Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Electronic Supporting Information

Denovo synthesis of polyhydroxy aminocyclohexanes

Anobick Sar, Sergey Lindeman, and William A. Donaldson*

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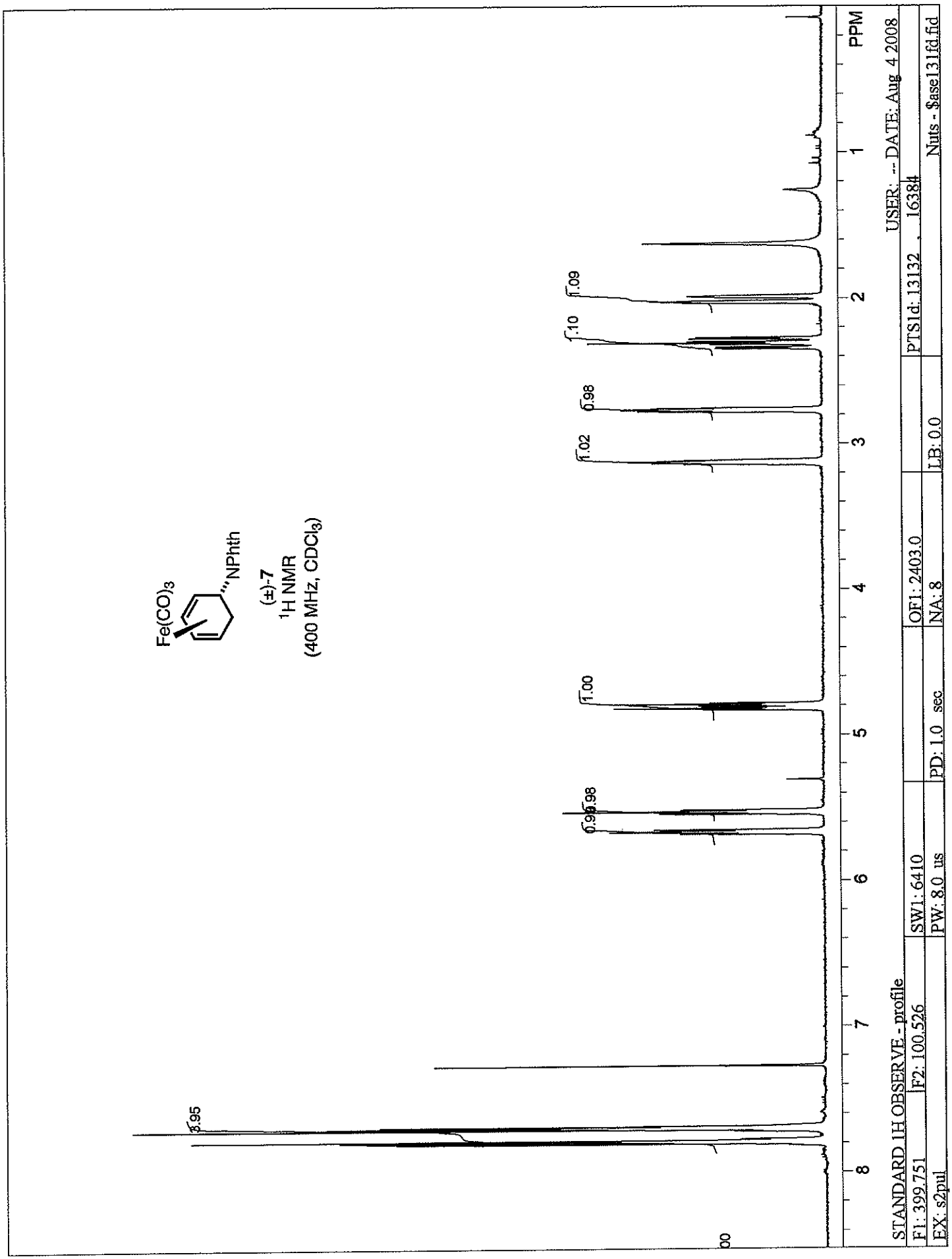
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(±)-7
1H NMR
(400 MHz, CDCl3)
3.95
0.99
0.98
1.00
1.02
0.98
1.10
1.09
PPM
1
2
3
4
5
6
7
8
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F2: 100.526
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OF1: 2403.0
PTS1d: 13132 , 16384
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PD: 1.0 sec
NA: 8
LB: 0.0
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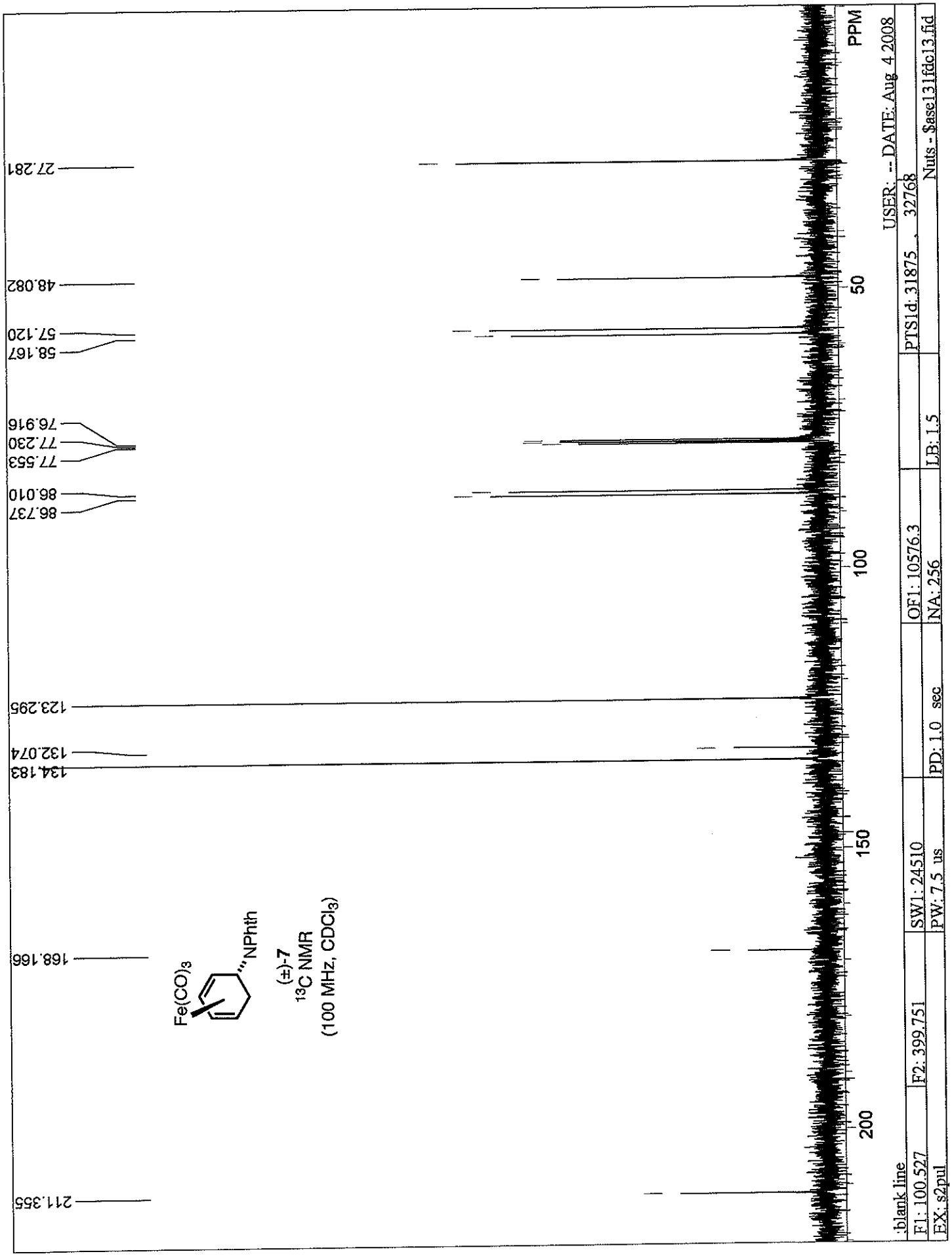

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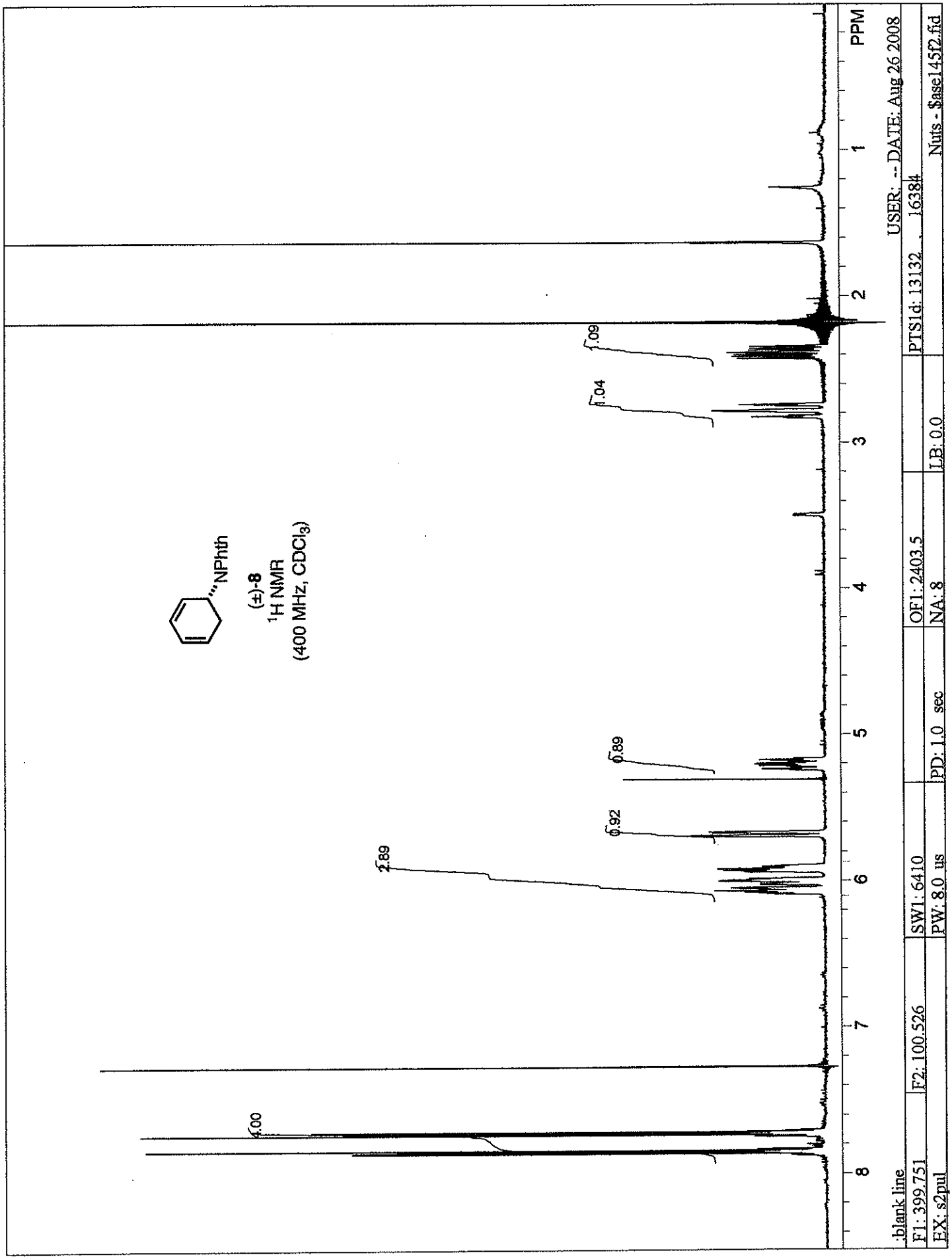

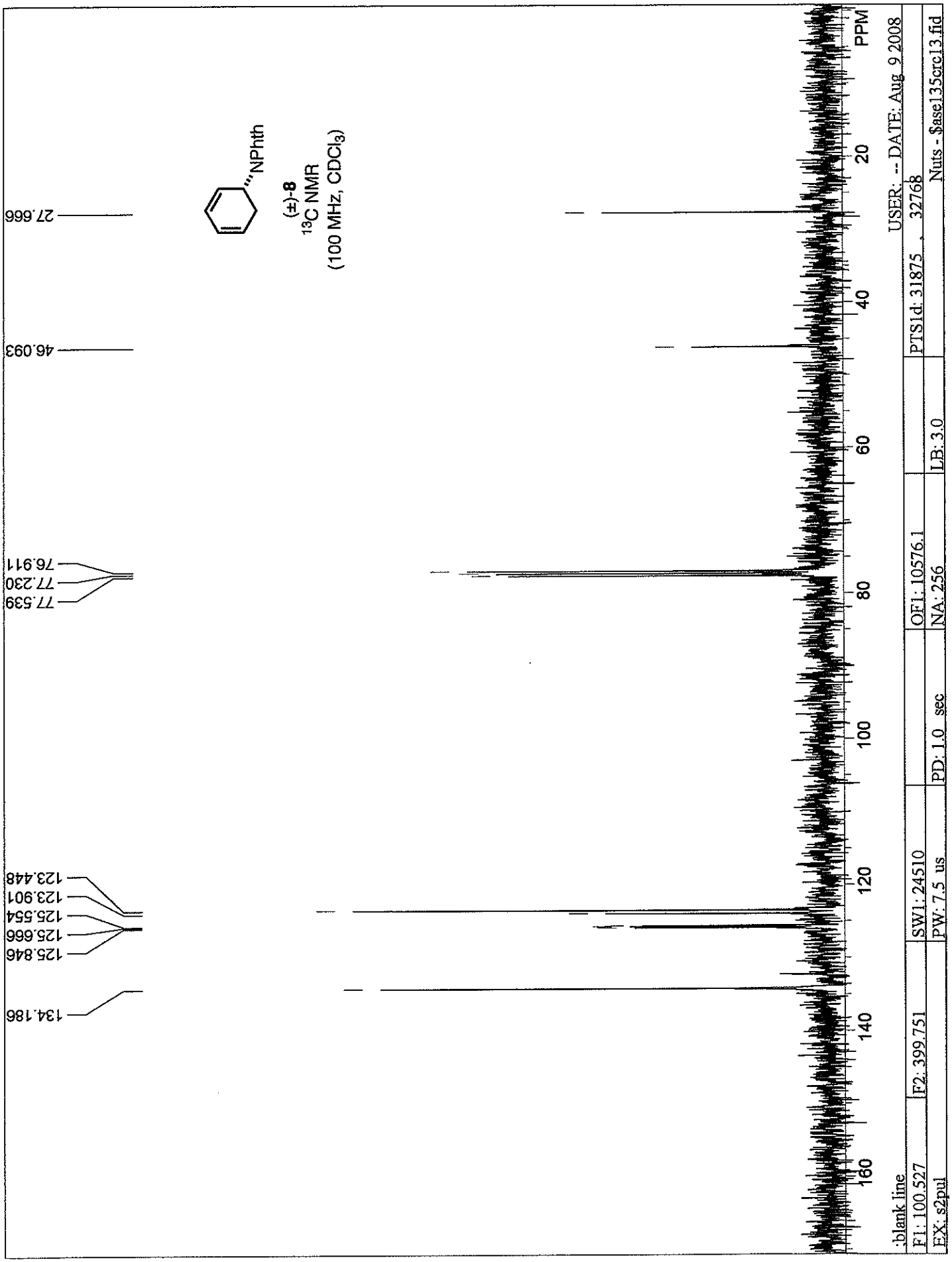
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(±)-8
^{13}C NMR
(100 MHz, $CDCl_3$)
27.666
46.093
76.911
77.230
77.539
123.448
123.901
125.554
125.666
125.846
134.186
PPM
USER: -- DATE: Aug 9 2008
PTS1d: 31875 , 32768
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LB: 3.0
NA: 256
SW1: 24510
PD: 1.0 sec
PW: 7.5 us
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F1: 100.527
EX: s2pul
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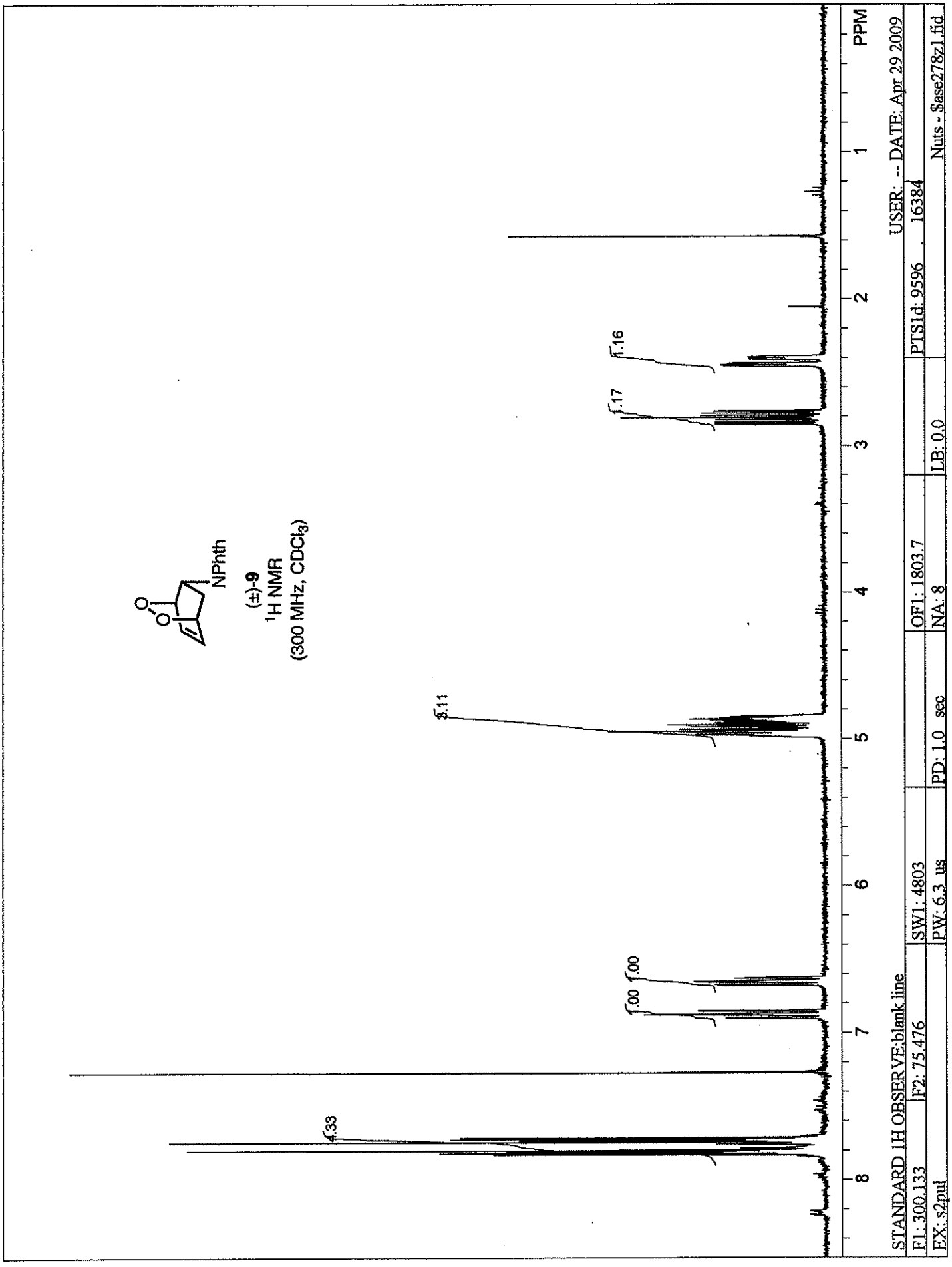

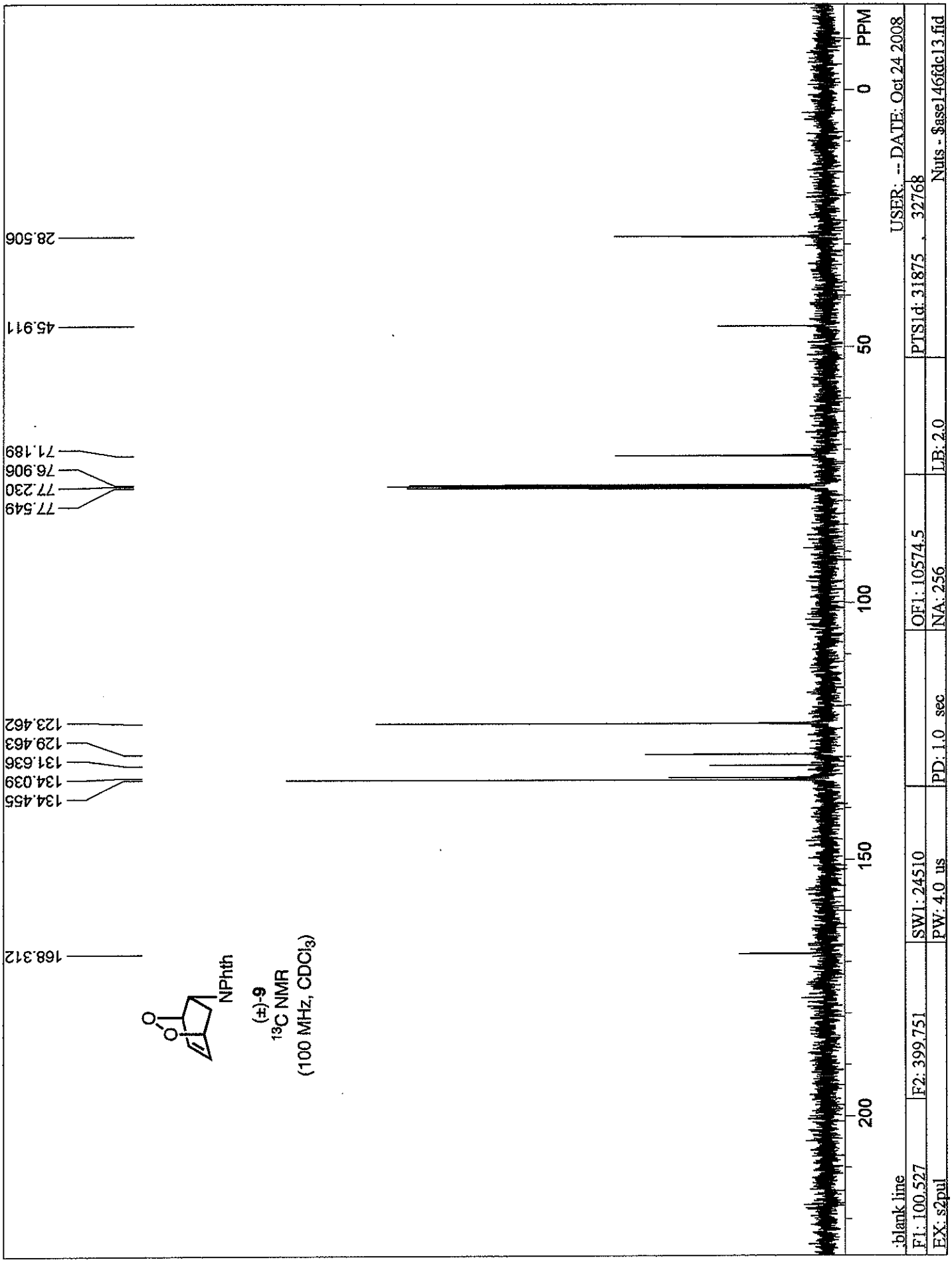
NPhth
(±)-9
^{13}C NMR
(100 MHz, $CDCl_3$)
168.312
134.455
134.039
131.636
129.463
123.462
77.549
77.230
76.906
71.189
45.911
28.506
PPM
USER: -- DATE: Oct 24 2008
blank line
F1: 100.527
F2: 399.751
SW1: 24510
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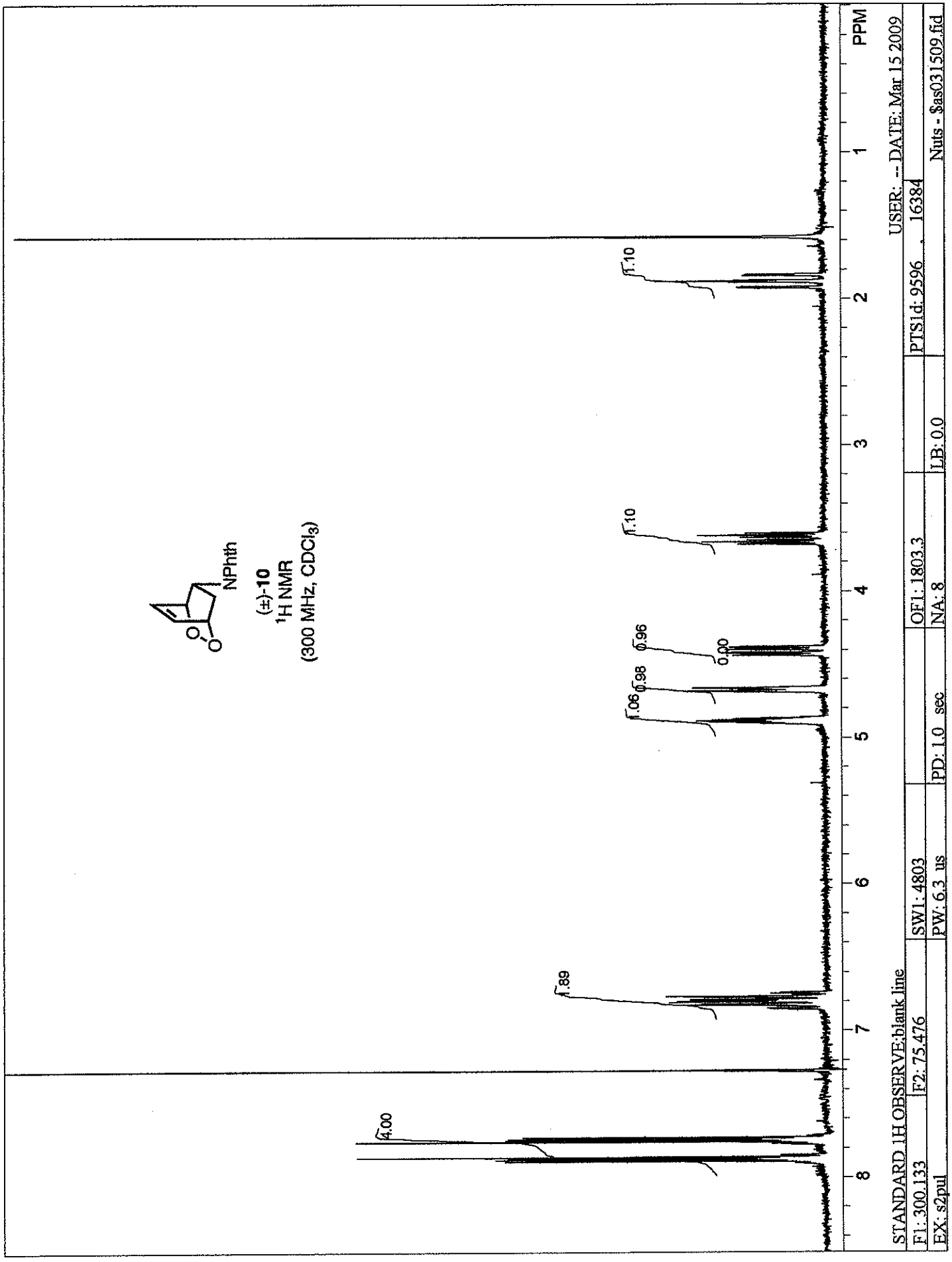
Nuts - $ase146fdc13.fid


NPhth
(±)-10
^{1}H NMR
(300 MHz, CDCl$_3$)
4.00
1.89
1.10
1.10
1.06
0.98
0.96
0.00
PPM
USER: -- DATE: Mar 15 2009
STANDARD 1H OBSERVE:blank line
F1: 300.133
F2: 75.476
SW1: 4803
OF1: 1803.3
PTS1d: 9596
16384
EX: s2pul
PW: 6.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $as031509.fid

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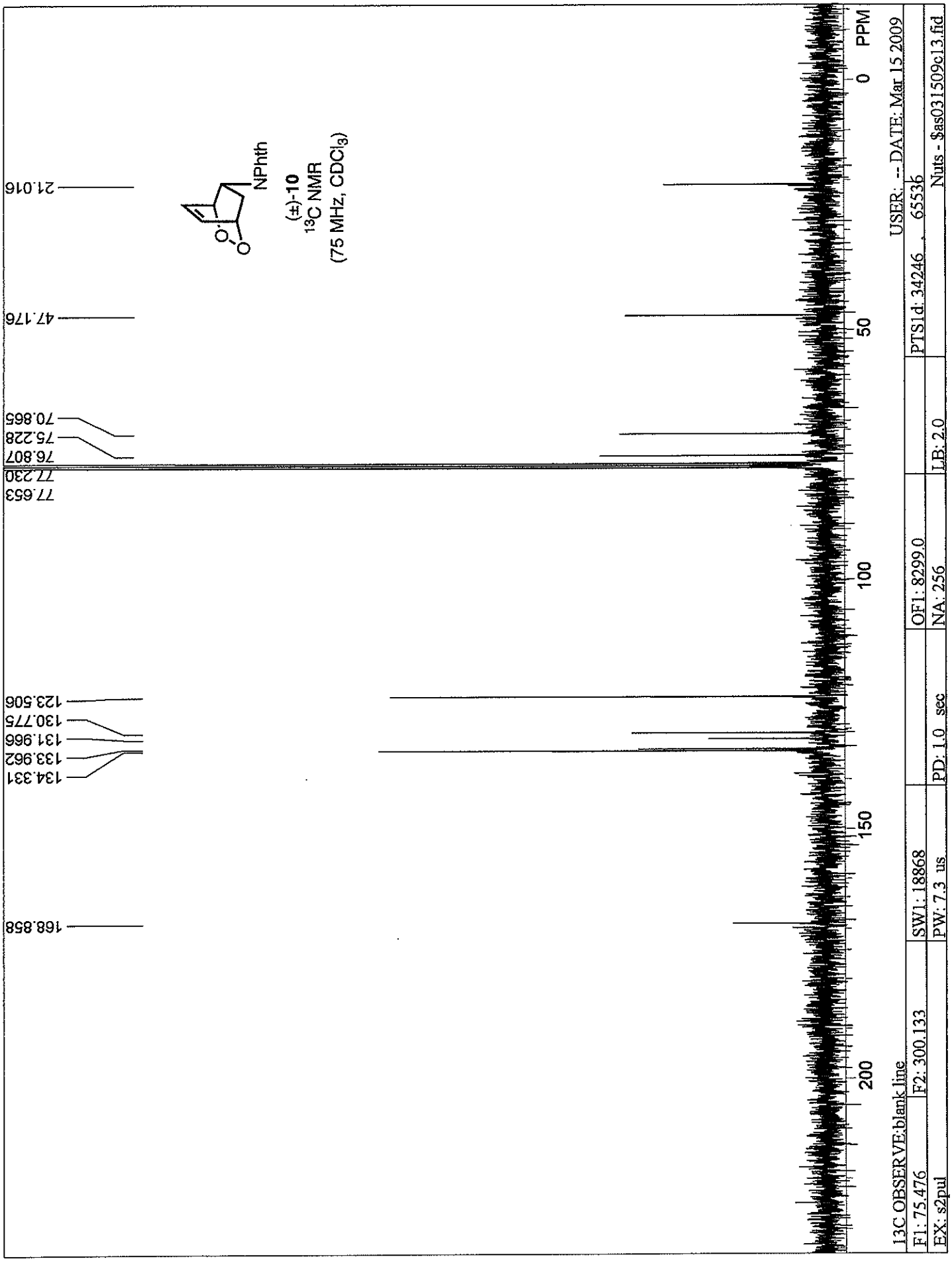

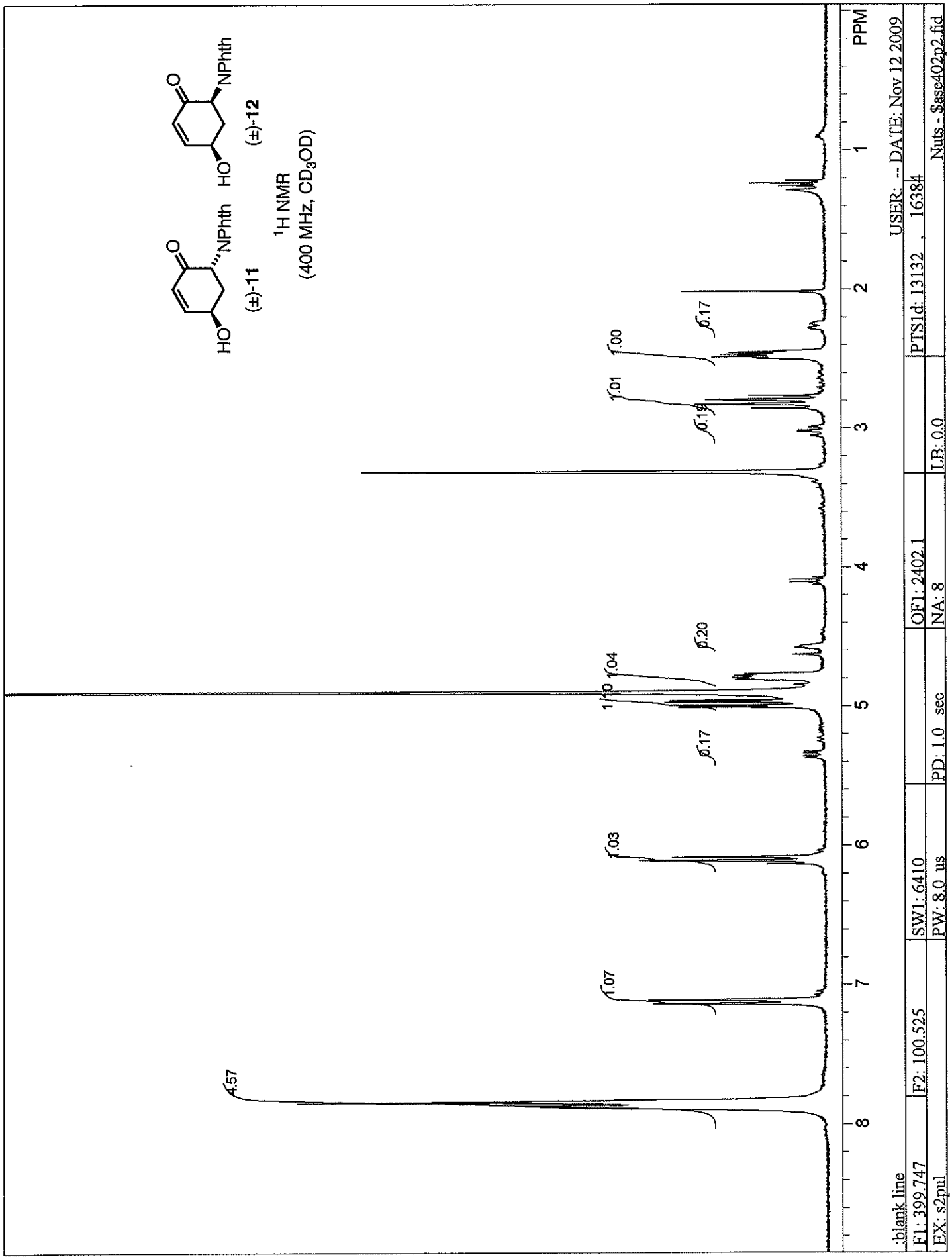
(±)-11
(±)-12
^{1}H NMR
(400 MHz, CD_3OD)
NPhth
HO
PPM
4.57
1.07
1.03
1.10
1.04
0.17
0.20
0.17
1.01
1.00
0.19
USER: -- DATE: Nov 12 2009
PTS1d: 13132 , 16384
Nuts - $ase402p2.fid
blank line
F1: 399.747
F2: 100.525
SW1: 6410
OF1: 2402.1
EX: s2pul
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.0

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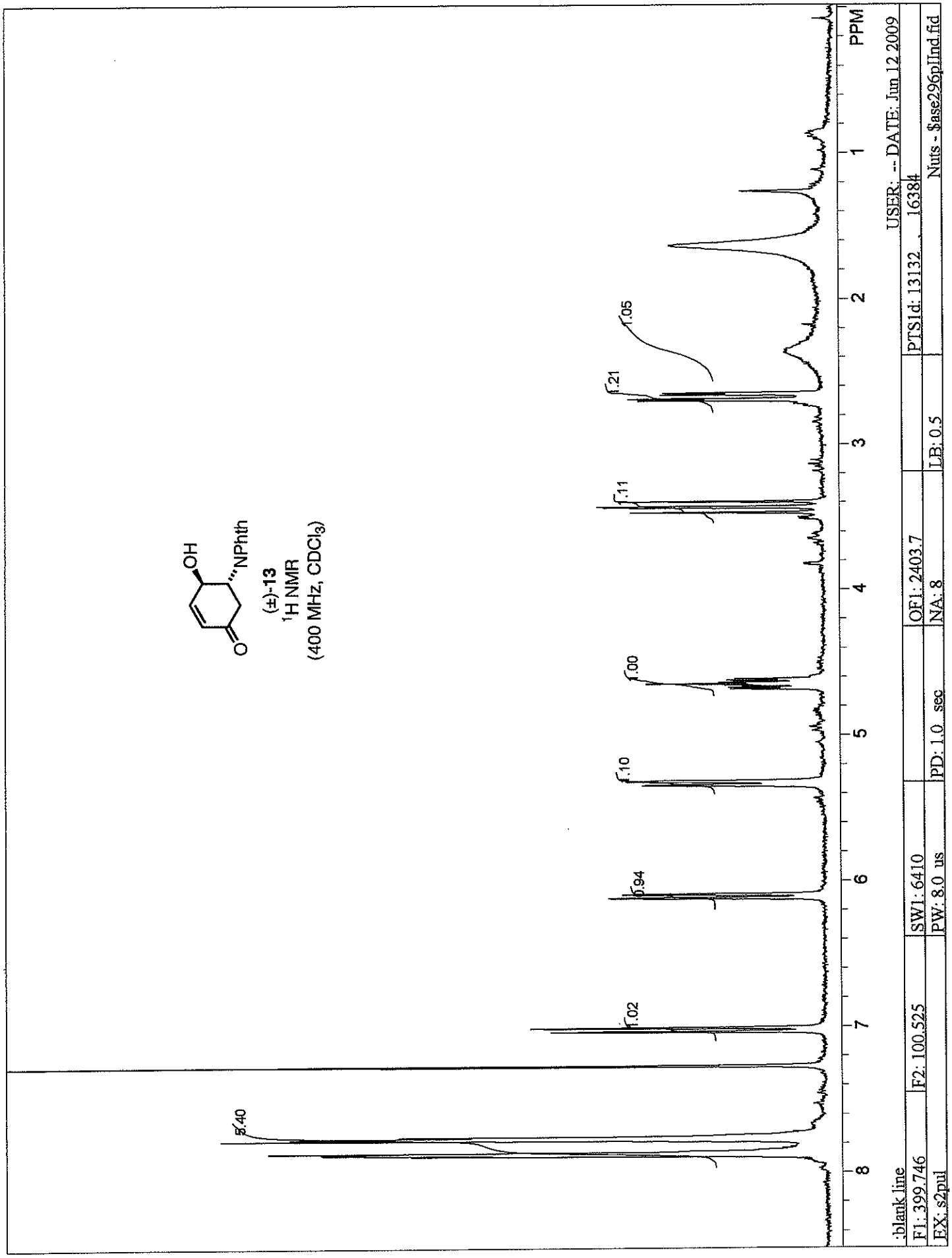

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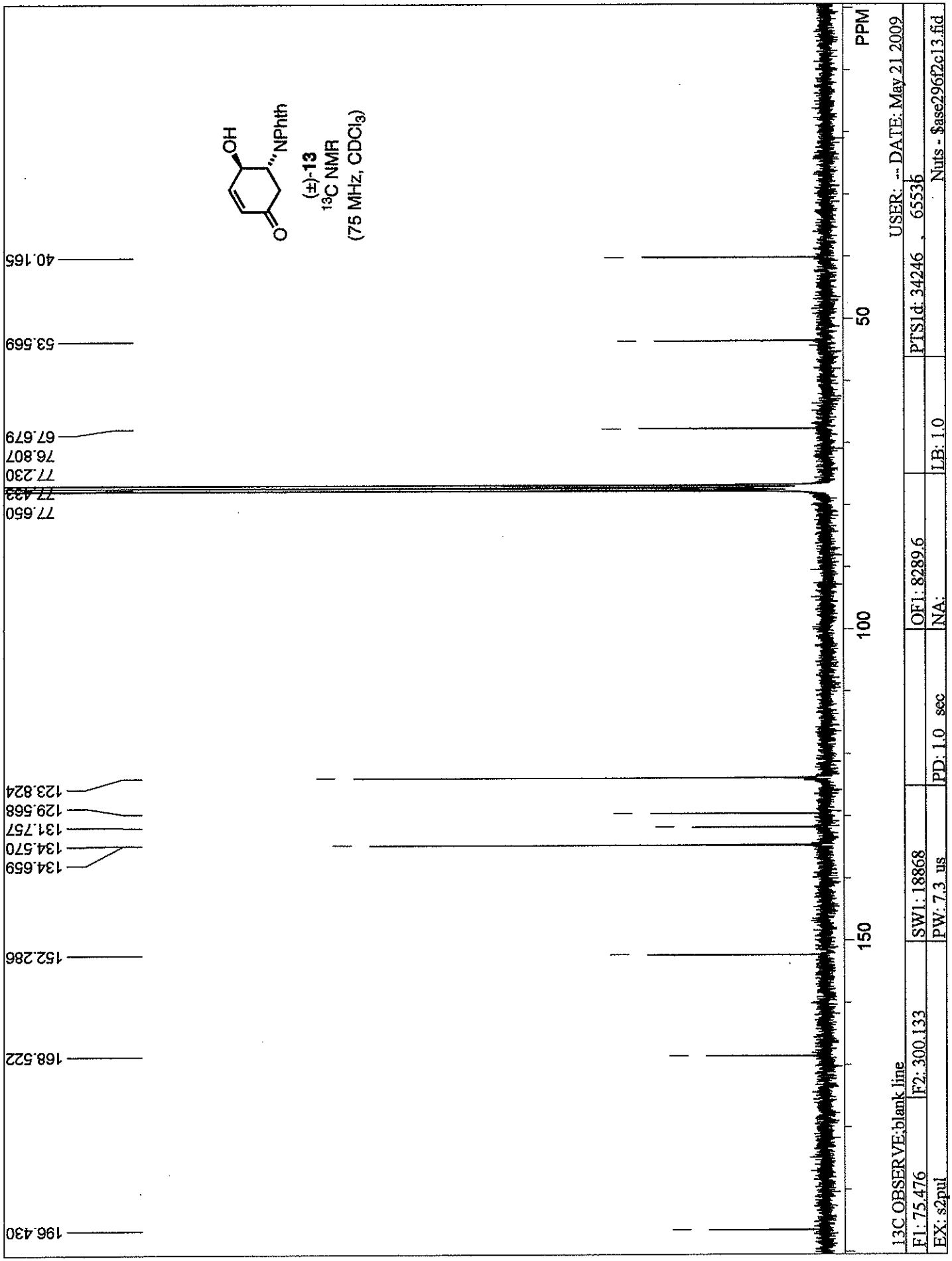

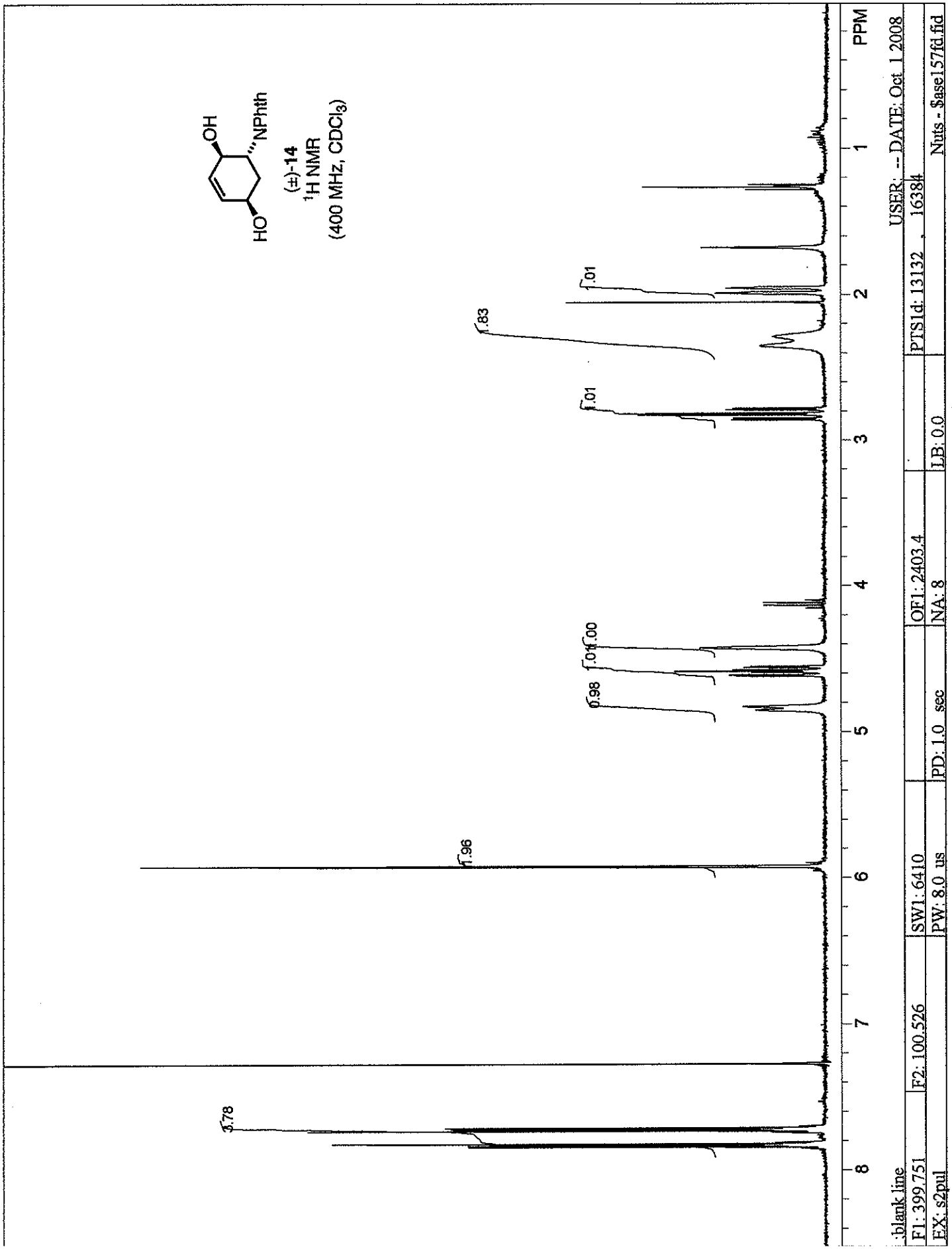
OH
NPhth
HO
(±)-14
^{1}H NMR
(400 MHz, CDCl$_3$)
5.78
1.96
0.98
1.00
1.01
1.83
1.01
PPM
USER: -- DATE: Oct 1 2008
.blank line
F1: 399.751
F2: 100.526
SW1: 6410
OF1: 2403.4
PTS1d: 13132 . 16384
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.0
EX: s2pul
Nuts - $ase157fd.fid

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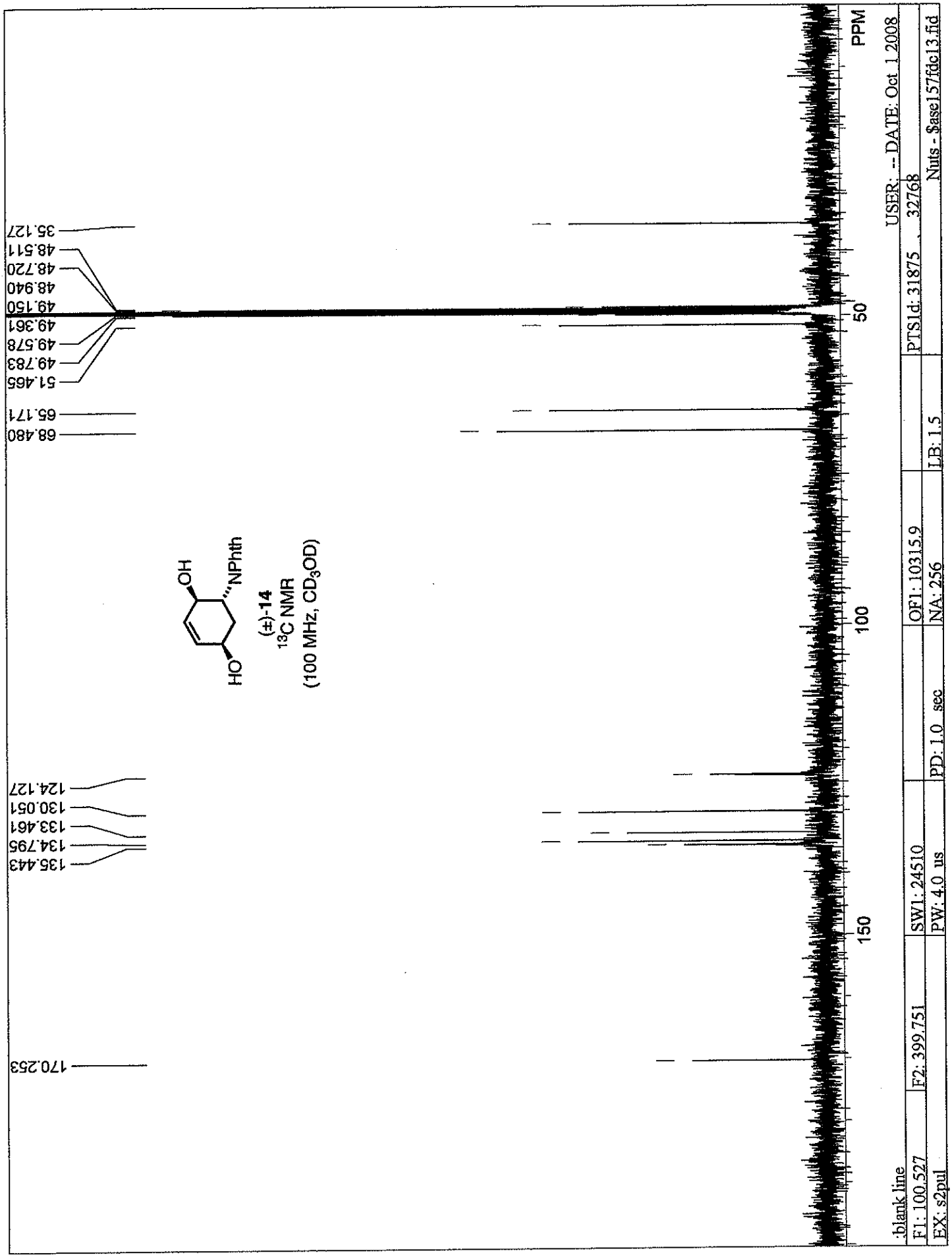

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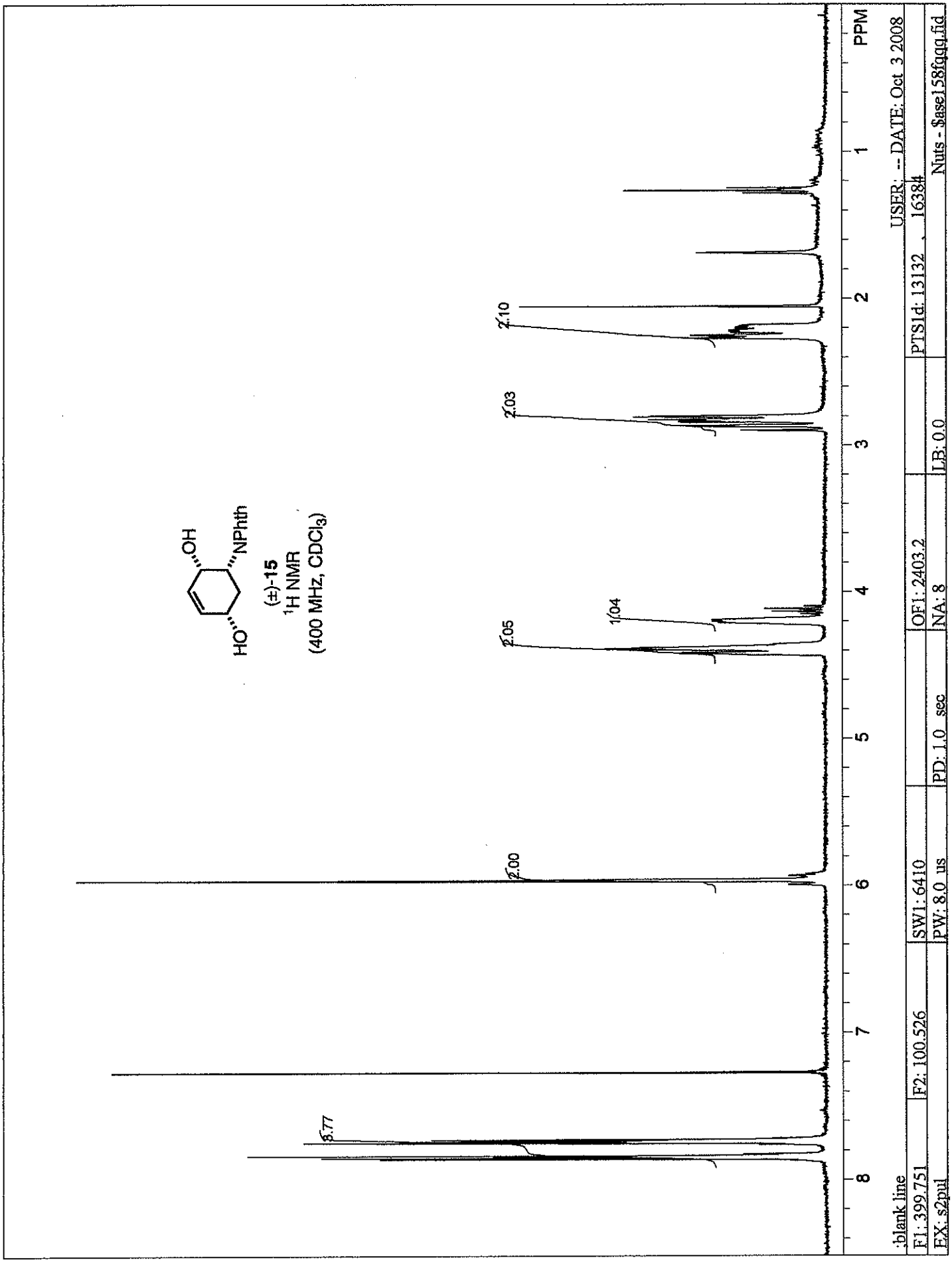

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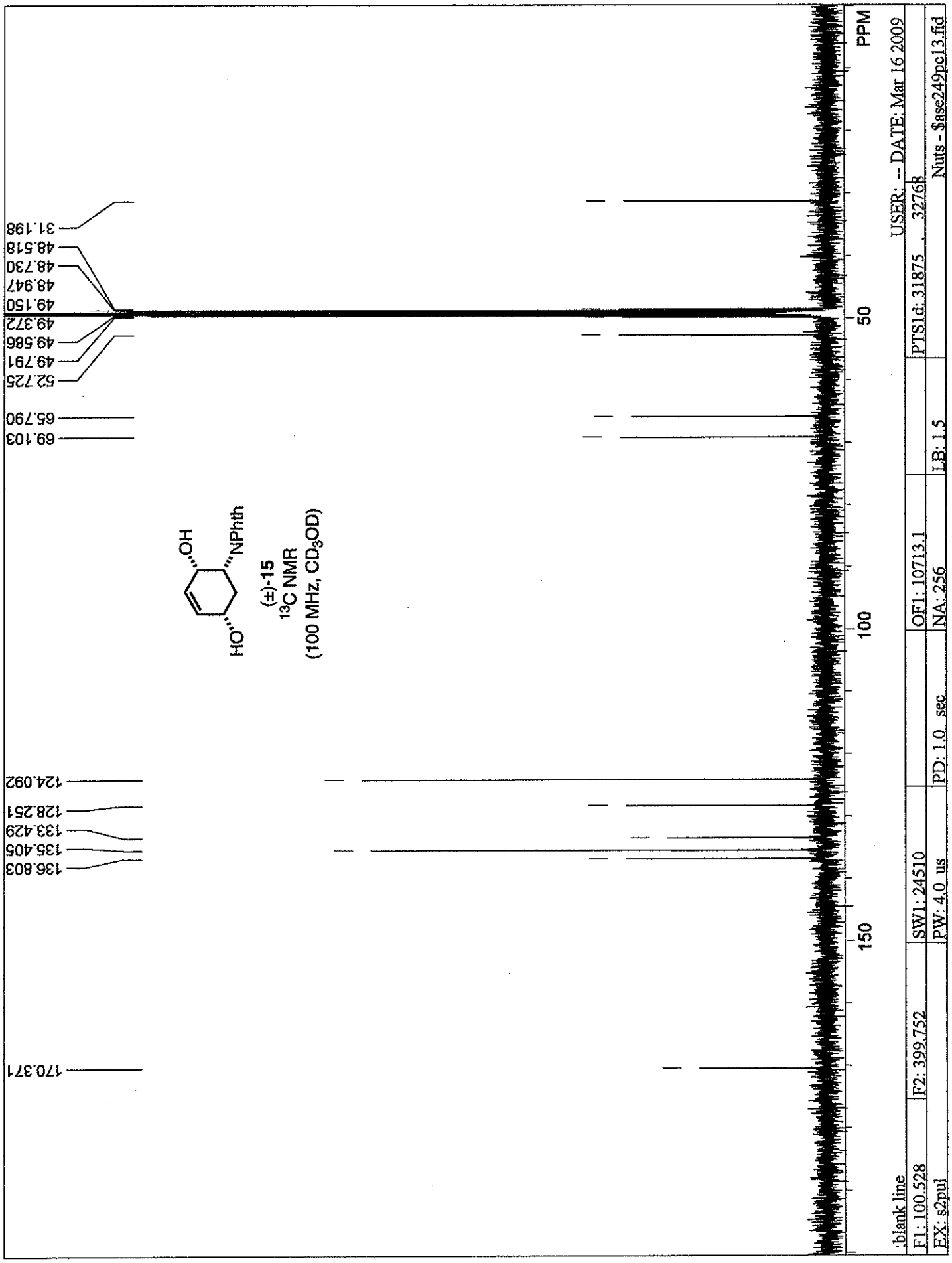

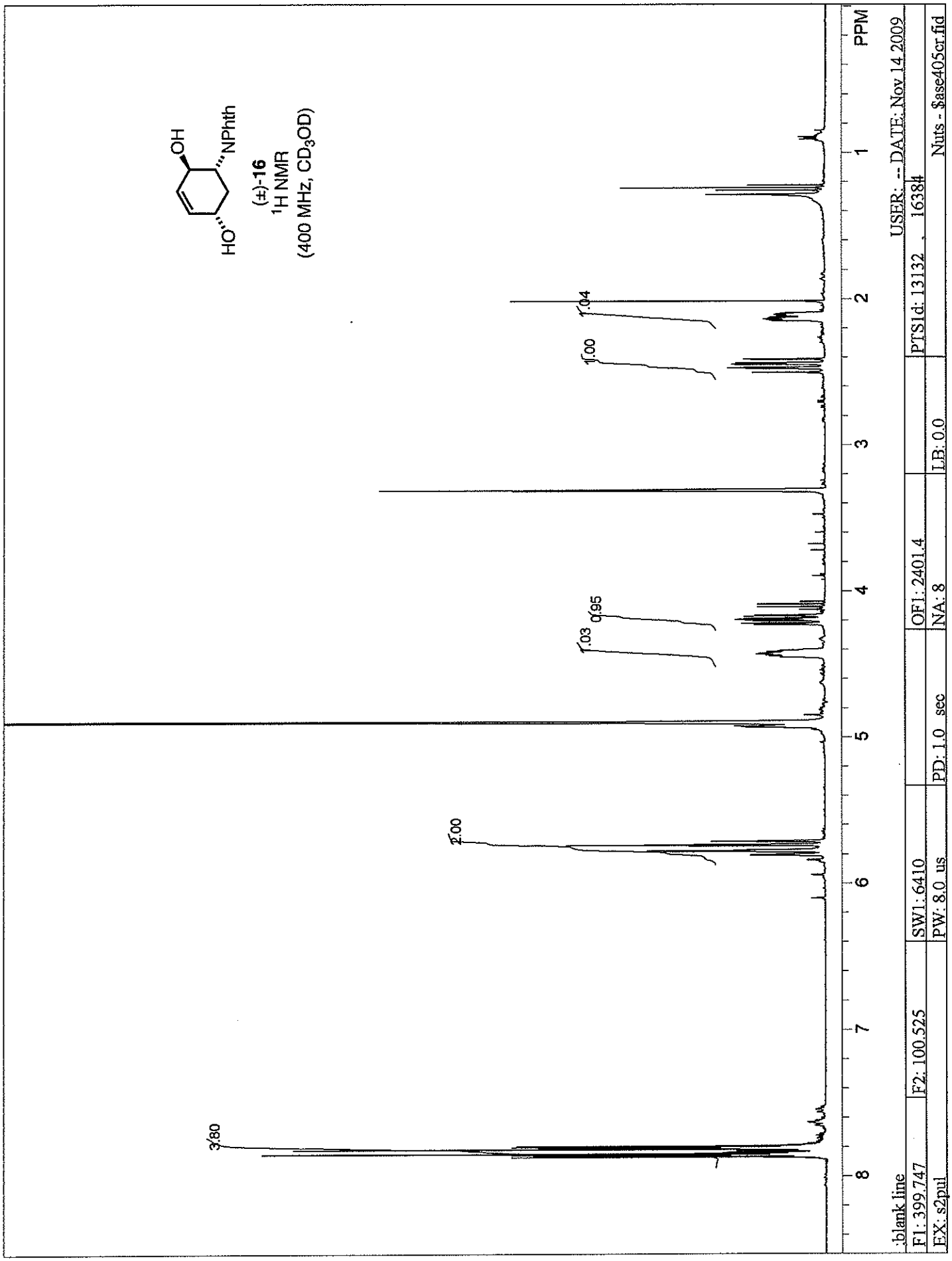
OH
NPhth
HO
(±)-16
^{1}H NMR
(400 MHz, CD_3OD)
3.80
2.00
1.03
0.95
1.00
1.04
PPM
USER: -- DATE: Nov 14 2009
blank line
F1: 399.747
F2: 100.525
SW1: 6410
OF1: 2401.4
PTS1d: 13132 , 16384
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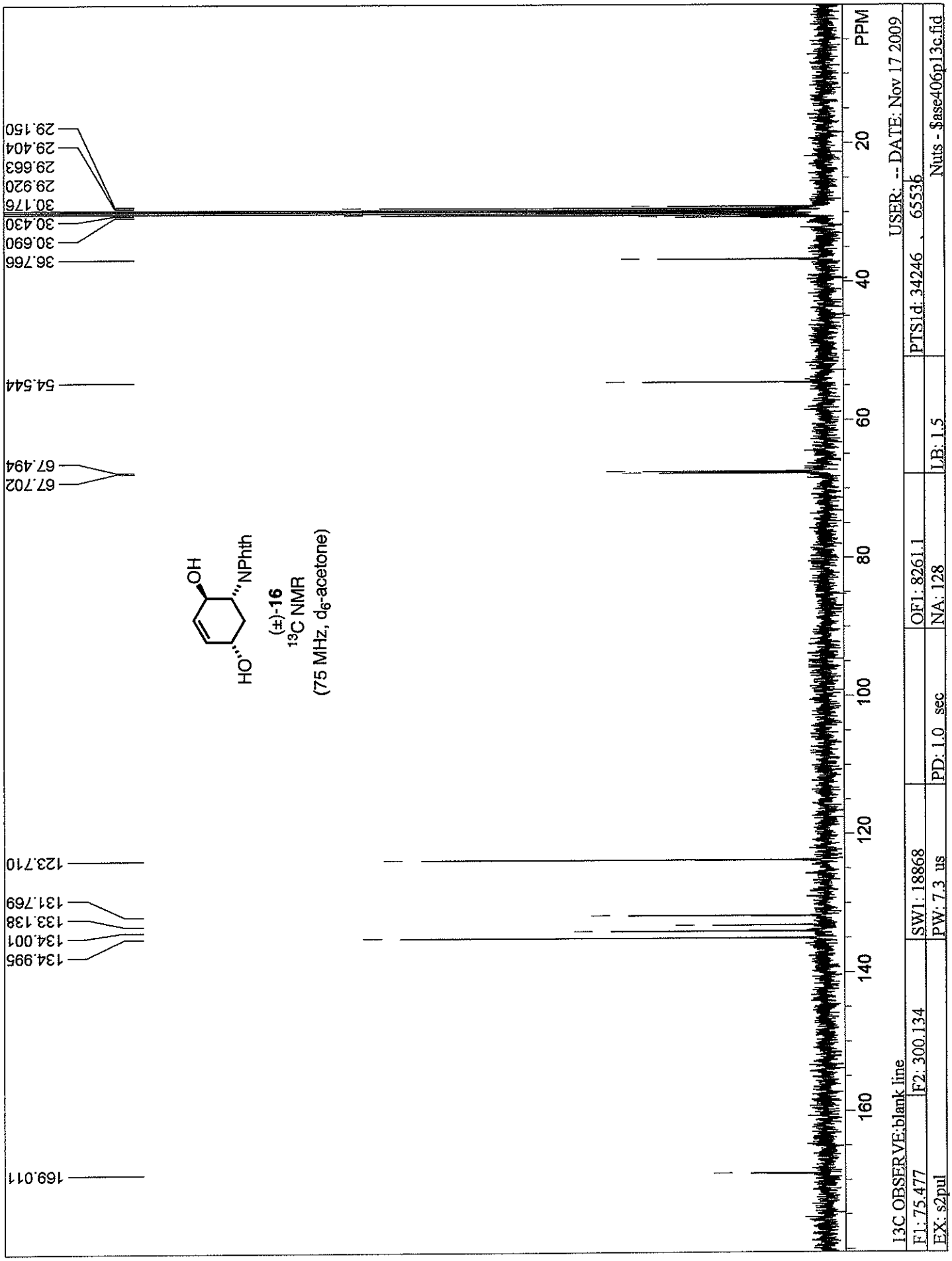
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $ase405cr.fid


OH
NPhth
HO
(±)-16
^{13}C NMR
(75 MHz, d_6-acetone)
29.150
29.404
29.663
29.920
30.176
30.430
30.690
36.766
54.544
67.494
67.702
123.710
131.769
133.138
134.001
134.995
169.011
PPM
13C OBSERVE:blank line
F1: 75.477
F2: 300.134
EX: s2pul
SW1: 18868
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PD: 1.0 sec
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PTS1d: 34246 , 65536
USER: -- DATE: Nov 17 2009
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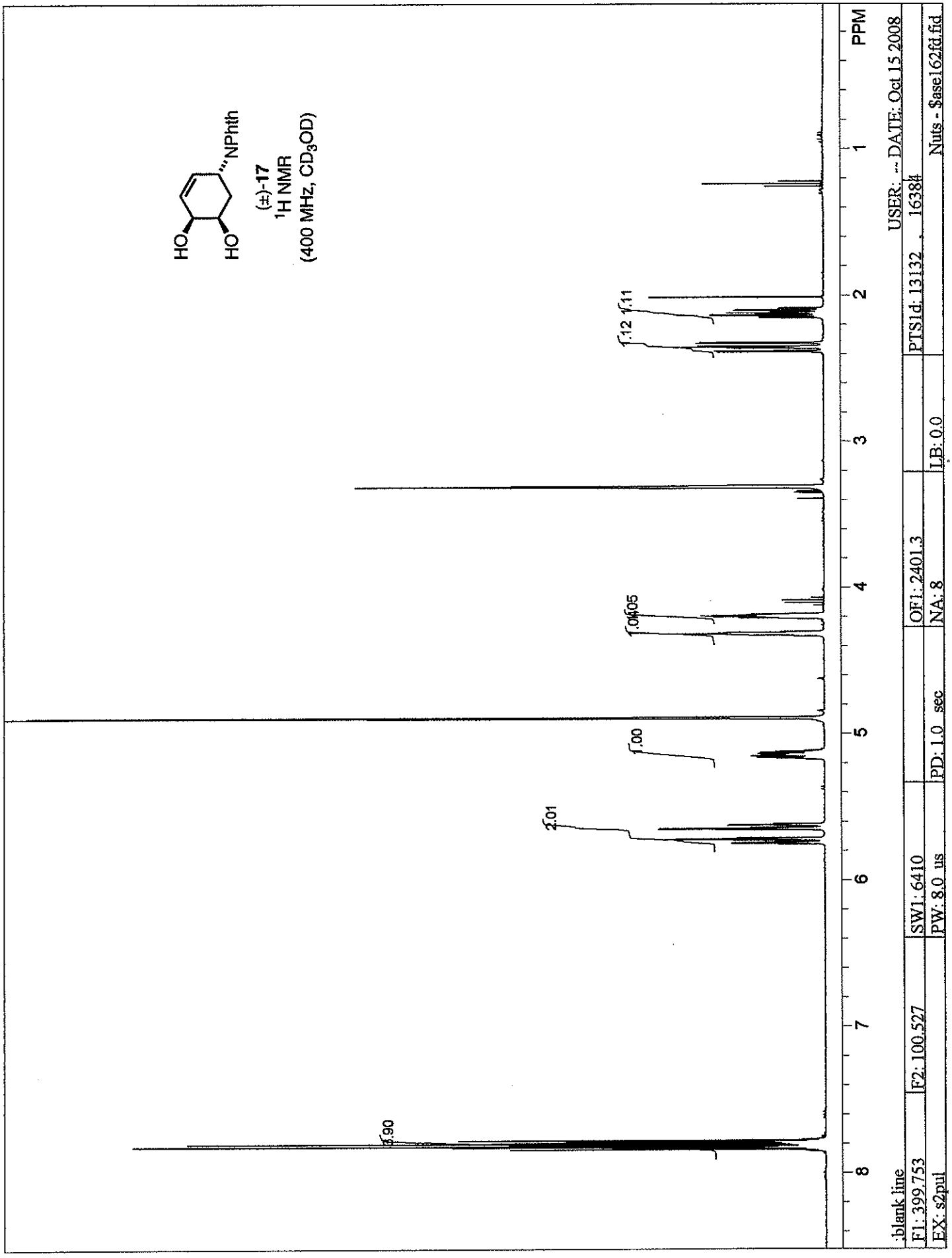

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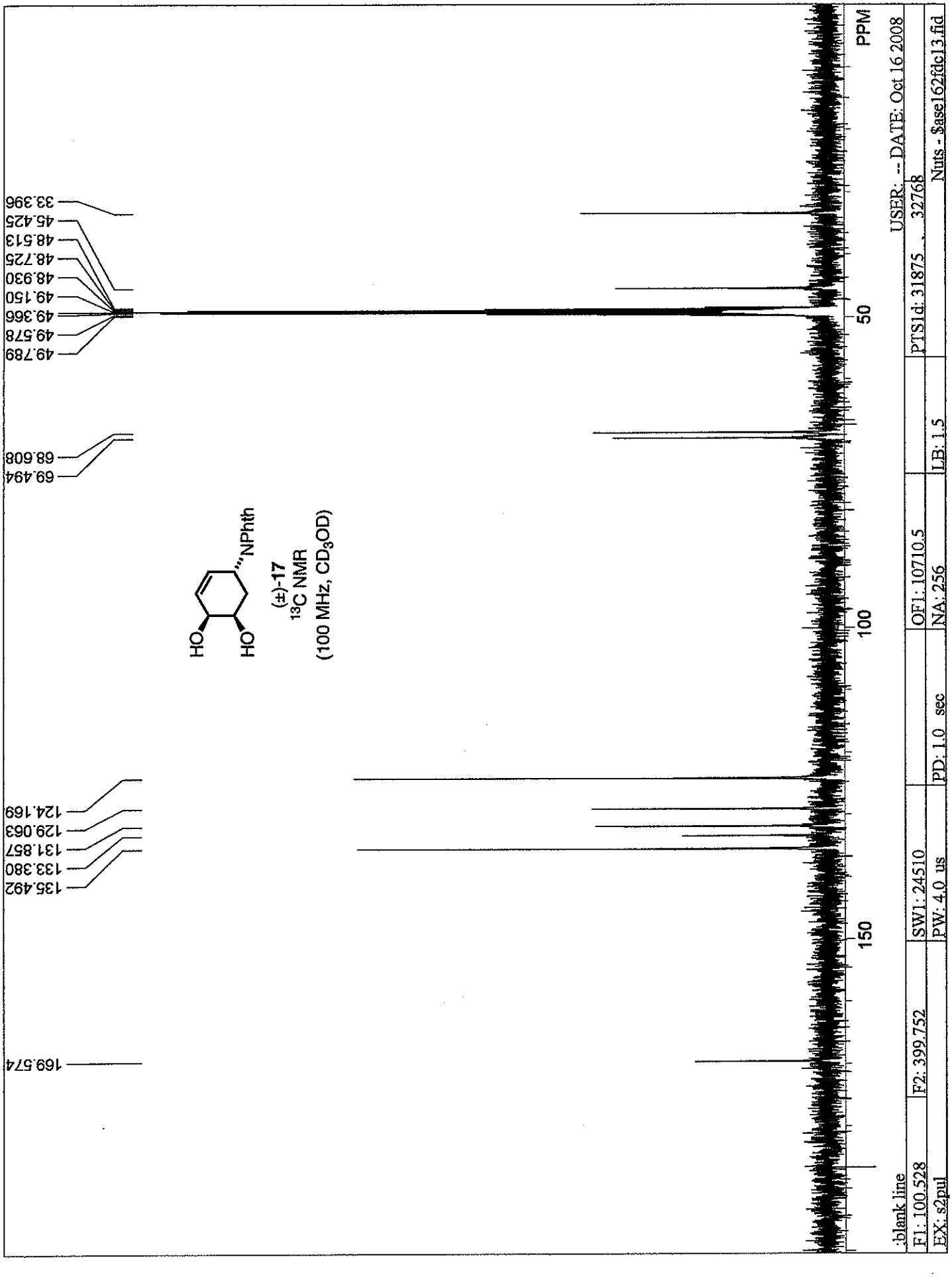

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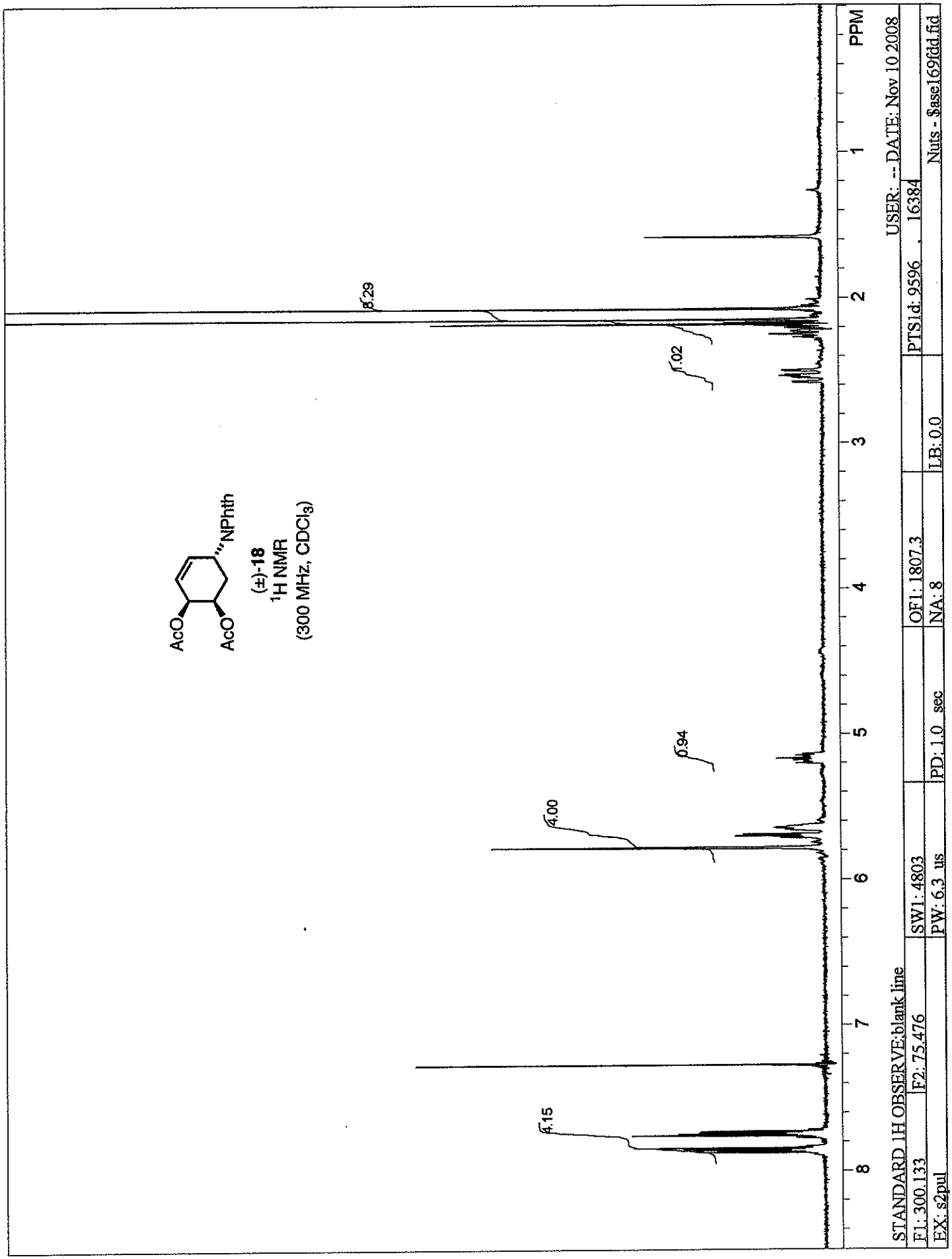

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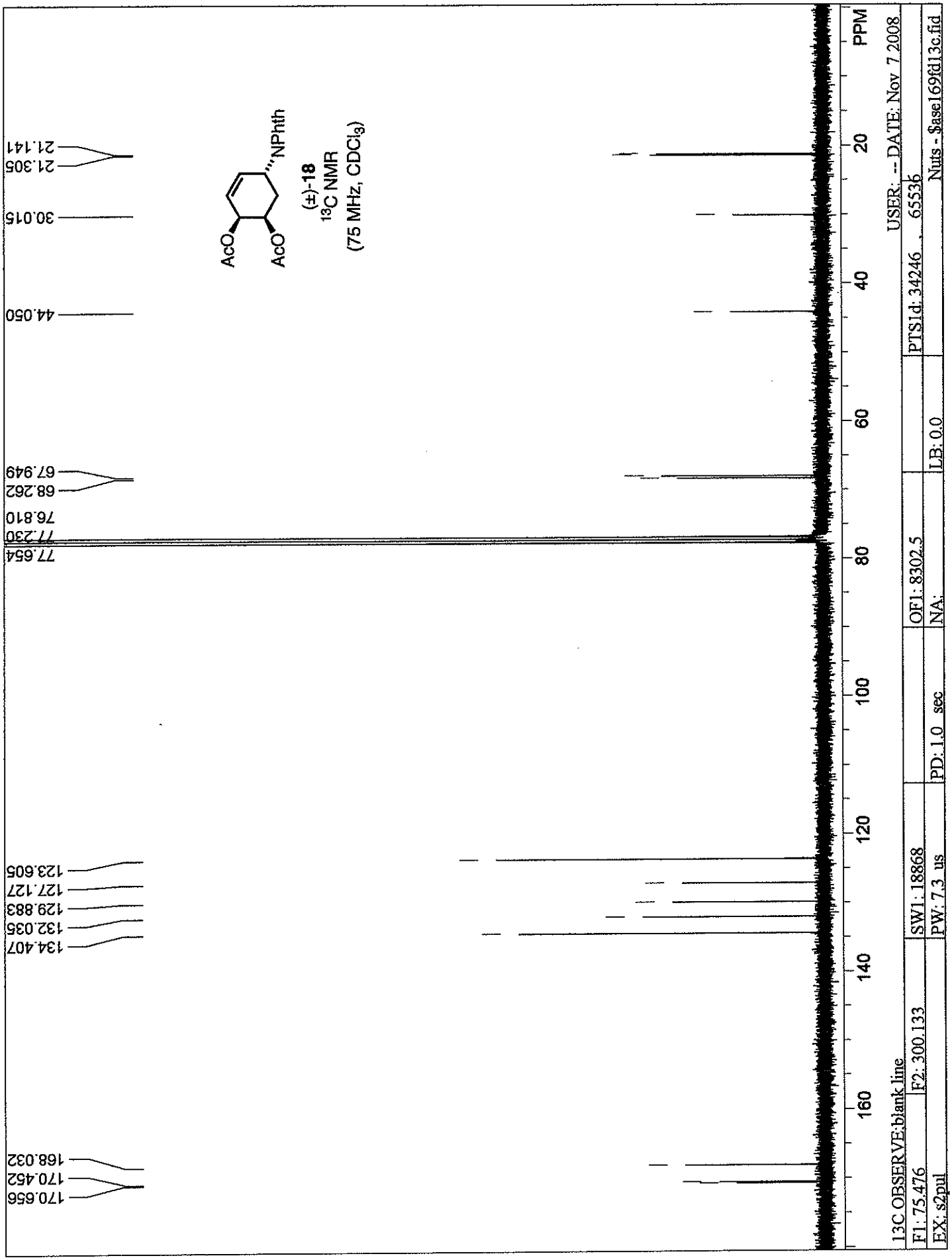

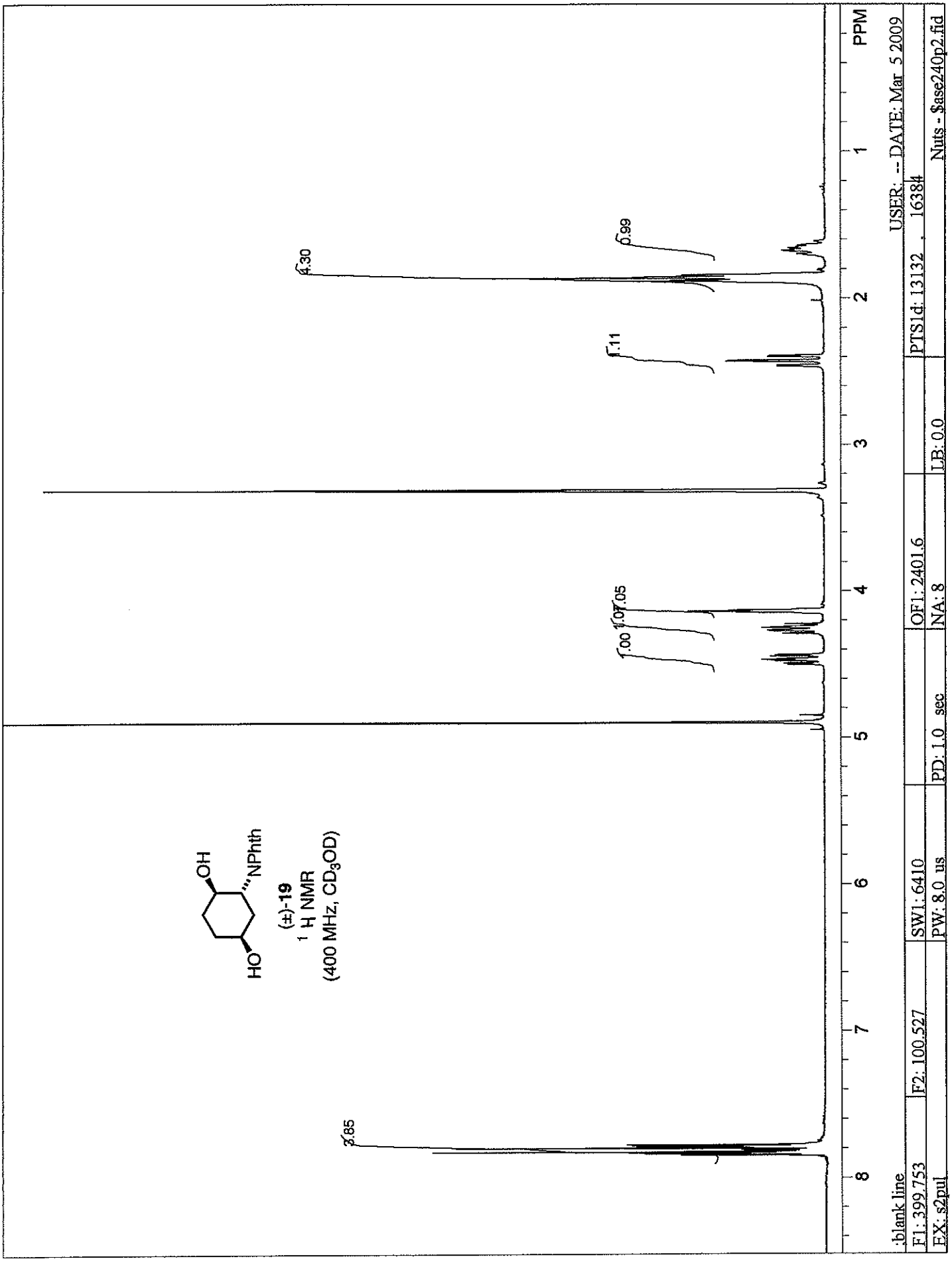
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NPhth
HO
(±)-19
^{1}H NMR
(400 MHz, CD_3OD)
3.85
4.30
1.00
1.07
1.05
1.11
0.99
PPM
USER: -- DATE: Mar 5 2009
blank line
F1: 399.753
F2: 100.527
SW1: 6410
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PTS1d: 13132 , 16384
EX: s2pul
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.0
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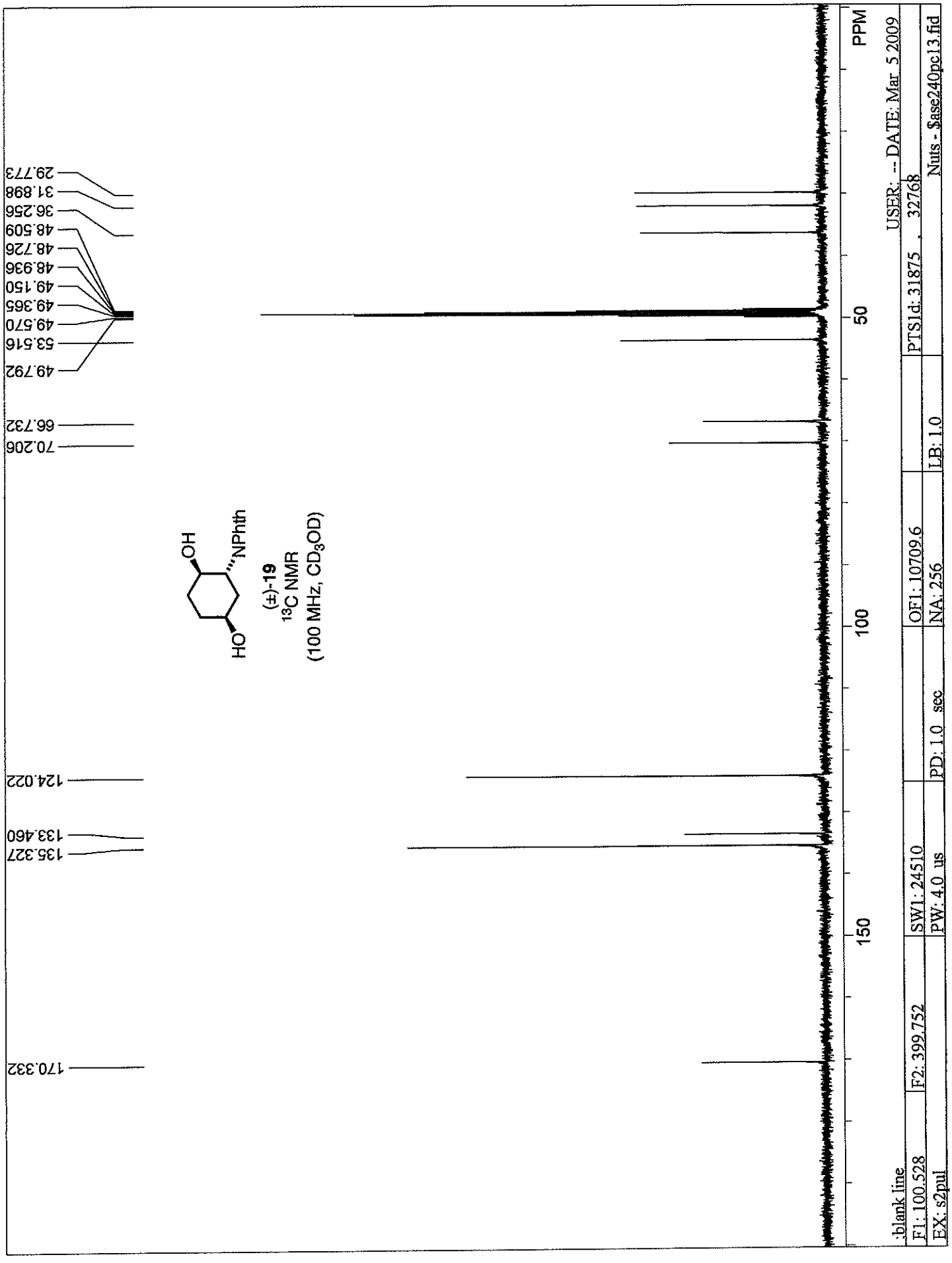

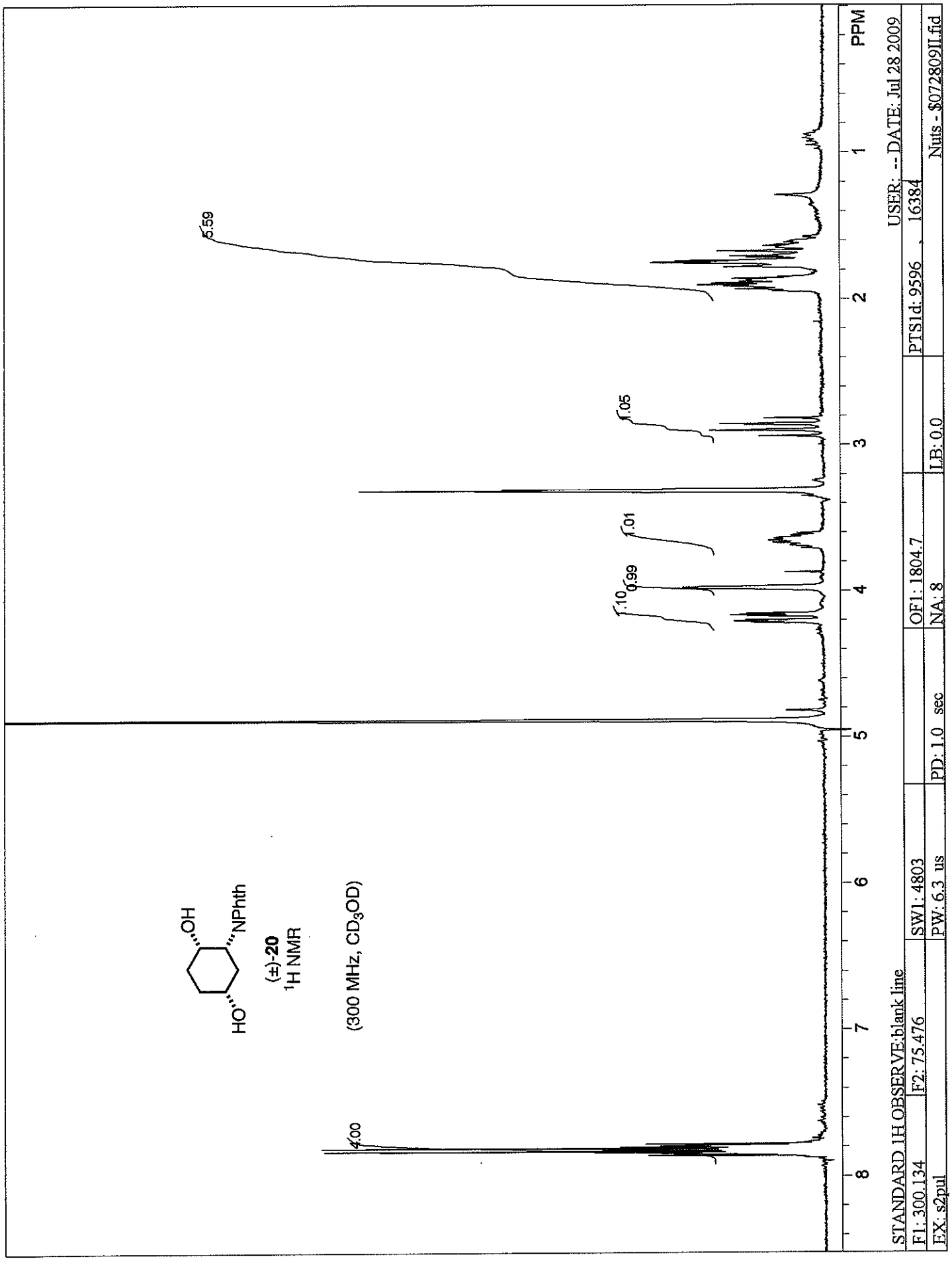
OH
NPhth
HO
(±)-20
^{1}H NMR
(300 MHz, CD_3OD)
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5.59
1.05
1.01
0.99
1.10
PPM
STANDARD 1H OBSERVE:blank line
F1: 300.134
F2: 75.476
SW1: 4803
OF1: 1804.7
PTS1d: 9596
16384
EX: s2pul
PW: 6.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $072809IL.fid
USER: -- DATE: Jul 28 2009

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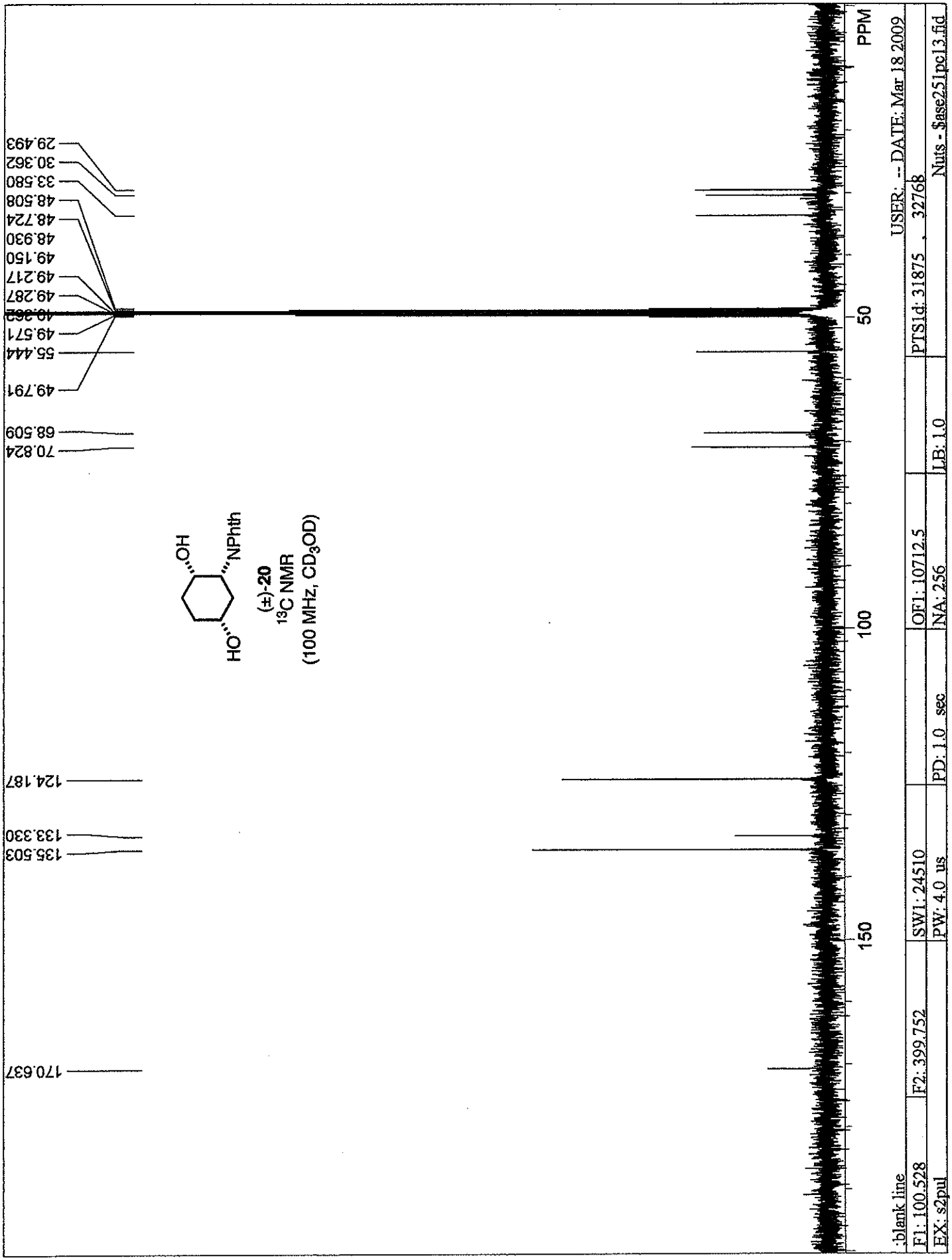

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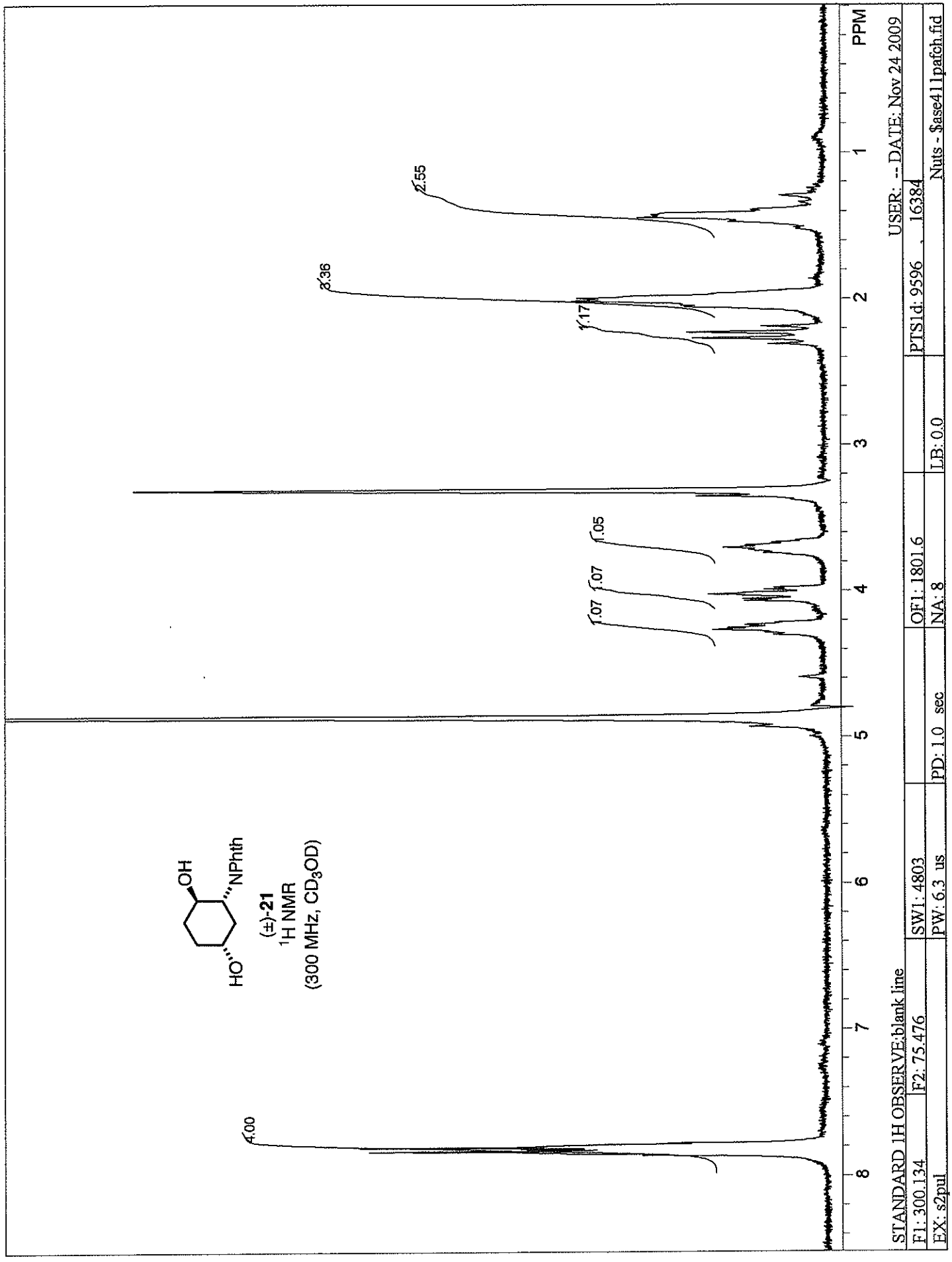

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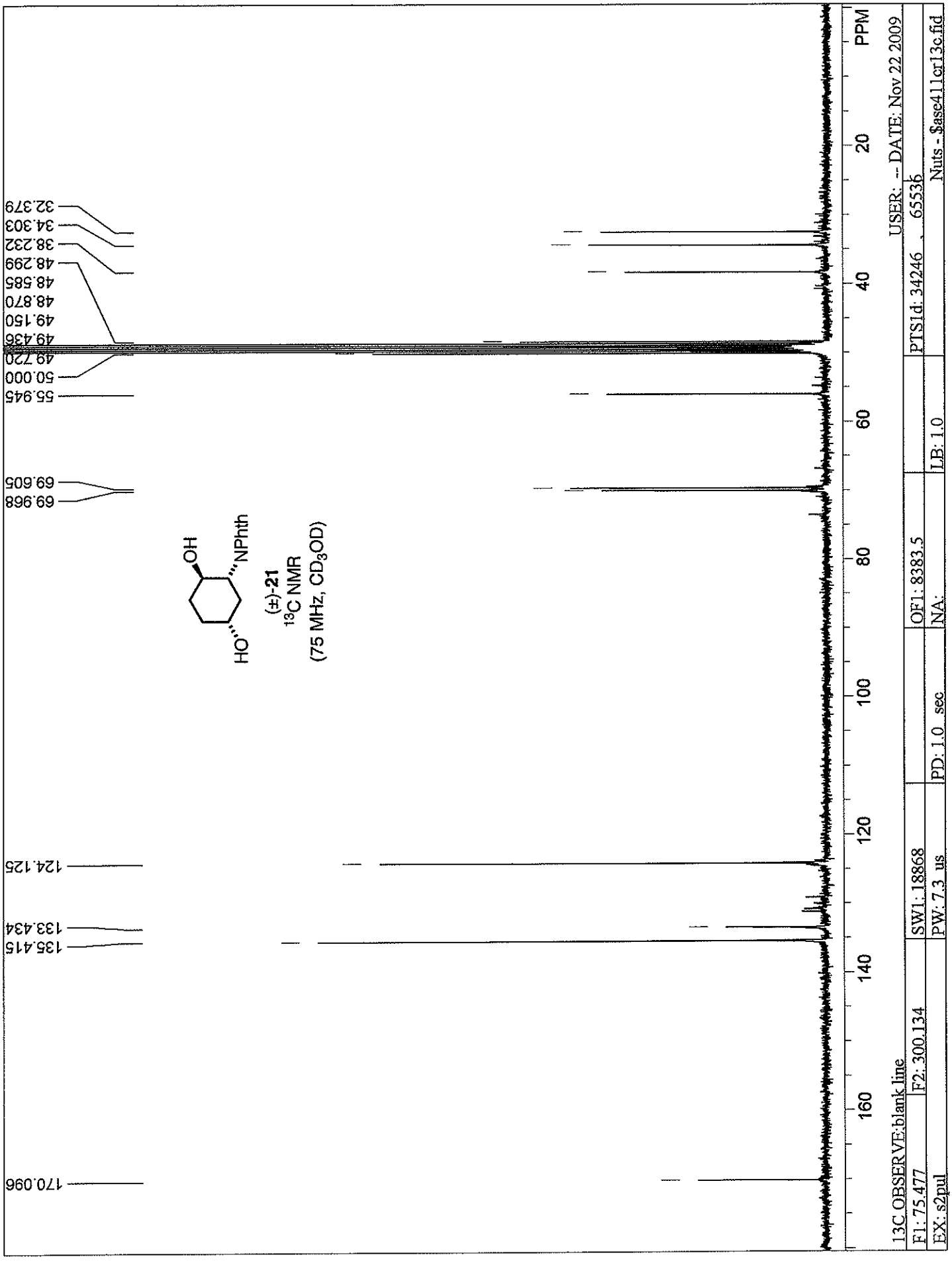

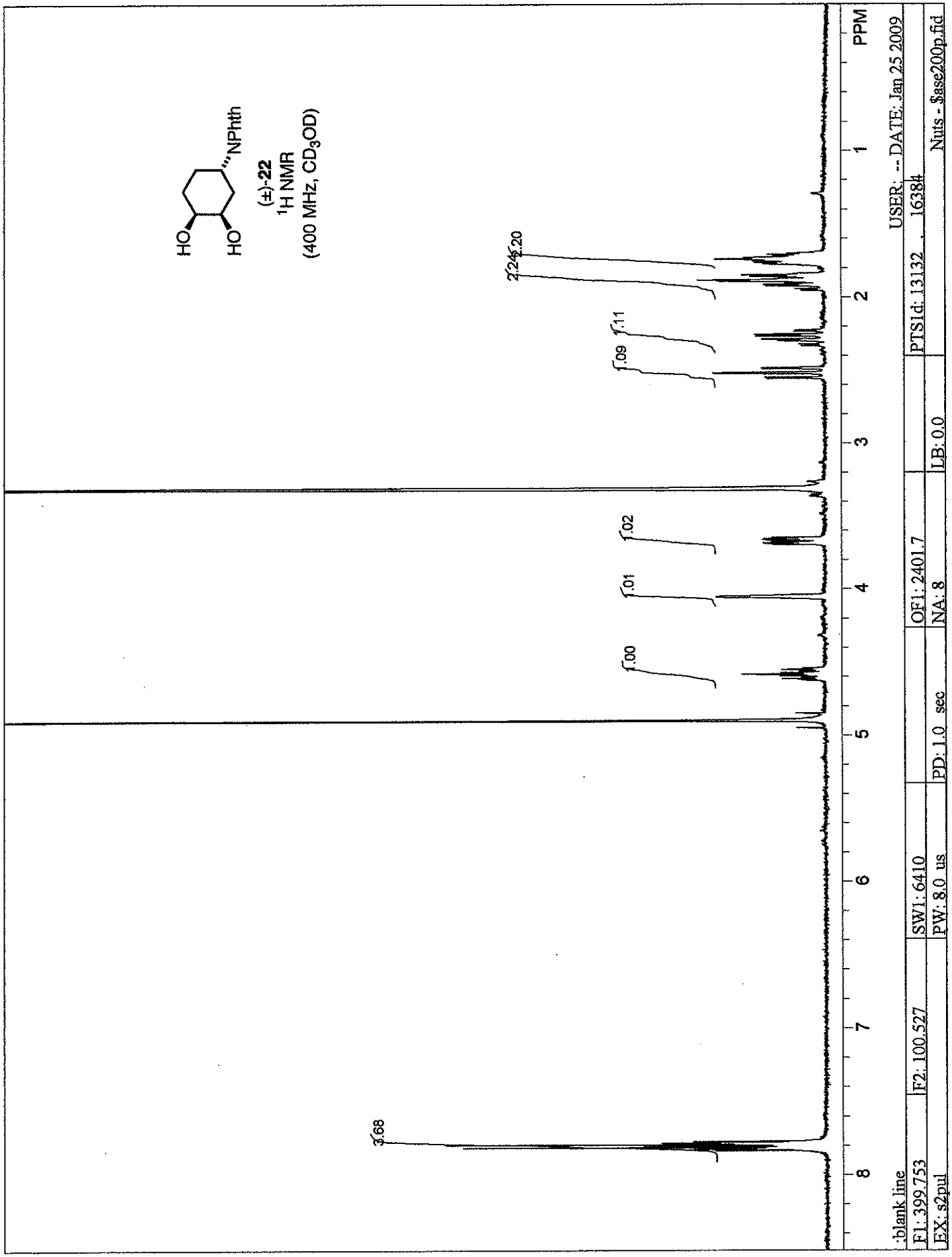
NPhth
HO
HO
(±)-22
^{1}H NMR
(400 MHz, CD_3OD)
3.68
1.00
1.01
1.02
1.09
1.11
2.24
2.20
PPM
USER: -- DATE: Jan 25 2009
blank line
F1: 399.753
F2: 100.527
SW1: 6410
PW: 8.0 us
PD: 1.0 sec
OF1: 2401.7
NA: 8
LB: 0.0
PTS1d: 13132
16384
EX: s2pul
Nuts - $ase200p.fid

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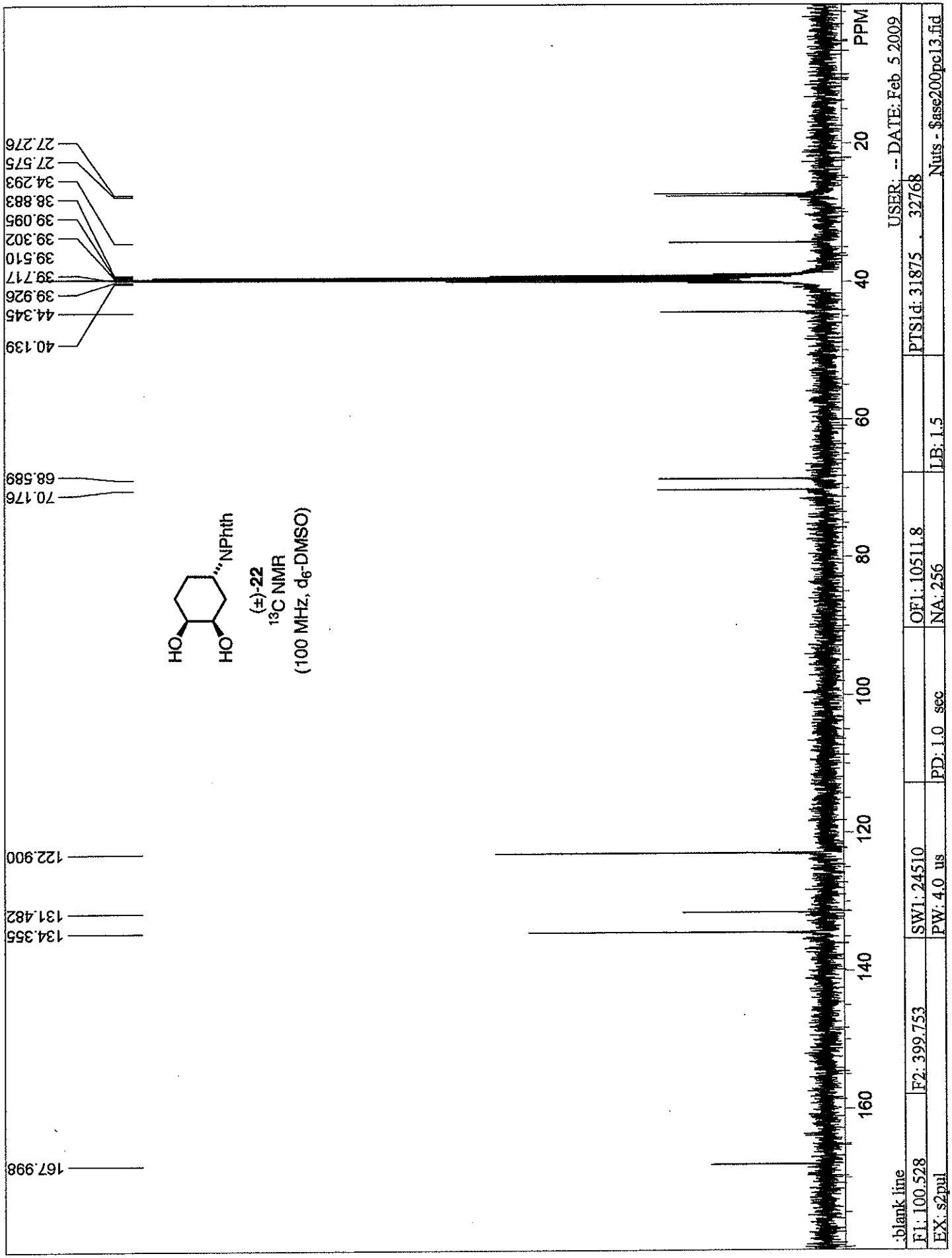

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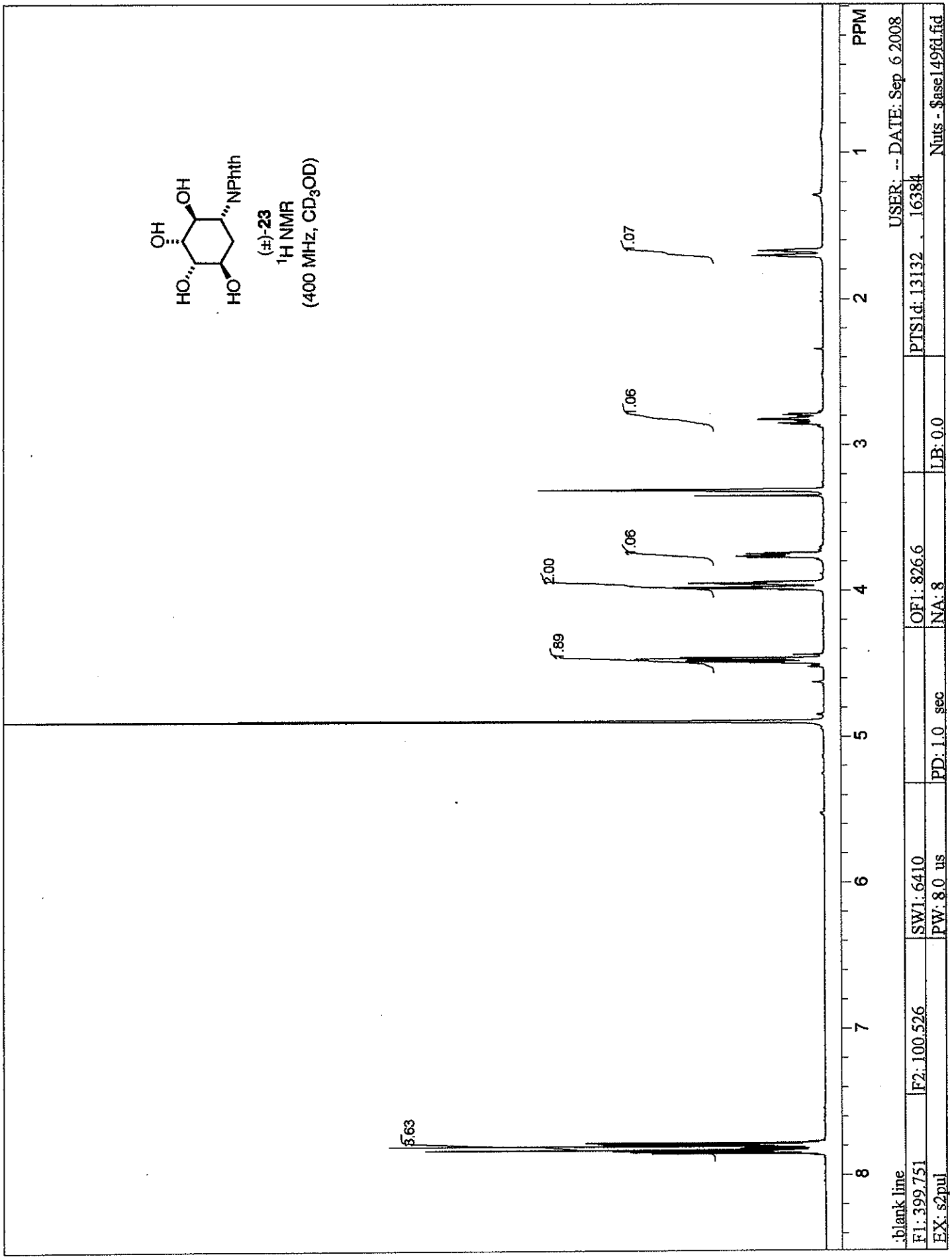

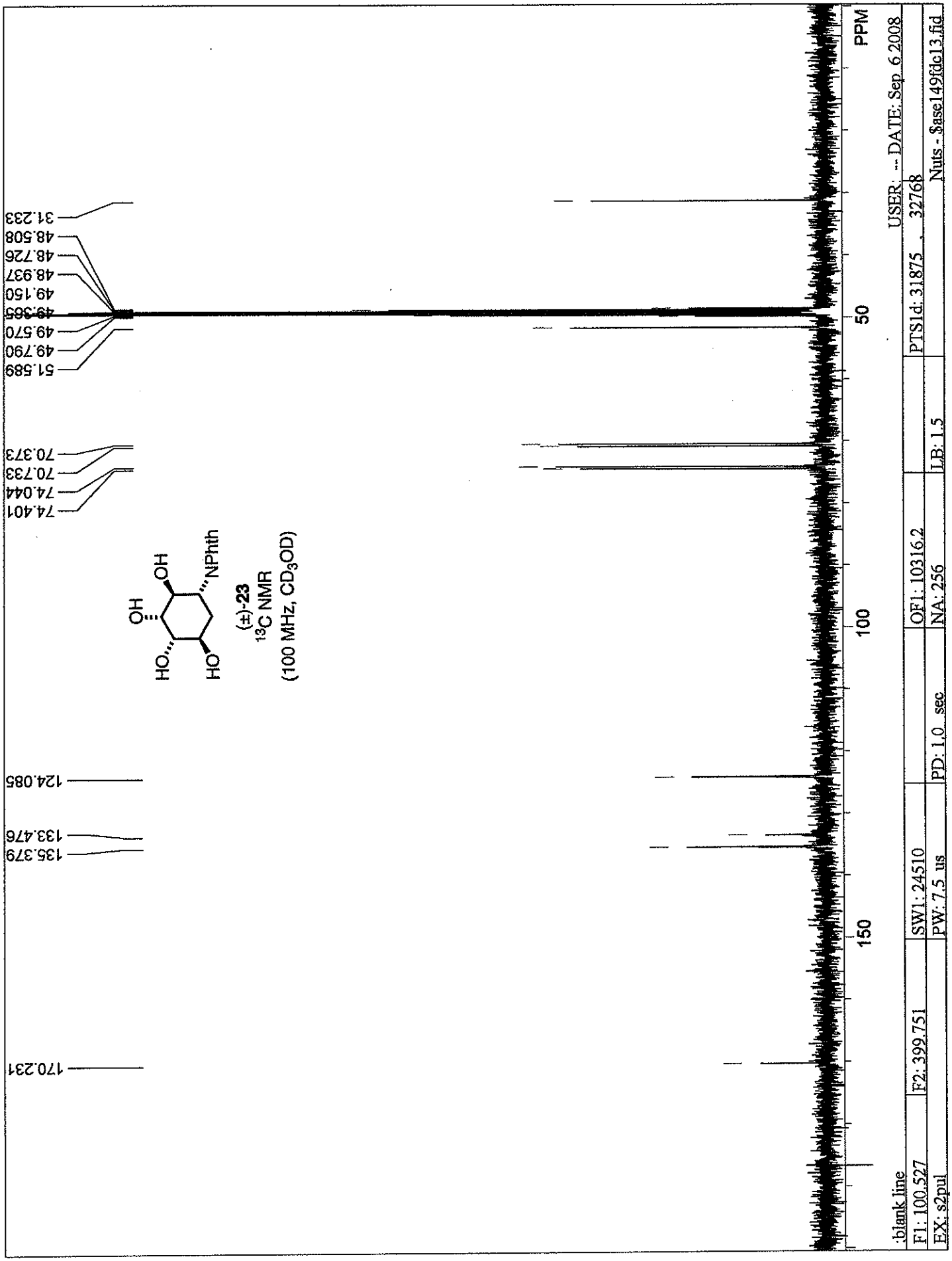
(±)-23
^{13}C NMR
(100 MHz, CD_3OD)
NPhth
OH
OH
HO
HO
31.233
48.508
48.726
48.937
49.150
49.365
49.570
49.790
51.589
70.373
70.733
74.044
74.401
124.085
133.476
135.379
170.231
PPM
50
100
150
USER: -- DATE: Sep 6 2008
PTS1d: 31875 32768
LB: 1.5
Nuts - $ase149fdc13.fid
OF1: 10316.2
NA: 256
SW1: 24510
PW: 7.5 us
PD: 1.0 sec
F2: 399.751
F1: 100.527
EX: s2pul
:blank line

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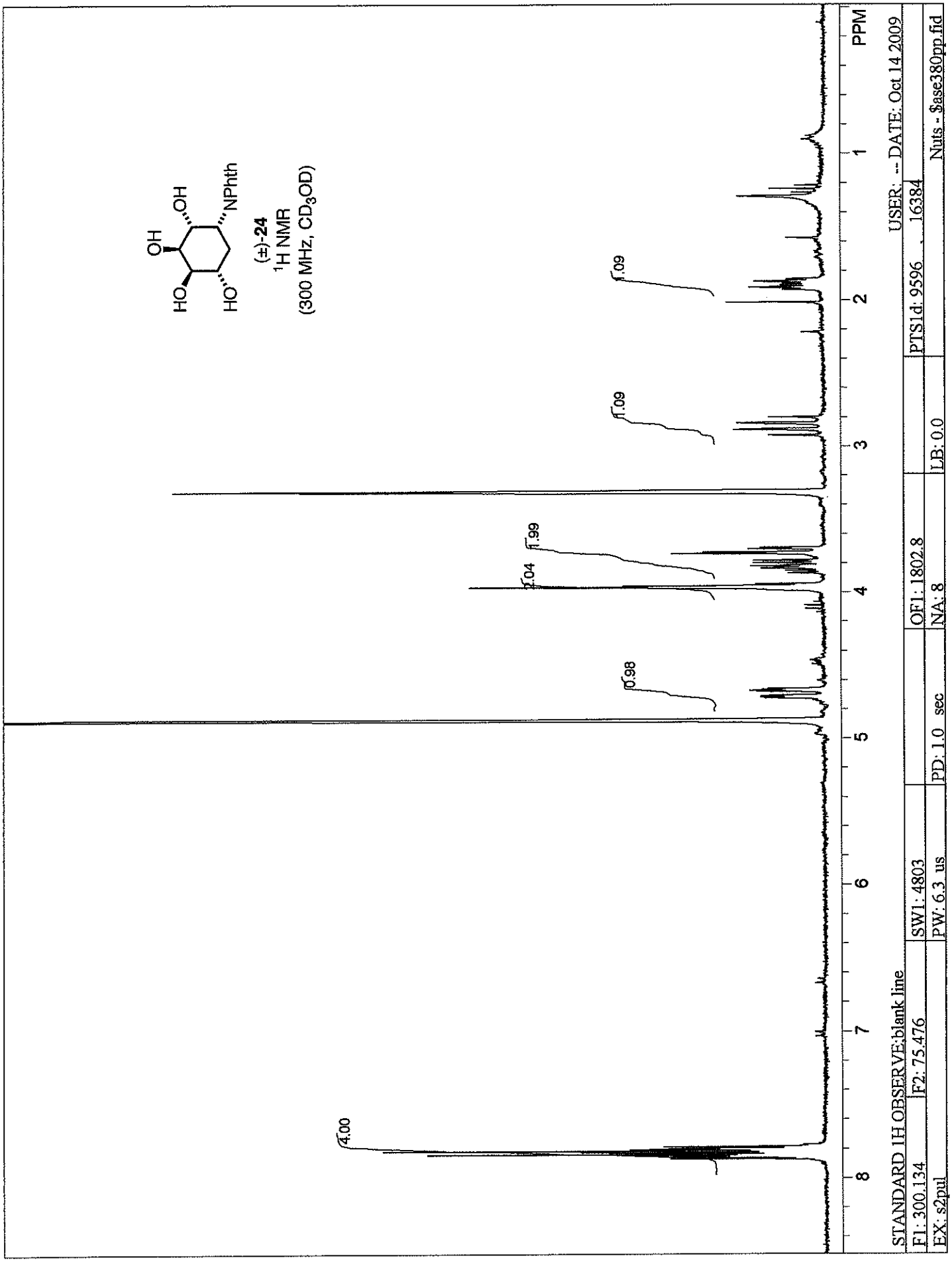
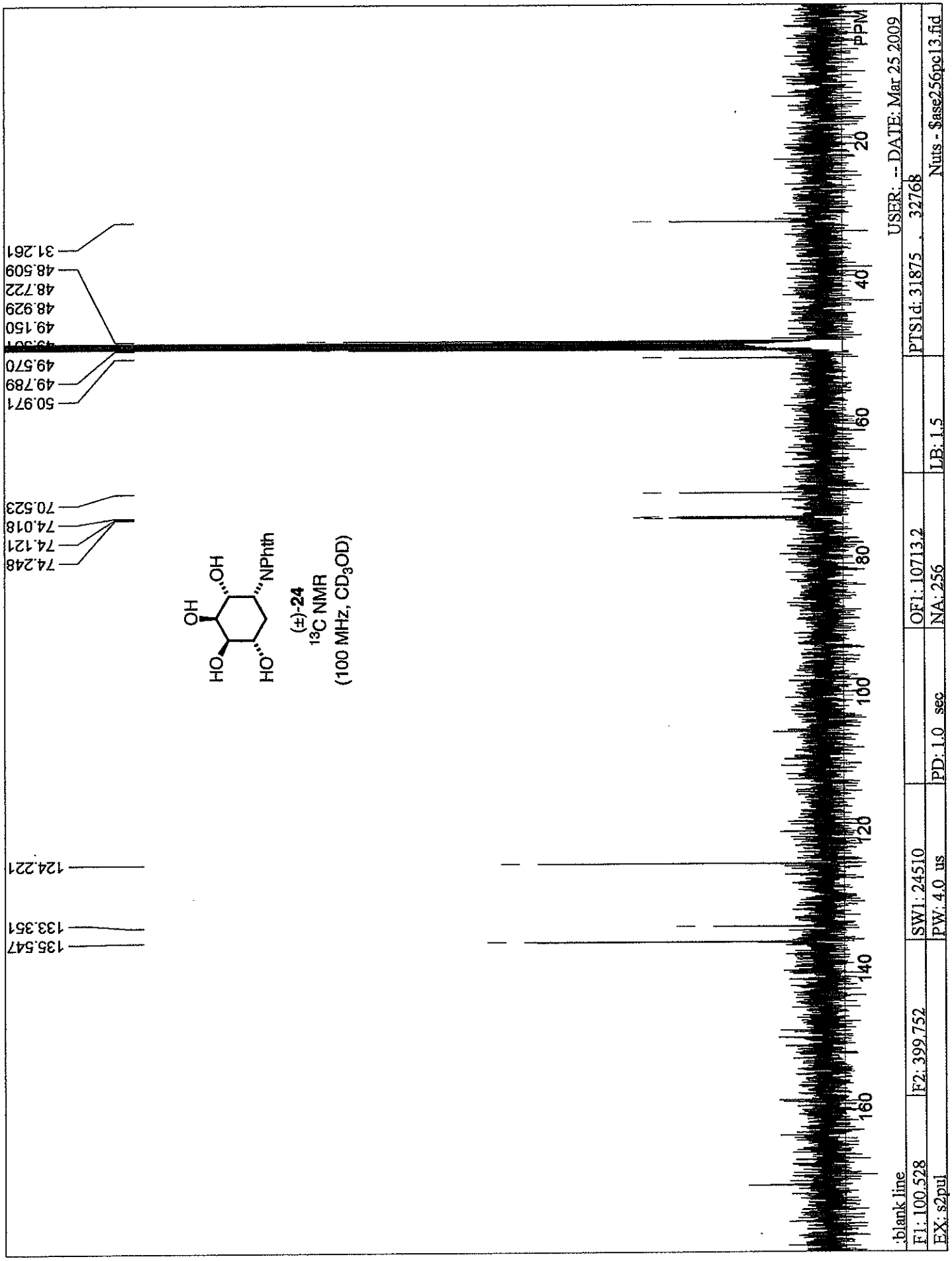
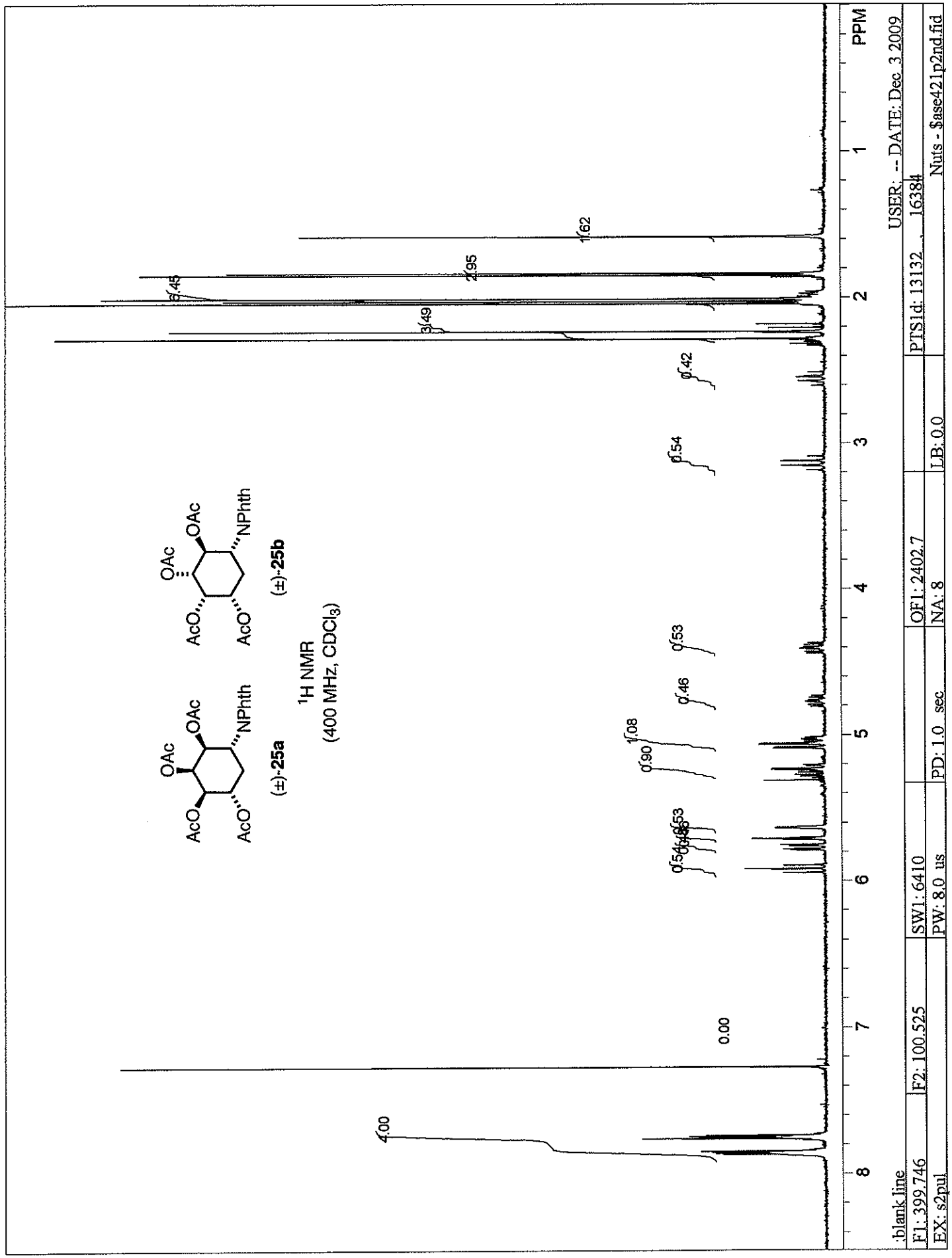
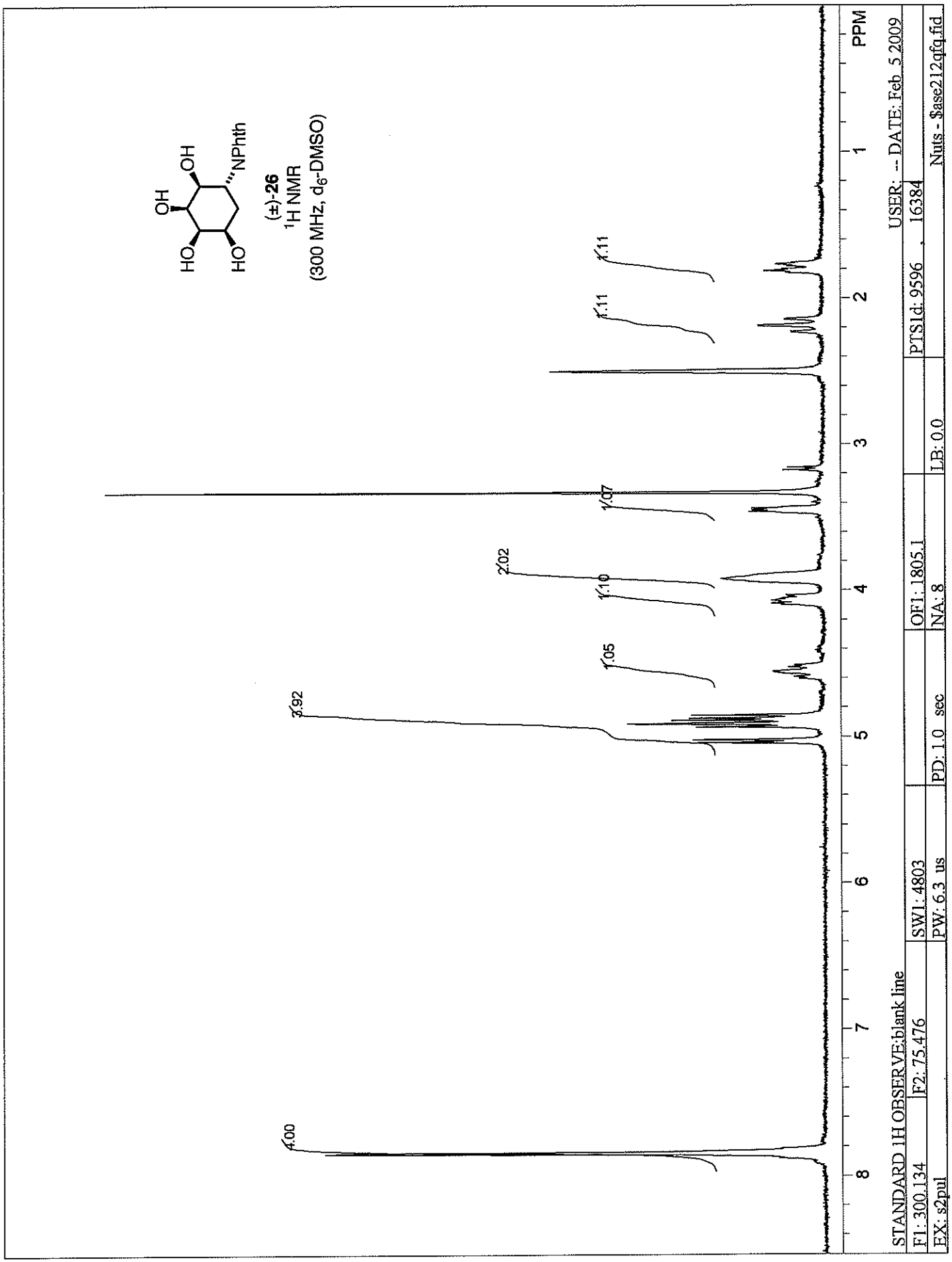
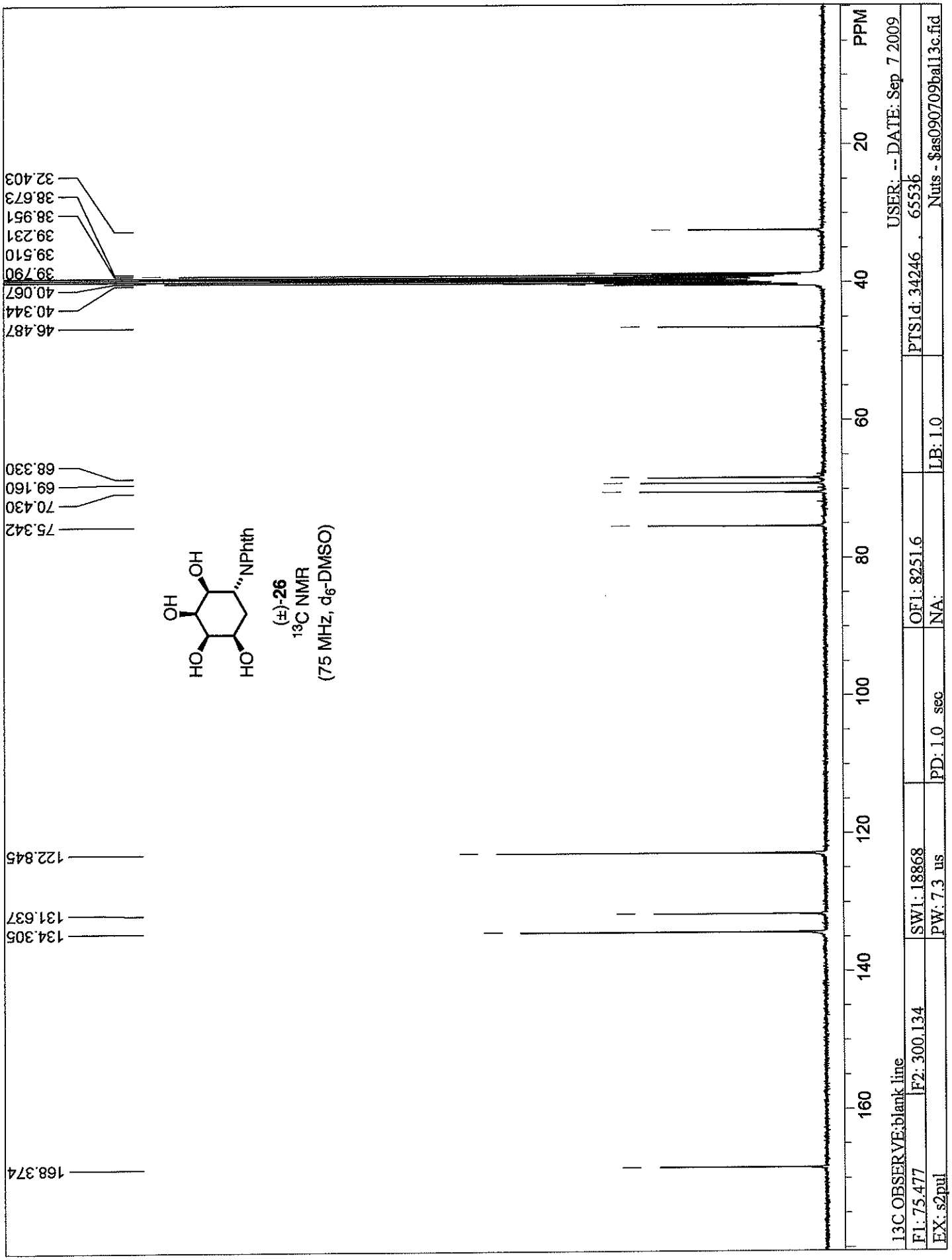
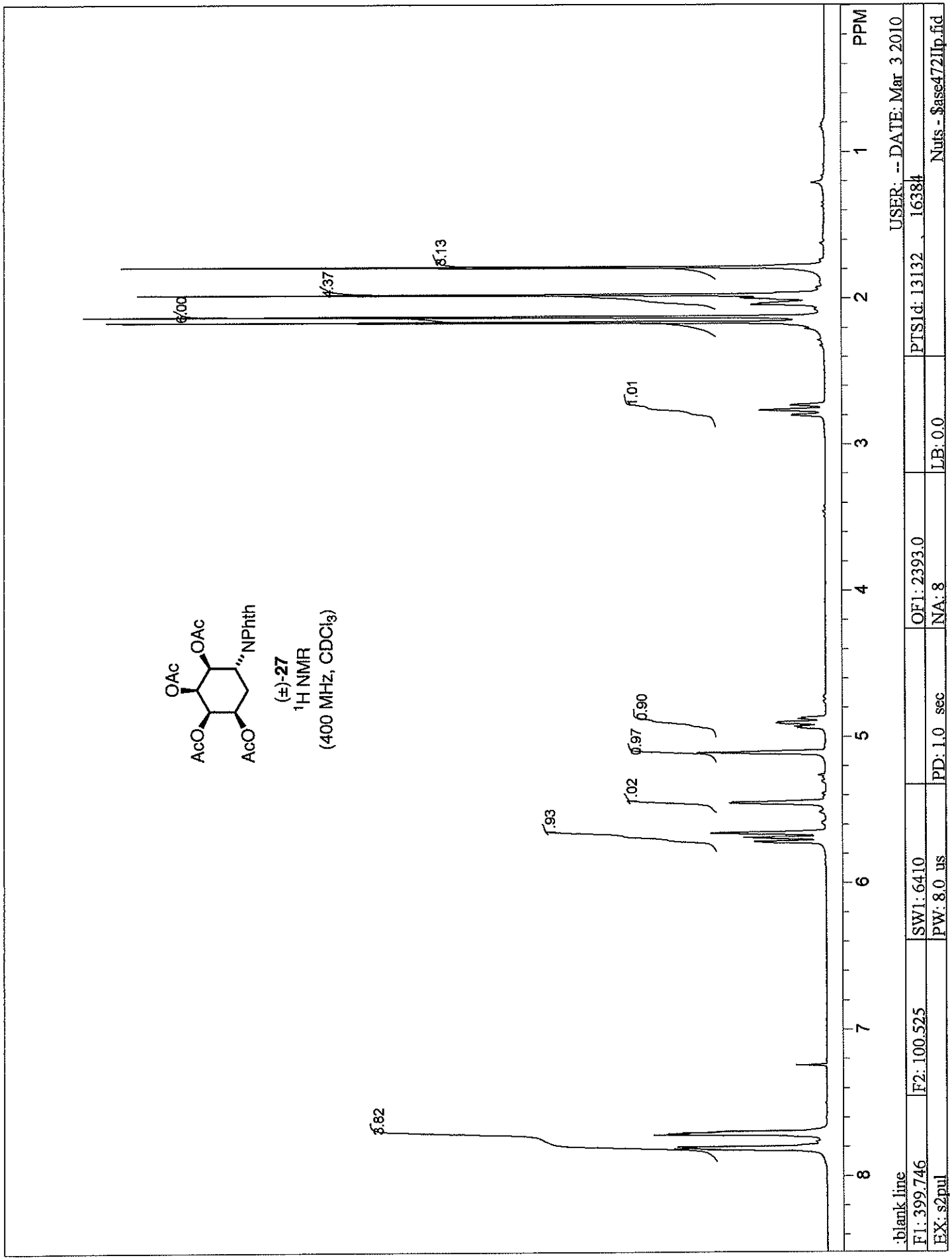



31.261
48.509
48.722
48.929
49.150
49.570
49.789
50.971
70.523
74.018
74.121
74.248
124.221
133.351
135.547
OH
OH
HO
HO
NPhth
(±)-24
13C NMR
(100 MHz, CD3OD)
ppm
20
40
60
80
100
120
140
160
USER: -- DATE: Mar 25 2009
PTS1d: 31875 32768
Nuts - $ase256pc13.fid
OF1: 10713.2
LB: 1.5
NA: 256
SW1: 24510
PW: 4.0 us
PD: 1.0 sec
F1: 100.528
F2: 399.752
EX: s2pul
.blank line


OAc
OAc
AcO
AcO
NPhth
(±)-25a
OAc
OAc
AcO
AcO
NPhth
(±)-25b
^{1}H NMR
(400 MHz, CDCl$_3$)
4.00
0.00
1.08
0.90
0.46
0.53
0.54
0.42
3.49
6.45
1.95
1.62
PPM
:blank line
F1: 399.746
F2: 100.525
EX: s2pul
SW1: 6410
PW: 8.0 us
PD: 1.0 sec
OF1: 2402.7
NA: 8
LB: 0.0
PTS1d: 13132
16384
USER: -- DATE: Dec 3 2009
Nuts - $ase421p2nd.fid


OH
OH
HO
HO
NPhth
(±)-26
1H NMR
(300 MHz, d6-DMSO)
4.00
3.92
2.02
1.10
1.05
1.07
1.11
1.11
PPM
USER: -- DATE: Feb 5 2009
STANDARD 1H OBSERVE:blank line
F1: 300.134
F2: 75.476
SW1: 4803
OF1: 1805.1
PTS1d: 9596
16384
EX: s2pul
PW: 6.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $ase212qfq.fid


32.403
38.673
38.951
39.231
39.510
39.790
40.067
40.344
46.487
68.330
69.160
70.430
75.342
122.845
131.637
134.305
168.374
OH
OH
HO
HO
NPhth
(±)-26
13C NMR
(75 MHz, d6-DMSO)
PPM
20
40
60
80
100
120
140
160
USER: -- DATE: Sep 7 2009
PTS1d: 34246 , 65536
Nuts - $as090709bal13c.fid
OF1: 8251.6
LB: 1.0
NA:
SW1: 18868
PD: 1.0 sec
PW: 7.3 us
13C OBSERVE:blank line
F1: 75.477
F2: 300.134
EX: s2pul


OAc
OAc
AcO
AcO
NPhth
(±)-27
1H NMR
(400 MHz, CDCl3)
8.82
1.93
1.02
0.97
0.90
1.01
6.00
4.37
3.13
PPM
USER: -- DATE: Mar 3 2010
.blank line
F1: 399.746
F2: 100.525
EX: s2pul
SW1: 6410
PW: 8.0 us
PD: 1.0 sec
OF1: 2393.0
NA: 8
PTS1d: 13132
16384
LB: 0.0
Nuts - $ase472IIp.fid

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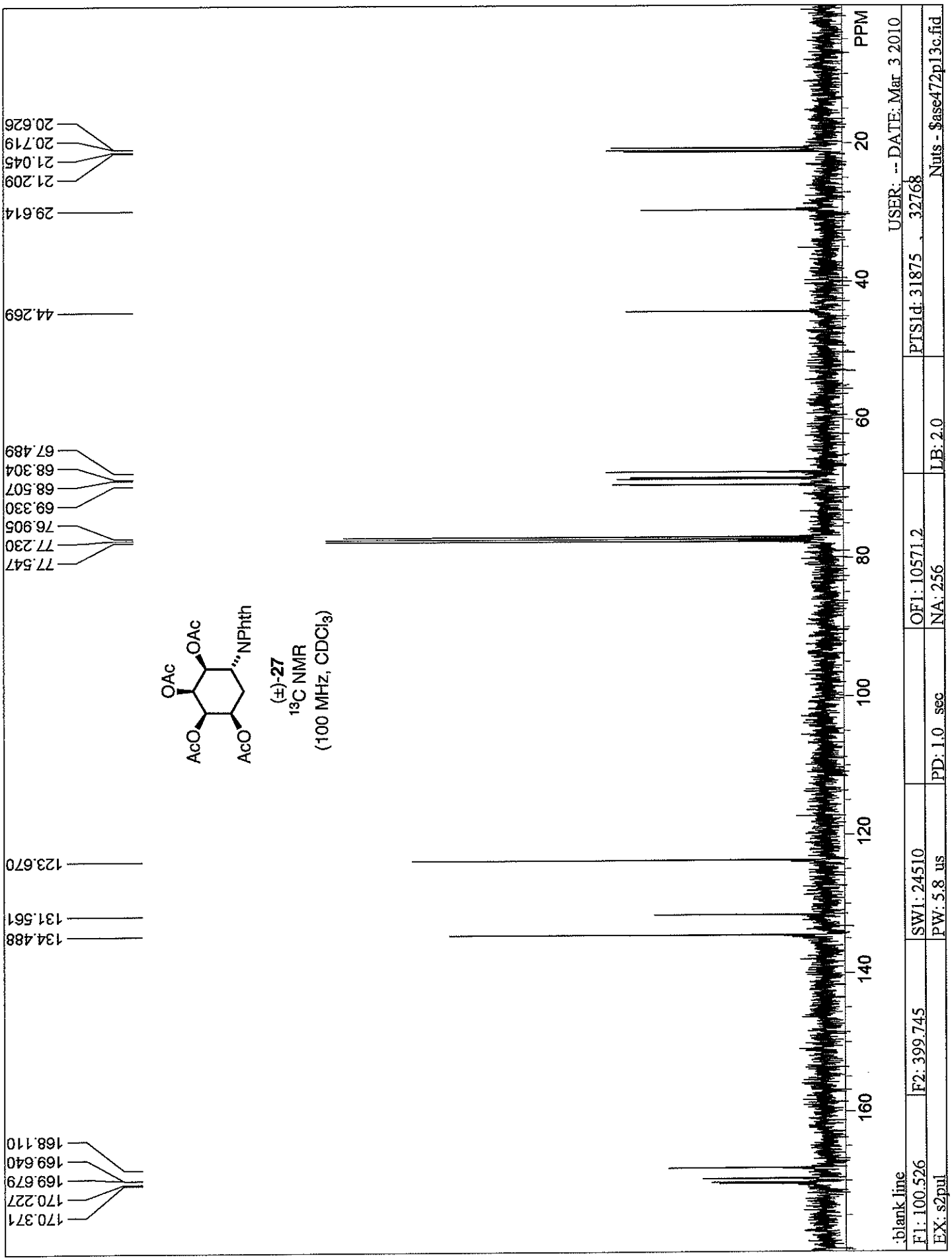
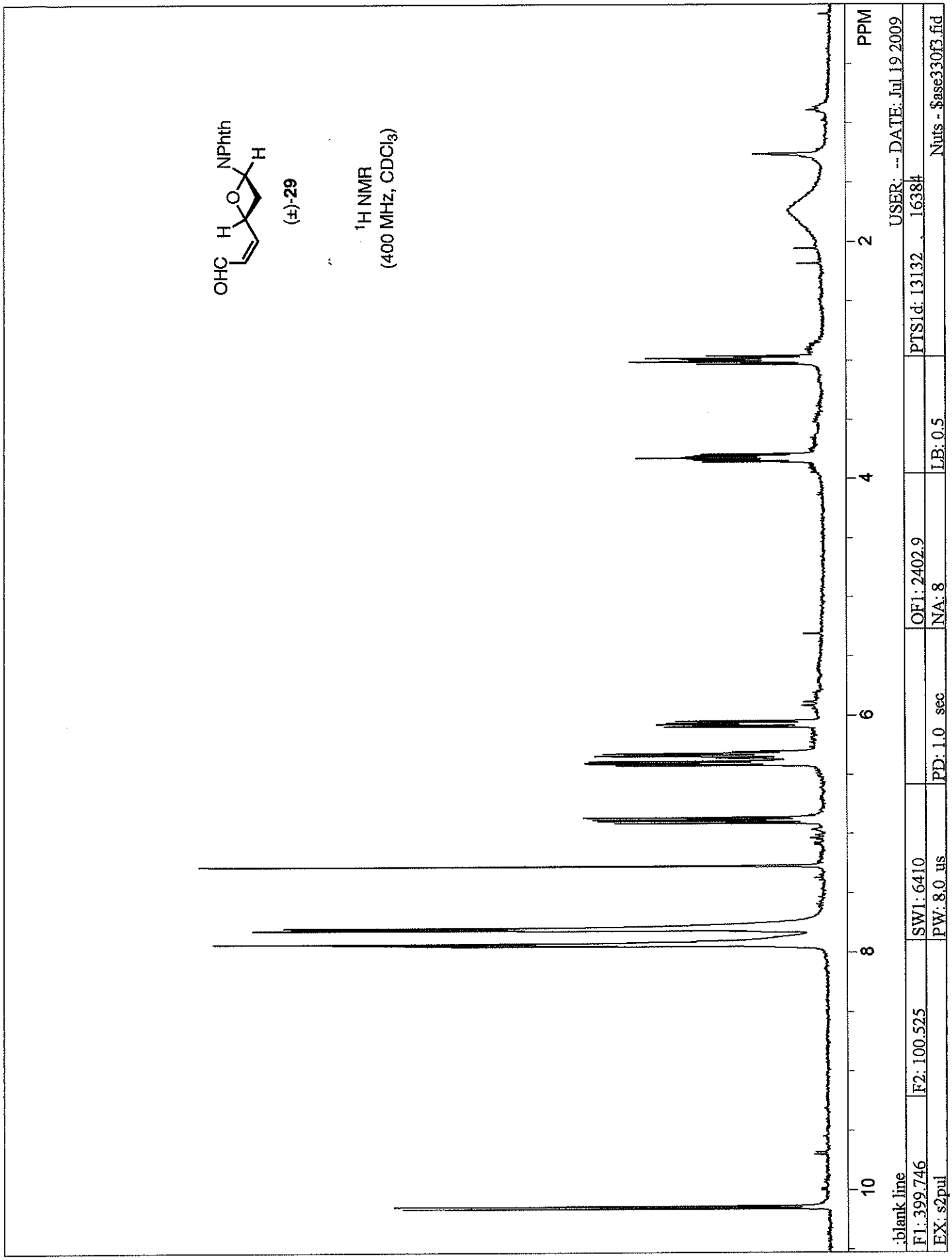
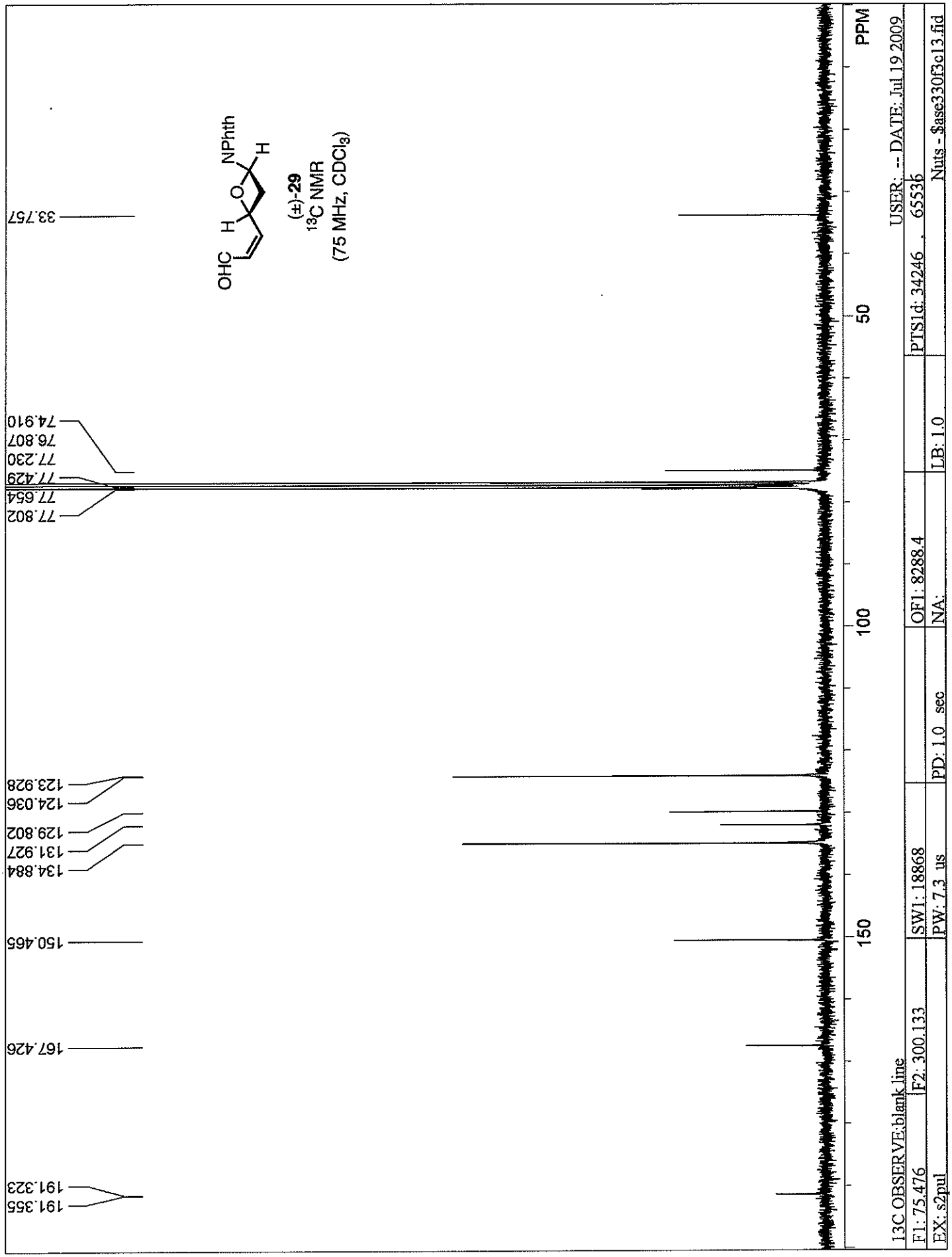



NPhth
H
O
H
OHC
(±)-29
^{1}H NMR
(400 MHz, CDCl$_3$)
PPM
10
8
6
4
2
USER: -- DATE: Jul 19 2009
.blank line
F1: 399.746
F2: 100.525
SW1: 6410
OF1: 2402.9
PTS1d: 13132 , 1638
EX: s2pul
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.5
Nuts - $ase330f3.fid


NPhth
O
H
OHC
H
(±)-29
^{13}C NMR
(75 MHz, CDCl$_3$)
33.757
74.910
76.807
77.230
77.429
77.654
77.802
123.928
124.036
129.802
131.927
134.884
150.465
167.426
191.323
191.355
PPM
50
100
150
USER: -- DATE: Jul 19 2009
13C OBSERVE:blank line
F1: 75.476
F2: 300.133
SW1: 18868
OF1: 8288.4
PTS1d: 34246 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA:
LB: 1.0
Nuts - $ase330f3c13.fid

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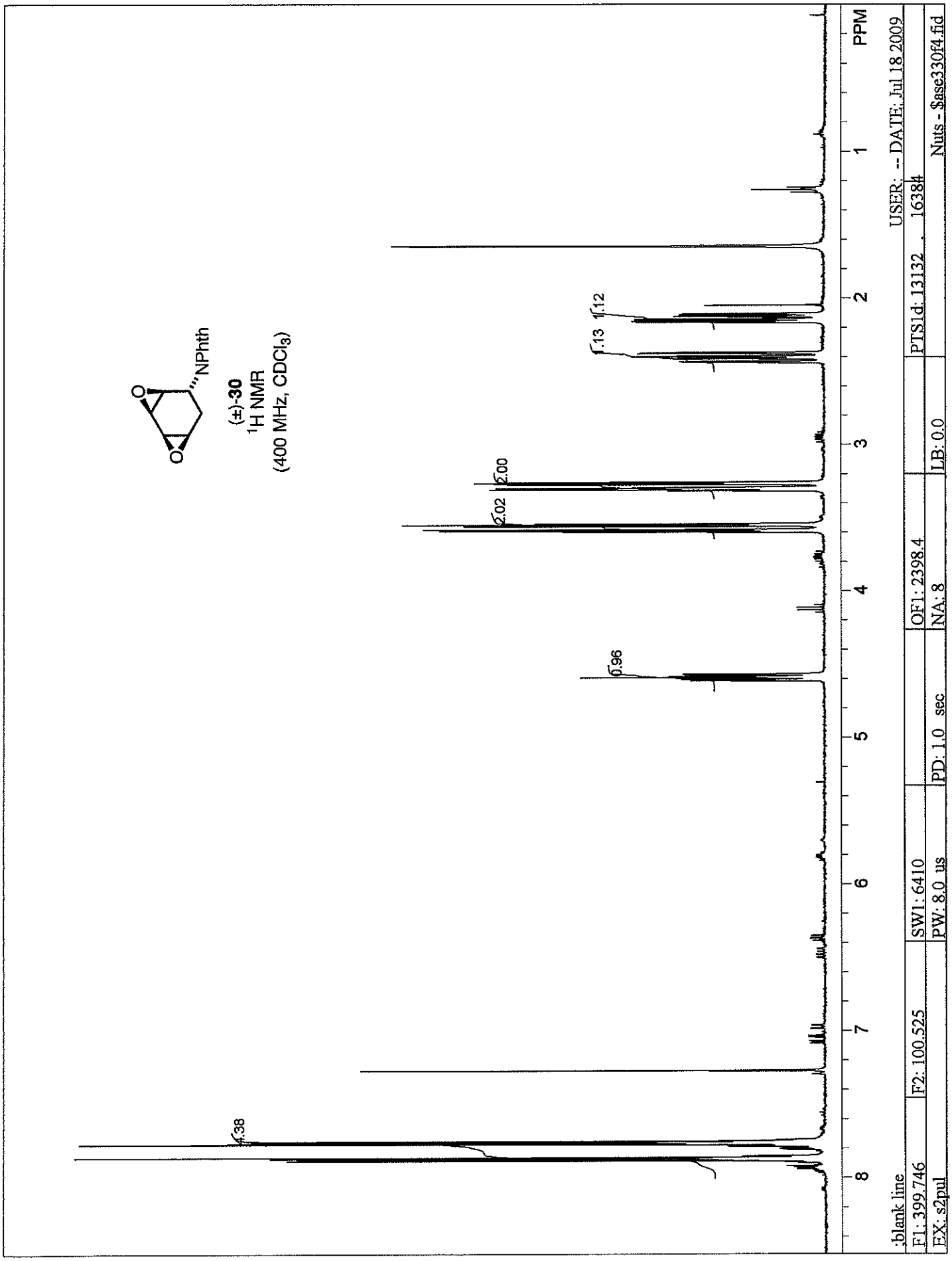

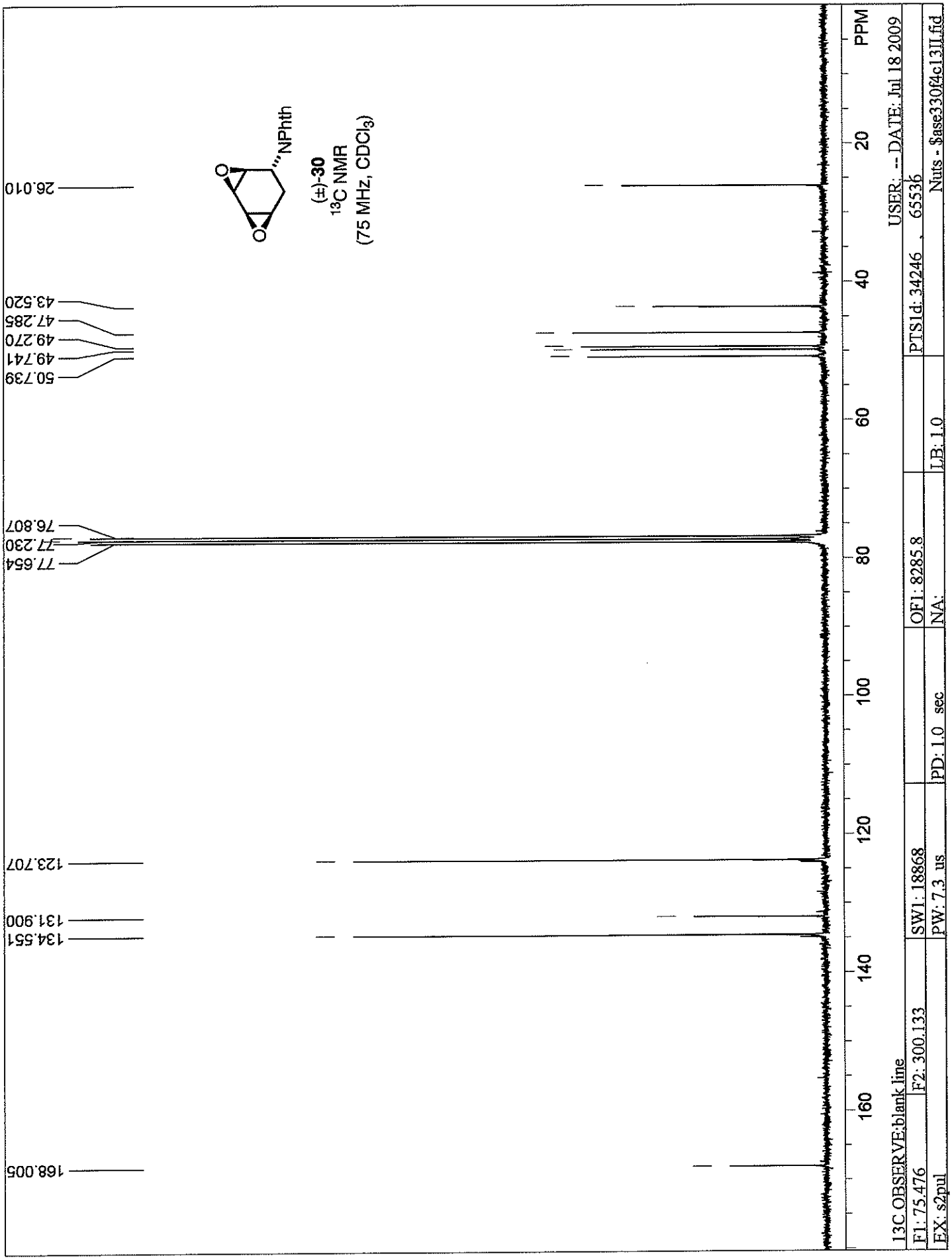
NPhth
(±)-30
^{13}C NMR
(75 MHz, CDCl$_3$)
26.010
43.520
47.285
49.270
49.741
50.739
76.807
77.230
77.654
123.707
131.900
134.551
168.005
PPM
USER: -- DATE: Jul 18 2009
13C OBSERVE:blank line
F1: 75.476
F2: 300.133
SW1: 18868
OF1: 8285.8
PTS1d: 34246 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA:
LB: 1.0
Nuts - $ase330f4c13II.fid

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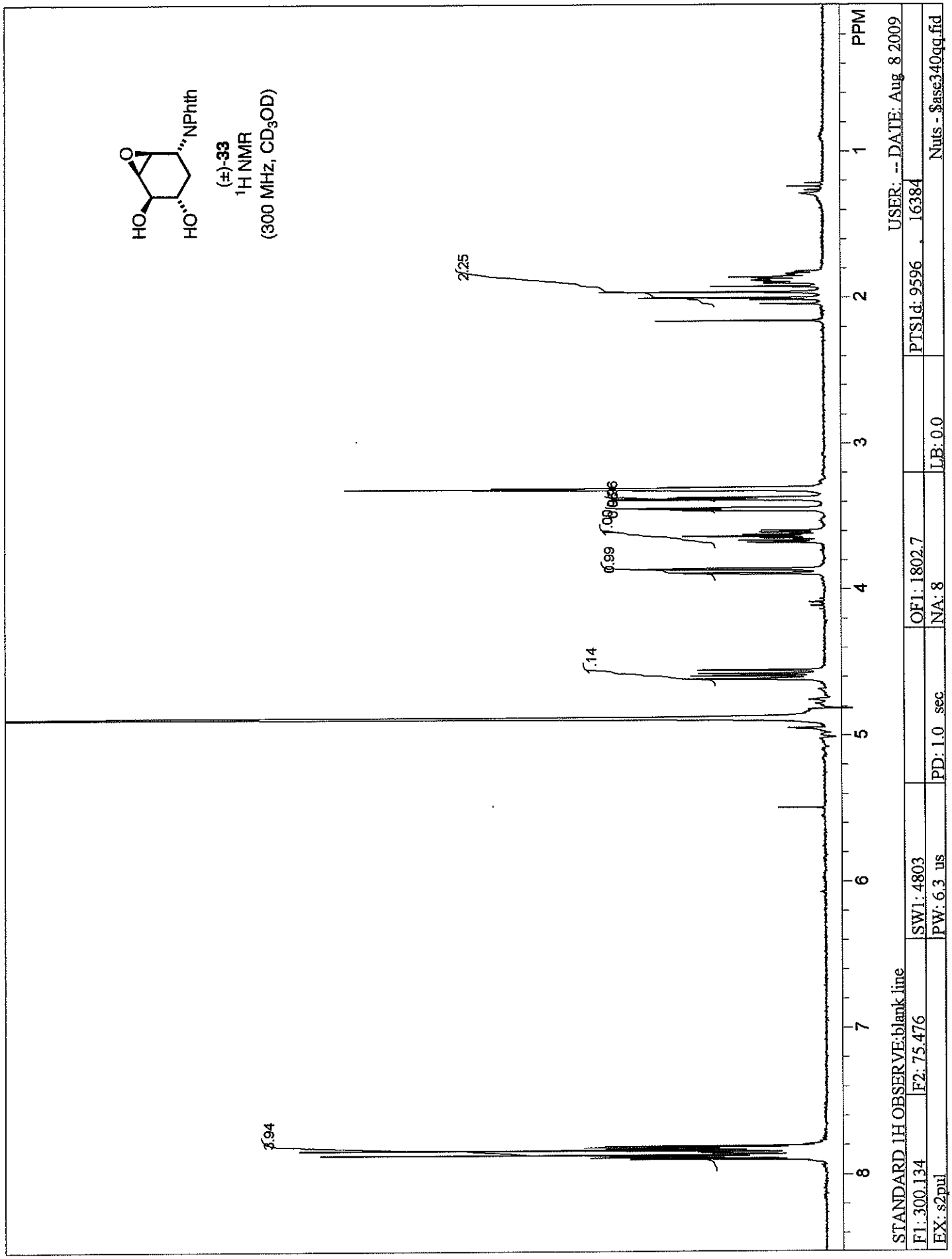

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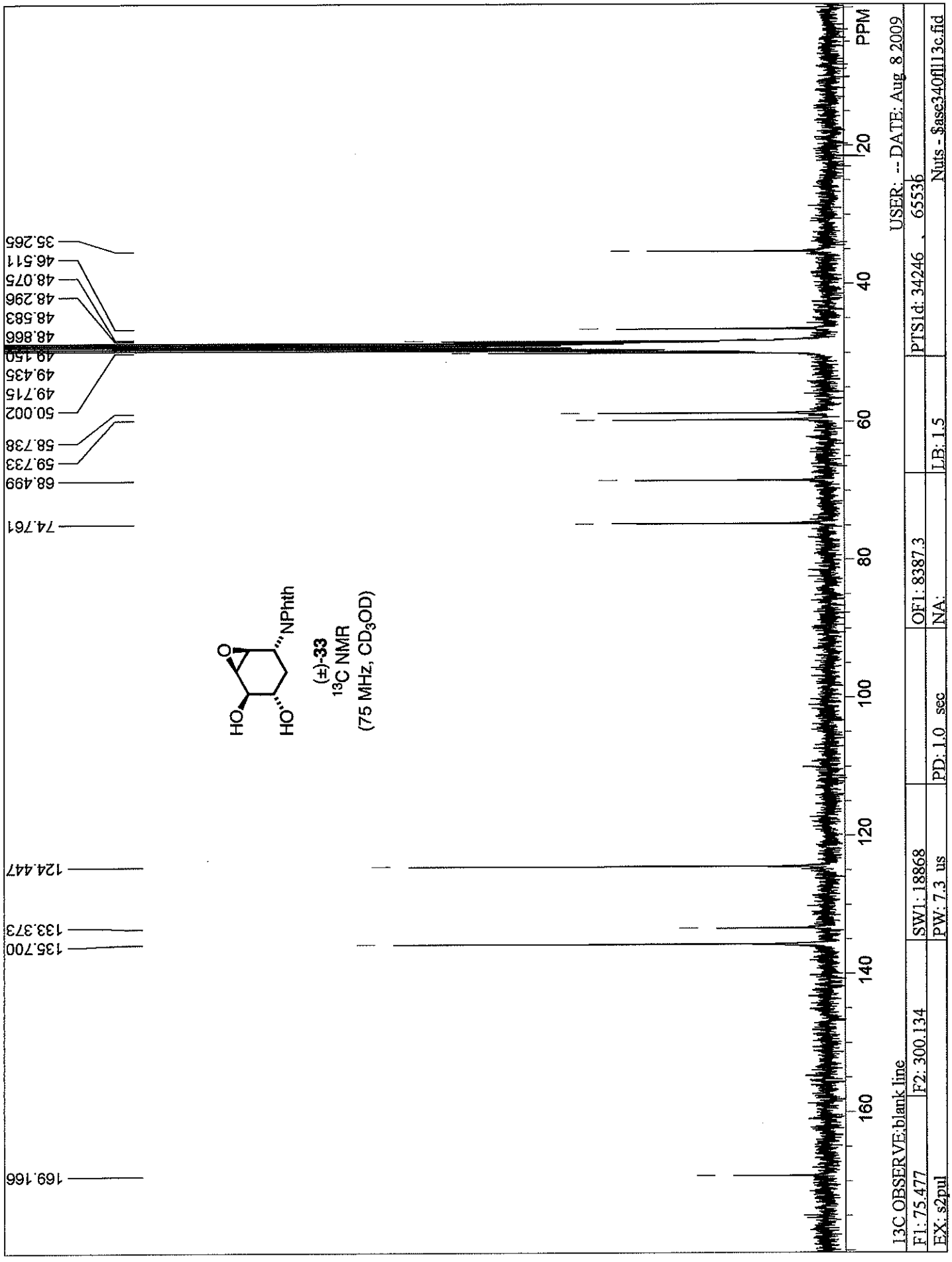

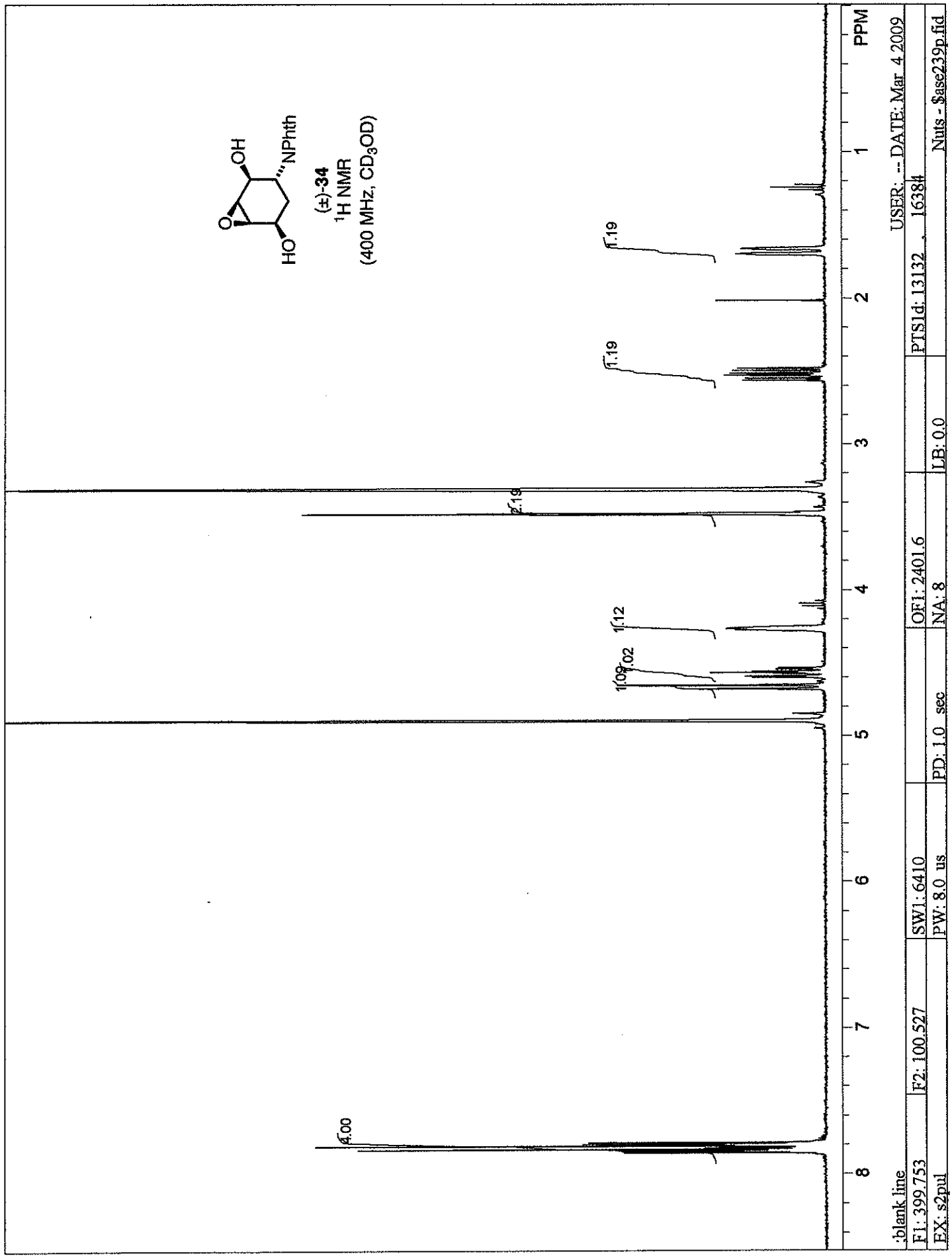
OH
NPhth
O
HO
(±)-34
^{1}H NMR
(400 MHz, CD_3OD)
4.00
2.19
1.12
1.09
1.02
1.19
1.19
PPM
USER: -- DATE: Mar 4 2009
:blank line
F1: 399.753
F2: 100.527
SW1: 6410
OF1: 2401.6
PTS1d: 13132 . 16384
EX: s2pul
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $ase239p.fid

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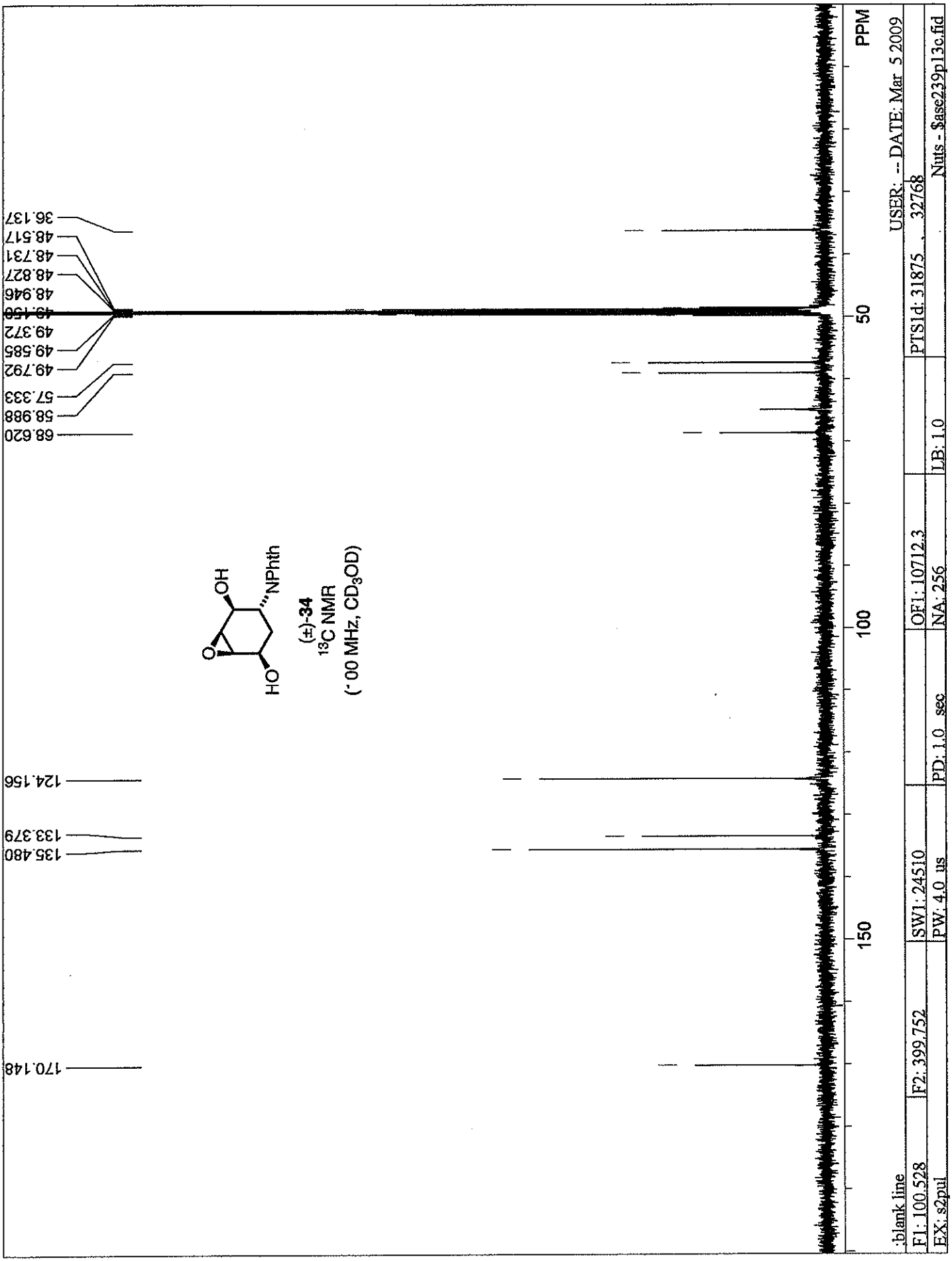
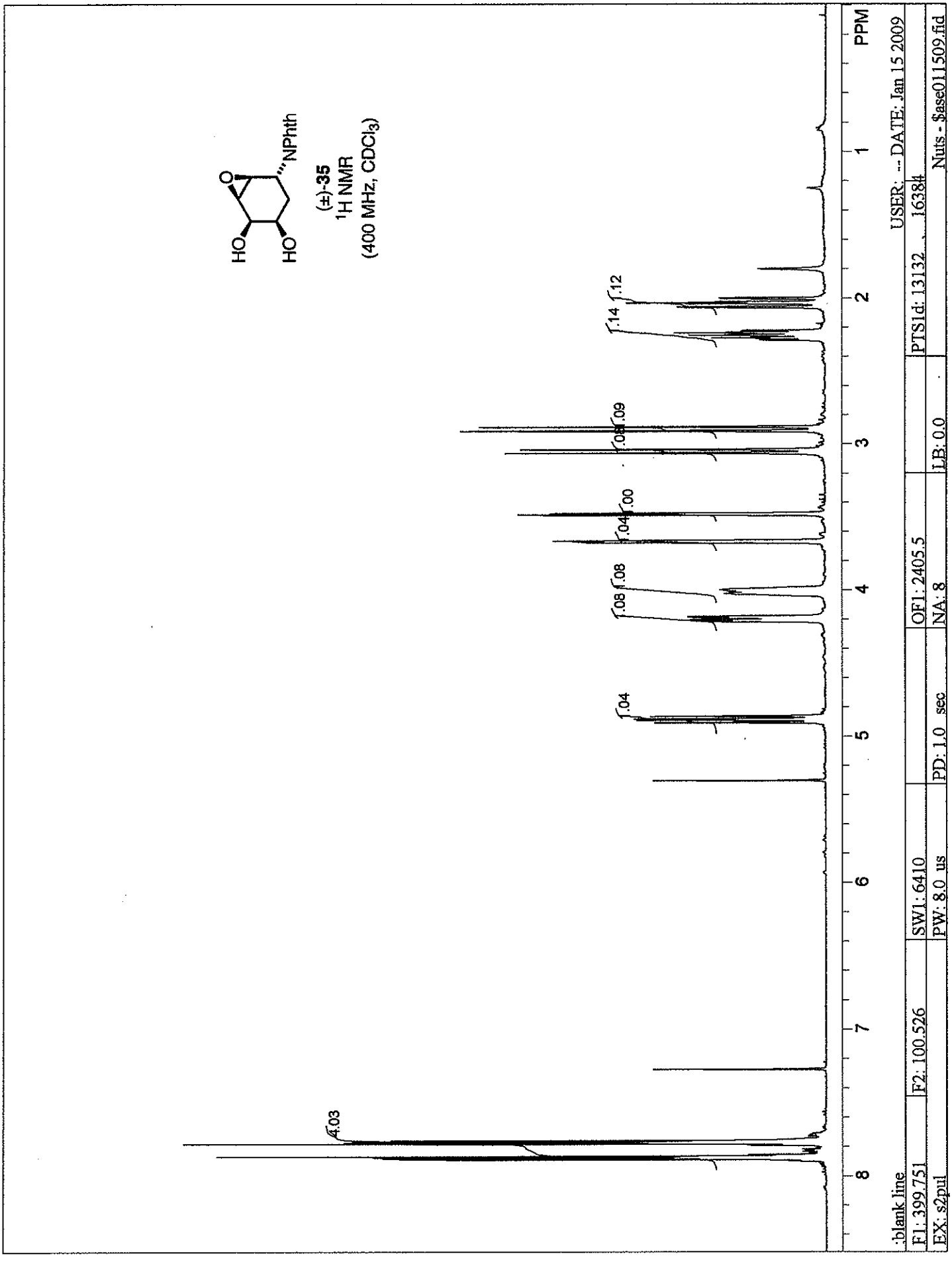
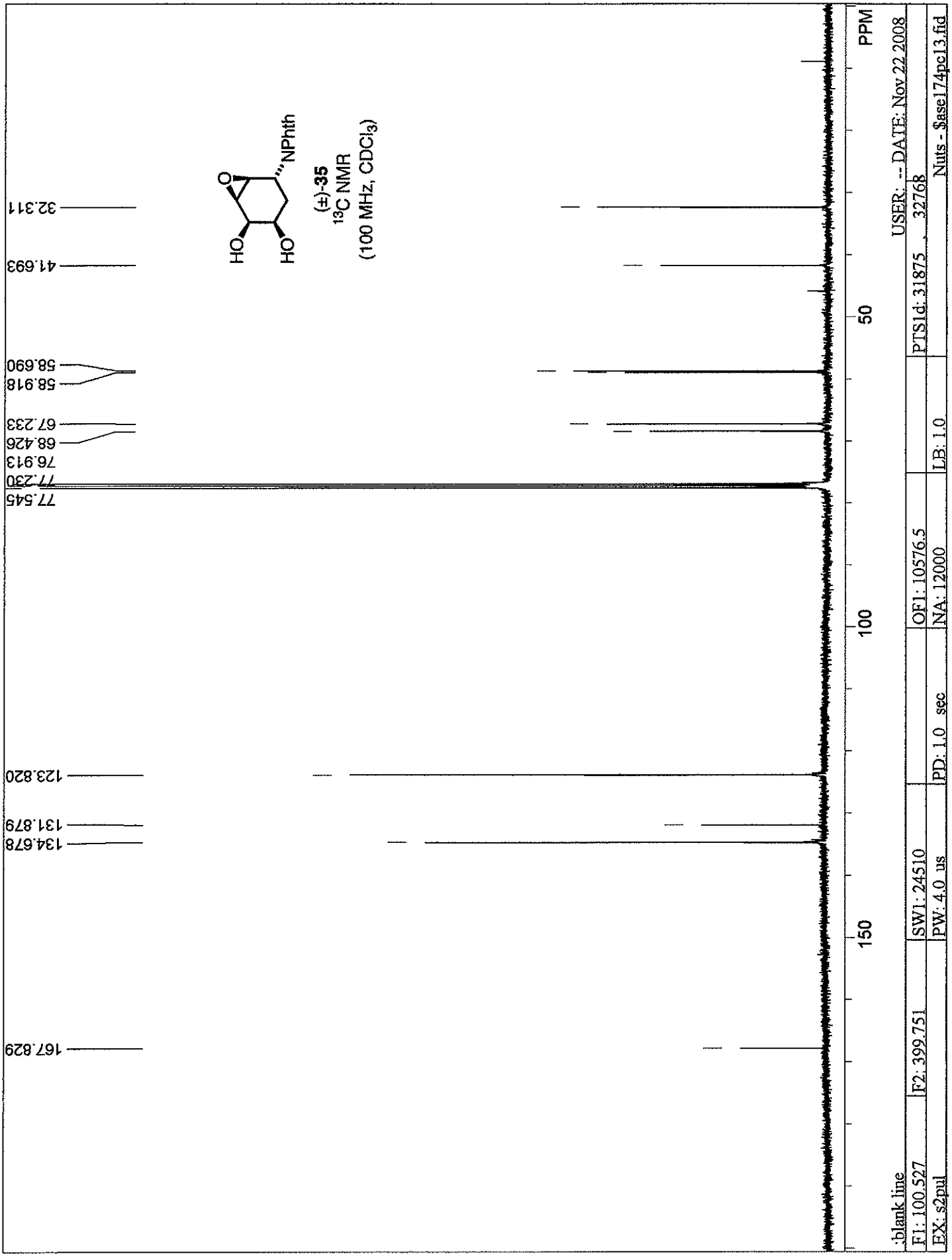
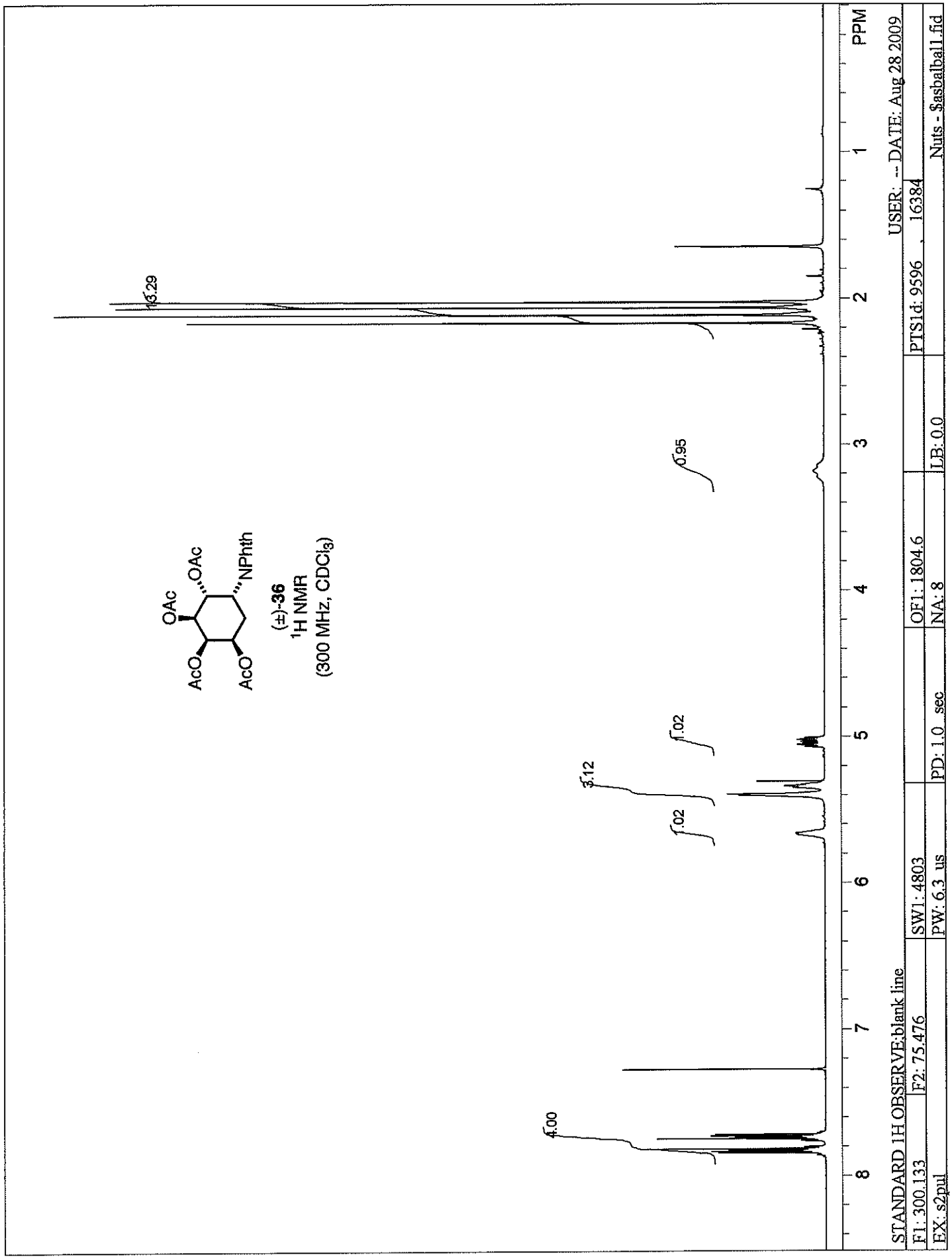



NPhth
HO
HO
(±)-35
1H NMR
(400 MHz, CDCl3)
4.03
1.04
1.08
1.08
1.04
1.00
1.08
1.09
1.14
1.12
PPM
USER: -- DATE: Jan 15 2009
PTS1d: 13132 1638#
Nuts - $ase011509.fid
.blank line
F1: 399.751
F2: 100.526
SW1: 6410
OF1: 2405.5
EX: s2pul
PW: 8.0 us
PD: 1.0 sec
NA: 8
LB: 0.0


(±)-35
^{13}C NMR
(100 MHz, $CDCl_3$)
NPhth
HO
HO
32.311
41.693
58.690
58.918
67.233
68.426
76.913
77.230
77.545
123.820
131.879
134.678
167.829
PPM
50
100
150
USER: -- DATE: Nov 22 2008
PTS1d: 31875 32768
Nuts - $ase174pc13.fid
OF1: 10576.5
NA: 12000
LB: 1.0
:blank line
F1: 100.527
F2: 399.751
SW1: 24510
PW: 4.0 us
PD: 1.0 sec
EX: s2pul


OAc
OAc
NPhth
AcO
AcO
(±)-36
^{1}H NMR
(300 MHz, $CDCl_3$)
6.29
0.95
3.12
1.02
1.02
4.00
PPM
USER: -- DATE: Aug 28 2009
STANDARD 1H OBSERVE:blank line
F1: 300.133
F2: 75.476
SW1: 4803
OF1: 1804.6
PTS1d: 9596
16384
EX: s2pul
PW: 6.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $asbalball.fid

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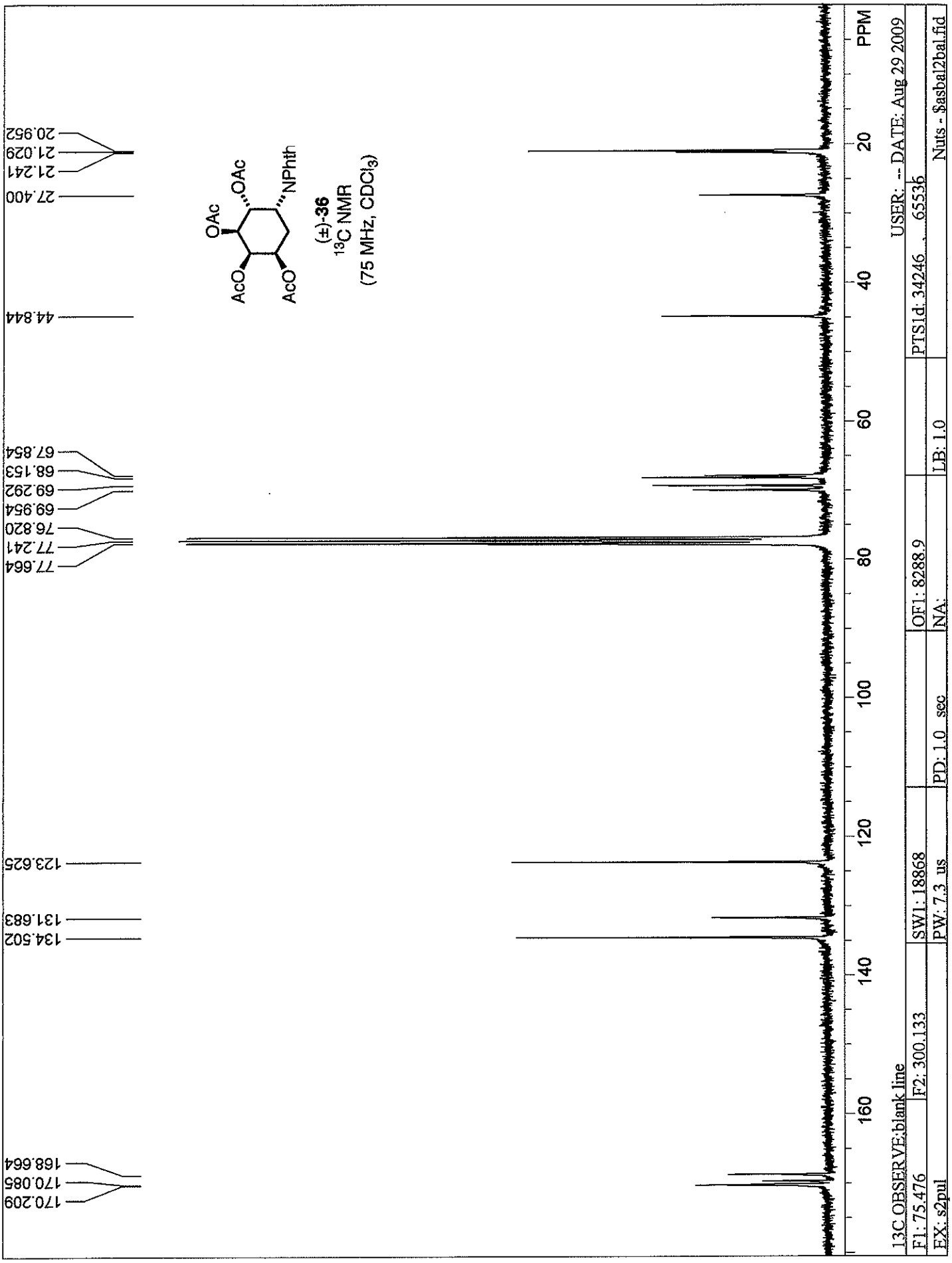

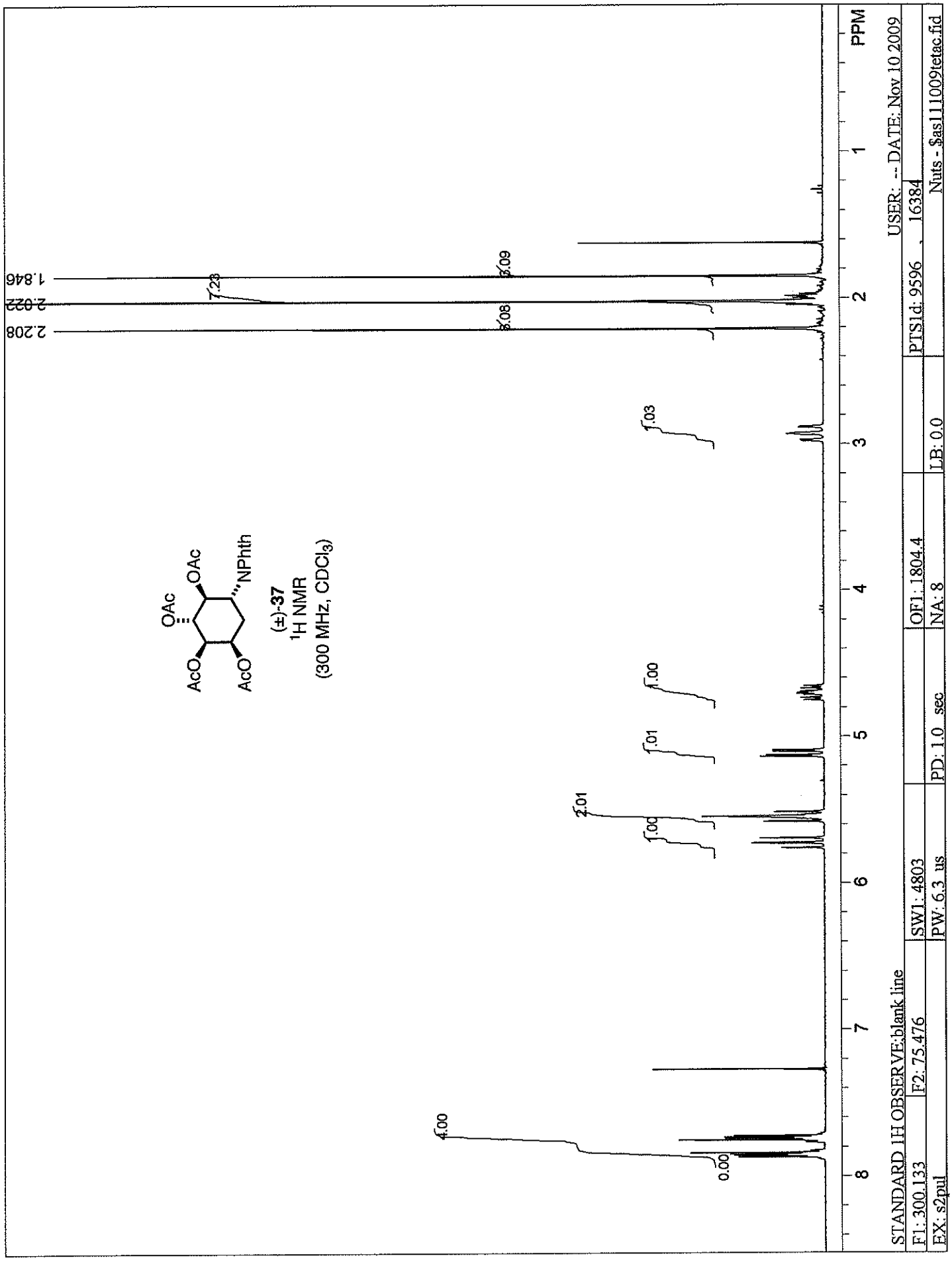
OAc
OAc
NPhth
AcO
AcO
(±)-37
^{1}H NMR
(300 MHz, $CDCl_3$)
1.846
2.022
2.208
7.23
6.09
6.08
1.03
1.00
1.01
2.01
1.00
4.00
0.00
PPM
USER: -- DATE: Nov 10 2009
STANDARD 1H OBSERVE:blank line
F1: 300.133
F2: 75.476
SW1: 4803
OF1: 1804.4
PTS1d: 9596
16384
EX: s2pul
PW: 6.3 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $as111009tetac.fid

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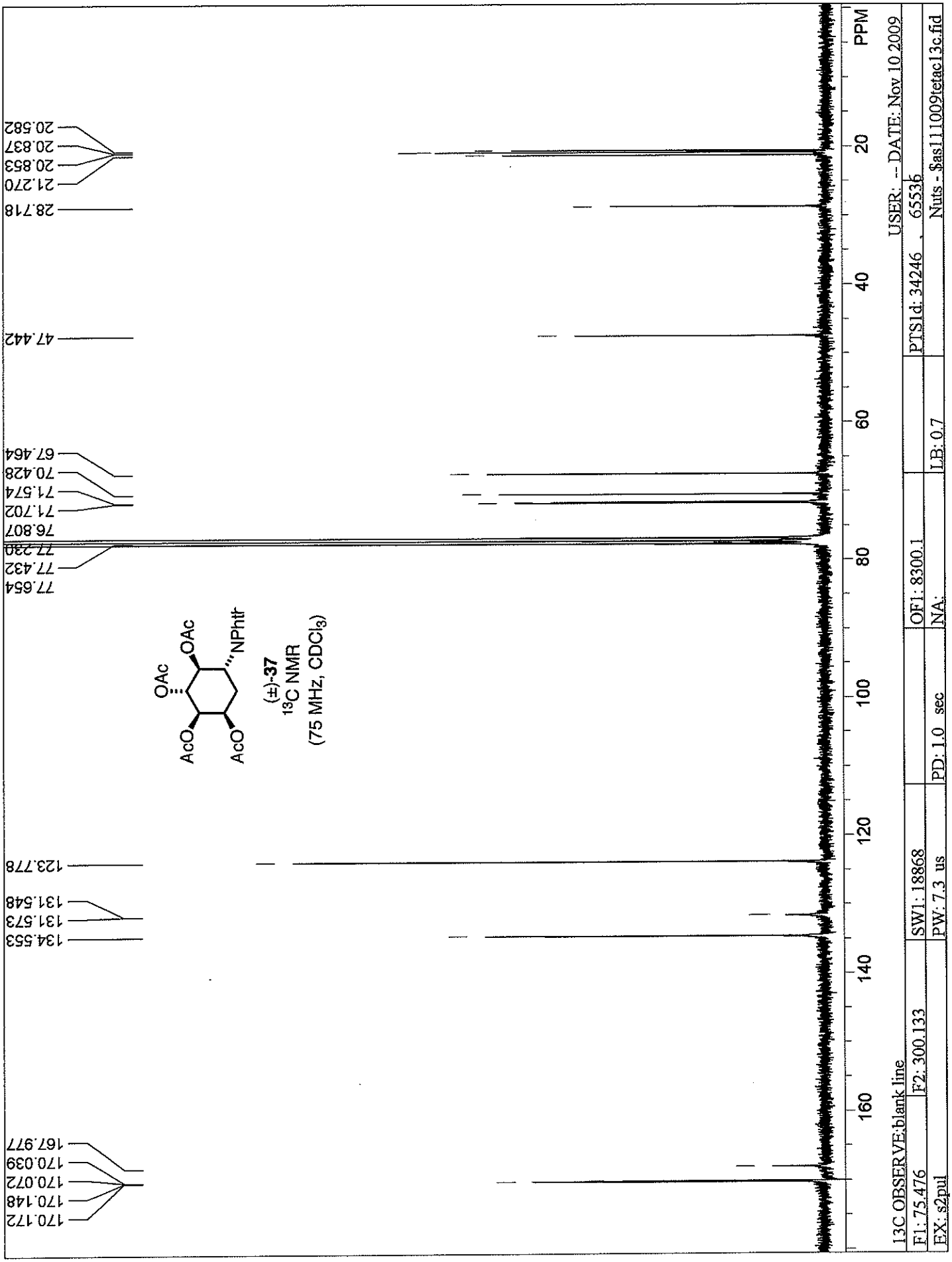
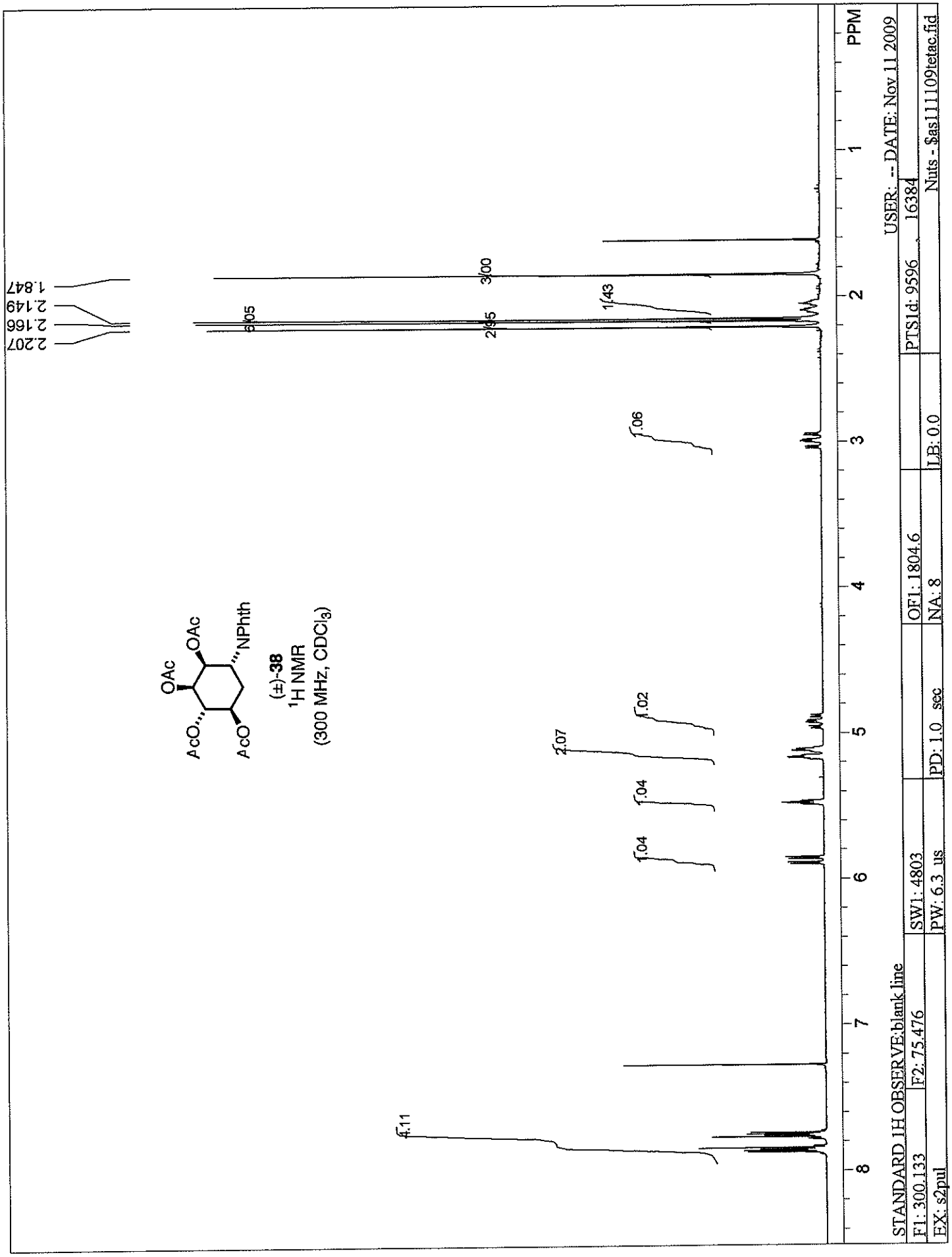
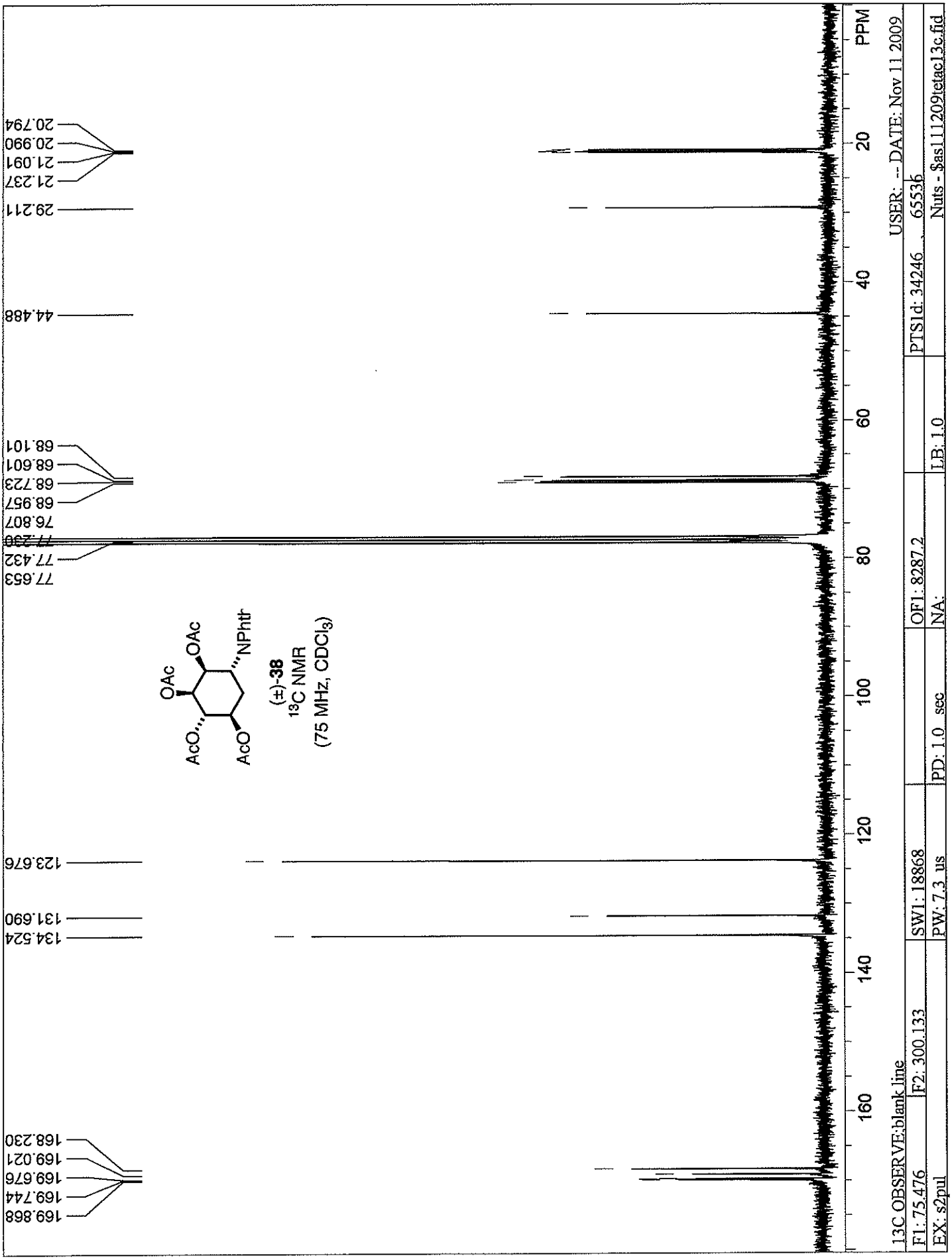
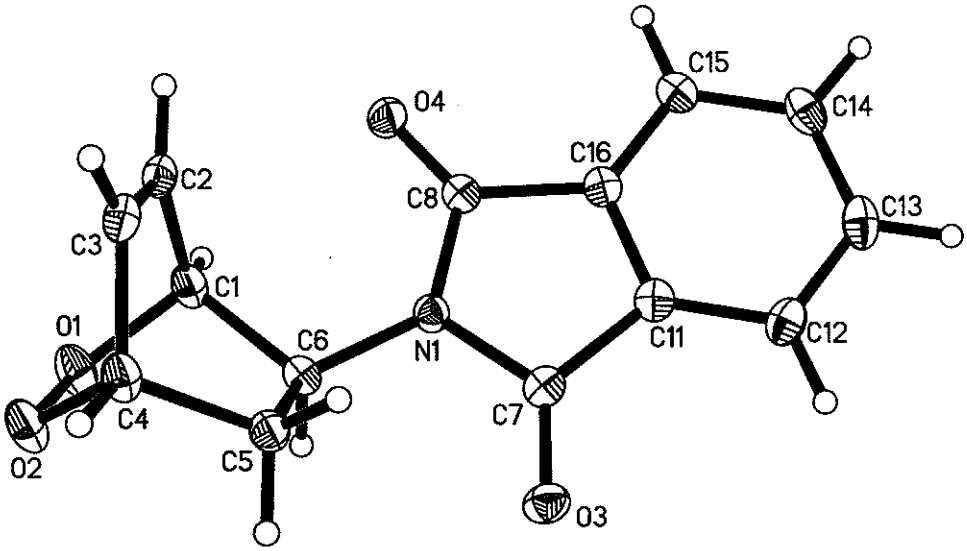
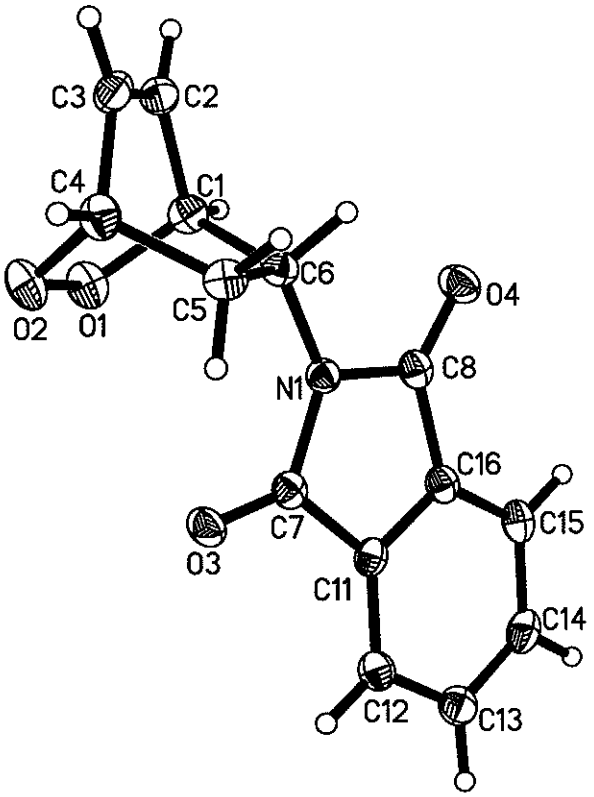



OAc
OAc
NPhth
AcO
AcO
(±)-38
^{1}H NMR
(300 MHz, CDCl$_3$)
2.207
2.166
2.149
1.847
6.05
3.00
2.05
1.43
1.06
2.07
1.02
1.04
1.04
4.11
PPM
USER: -- DATE: Nov 11 2009
STANDARD 1H OBSERVE:blank line
F1: 300.133
F2: 75.476
EX: s2pul
SW1: 4803
PW: 6.3 us
PD: 1.0 sec
OF1: 1804.6
NA: 8
LB: 0.0
PTS1d: 9596
16384
Nuts - $as111109tetac.fid

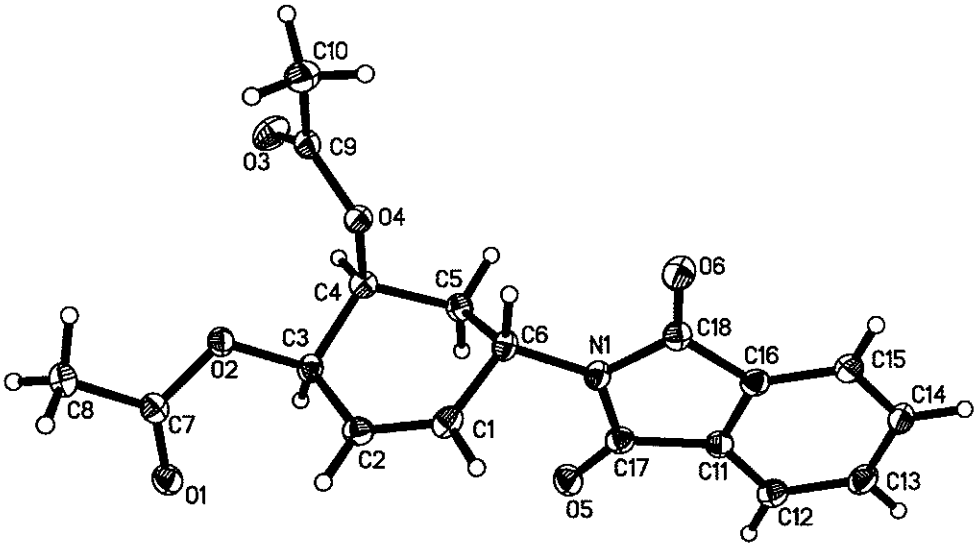

OAc
OAc
NPhth
AcO
AcO
(±)-38
^{13}C NMR
(75 MHz, CDCl$_3$)
169.868
169.744
169.676
169.021
168.230
134.524
131.690
123.676
77.653
77.432
77.230
76.807
68.957
68.723
68.601
68.101
44.488
29.211
21.237
21.091
20.990
20.794
PPM
13C OBSERVE:blank line
F1: 75.476
F2: 300.133
EX: s2pul
SW1: 18868
PW: 7.3 us
PD: 1.0 sec
OF1: 8287.2
NA:
LB: 1.0
PTS1d: 34246
65536
USER: -- DATE: Nov 11 2009
Nuts - $as111209tetac13c.fid



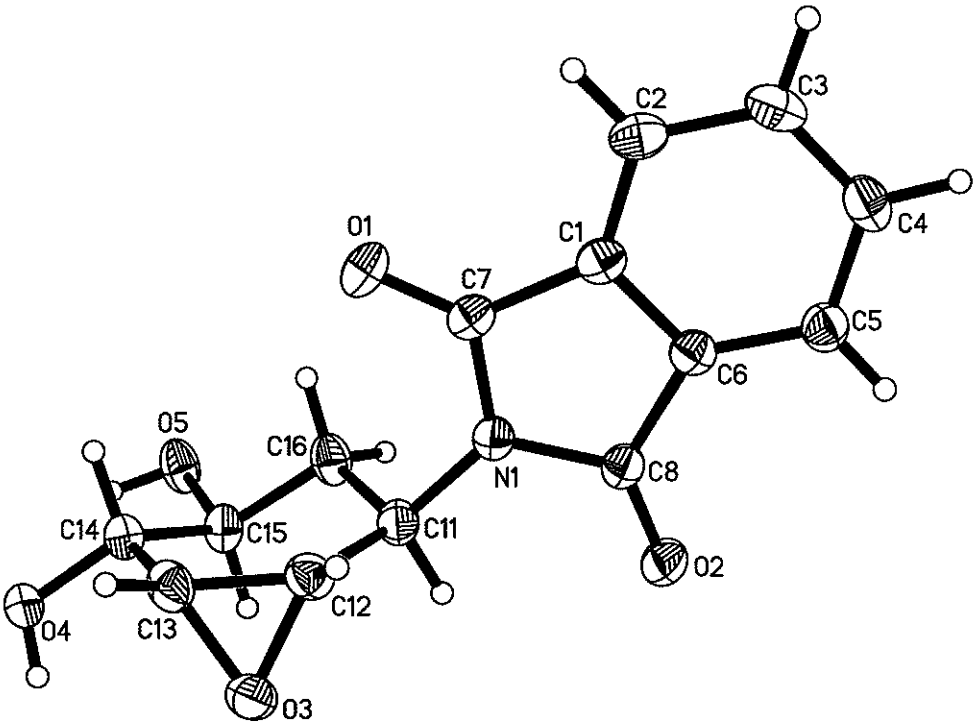
ORTEP of (±)-**9**



ORTEP of (±)-**10**



ORTEP of (±)-**18**



ORTEP of (±)-**33**

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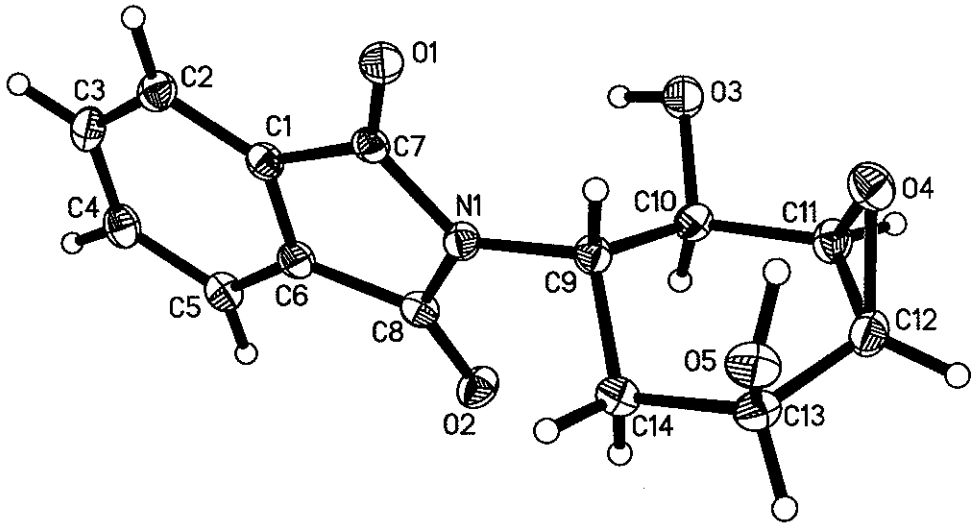
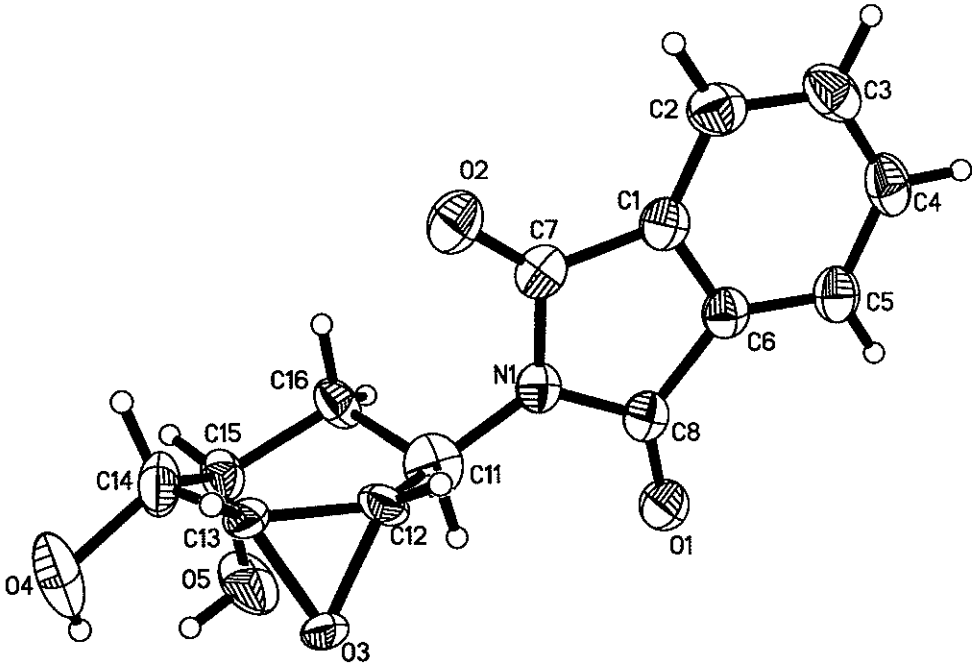


ORTEP of (±)-**34**



ORTEP of (±)-**35**