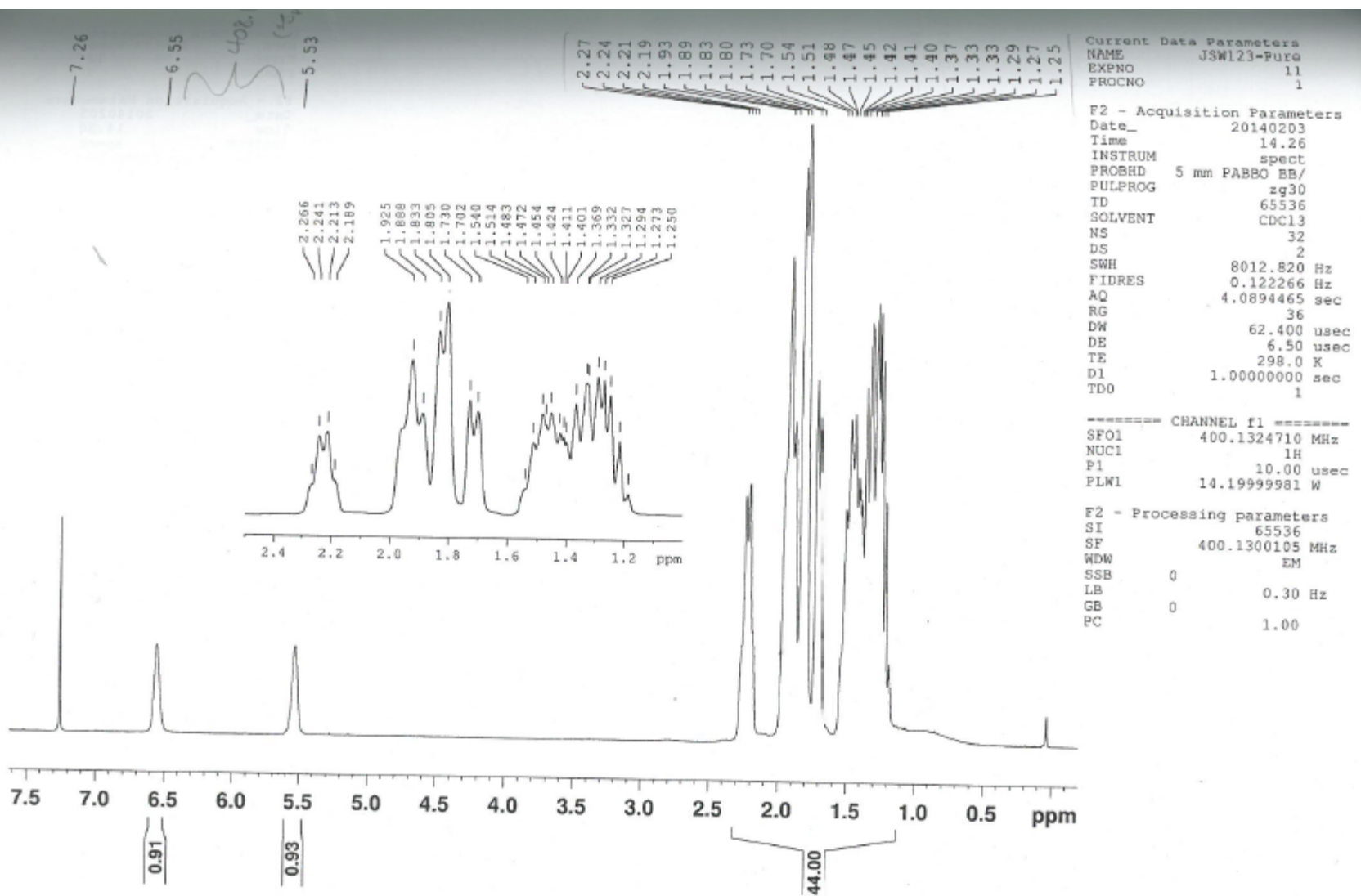


Synthetic and Structural Studies of Phosphine Coordinated Boronium Salts

Anthony F. Hill*^a and Jas S. Ward^a

^aResearch School of Chemistry, Australian National University, Canberra, ACT 2601, Australia.

Electronic Supporting Information (ESI)



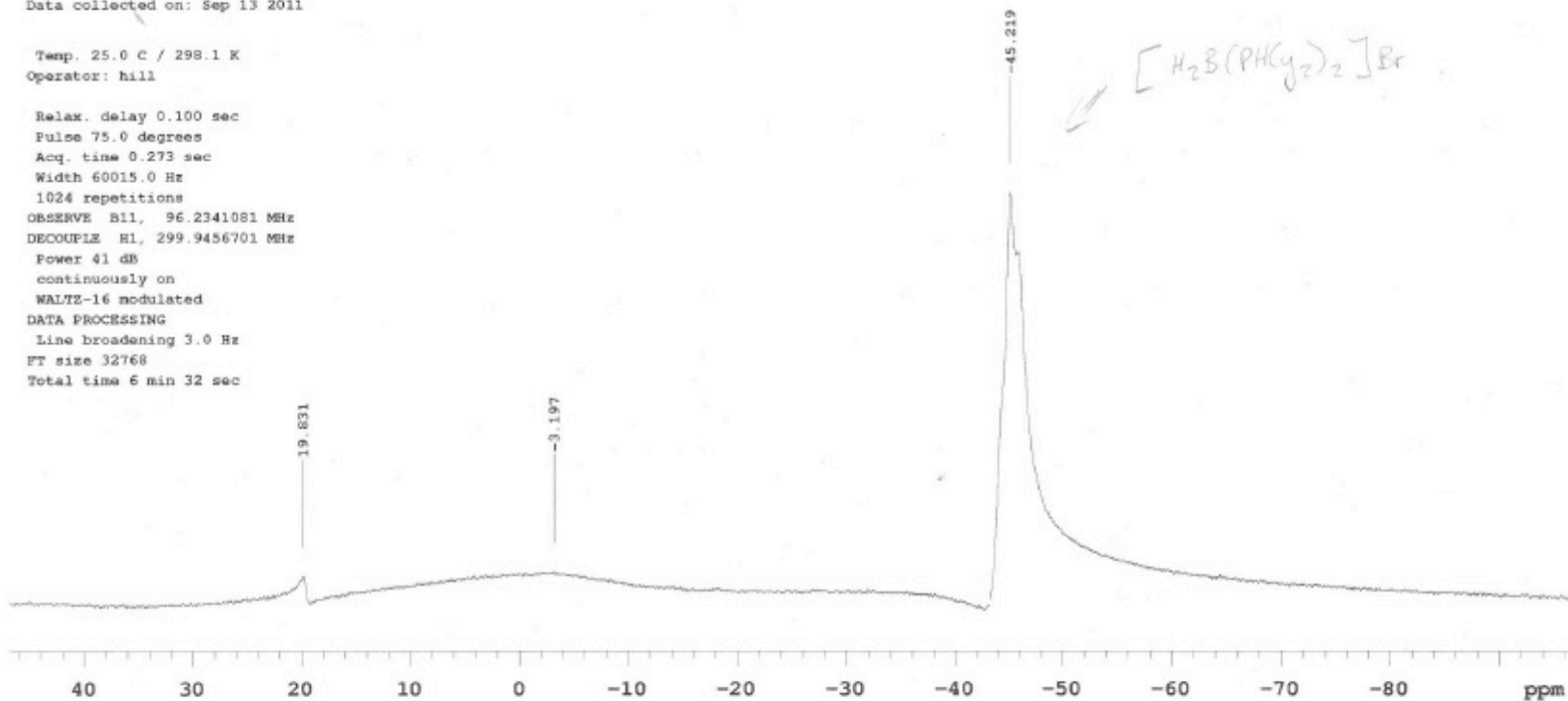
Compound 1 - ¹H NMR spectrum

JSW123-DCM Pent. Precip.
 Sample Name:
 JSW123-DCM Pent. Precip.
 Data Collected on:
 purcell-inova300
 Archive directory:
 /home/hill/vnmrsys/data
 Sample directory:
 MS-43-highC_20110913_01
 FidFile: std1h

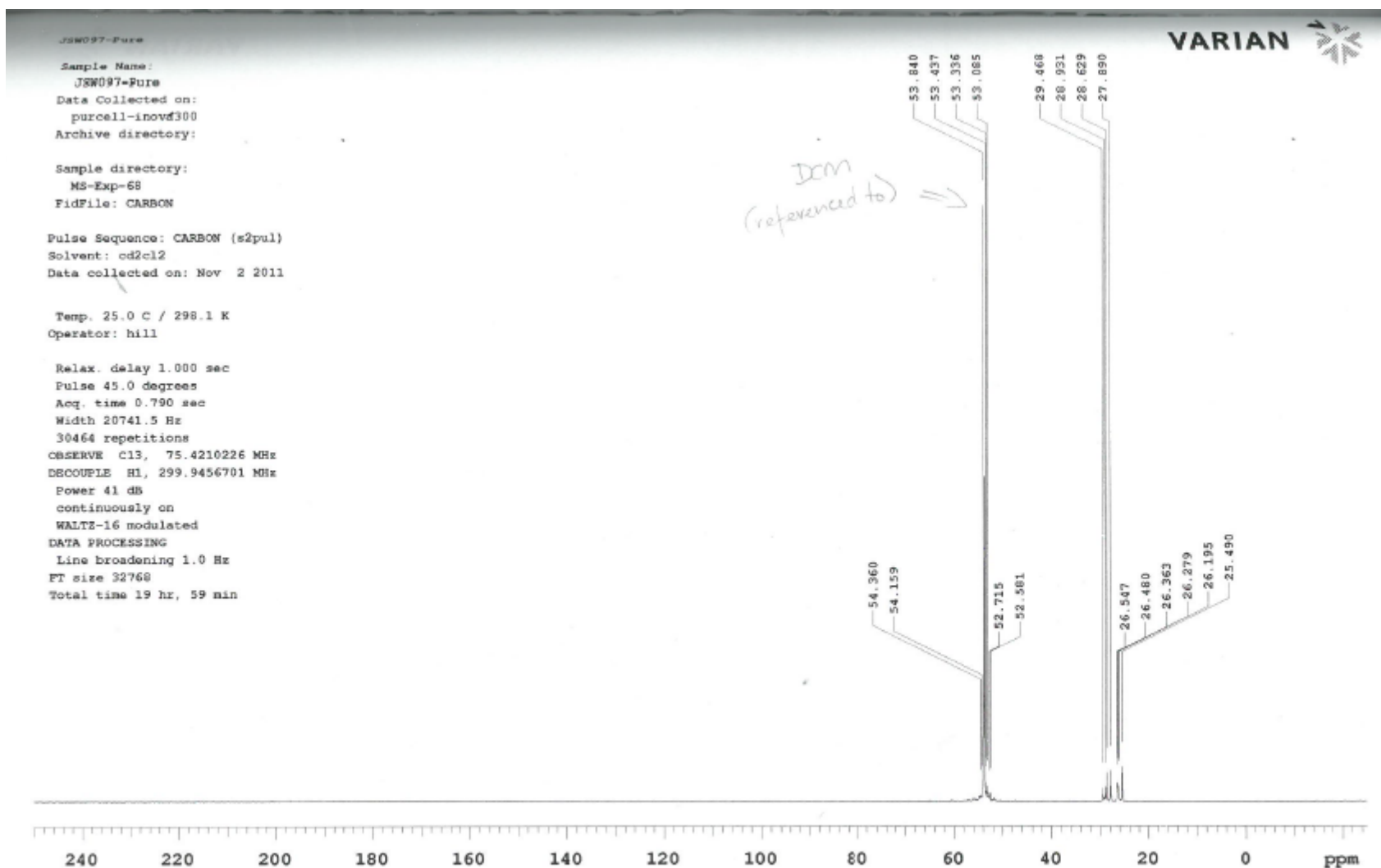
Pulse Sequence: std1h (s2pul)
 Solvent: cd2cl2
 Data collected on: Sep 13 2011

Temp. 25.0 C / 298.1 K
 Operator: hill

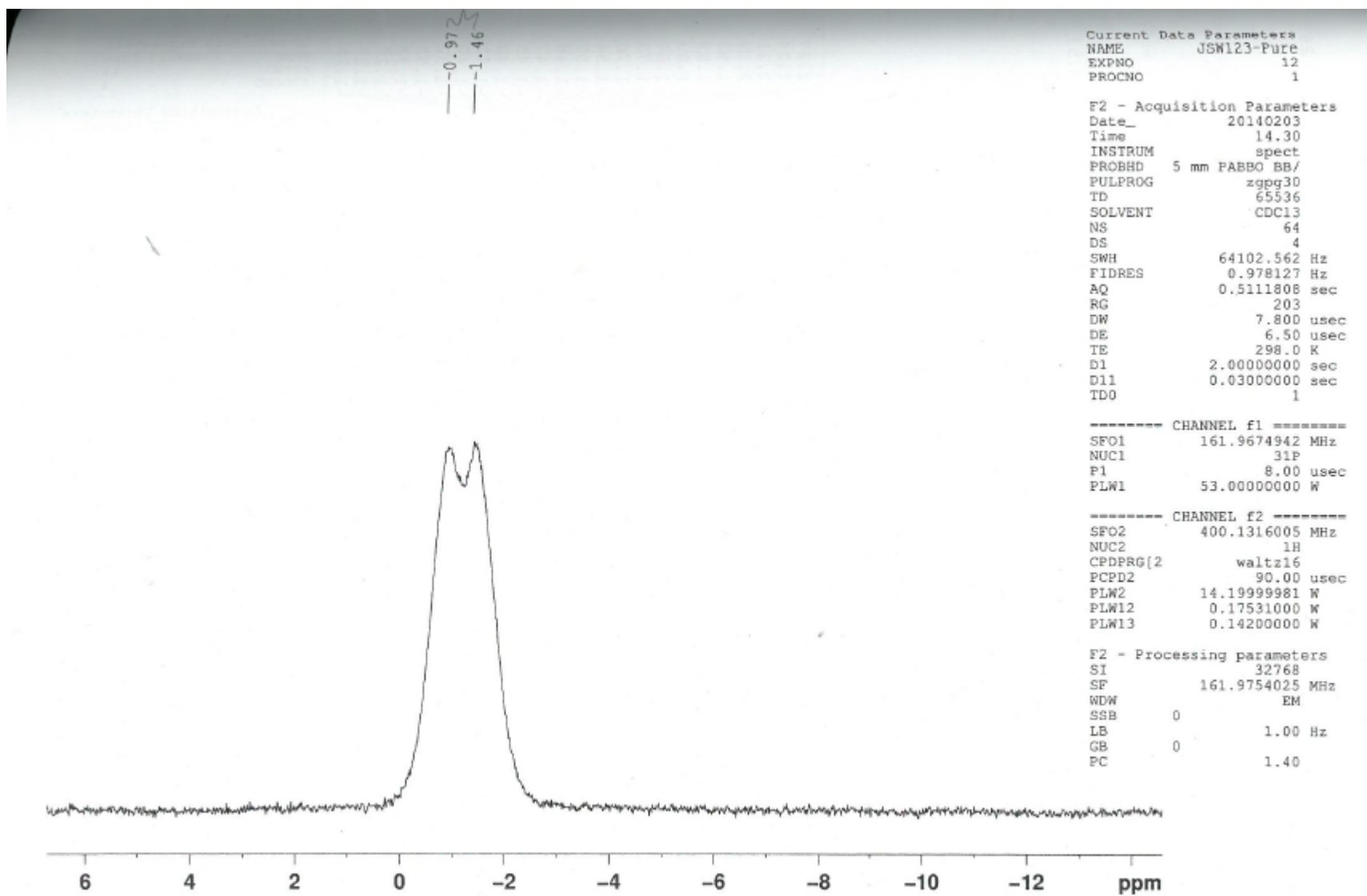
Relax. delay 0.100 sec
 Pulse 75.0 degrees
 Acq. time 0.273 sec
 Width 60015.0 Hz
 1024 repetitions
 OBSERVE B11, 96.2341081 MHz
 DECOUPLE H1, 299.9456701 MHz
 Power 41 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 32768
 Total time 6 min 32 sec



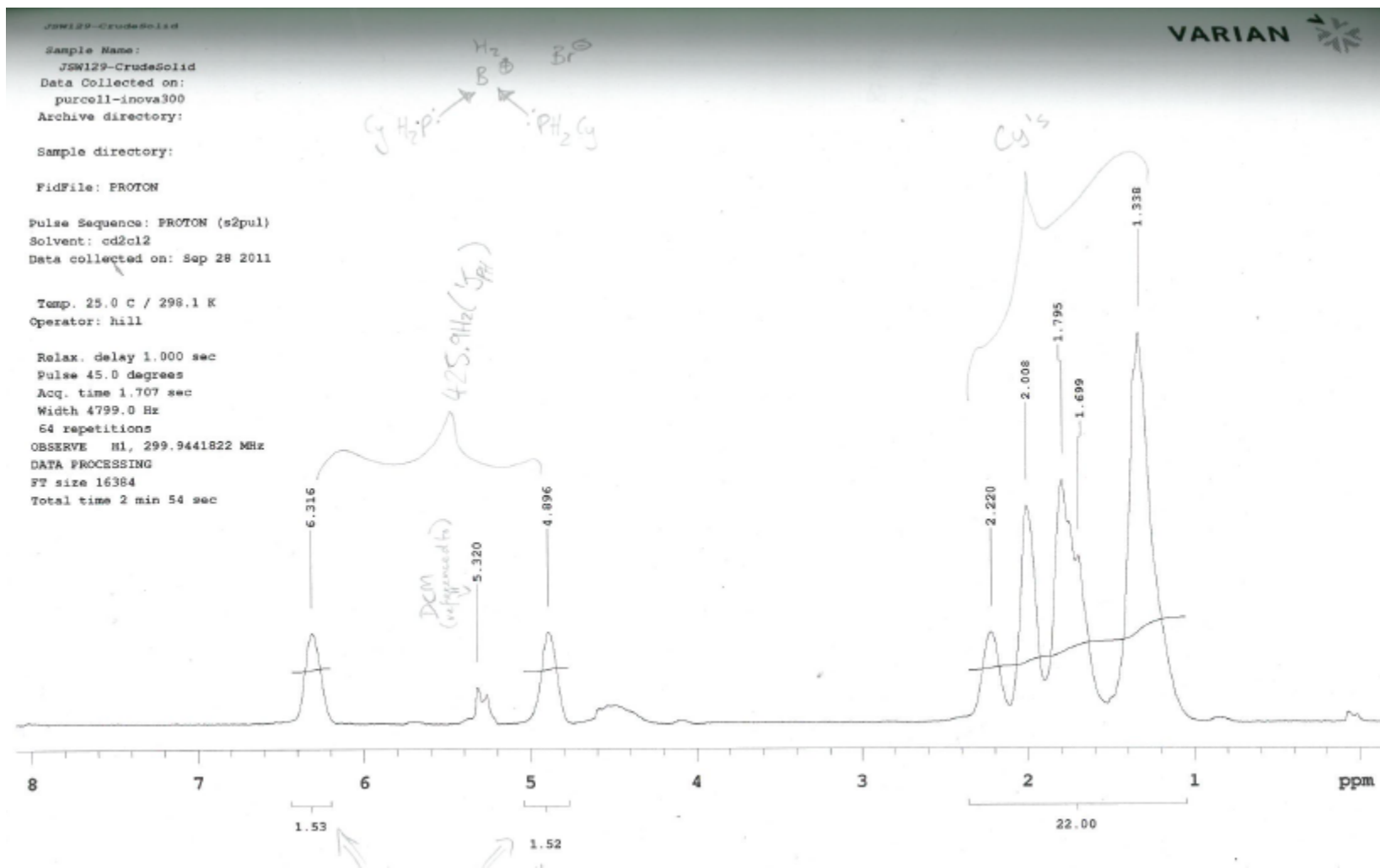
Compound 1 – $^{11}B\{^1H\}$ NMR spectrum



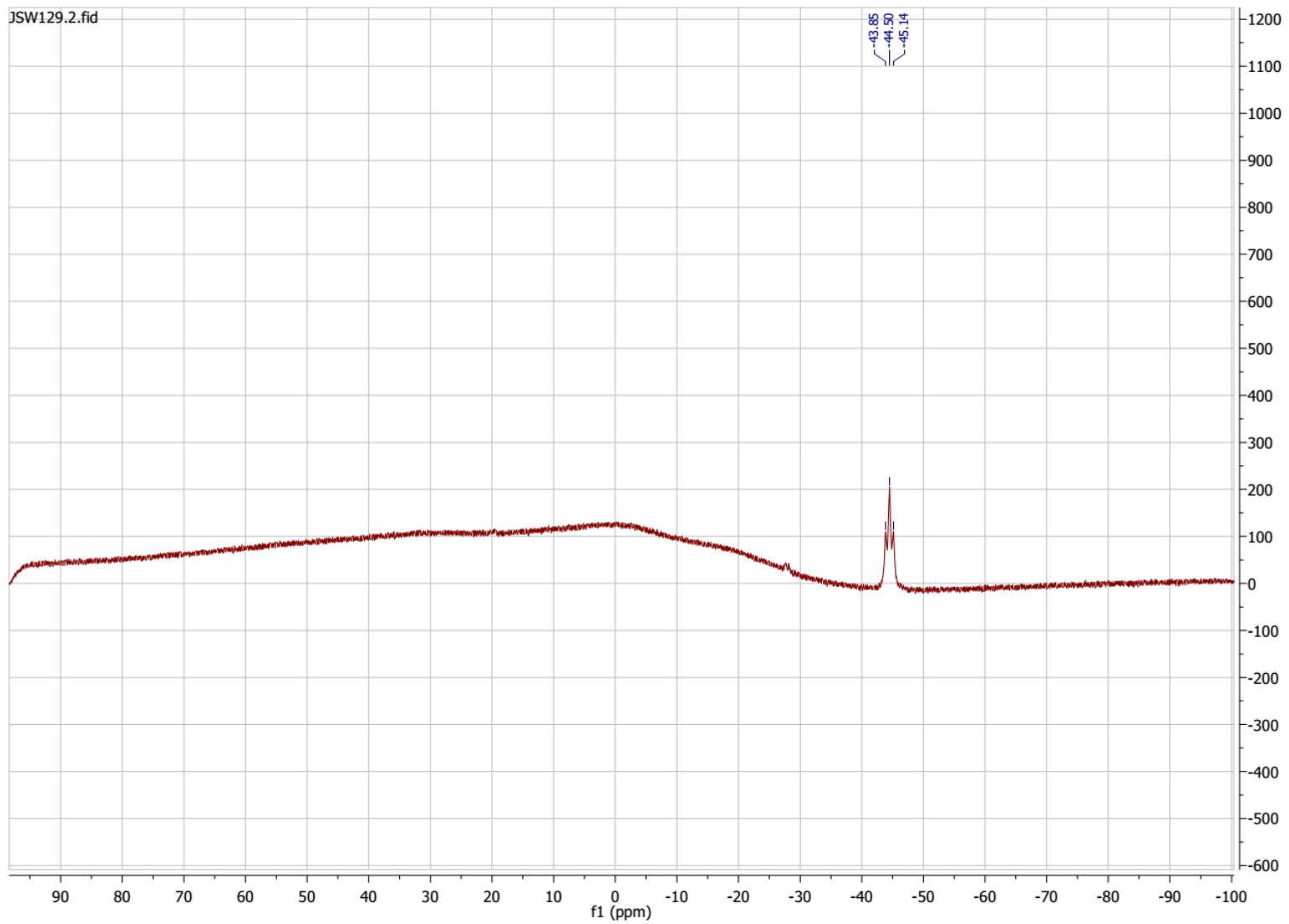
Compound 1 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



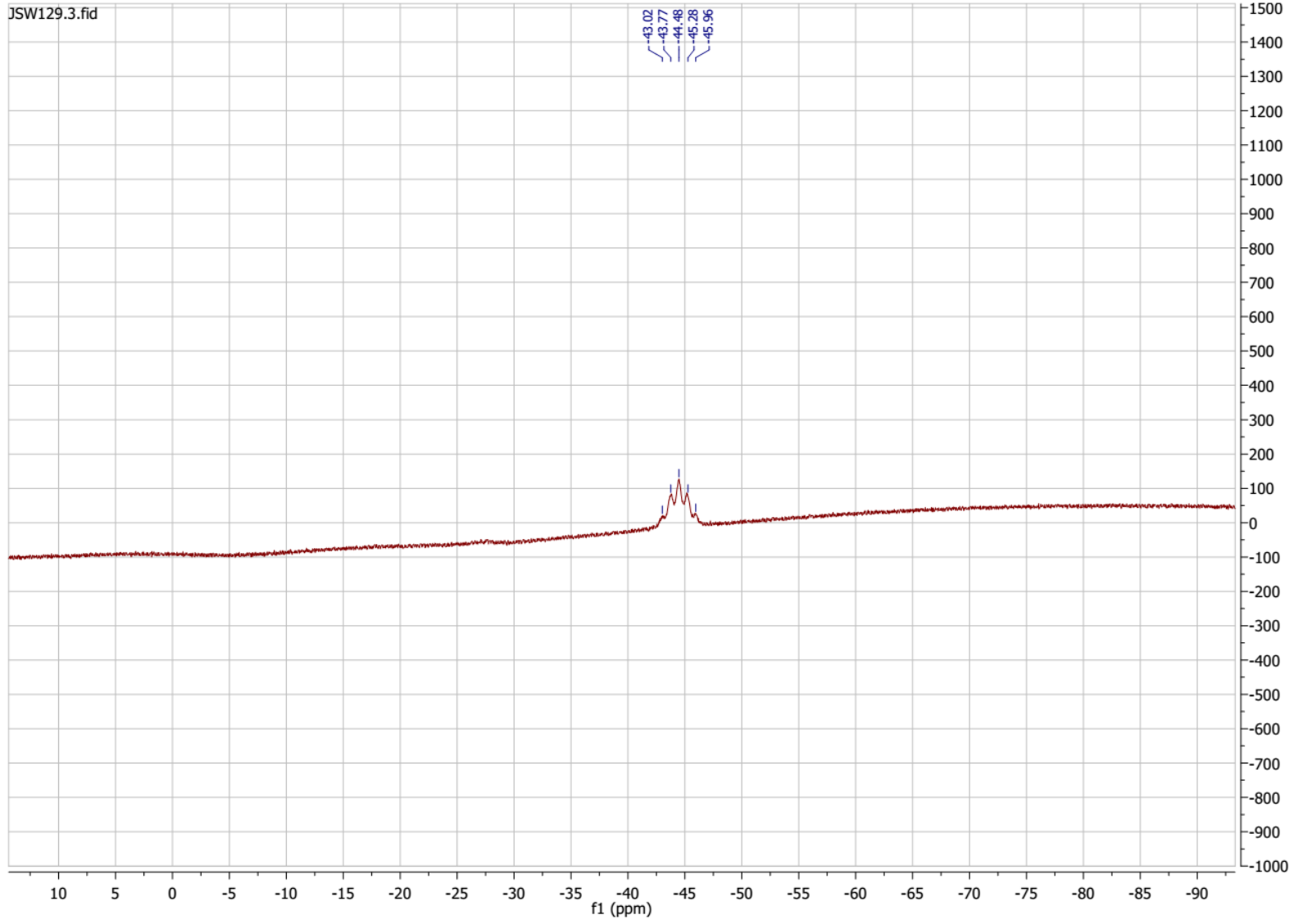
Compound 1 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 3 – ^1H NMR spectrum



Compound 3 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 3 – ^{11}B NMR spectrum

J5W129-Pure

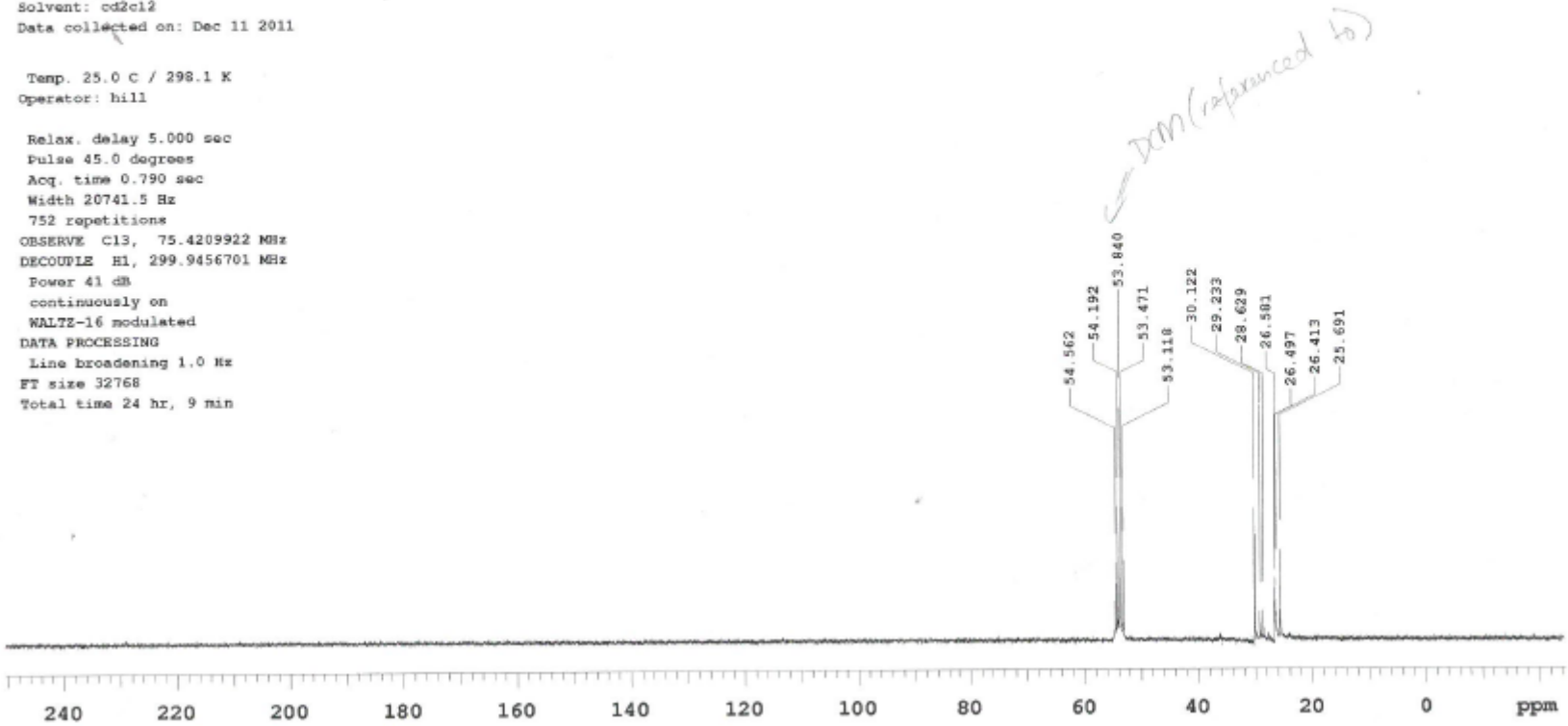
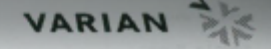
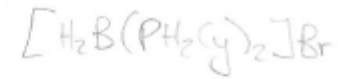
Sample Name:
J5W129-Pure
Data Collected on:
purcell-inova300
Archive directory:

Sample directory:
MS-Exp-79
FidFile: CARBON

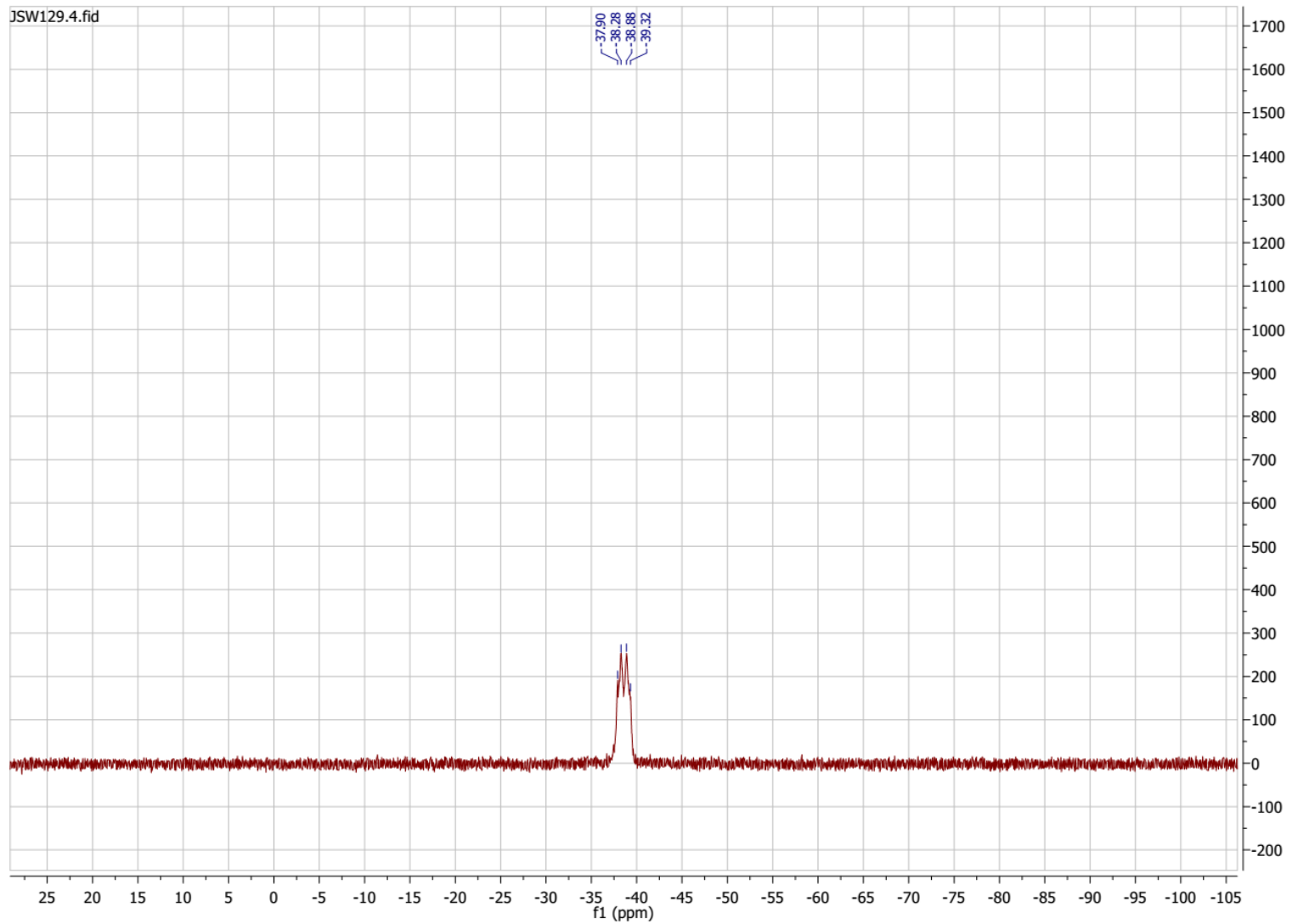
Pulse Sequence: CARBON (s2pul)
Solvent: cd2cl2
Data collected on: Dec 11 2011

Temp. 25.0 C / 298.1 K
Operator: hill

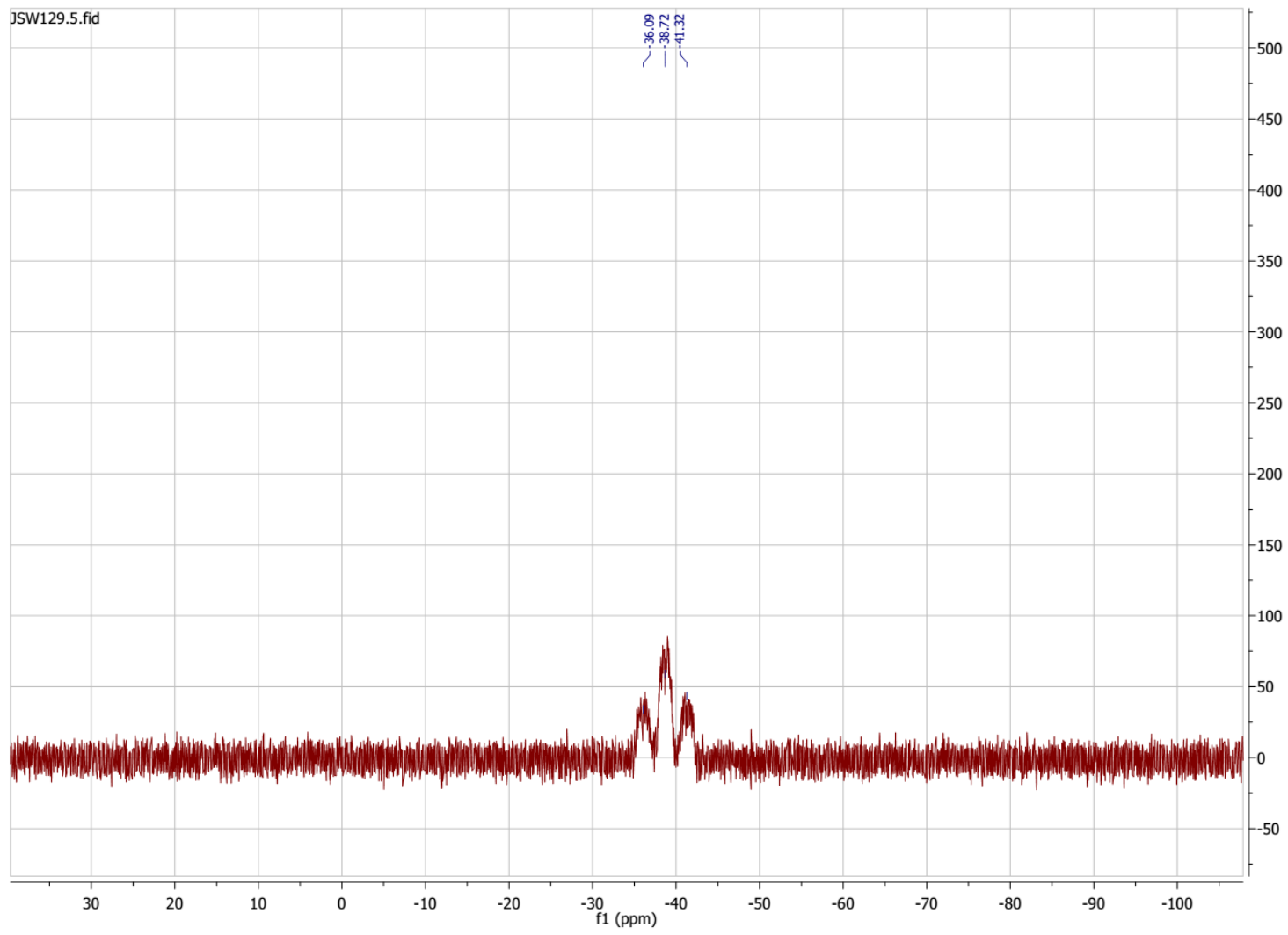
Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 0.790 sec
Width 20741.5 Hz
752 repetitions
OBSERVE C13, 75.4209922 MHz
DECOUPLE H1, 299.9456701 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 24 hr, 9 min



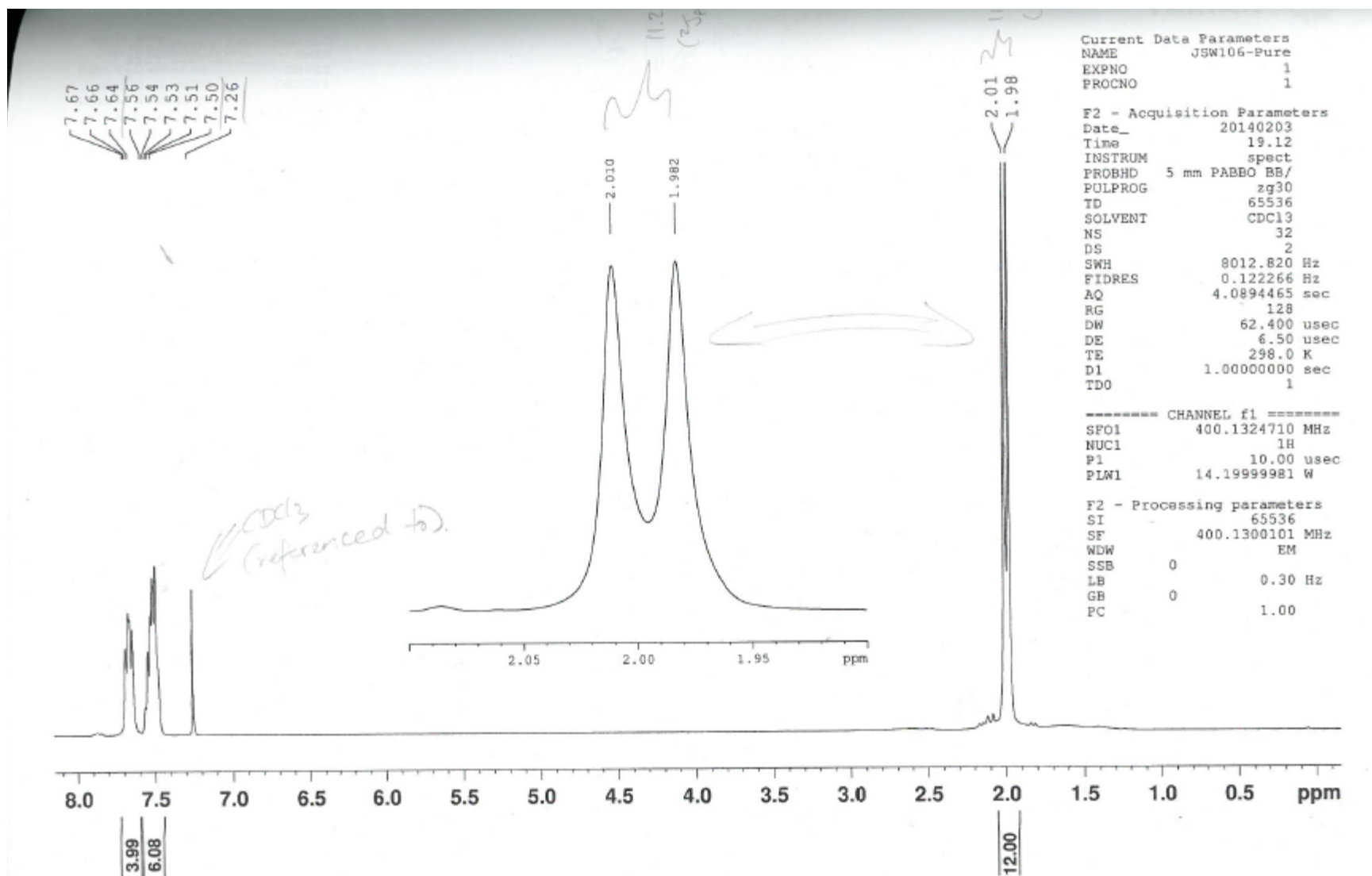
Compound 3 - $^{13}C\{^1H\}$ NMR spectrum



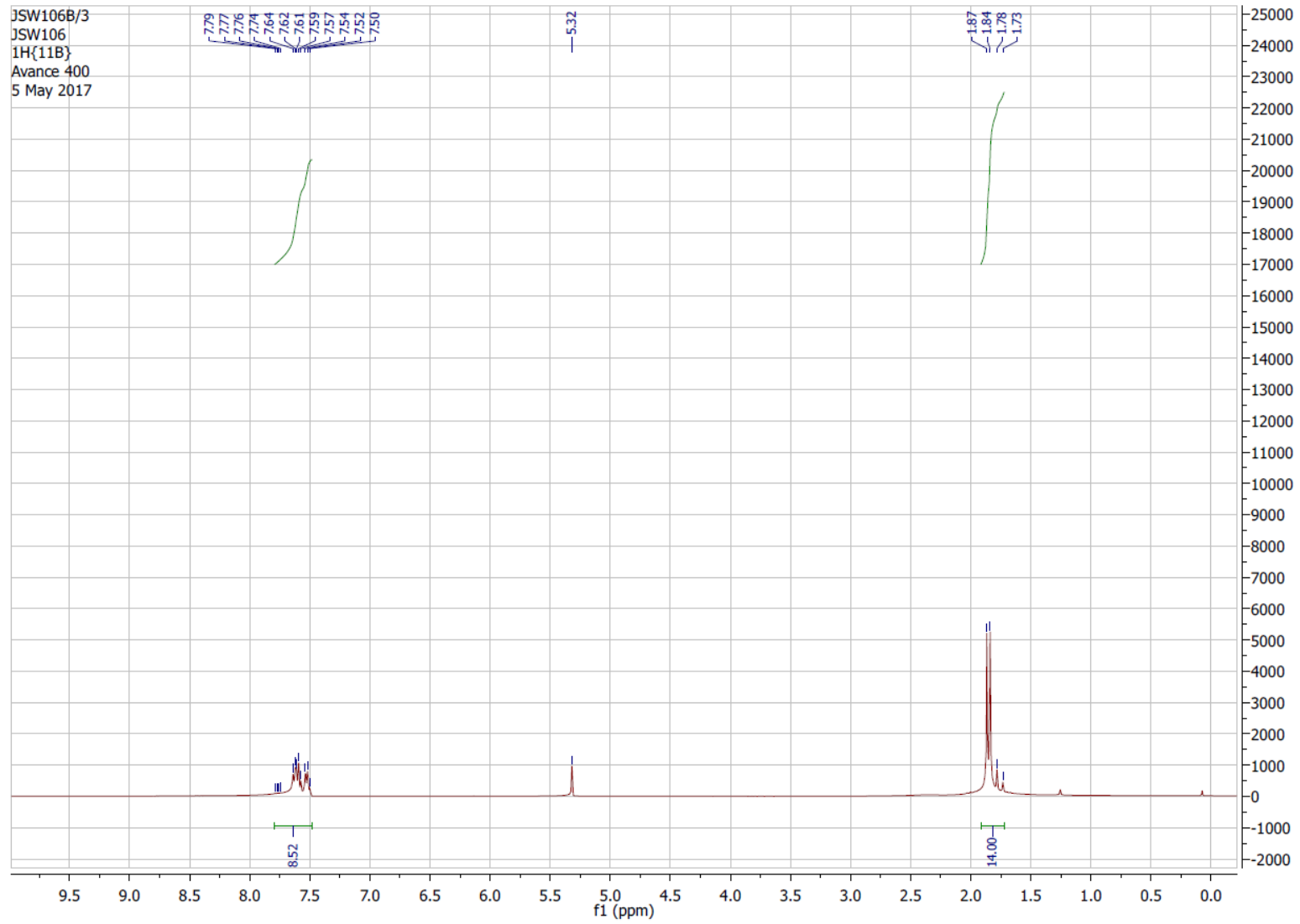
Compound 3 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



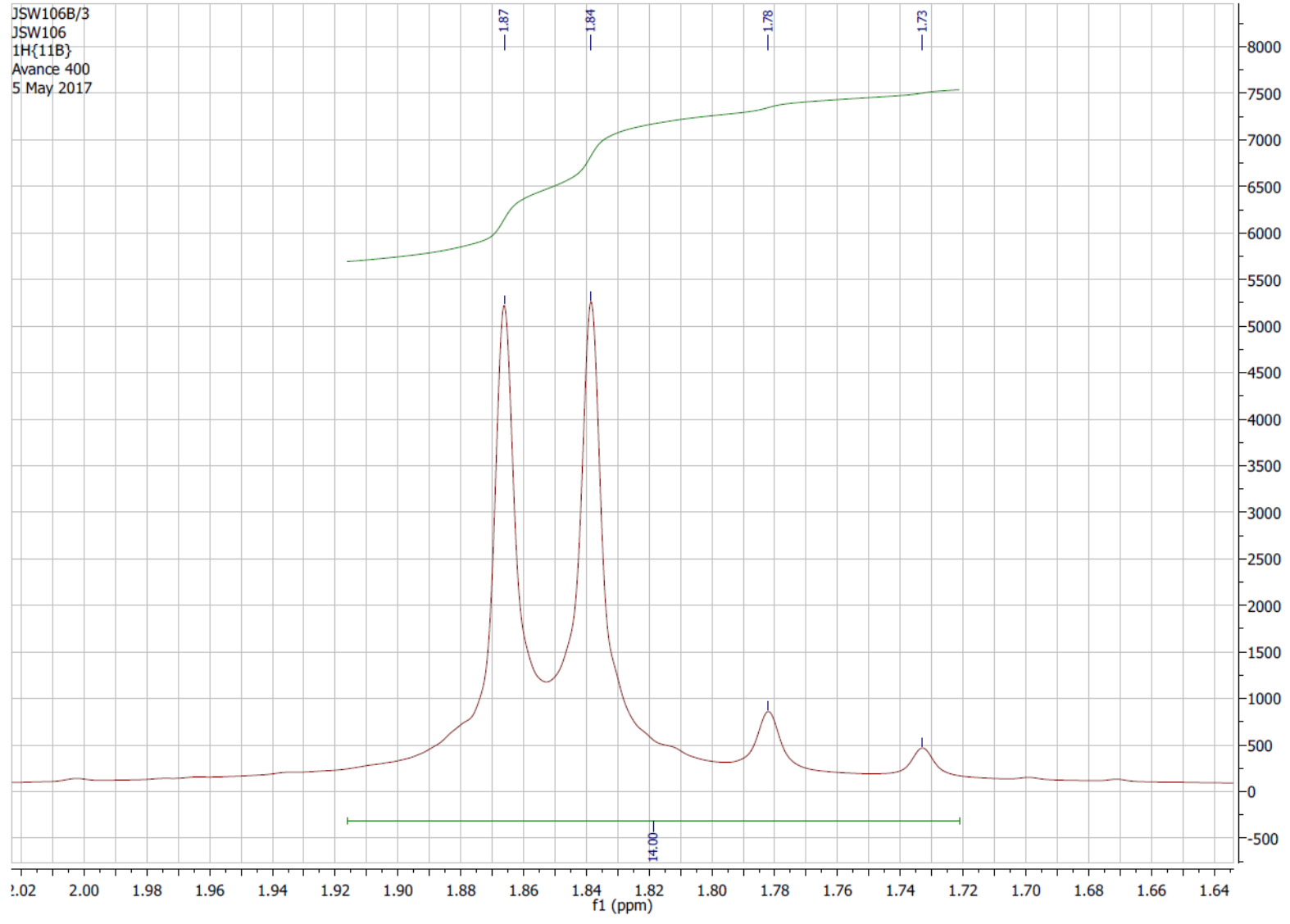
Compound 3 – ³¹P NMR spectrum



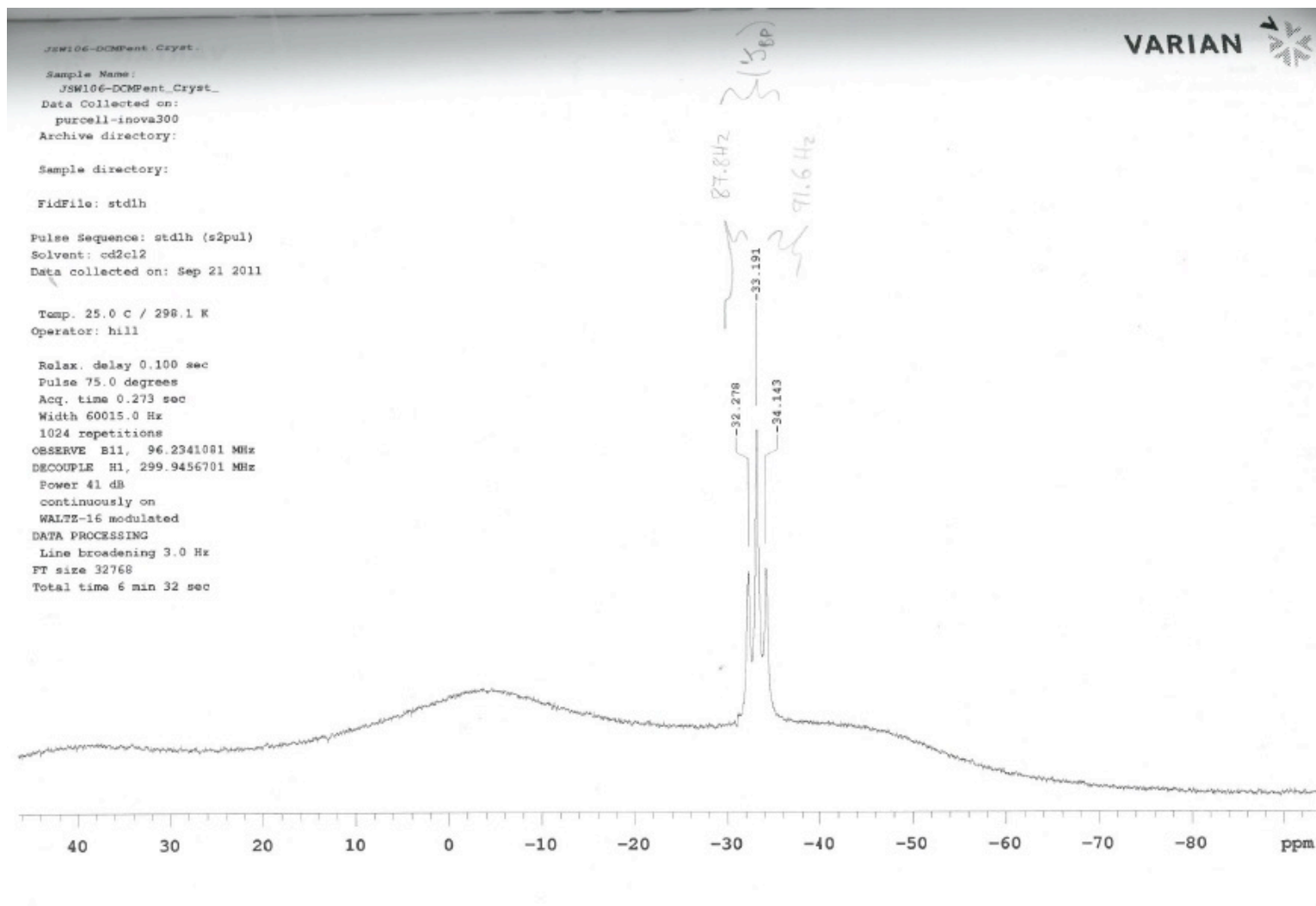
Compound 4 - ¹H NMR spectrum



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



Compound 4 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)



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

JSM106-DCMParnt_Cryst_

Sample Name:
JSM106-DCMParnt_Cryst_

Data Collected on:
purcell-inova300

Archive directory:

Sample directory:

FidFile: stdlh

Pulse Sequence: stdlh (s2pul)

Solvent: cd2cl2

Data collected on: Sep 21 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

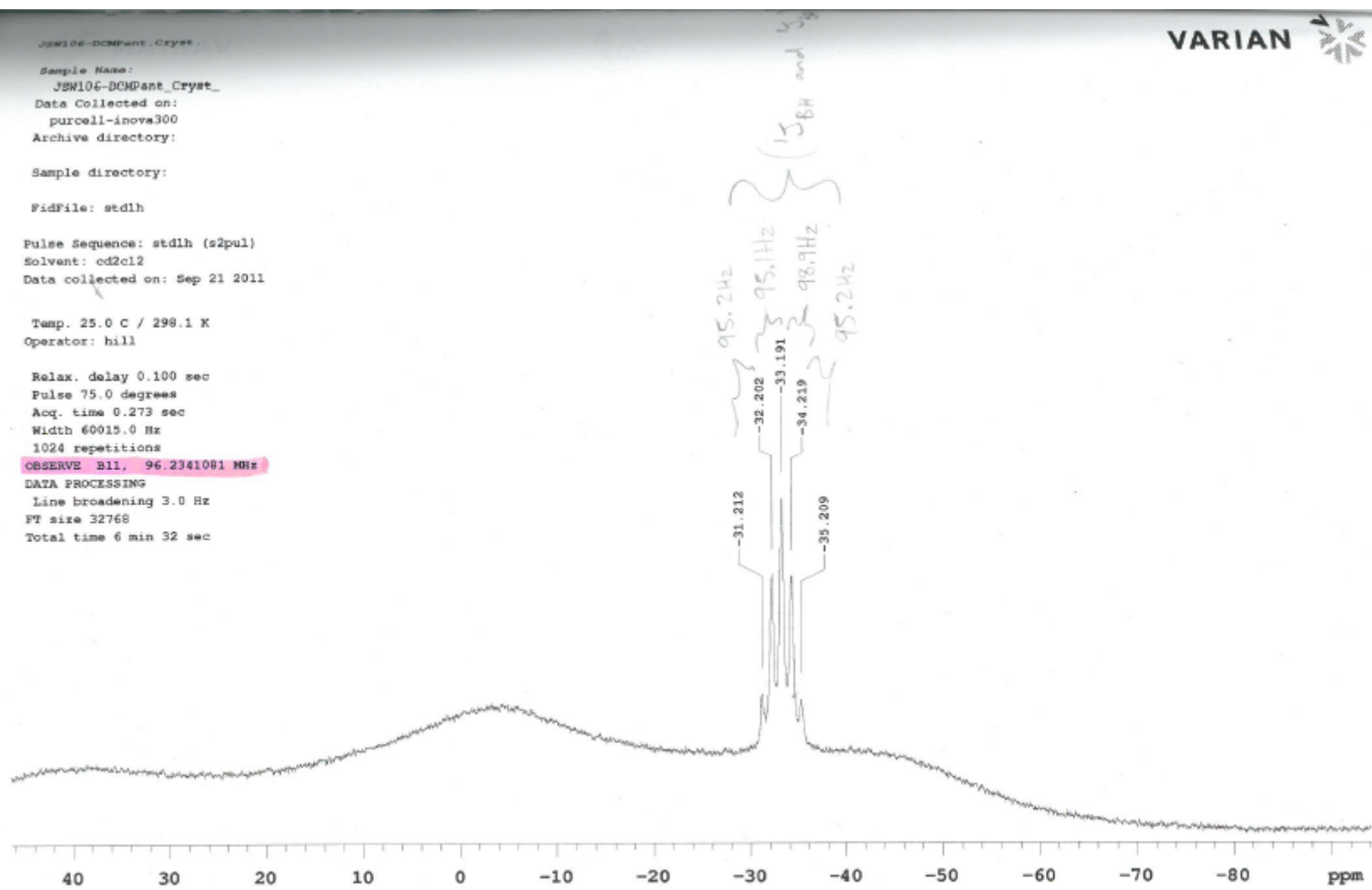
OBSERVE B11, 96.2341001 MHz

DATA PROCESSING

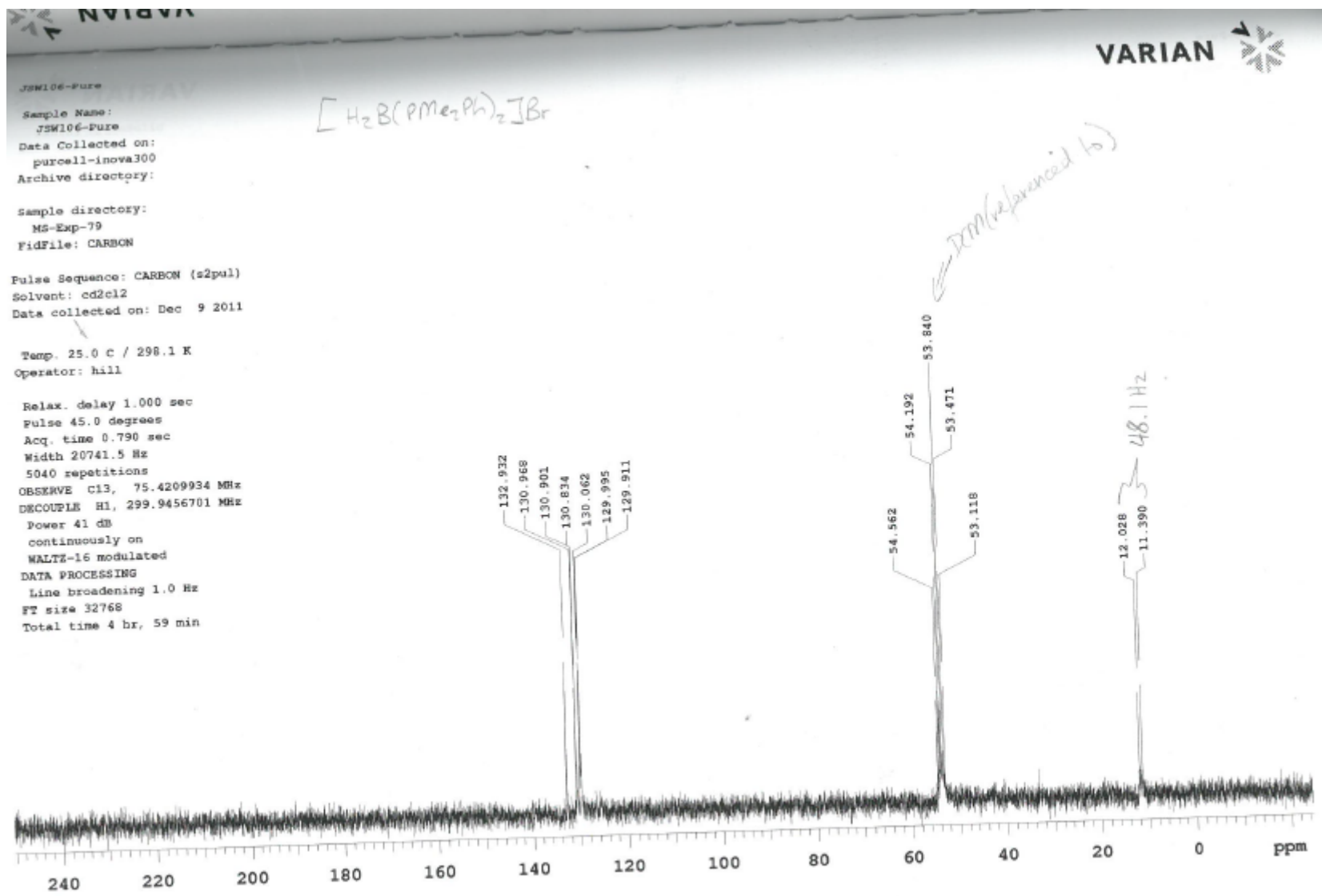
Line broadening 3.0 Hz

FT size 32768

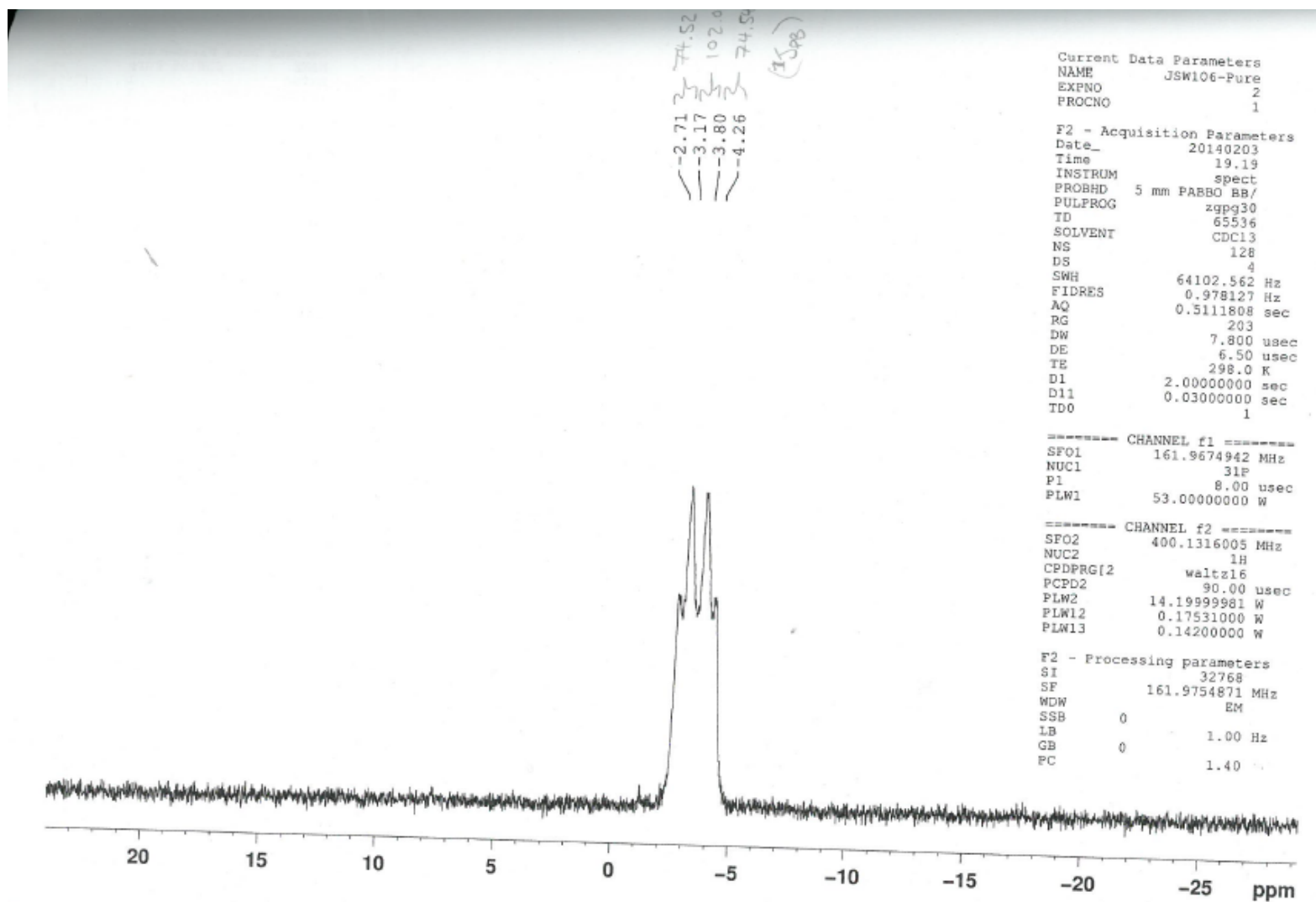
Total time 6 min 32 sec



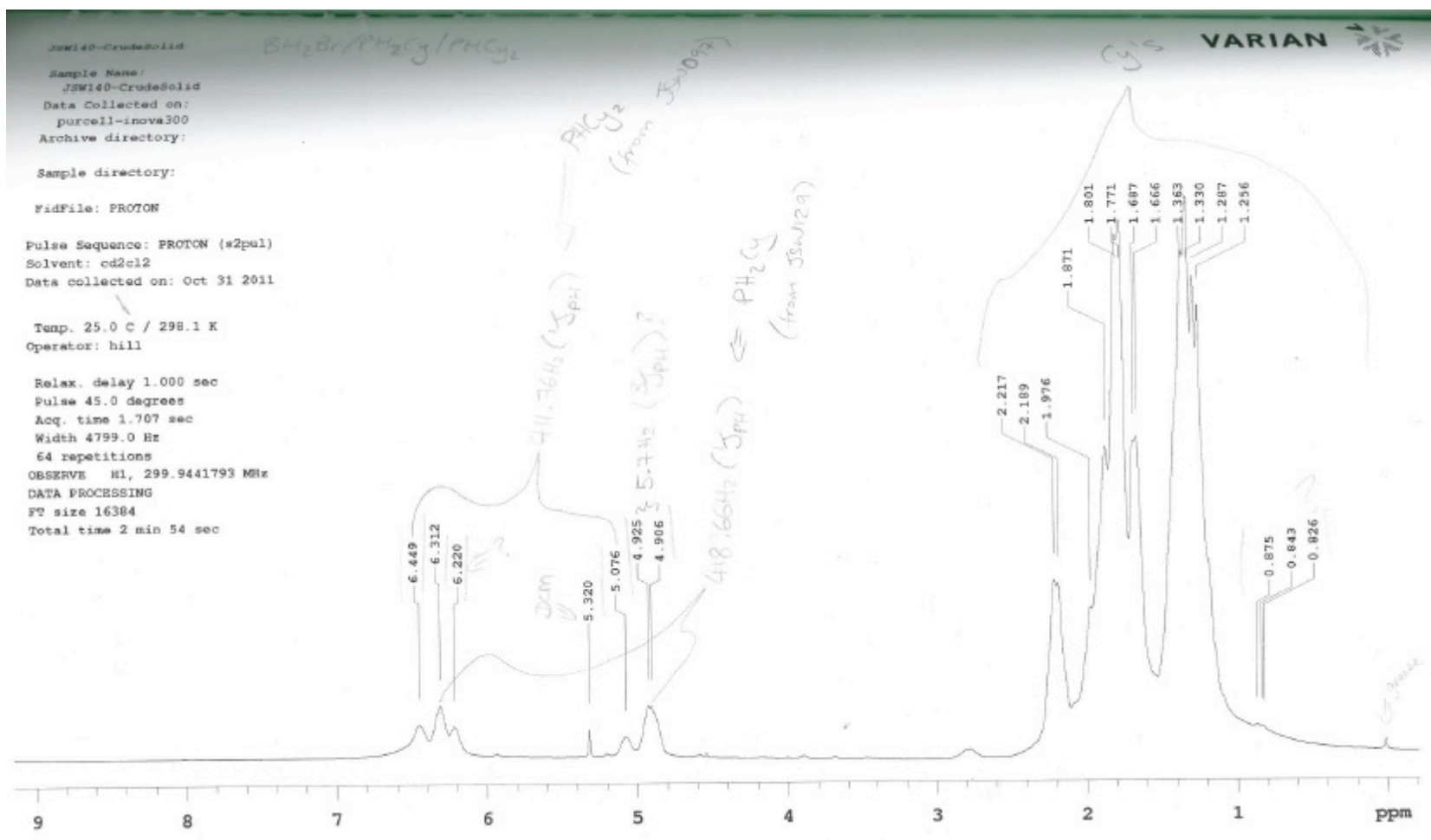
Compound 4 – ^{11}B NMR spectrum



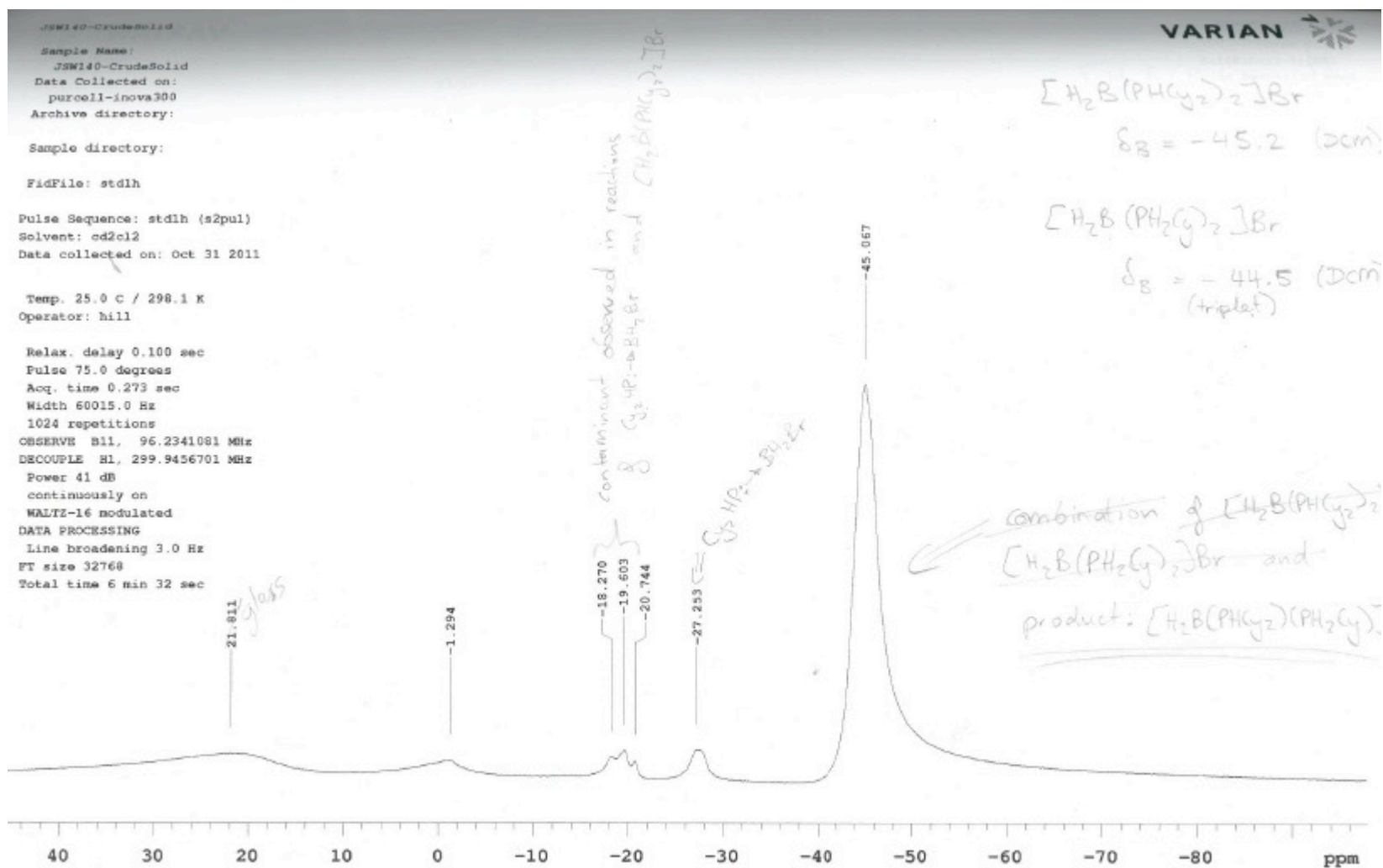
Compound 4 – ¹³C{¹H} NMR spectrum



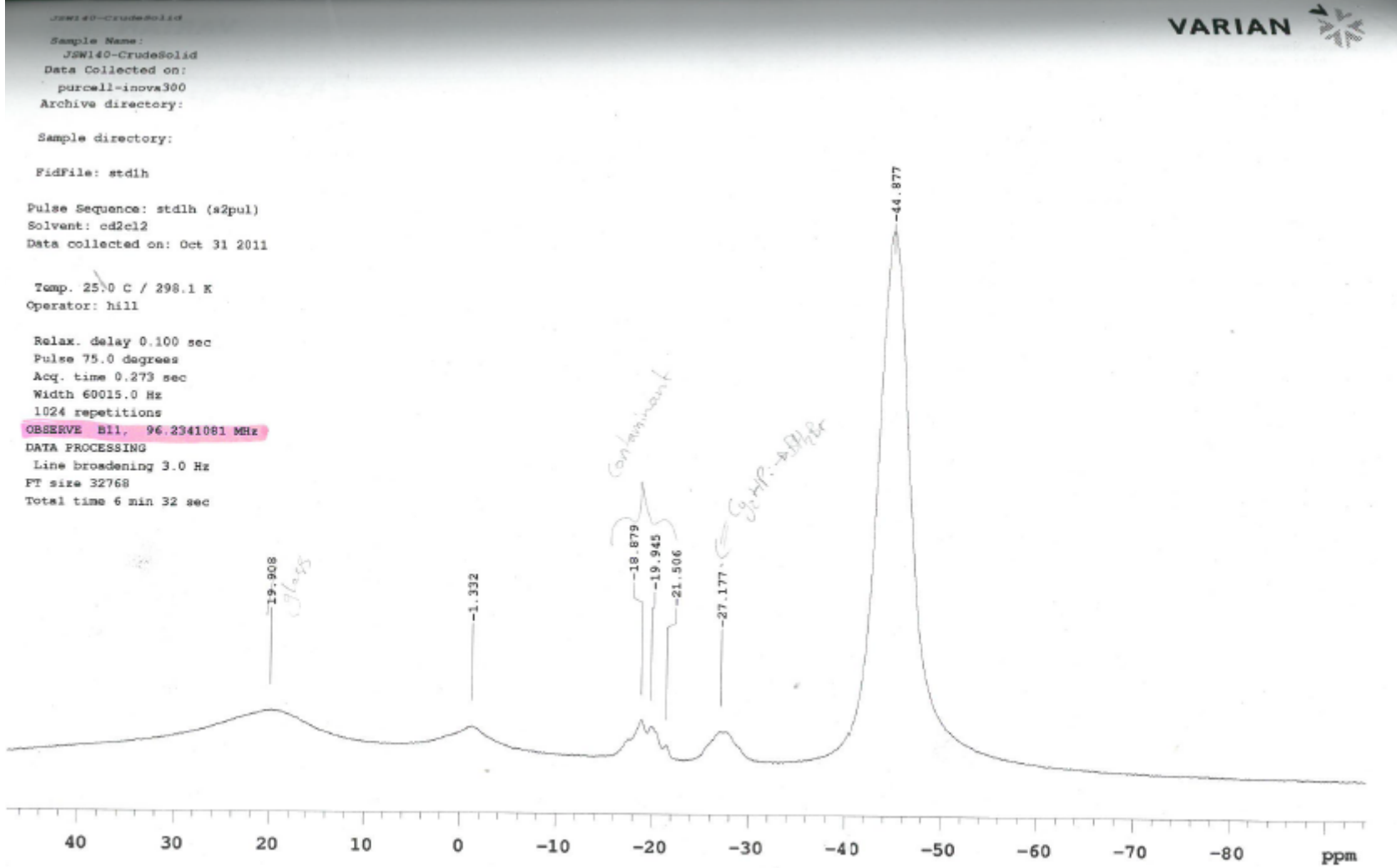
Compound 4 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



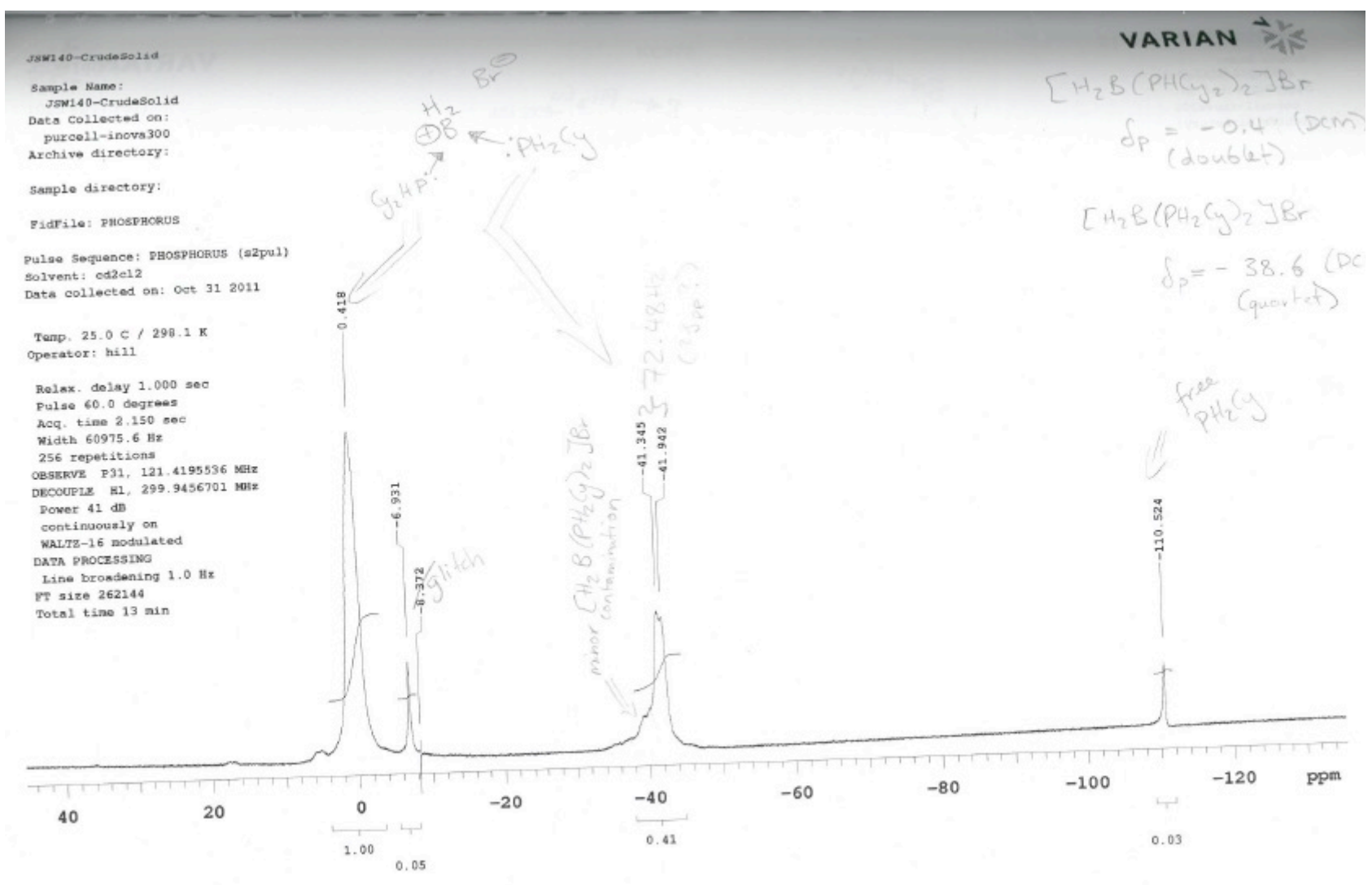
Compound 5 – ¹H NMR spectrum



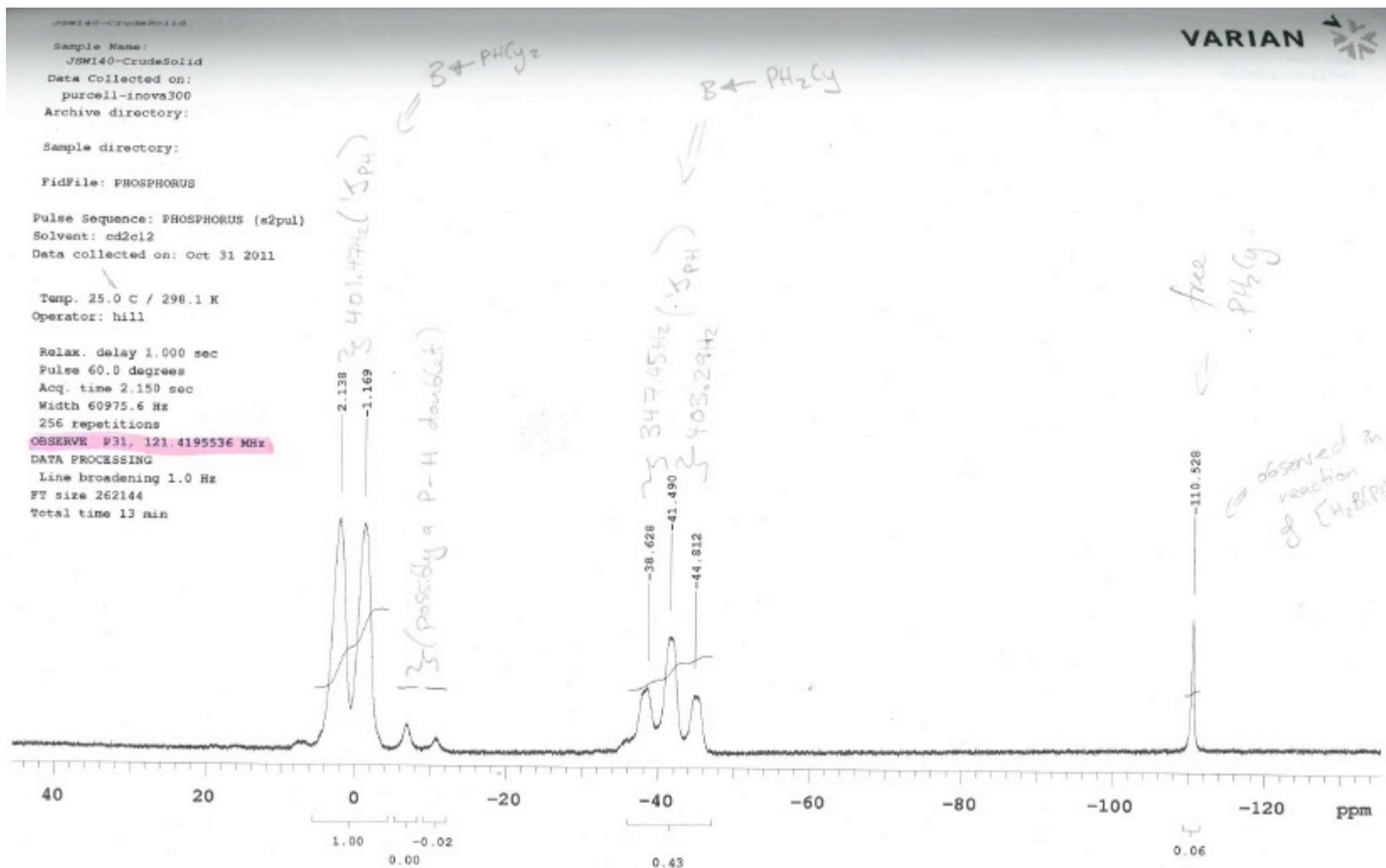
Compound 5 - $^{11}B\{^1H\}$ NMR spectrum



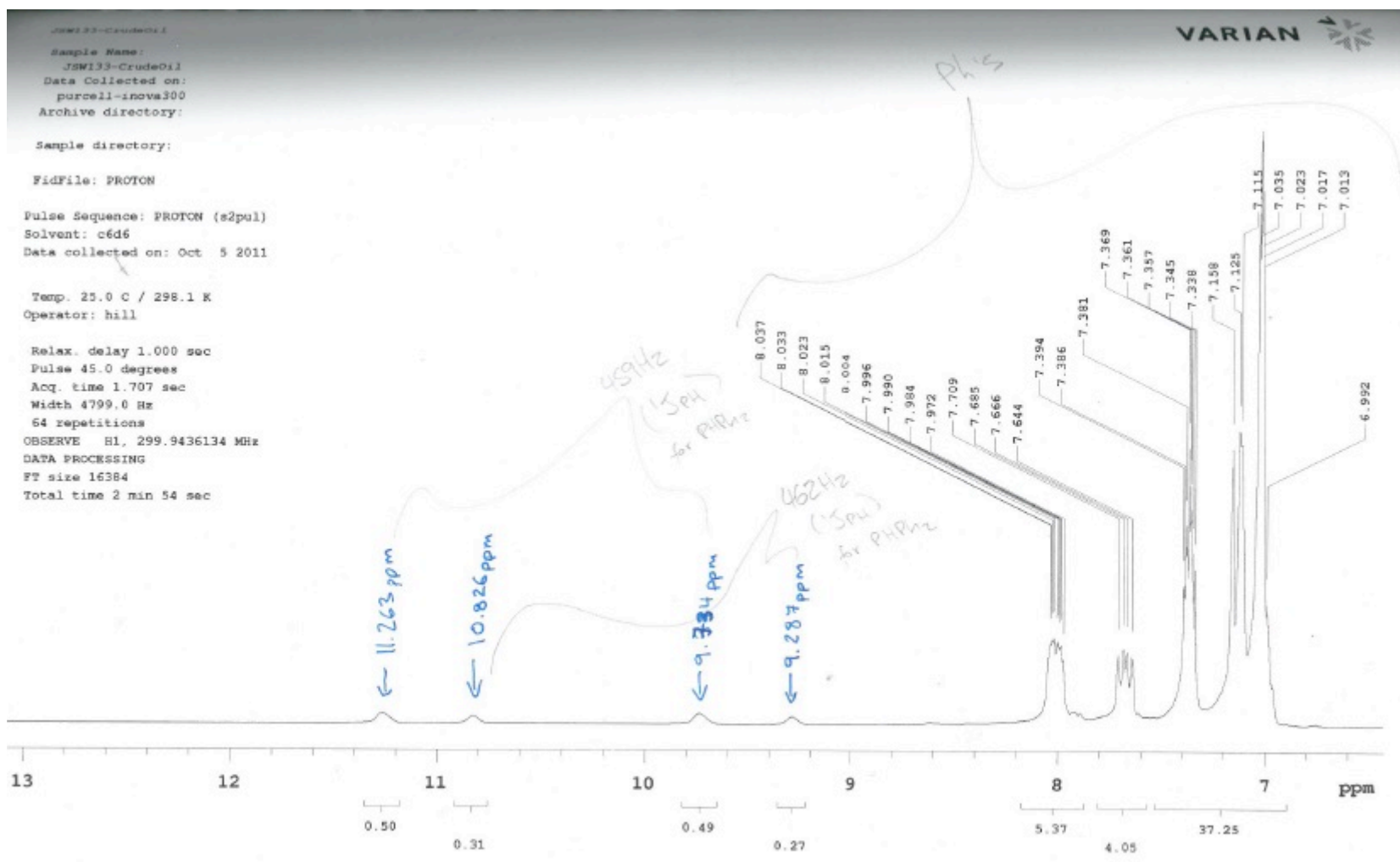
Compound 5 – ¹¹B NMR spectrum



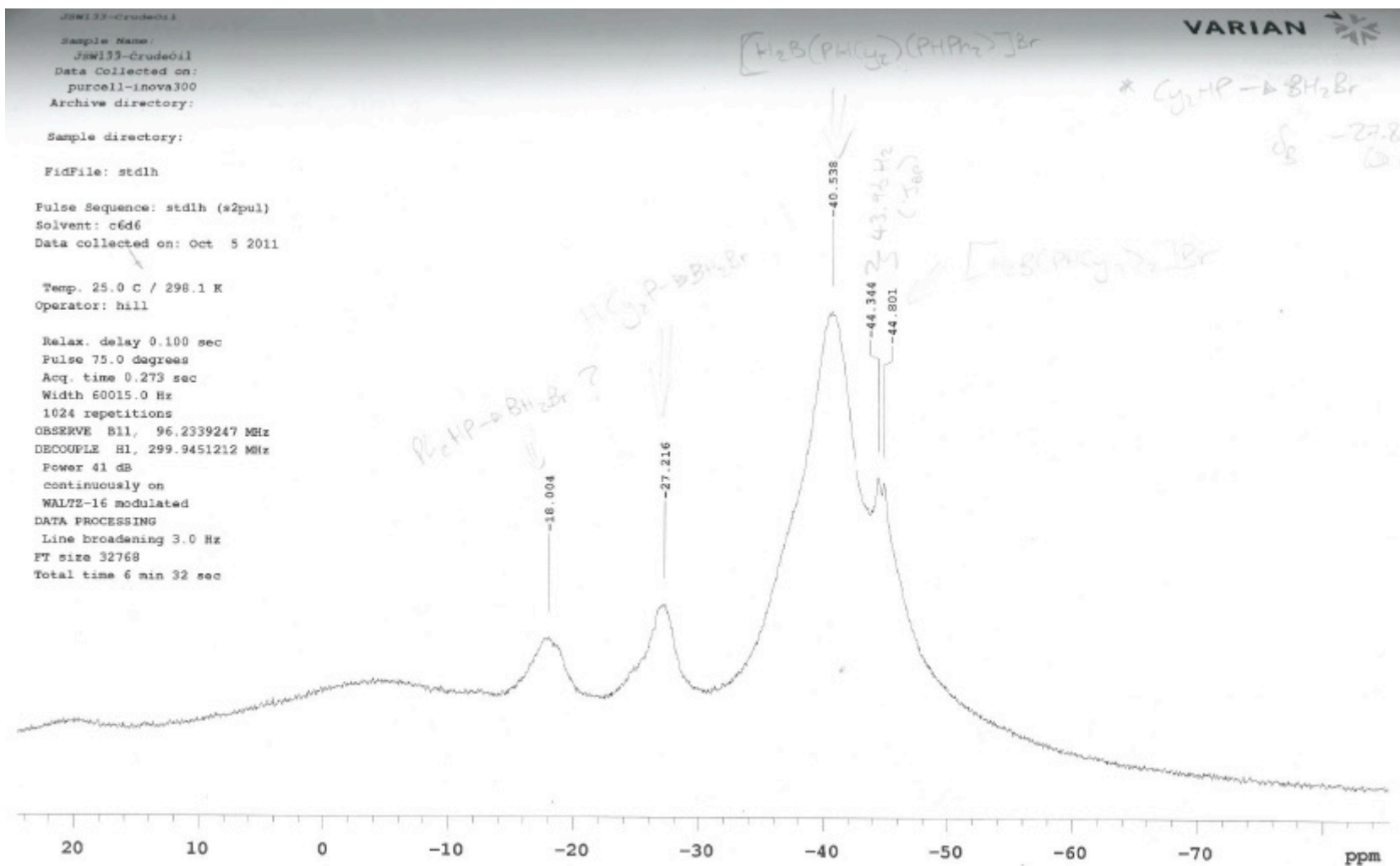
Compound 5 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



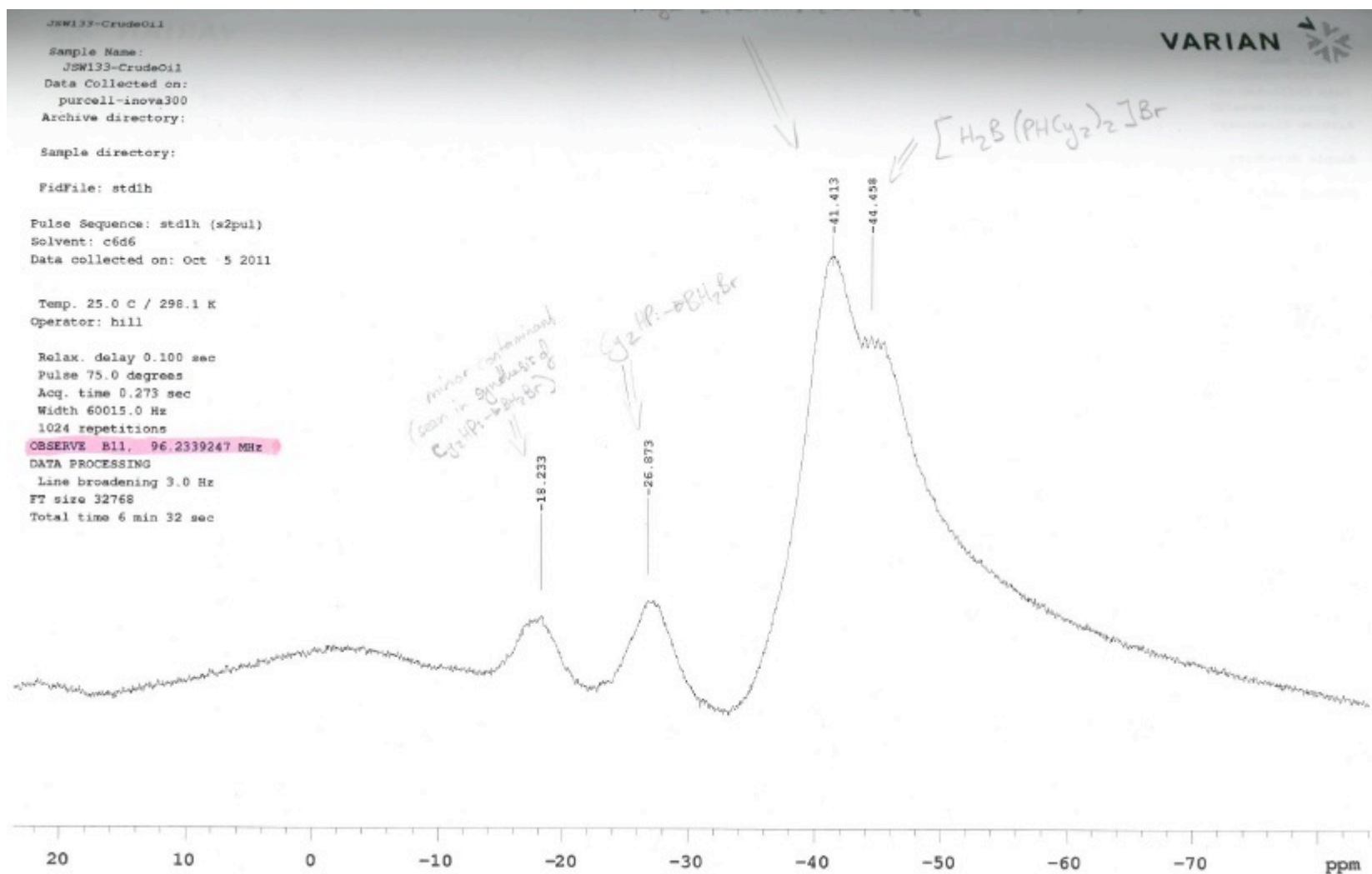
Compound 5 – ³¹P NMR spectrum



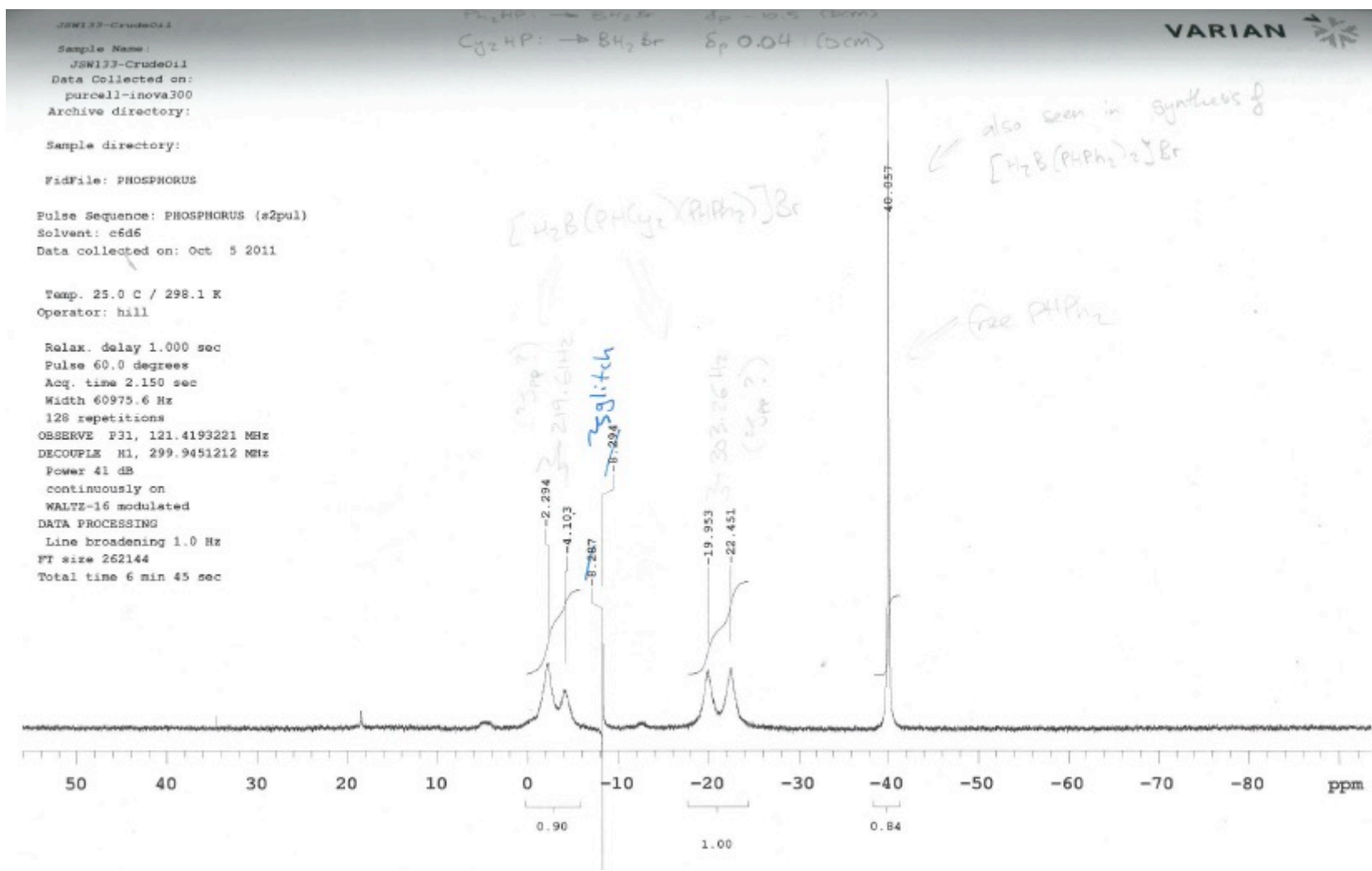
Compound 6 – ¹H NMR spectrum



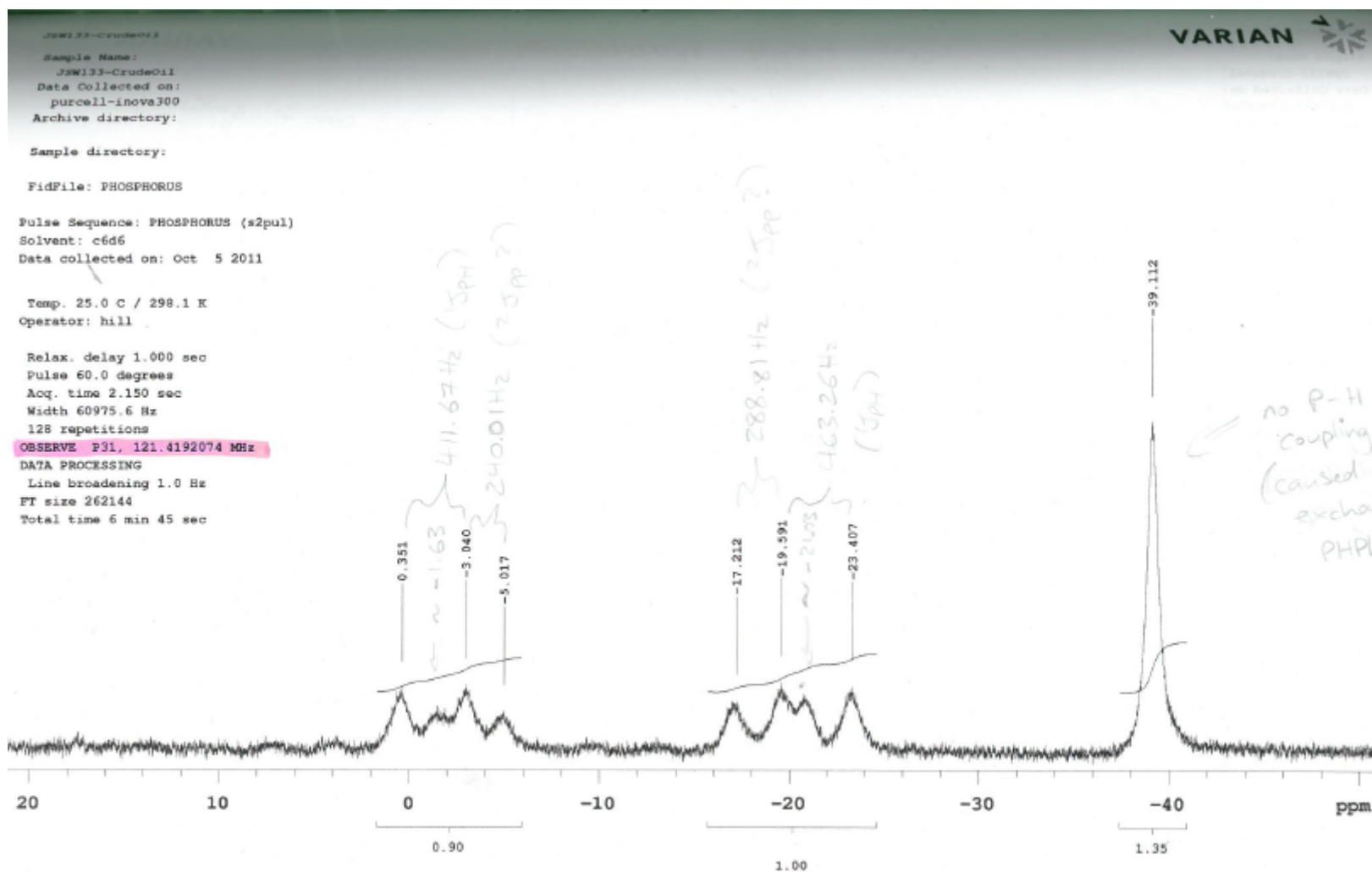
Compound 6 - $^{11}B\{^1H\}$ NMR spectrum



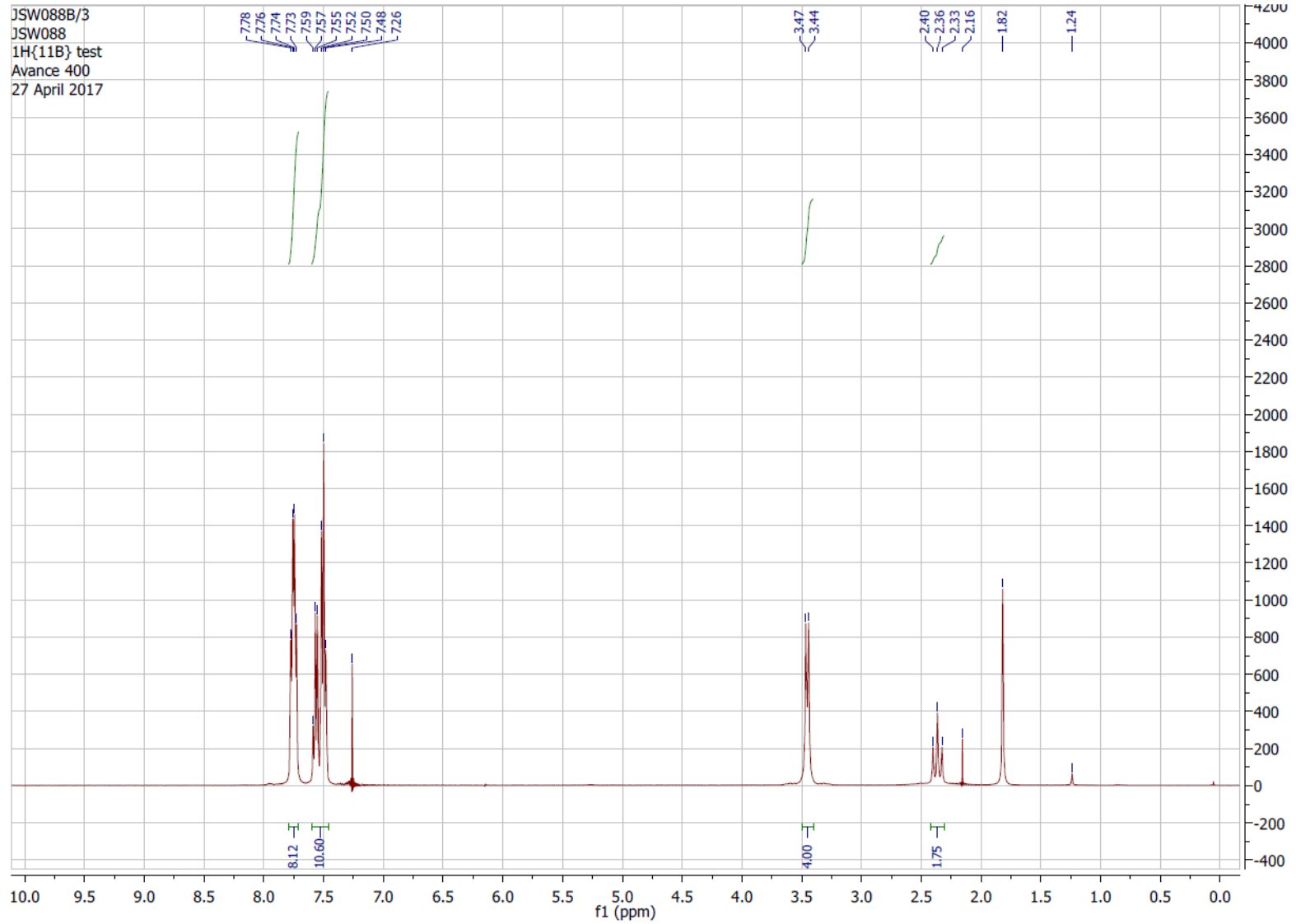
Compound 6 – ^{11}B NMR spectrum



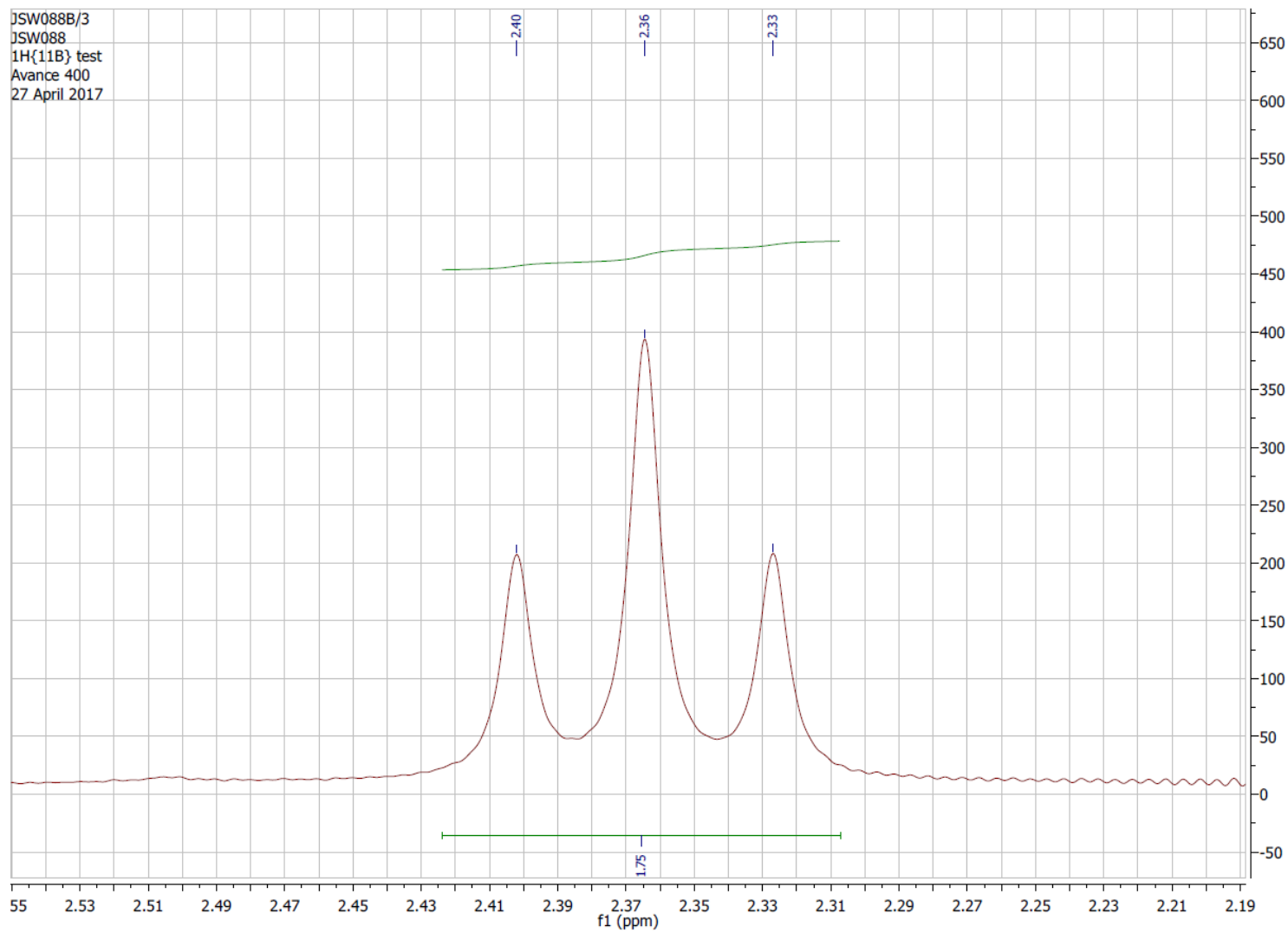
Compound 6 – $^{31}P\{^1H\}$ NMR spectrum



Compound 6 – ³¹P NMR spectrum



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

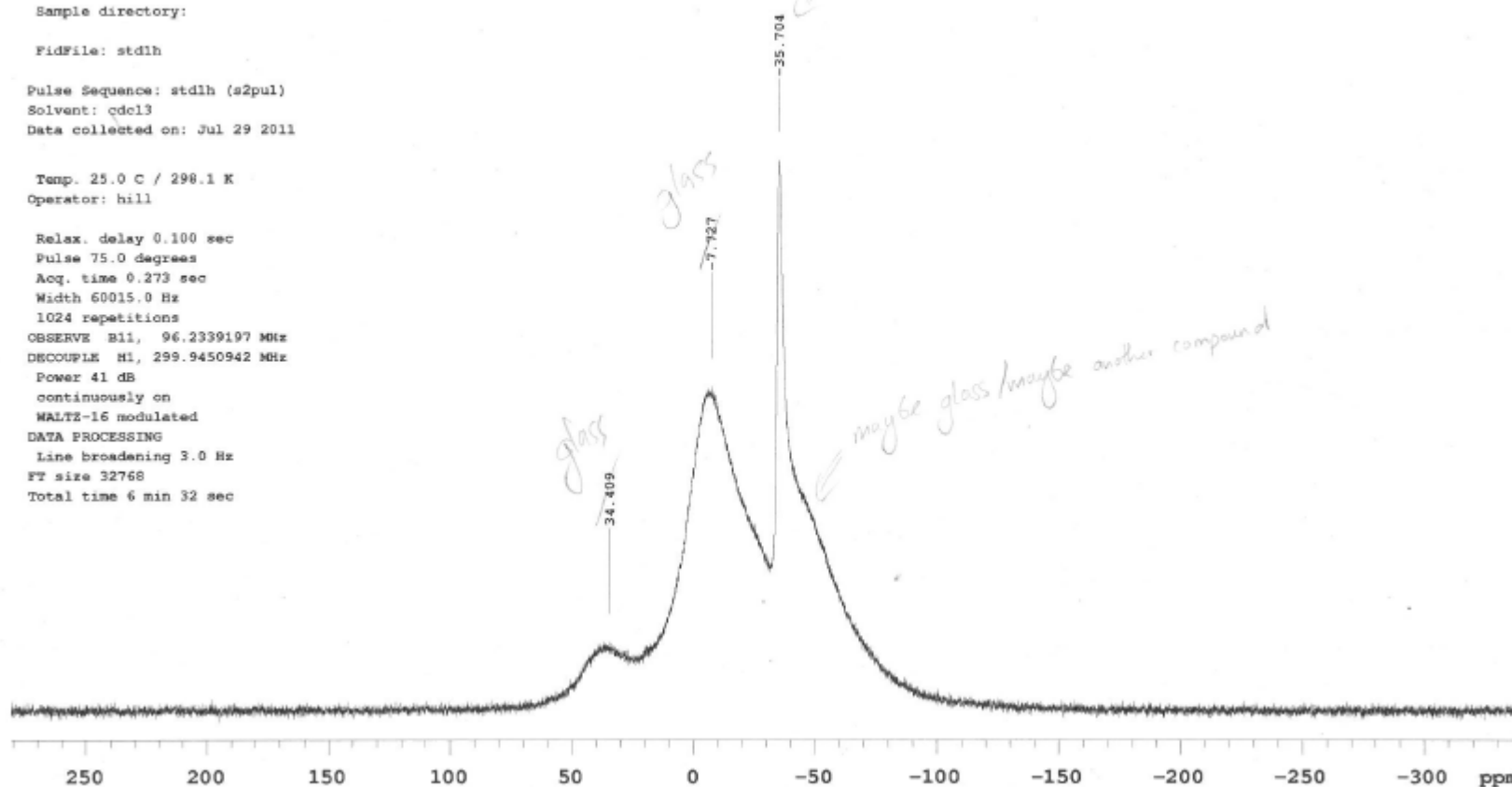


Compound 7 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonances)

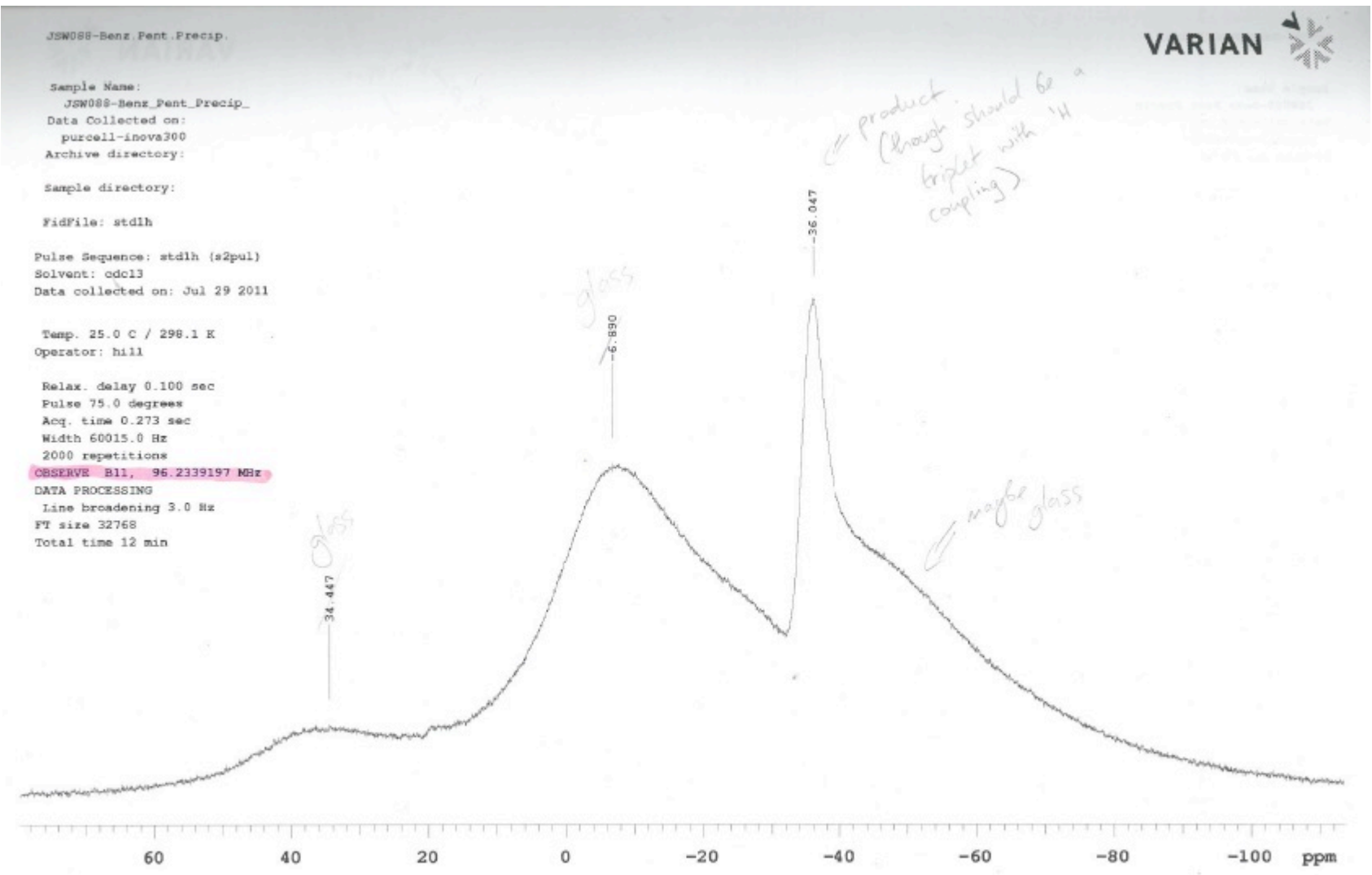
JSW088-Benz_Pent_Precip.

VARIAN 

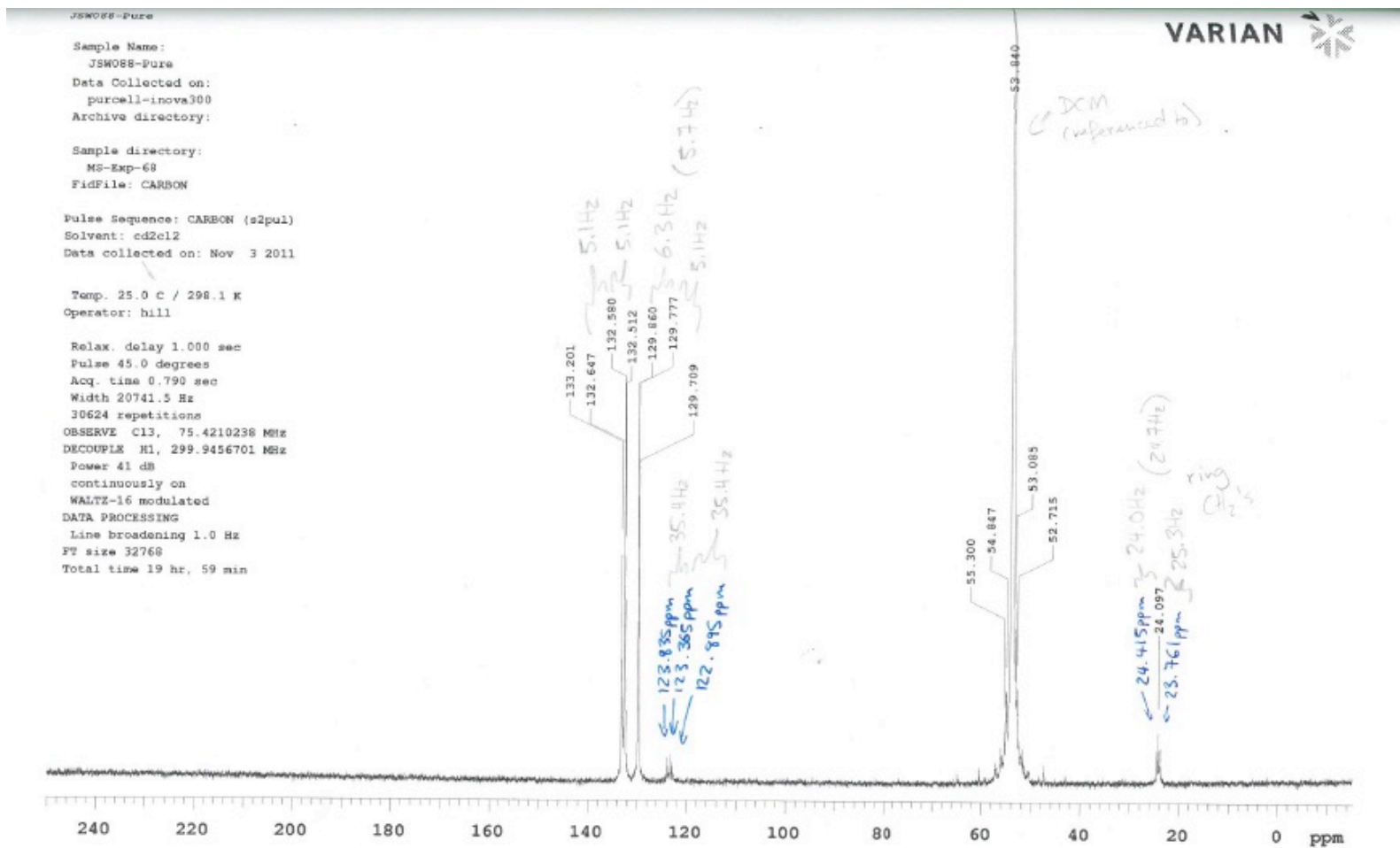
Sample Name:
JSW088-Benz_Pent_Precip_
Data Collected on:
purcell-inova300
Archive directory:
Sample directory:
FidFile: std1h
Pulse Sequence: std1h (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2011
Temp. 25.0 C / 298.1 K
Operator: hill
Relax. delay 0.100 sec
Pulse 75.0 degrees
Acq. time 0.273 sec
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



Compound 7 – ^{11}B 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:

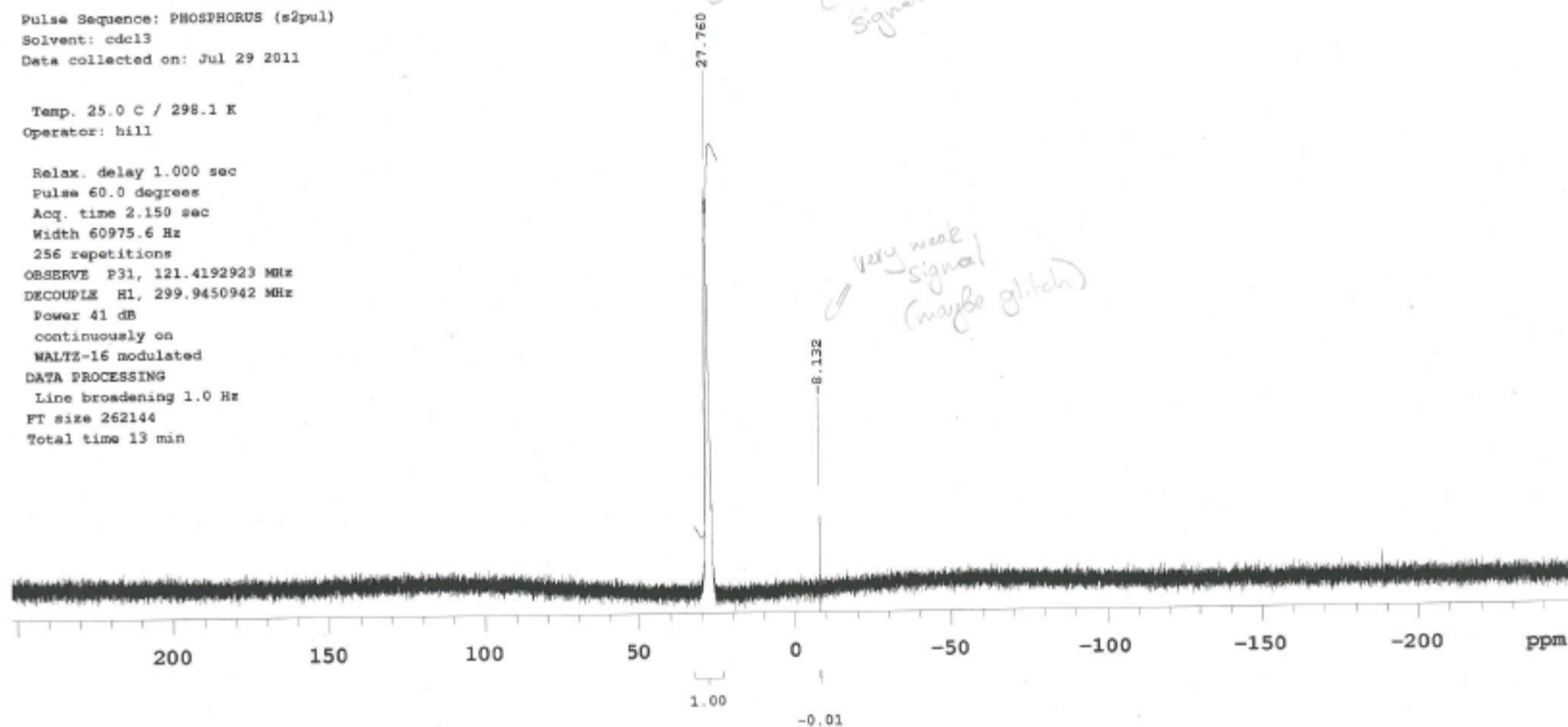
Sample directory:

Fidfile: PHOSPHORUS

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

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
256 repetitions
OBSERVE P31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 262144
Total time 13 min



Compound 7 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum

JSM179-Crystals



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

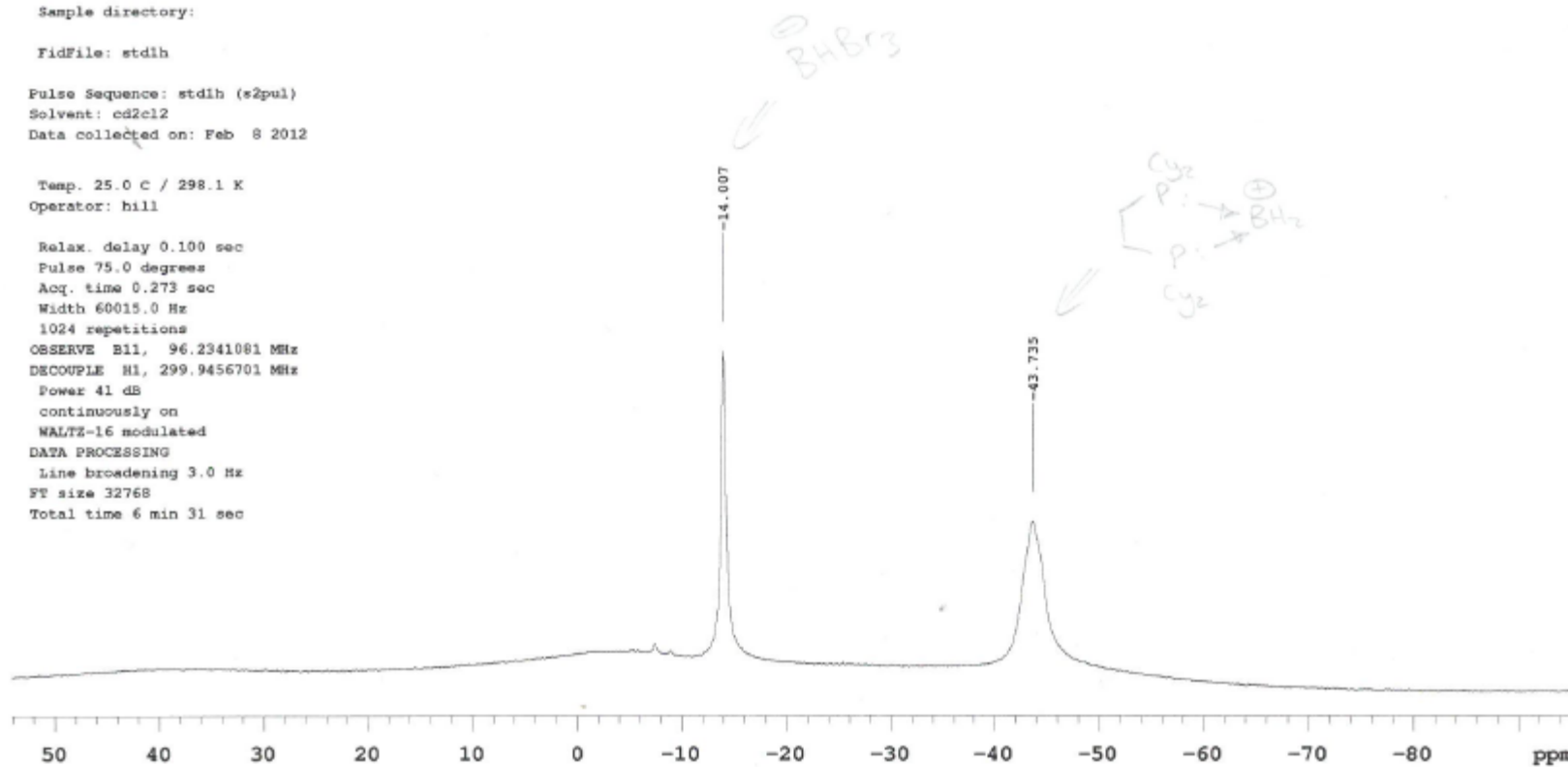
Sample directory:

FidFile: std1h

Pulse Sequence: std1h (s2pul)
Solvent: cd2cl2
Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K
Operator: bill

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
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



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

JSW179-Crystals

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

Sample directory:

FidFile: std1h

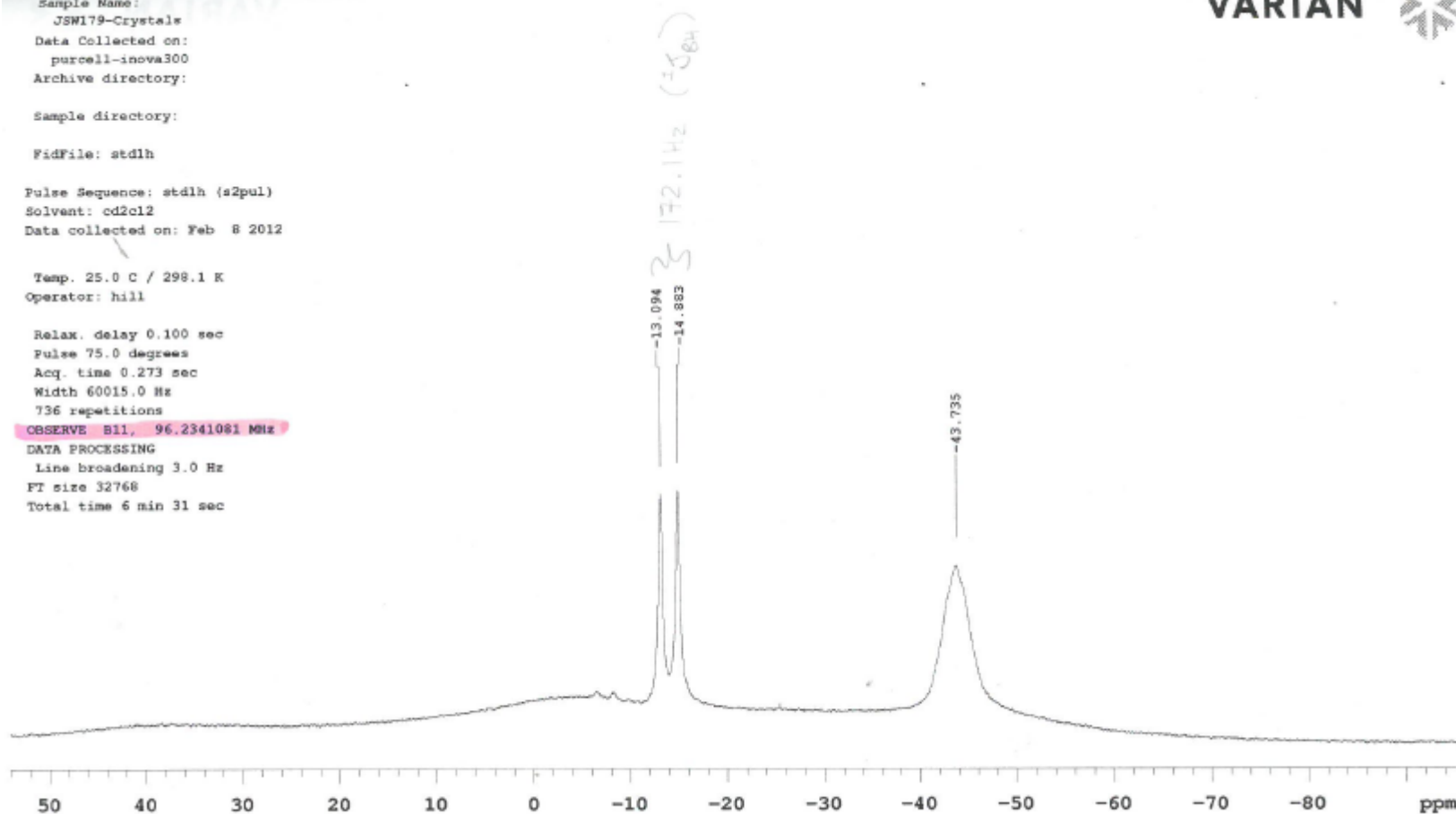
Pulse Sequence: std1h (s2pul)
Solvent: cd2cl2
Data collected on: Feb 8 2012

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



Compound 8 – ^{11}B NMR spectrum

JSW179-Crystals

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

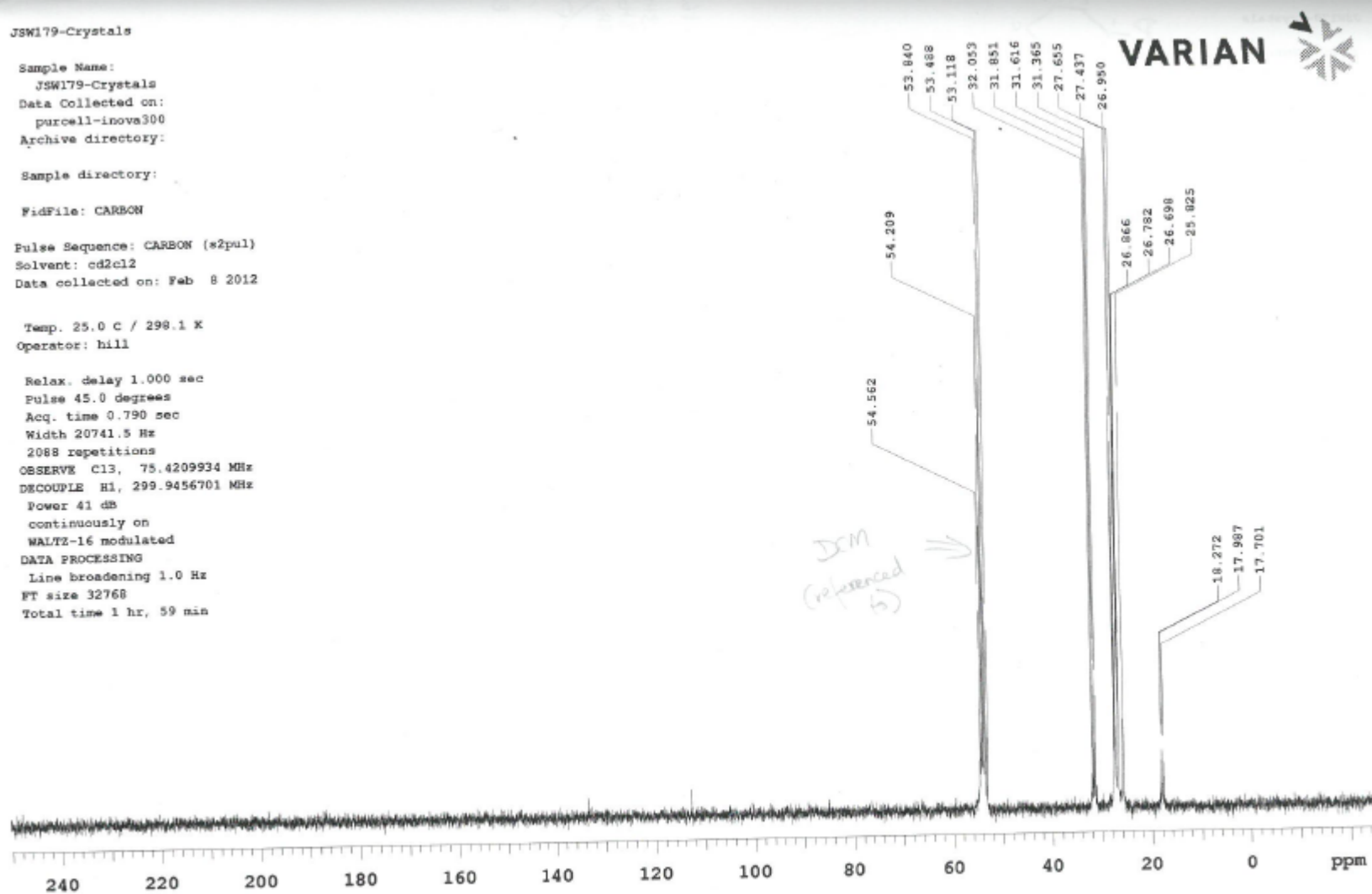
Sample directory:

Fidfile: CARBON

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

Temp. 25.0 C / 298.1 K
Operator: hill

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
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 1 hr, 59 min



Compound 8 - $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

JSM179-Crystals



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

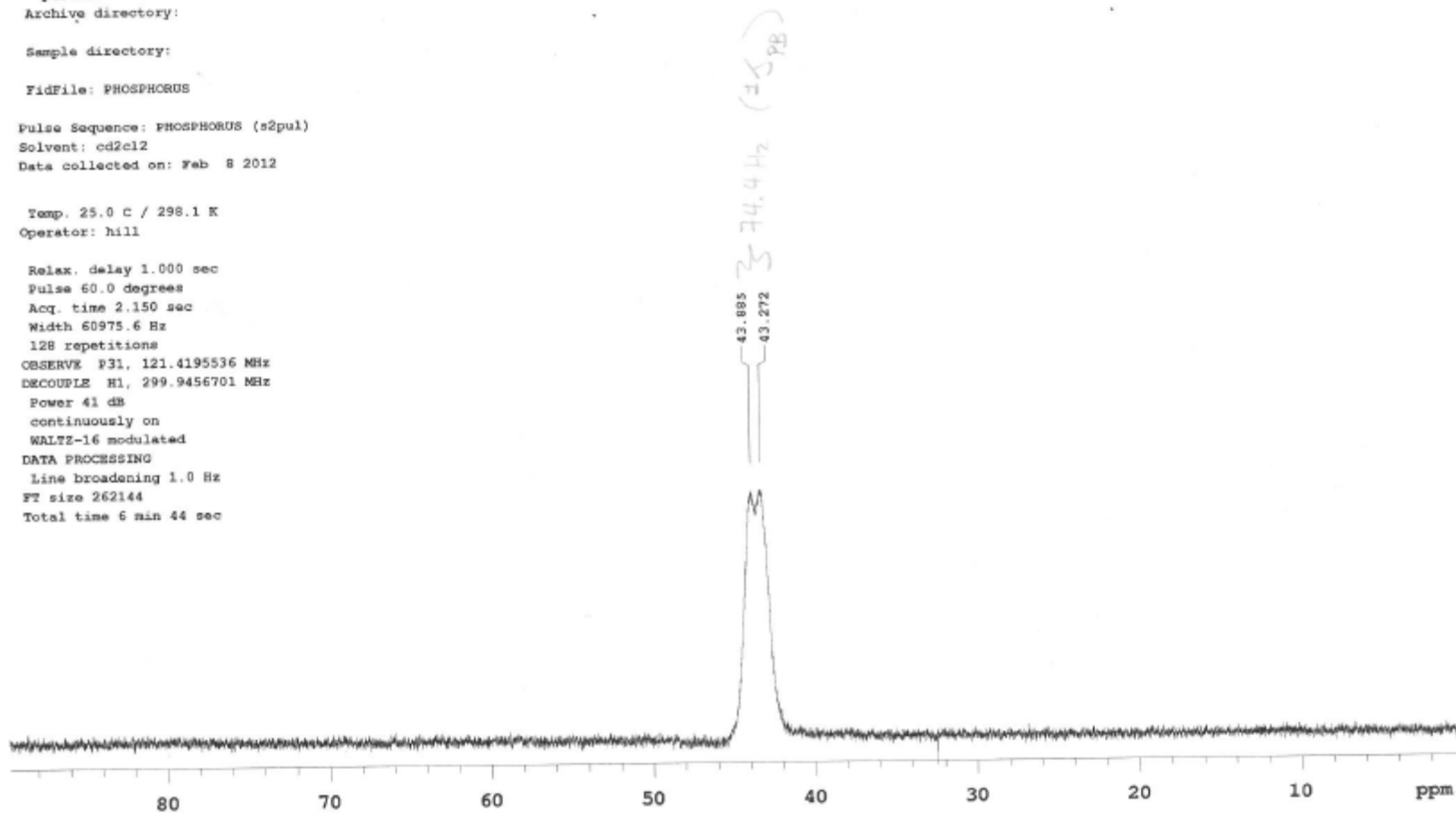
Sample directory:

FidFile: PHOSPHORUS

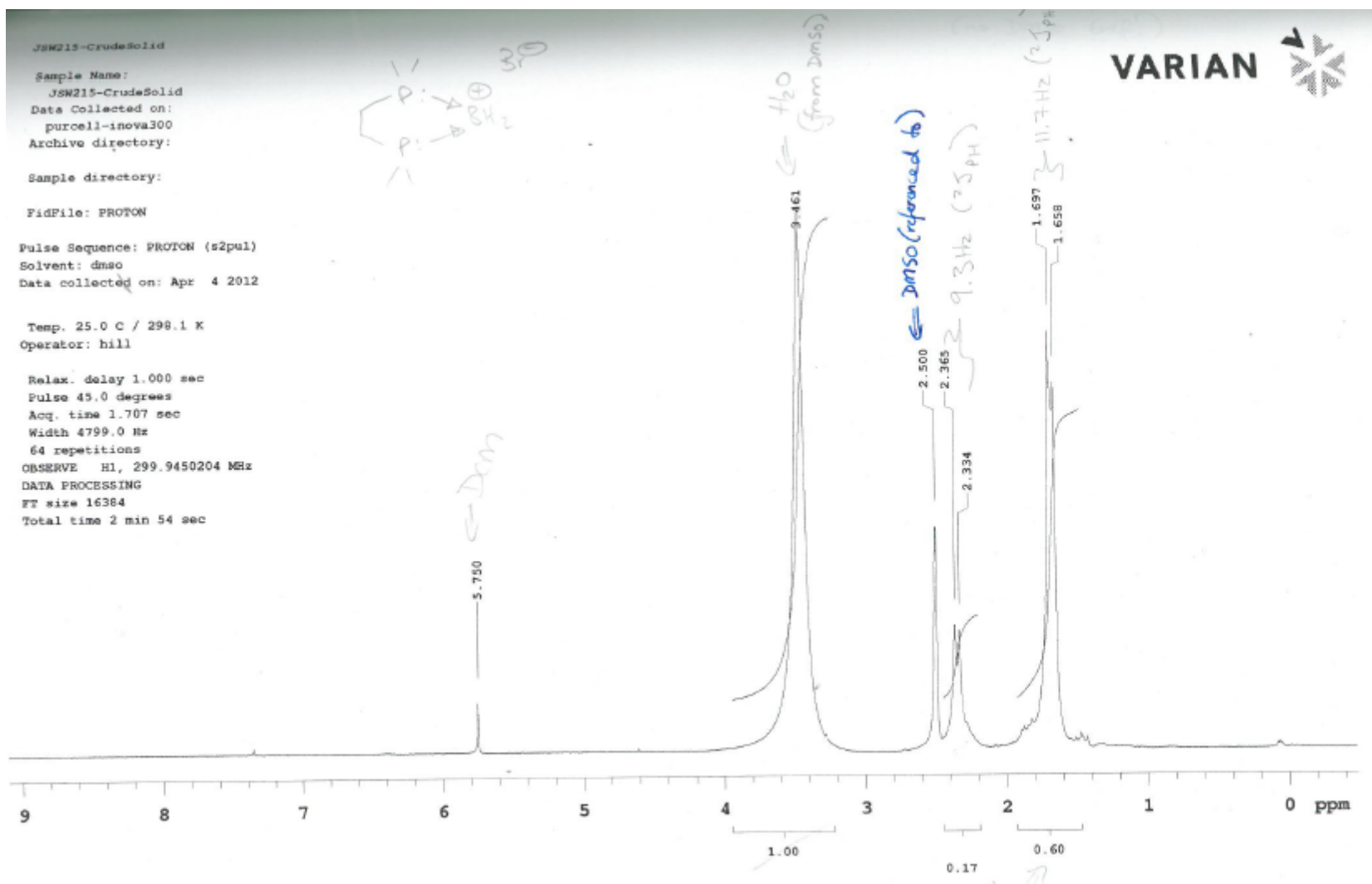
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cd2cl2
Data collected on: Feb 8 2012

Temp. 25.0 C / 298.1 K
Operator: hill

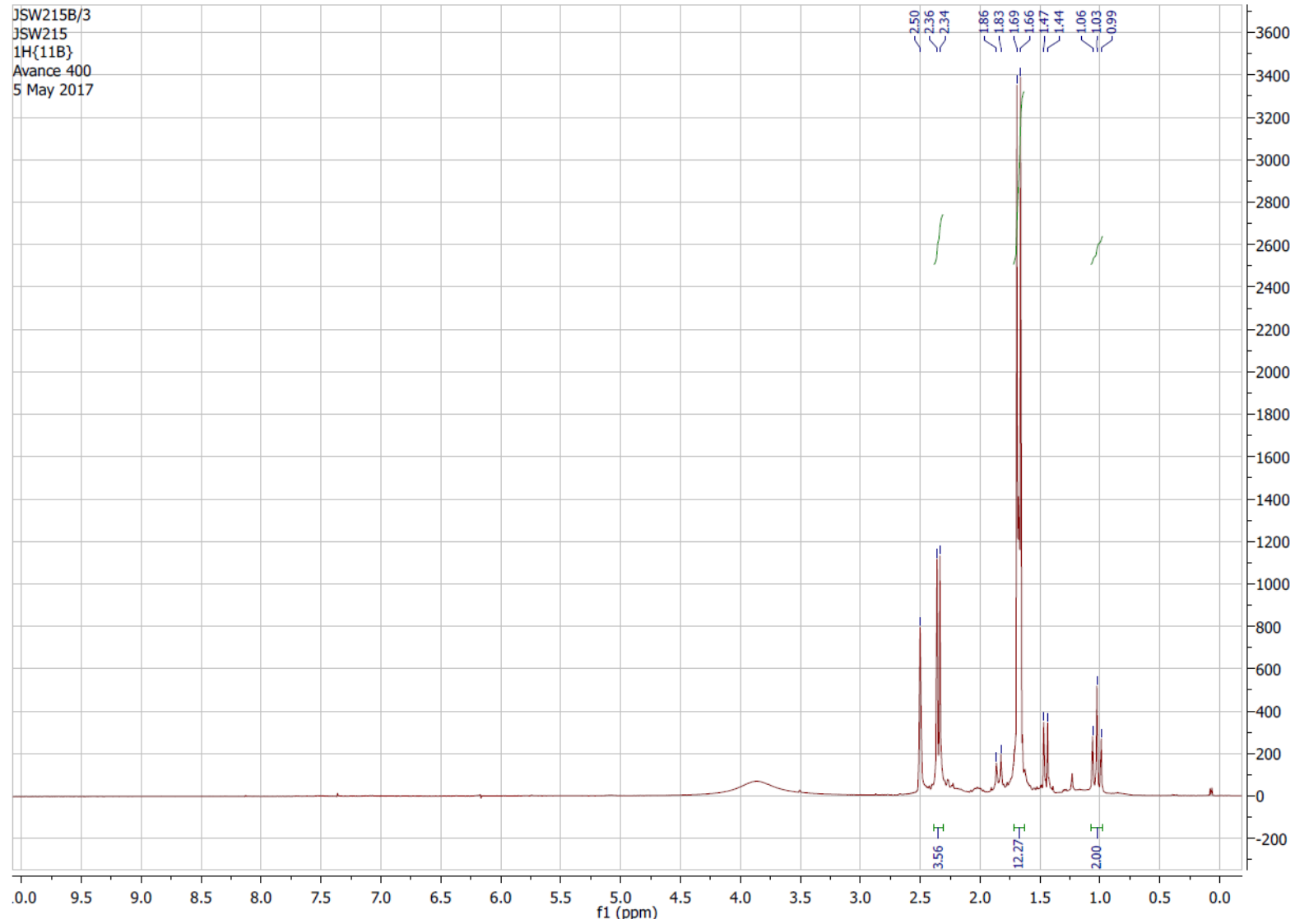
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 2.150 sec
Width 60975.6 Hz
128 repetitions
OBSERVE P31, 121.4195536 MHz
DECOUPLE H1, 299.9456701 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 262144
Total time 6 min 44 sec



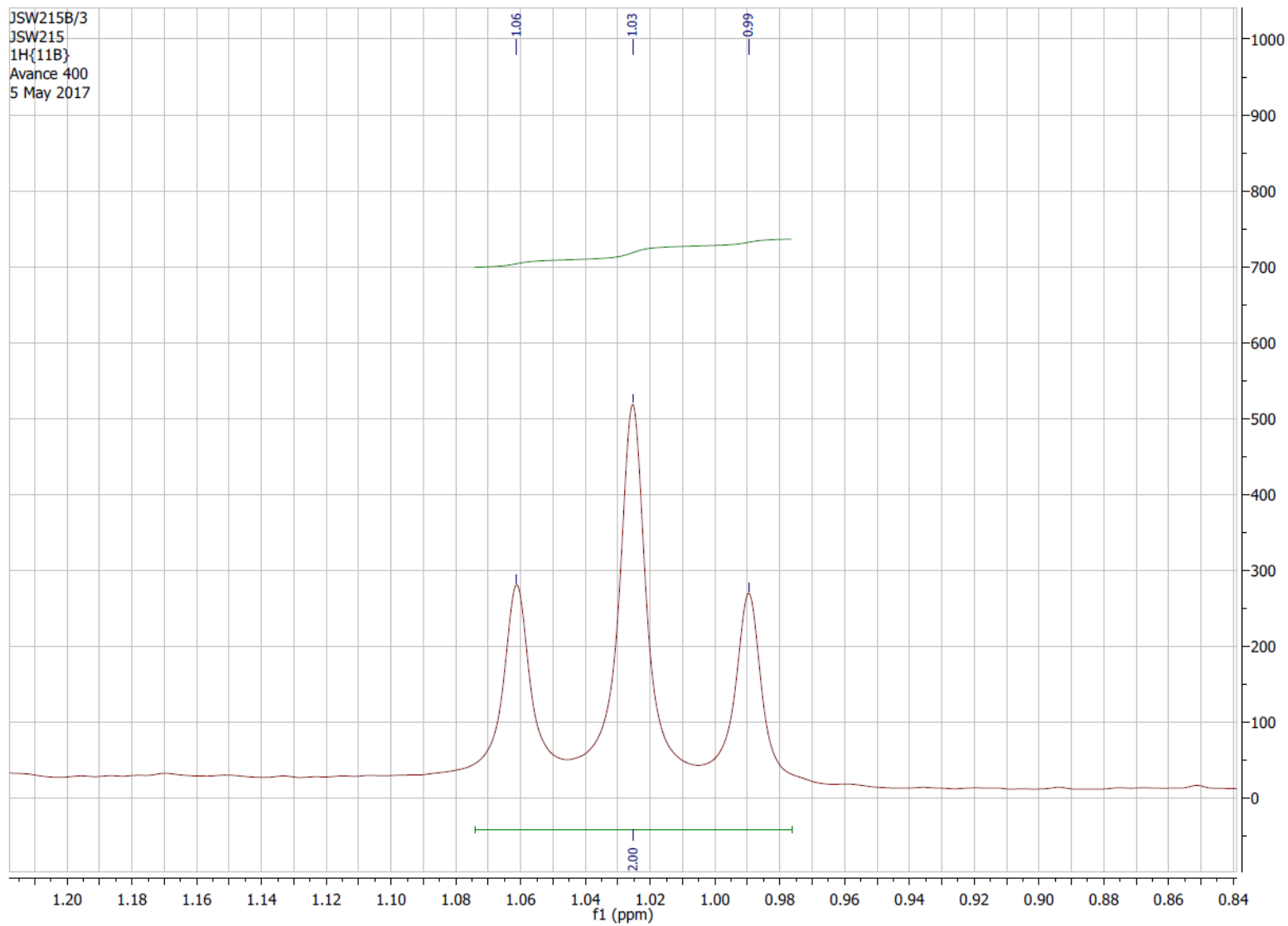
Compound 8 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



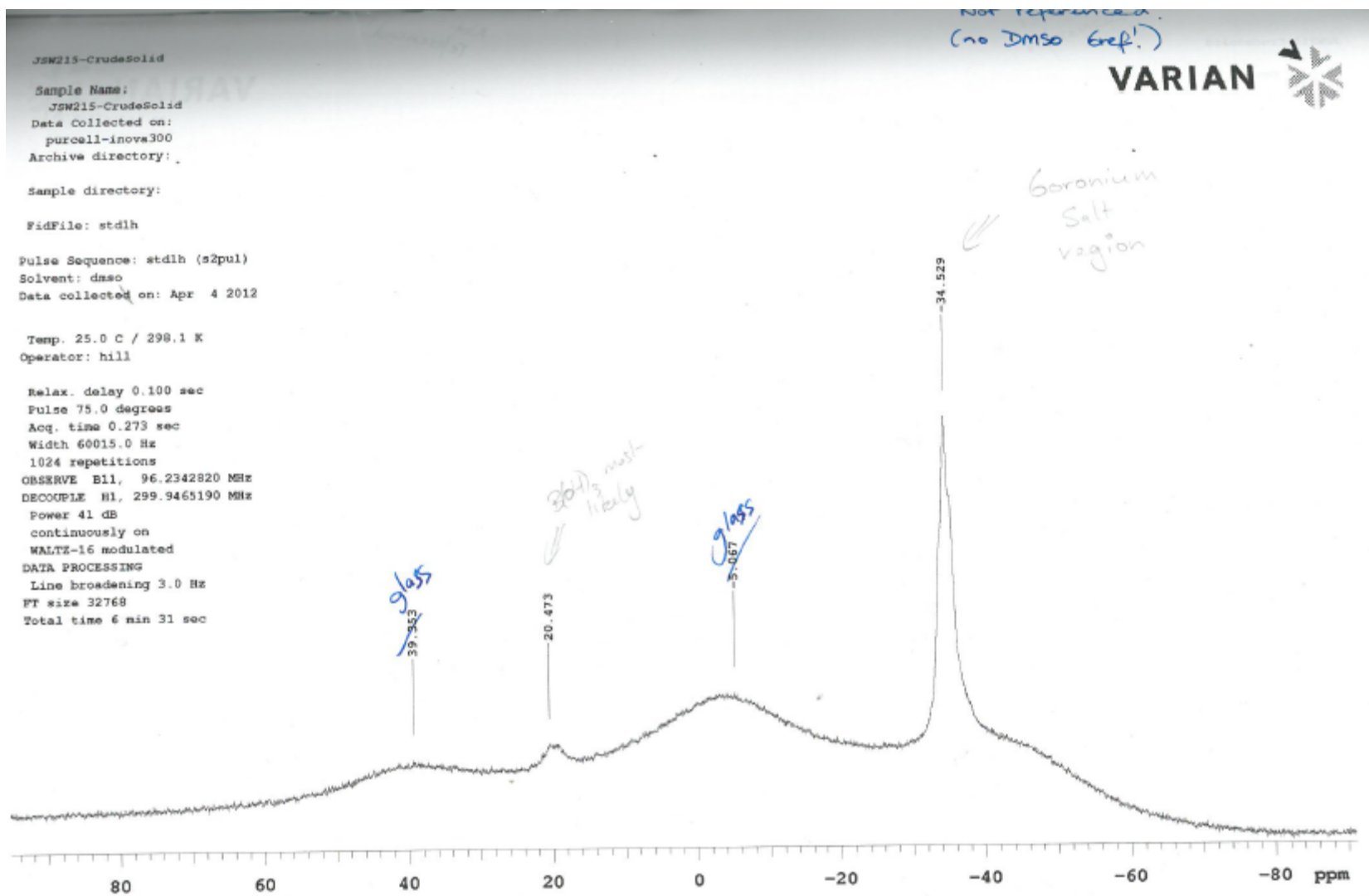
Compound 9 – ¹H NMR spectrum



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

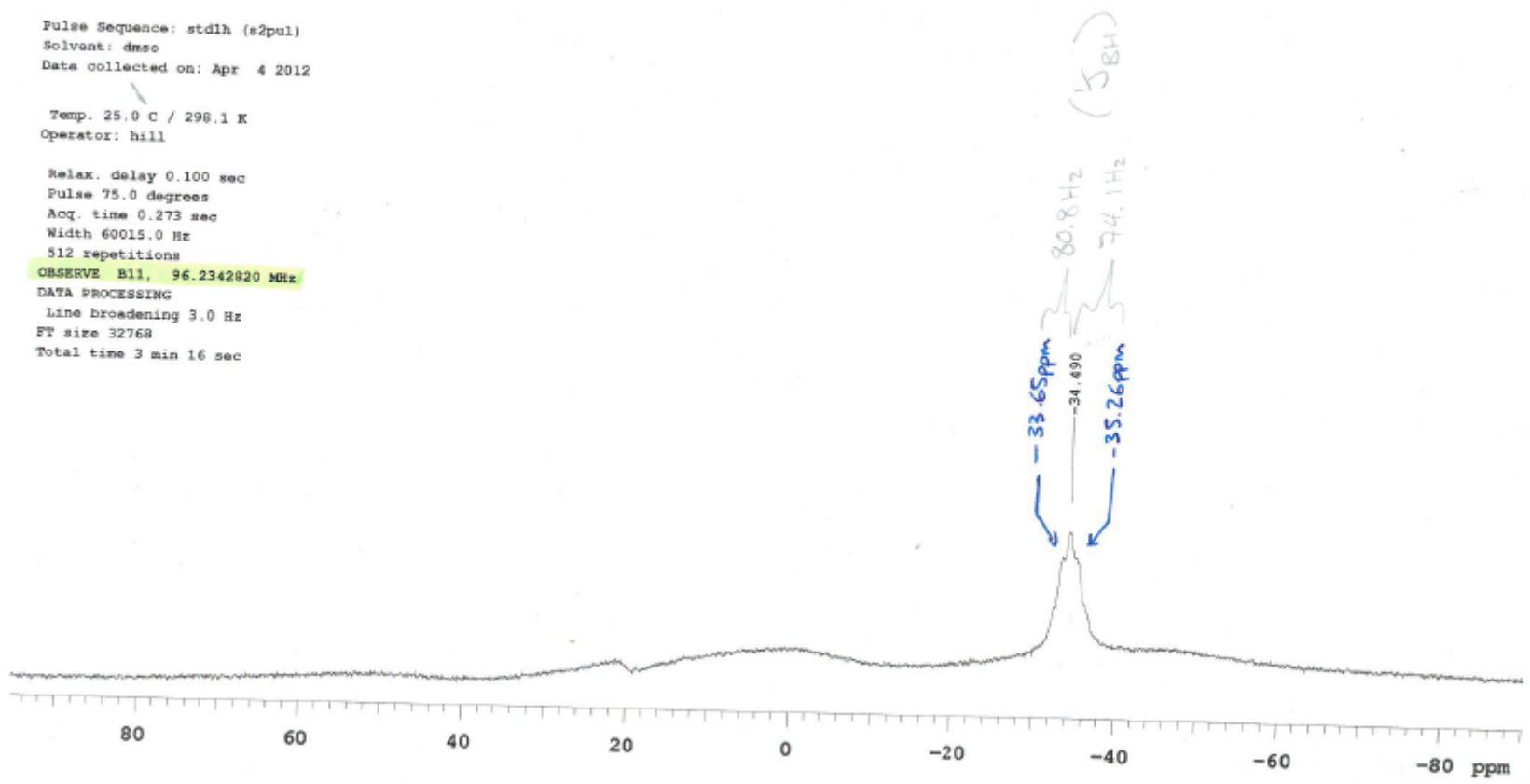


Compound 9 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)

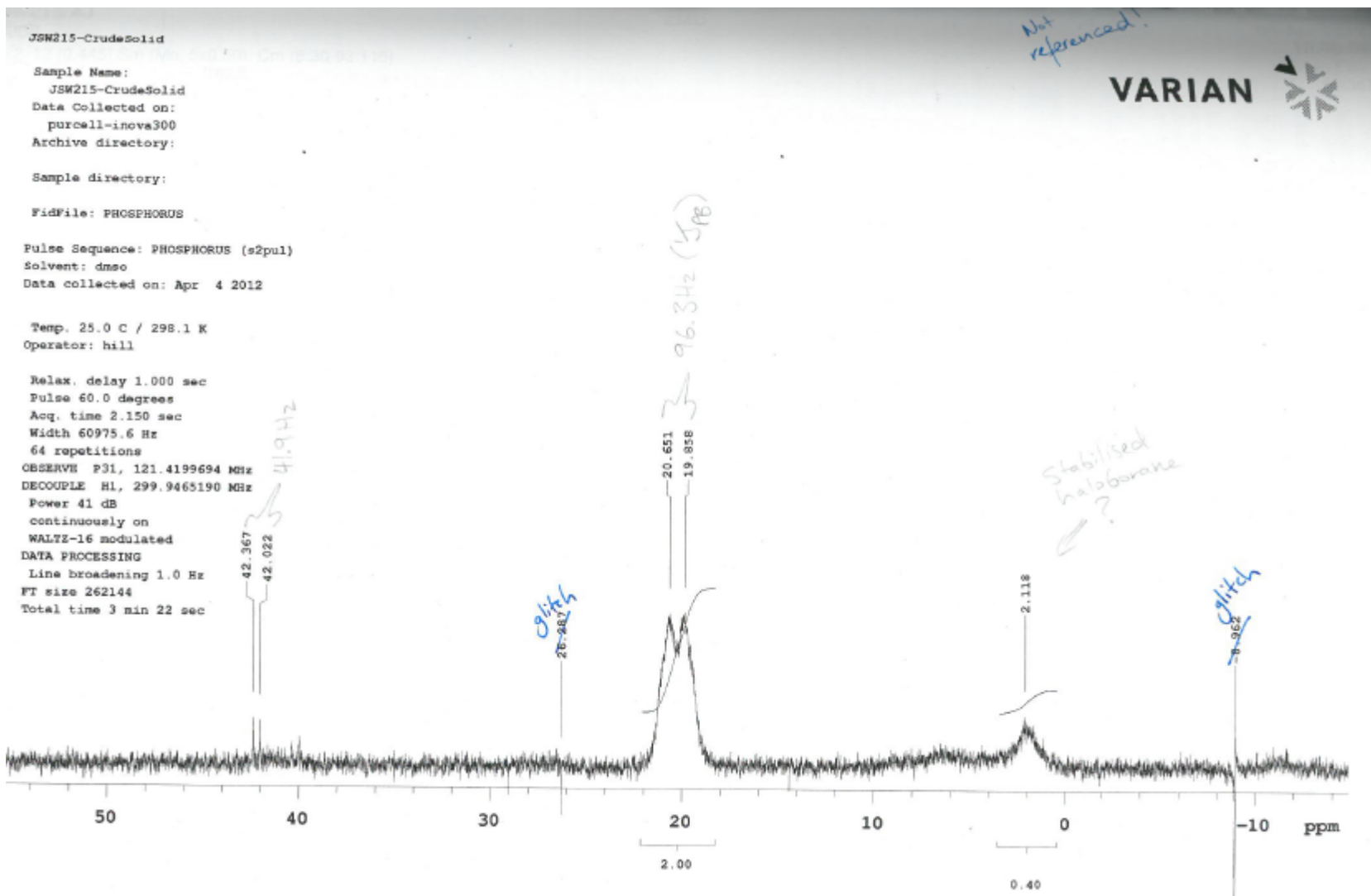


Compound 9 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum

Sample Name: JSM215-CrudeSolid
Data Collected on: purcell-inova300
Archive directory:
Sample directory:
FidFile: std1h
Pulse Sequence: std1h (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



Compound 9 – ¹¹B NMR spectrum



Compound 9 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum

JSM322-Precipitate

Sample Name:
JSM322-Precipitate
Data Collected on:
purcell-ineva300
Archive directory:

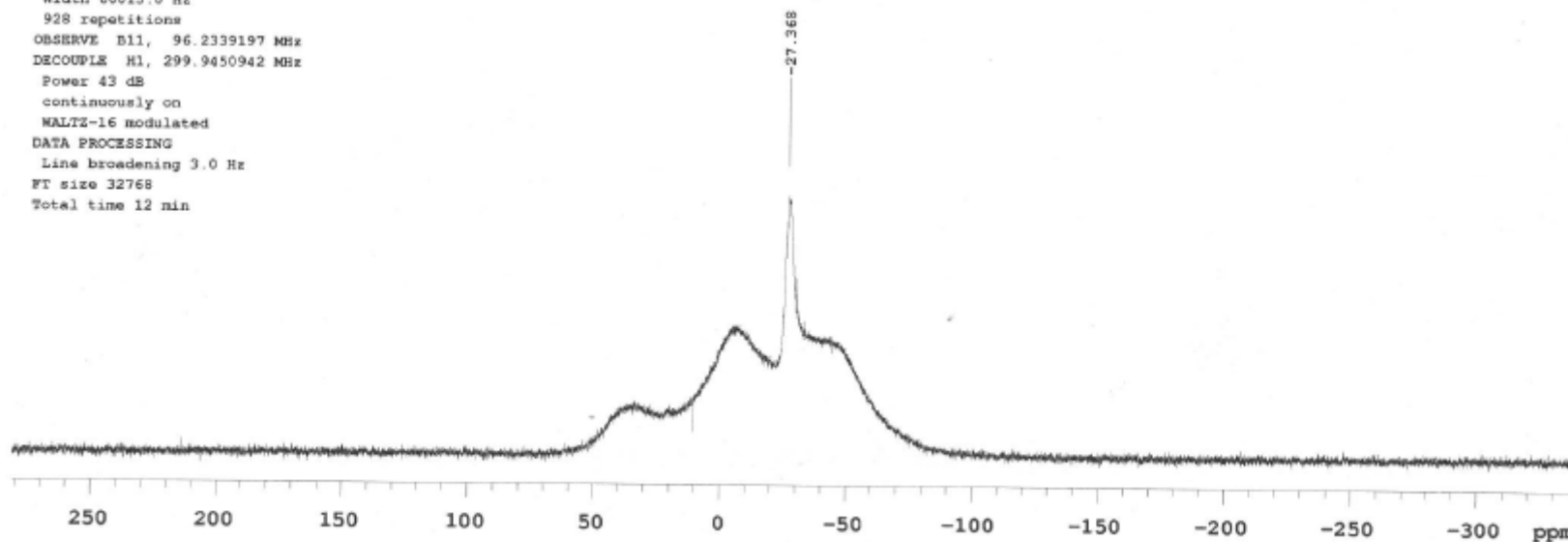
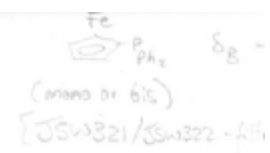
Sample directory:

FidFile: stdlh

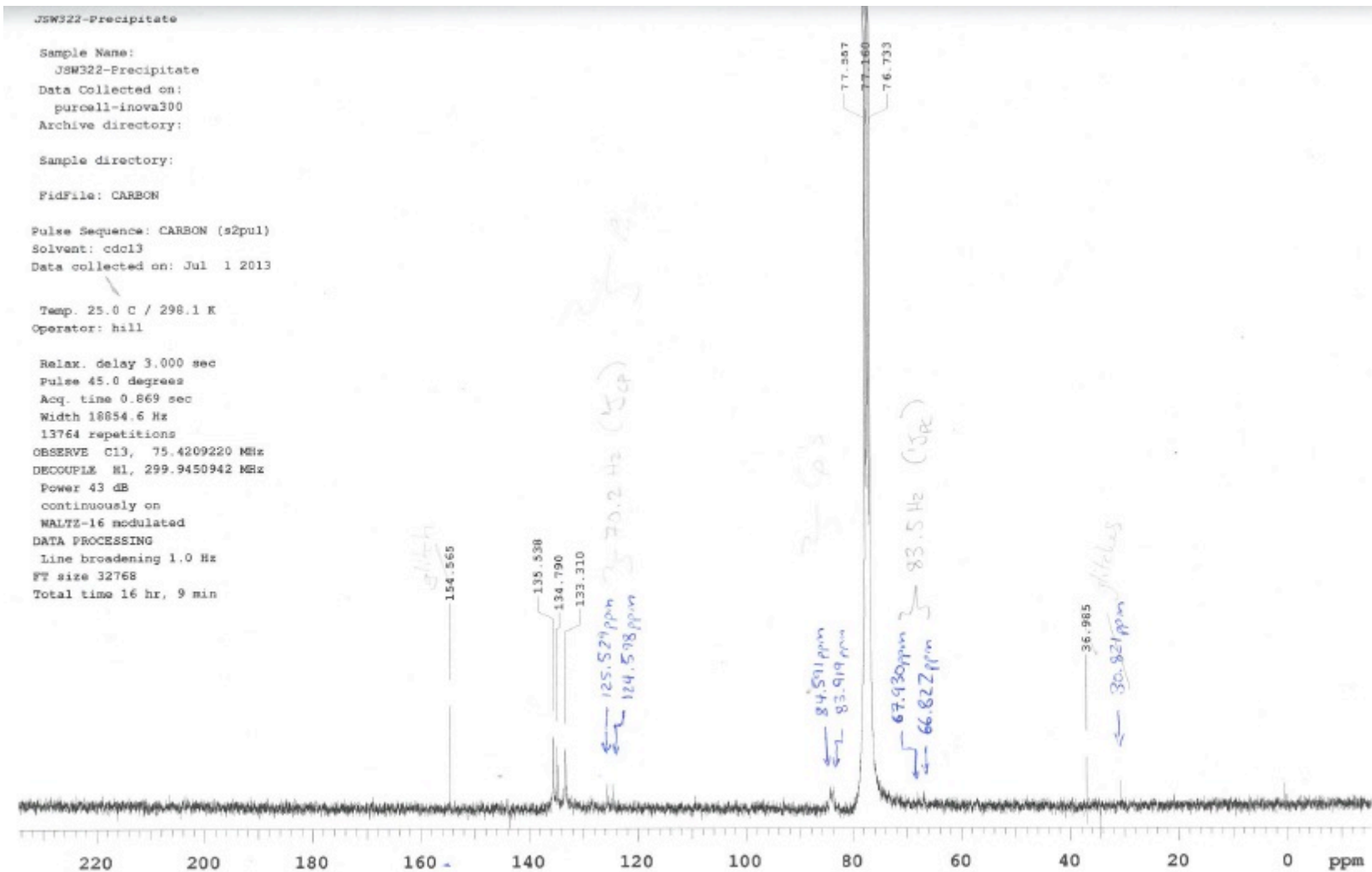
Pulse Sequence: stdlh (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2013

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 B11, 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



Compound 10 - ¹¹B{¹H} NMR spectrum



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

JSW322-Precipitate

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

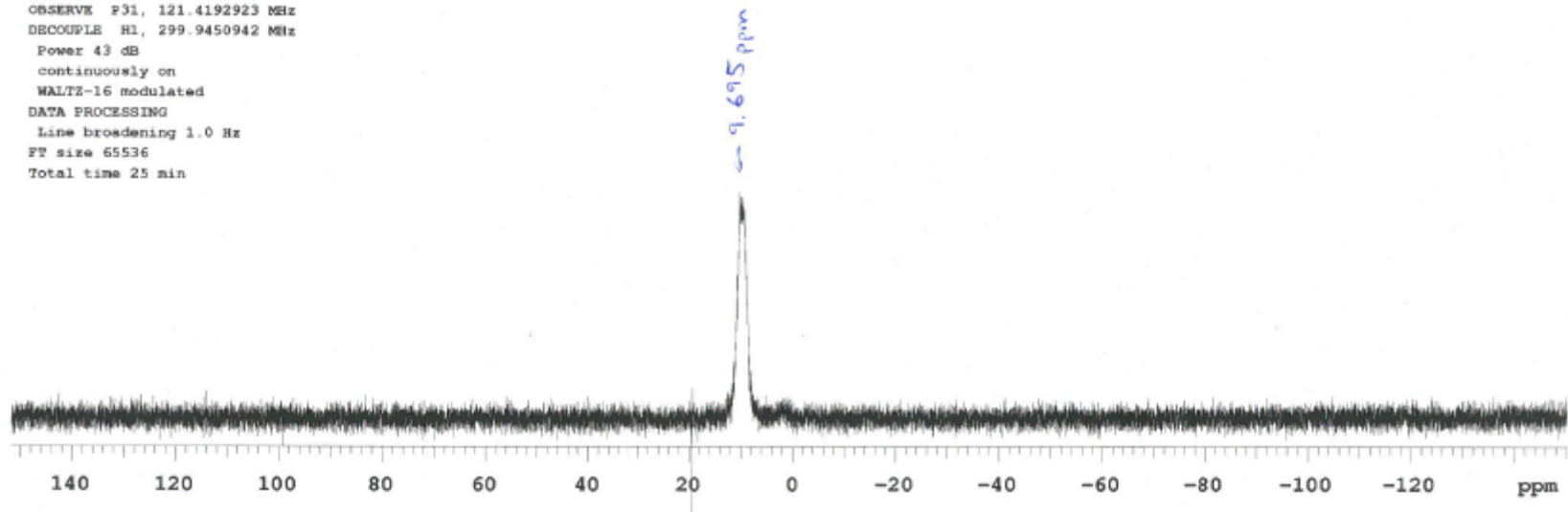
Sample directory:

FidFile: PHOSPHORUS

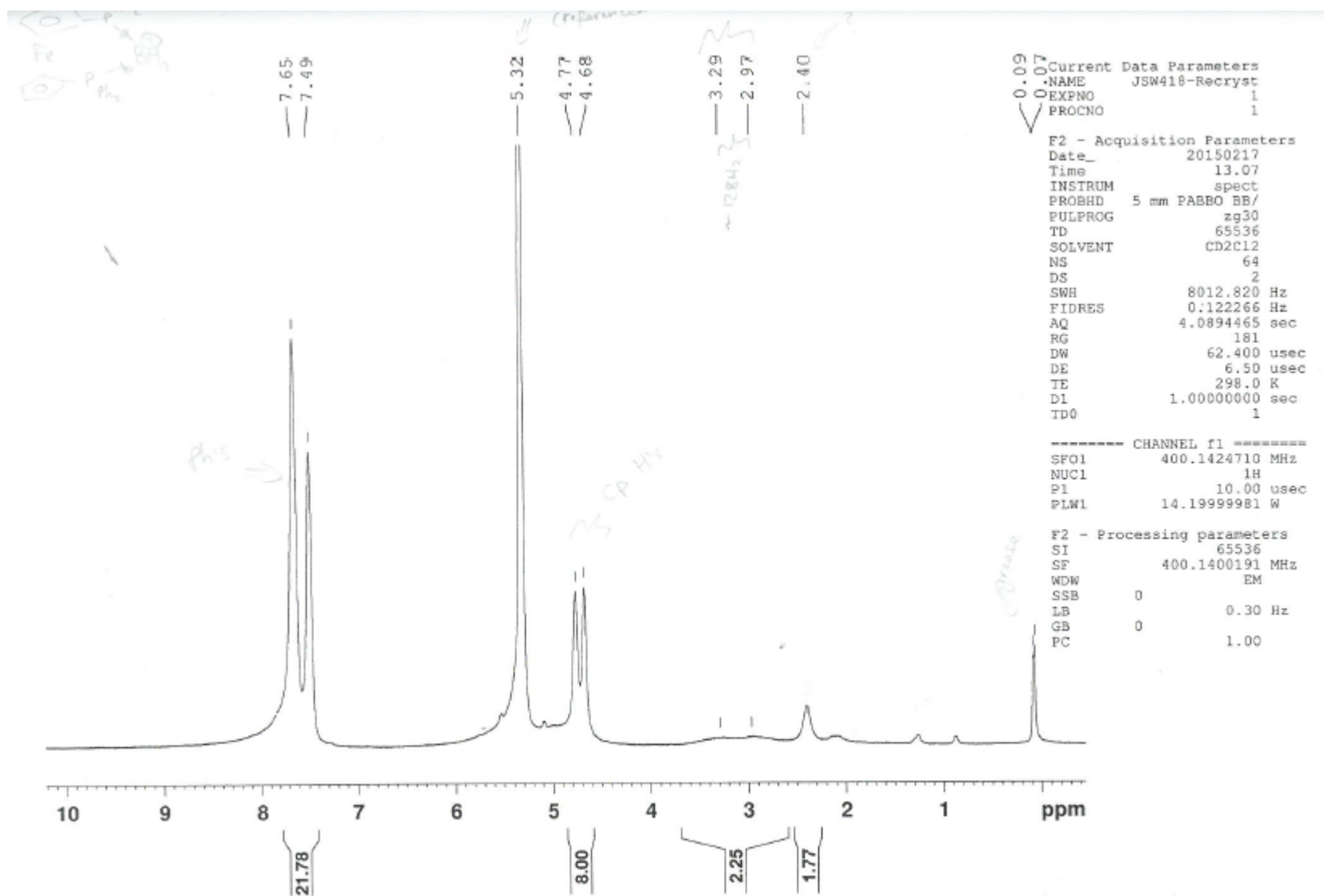
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdcl3
Data collected on: Jul 1 2013

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
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



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



Compound 11 - ^1H NMR spectrum

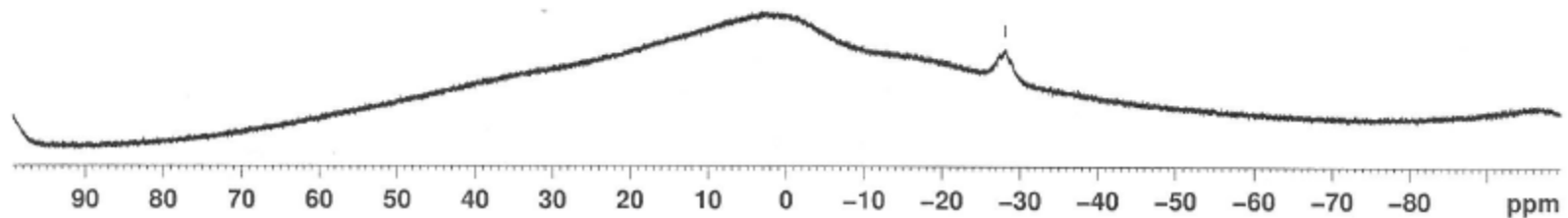
---28.07

Current Data Parameters
NAME JSW418-Recryst
EXPNO 3
PROCNO 1

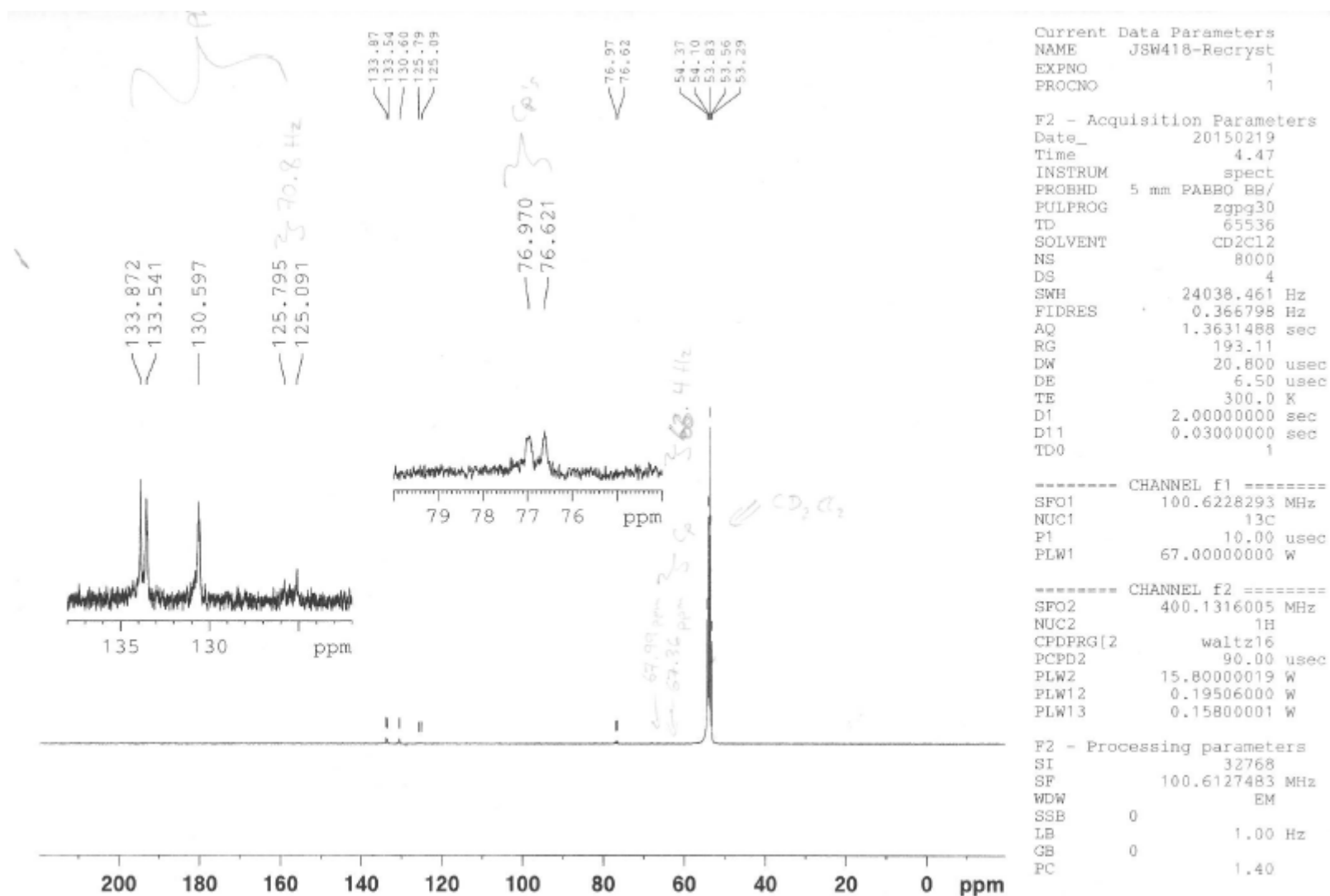
F2 - Acquisition Parameters
Date_ 20150217
Time 13.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg
TD 65536
SOLVENT CD2Cl2
NS 200
DS 4
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845056 sec
RG 203
DW 19.600 usec
DE 6.50 usec
TE 298.0 K
D1 0.20000000 sec
TD0 1

==== CHANNEL f1 =====
SF01 128.3808136 MHz
NUC1 11B
P1 9.00 usec
PLW1 63.97299957 W

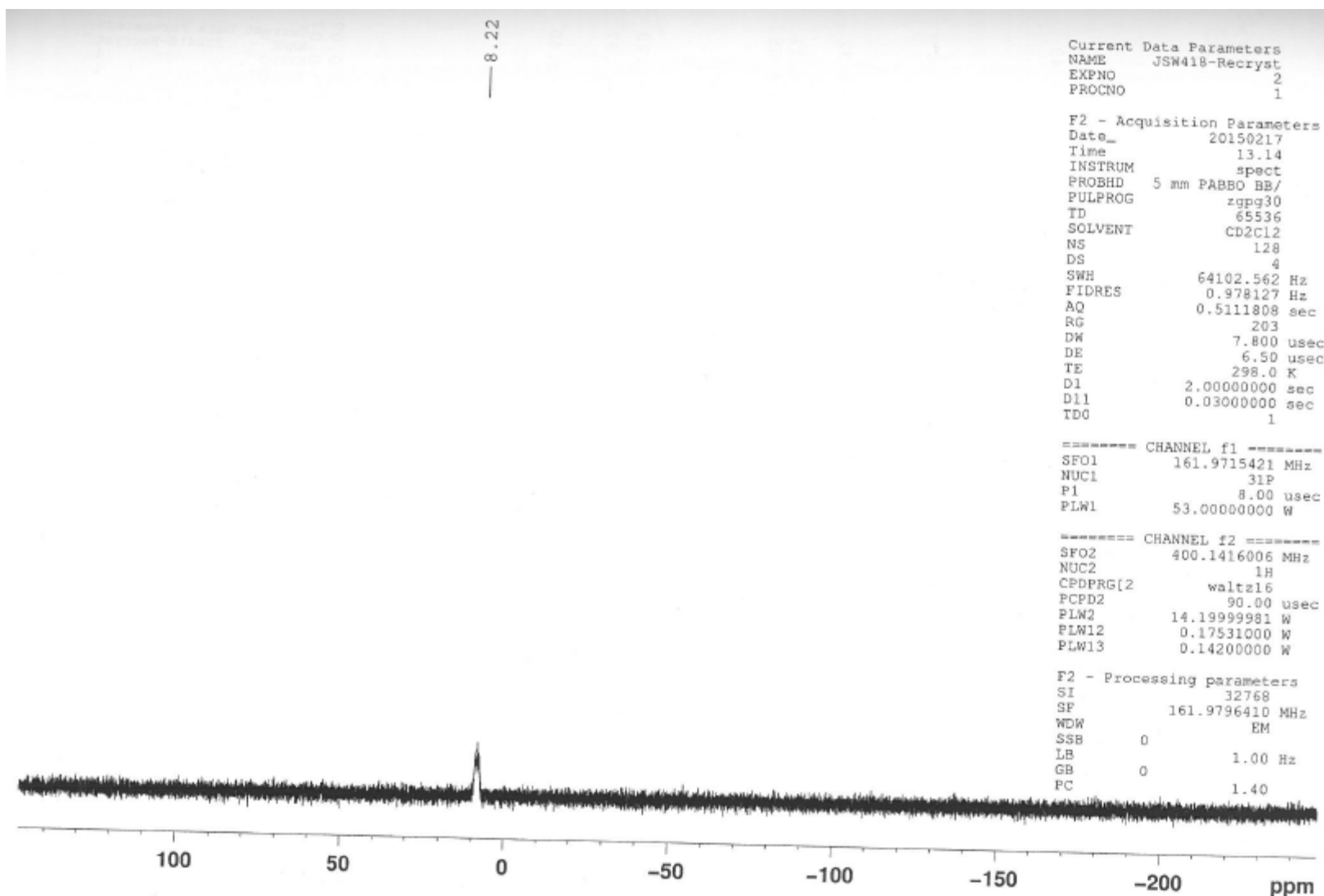
F2 - Processing parameters
SI 32768
SF 128.3808140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



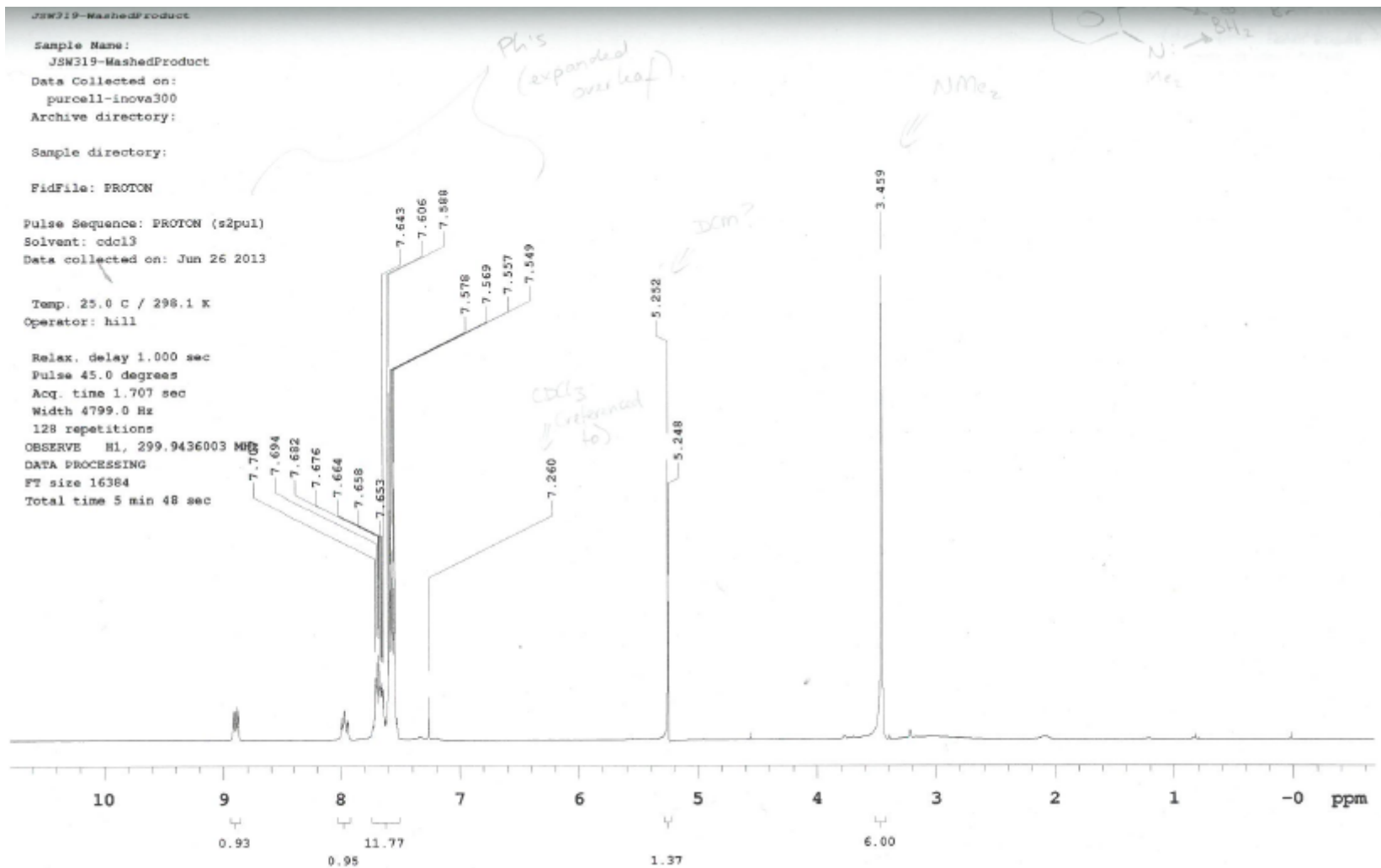
Compound 11 - $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



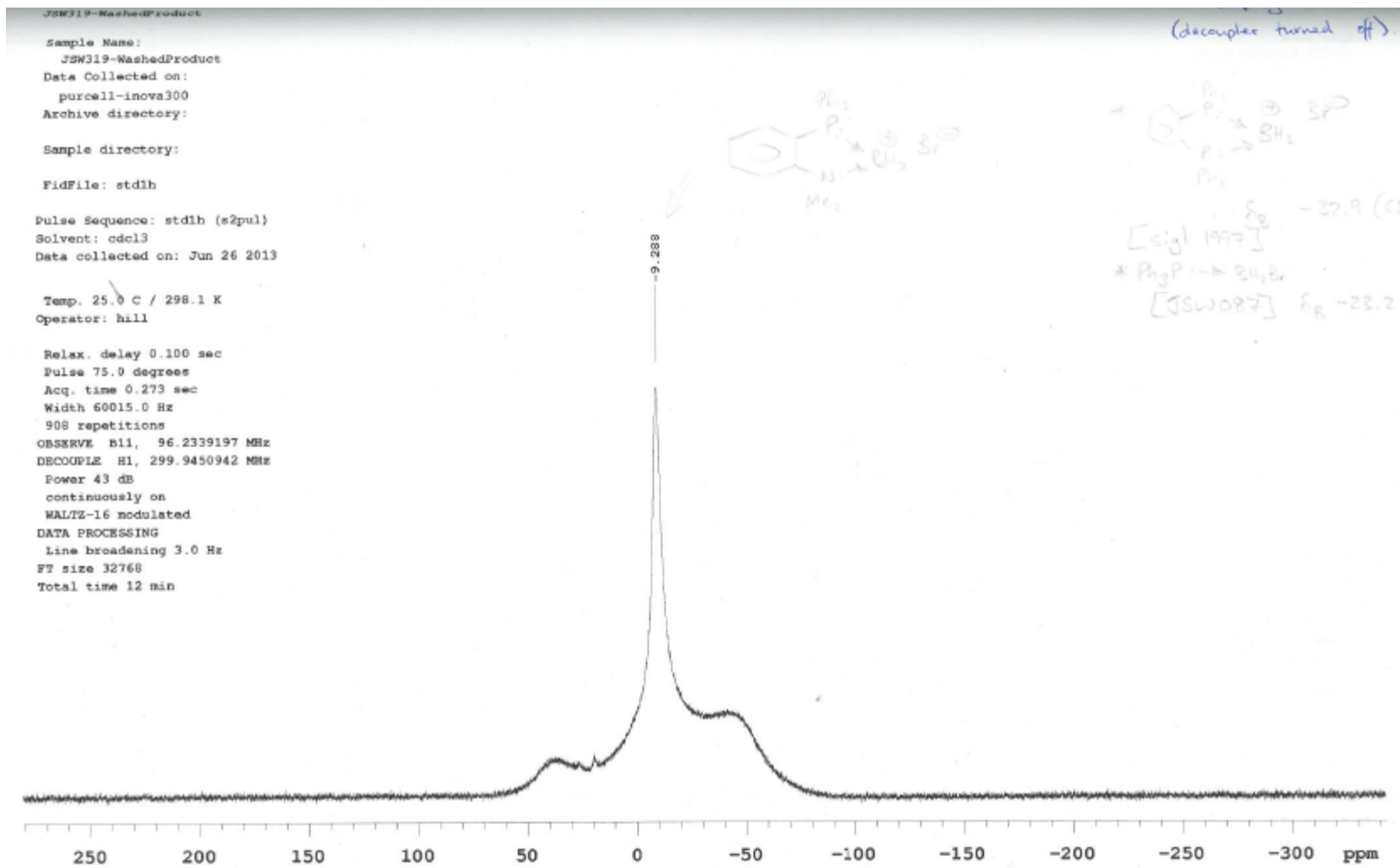
Compound 11 - $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



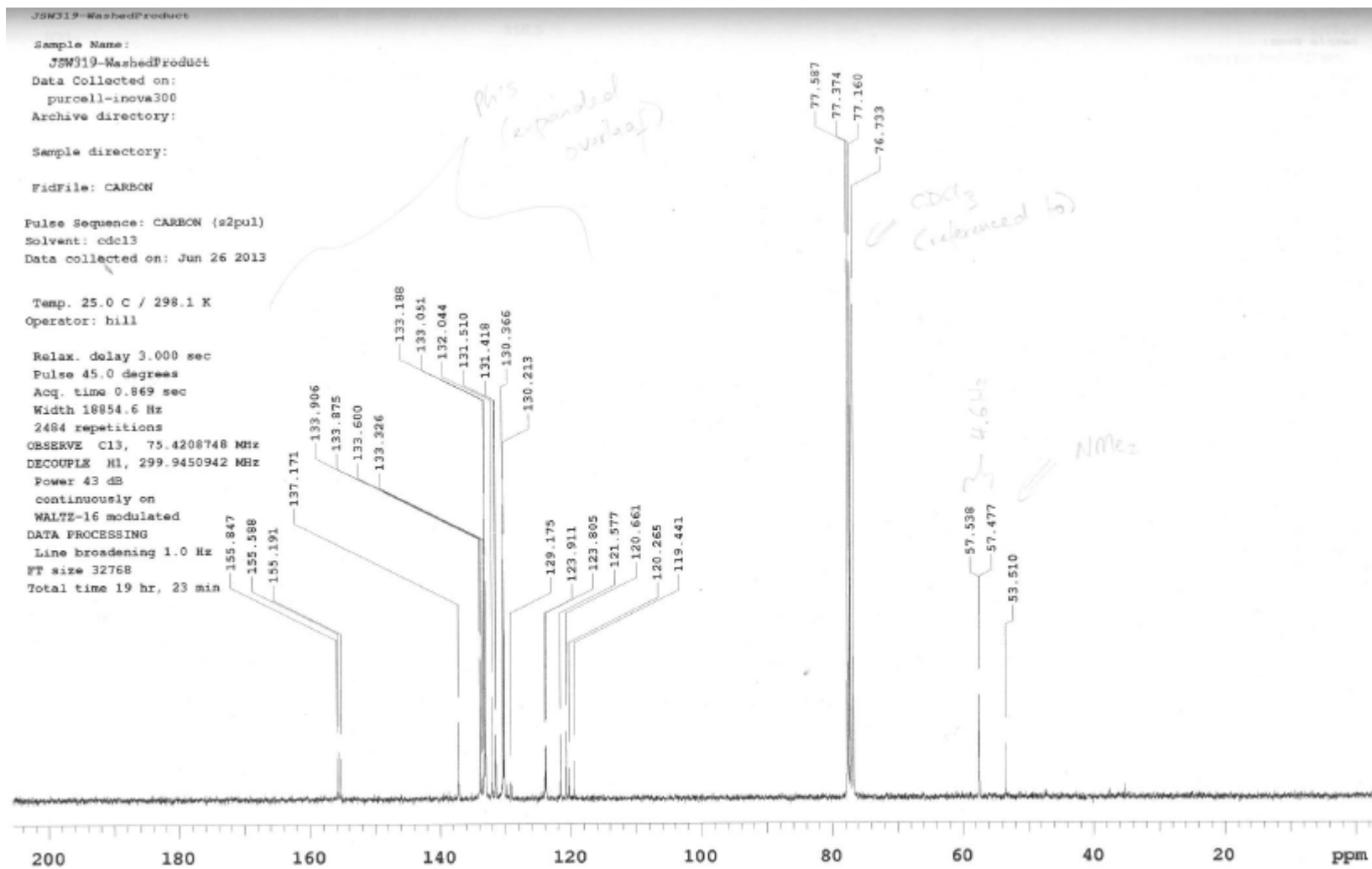
Compound 11 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



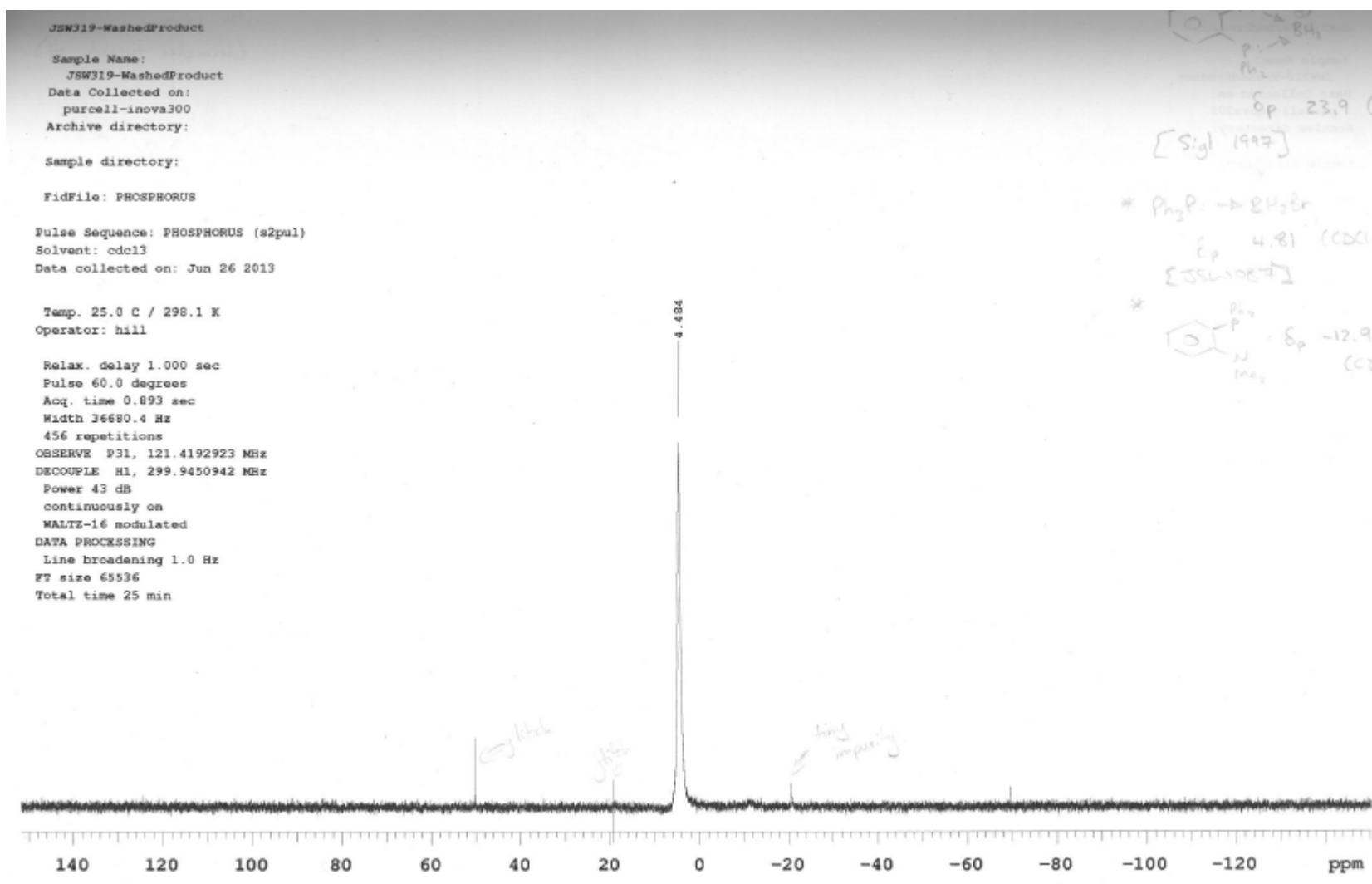
Compound 12 - ¹H NMR spectrum



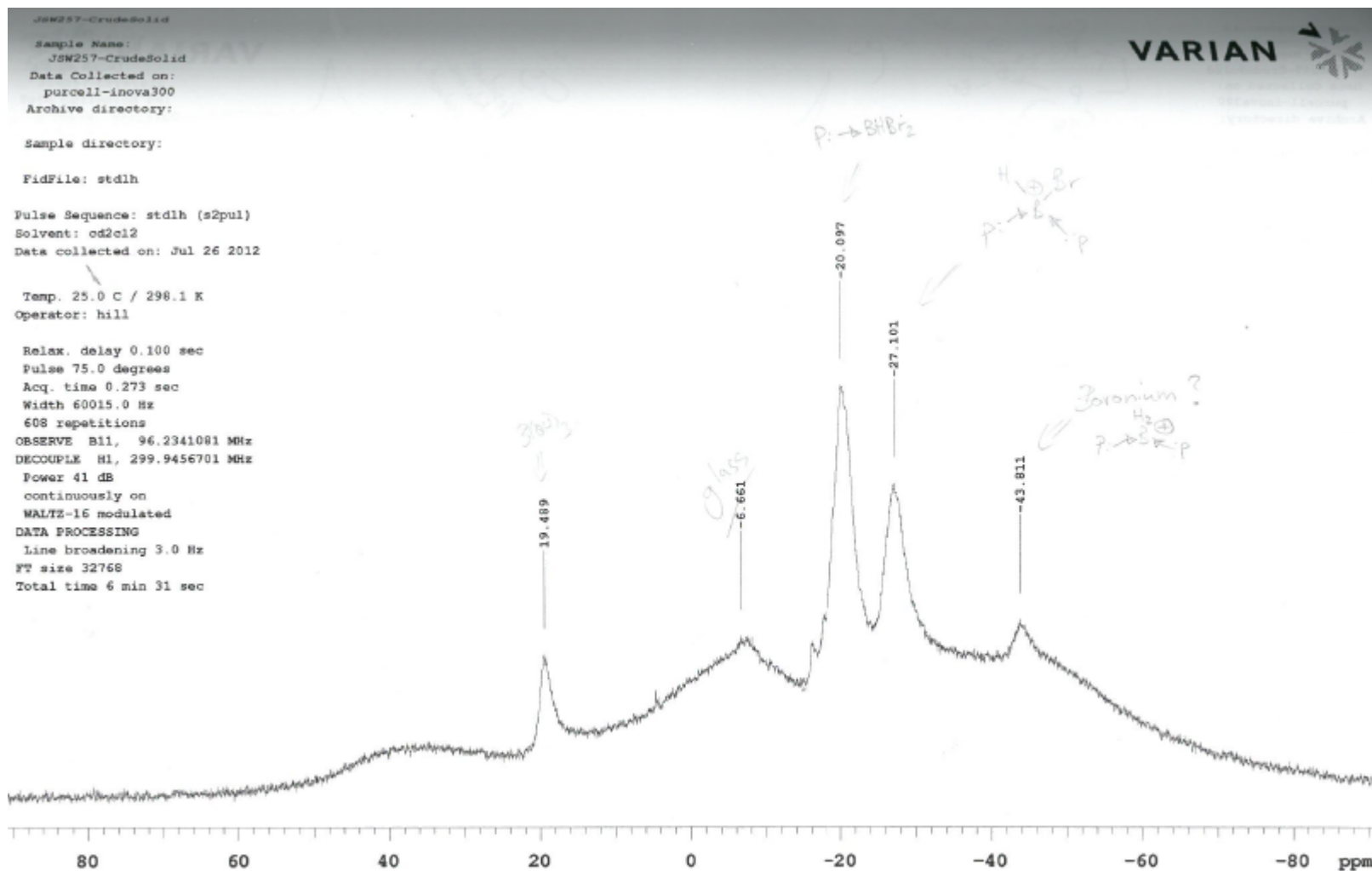
Compound 12 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



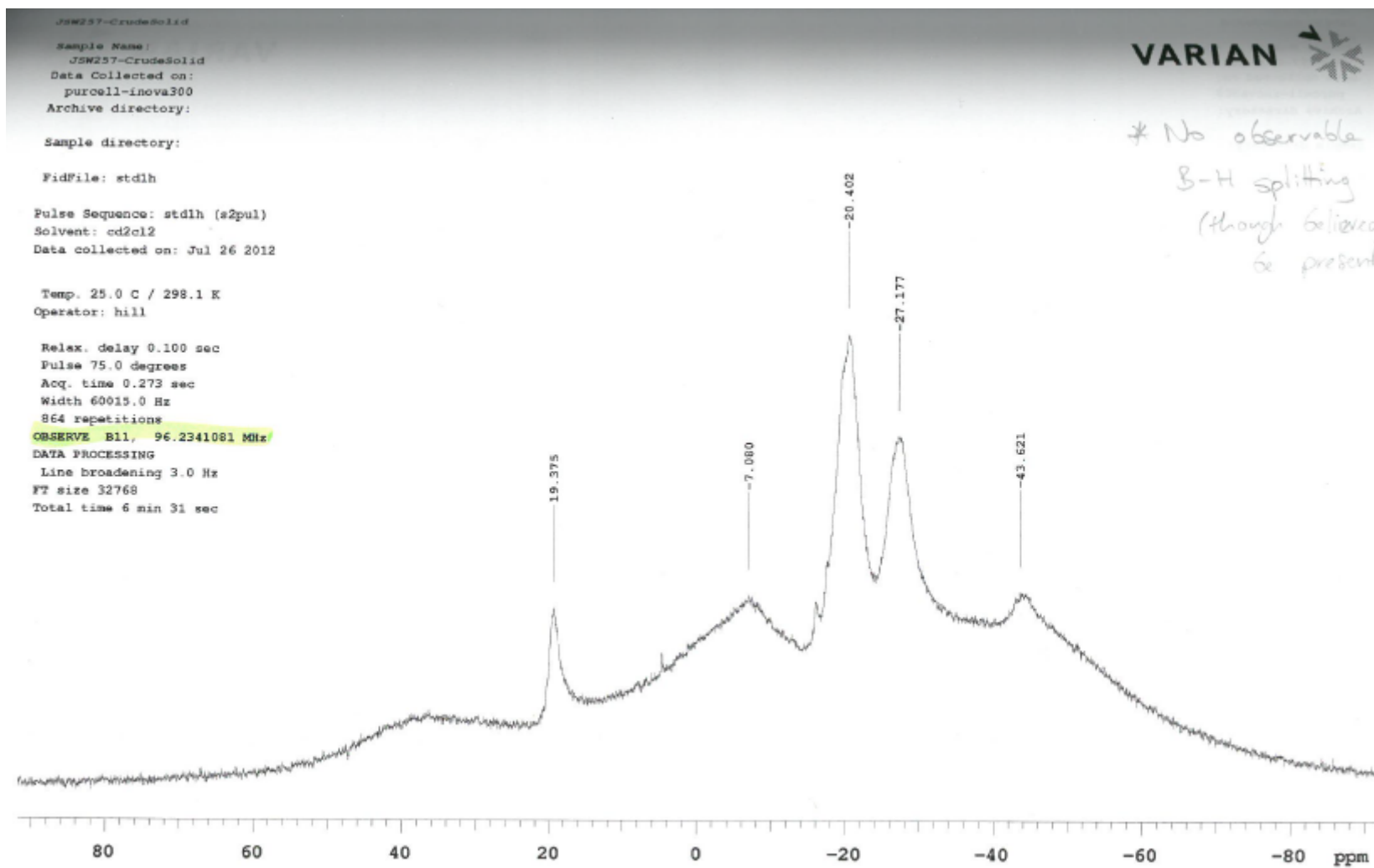
Compound 12 - $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



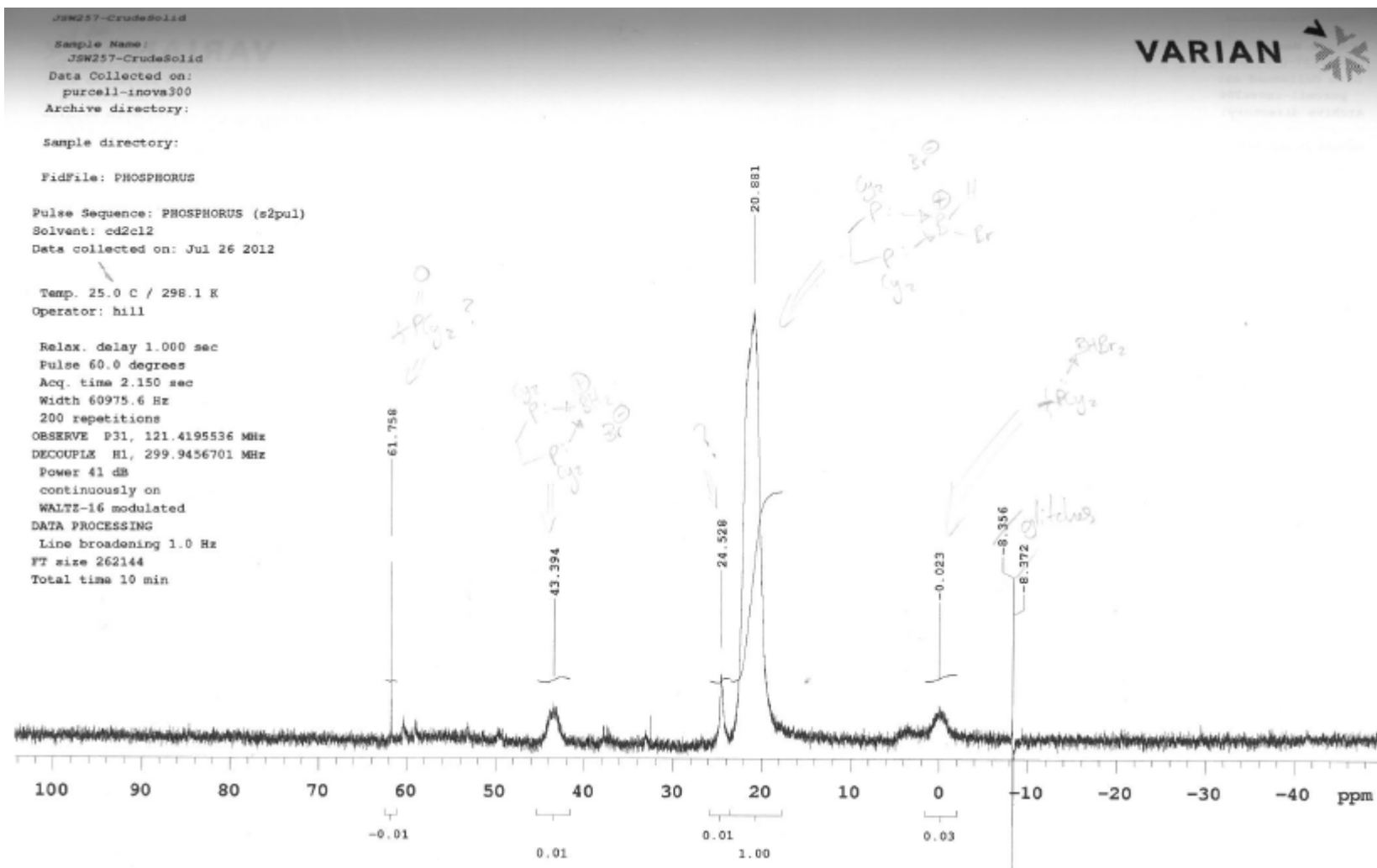
Compound 12 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



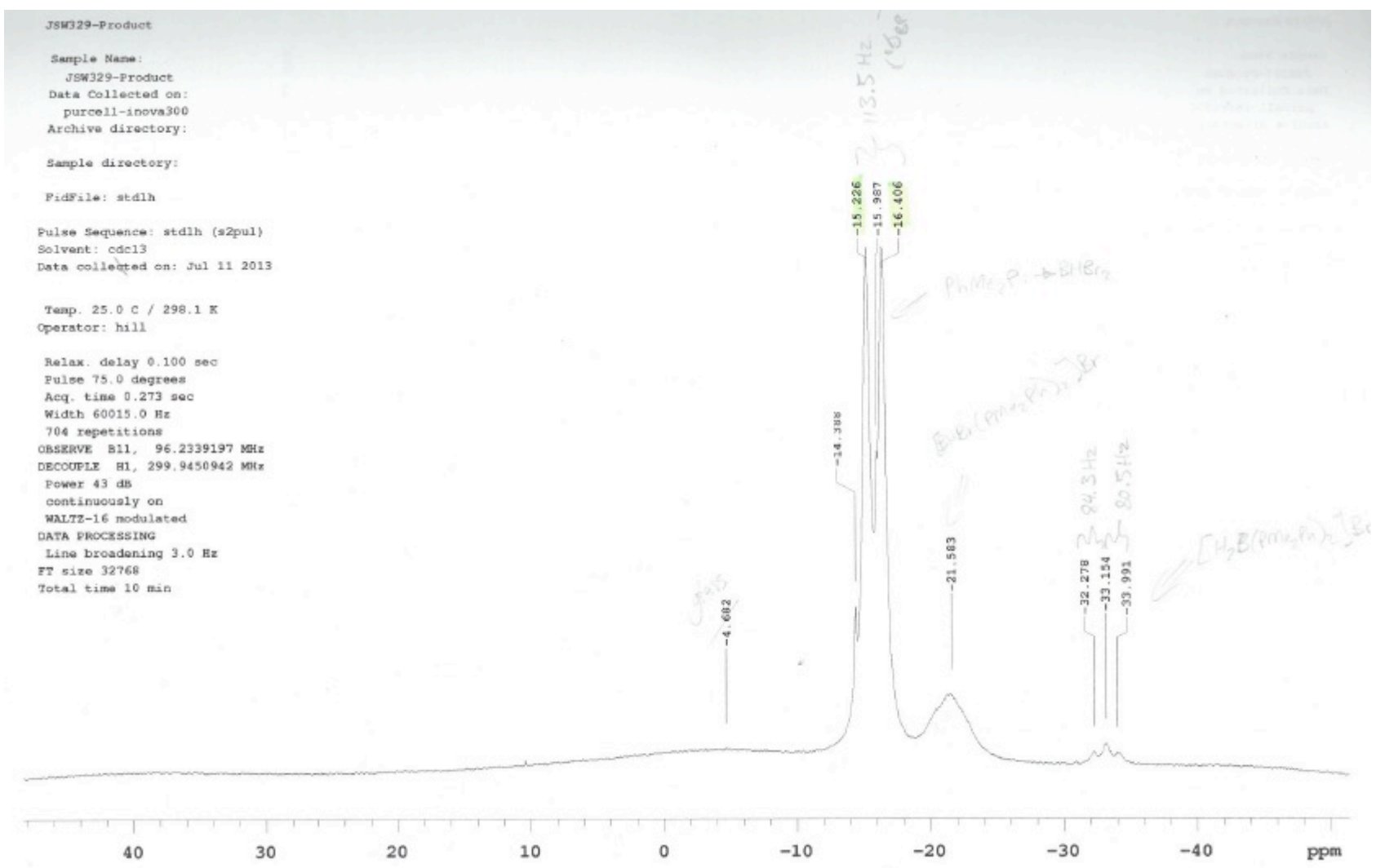
Compound 13 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 13 - ^{11}B NMR spectrum



Compound 13 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 14 – ¹¹B{¹H} NMR spectrum

JSM329-Product

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

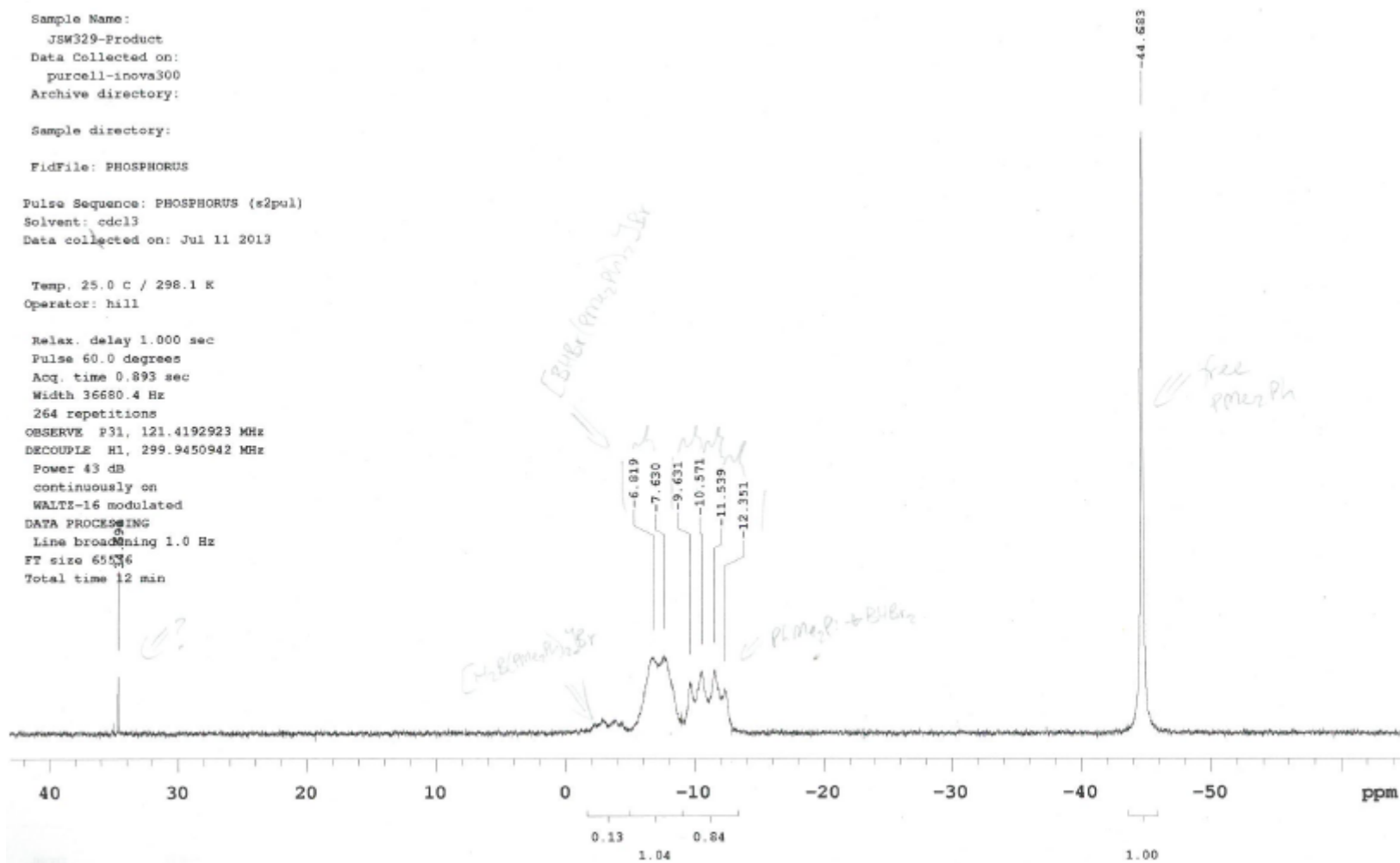
Sample directory:

FidFile: PHOSPHORUS

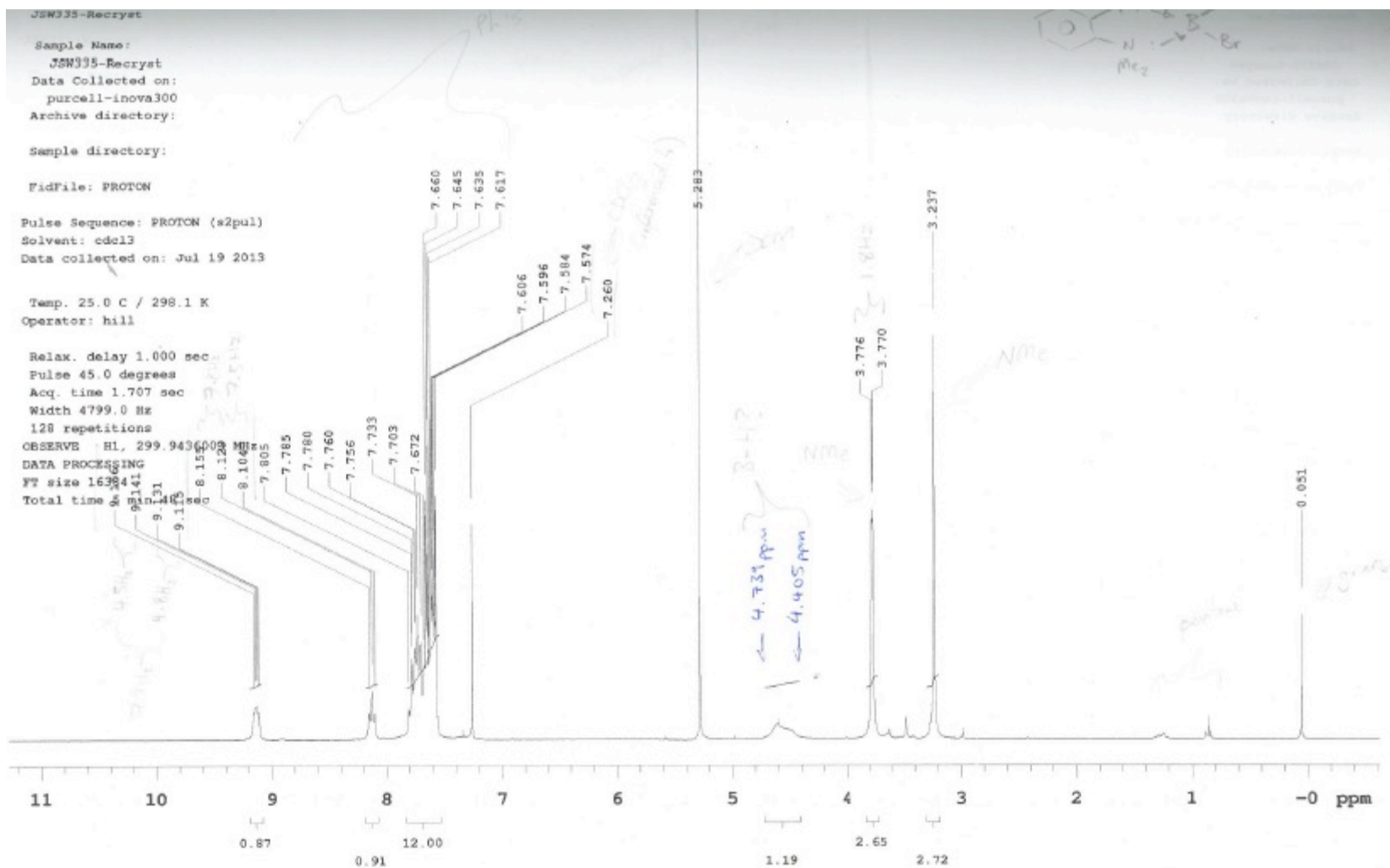
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdcl3
Data collected on: Jul 11 2013

Temp. 25.0 c / 298.1 K
Operator: hill

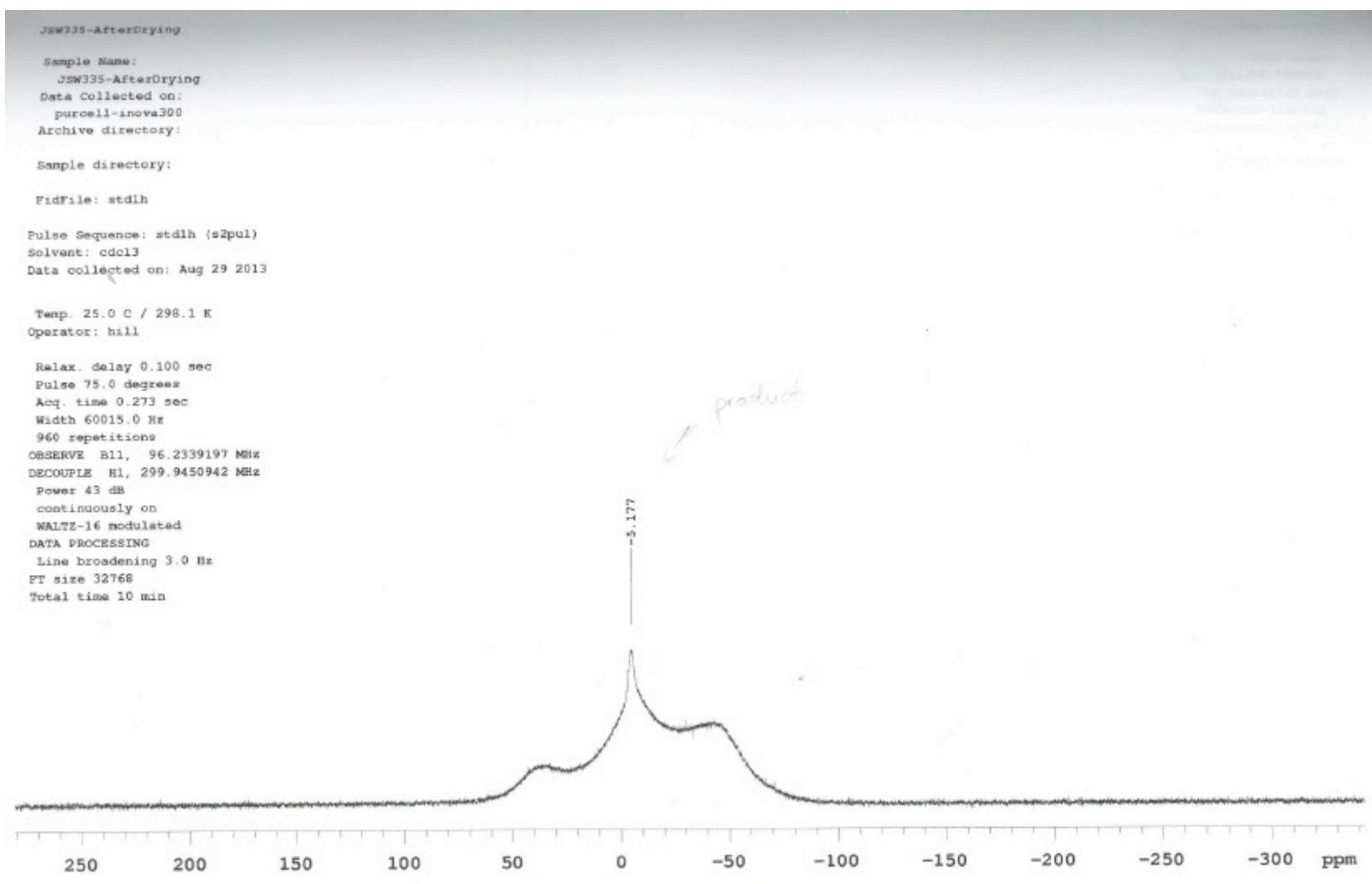
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
264 repetitions
OBSERVE F31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 12 min



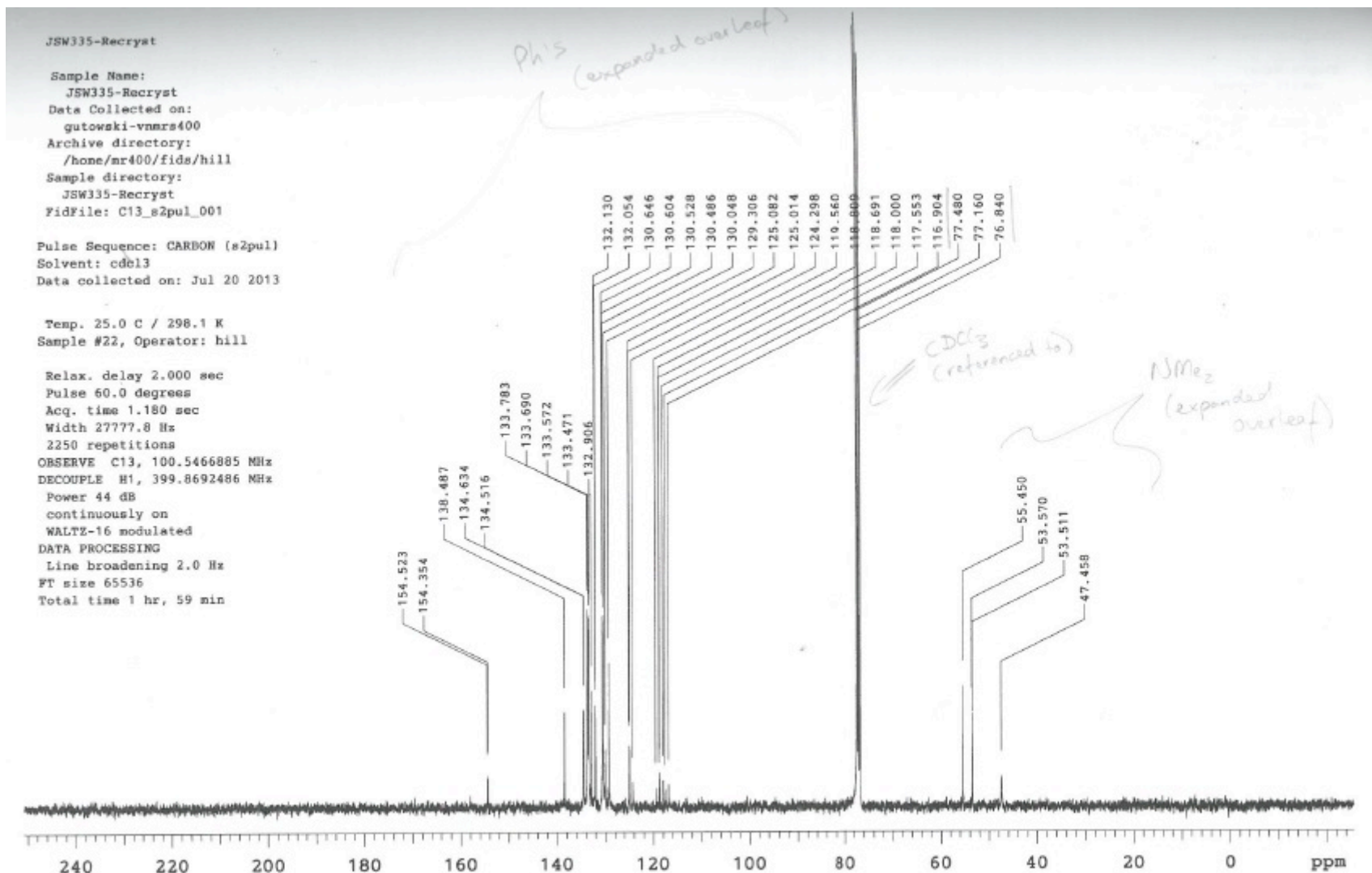
Compound 14 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



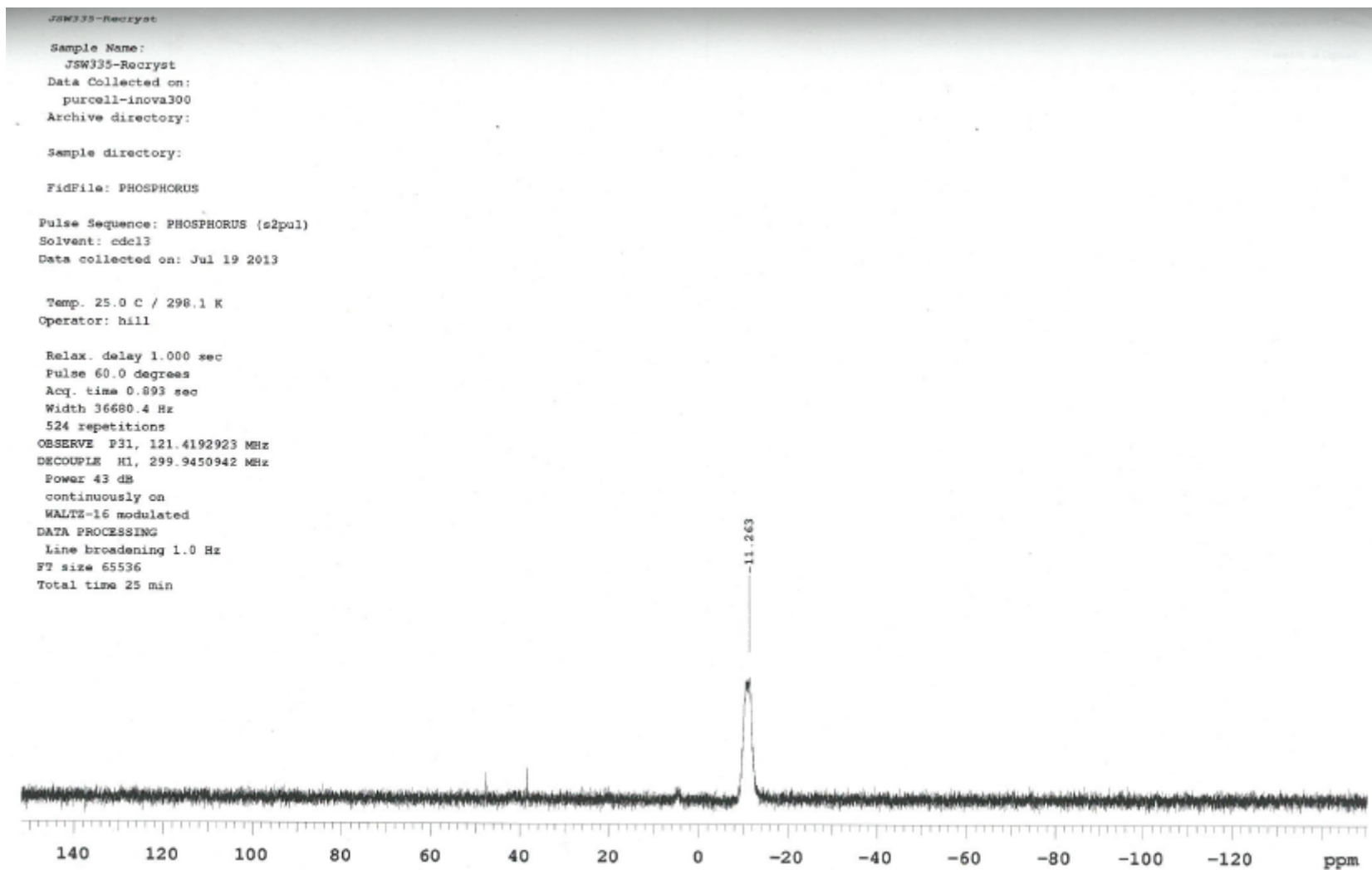
Compound 15 – ^1H NMR spectrum



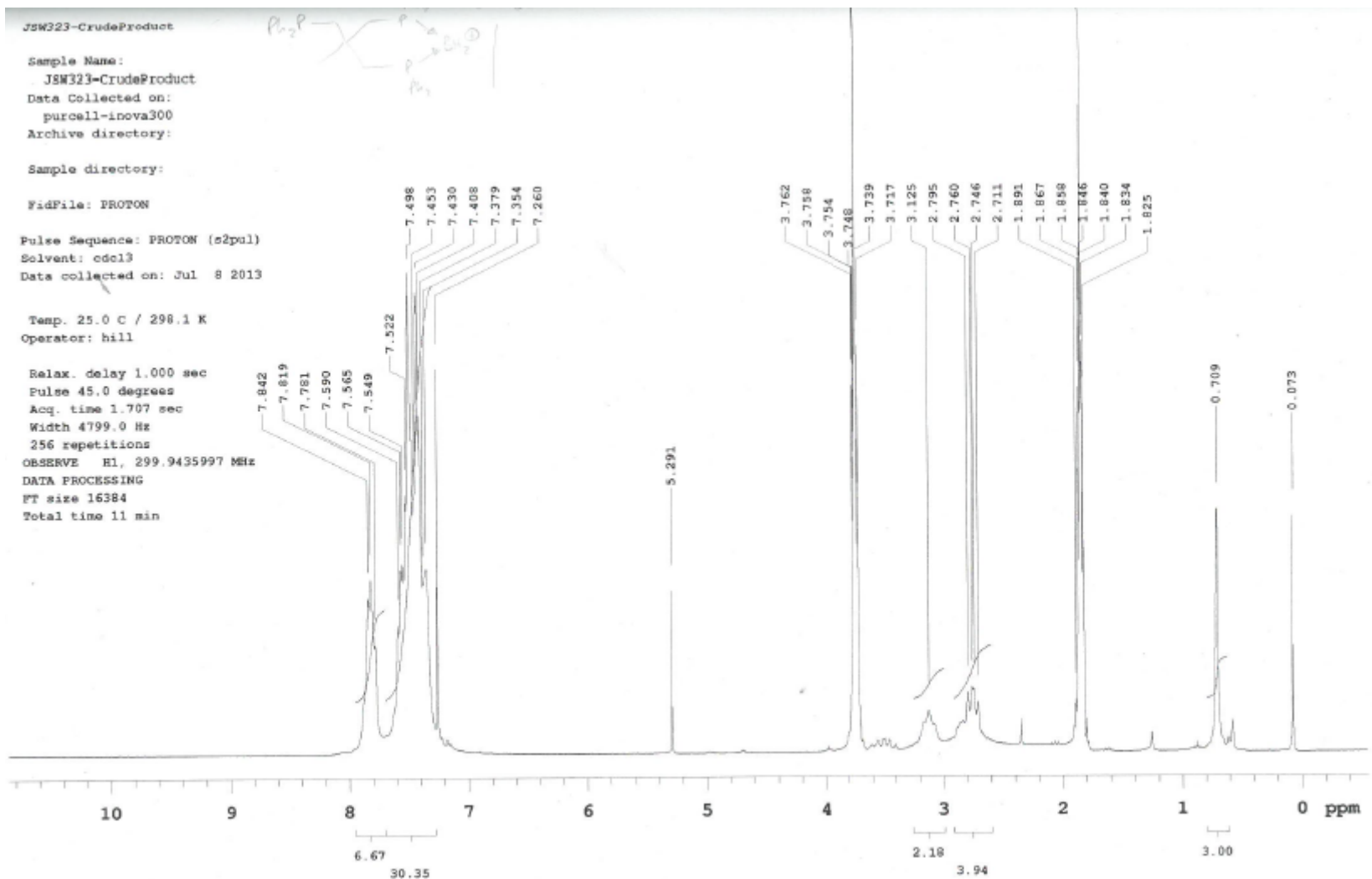
Compound 15 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



Compound 15 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



Compound 15 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 17 - ^1H NMR spectrum

JSW323-CrudeProduct

Sample Name:

JSW323-CrudeProduct

Data Collected on:

purcell-inova300

Archive directory:

Sample directory:

FidFile: std1h

Pulse Sequence: std1h (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

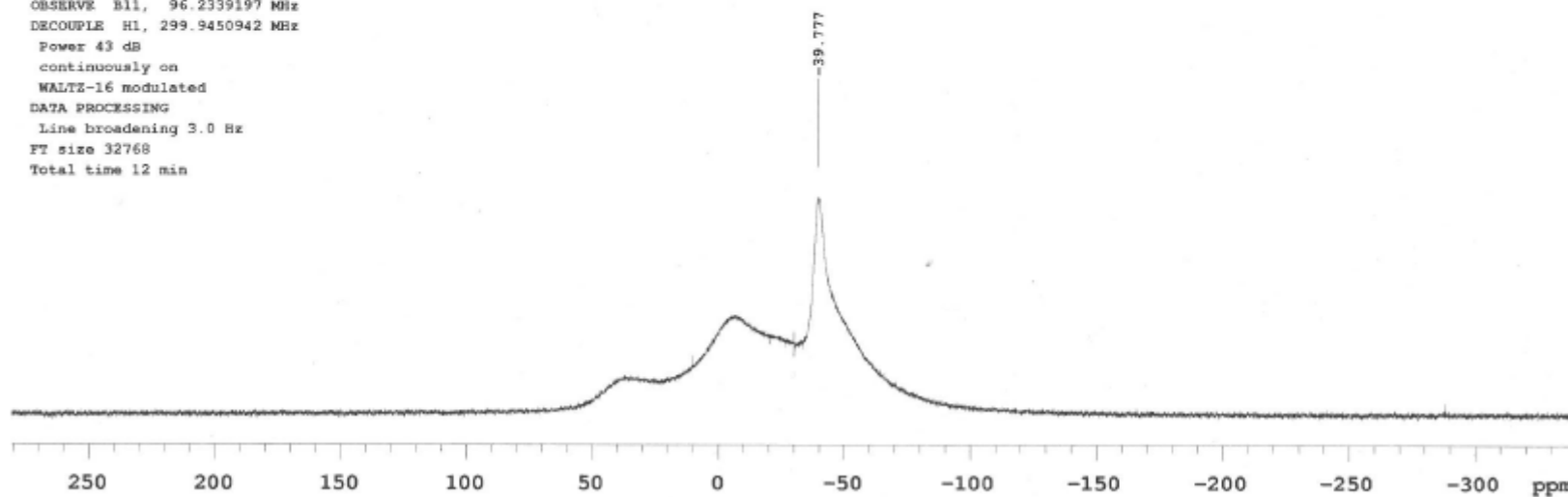
WALTZ-16 modulated

DATA PROCESSING

Line broadening 3.0 Hz

FT size 32768

Total time 12 min



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

JSM323-CrudeProduct

Sample Name:
JSM323-CrudeProduct
Data Collected on:
parcell-inova300
Archive directory:

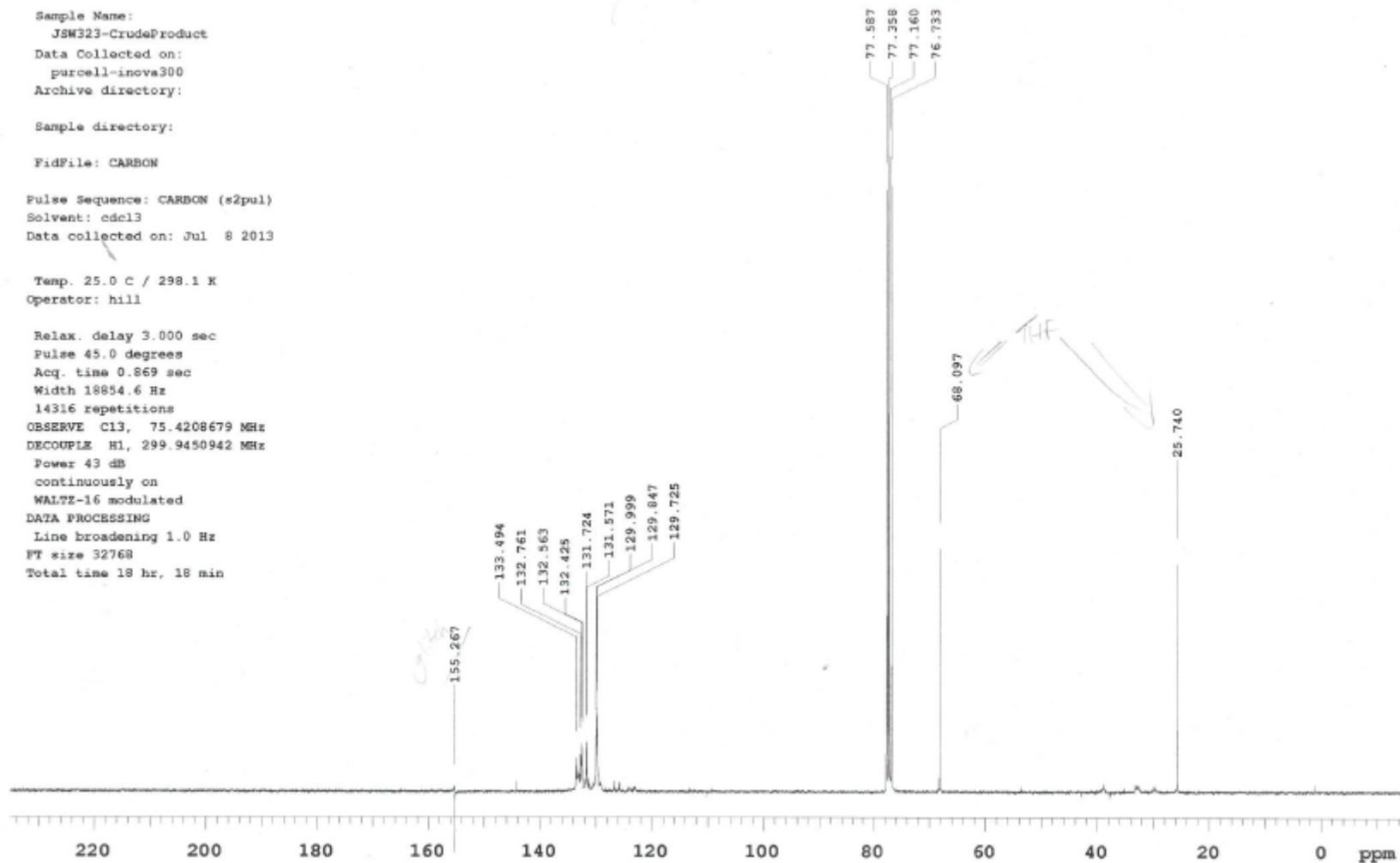
Sample directory:

FidFile: CARBON

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
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 - $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum

J5W323-CrudeProduct

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

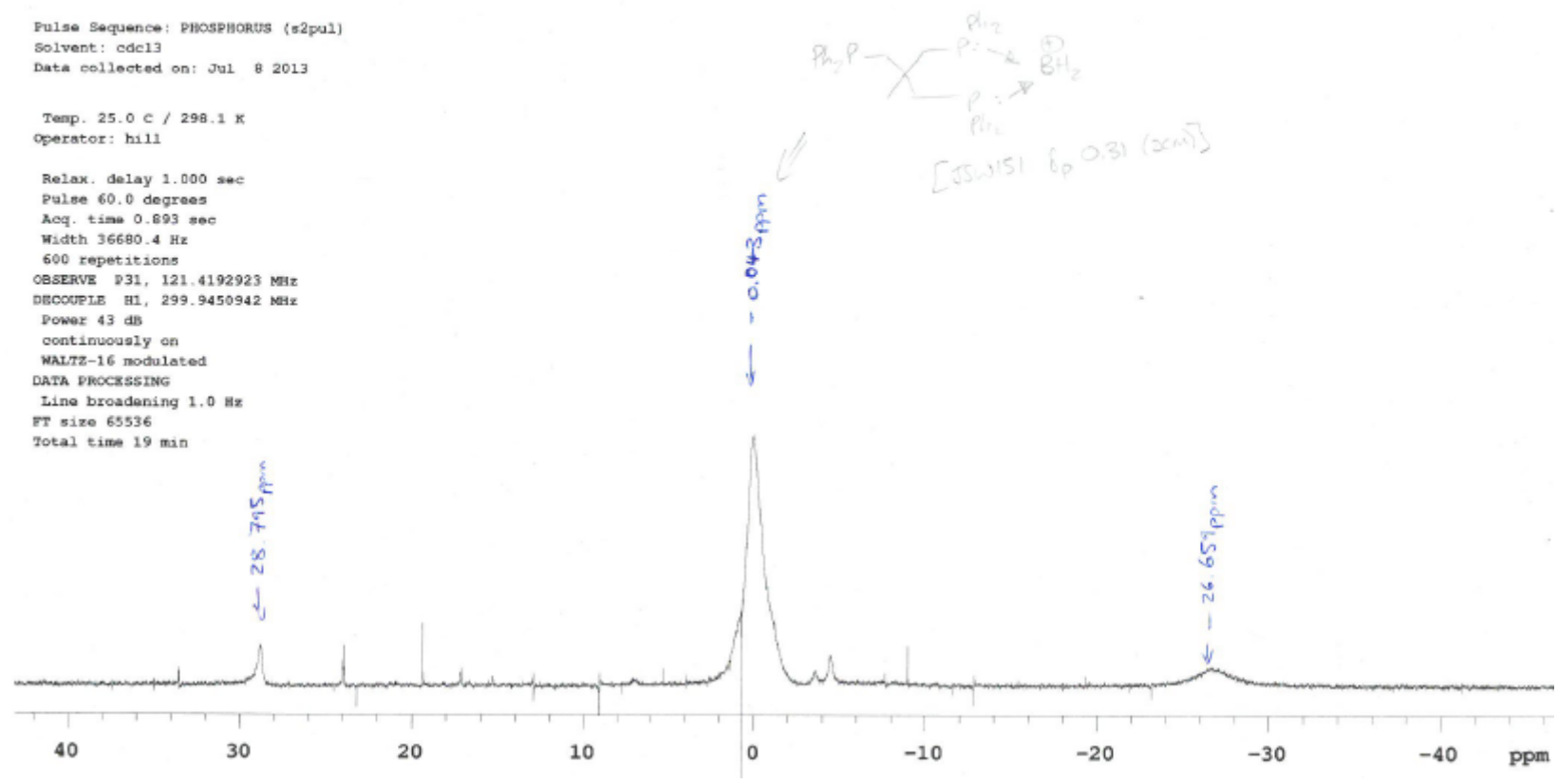
Sample directory:

Fidfile: PHOSPHORUS

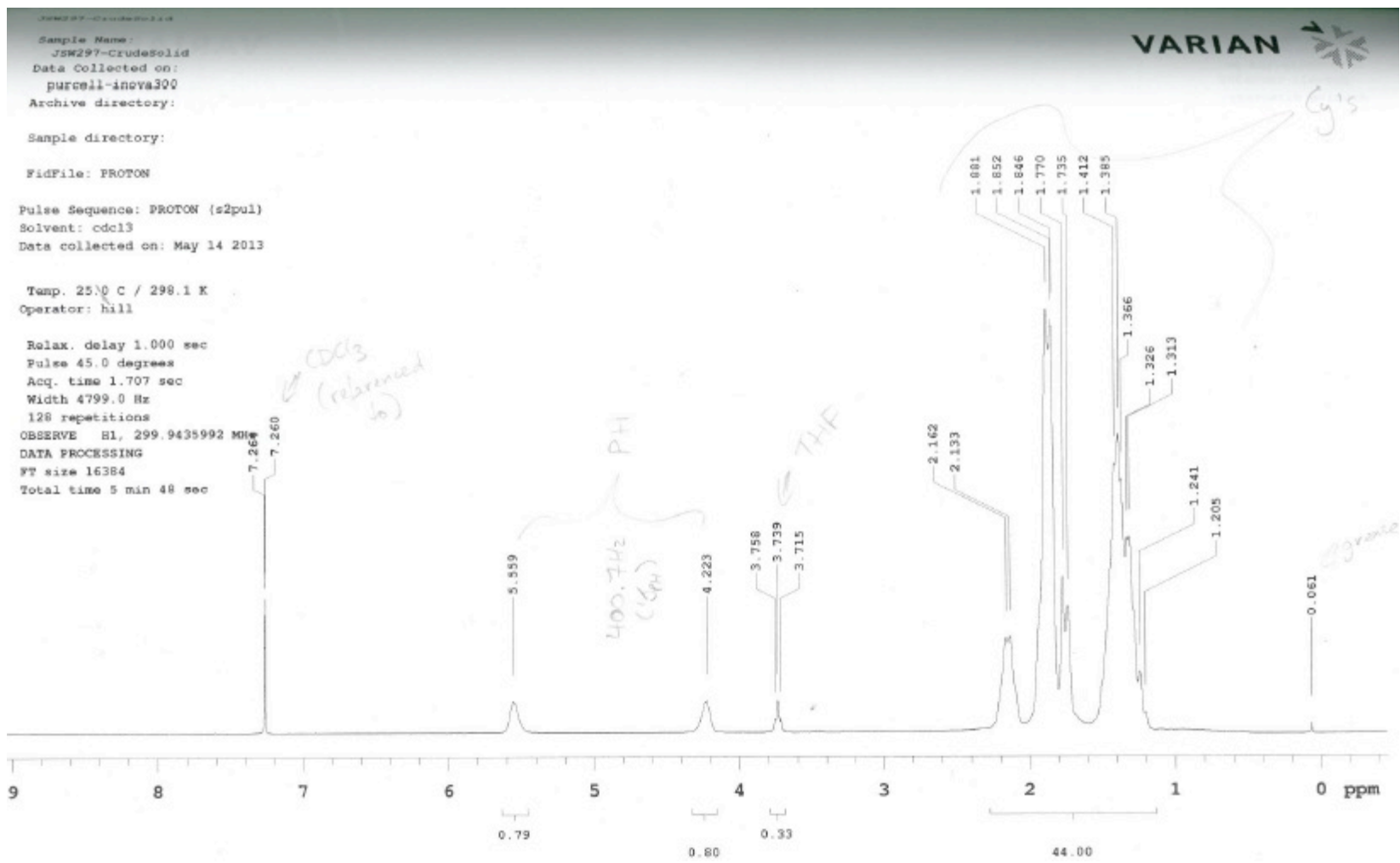
Pulse Sequence: PHOSPHORUS (s2pul)
Solvent: cdc13
Data collected on: Jul 8 2013

Temp. 25.0 C / 298.1 K
Operator: hill

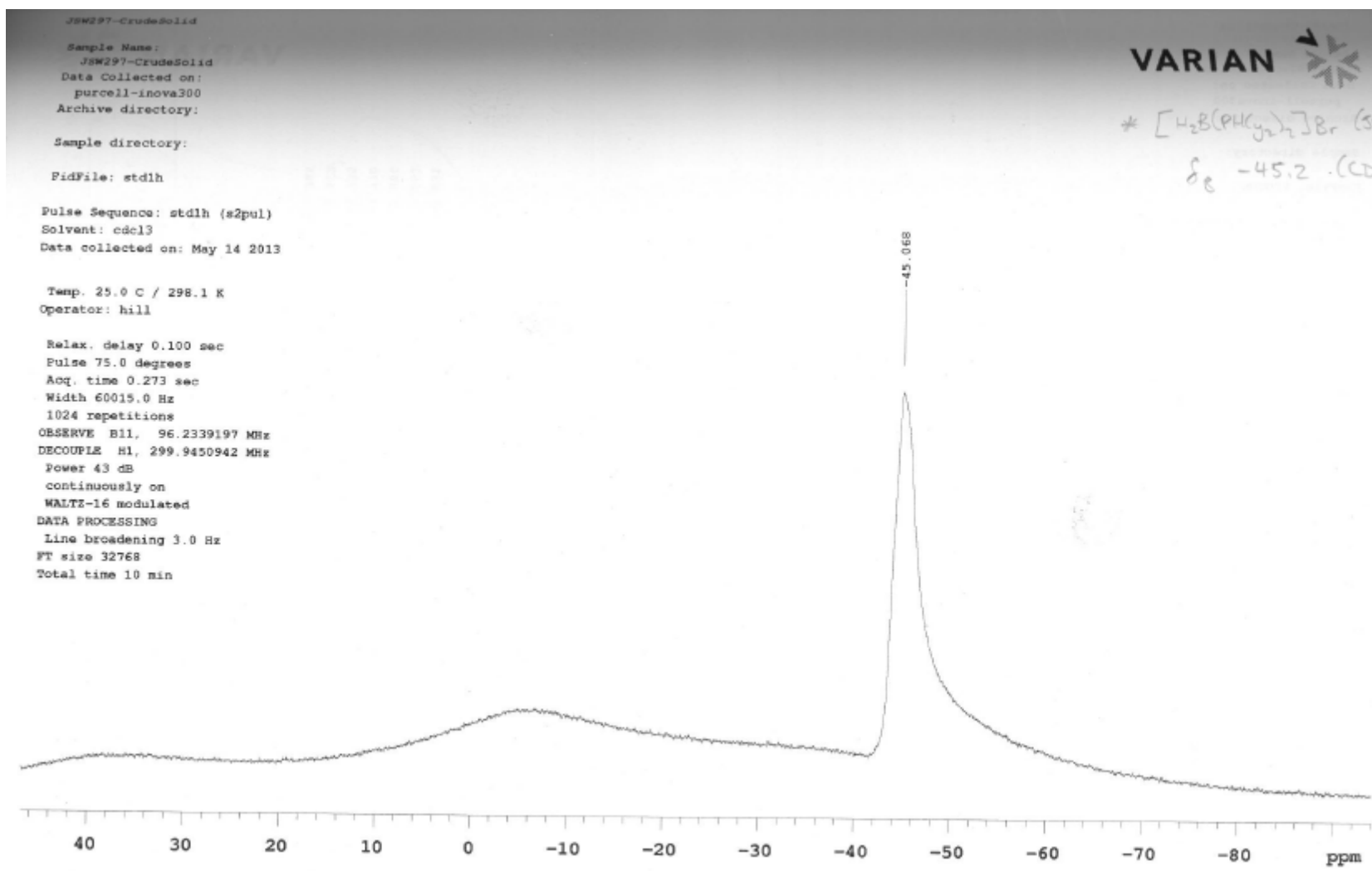
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 0.893 sec
Width 36680.4 Hz
600 repetitions
OBSERVE P31, 121.4192923 MHz
DECOUPLE H1, 299.9450942 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 19 min



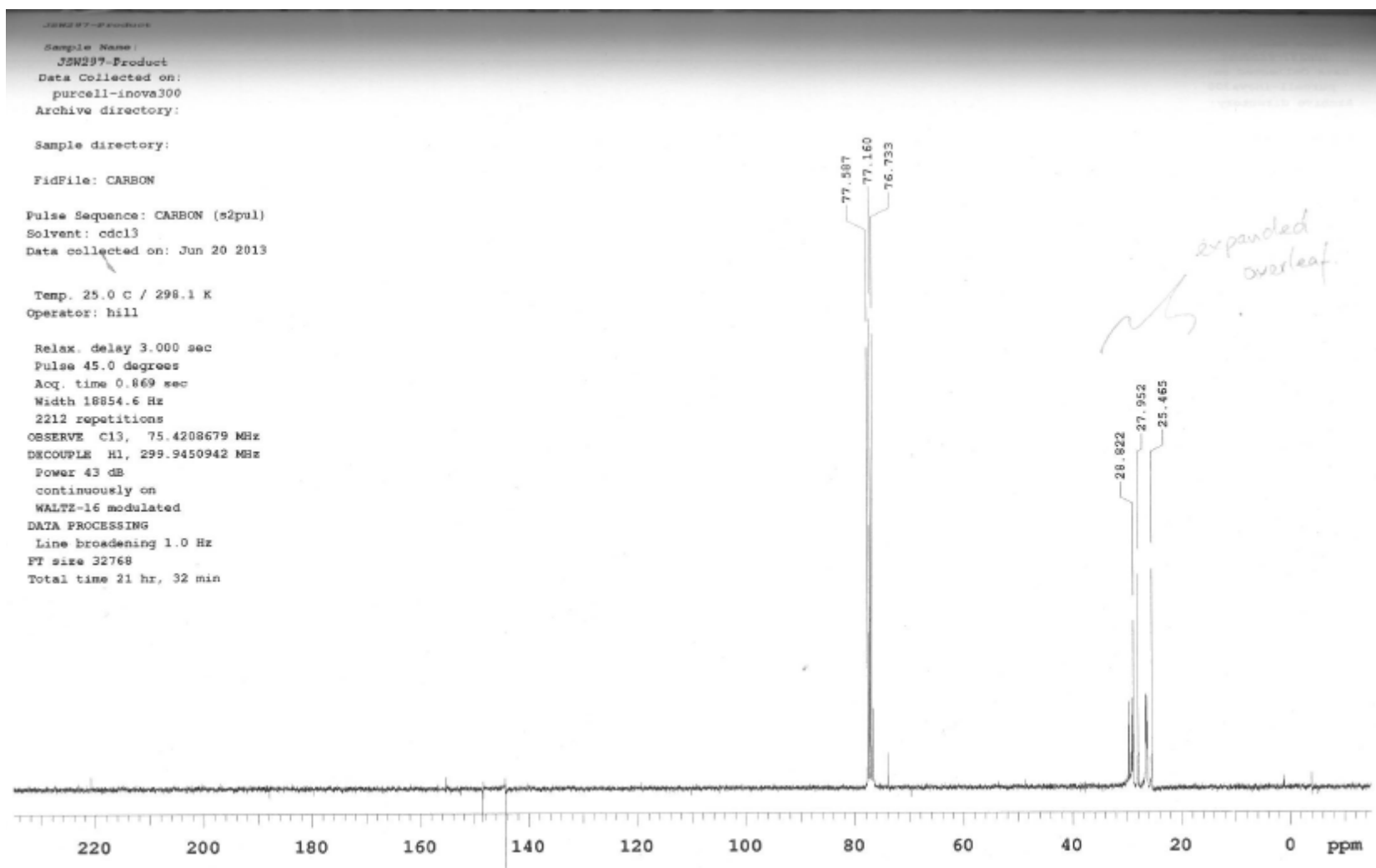
Compound 17 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



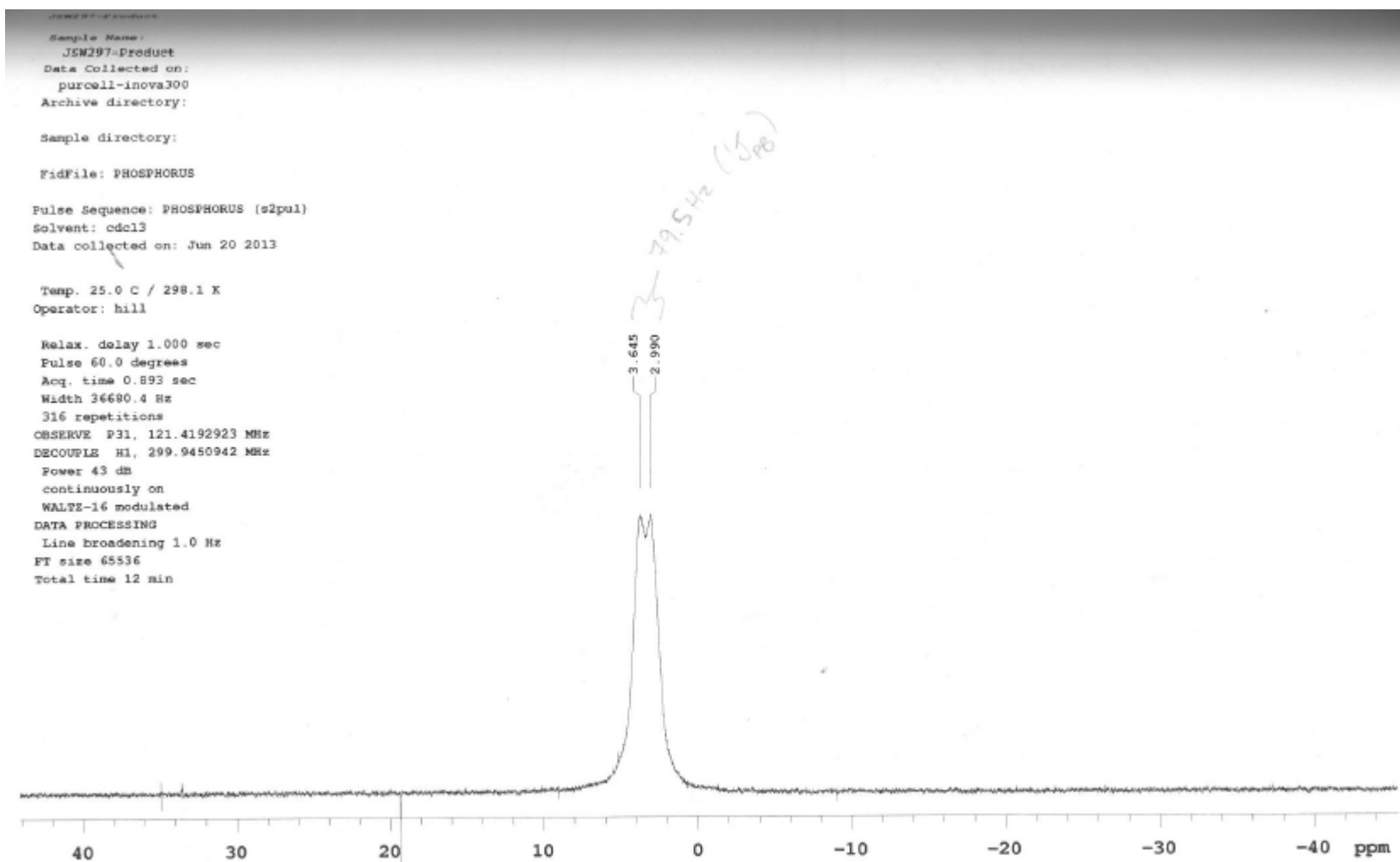
Compound 18 – ¹H NMR spectrum



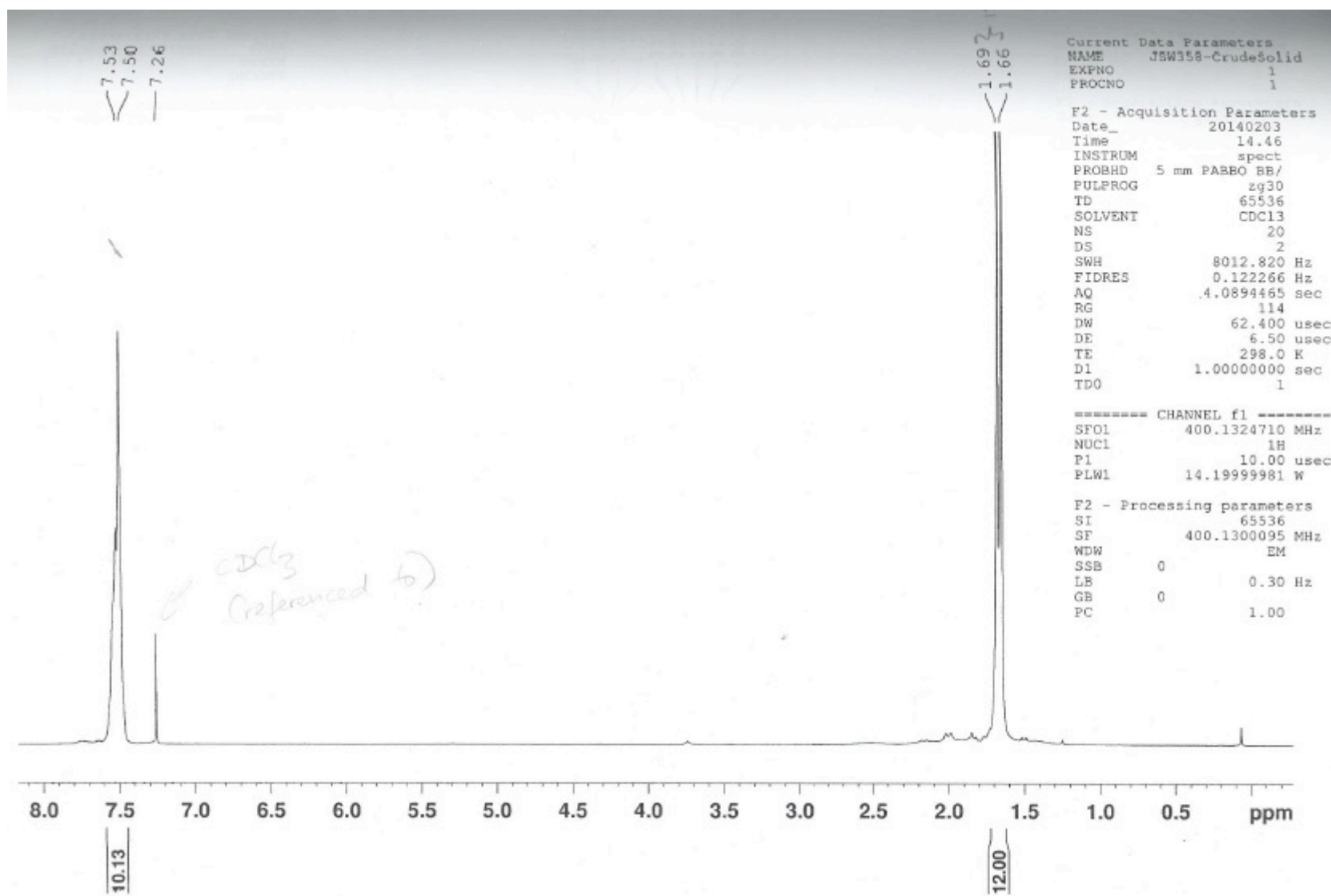
Compound 18 - $^{11}B\{^1H\}$ NMR spectrum



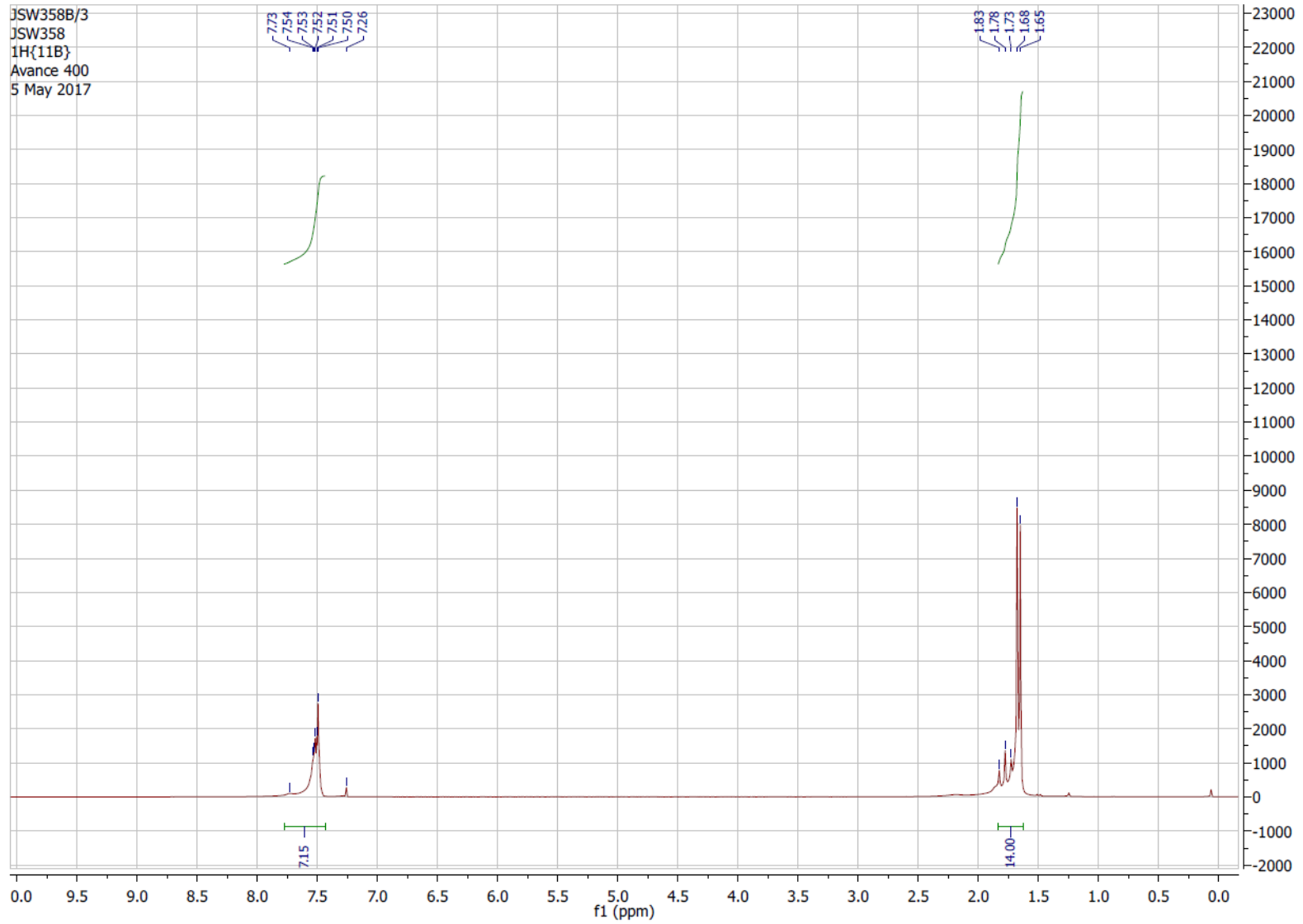
Compound 18 - $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



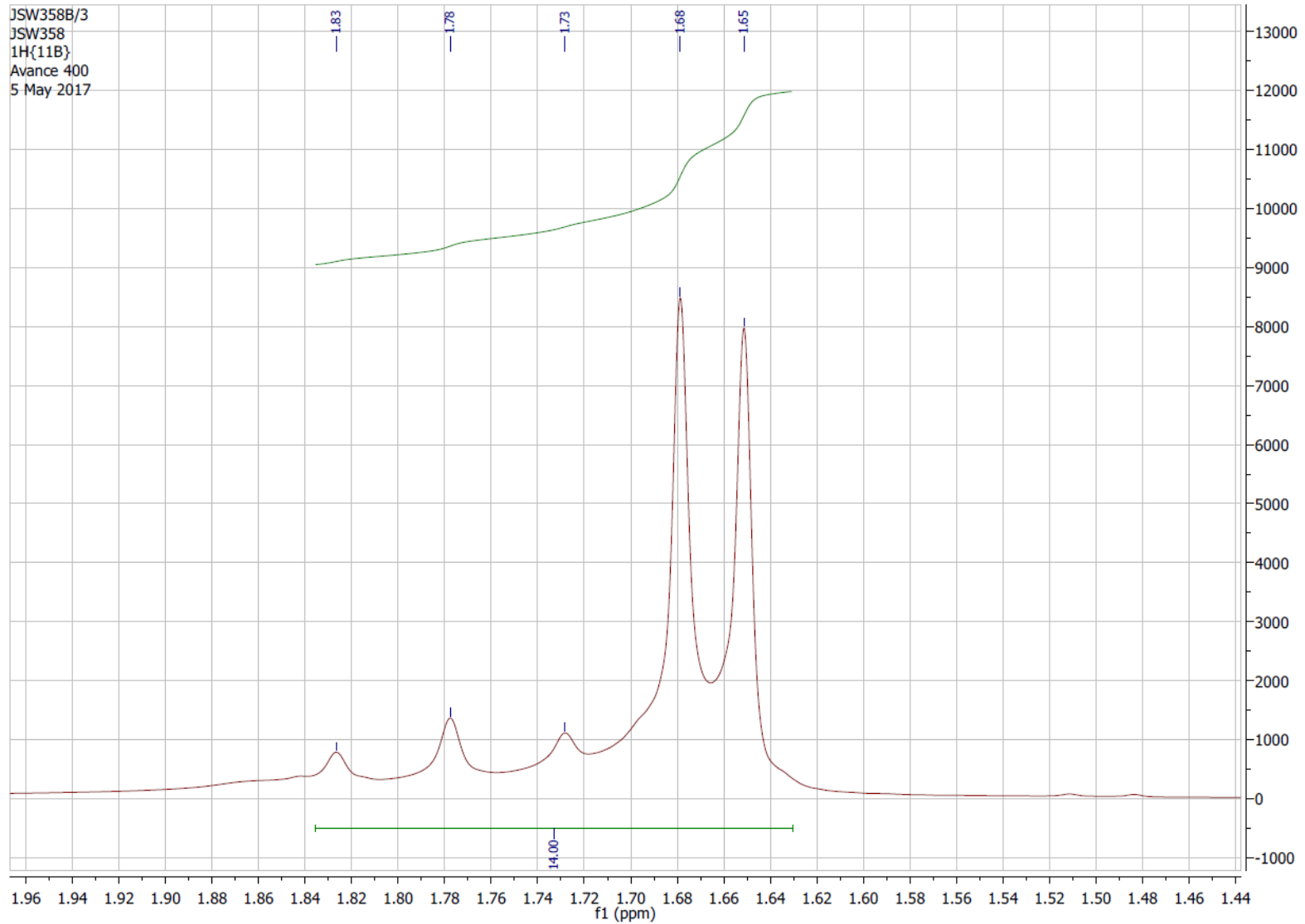
Compound 18 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



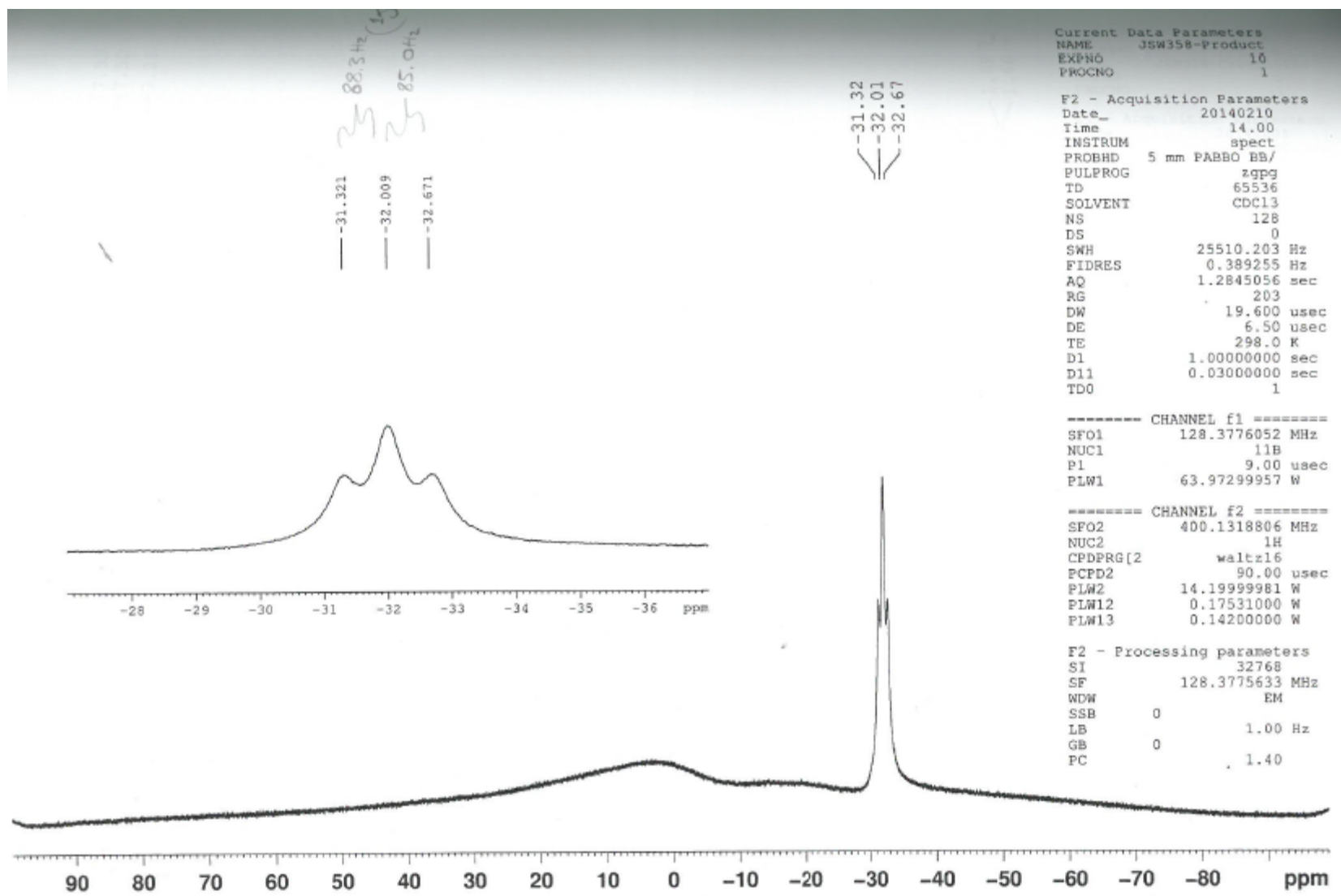
Compound 19 – ¹H NMR spectrum



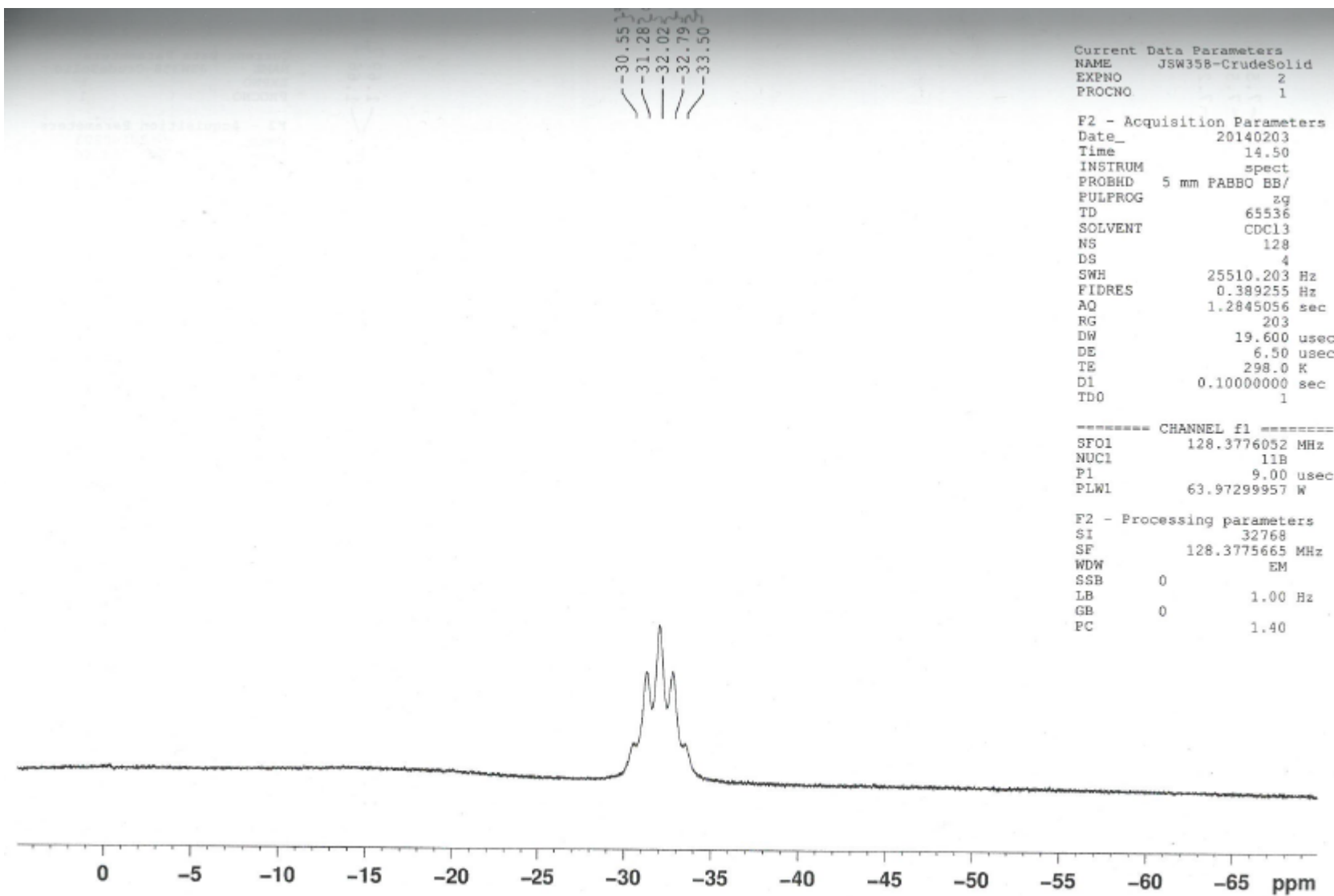
Compound 19 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum



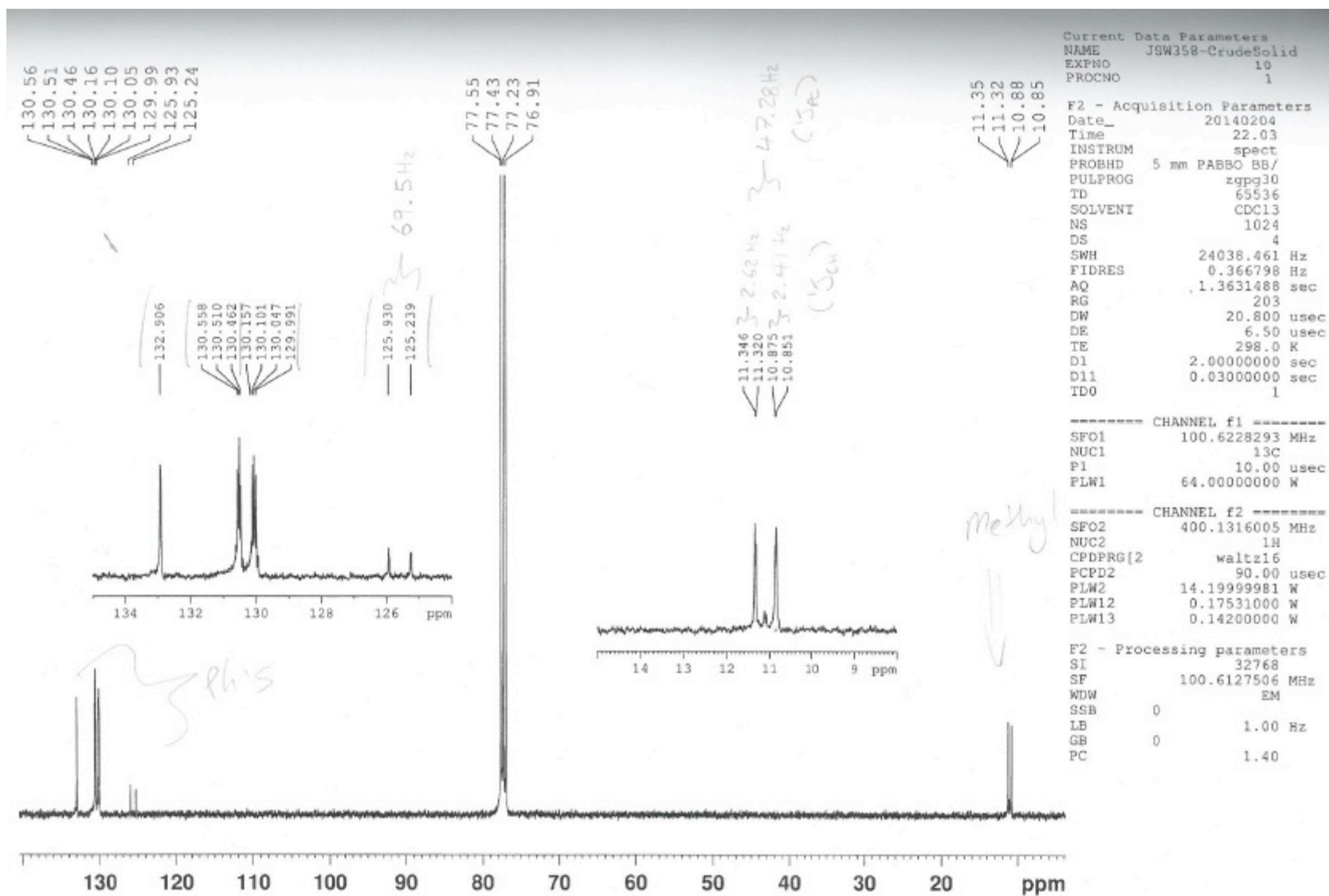
Compound 19 – $^1\text{H}\{^{11}\text{B}\}$ NMR spectrum (Expansion of BH_2 resonance)



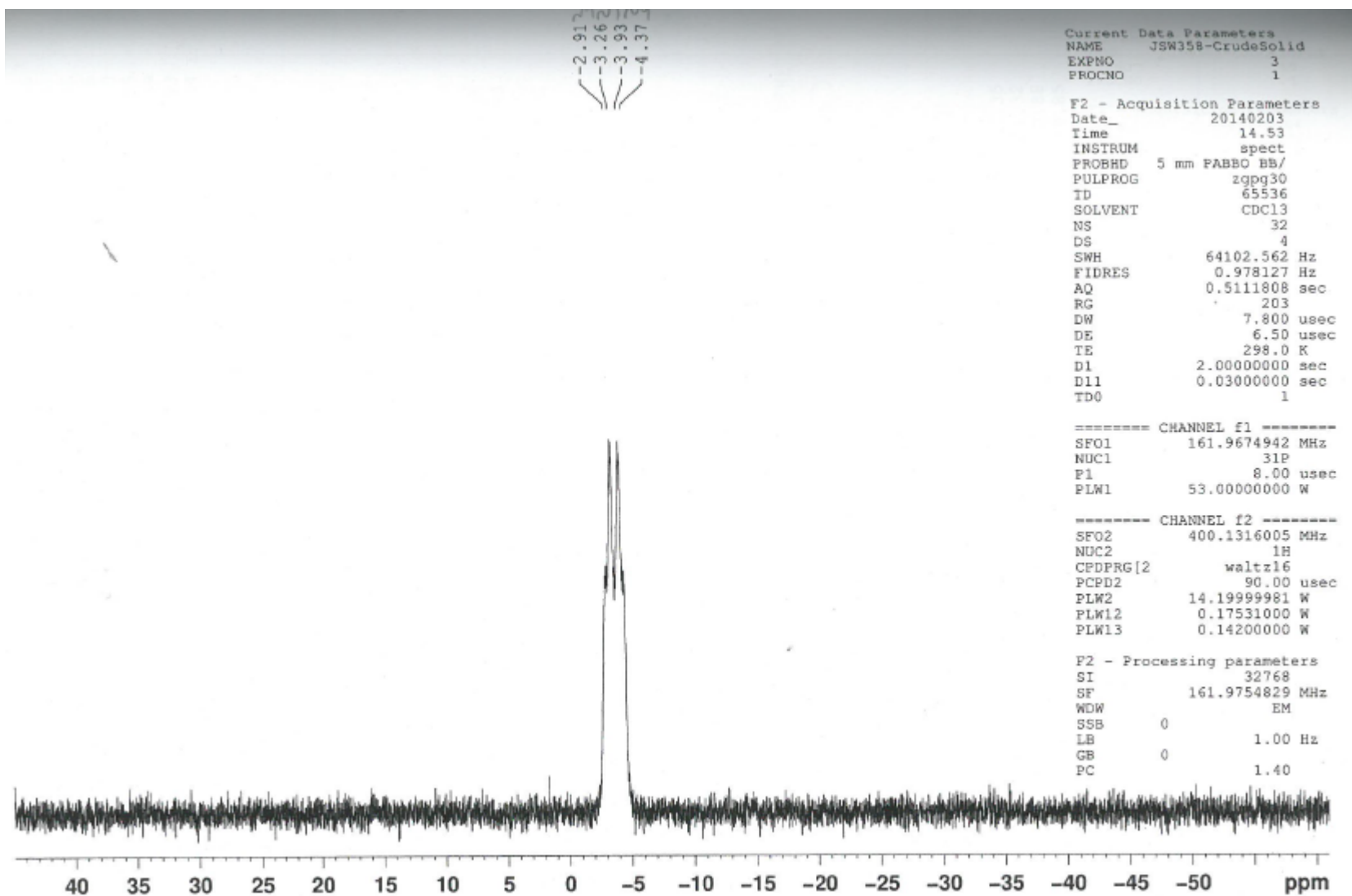
Compound 19 – $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum



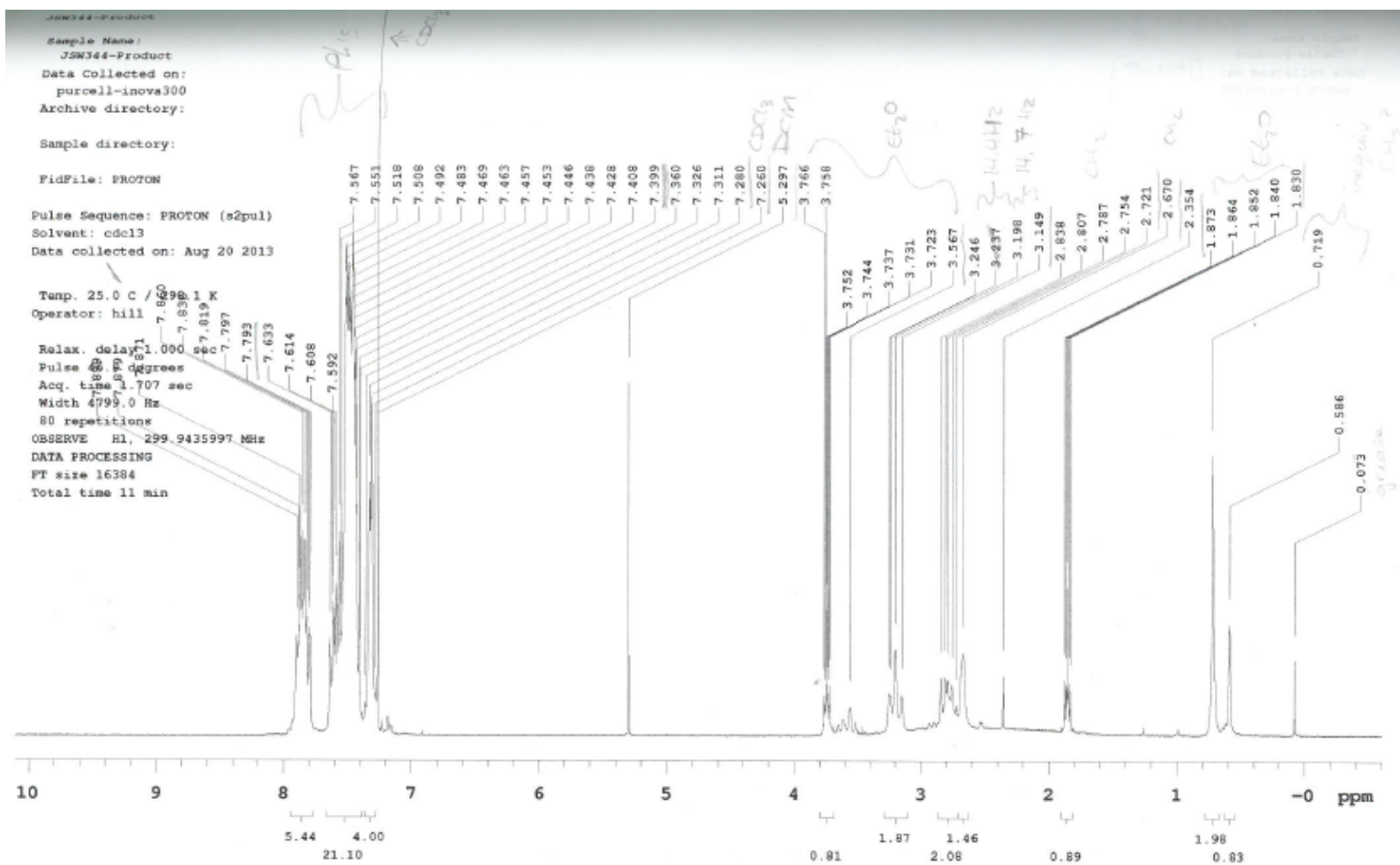
Compound 19 - ^{11}B NMR spectrum



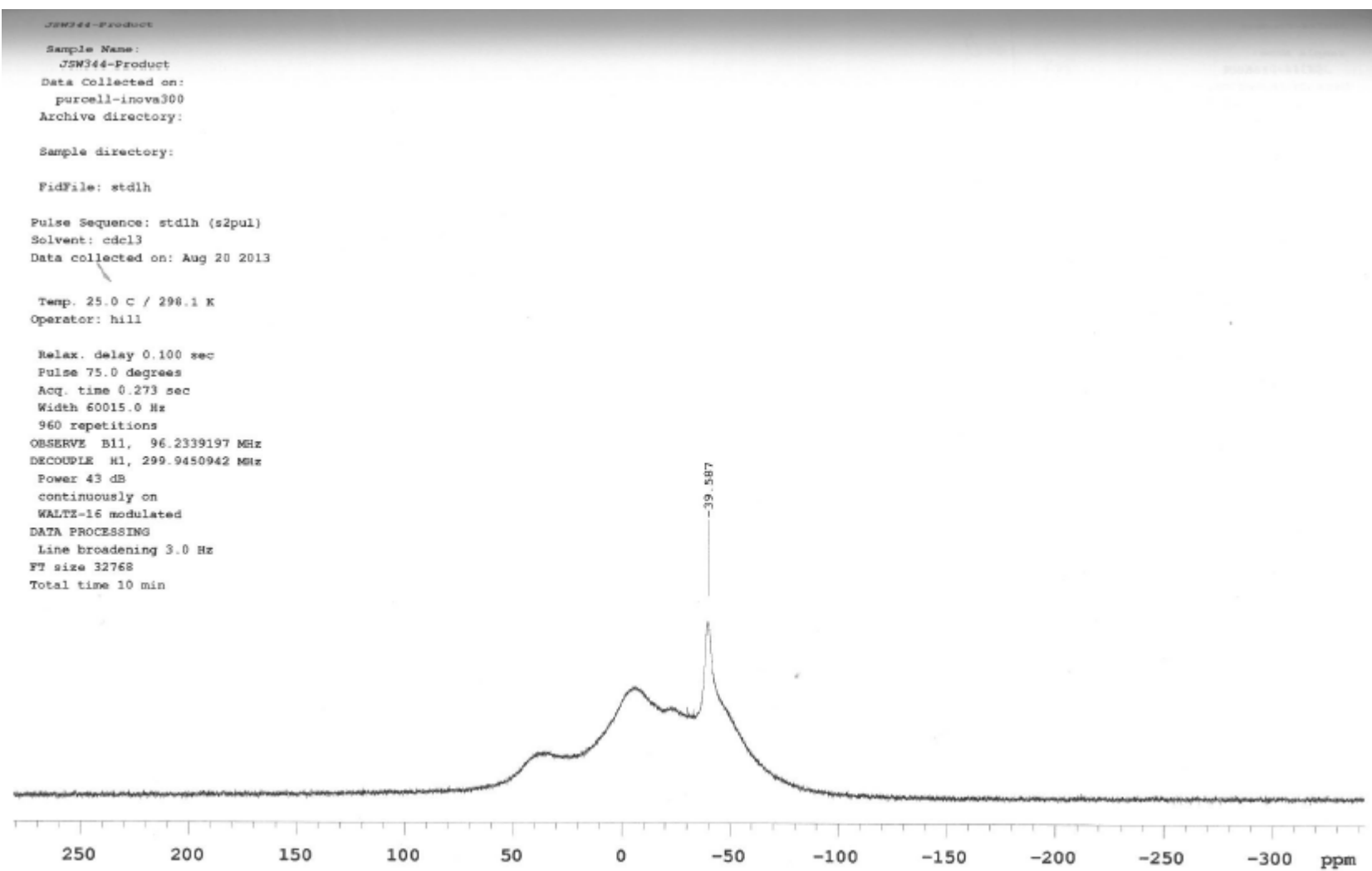
Compound 19 – $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum



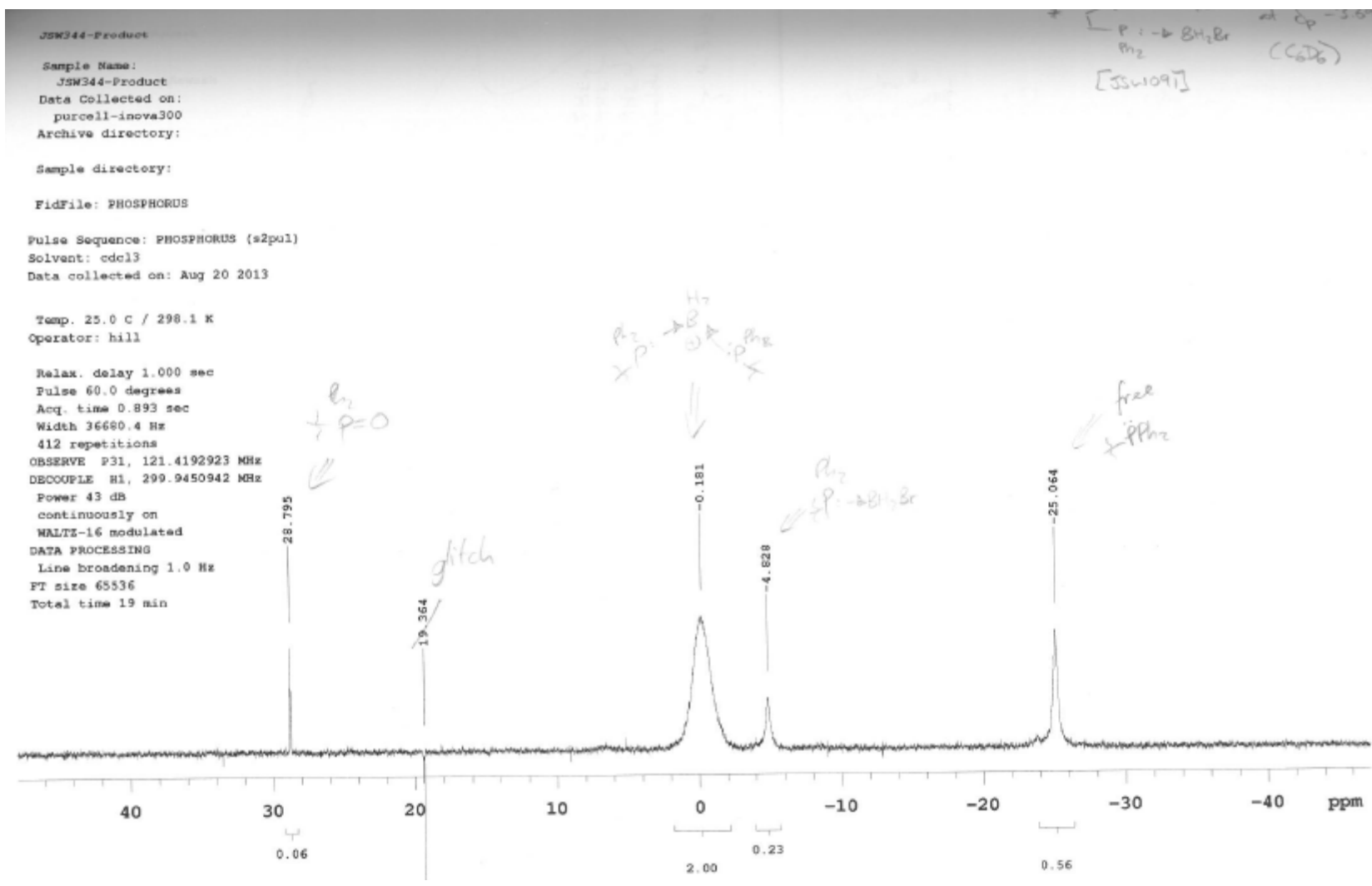
Compound 19 – $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum



Compound 20 - ^1H NMR spectrum



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



Compound 20 - $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum