

Controlled TfOH- or AuCl-catalyzed cycloisomerization and tandem hydrolytic defluorination of 2,3-allenyl perfluoroalkyl ketones

Can Xue, Xin Huang, Shangze Wu, Chunling Fu *and Shengming Ma*

Laboratory of Molecular Recognition and Synthesis, Department of Chemistry,
Zhejiang University, Hangzhou 310027, Zhejiang, People's Republic of China

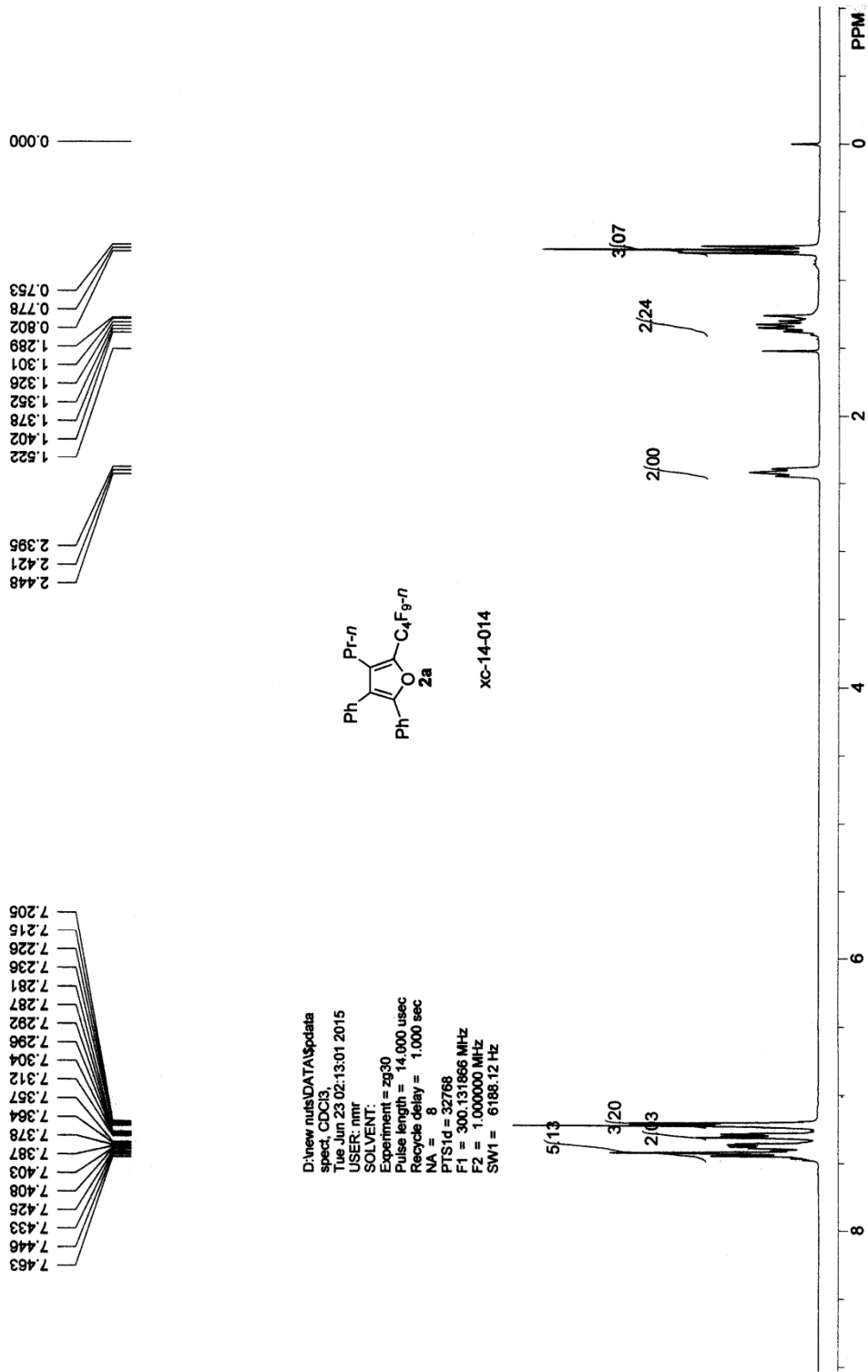
* Corresponding author. Tel.: 86-21-55665687; Fax: 86-21-64167510.

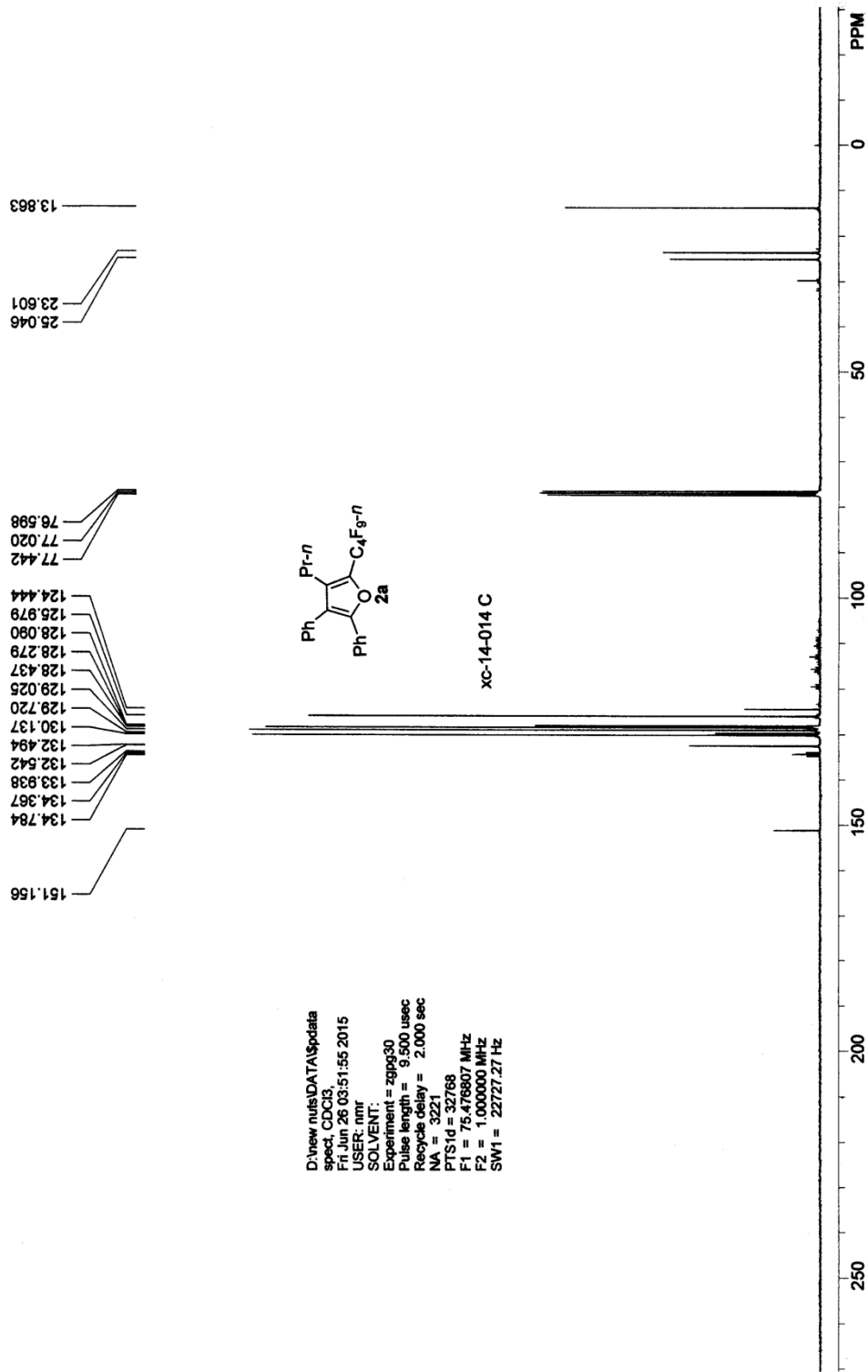
masm@sioc.ac.cn

Supporting Information

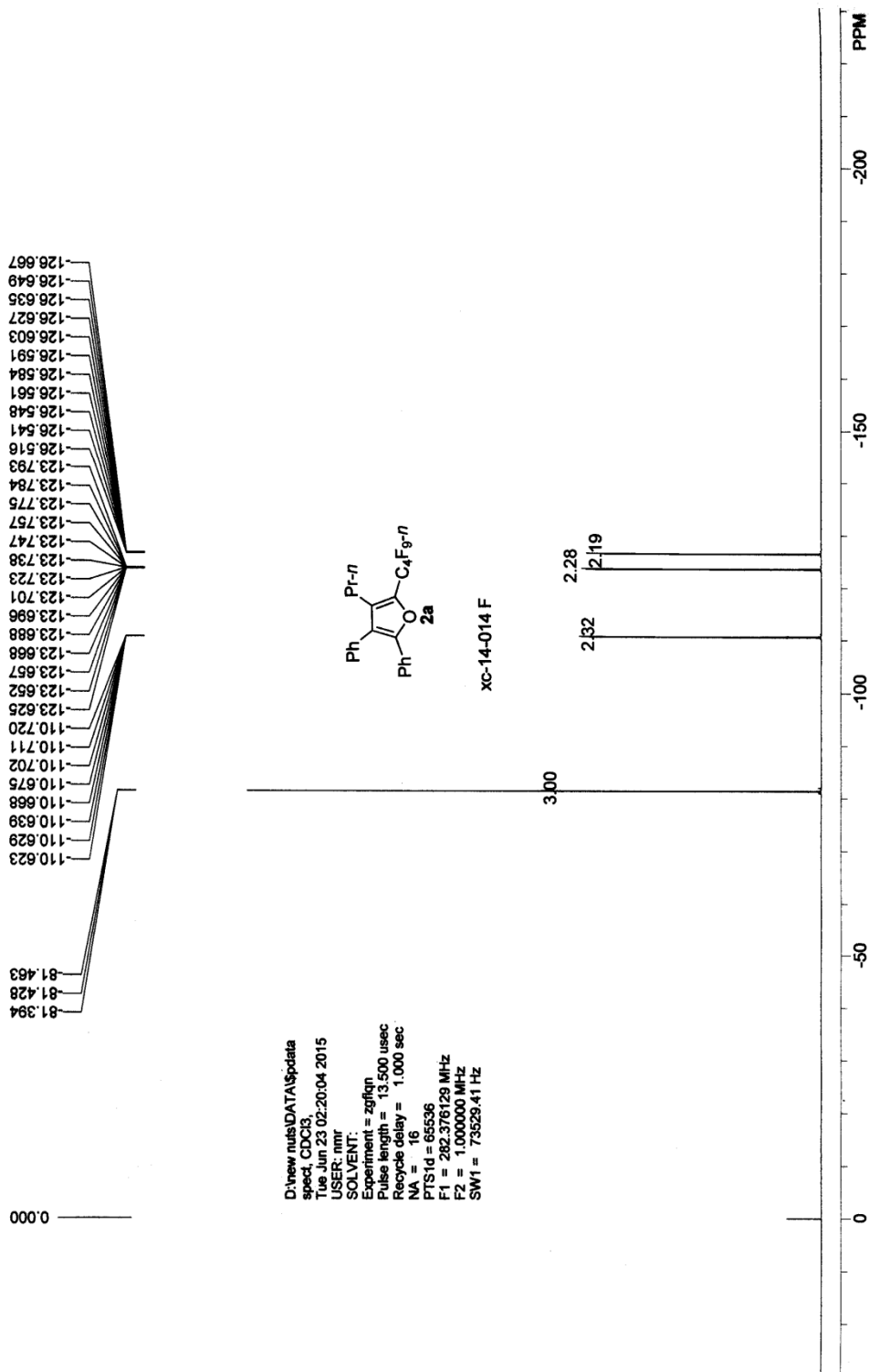
Index

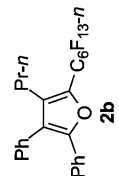
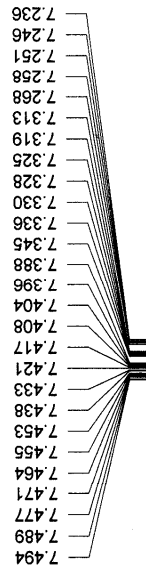
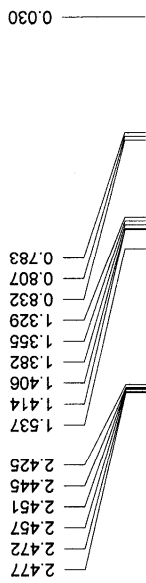
1. ^1H NMR, ^{13}C NMR, ^{19}F NMR and NOE Spectra of these Compounds S1





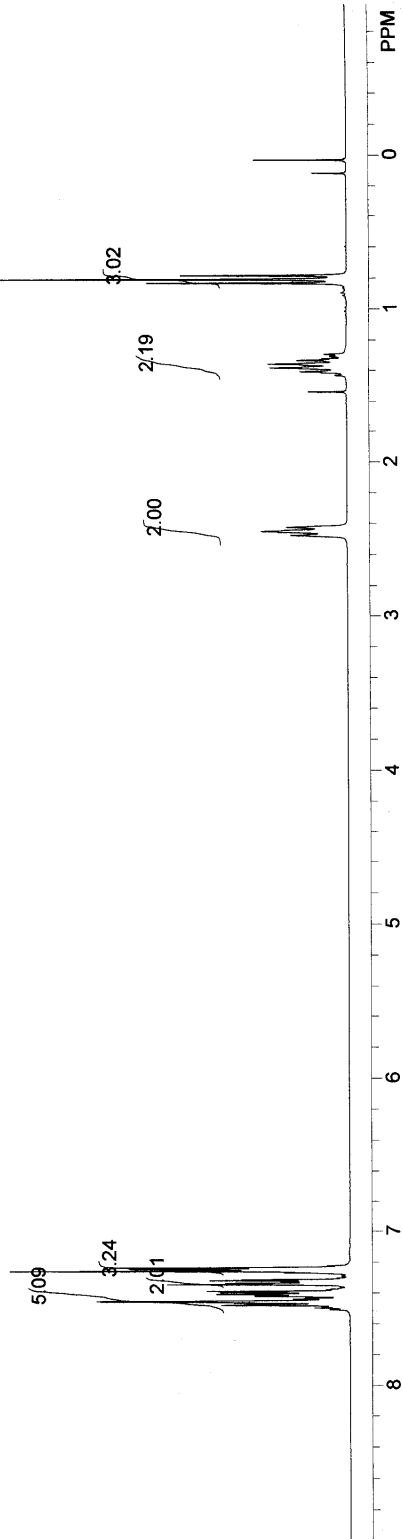
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 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

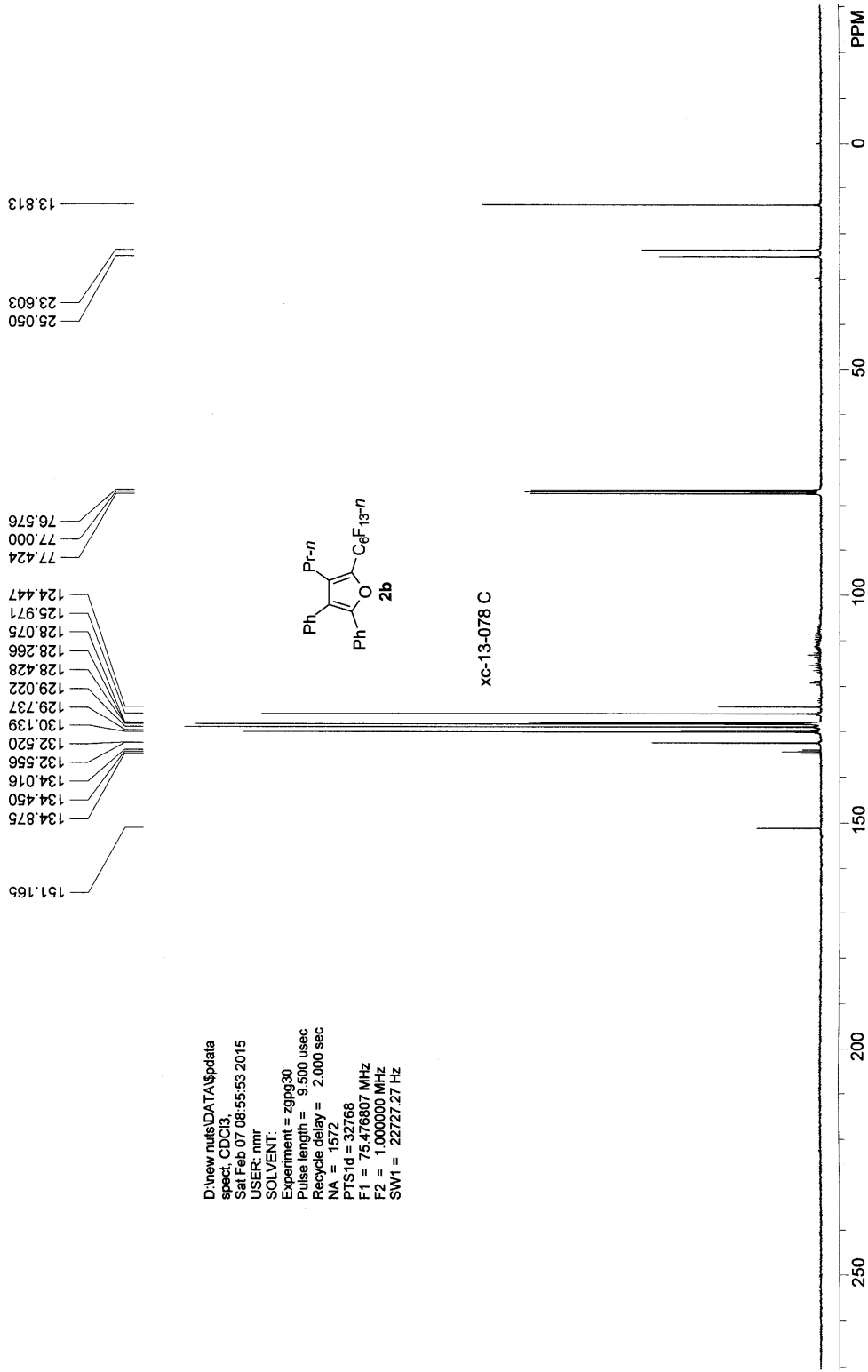




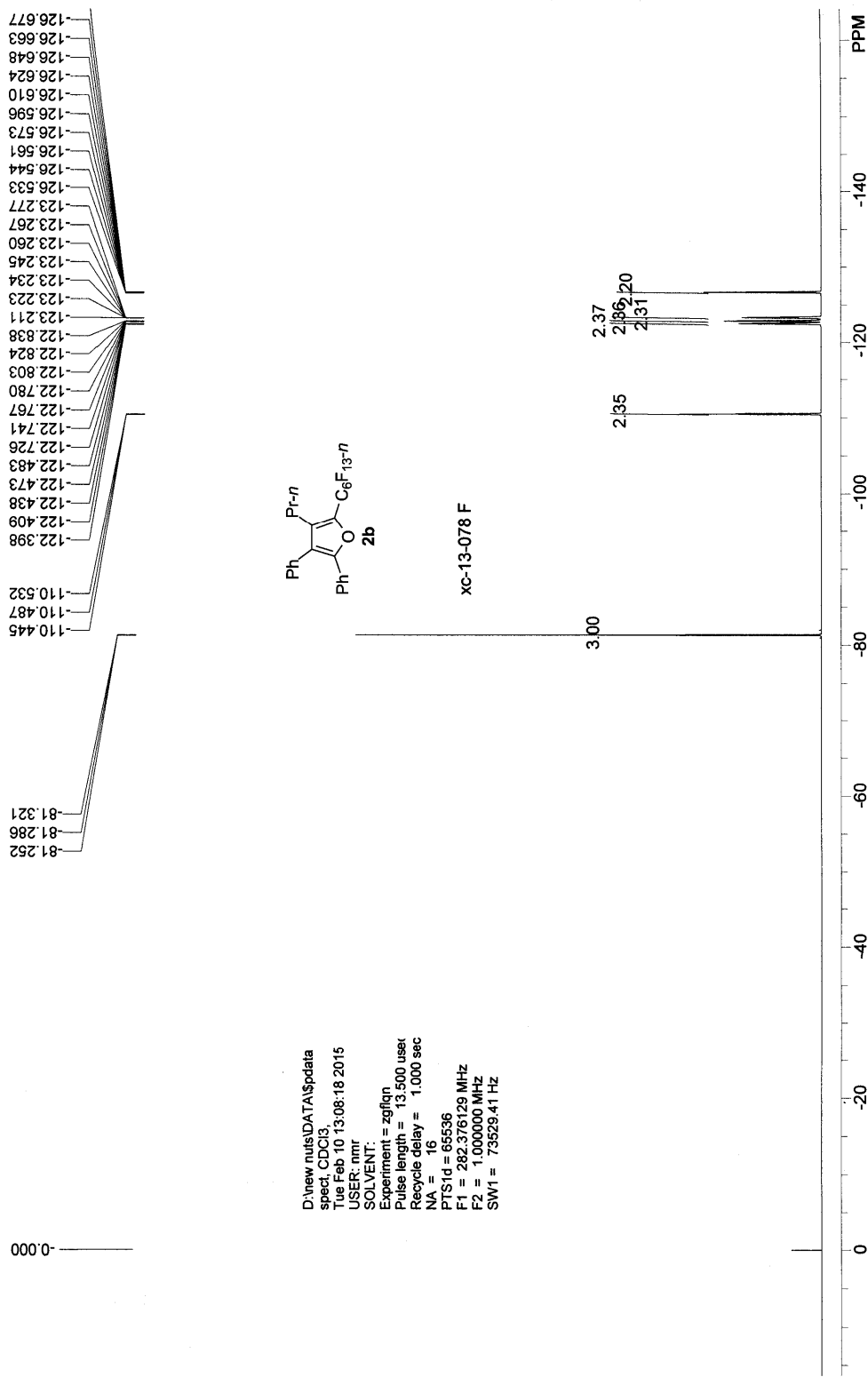
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 spect, CDCI3,
 Tue Feb 10 13:01:56 2015
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
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 NA = 8
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 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SW1 = 6188.12 Hz

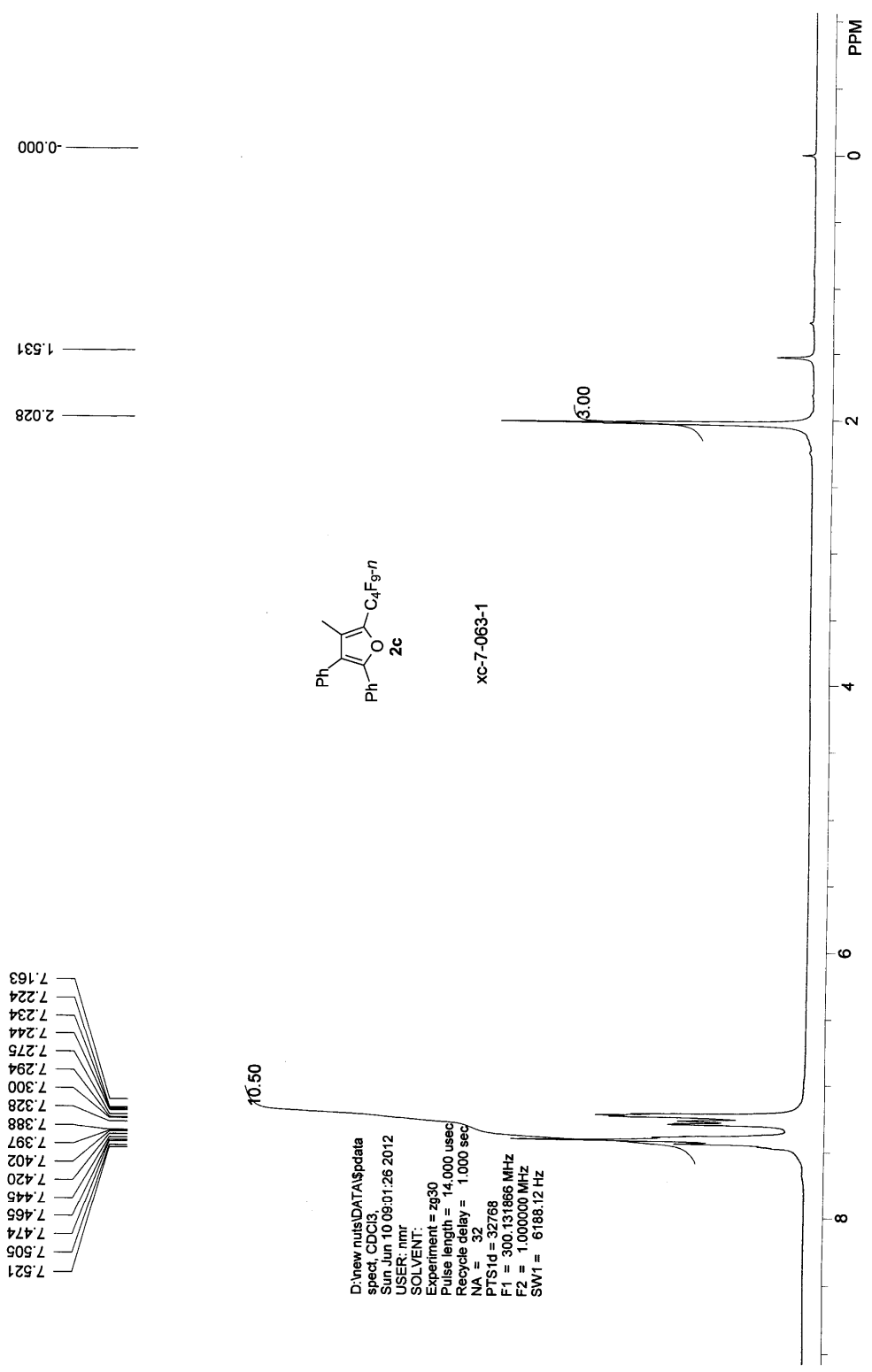
xc-13-078

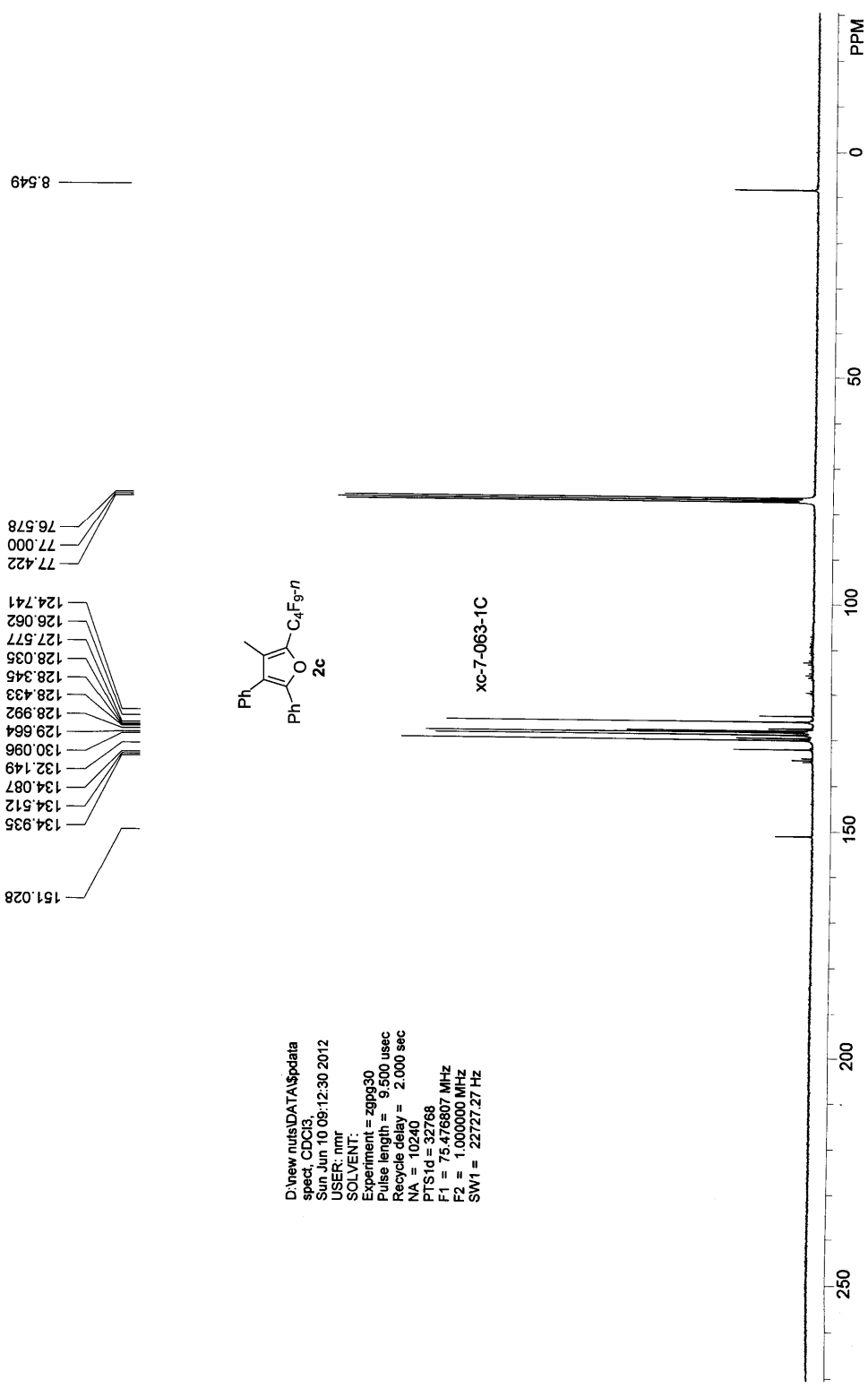




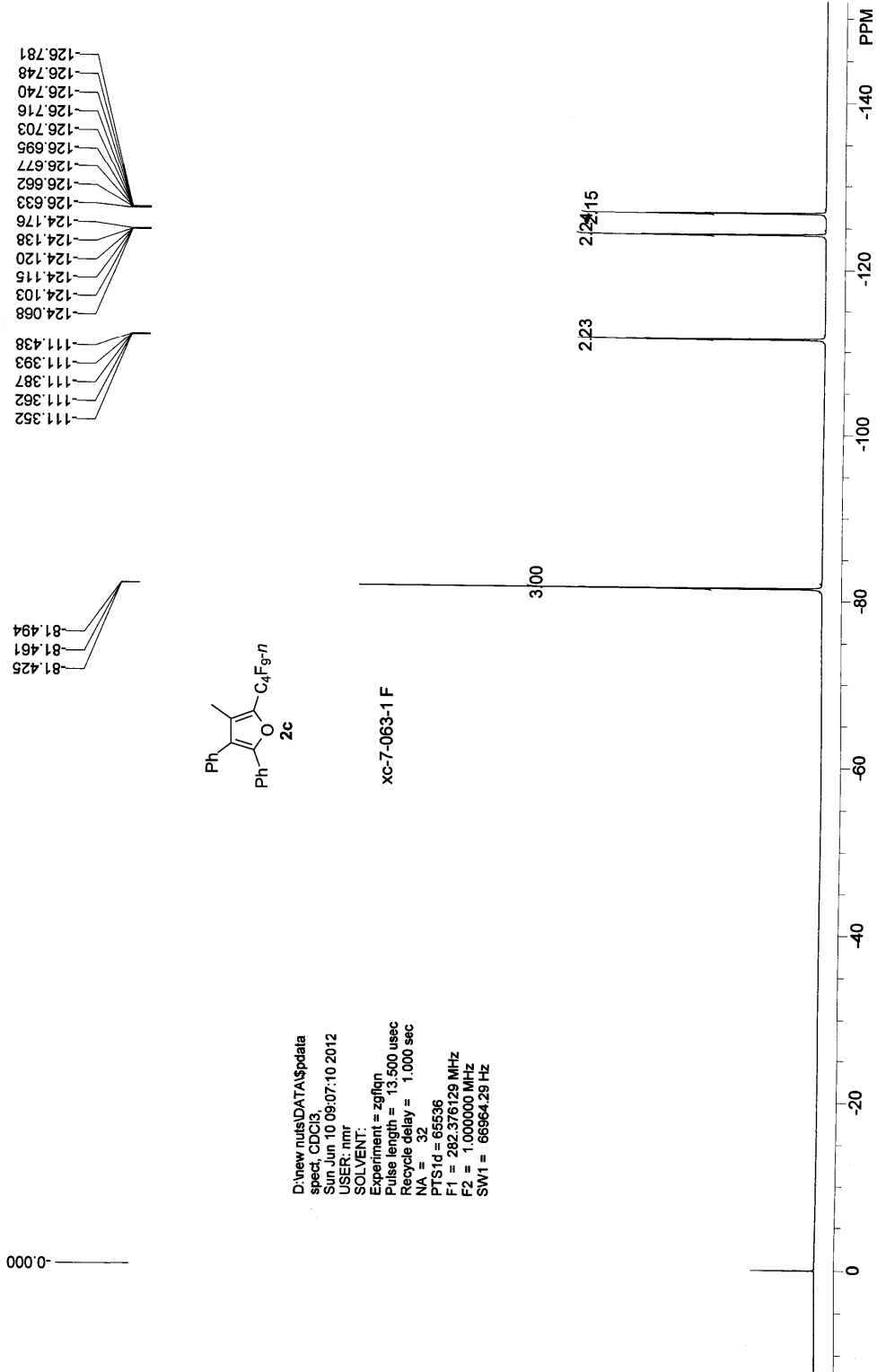
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 spect. CDC13
 Sat Feb 07 08:55:53 2015
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 SW1 = 22727.27 Hz

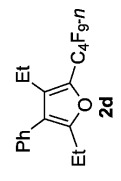
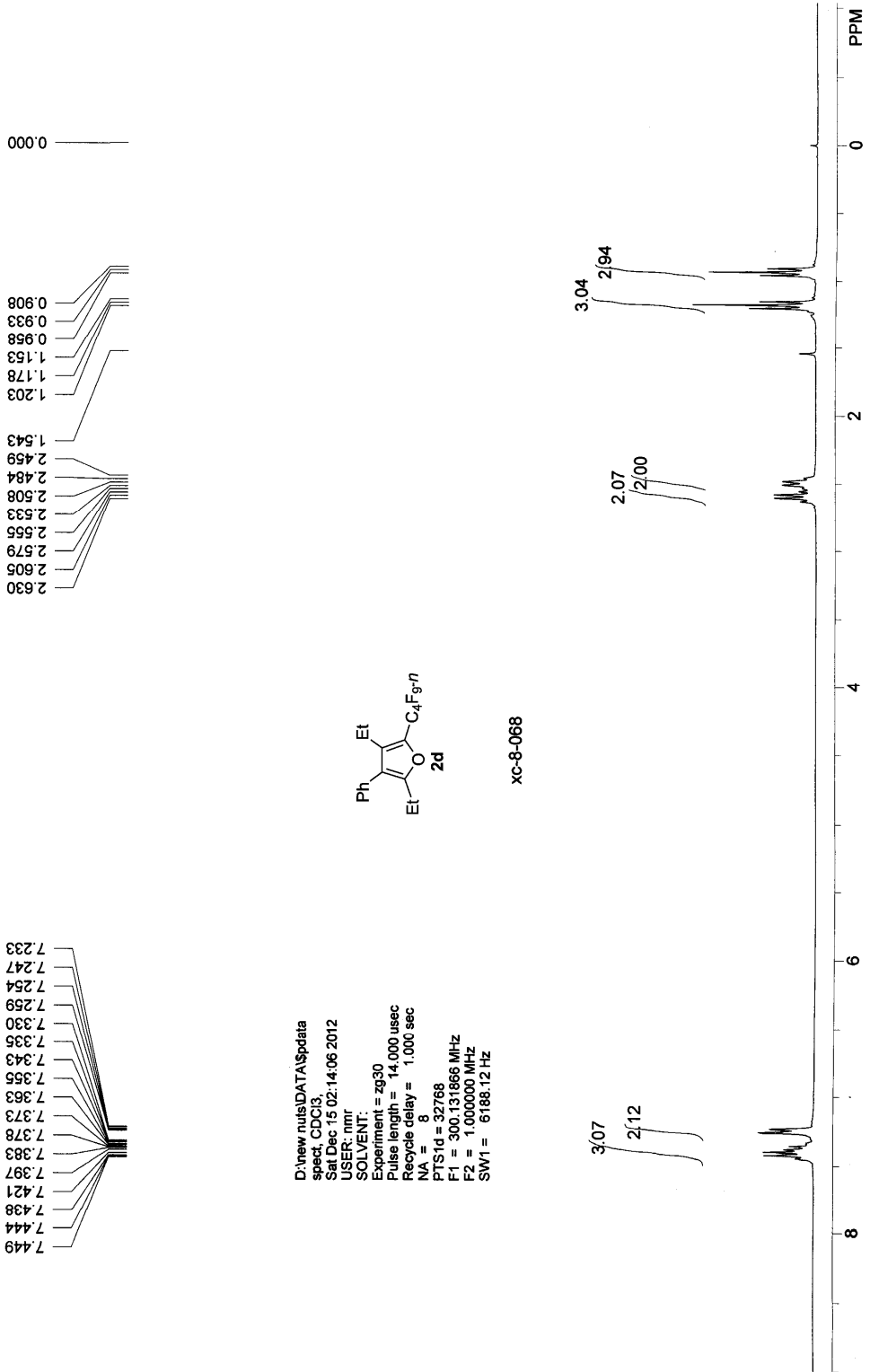






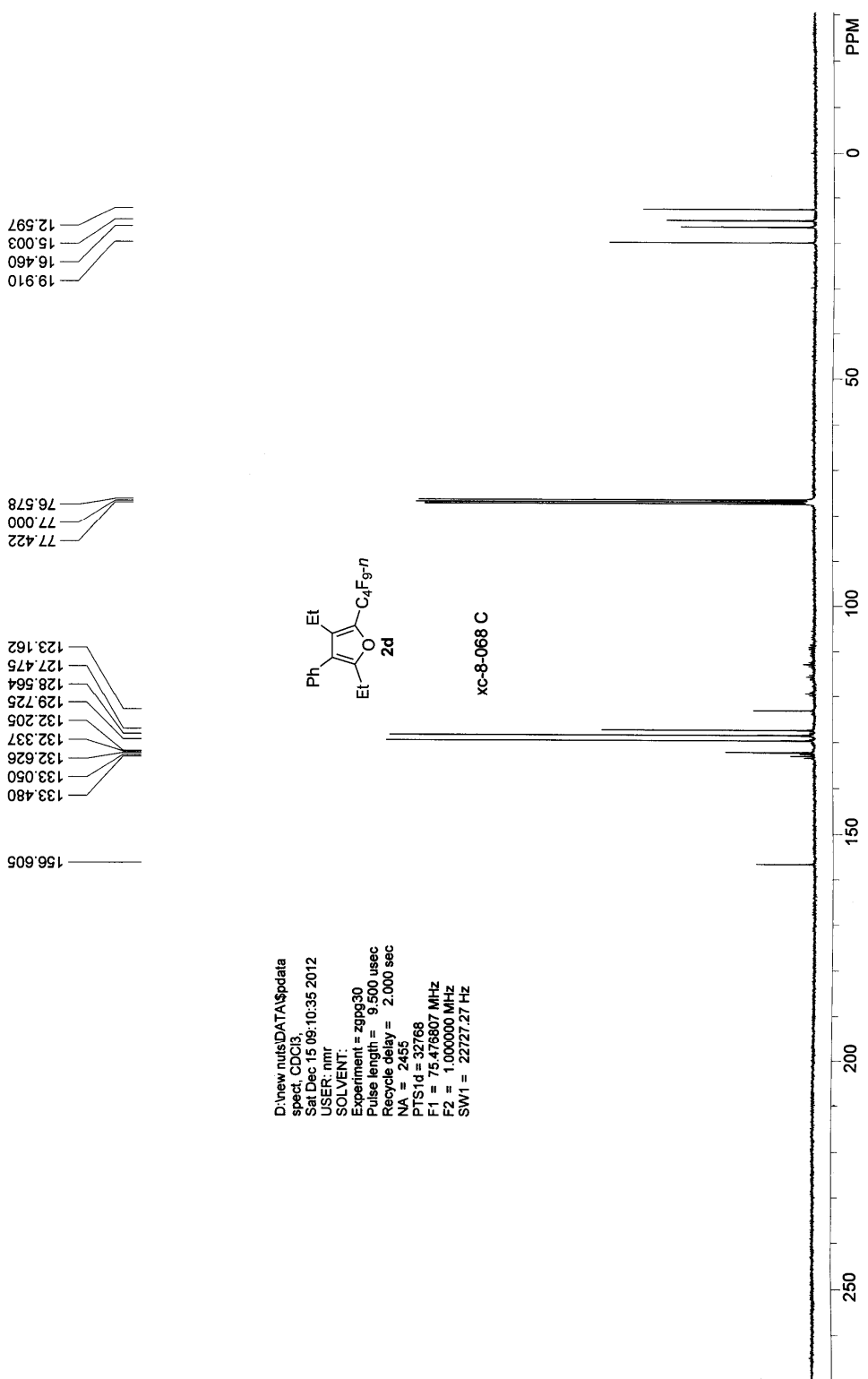
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 SOLVENT:
 Experiment = zppg30
 Pulse length = 9.500 usec
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 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

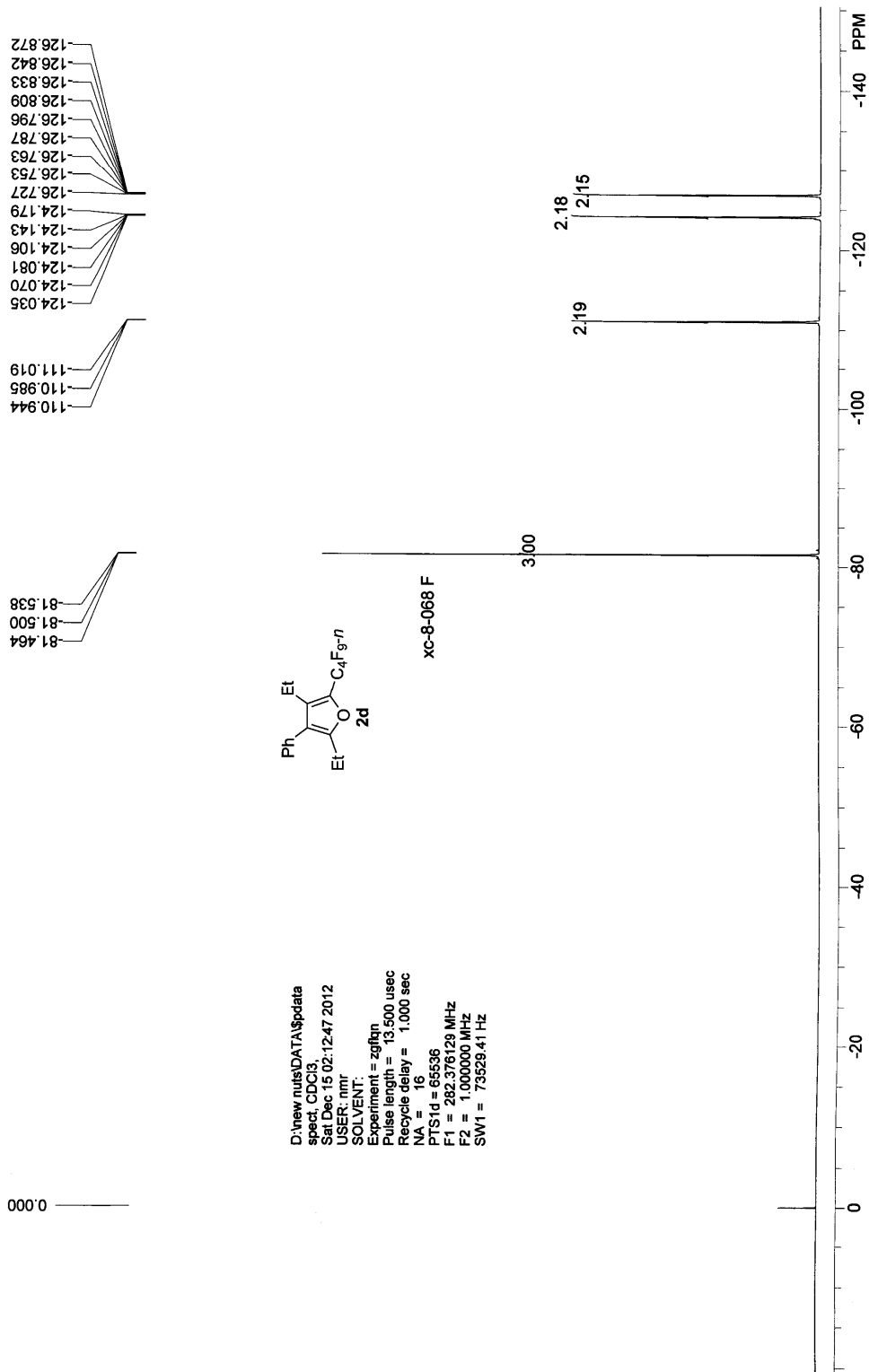




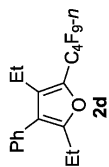
xc-8-068

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 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
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 P1S1d = 32768
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 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

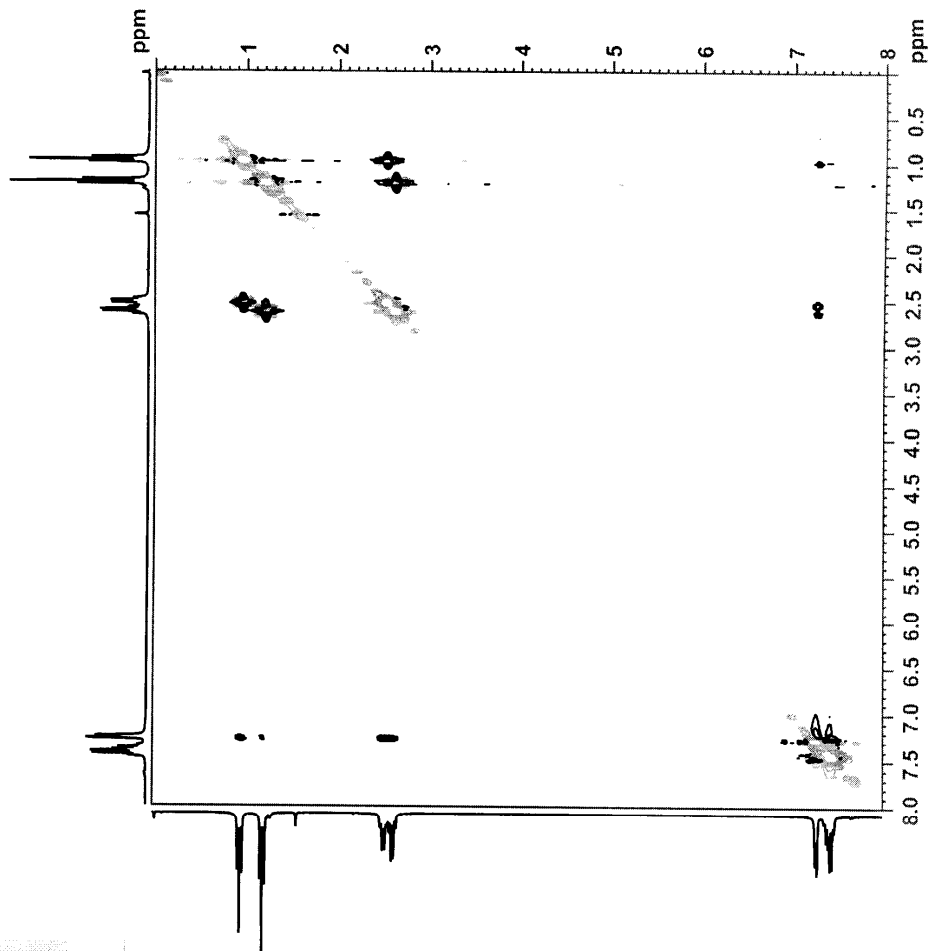


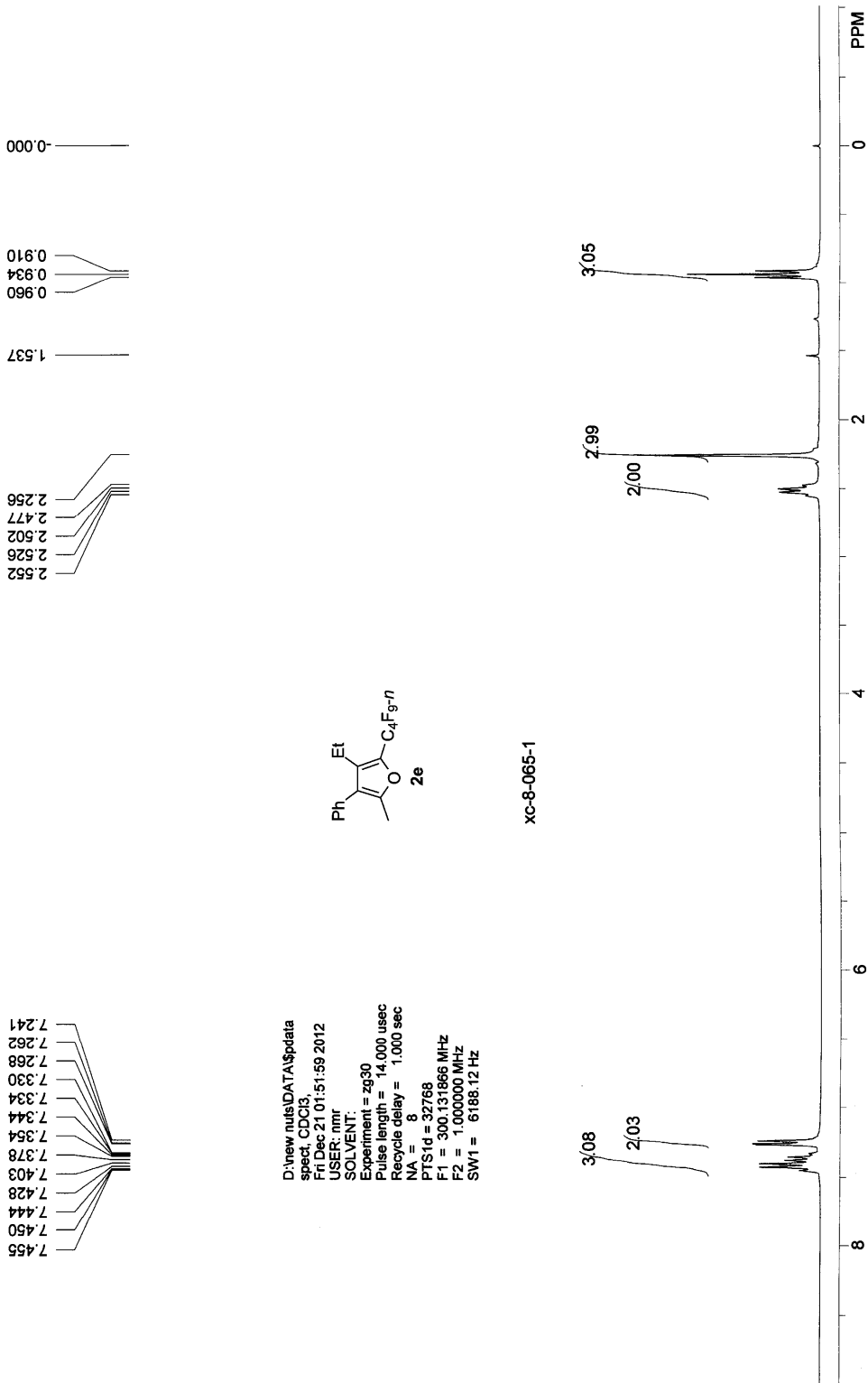


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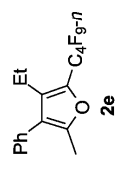


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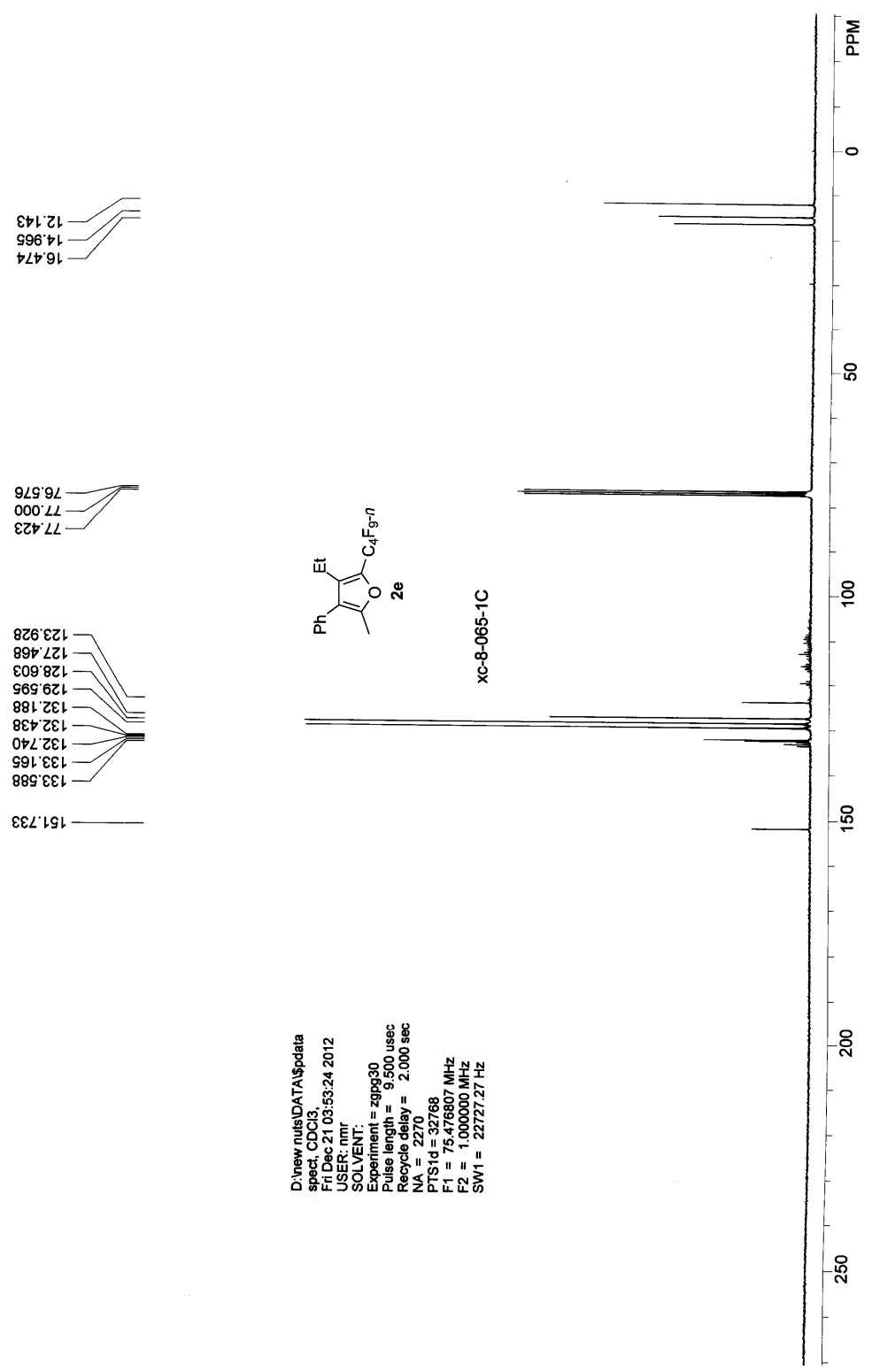


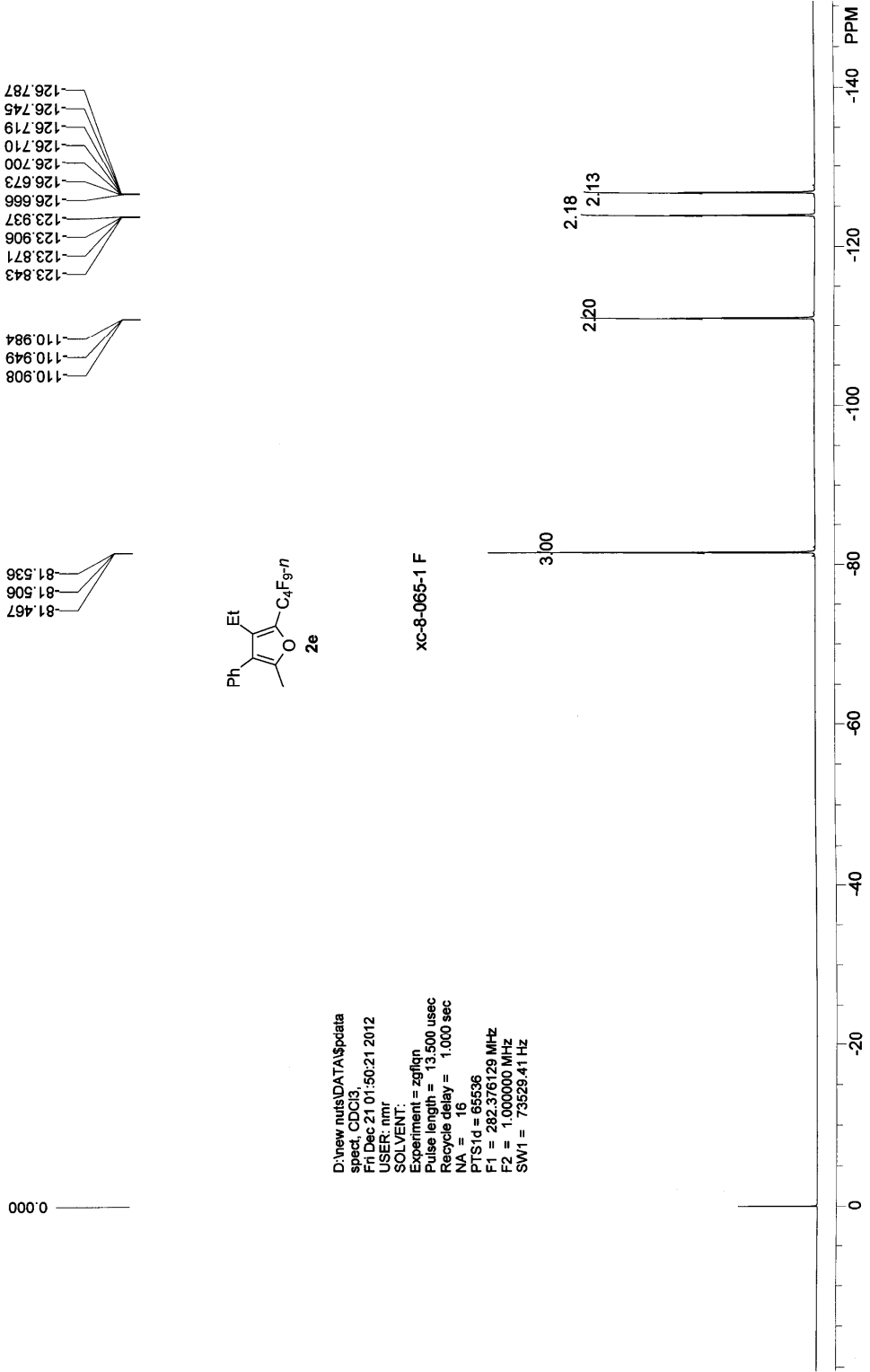


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 Pulse length = 9.500 usec
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 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



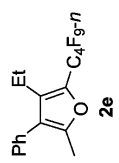
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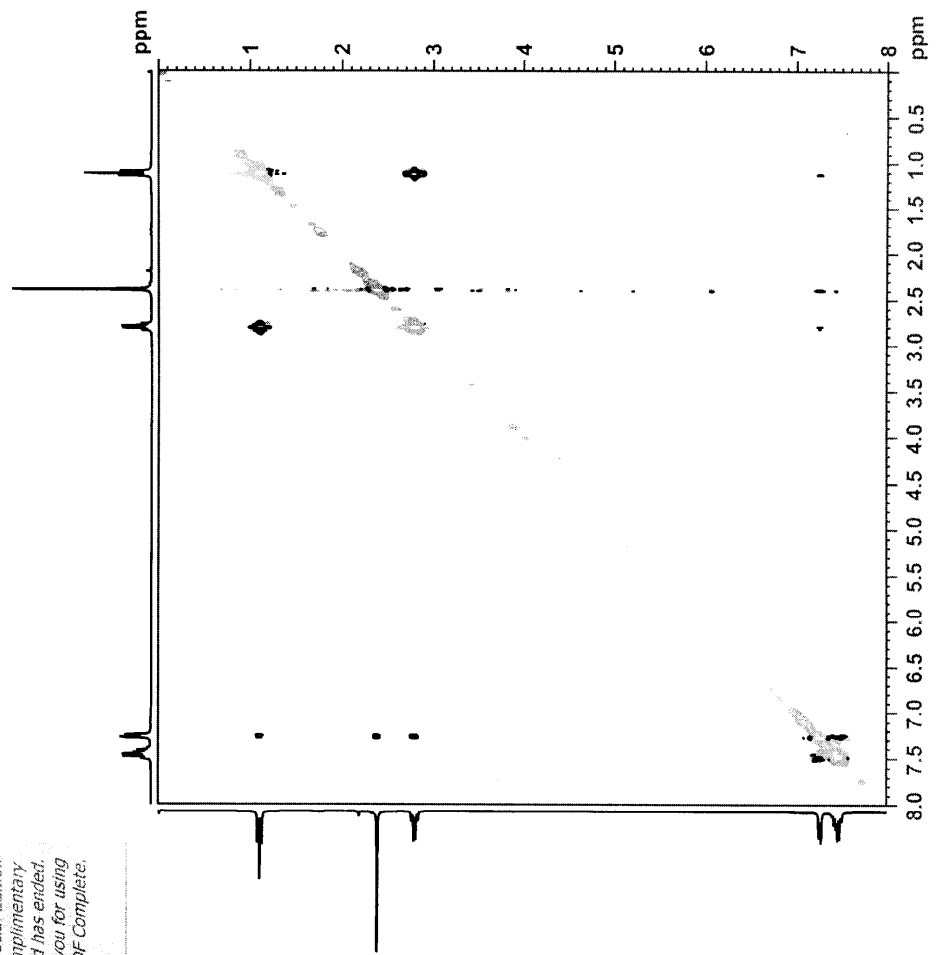


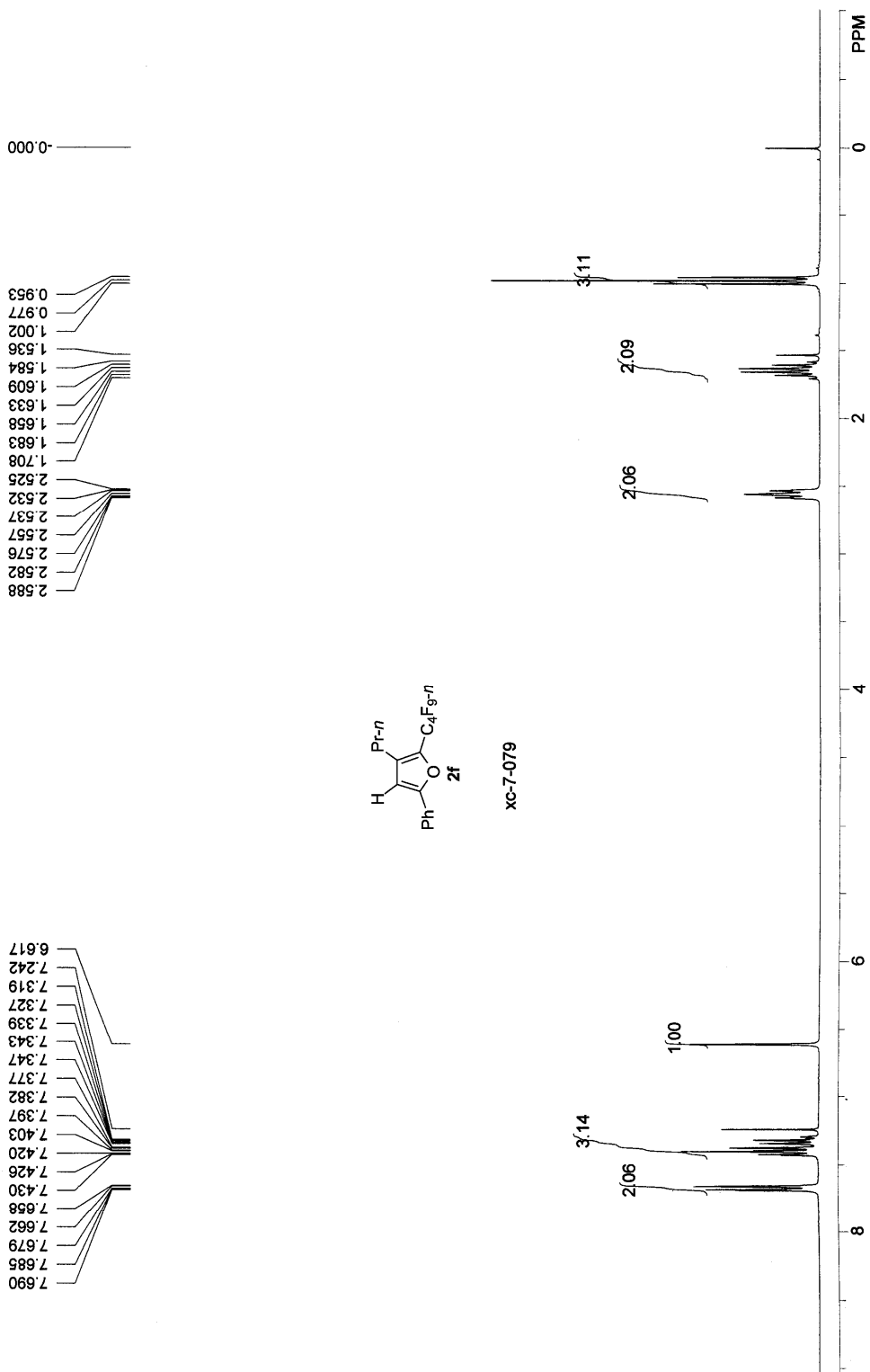
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Experiment = zgpgn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
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F2 = 100.0000 MHz
SWH = 73529.41 HZ

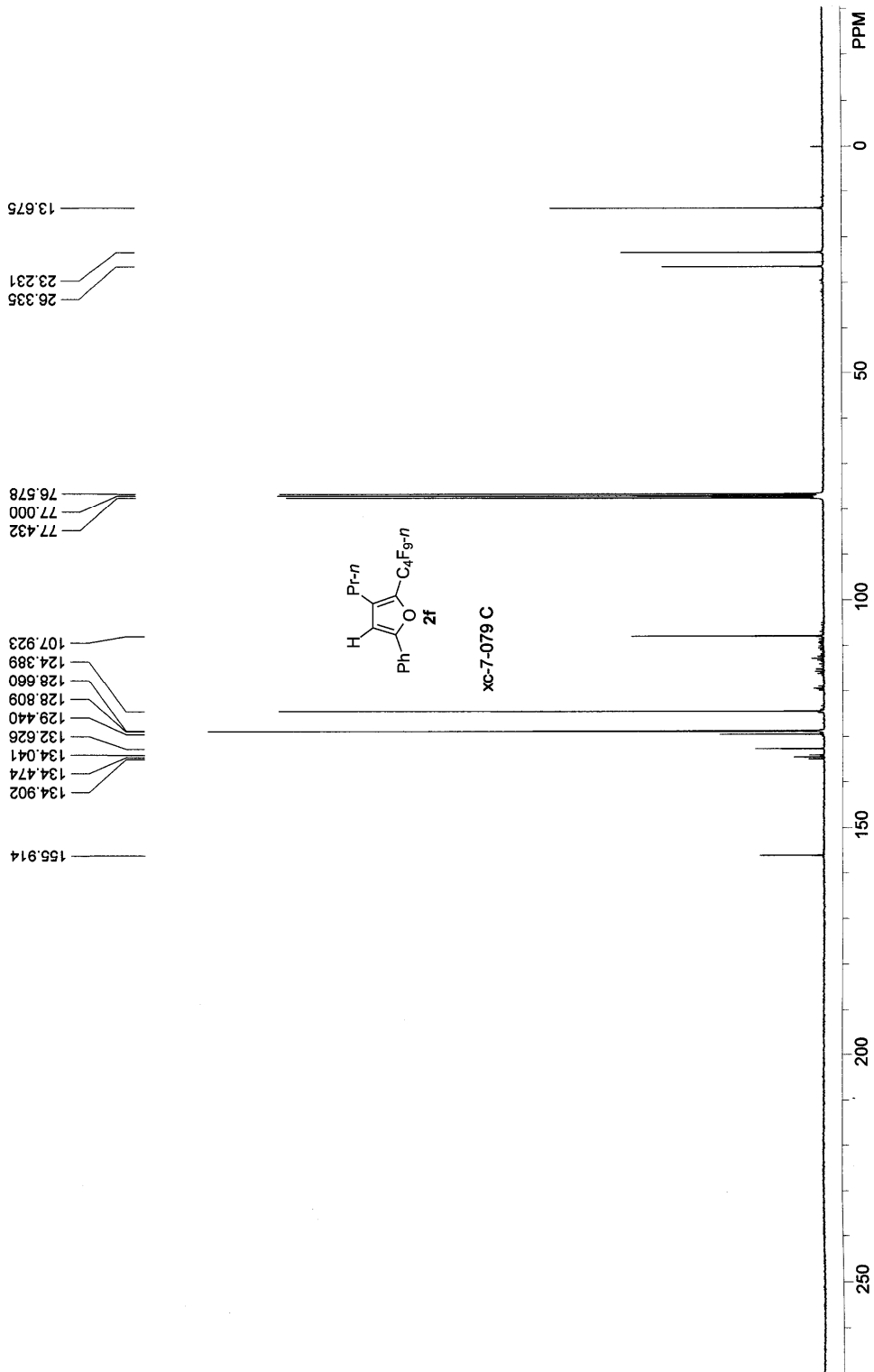
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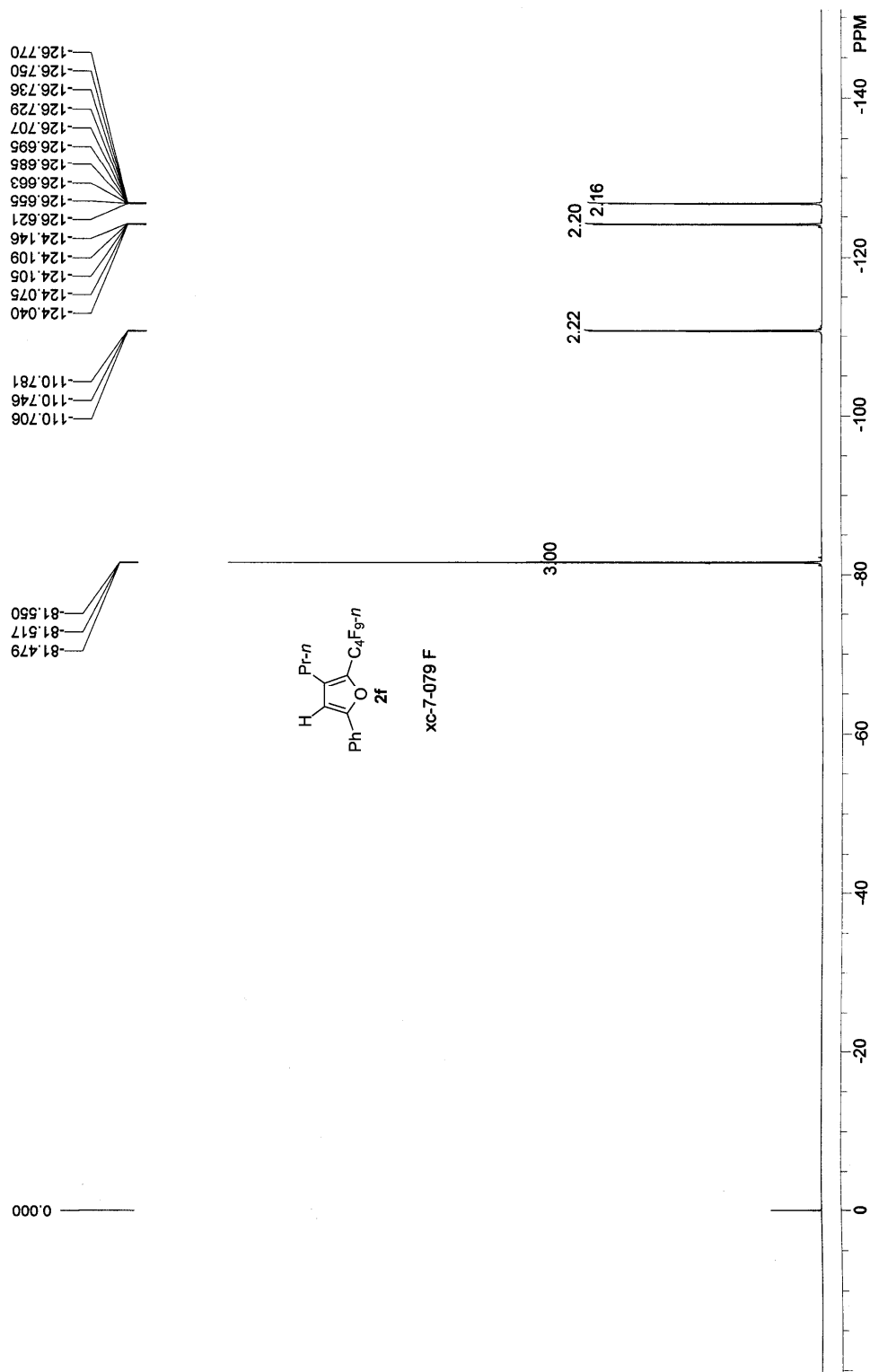


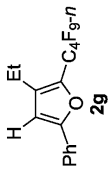
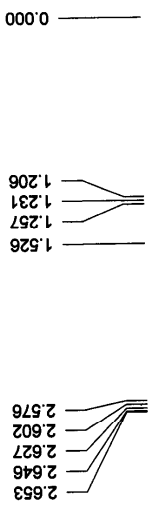
xc-8-065-noe





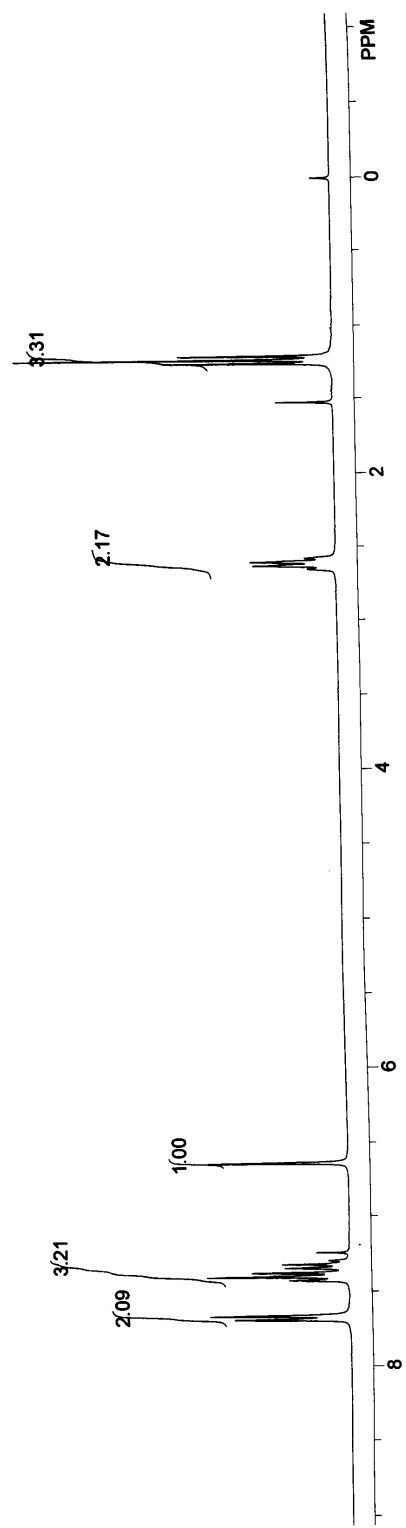
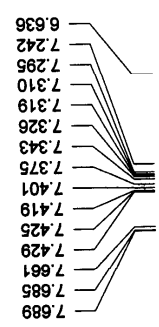


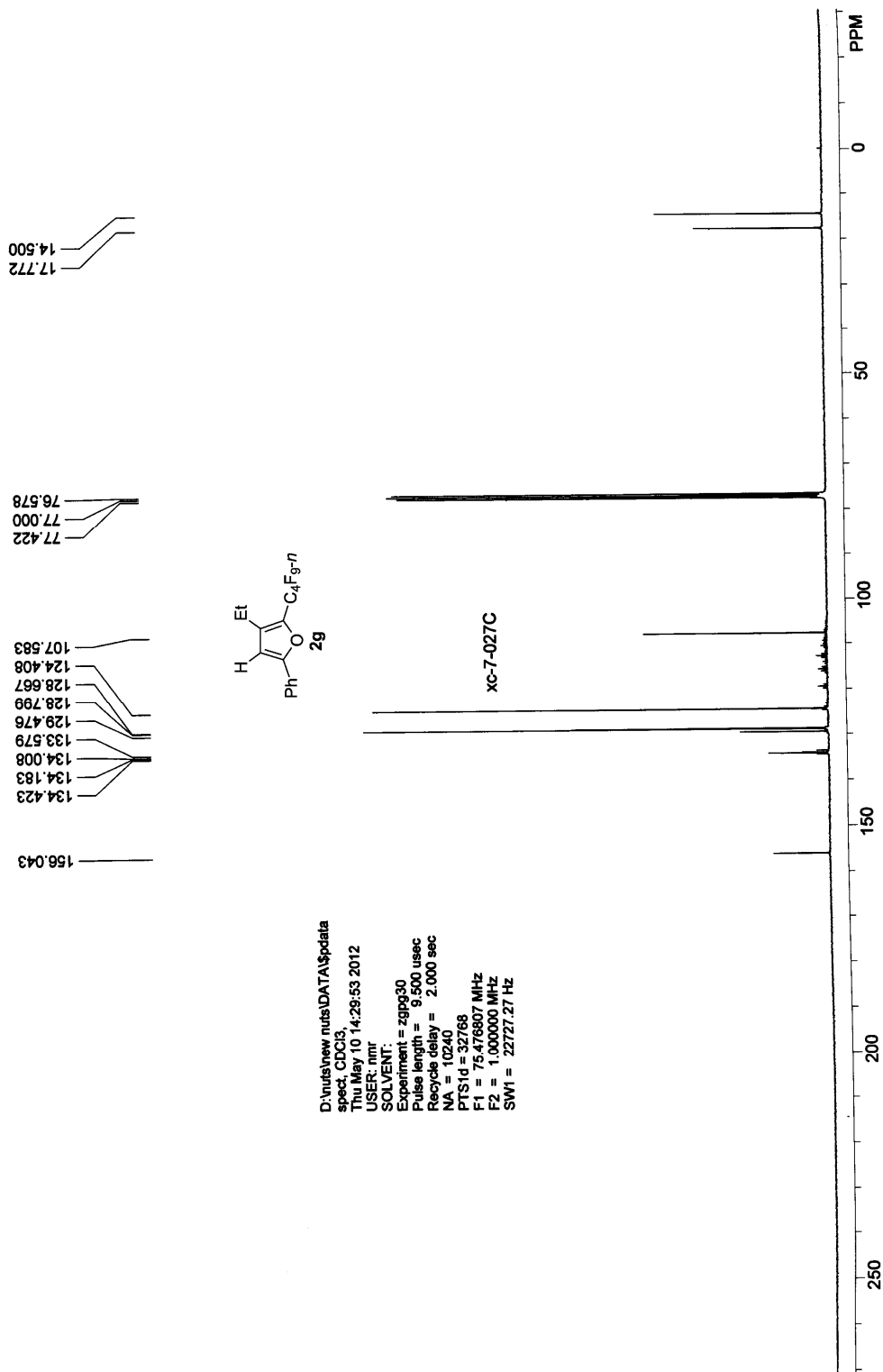


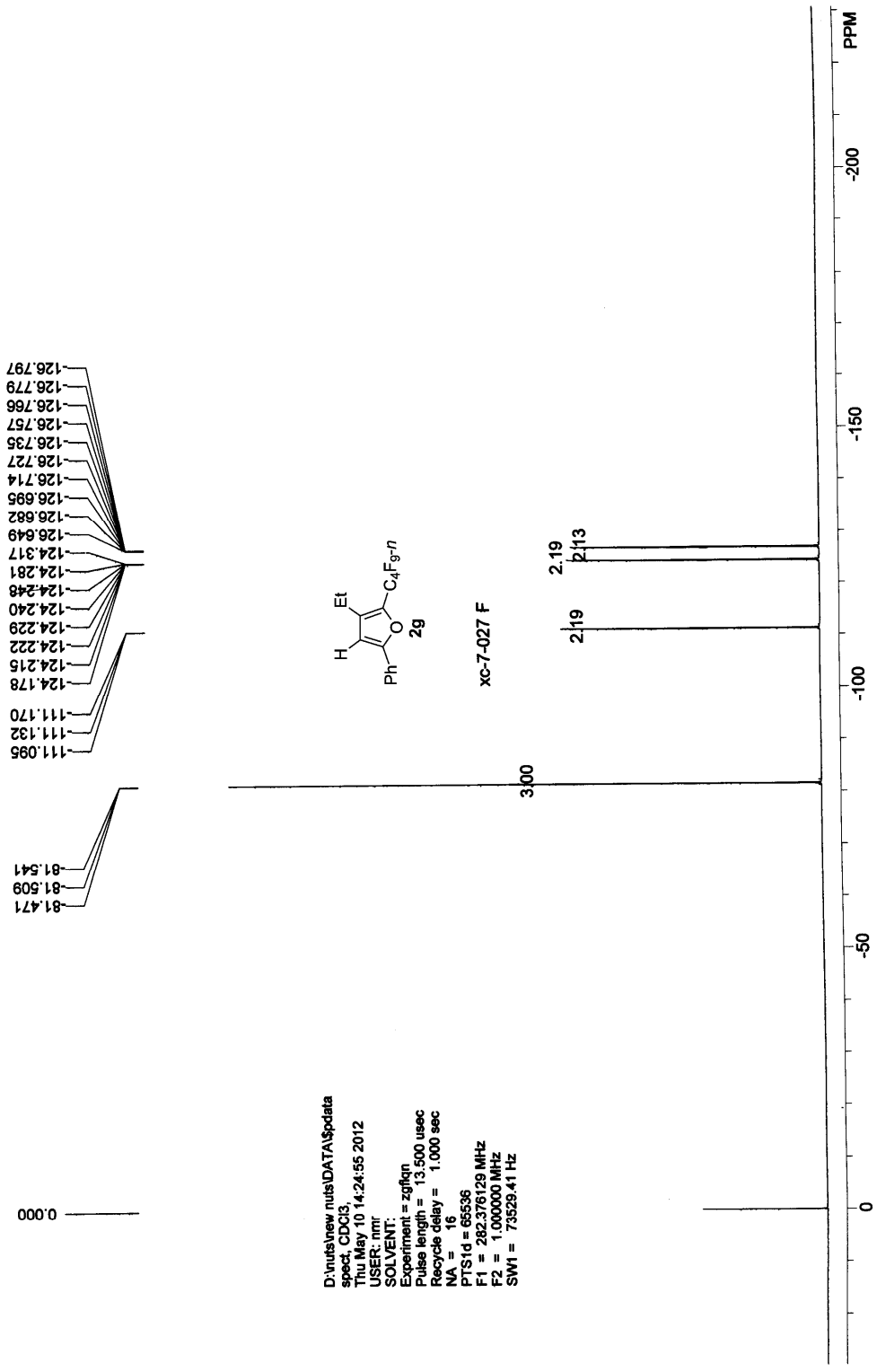


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 SOLVENT: nmr
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
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 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

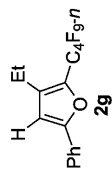
xc-7-027



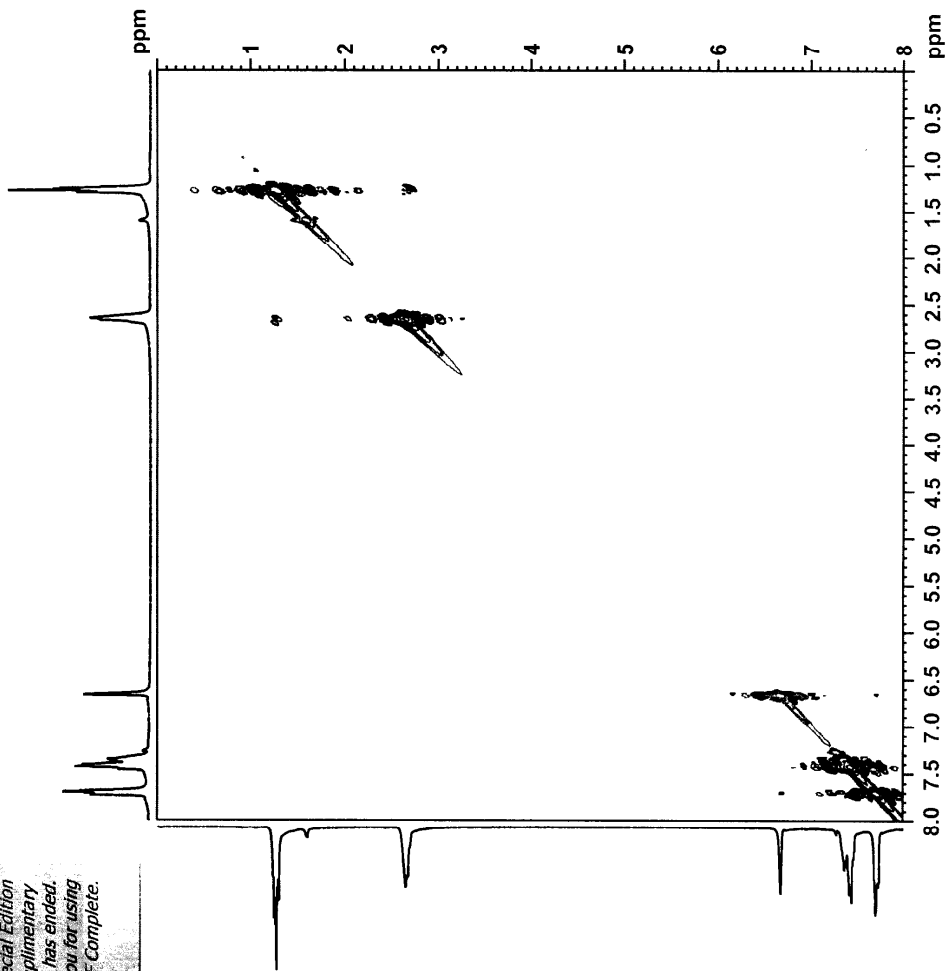


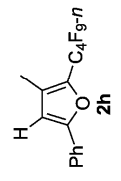
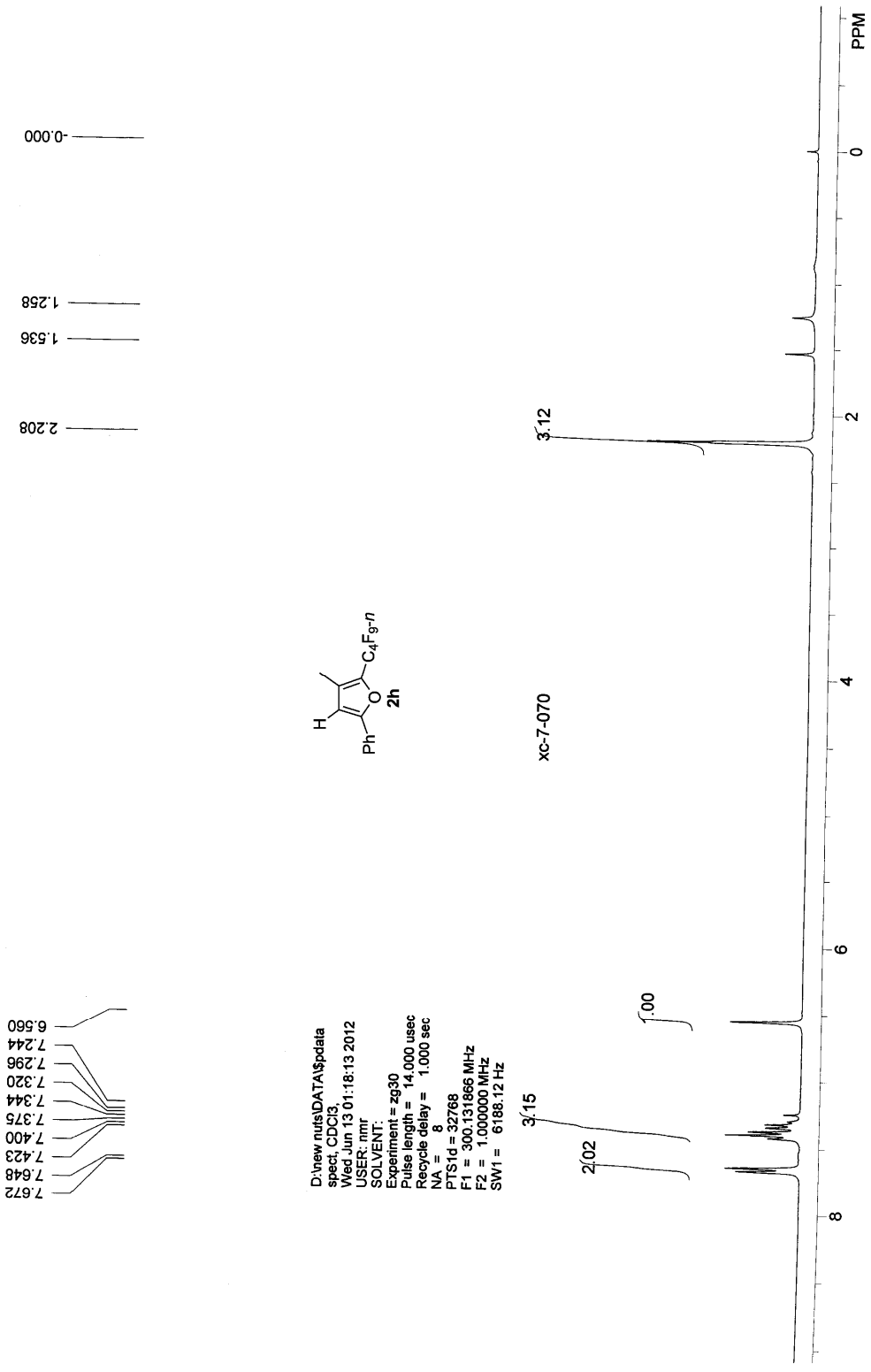


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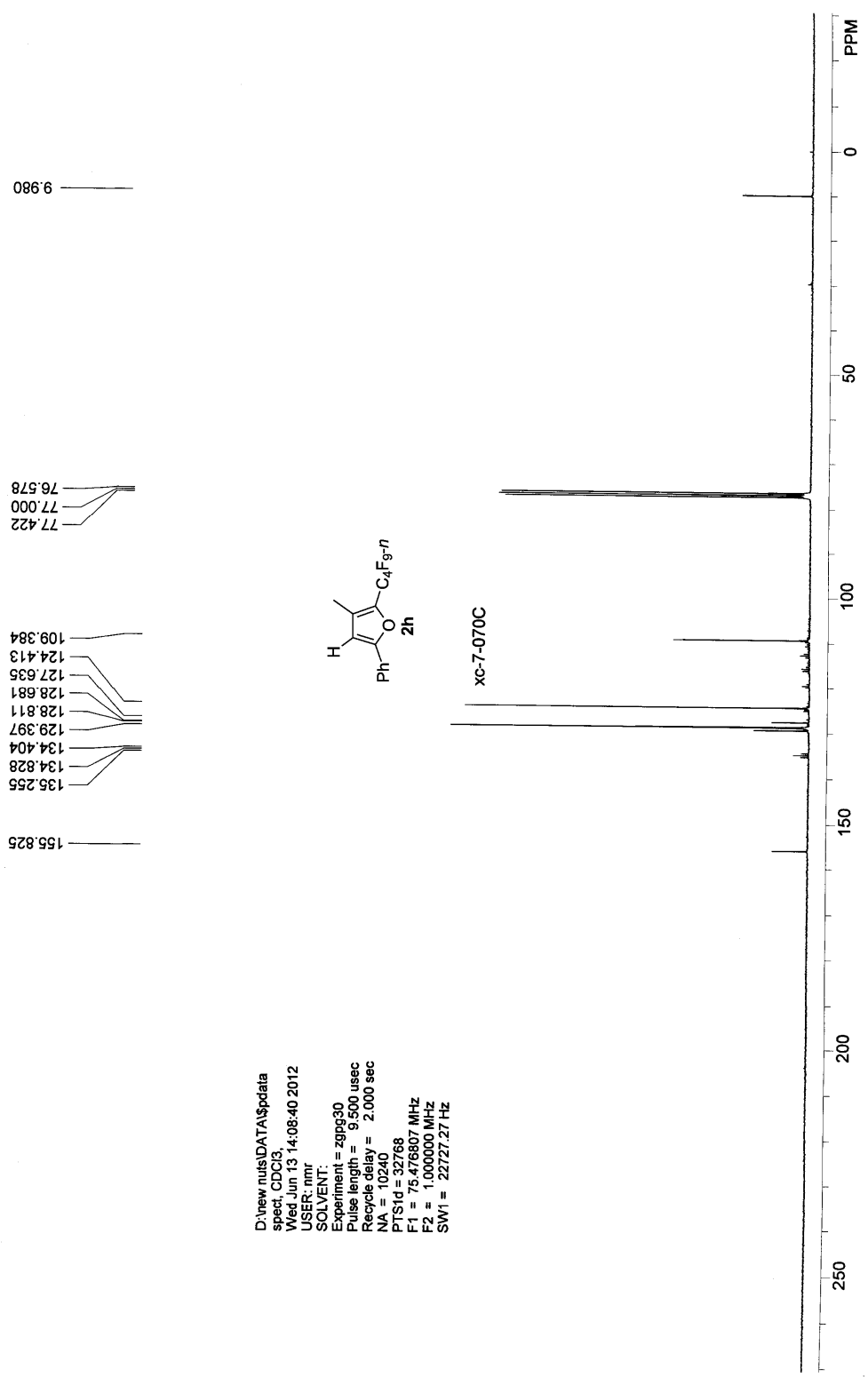
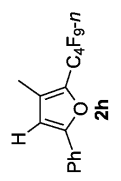
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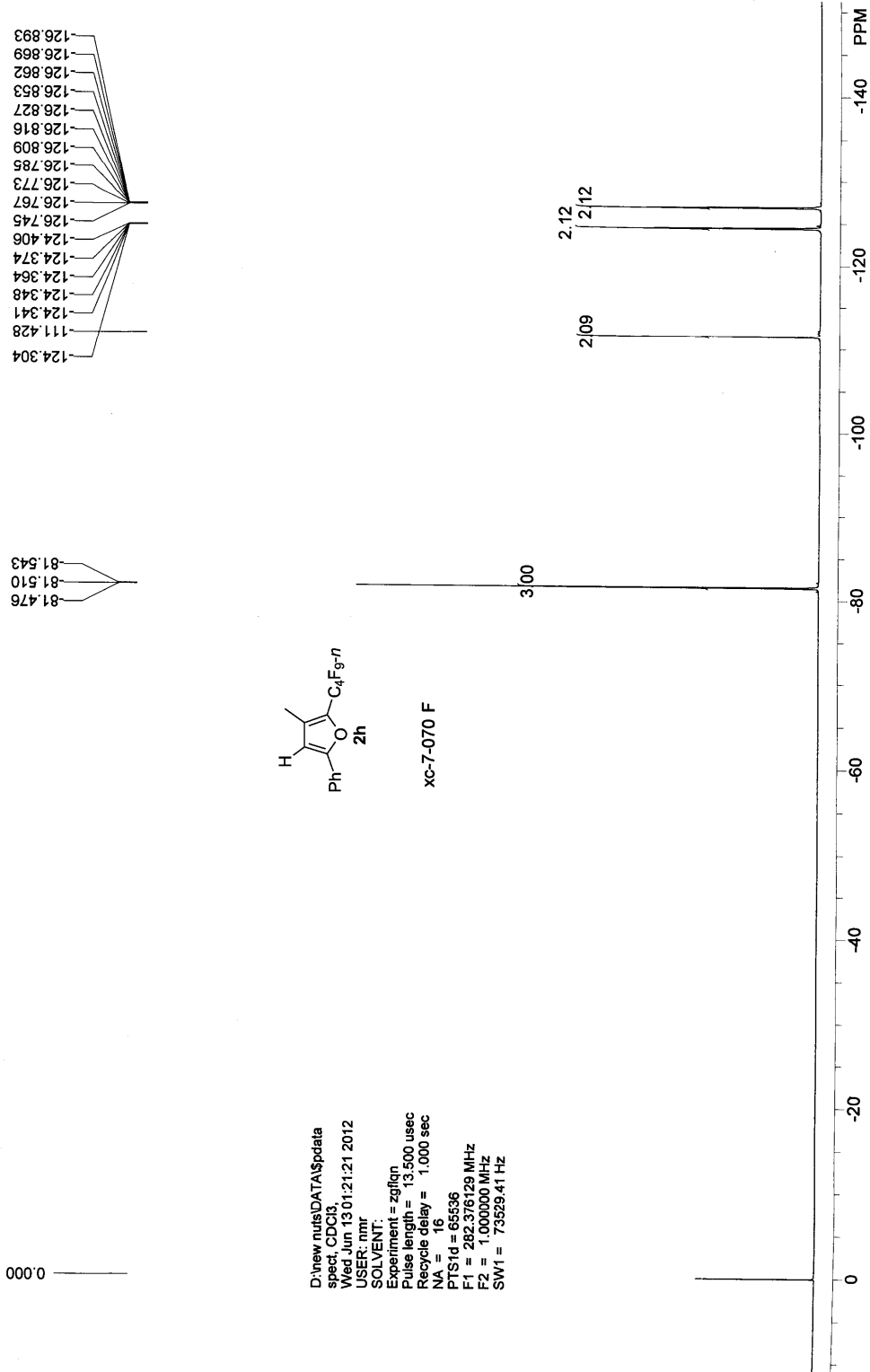


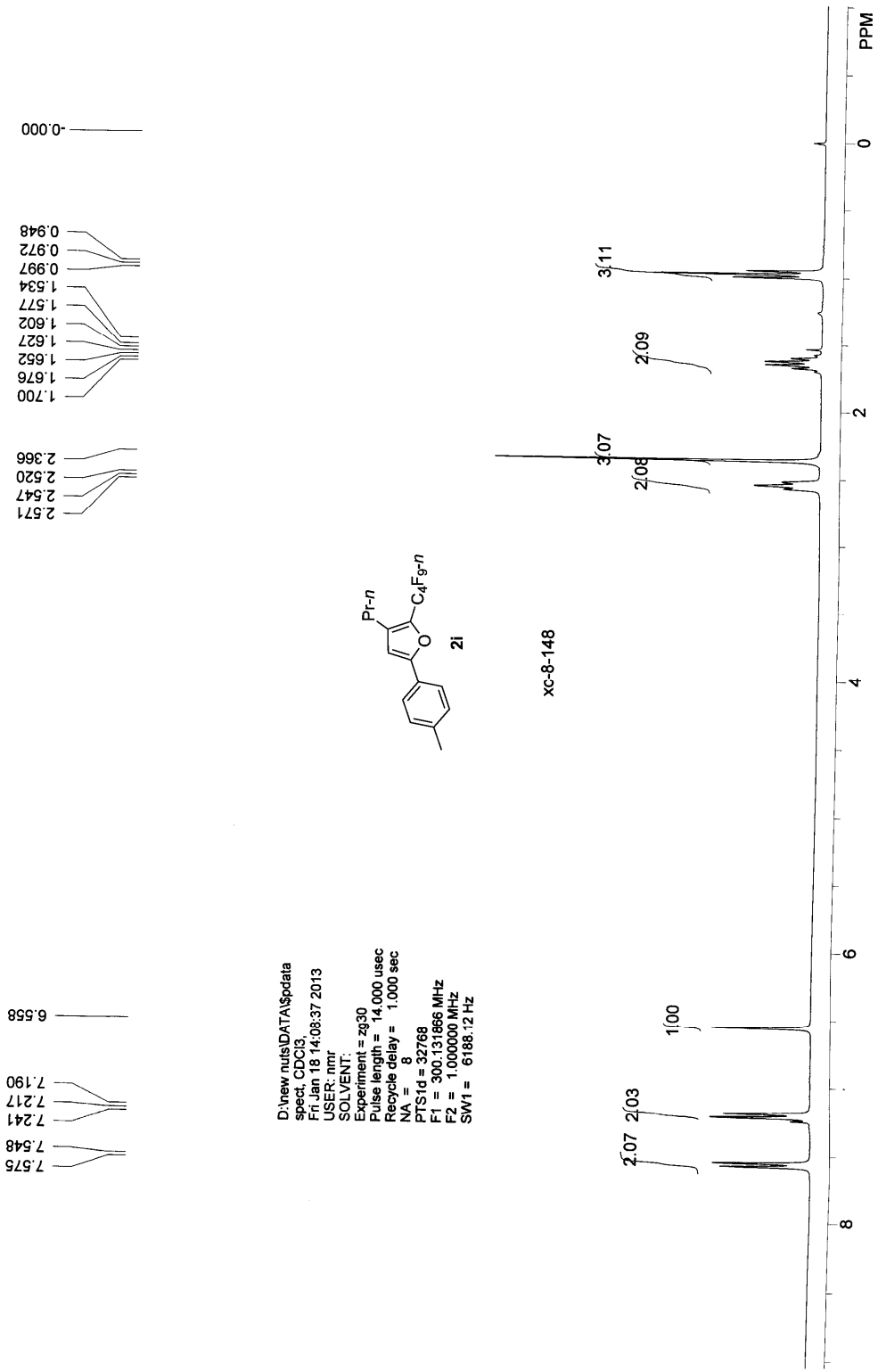


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 spect, CDCI3,
 Wed Jun 13 01:18:13 2012
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTS1a = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

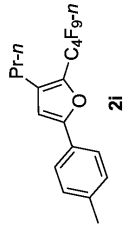
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 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 10240
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.0000000 MHz
 SW1 = 22727.27 Hz

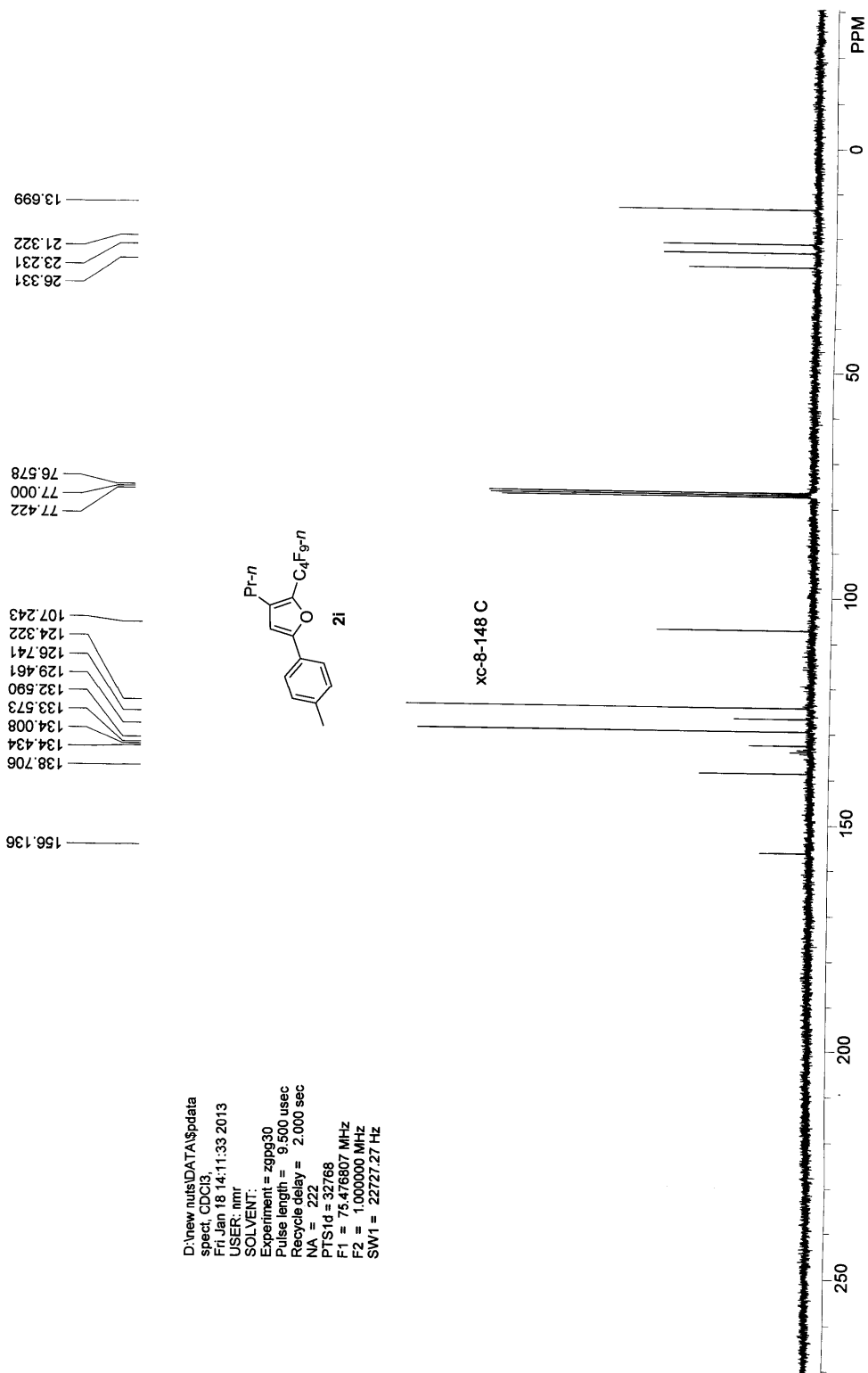




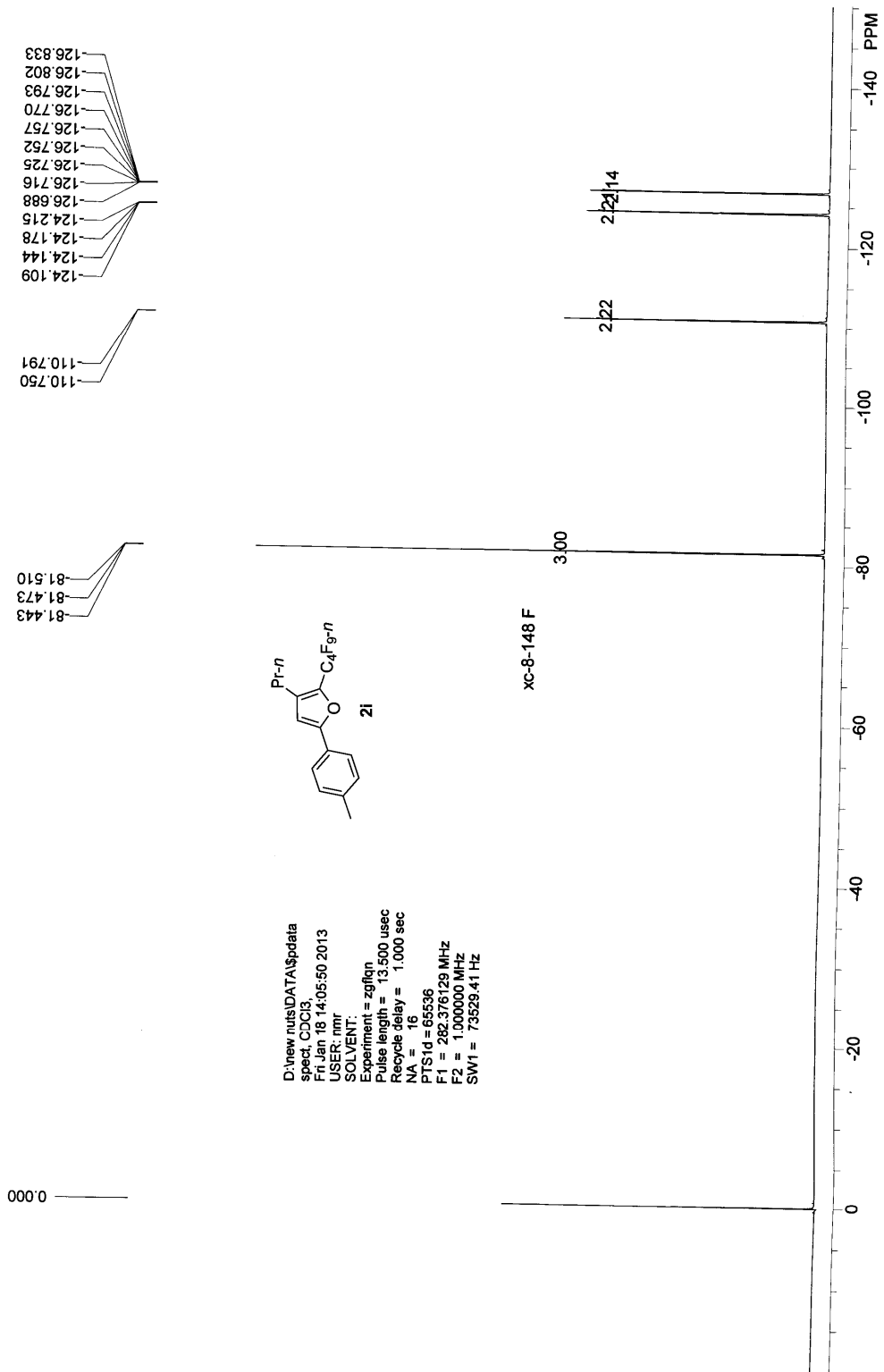


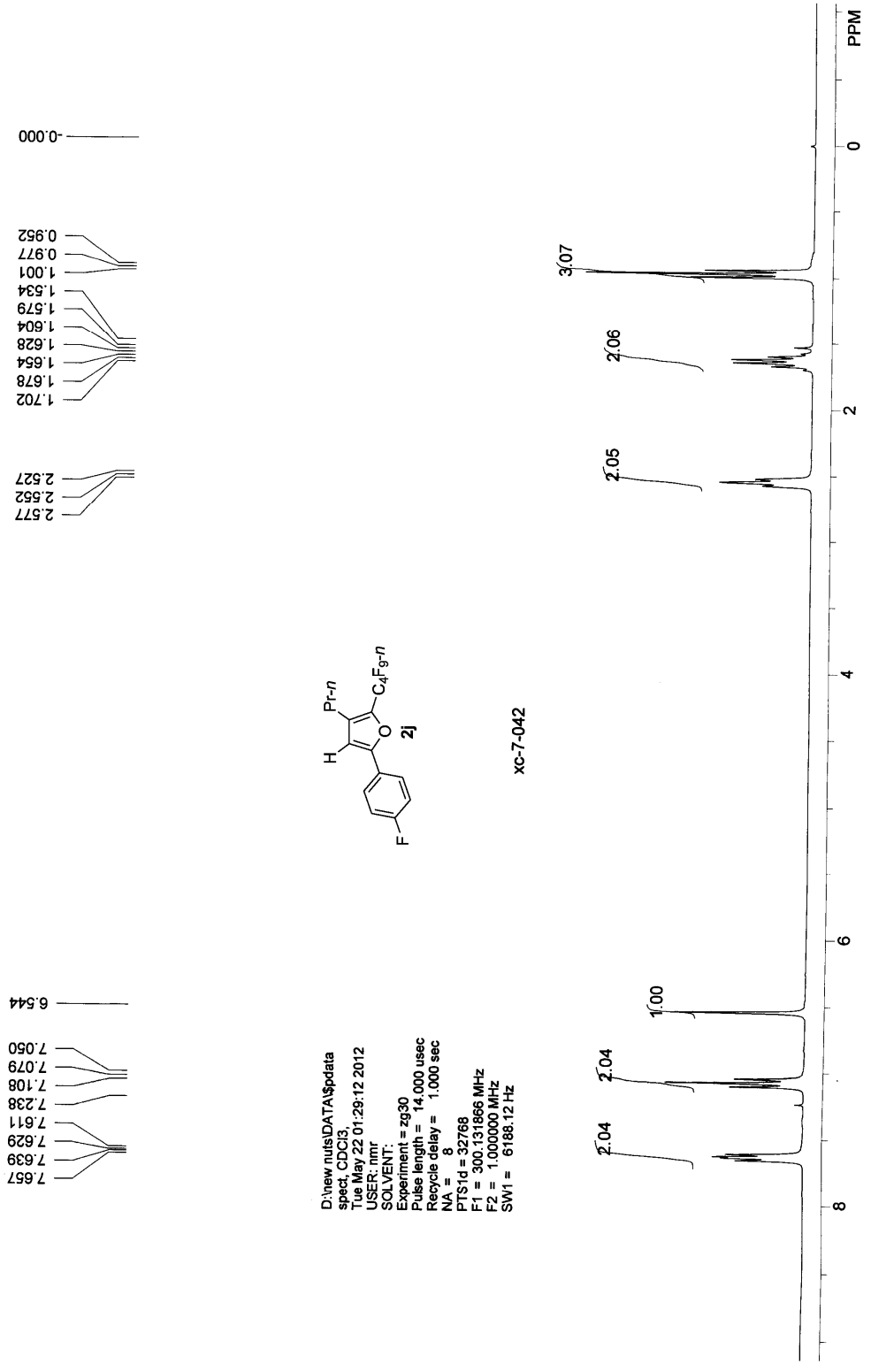
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 Fri Jan 18 14:08:37 2013
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SW1 = 6188.12 Hz





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 spect, CDCI8,
 Fri Jan 18 14:11:33 2013
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
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 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

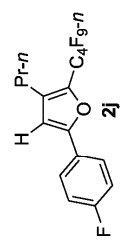




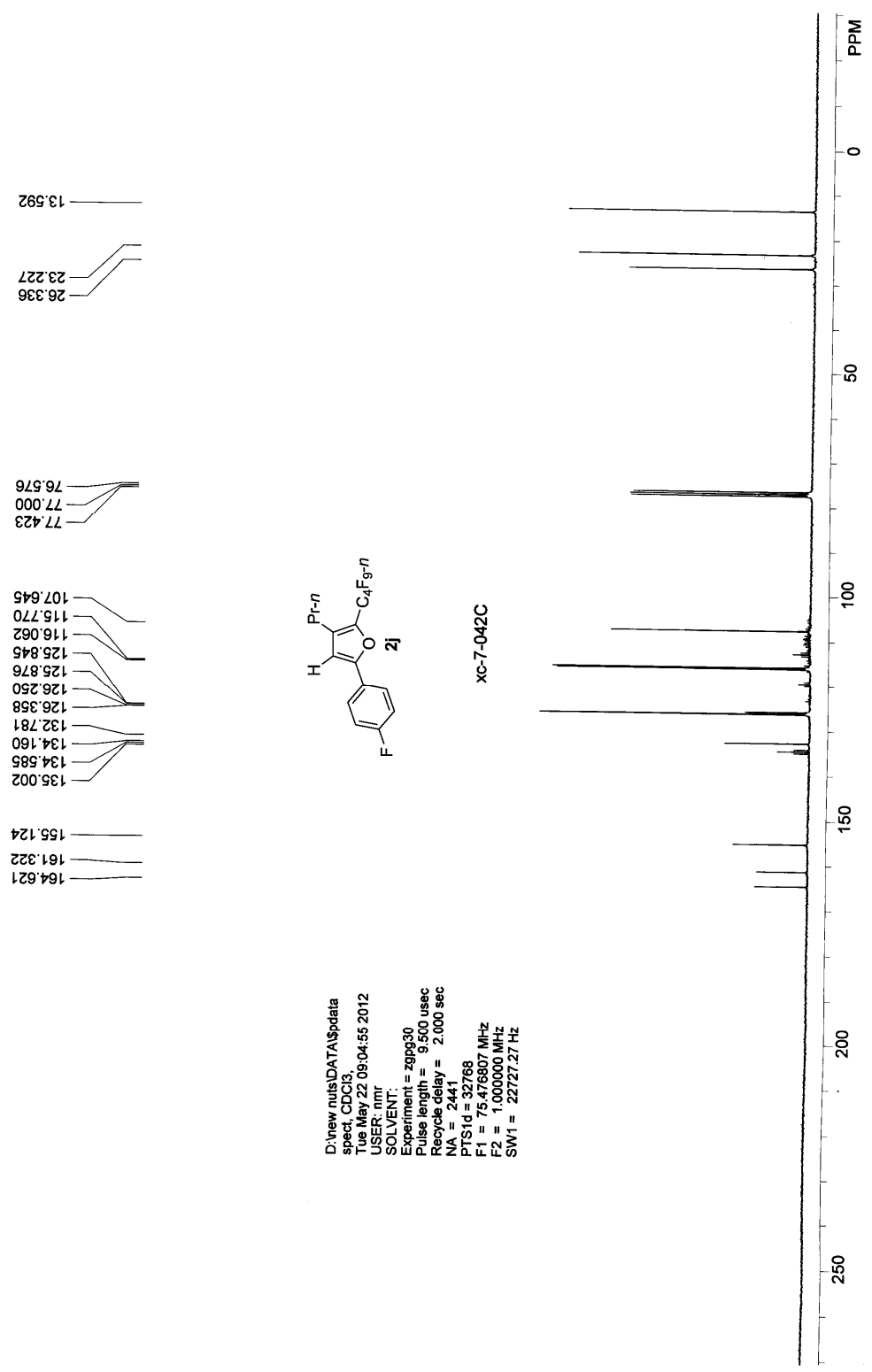
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 spect_CDCl3
 Tue May 22 01:29:12 2012
 USER: mmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 P1S1d = 32768
 F1 = 300,131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz

xc-7-042

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 spect, CDCIS,
 Tue May 22 09:04:55 2012
 USER: nmr
 SOLVENT: nmr
 Experiment = z9pg30
 Pulse length = 9.500 usec
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 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

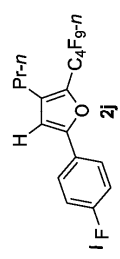


xc-7-042C



0.000

D:\newnuts\DATA\spdata
spect, CDCI3,
Tue May 22 01:32:20 2012
USER: nmr
SOLVENT:
Experiment = zgpgn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTSD = 65536
F1 = 282.376129 MHz
F2 = 1,000,000 MHz
SWH = 73529.41 Hz

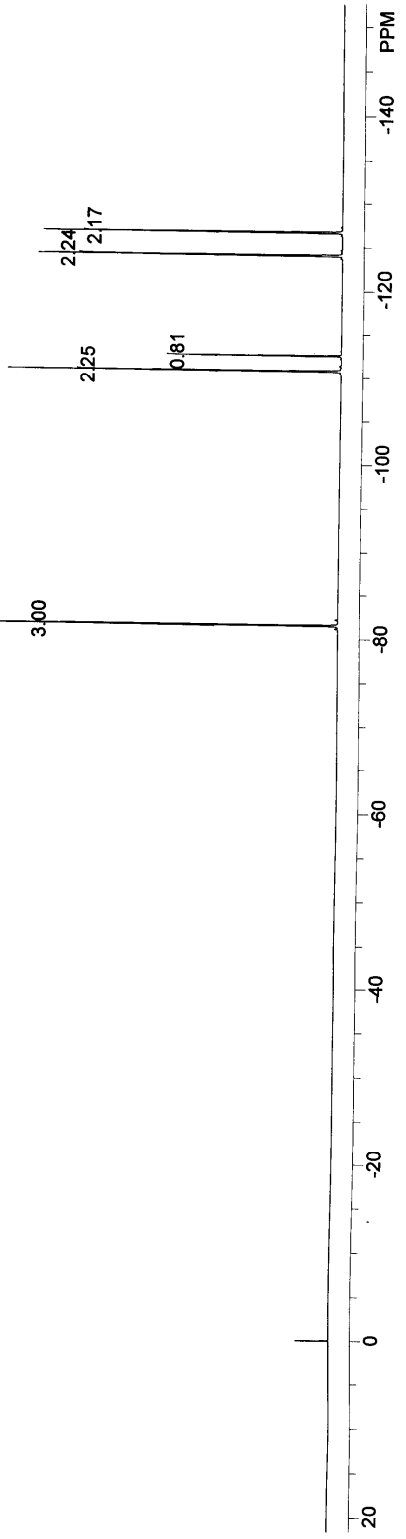


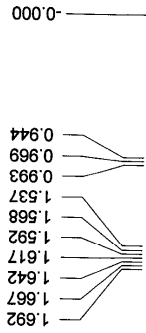
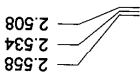
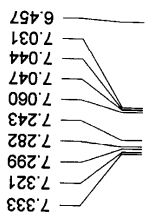
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81.518
81.524

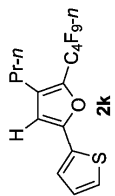
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112.555
112.574

124.057
124.092
126.653
126.694
126.729

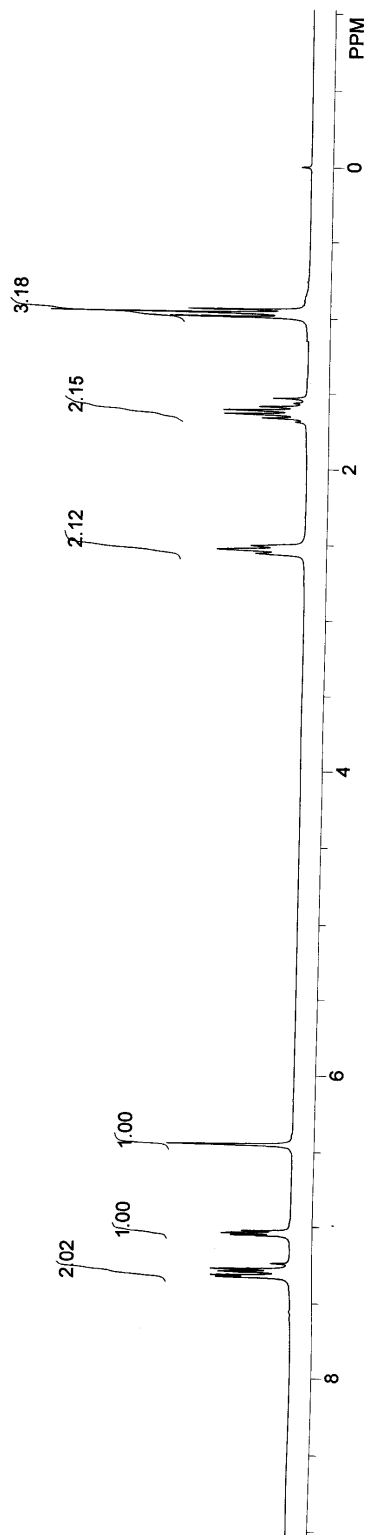


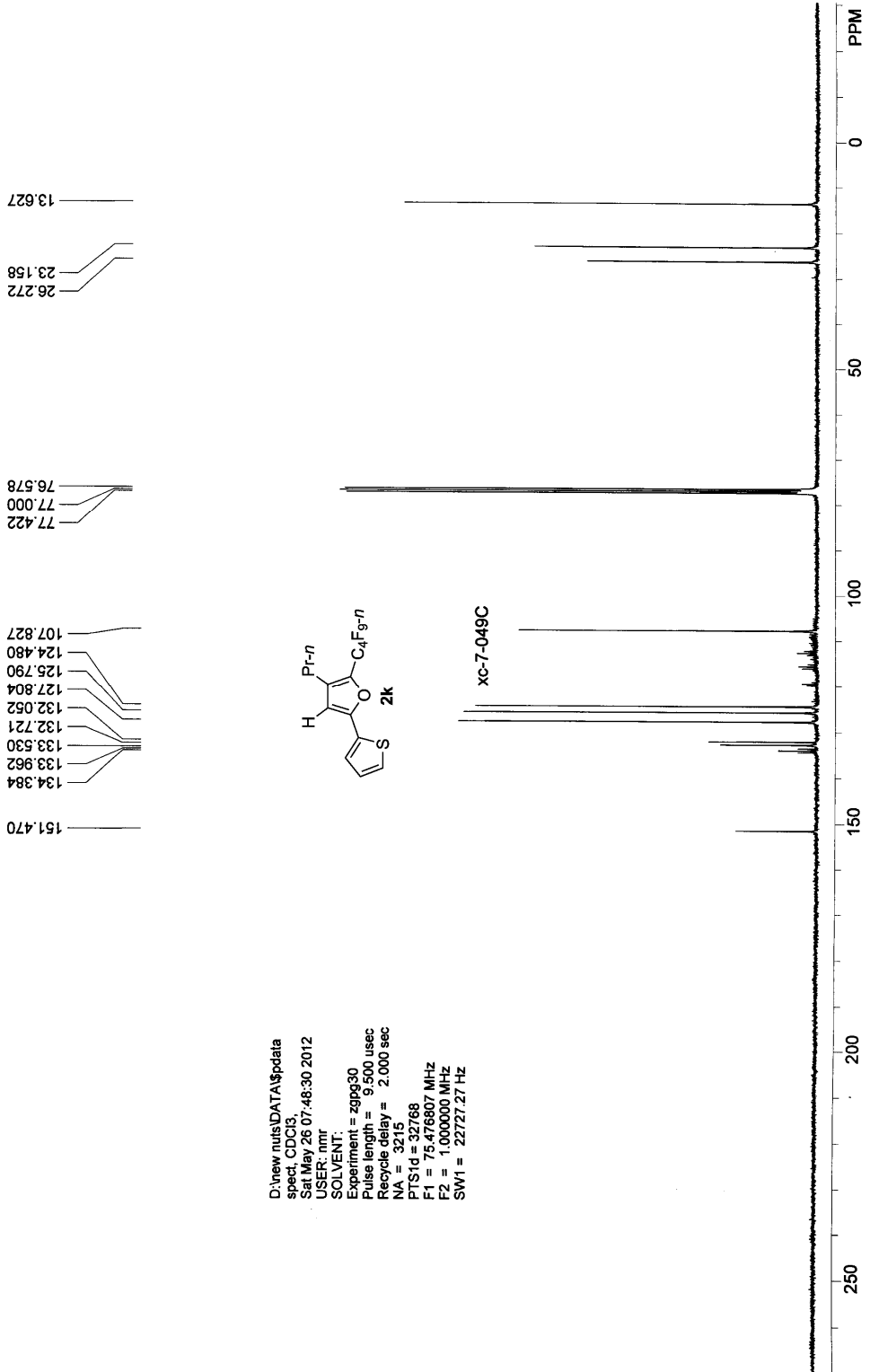


D:\new\nu1\data\spdata
spect, CDCl3,
Sat May 26 07:27:11 2012
USER: nmr
SOLVENT: nmr
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
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F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6186.12 Hz



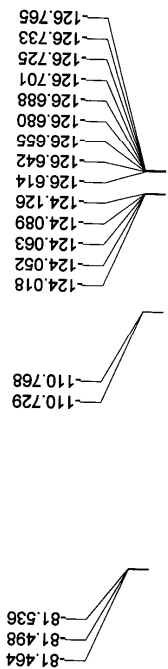
xc-7-049





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 Sat May 26 07:48:30 2012
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 3215
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

-0.000



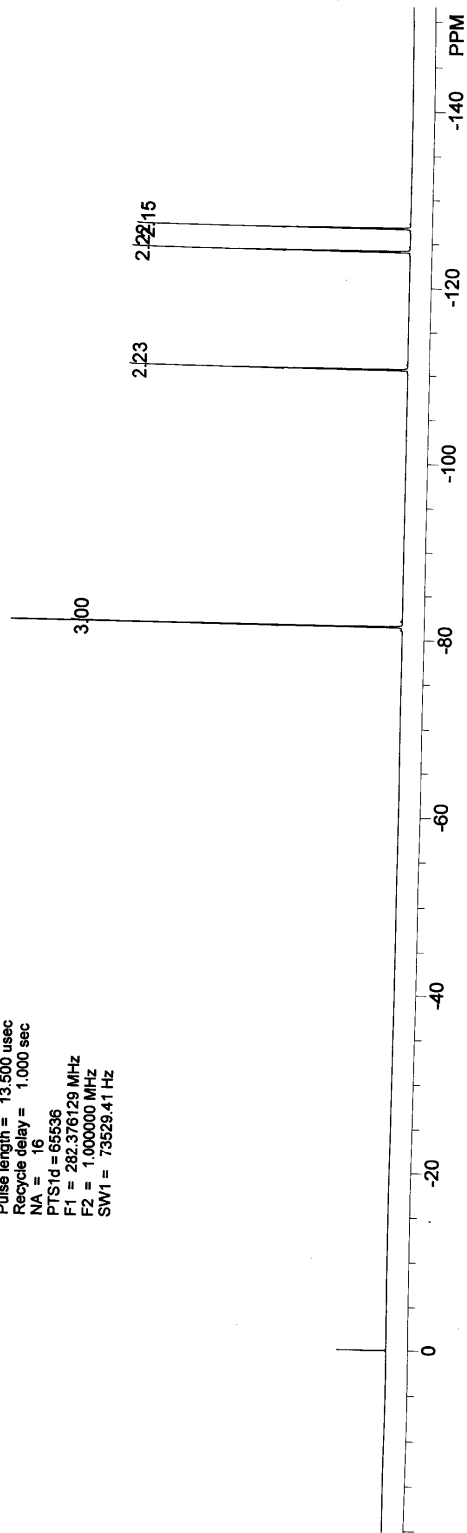
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81.498
81.536


110.729
110.768

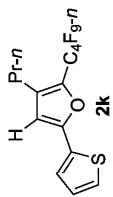
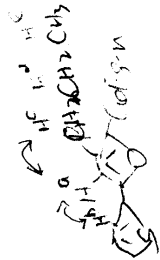
124.018
124.052
124.063
124.089
124.126
126.614
126.642
126.655
126.680
126.688
126.701
126.725
126.733
126.765

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spect_CDCl3
Sat May 26 07:30:29 2012
USER: nmr
SOLVENT:
Experiment = zgfgn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

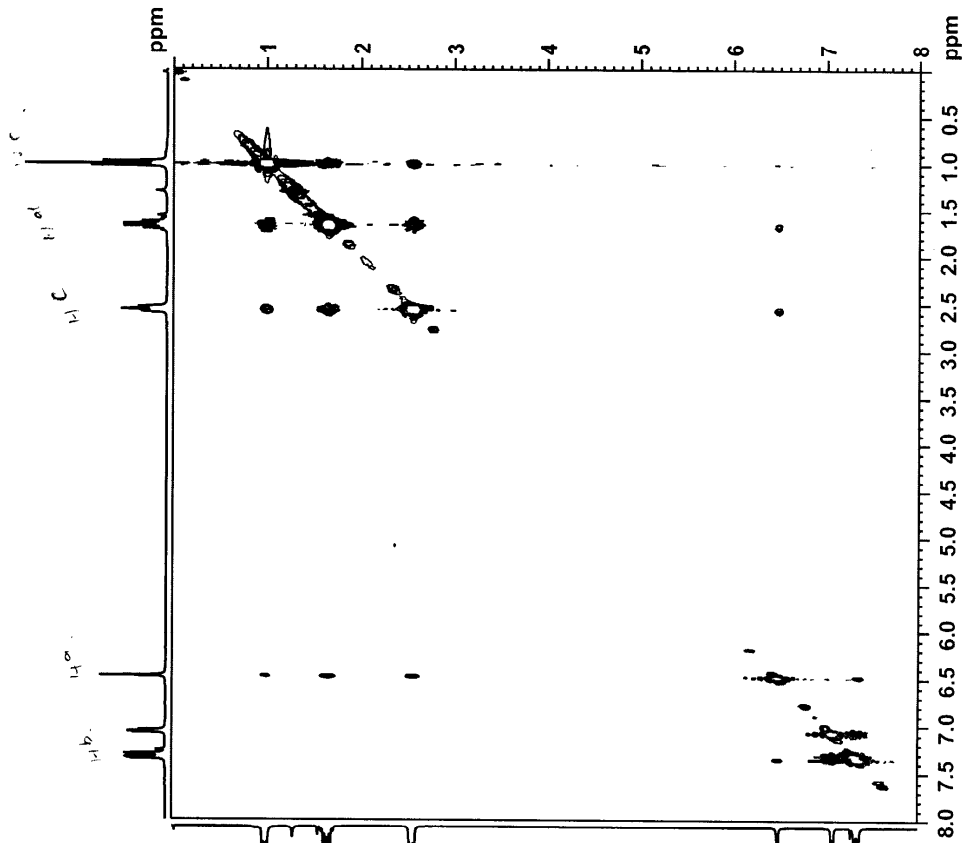
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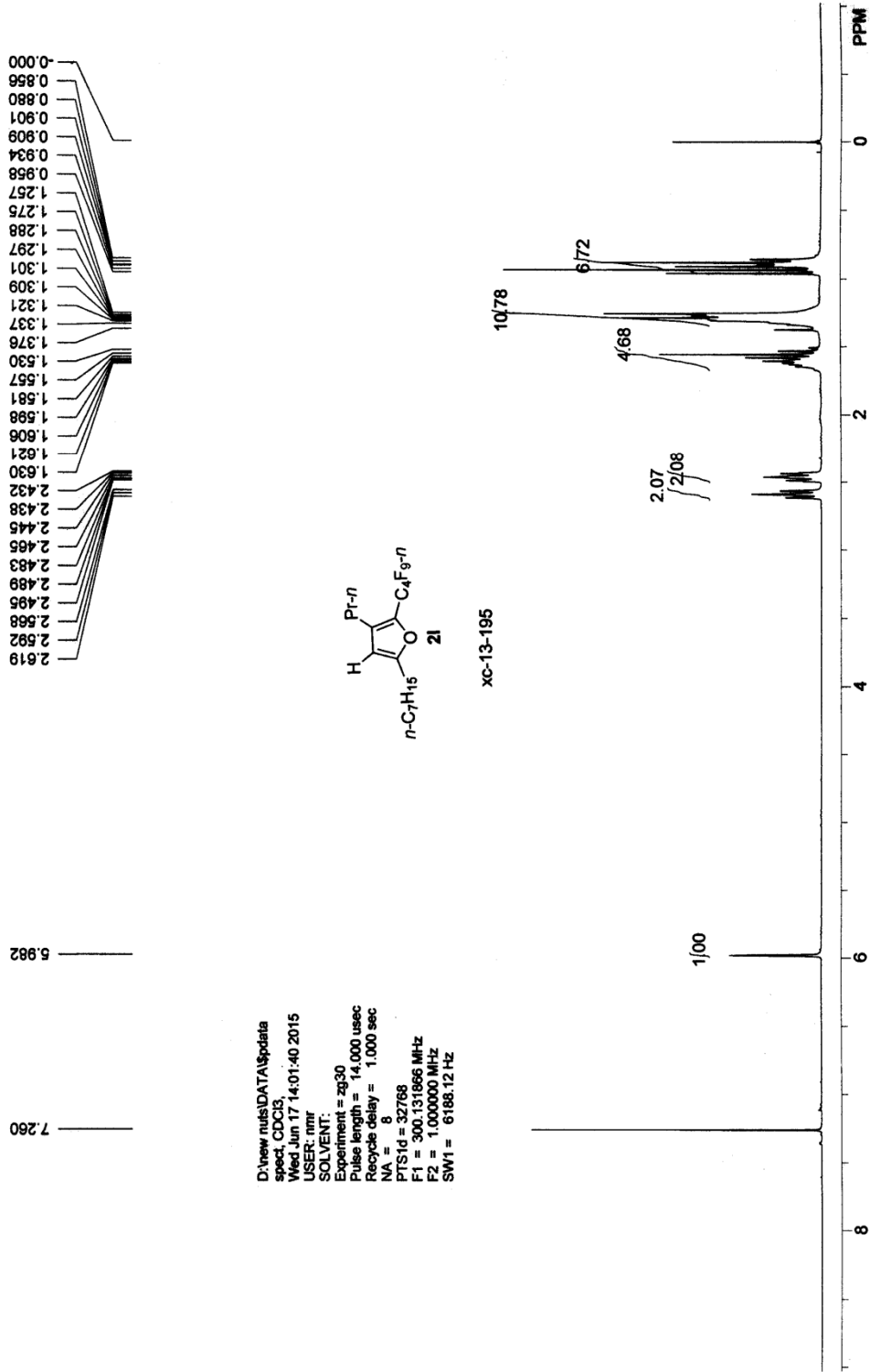



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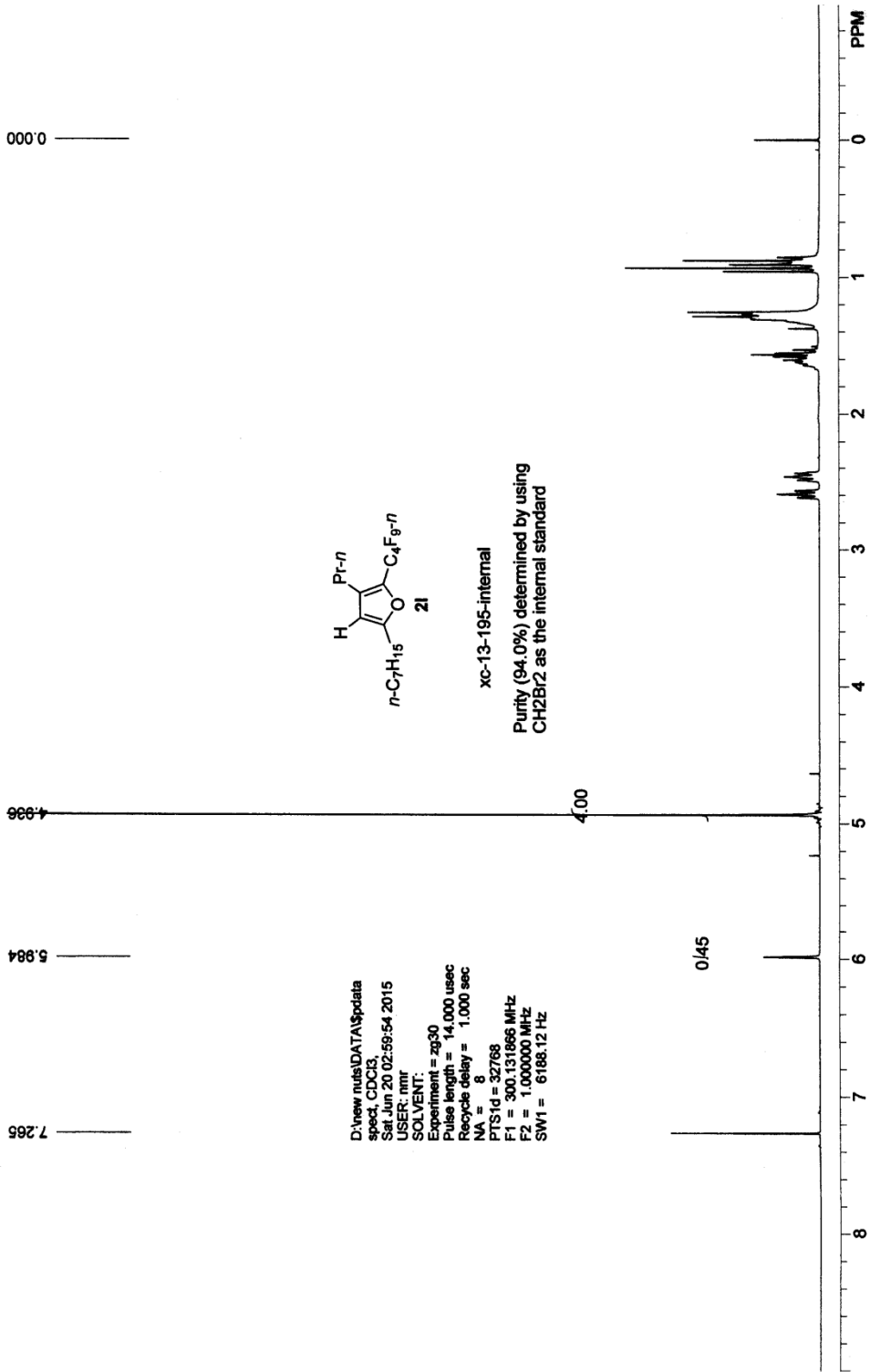


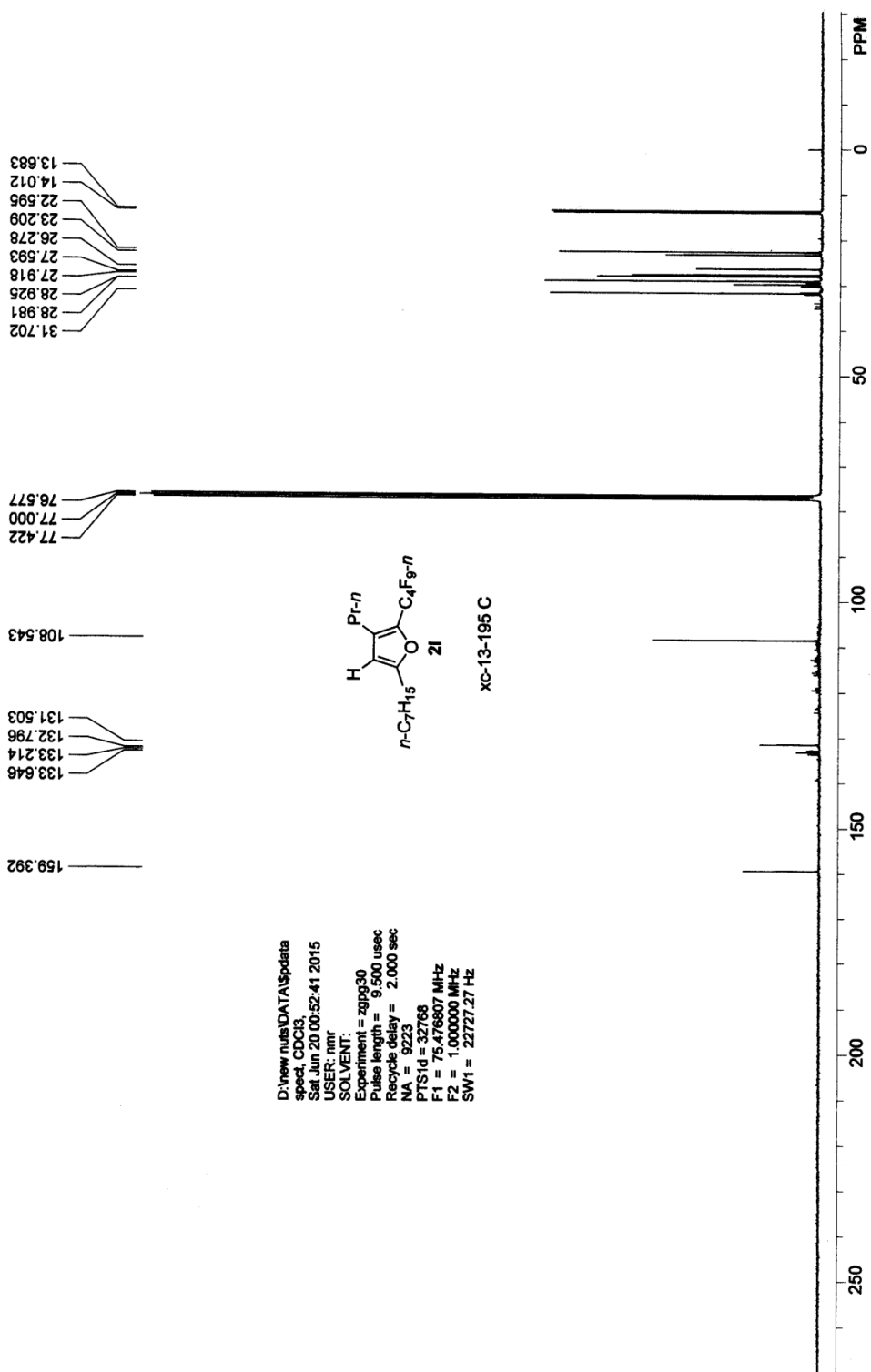
xc-7-049-noe

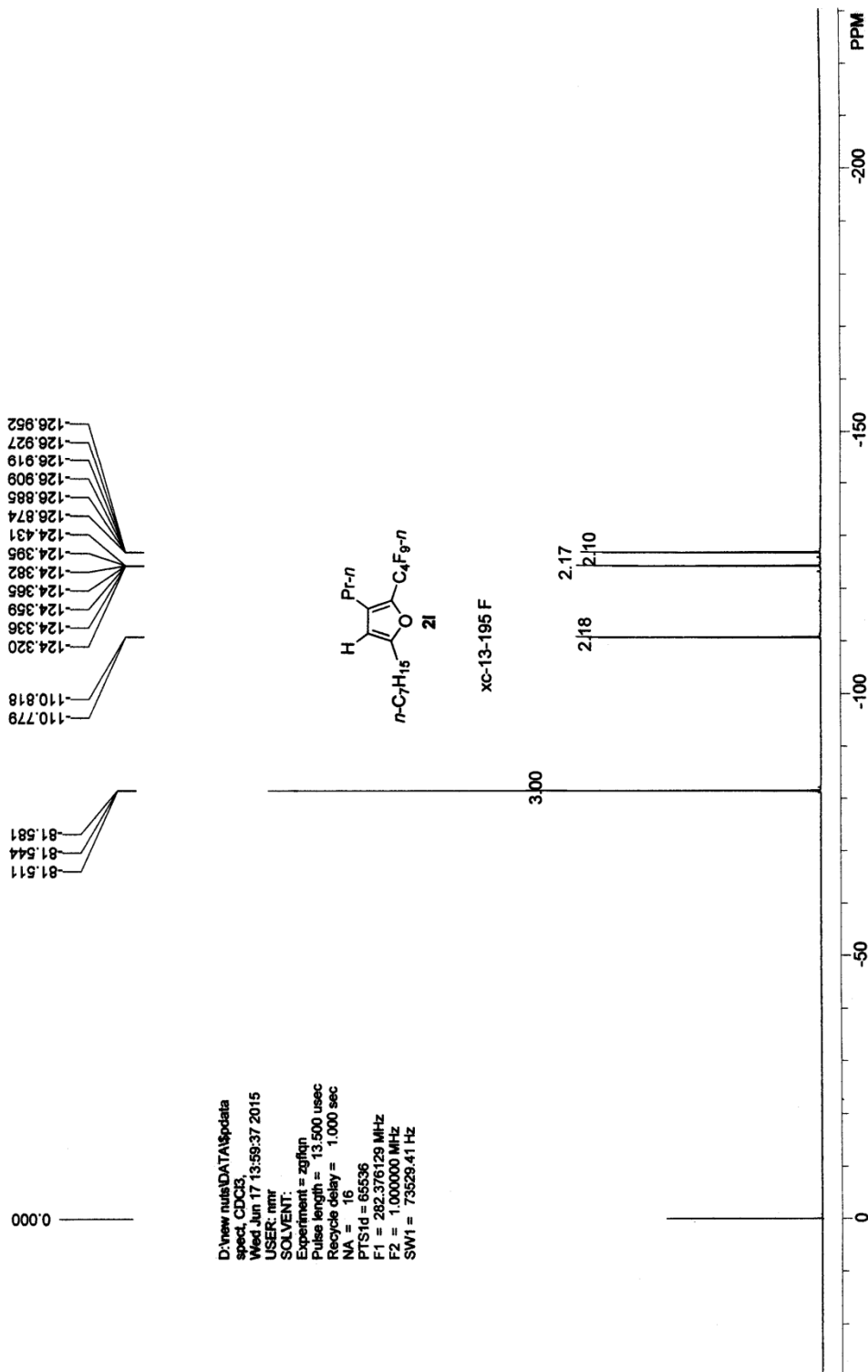




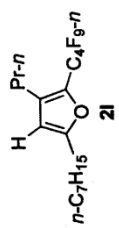
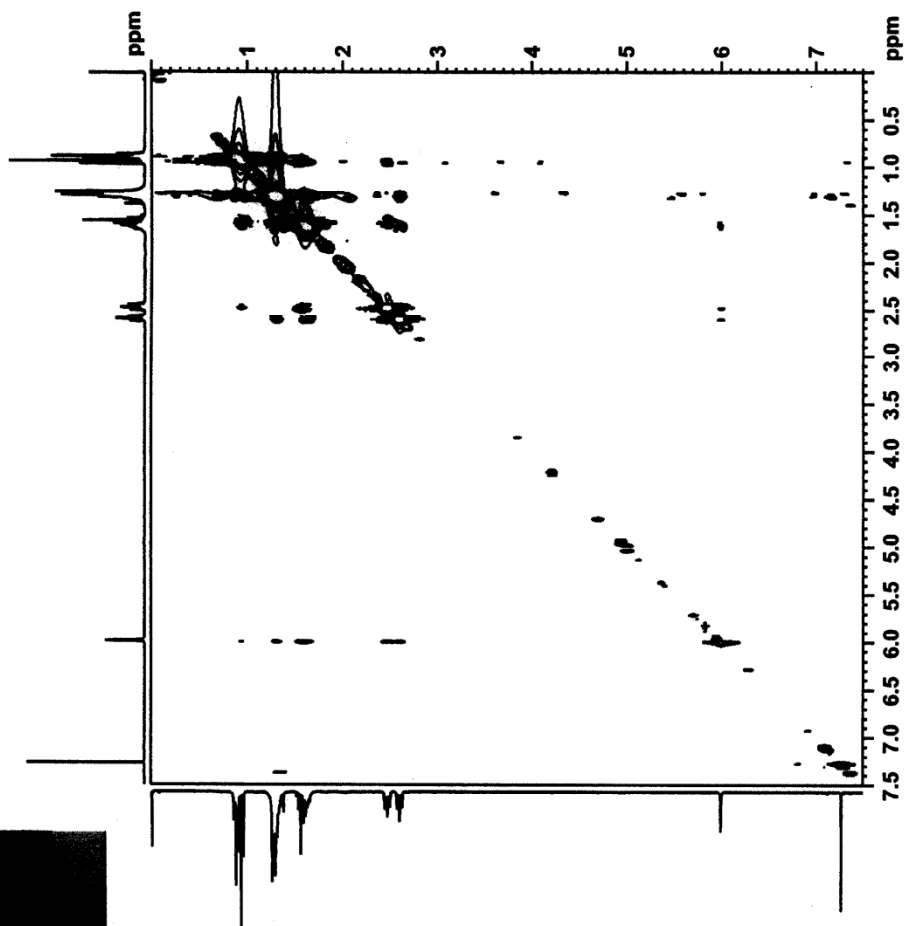
D:\new nuSIDATA\pdata
 spect CDCl3
 Wed Jun 17 14:01:40 2015
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz



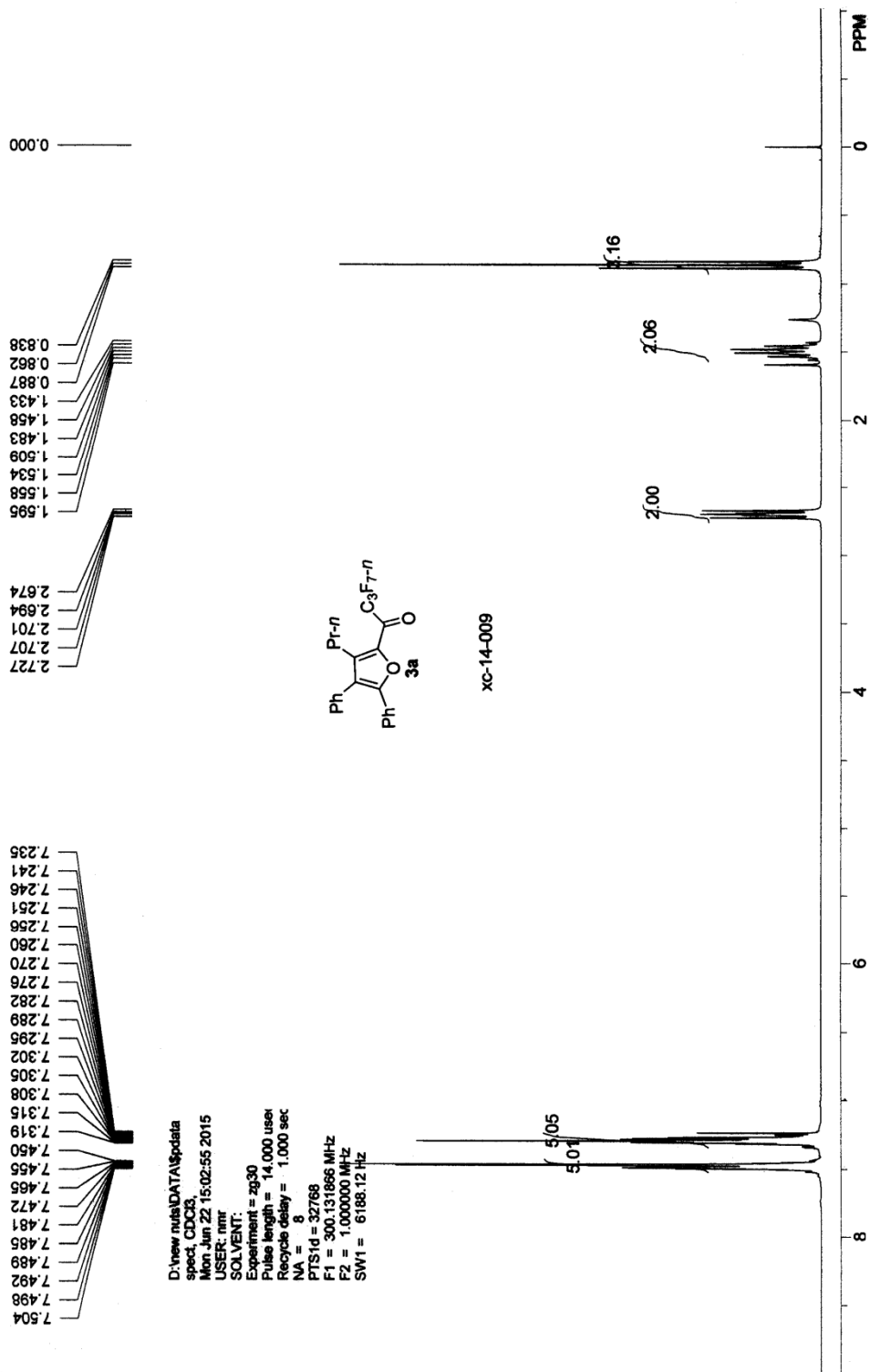


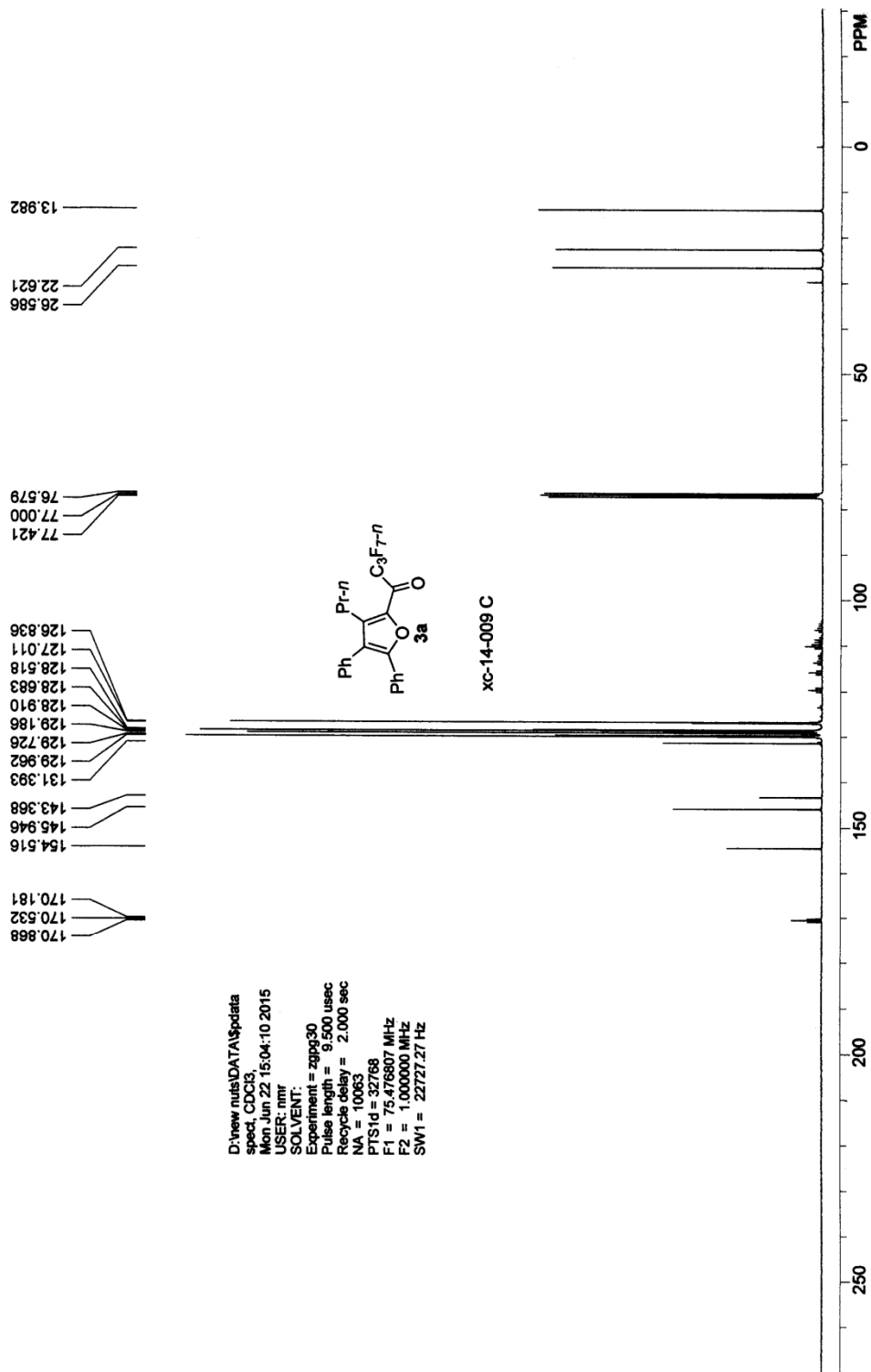


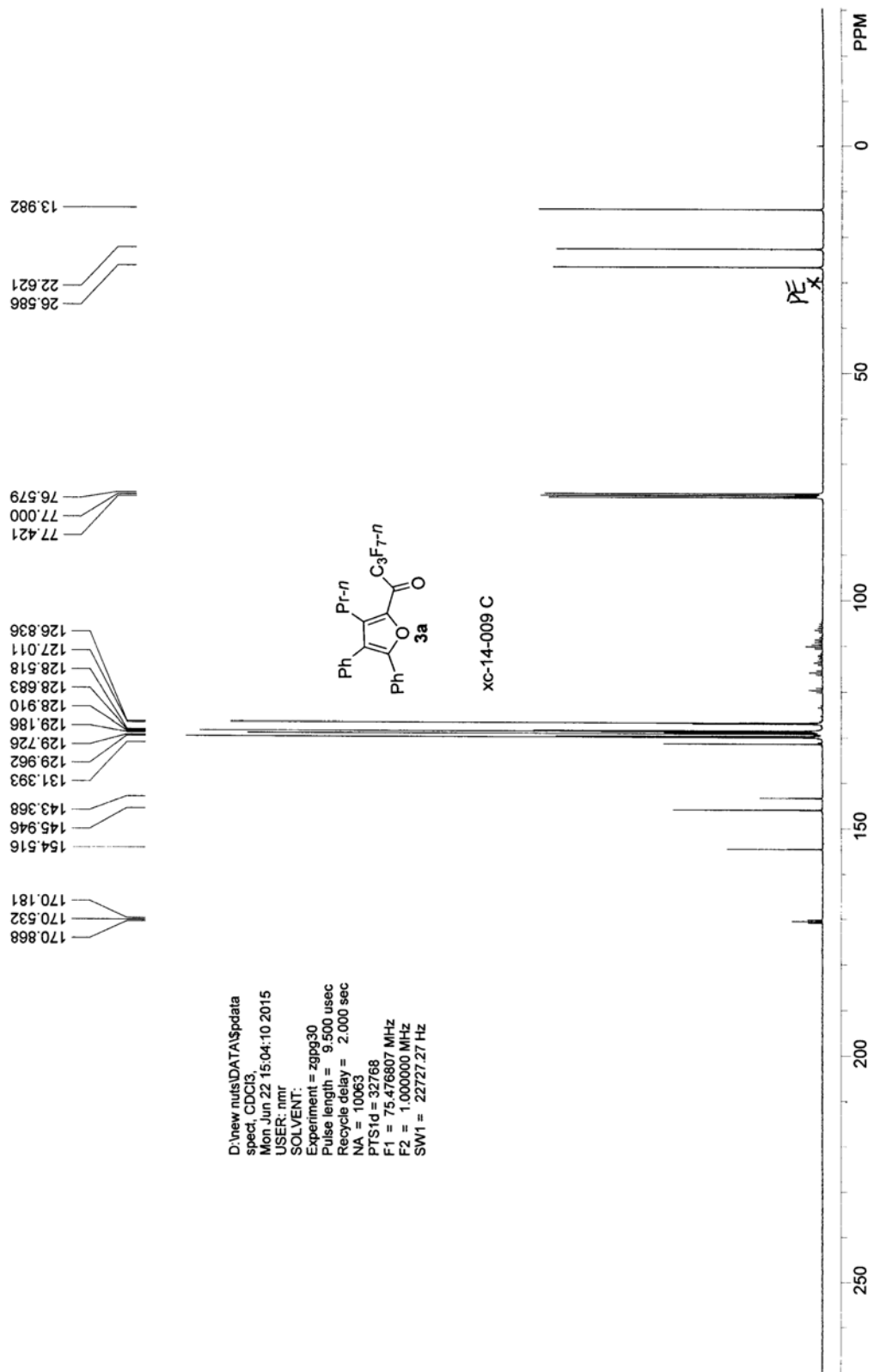
D:\new nuis\DATA\spdata
 spect, CDCCl3,
 Wed Jun 17 13:59:37 2015
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 13.500 usec
 Recycle delay = 1.000 sec
 NA = 16
 P1 = 65536
 F1 = 282.376129 MHz
 F2 = 1.000000 MHz
 SWH = 73529.41 Hz

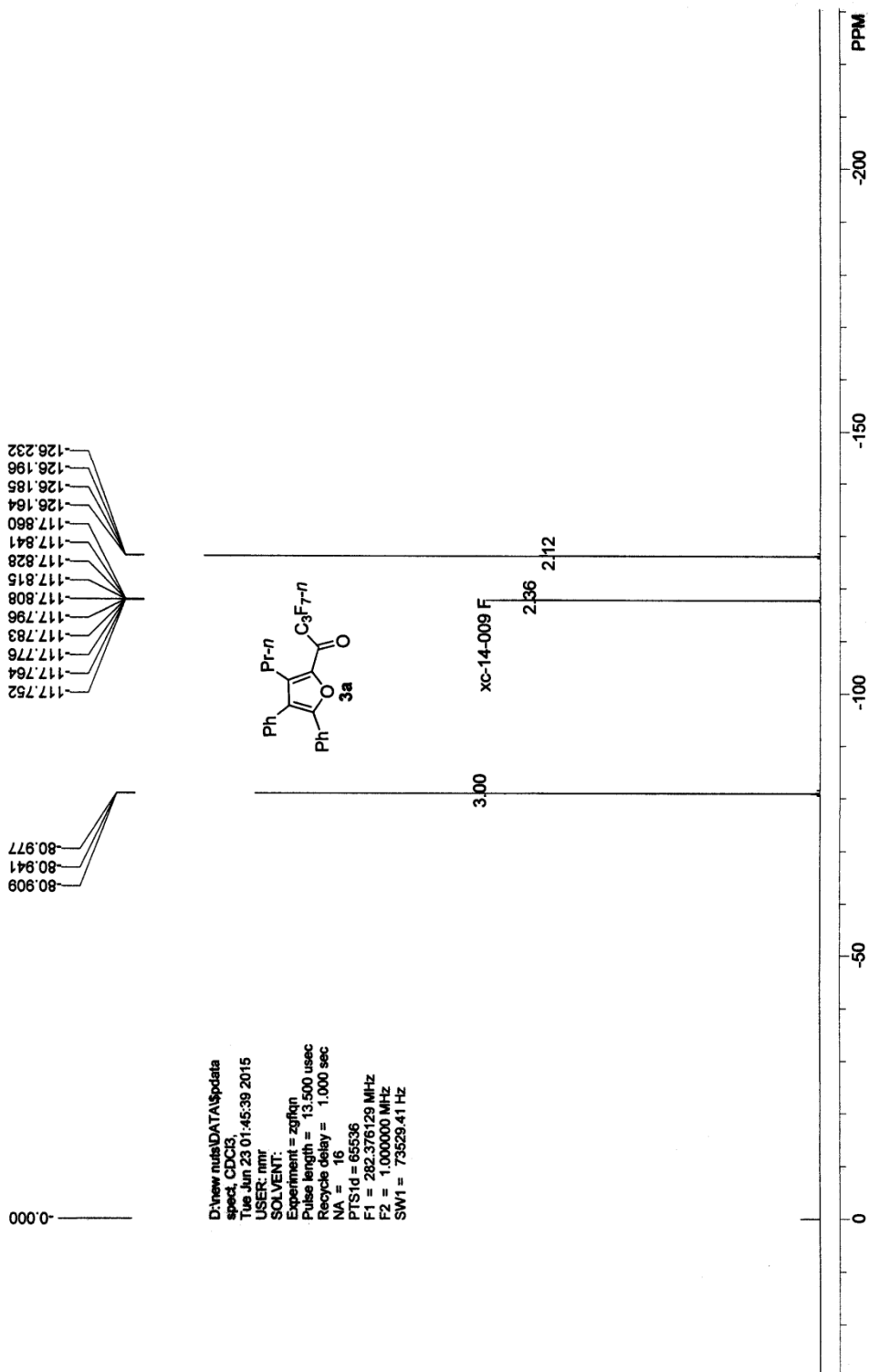


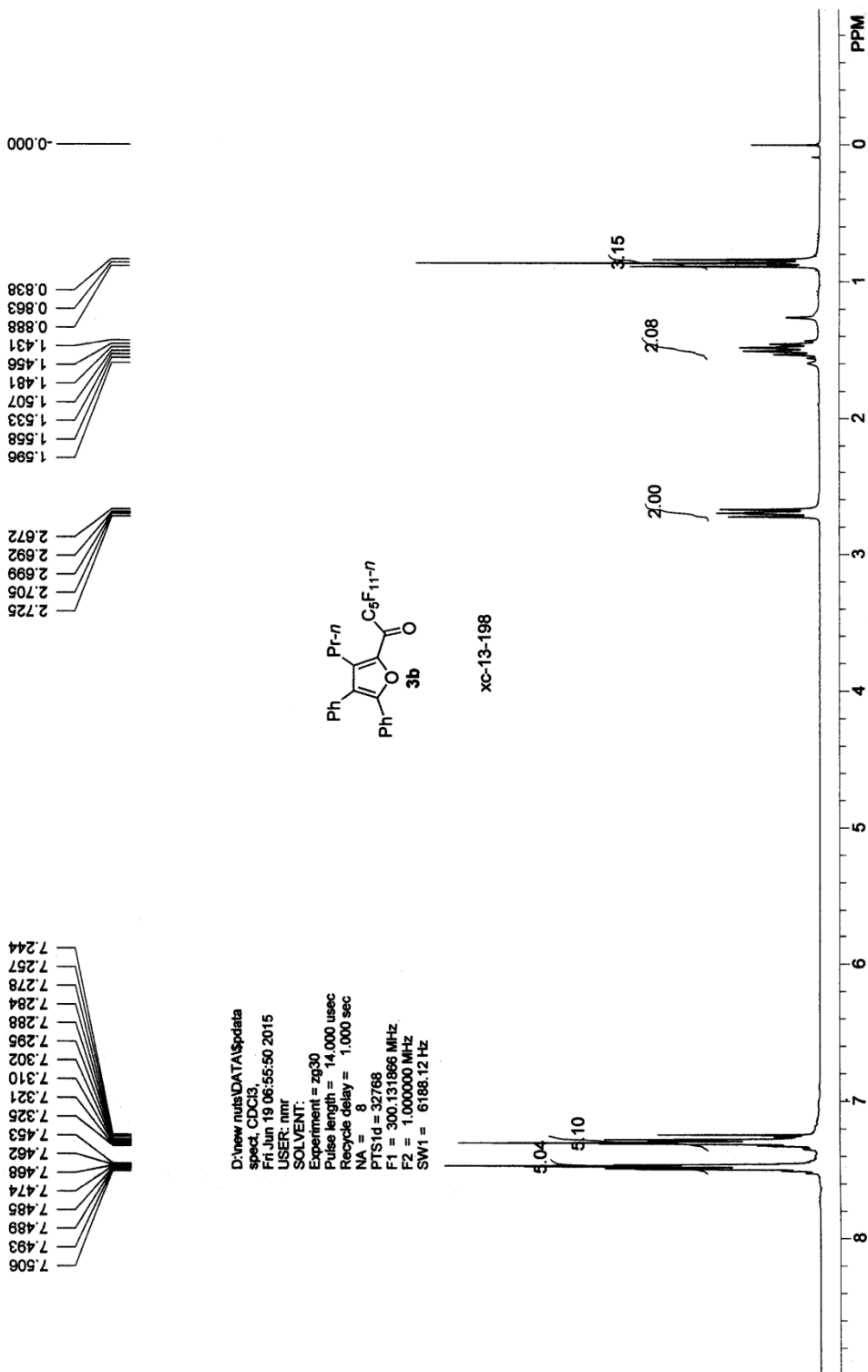
xc-13-195-noe

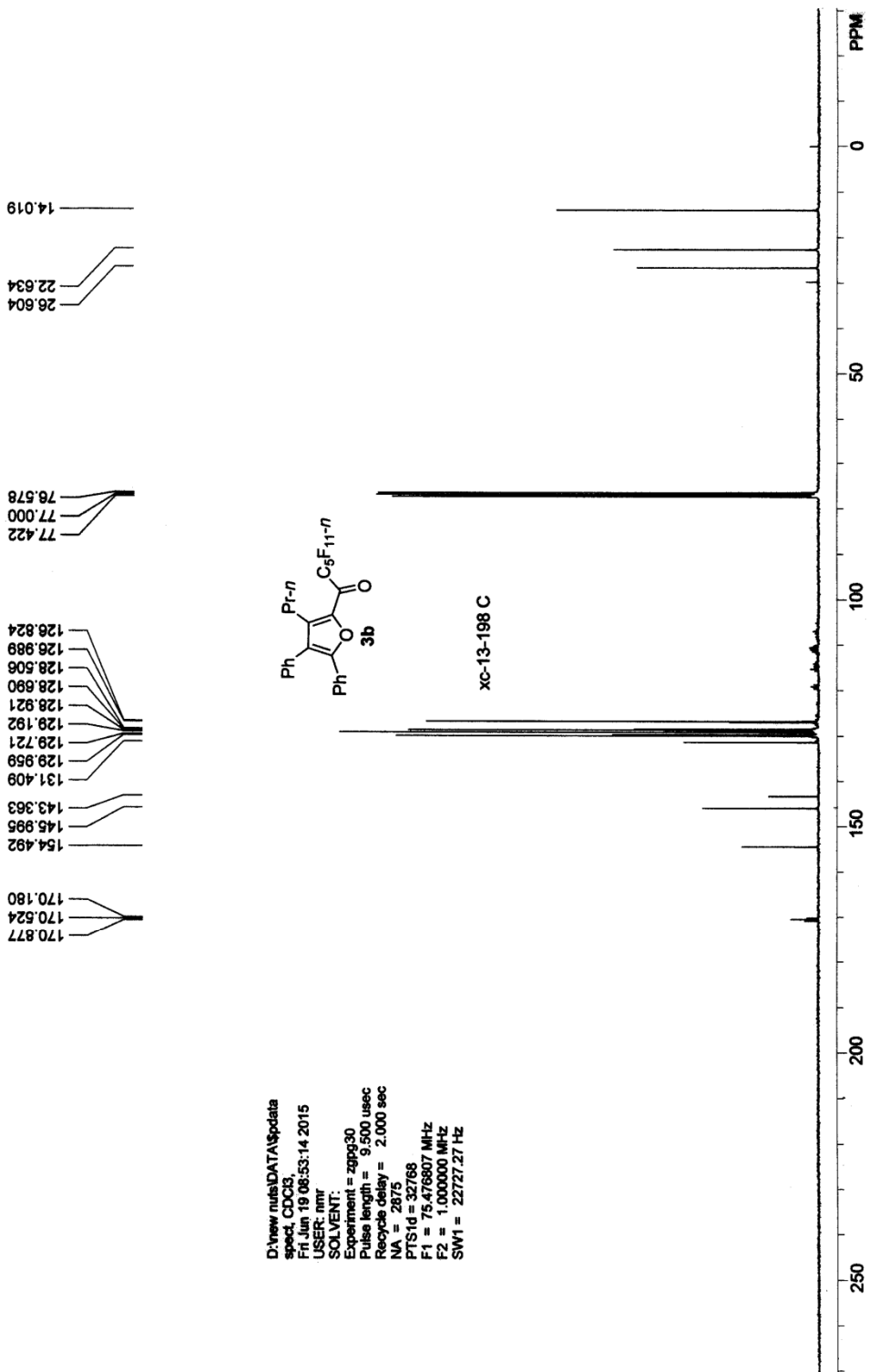




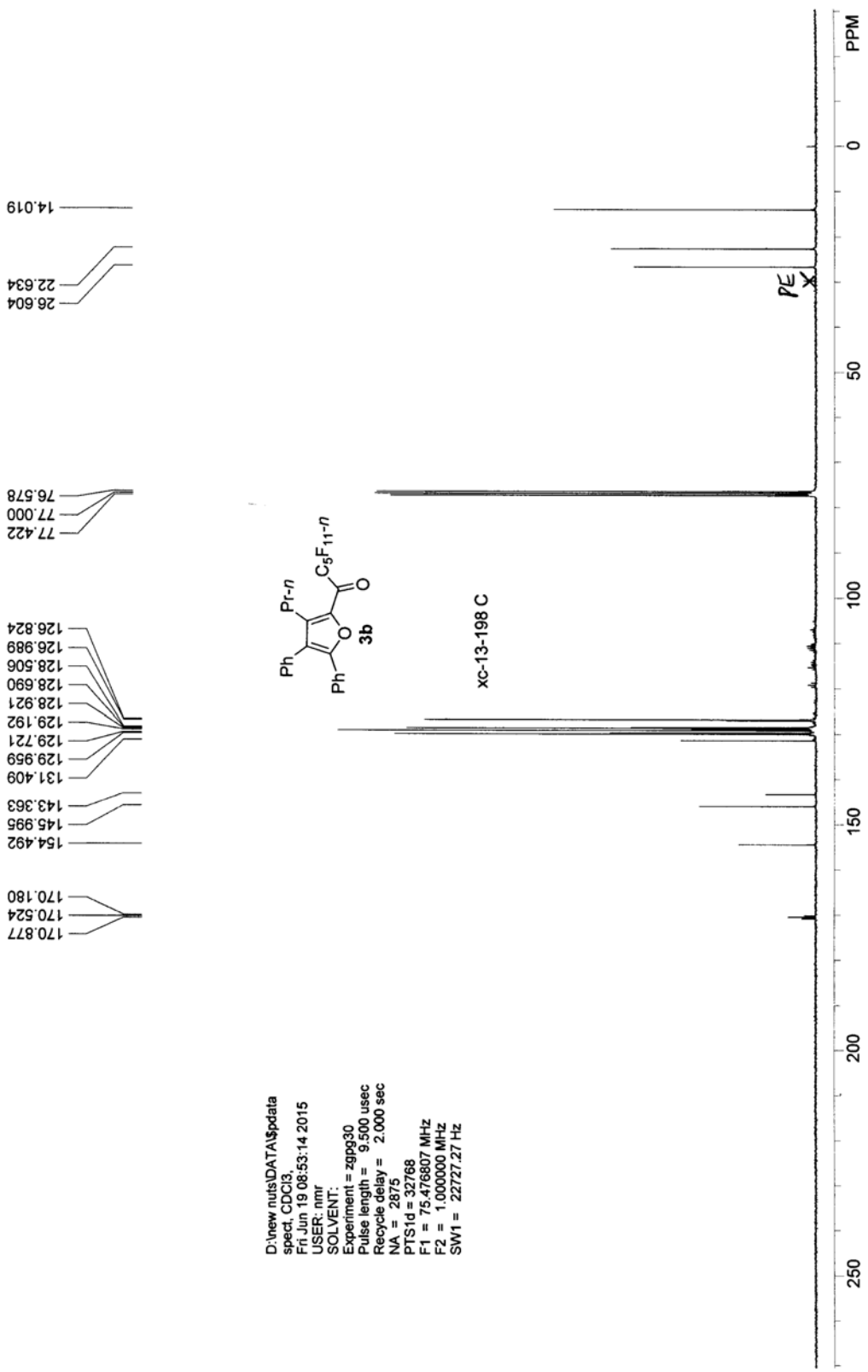




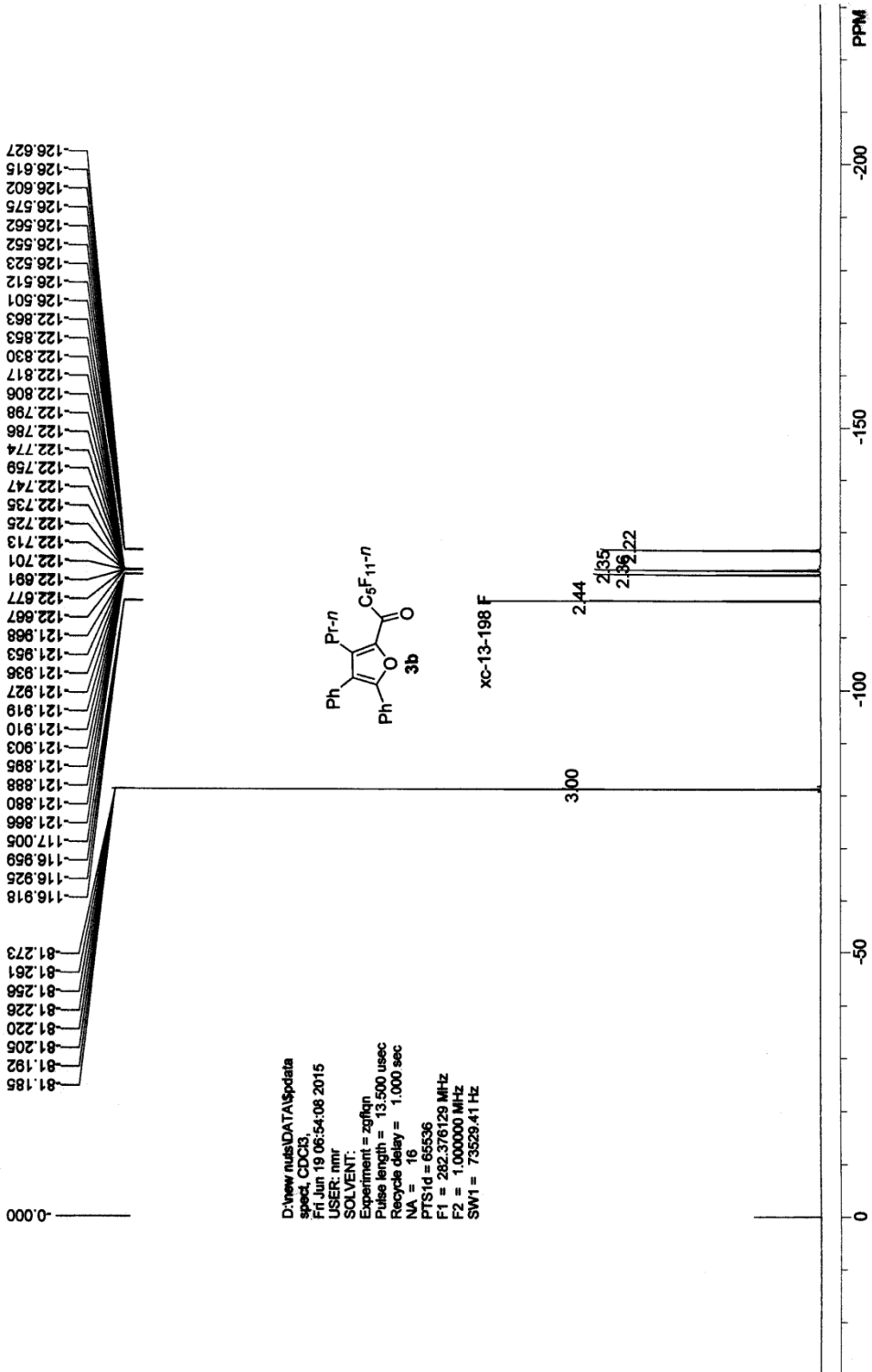




D:\new nutis\DATA\spdata
 spect, CQC8,
 Fri Jun 19 08:53:14 2015
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 2675
 P1S1d = 32768
 P1 = 75.476807 MHz
 P2 = 1.000000 MHz
 SW1 = 22727.27 Hz

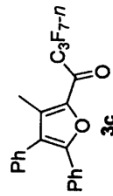


D:\new nuts\DATA\p\data
 spect, CDCI3.
 Fri Jun 19 08:53:14 2015
 USER: nmf
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 2875
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



7.519
7.514
7.507
7.504
7.501
7.494
7.488
7.486
7.481
7.475
7.472
7.470
7.467
7.463
7.333
7.324
7.319
7.313
7.306
7.296
7.287
7.278
7.273
7.268
7.244

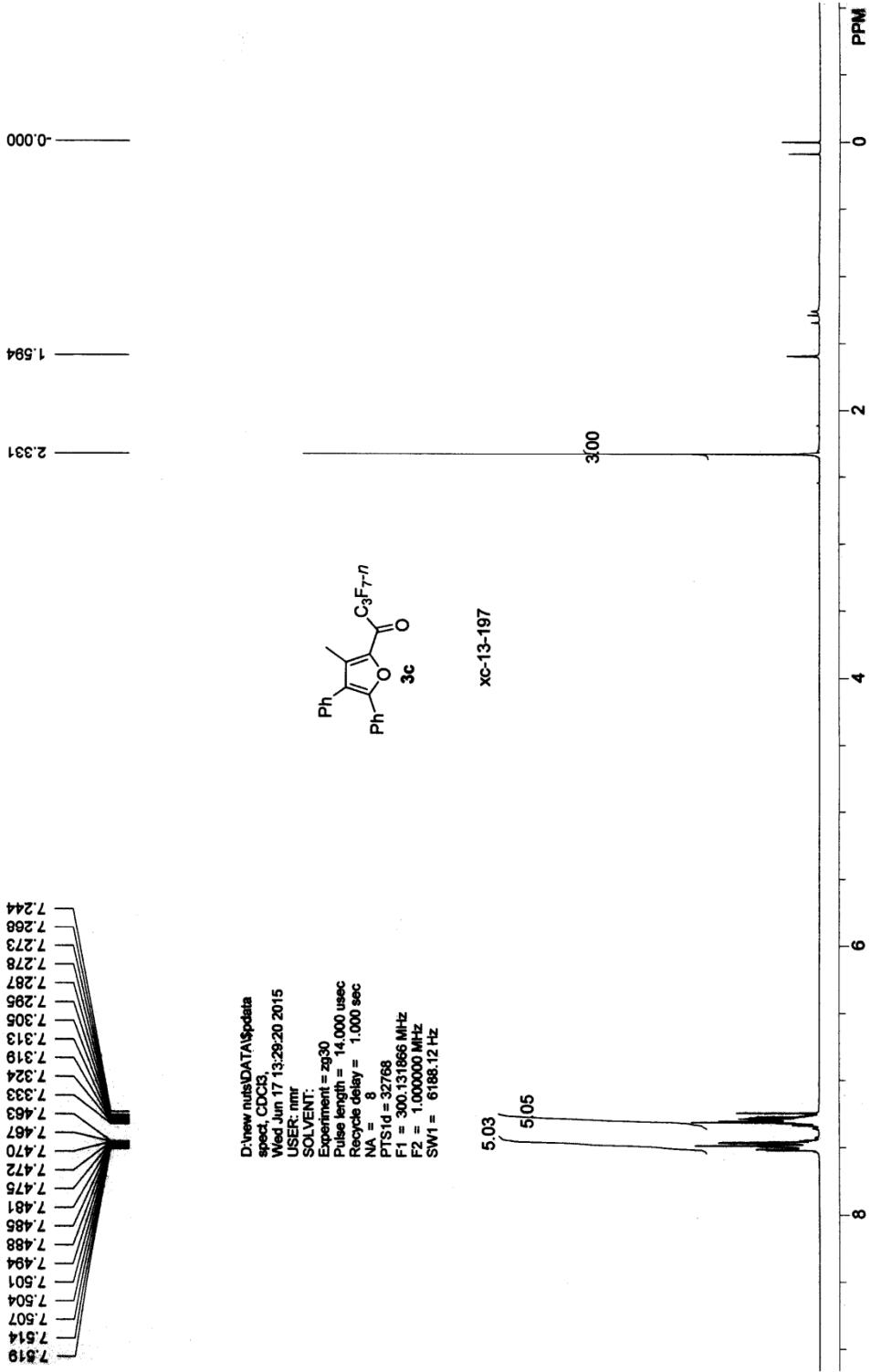
D:\new nuts\DATA\Spdata
spect_CD03
Wed Jun 17 13:29:20 2015
USER: nmr
SOLVENT:
Experiment = z930
Pulse length = 14,000 usec
Recycle delay = 1,000 sec
NA = 8
PTSD = 32768
F1 = 300.131866 MHz
F2 = 1,000,000 MHz
SW1 = 6188.12 Hz

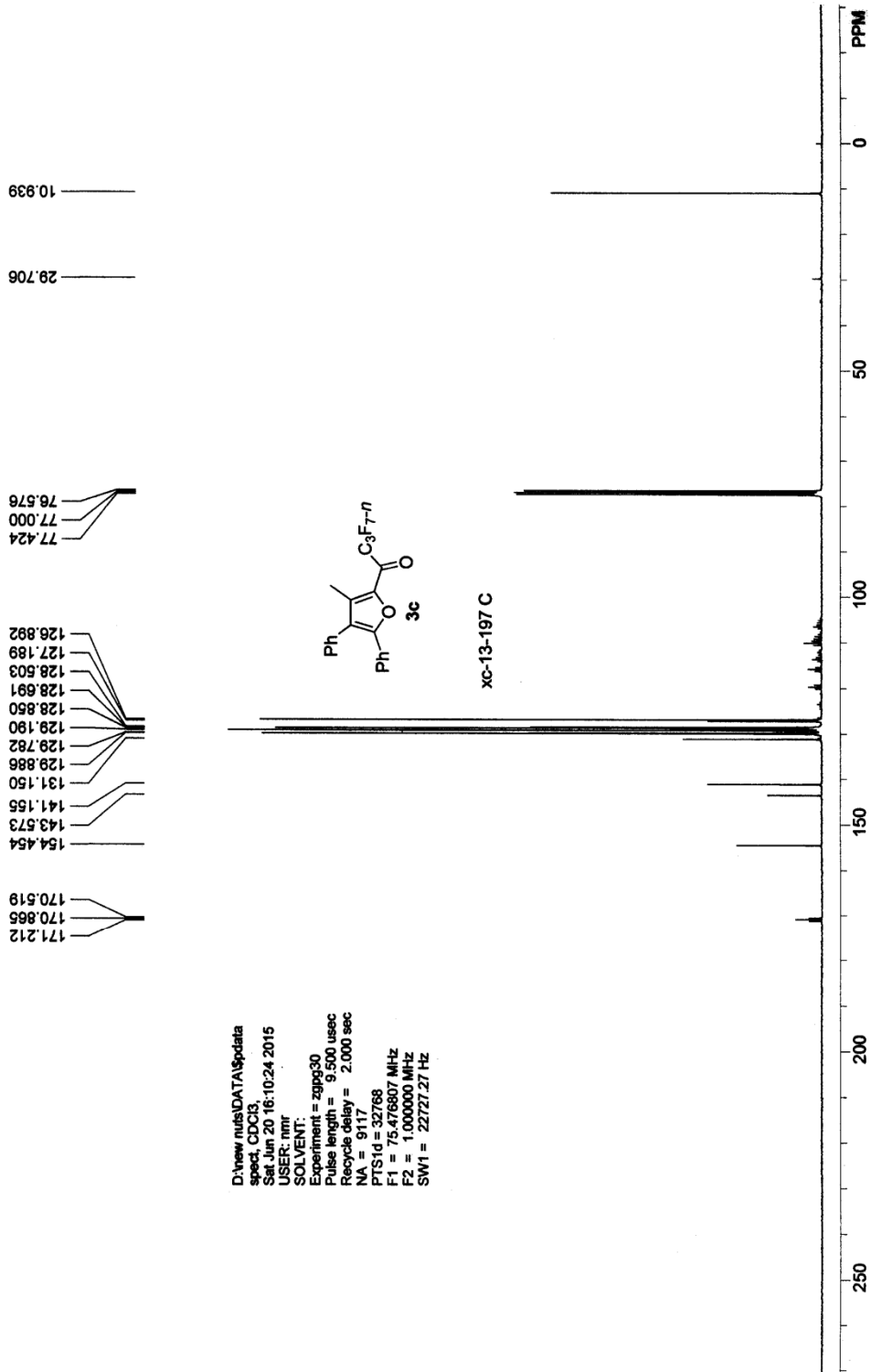


5.03
5.05

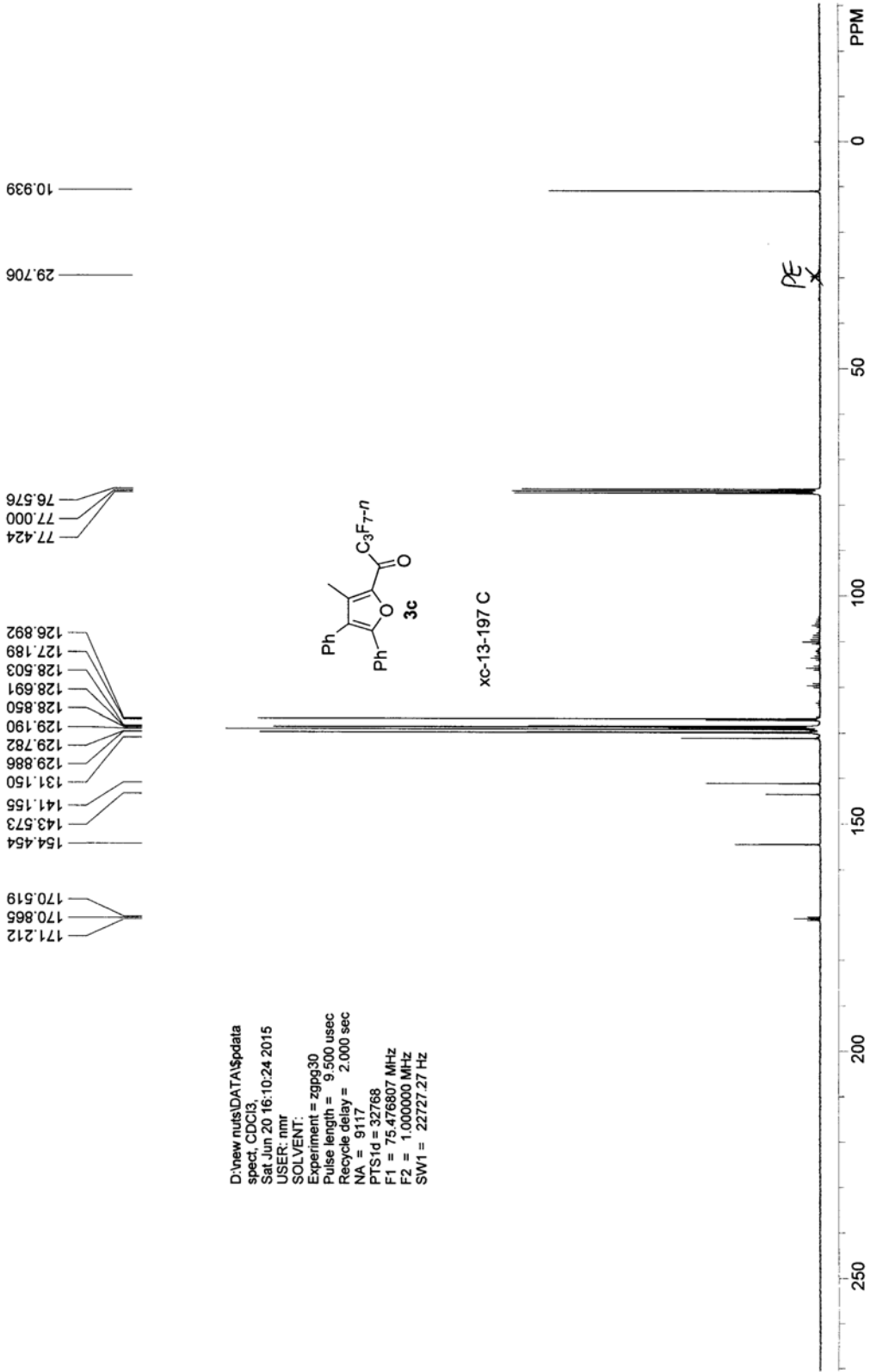
3.00

xc-13-197

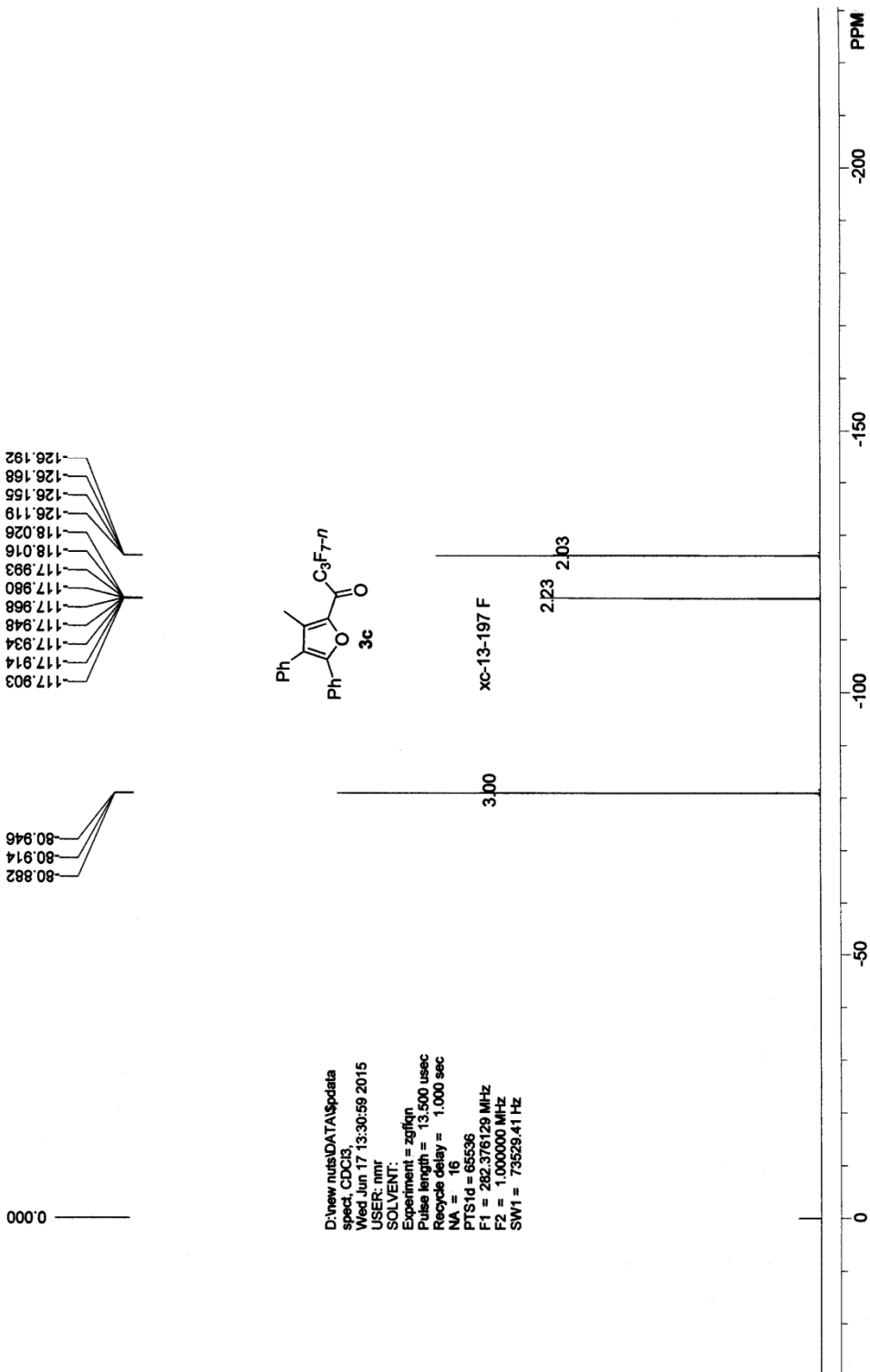


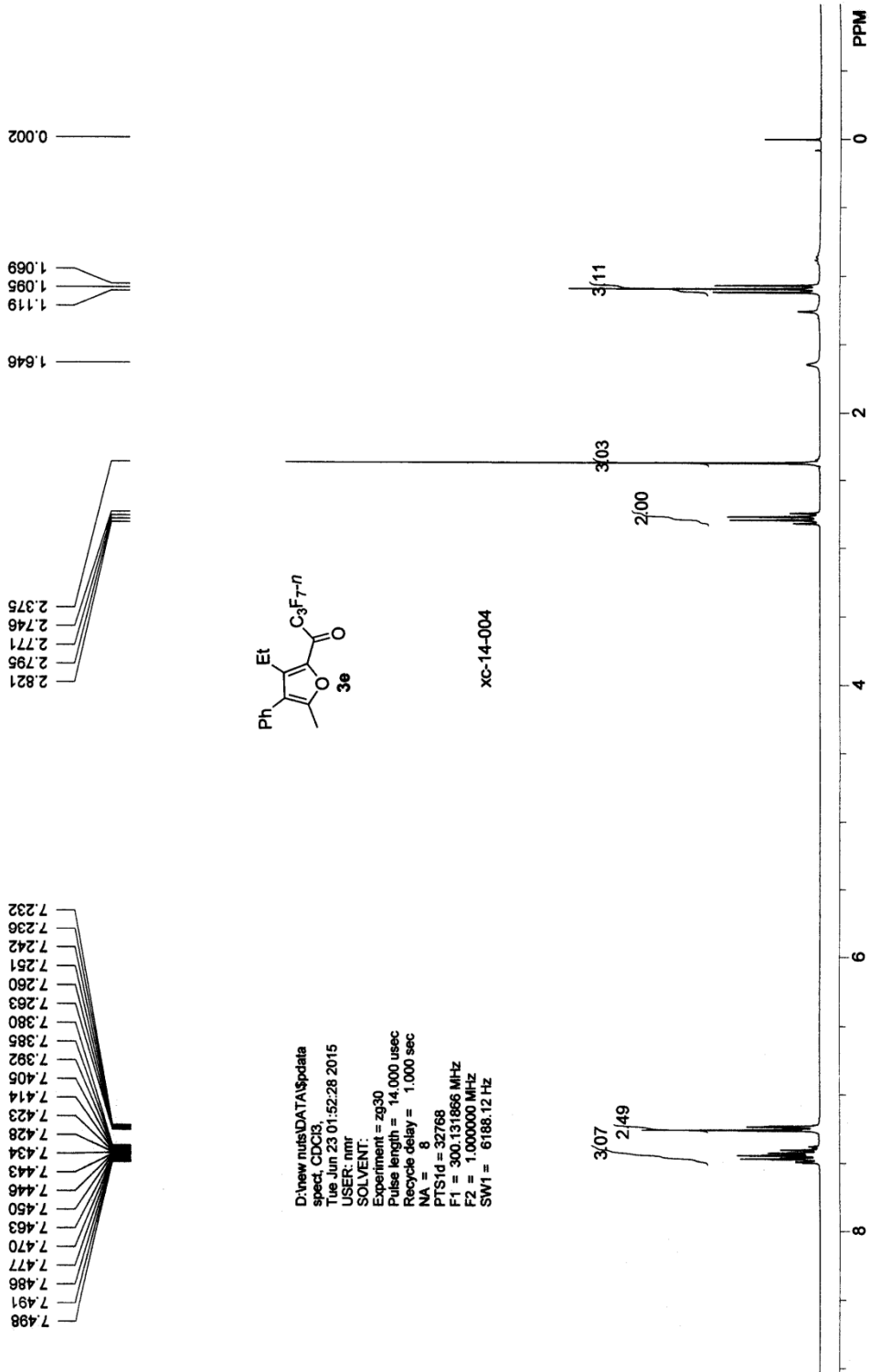


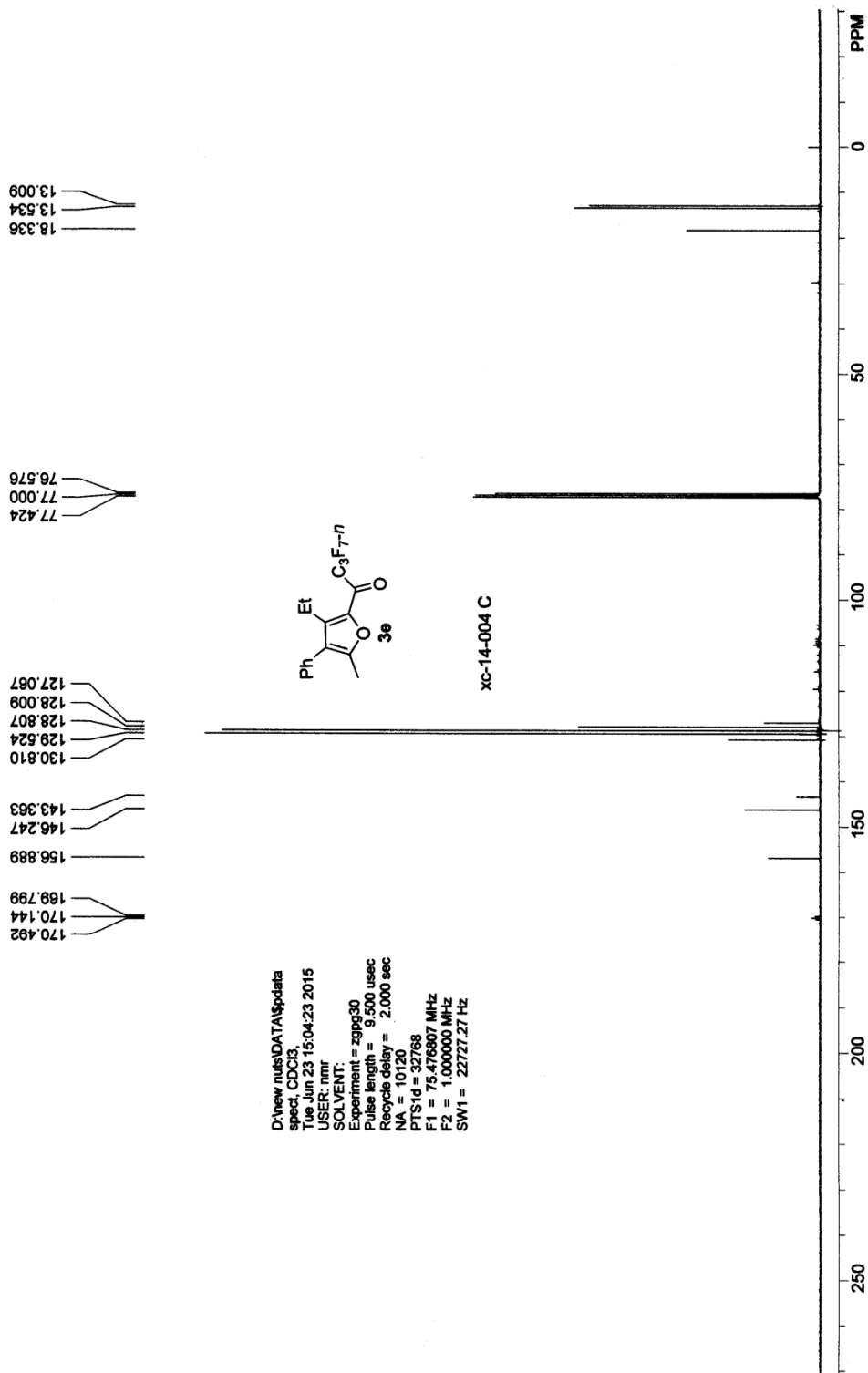
D:\new nus\DATA\sp\data
 spect, CDC13,
 Sat Jun 20 16:10:24 2015
 USER: nmr
 SOLVENT:
 Experiment = zppg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 9117
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

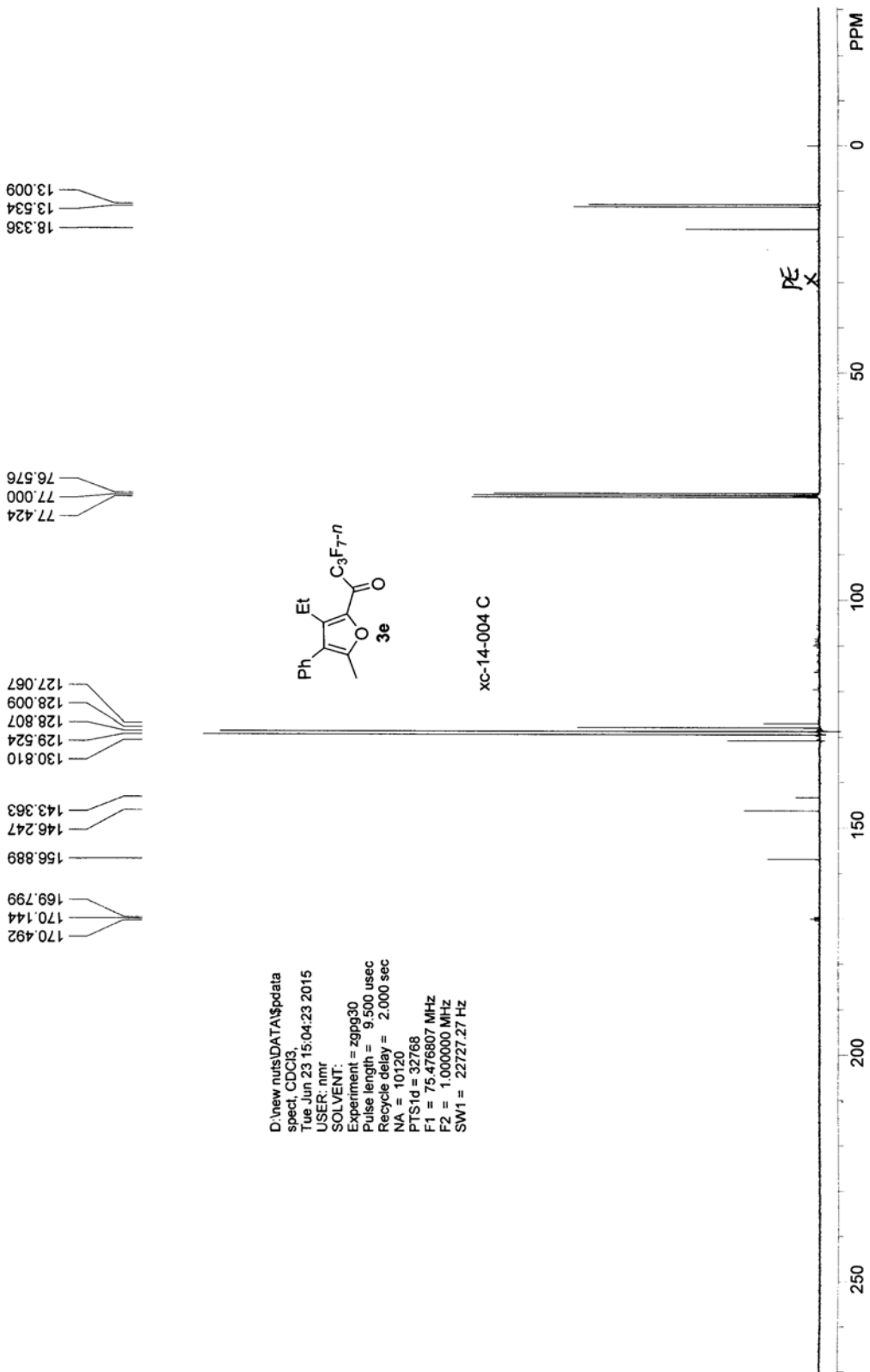


D:\new nuts\DATA\spdata
 spect, CDCl3,
 Sat Jun 20 16:10:24 2015
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 9117
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

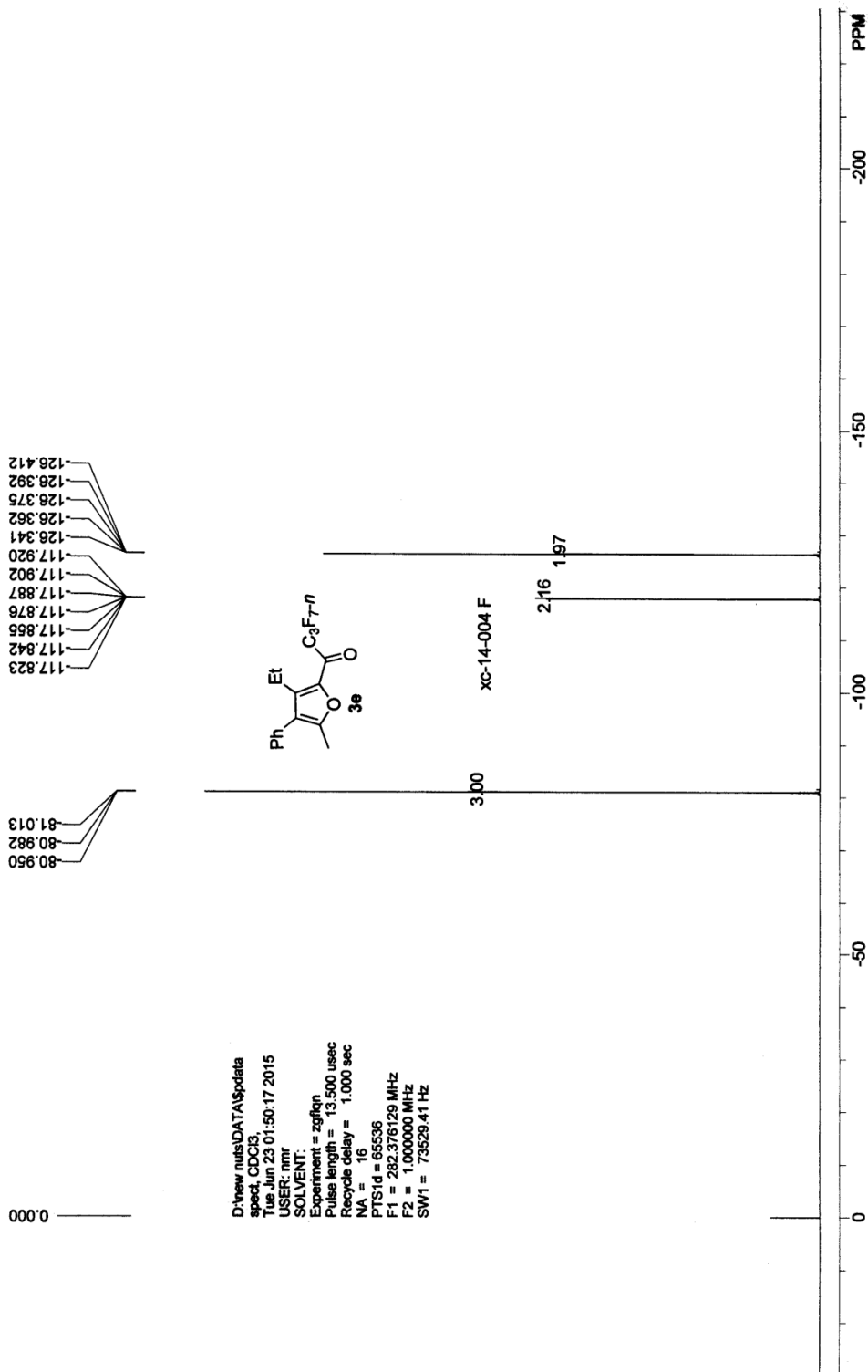


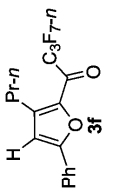
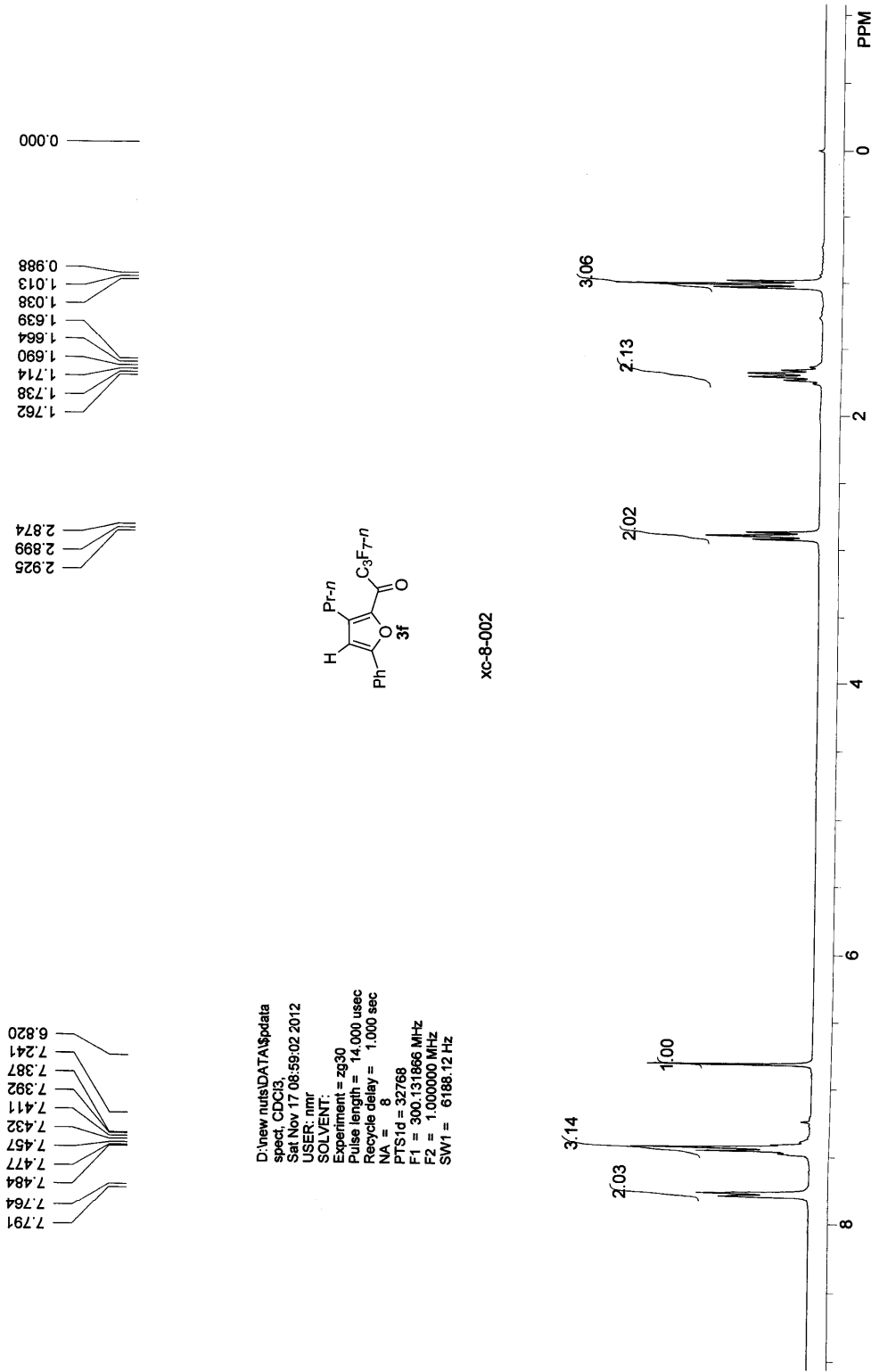






D:\new_nuis\DATA\p\data
 spect, CDCI3,
 Tue Jun 23 15:04:23 2015
 USER: nmr
 SOLVENT:
 Experiment = zpgg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 10120
 P1S1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz





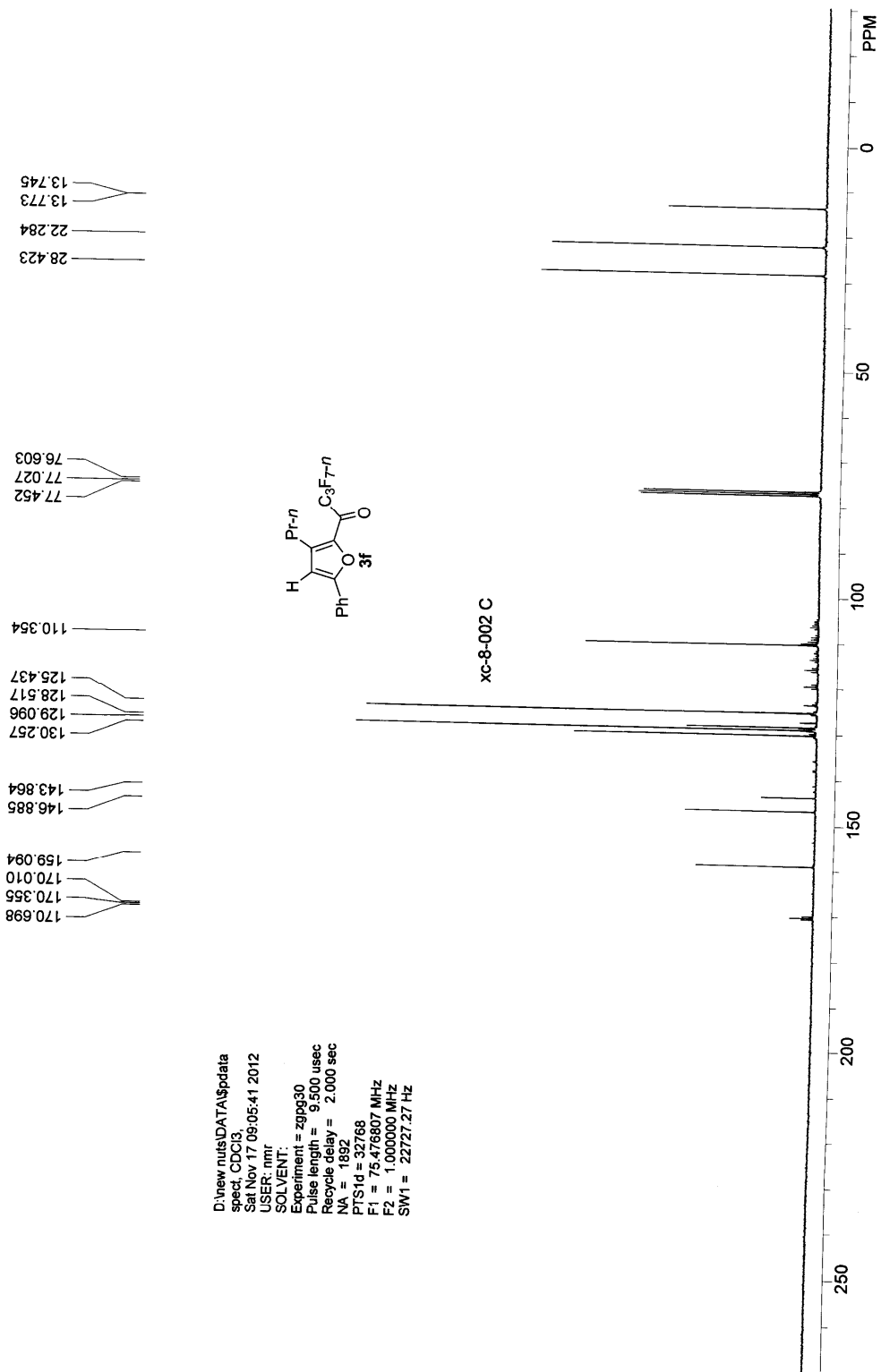
xc-8-002

D:\new nuts\DATA\pdata
 spect, CDCI3,
 Sat Nov 17 08:59:02 2012
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz

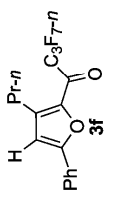
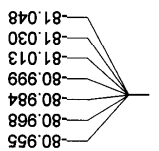
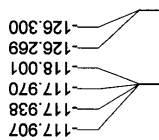
7.791
 7.764
 7.484
 7.477
 7.457
 7.432
 7.411
 7.392
 7.387
 7.241
 6.820

2.926
 2.899
 2.874

1.762
 1.738
 1.714
 1.690
 1.664
 1.639
 1.038
 1.013
 0.988



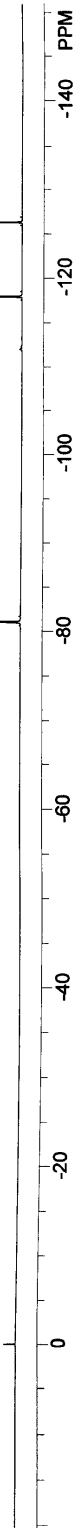
D:\new nuts\DATA\spdata
 spect, CDCI3,
 Sat Nov 17 09:05:41 2012
 USER: nmr
 SOLVENT:
 Experiment = zq9930
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 1892
 P1S1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

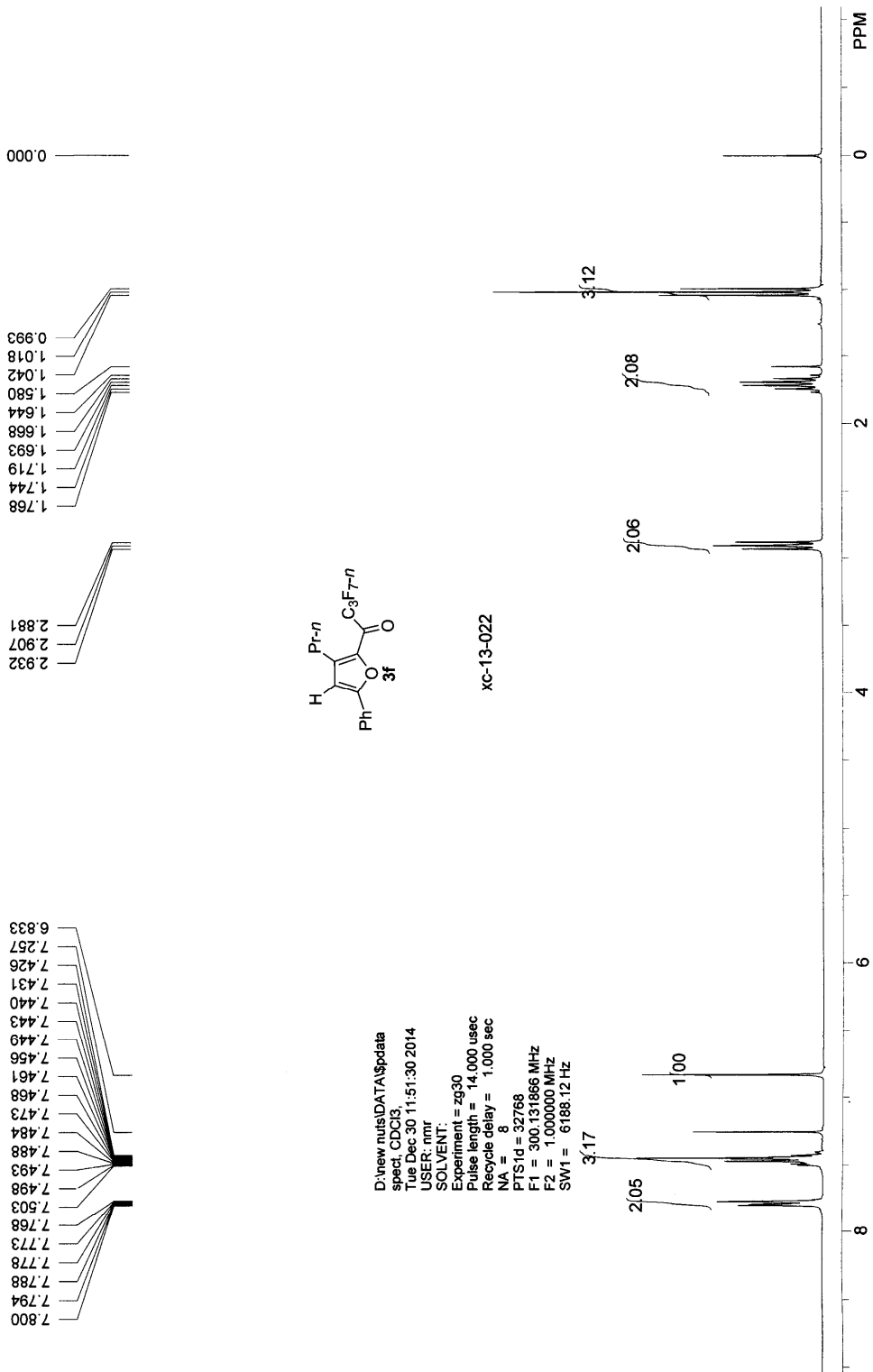


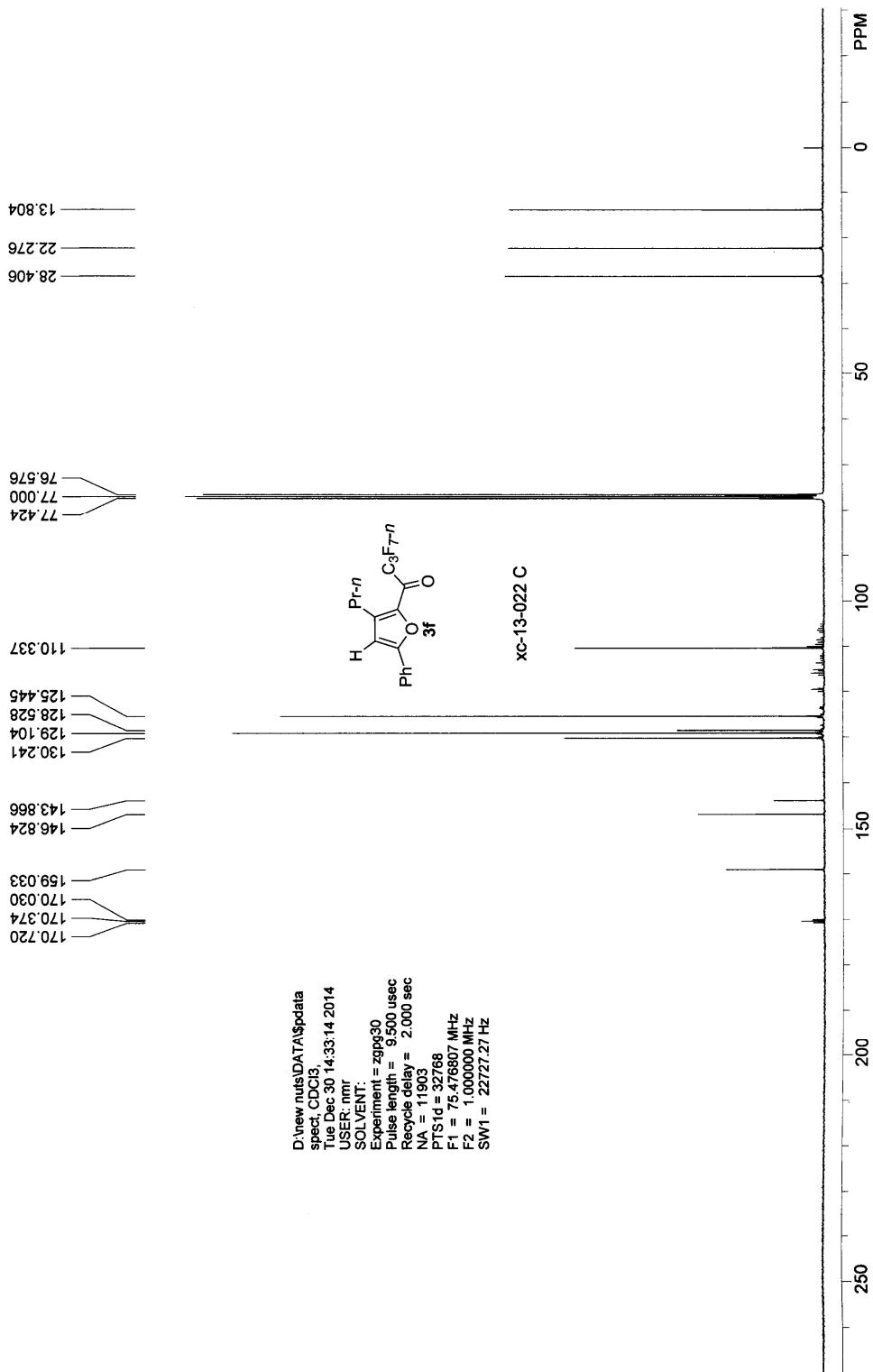
xc-8-002 F

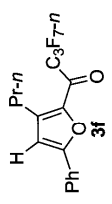
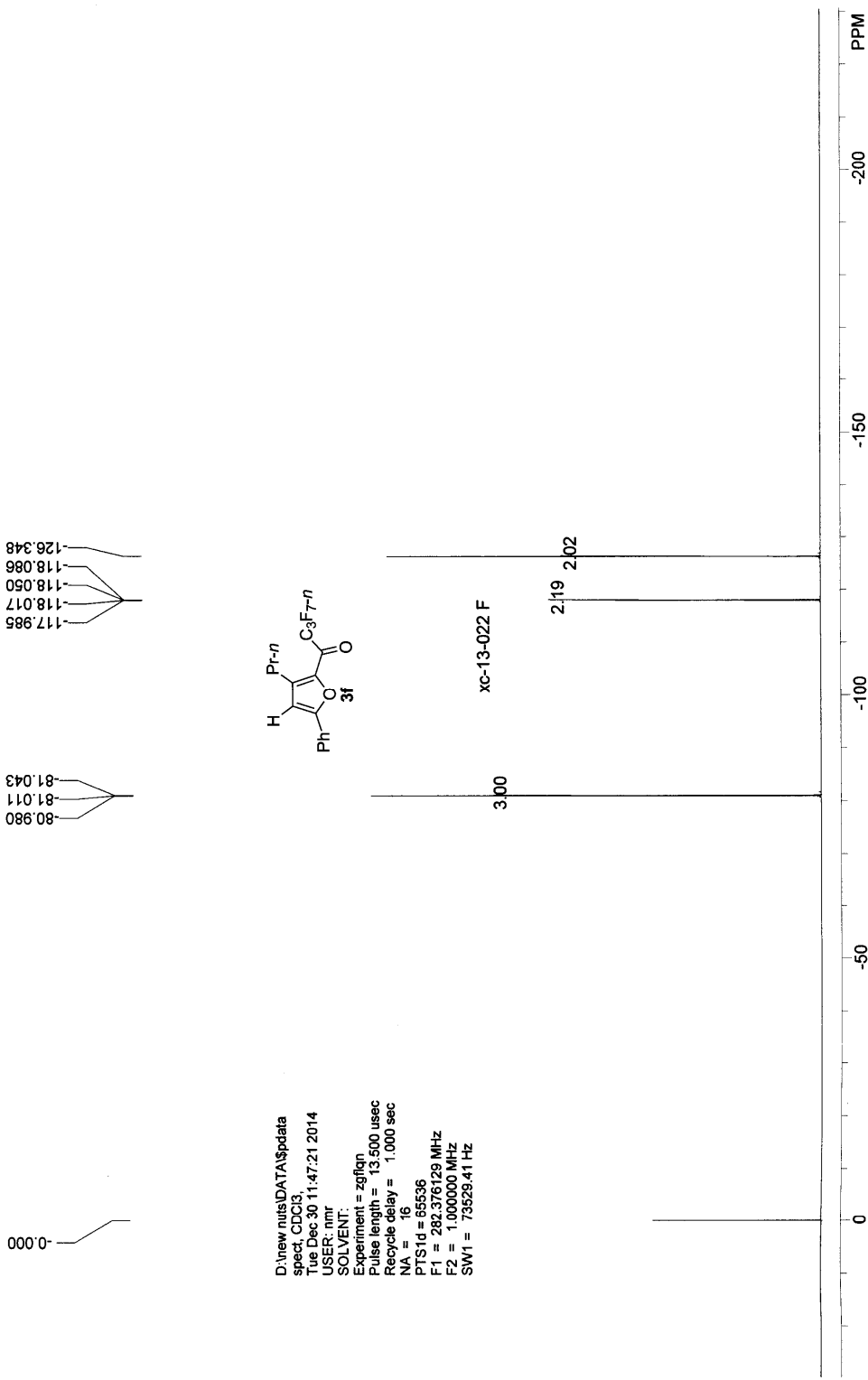
D:\new nuts\DATA\data
spect, CDCl₃,
Sat Nov 17 09:02:54 2012
USER: nmr
SOLVENT:
Experiment = zoffen
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTSD1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

0.000

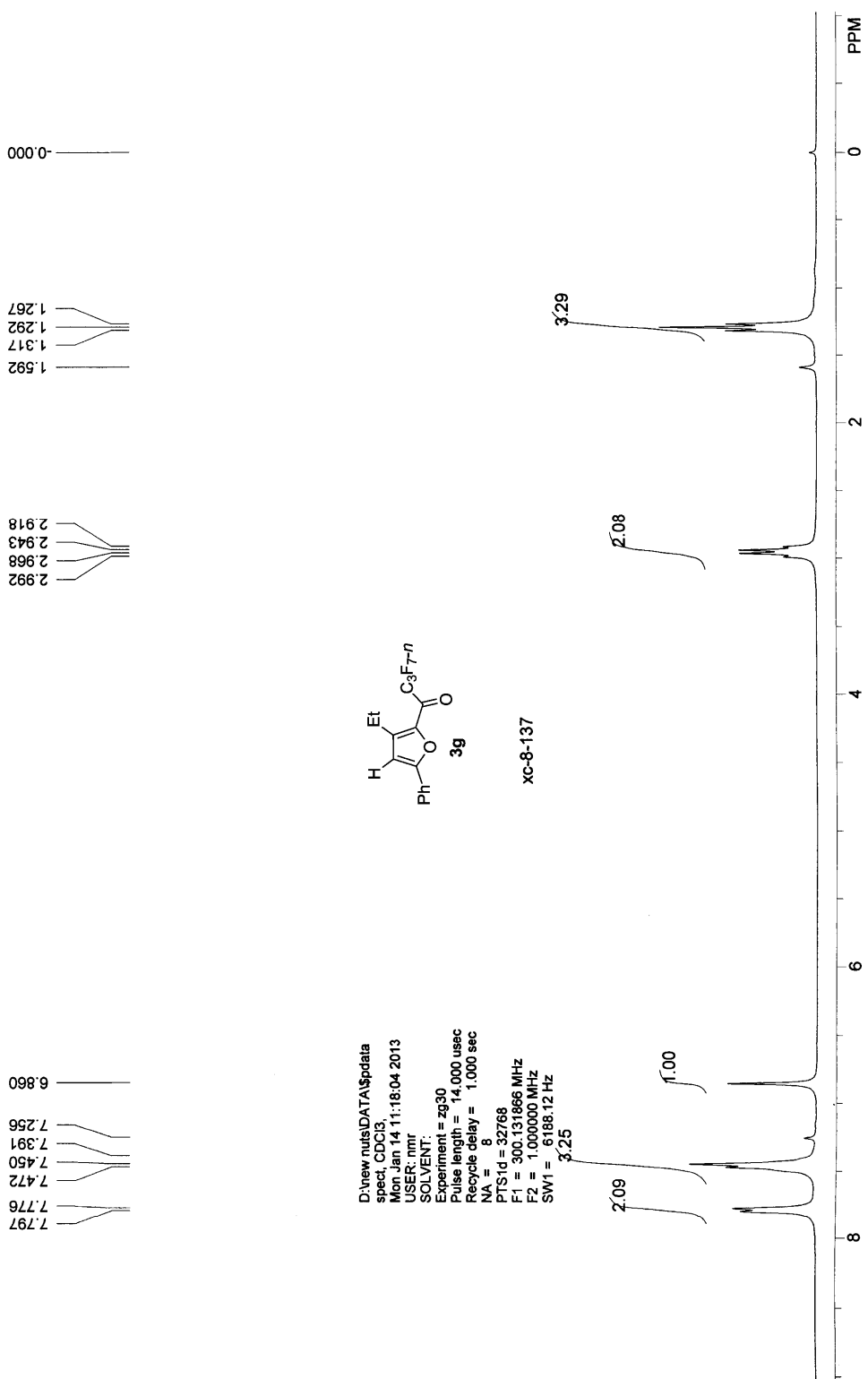


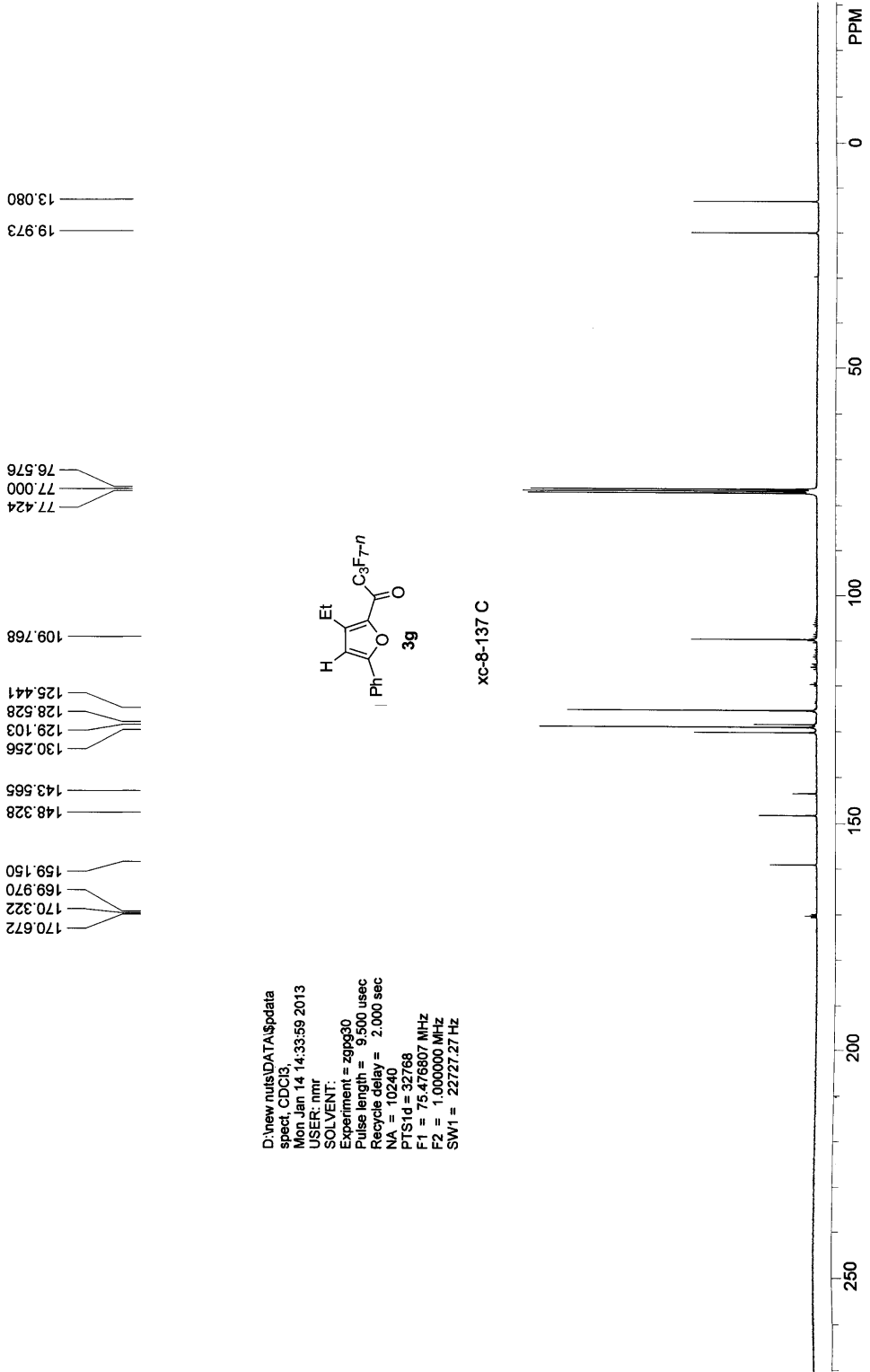




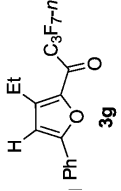


D:\new nusi\DATA\spdata
 spect, CDC13,
 Tue Dec 30 11:47:21 2014
 USER: nmr
 SOLVENT:
 Experiment = zgfgn
 Pulse length = 13.500 usec
 Recycle delay = 1.000 sec
 NA = 16
 PTS1d = 65536
 F1 = 282.376129 MHz
 F2 = 1.000000 MHz
 SW1 = 73529.41 Hz

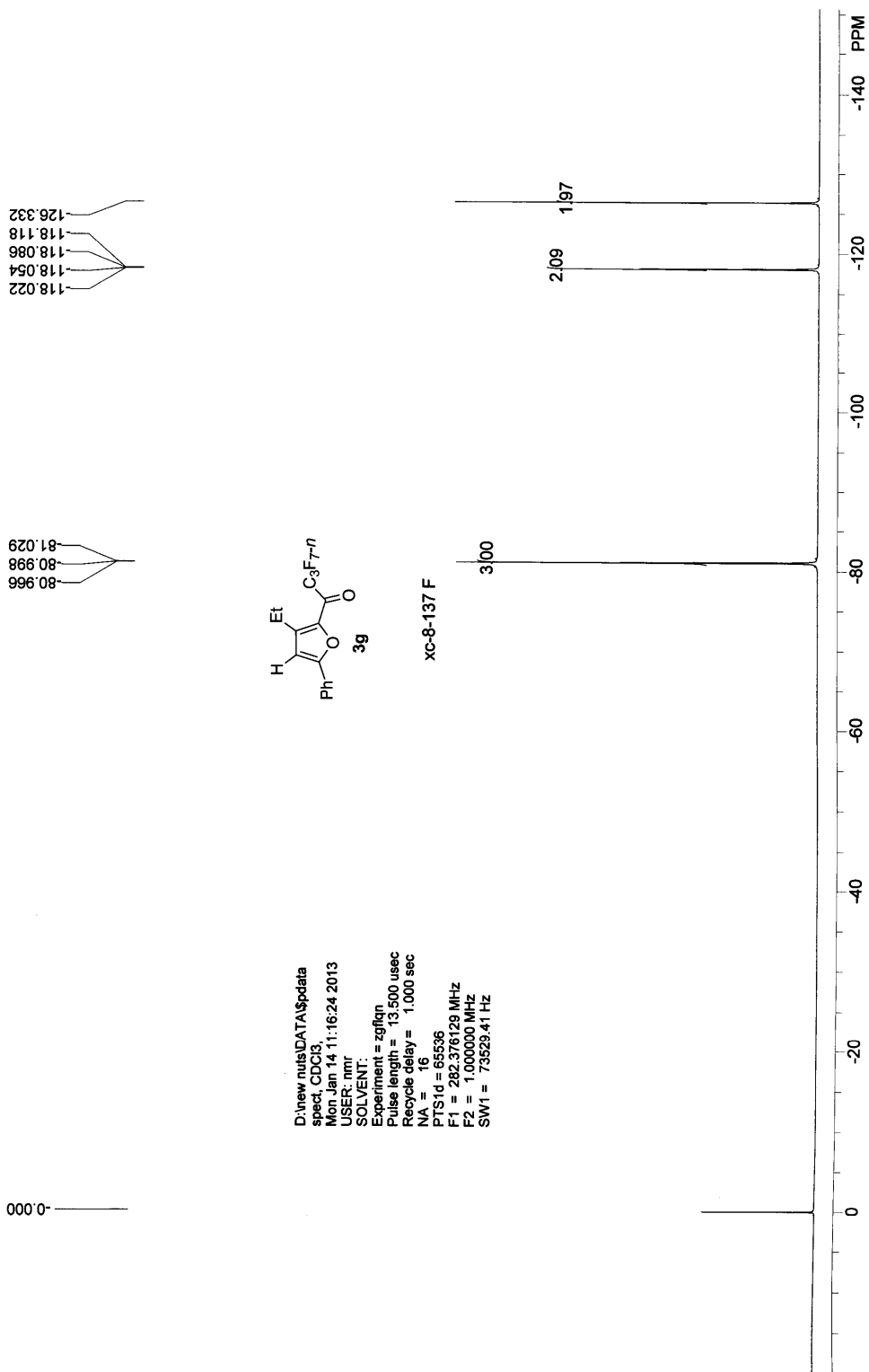


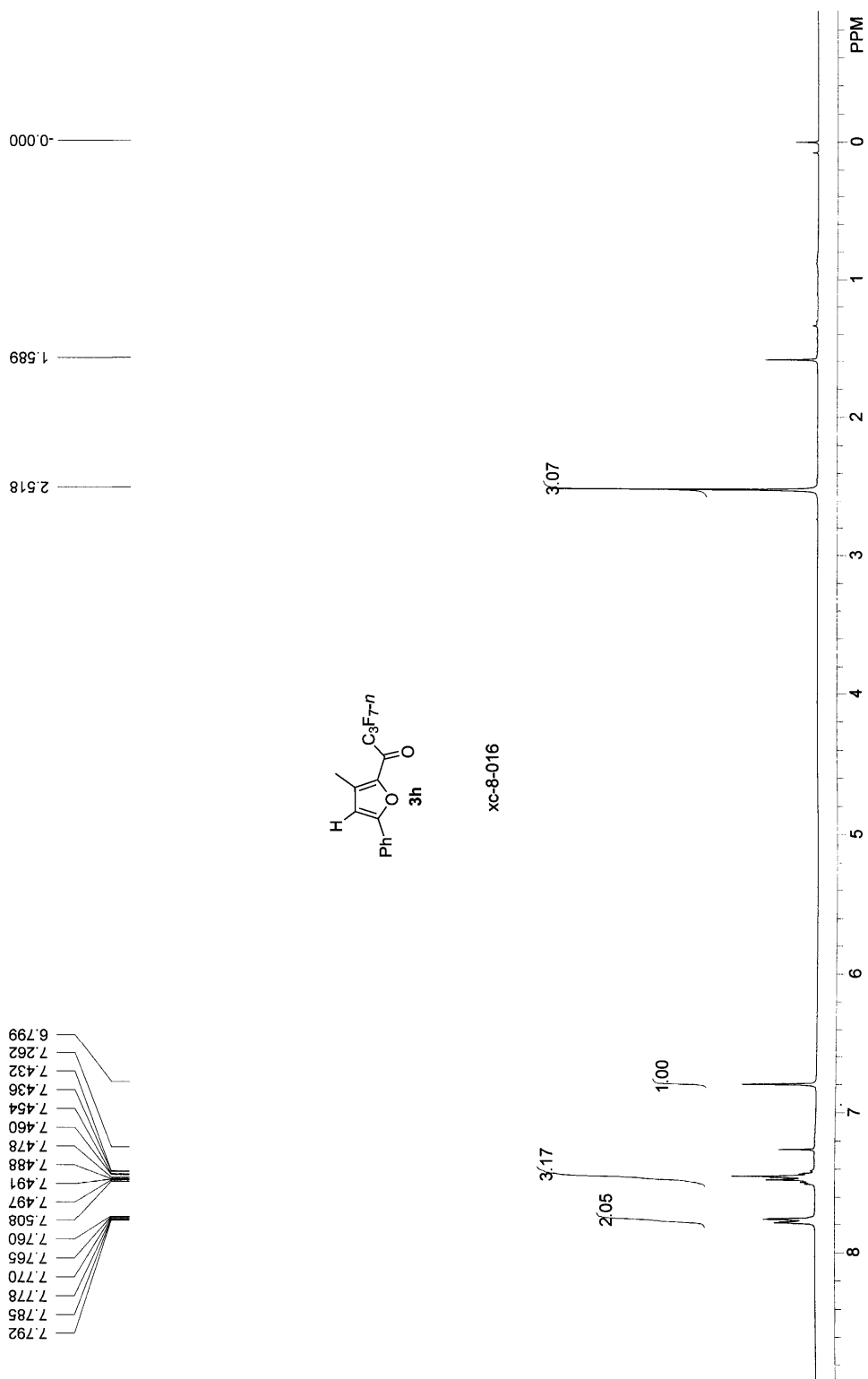


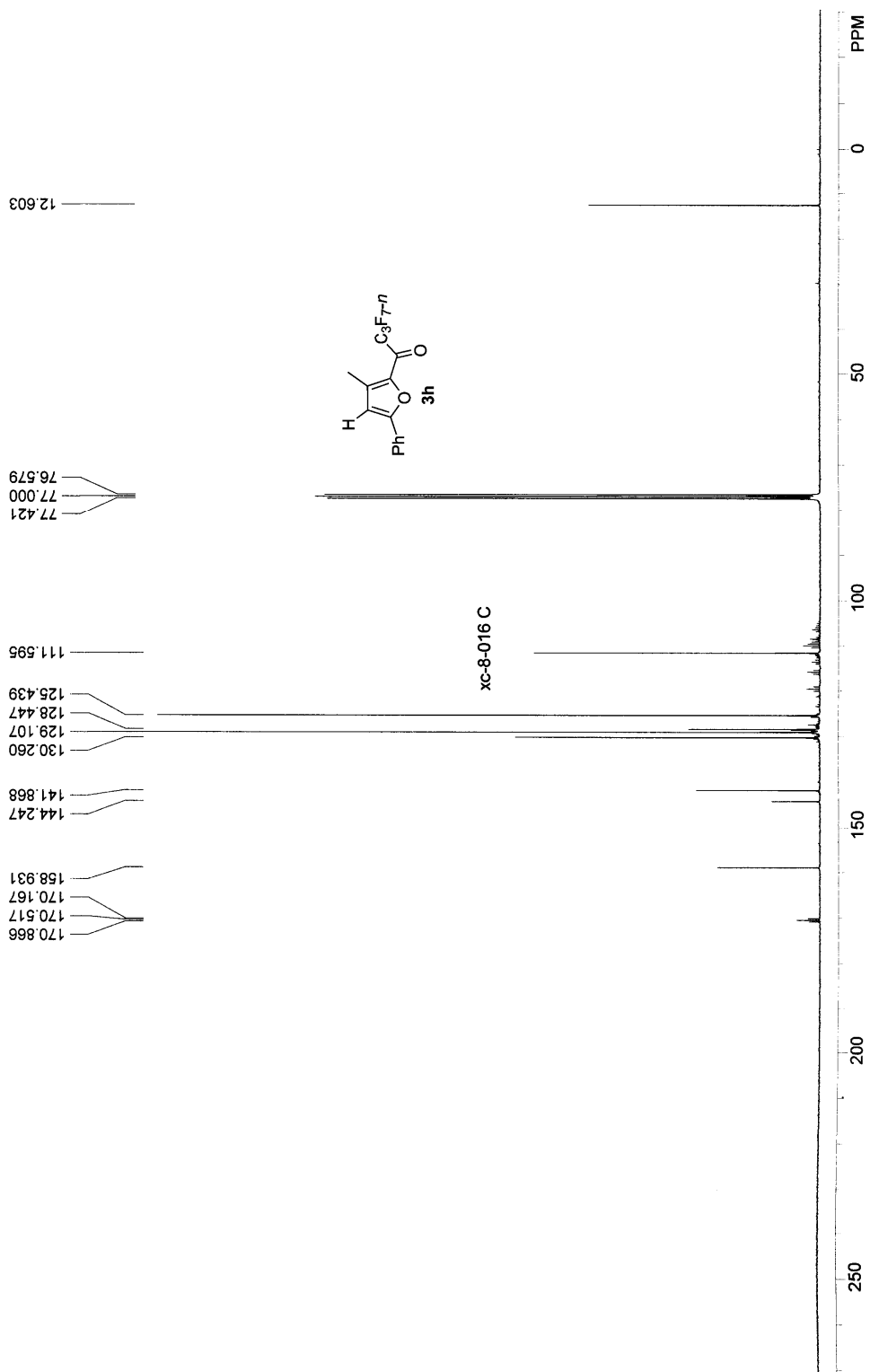
D:\new nuts\DATA\data
spect, CDC13,
Mon Jan 14 14:33:59 2013
USER: nmr
SOLVENT:
Experiment = zppg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10240
PTSD1 = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

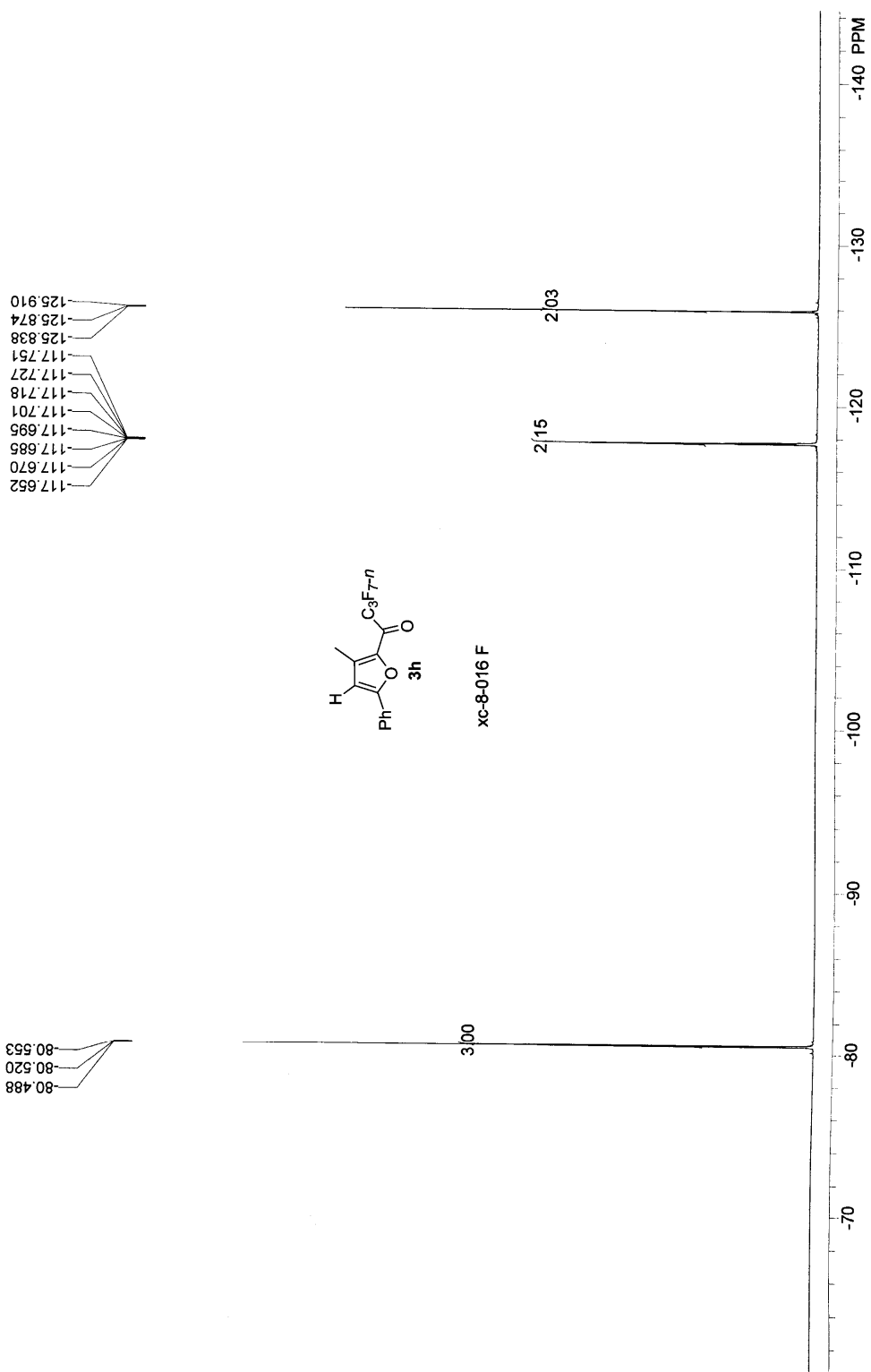


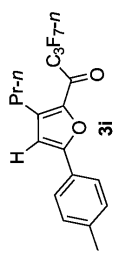
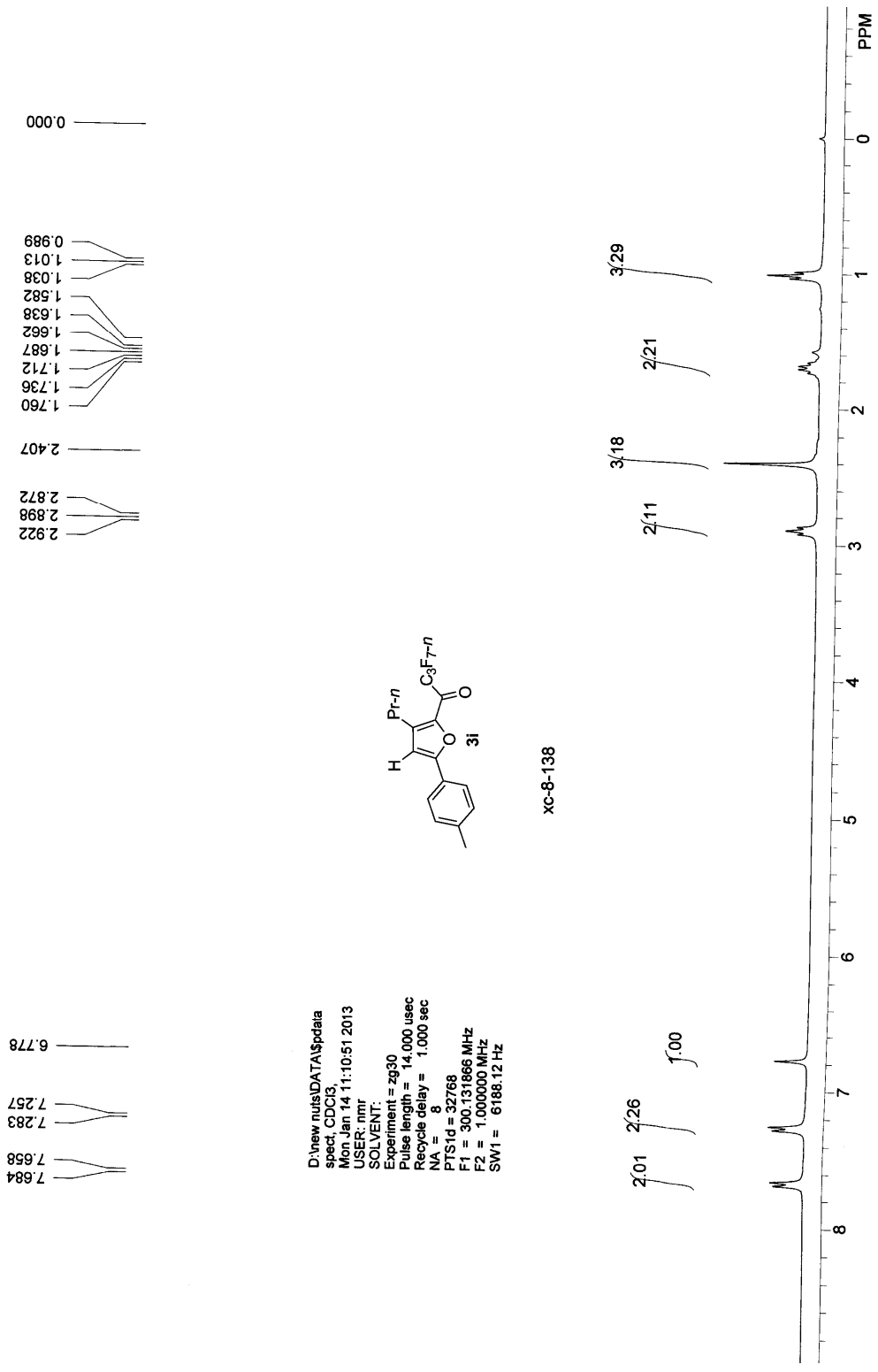
xc-8-137 C





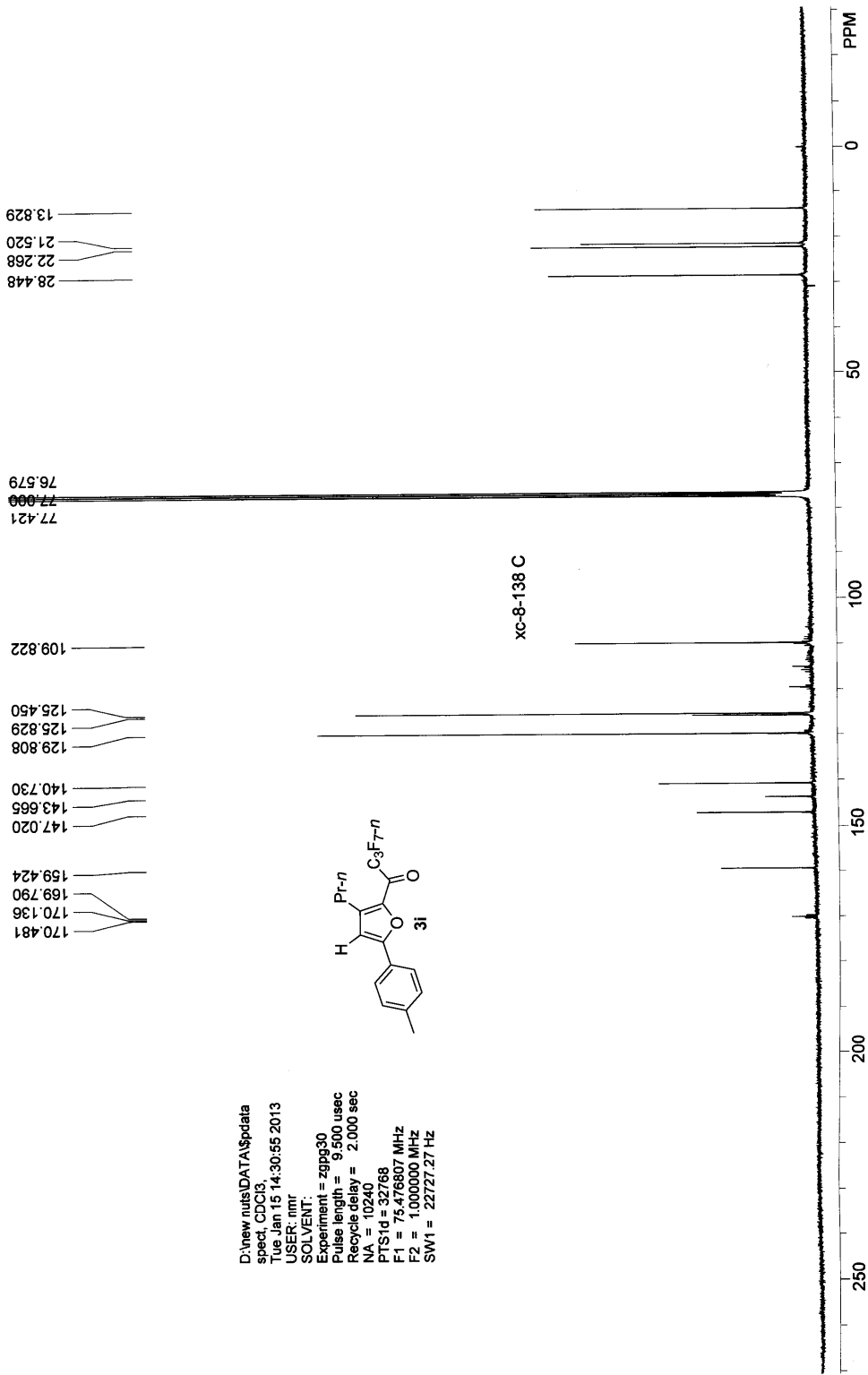


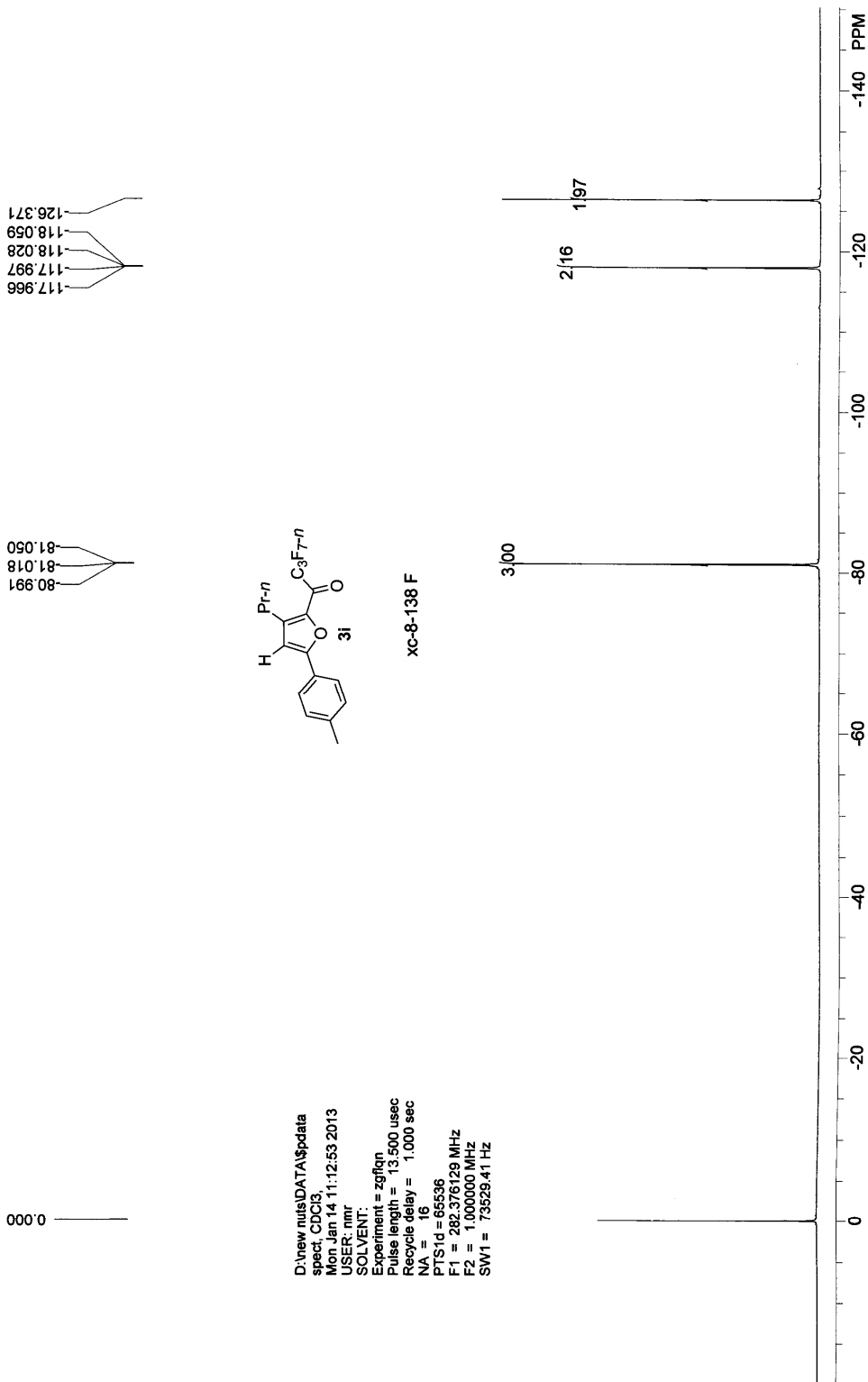


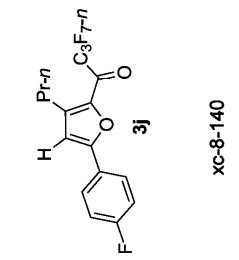
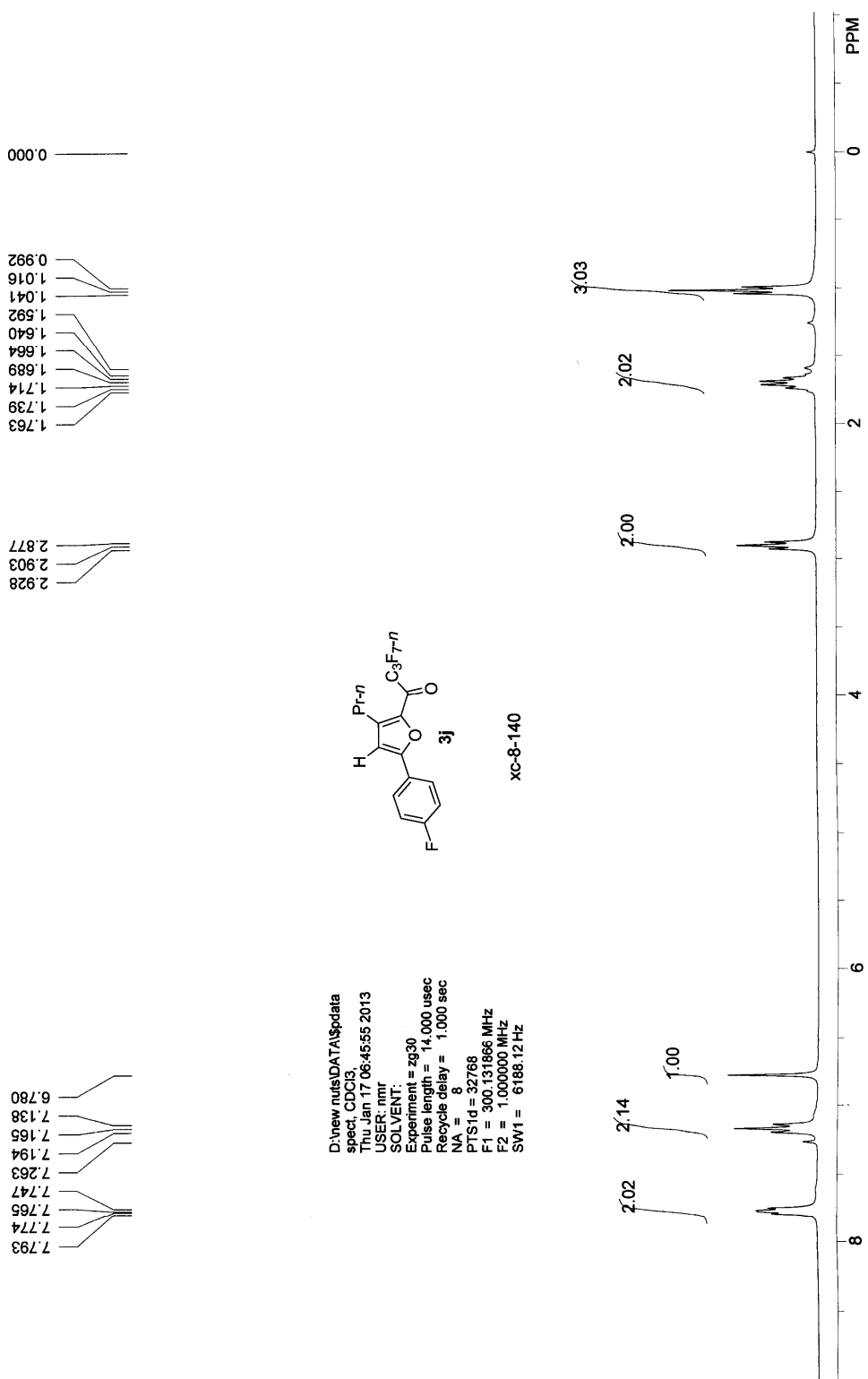


XC-8-138

D:\new nmr\DATA\spdata
 spect, CDCl3,
 Mon Jan 14 11:10:51 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz







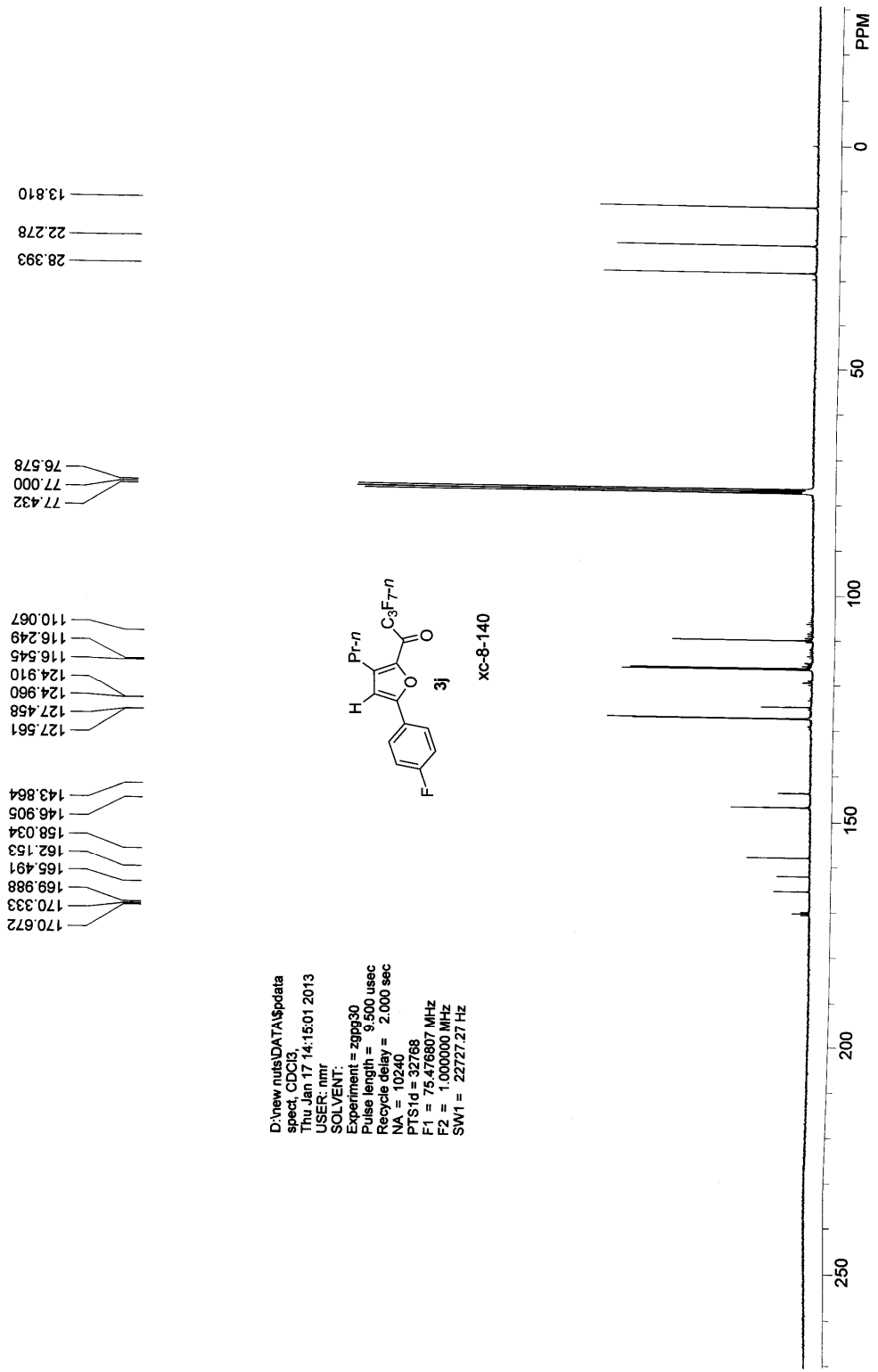
D:\new_nuts\DATA\1Sp\data
 spect, CDC18,
 Thu Jan 17 06:45:55 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

7.793
 7.774
 7.765
 7.747
 7.263
 7.194
 7.165
 7.138
 6.780

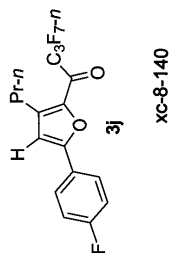
2.928
 2.903
 2.877

1.763
 1.739
 1.714
 1.689
 1.664
 1.640
 1.592
 1.041
 1.016
 0.992

0.000

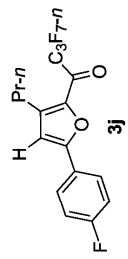


D:\new_nuts\DATA\spdata
 spect, CDCl3,
 Thu Jan 17 14:15:01 2013
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 10240
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



126.361
118.092
118.060
118.028
117.997
109.481
109.466
109.453
109.434
109.416
109.406
109.386

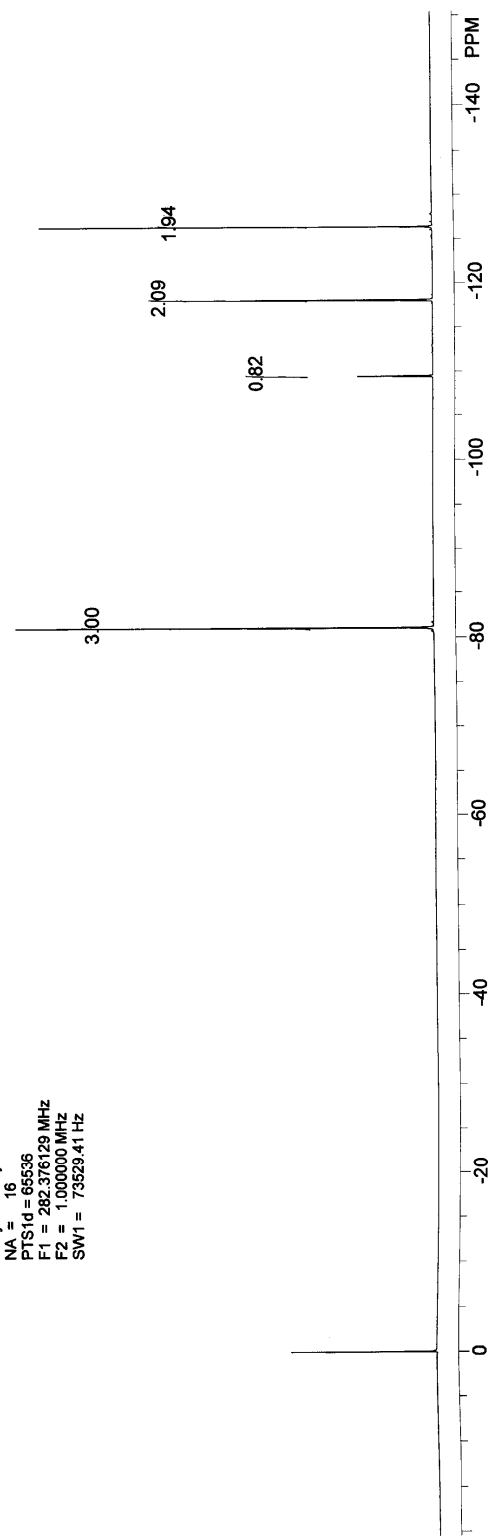
81.030
80.998
80.967

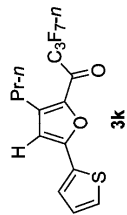
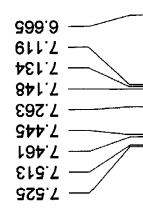
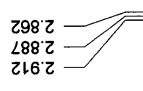
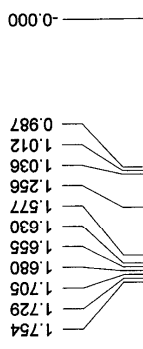


xc-g-140 F

D:\new nuts\DATA\pdata
spect, CDC13,
Thu Jan 17 06:49:33 2013
USER: nmr
SOLVENT:
Experiment = zgfgin
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 66536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

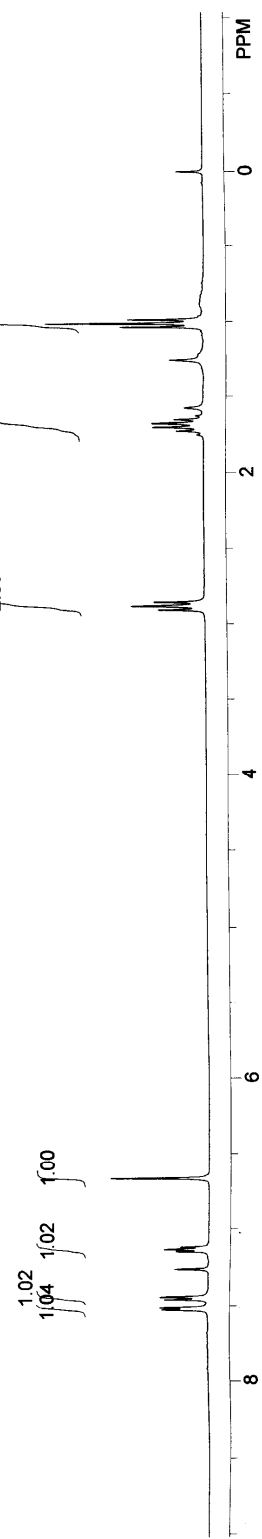
0.000

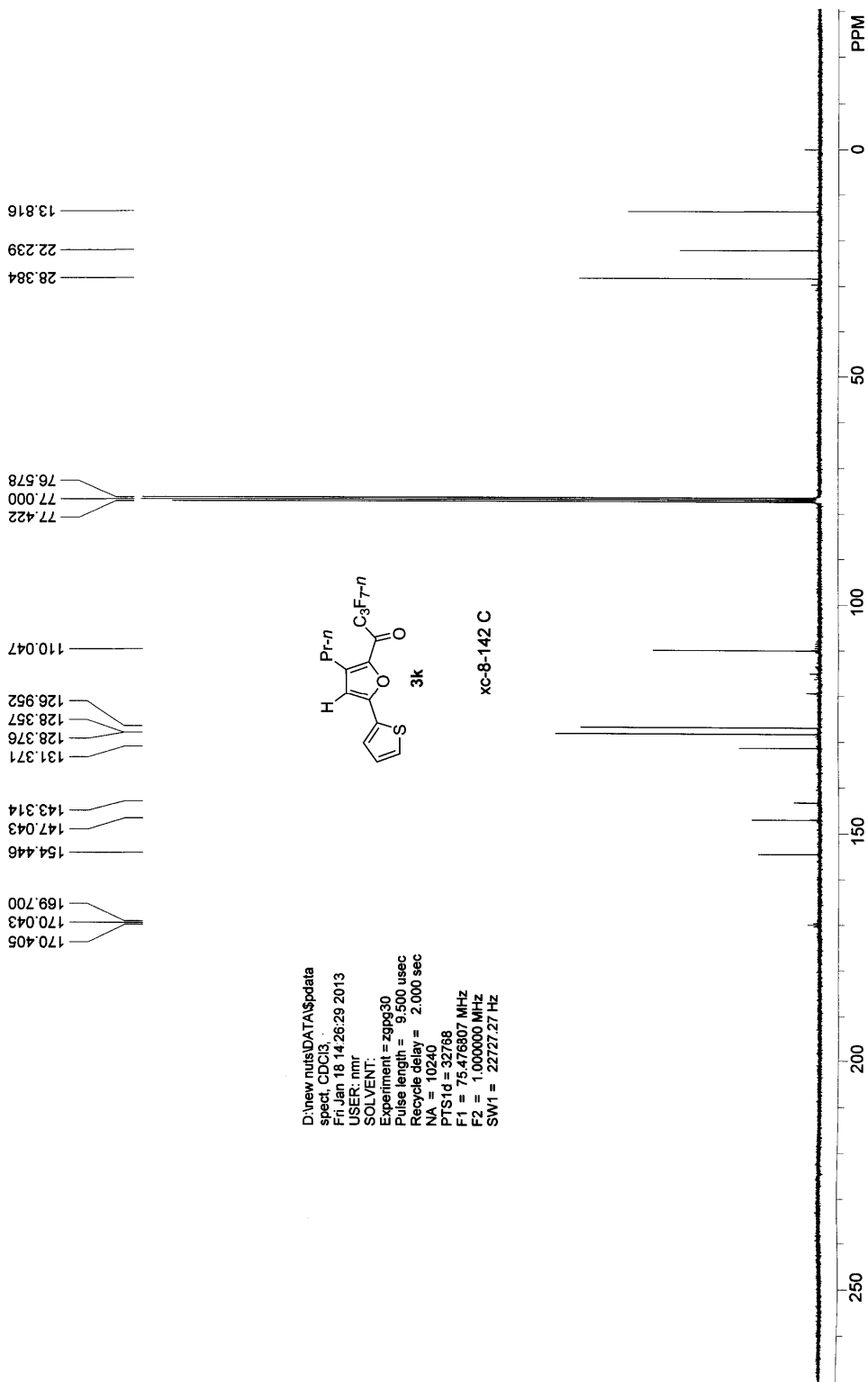




D:\new nuts\DATA\spdata
 spect, CDC13,
 Thu Jan 17 05:48:39 2013
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1S1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

xc-8-142

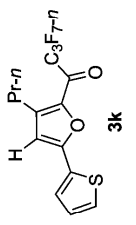




126.373
126.337
126.301
118.066
118.034
118.003
117.972

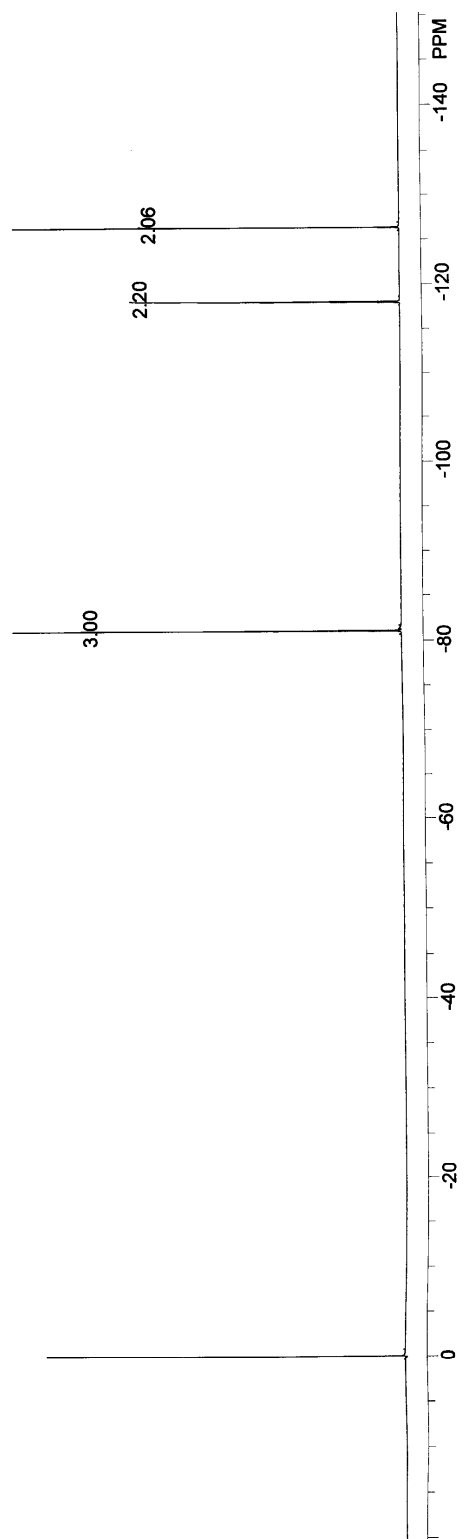
81.081
81.049
81.017

0.000



XC-8-142 F

D:\new nuts\DATA\spdata
spect_CDC13
Thu Jan 17 05:51:23 2013
USER: lmr
SOLVENT:
Experiment = zgfgn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

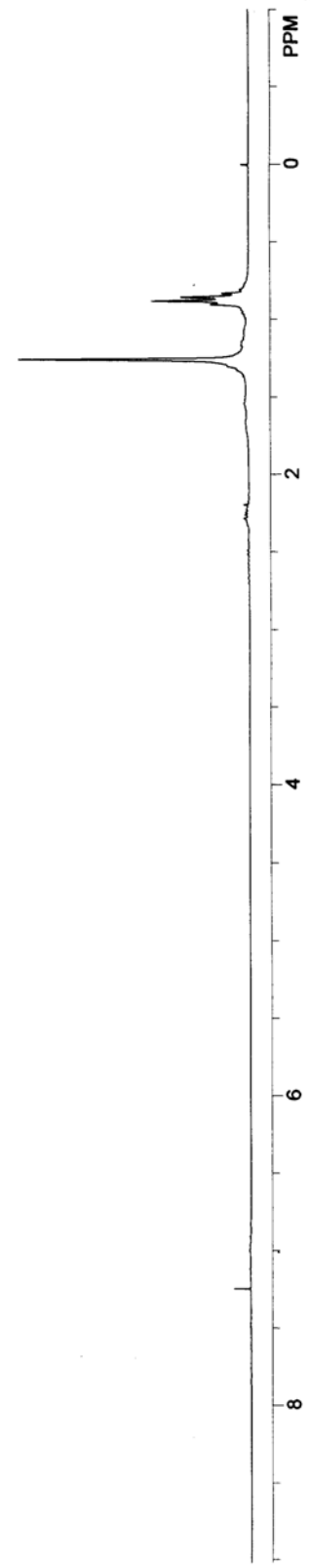


0.000
1.303
1.260
0.902
0.881
0.856
0.831

7.246

D:\new nuts\DATA\spdata
spect, CDCI3,
Wed Jan 14 09:03:54 2009
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

PE



29.708

77.422
77.000
76.576

PE

