

Appendix 4

*Spectra Relevant to Chapter 4:
Enantioselective Total Synthesis of Epidithiodiketopiperazine
Natural Product (–)-Acetylpoaranotin*

WHX-7-197-F-RE-INDY-CDCL3

Sample Name:

WHX-7-197-F-RE-INDY-CDCL3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/hxwang/vnmrsys/data

Sample directory:

WHX-7-197-F-RE-INDY-CDCL3
FidFile: WHX-7-197-F-RE-INDY-CDCL3

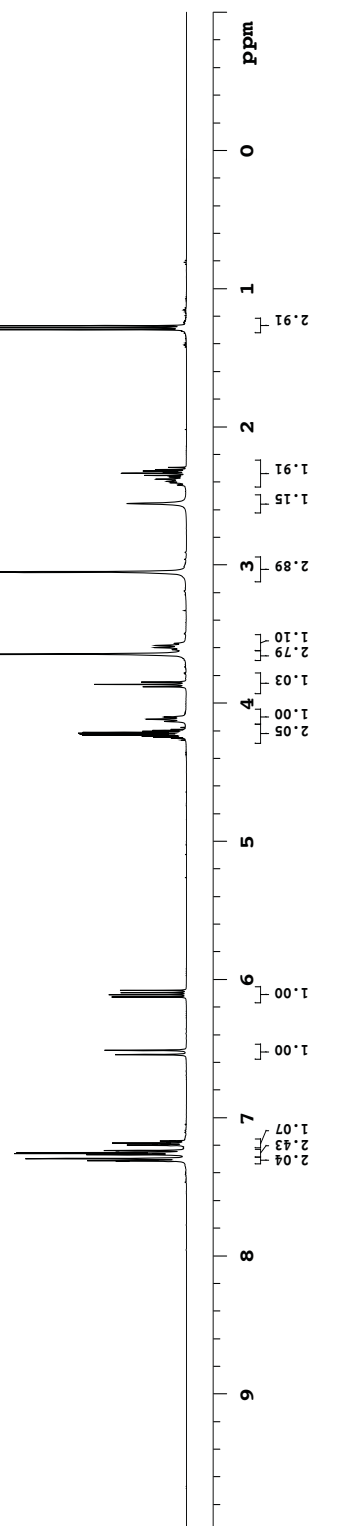
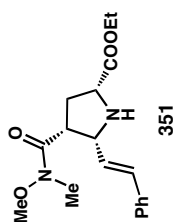
Pulse Sequence: PROTON (s2pul)

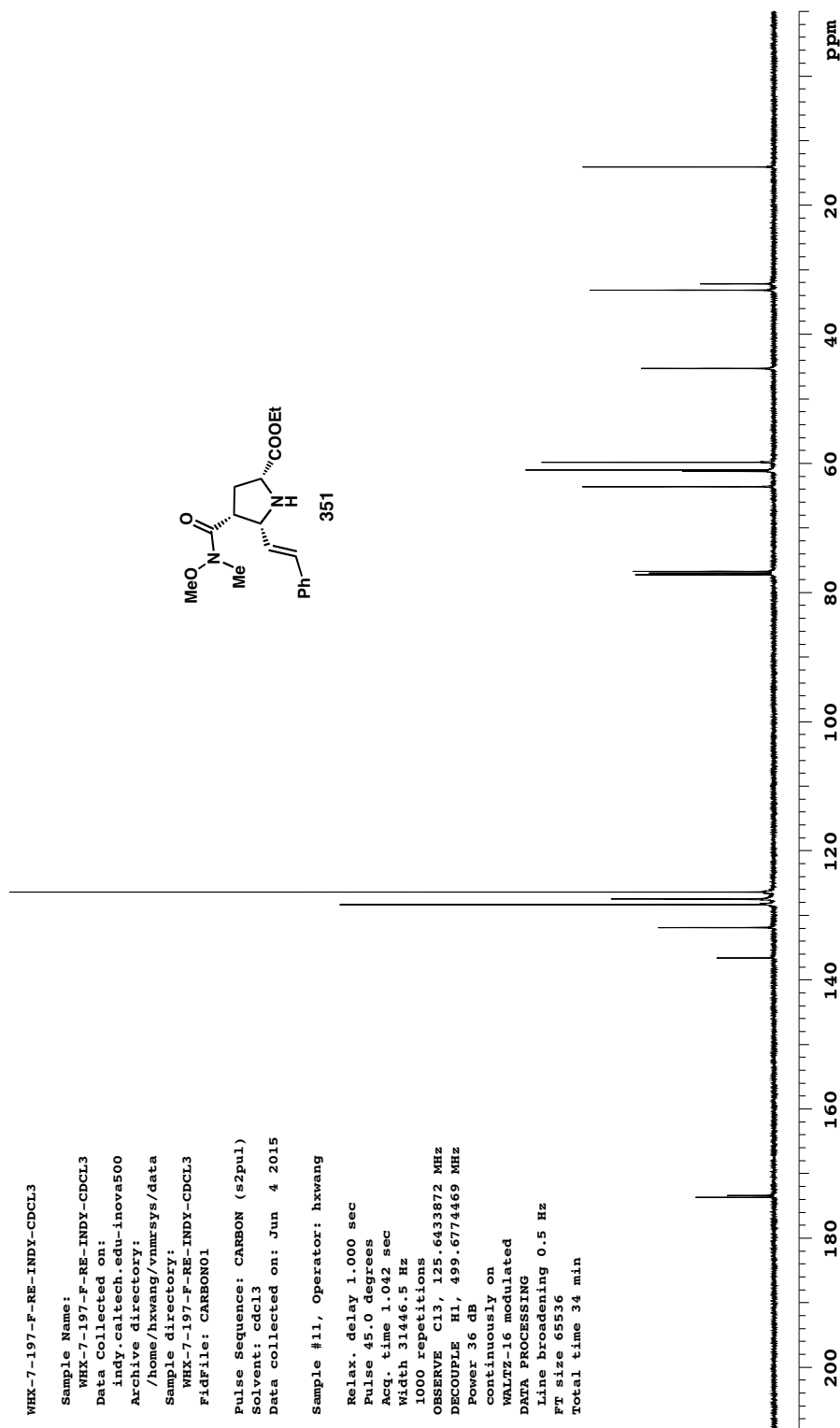
Solvent: cdcl3
Data collected on: Jun 4 2015

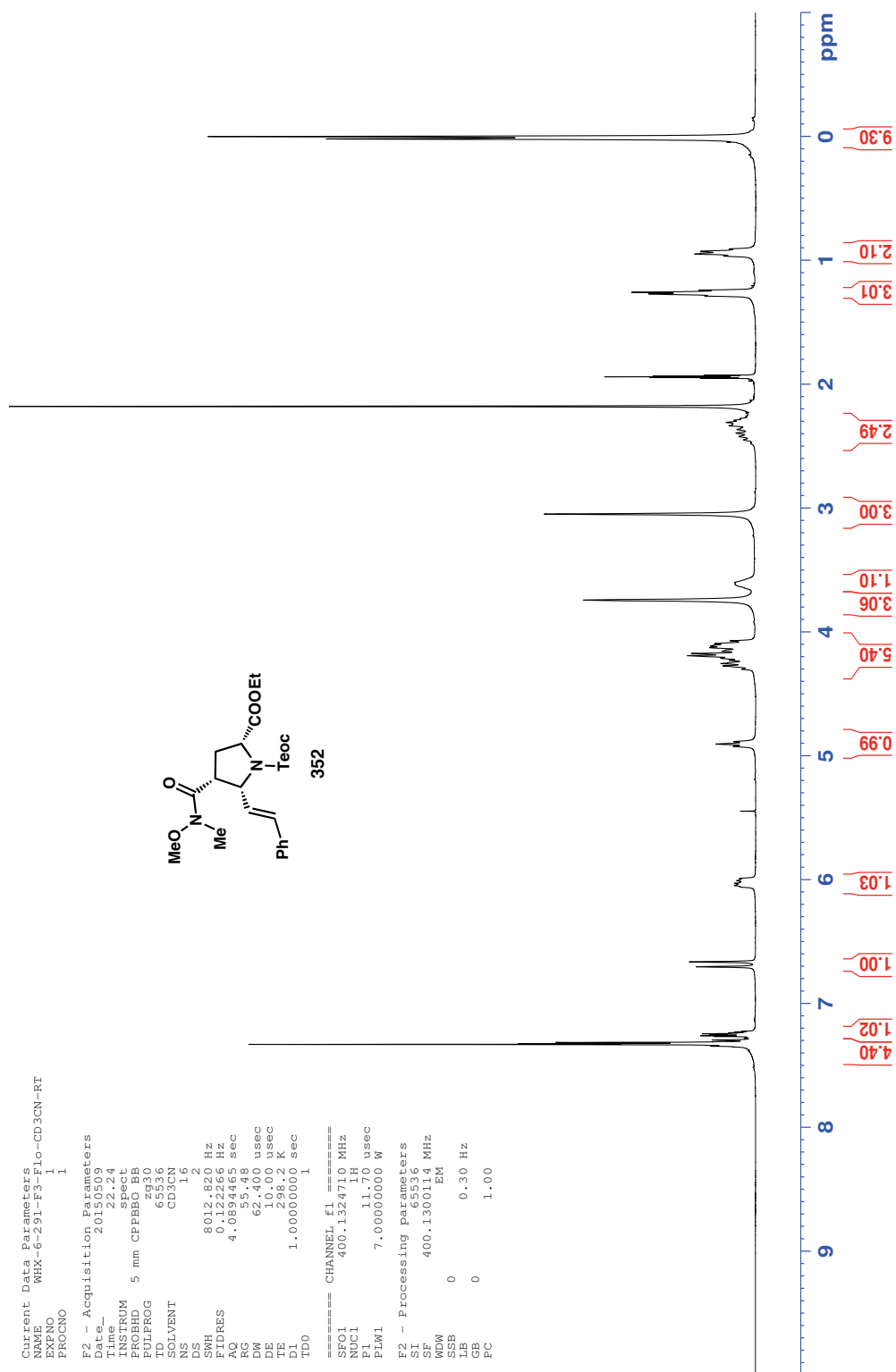
Sample #11, Operator: hxwang

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions

OBSERVE H1, 499.6749695 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec







WHX-6-291-F3-Siena-CD3CN-60C

Sample Name:

WHX-6-291-F3-Siena-CD3CN-60C
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hhwang/vnmrsys/data

Sample directory:

WHX-6-291-F3-Siena-CD3CN-60C
FidFile: WHX-6-291-F3-Siena-CD3CN-60C-Proton

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn
Data collected on: May 9 2015

Temp. 60.0 C / 333.1 K

Operator: hhwang

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

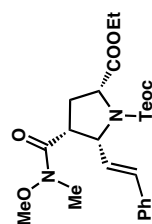
8 repetitions

OBSERVE H1, 399.7970633 MHz

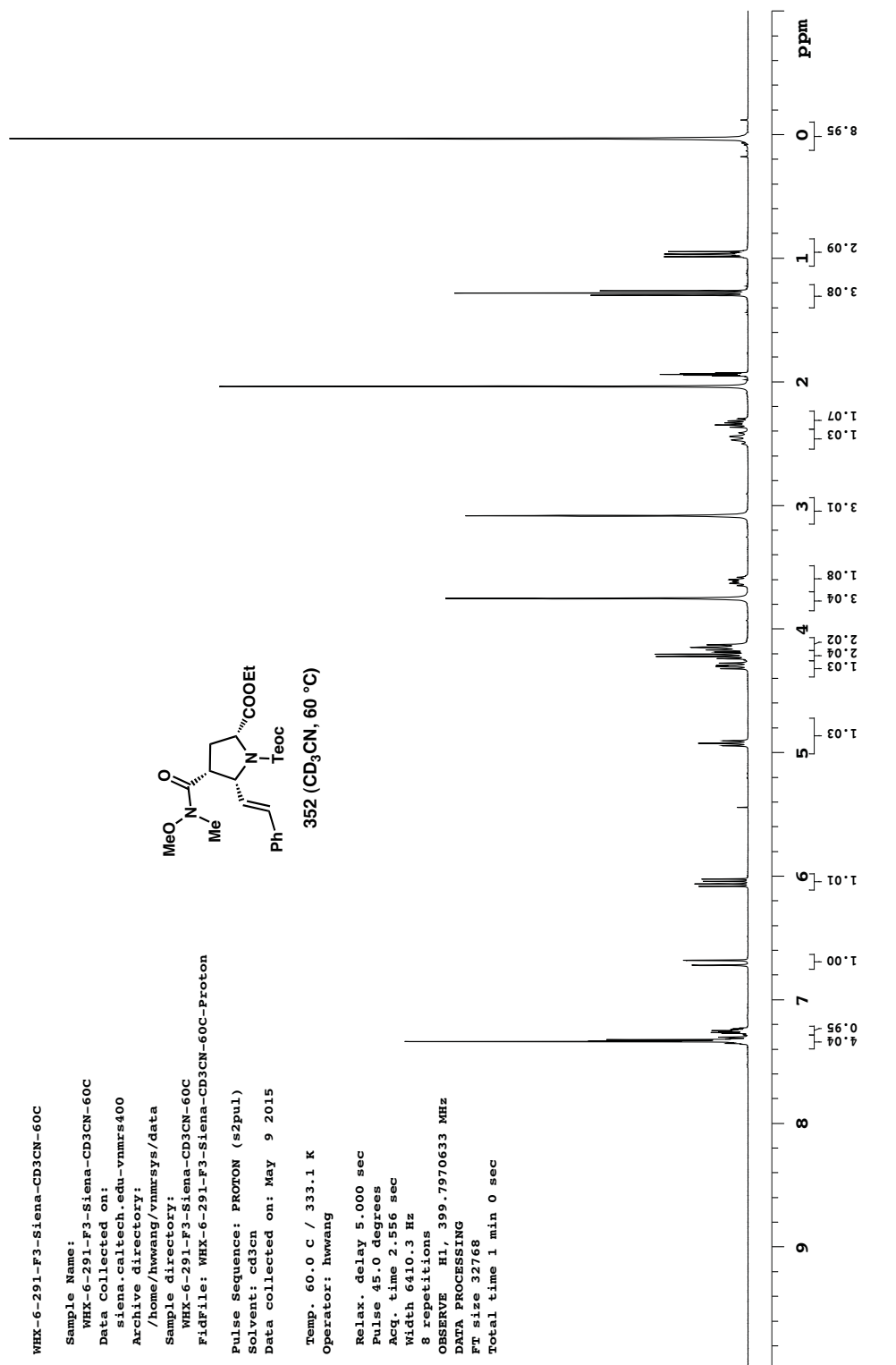
DATA PROCESSING

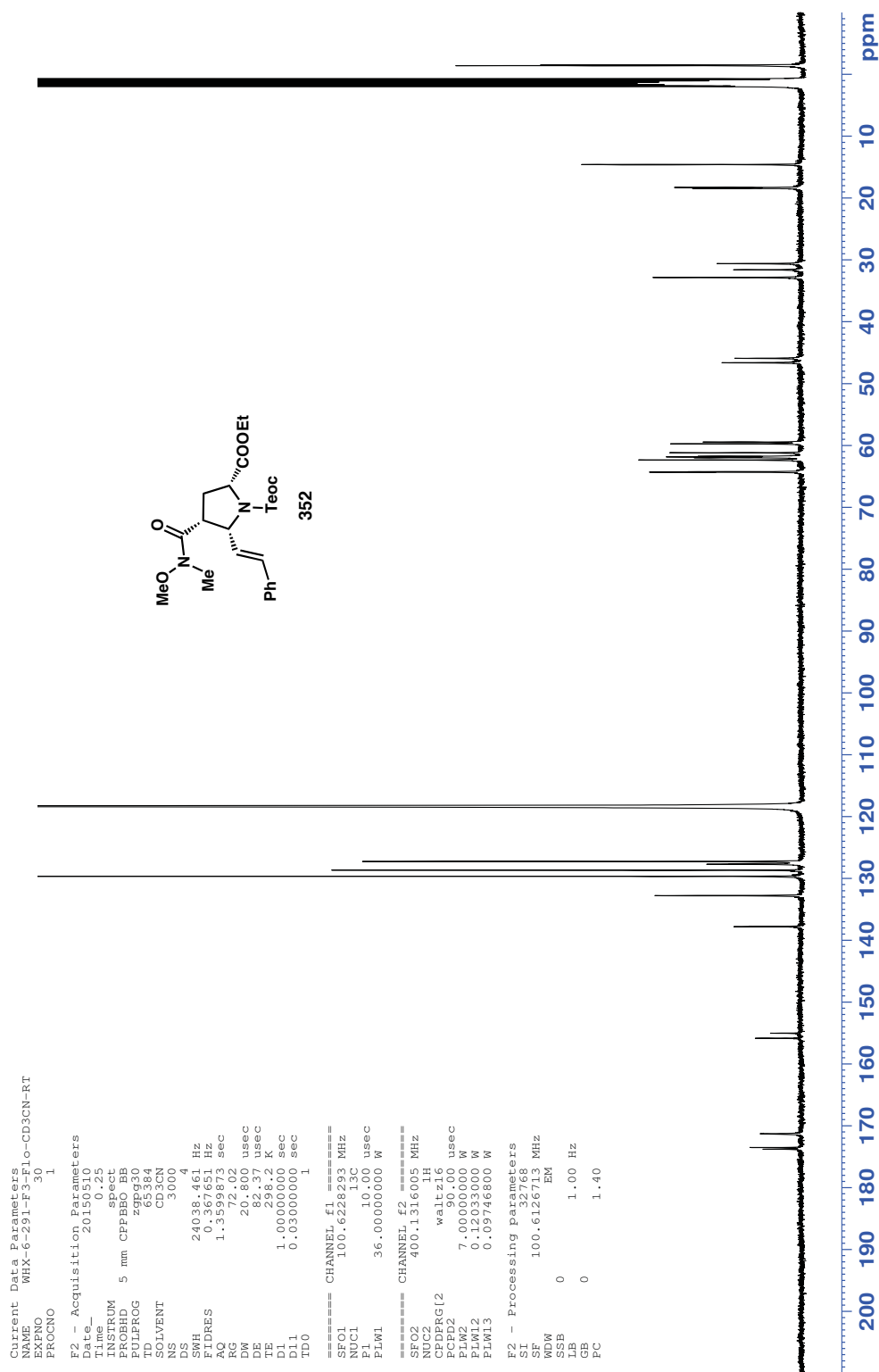
FT size 32768

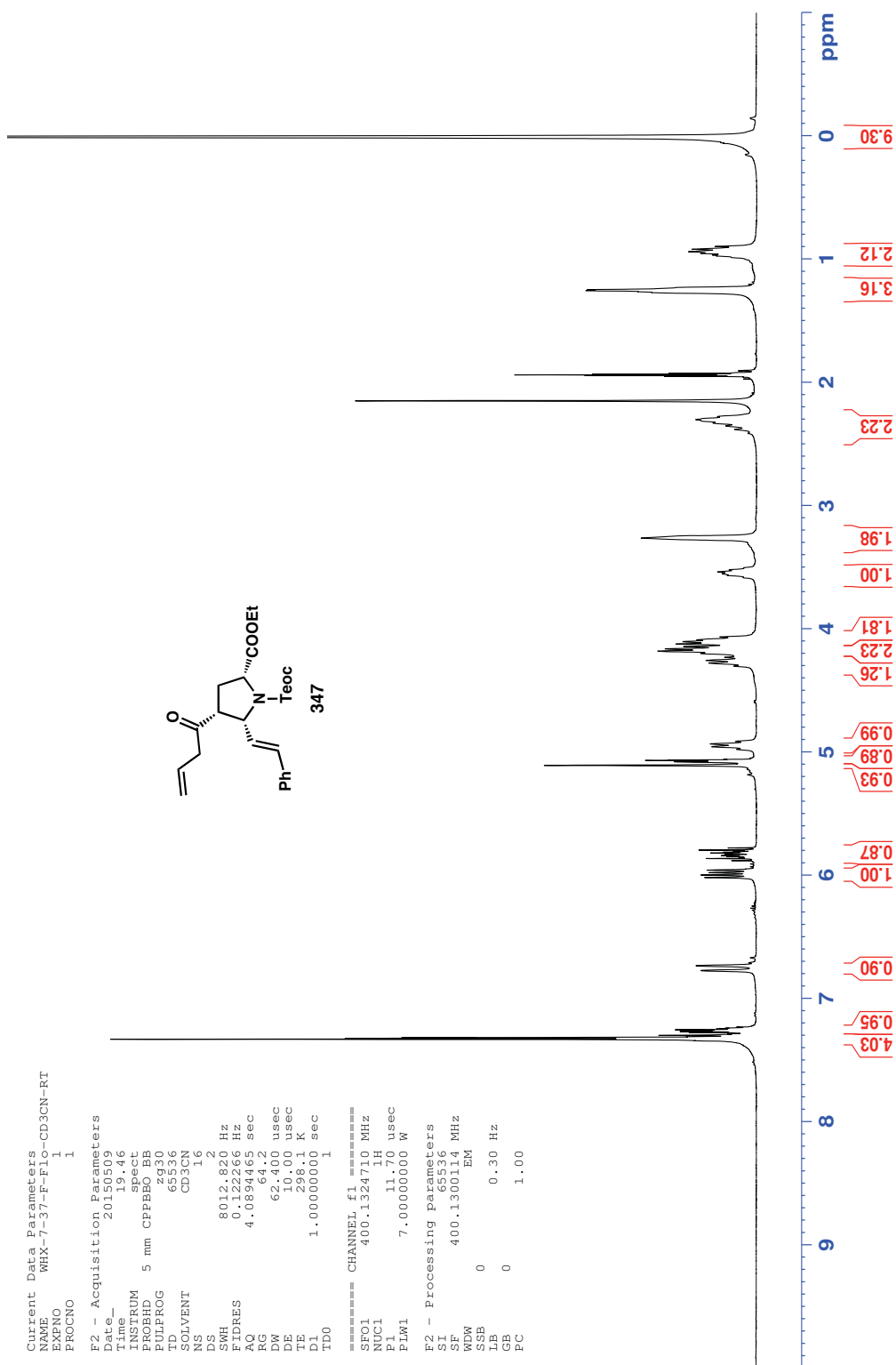
Total time 1 min 0 sec



352 (CD₃CN, 60 °C)







WHX-7-37-F-Siena-CD3CN-60C-H

Sample Name:

WHX-7-37-F-Siena-CD3CN-60C-H
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-37-F-Siena-CD3CN-60C-H
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: May 9 2015

Temp. 60.0 C / 333.1 K

Sample #1, Operator: hwang

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

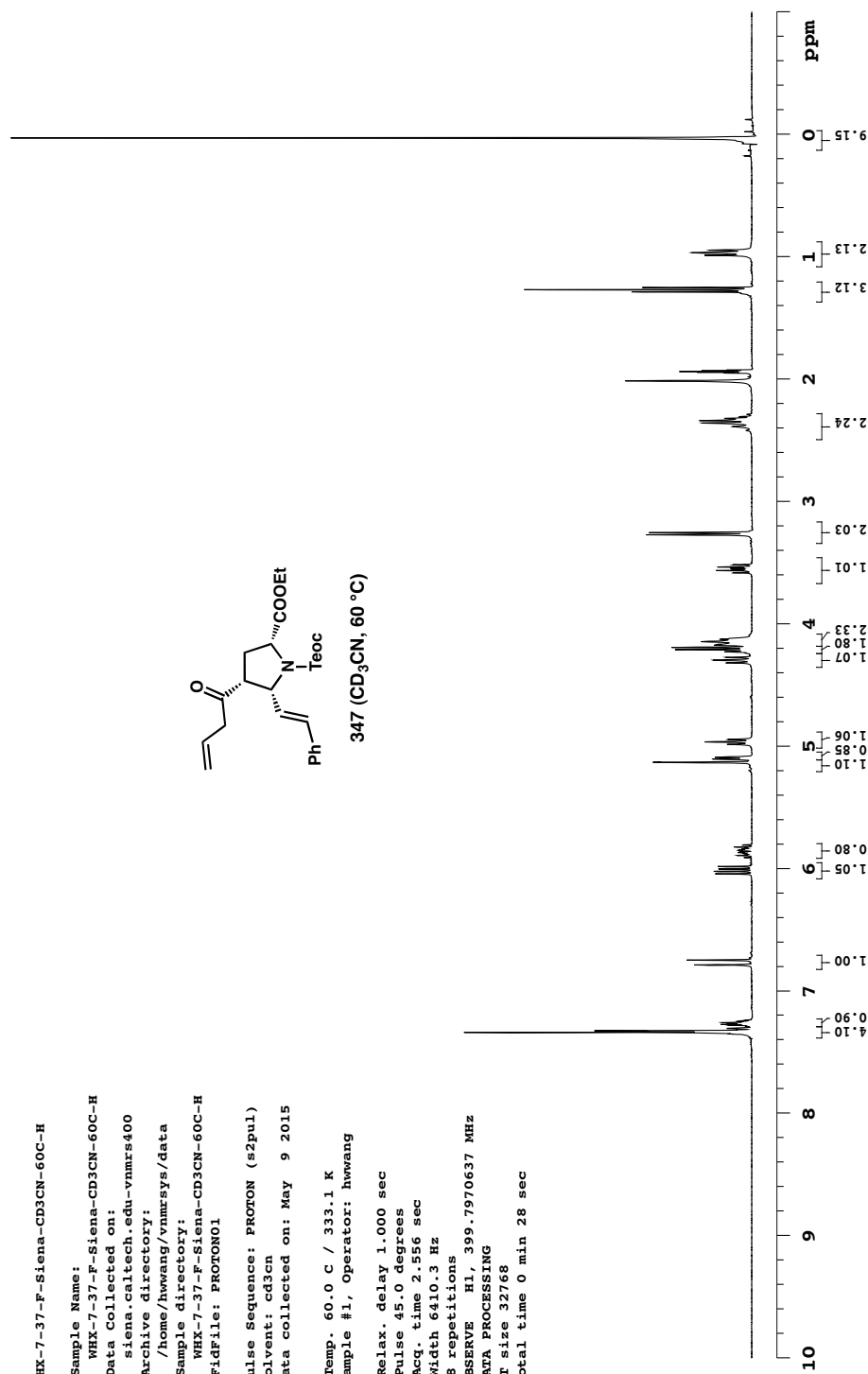
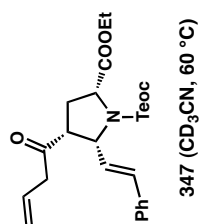
8 repetitions

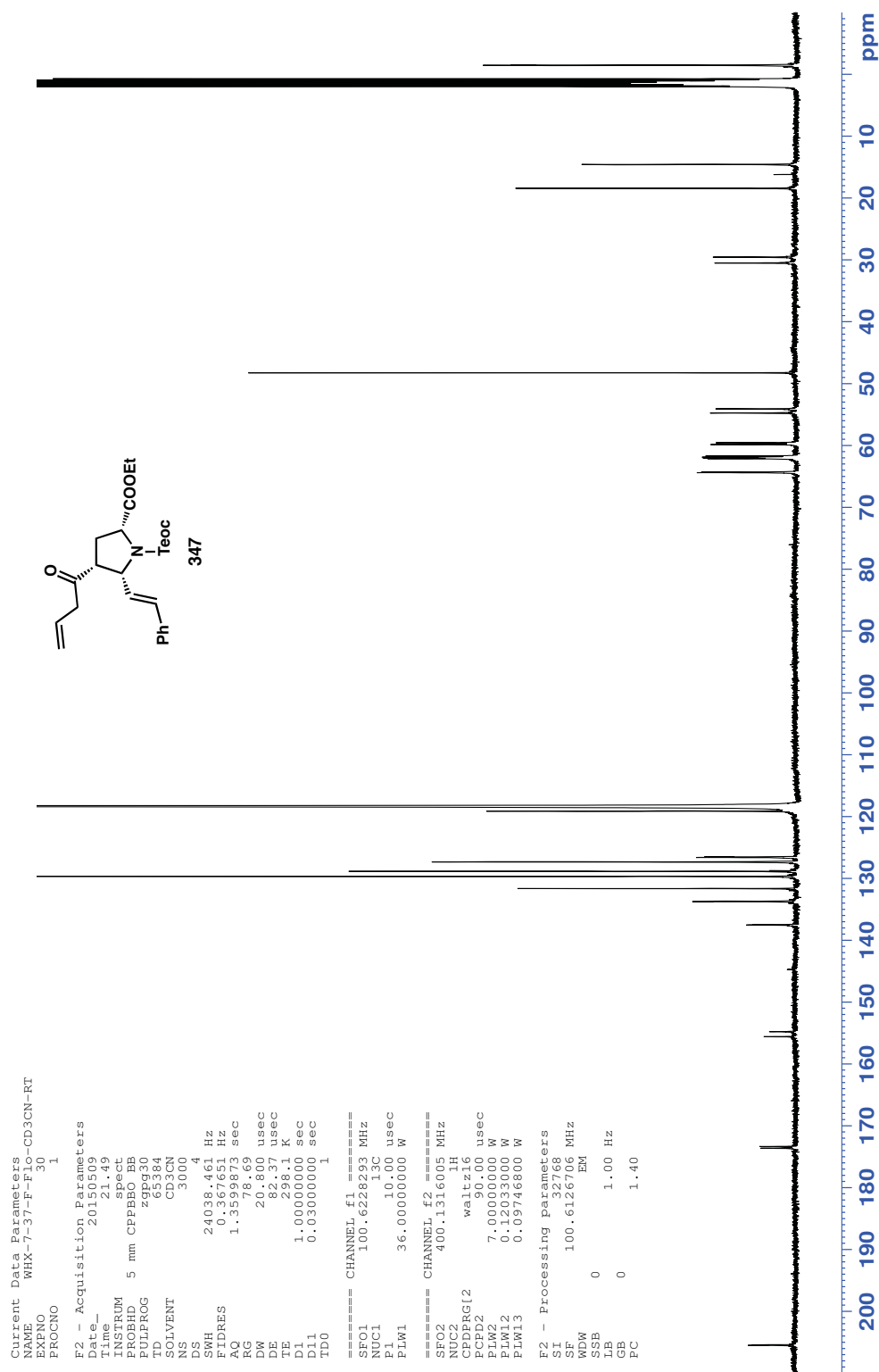
OBSERVE HL, 399.7970637 MHz

DATA PROCESSING

FT size 32768

Total time 0 min 28 sec





WHX-7-177-F-Siena-CD3CN

Sample Name:

WHX-7-177-F-Siena-CD3CN

Data Collected on:

sienna.caltech.edu-vnmrs400

Archive directory:

/home/hwang/vnmrsys/data

Sample directory:

WHX-7-177-F-Siena-CD3CN

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn

Data collected on: May 11 2015

Temp. 25.0 C / 298.1 K

Sample #2, Operator: hwang

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

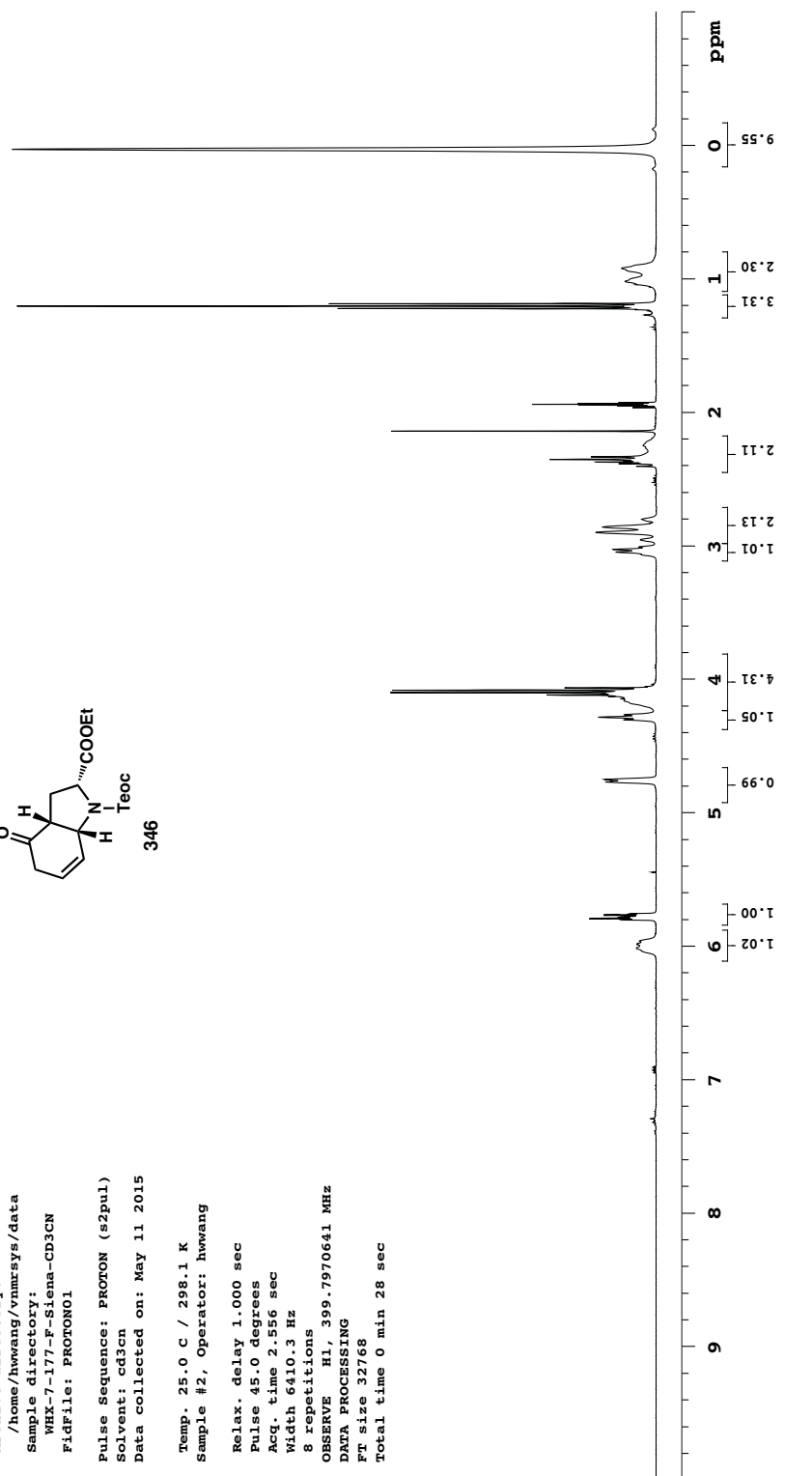
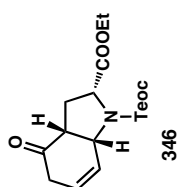
8 repetitions

OBSERVE H1, 399.7970641 MHz

DATA PROCESSING

FT size 32768

Total time 0 min 28 sec



WHX-7-177-F-Siena-CD3CN-65C

Sample Name:

WHX-7-177-F-Siena-CD3CN-65C

Data Collected on:

siena.caltech.edu-vnmrs400

Archive directory:

/home/hwwang/vnmrsys/data

Sample directory:

WHX-7-177-F-Siena-CD3CN-65C

FidFile: WHX-7-177-F-Siena-CD3CN-65C-Proton

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn

Data collected on: May 11 2015

Temp. 65.0 C / 338.1 K

Operator: hwwang

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

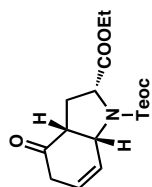
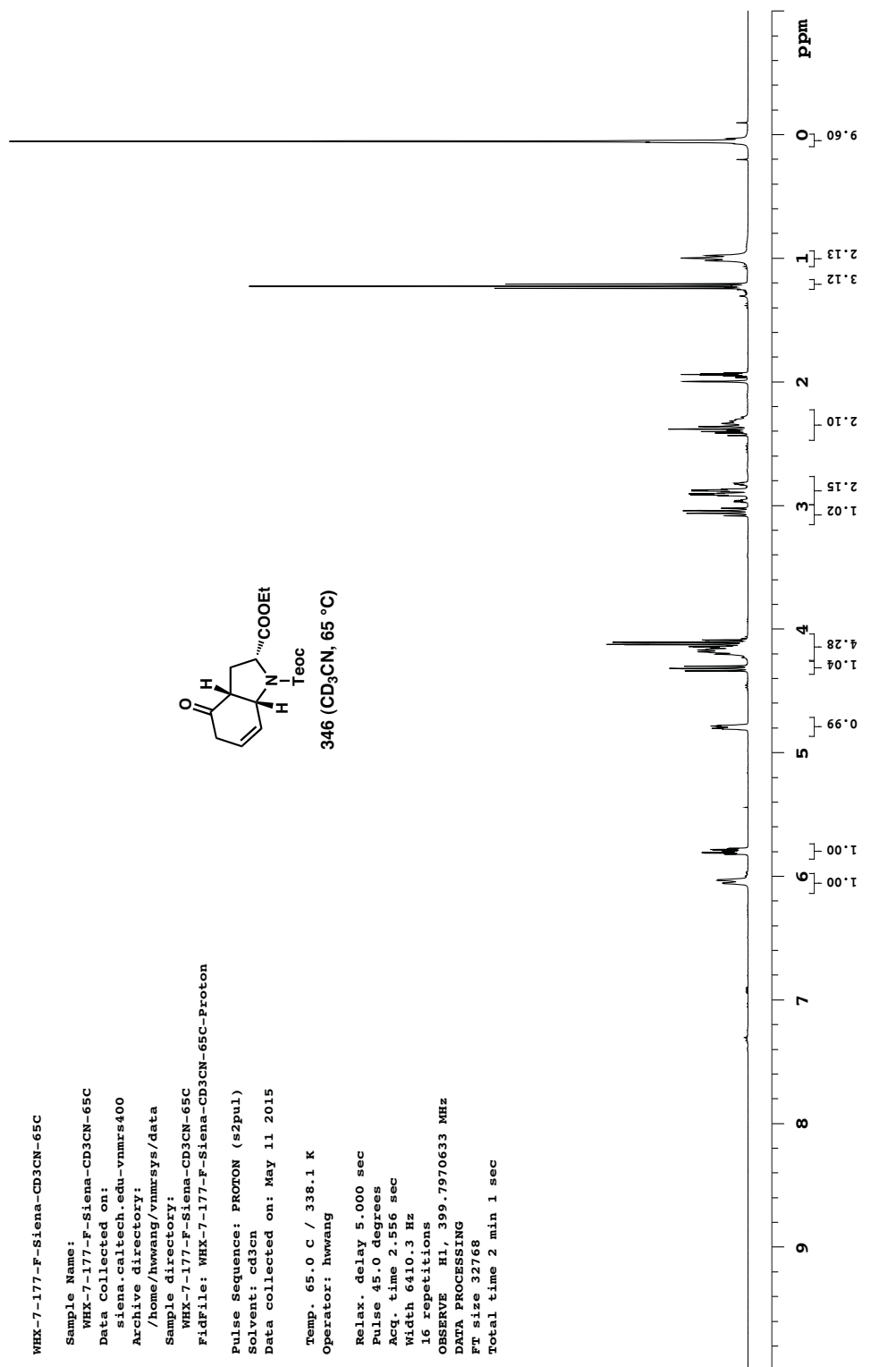
16 repetitions

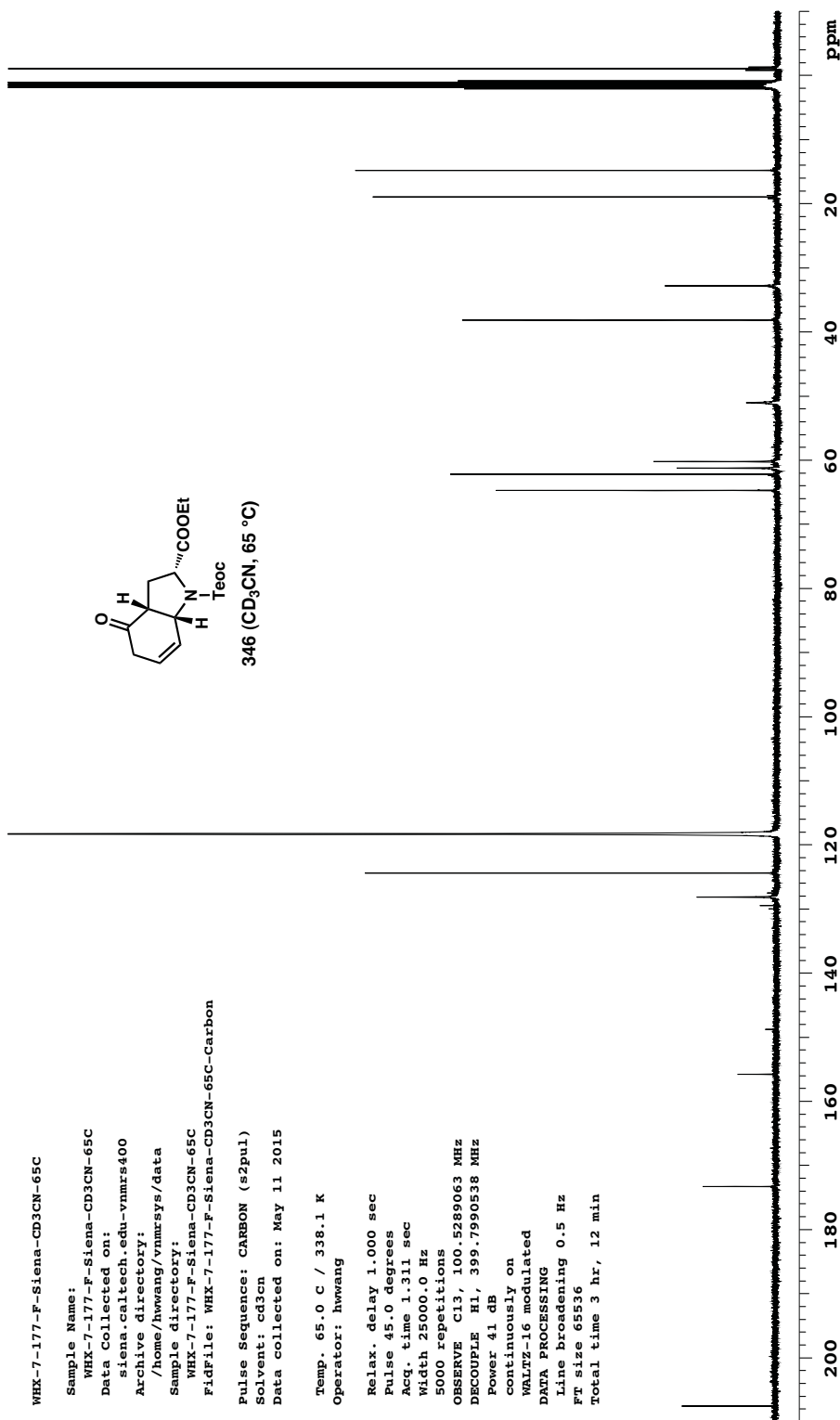
OBSERVE H1, 399.7970633 MHz

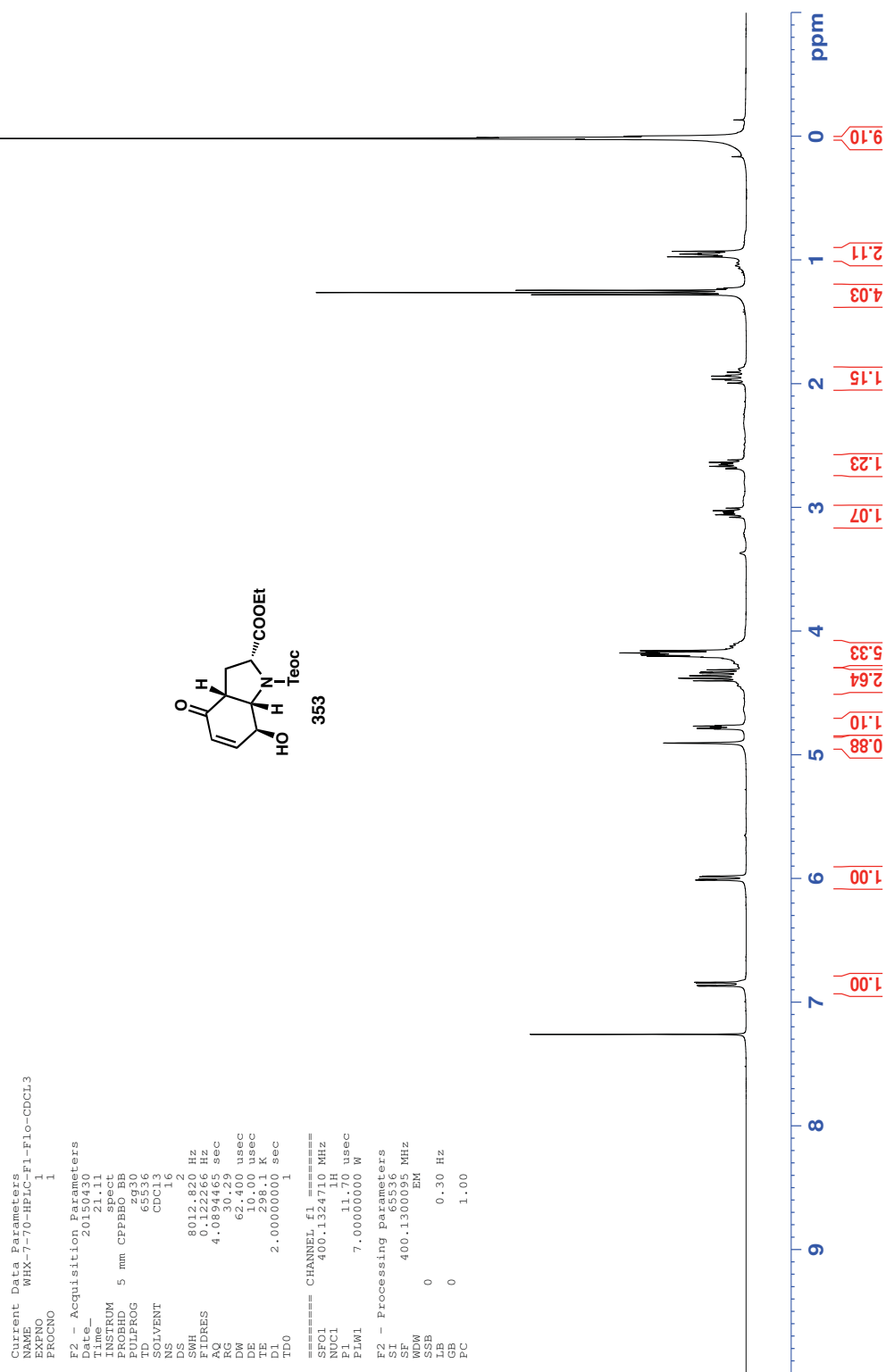
DATA PROCESSING

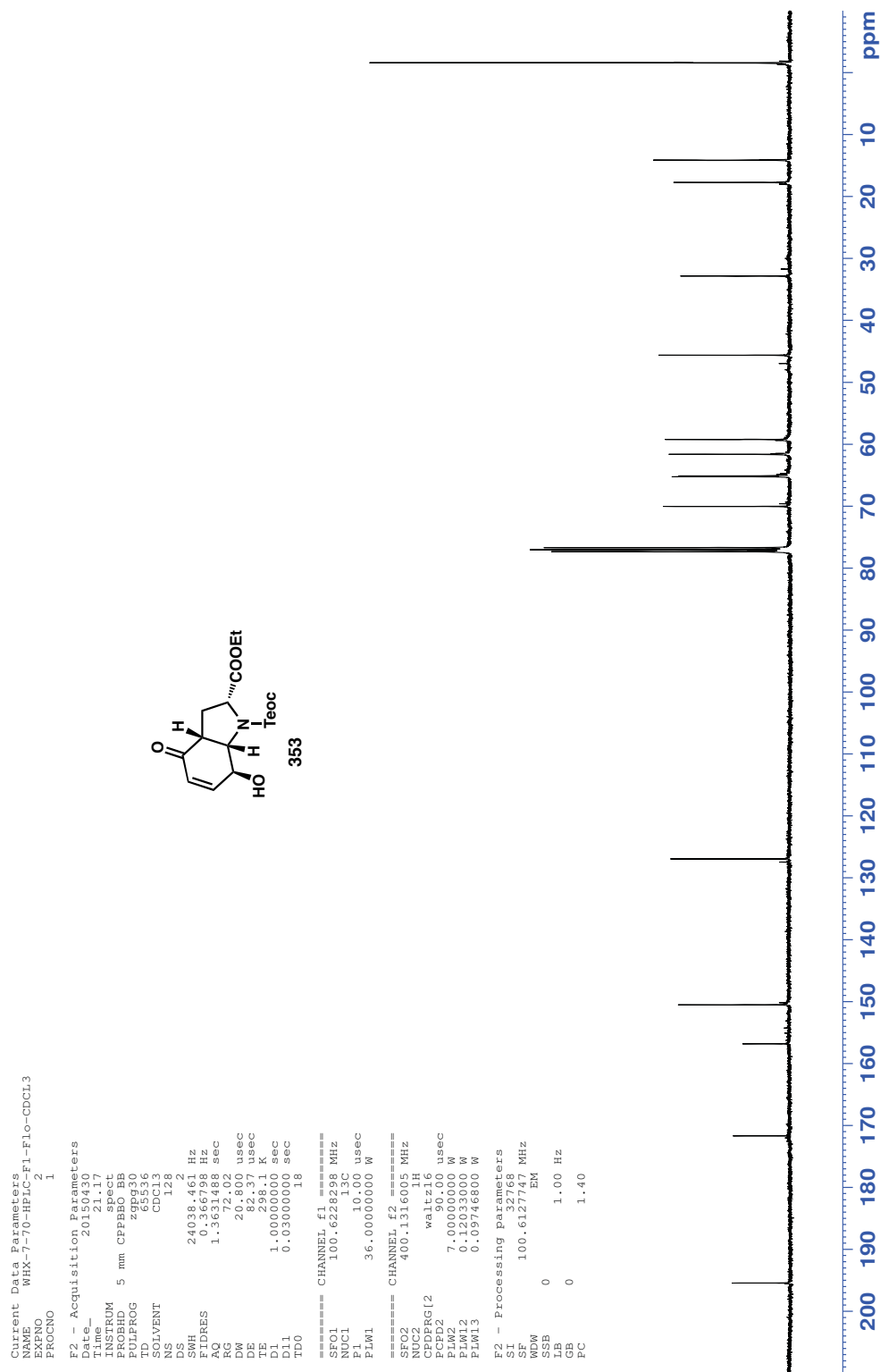
FT size 32768

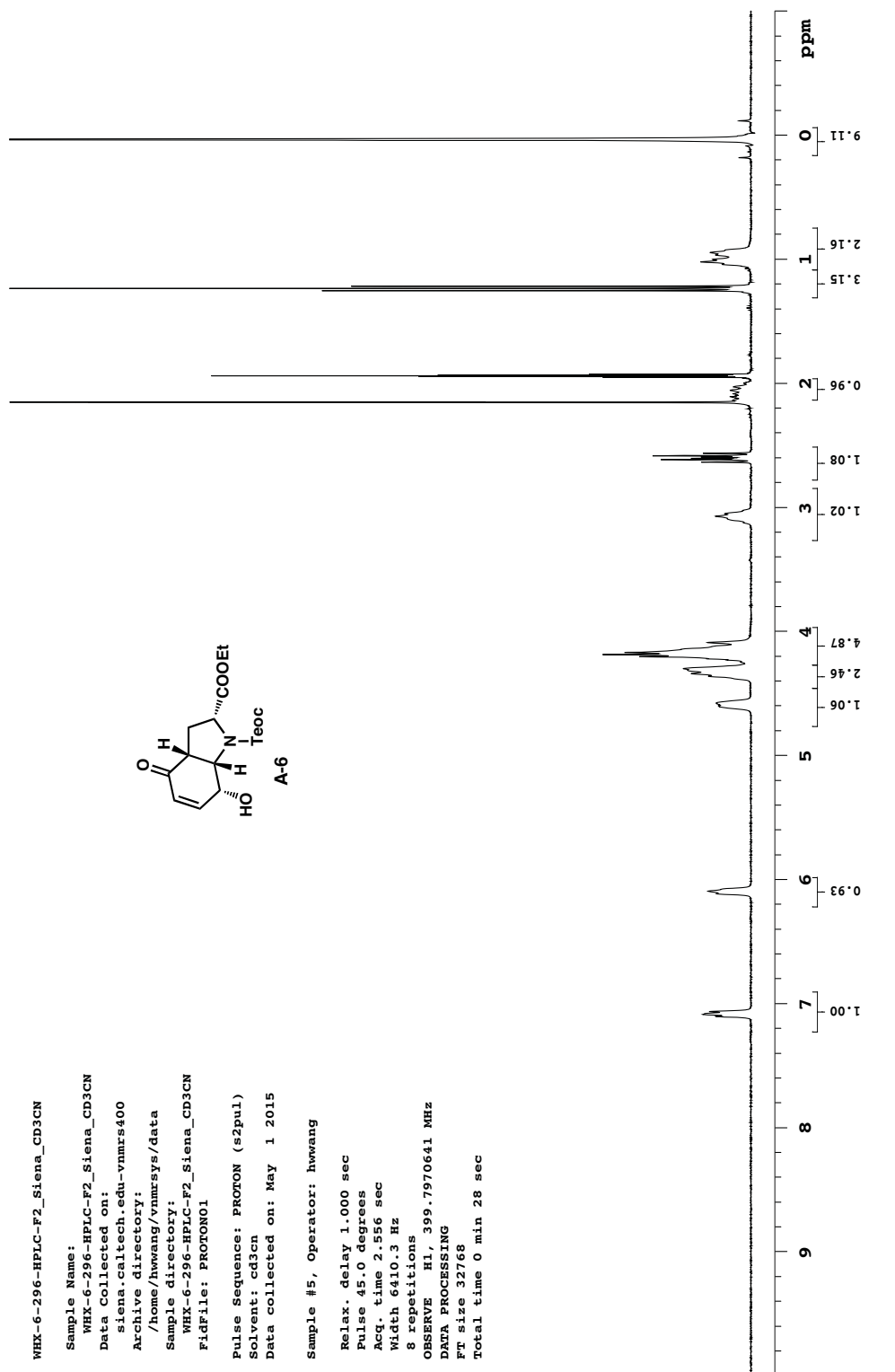
Total time 2 min 1 sec

346 (CD₃CN, 65 °C)









WHX-6-296-HPLC-F2_Siena_CD3CN-60C

Sample Name:

WHX-6-296-HPLC-F2_Siena_CD3CN-60C

Data Collected on:

siena.caltech.edu-vnmrs400

Archive directory:

/home/hhwang/vnmrsys/data

Sample directory:

WHX-6-296-HPLC-F2_Siena_CD3CN-60C

FidFile: WHX-6-296-HPLC-F2-Siena-CD3CN-60C-re

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn

Data collected on: May 1 2015

Temp. 60.0 C / 333.1 K

Operator: hhwang

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

16 repetitions

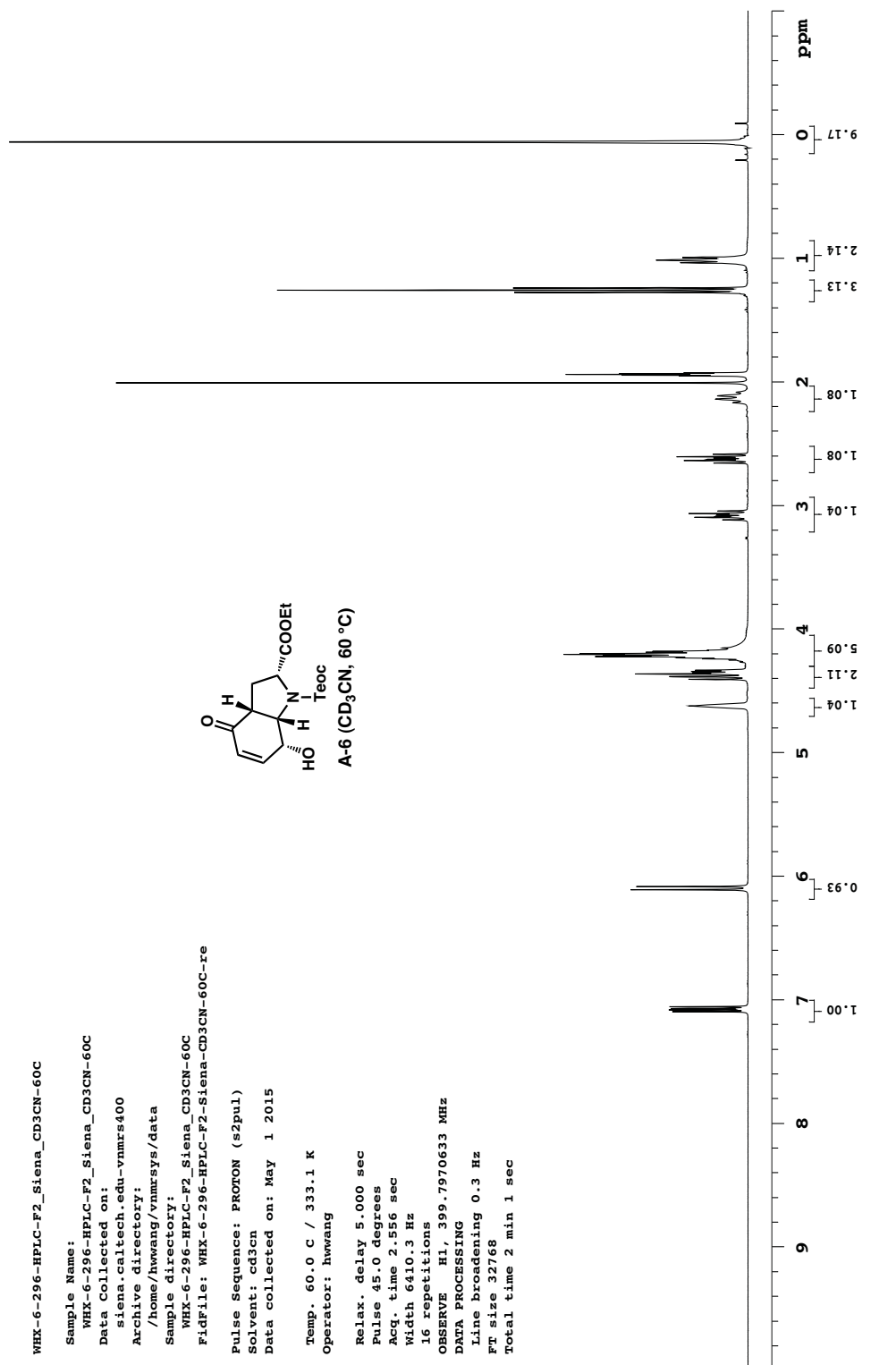
OBSERVE H1, 399.7970633 MHz

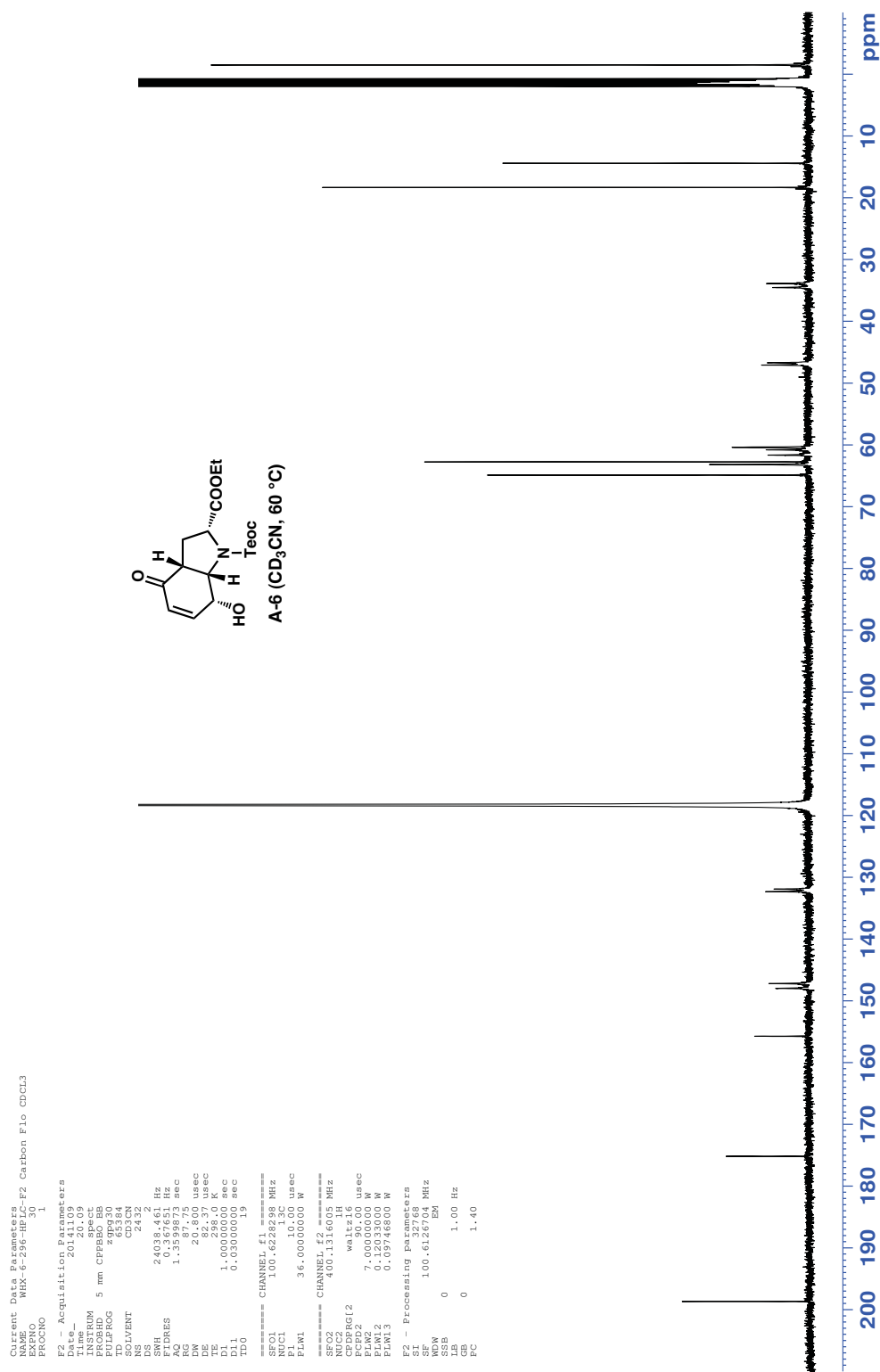
DATA PROCESSING

Line broadening 0.3 Hz

FT size 32768

Total time 2 min 1 sec





WHX-7-136-F_Siena_CD3CN

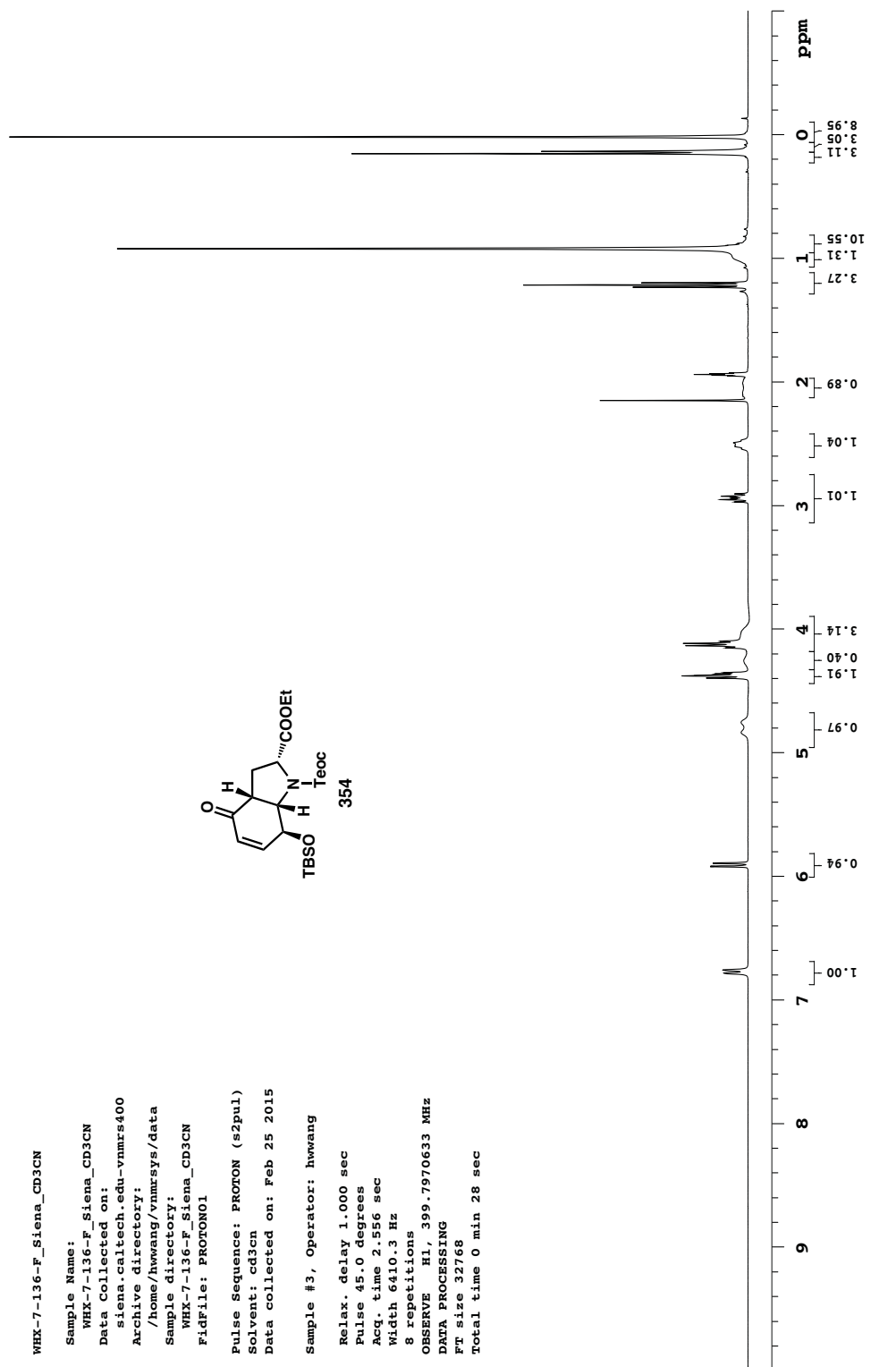
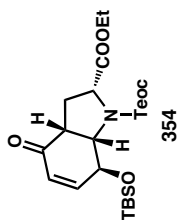
Sample Name:
 WHX-7-136-F_Siena_CD3CN
 Data Collected on:
 siena.caltech.edu-vnmrs400
 Archive directory:
 /home/hwang/vnmrsys/data
 Sample directory:
 WHX-7-136-F_Siena_CD3CN
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cd3cn
 Data collected on: Feb 25 2015

Sample #3, Operator: hwang

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.556 sec
 Width 6410.3 Hz
 8 repetitions

OBSERVE R1, 399.7970633 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min 28 sec



WHX-7-136-F-60C_Siena_CD3CN

Sample Name:

WHX-7-136-F-60C_Siena_CD3CN
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-136-F-60C_Siena_CD3CN
FidFile: PROTON01

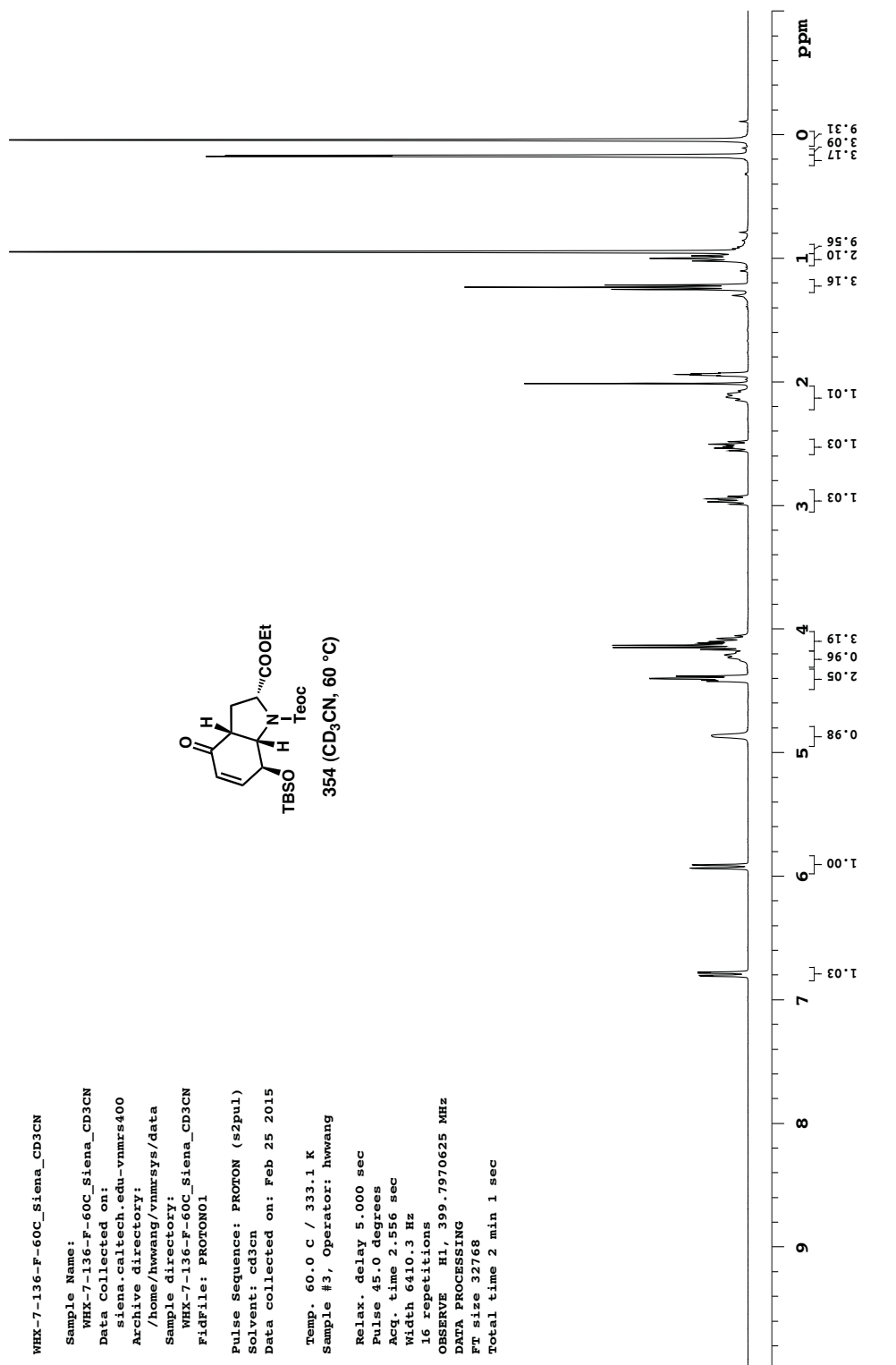
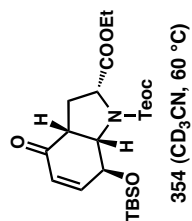
Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: Feb 25 2015

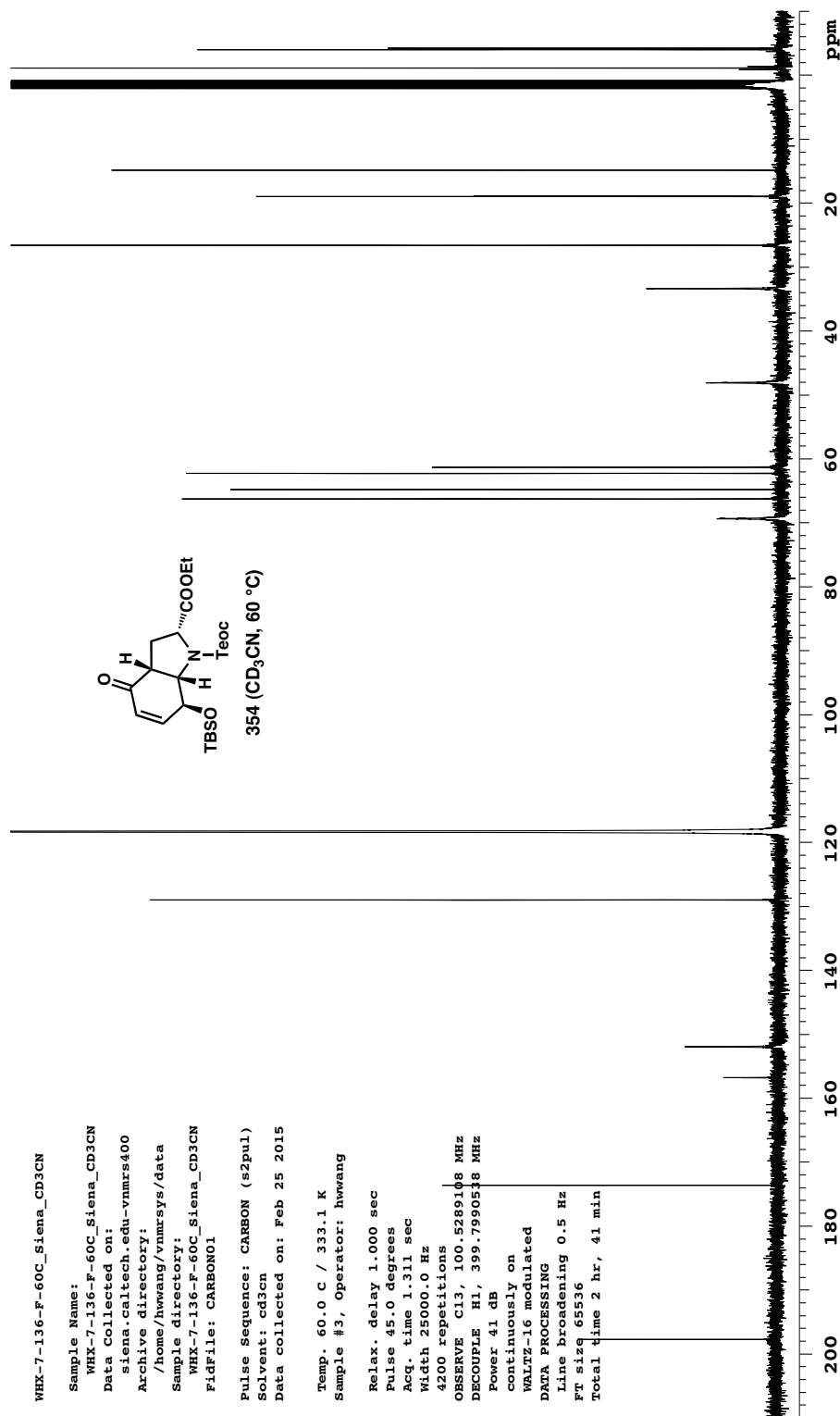
Temp. 60.0 C / 333.1 K

Sample #3, Operator: hwang

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions

OBSERVE H1, 399.7970625 MHz
DATA PROCESSING
FT size 32768
Total time 2 min 1 sec





WHX-7-171-F1_Siena_CD3CN

Sample Name:

WHX-7-171-F1_Siena_CD3CN

Data Collected on:

siena.caltech.edu-vnmrs400

Archive directory:

/home/hwang/vnmrsys/data

Sample directory:

WHX-7-171-F1_Siena_CD3CN

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn

Data collected on: May 6 2015

Temp. 25.0 C / 298.1 K

Sample #6, Operator: hwang

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

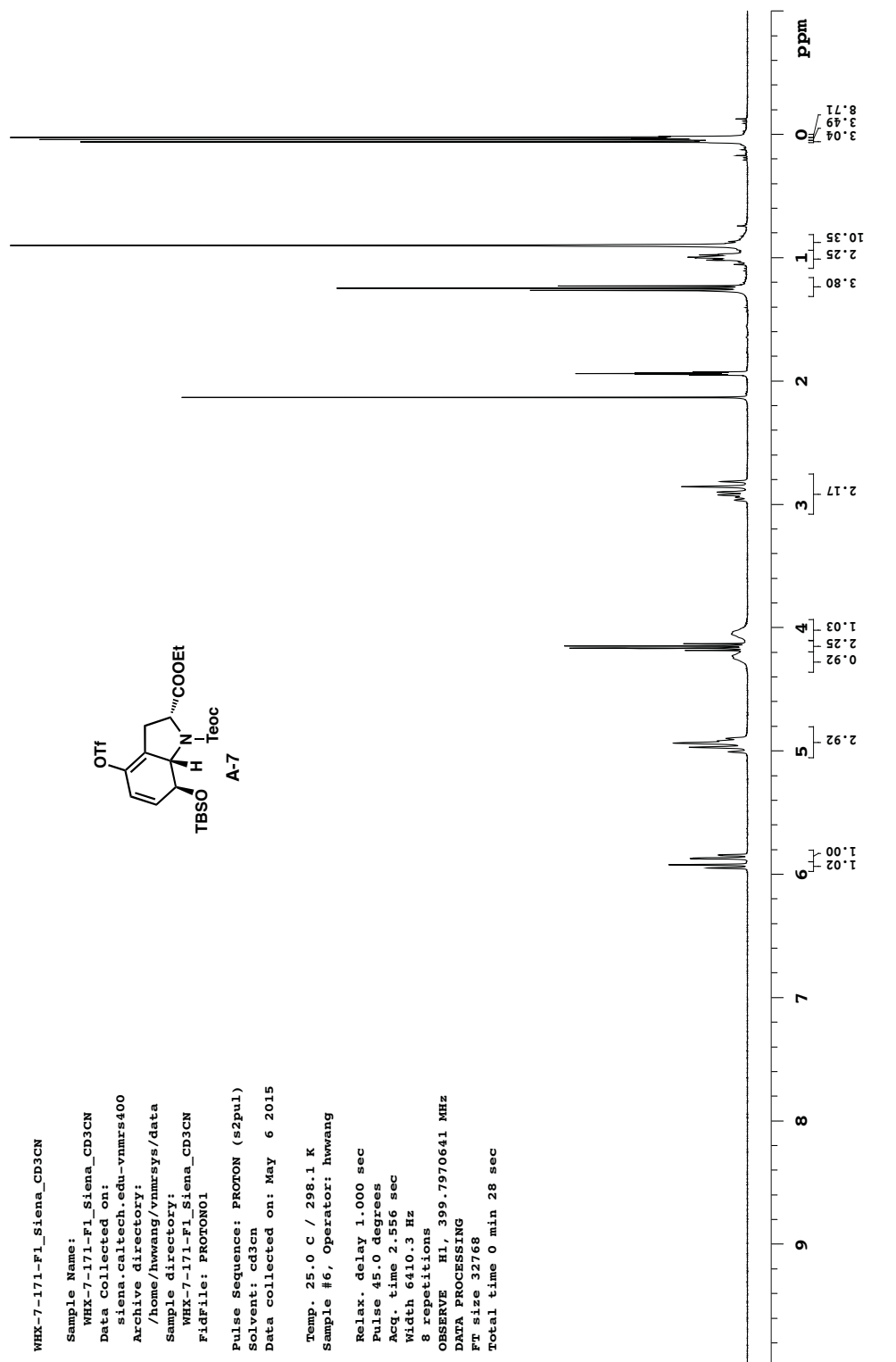
8 repetitions

OBSERVE H1, 399.7970641 MHz

DATA PROCESSING

FT size 32768

Total time 0 min 28 sec



WHX-7-171-F1_Siena_CD3CN-60C

Sample Name:

WHX-7-171-F1_Siena_CD3CN-60C
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-171-F1_Siena_CD3CN-60C
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: May 6 2015

Temp. 60.0 C / 333.1 K

Sample #6, Operator: hwang

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

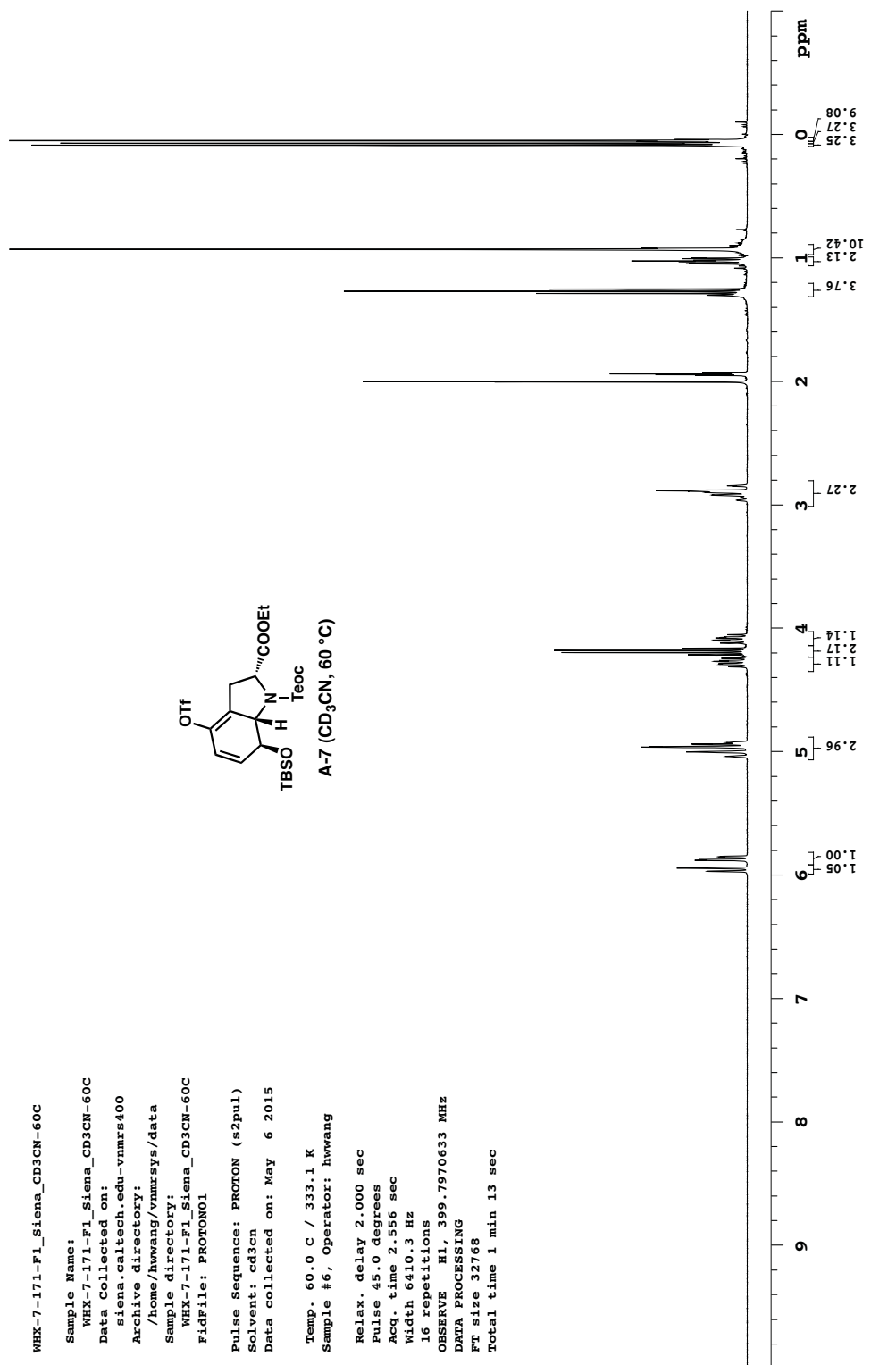
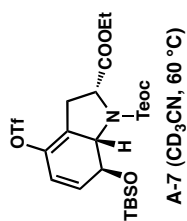
16 repetitions

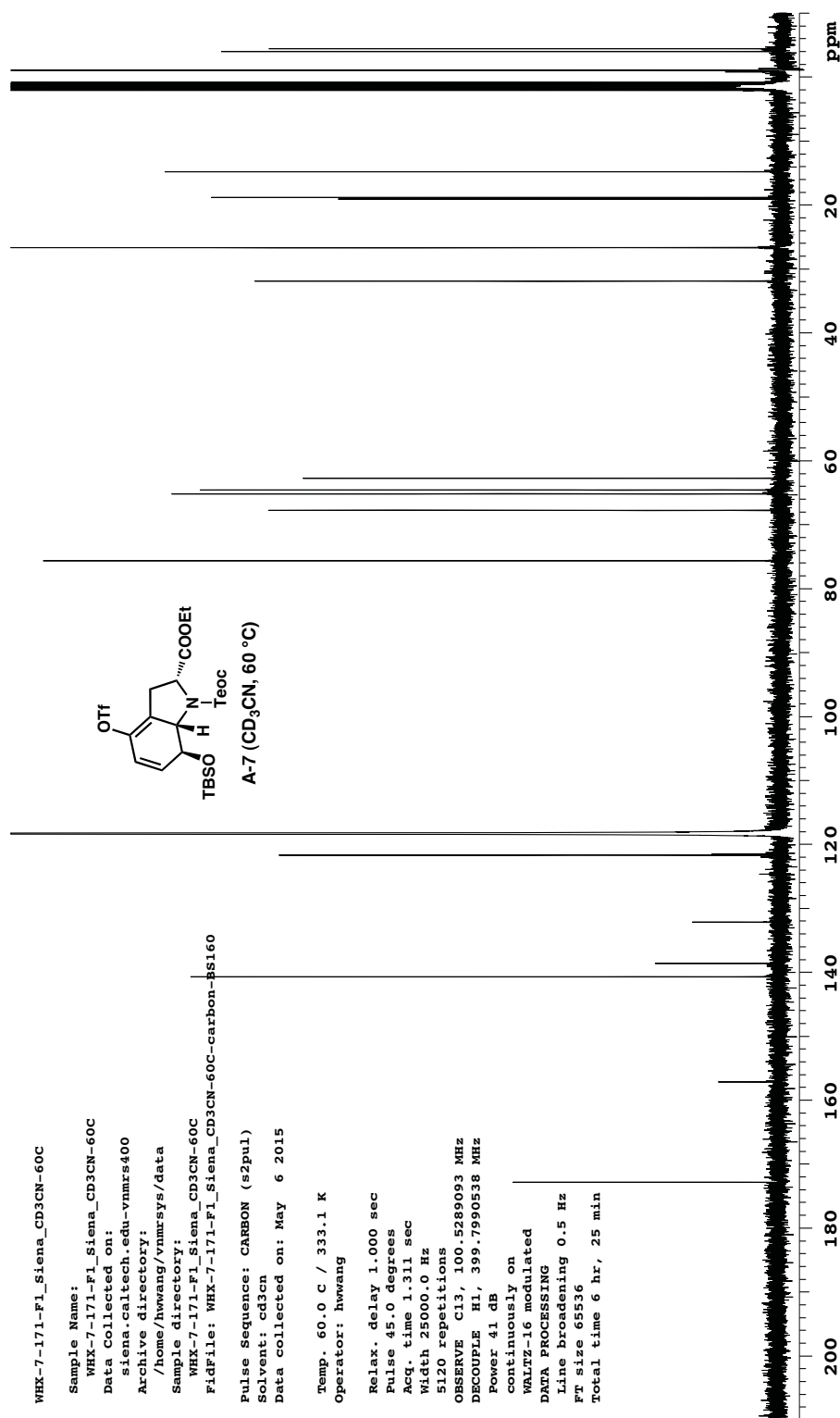
OBSERVE H1, 399.7970633 MHz

DATA PROCESSING

FT size 32768

Total time 1 min 13 sec





WHX-7-172-F_Siena_CD3CN

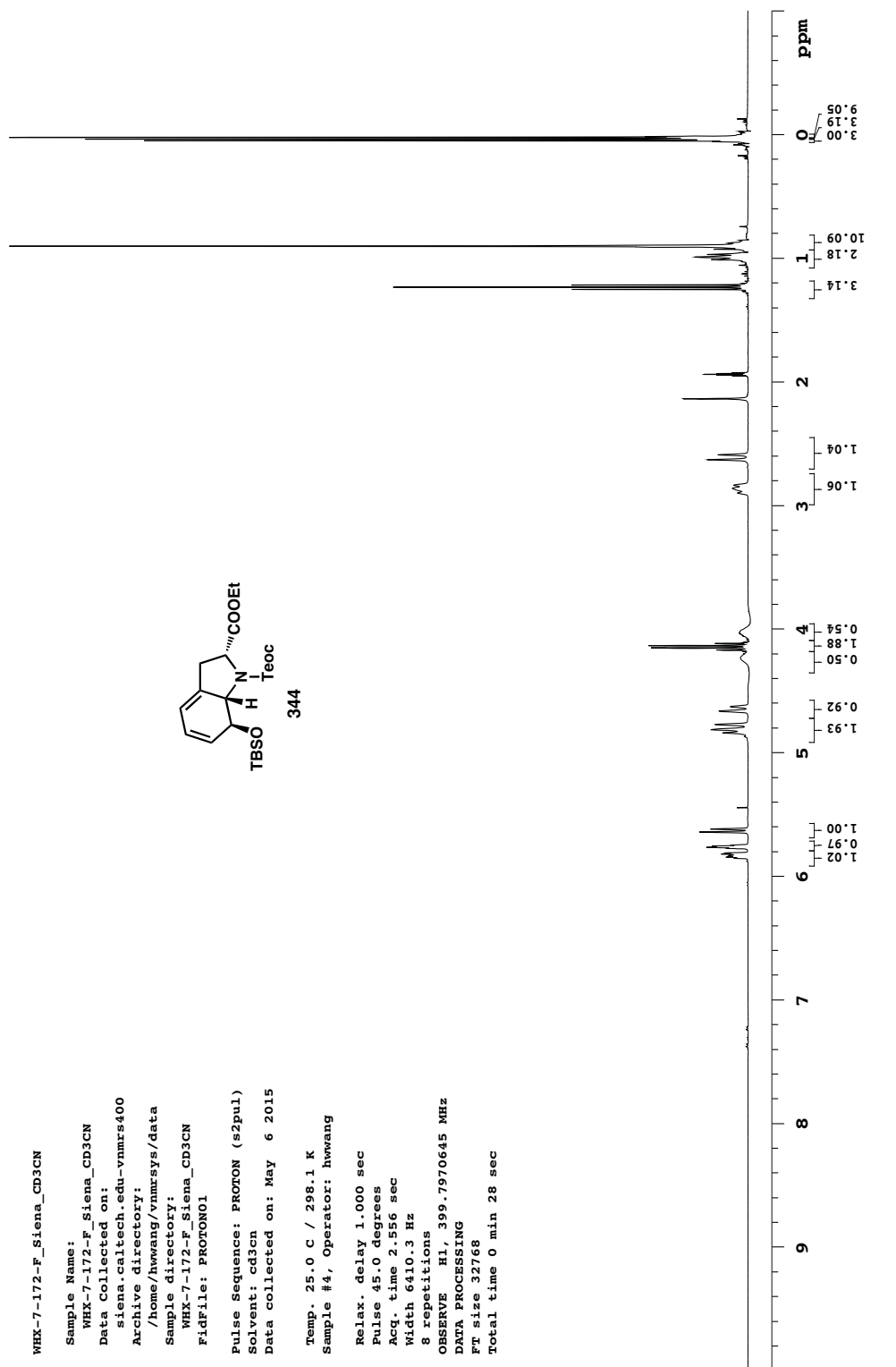
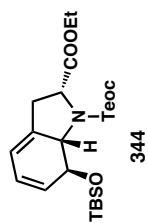
Sample Name:
WHX-7-172-F_Siena_CD3CN
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-172-F_Siena_CD3CN
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: May 6 2015

Temp. 25.0 C / 298.1 K
Sample #4, Operator: hwang

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions

OBSERVE H1, 399.7970645 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec



WHX-7-172-F_Siena_CD3CN-60C

Sample Name:

WHX-7-172-F_Siena_CD3CN-60C
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hhwang/vnmrsys/data

Sample directory:

WHX-7-172-F_Siena_CD3CN-60C
FidFile: WHX-7-172-F-Siena-CD3CN-60C-proton

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn
Data collected on: May 6 2015

Temp. 60.0 C / 333.1 K

Operator: hhwang

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

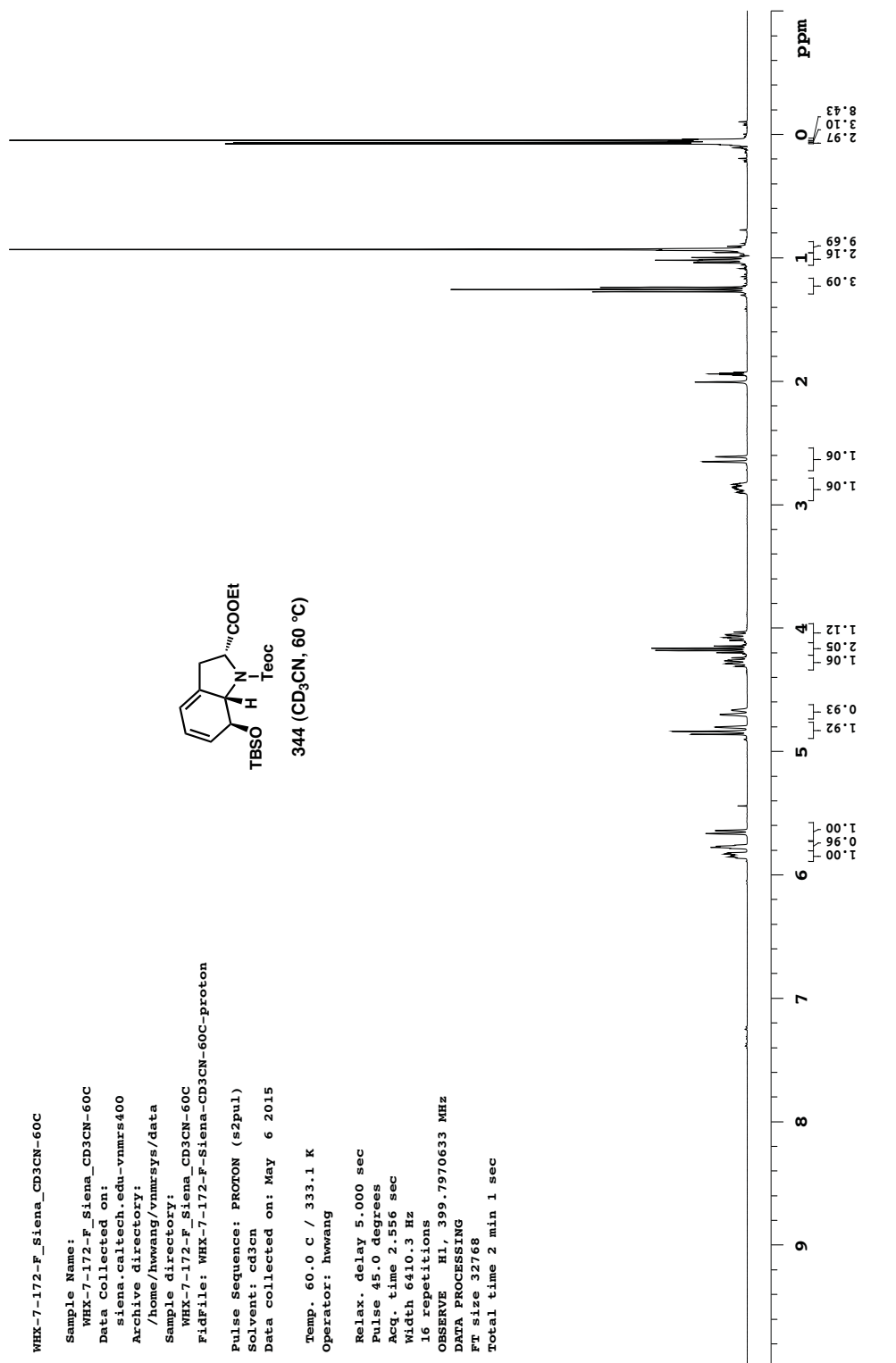
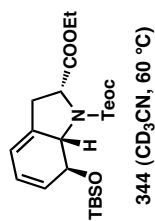
16 repetitions

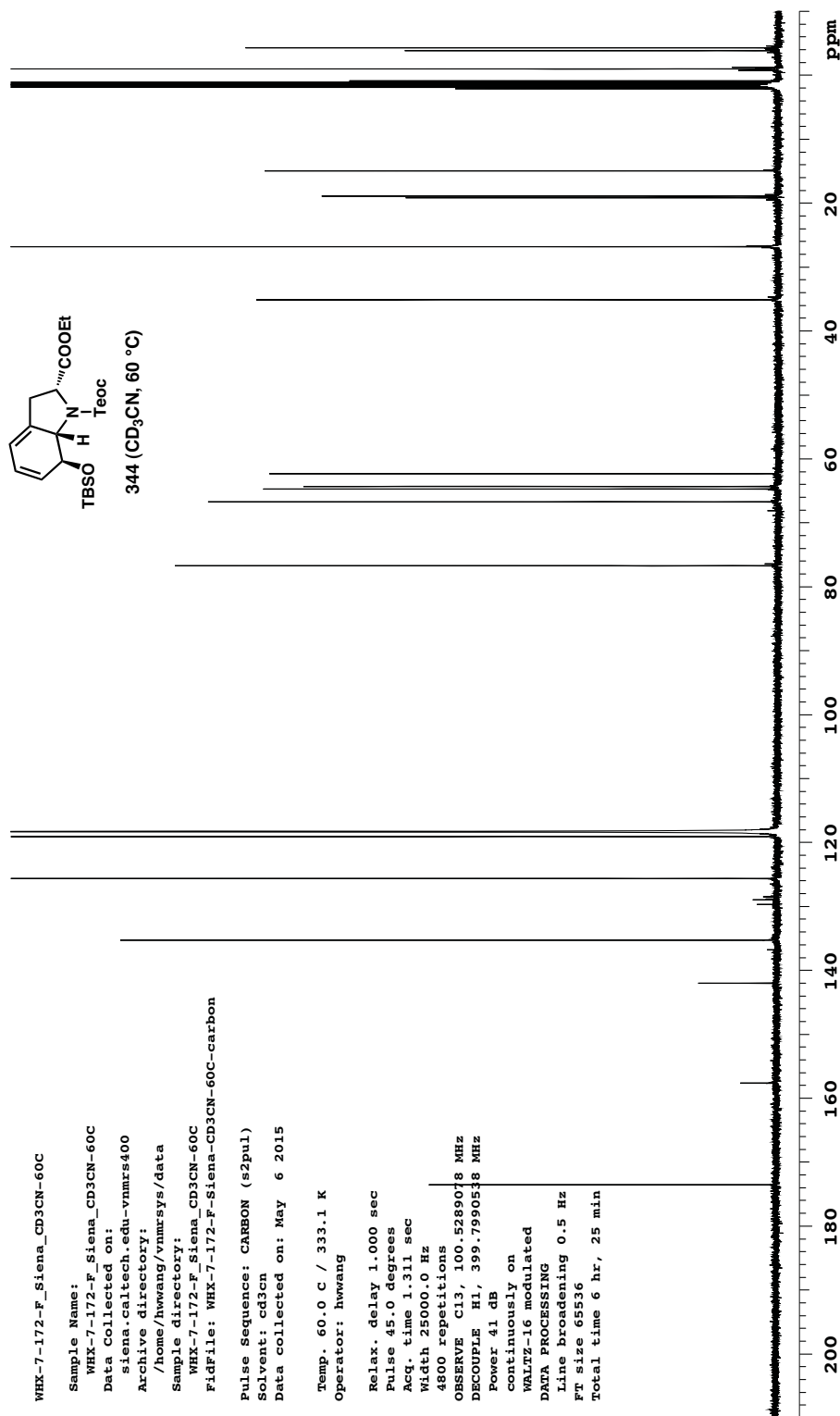
OBSERVE H1, 399.7970633 MHz

DATA PROCESSING

FT size 32768

Total time 2 min 1 sec





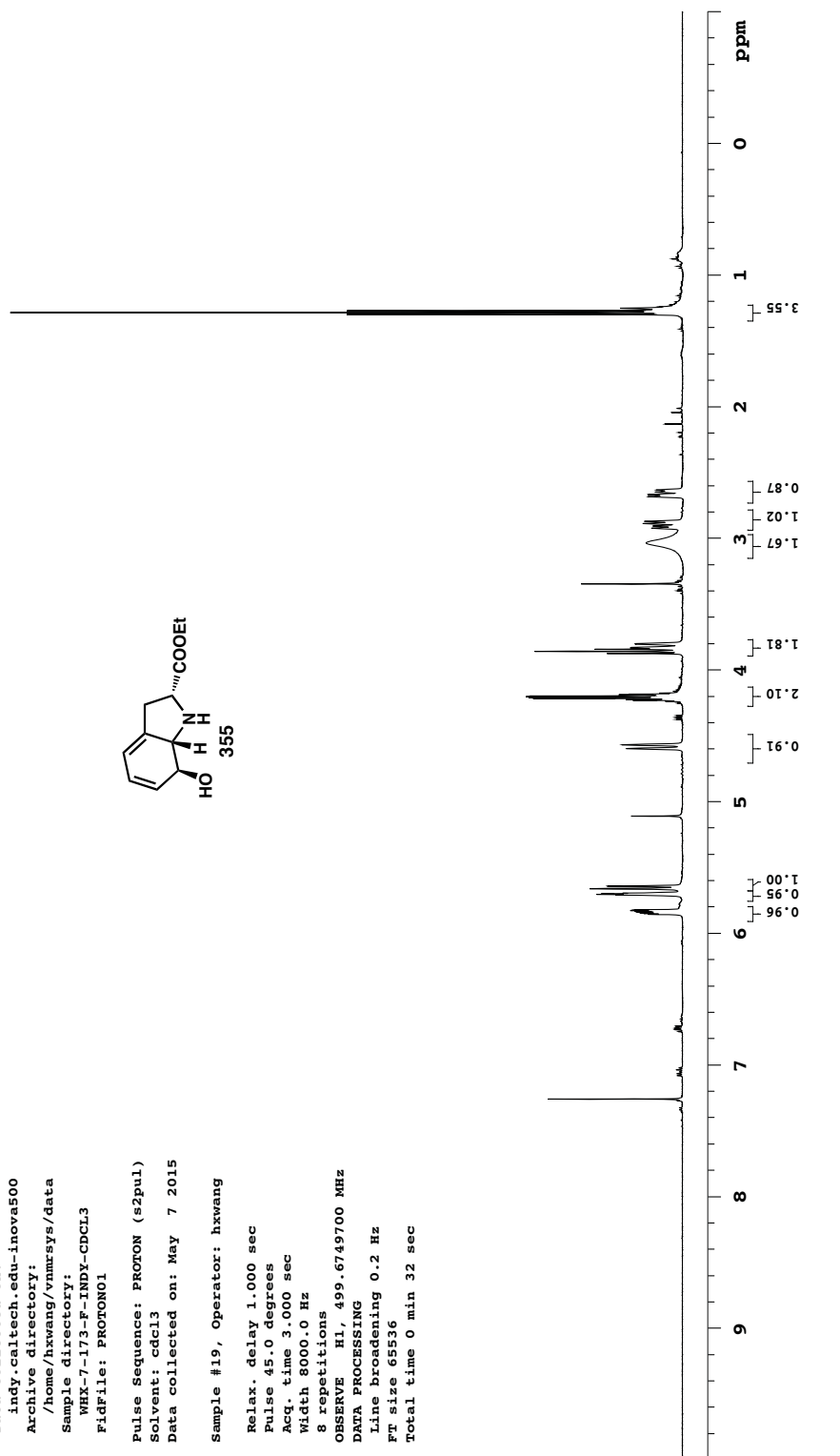
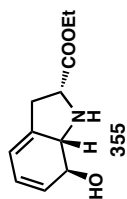
WHX-7-173-F-INDY-CDCL3

```
Sample Name: WHX-7-173-F-INDY-CDCL3
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/hxwang/vnmrsys/data
Sample directory: WHX-7-173-F-INDY-CDCL3
Fidfile: PROTON01
```

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 7 2015

Sample #19, Operator: hxxwang

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.6749700 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FFT size 65536
Total time 0 min 32 sec



WHX-7-173-F-INDY-CDCL3

Sample Name:

WHX-7-173-F-INDY-CDCL3

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/hxwang/vnmrsys/data

Sample directory:

WHX-7-173-F-INDY-CDCL3

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: May 7 2015

Sample #20, Operator: hxwang

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1500 repetitions

OBSERVE C13, 125.6433729 MHz

DECOUPLE H1, 499.677469 MHz

Power 36 dB

continuously on

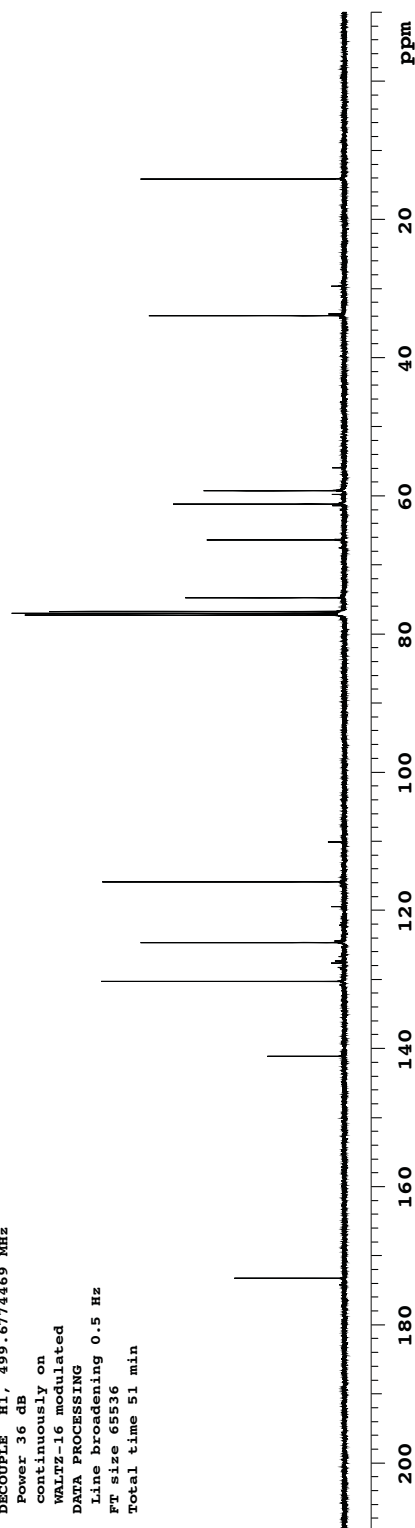
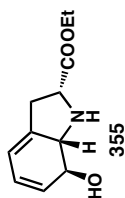
WALTZ-16 modulated

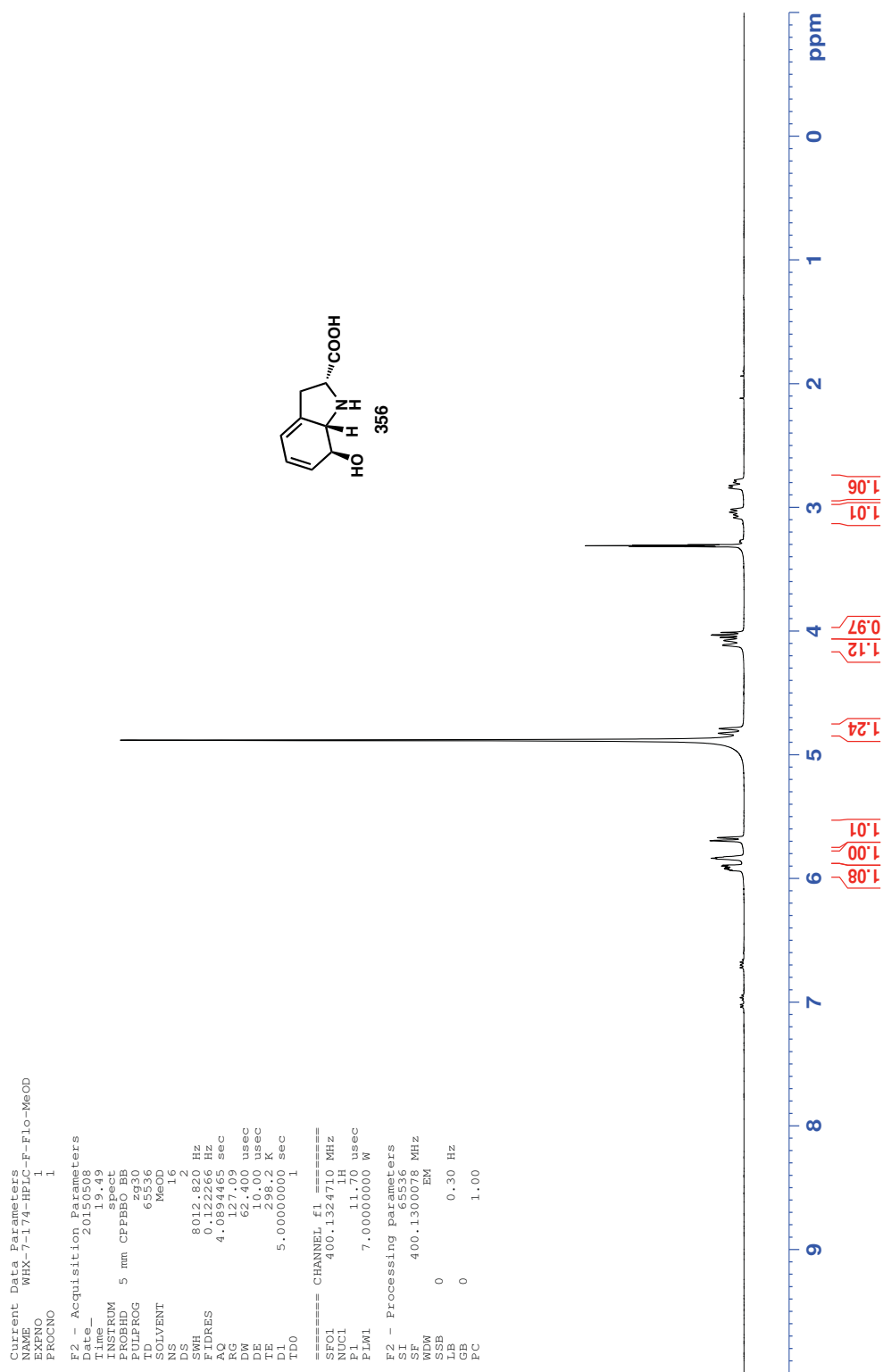
DATA PROCESSING

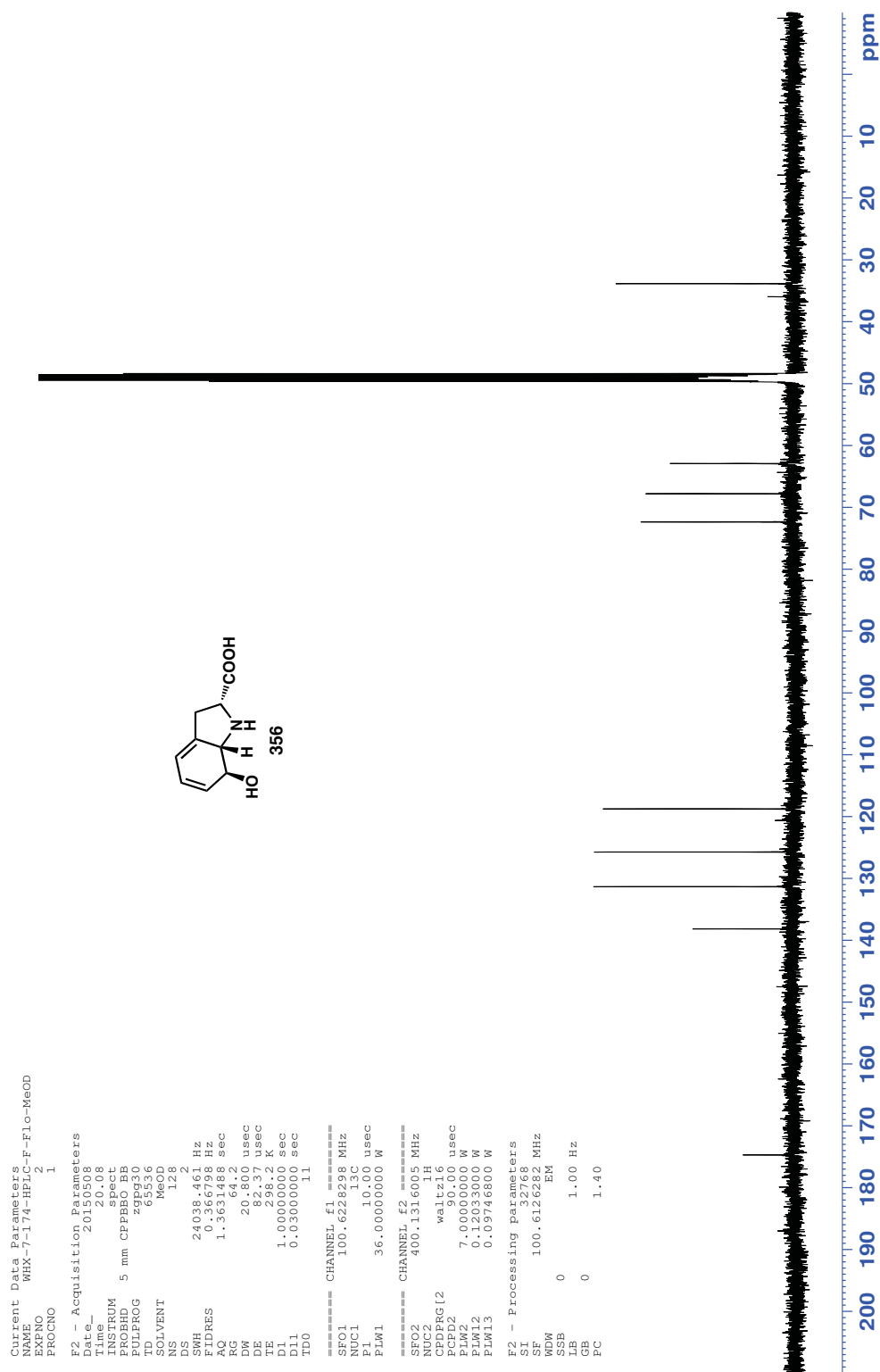
Line broadening 0.5 Hz

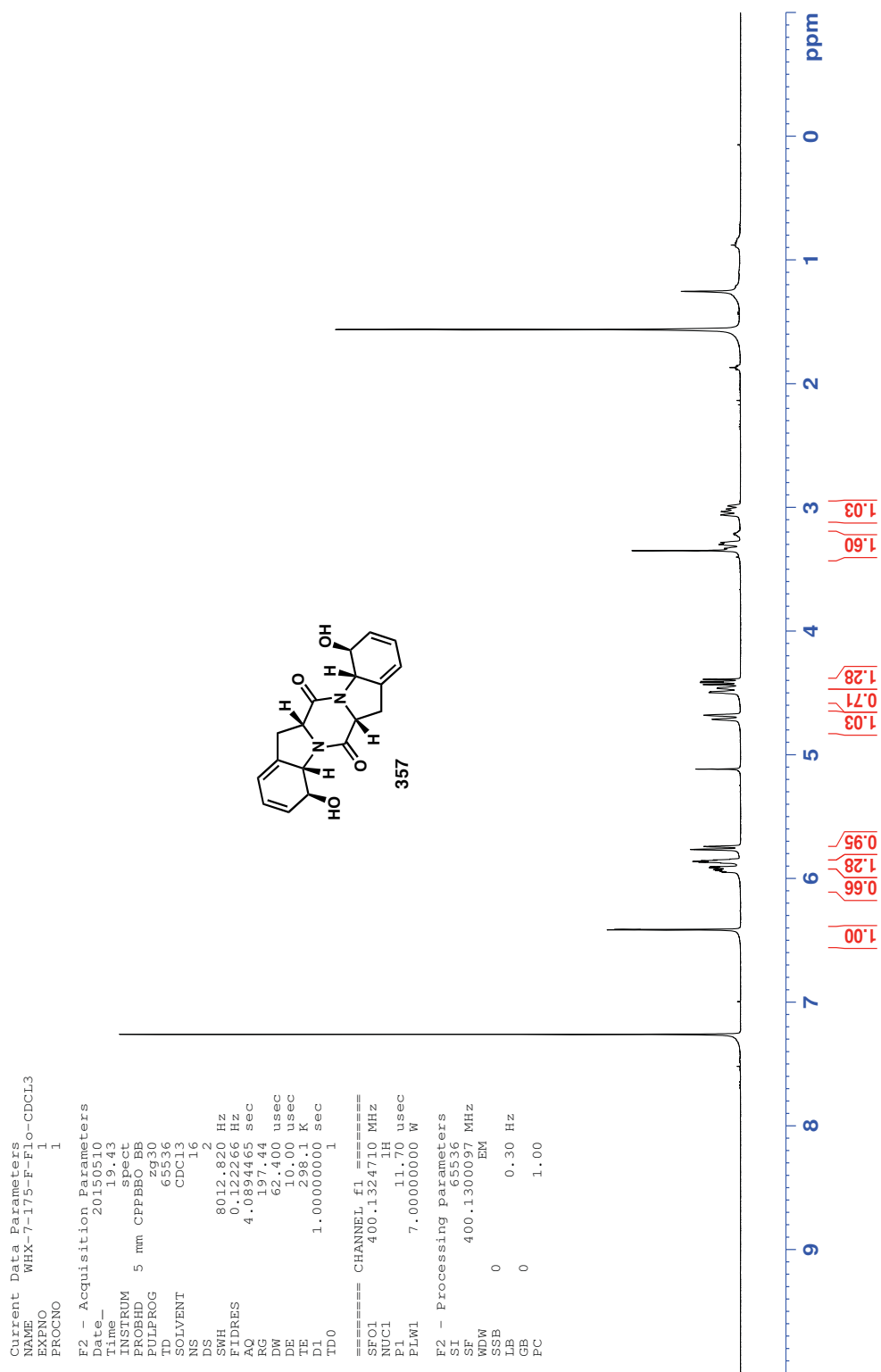
Ft size 65536

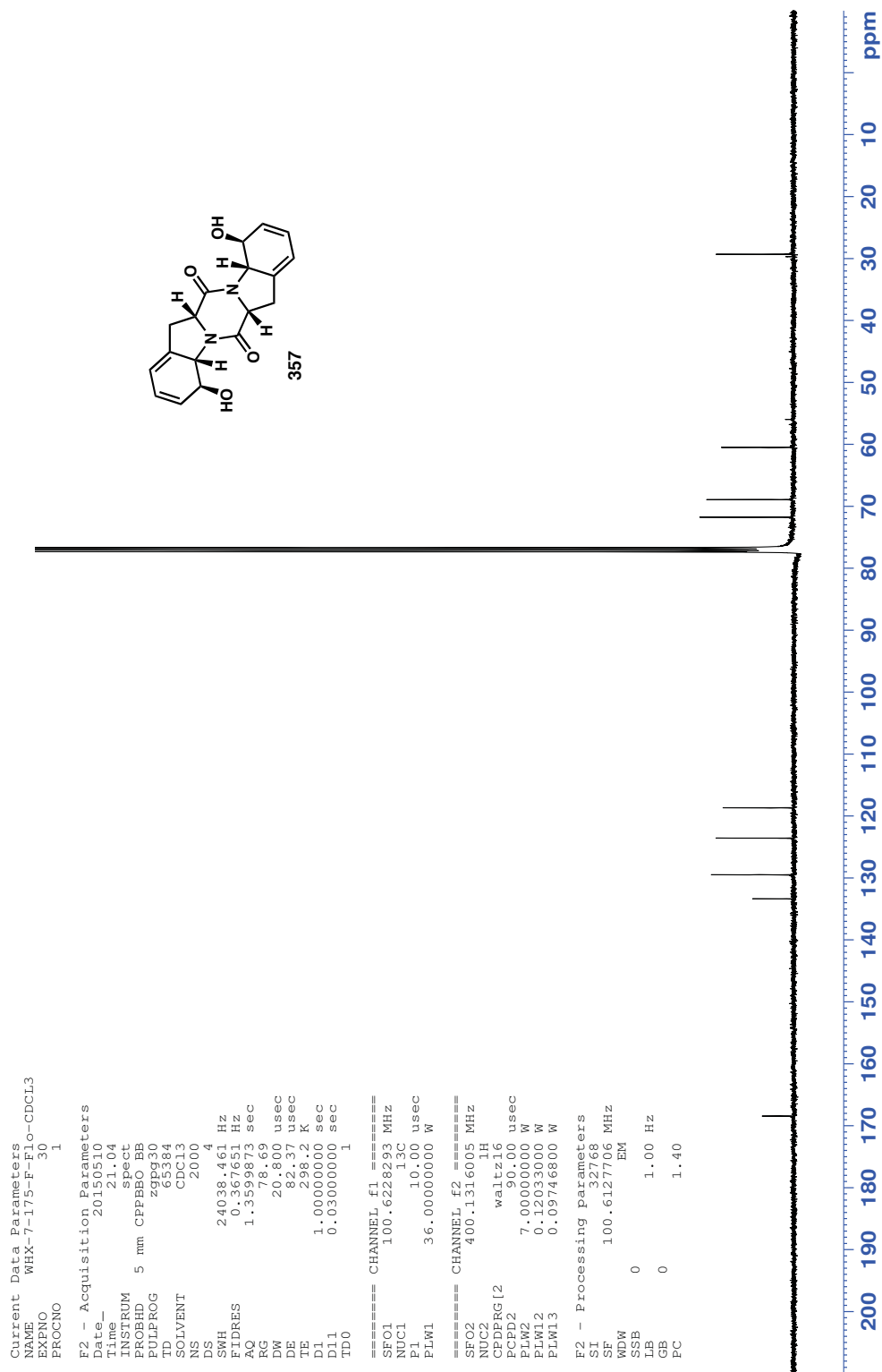
Total time 51 min

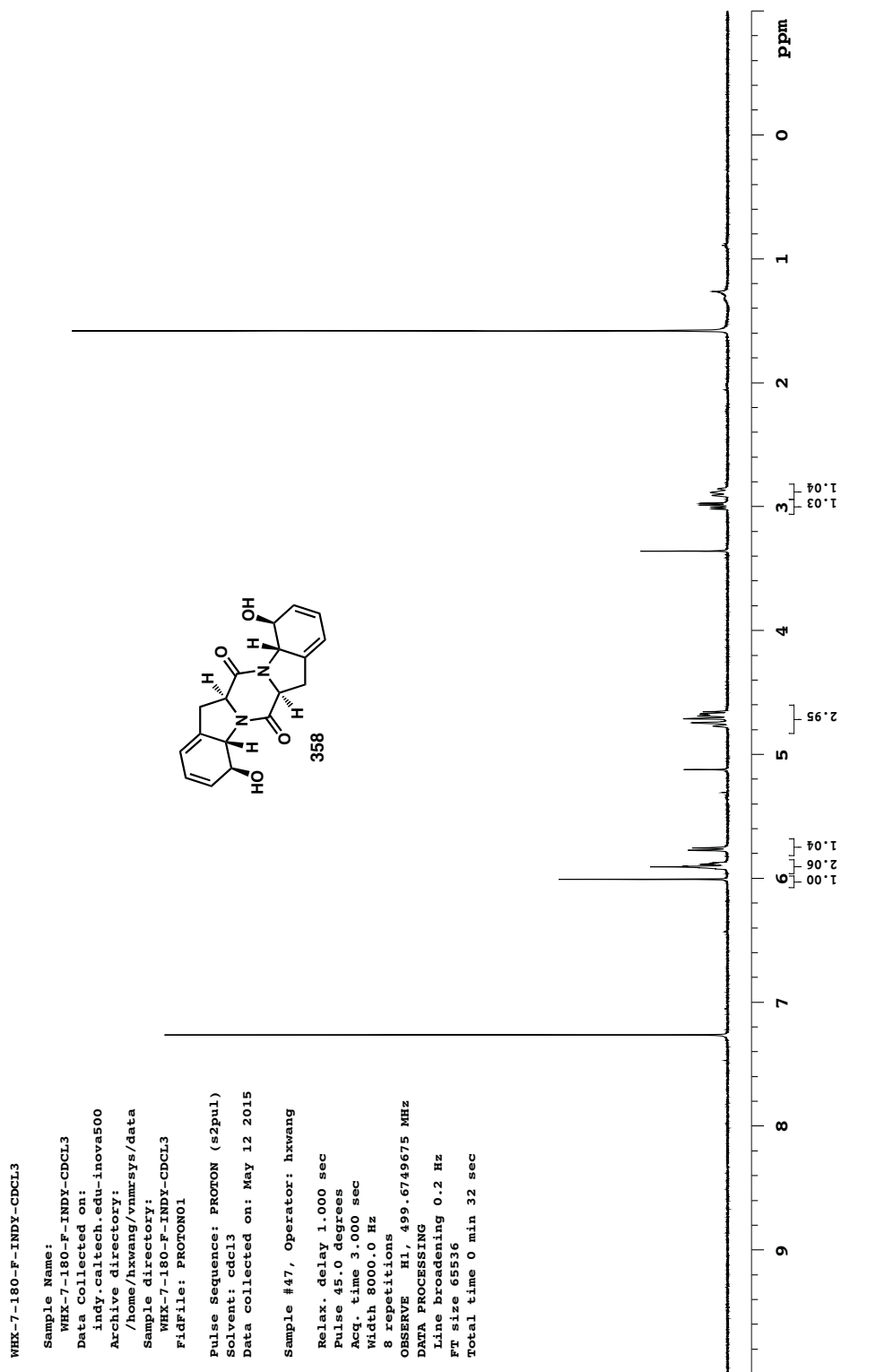


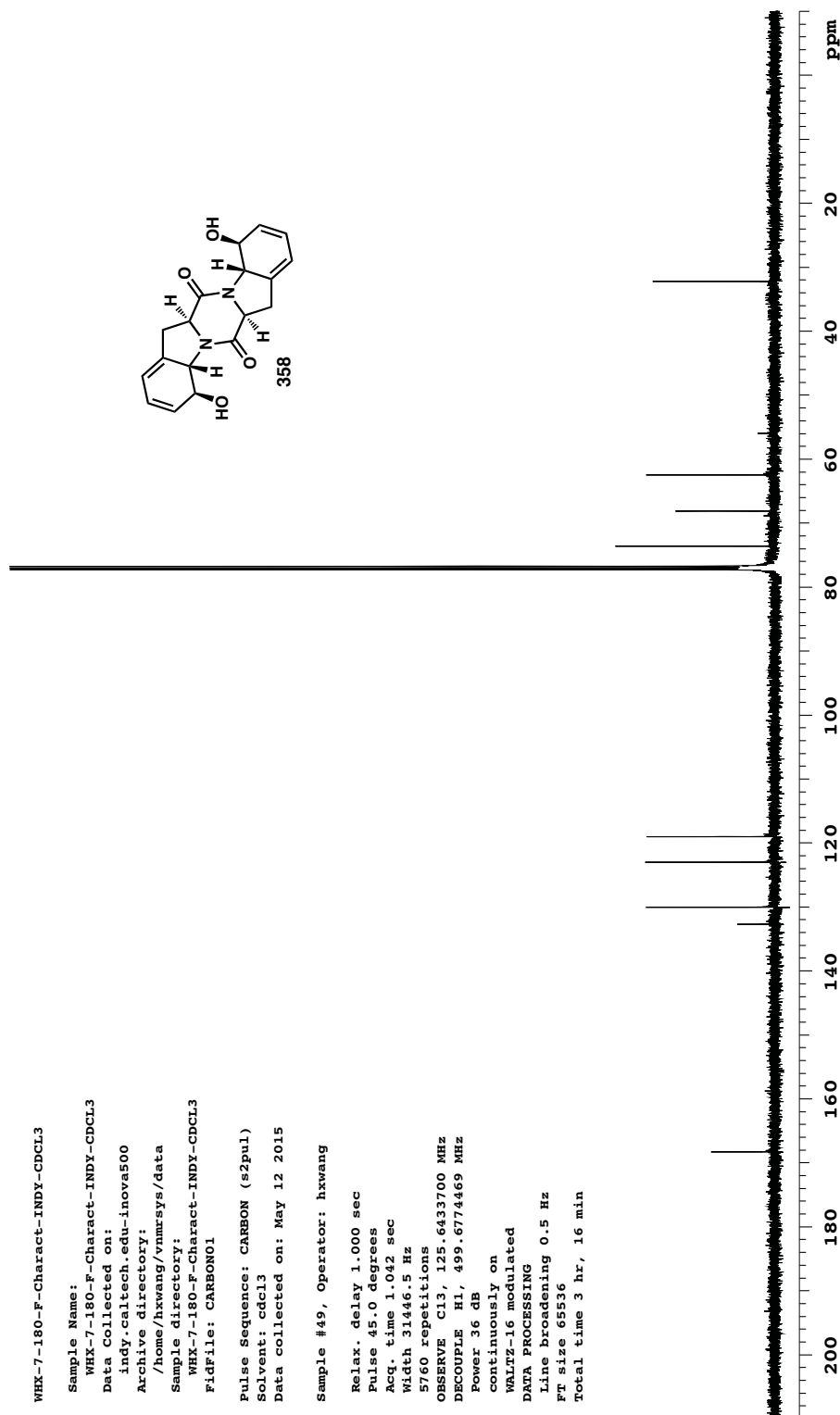












WHX-7-189-re-INDY-CDCL3

Sample Name:

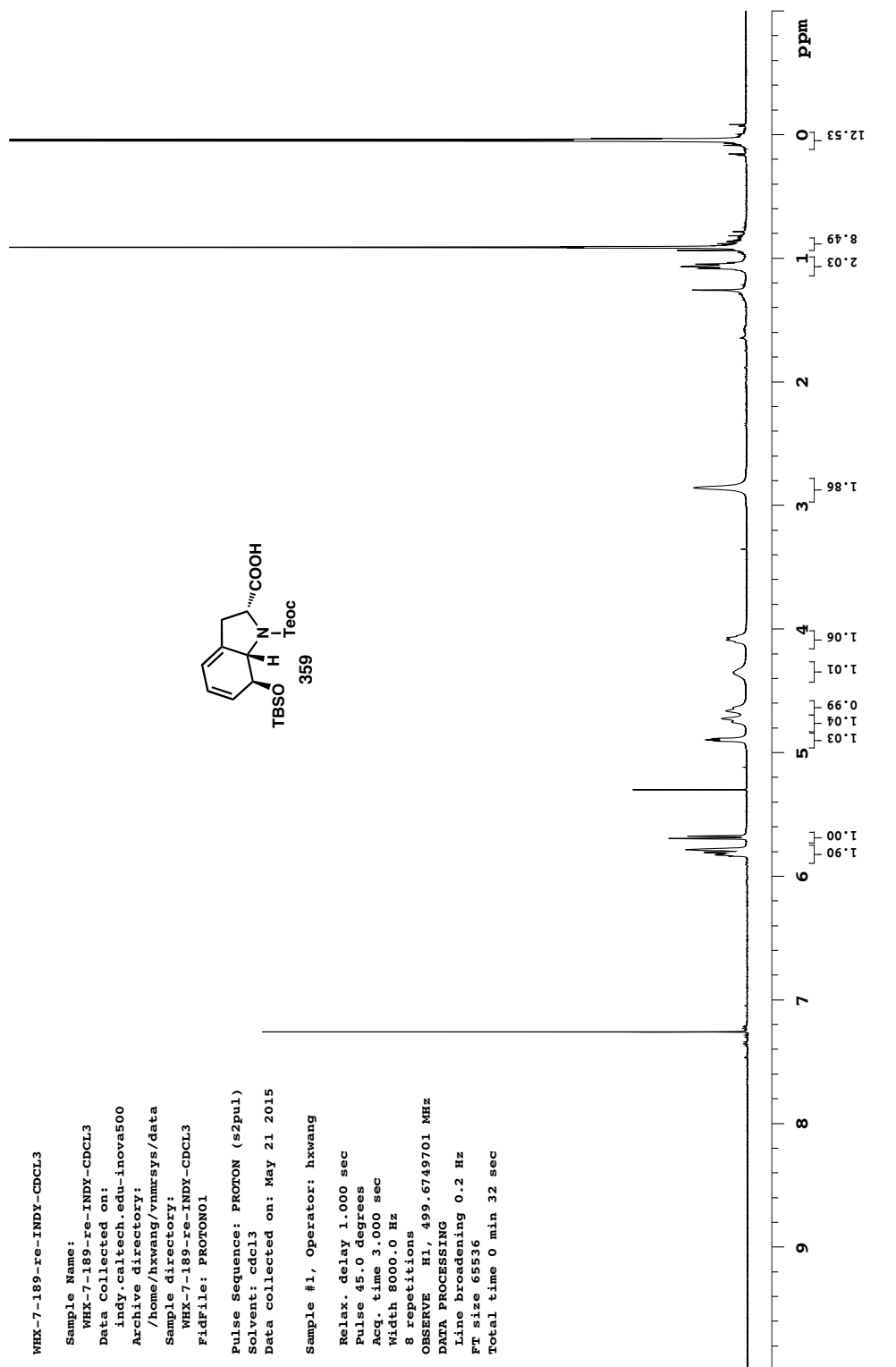
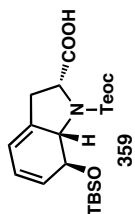
WHX-7-189-re-INDY-CDCL3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/hxwang/vnmrsys/data
Sample directory:
WHX-7-189-re-INDY-CDCL3
FidFile: PROTON01

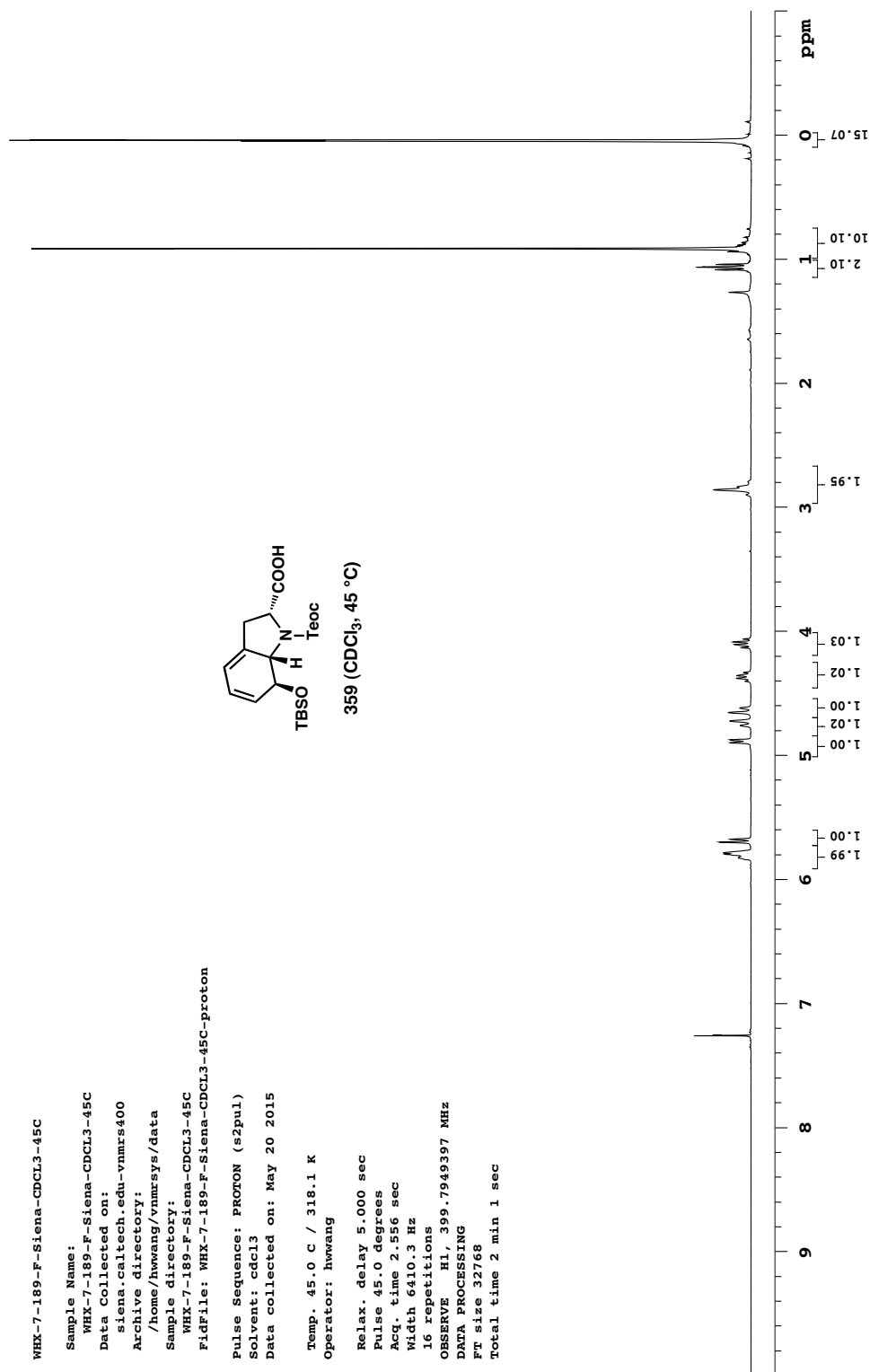
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 21 2015

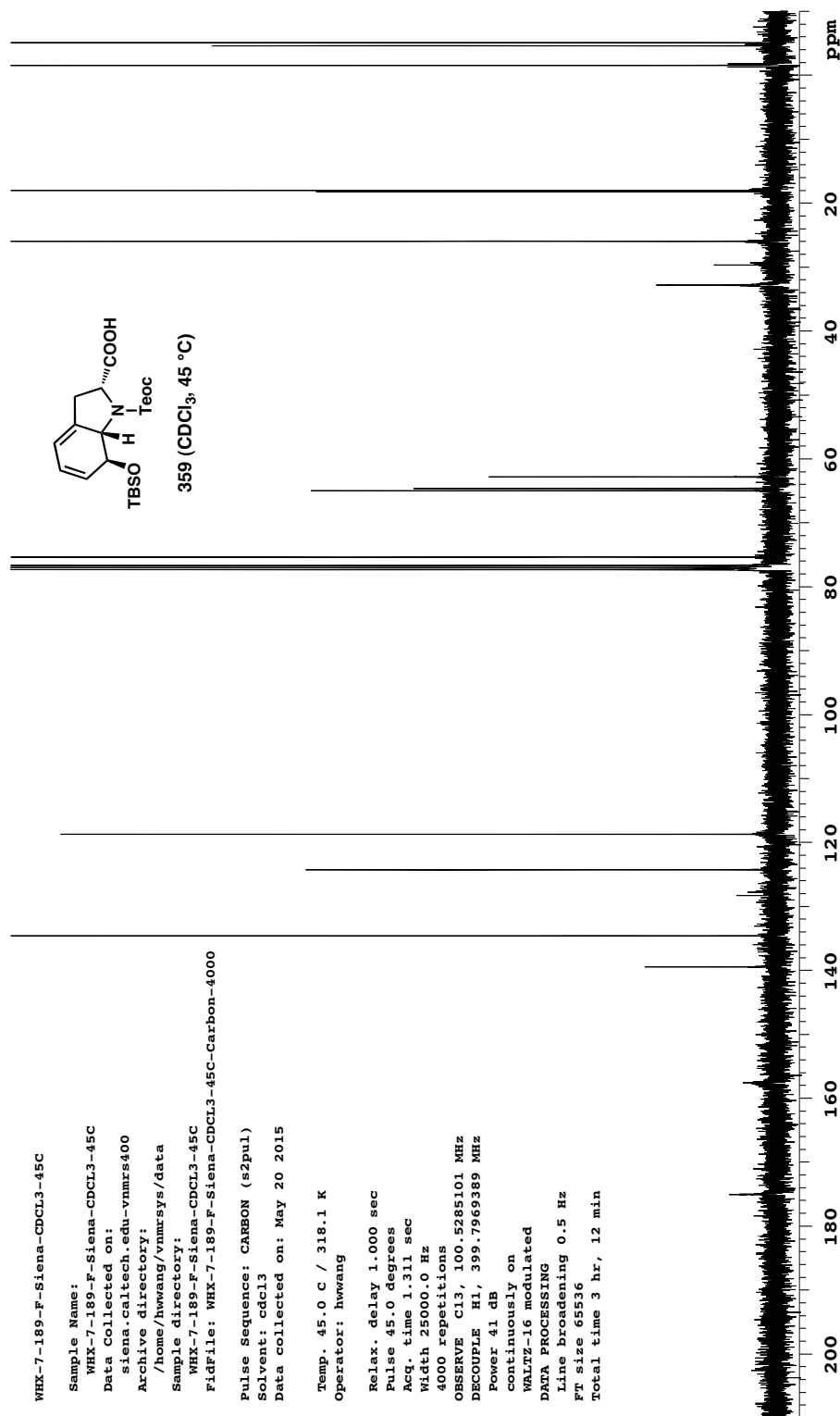
Sample #1, Operator: hxwang

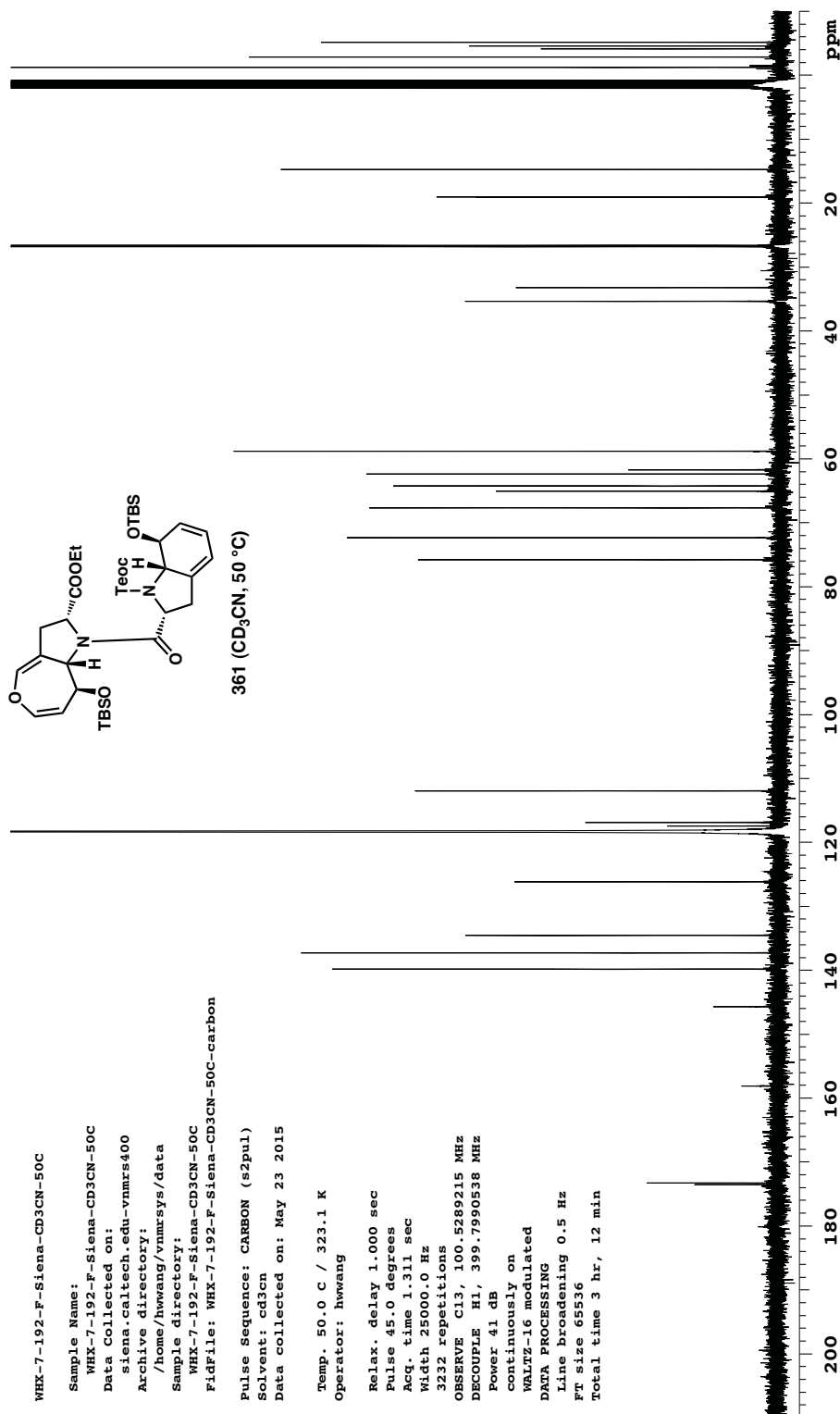
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
8 repetitions

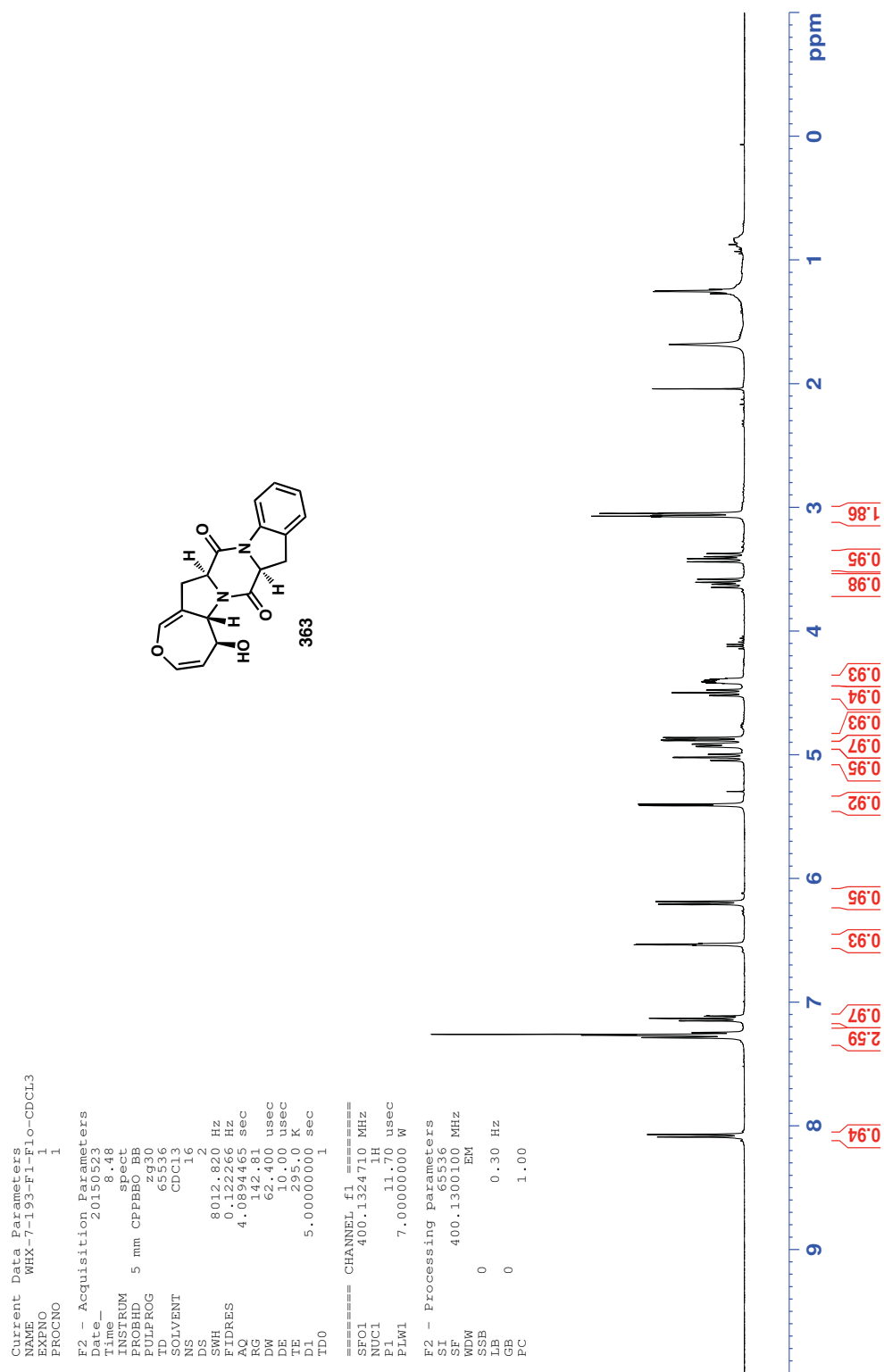
OBSERVE H1, 499.6749701 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 32 sec

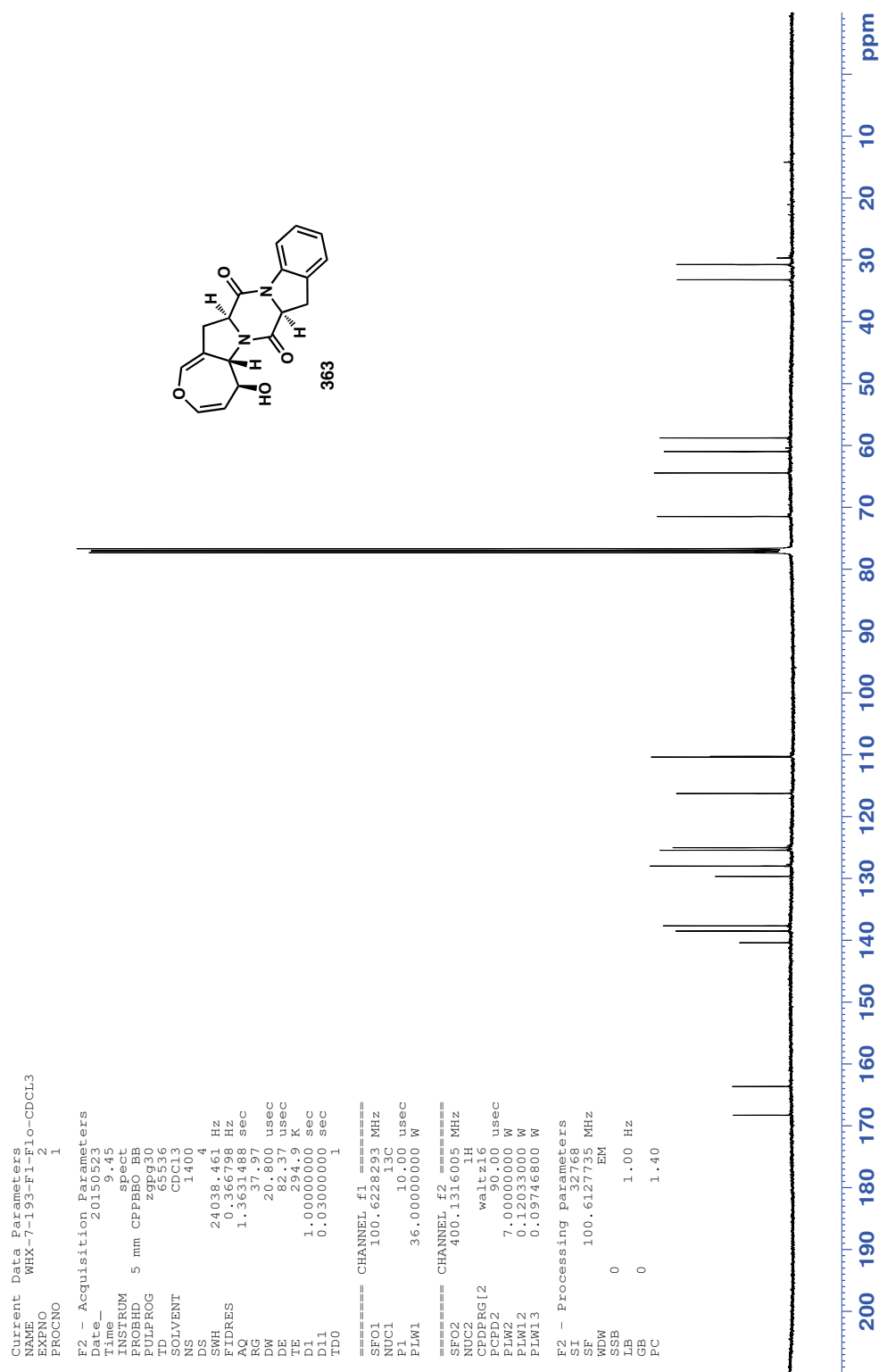












WHX-7-127-F_Siena_CDCL3

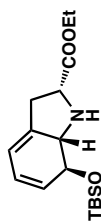
Sample Name:
WHX-7-127-F_Siena_CDCL3
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-127-F_Siena_CDCL3
FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 18 2015

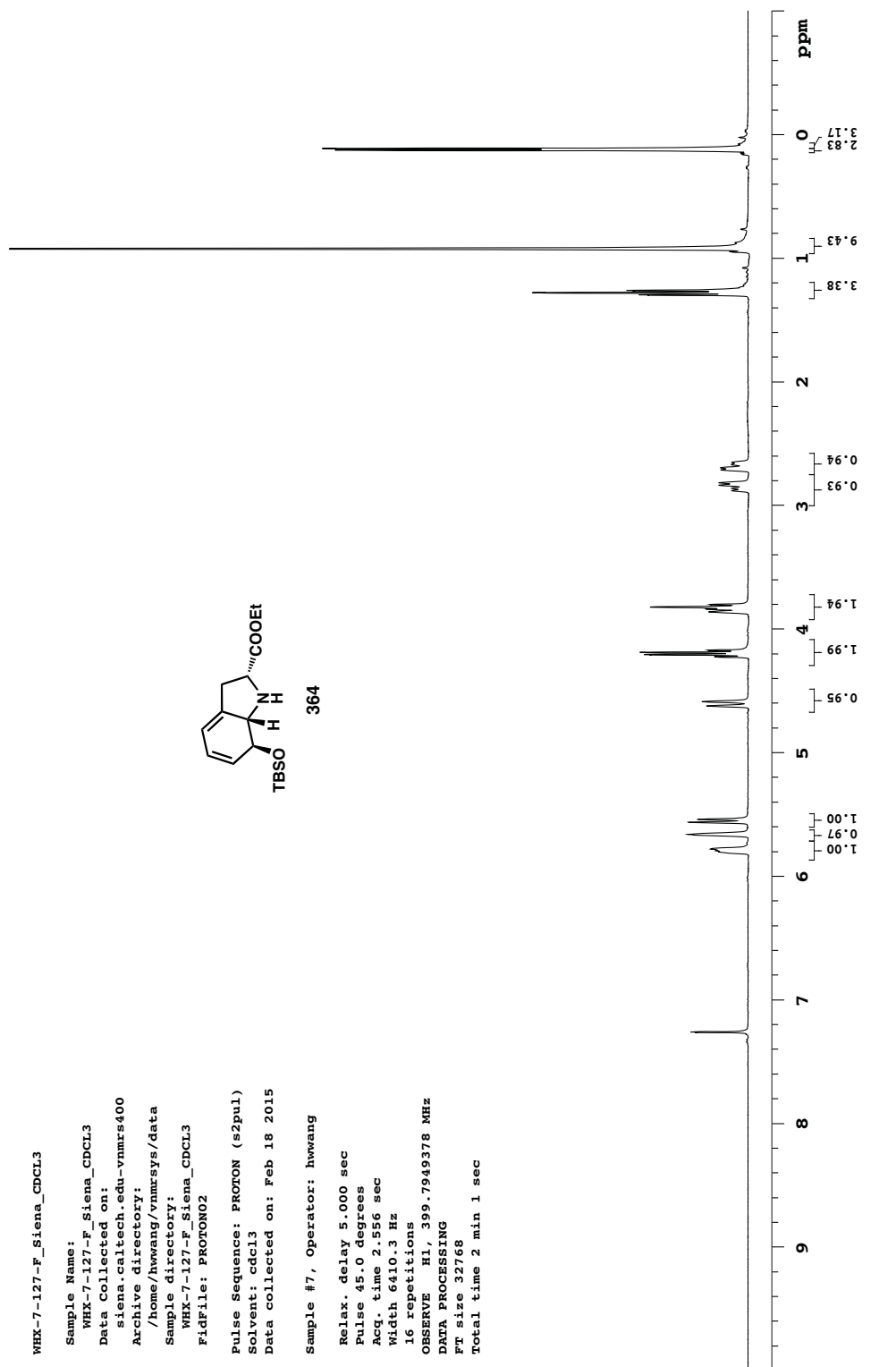
Sample #7, Operator: hwang

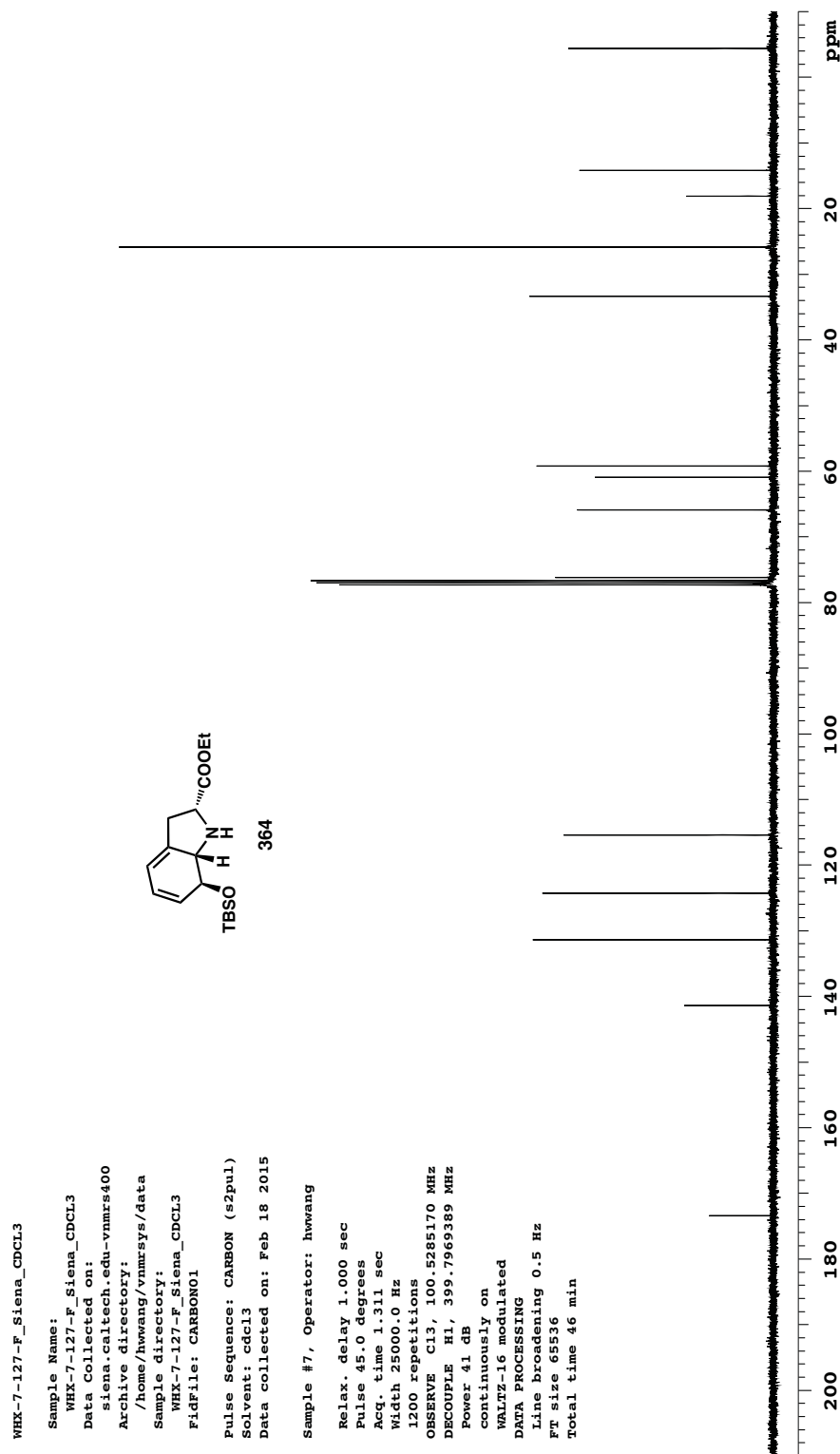
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions

OBSERVE H1, 399.7949378 MHz
DATA PROCESSING
Ft size 32768
Total time 2 min 1 sec



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WHX-7-132-F-FID-CD3CN

Sample Name:

WHX-7-132-F-FID-CD3CN
Data Collected on:
siena.caltech.edu-vnmrs400
Archive directory:
/home/hwang/vnmrsys/data
Sample directory:
WHX-7-132-F-FID-CD3CN
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cd3cn
Data collected on: May 8 2015

Temp. 25.0 C / 298.1 K

Sample #6, Operator: hwang

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

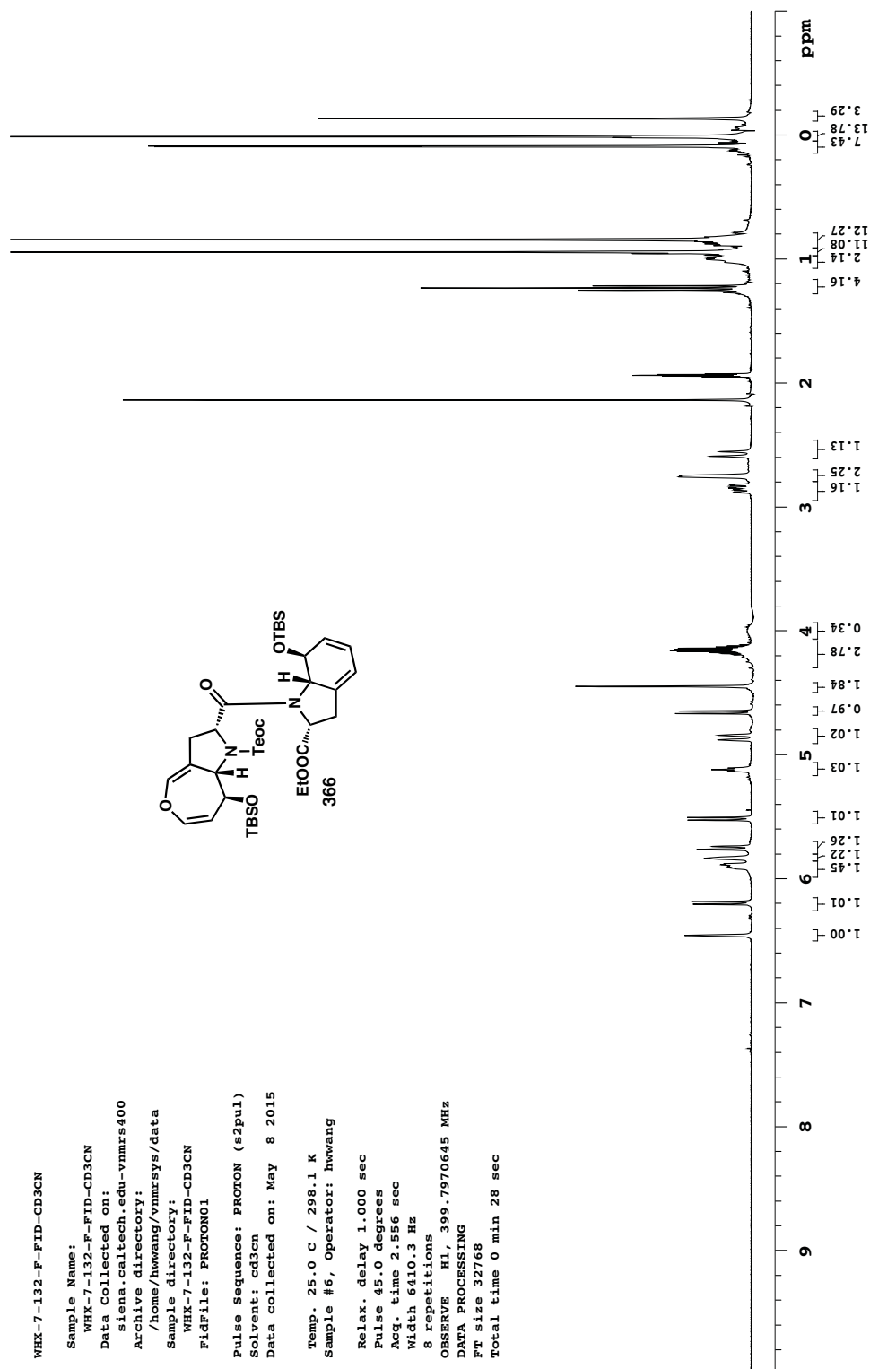
8 repetitions

OBSERVE HL, 399.7970645 MHz

DATA PROCESSING

FT size 32768

Total time 0 min 28 sec



WHX-7-132-F-FID-CD3CN-60C

Sample Name:

WHX-7-132-F-FID-CD3CN-60C
Data Collected on:
sienna.caltech.edu-vnmrs400
Archive directory:
/home/hhwang/vnmrsys/data

Sample directory:

WHX-7-132-F-FID-CD3CN-60C
FidFile: WHX-7-132-F-Siena-CD3CN-60C-Proton

Pulse Sequence: PROTON (s2pul)

Solvent: cd3cn
Data collected on: May 8 2015

Temp. 60.0 C / 333.1 K

Operator: hhwang

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

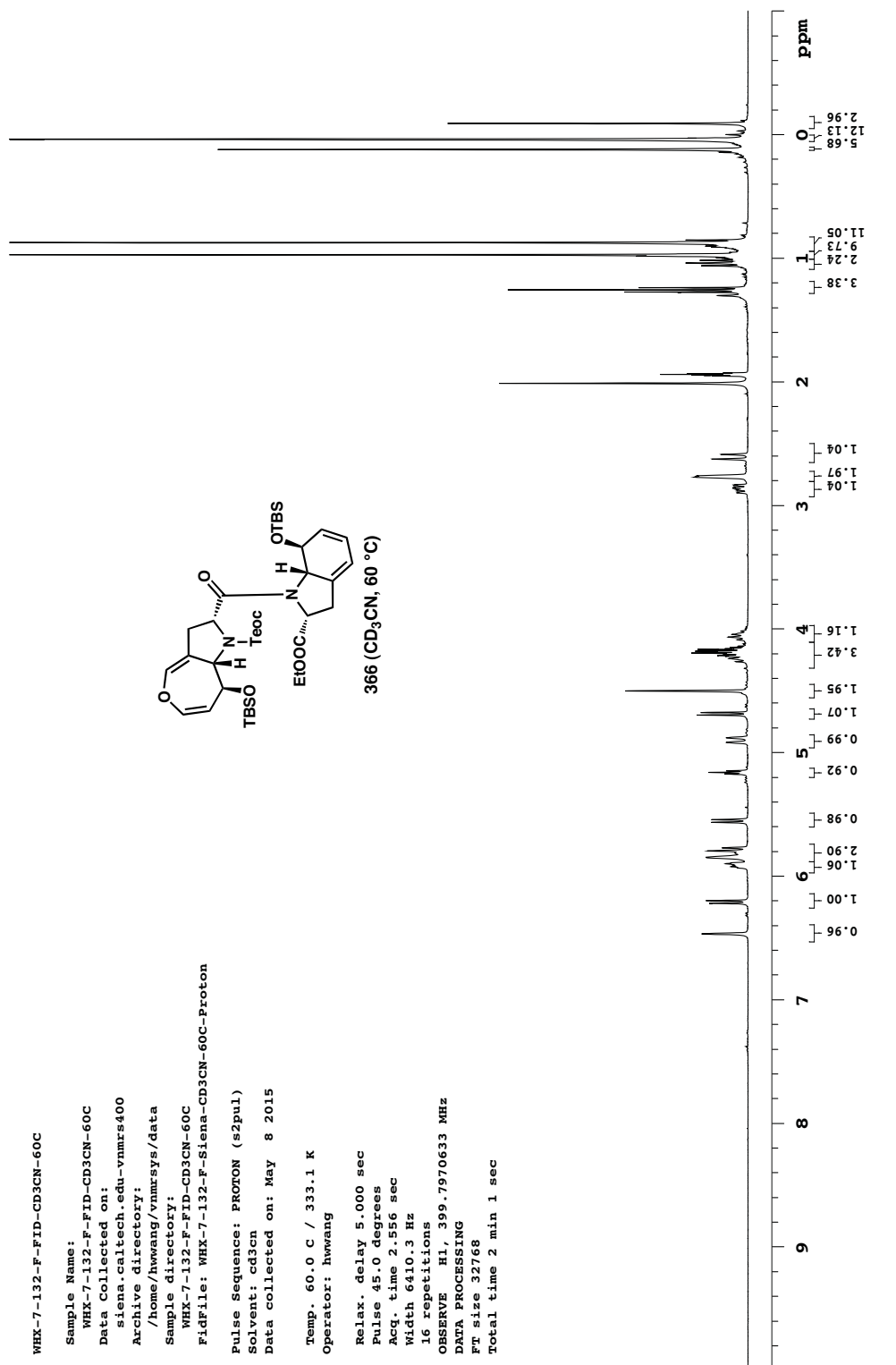
16 repetitions

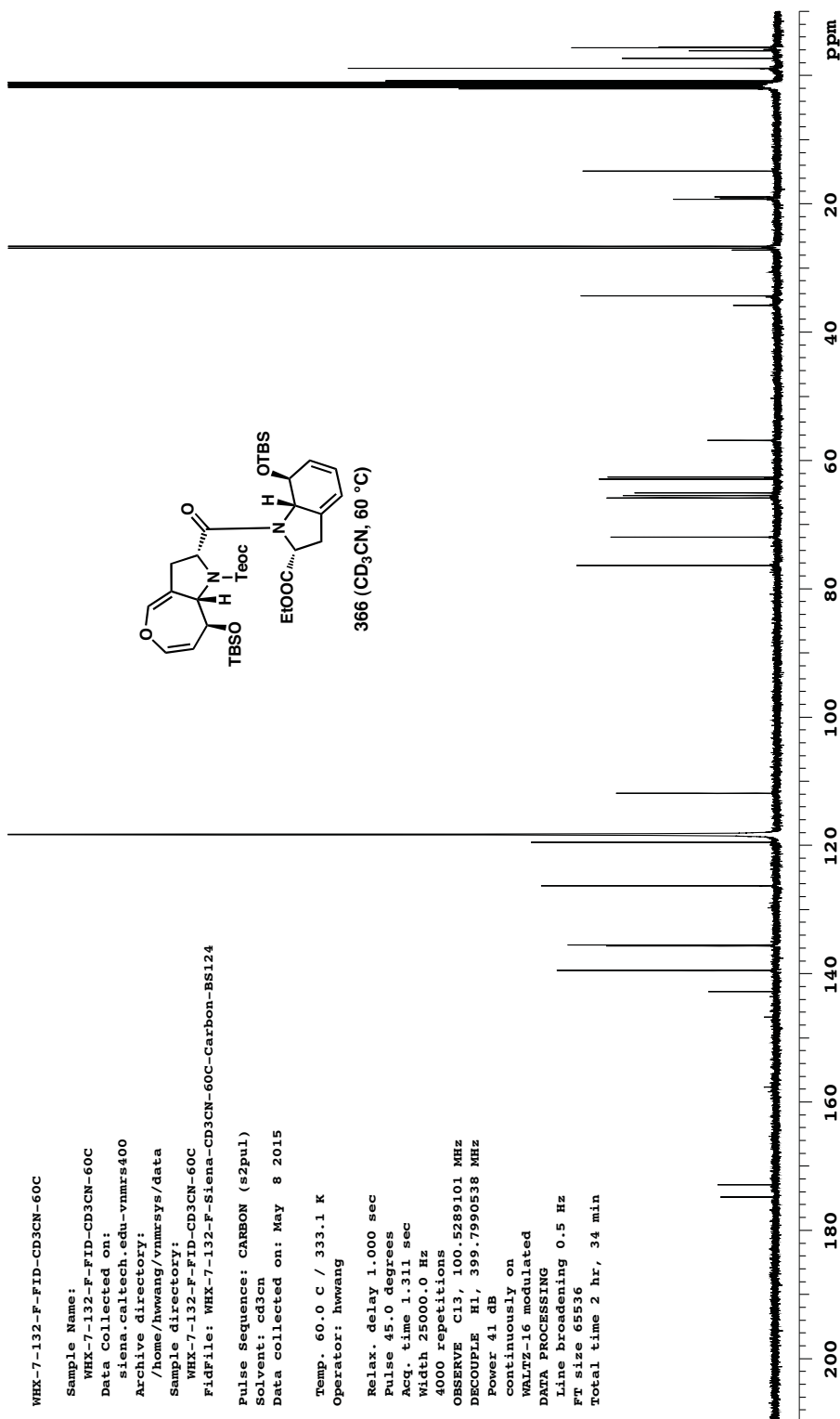
OBSERVE H1, 399.7970633 MHz

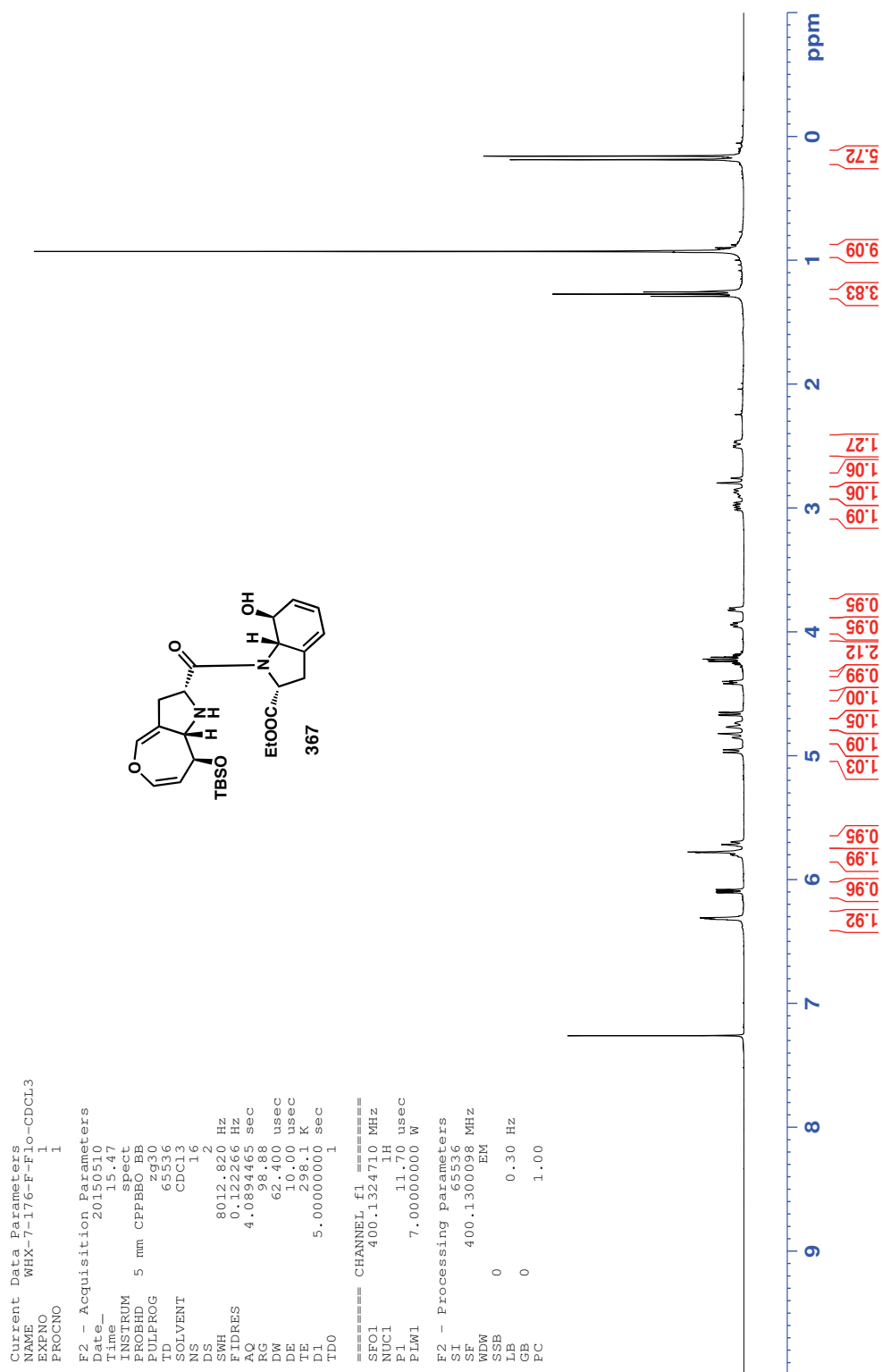
DATA PROCESSING

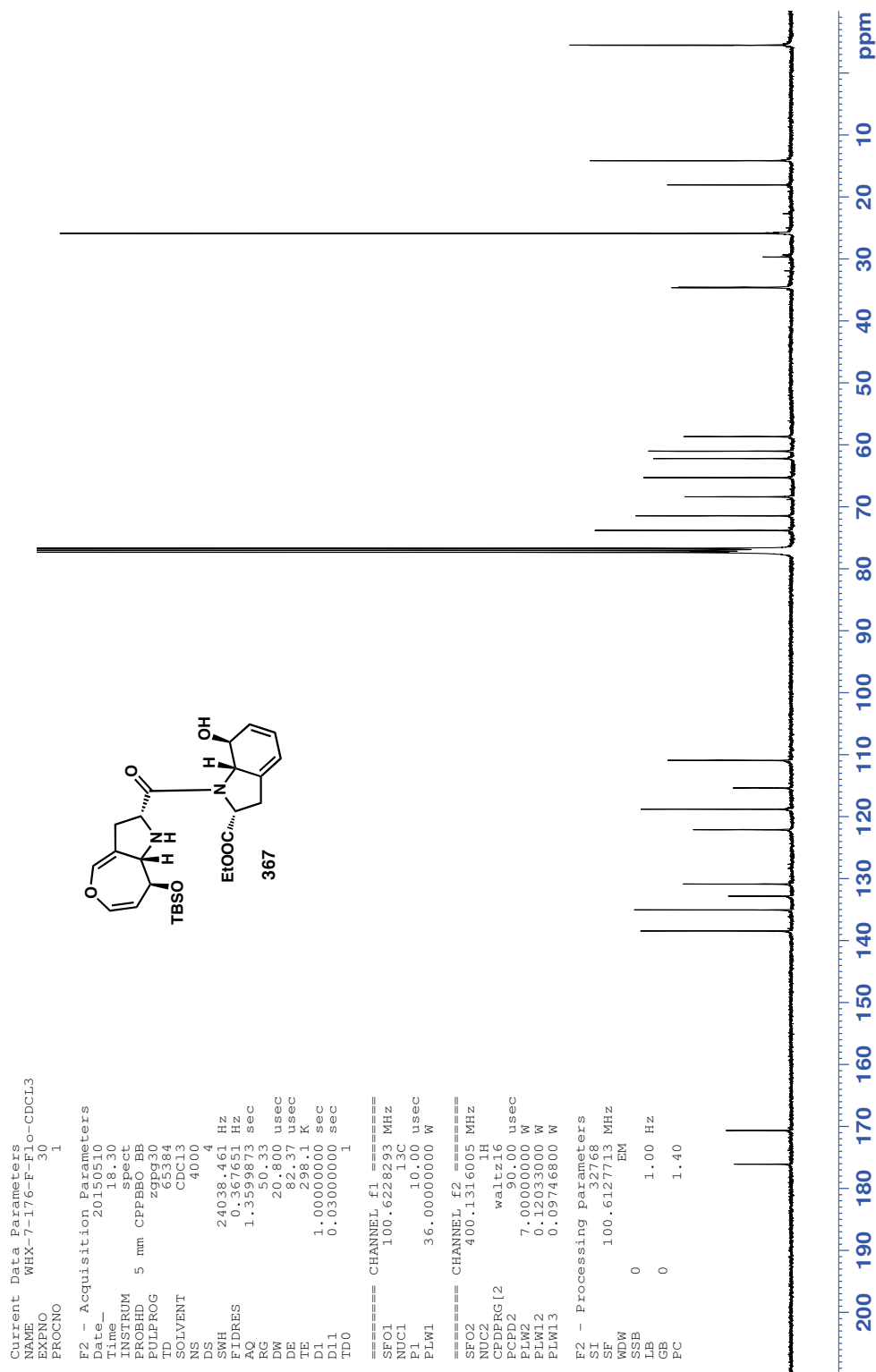
FT size 32768

Total time 2 min 1 sec









WHX-7-179-F-Character-INDY-CDCL3

Sample Name:

WHX-7-179-F-Character-INDY-CDCL3
 Data Collected on:
 indy.caltech.edu-inova500
 Archive directory:
 /home/hxwang/vnmrsys/data
 Sample directory:
 WHX-7-179-F-Character-INDY-CDCL3
 FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: May 12 2015

Sample #48, Operator: hxwang

Relax. delay 5.000 sec
 Pulse 45.0 degrees
 Acq. time 3.000 sec
 Width 8000.0 Hz
 16 repetitions

OBSERVE H1, 499.6749701 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 2 min 8 sec

