

**Gram Scale Synthesis of 7-Methoxy-*O*-methylumkonal, Clausine-O,
Clausine-K, Clausine-H, 7-Methoxylumkonal, and Methyl
2-Hydroxy-7-methoxy-9*H*-carbazole-3-carboxylate**

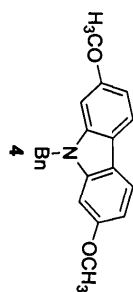
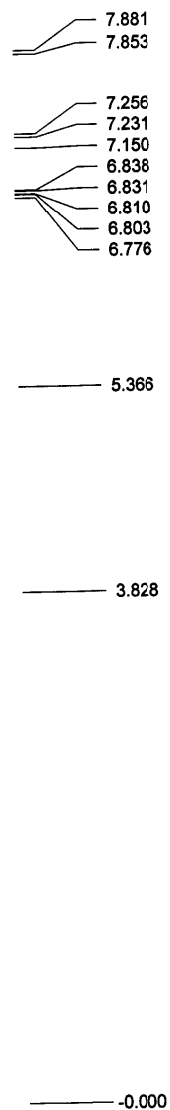
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Zhejiang University, Hangzhou 310027, Zhejiang, People's Republic of China

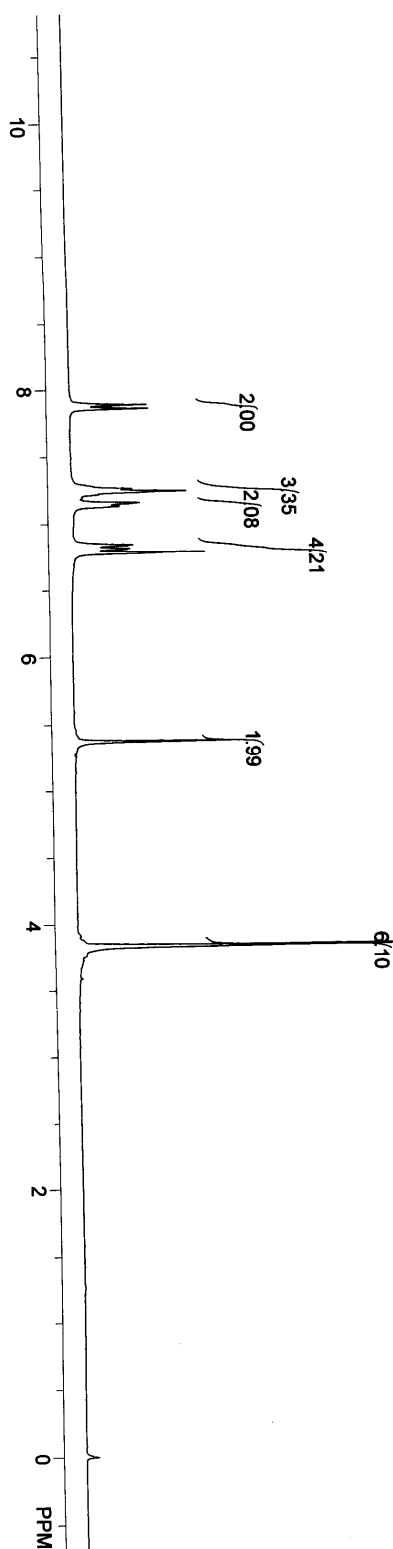
Fax: (+86) 21-62609305

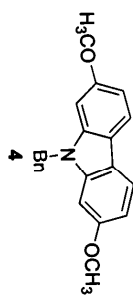
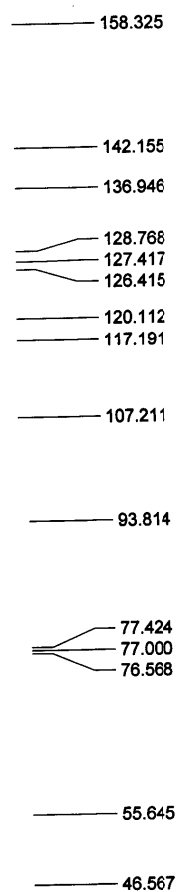
E-mail: masm@sioc.ac.cn

Supporting Information



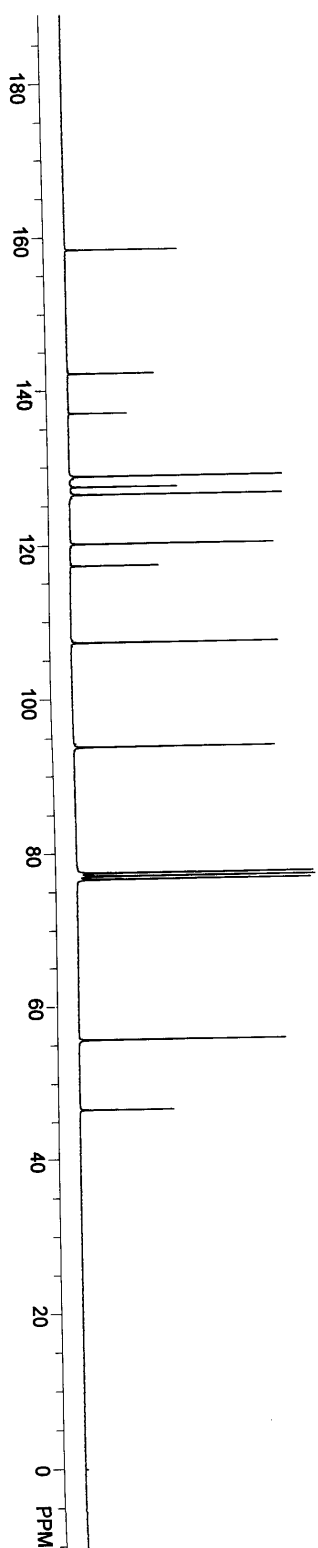
Spec: CDCl₃
Fri Sep 20 13:18:16 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
P1S1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SWH = 6188.12 Hz

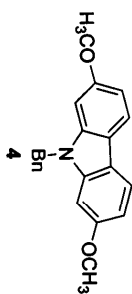
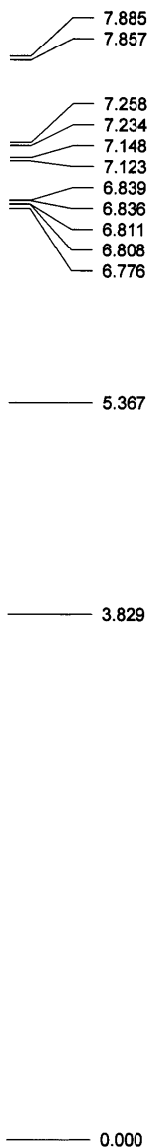




spect. CDCl₃,
 Sat Sep 28 13:18:16 2013
 USER: nmr
 SOLVENT: nmr
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 12376
 P1 = 12376
 P1S1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

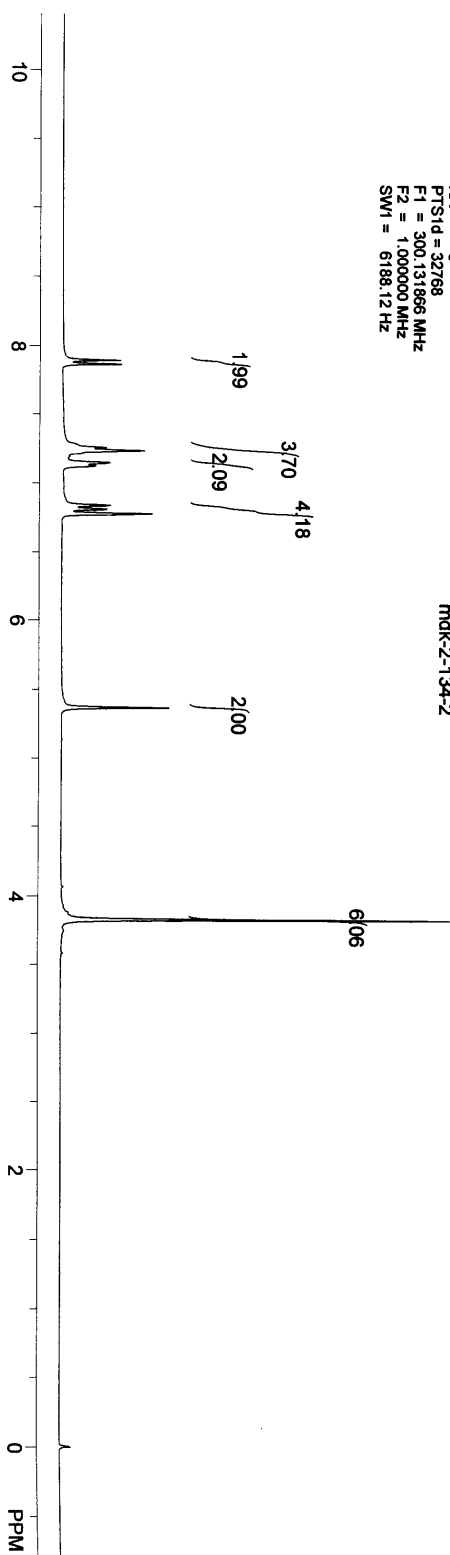
mdk-2-134

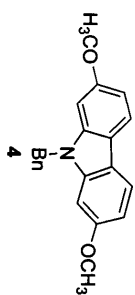
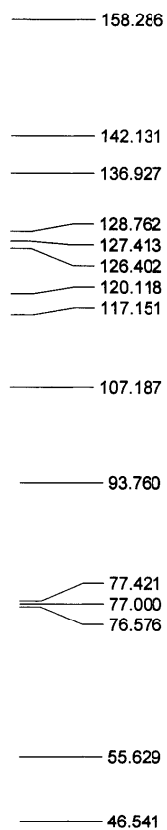




Spect. CDCl₃
 Sat Sep 28 13:15:56 2013
 USER: mhr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTSD = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

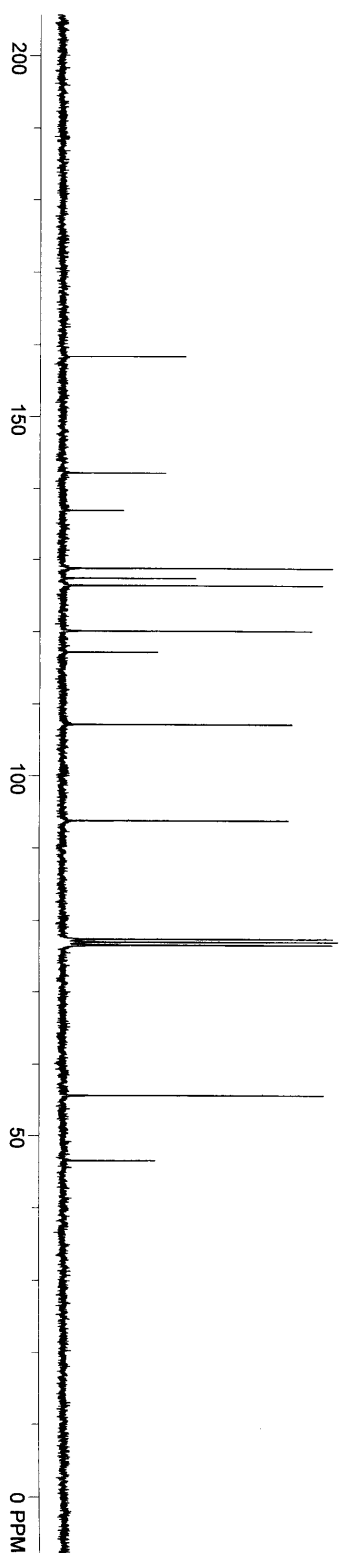
mdk-2-134-2

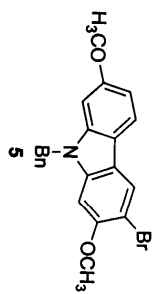
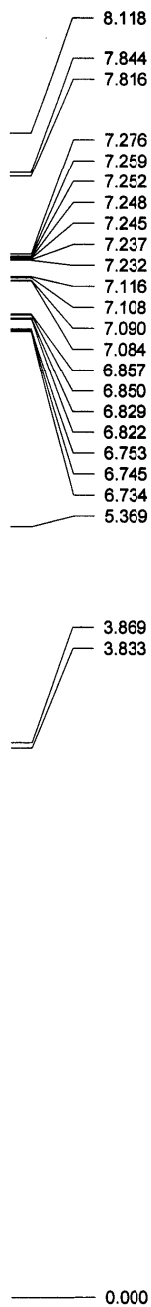




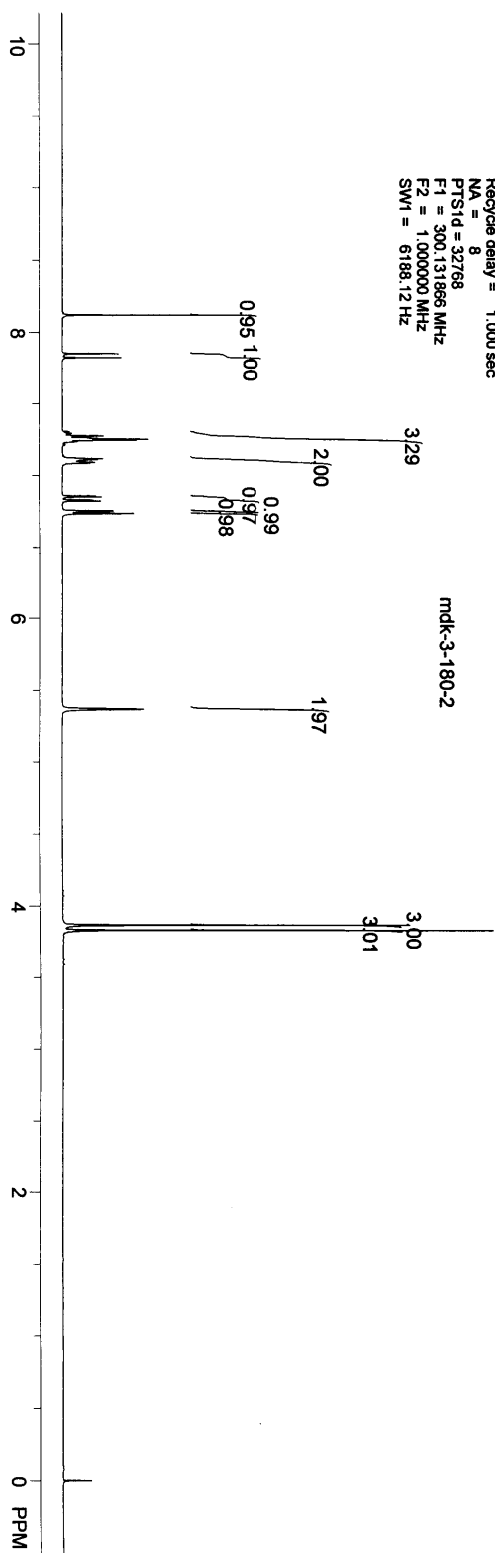
spect, CDCl₃,
 Fri Sep 20 13:20:35 2013
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 288
 P1S1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.30 Hz

mdk-2-134-2

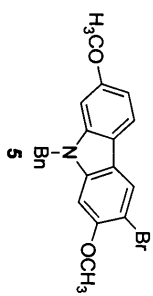




spect, CDCl₃,
Wed Jan 22 13:28:27 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTStd = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

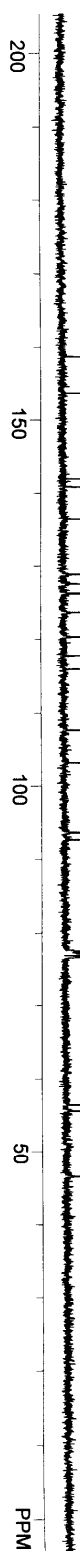


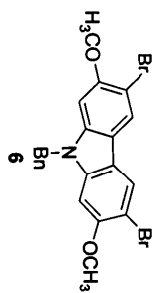
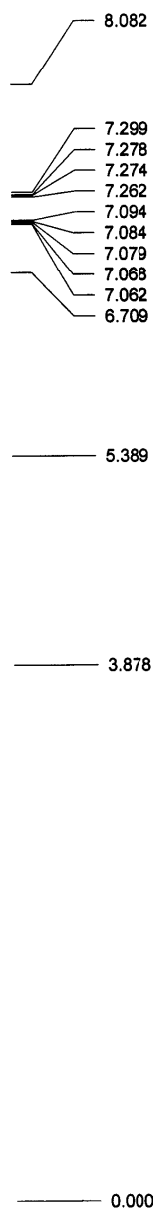
158.574
153.625
141.961
140.832
136.438
128.831
127.552
126.234
123.631
120.324
117.739
115.973
107.724
103.123
93.667
92.678
77.422
77.030
76.538
56.419
55.594
46.435



spec: CDCl₃
Mon Jan 20 12:41:53 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 280
PTSD = 32768
F1 = 75.476807 MHz
F2 = 100.000000 MHz
SWH = 22727.27 Hz

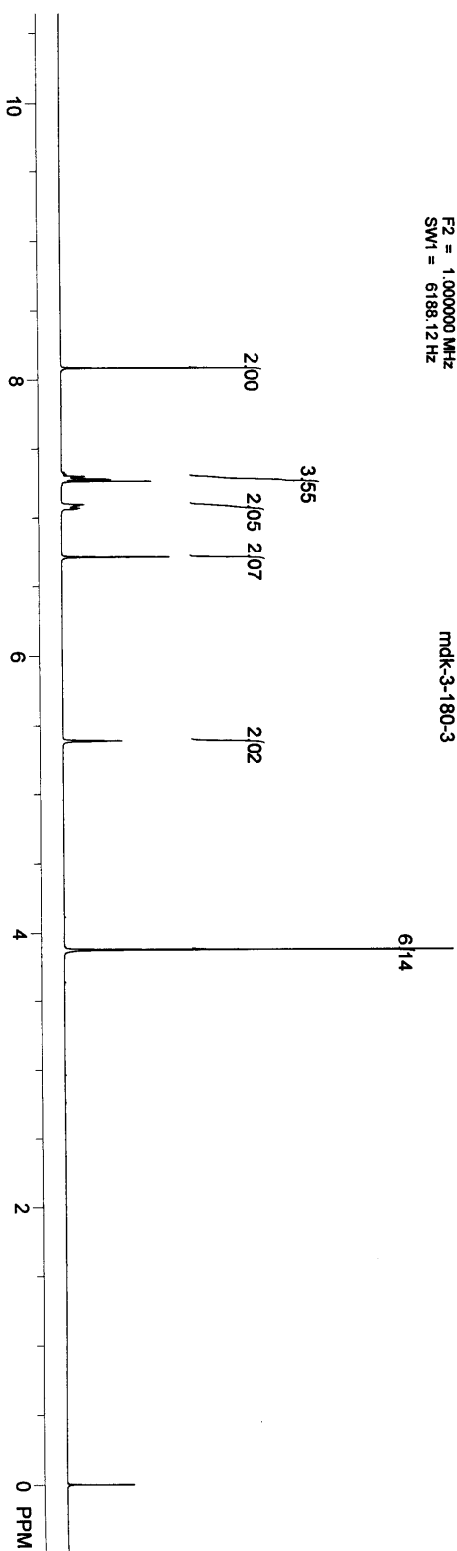
mdk-3-180-2



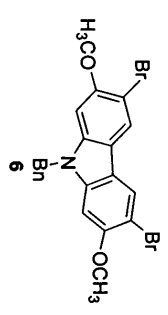


spect, CDCl₃
Wed Jan 22 07:05:50 2014
USER: mhr
SOLVENT: mhr
Experiment: zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTSD = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SWH = 6188.12 Hz

mdk-3-180-3

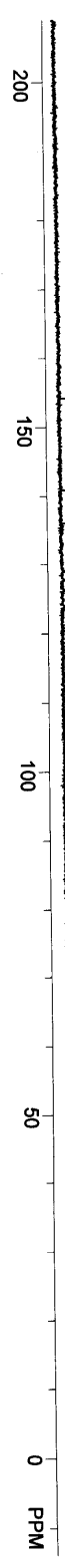


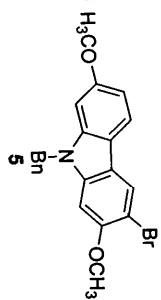
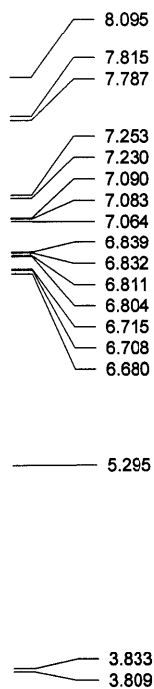
- 154.191
- 140.862
- 136.054
- 128.991
- 127.800
- 126.199
- 123.945
- 116.770
- 103.820
- 92.849
- 77.422
- 77.000
- 76.579
- 56.513
- 46.726



Specd CDCl₃
 Tue Jan 07 13:50:29 2014
 USER: nmr
 SOLVENT:
 Experiment: = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 10878
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SWH = 22727.27 Hz

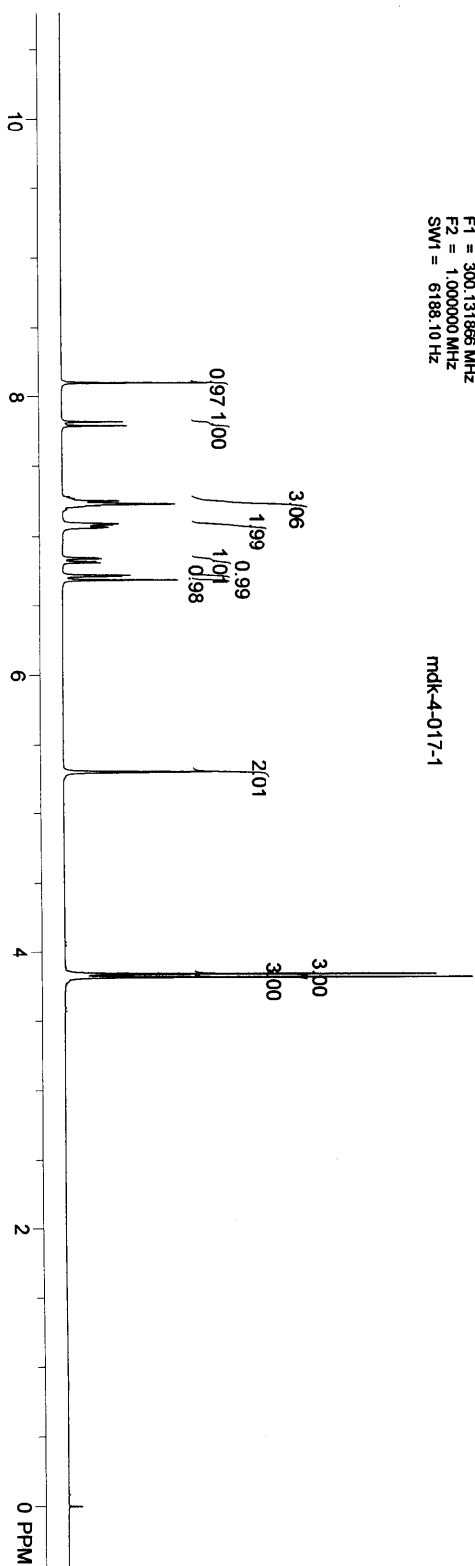
mdk-3-180-3

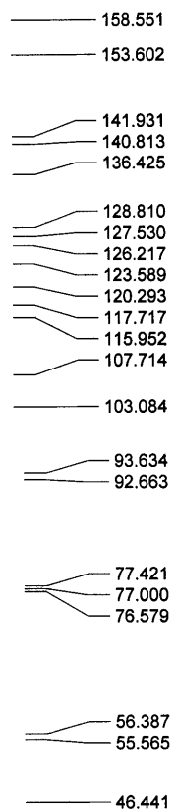




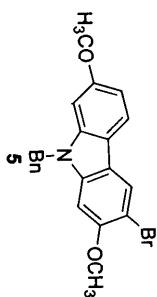
spect. CDCl₃
 Wed Feb 26 00:37:25 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 P2 = 300.131666 MHz
 F2 = 1,000,000 MHz
 SWH = 6188.10 Hz

mdk-4-017-1

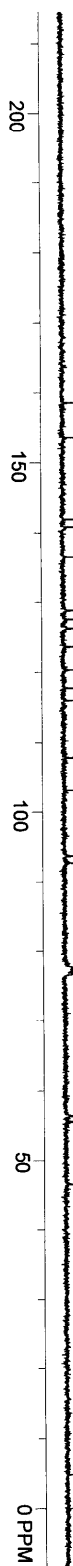


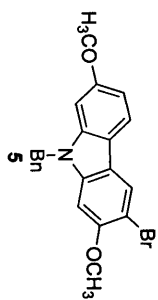


spec1, CDCl₃,
Wed Feb 26 00:41:41 2014
USER: nmr
SOLVENT:
Experiment = z9p930
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 278
PTStd = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SWH = 22727.30 Hz



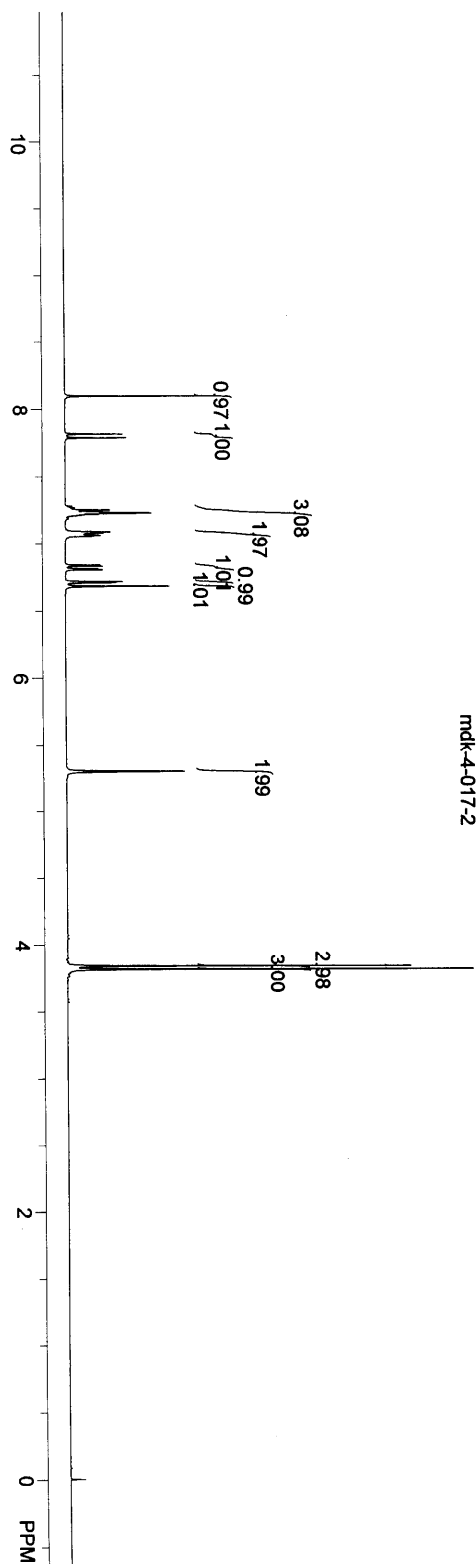
mdk-4-017-1



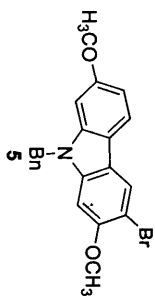


spect, CDCl₃,
 Wed Feb 26 01:45:18 2014
 USER: nmr
 SOLVENT: nmr
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32758
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

mdk-4-017-2

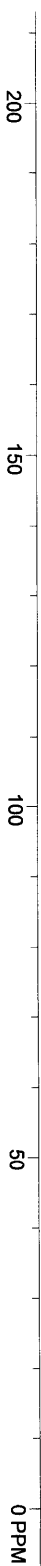


158.545
 153.604
 141.932
 140.813
 136.425
 128.807
 127.526
 126.216
 123.587
 120.292
 117.719
 115.953
 107.712
 103.094
 93.627
 92.654
 77.423
 77.000
 76.575
 56.385
 55.553
 46.440



spect, CDCl₃,
 Wed Feb 26 01:46:54 2014
 USER: nmf
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 1040
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SWH = 22727.30 Hz

mdk-4-017-2



11.180

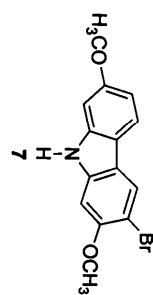
8.244
7.955
7.927

7.138
7.004
6.998
6.805
6.798
6.777
6.770

3.947
3.861

3.423

2.540



spect. DMSO-d₆
F1: Oct 25 01:12:10 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTSD = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SWH = 6188.12 Hz

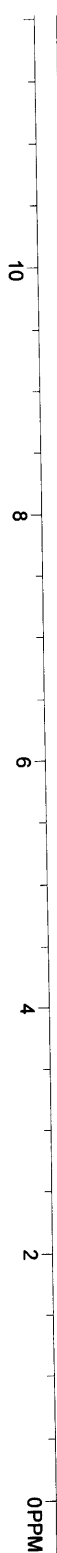
mdk-2-091

0.99

0.96 1.00

0.99 1.02
1.01

3.02
3.19

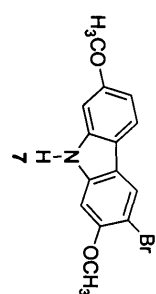


158.949
153.702
142.137
140.943

124.113
121.442
118.403
116.436
108.860
102.643
95.767
95.637

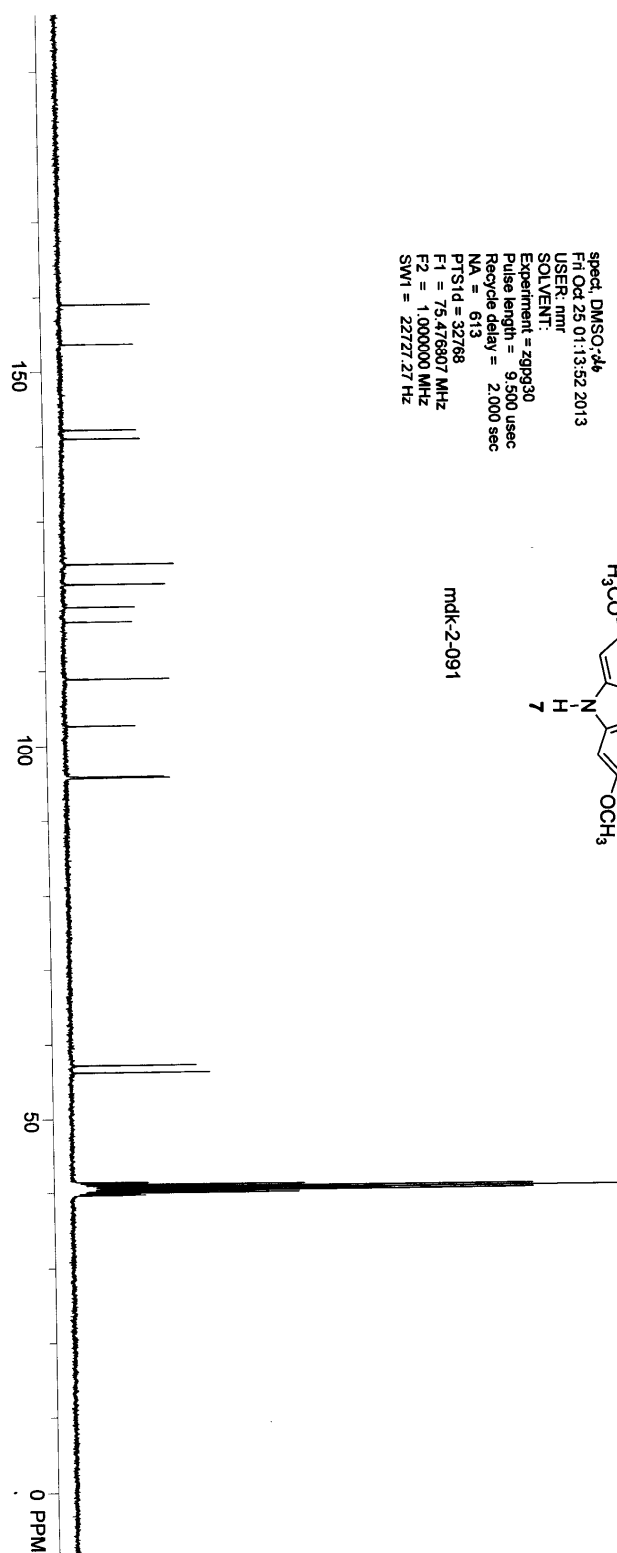
57.168
56.195

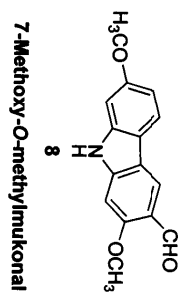
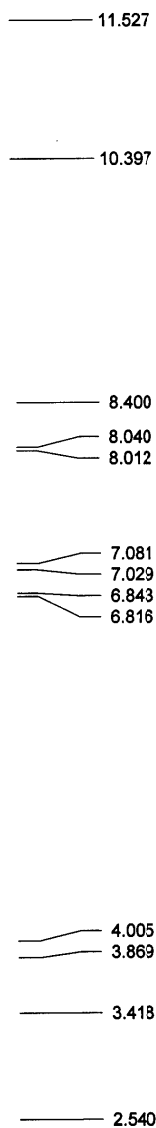
41.286
41.007
40.721
40.450
40.174
39.894
39.612



speed, DMSO-d₆
F1 Qd 25 01:13:52 2013
USER: nmf
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 613
PTSD = 32768
F1 = 75.47807 MHz
F2 = 1.000000 MHz
SWH = 22727.27 Hz

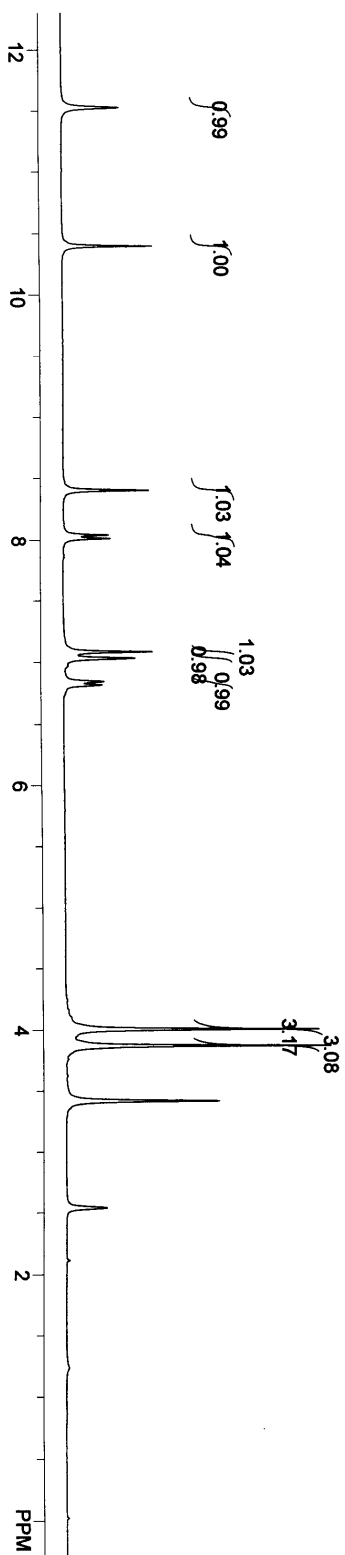
mdk-2-091





spect, DMSO-d₆
 Fri Jan 25 11:46:50 2013
 USER: nmf
 SOLVENT:
 Experiment: = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 P2 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

mdk-1-149



189.031

161.241
159.396

146.501
142.838

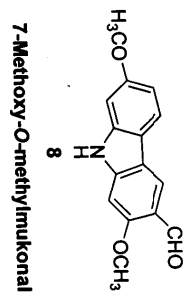
121.848
120.618
118.606
117.768
117.360
109.435

96.227
94.039

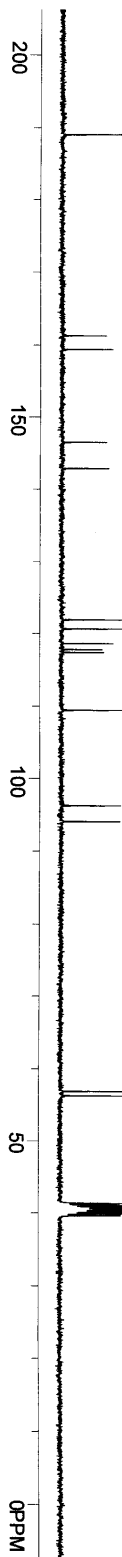
56.675
56.264

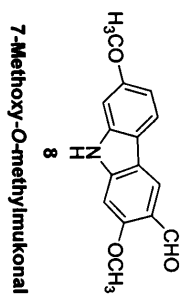
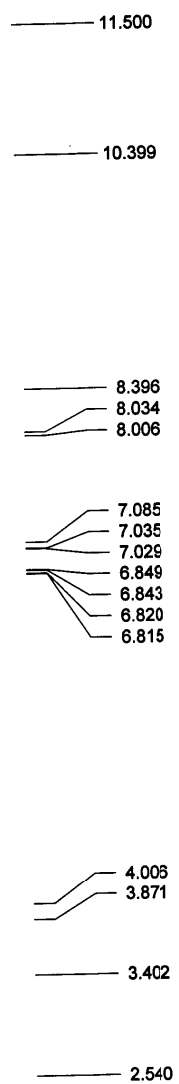
41.283
40.995
40.723
40.450
40.169
39.683
39.613

spec, DMSO- d_6
Fri Jan 25 12:22:03 2013
USER: nmr
SOLVENT:
Experiment = zpg30
Pulse length = 9.500 μ sec
Recycle delay = 2.000 sec
NA = 384
PTSID = 32768
F1 = 75.476907 MHz
F2 = 100.0000 MHz
SWH = 22727.27 Hz



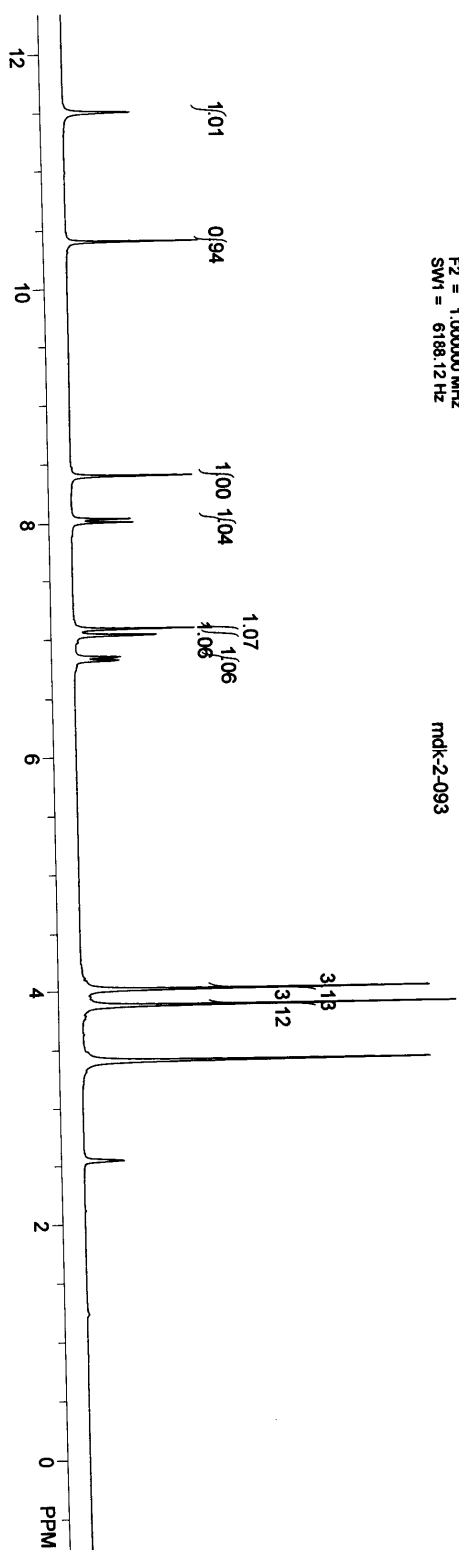
mdk-1-149





spect, DMSO-d₆
 Sun Jul 07 13:43:06 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PRSTD = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SWH = 6188.12 Hz

mdk-2-093



188.974

161.198
159.377

146.461
142.803

121.742
120.559
118.628
117.750
117.342
109.369

96.260
94.032

56.851
56.238

41.292
41.011
40.732
40.450
40.180
39.898
39.611

spect. DMSO-*d*₆

Sun Jul 07 13:44:46 2013

USER: nmr

SOLVENT:

Experiment = zgpg30

Pulse length = 9.500 usec

Recycle delay = 2.000 sec

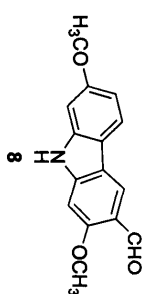
NA = 10240

PTSD = 32768

F1 = 75.476807 MHz

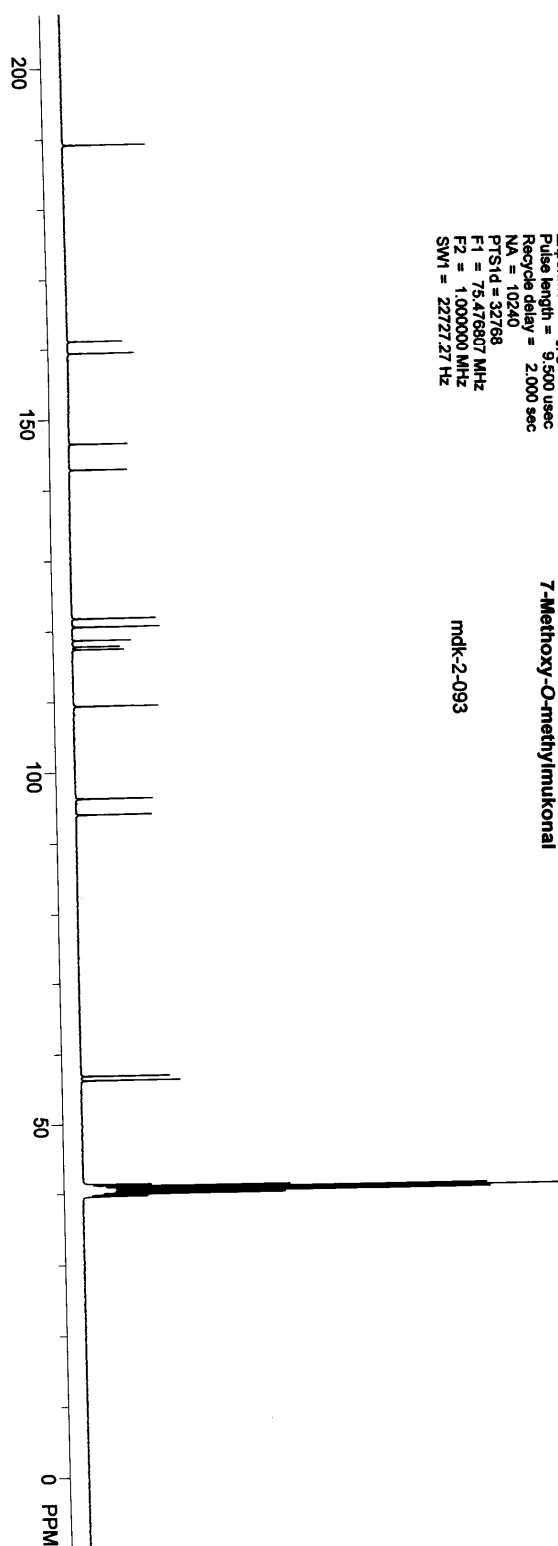
F2 = 1.000000 MHz

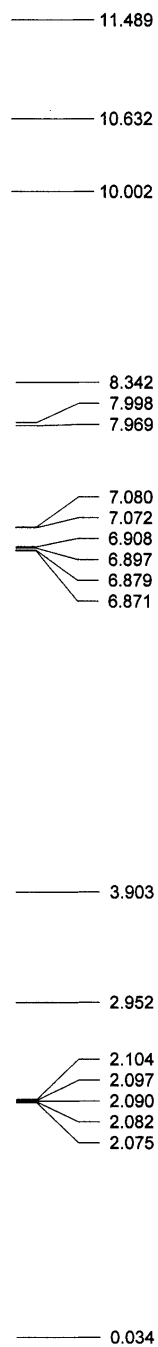
SWH = 22727.27 Hz



7-Methoxy-O-methylmukonal

mdk-2-093

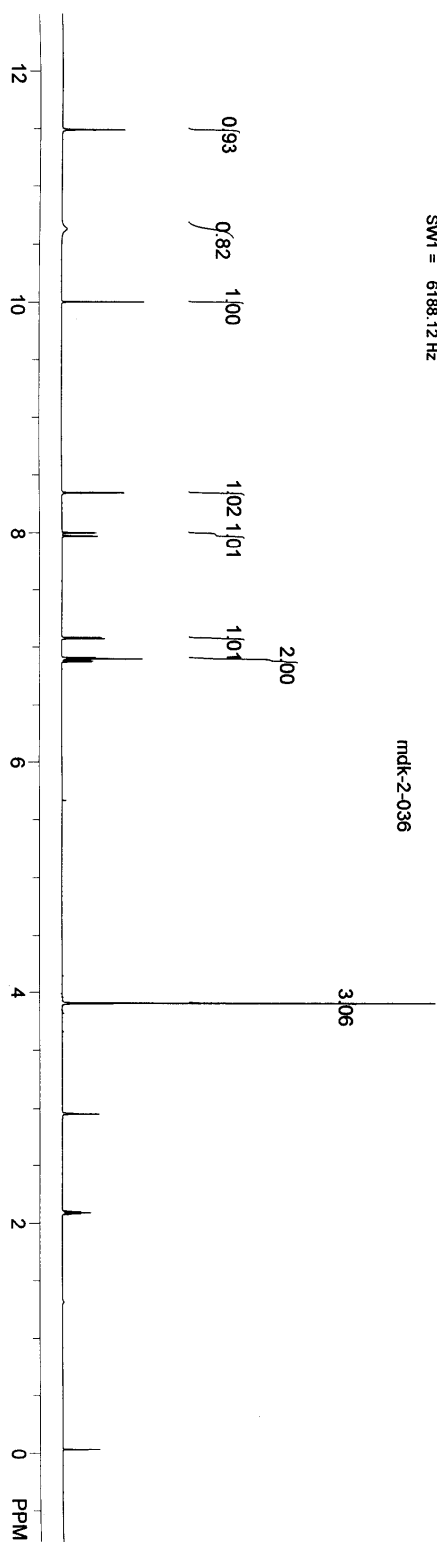


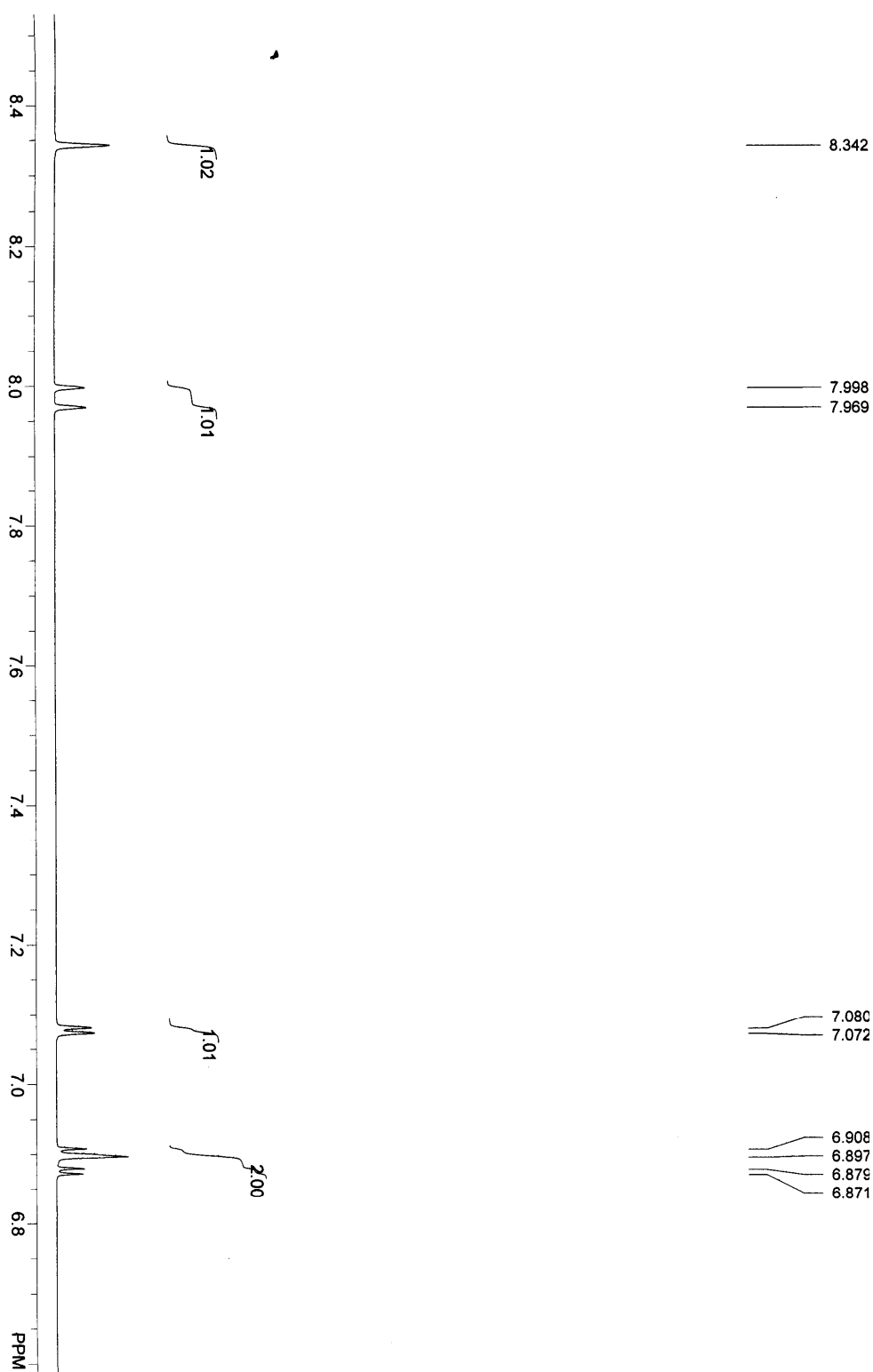


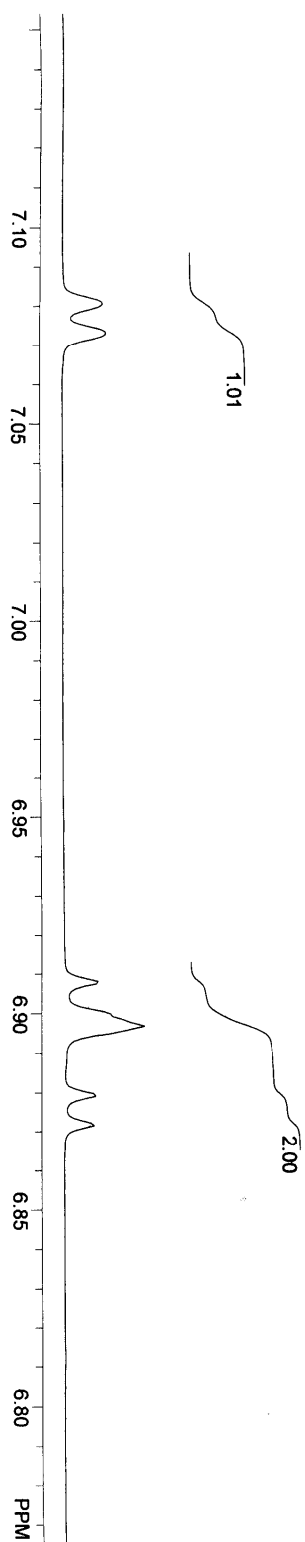
Spec: Acetone,
Fri Apr 18 07:04:24 2014
USER: nmf
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTSD = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SWH = 6188.12 Hz

7-Methoxymukonal

mdk-2-036

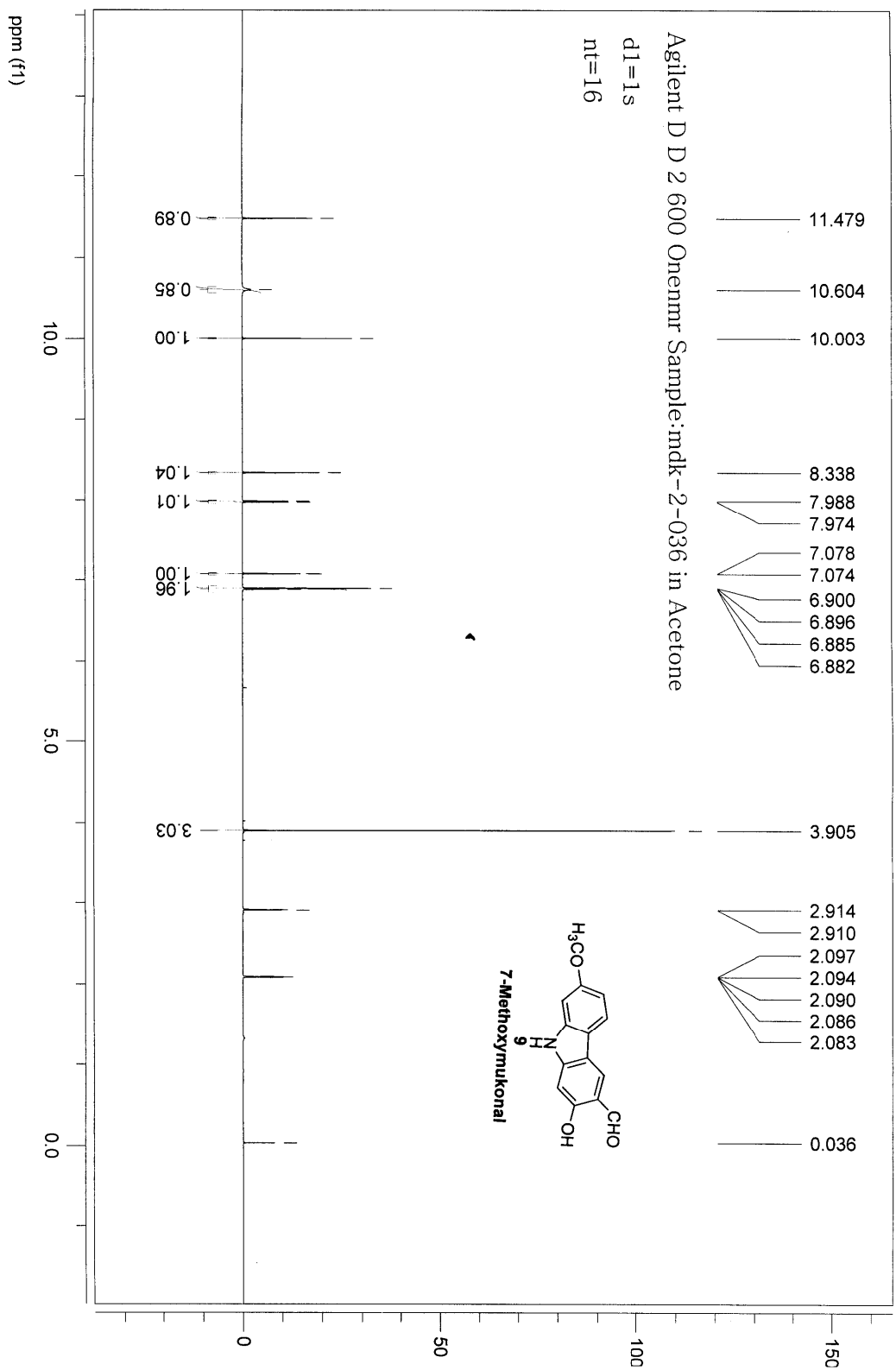


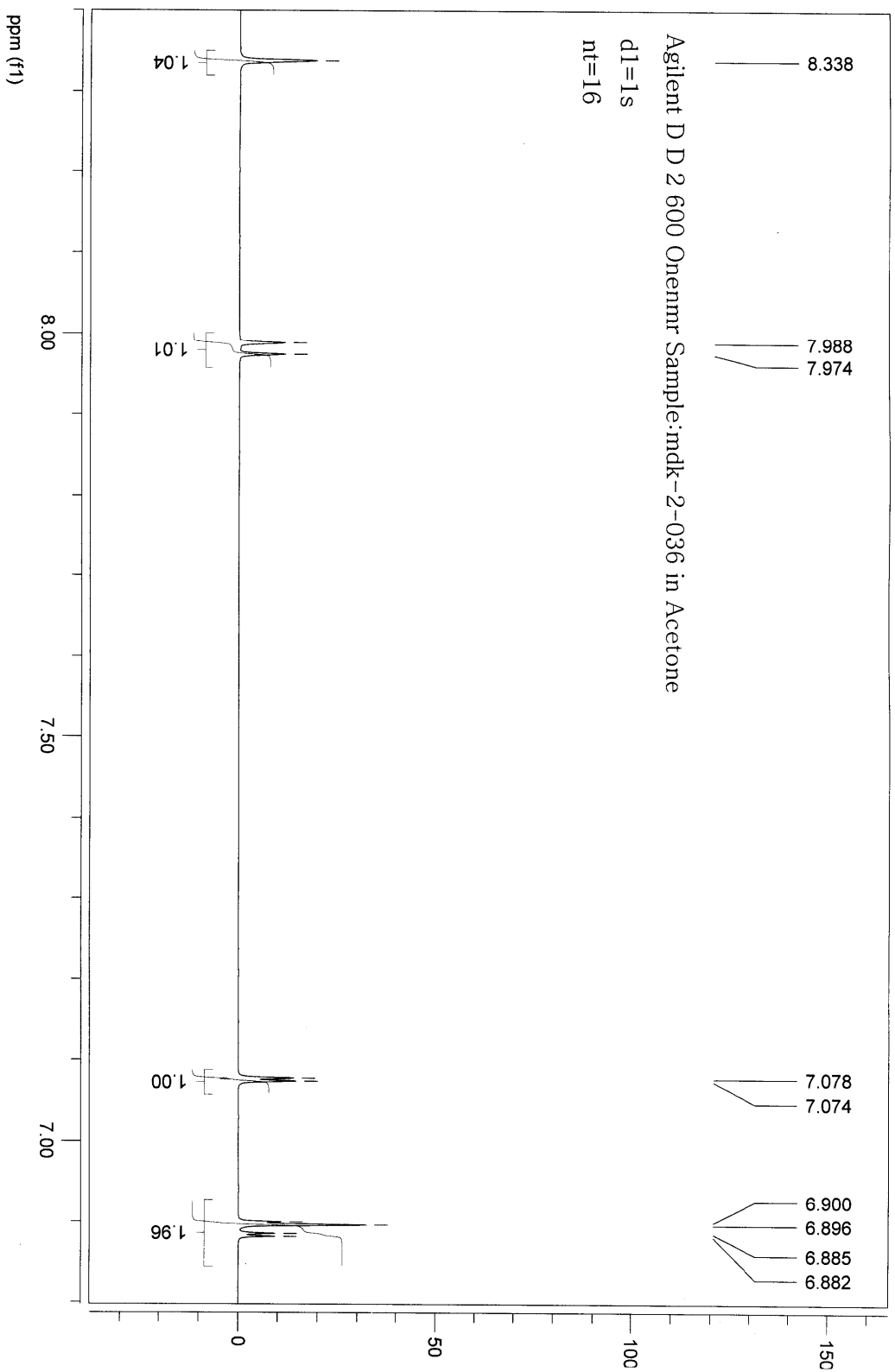




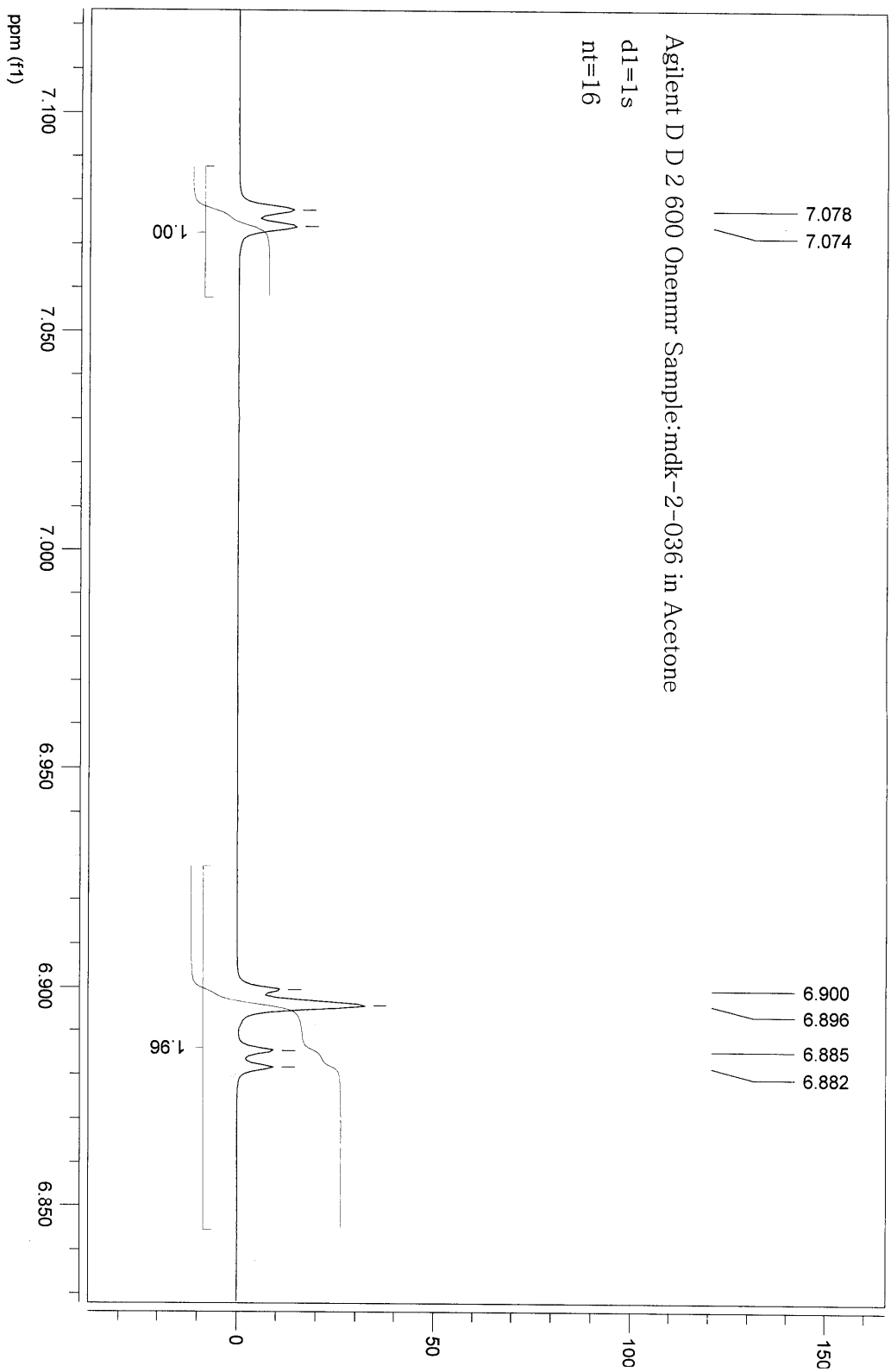
7.080
7.072

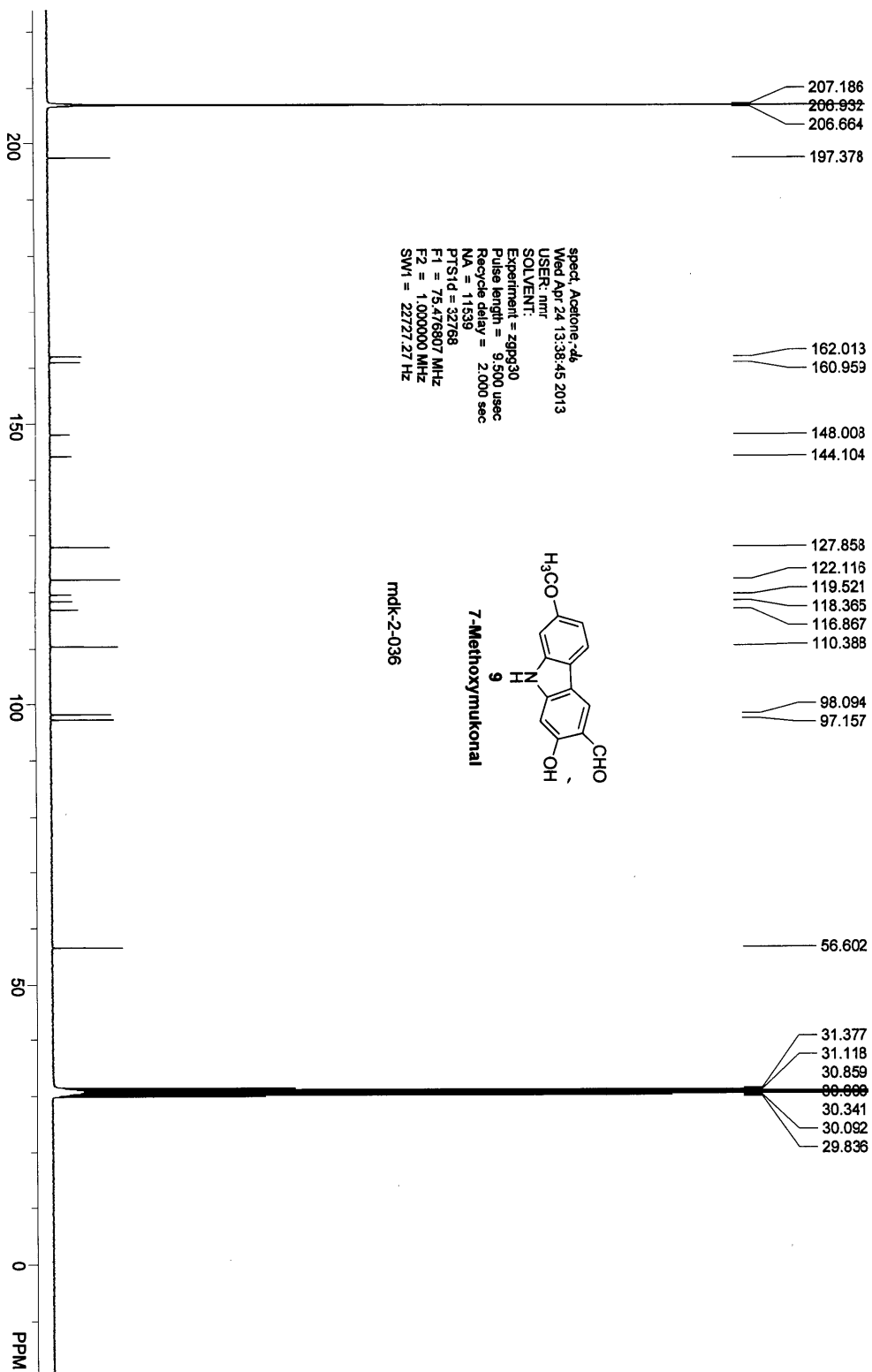
6.908
6.897
6.879
6.871





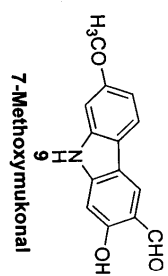
Agilent D D 2 600 Onenmr Sample:mdk-2-036 in Acetone
d1=1s
nt=16



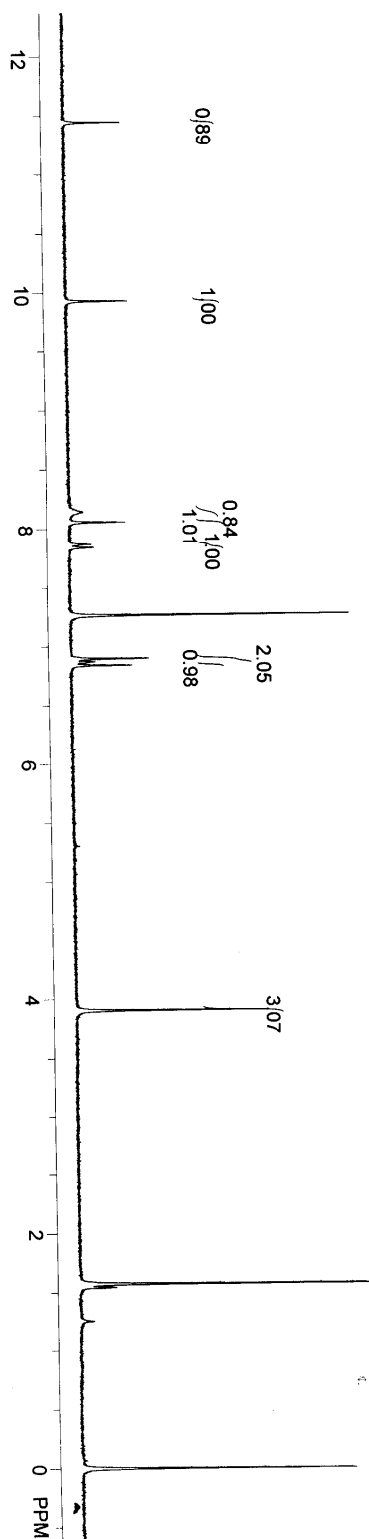


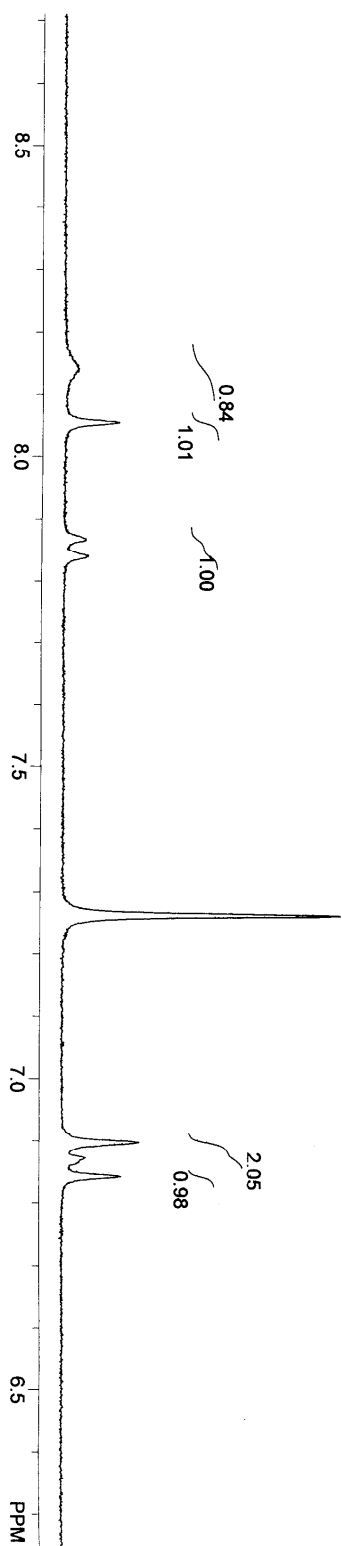


spect, CDCl₃,
 Fri Apr 18 06:37:43 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 P1 = 32.768
 P2 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz



mdk-2-036-CDCl₃





8.139

8.055

7.867

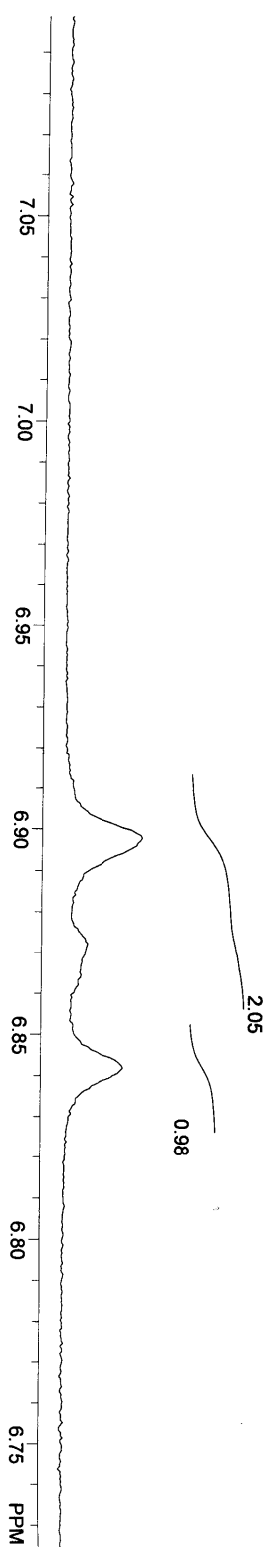
7.840

7.263

6.898

6.872

6.842



6.898

6.872

6.842

2-036-CDCl3



Current Data Parameters
NAME 2-036-CDCl3
EXPNO 1
PROCNO 1

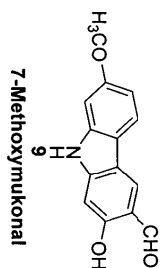
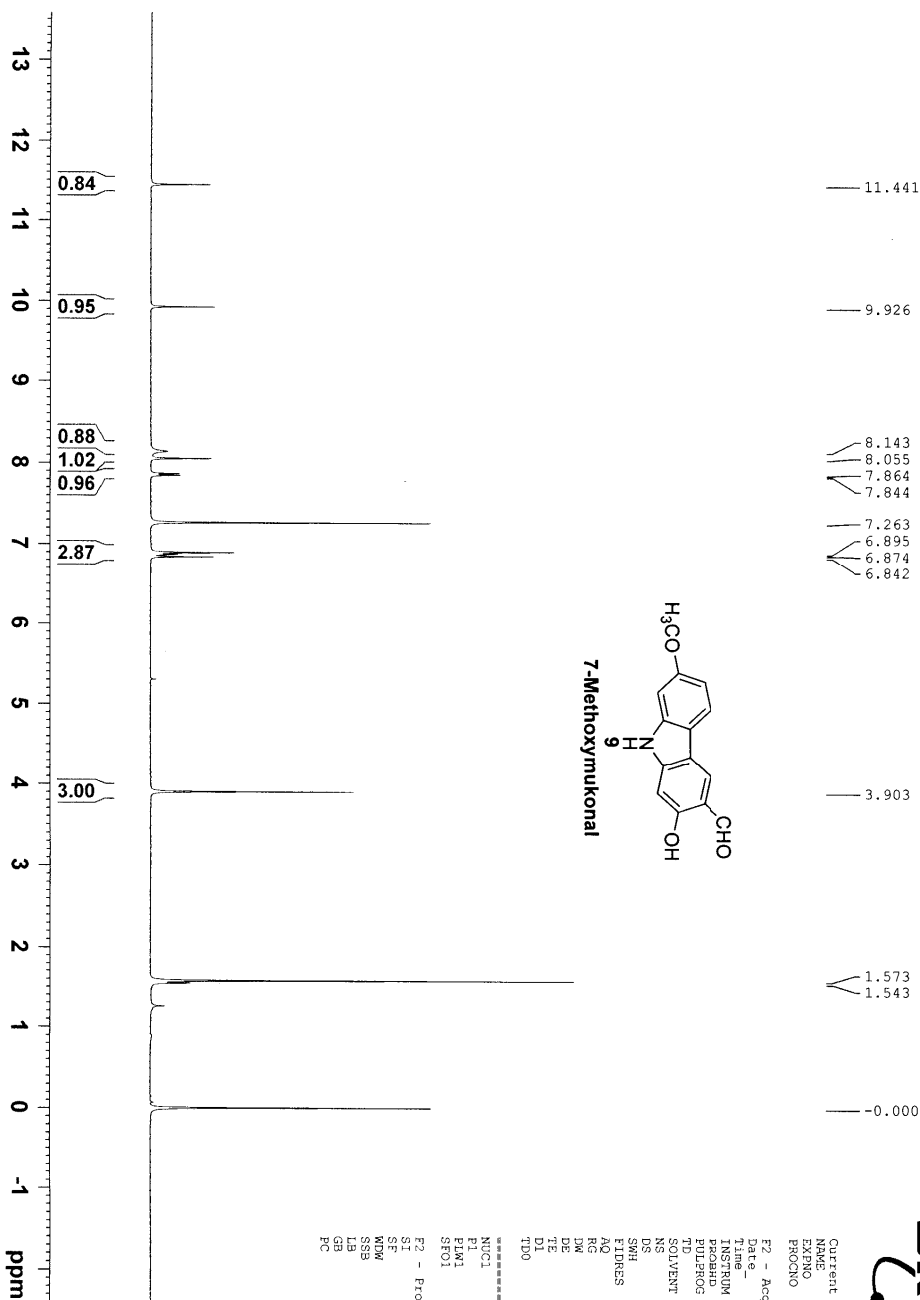
F2 - Acquisition Parameters

Date_ 20140418
Time 13.12
INSTRUM spect
PROBHD 5 mm zgr30
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 1
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 193.34
RG 50.000 usec
RM 0.050 usec
TE 293.3 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 12.5000000 W
SFO1 400.1328009 MHz

F2 - Processing parameters

SI 32768
SF 400.130008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



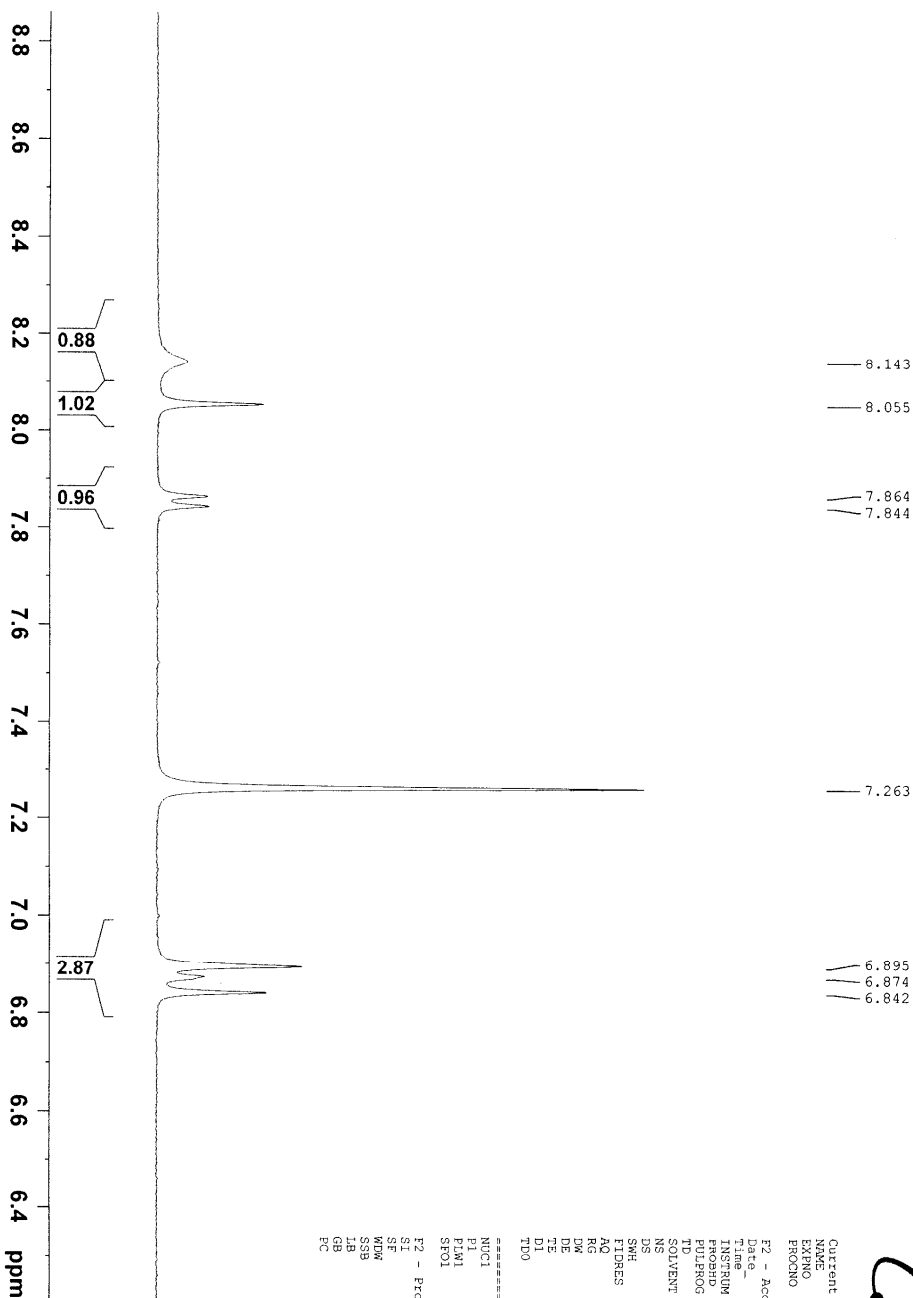
2-036-CDCl3

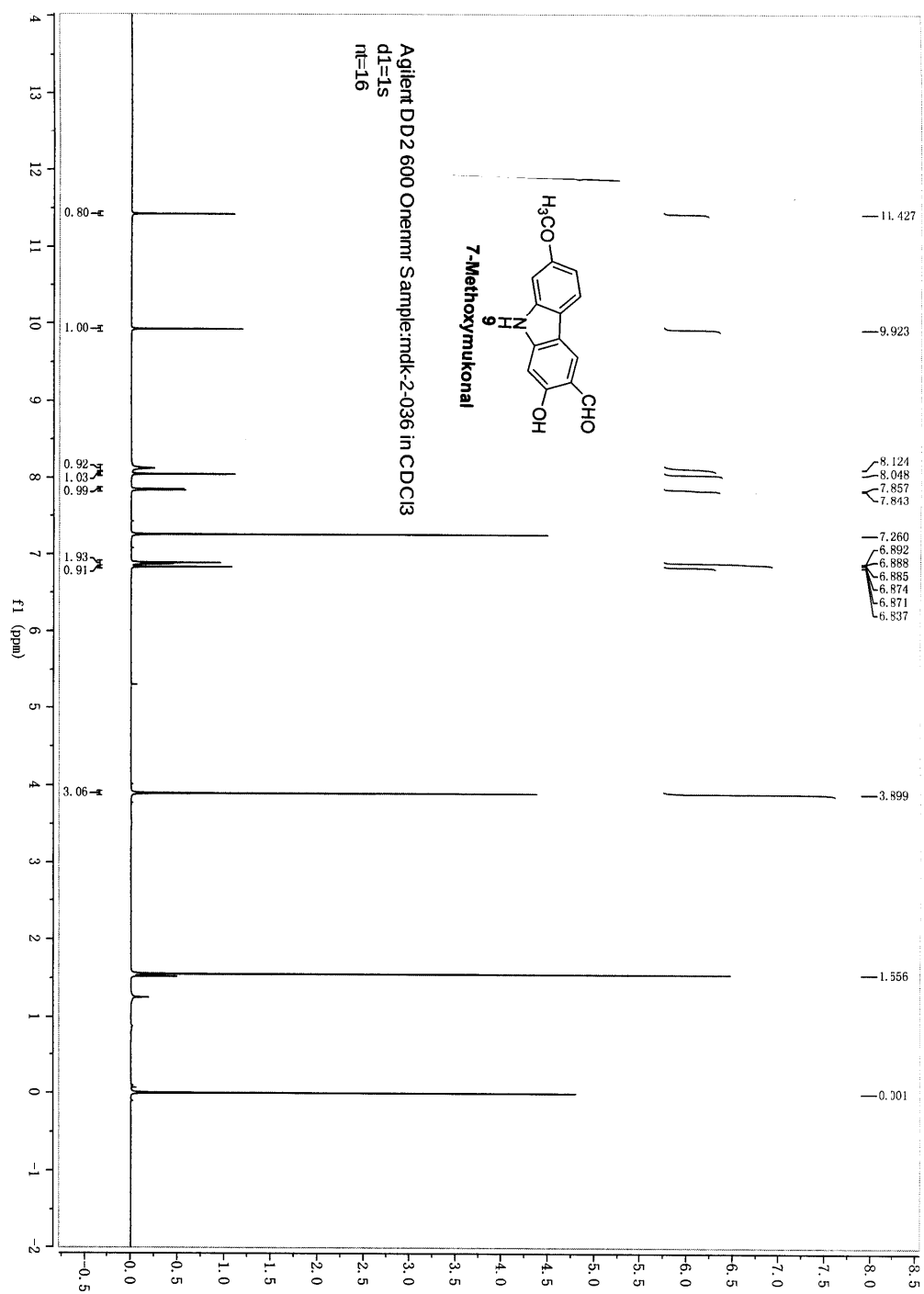


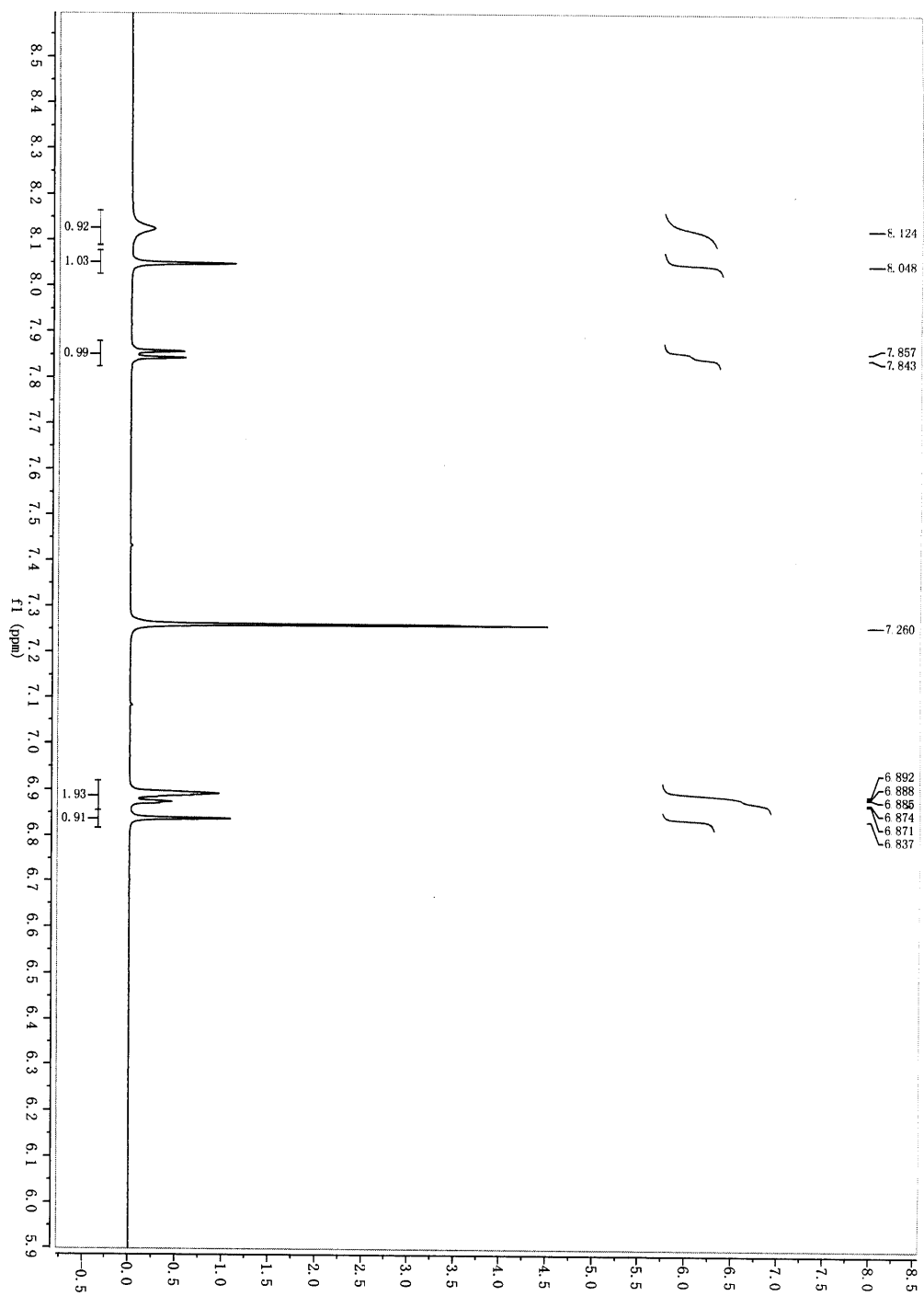
Current Data Parameters
NAME 2-036-CDCl3
EXPNO 1
PROCNO 1

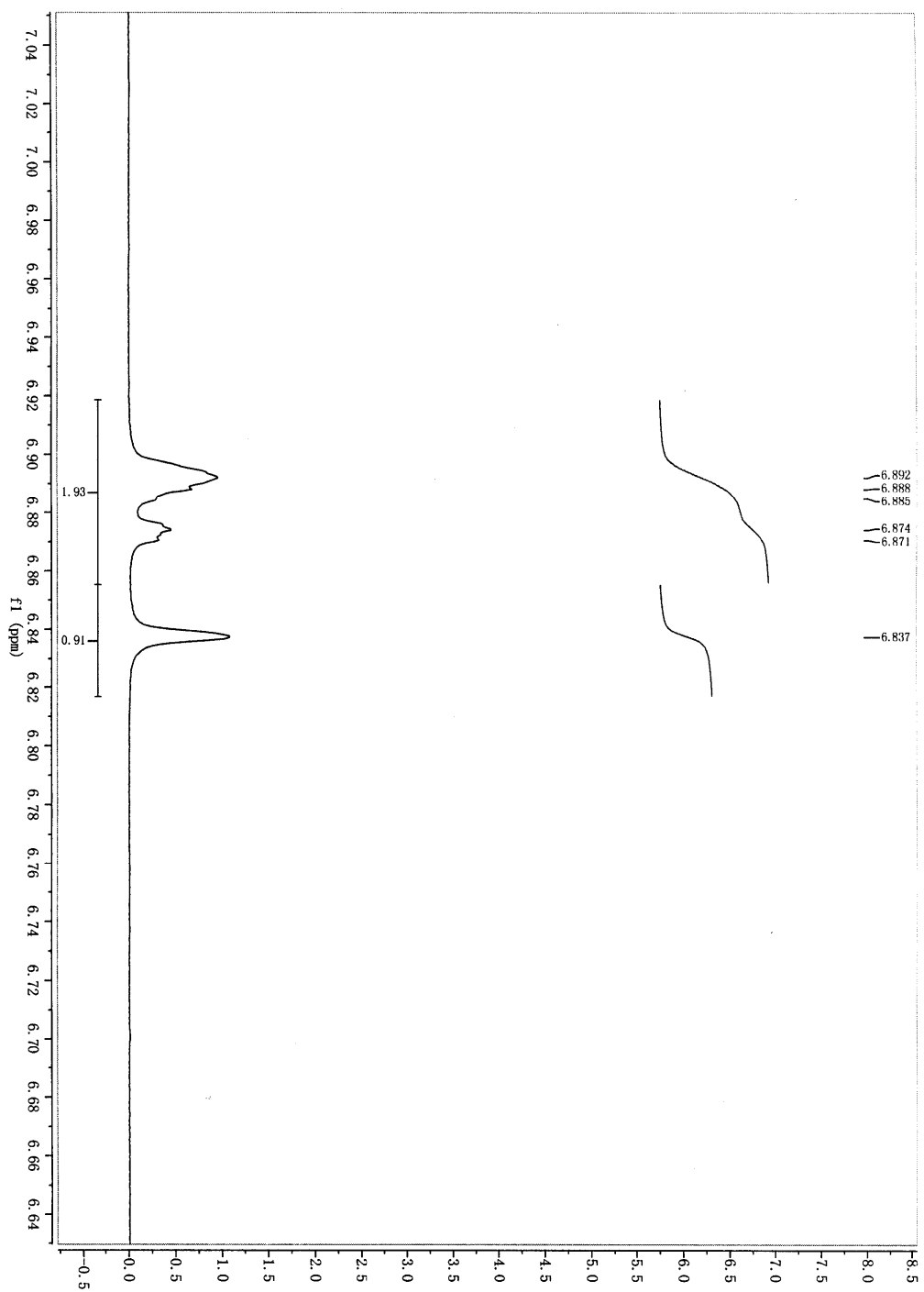
F2 - Acquisition Parameters
Date_ 20140418
Time 15.15
INSTRUM spect
PROBHD 5 mm PABBO
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SSB 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 193.34
DW 50.000 usec
DE 6.50 usec
TE 293.2 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.00 usec
PL1 12.5000000 W
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.130008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

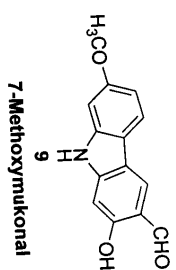
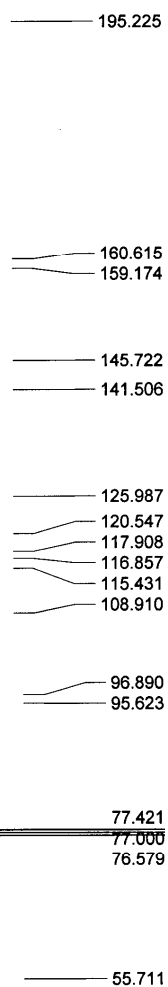




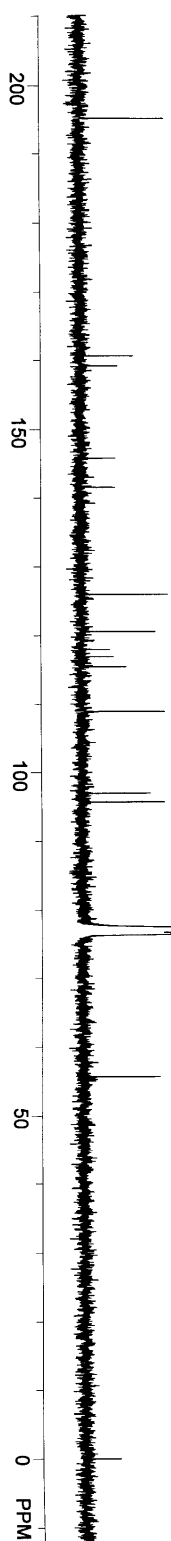


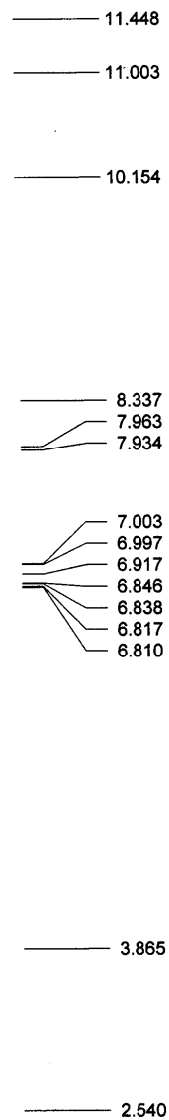


spect, CDCl3
Mon May 06 13:41:59 2013
USER: nmf
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10989
PTSD = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SWH = 22727.27 Hz

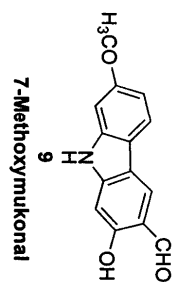


mdk-2-036

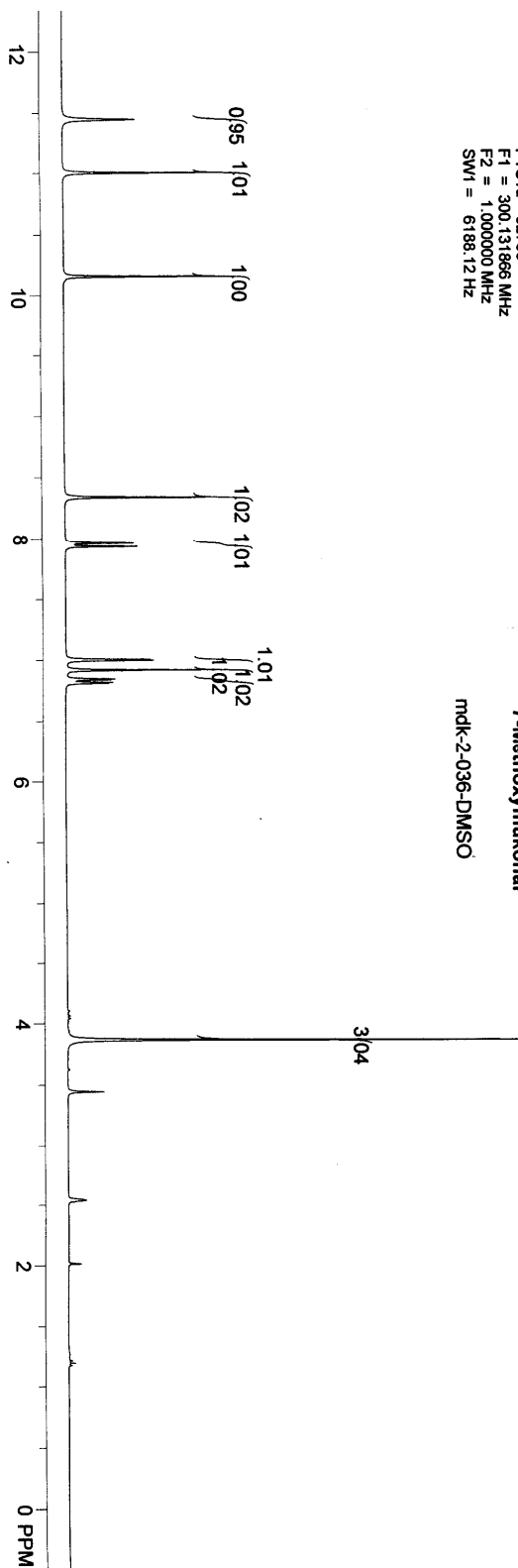




spect. DMSO-d6
 Fri May 30 07:14:24 2014
 USER: nmf
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usac
 Recycle delay = 1,000 sec
 NA = 8
 PTSTd = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SWH = 6188.12 Hz



mdk-2-036-DMSO



193.722

160.265
159.436

146.901
143.016

124.289
121.507
118.028
117.346
116.544
109.231

97.282
96.377

56.253

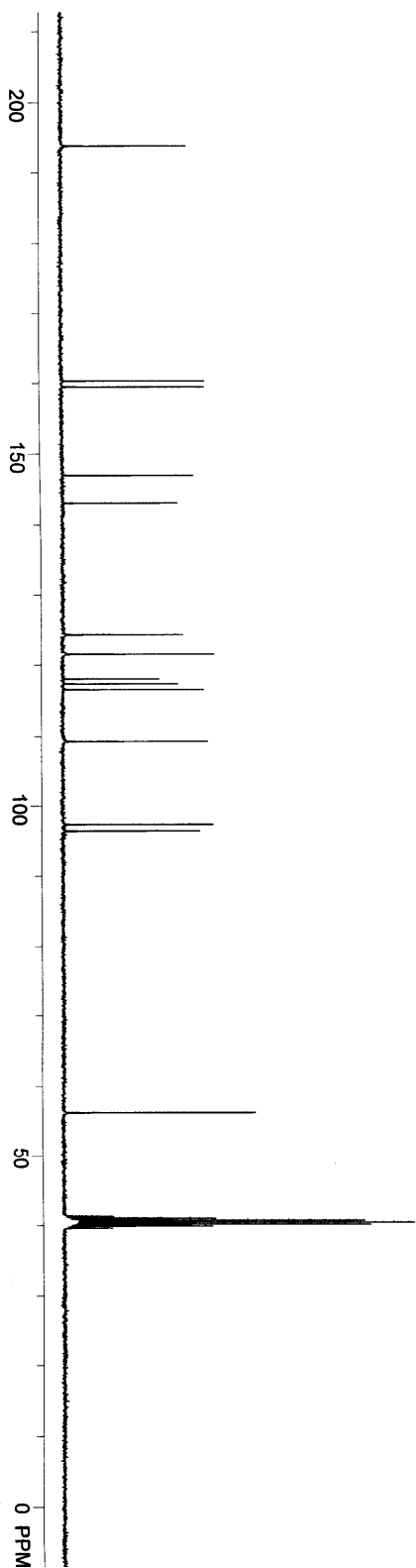
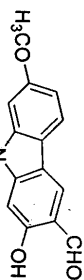
41.294
41.016
40.736
40.450
40.179
39.902
39.623

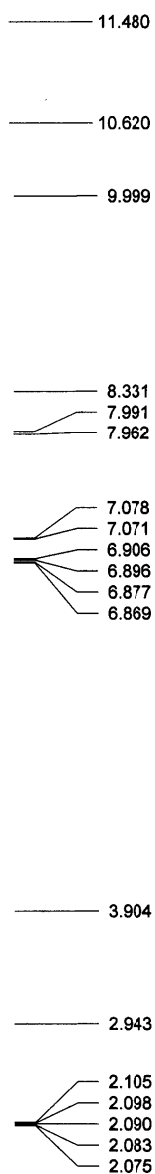
spec: DMSO-d6
Fri May 30 07:16:09 2014
USER: mtr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 376
PTSD = 32768
F1 = 75.476807 MHz
F2 = 100.00000 MHz
SWH = 22727.27 Hz

mdk-2-036-DMSO

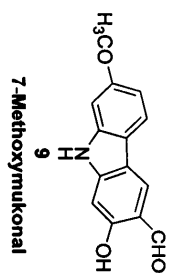
7-Methoxymukonal

9

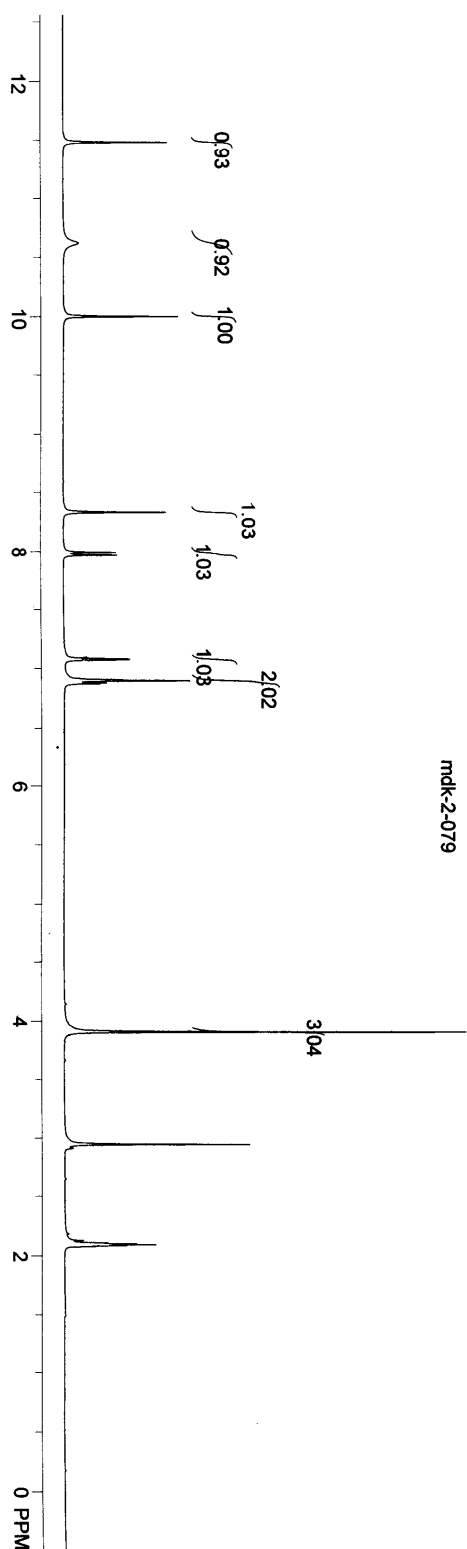


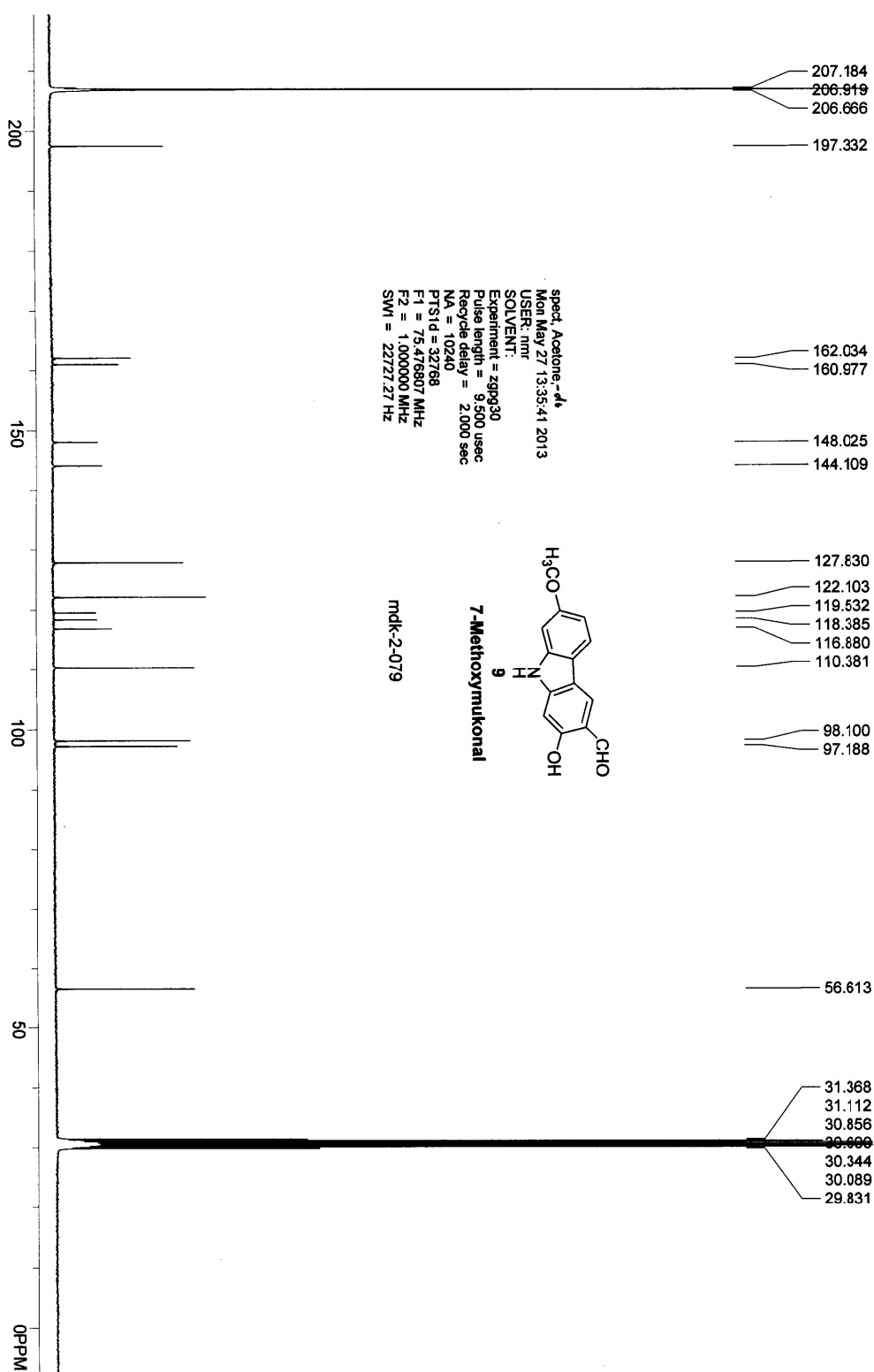


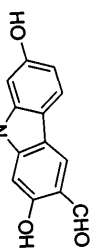
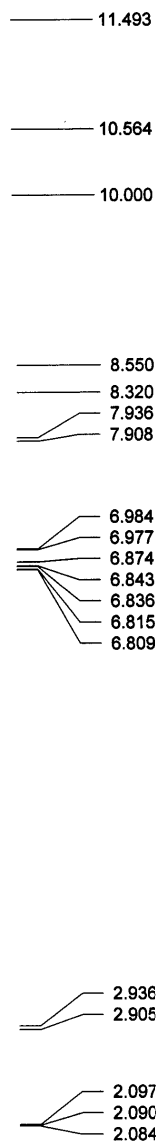
Spect, Acetone-d₆
 Sun May 26 12:25:44 2013
 USER: nmr
 SOLVENT:
 Experiment: z930
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32.768
 P2 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz



mdk-2-079



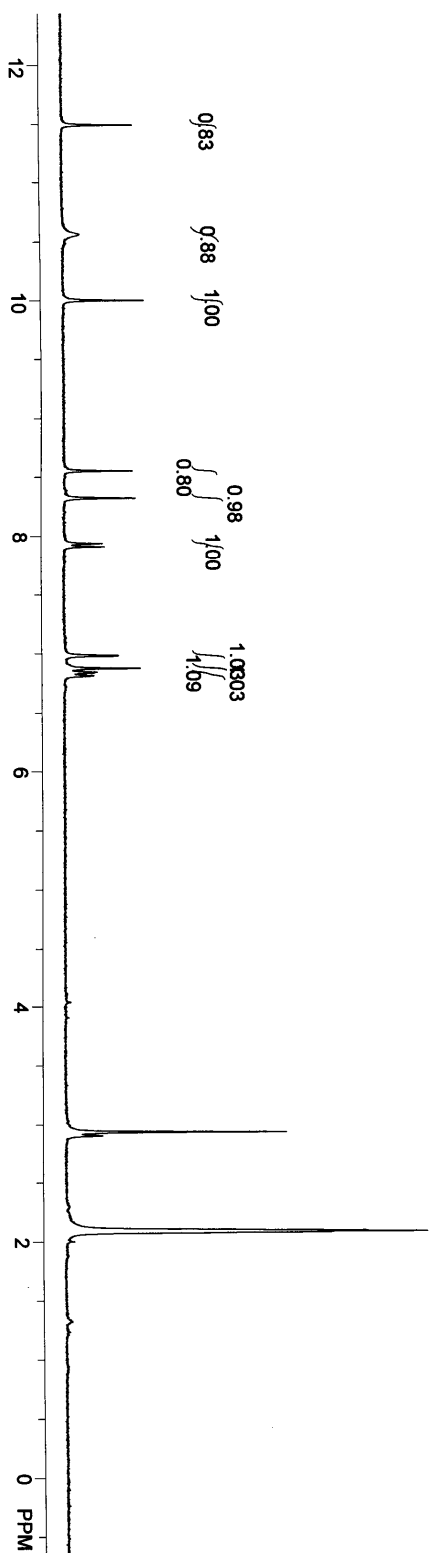


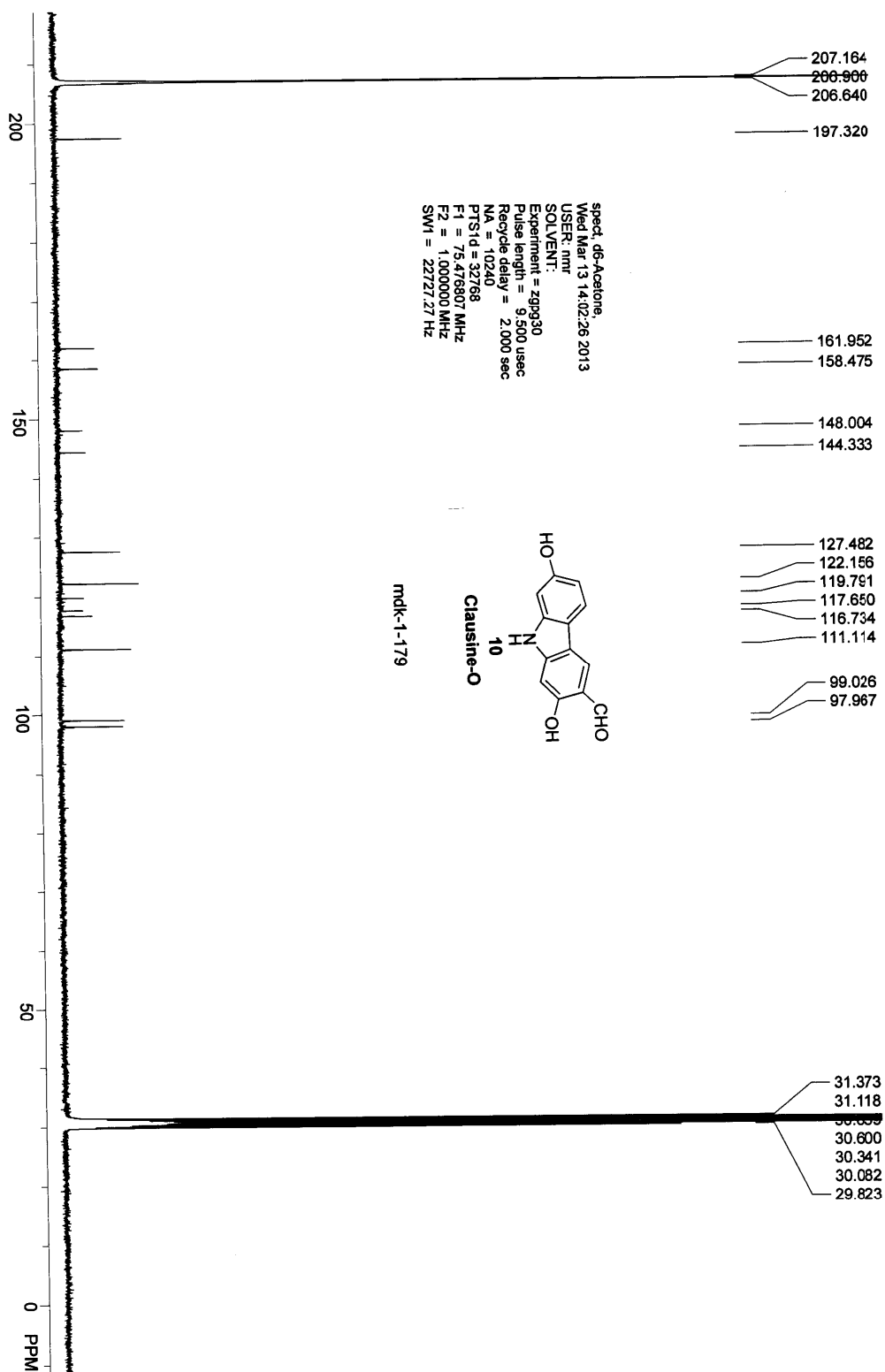


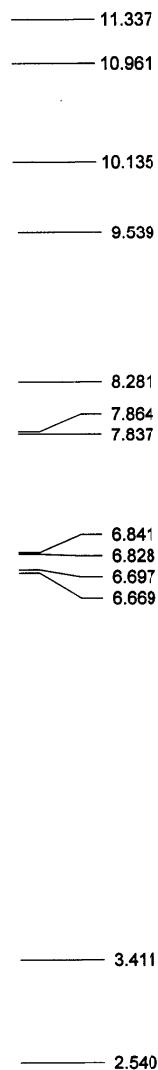
Clausine-O

mdk-1-179

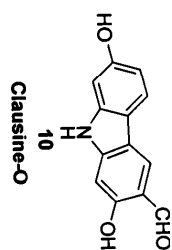
spect: Acetone-d₆
 Tue Mar 12 02:52:58 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 P2 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz



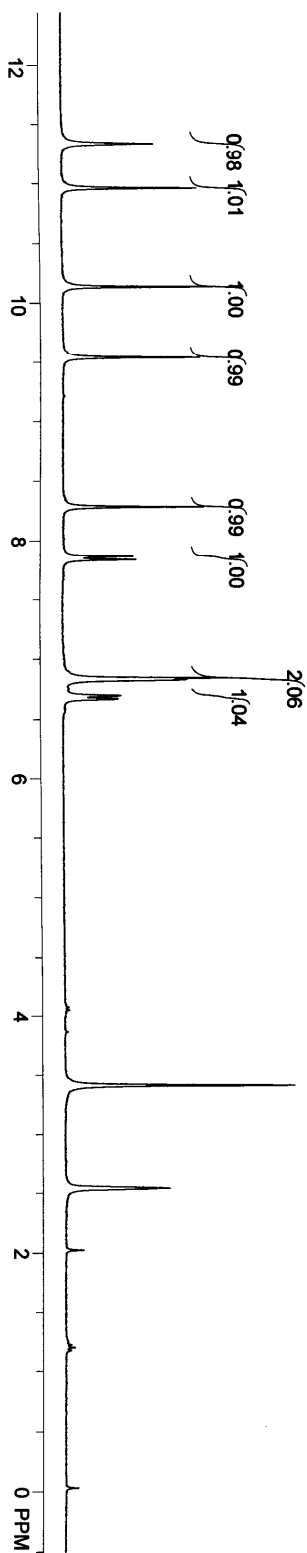


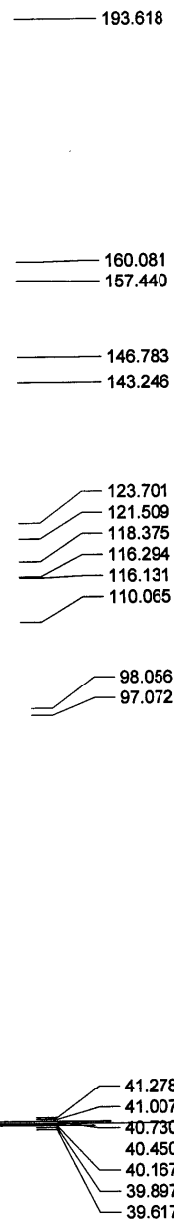


spect, DMSO-d₆
 Tue Mar 26 13:35:29 2013
 USER: nmf
 SOLVENT:
 Experiment = z930
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1STD = 32.768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

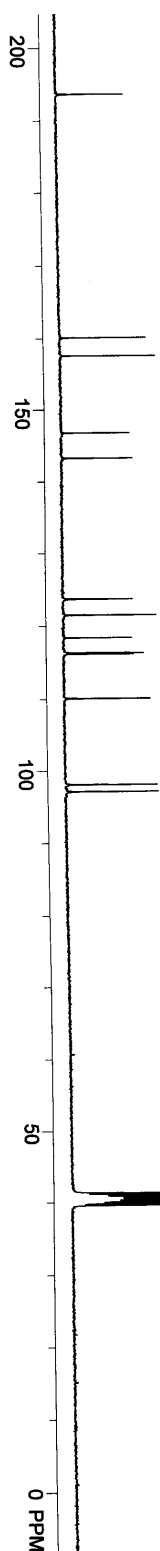
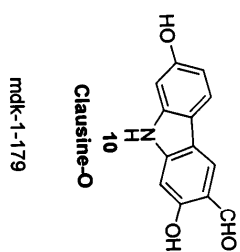


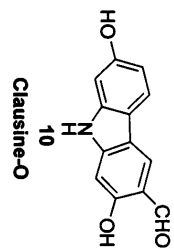
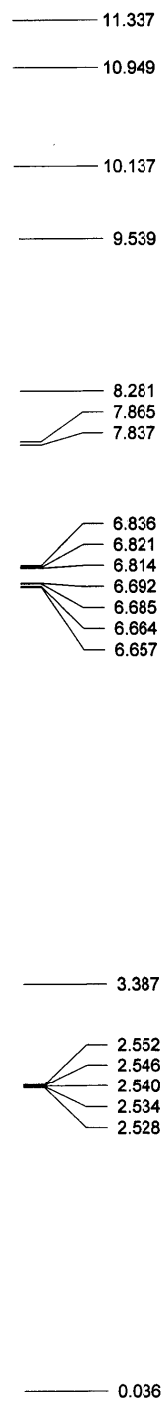
mdk-1-179





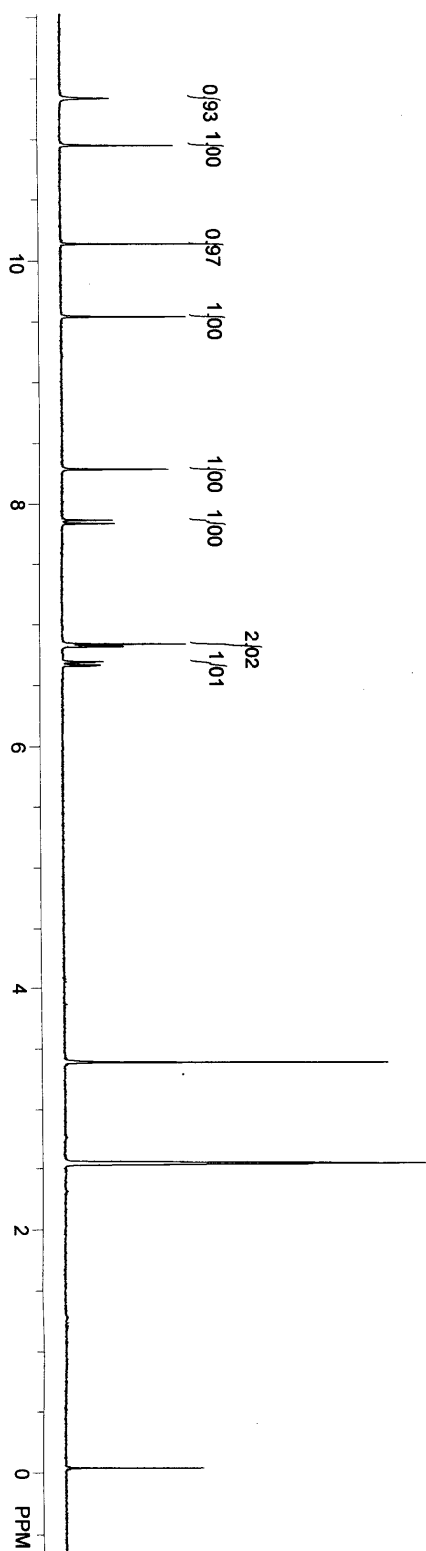
specd, d6-DMSO,
Tue Mar 26 13:49:38 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10240
PTSD = 32768
F1 = 75.476807 MHz
F2 = 100.626000 MHz
SWH = 22727.27 Hz





Spec: DMSO-^d₆
Wed Feb 26 00:33:01 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTSD = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

mdk-2-095



193.706

160.130
157.471

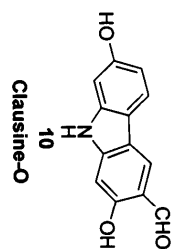
146.819
143.284

123.776
121.535
118.416
116.321
116.163
110.105

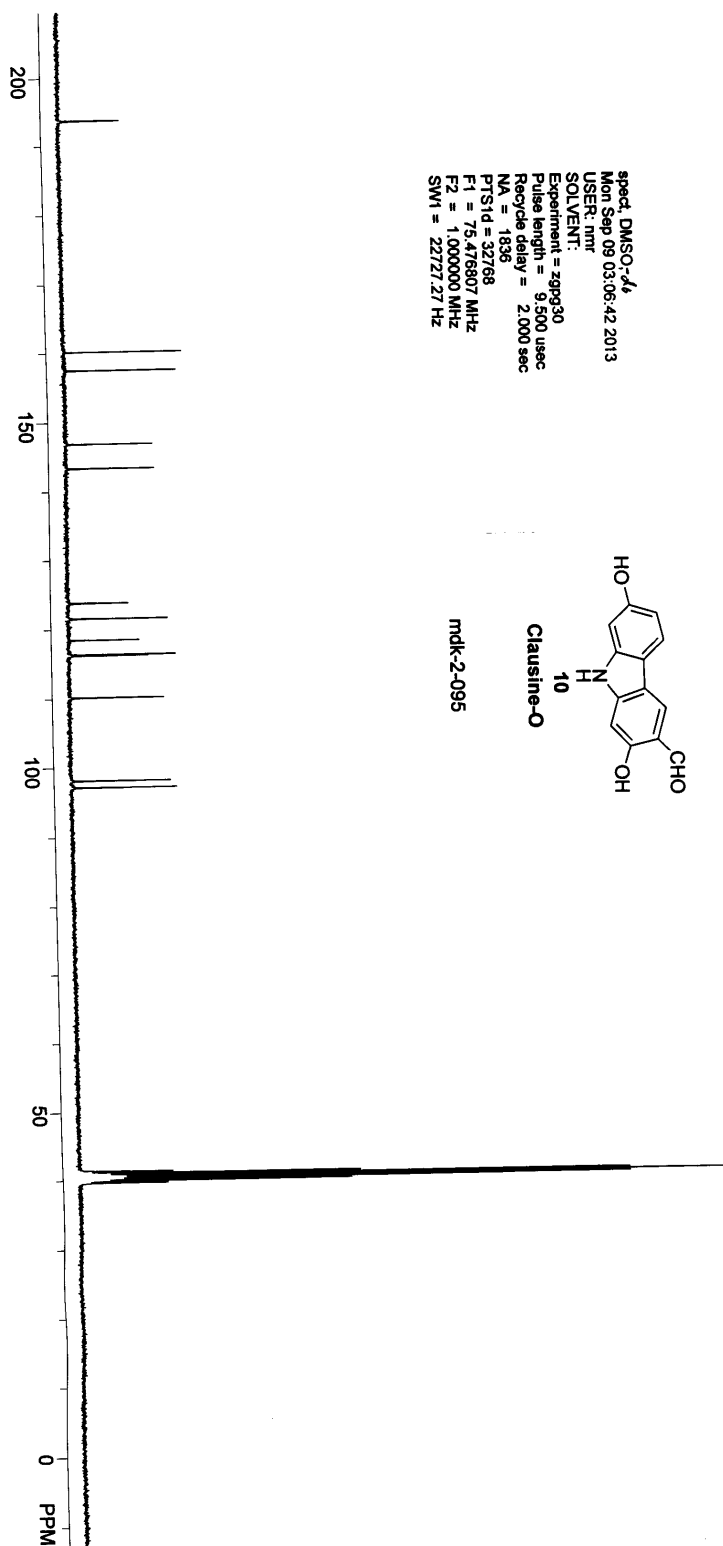
98.093
97.110

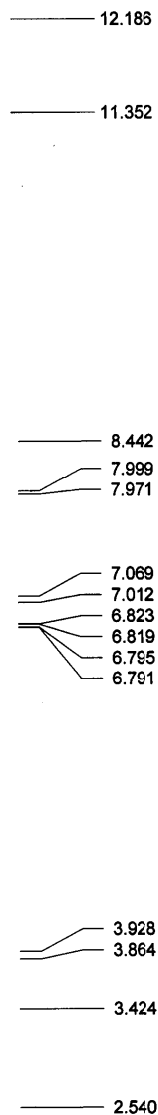
41.288
41.007
40.721
40.450
40.175
39.894
39.609

spec: DMSO- d_6
Mon Sep 09 03:06:42 2013
USER: nmr
SOLVENT:
Experiment = zpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1836
PTStd = 32768
F1 = 73.476807 MHz
F2 = 1,000000 MHz
SWH = 22727.27 Hz

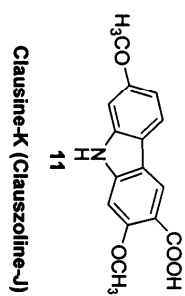


mdk-2-095

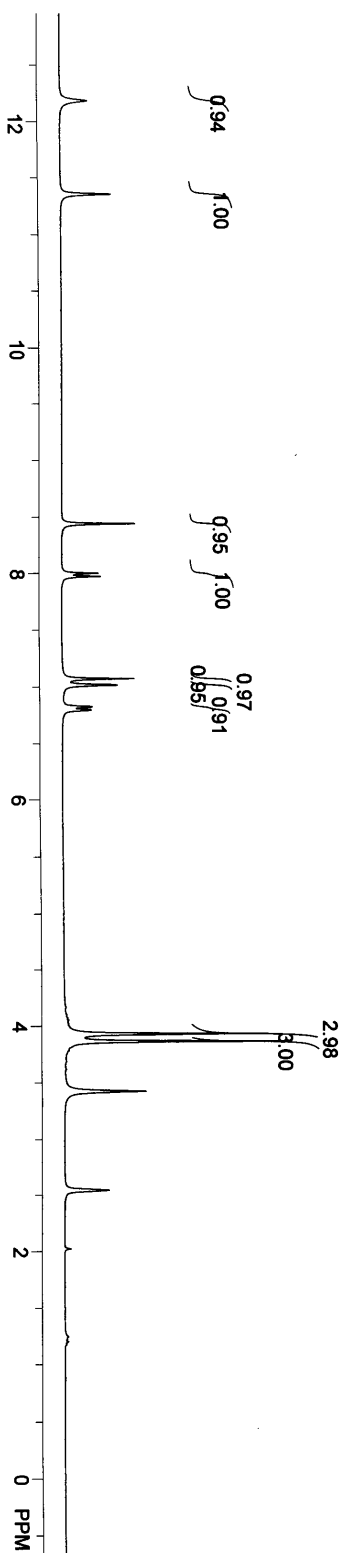


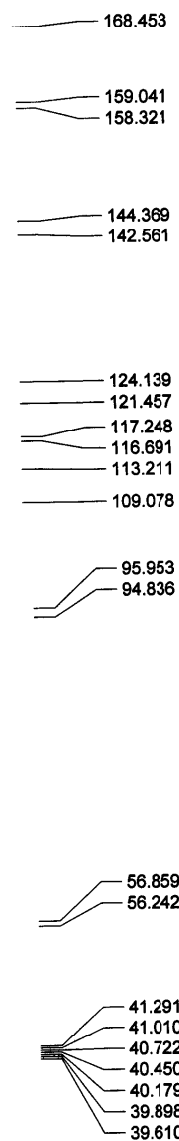


spect: DMSO- d_6
 Sun Mar 10 08:37:30 2013
 USER: mnr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

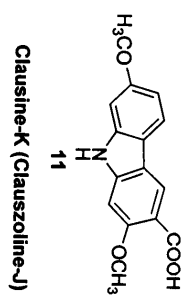


mdk-1-152

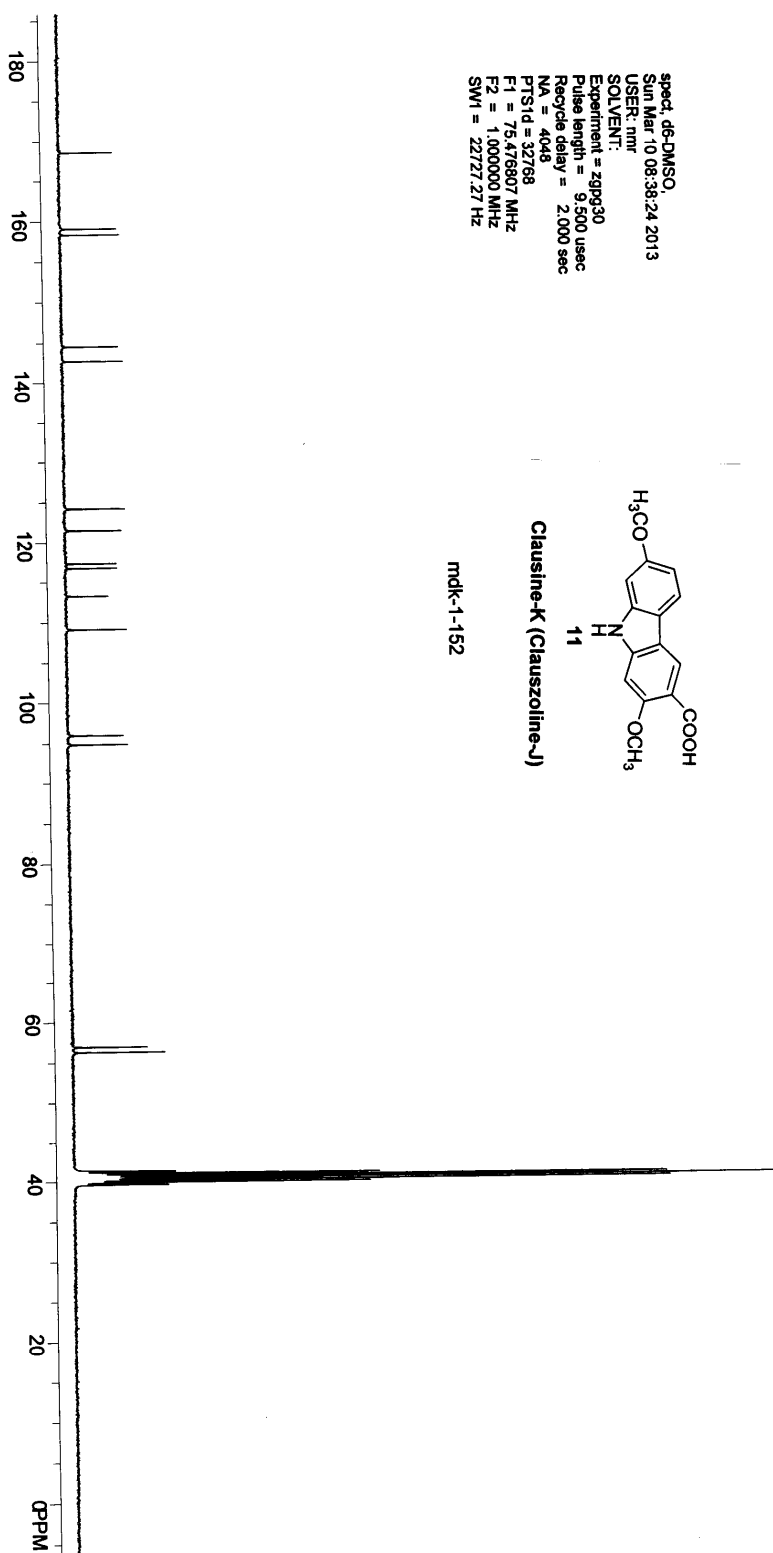


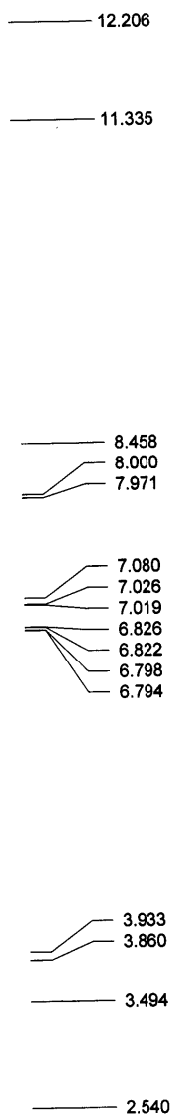


spect, d6-DMSO,
 Sun Mar 10 08:36:24 2013
 USER: nmf
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 4046
 PTStd = 52768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SWH = 22727.27 Hz

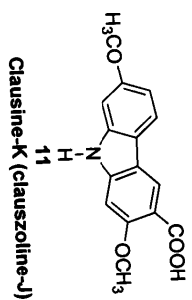


mdk-1-152

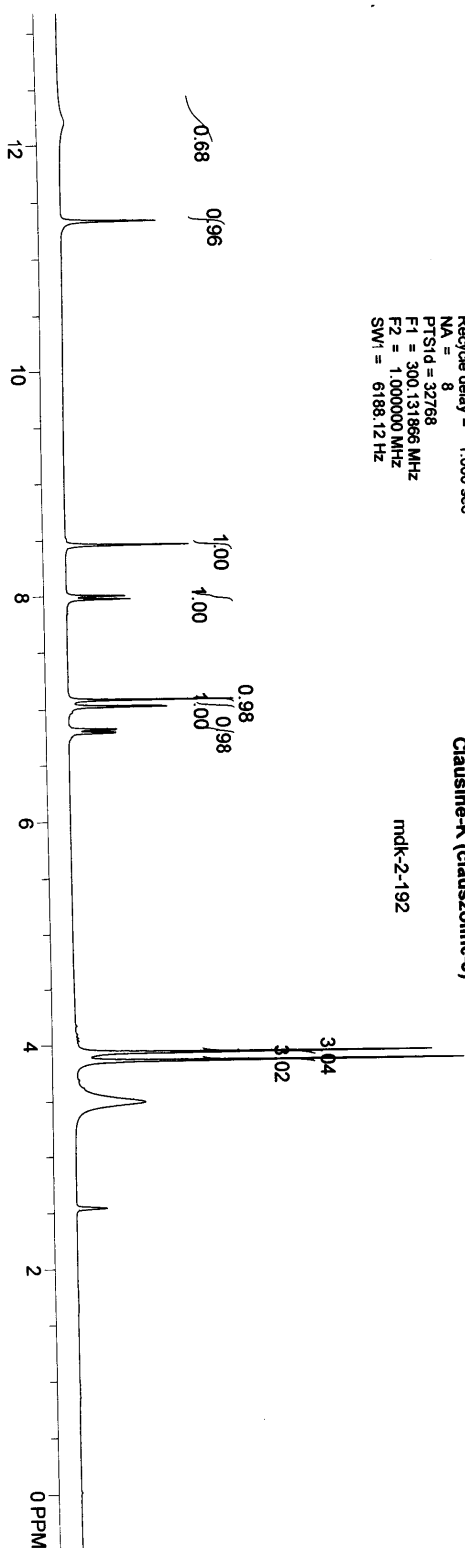


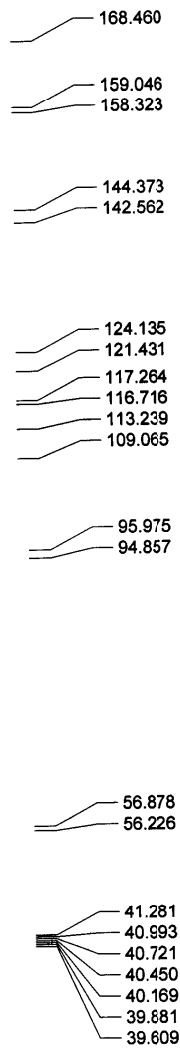


spect, DMSO- d_6
 Wed Jan 22 13:31:42 2014
 USER: nmr
 SOLVENT:
 Experiment = zq30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1SID = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

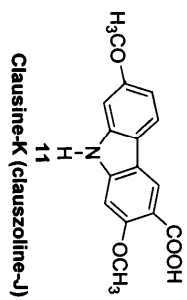


mdk-2-192

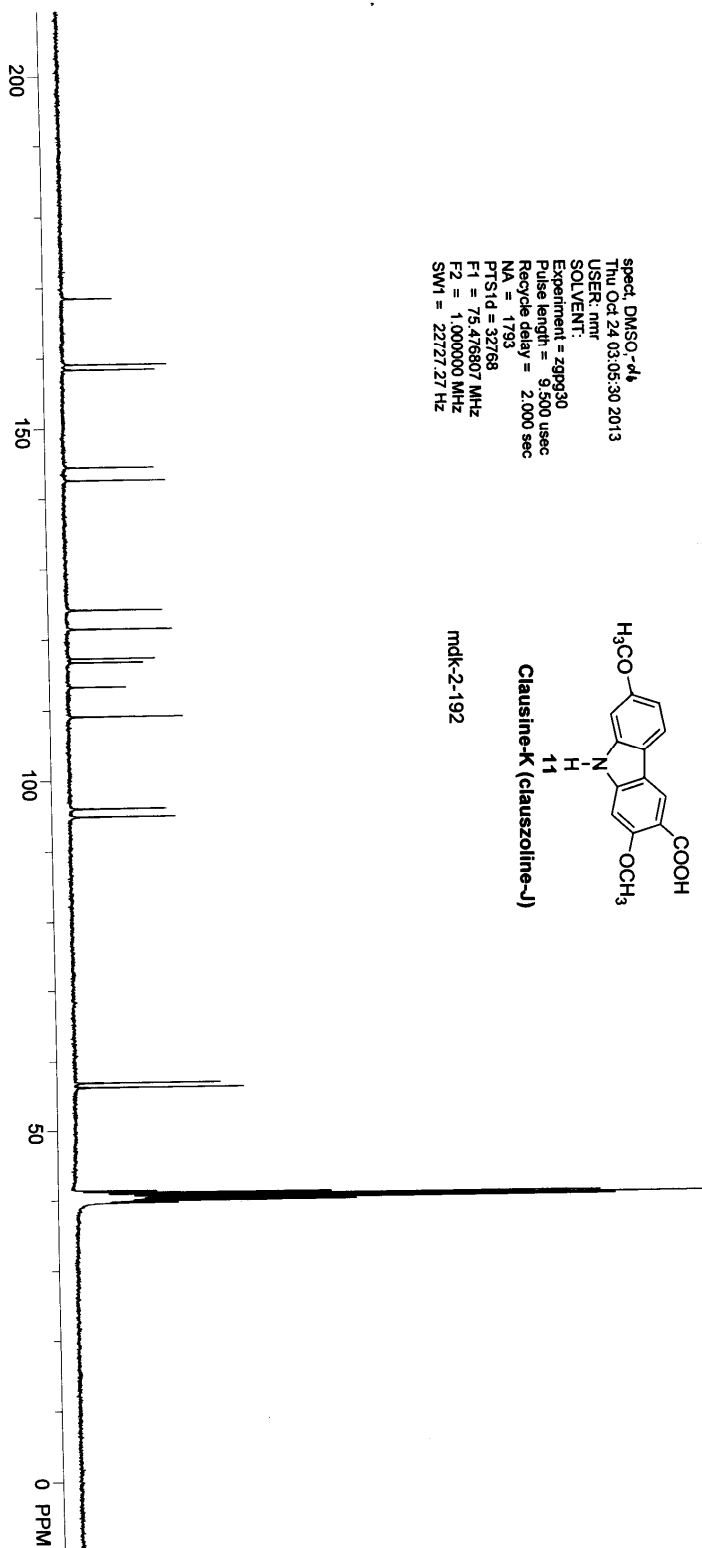


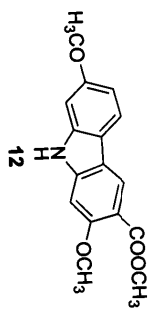
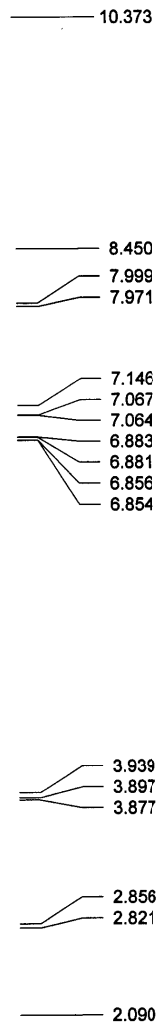


spec: DMSO-*d*₆
Thu Oct 24 03:05:30 2013
USER: mtr
SOLVENT:
Experiment: zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1793
PTSD = 32768
F1 = 75.476907 MHz
F2 = 1.000000 MHz
SWH = 22727.27 Hz



mdk-2-192

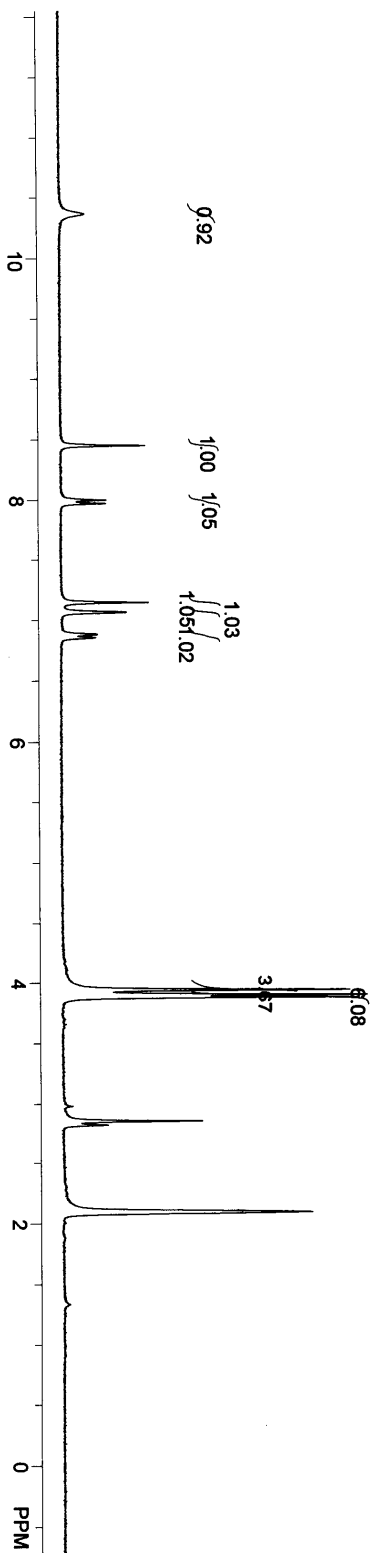


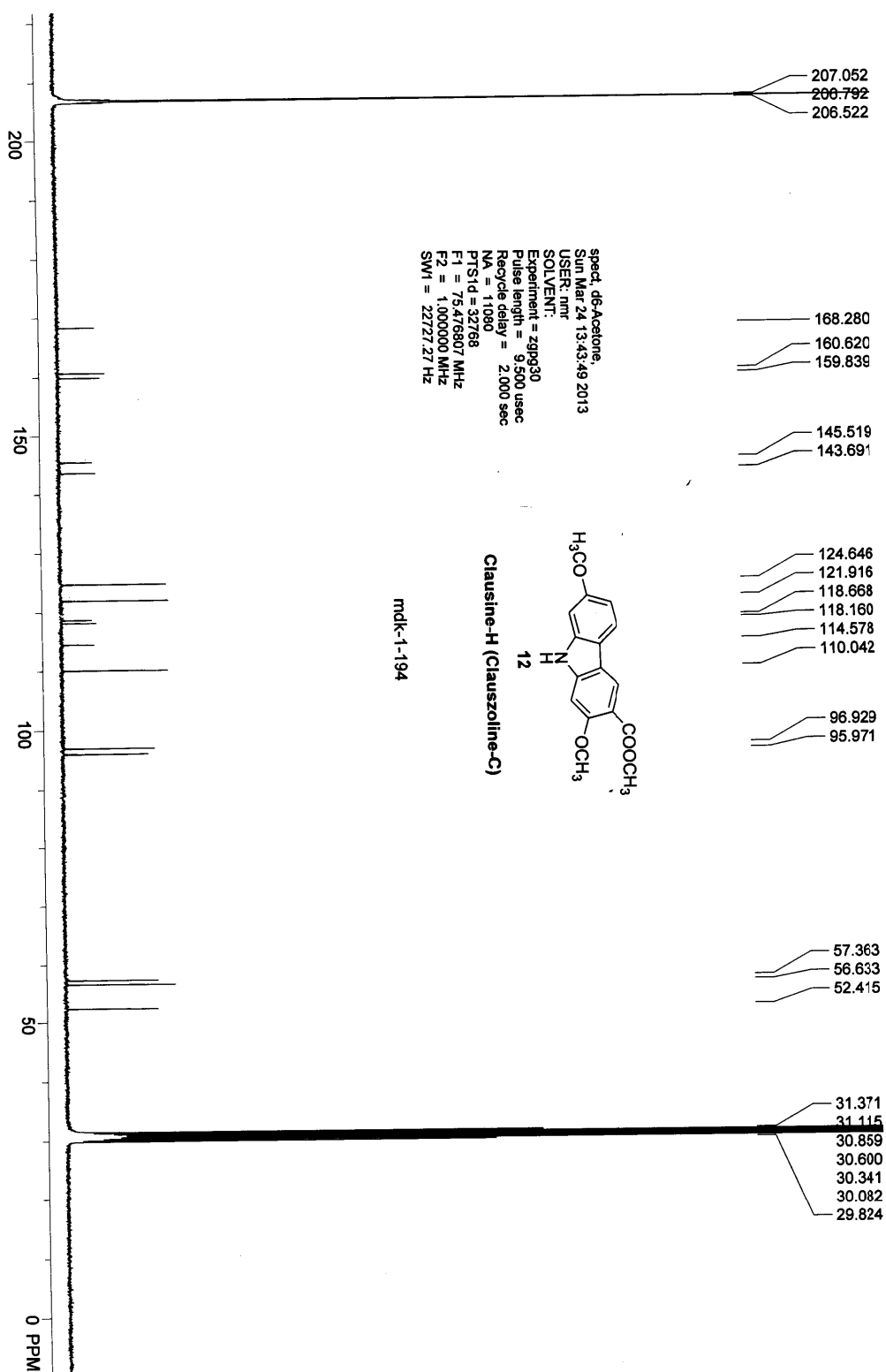


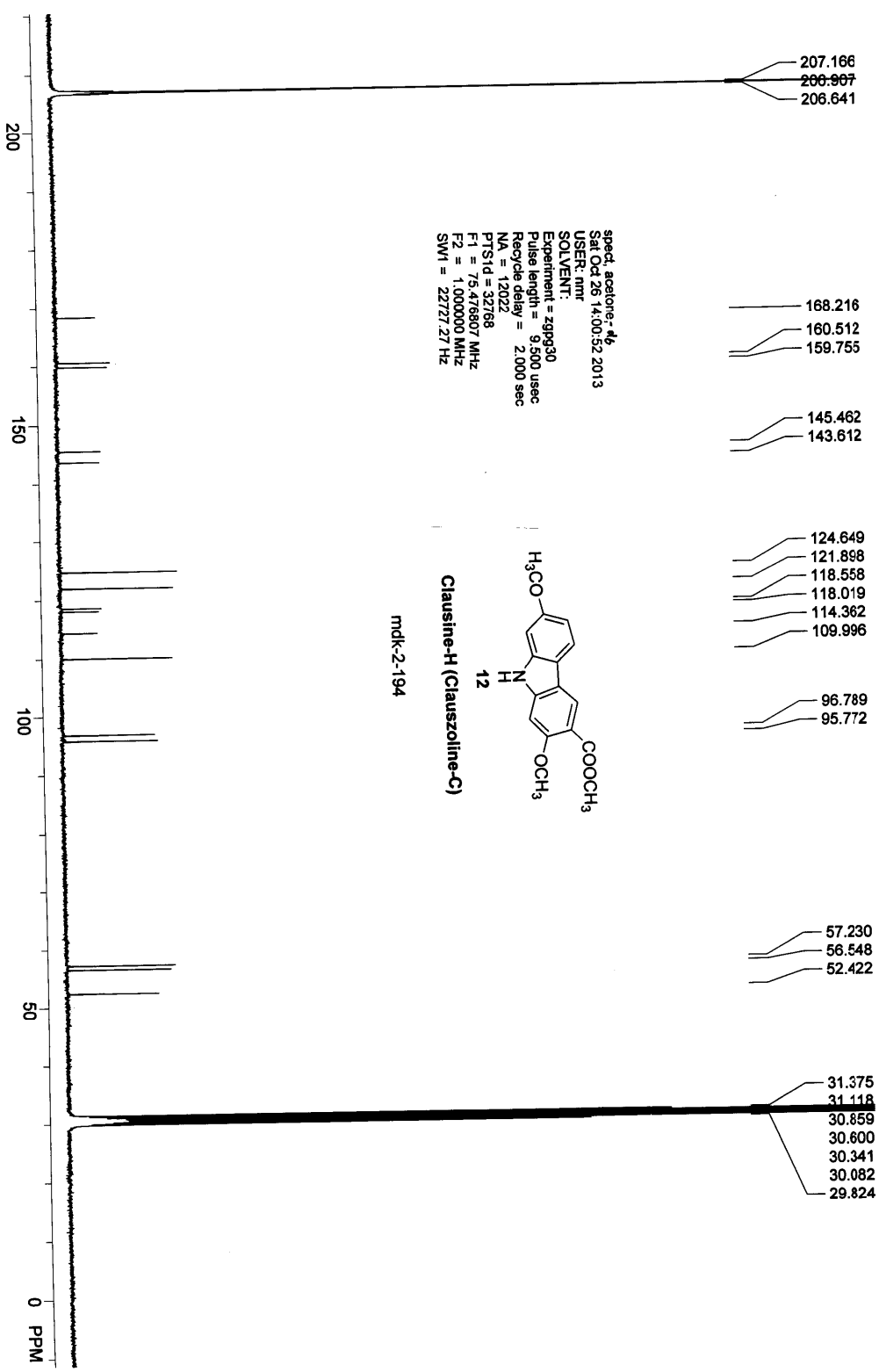
Clausine-H (Clauszoline-C)

spect Acetone-d₆
 Sun Mar 24 13:42:07 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTSD = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

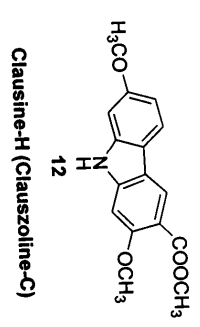
mdk-1-194



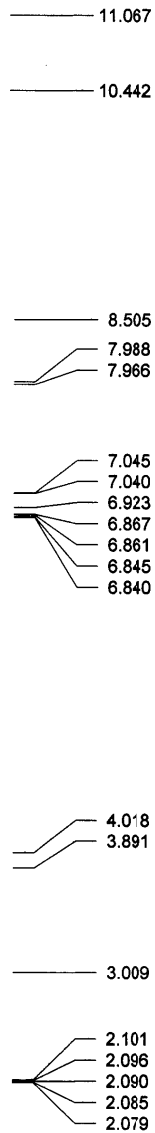




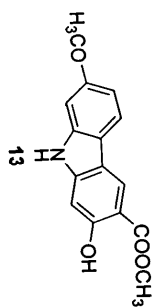
spec: acetone-^d₆
Sat Oct 26 14:00:52 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 12022
P1STD = 32768
F1 = 75.476807 MHz
F2 = 1,000000 MHz
SWH = 22727.27 Hz



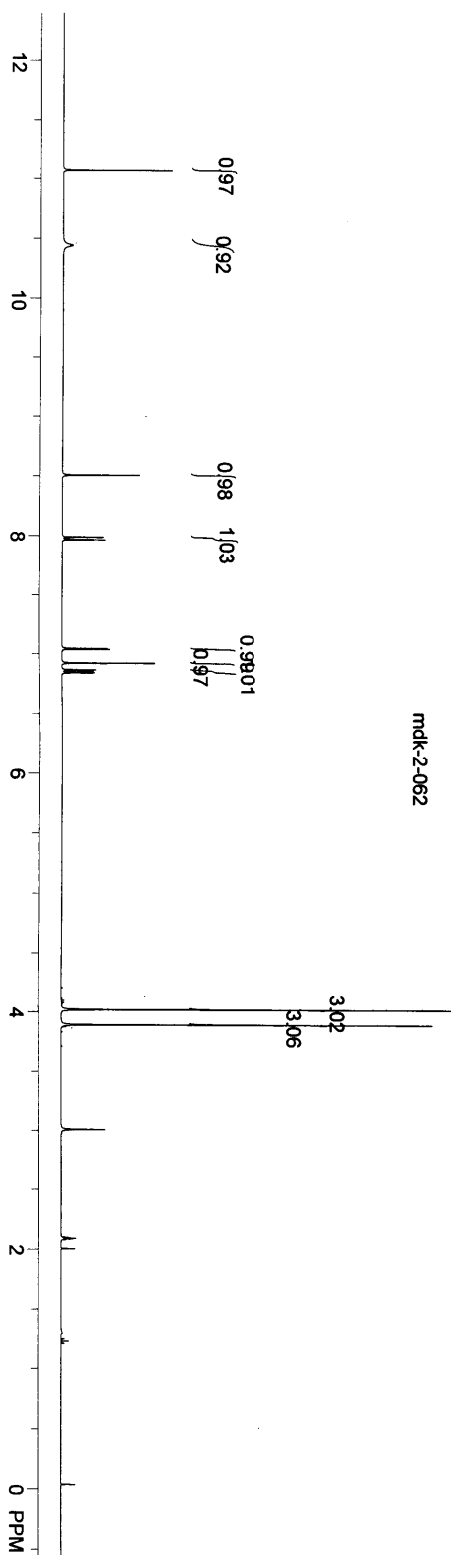
mdk-2-194

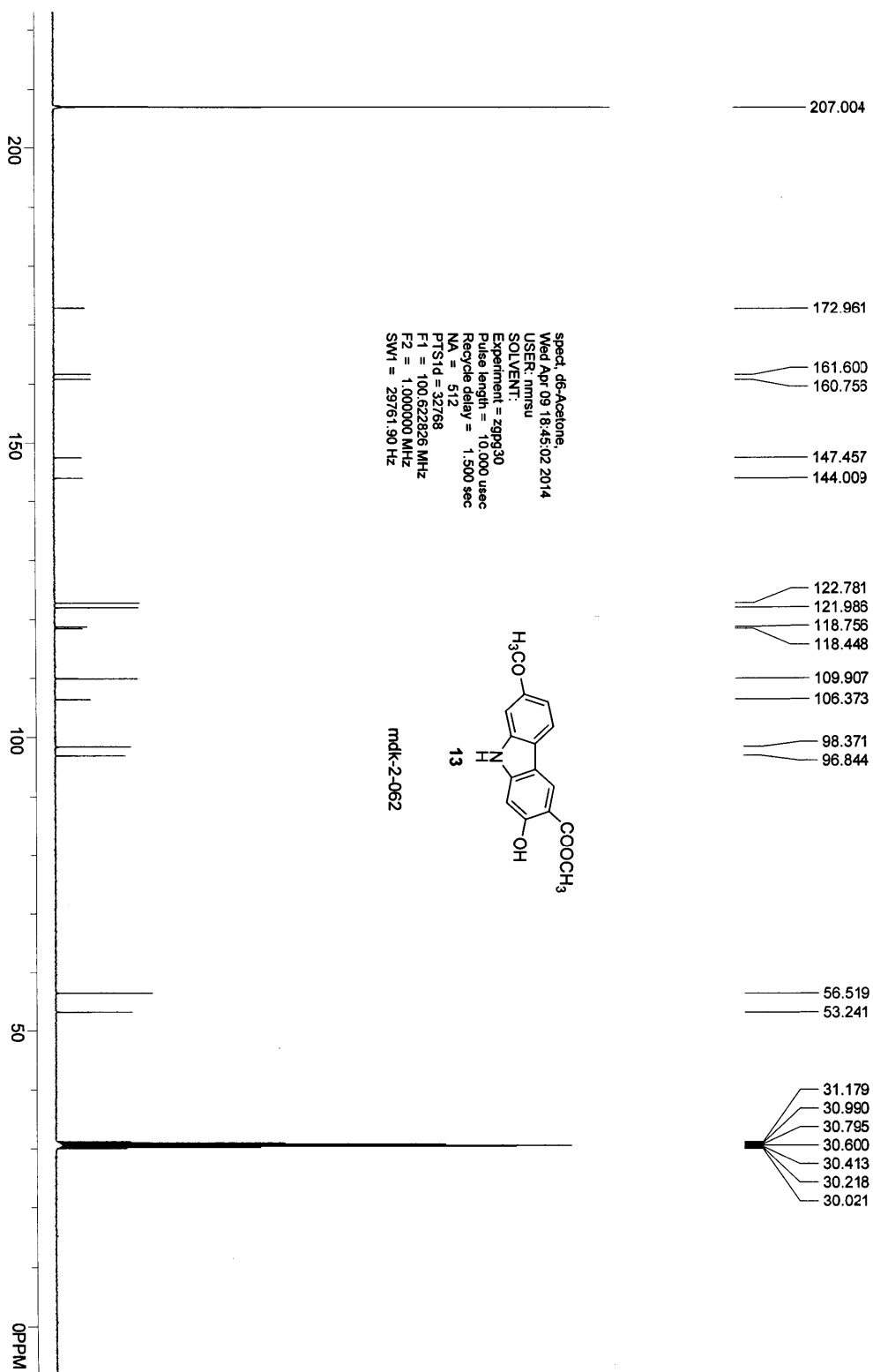


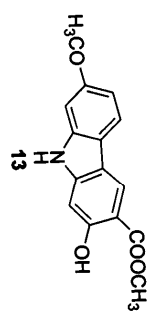
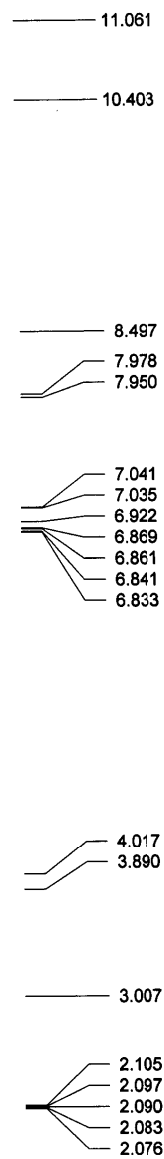
spect, d6-Acetone,
 Wed Apr 09 18:21:01 2014
 USER: nmrsu
 SOLVENT:
 Experiment = z930
 Pulse length = 15.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 P1STD = 65536
 F1 = 400.132813 MHz
 F2 = 1.000000 MHz
 SWH = 10000.00 Hz



mdk-2-062

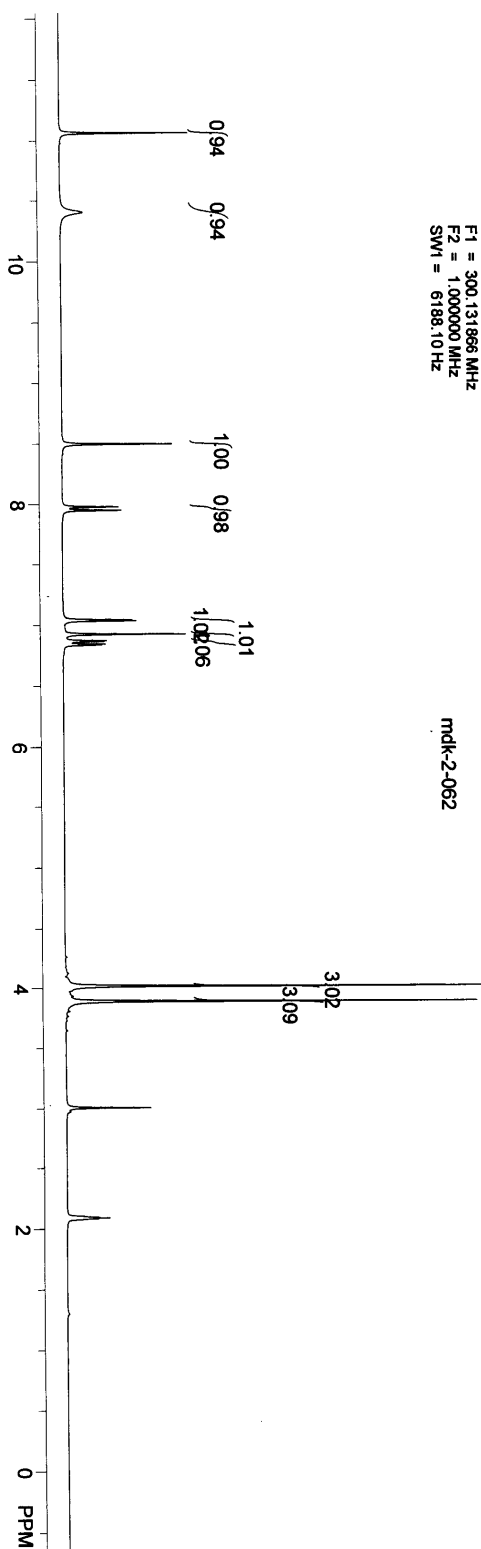


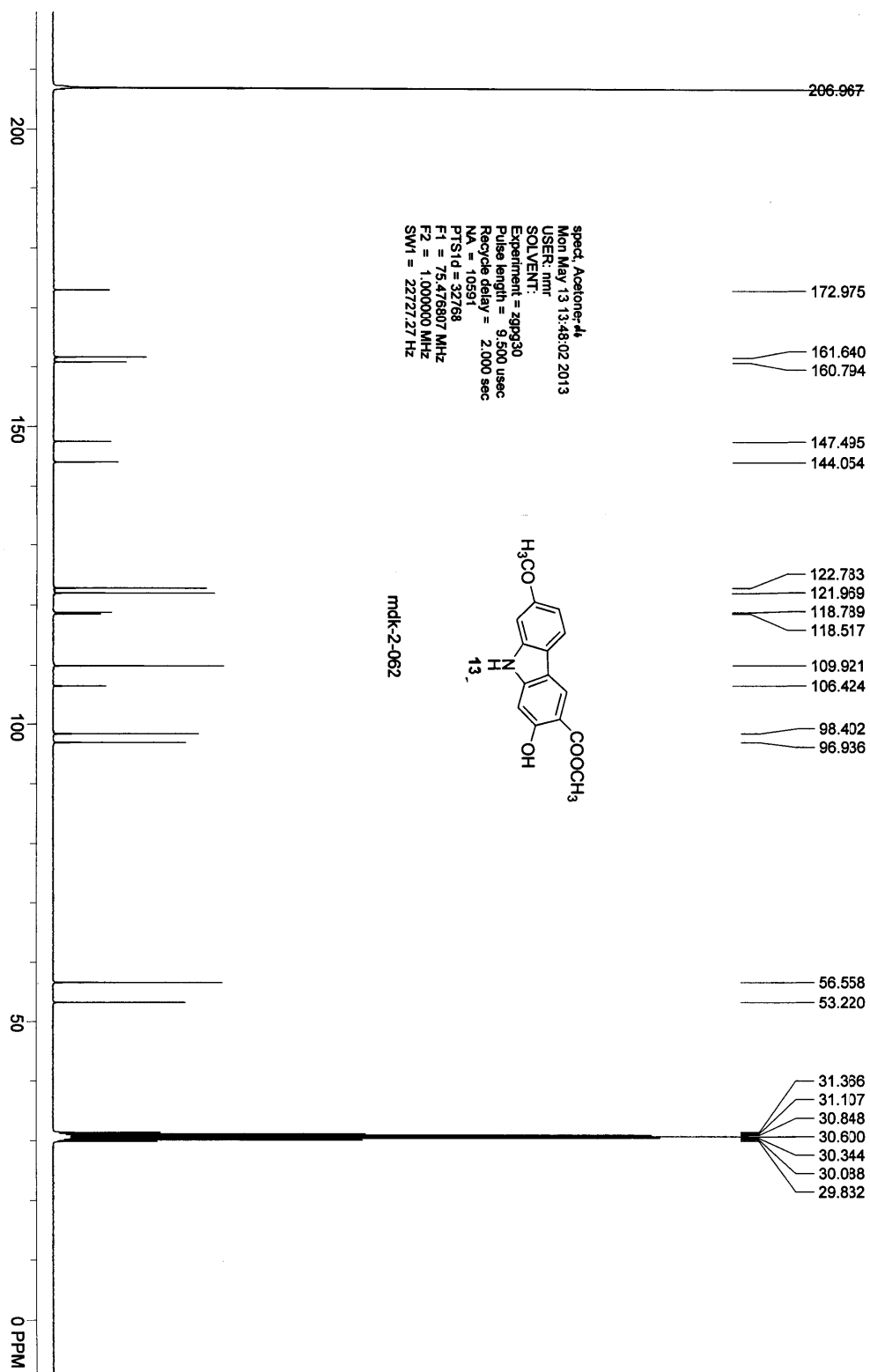


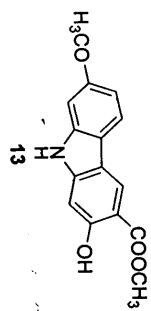
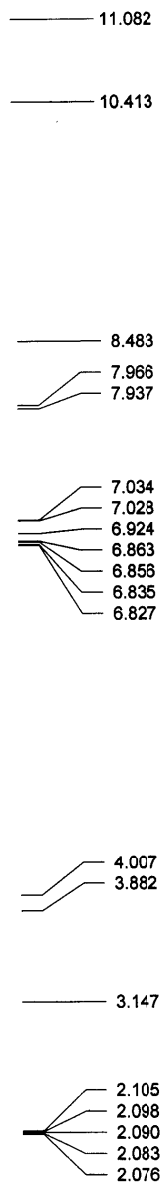


spect, Acetone-d₆
 Mon May 13 13:46:05 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1STD = 32768
 F1 = 300,131866 MHz
 F2 = 1,000000 MHz
 SWH = 6188.10 Hz

mdk-2-062

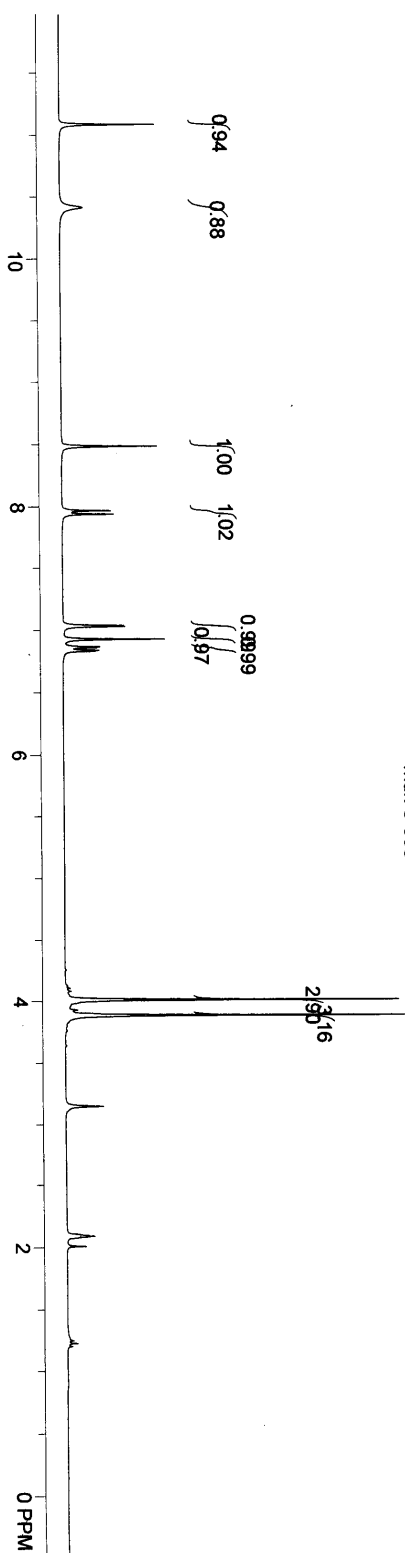


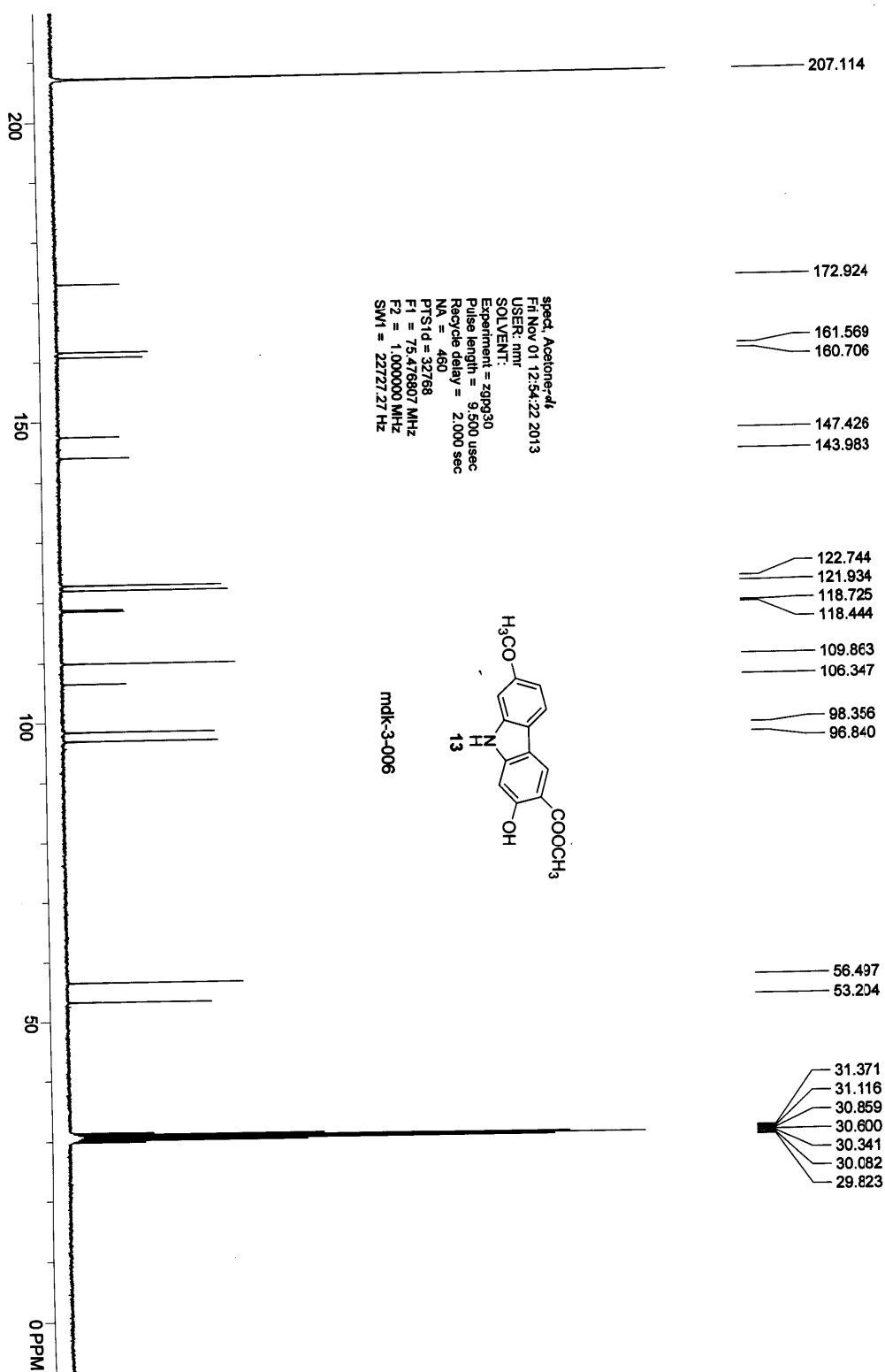


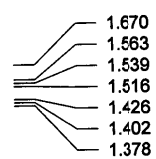
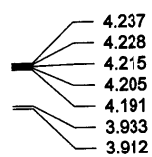
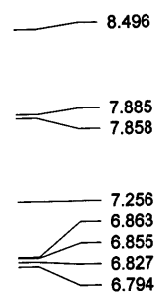


spect, Acetone-d₆
 Fri Nov 01 12:52:46 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 P2 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

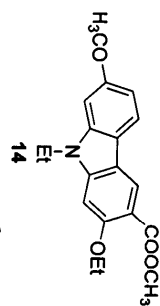
mdk-3-006





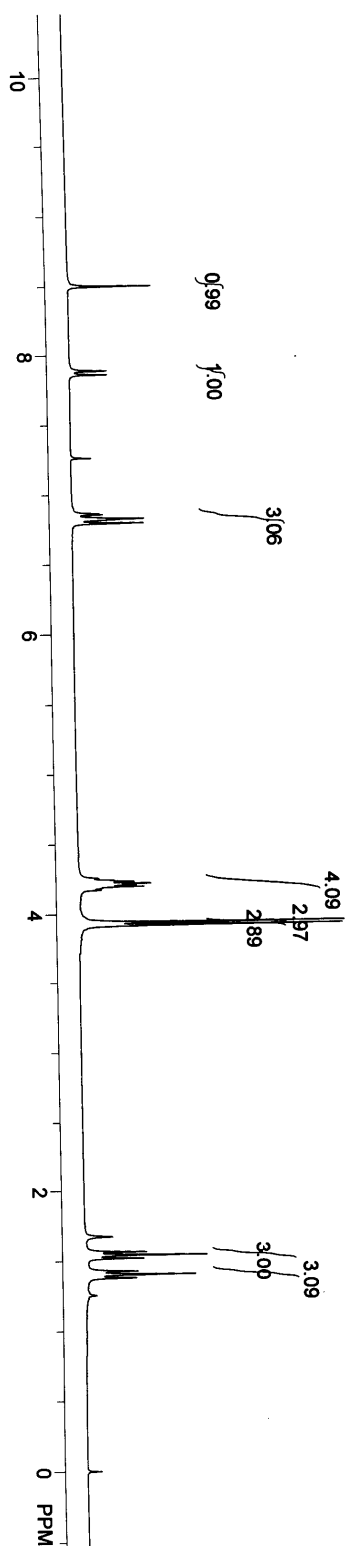


-0.000

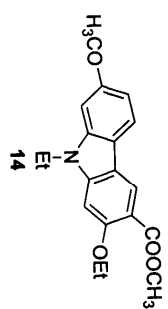


spect, CDCl₃,
 Thu Sep 12 03:18:34 2013
 USER: nmf
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

mdk-2-098



167.169
158.676
157.909
143.683
141.882
123.821
120.543
117.044
116.443
112.252
107.503
93.703
93.353
77.424
77.000
76.569
65.475
55.709
51.628
37.579
14.846
13.438



mdk-2-098

spect CDCI3
 Thu Sep 12 03:20:25 2013
 USER: nmf
 SOLVENT: zgp30
 Experiment = zgp30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 1592
 PtsId = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SWH = 22727.27 Hz

