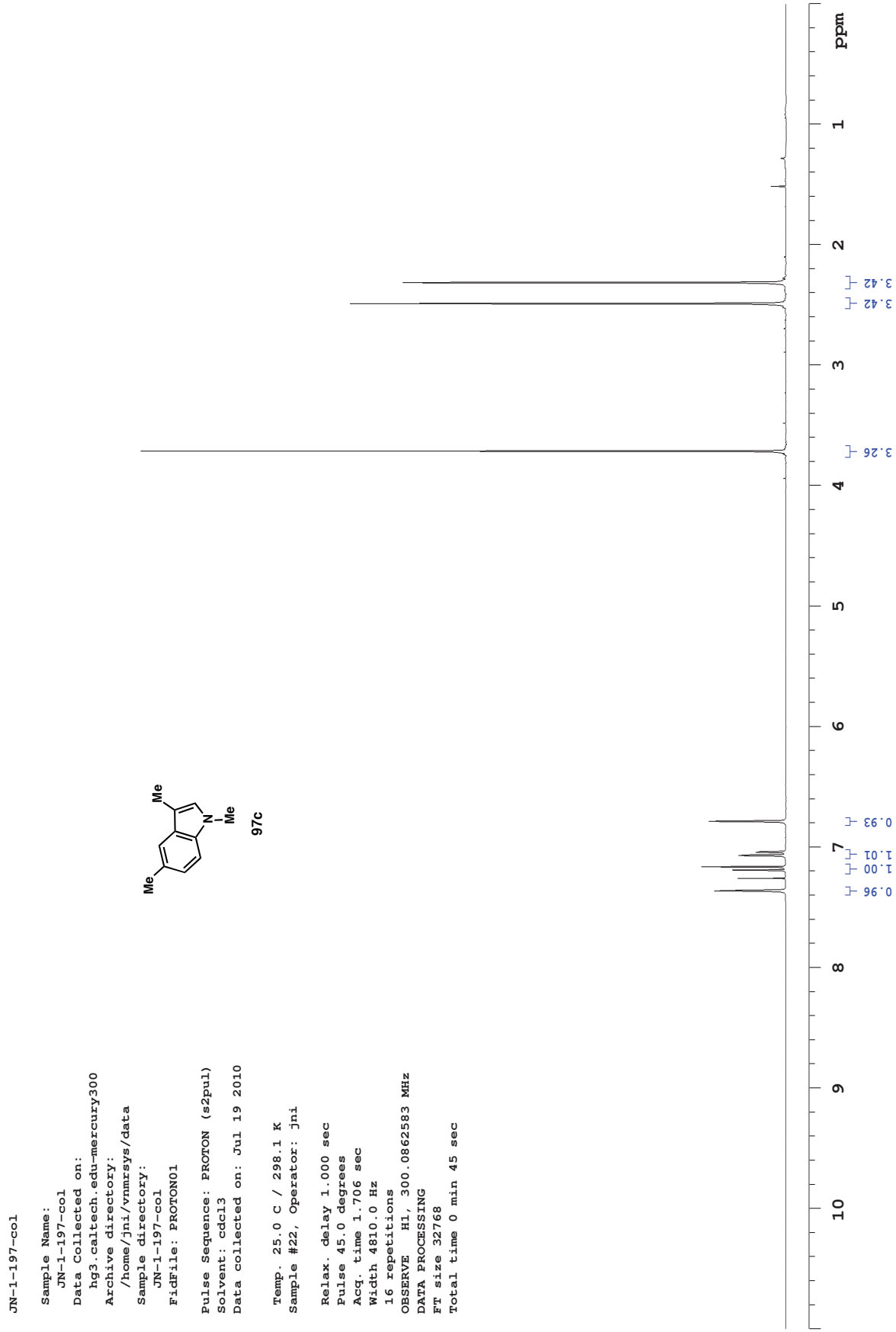
# *APPENDIX 1*

*Spectra Relevant to Chapter 2:*

*Enantioselective Synthesis of Pyrroloindolines by a Formal (3 + 2) Cycloaddition*


JN-1-197-col
Sample Name:
JN-1-197-col
Data Collected on:
hg3.caltech.edu-mercury300
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-197-col
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010
Temp. 25.0 C / 298.1 K
Sample #22, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862583 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 45 sec
Me
Me
N
Me
97c
ppm
10
9
8
7
6
5
4
3
2
1
0.96
1.00
1.01
0.93
3.26
3.42
3.42

JN-1-197

Sample Name:
JN-1-197
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-197
FidFile: CARBON01

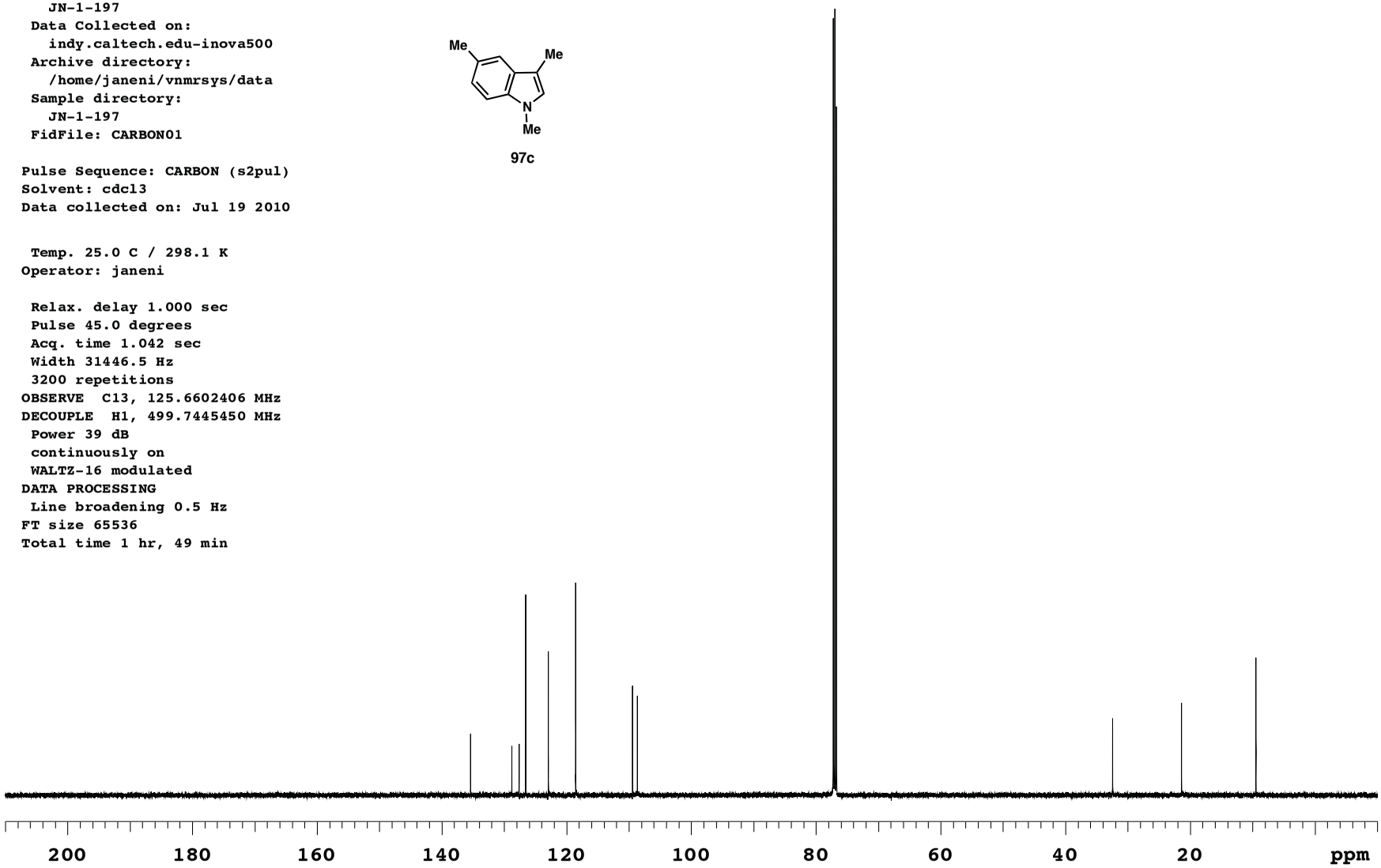
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3200 repetitions
OBSERVE C13, 125.6602406 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 49 min




97c



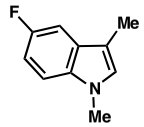
JN-1-183-col2

Sample Name:
JN-1-183-col2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-183-col2
FidFile: PROTON01

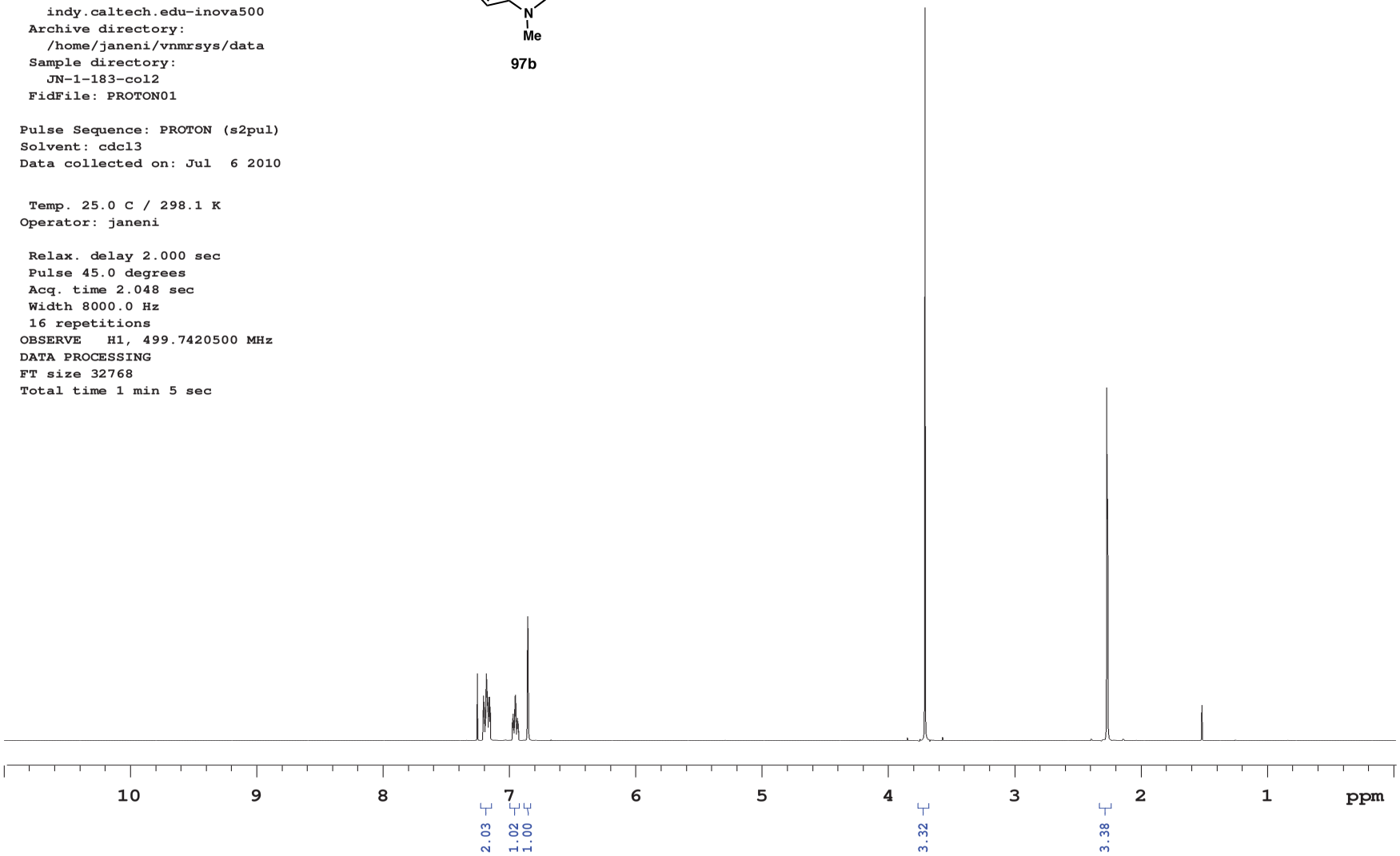
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 6 2010

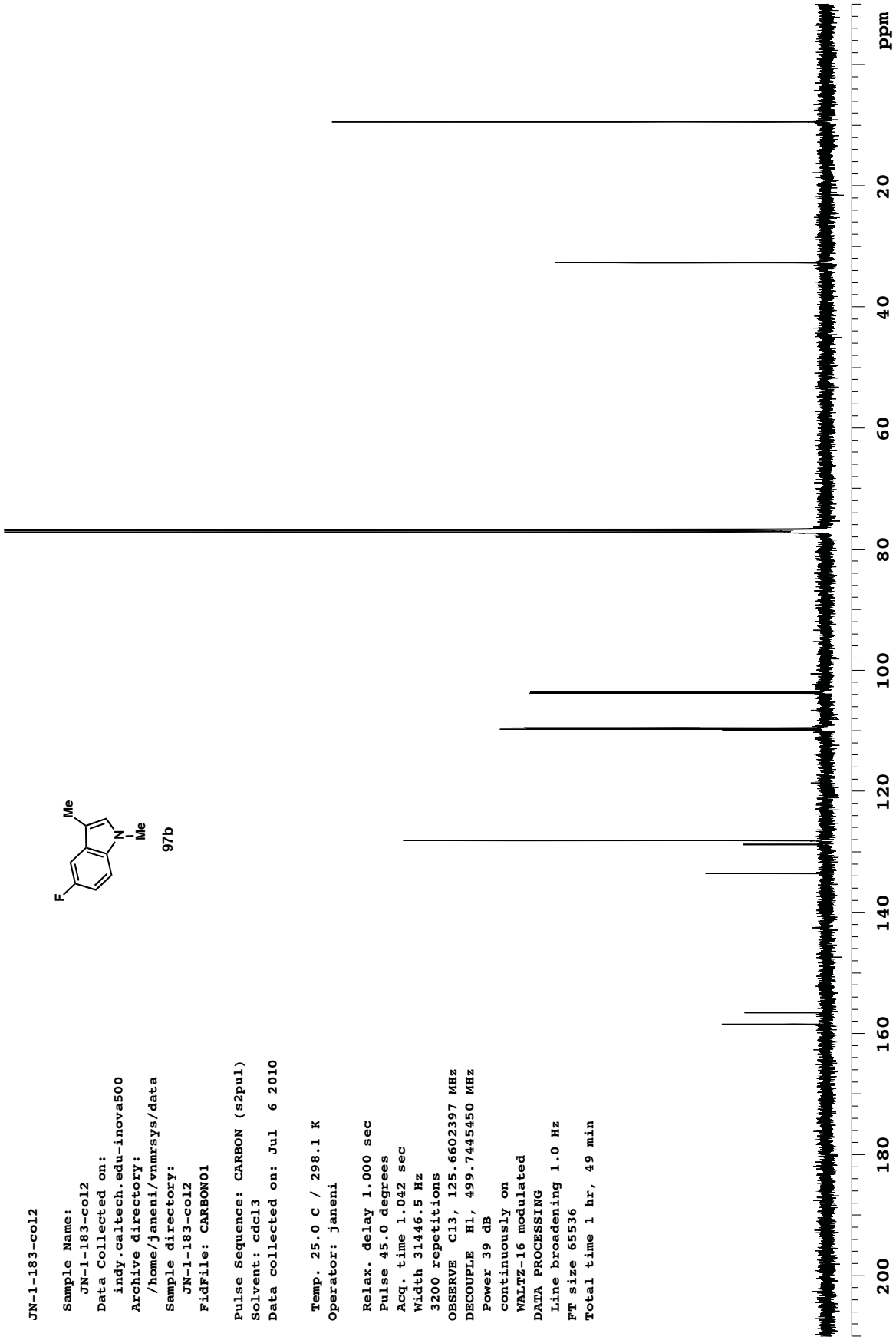
Temp. 25.0 C / 298.1 K
Operator: janeni

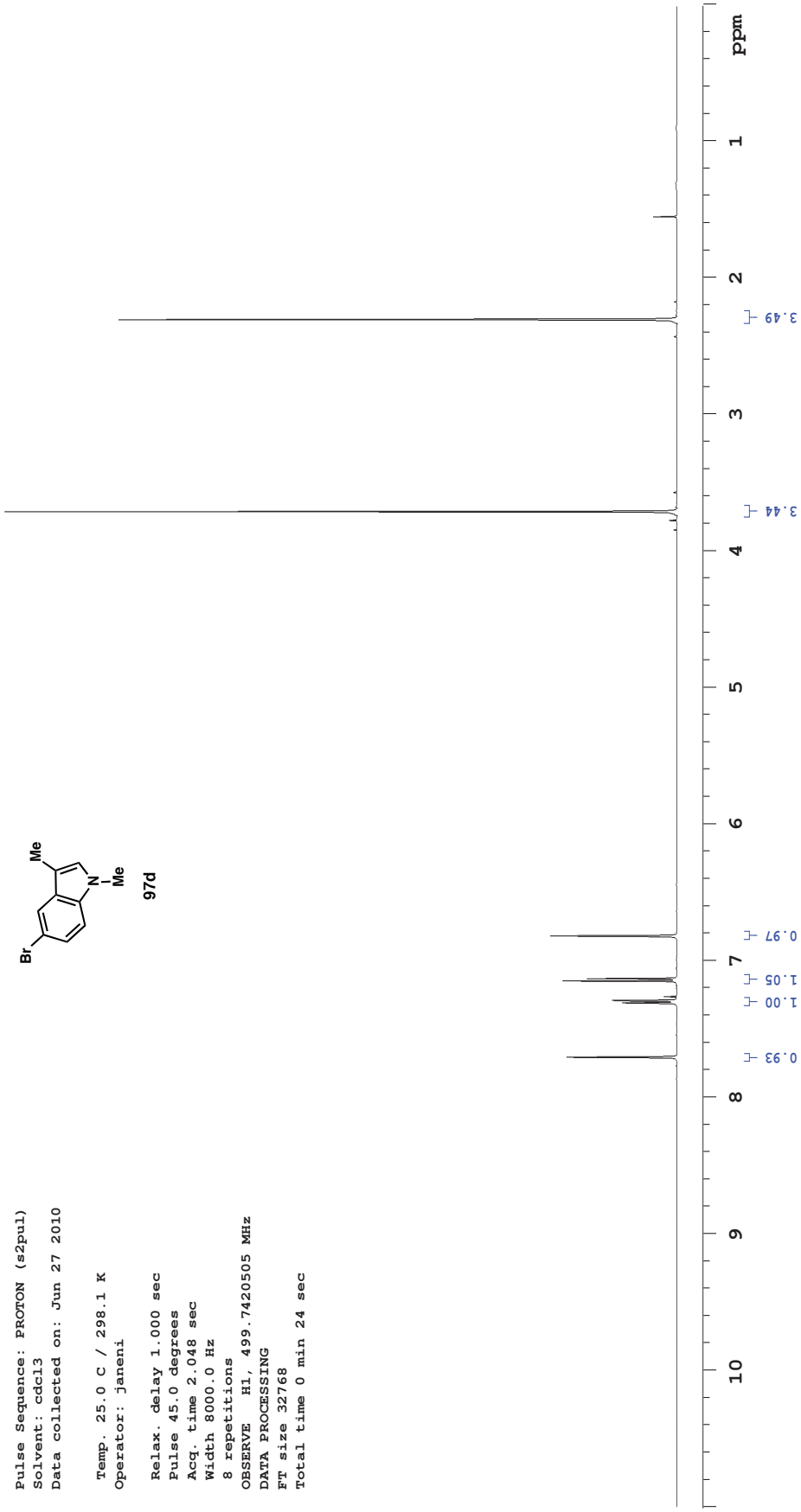
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420500 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 5 sec



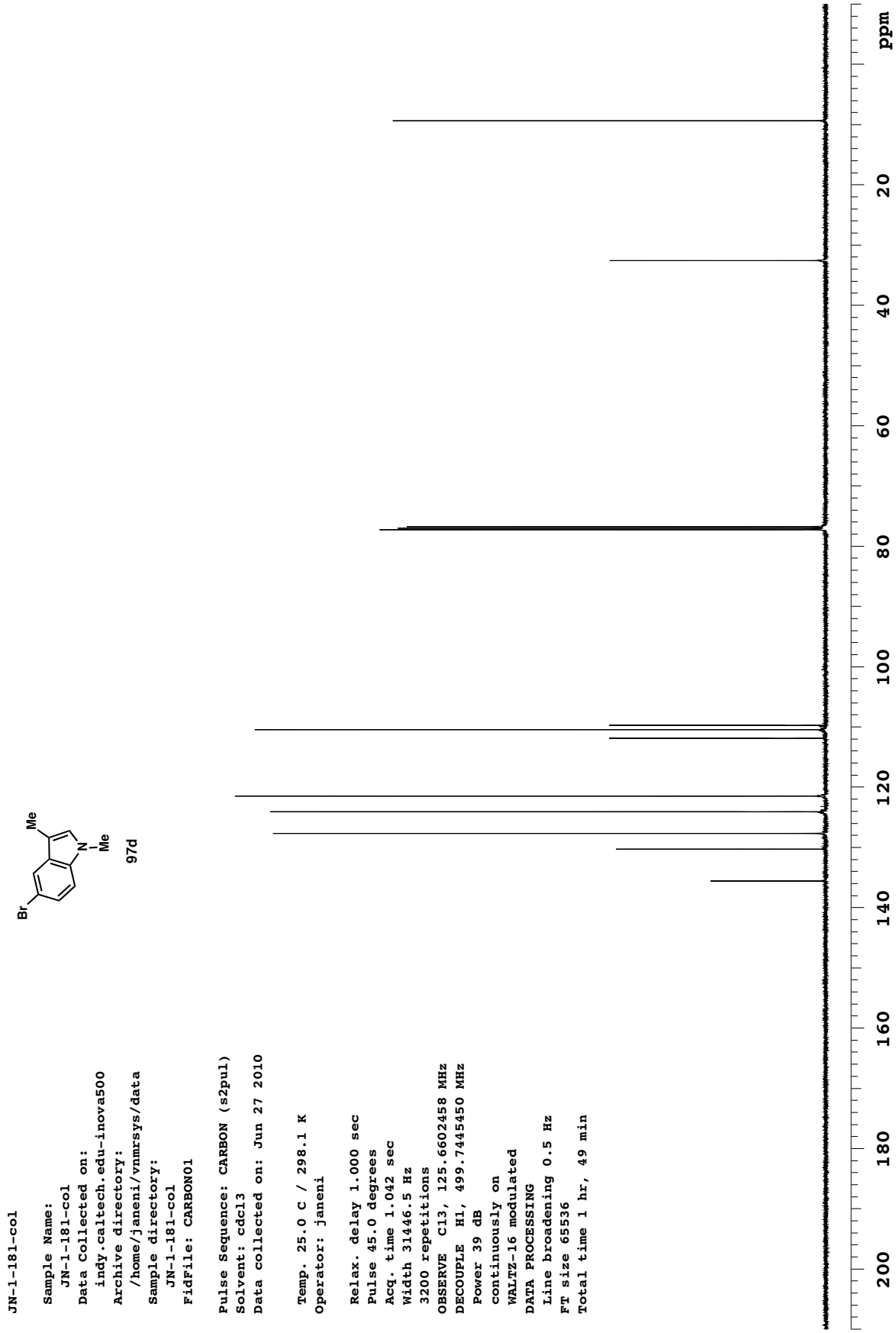

97b




JN-1-183-col2
Sample Name:
JN-1-183-col2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-183-col2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 6 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3200 repetitions
OBSERVE C13, 125.6602397 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 49 min
Me
N
Me
F
97b
ppm
200
180
160
140
120
100
80
60
40
20




```
JN-1-181-col

  Sample Name:
     JN-1-181-col
  Data Collected on:
     indy.caltech.edu-inova500
  Archive directory:
     /home/janeni/vnmrsys/data
  Sample directory:
     JN-1-181-col
  FidFile: PROTON01

  Pulse Sequence: PROTON (s2pul)
  Solvent: cdcl3
  Data collected on: Jun 27 2010

   Temp. 25.0 C / 298.1 K
  Operator: janeni

   Relax. delay 1.000 sec
   Pulse 45.0 degrees
   Acq. time 2.048 sec
   Width 8000.0 Hz
   8 repetitions
  OBSERVE   H1, 499.7420505 MHz
  DATA PROCESSING
  FT size 32768
  Total time 0 min 24 sec
```


JN-1-181-col
Sample Name:
JN-1-181-col
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-181-col
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 27 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
width 31446.5 Hz
3200 repetitions
OBSERVE C13, 125.6602458 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 49 min
Br
Me
N
Me
97d
ppm
200
180
160
140
120
100
80
60
40
20

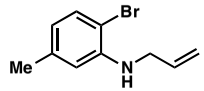
JN-1-199-col

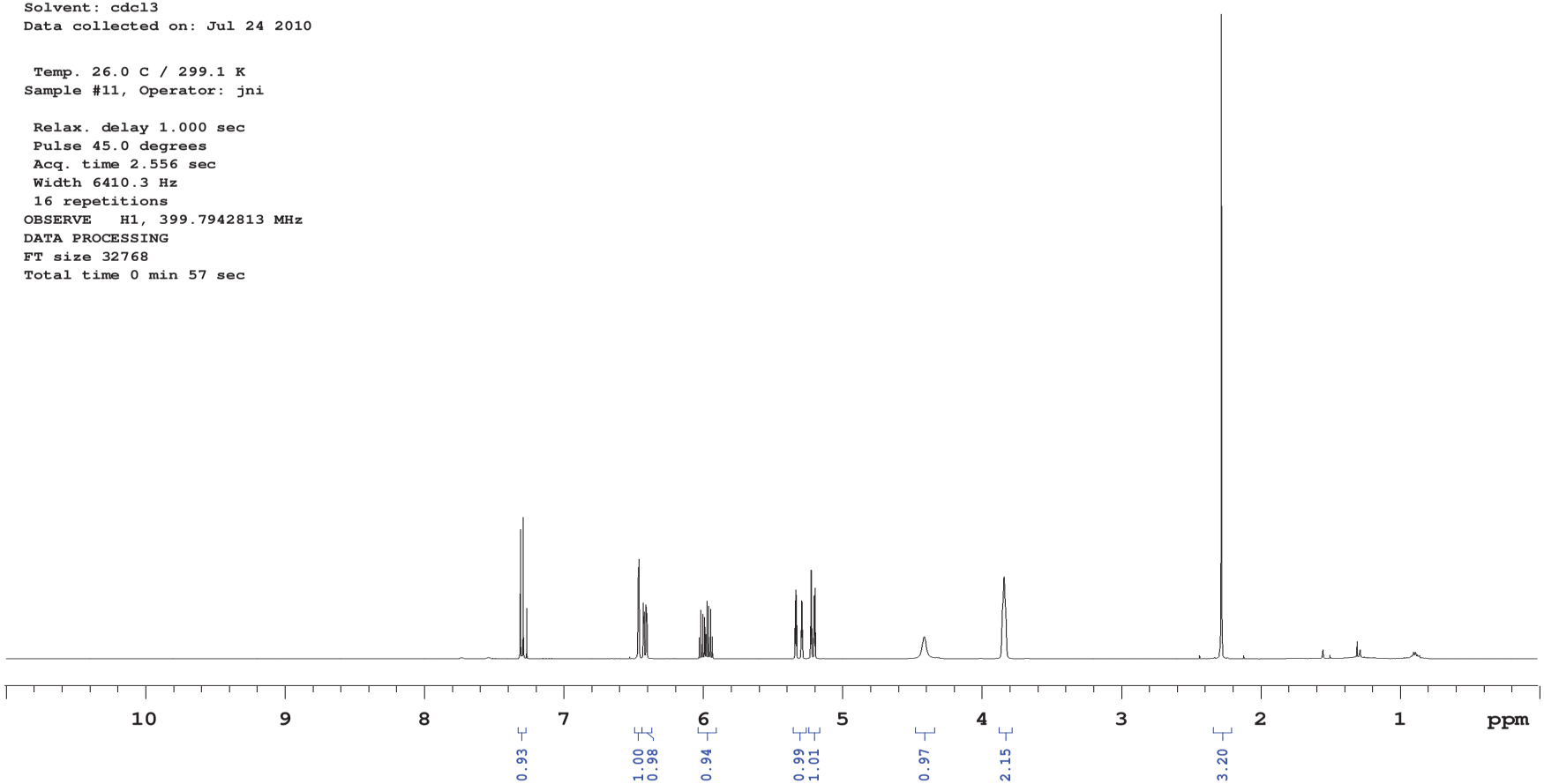
Sample Name:
JN-1-199-col
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-199-col
FidFile: PROTON01

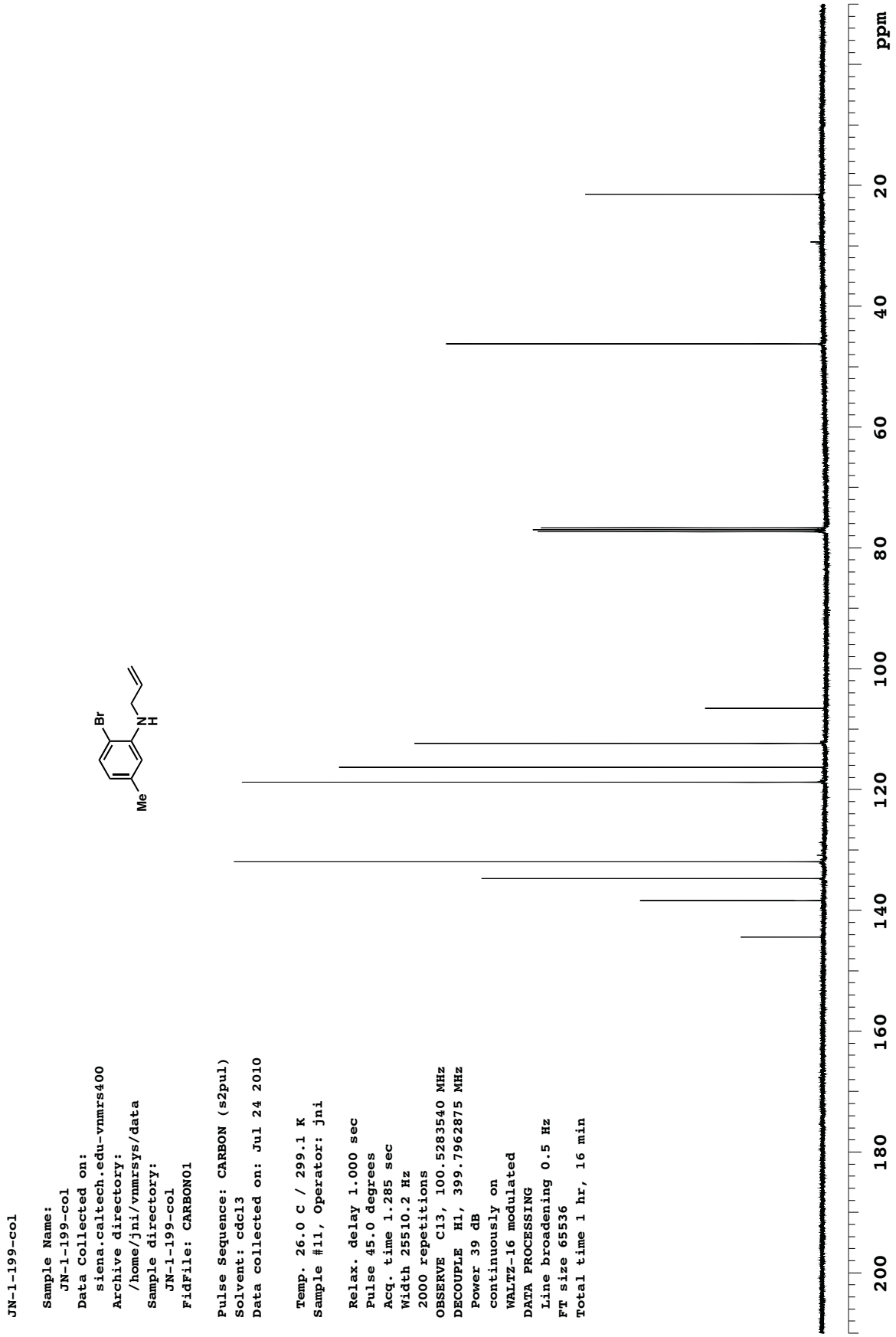
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010

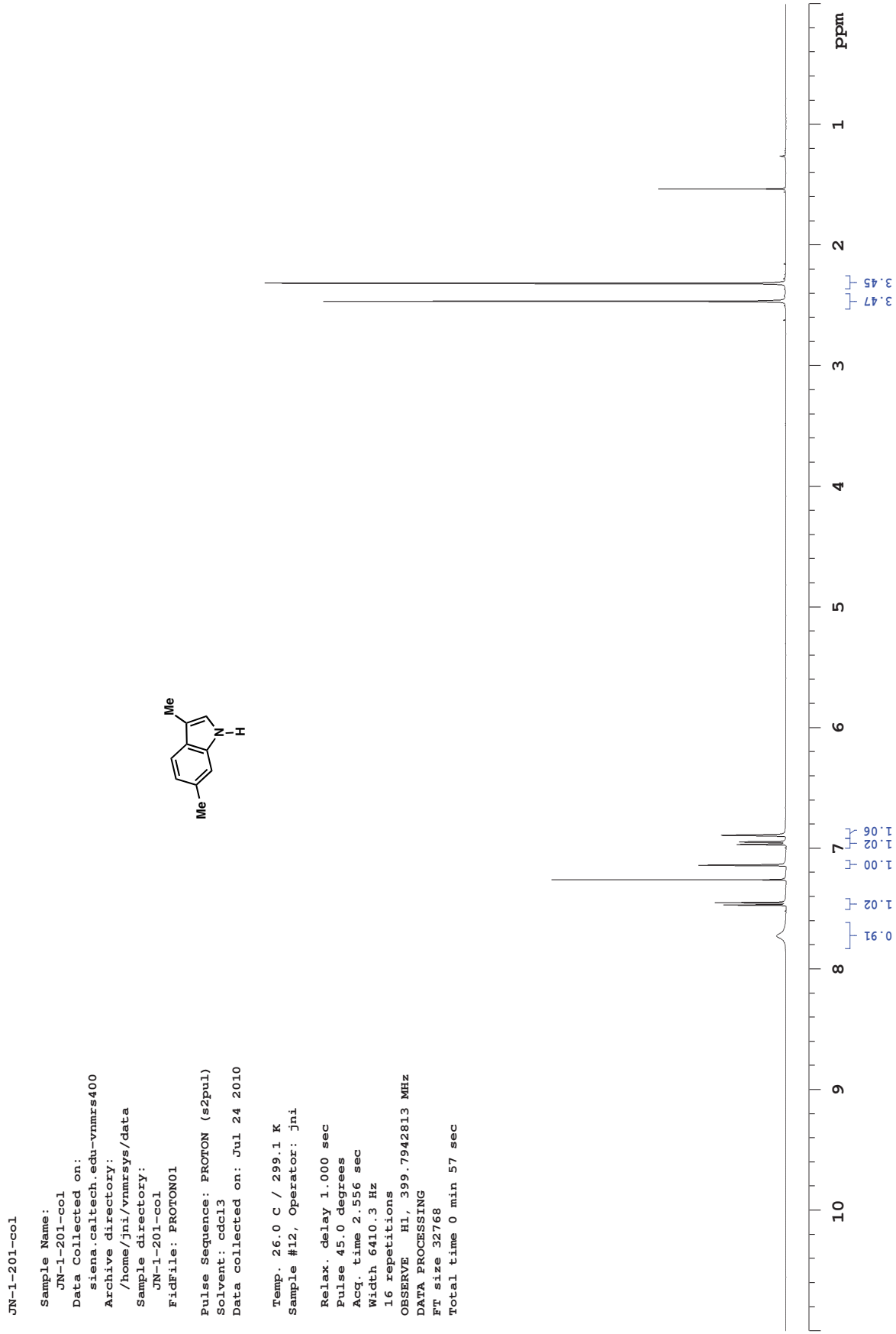
Temp. 26.0 C / 299.1 K
Sample #11, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 57 sec




JN-1-199-col
Sample Name:
JN-1-199-col
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-199-col
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010
Temp. 26.0 C / 299.1 K
Sample #11, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
2000 repetitions
OBSERVE C13, 100.5283540 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min
Br
N
H
Me
ppm
200
180
160
140
120
100
80
60
40
20


JN-1-201-col
Sample Name:
JN-1-201-col
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-201-col
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010
Temp. 26.0 C / 299.1 K
Sample #12, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 57 sec
Me
N
H
Me
ppm
1
2
3
4
5
6
7
8
9
10
3.45
3.47
1.06
1.02
1.00
1.02
0.91

JN-1-201-col

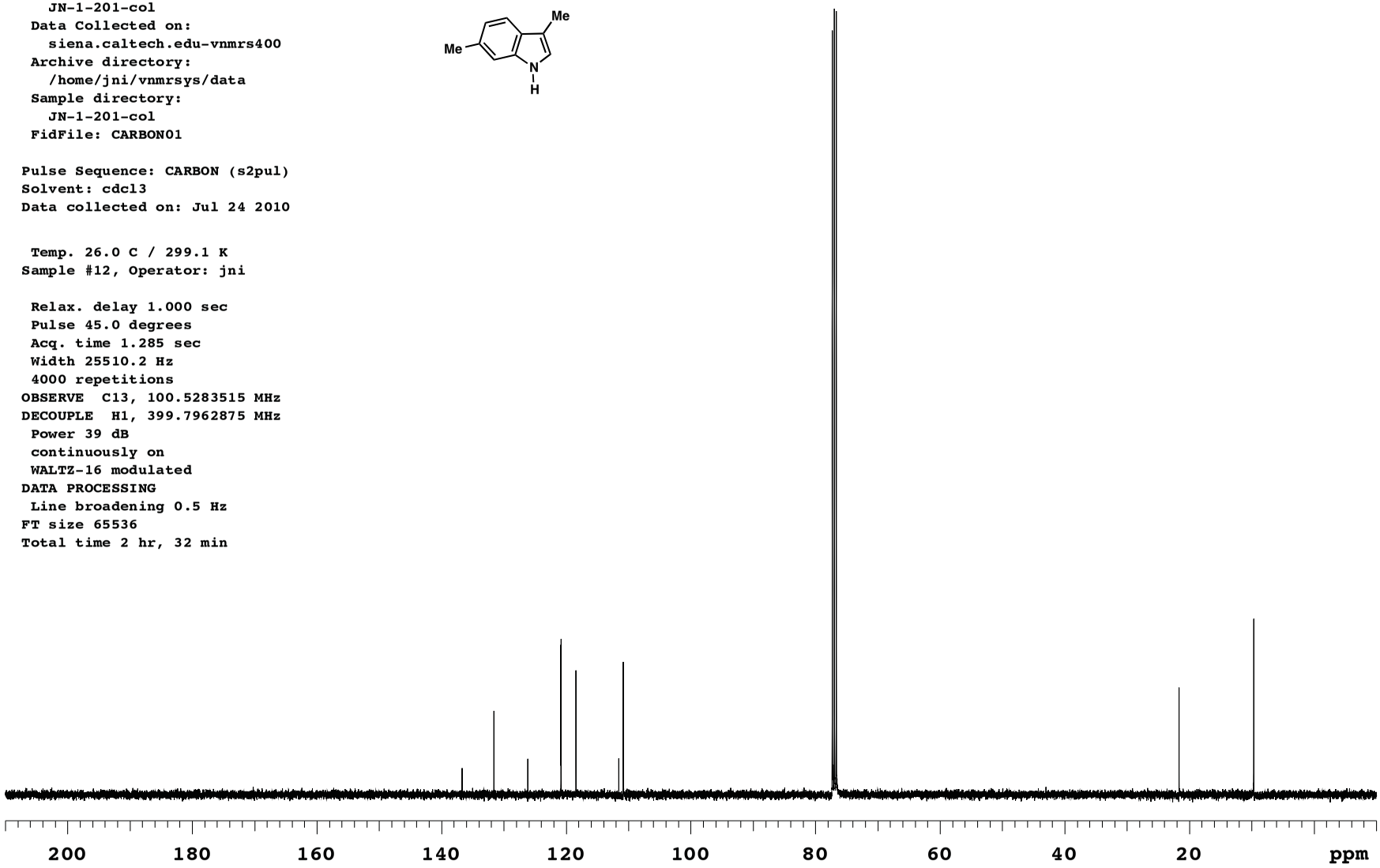
Sample Name:
JN-1-201-col
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-201-col
FidFile: CARBON01

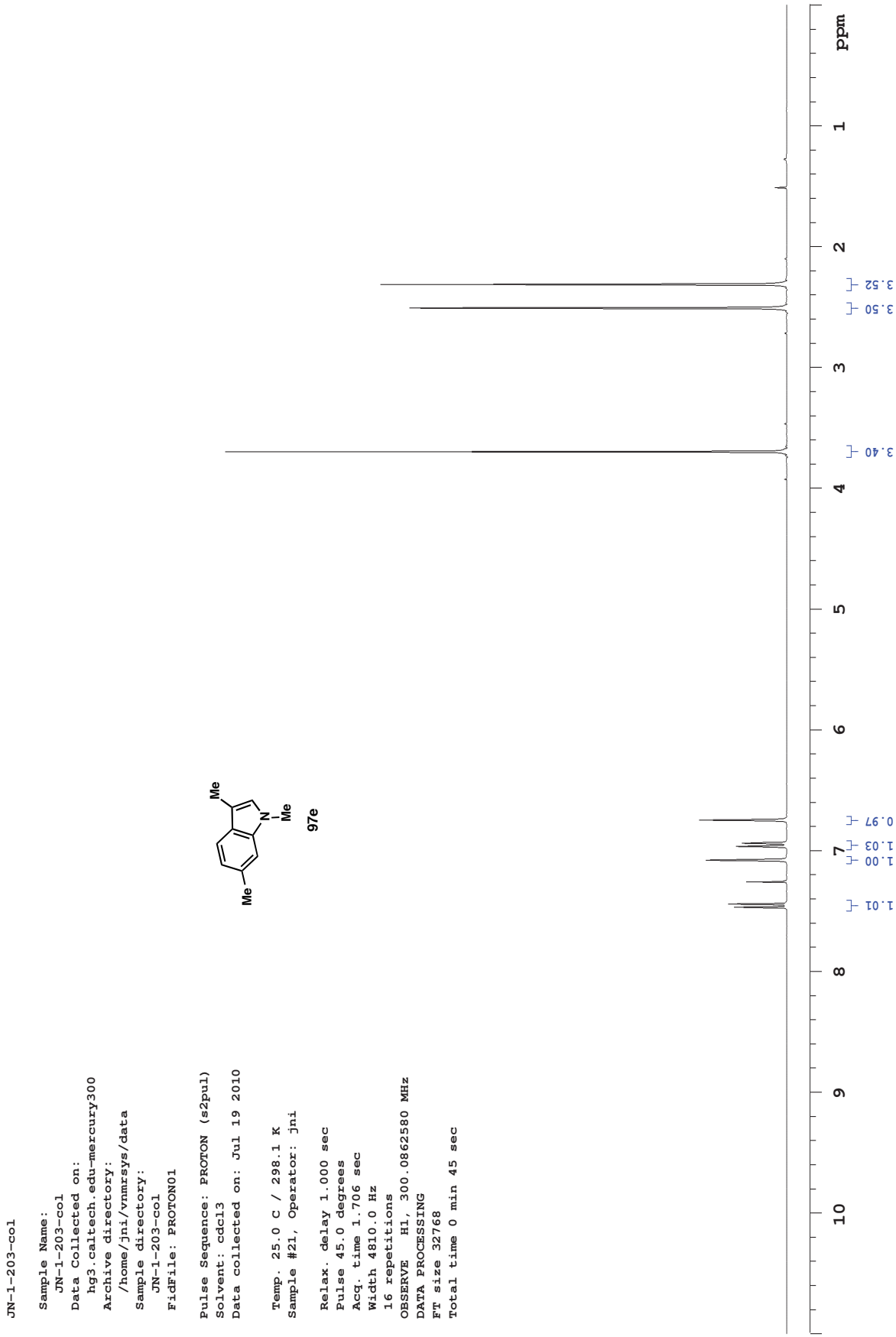
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2010

Temp. 26.0 C / 299.1 K
Sample #12, Operator: jni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
4000 repetitions
OBSERVE C13, 100.5283515 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 hr, 32 min




JN-1-203-col
Sample Name:
JN-1-203-col
Data Collected on:
hg3.caltech.edu-mercury300
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-203-col
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010
Temp. 25.0 C / 298.1 K
Sample #21, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862580 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 45 sec
Me
N
Me
Me
97e
ppm
10
9
8
7
6
5
4
3
2
1
1.01
1.00
1.03
0.97
3.40
3.50
3.52

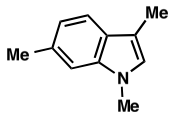
JN-1-203

Sample Name:
JN-1-203
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-203
FidFile: CARBON01

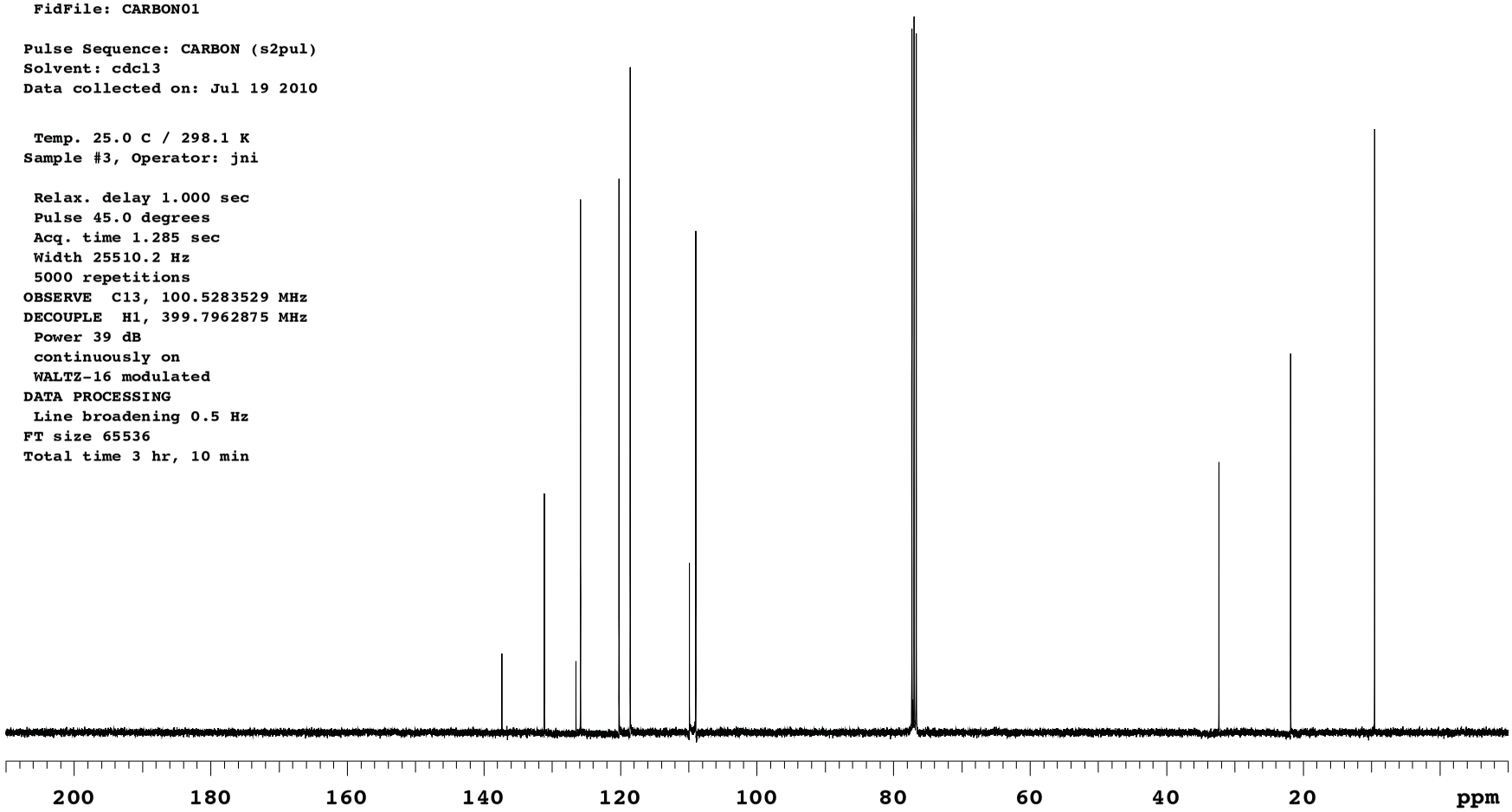
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

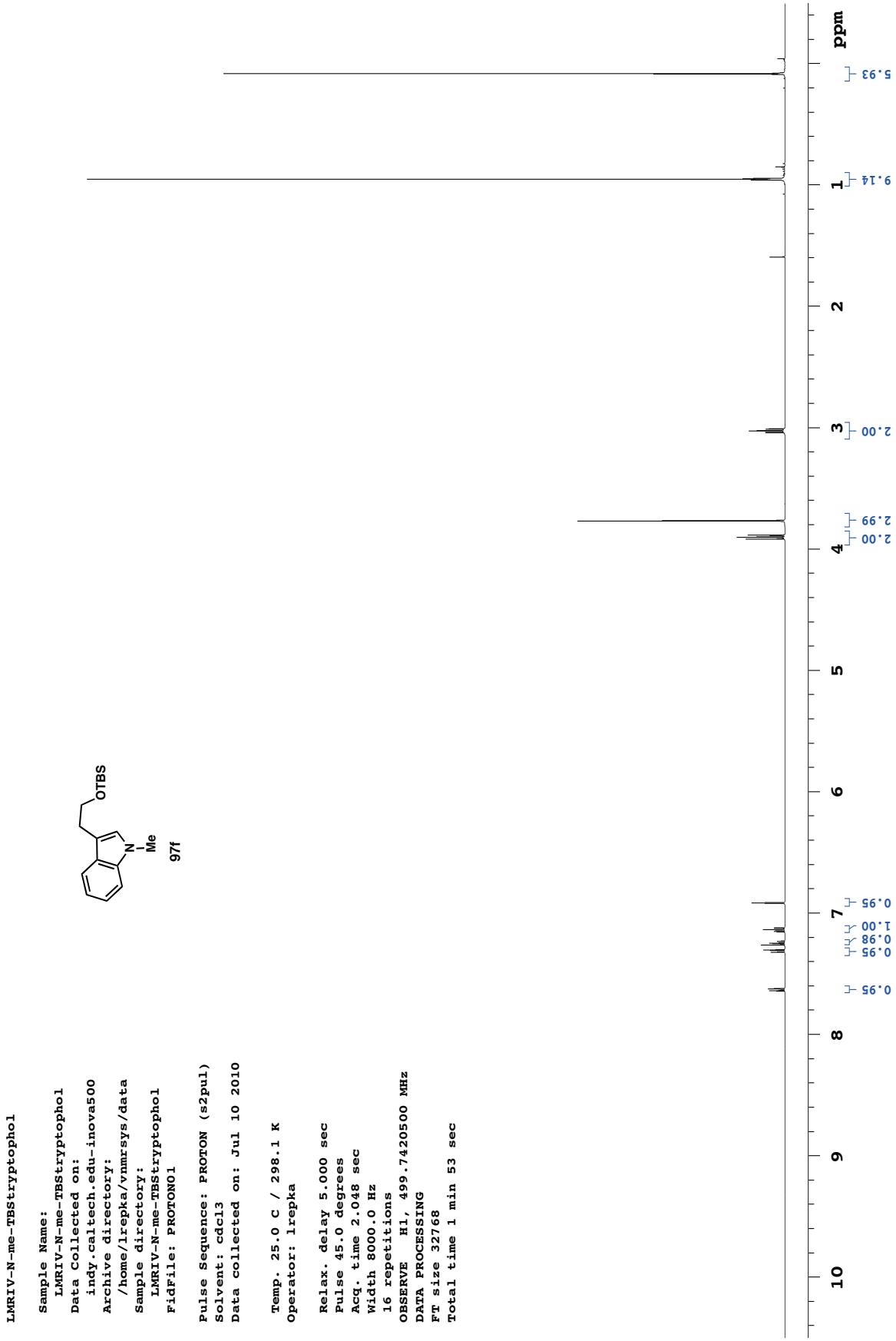
Temp. 25.0 C / 298.1 K
Sample #3, Operator: jni

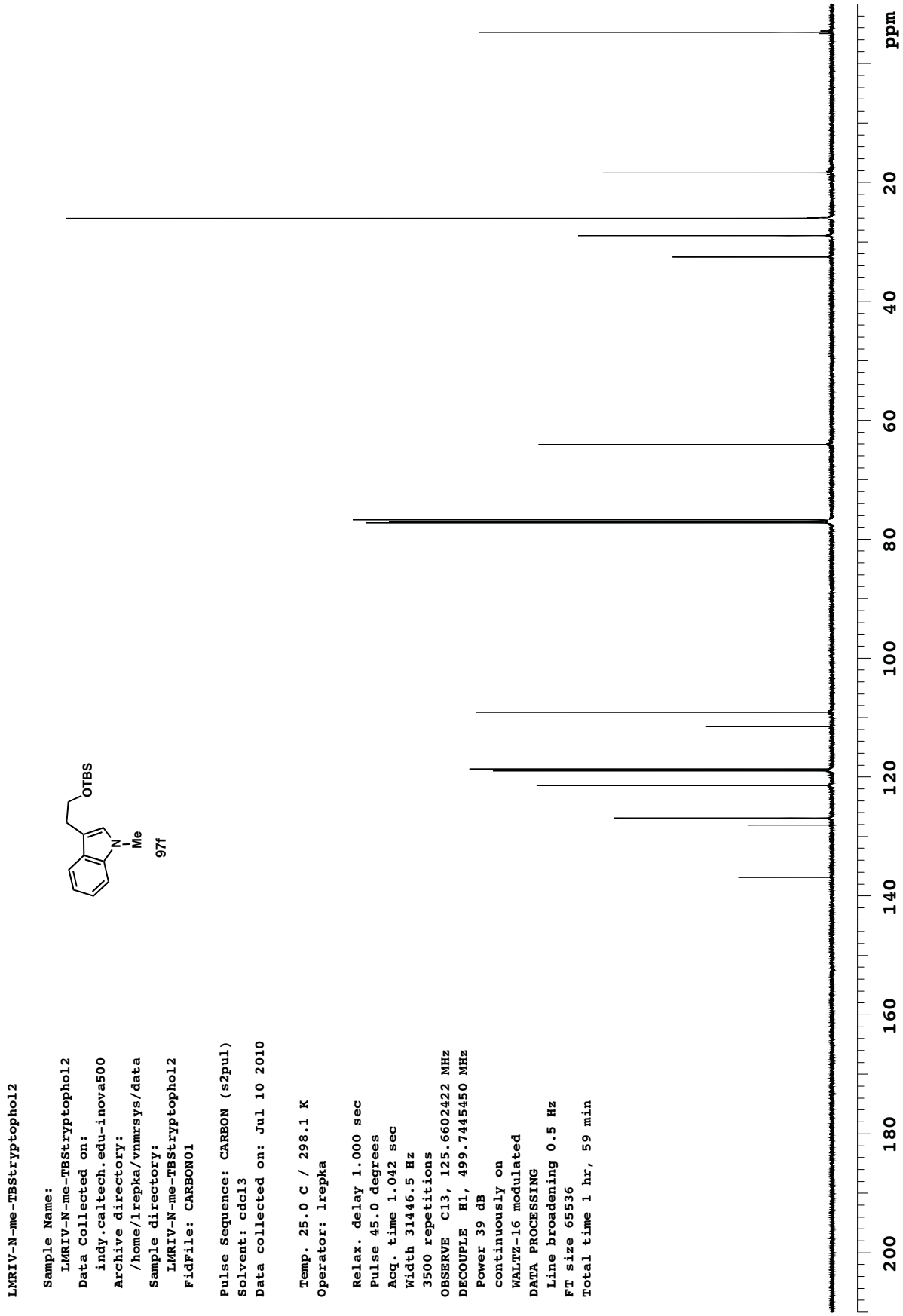
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
5000 repetitions
OBSERVE C13, 100.5283529 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 10 min



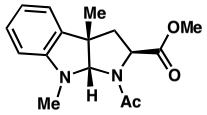

97e



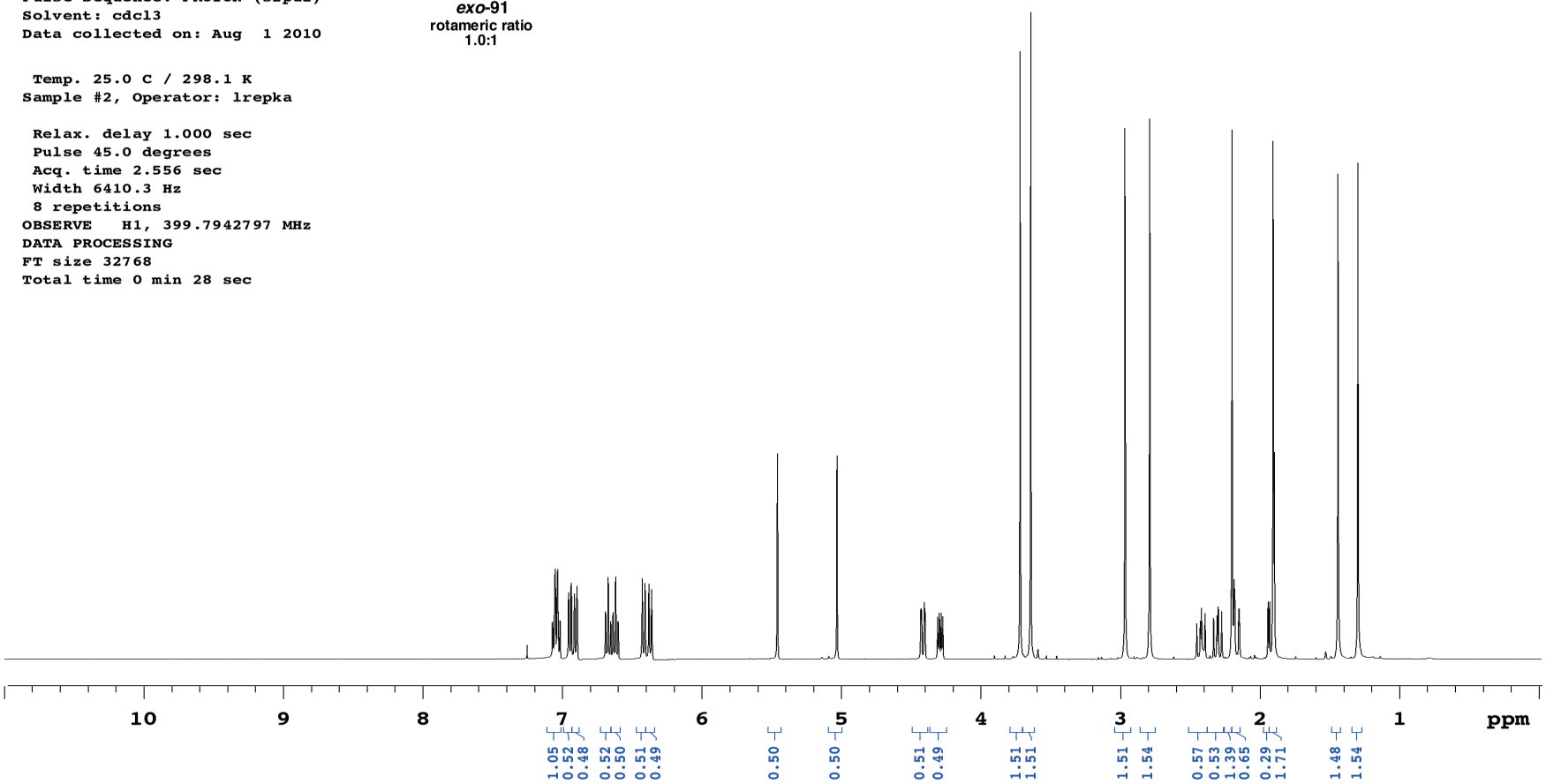

LMRIV-N-me-TBStryptophol
Sample Name:
LMRIV-N-me-TBStryptophol
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-N-me-TBStryptophol
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2010
Temp. 25.0 C / 298.1 K
Operator: lrepka
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420500 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 53 sec
OTBS
N
Me
97f
ppm
10
9
8
7
6
5
4
3
2
1
5.93
9.14
2.00
2.99
2.00
0.95
1.00
0.98
0.95
0.95

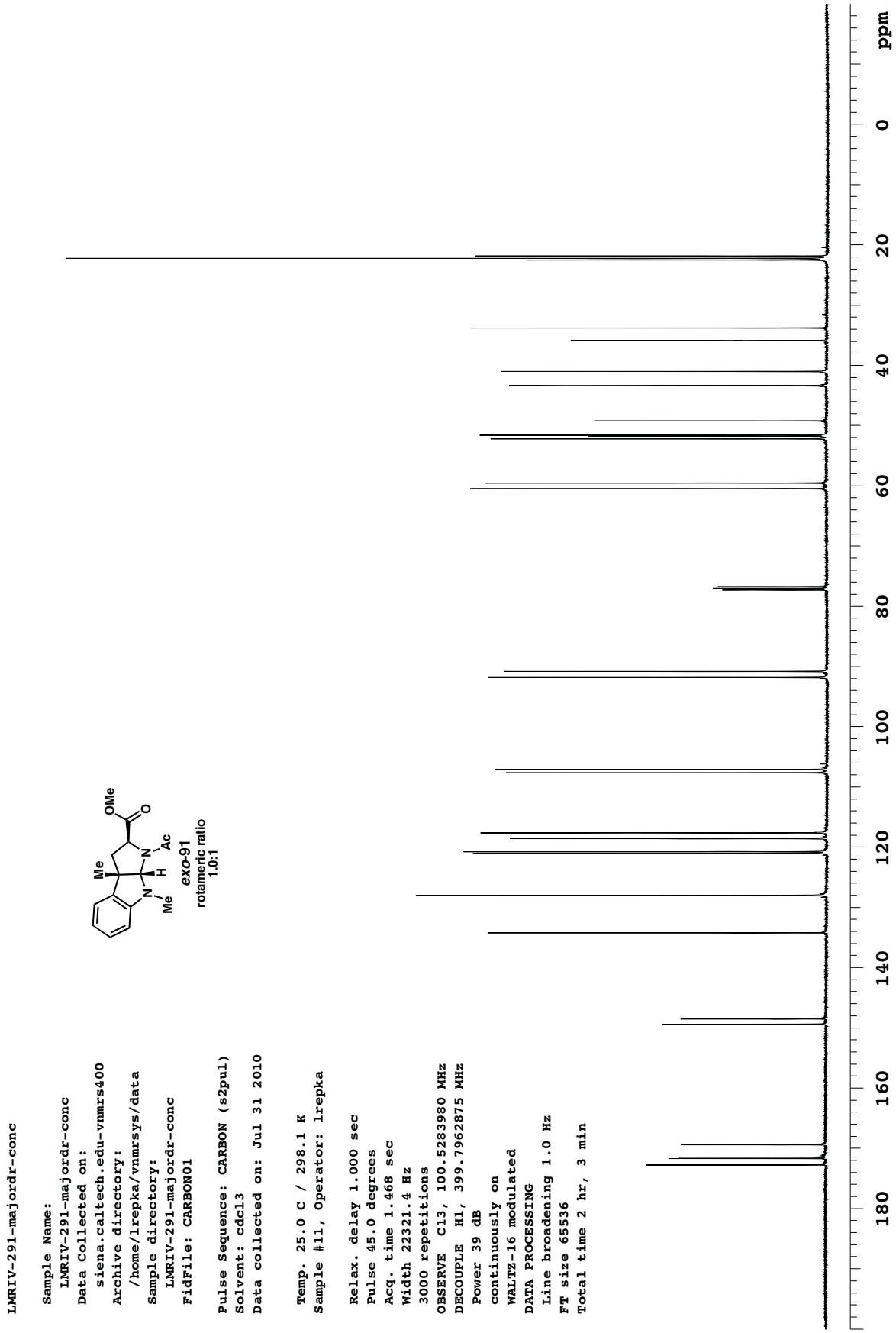

LMRIV-N-me-TBStryptophol2
Sample Name:
LMRIV-N-me-TBStryptophol2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-N-me-TBStryptophol2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2010
Temp. 25.0 C / 298.1 K
Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3500 repetitions
OBSERVE C13, 125.6602422 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 59 min
OTBS
N
Me
97f
ppm
200
180
160
140
120
100
80
60
40
20

```
LMRIV-291-majordr-hsqc

 Sample Name:
   LMRIV-291-majordr-hsqc
 Data Collected on:
   siena.caltech.edu-vnmrs400
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-291-majordr-hsqc
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug  1 2010

 Temp. 25.0 C / 298.1 K
Sample #2, Operator: lrepka

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.556 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE   H1, 399.7942797 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
```




*exo*-91
rotameric ratio
1.0:1




LMRIV-291-majordr-conc
Sample Name:
LMRIV-291-majordr-conc
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-291-majordr-conc
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2010
Temp. 25.0 C / 298.1 K
Sample #11, Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.468 sec
width 22321.4 Hz
3000 repetitions
OBSERVE C13, 100.5283980 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 2 hr, 3 min
OMe
O
Me
N
Ac
H
N
Me
exo-91
rotameric ratio
1.0:1
ppm
0
20
40
60
80
100
120
140
160
180

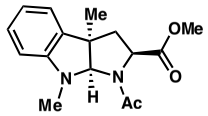
LMRIV-291-minordr-hsqc

Sample Name:
LMRIV-291-minordr-hsqc
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-291-minordr-hsqc
FidFile: PROTON01

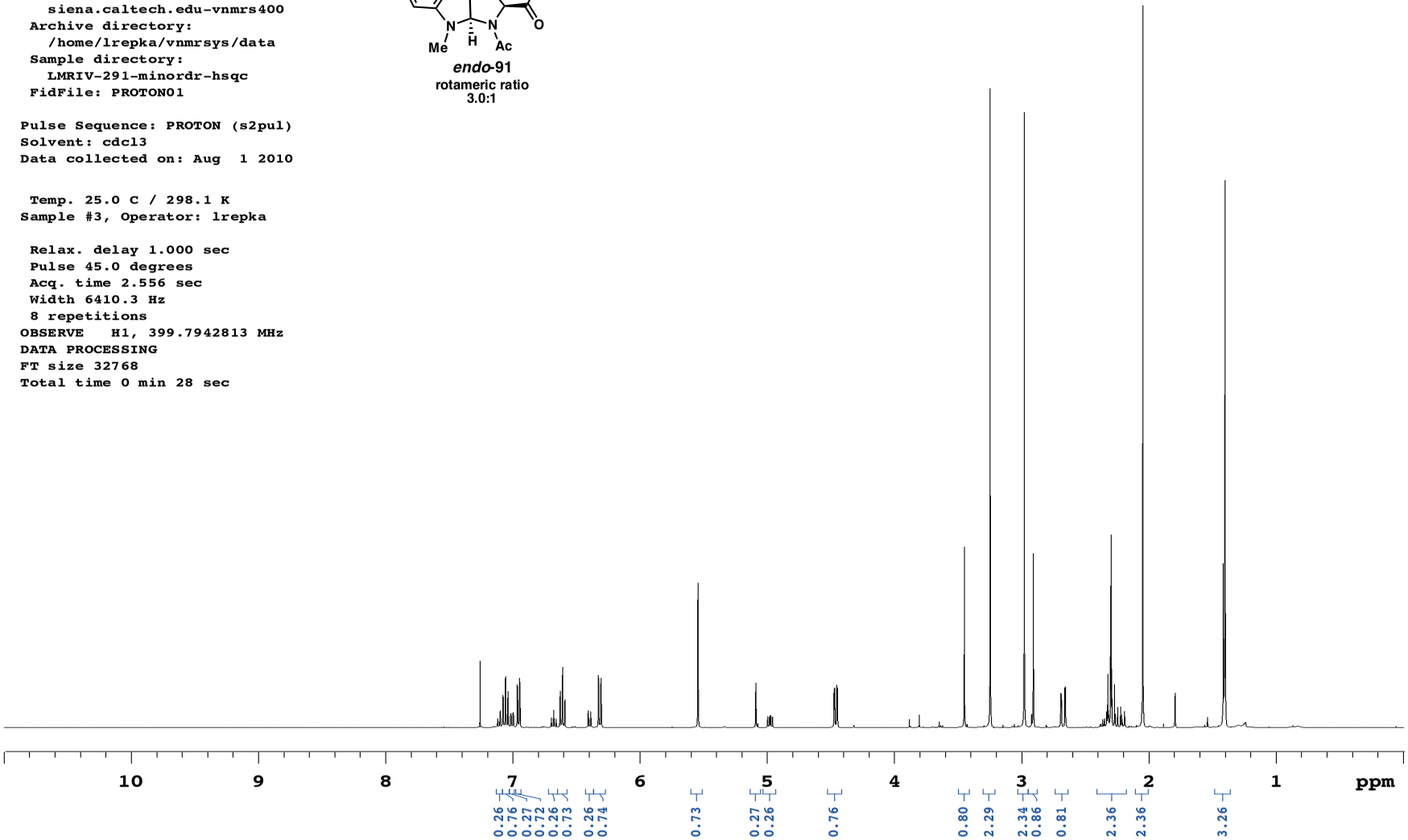
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 1 2010

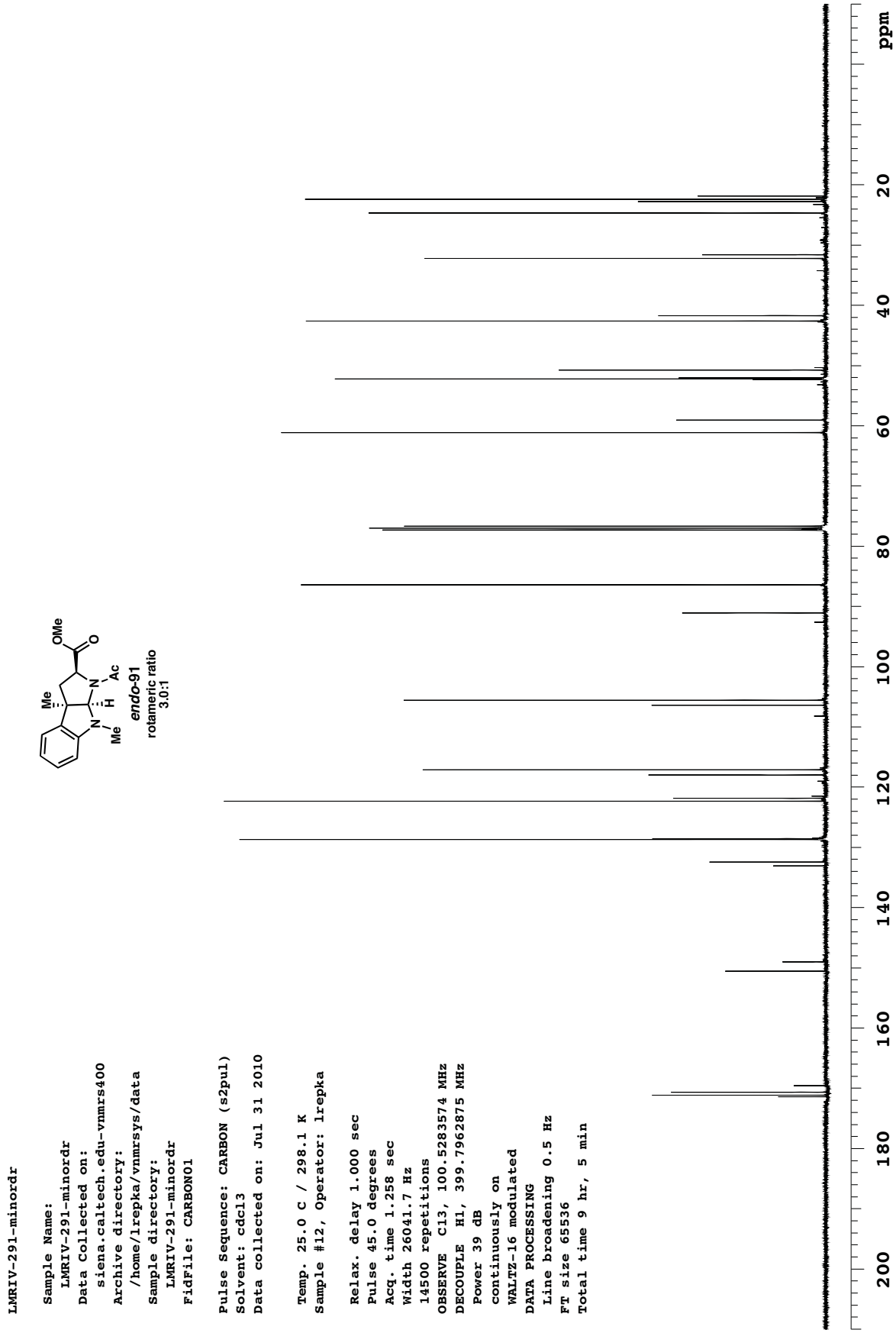
Temp. 25.0 C / 298.1 K
Sample #3, Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec

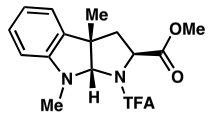



*endo*-91
rotameric ratio
3.0:1



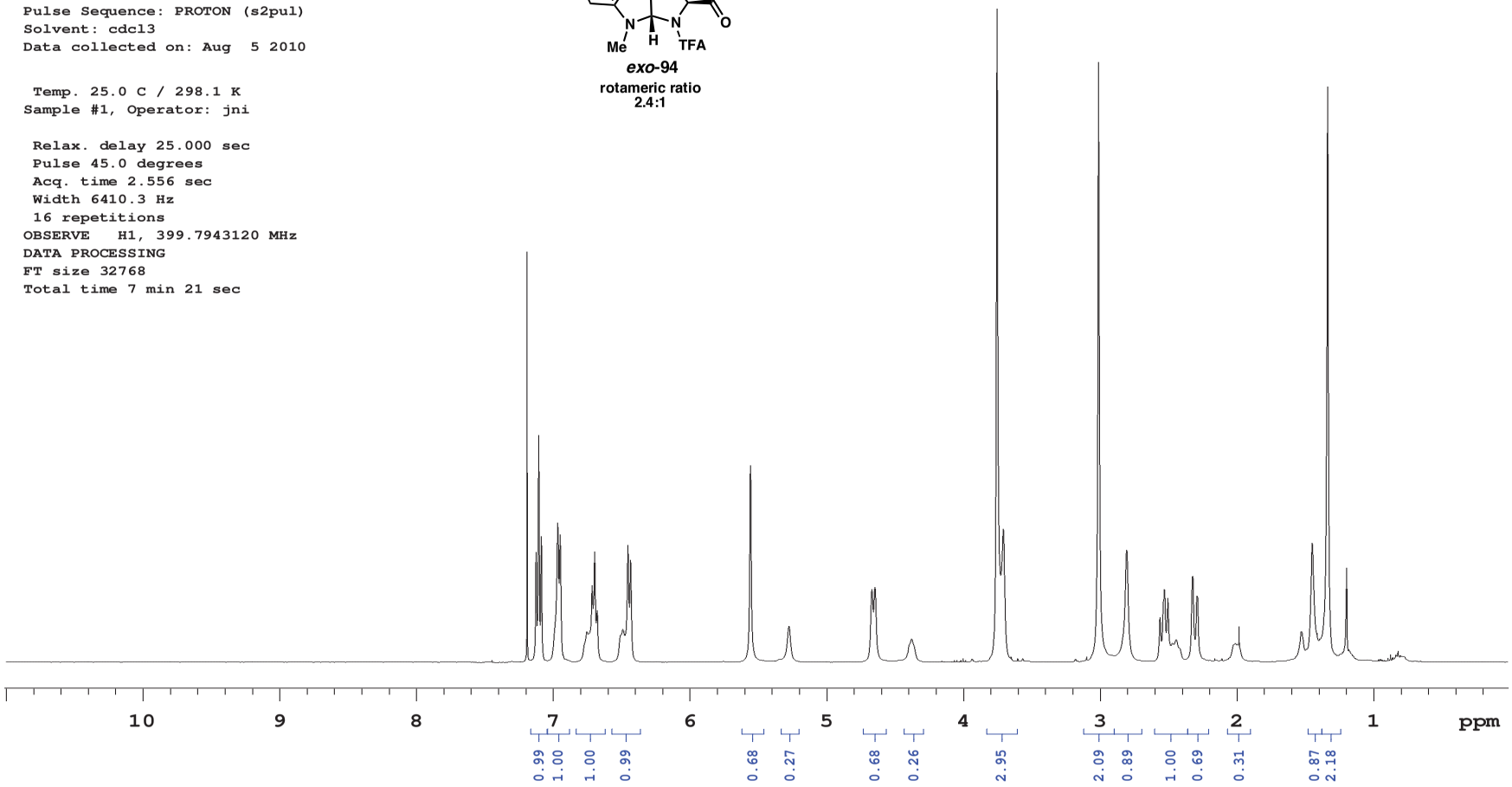

LMRIV-291-minordr
Sample Name:
LMRIV-291-minordr
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-291-minordr
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2010
Temp. 25.0 C / 298.1 K
Sample #12, Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.258 sec
width 26041.7 Hz
14500 repetitions
OBSERVE C13, 100.5283574 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 9 hr, 5 min
OMe
O
Me
N
H
Ac
N
Me
endo-91
rotameric ratio
3.0:1
ppm
200
180
160
140
120
100
80
60
40
20

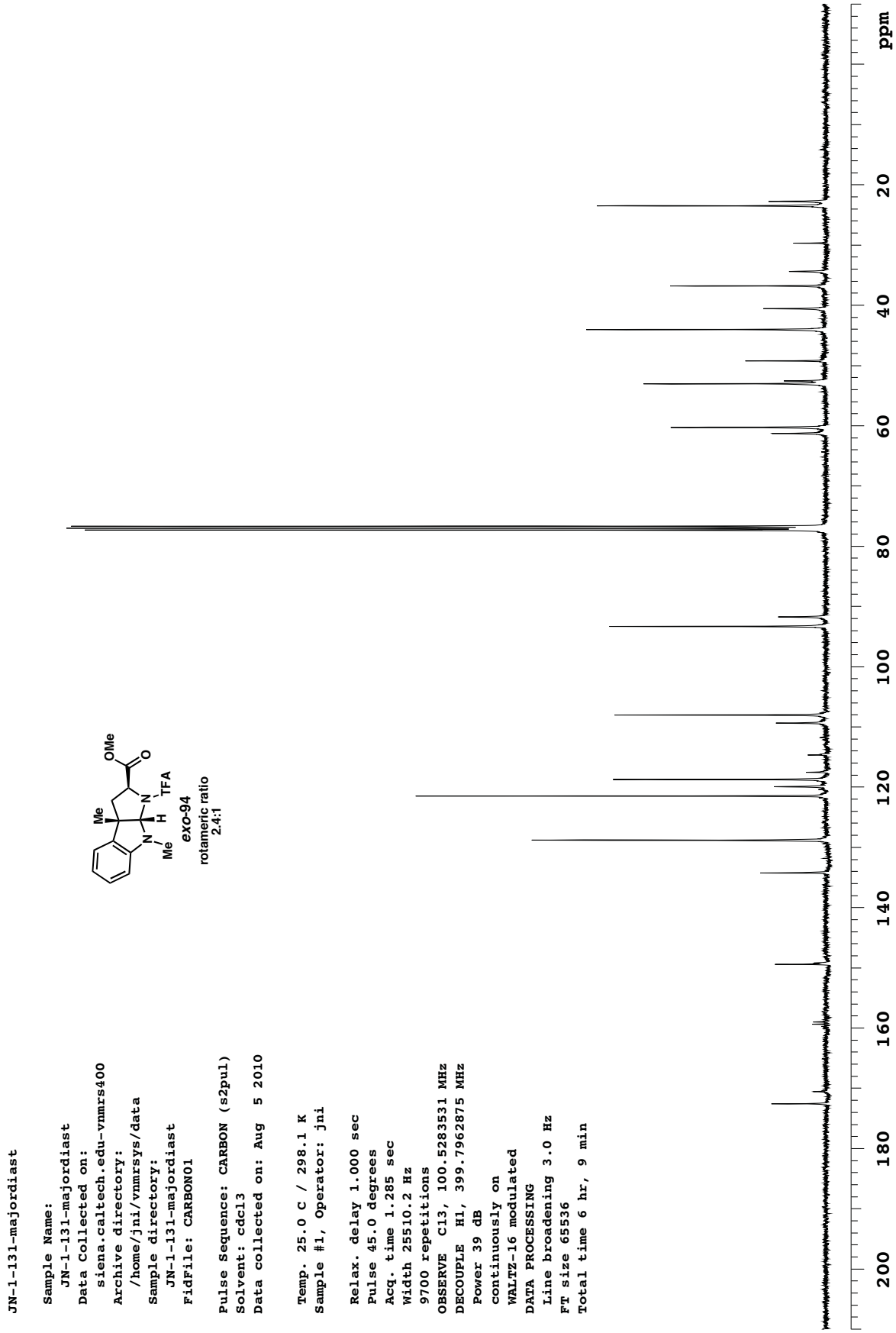
```
JN-1-131-majordiast

 Sample Name:
   JN-1-131-majordiast
 Data Collected on:
   siena.caltech.edu-vnmrs400
 Archive directory:
   /home/jni/vnmrsys/data
 Sample directory:
   JN-1-131-majordiast
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug  5 2010

 Temp. 25.0 C / 298.1 K
Sample #1, Operator: jni

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 2.556 sec
 Width 6410.3 Hz
 16 repetitions
OBSERVE   H1, 399.7943120 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec
```

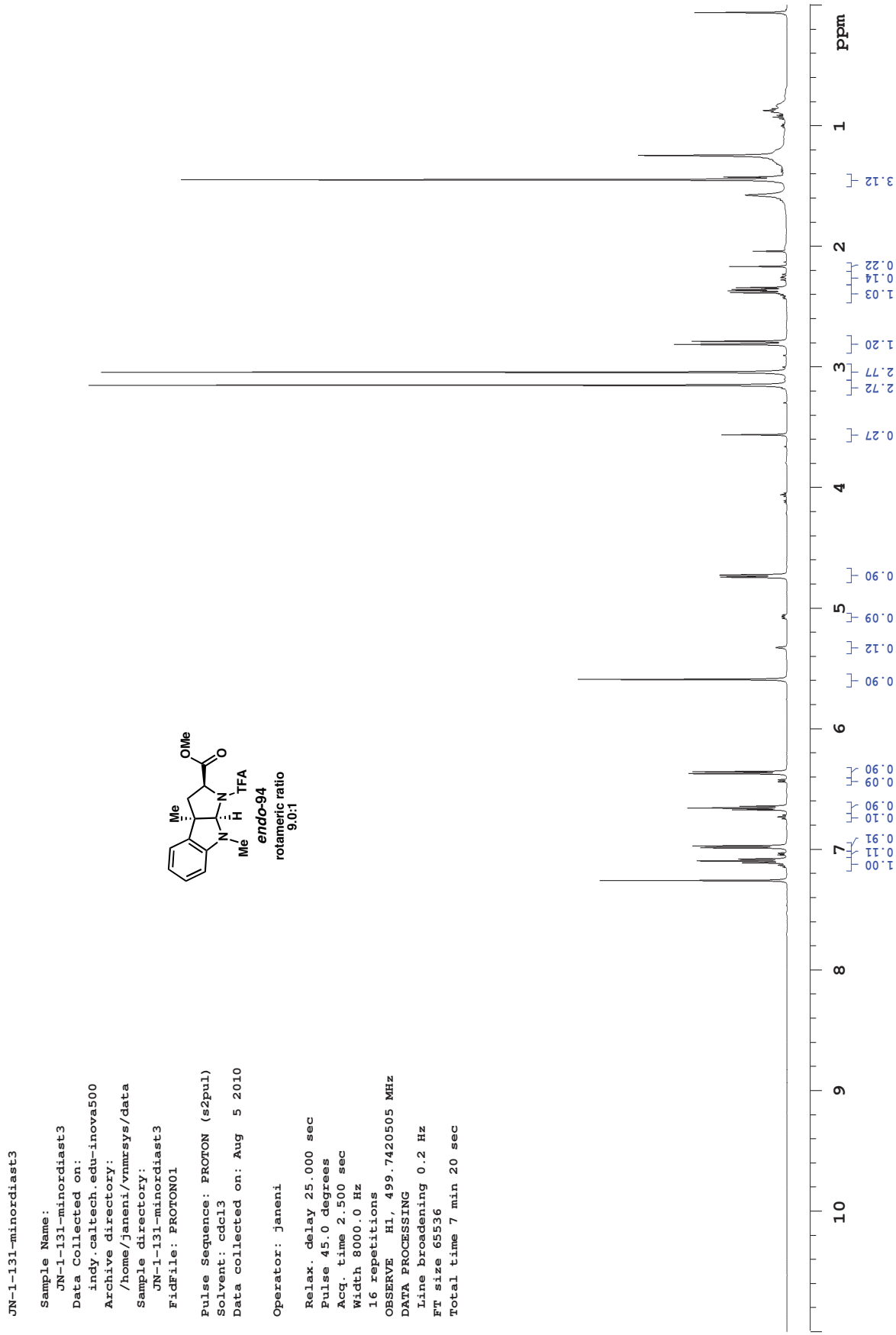


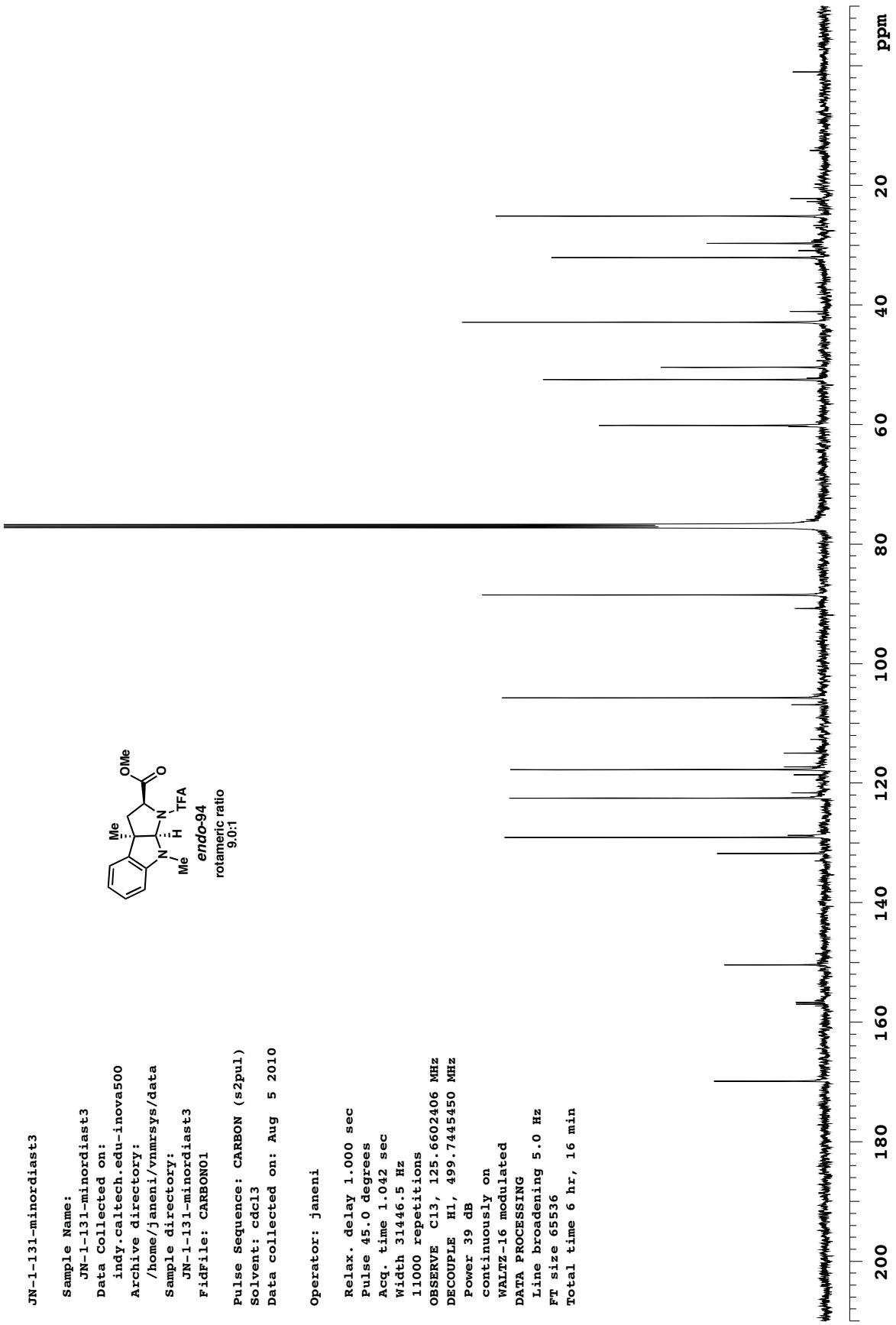

***exo*-94**

**rotameric ratio**
**2.4:1**




JN-1-131-majordiast
Sample Name:
JN-1-131-majordiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-131-majordiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010
Temp. 25.0 C / 298.1 K
Sample #1, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
9700 repetitions
OBSERVE C13, 100.5283531 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 6 hr, 9 min
OMe
O
Me
N
H
TFA
N
Me
exo-94
rotameric ratio
2.4:1
ppm
200
180
160
140
120
100
80
60
40
20


JN-1-131-minordiast3
Sample Name:
JN-1-131-minordiast3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-131-minordiast3
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010
Operator: janeni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec
OMe
O
Me
N
H
TFA
N
Me
endo-94
rotameric ratio
9.0:1
ppm
1
2
3
4
5
6
7
8
9
10
3.12
0.22
0.14
1.03
1.20
2.77
2.72
0.27
0.90
0.09
0.12
0.90
0.90
0.09
0.90
0.10
0.91
0.11
1.00


JN-1-131-minordiast3
Sample Name:
JN-1-131-minordiast3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-131-minordiast3
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 5 2010
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
11000 repetitions
OBSERVE C13, 125.6602406 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 6 hr, 16 min
OMe
O
Me
N
H
TFA
N
Me
endo-94
rotameric ratio
9.0:1
ppm
200
180
160
140
120
100
80
60
40
20

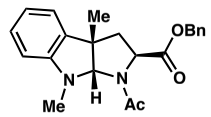
JN-1-151-major

Sample Name:
JN-1-151-major
Data Collected on:
hg3.caltech.edu-mercury300
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-151-major
FidFile: PROTON01

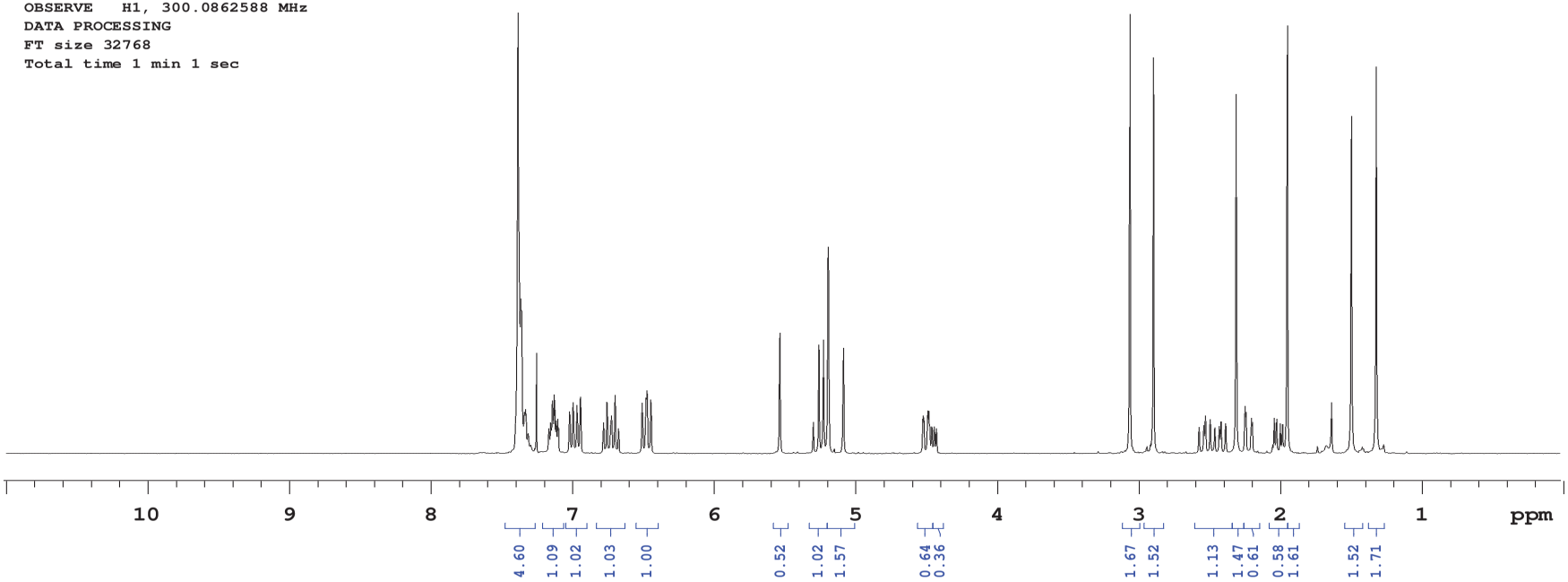
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 24 2010

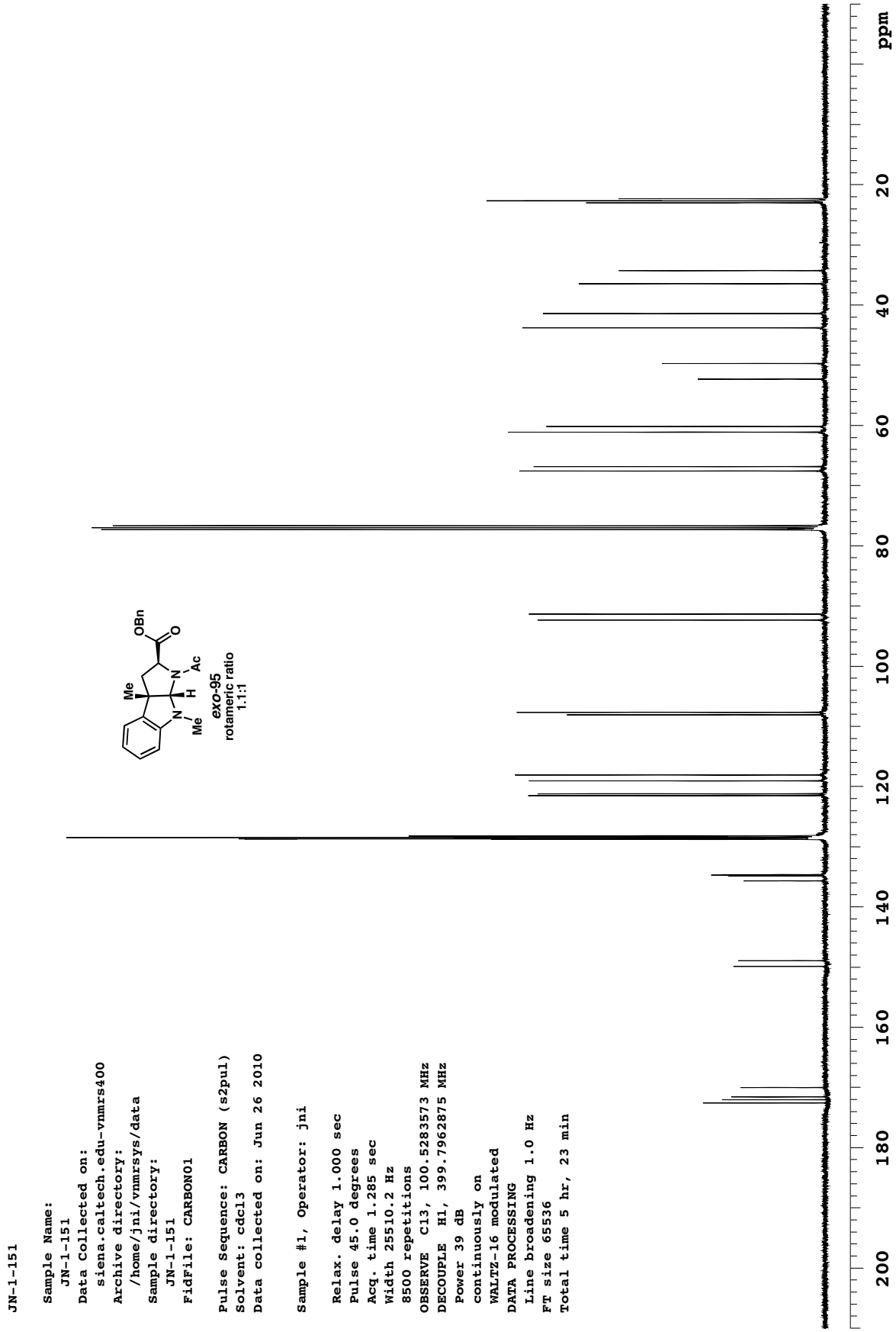
Temp. 25.0 C / 298.1 K
Sample #20, Operator: jni

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862588 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 1 sec




*exo*-95
rotameric ratio
1.1:1




JN-1-151
Sample Name:
JN-1-151
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-151
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 26 2010
Sample #1, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
8500 repetitions
OBSERVE C13, 100.5283573 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 5 hr, 23 min
OBn
O
Me
N
Ac
H
N
Me
exo-95
rotameric ratio
1.1:1
200
180
160
140
120
100
80
60
40
20
ppm

JN-1-151-minordiast3

Sample Name:
JN-1-151-minordiast3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-151-minordiast3
FidFile: PROTON01

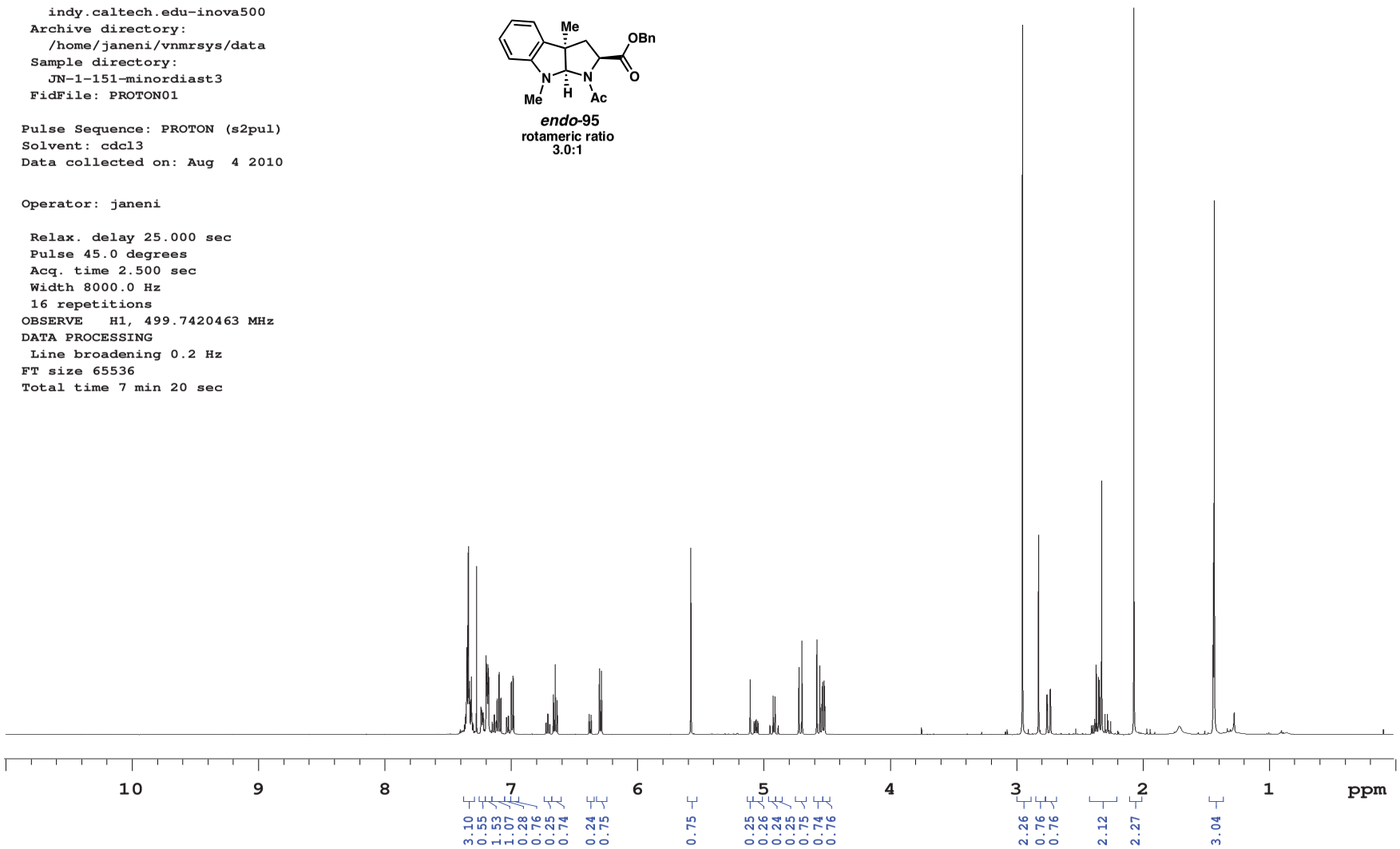
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 4 2010

Operator: janeni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec




***endo*-95**
**rotameric ratio**
**3.0:1**



JN-1-151-minordiast3

Sample Name:
JN-1-151-minordiast3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-151-minordiast3
FidFile: CARBON01

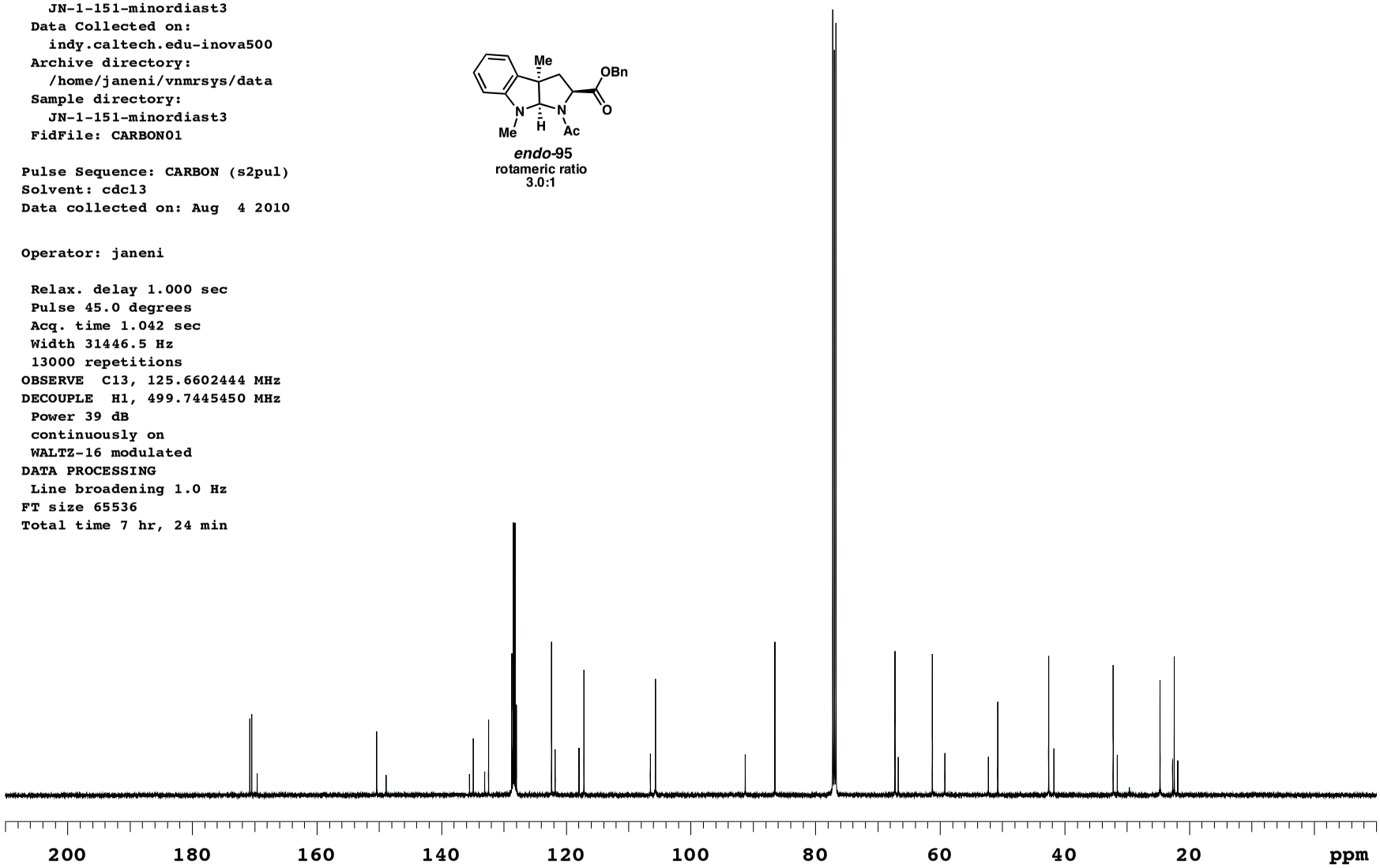
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 4 2010

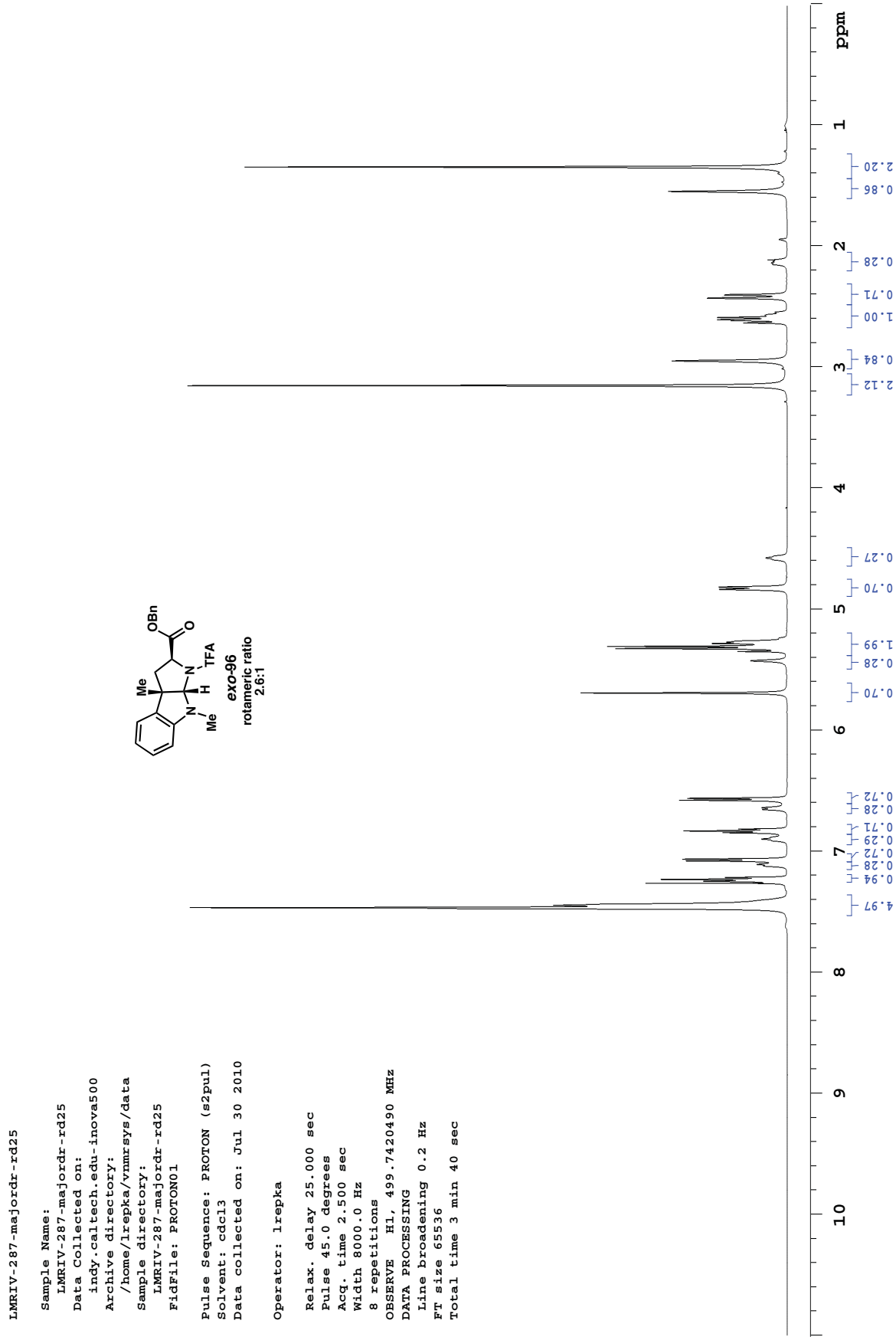
Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602444 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 24 min




*endo*-95
rotameric ratio
3.0:1




LMRIV-287-majordr-rd25
Sample Name:
LMRIV-287-majordr-rd25
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-287-majordr-rd25
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010
Operator: lrepka
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420490 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 3 min 40 sec
OBn
O
Me
N
H
TFA
N
Me
exo-96
rotameric ratio
2.6:1
ppm
1
2
3
4
5
6
7
8
9
10
2.20
0.86
0.28
0.71
1.00
0.84
2.12
0.27
0.70
1.99
0.28
0.70
0.72
0.28
0.71
0.29
0.72
0.28
0.94
4.97

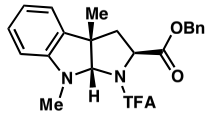
LMRIV-scale-up-12h-majordr

Sample Name:
LMRIV-scale-up-12h-majordr
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-scale-up-12h-majordr
FidFile: CARBON01

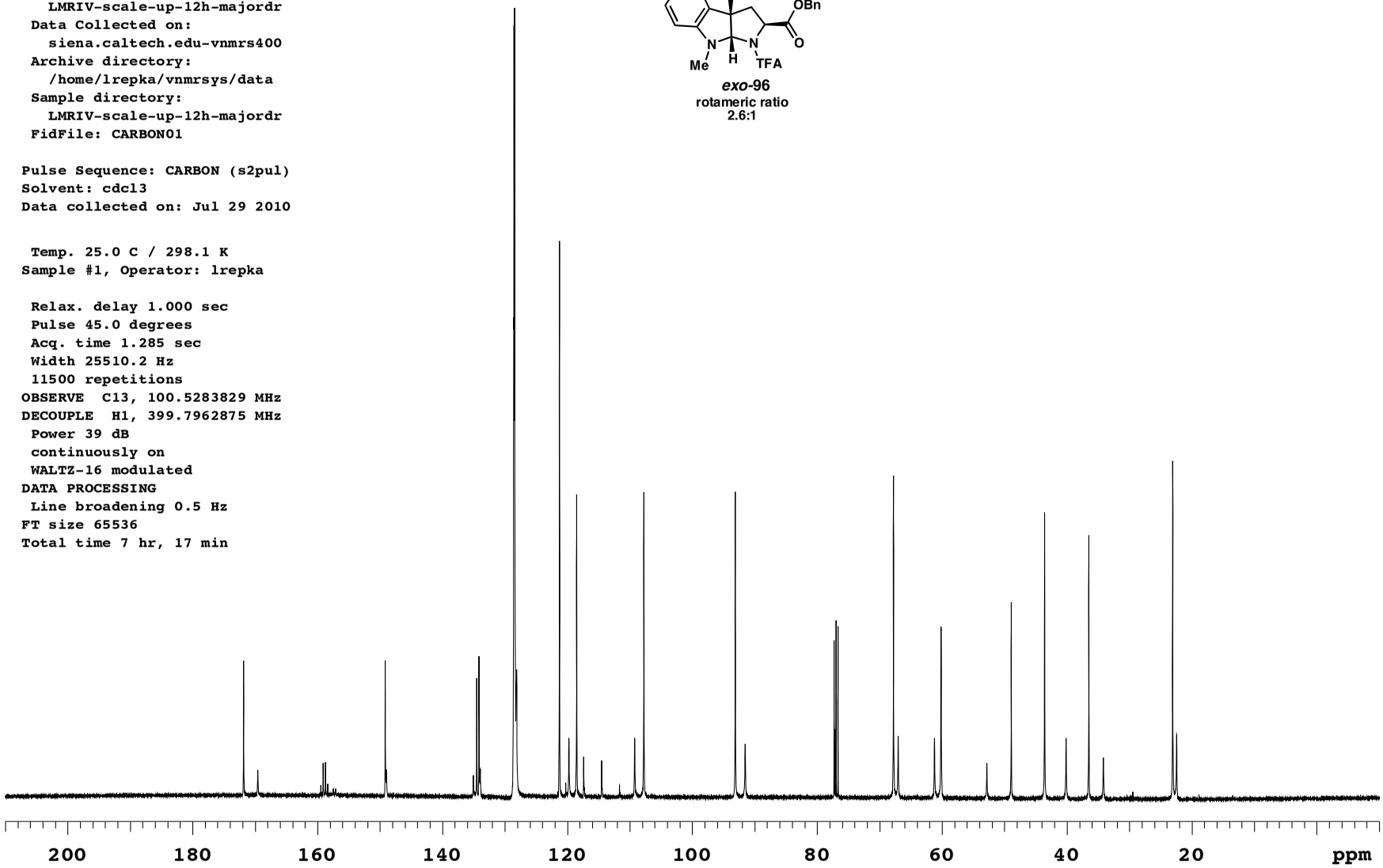
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2010

Temp. 25.0 C / 298.1 K
Sample #1, Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283829 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 17 min



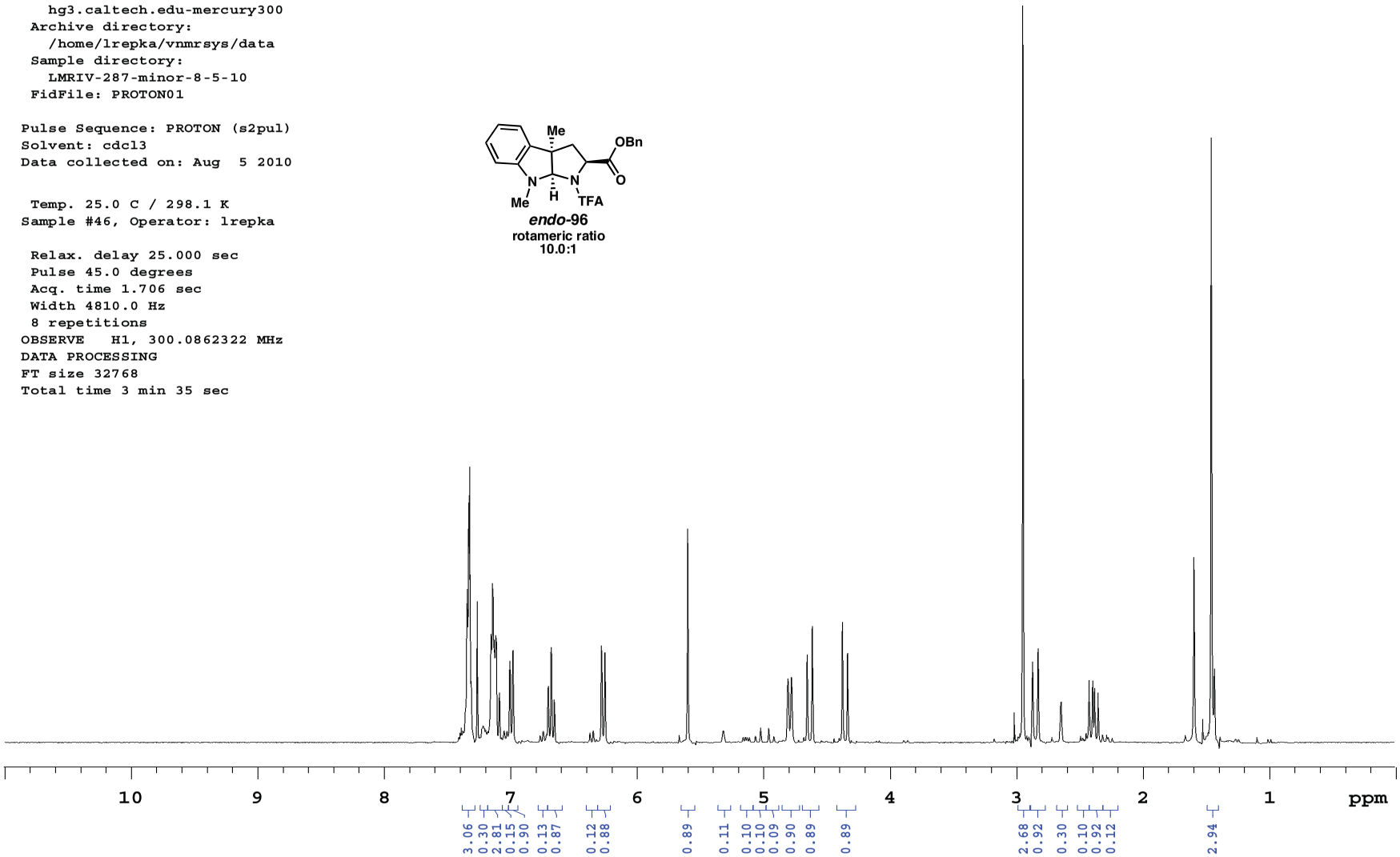

*exo*-96
rotameric ratio
2.6:1



```
LMRIV-287-minor-8-5-10

 Sample Name:
   LMRIV-287-minor-8-5-10
 Data Collected on:
   hg3.caltech.edu-mercury300
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-287-minor-8-5-10
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug  5 2010

 Temp. 25.0 C / 298.1 K
Sample #46, Operator: lrepka

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 1.706 sec
 Width 4810.0 Hz
 8 repetitions
OBSERVE   H1, 300.0862322 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 35 sec
```




***endo*-96**
**rotameric ratio**
**10.0:1**



LMRIV-scale-up-12h-minordr

Sample Name:
LMRIV-scale-up-12h-minordr
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-scale-up-12h-minordr
FidFile: CARBON01

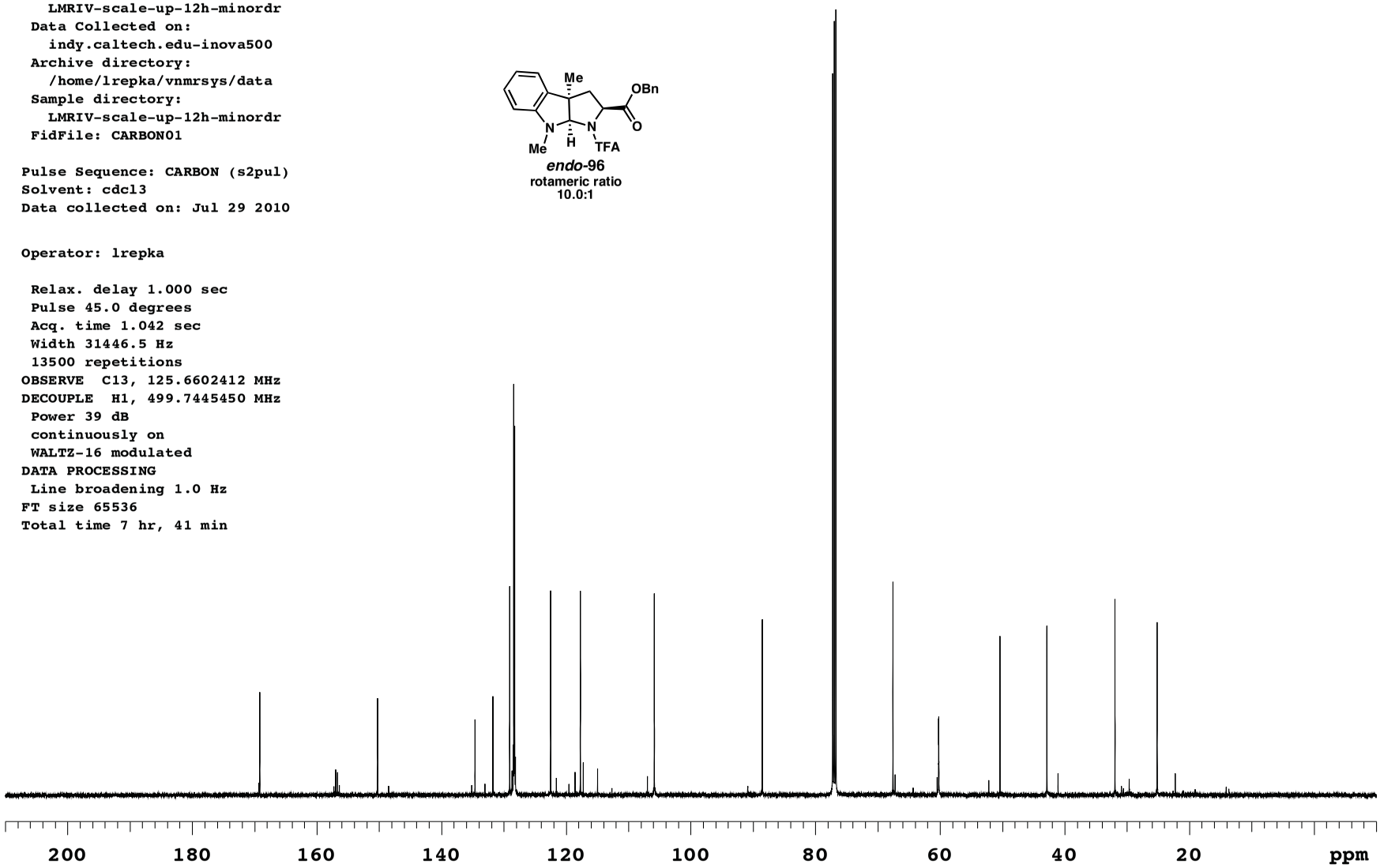
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2010

Operator: lrepka

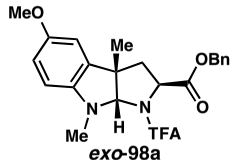
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13500 repetitions
OBSERVE C13, 125.6602412 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 41 min



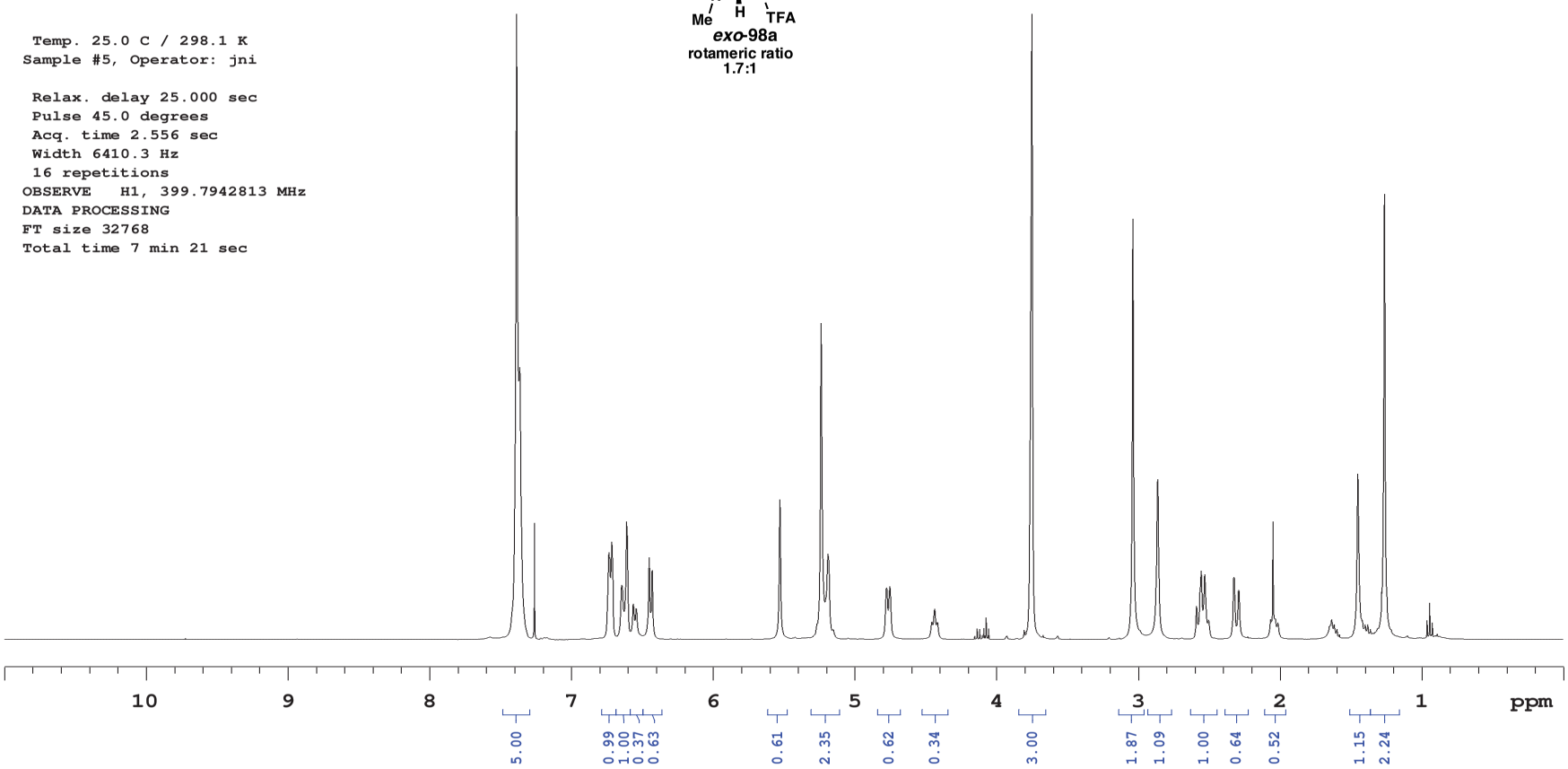

*endo*-96
rotameric ratio
10.0:1

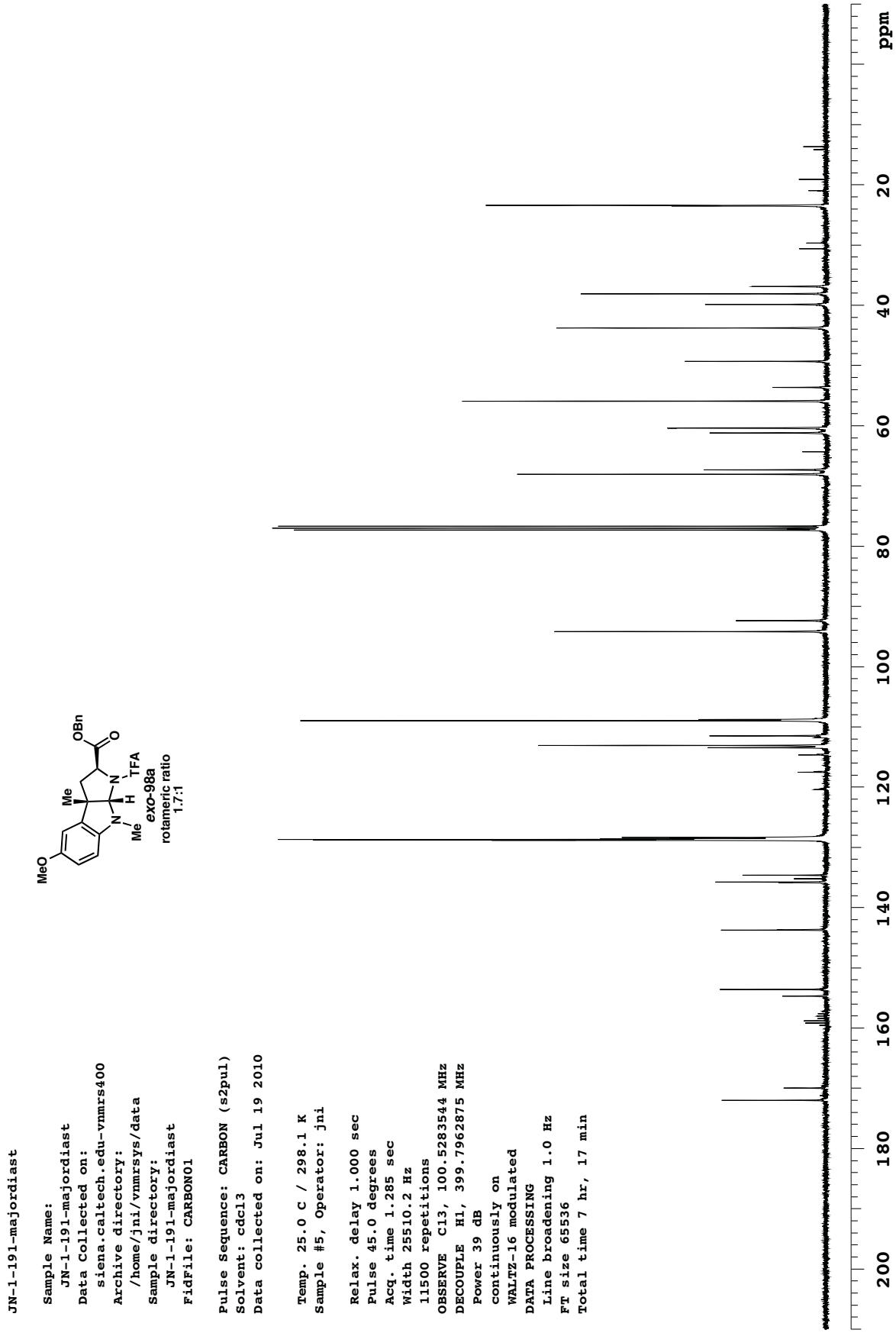


```
JN-1-191-majordiast

 Sample Name:
   JN-1-191-majordiast
 Data Collected on:
   siena.caltech.edu-vnmrs400
 Archive directory:
   /home/jni/vnmrsys/data
 Sample directory:
   JN-1-191-majordiast
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

 Temp. 25.0 C / 298.1 K
Sample #5, Operator: jni

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 2.556 sec
 Width 6410.3 Hz
 16 repetitions
OBSERVE   H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec
```




*exo*-98a
rotameric ratio
1.7:1




JN-1-191-majordiast
Sample Name:
JN-1-191-majordiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-191-majordiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010
Temp. 25.0 C / 298.1 K
Sample #5, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283544 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 17 min
MeO
Me
OBn
O
N
H
TFA
N
Me
exo-98a
rotameric ratio
1.7:1
200
180
160
140
120
100
80
60
40
20
ppm

JN-1-191-minordiast

Sample Name:
JN-1-191-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-191-minordiast
FidFile: PROTON01

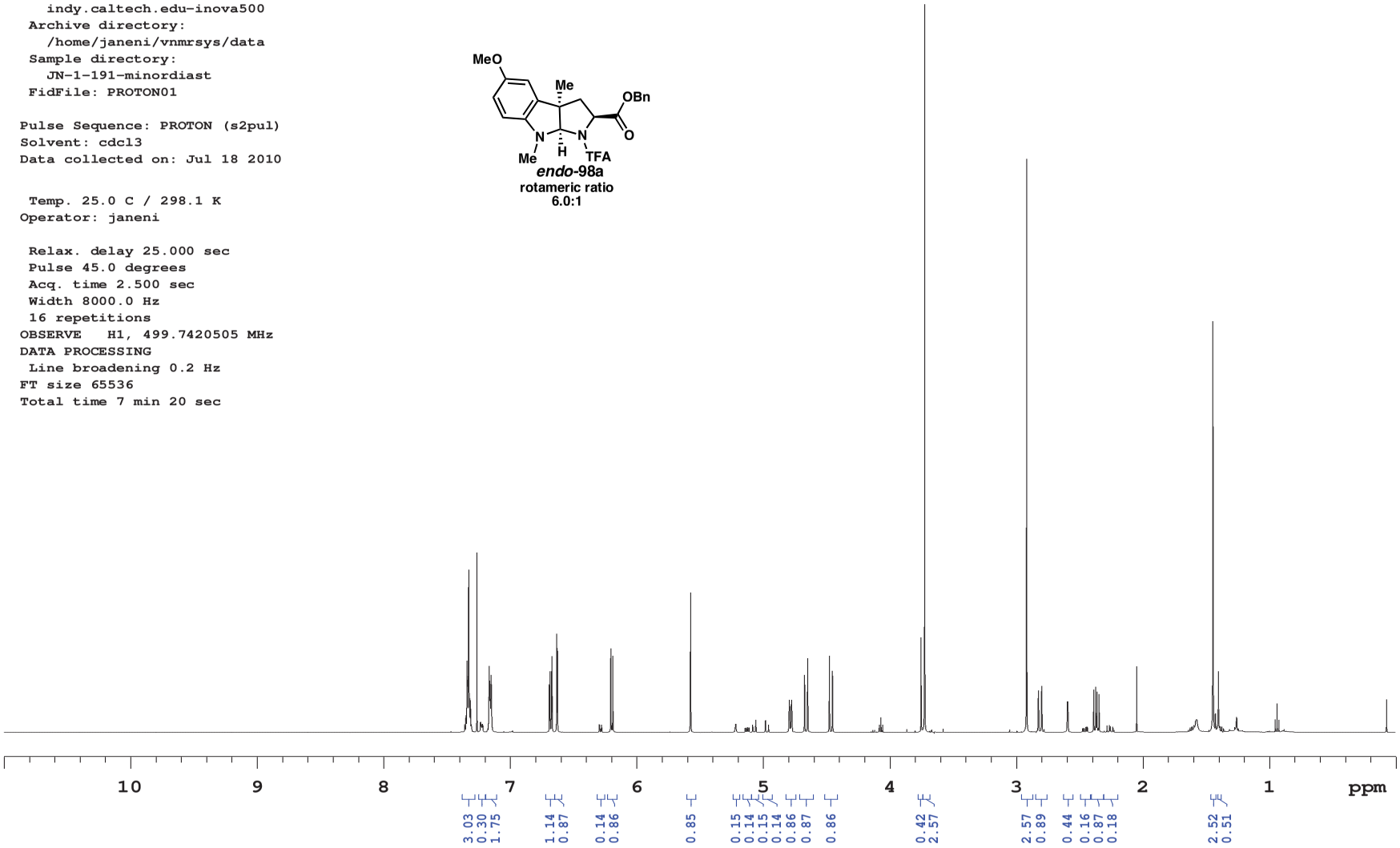
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 18 2010

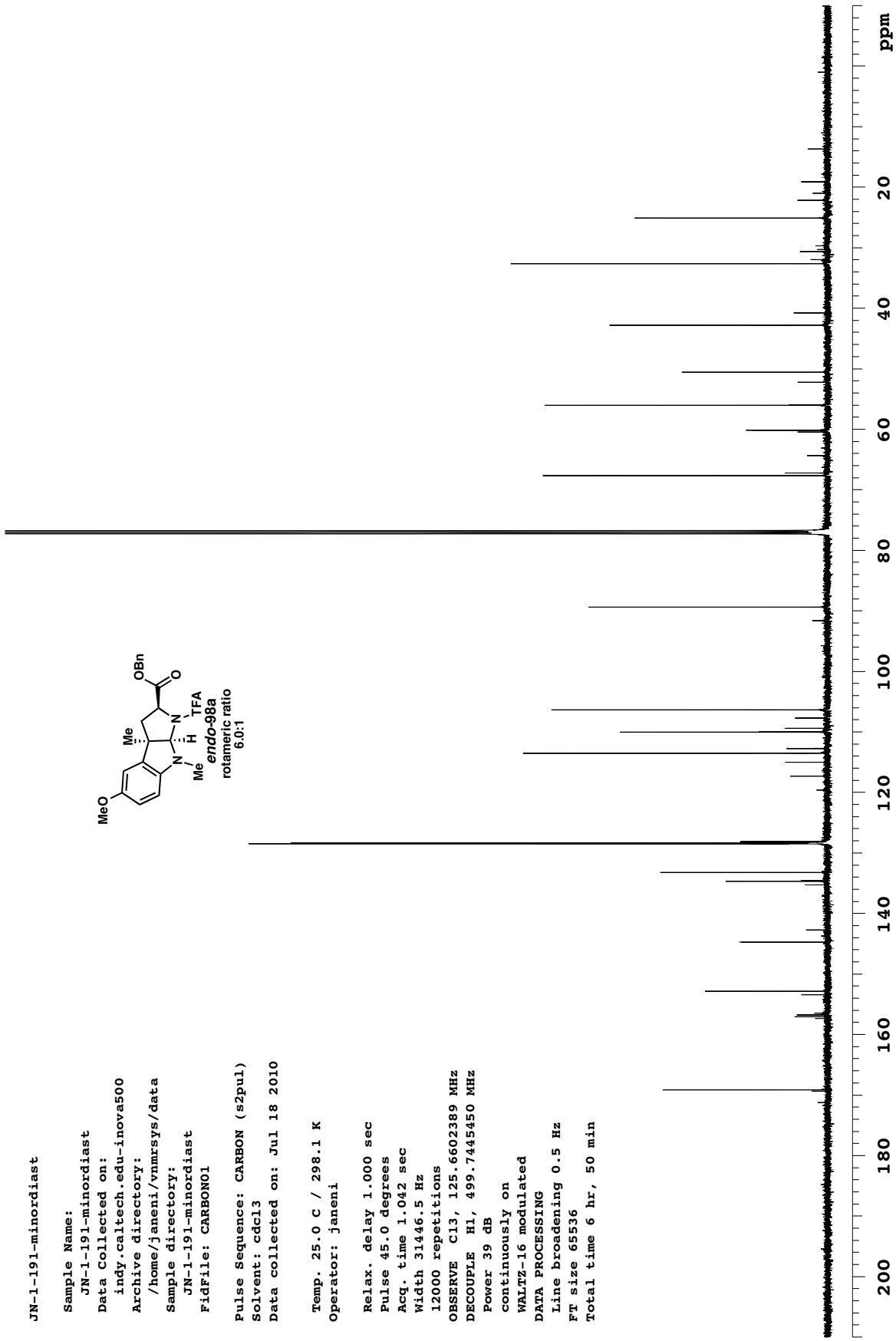
Temp. 25.0 C / 298.1 K
Operator: janeni

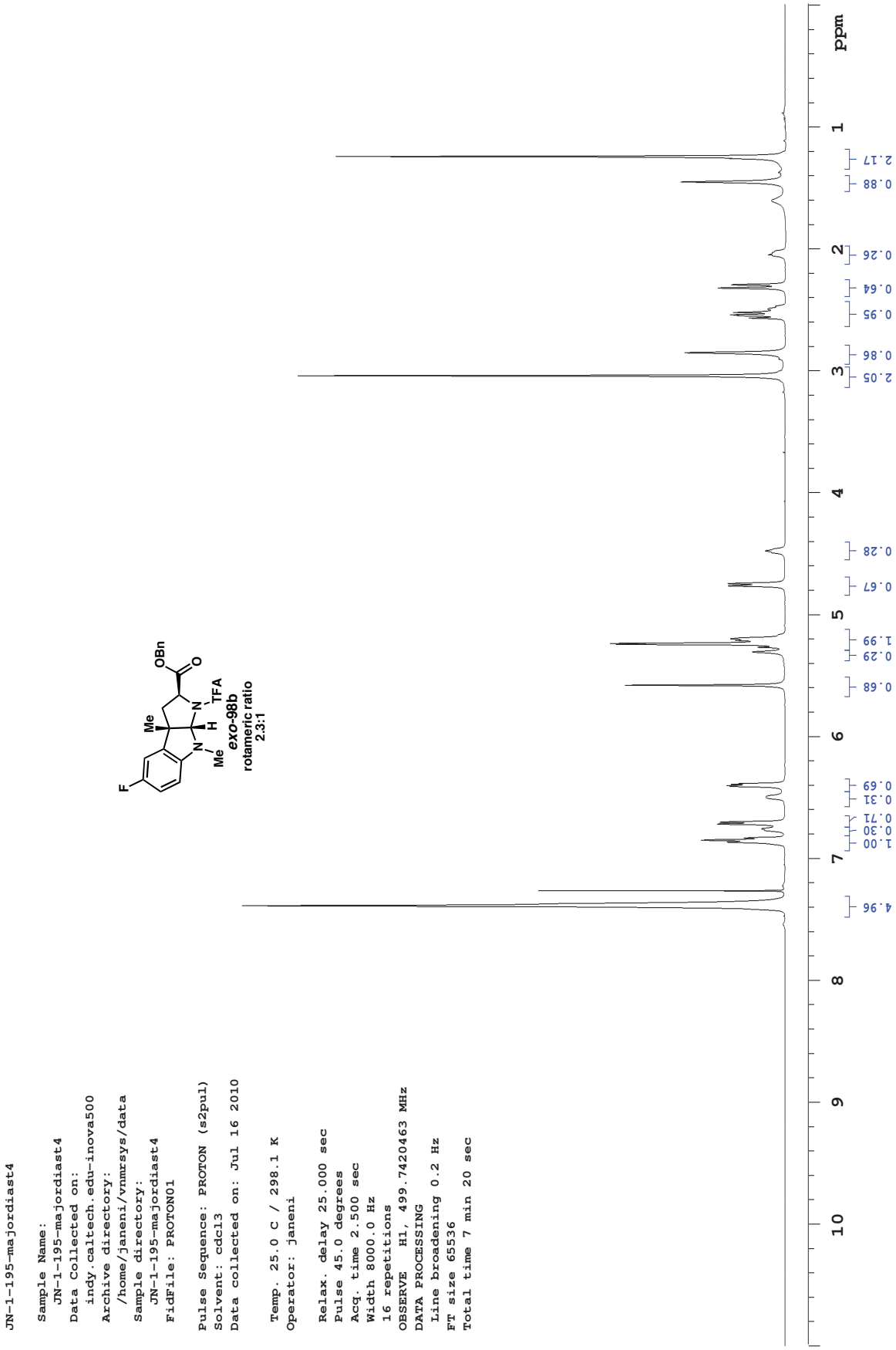
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420505 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

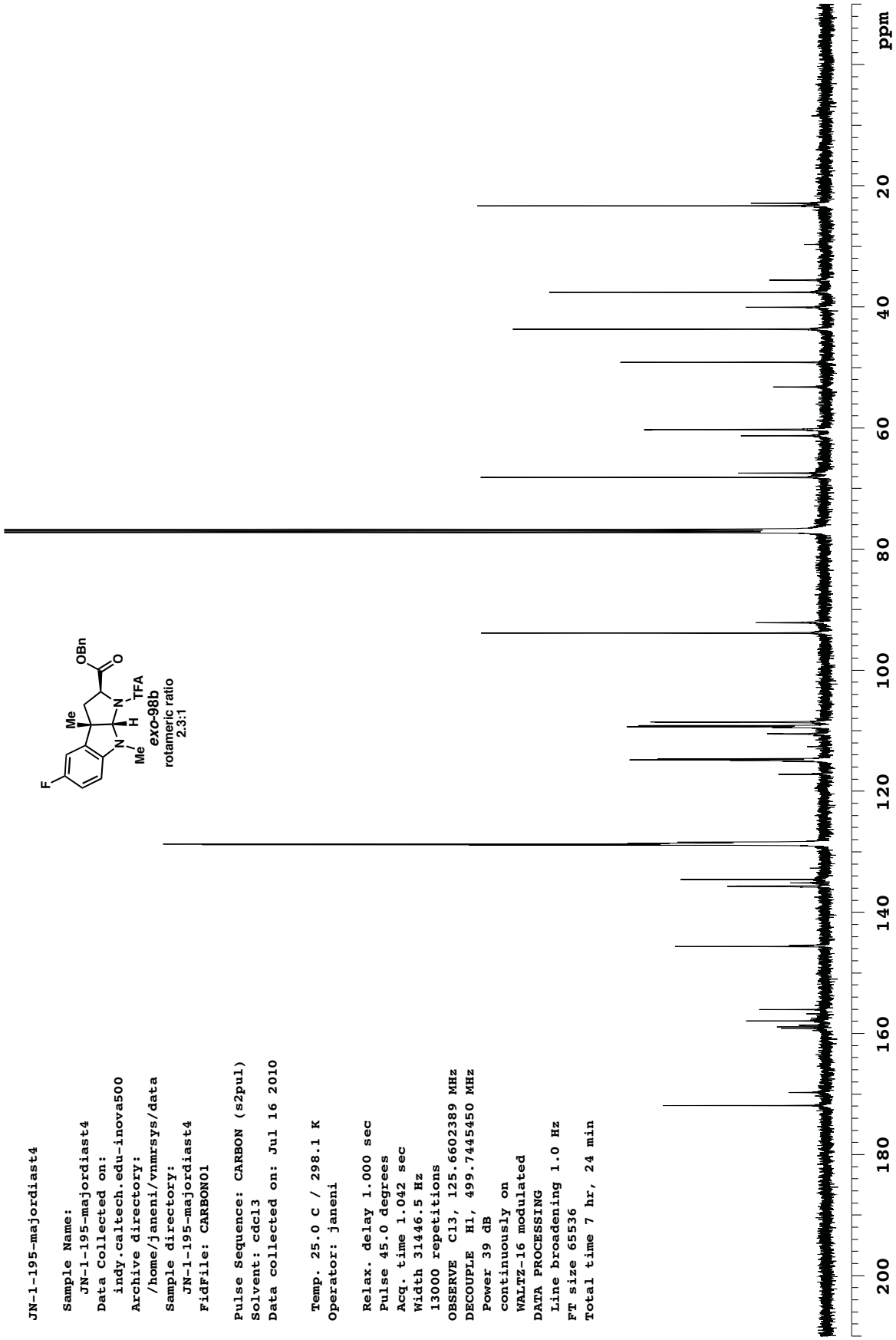



*endo*-98a
rotameric ratio
6.0:1




JN-1-191-minordiast
Sample Name:
JN-1-191-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-191-minordiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 18 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
12000 repetitions
OBSERVE C13, 125.6602389 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 6 hr, 50 min
MeO
Me
Me
OBn
O
N
H
N
TFA
endo-98a
rotameric ratio
6.0:1
ppm
200
180
160
140
120
100
80
60
40
20


JN-1-195-majordiast4
Sample Name:
JN-1-195-majordiast4
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-195-majordiast4
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 16 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec
OBn
O
Me
N
TFA
H
N
Me
F
exo-98b
rotameric ratio
2.3:1
ppm
1
2
3
4
5
6
7
8
9
10
2.17
0.88
0.26
0.64
0.95
0.86
2.05
0.28
0.67
1.99
0.29
0.68
0.69
0.31
0.71
0.30
1.00
4.96


JN-1-195-majordiast4
Sample Name:
JN-1-195-majordiast4
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-195-majordiast4
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 16 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602389 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 24 min
OBn
O
Me
N
TFA
H
N
Me
F
exo-98b
rotameric ratio
2.3:1
ppm
200
180
160
140
120
100
80
60
40
20

JN-1-195-minordiast

Sample Name:
JN-1-195-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-195-minordiast
FidFile: PROTON01

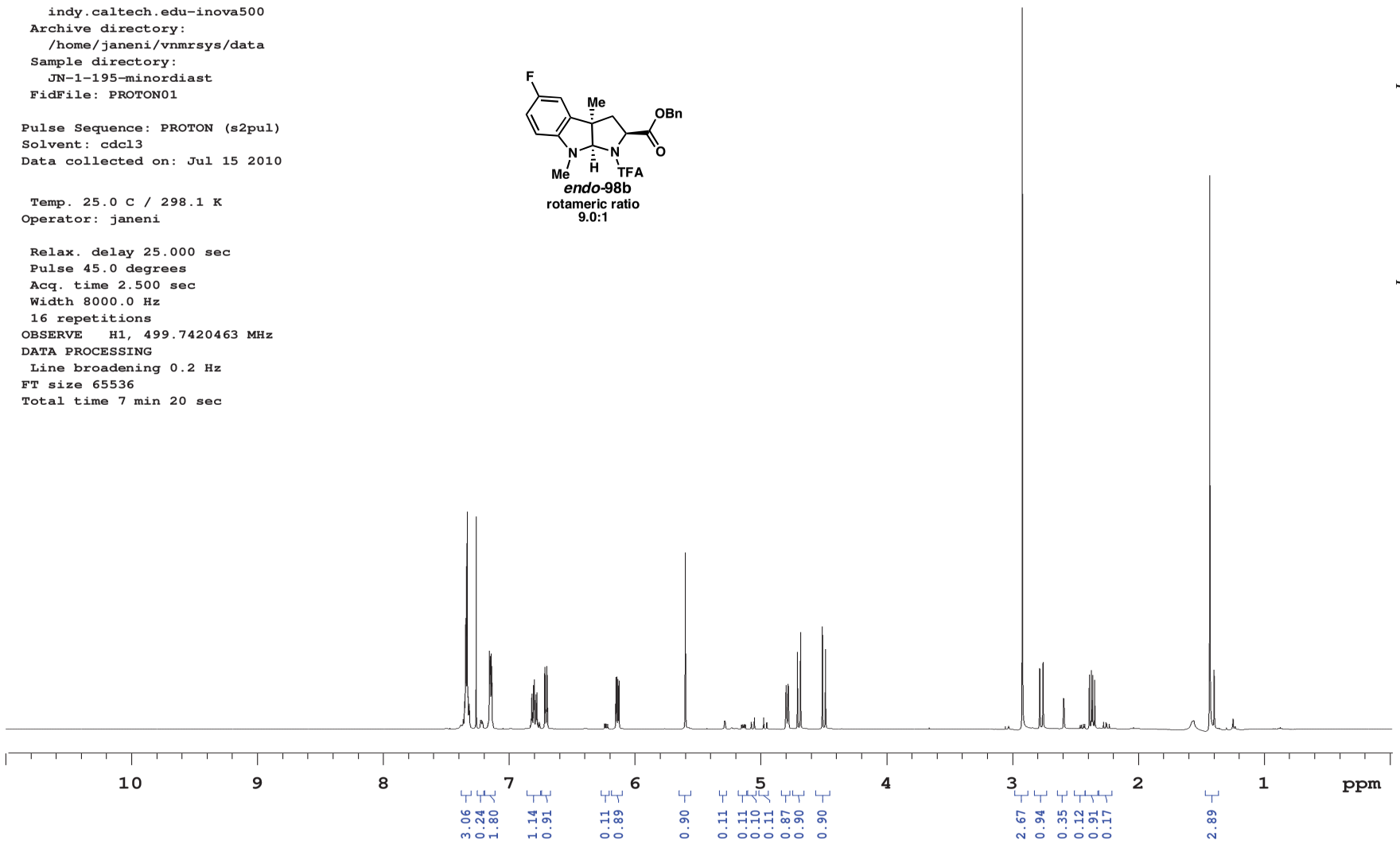
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 15 2010

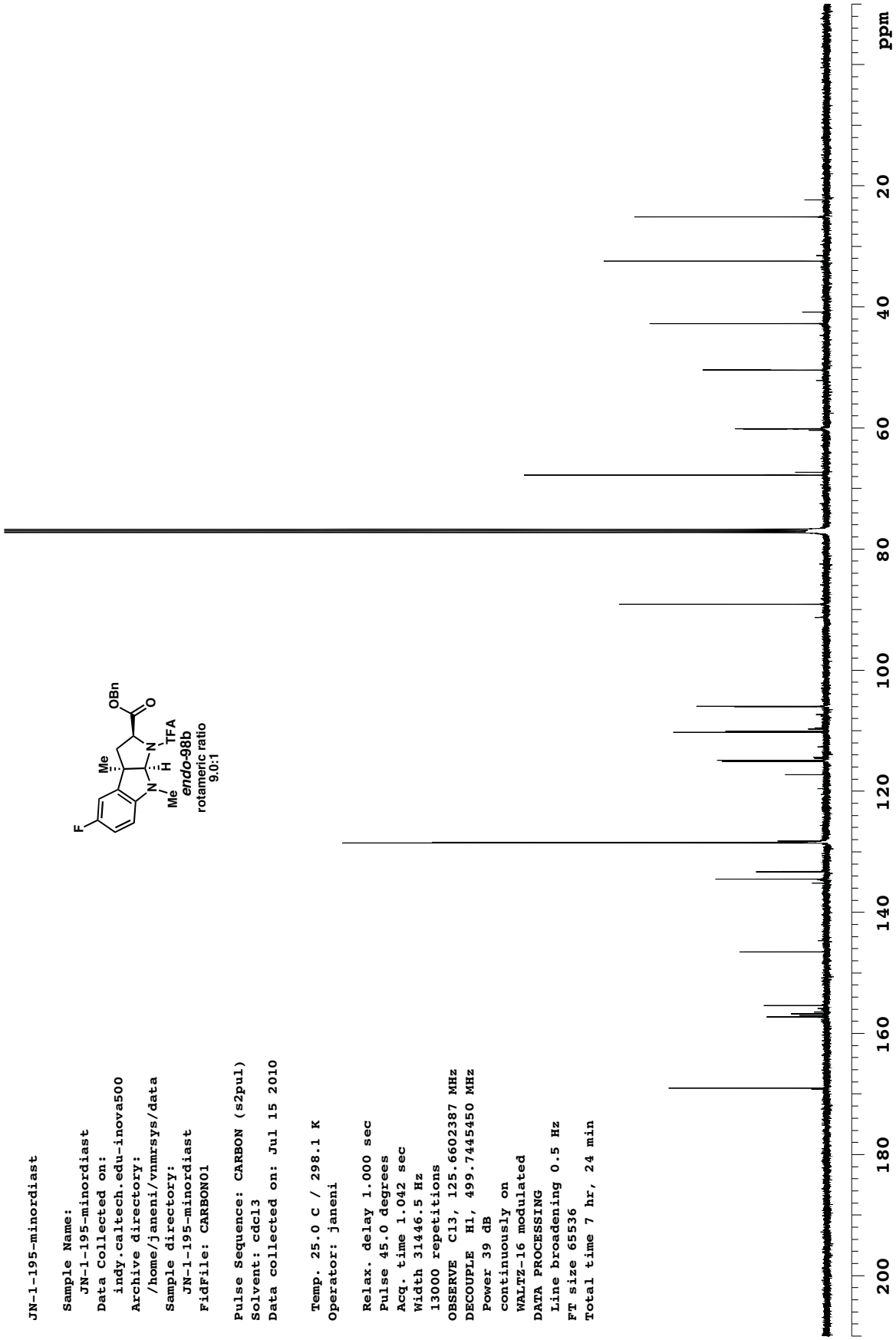
Temp. 25.0 C / 298.1 K
Operator: janeni

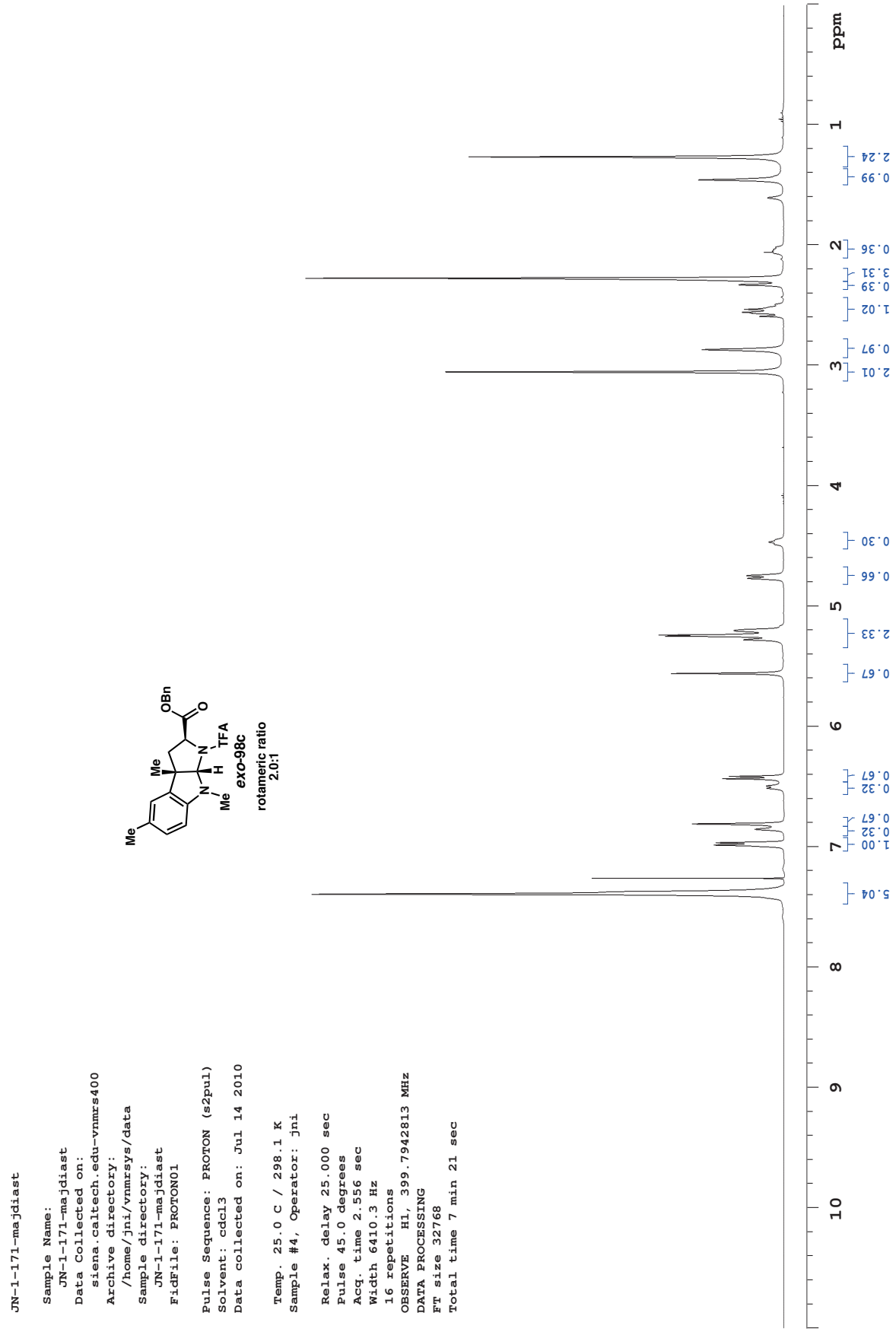
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec

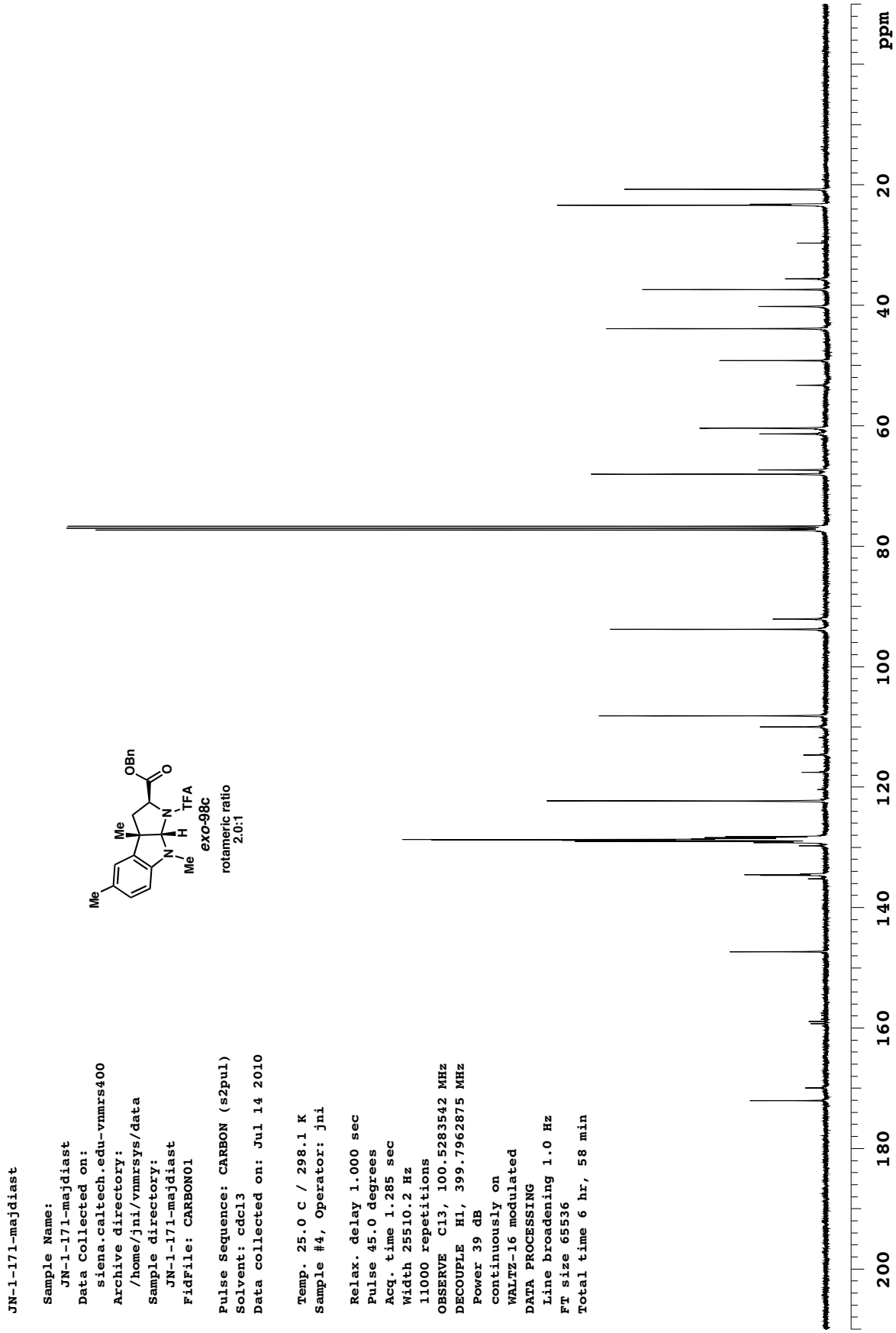


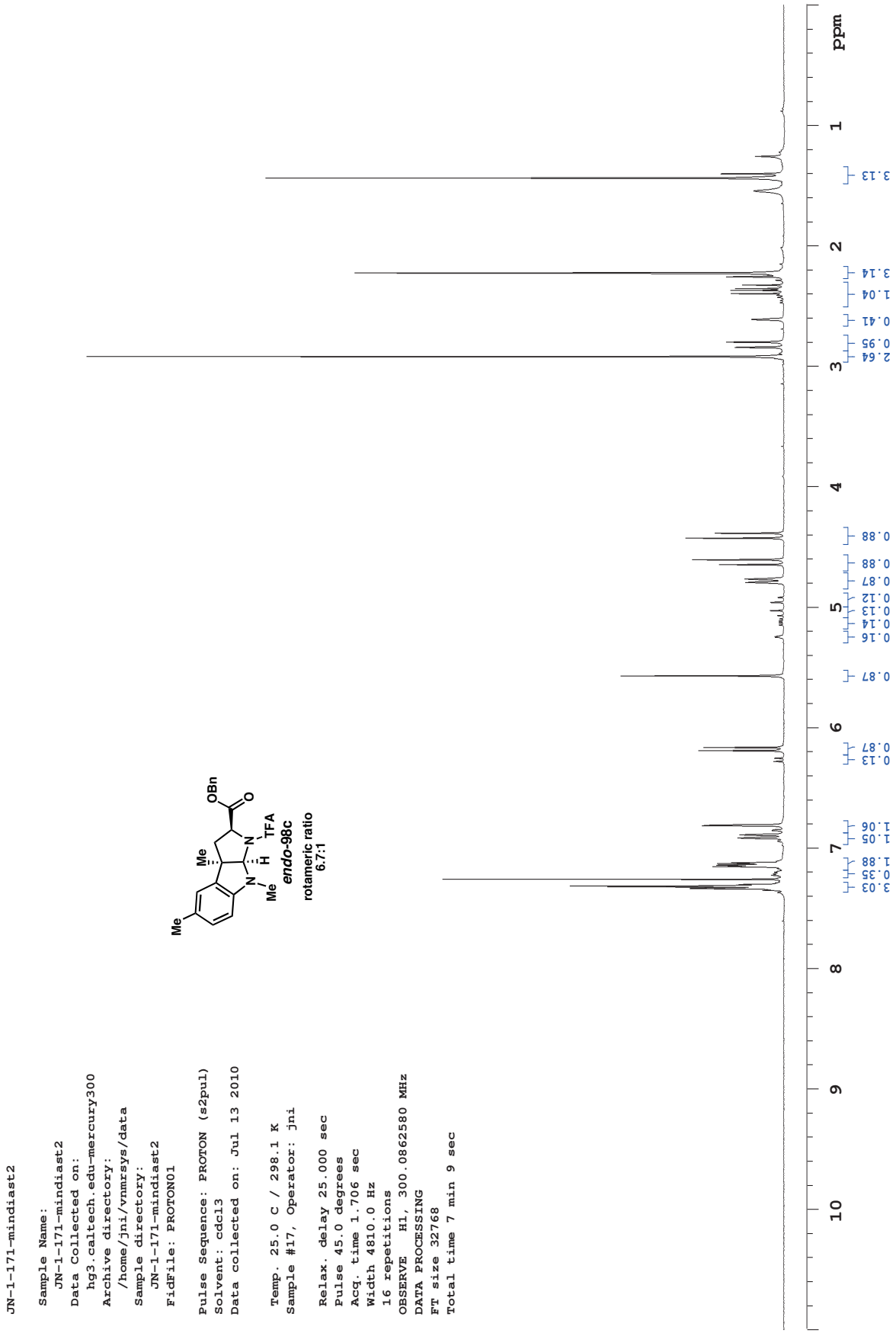

*endo*-98b
rotameric ratio
9.0:1

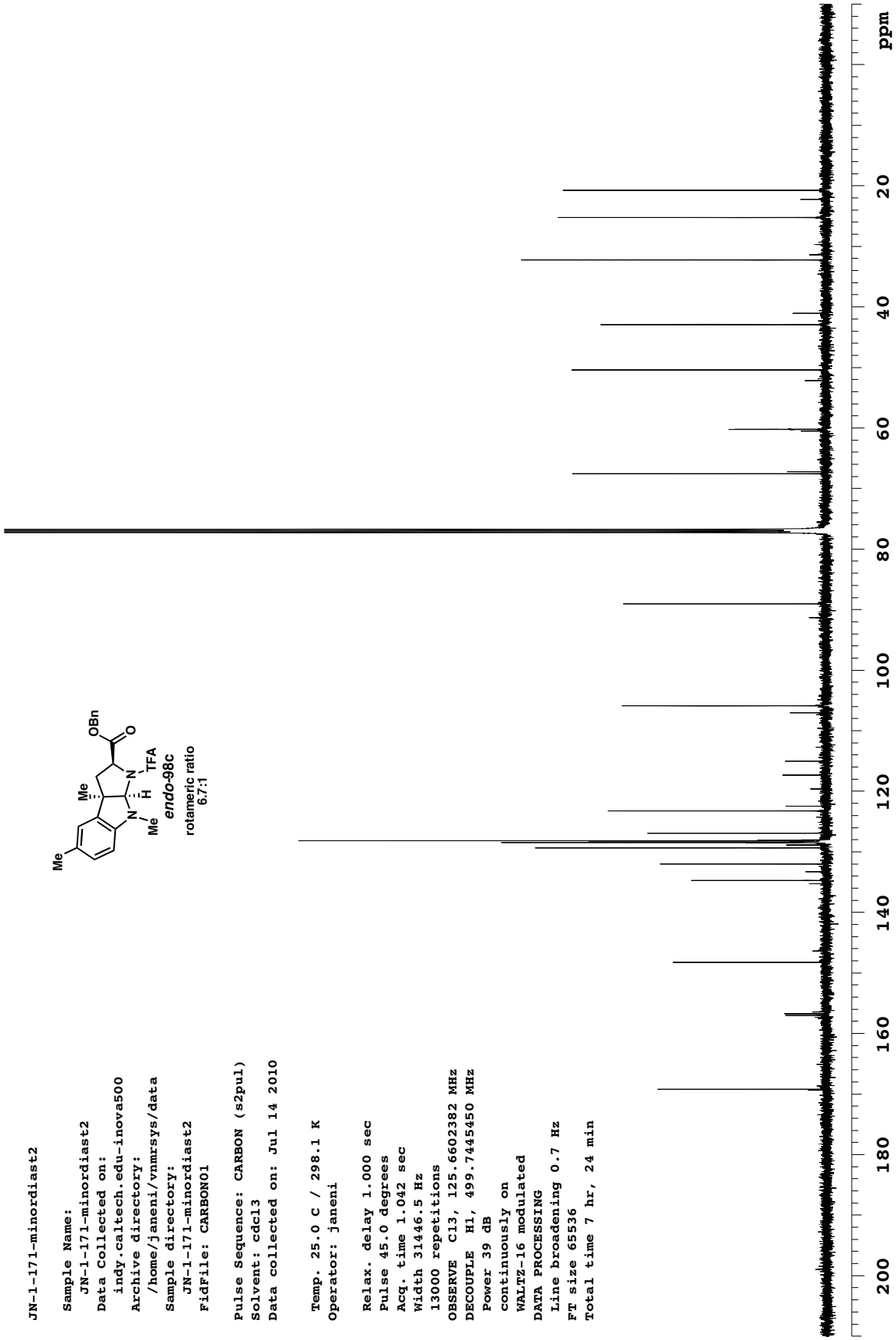



JN-1-195-minordiast
Sample Name:
JN-1-195-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-195-minordiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 15 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602387 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 24 min
OBn
O
Me
N
H
TFA
N
Me
F
endo-98b
rotameric ratio
9.0:1
ppm
200
180
160
140
120
100
80
60
40
20

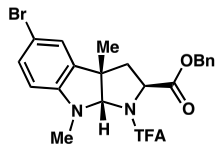

JN-1-171-majdiast
Sample Name:
JN-1-171-majdiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-171-majdiast
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010
Temp. 25.0 C / 298.1 K
Sample #4, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942813 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec
OBn
O
Me
N
TFA
H
N
Me
Me
exo-98c
rotameric ratio
2.0:1
ppm
1
2
3
4
5
6
7
8
9
10
2.24
0.99
0.36
3.31
0.39
1.02
0.97
2.01
0.30
0.66
2.33
0.67
0.67
0.32
0.67
0.32
1.00
5.04


JN-1-171-majdiast
Sample Name:
JN-1-171-majdiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-171-majdiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010
Temp. 25.0 C / 298.1 K
Sample #4, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
11000 repetitions
OBSERVE C13, 100.5283542 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 6 hr, 58 min
Me
Me
OBn
O
N
H
TFA
N
Me
exo-98c
rotameric ratio
2.0:1
ppm
200
180
160
140
120
100
80
60
40
20

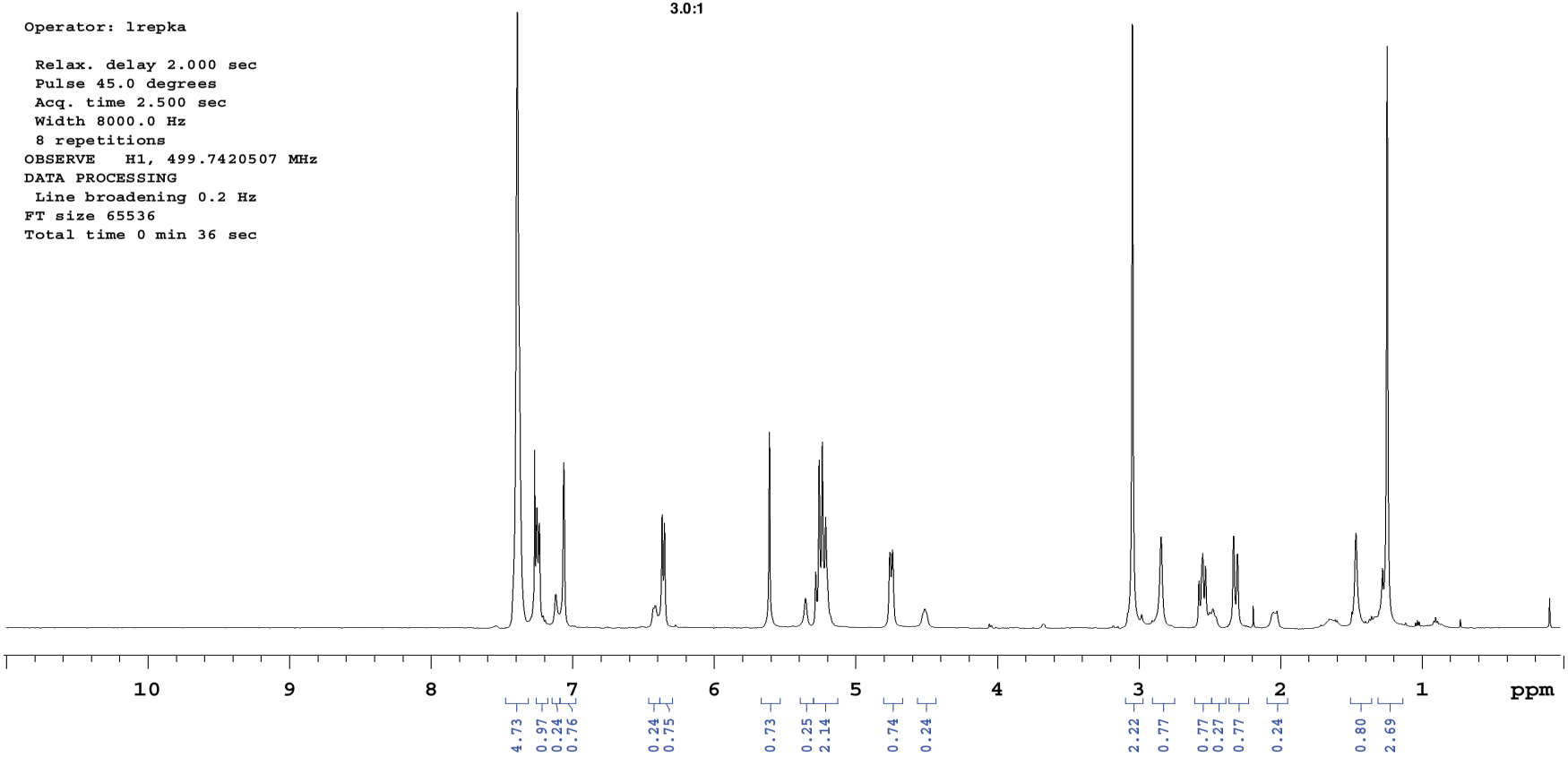

JN-1-171-mindiast2
Sample Name:
JN-1-171-mindiast2
Data Collected on:
hg3.caltech.edu-mercury300
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-171-mindiast2
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 13 2010
Temp. 25.0 C / 298.1 K
Sample #17, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4810.0 Hz
16 repetitions
OBSERVE H1, 300.0862580 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 9 sec
OBn
O
Me
N
TFA
H
N
Me
Me
endo-98c
rotameric ratio
6.7:1
ppm
1
2
3
4
5
6
7
8
9
10
3.13
3.14
1.04
0.41
0.95
2.64
0.88
0.88
0.87
0.12
0.13
0.14
0.16
0.87
0.87
0.13
1.06
1.05
1.88
0.35
3.03

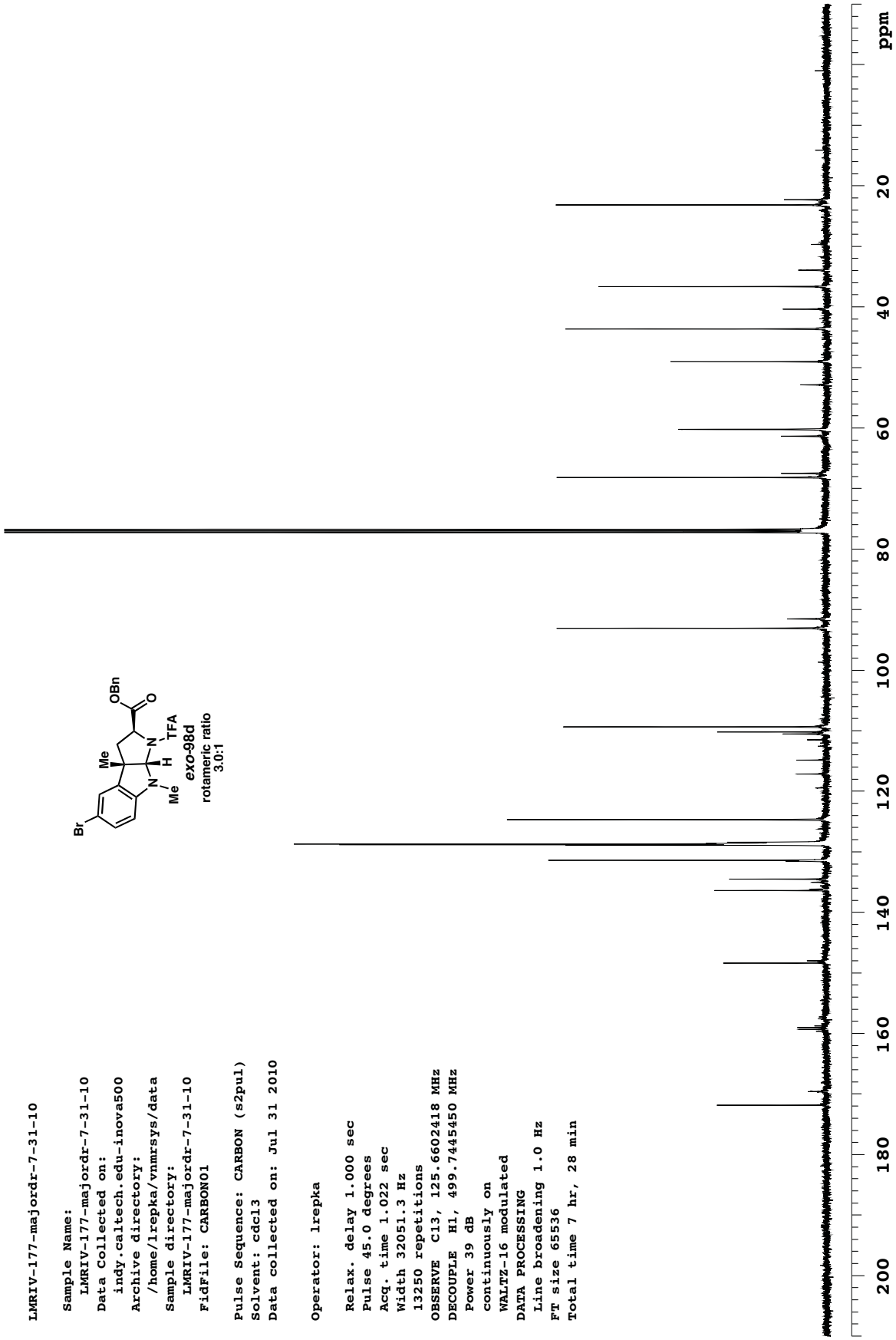

JN-1-171-minordiast2
Sample Name:
JN-1-171-minordiast2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-171-minordiast2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010
Temp. 25.0 C / 298.1 K
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602382 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.7 Hz
FT size 65536
Total time 7 hr, 24 min
Me
Me
Me
OBn
O
N
H
N
TFA
endo-98c
rotameric ratio
6.7:1
ppm
200
180
160
140
120
100
80
60
40
20

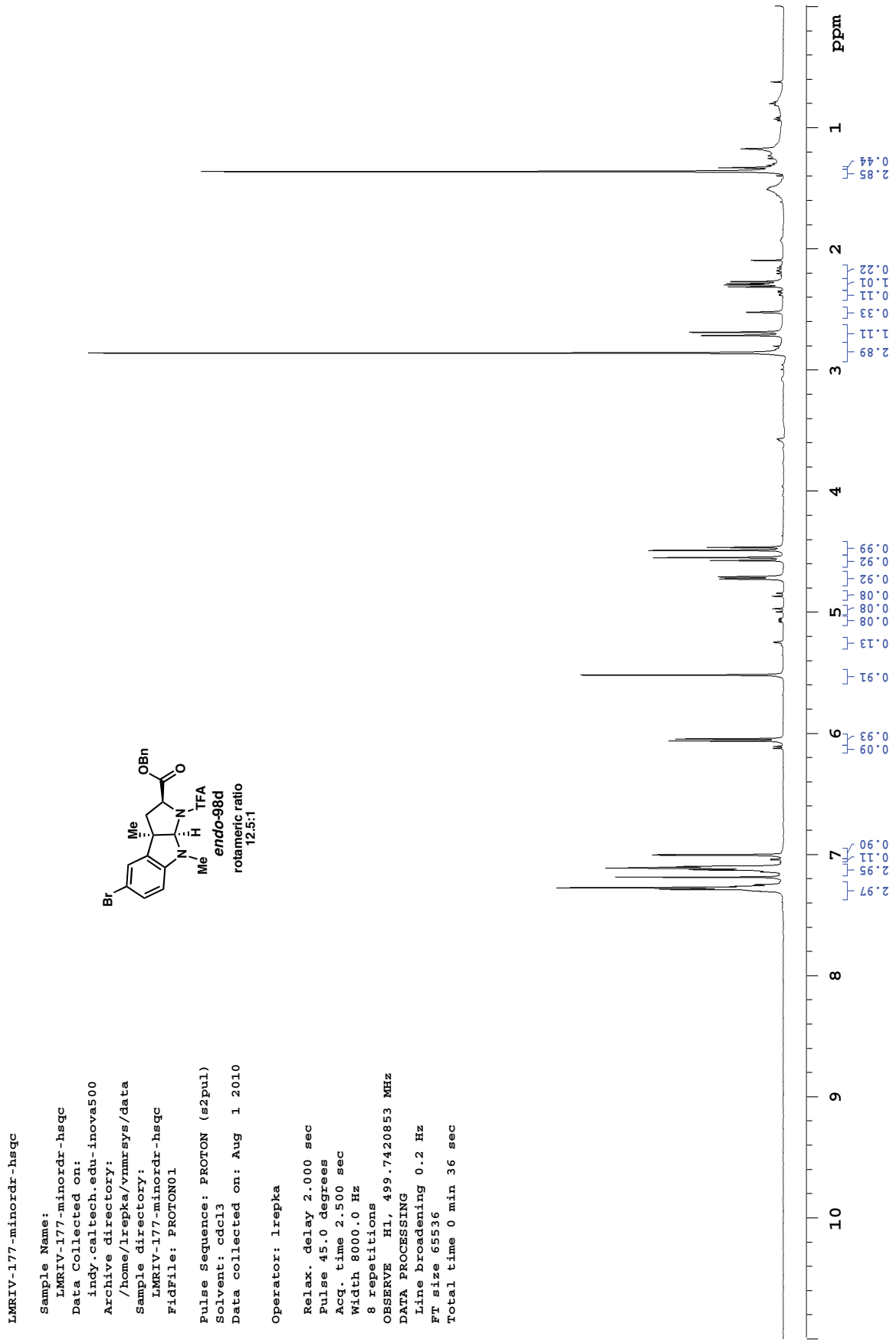
```
LMRIV-177-majordr-hsqc2

 Sample Name:
   LMRIV-177-majordr-hsqc2
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-177-majordr-hsqc2
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2010

Operator: lrepka

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
OBSERVE   H1, 499.7420507 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec
```

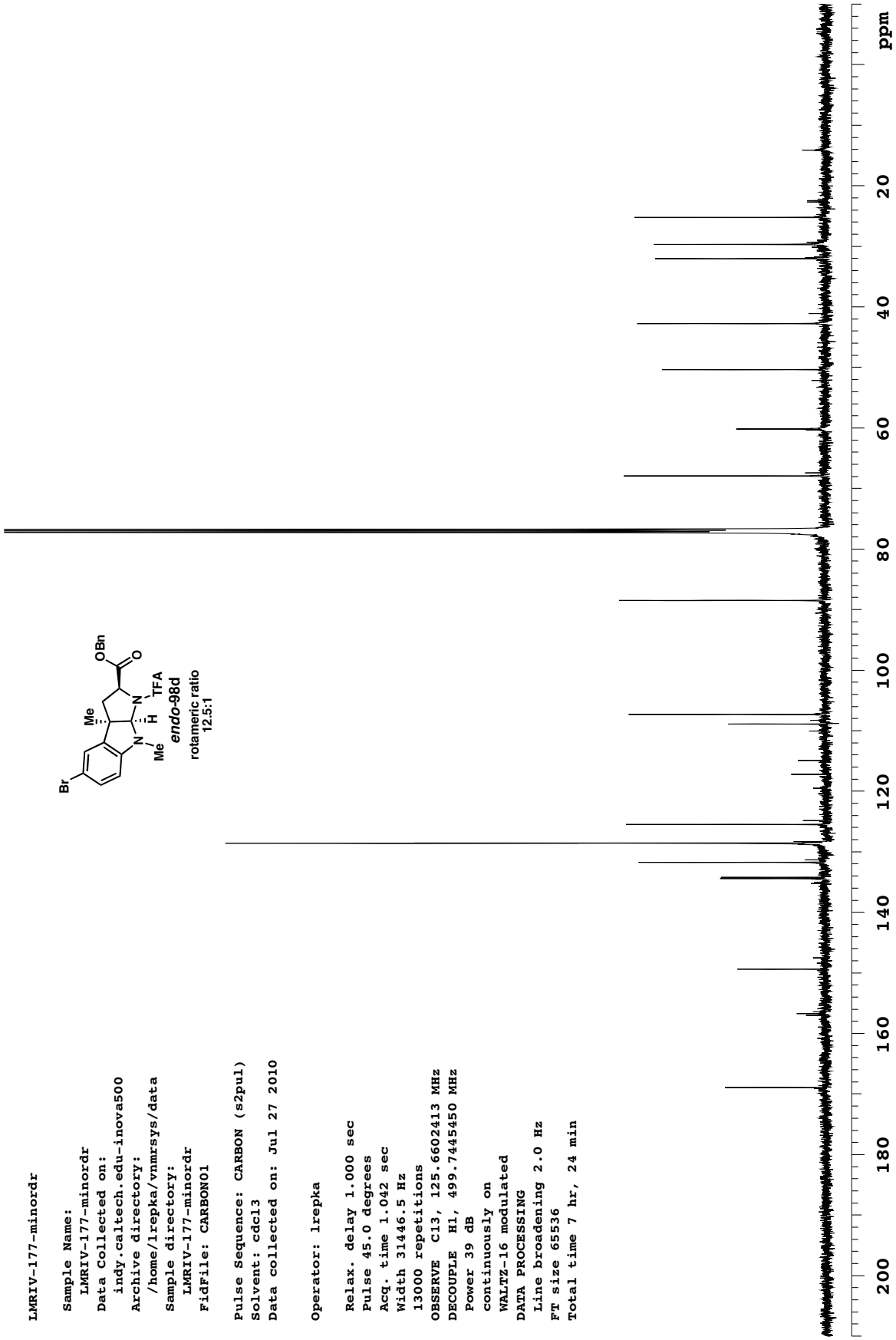



*exo*-98d
rotameric ratio
3.0:1

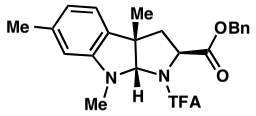



LMRIV-177-majordr-7-31-10
Sample Name:
LMRIV-177-majordr-7-31-10
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-177-majordr-7-31-10
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2010
Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.022 sec
Width 32051.3 Hz
13250 repetitions
OBSERVE C13, 125.6602418 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 28 min
OBn
O
Me
N
TFA
H
N
Me
Br
exo-98d
rotameric ratio
3.0:1
ppm
200
180
160
140
120
100
80
60
40
20

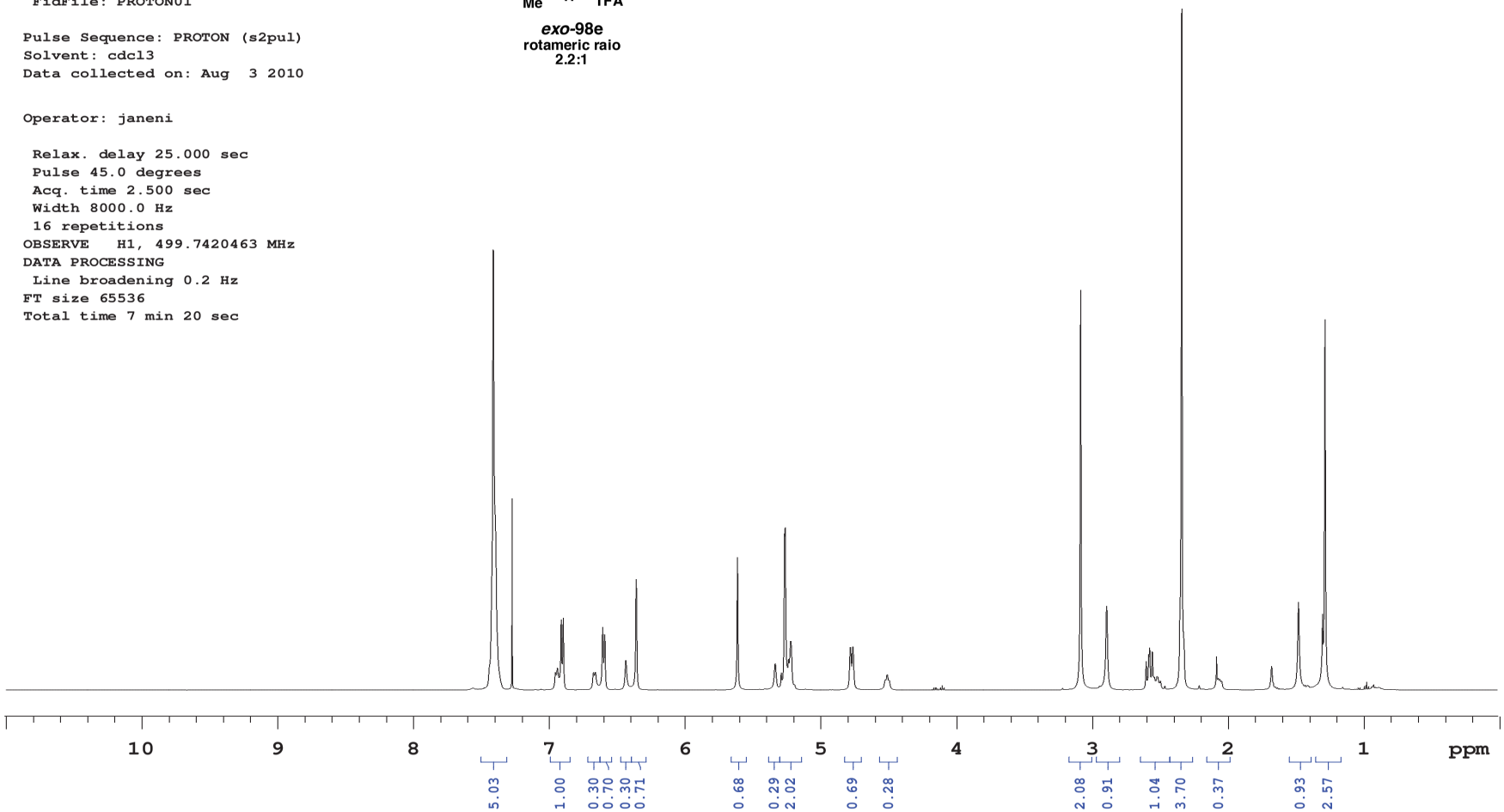

LMRIV-177-minordr-hsqc
Sample Name:
LMRIV-177-minordr-hsqc
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-177-minordr-hsqc
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 1 2010
Operator: lrepka
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420853 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 36 sec
Br
Me
OBn
O
N
TFA
H
N
Me
endo-98d
rotameric ratio
12.5:1
ppm
1
2
3
4
5
6
7
8
9
10
2.85
0.44
0.22
1.01
0.11
0.33
1.11
2.89
0.99
0.92
0.92
0.08
0.08
0.08
0.13
0.91
0.93
0.09
0.90
0.11
2.95
2.97


LMRIV-177-minordr
Sample Name:
LMRIV-177-minordr
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-177-minordr
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2010
Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602413 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 7 hr, 24 min
OBn
O
Me
N
H
TFA
N
Me
Br
endo-98d
rotameric ratio
12.5:1
ppm
200
180
160
140
120
100
80
60
40
20

```
JN-1-215-majordiast2

 Sample Name:
   JN-1-215-majordiast2
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/janeni/vnmrsys/data
 Sample directory:
   JN-1-215-majordiast2
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug  3 2010

Operator: janeni

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
OBSERVE   H1, 499.7420463 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec
```




***exo*-98e**
**rotameric raio**
**2.2:1**



JN-1-215-majordiast2

Sample Name:
JN-1-215-majordiast2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-215-majordiast2
FidFile: CARBON01

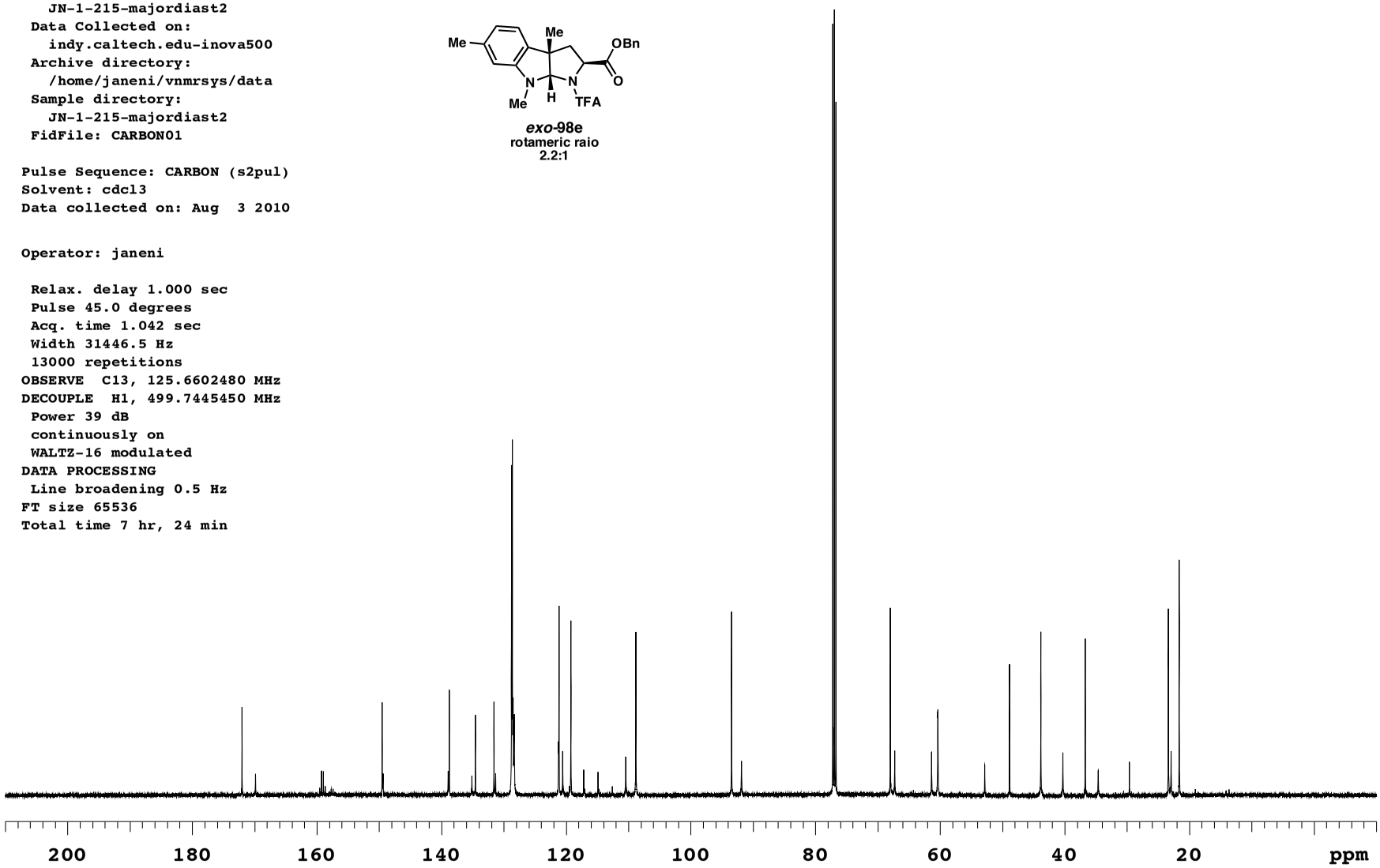
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 3 2010

Operator: janeni

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602480 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 24 min




*exo*-98e
rotameric raio
2.2:1



JN-1-215-minordiast2

Sample Name:
JN-1-215-minordiast2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-215-minordiast2
FidFile: PROTON01

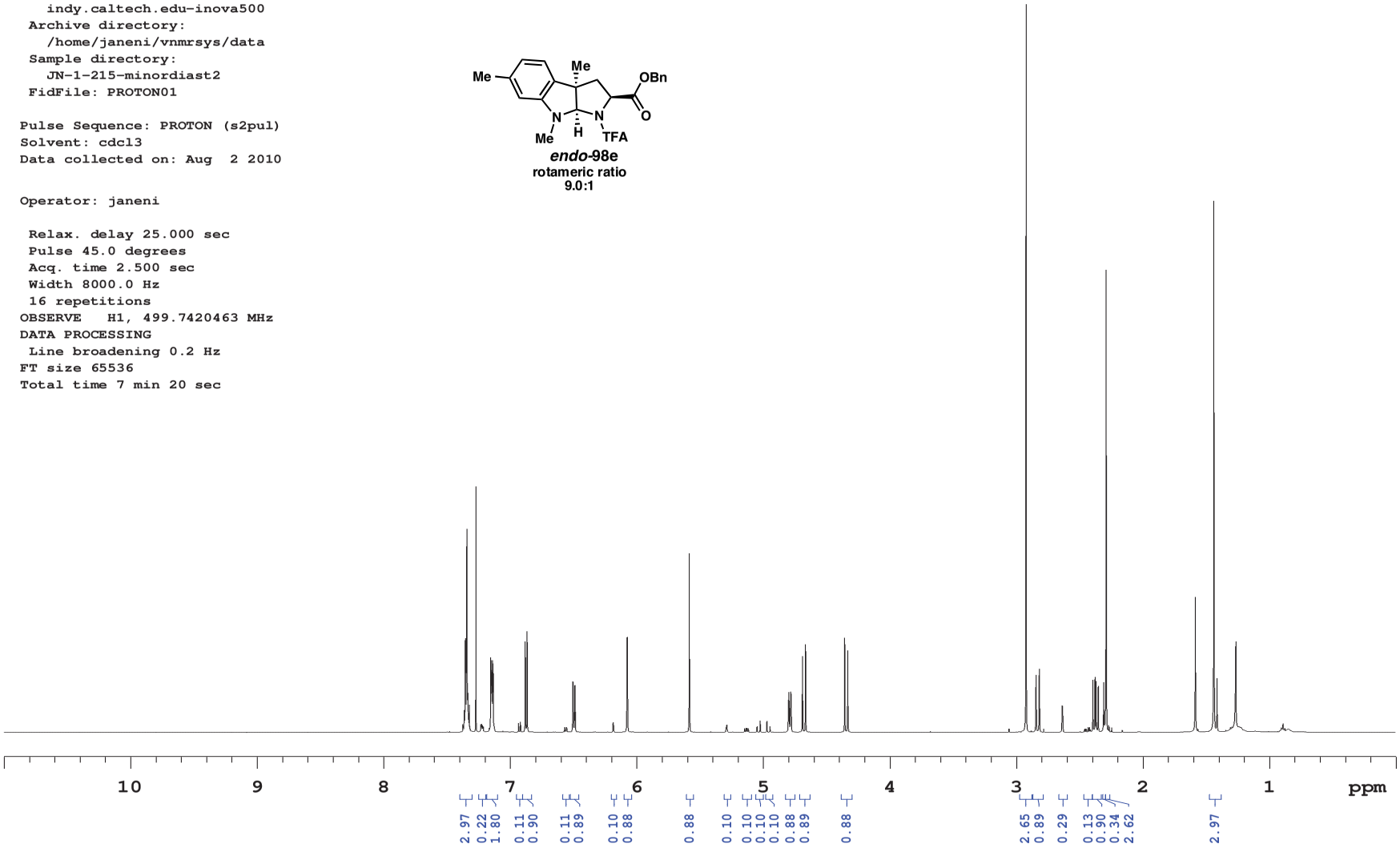
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2010

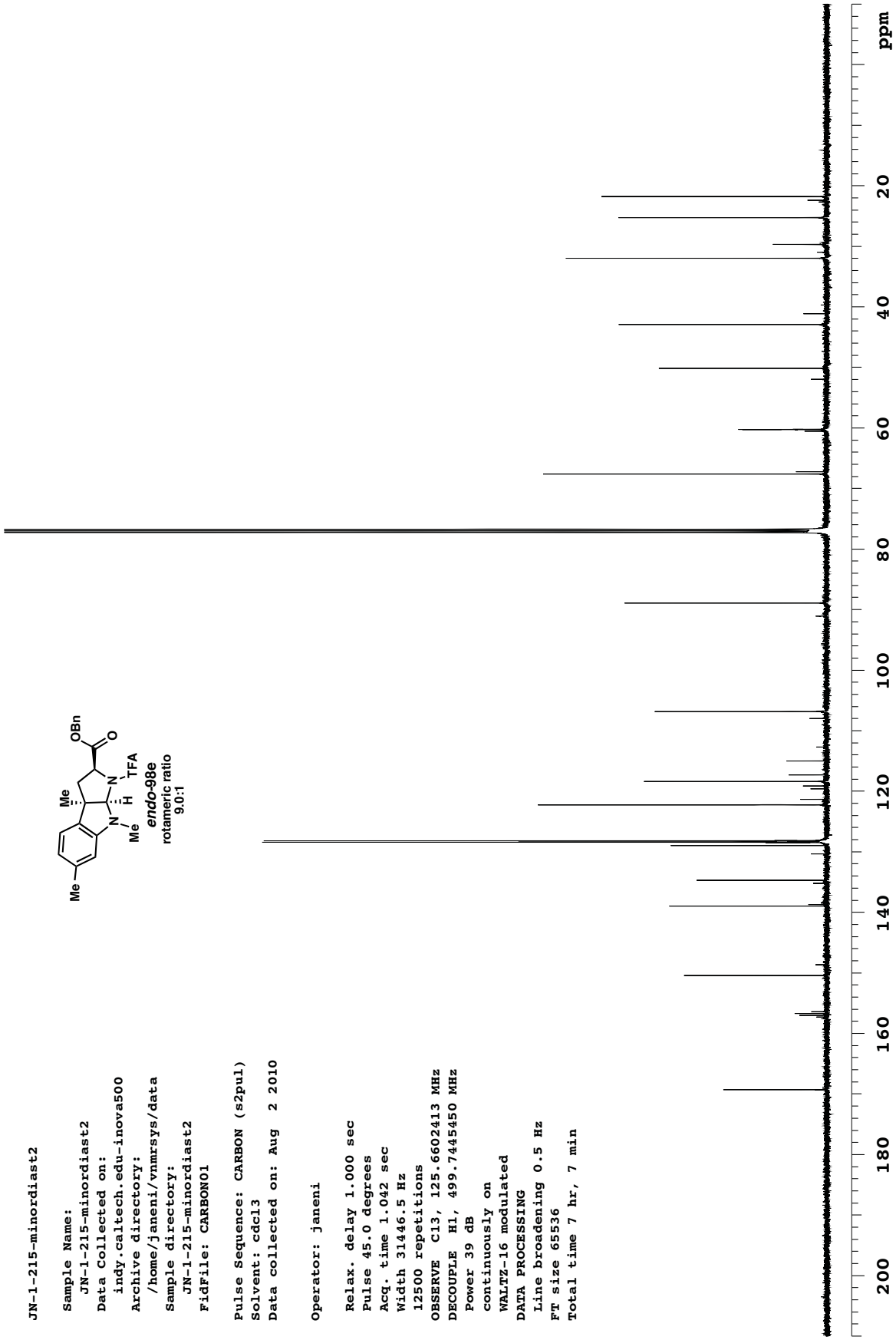
Operator: janeni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec




*endo*-98e
rotameric ratio
9.0:1



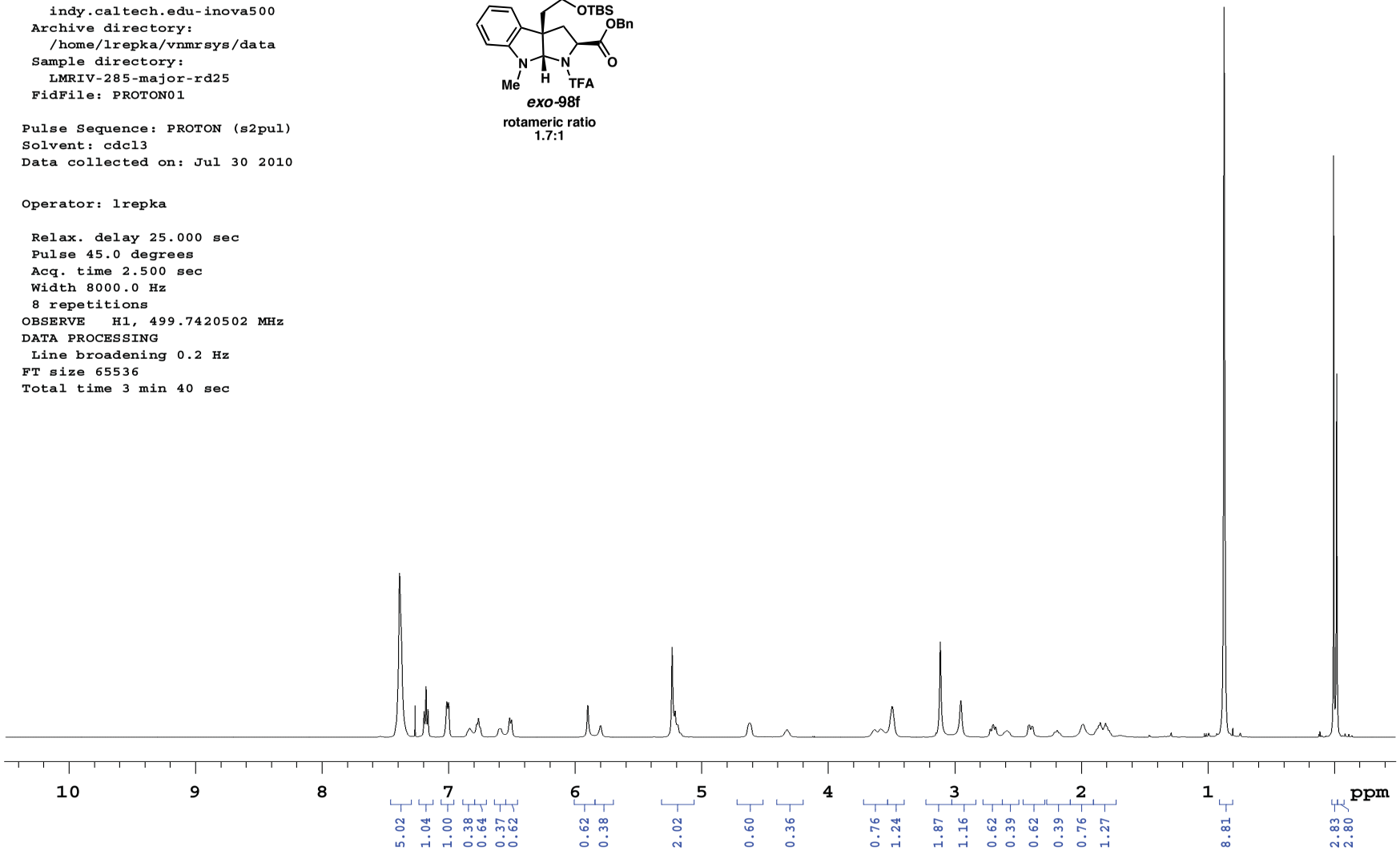

JN-1-215-minordiast2
Sample Name:
JN-1-215-minordiast2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-215-minordiast2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2010
Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
12500 repetitions
OBSERVE C13, 125.6602413 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 7 min
OBn
O
Me
N
H
TFA
N
Me
Me
endo-98e
rotameric ratio
9.0:1
ppm
200
180
160
140
120
100
80
60
40
20

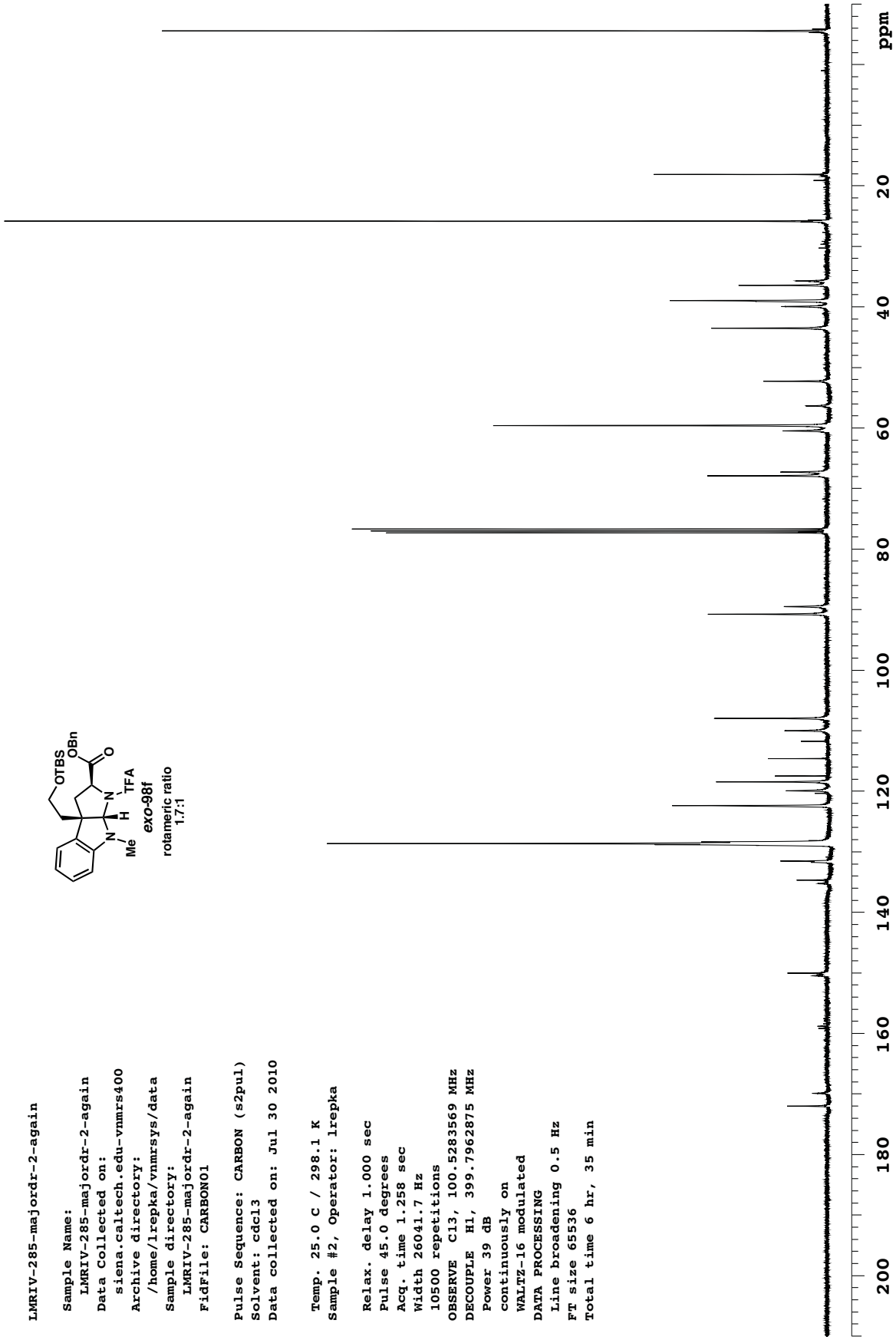
```
LMRIV-285-major-rd25

 Sample Name:
   LMRIV-285-major-rd25
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-285-major-rd25
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010

Operator: lrepka

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
OBSERVE   H1, 499.7420502 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 3 min 40 sec
```



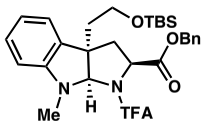

*exo*-98f

rotameric ratio
1.7:1

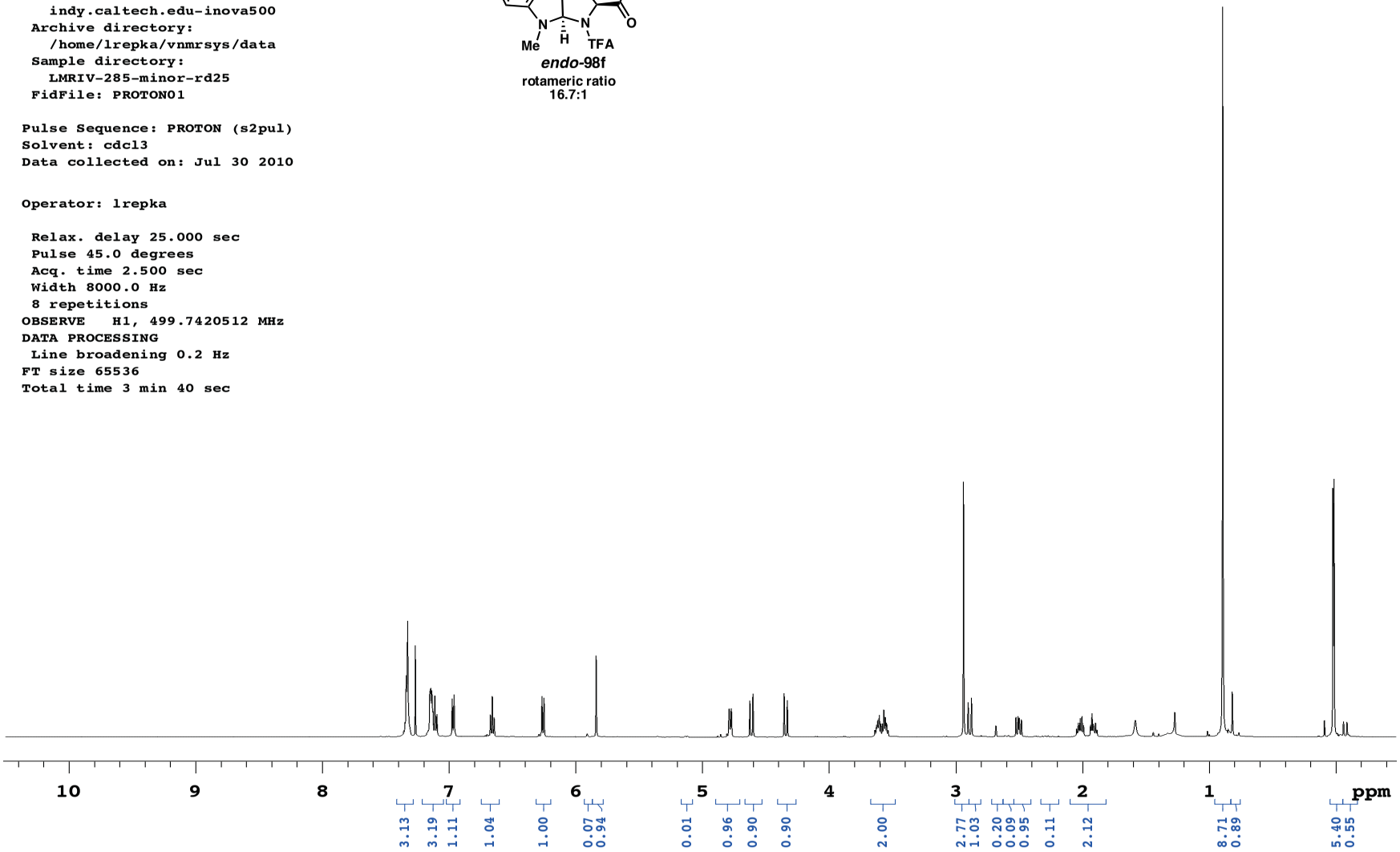


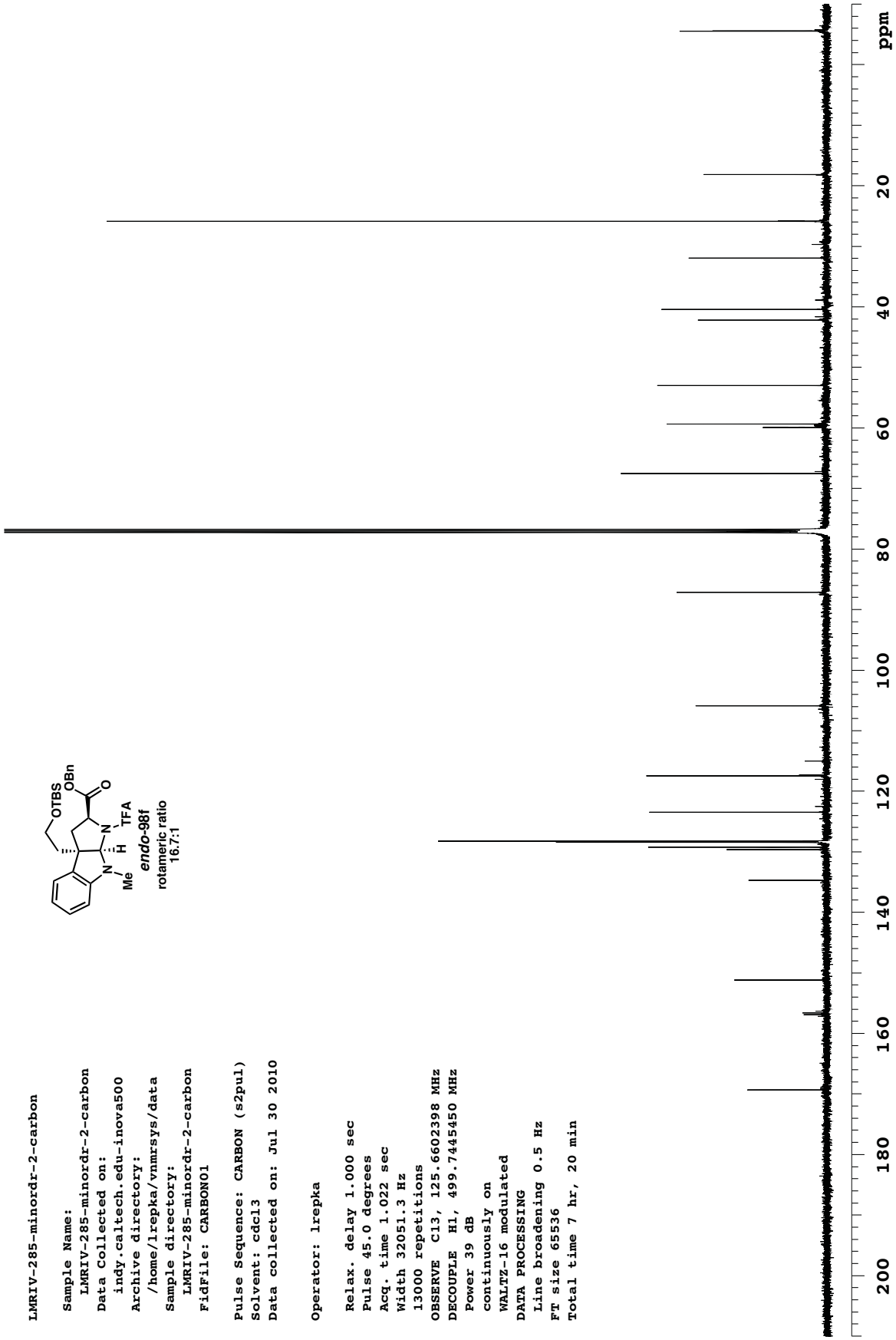

LMRIV-285-majordr-2-again
Sample Name:
LMRIV-285-majordr-2-again
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-285-majordr-2-again
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010
Temp. 25.0 C / 298.1 K
Sample #2, Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.258 sec
Width 26041.7 Hz
10500 repetitions
OBSERVE C13, 100.5283569 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 6 hr, 35 min
OTBS
OBn
O
N
H
TFA
N
Me
exo-98f
rotameric ratio
1.7:1
200
180
160
140
120
100
80
60
40
20
ppm

```
LMRIV-285-minor-rd25

 Sample Name:
   LMRIV-285-minor-rd25
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-285-minor-rd25
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010

Operator: lrepka

 Relax. delay 25.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
OBSERVE   H1, 499.7420512 MHz
DATA PROCESSING
 Line broadening 0.2 Hz
FT size 65536
Total time 3 min 40 sec
```

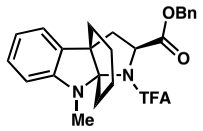



*endo*-98f
rotameric ratio
16.7:1

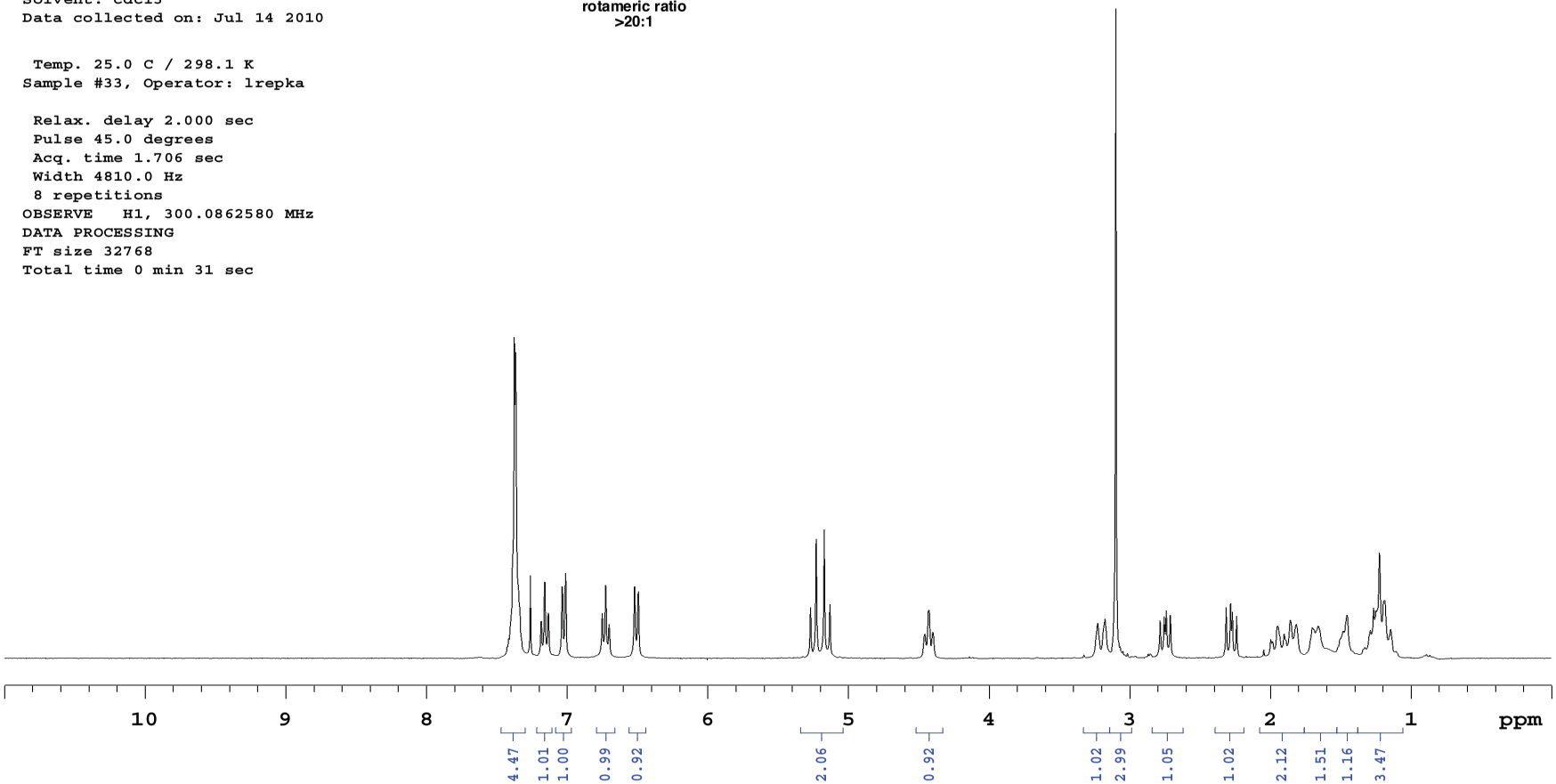


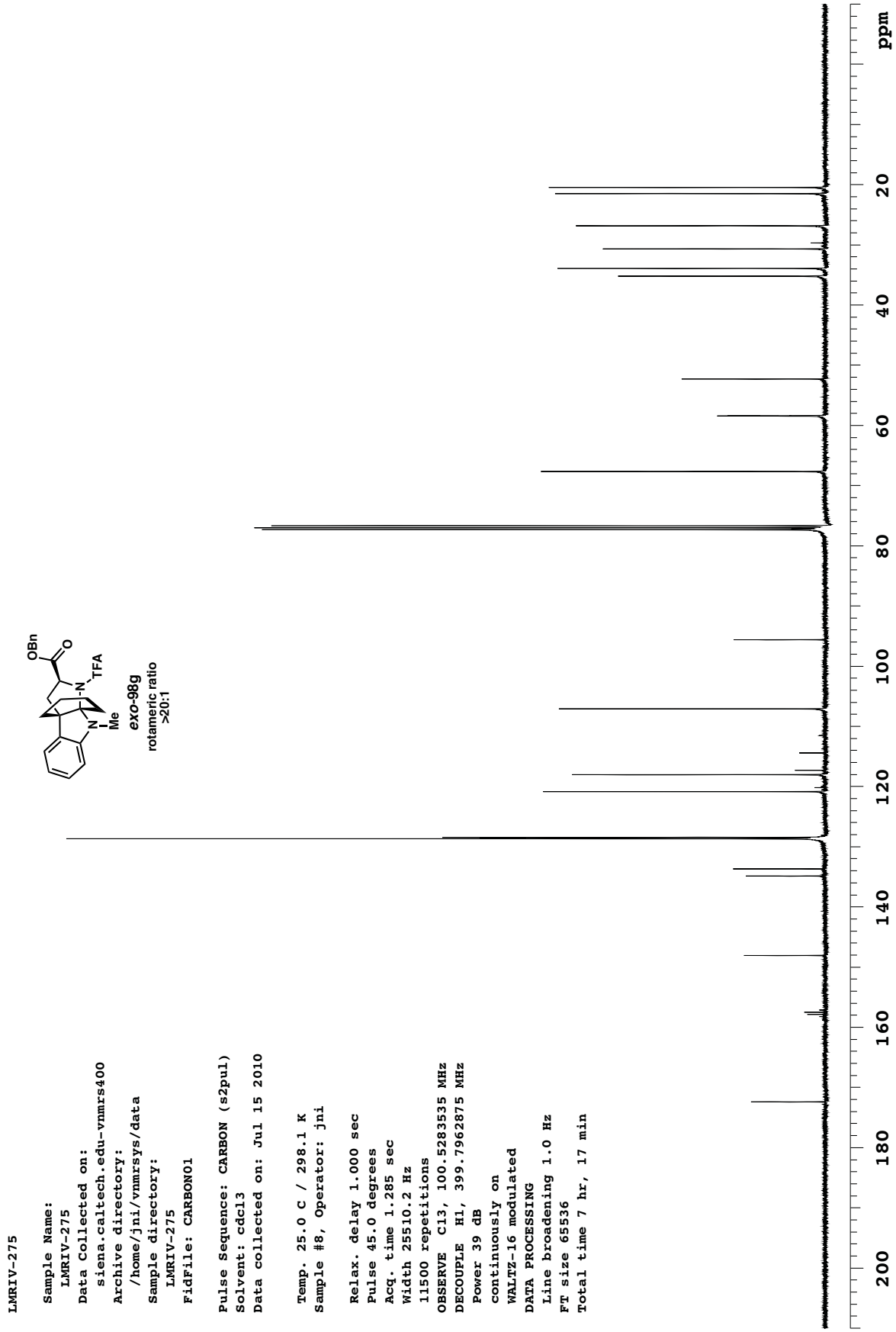

LMRIV-285-minordr-2-carbon
Sample Name:
LMRIV-285-minordr-2-carbon
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-285-minordr-2-carbon
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2010
Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.022 sec
Width 32051.3 Hz
13000 repetitions
OBSERVE C13, 125.6602398 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 20 min
OTBS
OBn
O
N
H
TFA
N
Me
endo-98f
rotameric ratio
16.7:1
ppm
200
180
160
140
120
100
80
60
40
20

```
LMRIV-275-pentanewash

 Sample Name:
   LMRIV-275-pentanewash
 Data Collected on:
   hg3.caltech.edu-mercury300
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-275-pentanewash
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 14 2010

 Temp. 25.0 C / 298.1 K
Sample #33, Operator: lrepka

 Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 1.706 sec
 Width 4810.0 Hz
 8 repetitions
OBSERVE   H1, 300.0862580 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 31 sec
```




***exo*-98g**
**rotameric ratio >20:1**




LMRIV-275
Sample Name:
LMRIV-275
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
LMRIV-275
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 15 2010
Temp. 25.0 C / 298.1 K
Sample #8, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
11500 repetitions
OBSERVE C13, 100.5283535 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 17 min
OBn
O
N
TFA
N
Me
exo-98g
rotameric ratio
>20:1
ppm
200
180
160
140
120
100
80
60
40
20

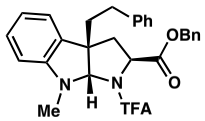
LMRIV-267-major diastereomer

Sample Name:
LMRIV-267-majordr
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-267-majordr
FidFile: PROTON01

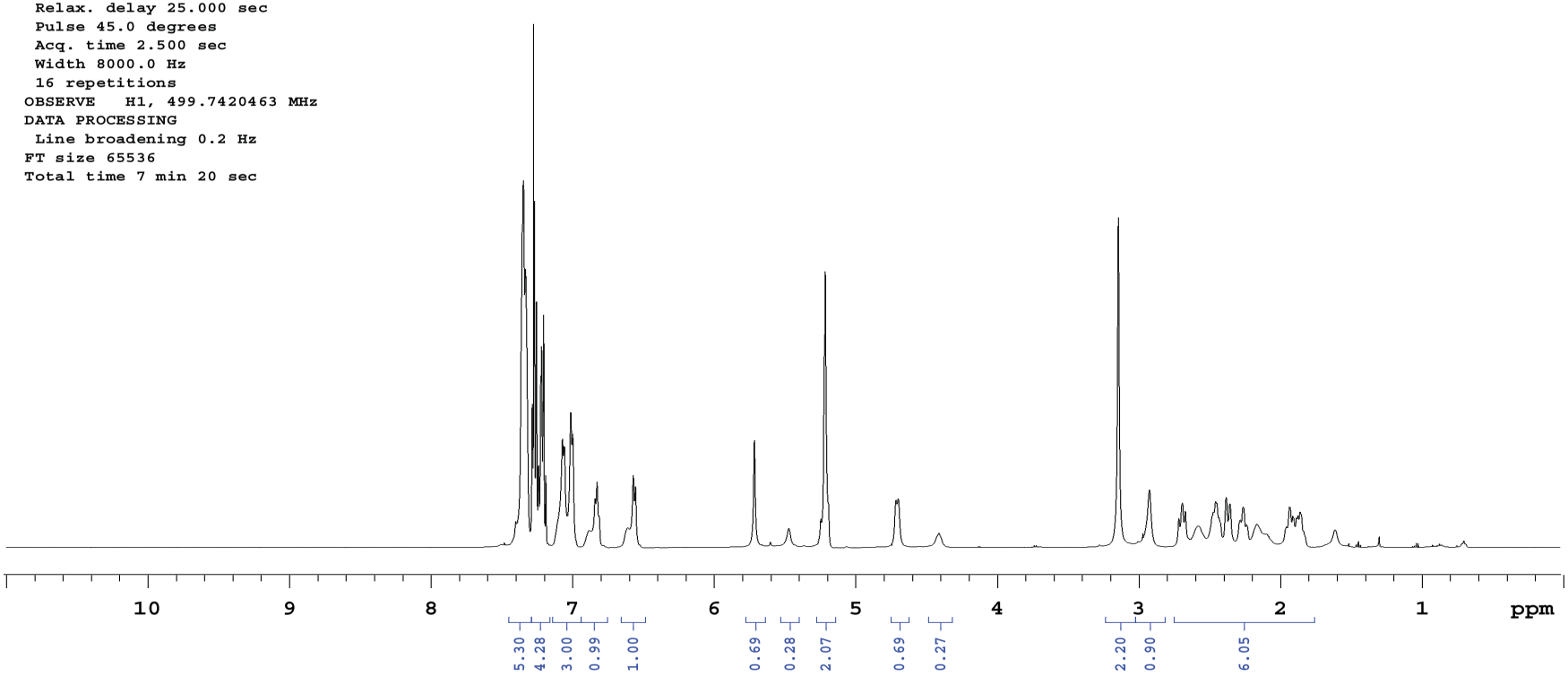
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec




*exo*-98h
rotameric ratio
2.6:1



LMRIV-267-majorshigemi

Sample Name:
LMRIV-267-majorshigemi
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-267-majorshigemi
FidFile: CARBON01

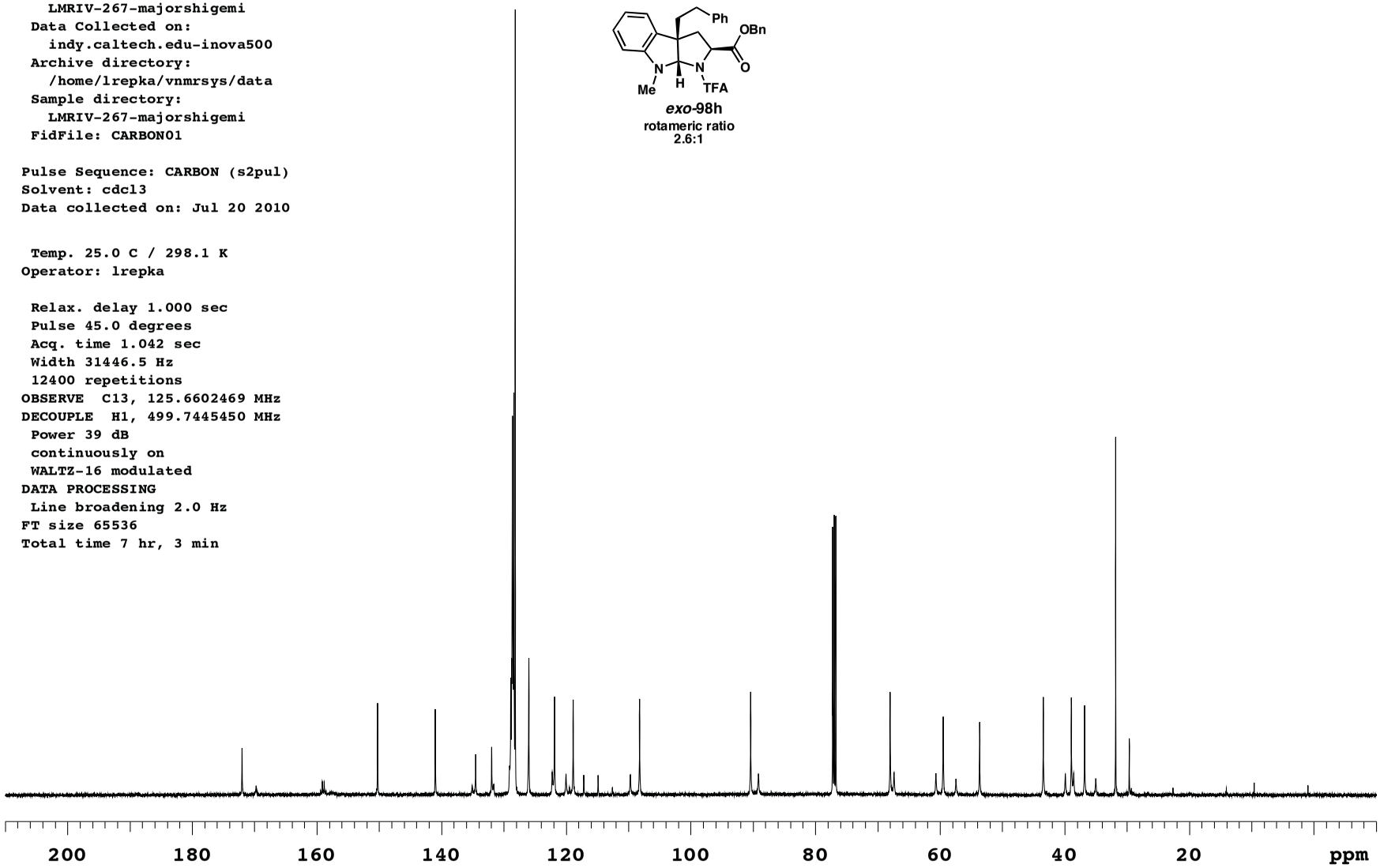
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2010

Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
12400 repetitions
OBSERVE C13, 125.6602469 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 7 hr, 3 min




*exo*-98h
rotameric ratio
2.6:1



LMRIV-267-minor3

Sample Name:
LMRIV-267-minor3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRIV-267-minor3_20100717_01
FidFile: data_s2pul_001

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Jul 17 2010

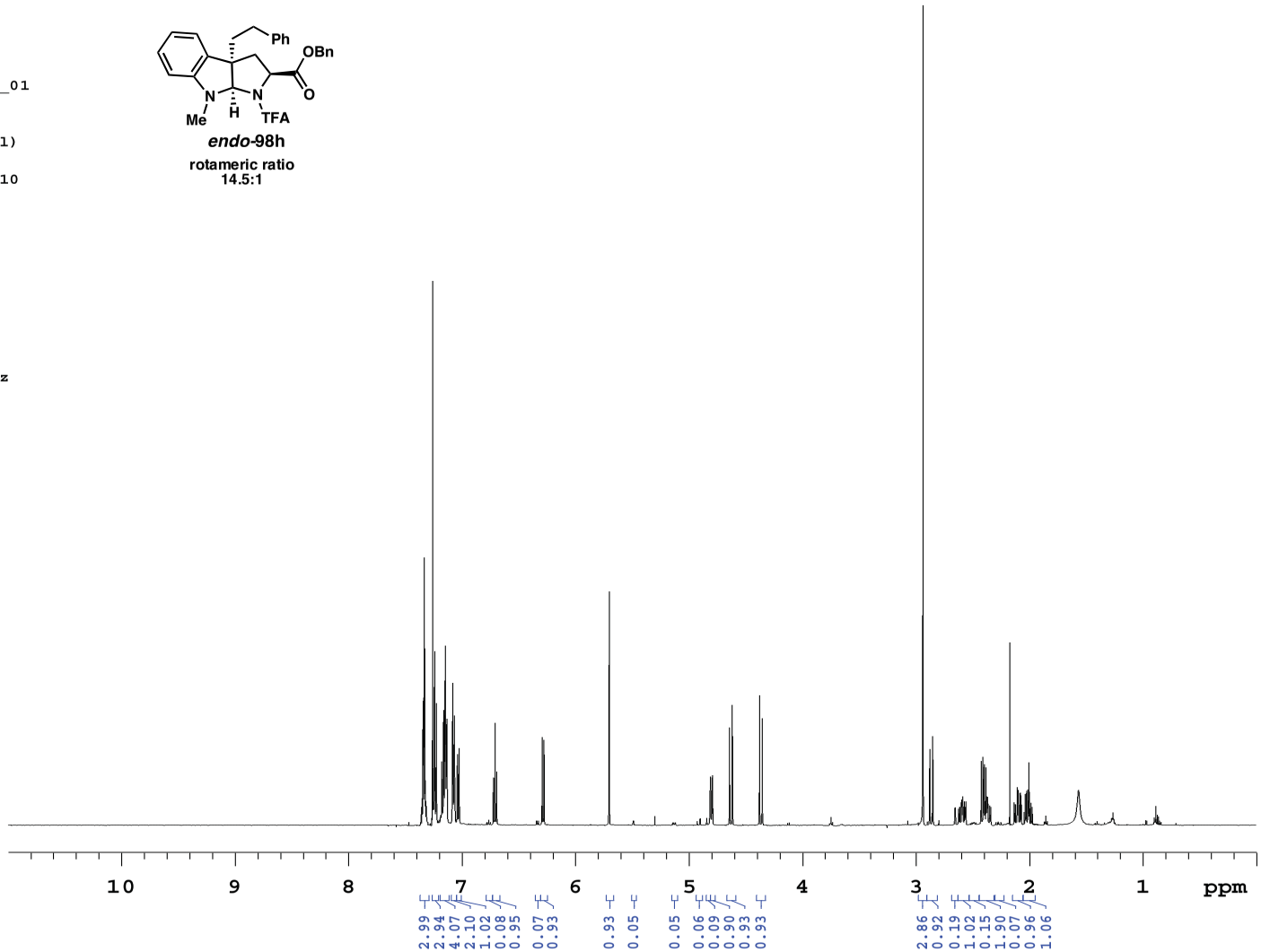
Temp. 25.0 C / 298.1 K
Operator: lrepka

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 7996.0 Hz
8 repetitions
OBSERVE H1, 499.7420487 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 36 sec




***endo*-98h**

**rotameric ratio 14.5:1**

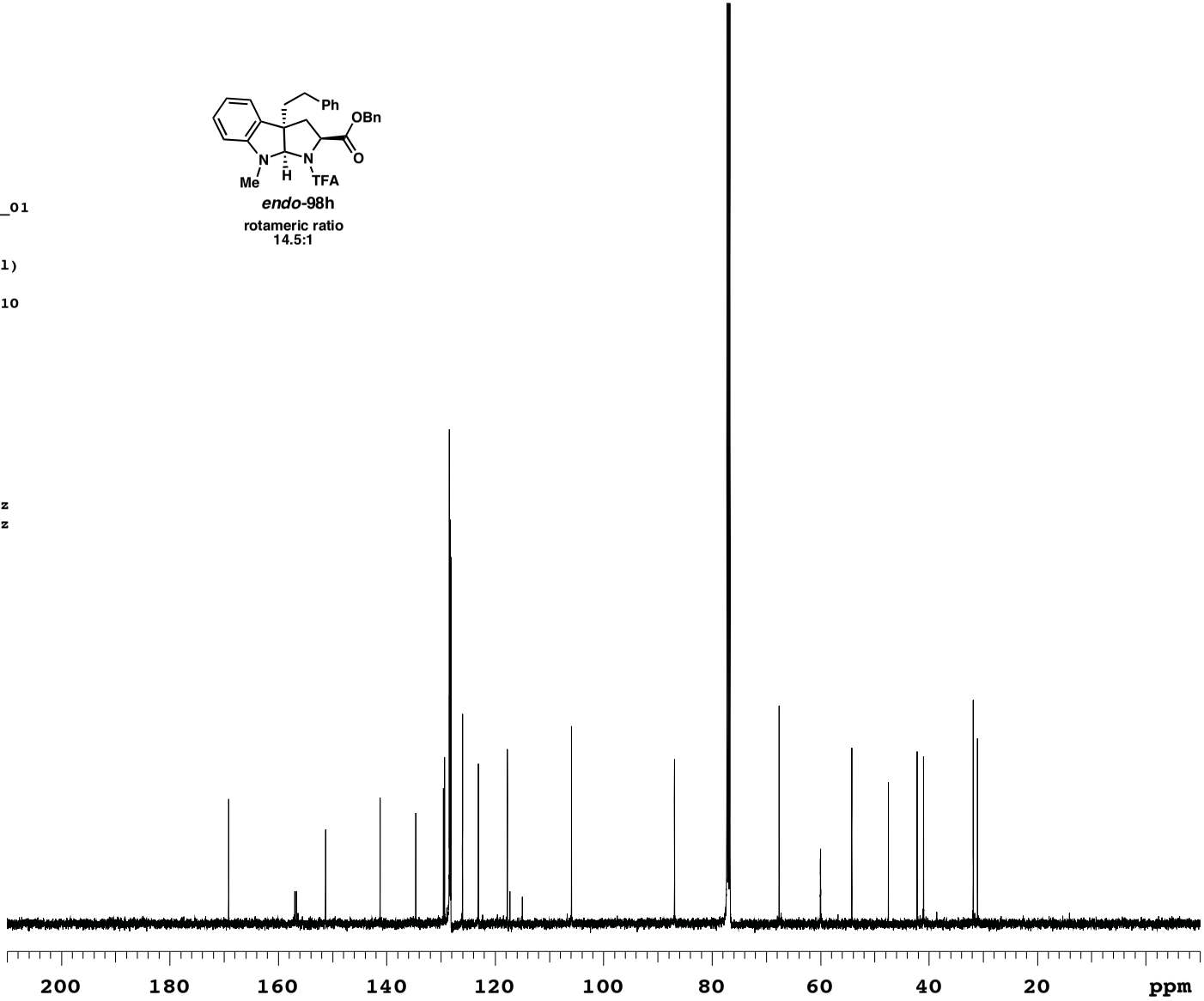


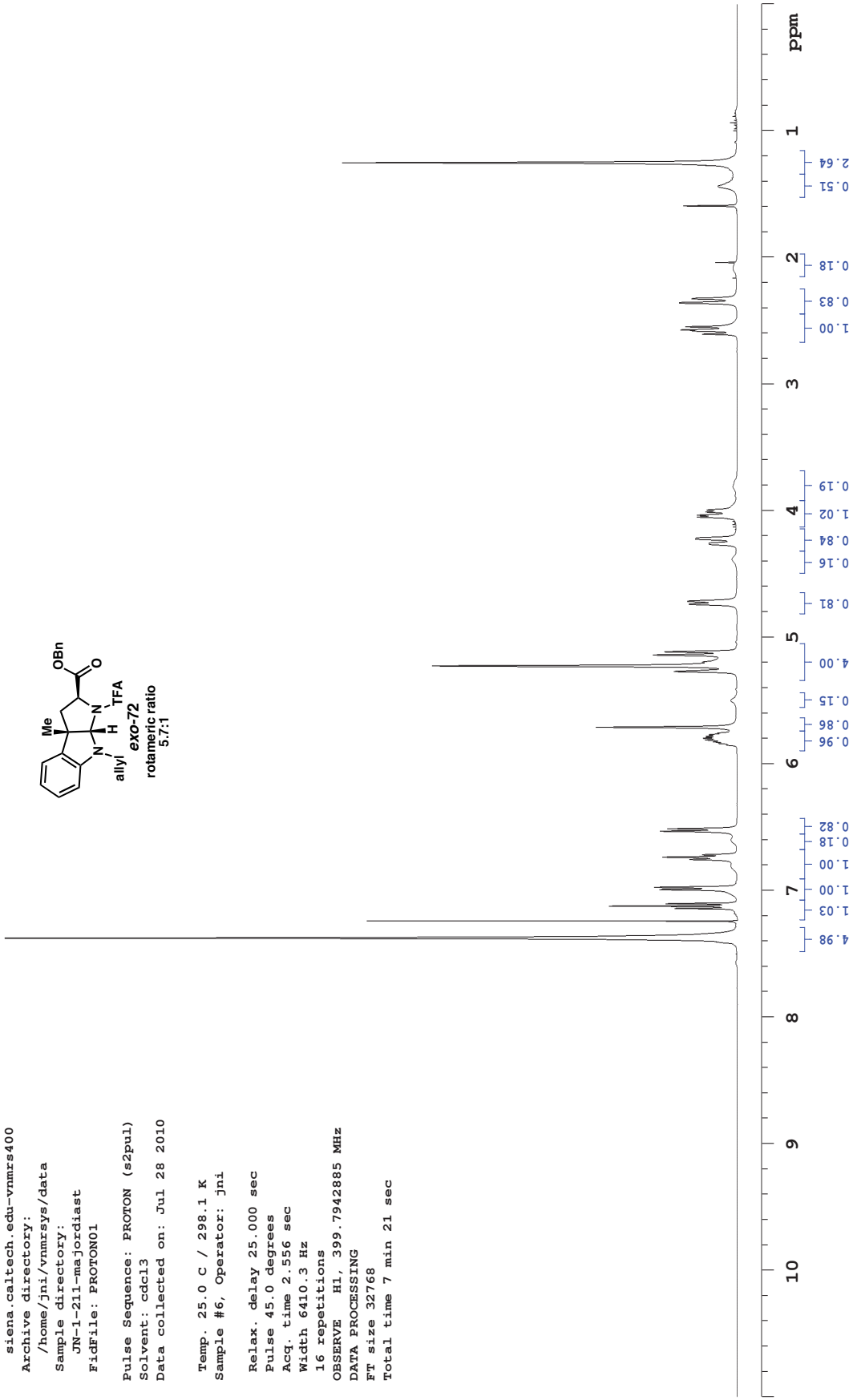
```
LMRIV-267-minor3

 Sample Name:
   LMRIV-267-minor3
 Data Collected on:
   indy.caltech.edu-inova500
 Archive directory:
   /home/lrepka/vnmrsys/data
 Sample directory:
   LMRIV-267-minor3_20100717_01
 FidFile: data_s2pul_002

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Jul 17 2010


 Temp. 25.0 C / 298.1 K
Operator: lrepka

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.043 sec
 Width 31409.5 Hz
 12000 repetitions
OBSERVE  C13, 125.6602386 MHz
DECOUPLE  H1, 499.7445450 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 65536
Total time 6 hr, 50 min
```

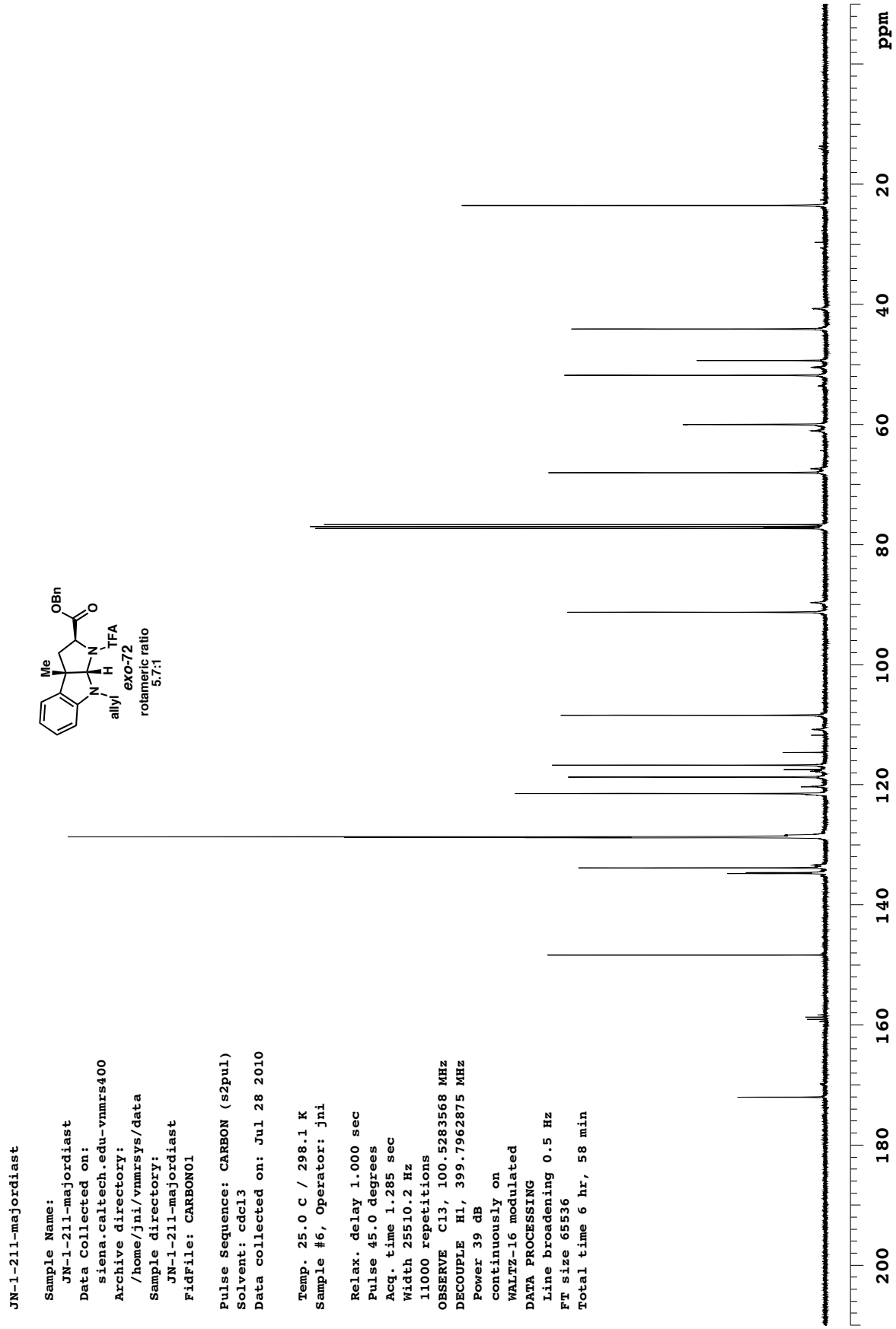



*endo*-98h

rotameric ratio
14.5:1




JN-1-211-majordiast
Sample Name:
JN-1-211-majordiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-211-majordiast
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 28 2010
Temp. 25.0 C / 298.1 K
Sample #6, Operator: jni
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7942885 MHz
DATA PROCESSING
FT size 32768
Total time 7 min 21 sec
OBn
O
Me
N
TFA
H
N
allyl
exo-72
rotameric ratio
5.7:1
ppm
1
2
3
4
5
6
7
8
9
10
2.64
0.51
0.18
0.83
1.00
0.19
1.02
0.84
0.16
0.81
4.00
0.15
0.86
0.96
0.82
0.18
1.00
1.00
1.03
4.98


JN-1-211-majordiast
Sample Name:
JN-1-211-majordiast
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/jni/vnmrsys/data
Sample directory:
JN-1-211-majordiast
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 28 2010
Temp. 25.0 C / 298.1 K
Sample #6, Operator: jni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
width 25510.2 Hz
11000 repetitions
OBSERVE C13, 100.5283568 MHz
DECOUPLE H1, 399.7962875 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 6 hr, 58 min
OBn
O
Me
N
TFA
H
N
allyl
exo-72
rotameric ratio
5.7:1
ppm
200
180
160
140
120
100
80
60
40
20

JN-1-211-minordiast

Sample Name:
JN-1-211-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-211-minordiast
FidFile: PROTON01

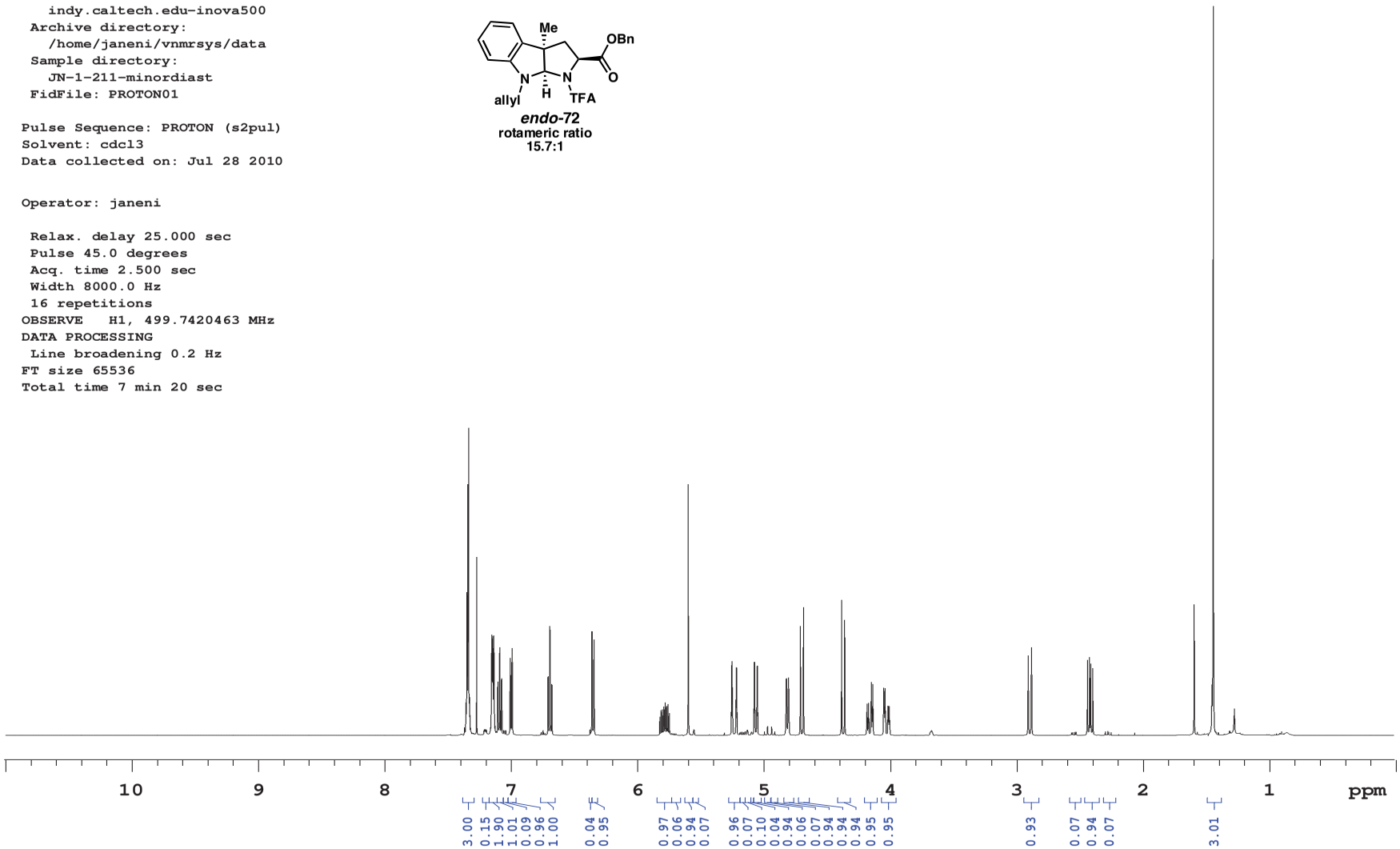
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 28 2010

Operator: janeni

Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7420463 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 7 min 20 sec




*endo*-72
rotameric ratio
15.7:1



JN-1-211-minordiast

Sample Name:
JN-1-211-minordiast
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-1-211-minordiast
FidFile: CARBON01

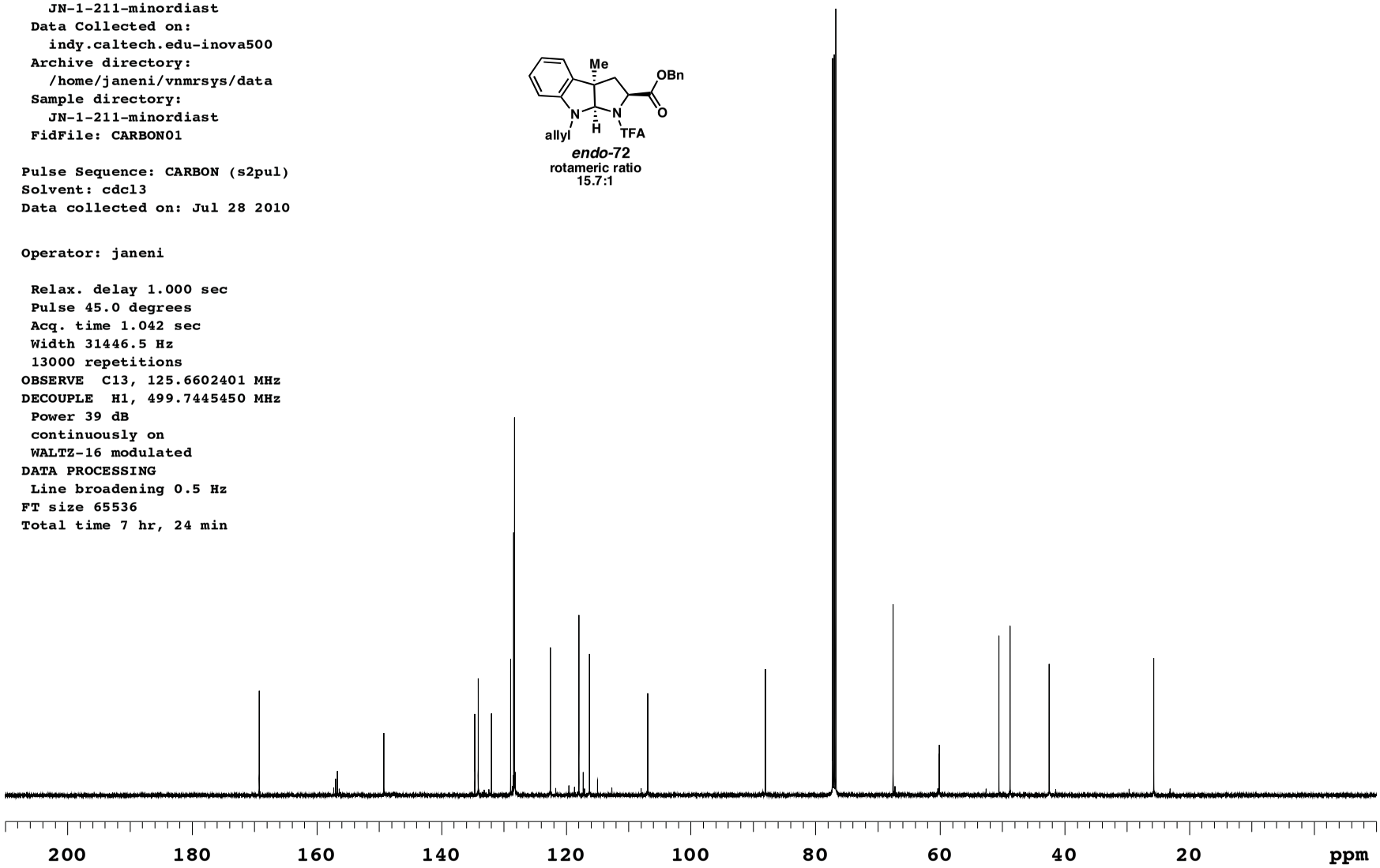
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 28 2010

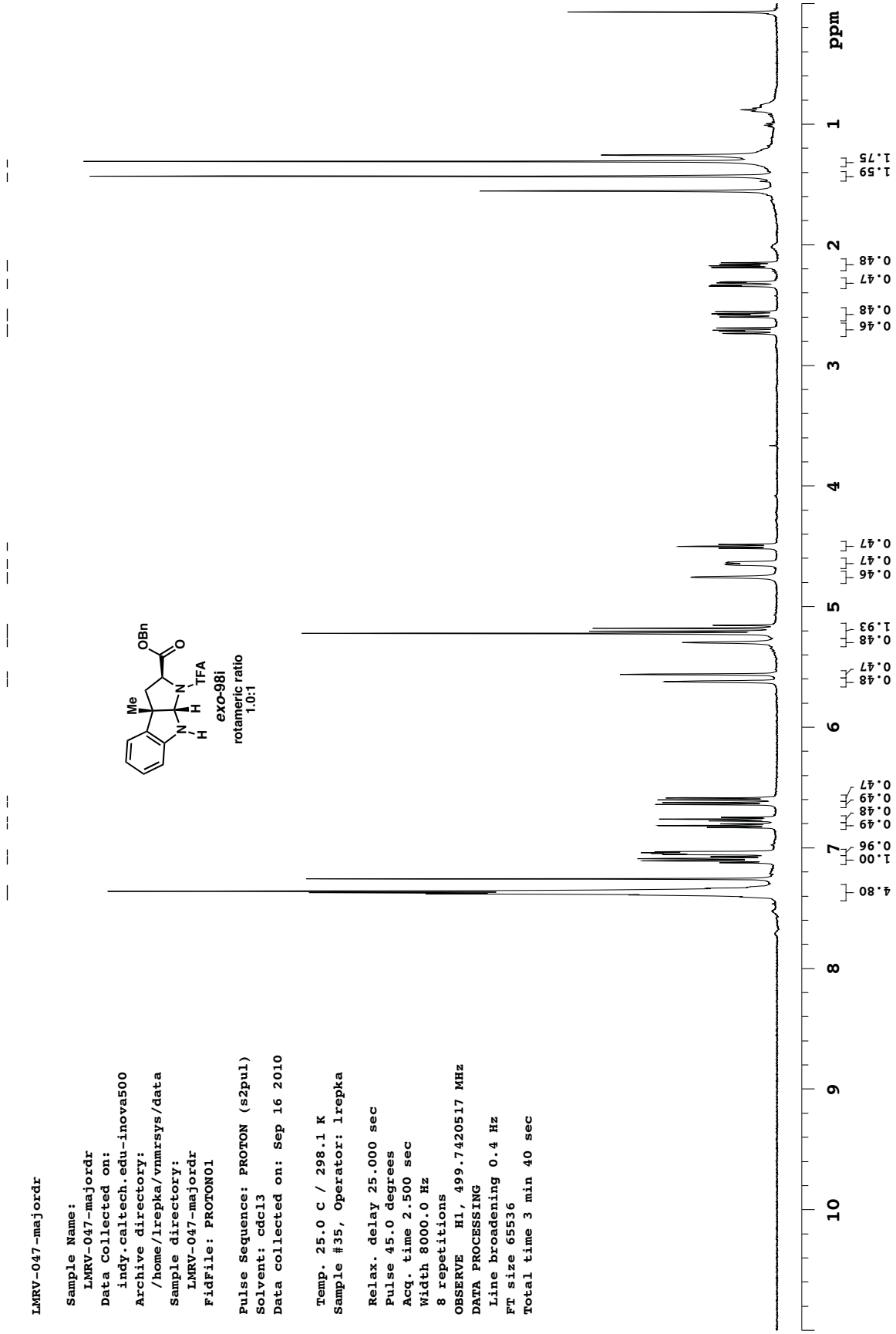
Operator: janeni

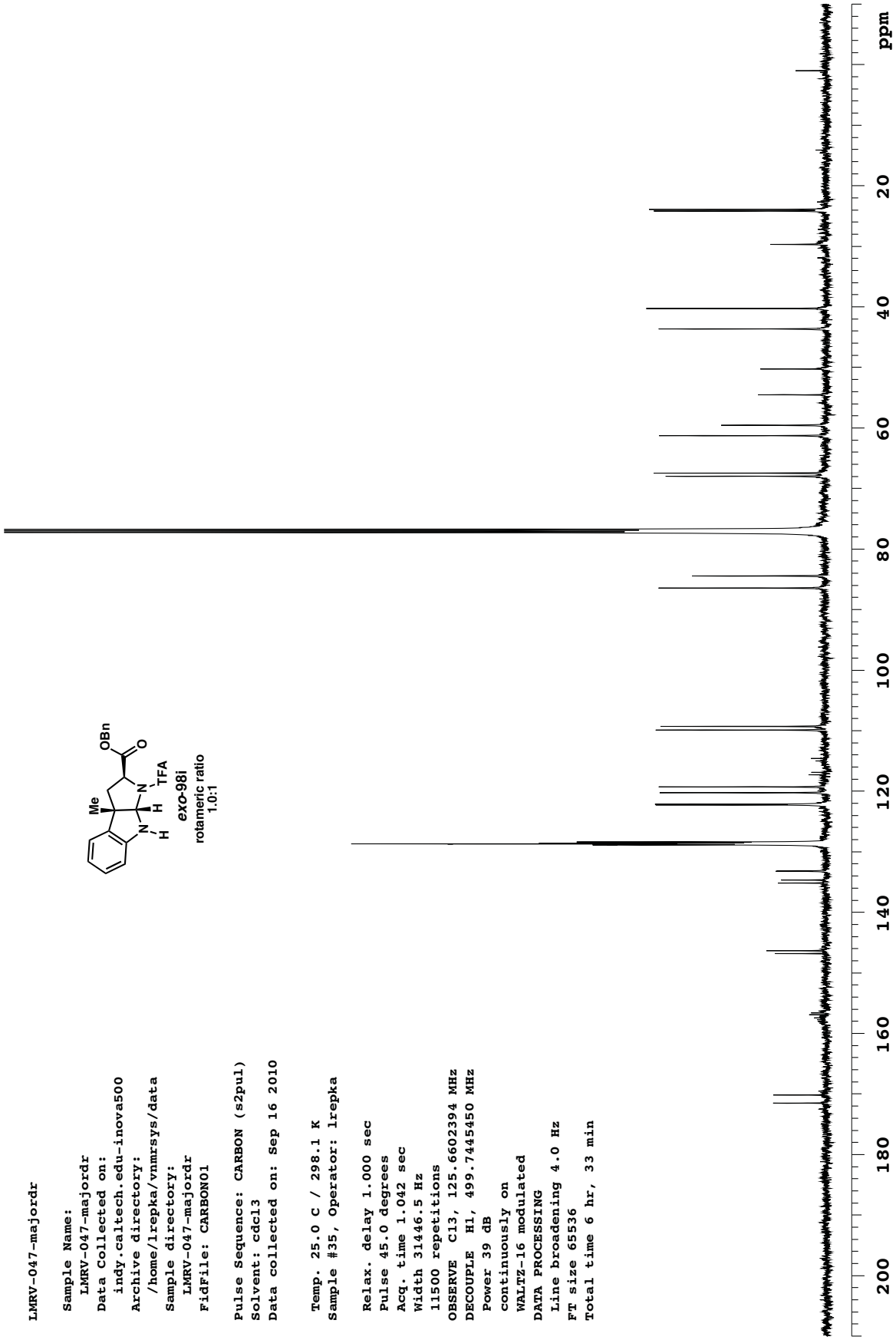
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
13000 repetitions
OBSERVE C13, 125.6602401 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 7 hr, 24 min

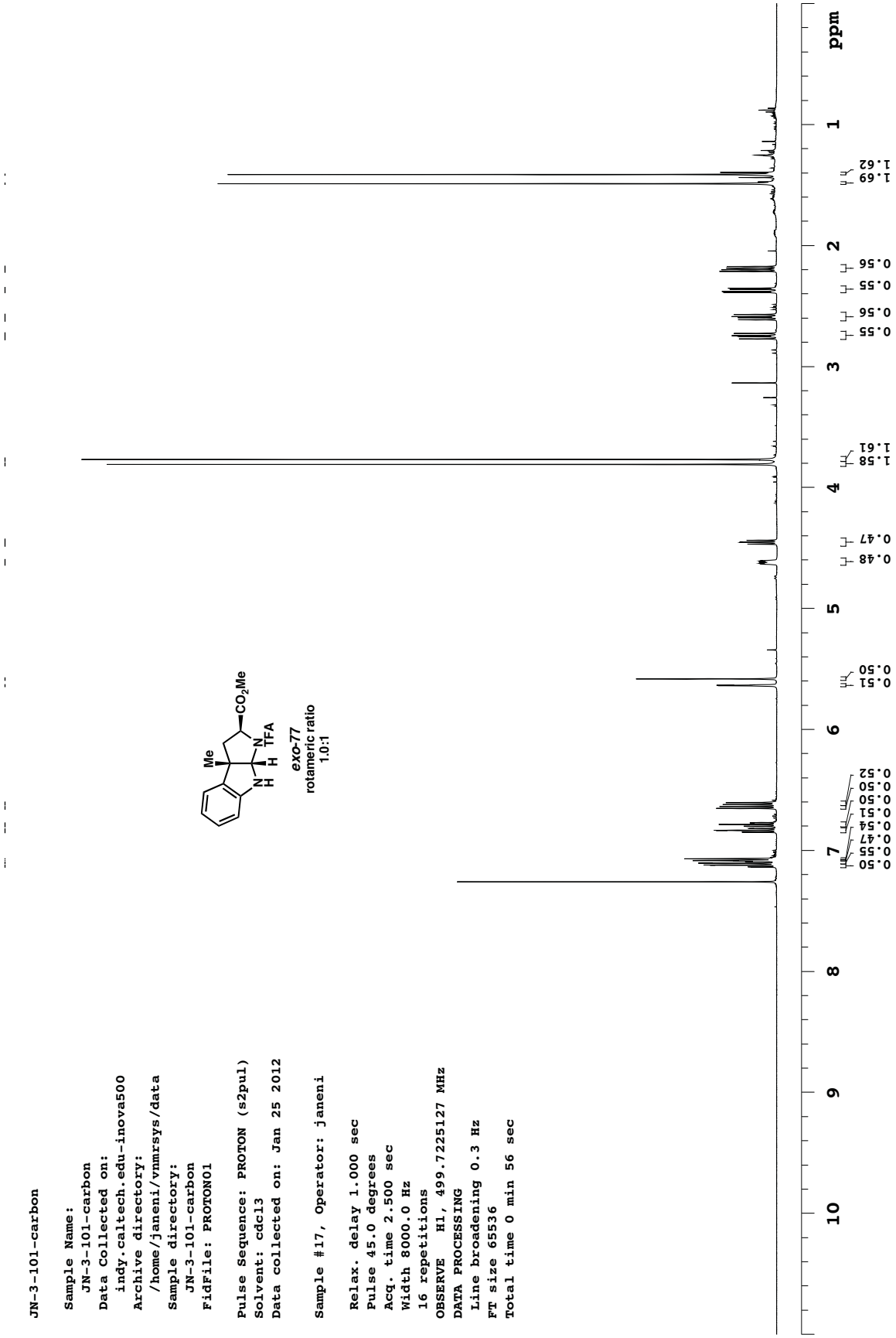


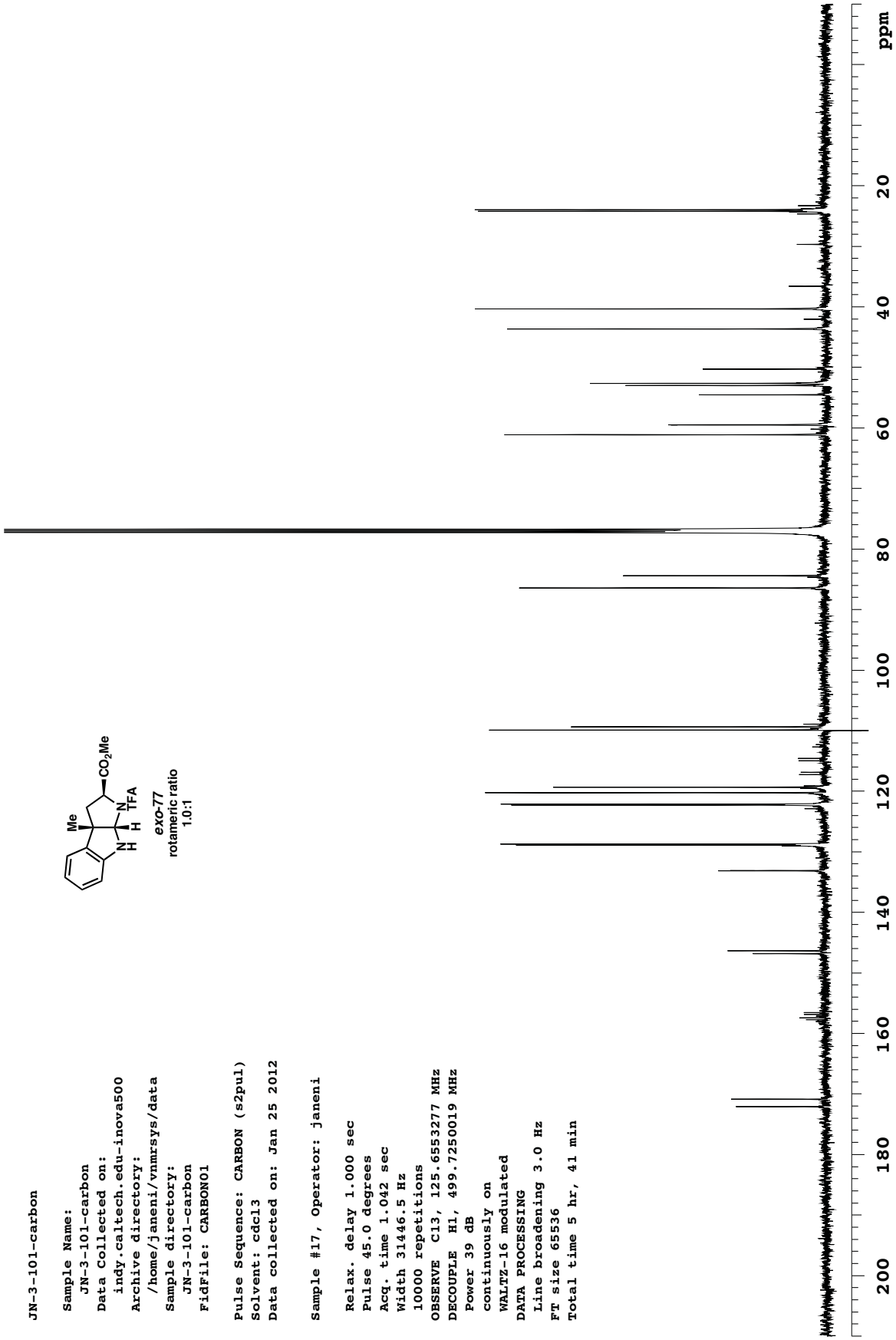

*endo*-72
rotameric ratio
15.7:1

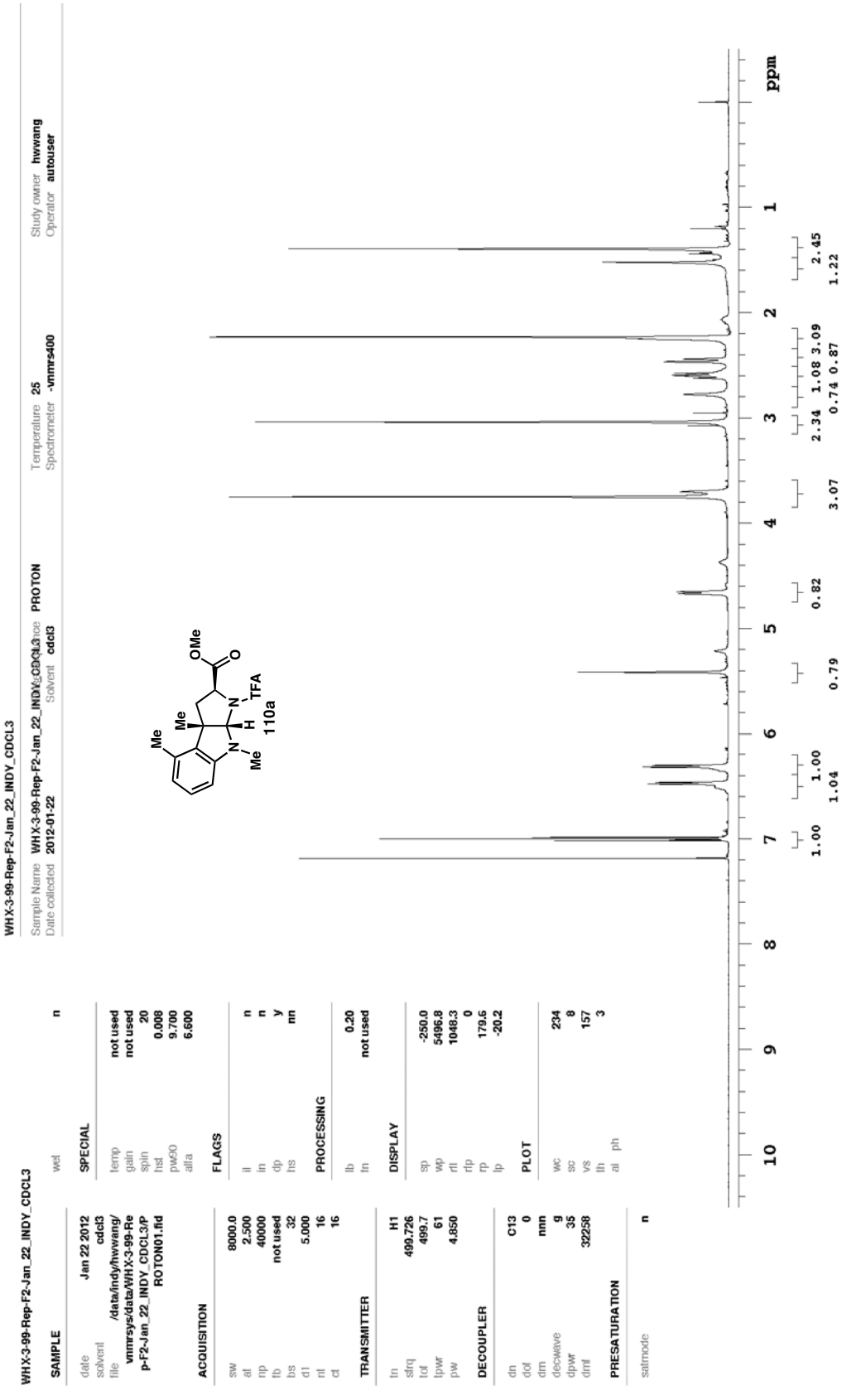



LMRV-047-majordr
Sample Name:
LMRV-047-majordr
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRV-047-majordr
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 16 2010
Temp. 25.0 C / 298.1 K
Sample #35, Operator: lrepka
Relax. delay 25.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7420517 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 3 min 40 sec
OBn
O
Me
N
TFA
H
H
N
H
exo-98i
rotameric ratio
1.0:1
ppm
1
2
3
4
5
6
7
8
9
10
1.75
1.59
0.48
0.47
0.48
0.46
0.47
0.47
0.46
1.93
0.48
0.47
0.48
0.47
0.49
0.48
0.49
0.96
1.00
4.80


LMRV-047-majordr
Sample Name:
LMRV-047-majordr
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/lrepka/vnmrsys/data
Sample directory:
LMRV-047-majordr
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 16 2010
Temp. 25.0 C / 298.1 K
Sample #35, Operator: lrepka
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
11500 repetitions
OBSERVE C13, 125.6602394 MHz
DECOUPLE H1, 499.7445450 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 65536
Total time 6 hr, 33 min
Me
OBn
O
N
H
TFA
N
H
exo-98i
rotameric ratio
1.0:1
ppm
200
180
160
140
120
100
80
60
40
20


JN-3-101-carbon
Sample Name:
JN-3-101-carbon
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-3-101-carbon
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2012
Sample #17, Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7225127 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 56 sec
Me
CO2Me
N
H
N
H
TFA
exo-77
rotameric ratio
1.0:1
ppm
10
9
8
7
6
5
4
3
2
1
0.50
0.55
0.47
0.54
0.51
0.50
0.50
0.52
0.51
0.50
0.48
0.47
1.58
1.61
0.55
0.56
0.55
0.56
1.69
1.62


JN-3-101-carbon
Sample Name:
JN-3-101-carbon
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/janeni/vnmrsys/data
Sample directory:
JN-3-101-carbon
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2012
Sample #17, Operator: janeni
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
10000 repetitions
OBSERVE C13, 125.6553277 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 5 hr, 41 min
Me
CO2Me
N
H
N
H
TFA
exo-77
rotameric ratio
1.0:1
ppm
200
180
160
140
120
100
80
60
40
20


WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3
Sample Name WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3
Date collected 2012-01-22
Solvent cdcl3
PROTON
Temperature 25
Spectrometer -vnmrs400
Study owner hwwang
Operator autouser
OMe
Me
Me
Me
N
N
H
TFA
110a
ppm
10
9
8
7
6
5
4
3
2
1
1.00
1.04
1.00
0.79
0.82
3.07
2.34
1.08
3.09
0.74
0.87
2.45
1.22
WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3
SAMPLE
date Jan 22 2012
solvent cdcl3
file /data/indy/hwwang/vnmrsys/data/WHX-3-99-Re p-F2-Jan_22_INDY_CDCL3/P ROTON01.fid
ACQUISITION
sw 8000.0
at 2.500
np 40000
fb not used
bs 32
d1 5.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 499.726
tof 499.7
tpwr 61
pw 4.850
DECOUPLER
dn C13
dof 0
dm nnn
decwave g
dpwr 35
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pwx0 9.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
ln not used
DISPLAY
sp -250.0
wp 5496.8
rf1 1048.3
rf0 0
rp 179.6
lp -20.2
PLOT
wc 234
sc 8
vs 157
th 3
al ph

WHX-3-99-Rep-F2-Jan 22 INDY CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 22 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 14000 |
| ct | 14000 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lstfid | -3 |
| fn | not used |

**DISPLAY**

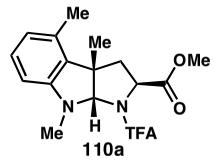
| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | -179.6 |
| lp | 125.4 |

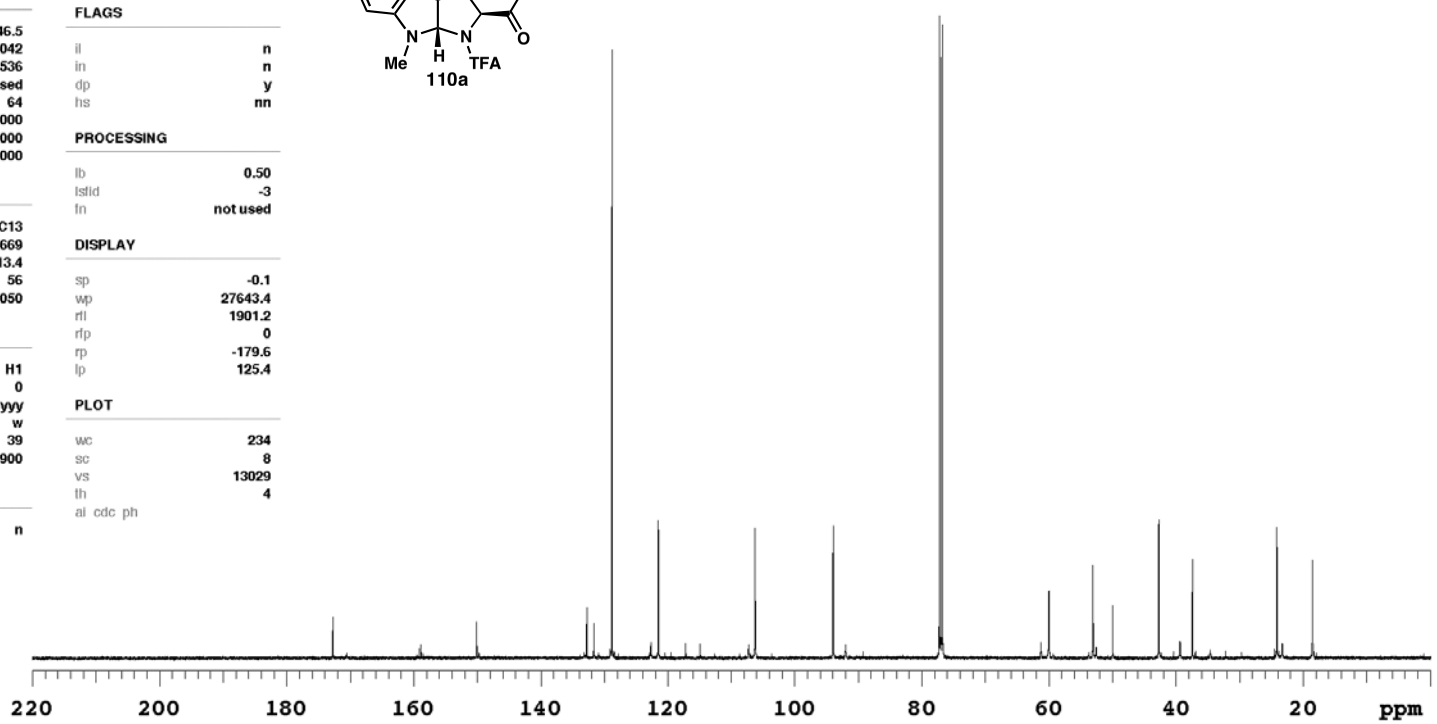
**PLOT**

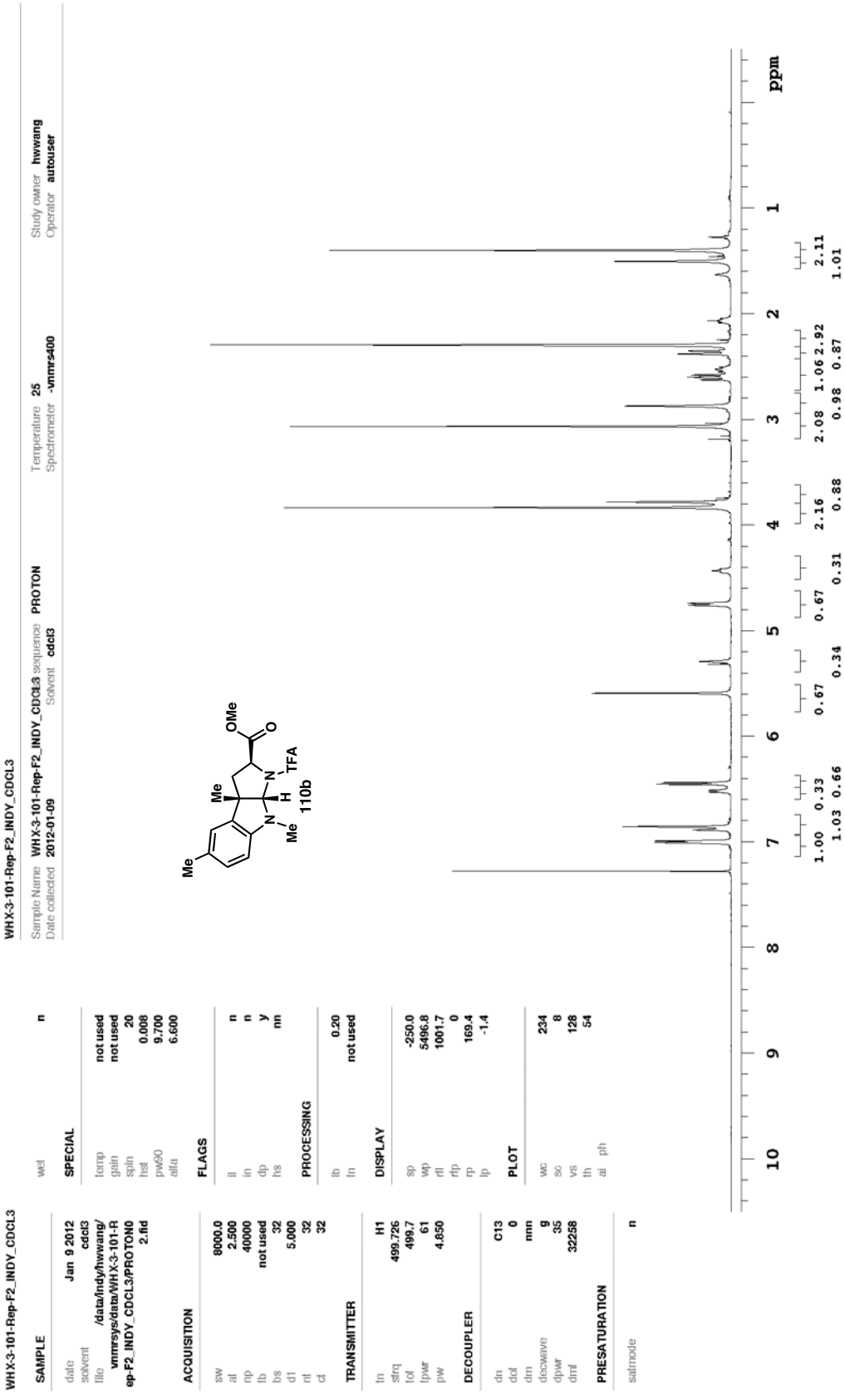
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 13029 |
| th | 4 |
| al cdc ph | |

WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-99-Rep-F2-Jan_22_INDY_CDCL3 | Experiment | CARBON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2012-01-22 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |




WHX-3-101-Rep-F2_INDY_CDCL3
SAMPLE
date Jan 9 2012
solvent cdcl3
file /data/indy/hwwang/vnmrsys/data/WHX-3-101-Rep-F2_INDY_CDCL3/PROTON0
2.fid
ACQUISITION
sw 8000.0
at 2.500
np 40000
fb not used
bs 32
d1 5.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 499.726
tof 499.7
tpwr 61
pw 4.850
DECOUPLER
dn C13
dof 0
dm nnn
decwave g
dpwr 35
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 9.700
alfa 6.600
FLAGS
ll n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -250.0
wp 5496.8
rf1 1001.7
rf0 0
rp 169.4
lp -1.4
PLOT
wc 234
sc 8
vs 128
th 54
al ph
WHX-3-101-Rep-F2_INDY_CDCL3
Sample Name WHX-3-101-Rep-F2_INDY_CDCL3 sequence PROTON
Date collected 2012-01-09 Solvent cdcl3
Temperature 25
Spectrometer -vnmrs400
Study owner hwwang
Operator autouser
Me
Me
OMe
O
N
TFA
H
110b
N
Me
10 9 8 7 6 5 4 3 2 1 ppm
1.00 0.33 0.67 0.67 0.67 2.16 2.08 1.06 2.92 2.11
1.03 0.66 0.34 0.31 0.88 0.98 0.87 1.01

WHX-3-101-Rep-F2_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 9 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-101-Rep-F2_INDY_CDCL3/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 13000 |
| ct | 13000 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lstid | -3 |
| fn | not used |

**DISPLAY**

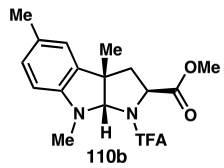
| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | -168.3 |
| lp | 127.7 |

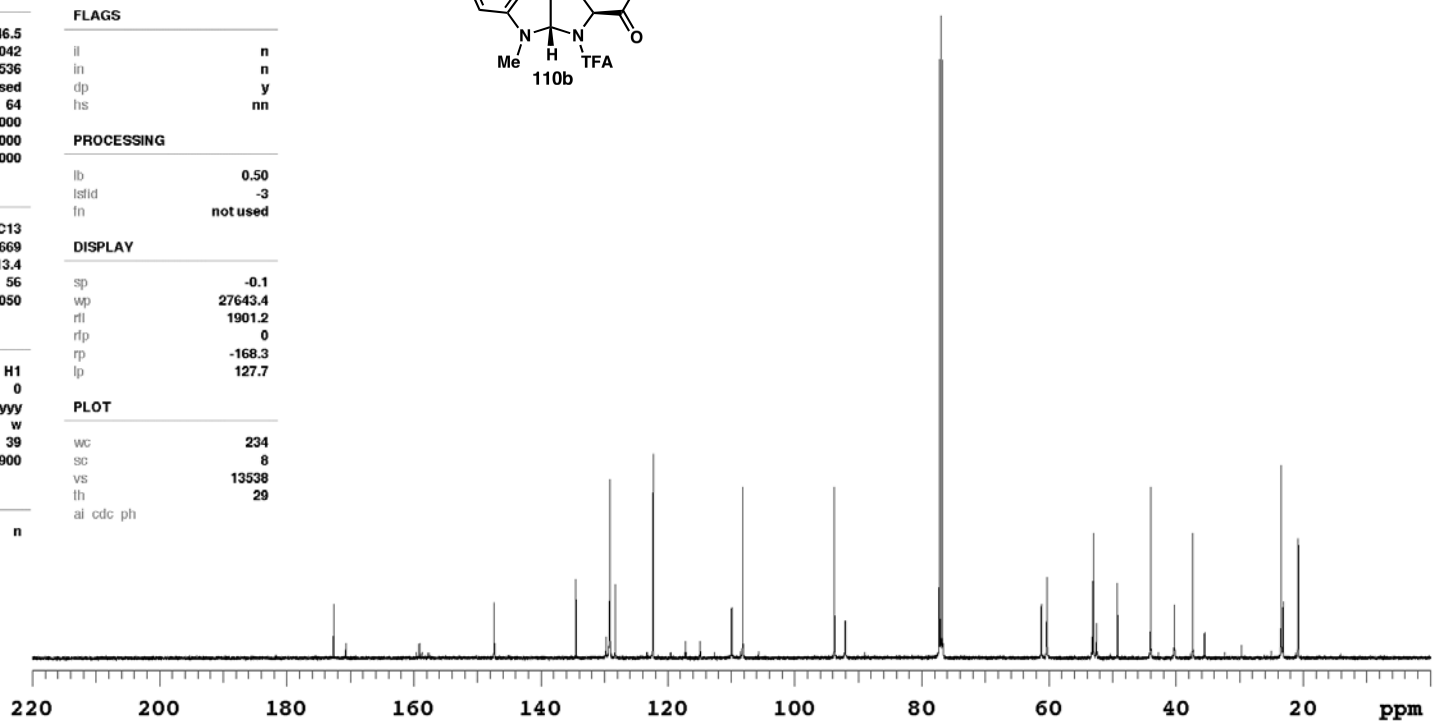
**PLOT**

| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 13538 |
| th | 29 |
| ai cdc ph | |

WHX-3-101-Rep-F2_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-101-Rep-F2_INDY_CDCL3 | sequence | CARBON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2012-01-09 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |



WHX-3-102-Rep-F2_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 10 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-102-Rep-F2_INDY_CDCL3/PROTON02.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 8000.0 |
| at | 2.500 |
| np | 40000 |
| fb | not used |
| bs | 32 |
| d1 | 5.000 |
| nt | 32 |
| ct | 32 |

**TRANSMITTER**

| | |
|---|---|
| tn | H1 |
| sfrq | 499.726 |
| tof | 499.7 |
| tpwr | 61 |
| pw | 4.850 |

**DECOUPLER**

| | |
|---|---|
| dn | C13 |
| dof | 0 |
| dm | nnn |
| decwave | g |
| dpwr | 35 |
| dmf | 32258 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | not used |
| spin | 20 |
| hst | 0.008 |
| pw90 | 9.700 |
| alfa | 6.600 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.20 |
| fn | not used |

**DISPLAY**

| | |
|---|---|
| sp | -250.0 |
| wp | 5496.8 |
| rfl | 1001.7 |
| rfp | 0 |
| rp | 164.3 |
| lp | 11.7 |

**PLOT**

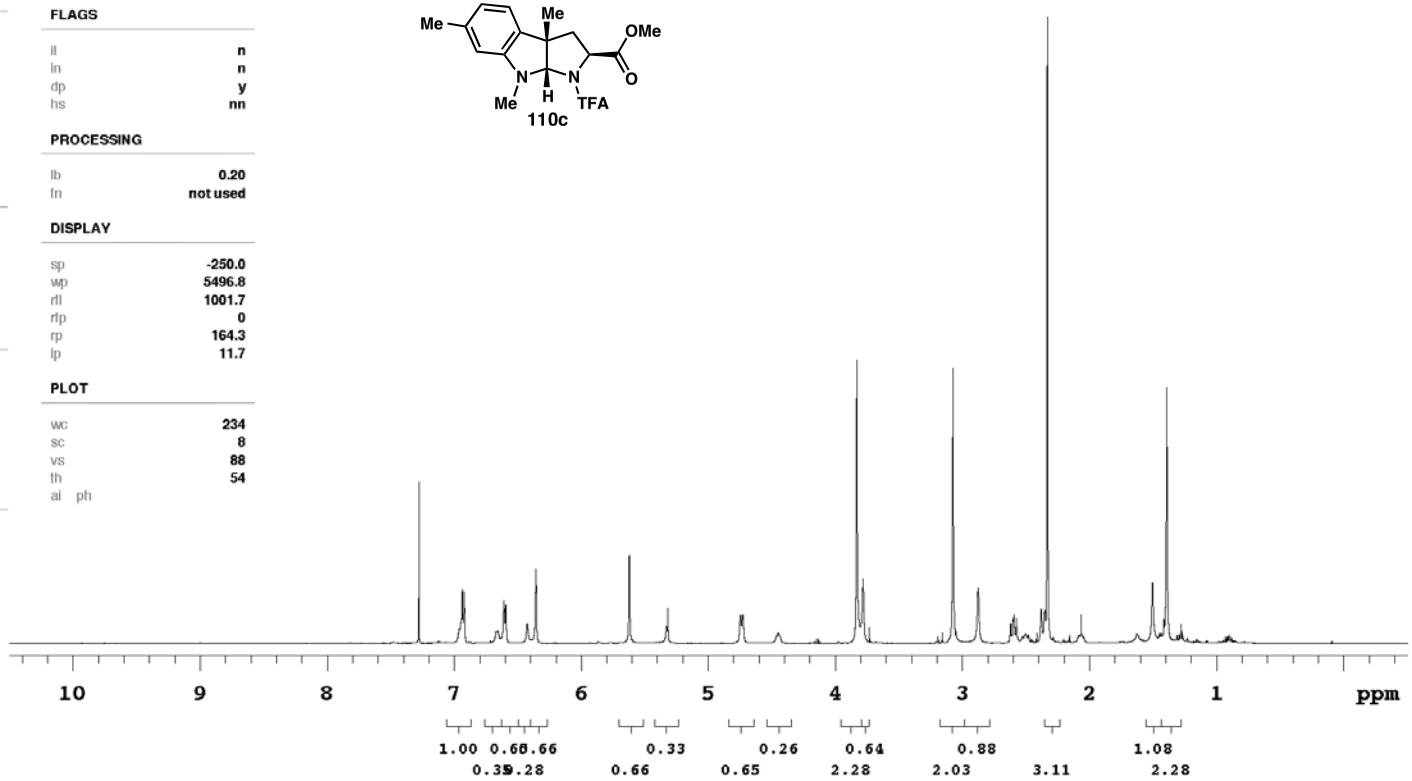
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 88 |
| th | 54 |
| ai ph | |

WHX-3-102-Rep-F2_INDY_CDCL3

Sample Name WHX-3-102-Rep-F2_INDY_CDCL3 sequence PROTON Temperature 25 Study owner hwwang
Date collected 2012-01-10 Solvent cdcl3 Spectrometer -vnmrs400 Operator autouser




110c



WHX-3-102-Rep-F2_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 11 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-102-Rep-F2_INDY_CDCL3/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 8500 |
| ct | 8500 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lstid | -3 |
| fn | not used |

**DISPLAY**

| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | -164.8 |
| lp | 120.7 |

**PLOT**

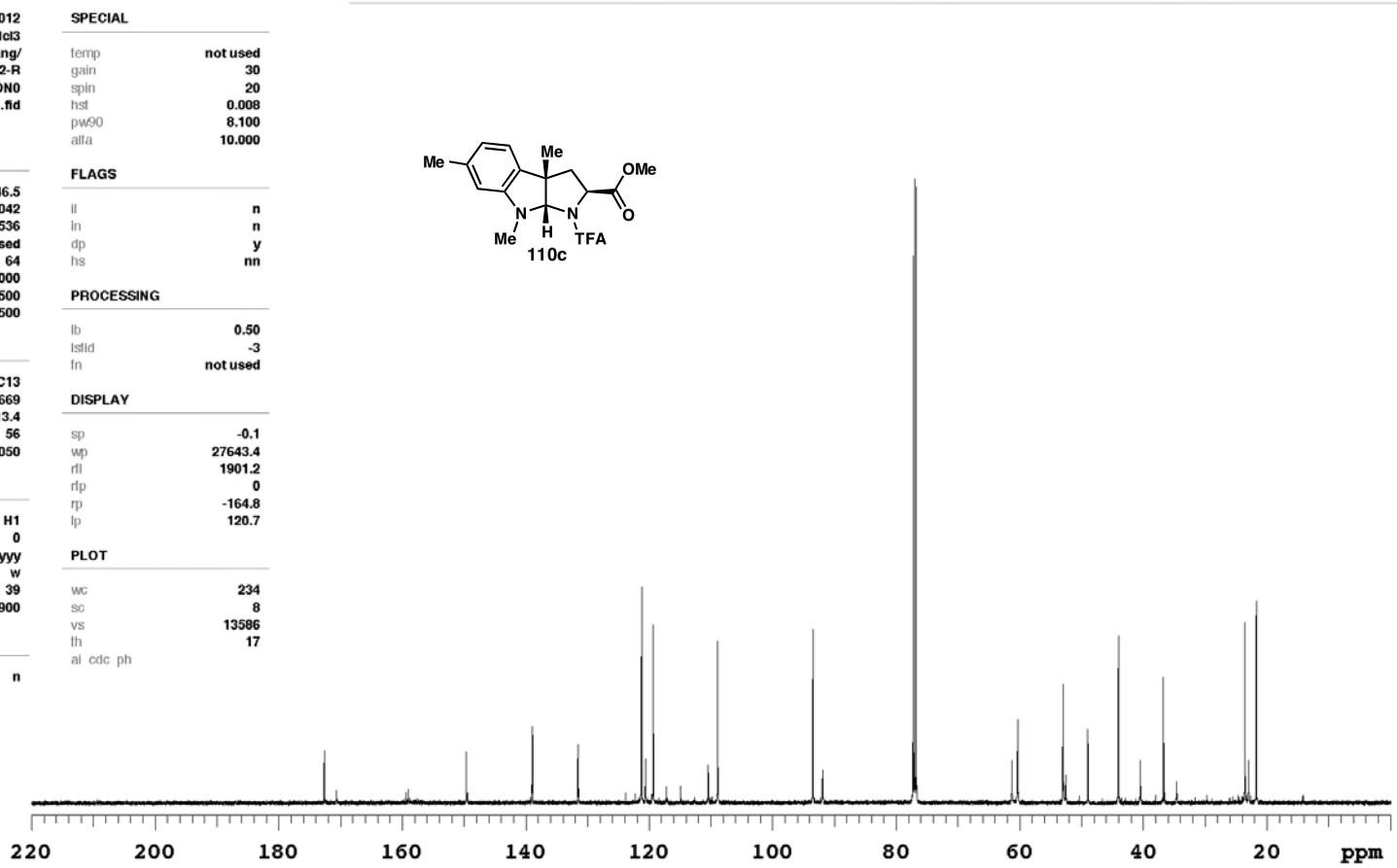
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 13586 |
| th | 17 |
| al cdc ph | |

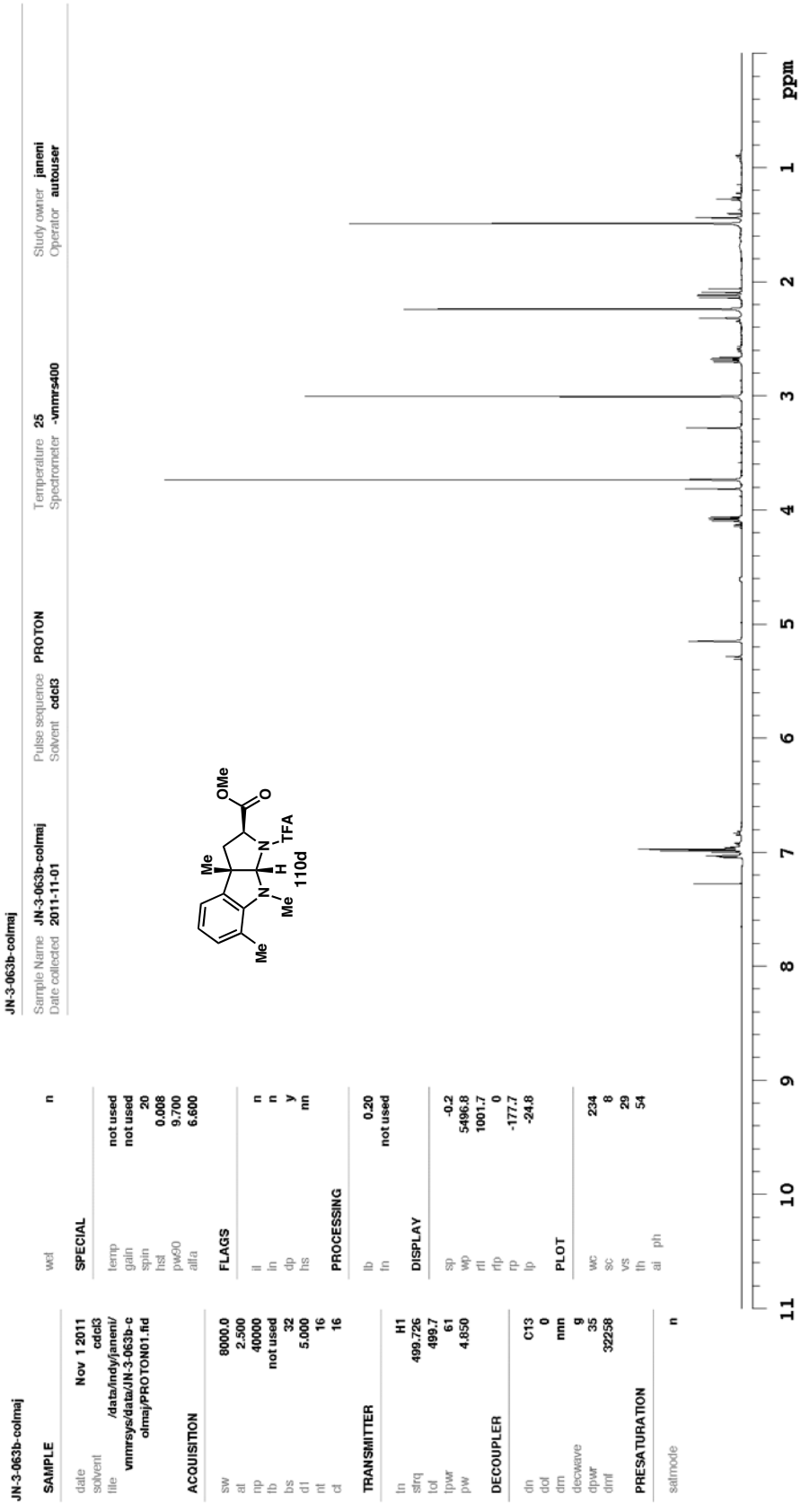
WHX-3-102-Rep-F2_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-102-Rep-F2_INDY_CDCL3 | sequence | CARBON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2012-01-11 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |



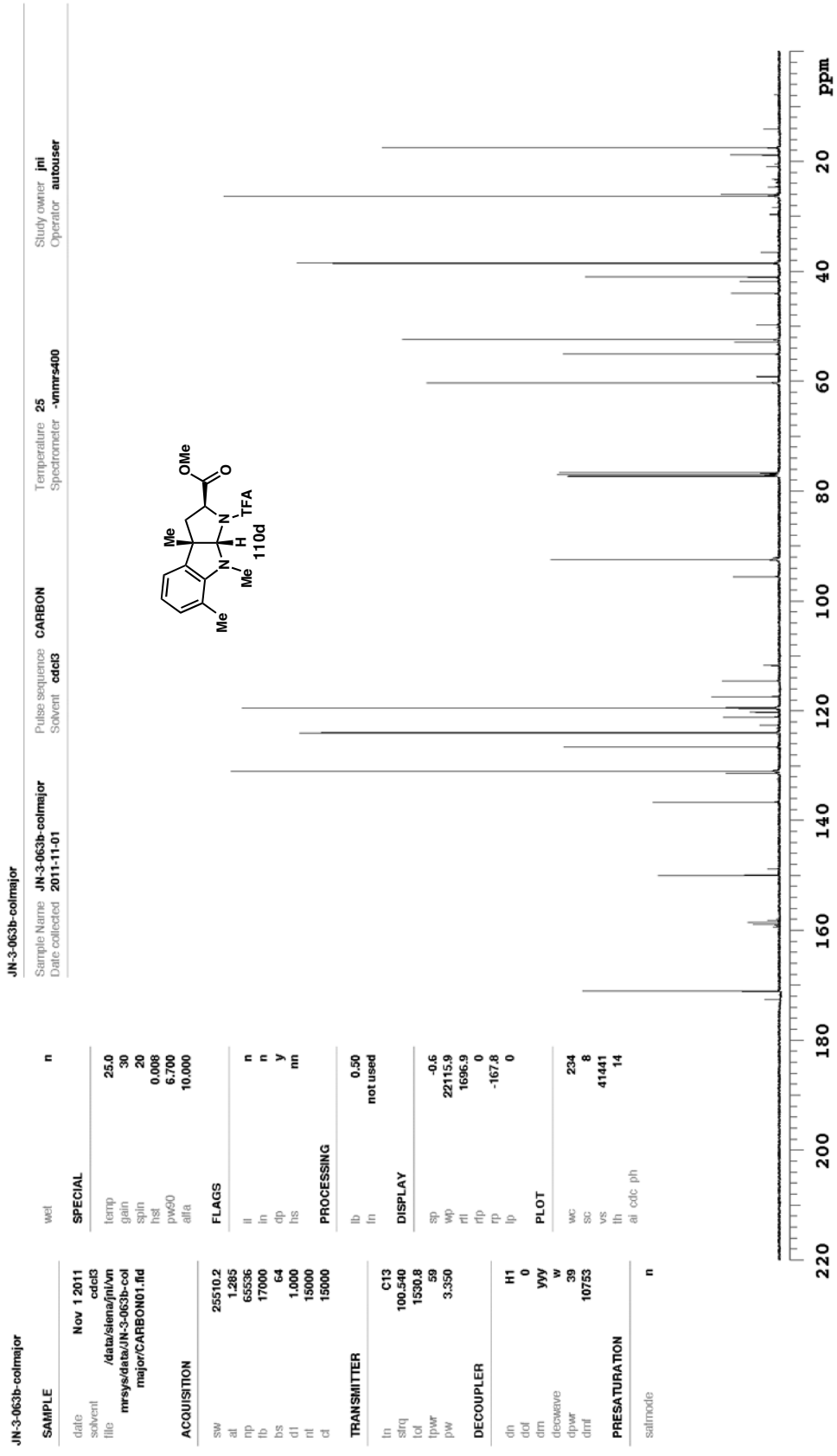

110c

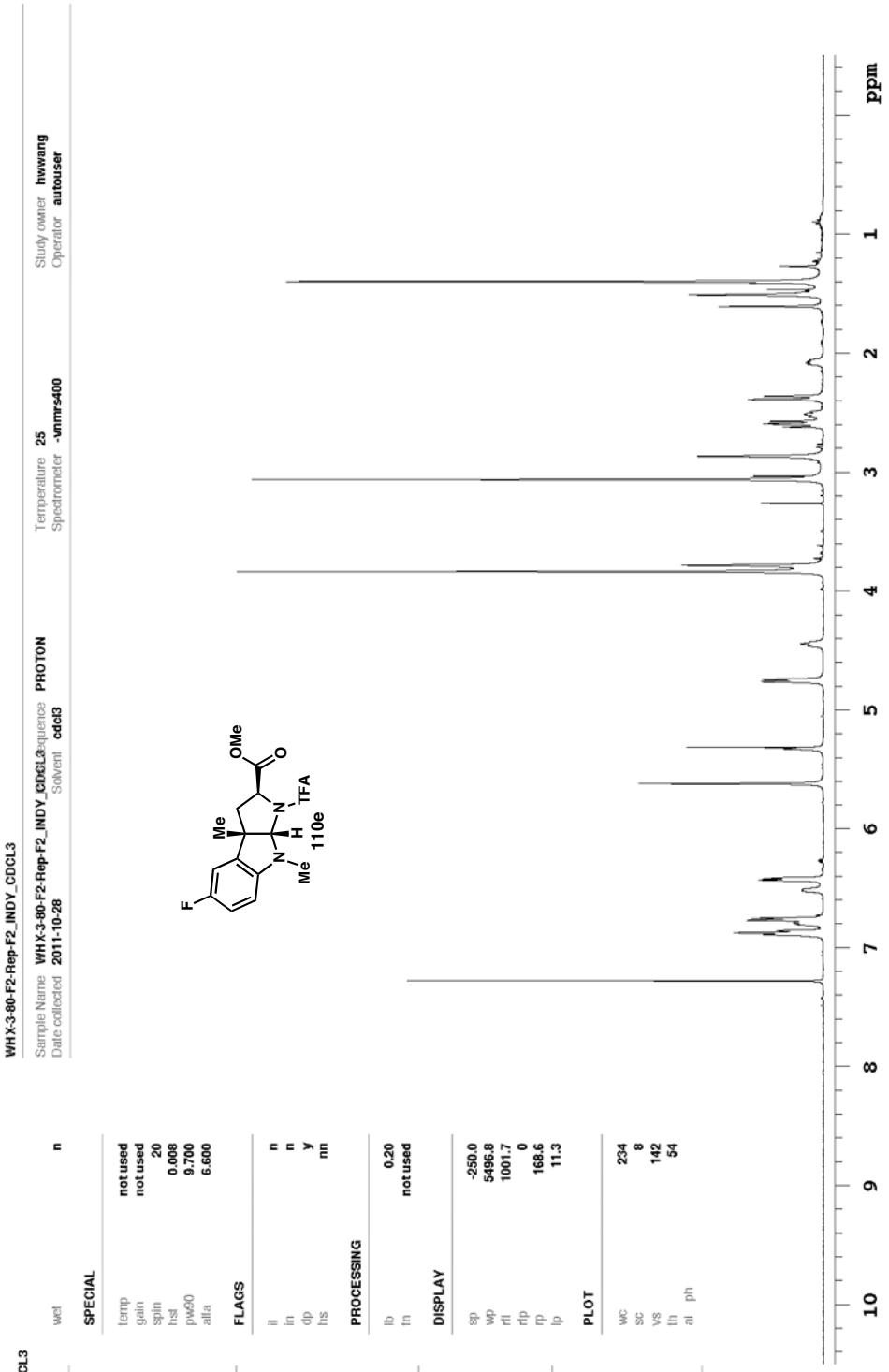





JN-3-063b-colmaj

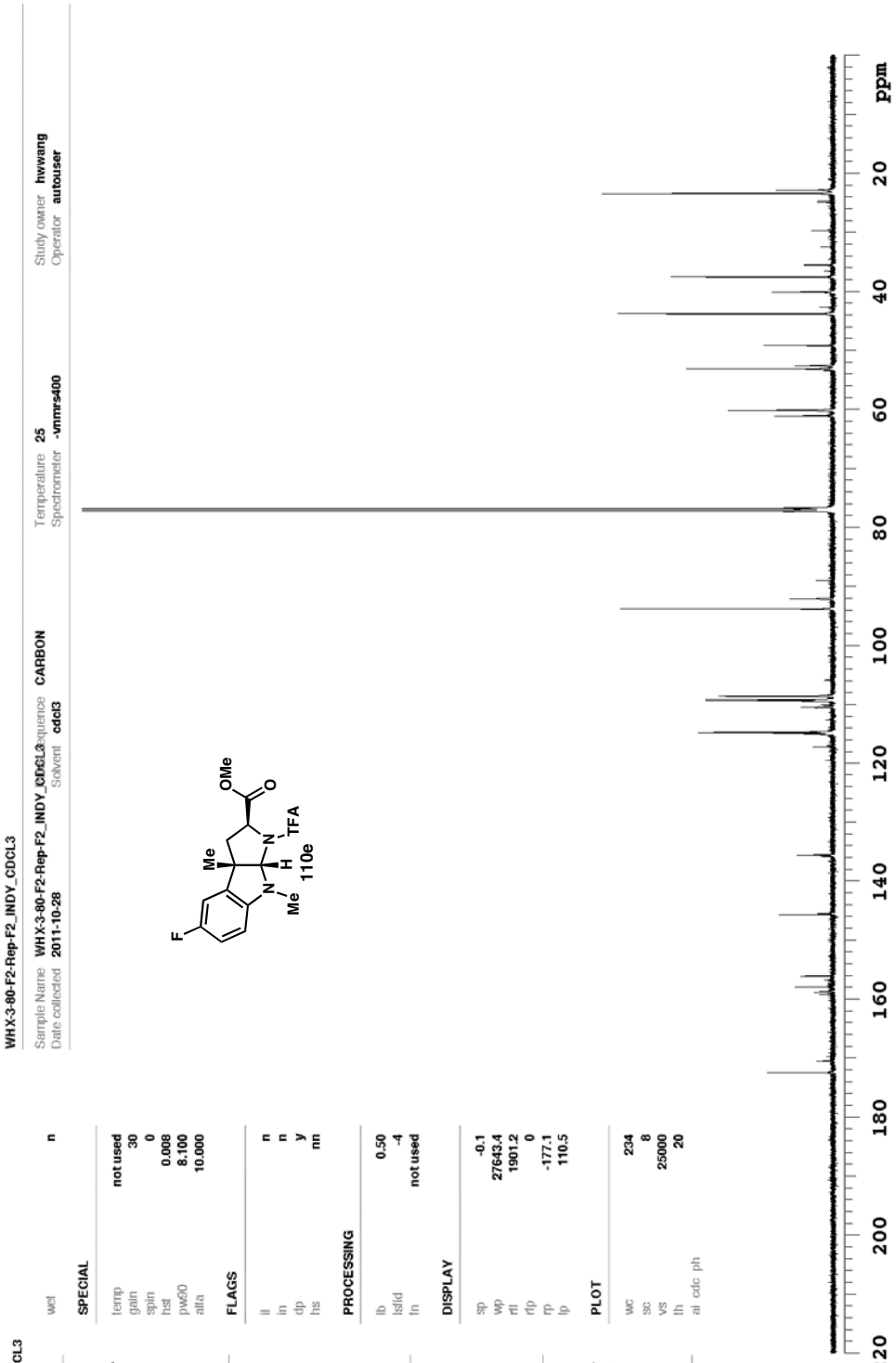
| | | | |
|---|---|---|---|
| **SAMPLE** | | wet | n |
| date | Nov 1 2011 | **SPECIAL** | |
| solvent | cdcl3 | temp | not used |
| file | /data/indy/janeni/vnmrsys/data/JN-3-063b-colmaj/PROTON01.fid | gain | not used |
| | | spin | 20 |
| | | hst | 0.008 |
| **ACQUISITION** | | pw90 | 9.700 |
| | | alfa | 6.600 |
| sw | 8000.0 | **FLAGS** | |
| at | 2.500 | il | n |
| np | 40000 | in | n |
| fb | not used | dp | y |
| bs | 32 | hs | nn |
| d1 | 5.000 | | |
| nt | 16 | **PROCESSING** | |
| ct | 16 | | |
| **TRANSMITTER** | | lb | 0.20 |
| | | fn | not used |
| tn | H1 | **DISPLAY** | |
| sfrq | 499.726 | | |
| tof | 499.7 | sp | -0.2 |
| tpwr | 61 | wp | 5496.8 |
| pw | 4.850 | rf1 | 1001.7 |
| **DECOUPLER** | | rfp | 0 |
| | | rp | -177.7 |
| dn | C13 | lp | -24.8 |
| dof | 0 | **PLOT** | |
| dm | nnn | | |
| decwave | g | wc | 234 |
| dpwr | 35 | sc | 8 |
| dmf | 32258 | vs | 29 |
| **PRESATURATION** | | th | 54 |
| | | ai ph | |
| satmode | n | | |


JN-3-063b-colmajor
Sample Name JN-3-063b-colmajor
Date collected 2011-11-01
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Spectrometer -vnmrs400
Study owner jni
Operator autouser
OMe
O
N
TFA
H
110d
Me
N
Me
Me
SAMPLE
date Nov 1 2011
solvent cdcl3
file /data/siena/jni/vn
mrsys/data/JN-3-063b-col
major/CARBON01.fid
ACQUISITION
sw 25510.2
at 1.285
np 65536
fb 17000
bs 64
d1 1.000
nt 15000
ct 15000
TRANSMITTER
tn C13
sfrq 100.540
tof 1530.8
tpwr 59
pw 3.350
DECOUPLER
dn H1
dof 0
dm yyy
decwave w
dpwr 39
dmf 10753
PRESATURATION
satmode n
wet n
SPECIAL
temp 25.0
gain 30
spin 20
hst 0.008
pw90 6.700
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
ln not used
DISPLAY
sp -0.6
wp 22115.9
rf1 1696.9
rf2 0
rp -167.8
lp 0
PLOT
wc 234
sc 8
vs 41441
th 14
ai cdc ph
ppm
220
200
180
160
140
120
100
80
60
40
20




**WHX-3-80-F2-Rep-F2_INDY_CDCL3**

| | | | |
|---|---|---|---|
| **SAMPLE** | | wet | n |
| date | Oct 28 2011 | **SPECIAL** | |
| solvent | cdcl3 | | |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-80-F2-Rep-F2_INDY_CDCL3/PROTON01.fid | temp | not used |
| | | gain | not used |
| | | spin | 20 |
| | | hst | 0.008 |
| | | pw90 | 9.700 |
| | | alfa | 6.600 |
| **ACQUISITION** | | **FLAGS** | |
| sw | 8000.0 | il | n |
| at | 2.500 | in | n |
| np | 40000 | dp | y |
| fb | not used | hs | nn |
| bs | 32 | | |
| d1 | 5.000 | **PROCESSING** | |
| nt | 16 | | |
| ct | 16 | lb | 0.20 |
| | | fn | not used |
| **TRANSMITTER** | | **DISPLAY** | |
| tn | H1 | | |
| sfrq | 499.726 | sp | -250.0 |
| tof | 499.7 | wp | 5496.8 |
| tpwr | 61 | rf1 | 1001.7 |
| pw | 4.850 | rfp | 0 |
| **DECOUPLER** | | rp | 168.6 |
| | | lp | 11.3 |
| dn | C13 | **PLOT** | |
| dof | 0 | | |
| dm | nnn | wc | 234 |
| decwave | g | sc | 8 |
| dpwr | 35 | vs | 142 |
| dmf | 32258 | th | 54 |
| **PRESATURATION** | | al ph | |
| satmode | n | | |




WHX-3-80-F2-Rep-F2_INDY_CDCL3

| | | | |
|---|---|---|---|
| **SAMPLE** | | wet | n |
| date | Oct 28 2011 | **SPECIAL** | |
| solvent | cdcl3 | temp | not used |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-80-F2-Rep-F2_INDY_CDCL3/CARBON/N01.fid | gain | 30 |
| | | spin | 0 |
| | | hst | 0.008 |
| | | pw90 | 8.100 |
| | | alfa | 10.000 |
| **ACQUISITION** | | **FLAGS** | |
| sw | 31446.5 | il | n |
| at | 1.042 | in | n |
| np | 65536 | dp | y |
| fb | not used | hs | nn |
| bs | 64 | | |
| d1 | 1.000 | | |
| nt | 10000 | **PROCESSING** | |
| ct | 10000 | lb | 0.50 |
| | | lsfid | -4 |
| **TRANSMITTER** | | fn | not used |
| tn | C13 | **DISPLAY** | |
| sfrq | 125.669 | sp | -0.1 |
| tof | 1913.4 | wp | 27643.4 |
| tpwr | 56 | rf1 | 1901.2 |
| pw | 4.050 | rfp | 0 |
| **DECOUPLER** | | rp | -177.1 |
| dn | H1 | lp | 110.5 |
| dof | 0 | | |
| dm | yyy | **PLOT** | |
| decpwave | w | | |
| dpwr | 39 | wc | 234 |
| dmf | 11900 | sc | 8 |
| | | vs | 25000 |
| **PRESATURATION** | | th | 20 |
| satmode | n | ai cdc ph | |

WHX-3-81-F2-Rep-Re_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Nov 5 2011 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/ vnmrsys/data/WHX-3-81-F2 -Rep-Re_INDY_CDCL3/PROTO N02.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 8000.0 |
| at | 2.500 |
| np | 40000 |
| fb | not used |
| bs | 32 |
| d1 | 5.000 |
| nt | 32 |
| ct | 32 |

**TRANSMITTER**

| | |
|---|---|
| tn | H1 |
| sfrq | 499.726 |
| tof | 499.7 |
| tpwr | 61 |
| pw | 4.850 |

**DECOUPLER**

| | |
|---|---|
| dn | C13 |
| dof | 0 |
| dm | nnn |
| decwave | g |
| dpwr | 35 |
| dmf | 32258 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | not used |
| spin | 20 |
| hst | 0.008 |
| pw90 | 9.700 |
| alfa | 6.600 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.20 |
| fn | not used |

**DISPLAY**

| | |
|---|---|
| sp | -250.0 |
| wp | 5496.8 |
| rfl | 1001.7 |
| rfp | 0 |
| rp | 166.0 |
| lp | -2.3 |

**PLOT**

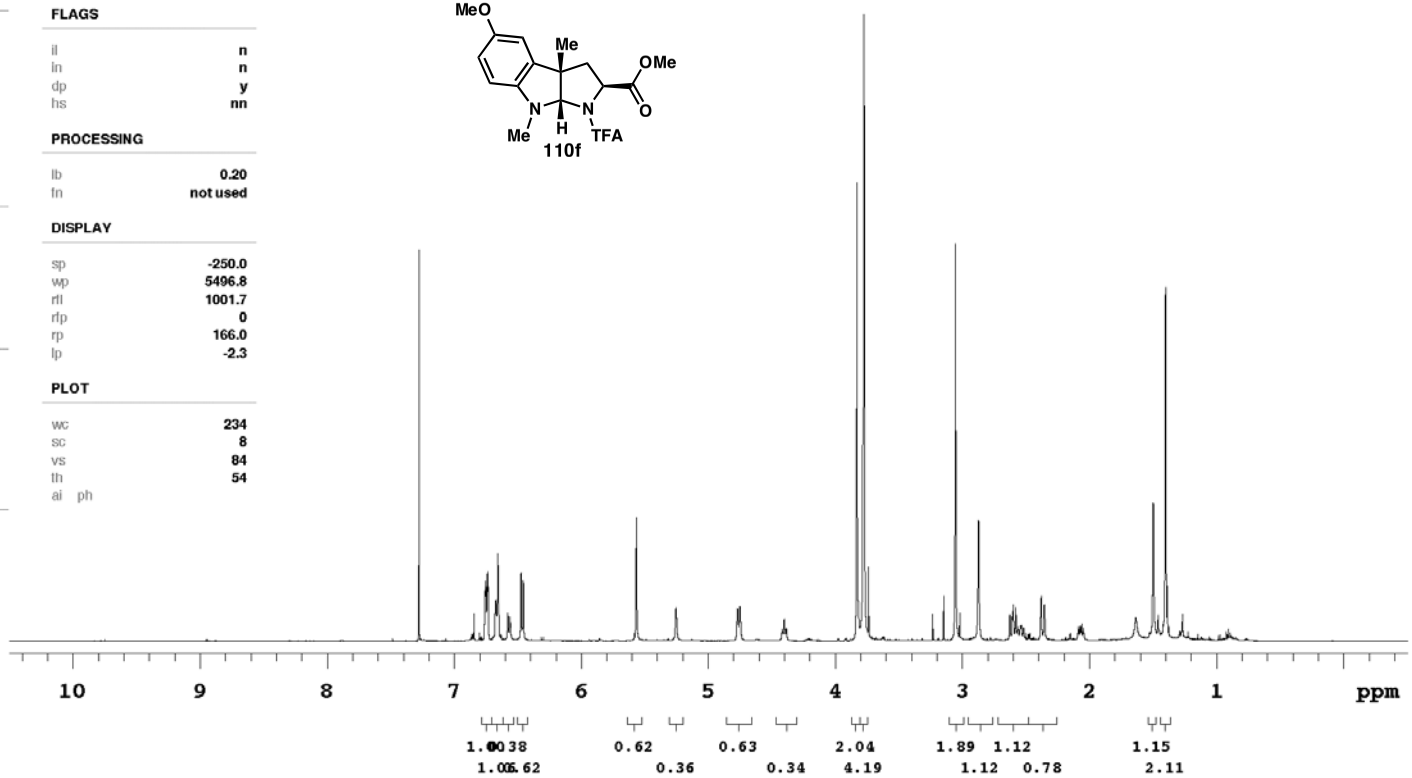
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 84 |
| th | 54 |
| ai ph | |

WHX-3-81-F2-Rep-Re_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-81-F2-Rep-Re_INDY_CDCL3 | Pulse Sequence | PROTON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2011-11-05 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |




110f



WHX-3-81-F2-Rep-Re_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Nov 5 2011 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-81-F2-Rep-Re_INDY_CDCL3/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 13850 |
| ct | 13850 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lstfid | -3 |
| fn | not used |

**DISPLAY**

| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | -173.0 |
| lp | 137.4 |

**PLOT**

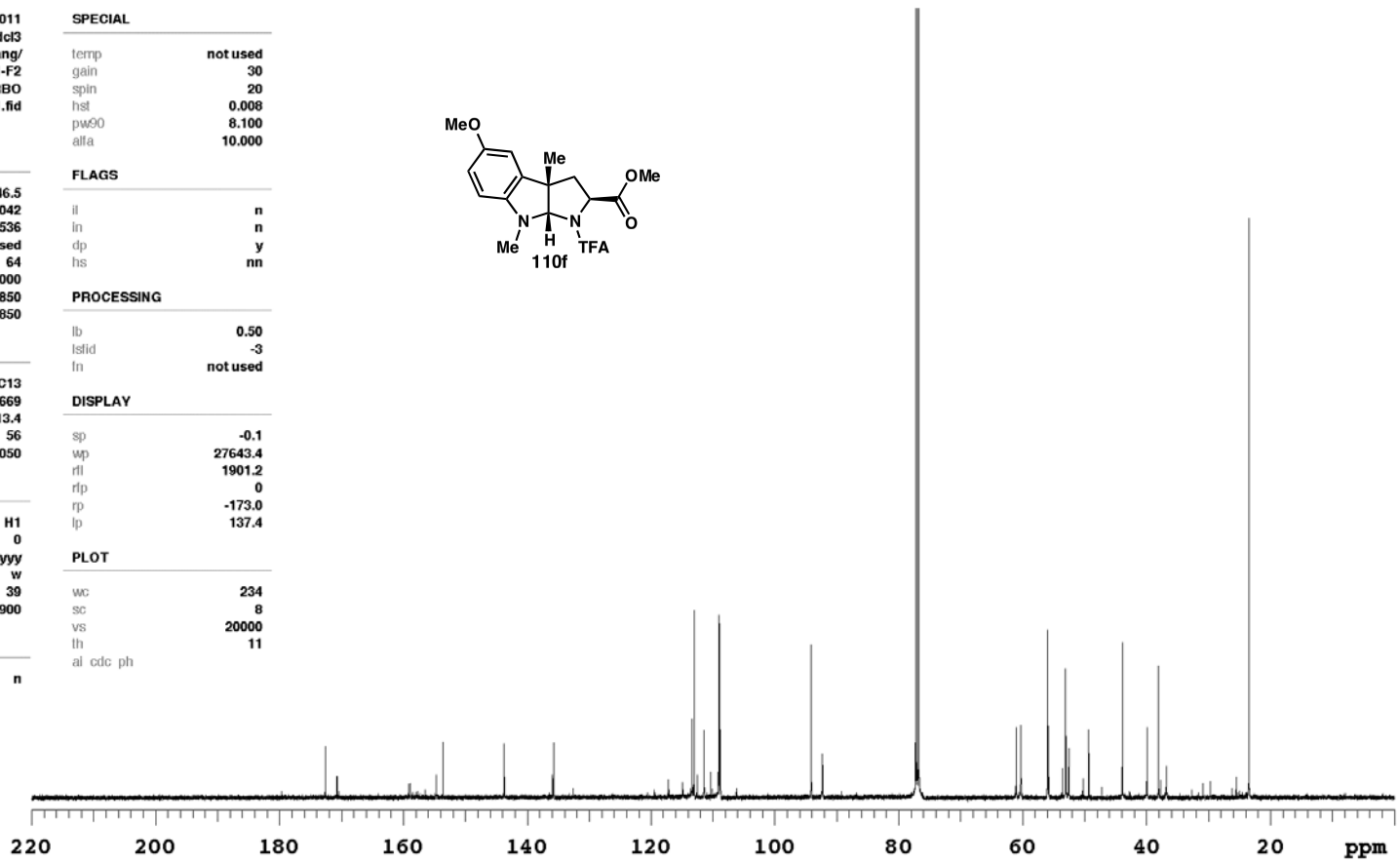
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 20000 |
| th | 11 |

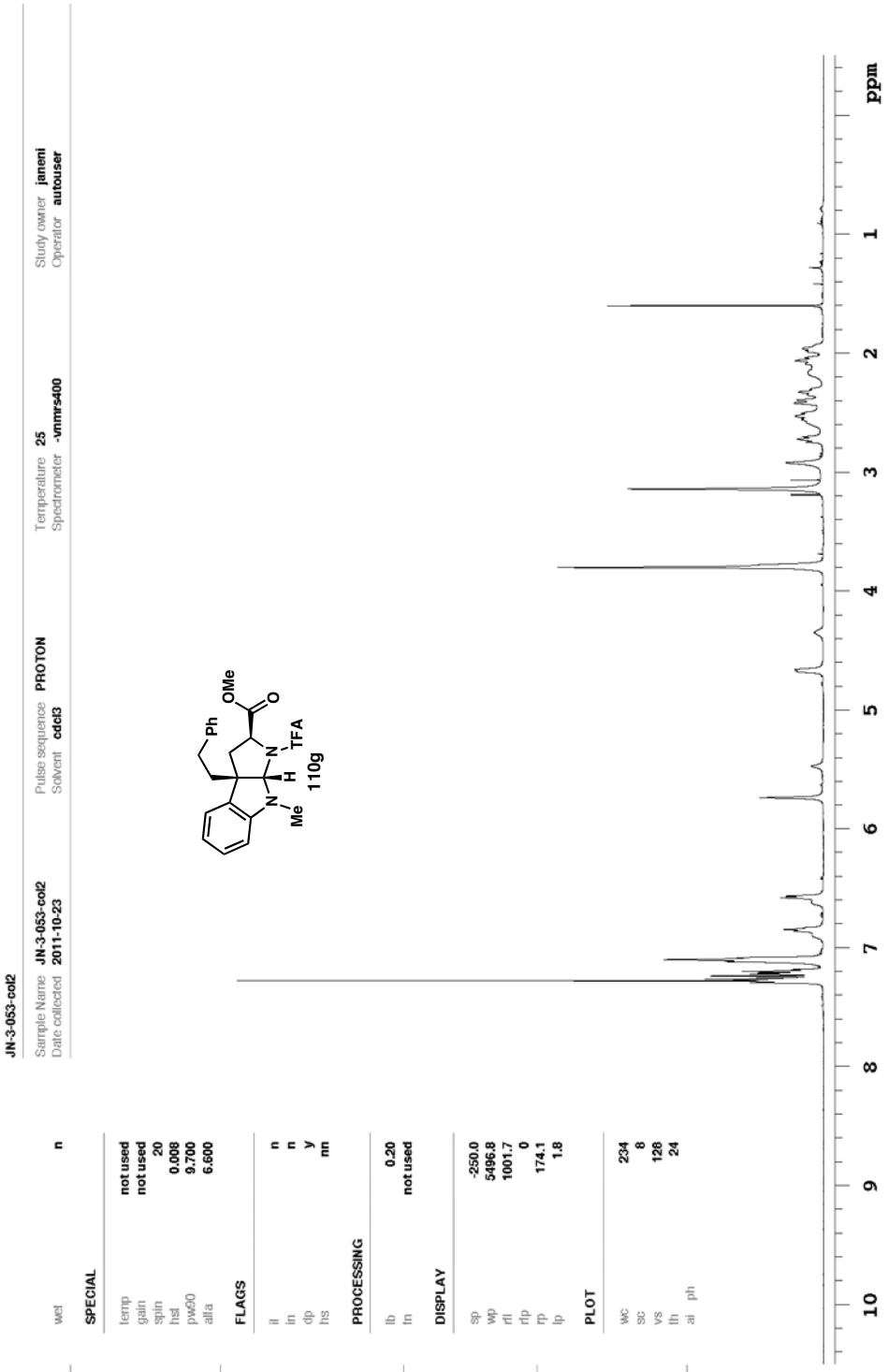
al cdc ph

WHX-3-81-F2-Rep-Re_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-81-F2-Rep-Re_INDY_CDCL3 | Pulse Sequence | CARBON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2011-11-05 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |




JN-3-053-col2
Sample Name JN-3-053-col2
Date collected 2011-10-23
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Spectrometer -vnmrs400
Study owner janeni
Operator autouser
OMe
O
Ph
N
TFA
H
N
Me
110g
ppm
10
9
8
7
6
5
4
3
2
1
0.53 0.99 1.00
2.57 1.02
0.68
0.30
0.69
0.29
2.99
2.07 0.75
0.89
5.28
JN-3-053-col2
SAMPLE
date Oct 23 2011
solvent cdcl3
file /data/mdy/janeni/vnmrsys/data/JN-3-053-co l2/PROTON02.fid
ACQUISITION
sw 8000.0
at 2.500
np 40000
fb not used
bs 32
d1 25.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 499.726
tof 499.7
tpwr 61
pw 4.850
DECOUPLER
dn C13
dof 0
dm nnn
decoupler
dpwr 35
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 9.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
fn not used
DISPLAY
sp -250.0
wp 5496.8
rf1 1001.7
rfp 0
rp 174.1
lp 1.8
PLOT
wc 234
sc 8
vs 128
th 24
ai ph

JN-3-053-col2

**SAMPLE**

| | |
|---|---|
| date | Oct 23 2011 |
| solvent | cdcl3 |
| file | /data/indy/janeni/vnmrsys/data/JN-3-053-col2/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 13000 |
| ct | 13000 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 0 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lsfid | -4 |
| fn | not used |

**DISPLAY**

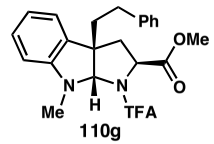
| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | -178.9 |
| lp | 106.4 |

**PLOT**

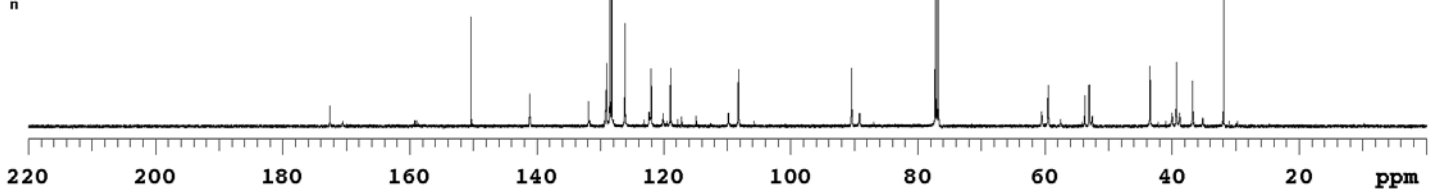
| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 11429 |
| th | 4 |
| ai cdc ph | |

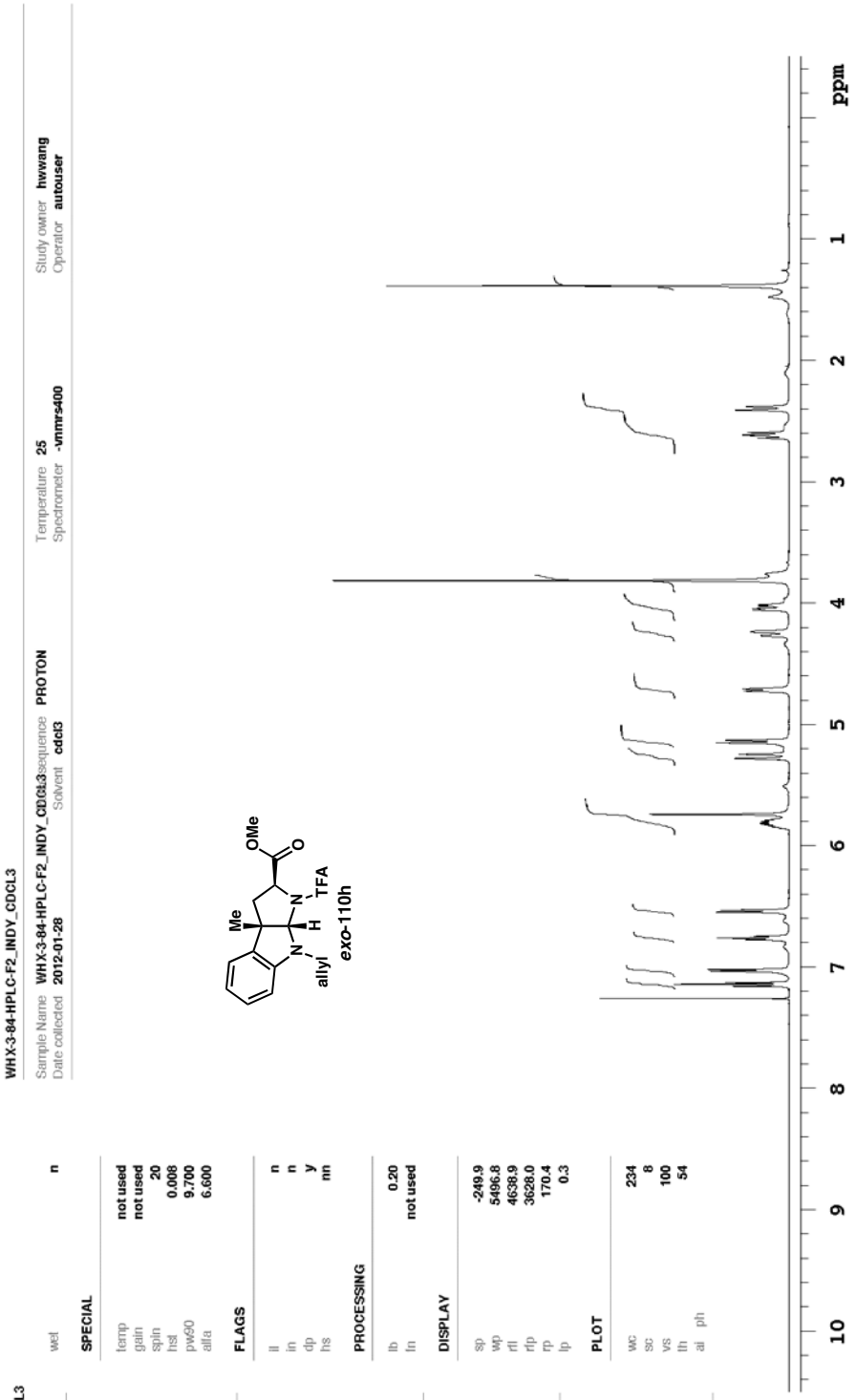
JN-3-053-col2

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | JN-3-053-col2 | Pulse sequence | CARBON | Temperature | 25 | Study owner | janeni |
| Date collected | 2011-10-23 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |




110g




WHX-3-84-HPLC-F2_INDY_CDCL3
SAMPLE
date Jan 28 2012
solvent cdcl3
file /data/lndy/hwwang/vnmrsys/data/WHX-3-84-HPLC-F2_INDY_CDCL3/PROTON0
ACQUISITION
sw 8000.0
at 2.500
np 40000
fb not used
bs 32
d1 5.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 499.726
tof 499.7
tpwr 61
pw 4.850
DECOUPLER
dn C13
dof 0
dm nnn
decwave g
dpwr 35
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 9.700
alfa 6.600
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.20
ln not used
DISPLAY
sp -249.9
wp 5496.8
rf1 4638.9
rf2 3628.0
rp 170.4
lp 0.3
PLOT
wc 234
sc 8
vs 100
th 54
al ph
WHX-3-84-HPLC-F2_INDY_CDCL3
Sample Name WHX-3-84-HPLC-F2_INDY_CDCL3 sequence PROTON
Date collected 2012-01-28 Solvent cdcl3
Temperature 25
Spectrometer -vnmrs400
Study owner hwwang
Operator autouser
Me
OMe
O
N
TFA
H
N
allyl
exo-110h
1.16 0.99
1.16 1.00
2.14
1.28
1.11 0.97
1.01 3.41
1.20
2.20
2.91
10
9
8
7
6
5
4
3
2
1
ppm

WHX-3-84-HPLC-F2_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 28 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-84-HPLC-F2_INDY_CDCL3/CARBON02.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 13500 |
| ct | 13500 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1900.8 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lsfid | -3 |
| fn | not used |

**DISPLAY**

| | |
|---|---|
| sp | -0.2 |
| wp | 27643.4 |
| rfl | 1913.8 |
| rfp | 0 |
| rp | -162.8 |
| lp | 104.7 |

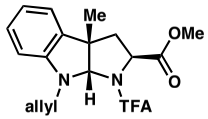
**PLOT**

| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 12120 |
| th | 7 |

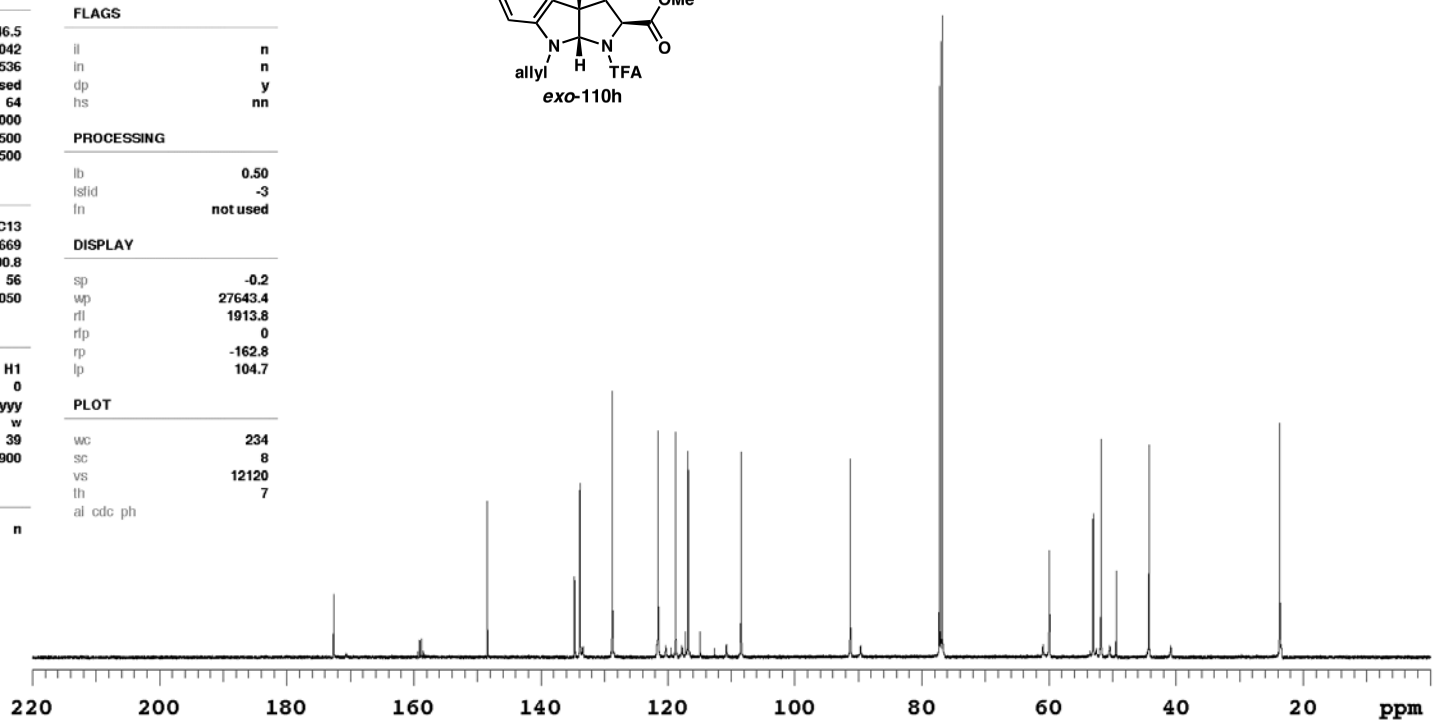
al cdc ph

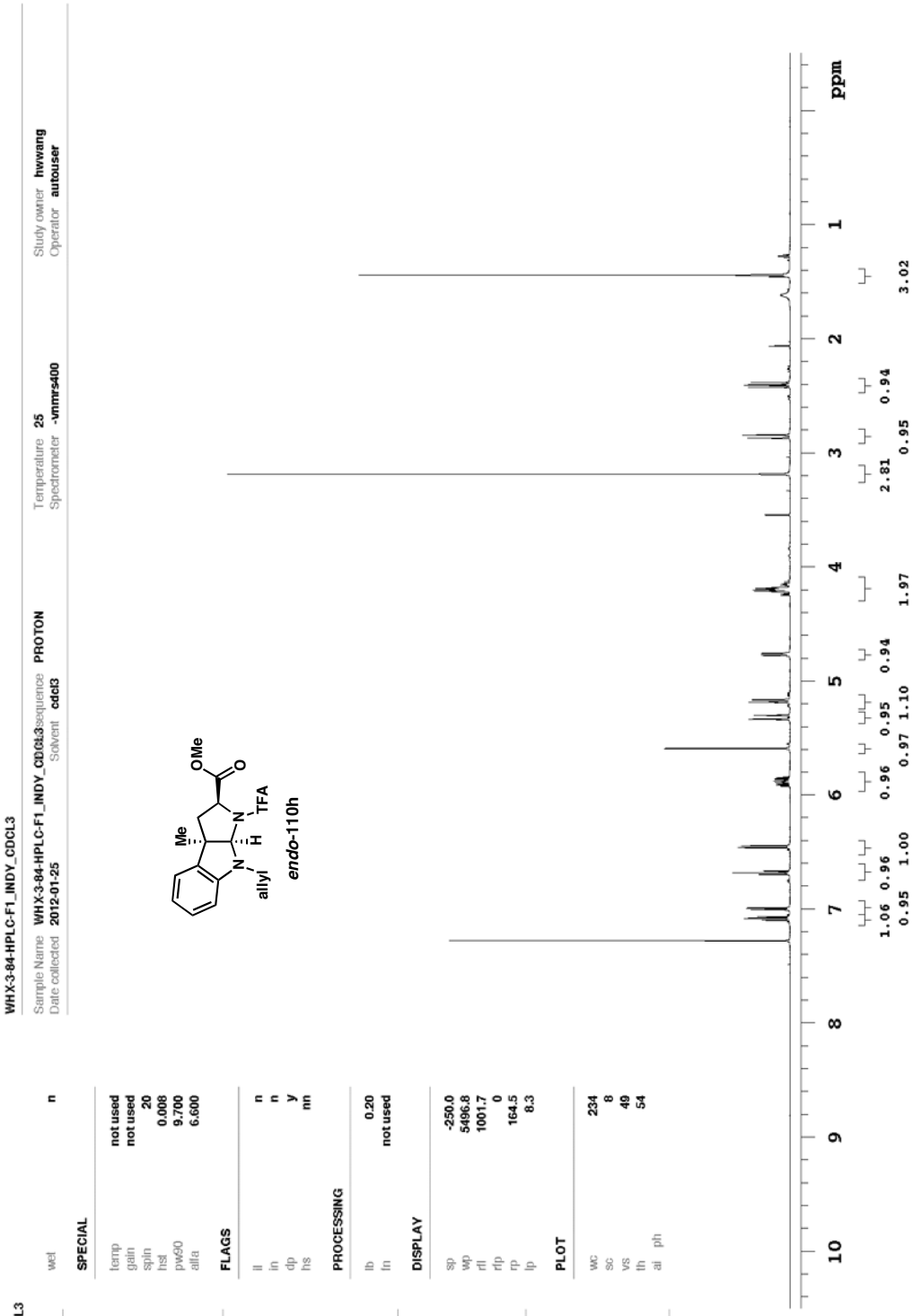
WHX-3-84-HPLC-F2_INDY_CDCL3

Sample Name WHX-3-84-HPLC-F2_INDY_CDCL3 Pulse sequence CARBON Temperature 25 Study owner hwwang
Date collected 2012-01-28 Solvent cdcl3 Spectrometer -vnmrs400 Operator autouser




*exo*-110h






WHX-3-84-HPLC-F1_INDY_CDCL3

| | | | |
|---|---|---|---|
| **SAMPLE** | | wet | n |
| date | Jan 25 2012 | **SPECIAL** | |
| solvent | cdcl3 | temp | not used |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-84-HPLC-F1_INDY_CDCL3/PROTON0 | gain | not used |
| | | spin | 20 |
| | | hst | 0.008 |
| | | pw90 | 9.700 |
| | | alfa | 6.600 |
| **ACQUISITION** | | **FLAGS** | |
| sw | 8000.0 | il | n |
| at | 2.500 | in | n |
| np | 40000 | dp | y |
| fb | not used | hs | nn |
| bs | 32 | | |
| d1 | 5.000 | **PROCESSING** | |
| nt | 32 | lb | 0.20 |
| ct | 32 | fn | not used |
| **TRANSMITTER** | | **DISPLAY** | |
| tn | H1 | sp | -250.0 |
| sfrq | 499.726 | wp | 5496.8 |
| tof | 499.7 | rf1 | 1001.7 |
| tpwr | 61 | rfp | 0 |
| pw | 4.850 | rp | 164.5 |
| **DECOUPLER** | | lp | 8.3 |
| dn | C13 | **PLOT** | |
| dof | 0 | wc | 234 |
| dm | nnn | sc | 8 |
| decoupler | g | vs | 49 |
| dpwr | 35 | th | 54 |
| dmf | 32258 | | |
| **PRESATURATION** | | | |
| satmode | n | | |

WHX-3-84-HPLC-F2_INDY_CDCL3

**SAMPLE**

| | |
|---|---|
| date | Jan 26 2012 |
| solvent | cdcl3 |
| file | /data/indy/hwwang/vnmrsys/data/WHX-3-84-HPLC-F2_INDY_CDCL3/CARBON01.fid |

**ACQUISITION**

| | |
|---|---|
| sw | 31446.5 |
| at | 1.042 |
| np | 65536 |
| fb | not used |
| bs | 64 |
| d1 | 1.000 |
| nt | 13500 |
| ct | 13500 |

**TRANSMITTER**

| | |
|---|---|
| tn | C13 |
| sfrq | 125.669 |
| tof | 1913.4 |
| tpwr | 56 |
| pw | 4.050 |

**DECOUPLER**

| | |
|---|---|
| dn | H1 |
| dof | 0 |
| dm | yyy |
| decwave | w |
| dpwr | 39 |
| dmf | 11900 |

**PRESATURATION**

| | |
|---|---|
| satmode | n |
| wet | n |

**SPECIAL**

| | |
|---|---|
| temp | not used |
| gain | 30 |
| spin | 20 |
| hst | 0.008 |
| pw90 | 8.100 |
| alfa | 10.000 |

**FLAGS**

| | |
|---|---|
| il | n |
| in | n |
| dp | y |
| hs | nn |

**PROCESSING**

| | |
|---|---|
| lb | 0.50 |
| lstid | -3 |
| ln | not used |

**DISPLAY**

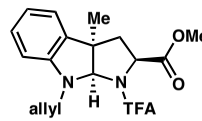
| | |
|---|---|
| sp | -0.1 |
| wp | 27643.4 |
| rfl | 1901.2 |
| rfp | 0 |
| rp | 178.8 |
| lp | 119.1 |

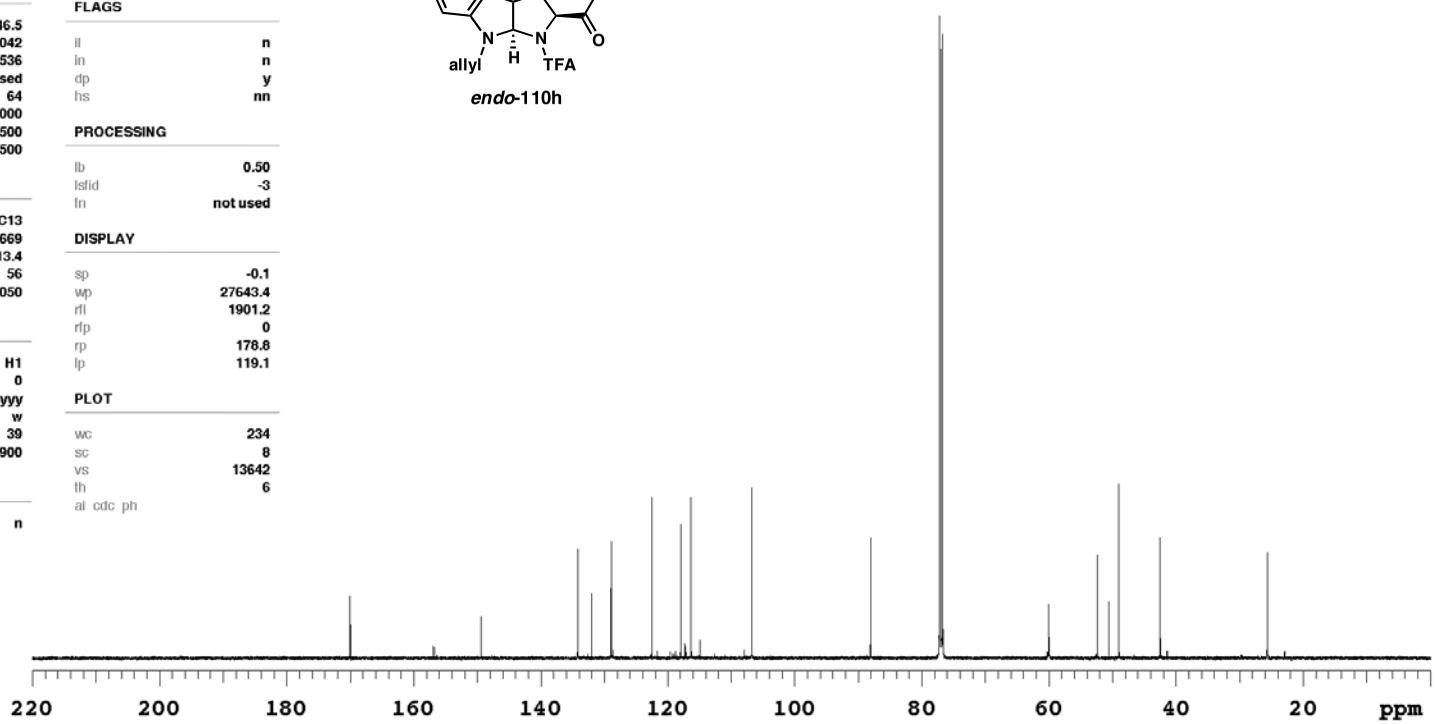
**PLOT**

| | |
|---|---|
| wc | 234 |
| sc | 8 |
| vs | 13642 |
| th | 6 |
| ai cdc ph | |

WHX-3-84-HPLC-F2_INDY_CDCL3

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Sample Name | WHX-3-84-HPLC-F2_INDY_CDCL3 | Pulse sequence | CARBON | Temperature | 25 | Study owner | hwwang |
| Date collected | 2012-01-26 | Solvent | cdcl3 | Spectrometer | -vnmrs400 | Operator | autouser |




*endo*-110h