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Supporting Information Available

CuO/I_2-mediated intramolecular annulation for the synthesis of 2-aroyl-3-hydroxy-4-iodonaphthalenes

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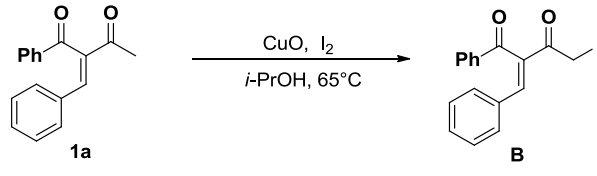
1. General

All substrates and reagents were commercially available and used without further purification. TLC analysis was performed using pre-coated glass plates. Column chromatography was performed using silica gel (200-300 mesh). IR spectra were recorded on a Perkin-Elmer PE-983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. ^1H spectra were recorded in $CDCl_3$ 600 MHz NMR spectrometers and resonances (δ) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C spectra were recorded in $CDCl_3$ 100/150 MHz NMR spectrometers and resonances (δ) are given in ppm. HRMS were obtained on a Bruker Apex-Ultra 7.0T FTMS equipped with an electrospray source (ESI). The X-ray crystal structure determination of **2a**, **2i** and **2j** were obtained on a Bruker SMART APEX CCD system. Melting points were determined using XT-4 apparatus and not corrected.

2. General procedure for synthesis of 2 (2a as an example)

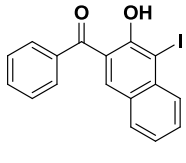
A mixture of (*Z*)-2-benzylidene-1-phenylbutane-1,3-dione **1a** (1.0 mmol, 1.0 equiv), CuO (3.0 mmol, 3.0 equiv) and I_2 (2.0 mmol, 2.0 equiv) in 5 mL ethanol at reflux for 36 h, then filter the precipitate and add CuO (3.0 mmol, 3.0 equiv) and I_2 (2.0 mmol, 2.0 equiv) to mother liquor. Continue refluxing for 12 h till almost completed conversion of the substrates by TLC analysis. Then extract with EtOAc three times (3 × 50 mL). The extract was washed with 10% $Na_2S_2O_3$ solution (w/w), dried over anhydrous Na_2SO_4 and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to afford the product **2a** as a yellow solid.

General procedure for synthesis of B



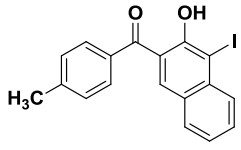
Finely powdered CuO (0.40 g, 5.0 mmol) and I_2 (1.27 g, 5.0 mmol) were added to a well-stirred soln of (*Z*)-2-benzylidene-1-phenylbutane-1,3-dione **1a** (5.0 mmol) in *i*-PrOH (20 mL). The mixture was stirred for 5 min and then was heated at 65 °C for 10 h. After disappearance of the reactant (TLC), the mixture was filtered and the solvent was removed under reduced pressure (Note: the mixture could not be treated with $Na_2S_2O_3$ because it reacted with the iodinated product at r.t.), then direct purification of the residue by column chromatography gave the target products in 68% yield.

3. Spectral data of compounds 2 and B.

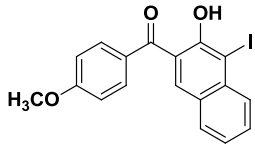


(3-hydroxy-4-iodonaphthalen-2-yl)(phenyl)methanone (2a). Yield 80%; Yellow solid; m.p. 160.2–162.8 °C; ^1H NMR ($CDCl_3$, 600 MHz): δ 12.25 (s, 1H), 8.18 (s, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.76 (d, J = 7.2 Hz, 2H), 7.71–7.64 (m, 3H), 7.57 (d, J = 7.2 Hz, 2H), 7.38 (t, J = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 201.1, 156.7, 138.6, 137.5, 137.3, 132.6, 131.5, 130.7, 130.2, 129.6, 128.6,

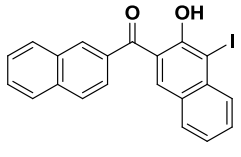
127.4, 124.7, 120.3, 86.9; IR (KBr): 1630, 1345, 1287, 1242 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{17}H_{12}IO_2$: 374.9877; found: 374.9873.



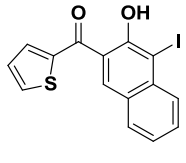
(3-hydroxy-4-iodonaphthalen-2-yl)(p-tolyl)methanone (2b). Yield 62%; Yellow solid; m.p. 147.3–148.8 °C; ^1H NMR ($CDCl_3$, 600 MHz): δ 12.26 (s, 1H), 8.19 (s, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.71–7.64 (m, 4H), 7.39–7.35 (m, 3H), 2.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 200.7, 156.8, 143.7, 138.5, 137.3, 134.6, 131.4, 130.7, 130.1, 129.9, 129.3, 127.4, 124.7, 120.5, 86.8, 21.7; IR (KBr): 1630, 1341, 1322, 1283 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{14}IO_2$: 389.0033; found: 389.0033.



(3-hydroxy-4-iodonaphthalen-2-yl)(4-methoxyphenyl)methanone (2c). Yield 65%; Yellow solid; m.p. 112.4–113.4 °C; ^1H NMR ($CDCl_3$, 600 MHz): δ 12.17 (s, 1H), 8.18 (s, 1H), 8.13 (d, J = 7.8 Hz, 1H), 7.79 (d, J = 8.4 Hz, 2H), 7.72 (d, J = 7.8 Hz, 1H), 7.64 (t, J = 7.8 Hz, 1H), 7.38 (t, J = 7.8 Hz, 1H), 7.04 (d, J = 7.8 Hz, 2H), 3.93 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 199.3, 163.5, 156.6, 138.3, 136.8, 132.3, 131.2, 130.7, 130.1, 129.7, 127.4, 124.6, 120.7, 113.9, 86.7, 55.6; IR (KBr): 1632, 1605, 1329, 1263, 1176 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{14}IO_3$: 404.9982; found: 404.9986.

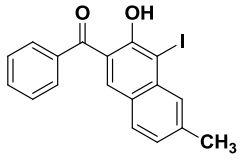


(3-hydroxy-4-iodonaphthalen-2-yl)(naphthalen-2-yl)methanone (2d). Yield 57%; Yellow solid; m.p. 173.3–175.8 °C; ^1H NMR ($CDCl_3$, 600 MHz): δ 12.25 (s, 1H), 8.25–8.24 (m, 2H), 8.15 (d, J = 8.4 Hz, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.97–7.95 (m, 2H), 7.85 (d, J = 8.4 Hz, 1H), 7.69–7.65 (m, 3H), 7.61 (t, J = 7.8 Hz, 1H), 7.38 (t, J = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 200.9, 156.7, 138.6, 137.5, 135.1, 134.5, 132.1, 131.5, 131.2, 130.7, 130.2, 129.3, 128.6, 127.9, 127.5, 127.2, 125.4, 124.7, 120.6, 86.9; IR (KBr): 1635, 1286 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{21}H_{14}IO_2$: 425.0033; found: 425.0028.

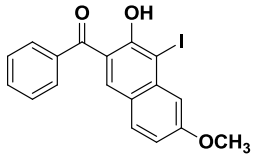


(3-hydroxy-4-iodonaphthalen-2-yl)(thiophen-2-yl)methanone (2e). Yield 51%; Yellow solid; m.p. 158.7–160.4 °C; ^1H NMR ($CDCl_3$, 600 MHz): δ 11.73 (s, 1H), 8.49 (s, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.84 (d, J = 5.4 Hz, 1H), 7.81–7.79 (m, 2H), 7.67 (t, J = 7.8 Hz, 1H), 7.42 (t, J = 7.2 Hz, 1H),

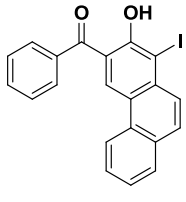
7.27–7.26 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 190.6, 155.9, 141.7, 138.3, 135.5, 135.4, 135.1, 131.3, 130.8, 130.1, 128.2, 127.6, 124.8, 120.8, 86.9; IR (KBr): 1628, 1571, 1411, 1287, 1232, 1222 cm^{-1}; HRMS (ESI): m/z $[M + Na]^+$ calcd for $C_{15}H_9INaO_2S$: 402.9260; found: 402.9264.



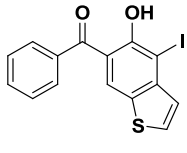
(3-hydroxy-4-iodo-6-methylnaphthalen-2-yl)(phenyl)methanone (2f). Yield 72%; Yellow solid; m.p. 167.4–168.9 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 12.35 (s, 1H), 8.11 (s, 1H), 7.89 (s, 1H), 7.74 (d, J = 7.8 Hz, 2H), 7.66 (t, J = 7.8 Hz, 1H), 7.58–7.54 (m, 3H), 7.19 (d, J = 8.4 Hz, 1H), 2.57 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 201.0, 156.9, 142.4, 138.7, 137.4, 137.3, 132.4, 130.1, 129.7, 129.5, 128.5, 127.1, 125.7, 119.5, 86.1, 22.3; IR (KBr): 1629, 1444, 1341, 1309, 1283 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{14}IO_2$: 389.0033; found: 389.0035.



(3-hydroxy-4-iodo-6-methoxynaphthalen-2-yl)(phenyl)methanone (2g). Yield 68%; Yellow solid; m.p. 138.1–138.9 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 12.59 (s, 1H), 8.05 (s, 1H), 7.73 (d, J = 8.4 Hz, 2H), 7.65 (t, J = 7.8 Hz, 1H), 7.58–7.53 (m, 3H), 7.39 (s, 1H), 6.97 (d, J = 9.0 Hz, 1H), 4.00 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 200.8, 162.6, 157.8, 140.8, 137.5, 137.2, 132.3, 132.2, 129.4, 128.5, 122.6, 118.0, 117.8, 109.4, 85.5, 55.6; IR (KBr): 1635, 1500, 1341, 1312, 1228 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{18}H_{14}IO_3$: 404.9982; found: 404.9977.

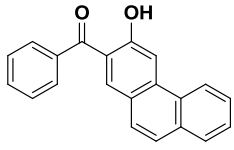


(2-hydroxy-1-iodophenanthren-3-yl)(phenyl)methanone (2h). Yield 59%; Yellow solid; m.p. 166.2–167.9 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 12.66 (s, 1H), 8.79 (s, 1H), 8.09 (d, J = 7.8 Hz, 1H), 7.96 (d, J = 9.6 Hz, 1H), 7.76–7.75 (m, 3H), 7.72–7.67 (m, 2H), 7.57 (t, J = 7.2 Hz, 2H), 7.52–7.48 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 200.8, 158.4, 138.4, 137.3, 132.8, 132.6, 130.6, 130.3, 130.2, 129.7, 129.5, 128.9, 128.5, 127.9, 126.6, 123.3, 121.6, 118.4, 89.5; IR (KBr): 1615, 1402 cm^{-1}; HRMS (ESI): m/z $[M + H]^+$ calcd for $C_{21}H_{14}IO_2$: 425.0033; found: 425.0034.

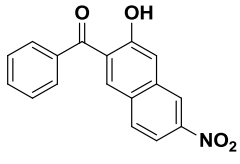


(5-hydroxy-4-iodobenzo[b]thiophen-6-yl)(phenyl)methanone (2i). Yield 43%; Yellow solid; m.p. 133.4–134.6 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 12.66 (s, 1H), 8.07 (s, 1H), 7.77 (d, J = 5.4 Hz, 1H),

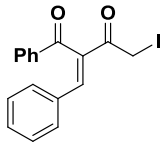
7.70 (d, J = 7.8 Hz, 2H), 7.64 (t, J = 7.2 Hz, 1H), 7.54 (t, J = 7.8 Hz, 2H), 7.44 (d, J = 5.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 200.8, 158.1, 148.6, 137.4, 134.3, 132.3, 129.3, 129.0, 128.7, 128.6, 128.1, 117.4, 80.3; IR (KBr): 1618, 1388, 1332, 1263, 1235 cm^{-1}; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{15}H_{10}IO_2S$: 380.9441; found: 380.9435.



(3-hydroxyphenanthren-2-yl)(phenyl)methanone (2j). Yield 85%; Yellow solid; m.p. 140.1–141.1 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 11.63 (s, 1H), 8.59 (bs, 1H), 8.21 (s, 1H), 8.13 (s, 1H), 7.82 (d, J = 3.0 Hz, 1H), 7.79 (d, J = 7.2 Hz, 2H), 7.67–7.64 (m, 3H), 7.58–7.53 (m, 4H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 201.4, 159.5, 137.9, 136.6, 135.5, 133.5, 132.2, 129.5, 128.7, 128.55, 128.47, 126.9, 126.7, 125.2, 124.7, 123.9, 119.6, 109.7; IR (KBr): 1641, 1332, 1211 cm^{-1}; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{21}H_{15}O_2$: 299.1067; found: 299.1067.

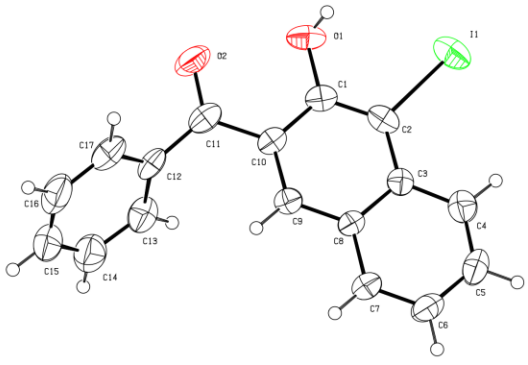


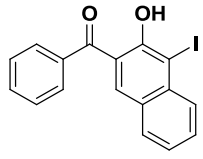
(3-hydroxy-6-nitronaphthalen-2-yl)(phenyl)methanone (2k). Yield 38%; Yellow solid; m.p. 144.8–146.3 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 11.16 (s, 1H), 8.63 (s, 1H), 8.27 (s, 1H), 8.04 (d, J = 9.0 Hz, 1H), 7.89 (d, J = 9.0 Hz, 1H), 7.79 (d, J = 7.8 Hz, 2H), 7.71 (t, J = 7.2 Hz, 1H), 7.61–7.58 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 201.3, 158.5, 148.0, 137.2, 136.3, 135.9, 132.9, 131.2, 129.6, 128.7, 128.6, 123.5, 122.6, 117.2, 114.7; IR (KBr): 1643, 1531, 1342, 1294, 1215 cm^{-1}; HRMS (ESI): m/z [M + H]$^+$ calcd for $C_{17}H_{12}NO_4$: 294.0761; found: 294.0754.



(E)-2-benzylidene-4-iodo-1-phenylbutane-1,3-dione (B). Yield 68%; Yellow solid; m.p. 120.1–121.8 °C; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.98 (s, 1H), 7.95 (d, J = 7.8 Hz, 2H), 7.52 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.8 Hz, 2H), 7.32 (t, J = 7.8 Hz, 2H), 7.29–7.24 (m, 1H), 7.22 (t, J = 7.8 Hz, 2H), 4.20 (s, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 196.9, 190.6, 143.6, 135.8, 135.7, 134.1, 132.5, 130.8, 130.5, 129.4, 128.8, 128.7, 2.3; IR (KBr): 3450, 3050, 1668, 1639, 1616, 1325, 1259, 1234, 915, 750, 689 cm^{-1}; HRMS (ESI): m/z [M + Na]$^+$ calcd for $C_{17}H_{13}INaO_2$: 398.9852; found: 398.9858.

4. Molecular structure and crystallographic data of compounds 2a, 2i and 2j.

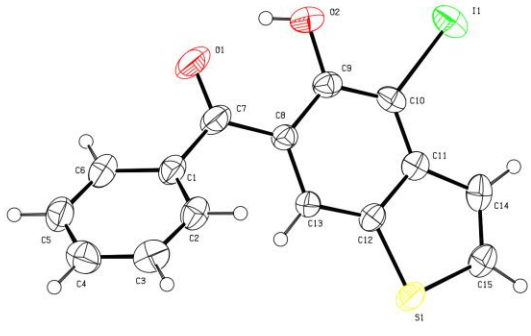


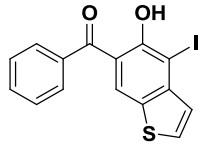


2a

Figure S1 X-ray crystal structure of compounds **2a**

Crystal Data for Compound **2a**: $C_{17}H_{11}IO_2$, MW = 374.16, monoclinic, a = 11.327(4) Å, b = 11.780(5) Å, c = 10.747(4) Å, α= 90.00 °, β= 94.540(4)°, γ= 90.00 °, V = 1429.5(9) $Å^3$, T = 296(2) K , space group P2(1)/c, Z = 4, m(Mo–Ka) = 2.238 mm^{-1}, 6008 reflections collected, 2592 unique [R(int) = 0.0273] which were used in all calculations. The final wR2 (F2) was 0.0771. CCDC 986777 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.





2i

Figure S2 X-ray crystal structure of compounds **2i**

Crystal Data for Compound **2i**: $C_{15}H_9IO_2S$, MW = 380.18, orthorhombic, a = 16.603(3) Å, b = 4.1488(8) Å, c = 19.295(4) Å, α= 90.00 °, β= 90.00°, γ= 90.00 °, V = 1329.1(4) $Å^3$, T = 296(2) K , space group Pna2(1), Z = 4, m(Mo–Ka) = 2.560 mm^{-1}, 6961 reflections collected, 2584 unique [R(int) = 0.0590] which were used in all calculations. The final wR2 (F2) was 0.0872. CCDC 986778 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

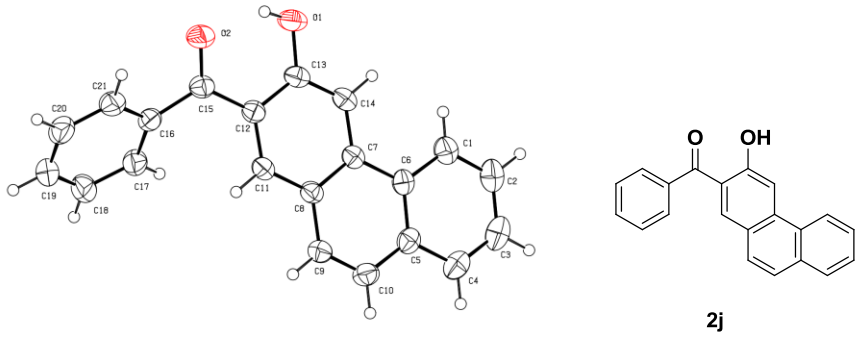
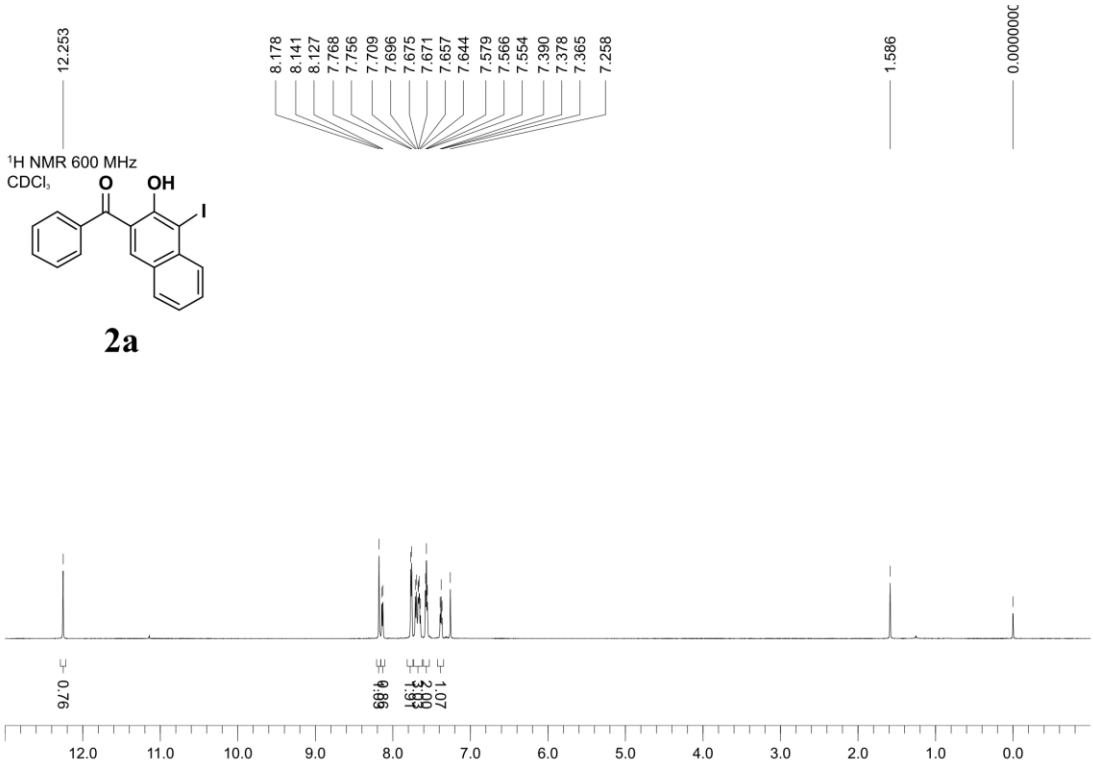
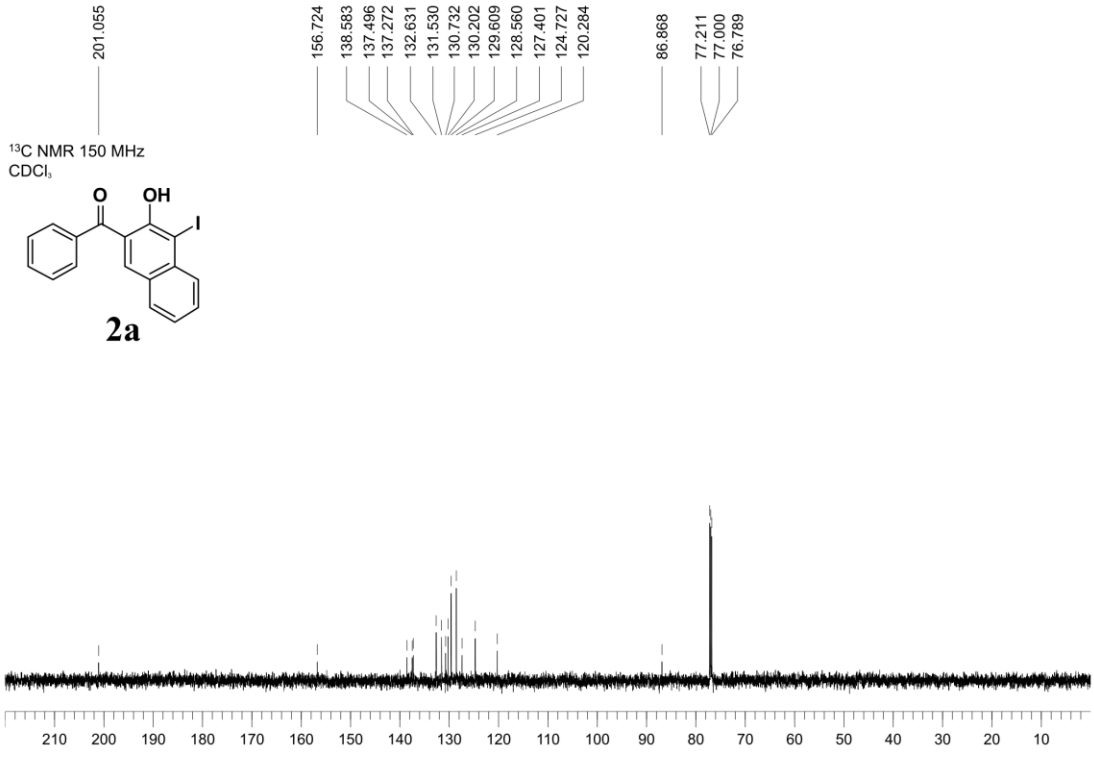


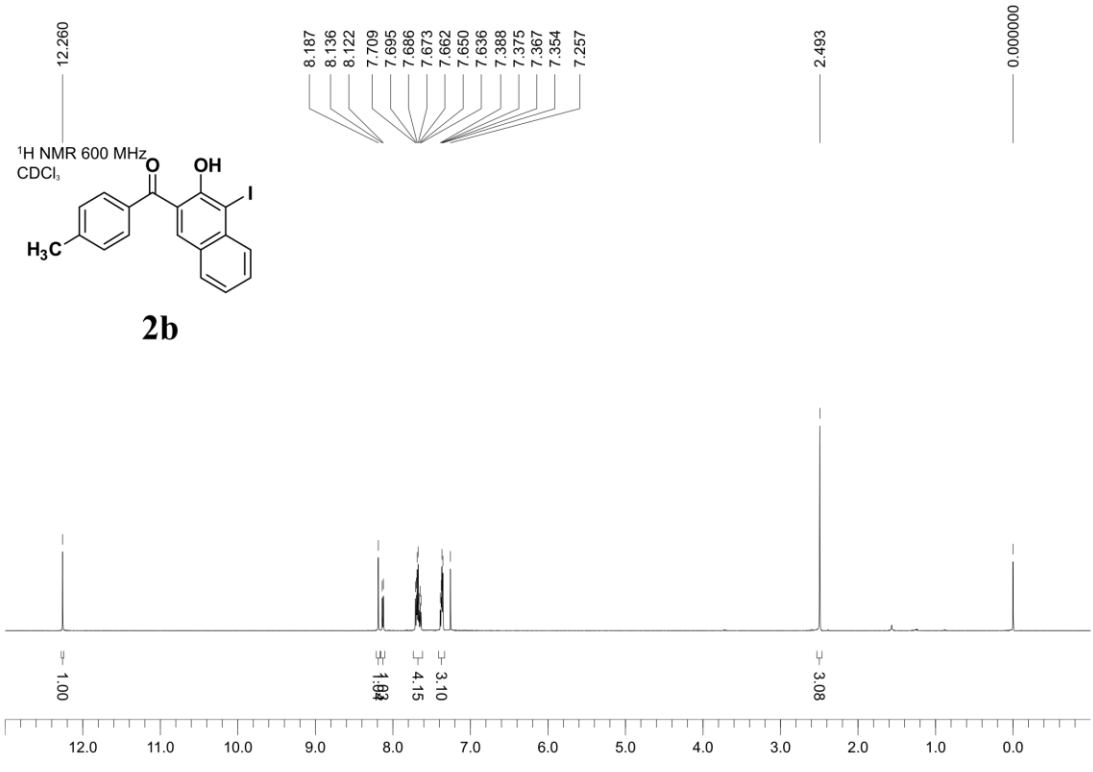
Figure S3 X-ray crystal structure of compounds **2j**

Crystal Data for Compound **2j**: $C_{21}H_{14}O_2$, MW = 298.32, monoclinic, a = 13.966(2) Å, b = 12.7793(19) Å, c = 8.4353(13) Å, α= 90.00°, β= 100.374(2)°, γ= 90.00 °, V = 1480.9(4) $Å^3$, T = 296(2) K , space group P2(1)/c, Z = 4, m(Mo–Ka) = 0.085 mm^{-1}, 7985 reflections collected, 2741 unique [R(int) = 0.0236] which were used in all calculations. The final wR2 (F2) was 0.1291. CCDC 986779 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

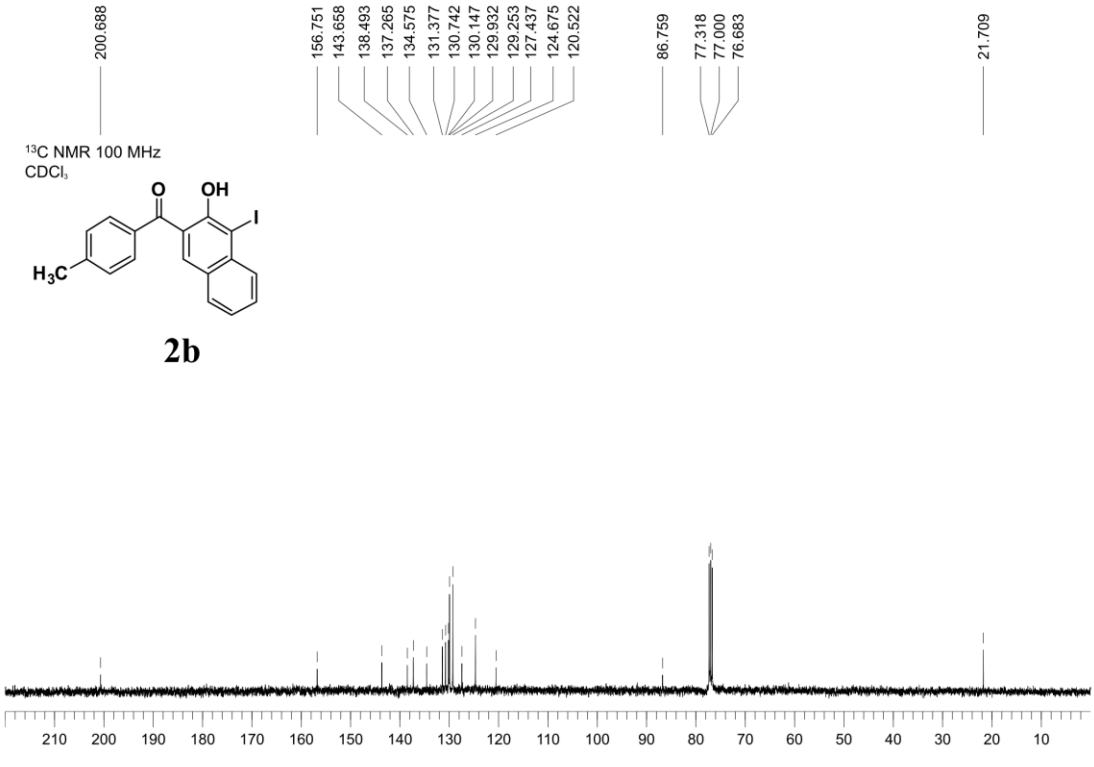
5. ^{1}H NMR and ^{13}C NMR spectra of compounds 2 and B.



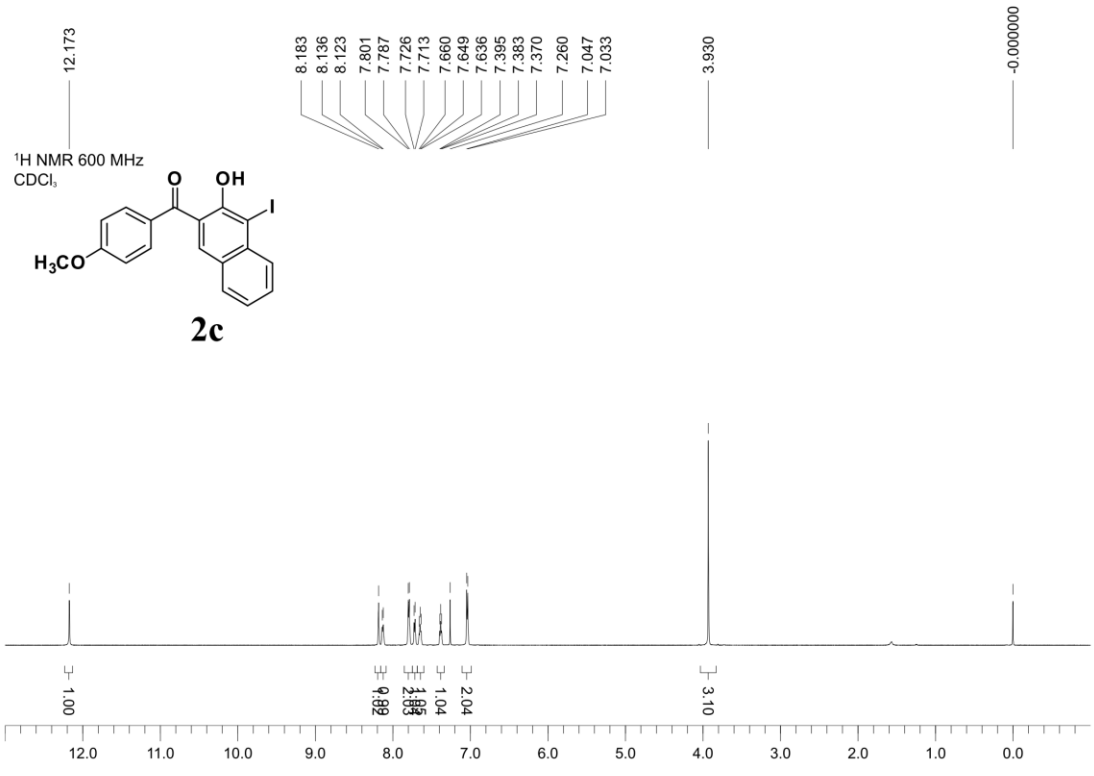


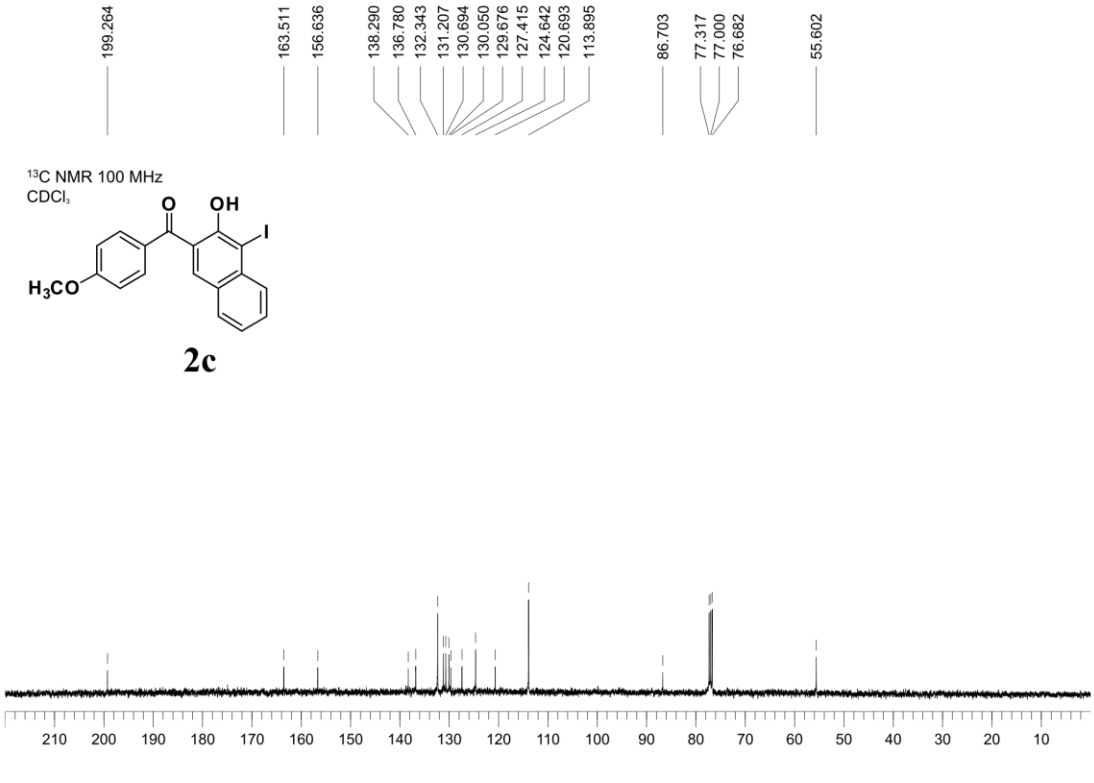

12.260
8.187
8.136
8.122
7.709
7.695
7.686
7.673
7.662
7.650
7.636
7.388
7.375
7.367
7.354
7.257
2.493
0.000000
^{1}H NMR 600 MHz
$CDCl_3$
O
OH
I
H_3C
1.00
1.03
4.15
3.10
3.08
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

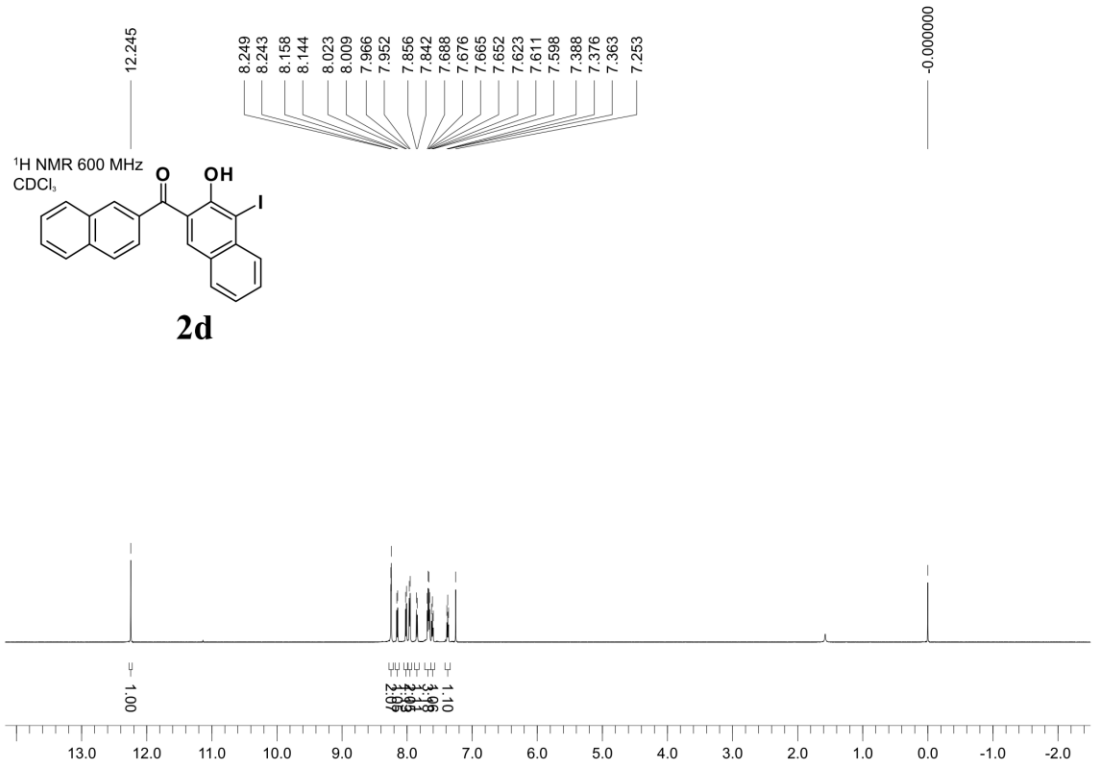
2b

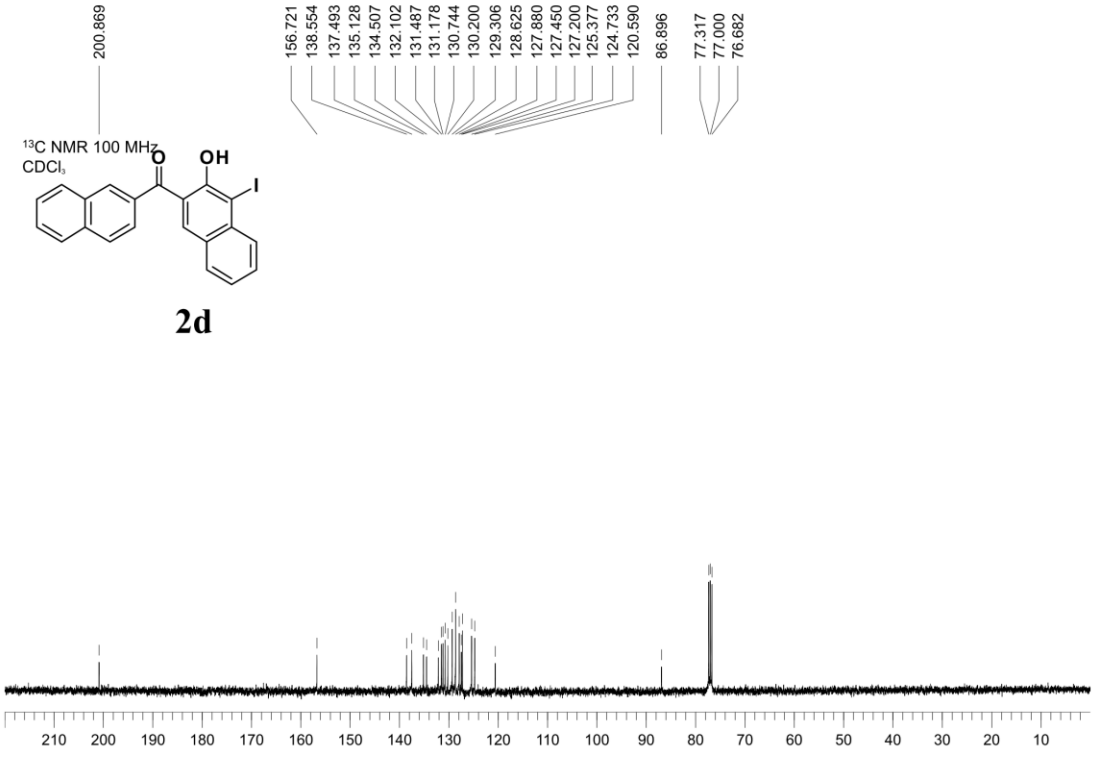

200.688
156.751
143.658
138.493
137.265
134.575
131.377
130.742
130.147
129.932
129.253
127.437
124.675
120.522
86.759
77.318
77.000
76.683
21.709
^{13}C NMR 100 MHz
$CDCl_3$
O
OH
I
H_3C
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

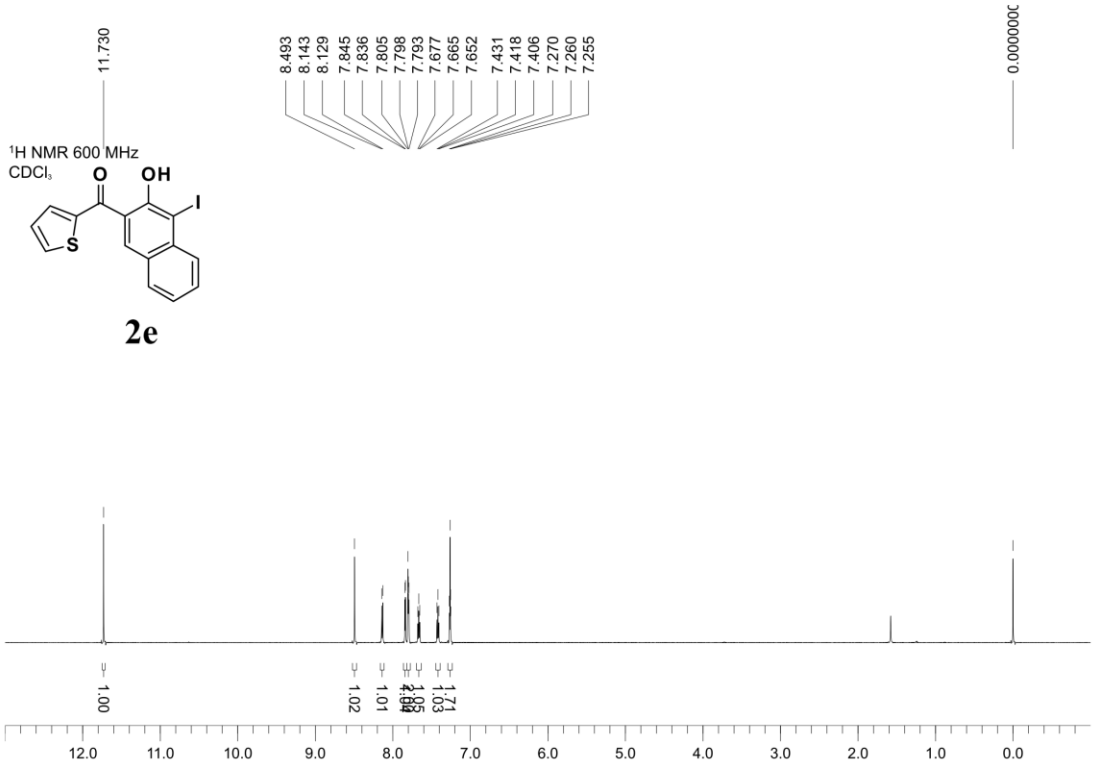
2b


12.173
8.183
8.136
8.123
7.801
7.787
7.726
7.713
7.660
7.649
7.636
7.395
7.383
7.370
7.260
7.047
7.033
3.930
-0.000000
1H NMR 600 MHz
CDCl3
O
OH
I
H3CO
2c
1.00
9.09
2.03
1.04
2.04
3.10
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

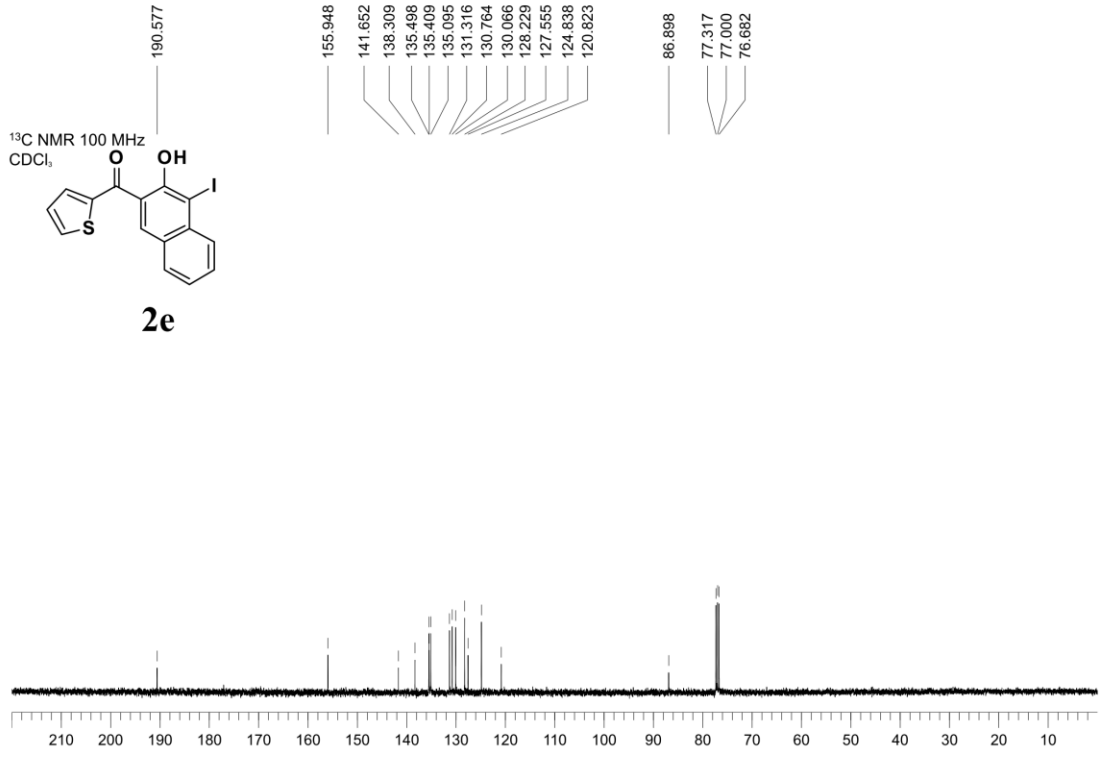

199.264
163.511
156.636
138.290
136.780
132.343
131.207
130.694
130.050
129.676
127.415
124.642
120.693
113.895
86.703
77.317
77.000
76.682
55.602
13C NMR 100 MHz
CDCl3
O
OH
I
H3CO
2c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


12.245
8.249
8.243
8.158
8.144
8.023
8.009
7.966
7.952
7.856
7.842
7.688
7.676
7.665
7.652
7.623
7.611
7.598
7.388
7.376
7.363
7.253
-0.000000
1H NMR 600 MHz
CDCl3
O
OH
I
2d
1.00
1.10
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.0

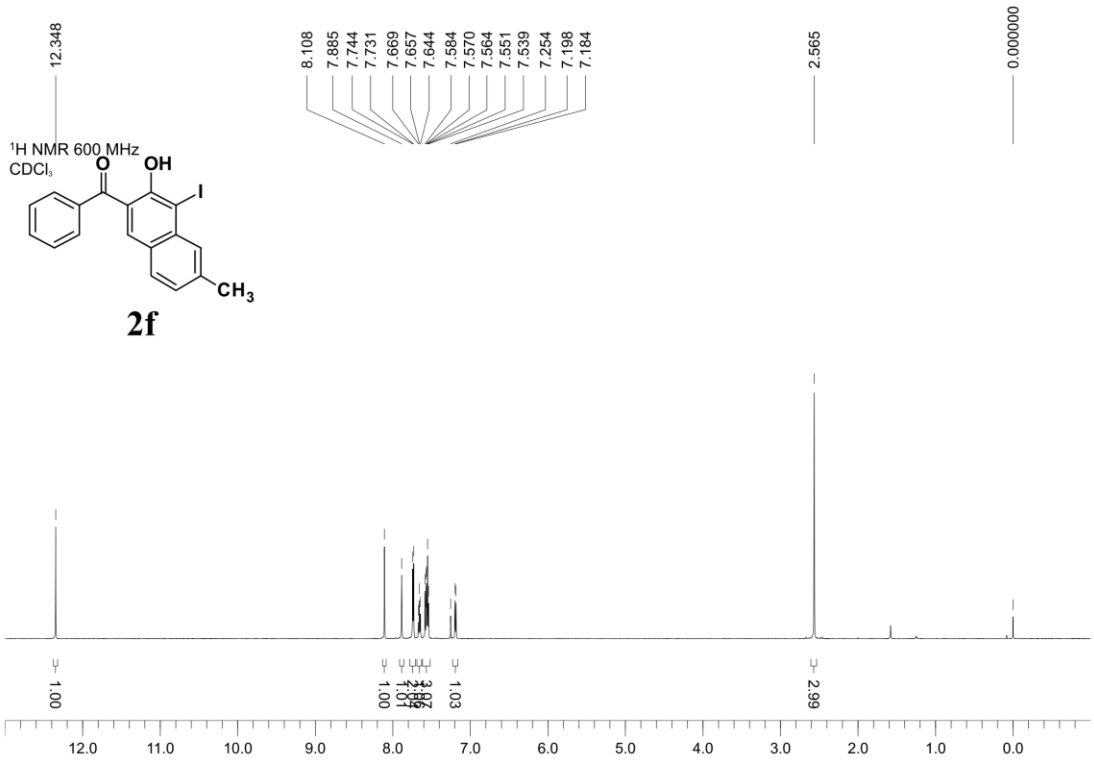

200.869
156.721
138.554
137.493
135.128
134.507
132.102
131.487
131.178
130.744
130.200
129.306
128.625
127.880
127.450
127.200
125.377
124.733
120.590
86.896
77.317
77.000
76.682
13C NMR 100 MHz
CDCl3
O
OH
I
2d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


11.730
8.493
8.143
8.129
7.845
7.836
7.805
7.798
7.793
7.677
7.665
7.652
7.431
7.418
7.406
7.270
7.260
7.255
0.000000
1H NMR 600 MHz
CDCl3
O
OH
I
S
1.00
1.02
1.01
1.03
1.11
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

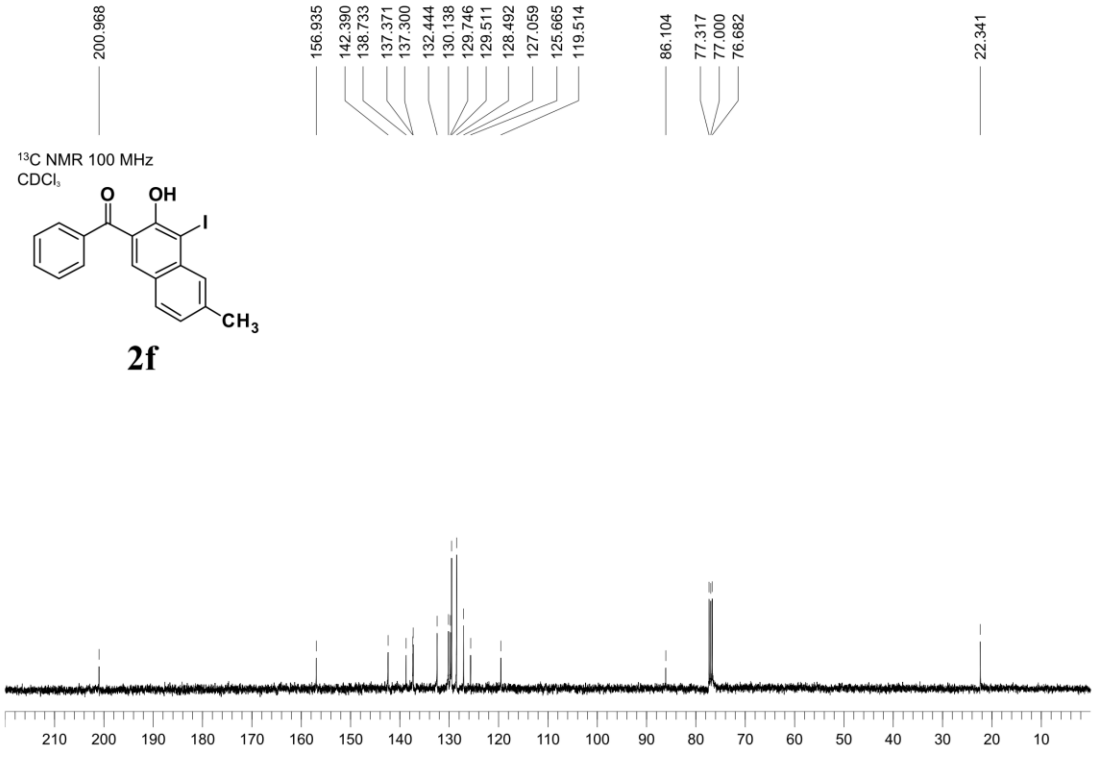
2e


190.577
155.948
141.652
138.309
135.498
135.409
135.095
131.316
130.764
130.066
128.229
127.555
124.838
120.823
86.898
77.317
77.000
76.682
13C NMR 100 MHz
CDCl3
O
OH
I
S
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

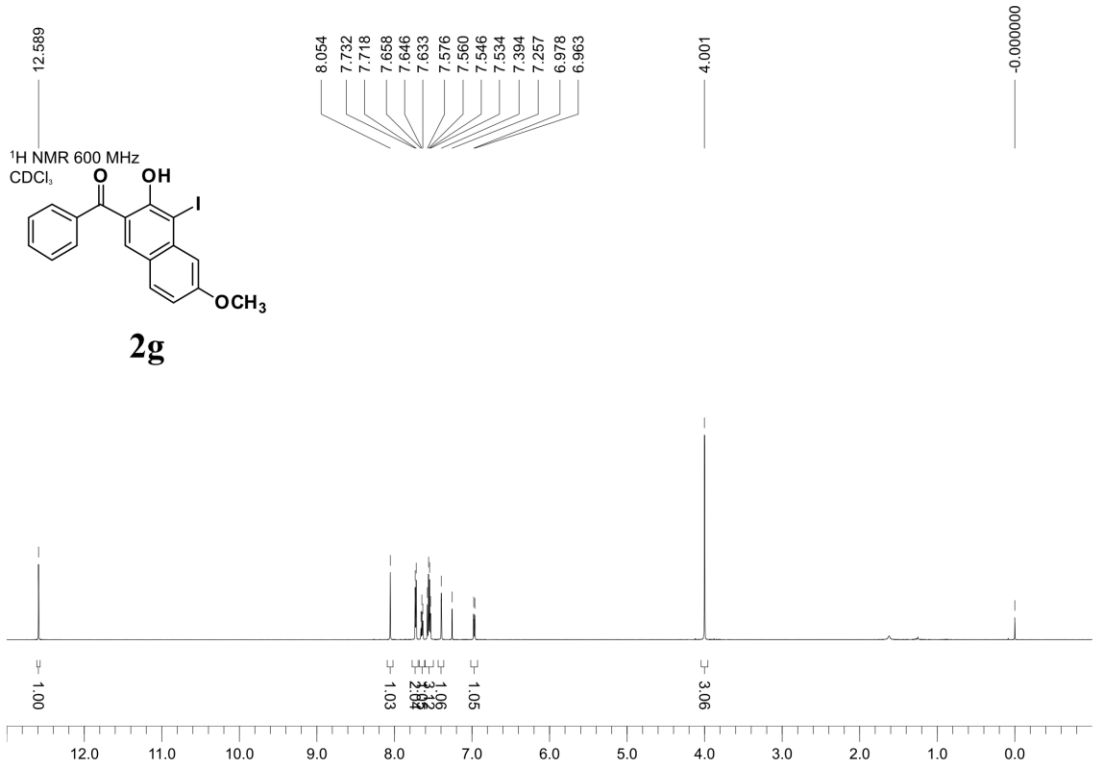
2e


12.348
8.108
7.885
7.744
7.731
7.669
7.657
7.644
7.584
7.570
7.564
7.551
7.539
7.254
7.198
7.184
2.565
0.000000
^{1}H NMR 600 MHz
$CDCl_3$
O
OH
I
CH_3
1.00
1.00
1.03
2.99
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

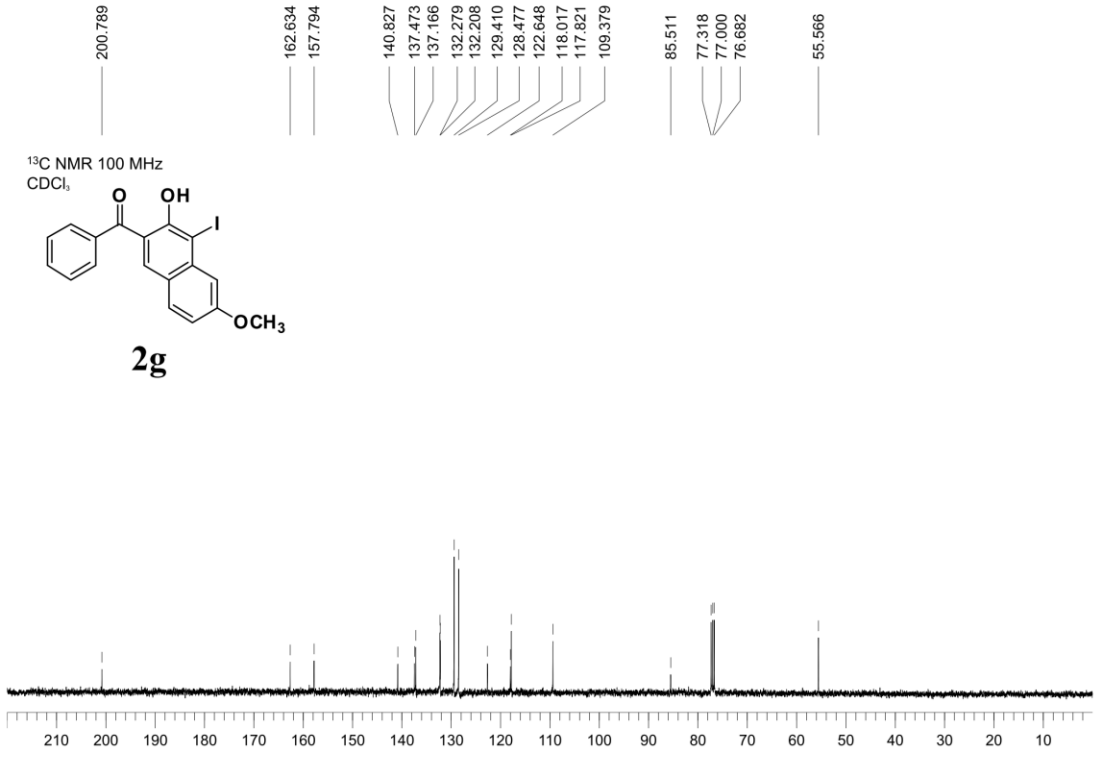
2f


200.968
156.935
142.390
138.733
137.371
137.300
132.444
130.138
129.746
129.511
128.492
127.059
125.665
119.514
86.104
77.317
77.000
76.682
22.341
^{13}C NMR 100 MHz
$CDCl_3$
O
OH
I
CH_3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

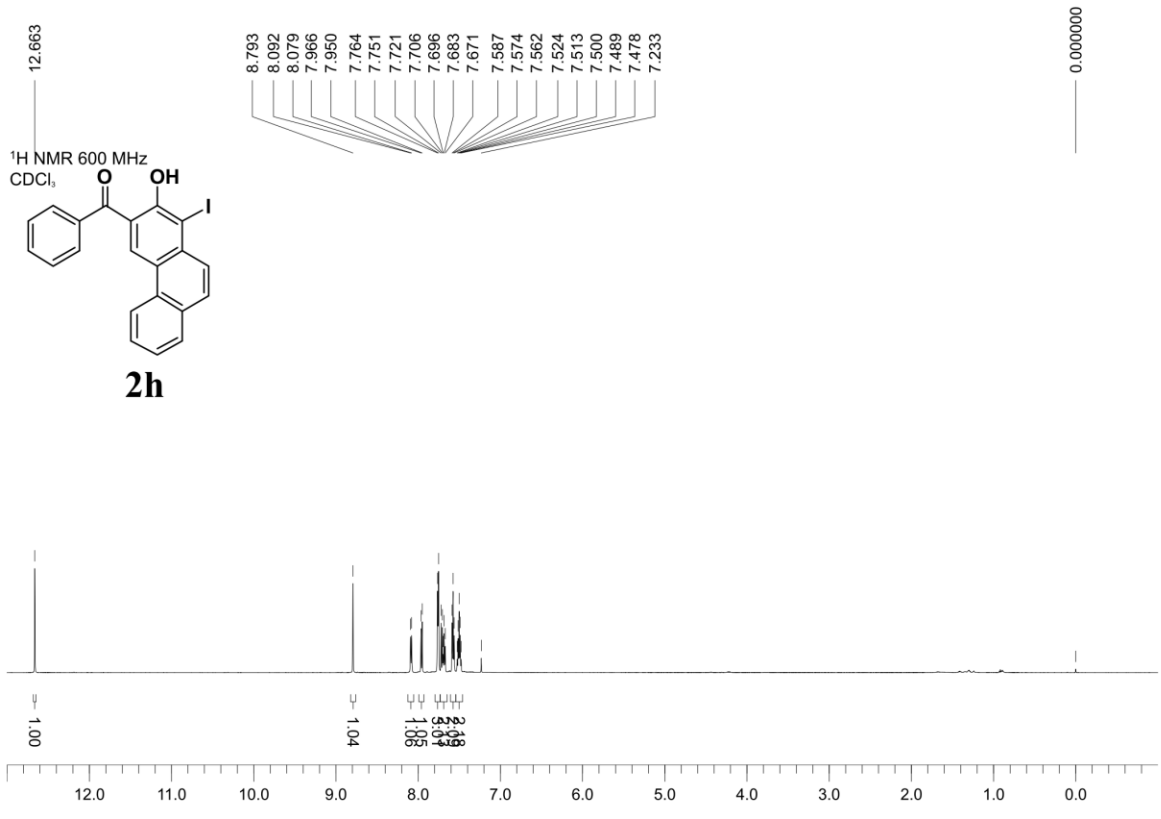
2f

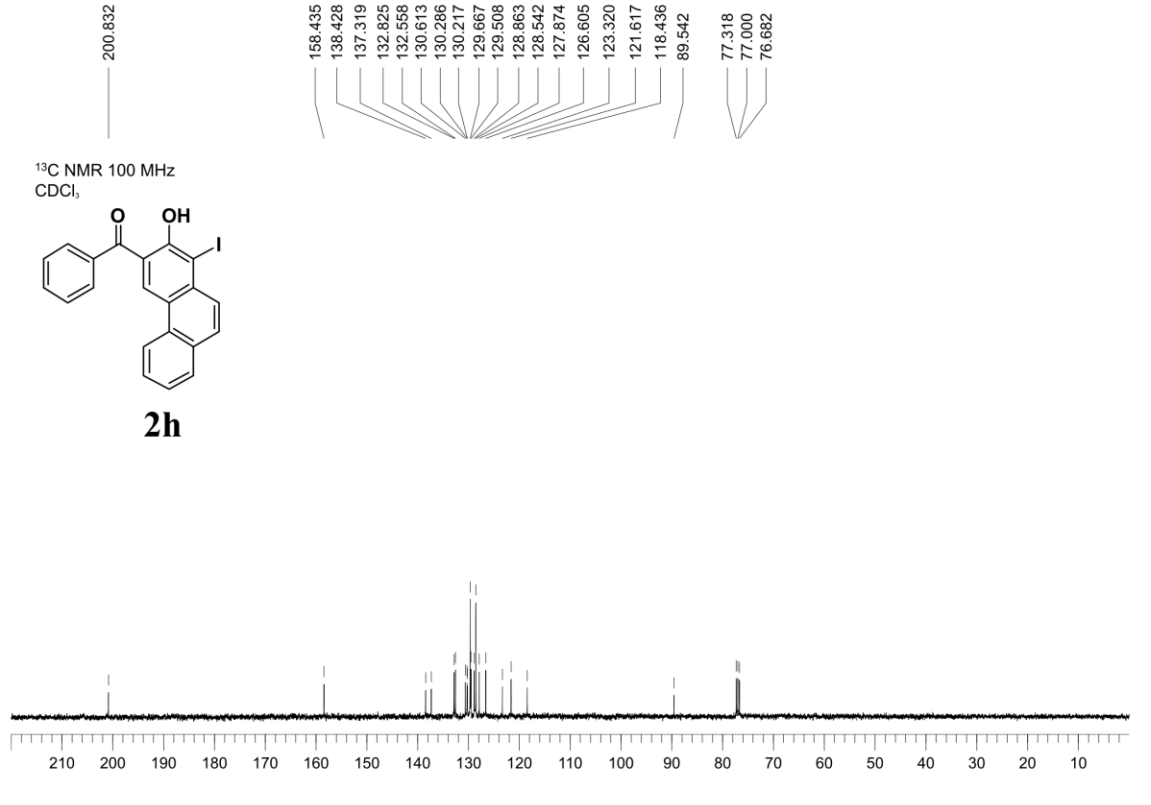

12.589
8.054
7.732
7.718
7.658
7.646
7.633
7.576
7.560
7.546
7.534
7.394
7.257
6.978
6.963
4.001
-0.000000
1H NMR 600 MHz
CDCl3
O
OH
I
OCH3
1.00
1.03
1.06
1.05
3.06
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

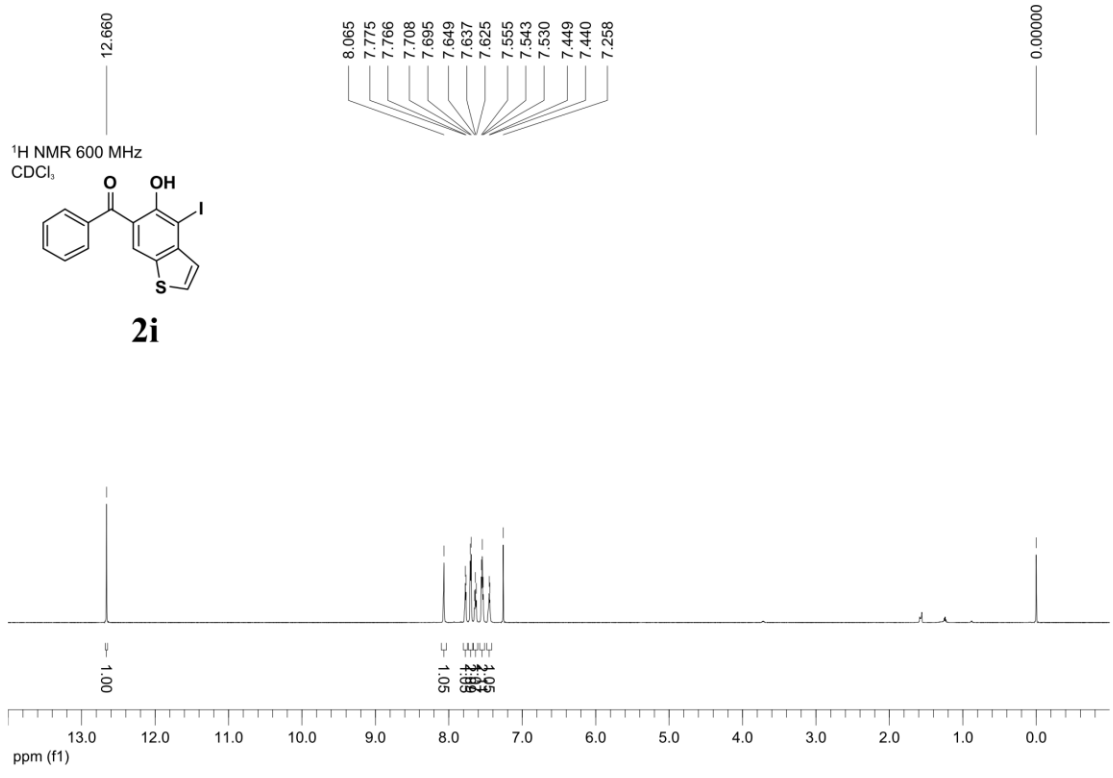
2g

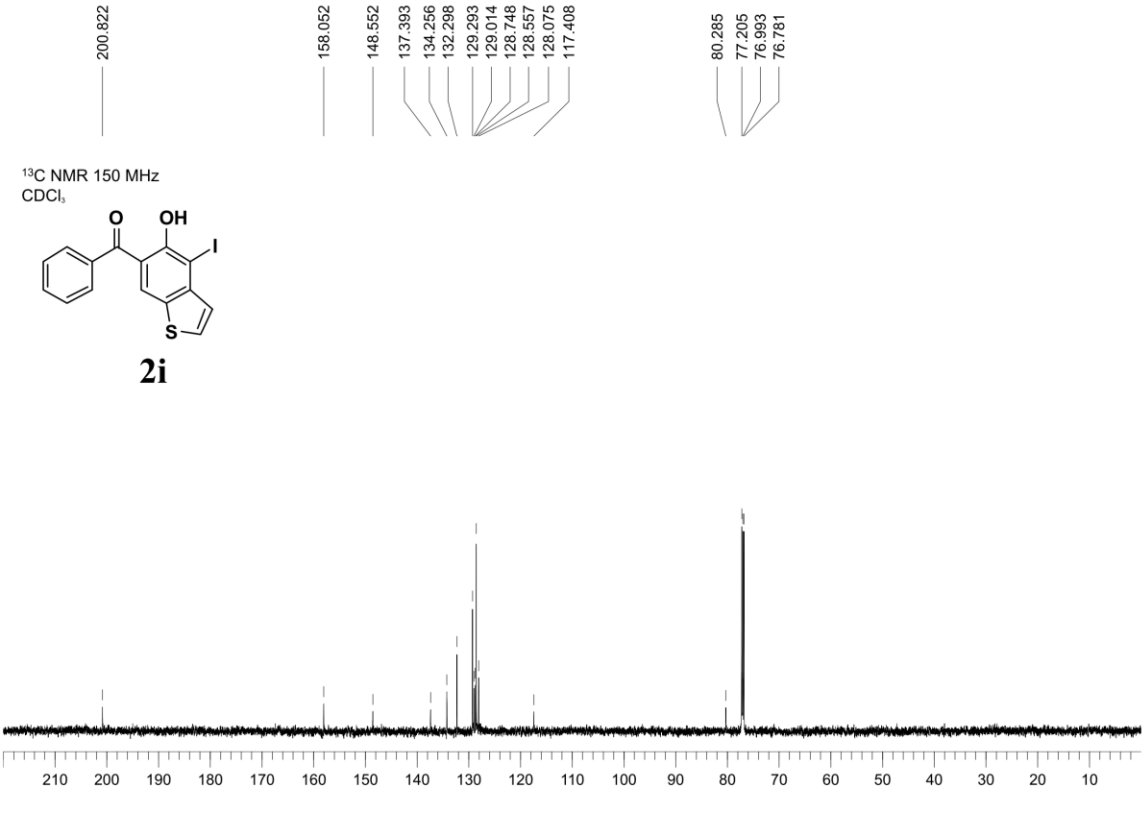

200.789
162.634
157.794
140.827
137.473
137.166
132.279
132.208
129.410
128.477
122.648
118.017
117.821
109.379
85.511
77.318
77.000
76.682
55.566
13C NMR 100 MHz
CDCl3
O
OH
I
OCH3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

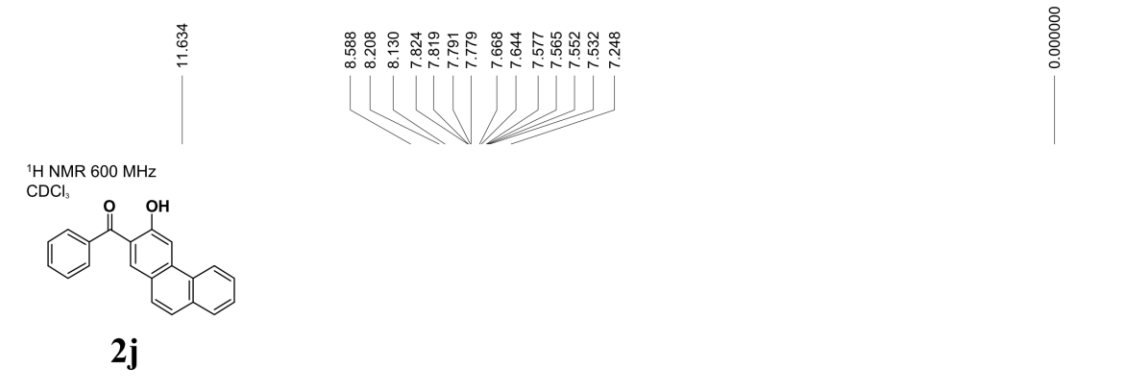
2g


12.663
8.793
8.092
8.079
7.966
7.950
7.764
7.751
7.721
7.706
7.696
7.683
7.671
7.587
7.574
7.562
7.524
7.513
7.500
7.489
7.478
7.233
0.000000
1H NMR 600 MHz
CDCl3
O
OH
I
2h
1.00
1.04
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

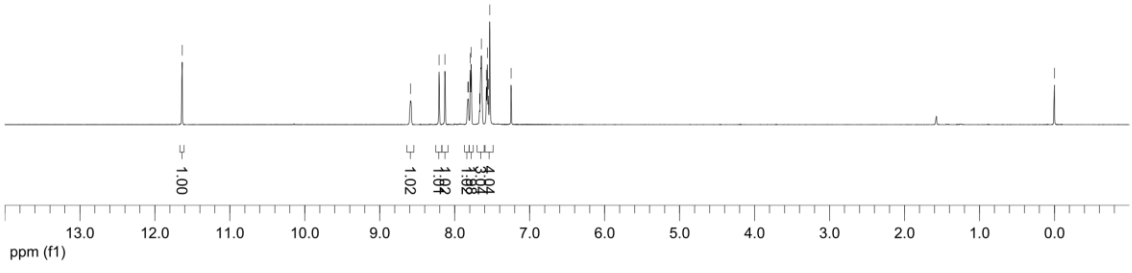

200.832
158.435
138.428
137.319
132.825
132.558
130.613
130.286
130.217
129.667
129.508
128.863
128.542
127.874
126.605
123.320
121.617
118.436
89.542
77.318
77.000
76.682
13C NMR 100 MHz
CDCl3
O
OH
I
2h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

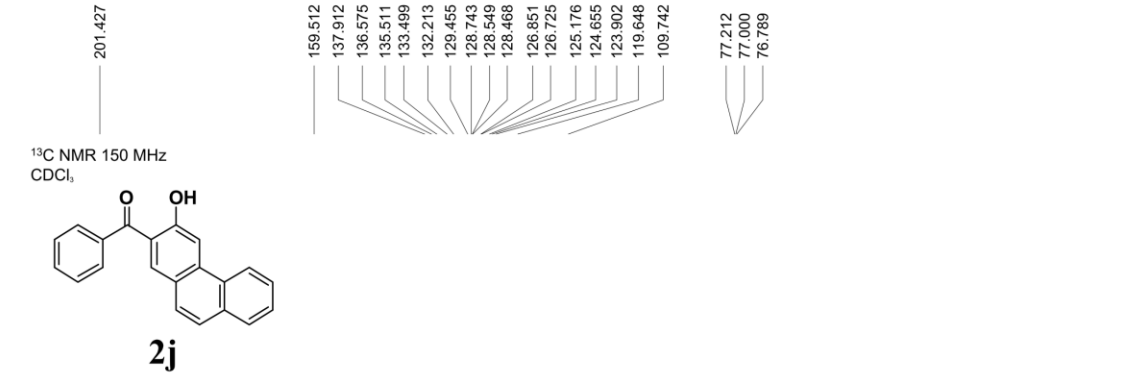

12.660
8.065
7.775
7.766
7.708
7.695
7.649
7.637
7.625
7.555
7.543
7.530
7.449
7.440
7.258
0.00000
1H NMR 600 MHz
CDCl3
O
OH
I
S
2i
1.00
1.05
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)


200.822
158.052
148.552
137.393
134.256
132.298
129.293
129.014
128.748
128.557
128.075
117.408
80.285
77.205
76.993
76.781
13C NMR 150 MHz
CDCl3
O
OH
I
S
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

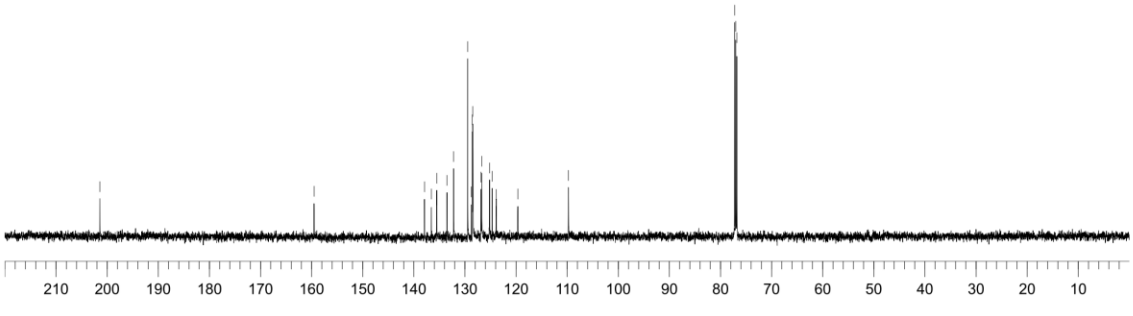

11.634
8.588
8.208
8.130
7.824
7.819
7.791
7.779
7.668
7.644
7.577
7.565
7.552
7.532
7.248
0.000000
1H NMR 600 MHz
CDCl3
O
OH

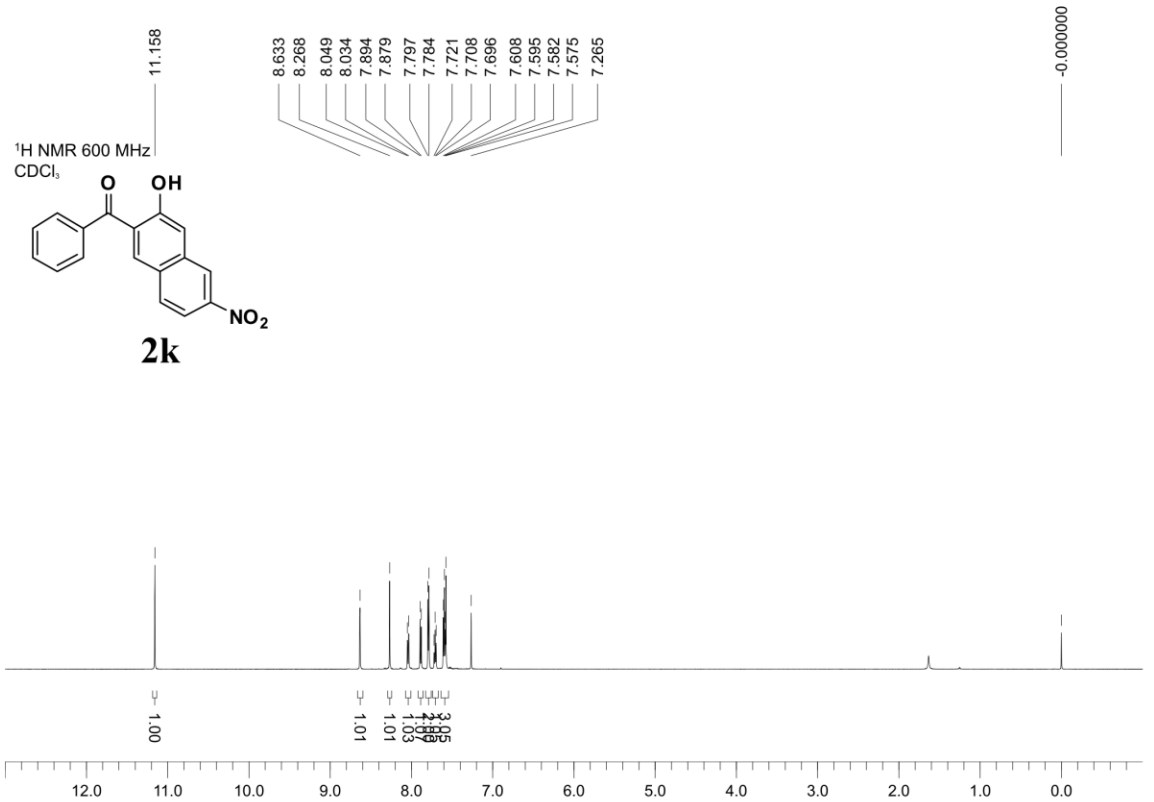
2j

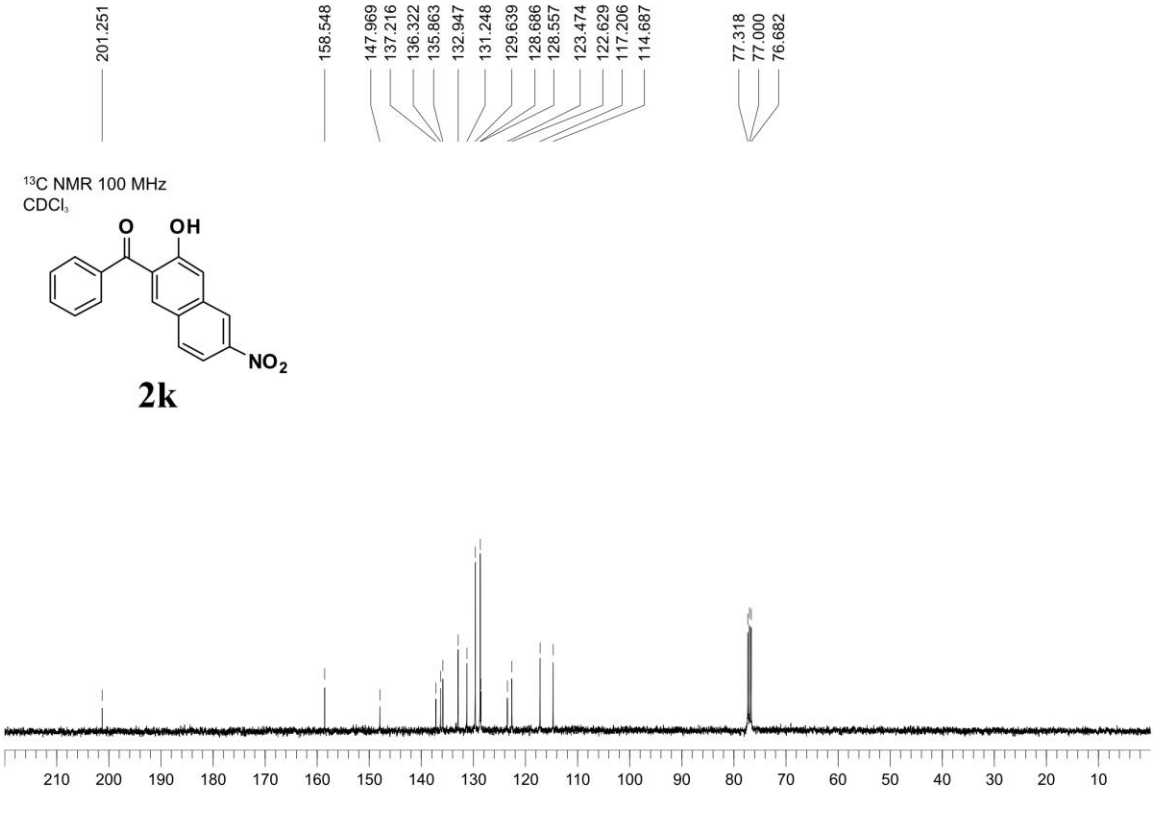

1.00
1.02
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

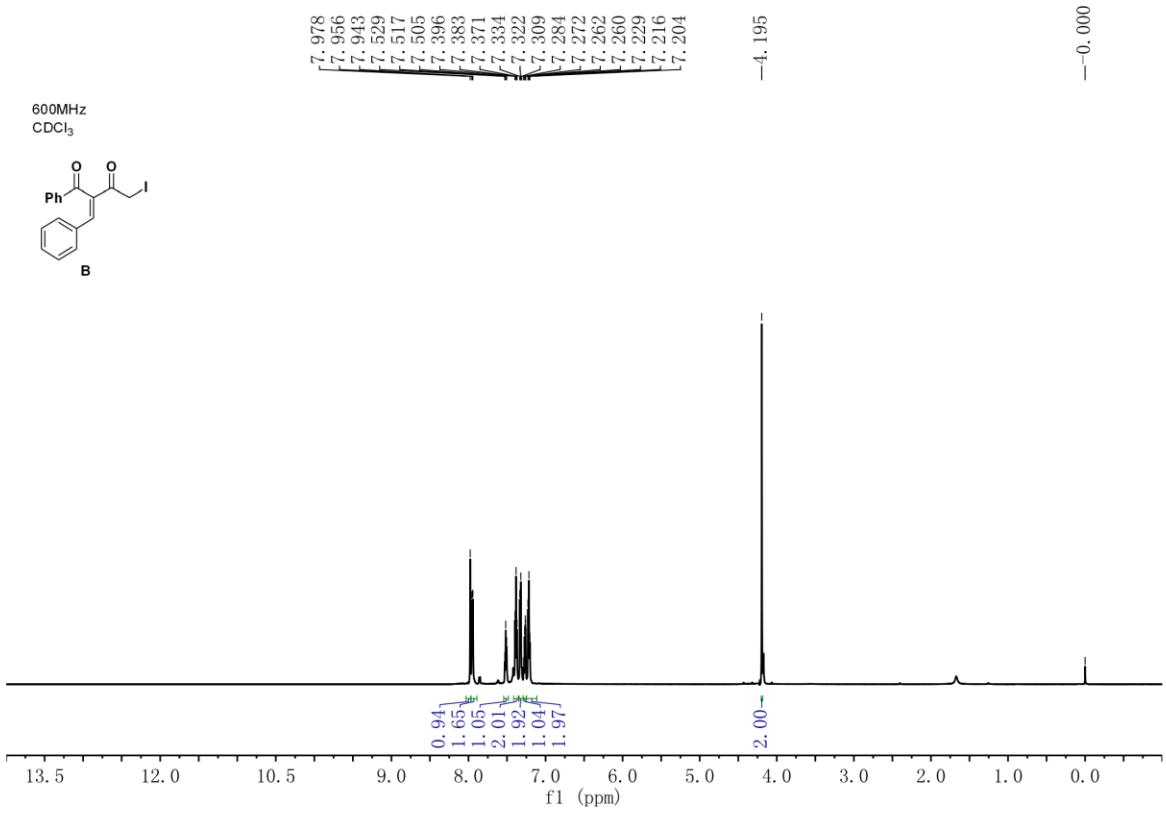

201.427
159.512
137.912
136.575
135.511
133.499
132.213
129.455
128.743
128.549
128.468
126.851
126.725
125.176
124.655
123.902
119.648
109.742
77.212
77.000
76.789
13C NMR 150 MHz
CDCl3
O
OH

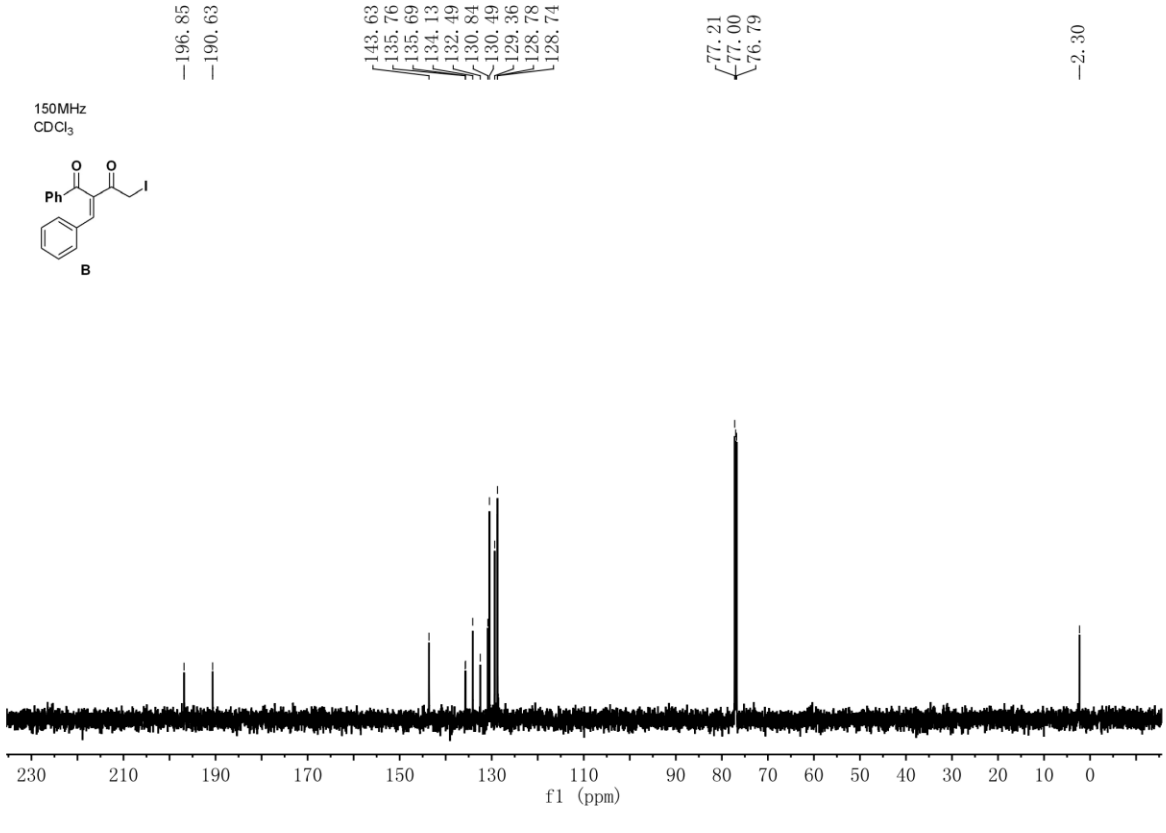
2j


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


11.158
8.633
8.268
8.049
8.034
7.894
7.879
7.797
7.784
7.721
7.708
7.696
7.608
7.595
7.582
7.575
7.265
-0.000000
1H NMR 600 MHz
CDCl3
O
OH
NO2
2k
1.00
1.01
1.01
1.03
3.05
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


201.251
158.548
147.969
137.216
136.322
135.863
132.947
131.248
129.639
128.686
128.557
123.474
122.629
117.206
114.687
77.318
77.000
76.682
13C NMR 100 MHz
CDCl3
O
OH
NO2
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


7.978
7.956
7.943
7.529
7.517
7.505
7.396
7.383
7.371
7.334
7.322
7.309
7.284
7.272
7.262
7.260
7.229
7.216
7.204
4.195
0.000
600MHz
CDCl3
Ph
O
O
I
B
0.94
1.65
1.05
2.01
1.92
1.04
1.97
2.00
13.5
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


196.85
190.63
143.63
135.76
135.69
134.13
132.49
130.84
130.49
129.36
128.78
128.74
77.21
77.00
76.79
2.30
150MHz
CDCl3
Ph
O
O
I
B
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)