Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

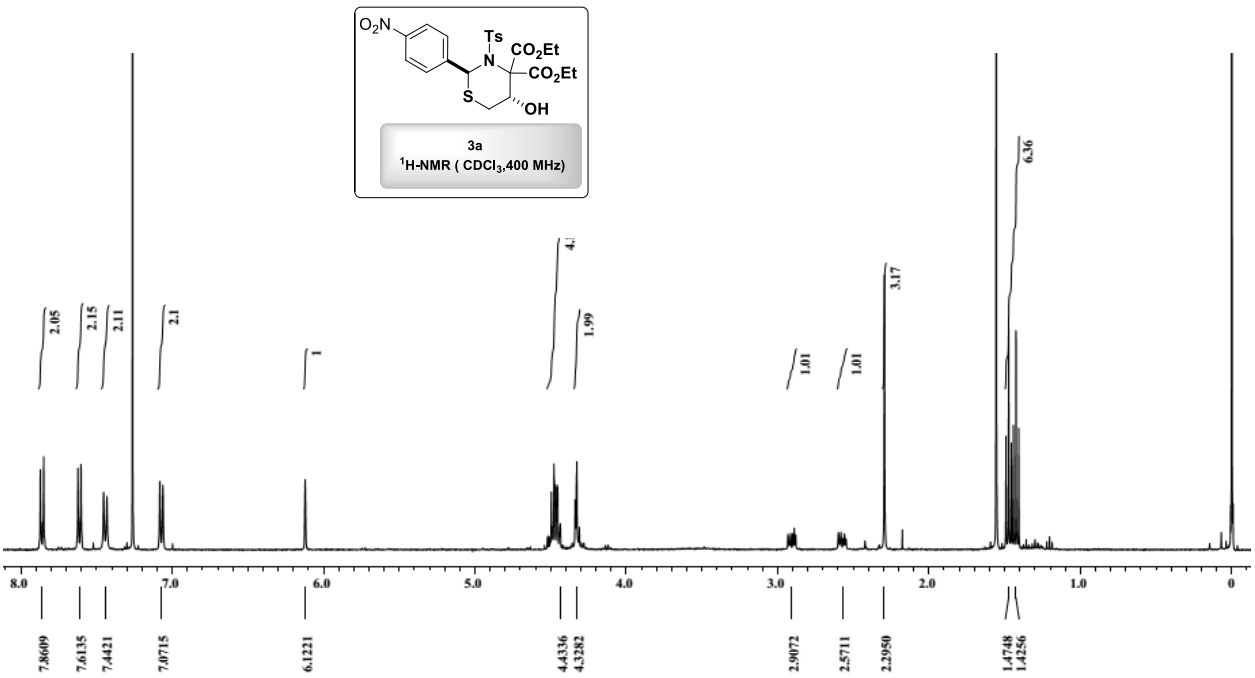
Construction of Thiazines and Oxathianes via [3+3] Annulation of *N*-Tosylaziridinedicarboxylates and Oxiranes with 1,4-dithian-2,5-diol : Application Towards the Synthesis of Bioactive Molecules

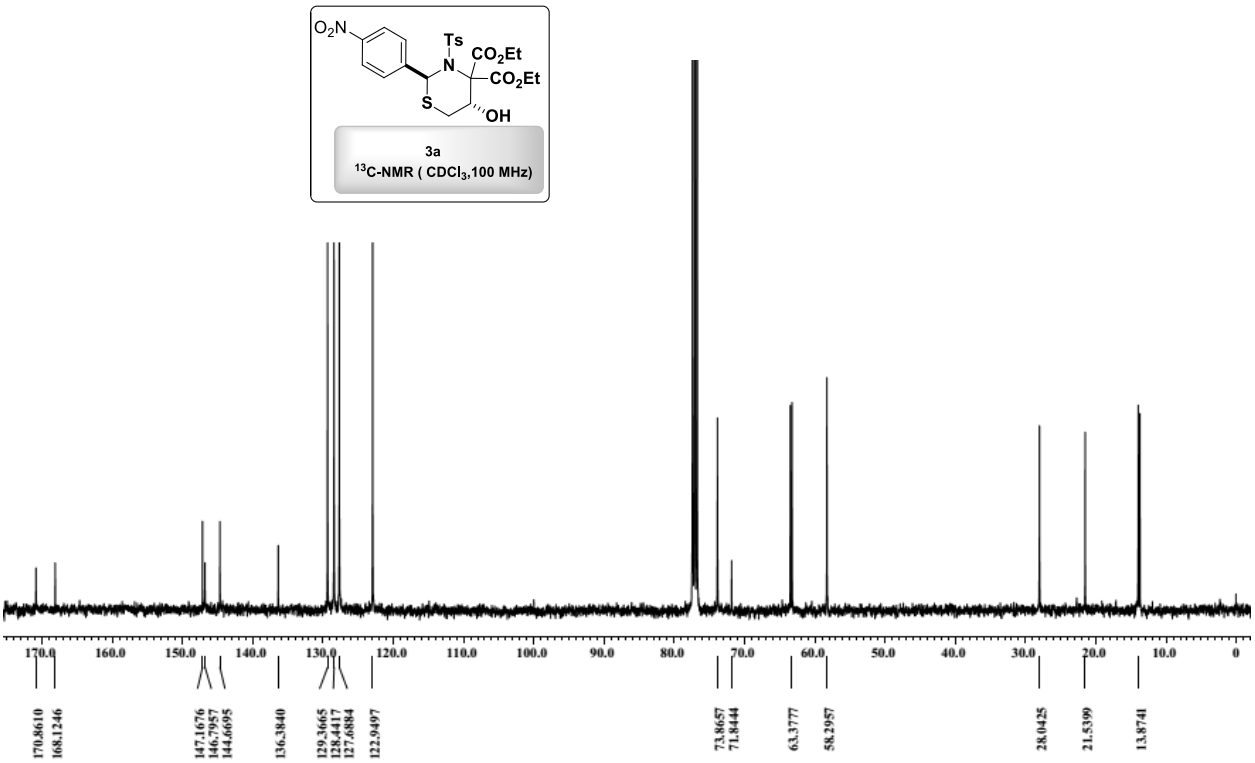
Rohit Kumar Varshnaya, Prabal Banerjee

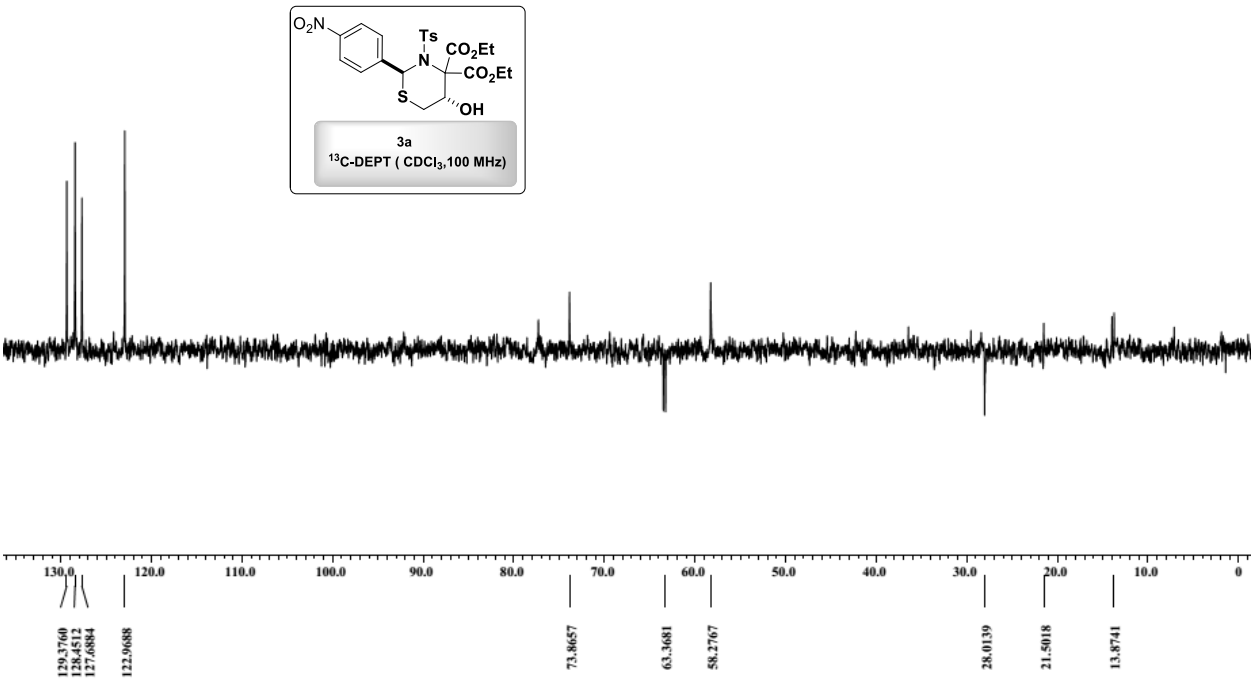
Department of Chemistry, Indian Institute of Technology Ropar,

Nangal Road, Rupnagar, Punjab 140001, India,

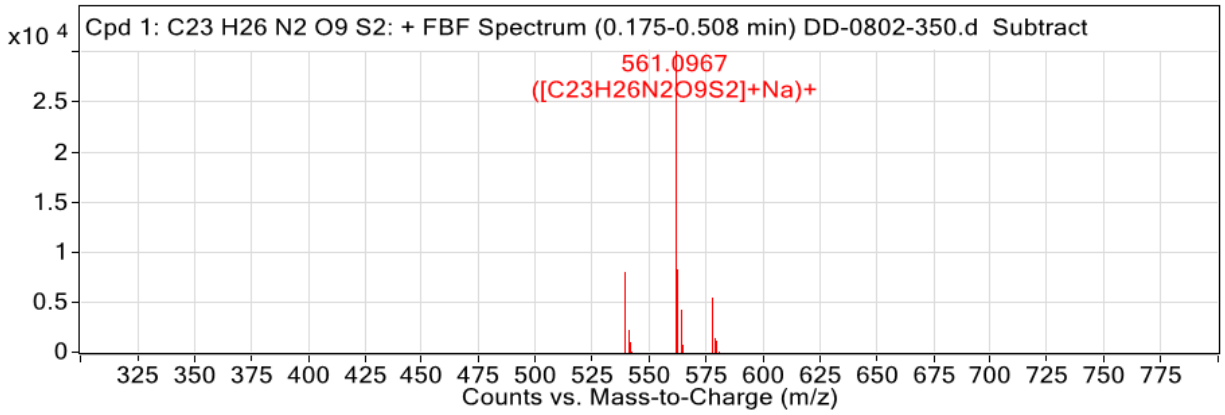
E-mail: prabal@iitrpr.ac.in

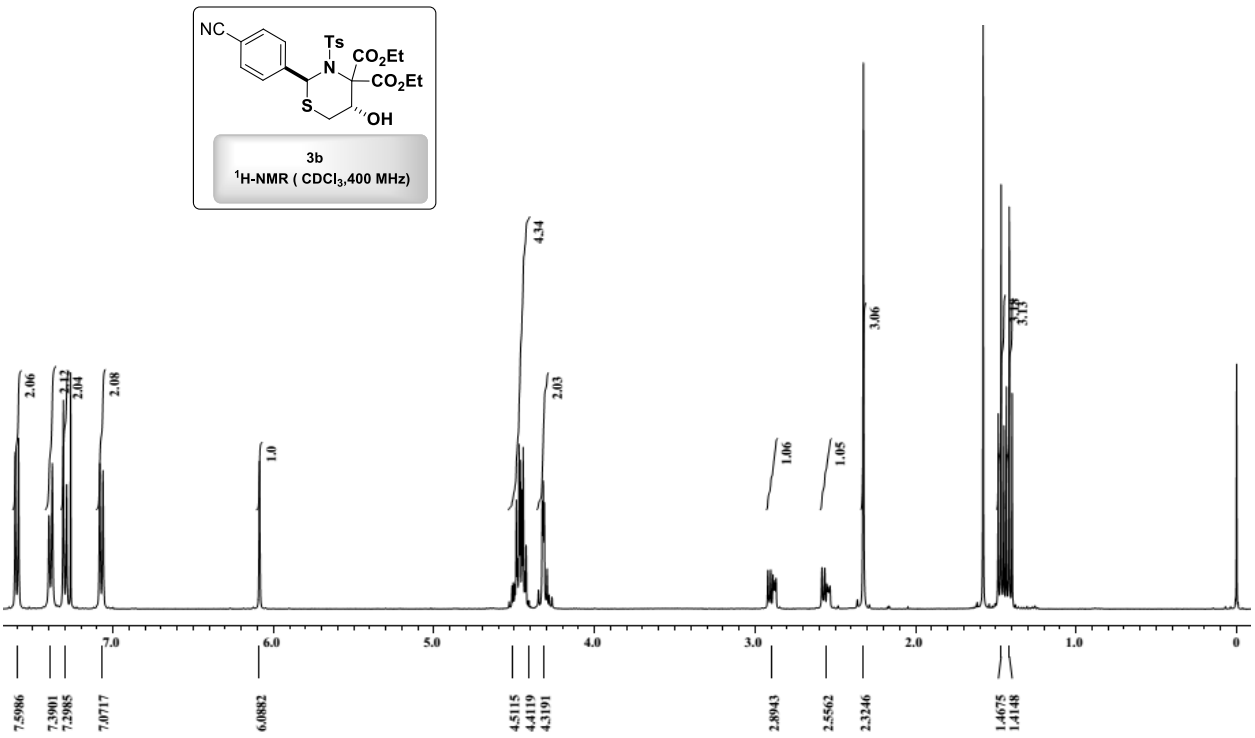

3a
1H-NMR (CDCl3,400 MHz)
2.05
2.15
2.11
2.1
1
1.99
1.01
1.01
3.17
6.36
7.8609
7.6135
7.4421
7.0715
6.1221
4.4336
4.3282
2.9072
2.5711
2.2950
1.4748
1.4256

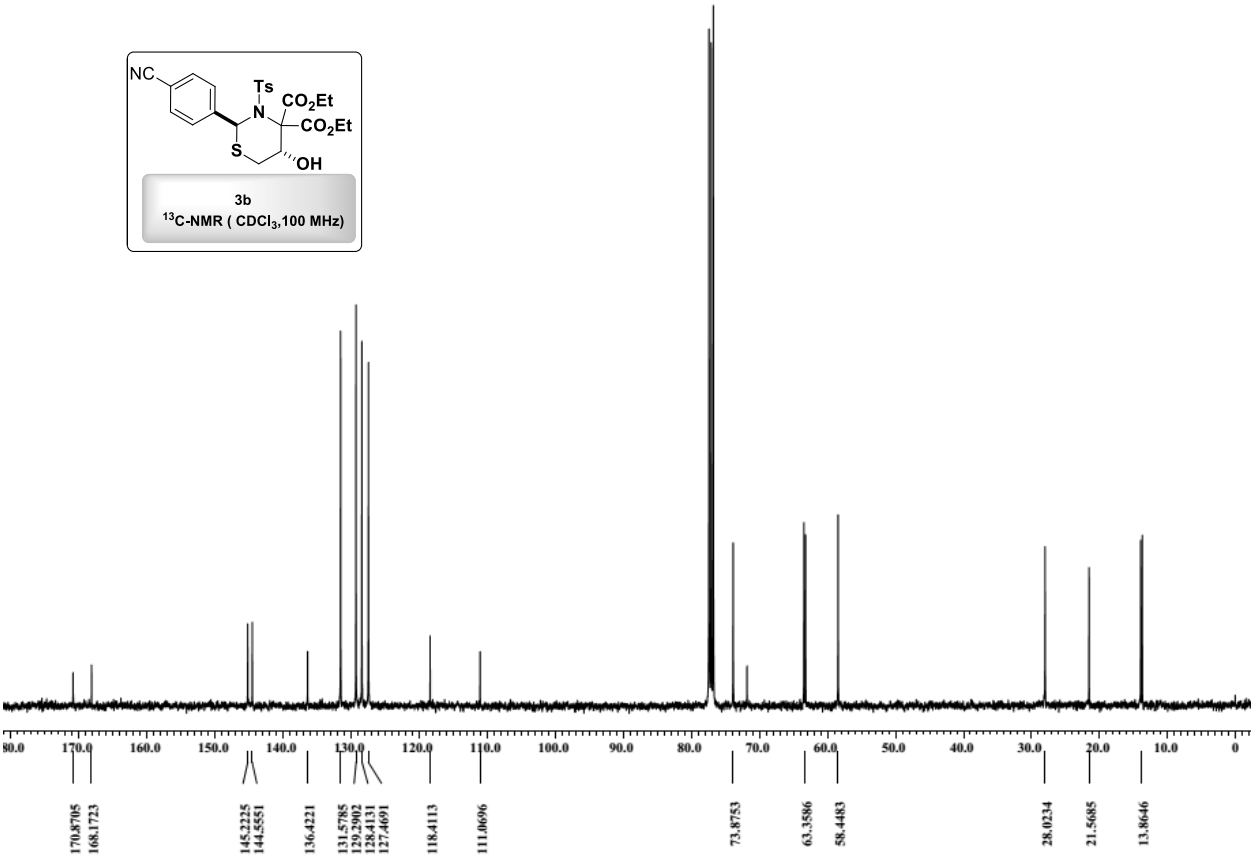

3a
13C-NMR (CDCl3,100 MHz)
170.8610
168.1246
147.1676
146.7957
144.6695
136.3840
129.3665
128.4417
127.6884
122.9497
73.8657
71.8444
63.3777
58.2957
28.0425
21.5399
13.8741

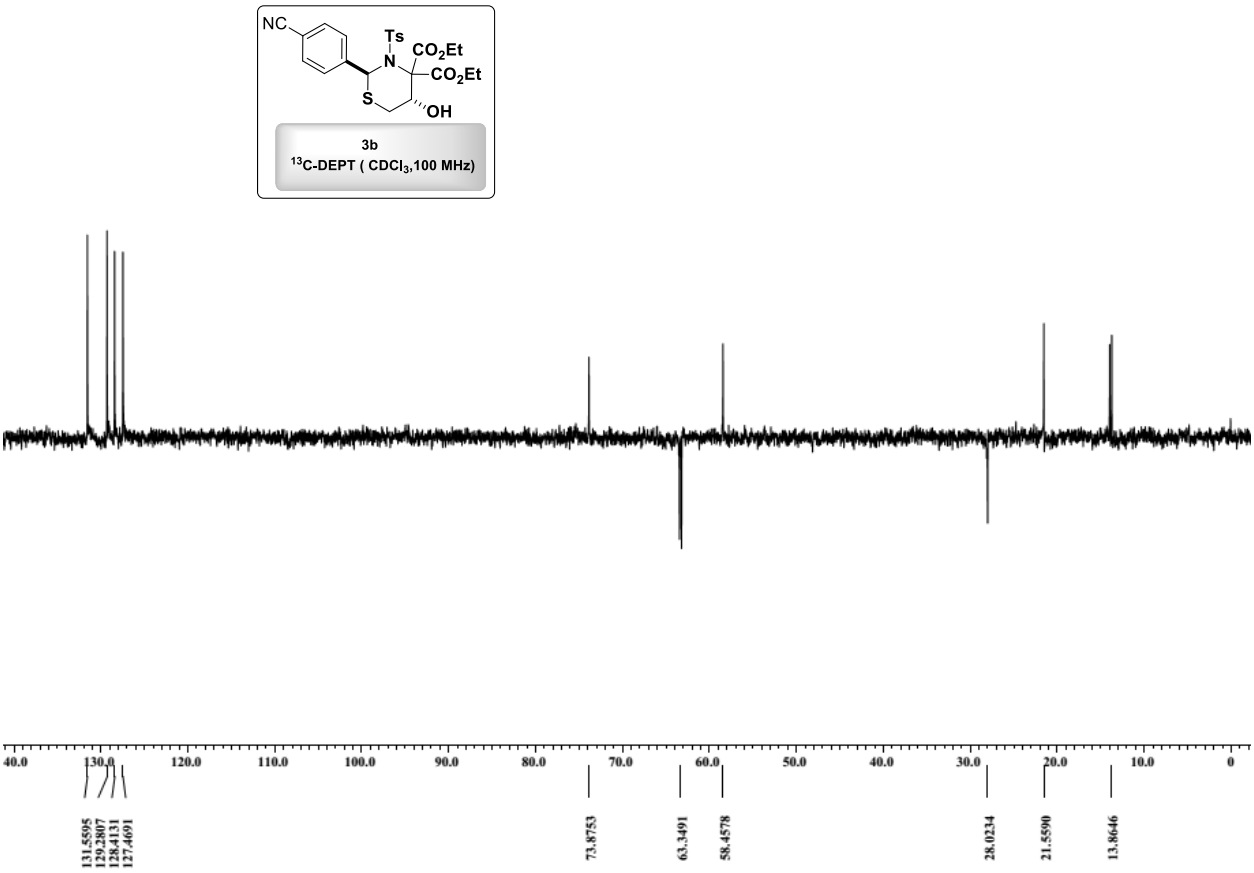

O2N
Ts
N
CO2Et
CO2Et
S
OH
3a
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.3760
128.4512
127.6884
122.9688
73.8657
63.3681
58.2767
28.0139
21.5018
13.8741

HRMS of 3a:

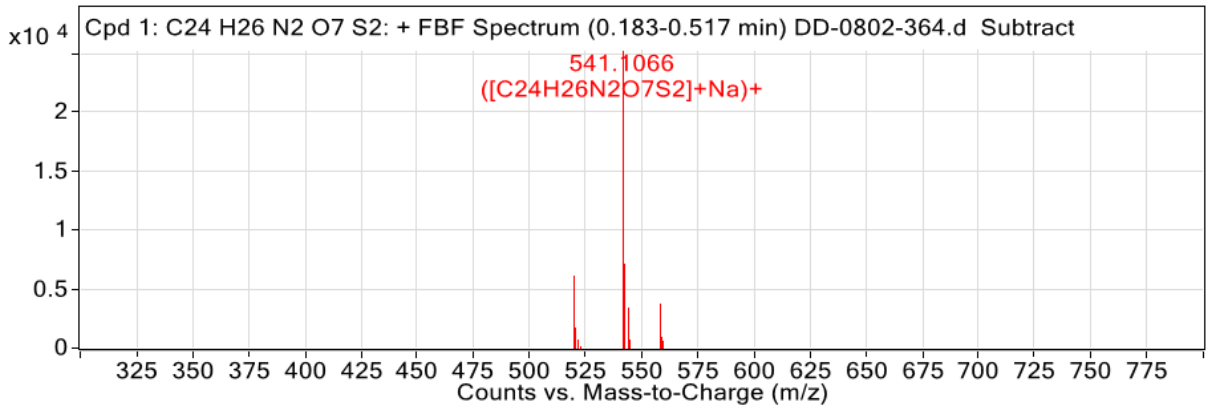

x10 4
Cpd 1: C23 H26 N2 O9 S2: + FBF Spectrum (0.175-0.508 min) DD-0802-350.d Subtract
561.0967
([C23H26N2O9S2]+Na)+
2.5
2
1.5
1
0.5
0
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

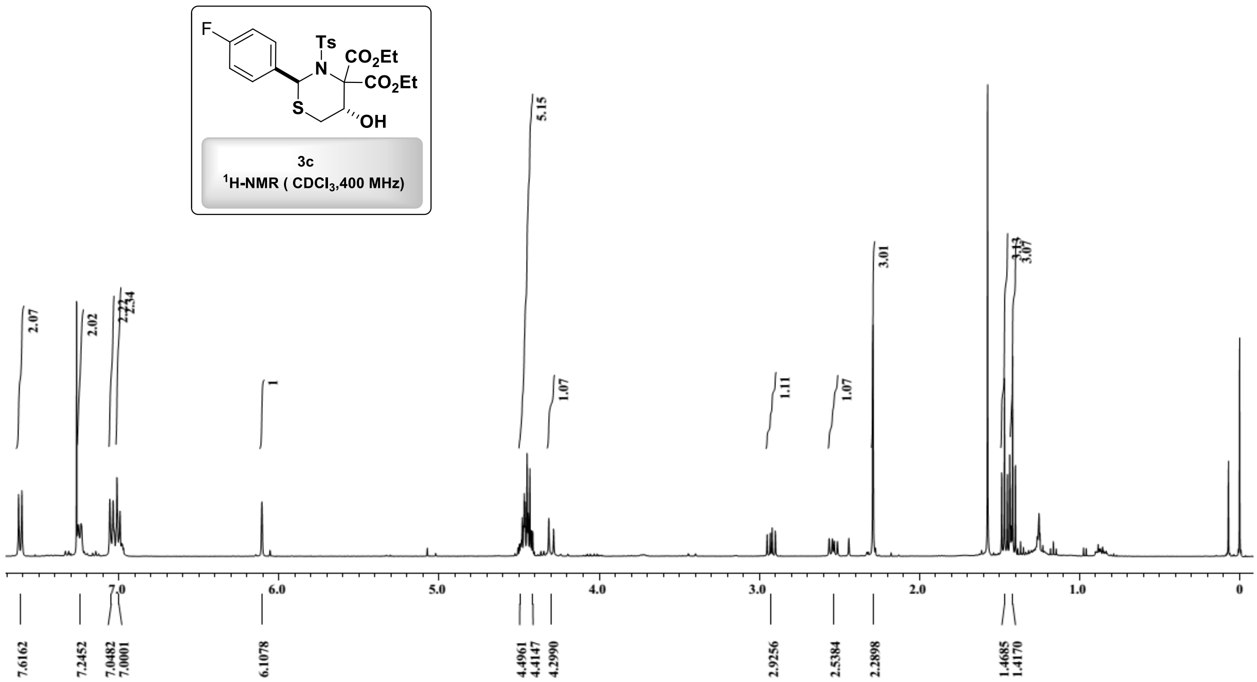

NC
Ts
N
CO2Et
CO2Et
S
OH
3b
1H-NMR (CDCl3,400 MHz)
2.06
2.13
2.04
2.08
1.0
4.34
2.03
1.06
1.05
3.06
3.13
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.5986
7.3901
7.2985
7.0717
6.0882
4.5115
4.4119
4.3191
2.8943
2.5562
2.3246
1.4675
1.4148

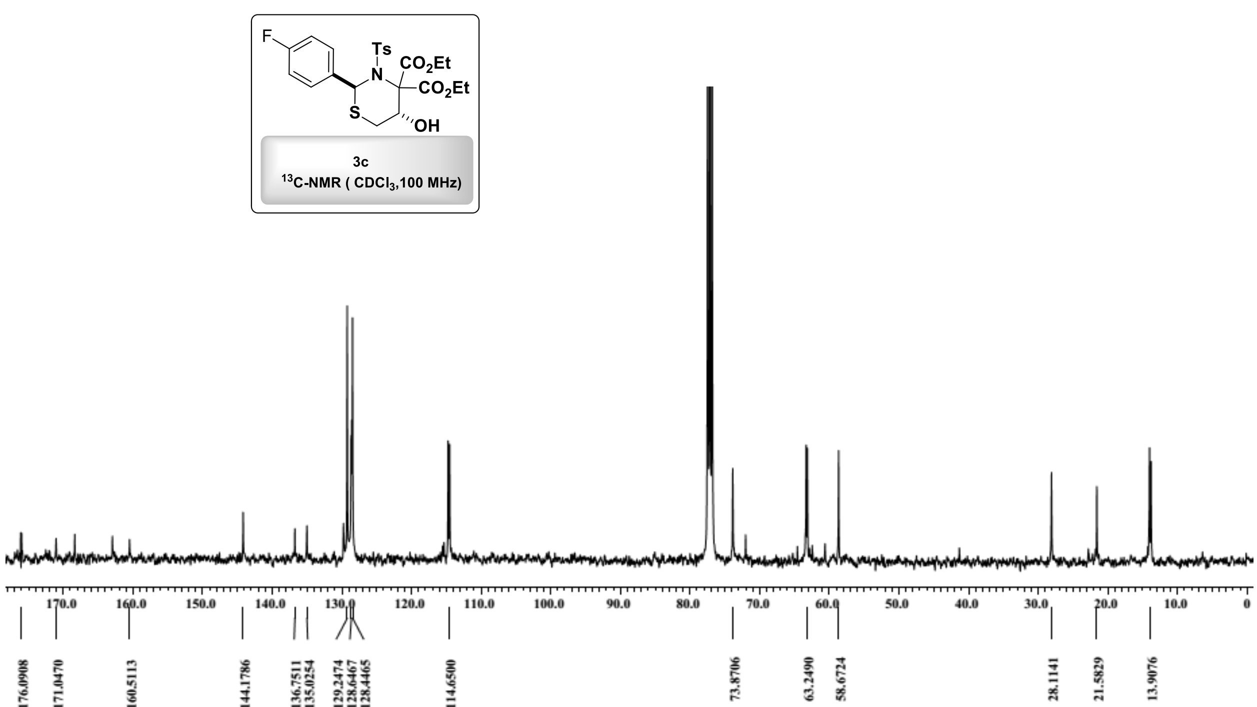

NC
Ts
N
CO2Et
CO2Et
S
OH
3b
13C-NMR (CDCl3,100 MHz)
80.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.8705
168.1723
145.2235
144.5551
136.4221
131.5785
129.2902
128.4111
127.4691
118.4113
111.0696
73.8753
63.3586
58.4483
28.0234
21.5685
13.8646

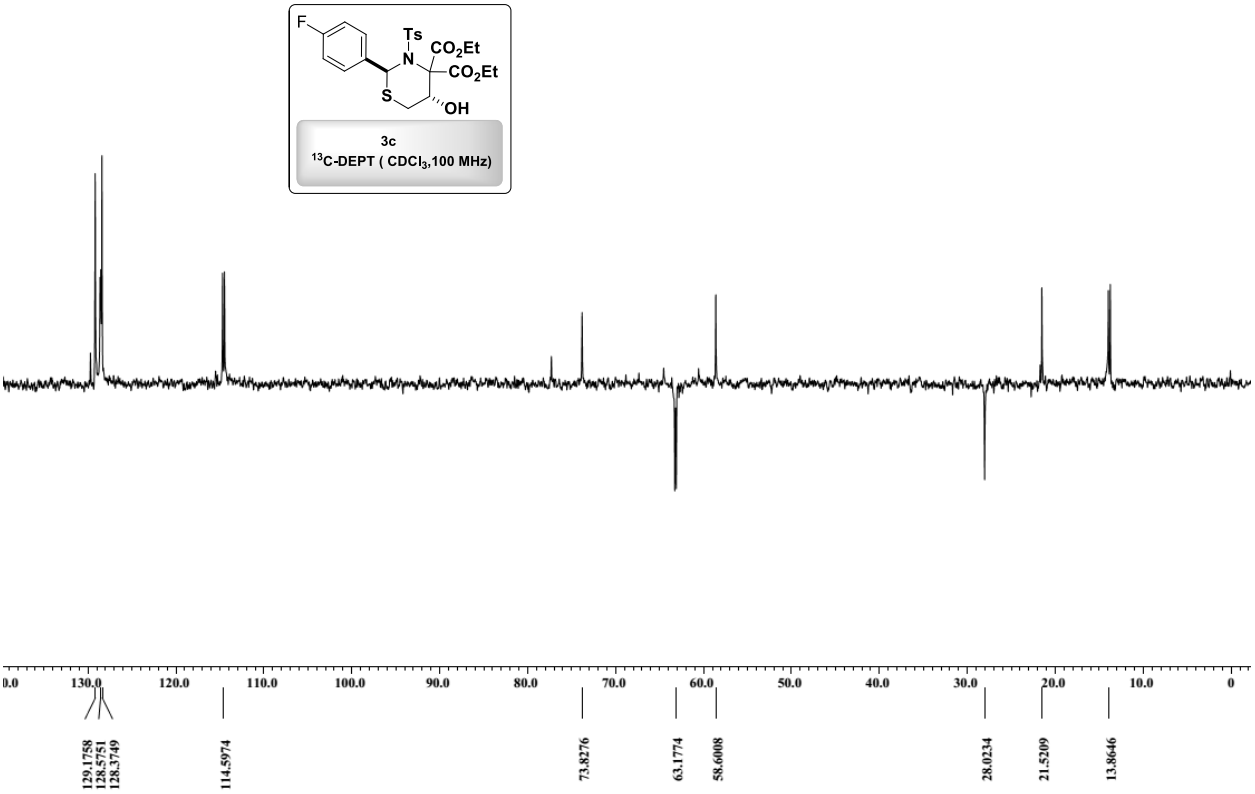

NC
Ts
N
CO2Et
CO2Et
S
OH
3b
13C-DEPT (CDCl3,100 MHz)
40.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
131.5595
129.2807
128.4131
127.4691
73.8753
63.3491
58.4578
28.0234
21.5590
13.8646

HRMS of 3b:

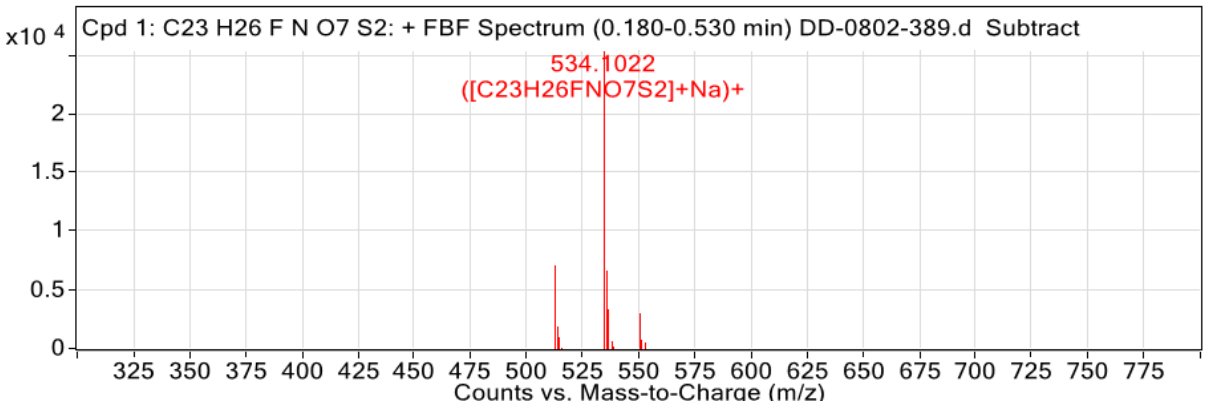

x10 4
Cpd 1: C24 H26 N2 O7 S2: + FBF Spectrum (0.183-0.517 min) DD-0802-364.d Subtract
541.1066
([C24H26N2O7S2]+Na)+
2
1.5
1
0.5
0
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

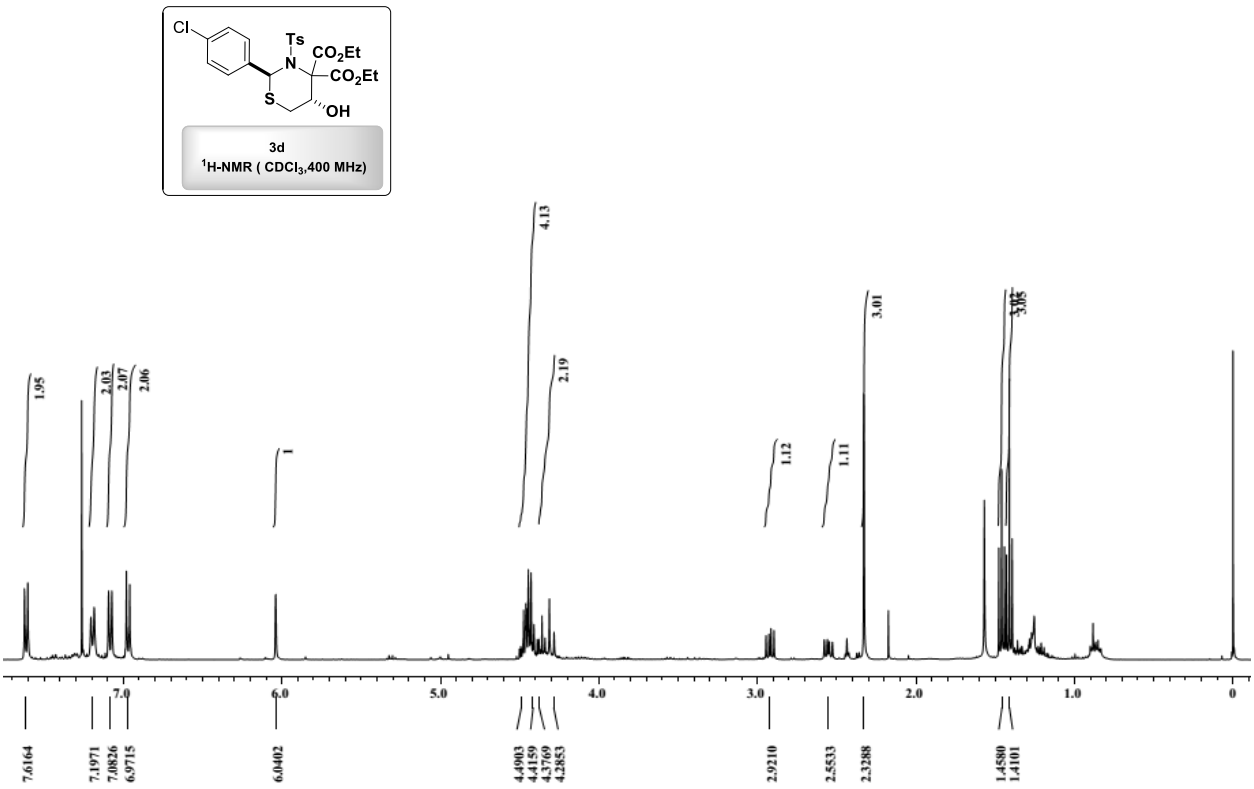

F
Ts
N
CO2Et
CO2Et
S
OH
3c
1H-NMR (CDCl3,400 MHz)
7.6162
7.2452
7.0482
7.0001
6.1078
4.4961
4.4147
4.2990
2.9256
2.5384
2.2898
1.4685
1.4170

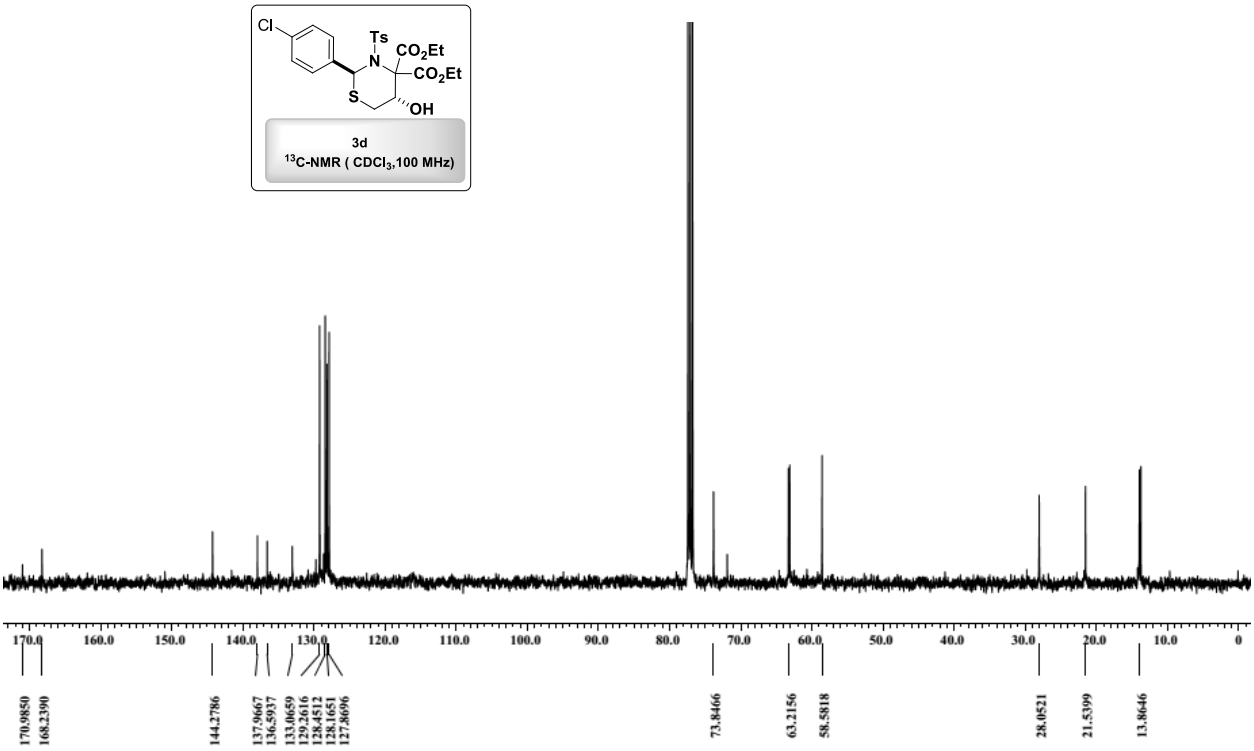

F
Ts
N
CO2Et
CO2Et
S
OH
3c
13C-NMR (CDCl3,100 MHz)
176.0908
171.0470
160.5113
144.1786
136.7511
135.0254
129.2474
128.6467
128.4465
114.6500
73.8706
63.2490
58.6724
28.1141
21.5829
13.9076

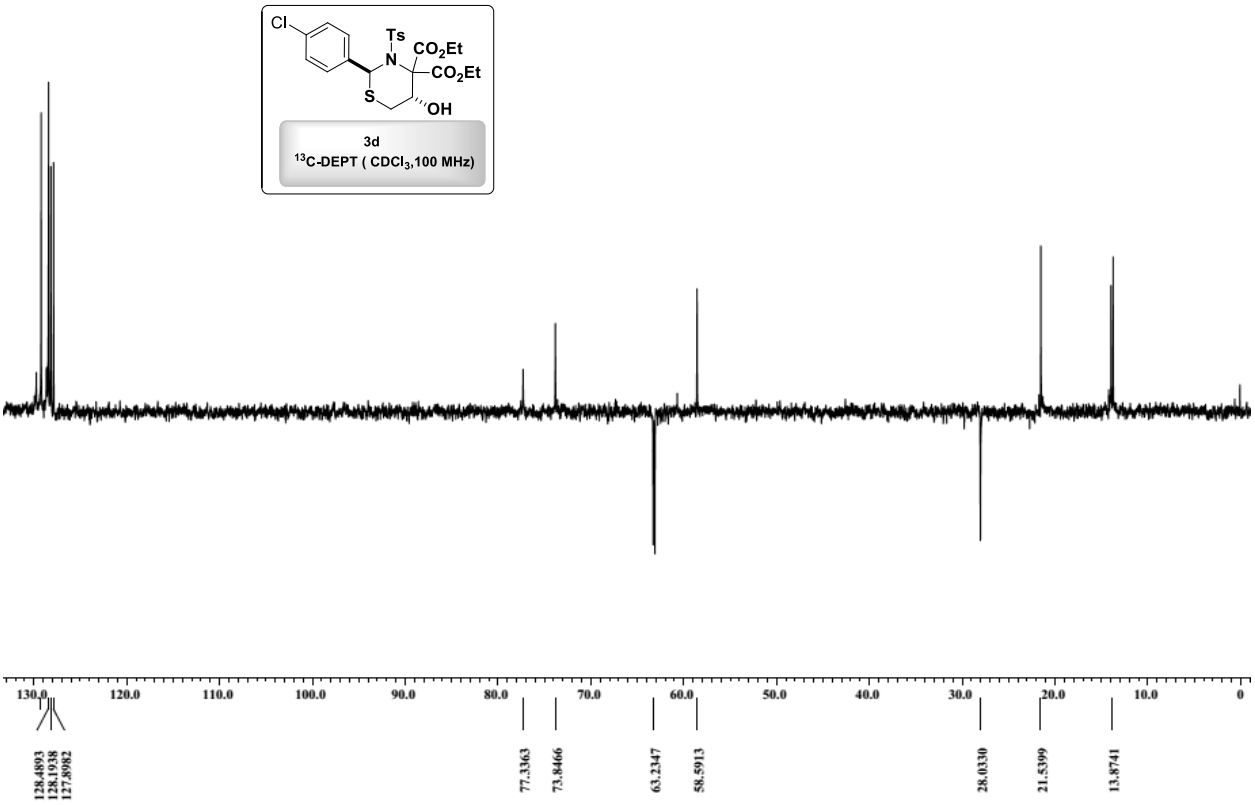

F
Ts
N
CO2Et
CO2Et
S
OH
3c
13C-DEPT (CDCl3,100 MHz)
0.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.1758
128.5751
128.3749
114.5974
73.8276
63.1774
58.6008
28.0234
21.5209
13.8646

HRMS of 3c:

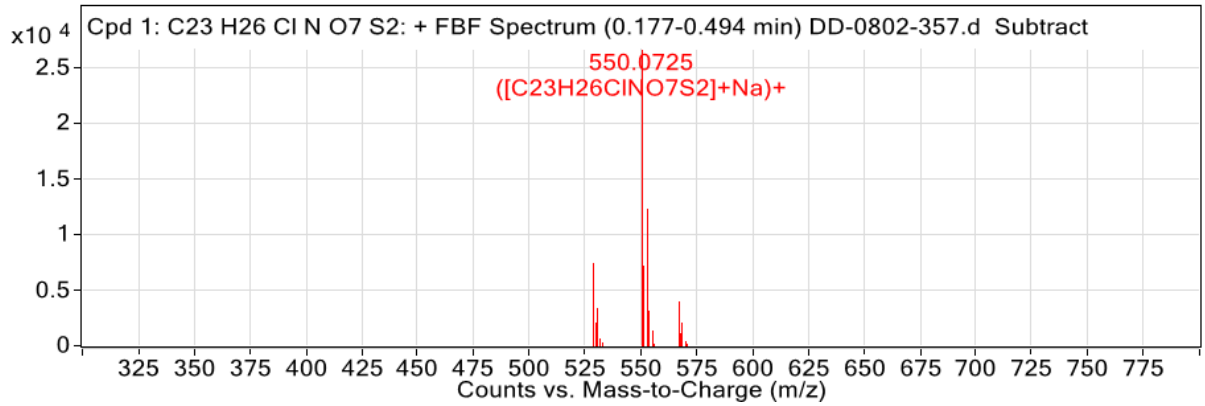

x10 4
Cpd 1: C23 H26 F N O7 S2: + FBF Spectrum (0.180-0.530 min) DD-0802-389.d Subtract
534.1022
([C23H26FNO7S2]+Na)+
2
1.5
1
0.5
0
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

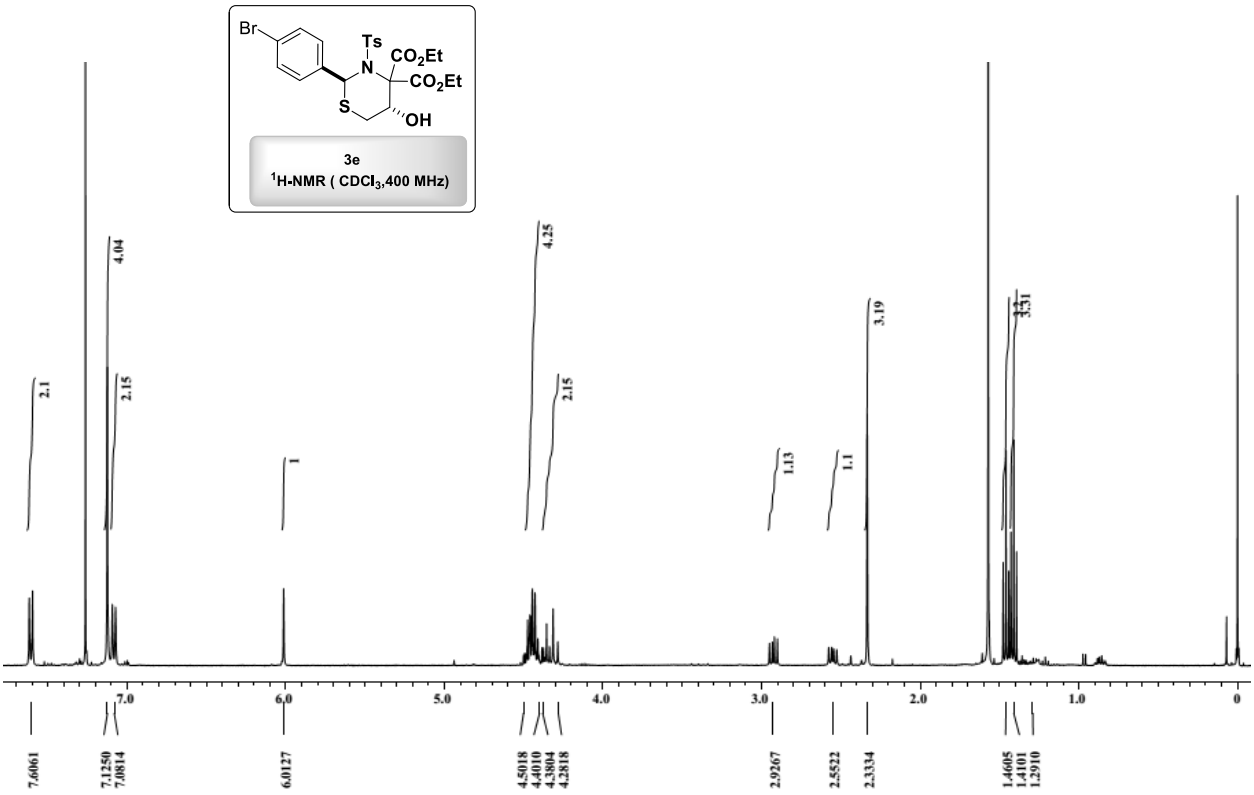

Cl
Ts
N
CO2Et
CO2Et
S
OH
3d
1H-NMR (CDCl3,400 MHz)
1.95
2.03
2.07
2.06
1
4.13
2.19
1.12
1.11
3.01
7.6164
7.1971
7.0826
6.9715
6.0402
4.4903
4.4159
4.3769
4.2853
2.9210
2.5533
2.3288
1.4580
1.4101

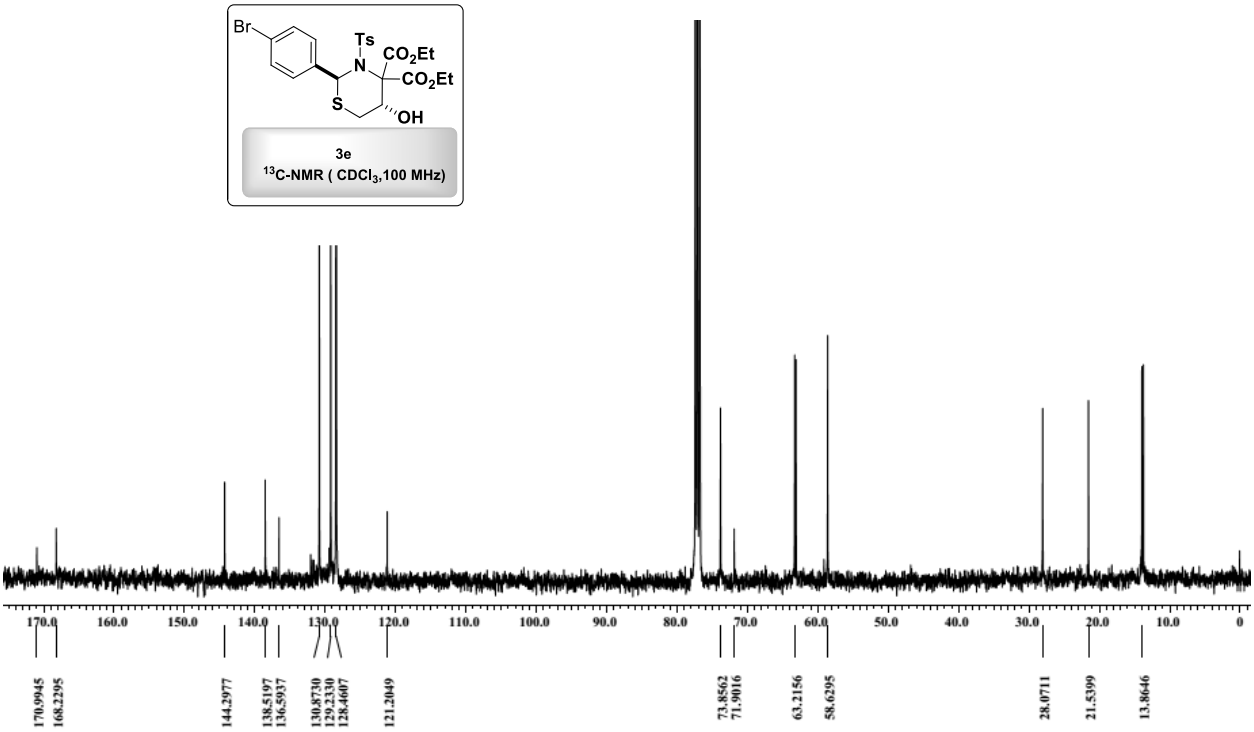

Cl
Ts
N
CO2Et
CO2Et
S
OH
3d
13C-NMR (CDCl3,100 MHz)
170.9850
168.2390
144.2786
137.9667
136.5937
133.0659
129.2616
128.4512
128.1651
127.8696
73.8466
63.2156
58.5818
28.0521
21.5399
13.8646

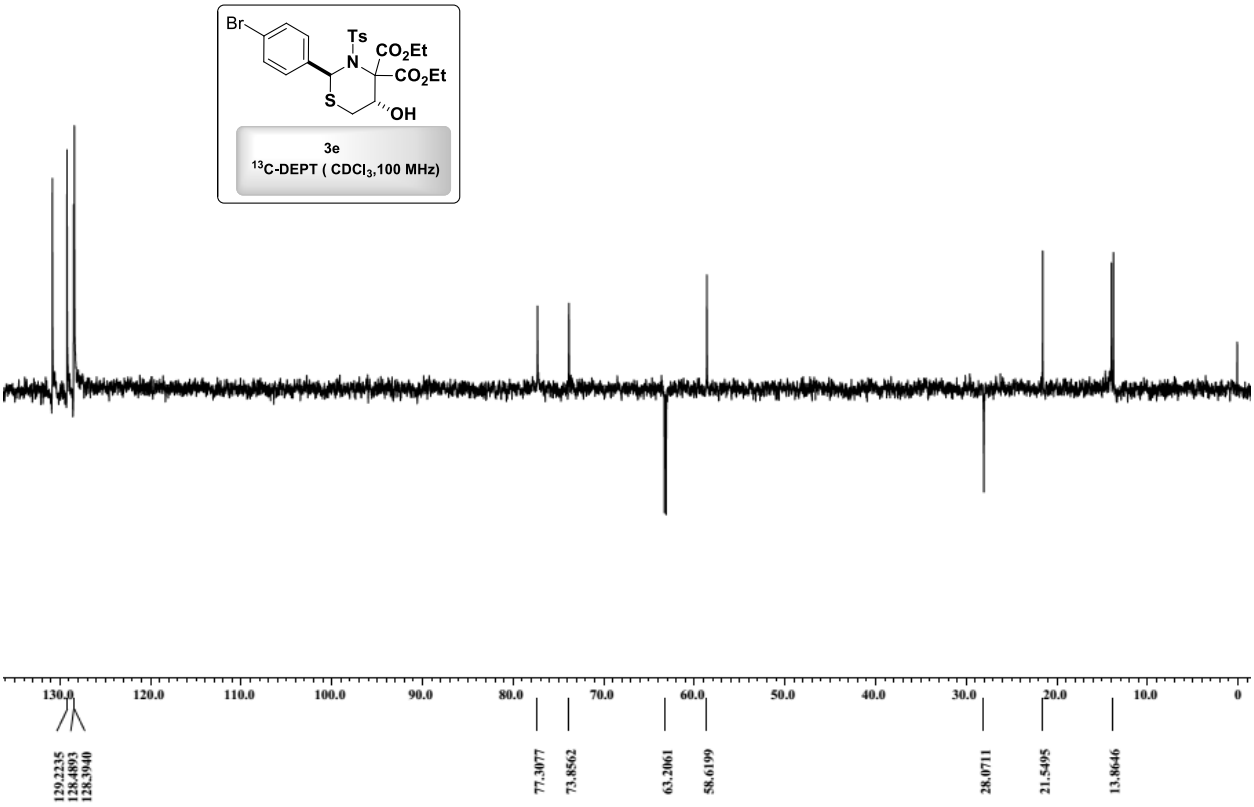

Cl
Ts
N
CO2Et
CO2Et
S
OH
3d
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
128.4893
128.1938
127.8982
77.3363
73.8466
63.2347
58.5913
28.0330
21.5399
13.8741

HRMS of 3d:

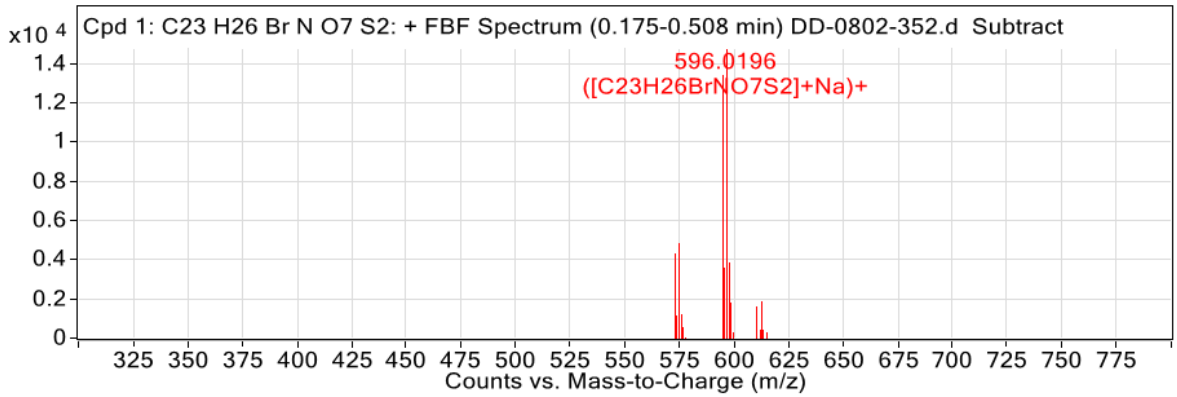

x10 4
Cpd 1: C23 H26 Cl N O7 S2: + FBF Spectrum (0.177-0.494 min) DD-0802-357.d Subtract
550.0725
([C23H26ClNO7S2]+Na)+
2.5
2
1.5
1
0.5
0
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

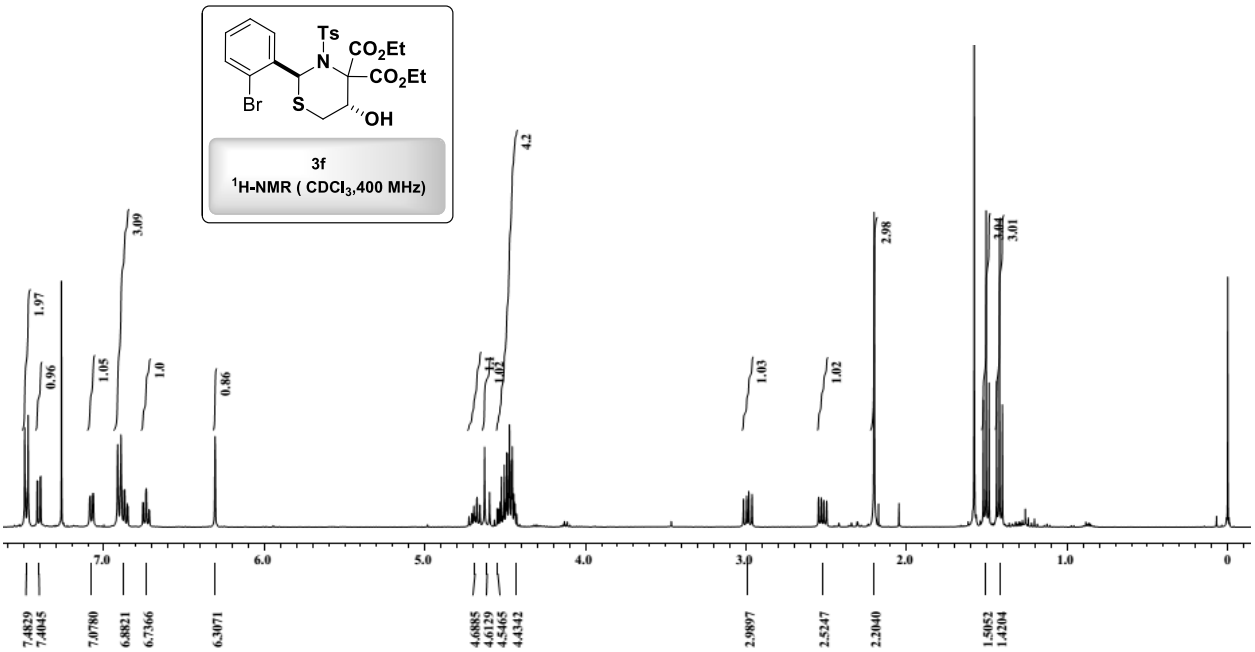

Br
Ts
N
CO2Et
CO2Et
S
OH
3e
1H-NMR (CDCl3,400 MHz)
2.1
4.04
2.15
1
4.25
2.15
1.13
1.1
3.19
3.31
7.6061
7.1250
7.0814
6.0127
4.5018
4.4010
4.3804
4.2818
2.9267
2.5522
2.3334
1.4605
1.4101
1.2910

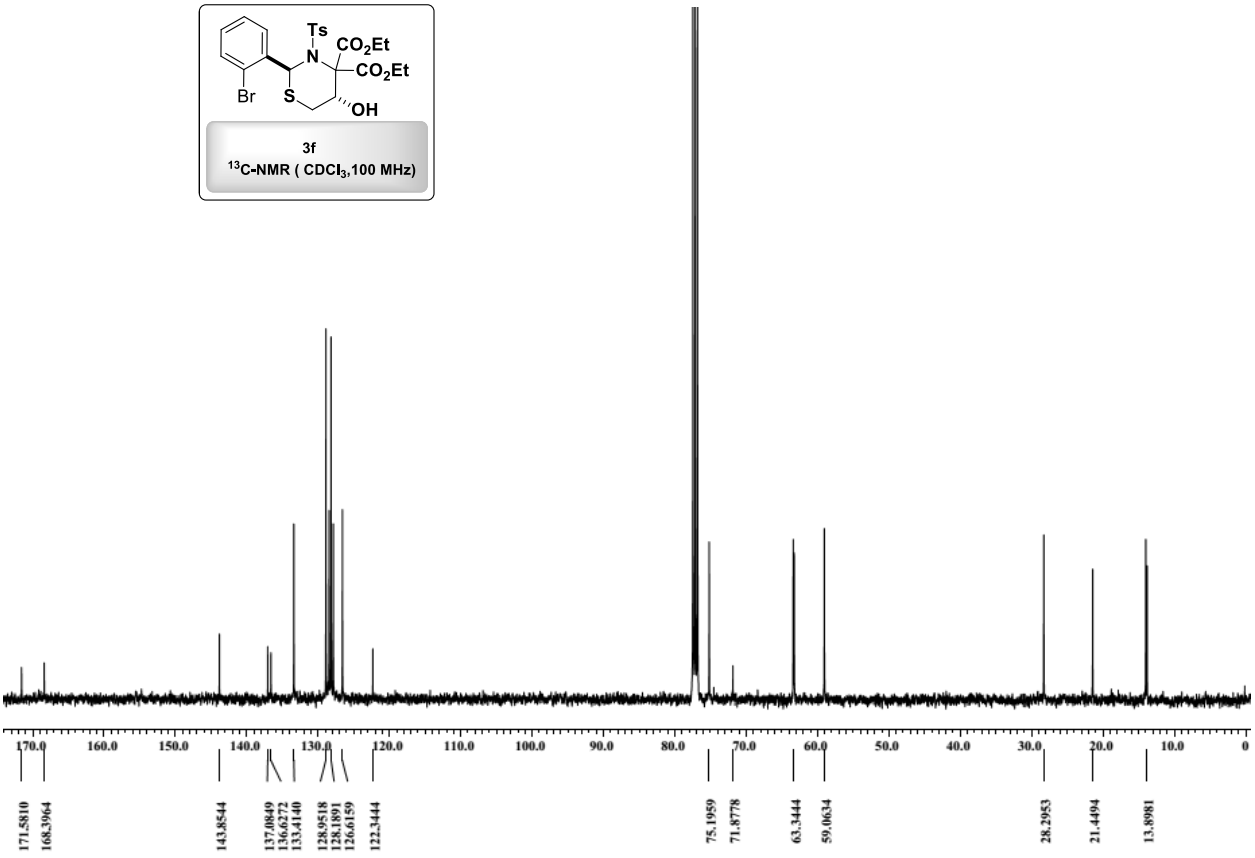

Br
Ts
N
CO2Et
CO2Et
S
OH
3e
13C-NMR (CDCl3,100 MHz)
170.9945
168.2295
144.2977
138.5197
136.5937
130.8730
129.2330
128.4607
121.2049
73.8562
71.9016
63.2156
58.6295
28.0711
21.5399
13.8646

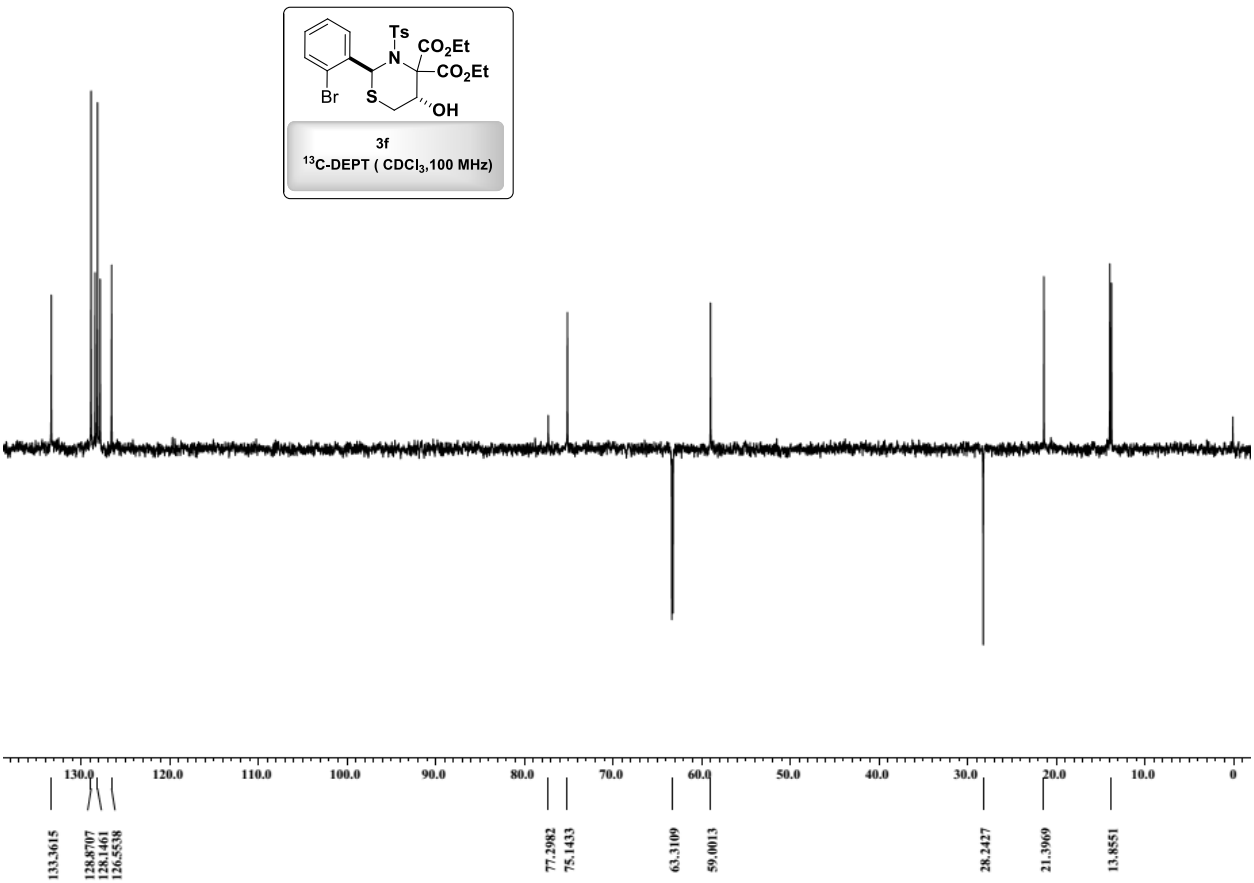

Br
Ts
N
CO2Et
CO2Et
S
OH
3e
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.2235
128.4893
128.3940
77.3077
73.8562
63.2061
58.6199
28.0711
21.5495
13.8646

HRMS of 3e:

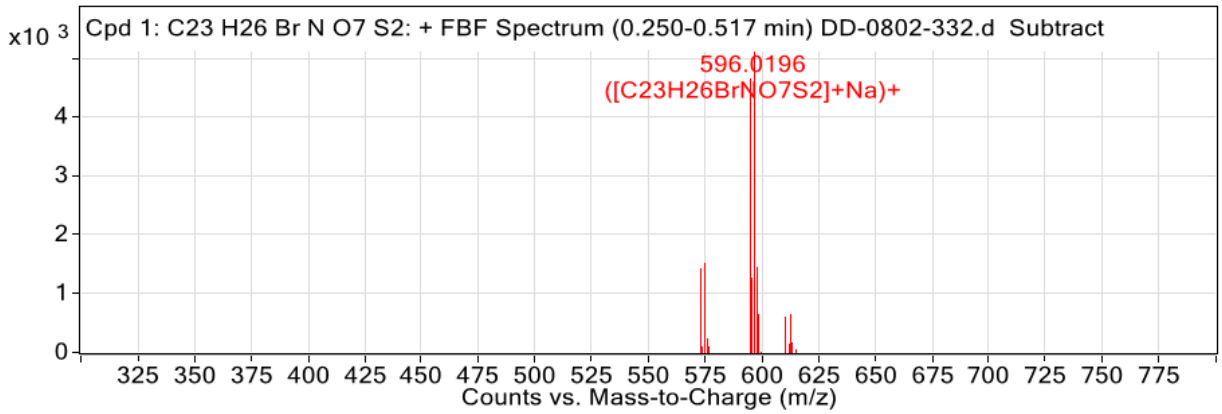

Cpd 1: C23 H26 Br N O7 S2: + FBF Spectrum (0.175-0.508 min) DD-0802-352.d Subtract
x10 4
1.4
1.2
1
0.8
0.6
0.4
0.2
0
596.0196
([C23H26BrNO7S2]+Na)+
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

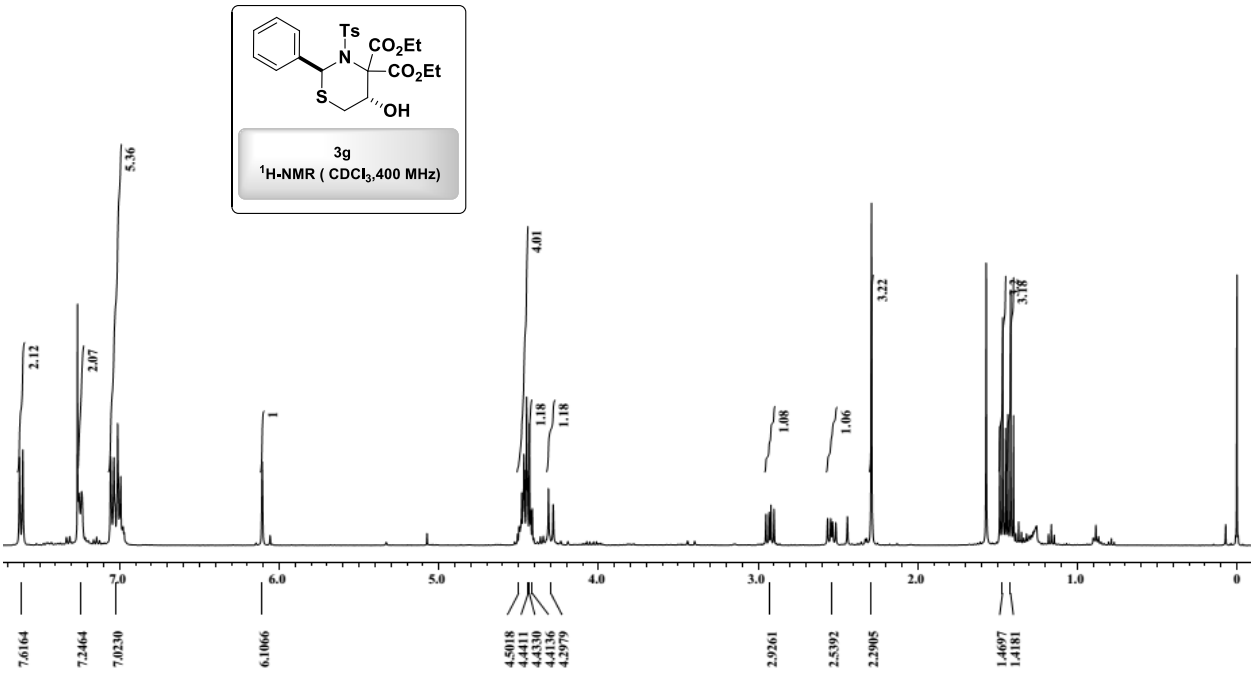

Ts
N
CO2Et
CO2Et
Br
S
OH
3f
1H-NMR (CDCl3,400 MHz)
1.97
0.96
1.05
3.09
1.0
0.86
4.2
1.03
1.02
2.98
3.04
3.01
7.4829
7.4045
7.0780
6.8821
6.7366
6.3071
4.6885
4.6129
4.5465
4.4342
2.9897
2.5247
2.2040
1.5052
1.4204

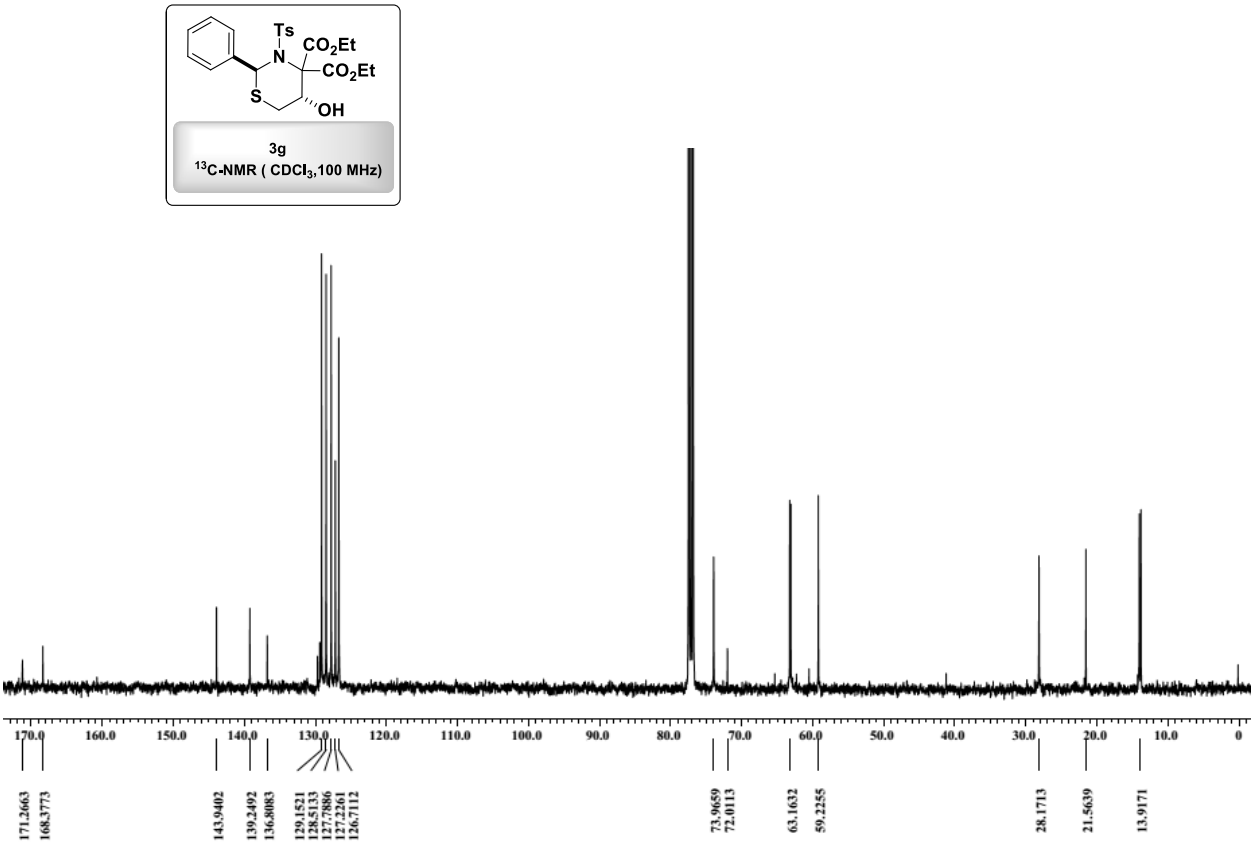

Ts
N
CO2Et
CO2Et
Br
S
OH
3f
13C-NMR (CDCl3,100 MHz)
171.5810
168.3964
143.8544
137.0849
136.6272
133.4140
128.9518
128.1891
126.6159
122.3444
75.1959
71.8778
63.3444
59.0634
28.2953
21.4494
13.8981

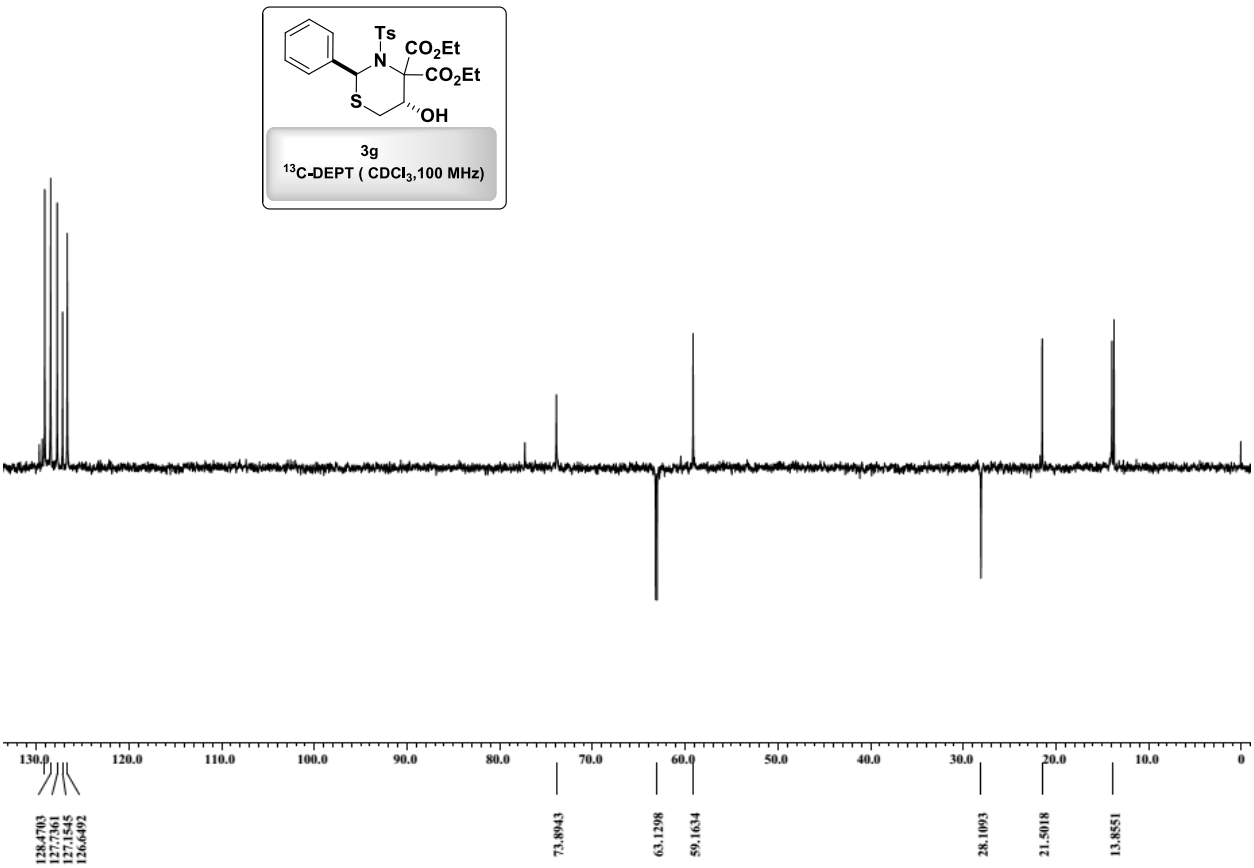

Ts
N
CO2Et
CO2Et
Br
S
OH
3f
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
133.3615
128.8707
128.1461
126.5538
77.2982
75.1433
63.3109
59.0013
28.2427
21.3969
13.8551

HRMS of 3f:

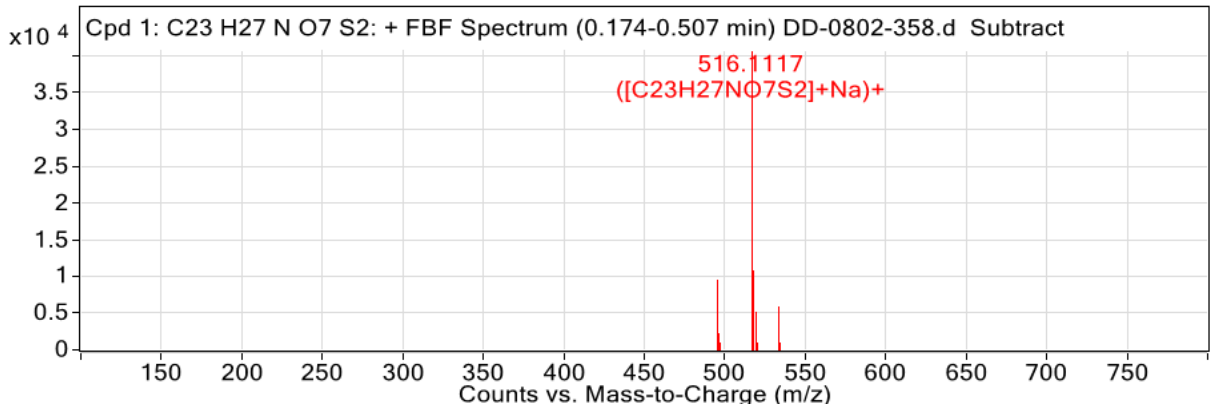

Cpd 1: C23 H26 Br N O7 S2: + FBF Spectrum (0.250-0.517 min) DD-0802-332.d Subtract
x10 3
4
3
2
1
0
596.0196
([C23H26BrNO7S2]+Na)+
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

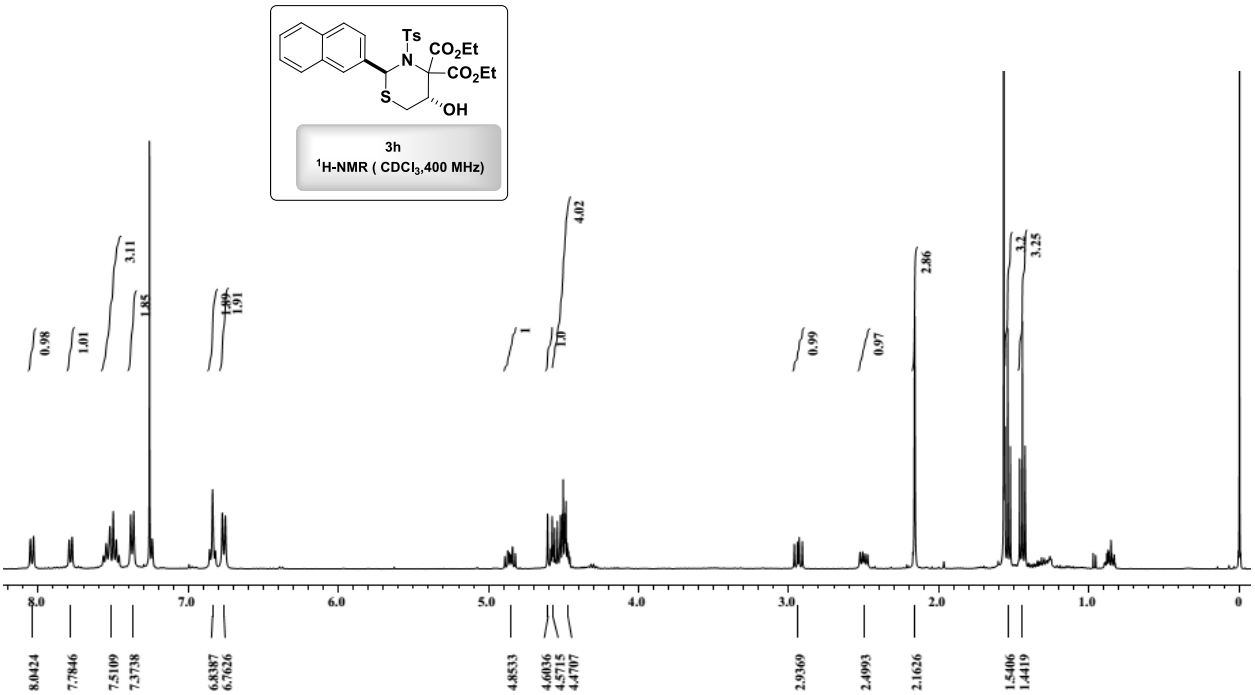

Ts
N
CO2Et
CO2Et
S
OH
3g
1H-NMR (CDCl3,400 MHz)
5.36
2.12
2.07
1
4.01
1.18
1.18
1.08
1.06
3.22
3.18
7.6164
7.2464
7.0230
6.1066
4.5018
4.4411
4.4330
4.4136
4.2979
2.9261
2.5392
2.2905
1.4697
1.4181

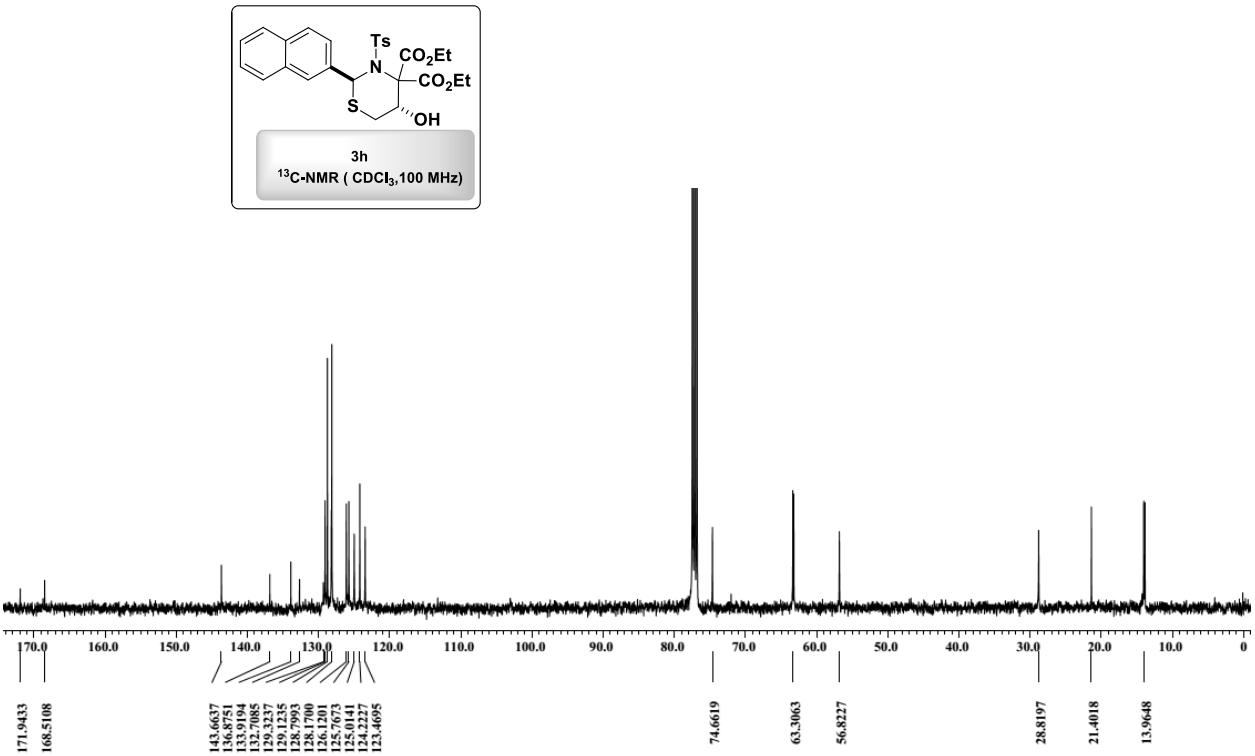

Ts
N
CO2Et
CO2Et
S
OH
3g
13C-NMR (CDCl3,100 MHz)
171.2663
168.3773
143.9402
139.2492
136.8083
129.1521
128.5133
127.7886
127.2261
126.7112
73.9659
72.0113
63.1632
59.2255
28.1713
21.5639
13.9171

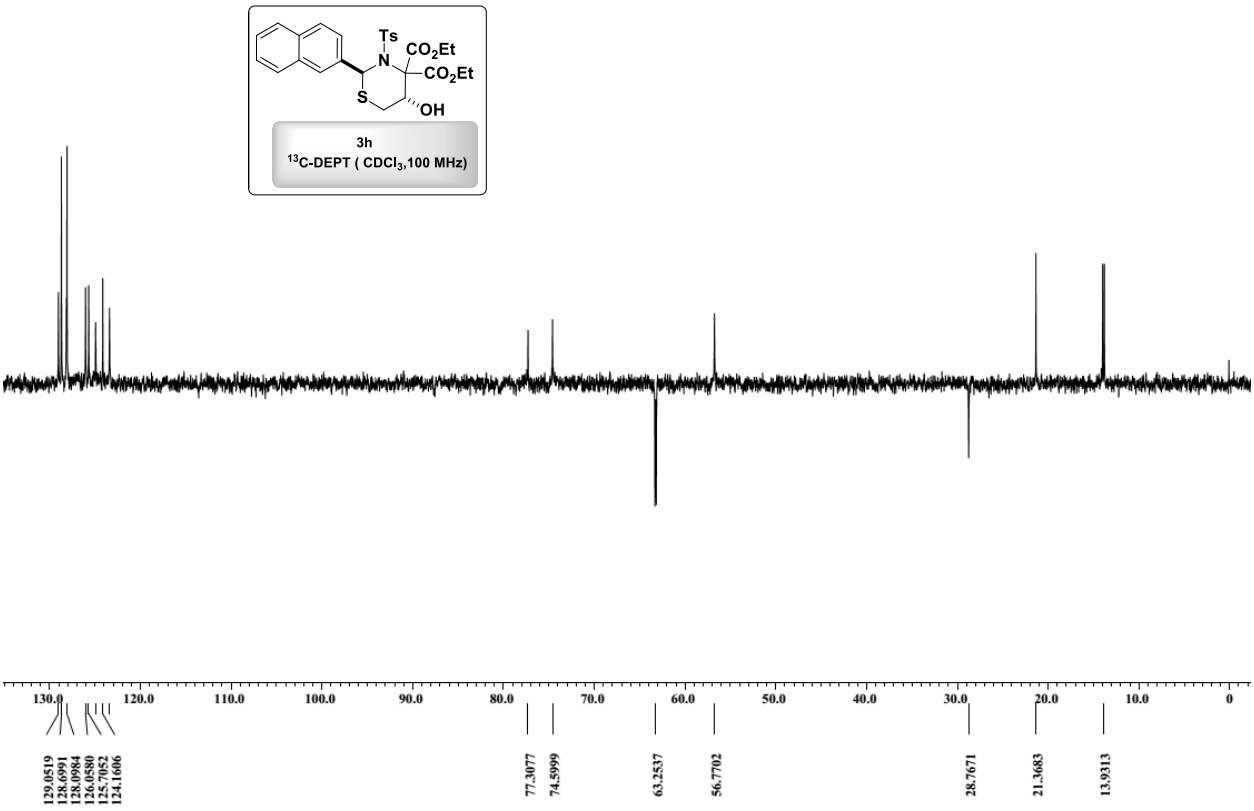

Ts
N
CO2Et
CO2Et
S
OH
3g
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
128.4703
127.7361
127.1545
126.6492
73.8943
63.1298
59.1634
28.1093
21.5018
13.8551

HRMS of 3g:

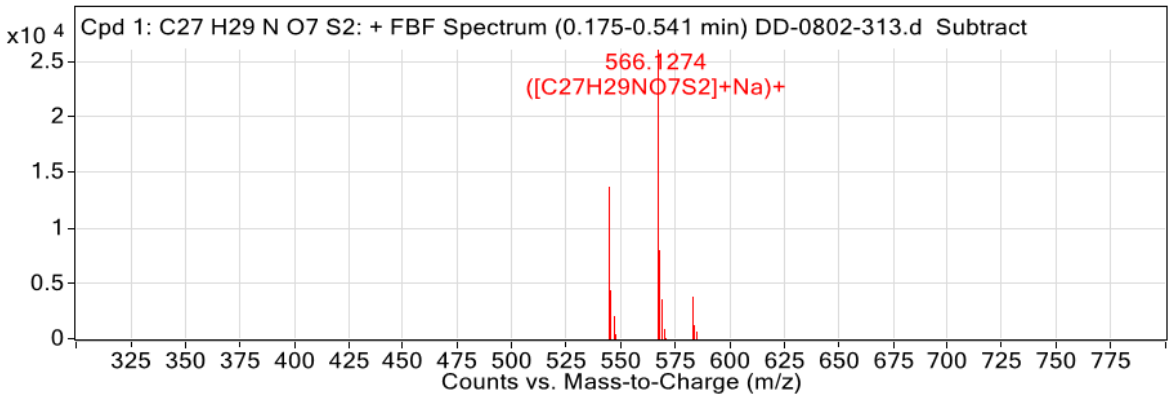

x10 4
Cpd 1: C23 H27 N O7 S2: + FBF Spectrum (0.174-0.507 min) DD-0802-358.d Subtract
516.1117
([C23H27NO7S2]+Na)+
3.5
3
2.5
2
1.5
1
0.5
0
150
200
250
300
350
400
450
500
550
600
650
700
750
Counts vs. Mass-to-Charge (m/z)

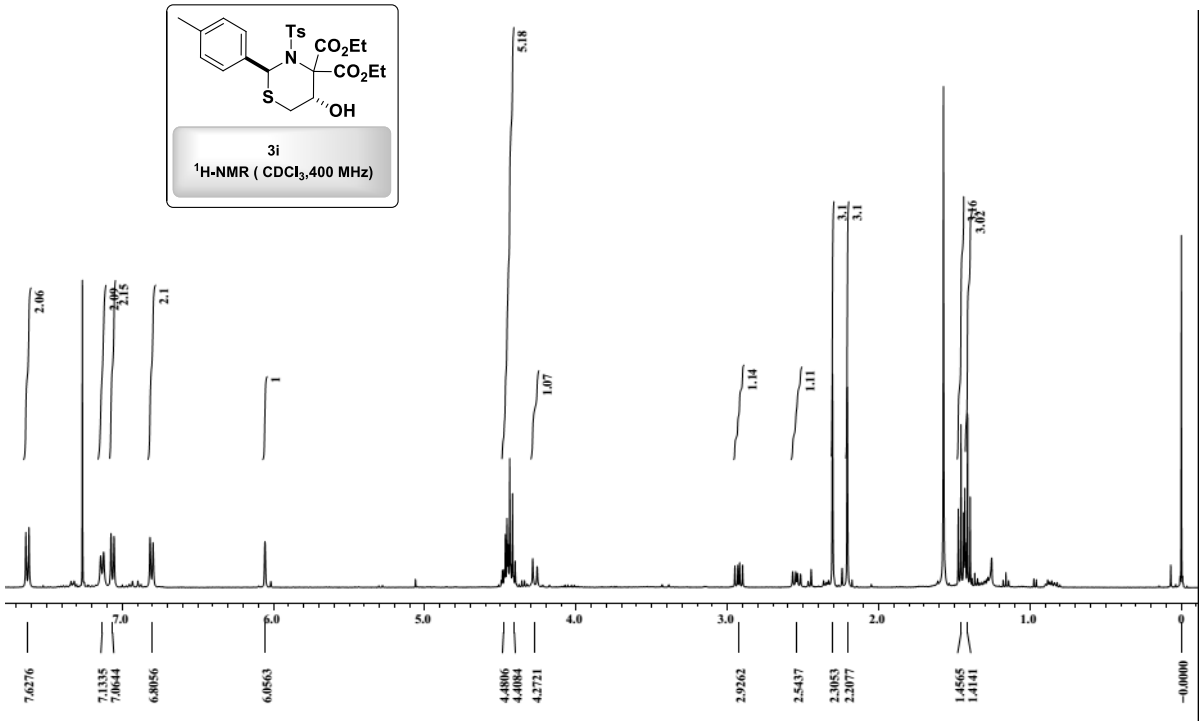

Ts
N
CO2Et
CO2Et
S
OH
3h
1H-NMR (CDCl3,400 MHz)
0.98
1.01
3.11
1.85
1.89
1.91
1
1.0
4.02
0.99
0.97
2.86
3.2
3.25
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0424
7.7846
7.5109
7.3738
6.8387
6.7626
4.8533
4.6036
4.5715
4.4707
2.9369
2.4993
2.1626
1.5406
1.4419

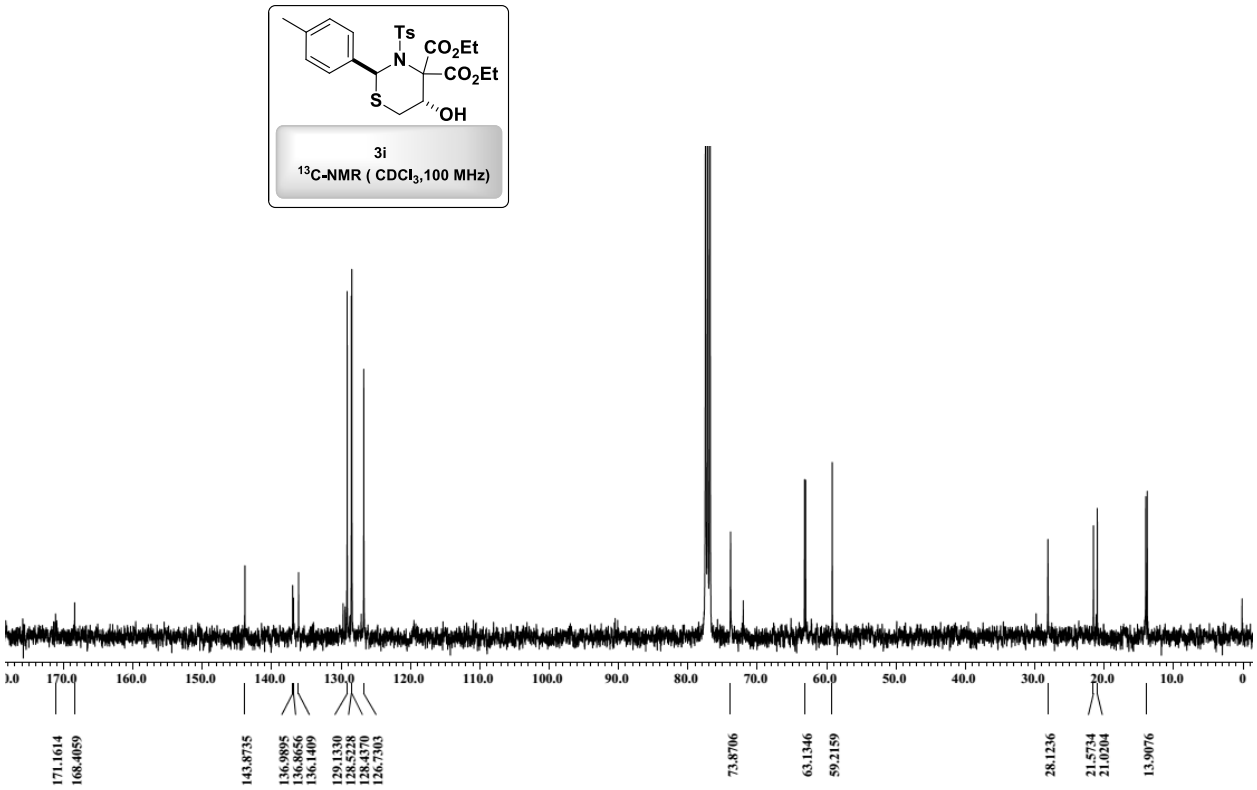

Ts
N
CO2Et
CO2Et
S
OH
3h
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
171.9433
168.5108
143.6637
136.8751
133.9194
132.7085
129.3237
129.1235
128.7993
128.1700
126.1201
125.7673
125.0141
124.2227
123.4695
74.6619
63.3063
56.8227
28.8197
21.4018
13.9648

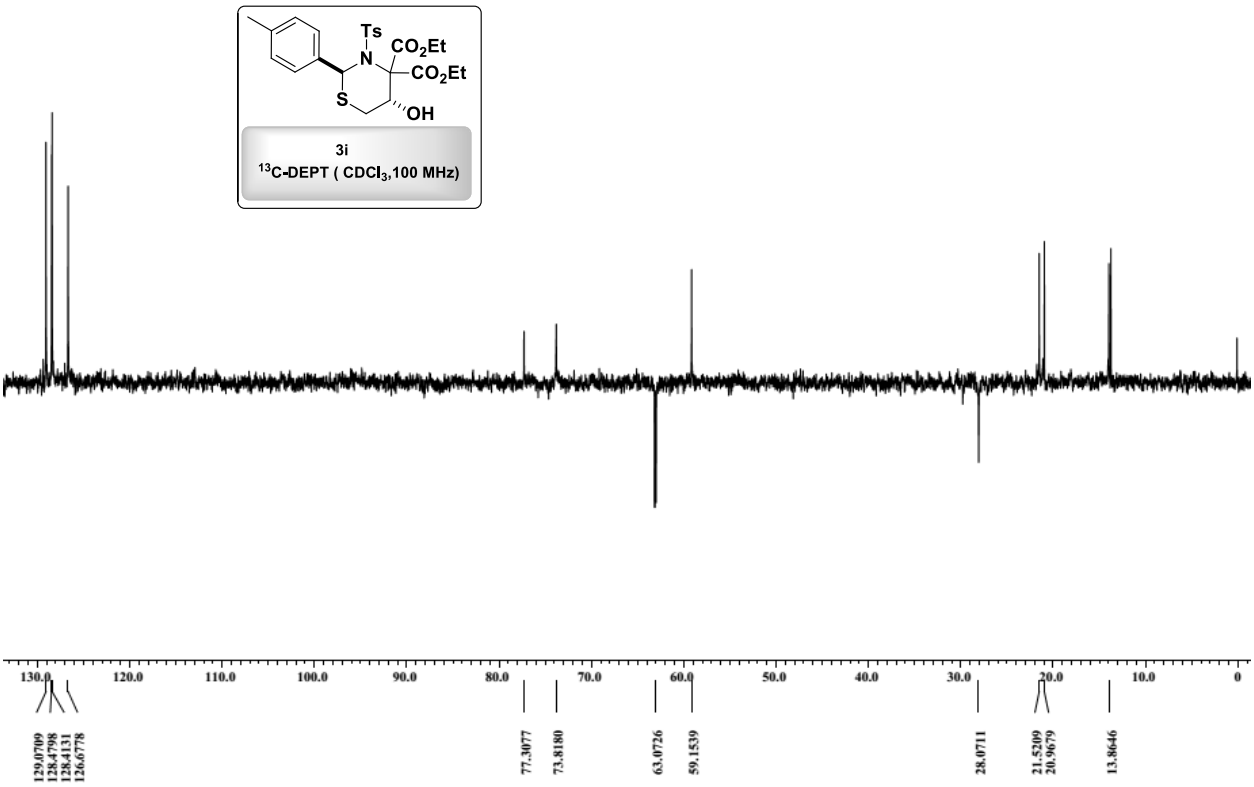

Ts
N
CO2Et
CO2Et
S
OH
3h
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.0519
128.6991
128.0984
126.0580
125.7052
124.1606
77.3077
74.5999
63.2537
56.7702
28.7671
21.3683
13.9313

HRMS of 3h:

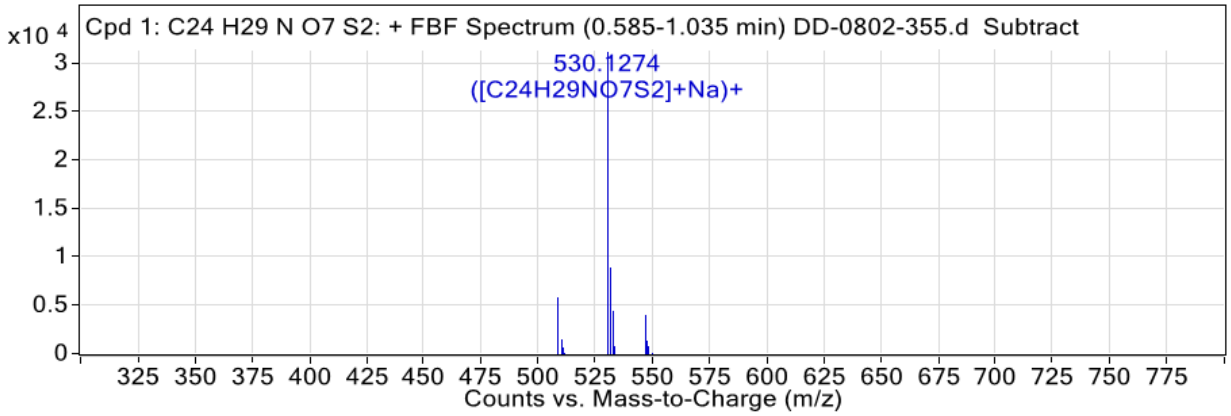

Cpd 1: C27 H29 N O7 S2: + FBF Spectrum (0.175-0.541 min) DD-0802-313.d Subtract
x10 4
2.5
2
1.5
1
0.5
0
566.1274
([C27H29NO7S2]+Na)+
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

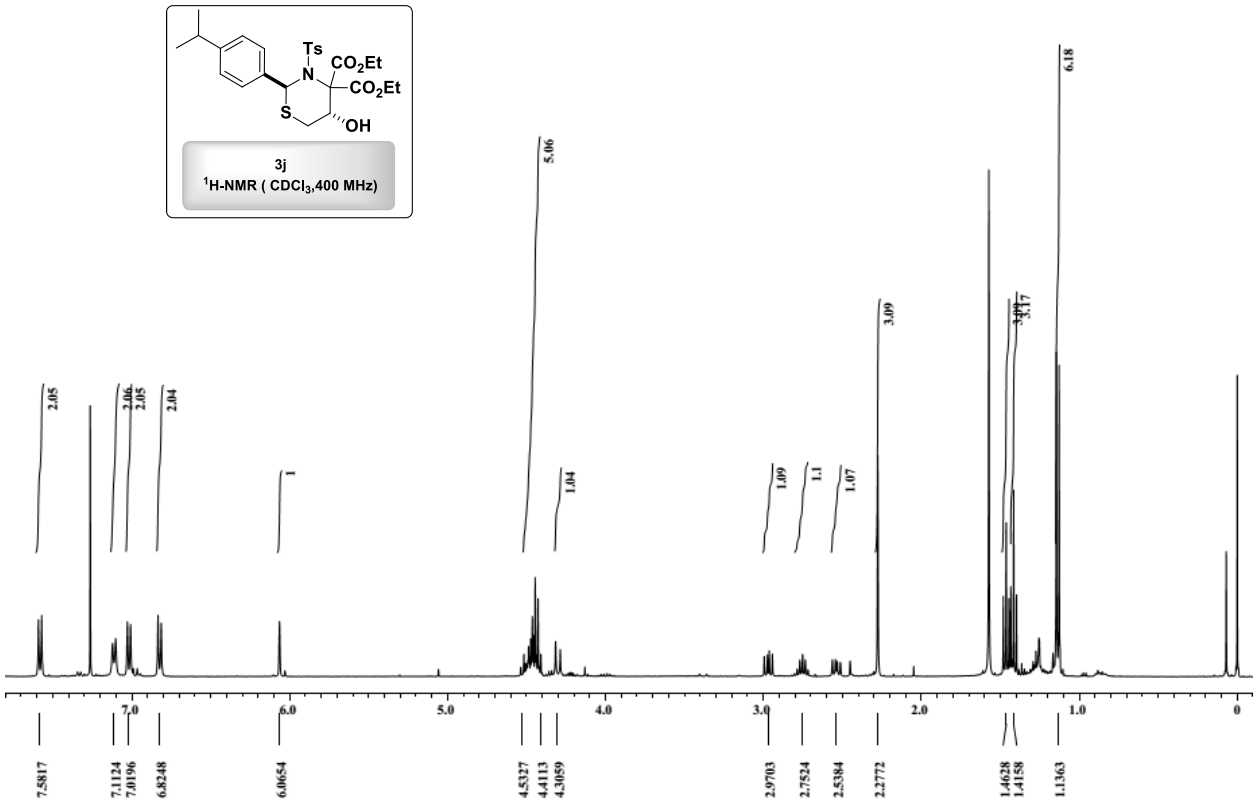

Ts
N
CO2Et
CO2Et
S
OH
3i
1H-NMR (CDCl3,400 MHz)
5.18
2.06
2.09
2.15
2.1
1
1.07
1.14
1.11
3.1
3.1
3.16
3.02
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.6276
7.1335
7.0644
6.8056
6.0563
4.4806
4.4084
4.2721
2.9262
2.5437
2.3053
2.2077
1.4565
1.4141
-0.0000

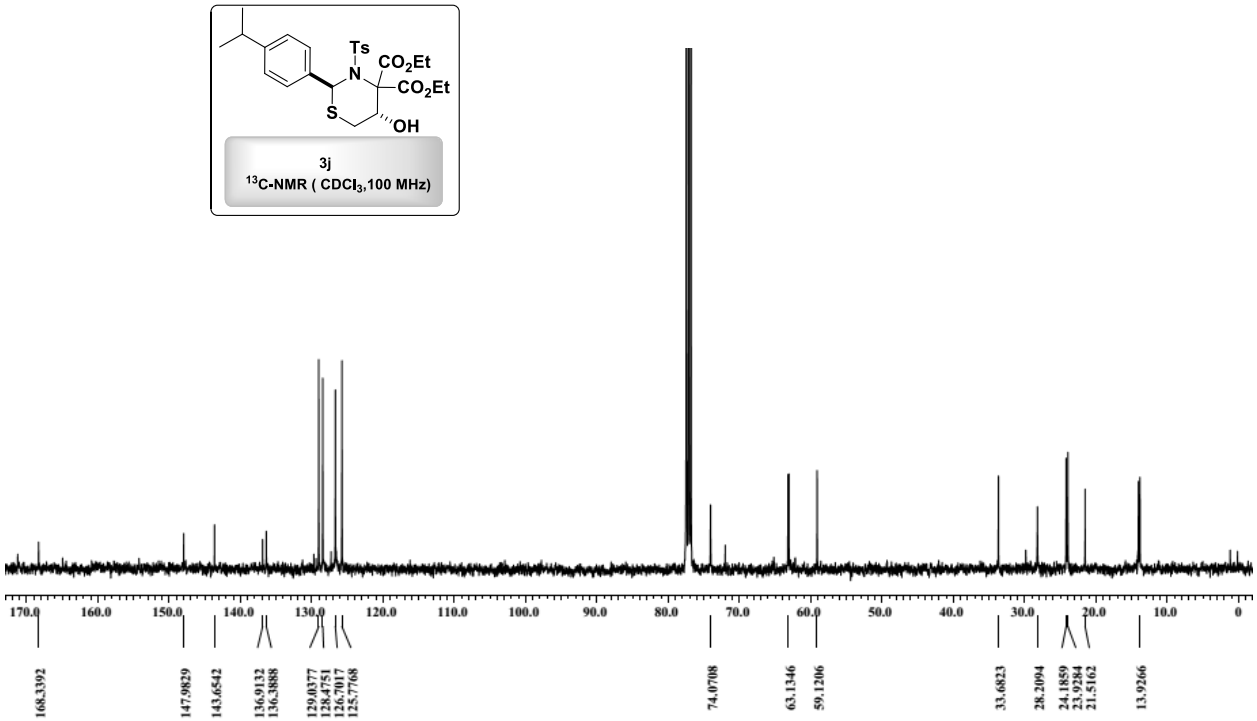

Ts
N
CO2Et
CO2Et
S
OH
3i
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
171.1614
168.4059
143.8735
136.9895
136.8656
136.1409
129.1330
128.5228
128.4370
126.7303
73.8706
63.1346
59.2159
28.1236
21.5734
21.0204
13.9076

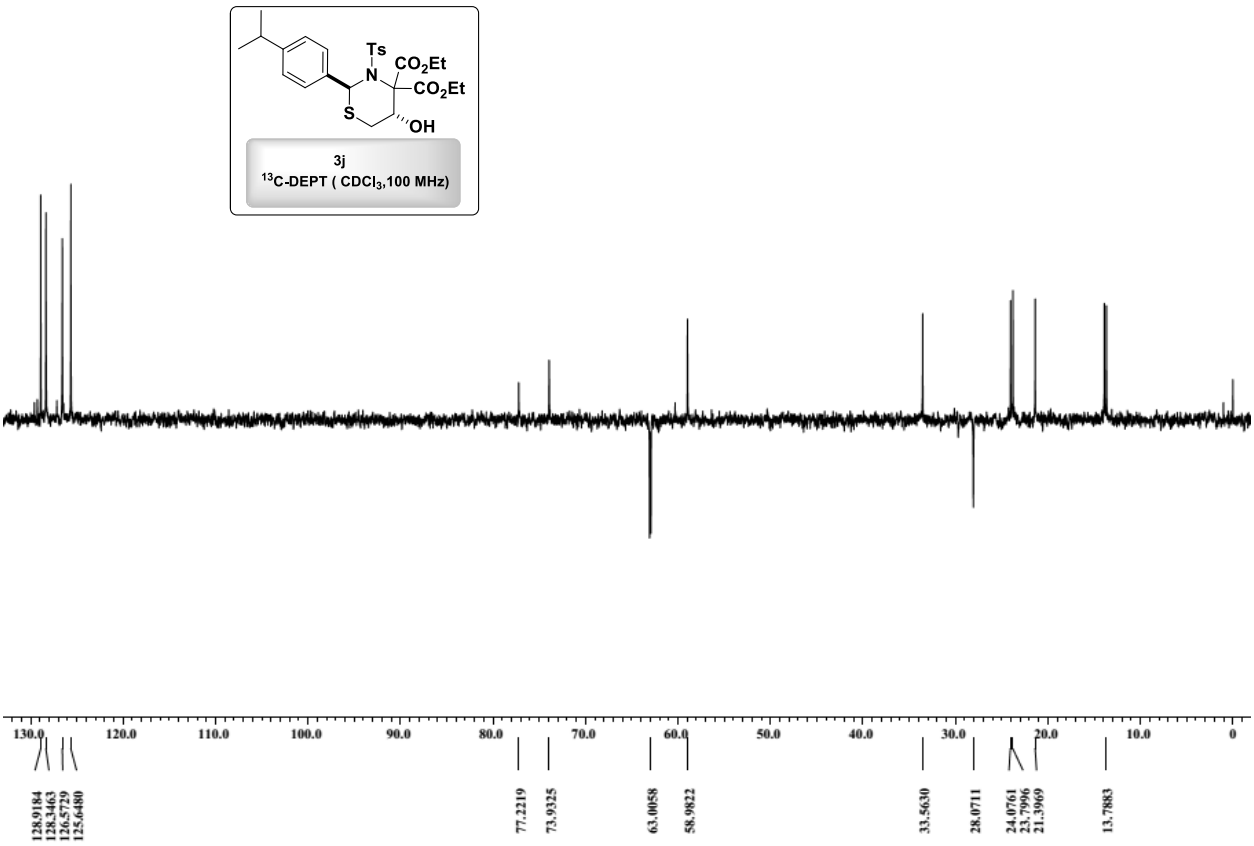

Ts
N
CO2Et
CO2Et
S
OH
3i
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.0709
128.4798
128.4131
126.6778
77.3077
73.8180
63.0726
59.1539
28.0711
21.5209
20.9679
13.8646

HRMS of 3i:

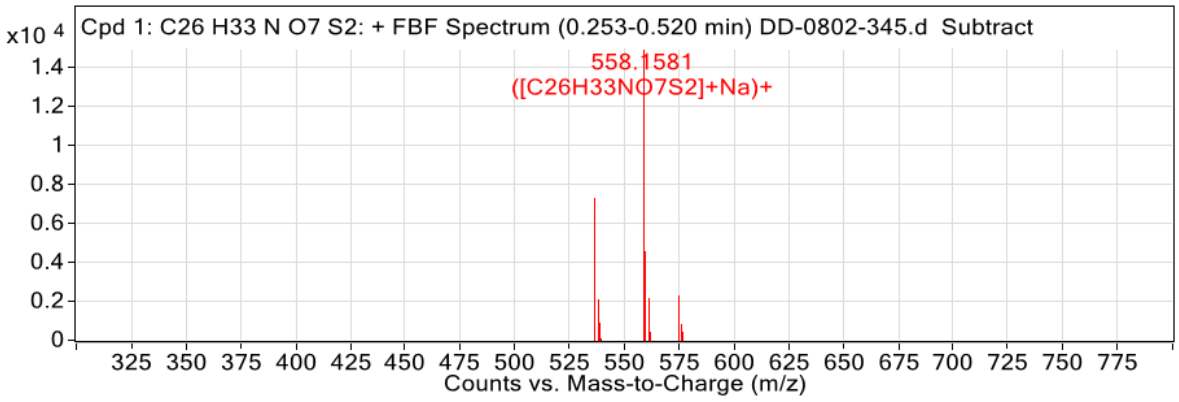

Cpd 1: C24 H29 N O7 S2: + FBF Spectrum (0.585-1.035 min) DD-0802-355.d Subtract
x10 4
3
2.5
2
1.5
1
0.5
0
530.1274
([C24H29NO7S2]+Na)+
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

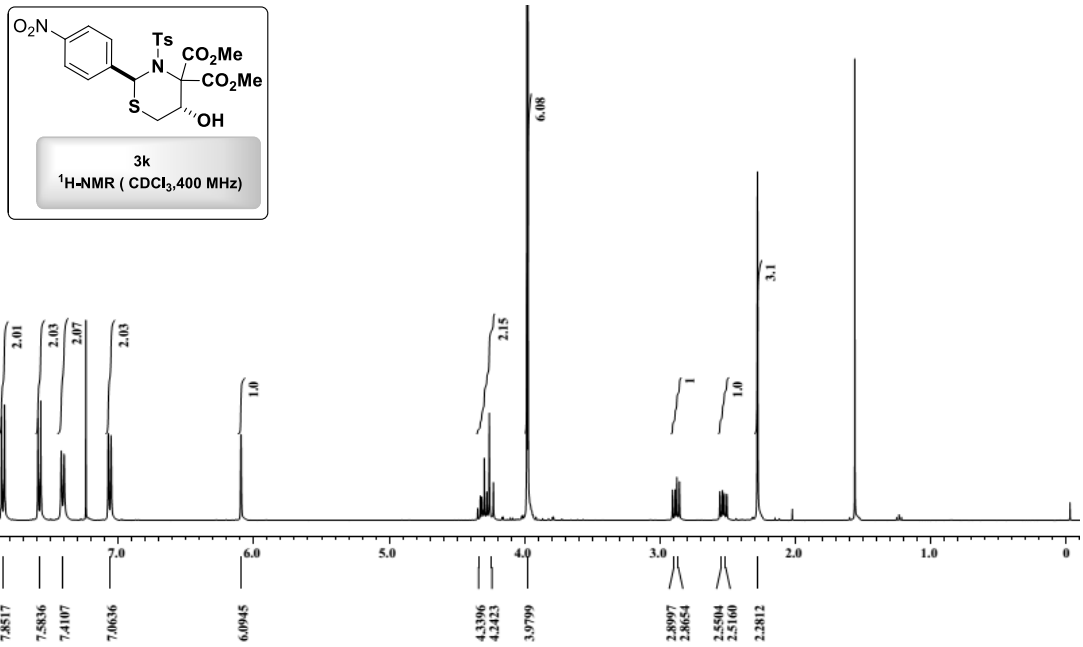

Ts
N
CO2Et
CO2Et
S
OH
3j
1H-NMR (CDCl3,400 MHz)
2.05
2.06
2.05
2.04
1
5.06
1.04
1.09
1.1
1.07
3.09
6.18
7.5817
7.1124
7.0196
6.8248
6.0654
4.5327
4.4113
4.3059
2.9703
2.7524
2.5384
2.2772
1.4628
1.4158
1.1363

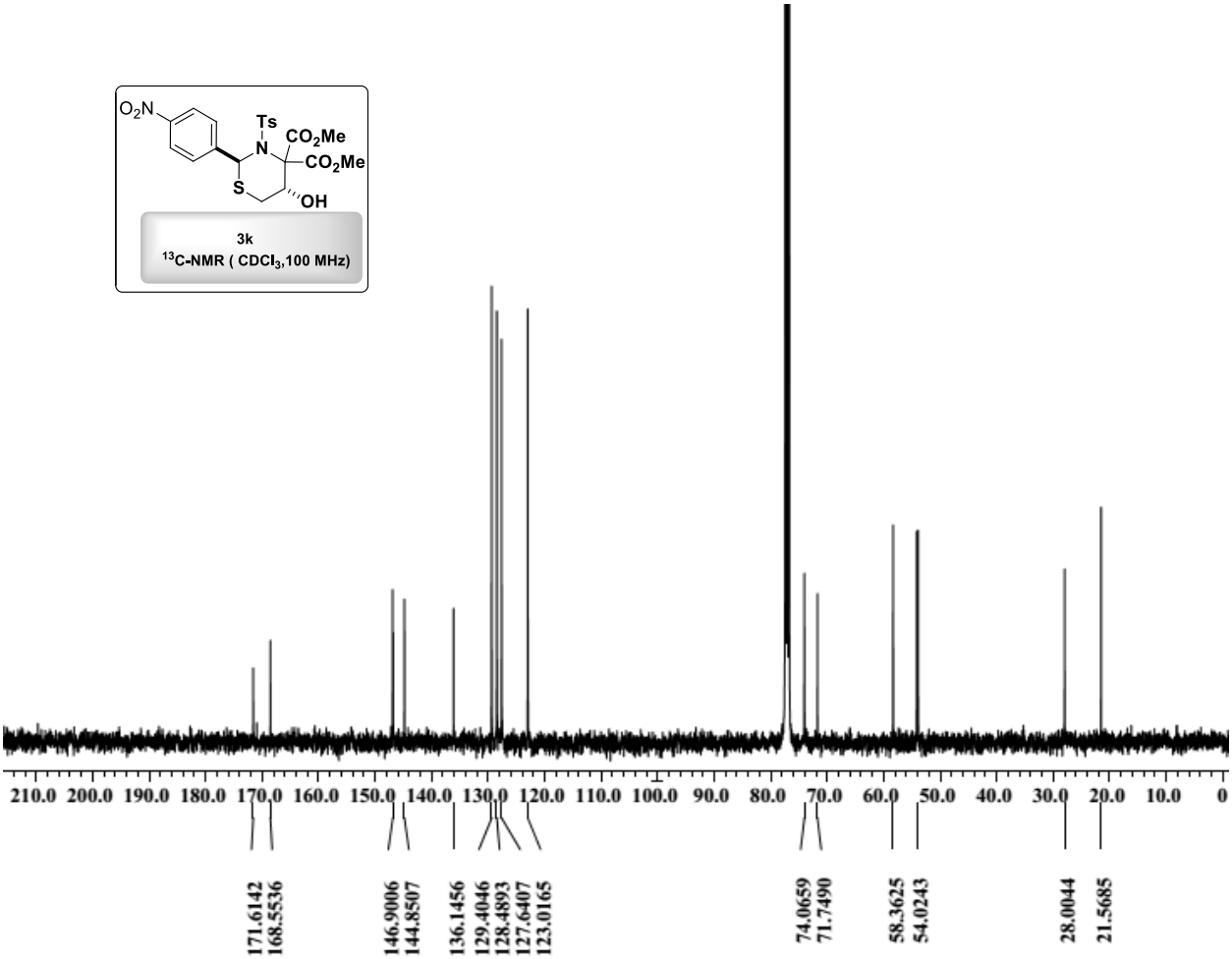

Ts
N
CO2Et
CO2Et
S
OH
3j
13C-NMR (CDCl3,100 MHz)
168.3392
147.9829
143.6542
136.9132
136.3888
129.0377
128.4751
126.7017
125.7768
74.0708
63.1346
59.1206
33.6823
28.2094
24.1859
23.9284
21.5162
13.9266


Ts
N
CO2Et
CO2Et
S
OH
3j
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
128.9184
128.3463
126.5729
125.6480
77.2219
73.9325
63.0058
58.9822
33.5630
28.0711
24.0761
23.7996
21.3969
13.7883

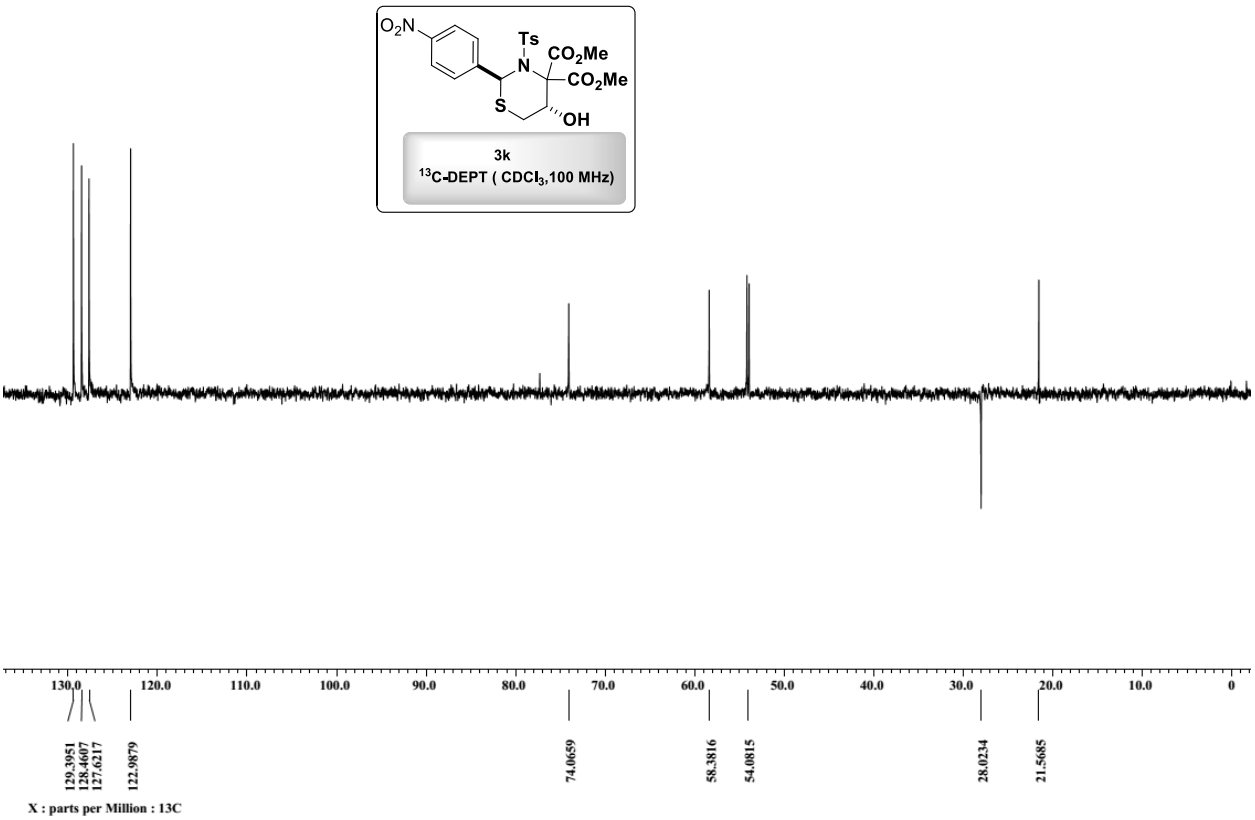
HRMS of 3j:


Cpd 1: C26 H33 N O7 S2: + FBF Spectrum (0.253-0.520 min) DD-0802-345.d Subtract
x10 4
1.4
1.2
1
0.8
0.6
0.4
0.2
0
558.1581
([C26H33NO7S2]+Na)+
325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775
Counts vs. Mass-to-Charge (m/z)

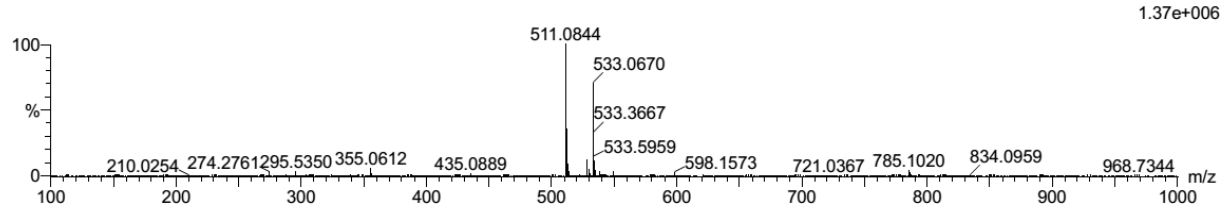


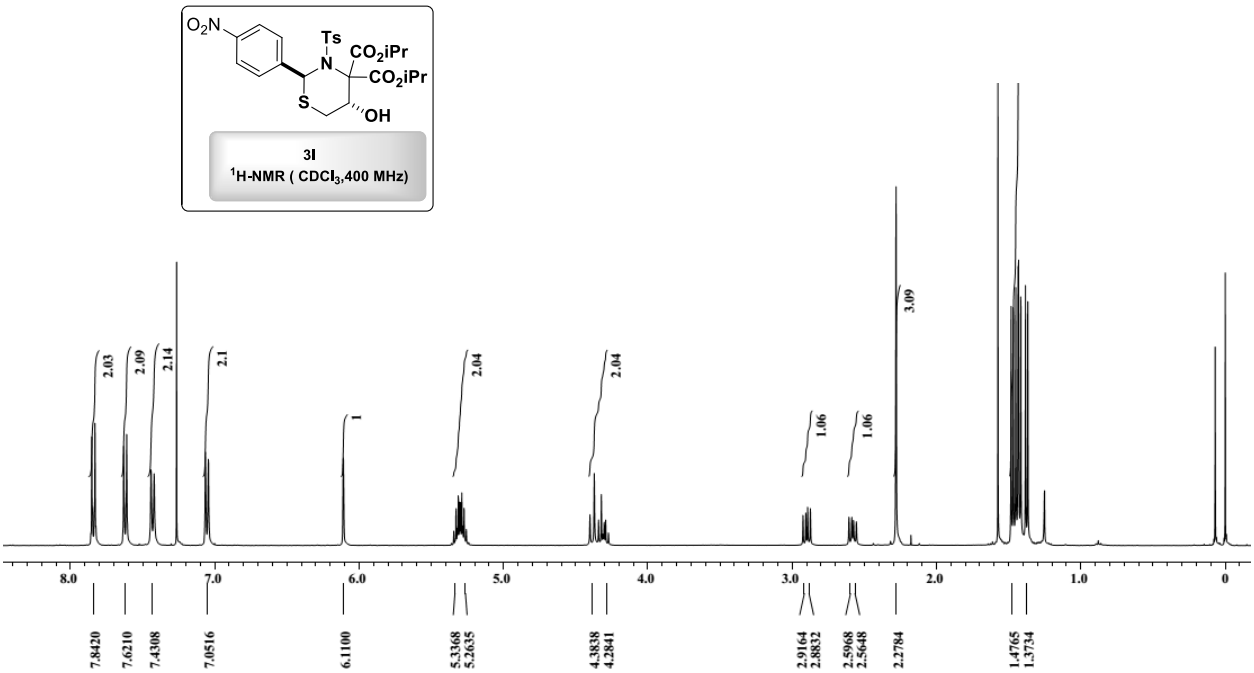


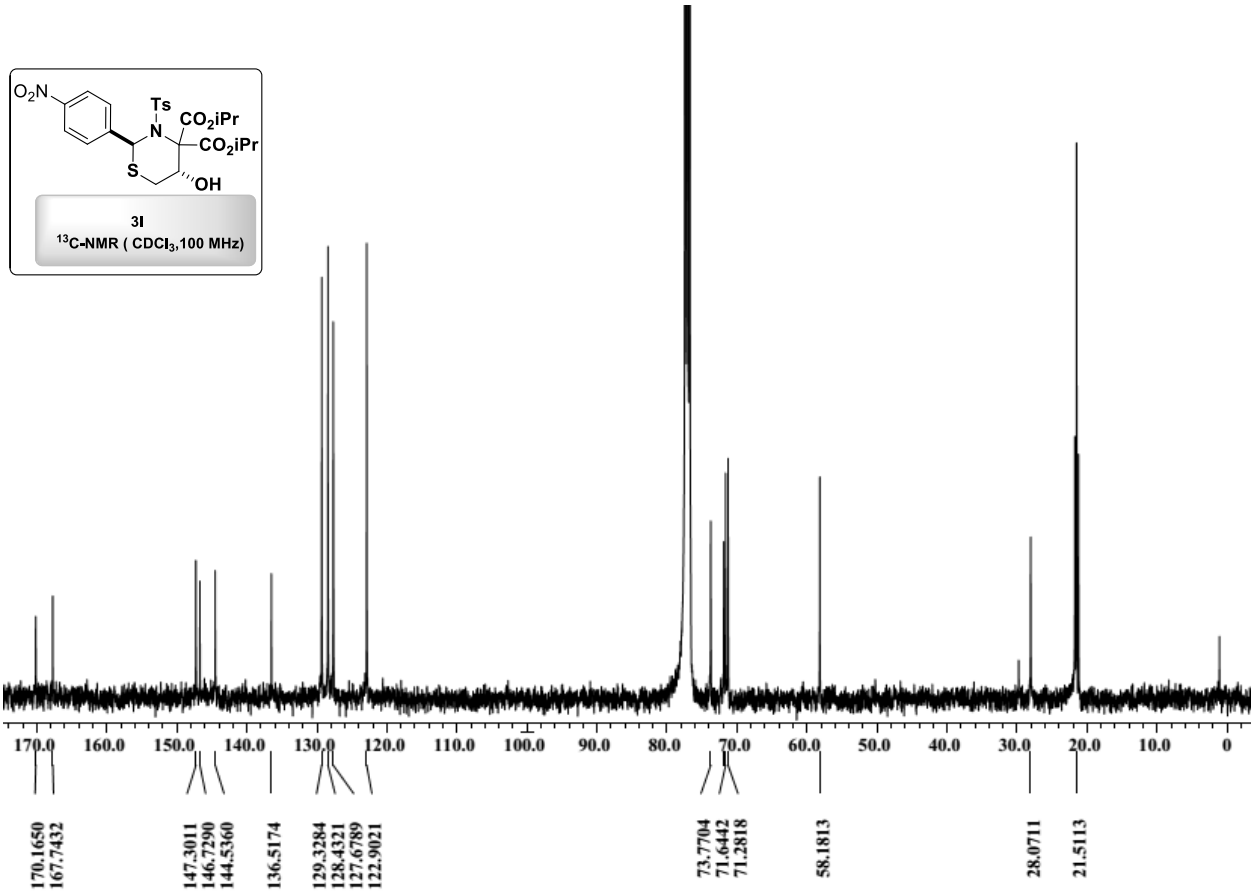
X : parts per Million : 13C

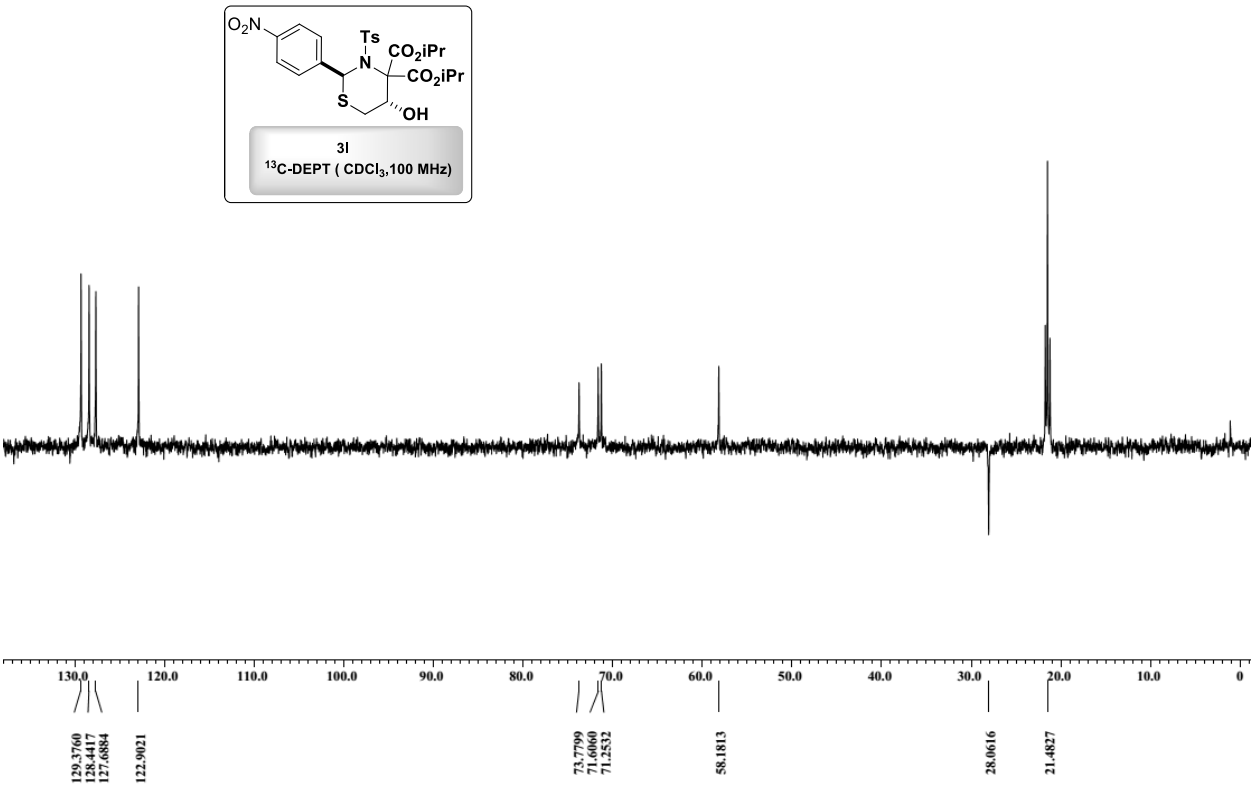

O2N
Ts
N
CO2Me
CO2Me
S
OH
3k
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.3951
128.4607
127.6217
122.9879
74.0659
58.3816
54.0815
28.0234
21.5685
X : parts per Million : 13C

HRMS of 3k:

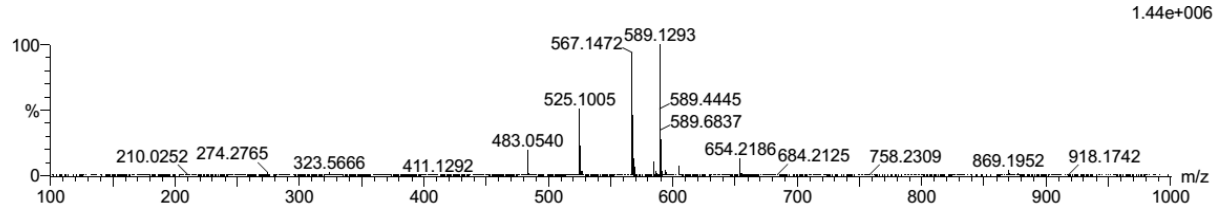

1.37e+006
100
%
0
511.0844
533.0670
533.3667
533.5959
210.0254
274.2761
295.5350
355.0612
435.0889
598.1573
721.0367
785.1020
834.0959
968.7344
m/z
100
200
300
400
500
600
700
800
900
1000

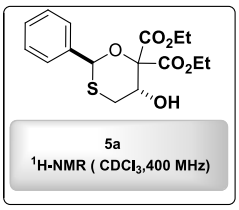

3l
^{1}H-NMR ($CDCl_3$,400 MHz)
2.03
2.09
2.14
2.1
1
2.04
2.04
1.06
1.06
3.09
7.8420
7.6210
7.4308
7.0516
6.1100
5.3368
5.2635
4.3838
4.2841
2.9164
2.8832
2.5968
2.5648
2.2784
1.4765
1.3734

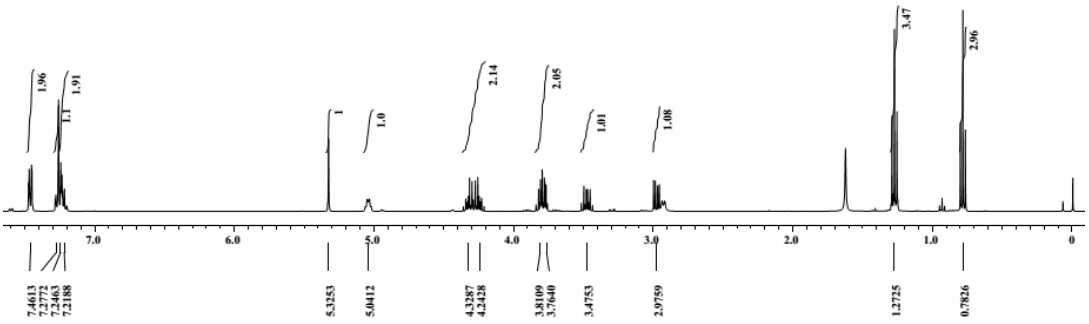

3l
^{13}C-NMR ($CDCl_3$,100 MHz)
170.1650
167.7432
147.3011
146.7290
144.5360
136.5174
129.3284
128.4321
127.6789
122.9021
73.7704
71.6442
71.2818
58.1813
28.0711
21.5113


O2N
Ts
N
CO2iPr
CO2iPr
S
OH
3l
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.3760
128.4417
127.6884
122.9021
73.7799
71.6060
71.2532
58.1813
28.0616
21.4827

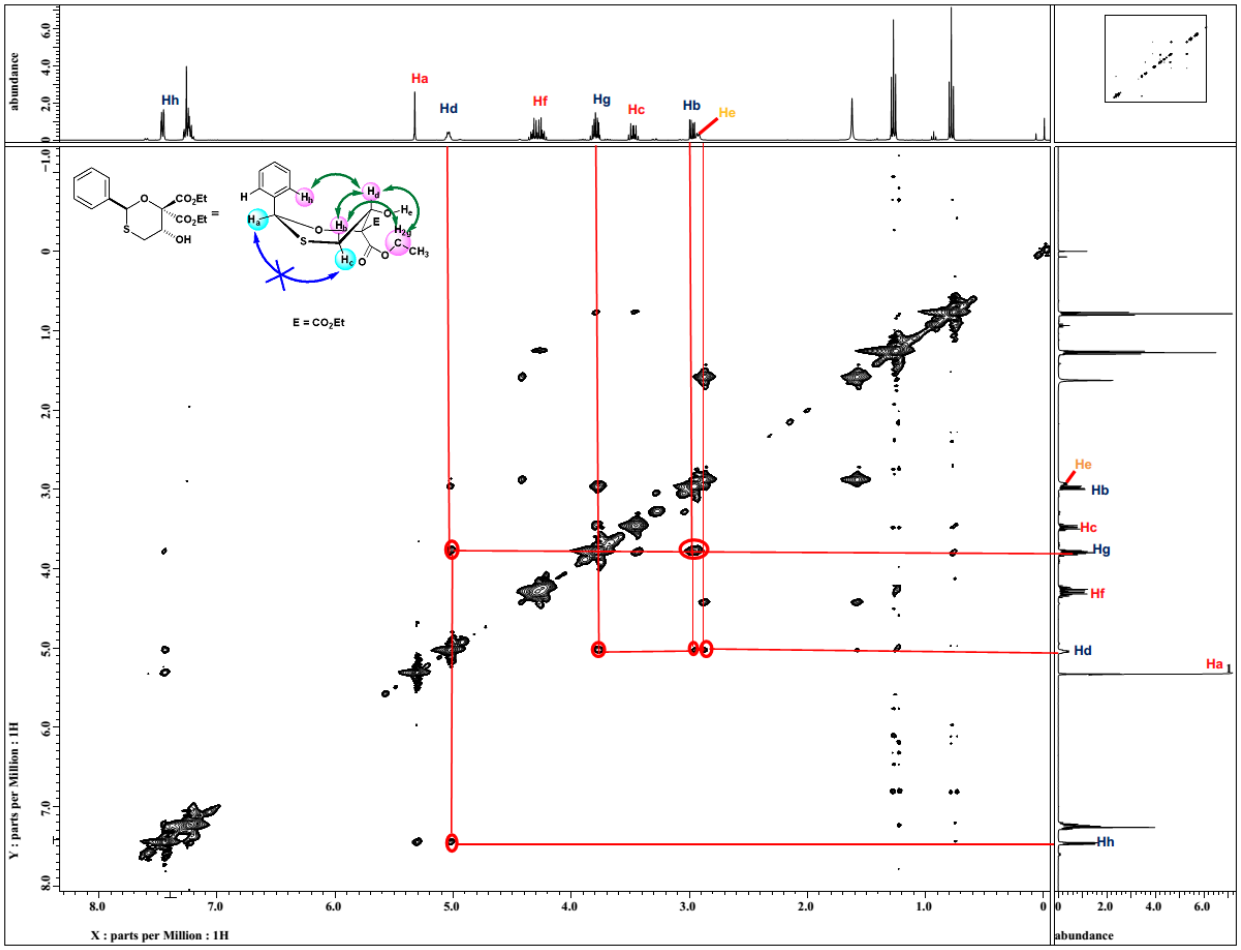
HRMS of 3l:


1.44e+006
100
%
0
567.1472
589.1293
525.1005
589.4445
589.6837
483.0540
654.2186
684.2125
758.2309
869.1952
918.1742
210.0252
274.2765
323.5666
411.1292
m/z
100
200
300
400
500
600
700
800
900
1000

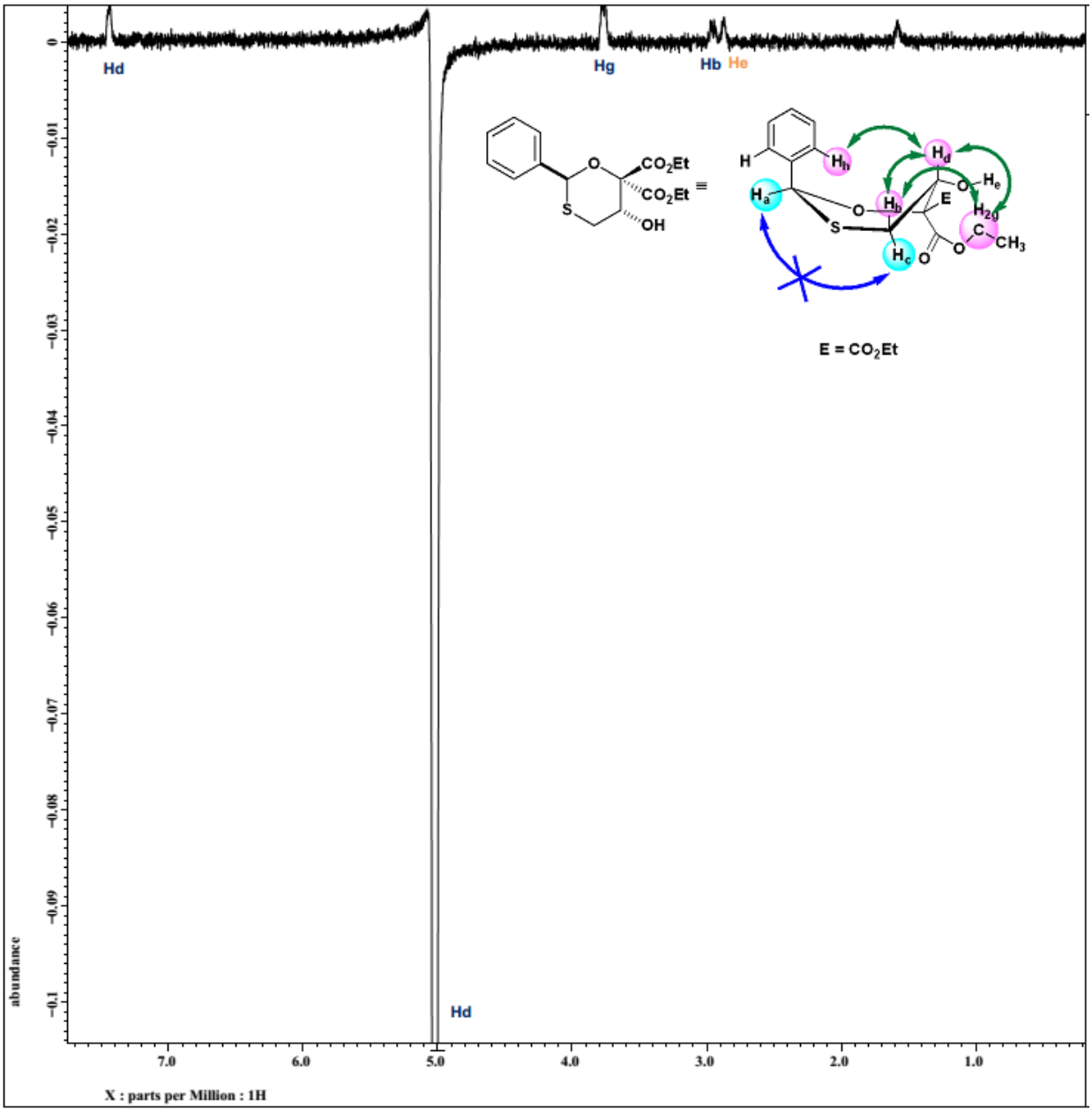


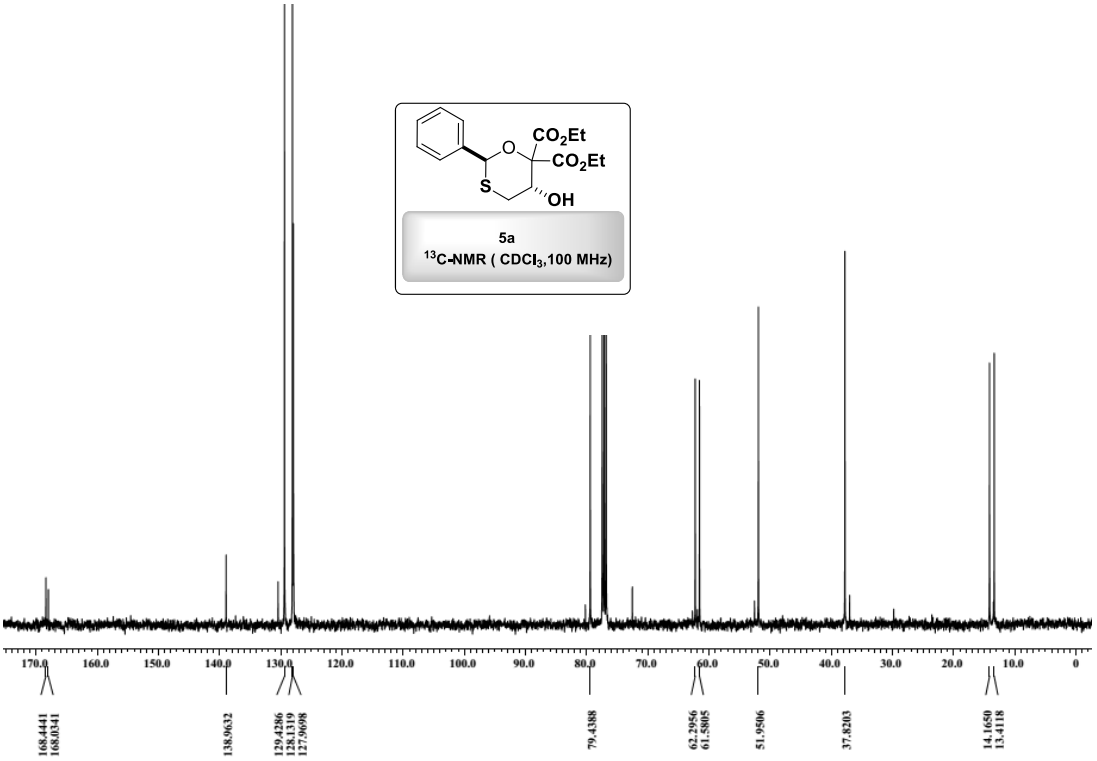


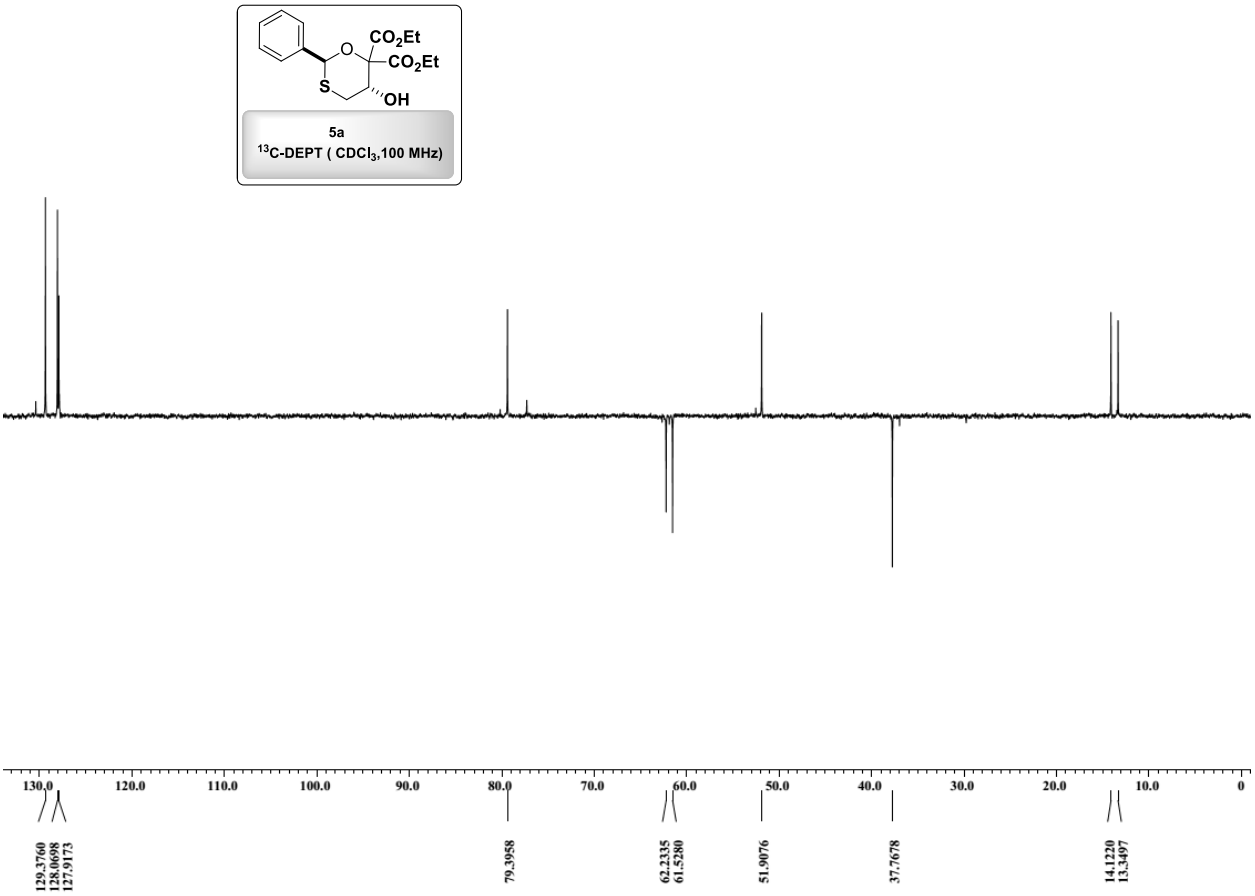
NOESY (400 MHz) of **5a**



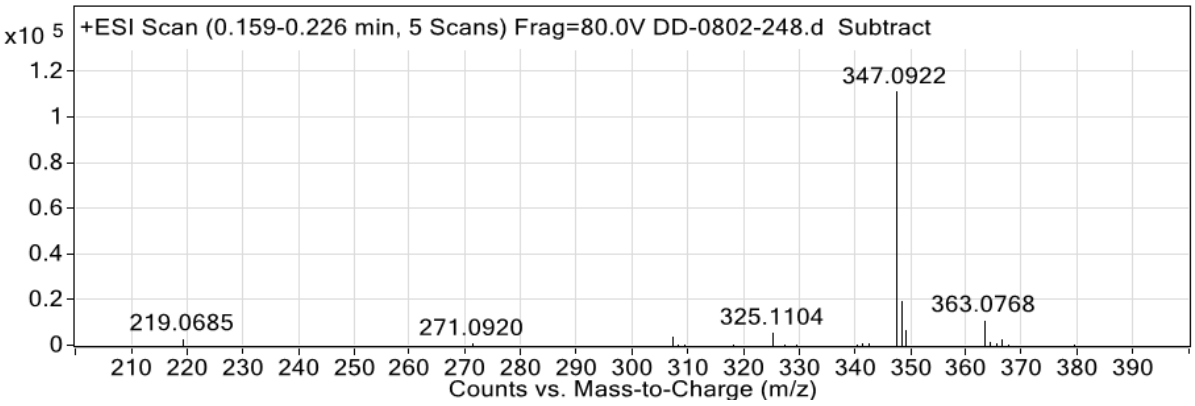
1D NOESY OF **5a**

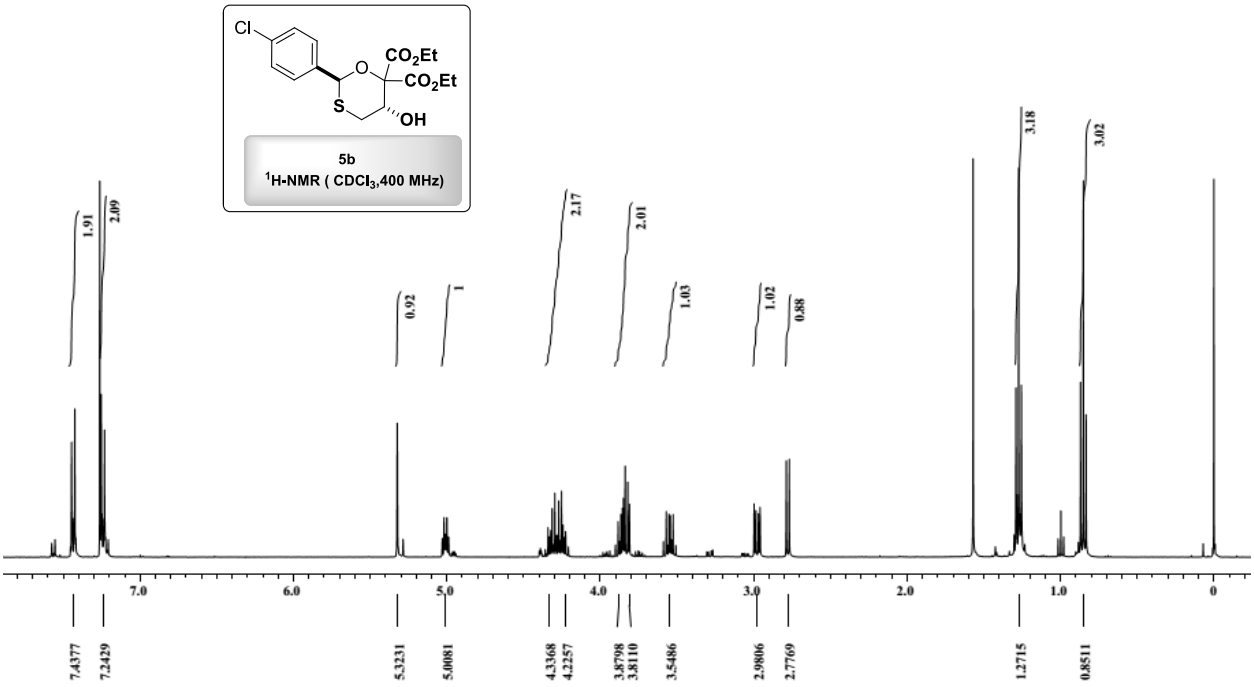


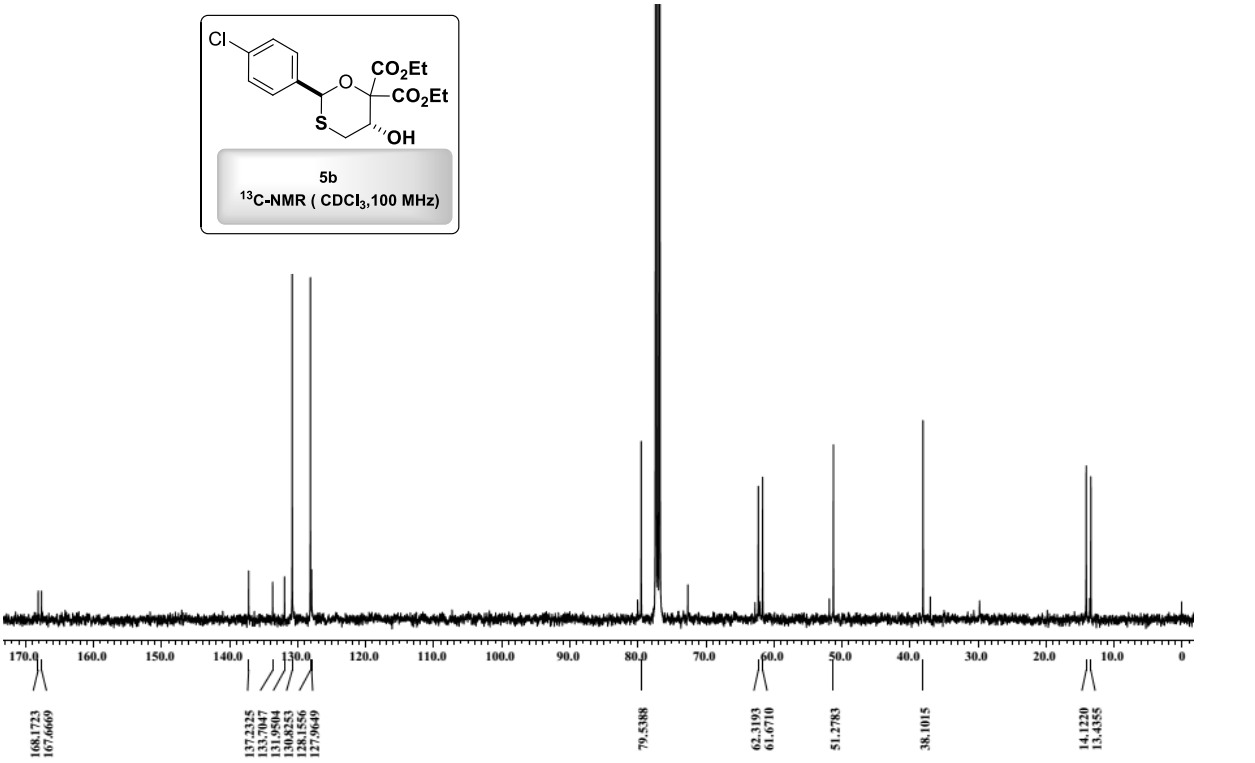

CO2Et
CO2Et
O
S
OH
5a
13C-NMR (CDCl3,100 MHz)
170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
168.4441
168.0341
138.9632
129.4286
128.1319
127.9698
79.4388
62.2956
61.5805
51.9506
37.8303
14.1650
13.4118

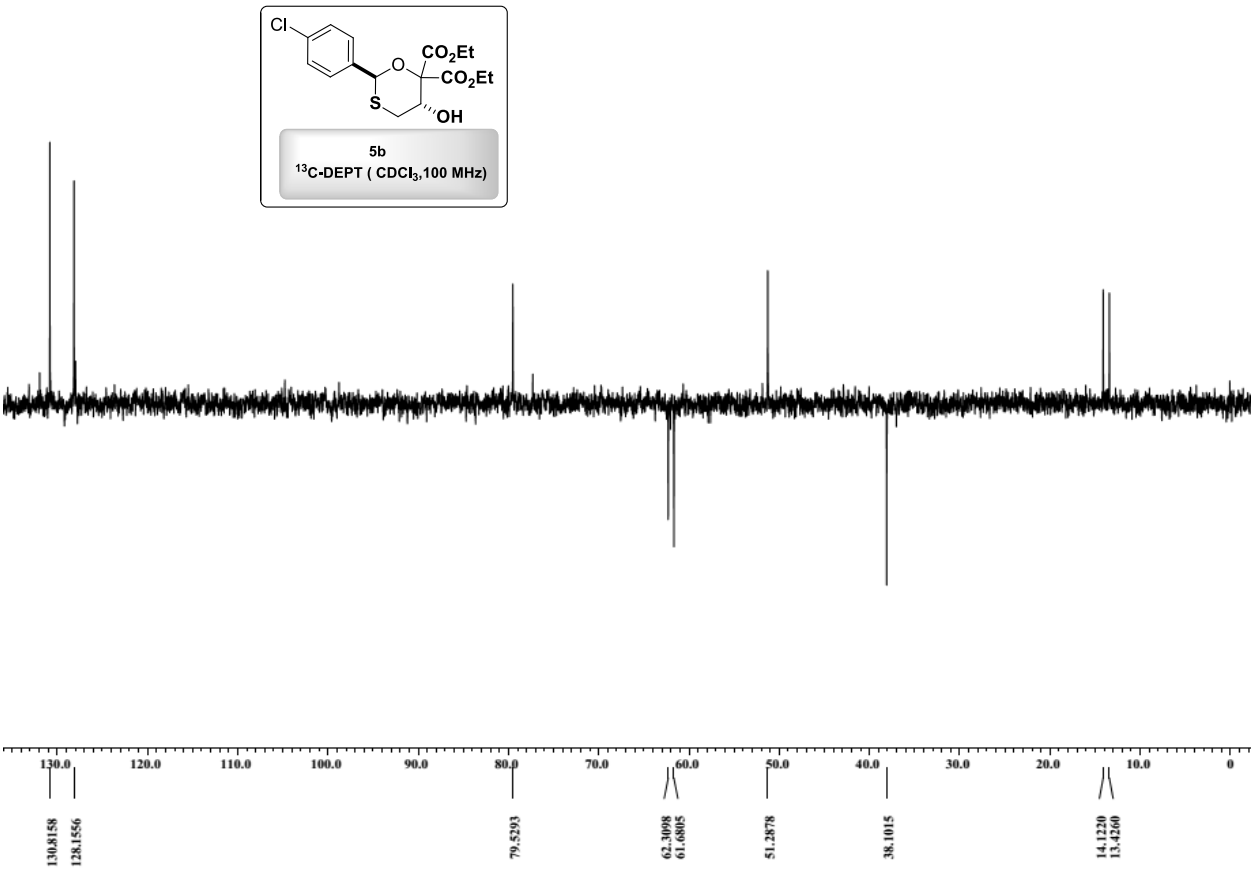

CO2Et
CO2Et
O
S
OH
5a
13C-DEPT (CDCl3,100 MHz)
130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
129.3760
128.0698
127.9173
79.3958
62.2335
61.5280
51.9076
37.7678
14.1220
13.3497

HRMS of 5a:

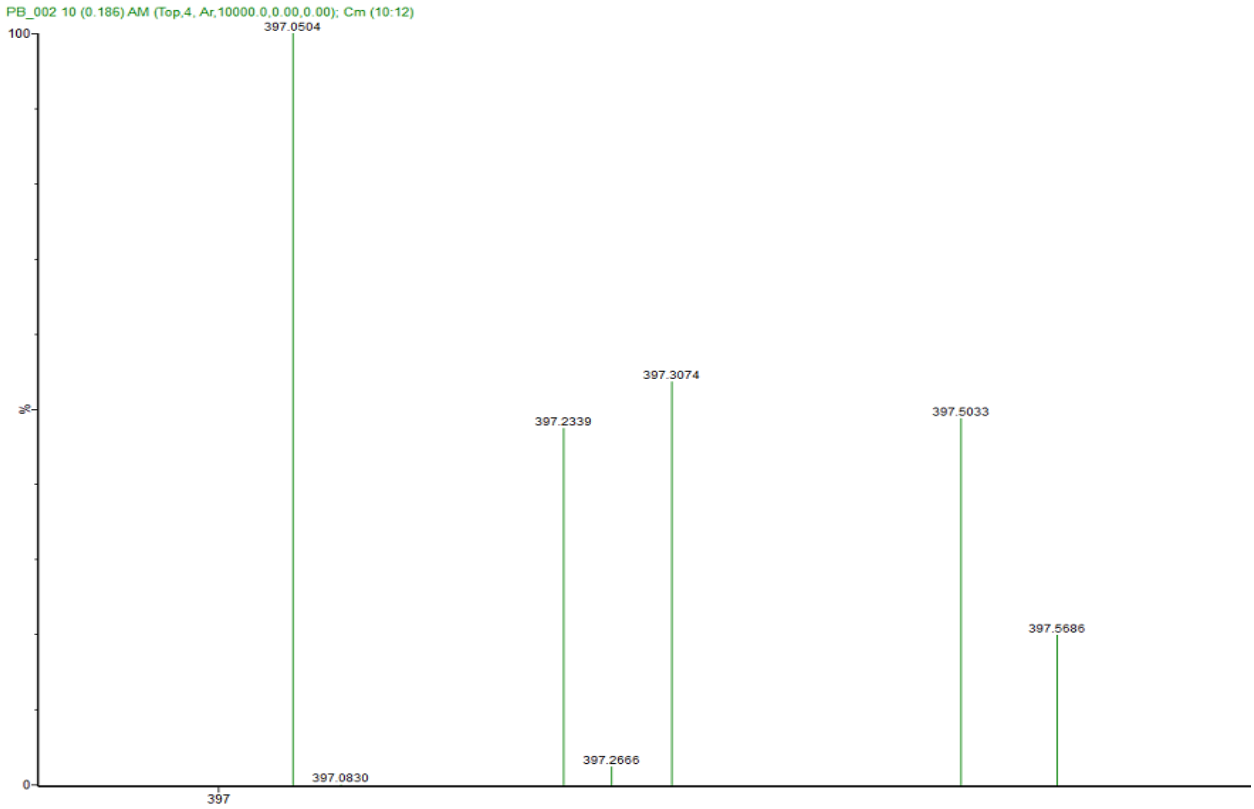

+ESI Scan (0.159-0.226 min, 5 Scans) Frag=80.0V DD-0802-248.d Subtract
x10 5
347.0922
363.0768
325.1104
219.0685
271.0920
Counts vs. Mass-to-Charge (m/z)

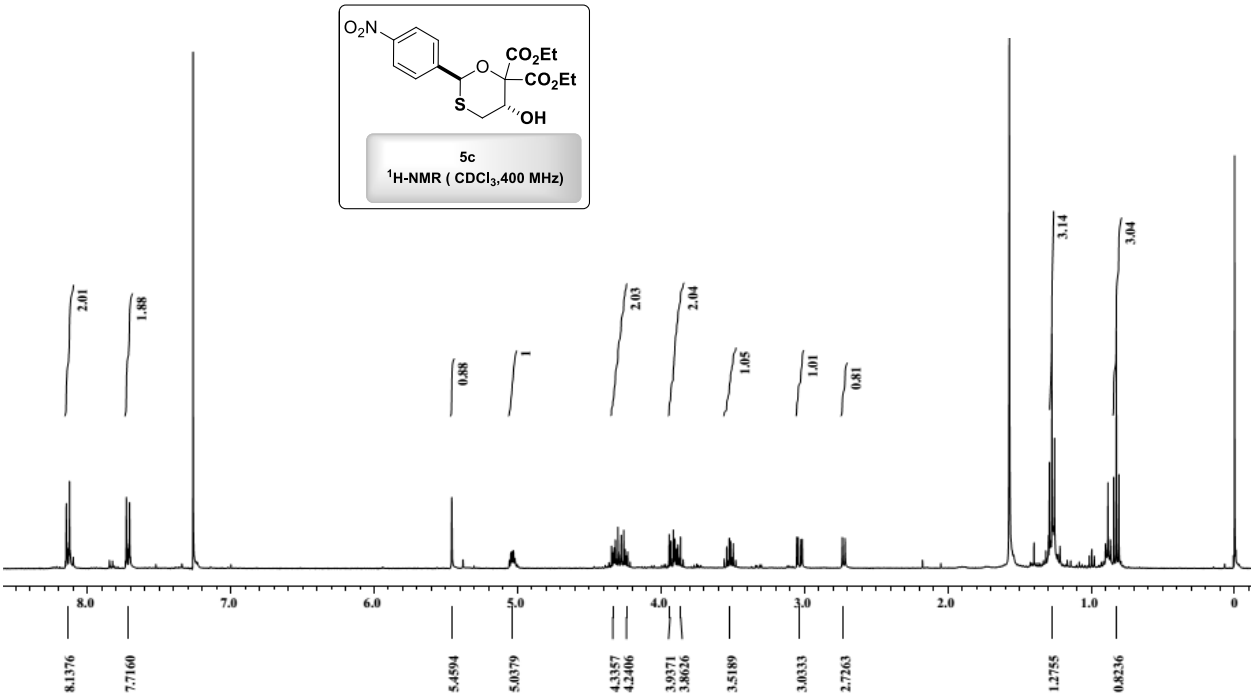

Cl
CO2Et
CO2Et
O
S
OH
5b
1H-NMR (CDCl3,400 MHz)
1.91
2.09
0.92
1
2.17
2.01
1.03
1.02
0.88
3.18
3.02
7.4377
7.2429
5.3231
5.0081
4.3368
4.2257
3.8798
3.8110
3.5486
2.9806
2.7769
1.2715
0.8511


Cl
CO2Et
CO2Et
O
S
OH
5b
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.1723
167.6669
137.2325
133.7047
131.9504
130.8253
128.1556
127.7649
79.5388
62.3193
61.6710
51.2783
38.1015
14.1220
13.4355

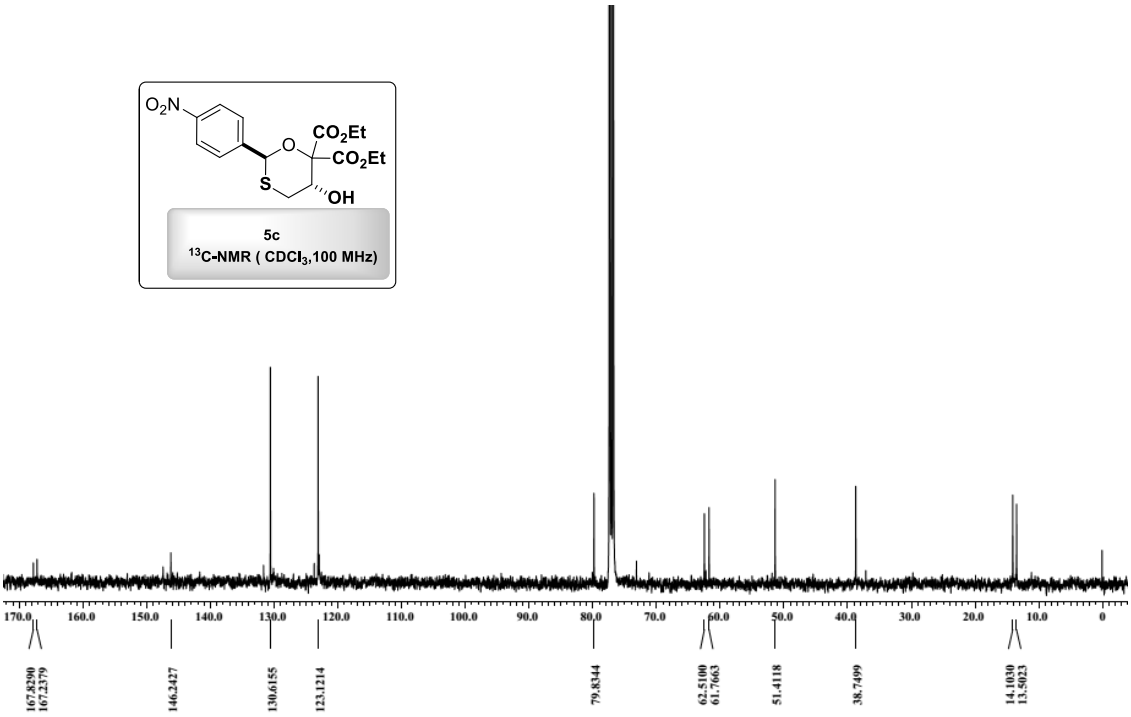

Cl
CO2Et
CO2Et
O
S
OH
5b
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
130.8158
128.1556
79.5293
62.3098
61.6805
51.2878
38.1015
14.1220
13.4260

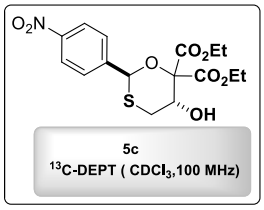
HRMS of 5b:


PB_002 10 (0.186) AM (Top,4, Ar,10000.0,0.00,0.00); Cm (10:12)
100
%
0
397
397.0504
397.0830
397.2339
397.2666
397.3074
397.5033
397.5686

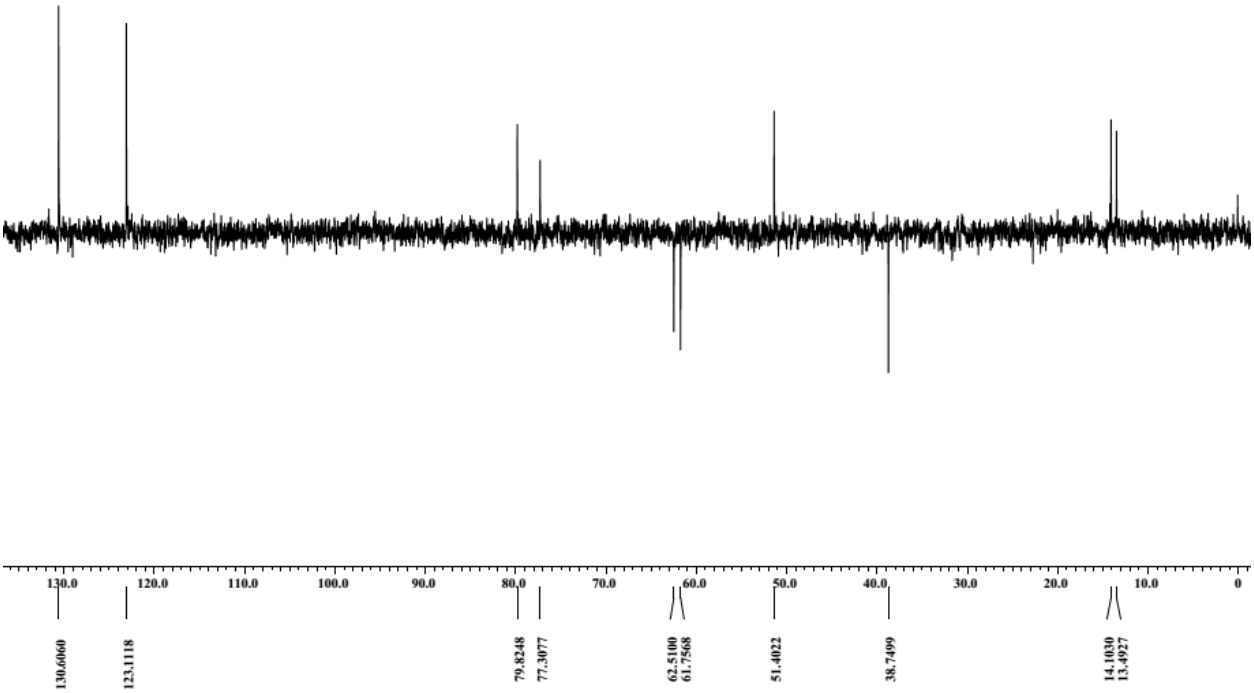

O2N
CO2Et
CO2Et
O
S
OH
5c
1H-NMR (CDCl3,400 MHz)
2.01
1.88
0.88
1
2.03
2.04
1.05
1.01
0.81
3.14
3.04
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.1376
7.7160
5.4594
5.0379
4.3357
4.2406
3.9371
3.8626
3.5189
3.0333
2.7263
1.2755
0.8236

5c
^{13}C-NMR ($CDCl_3$,100 MHz)

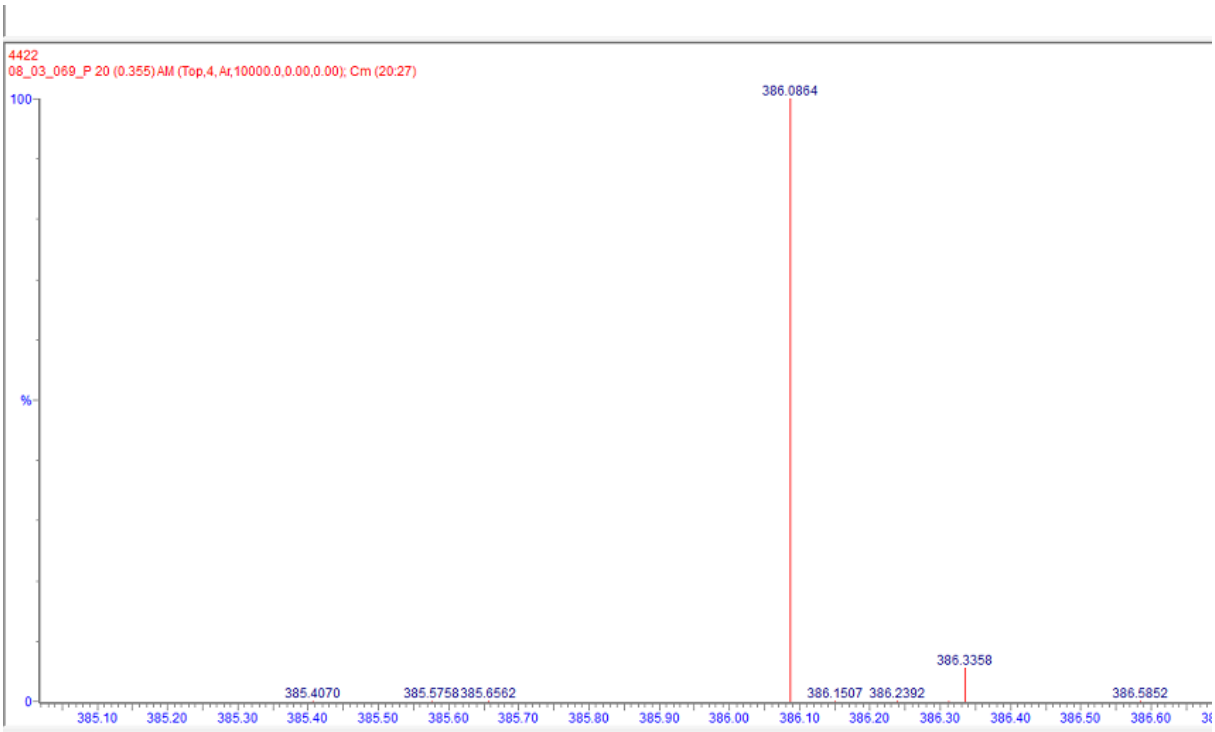


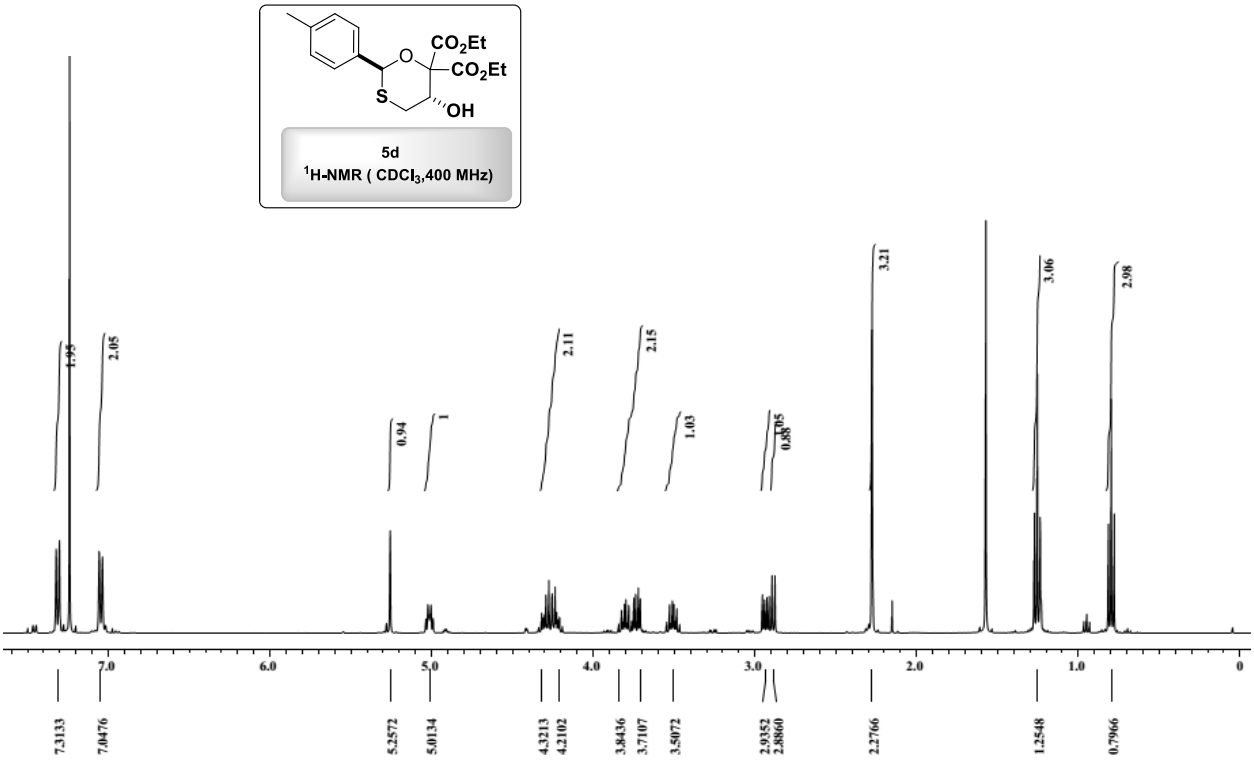


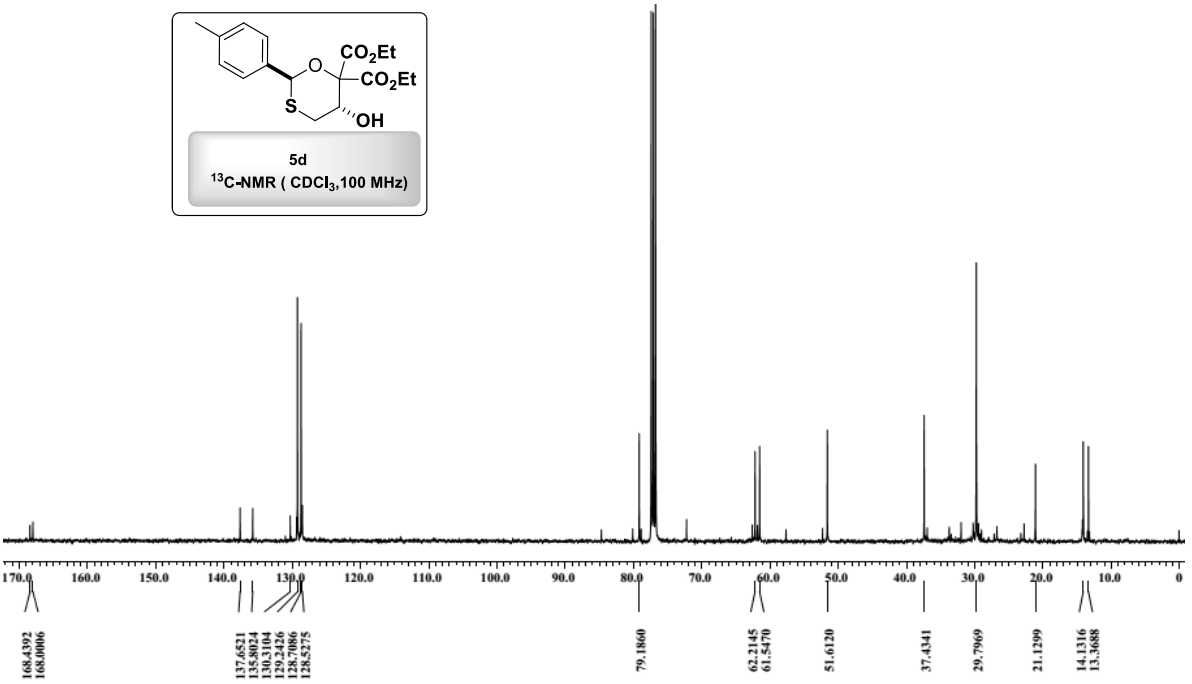
5c
^{13}C-DEPT ($CDCl_3$,100 MHz)

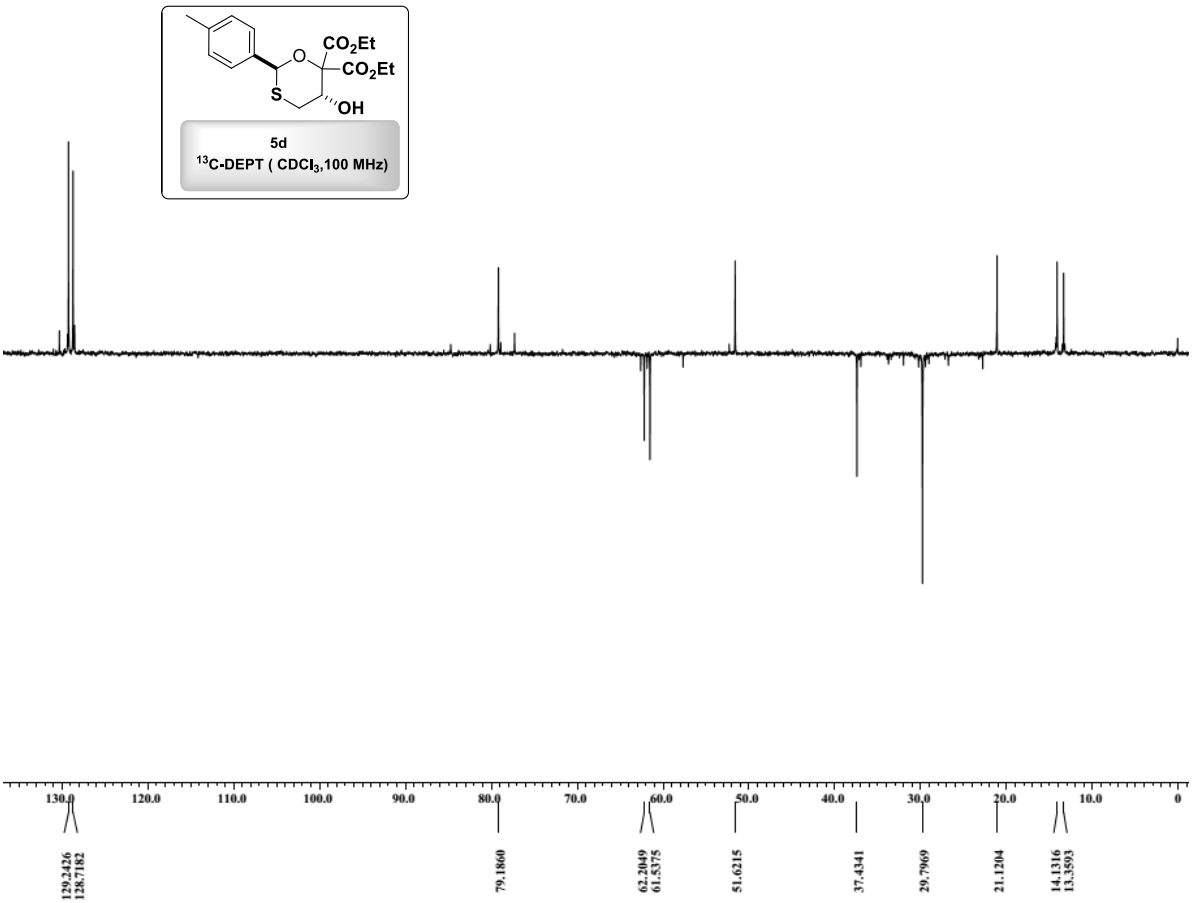


HRMS of 5c:

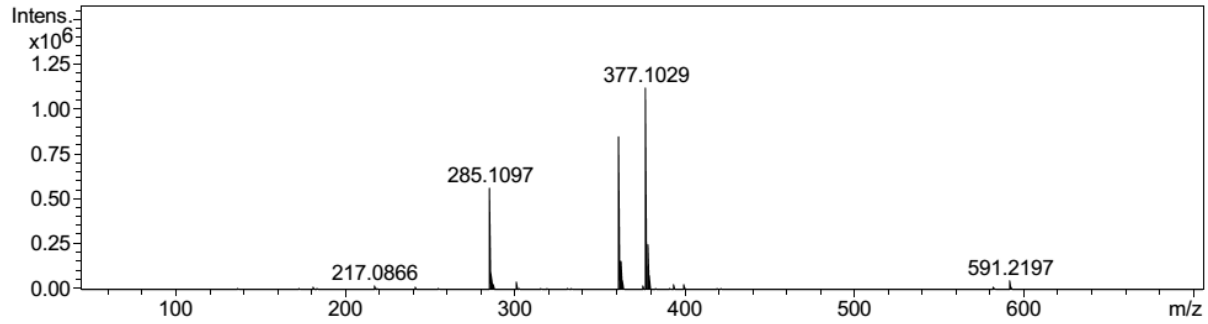

4422
08_03_069_P 20 (0.355) AM (Top,4, Ar,10000.0,0.00,0.00); Cm (20:27)
100
%
0
386.0864
386.3358
385.4070
385.5758
385.6562
386.1507
386.2392
386.5852

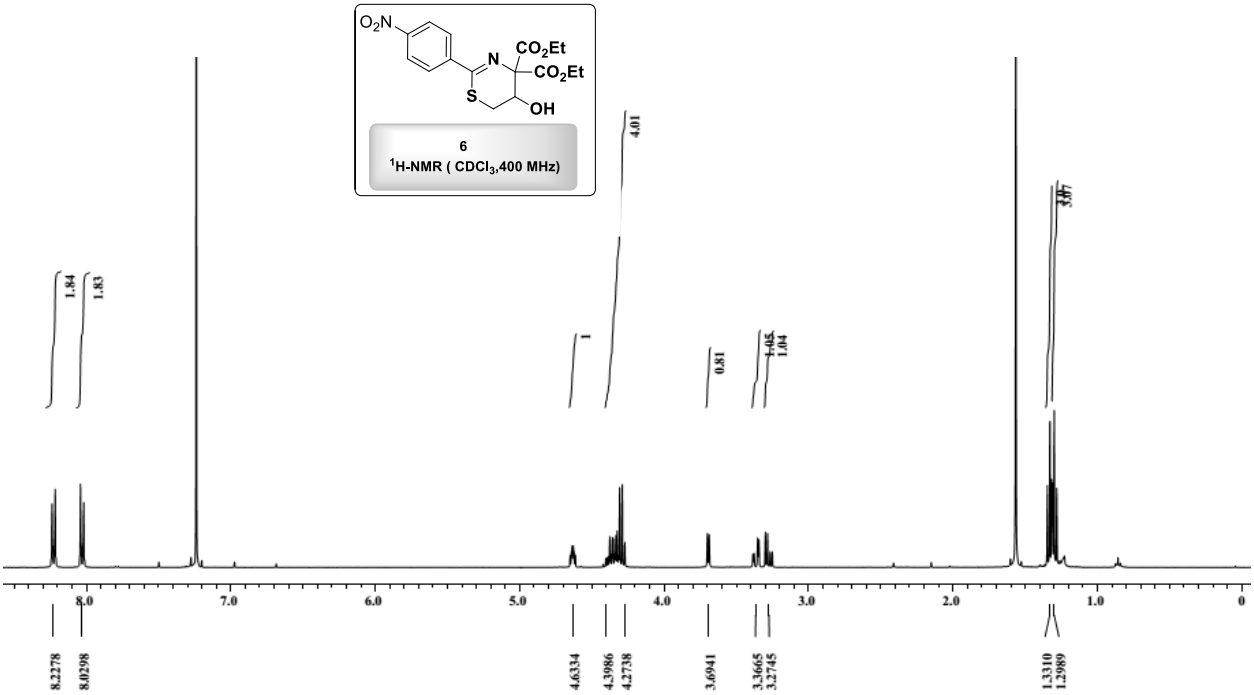

CO2Et
CO2Et
O
S
OH
5d
1H-NMR (CDCl3,400 MHz)
1.95
2.05
0.94
1
2.11
2.15
1.03
0.85
0.85
3.21
3.06
2.98
7.3133
7.0476
5.2572
5.0134
4.3213
4.2102
3.8436
3.7107
3.5072
2.9352
2.8860
2.2766
1.2548
0.7966

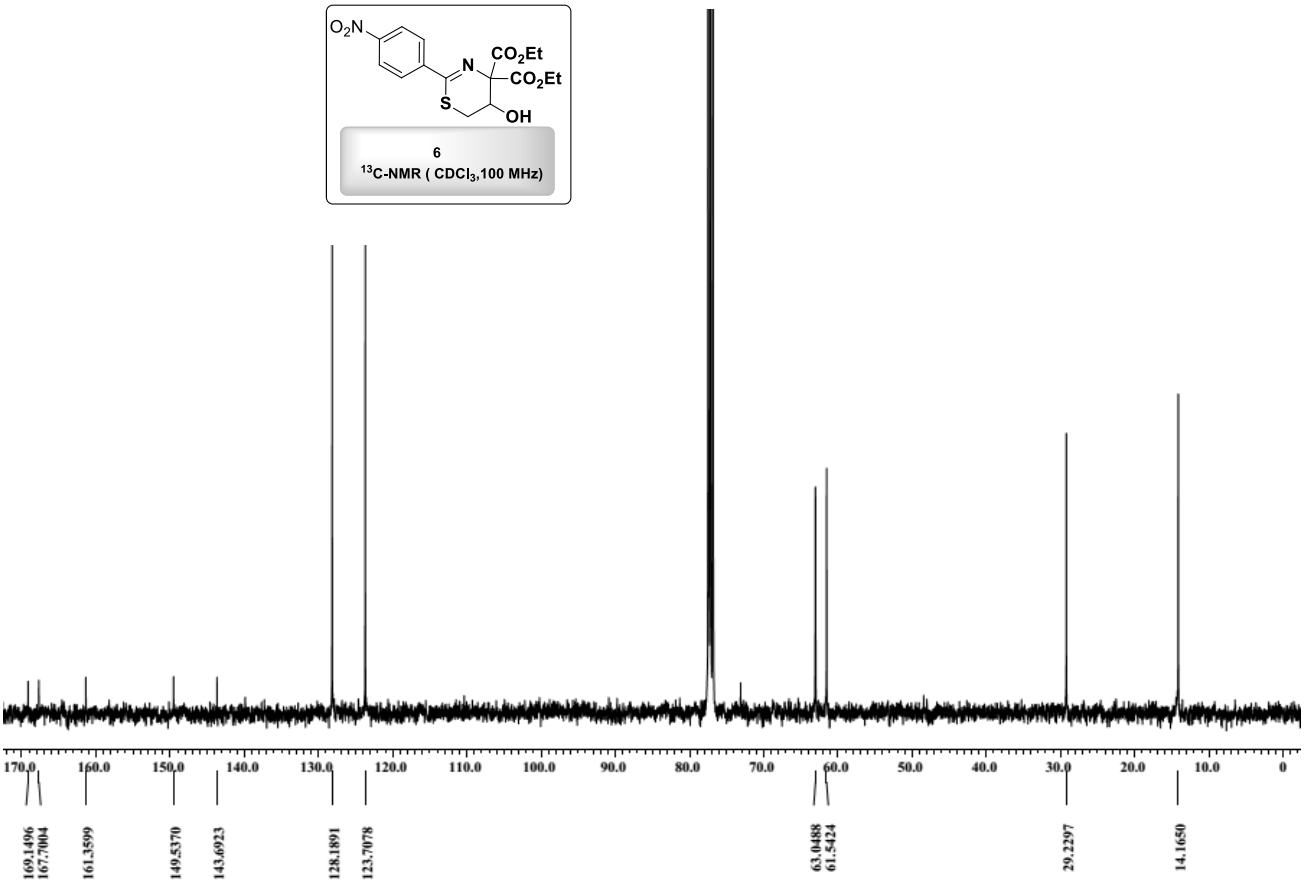

CO2Et
CO2Et
O
S
OH
5d
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.4392
168.0006
137.6521
135.8024
130.3104
129.2426
128.7086
128.5275
79.1860
62.2145
61.5470
51.6120
37.4341
29.7969
21.1299
14.1316
13.3688

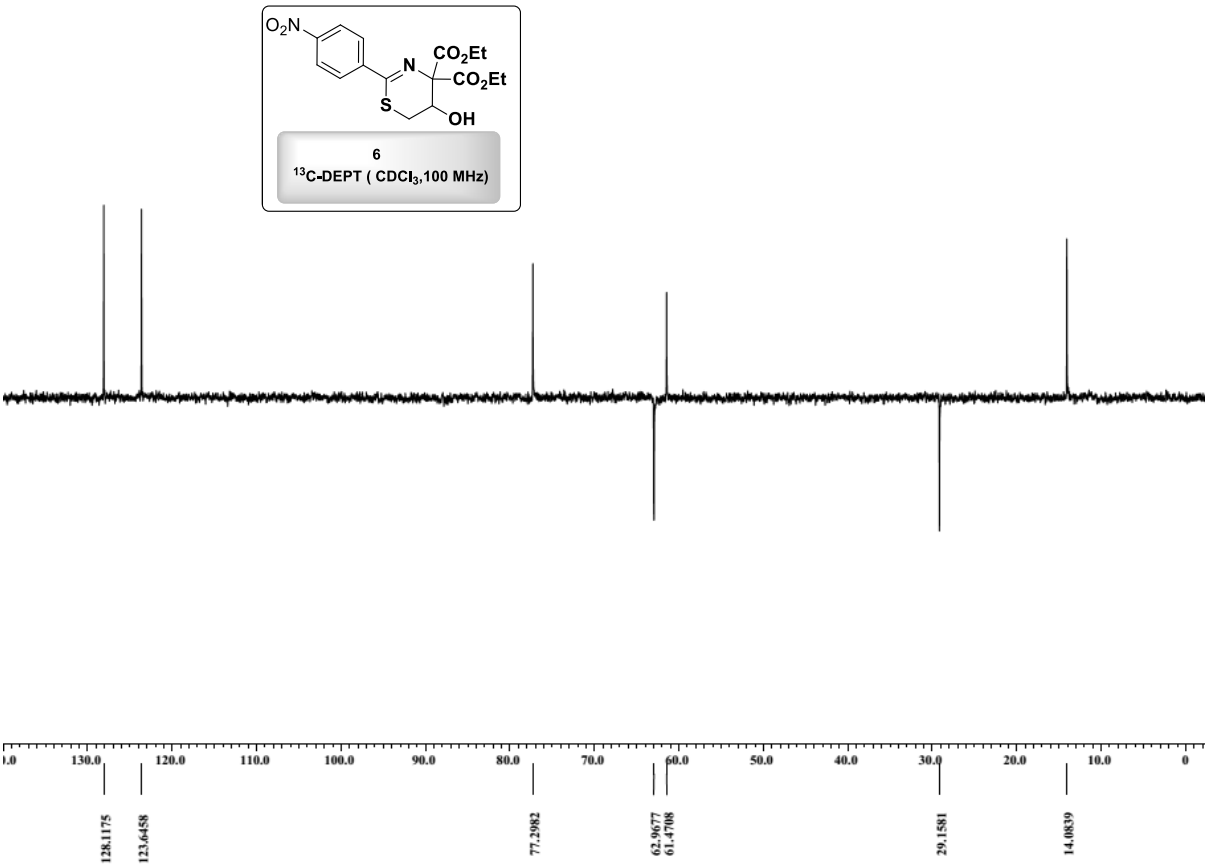

CO2Et
CO2Et
O
S
OH
5d
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.2426
128.7182
79.1860
62.2049
61.5375
51.6215
37.4341
29.7969
21.1204
14.1316
13.3593

HRMS of 5d:

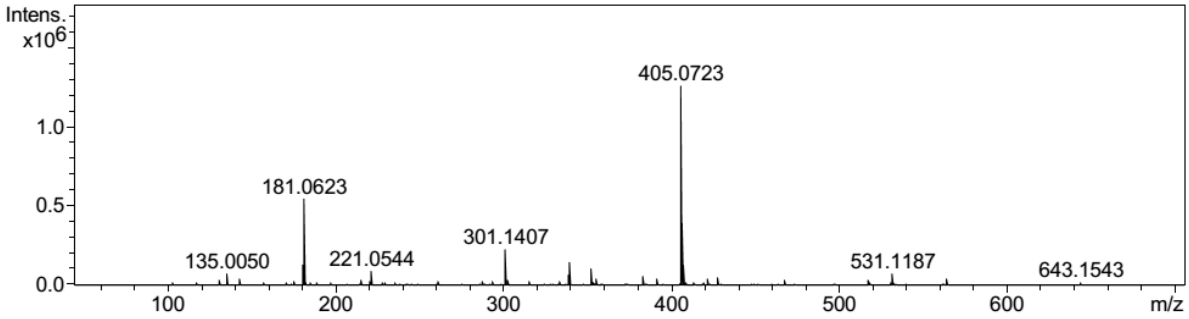

Intens.
x10^6
1.25
1.00
0.75
0.50
0.25
0.00
377.1029
285.1097
217.0866
591.2197
100
200
300
400
500
600
m/z

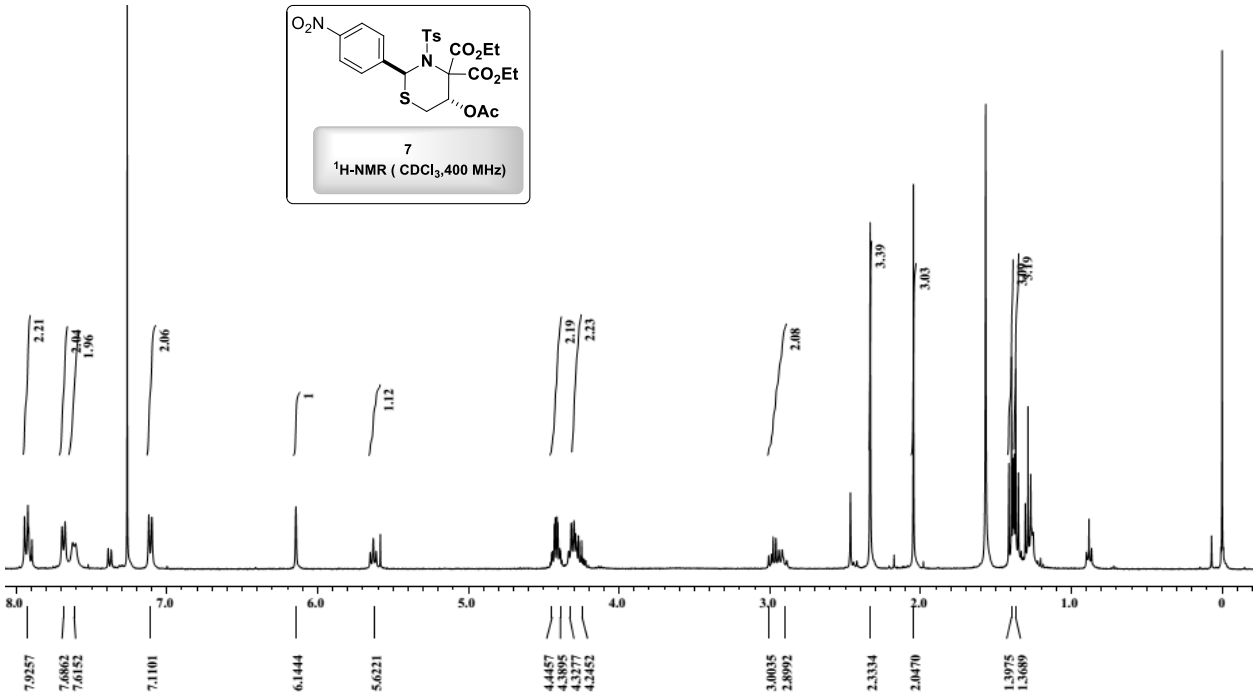

O2N
CO2Et
CO2Et
N
S
OH
6
1H-NMR (CDCl3,400 MHz)
1.84
1.83
1
4.01
0.81
1.05
1.04
3.07
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.2278
8.0298
4.6334
4.3986
4.2738
3.6941
3.3665
3.2745
1.3310
1.2989

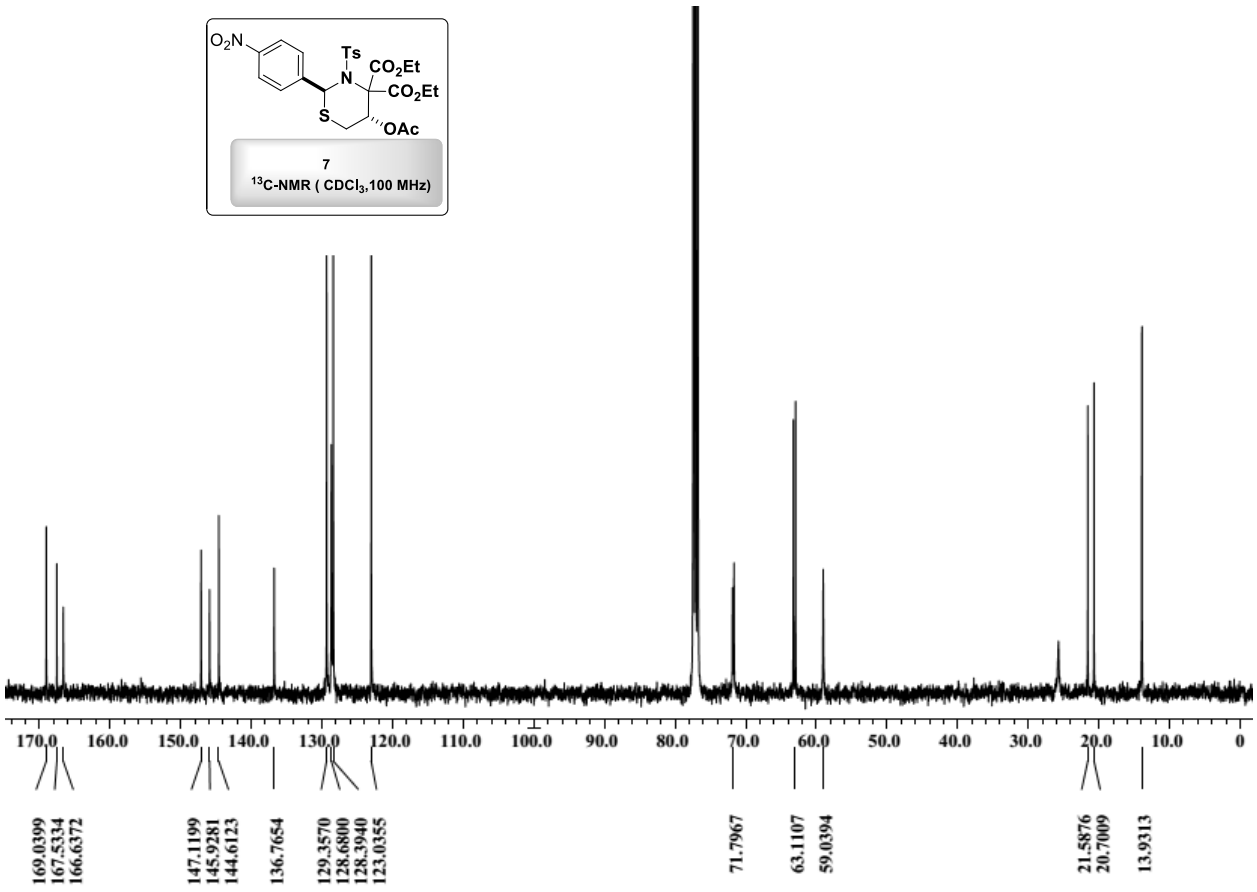

O2N
CO2Et
CO2Et
N
S
OH
6
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
169.1496
167.7004
161.3599
149.5370
143.6923
128.1891
123.7078
63.0488
61.5424
29.2297
14.1650

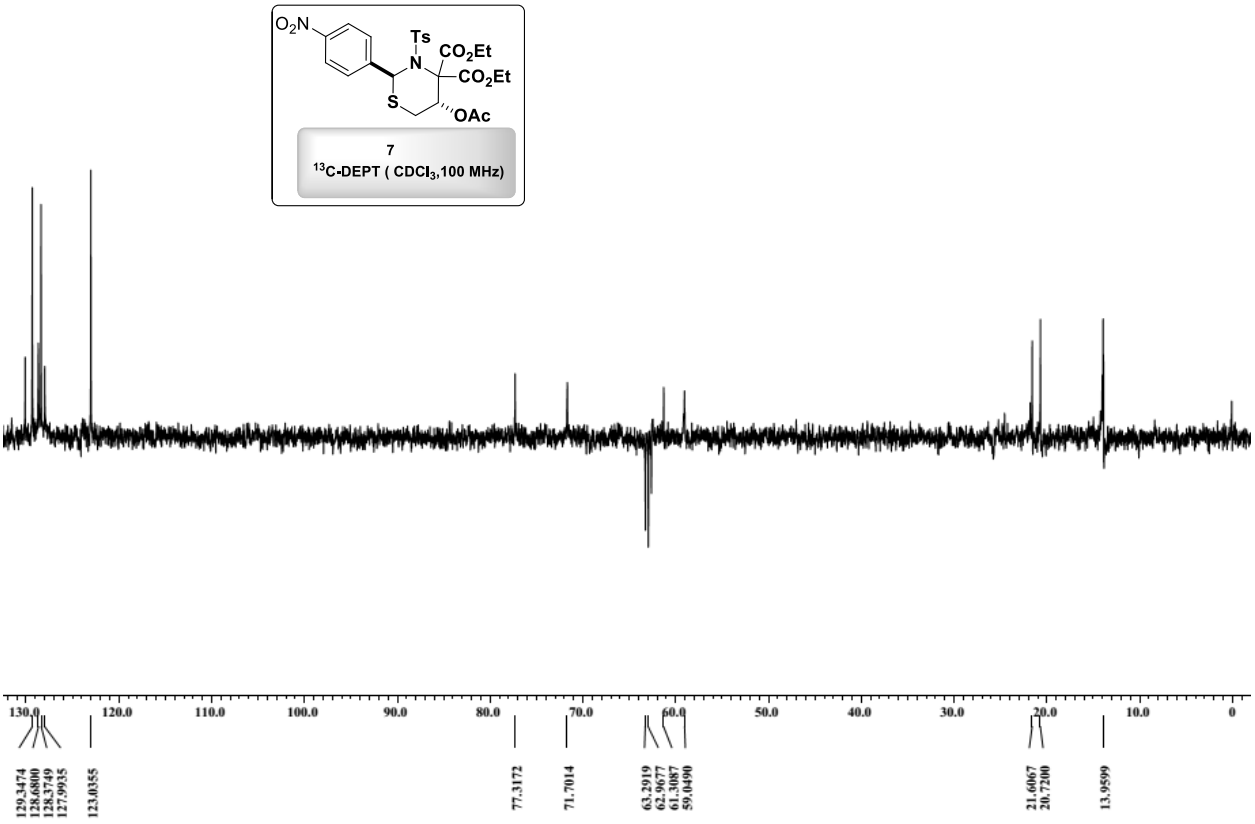

O2N
CO2Et
CO2Et
N
S
OH
6
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
128.1175
123.6458
77.2982
62.9677
61.4708
29.1581
14.0839

HRMS of 6:

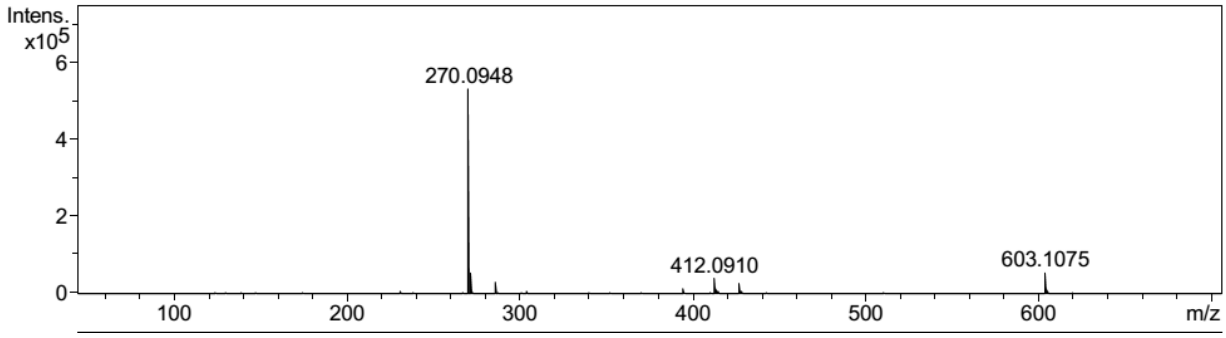

Intens.
x10^6
1.0
0.5
0.0
405.0723
181.0623
301.1407
135.0050
221.0544
531.1187
643.1543
100
200
300
400
500
600
m/z

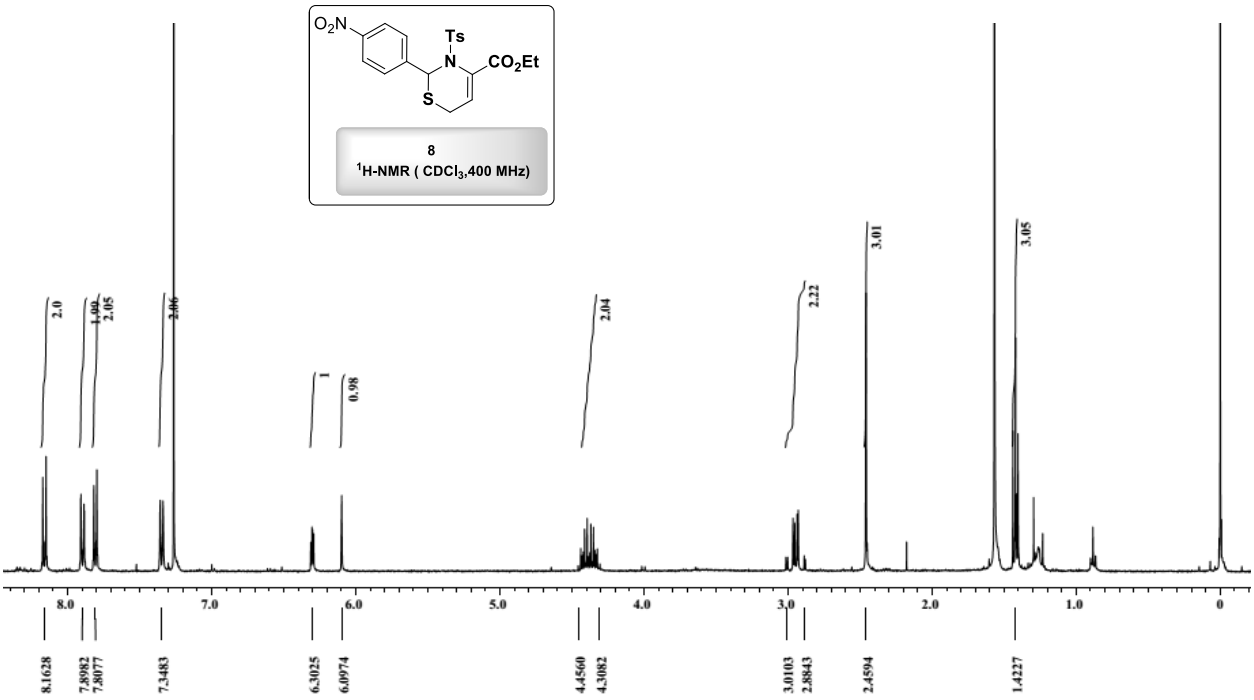

O2N
Ts
N
CO2Et
CO2Et
S
OAc
7
1H-NMR (CDCl3,400 MHz)
2.21
3.44
1.96
2.06
1
1.12
2.19
2.23
2.08
3.39
3.03
3.19
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.9257
7.6862
7.6152
7.1101
6.1444
5.6221
4.4457
4.3895
4.3277
4.2452
3.0035
2.8992
2.3334
2.0470
1.3975
1.3689

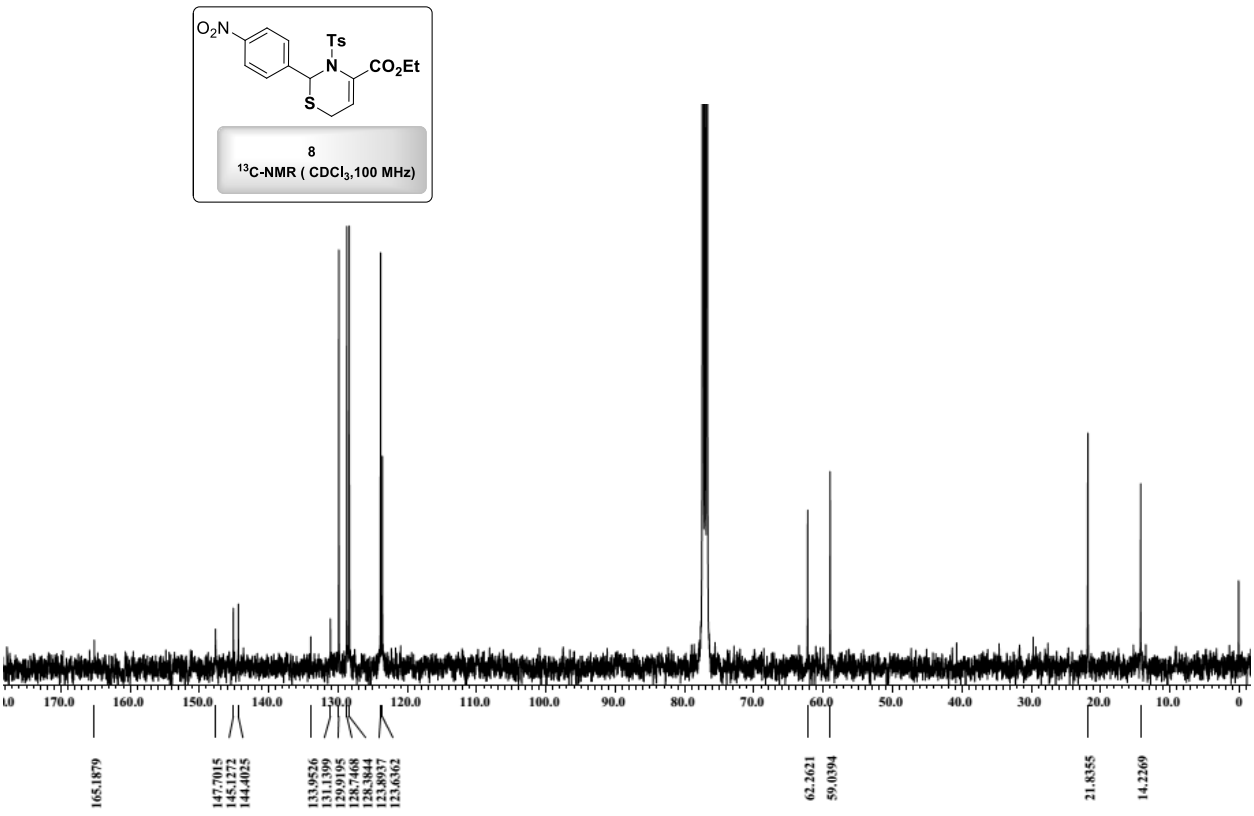

O2N
Ts
N
CO2Et
CO2Et
S
OAc
7
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
169.0399
167.5334
166.6372
147.1199
145.9281
144.6123
136.7654
129.3570
128.6800
128.3940
123.0355
71.7967
63.1107
59.0394
21.5876
20.7009
13.9313

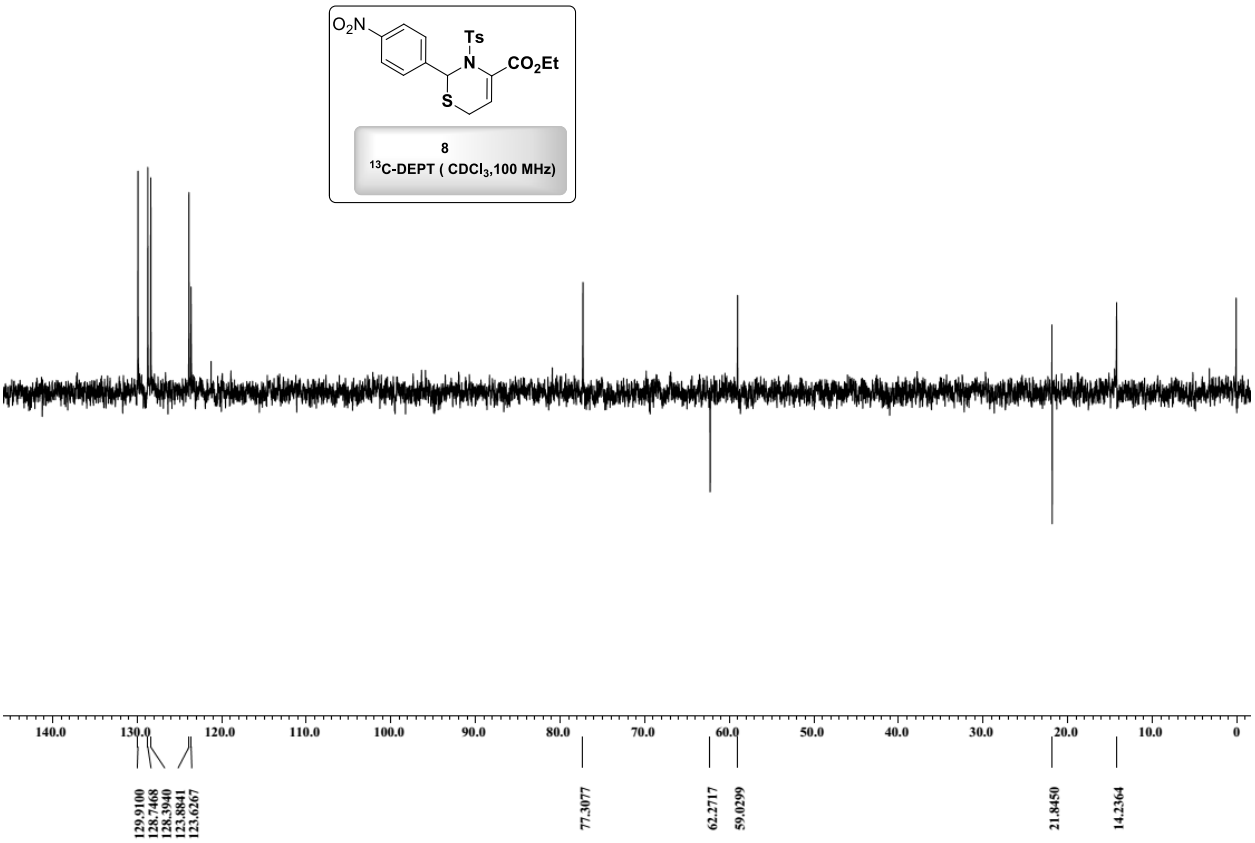

O2N
Ts
N
CO2Et
CO2Et
S
OAc
7
13C-DEPT (CDCl3,100 MHz)
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
139.3474
128.6400
128.3740
127.9935
123.0355
77.3172
71.7014
63.2919
62.9677
61.3087
59.0490
21.6067
20.7200
13.9599

HRMS of 7:

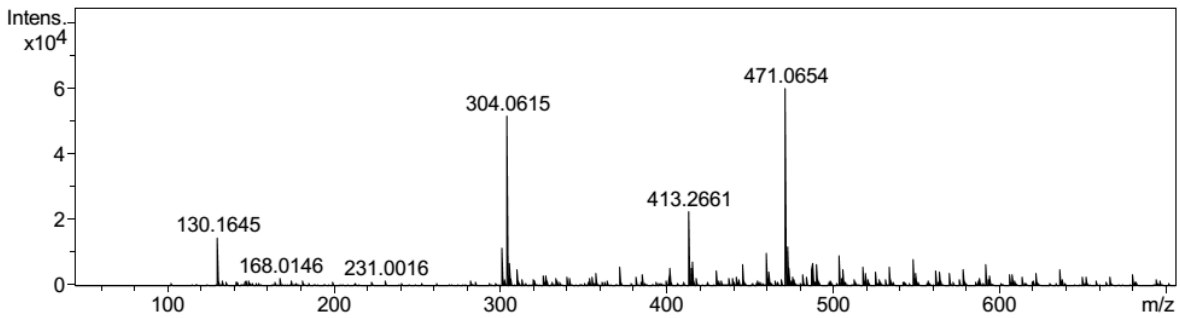

Intens.
x10^5
6
4
2
0
270.0948
412.0910
603.1075
100
200
300
400
500
600
m/z


O2N
Ts
N
CO2Et
S
8
1H-NMR (CDCl3,400 MHz)
2.0
1.00
2.05
3.06
1
0.98
2.04
2.22
3.01
3.05
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.1628
7.8982
7.8077
7.3483
6.3025
6.0974
4.4560
4.3082
3.0103
2.8843
2.4594
1.4227


O2N
Ts
N
CO2Et
S
8
13C-NMR (CDCl3,100 MHz)
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
165.1879
147.7015
145.1372
144.4025
133.9526
131.1399
129.9195
128.7468
128.3844
123.8937
123.6362
62.2621
59.0394
21.8355
14.2269


O2N
Ts
N
CO2Et
S
8
13C-DEPT (CDCl3,100 MHz)
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
129.9100
128.7468
128.3940
123.8841
123.6267
77.3077
62.2717
59.0299
21.8450
14.2364

HRMS of 8:


Intens.
x10^4
6
4
2
0
471.0654
304.0615
413.2661
130.1645
168.0146
231.0016
100
200
300
400
500
600
m/z

3. X-Ray diffraction:

For the determination of X-ray crystal structures of **3b** a single crystal was selected and mounted with paratone oil on a glass fiber using gum. The data was collected at 293K on a CMOS based Bruker D8 Venture PHOTON 100 diffractometer equipped with a INCOATEC micro-focus source with graphite monochromatic Mo Kα radiation (λ = 0.71073 Å) operation at 50 kV and 30 mA. For the integration of diffraction profiles SAINT program[1] was used. Absorption correction was done applying SADABS program.[2] The crystal structure was solved by SIR 92[3] and refined by full matrix least square method using SHELXL-97[4] WinGX system, Ver 1.70.01.[5] All the non-hydrogen atoms in the structure were located the Fourier map and refined anisotropically. The hydrogen atoms were fixed by HFIX in their ideal positions and refined using riding model with isotropic thermal parameters. The crystal structure (excluding structure factor) has been deposited to Cambridge Crystallographic Data Centre and allocated deposition number: **CCDC 1536757**

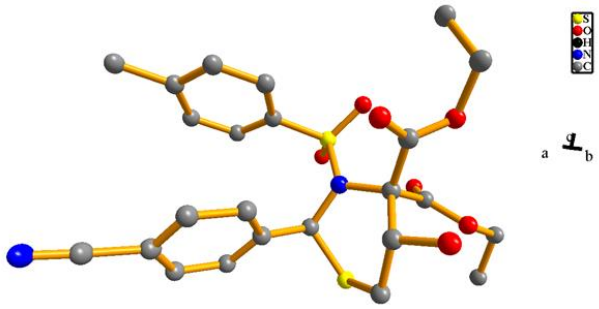


Fig1. X-ray crystal structure of compound 4b

CCDC No.	CCDC 1536757
Formula	C24 H26 N2 O7 S2
Formula weight	518.61
Crystal System	Triclinic
Space group	P-1
a, b, c (Å)	7.986(9), 9.532(12), 16.242(2)
α, β, γ (°)	79.589(4), 83.633(4), 86.893(4)
V (Å^3)	1208.0(3)
Z	2
Calculated Density (g/cm^3)	1.426
Absorption coefficient (mm^{-1})	0.269
F(000)	544
Crystal Size (mm^3)	0.18 x 0.23 x 0.32
Theta range for data collection:	2.3° to 28.3°
Data set	-10: 10 ; -12: 12 ; -21: 21
Reflection	62257
Independent refl.	6000 [R(int) = 0.030]
data [I > 2σ(I)]	5279
R indices (all data)	R = 0.0392, wR$_2$ = 0.1120
S	1.06

Min. and Max. Resd. Dens. (e/Å^3)	-0.47and 0.50

Table 1: Selected bond lengths [Å] of 3b

Atoms	**Bond lengths [Å]**	**Atoms**	**Bond lengths [Å]**
S(1)-O(1)	1.4291(12)	N(1)-C(24)	1.4737(19)
S(1) –O(2)	1.4301(13)	N(1)-C(23)	1.137(2)
S(1)-N(1)	1.6692(12)	C(1)-C(3)	1.386(2)
S(1)-C(1)	1.7563(17)	C(1)-C(2)	1.386(2)
S(2)-C(15)	1.802(2)	C(14)-C(24)	1.563(2)
S(2)-C(16)	1.8061(16)	C(14)-C(15)	1.524(2)
O(3)-C(11)	1.1935(19)	C(16)-C(17)	1.523(2)
O(4)-C(11)	1.3405(19)	C(17)-C(19)	1.395(2)
O(4)-C(12)	1.463(2)	C(17)-C(18)	1.387(2)
O(5)-C(8)	1.3206(19)	C(18)-C(20)	1.387(2)
O(5)-C(9)	1.469(2)	C(19)-C(21)	1.378(2)
O(7)-C(15)	1.3204(19)	C(20)-C(22)	1.389(2)
O(6)-C(8)	1.199(2)	C(21)-C(22)	1.394(2)
O(7)-C(14)	1.414(2)	C(22)-C(23)	1.445(2)
O(7)-H(7)	0.8200	C(13)-C(14)	1.493(3)
N(1)-C(16)	1.4771(19)		

Table 2: Selected bond angles [º] of 3b

Atoms	Bond angles[º]	Atoms	Bond angles[º]
O(1)-S(1)-O(2)	120.35(8)	O(4)-C(11)-C(24)	111.46(12)
O(1)-S(1)-N(2)	105.96(7)	O(3)-C(11)-C(4)	124.71(14)
O(1)-S(1))-C(1)	109.64(7)	O(4)-C(12)-C(13)	110.44(16)
O(2)-S(1)-N(1)	109.87(7)	O(7)-C(14)-C(15)	110.50(14)
O(2)-S(1))-C(1)	107.06(8)	O(7)-C(14)-C(24)	112.11(13)
N(1)-S(1))-C(1)	102.57(7)	C(15)-C(14)-C(24)	114.81(14)
C(15)-S(2)-C(16)	99.98(8)	S(2)-C(15)-C(14)	113.38(12)
C(11)-O(4)-C(12)	115.67(13)	S(2)-C(16)-N(1)	112.32(10)
C(8)-O(5)-C(9)	116.13(14)	S(2)-C(16)-C(17)	114.35(10)
C(14)-H(7)-O(7)	109.00	N(1)-C(16)-C(17)	113.61(12)
S(1)-N(1)-C(24)	120.33(9)	C(16)-C(17)-C(18)	123.15(13)
C(16)-N(1)-C(24)	120.11(11)	C(16)-C(17)-C(19)	117.43(13)
S(1)-N(1)-C(16)	114.11(10)	C(18)-C(17)-C(19)	119.20(14)
S(1)-C(1)-C(2)	119.44(12)	C(17)-C(18)-C(20)	120.59(15)
C(2)-C(1)-C(3)	121.01(15)	C(17)-C(19)-C(21)	120.90(15)
S(1)-C(1)-C(3)	119.50(12)	C(18)-C(20)-C(22)	119.45(15)
C(1)-C(2)-C(5)	118.92(17)	C(19)-C(21)-C(22)	119.28(16)
C(1)-C(3)-C(4)	118.81(15)	C(20)-C(22)-C(23)	119.26(15)
C(3)-C(4)-C(6)	121.42(16)	C(21)-C(22)-C(23)	120.11(16)
C(2)-C(5)-C(6)	121.46(19)	C(20)-C(22)-C(21)	120.57(15)
C(4)-C(6)-C(5)	118.39(17)	N(2)-C(23)-C(22)	177.9(2)
C(5)-C(6)-C(7)	119.97(17)	N(1)-C(24)-C(11)	110.21(12)
C(4)-C(6)-C(7)	121.65(16)	N(1)-C(24)-C(14)	108.07(12)
O(5)-C(8)-O(6)	126.27(15)	N(1)-C(24)-C(8)	108.93(11)
O(6)-C(8)-C(24)	121.18(14)	C(8)-C(24)-C(14)	102.89(12)
O(5)-C(8)-C(24)	112.46(13)	C(11)-C(24)-C(14)	113.96(12)
O(5)-C(9)-C(10)	112.50(15)	C (8)-C(24)-C(11)	112.44(12)
O(3)-C(11)-C(24)	111.63(13)	C(1)-C(2)-H(2)	121.00

Table 3: Selected hydrogen bonding geometry [Å, º] for a compound 3b

D--H.. **A**	**D**..**H**	**H**..**A**	**D**..**A**	**D--H**..**A**
C(3)--H(3)..O(2)	0.9300	2.5200	2.885(2)	104.00
C(16)--H(16)..O(2)	0.9800	2.3100	2.906(2)	118.00
C(18)--H(18)..N(1)	0.9300	2.5800	2.896(2)	100.00
C(20)--H(20)..O(2)	0.9300	2.5800	3.372(2)	143.00
C(20)--H(20)..O(3)	0.9300	2.4700	3.214(2)	137.00

4. Reference:

(1) Bruker, SAINT V7.68A, Bruker AXS Inc., Madison (WI, U.S.A), **2005.**
(2) G. M. Sheldrick, SADABS 2008/2, GÖttingen, **2008**.

(3) A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, *J. Appl. Cryst*. **1993**, *26*, 343.
(4) G. M. Sheldrick, SHELXL-97, Program for *Crystal Structure Solution and Refinement*; University of GÖttingen, Germany, **1997.**
(5) L. Farrugia, WinGX-A Windows Program for Crystal Structure Analysis, *J. Appl. Cryst*. **1999**, *32*, 837.