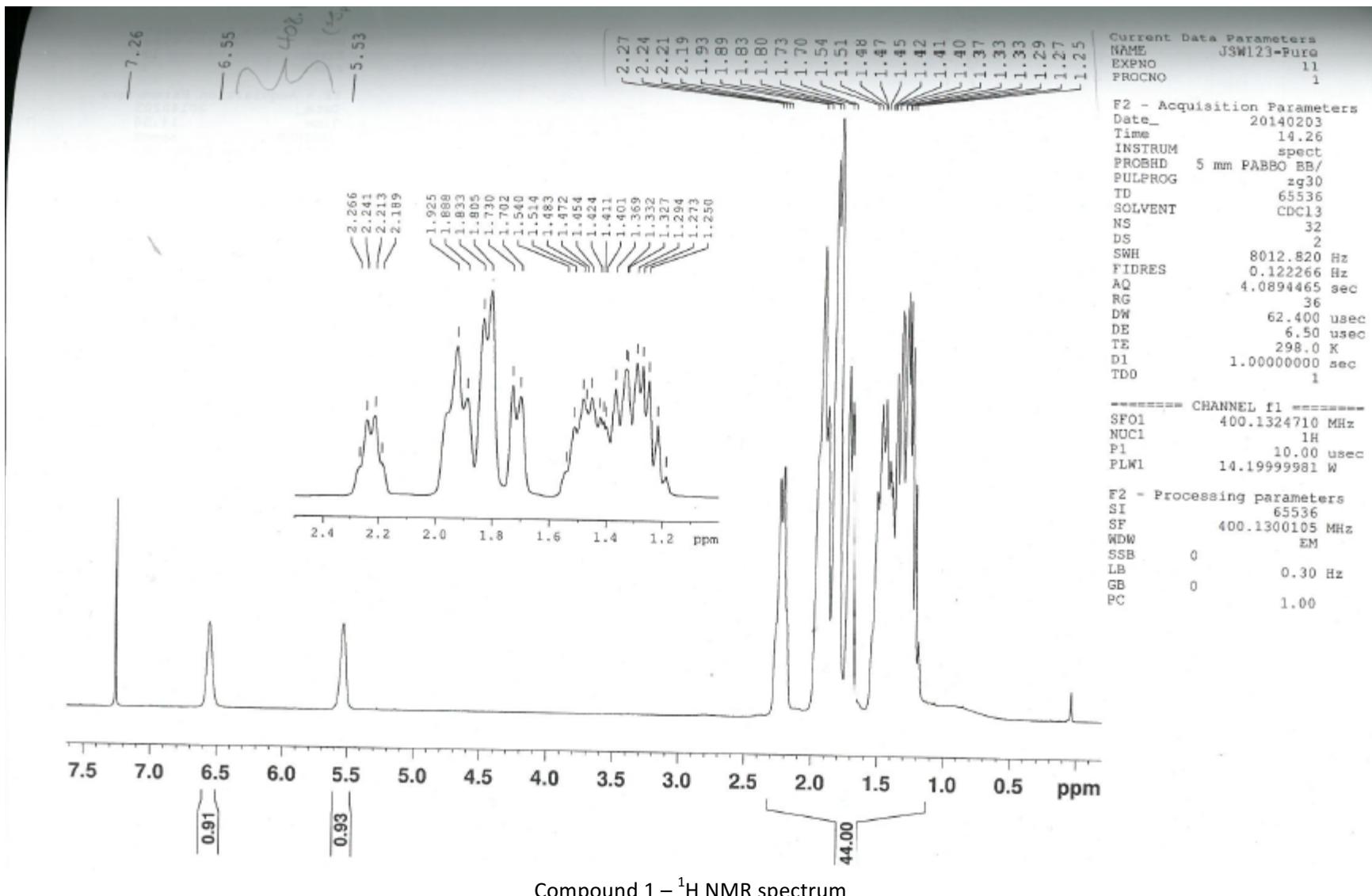


**Synthetic and Structural Studies of
Phosphine Coordinated Boronium Salts**

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Electronic Supporting Information (ESI)



Compound 1 – ^1H NMR spectrum

```

JSM123-DCMPent_Precip.p
Sample Name:
  JSM123-DCMPent_Precip_
Data Collected on:
  purcell-inova300
Archive directory:
  /home/hill/vnmrsys/data
Sample directory:
  MS-43-highC_20110913_01
FidFile: stdlh

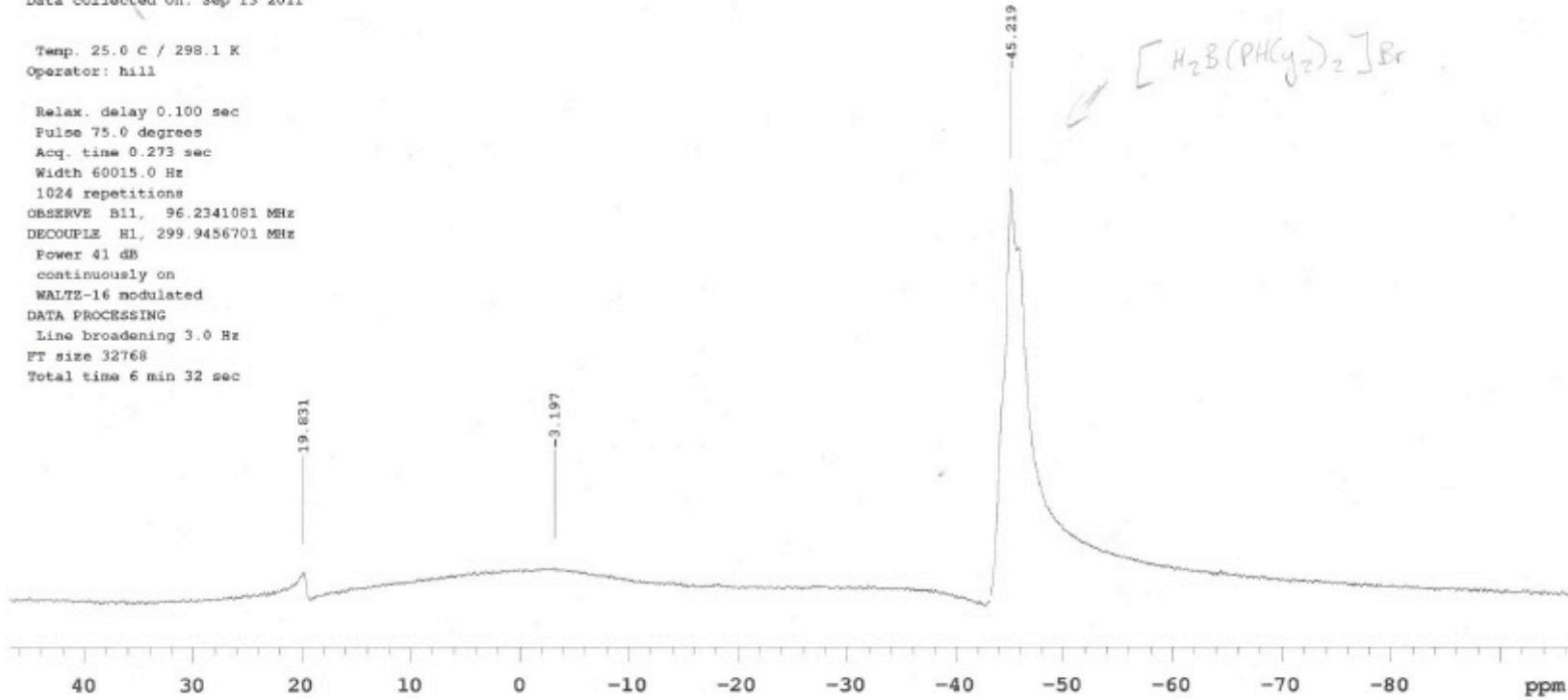
Pulse Sequence: stdlh (s2pul)
Solvent: cd2cl2
Data collected on: Sep 13 2011

Temp. 25.0 C / 298.1 K
Operator: hill

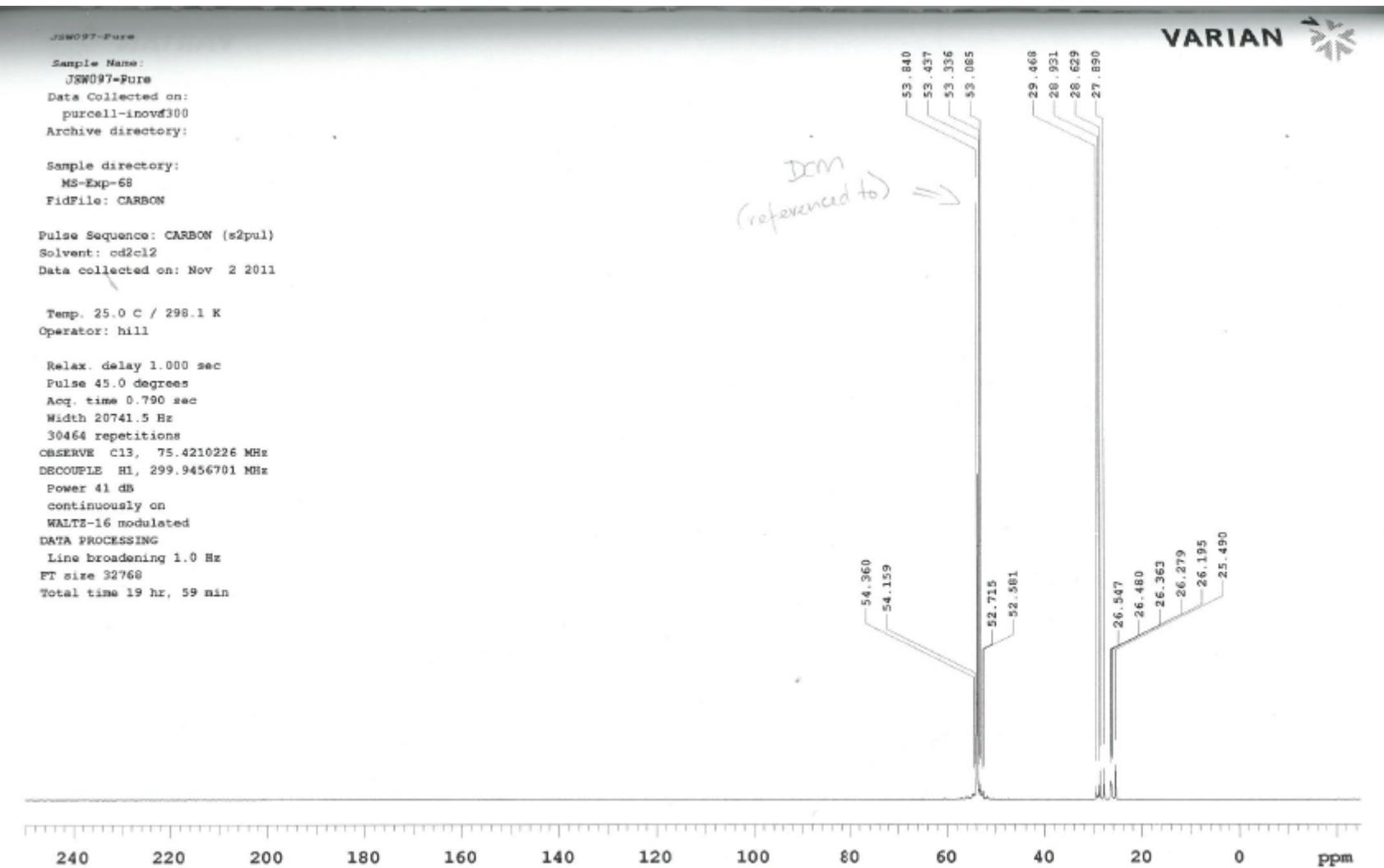
Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1024 repetitions
OBSERVE H1, 96.2341081 MHz
DECOUPLE H1, 299.9456701 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 6 min 32 sec

```

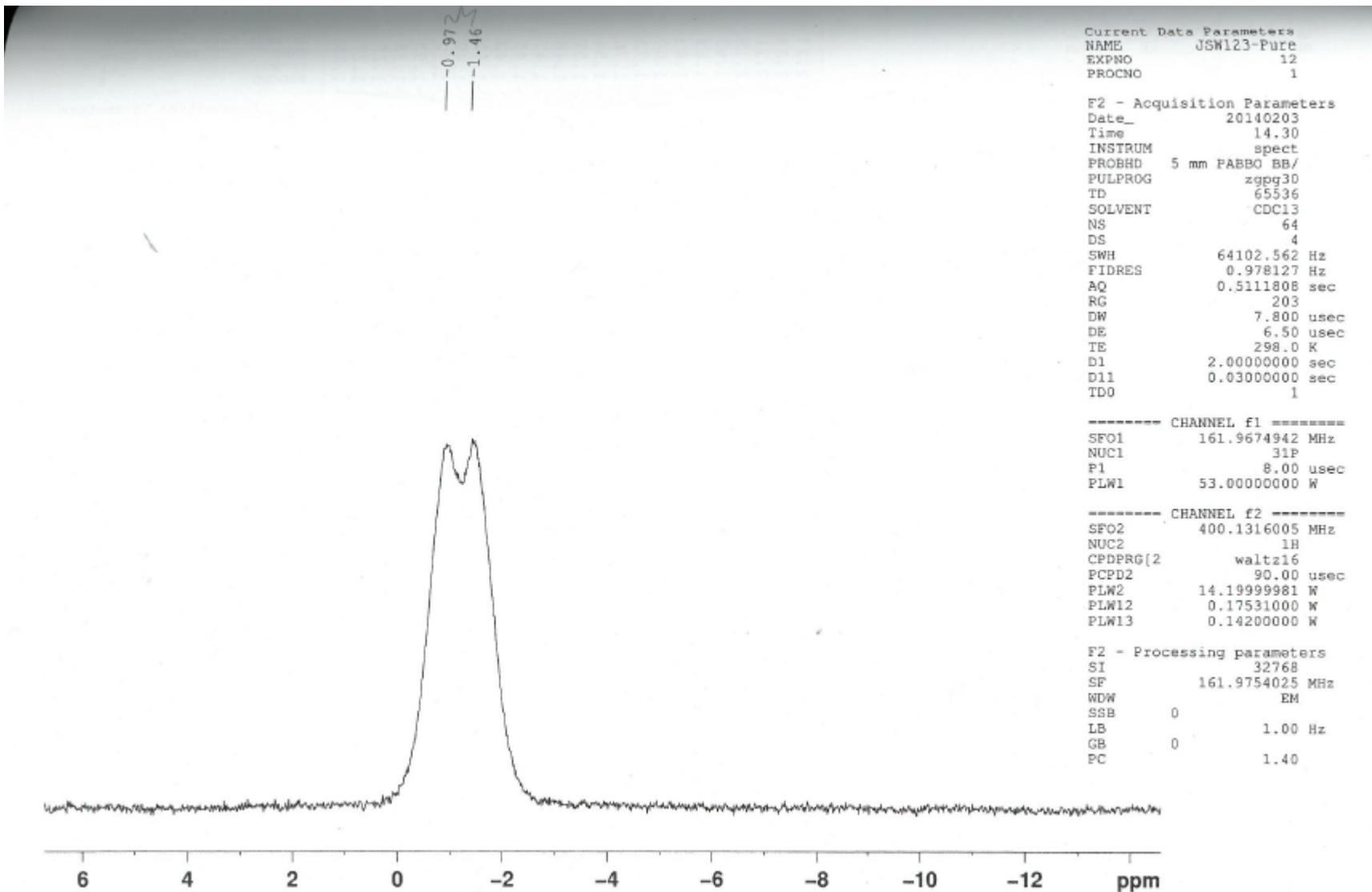
VARIAN



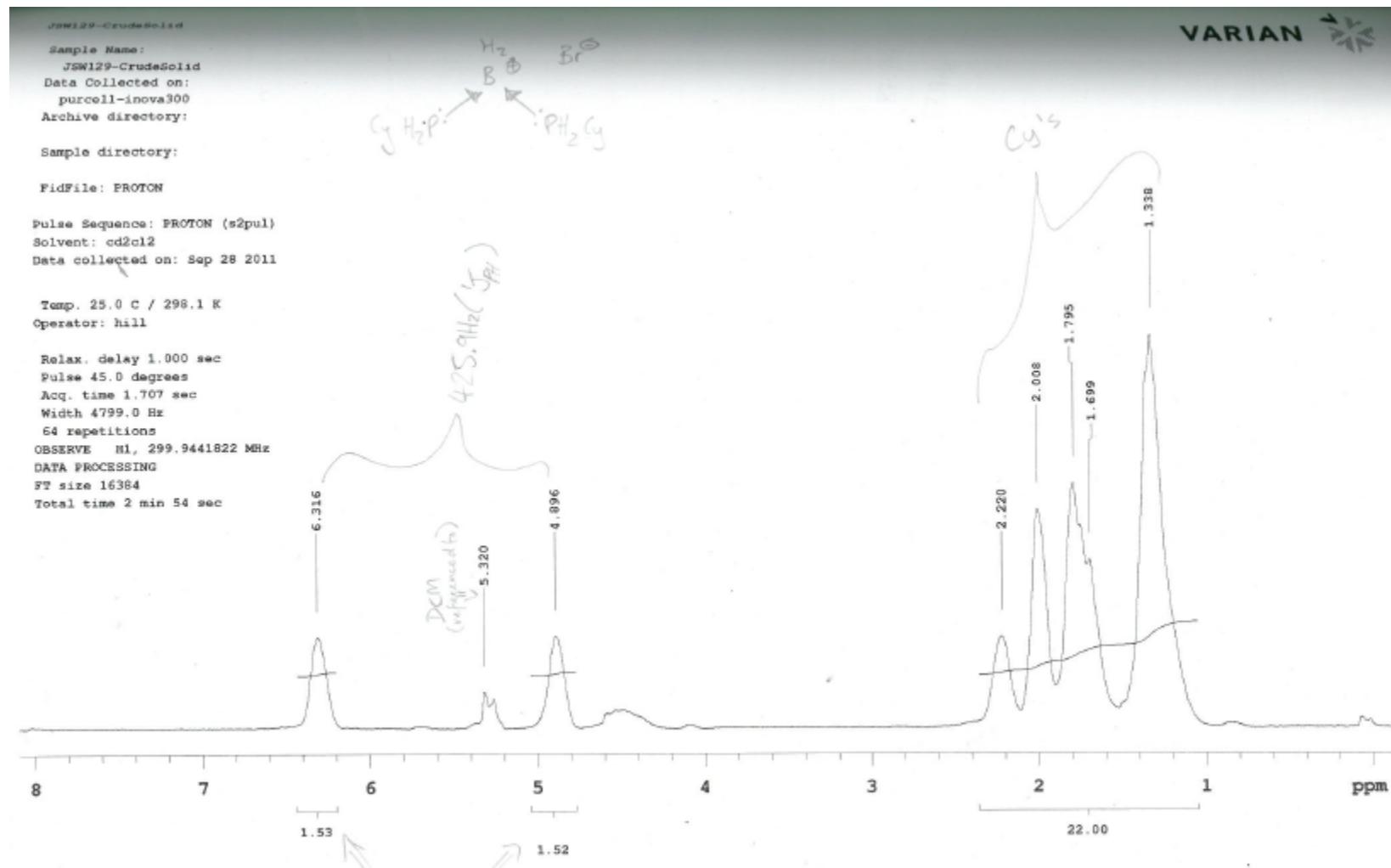
Compound 1 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



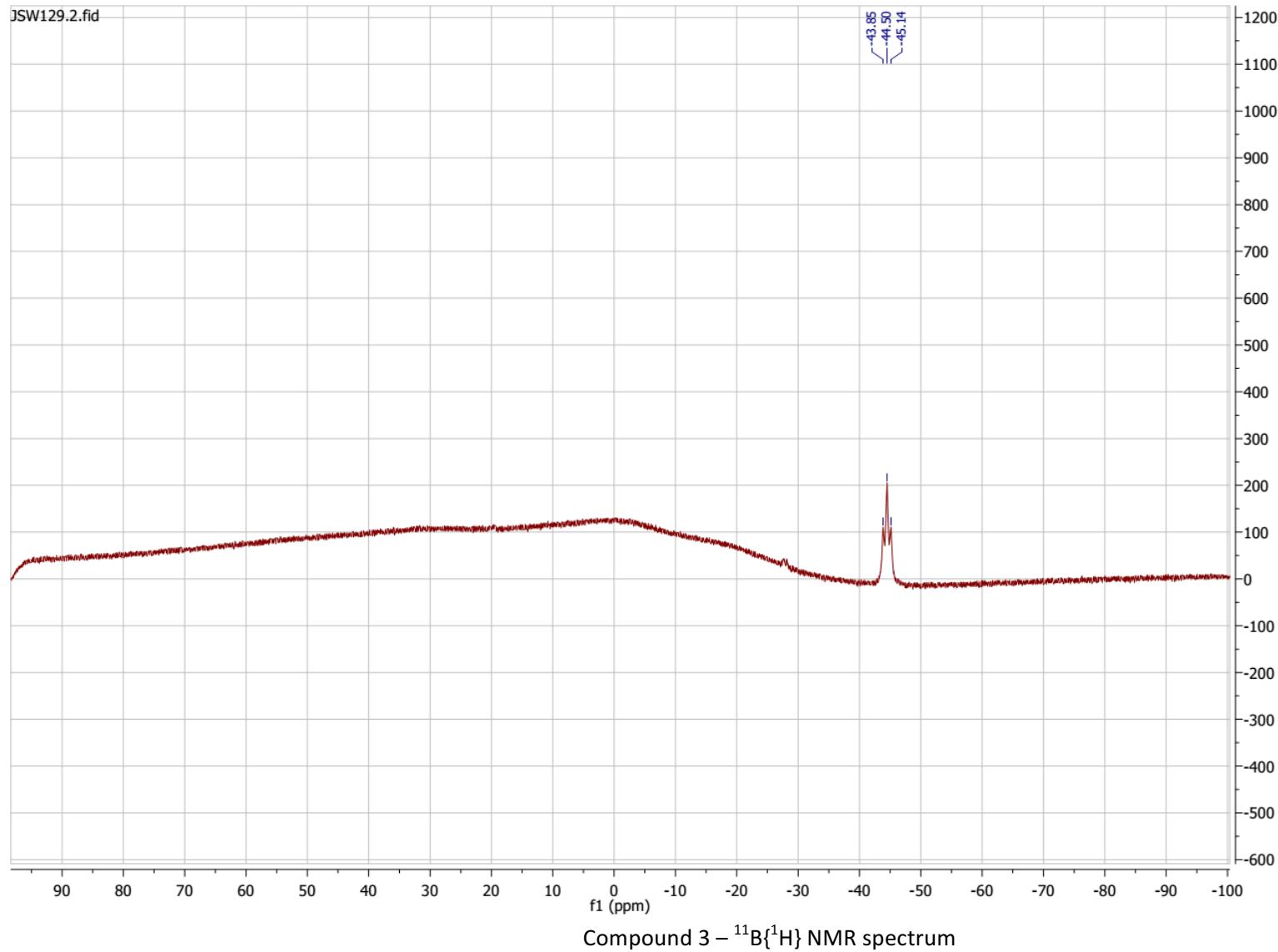
Compound 1 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

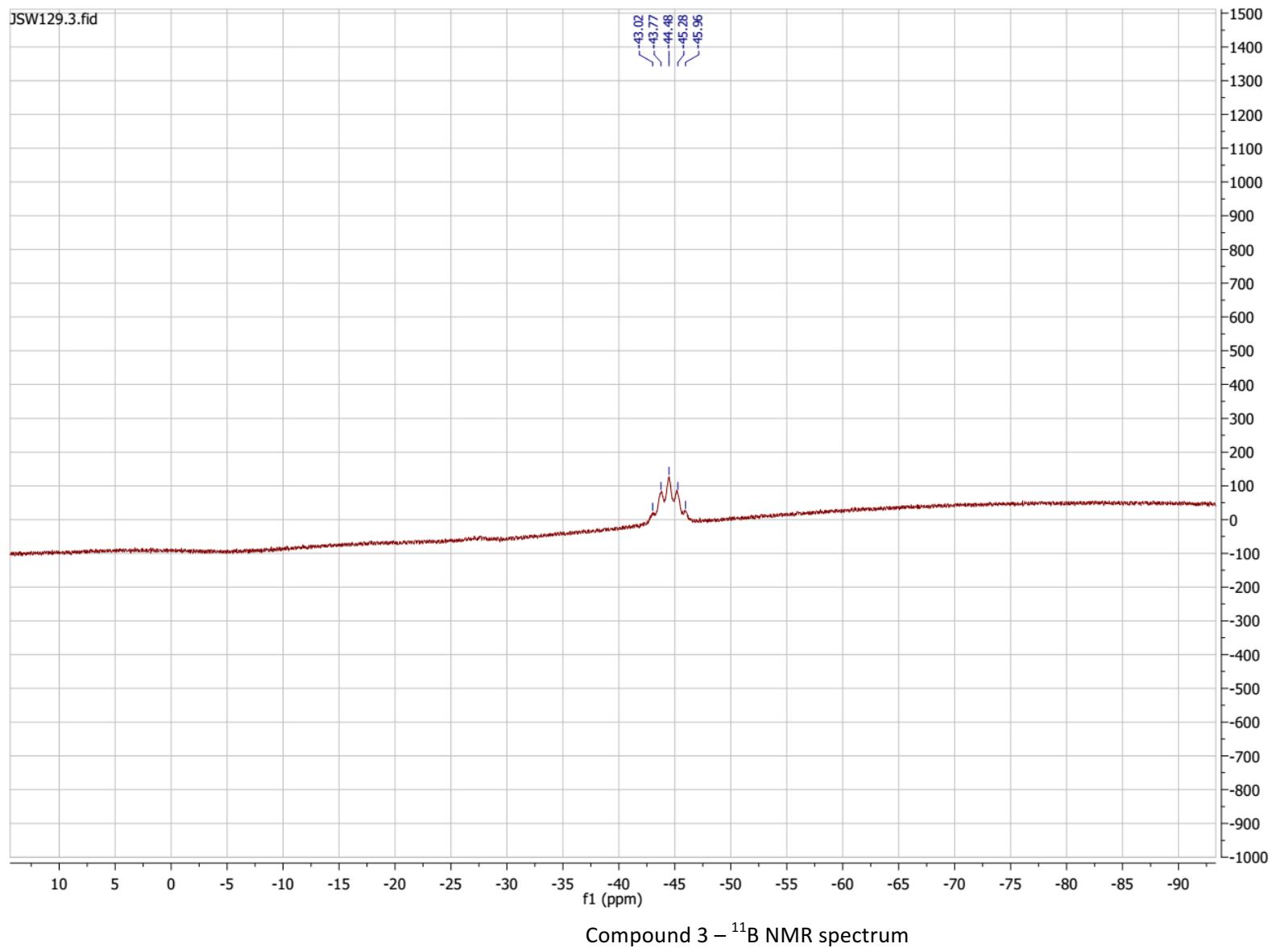


Compound 1 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum



Compound 3 – ^1H NMR spectrum





JSW129-Pure

Sample Name:
JSW129-Pure
Data Collected on:
purcell-inova300
Archive directory:

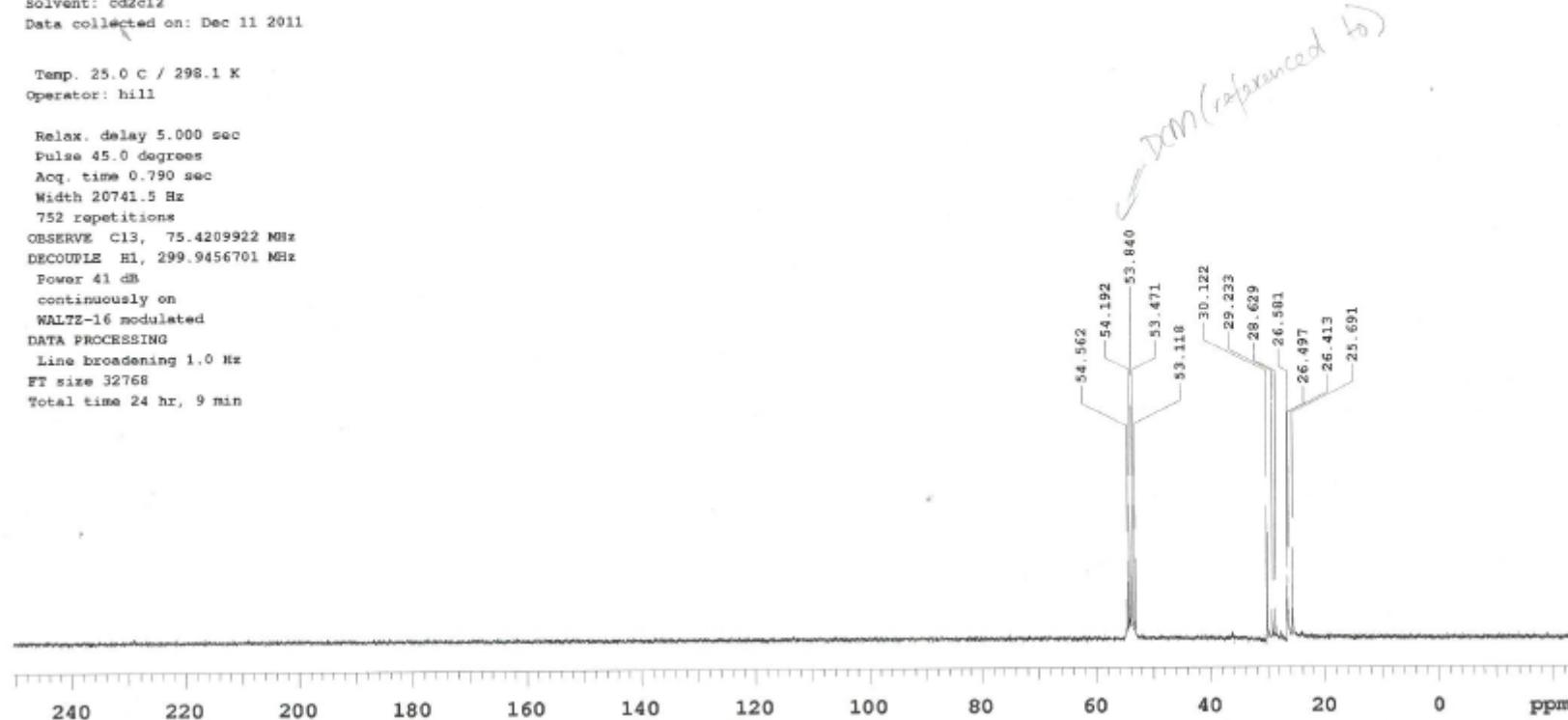
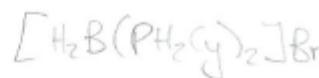
Sample directory:
MS-Exp-79
FidFile: CARBON

Pulse Sequence: CARBON (s2pal)
Solvent: cd2c12
Data collected on: Dec 11 2011

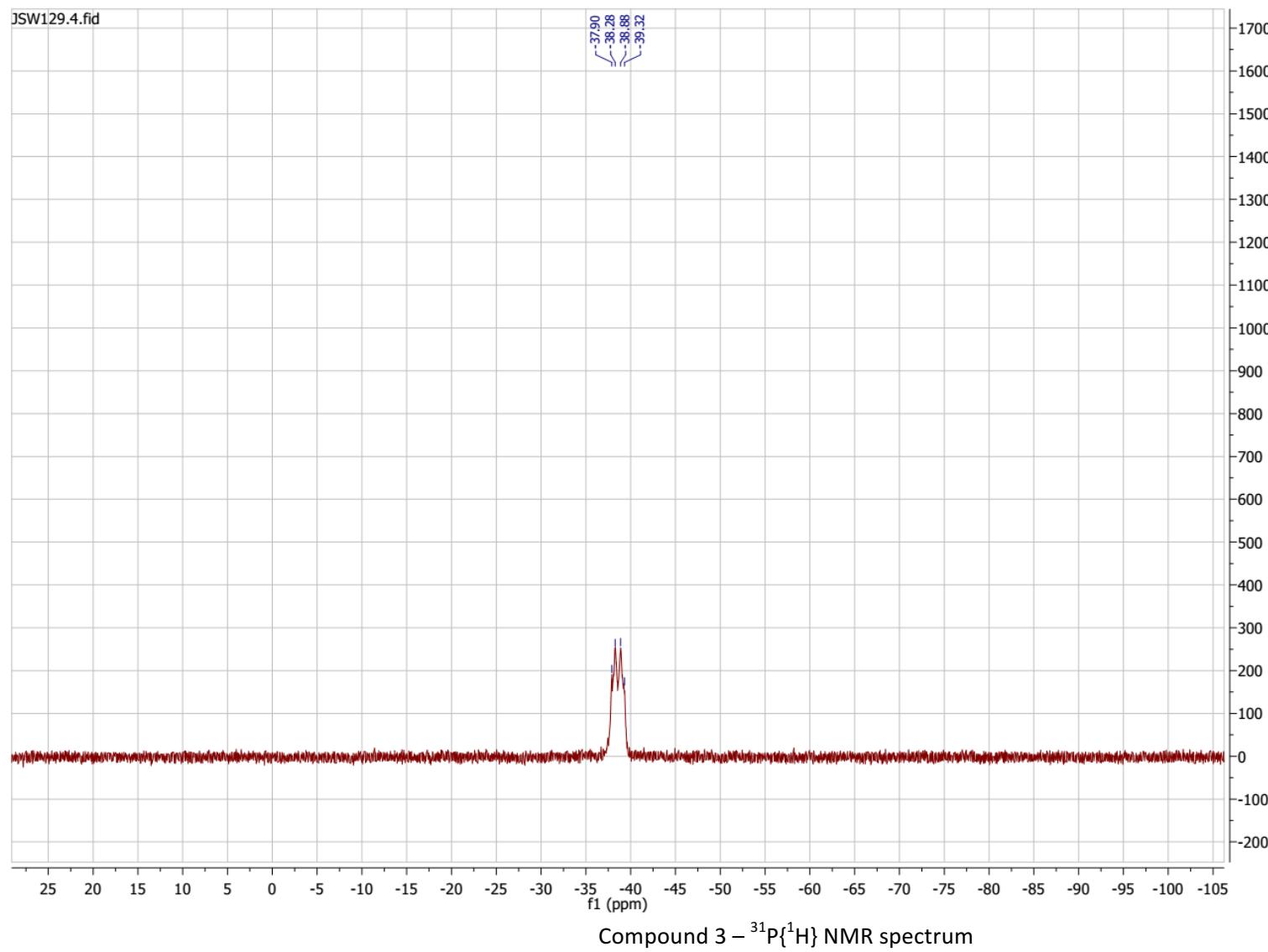
Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 0.790 sec
Width 20741.5 Hz
752 repetitions
OBSERVE C13, 75.4209922 MHz
DECOUPLE H1, 299.9456701 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 24 hr, 9 min

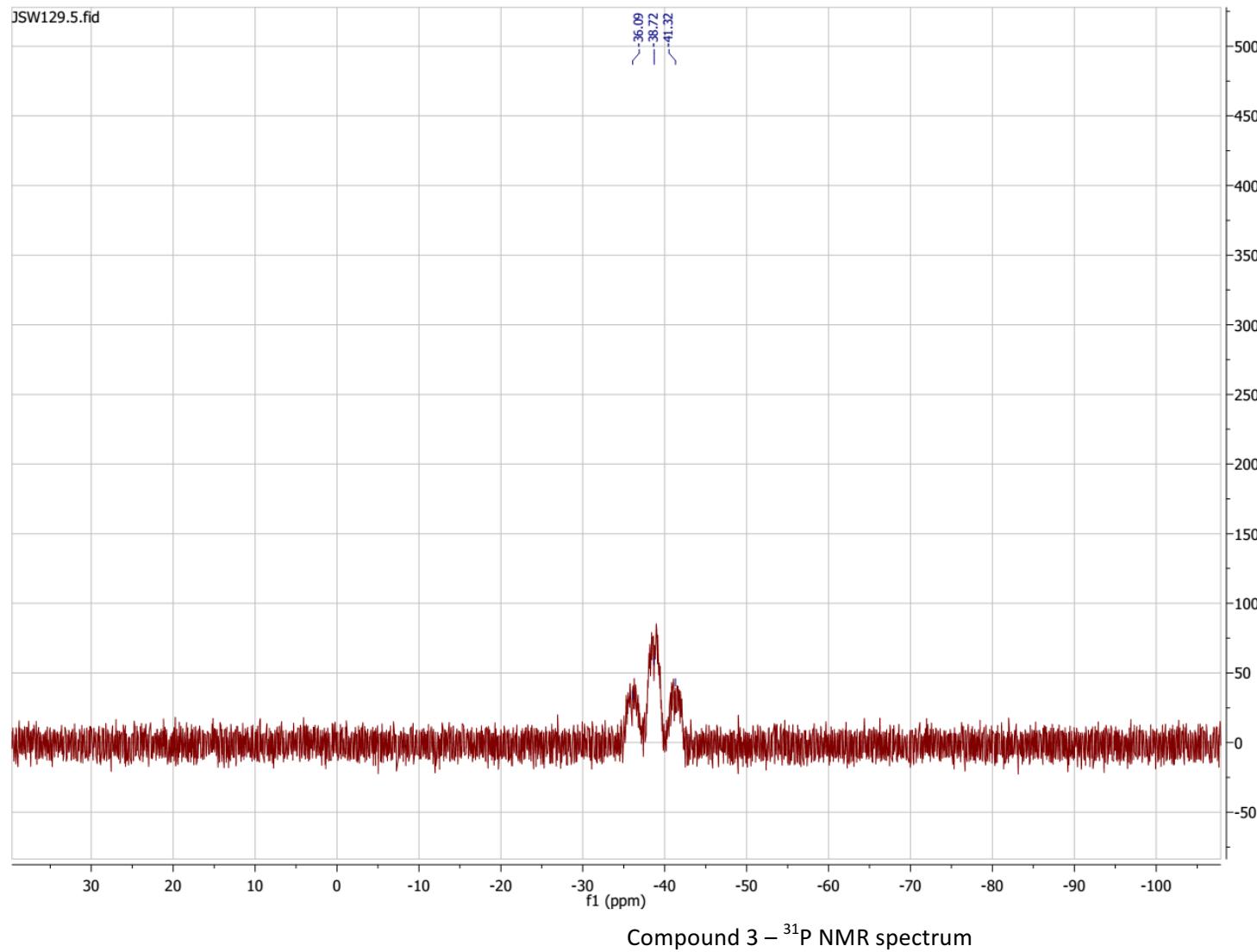
VARIAN

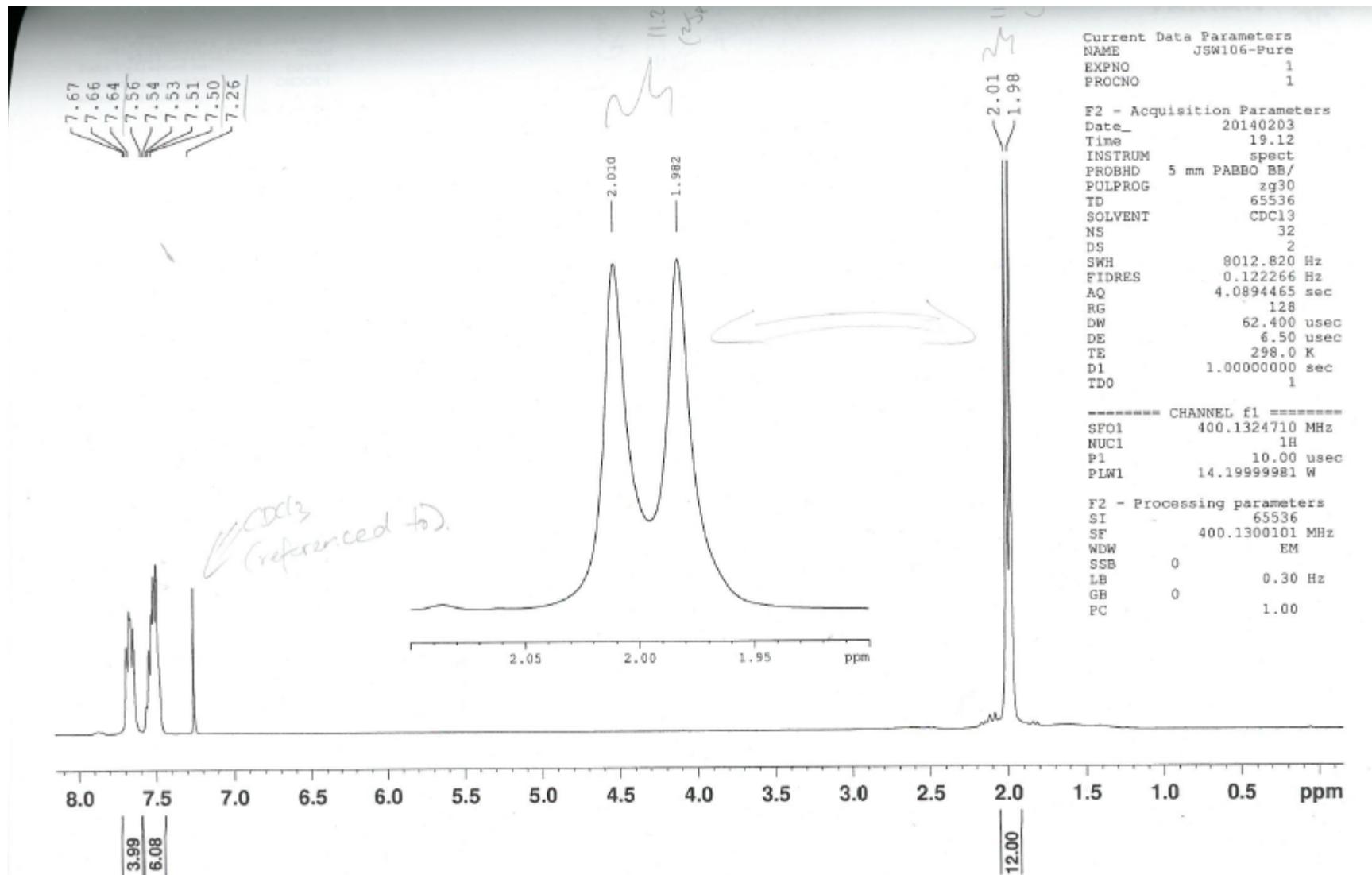


Compound 3 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

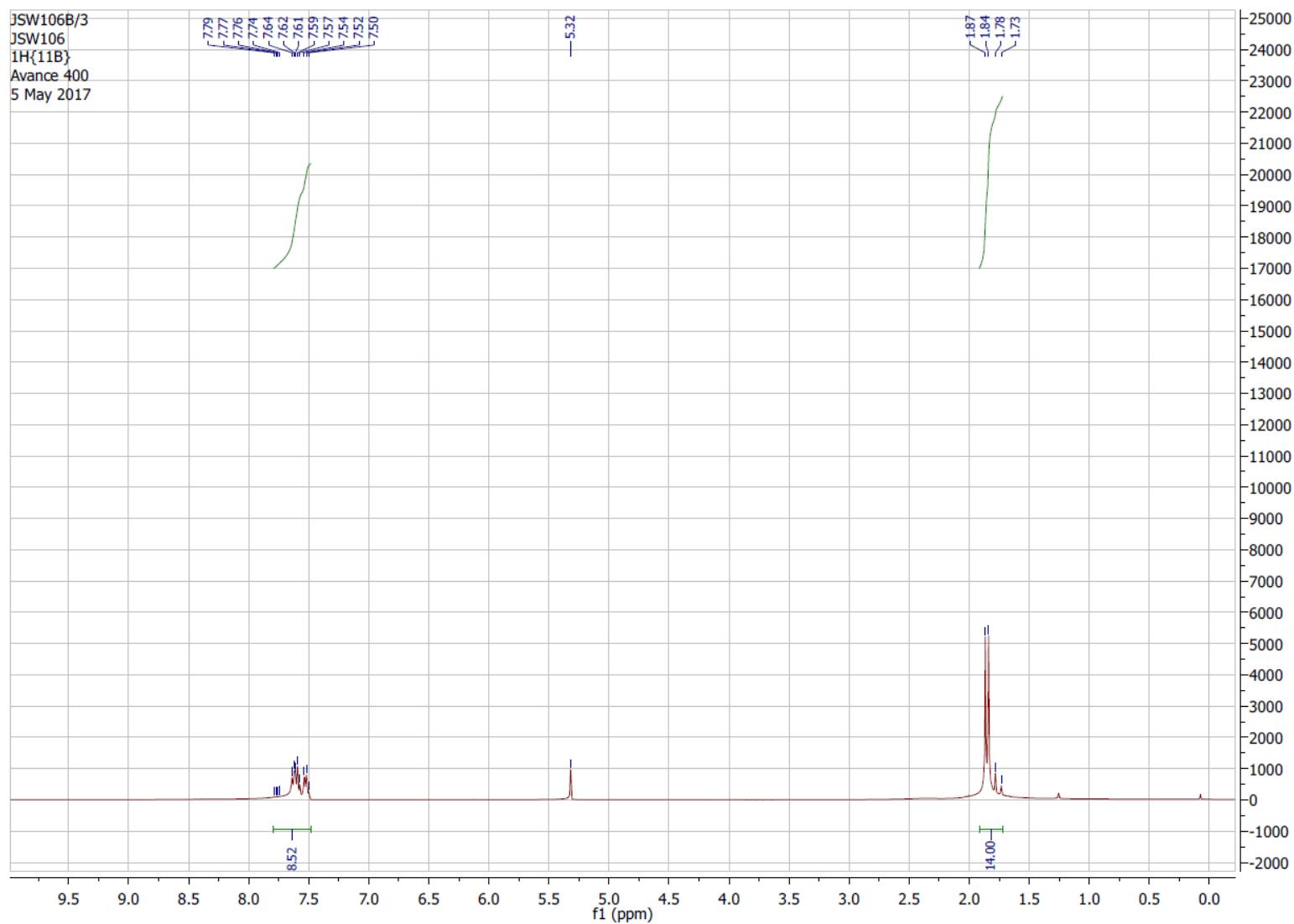


JSW129.5.fid

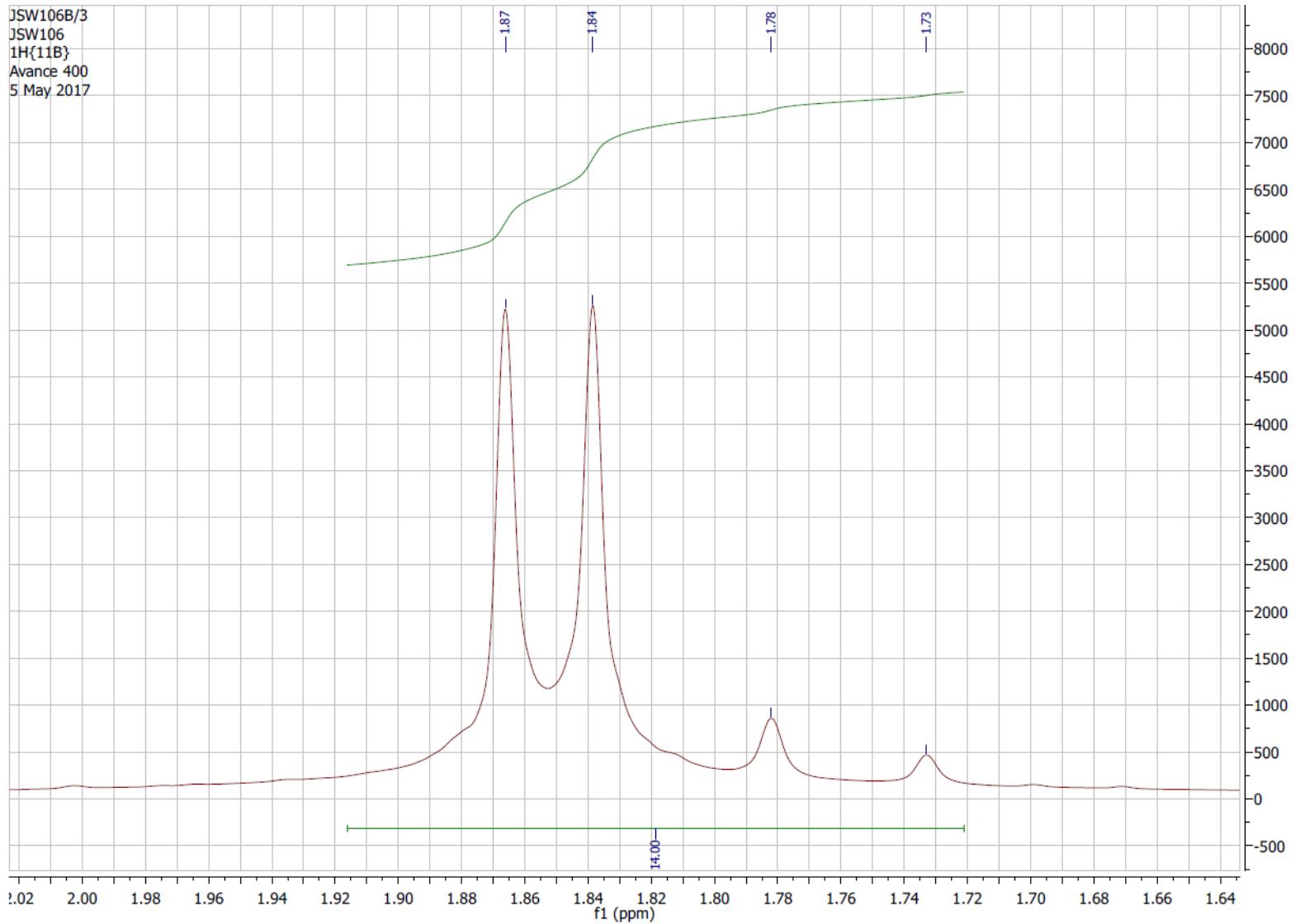




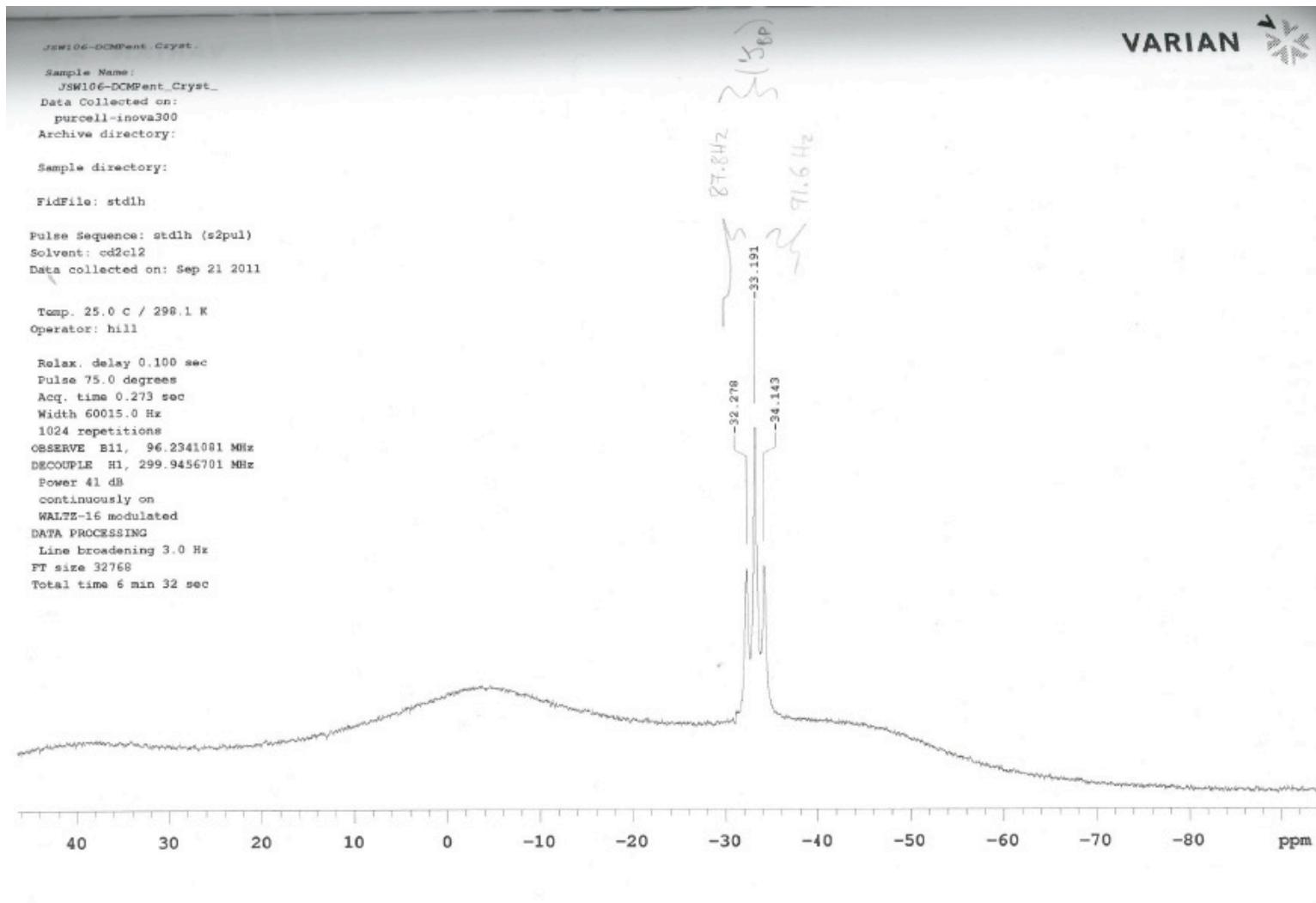
Compound 4 – ¹H NMR spectrum



Compound 4 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum



Compound 4 – $^1\text{H}\{\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)



Sample Name: JBW106-DCMPant_Cryst
Data Collected on: purcell-inova300
Archive directory:

Sample directory:

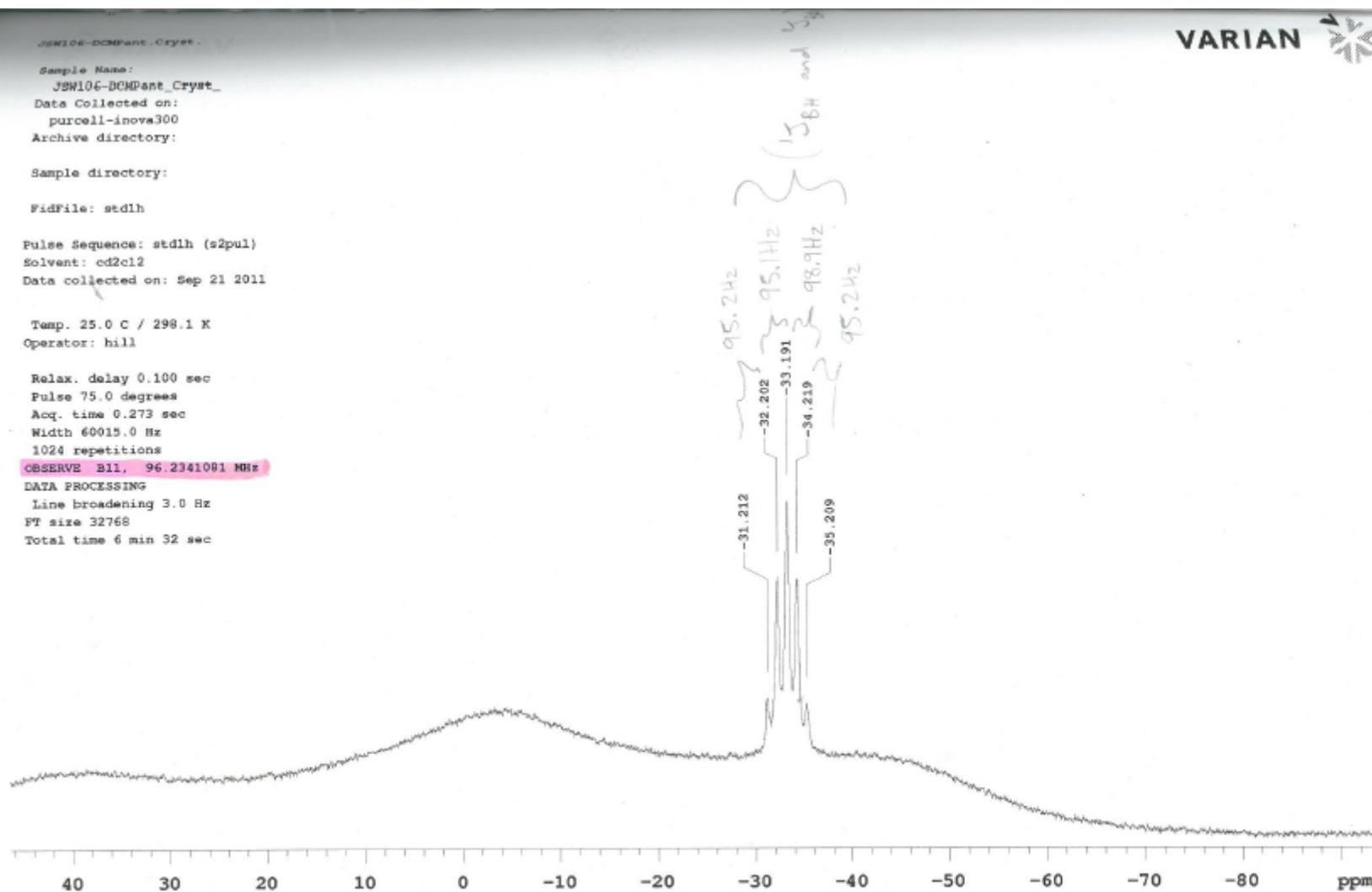
FidFile: stdlh

Pulse Sequence: stdlh (s2p
Solvent: cd2cl2
Data collected on: Sep 21

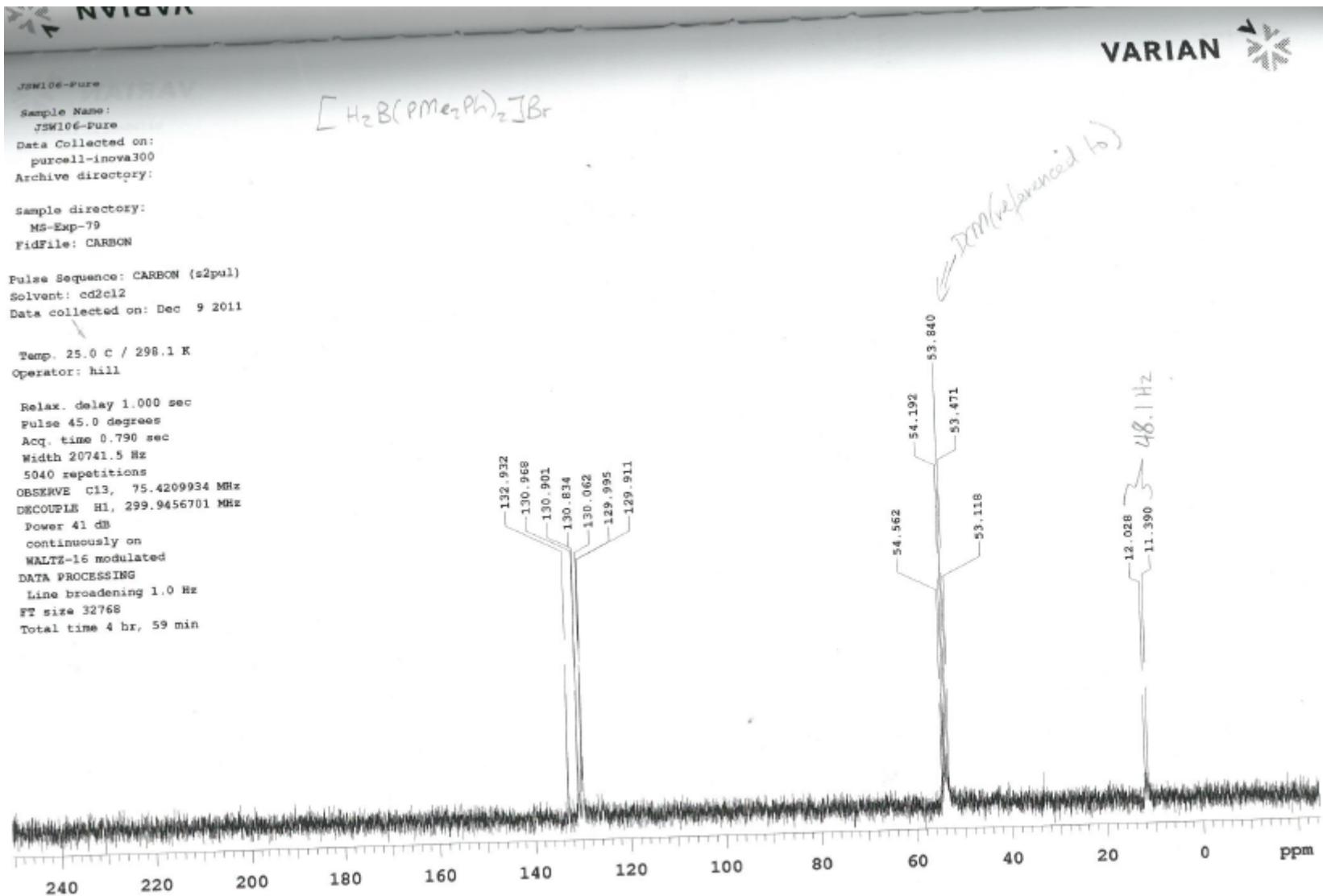
Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1024 repetitions
OBSERVE B1L 96.2341081
DATA PROCESSING
Line broadening 3.0 Hz
PT size 32768
Total time 6 min 32 sec

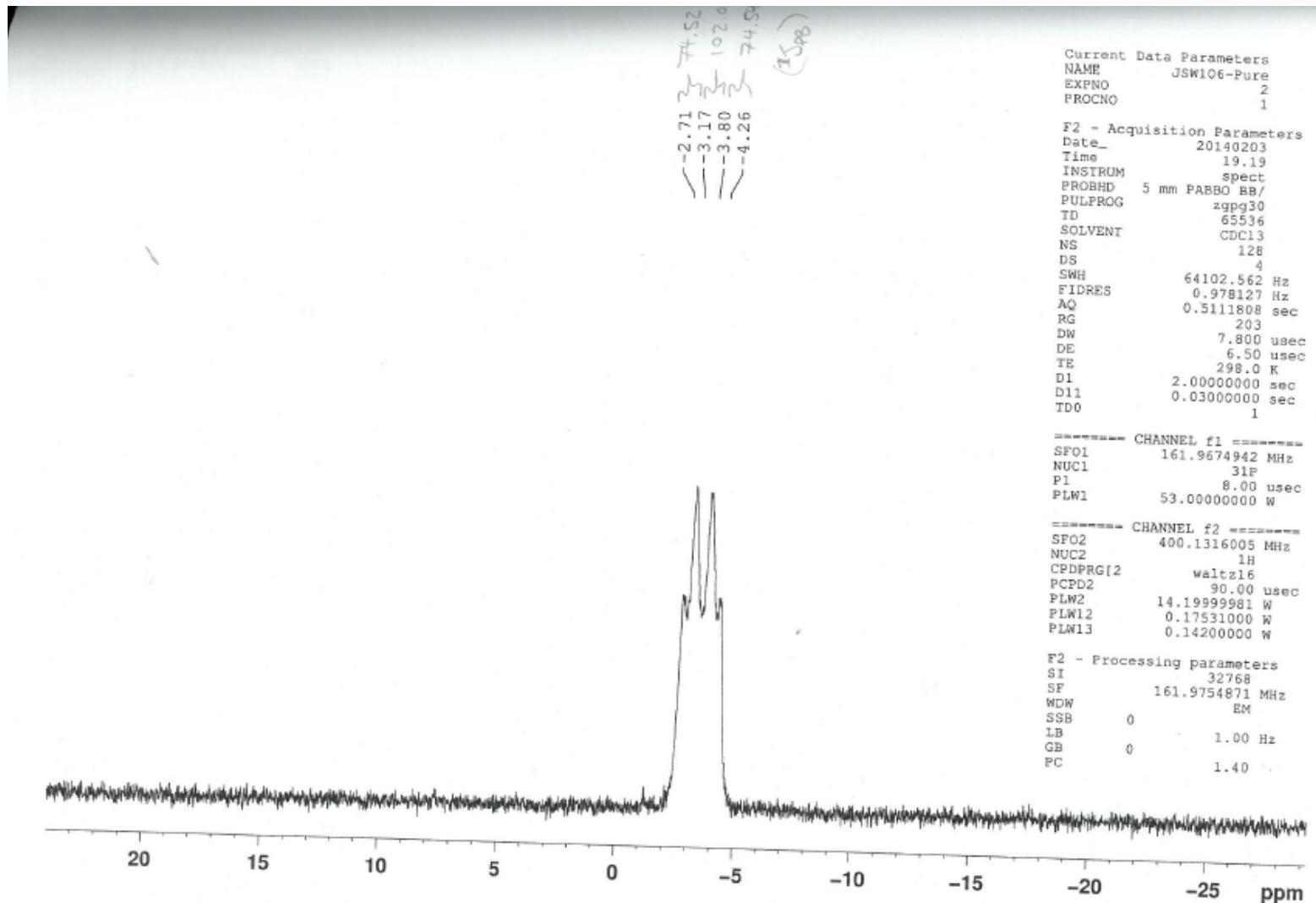
VARIAN



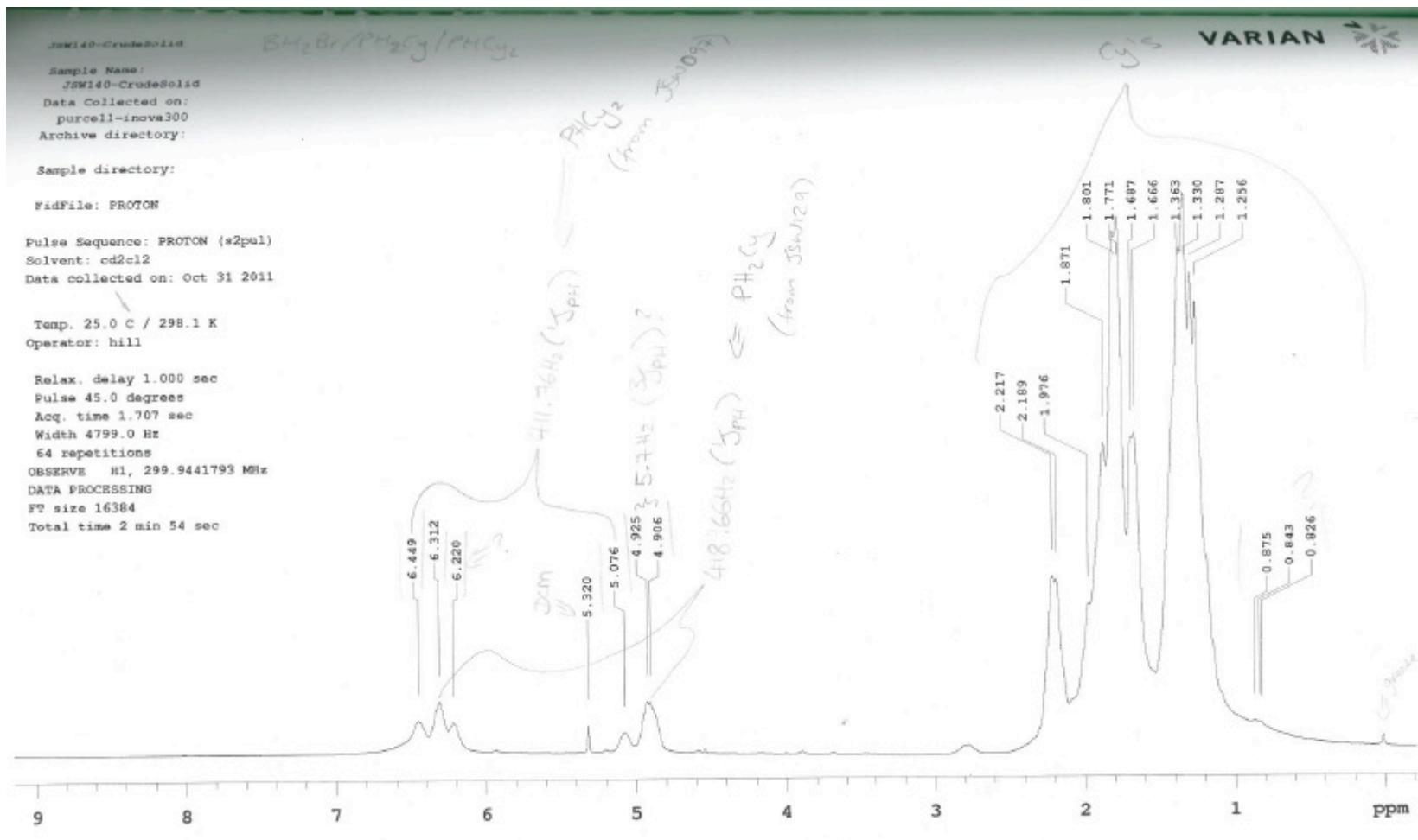
Compound 4 – ^{11}B NMR spectrum



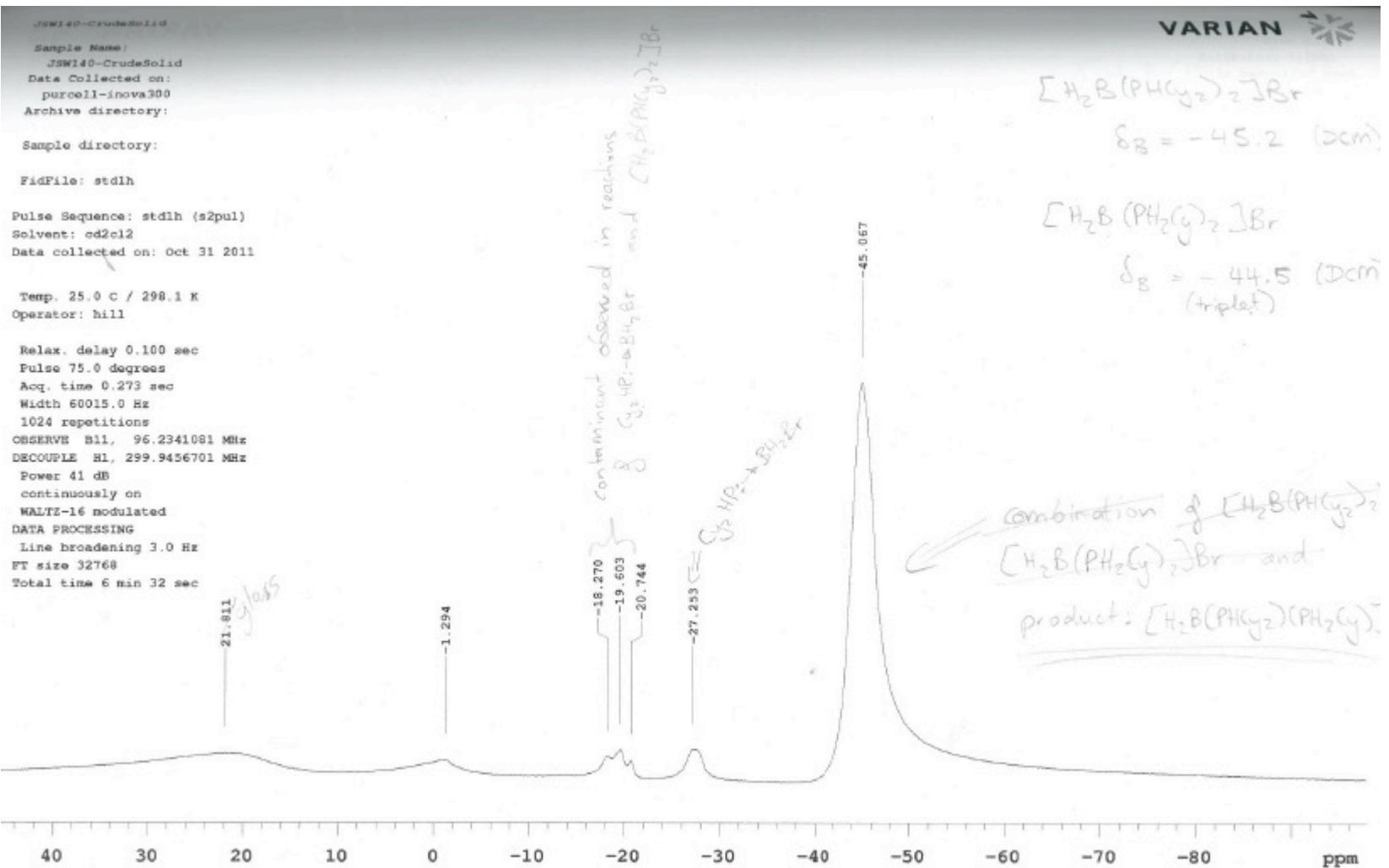
Compound 4 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



Compound 4 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum



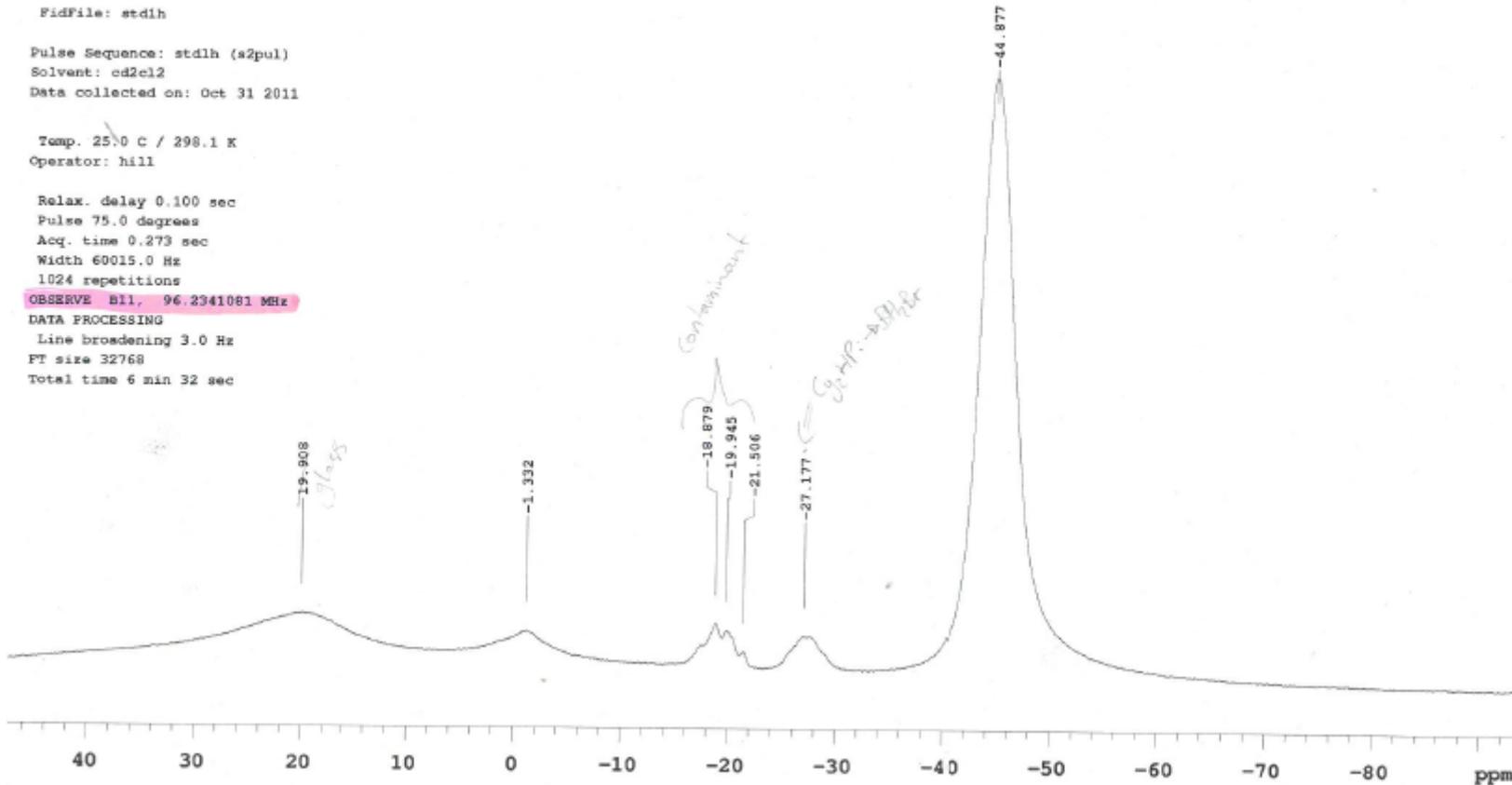
Compound 5 – ^1H NMR spectrum



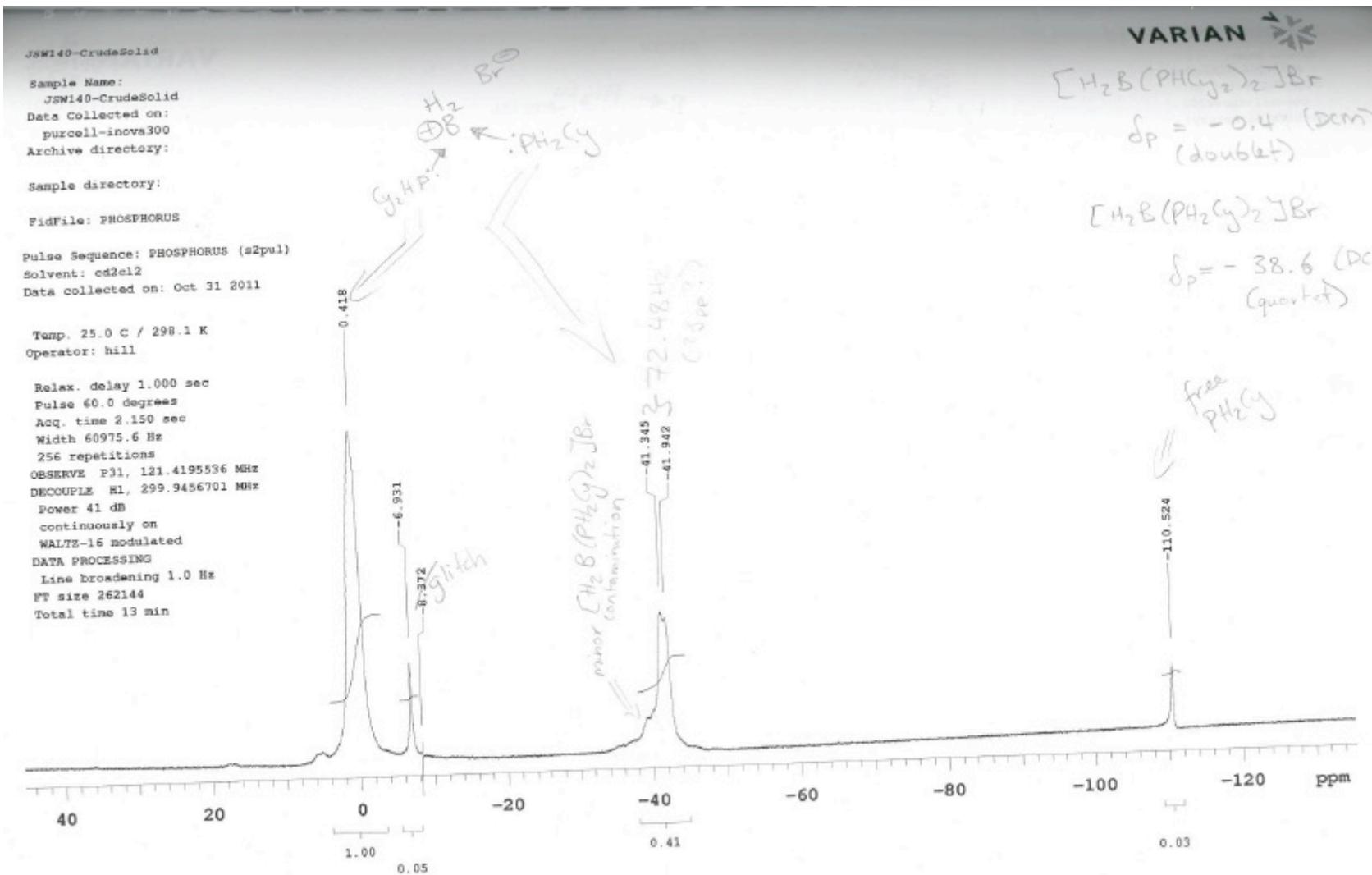
Compound 5 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

VARIAN

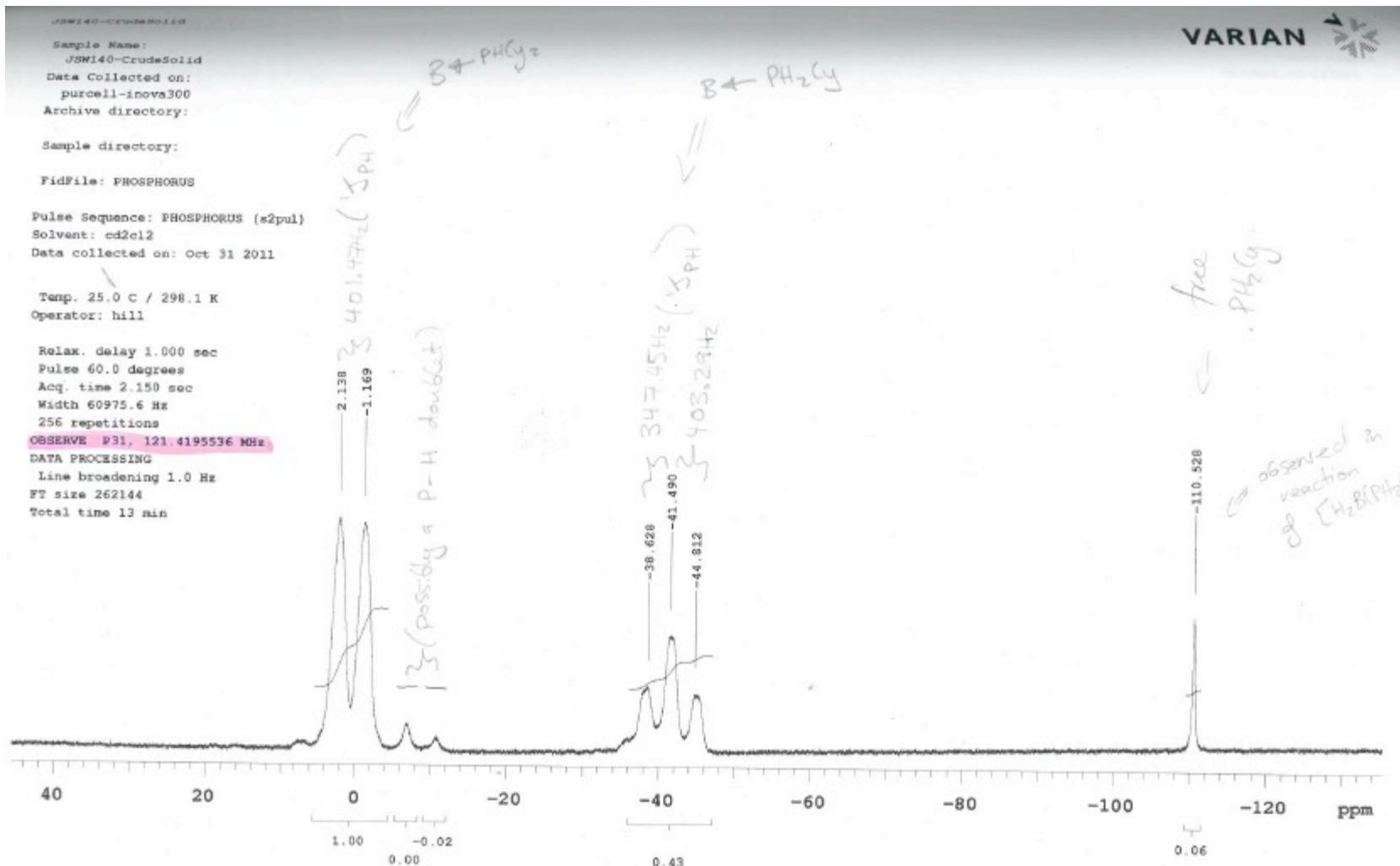
Sample Name: JSW140-CrudeSolid
Data Collected on: purcell-innova300
Archive directory:
Sample directory:
FidFile: stdlh
Pulse Sequence: stdlh (s2pul)
Solvent: cd2cl2
Data collected on: Oct 31 2011
Temp. 25.0 °C / 298.1 K
Operator: hill
Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1024 repetitions
OBSERVE B11, 96.2341081 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 6 min 32 sec



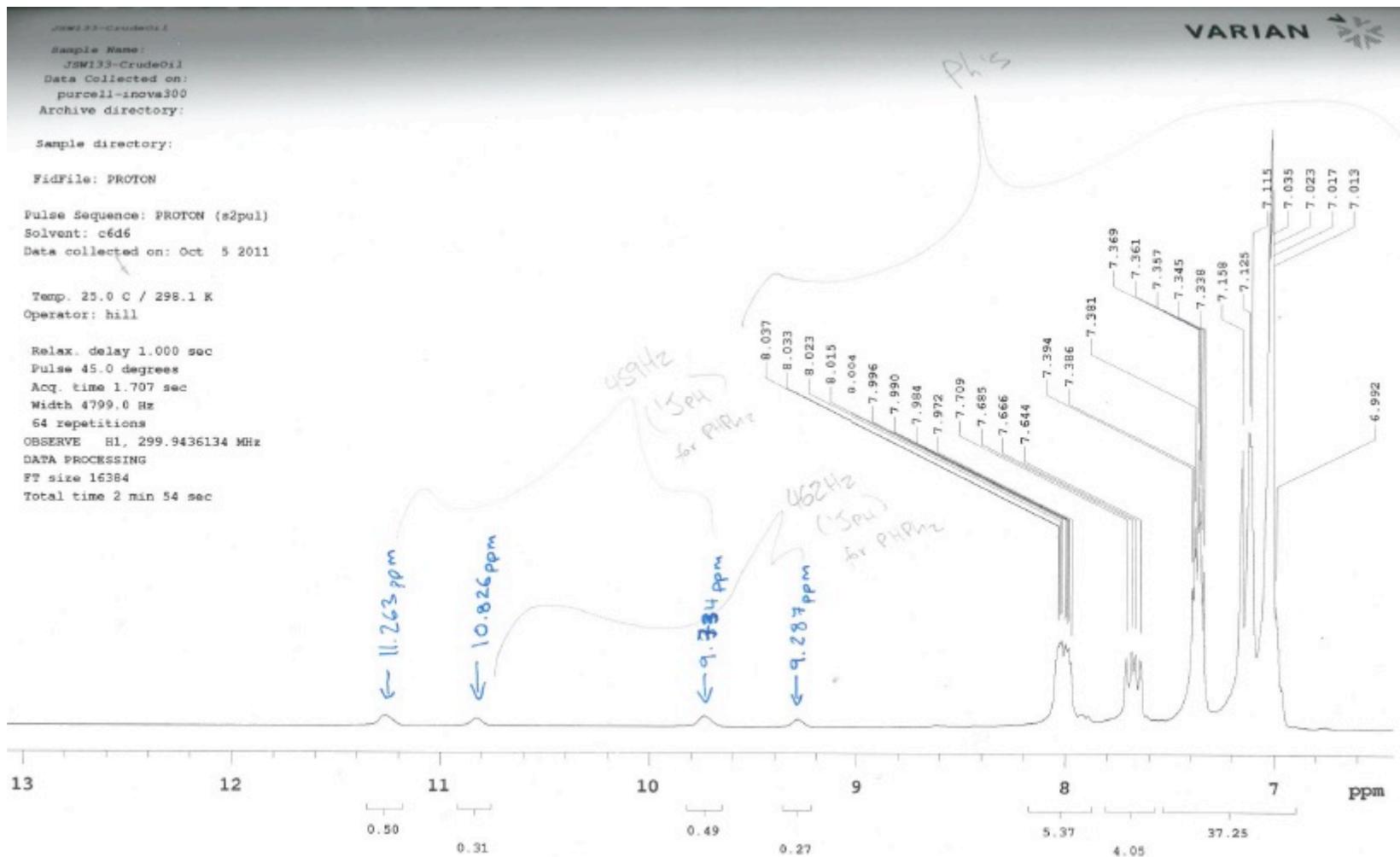
Compound 5 – ¹¹B NMR spectrum



Compound 5 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 5 – ^{31}P NMR spectrum



Compound 6 – ^1H NMR spectrum

JGW133-CrudeOil
 Sample Name:
 JGW133-CrudeOil
 Data Collected on:
 purcell-inova300
 Archive directory:

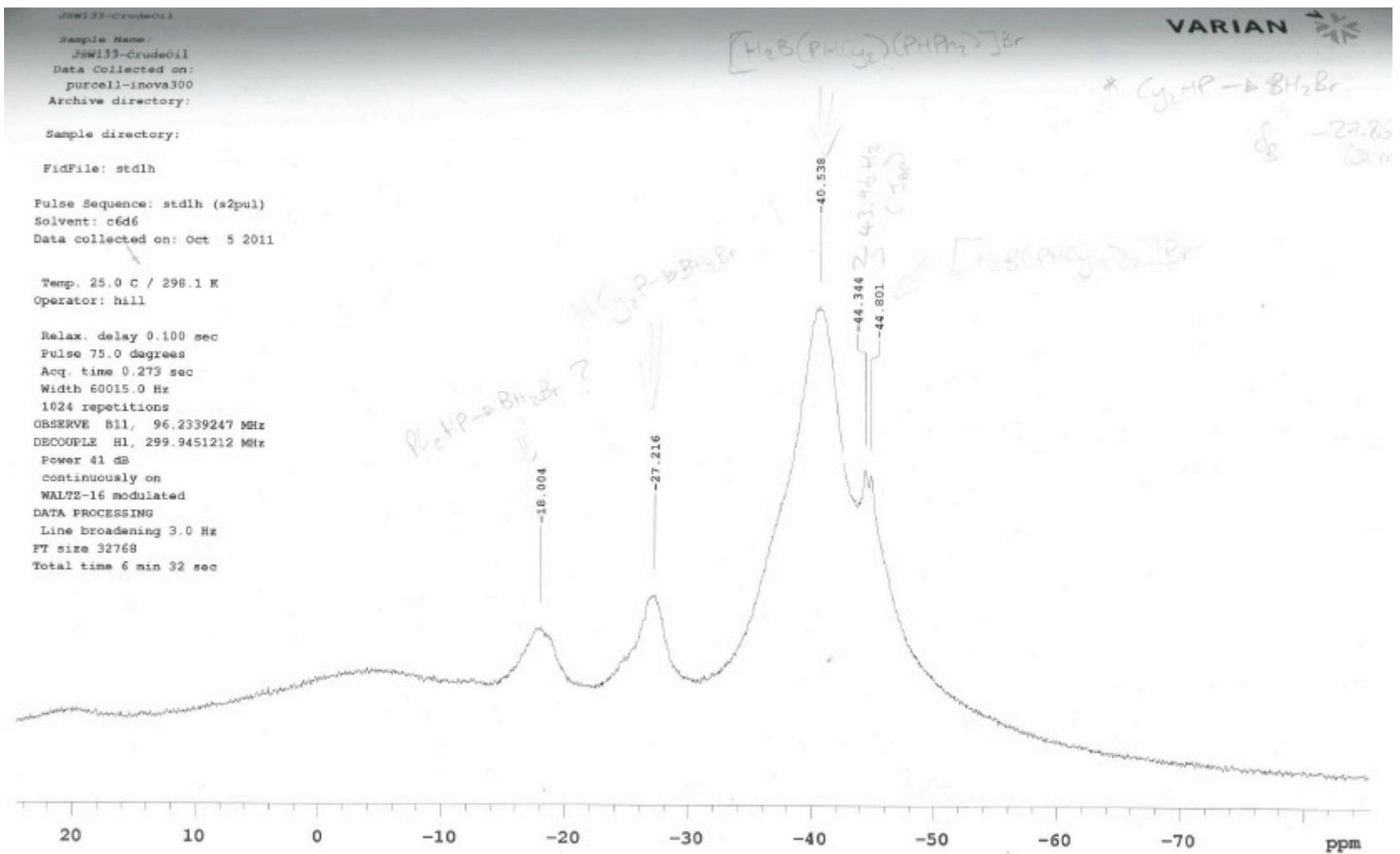
 Sample directory:

 FidFile: stdlh

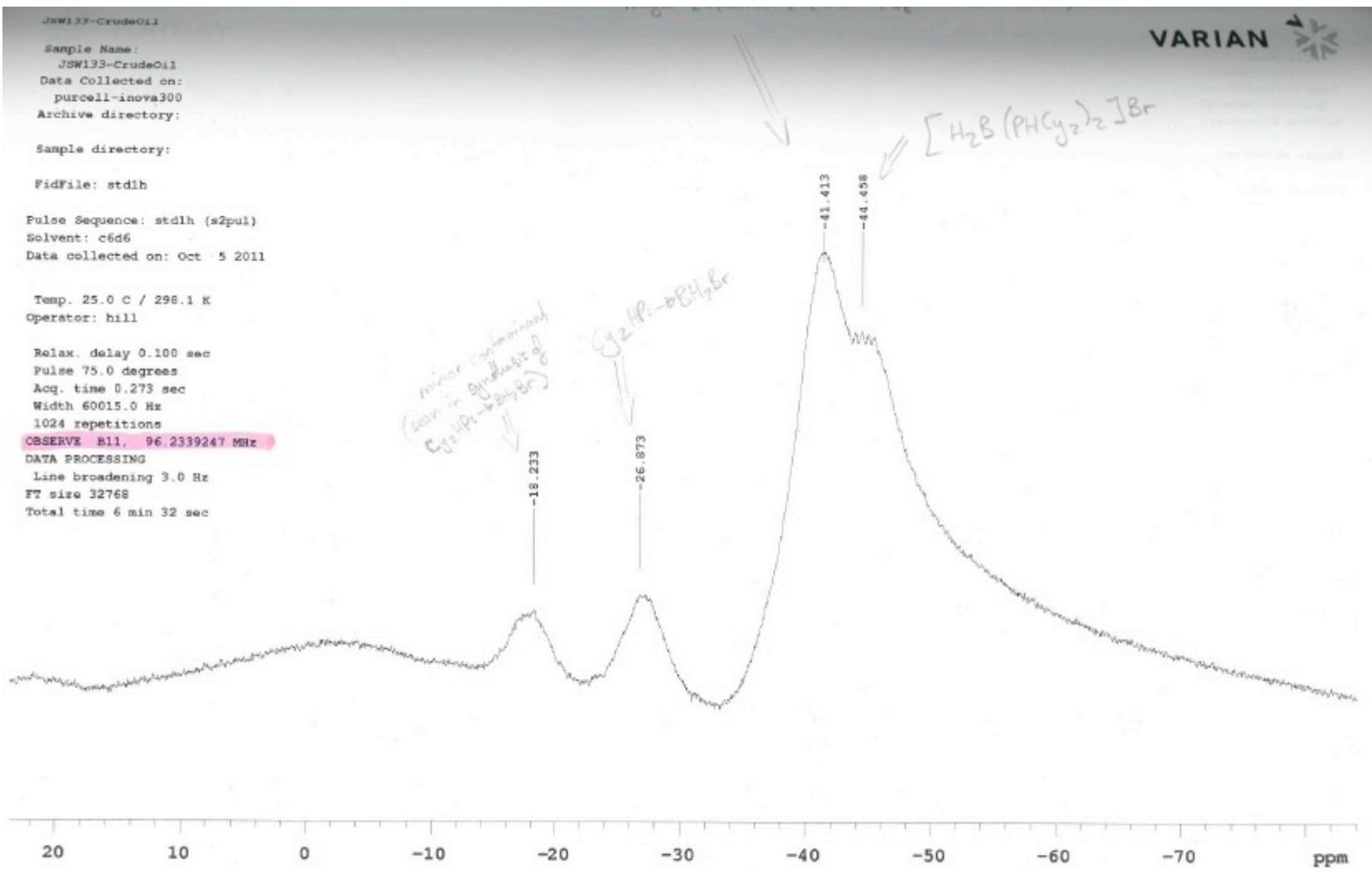
 Pulse Sequence: stdlh (s2pul)
 Solvent: c6d6
 Data collected on: Oct 5 2011

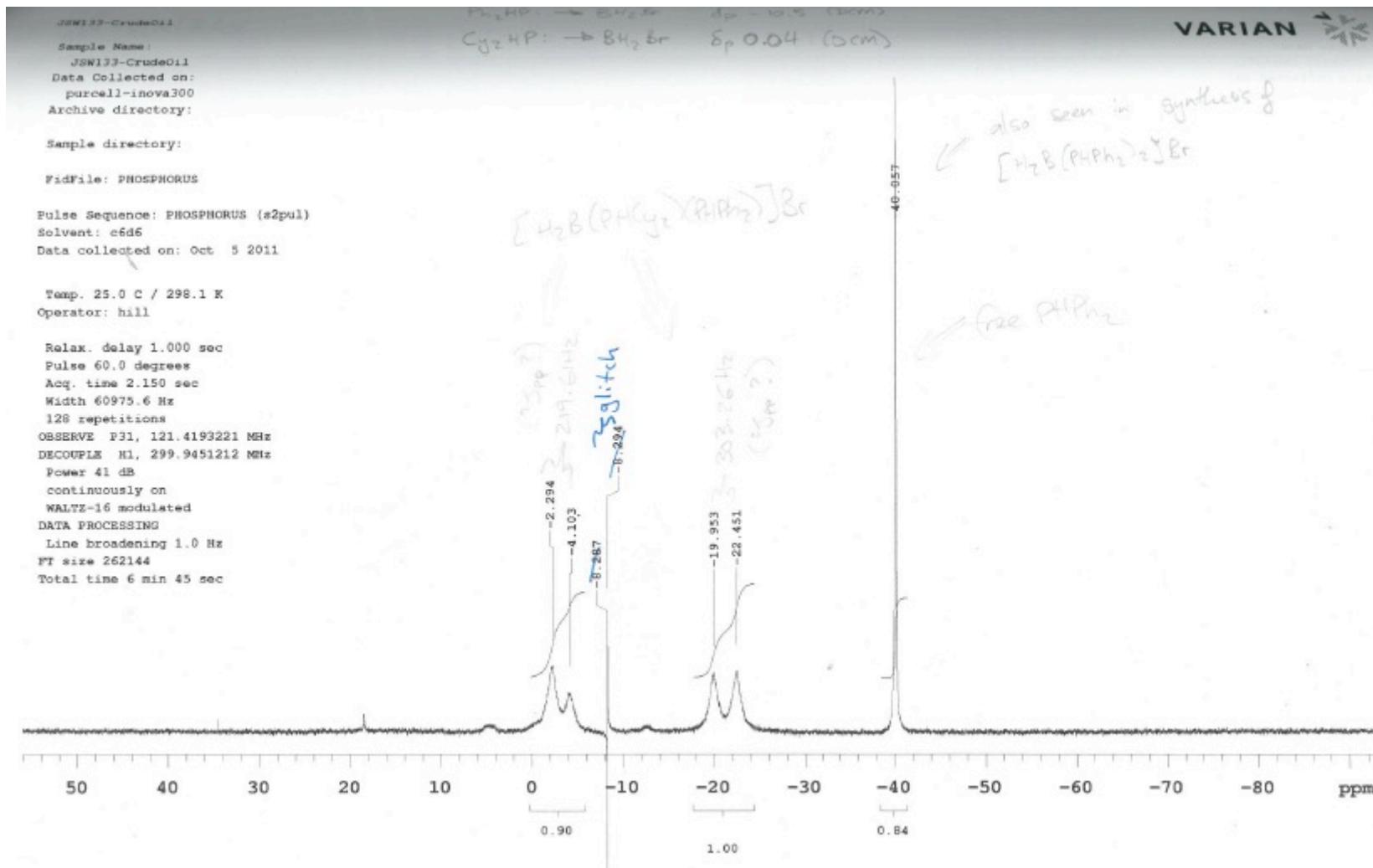
 Temp. 25.0 C / 298.1 K
 Operator: hill

 Relax. delay 0.100 sec
 Pulse 75.0 degrees
 Acq. time 0.273 sec
 Width 60015.0 Hz
 1024 repetitions
 OBSERVE B11, 96.2339247 MHz
 DECOUPLE H1, 299.9451212 MHz
 Power 41 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 32768
 Total time 6 min 32 sec

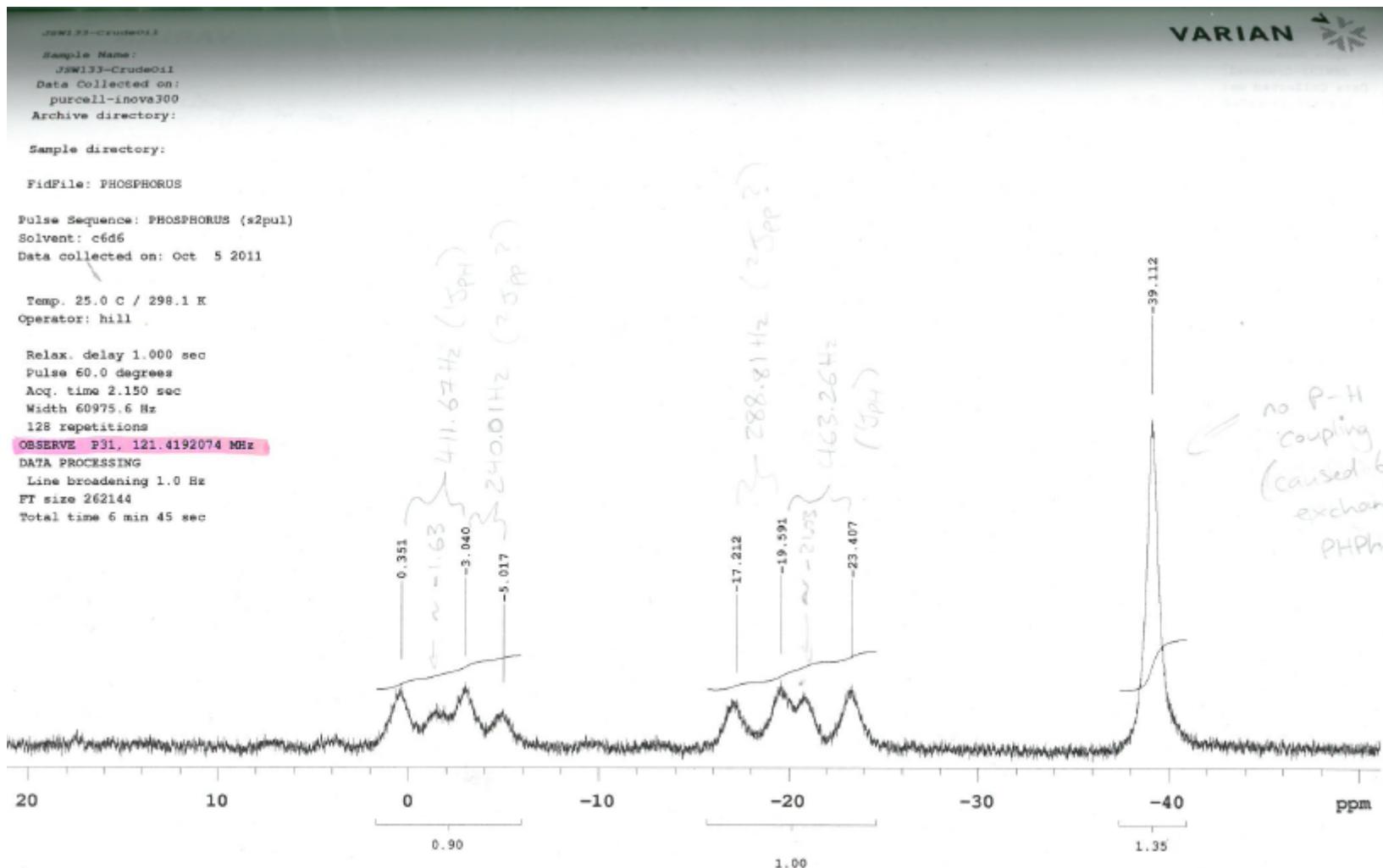


Compound 6 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum





Compound 6 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 6 – ^{31}P NMR spectrum

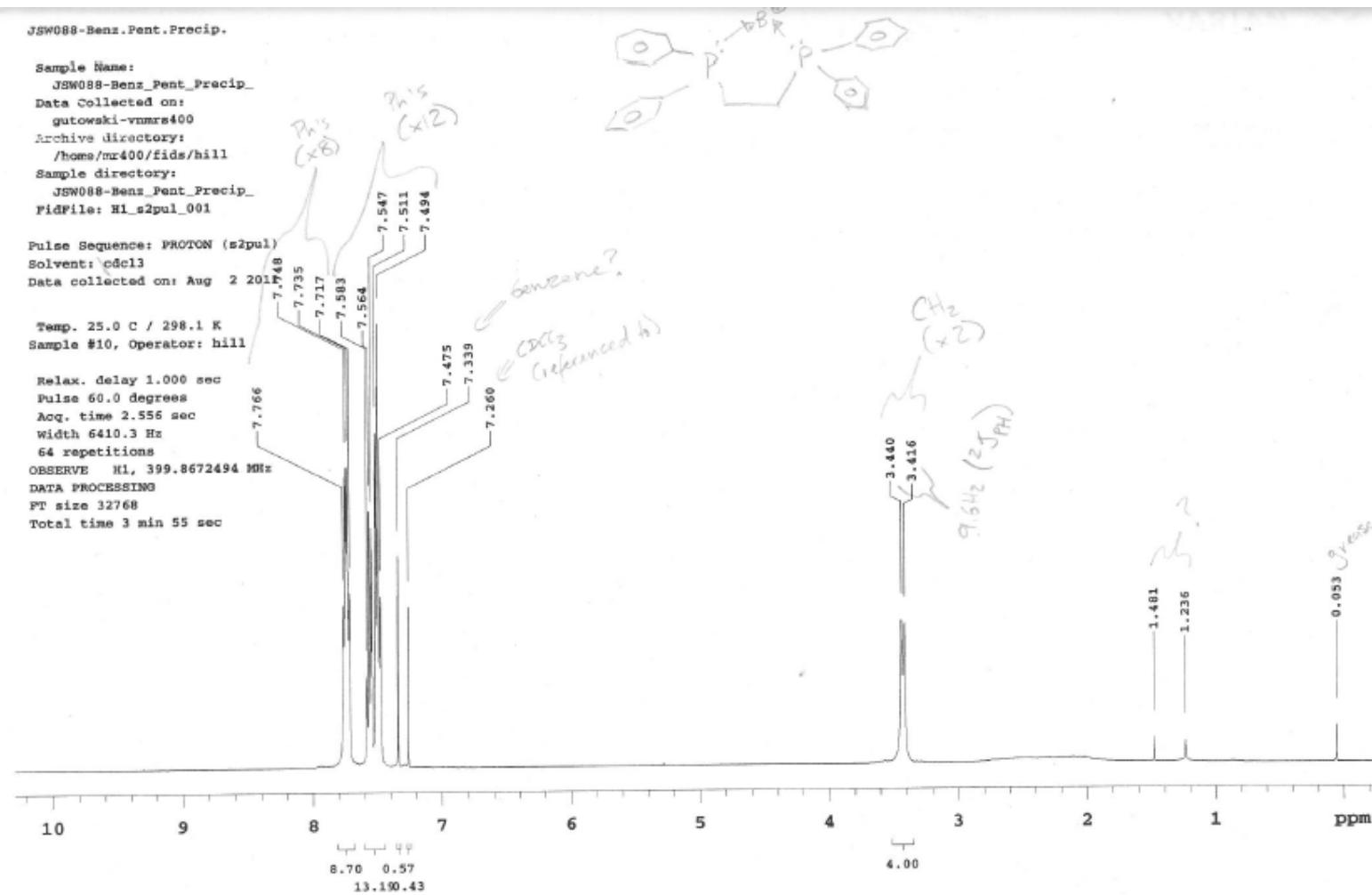
JSW088-Benz.Pent.Precip.

Sample Name:
JSW088-Benz_Pent_Precip.
Data Collected on:
gutowski-vnmrs400
Archive directory:
/home/mr400/fids/hill
Sample directory:
JSW088-Benz_Pent_Precip_
FidFile: H1_s2pul_001

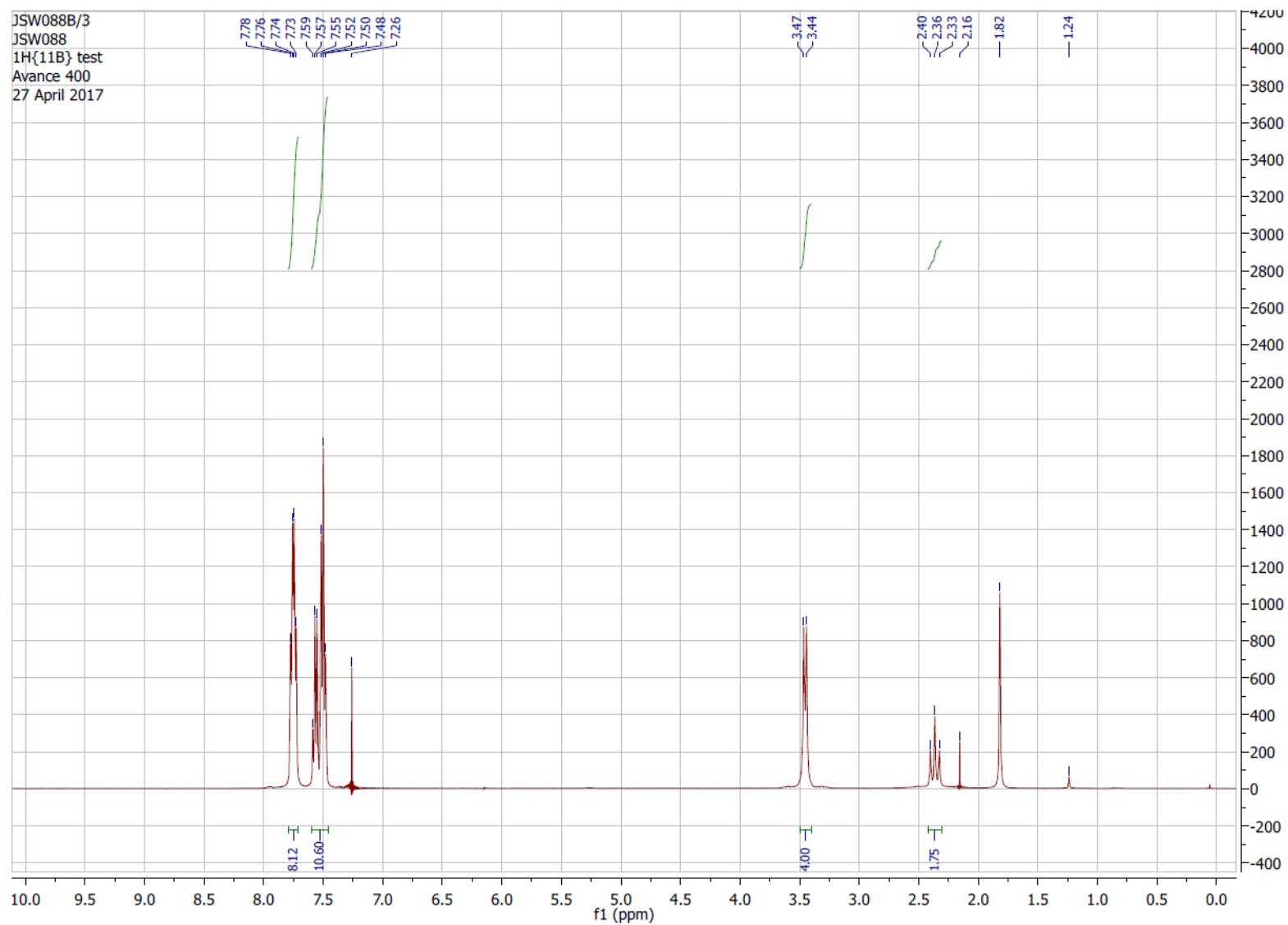
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 2 2011

Temp. 25.0 C / 298.1 K
Sample #10, Operator: hill

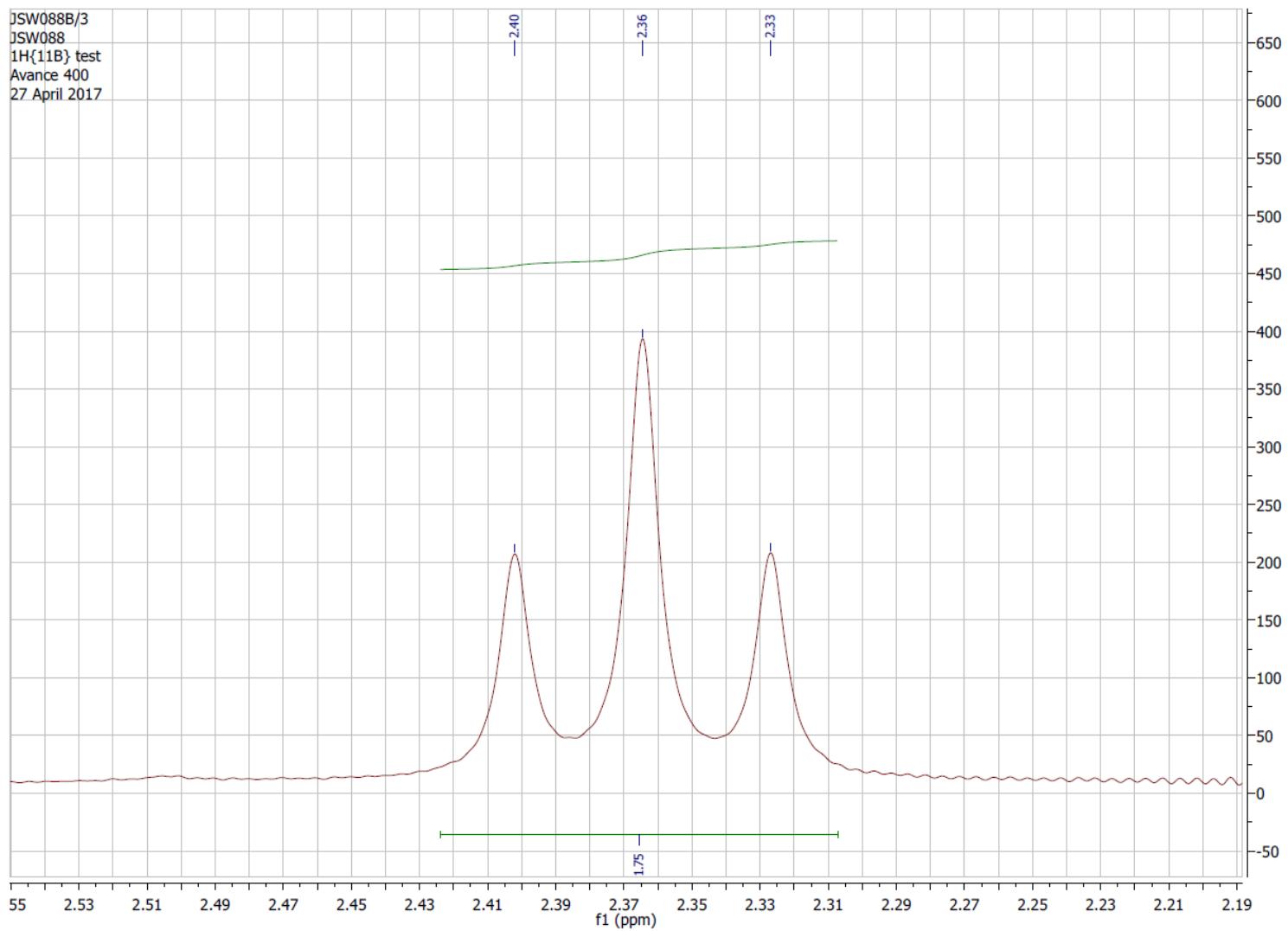
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.555 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.8672494 MHz
DATA PROCESSING
FT size 32768
Total time 3 min 55 sec



Compound 7 – ^1H NMR spectrum



Compound 7 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum



Compound 7 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonances)

JSW088-Benz_Pent_Precip.

VARIAN

Sample Name:
JSW088-Benz_Pent_Precip

Data Collected on:
purcell-inova300
Archive directory:

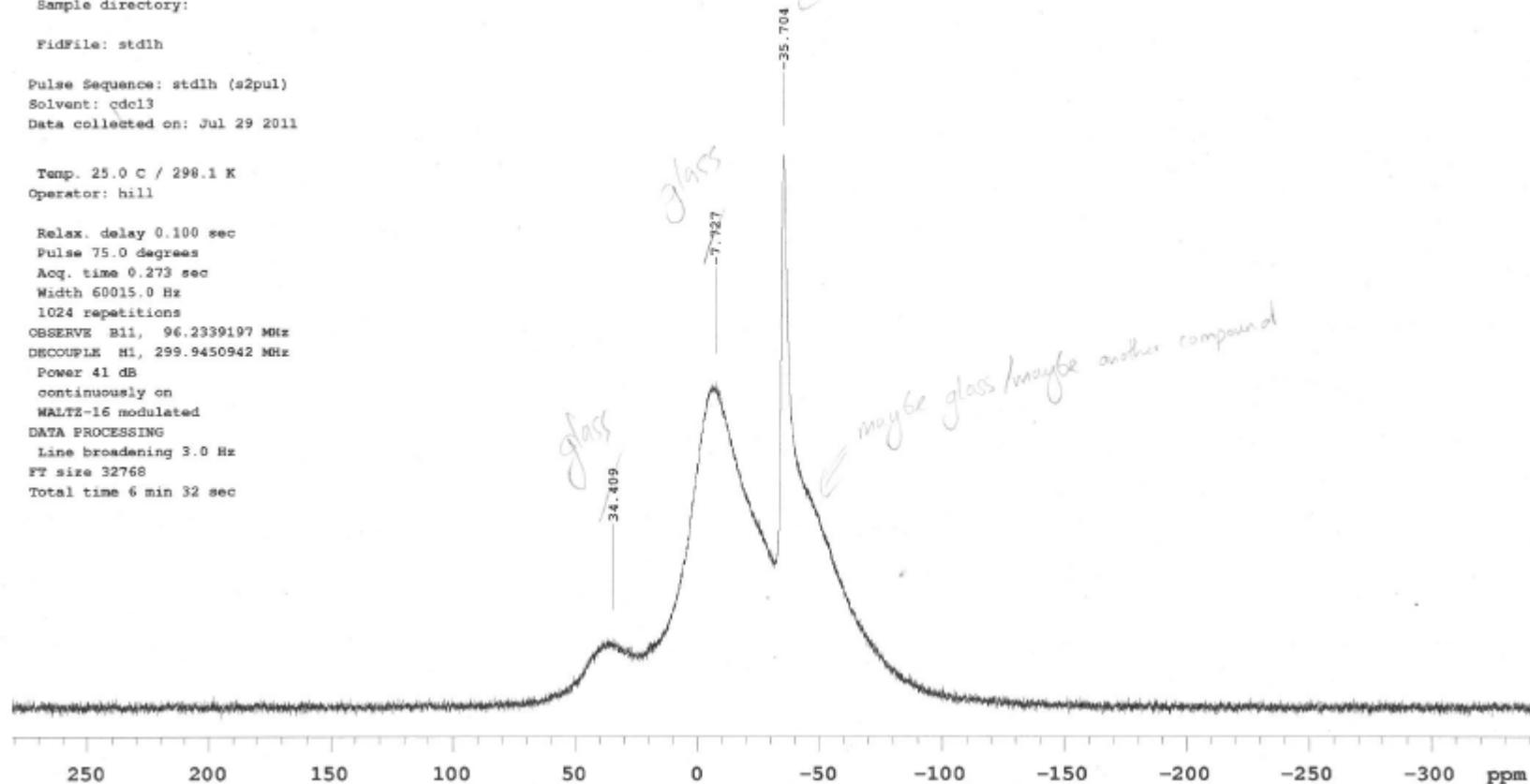
Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2011

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1024 repetitions
OBSERVE B11, 96.2339197 MHz
DECOUPLE H1, 299.9450942 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 6 min 32 sec



Compound 7 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

JSW088-Benz_Pent_Precip.

VARIAN

Sample Name:
JSW088-Benz_Pent_Precip.

Data Collected on:
purcell-inova300
Archive directory:

Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)

Solvent: cdcl3

Data collected on: Jul 29 2011

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 0.100 sec

Pulse 75.0 degrees

Acq. time 0.273 sec

Width 60015.0 Hz

2000 repetitions

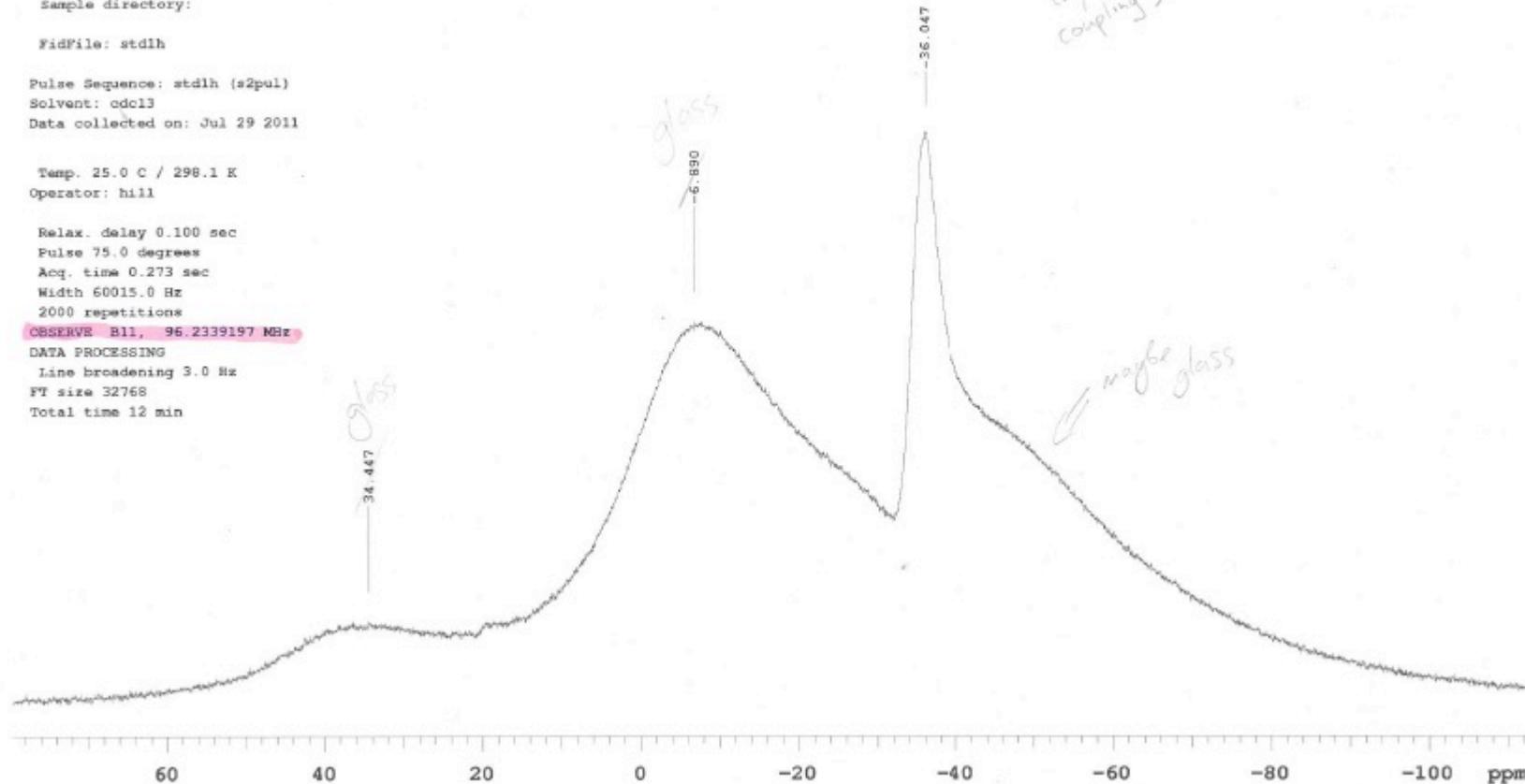
OBSERVE B11, 96.2339197 MHz

DATA PROCESSING

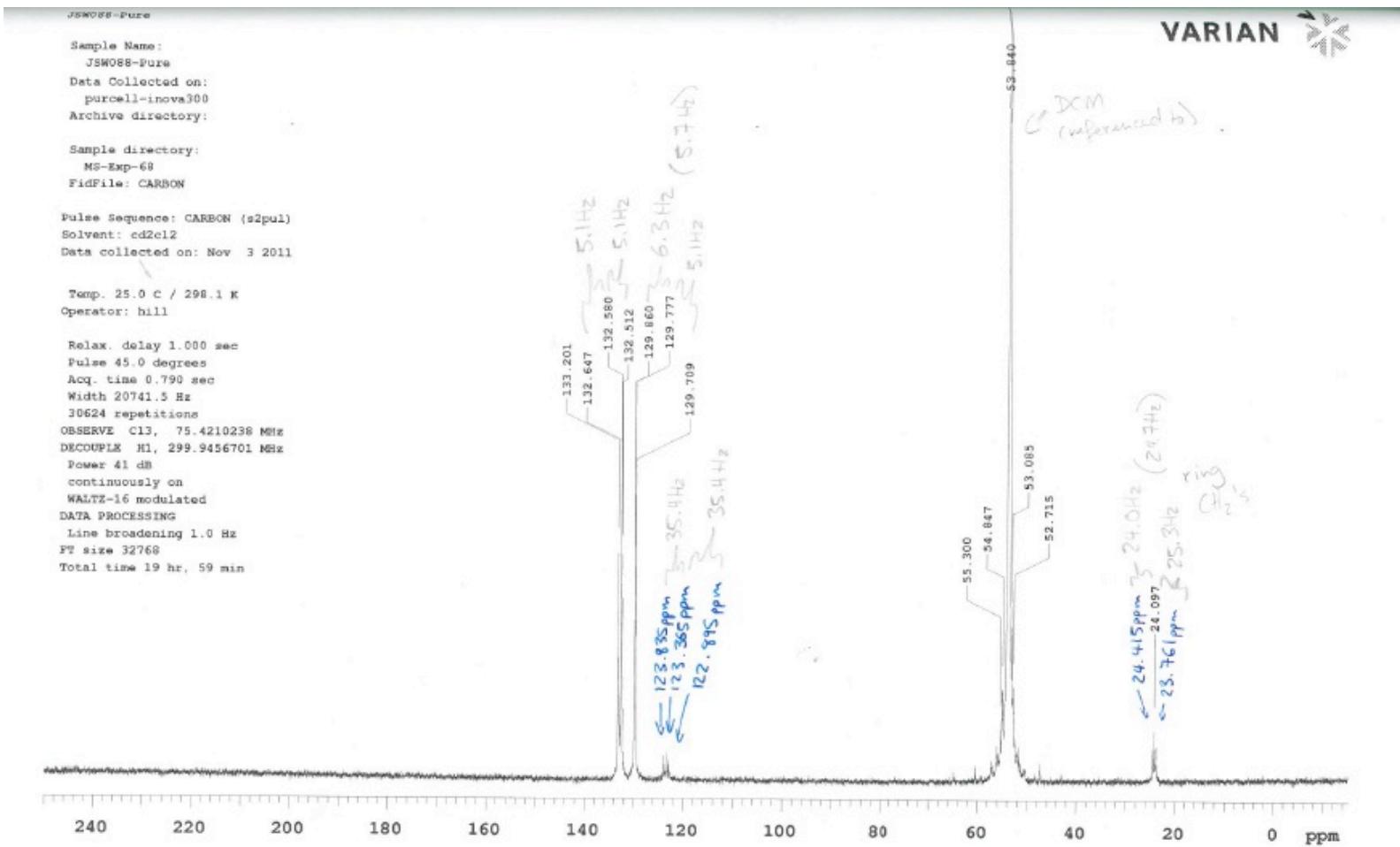
Line broadening 3.0 Hz

FT size 32768

Total time 12 min



Compound 7 – ¹¹B NMR spectrum



Compound 7 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

JSW088-Benz_Pent.Precip.

VARIAN

Sample Name:
JSW088-Benz_Pent_Precip_

Data Collected on:
purcell-inova300

Archive directory:

Sample directory:

FidFile: PHOSPHORUS

Pulse Sequence: PHOSPHORUS (s2pul)

Solvent: cdc13

Data collected on: Jul 29 2011

Temp. 25.0 C / 298.1 K

Operator: bill

Relax. delay 1.000 sec

Pulse 60.0 degrees

Acq. time 2.150 sec

Width 60975.6 Hz

256 repetitions

OBSERVE P31, 121.4192923 MHz

DECOUPLE H1, 299.9450942 MHz

Power 41 dB

continuously on

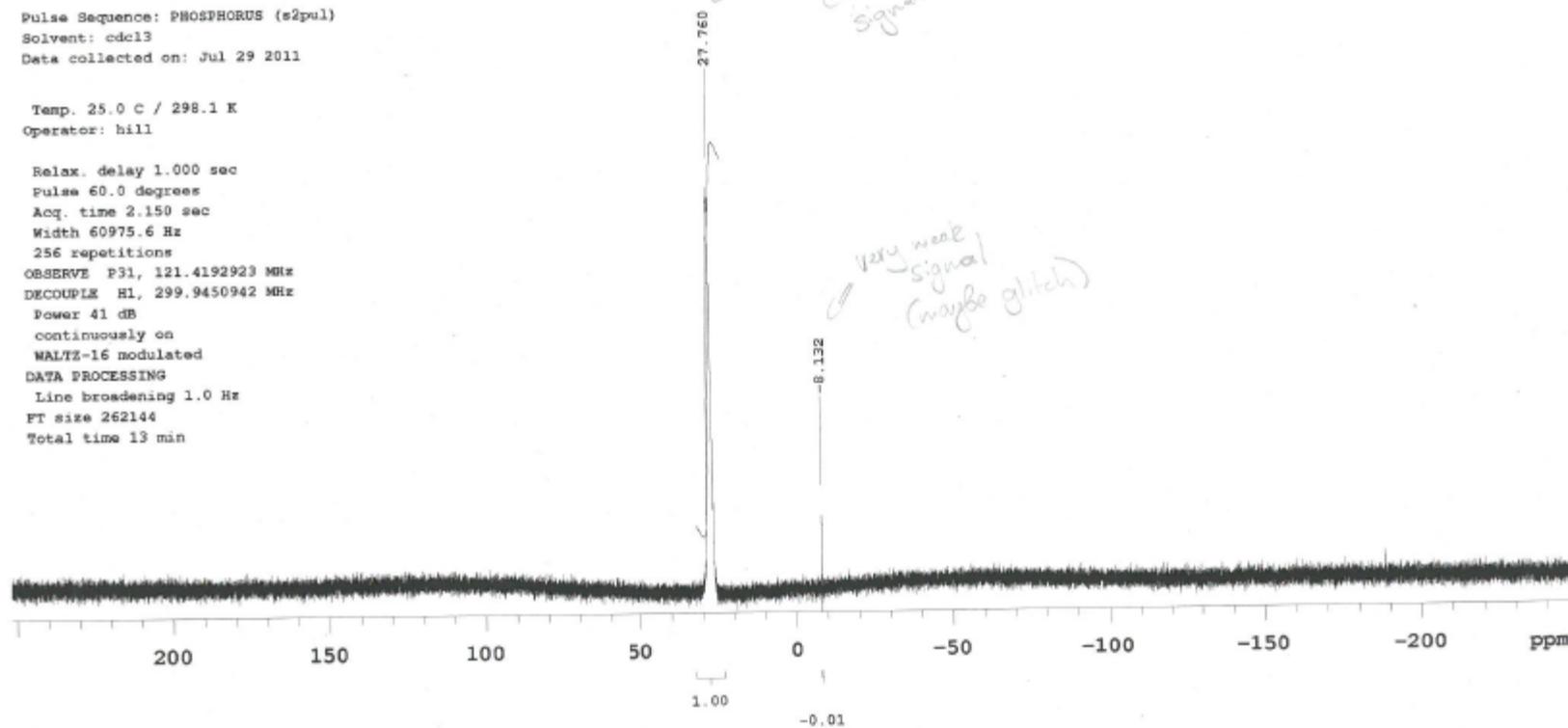
MALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 262144

Total time 13 min



Compound 7 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

JSM179-crystals

Sample Name:
JSM179-Crystals
Data Collected on:
purcell-inova300
Archive directory:

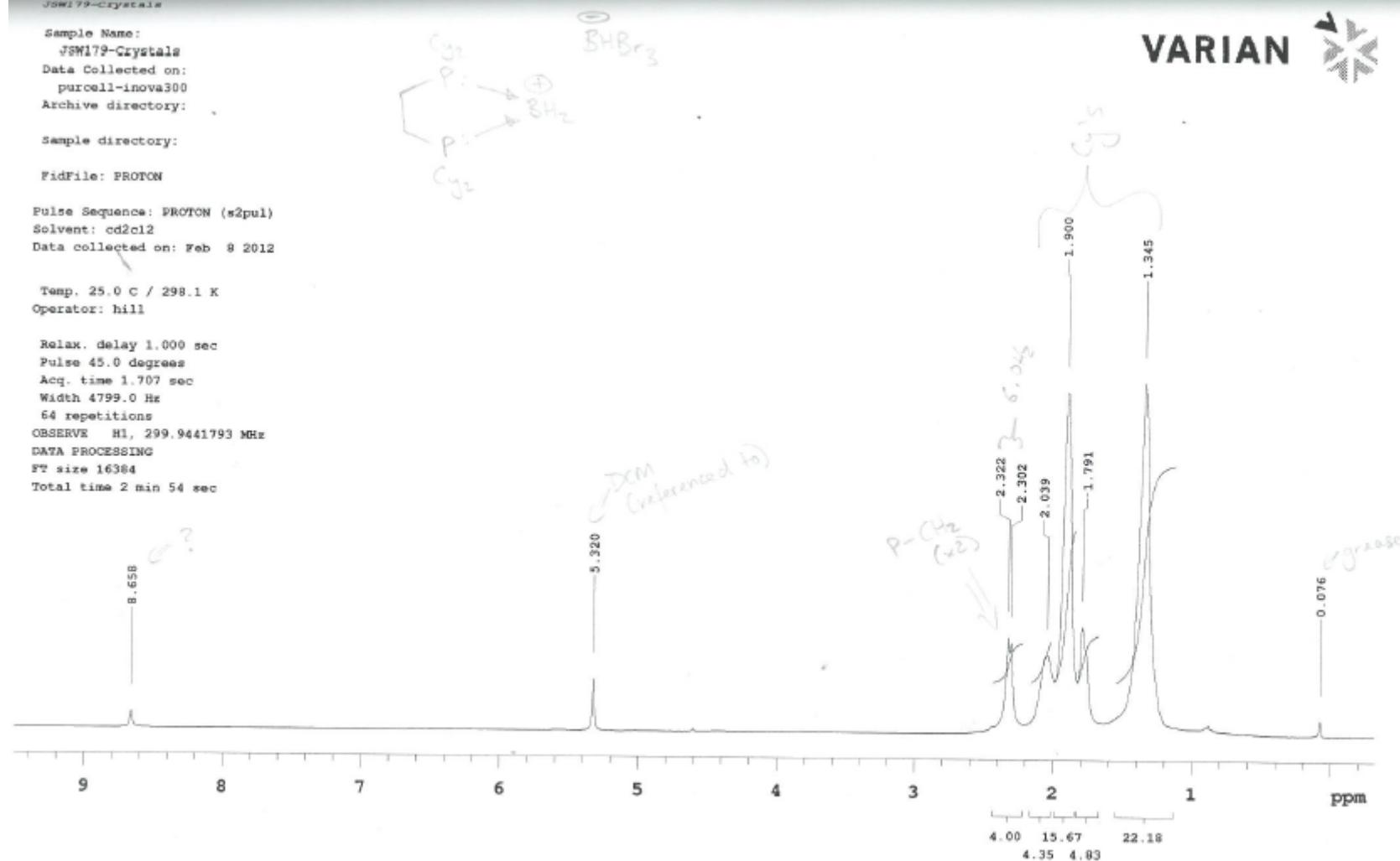
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cd2cl2
Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.707 sec
Width 4799.0 Hz
64 repetitions
OBSERVE H1, 299.9441793 MHz
DATA PROCESSING
FT size 16384
Total time 2 min 54 sec



Compound 8 – ¹H NMR spectrum

JSW179-Crystals

Sample Name:

JSW179-Crystals

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (*2pul)

Solvent: cd2cl2

Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 0.100 sec

Pulse 75.0 degrees

Acq. time 0.273 sec

Width 60015.0 Hz

1024 repetitions

OBSERVE B11, 96.2341081 MHz

DECOPPLE 1H, 299.9456701 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

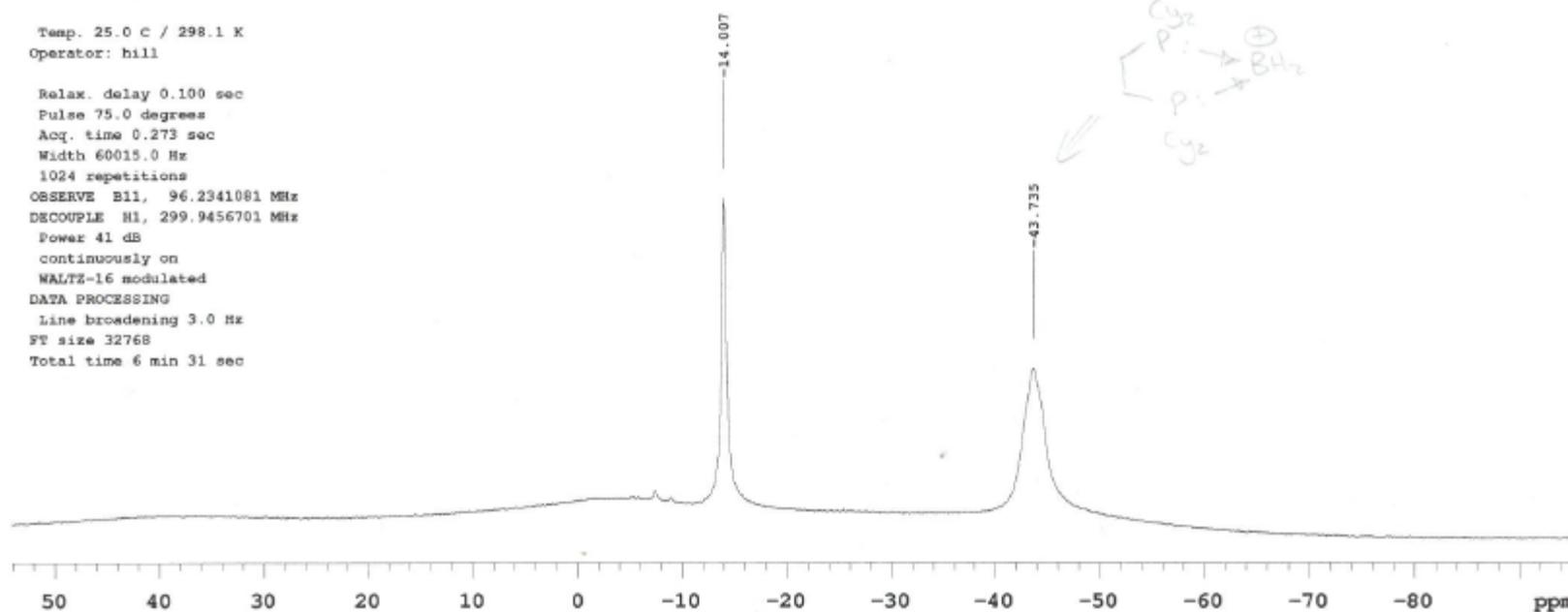
DATA PROCESSING

Line broadening 3.0 Hz

FT size 32768

Total time 6 min 31 sec

VARIAN



Compound 8 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

JSW179-Crystals
Sample Name:
JSW179-Crystals
Data Collected on:
percell-inova300
Archive directory:

Sample directory:

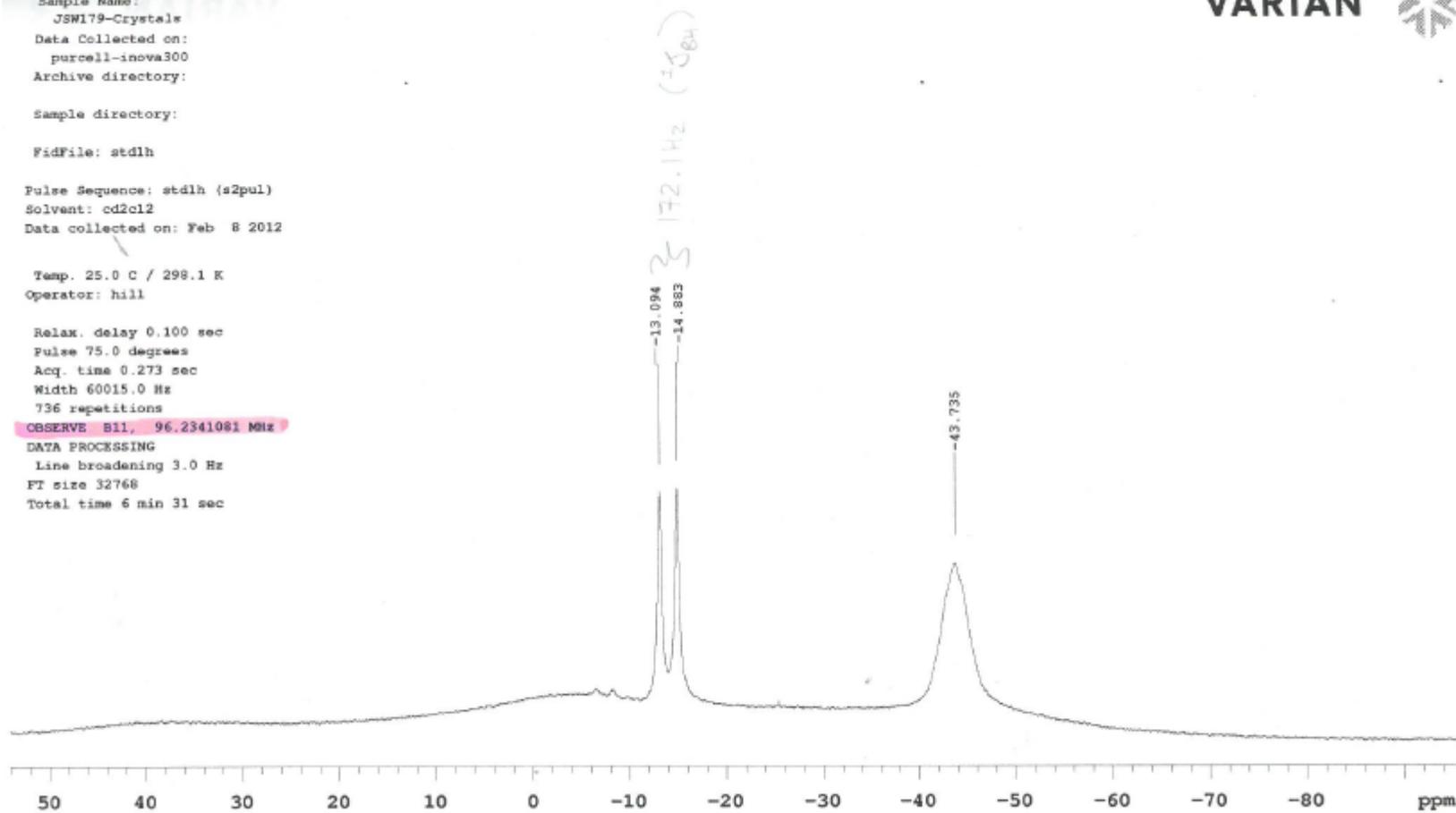
Fidfile: stdlh

Pulse Sequence: stdlh (s2pul)
Solvent: cd2cl2
Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
736 repetitions
OBSERVE B11, 96.2341081 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 6 min 31 sec

VARIAN



Compound 8 – ¹¹B NMR spectrum

JSW179-Crystals

Sample Name:

JSW179-Crystals

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)

Solvent: cd2cl2

Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K

Operator: bill

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 0.790 sec

Width 20741.5 Hz

2088 repetitions

OBSERVE C13, 75.4209934 MHz

DISCOUPLE H1, 299.9456701 MHz

Power 41 dB

continuously on

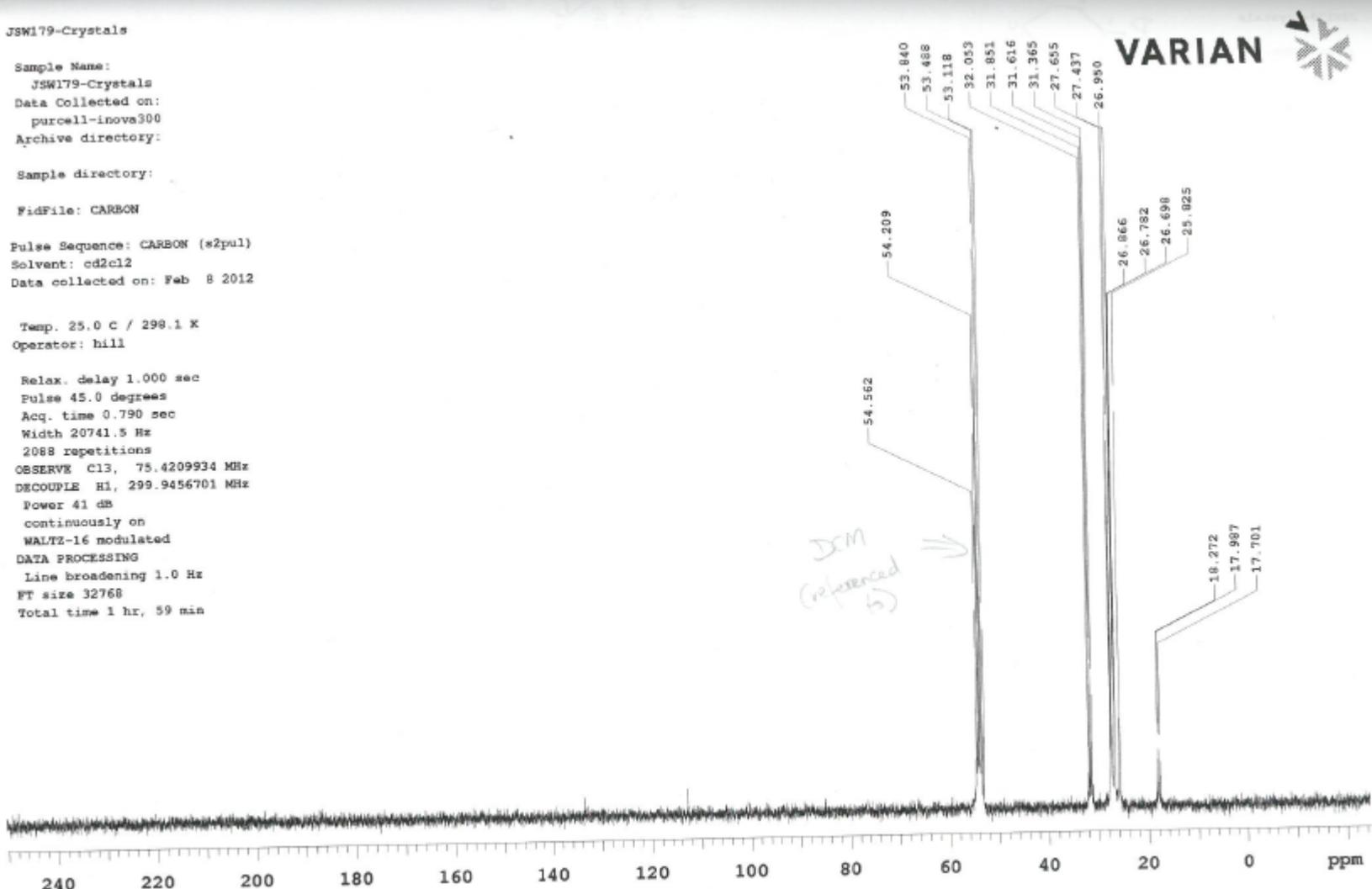
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 1 hr, 59 min



Compound 8 – ¹³C{¹H} NMR spectrum

JSW179-Crystals

Sample Name:

JSW179-Crystals

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: PHOSPHORUS

Pulse Sequence: PHOSPHORUS (s2pul)

Solvent: cd2c12

Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 1.000 sec

Pulse 60.0 degrees

Acq. time 2.150 sec

Width 60975.6 Hz

128 repetitions

OBSERVE P31, 121.4195536 MHz

DECOUPLE H1, 299.9456701 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

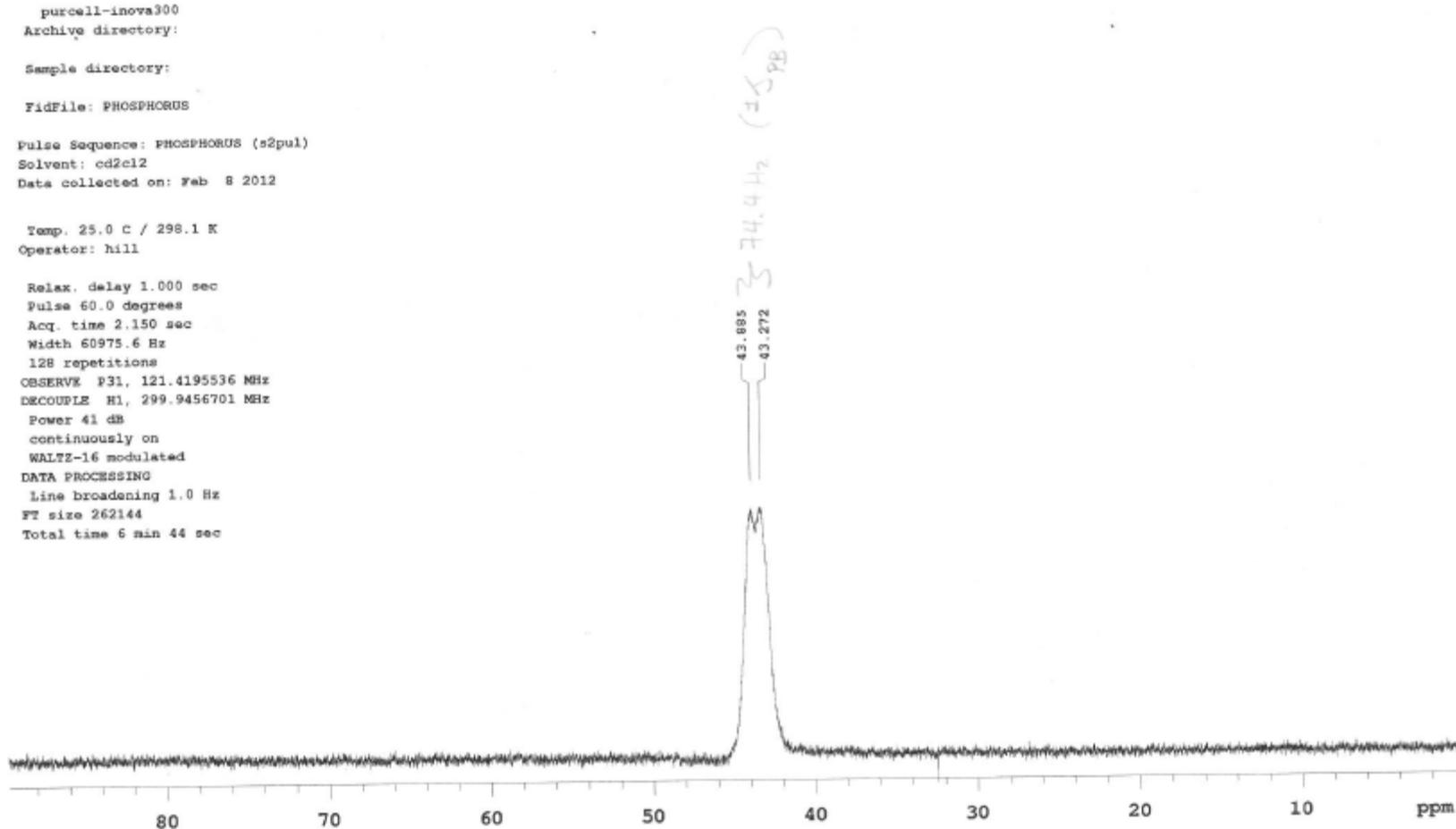
DATA PROCESSING

Line broadening 1.0 Hz

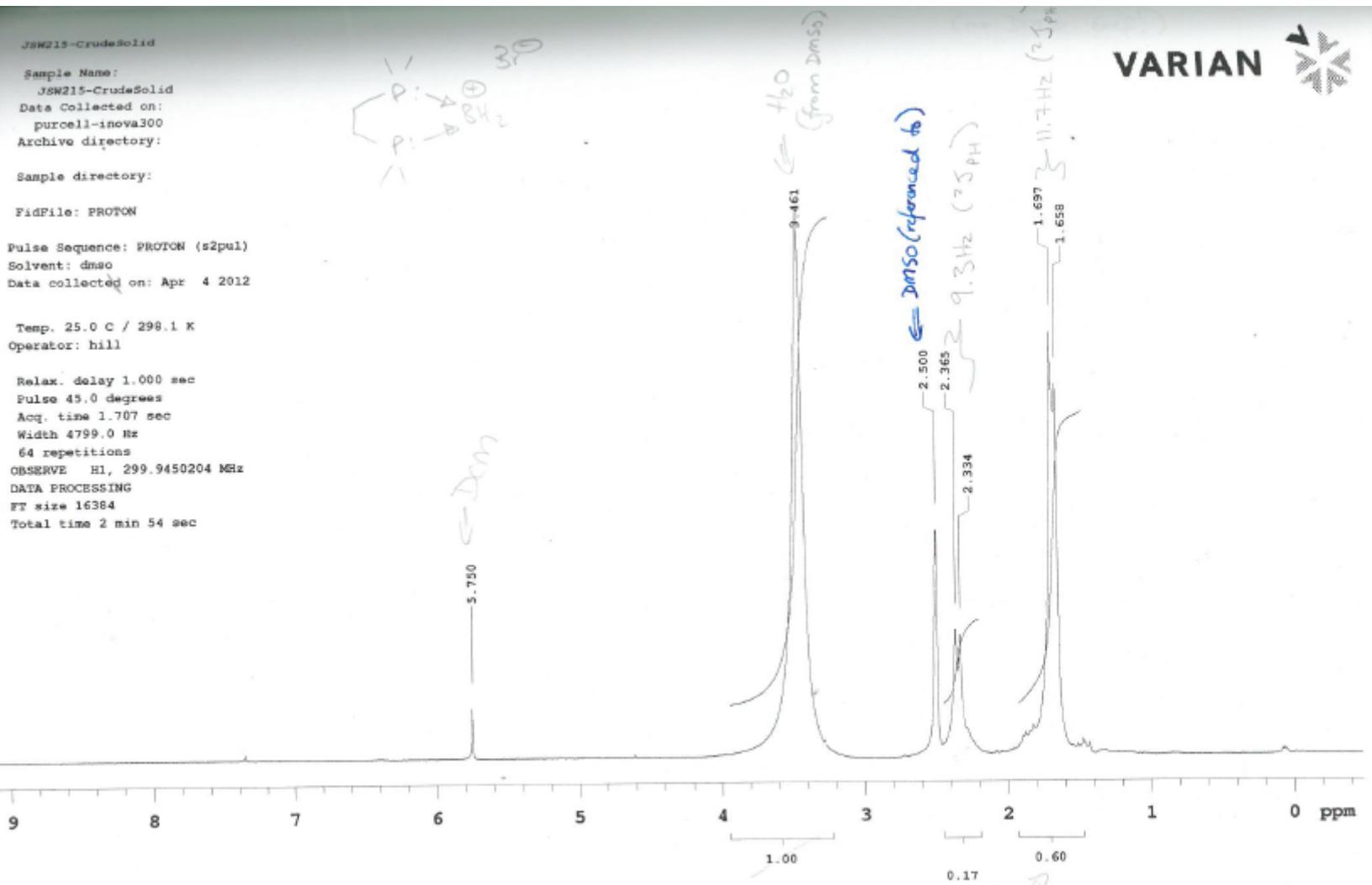
FT size 262144

Total time 6 min 44 sec

VARIAN



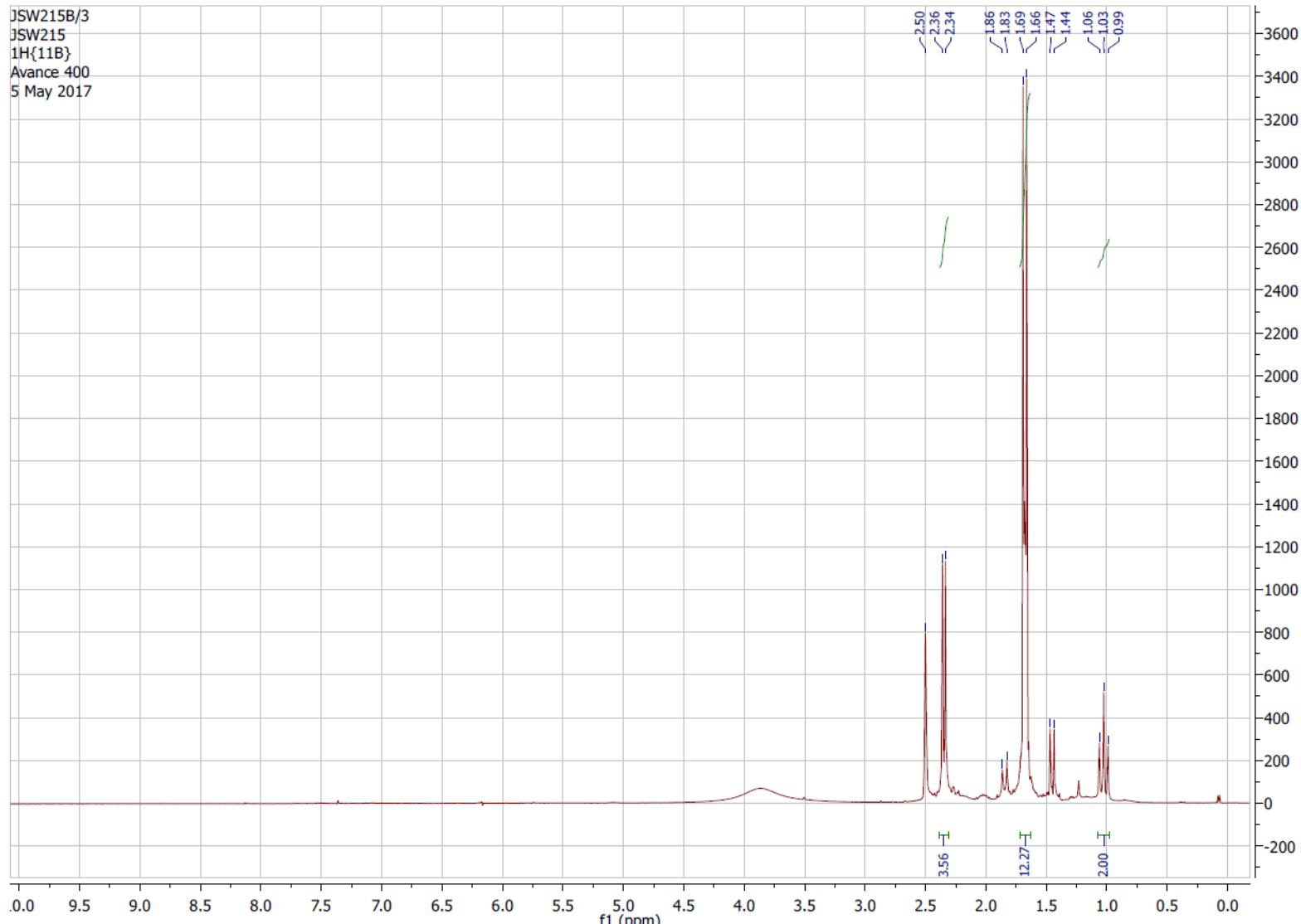
Compound 8 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



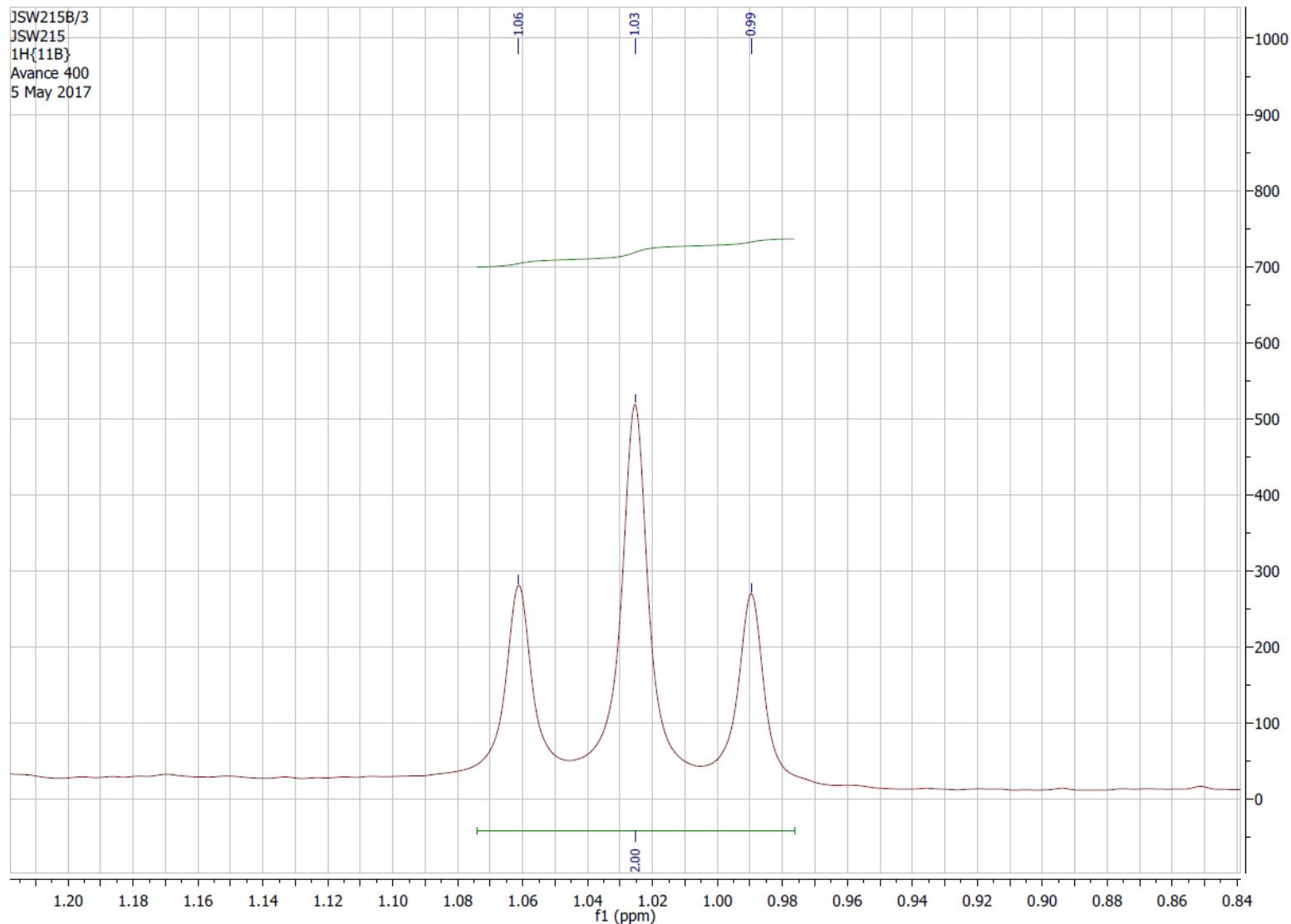
Compound 9 – ^1H NMR spectrum

VARIAN

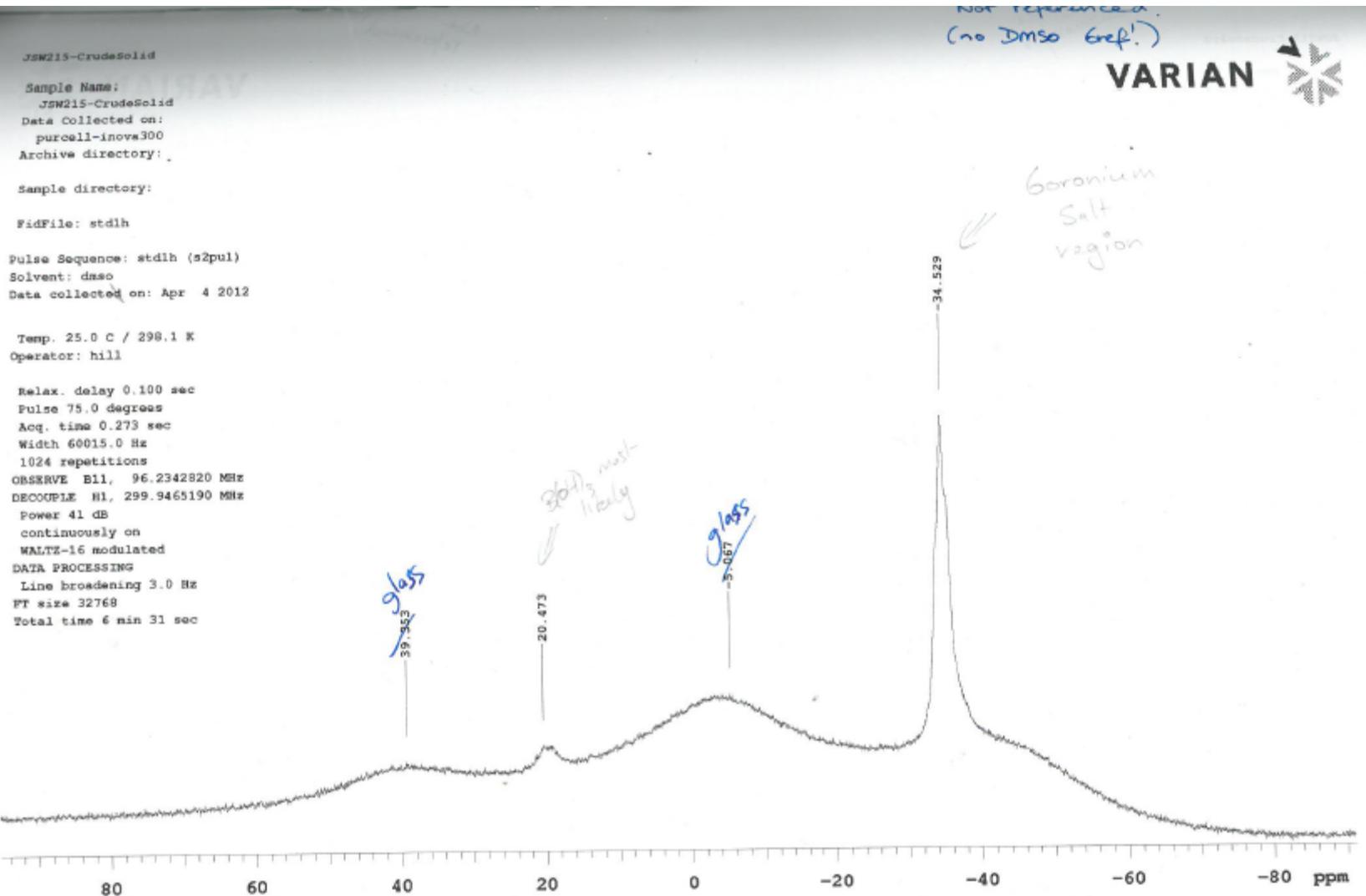




Compound 9 – ¹H{¹¹B} NMR spectrum



Compound 9 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)



Compound 9 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

Sample Name: JSW215-CrudeSolid
Data Collected on: purcell-inova300
Archive directory:

Sample directory:

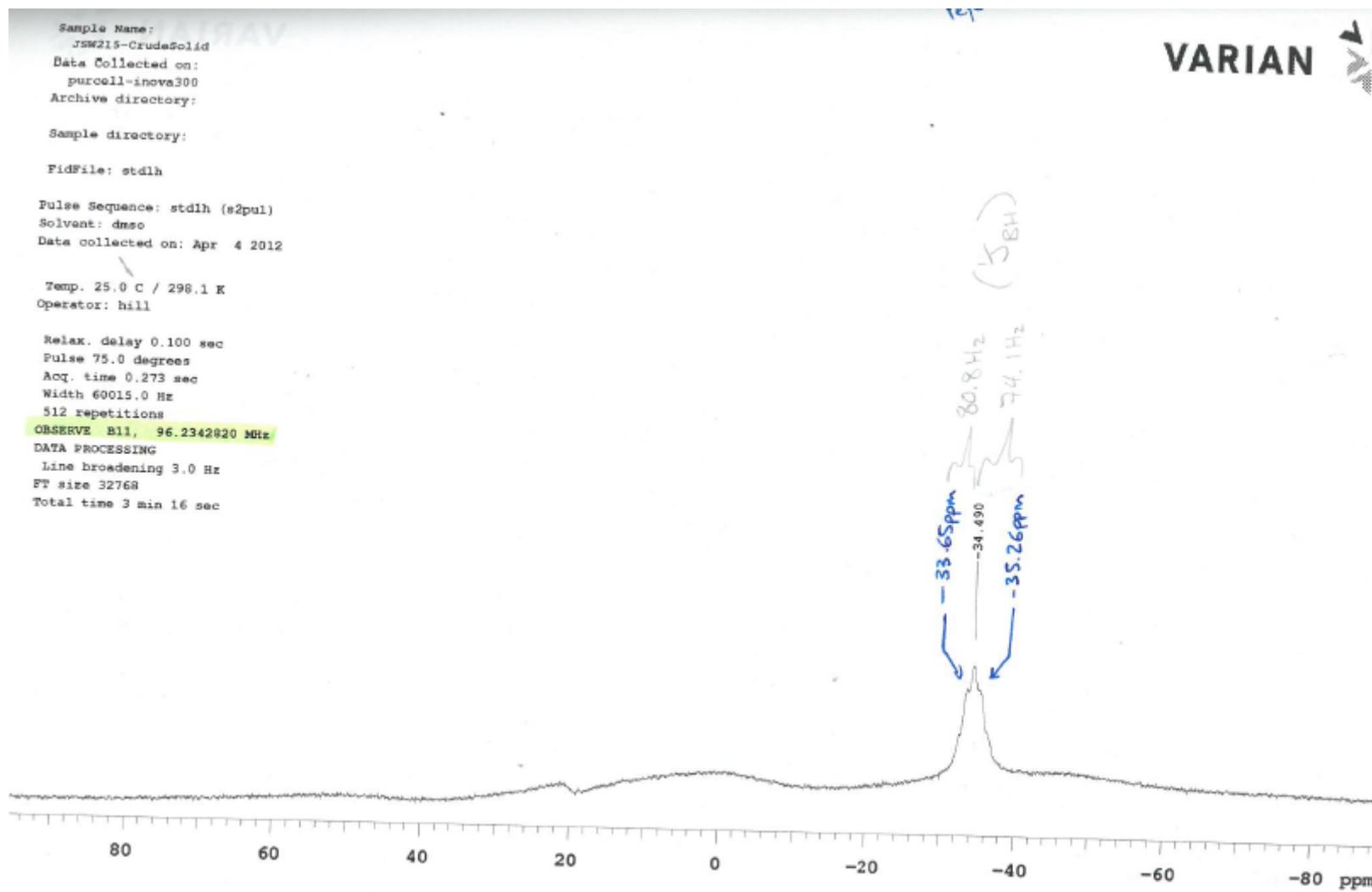
FidFile: stdlh

Pulse Sequence: stdlh (s2pul)
Solvent: dmsc
Data collected on: Apr 4 2012

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
512 repetitions
OBSERVE B11, 96.2342820 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 3 min 16 sec

VARIAN



Compound 9 – ^{11}B NMR spectrum

JSM215-CrudeSolid

Sample Name:
JSM215-CrudeSolid
Data Collected on:
purcell-inova300
Archive directory:

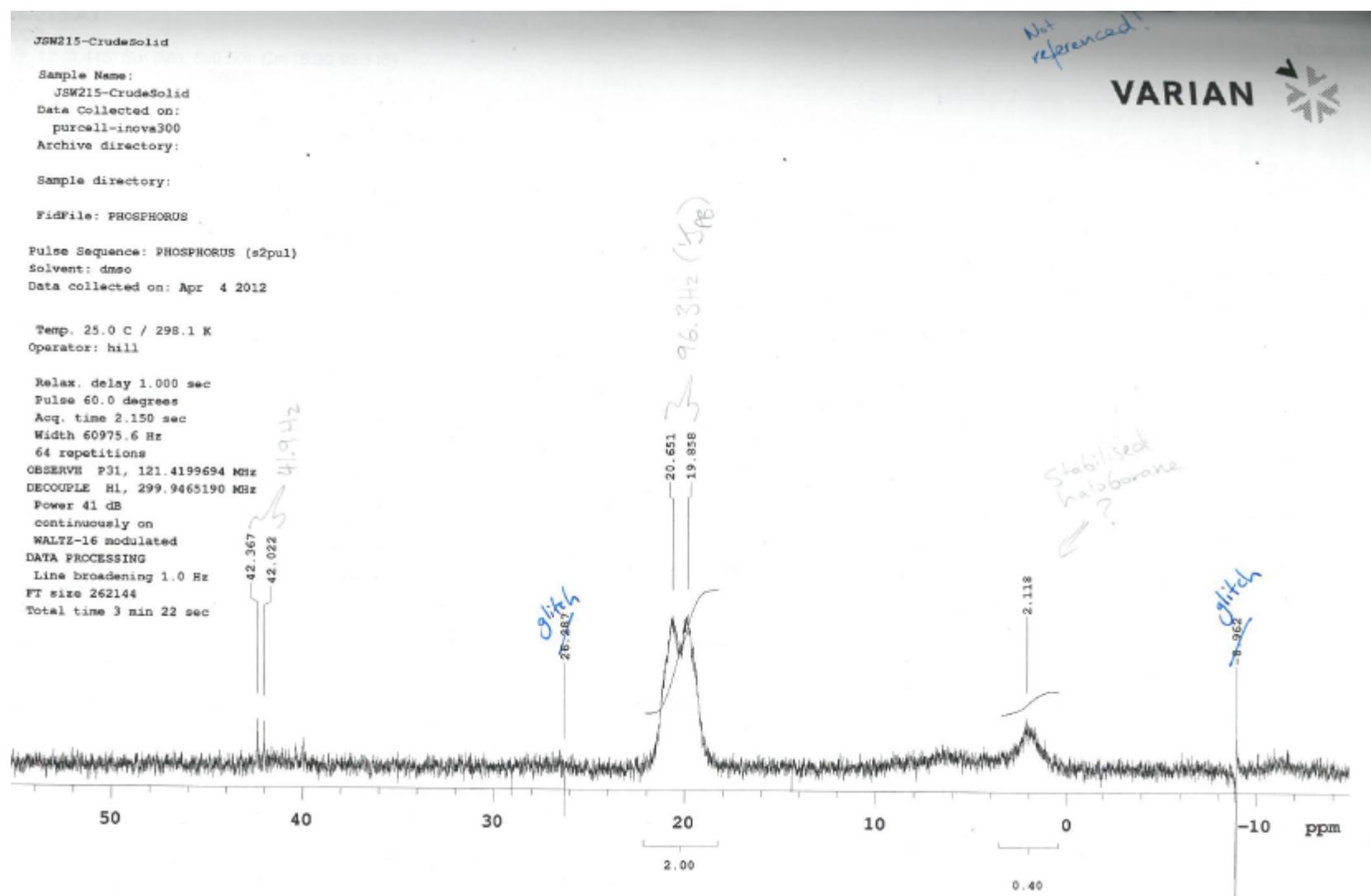
Sample directory:

FidFile: PHOSPHORUS

Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: dmso
Data collected on: Apr 4 2012

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.150 sec
Width 60975.6 Hz
64 repetitions
OBSERVE P31, 121.4199694 MHz
DECOUPLE H1, 299.9465190 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 262144
Total time 3 min 22 sec



Compound 9 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

JSW322-Precipitate
Sample Name:
JSW322-Precipitate
Data Collected on:
purcell-inova300
Archive directory:

Sample directory:

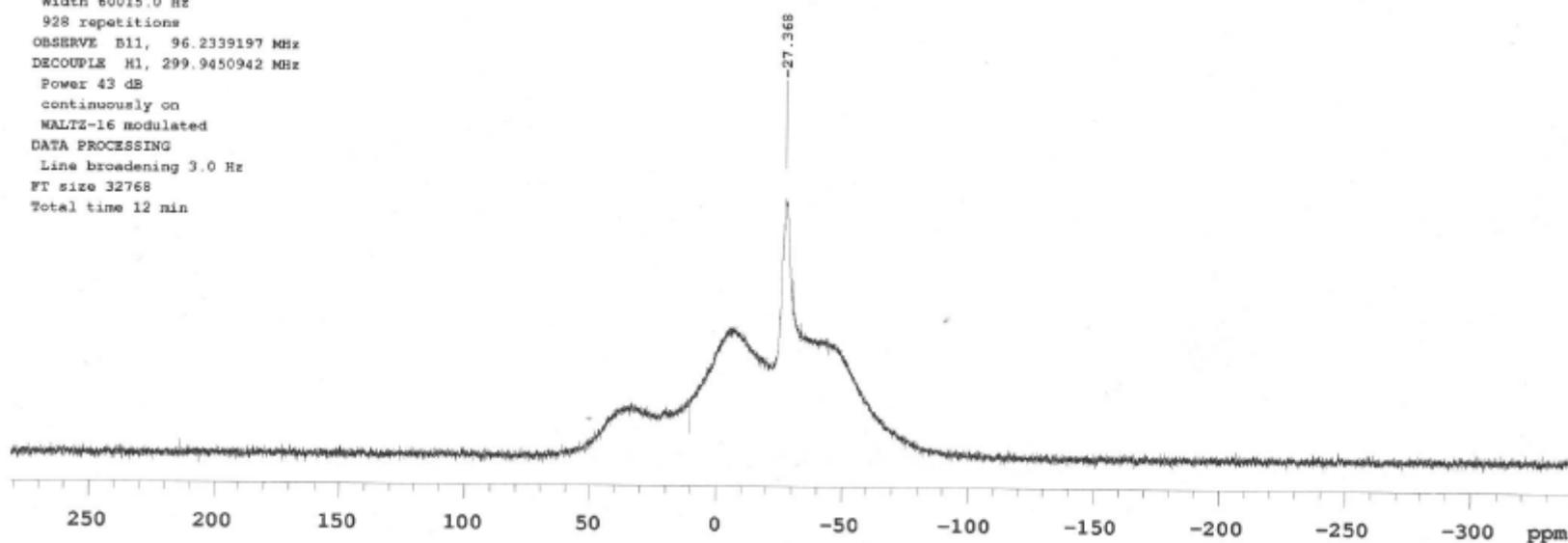
FidFile: stdlh

Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2013

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
928 repetitions
OBSERVE H1, 96.2339197 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
MALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 12 min

Fe
P ph₂ δ_B =
(mono or bis)
[JSW321/JSW322-44]



Compound 10 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

JSM322-Precipitate

Sample Name:
JSM322-Precipitate

Data Collected on:
purcell-inova300
Archive directory:

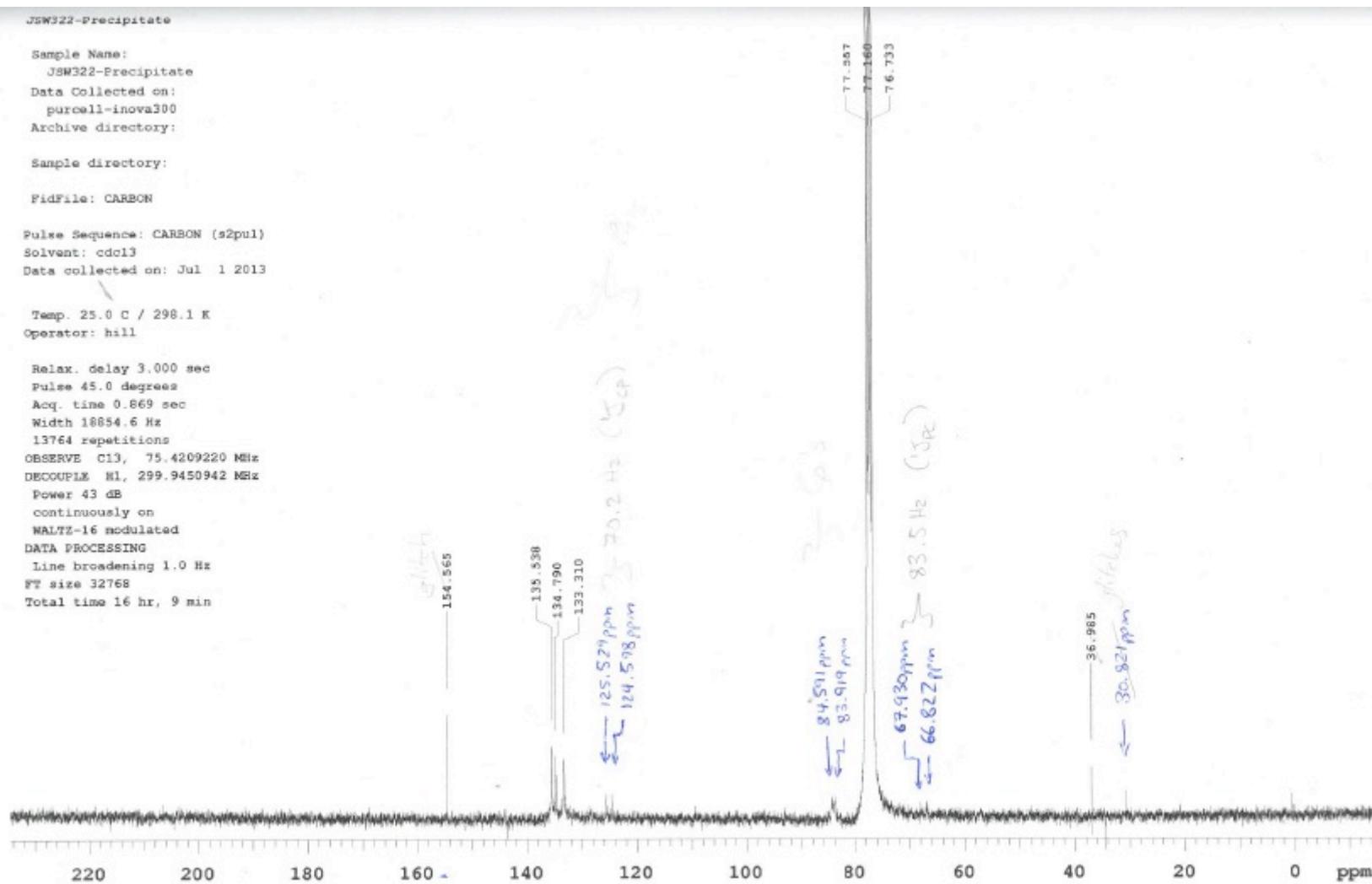
Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2013

Temp. 25.0 C / 298.1 K
Operator: hill

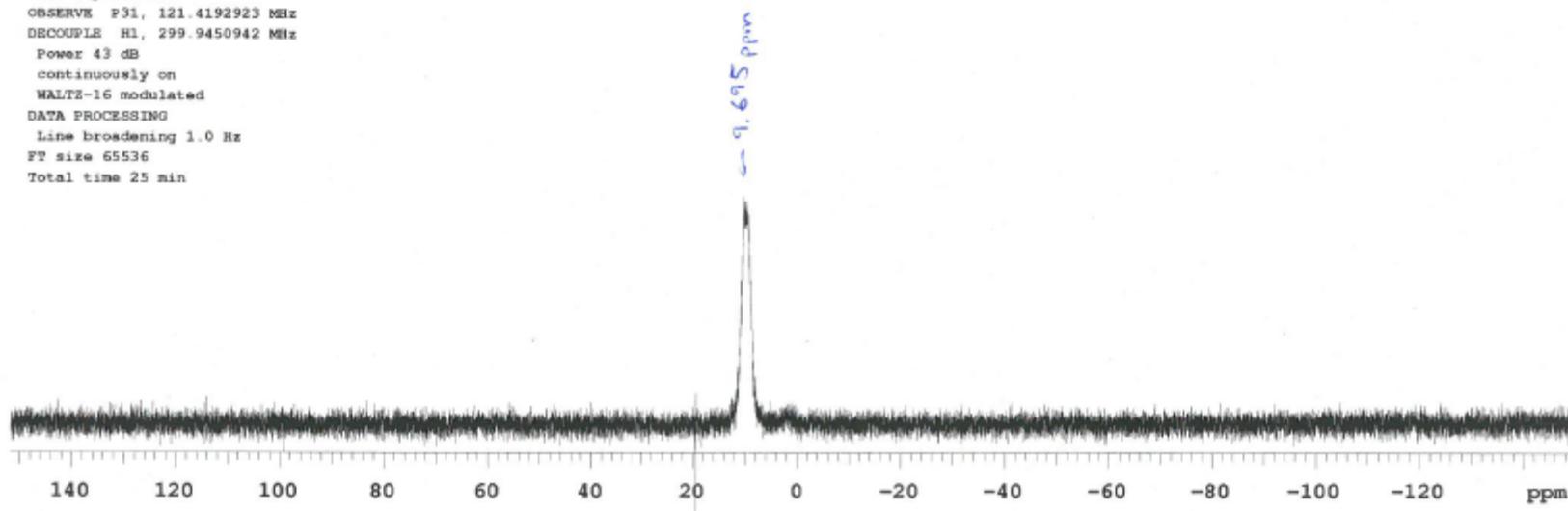
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 0.869 sec
Width 10854.6 Hz
13764 repetitions
OBSERVE C13, 75.4209220 MHz
DECOUPLE M1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 16 hr, 9 min



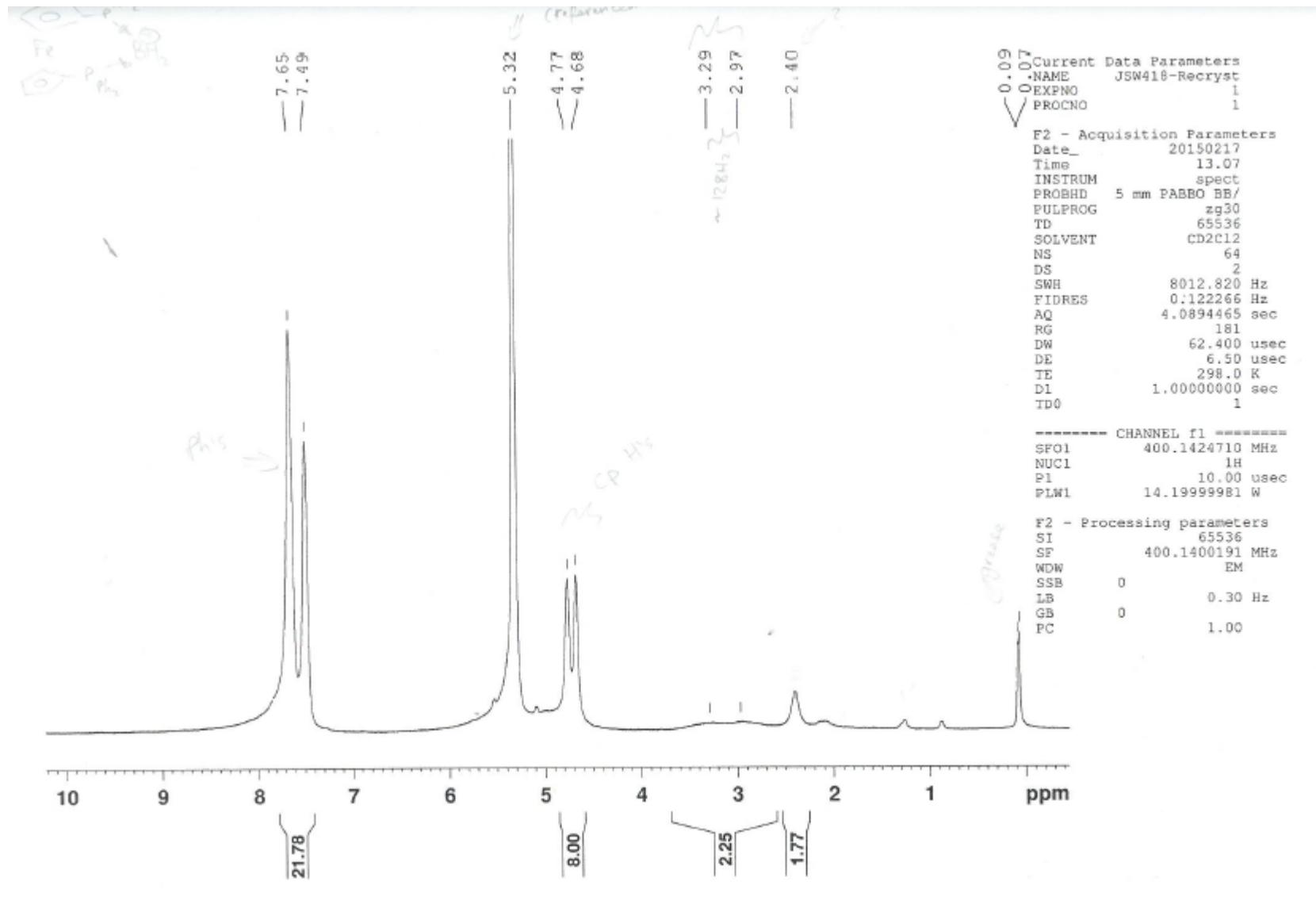
Compound 10 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

JSW322-Precipitate
Sample Name: JSW322-Precipitate
Data Collected on: purcell-inova300
Archive directory:
Sample directory:
FidFile: PHOSPHORUS
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: ccl4
Data collected on: Jul 1 2013
Temp. 25.0 C / 298.1 K
Operator: hill
Relax. delay 1.000 sec
Pulse 60.0 degrees
Aqg. time 0.893 sec
Width 36680.4 Hz
296 repetitions
OBSERVE F31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 25 min

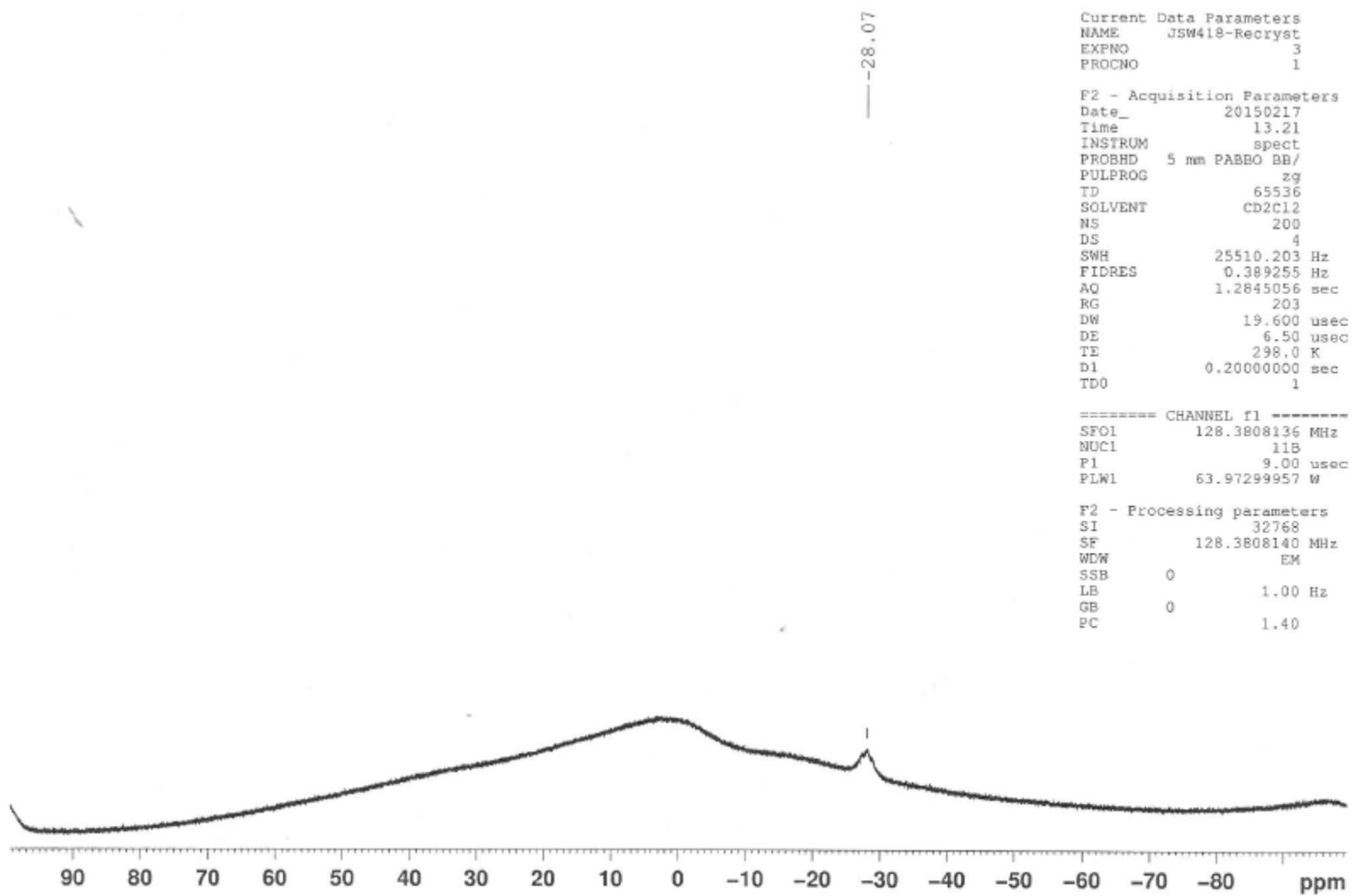
^31P TMS 0 ppm
(mono or bi) 1.9 ppm
[JSW321/JSW322-HKote]
1.9 ppm



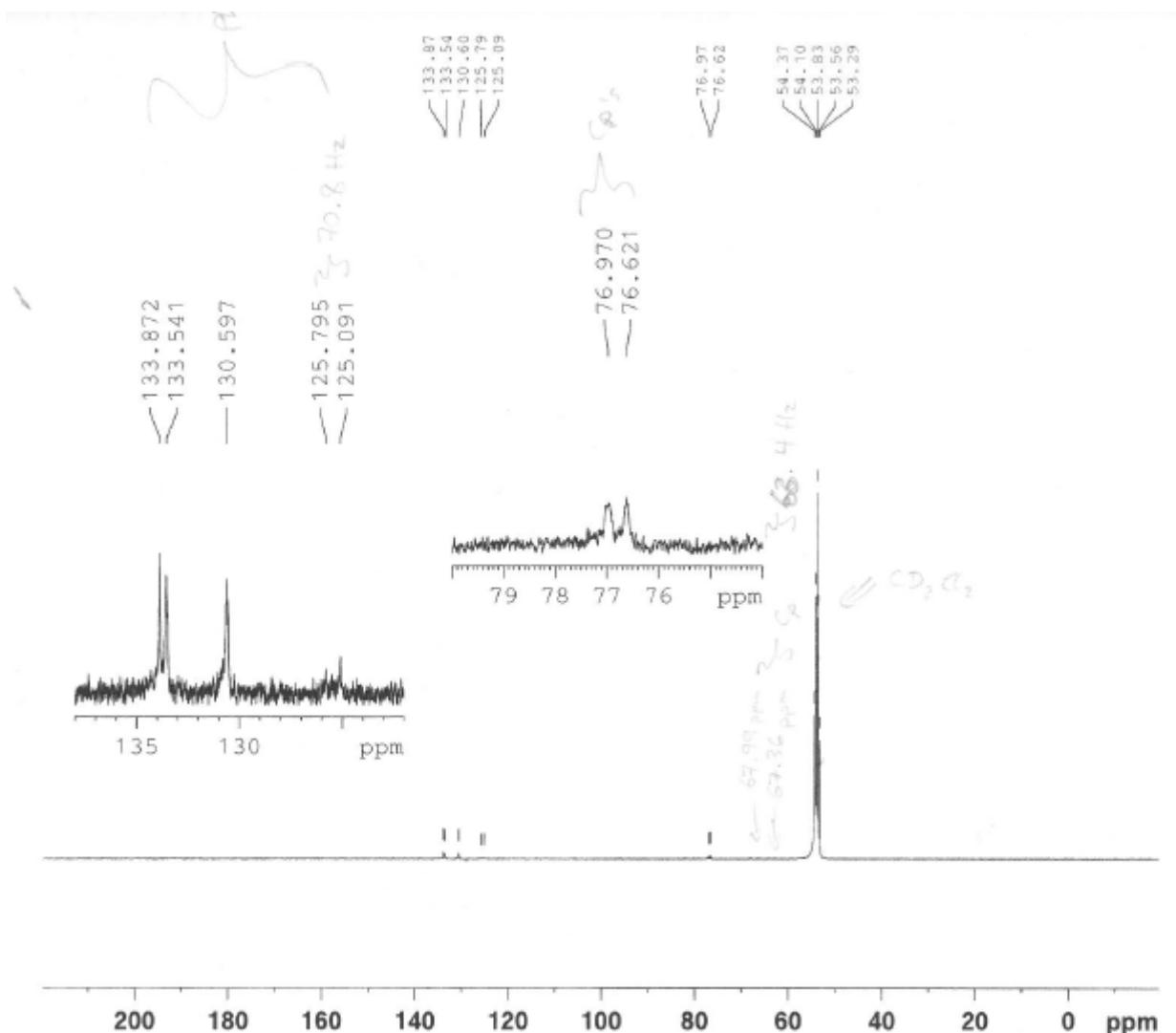
Compound 10 – $^{31}\text{P}\{{}^1\text{H}\}$ NMR spectrum



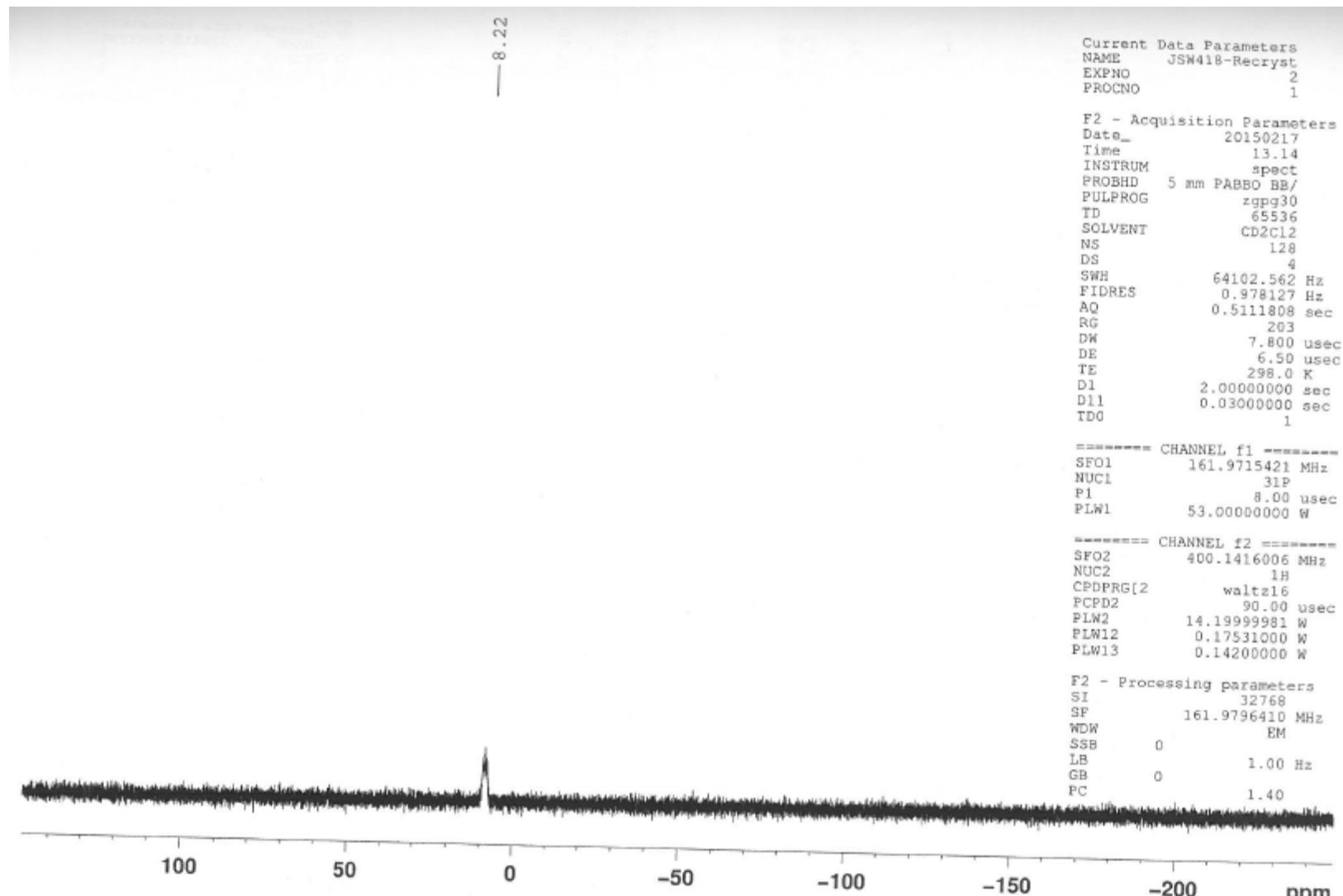
Compound 11 – ^1H NMR spectrum



Compound 11 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 11 – $^{13}\text{C}^{\{1\}\text{H}}$ NMR spectrum



Compound 11 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

JSW319-WashedProduct

Sample Name:

JSW319-WashedProduct

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 26 2013

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.707 sec

Width 4799.0 Hz

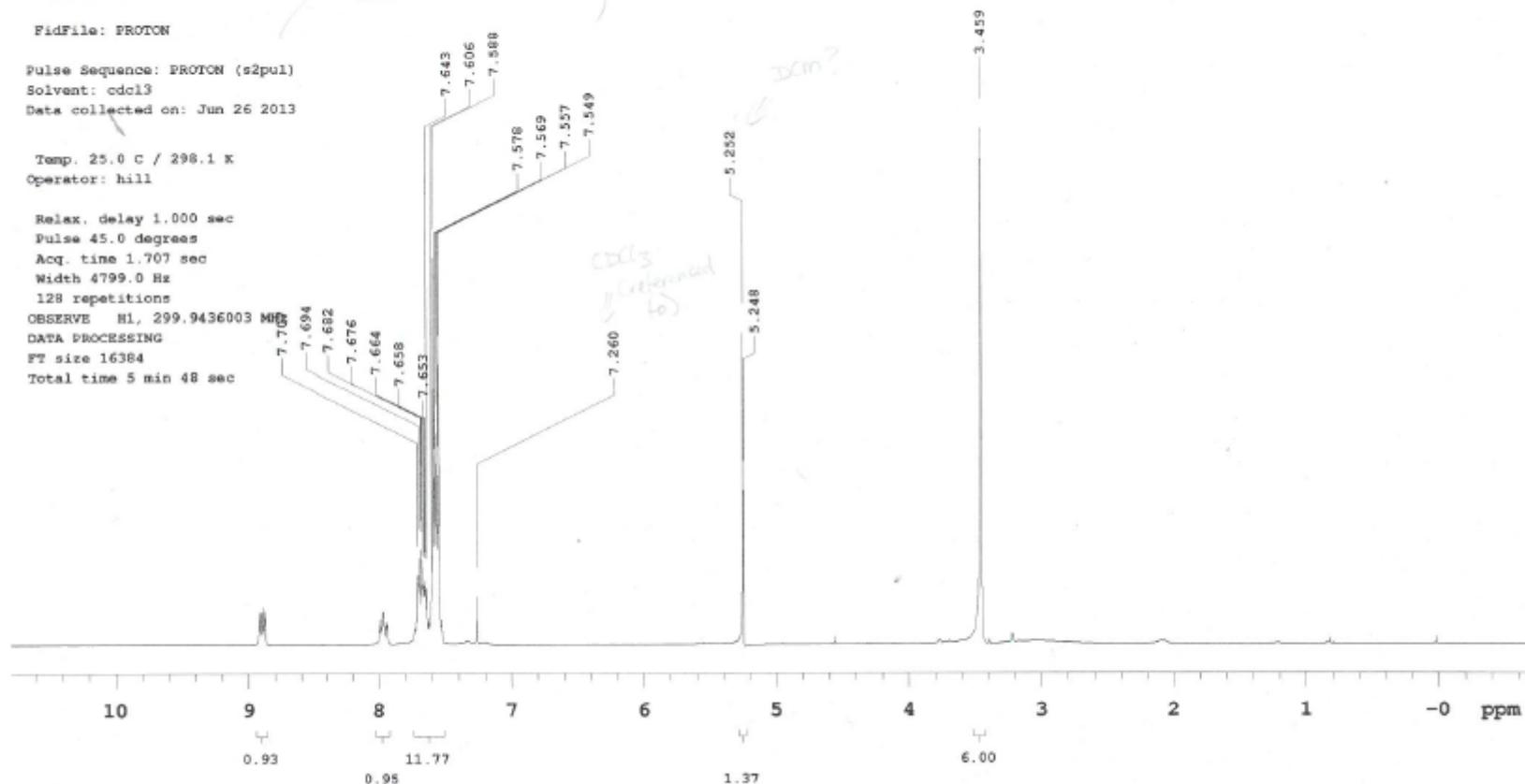
128 repetitions

OBSERVE H1, 299.9436003 MHz

DATA PROCESSING

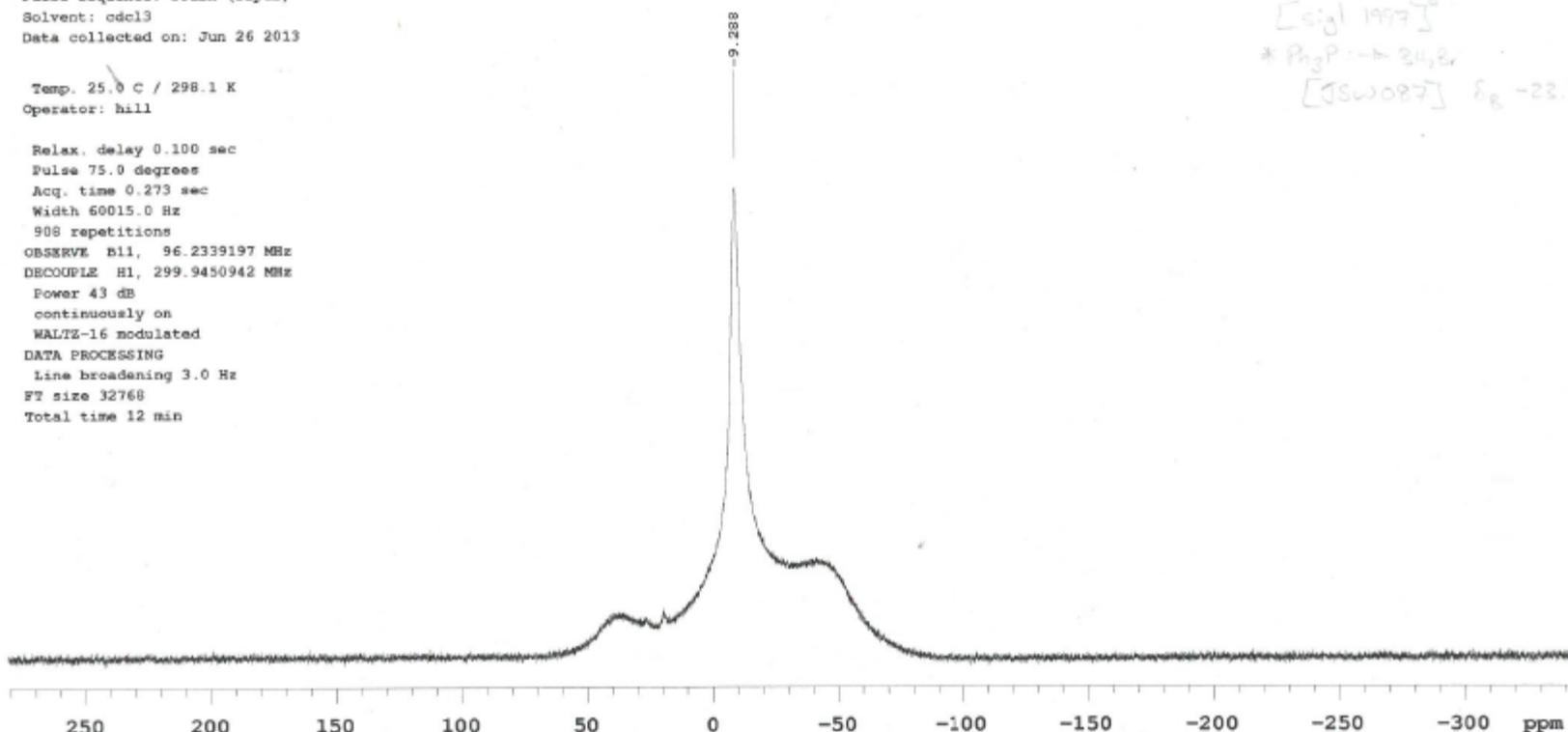
FT size 16384

Total time 5 min 48 sec

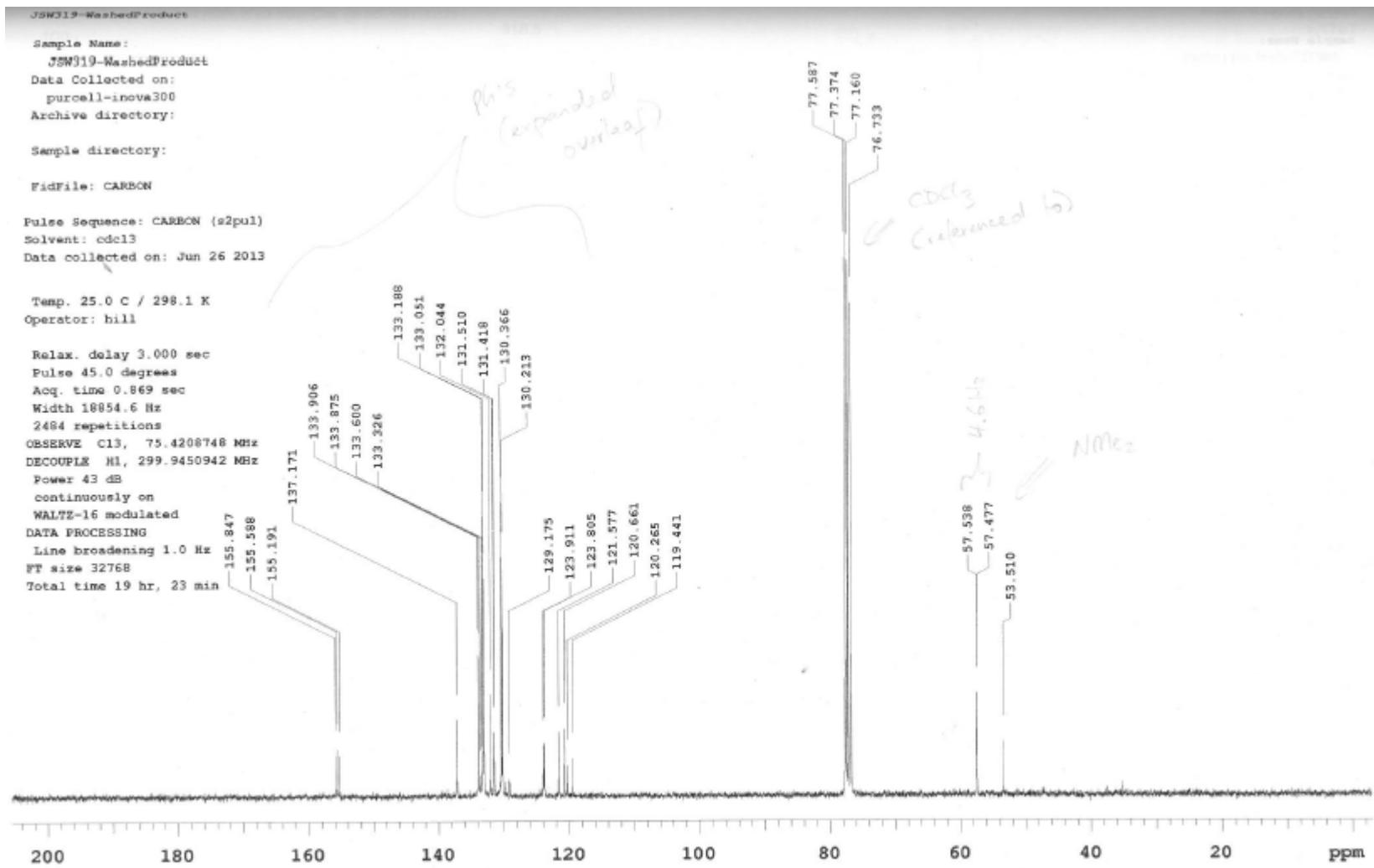


Compound 12 – ^1H NMR spectrum

JSW319-WashedProduct
 Sample Name: JSW319-WashedProduct
 Data Collected on: purcell-inova300
 Archive directory:
 Sample directory:
 FidFile: stdih
 Pulse Sequence: stdih (s2pul)
 Solvent: cdcl3
 Data collected on: Jun 26 2013
 Temp. 25.0 °C / 298.1 K
 operator: hill
 Relax. delay 0.100 sec
 Pulse 75.0 degrees
 Acq. time 0.273 sec
 Width 60015.0 Hz
 908 repetitions
 OBSERVE H1, 96.2339197 MHz
 DECOUPLE H1, 299.9450942 MHz
 Power 43 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 32768
 Total time 12 min



Compound 12 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

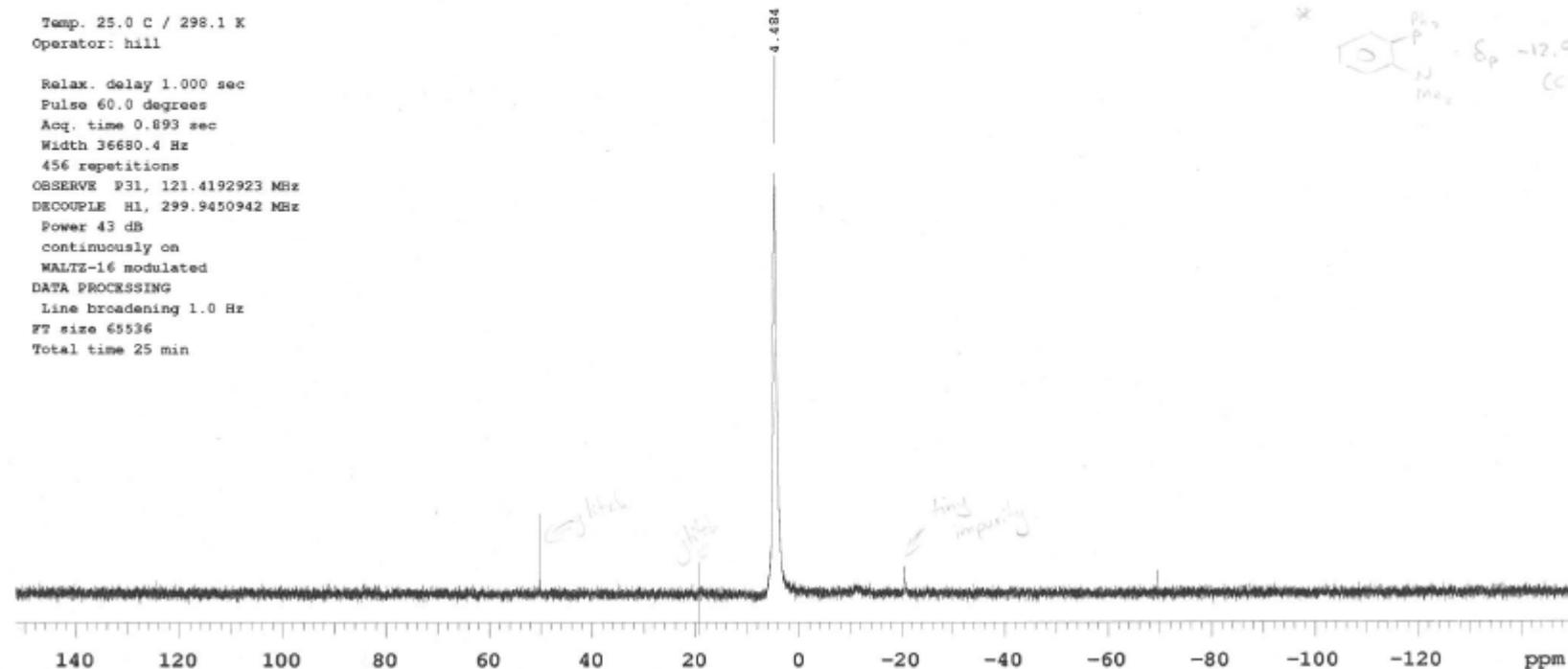


Compound 12 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

```

JSW319-WashedProduct
Sample Name:
JSW319-WashedProduct
Data Collected on:
purcell-inova300
Archive directory:
Sample directory:
FidFile: PHOSPHORUS
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdcl3
Data collected on: Jun 26 2013
Temp. 25.0 C / 298.1 K
Operator: hilf
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
456 repetitions
OBSERVE P31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 25 min

```



Compound 12 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

JSW257-CrudeSolid

Sample Name: JSW257-CrudeSolid

Data Collected on: purcell-inova300

Archive directory:

Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)

Solvent: cd2cl2

Data collected on: Jul 26 2012

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 0.100 sec

Pulse 75.0 degrees

Acq. time 0.273 sec

Width 60015.0 Hz

608 repetitions

OBSERVE B11, 96.2341081 MHz

DECOUPLE H1, 299.9456701 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

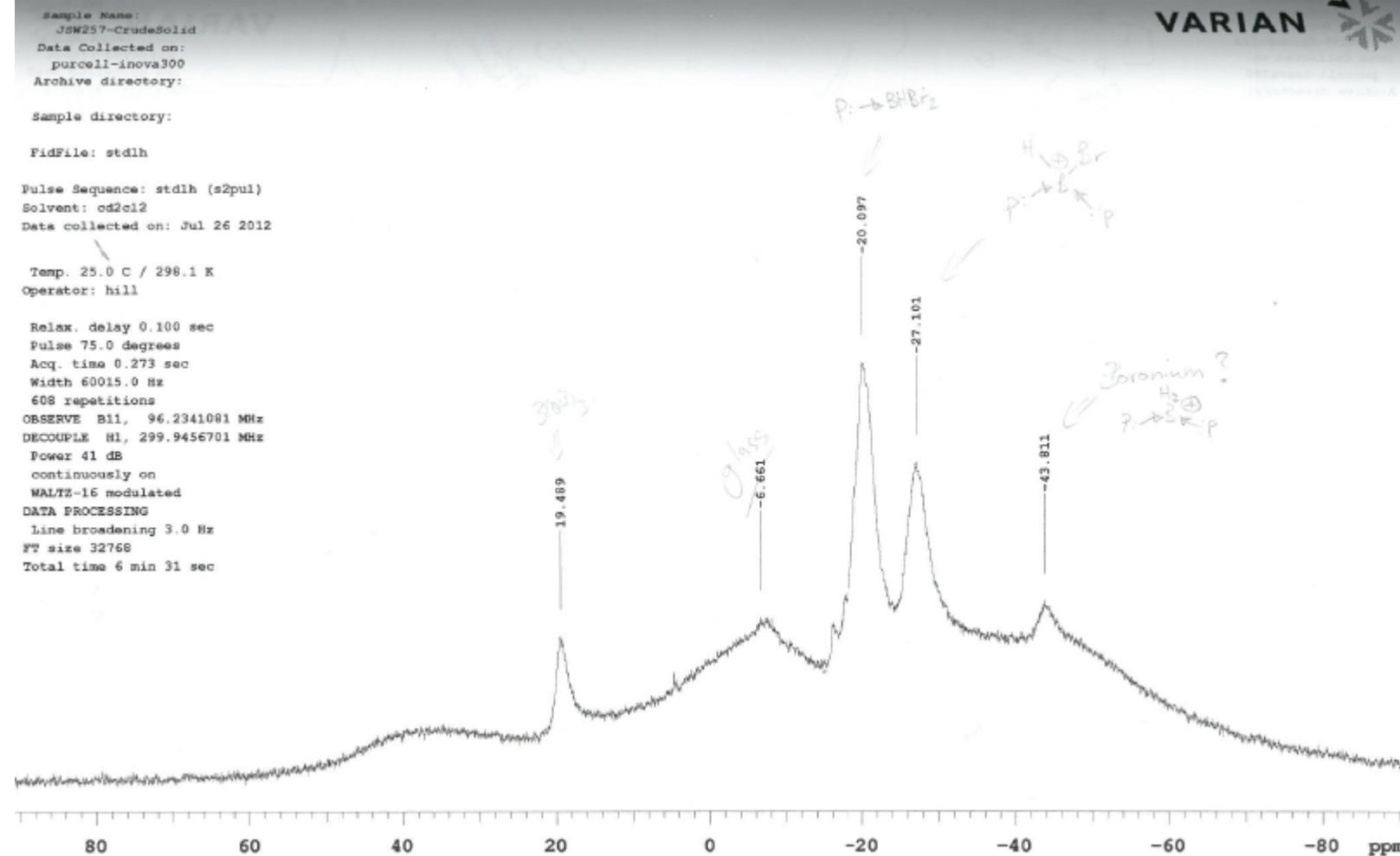
DATA PROCESSING

Line broadening 3.0 Hz

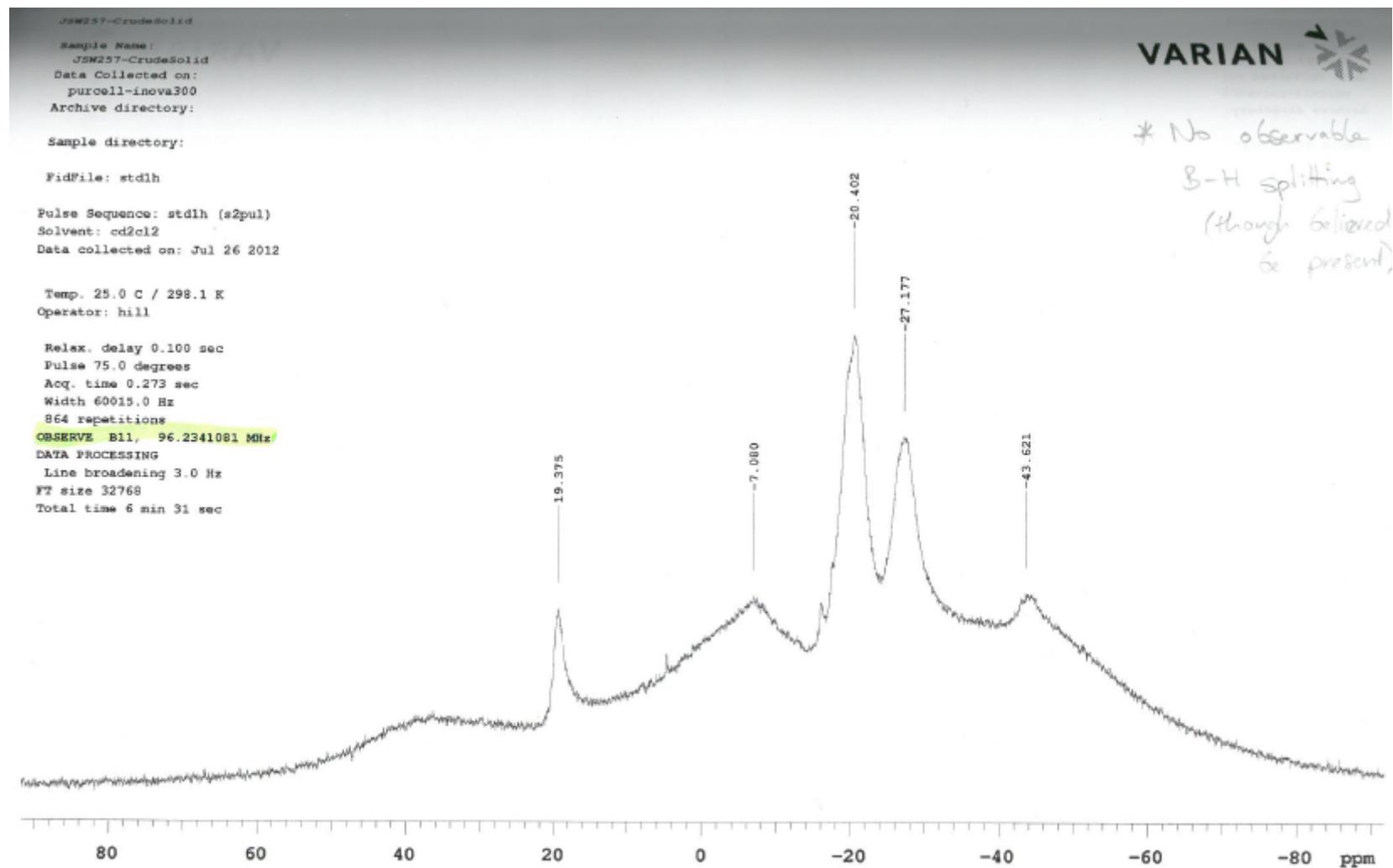
FT size 32768

Total time 6 min 31 sec

VARIAN



Compound 13 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 13 – ^{11}B NMR spectrum

JSW257-CrudeSolid

Sample Name:

JSW257-CrudeSolid

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: PHOSPHORUS

Pulse Sequence: PHOSPHORUS (s2pul)

Solvent: cd2cl2

Data collected on: Jul 26 2012

Temp. 25.0 C / 298.1 K

Operator: hill

Relax. delay 1.000 sec

Pulse 60.0 degrees

Acq. time 2.150 sec

Width 60975.6 Hz

200 repetitions

OBSERVE P31, 121.4195536 MHz

DECOUPLE H1, 299.9456701 MHz

Power 41 dB

continuously on

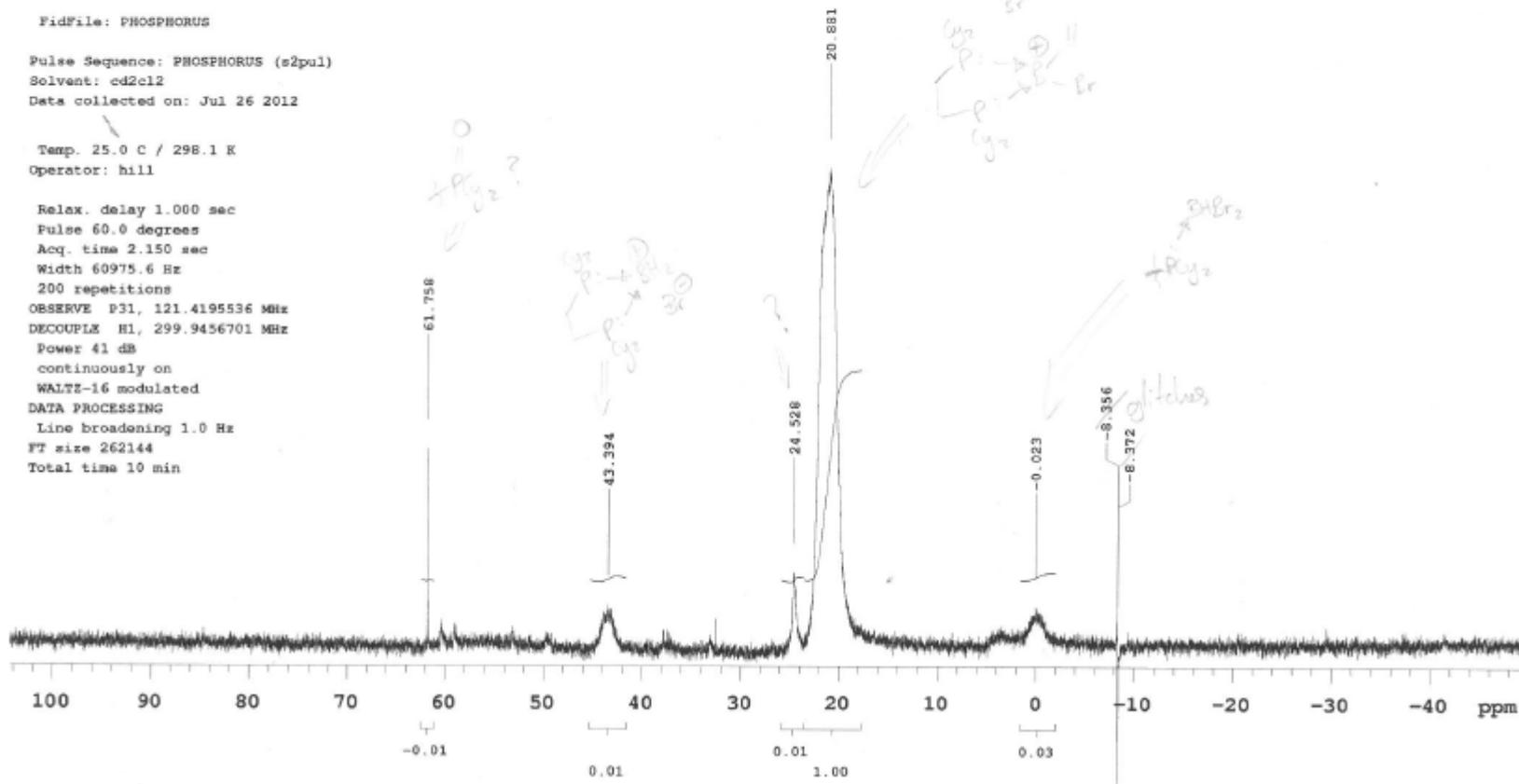
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 262144

Total time 10 min



Compound 13 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

JSW329-Product

Sample Name:
JSW329-Product
Data Collected on:
purcell-inova300
Archive directory:

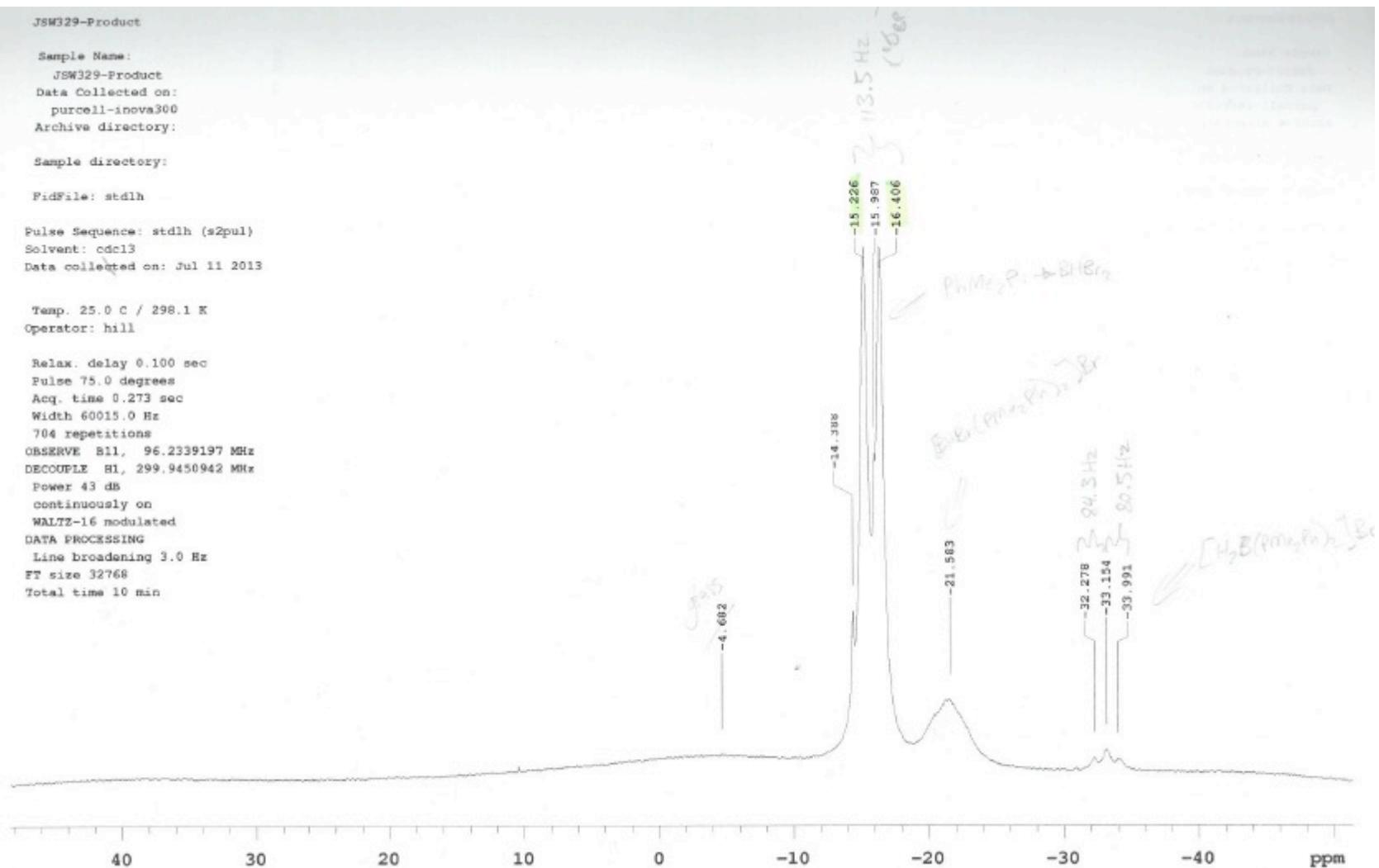
Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Jul 11 2013

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
704 repetitions
OBSERVE H1, 96.2339197 MHz
DECOUPLE B1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 10 min



Compound 14 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

JSN329-Product

Sample Name:
JSM329-Product
Data Collected on:
purcell-inova300
Archive directory:

Sample directory:

FidFile: PHOSPHORUS

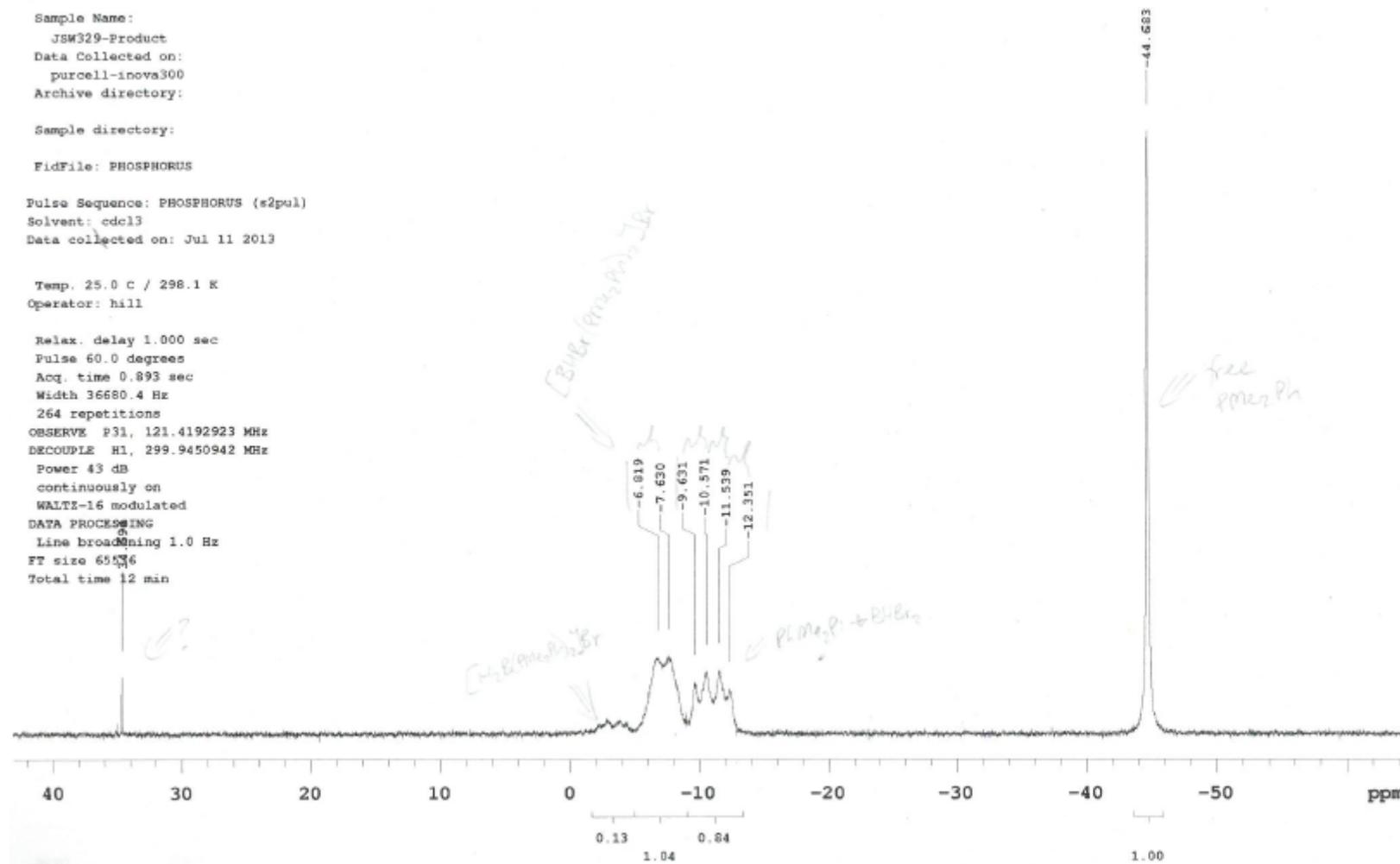
Pulse Sequence: PHOSPHORUS (π 2pul)
Solvent: cdcl3
Data collected on: Jul 11 2013

Temp. 25.0 °C / 298.1 K
Operator: hill

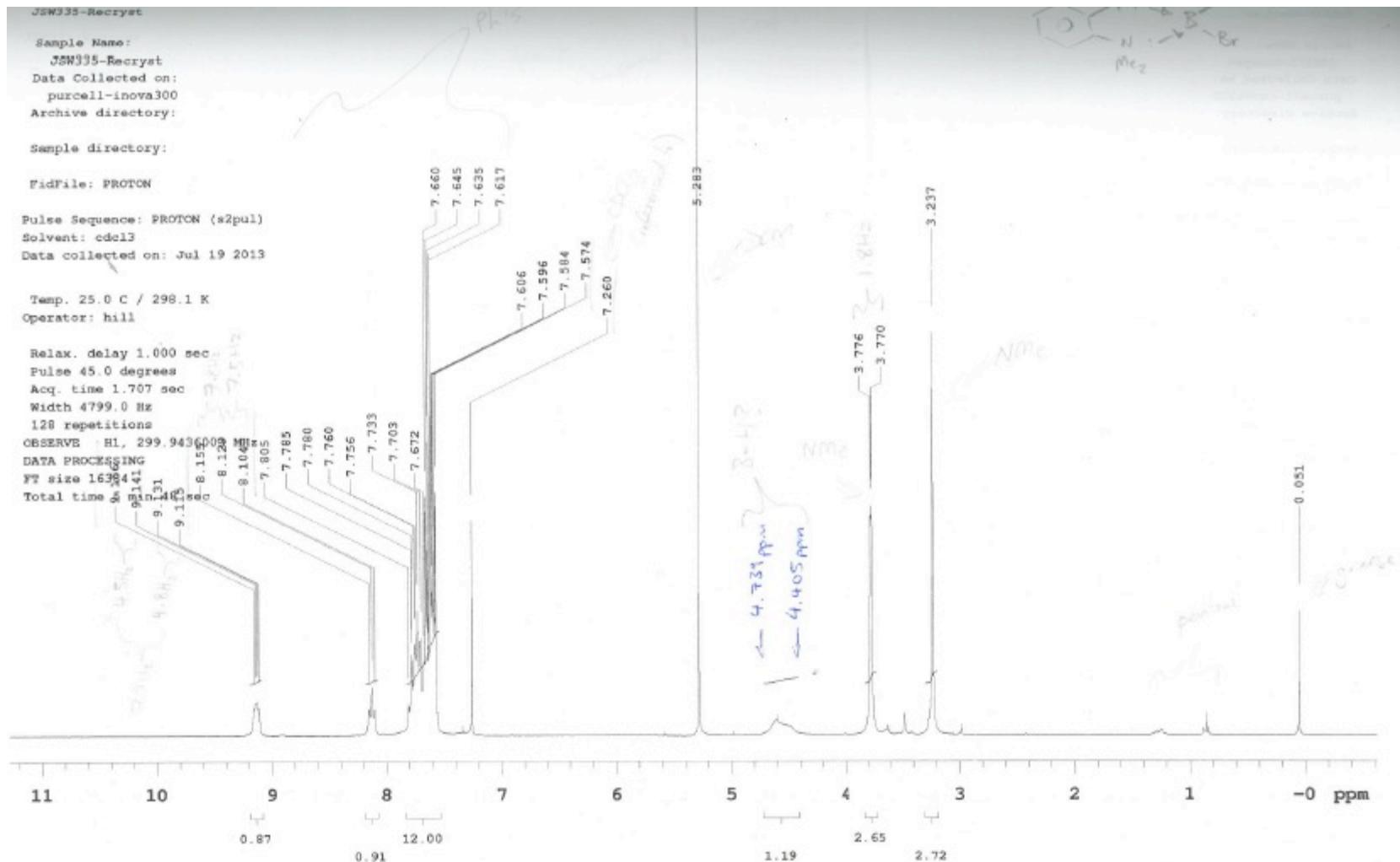
```

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
264 repetitions
OBSERVE F31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 12 min

```



Compound 14 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum



Compound 15 – ^1H NMR spectrum

JSW335-AfterDrying

Sample Name:

JSW335-AfterDrying

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)

Solvent: cdcl3

Data collected on: Aug 29 2013

Temp. 25.0 °C / 298.1 K

Operator: hill

Relax. delay 0.100 sec

Pulse 75.0 degrees

Acq. time 0.273 sec

Width 60015.0 Hz

960 repetitions

OBSERVE H1, 96.2339197 MHz

DECOUPLE H1, 299.9450942 MHz

Power 43 dB

continuously on

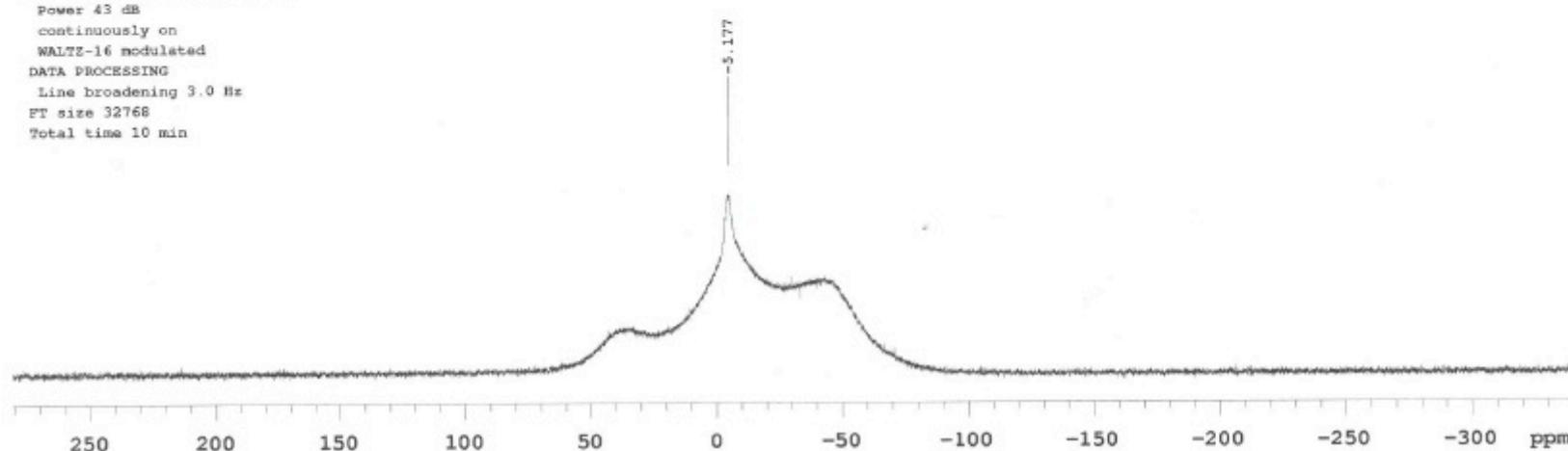
WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FT size 32768

Total time 10 min



Compound 15 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

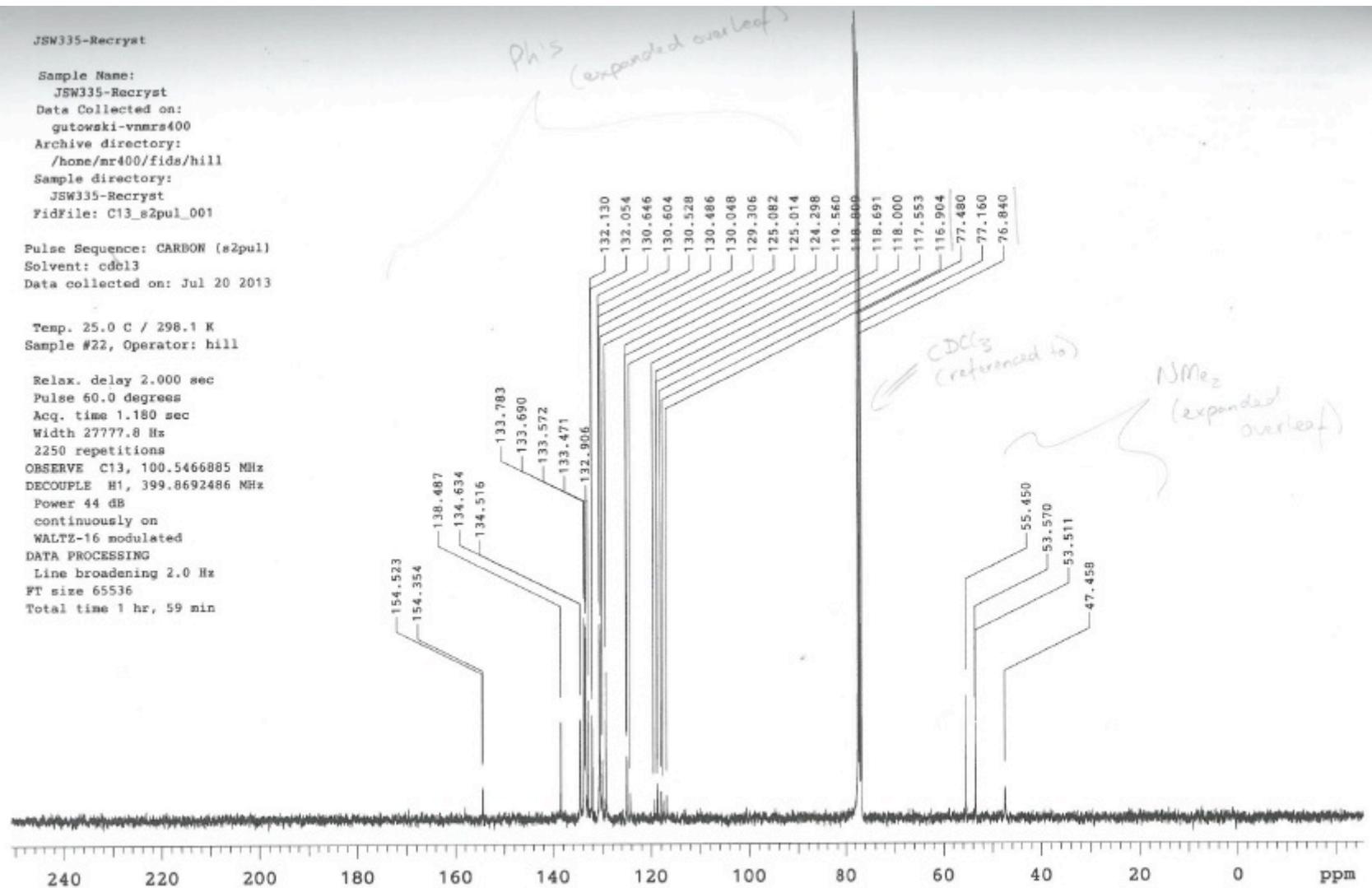
JSW335-Recryst

Sample Name:
JSW335-Recryst
Data Collected on:
gutowski-vnmrs400
Archive directory:
/home/mr400/fids/hill
Sample directory:
JSW335-Recryst
Piffile: C13_s2pul_001

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 20 2013

Temp. 25.0 C / 298.1 K
Sample #22, Operator: hill

Relax. delay 2.000 sec
Pulse 60.0 degrees
Acq. time 1.180 sec
Width 27777.8 Hz
2250 repetitions
OBSERVE C13, 100.5466885 MHz
DECOUPLE H1, 399.8692486 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min



Compound 15 – ¹³C{¹H} NMR spectrum

JSW335-Recryst
Sample Name:
JSW335-Recryst
Data Collected on:
purcell-inova300
Archive directory:

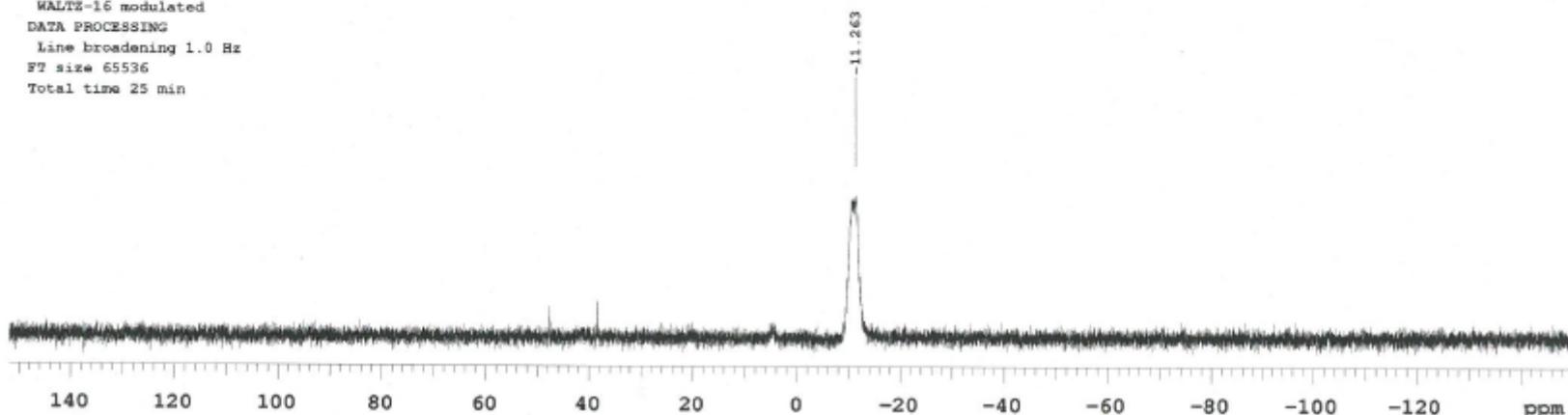
Sample directory:

FidFile: PHOSPHORUS

Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdcl3
Data collected on: Jul 19 2013

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
524 repetitions
OBSERVE P31, 121.4192923 MHz
DECOPPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 25 min



Compound 15 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum

J5W323-CrudeProduct

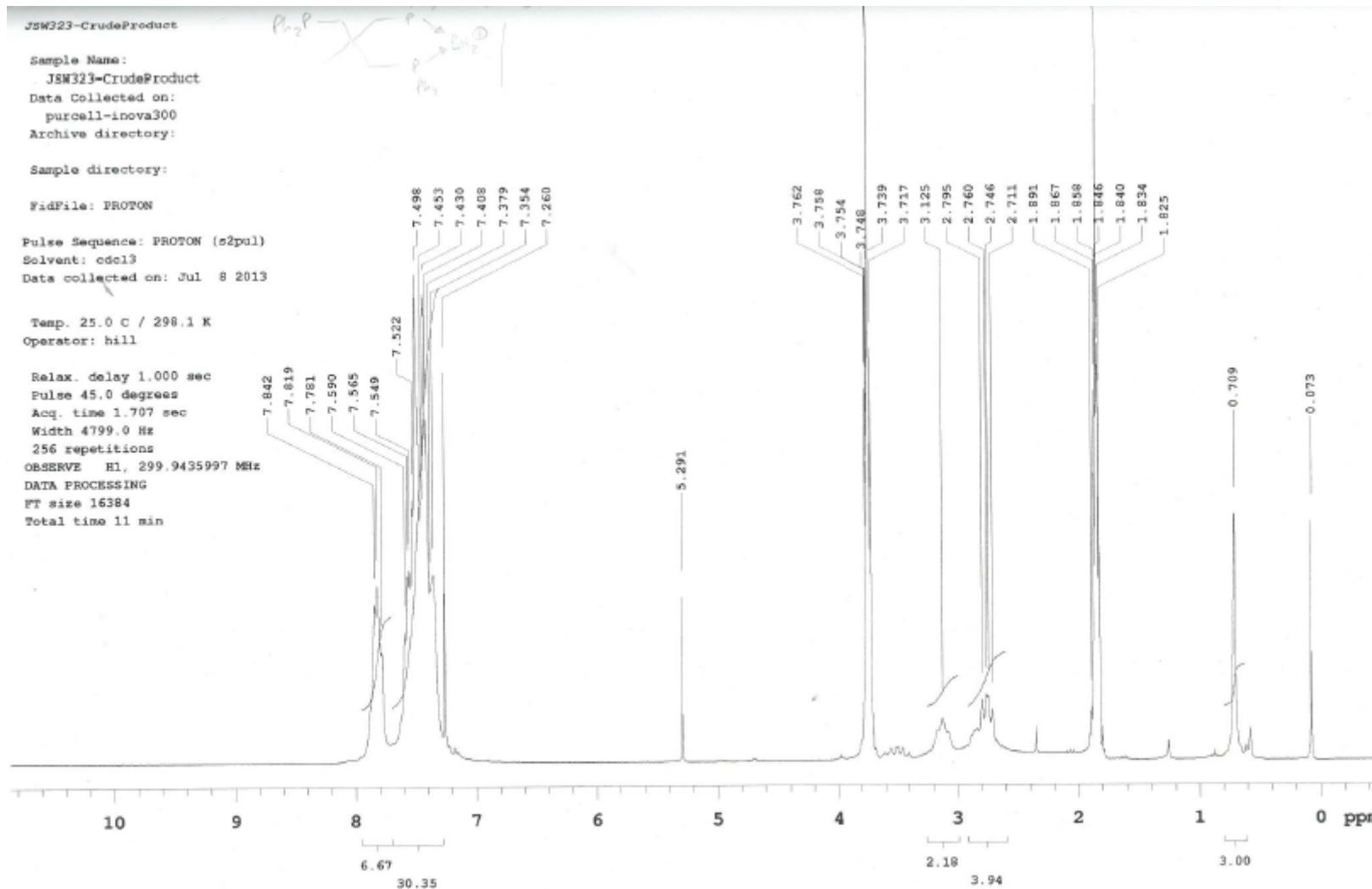
Sample Name:
J5W323-CrudeProduct
Data Collected on:
purcell-inova300
Archive directory:

Sample directory:
fidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 8 2013

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.707 sec
Width 4799.0 Hz
256 repetitions
OBSERVE H1, 299.9435997 MHz
DATA PROCESSING
FT size 16384
Total time 11 min



Compound 17 – ^1H NMR spectrum

JSW323-CrudeProduct

Sample Name:
JSW323-CrudeProduct

Data Collected on:
purcell-inova300
Archive directory:

Sample directory:

FidFile: stdlh

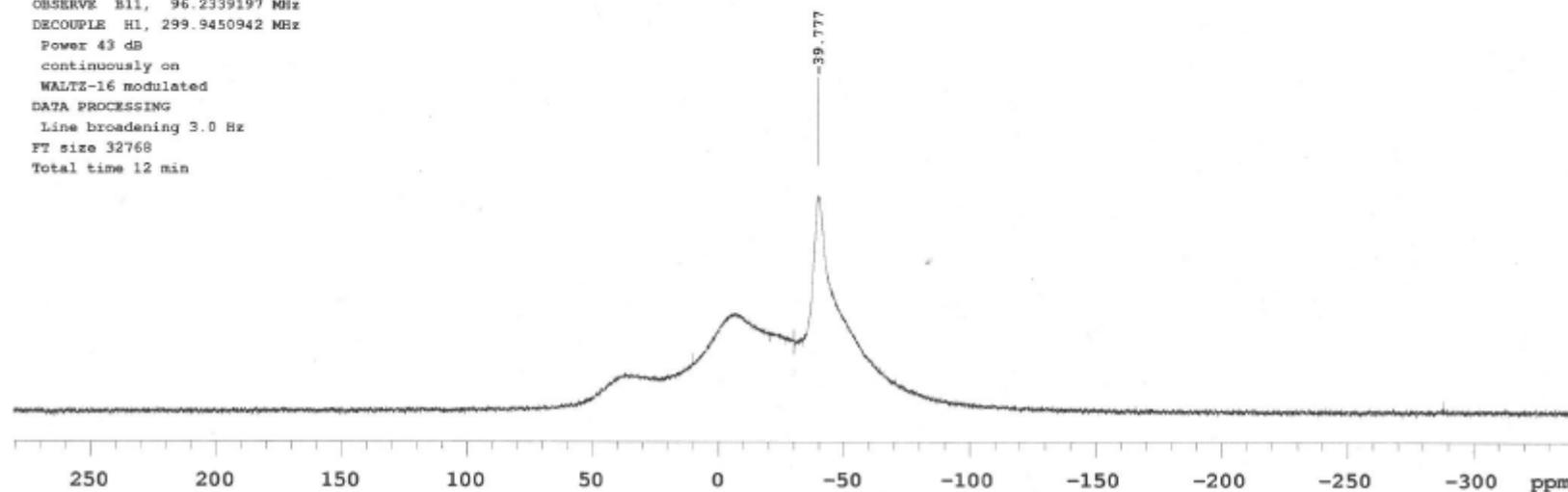
Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Jul 8 2013

Temp. 25.0 C / 298.1 K
operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1120 repetitions
OBSERVE B11, 96.2339197 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 12 min



[JSW15] $\delta_B = -39.1$ (2cm)



Compound 17 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

JSM323-CrudeProduct

Sample Name:
JSM323-CrudeProduct

Data Collected on:
purcell-inova300
Archive directory:

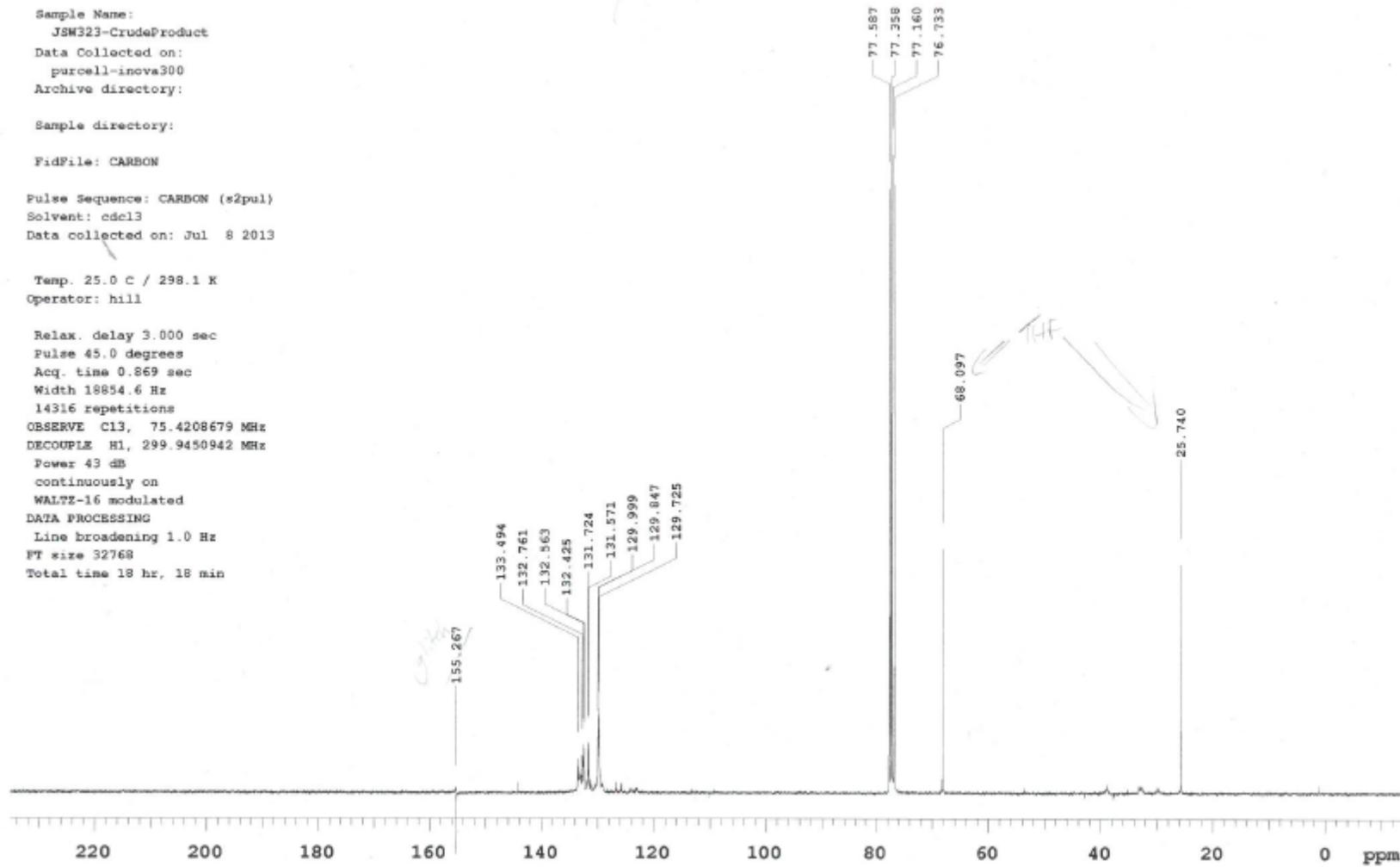
Sample directory:

FidFile: CARBON

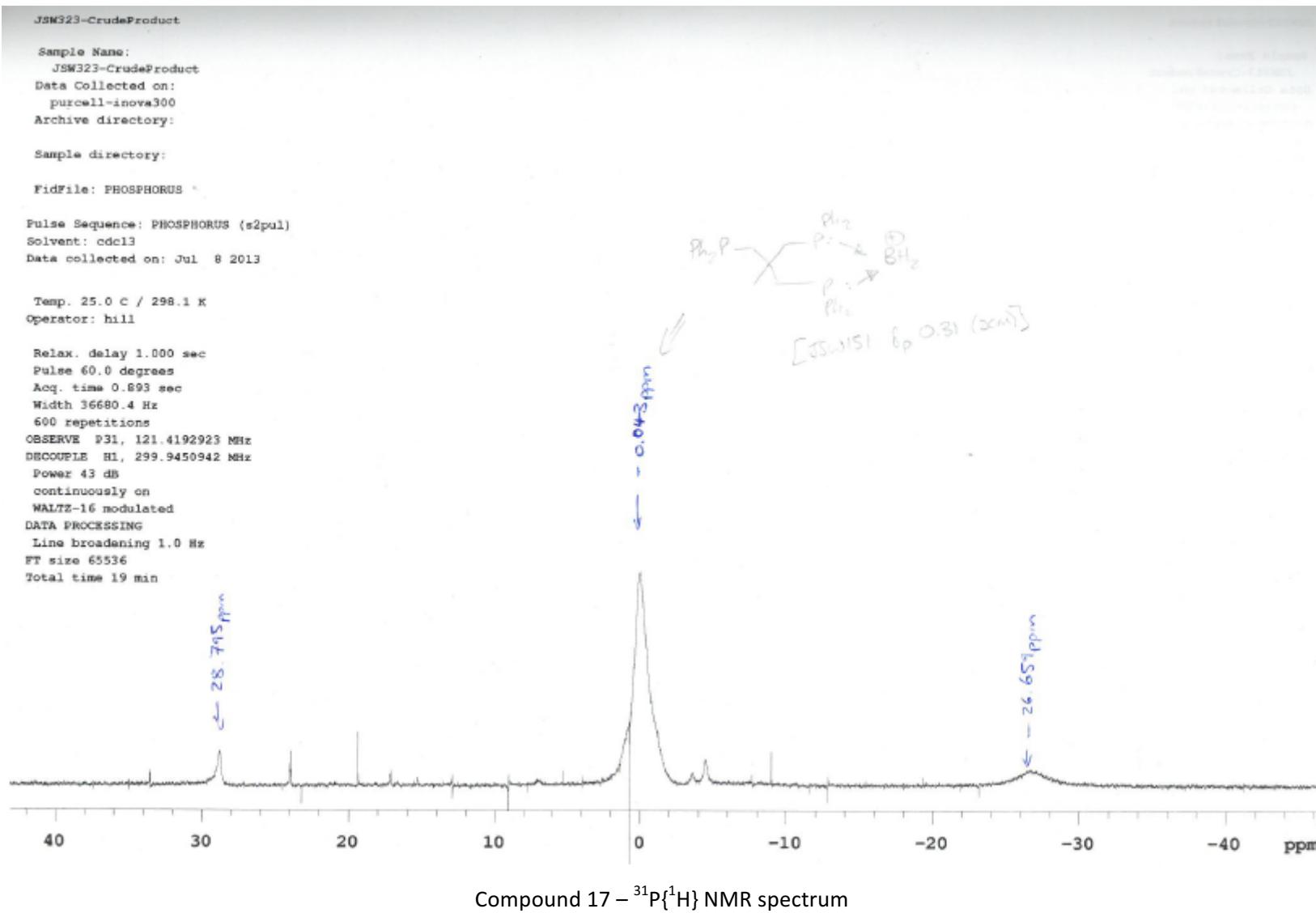
Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Jul 8 2013

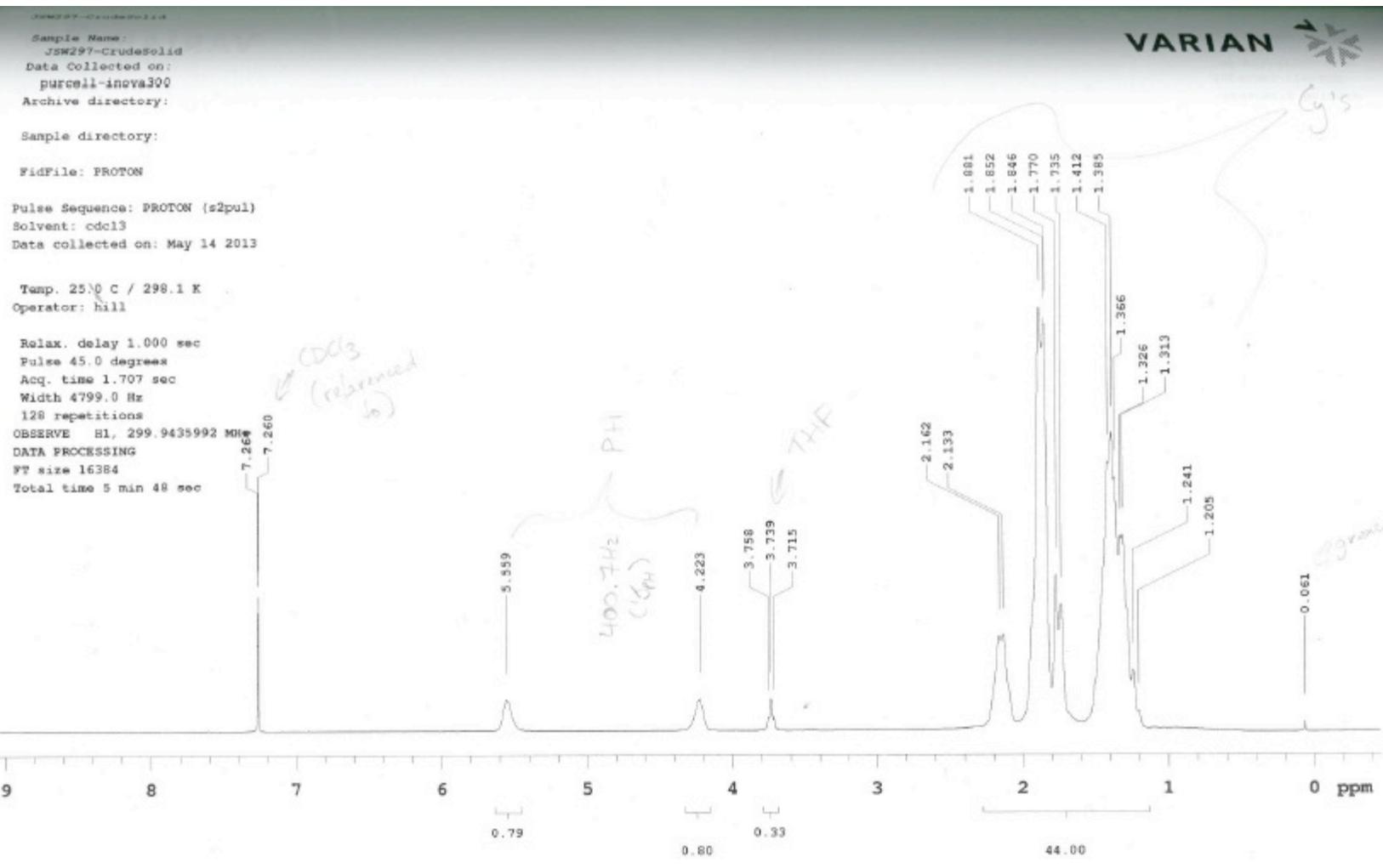
Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 0.869 sec
Width 18854.6 Hz
14316 repetitions
OBSERVE C13, 75.4208679 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 18 hr, 18 min



Compound 17 – $^{13}\text{C}\{\text{H}\}$ NMR spectrum





Compound 18 – ^1H NMR spectrum

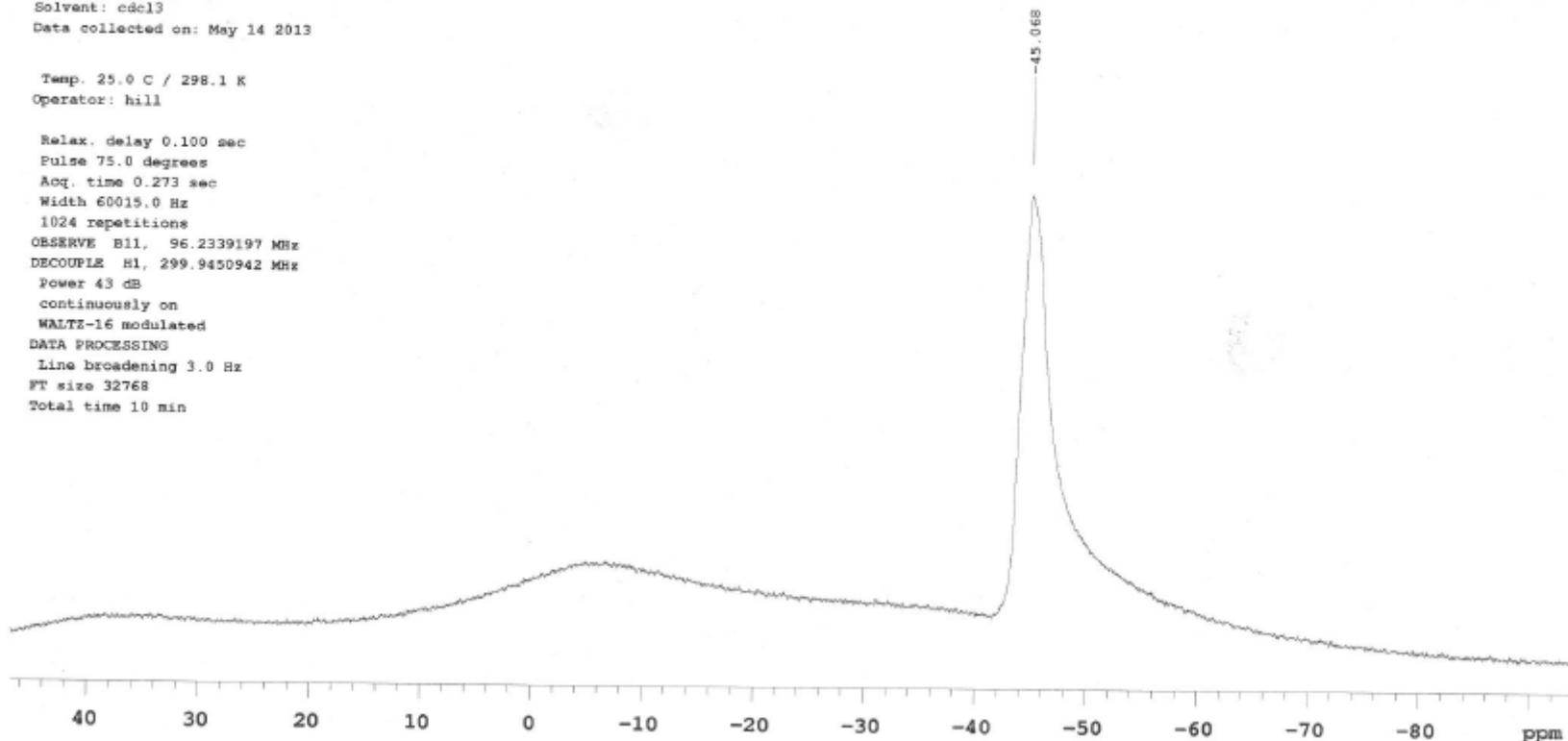
JSW297-CrudeSolid
Sample Name: JSW297-CrudeSolid
Data Collected on: purcell-inova300
Archive directory:
Sample directory:
FidFile: stdlh
Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: May 14 2013

Temp. 25.0 C / 298.1 K
Operator: hill

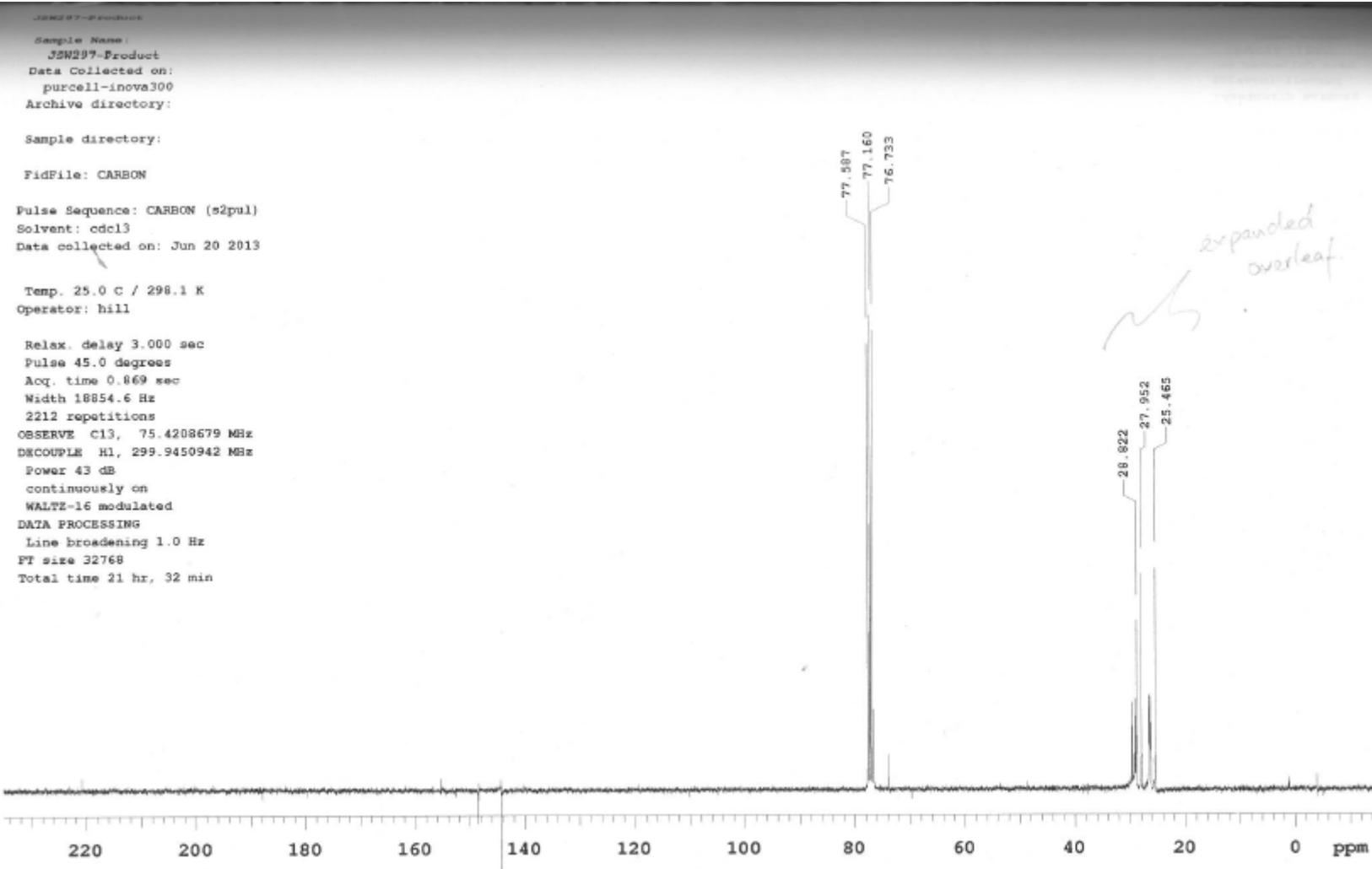
Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
1024 repetitions
OBSERVE H1, 96.2339197 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 10 min

VARIAN

* [H₂B(PH₃)₂]Br (s)
 δ_c -45.2 (CD)



Compound 18 – ¹¹B{¹H} NMR spectrum



Compound 18 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

Sample Name:
JSW297=Product
Data Collected on:
purcell-inova300
Archive directory:

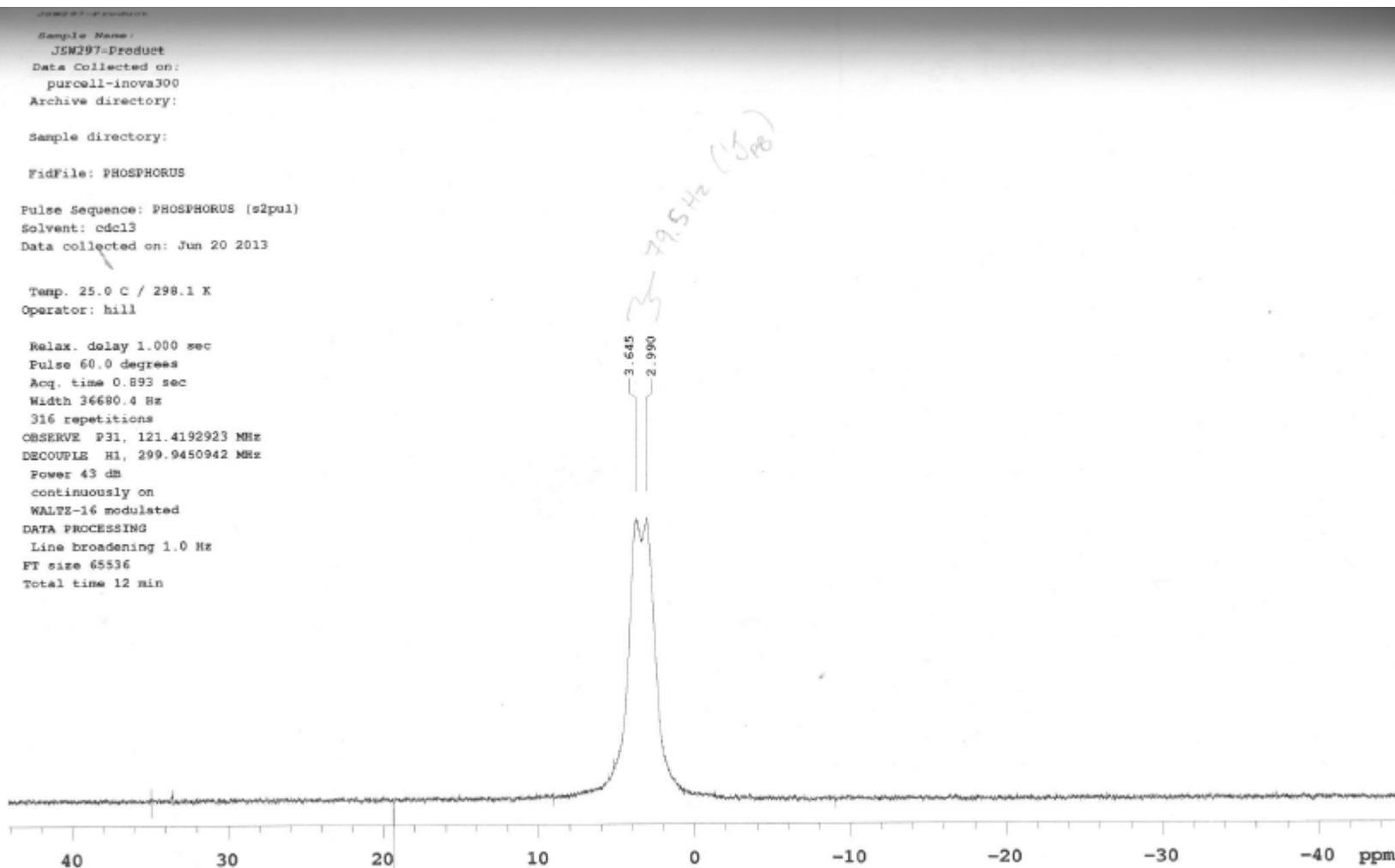
Sample directory:

FidFile: PHOSPHORUS

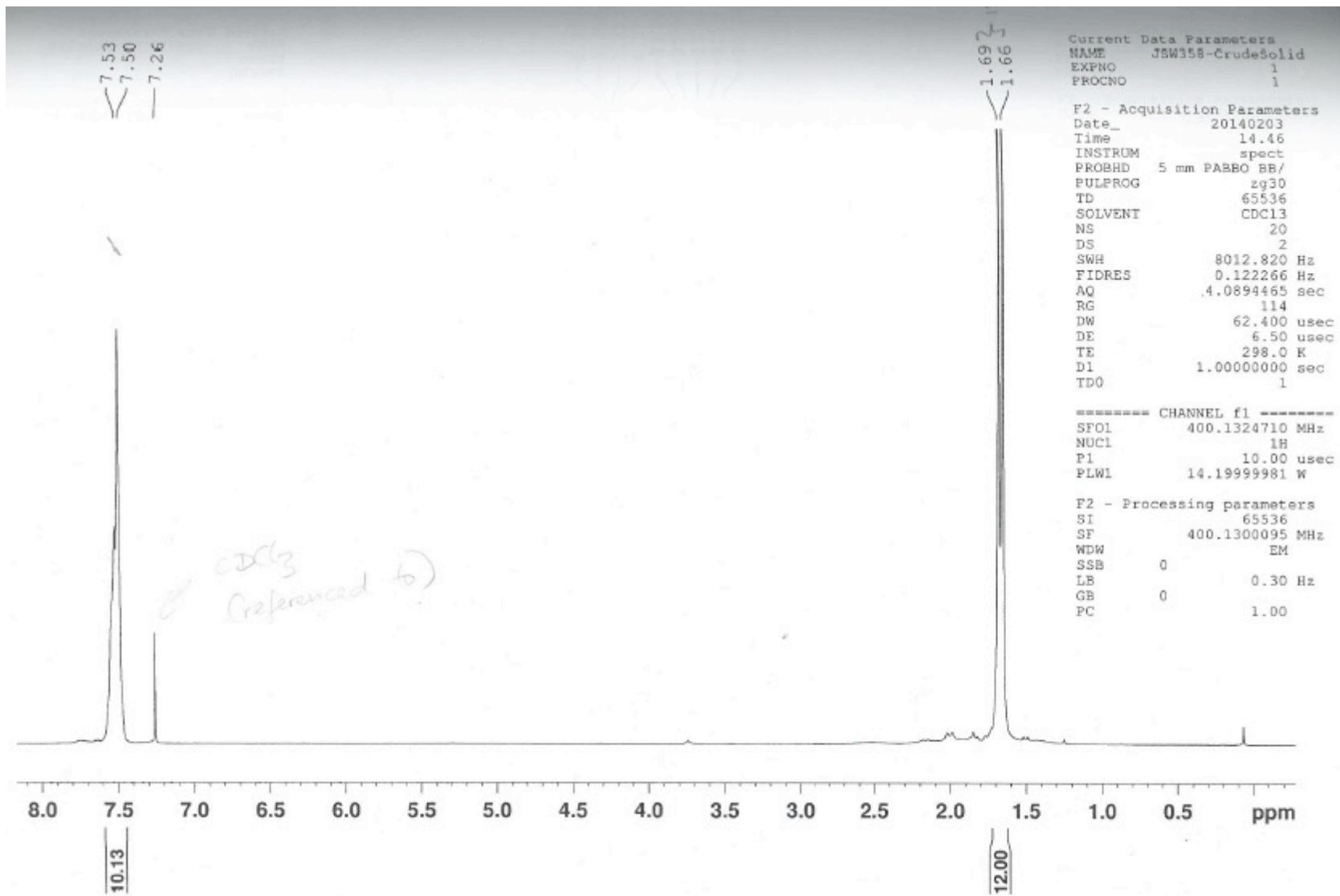
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdc13
Data collected on: Jun 20 2013

Temp. 25.0 C / 298.1 K
Operator: hill

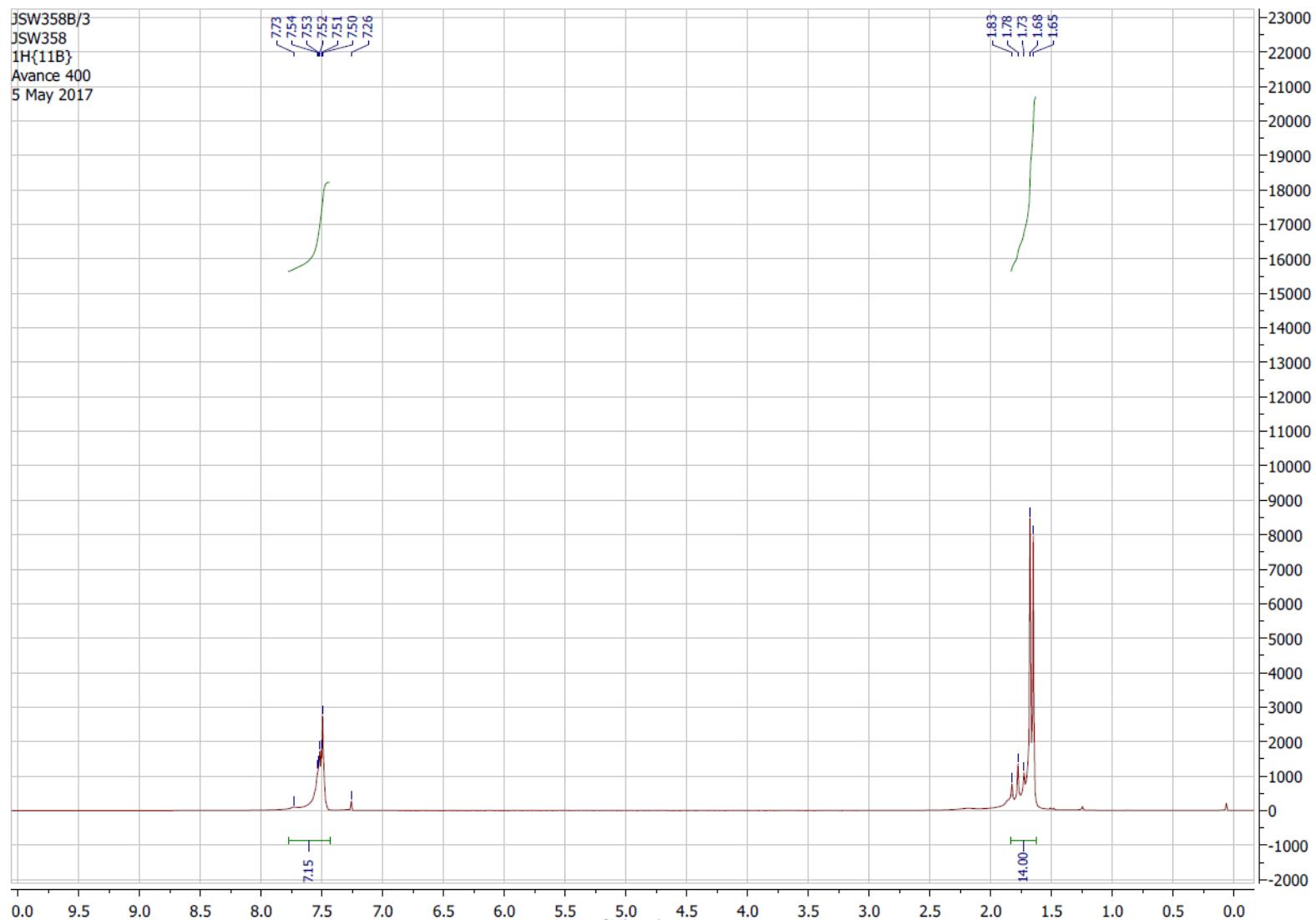
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
316 repetitions
OBSERVE P31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 12 min



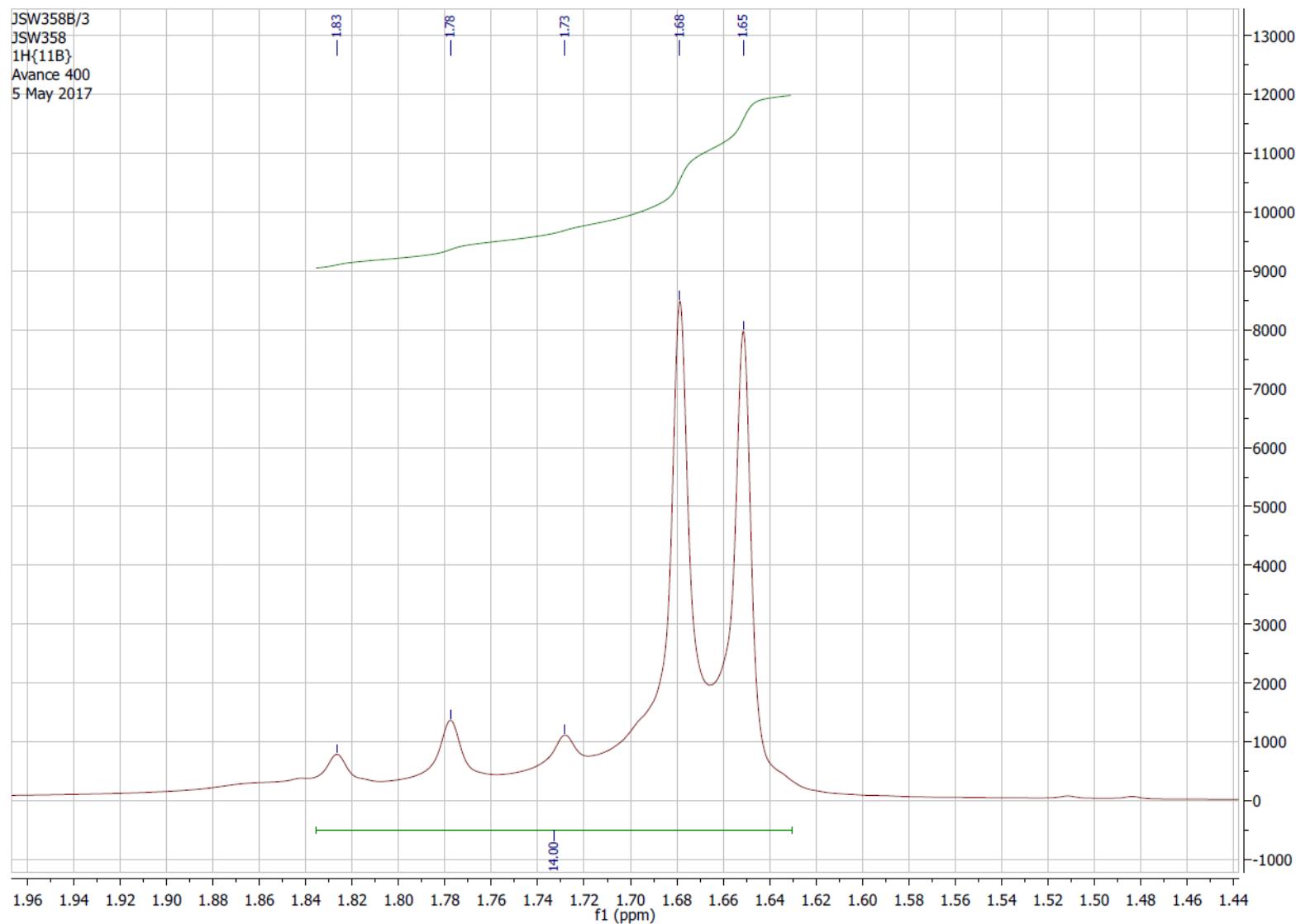
Compound 18 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



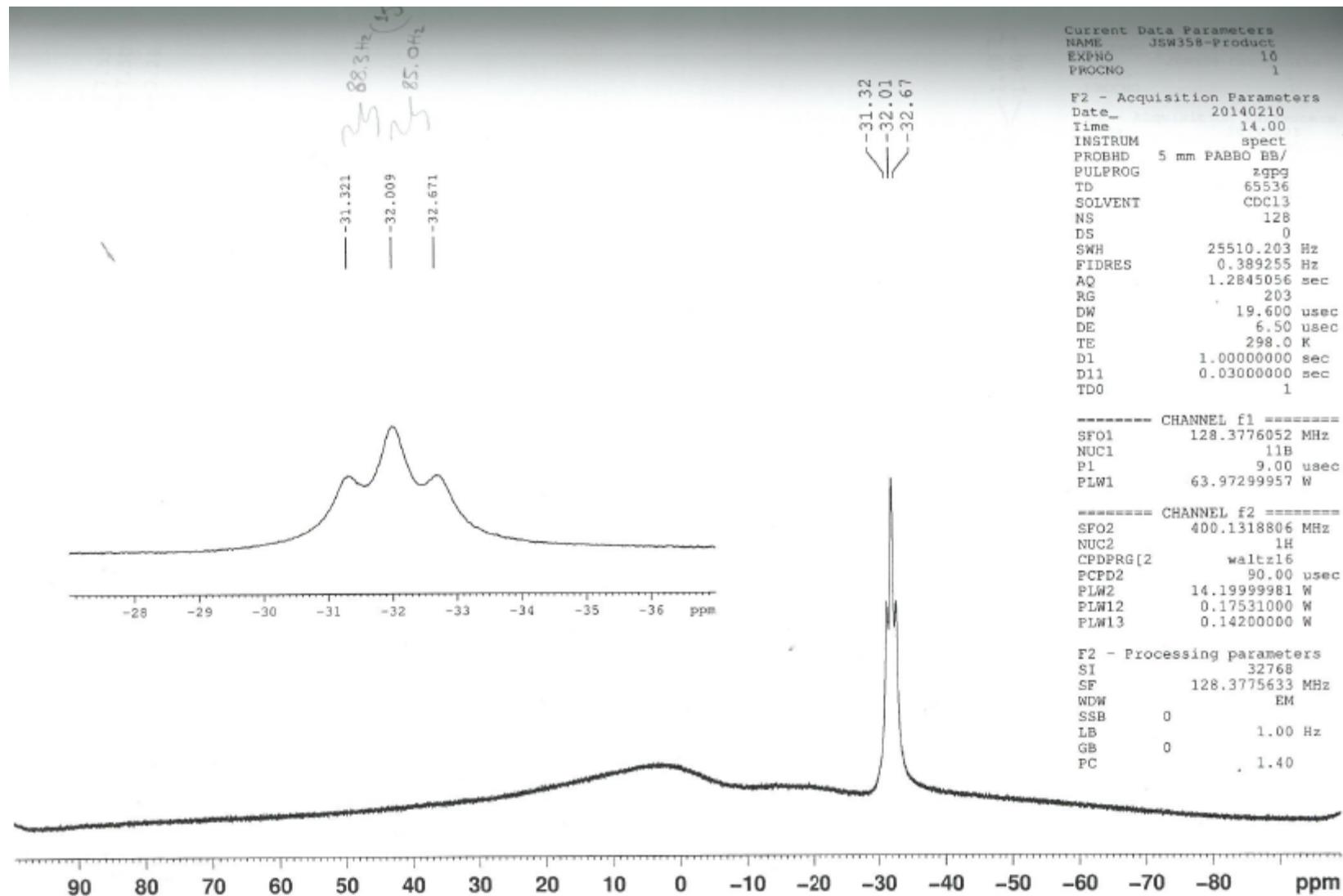
Compound 19 – ¹H NMR spectrum



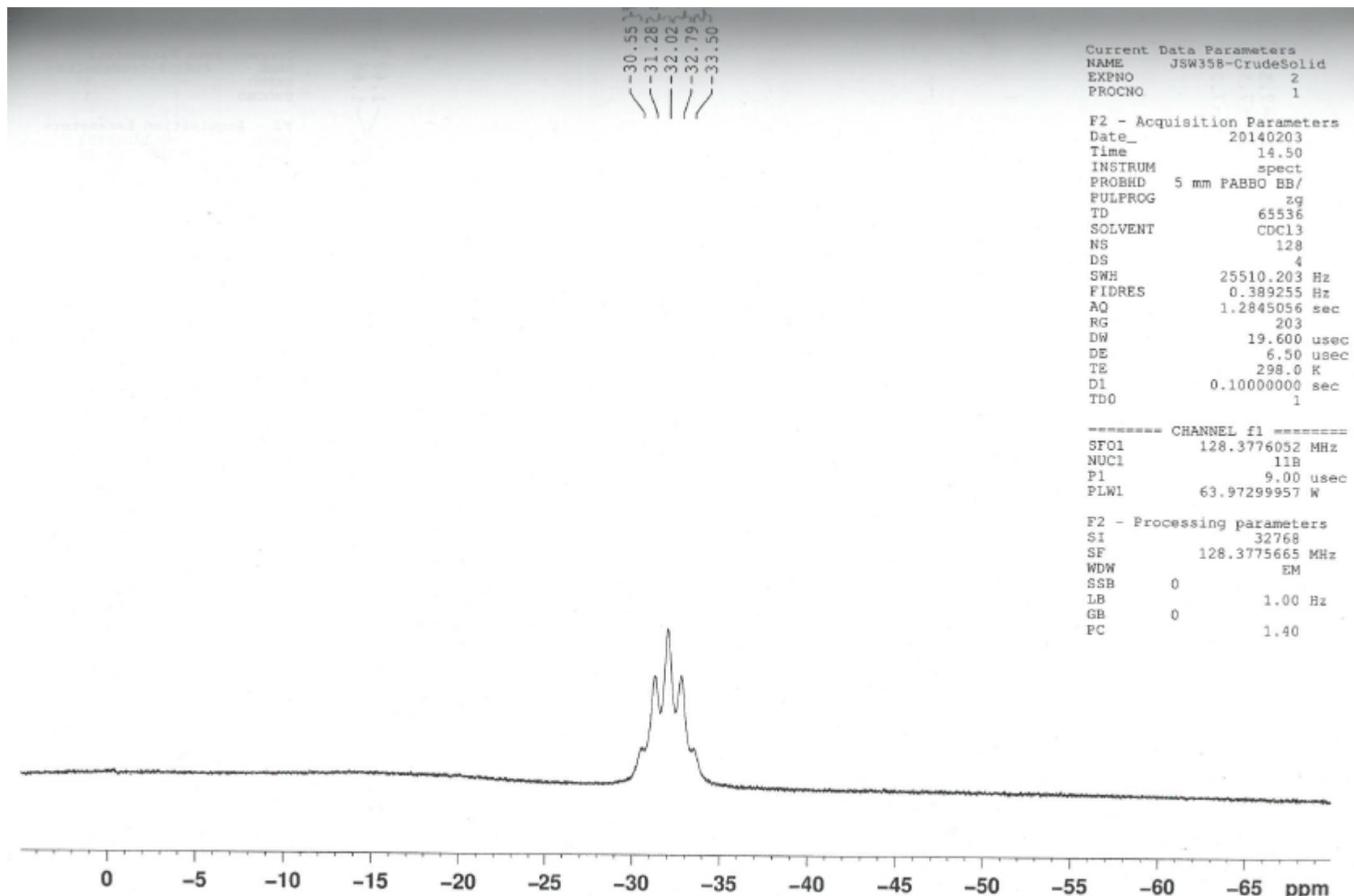
Compound 19 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum



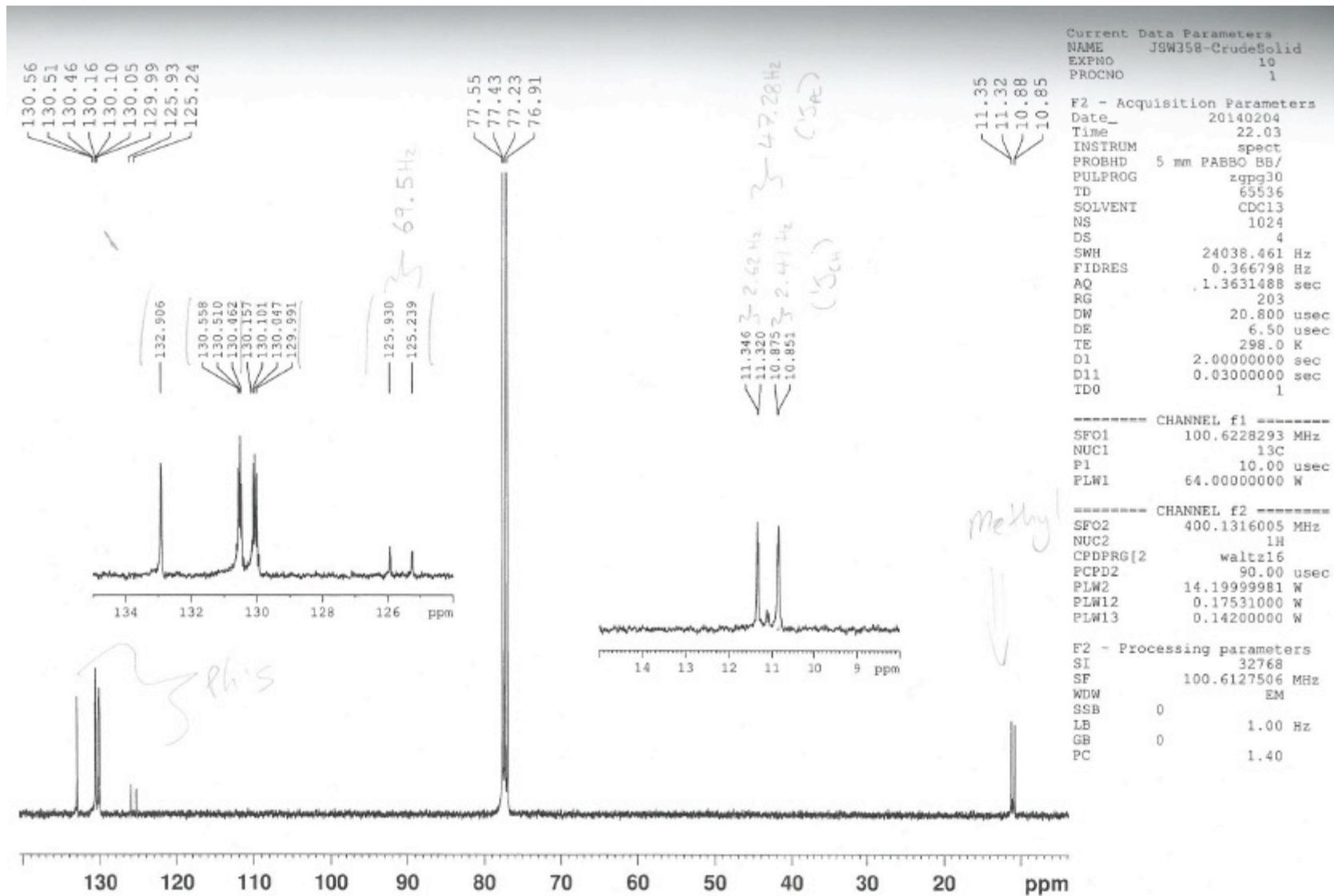
Compound 19 – $^1\text{H}\{{}^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)



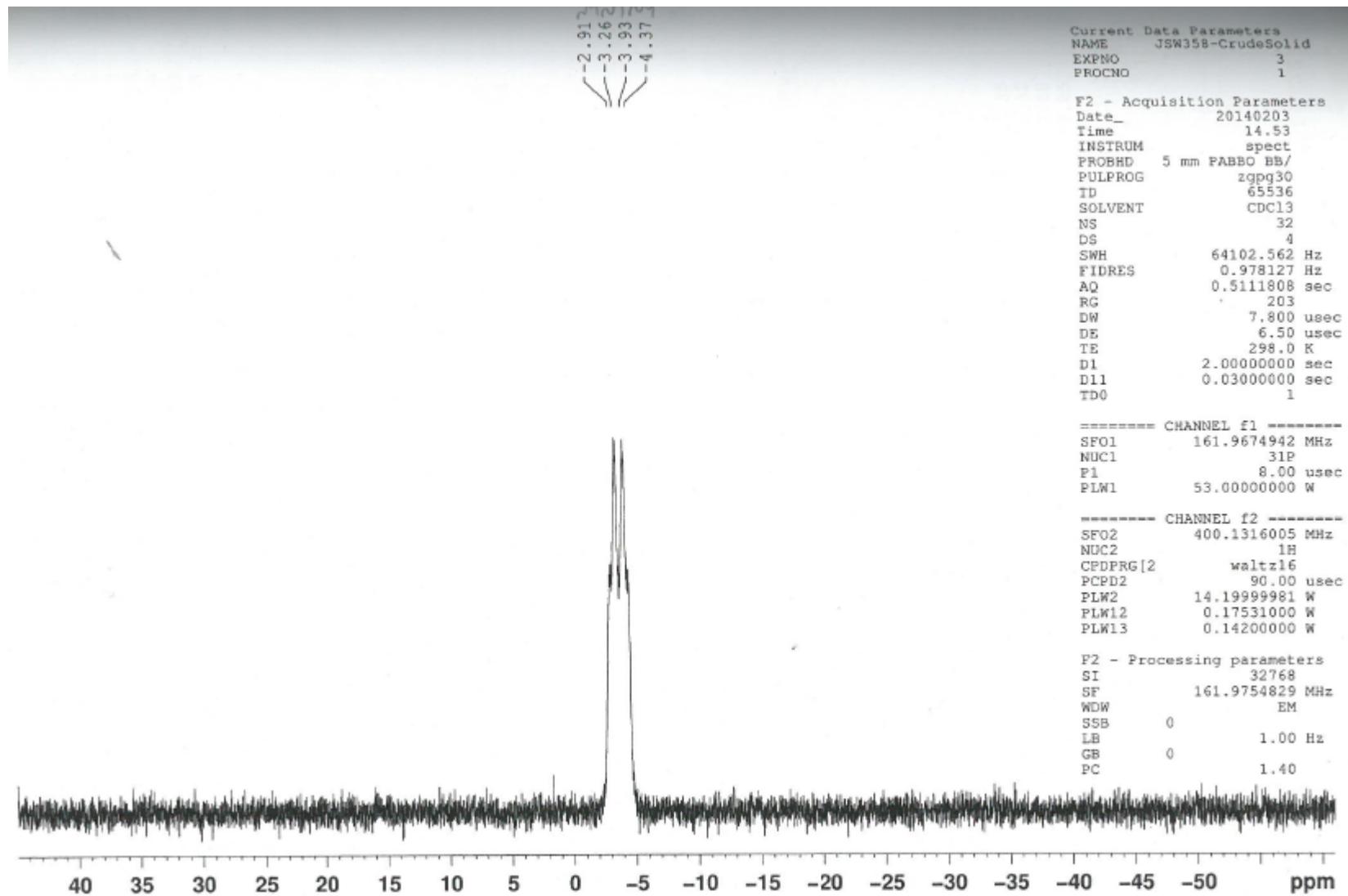
Compound 19 – $^{11}\text{B}\{\text{H}\}$ NMR spectrum



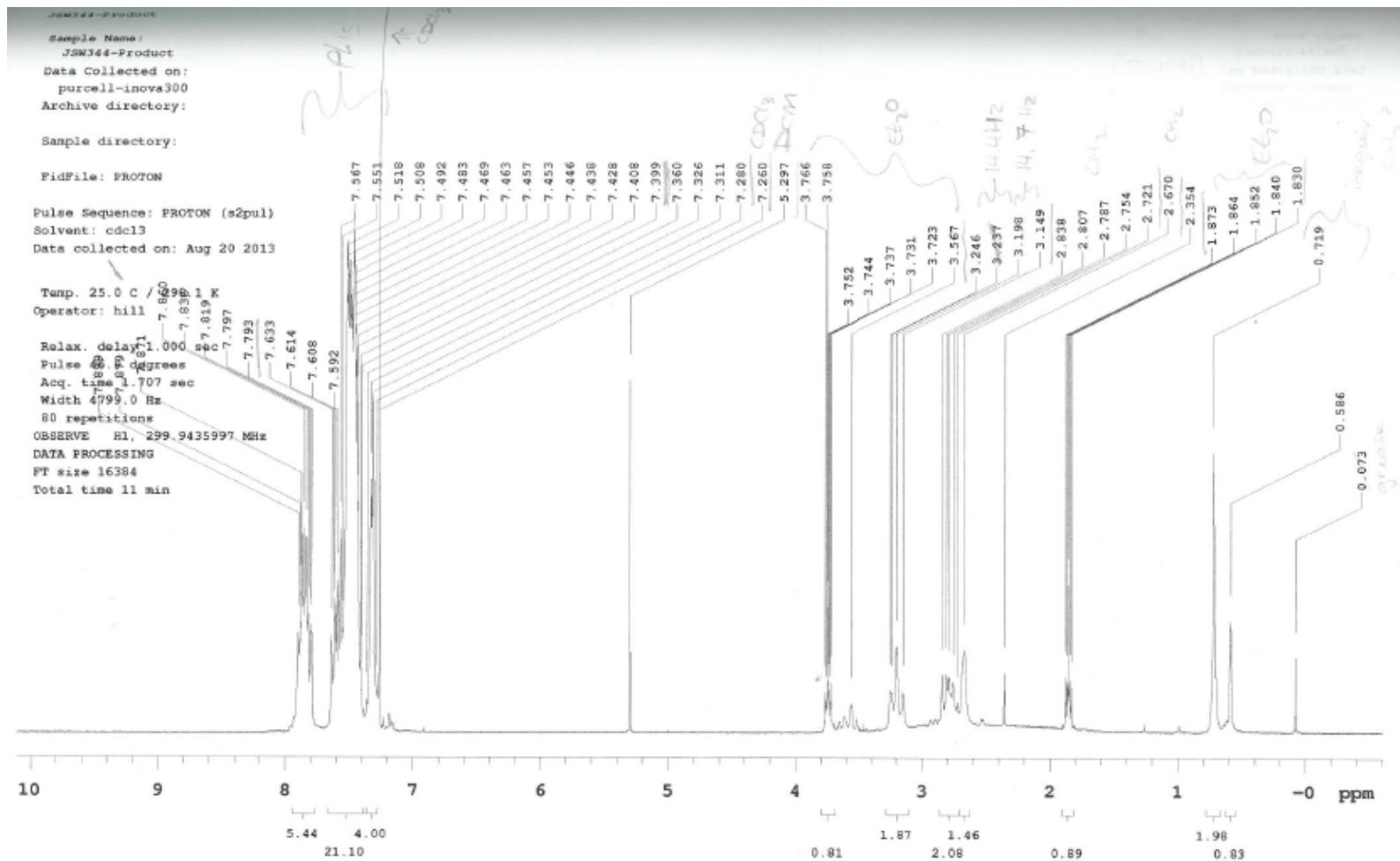
Compound 19 – ^{11}B NMR spectrum



Compound 19 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



Compound 19 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum



Compound 20 – ^1H NMR spectrum

JSW344-Product
Sample Name:
JSW344-Product
Data Collected on:
purcell-inova300
Archive directory:

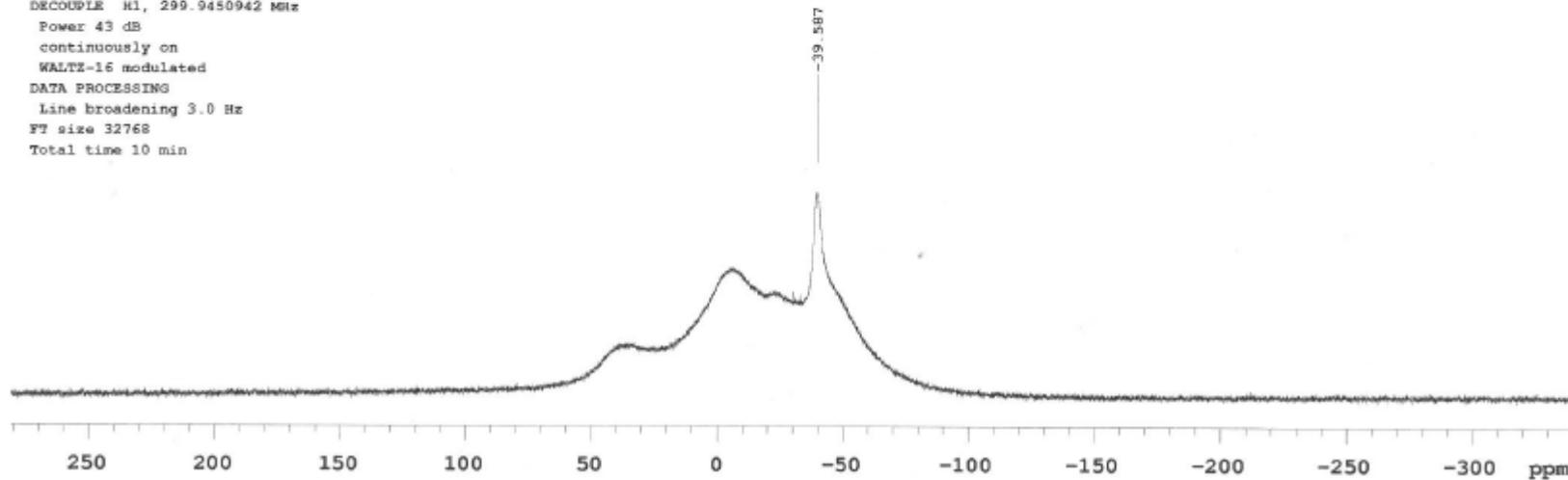
Sample directory:

FidFile: stdlh

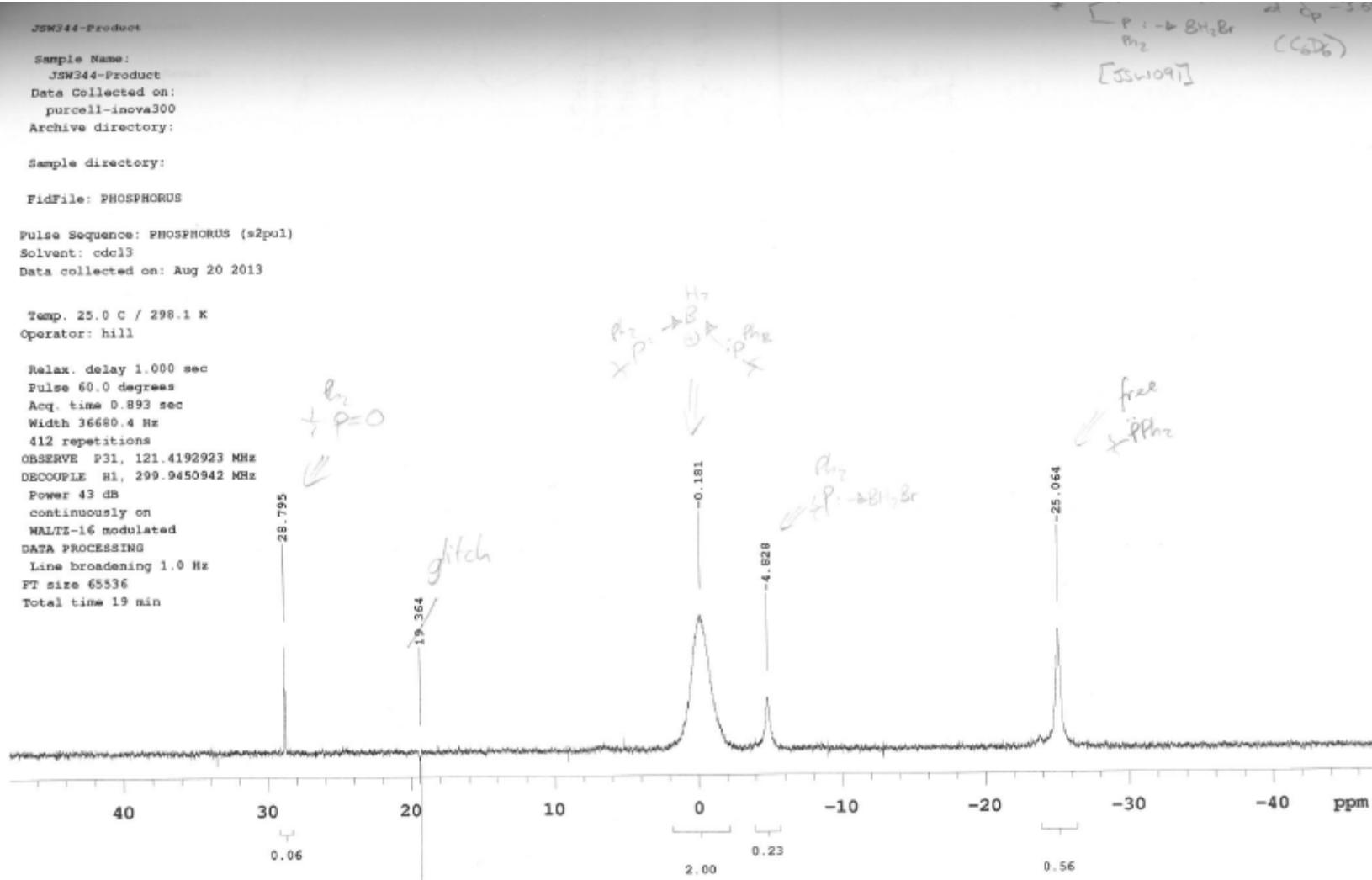
Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Aug 20 2013

Temp. 25.0 C / 298.1 K
Operator: hill

Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
Width 60015.0 Hz
960 repetitions
OBSERVE B11, 96.2339197 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 10 min



Compound 20 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 20 – $^{31}\text{P}\{\text{H}\}$ NMR spectrum