

Appendix 1

*Spectra Relevant to Chapter 2:
Total Synthesis of (–)-Maoecrystal Z*

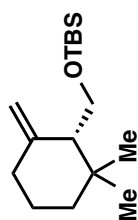
jc-1-245-A

Sample Name:
jc-1-245-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-1-245-A
FidFile: PROTON01

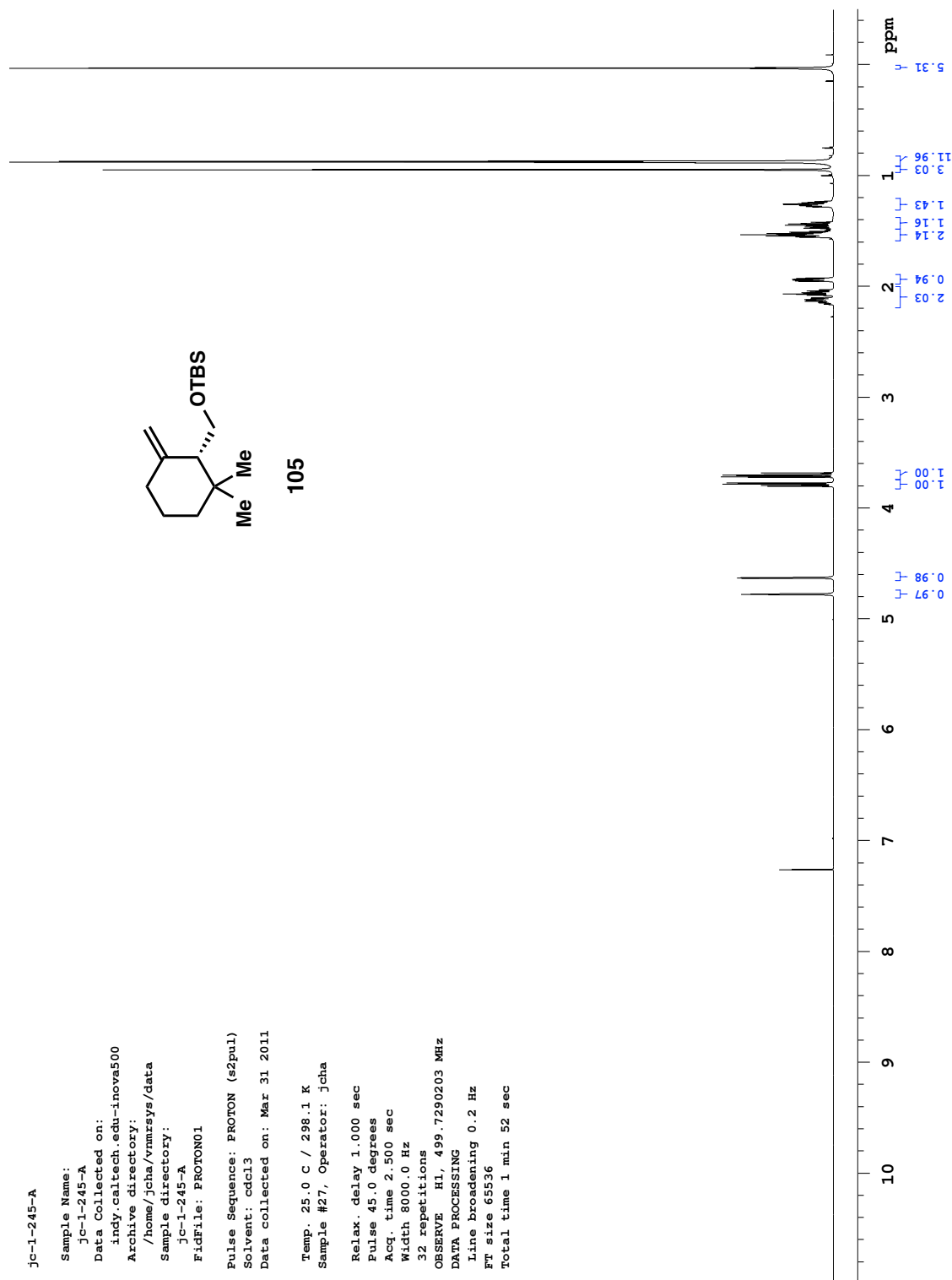
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 31 2011

Temp. 25.0 C / 298.1 K
Sample #27, Operator: jcha

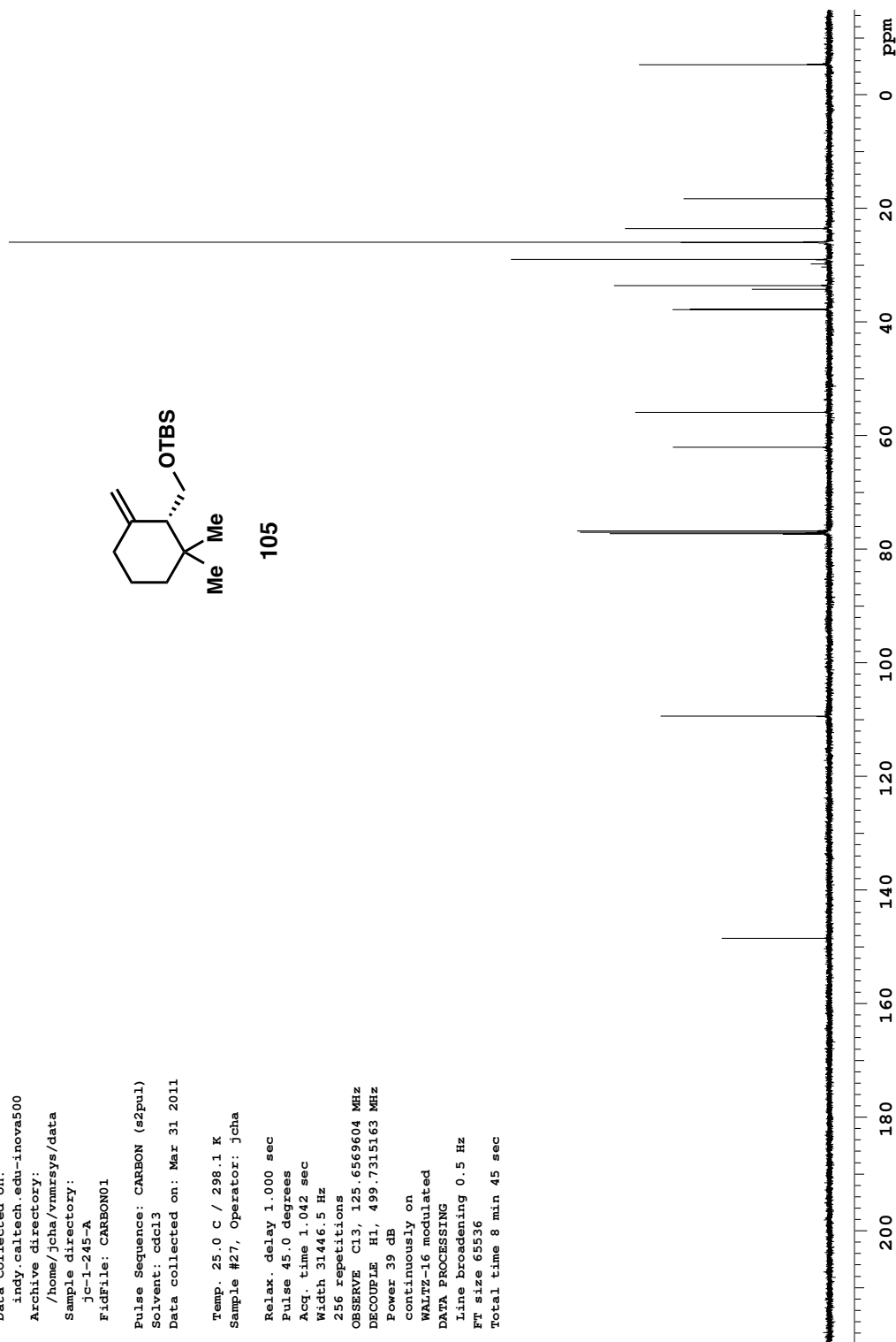
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec



105



105

CC1(C)C=C(C1)C(OC(C)(C)C(C)(C)C)C

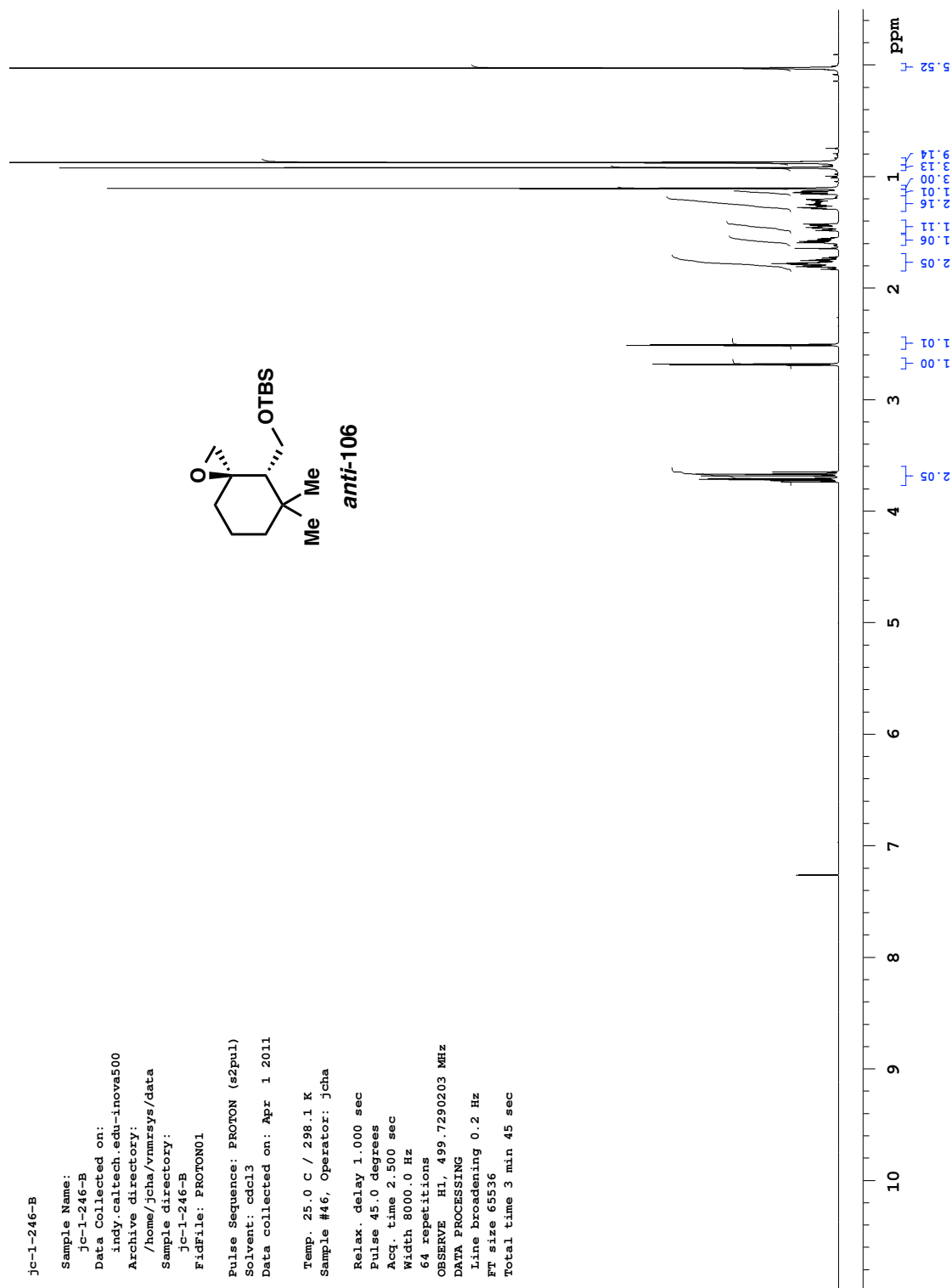
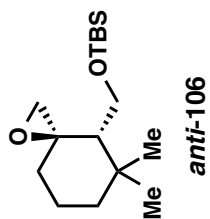
jc-l-246-B

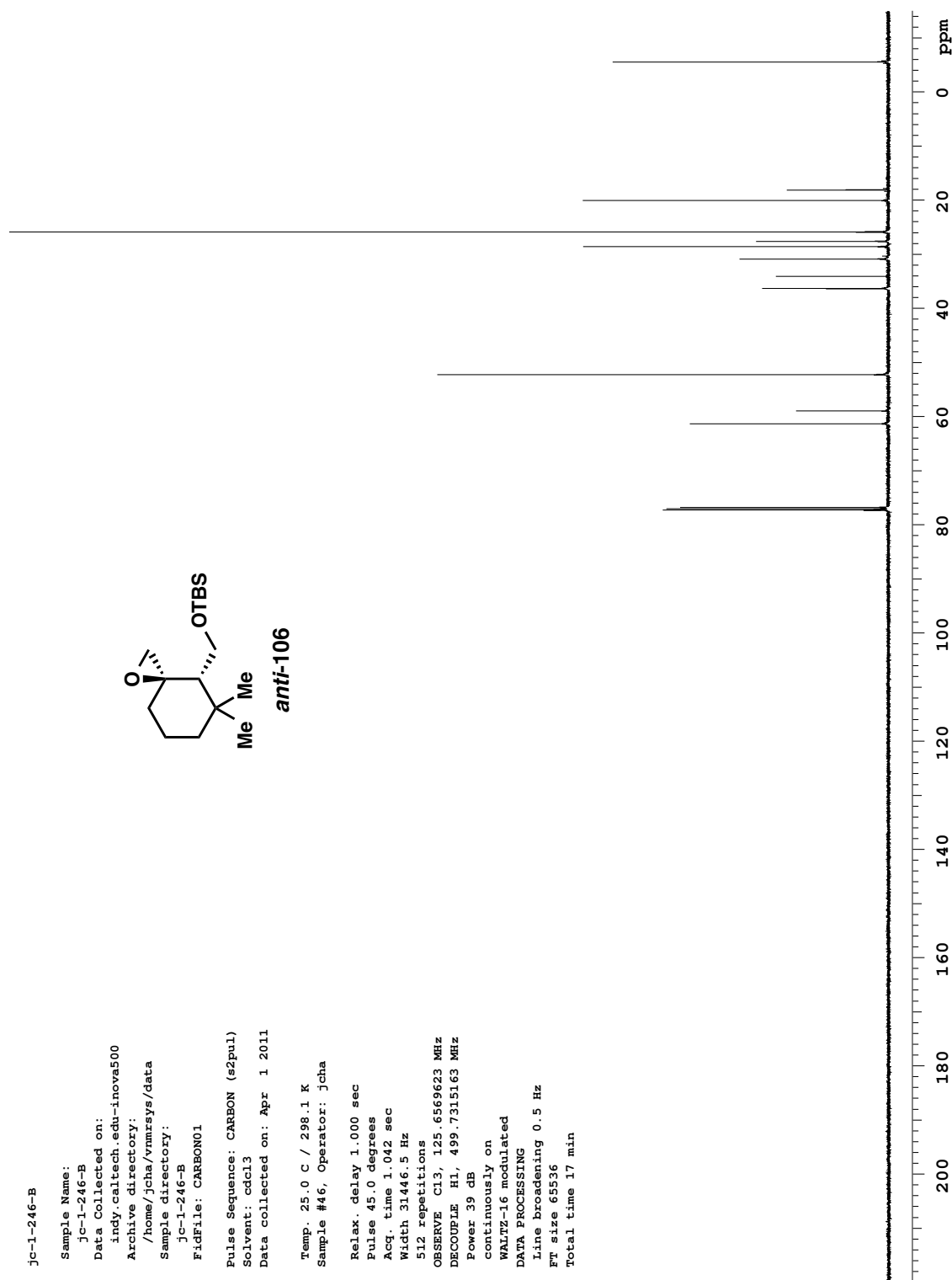
Sample Name:
jc-l-246-B
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-l-246-B
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 1 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 3 min 45 sec





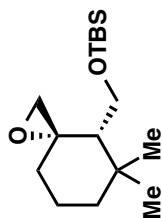
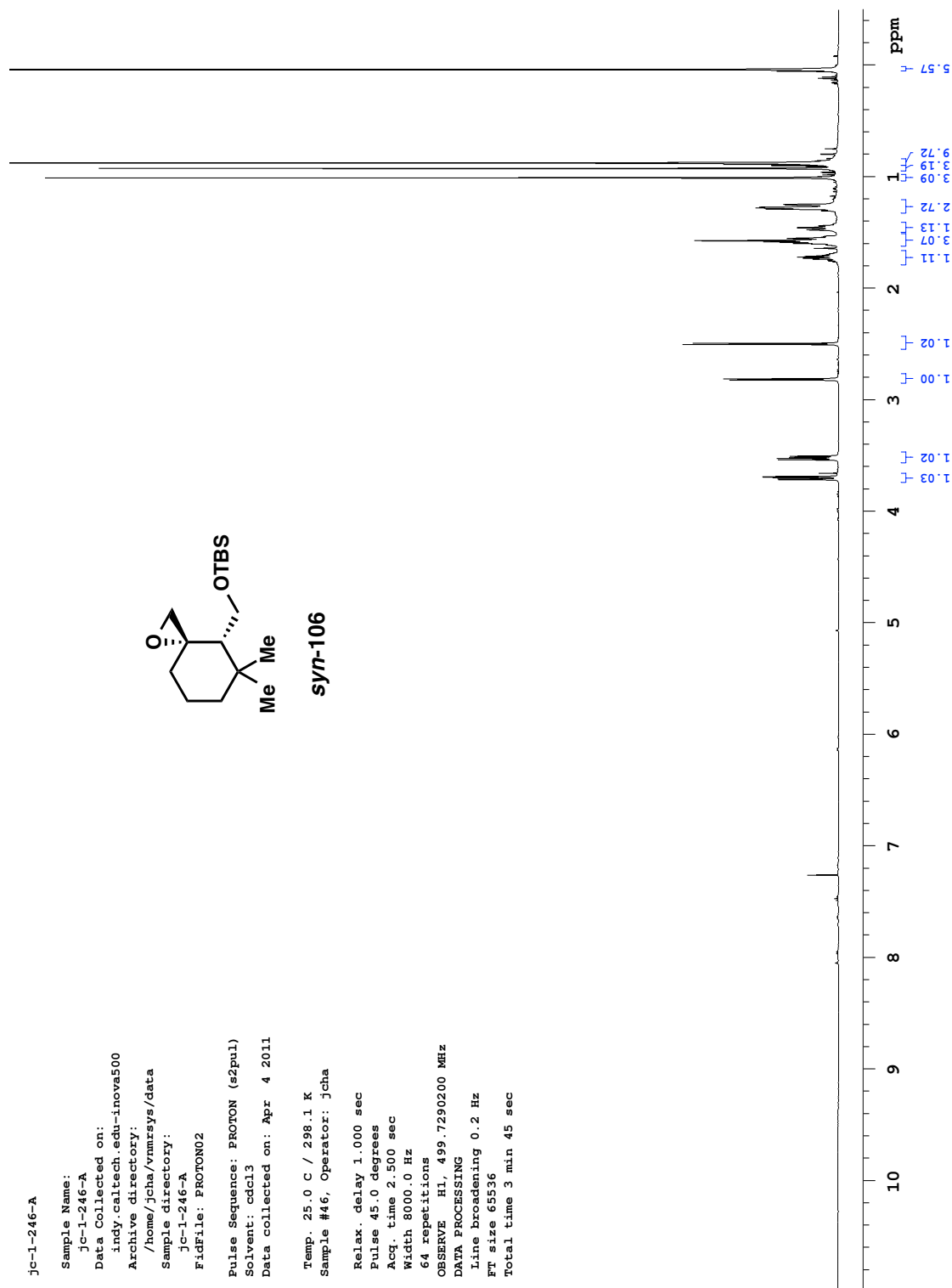
jc-1-246-A

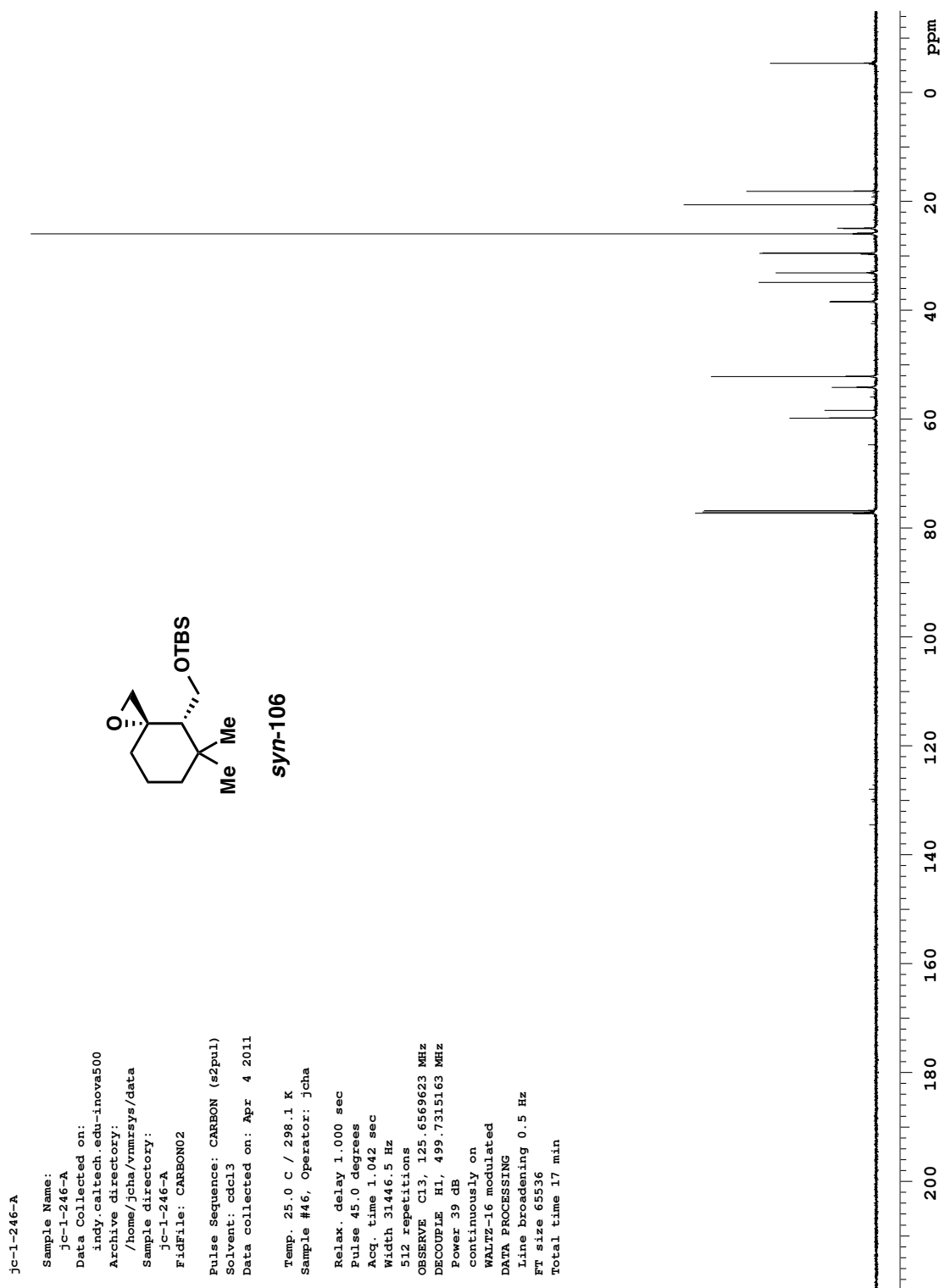
Sample Name:
jc-1-246-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-1-246-A
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 4 2011

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

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290200 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 3 min 45 sec

**syn-106**



jtsy-v-287

Sample Name:
jtsy-v-287f15char1

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/yeomanjo/vnmrSYS/data

Sample directory:
jtsy-v-287f15char1

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 26 2011

Temp. 25.0 C / 298.1 K

Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

16 repetitions

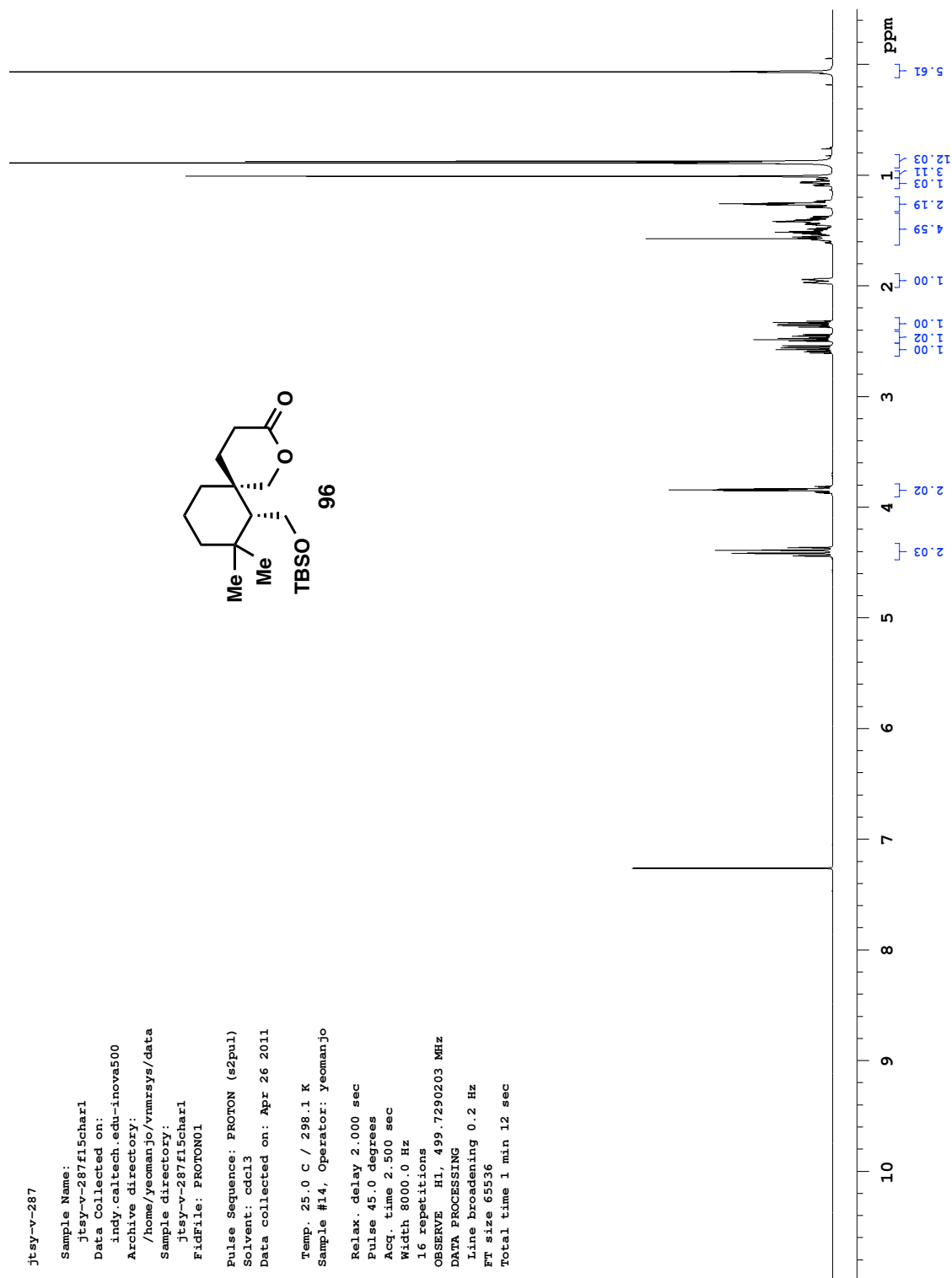
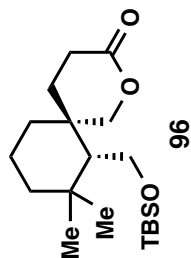
OBSERVE H1, 499.7290203 MHz

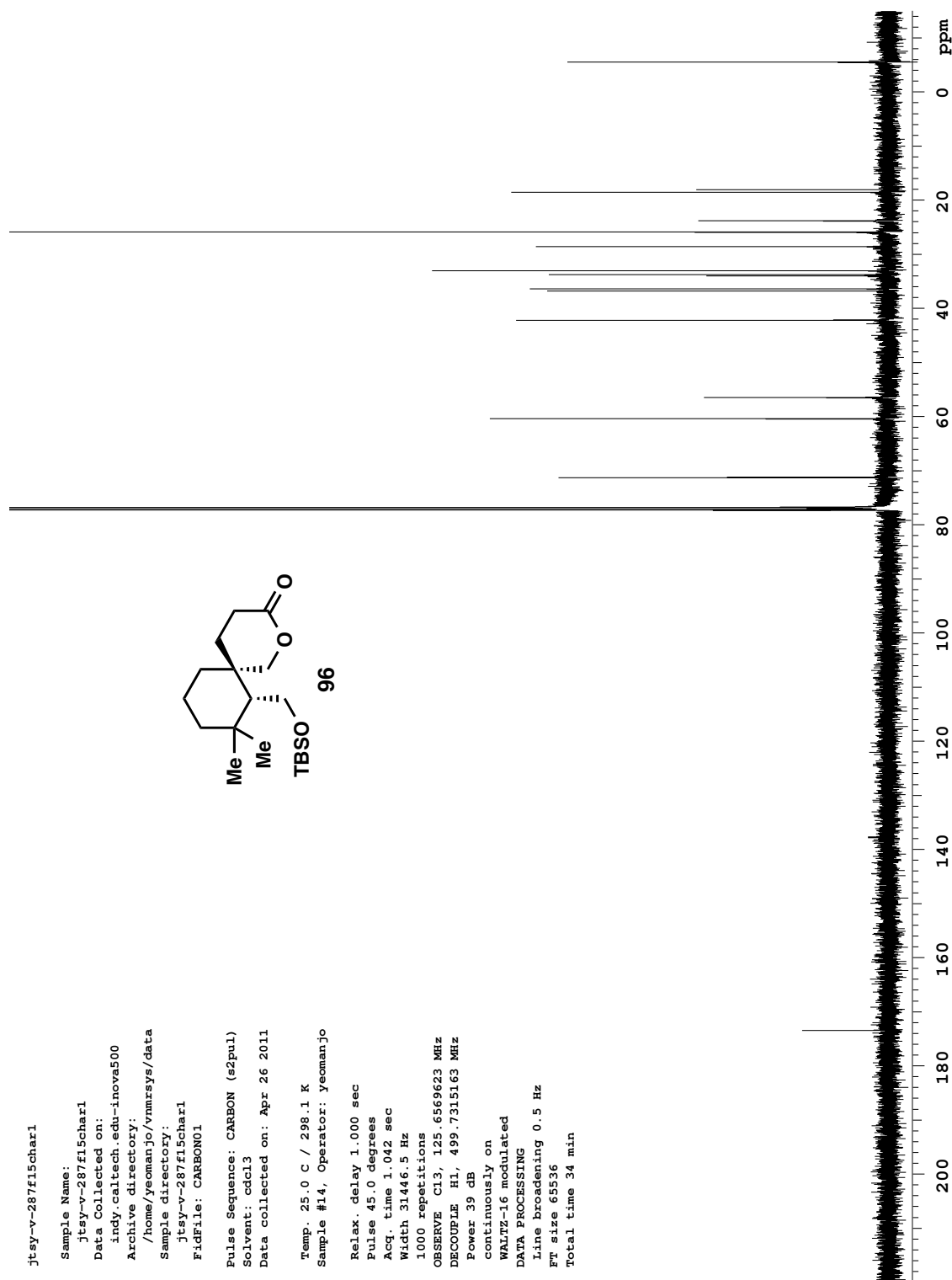
DATA PROCESSING

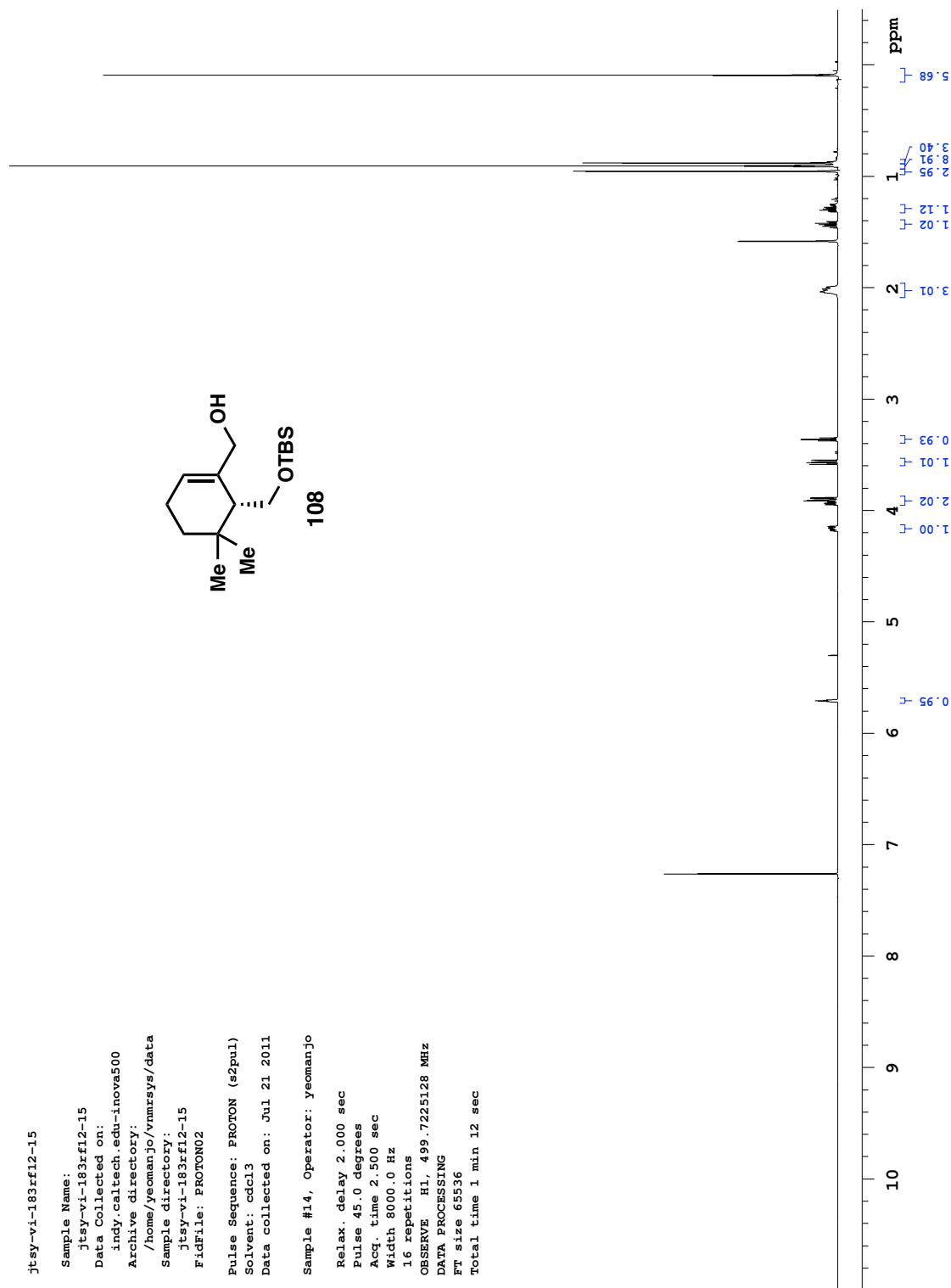
Line broadening 0.2 Hz

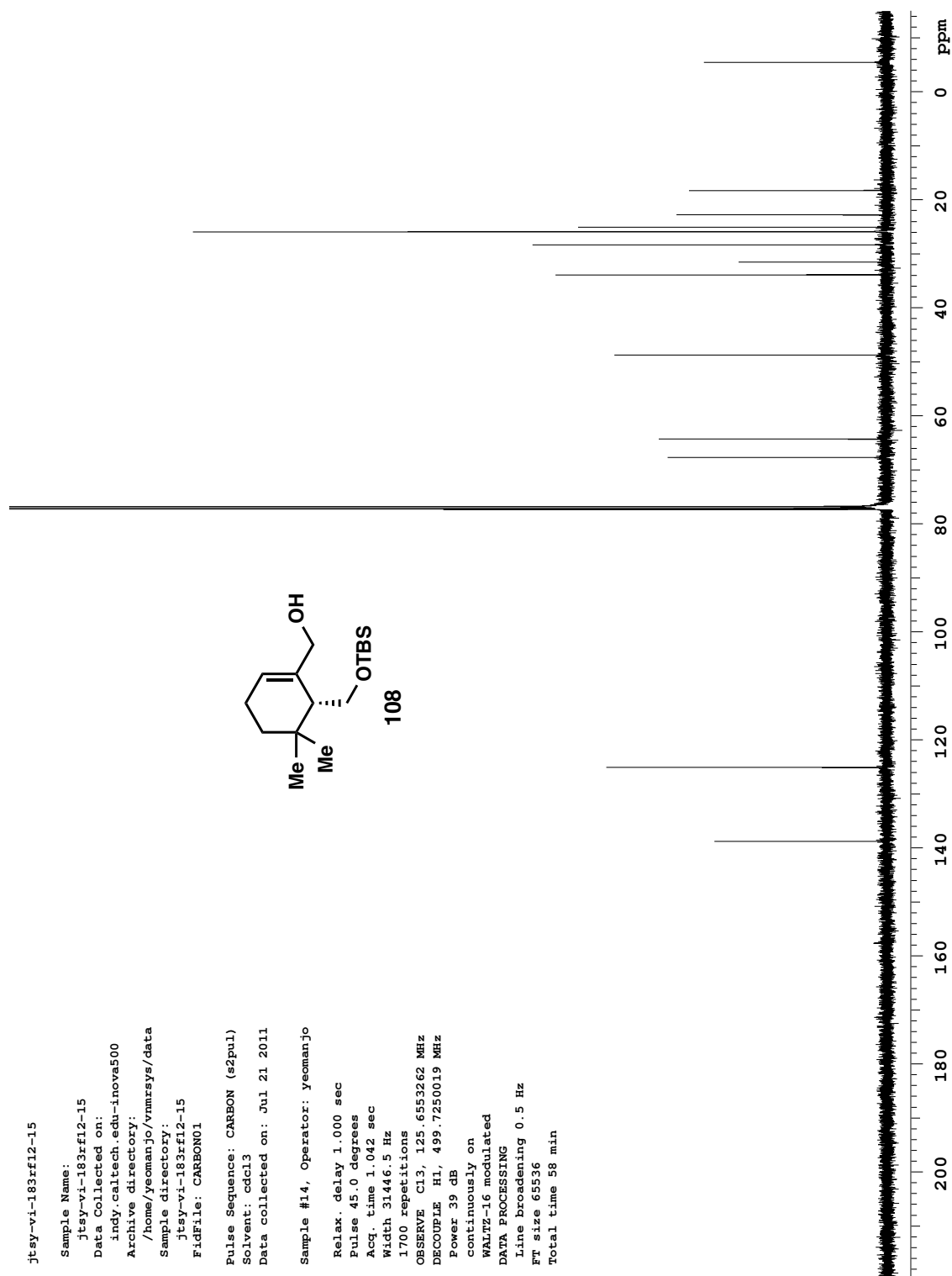
FT size 65536

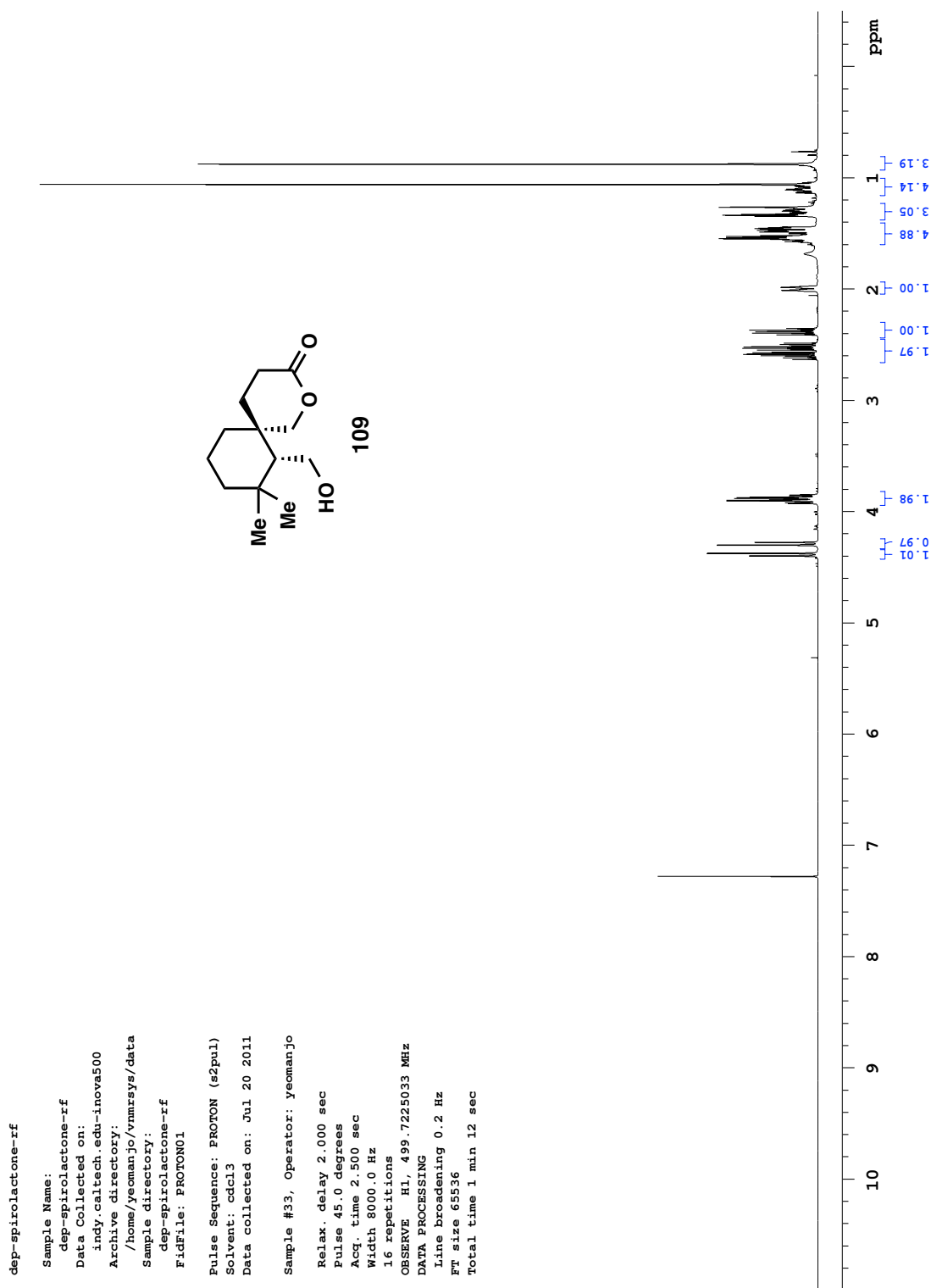
Total time 1 min 12 sec











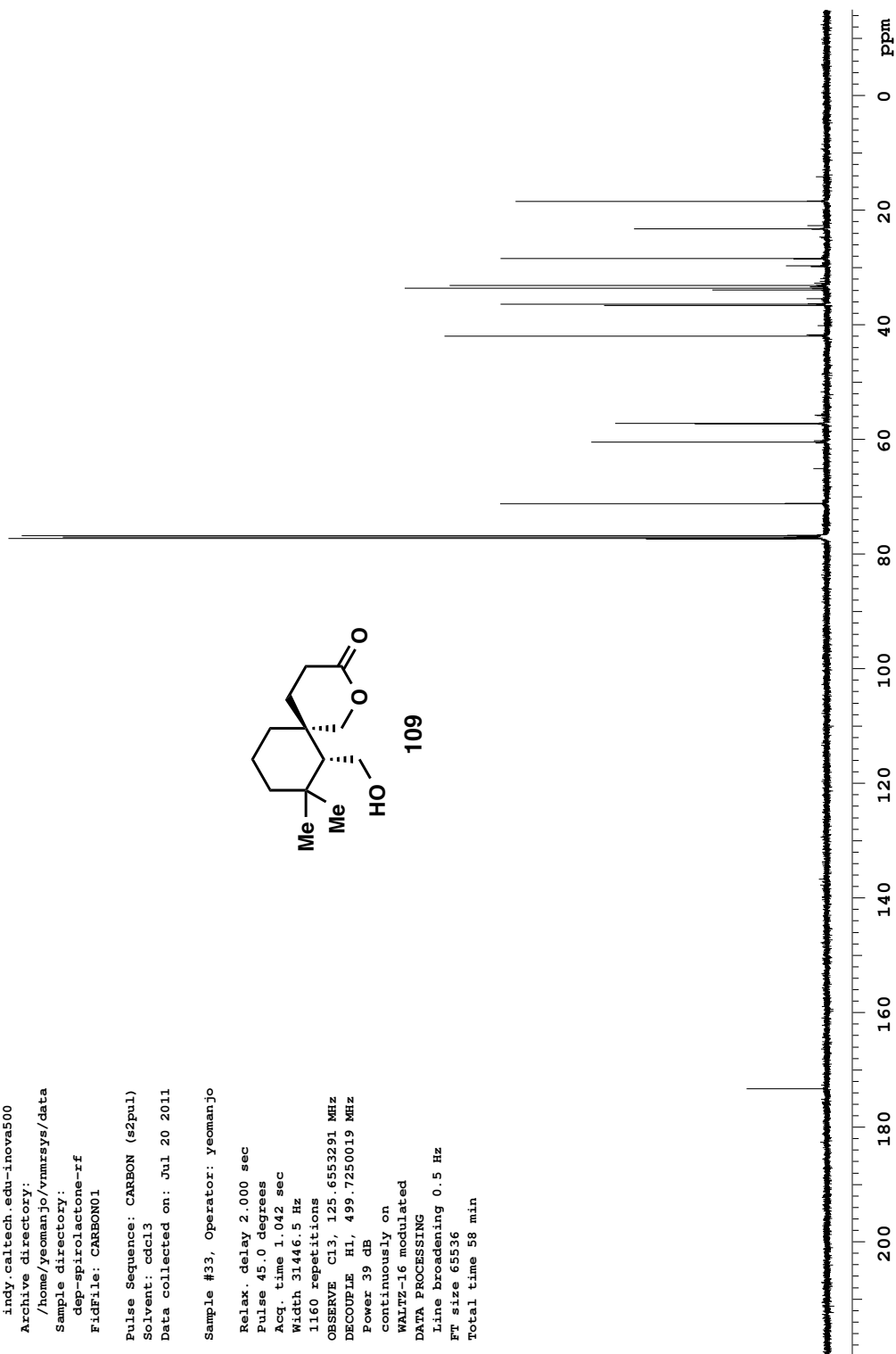
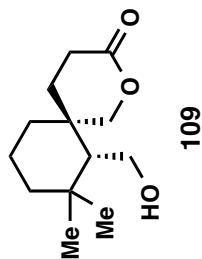
dep-spirolactone-rf

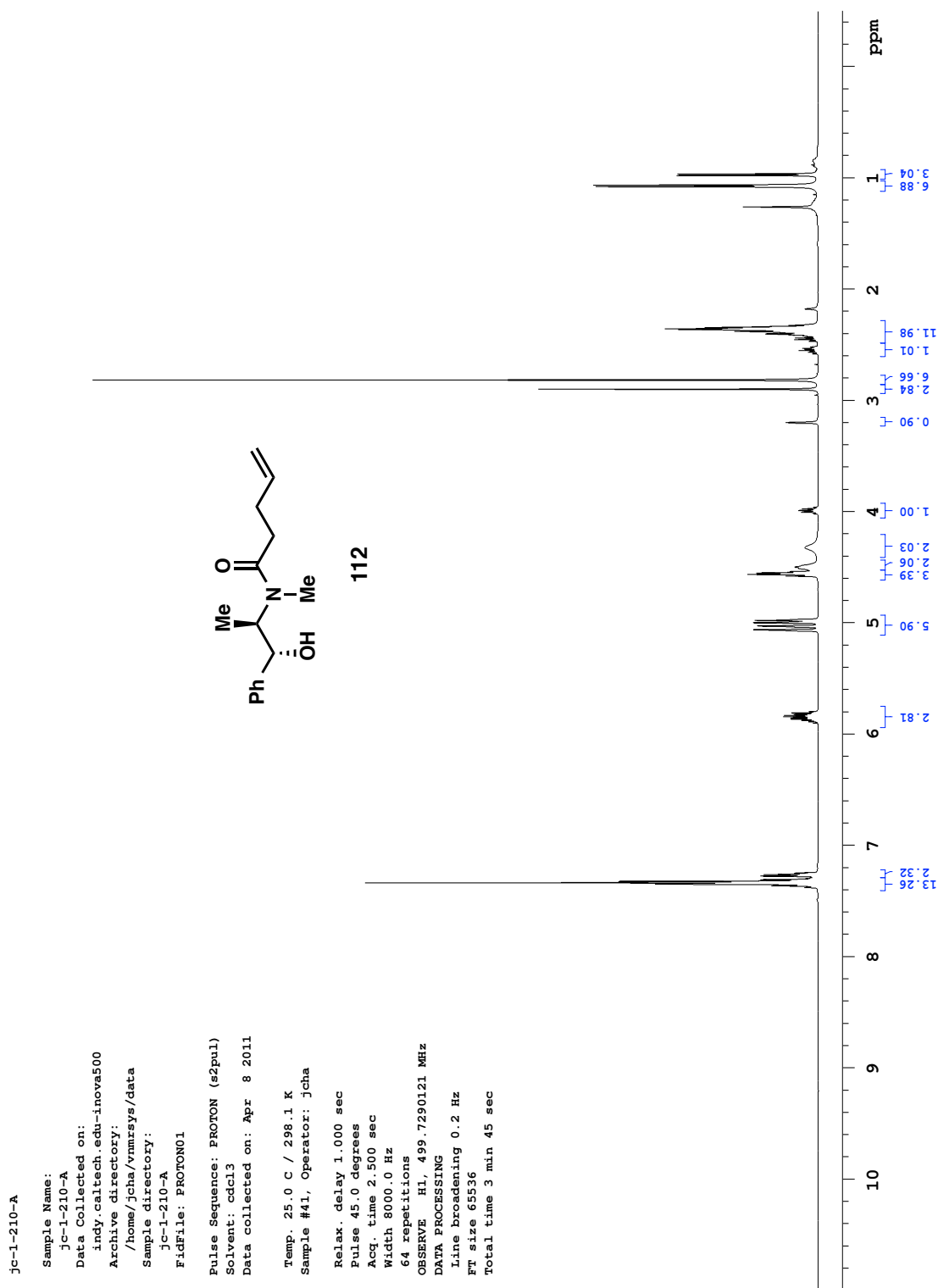
Sample Name:
dep-spirolactone-rf
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/yeomanjo/vnmrsys/data
Sample directory:
dep-spirolactone-rf
FidFile: CARBON01

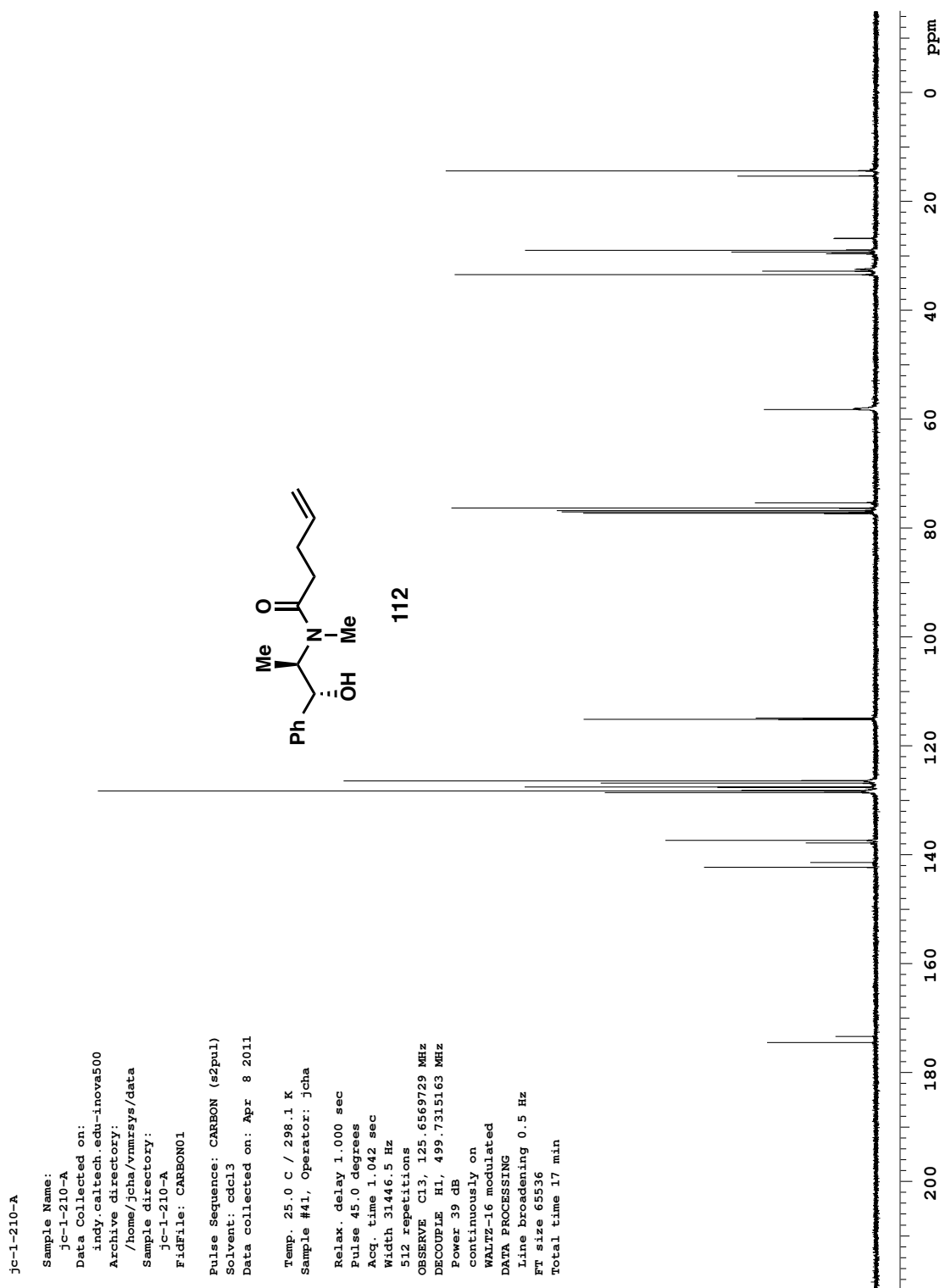
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2011

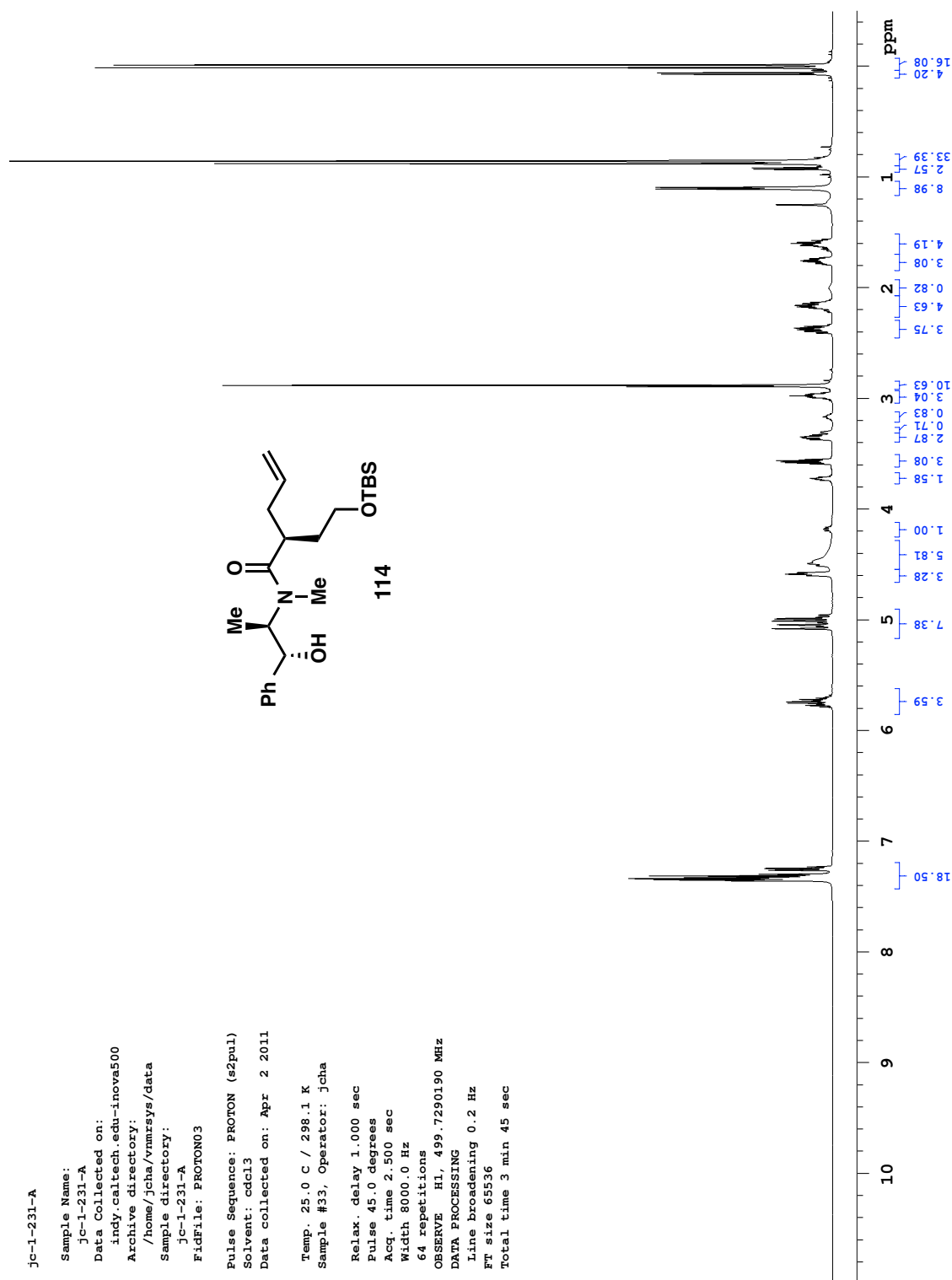
Sample #33, Operator: yeomanjo

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1160 repetitions
OBSERVE C13, 125.6553291 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Ft size 65536
Total time 58 min







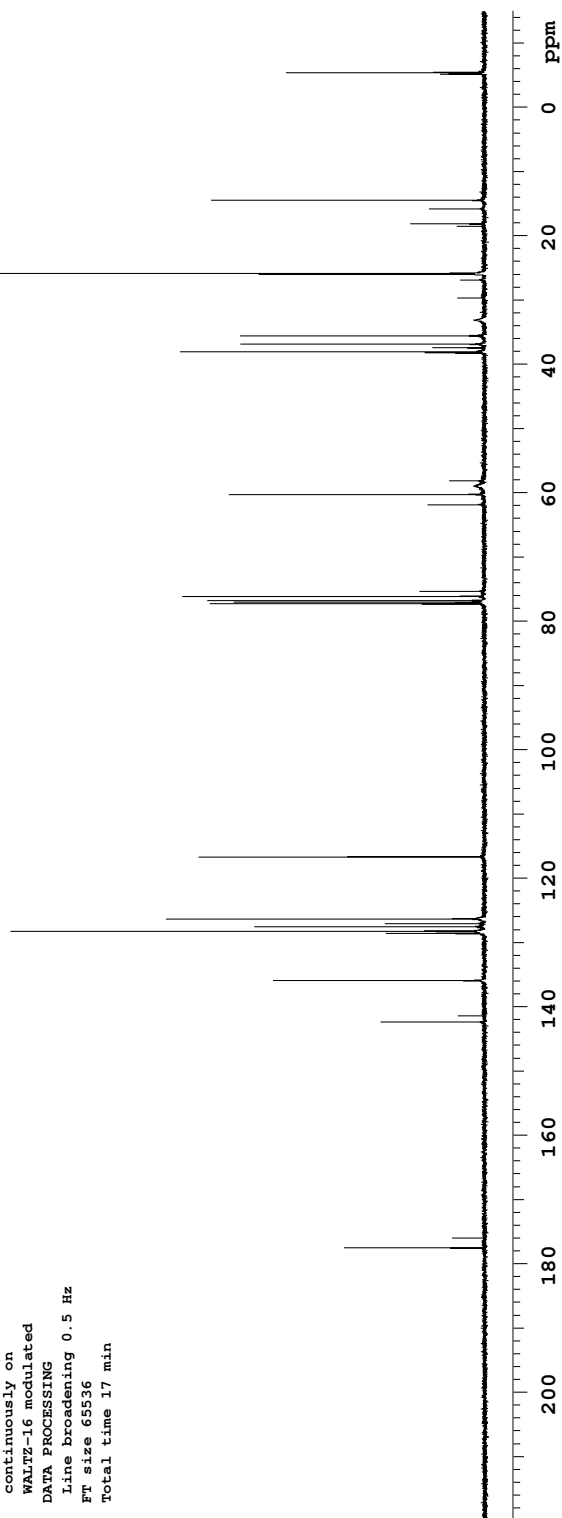
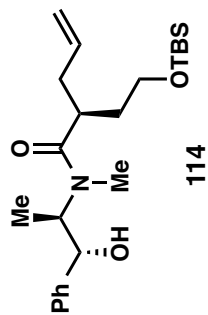


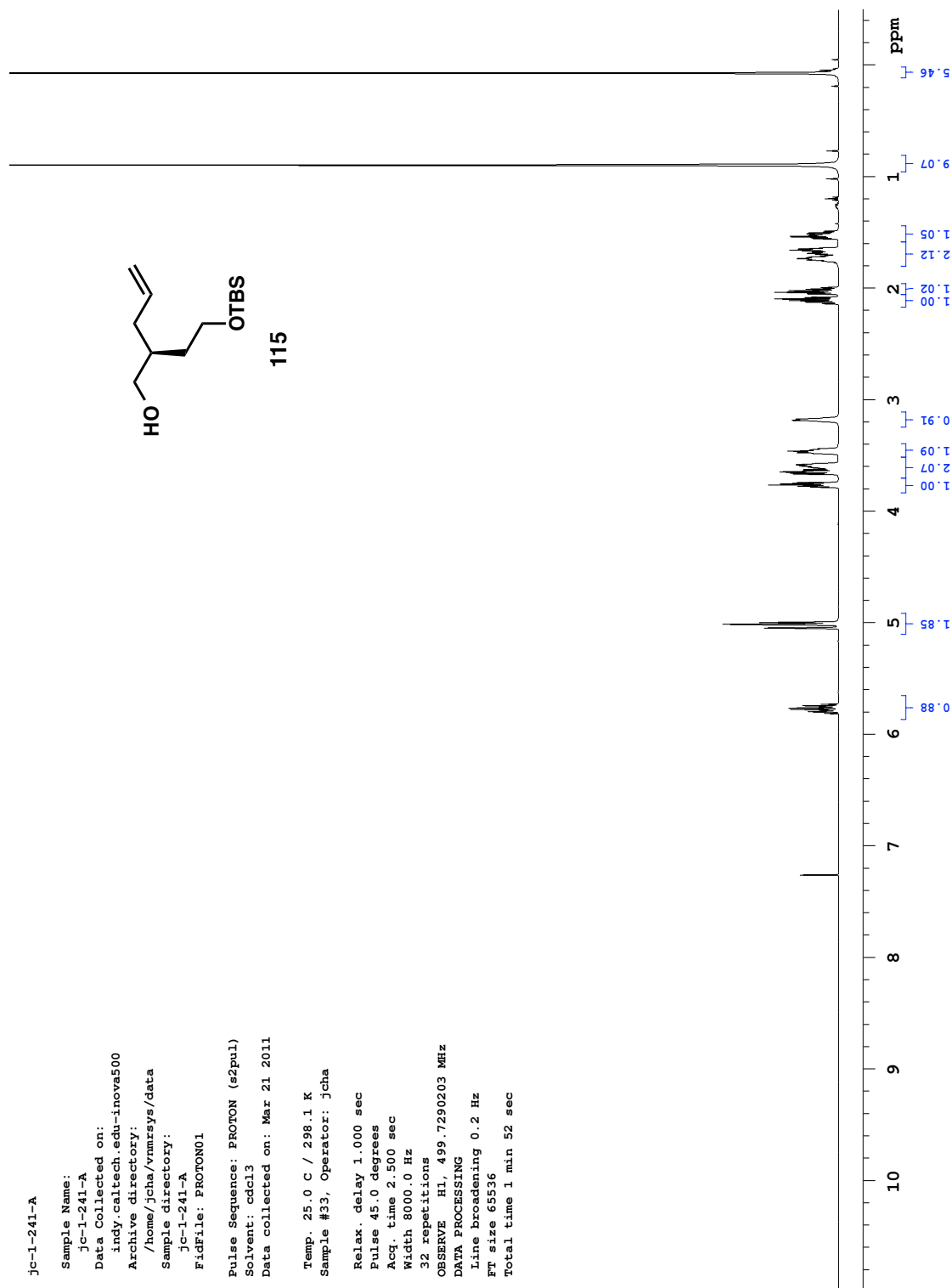
jc-1-231-A

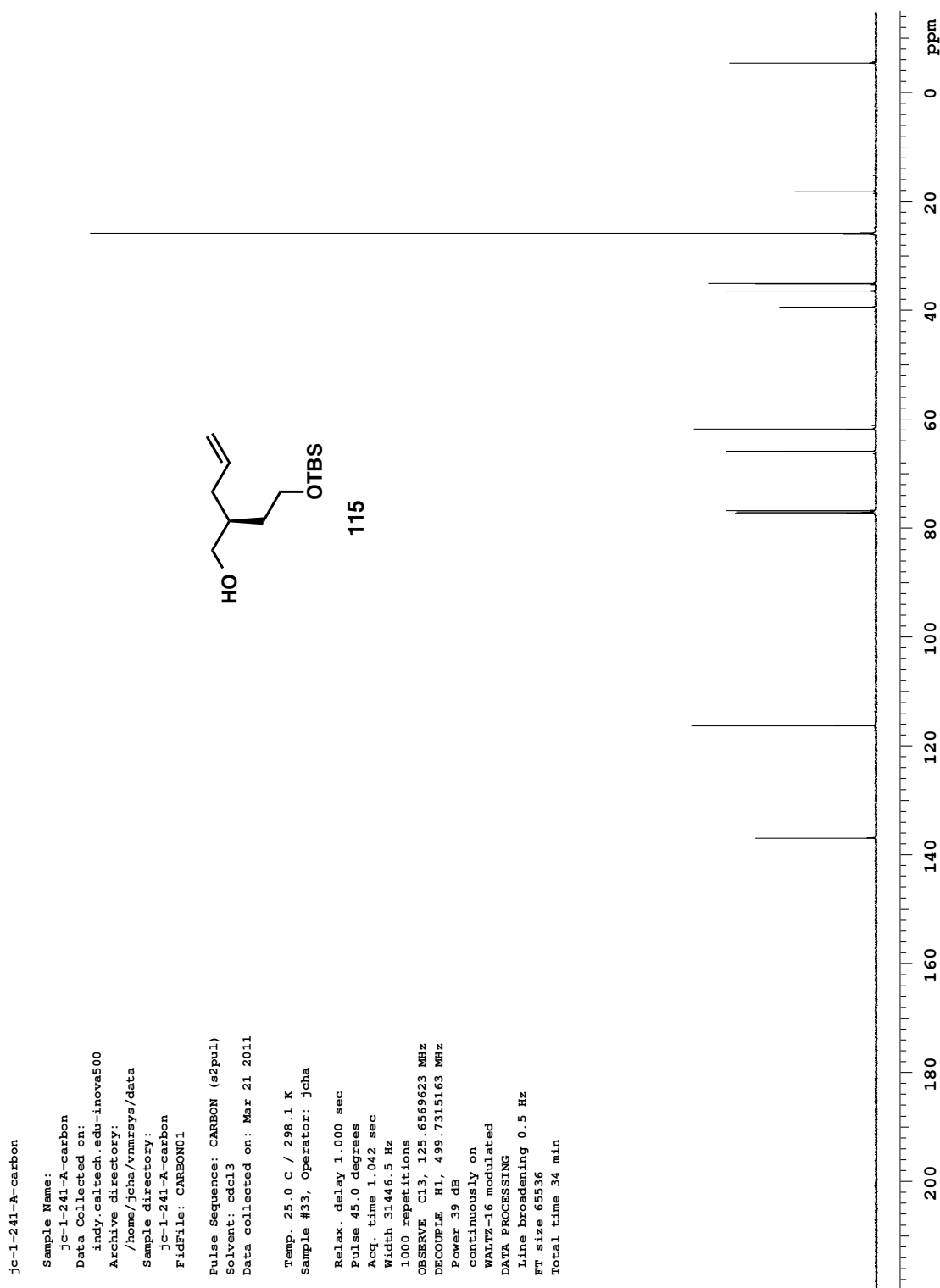
```
Sample Name:
jc-1-231-A
Data Collected on:
indy.caltech.edu-inoa500
Archive directory:
/home/jcha/vmrsys/data
Sample directory:
jc-1-231-A
FidFile: CARBON02
```

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 2 2011

```
Temp. 25.0 C / 298.1 K
Sample #33, Operator: jcha
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
512 repetitions
OBSERVE C13, 125.659671 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 17 min
```







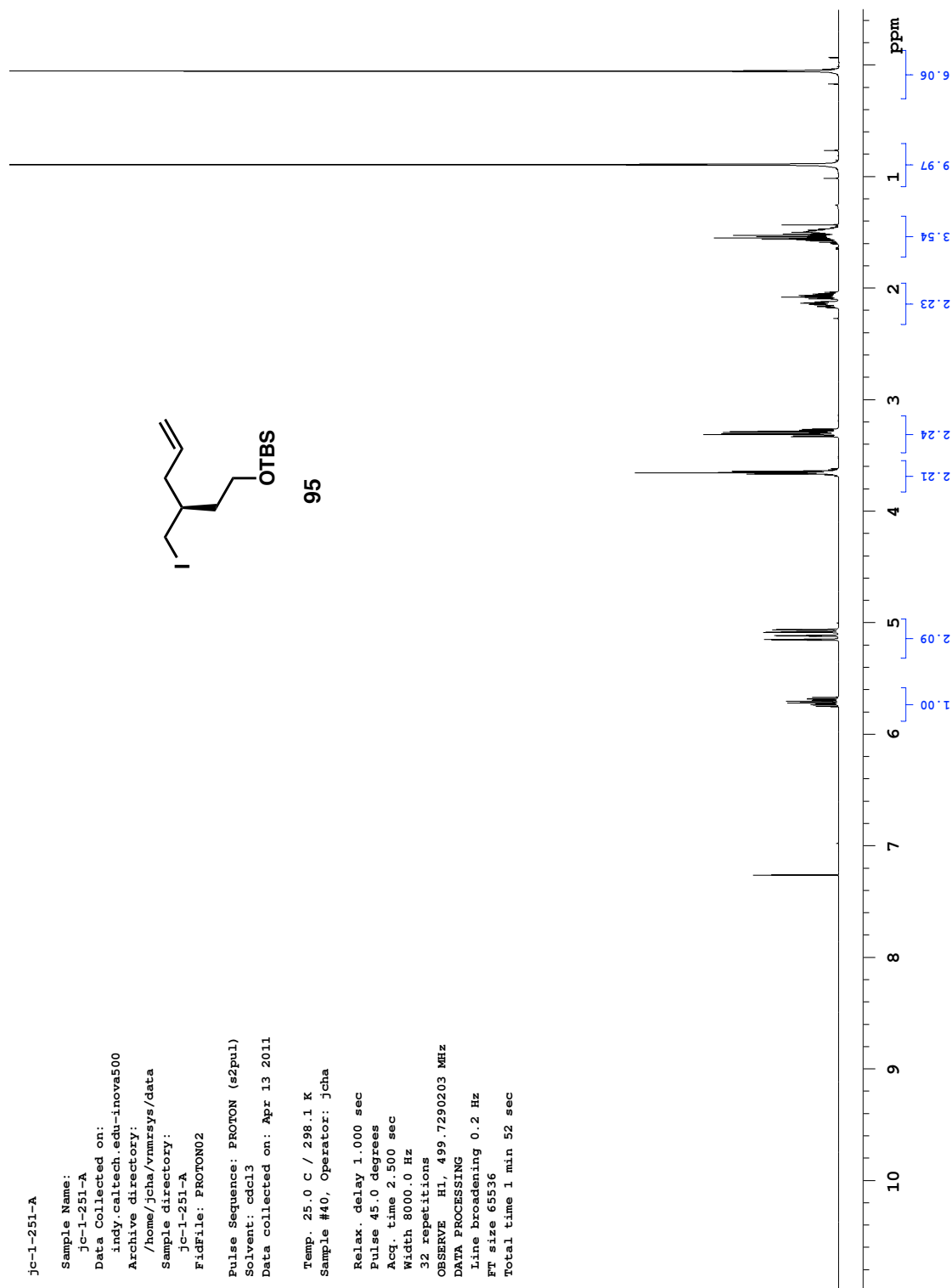
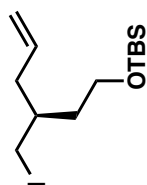
jc-1-251-A

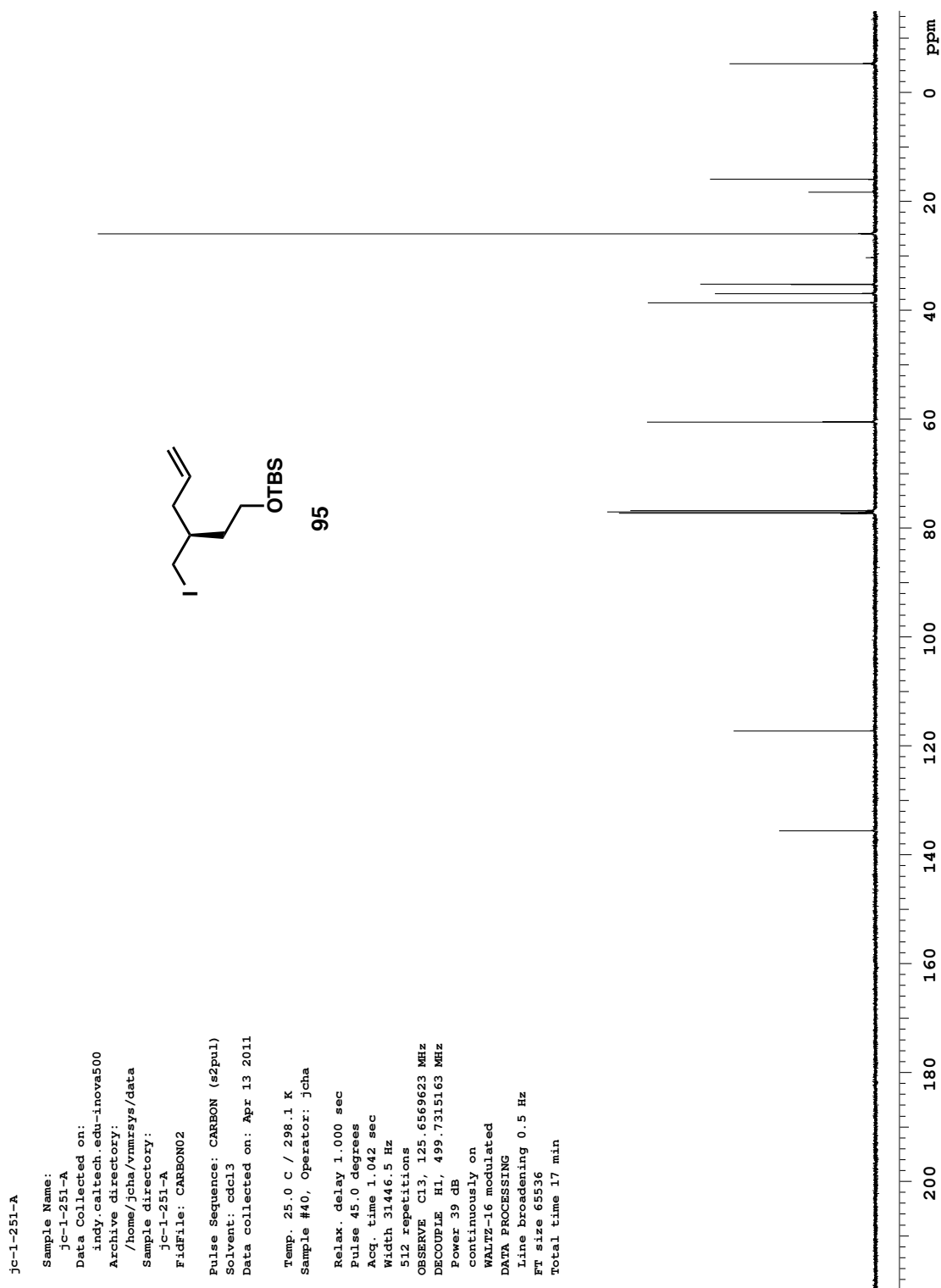
Sample Name:
jc-1-251-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-1-251-A
FidFile: PROTON02

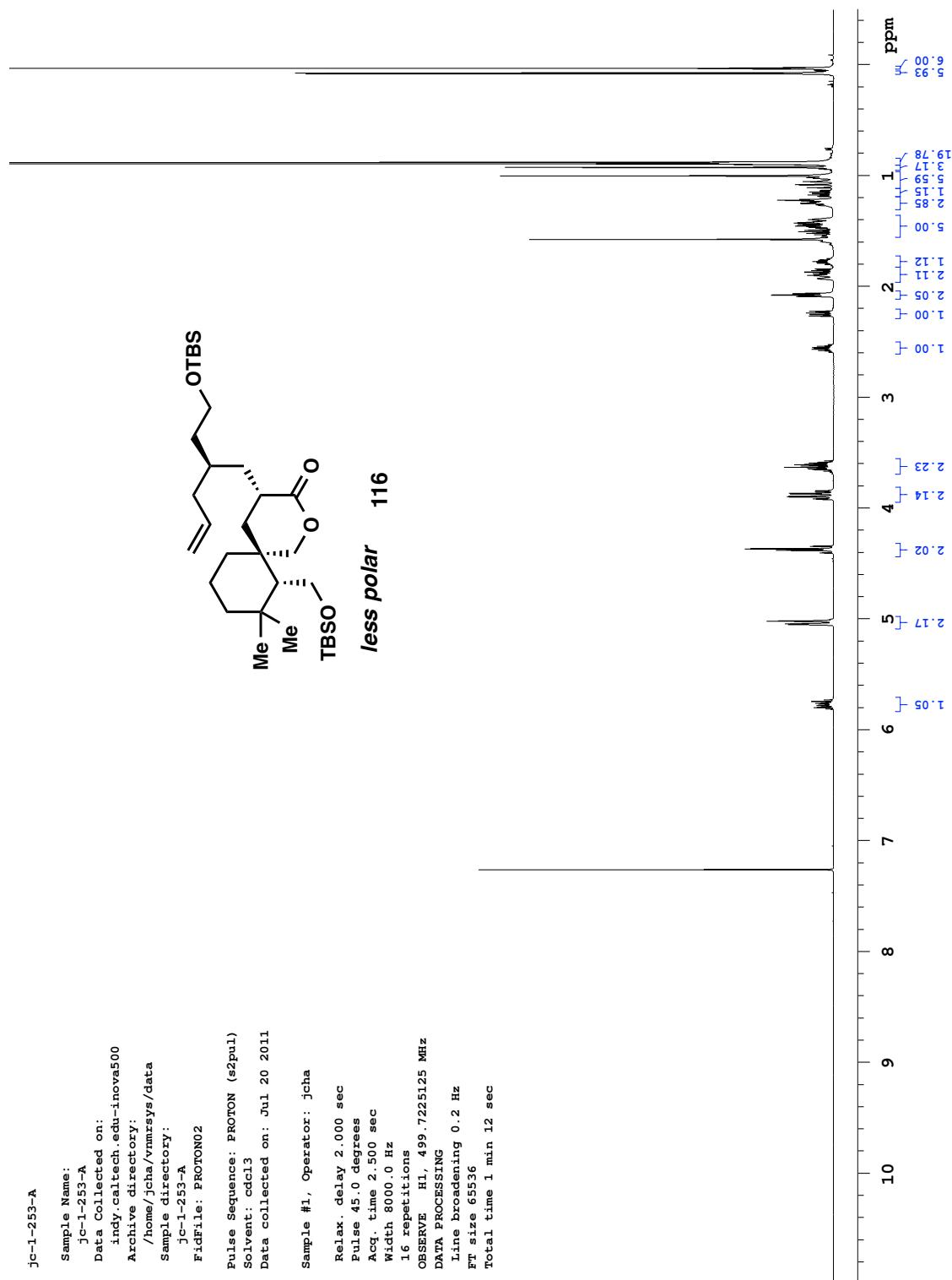
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 13 2011

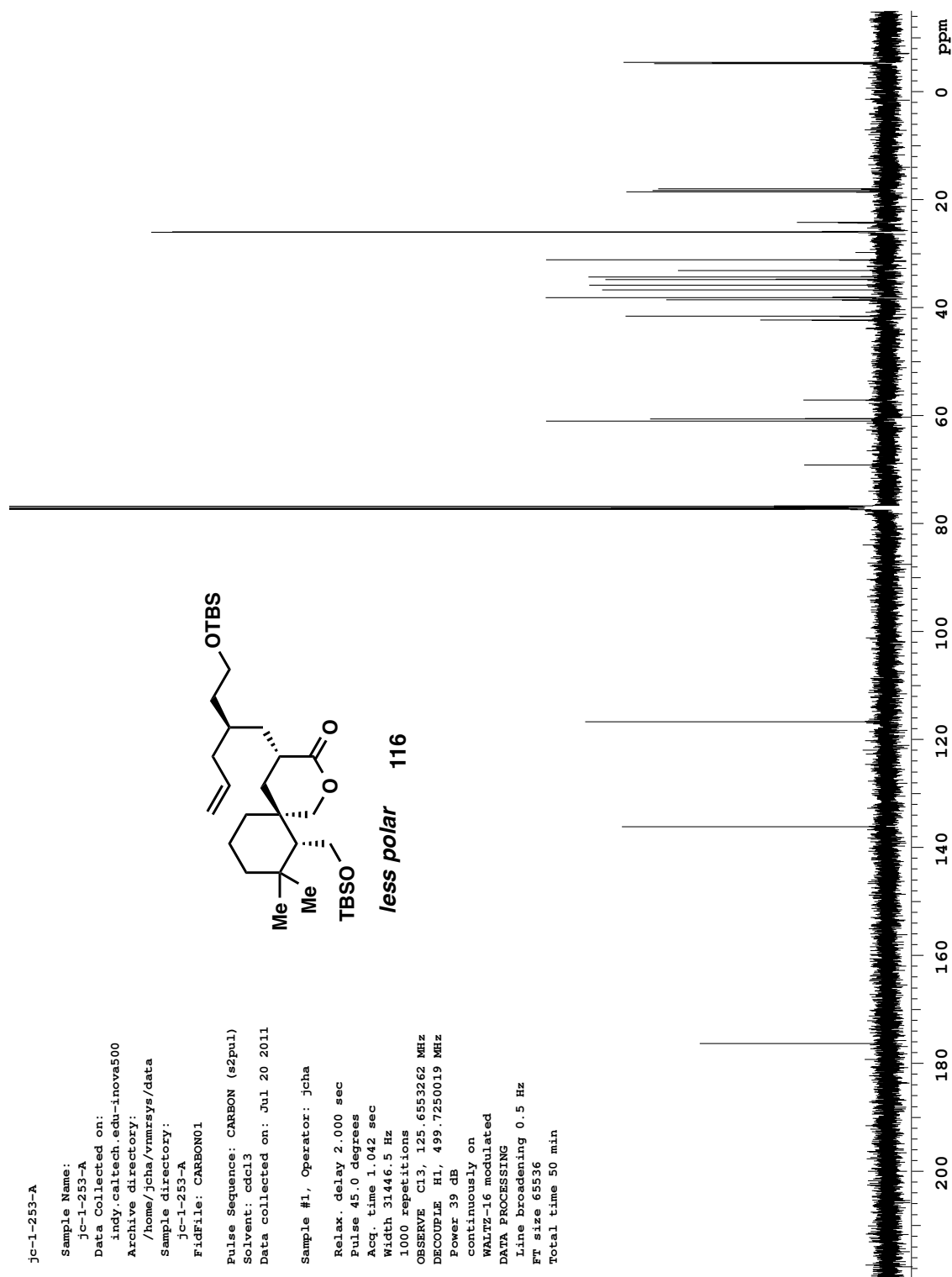
Temp. 25.0 C / 298.1 K
Sample #40, Operator: jcha

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min 52 sec









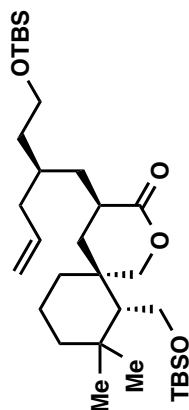
jc-1-253-B

Sample Name:
jc-1-253-B
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-1-253-B
Fidfile: PROTON01

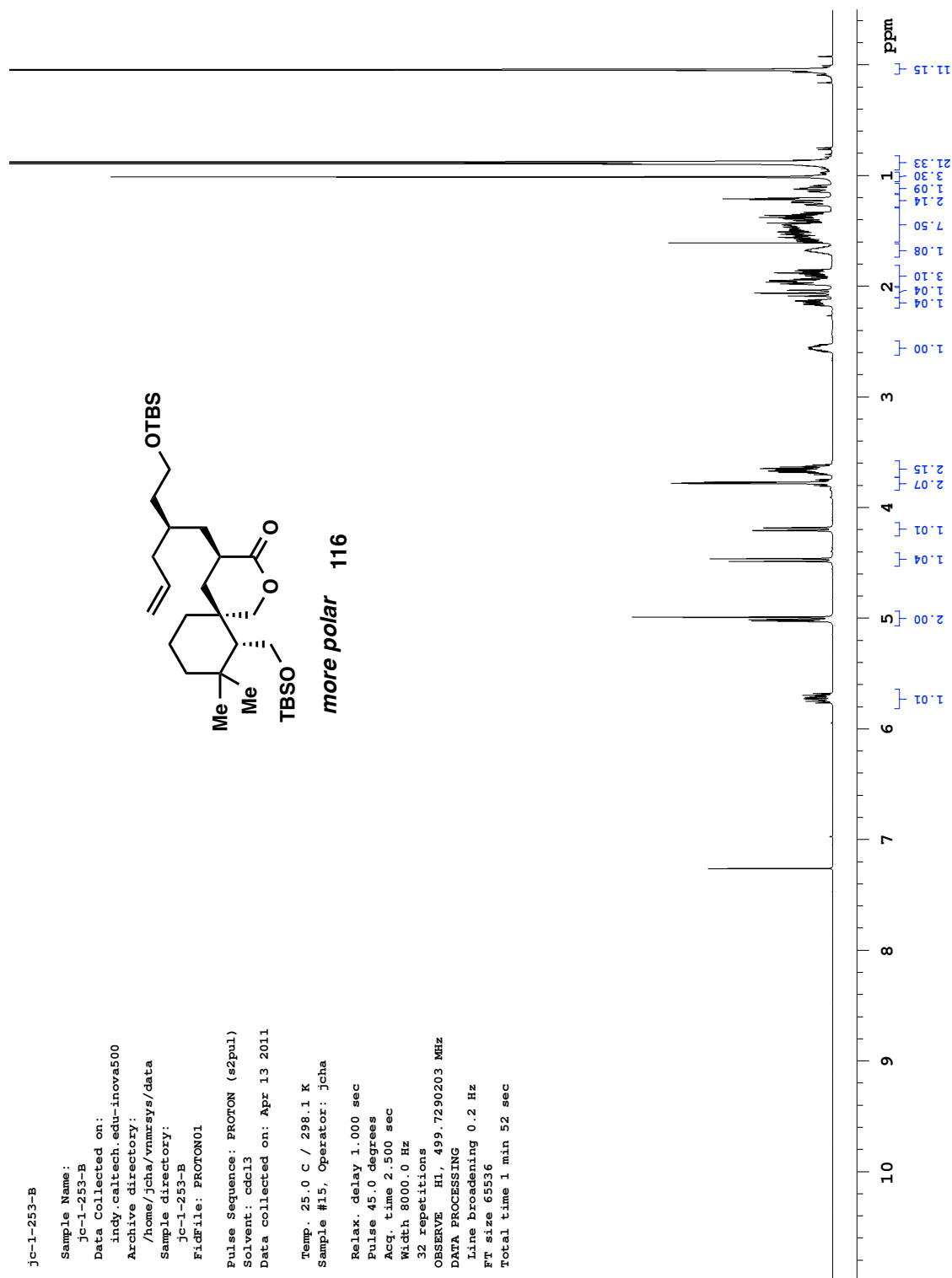
Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Apr 13 2011

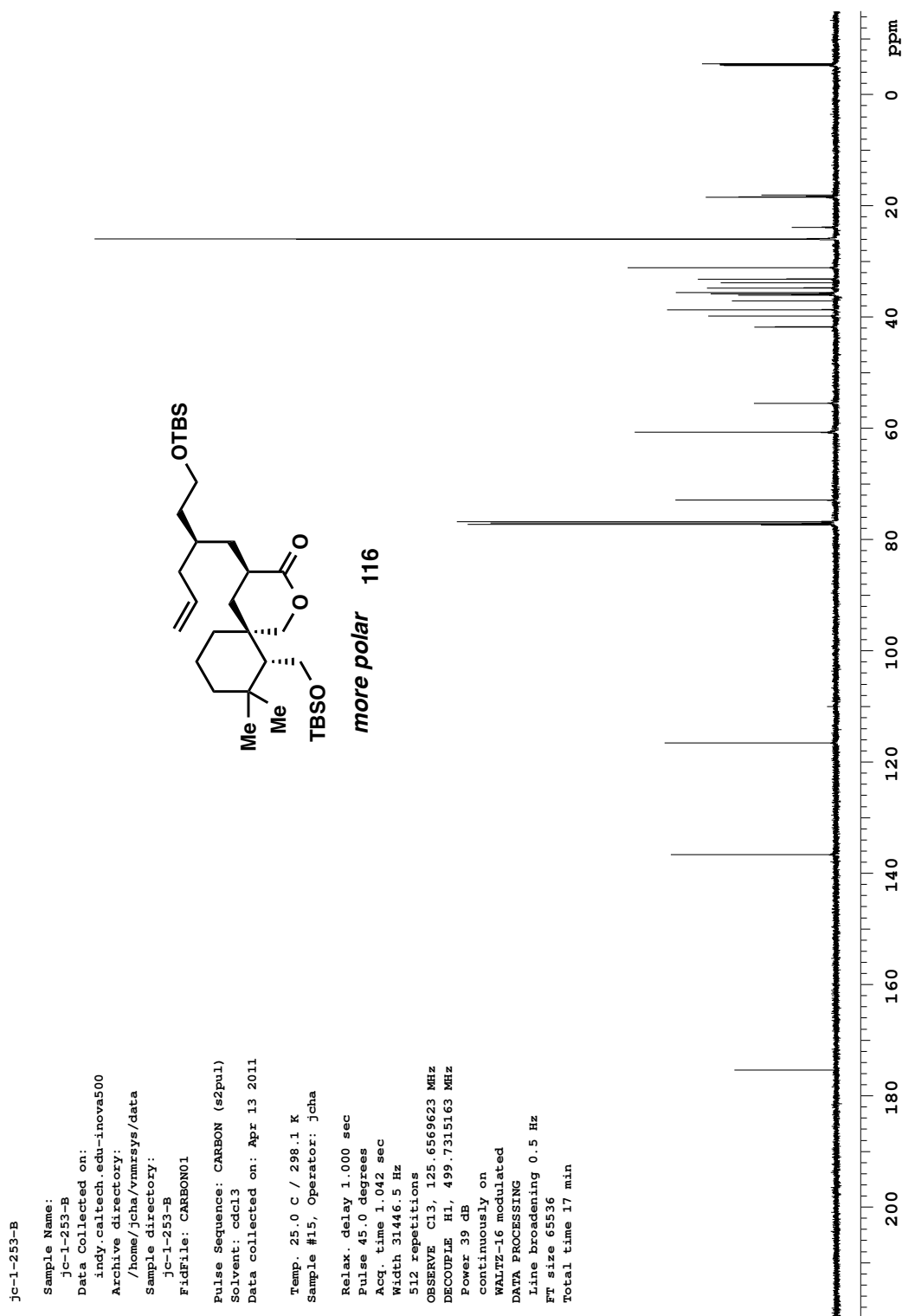
Temp. 25.0 C / 298.1 K
Sample #15, Operator: jcha

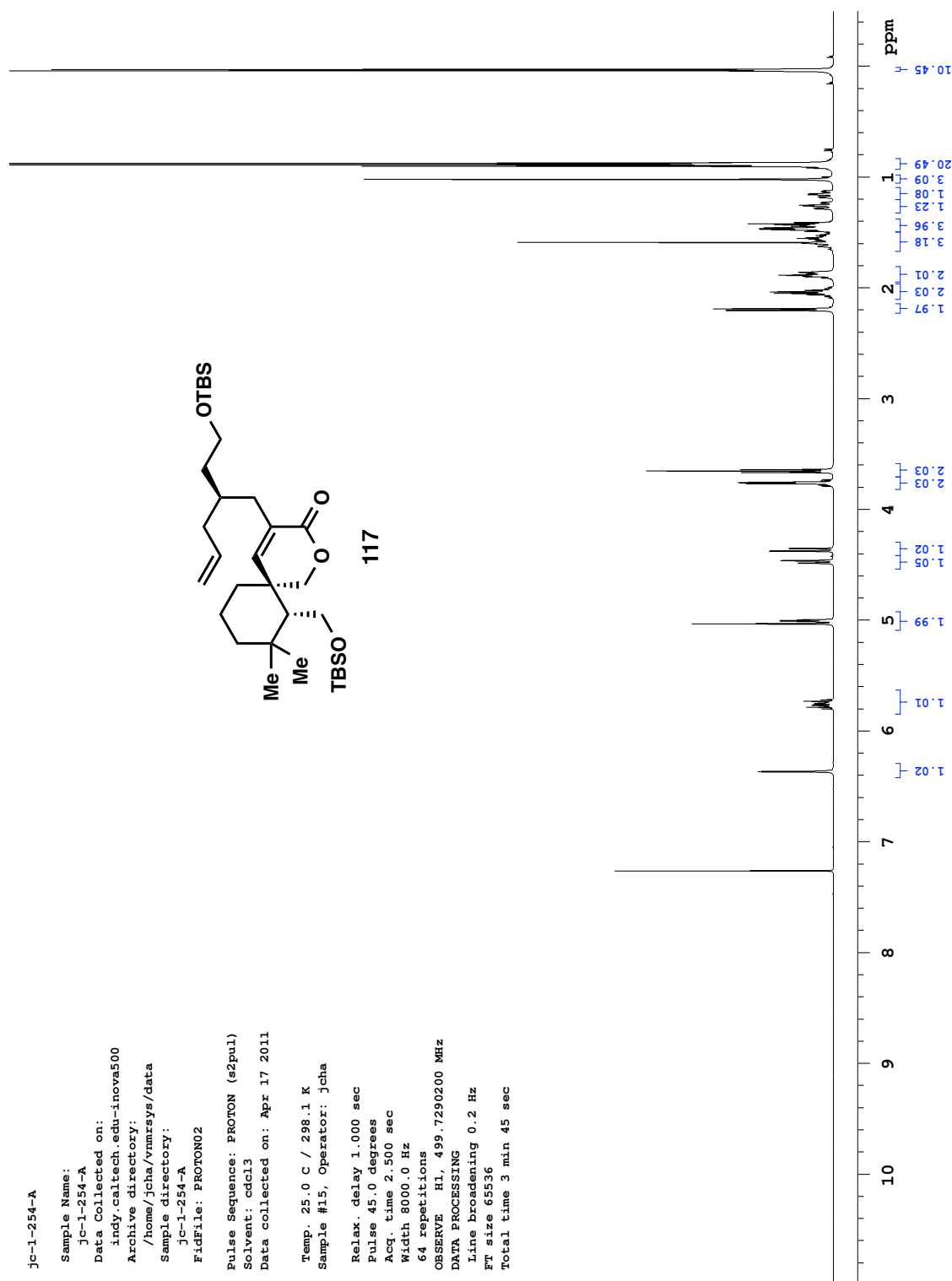
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec



more polar 116







jc-1-254-A

Sample Name:

jc-1-254-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vmrsys/data

Sample directory:

jc-1-254-A

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 17 2011

Temp. 25.0 C / 298.1 K

Sample #15, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1500 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

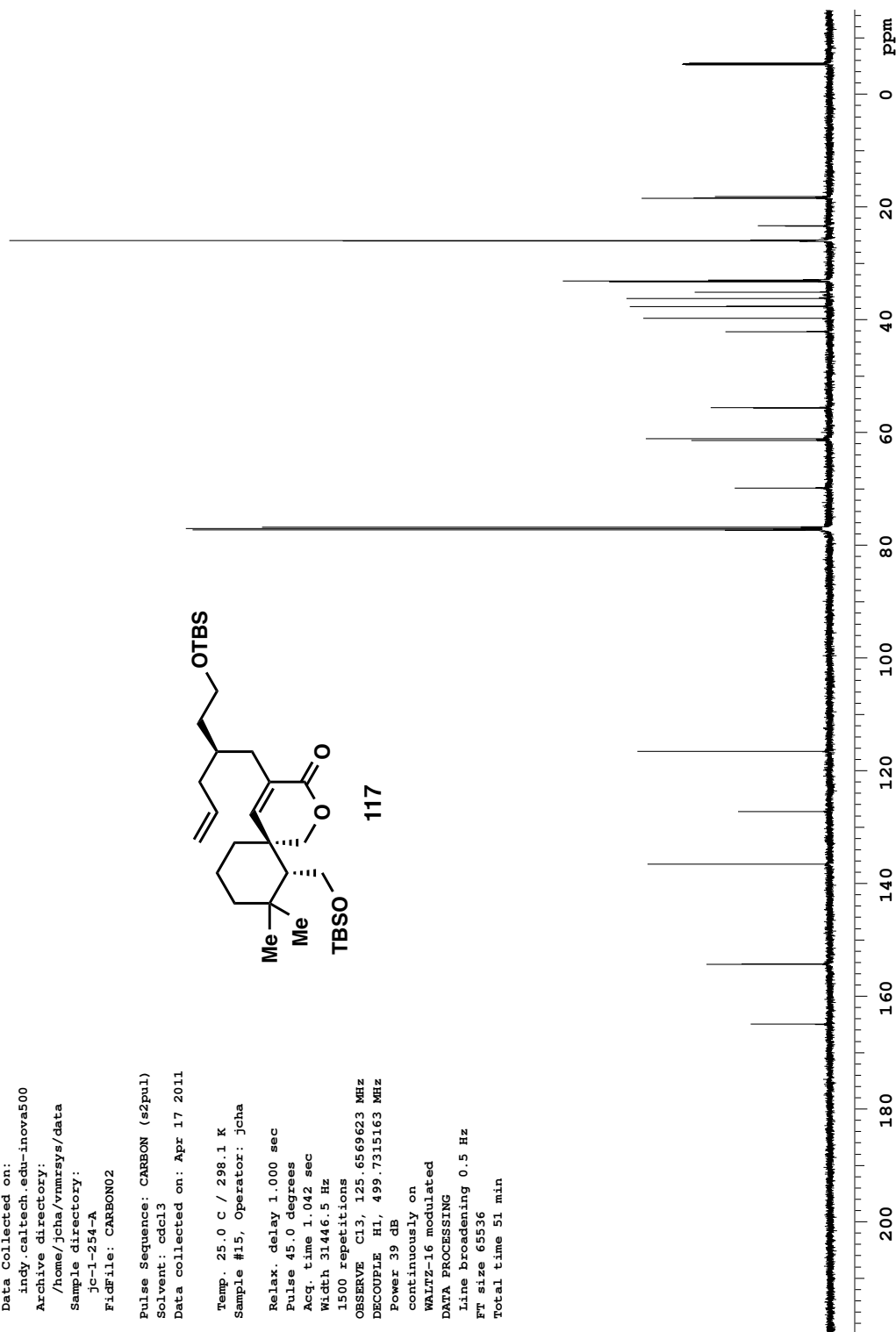
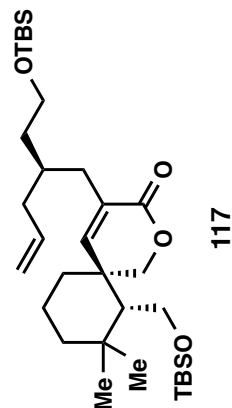
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

Ft size 65536

Total time 51 min



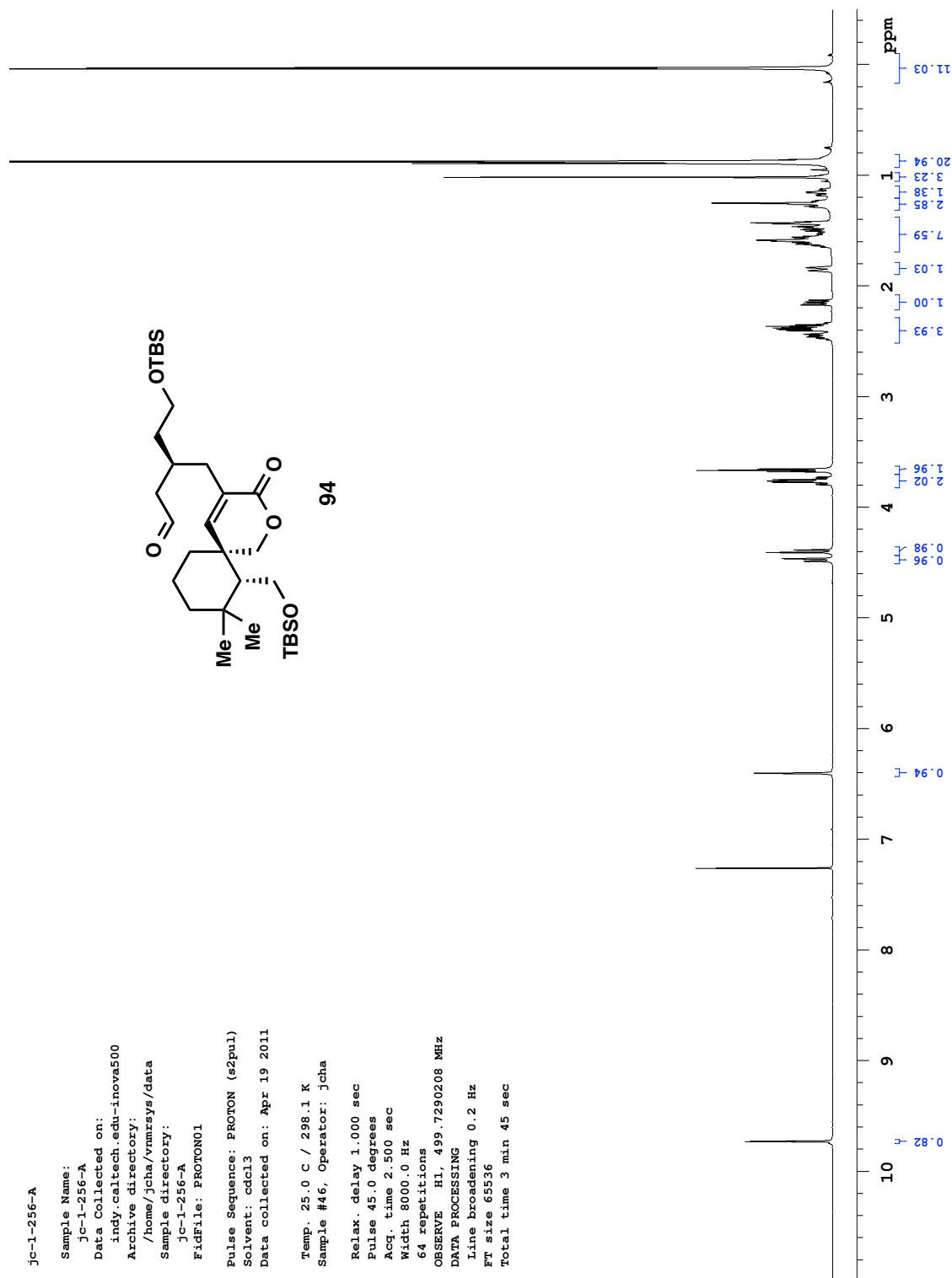
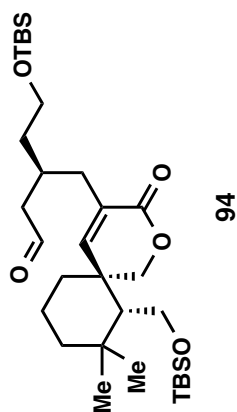
jc-1-256-A

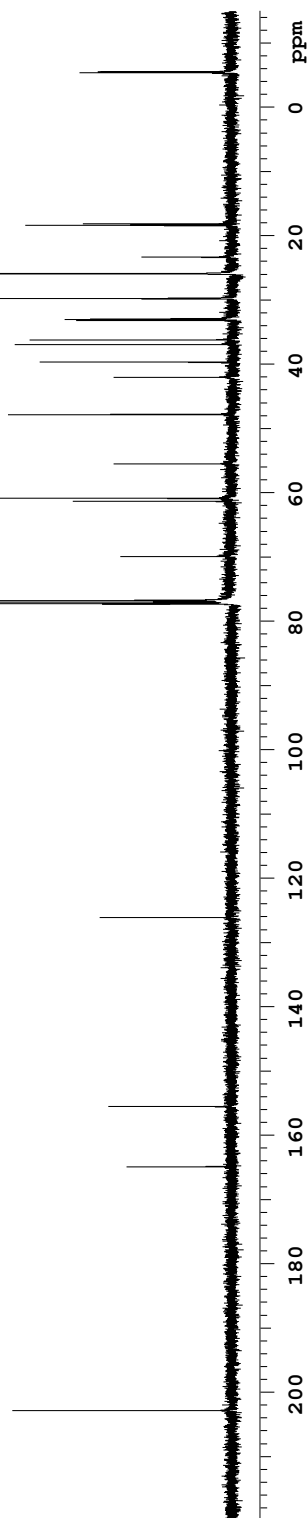
Sample Name:
jc-1-256-A
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vnmrsys/data
Sample directory:
jc-1-256-A
FidFile: PROTON01

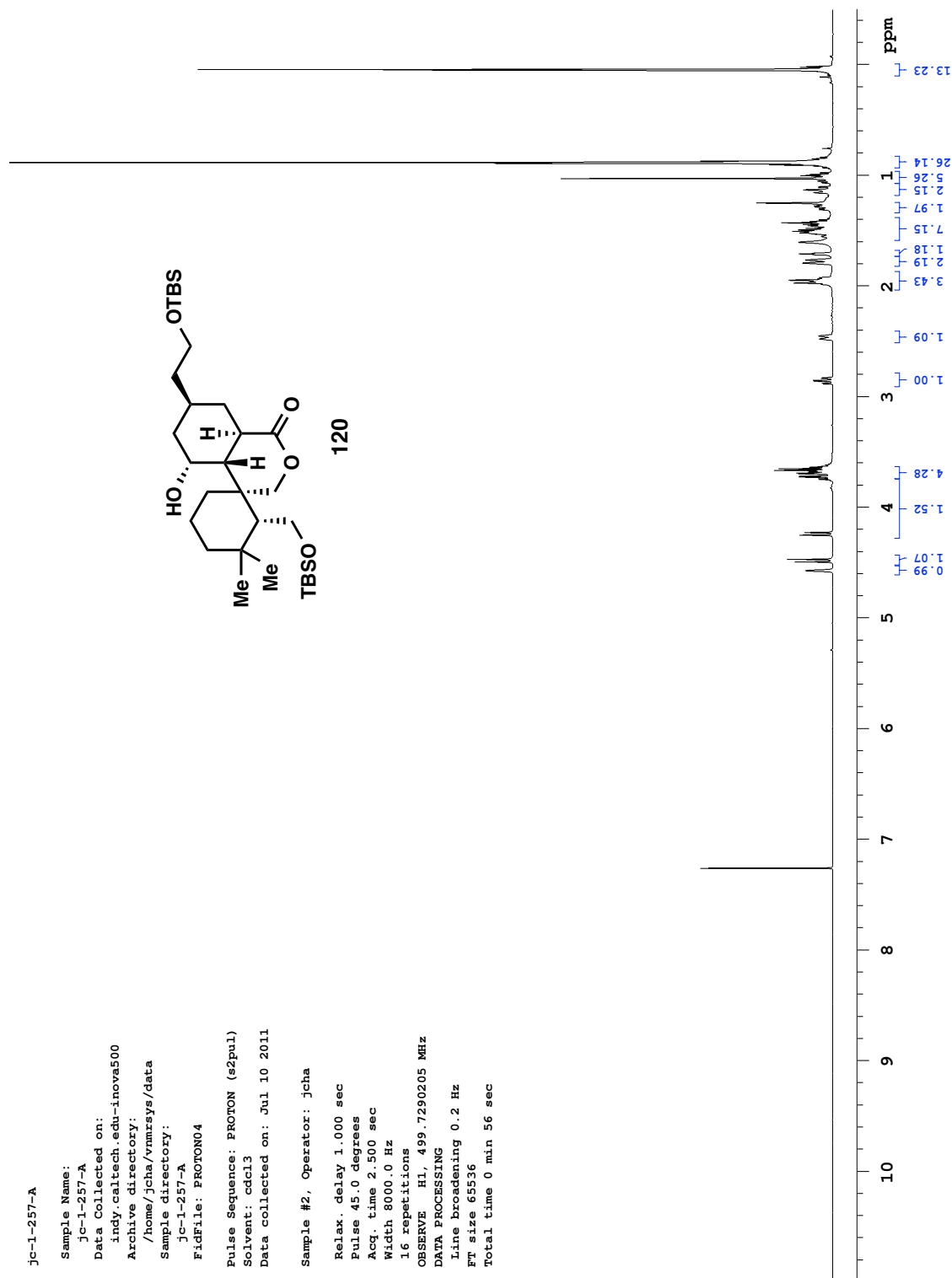
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 19 2011

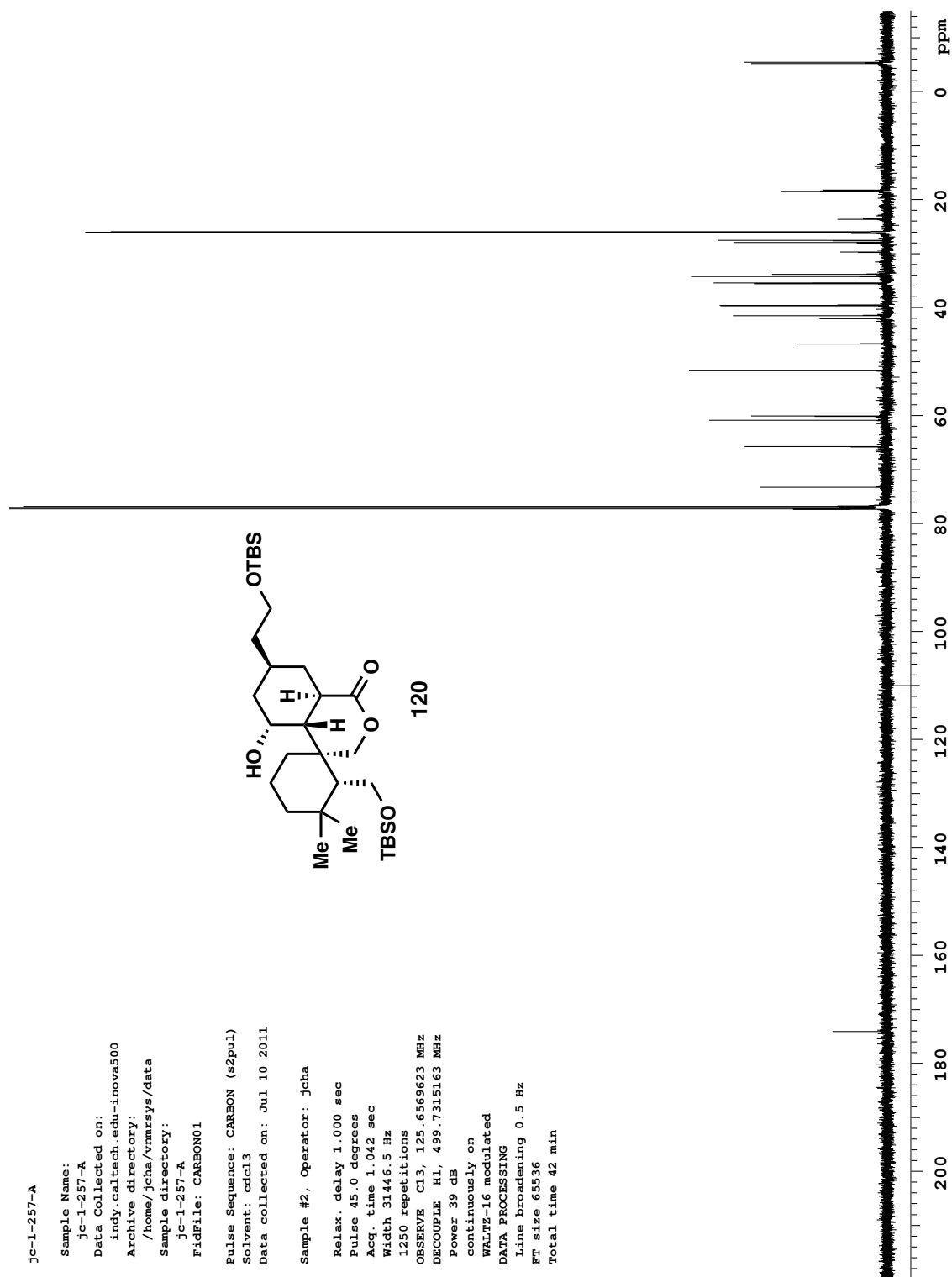
Temp. 25.0 C / 298.1 K
Sample #46, Operator: Jcha

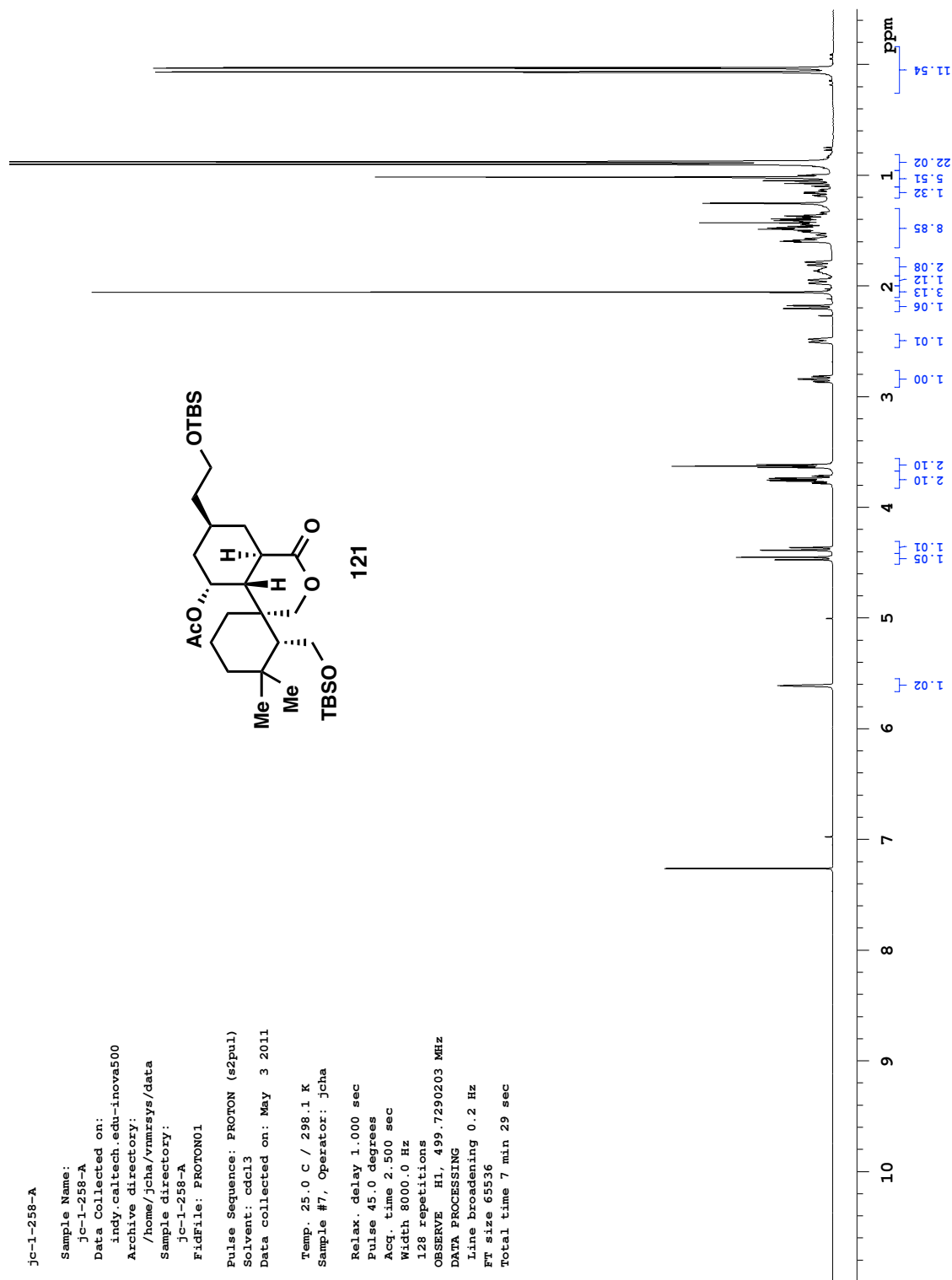
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290208 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 3 min 45 sec

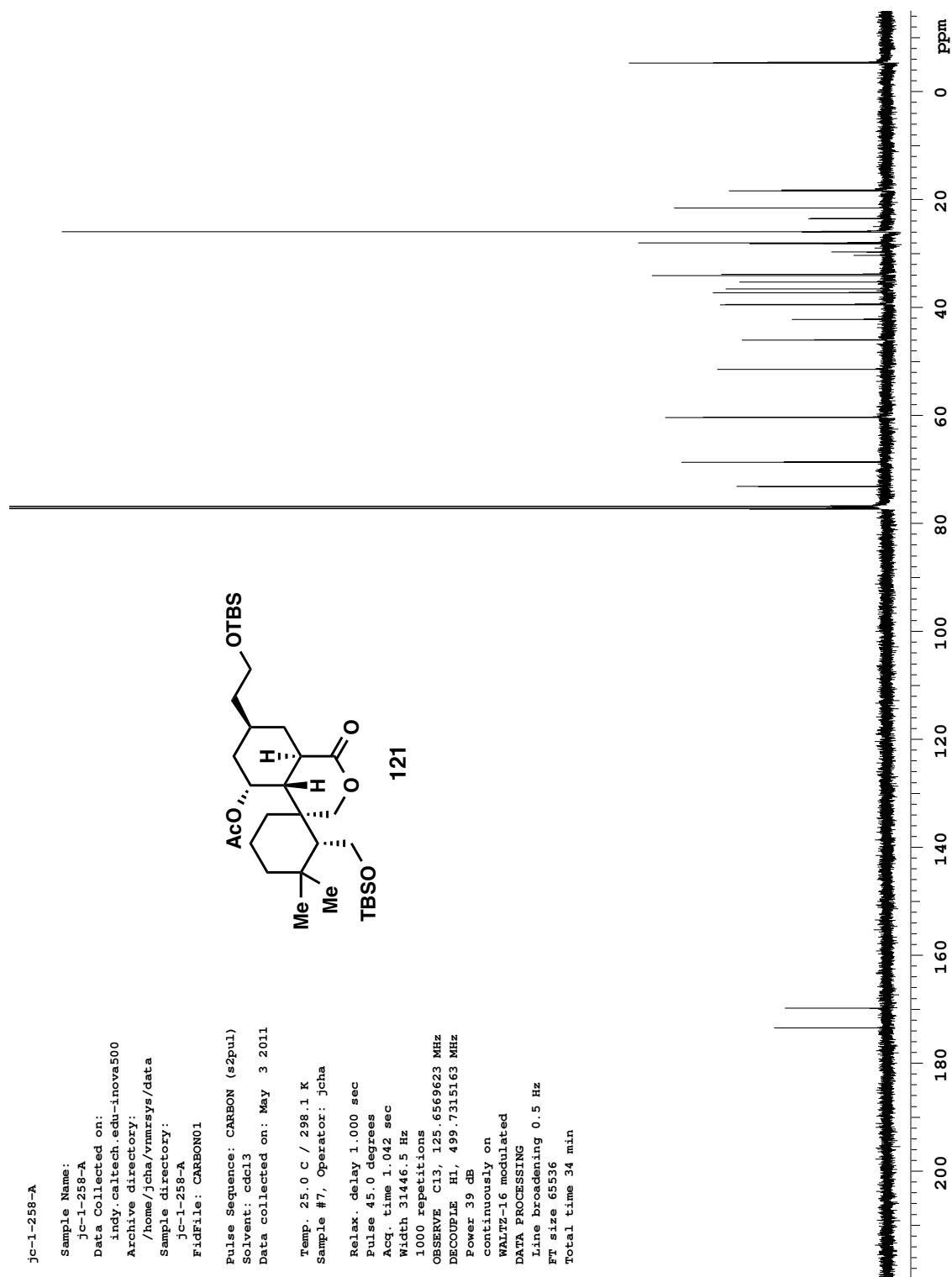


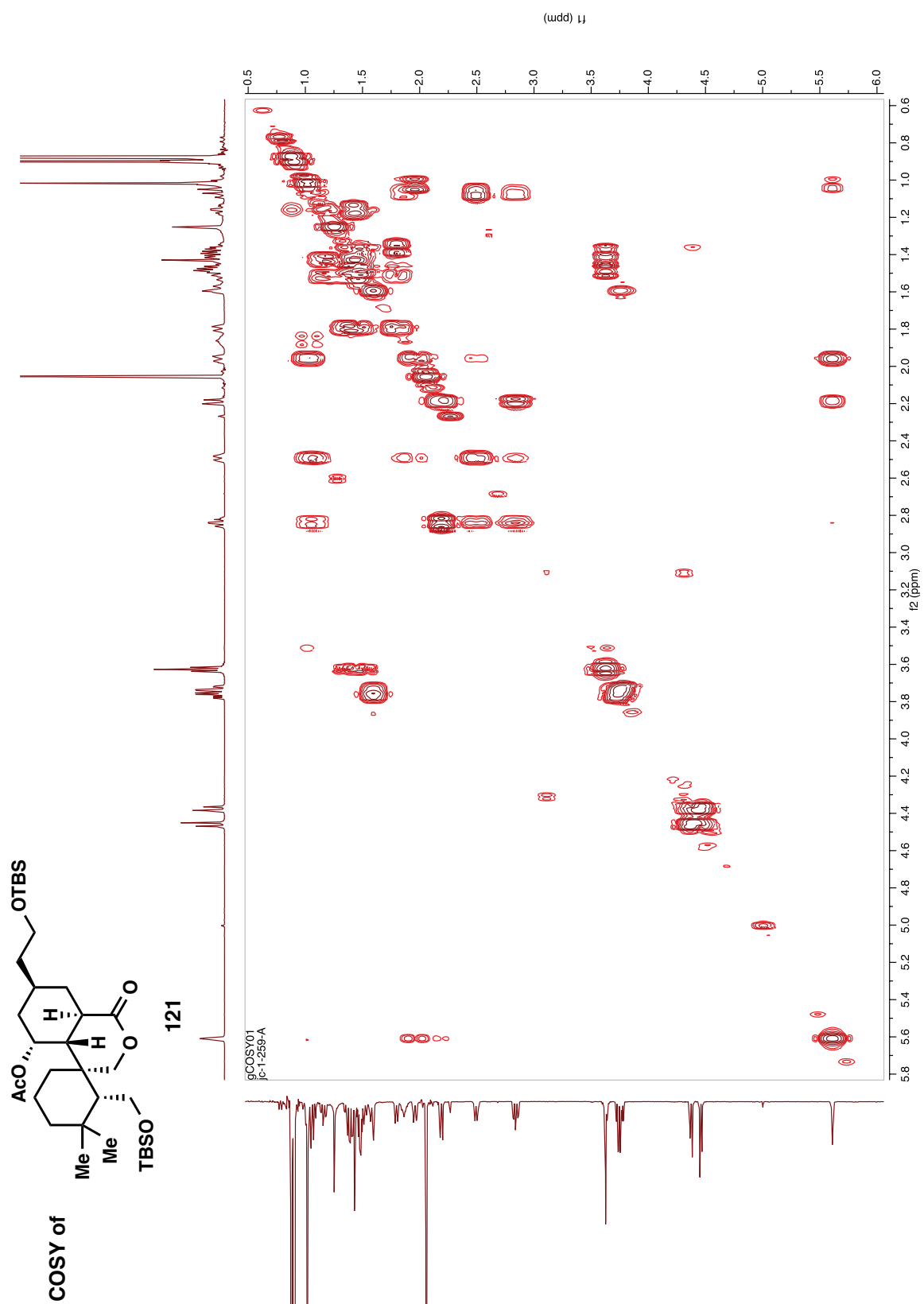


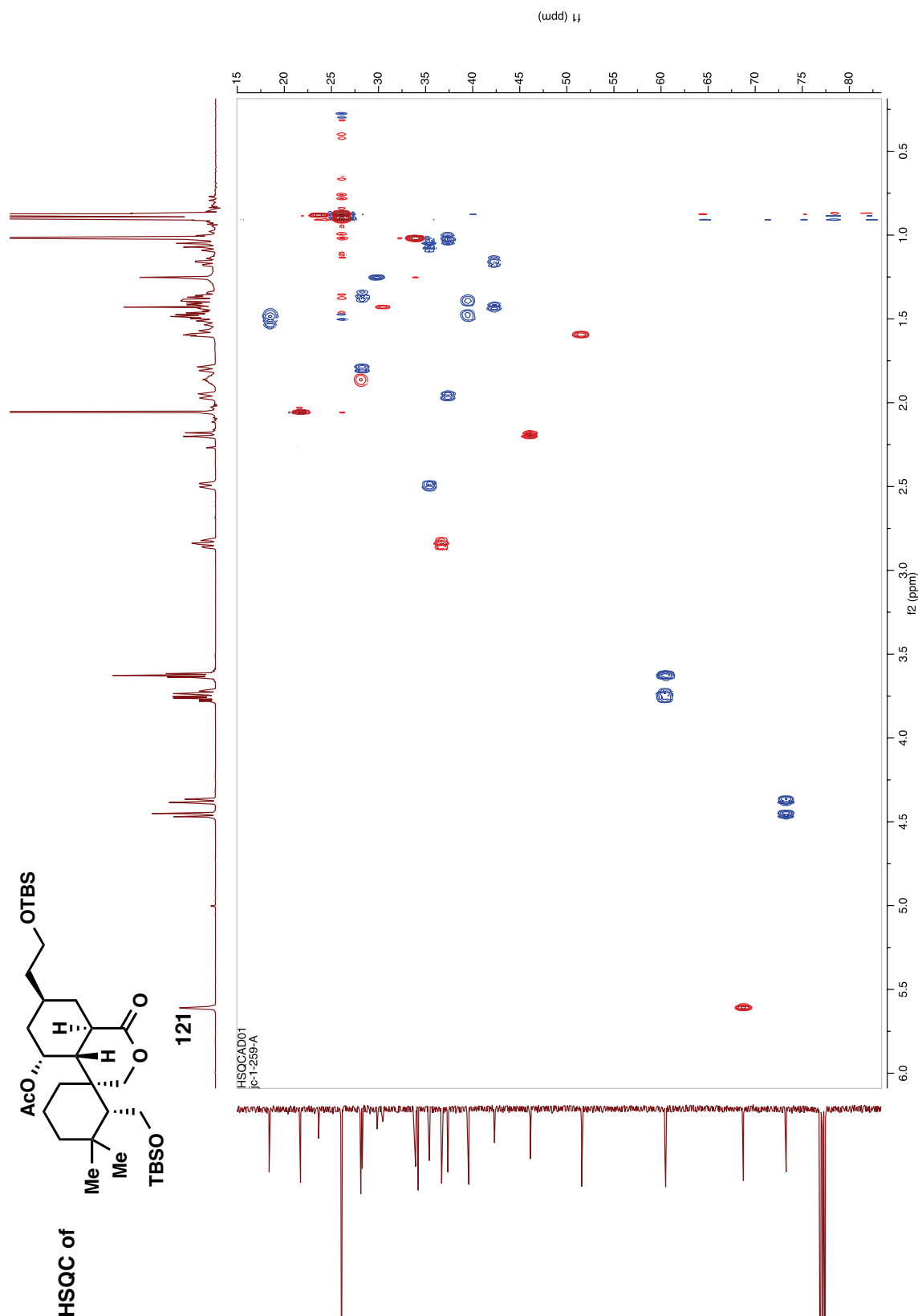


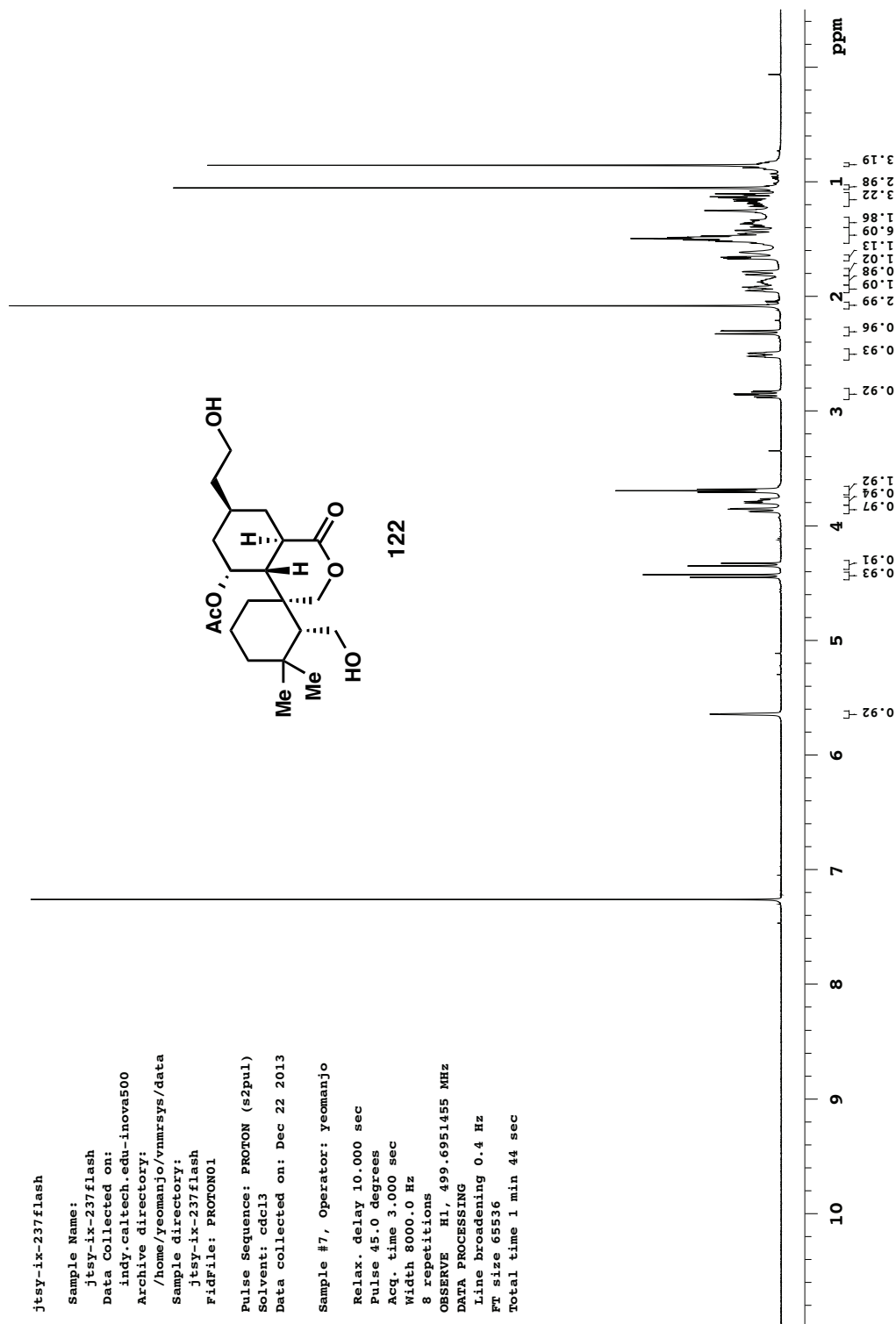


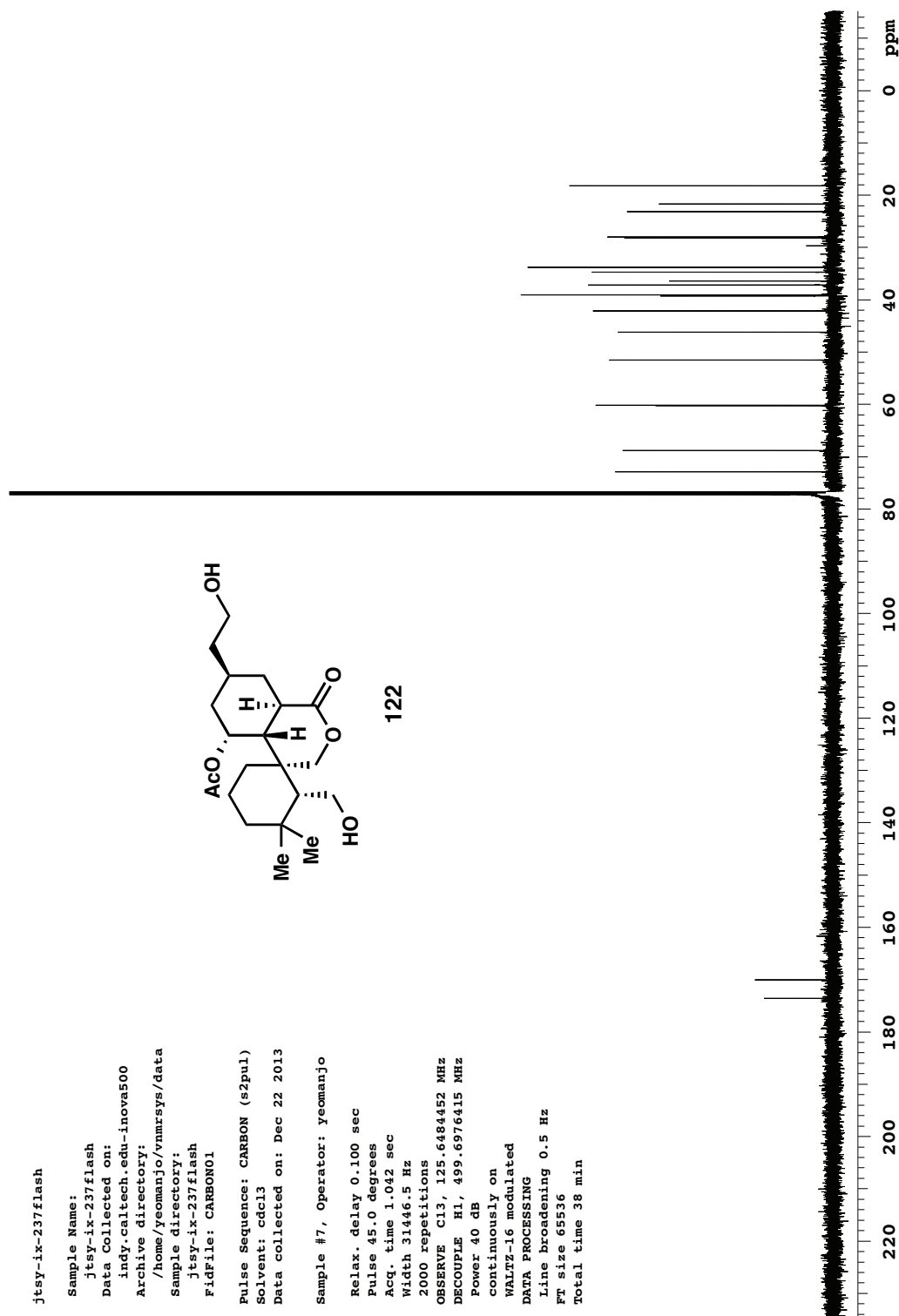


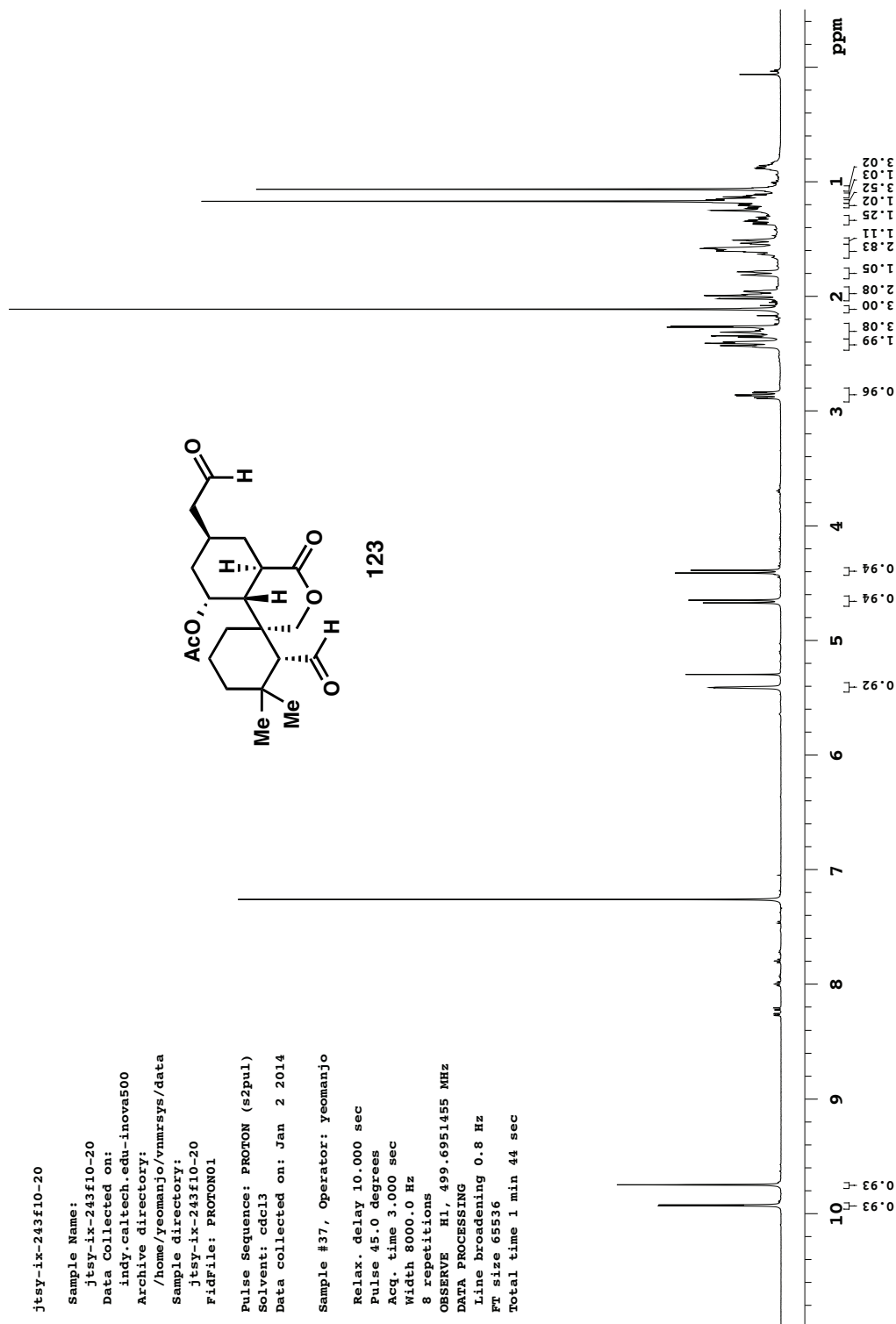












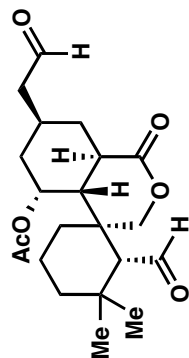
jtsy-ix-243f10-20

Sample Name:
jtsy-ix-243f10-20
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/yeomanjo/vnmrsys/data
Sample directory:
jtsy-ix-243f10-20
Fidfile: CARBON01

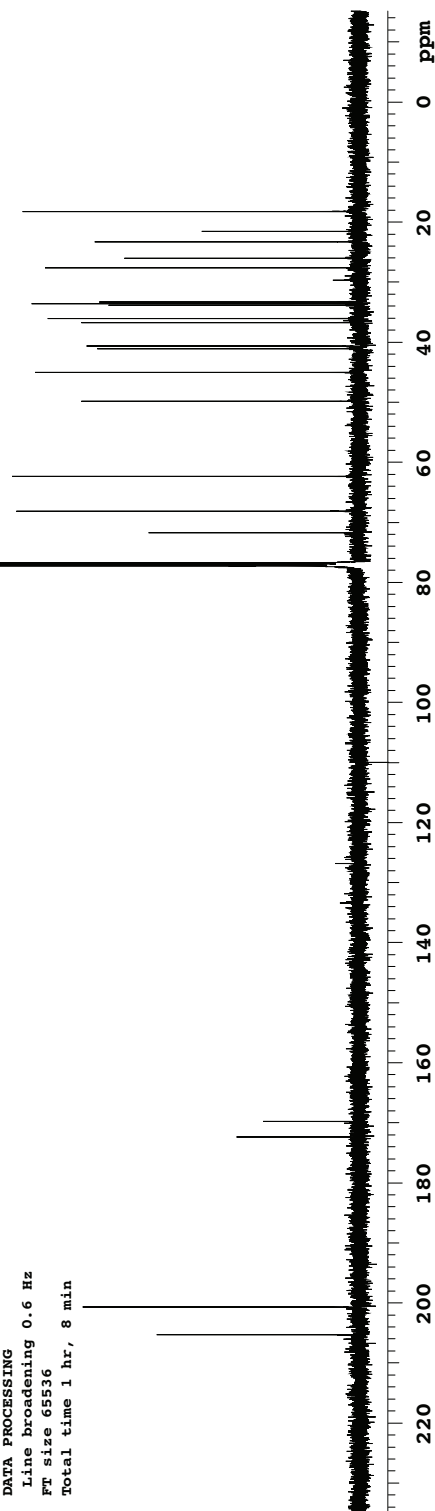
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 2 2014

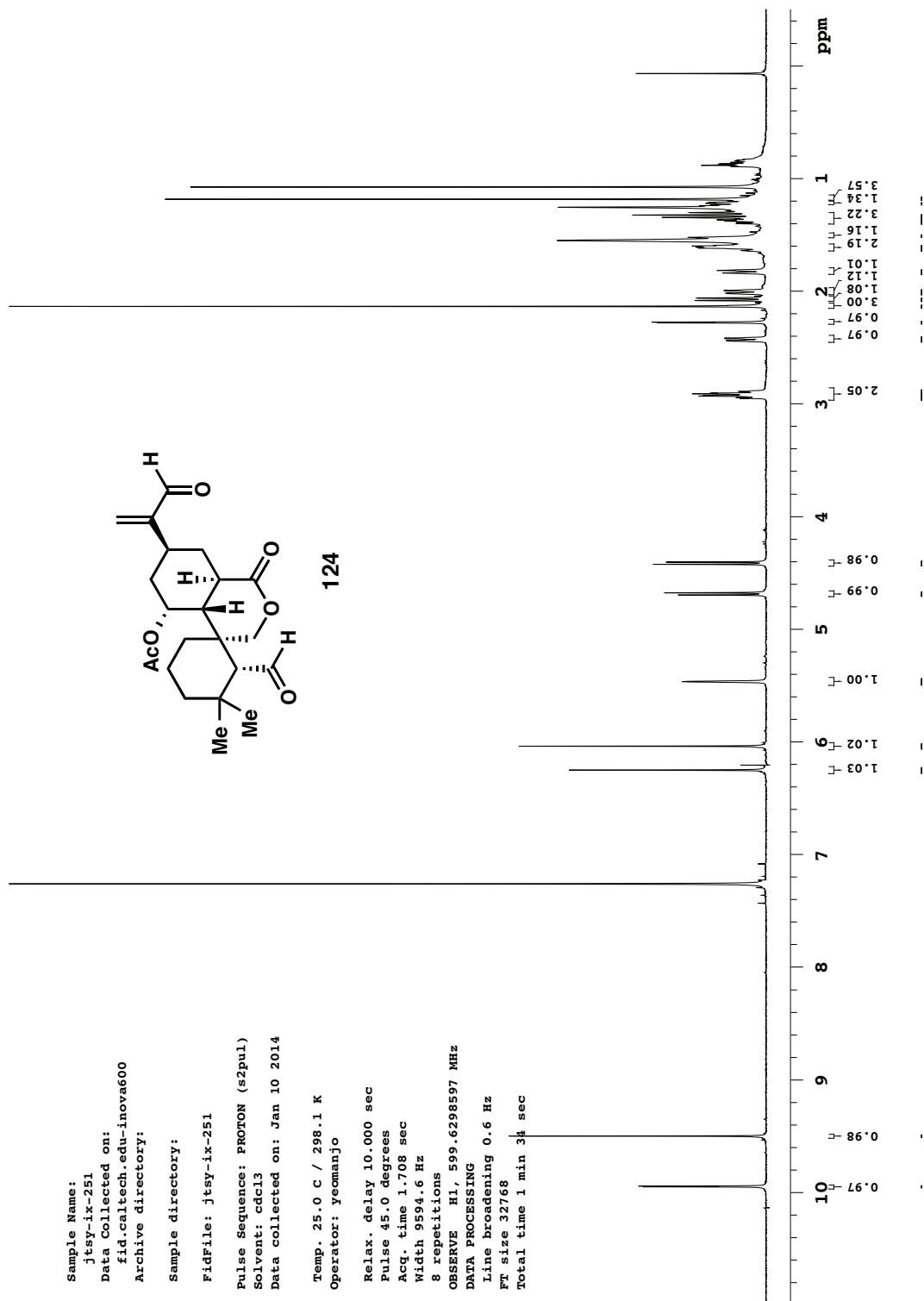
Sample #37, Operator: yeomanjo

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
2000 repetitions
OBSERVE C13, 125.6484452 MHz
DECOUPLE H1, 499.6976415 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.6 Hz
FT size 65536
Total time 1 hr, 8 min



123





jtsy-ix-251f25-33

Sample Name:
jtsy-ix-251f25-33
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/yeomanjo/vnmrsys/data
Sample directory:
jtsy-ix-251f25-33
Fidfile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014

Sample #1, Operator: yeomanjo

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3100 repetitions

OBSERVE C13, 125.6484443 MHz
DECOUPLE H1, 499.6976415 MHz
Power 40 dB
continuously on

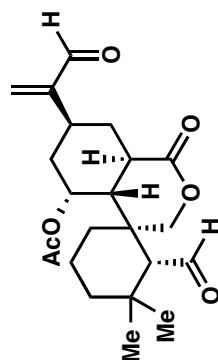
WALTZ-16 modulated

DATA PROCESSING

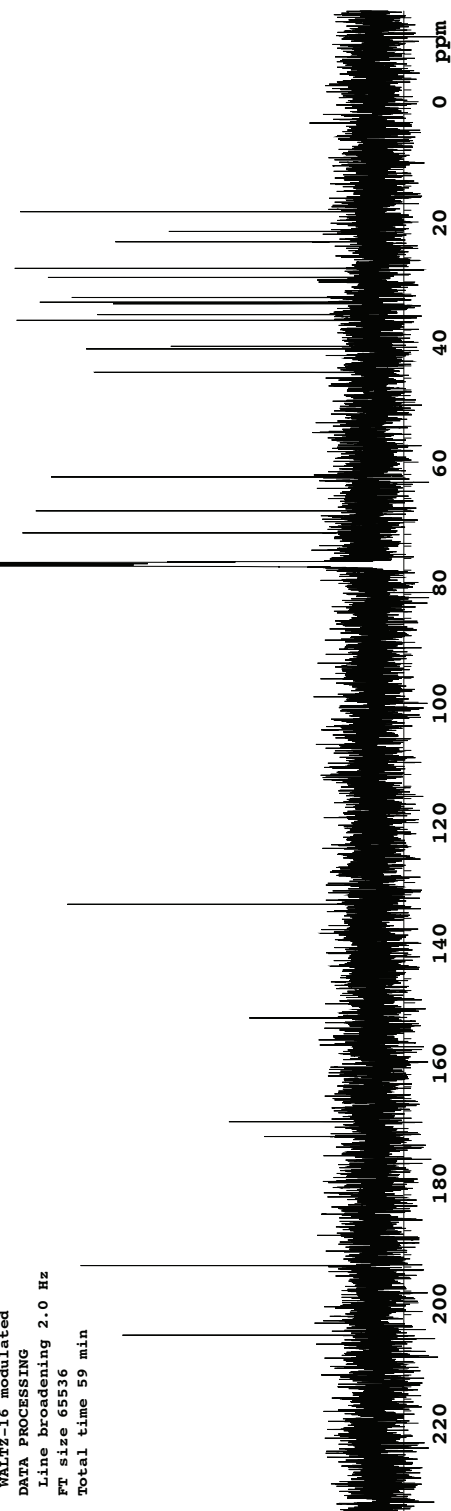
Line broadening 2.0 Hz

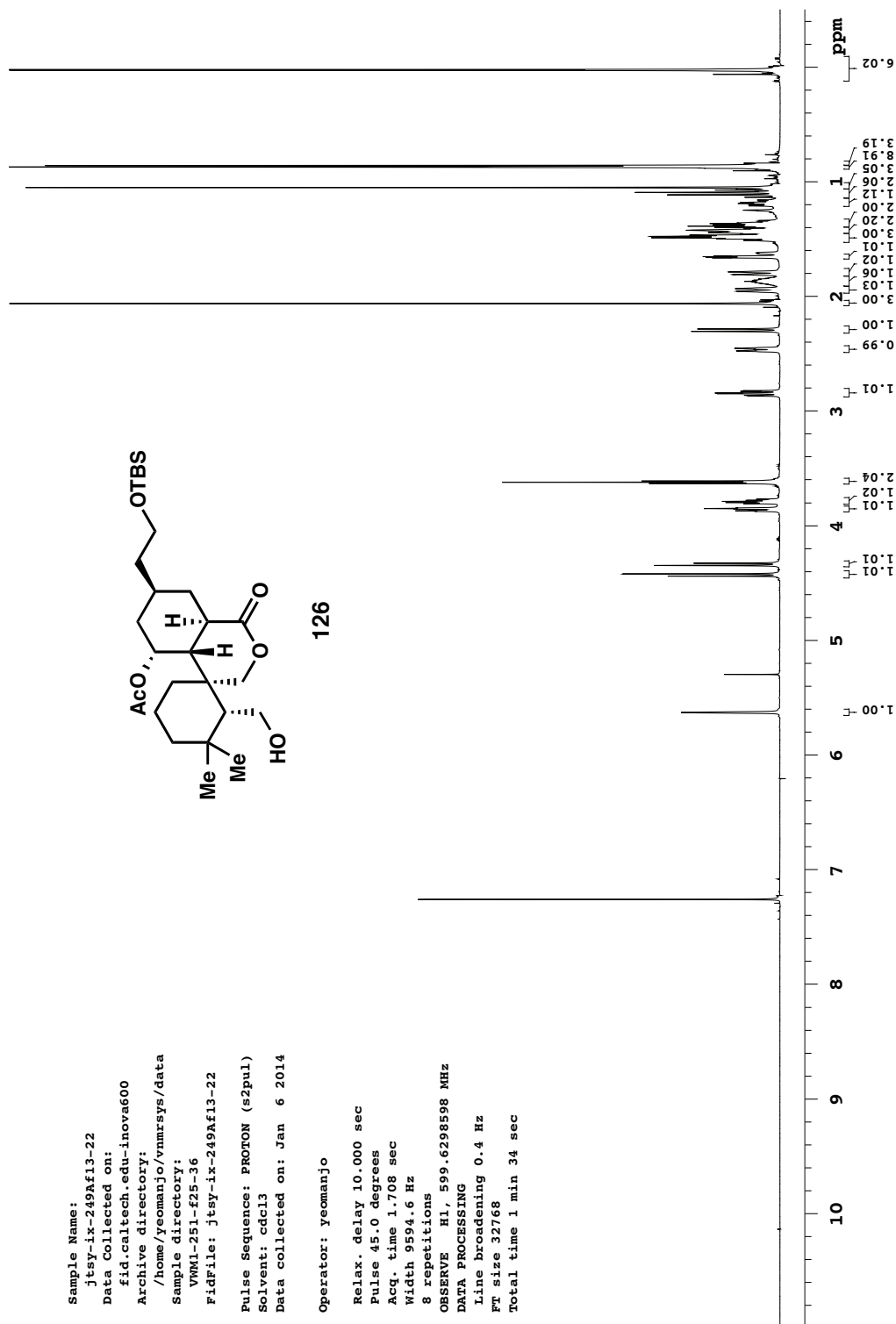
FT size 65536

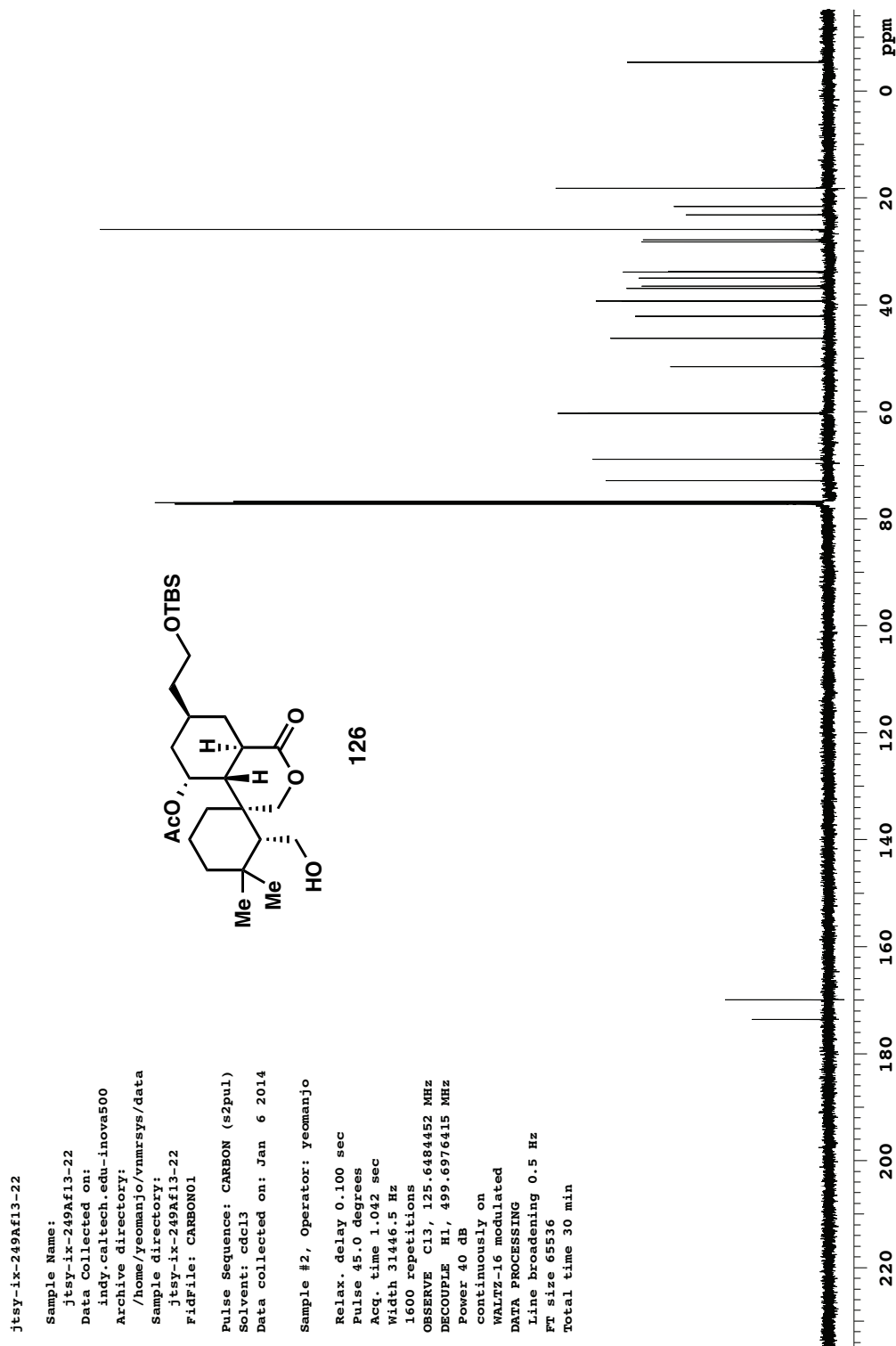
Total time 59 min

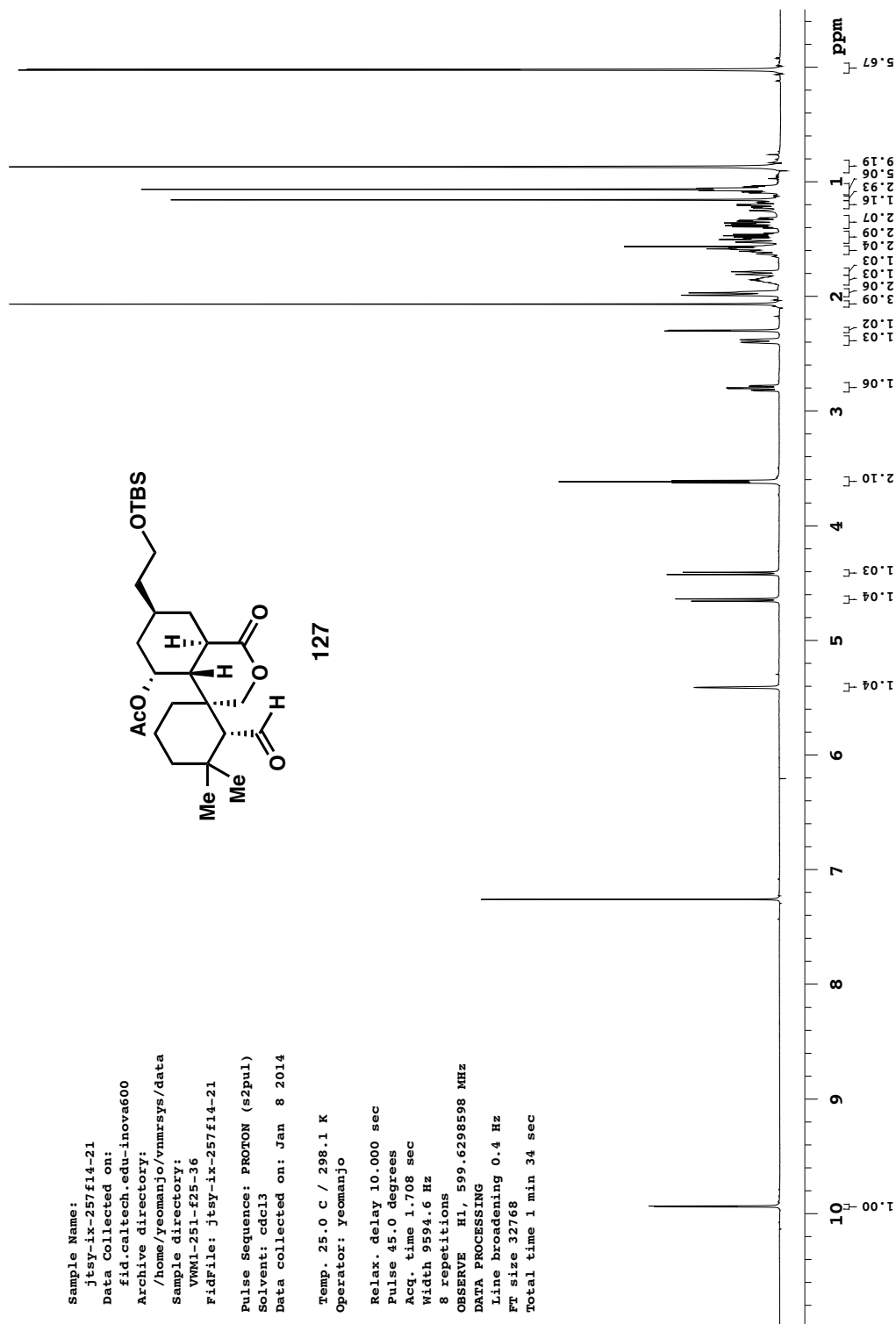


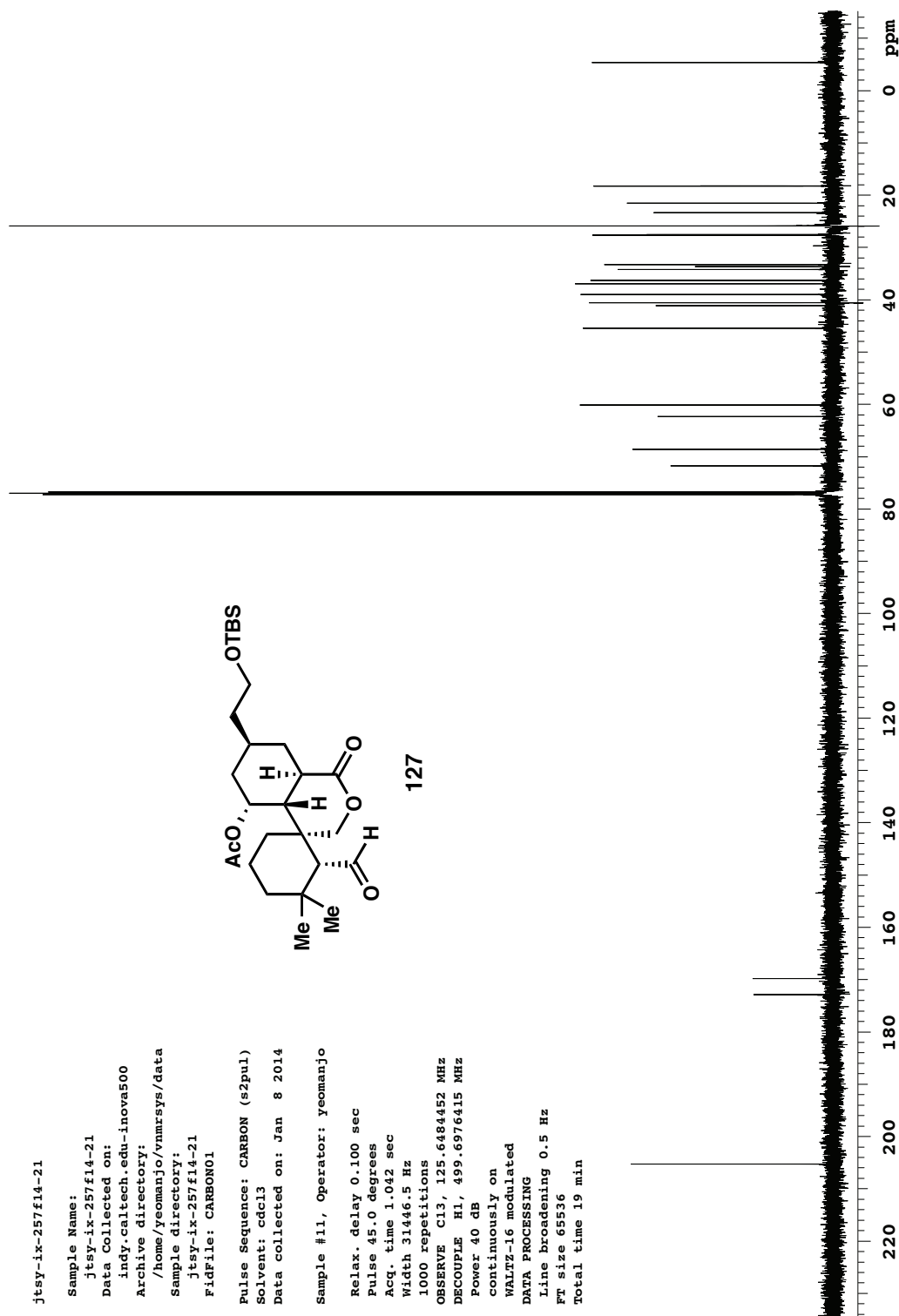
124

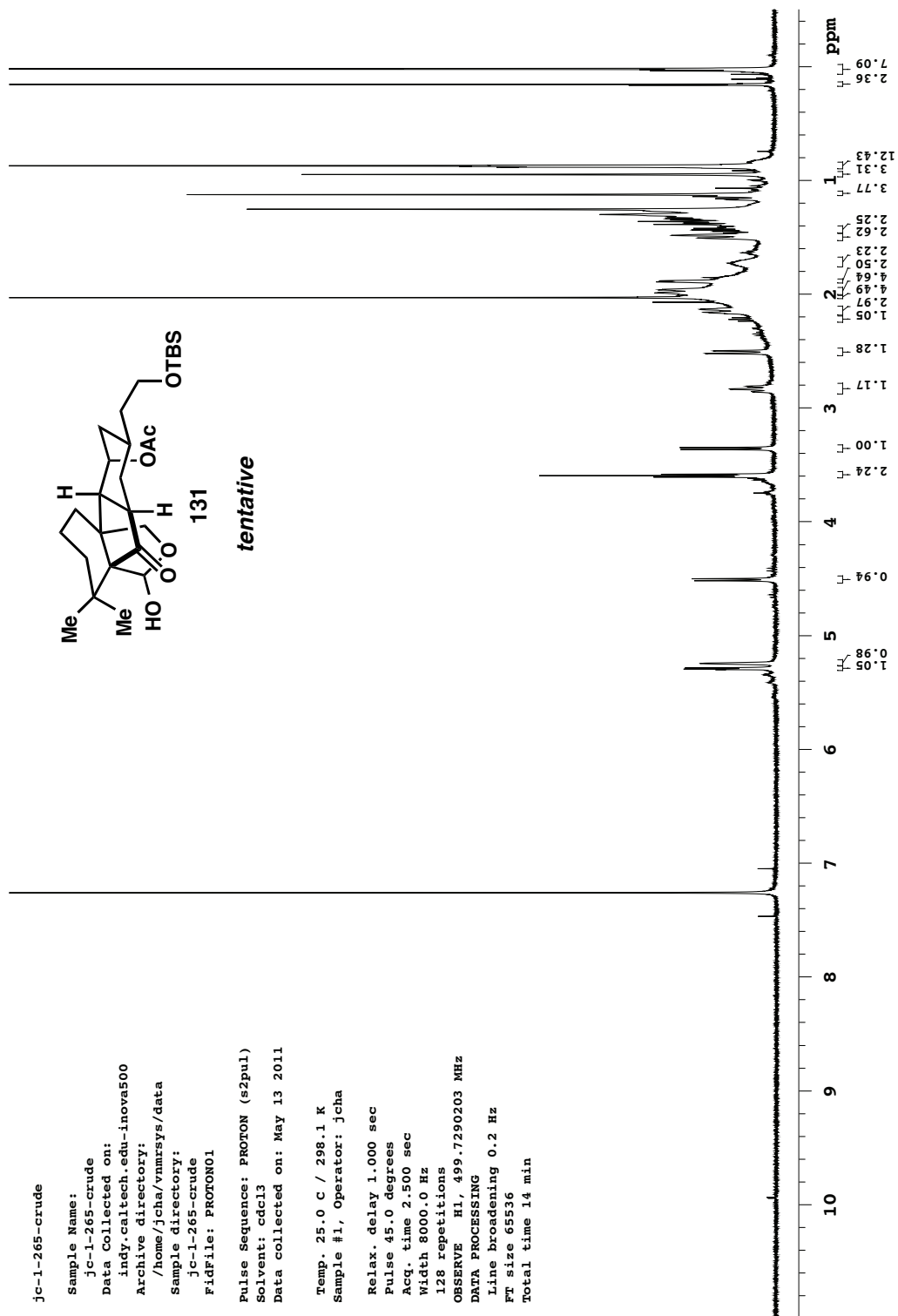


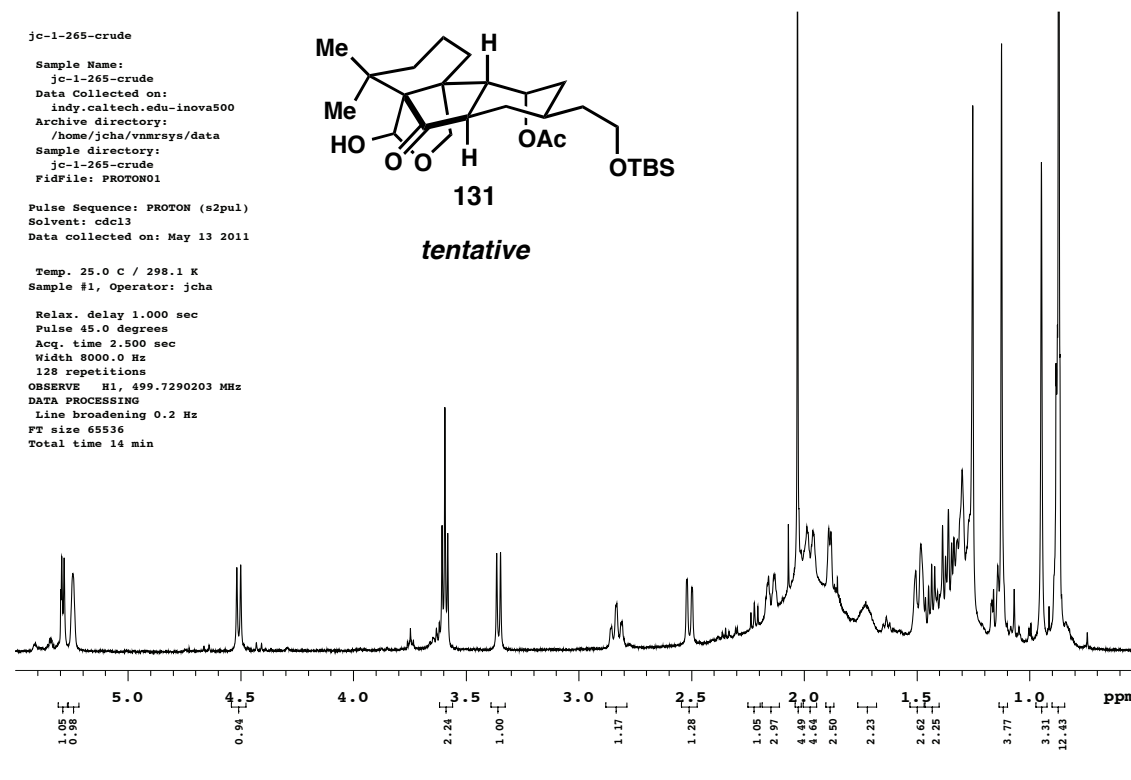
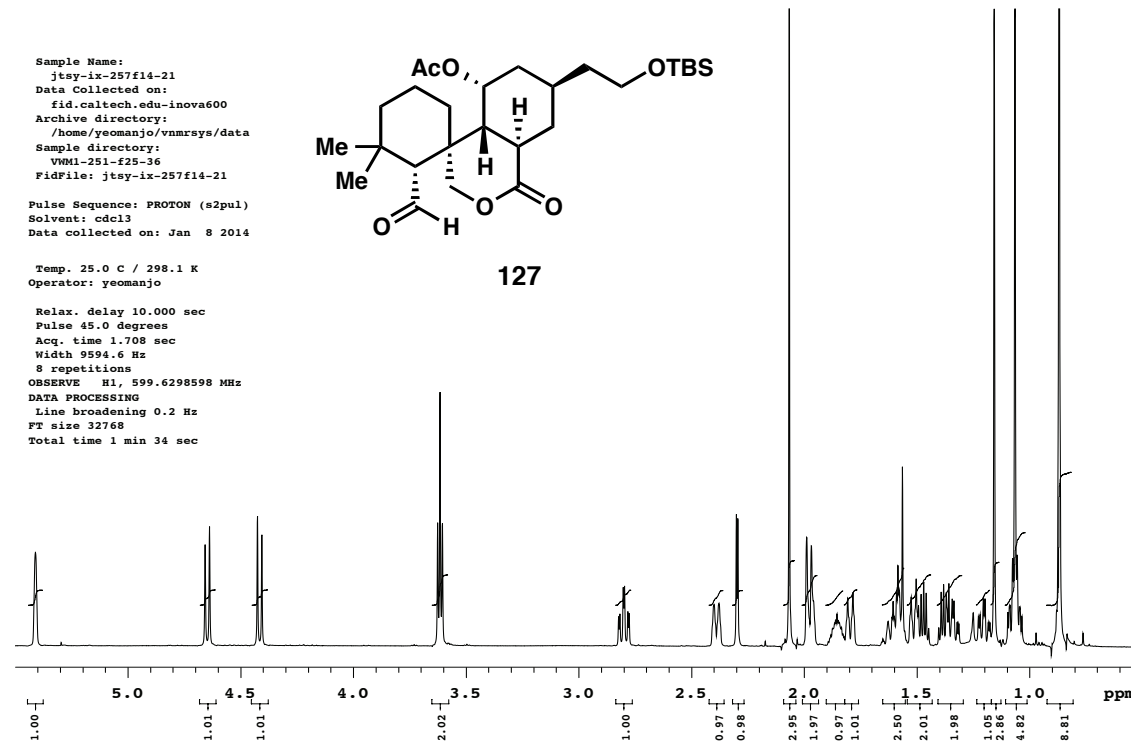








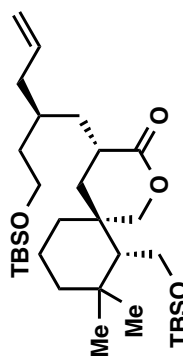




```

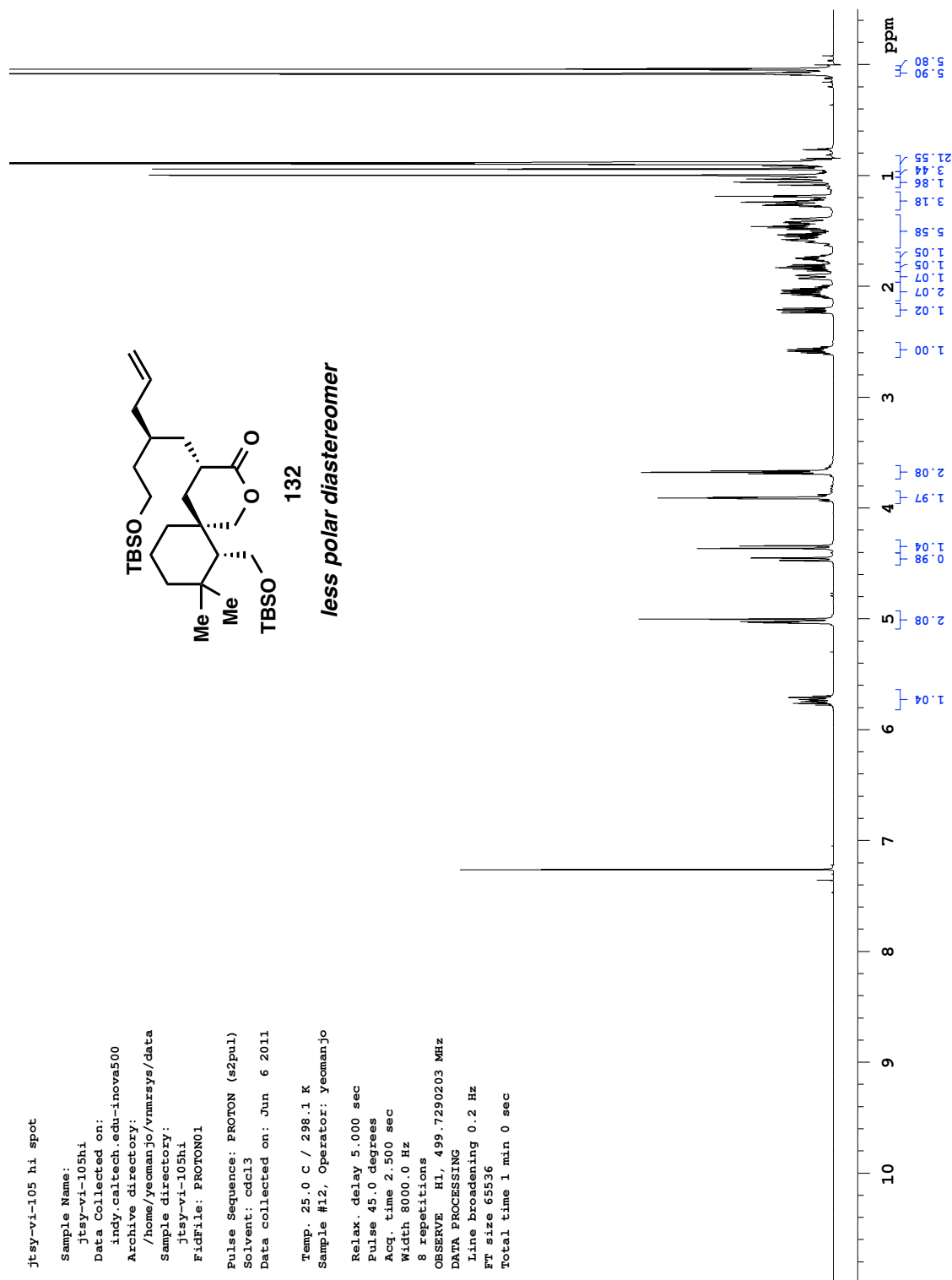
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7290200
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 0 sec

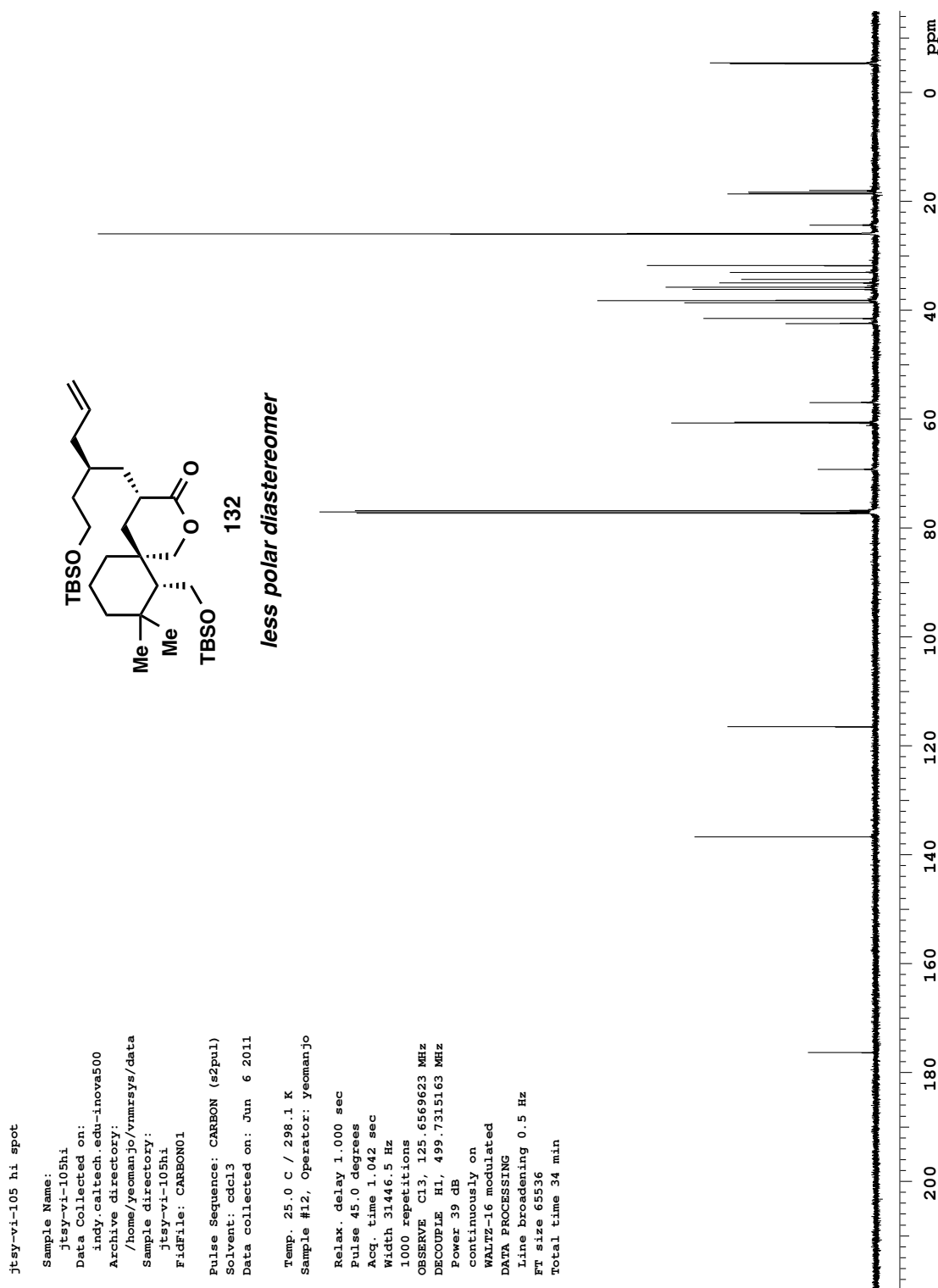
```



132

less polar diastereomer





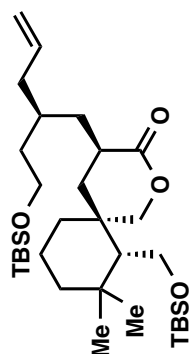
jtay-vi-129 minor fms 46-47

Sample Name:
jtay-vi-129min46-47
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/yeomanjo/vnmrSYS/data
Sample directory:
jtay-vi-129min46-47
FidFile: PROTON01

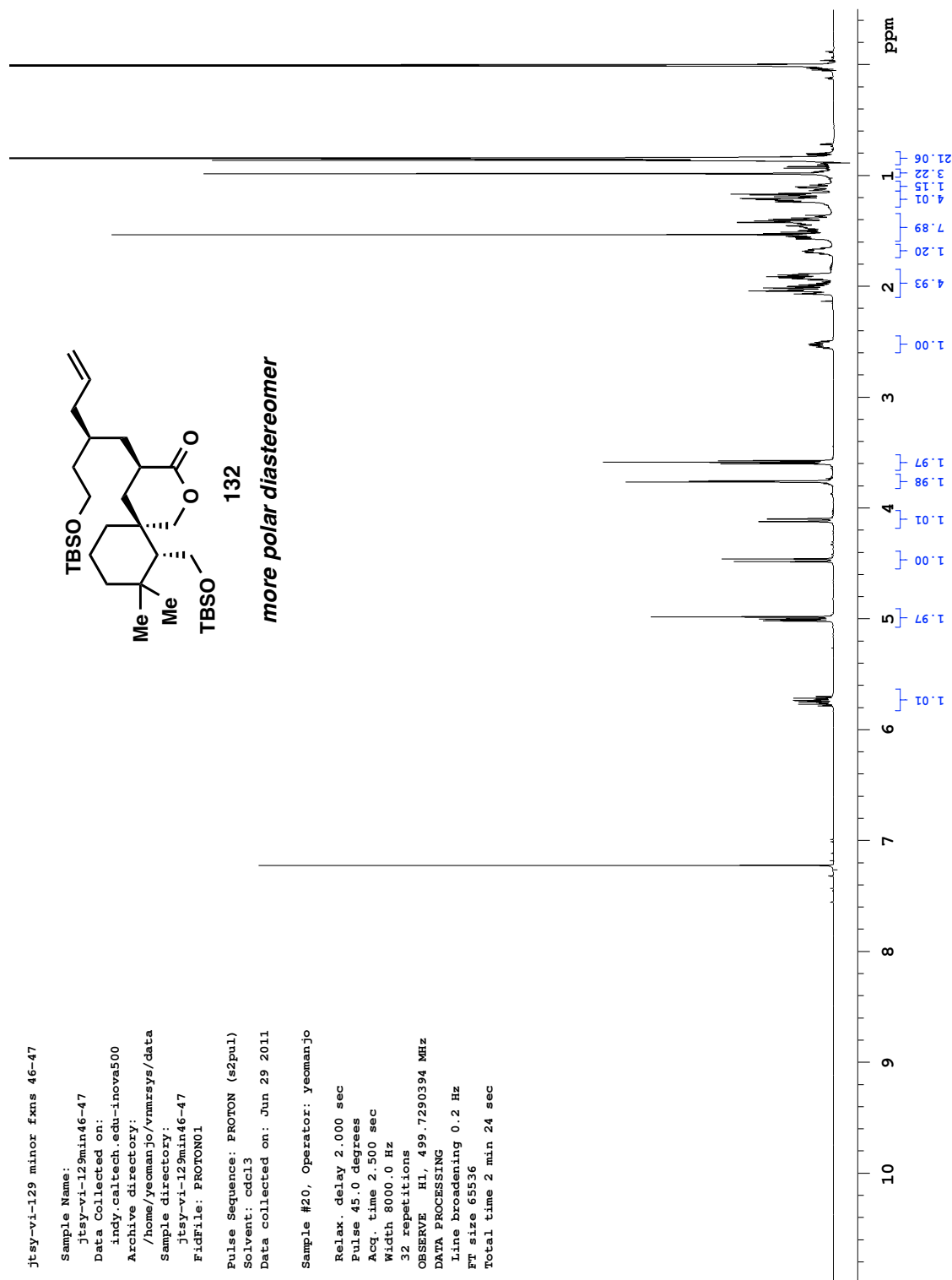
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 29 2011

Sample #20, Operator: yeomanjo

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7290394 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 24 sec



132

more polar diastereomer

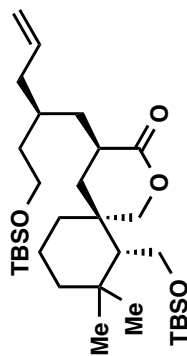
jtsey-vi-129 minor fxns 46-47

Sample Name:
jtsey-vi-129min46-47
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/yeomanjo/vmrays/data
Sample directory:
jtsey-vi-129min46-47
FidFile: CARBON01

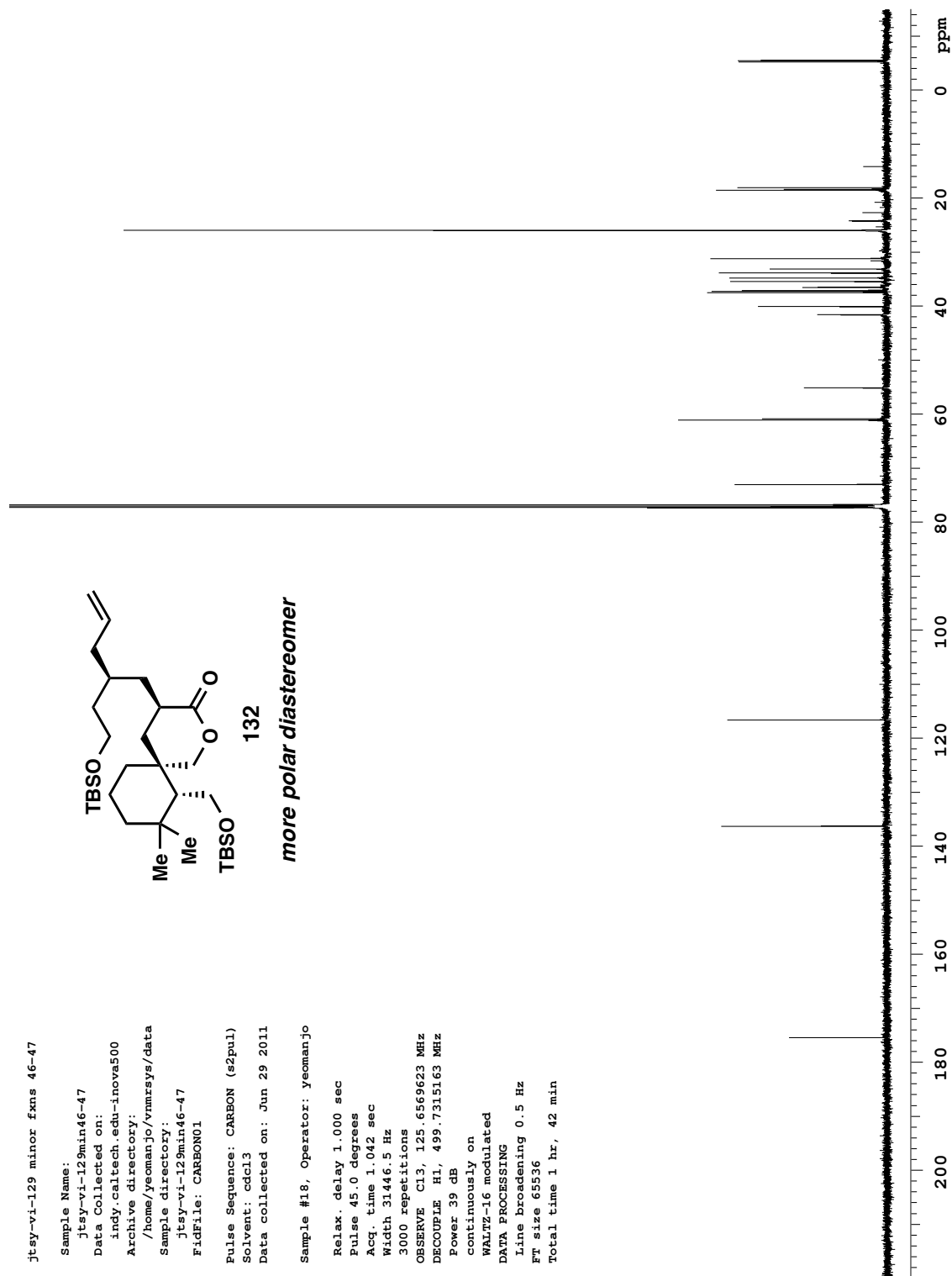
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 29 2011

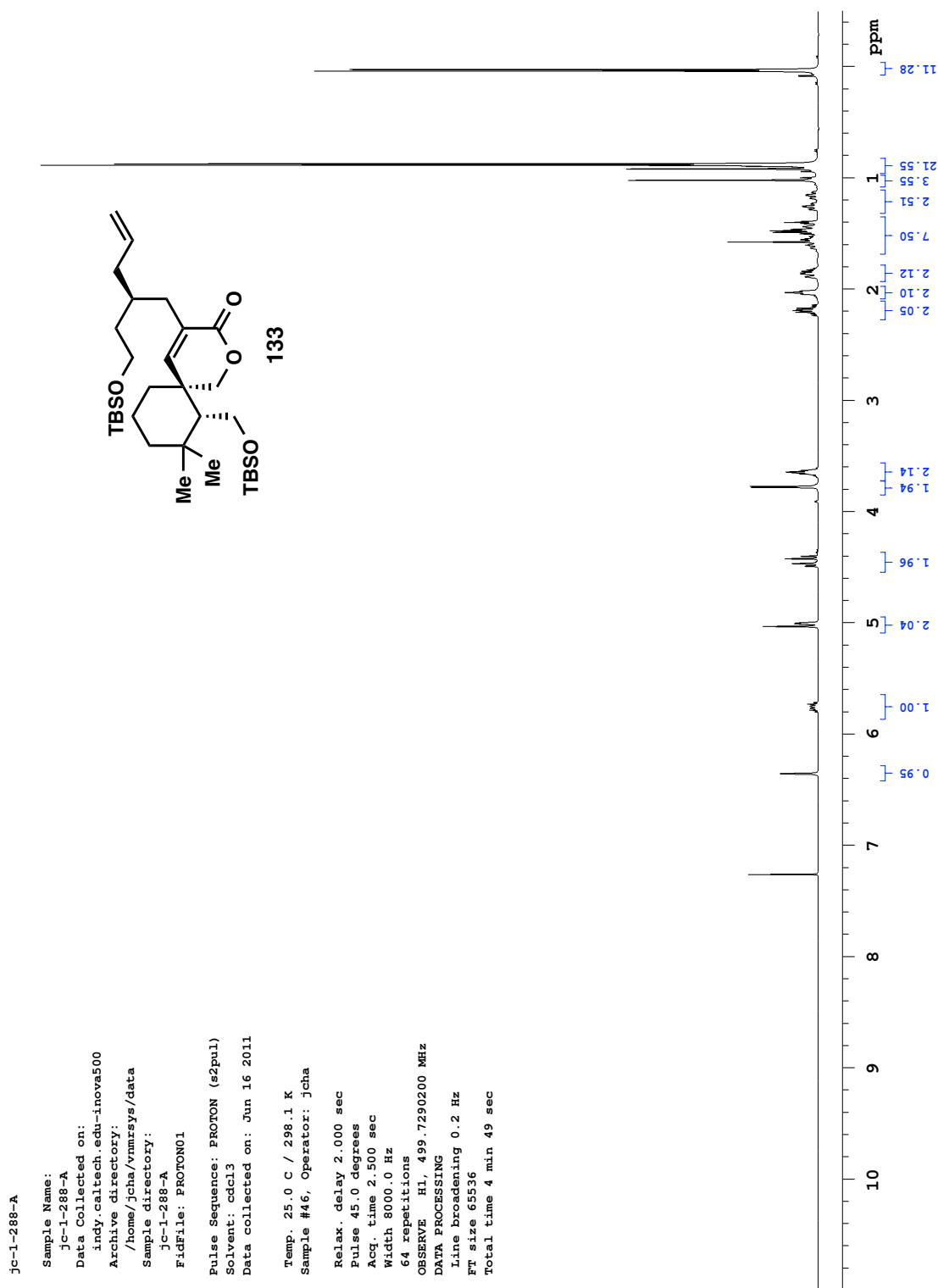
Sample #18, Operator: yeomanjo

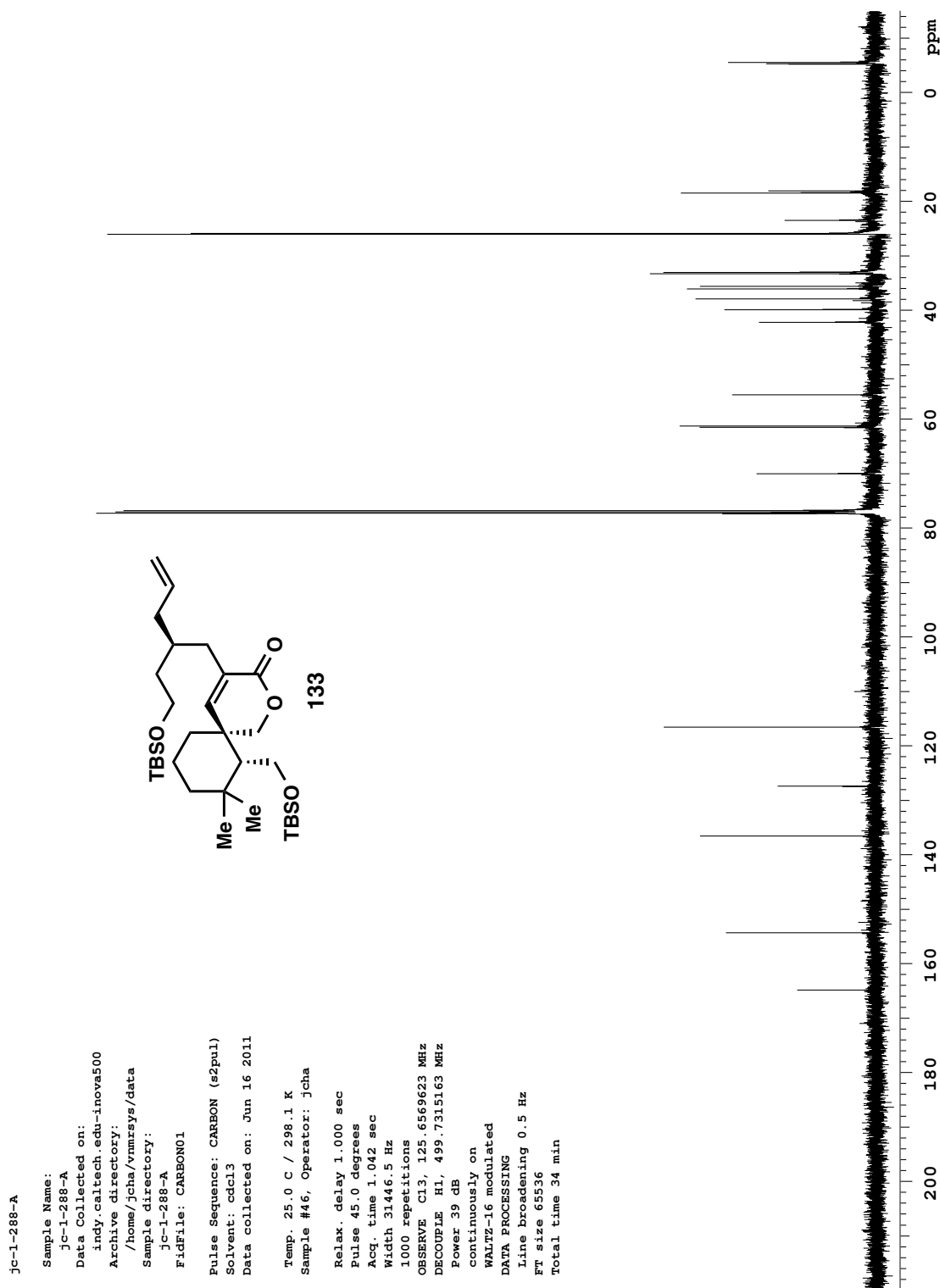
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
3000 repetitions
OBSERVE C13, 125.6569623 MHz
DECOUPLE H1, 499.7315163 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 42 min



132

more polar diastereomer





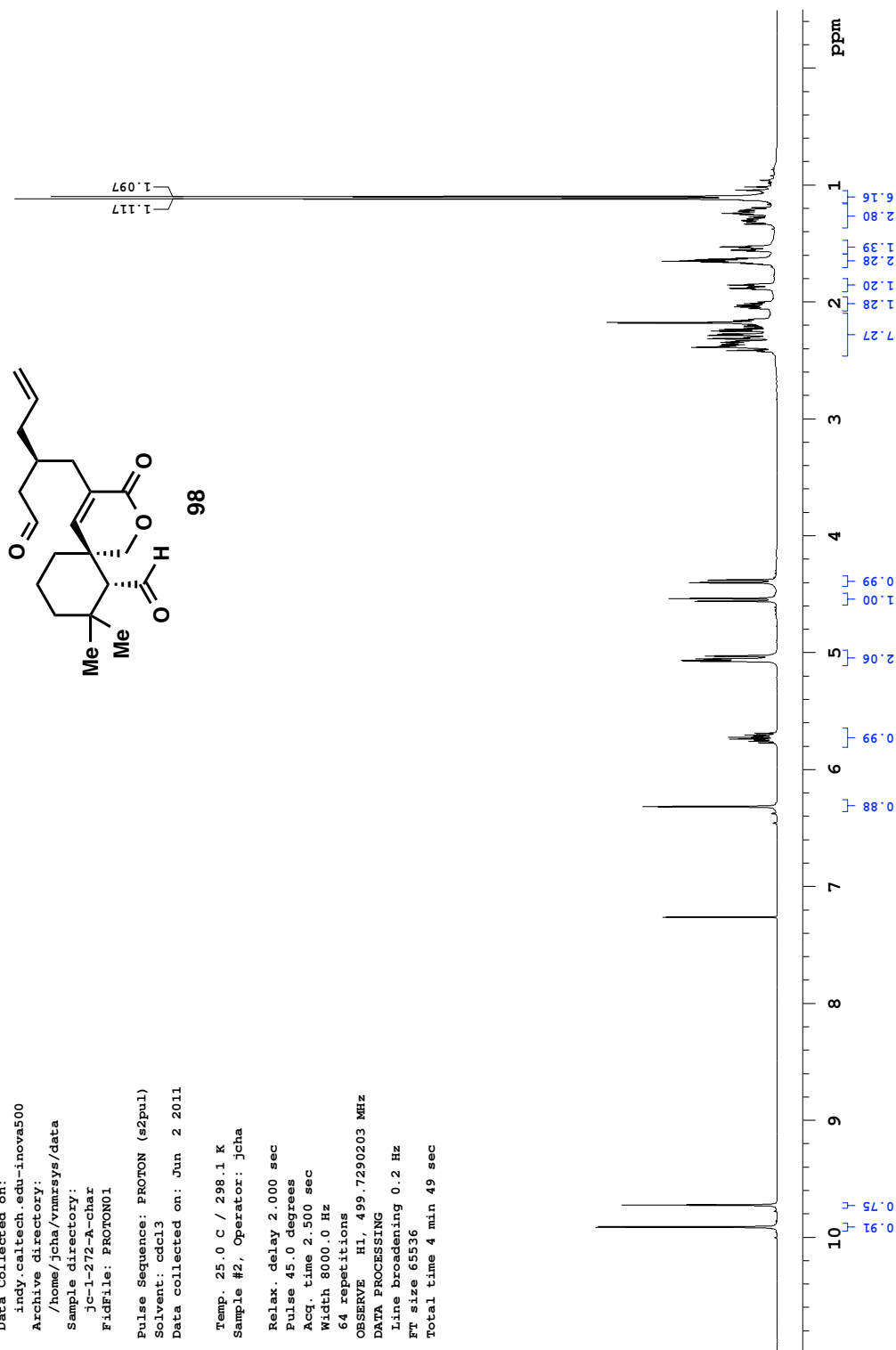
jc-1-272-A-char

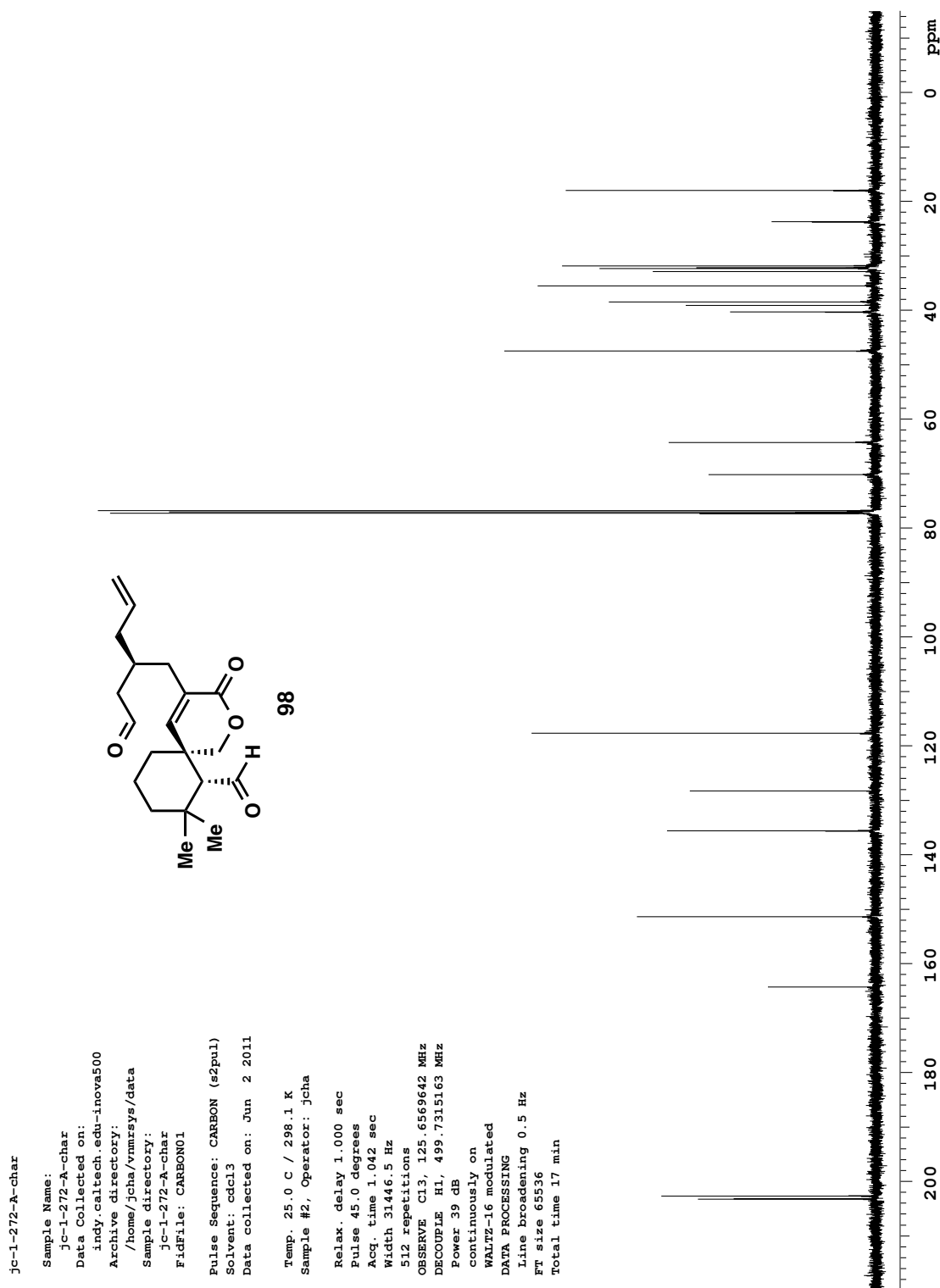
Sample Name:
jc-1-272-A-char
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/jcha/vmrsys/data
Sample directory:
jc-1-272-A-char
FidFile: PROTON01

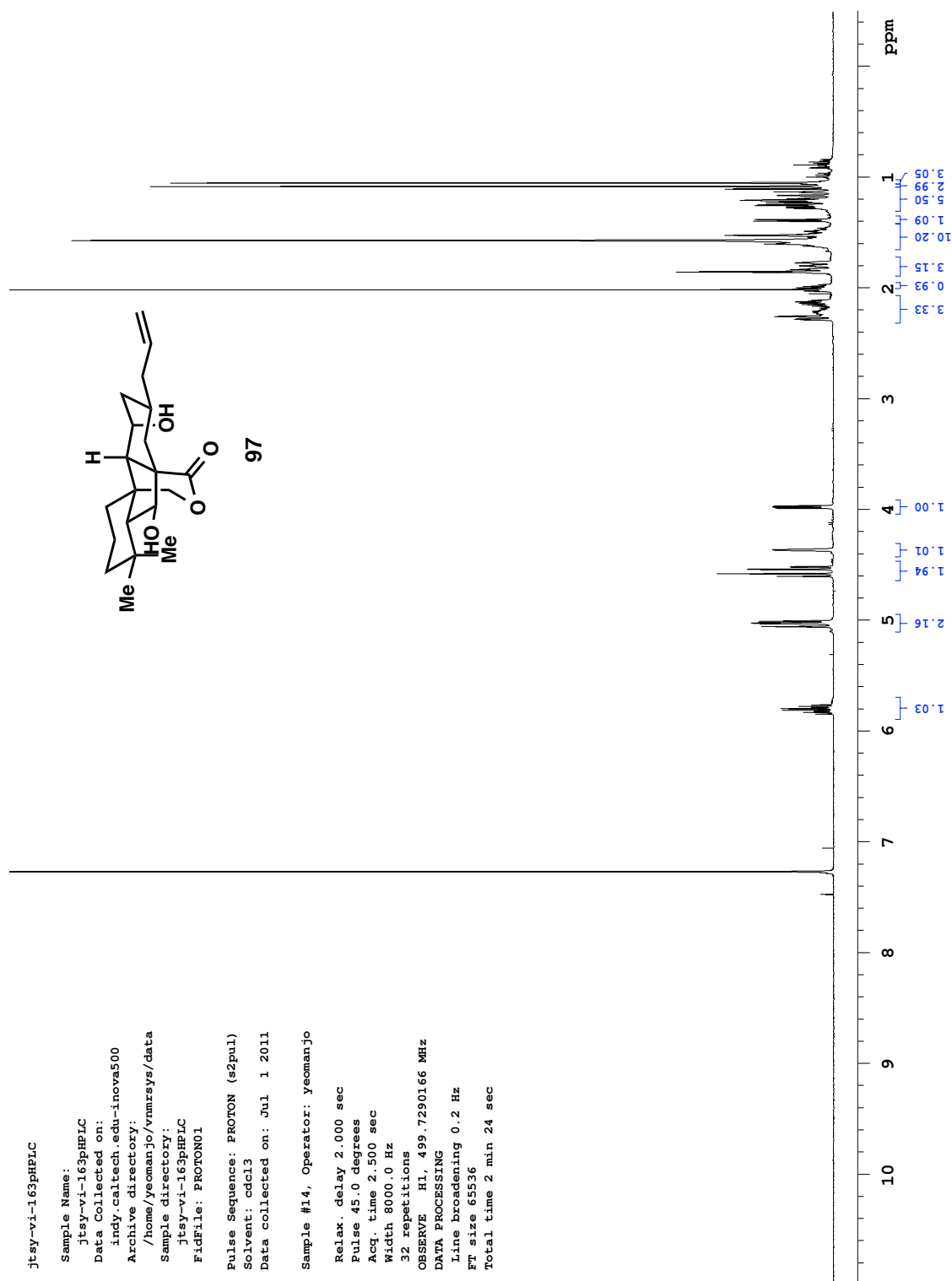
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 2 2011

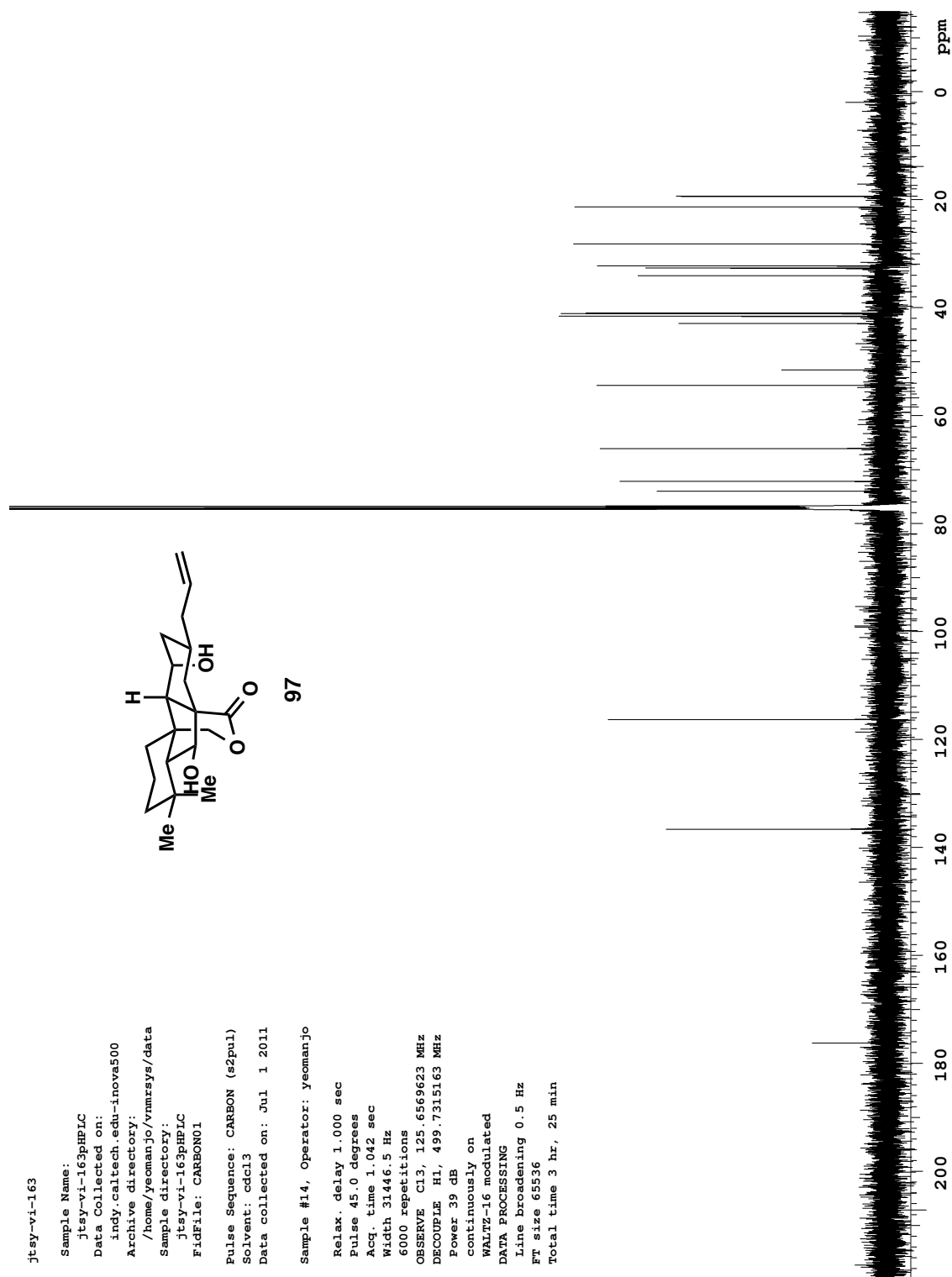
Temp. 25.0 C / 298.1 K
Sample #2, Operator: jcha

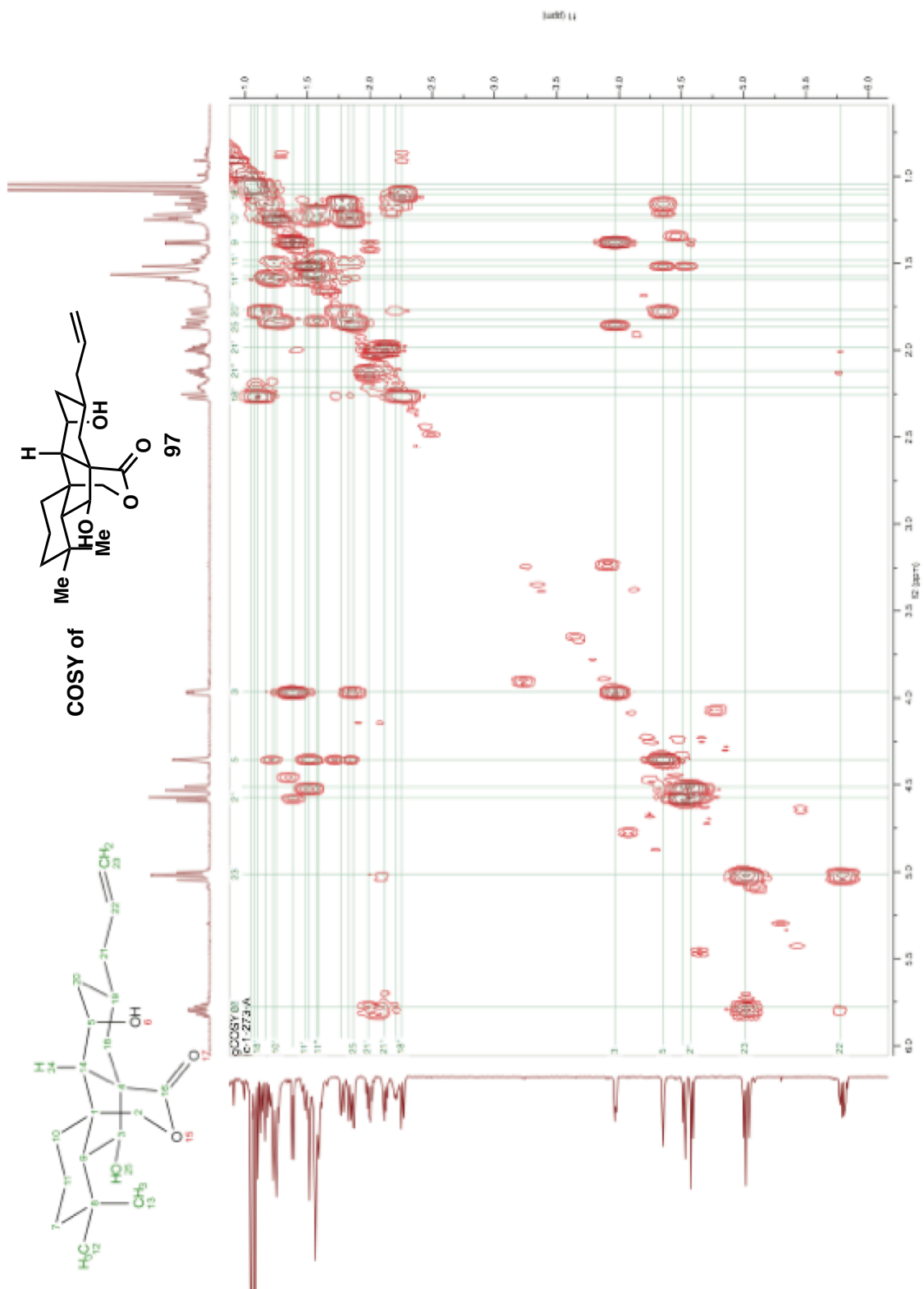
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7290203 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 4 min 49 sec

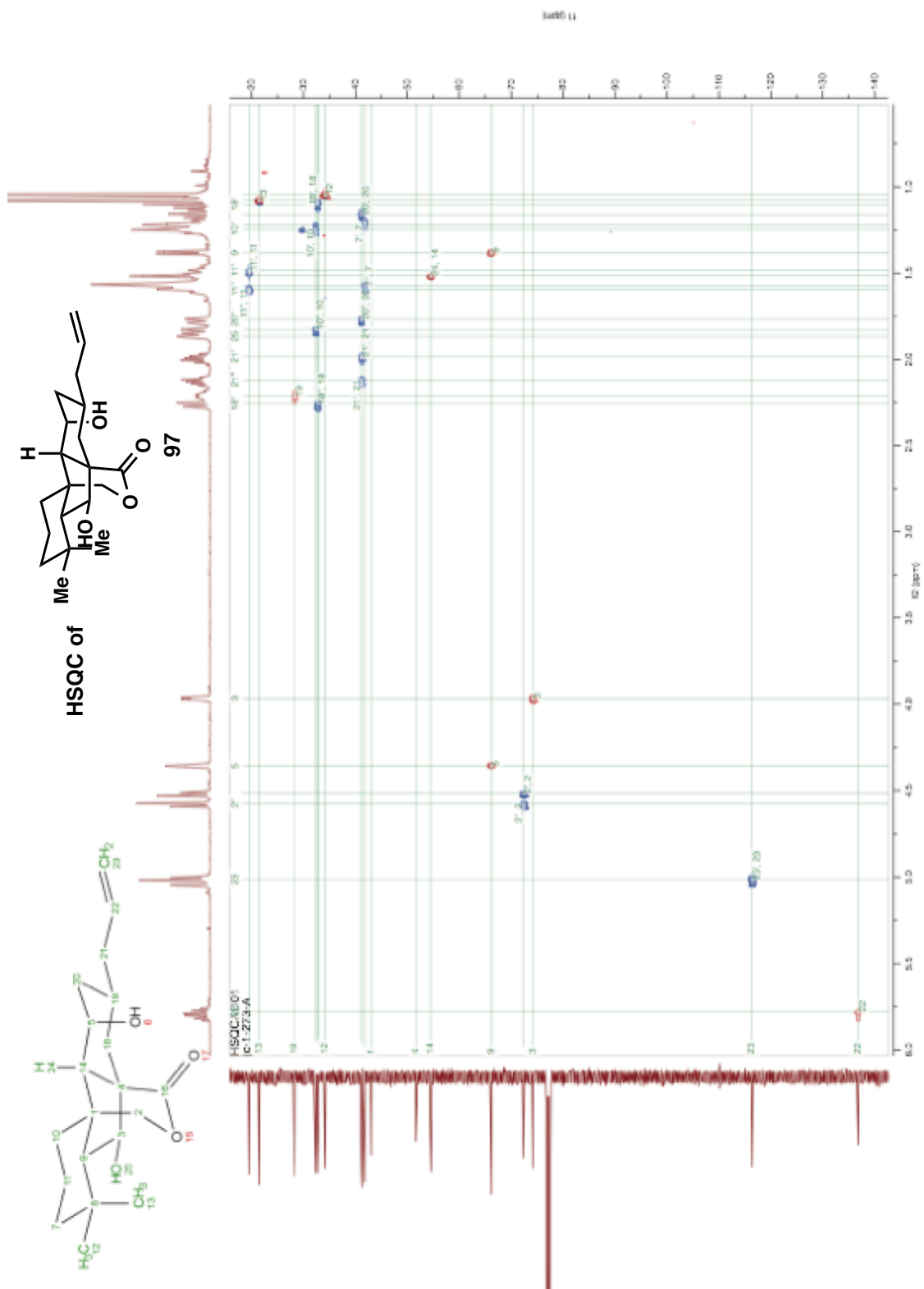


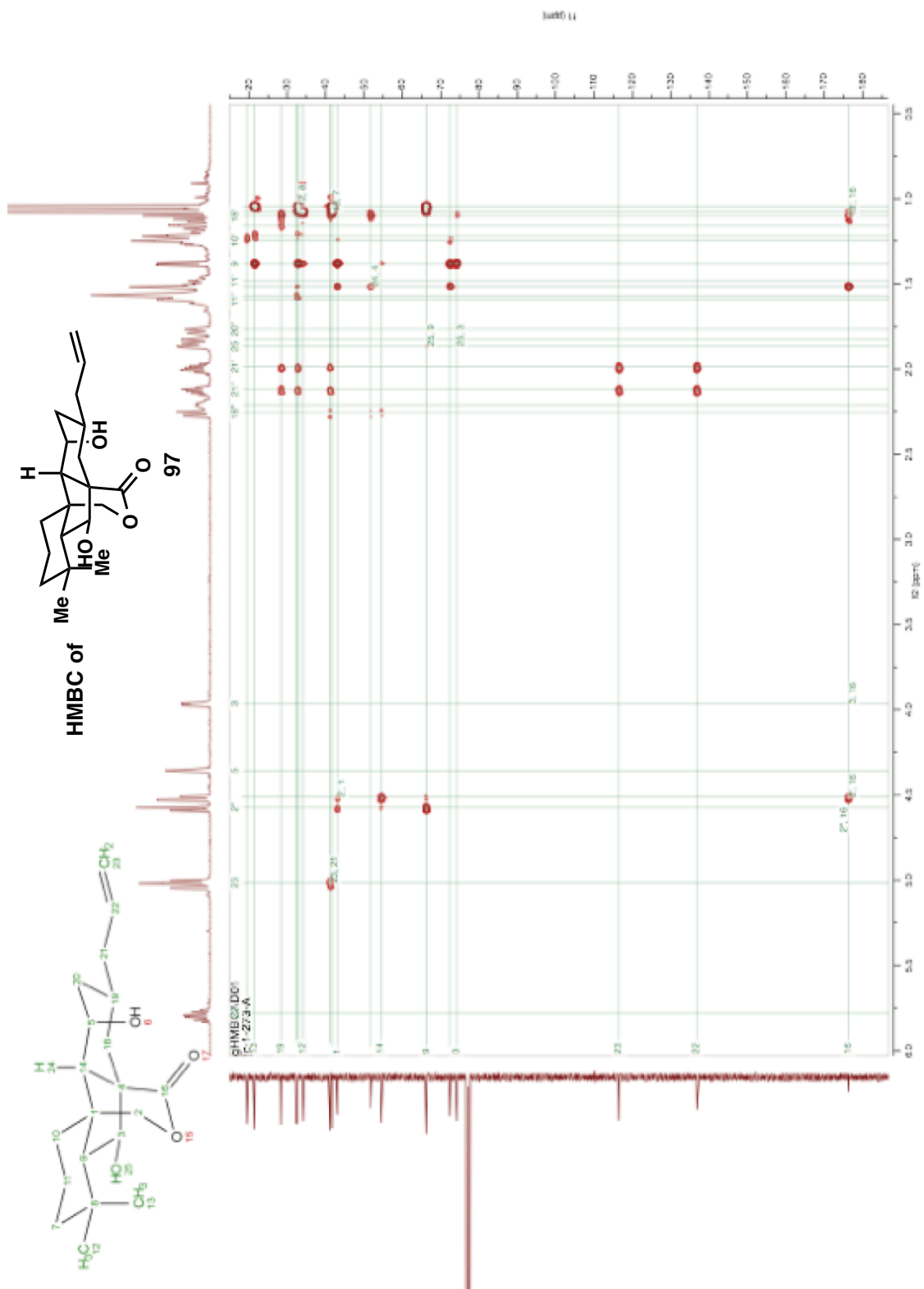


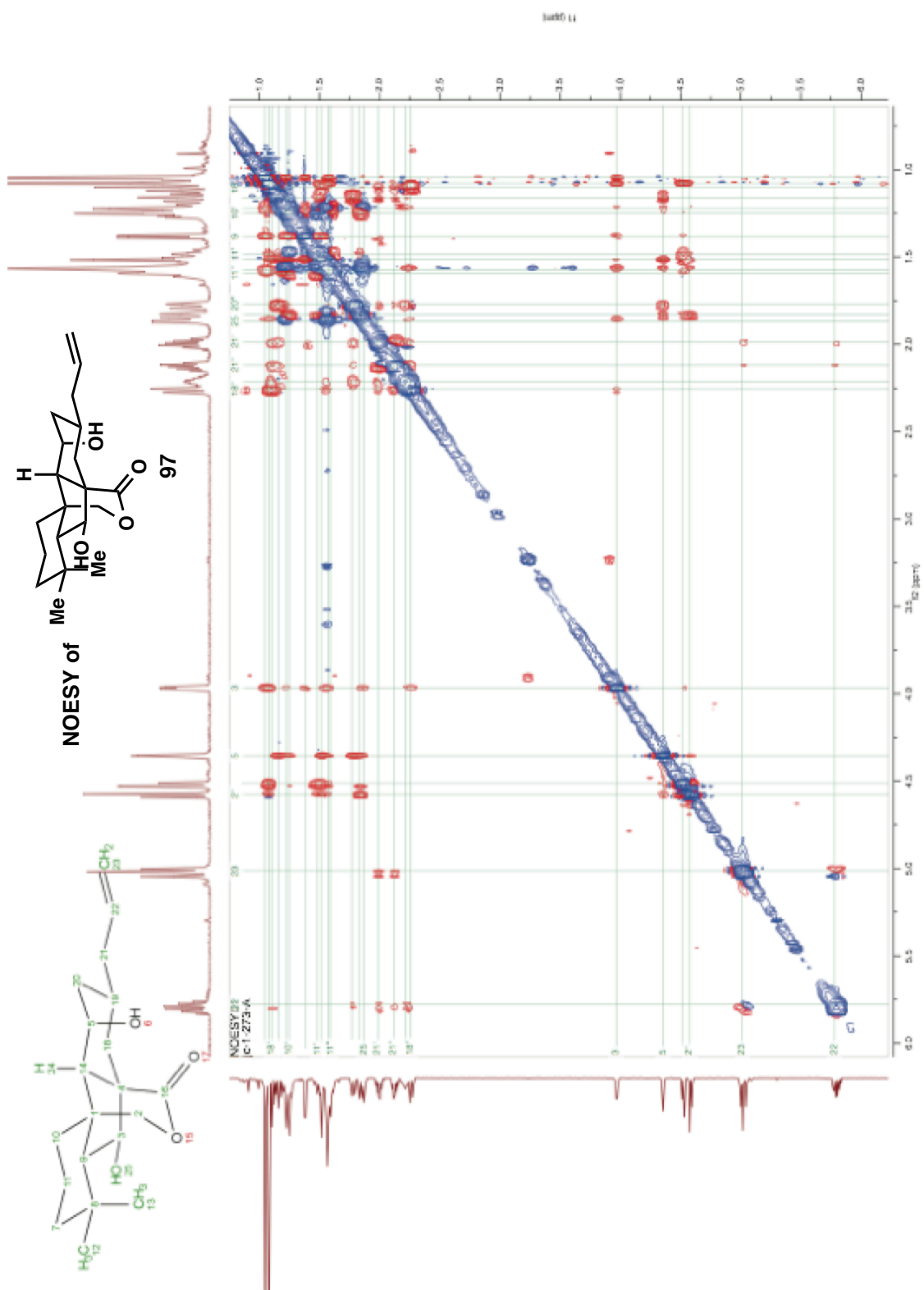


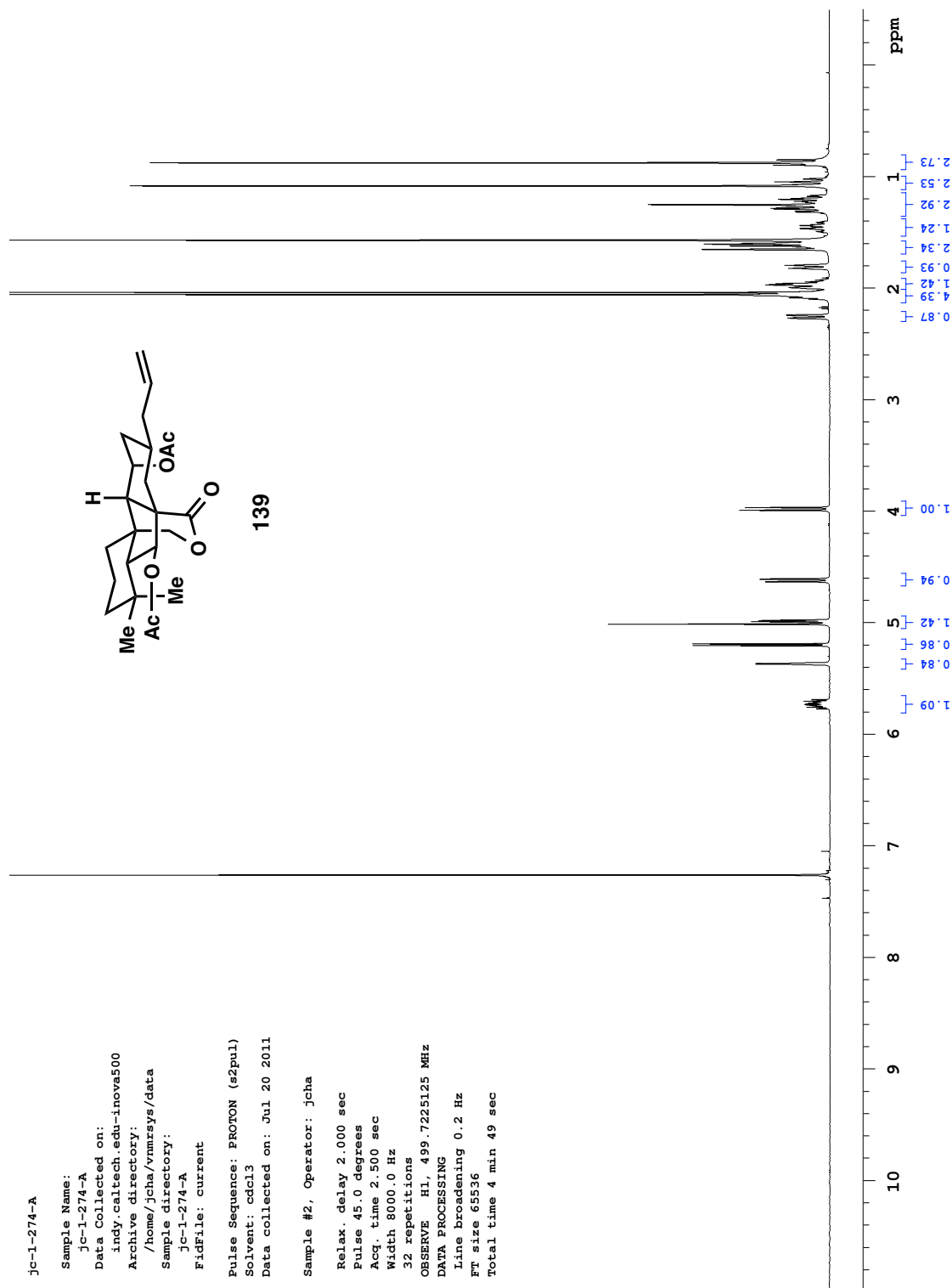


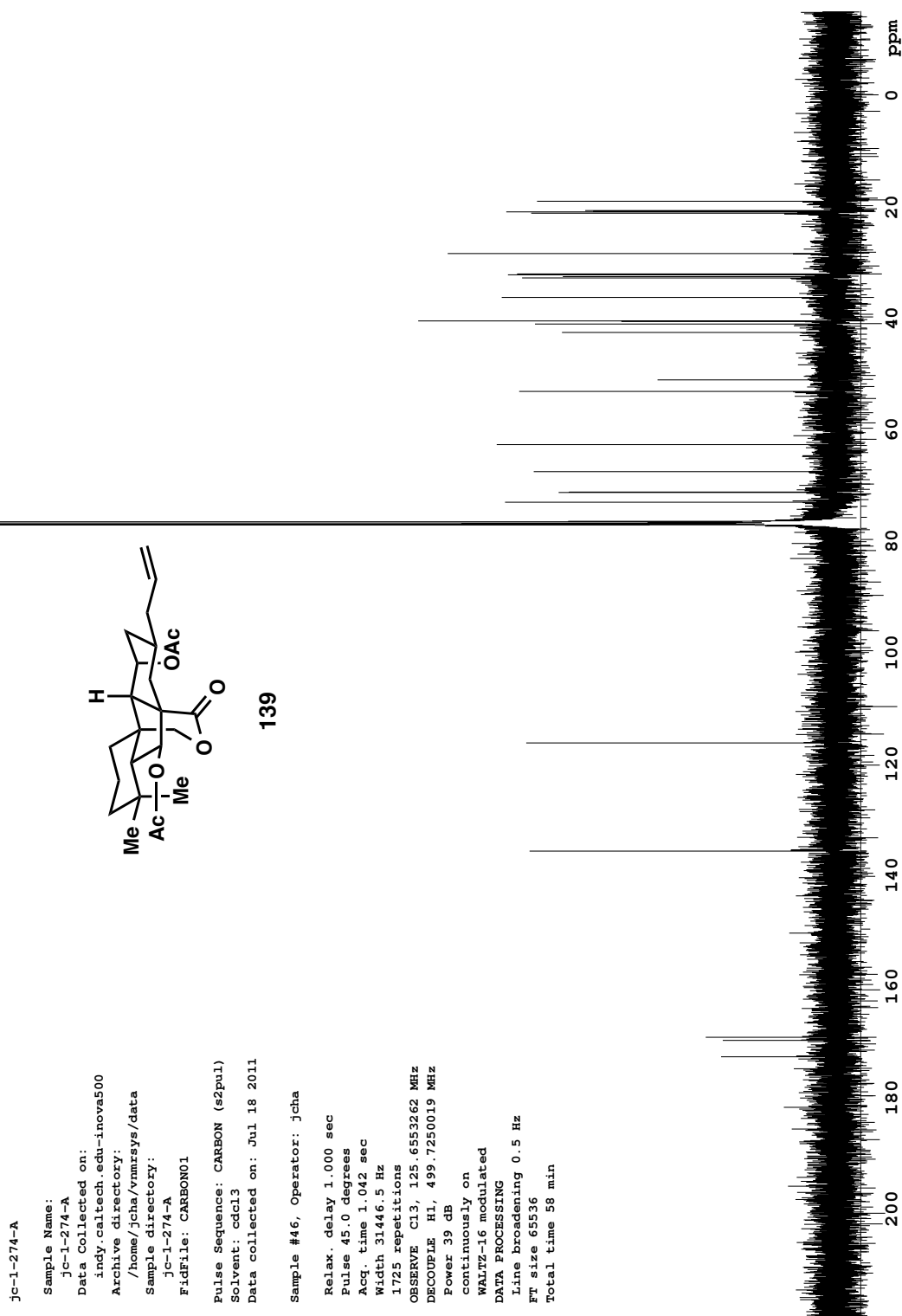


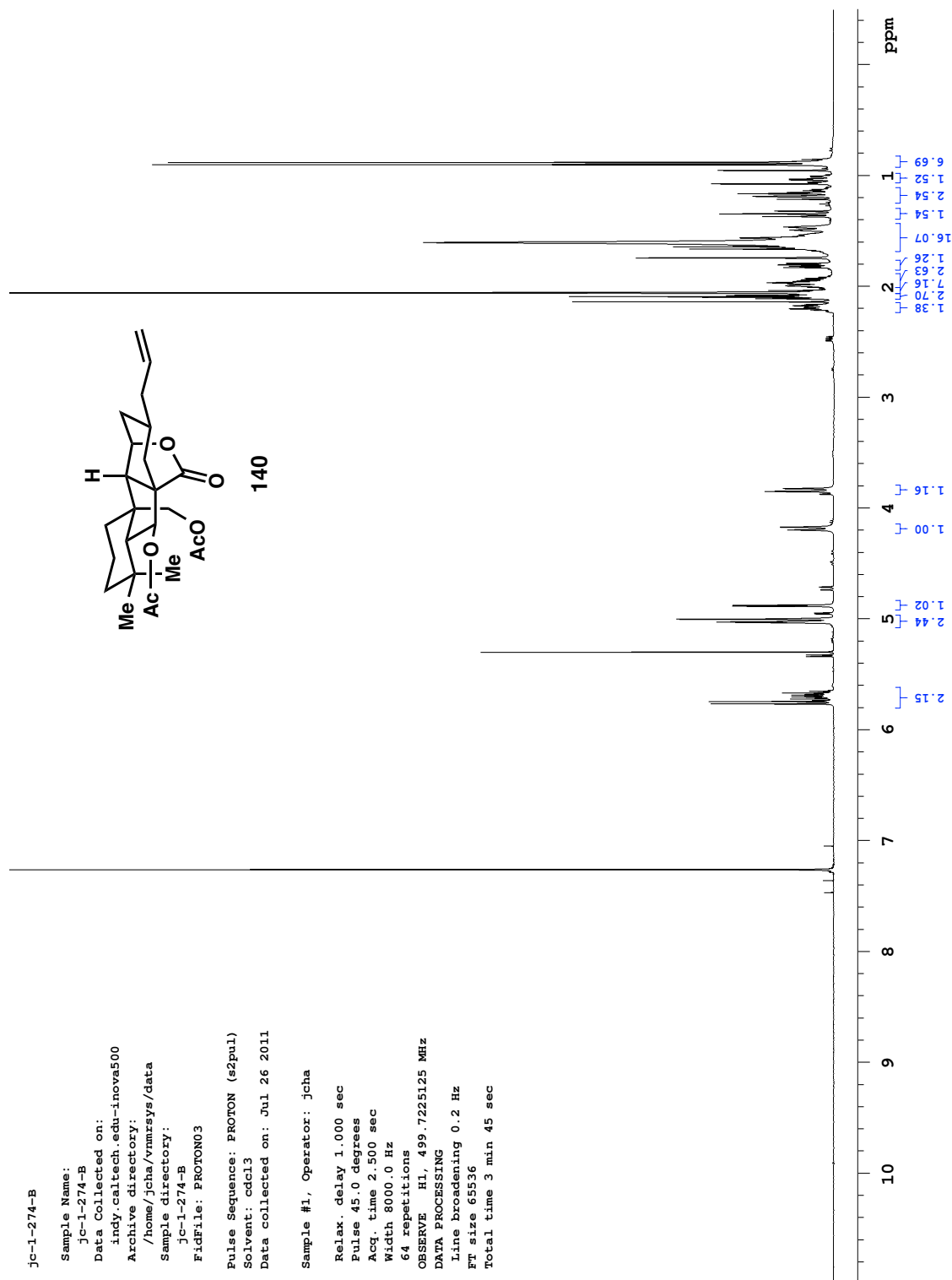


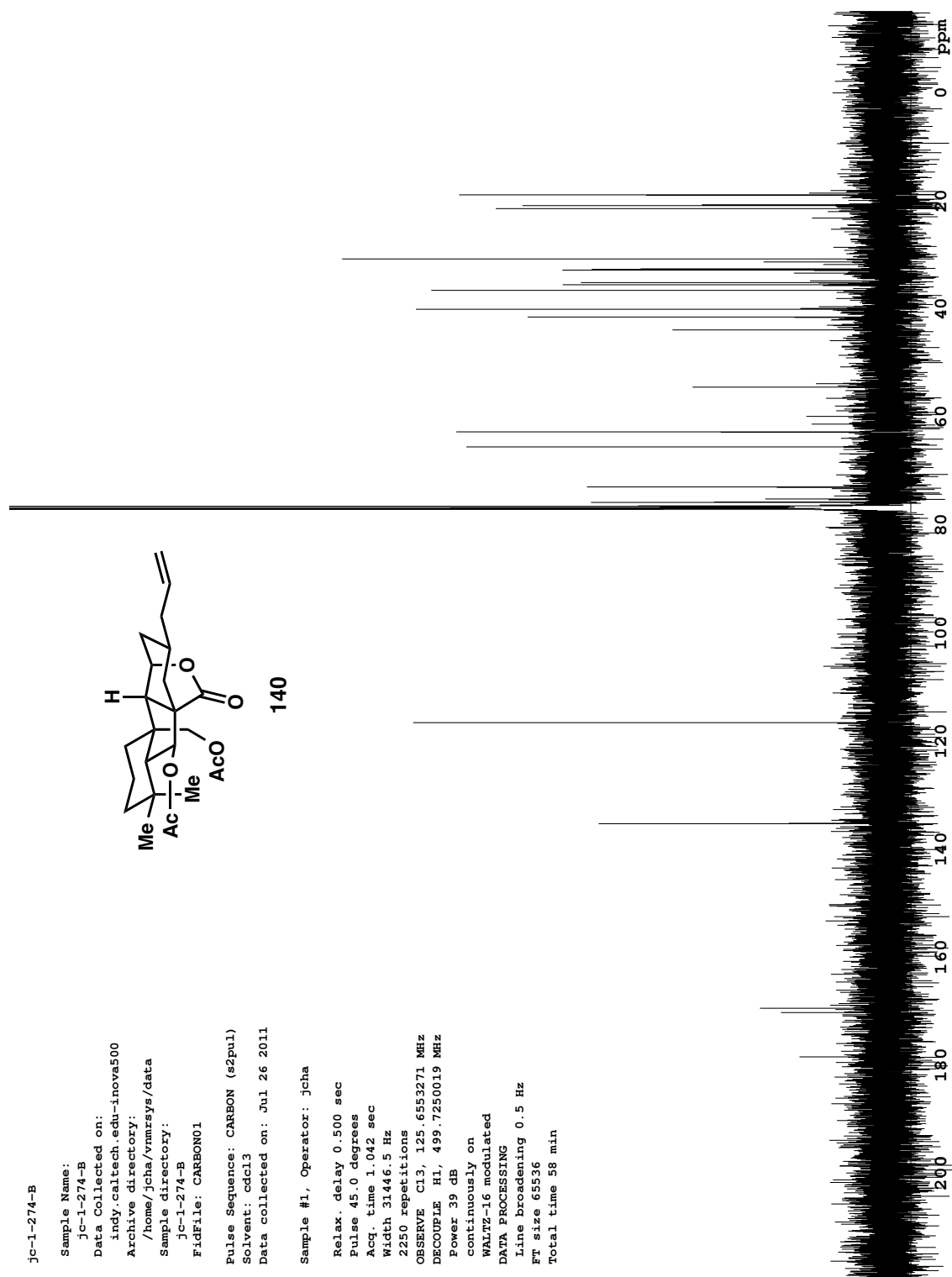


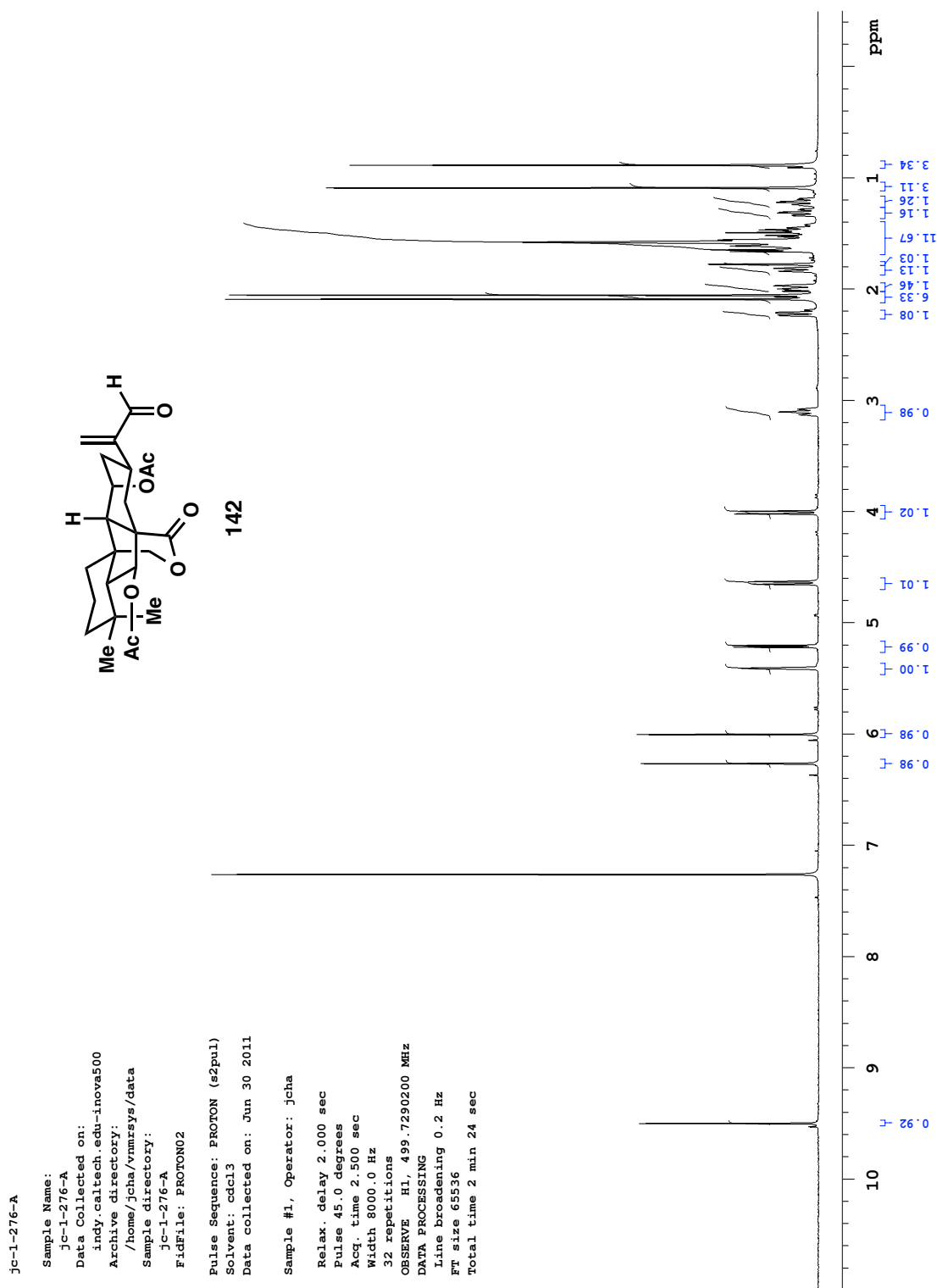


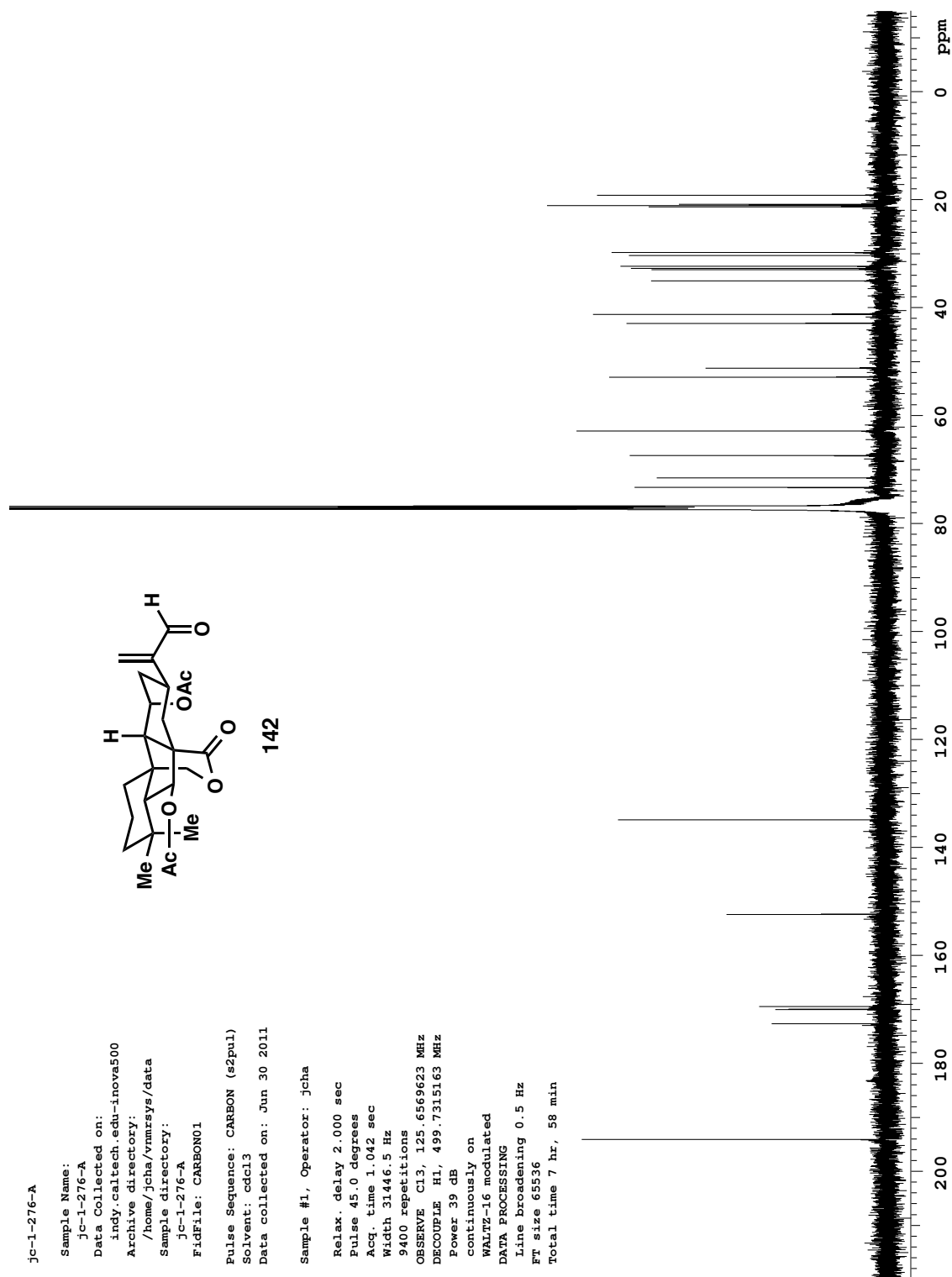


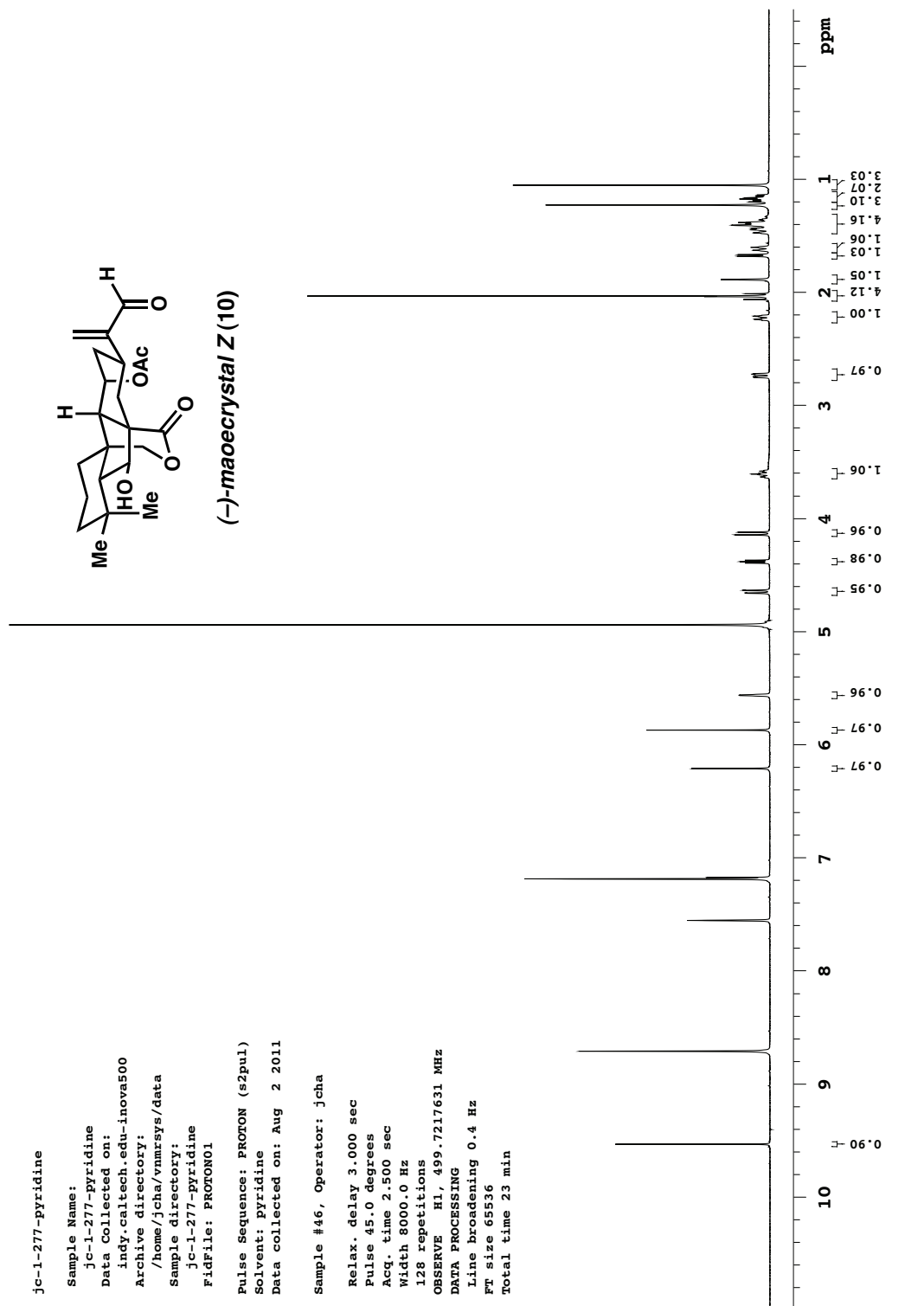


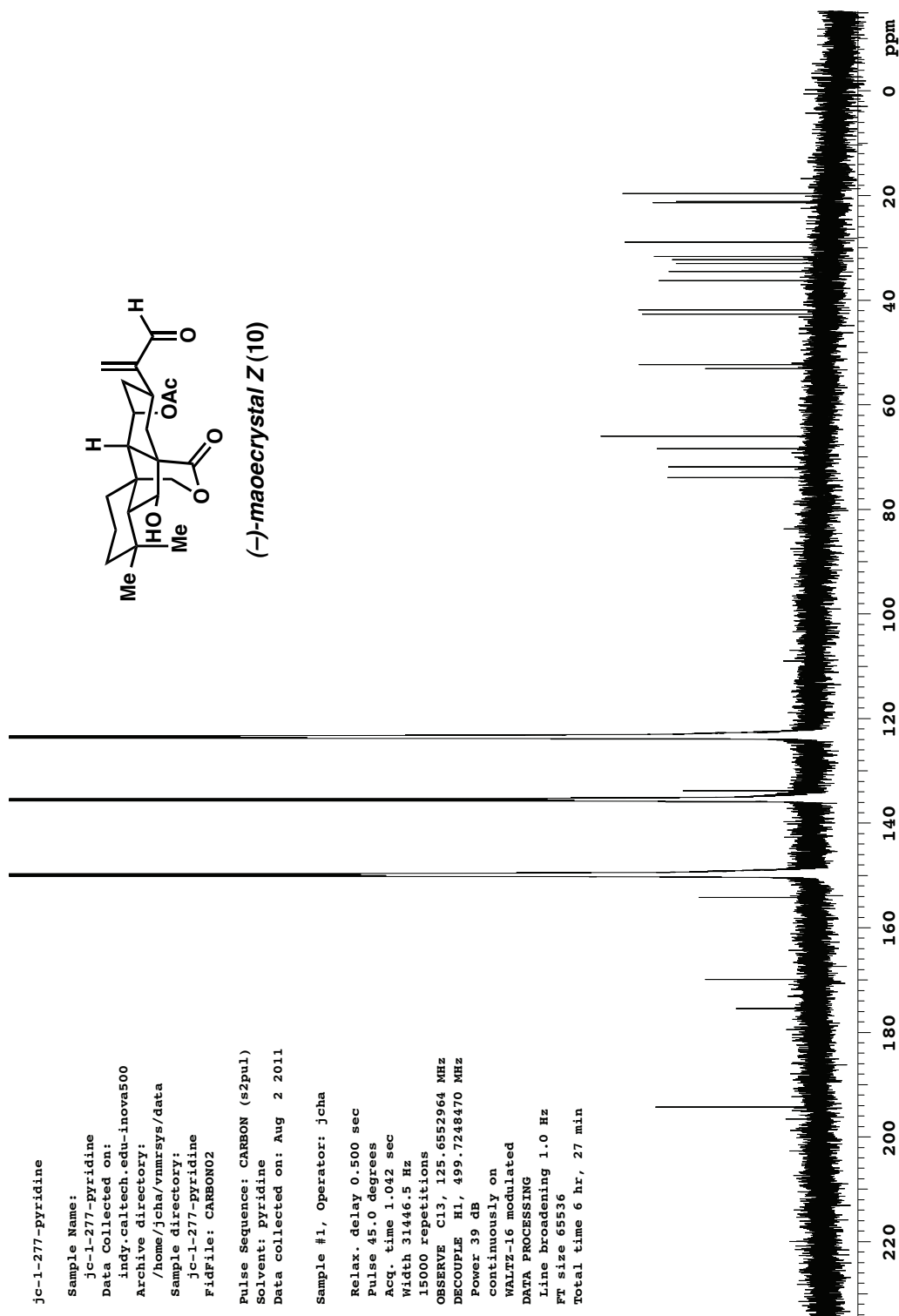


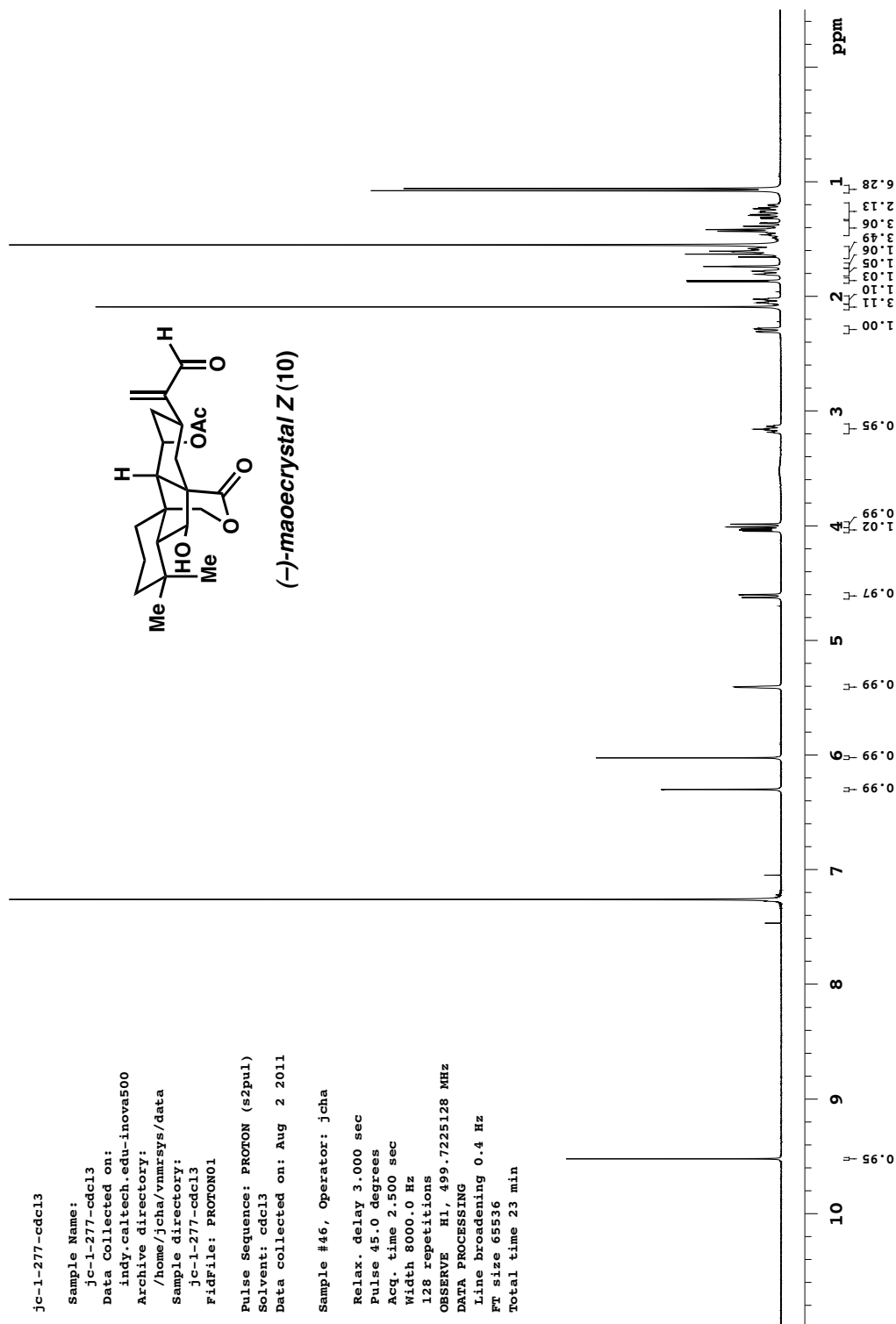




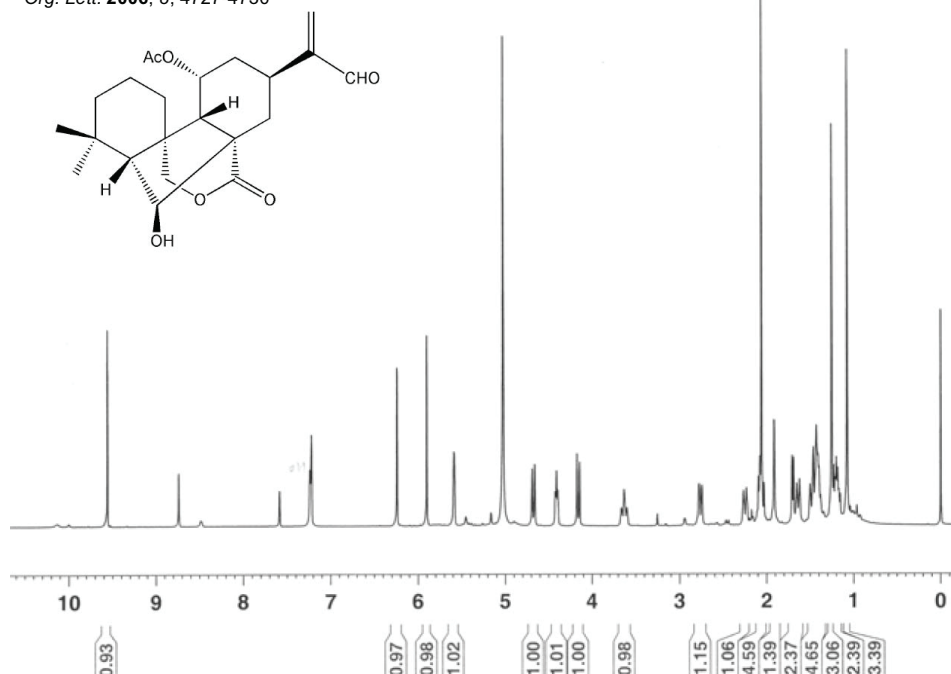








natural sample, from isolation paper:
Org. Lett. **2006**, 8, 4727-4730



pus res, operator: jcm
 lax. delay 3.000 sec
 lse 45.0 degrees
 q. time 2.500 sec
 dth 8000.0 Hz
 8 repetitions
 ERVE H1, 499.7217632 MHz
 A PROCESSING
 no broadening 0.2 Hz
 size 65536
 al time 23 min

synthetic sample

