Electronic Supplementary Information for

Metal-Free Tandem Reaction in Water:

An Efficient and Regioselective Synthesis of 3-Hydroxyisoindolin-1-ones

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Contents

X-ray crystallographic structure of compound 9Aa

Bond precision: C-C = 0.0046 A Wavelength=0.71073

Cell: a=9.2376(10) b=14.5788(16) c=14.9383(17)

alpha=66.775(2) beta=74.994(2) gamma=74.785(2)

Temperature: 293 K

	Calculated	Reported
Volume	1756.3(3)	1756.3(3)
Space group	P -1	P-1
Moiety formula	C22 H19 N O2	
Sum formula	C22 H19 N O2	C22 H19 N O2
Mr	329.38	329.38
Dx,g cm-3	1.246	1.246
Z	4	4
Mu (mm-1)	0.080	0.080
F000	696.0	696.0
F000'	696.30	
h,k,lmax	11,17,18	11,17,18
Nref	6535	6443
Tmin,Tmax	0.987,0.994	0.767,1.000
Tmin'	0.979	

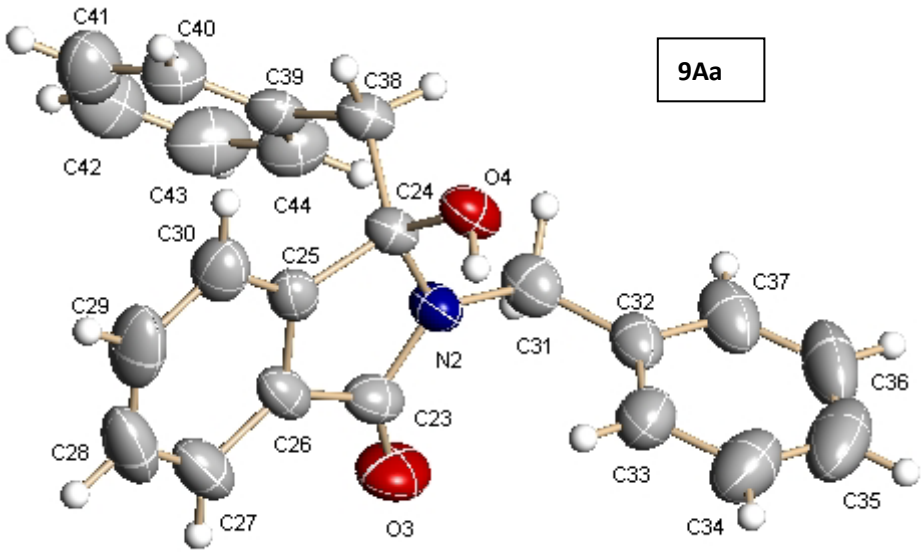
Correction method= EMPIRICAL

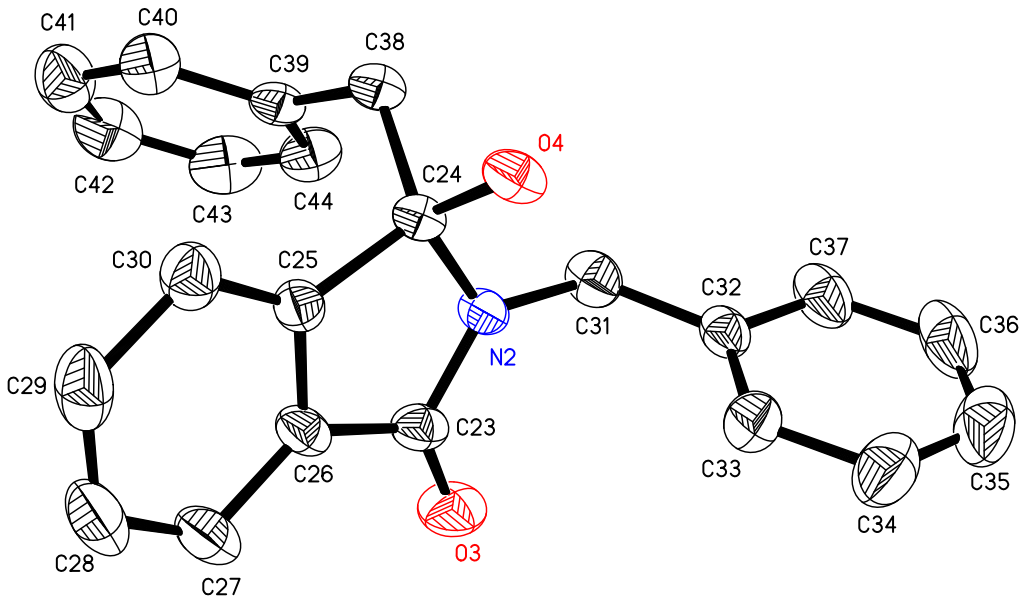
Data completeness= 0.986 Theta(max)= 25.500

R(reflections)= 0.0516(2759) wR2(reflections)= 0.1038(6443)

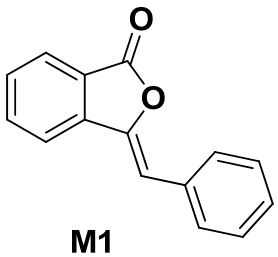
S = 0.844 Npar= 453

CCDC 755262 contains the supplementary crystallographic data for this paper. These data can be also obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.


9Aa
C41
C40
C39
C38
C42
C43
C44
C24
O4
C30
C25
C29
N2
C31
C32
C37
C36
C23
C28
C26
C33
C35
C27
O3
C34


C41
C40
C39
C38
O4
C42
C43
C44
C24
C30
C25
C37
C32
C36
N2
C31
C29
C23
C33
C35
C26
O3
C34
C28
C27

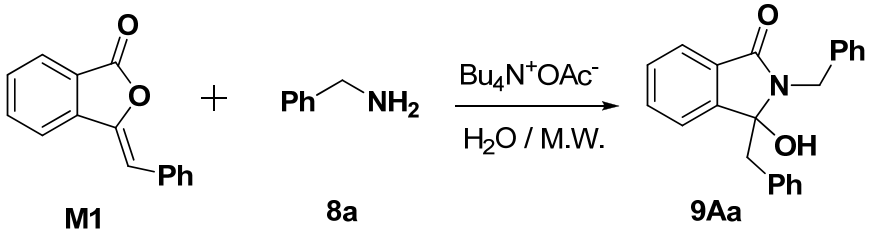
General Procedure for Synthesis of Intermediate M1



M1

To a suspension solution of 2-(phenylethynyl)benzoic acid **(7A**, 0.1 mmol) in H_2O (3 mL) were added $Bu_4N^+OAc^-$ (5 mol%). Subsequently, the reaction vial was sealed under Ar protection and the mixture was heated to 100 ^oC for 20 min under the participation of microwave irritation. The cold mixture was concentrated in vaccum, and the resulting residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 10/1) to afford the expected enol lactone intermediate 3-benzylideneisobenzofuran-1(3*H*)-one (**M1**). The characterization data obtained are as follows: ^1H NMR ($CDCl_3$, 300 MHz) δ 7.95 (d, J = 7.5 Hz, 1H), 7.86 (d, J = 7.8 Hz, 2H), 7.75 (m, 2H), 7.55 (t, 1H), 7.42 (t, 2H), 7.32 (m, 1H), 6.43 (s, 1H); LRMS (EI) *m/z* 222 (M^+). HRMS (EI) *m/z* calcd $C_{15}H_{10}O_2$ $(M)^+$ 222.0681, found 222.0690. These spectral data are consistent with previous reports.[S1]

Synthesis of the target product 9Aa from intermediate M1



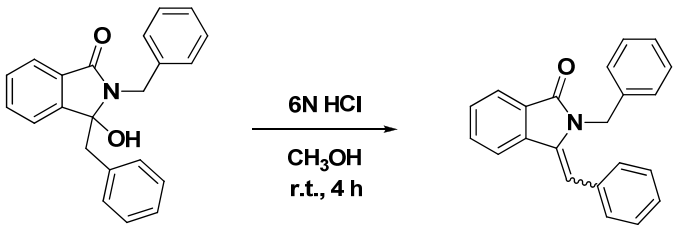
M1 **8a** **9Aa**

To a suspension solution of **M1** (0.1 mmol) in H_2O (3 mL) was added $Bu_4N^+OAc^-$ (5 mol%). After the mixture was stirred at room temperature for 10 min in the presence of Ar protection, benzyl amine (**8a**, 0.2 mmol) was added. Subsequently, the resulting mixture was underwent microwave irritation at 100 ^oC under Ar protection for 10 min. And then, the cold mixture was concentrated in vaccum, the resulting residue was purified by flash chromatography (petroleum ether/ethyl acetate = 4/1, v/v) to afford the expected product **9Aa** with 95% yield.

References:

[S1] (a) M. Uchiyama, H. Ozawa, K. Takuma, Y. Matsumoto, M. Yonehara, K. Hiroya, T. Sakamoto, *Org. Lett.,* 2006, **8**, 5517; (b) L. Zhou, H. F. Jiang, *Tetrahedron Lett.*, 2007, **48**, 8449; (c) E. Marchal, P. Uriac, B. Legouin, L. Toupet, P. van de Weghe, *Tetrahedron*, 2007, **63**, 9979; (d) C. Kanazawa, M. Terada, *Tetrahedron Lett.* 2007, **48**, 933.

Dehydration Reaction Study for Compound 9Aa



9Aa **11 (E/Z = 10/1)**
(98% yield)

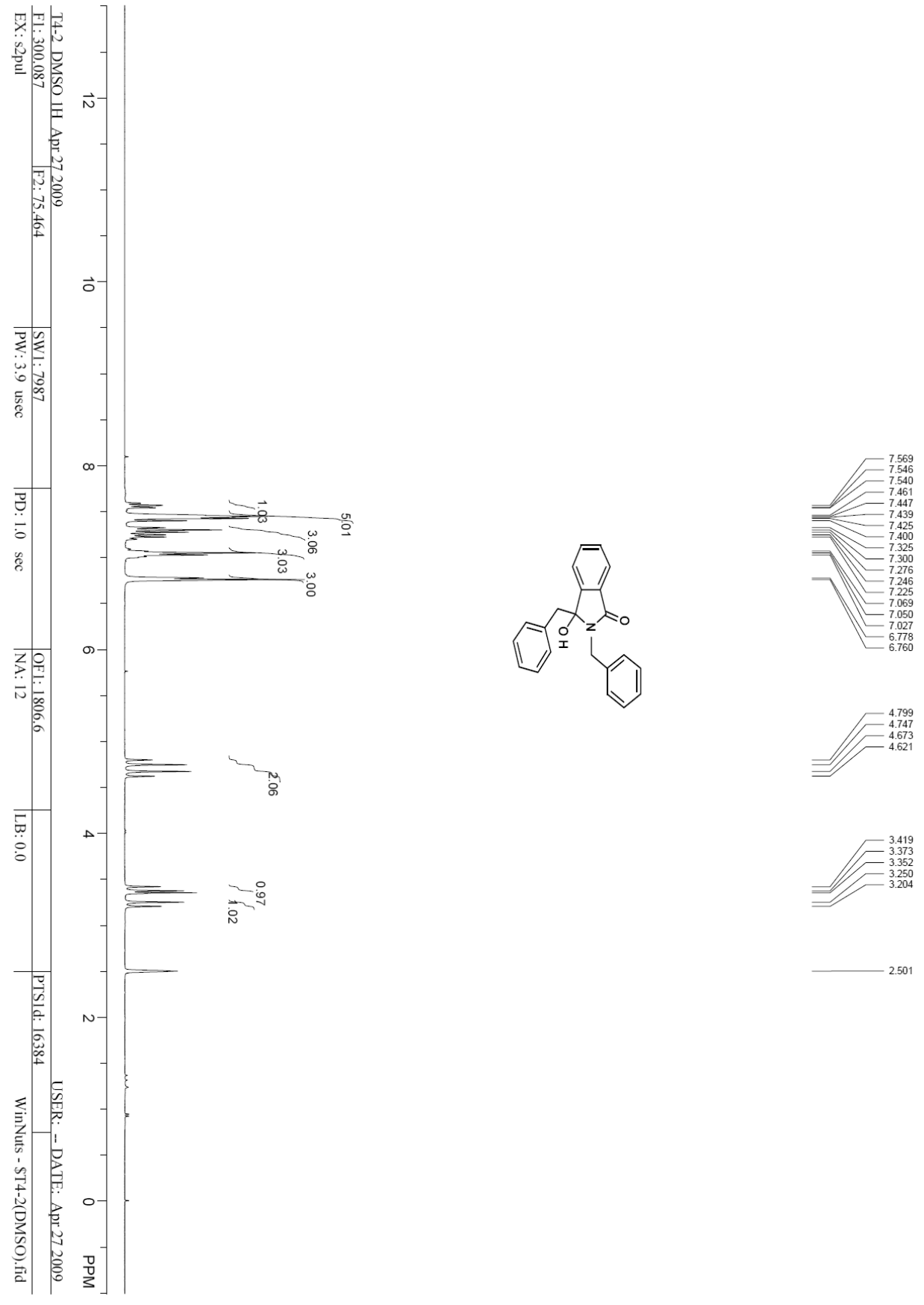
A solution of **9Aa** (0.5 mmol) in 6N hydrochloric acid solution (2 mL) and methanol (20 mL) was stirred at room temperature for 4 h. The solvent was removed under reduced pressure, and the residue was purified by a flash chromatography (petroleum ether/ethyl acetate = 25/1, v/v, as an eluent) to product (*E*)-**11** (as major product) and (*Z*)-**11**, respectively, and the E/*Z*-isomers were determined by comparing the ^{1}H NMR with the reported spectrum data.[S2]

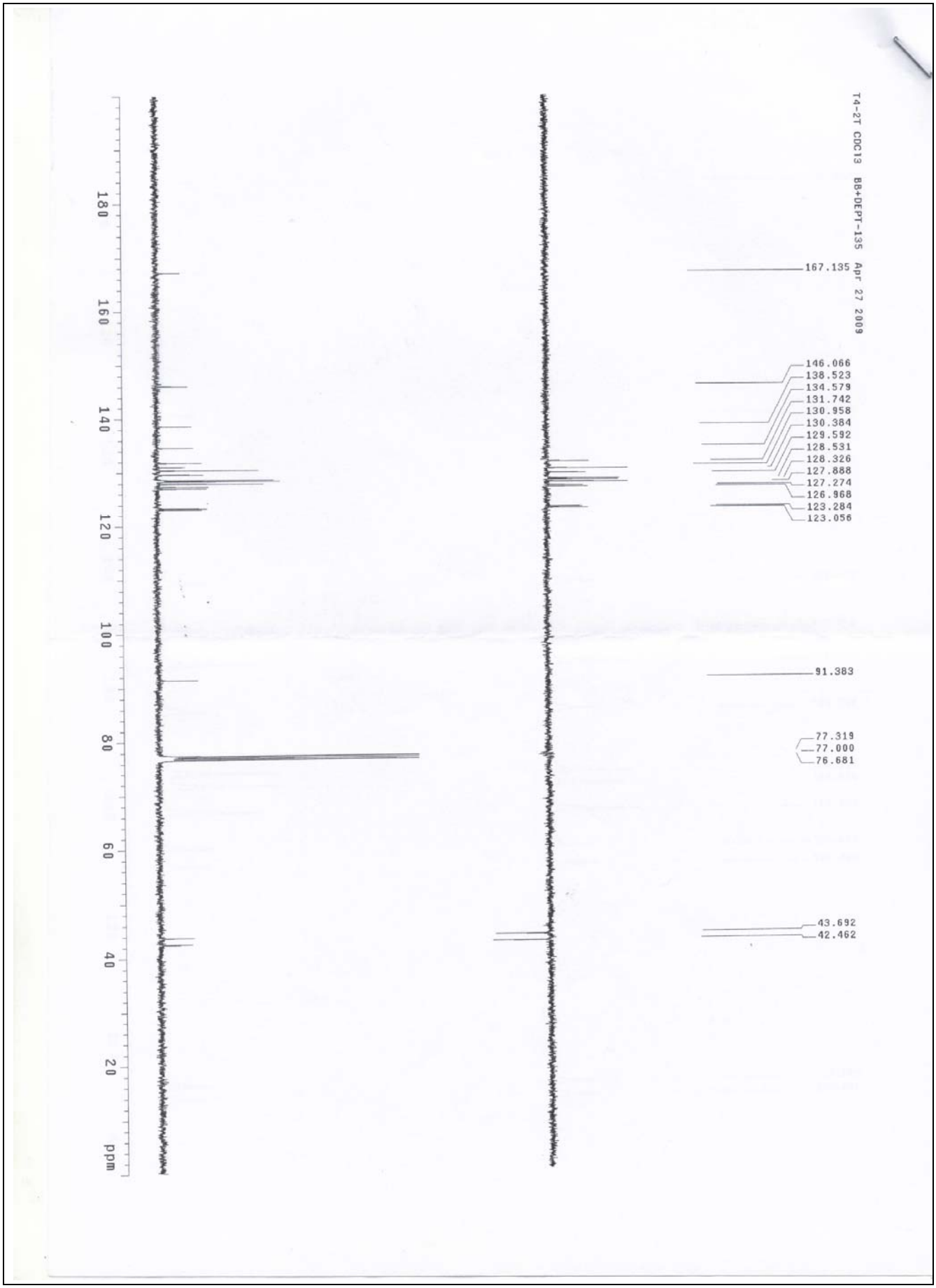
The characterization data are as follows: **(*E*)-11**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.91 (d, J = 7.2 Hz, 1H), 7.32 (m, 13H), 6.47 (s, 1H), 5.14 (s, 2H); LRMS (EI) *m/z* 311 (M^+). **(*Z*)-11**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.93 (d, J = 7.2 Hz, 1H), 7.74 (d, J = 7.5 Hz, 1H), 7.62 (t, 1H), 7.54 (t, 1H), 7.31 (m, 2H), 7.24 (m, 1H), 7.06 (m, 5H), 6.72 (s, 1H), 6.52 (d, J = 7.5 Hz, 2H), 4.93 (s, 2H); LRMS (EI) *m/z* 311 (M^+).

[S2] (a) L. Li, M. Wang, X. Zhang, Y. Jiang, D. Ma, *Org. Lett.* 2009**, 11**, 1309. (b) K. Cherry, A. Duchene, J. Thibonnet, J. L. Parrain, E. Anselmi, M. Abarbri, *Synthesis,* 2009, **2**, 257; (c) H. Cao, L. McNamee, H. Alper, *Org. Lett.,* 2008, **10**, 5281; (d) N. G. Kundu, M. W. Khan, *Tetrahedron*, 2000, **56**, 4777; (e) W. W. Khan, N. G. Kundu, *Synlett.,* 1997, **12**, 1435.

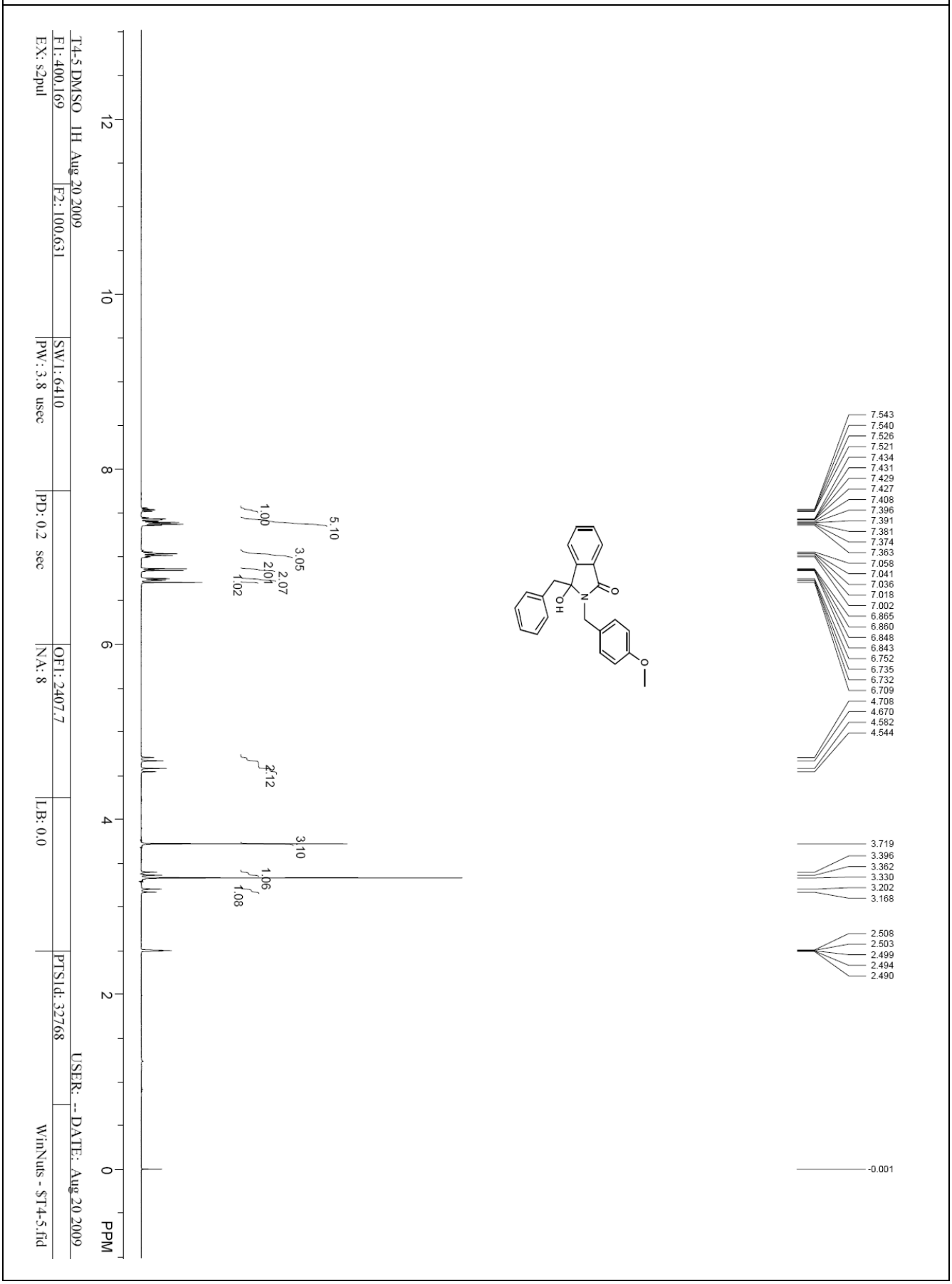
Copies of ^{1}H NMR and ^{13}C NMR of Compounds

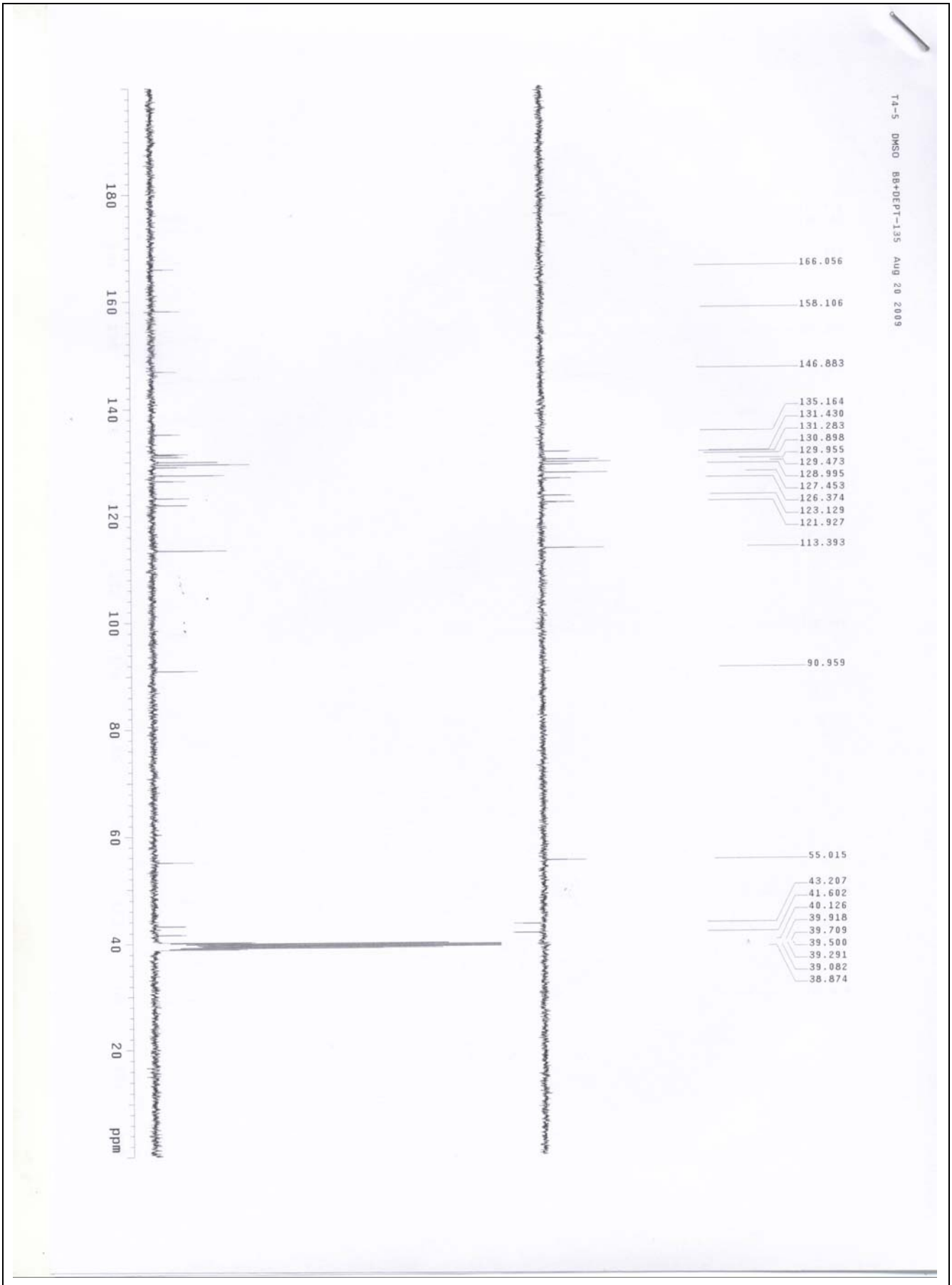
2,3-dibenzyl-3-hydroxyisoindolin-1-one (9Aa)



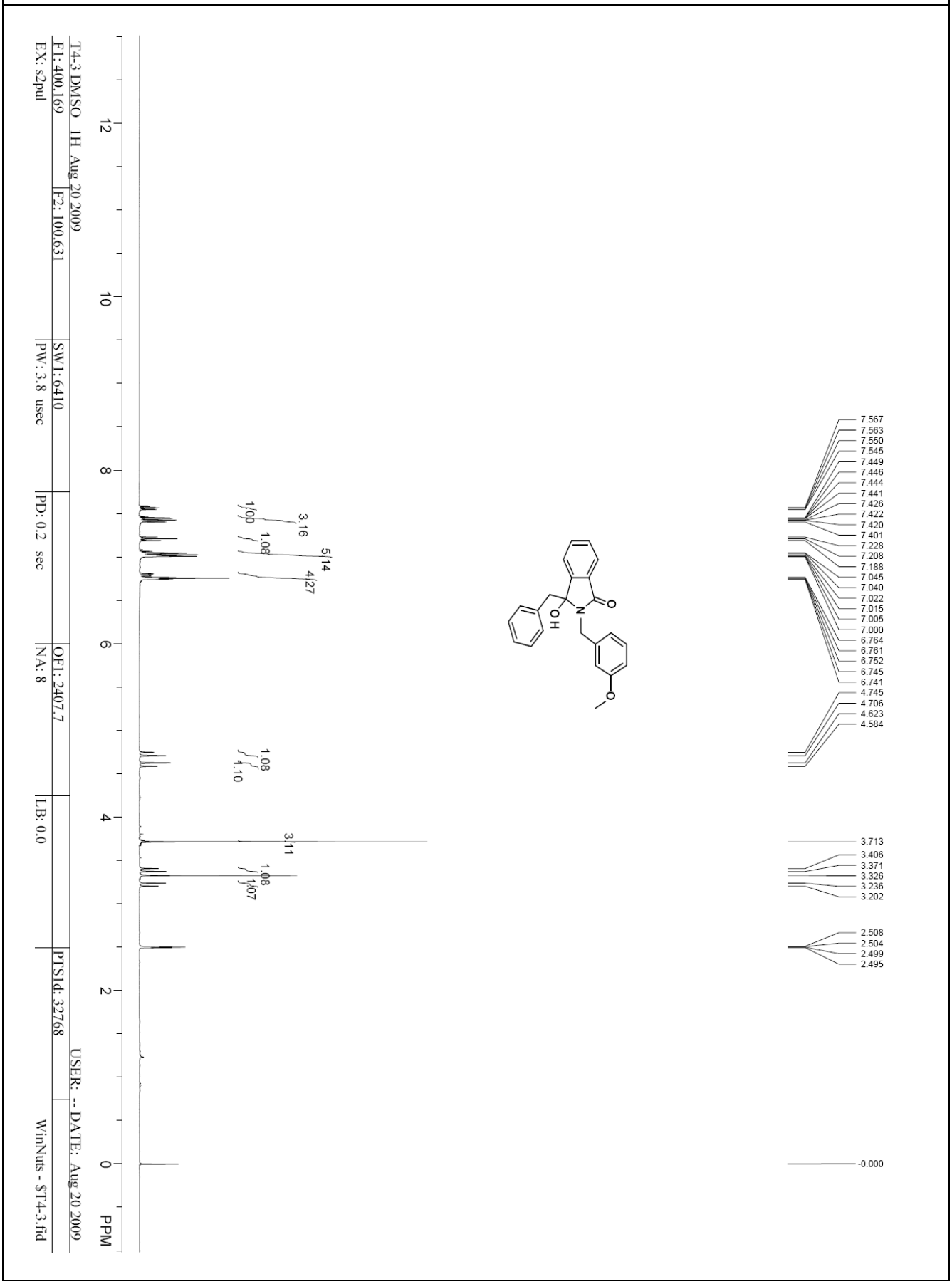

T4-2T CDCl3 BB+DEPT-135 Apr 27 2009
167.135
146.066
138.523
134.579
131.742
130.958
130.384
129.592
128.531
128.326
127.888
127.274
126.968
123.284
123.056
91.383
77.319
77.000
76.681
43.692
42.462
180
160
140
120
100
80
60
40
20
ppm

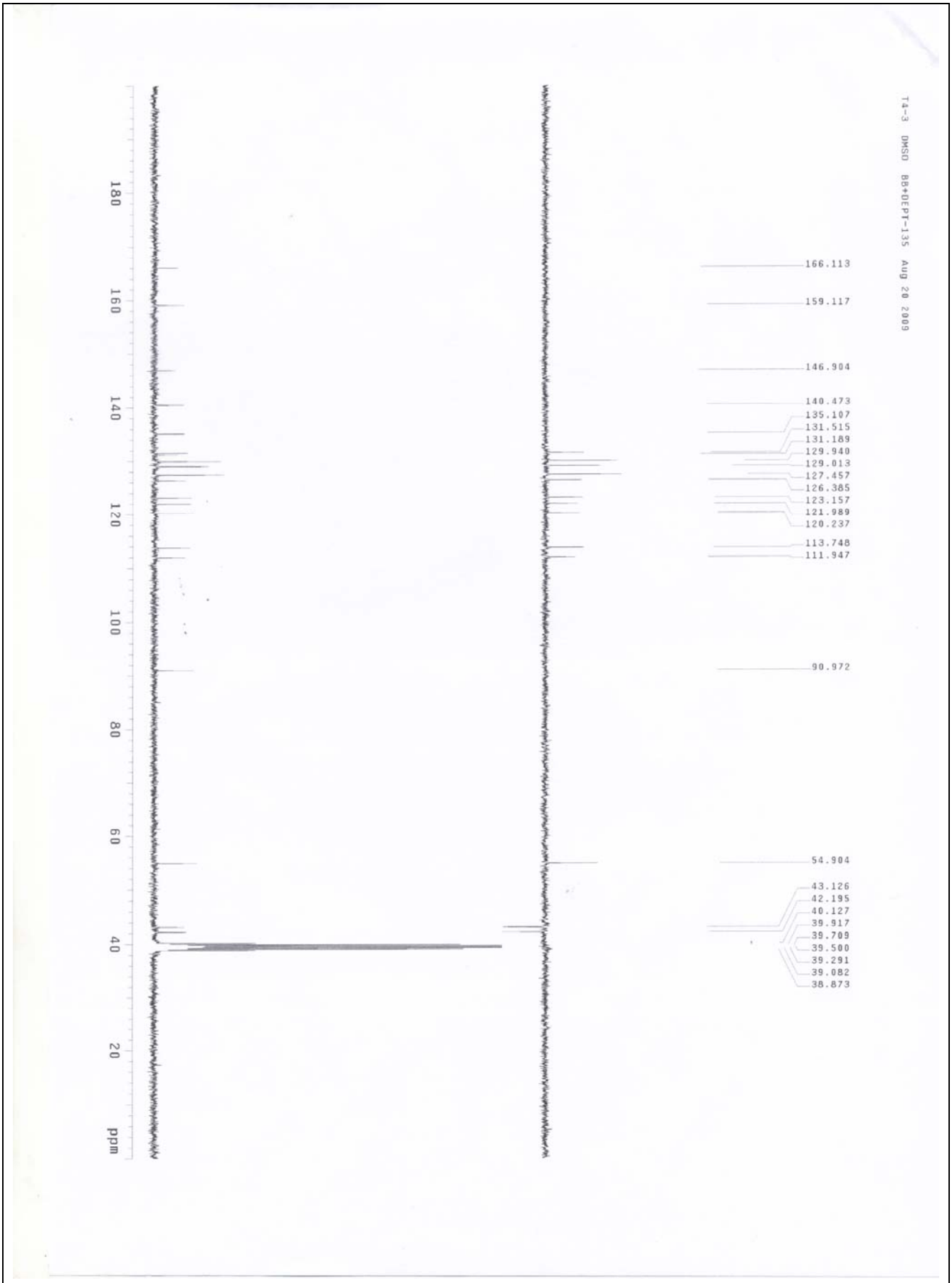
3-benzyl-3-hydroxy-2-(4-methoxybenzyl)isoindolin-1-one (9Ab)



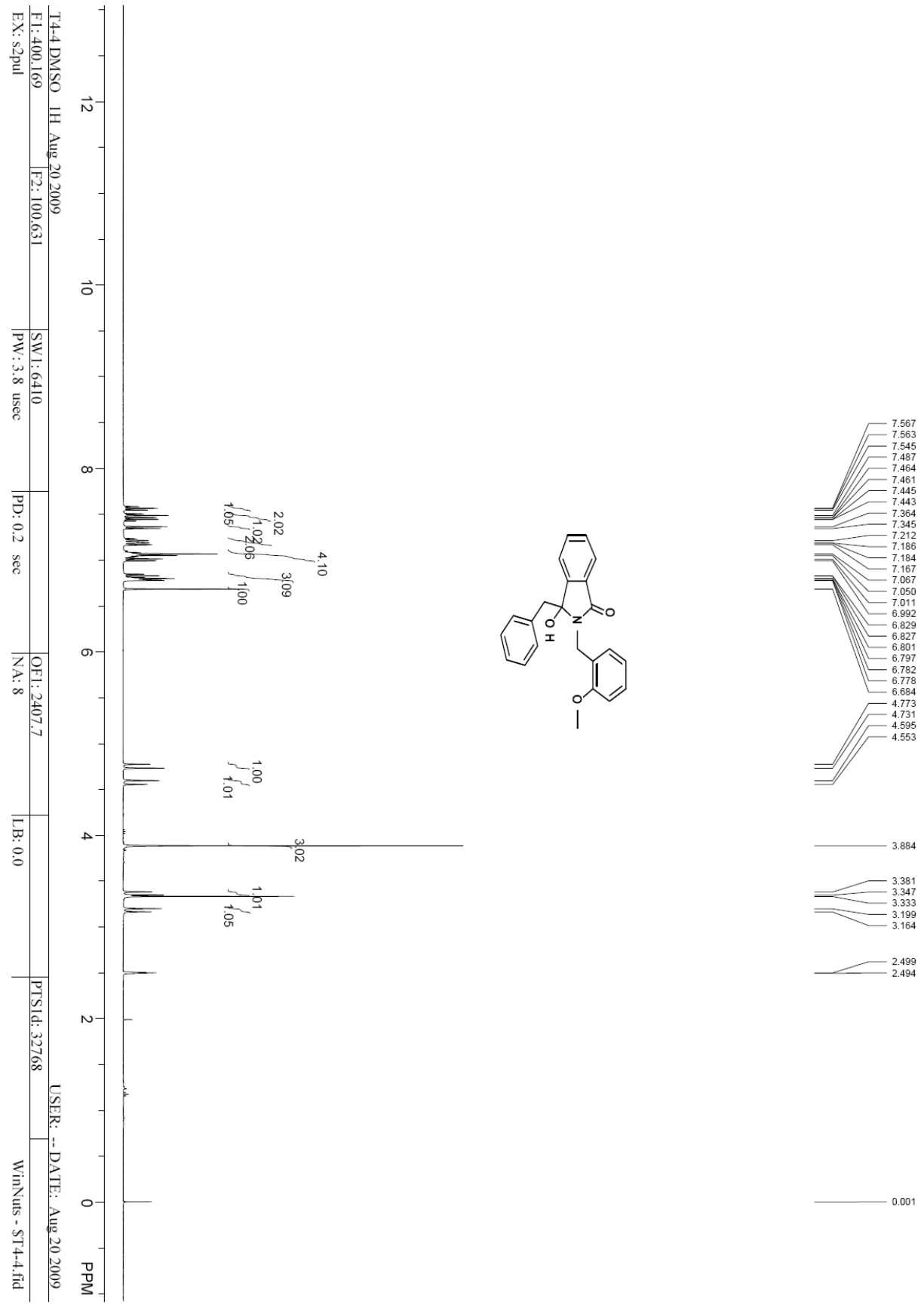

T4-5 DMSO BB+DEPT-135 Aug 20 2009
166.056
158.106
146.883
135.164
131.430
131.283
130.898
129.955
129.473
128.995
127.453
126.374
123.129
121.927
113.393
90.959
55.015
43.207
41.602
40.126
39.918
39.709
39.500
39.291
39.082
38.874
180
160
140
120
100
80
60
40
20
ppm

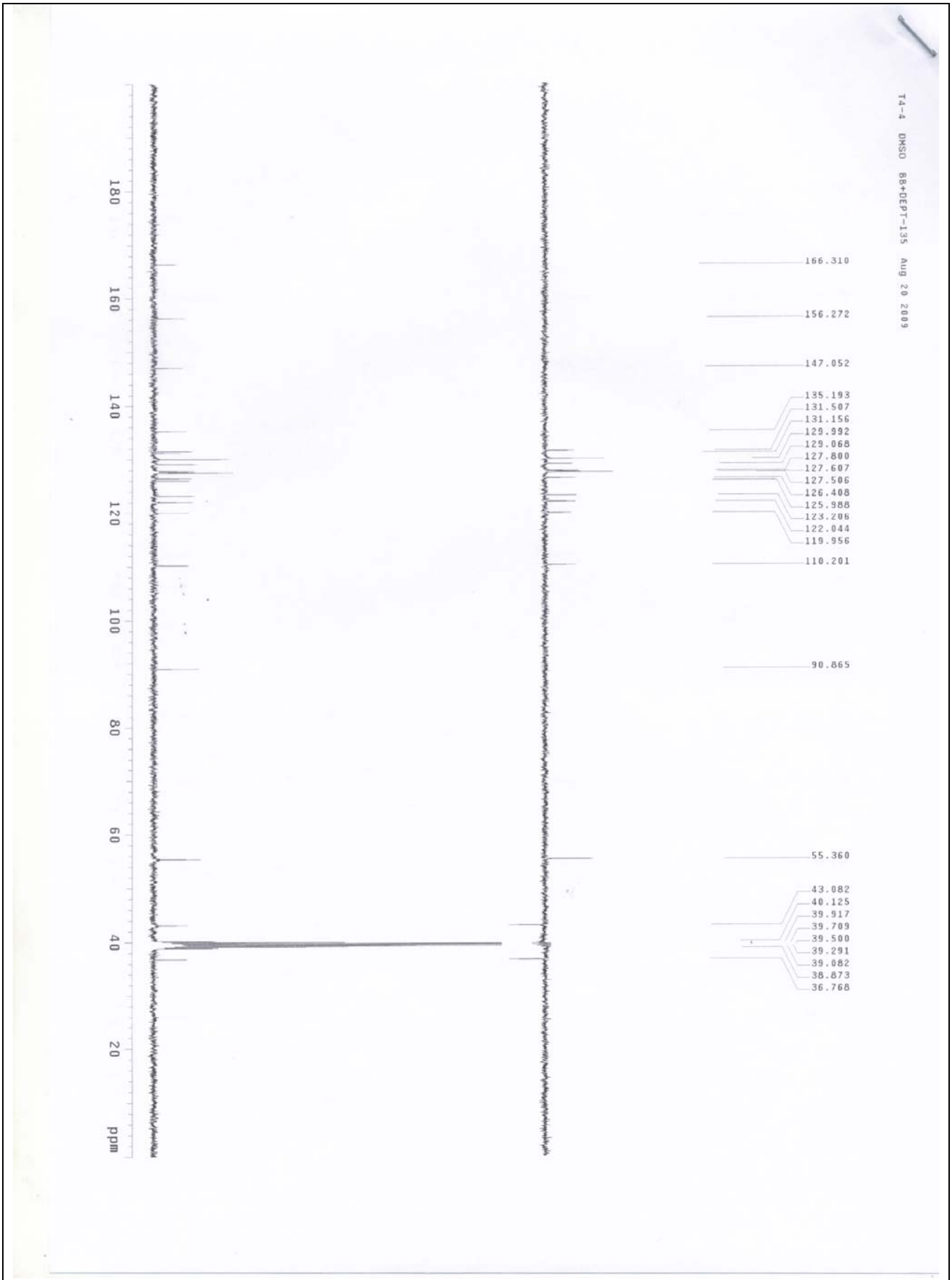
3-benzyl-3-hydroxy-2-(3-methoxybenzyl)isoindolin-1-one (9Ac)



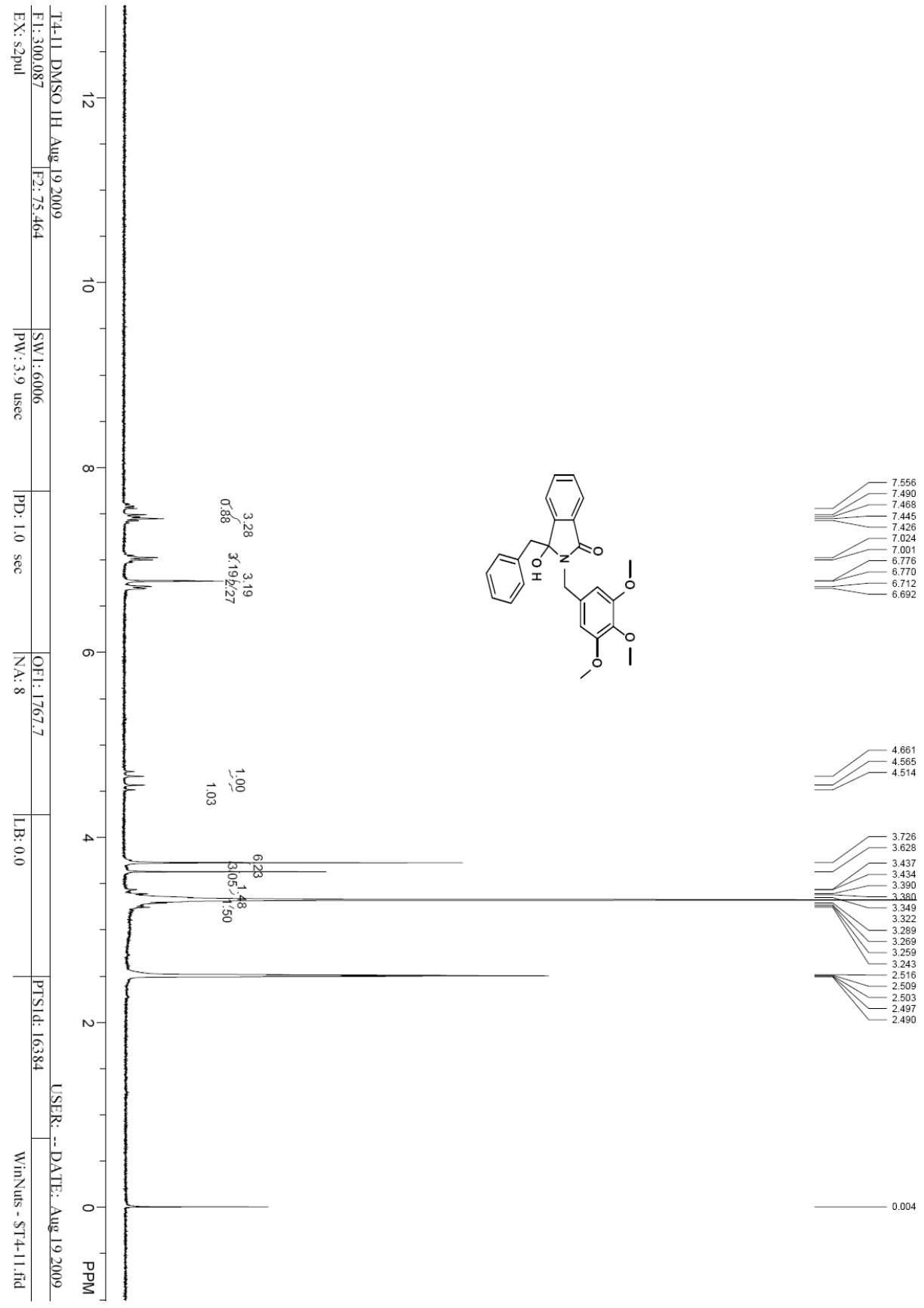

T4-3 DMSO BB+DEPT-135 Aug 20 2009
166.113
159.117
146.904
140.473
135.107
131.515
131.189
129.940
129.013
127.457
126.385
123.157
121.989
120.237
113.748
111.947
90.972
54.904
43.126
42.195
40.127
39.917
39.709
39.500
39.291
39.082
38.873
180
160
140
120
100
80
60
40
20
ppm

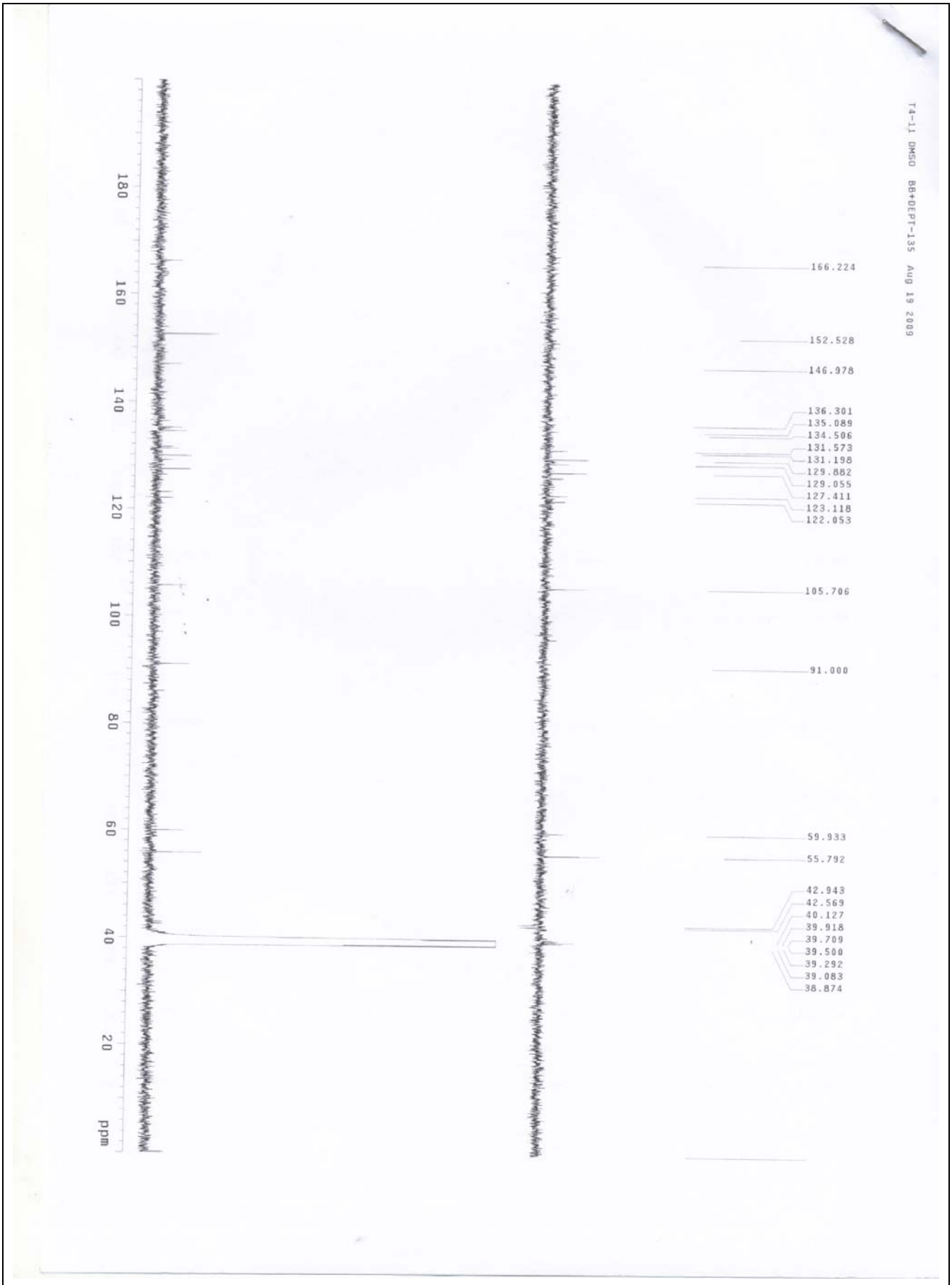
3-benzyl-3-hydroxy-2-(2-methoxybenzyl)isoindolin-1-one (9Ad)



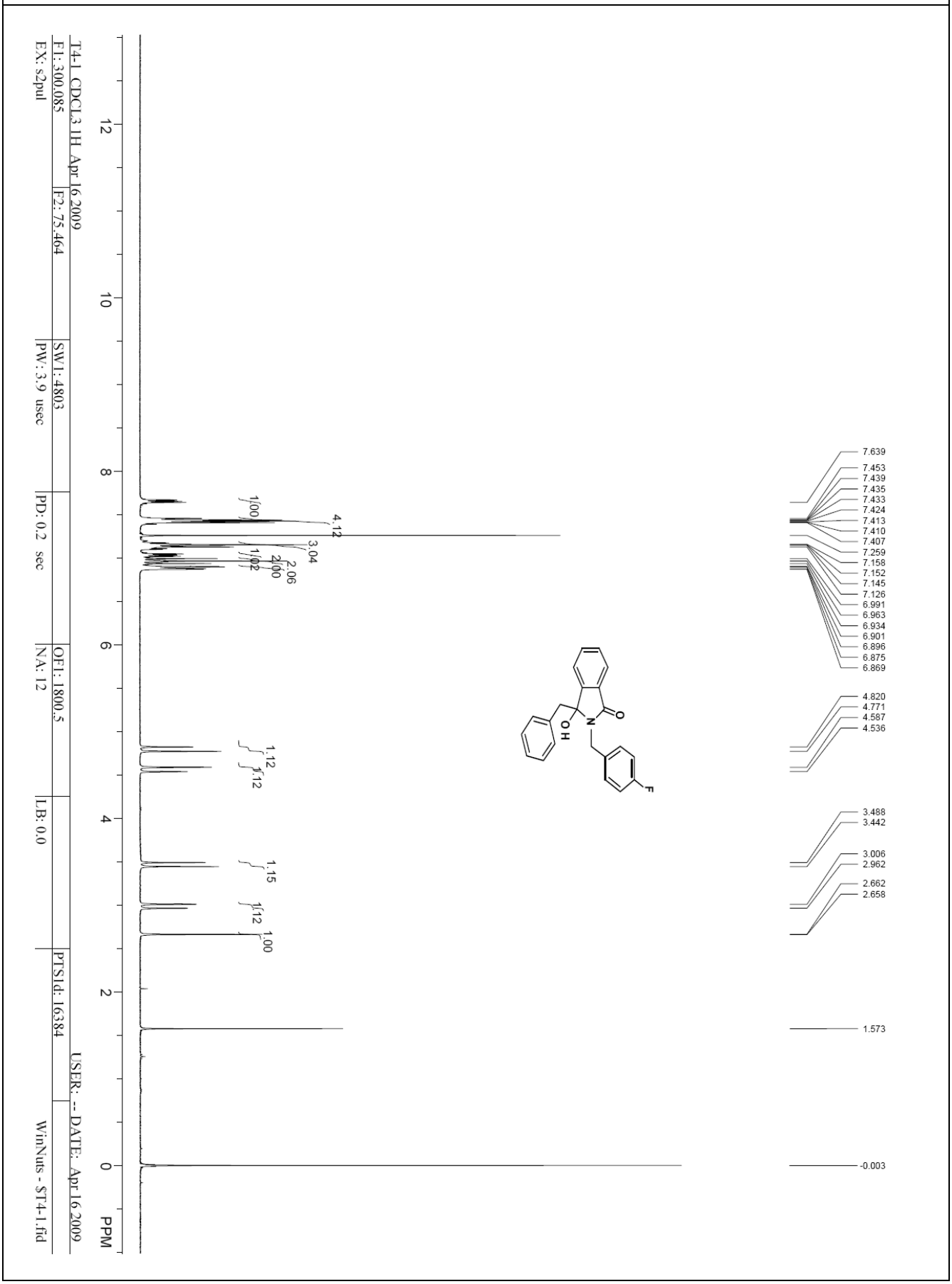

T4-4 DMSO BB+DEPT-135 Aug 20 2009
166.310
156.272
147.052
135.193
131.507
131.156
129.992
129.068
127.800
127.607
127.506
126.408
125.988
123.206
122.044
119.956
110.201
90.865
55.360
43.082
40.125
39.917
39.709
39.500
39.291
39.082
38.873
36.768
180
160
140
120
100
80
60
40
20
ppm

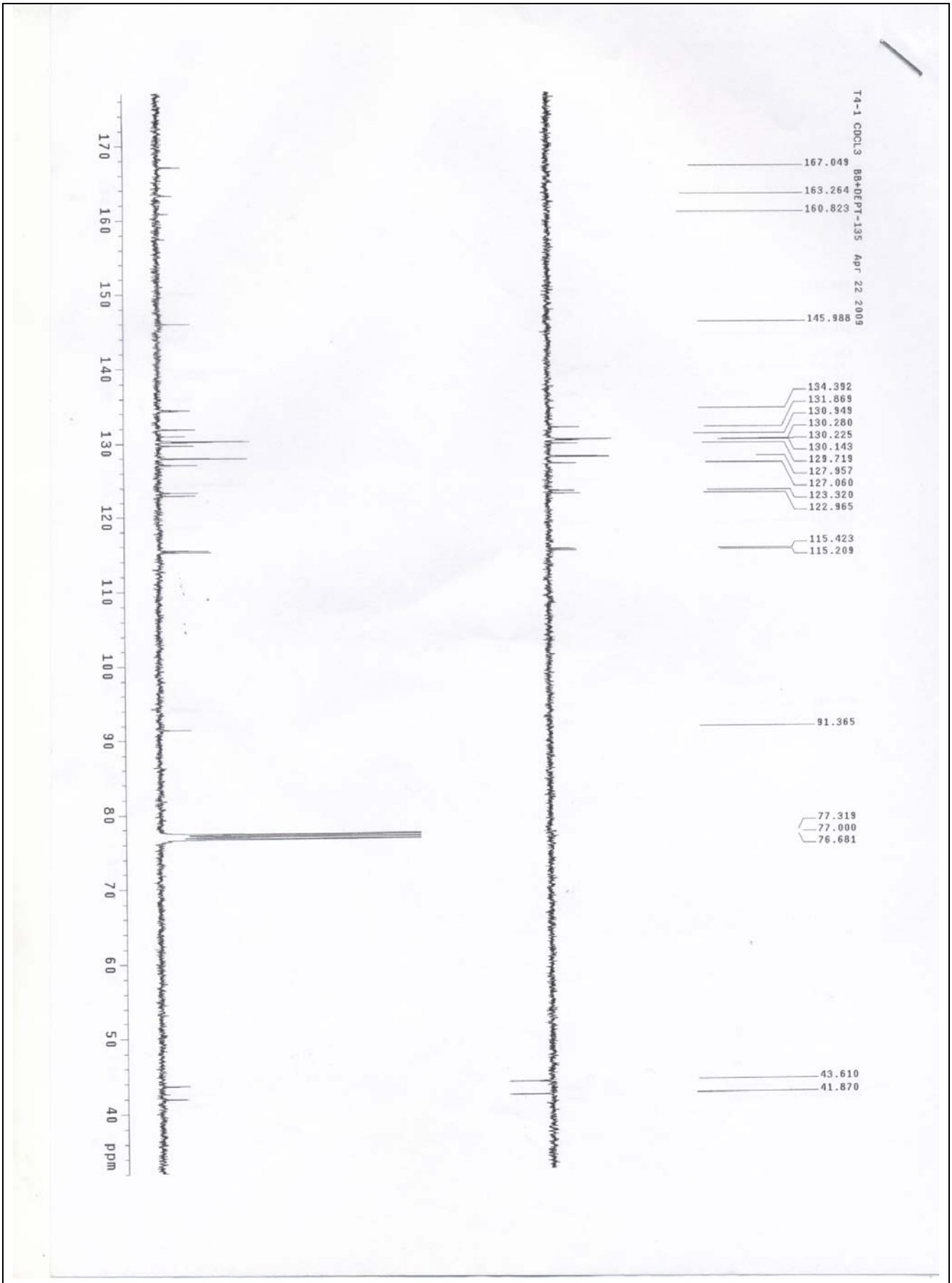
3-benzyl-3-hydroxy-2-(3,4,5-trimethoxybenzyl)isoindolin-1-one (9Ae)




T4-11 DMSO BB+DEPT-135 Aug 19 2009
166.224
152.528
146.978
136.301
135.089
134.506
131.573
131.198
129.882
129.055
127.411
123.118
122.053
105.706
91.000
59.933
55.792
42.943
42.569
40.127
39.918
39.709
39.500
39.292
39.083
38.874
180
160
140
120
100
80
60
40
20
ppm

3-benzyl-2-(4-fluorobenzyl)-3-hydroxyisoindolin-1-one (9Af)

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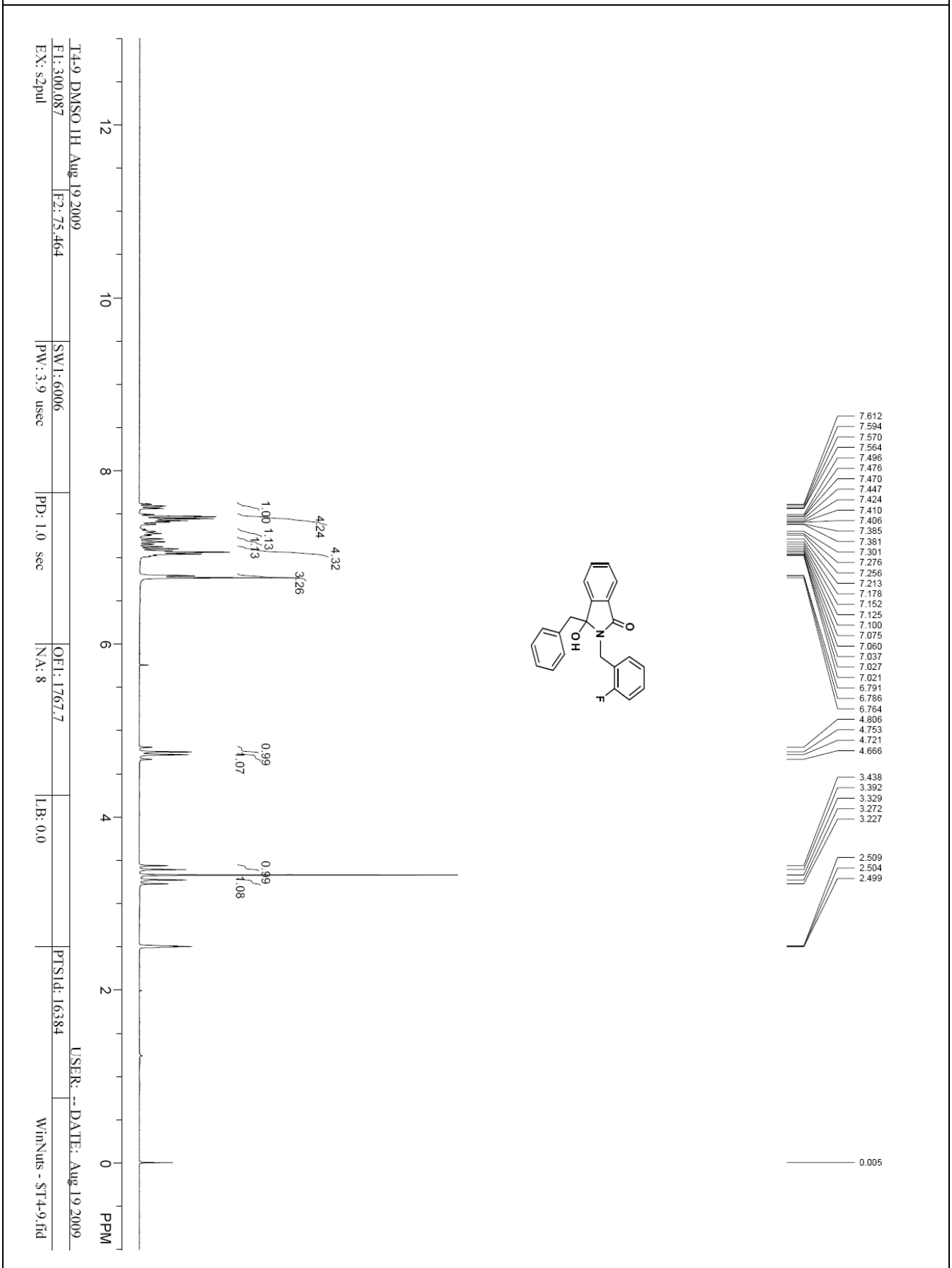
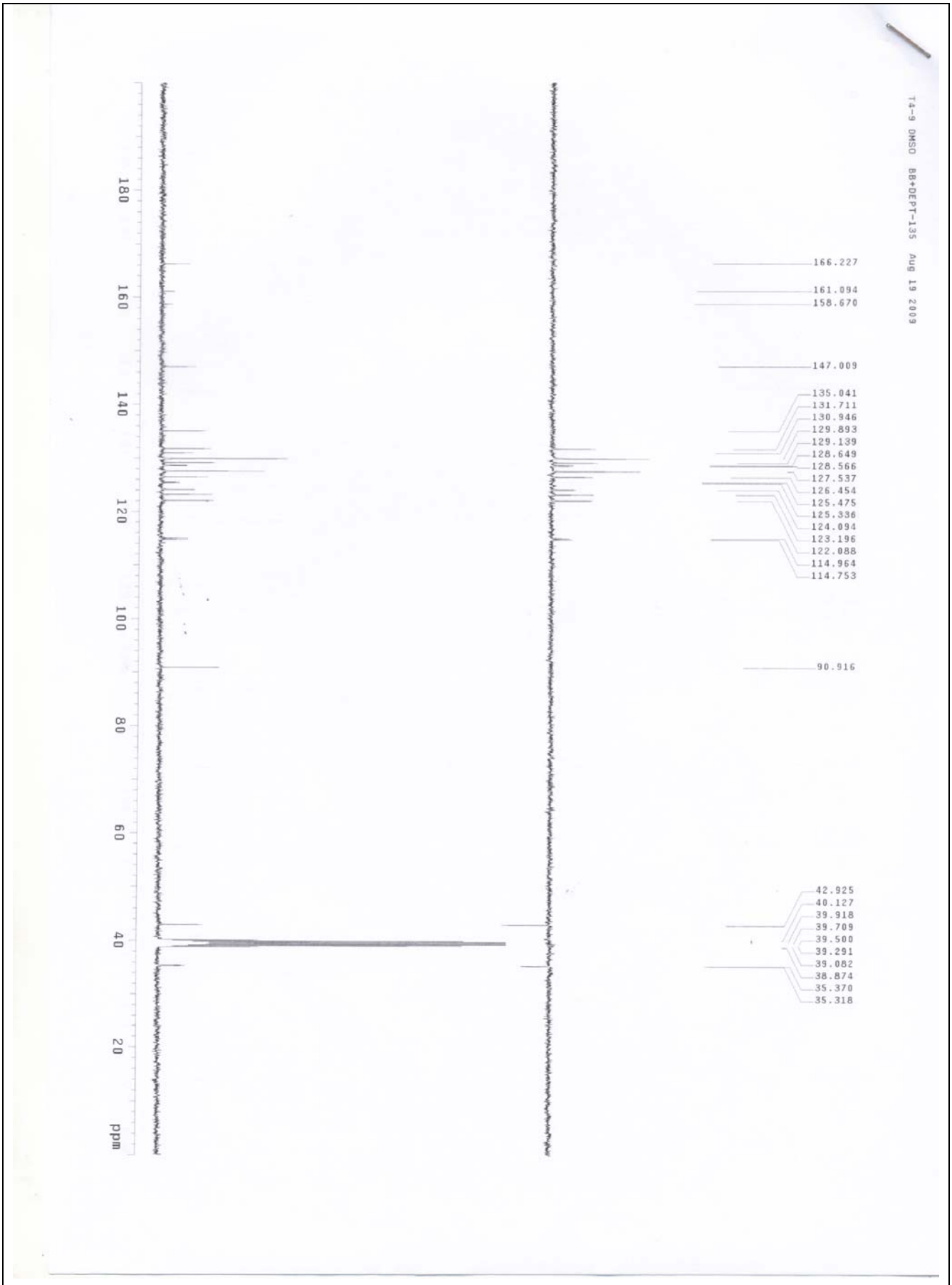
167.049
163.264
160.823
145.988
134.392
131.869
130.949
130.280
130.225
130.143
129.719
127.957
127.060
123.320
122.965
115.423
115.209
91.365
77.319
77.000
76.681
43.610
41.870
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm

3-benzyl-2-(2-fluorobenzyl)-3-hydroxyisoindolin-1-one (9Ag)

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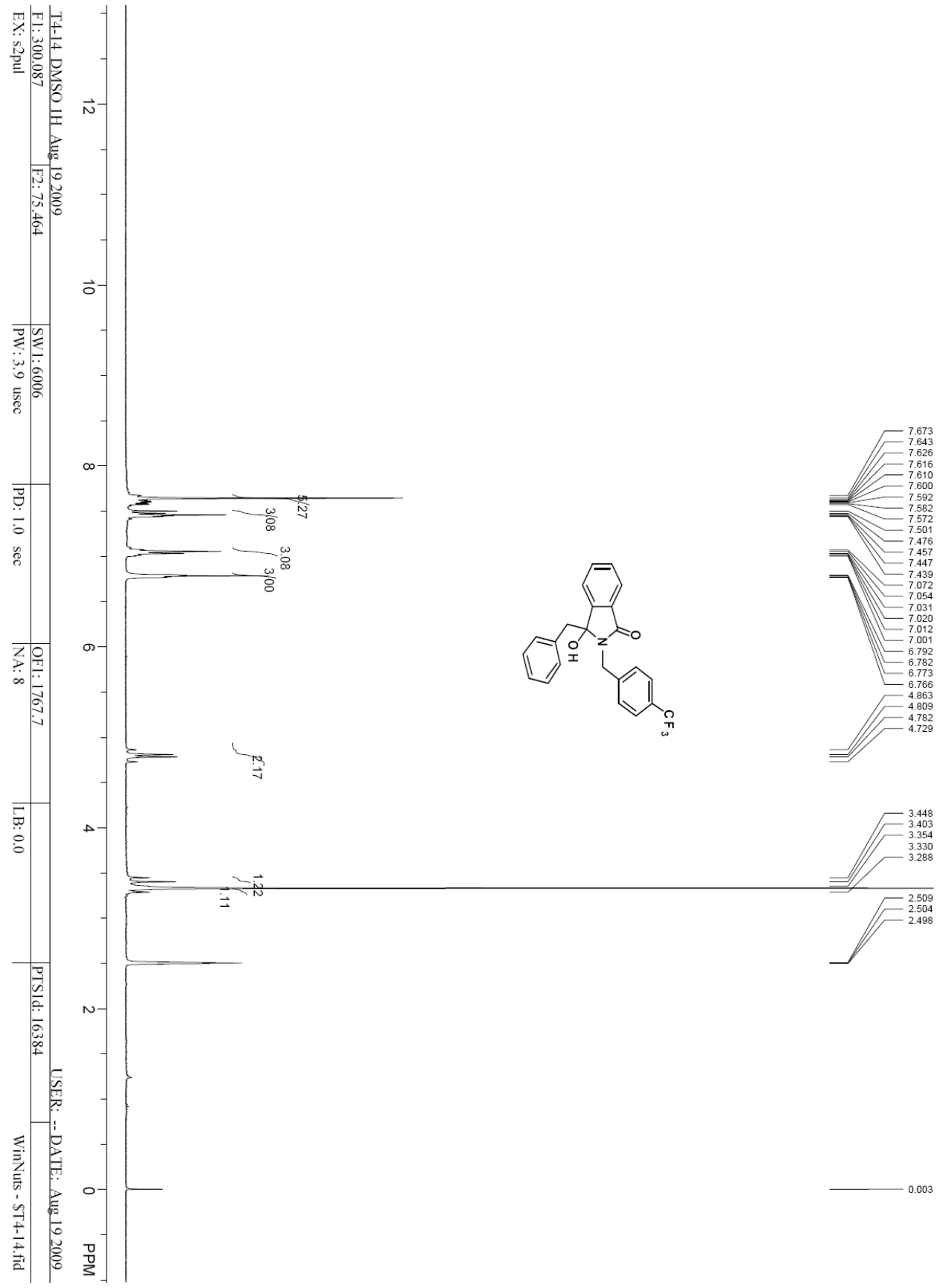
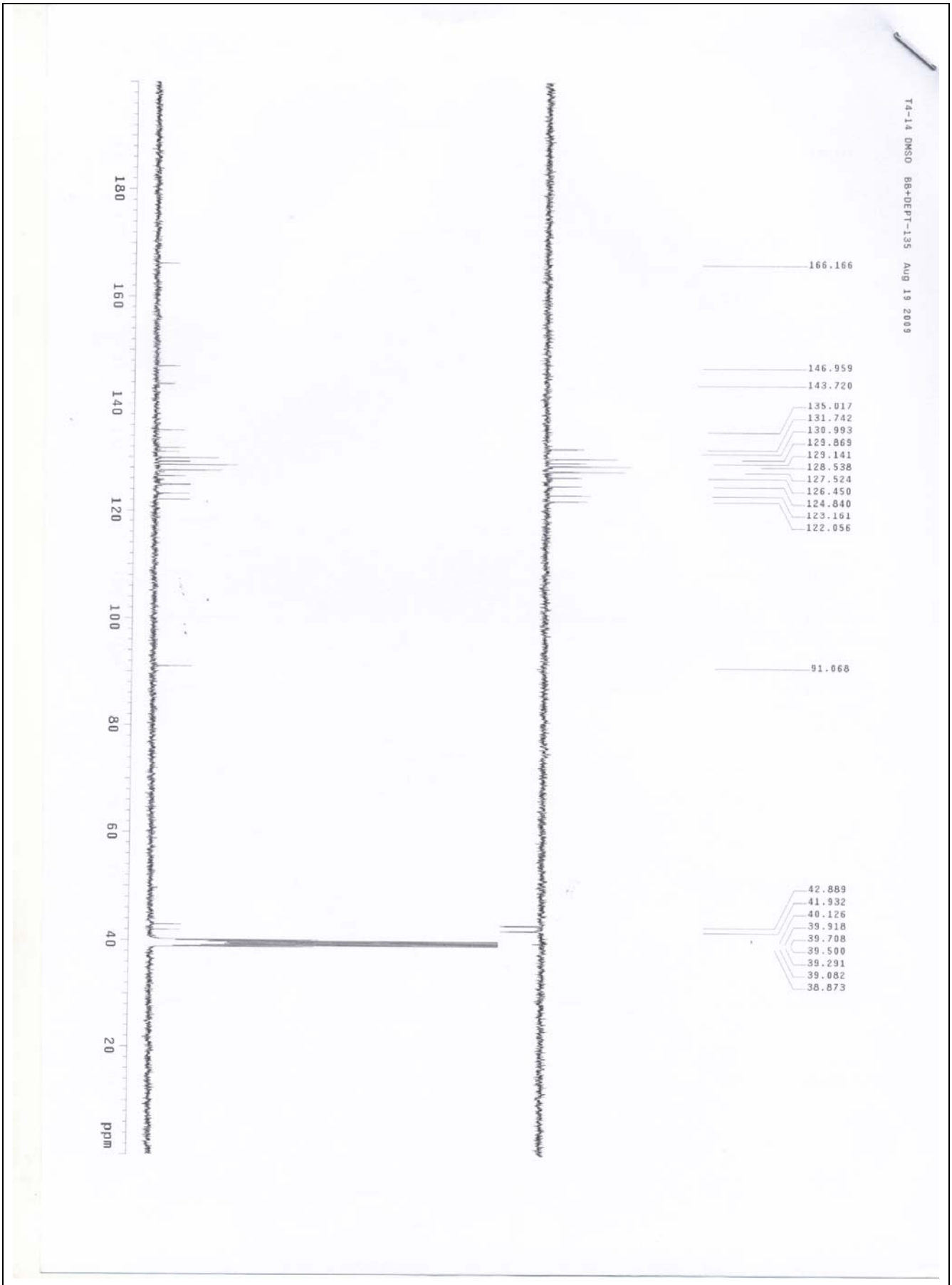
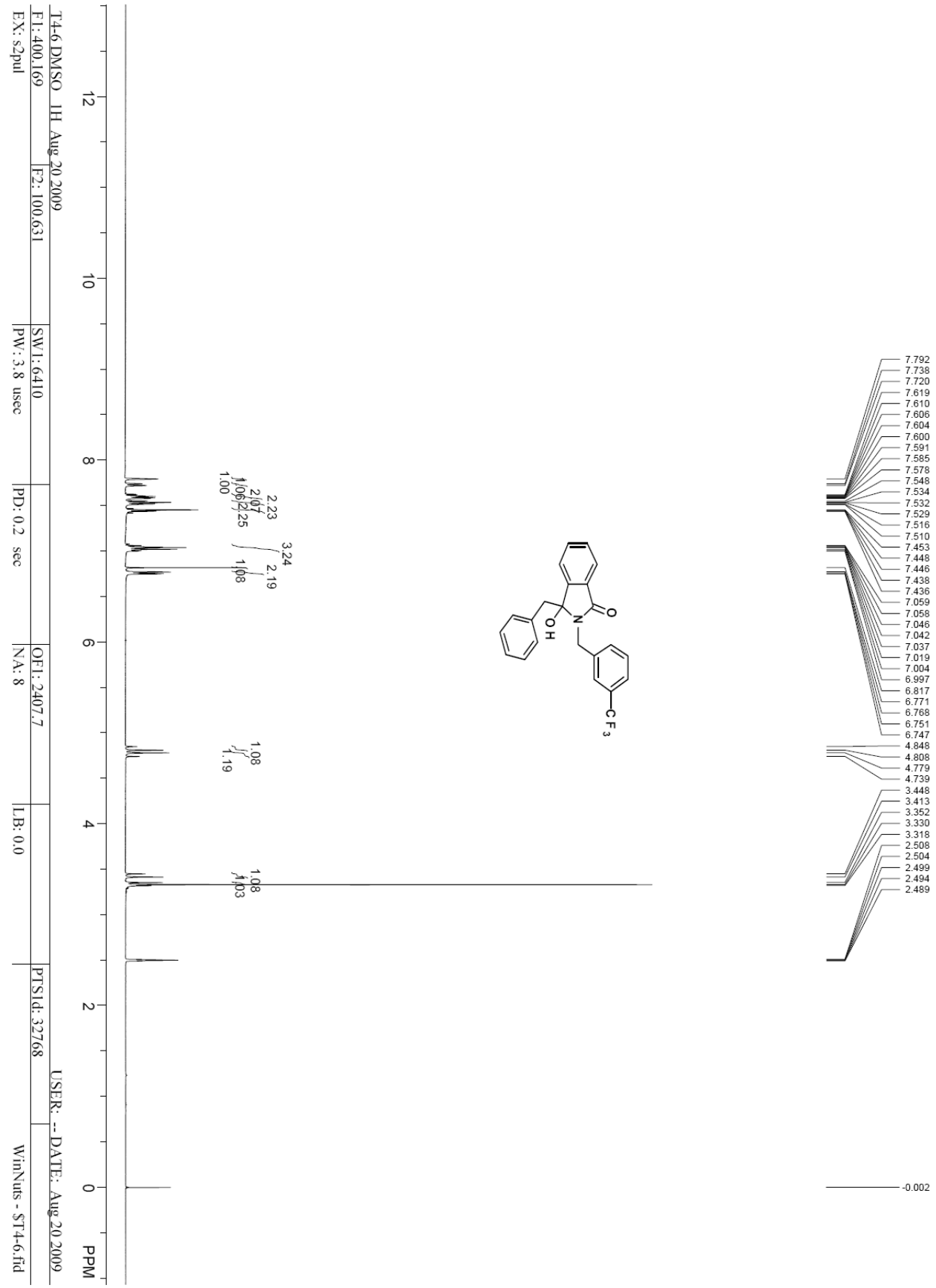
161.094
158.670
147.009
135.041
131.711
130.946
129.893
129.139
128.649
128.566
127.537
126.454
125.475
125.336
124.094
123.196
122.088
114.964
114.753
90.916
42.925
40.127
39.918
39.709
39.500
39.291
39.082
38.874
35.370
35.318
180
160
140
120
100
80
60
40
20
ppm

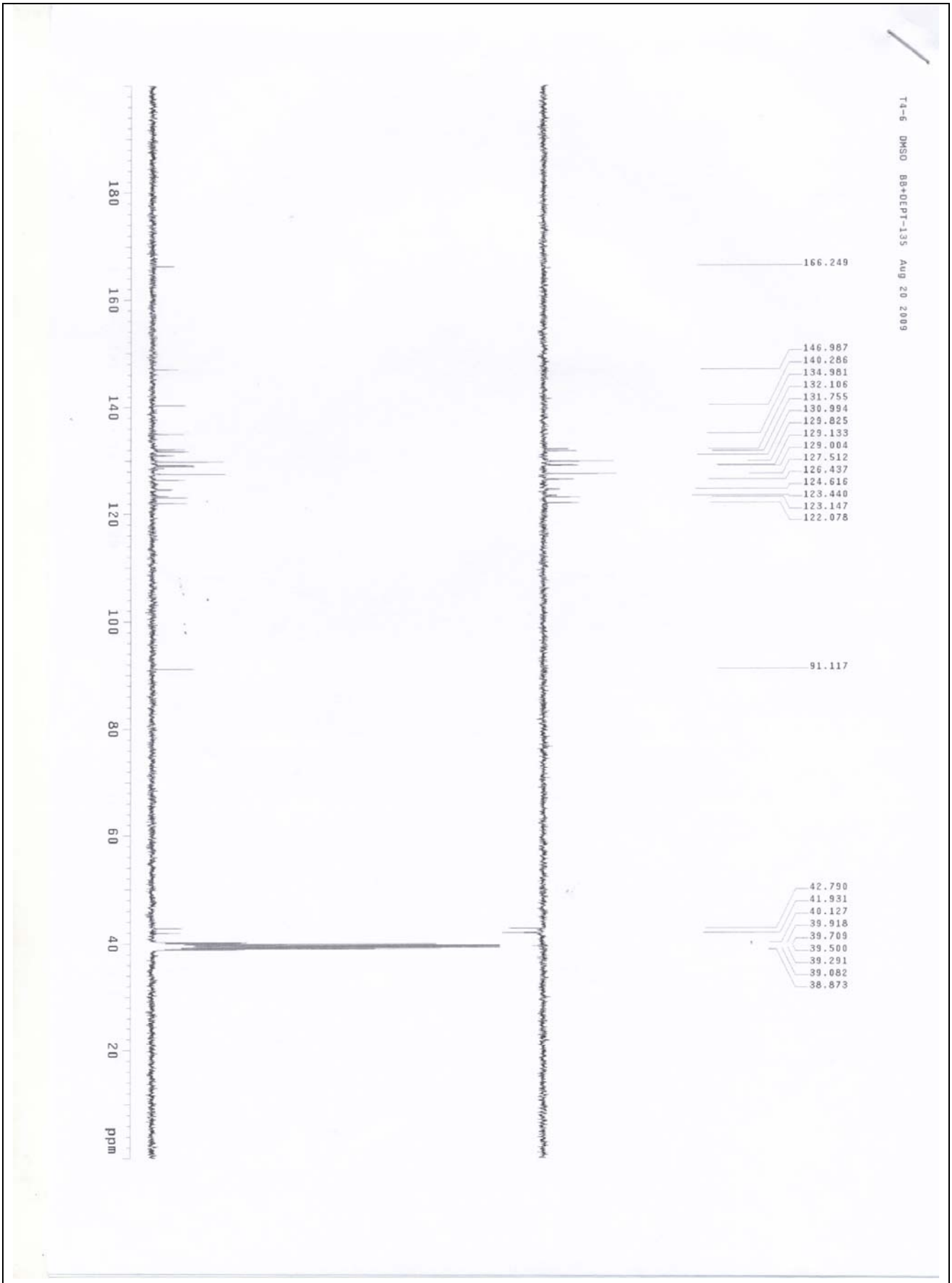
3-benzyl-3-hydroxy-2-(4-(trifluoromethyl)benzyl)isoindolin-1-one (9Ah)



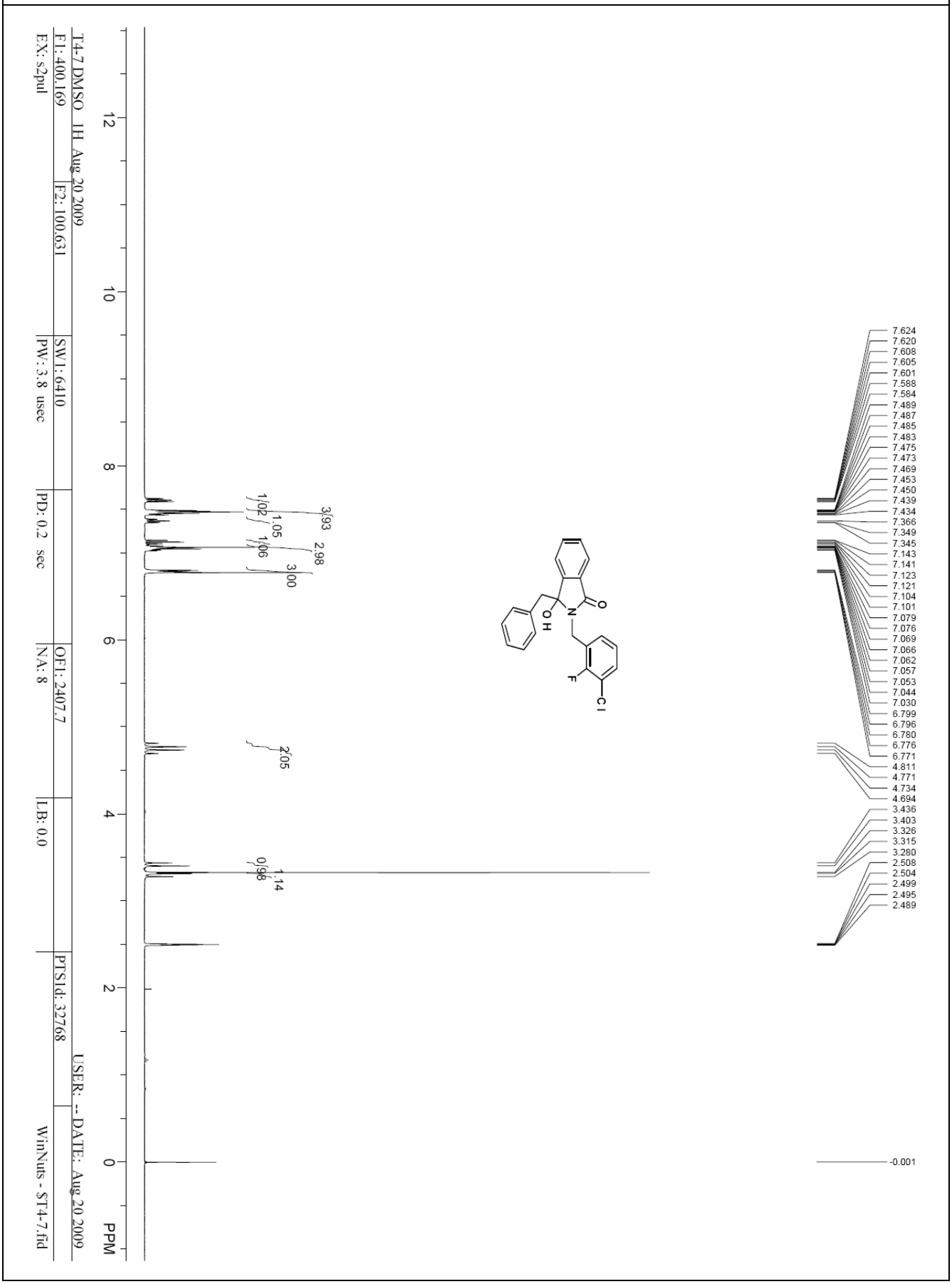

T4-14 DMSO BB+DEPT-135 Aug 19 2009
166.166
146.959
143.720
135.017
131.742
130.993
129.869
129.141
128.538
127.524
126.450
124.840
123.161
122.056
91.068
42.889
41.932
40.126
39.918
39.708
39.500
39.291
39.082
38.873
180
160
140
120
100
80
60
40
20
ppm

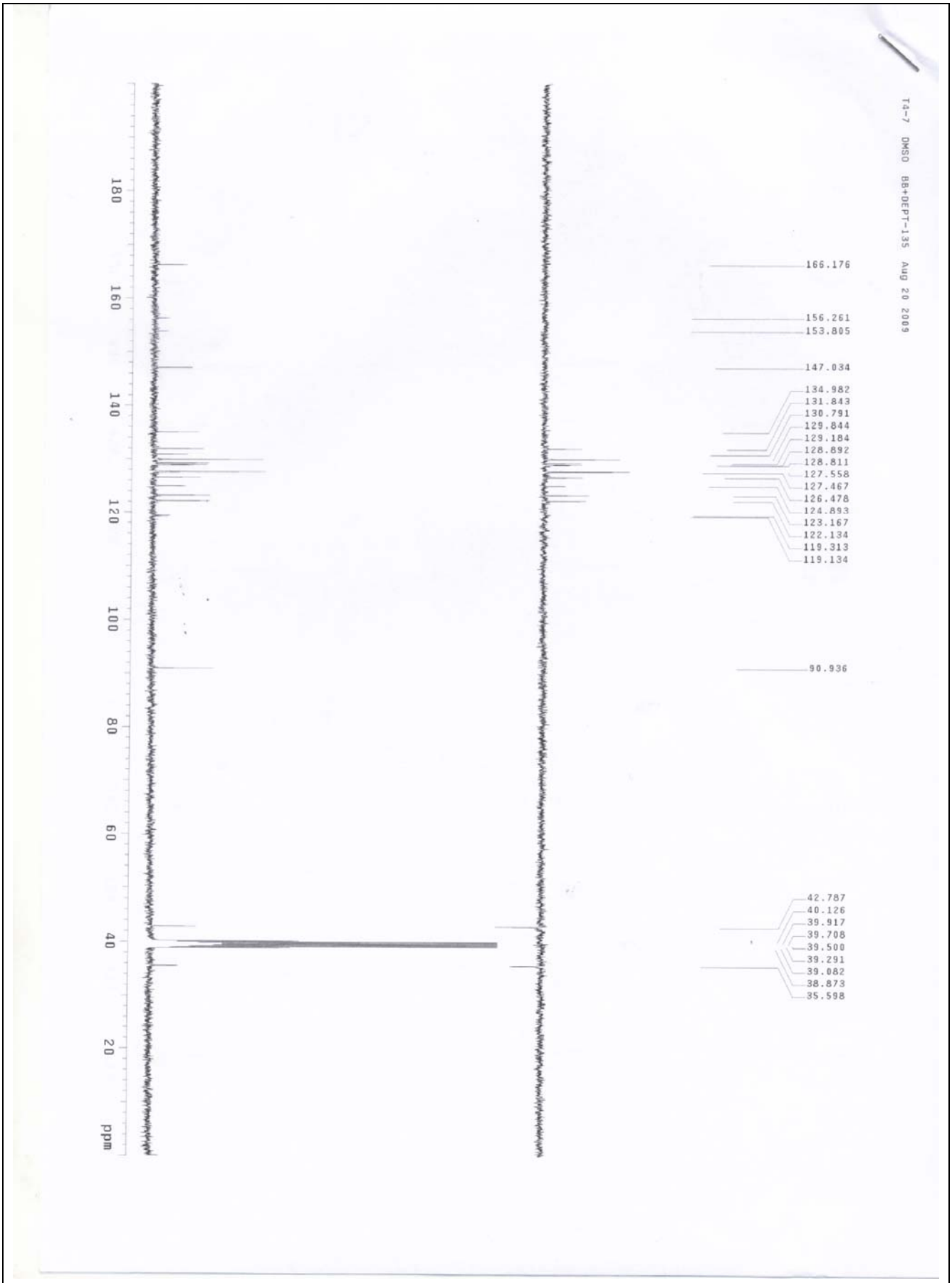
3-benzyl-3-hydroxy-2-(3-(trifluoromethyl)benzyl)isoindolin-1-one (9Ai)



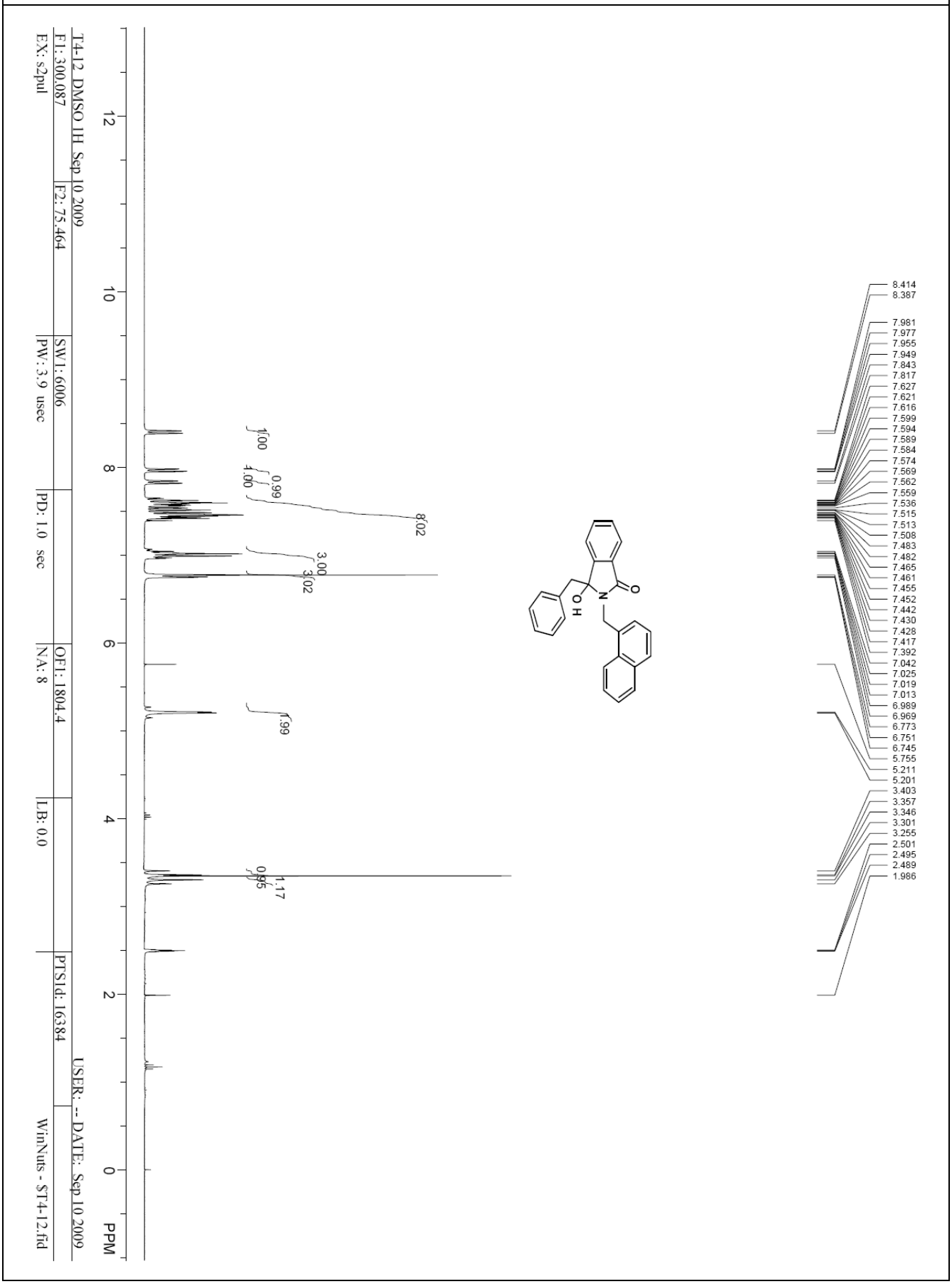

T4-6 DMSO BB+DEPT-135 Aug 20 2009
166.249
146.987
140.286
134.981
132.106
131.755
130.994
129.825
129.133
129.004
127.512
126.437
124.616
123.440
123.147
122.078
91.117
42.790
41.931
40.127
39.918
39.709
39.500
39.291
39.082
38.873
180
160
140
120
100
80
60
40
20
ppm

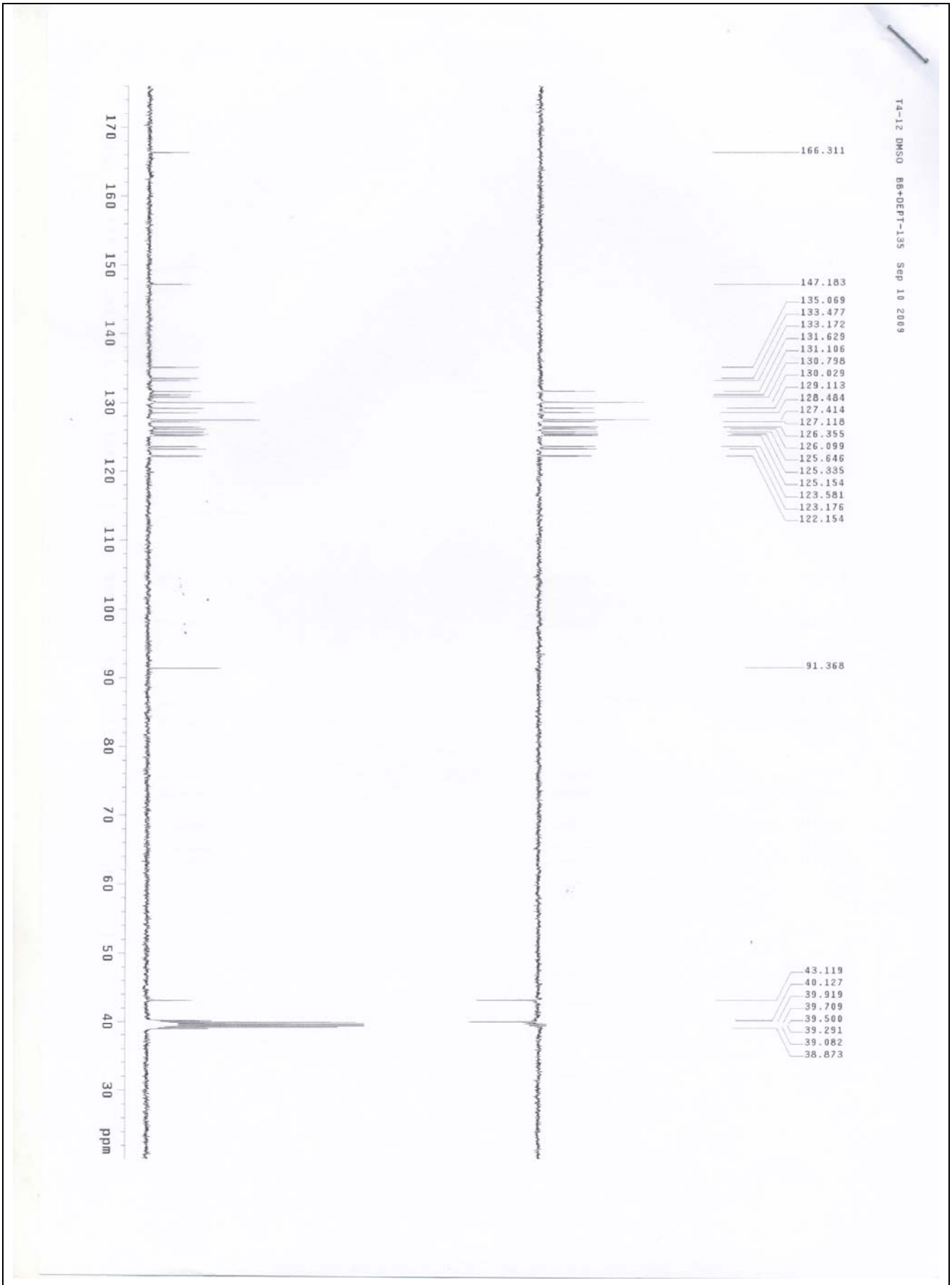
3-benzyl-2-(3-chloro-2-fluorobenzyl)-3-hydroxyisoindolin-1-one (9Aj)



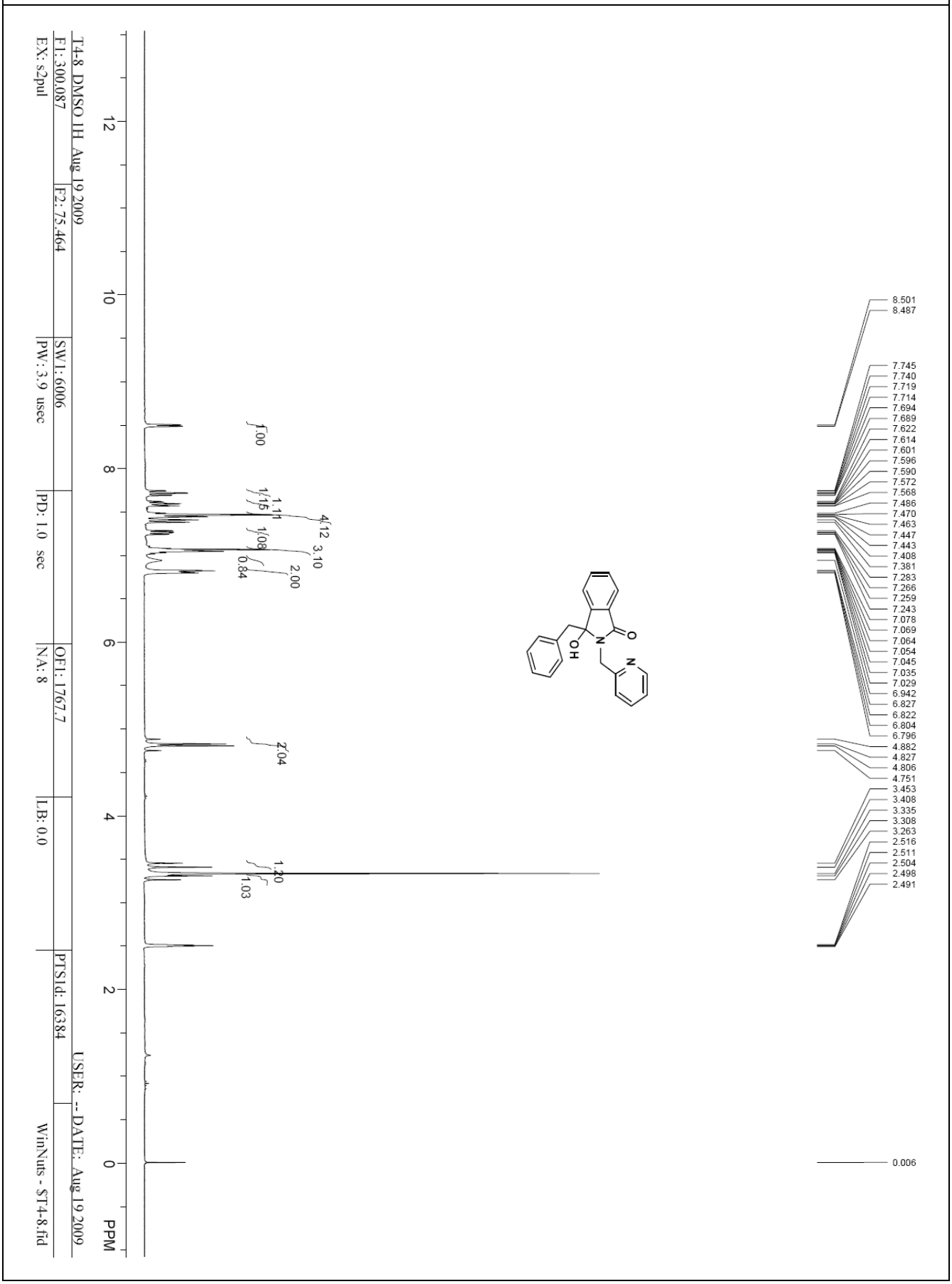

T4-7 DMSO BB+DEPT-135 Aug 20 2009
166.176
156.261
153.805
147.034
134.982
131.843
130.791
129.844
129.184
128.892
128.811
127.558
127.467
126.478
124.893
123.167
122.134
119.313
119.134
90.936
42.787
40.126
39.917
39.708
39.500
39.291
39.082
38.873
35.598
180
160
140
120
100
80
60
40
20
ppm

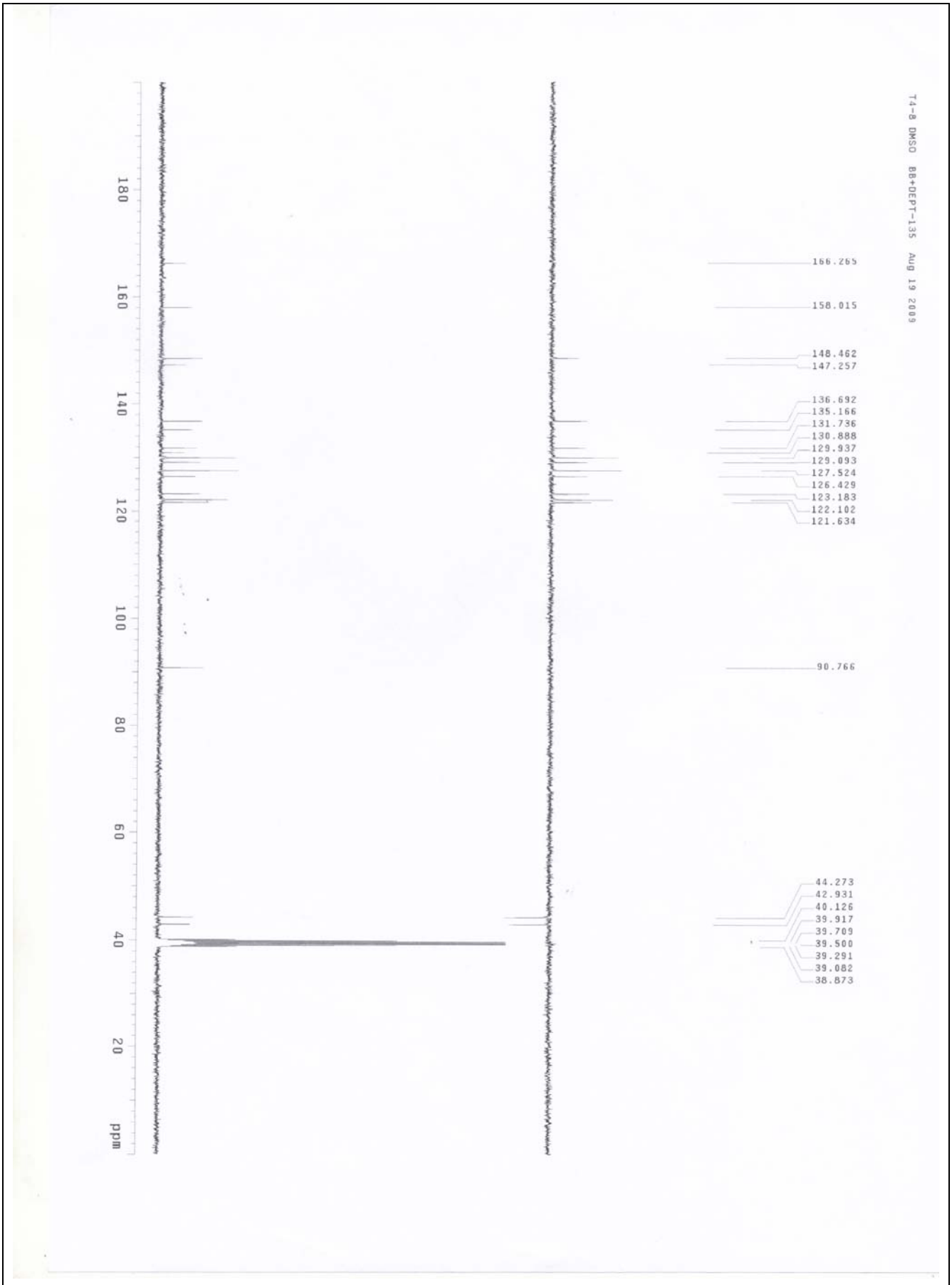
3-benzyl-3-hydroxy-2-(naphthalen-1-ylmethyl)isoindolin-1-one (9Ak)



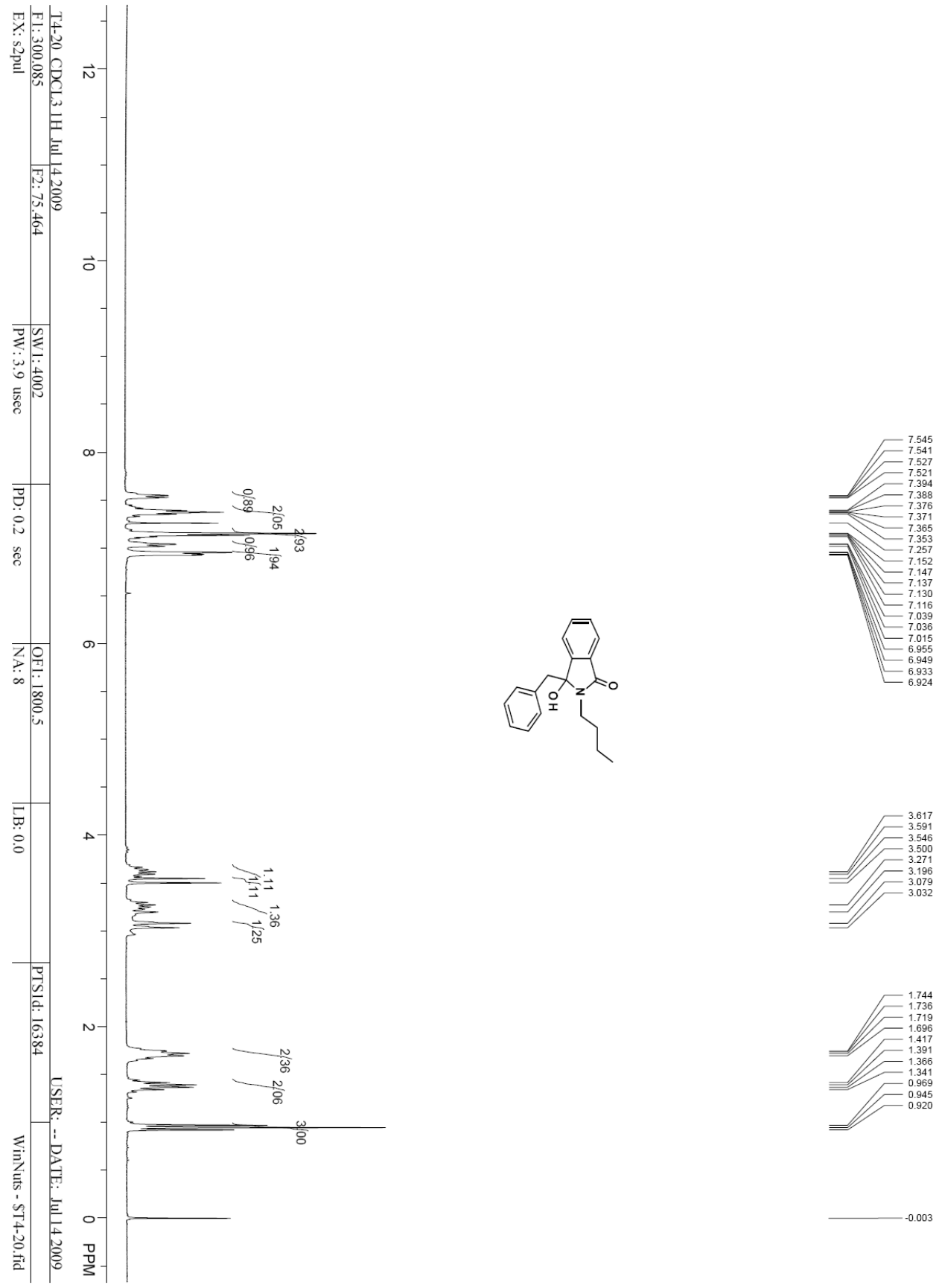

T4-12 DMSO BB+DEPT-135 Sep 10 2009
166.311
147.183
135.069
133.477
133.172
131.629
131.106
130.798
130.029
129.113
128.484
127.414
127.118
126.355
126.099
125.646
125.335
125.154
123.581
123.176
122.154
91.368
43.119
40.127
39.919
39.709
39.500
39.291
39.082
38.873
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

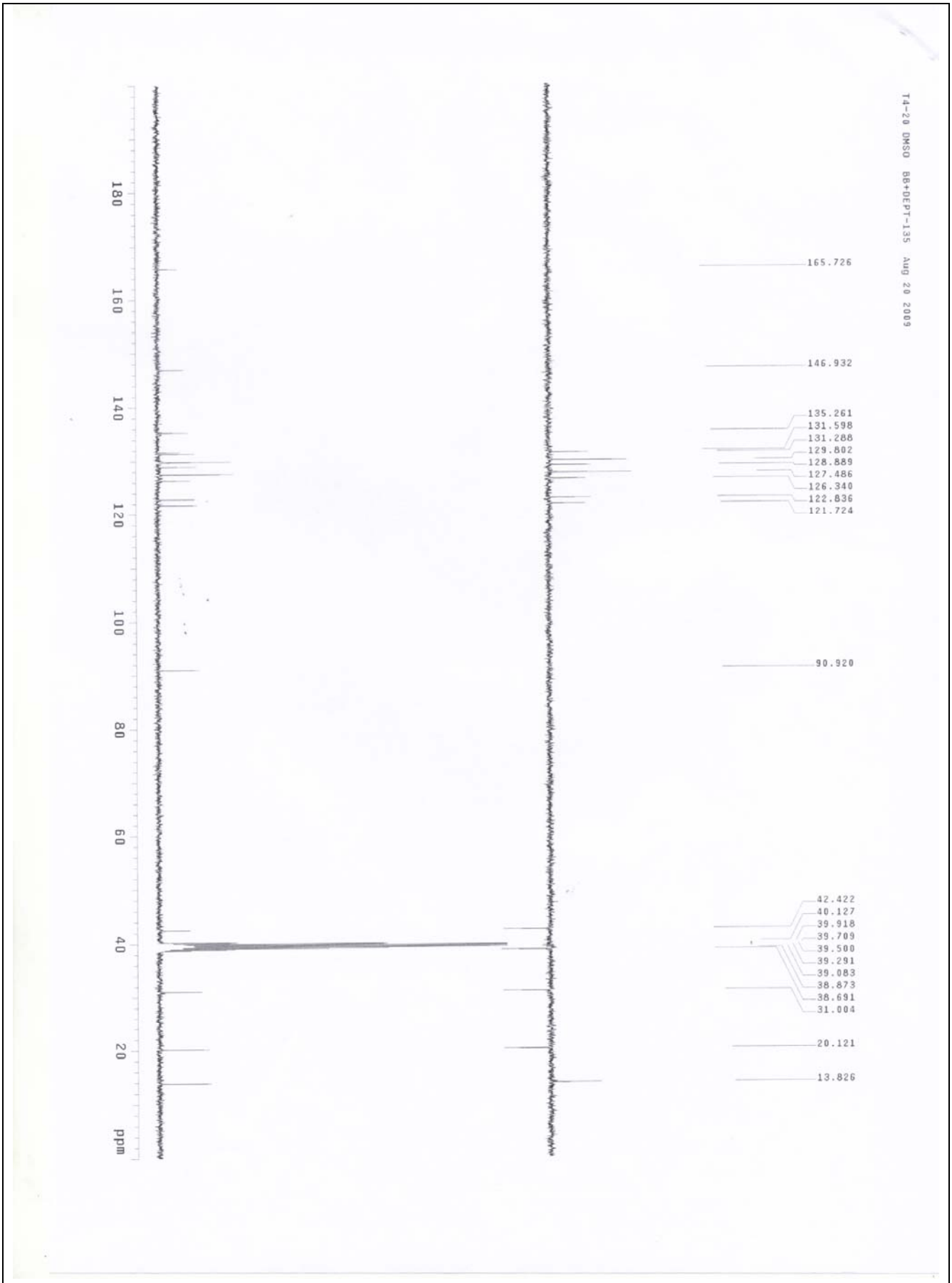
3-benzyl-3-hydroxy-2-(pyridin-2-ylmethyl)isoindolin-1-one (9Al)



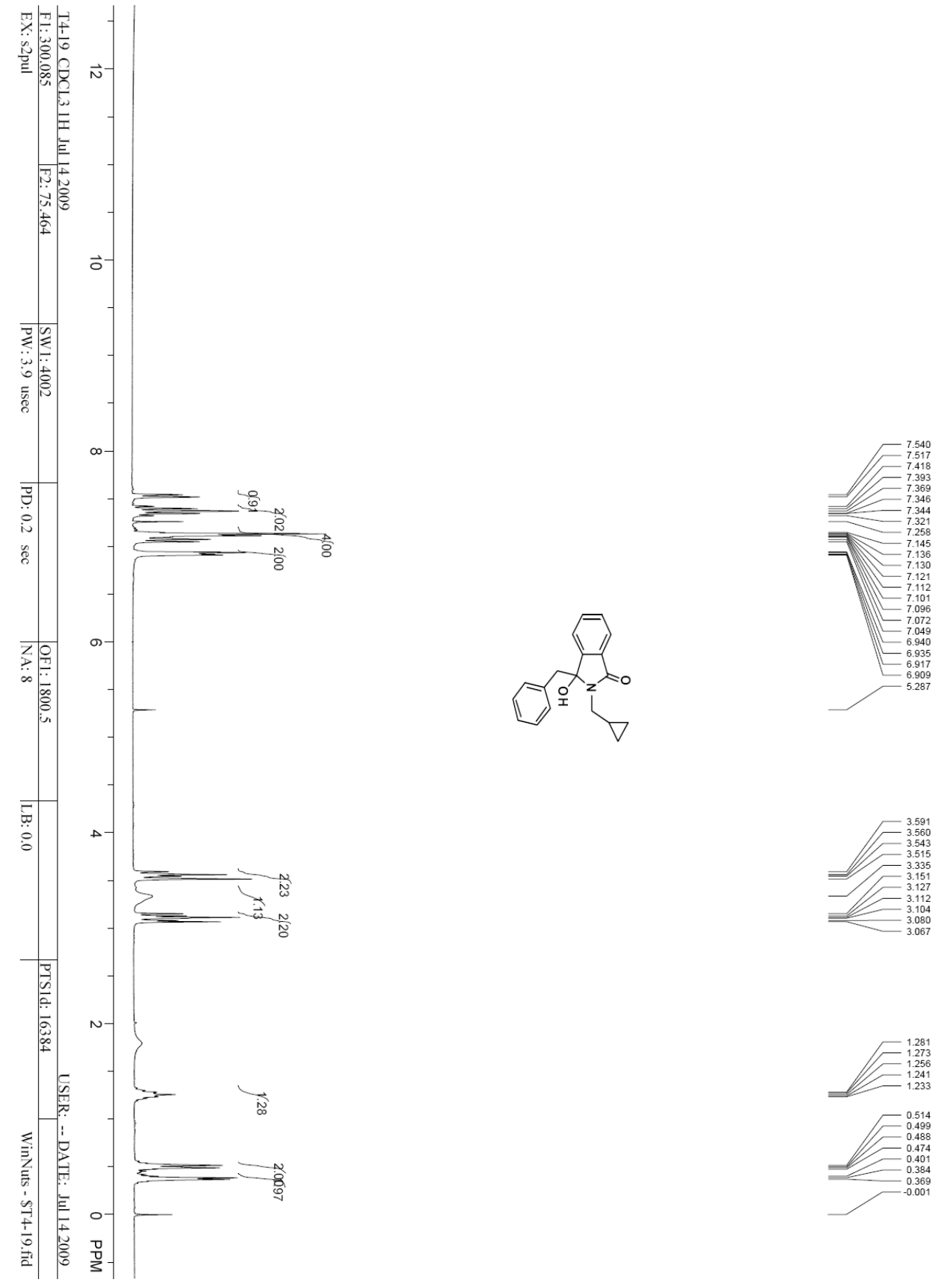

T4-8 DMSO BB+DEPT-135 Aug 19 2009
166.265
158.015
148.462
147.257
136.692
135.166
131.736
130.888
129.937
129.093
127.524
126.429
123.183
122.102
121.634
90.766
44.273
42.931
40.126
39.917
39.709
39.500
39.291
39.082
38.873
180
160
140
120
100
80
60
40
20
ppm

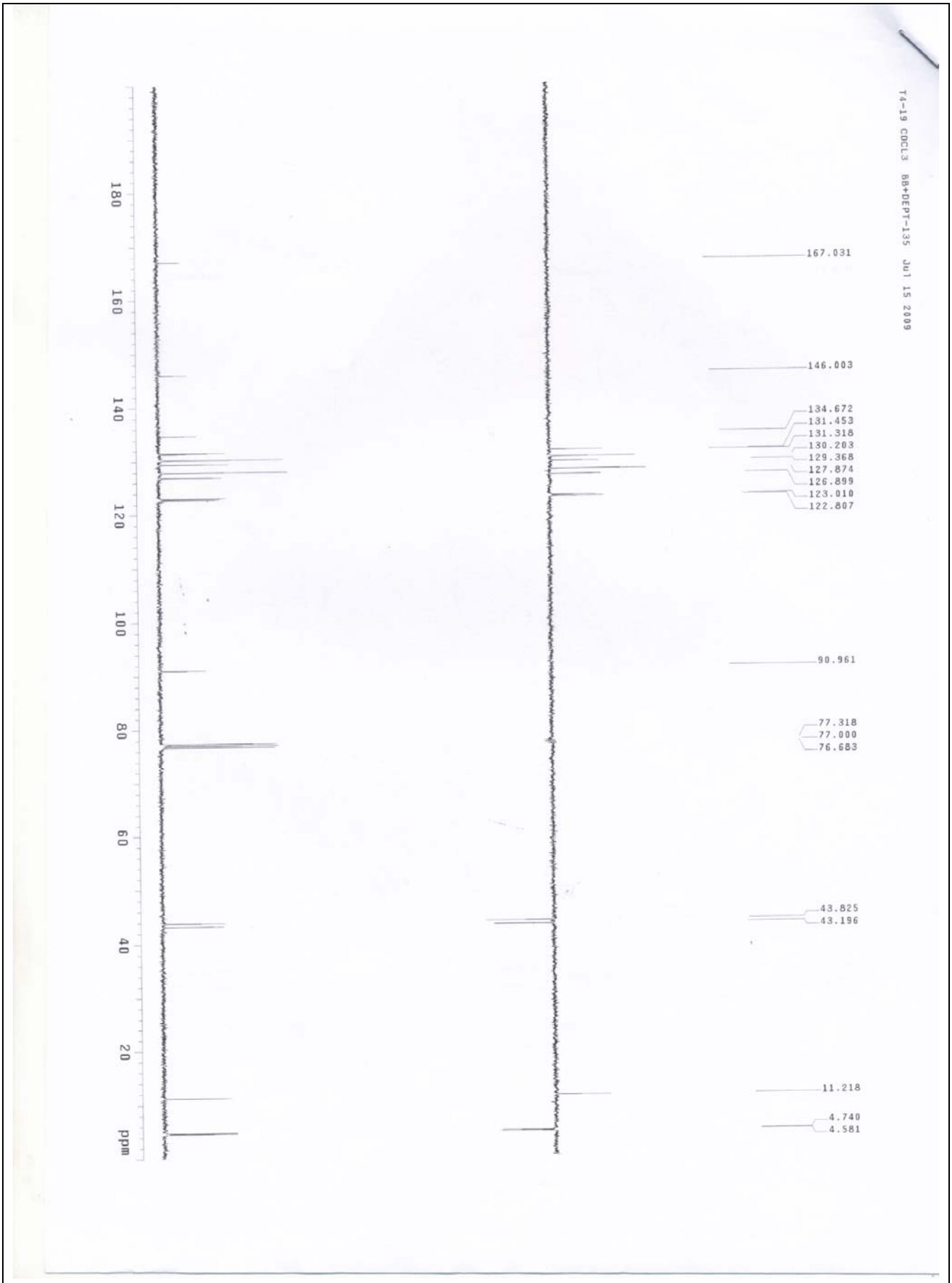
3-benzyl-2-butyl-3-hydroxyisoindolin-1-one (9Am)



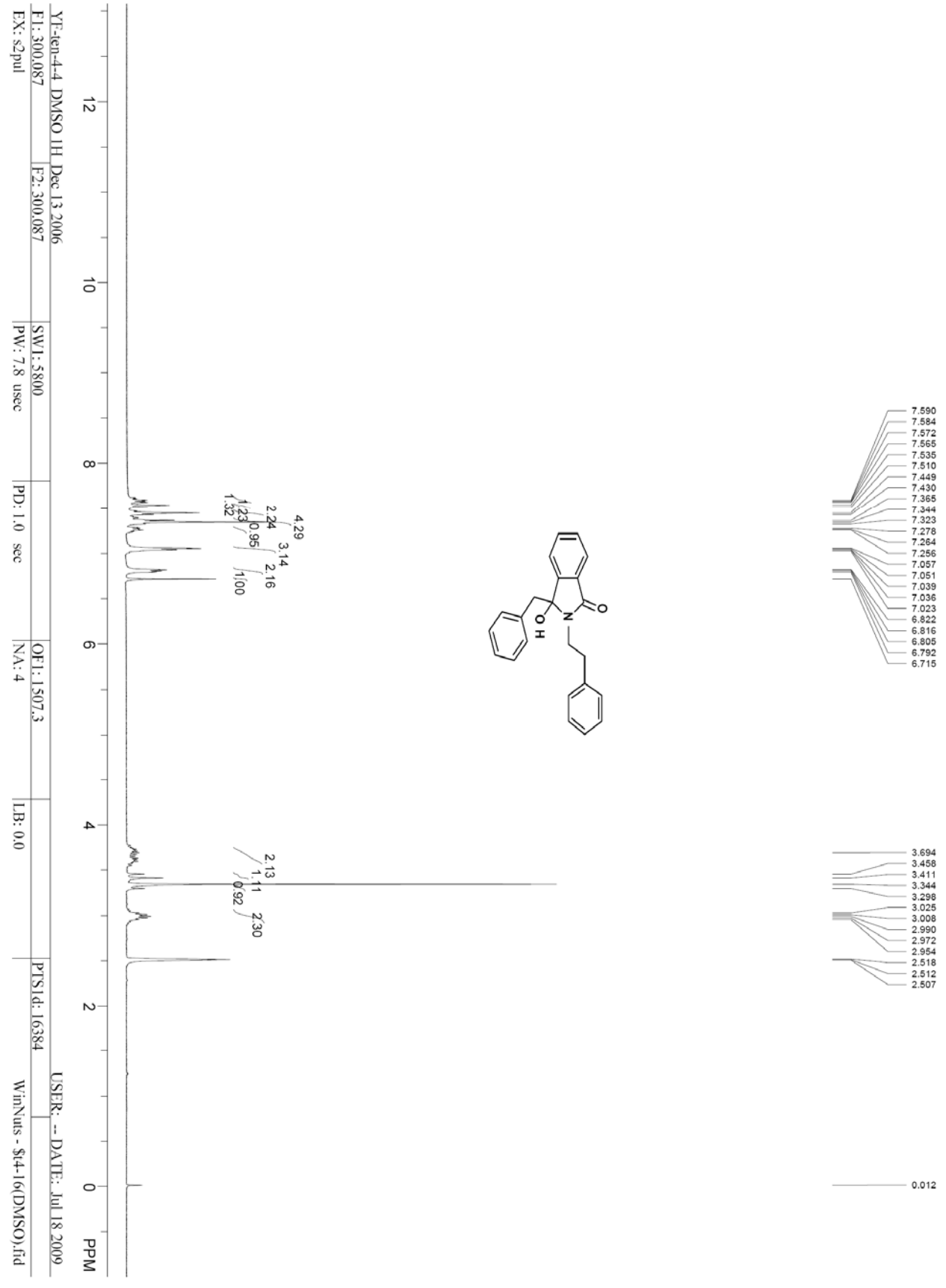

T4-20 DMSO BB+DEPT-135 Aug 20 2009
165.726
146.932
135.261
131.598
131.288
129.802
128.889
127.486
126.340
122.836
121.724
90.920
42.422
40.127
39.918
39.709
39.500
39.291
39.083
38.873
38.691
31.004
20.121
13.826
180
160
140
120
100
80
60
40
20
ppm

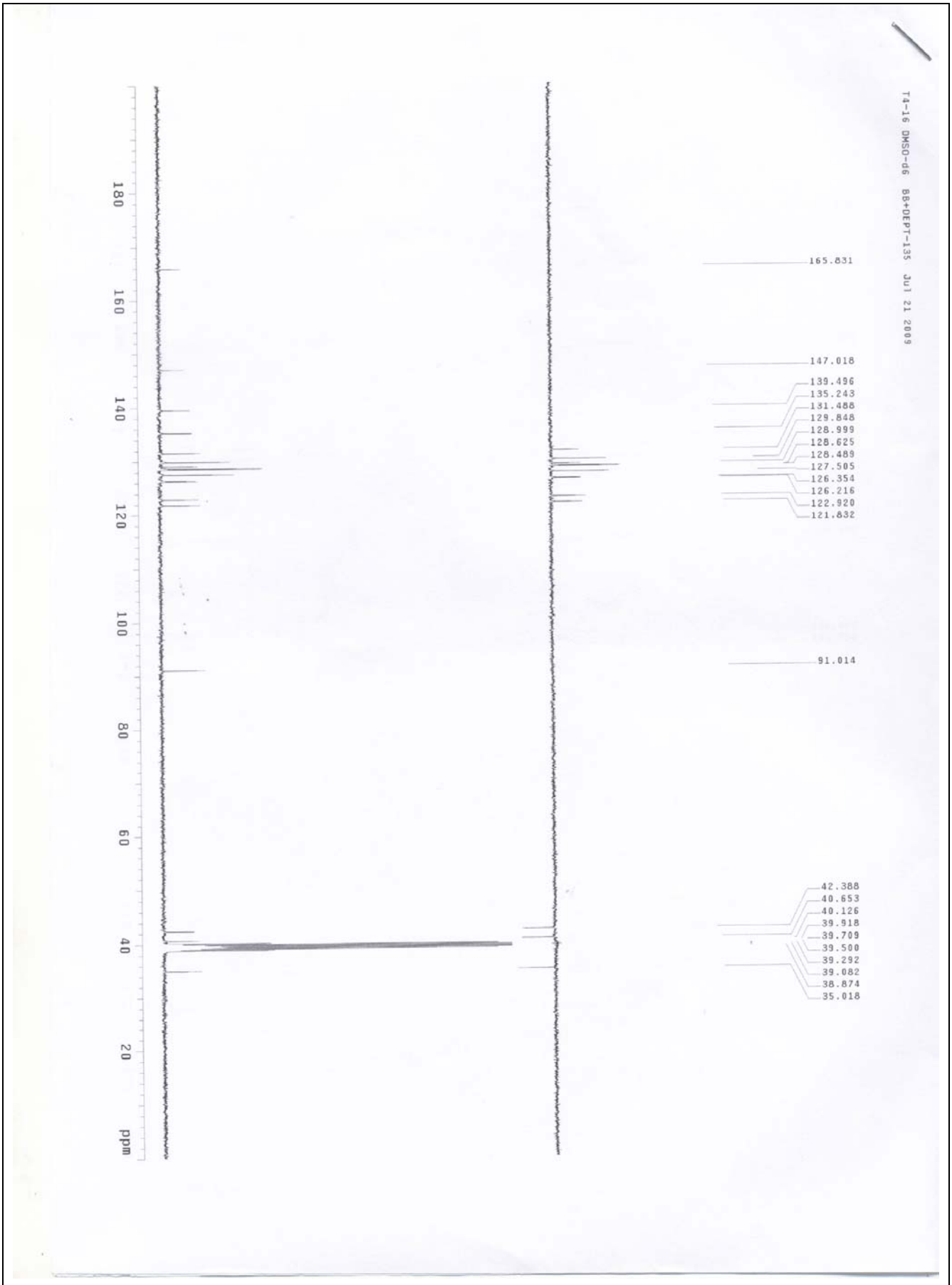
3-benzyl-2-(cyclopropylmethyl)-3-hydroxyisoindolin-1-one (9An)



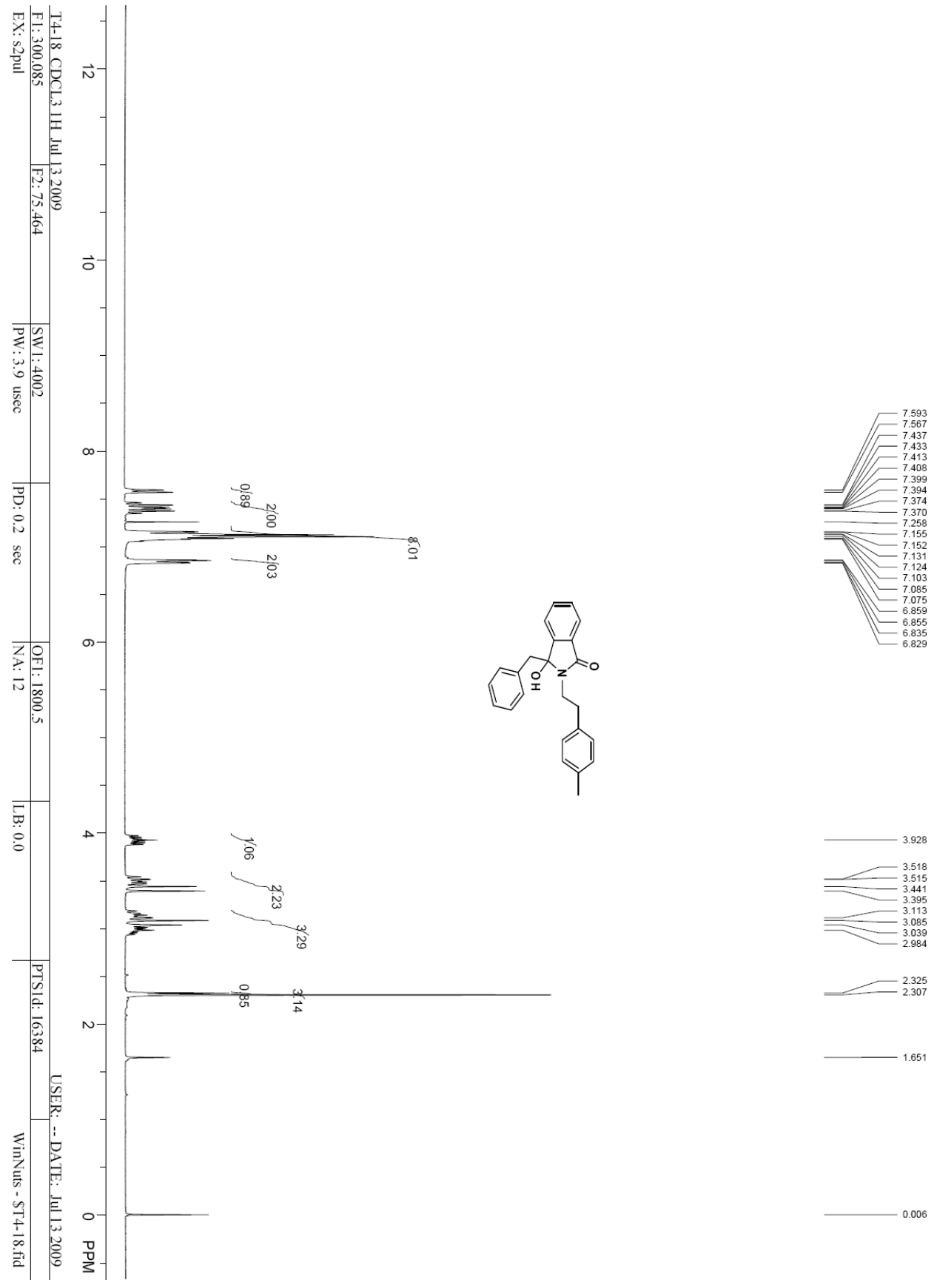

T4-19 CDCL3 BB+DEPT-135 Jul 15 2009
167.031
146.003
134.672
131.453
131.318
130.203
129.368
127.874
126.899
123.010
122.807
90.961
77.318
77.000
76.683
43.825
43.196
11.218
4.740
4.581
180
160
140
120
100
80
60
40
20
ppm

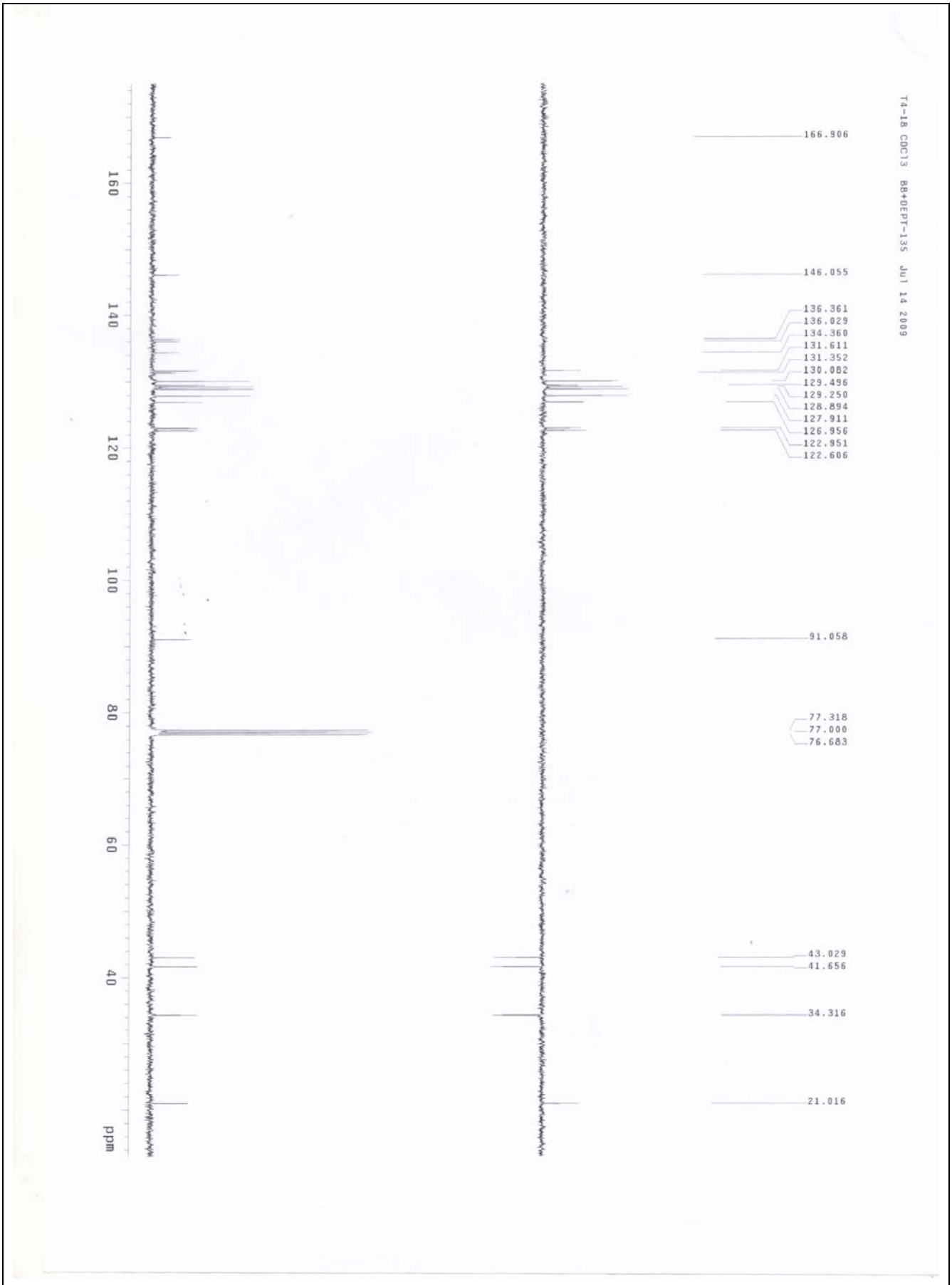
3-benzyl-3-hydroxy-2-phenethylisoindolin-1-one (9Ao)



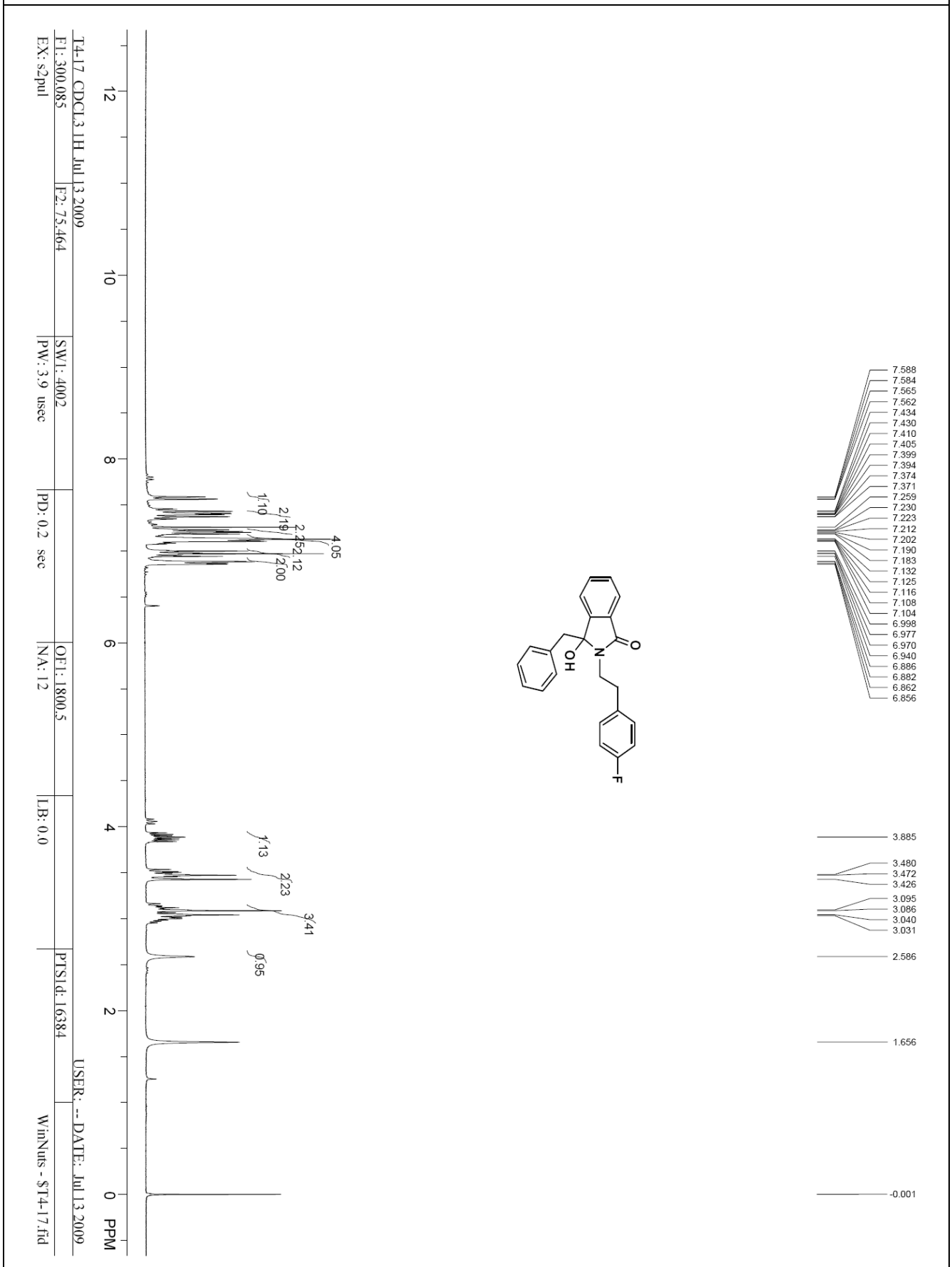

T4-16 DMSO-d6 BB+DEPT-135 Jul 21 2009
165.831
147.018
139.496
135.243
131.488
129.848
128.999
128.625
128.489
127.505
126.354
126.216
122.920
121.832
91.014
42.388
40.653
40.126
39.918
39.709
39.500
39.292
39.082
38.874
35.018
180
160
140
120
100
80
60
40
20
ppm

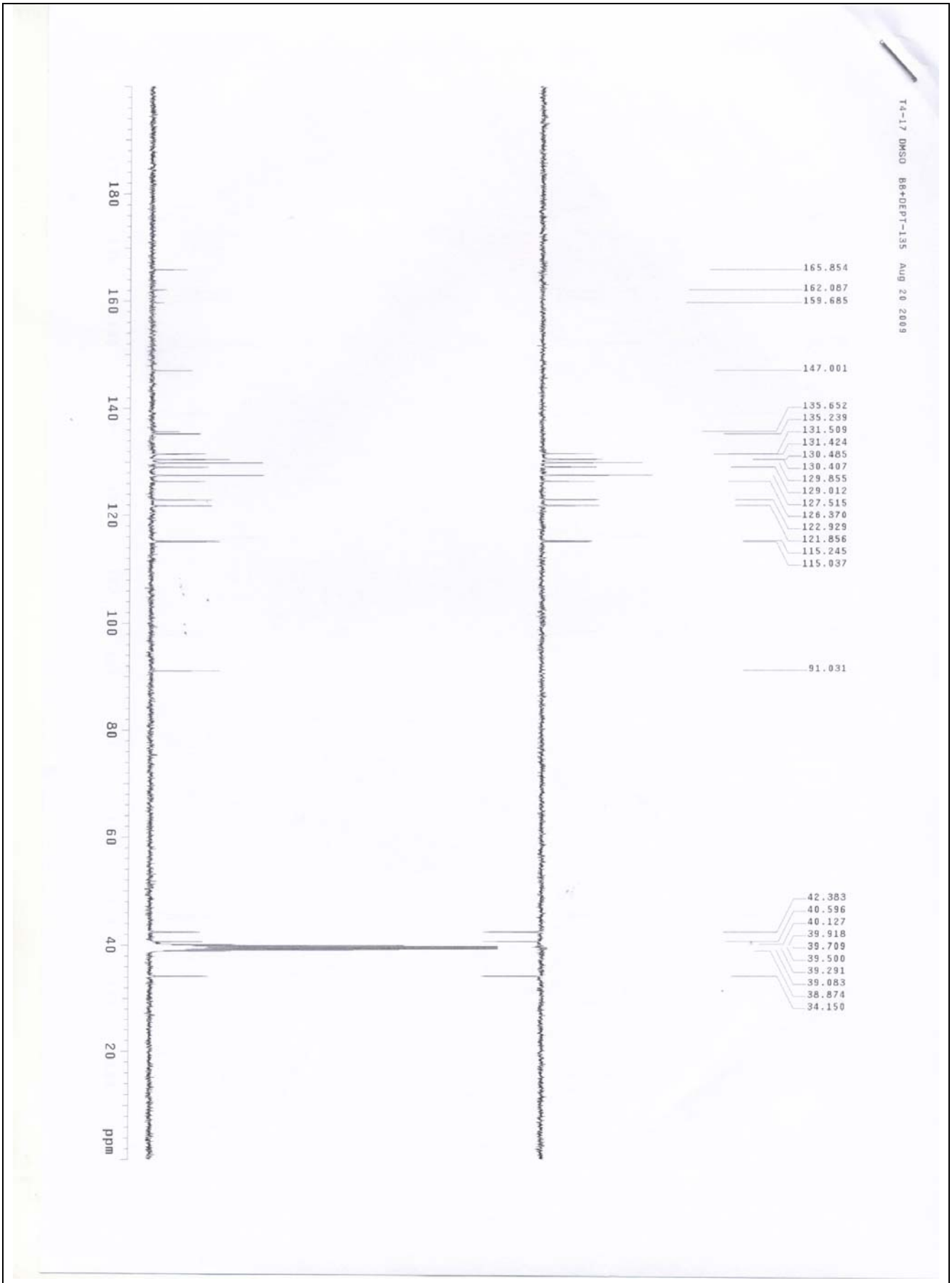
3-benzyl-3-hydroxy-2-(4-methylphenethyl)isoindolin-1-one (9Ap)



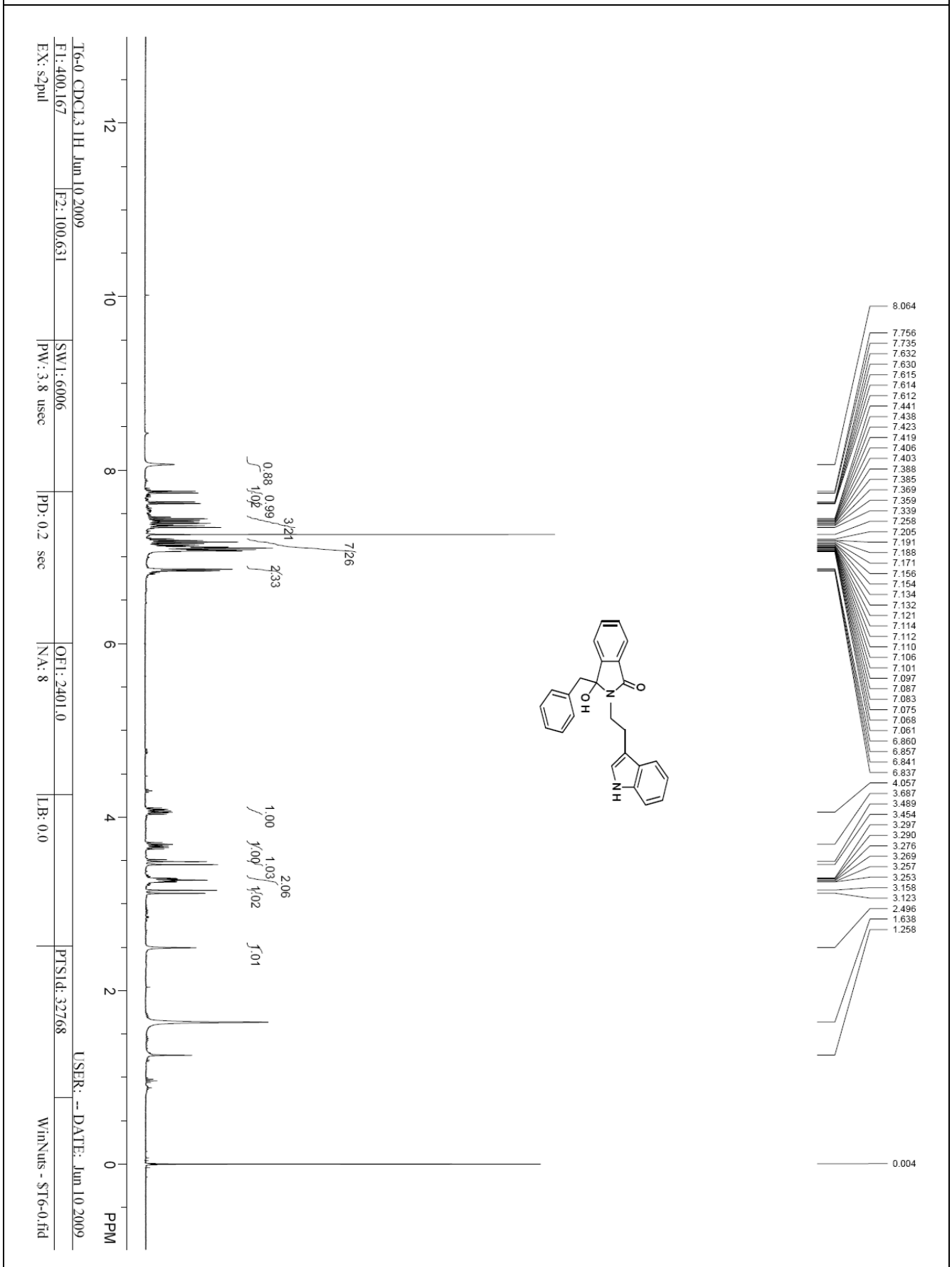

T4-18 CDCl3 BB+DEPT-135 Jul 14 2009
166.906
146.055
136.361
136.029
134.360
131.611
131.352
130.082
129.496
129.250
128.894
127.911
126.956
122.951
122.606
91.058
77.318
77.000
76.683
43.029
41.656
34.316
21.016
160
140
120
100
80
60
40
ppm

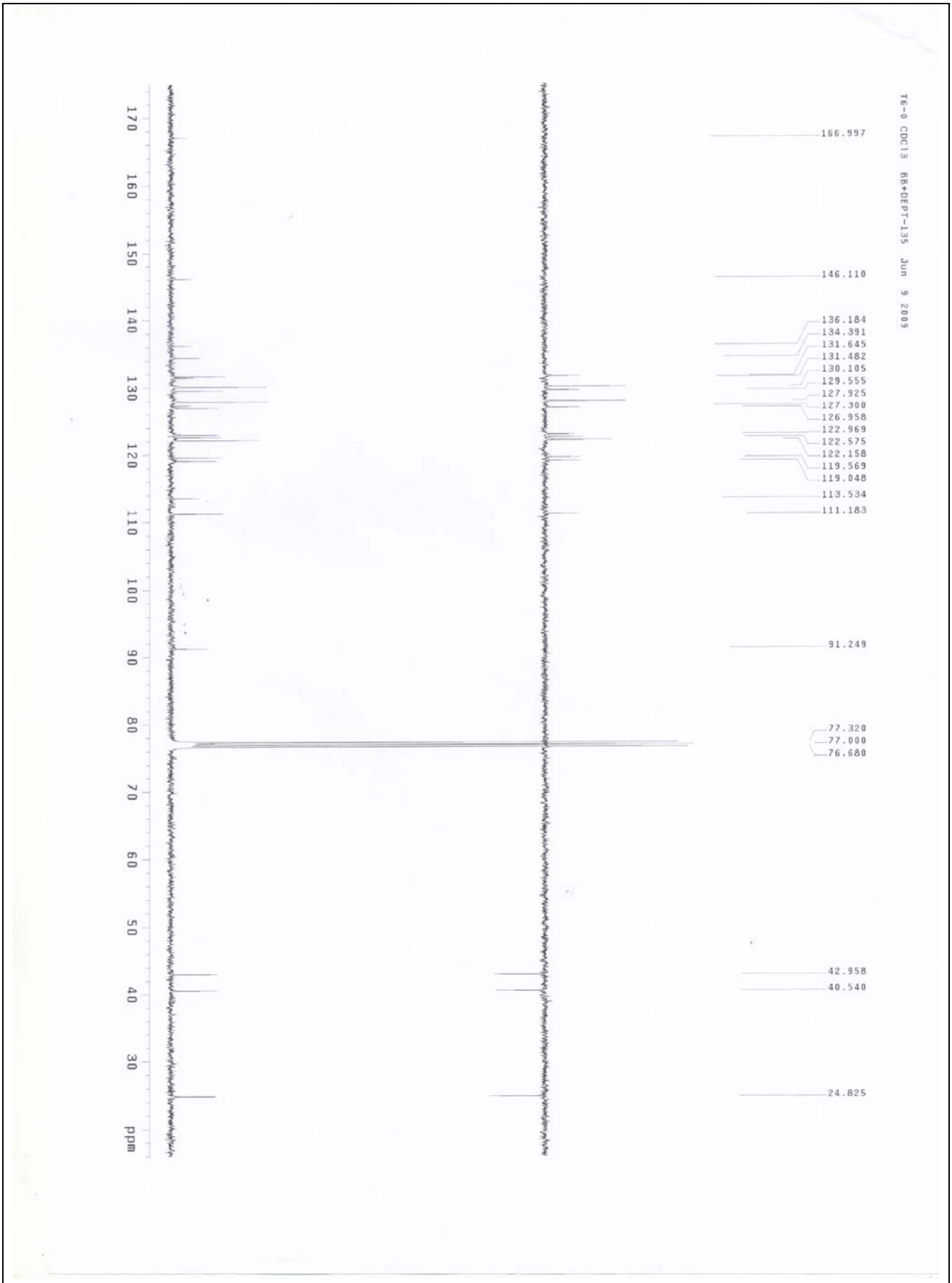
3-benzyl-2-(4-fluorophenethyl)-3-hydroxyisoindolin-1-one (9Aq)



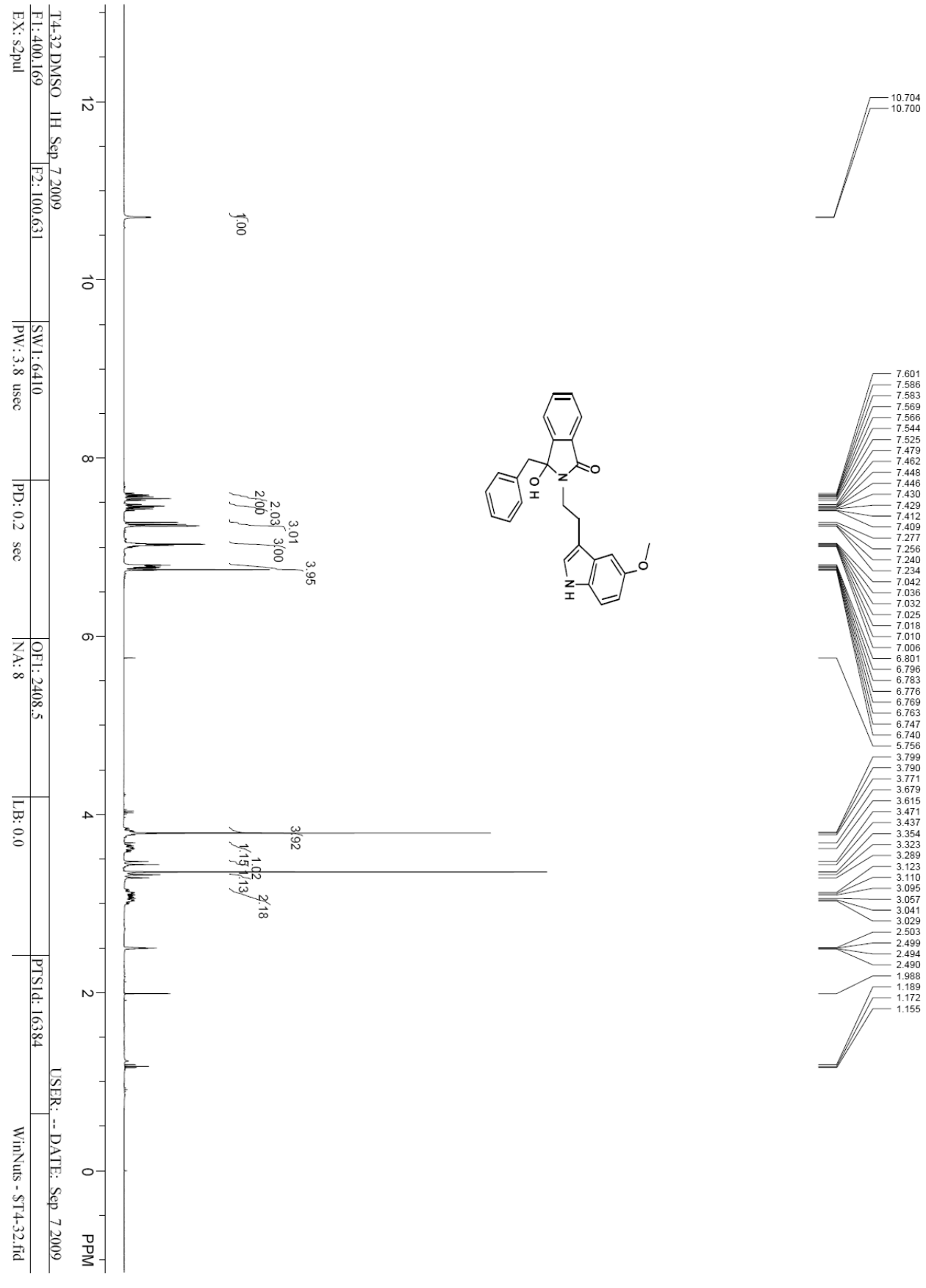

T4-17 DMSO BB+DEPT-135 Aug 20 2009
165.854
162.087
159.685
147.001
135.652
135.239
131.509
131.424
130.485
130.407
129.855
129.012
127.515
126.370
122.929
121.856
115.245
115.037
91.031
42.383
40.596
40.127
39.918
39.709
39.500
39.291
39.083
38.874
34.150
180
160
140
120
100
80
60
40
20
ppm

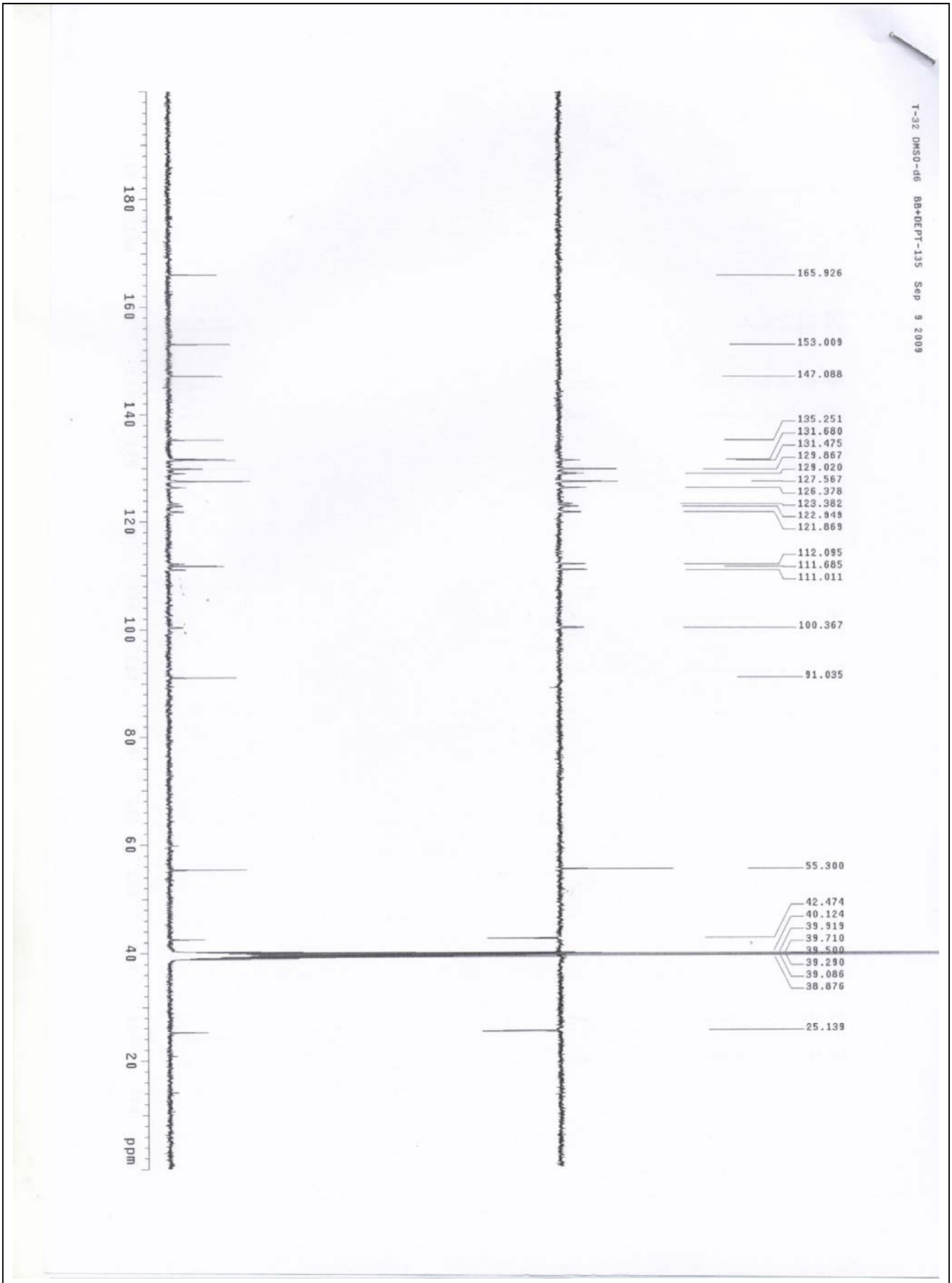
2-(2-(1H-indol-3-yl)ethyl)-3-benzyl-3-hydroxyisoindolin-1-one (9Ar)



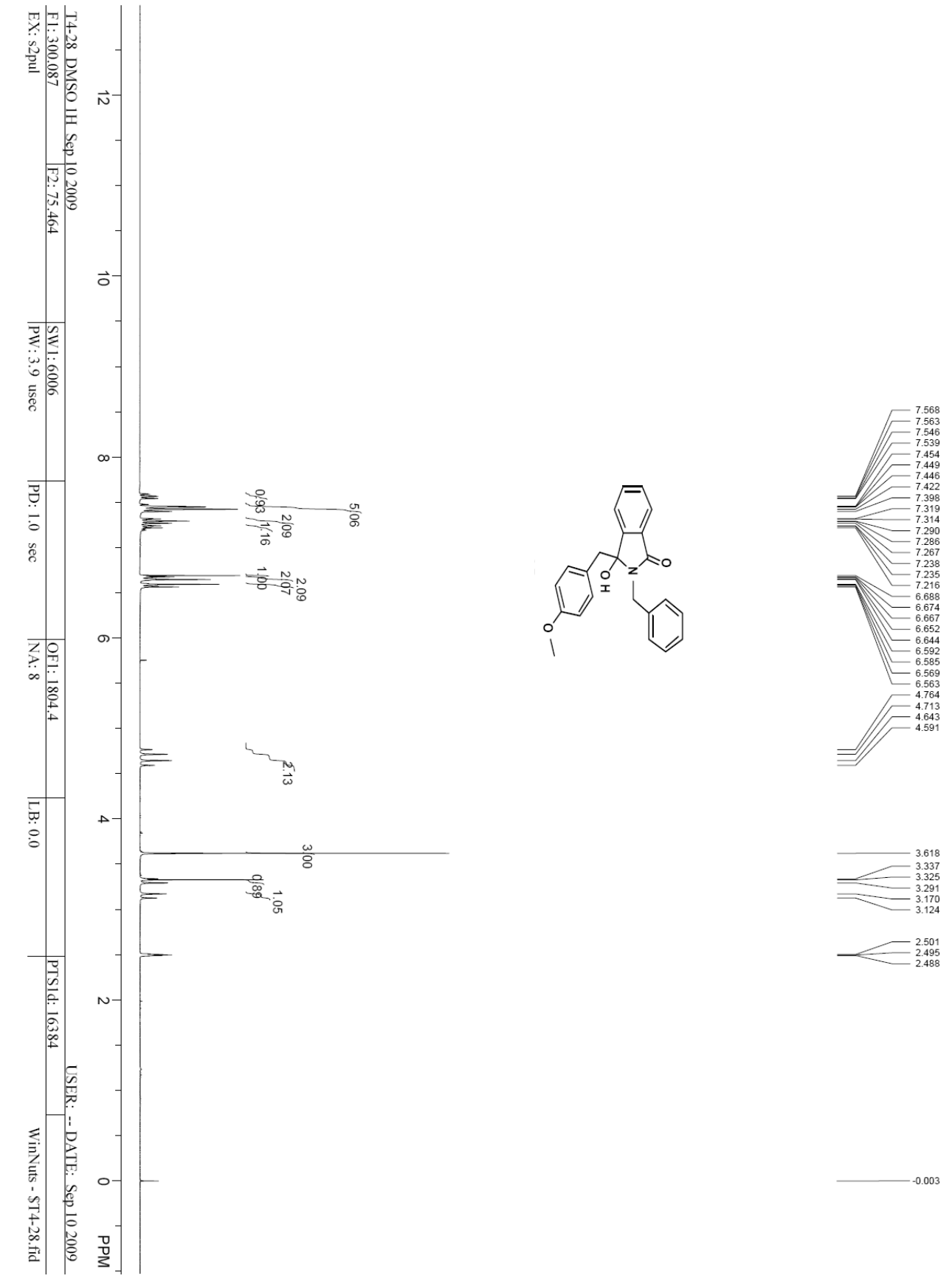

T6-0 CDCl3 BB+DEPT-135 Jun 9 2009
166.997
146.110
136.184
134.391
131.645
131.482
130.105
129.555
127.925
127.300
126.958
122.969
122.575
122.158
119.569
119.048
113.534
111.183
91.249
77.320
77.000
76.680
42.958
40.540
24.825
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

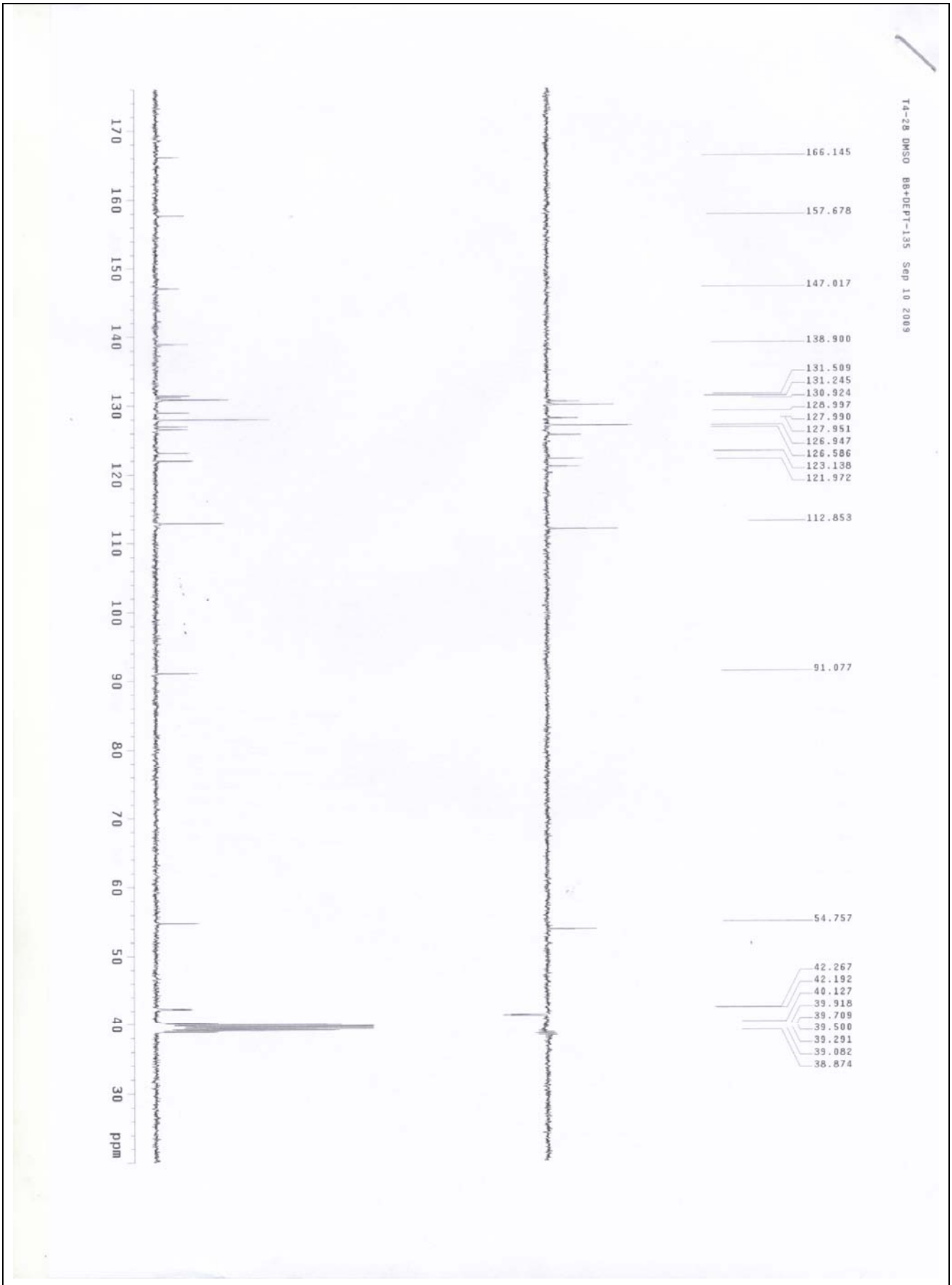
3-benzyl-3-hydroxy-2-(2-(5-methoxy-1H-indol-3-yl)ethyl)isoindolin-1-one (9As)



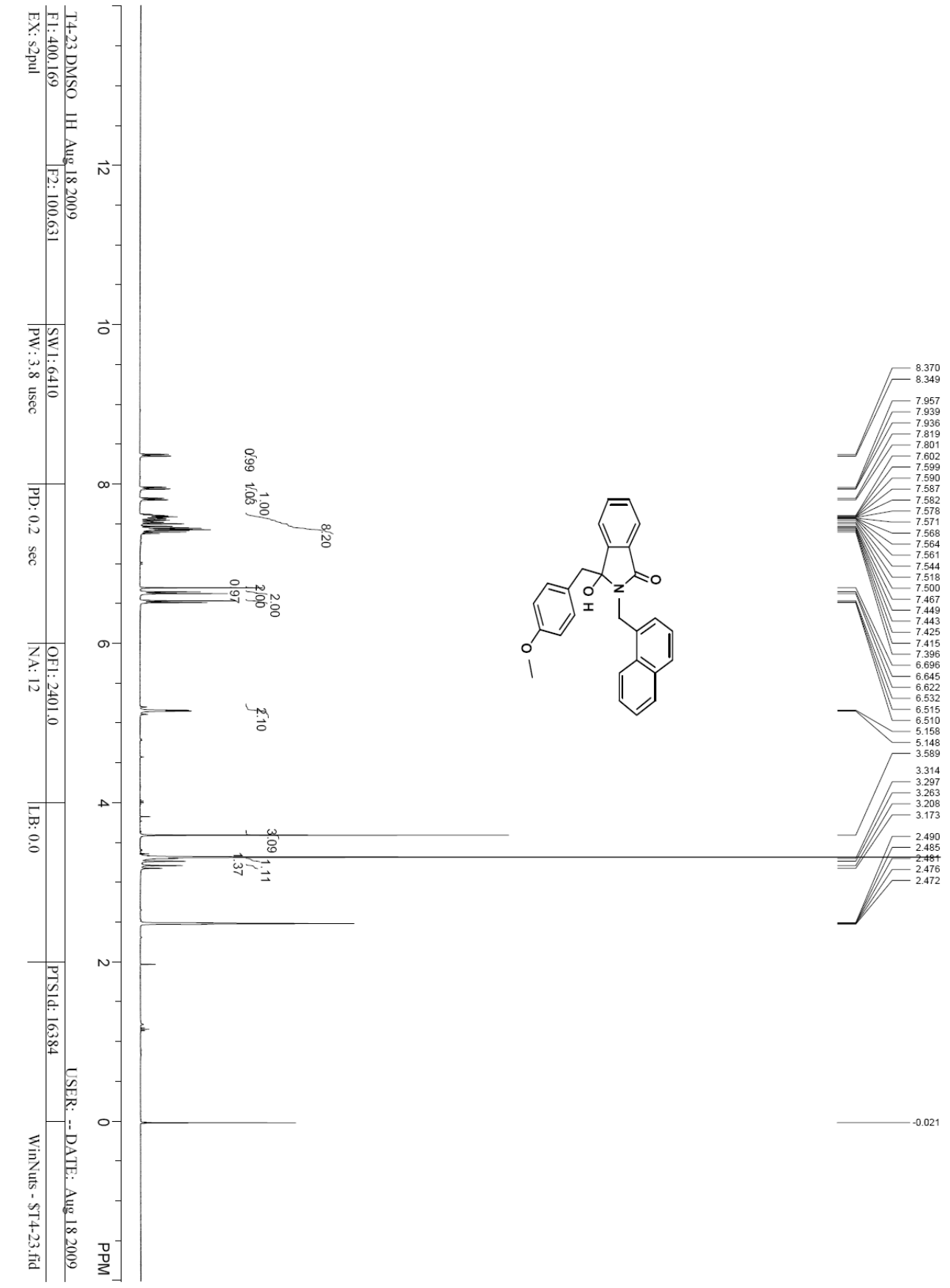

T-32 DMSO-d6 BB+DEPT-135 Sep 9 2009
165.926
153.009
147.088
135.251
131.680
131.475
129.867
129.020
127.567
126.378
123.382
122.949
121.869
112.095
111.685
111.011
100.367
91.035
55.300
42.474
40.124
39.919
39.710
39.500
39.290
39.086
38.876
25.139
180
160
140
120
100
80
60
40
20
ppm

2-benzyl-3-hydroxy-3-(4-methoxybenzyl)isoindolin-1-one (9Ba)



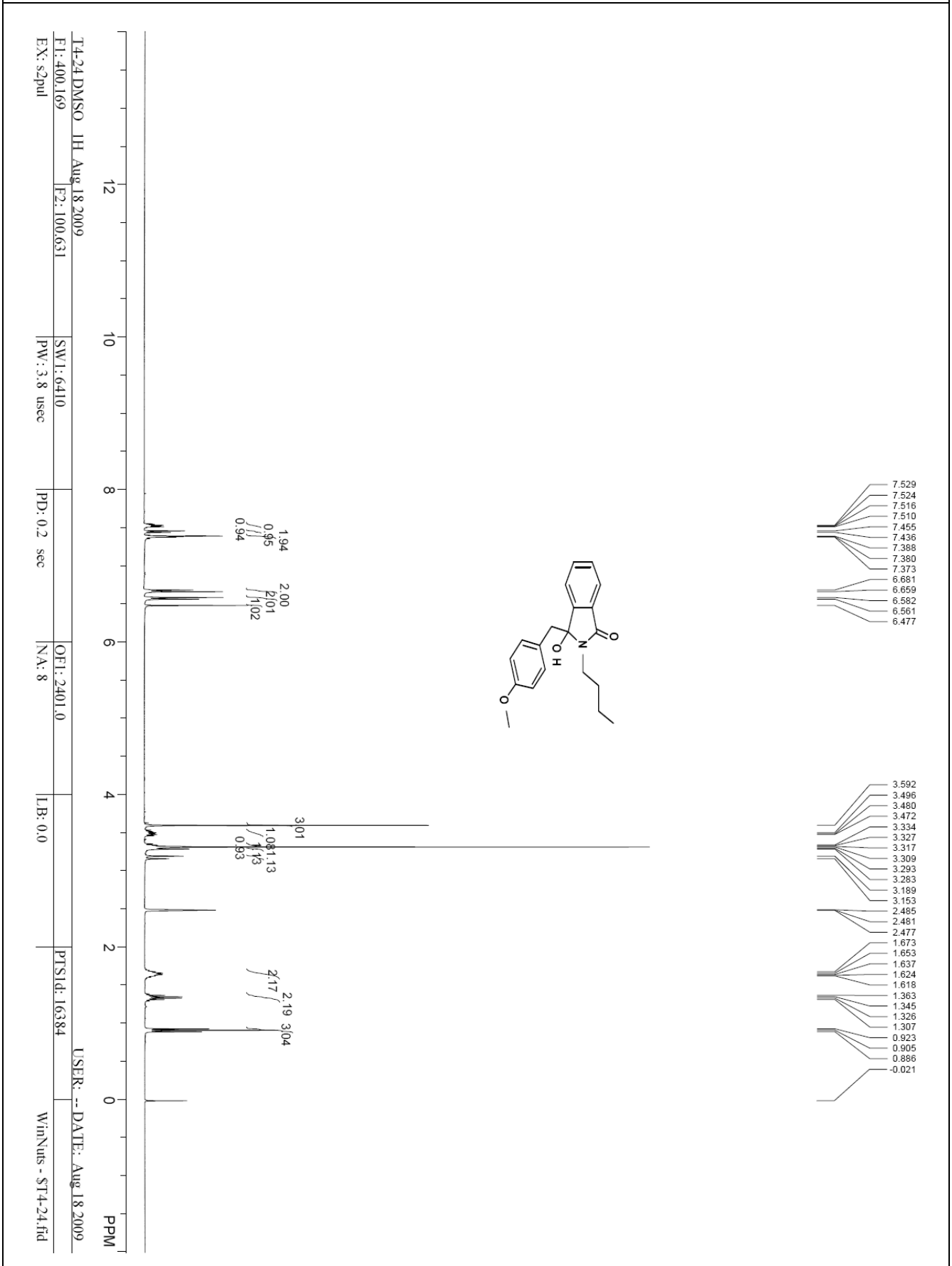

T4-28 DMSO BB+DEPT-135 Sep 10 2009
166.145
157.678
147.017
138.900
131.509
131.245
130.924
128.997
127.990
127.951
126.947
126.586
123.138
121.972
112.853
91.077
54.757
42.267
42.192
40.127
39.918
39.709
39.500
39.291
39.082
38.874
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

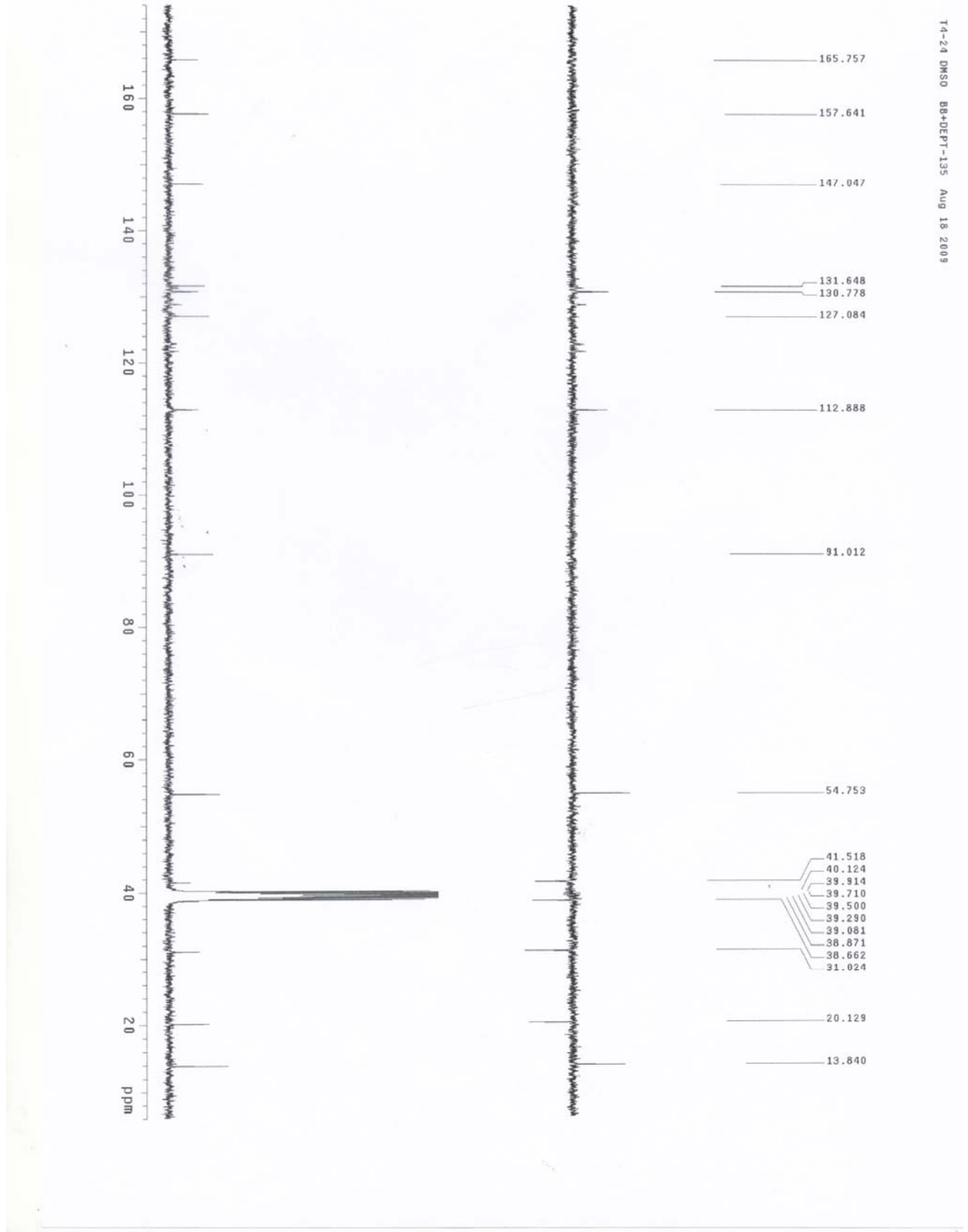
3-hydroxy-3-(4-methoxybenzyl)-2-(naphthalen-1-ylmethyl)isoindolin-1-one (9Bk)



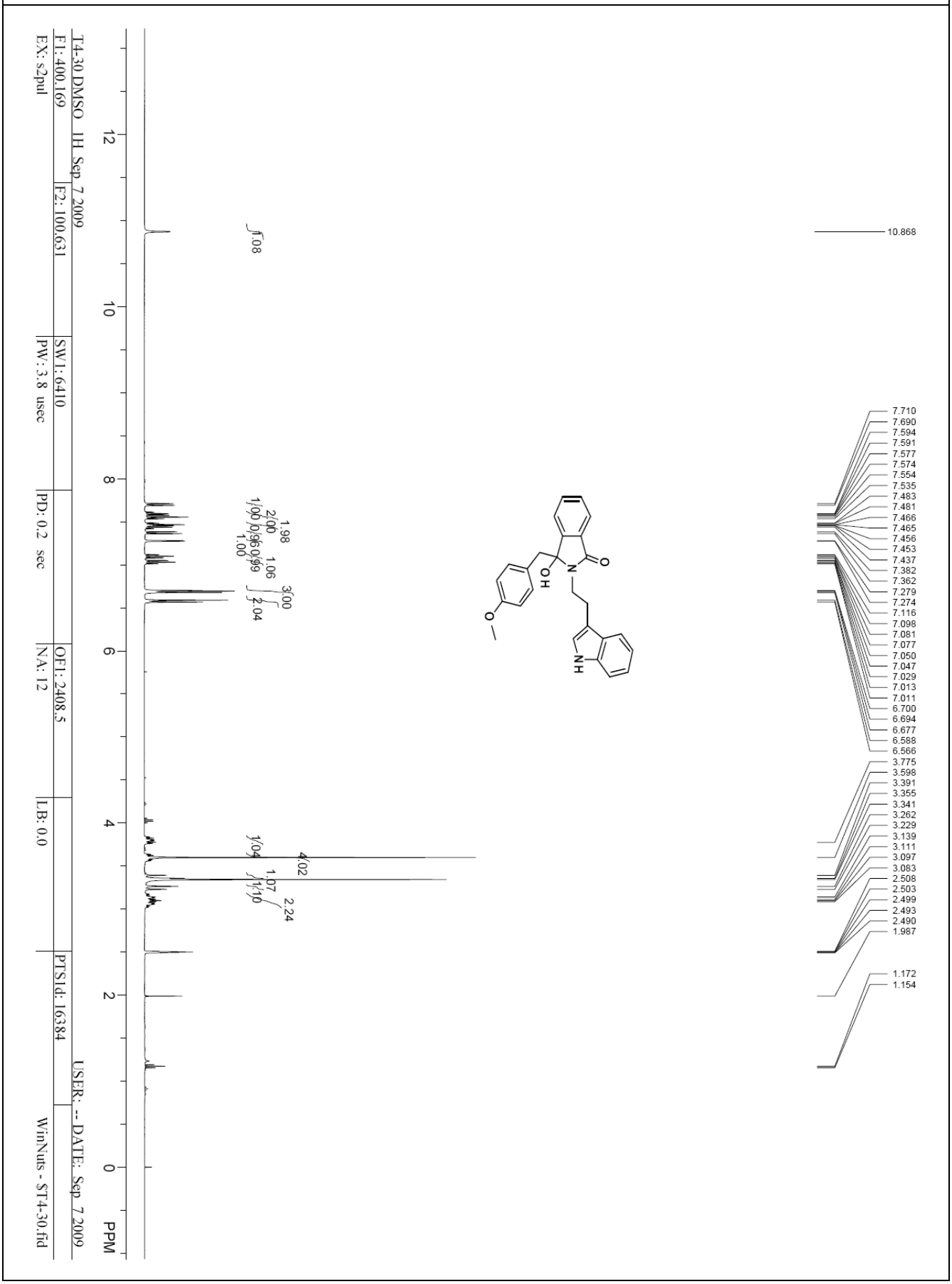

T4-23 DMSO-d6 BB+DEPT-135 Aug 18 2009
167.641
157.299
143.495
143.158
141.645
141.135
140.988
140.784
139.085
138.472
137.088
136.888
136.080
135.630
135.327
135.121
133.563
133.146
132.147
122.787
101.441
64.737
52.214
50.127
49.918
49.709
49.500
49.291
49.083
48.874
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm

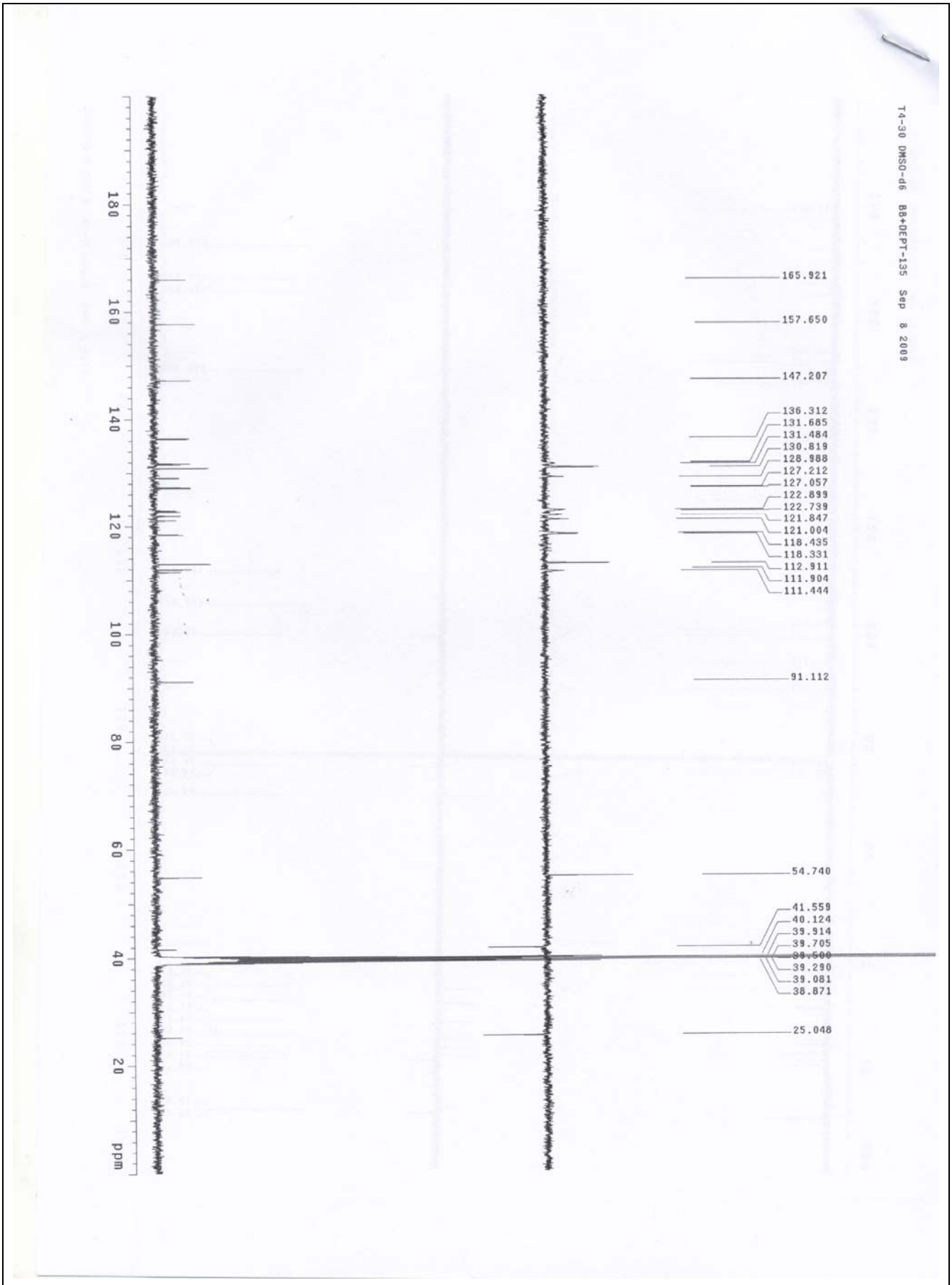
2-butyl-3-hydroxy-3-(4-methoxybenzyl)isoindolin-1-one (9Bm)



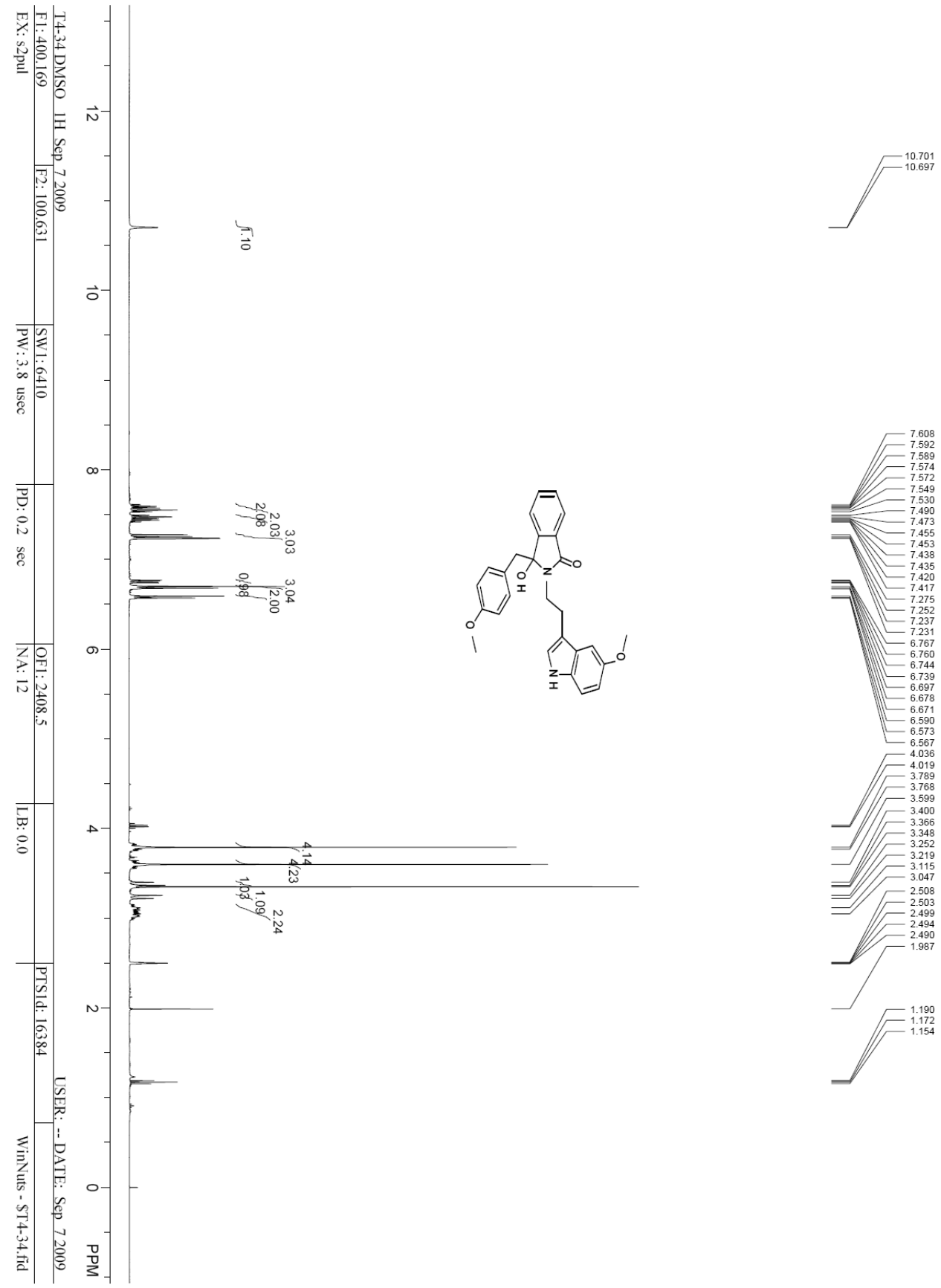

T4-24 DMSO BB+DEPT-135 Aug 18 2009
165.757
157.641
147.047
131.648
130.778
127.084
112.888
91.012
54.753
41.518
40.124
39.914
39.710
39.500
39.290
39.081
38.871
38.662
31.024
20.129
13.840
160
140
120
100
80
60
40
20
ppm

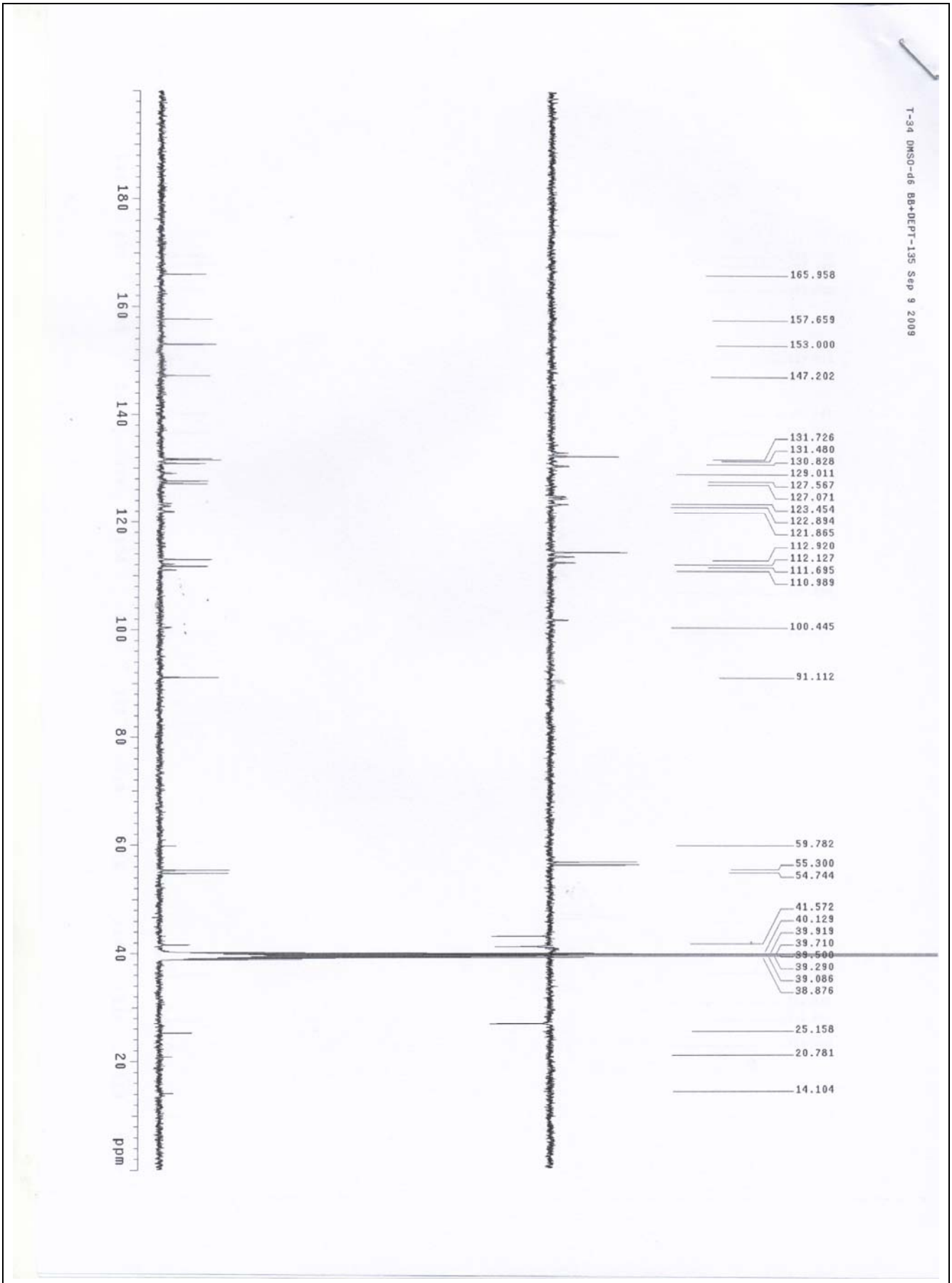
2-(2-(1H-indol-3-yl)ethyl)-3-hydroxy-3-(4-methoxybenzyl)isoindolin-1-one (9Br)



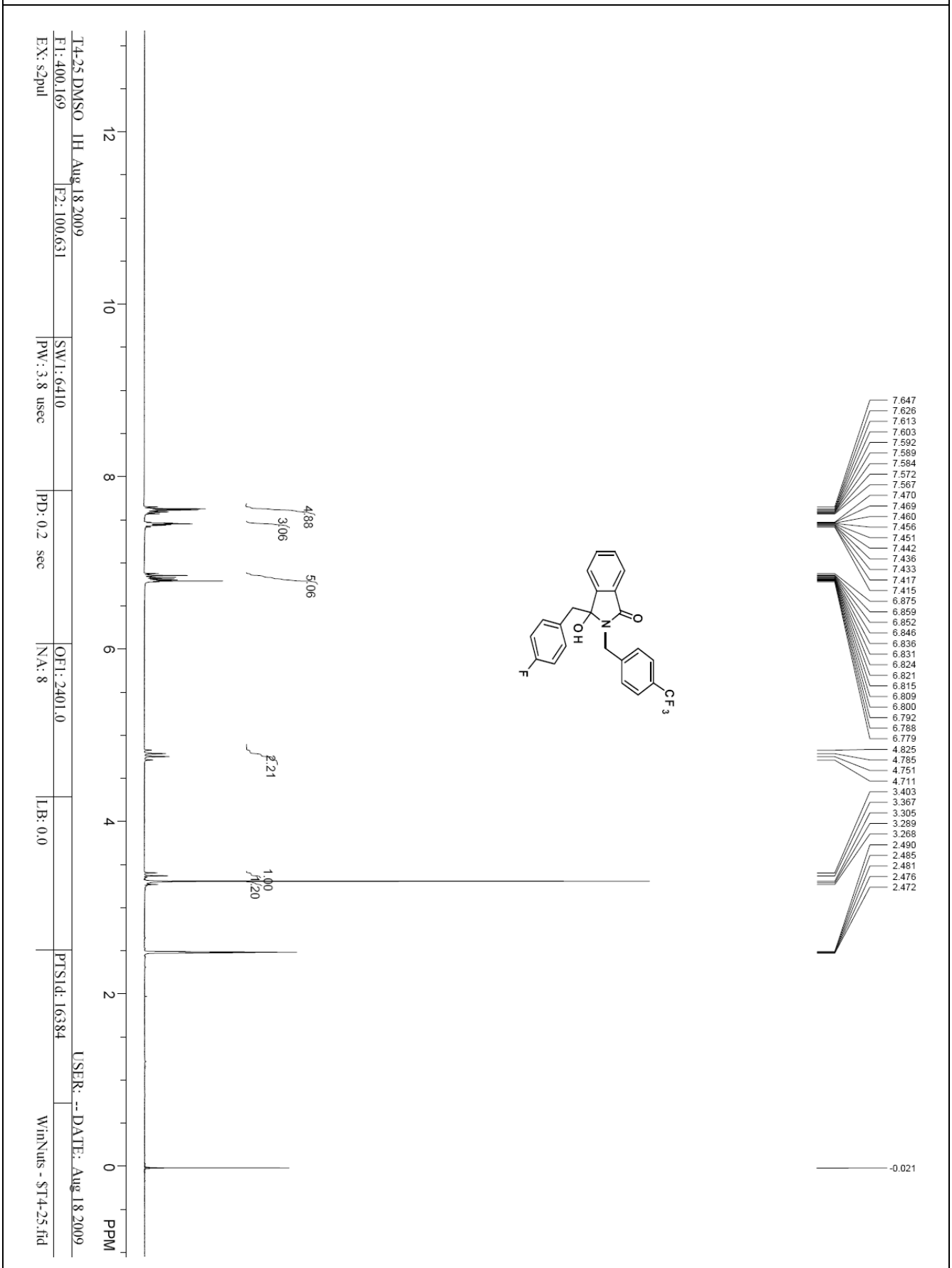

T4-30 DMSO-d6 BB+DEPT-135 Sep 8 2009
165.921
157.650
147.207
136.312
131.685
131.484
130.819
128.988
127.212
127.057
122.899
122.739
121.847
121.004
118.435
118.331
112.911
111.904
111.444
91.112
54.740
41.559
40.124
39.914
39.705
39.500
39.290
39.081
38.871
25.048
180
160
140
120
100
80
60
40
20
ppm

3-hydroxy-2-(2-(5-methoxy-1H-indol-3-yl)ethyl)-3-(4-methoxybenzyl)isoindolin-1-one (9Bs)



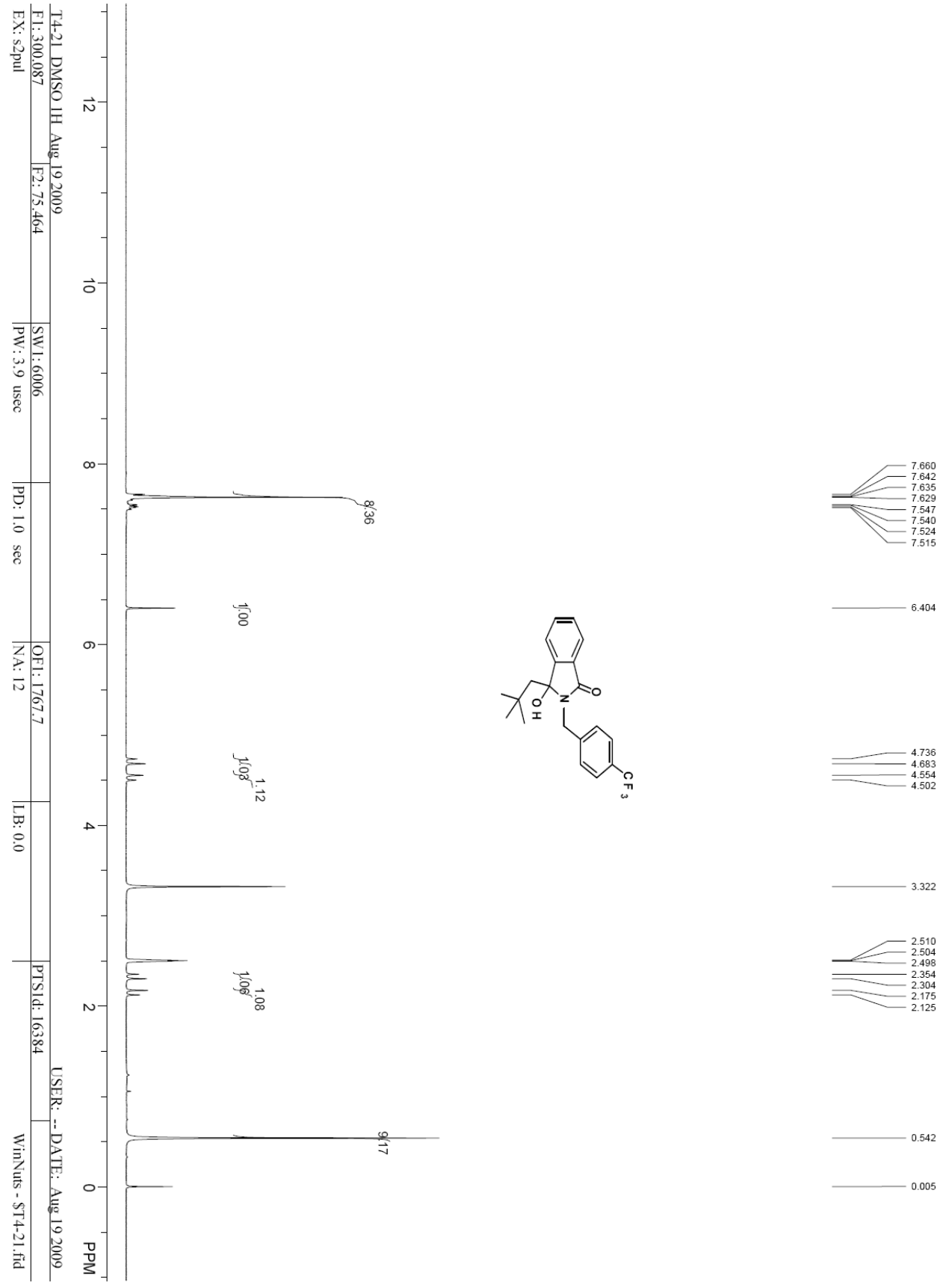

T-34 DMSO-d6 BB+DEPT-135 Sep 9 2009
165.958
157.659
153.000
147.202
131.726
131.480
130.828
129.011
127.567
127.071
123.454
122.894
121.865
112.920
112.127
111.695
110.989
100.445
91.112
59.782
55.300
54.744
41.572
40.129
39.919
39.710
39.500
39.290
39.086
38.876
25.158
20.781
14.104
180
160
140
120
100
80
60
40
20
ppm

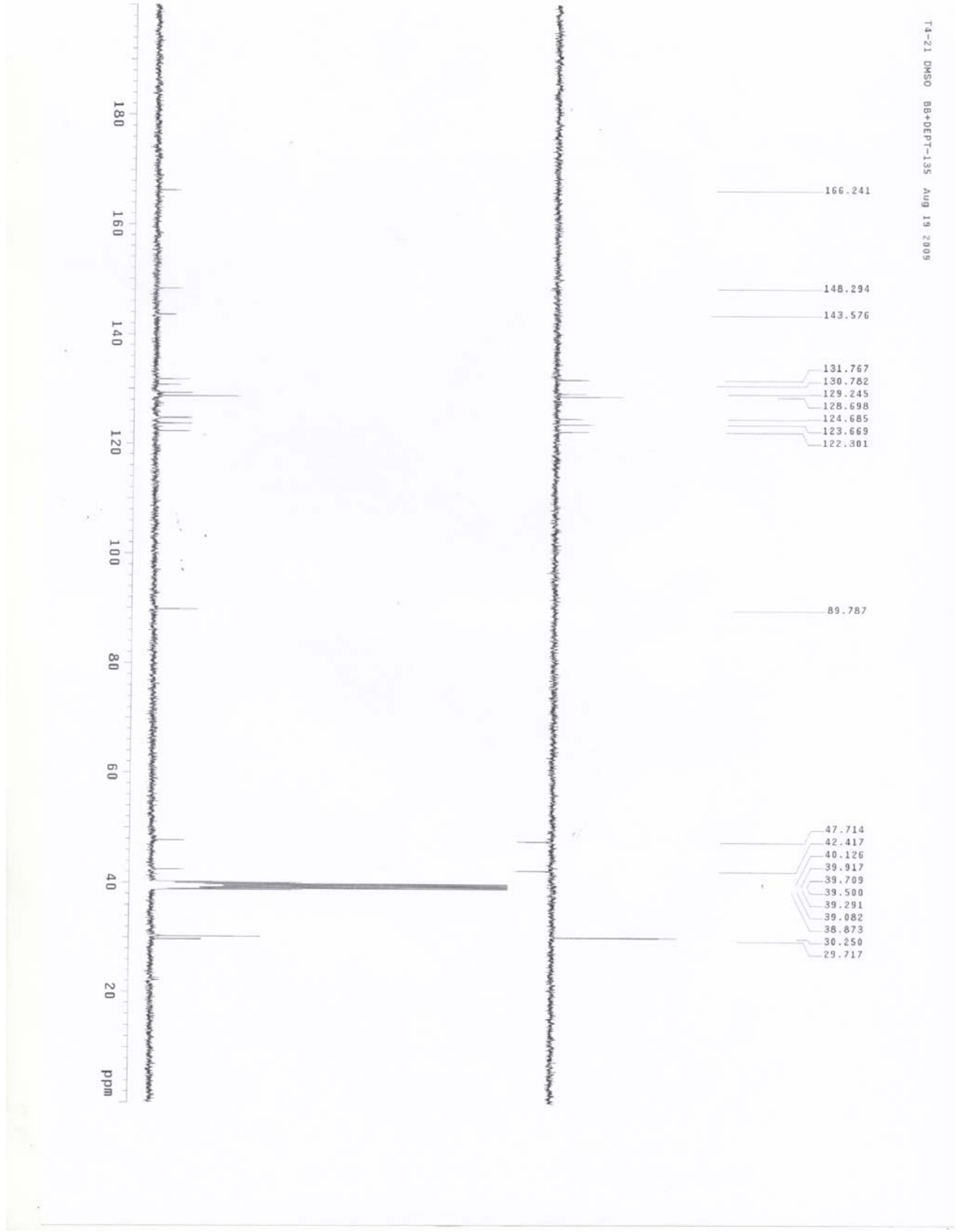
3-(4-fluorobenzyl)-3-hydroxy-2-(4-(trifluoromethyl)benzyl)isoindolin-1-one (9Ch)



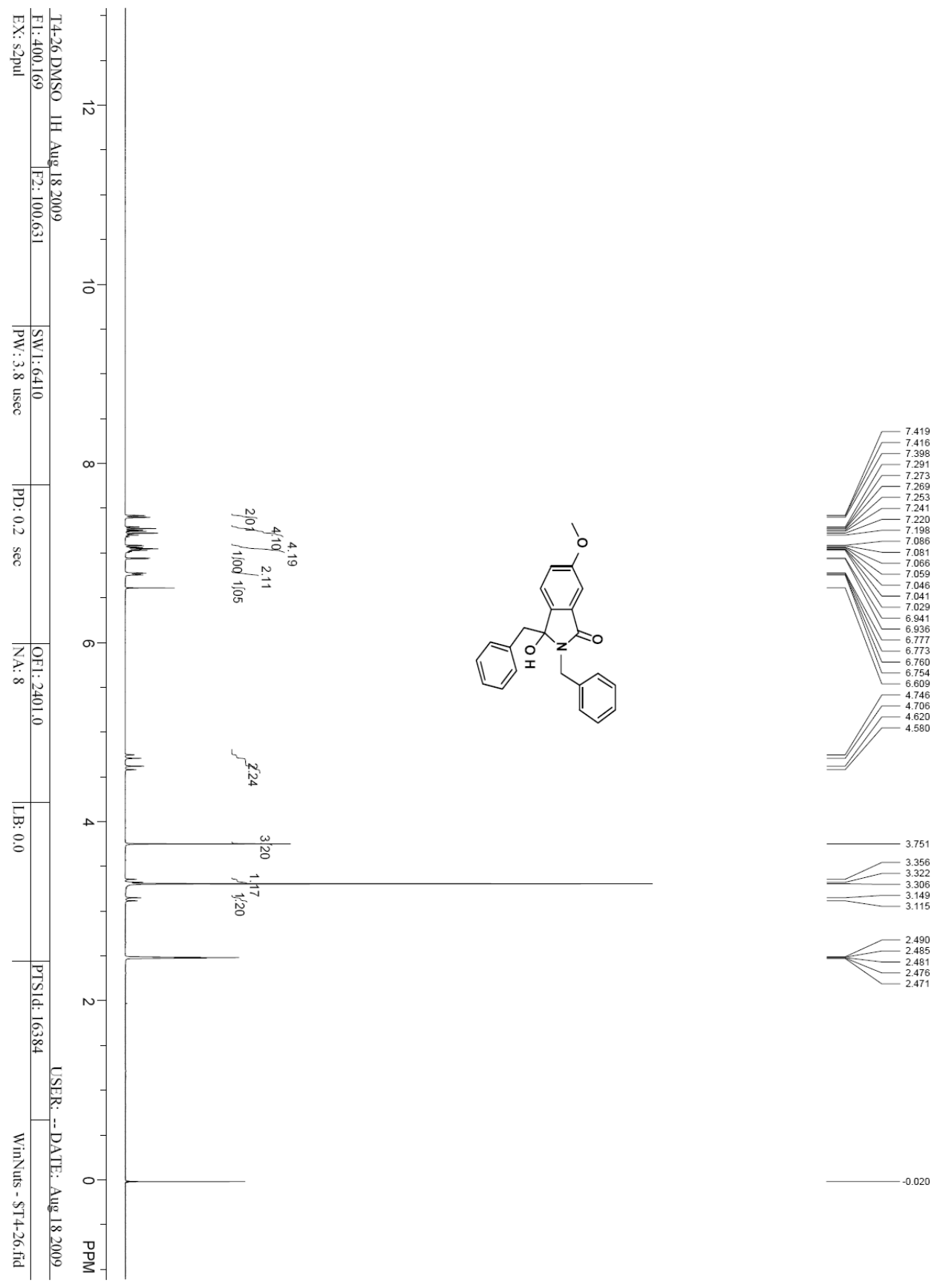

T4-25 DMSO-d6 BB+DEPT-135 Aug 18 2009
176.143
172.111
156.806
153.672
141.828
141.714
141.633
141.296
140.960
139.224
138.505
134.890
133.143
132.109
124.387
124.178
100.954
51.935
51.856
50.127
49.918
49.709
49.500
49.292
49.083
48.874
10.097
180
160
140
120
100
80
60
40
20
ppm

3-hydroxy-3-neopentyl-2-(4-(trifluoromethyl)benzyl)isoindolin-1-one (9Dh)



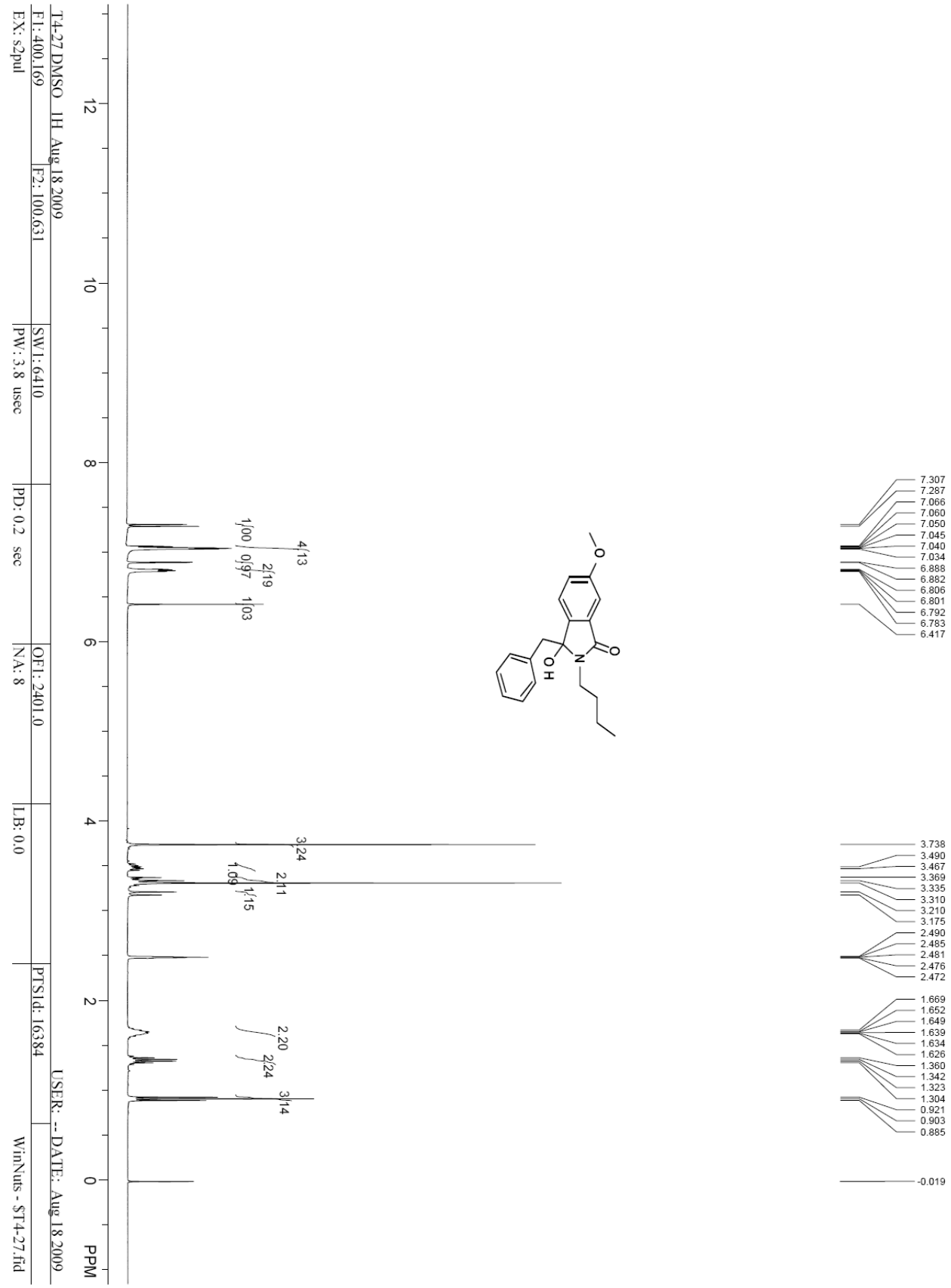

T4-21 DMSO BB+DEPT-135 Aug 19 2009
166.241
148.294
143.576
131.767
130.782
129.245
128.698
124.685
123.669
122.301
89.787
47.714
42.417
40.126
39.917
39.709
39.500
39.291
39.082
38.873
30.250
29.717
180
160
140
120
100
80
60
40
20
ppm

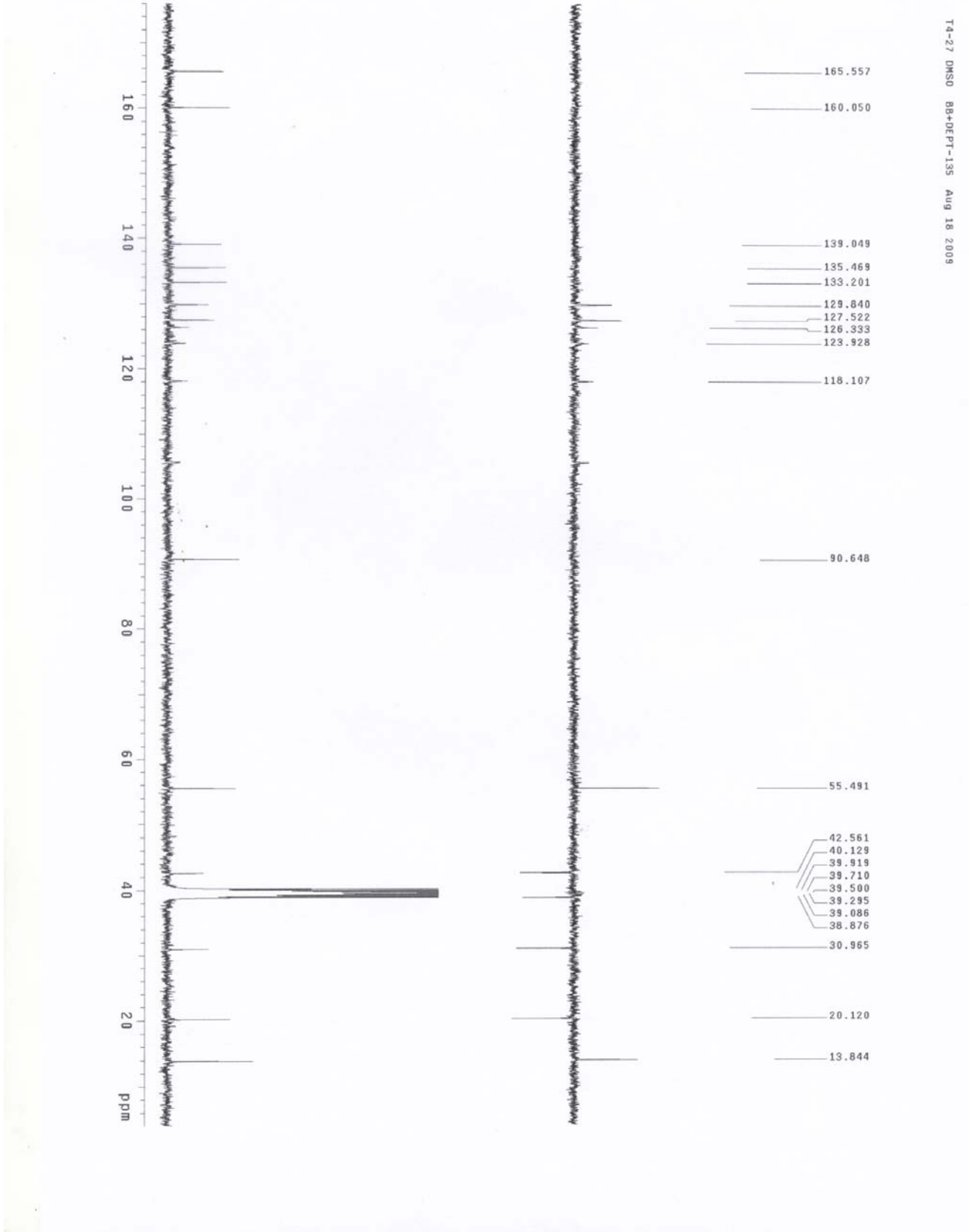
2,3-dibenzyl-3-hydroxy-6-methoxyisoindolin-1-one (9Ea)



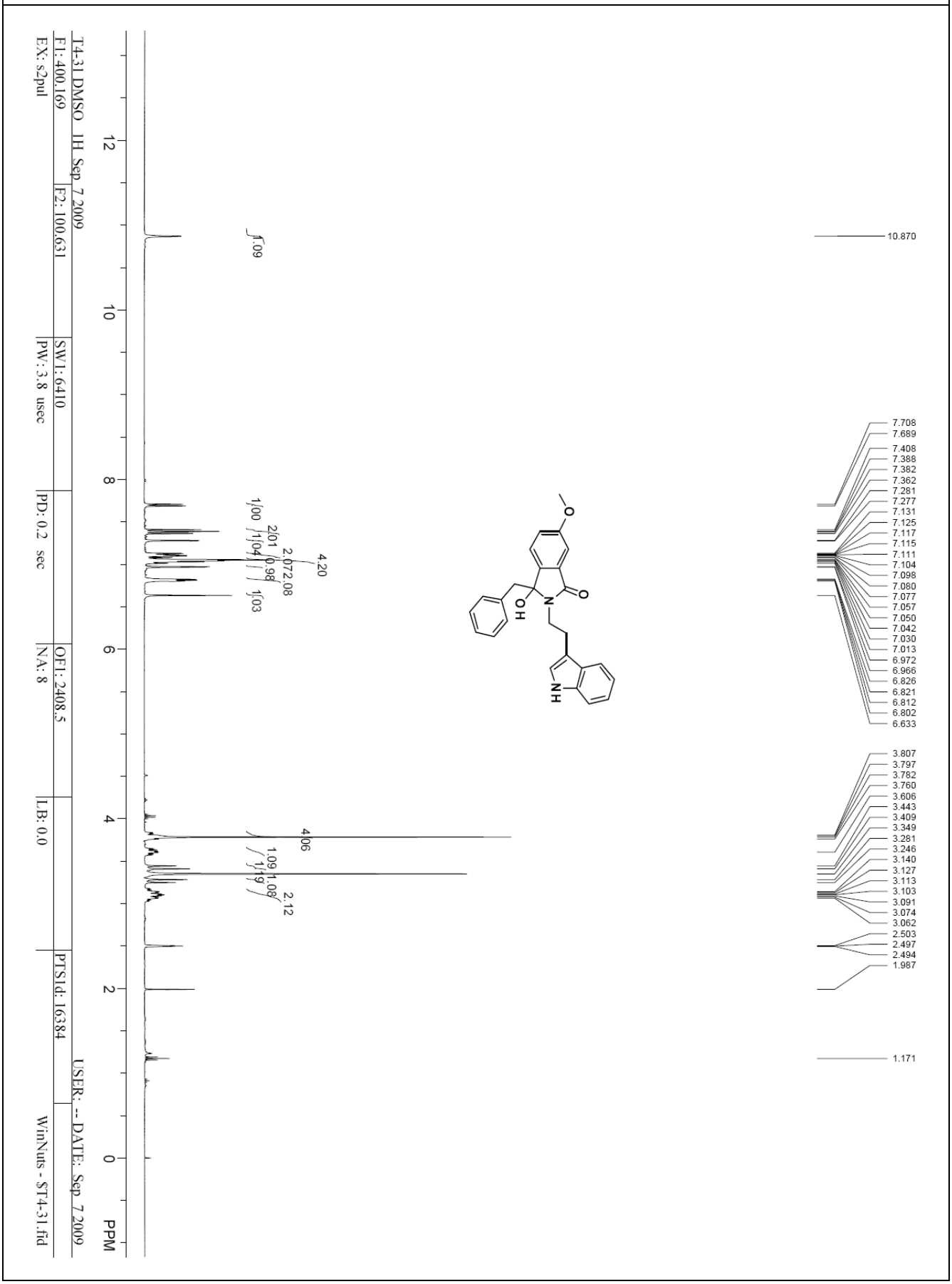

T4-26 DMSO-d6 BB+DEPT-135 Aug 18 2009
165.954
160.124
139.026
138.884
135.318
132.782
129.981
127.993
127.887
127.488
126.577
126.373
124.248
118.355
105.695
90.705
55.511
43.314
42.258
40.127
39.918
39.709
39.500
39.291
39.082
38.874
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

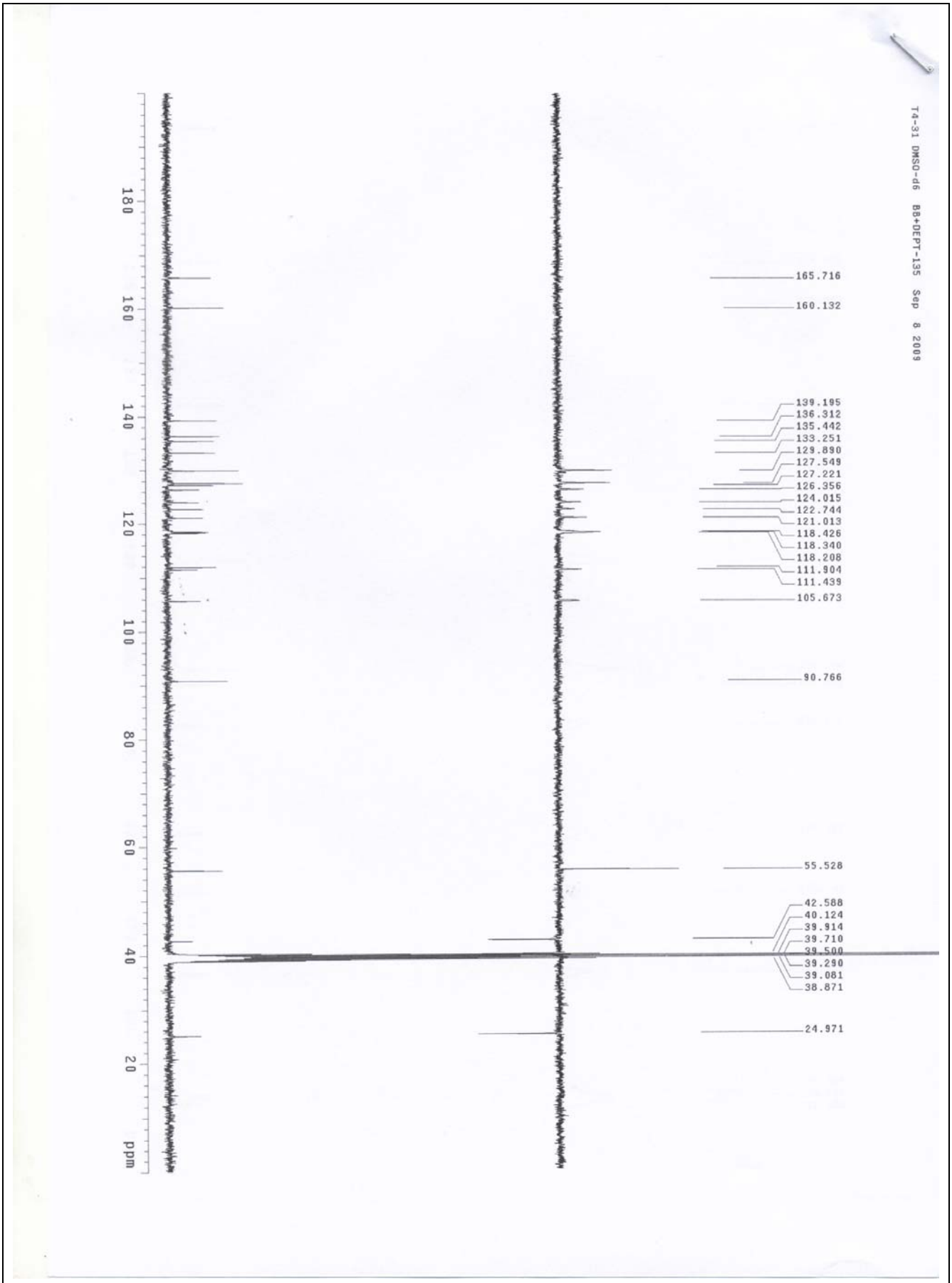
3-benzyl-2-butyl-3-hydroxy-6-methoxyisoindolin-1-one (9Em)



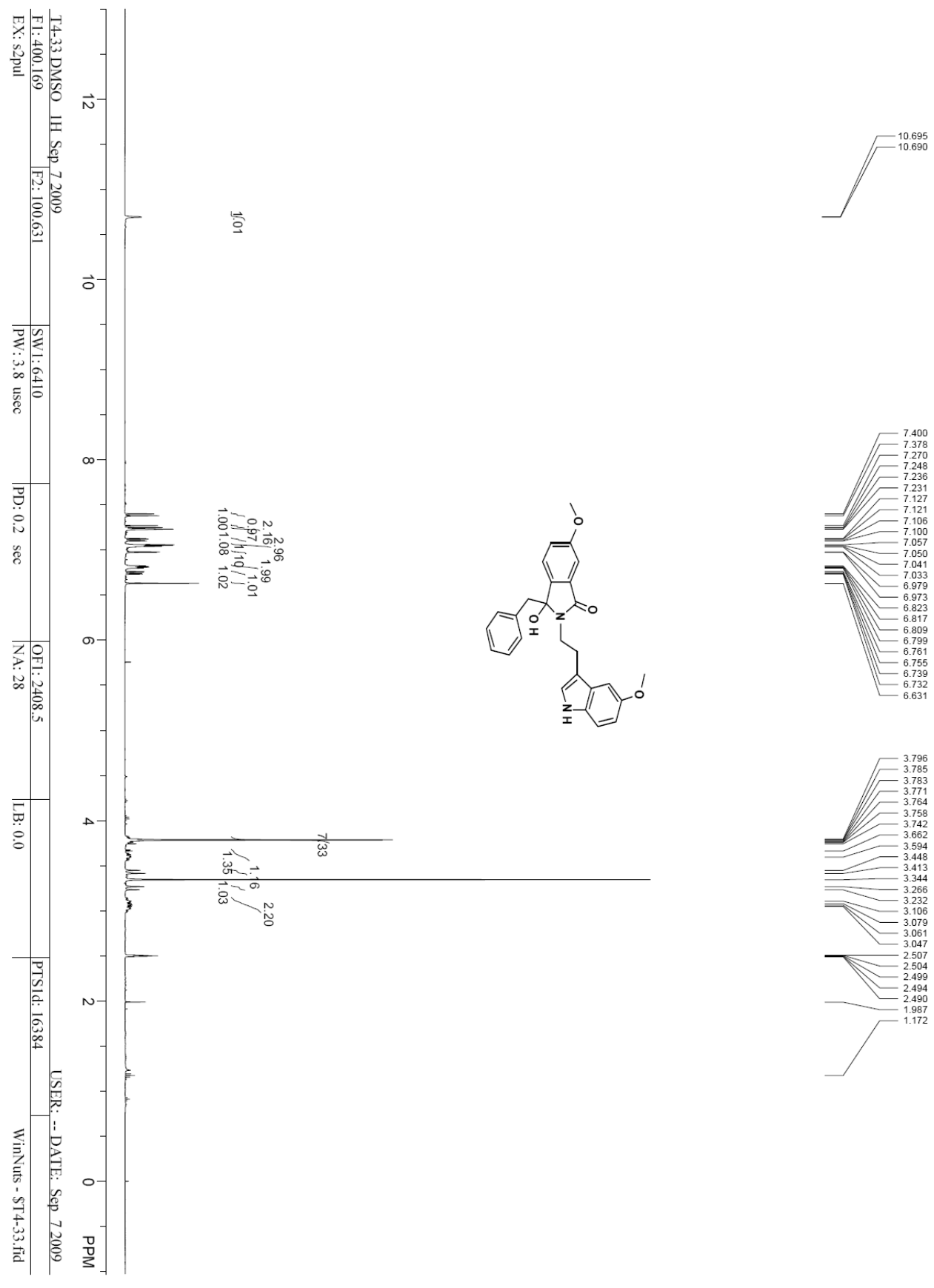

T4-27 DMSO BB+DEPT-135 Aug 18 2009
165.557
160.050
139.049
135.469
133.201
129.840
127.522
126.333
123.928
118.107
90.648
55.491
42.561
40.129
39.919
39.710
39.500
39.295
39.086
38.876
30.965
20.120
13.844
160
140
120
100
80
60
40
20
ppm

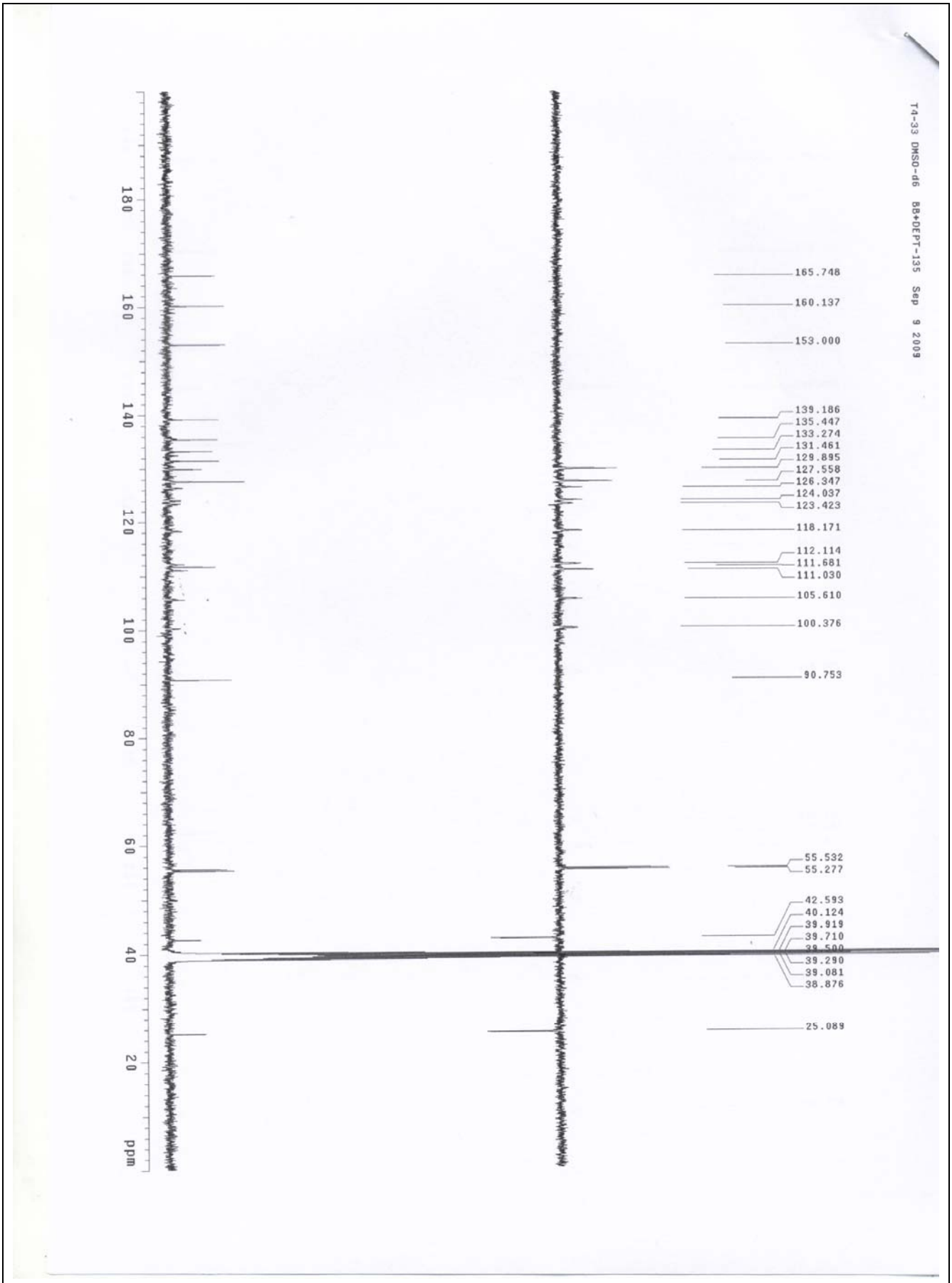
2-(2-(1H-indol-3-yl)ethyl)-3-benzyl-3-hydroxy-6-methoxyisoindolin-1-one (9Er)



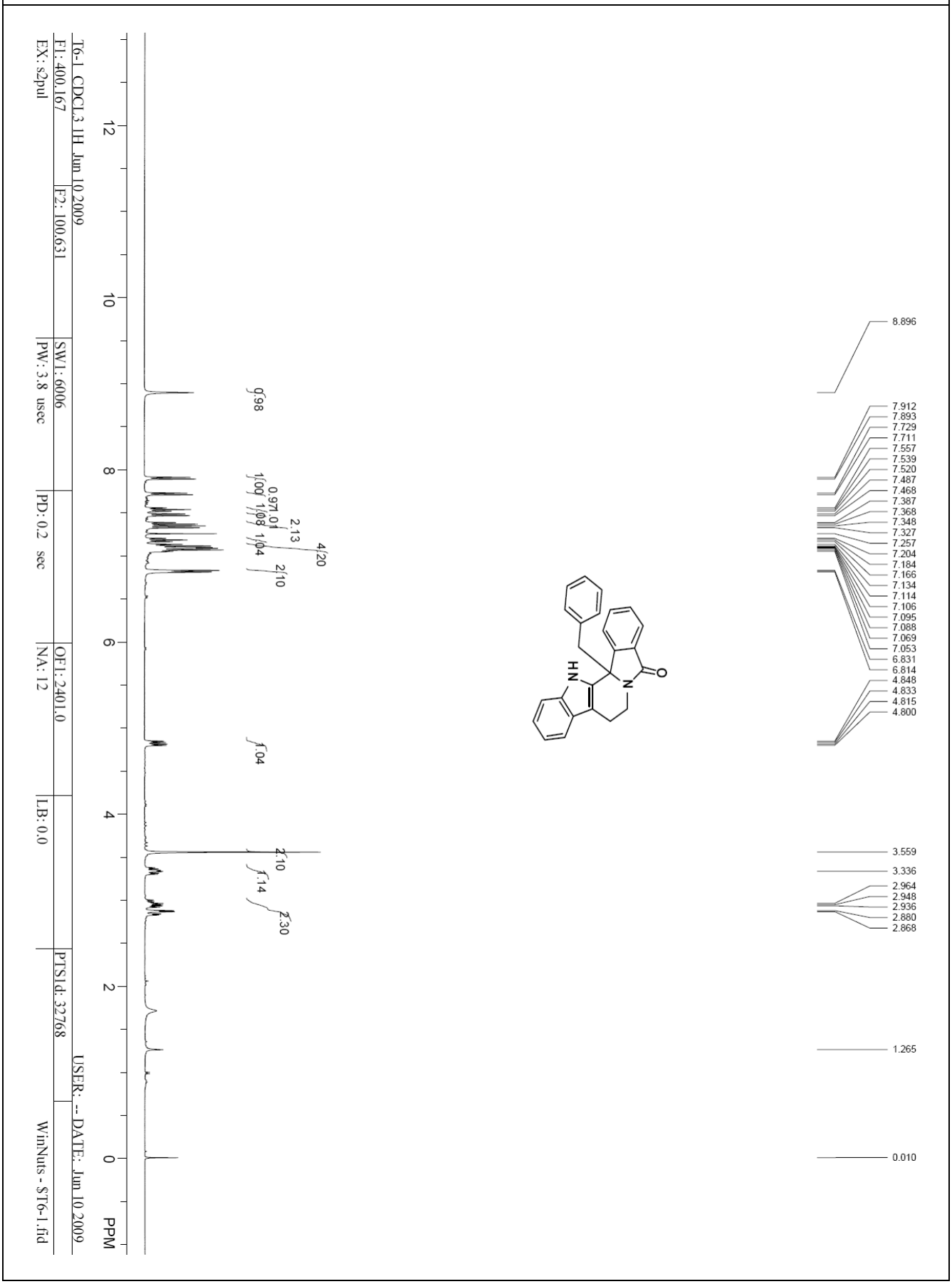

T4-31 DMSO-d6 BB+DEPT-135 Sep 8 2009
165.716
160.132
139.195
136.312
135.442
133.251
129.890
127.549
127.221
126.356
124.015
122.744
121.013
118.426
118.340
118.208
111.904
111.439
105.673
90.766
55.528
42.588
40.124
39.914
39.710
39.500
39.290
39.081
38.871
24.971
180
160
140
120
100
80
60
40
20
ppm

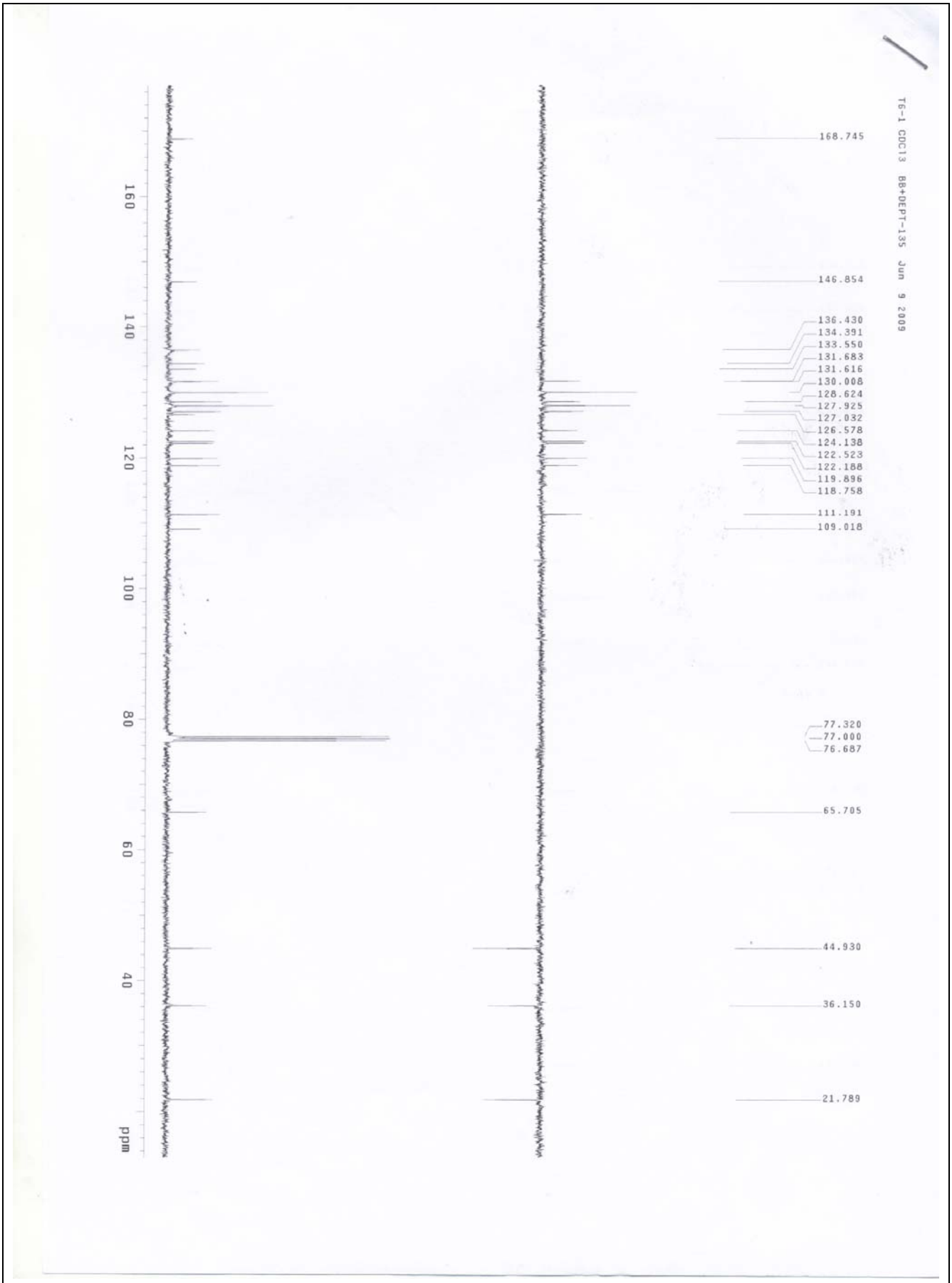
3-benzyl-3-hydroxy-6-methoxy-2-(2-(5-methoxy-1H-indol-3-yl)ethyl)isoindolin-1-one (9Es)



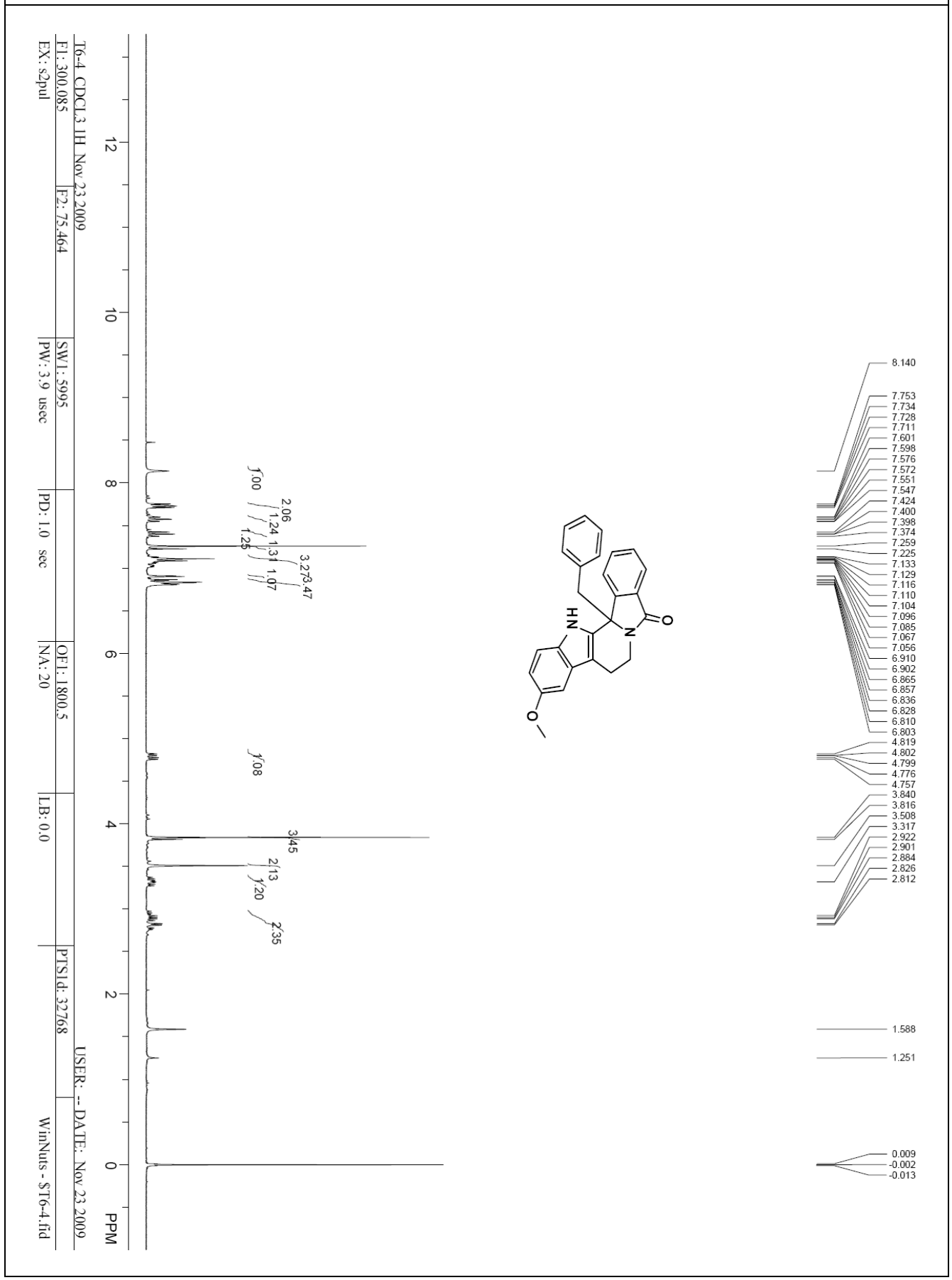

T4-33 DMSO-d6 BB+DEPT-135 Sep 9 2009
165.748
160.137
153.000
139.186
135.447
133.274
131.461
129.895
127.558
126.347
124.037
123.423
118.171
112.114
111.681
111.030
105.610
100.376
90.753
55.532
55.277
42.593
40.124
39.919
39.710
39.500
39.290
39.081
38.876
25.089
180
160
140
120
100
80
60
40
20
ppm

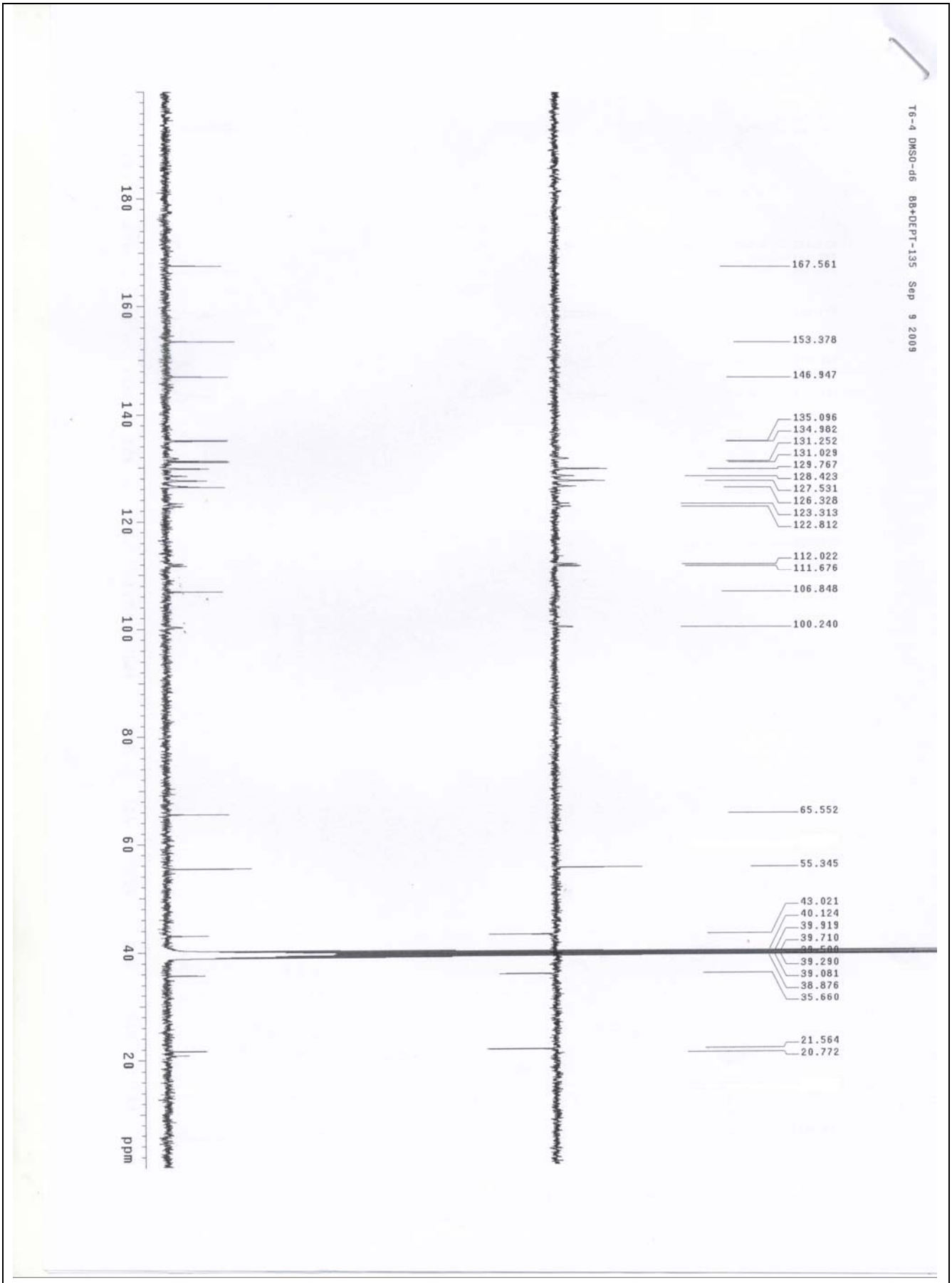
Compound 10Ar



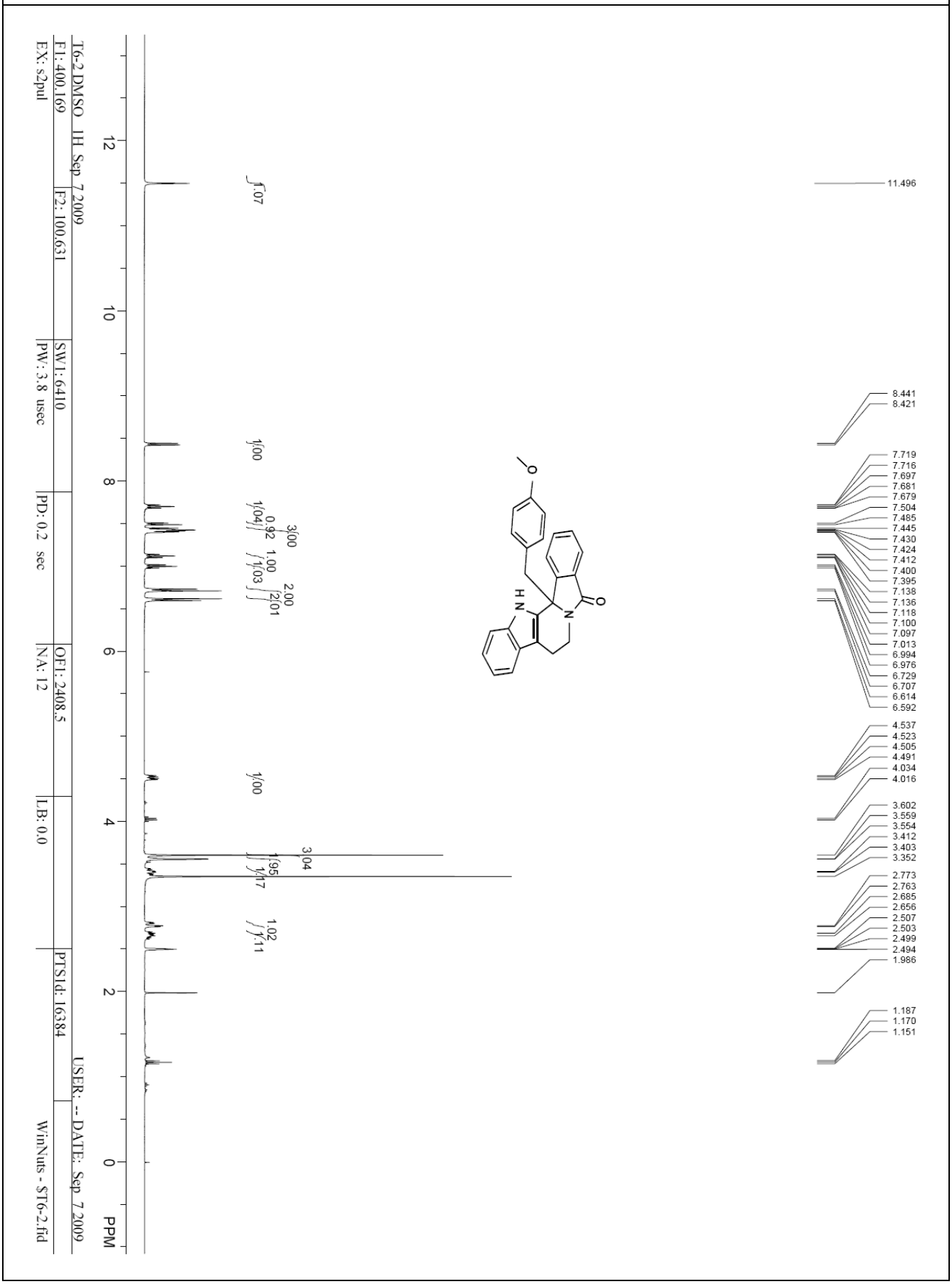

T6-1 CDCl3 BB+DEPT-135 Jun 9 2009
168.745
146.854
136.430
134.391
133.550
131.683
131.616
130.008
128.624
127.925
127.032
126.578
124.138
122.523
122.188
119.896
118.758
111.191
109.018
77.320
77.000
76.687
65.705
44.930
36.150
21.789
160
140
120
100
80
60
40
ppm

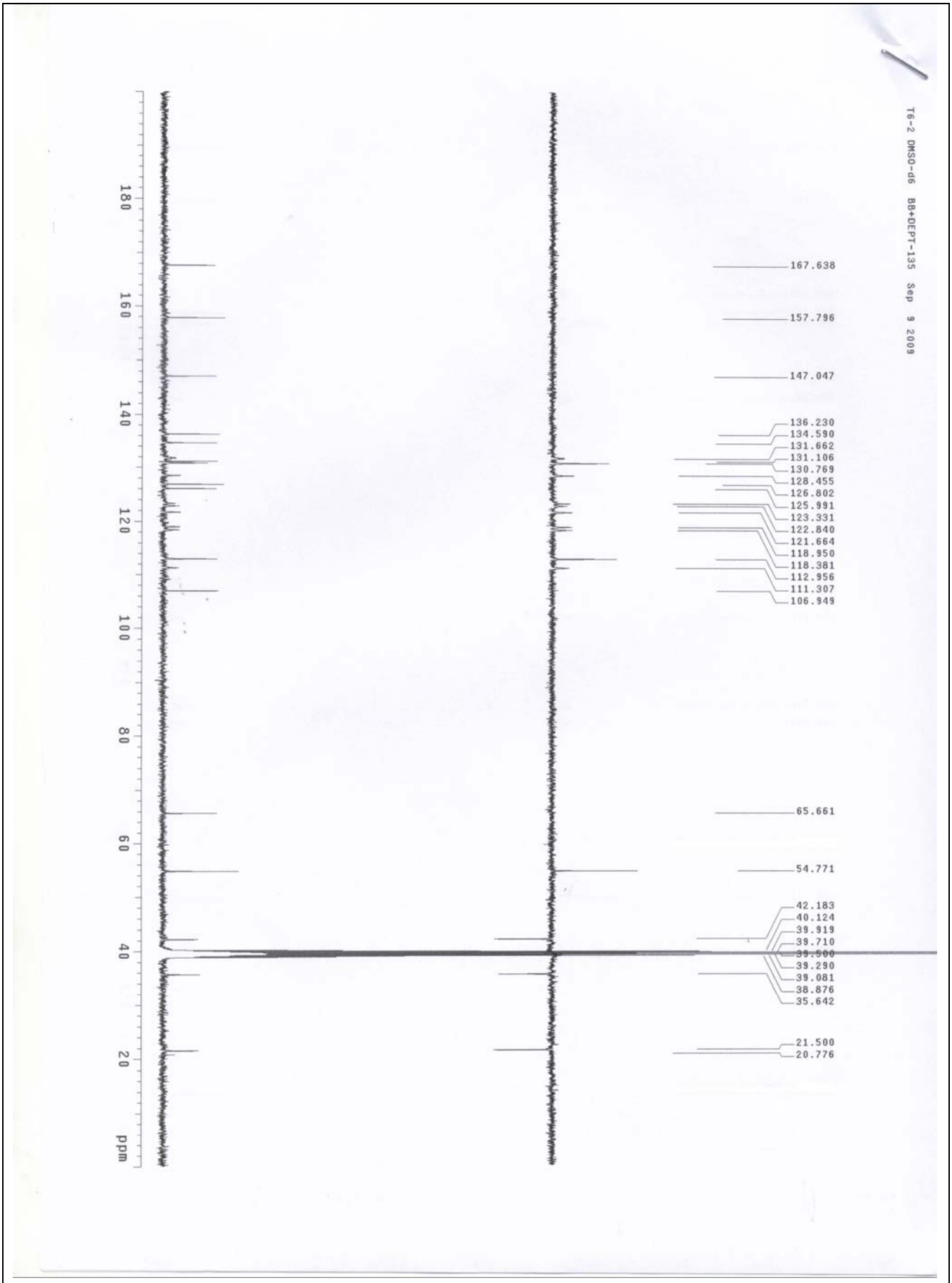
Compound 10As



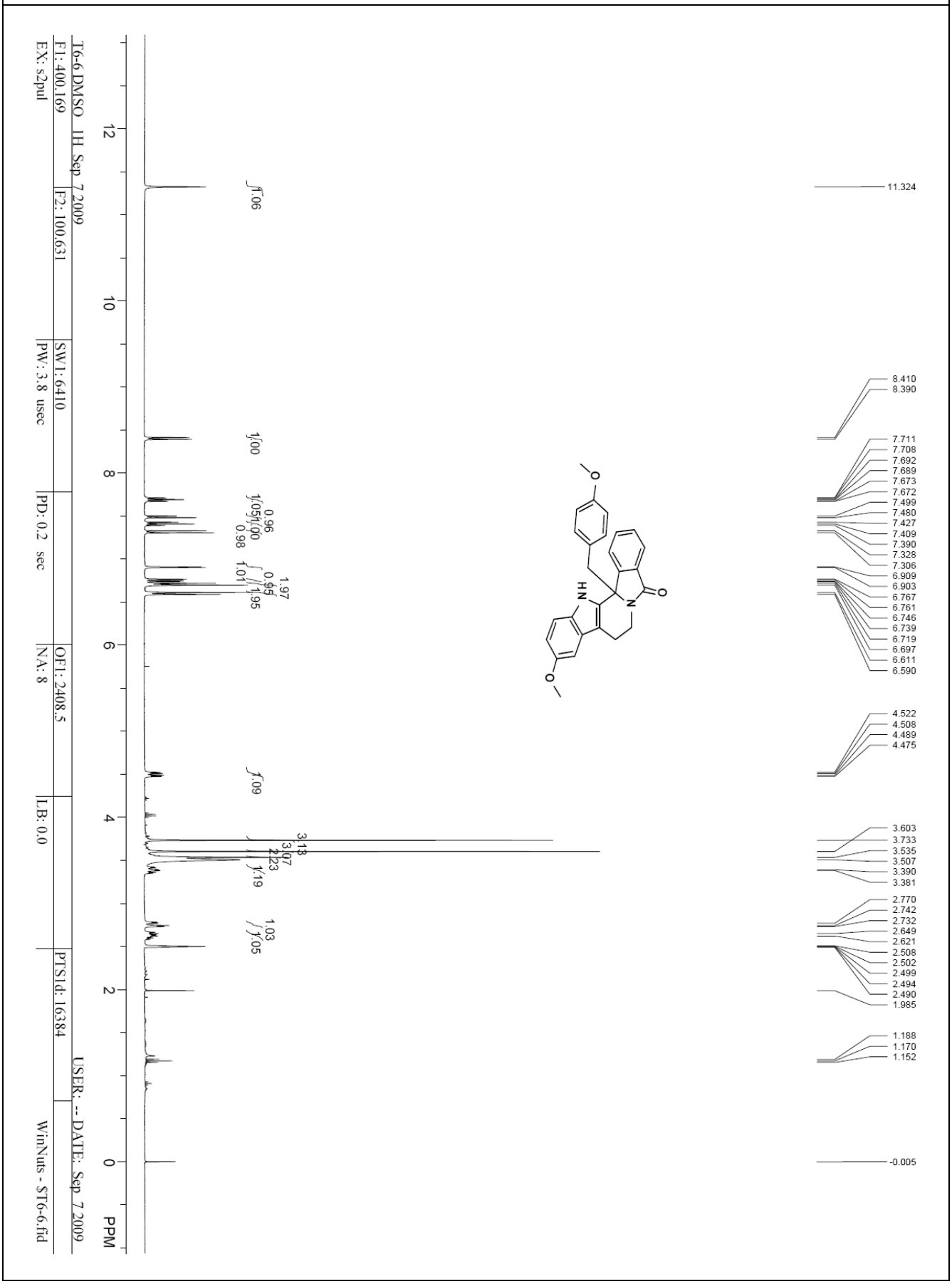

T6-4 DMSO-d6 BB+DEPT-135 Sep 9 2009
167.561
153.378
146.947
135.096
134.982
131.252
131.029
129.767
128.423
127.531
126.328
123.313
122.812
112.022
111.676
106.848
100.240
65.552
55.345
43.021
40.124
39.919
39.710
39.290
39.081
38.876
35.660
21.564
20.772
180
160
140
120
100
80
60
40
20
ppm

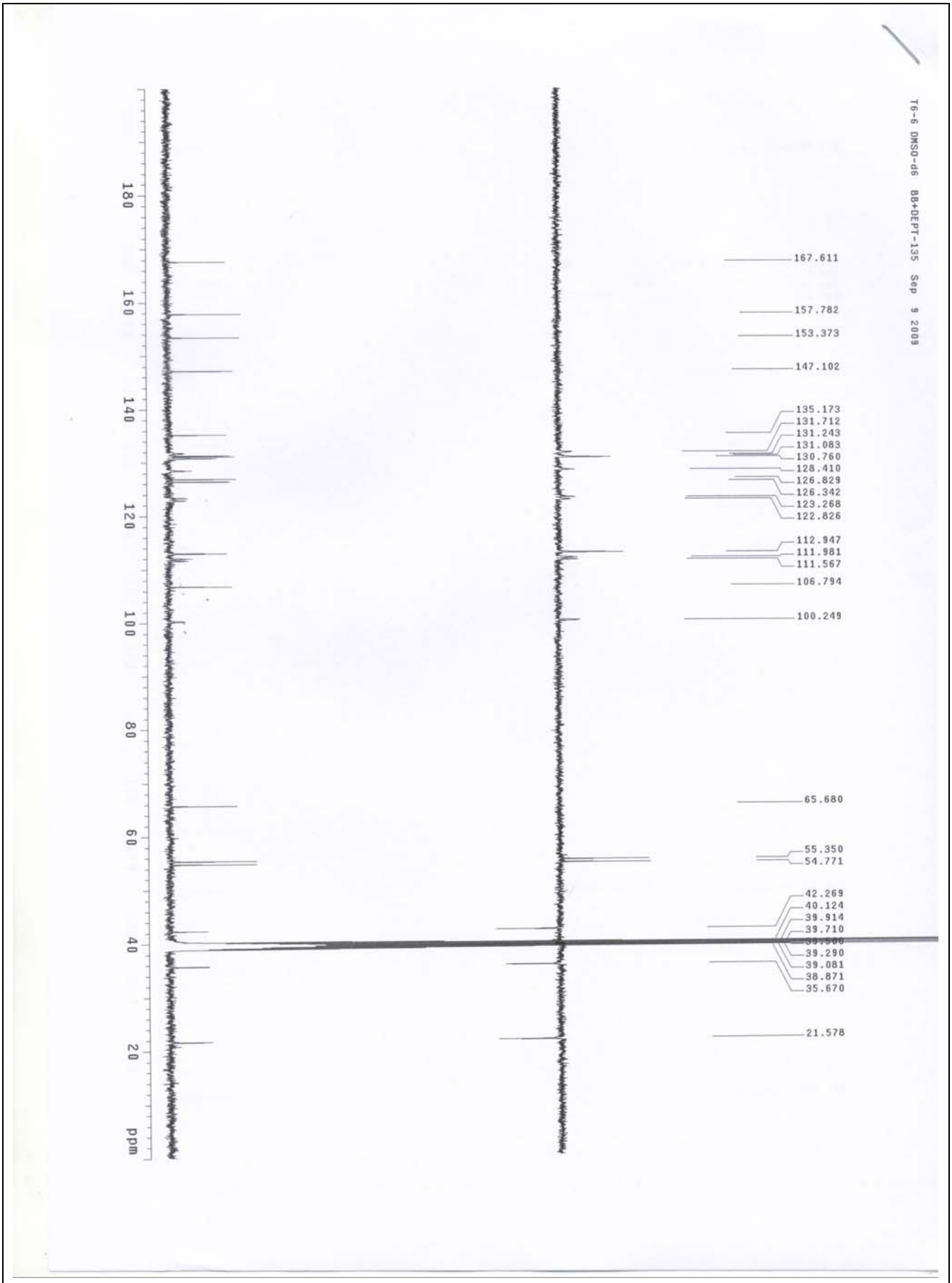
Compound 10Br



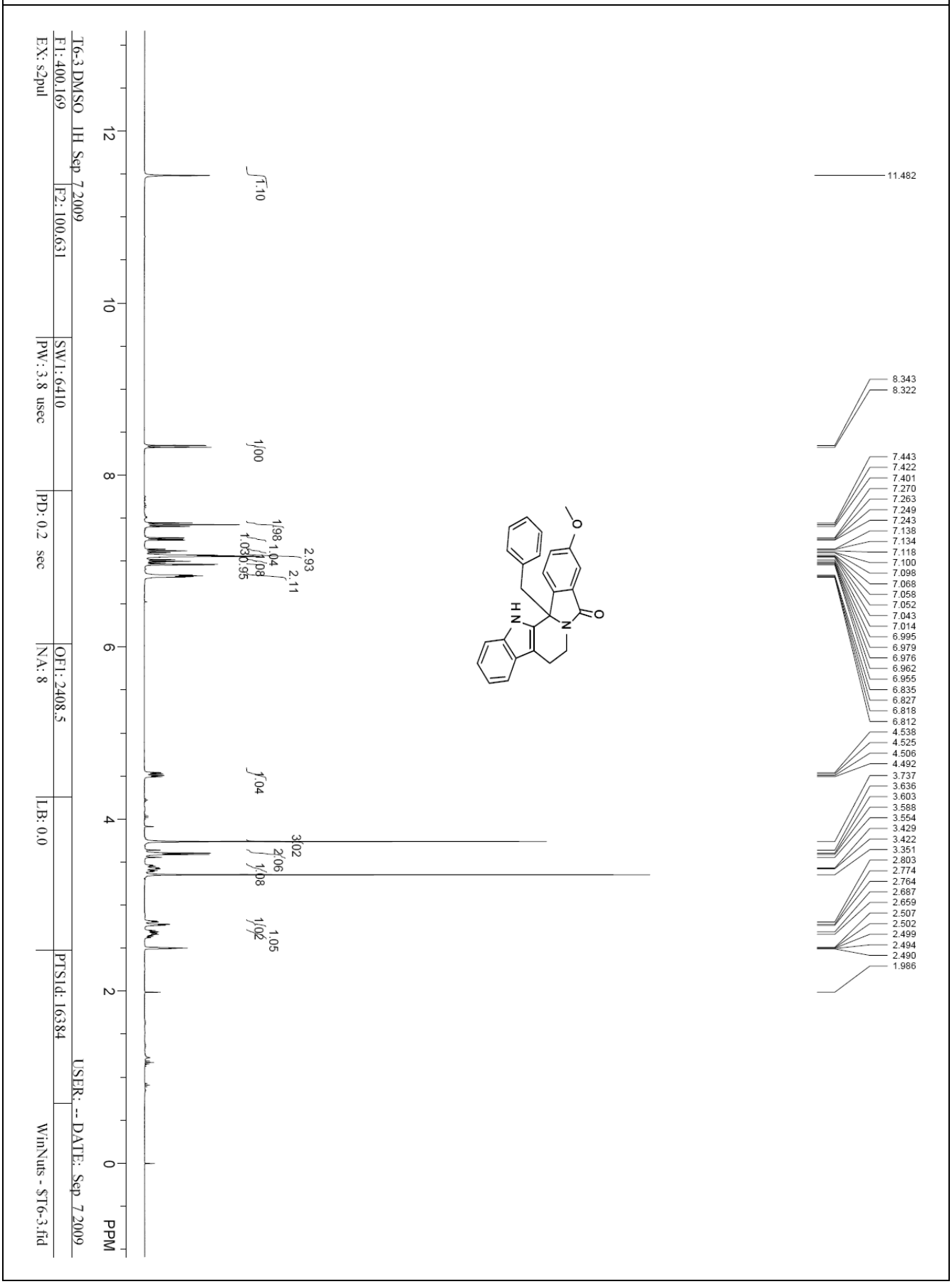

T6-2 DMSO-d6 BB+DEPT-135 Sep 9 2009
167.638
157.796
147.047
136.230
134.590
131.662
131.106
130.769
128.455
126.802
125.991
123.331
122.840
121.664
118.950
118.381
112.956
111.307
106.949
65.661
54.771
42.183
40.124
39.919
39.710
39.500
39.290
39.081
38.876
35.642
21.500
20.776
180
160
140
120
100
80
60
40
20
ppm

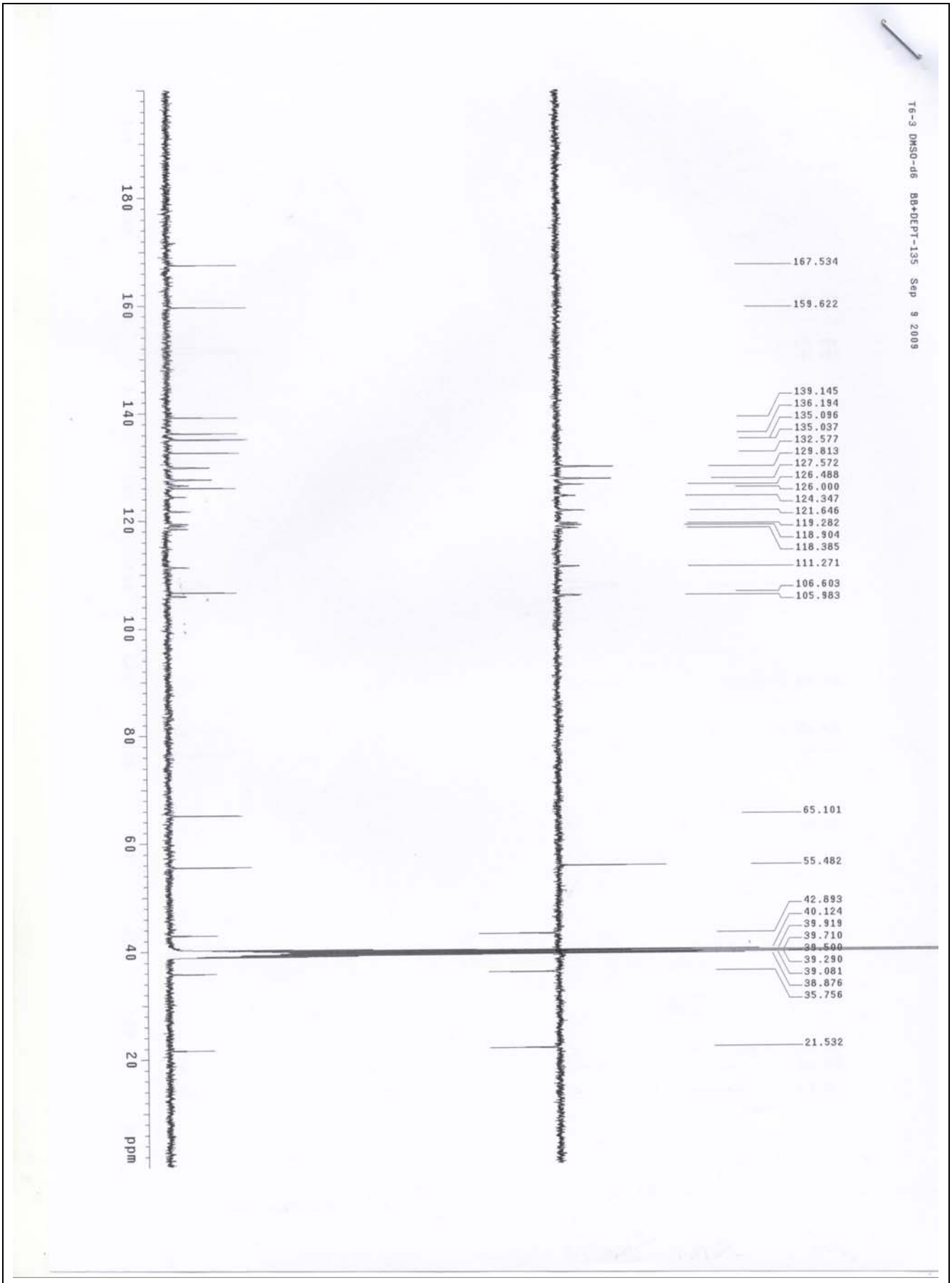
Compound 10Bs



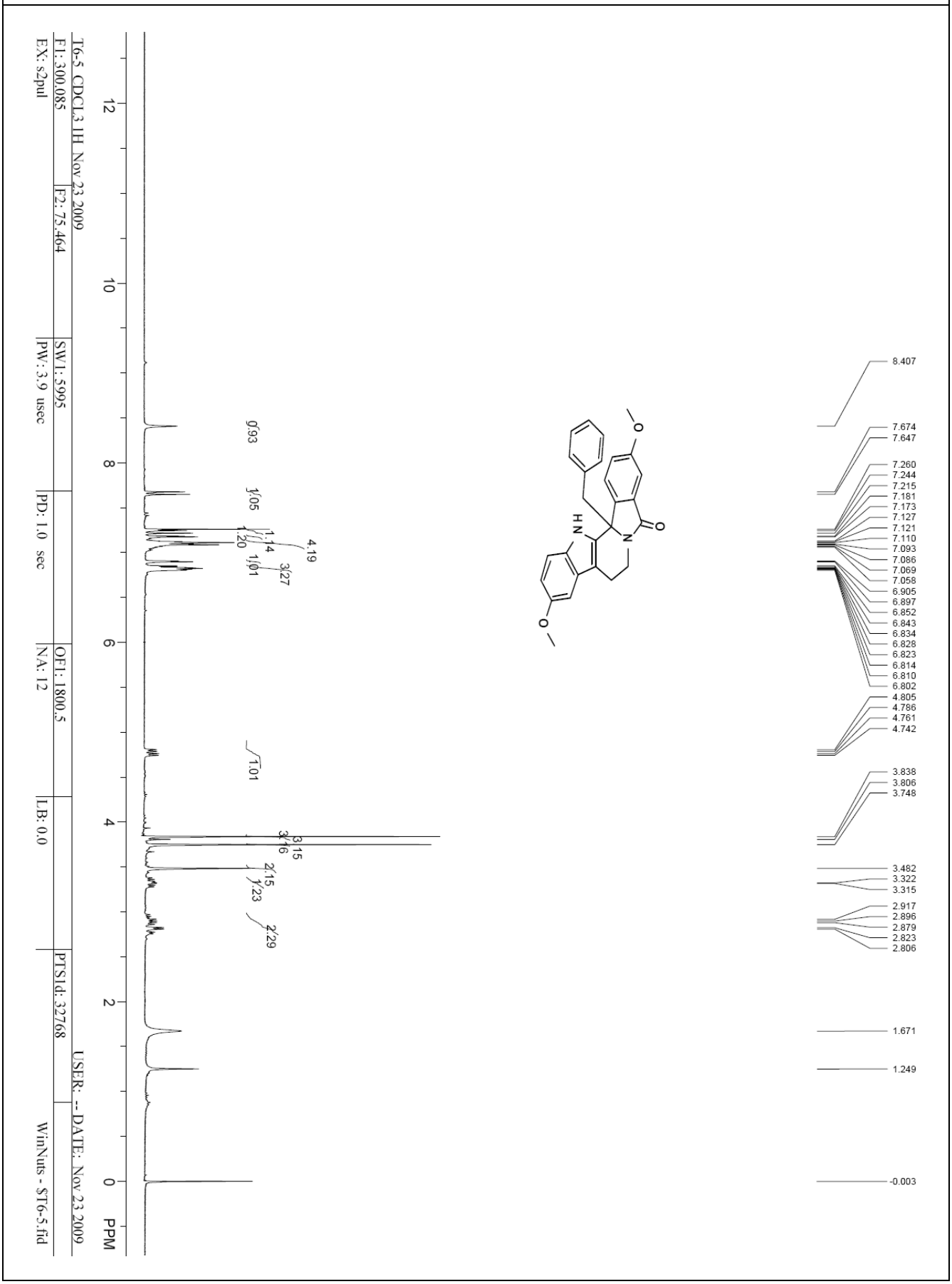

T6-6 DMSO-d6 BB+DEPT-135 Sep 9 2009
167.611
157.782
153.373
147.102
135.173
131.712
131.243
131.083
130.760
128.410
126.829
126.342
123.268
122.826
112.947
111.981
111.567
106.794
100.249
65.680
55.350
54.771
42.269
40.124
39.914
39.710
39.290
39.081
38.871
35.670
21.578
180
160
140
120
100
80
60
40
20
ppm

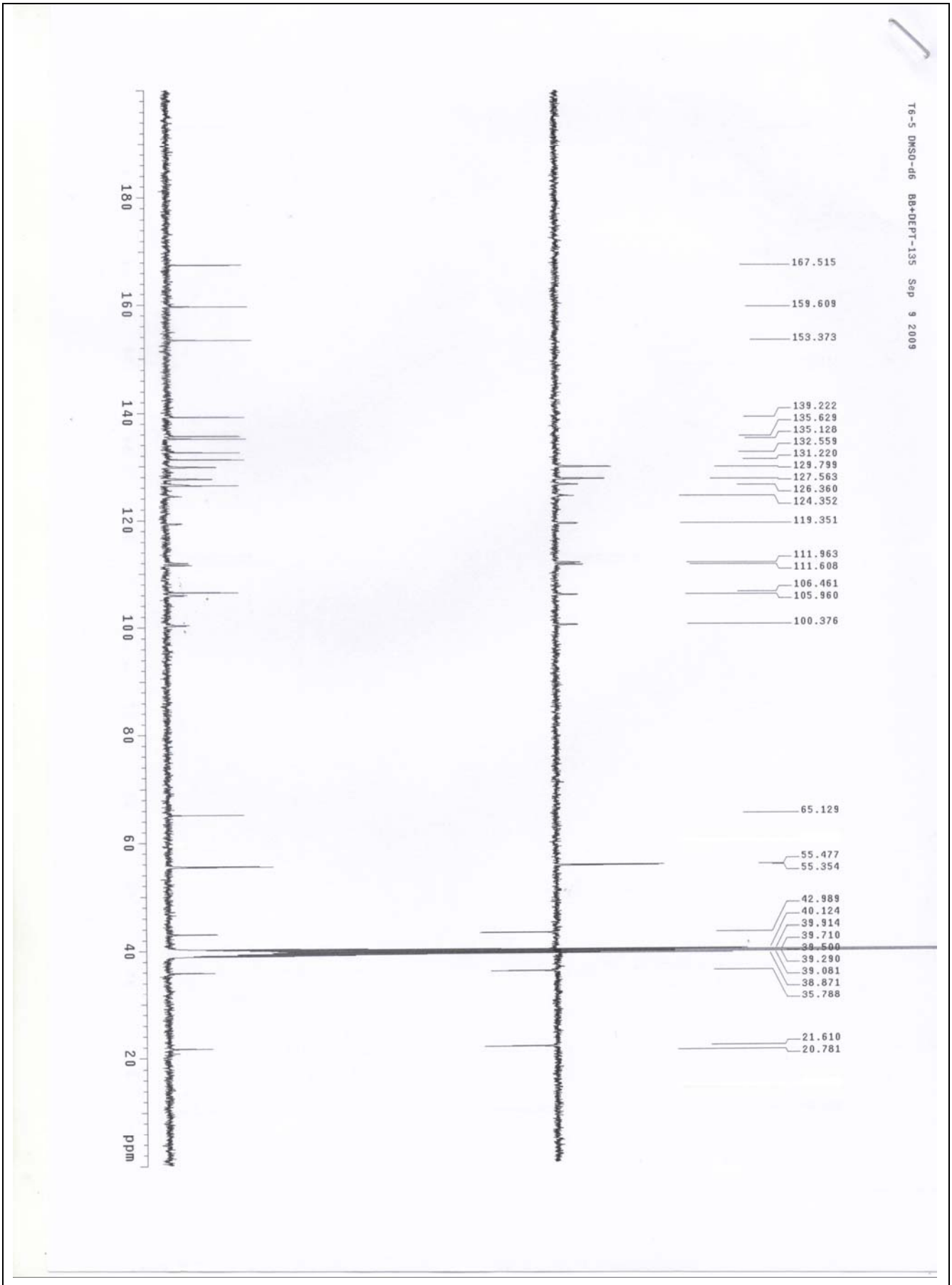
Compound 10Er



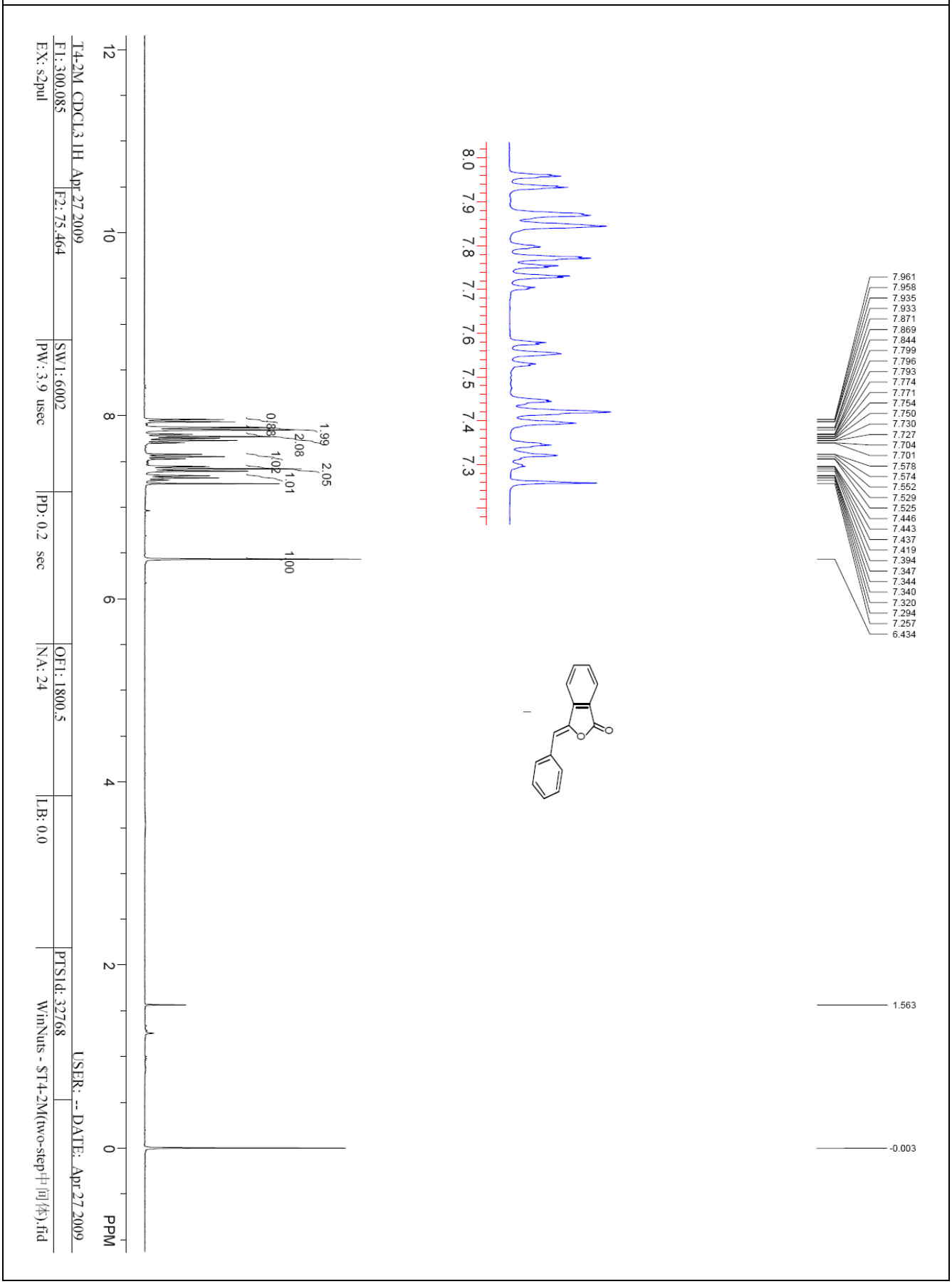

T6-3 DMSO-d6 BB+DEPT-135 Sep 9 2009
167.534
159.622
139.145
136.194
135.096
135.037
132.577
129.813
127.572
126.488
126.000
124.347
121.646
119.282
118.904
118.385
111.271
106.603
105.983
65.101
55.482
42.893
40.124
39.919
39.710
39.500
39.290
39.081
38.876
35.756
21.532
180
160
140
120
100
80
60
40
20
ppm

Compound 10Es

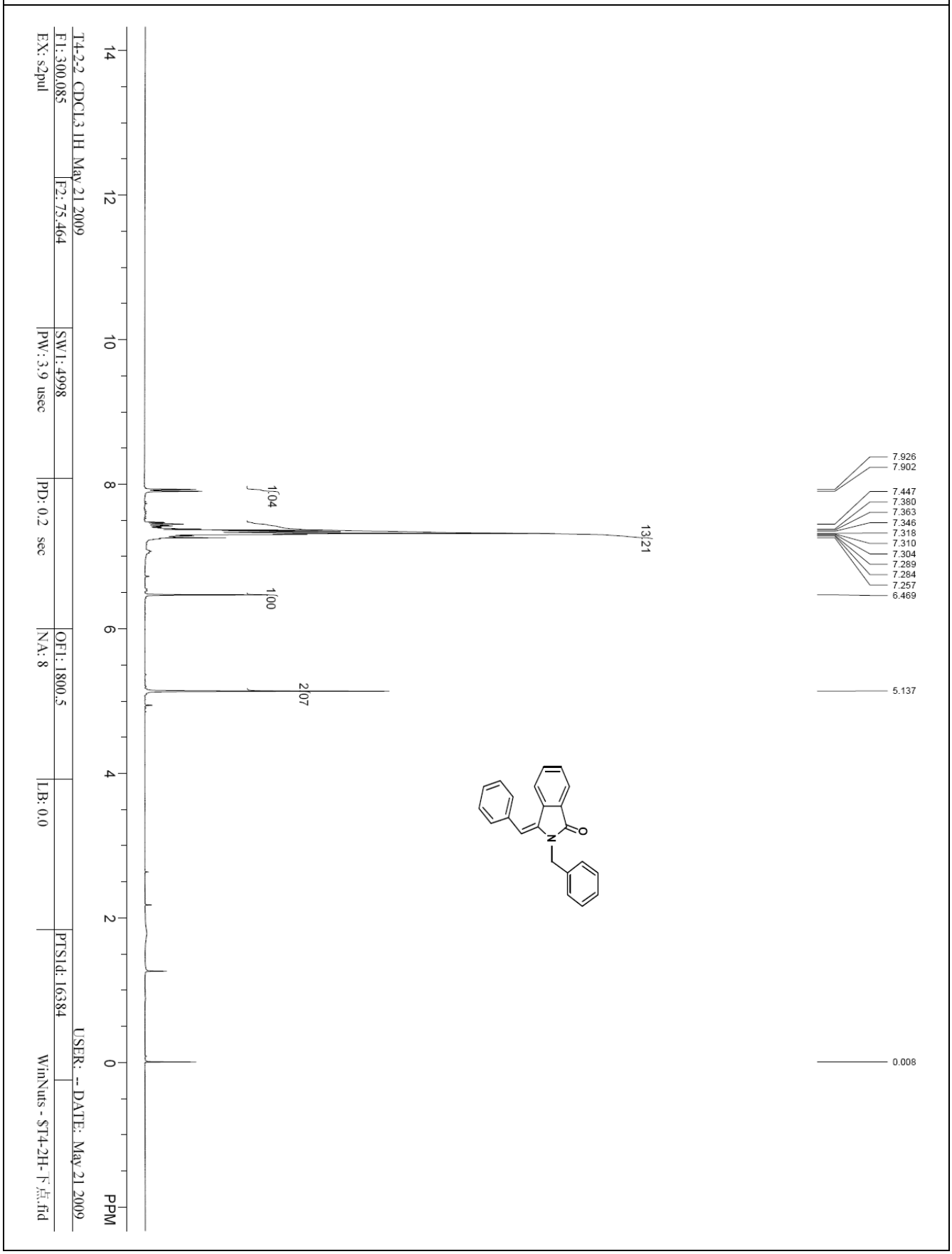



T6-5 DMSO-d6 BB+DEPT-135 Sep 9 2009
167.515
159.609
153.373
139.222
135.629
135.128
132.559
131.220
129.799
127.563
126.360
124.352
119.351
111.963
111.608
106.461
105.960
100.376
65.129
55.477
55.354
42.989
40.124
39.914
39.710
39.500
39.290
39.081
38.871
35.788
21.610
20.781
180
160
140
120
100
80
60
40
20
ppm

Intermediate **M1**



Dehydration product (*E*)-11



Dehydration product (*Z*)-11