 63.6 | | | | |
| 20 | 7990.4 | 79.450 | 20.1 | | | | |
| 21 | 7881.4 | 78.366 | 21.9 | | | | |
| 22 | 7776.7 | 77.326 | 76.8 | | | | |
| 23 | 7745.1 | 77.011 | 78.4 | | | | |
| 24 | 7712.6 | 76.688 | 73.0 | | | | |
| 25 | 6475.0 | 64.382 | 23.3 | | | | |
| 26 | 6466.2 | 64.294 | 20.6 | | | | |
| 27 | 5556.3 | 55.248 | 59.5 | | | | |
| 28 | 4690.0 | 46.633 | 28.0 | | | | |
| 29 | 4681.1 | 46.546 | 21.1 | | | | |
| 30 | 4046.1 | 40.231 | 23.8 | | | | |
| 31 | 4012.2 | 39.894 | 19.2 | | | | |
| 32 | 3896.6 | 38.744 | 30.2 | | | | |
| 33 | 3854.6 | 38.327 | 23.4 | | | | |
| 34 | 3379.7 | 33.595 | 21.9 | | | | |
| 35 | 3307.2 | 32.884 | 27.9 | | | | |
| 36 | 2889.5 | 28.731 | 126.0 | | | | |
| 37 | 2887.3 | 28.709 | 110.0 | | | | |
| 38 | 2852.7 | 28.365 | 12.5 | | | | |
| 39 | 2628.7 | 26.138 | 26.6 | | | | |




Standard Proton
File: JDN-III-5-1HNMR
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-III-5-1HNMR
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042599 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: May 27 2008
NHBoc
61
8
7
6
5
4
3
2
1
0 ppm
0.83
0.68
2.00
1.97
7.57
9.07


Std Carbon
File: JDN-III-5-13CNMR
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-III-5-13CNMR
INOVA-400 "Zr.Chem.LSA.UMich.Edu"
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
15264 repetitions
OBSERVE C13, 100.5712662 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 5 hr, 58 min, 32 sec
Acquisition date: Jun 27 2008
INDEX FREQUENCY PPM HEIGHT
1 15682.610 155.935 20.7
2 13567.559 134.905 47.8
3 11962.301 118.944 166.6
4 10564.055 105.040 25.3
5 7946.579 79.014 25.8
6 7775.665 77.315 1266.2
7 7764.615 77.205 120.9
8 7743.988 77.000 1343.0
9 7712.310 76.685 1263.4
10 4056.094 40.331 58.6
11 3703.217 36.822 95.9
12 2858.228 28.420 687.0
13 2828.024 28.120 97.6
14 1556.489 15.476 125.8
15 1339.900 13.323 111.8
NHBoc
61
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Std proton
File: JDN-III-29-1HNMR-pub
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-III-29-1HNMR-pub
INOVA-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649495 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Jun 27 2008
NHBoc
62
8
7
6
5
4
3
2
1
ppm
0.87
0.75
2.00
2.00
2.99
2.17
2.74
9.08

Std Carbon

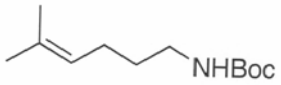
File: JDN-III-29-13CNMR

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
Operator: jdneukom
File: JDN-III-29-13CNMR
INOVA-400 "Md.Chem.LSA.UMich.Edu"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
256 repetitions
OBSERVE C13, 100.5712685 MHz
DECOUPLE H1, 399.9669644 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 6 min, 0 sec
Acquisition date: Jun 27 2008

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 15681.873 | 155.928 | 4.5 |
| 2 | 13292.772 | 132.173 | 12.5 |
| 3 | 12425.683 | 123.551 | 42.0 |
| 4 | 7939.948 | 78.948 | 4.9 |
| 5 | 7776.402 | 77.322 | 66.1 |
| 6 | 7743.988 | 77.000 | 67.1 |
| 7 | 7712.310 | 76.685 | 66.9 |
| 8 | 4050.937 | 40.279 | 12.9 |
| 9 | 3022.511 | 30.053 | 16.9 |
| 10 | 2856.018 | 28.398 | 162.0 |
| 11 | 2581.968 | 25.673 | 47.6 |
| 12 | 2542.923 | 25.285 | 20.5 |
| 13 | 1773.077 | 17.630 | 49.1 |

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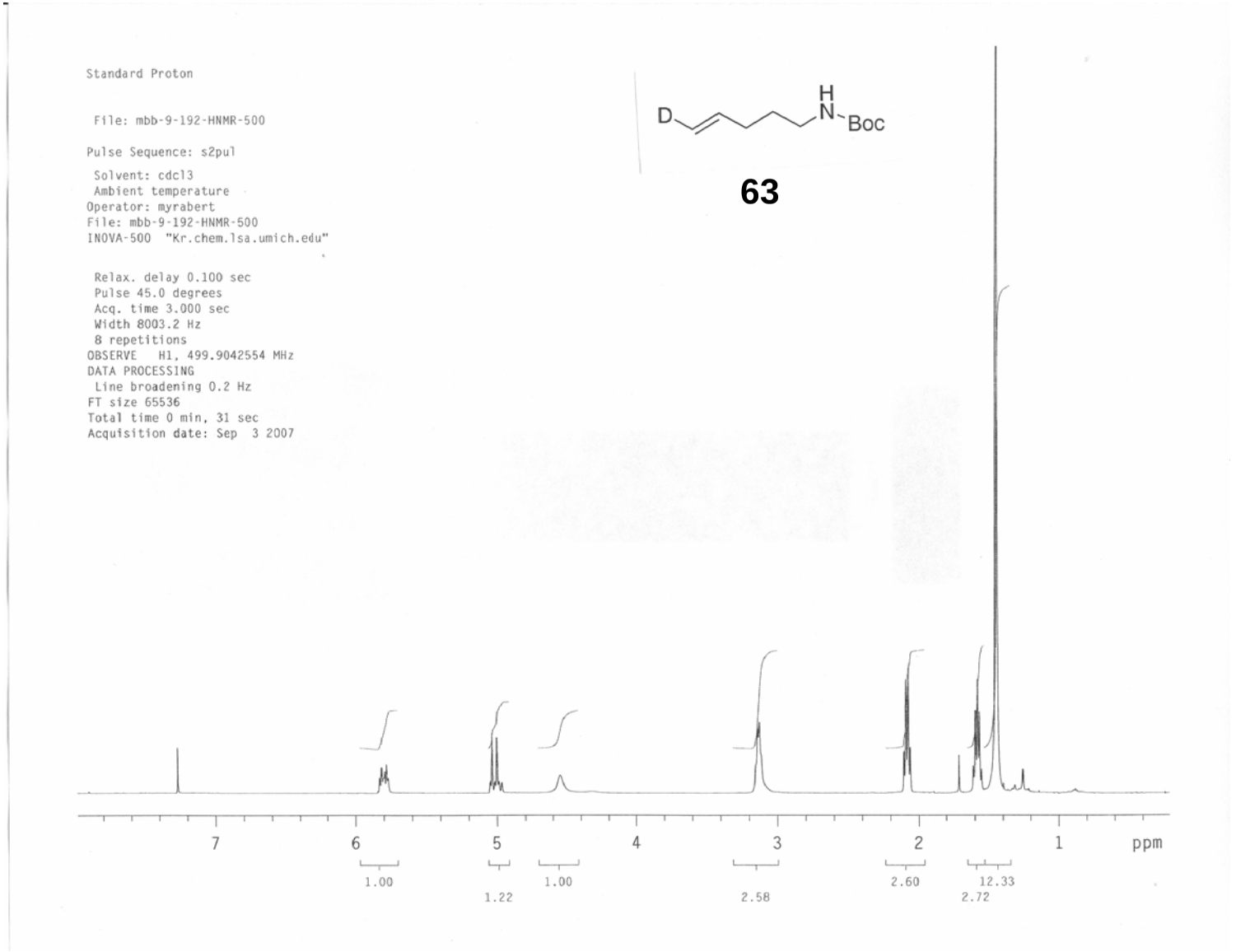
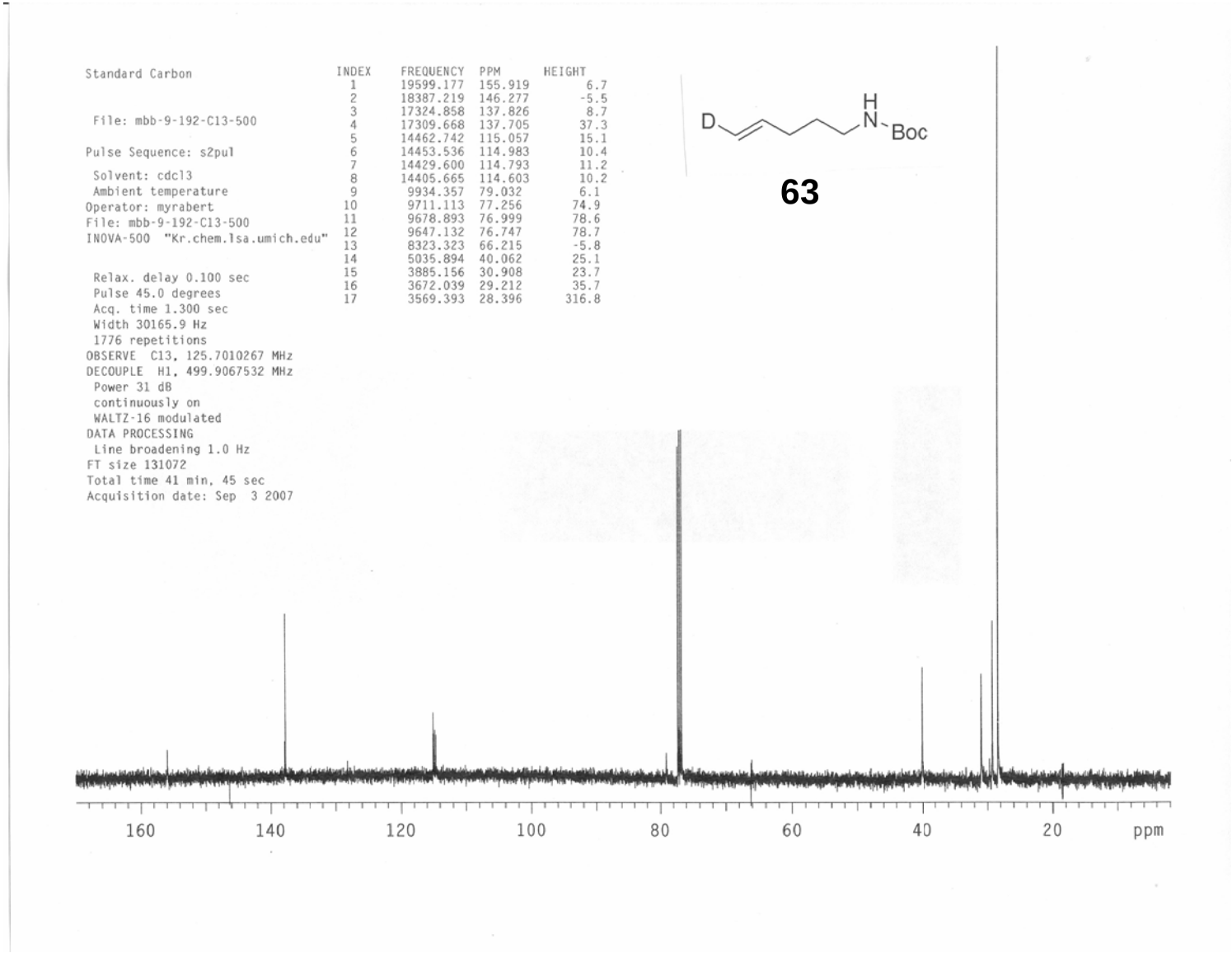
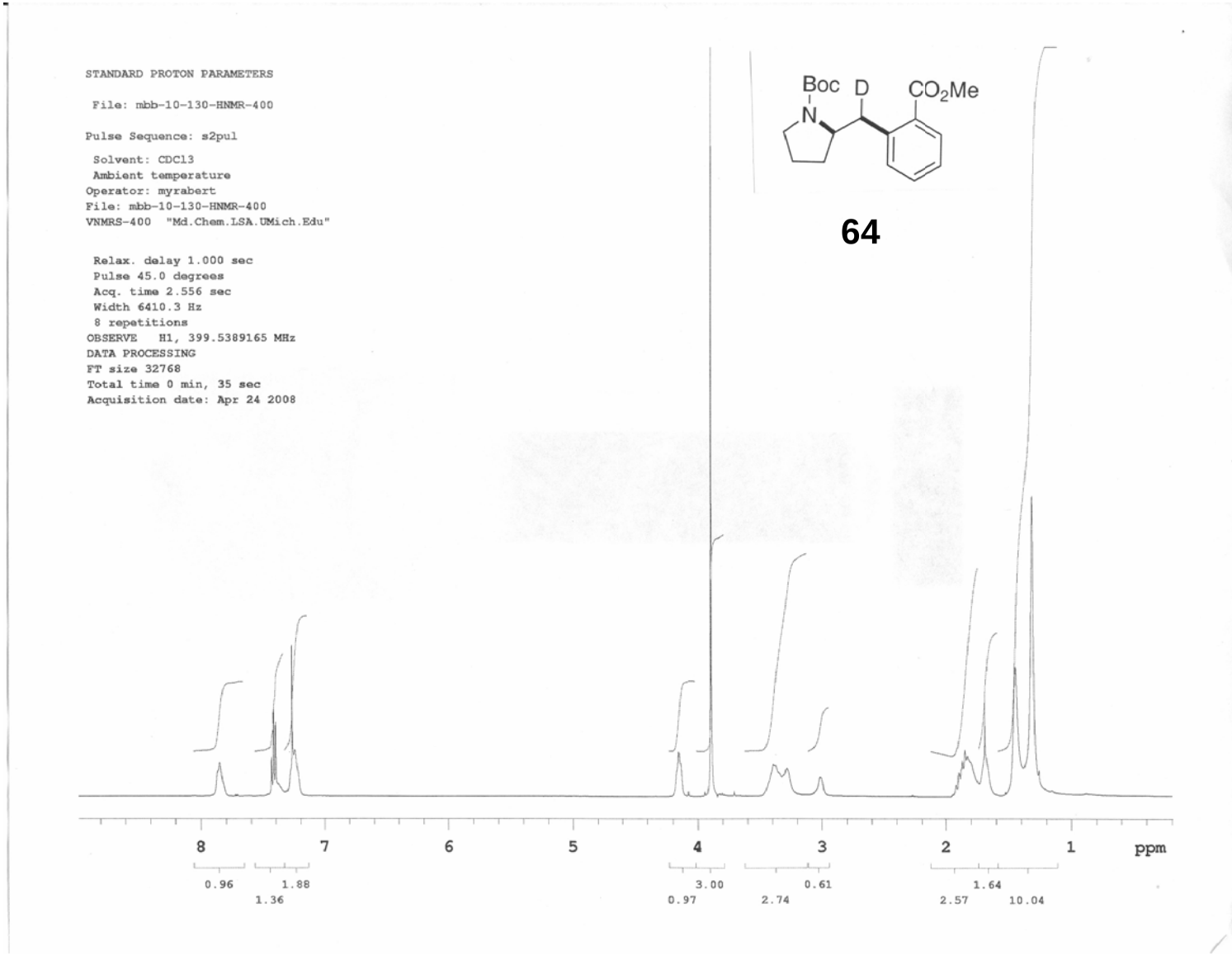
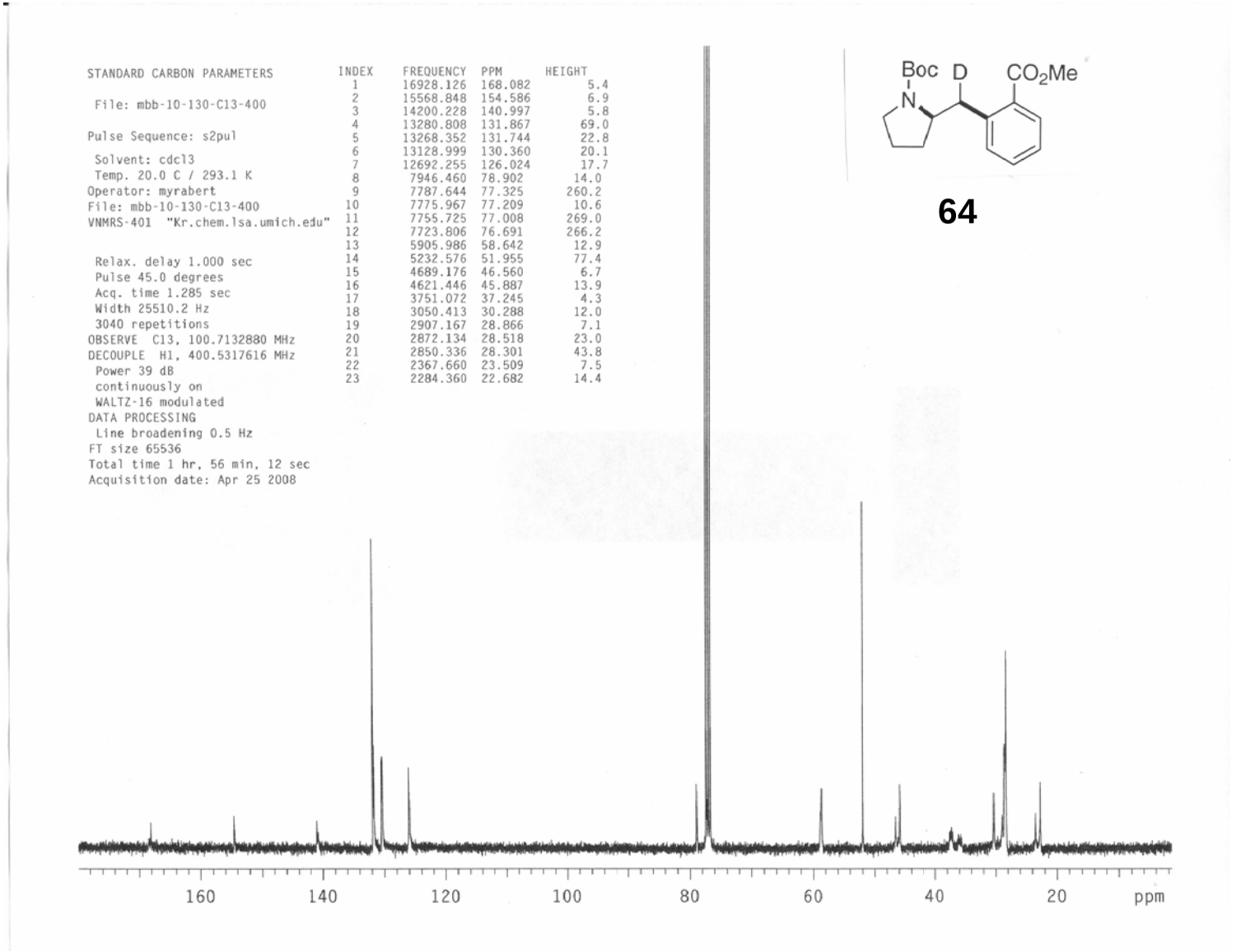
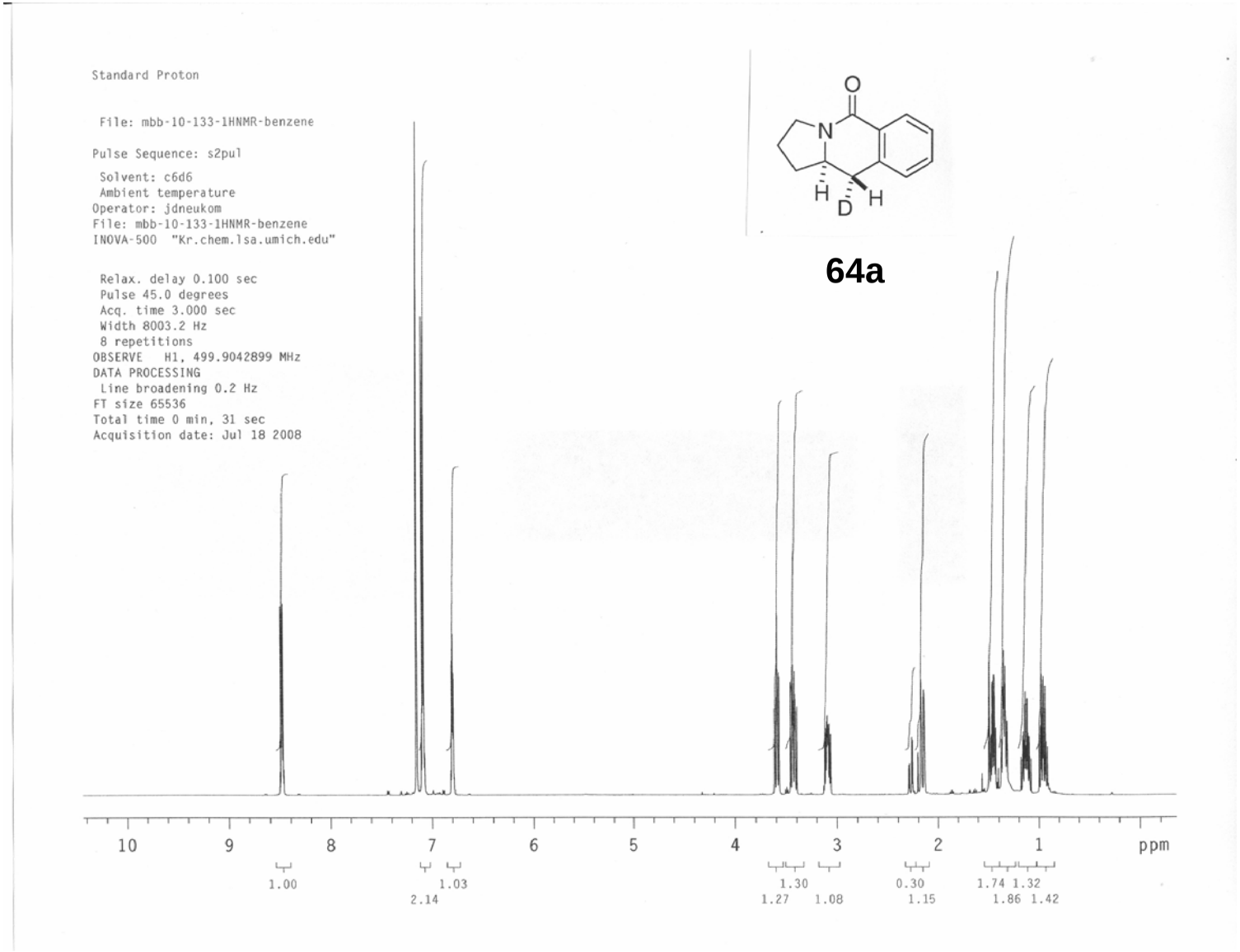
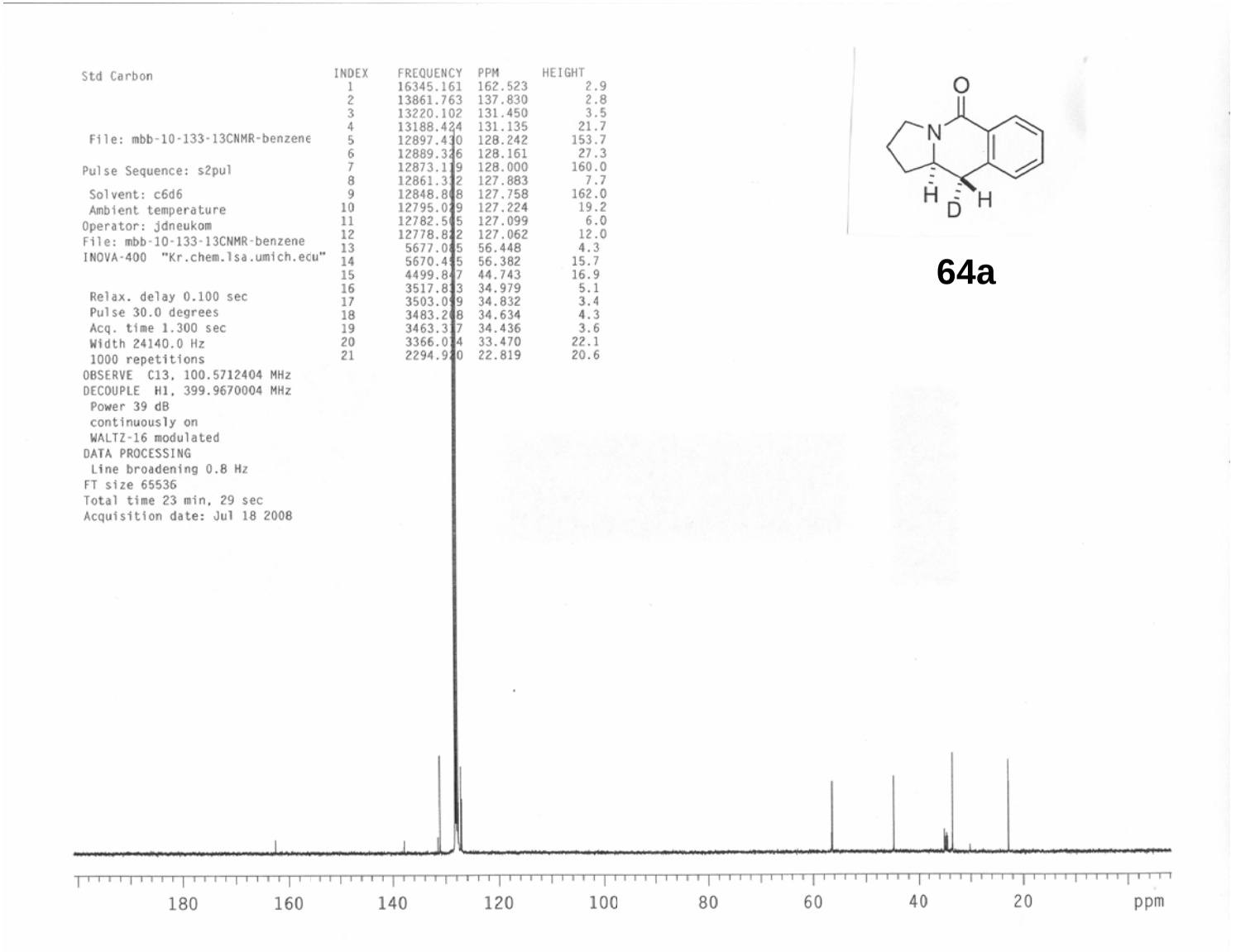

Standard Proton
File: mbb-9-192-HNMR-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-192-HNMR-500
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042554 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Sep 3 2007
D
H
N
Boc
63
7
6
5
4
3
2
1
ppm
1.00
1.22
1.00
2.58
2.60
12.33
2.72

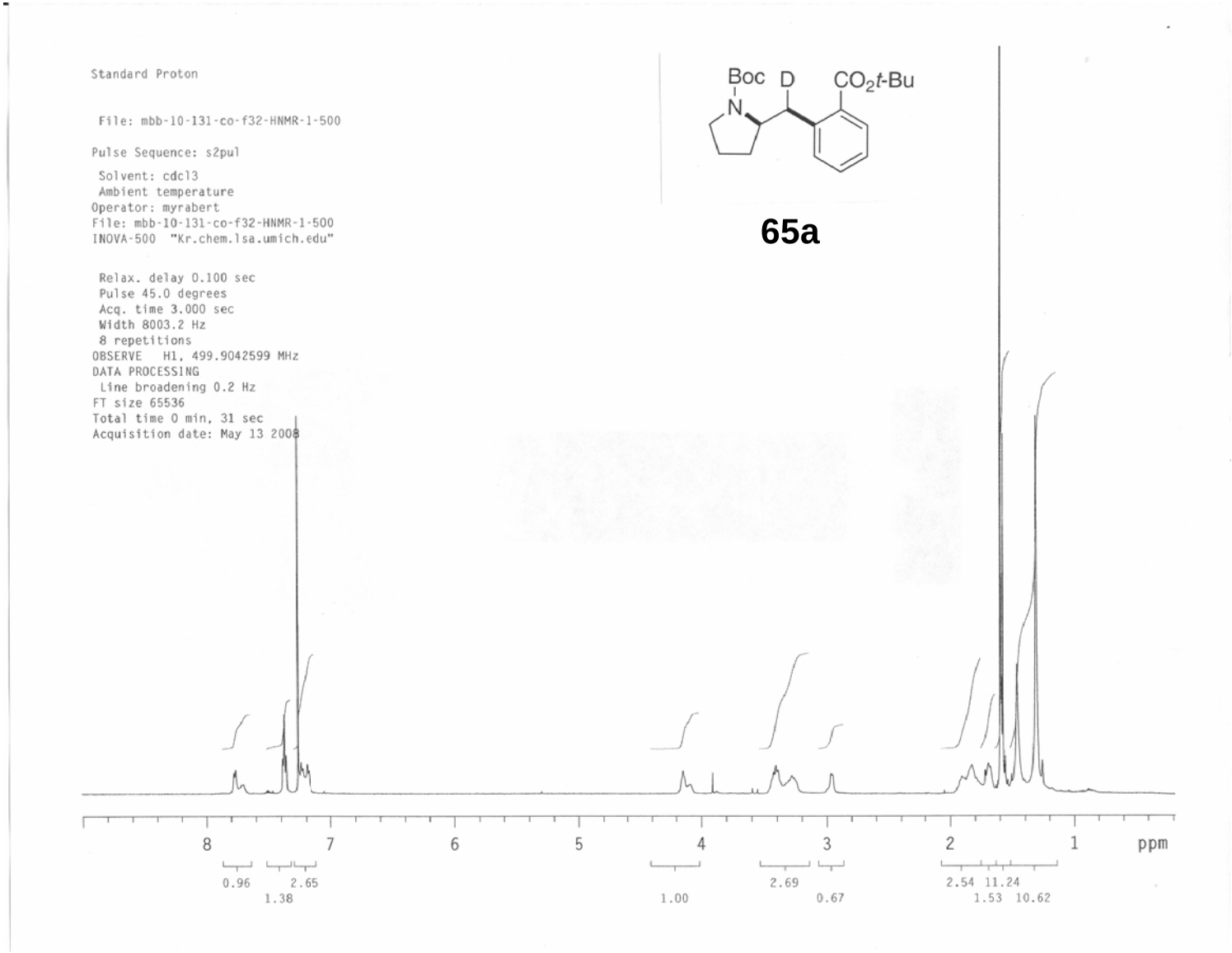

Standard Carbon
File: mbb-9-192-C13-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-9-192-C13-500
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
1776 repetitions
OBSERVE C13, 125.7010267 MHz
DECOUPLE H1, 499.9067532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 41 min, 45 sec
Acquisition date: Sep 3 2007
INDEX FREQUENCY PPM HEIGHT
1 19599.177 155.919 6.7
2 18387.219 146.277 -5.5
3 17324.858 137.826 8.7
4 17309.668 137.705 37.3
5 14462.742 115.057 15.1
6 14453.536 114.983 10.4
7 14429.600 114.793 11.2
8 14405.665 114.603 10.2
9 9934.357 79.032 6.1
10 9711.113 77.256 74.9
11 9678.893 76.999 78.6
12 9647.132 76.747 78.7
13 8323.323 66.215 -5.8
14 5035.894 40.062 25.1
15 3885.156 30.908 23.7
16 3672.039 29.212 35.7
17 3569.393 28.396 316.8
D
H
N
Boc
63
160
140
120
100
80
60
40
20
ppm

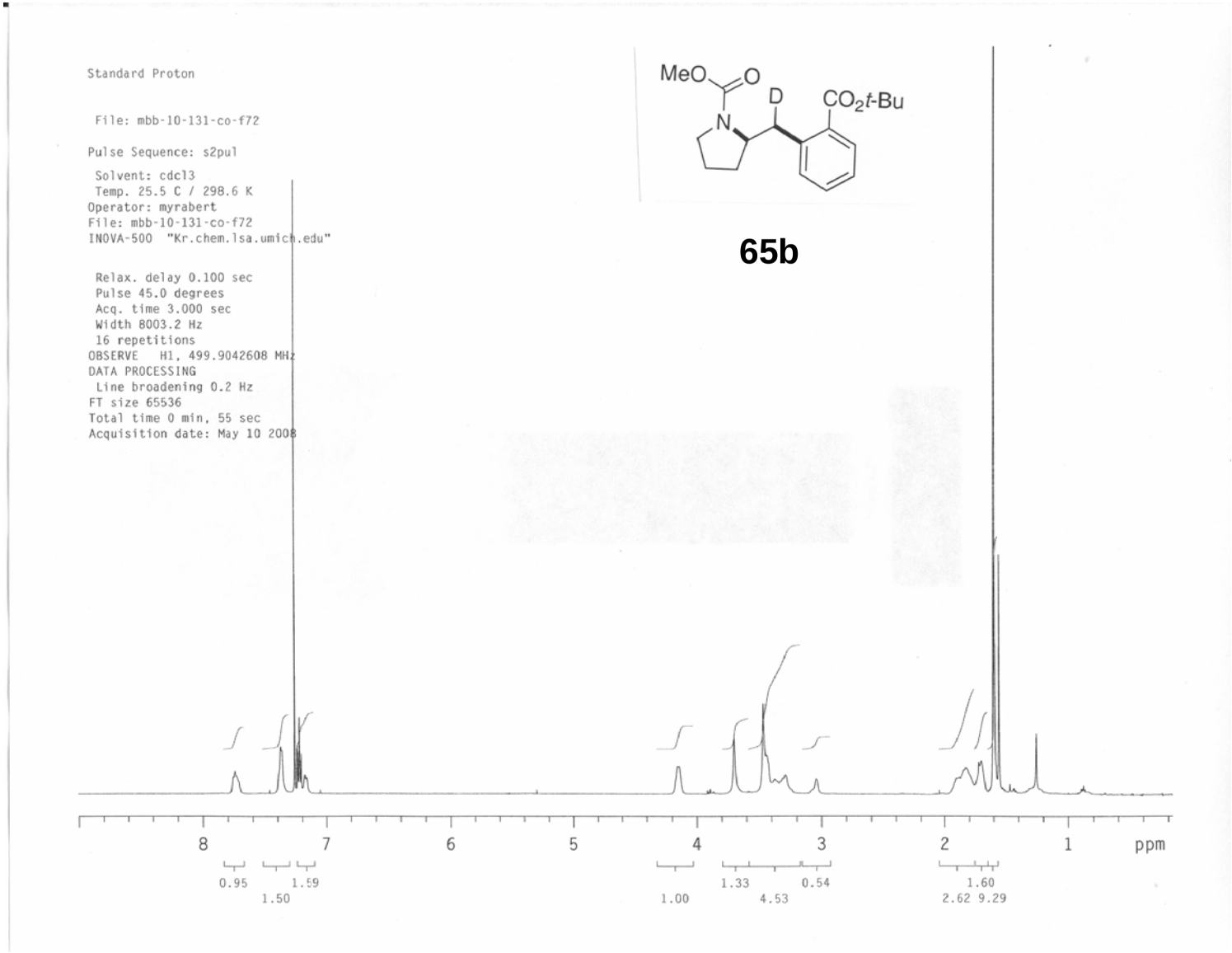

STANDARD PROTON PARAMETERS
File: mbb-10-130-HNMR-400
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: myrabert
File: mbb-10-130-HNMR-400
VNMRS-400 "Md.Chem.LSA.UMich.Edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.5389165 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec
Acquisition date: Apr 24 2008
Boc
D
CO2Me
N
64
8
7
6
5
4
3
2
1
ppm
0.96
1.88
1.36
3.00
0.97
0.61
2.74
1.64
2.57
10.04

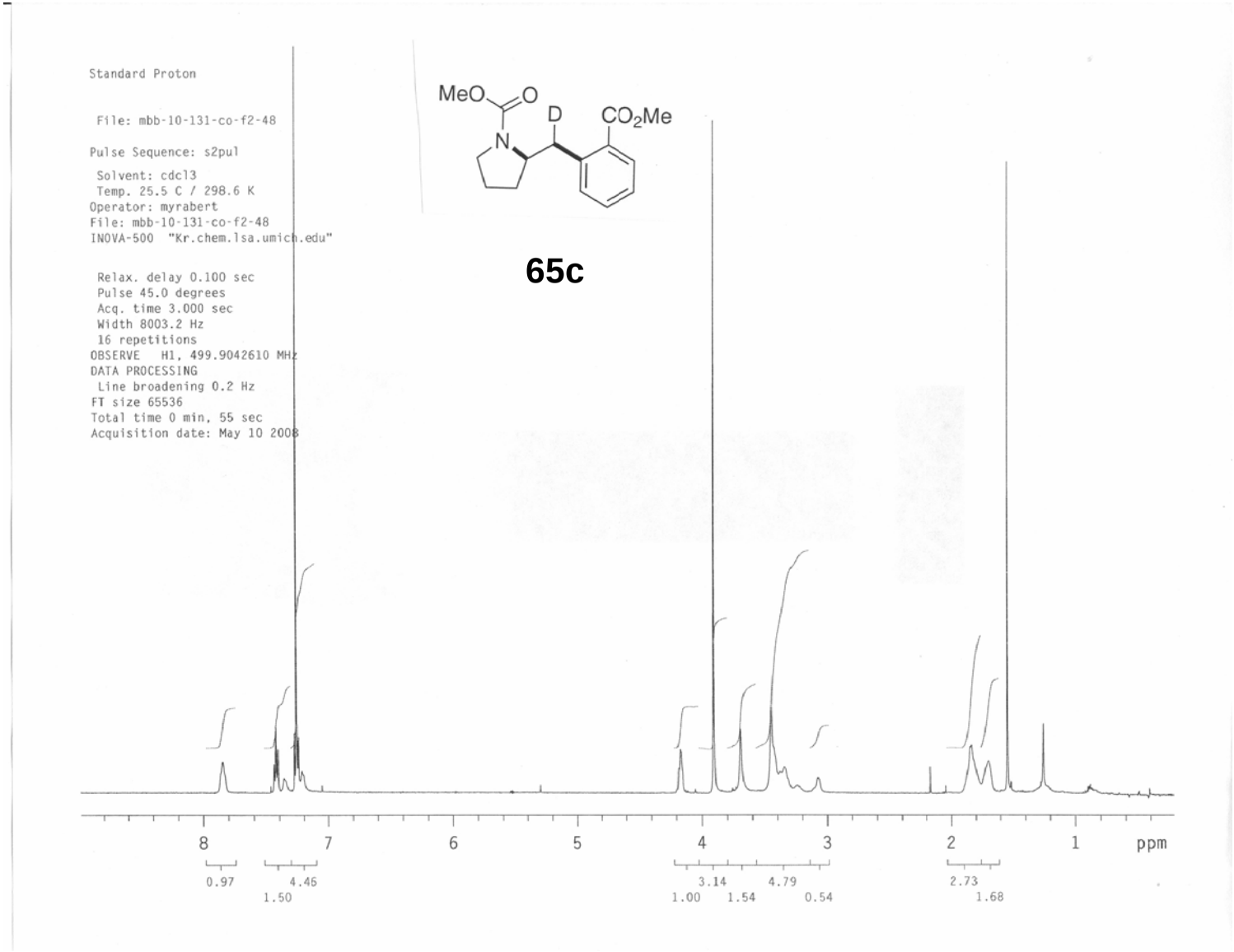

STANDARD CARBON PARAMETERS
File: mbb-10-130-C13-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 20.0 C / 293.1 K
Operator: myrabert
File: mbb-10-130-C13-400
VNMRS-401 "Kr.chem.lsa.umich.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
3040 repetitions
OBSERVE C13, 100.7132880 MHz
DECOUPLE H1, 400.5317616 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 56 min, 12 sec
Acquisition date: Apr 25 2008
INDEX FREQUENCY PPM HEIGHT
1 16928.126 168.082 5.4
2 15568.848 154.586 6.9
3 14200.228 140.997 5.8
4 13280.808 131.867 69.0
5 13268.352 131.744 22.8
6 13128.999 130.360 20.1
7 12692.255 126.024 17.7
8 7946.460 78.902 14.0
9 7787.644 77.325 260.2
10 7775.967 77.209 10.6
11 7755.725 77.008 269.0
12 7723.806 76.691 266.2
13 5905.986 58.642 12.9
14 5232.576 51.955 77.4
15 4689.176 46.560 6.7
16 4621.446 45.887 13.9
17 3751.072 37.245 4.3
18 3050.413 30.288 12.0
19 2907.167 28.866 7.1
20 2872.134 28.518 23.0
21 2850.336 28.301 43.8
22 2367.660 23.509 7.5
23 2284.360 22.682 14.4
Boc
D
CO2Me
N
64
160
140
120
100
80
60
40
20
ppm

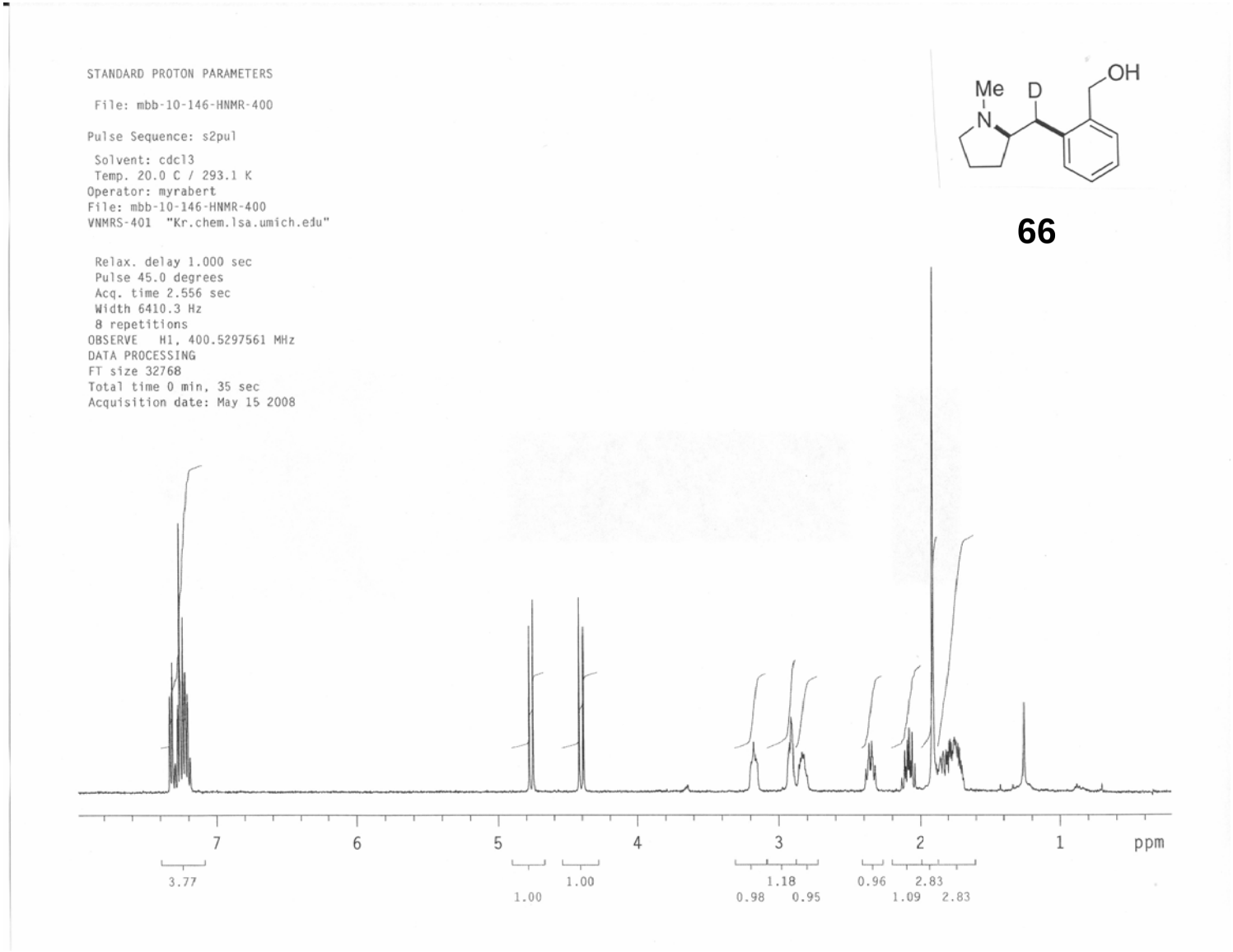

Standard Proton
File: mbb-10-133-1HNMR-benzene
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
Operator: jdneukom
File: mbb-10-133-1HNMR-benzene
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042899 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Jul 18 2008
O
N
H
D
H
64a
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.14
1.03
1.27
1.30
1.08
0.30
1.15
1.74
1.86
1.32
1.42


Std Carbon
File: mbb-10-133-13CNMR-benzene
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
Operator: jdneukom
File: mbb-10-133-13CNMR-benzene
INOVA-400 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 1.300 sec
Width 24140.0 Hz
1000 repetitions
OBSERVE C13, 100.5712404 MHz
DECOUPLE H1, 399.9670004 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 23 min, 29 sec
Acquisition date: Jul 18 2008
INDEX FREQUENCY PPM HEIGHT
1 16345.161 162.523 2.9
2 13861.763 137.830 2.8
3 13220.102 131.450 3.5
4 13188.424 131.135 21.7
5 12897.430 128.242 153.7
6 12889.326 128.161 27.3
7 12873.119 128.000 160.0
8 12861.312 127.883 7.7
9 12848.868 127.758 162.0
10 12795.019 127.224 19.2
11 12782.565 127.099 6.0
12 12778.812 127.062 12.0
13 5677.085 56.448 4.3
14 5670.455 56.382 15.7
15 4499.847 44.743 16.9
16 3517.813 34.979 5.1
17 3503.019 34.832 3.4
18 3483.268 34.634 4.3
19 3463.317 34.436 3.6
20 3366.014 33.470 22.1
21 2294.930 22.819 20.6
O
N
H
D
H
64a
180
160
140
120
100
80
60
40
20
ppm


Standard Proton
File: mbb-10-131-co-f32-HNMR-1-500
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: myrabert
File: mbb-10-131-co-f32-HNMR-1-500
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042599 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: May 13 2008
Boc
D
CO2t-Bu
N
65a
8
7
6
5
4
3
2
1
ppm
0.96
2.65
1.38
1.00
2.69
0.67
2.54
11.24
1.53
10.62


Standard Proton
File: mbb-10-131-co-f72
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.5 C / 298.6 K
Operator: myrabert
File: mbb-10-131-co-f72
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
16 repetitions
OBSERVE H1, 499.9042608 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Acquisition date: May 10 2008
MeO
O
N
D
CO2t-Bu
65b
8
7
6
5
4
3
2
1
ppm
0.95
1.59
1.50
1.00
1.33
0.54
4.53
1.60
2.62 9.29


Standard Proton
File: mbb-10-131-co-f2-48
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.5 C / 298.6 K
Operator: myrabert
File: mbb-10-131-co-f2-48
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
16 repetitions
OBSERVE H1, 499.9042610 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 55 sec
Acquisition date: May 10 2008
MeO
O
N
D
CO2Me
65c
8
7
6
5
4
3
2
1
ppm
0.97
4.45
1.50
3.14
4.79
1.00
1.54
0.54
2.73
1.68


STANDARD PROTON PARAMETERS
File: mbb-10-146-HNMR-400
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 20.0 C / 293.1 K
Operator: myrabert
File: mbb-10-146-HNMR-400
VNMRS-401 "Kr.chem.lsa.umich.edu"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.5297561 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec
Acquisition date: May 15 2008
Me
N
D
OH
66
7
6
5
4
3
2
1
ppm
3.77
1.00
1.00
1.18
0.98
0.95
0.96
2.83
1.09
2.83

STANDARD CARBON PARAMETERS

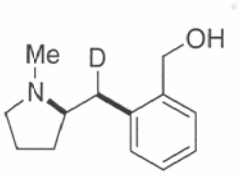
File: mbb-10-146-C13-400

Pulse Sequence: s2pul

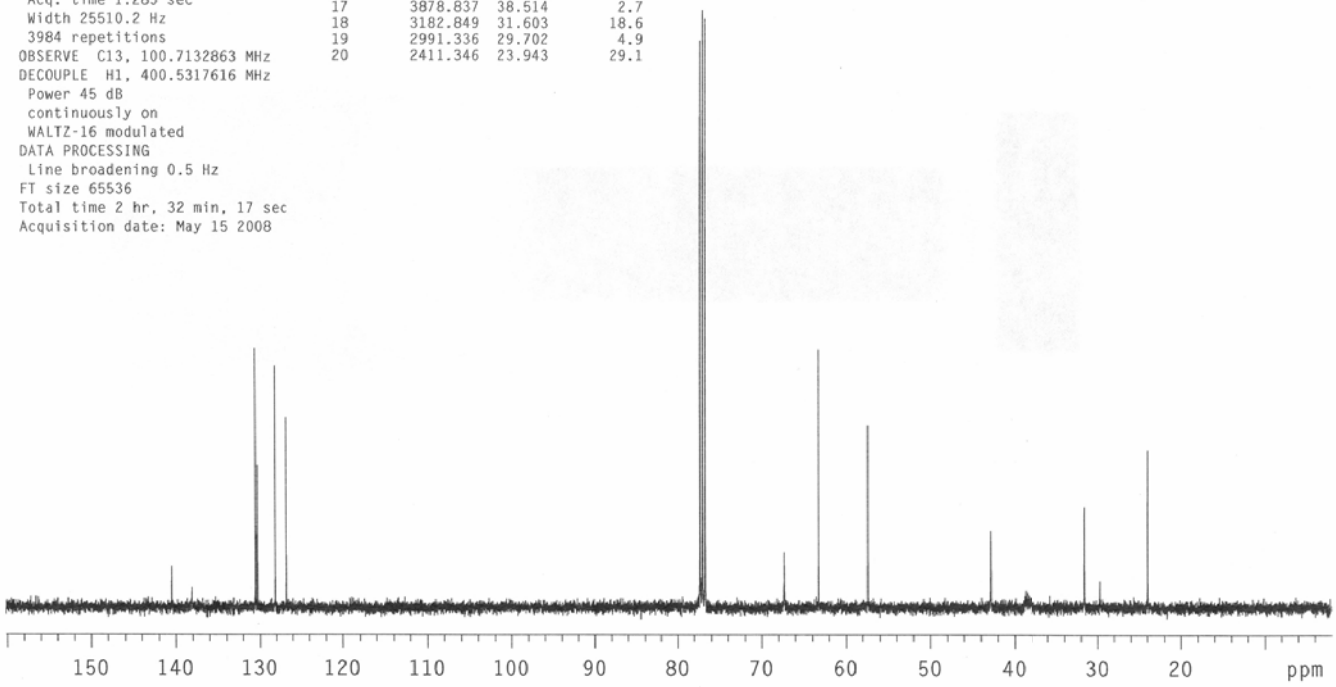
Solvent: cdcl3
Temp. 20.0 C / 293.1 K
Operator: myrabert
File: mbb-10-146-C13-400
VNMRS-401 "Kr.chem.lsa.umich.edu"

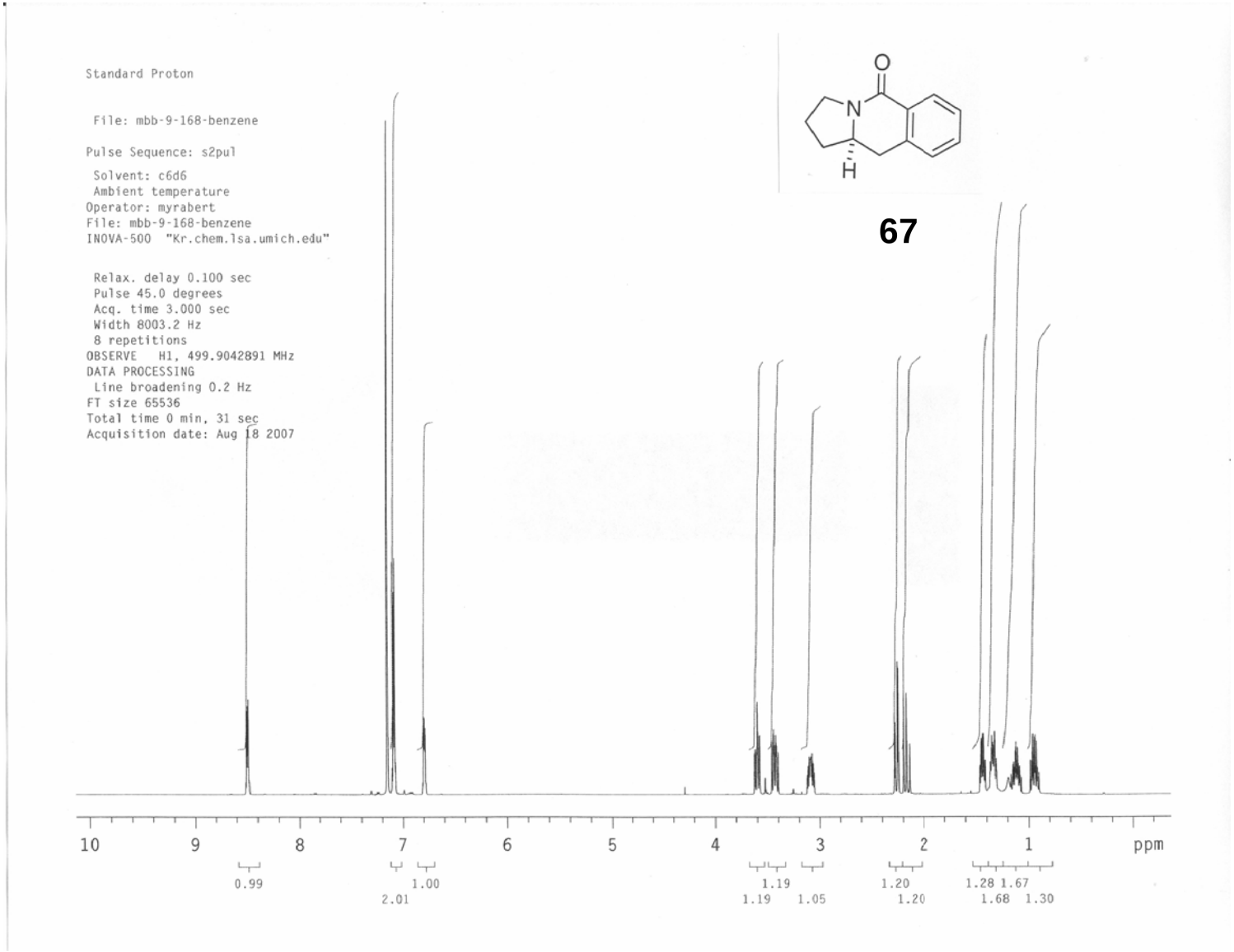
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
3984 repetitions
OBSERVE C13, 100.7132863 MHz
DECOUPLE H1, 400.5317616 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 hr, 32 min, 17 sec
Acquisition date: May 15 2008

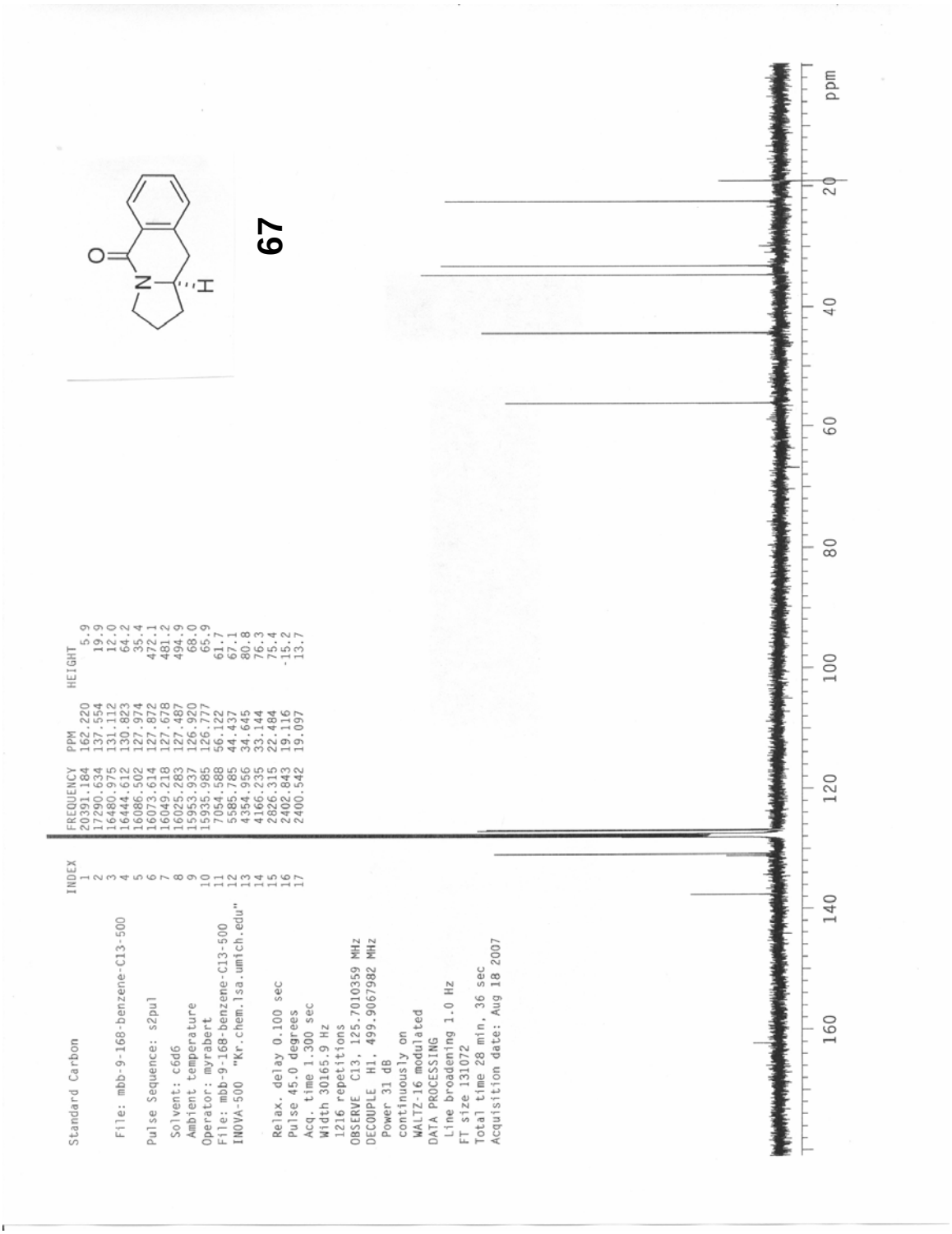
| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 14149.715 | 140.495 | 7.5 |
| 2 | 13909.934 | 138.114 | 3.7 |
| 3 | 13143.880 | 130.508 | 47.8 |
| 4 | 13125.974 | 130.330 | 13.3 |
| 5 | 13122.860 | 130.299 | 26.2 |
| 6 | 12907.213 | 128.158 | 44.6 |
| 7 | 12760.853 | 126.705 | 35.0 |
| 8 | 7787.734 | 77.326 | 105.0 |
| 9 | 7776.835 | 77.218 | 5.5 |
| 10 | 7755.815 | 77.009 | 110.7 |
| 11 | 7723.896 | 76.692 | 109.2 |
| 12 | 6776.450 | 67.285 | 10.3 |
| 13 | 6366.954 | 63.219 | 47.7 |
| 14 | 5781.514 | 57.406 | 33.7 |
| 15 | 4311.688 | 42.812 | 14.2 |
| 16 | 3895.964 | 38.684 | 3.1 |
| 17 | 3878.837 | 38.514 | 2.7 |
| 18 | 3182.849 | 31.603 | 18.6 |
| 19 | 2991.336 | 29.702 | 4.9 |
| 20 | 2411.346 | 23.943 | 29.1 |

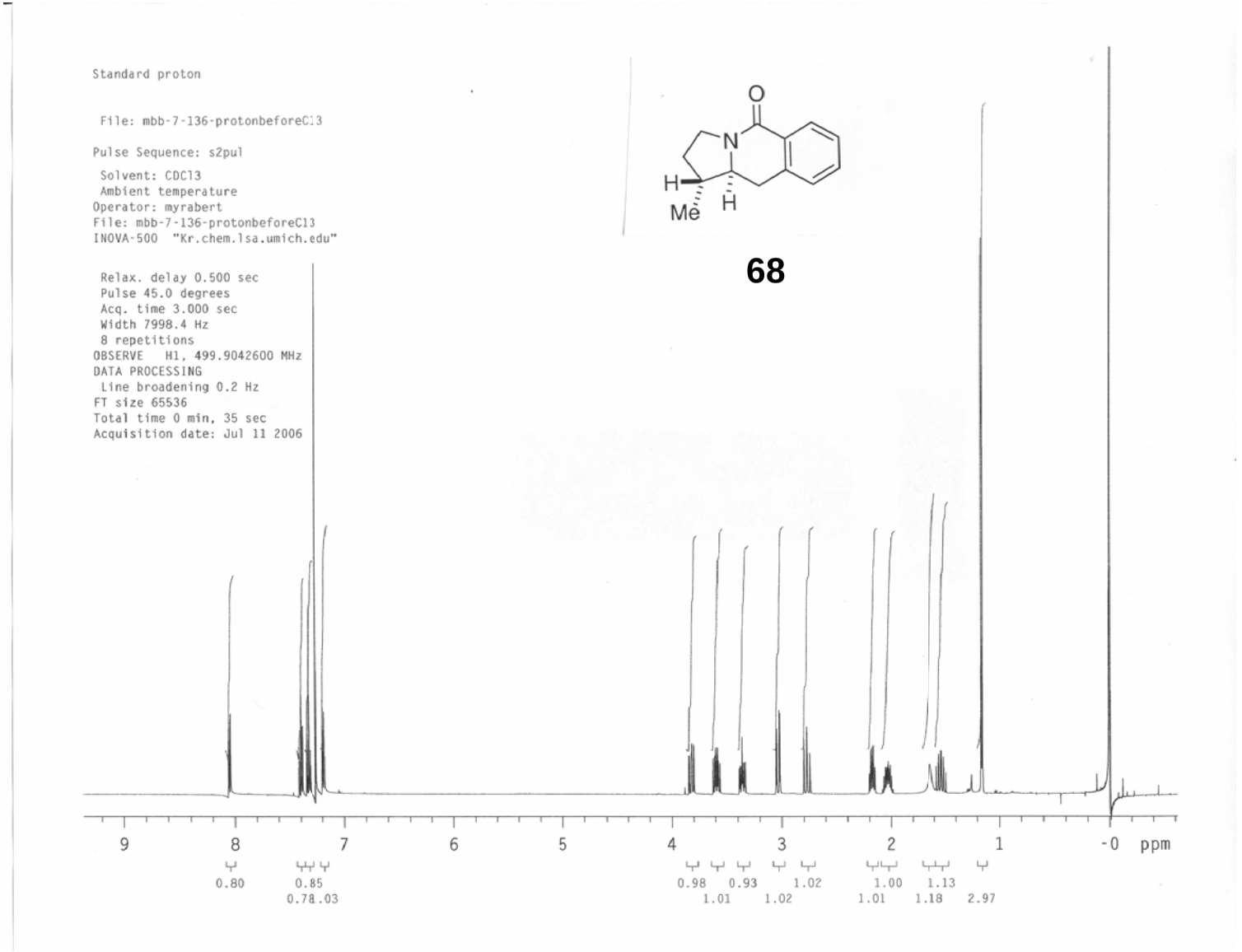



**66**




Standard Proton
File: mbb-9-168-benzene
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
Operator: myrabert
File: mbb-9-168-benzene
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8003.2 Hz
8 repetitions
OBSERVE H1, 499.9042891 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 31 sec
Acquisition date: Aug 18 2007
O
N
H
67
10
9
8
7
6
5
4
3
2
1
ppm
0.99
2.01
1.00
1.19
1.19
1.05
1.20
1.20
1.28 1.67
1.68 1.30


67
Standard Carbon
File: mbb-9-168-benzene-C13-500
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
Operator: myrabert
File: mbb-9-168-benzene-C13-500
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
1216 repetitions
OBSERVE C13, 125.7010359 MHz
DECOUPLE H1, 499.9067982 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 28 min, 36 sec
Acquisition date: Aug 18 2007
INDEX FREQUENCY PPM HEIGHT
1 20391.184 162.220 5.9
2 17290.634 137.554 19.9
3 16480.975 131.112 12.0
4 16444.612 130.823 64.2
5 16086.502 127.974 35.4
6 16073.614 127.872 472.1
7 16049.218 127.678 481.2
8 16025.283 127.487 494.9
9 15953.937 126.920 68.0
10 15935.985 126.777 65.9
11 7054.588 56.122 61.7
12 5585.785 44.437 67.1
13 4354.956 34.645 80.8
14 4166.235 33.144 76.3
15 2826.315 22.484 75.4
16 2402.843 19.116 -15.2
17 2400.542 19.097 13.7
160
140
120
100
80
60
40
20
ppm


Standard proton
File: mbb-7-136-protonbeforeC13
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: myrabert
File: mbb-7-136-protonbeforeC13
INOVA-500 "Kr.chem.lsa.umich.edu"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7998.4 Hz
8 repetitions
OBSERVE H1, 499.9042600 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 35 sec
Acquisition date: Jul 11 2006
O
N
H
Me
H
68
9
8
7
6
5
4
3
2
1
-0
ppm
0.80
0.85
0.78
1.03
0.98
0.93
1.02
1.01
1.02
1.00
1.13
1.01
1.18
2.97

Standard carbon

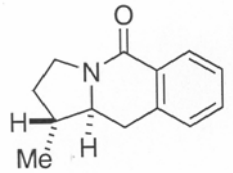
File: MBB-7-136-C13-500

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Operator: myrabert
File: MBB-7-136-C13-500
INOVA-500 "Kr.chem.lsa.umich.edu"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 30165.9 Hz
12144 repetitions
OBSERVE C13, 125.7010266 MHz
DECOUPLE H1, 499.9072532 MHz
Power 31 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 hr, 45 min, 15 sec
Acquisition date: Jul 11 2006

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 20520.314 | 163.247 | 4.9 |
| 2 | 17260.963 | 137.318 | 9.3 |
| 3 | 16522.650 | 131.444 | 48.8 |
| 4 | 16381.339 | 130.320 | 9.0 |
| 5 | 16031.514 | 127.537 | 46.4 |
| 6 | 15983.183 | 127.152 | 42.9 |
| 7 | 15968.914 | 127.039 | 42.0 |
| 8 | 9711.200 | 77.256 | 144.2 |
| 9 | 9678.979 | 77.000 | 151.2 |
| 10 | 9647.219 | 76.747 | 148.3 |
| 11 | 7911.905 | 62.942 | 41.6 |
| 12 | 5556.574 | 44.205 | 46.5 |
| 13 | 5232.066 | 41.623 | 42.6 |
| 14 | 4216.194 | 33.541 | 50.9 |
| 15 | 3981.444 | 31.674 | 47.8 |
| 16 | 1997.571 | 15.891 | 49.3 |
| 17 | 1995.270 | 15.873 | -5.3 |
| 18 | -2.412 | -0.019 | 79.3 |




**68**

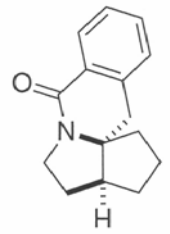
STANDARD PROTON PARAMETERS

File: mbb-10-124-HNMR-400

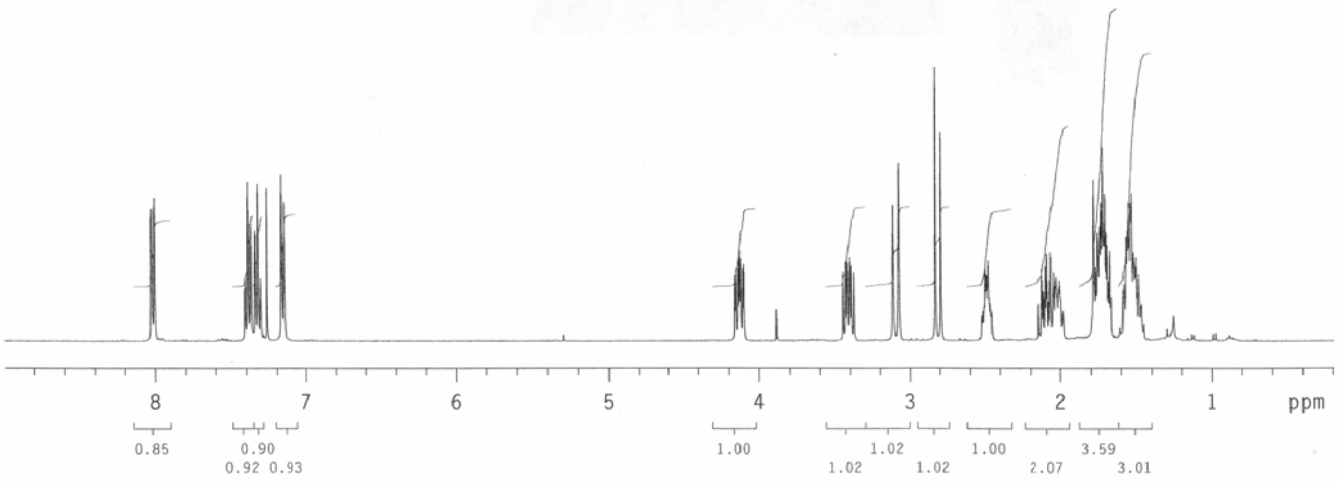
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 10.0 C / 283.1 K
Operator: myrabert
File: mbb-10-124-HNMR-400
VNMRS-401 "Kr.chem.lsa.umich.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.5297559 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec
Acquisition date: Apr 18 2008




**69**



STANDARD CARBON PARAMETERS

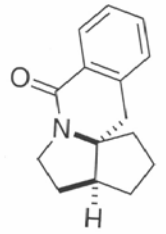
File: mbb-10-124-C13-400

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 10.0 C / 283.1 K
Operator: myrabert
File: mbb-10-124-C13-400
VNMRS-401 "Kr.chem.lsa.umich.edu"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
112 repetitions
OBSERVE C13, 100.7132888 MHz
DECOUPLE H1, 400.5317616 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 4 min, 16 sec
Acquisition date: Apr 18 2008

| INDEX | FREQUENCY | PPM | HEIGHT |
|---|---|---|---|
| 1 | 16332.498 | 162.168 | 9.2 |
| 2 | 13821.804 | 137.239 | 12.8 |
| 3 | 13230.137 | 131.364 | 31.9 |
| 4 | 13068.985 | 129.764 | 12.5 |
| 5 | 12872.801 | 127.816 | 37.2 |
| 6 | 12850.224 | 127.592 | 34.7 |
| 7 | 12790.279 | 126.997 | 33.2 |
| 8 | 7787.576 | 77.324 | 139.0 |
| 9 | 7755.657 | 77.007 | 143.0 |
| 10 | 7723.738 | 76.690 | 140.2 |
| 11 | 7261.303 | 72.099 | 21.2 |
| 12 | 5027.759 | 49.922 | 32.5 |
| 13 | 4447.769 | 44.163 | 27.8 |
| 14 | 4014.139 | 39.857 | 33.6 |
| 15 | 3826.519 | 37.994 | 38.6 |
| 16 | 3280.783 | 32.575 | 30.6 |
| 17 | 3065.915 | 30.442 | 32.0 |
| 18 | 2562.219 | 25.441 | 37.0 |




**69**