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Squaramide–catalysed asymmetric cascade reactions of 2,3-dioxopyrrolidines with 3-chlorooxindoles

Jiang-Bo Wen and Da-Ming Du*

School of Chemistry and Chemical Engineering, Beijing Institute of Technology, Beijing 100081, People's Republic of China
dudm@bit.edu.cn

Supporting Information

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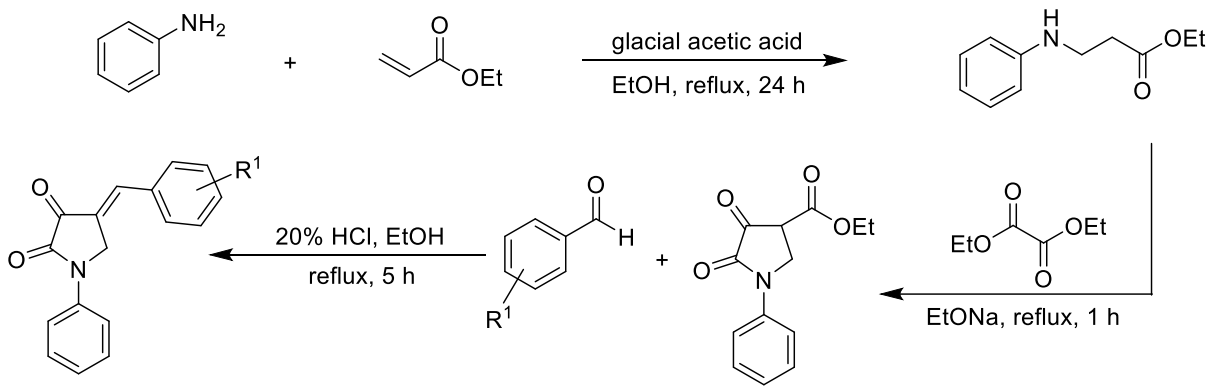
1. General information and starting materials

General information. All solvents commercially and available chemicals were used without further purification. The column chromatography was performed with silica gel (200−300 mesh) using mixtures of petroleum ether and ethyl acetate. Melting points were determined with a XT-4 melting-point apparatus without corrected. ^{1}H NMR spectra were measured with Bruker Ascend 400 MHz spectrometer, and chemical shifts were reported in δ (ppm) units relative to tetramethylsilane (TMS) as the internal standard. ^{13}C NMR spectra were measured at 100 MHz with Bruker Ascend 400 MHz spectrometer and chemical shifts are reported in δ (ppm) units relative to tetramethylsilane and referenced to the solvent peak ($CDCl_3$, δ(C) = 77.00 ppm, acetone-d6 δ(C) = 30.83 ppm, DMSO-d6 δ(C) = 39.43 ppm). ^{19}F NMR spectra were measured at 376 MHz with Bruker Ascend 400 MHz spectrometer. Proton coupling patterns are described as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), and broad (br). High-resolution mass spectra were obtained with an Agilent 6520 Accurate-Mass-Q-TOF MS system equipped with an electrospray ionization (ESI) source. The enantiomeric excesses were determined by chiral HPLC analysis using an Agilent 1200 LC instrument with Daicel Chiralpak IA, IB, IC or AD-H columns. Optical rotations were measured with Krüss P8000 polarimeter at the indicated concentration with the units of grams per 100 mL at 20 °C using sodium D light.

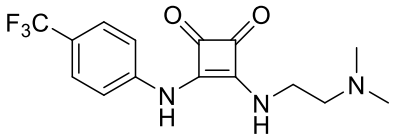
Starting materials. 1a−1t were prepared according to the literature.[1]



2a−2i were prepared according to the literature.[2] The squaramide organocatalysts were prepared by following the reported procedures. [3]

2. General procedure for the synthesis of compound 3

To a dried small bottle were added 2,3-dioxopyrrolidines **1** (0.1mmol), 3-chlorooxindoles **2** (0.12 mmol), squaramide catalyst **C1** (5 mol%) and NH_4HCO_3(0.1 mmol) in 1.0 mL of AcOEt at 0°C. The reaction mixture was stirred for 2 h and the progress of the reaction was monitored by TLC analysis (Petroleum ether/ ethyl acetate = 1:1). After the completion of the reaction, the crude product mixture was purified by flash column chromatography on silica (petroleum ether/ethyl acetate = 4:1) to afford the pure product **3**. The racemic standard of **3** was prepared using achiral catalyst **C16**.[4]



C16

References

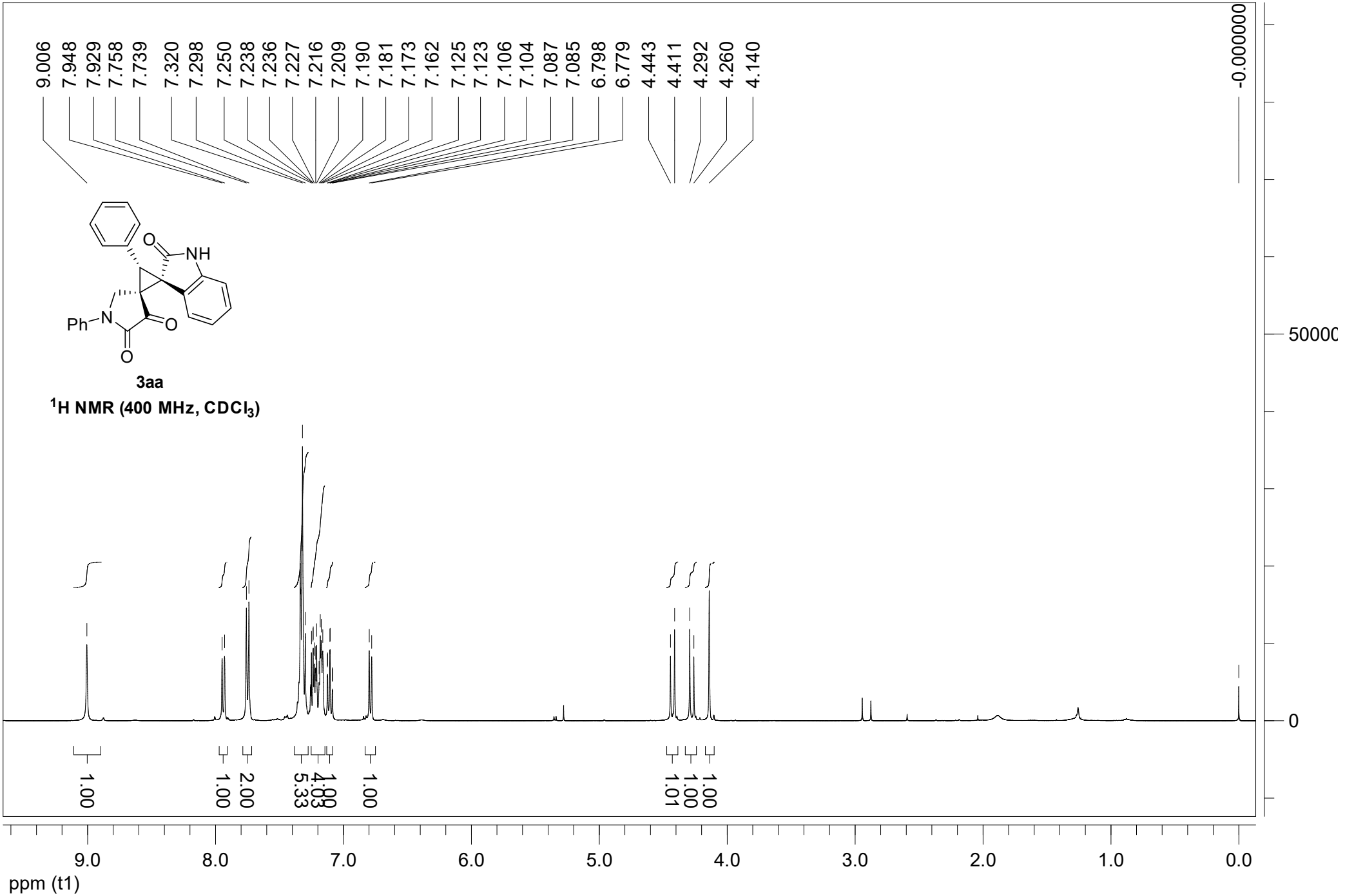
[1] X. Chen, L. Zhu, L. Fang, S. Yan and J. Lin, RSC Adv. **2014**, 4, 9926.

[2] a) J. Guillaumel, P. Demerseman, J.-M. Clavel, R. Royer, J. Heterocycl. Chem. **1980**, 17, 1531-1536. b) A. Noole, A. V. Malkov, T. Kanger, Synthesis **2013**, 45, 2520-2524. c) A. Noole, M. Ošeka, T. Pehk, M. Öeren, I. Järving, M. R. J. Elsegood, A. V. Malkov, M. Lopp, T. Kanger, Adv. Synth. Catal. **2013**, 355, 829-835. d) S. Paladhi, S. Y. Park, J. W. Yang, C. E. Song, Org. Lett. **2017**, 19, 5336-5339.

[3] a) Y. Zhu, J. P. Malerich, V. H. Rawal, Angew. Chem., Int. Ed. **2010**, 49, 153−156; Angew. Chem. 2010, 122, 157−160. b) W. Yang, D. M. Du, Org. Lett. **2010**, 12, 5450−5453. c) W. Yang, D. M. Du, Adv. Synth. Catal. **2011**, 353, 1241−1246.

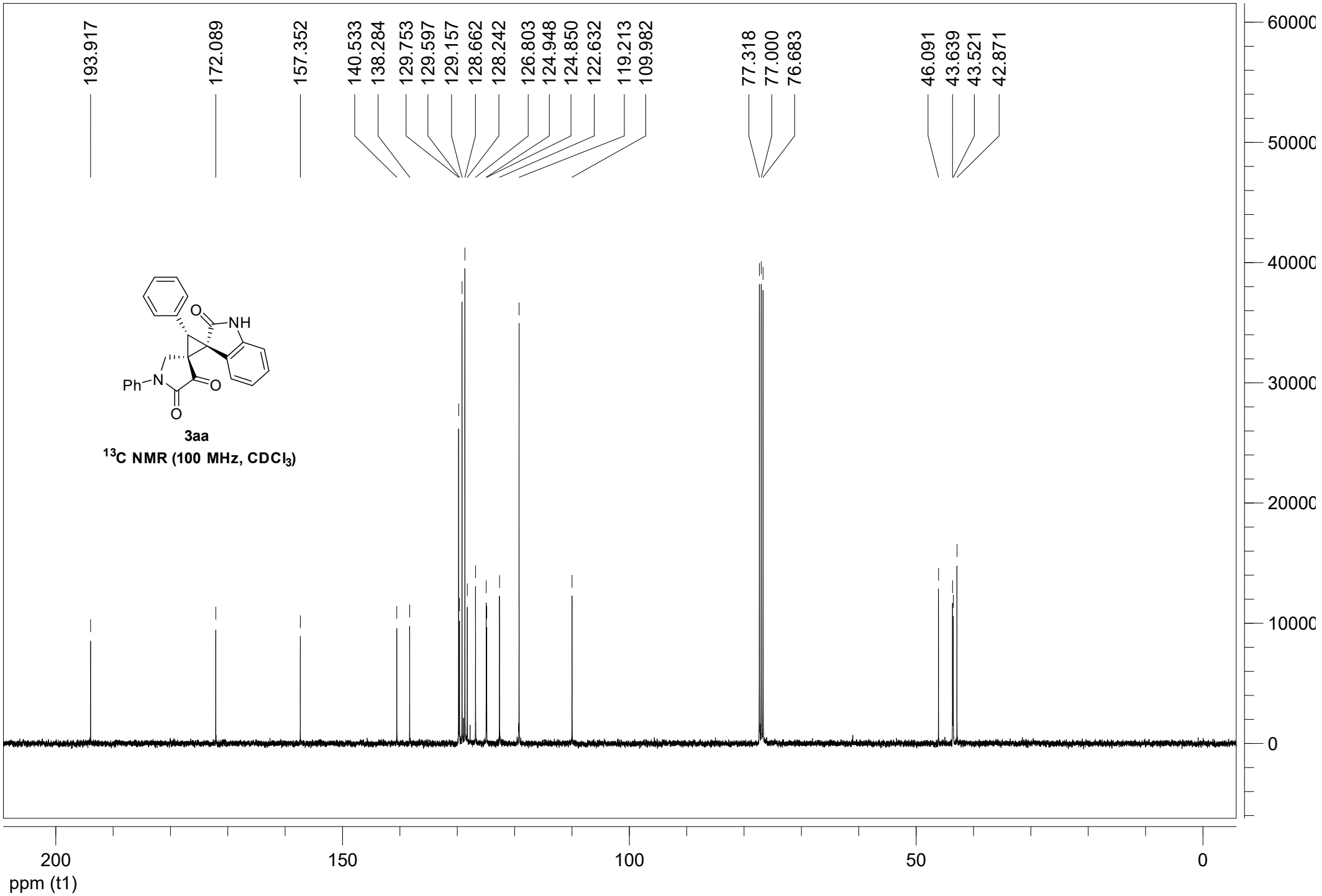
[4] S. Sopeña, E. Martin, E. -C. Escuder and A. -W. Kleij, ACS Catal. **2017**, 7, 3532.

3. Copies of ^{1}H, ^{13}C and ^{19}F NMR spectra of new products



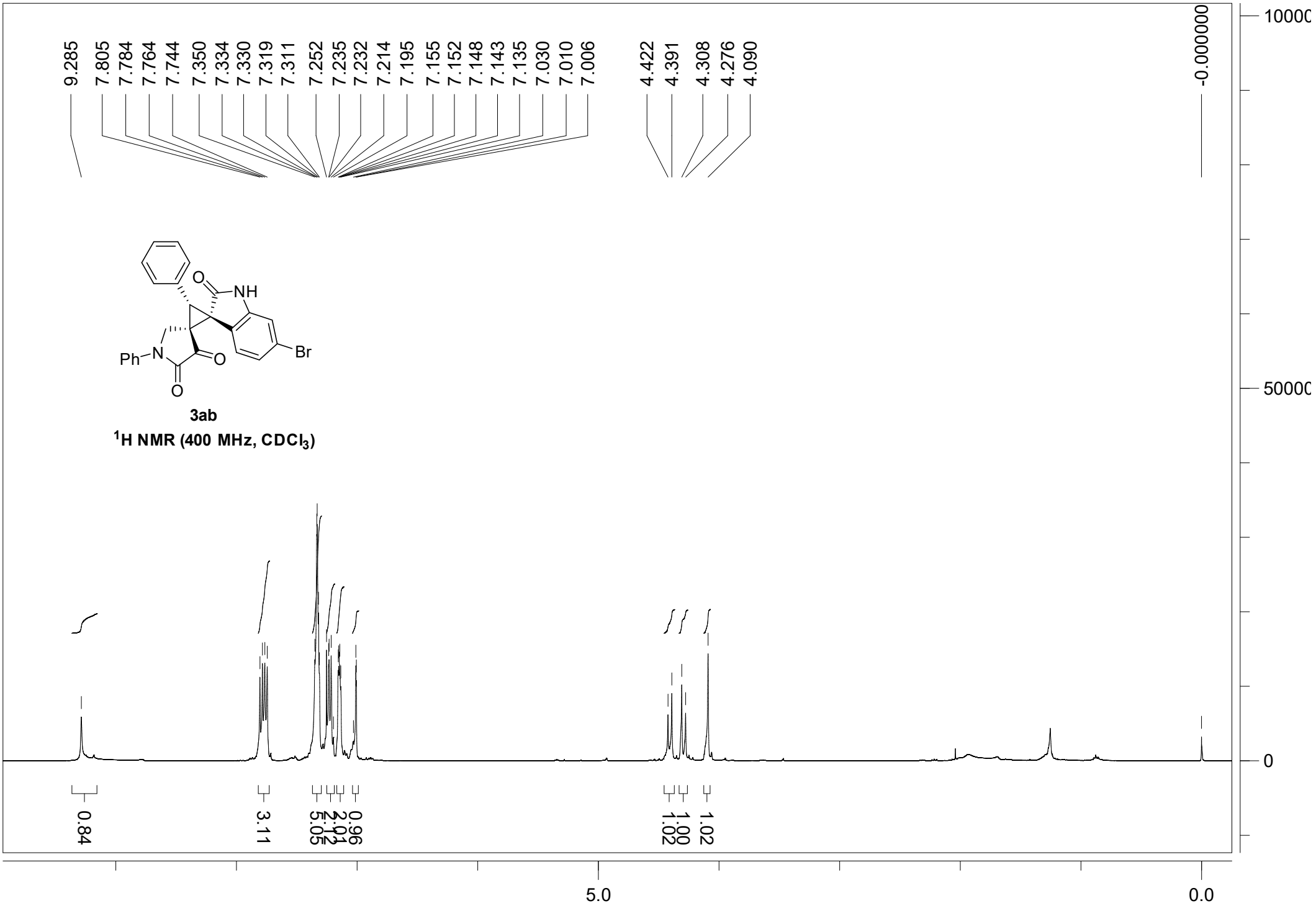
3aa

^{1}H NMR (400 MHz, $CDCl_3$)



3aa

^{13}C NMR (100 MHz, $CDCl_3$)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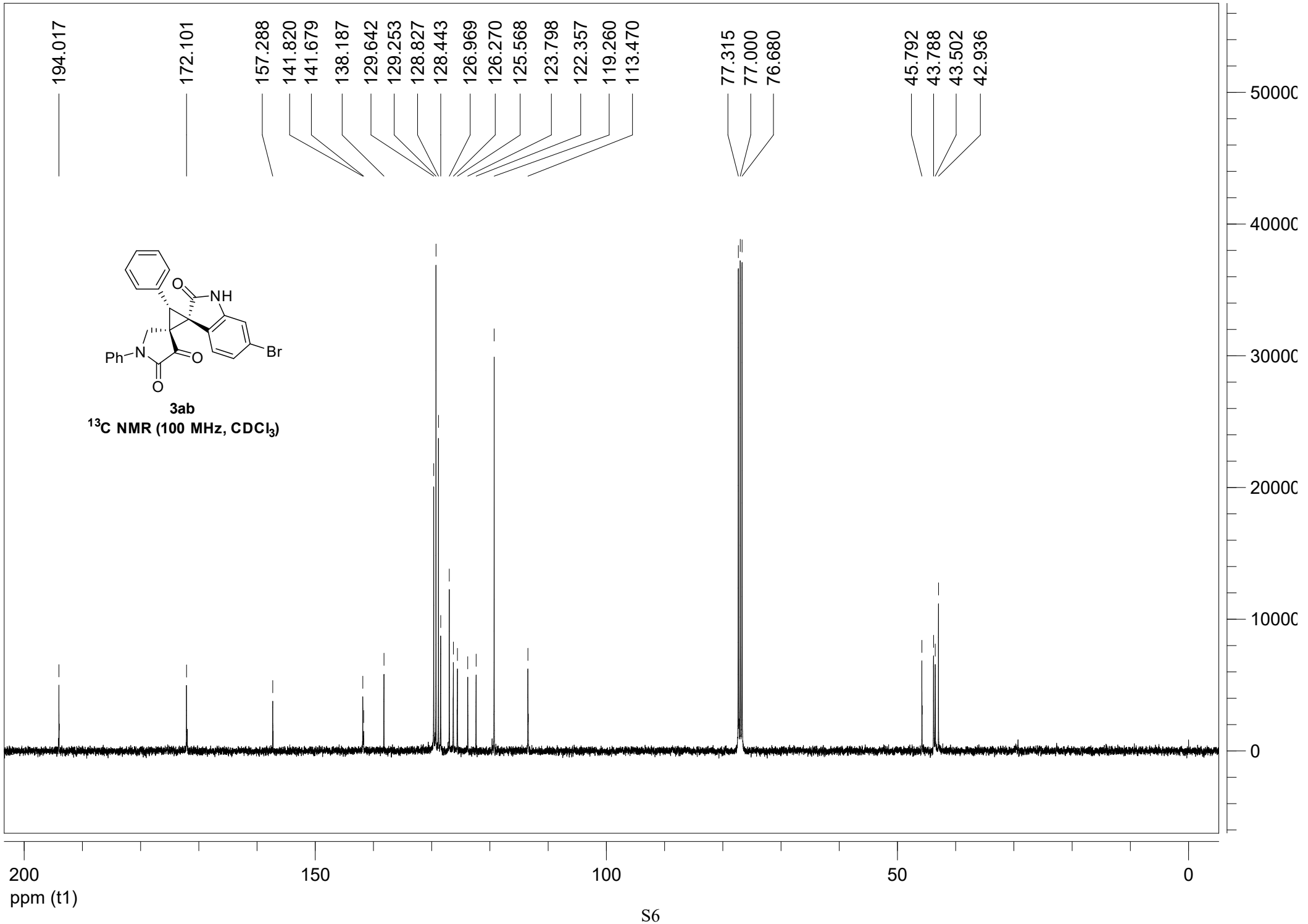
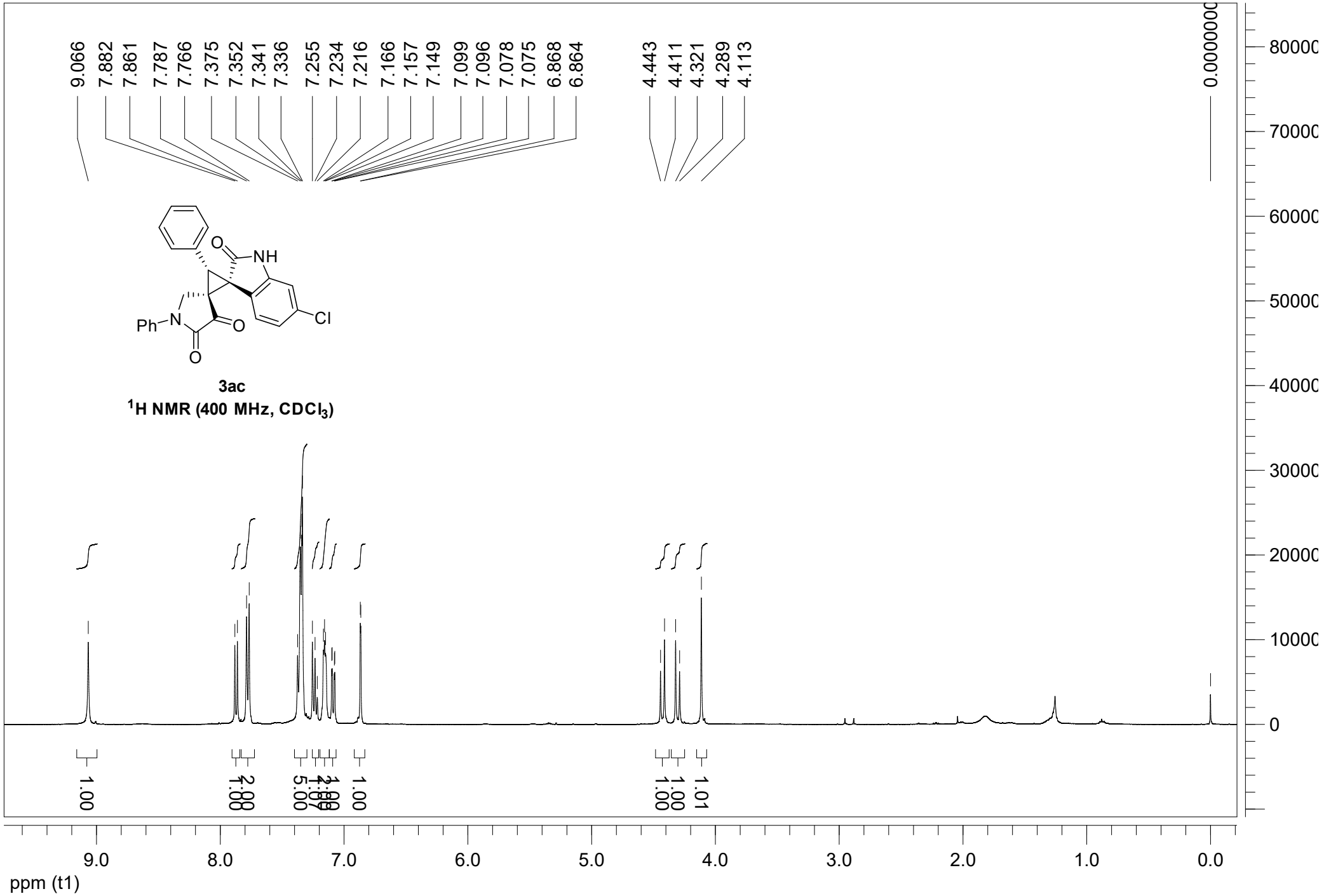
7.805
7.784
7.764
7.744
7.350
7.334
7.330
7.319
7.311
7.252
7.235
7.232
7.214
7.195
7.155
7.152
7.148
7.143
7.135
7.030
7.010
7.006
4.422
4.391
4.308
4.276
4.090
-0.000000
10000
50000
0
O
NH
Ph
N
O
O
Br
3ab
1H NMR (400 MHz, CDCl3)
0.84
3.11
5.05
2.01
2.12
0.96
1.02
1.00
1.02
5.0
0.0
ppm (t1)



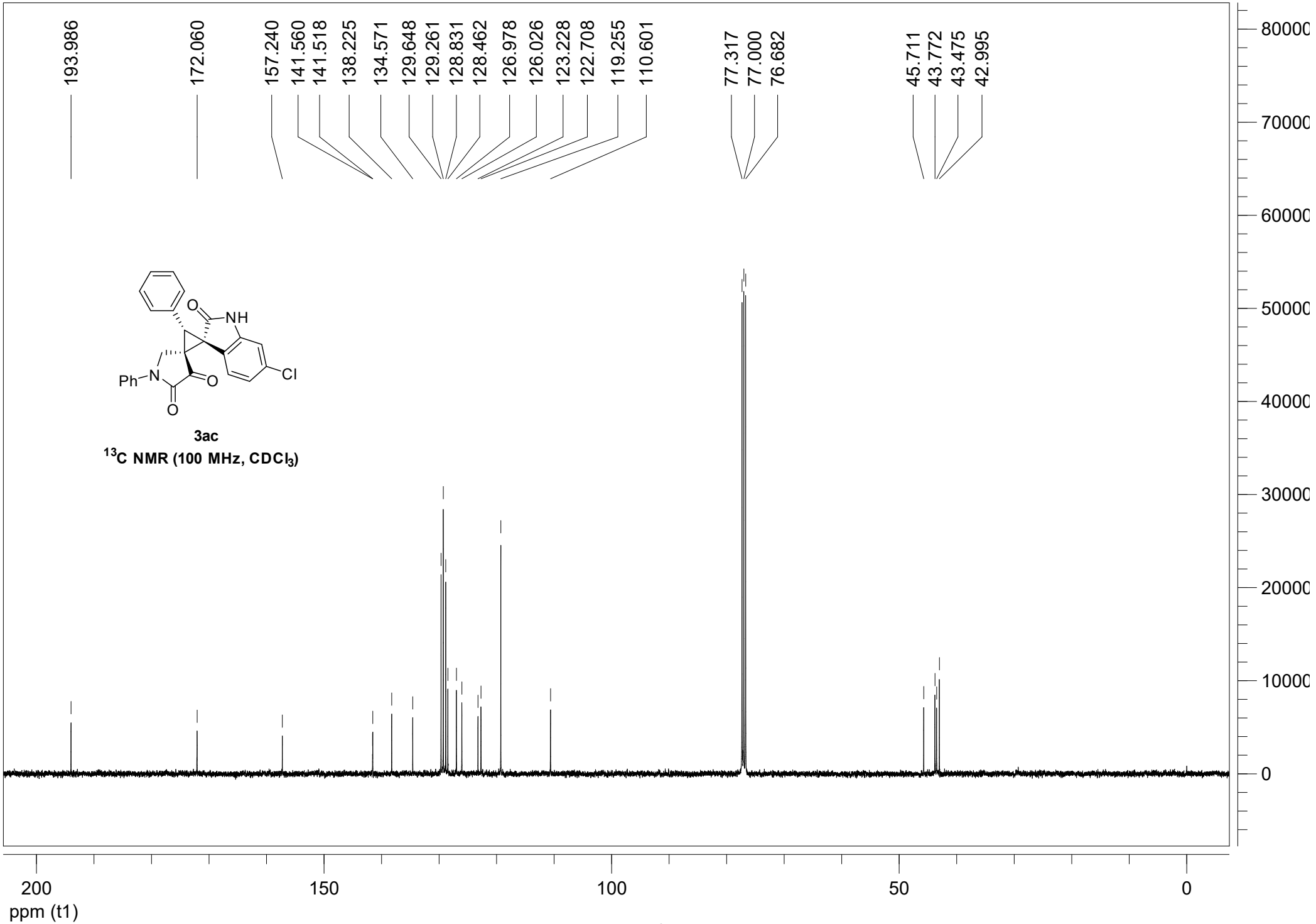
3ab

^{13}C NMR (100 MHz, $CDCl_3$)



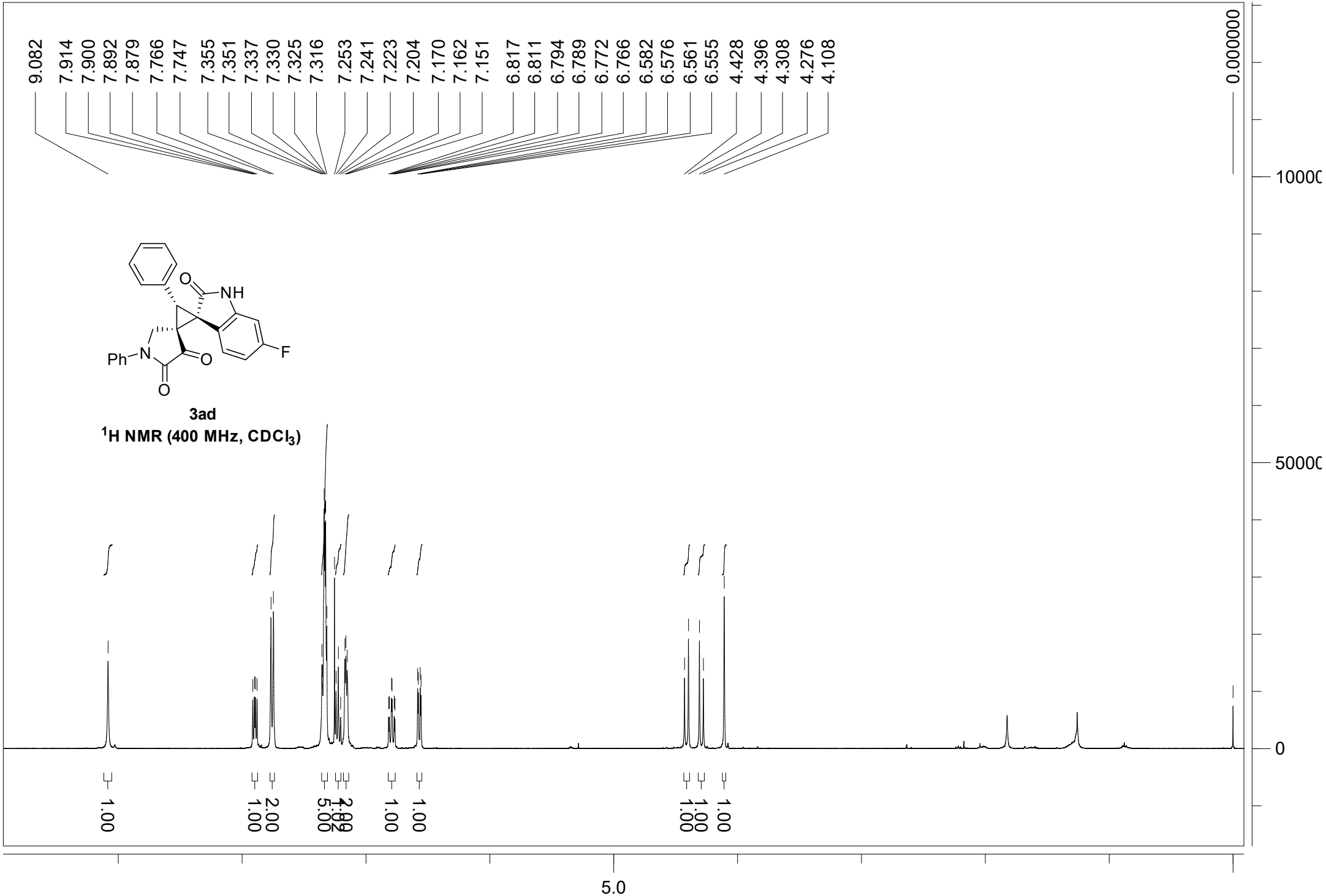
3ac

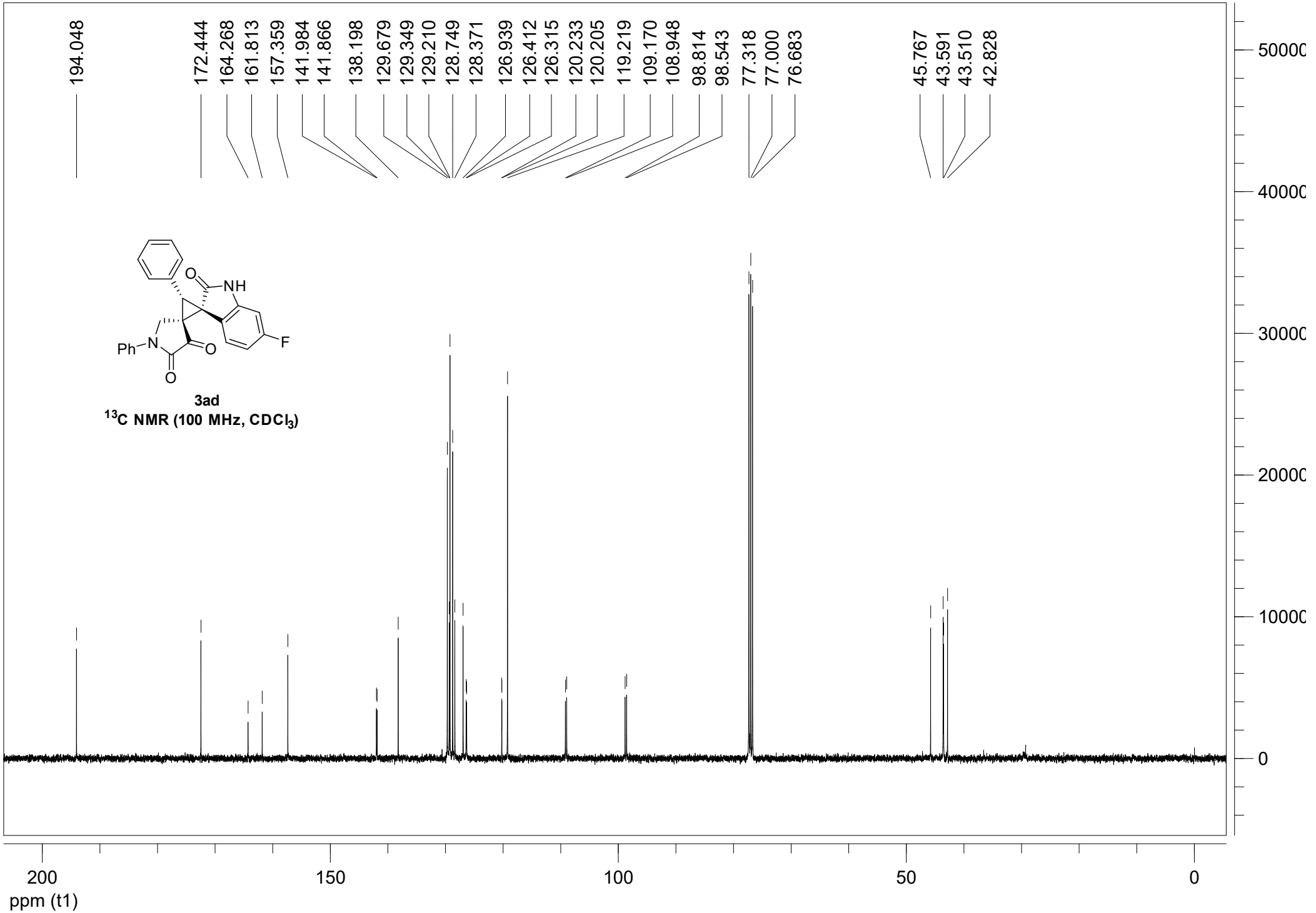
^{1}H NMR (400 MHz, $CDCl_3$)



3ac

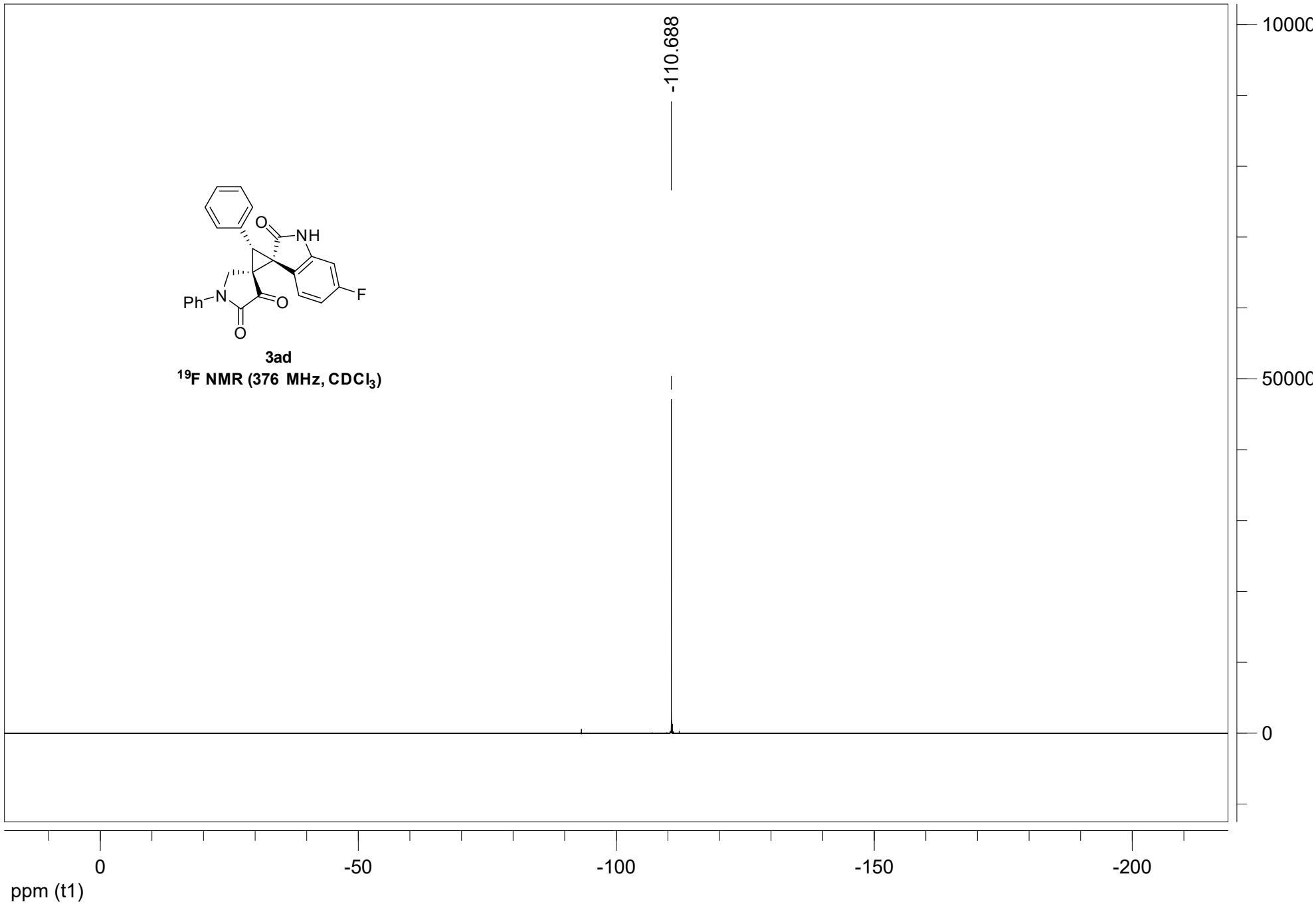
^{13}C NMR (100 MHz, $CDCl_3$)


9.082
7.914
7.900
7.892
7.879
7.766
7.747
7.355
7.351
7.337
7.330
7.325
7.316
7.253
7.241
7.223
7.204
7.170
7.162
7.151
6.817
6.811
6.794
6.789
6.772
6.766
6.582
6.576
6.561
6.555
4.428
4.396
4.308
4.276
4.108
0.000000
10000
50000
0
O
NH
Ph
N
O
O
F
3ad
1H NMR (400 MHz, CDCl3)
1.00
1.00
2.00
5.00
2.00
1.00
1.00
1.00
1.00
1.00
5.0
ppm (t1)



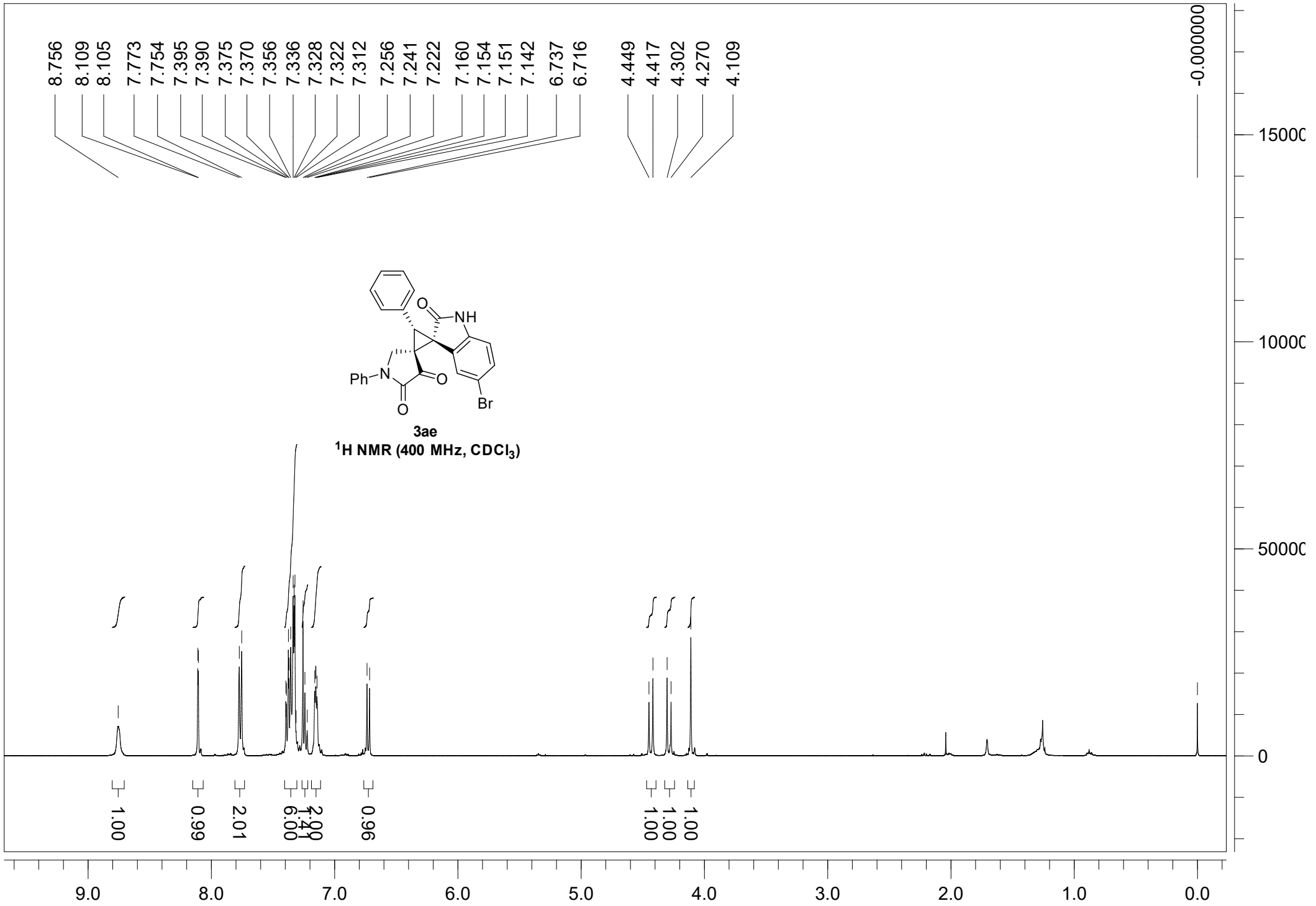
3ad

^{13}C NMR (100 MHz, $CDCl_3$)



3ad

^{19}F NMR (376 MHz, $CDCl_3$)



3ae

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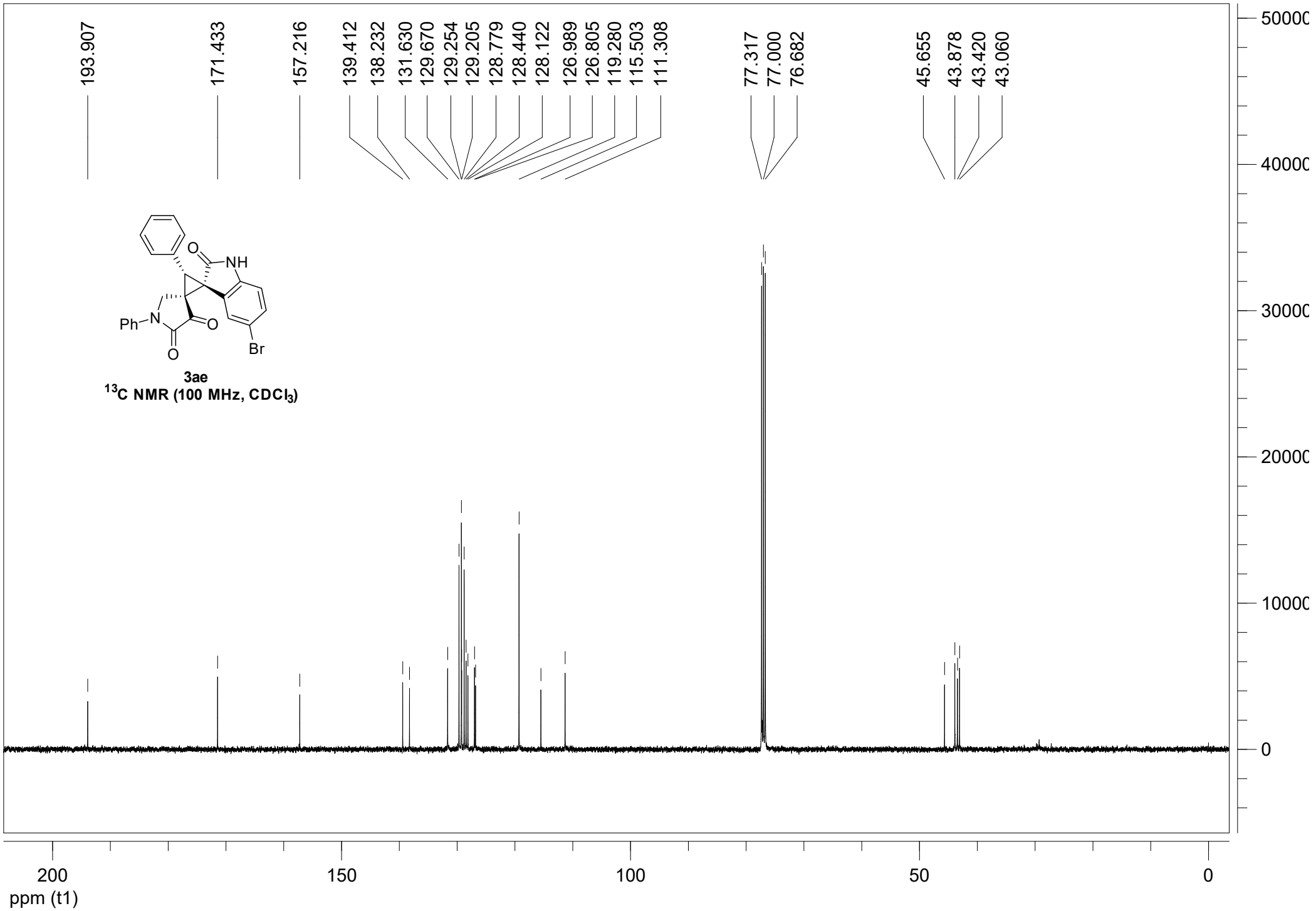
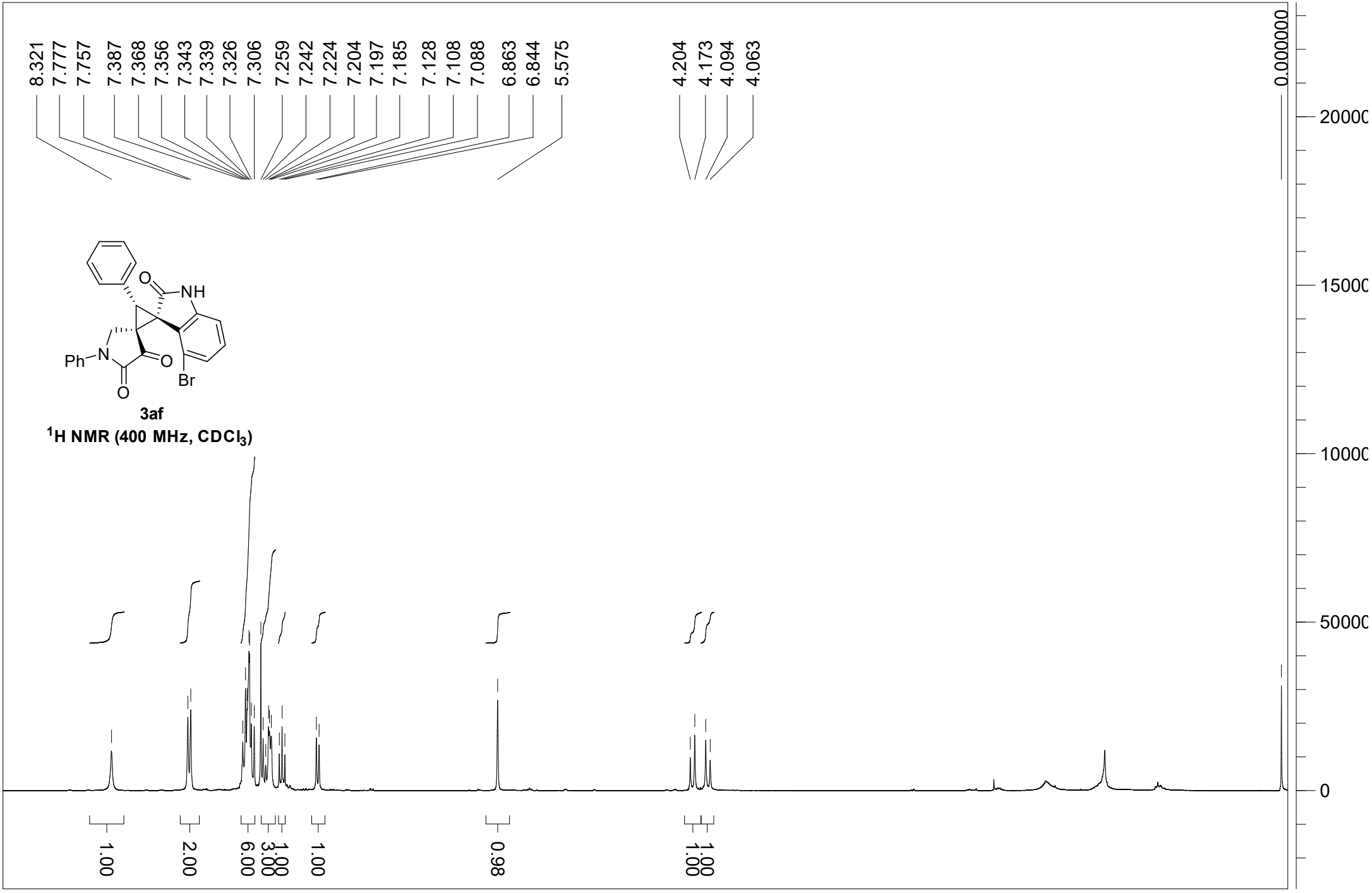


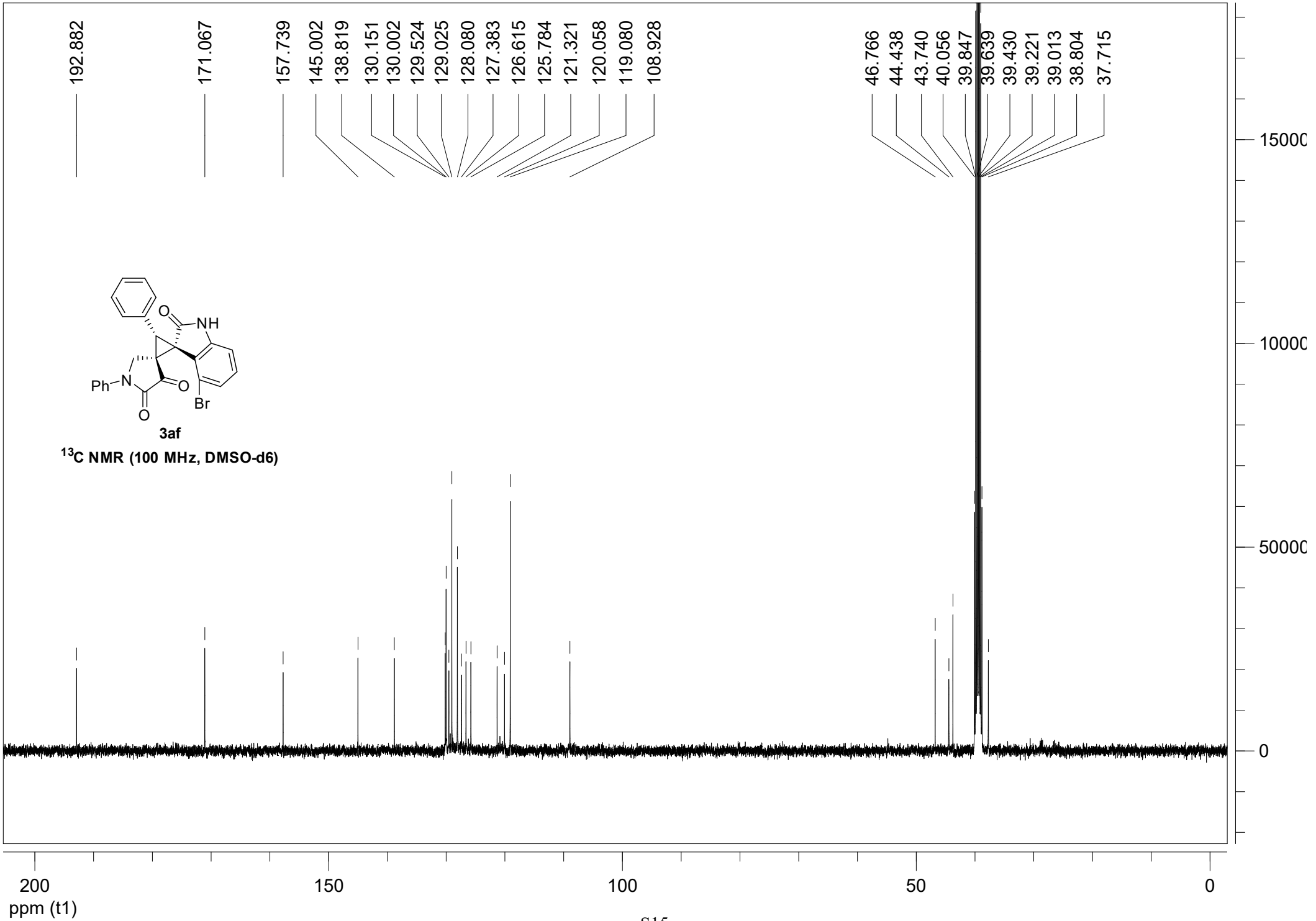
3ae

^{13}C NMR (100 MHz, $CDCl_3$)



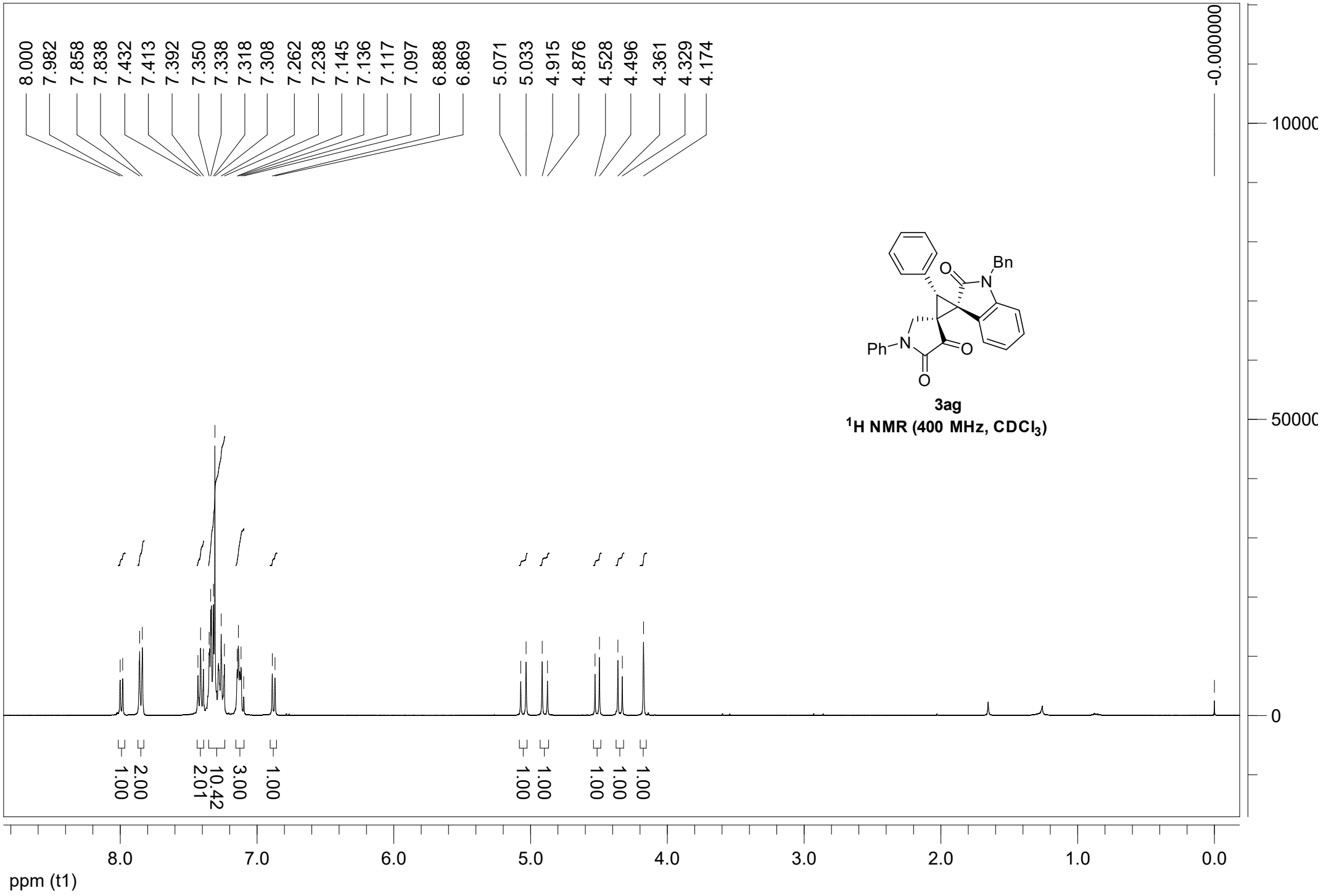
3af

^{1}H NMR (400 MHz, $CDCl_3$)



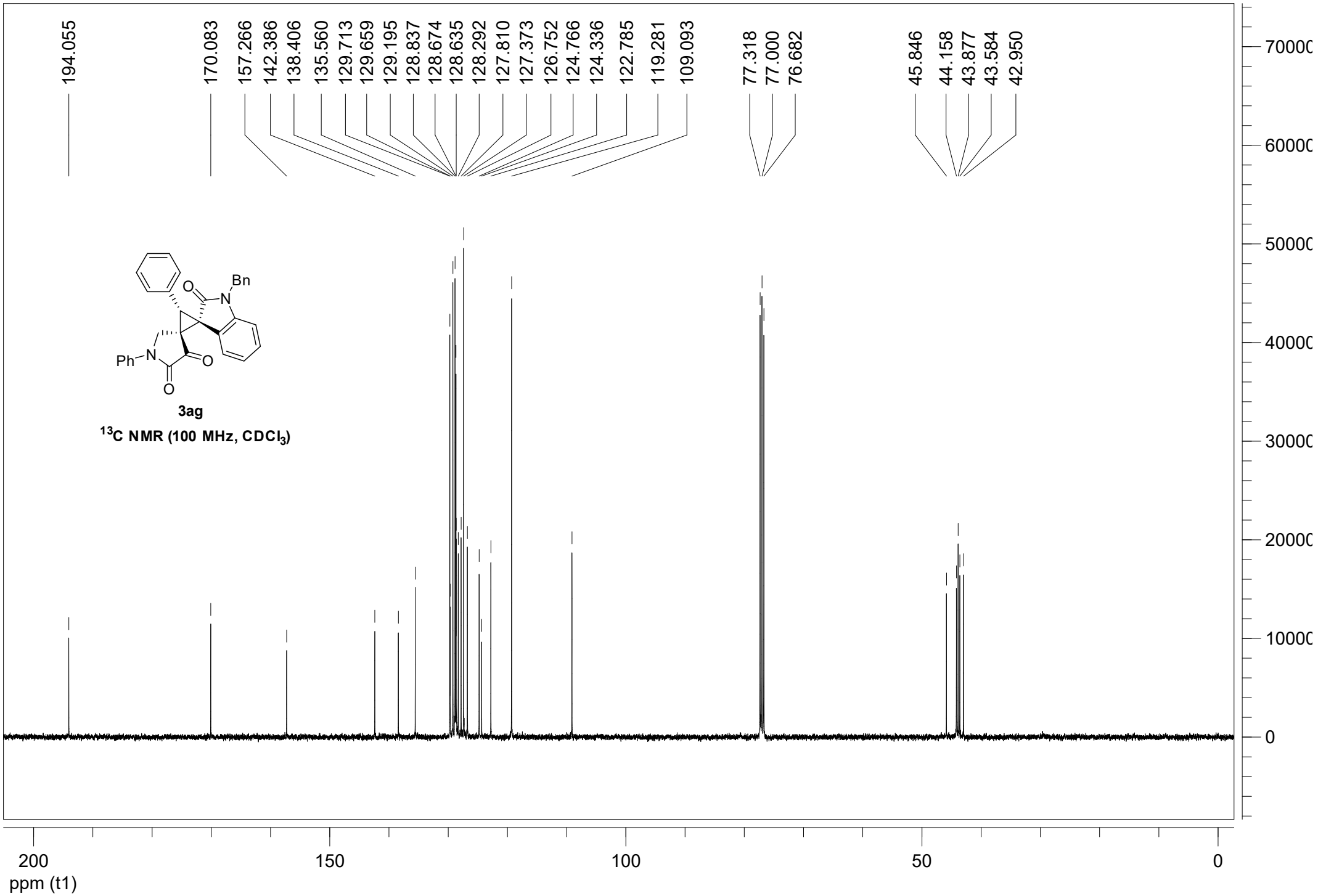
3af

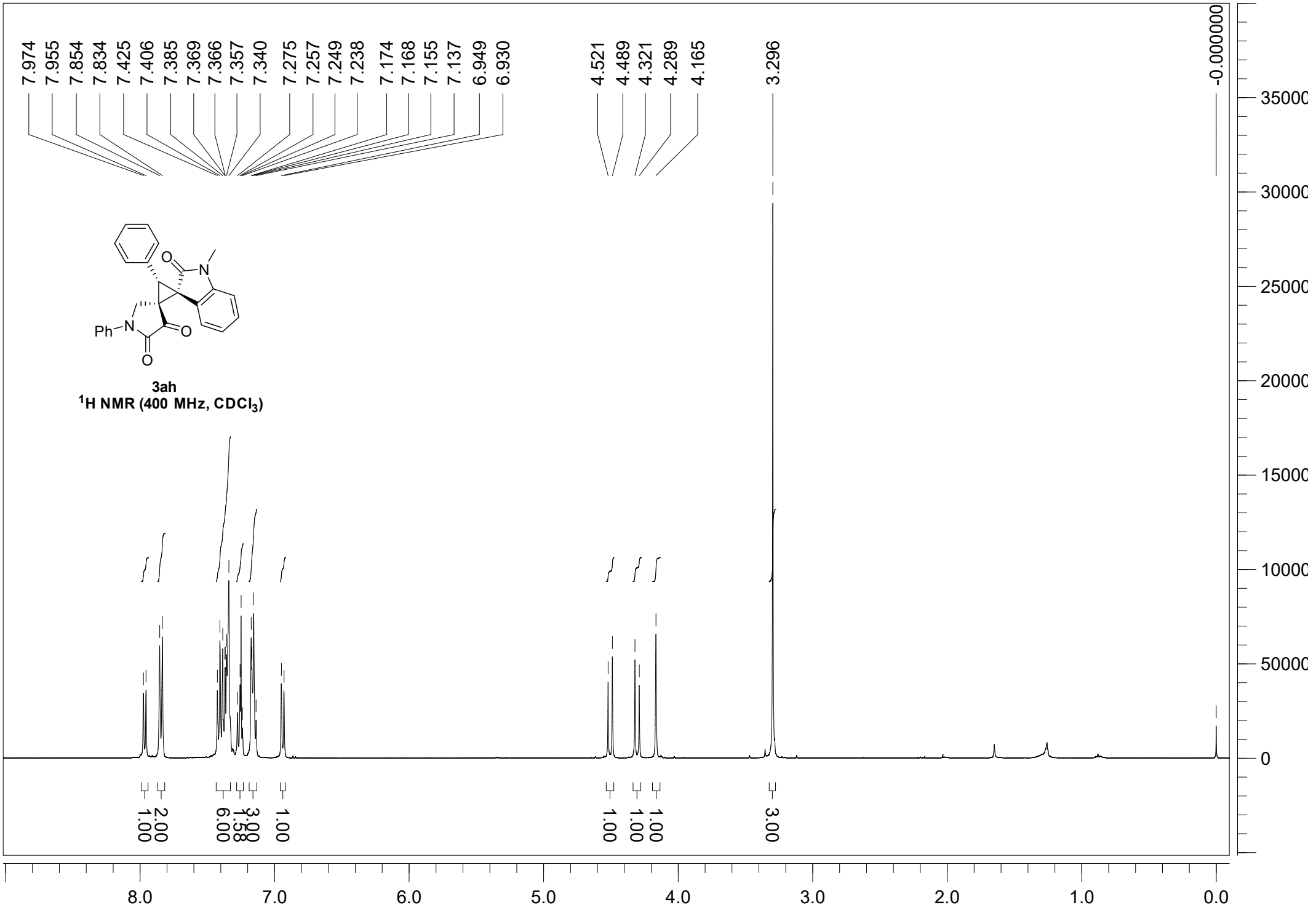
^{13}C NMR (100 MHz, DMSO-d6)



3ag

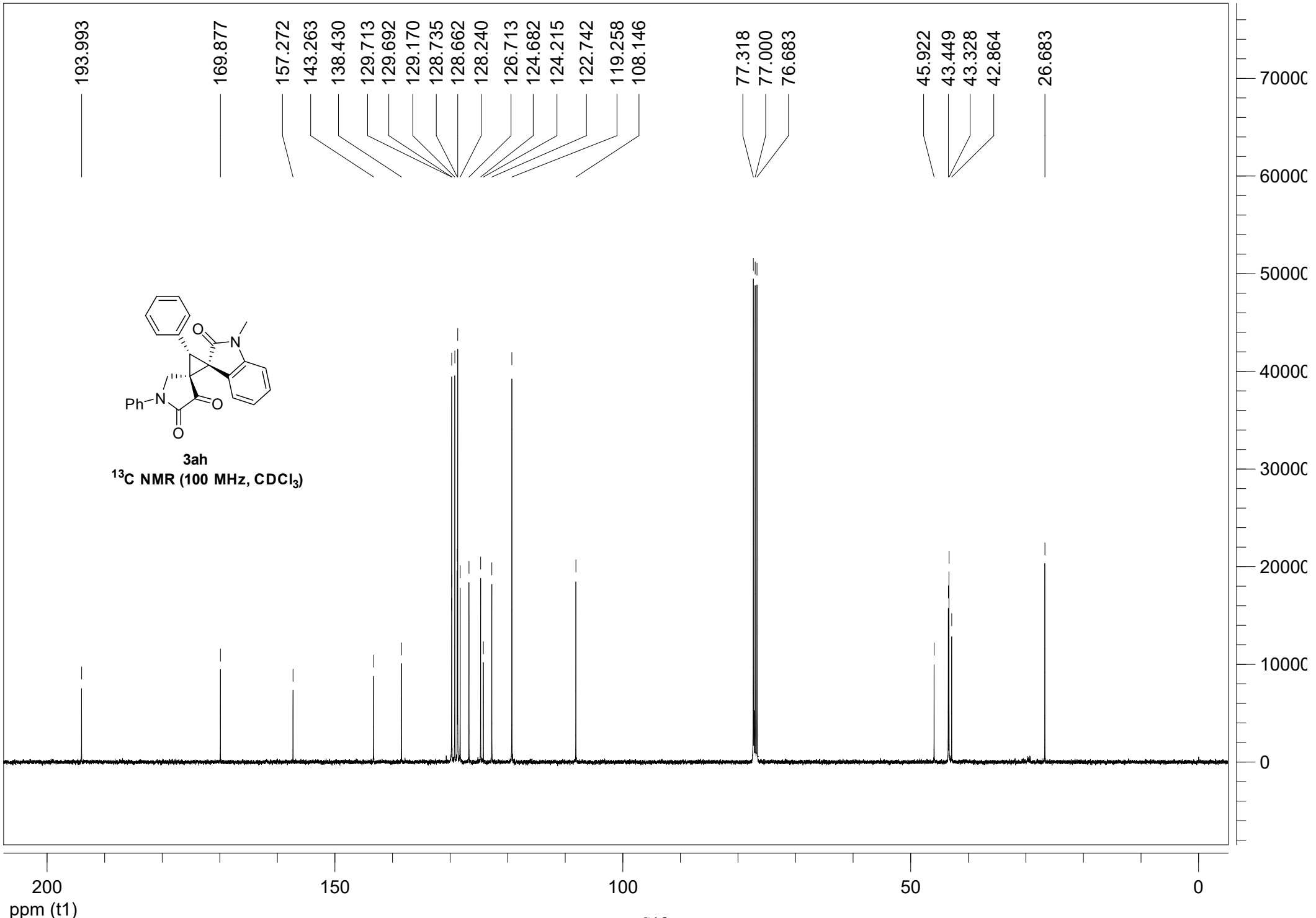
^{1}H NMR (400 MHz, $CDCl_3$)

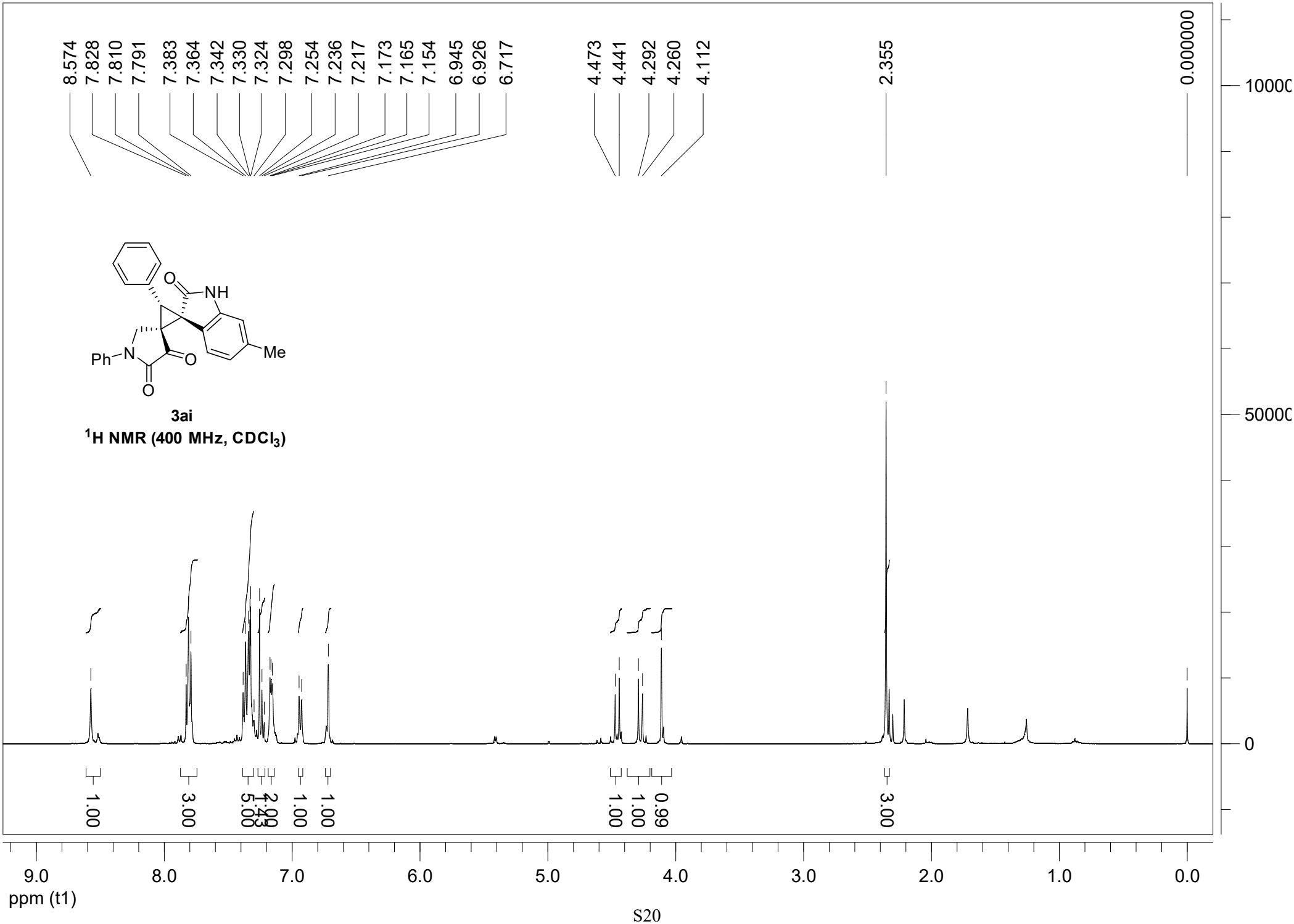

194.055
170.083
157.266
142.386
138.406
135.560
129.713
129.659
129.195
128.837
128.674
128.635
128.292
127.810
127.373
126.752
124.766
124.336
122.785
119.281
109.093
77.318
77.000
76.682
45.846
44.158
43.877
43.584
42.950
Bn
N
O
Ph
N
O
O
3ag
13C NMR (100 MHz, CDCl3)
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)



3ah

^{1}H NMR (400 MHz, $CDCl_3$)


193.993
169.877
157.272
143.263
138.430
129.713
129.692
129.170
128.735
128.662
128.240
126.713
124.682
124.215
122.742
119.258
108.146
77.318
77.000
76.683
45.922
43.449
43.328
42.864
26.683
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)
O
N
Ph
N
O
O
3ah
13C NMR (100 MHz, CDCl3)


8.574
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7.810
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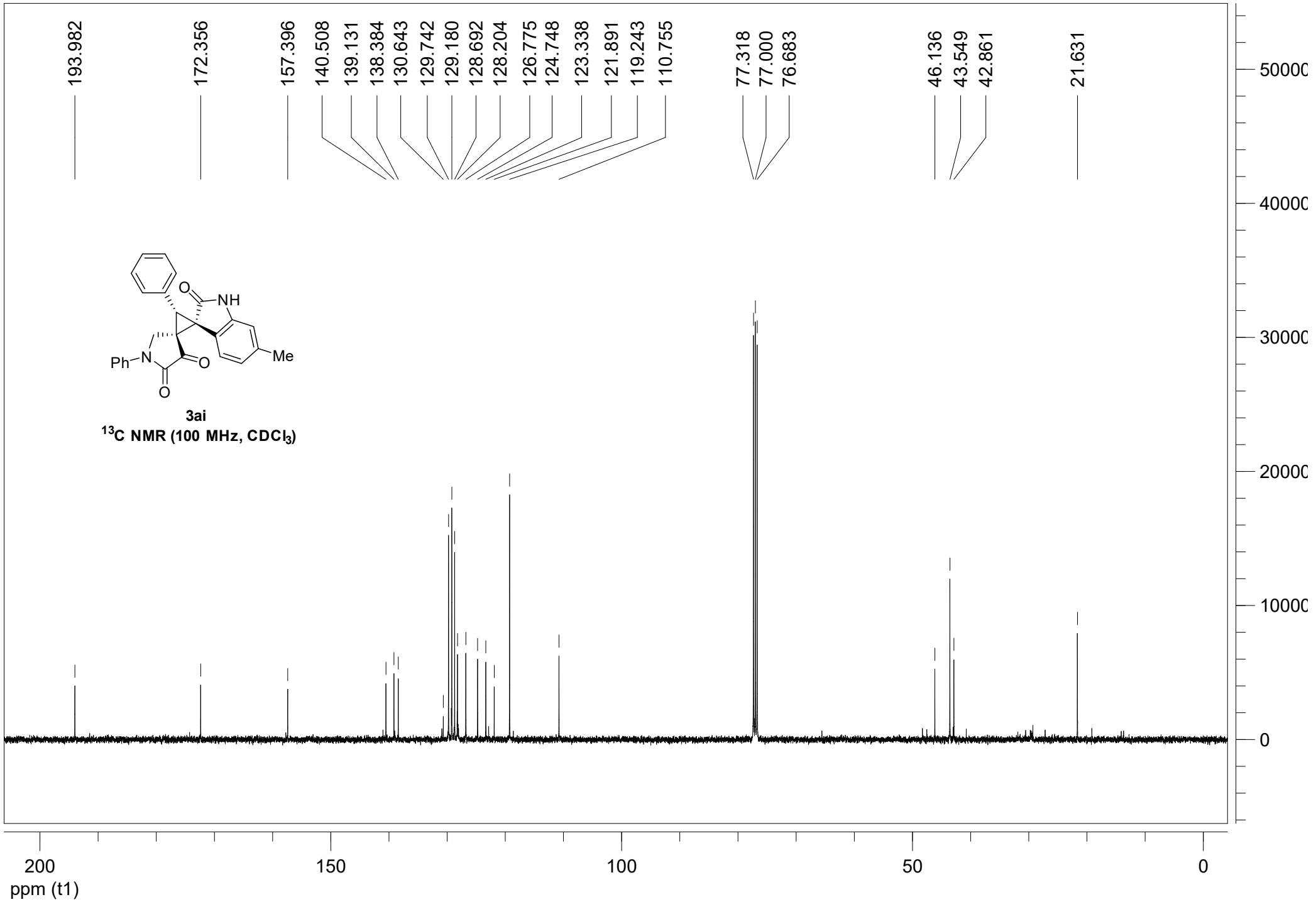
7.383
7.364
7.342
7.330
7.324
7.298
7.254
7.236
7.217
7.173
7.165
7.154
6.945
6.926
6.717
4.473
4.441
4.292
4.260
4.112
2.355
0.000000
O
NH
Ph
N
O
O
Me
3ai
1H NMR (400 MHz, CDCl3)
1.00
3.00
5.00
1.43
2.00
1.00
1.00
1.00
1.00
0.99
3.00
10000
50000
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


193.982
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157.396
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139.131
138.384
130.643
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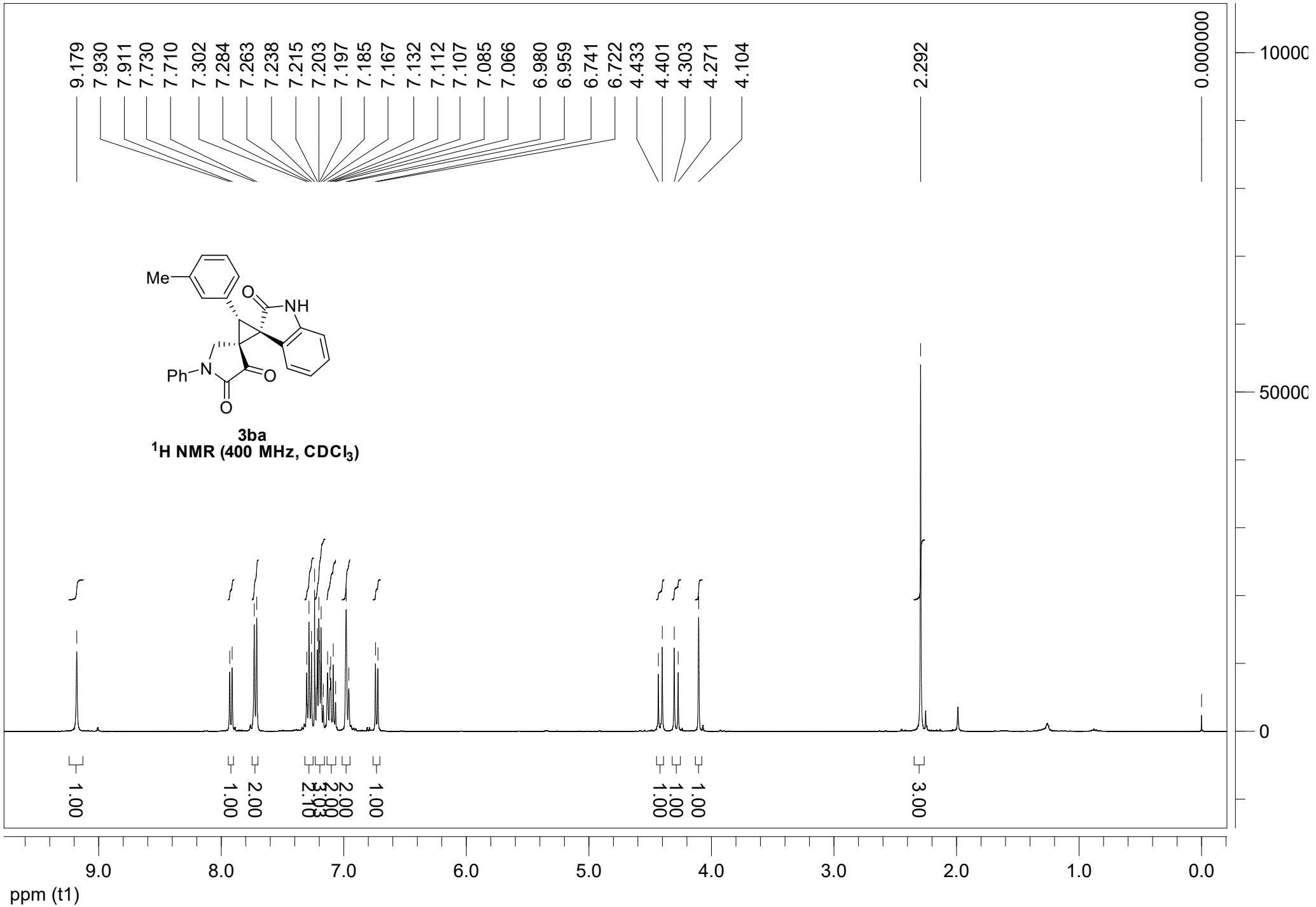
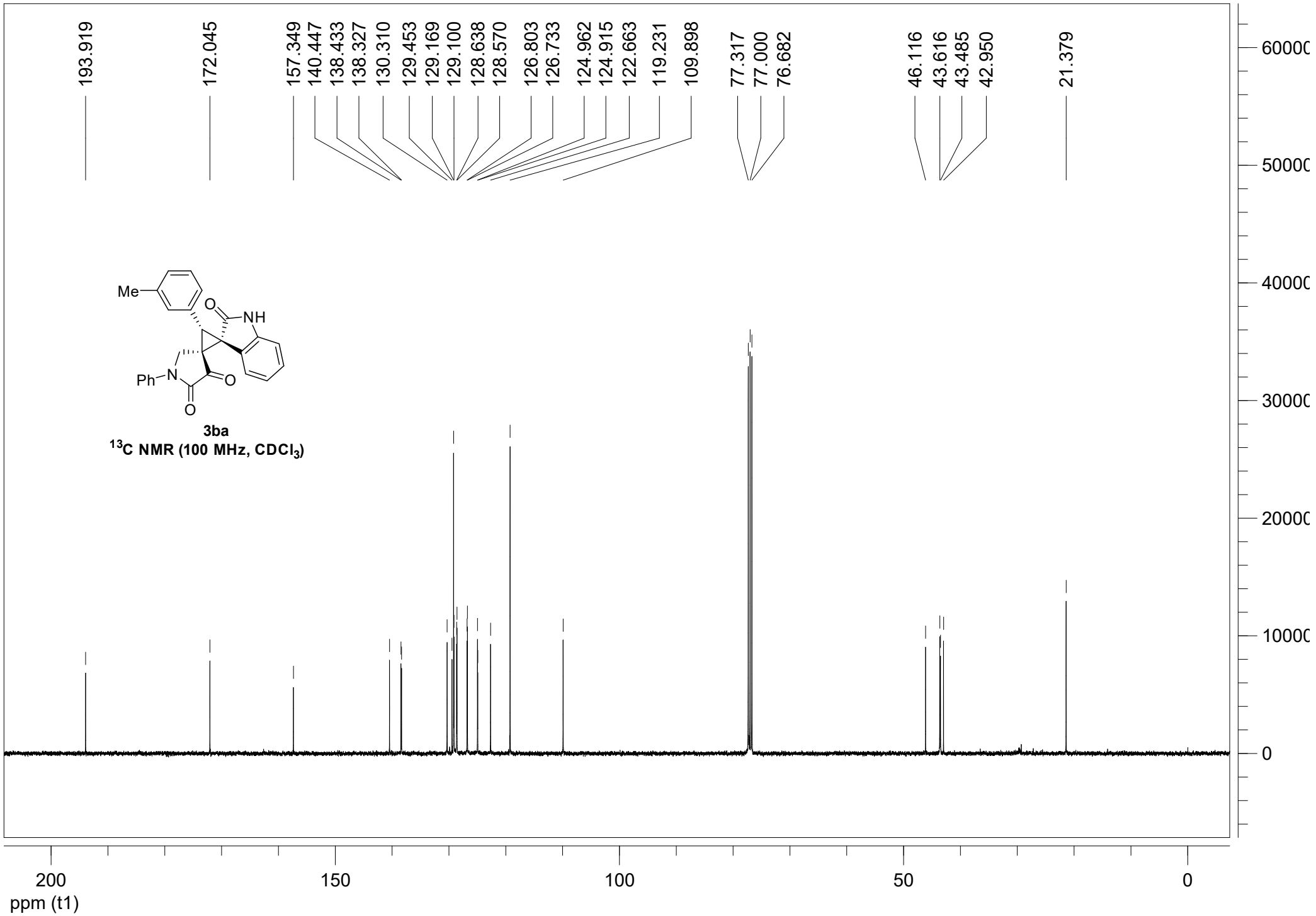
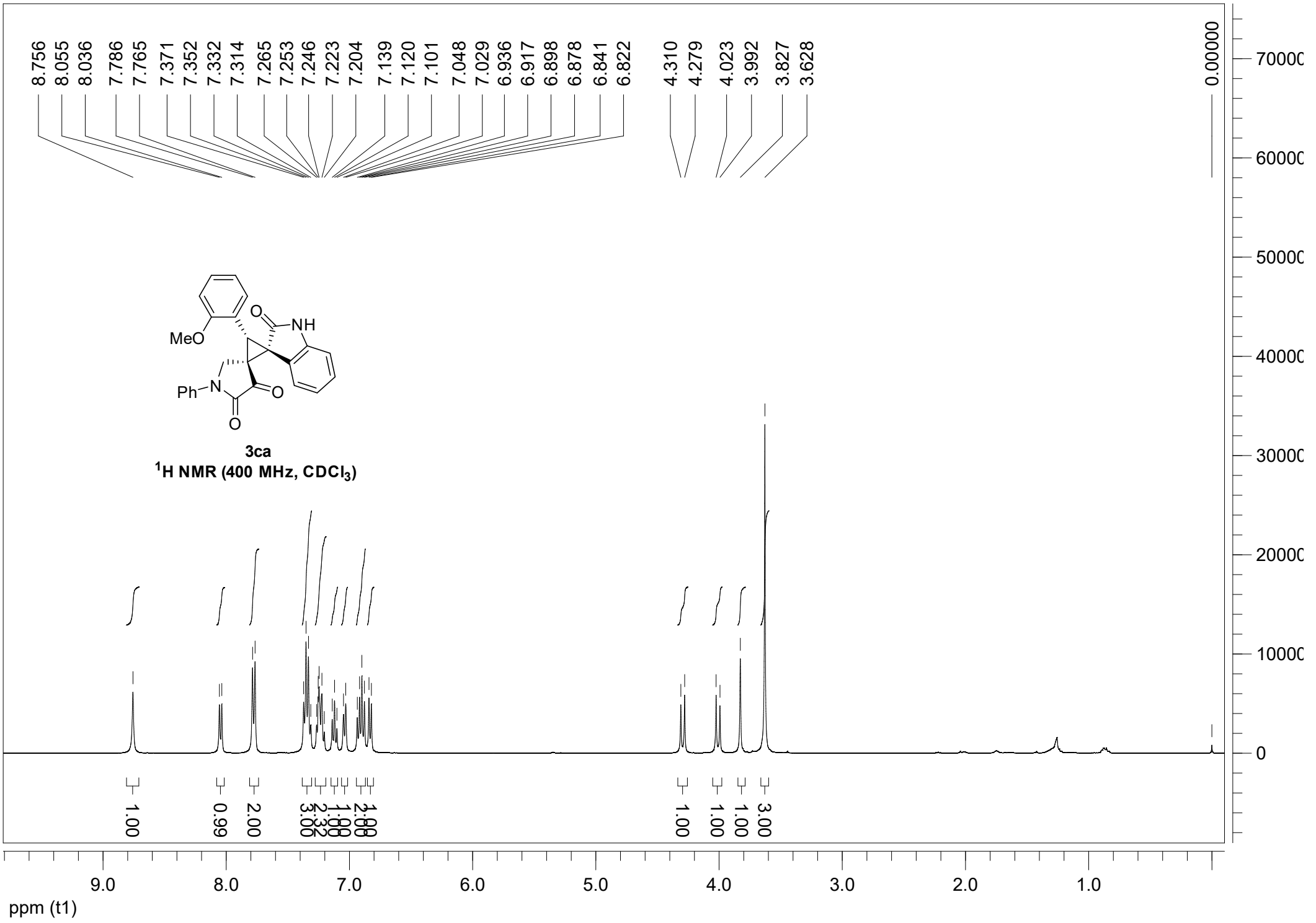
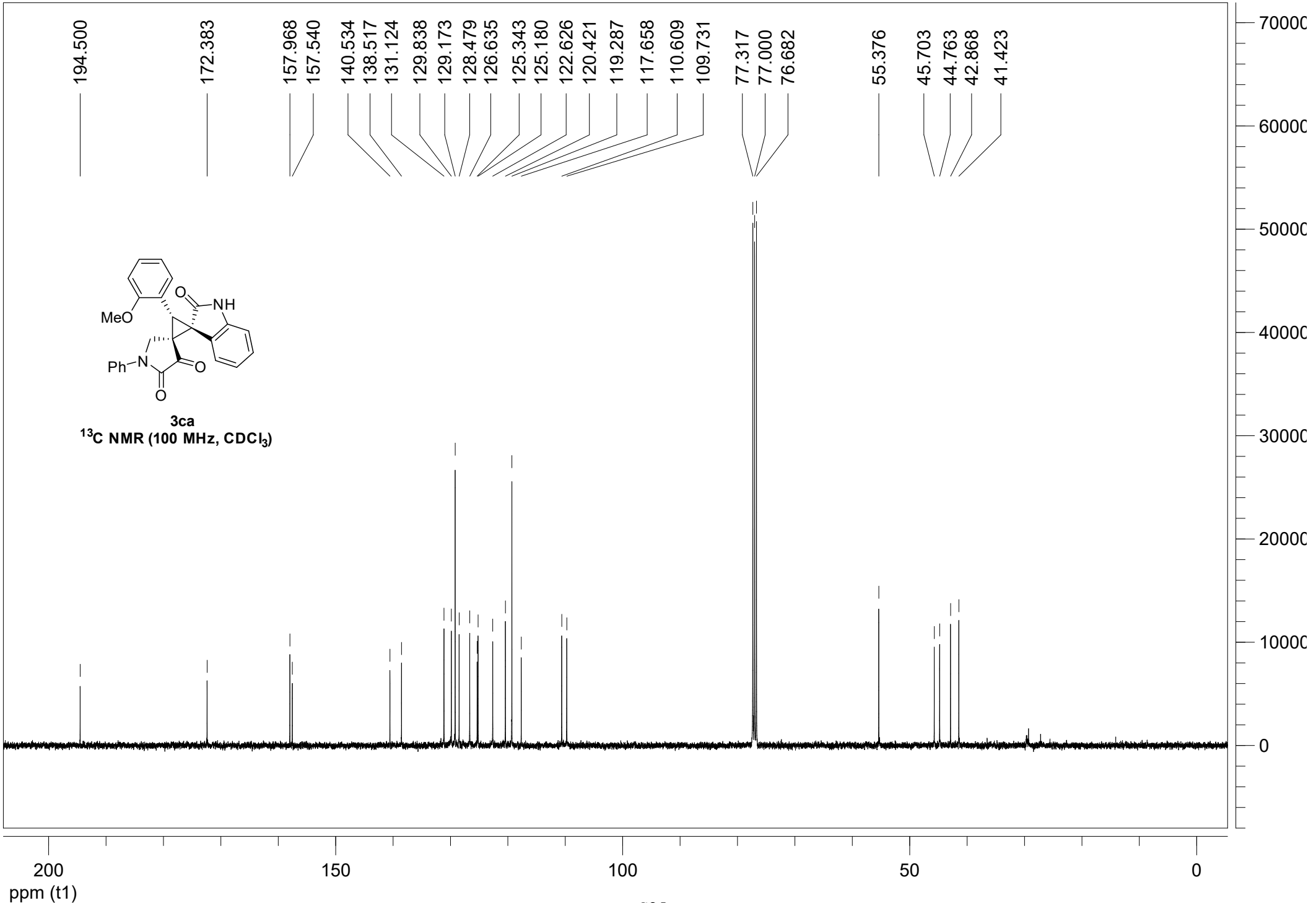
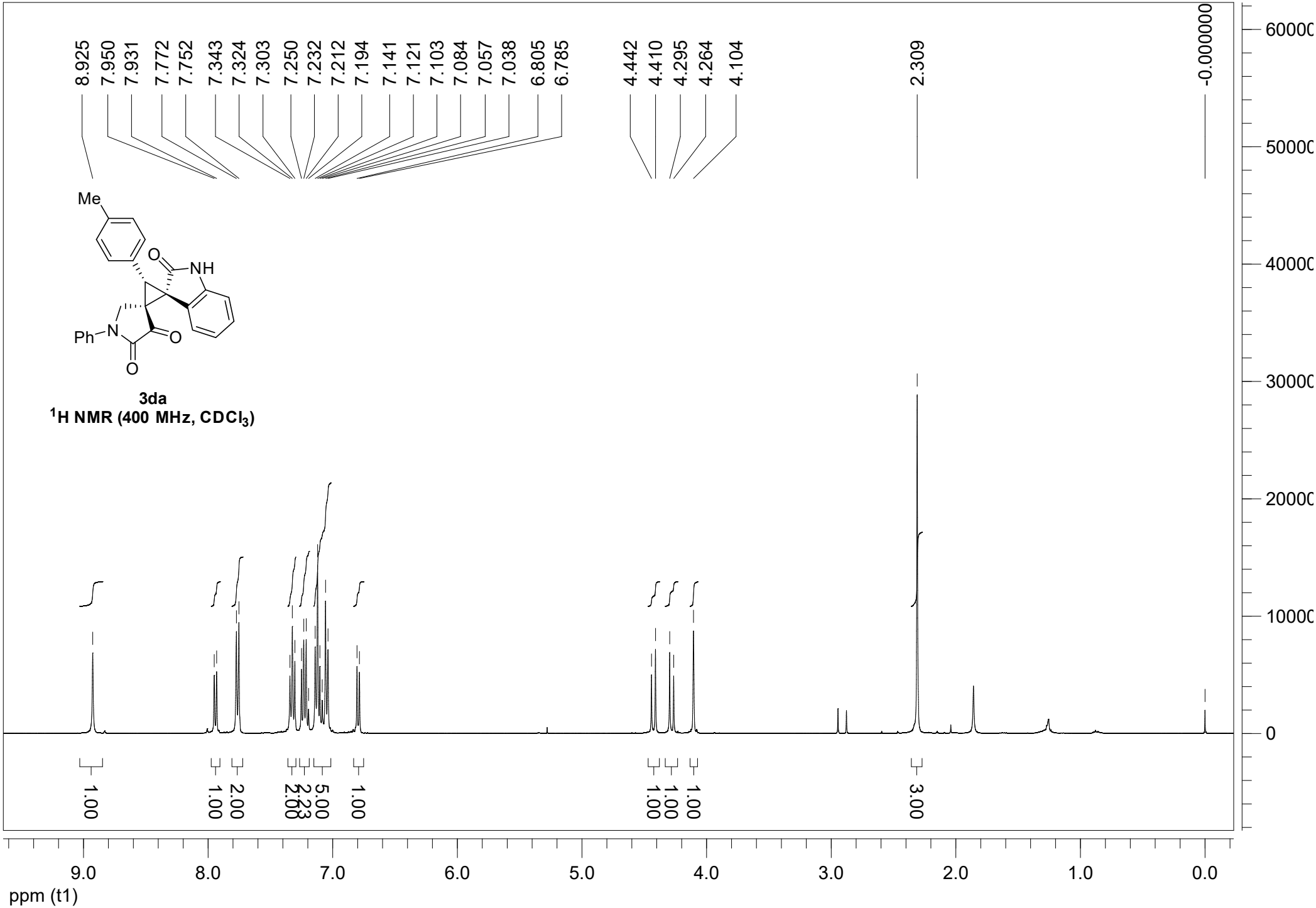
129.180
128.692
128.204
126.775
124.748
123.338
121.891
119.243
110.755
77.318
77.000
76.683
46.136
43.549
42.861
21.631
O
NH
Ph
N
O
O
Me
3ai
13C NMR (100 MHz, CDCl3)
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

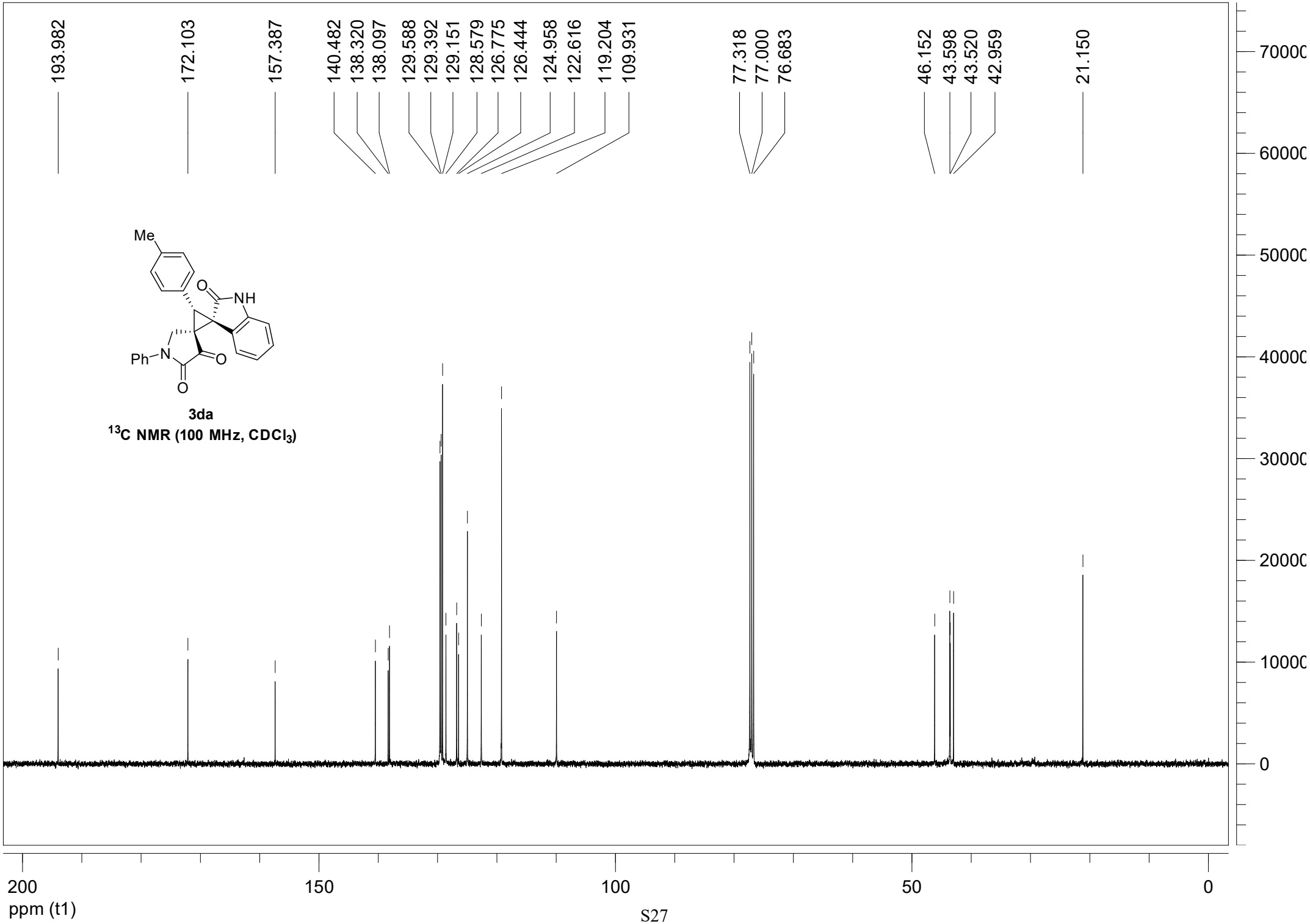

9.179
7.930
7.911
7.730
7.710
7.302
7.284
7.263
7.238
7.215
7.203
7.197
7.185
7.167
7.132
7.112
7.107
7.085
7.066
6.980
6.959
6.741
6.722
4.433
4.401
4.303
4.271
4.104
2.292
0.000000
Me
O
NH
Ph
N
O
O
3ba
1H NMR (400 MHz, CDCl3)
10000
50000
0
1.00
1.00
2.00
2.10
3.03
2.00
1.00
1.00
1.00
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

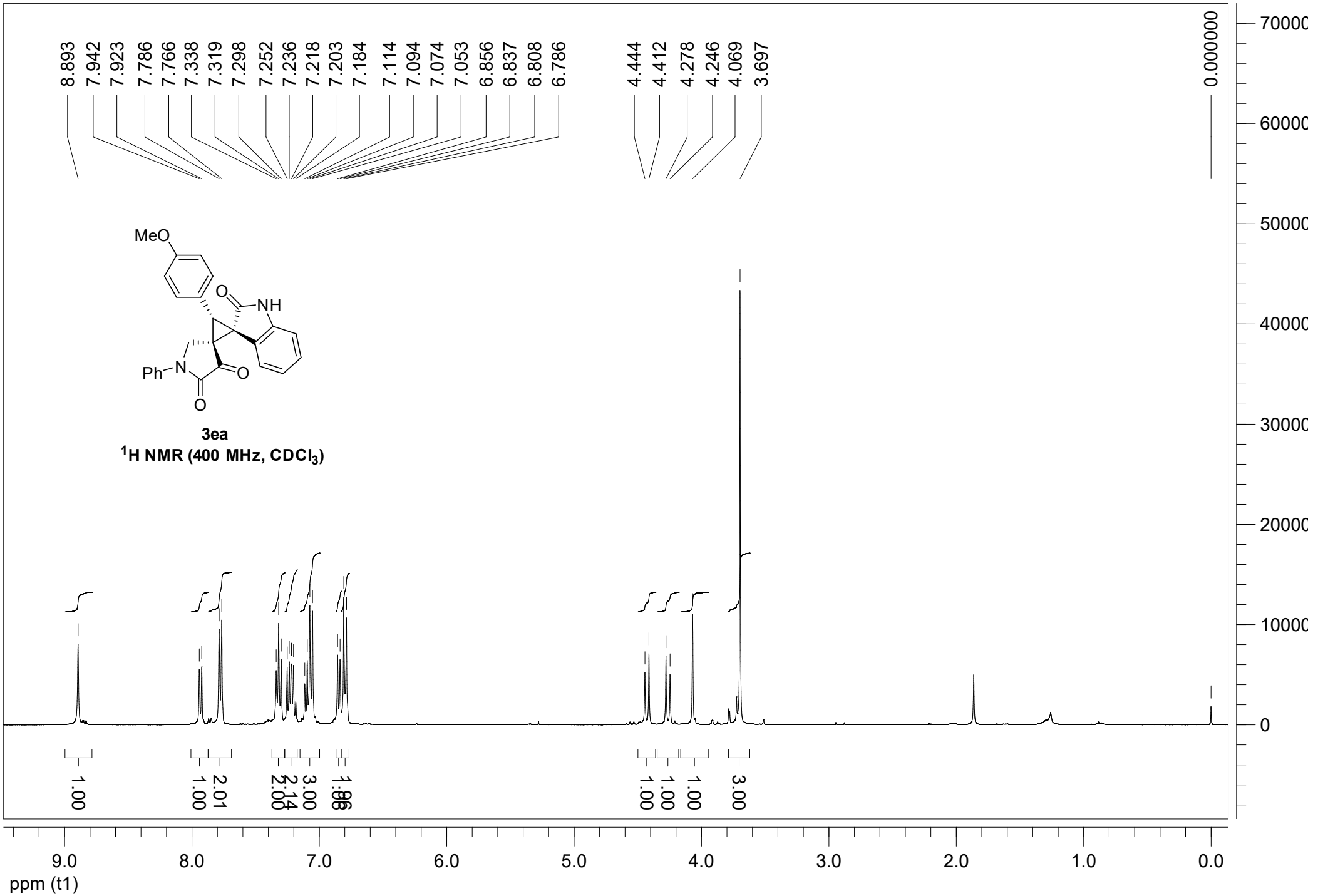

193.919
172.045
157.349
140.447
138.433
138.327
130.310
129.453
129.169
129.100
128.638
128.570
126.803
126.733
124.962
124.915
122.663
119.231
109.898
77.317
77.000
76.682
46.116
43.616
43.485
42.950
21.379
Me
O
NH
Ph
N
O
O
3ba
13C NMR (100 MHz, CDCl3)
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

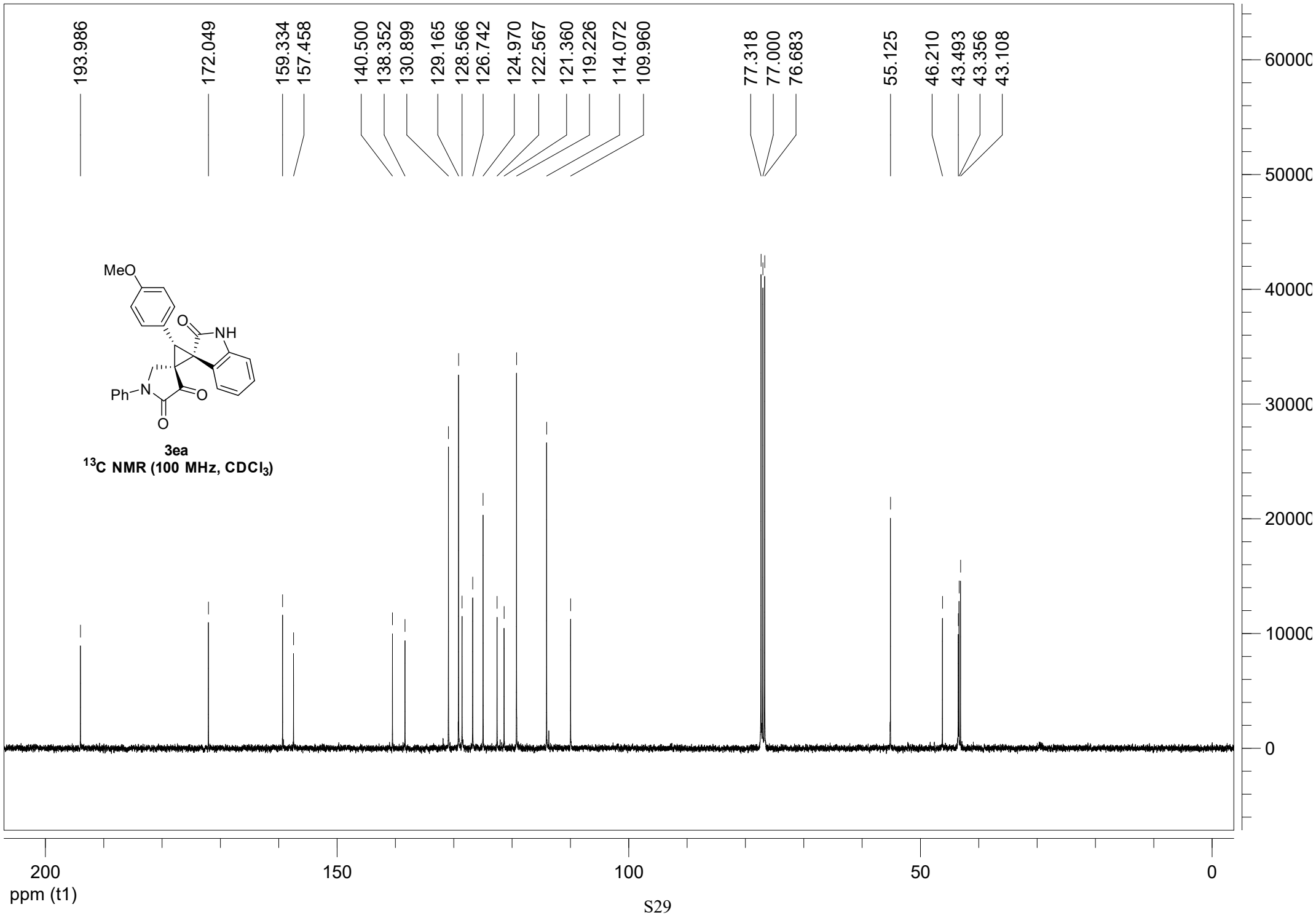

8.756
8.055
8.036
7.786
7.765
7.371
7.352
7.332
7.314
7.265
7.253
7.246
7.223
7.204
7.139
7.120
7.101
7.048
7.029
6.936
6.917
6.898
6.878
6.841
6.822
4.310
4.279
4.023
3.992
3.827
3.628
0.00000
MeO
NH
O
Ph
N
O
O
3ca
1H NMR (400 MHz, CDCl3)
1.00
0.99
2.00
3.06
2.32
1.00
2.00
1.00
1.00
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


194.500
172.383
157.968
157.540
140.534
138.517
131.124
129.838
129.173
128.479
126.635
125.343
125.180
122.626
120.421
119.287
117.658
110.609
109.731
77.317
77.000
76.682
55.376
45.703
44.763
42.868
41.423
MeO
NH
O
Ph
N
O
O
3ca
13C NMR (100 MHz, CDCl3)
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


8.925
7.950
7.931
7.772
7.752
7.343
7.324
7.303
7.250
7.232
7.212
7.194
7.141
7.121
7.103
7.084
7.057
7.038
6.805
6.785
4.442
4.410
4.295
4.264
4.104
2.309
-0.000000
Me
O
NH
Ph
N
O
O
3da
1H NMR (400 MHz, CDCl3)
60000
50000
40000
30000
20000
10000
0
1.00
1.00
2.00
2.03
2.23
5.00
1.00
1.00
1.00
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

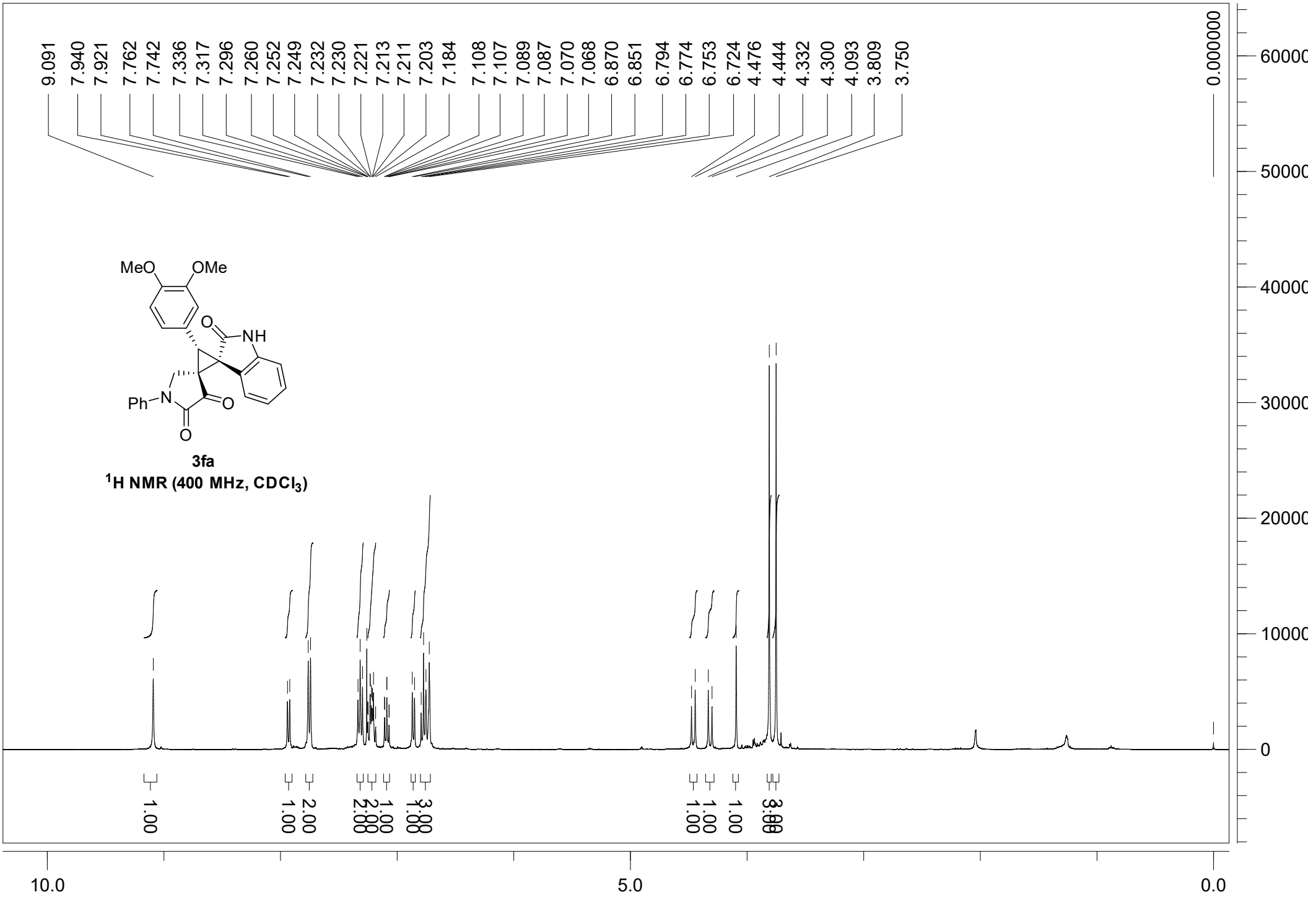

193.982
172.103
157.387
140.482
138.320
138.097
129.588
129.392
129.151
128.579
126.775
126.444
124.958
122.616
119.204
109.931
77.318
77.000
76.683
46.152
43.598
43.520
42.959
21.150
Me
O
NH
Ph
N
O
O
3da
13C NMR (100 MHz, CDCl3)
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

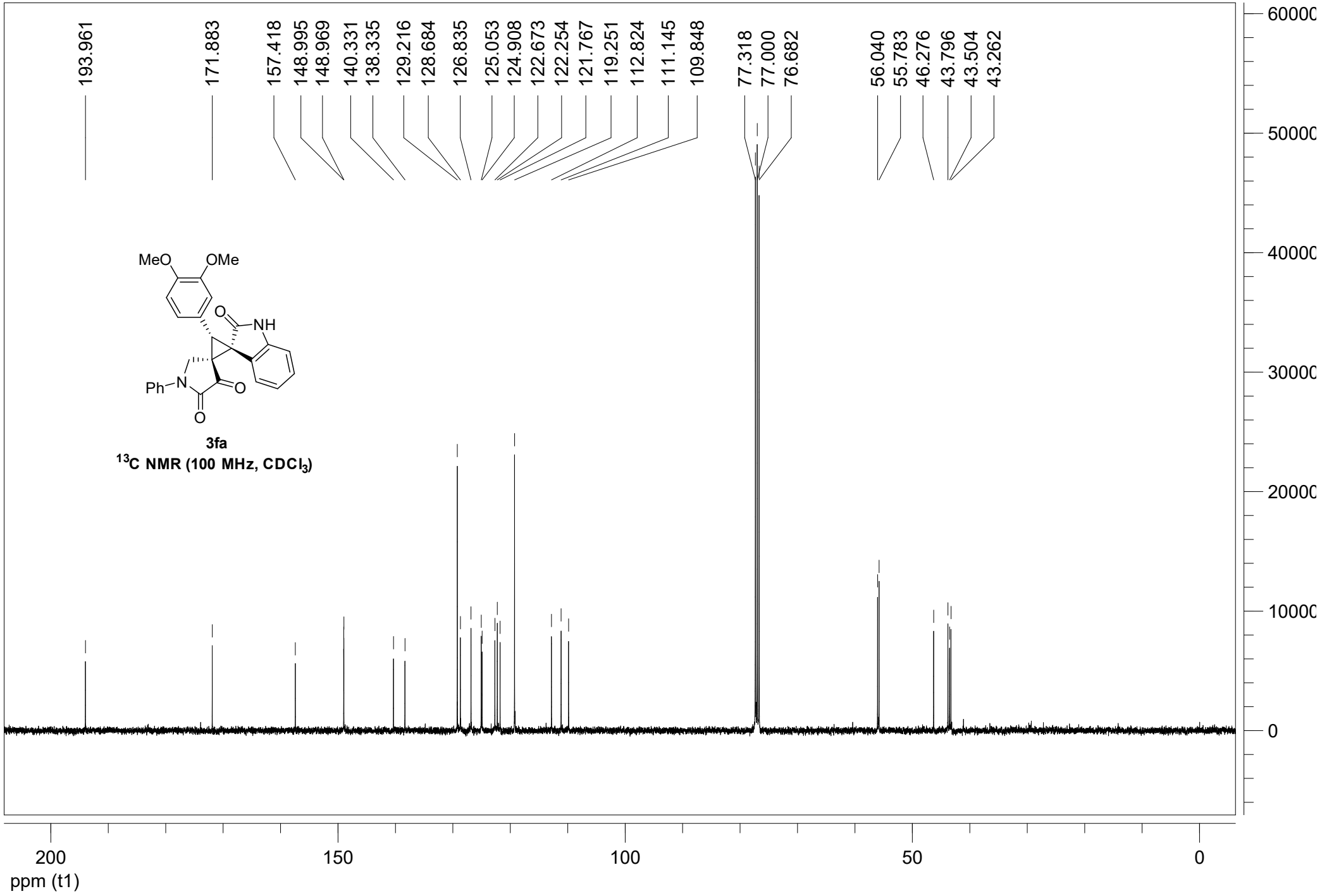

8.893
7.942
7.923
7.786
7.766
7.338
7.319
7.298
7.252
7.236
7.218
7.203
7.184
7.114
7.094
7.074
7.053
6.856
6.837
6.808
6.786
4.444
4.412
4.278
4.246
4.069
3.697
0.000000
MeO
O
NH
Ph
N
O
O
3ea
1H NMR (400 MHz, CDCl3)
1.00
1.00
2.01
2.00
2.14
3.00
1.96
1.00
1.00
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

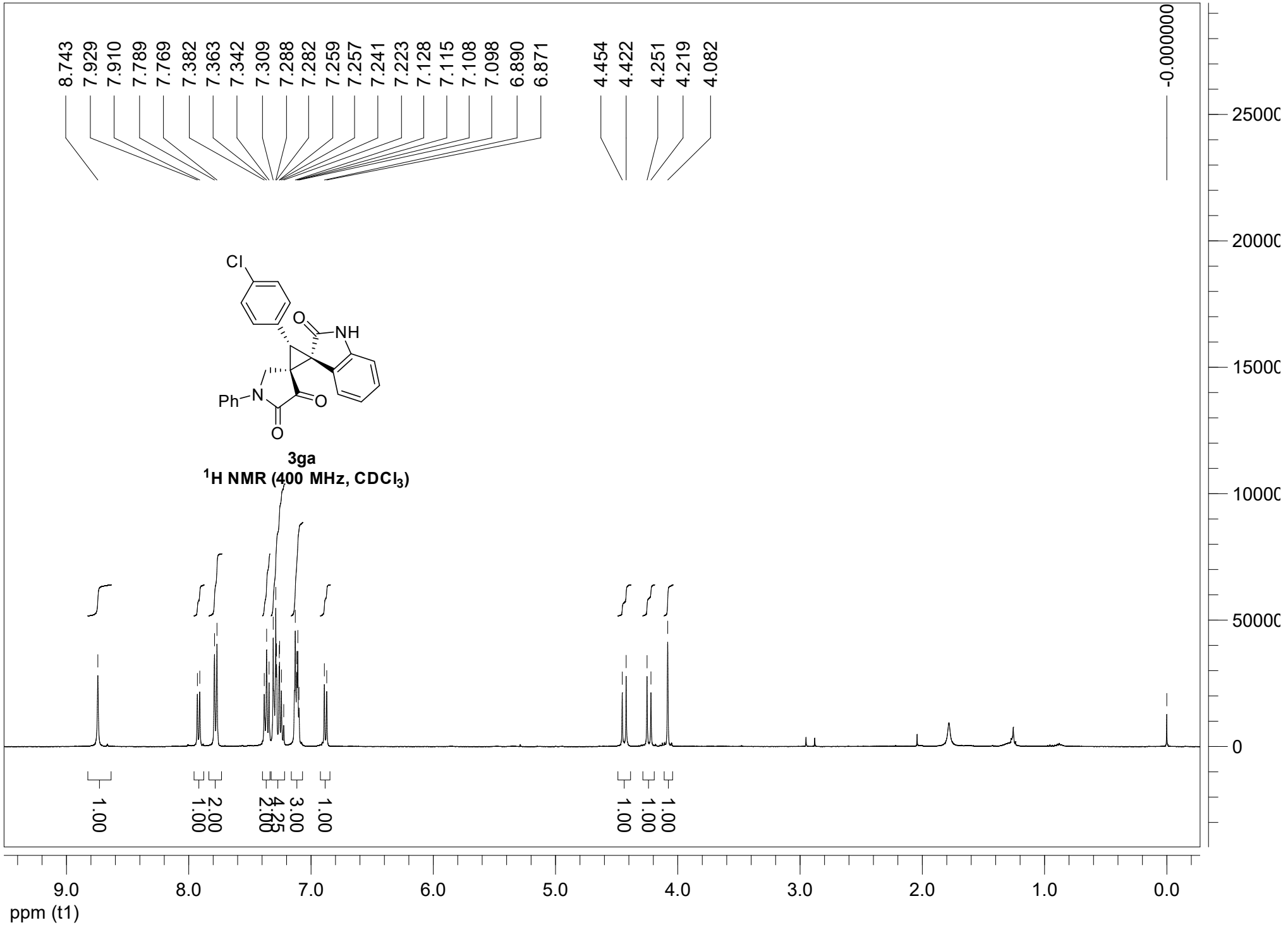


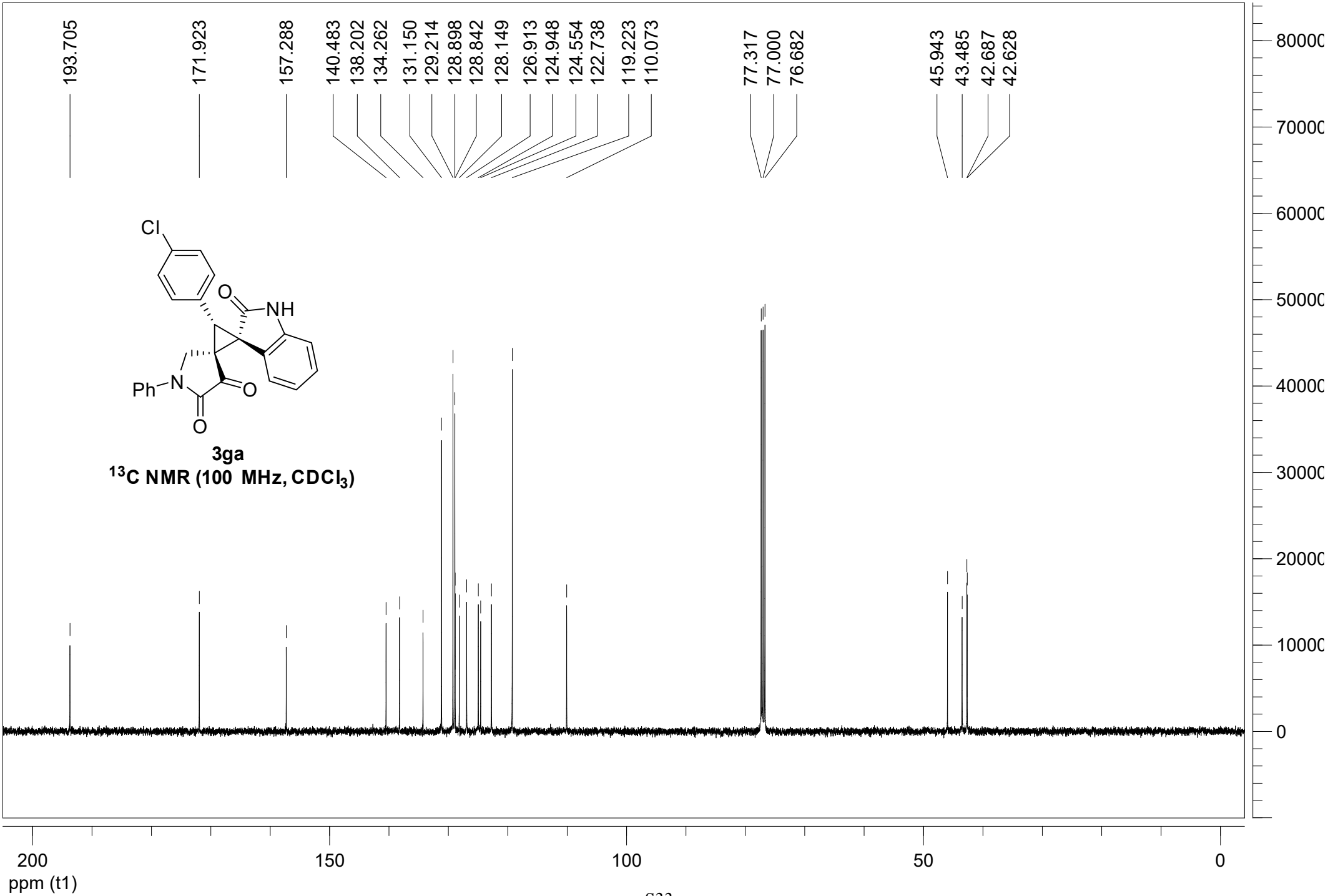
3ea

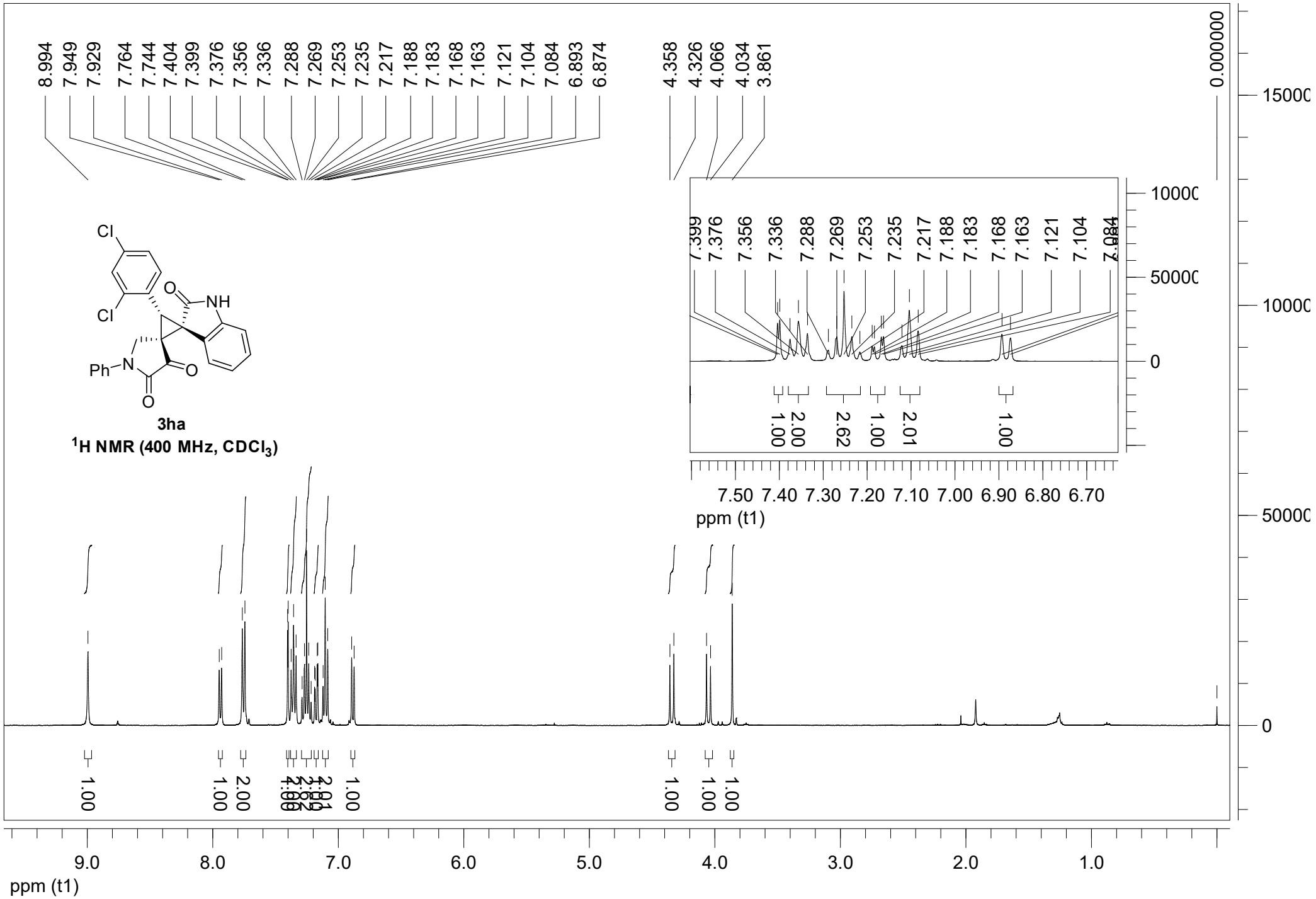
^{13}C NMR (100 MHz, $CDCl_3$)

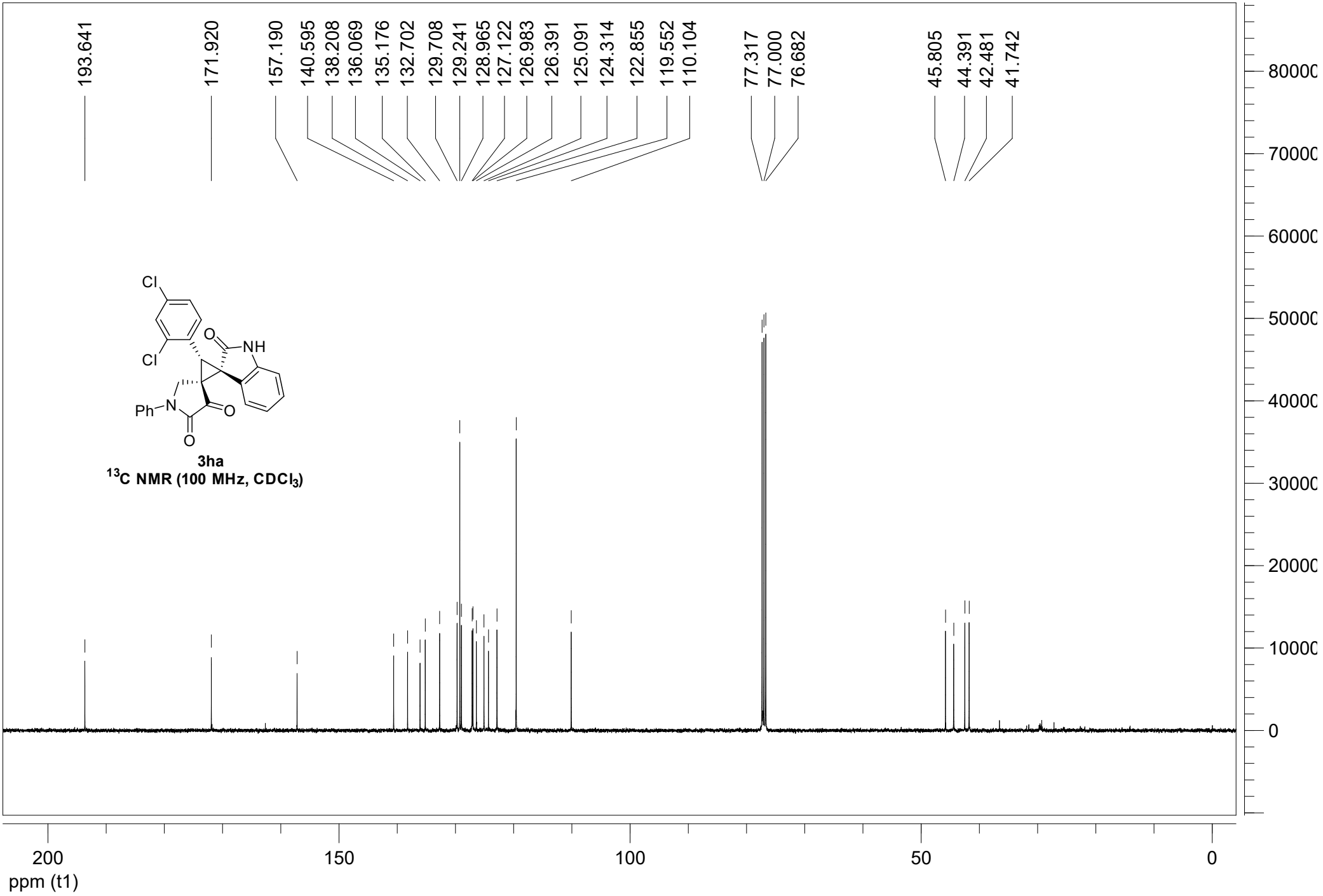

9.091
7.940
7.921
7.762
7.742
7.336
7.317
7.296
7.260
7.252
7.249
7.232
7.230
7.221
7.213
7.211
7.203
7.184
7.108
7.107
7.089
7.087
7.070
7.068
6.870
6.851
6.794
6.774
6.753
6.724
4.476
4.444
4.332
4.300
4.093
3.809
3.750
0.000000
MeO
OMe
O
NH
Ph
N
O
O
3fa
1H NMR (400 MHz, CDCl3)
1.00
1.00
2.00
2.00
2.00
1.00
3.00
1.00
1.00
1.00
3.00
10.0
ppm (t1)
5.0
0.0

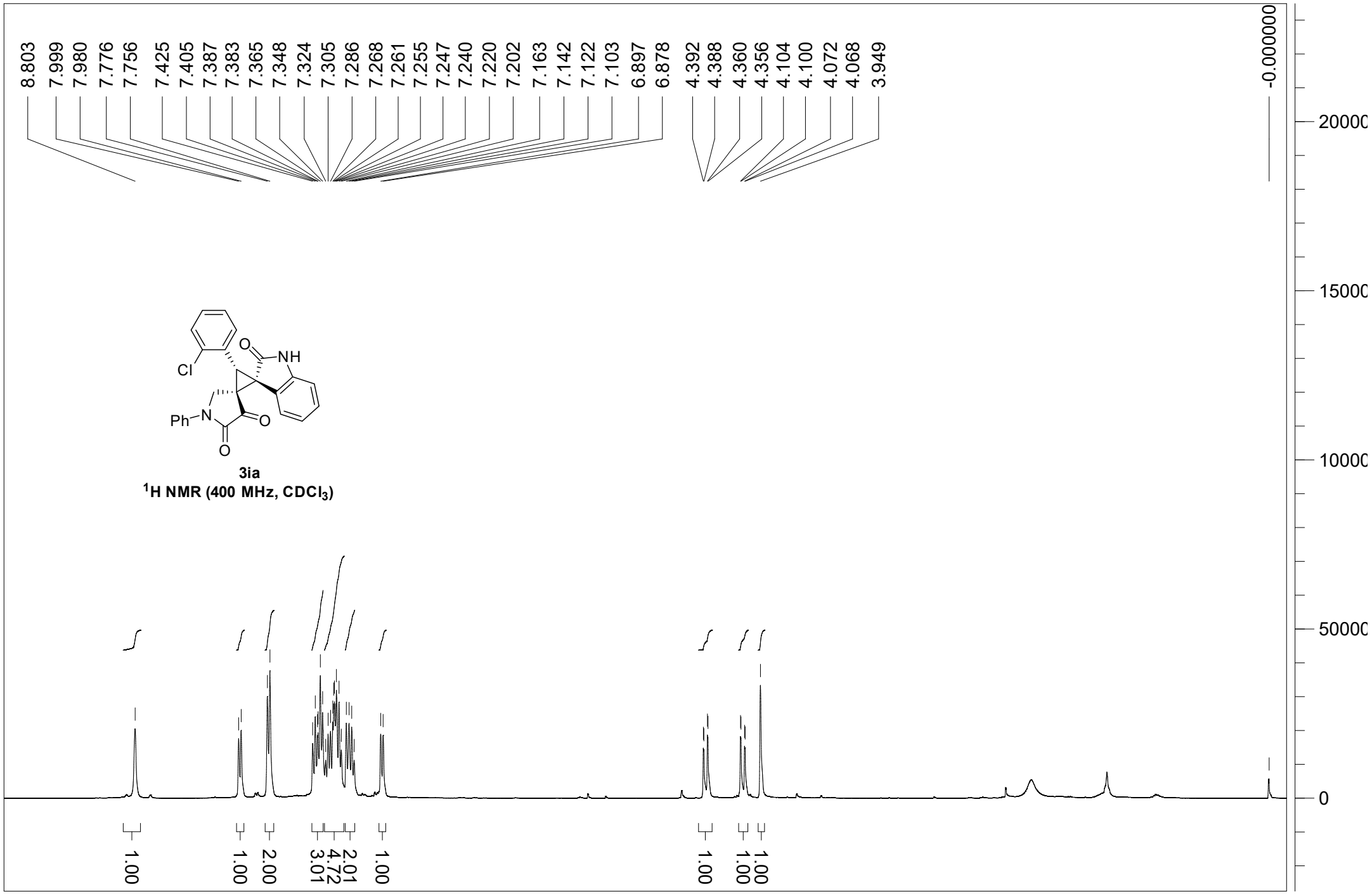

193.961
171.883
157.418
148.995
148.969
140.331
138.335
129.216
128.684
126.835
125.053
124.908
122.673
122.254
121.767
119.251
112.824
111.145
109.848
77.318
77.000
76.682
56.040
55.783
46.276
43.796
43.504
43.262
MeO
OMe
O
NH
Ph
N
O
O
3fa
13C NMR (100 MHz, CDCl3)
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

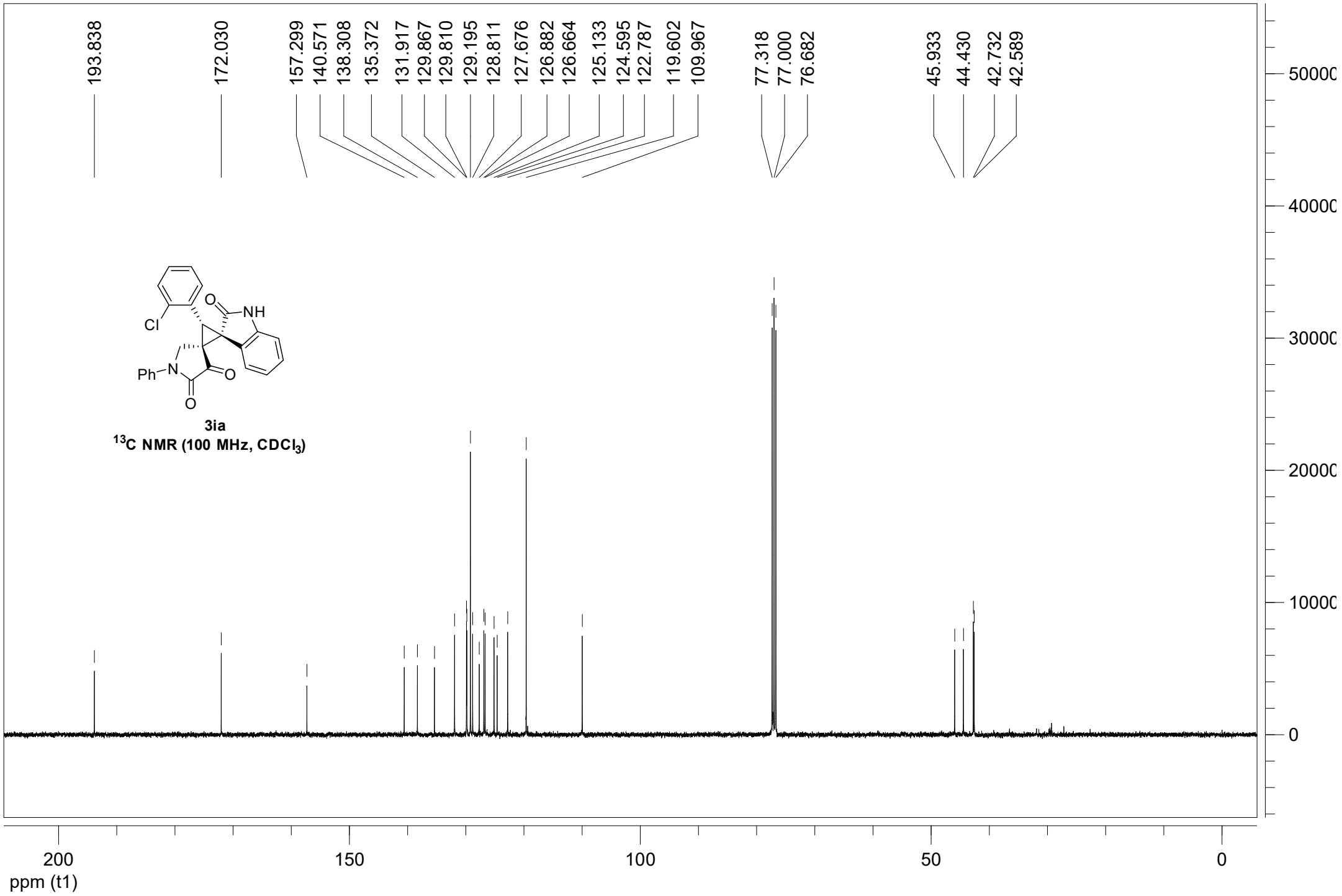

8.743
7.929
7.910
7.789
7.769
7.382
7.363
7.342
7.309
7.288
7.282
7.259
7.257
7.241
7.223
7.128
7.115
7.108
7.098
6.890
6.871
4.454
4.422
4.251
4.219
4.082
-0.000000
Cl
O
NH
Ph
N
O
O
3ga
1H NMR (400 MHz, CDCl3)
1.00
1.00
2.00
4.25
3.00
1.00
1.00
1.00
1.00
25000
20000
15000
10000
5000
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

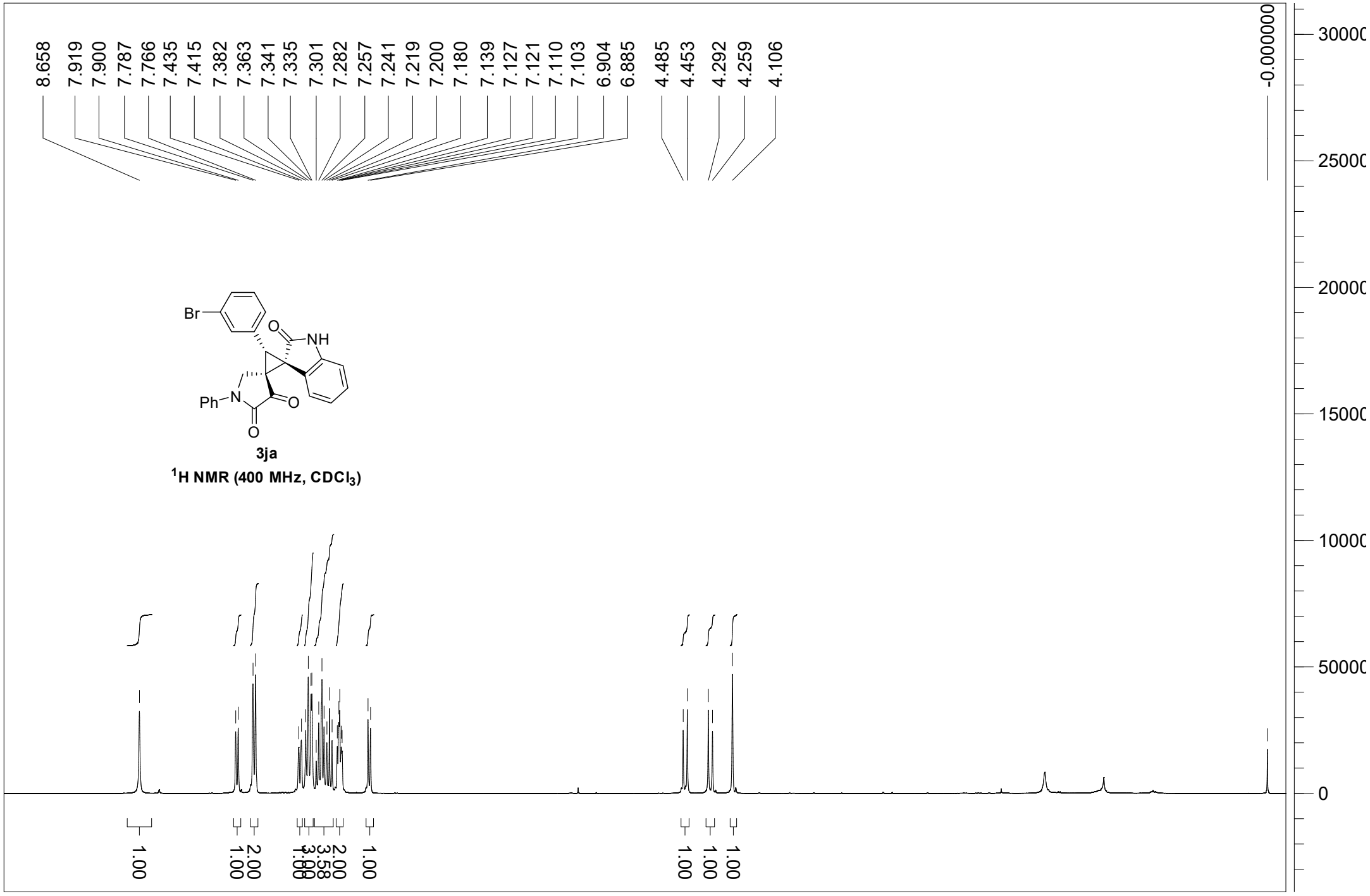

193.705
171.923
157.288
140.483
138.202
134.262
131.150
129.214
128.898
128.842
128.149
126.913
124.948
124.554
122.738
119.223
110.073
77.317
77.000
76.682
45.943
43.485
42.687
42.628
Cl
O
NH
Ph
N
O
O
3ga
13C NMR (100 MHz, CDCl3)
80000
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

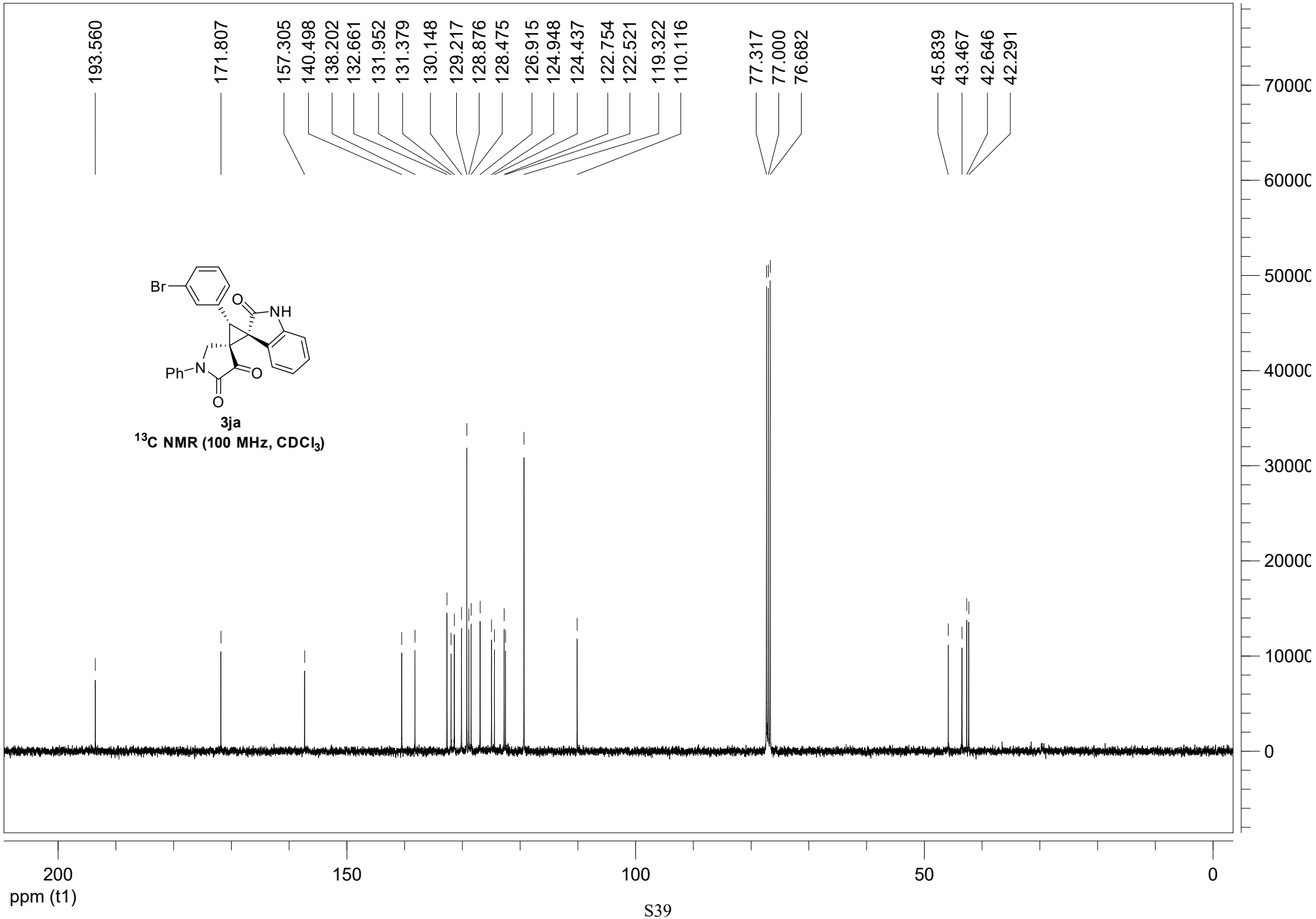

8.994
7.949
7.929
7.764
7.744
7.404
7.399
7.376
7.356
7.336
7.288
7.269
7.253
7.235
7.217
7.188
7.183
7.168
7.163
7.121
7.104
7.084
6.893
6.874
4.358
4.326
4.066
4.034
3.861
0.000000
Cl
Cl
O
NH
Ph
N
O
O
3ha
1H NMR (400 MHz, CDCl3)
1.00
2.00
2.62
1.00
2.01
1.00
7.50 7.40 7.30 7.20 7.10 7.00 6.90 6.80 6.70
ppm (t1)
1.00
1.00
2.00
1.00
1.00
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

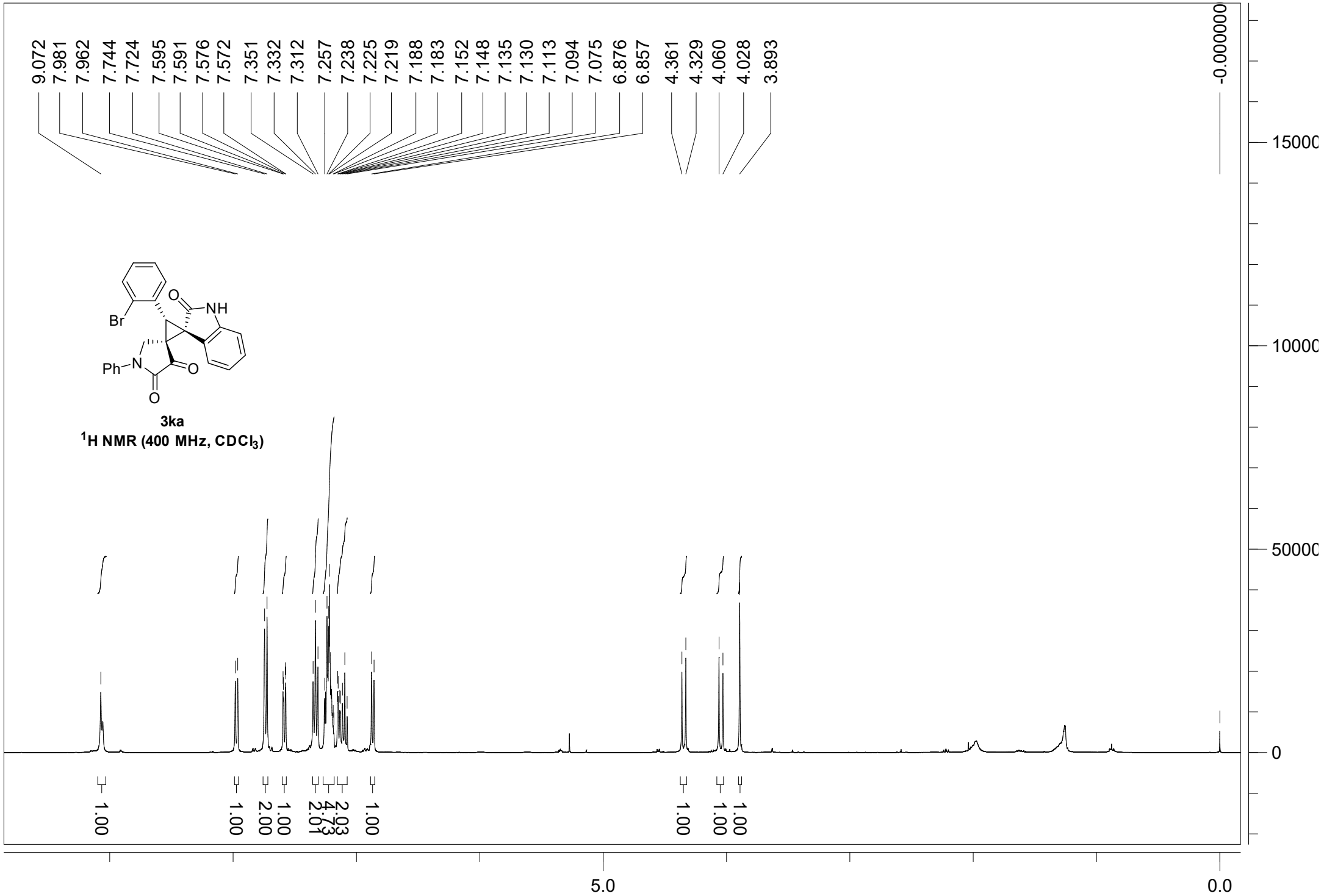

193.641
171.920
157.190
140.595
138.208
136.069
135.176
132.702
129.708
129.241
128.965
127.122
126.983
126.391
125.091
124.314
122.855
119.552
110.104
77.317
77.000
76.682
45.805
44.391
42.481
41.742
Cl
Cl
O
NH
Ph
N
O
O
3ha
13C NMR (100 MHz, CDCl3)
80000
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

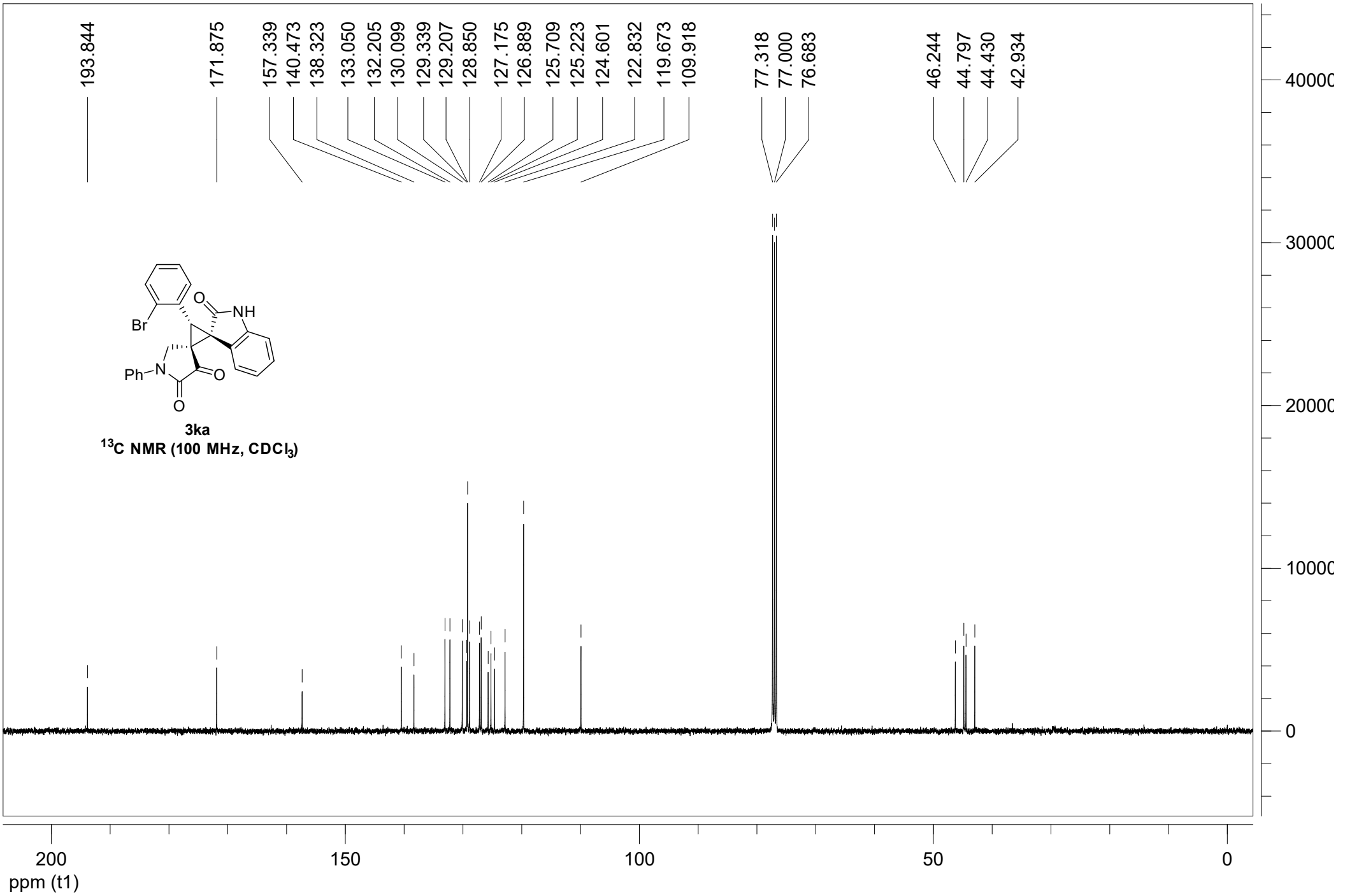

8.803
7.999
7.980
7.776
7.756
7.425
7.405
7.387
7.383
7.365
7.348
7.324
7.305
7.286
7.268
7.261
7.255
7.247
7.240
7.220
7.202
7.163
7.142
7.122
7.103
6.897
6.878
4.392
4.388
4.360
4.356
4.104
4.100
4.072
4.068
3.949
-0.000000
Cl
NH
O
Ph
N
O
O
O
3ia
1H NMR (400 MHz, CDCl3)
20000
15000
10000
50000
0
1.00
1.00
2.00
3.01
4.72
2.01
1.00
1.00
1.00
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

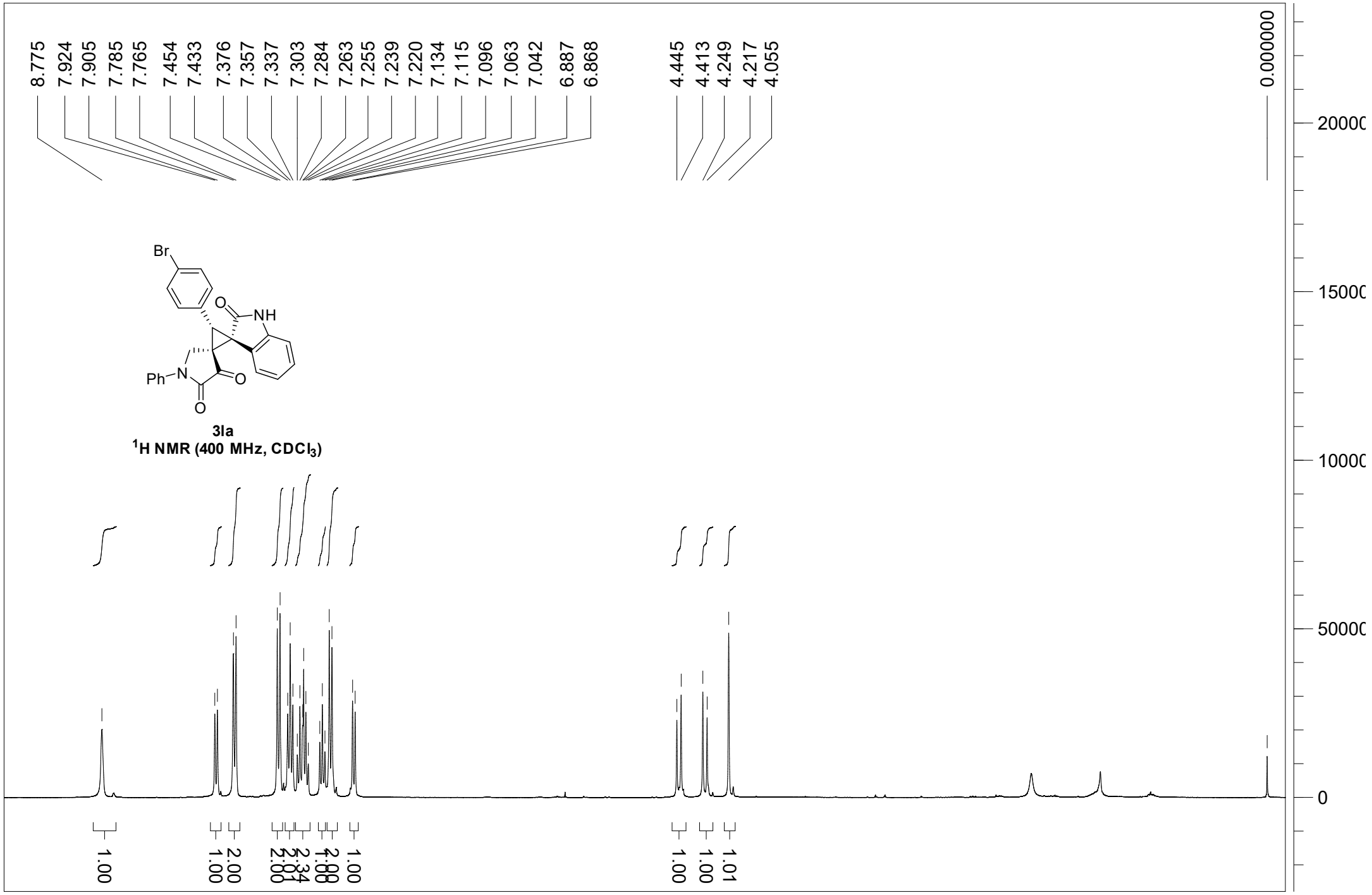

193.838
172.030
157.299
140.571
138.308
135.372
131.917
129.867
129.810
129.195
128.811
127.676
126.882
126.664
125.133
124.595
122.787
119.602
109.967
77.318
77.000
76.682
45.933
44.430
42.732
42.589
Cl
O
NH
Ph
N
O
O
3ia
13C NMR (100 MHz, CDCl3)
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


8.658
7.919
7.900
7.787
7.766
7.435
7.415
7.382
7.363
7.341
7.335
7.301
7.282
7.257
7.241
7.219
7.200
7.180
7.139
7.127
7.121
7.110
7.103
6.904
6.885
4.485
4.453
4.292
4.259
4.106
-0.000000
Br
O
NH
Ph
N
O
O
3ja
1H NMR (400 MHz, CDCl3)
1.00
1.00
2.00
1.00
3.58
3.00
2.00
1.00
1.00
1.00
1.00
30000
25000
20000
15000
10000
5000
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


193.560
171.807
157.305
140.498
138.202
132.661
131.952
131.379
130.148
129.217
128.876
128.475
126.915
124.948
124.437
122.754
122.521
119.322
110.116
77.317
77.000
76.682
45.839
43.467
42.646
42.291
Br
O
NH
Ph
N
O
O
3ja
13C NMR (100 MHz, CDCl3)
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

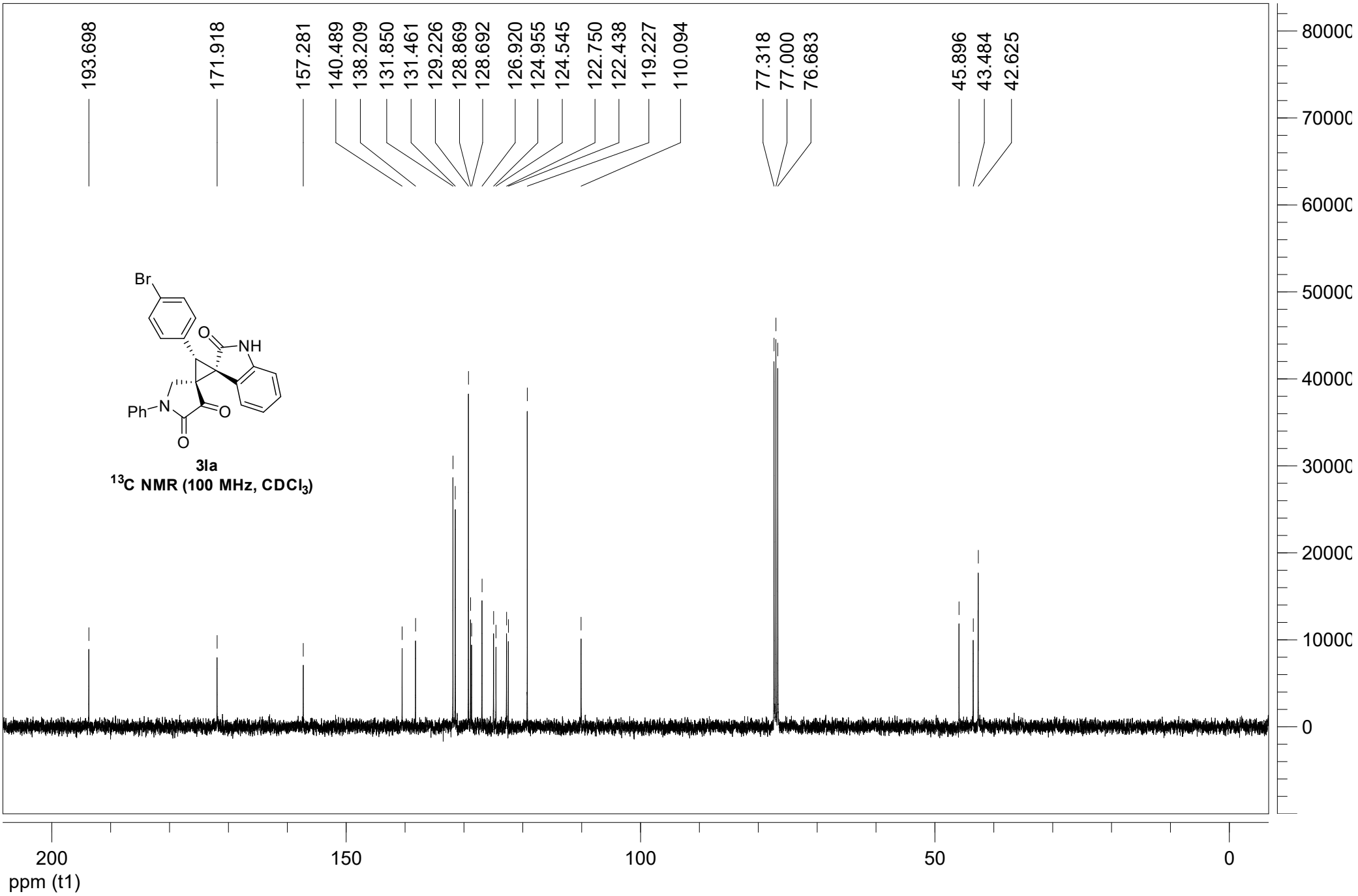

9.072
7.981
7.962
7.744
7.724
7.595
7.591
7.576
7.572
7.351
7.332
7.312
7.257
7.238
7.225
7.219
7.188
7.183
7.152
7.148
7.135
7.130
7.113
7.094
7.075
6.876
6.857
4.361
4.329
4.060
4.028
3.893
-0.000000
Br
NH
O
Ph
N
O
O
3ka
1H NMR (400 MHz, CDCl3)
15000
10000
50000
0
1.00
1.00
2.00
1.00
2.01
4.23
2.03
1.00
1.00
1.00
1.00
5.0
0.0
ppm (t1)

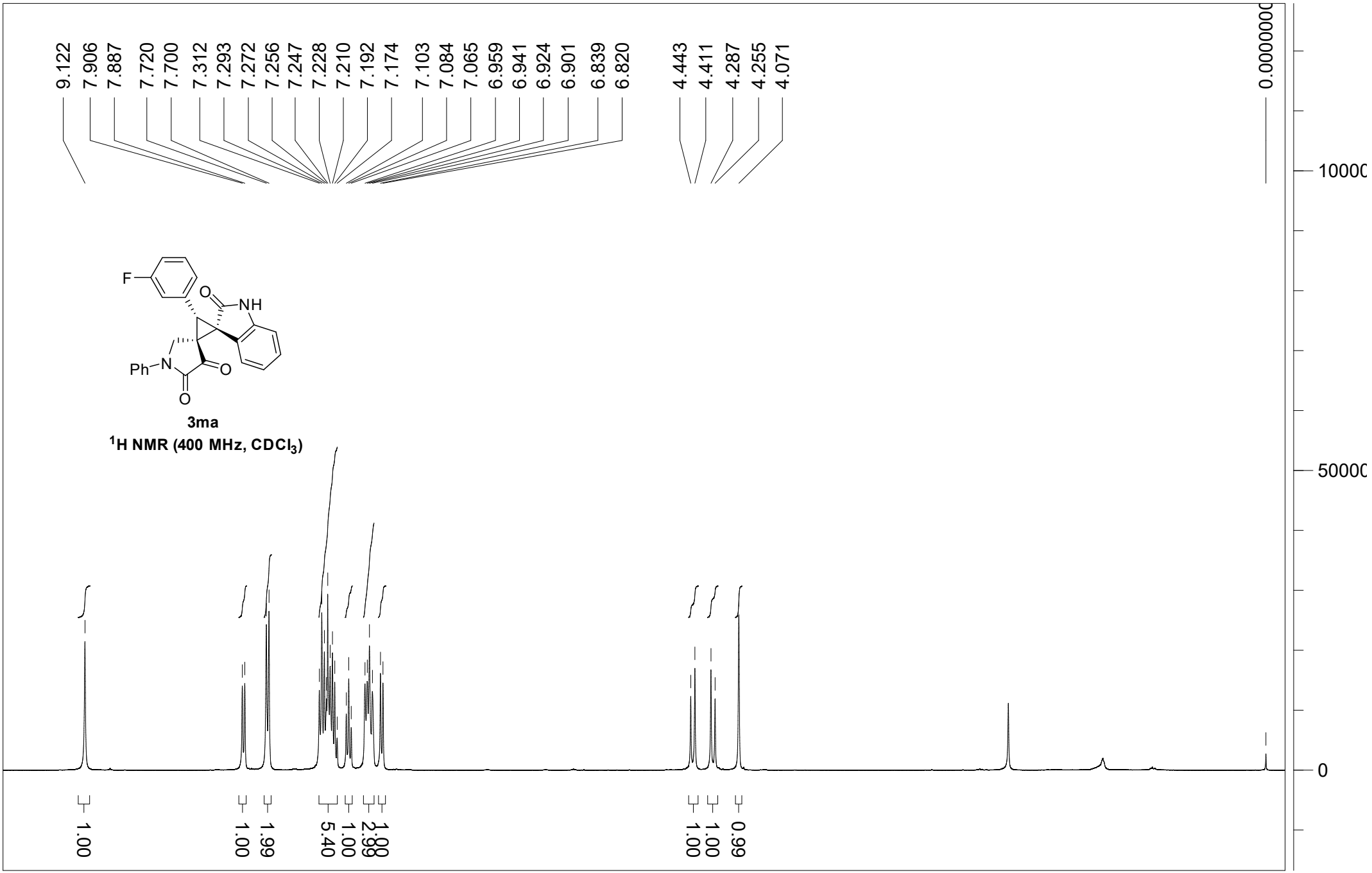

193.844
171.875
157.339
140.473
138.323
133.050
132.205
130.099
129.339
129.207
128.850
127.175
126.889
125.709
125.223
124.601
122.832
119.673
109.918
77.318
77.000
76.683
46.244
44.797
44.430
42.934
40000
30000
20000
10000
0
Br
O
NH
Ph
N
O
O
3ka
13C NMR (100 MHz, CDCl3)
200
150
100
50
0
ppm (t1)

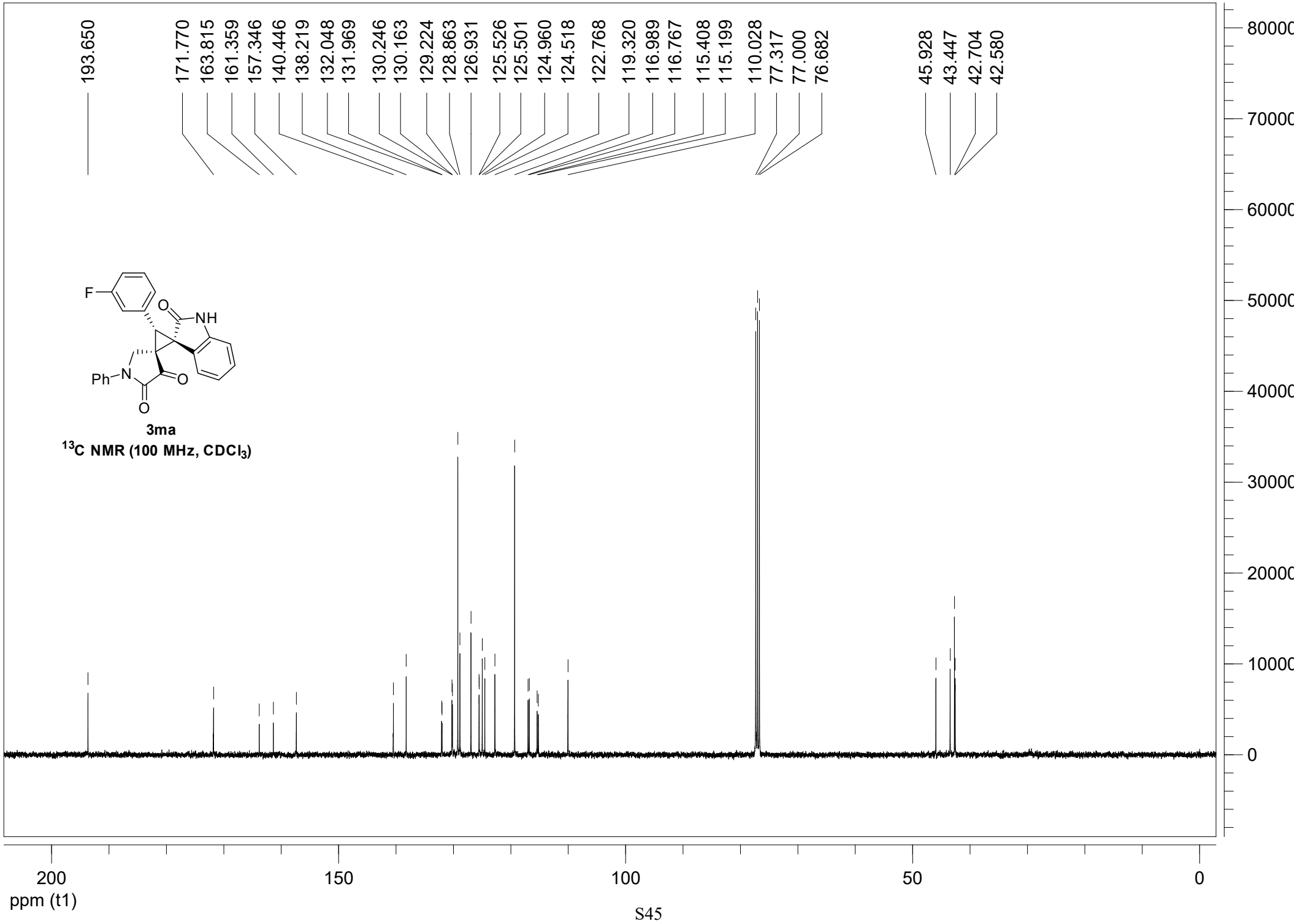


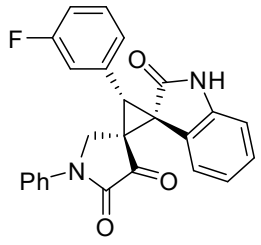
3la

^{1}H NMR (400 MHz, $CDCl_3$)


193.698
171.918
157.281
140.489
138.209
131.850
131.461
129.226
128.869
128.692
126.920
124.955
124.545
122.750
122.438
119.227
110.094
77.318
77.000
76.683
45.896
43.484
42.625
Br
O
NH
Ph
N
O
O
3la
13C NMR (100 MHz, CDCl3)
80000
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

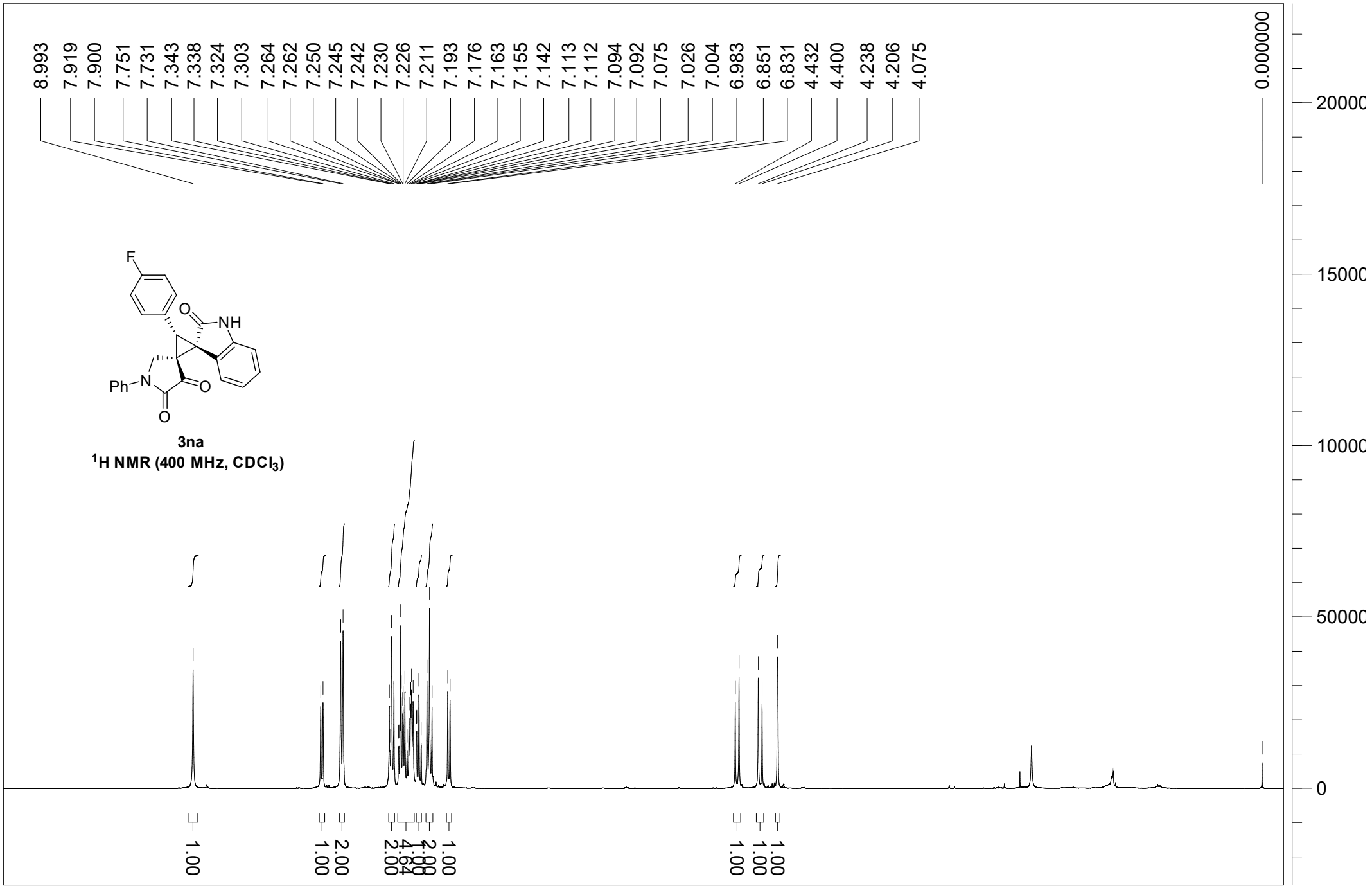

9.122
7.906
7.887
7.720
7.700
7.312
7.293
7.272
7.256
7.247
7.228
7.210
7.192
7.174
7.103
7.084
7.065
6.959
6.941
6.924
6.901
6.839
6.820
4.443
4.411
4.287
4.255
4.071
0.000000
F
O
NH
Ph
N
O
O
O
3ma
1H NMR (400 MHz, CDCl3)
10000
50000
0
1.00
1.00
1.99
5.40
1.00
2.88
1.00
1.00
1.00
0.99
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


193.650
171.770
163.815
161.359
157.346
140.446
138.219
132.048
131.969
130.246
130.163
129.224
128.863
126.931
125.526
125.501
124.960
124.518
122.768
119.320
116.989
116.767
115.408
115.199
110.028
77.317
77.000
76.682
45.928
43.447
42.704
42.580
F
O
NH
Ph
N
O
O
3ma
13C NMR (100 MHz, CDCl3)
80000
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)



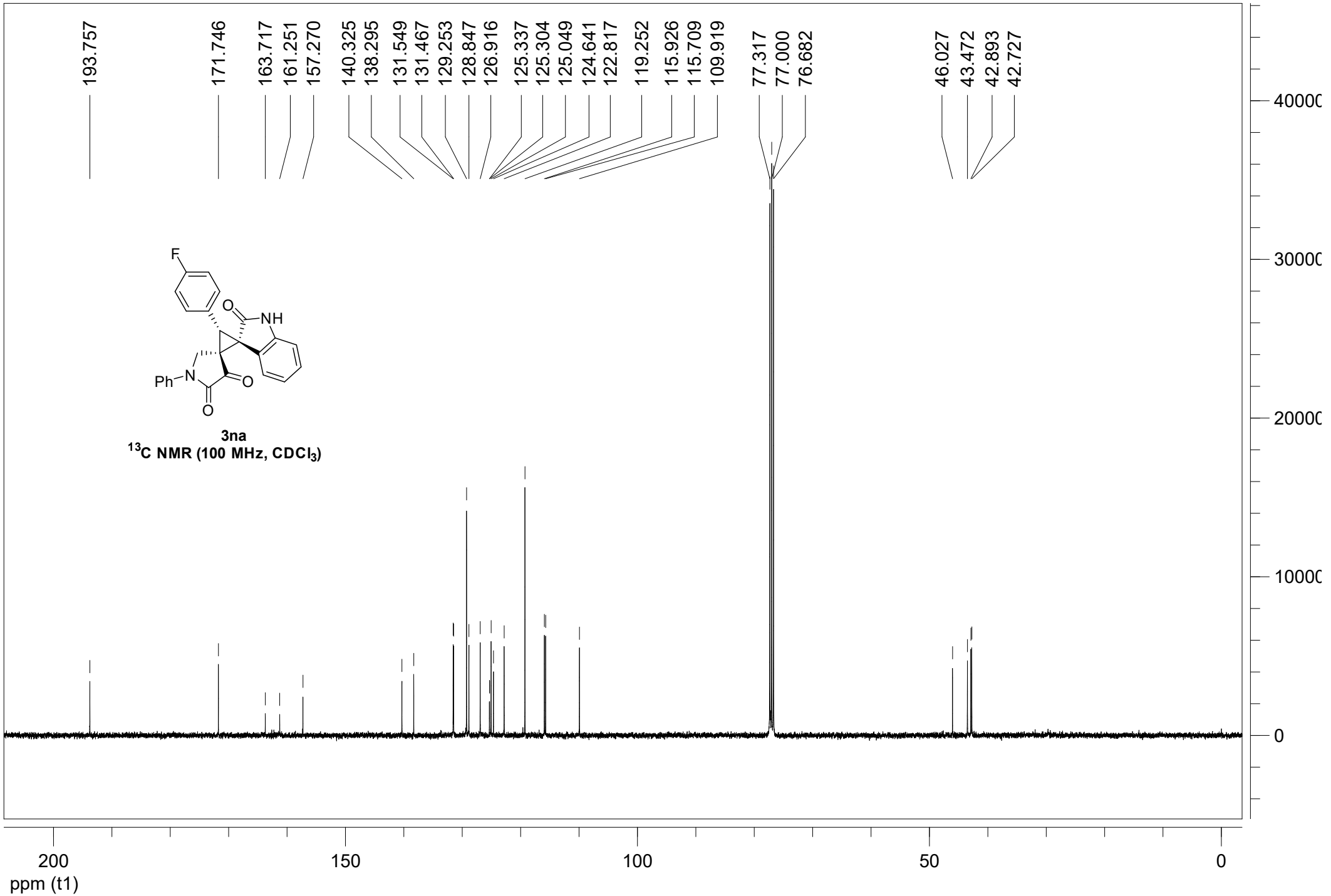
3ma

^{19}F NMR (376 MHz, $CDCl_3$)



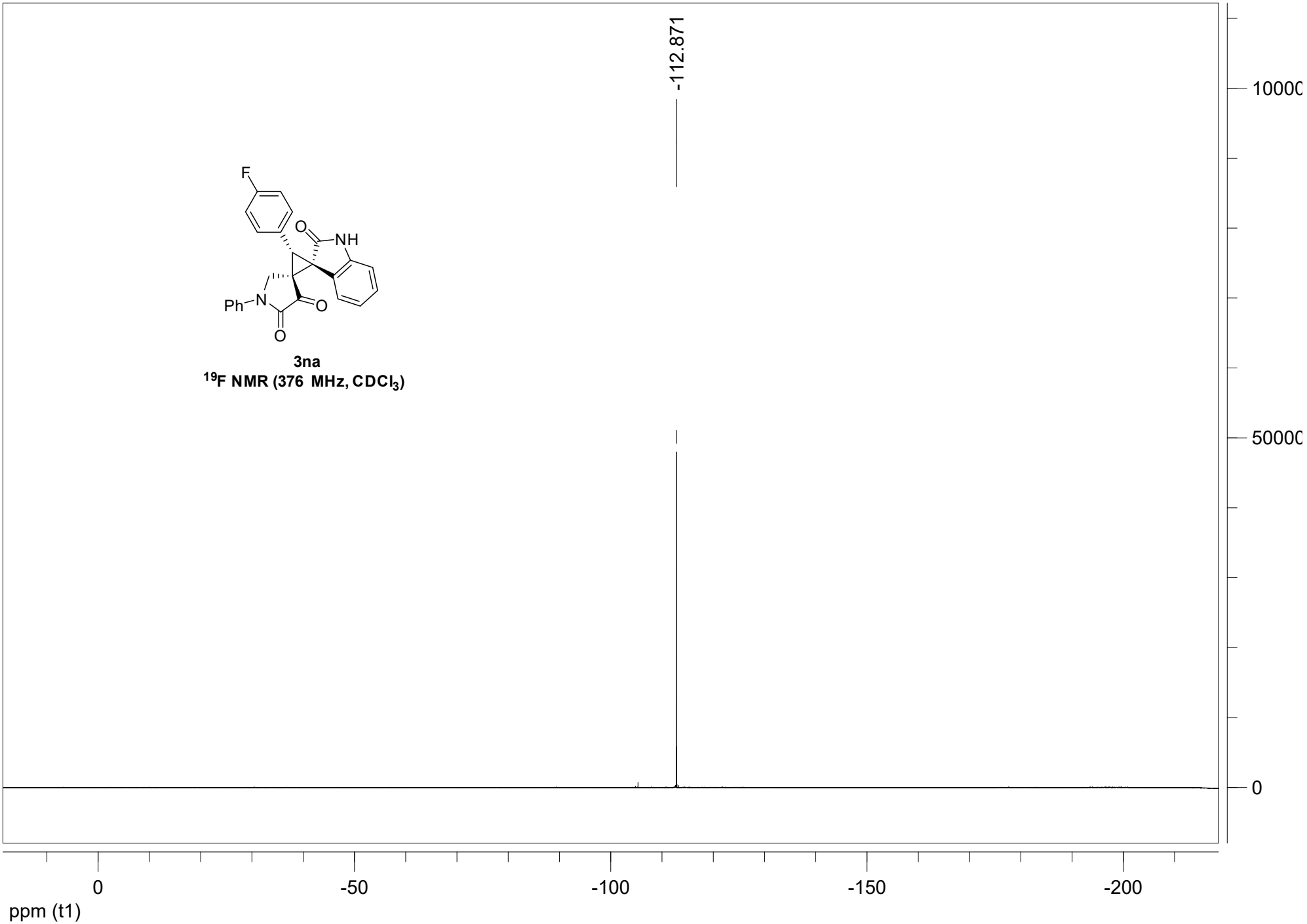
3na

^{1}H NMR (400 MHz, $CDCl_3$)



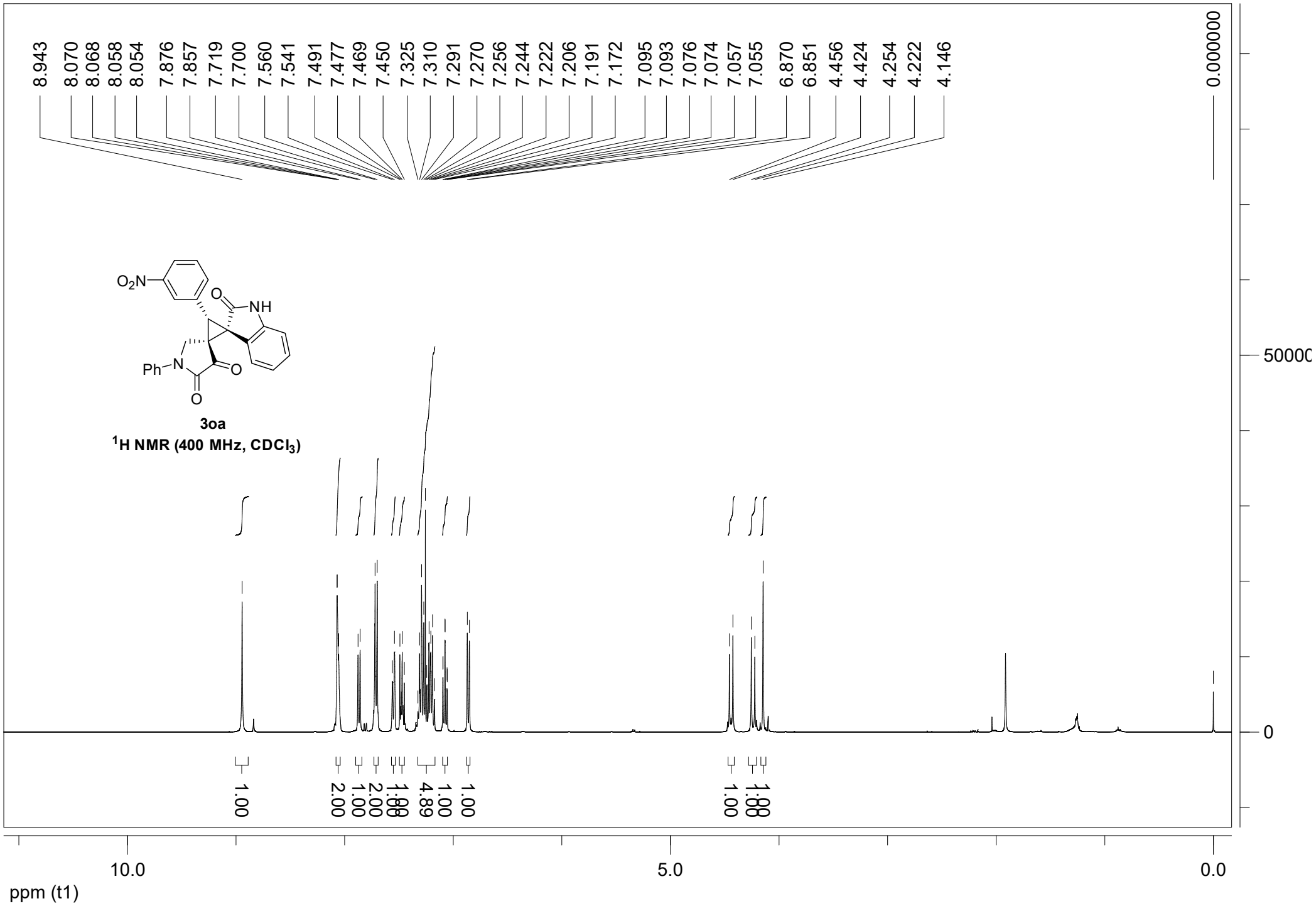
3na

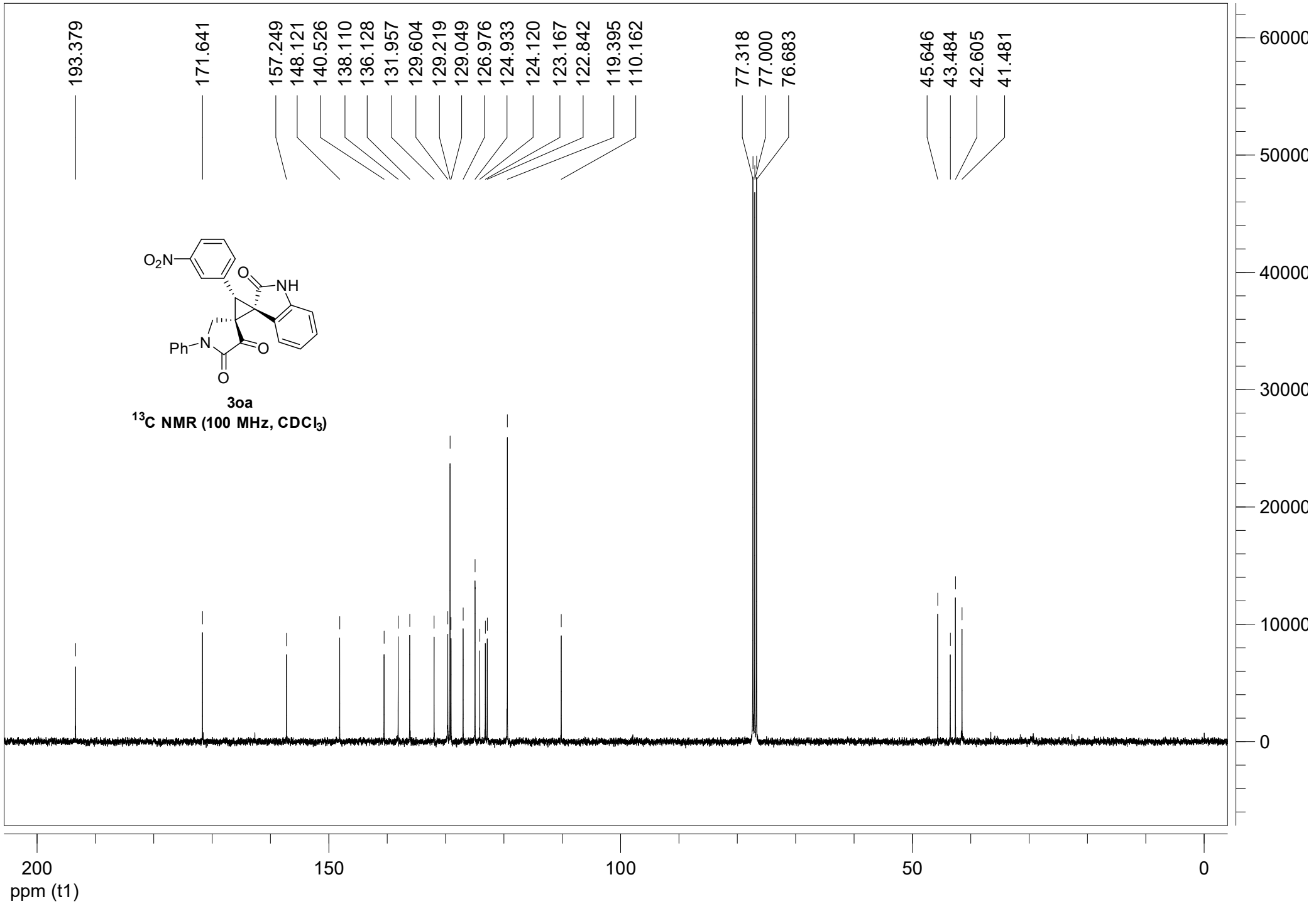
^{13}C NMR (100 MHz, $CDCl_3$)

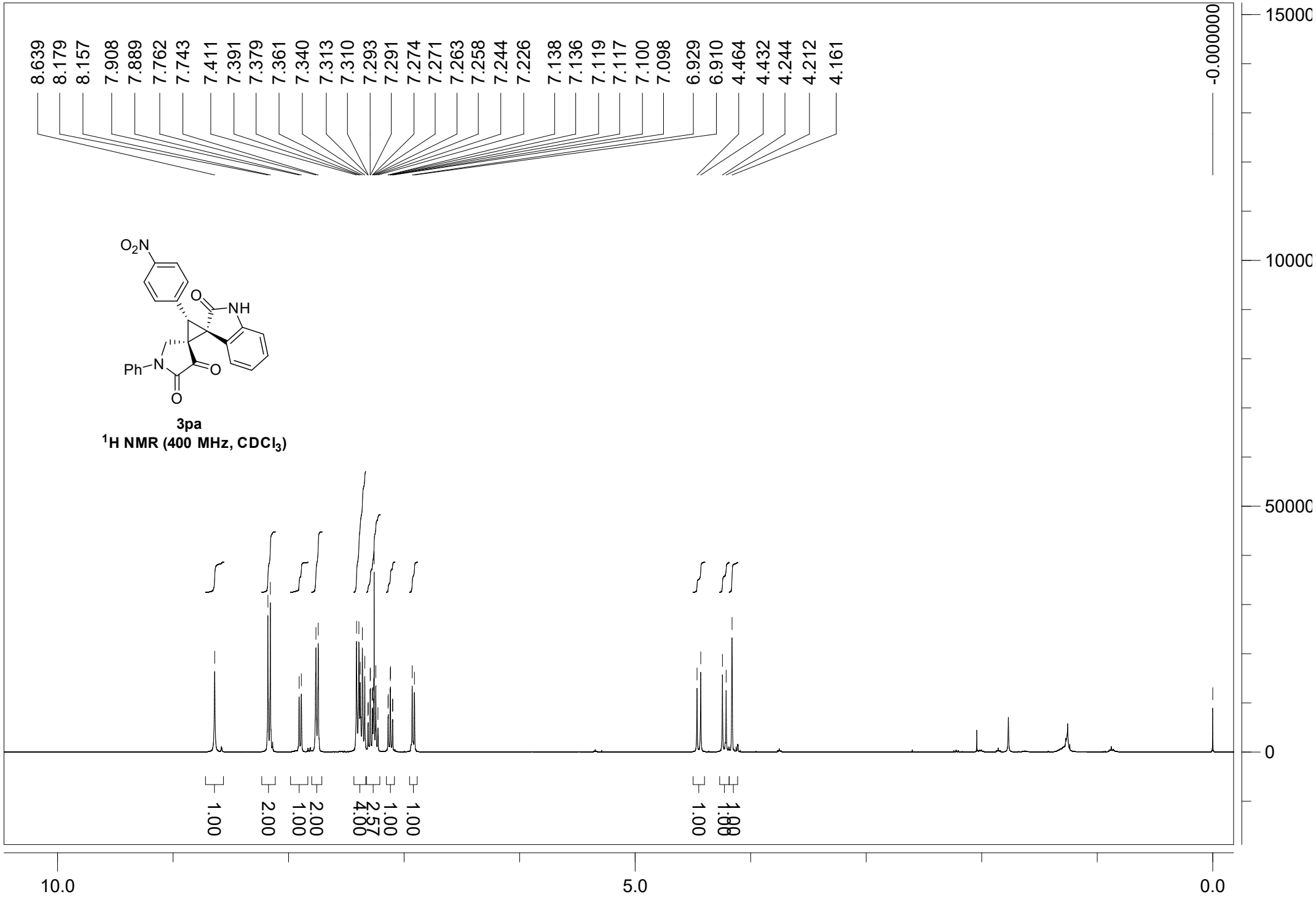


3na

^{19}F NMR (376 MHz, $CDCl_3$)

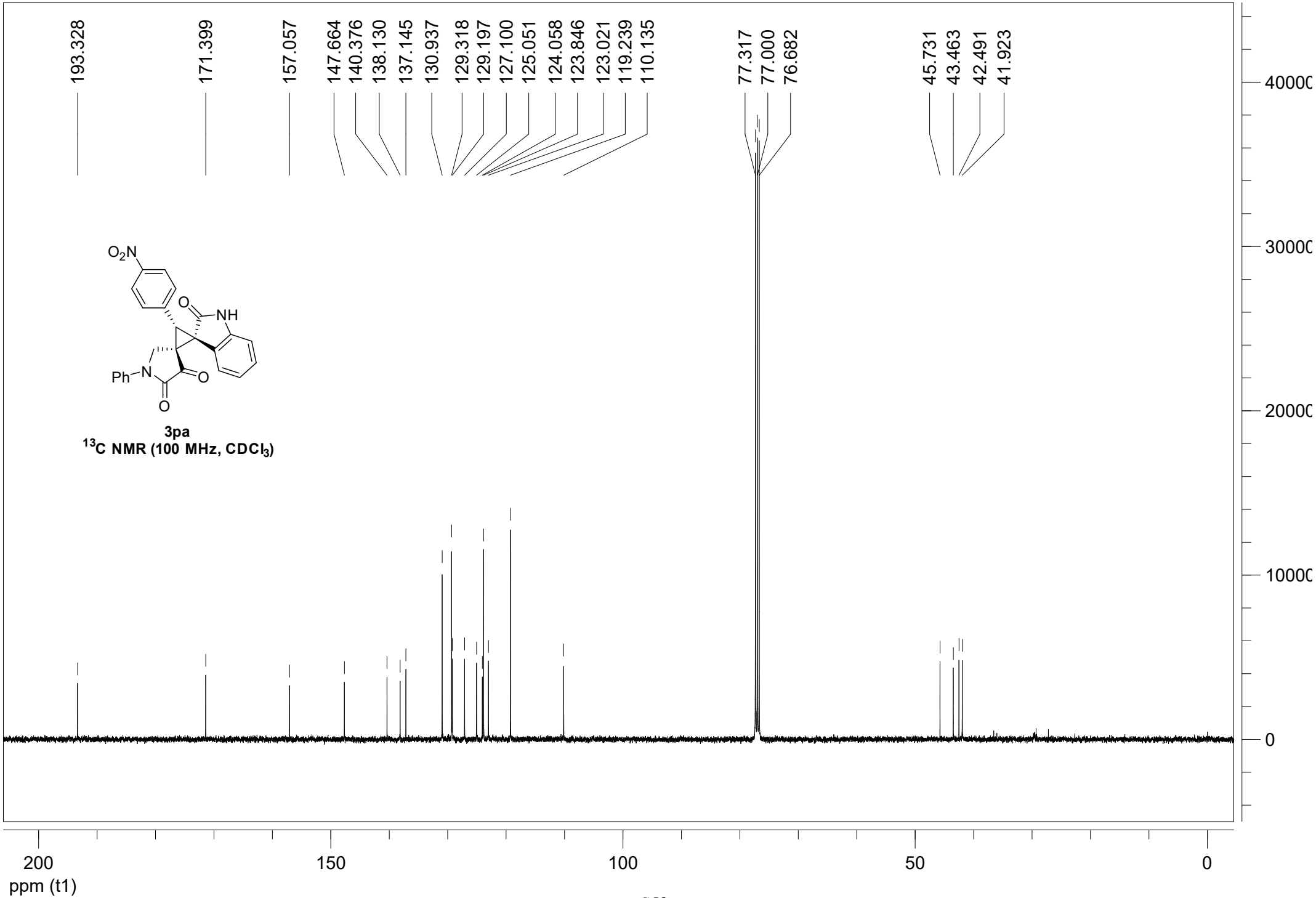

8.943
8.070
8.068
8.058
8.054
7.876
7.857
7.719
7.700
7.560
7.541
7.491
7.477
7.469
7.450
7.325
7.310
7.291
7.270
7.256
7.244
7.222
7.206
7.191
7.172
7.095
7.093
7.076
7.074
7.057
7.055
6.870
6.851
4.456
4.424
4.254
4.222
4.146
0.000000
O2N
O
NH
Ph
N
O
O
O
3oa
1H NMR (400 MHz, CDCl3)
50000
0
1.00
2.00
1.00
2.00
1.00
1.00
4.89
1.00
1.00
1.00
1.00
10.0
5.0
0.0
ppm (t1)


193.379
171.641
157.249
148.121
140.526
138.110
136.128
131.957
129.604
129.219
129.049
126.976
124.933
124.120
123.167
122.842
119.395
110.162
77.318
77.000
76.683
45.646
43.484
42.605
41.481
O2N
O
NH
Ph
N
O
O
O
3oa
13C NMR (100 MHz, CDCl3)
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)



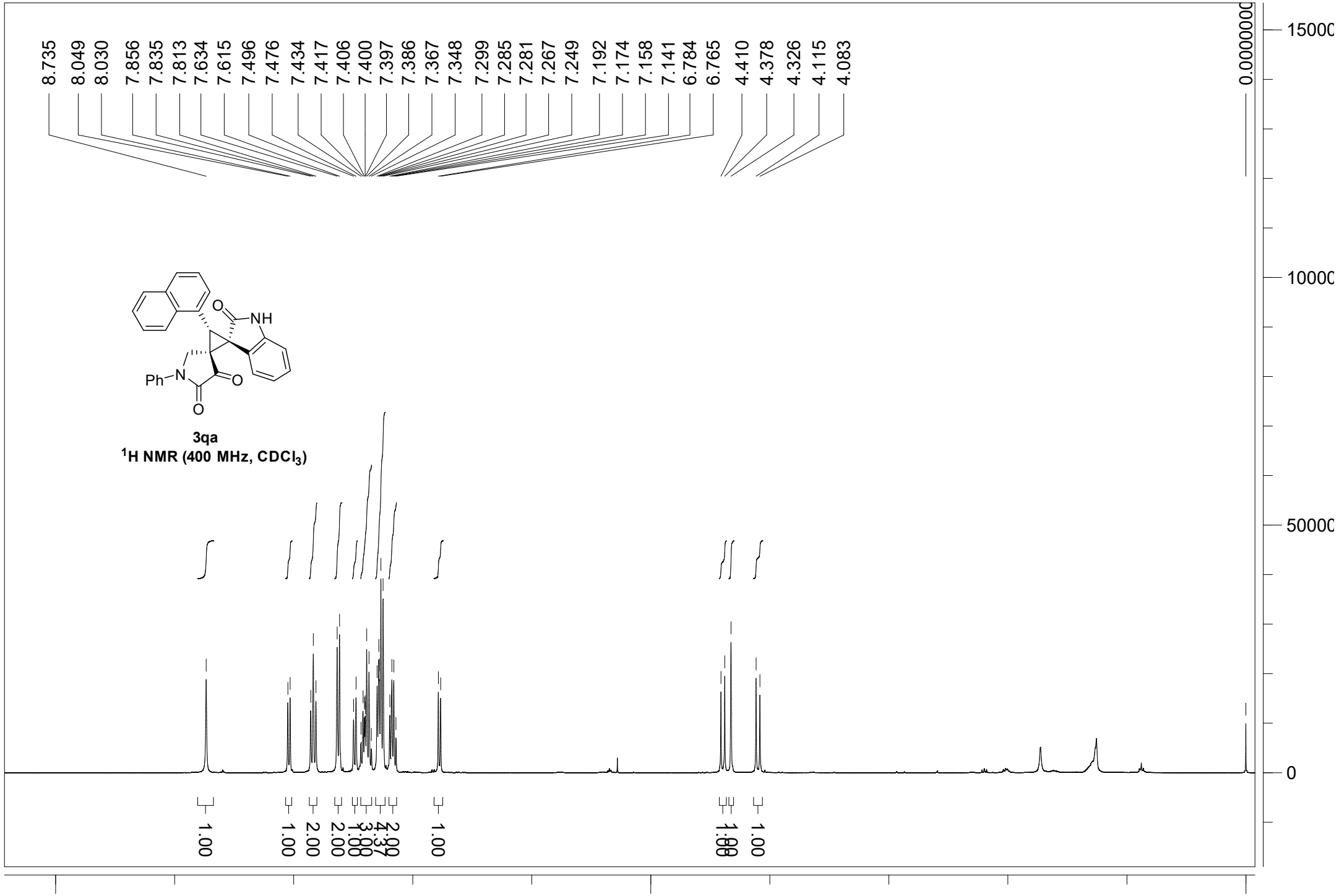
3pa

^{1}H NMR (400 MHz, $CDCl_3$)



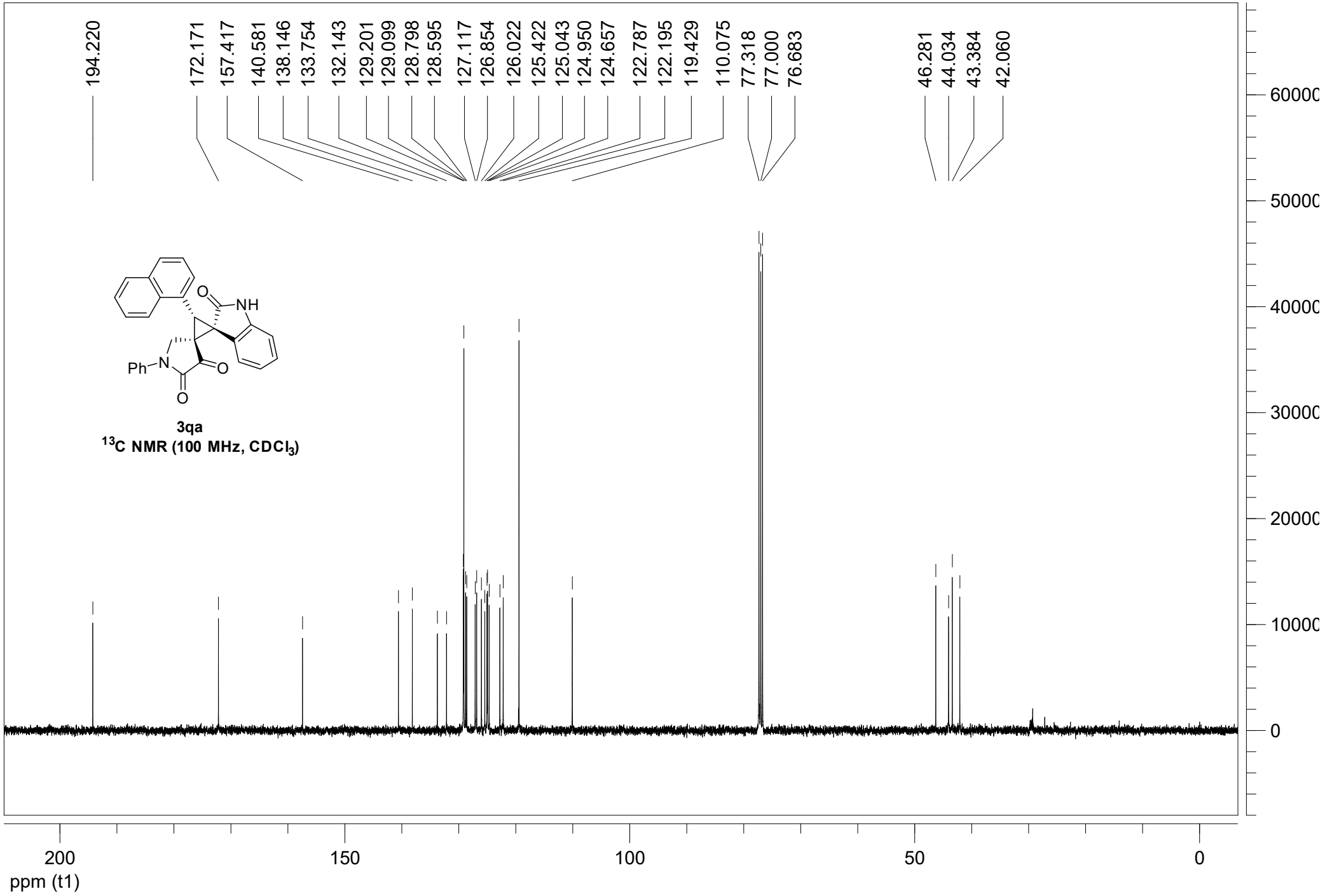
3pa

^{13}C NMR (100 MHz, $CDCl_3$)

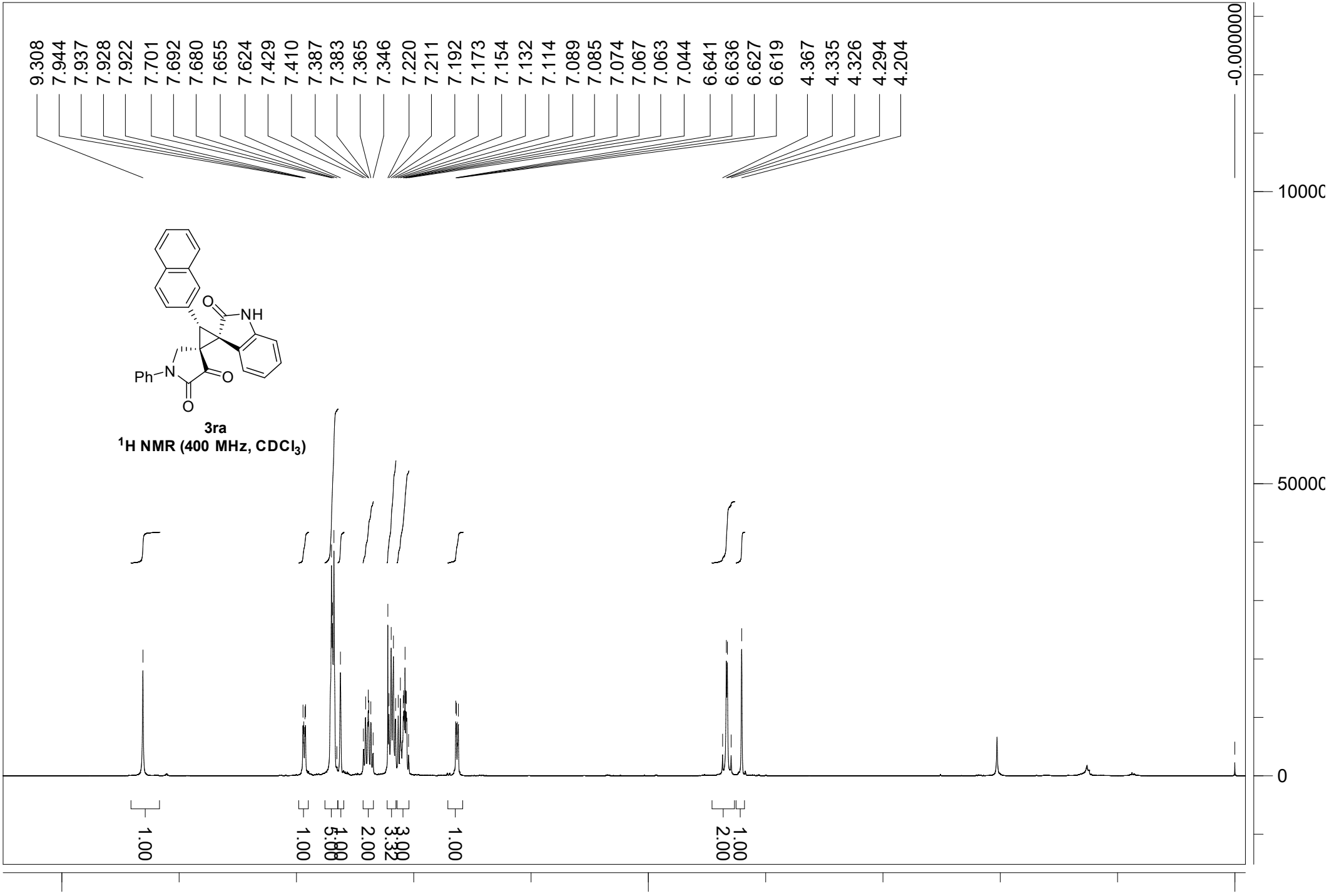


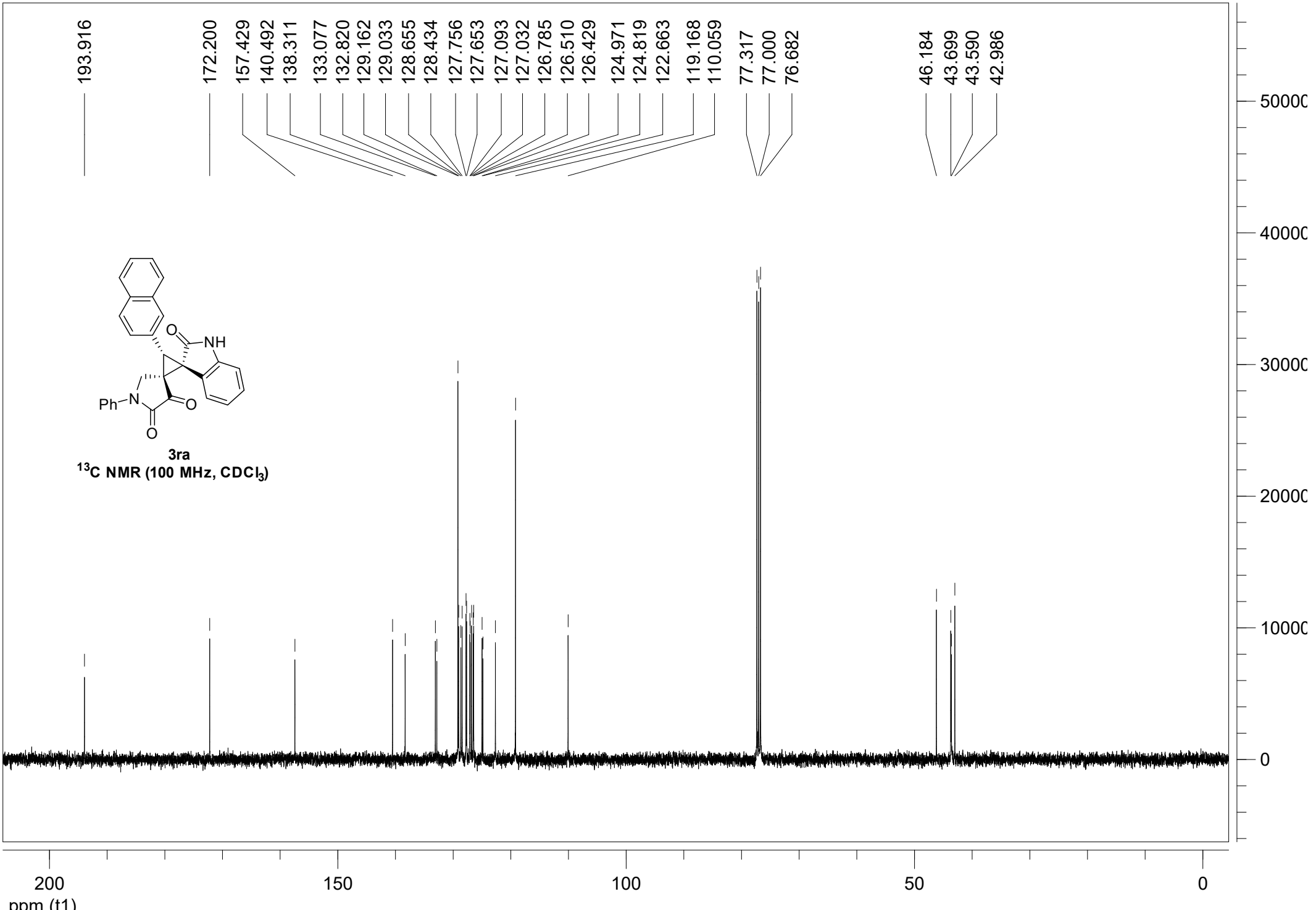
3qa

^{1}H NMR (400 MHz, $CDCl_3$)



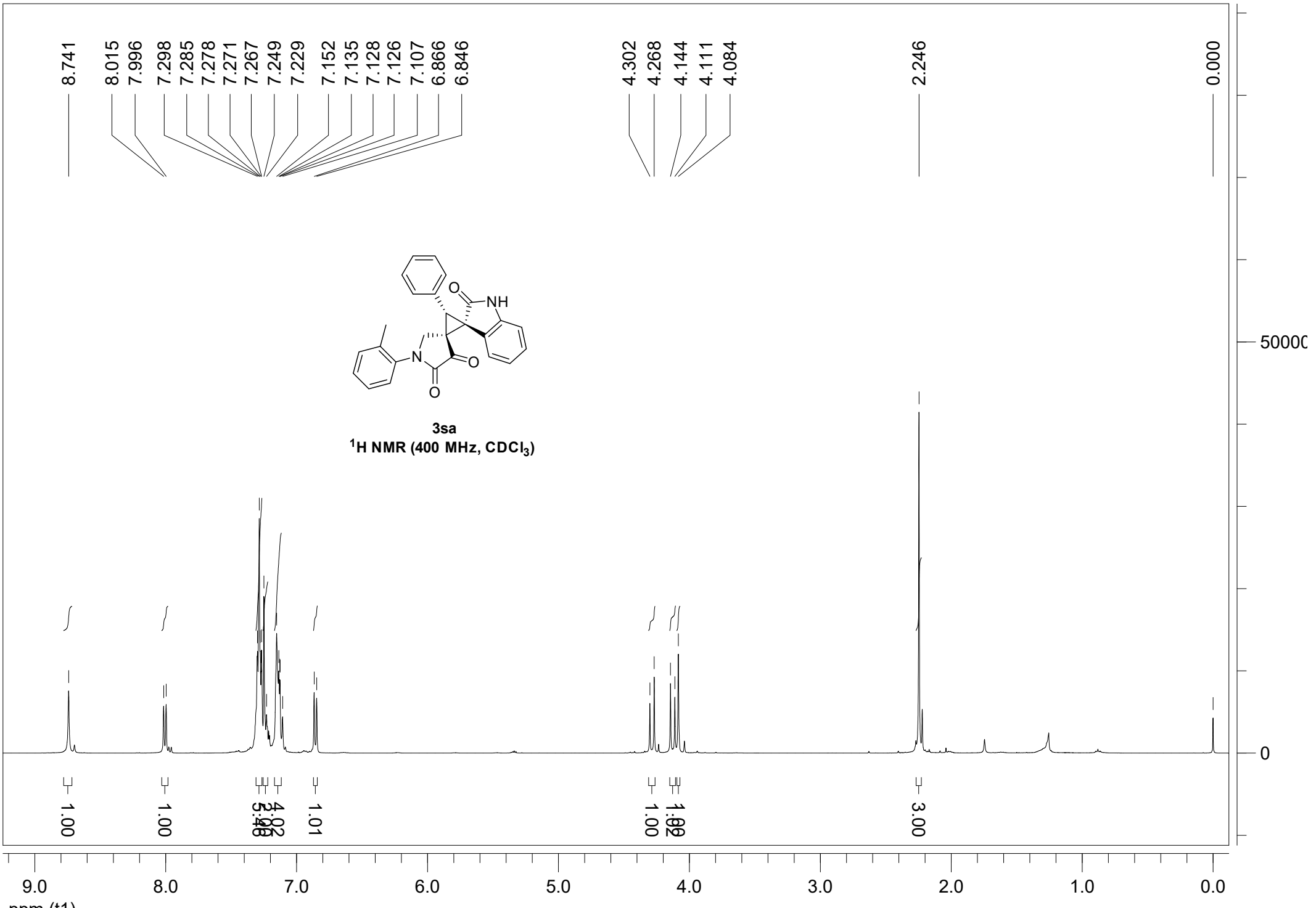
3qa
^{13}C NMR (100 MHz, $CDCl_3$)

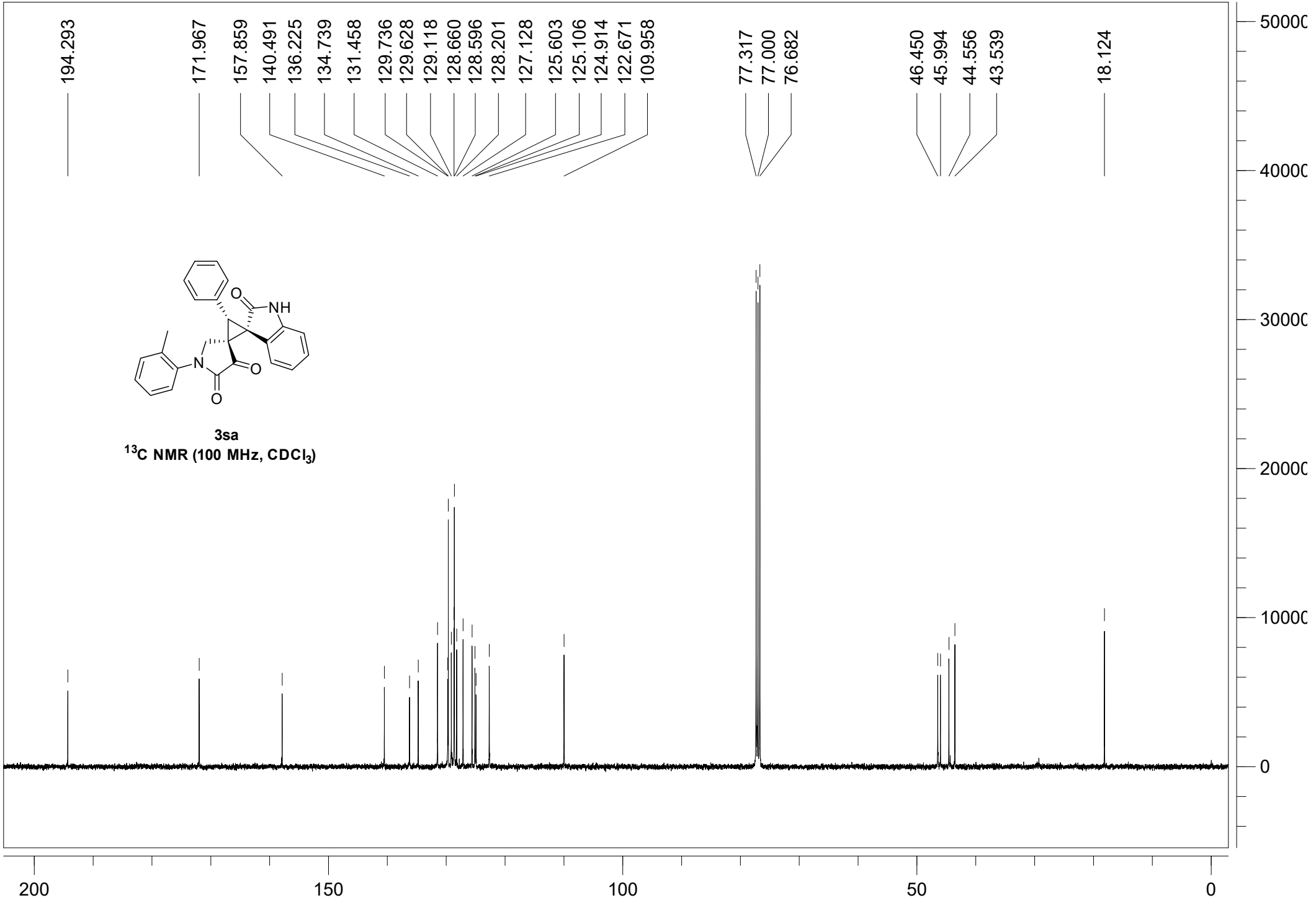

9.308
7.944
7.937
7.928
7.922
7.701
7.692
7.680
7.655
7.624
7.429
7.410
7.387
7.383
7.365
7.346
7.220
7.211
7.192
7.173
7.154
7.132
7.114
7.089
7.085
7.074
7.067
7.063
7.044
6.641
6.636
6.627
6.619
4.367
4.335
4.326
4.294
4.204
-0.000000
10000
50000
0
NH
O
Ph
N
O
O
3ra
1H NMR (400 MHz, CDCl3)
1.00
1.00
5.00
1.00
2.00
3.32
3.00
1.00
2.00
1.00
10.0
5.0
ppm (t1)

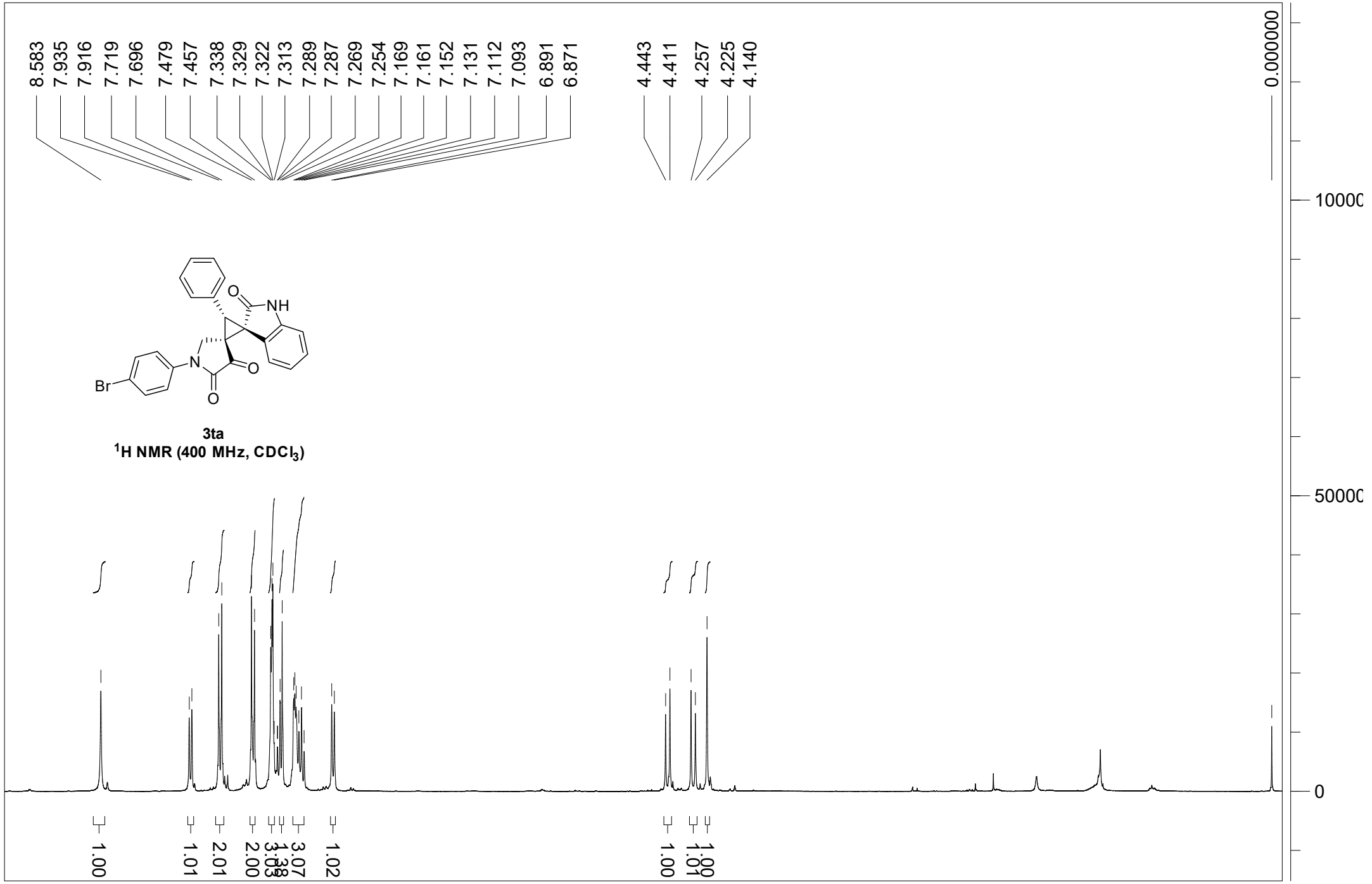


3ra

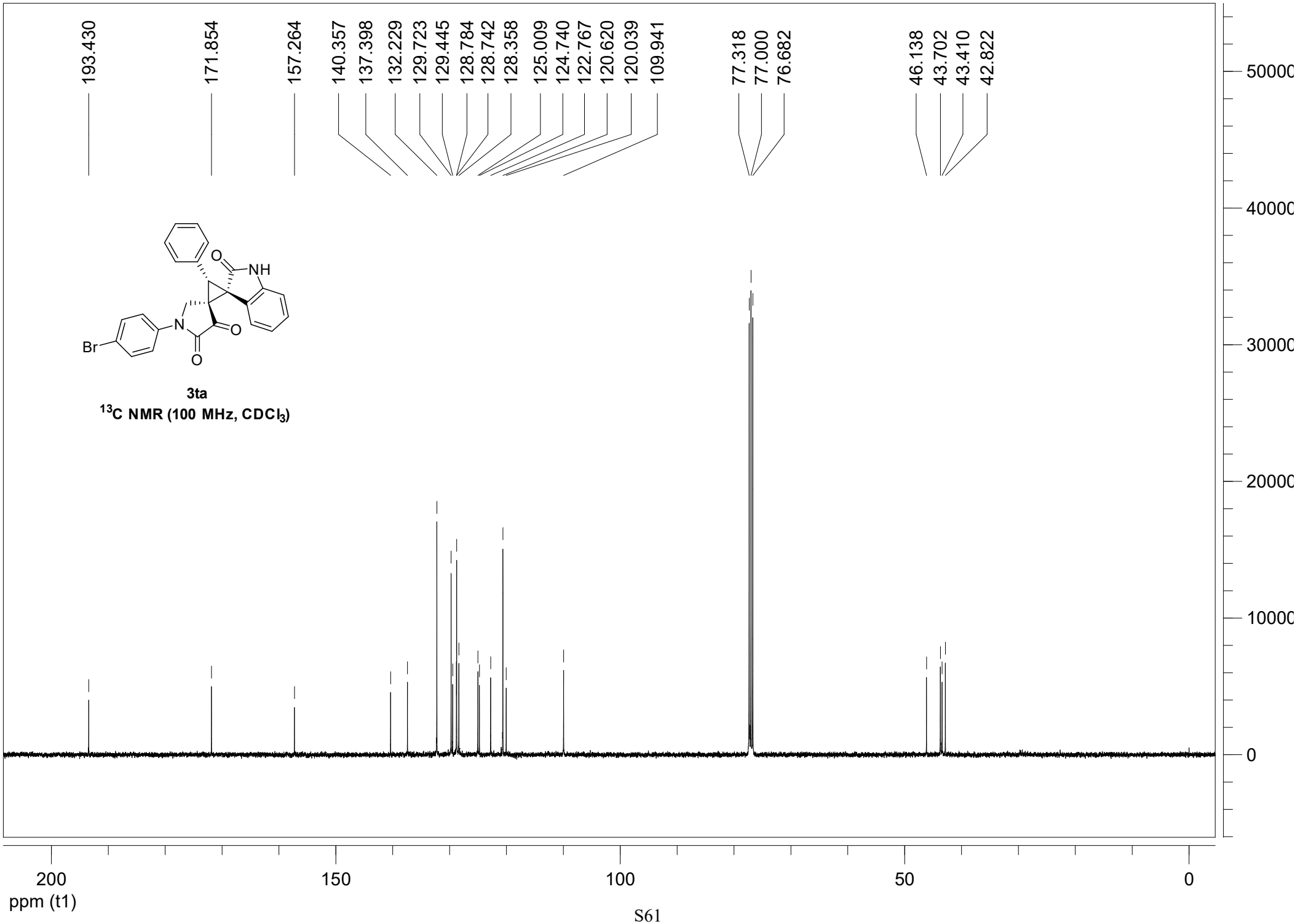
^{13}C NMR (100 MHz, $CDCl_3$)

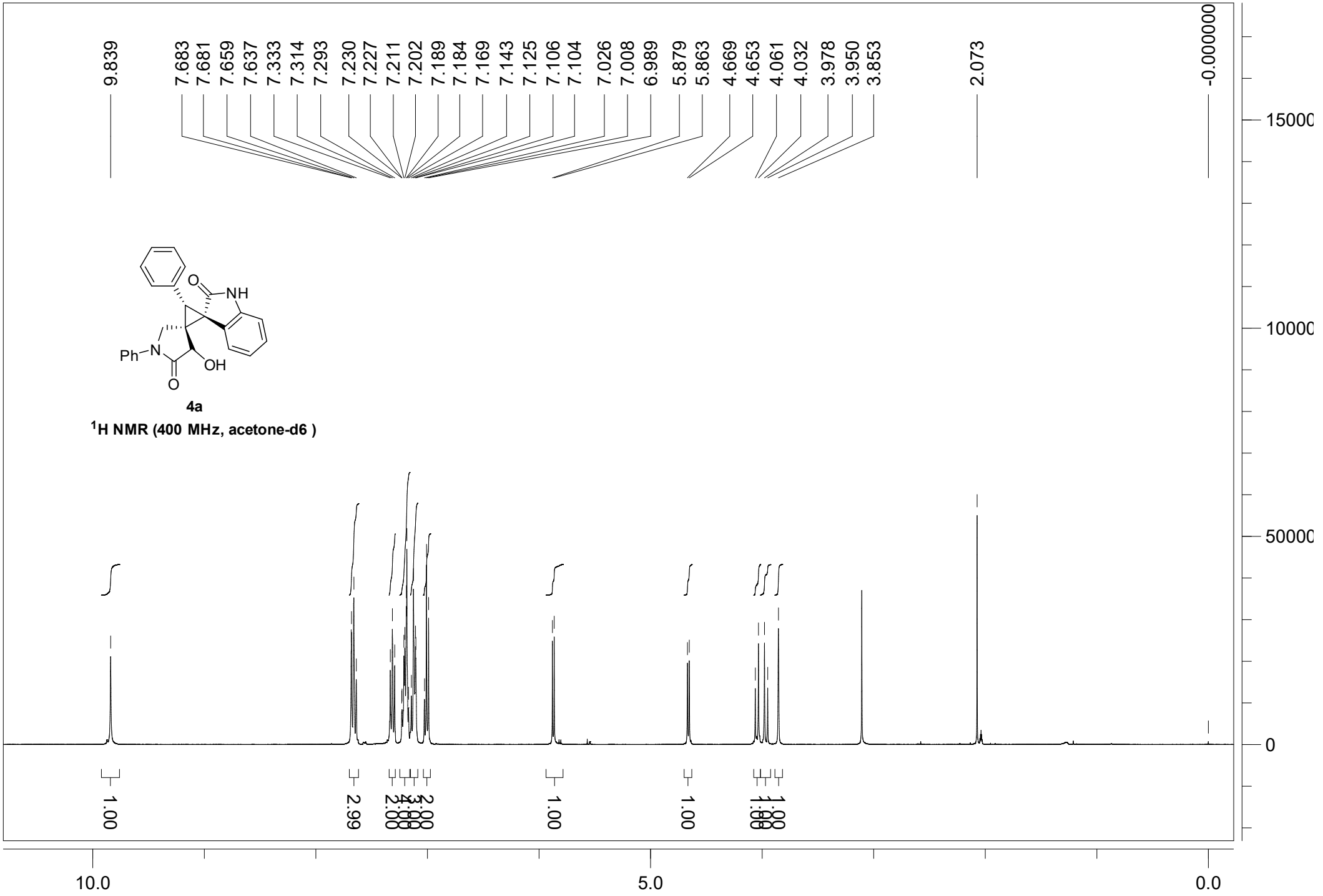

8.741
8.015
7.996
7.298
7.285
7.278
7.271
7.267
7.249
7.229
7.152
7.135
7.128
7.126
7.107
6.866
6.846
4.302
4.268
4.144
4.111
4.084
2.246
0.000
O
NH
N
O
O
3sa
1H NMR (400 MHz, CDCl3)
50000
0
1.00
1.00
1.01
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

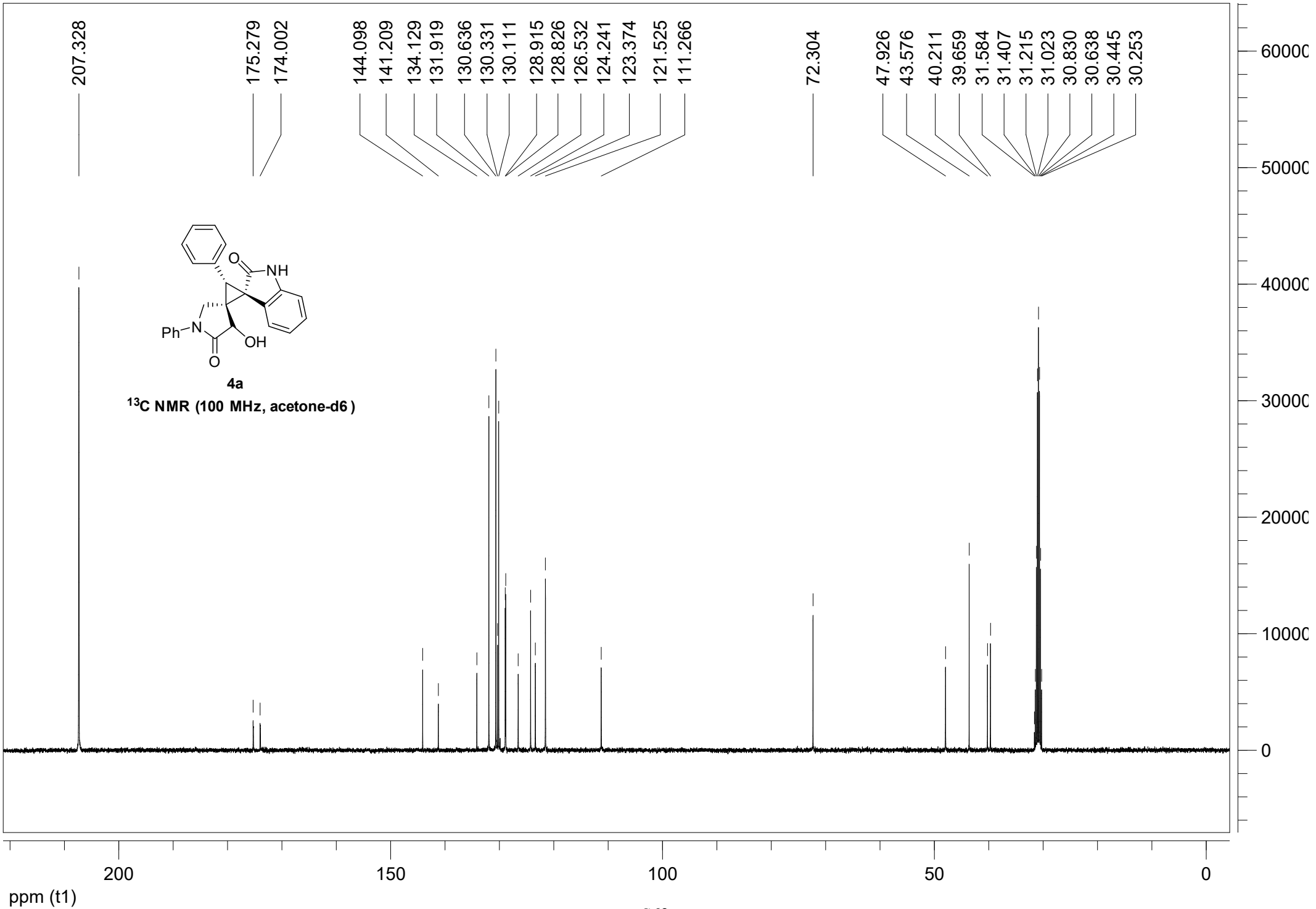

194.293
171.967
157.859
140.491
136.225
134.739
131.458
129.736
129.628
129.118
128.660
128.596
128.201
127.128
125.603
125.106
124.914
122.671
109.958
77.317
77.000
76.682
46.450
45.994
44.556
43.539
18.124
O
NH
N
O
O
3sa
13C NMR (100 MHz, CDCl3)
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)



3ta
^{1}H NMR (400 MHz, $CDCl_3$)

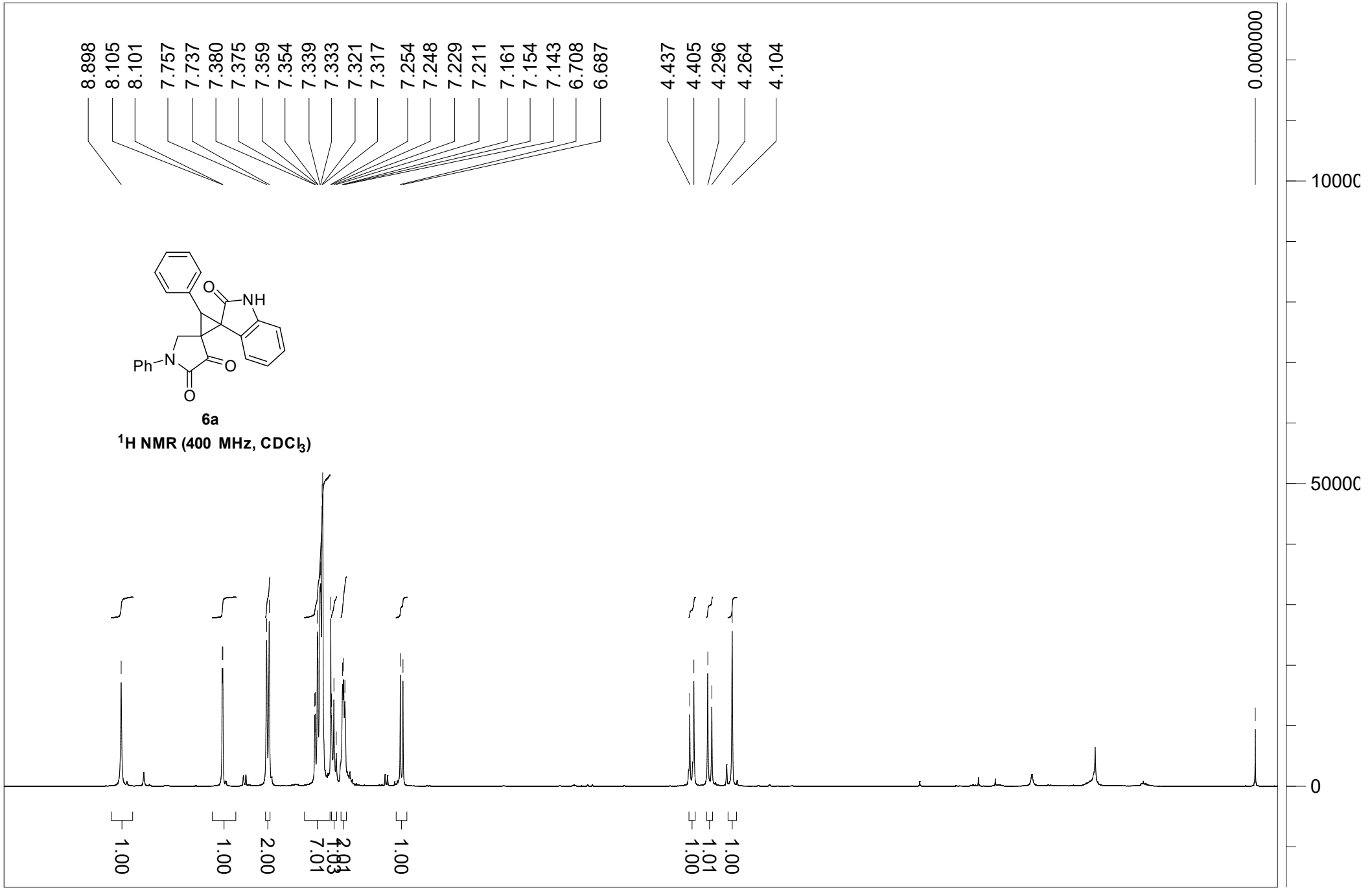

193.430
171.854
157.264
140.357
137.398
132.229
129.723
129.445
128.784
128.742
128.358
125.009
124.740
122.767
120.620
120.039
109.941
77.318
77.000
76.682
46.138
43.702
43.410
42.822
O
NH
N
O
O
Br
3ta
13C NMR (100 MHz, CDCl3)
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


9.839
7.683
7.681
7.659
7.637
7.333
7.314
7.293
7.230
7.227
7.211
7.202
7.189
7.184
7.169
7.143
7.125
7.106
7.104
7.026
7.008
6.989
5.879
5.863
4.669
4.653
4.061
4.032
3.978
3.950
3.853
2.073
-0.000000
O
NH
Ph
N
OH
O
4a
1H NMR (400 MHz, acetone-d6)
15000
10000
50000
0
1.00
2.99
2.00
1.00
1.00
1.00
1.00
10.0
5.0
0.0
ppm (t1)



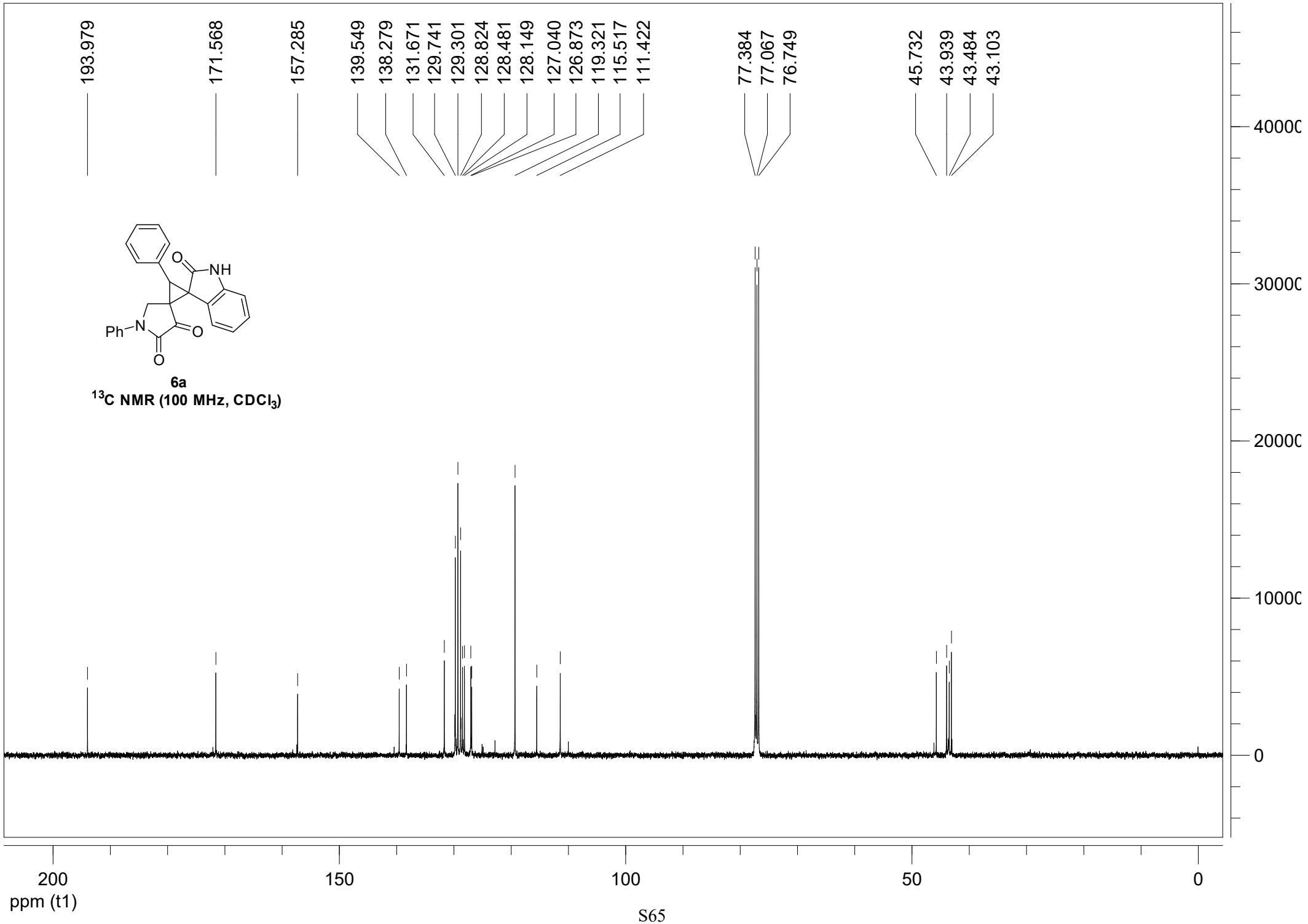
4a

^{13}C NMR (100 MHz, acetone-d6)



6a

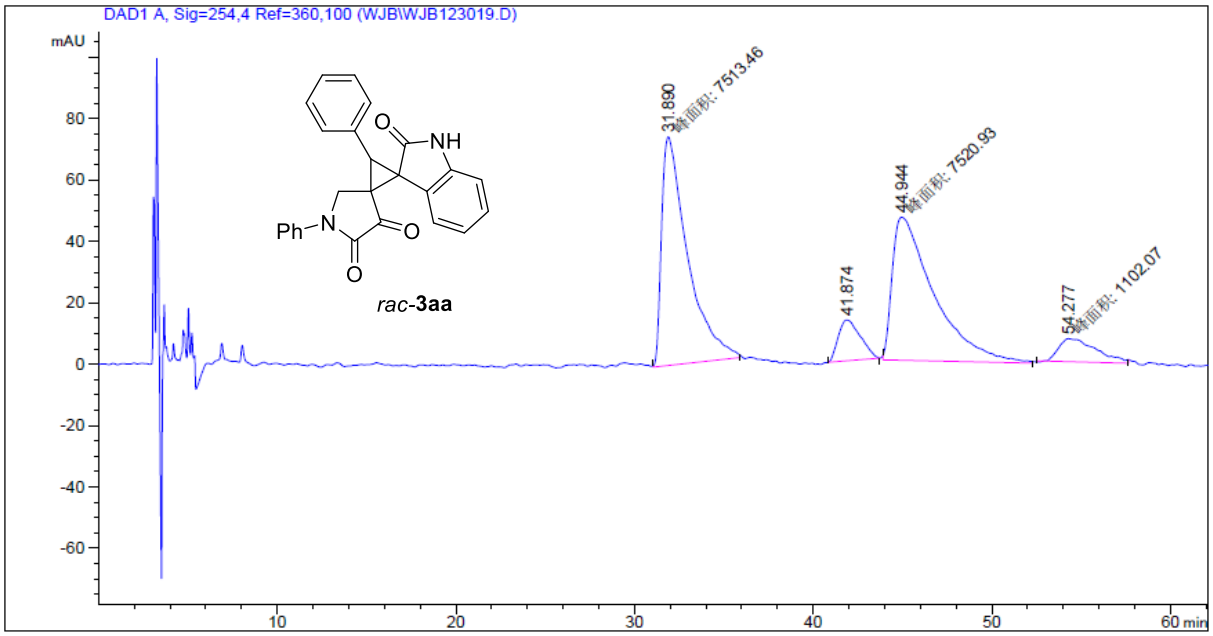
^{1}H NMR (400 MHz, $CDCl_3$)



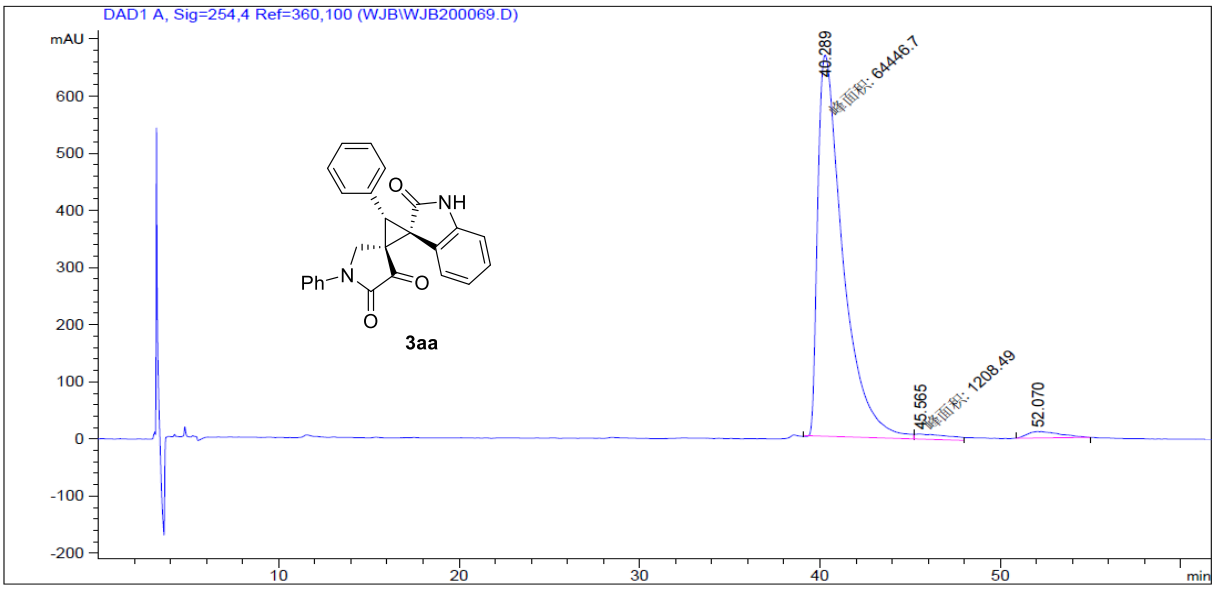
6a

^{13}C NMR (100 MHz, $CDCl_3$)

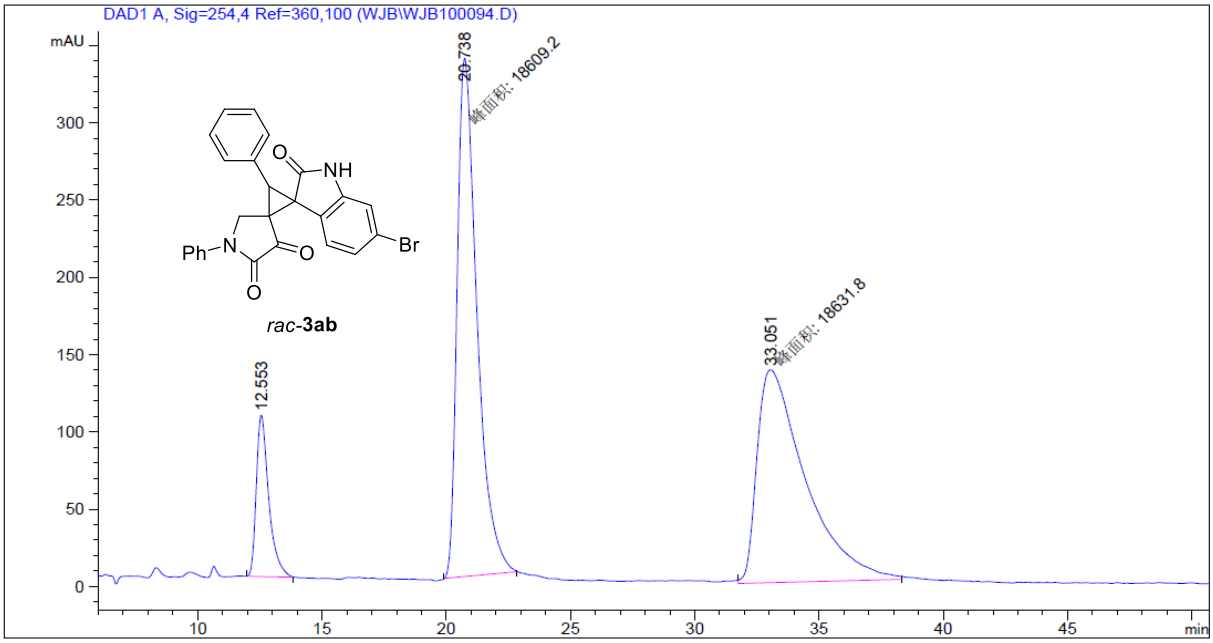
4. Copies of HPLC chromatograms of products



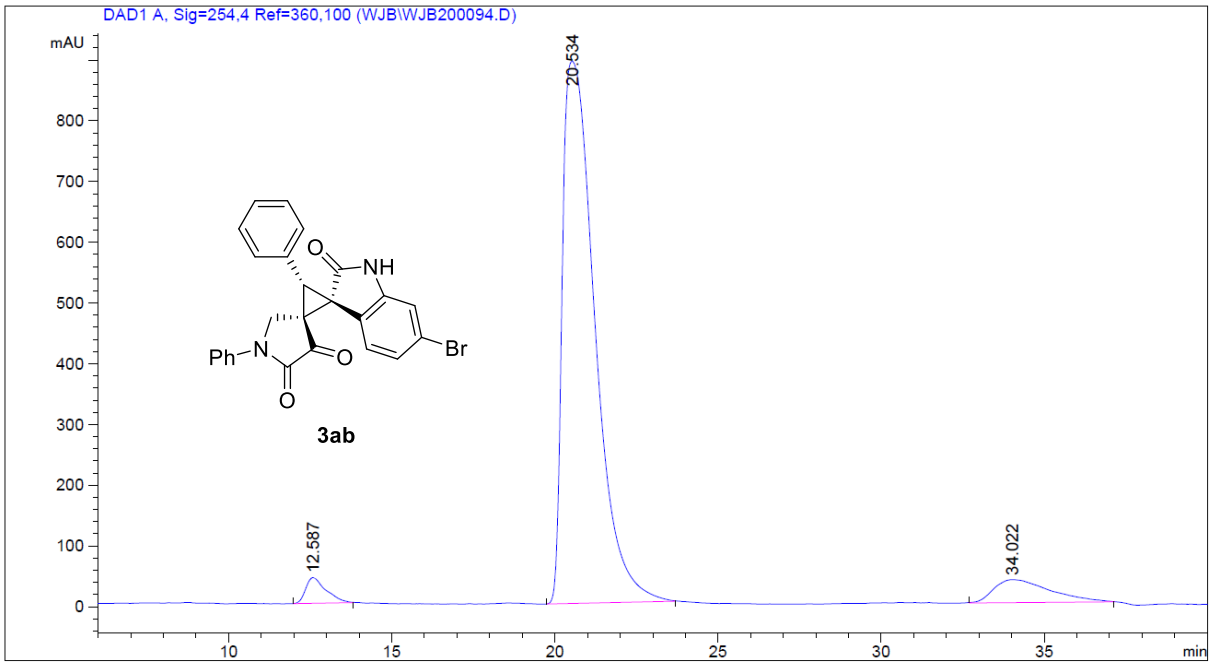
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	31.890	1.6797	7513.46387	74.55093	43.4875
2	41.874	1.0170	1140.81799	13.29632	6.6030
3	44.944	2.6883	7520.92822	46.62727	43.5307
4	54.277	2.4545	1102.06836	7.48327	6.3787



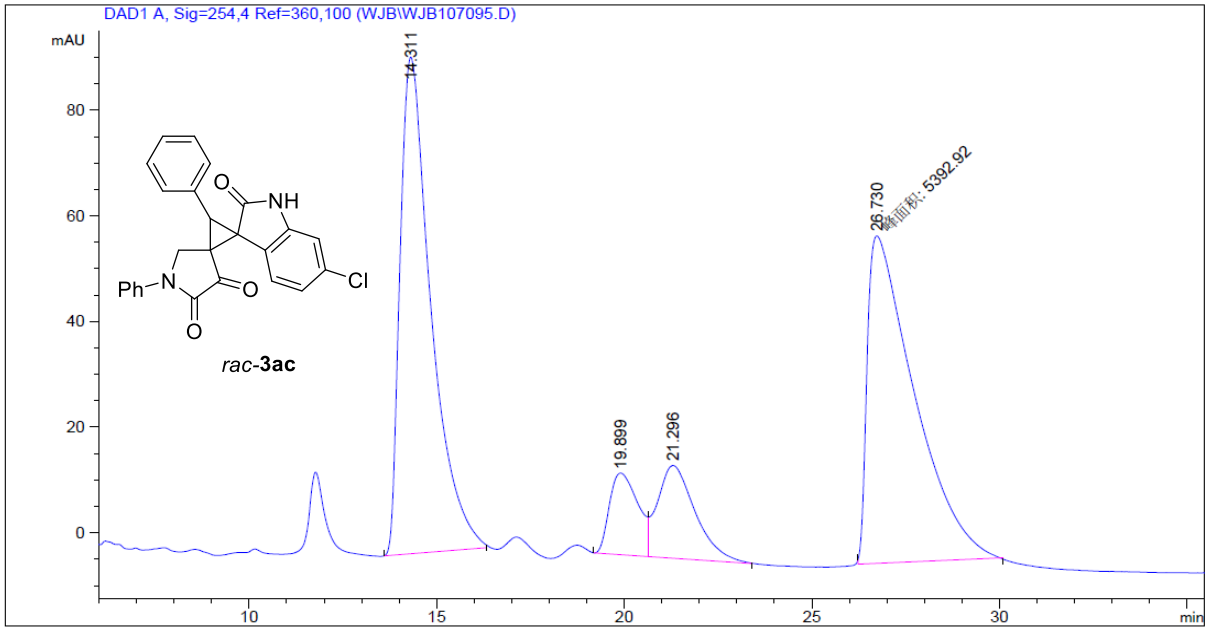
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	40.289	1.6104	6.44467e4	666.99982	96.0759
2	45.565	2.2833	1208.48877	8.82124	1.8016
3	52.070	1.5139	1423.78442	11.06019	2.1225



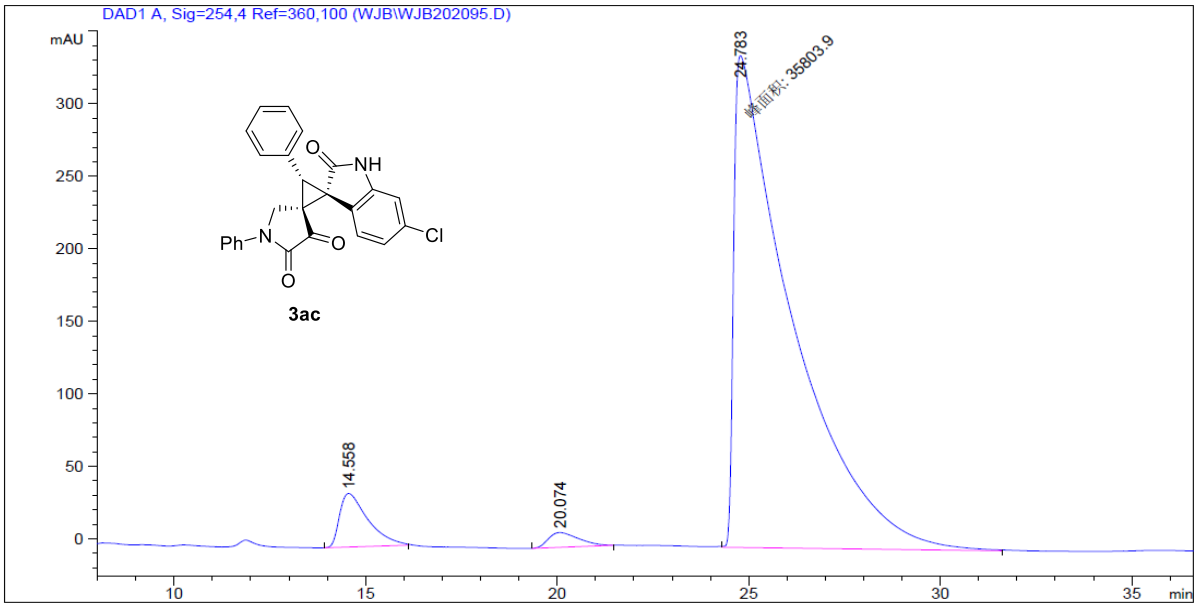
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	12.553	0.5201	3630.78125	104.31587	8.8833
2	20.738	0.9248	1.86092e4	335.36548	45.5306
3	33.051	2.2572	1.86318e4	137.57329	45.5860



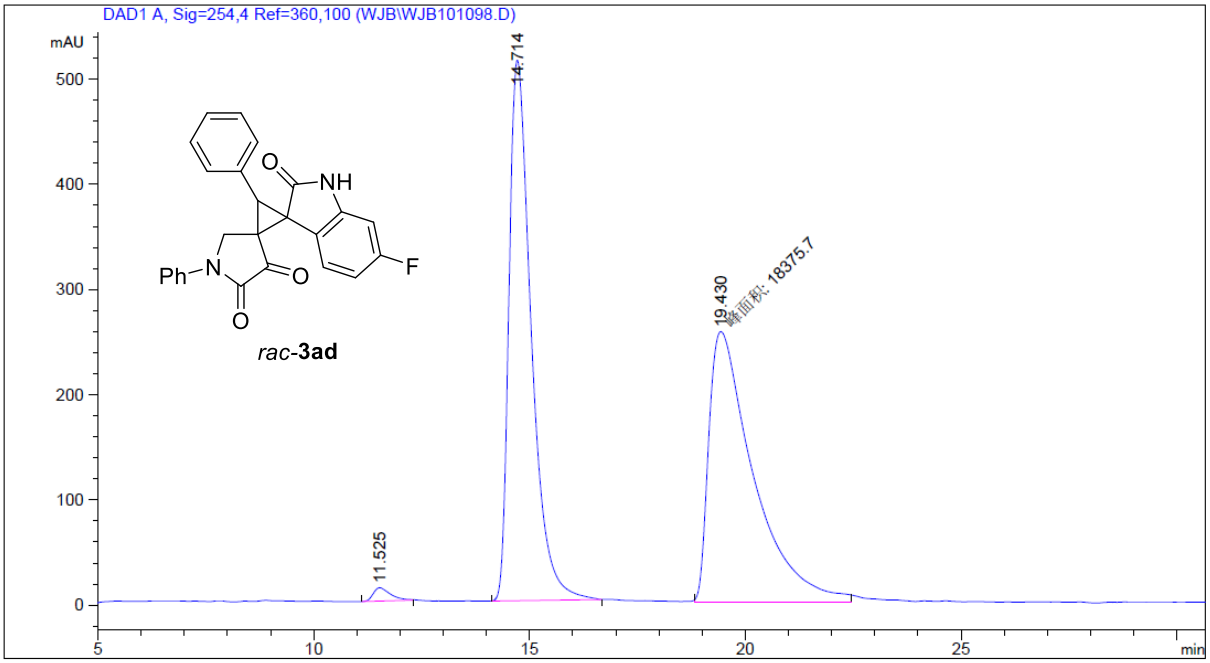
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	12.587	0.6254	1820.58130	42.35913	2.6792
2	20.534	0.9126	6.15782e4	892.90381	90.6213
3	34.022	1.4710	4552.39062	37.85654	6.6995



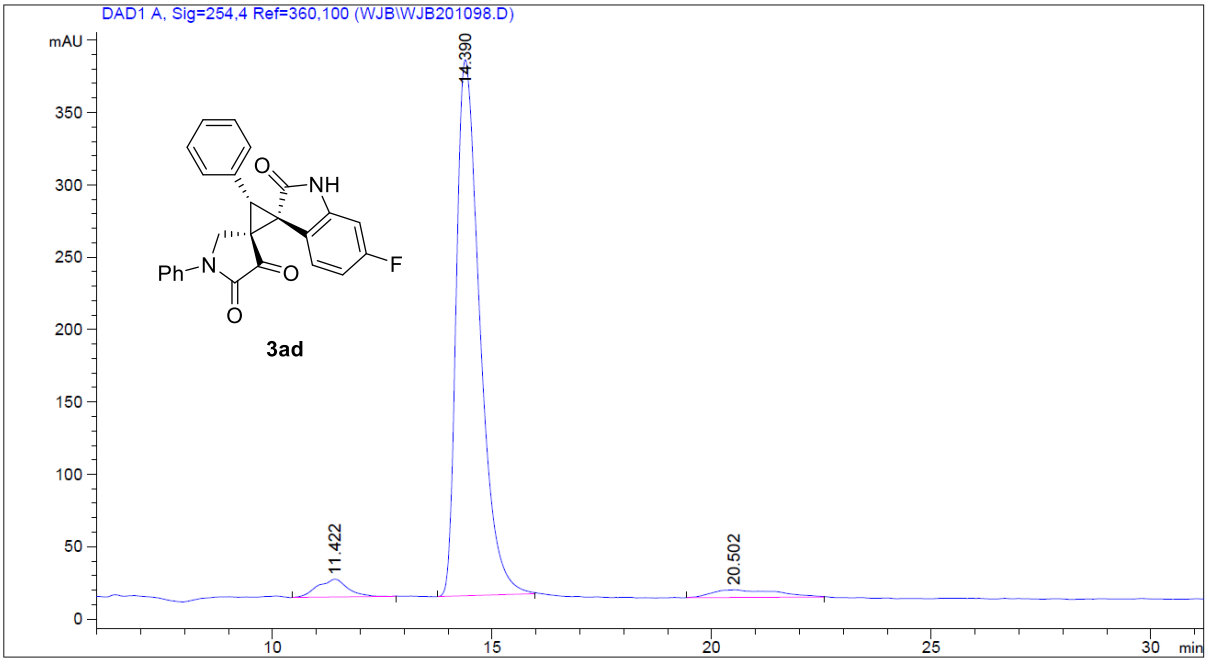
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	14.311	0.8521	5385.45947	94.03922	42.0035
2	19.899	0.7582	819.05292	15.45826	6.3881
3	21.296	0.9309	1224.02295	17.56669	9.5467
4	26.730	1.4499	5392.91797	61.99150	42.0617



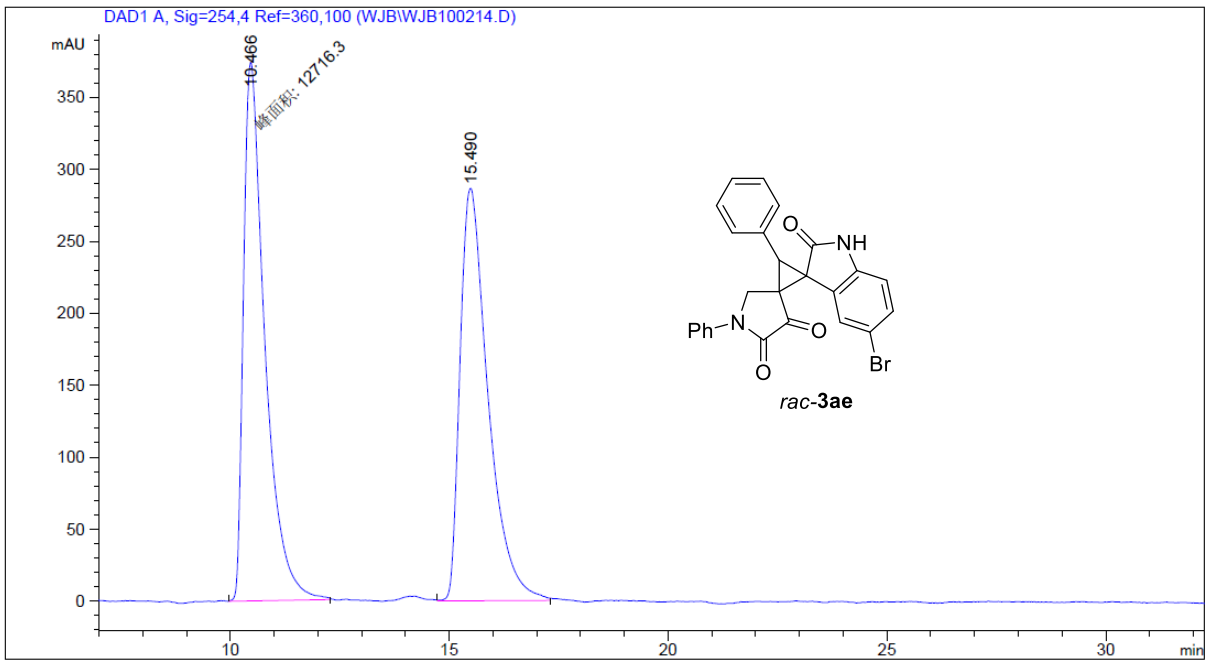
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	14.558	0.7415	1903.02344	36.90892	4.9730
2	20.074	0.7563	559.96442	10.20267	1.4633
3	24.783	1.7602	3.58039e4	339.00955	93.5637



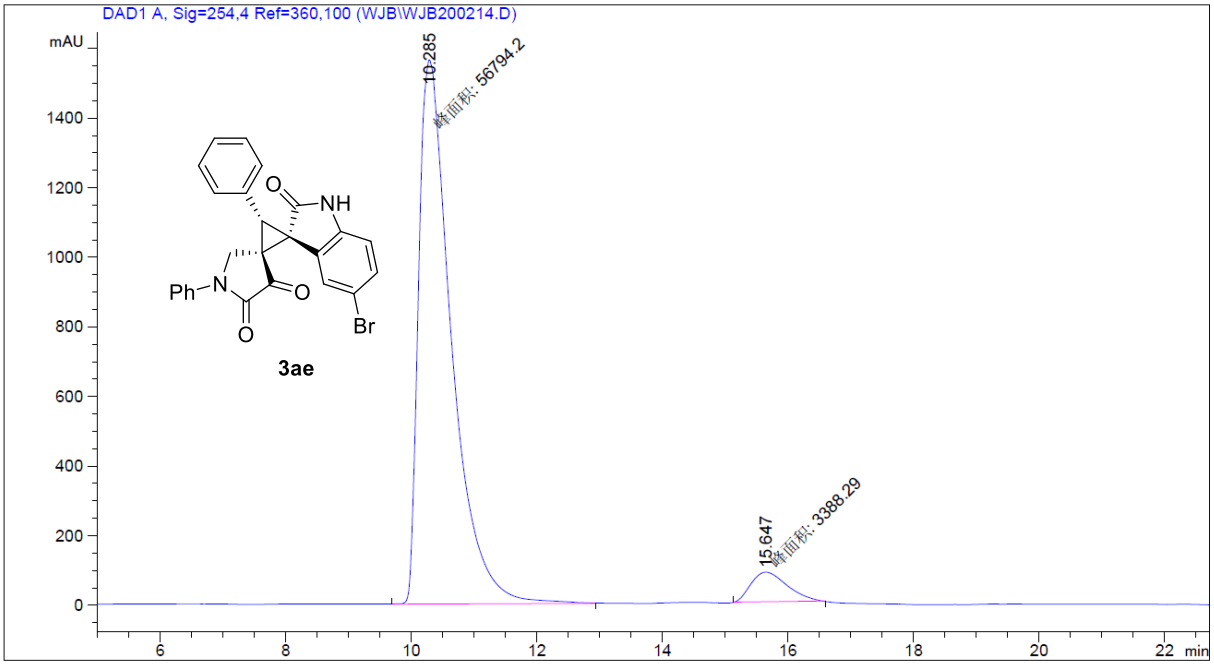
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.525	0.3996	350.15421	12.70099	0.9429
2	14.714	0.5380	1.84099e4	513.85010	49.5746
3	19.430	1.1924	1.83757e4	256.85327	49.4825



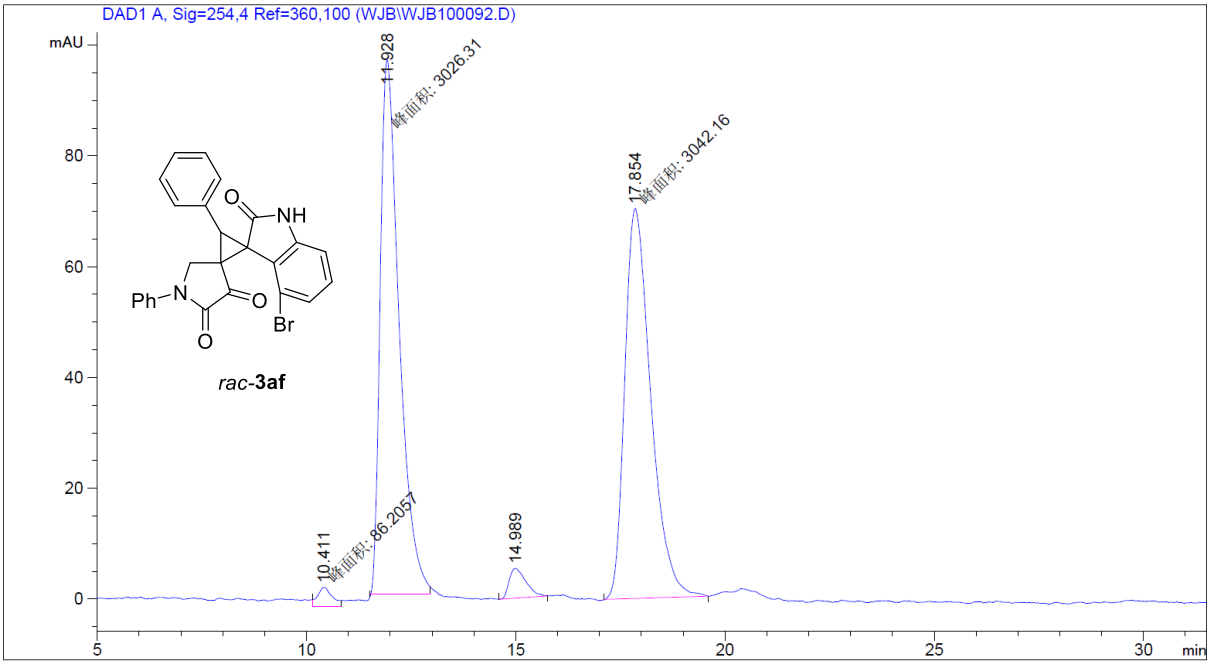
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.422	0.6220	582.56946	12.26800	3.9209
2	14.390	0.5644	1.37237e4	370.30884	92.3651
3	20.502	1.2362	551.83246	5.24205	3.7140



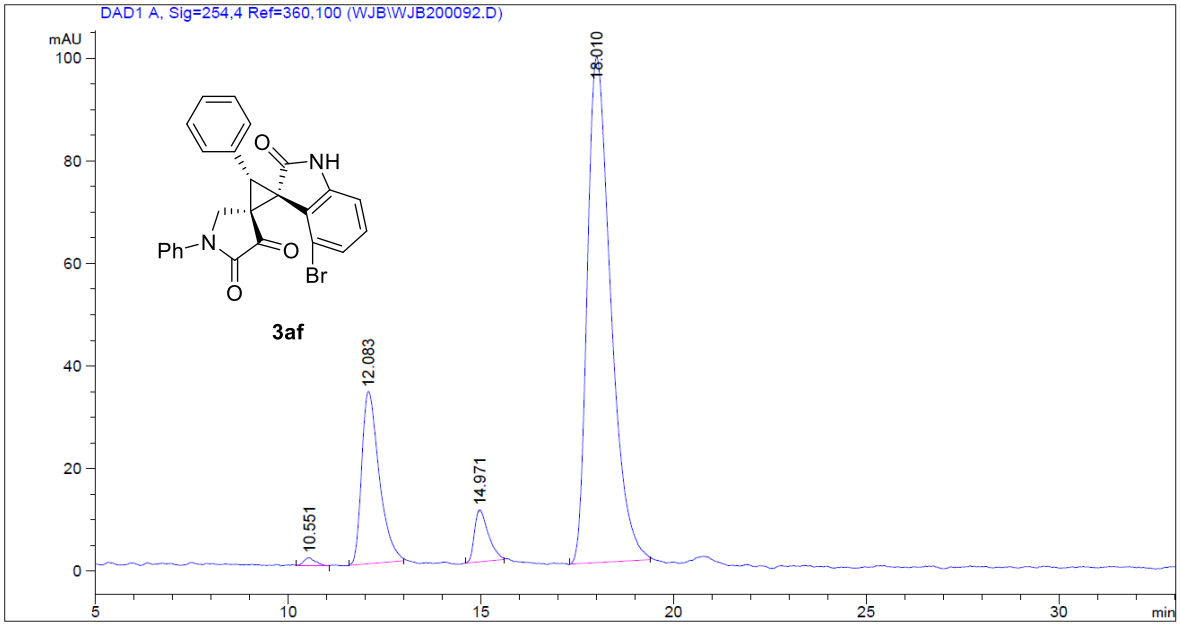
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	10.466	0.5663	1.27163e4	374.26309	49.9555
2	15.490	0.6567	1.27390e4	286.50803	50.0445



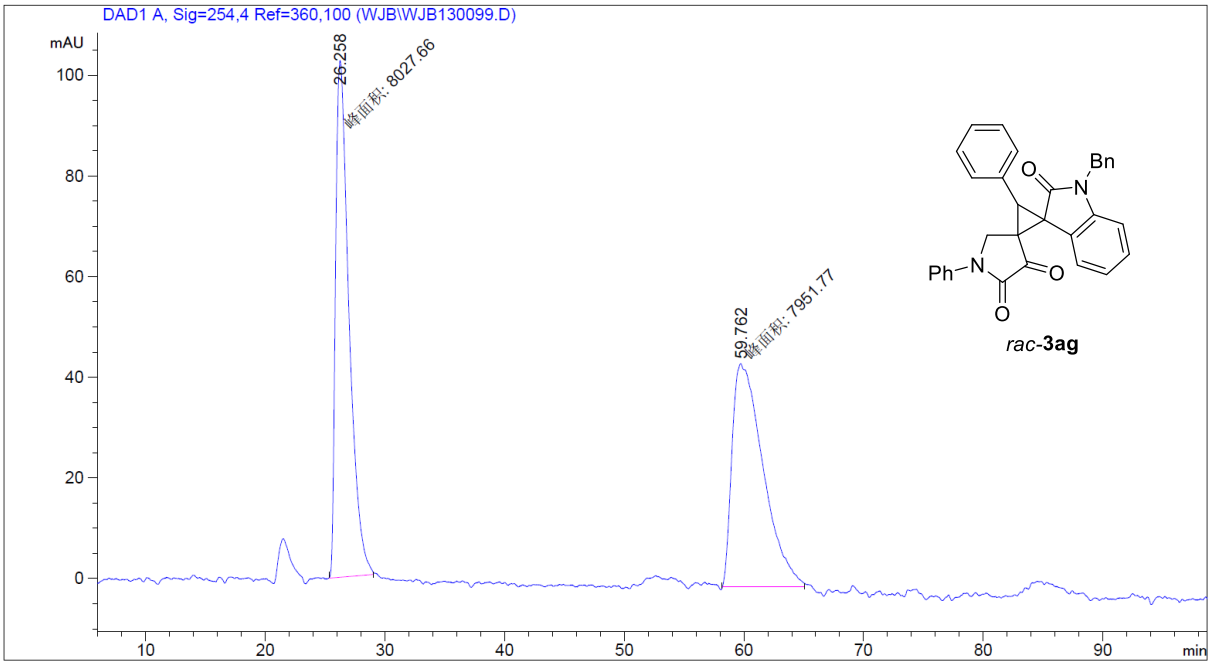
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	10.285	0.6053	5.67942e4	1563.85767	94.3700
2	15.647	0.6658	3388.29102	84.81294	5.6300



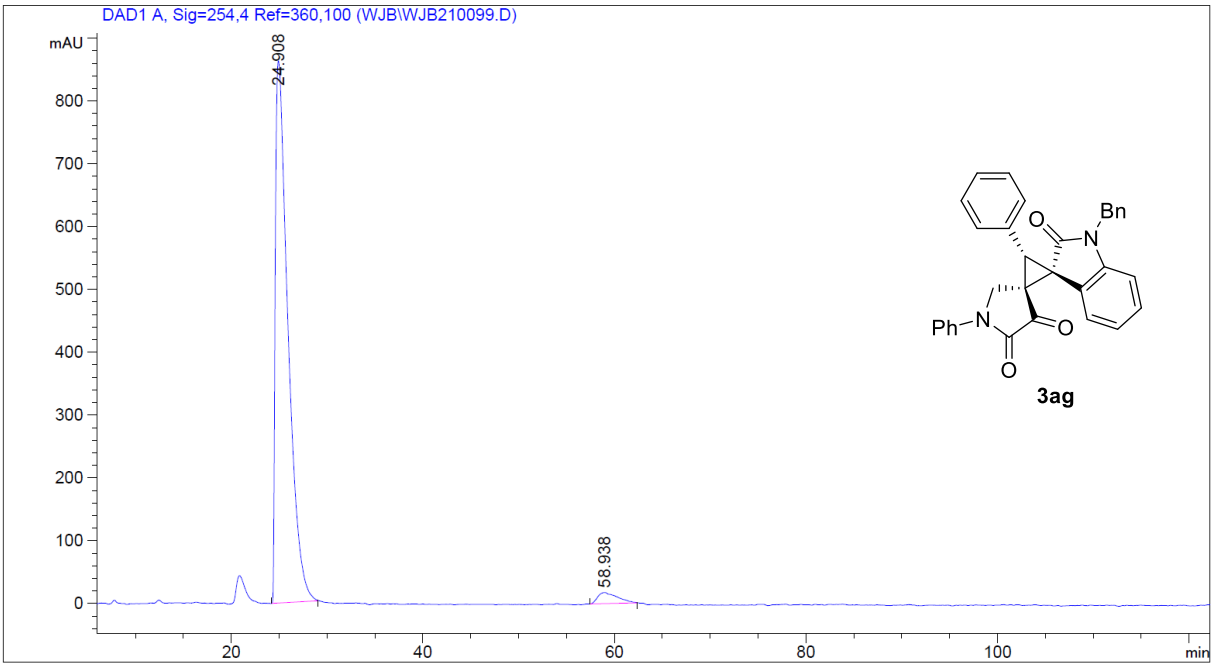
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	10.411	0.4115	86.20573	3.49185	1.3677
2	11.928	0.5234	3026.30713	96.36909	48.0129
3	14.989	0.3834	148.43542	5.38812	2.3589
4	17.854	0.7204	3042.16089	70.38170	48.2644



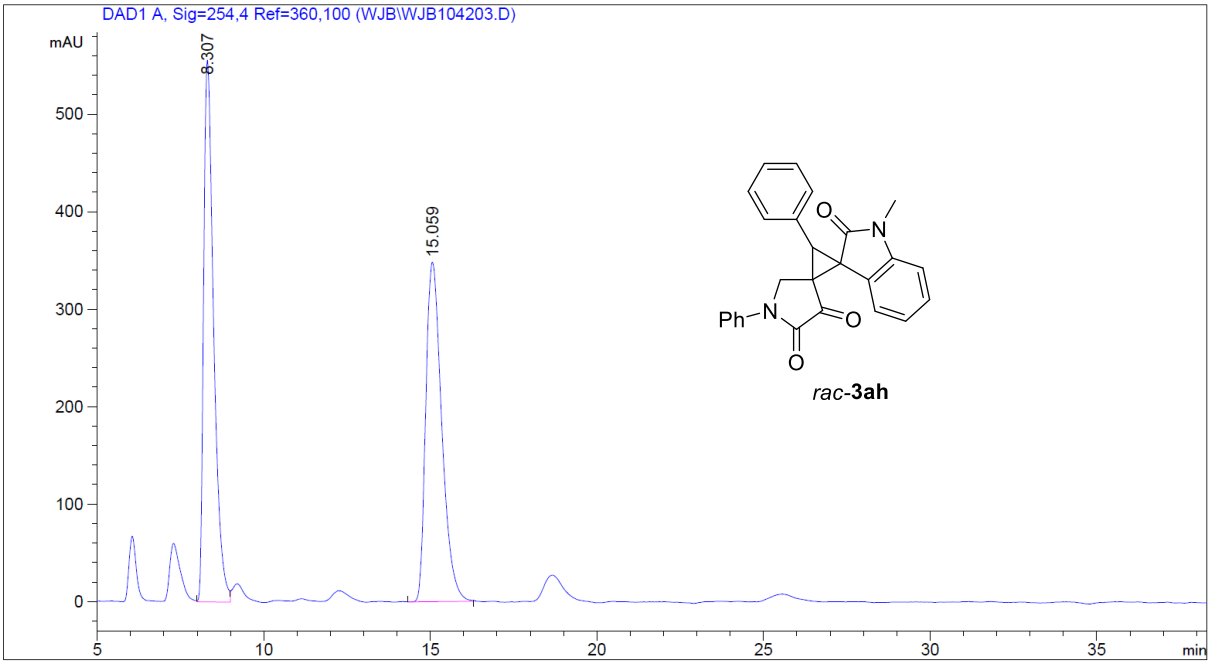
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	10.551	0.2364	28.59163	1.44783	0.5178
2	12.083	0.4573	1064.43164	33.62897	19.2756
3	14.971	0.3653	253.35223	10.14543	4.5879
4	18.010	0.6293	4175.80908	98.76486	75.6188



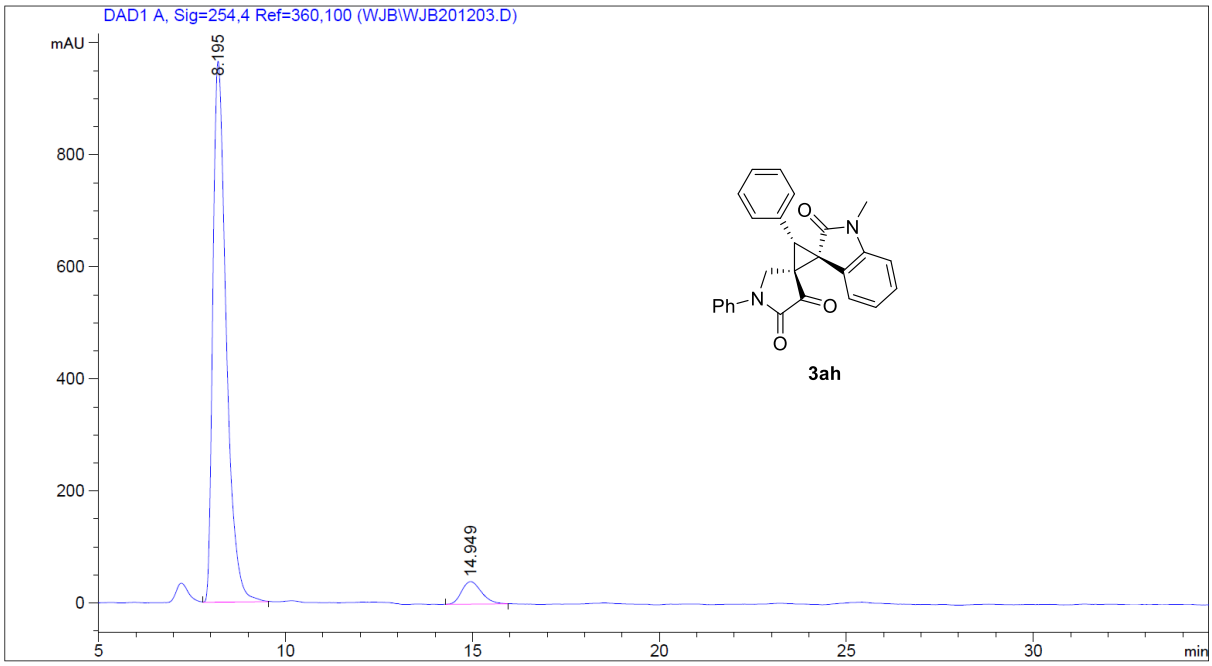
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	26.258	1.3028	8027.66162	102.69595	50.2375
2	59.762	2.9951	7951.76563	44.24814	49.7625



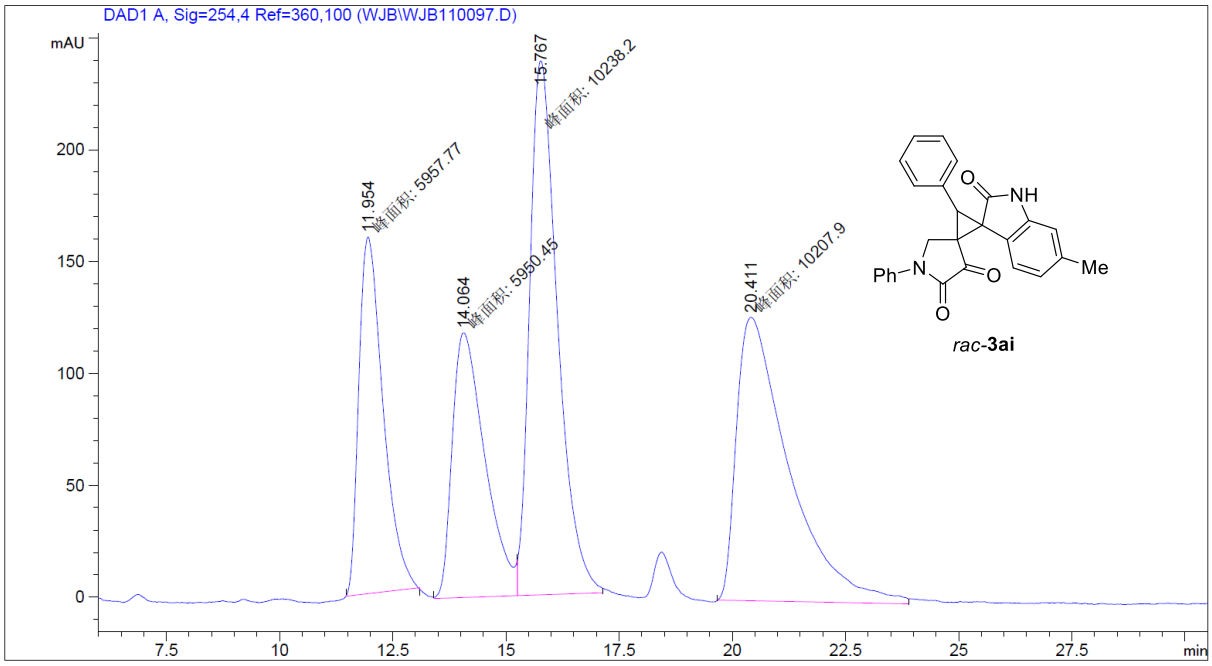
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	24.908	1.1926	7.90282e4	862.83954	96.9164
2	58.938	1.6617	2514.43677	17.73924	3.0836



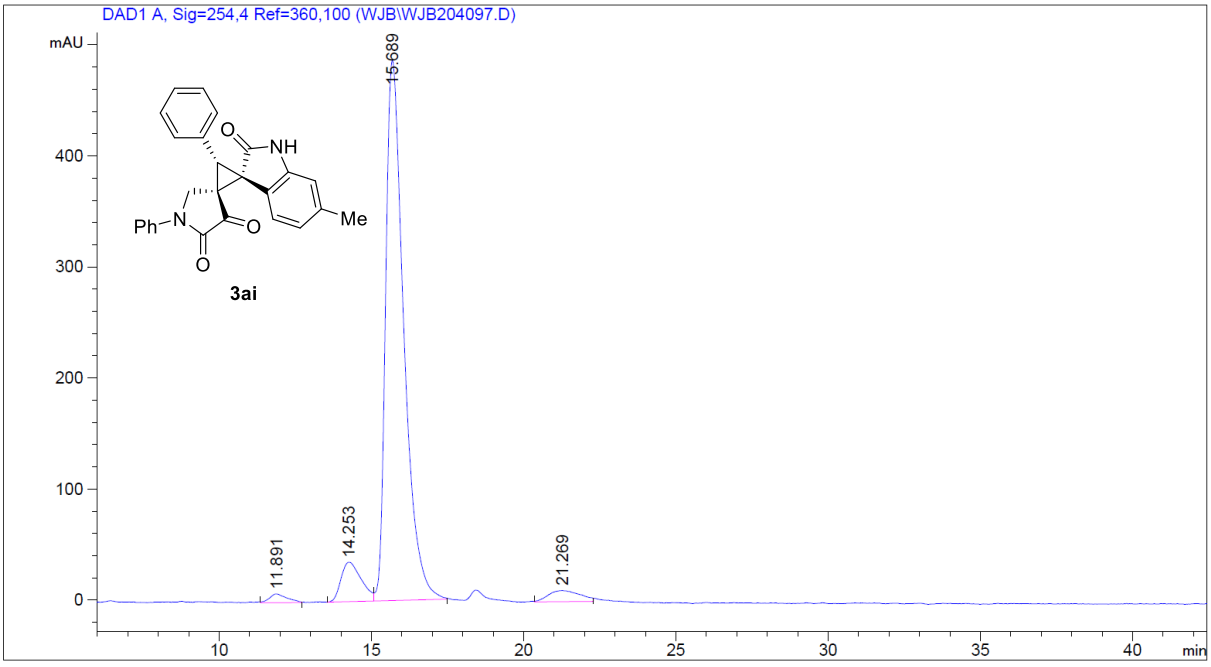
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.307	0.3212	1.16899e4	556.13867	50.1681
2	15.059	0.5188	1.16116e4	348.41922	49.8319



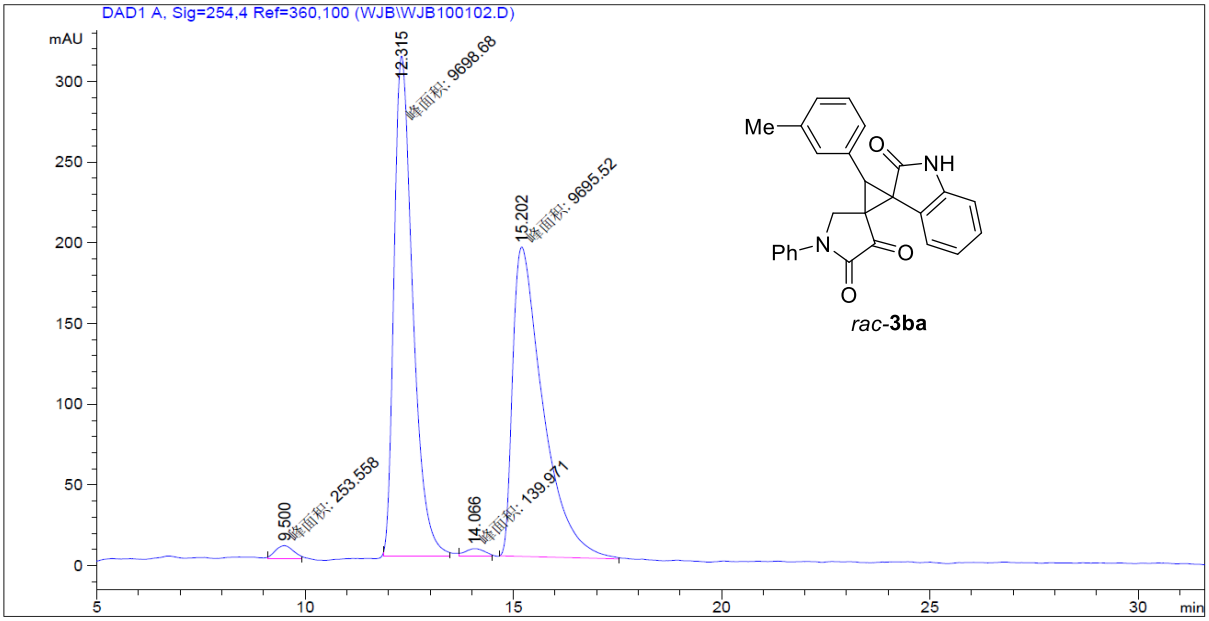
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.195	0.3773	2.39438e4	965.73016	94.1814
2	14.949	0.5146	1479.26074	40.08051	5.8186



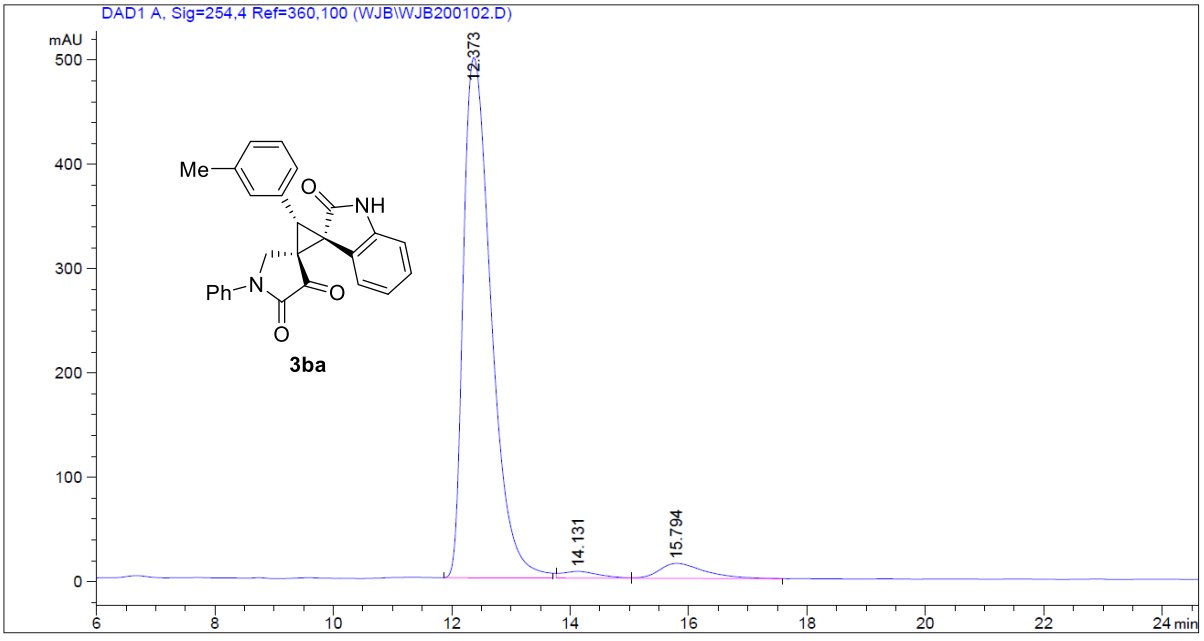
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.954	0.6226	5957.76953	159.48012	18.4141
2	14.064	0.8382	5950.44531	118.31105	18.3915
3	15.767	0.7146	1.02382e4	238.78651	31.6441
4	20.411	1.3420	1.02079e4	126.77451	31.5503



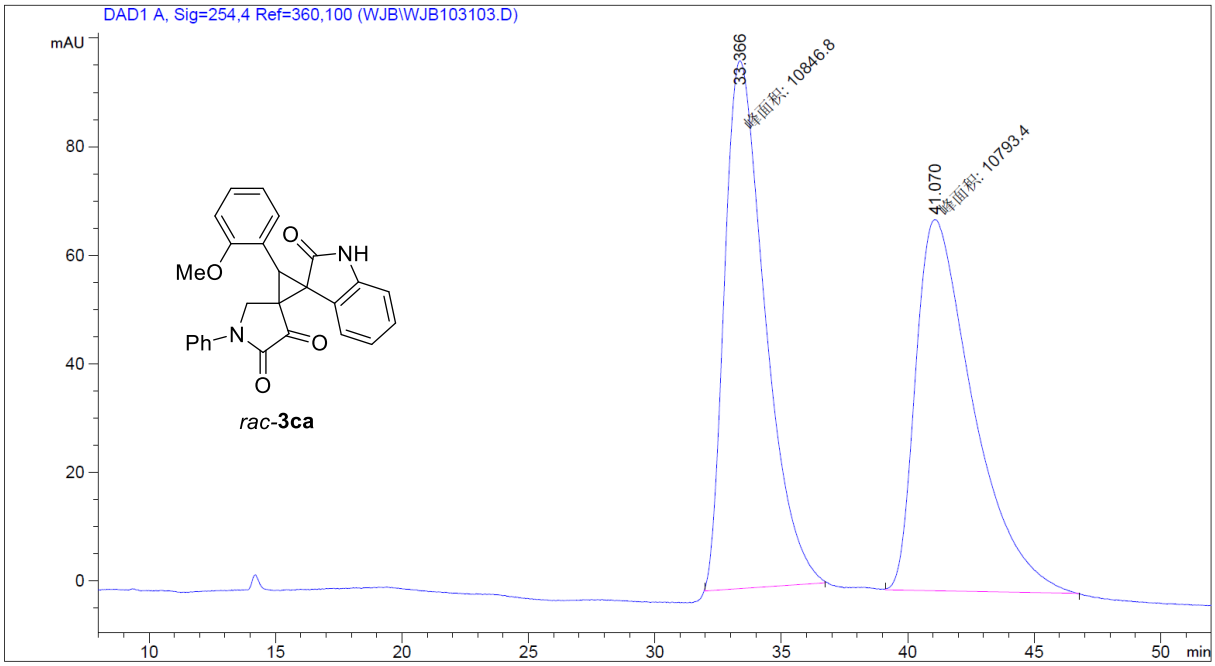
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.891	0.4779	310.71732	7.78130	1.3603
2	14.253	0.5871	1641.55920	35.52750	7.1865
3	15.689	0.5856	2.01665e4	486.46790	88.2860
4	21.269	0.8431	723.47046	10.08322	3.1673



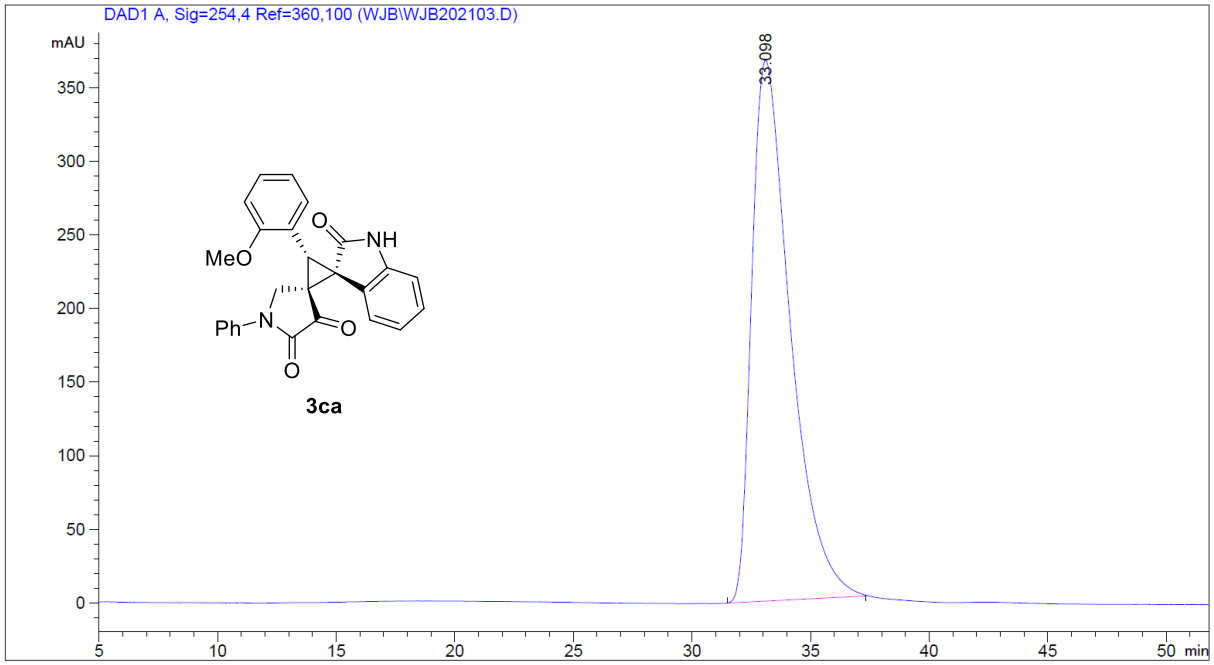
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	9.500	0.5111	253.55803	8.26890	1.2814
2	12.315	0.5223	9698.67578	309.50089	49.0136
3	14.066	0.5268	139.97141	4.42847	0.7074
4	15.202	0.8434	9695.52051	191.59204	48.9976



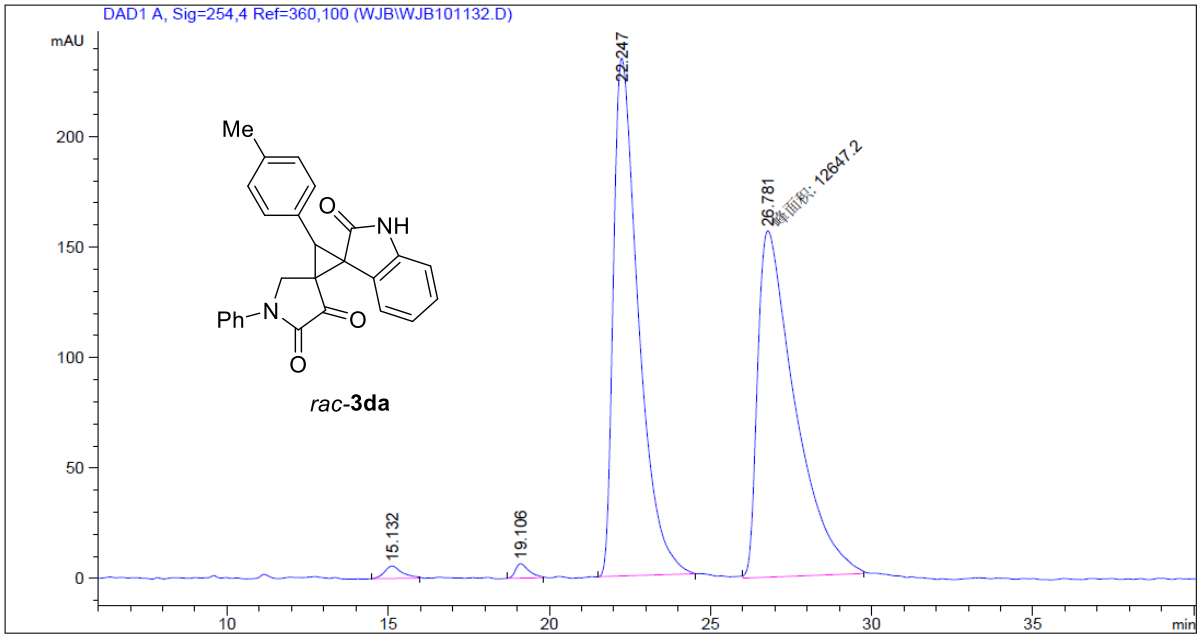
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	12.373	0.5067	1.62701e4	498.53784	93.9008
2	14.131	0.5168	282.63165	6.53153	1.6312
3	15.794	0.6582	774.17444	14.377723	4.4680



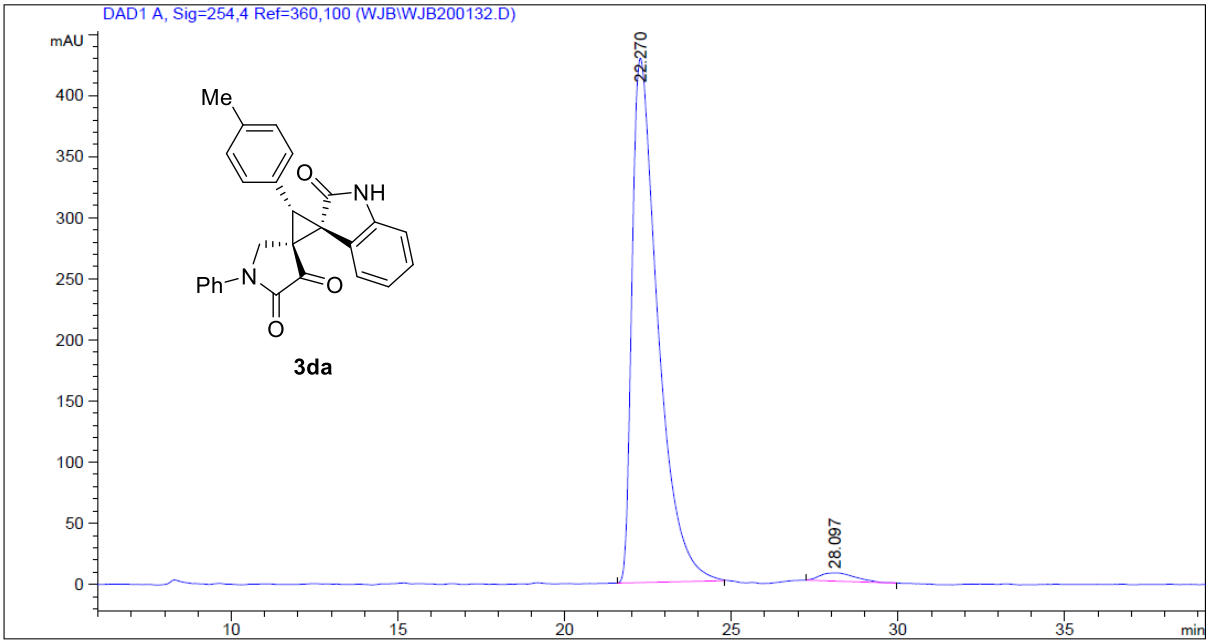
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	33.366	1.8584	1.08468e4	97.27657	50.1233
2	41.070	2.6320	1.07934e4	68.34662	49.8767



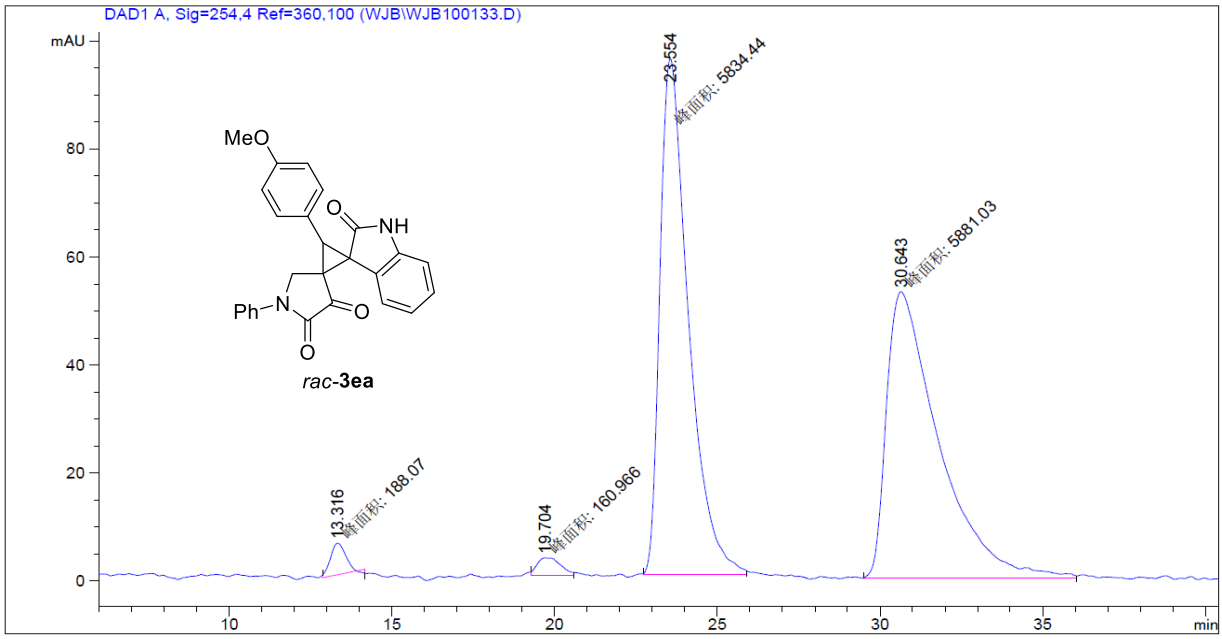
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	33.098	1.6031	4.15932e4	367.31366	100.0000



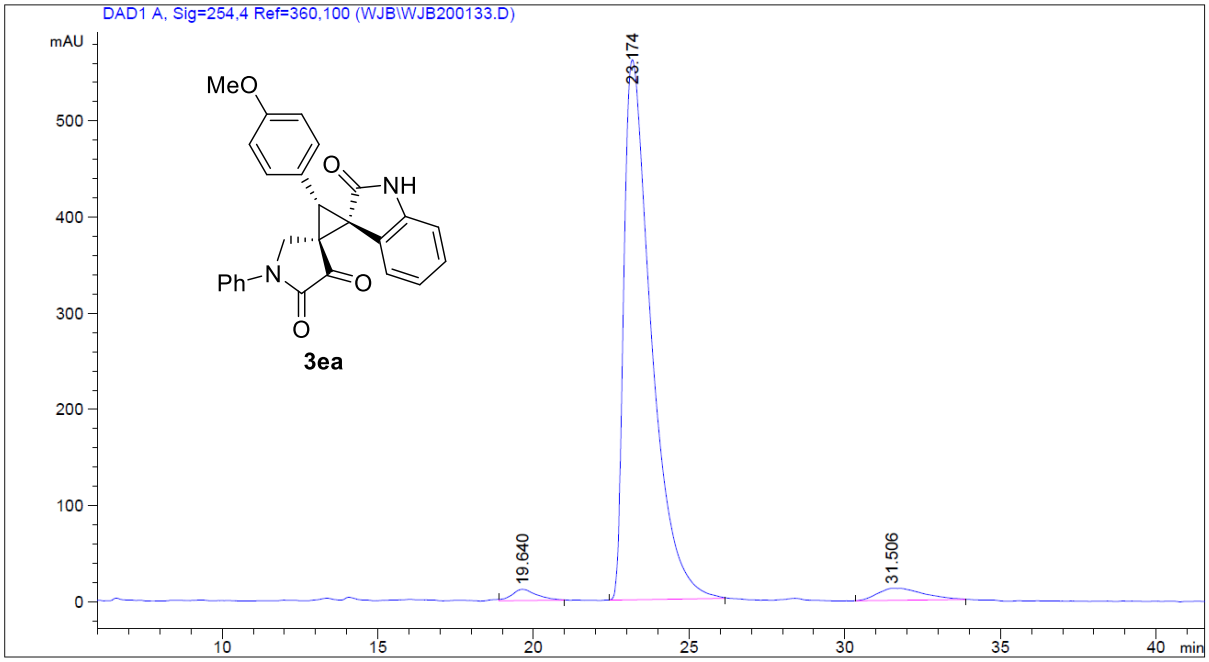
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	15.132	0.4623	214.45787	5.58109	0.8340
2	19.106	0.4137	190.01068	6.52613	0.7390
3	22.247	0.7961	1.26617e4	234.42819	49.2416
4	26.781	1.3450	1.26472e4	156.71603	49.1854



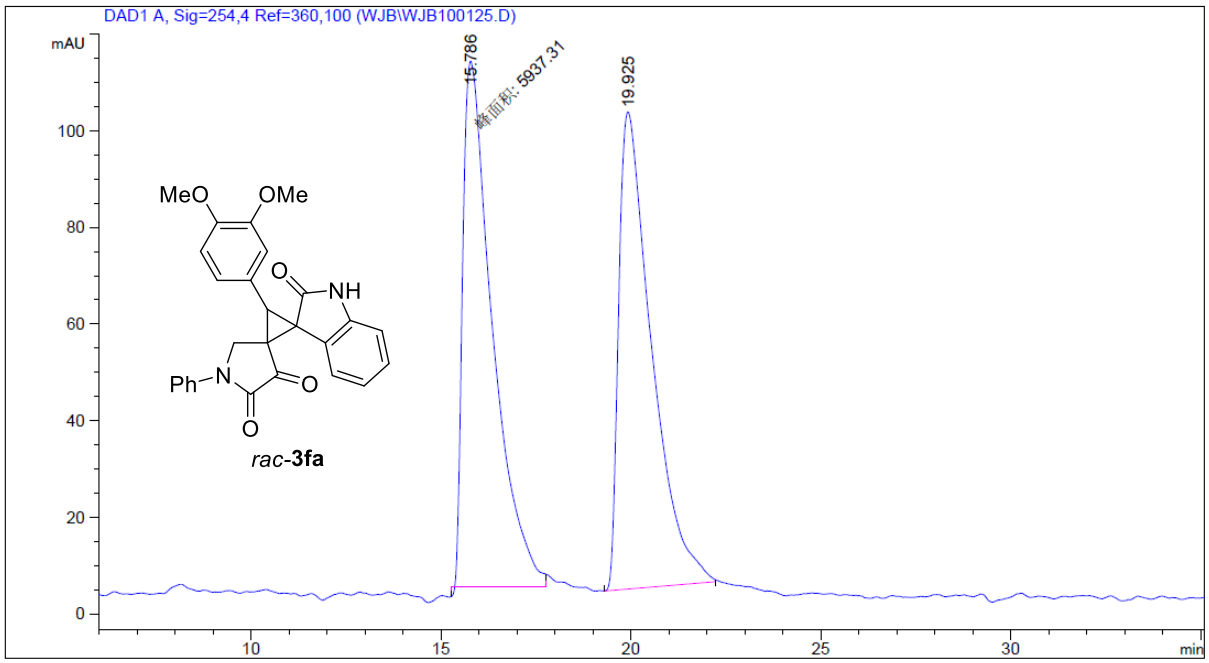
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	22.270	0.7969	2.31223e4	428.95096	97.9014
2	28.097	0.8698	495.65039	6.74117	2.0986



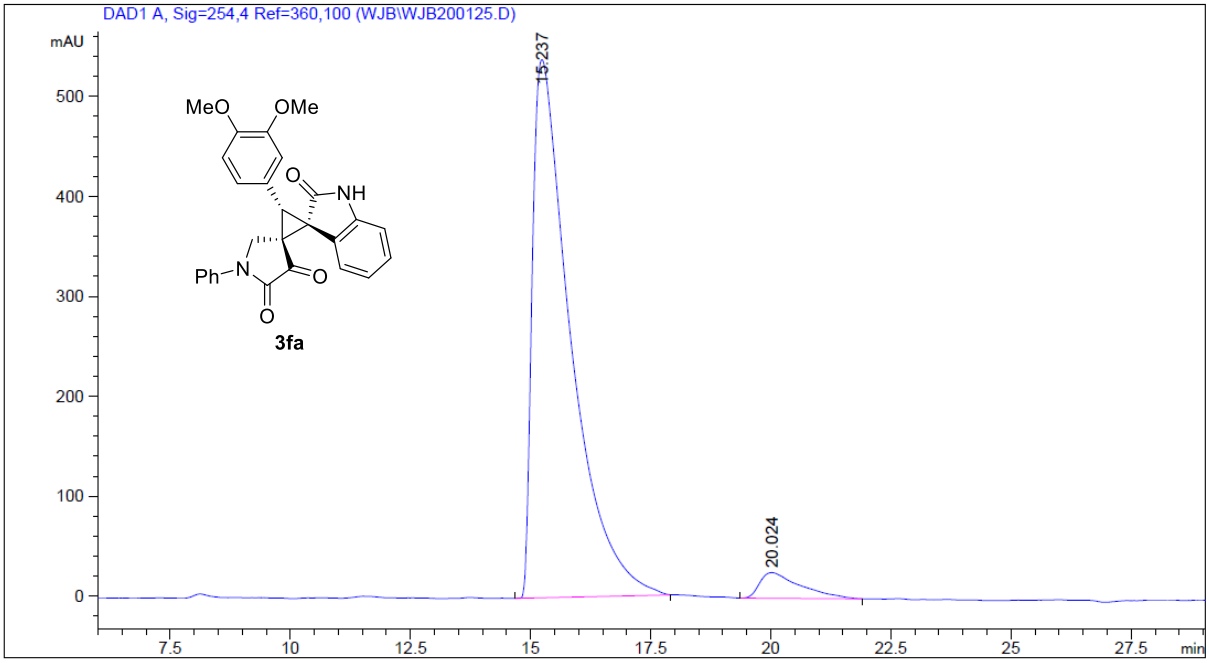
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.316	0.5345	188.06975	5.86430	1.5589
2	19.704	0.8329	160.96613	3.22095	1.3342
3	23.554	1.0176	5834.44287	95.56062	48.3604
4	30.643	1.8505	5881.03320	52.96864	48.7465



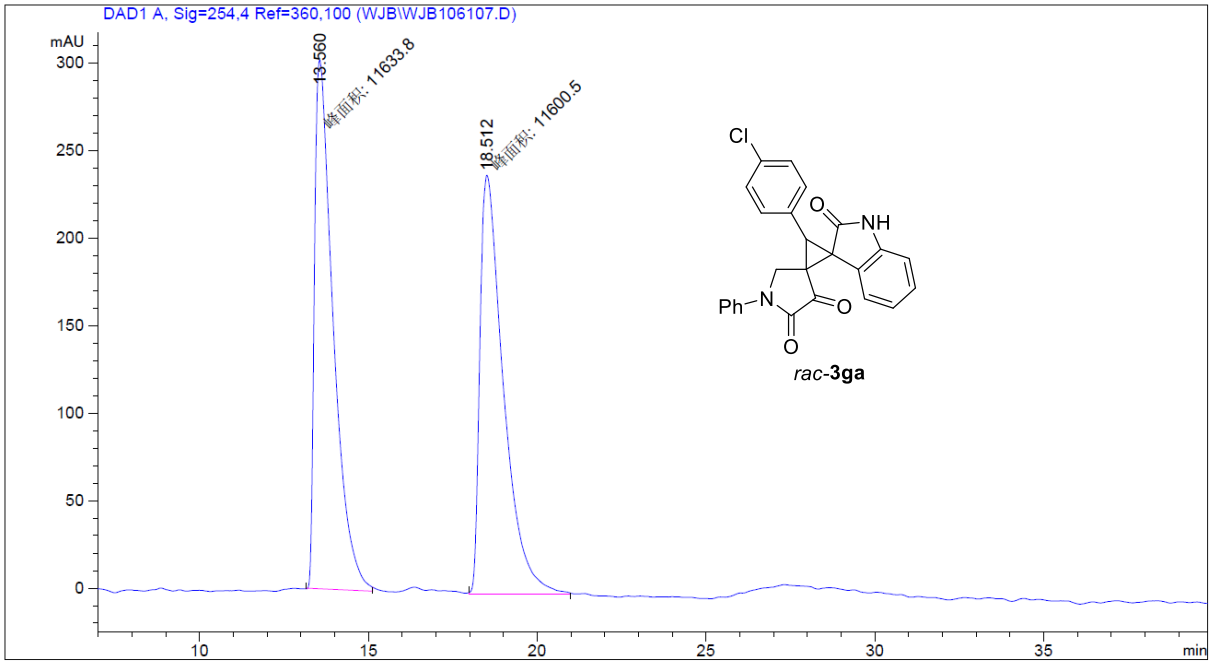
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	19.640	0.6703	651.05811	11.75318	1.7965
2	23.174	0.8948	3.43175e4	561.65112	94.6931
3	31.506	1.2001	1272.21533	12.51369	3.5105



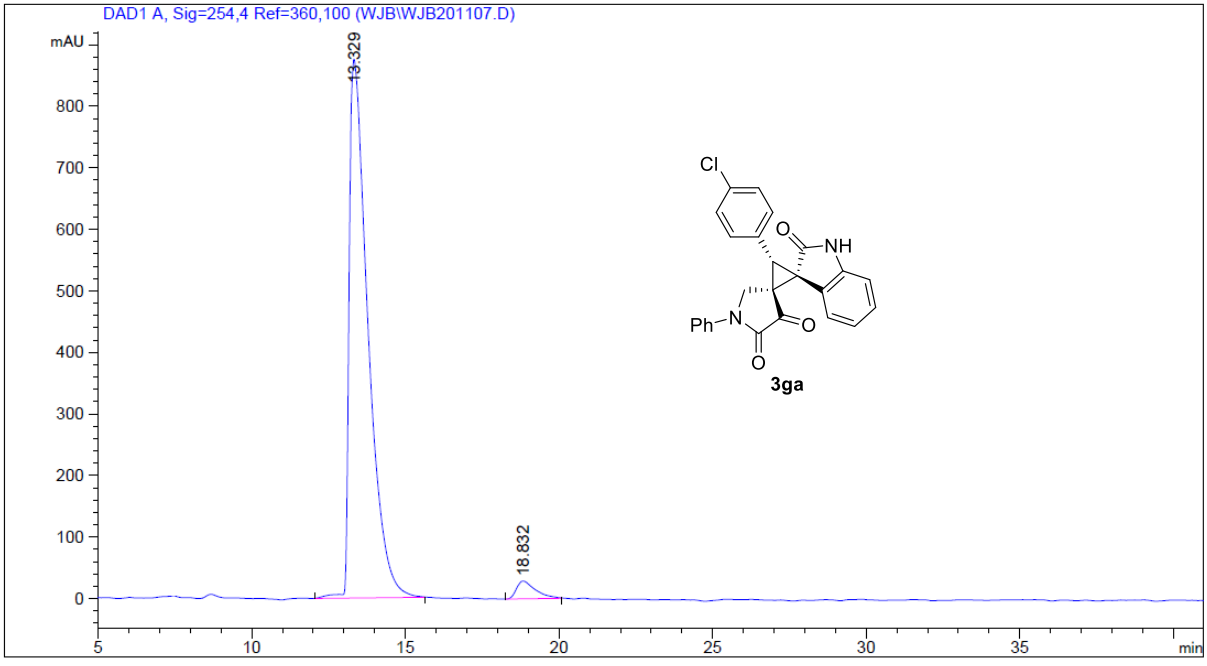
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	15.786	0.9084	5937.30908	108.92759	49.9670
2	19.925	0.8182	5945.16016	98.77988	50.0330



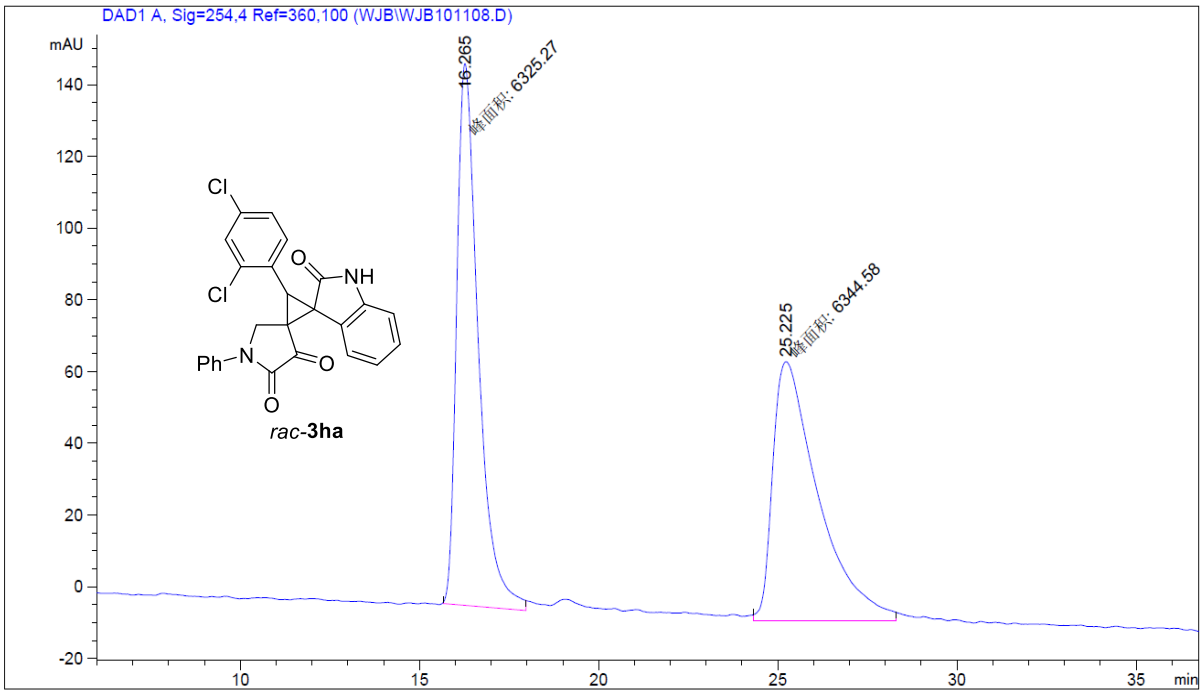
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	15.237	0.7710	2.97728e4	538.52228	95.1430
2	20.024	0.7750	1519.88843	25.73763	4.8570



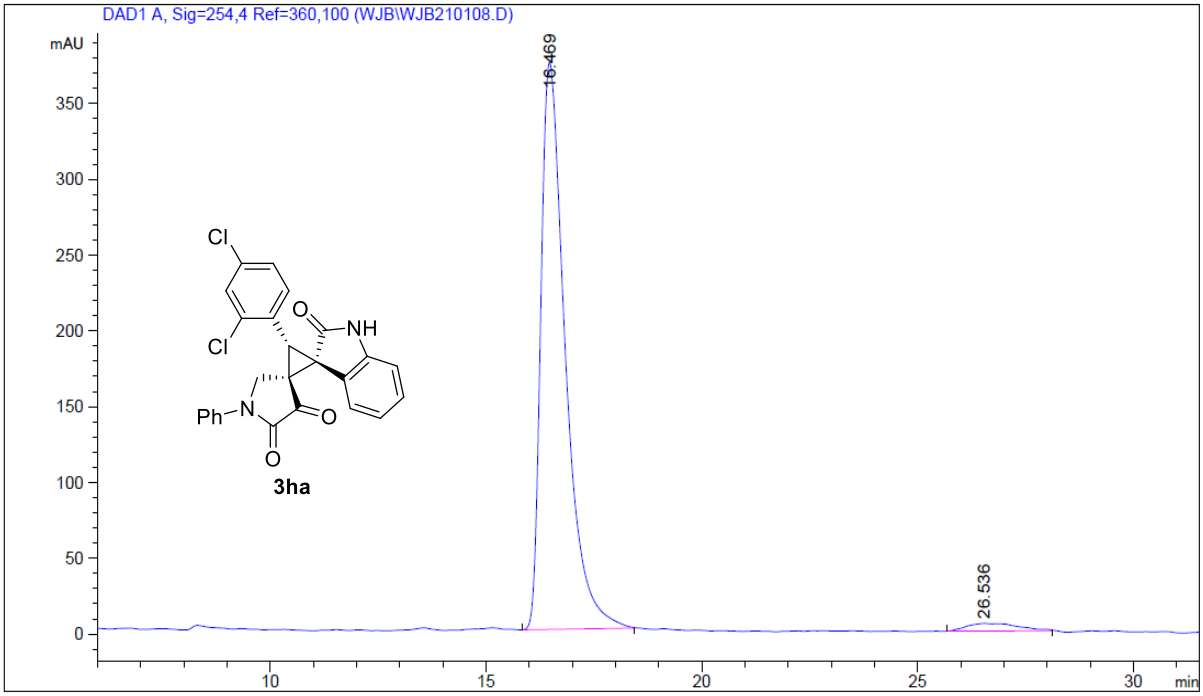
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.560	0.6422	1.16338e4	301.93530	50.0715
2	18.512	0.8083	1.16005e4	239.20134	49.9285



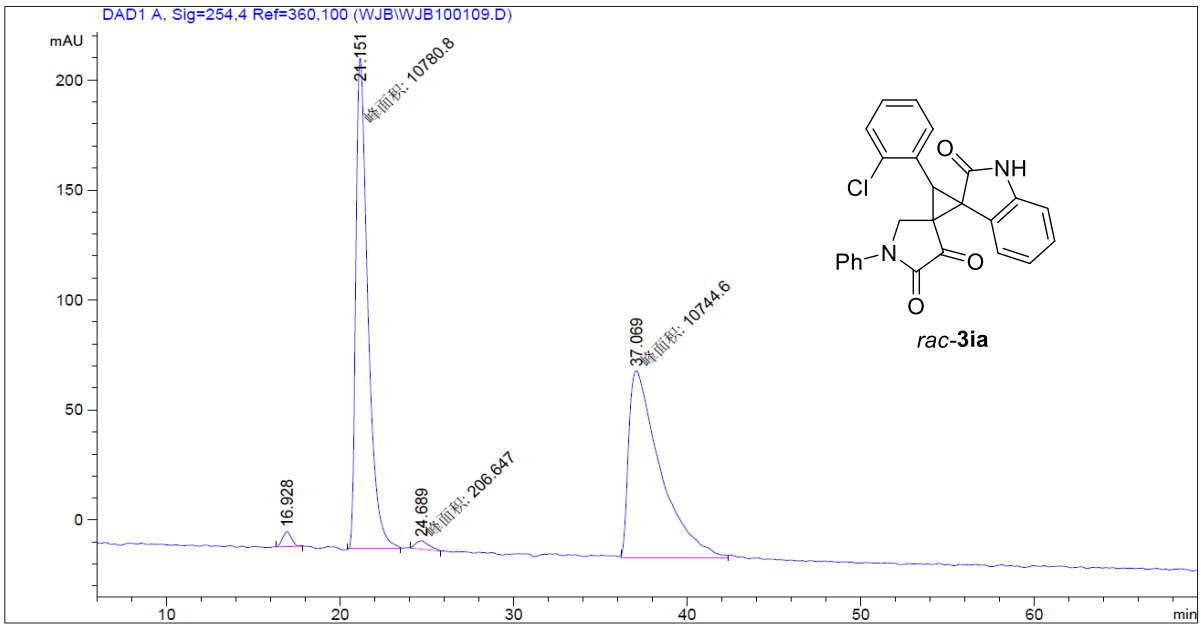
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.329	0.6247	3.71313e4	875.75018	96.7577
2	18.832	0.5983	1244.25024	28.90086	3.2423



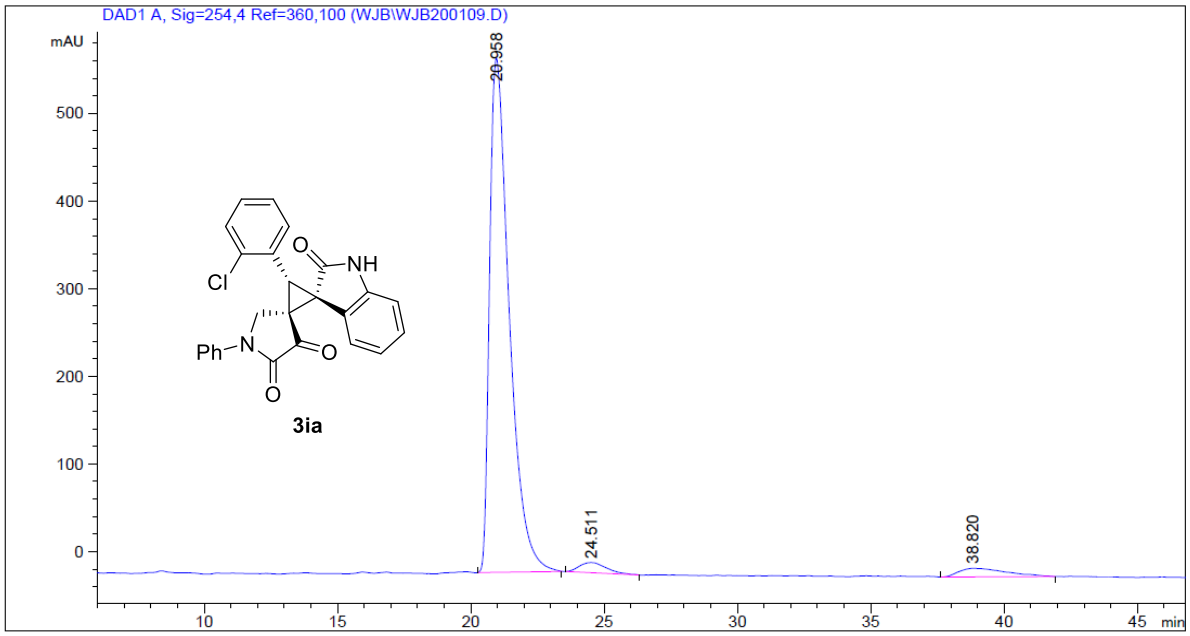
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	16.265	0.6978	6325.27393	151.07901	49.9238
2	25.225	1.4669	6344.57910	72.08625	50.0762



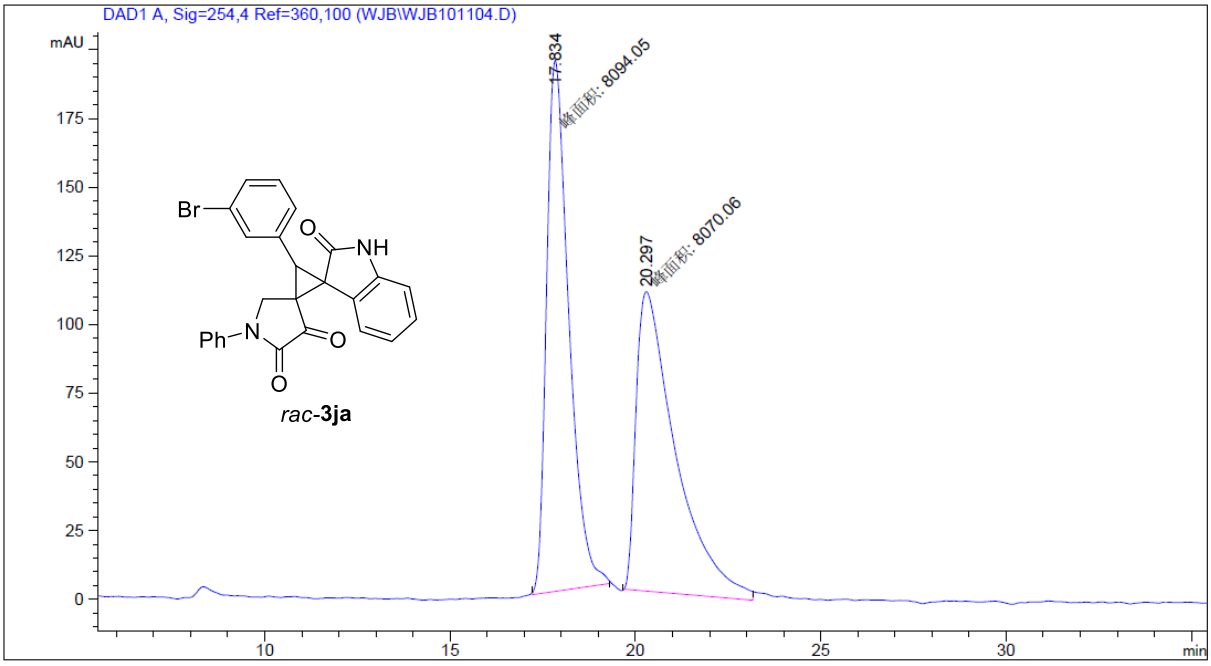
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	16.469	0.5907	1.47036e4	374.01151	97.2248
2	26.536	0.9699	419.69995	5.10112	2.7752



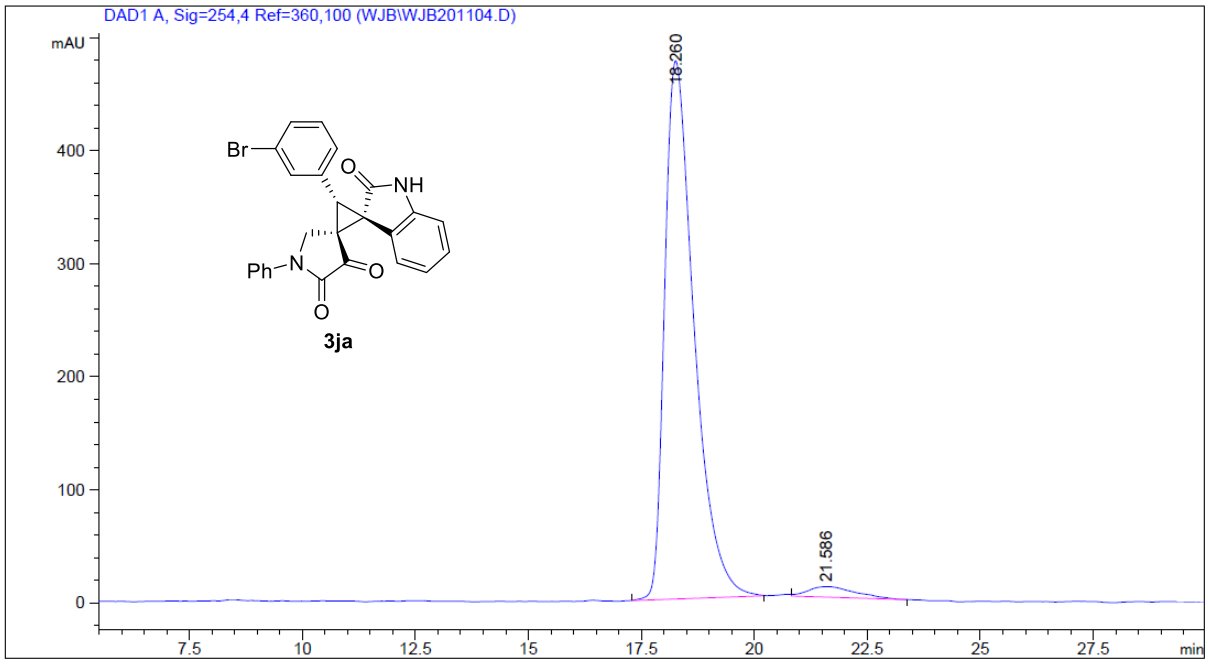
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	16.928	0.4534	240.29655	6.71694	1.0936
2	21.151	0.8060	1.107808e4	222.92033	49.0654
3	24.689	0.8976	206.64734	3.83692	0.9405
4	37.069	2.1034	1.07446e4	85.13586	48.9004



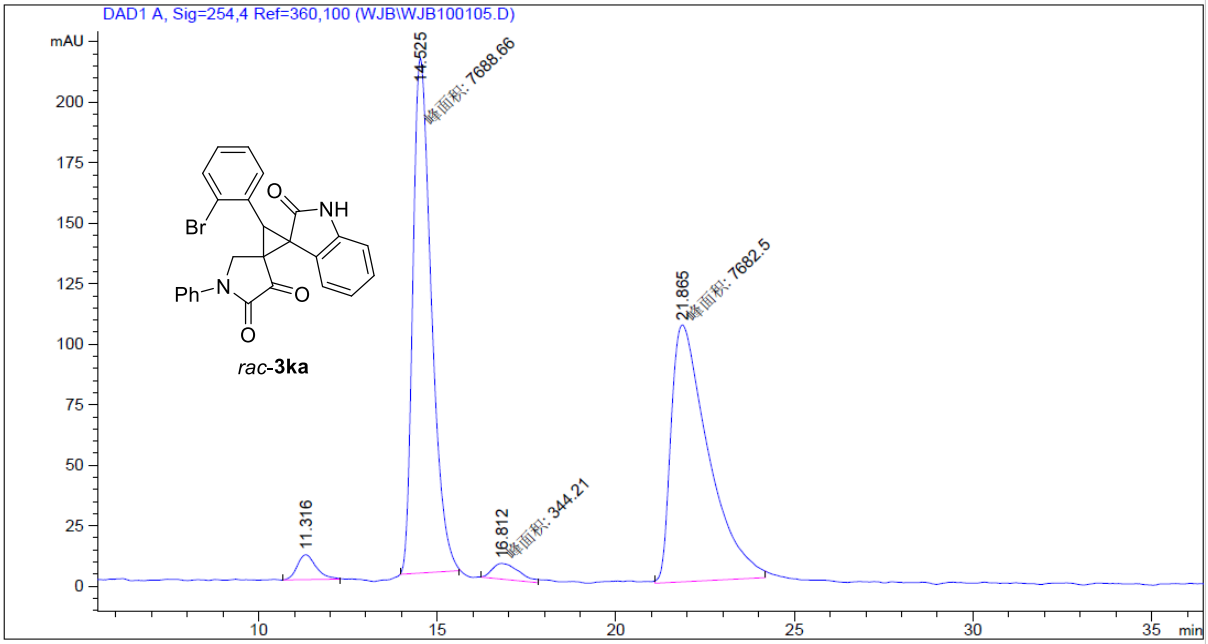
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	20.958	0.7740	3.00591e4	586.47632	93.6700
2	24.511	0.8239	810.36383	11.64394	2.5252
3	38.820	1.4477	1220.95837	9.96314	3.8047



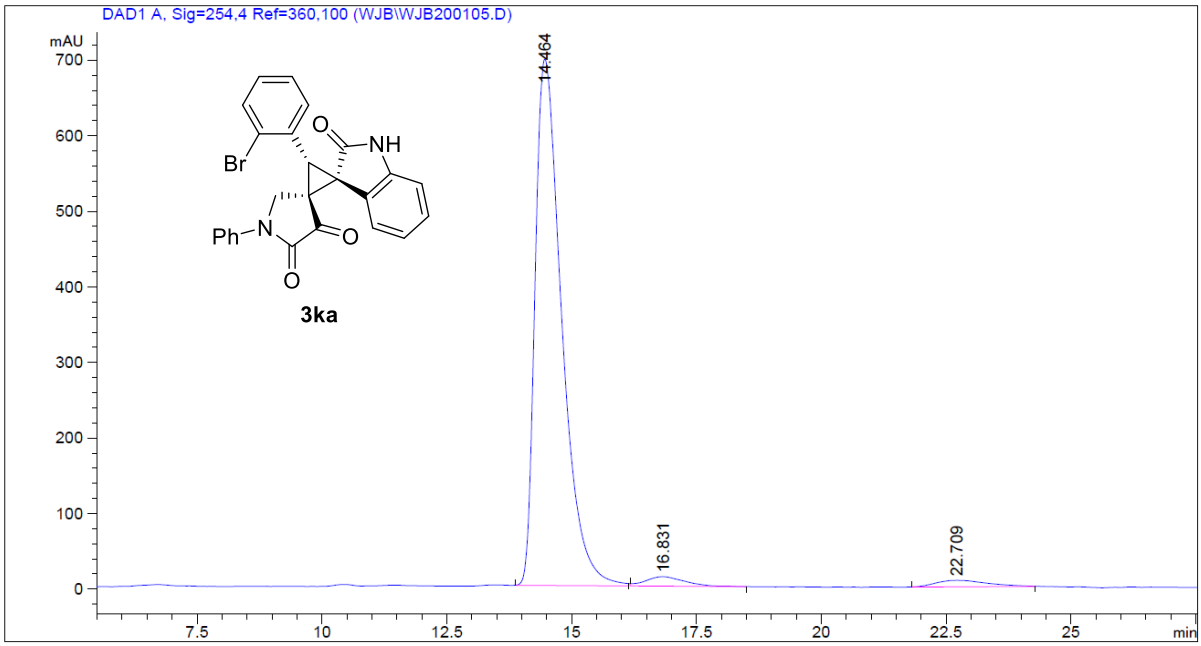
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	17.834	0.6977	8094.05078	193.36087	50.0742
2	20.297	1.2337	8070.05615	109.02421	49.9258



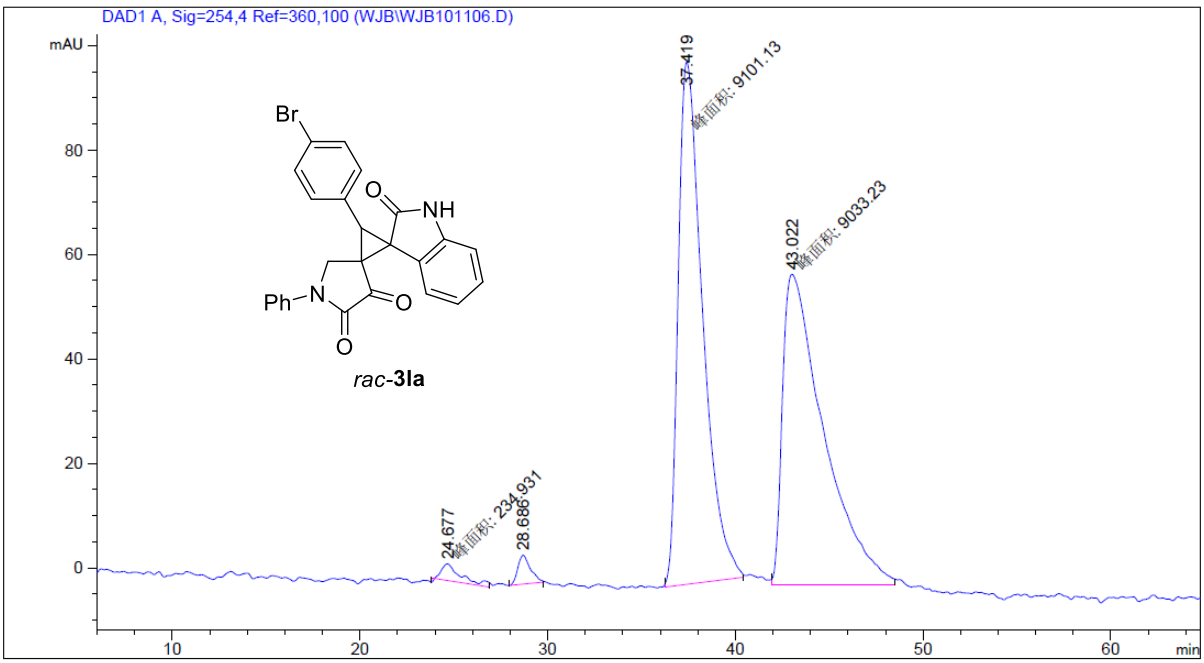
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	18.260	0.6816	2.15397e4	476.33276	96.9515
2	21.586	0.8566	677.27881	9.33306	3.0485



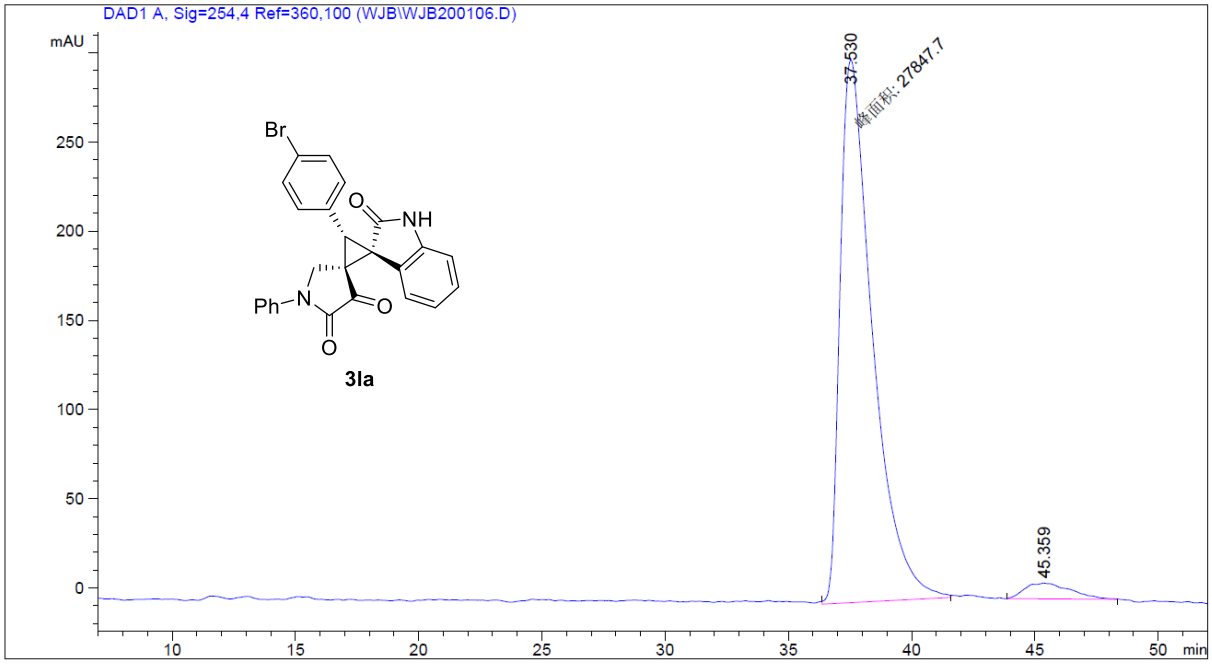
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.316	0.4469	378.20505	10.28721	2.3500
2	14.525	0.6026	7688.66357	212.66362	47.7747
3	16.812	0.8756	344.20959	6.55153	2.1388
4	21.865	1.2061	7682.50195	106.16322	47.7364



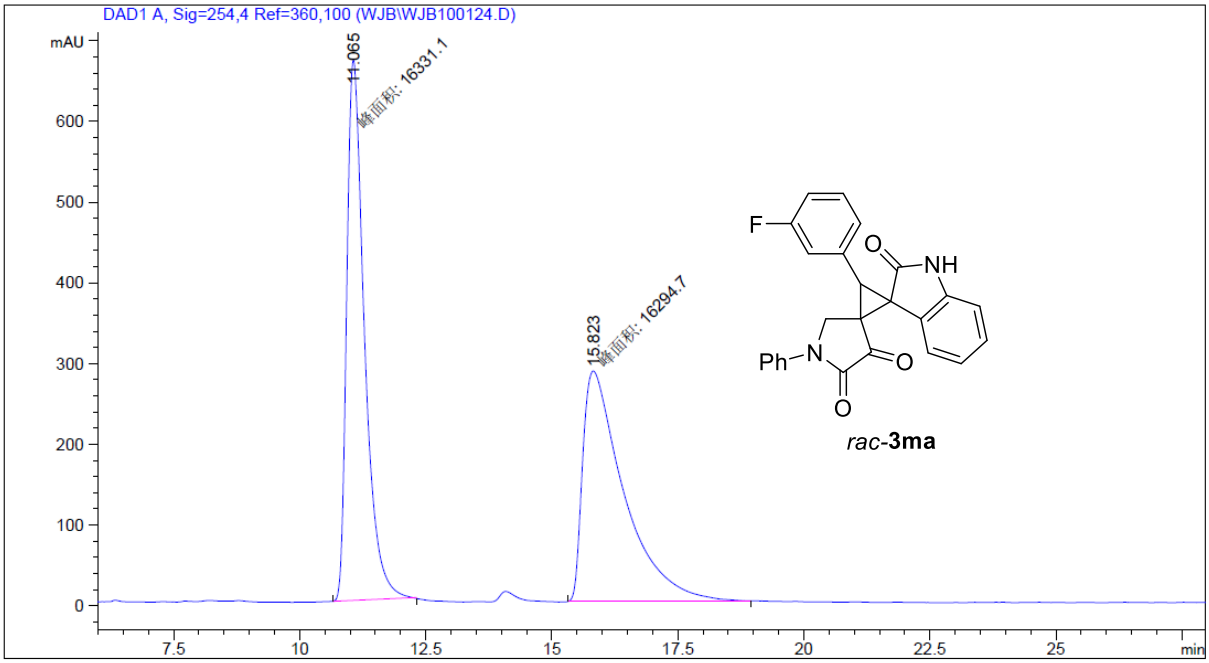
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	14.464	0.5616	2.57650e4	696.50659	95.1855
2	16.831	0.6734	688.44202	12.64106	2.5434
3	22.709	0.8509	614.74603	8.52860	2.2711



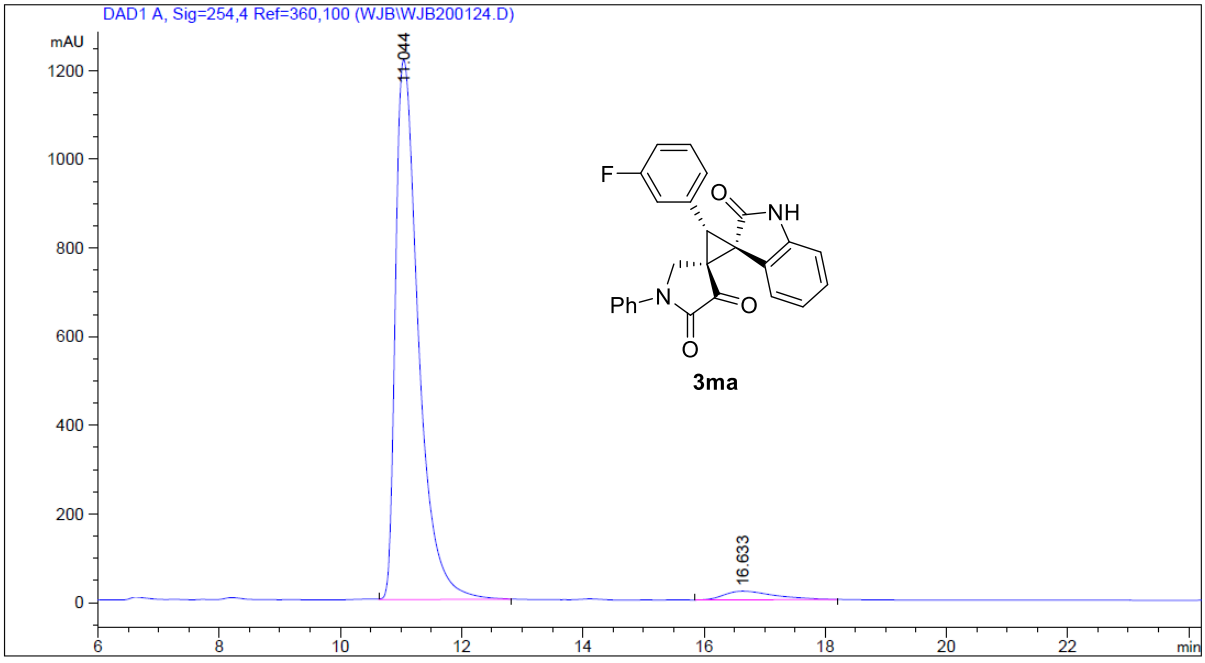
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	24.677	1.2245	234.93140	3.19771	1.2611
2	28.686	0.5568	259.44336	5.57646	1.3927
3	37.419	1.5157	9101.13086	100.07813	48.8553
4	43.022	2.5326	9033.22852	59.44736	48.4908



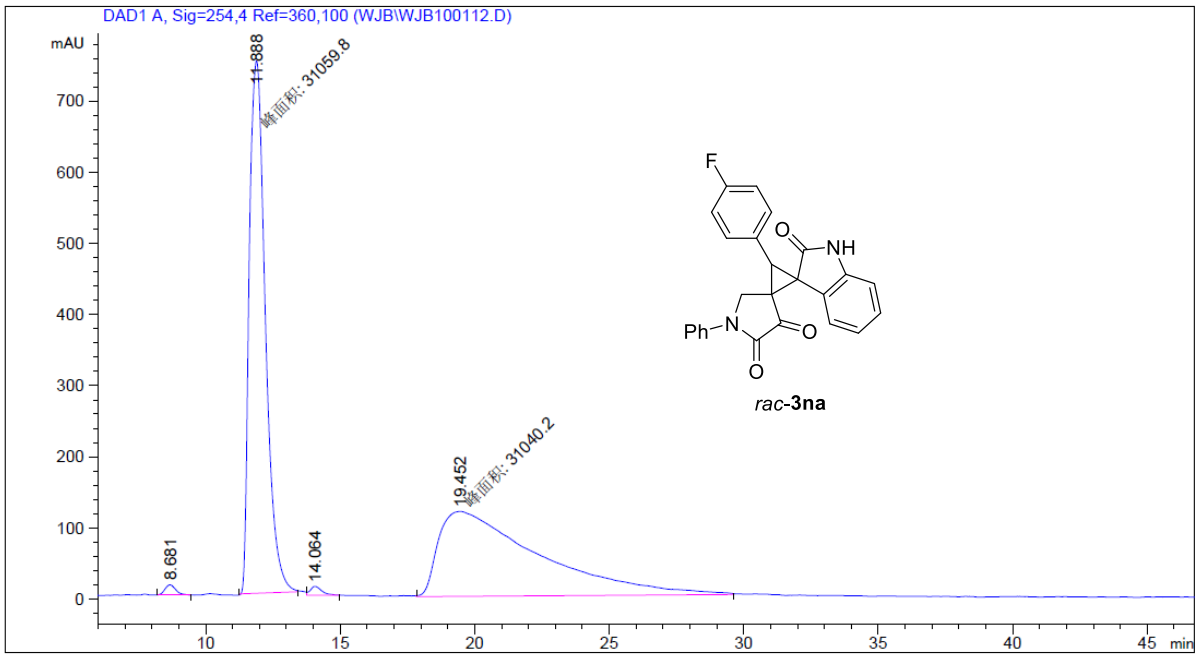
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	37.530	1.5257	2.78477e4	304.20969	96.0642
2	45.359	1.5299	1140.92664	8.78059	3.9358



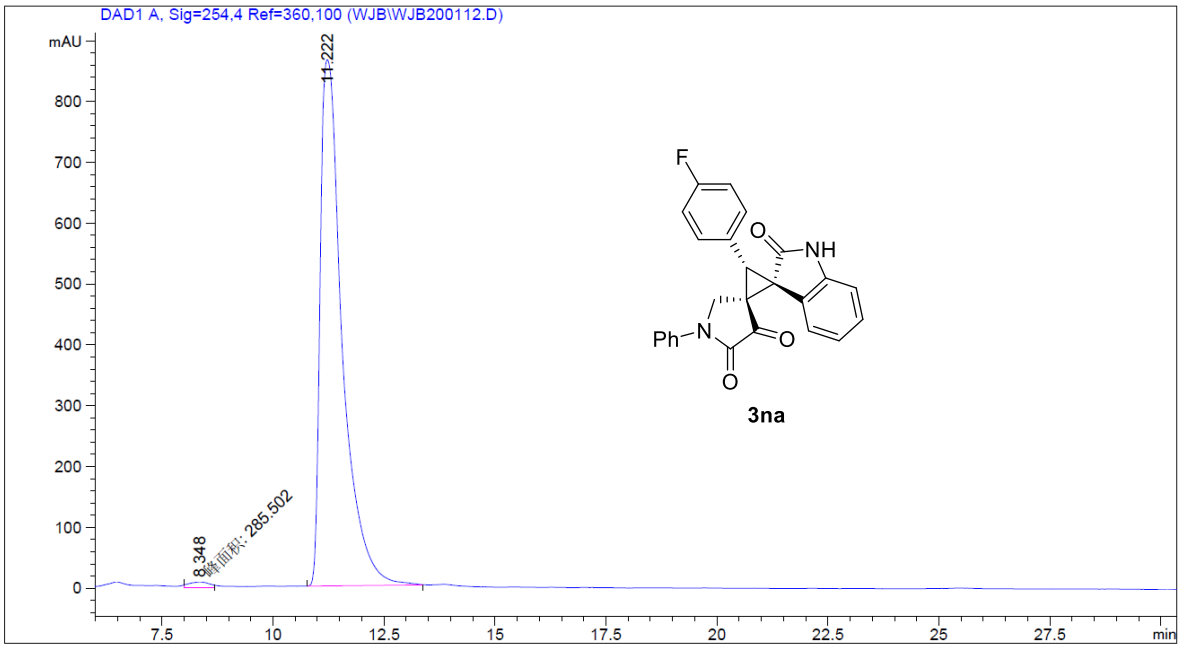
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.065	0.4064	1.63311e4	669.75769	50.0558
2	15.823	0.9505	1.62947e4	285.70963	49.9442



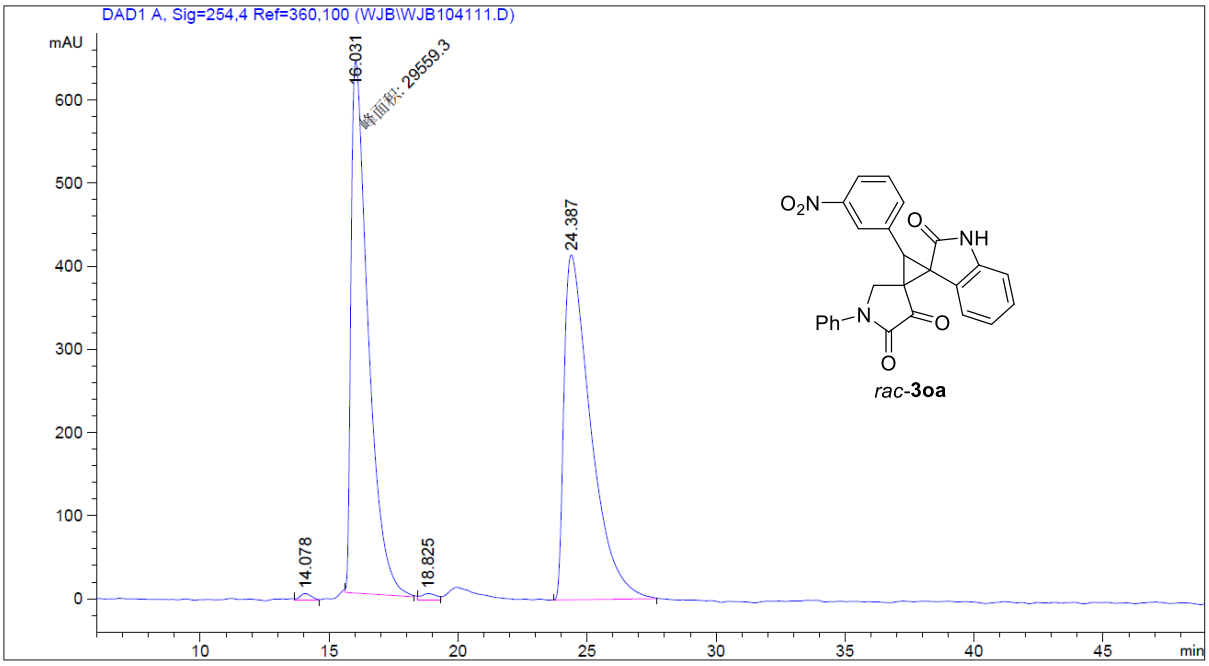
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	11.044	0.3993	3.20773e4	1217.95740	96.6204
2	16.633	0.7453	1121.99243	19.44107	3.3796



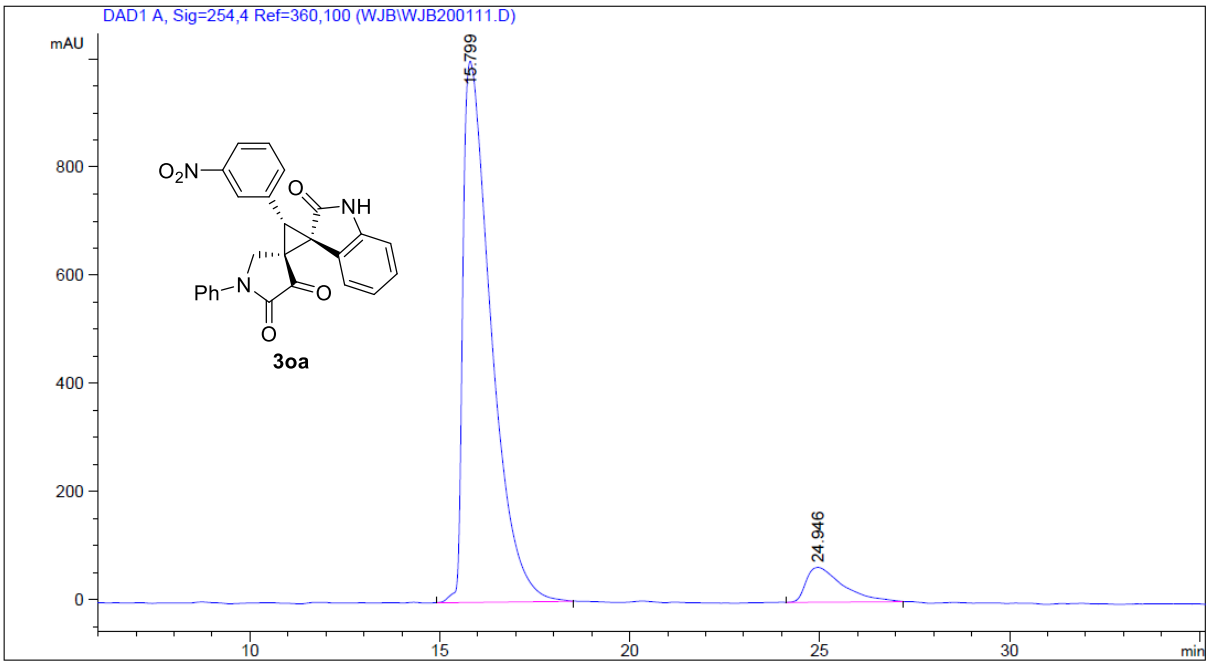
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.681	0.4055	405.31360	14.52948	0.6446
2	11.888	0.6916	3.10598e4	748.53577	49.3979
3	14.064	0.4053	371.46979	12.47728	0.5908
4	19.452	4.3283	3.10402e4	119.52375	49.3667



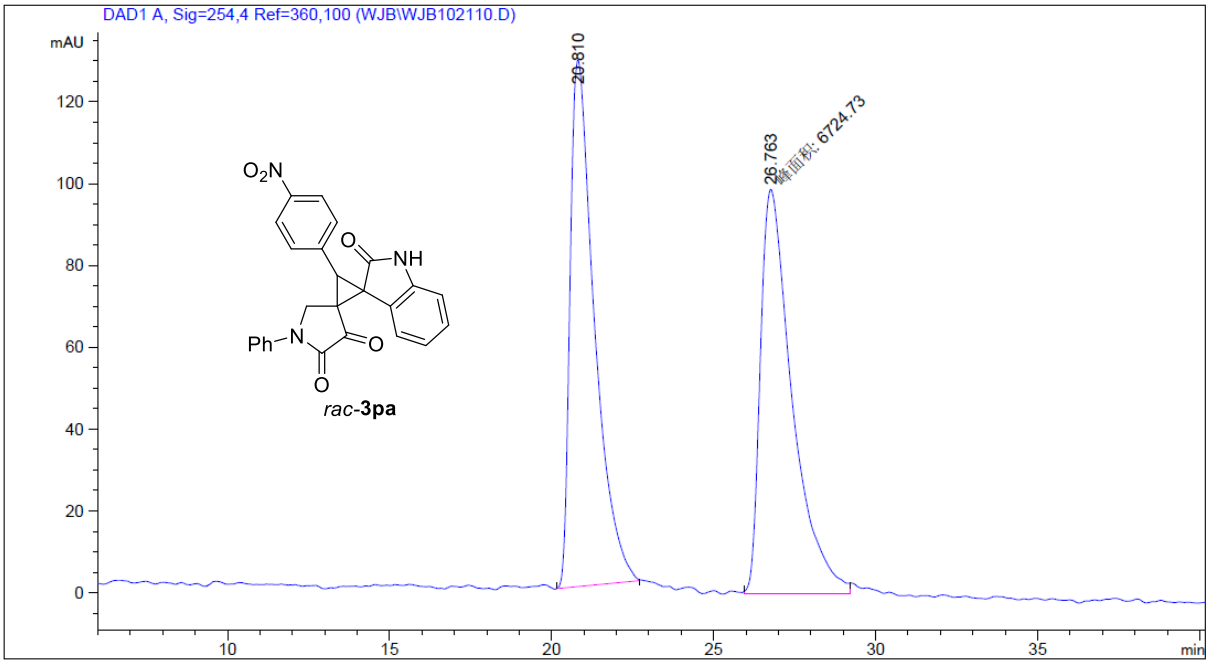
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.348	0.5298	285.50159	8.98092	0.9487
2	11.222	0.5181	2.98097e4	864.88324	99.0513



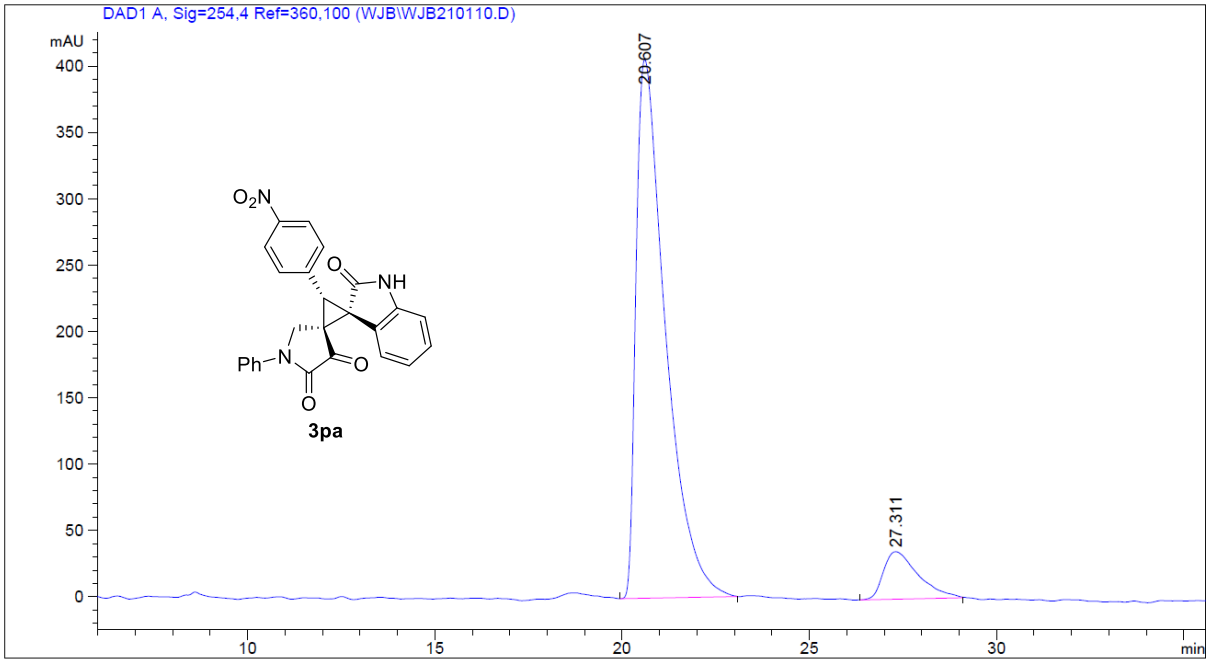
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	14.078	0.4271	261.86365	8.27660	0.4364
2	16.031	0.7695	2.95593e4	640.23151	49.2573
3	18.825	0.4999	326.87766	7.78484	0.5447
4	24.387	0.9980	2.98619e4	414.72940	49.7616



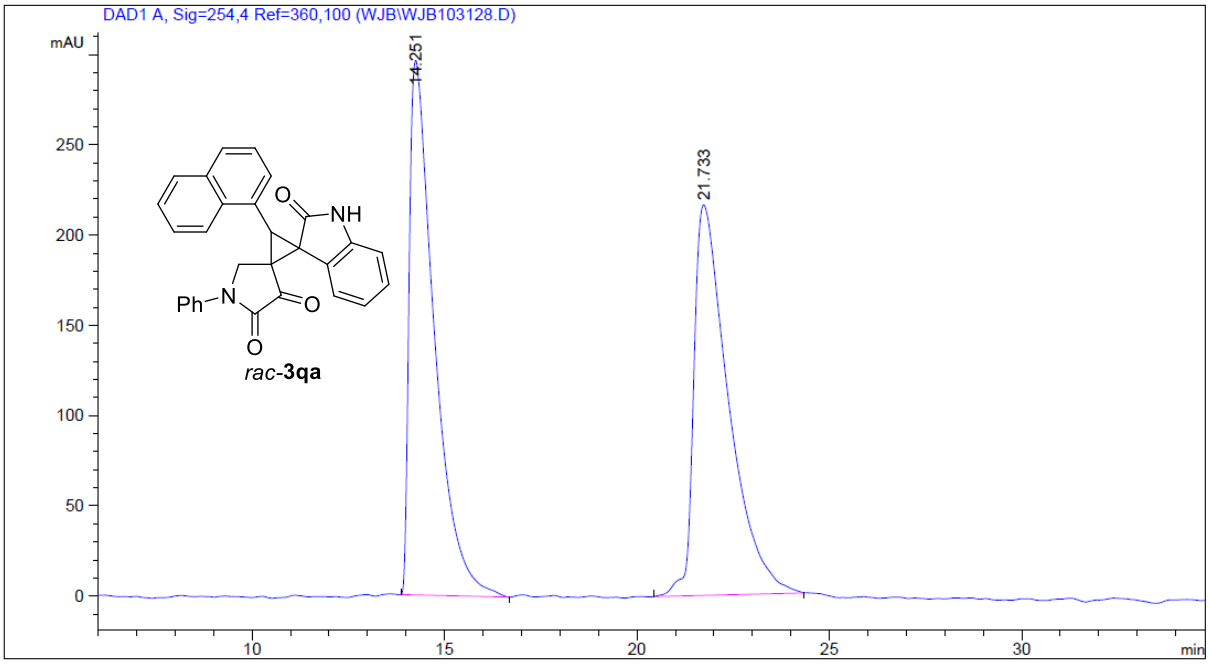
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	15.799	0.7501	5.16541e4	1000.81061	92.2739
2	24.946	0.9359	4325.02783	64.36931	7.7261



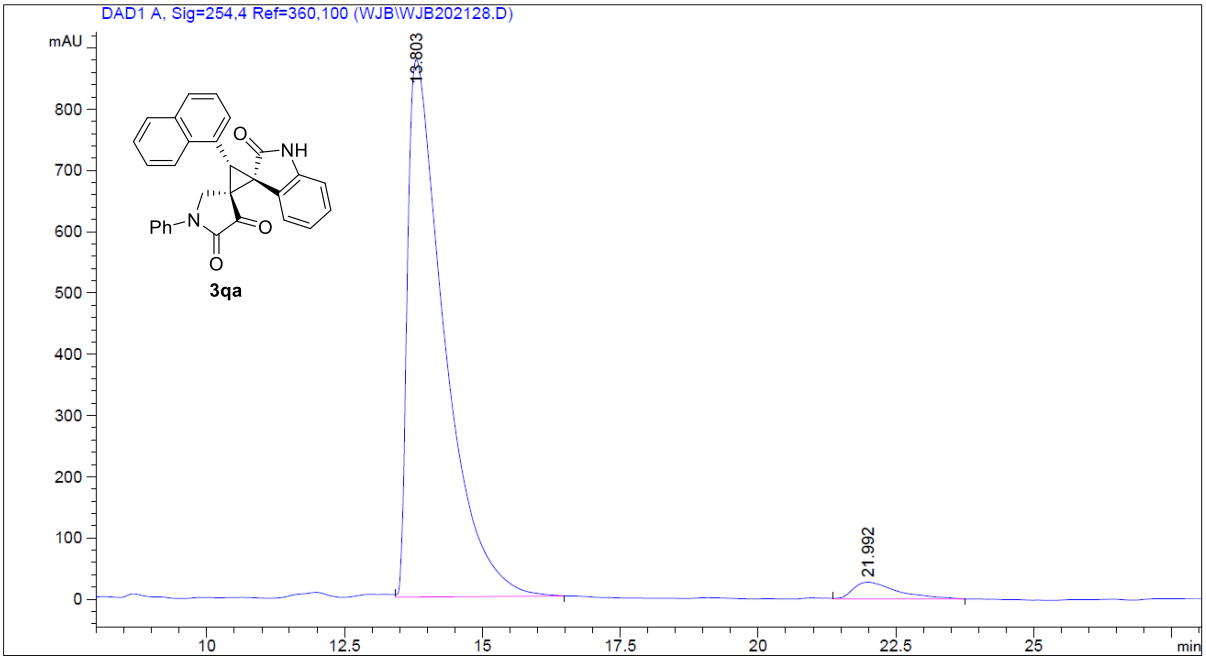
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	20.810	0.7465	6713.29492	128.72269	49.9575
2	26.763	1.1345	6724.72607	98.78780	50.0425



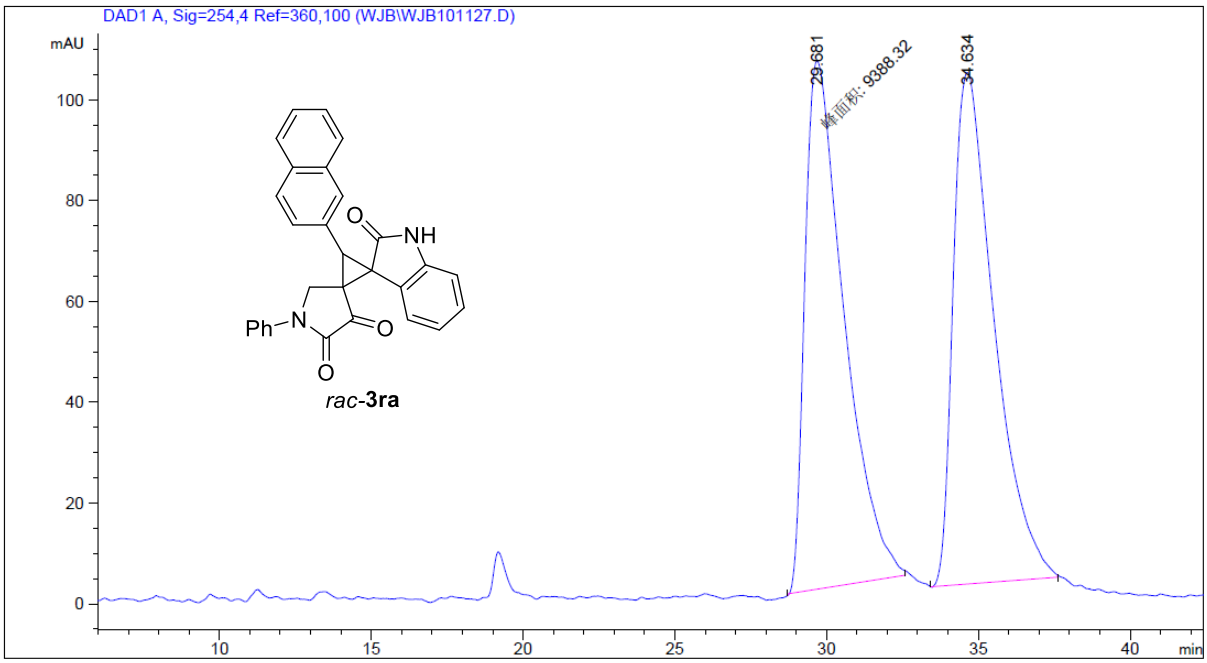
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	20.607	0.7973	2.22445e4	405.97086	90.5807
2	27.311	0.8911	2313.14893	35.83347	9.4193



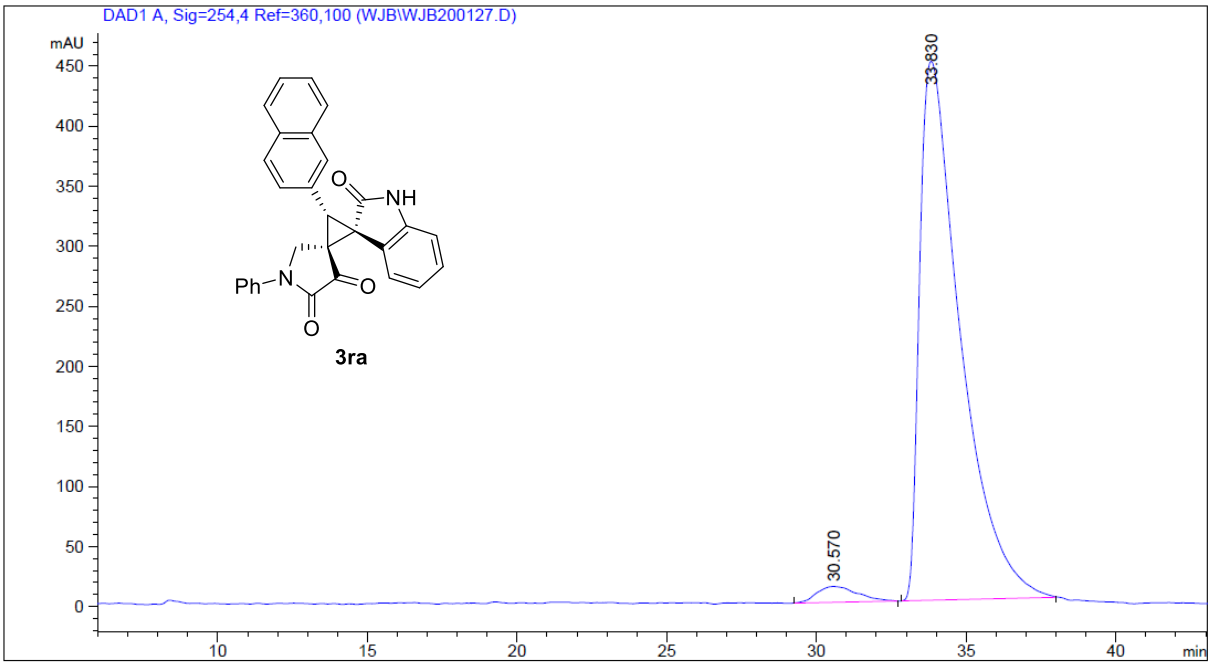
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	14.251	0.6567	1.34216e4	296.19766	49.7656
2	21.733	0.8865	1.35481e4	216.38805	50.2344



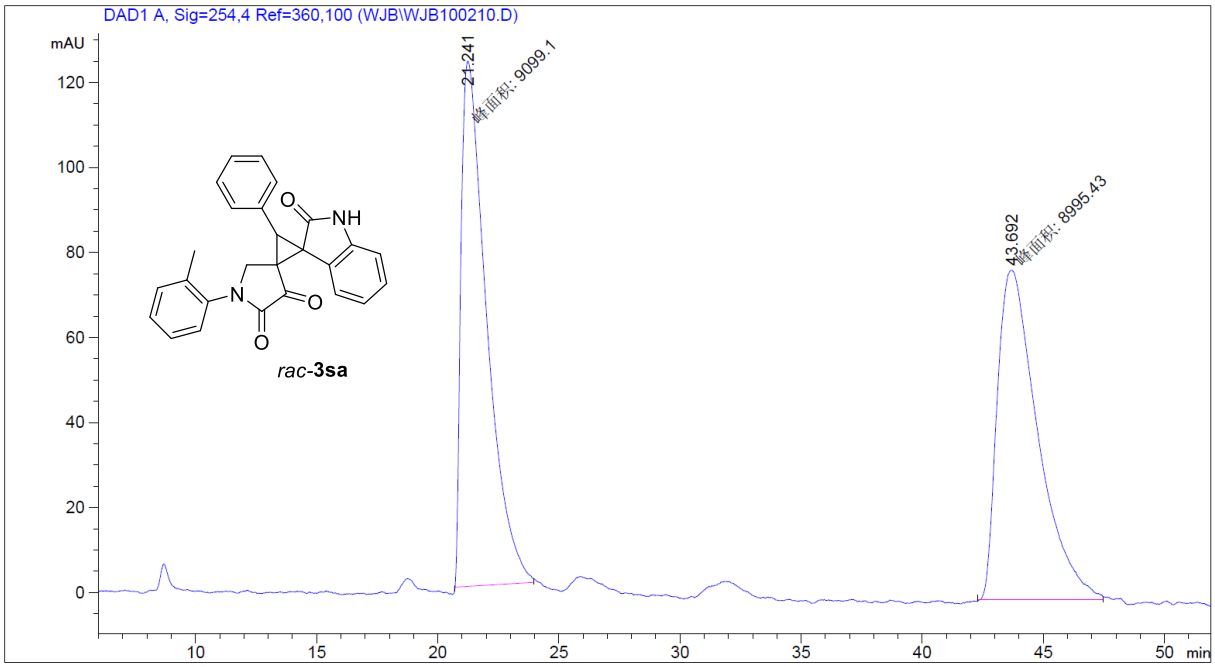
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.803	0.6595	4.05892e4	877.67865	96.3428
2	21.992	0.7535	1540.76660	27.33979	3.6572



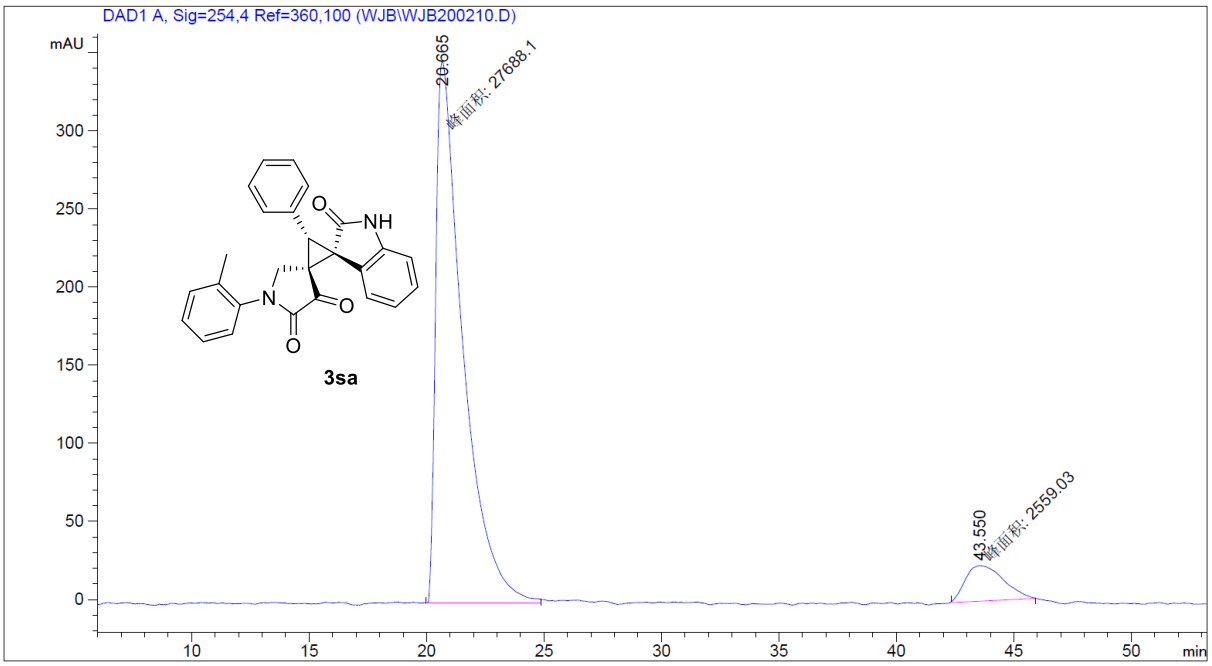
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	29.681	1.4931	9388.31934	104.79726	49.7929
2	34.634	1.2460	9466.41211	101.51268	50.2071



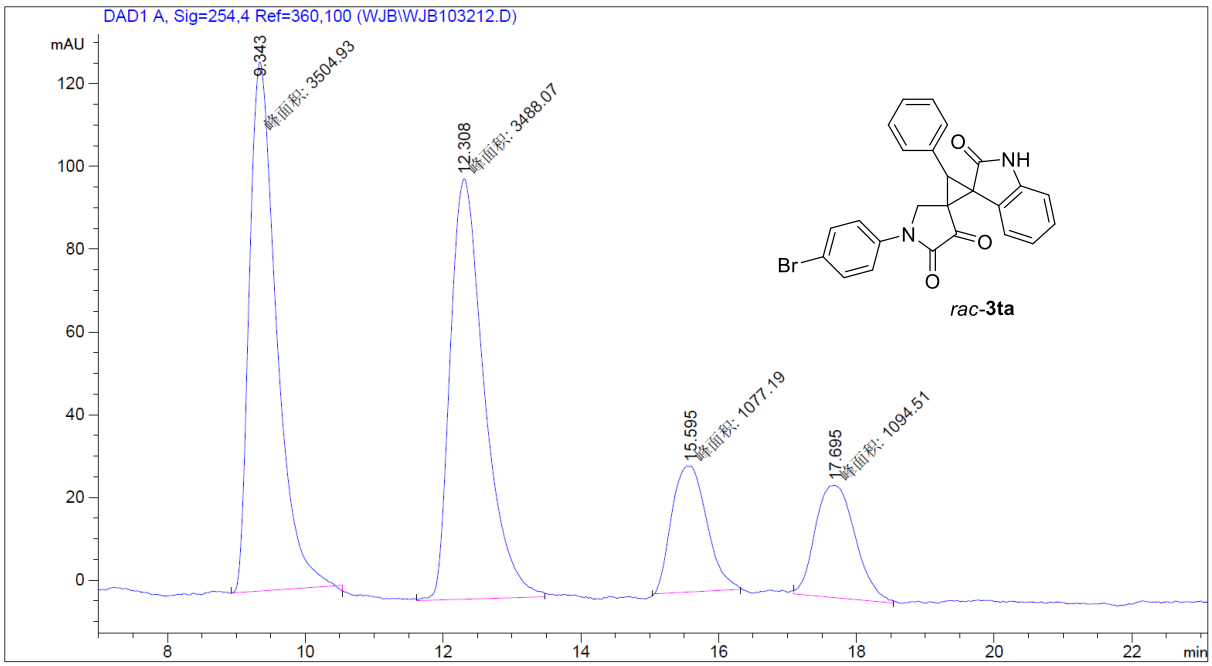
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	30.570	1.1135	1245.01160	13.18698	2.8418
2	33.830	1.3358	4.25657e4	449.08600	97.1582



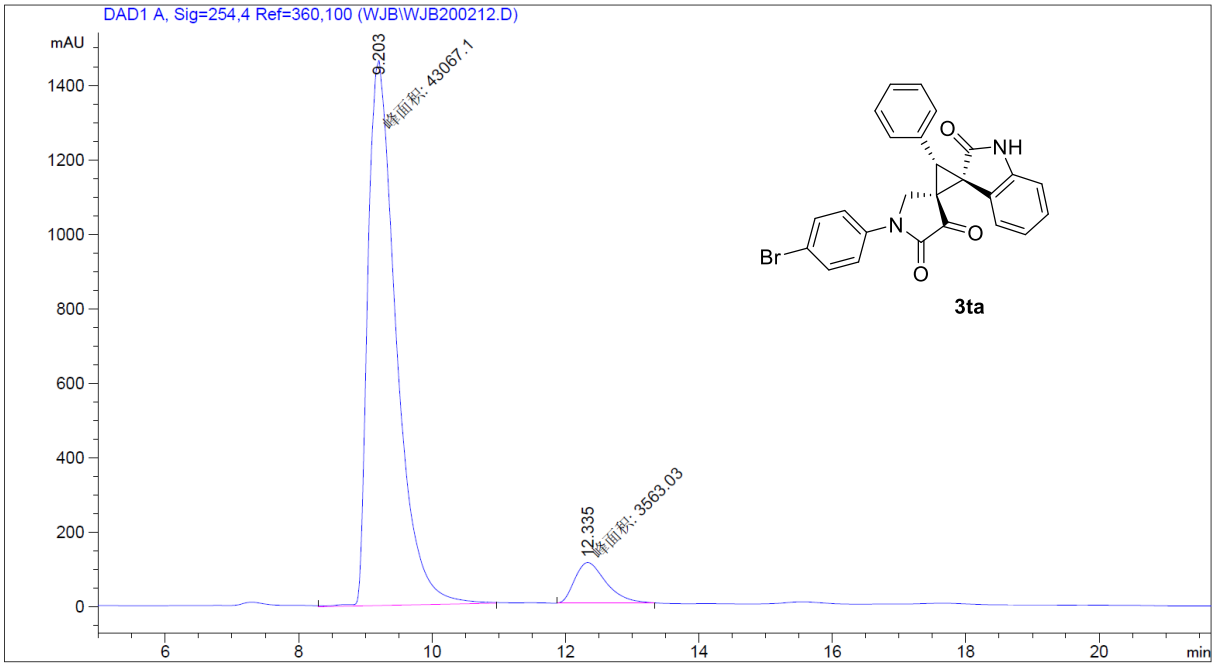
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	21.241	1.2271	9099.09766	123.58936	50.2865
2	43.692	1.9358	8995.42969	77.44735	49.7135



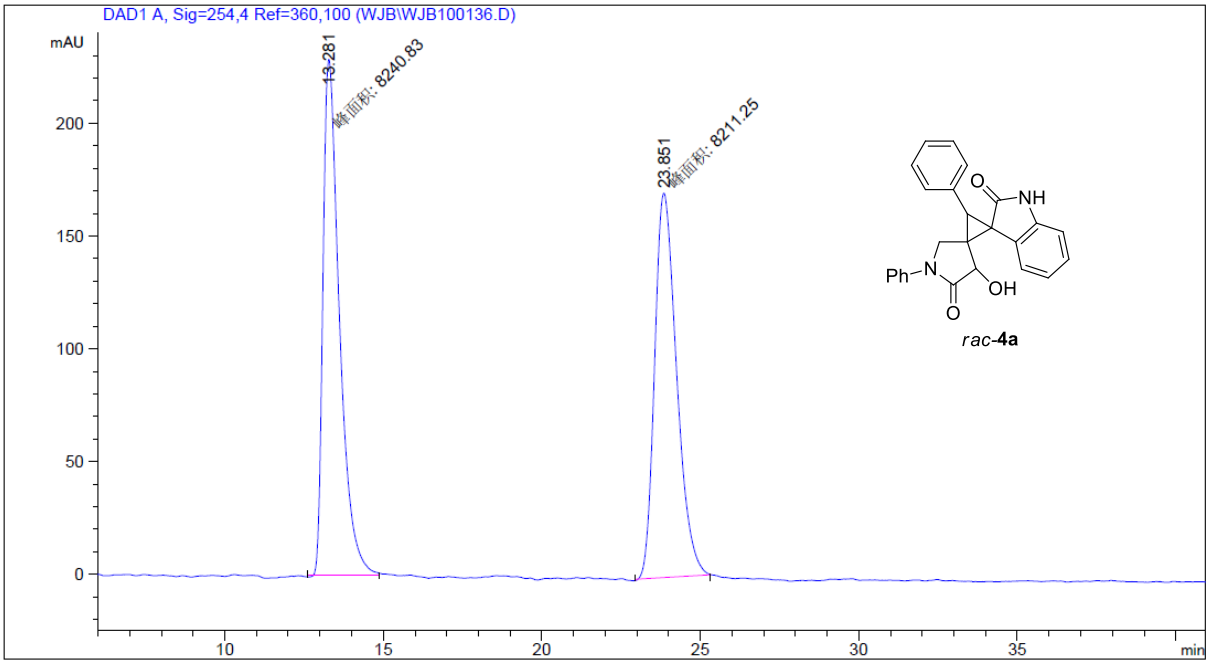
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	20.665	1.3309	2.76881e4	346.72131	91.5396
2	43.550	1.8733	2559.03125	22.76762	8.4604



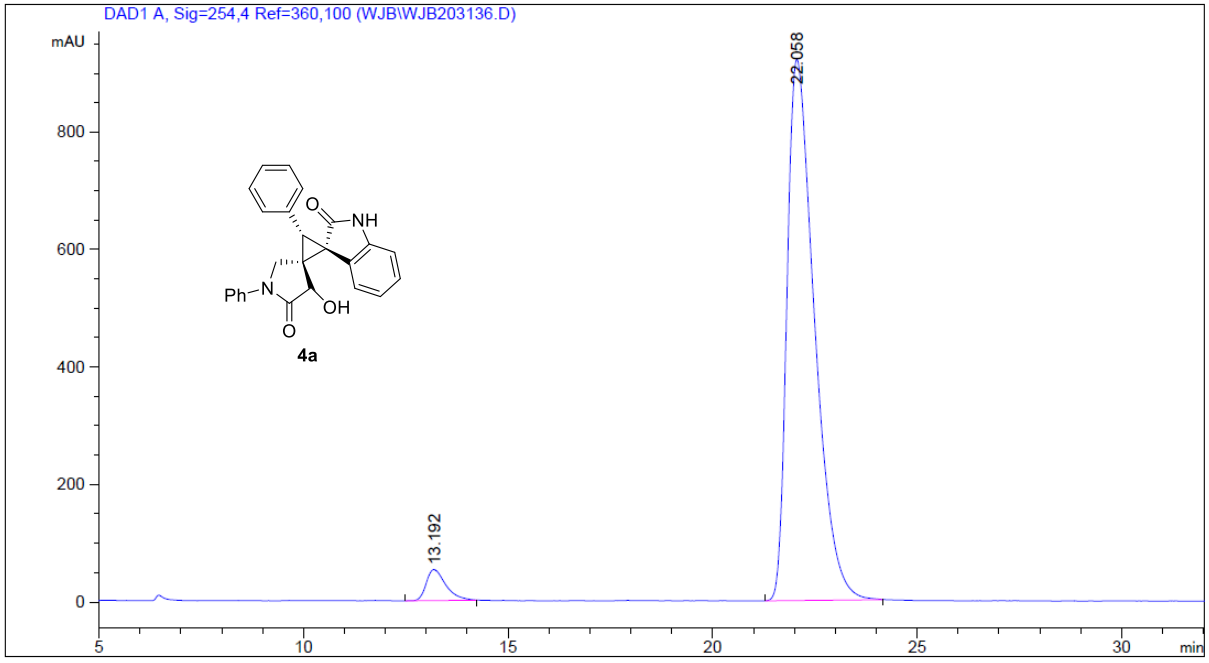
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	9.343	0.4568	3504.93188	127.87173	38.2438
2	12.308	0.5720	3488.07300	101.63976	38.0598
3	15.595	0.5905	1077.19214	30.40416	11.7537
4	17.695	0.6714	1094.50964	27.17075	11.9427



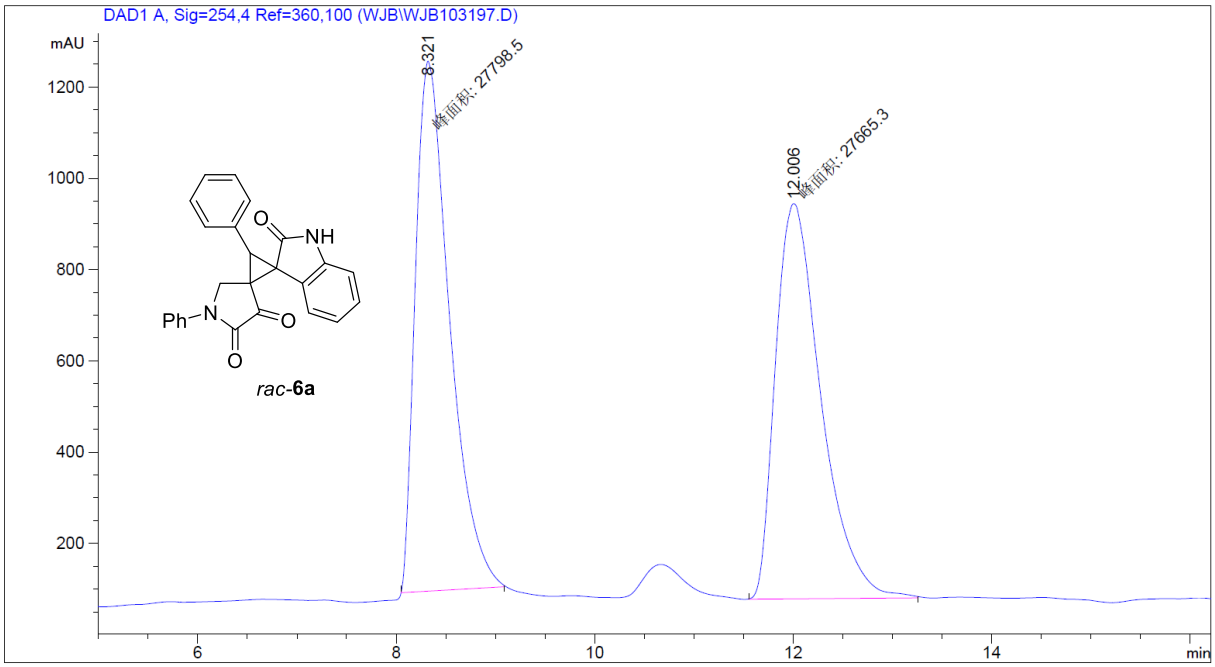
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	9.203	0.4902	4.30671e4	1464.29041	92.3590
2	12.335	0.5452	3563.02954	108.92046	7.6410



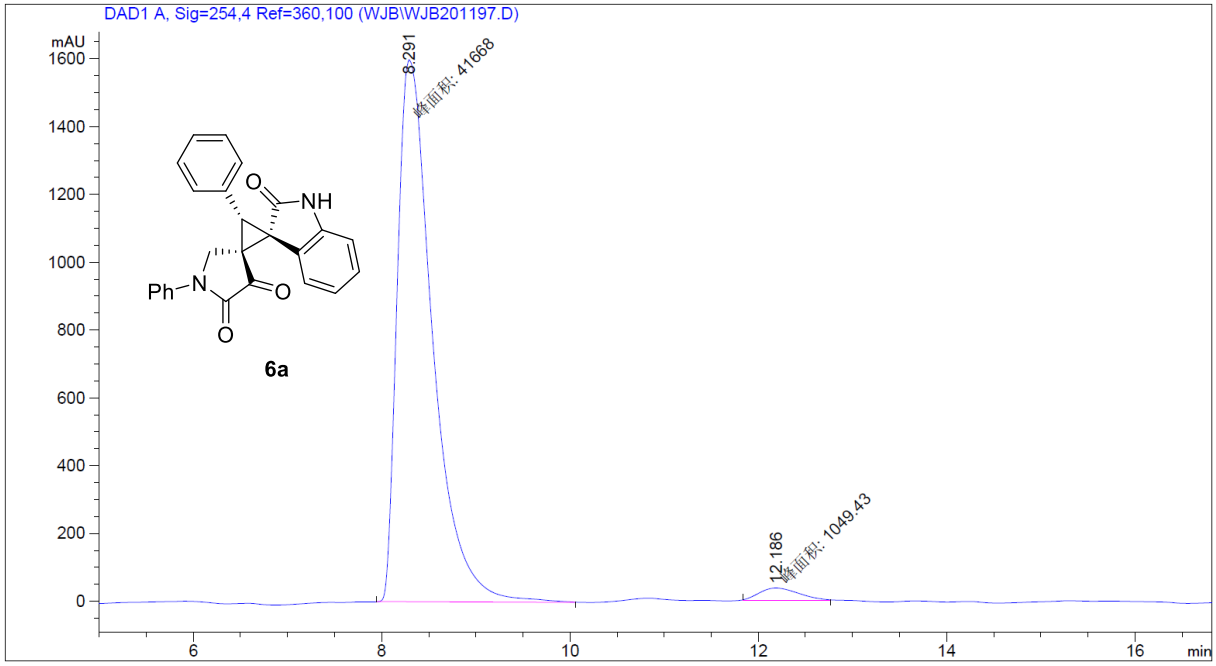
#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.281	0.6010	8240.82813	228.54436	50.0899
2	23.851	0.8031	8211.25195	170.39844	49.9101



#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	13.192	0.4852	1766.45776	52.95348	3.9628
2	22.058	0.7022	4.28100e4	921.09808	96.0372



#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.321	0.3989	2.77985e4	1161.57153	50.1200
2	12.006	0.3525	2.76653e4	865.83978	49.8800



#	Ret Time(min)	Width(min)	Area(Mau*s)	Height(mAU)	Area(%)
1	8.291	0.4343	4.16680e4	1598.86670	97.5433
2	12.186	0.4840	1049.42615	36.13918	2.4567