The Synthesis of Chromeno[2,3-d]imidazol-9(1H)-ones *via* Tandem Reactions of 3-Iodochromones with Amidines Involving Copper-Catalyzed C-H Functionalization and C-O Bond Formation**

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Supporting Information

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1. Experimental Section

All starting materials and solvents were used as received from commercial suppliers without further purification. All reactions were conducted under air atmosphere and were heated in oil bath, where an external thermometer was employed for temperature control. All ^{1}H NMR and ^{13}C NMR spectra were measured in $CDCl_3$ or DMSO-d_6 or CF_3COOD by Varian Mercury-VX 500M or 400M or 300M NMR spectrometer. Chemical shifts were expressed in ppm and J values were given in Hz. High resolution mass spectra were recorded on Micromass Ultra Q-TOF (ESI) or Thermo – DFS (EI). Column chromatography was performed with 200-300 mesh silica gel using flash column techniques.

General procedure for the synthesis of Imidazochromones (4a-4n)

To a 10mL round bottomed flask was added the iodo-chromone (0.4 mmol), amidines hydrochloride (0.4 mmol), potassium carbonate (1.2 mmol), CuI (0.04 mmol), DMF (3mL) in sequence. This mixture was heated to 120℃ with stirring for 18h under air atmosphere, after which the reaction mixture was cooled down to room temperature, and concentrated in vacuo.

For the instances which used acyclic amidines(4a-4i)

The residue above was diluted with ETDA aqueous solution(0.1M) 25mL, extracted with ethyl acetate (10 mL×3), dichloromethane(10 mL×2). The combined organic layers were washed with brine(10 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo to give the residue, which was further purified by silica gel chromatography (dichloromethane/methanol 100:0 to 20:1) to afford the title product.

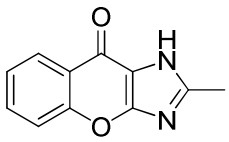
For the instances which used aromatic amidines (4j-4n)

The residue above was diluted with ETDA aqueous solution(0.1M) 25mL, shaked to be uniform ,filtered and washed with water (10 mL), ethyl acetate (10mL), The filter cake was recrystallized by a mixture of solvent (methanol: TFA = 10:1) to give the title product.

General procedure for the synthesis of the other substrates(6a,6c,6e,6f)

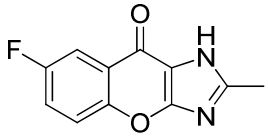
To a 10mL round bottomed flask was added the ring-opening material (0.5 mmol), , potassium carbonate (0.5 mmol), CuI (0.05 mmol), DMF (3 mL) in sequence. This mixture was heated to 120℃ with stirring for 3h under air atmosphere. After the reaction finished, the mixture was cooled down to ambient temperature, and concentrated in vacuo. This residue was diluted with ETDA aqueous solution(0.1M) 25mL, extracted with ethyl acetate (10 mL×3), washed with brine (10 mL), dried over anhydrous Na_2SO_4, filtered and concentrated in vacuo to give the crude product, which was further purified by silica gel chromatography (dichloromethane/methanol 100:0 to 20:1) to afford the title product.

2. Characterization Data



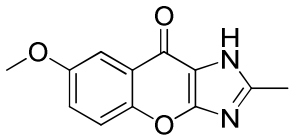
2-methylchromeno[3,2-d]imidazol-9(1H)-one(4a)

As a light yellow powder (64 mg, 82%). m.p. 295-297 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.44 (s, 1H), 8.18 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.81 – 7.72 (m, 1H), 7.71 (d, *J* = 7.9 Hz, 1H), 7.50 – 7.43 (m, 1H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 166.61, 159.82, 154.34, 150.33, 133.52, 125.96, 124.73, 123.81, 118.78, 112.97, 15.12. HRMS (ESI), calcd for $C_{11}H_9N_2O_2$ (M+H) 201.0664, found: 201.0662.



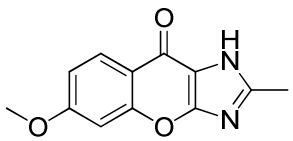
7-fluoro-2-methylchromeno[3,2-d]imidazol-9(1H)-one(4b)

As a white powder (49 mg, 57%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 7.95 – 7.87 (m, 1H), 7.63 (dd, *J* = 9.2, 3.4 Hz, 1H), 7.51 (t, *J* = 6.8 Hz, 1H), 2.80 (s, 3H). ^{13}C NMR (101 MHz, CF_3COOD) δ 170.70, 162.70 (d, $J_{C\text{-}F}$=250 Hz), 153.48, 152.54, 150.57, 127.11 (d, $J_{C\text{-}F}$=26 Hz), 125.62(d, $J_{C\text{-}F}$=8.1Hz), 122.83 (d, $J_{C\text{-}F}$=8.3 Hz), 114.12, 113.87, 13.63. HRMS (ESI), calcd for $C_{11}H_7N_2O_2FNa$ (M+Na) 241.0389, found: 241.0392.



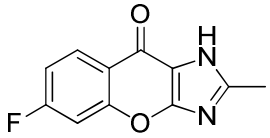
7-methoxy-2-methylchromeno[3,2-d]imidazol-9(1H)-one(4c)

As a white powder (36mg, 40%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 7.91 (d, *J* = 2.9 Hz, 1H), 7.76 (d, *J* = 9.3 Hz, 1H), 7.64 (dd, *J* = 9.3, 3.0 Hz, 1H), 4.05 (s, 3H), 3.00 (s, 3H). ^{13}C NMR (101 MHz, CF_3COOD) δ 171.22, 160.47, 152.46, 152.22, 150.24, 128.44, 125.02, 122.11, 114.11, 109.11, 57.85, 13.57. HRMS (ESI), calcd for $C_{12}H_{11}N_2O_3$ (M+H) 231.0770, found: 231.0770.



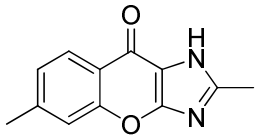
6-methoxy-2-methylchromeno[3,2-d]imidazol-9(1H)-one(4d)

As a white powder (76mg, 83%). decompose at 300 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.35 (s, 1H), 8.05 (d, *J* = 8.9 Hz, 1H), 7.22 (d, *J* = 2.4 Hz, 1H), 7.03 (dd, *J* = 8.9, 2.4 Hz, 1H), 3.89 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 166.46, 163.46, 159.64, 156.11, 149.37, 127.10, 117.29, 113.45, 112.61, 101.66, 56.40, 14.99. HRMS (ESI), calcd for $C_{12}H_{11}N_2O_3$ (M+H) 231.0770, found: 231.0770.



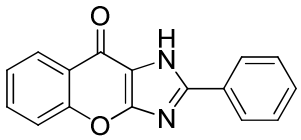
6-fluoro-2-methylchromeno[2,3-d]imidazol-9(1H)-one (4e)

As a light yellow powder (36mg, 42%). m.p. >300 °C. ^{1}H NMR (500 MHz, DMSO) δ 8.21 (m, 1H), 7.67 (d, J = 8.9 Hz, 1H), 7.34 (m, 1H), 2.44 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 165.81, 164.60 (d, $J_{C\text{-}F}$ = 249.8 Hz), 160.05, 155.25, 155.14, 150.34, 128.35 (d, $J_{C\text{-}F}$ = 10.7 Hz), 120.83, 112.97 (d, $J_{C\text{-}F}$ = 22.5 Hz), 105.57 (d, $J_{C\text{-}F}$ = 26.2 Hz), 15.08. HRMS (ESI), calcd for $C_{11}H_8N_2O_2F$ (M+H) 219.0570, found: 219.0573.



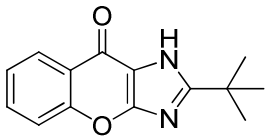
2,6-dimethylchromeno[3,2-d]imidazol-9(1H)-one(4f)

As a white powder (55mg, 65%). decompose at 300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.17 (d, J = 8.2 Hz, 1H), 7.43 (s, 1H), 7.36 (d, J = 8.1 Hz, 1H), 2.80 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (126 MHz, CF_3C OOD) δ 171.52, 157.60, 152.86, 152.07, 149.71, 131.04, 128.43, 121.51, 120.23, 114.21, 22.59, 13.41. HRMS (ESI), calcd for $C_{12}H_{11}N_2O_2$ (M+H) 215.0821, found: 215.0822.



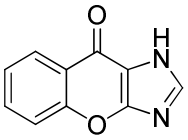
2-phenylchromeno[3,2-d]imidazol-9(1H)-one(4g)

As a light yllow powder (73mg, 70%). m.p. >300 °C. ^{1}H NMR (400 MHz, DMSO) δ 14.08 (s, 1H), 8.20 (dd, J = 7.7, 6.6 Hz, 3H), 7.77 (dd, J = 11.1, 4.1 Hz, 1H), 7.71 (d, J = 8.1 Hz, 1H), 7.61 – 7.40 (m, 4H). ^{13}C NMR (101 MHz, DMSO) δ 167.17, 160.00, 154.49, 149.15, 133.77, 131.14, 129.49, 129.00, 127.17, 126.04, 124.86, 123.91, 118.80, 114.37. HRMS (ESI), calcd for $C_{16}H_{10}N_2O_2Na$ (M+Na) 285.0640, found: 285.0641.



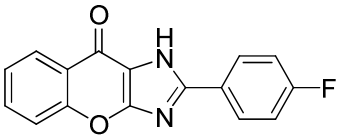
2-tert-butylchromeno[3,2-d]imidazol-9(1H)-one(4h)

As a white powder (85mg, 88%). m.p. 298-300°C. ^{1}H NMR (400 MHz, DMSO) δ 13.30 (s, 1H), 8.20 (d, J = 7.9 Hz, 1H), 7.77 (dd, J = 7.7, 8.0 Hz, 1H), 7.69 (d, J = 8.3 Hz, 1H), 7.47 (dd, J = 7.4, 7.8 Hz, 1H), 1. 37 (s, 9H). ^{13}C NMR (101 MHz, DMSO) δ 166.95, 161.03, 159.18, 154.38, 133.54, 126.03, 124.69, 123.8 3, 118.71, 113.26, 34.09, 29.30. HRMS (ESI), calcd for $C_{14}H_{14}N_2O_2Na$ (M+Na) 265.0953, found: 265.09 53.



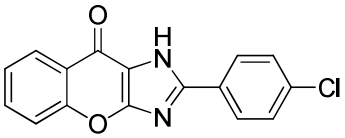
chromeno[3,2-d]imidazol-9(1H)-one(4i)

As a white powder (54mg, 73%). m.p. 268-270 °C. ^{1}H NMR (400 MHz, DMSO) δ 13.82 (s, 1H), 8.23 (s, 1H), 8.21 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.85 – 7.76 (dd, *J* = 7.7, 8.1 Hz, 1H), 7.74 (d, *J* = 8.2 Hz, 1H), 7.49 (d d, *J* = 7.4, 7.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 167.75, 159.62, 154.64, 140.25, 133.93, 126.07, 1 24.83, 123.66, 118.87, 112.88. HRMS (ESI), calcd for $C_{10}H_7N_2O_2$ (M+H) 187.0508, found: 187.0507.



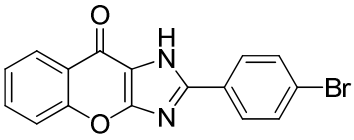
2-(4-fluorophenyl)chromeno[3,2-d]imidazol-9(1H)-one(4j)

As a white powder (70mg, 63%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.44 (d, *J* = 8.0 Hz, 1 H), 8.14 (m, 2H), 7.93 (dd, *J* = 7.8, 7.4 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.64 (dd, *J* = 7.5, 7.9 Hz, 1H), 7. 34 (m, 2H). ^{13}C NMR (101 MHz, CF_3COOD) δ 171.63, 169.94 (d, $J_{C\text{-}F}$ = 260.1 Hz), 157.47, 153.40, 148. 20, 139.22, 132.78 (d, $J_{C\text{-}F}$ = 10.1 Hz), 129.70, 128.88, 124.08, 120.50 (d, $J_{C\text{-}F}$= 10.6 Hz), 120.21, 118.39, 114.83. HRMS (ESI), calcd for $C_{16}H_{10}N_2O_2F$ (M+H) 281.0726, found: 281.0724.



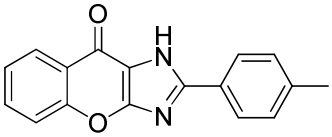
2-(4-chlorophenyl)chromeno[3,2-d]imidazol-9(1H)-one (4k)

As a white powder (70mg, 60%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.43 (d, *J* = 7.6 Hz, 1 H), 8.02 (d, *J* = 7.2 Hz, 2H), 7.92 (dd, *J* = 8.0,7.6 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.63 (m, 3H). ^{13}C NMR (101 MHz, CF_3COOD) δ 171.70, 157.55, 153.51, 148.24, 145.91, 139.34, 133.26, 131.06, 129. 79, 128.97, 124.15, 120.62, 120.48, 115.00. HRMS (ESI), calcd for $C_{16}H_{10}N_2O_2Cl$ (M+H) 297.0431, foun d: 297.0430.



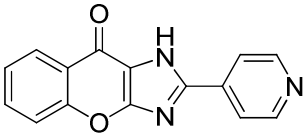
2-(4-bromophenyl)chromeno[3,2-d]imidazol-9(1H)-one (4l)

As a white powder (75mg, 58%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.66 (d, *J* = 7.8 Hz, 1 H), 8.16 (m, 3H), 8.03 (d, *J* = 8.1 Hz, 2H), 7.96 (d, *J* = 8.3 Hz, 1H), 7.86 (dd, *J* = 7.5, 8.0 Hz, 1H). ^{13}C N MR (101 MHz,CF_3COOD) δ 171.58, 157.42, 153.42, 148.28, 139.21, 136.24, 134.05, 130.78, 129.66, 12 8.84, 124.00, 120.79, 120.50, 114.88. HRMS (ESI), calcd for $C_{16}H_{10}N_2O_2Br$ (M+H) 340.9926, found: 34 0.9928.



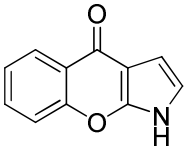
2-p-tolylchromeno[3,2-d]imidazol-9(1H)-one (4m)

As a whie powder (77mg, 70%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.42 (d, *J* = 7.7 Hz, 1 H), 7.95 (d, *J* = 7.9 Hz, 2H), 7.90 (dd, *J* = 8.0,7.8 Hz, 1H), 7.72 (d, *J* = 8.5 Hz, 1H), 7.62 (dd, *J* = 7.8, 8.0 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CF_3COOD) δ 171.68, 157.48, 153.32, 151.20, 149.44, 139.14, 133.51, 129.80, 129.71, 128.92, 124.28, 120.58, 118.96, 114.68, 22.66. HRMS (ESI), calcd for $C_{17}H_{13}N_2O_2$ (M+H) 277.0977, found: 277.0983.



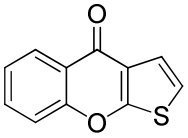
2-(pyridin-4-yl)chromeno[3,2-d]imidazol-9(1H)-one (4n)

As a whie powder (47mg, 45%). m.p. >300 °C. ^{1}H NMR (400 MHz, CF_3COOD) δ 8.97 (d, *J* = 6.1 Hz, 2 H), 8.74 (d, *J* = 6.0 Hz, 2H), 8.37 (d, *J* = 8.0 Hz, 1H), 7.86 (dd, *J* = 7.8, 8.0 Hz, 1H), 7.72 (d, *J* = 8.5 Hz, 1 H), 7.54 (dd, *J* = 7.7, 7.9 Hz, 1H). ^{13}C NMR (101 MHz, CF_3COOD) δ 173.48, 162.60, 158.35, 146.89, 145.67, 145.15, 139.07, 128.70, 128.61, 127.03, 123.98, 121.17, 121.04. HRMS (ESI), calcd for $C_{15}H_{10}N_3O_2$ (M+H) 264.0773, found: 264.0781.



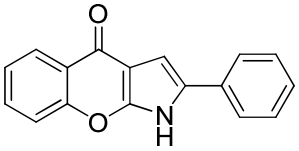
chromeno[2,3-b]pyrrol-4(1H)-one (6a)

As a gray powder (63mg, 68%). decompose at 280 °C. ^{1}H NMR (300 MHz, CD3OD) δ 8.30 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.80 – 7.70 (m, 1H), 7.61 (d, *J* = 8.5 Hz, 1H), 7.47 (dd, *J* = 7.5, 7.8 Hz, 1H), 6.82 (d, *J* = 3.7 Hz, 1H), 6.62 (d, *J* = 3.7 Hz, 1H). ^{13}C NMR (126 MHz, DMSO) δ 172.32, 153.94, 150.36, 133.22, 126.21, 124.70, 123.01, 117.95, 116.23, 105.94, 102.07. HRMS (ESI), calcd for $C_{11}H_8NO_2$ (M+H) 186.0555, found: 186.0552.



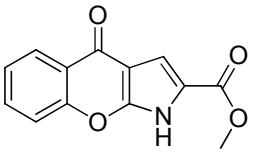
4H-thieno[2,3-b]chromen-4-one (6c)

As a light yellow powder (20mg, 20%). m.p. 160-162 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.71 (ddd, *J* = 8.7, 7.1, 1.7 Hz, 1H), 7.54 (dd, *J* = 8.6, 0.9 Hz, 1H), 7.50 – 7.41 (m, 2H), 6.92 (d, *J* = 6.0 Hz, 1H). ^{13}C NMR (126 MHz, DMSO) δ 172.30, 168.04, 156.71, 134.68, 126.32, 125.69, 123.29, 122.26, 120.93, 118.38, 117.98. HRMS (EI), calcd for $C_{11}H_6SO_2$ (M^+) 202.0089, found: 202.0085.



2-phenylchromeno[2,3-b]pyrrol-4(1H)-one (6e)

As a white powder (68mg, 52%). m.p. >300 °C. ^{1}H NMR (500 MHz, DMSO) δ 12.88 (s, 1H), 8.22 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 2H), 7.78 (s, 1H), 7.70 (s, 1H), 7.50 (dd, *J* = 7.1 ,7.3Hz, 1H), 7.44 (dd, *J* = 7.7, 7.9 Hz, 2H), 7.29 (dd, *J* = 7.4 ,7.6 Hz, 1H), 7.01 (s, 1H). ^{13}C NMR (126 MHz, DMSO) δ 17 2.06, 154.06, 151.89, 133.35, 131.62, 129.39, 129.28, 127.62, 126.35, 124.99, 124.66, 123.29, 118.16, 10 7.36, 99.04. HRMS (ESI), calcd for $C_{17}H_{12}NO_2$ (M+H) 262.0868, found: 262.0861.



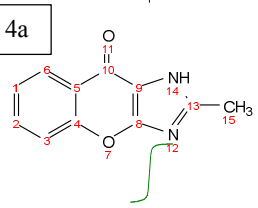
methyl 4-oxo-1,4-dihydrochromeno[2,3-b]pyrrole-2-carboxylate (6f)

As a white powder (30mg, 23%). m.p. 276-278 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.33 (s, 1H), 8.38 (d, *J* = 7.8 Hz, 1H), 7.69 (dd, *J* = 8.0, 7.8 Hz, 1H), 7.49 (d, *J* = 8.1 Hz, 1H), 7.43 (dd, *J* = 7.4, 7.8 Hz, 1H), 7.40 (s, 1H), 3.93 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 172.83, 160.97, 154.30, 151.69, 134.13, 126.33, 12 5.19, 122.69, 119.34, 118.14, 109.27, 107.19, 52.23. HRMS (ESI), calcd for $C_{13}H_{10}NO_4$ (M+H) 244.0610, found: 244.0602.

3. ^{1}H and ^{13}C NMR Spectra

H yimi
CB7148 1H

4a

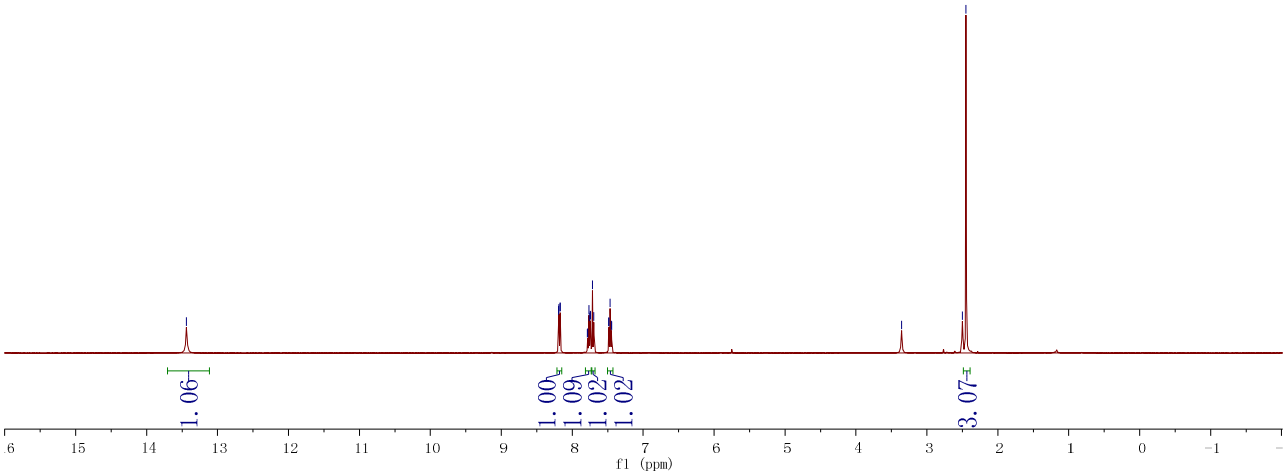


13.44

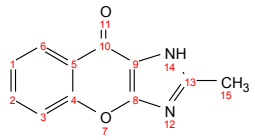
8.19
8.19
8.17
8.17
7.78
7.78
7.76
7.76
7.75
7.74
7.72
7.70
7.48
7.47
7.45
7.45

3.36

2.50
2.45



H yimi
CB7148 DMSO BB+DEPT135

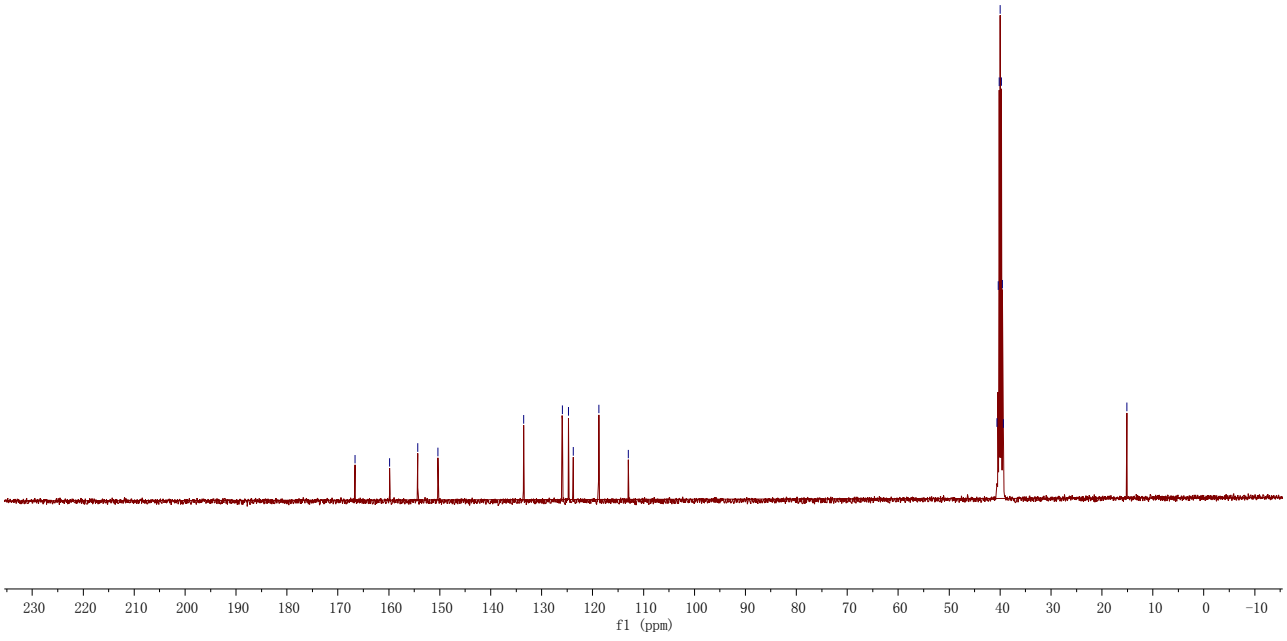


166.6
159.8
154.3
150.3

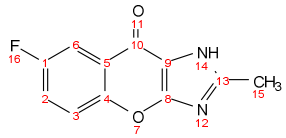
133.5
126.0
124.7
123.8
118.8
113.0

40.6
40.4
40.2
40.0
39.8
39.5
39.3

15.1

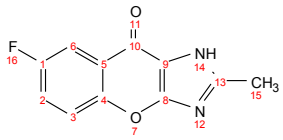


SJ-6Fyi
SJ 6Fyi CF3COOD 1H 20130222

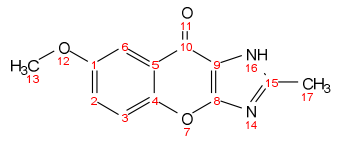


4b

SJ-6FYi_bb130222
SJ-6FYi TFA BB+DEPT-135

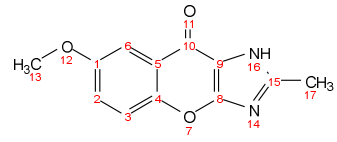


SJ-60MeY
SJ-60MeY CF3COOH

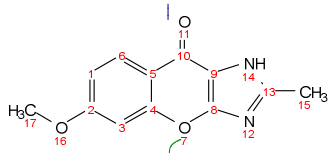


4c

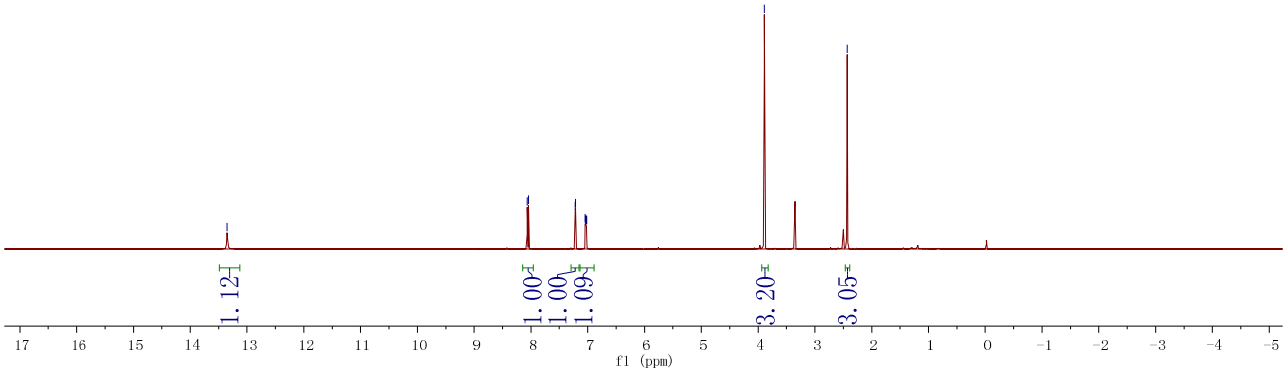
SJ 60MeYi bb130221
SJ-60MeYi TFA BB+DEPT-135



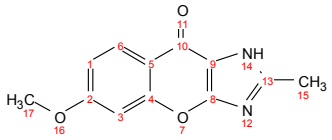
CB9026C
CB9026C DMSO 1H Jul 5 2013

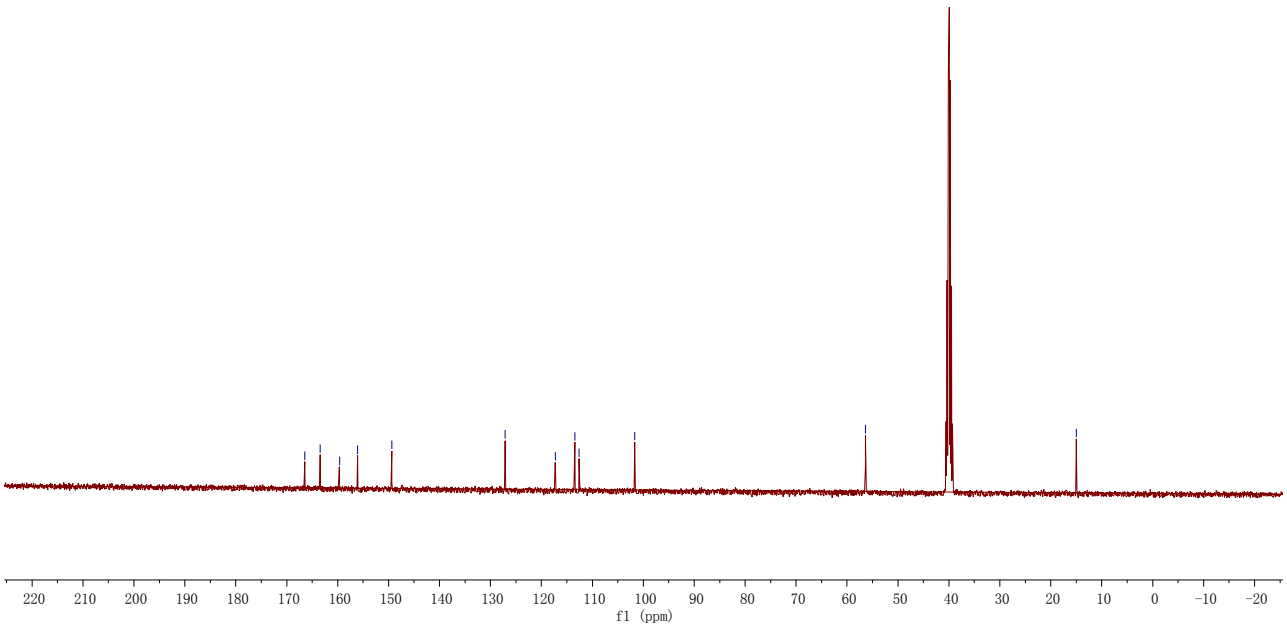


4d

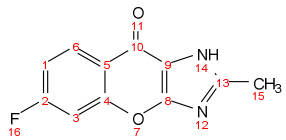


7omeyi
CB9026C DMSO BB+DEPT135





SJ-7Fyi
SJ 7Fyi DMSO 1H



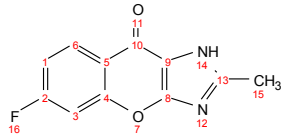
4e

8.2
7.7
7.6
7.3
2.4

1.00
1.06
1.04
2.80

f1 (ppm)

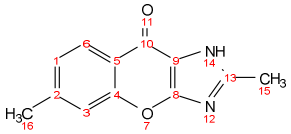
SJ-7Fyi
SJ-7Fyi DMSO BB+DEPT135



165.8
165.6
163.6
160.0
155.2
155.1
150.3
128.4
128.3
120.8
113.1
112.9
105.7
105.5
40.3
40.2
40.0
39.8
39.7
39.5
39.3
15.1

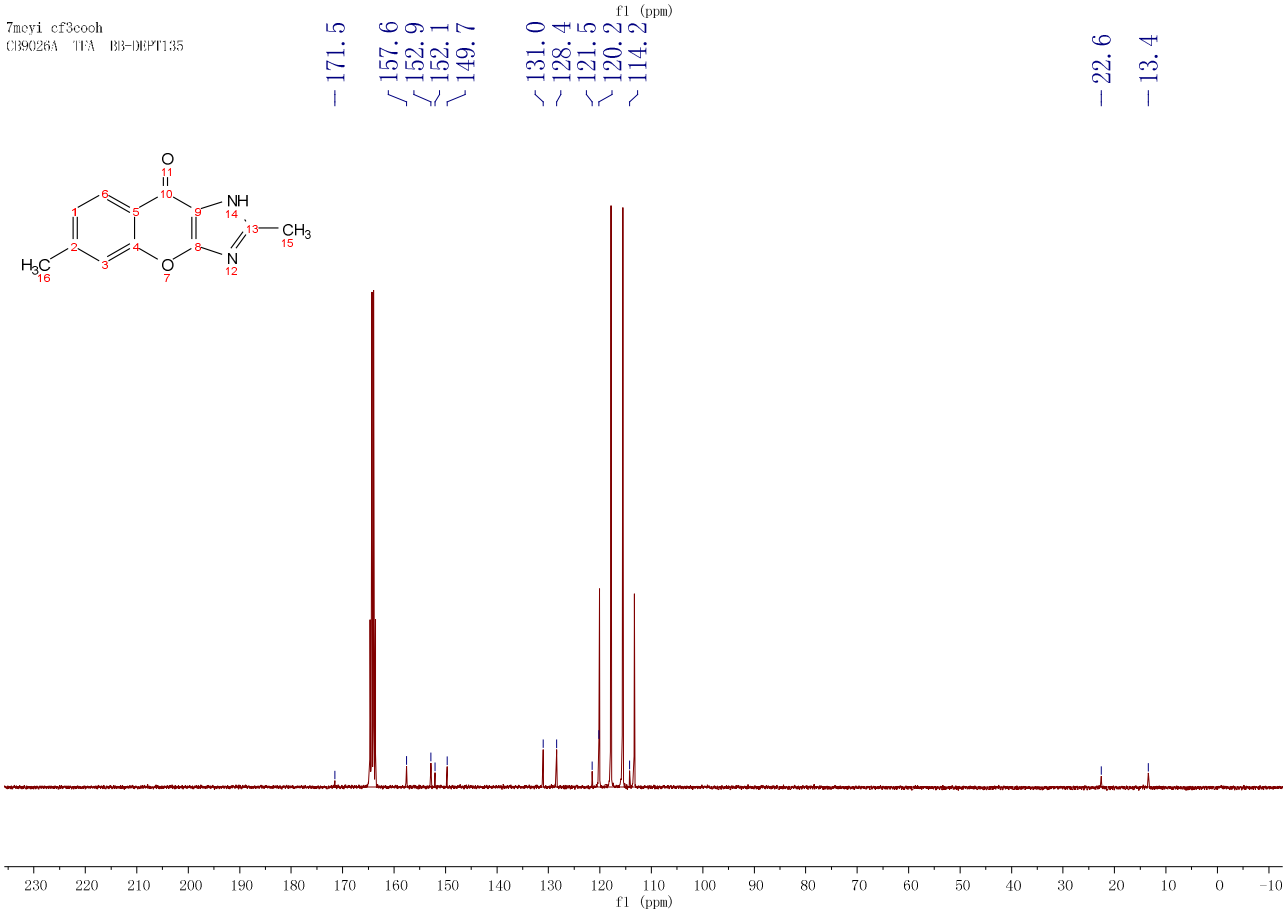
f1 (ppm)

CB9026A
CB9026A CF3COOD 20120705

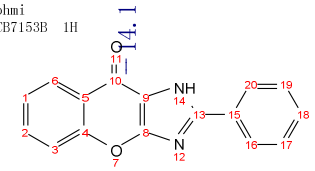


4f

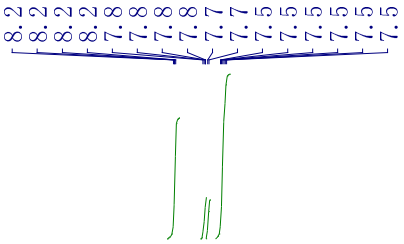
7meyi cf3cooh
CB9026A TFA BB-DEPT135

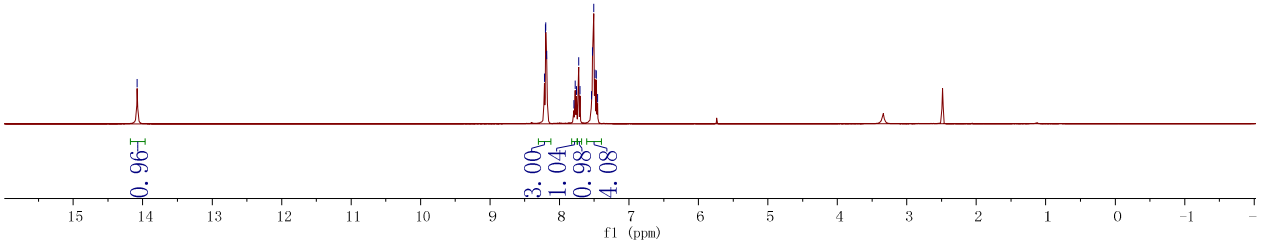


phmi
CB7153B 1H

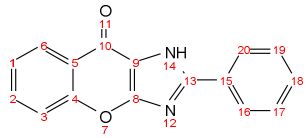


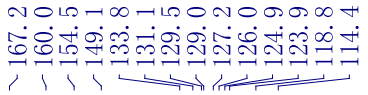
4g

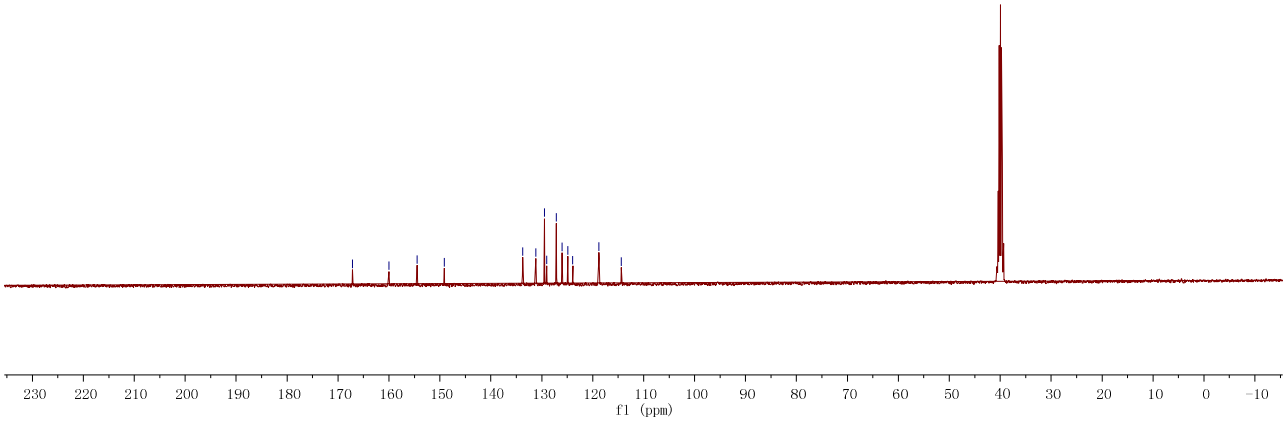




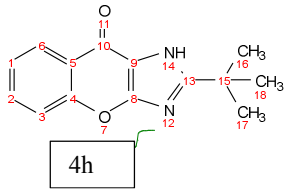
phmi
CB7153B DMSO BB+DEPT135





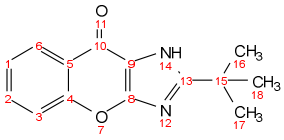


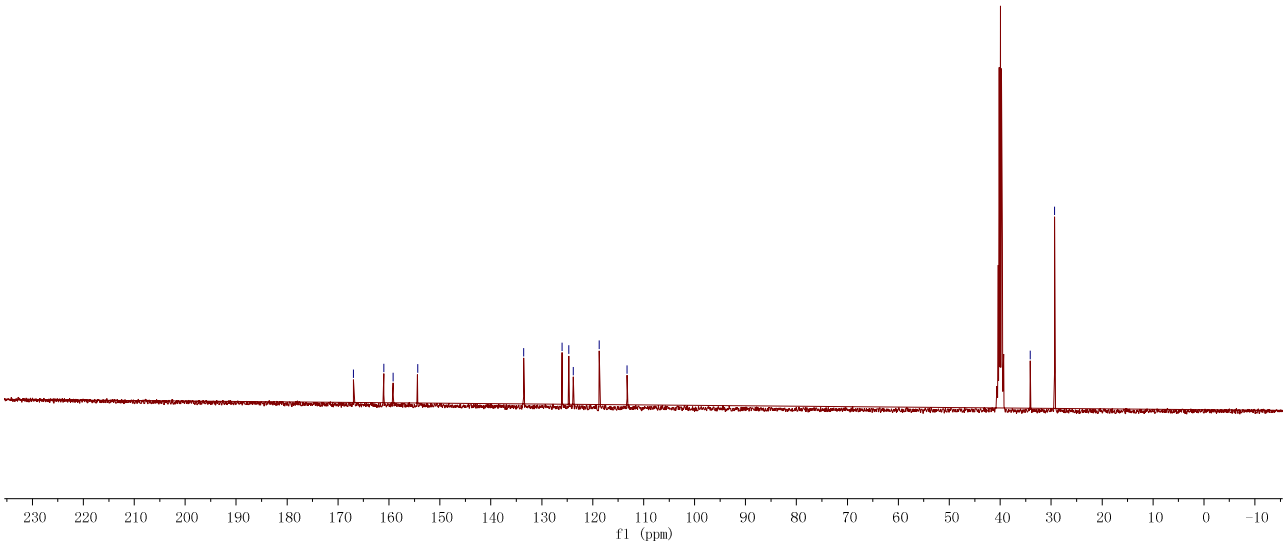
t-bu
CB7154A 1H



4h

t-bu
CB7154A DMSO BB+DEPT135

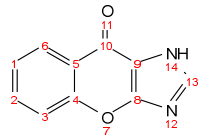




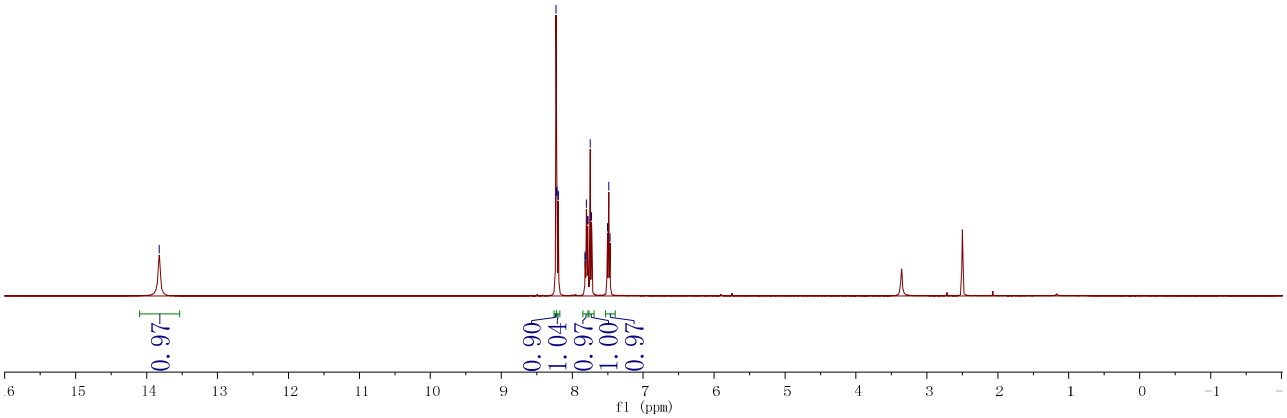
h h
CB7155A 1H

13.8

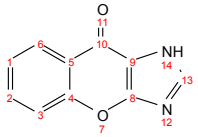
8.2 8.2 8.2 8.2 8.2 7.8 7.8 7.8 7.8 7.8 7.7 7.7 7.5 7.5 7.5



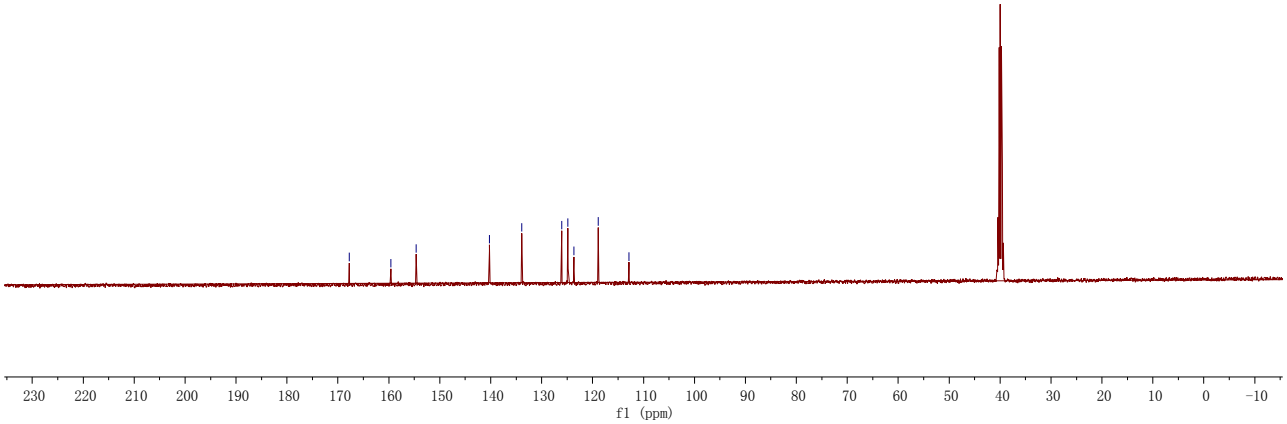
4i



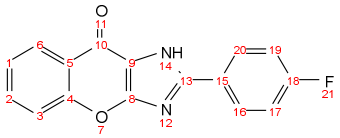
h h
CB7155A DMSO BB+DEPT135



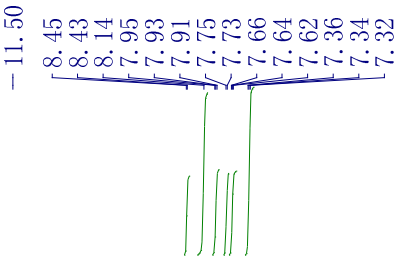
167.7
159.6
154.6
140.2
133.9
126.1
124.8
123.7
118.9
112.9

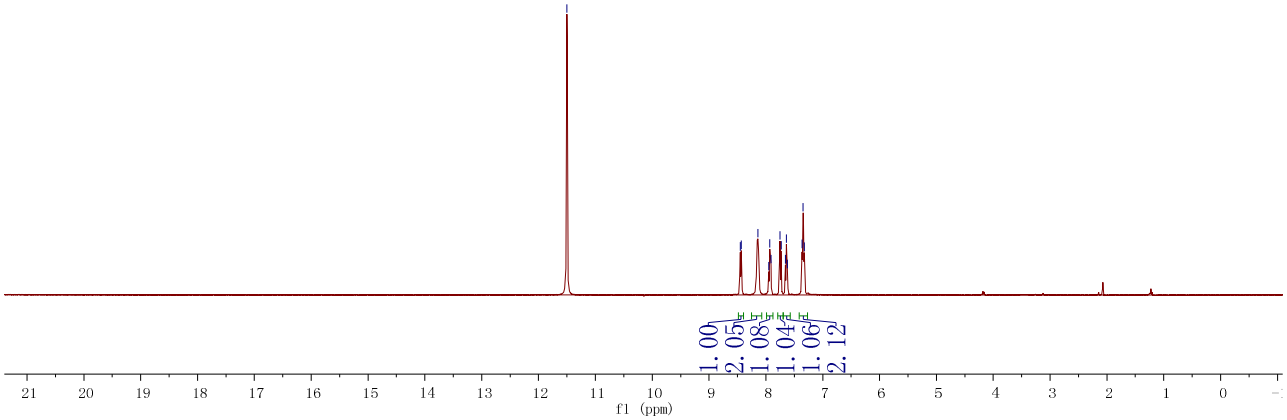


SJ-F2
SJ-F2 CF3COOD 1H 20121128

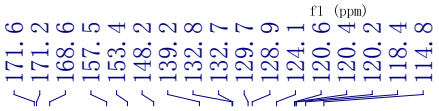


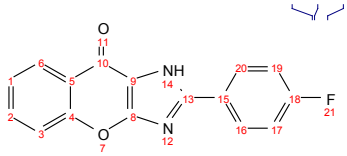
4j

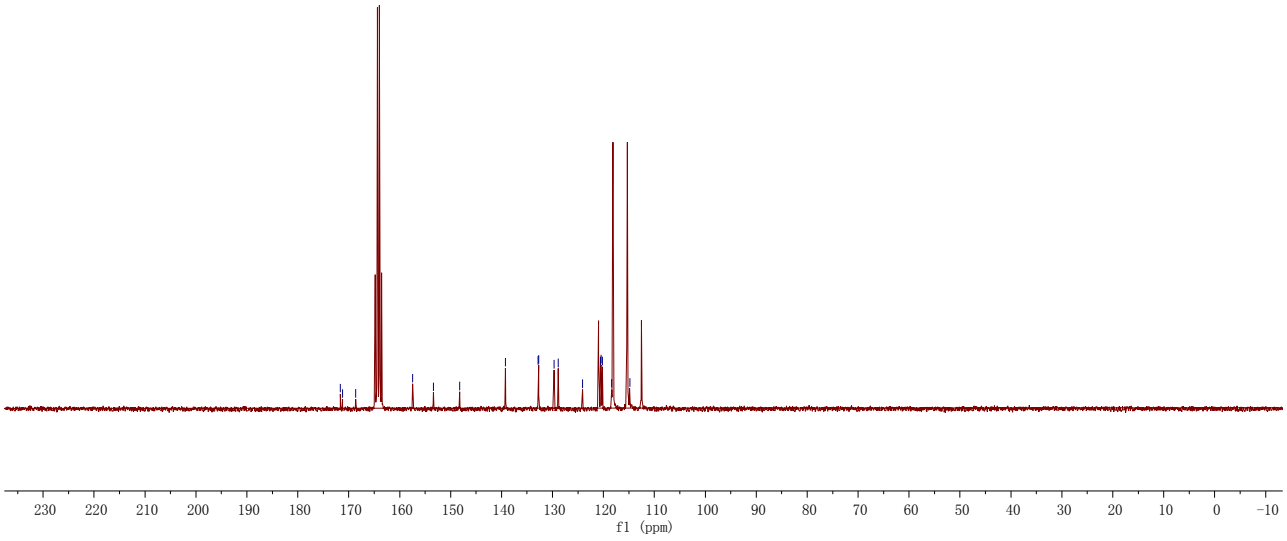




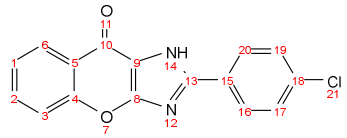
SJ-Fmi
SJ-Fmi TFA BB+DEPT135





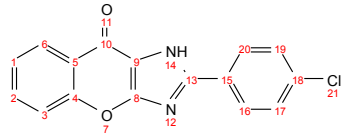


SJ-CL
SJ-CL CF3COOD 1H 20121205

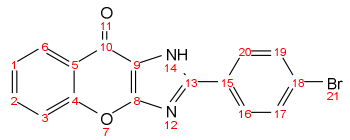


4k

SJ-ClYi_bb130222
SJ-ClYi TFA BB+DEPT-135

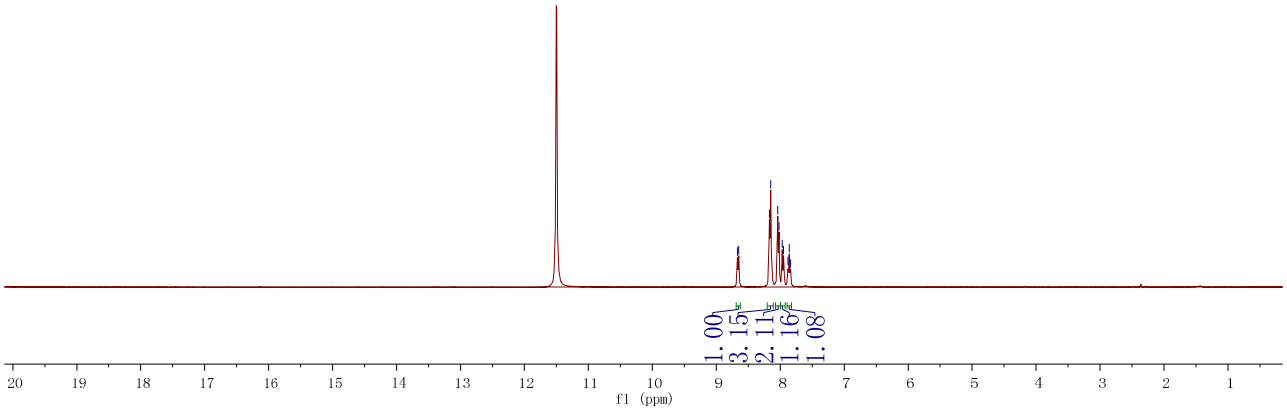


SJ-BrJ
SJ-BrJ in CF3COOH



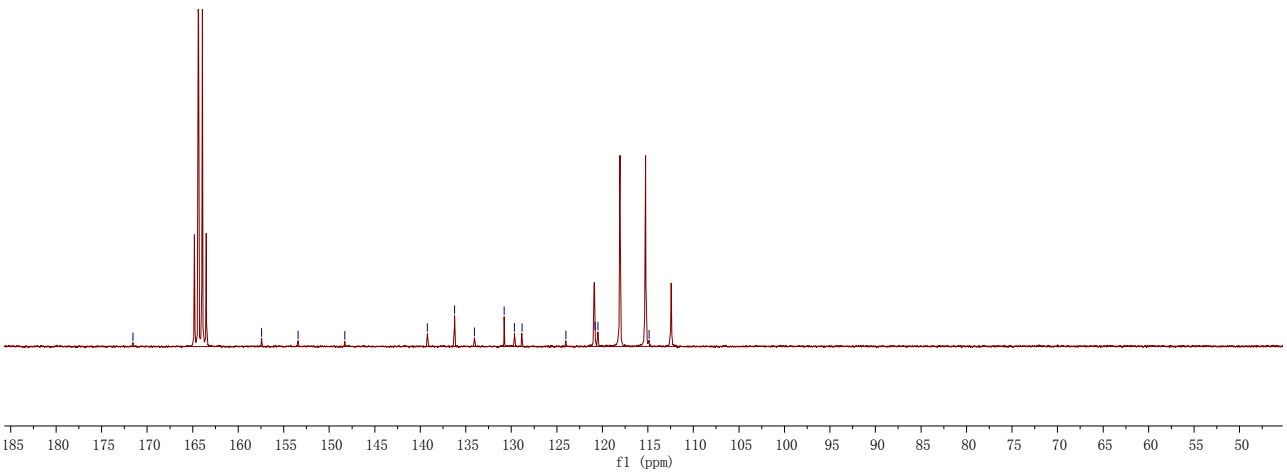
41

8.67
8.65
8.17
8.15
8.04
8.02
7.97
7.95
7.88
7.86
7.84

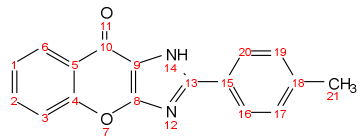


SJ-BrT
SJ-BrT TFA BB-DEPT135

171.58
157.42
153.42
148.28
139.21
136.24
134.05
130.78
129.66
128.84
124.00
120.79
120.50
114.88

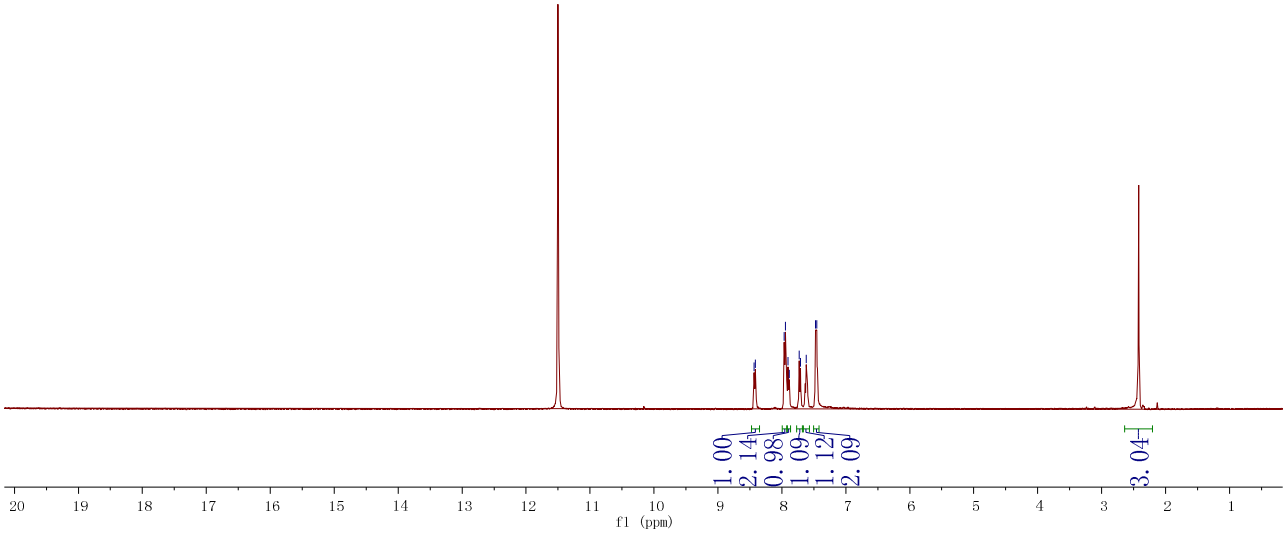


SJ-phch3
SJ phch3 CF3COOD 1H 20121205



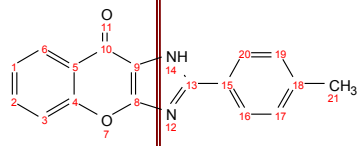
4m

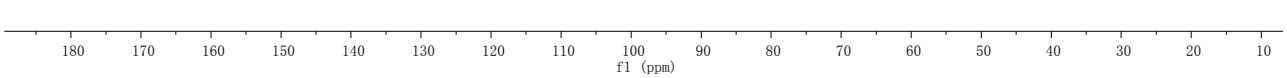
8.43
8.41
7.96
7.94
7.91
7.89
7.73
7.71
7.62
7.47
7.45



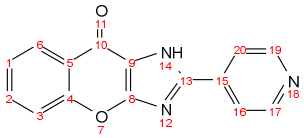
SJ-PhCH3Yi
SJ-PhCH3Yii TFA BB+DEPT135

171.68
164.86
164.43
164.00
163.56
157.48
153.32
151.20
149.44
139.14
133.51
129.80
129.71
128.92
124.28
121.07
120.58
118.96
118.26
115.44
114.68
112.63
22.66

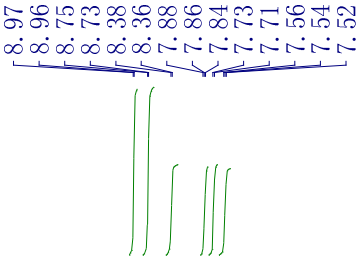


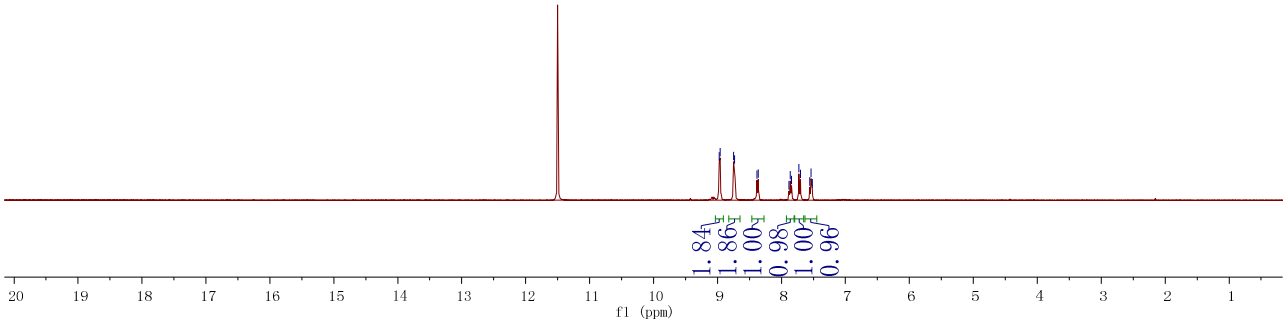


SJ-Py
SJ Py CF3COOD 1H 20121127

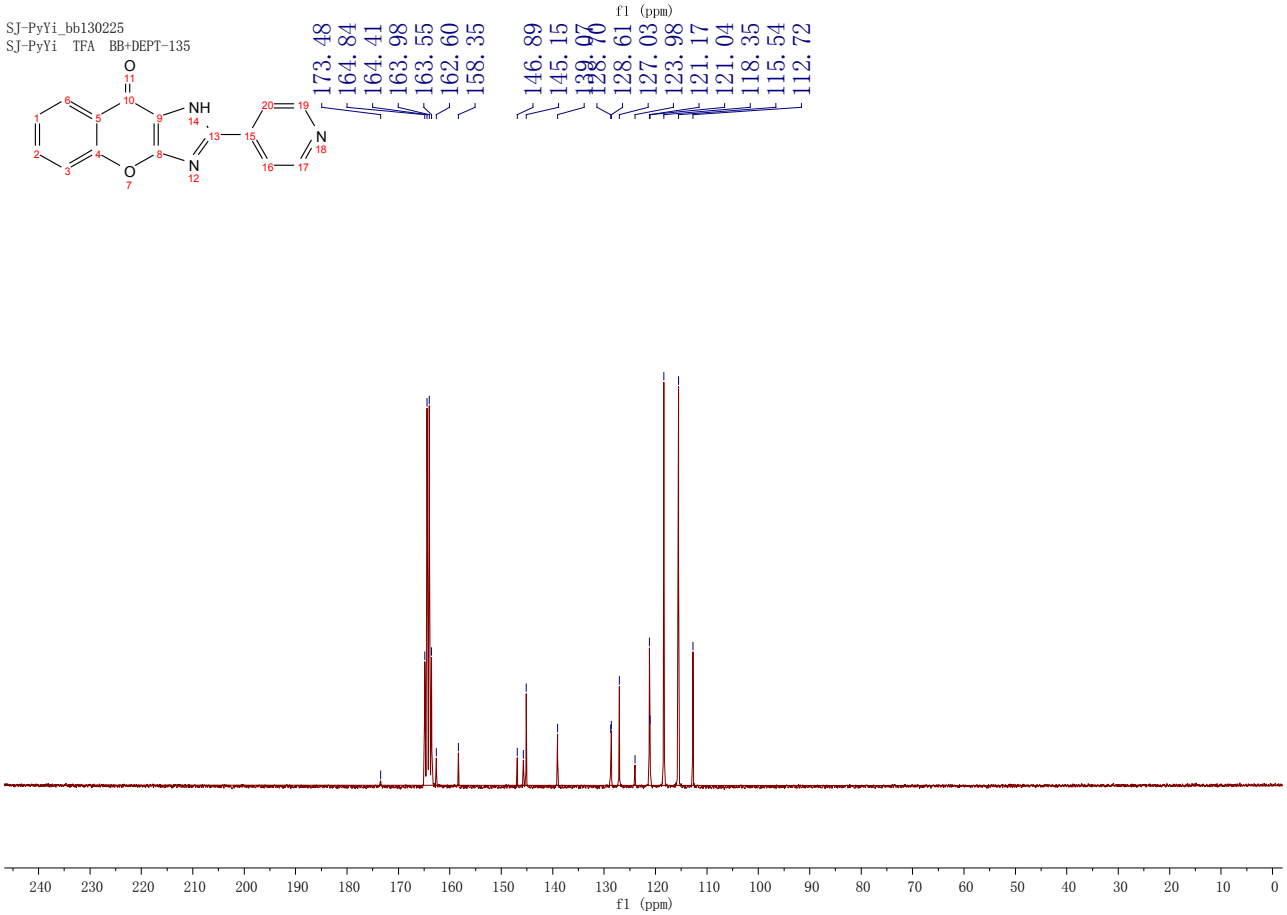


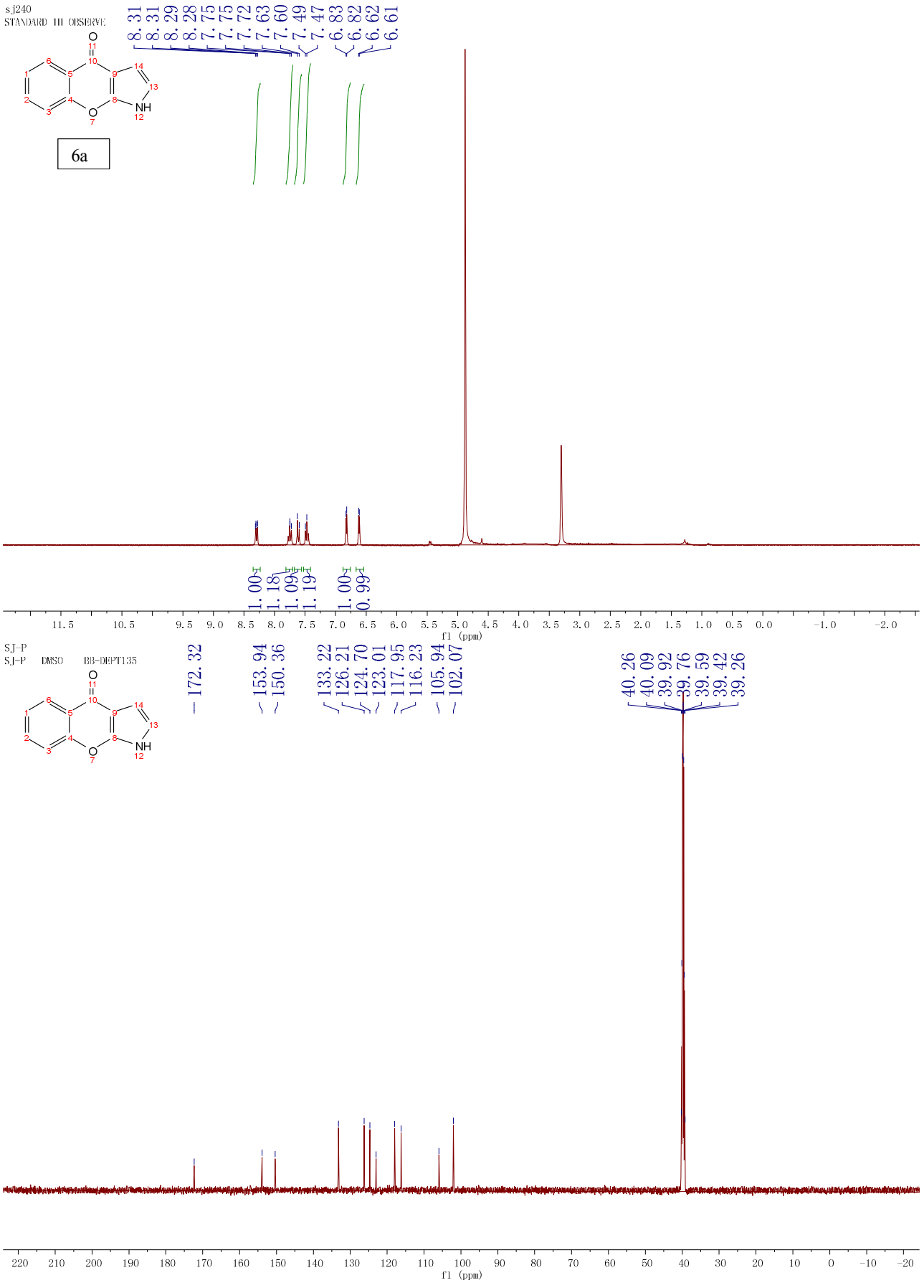
4n

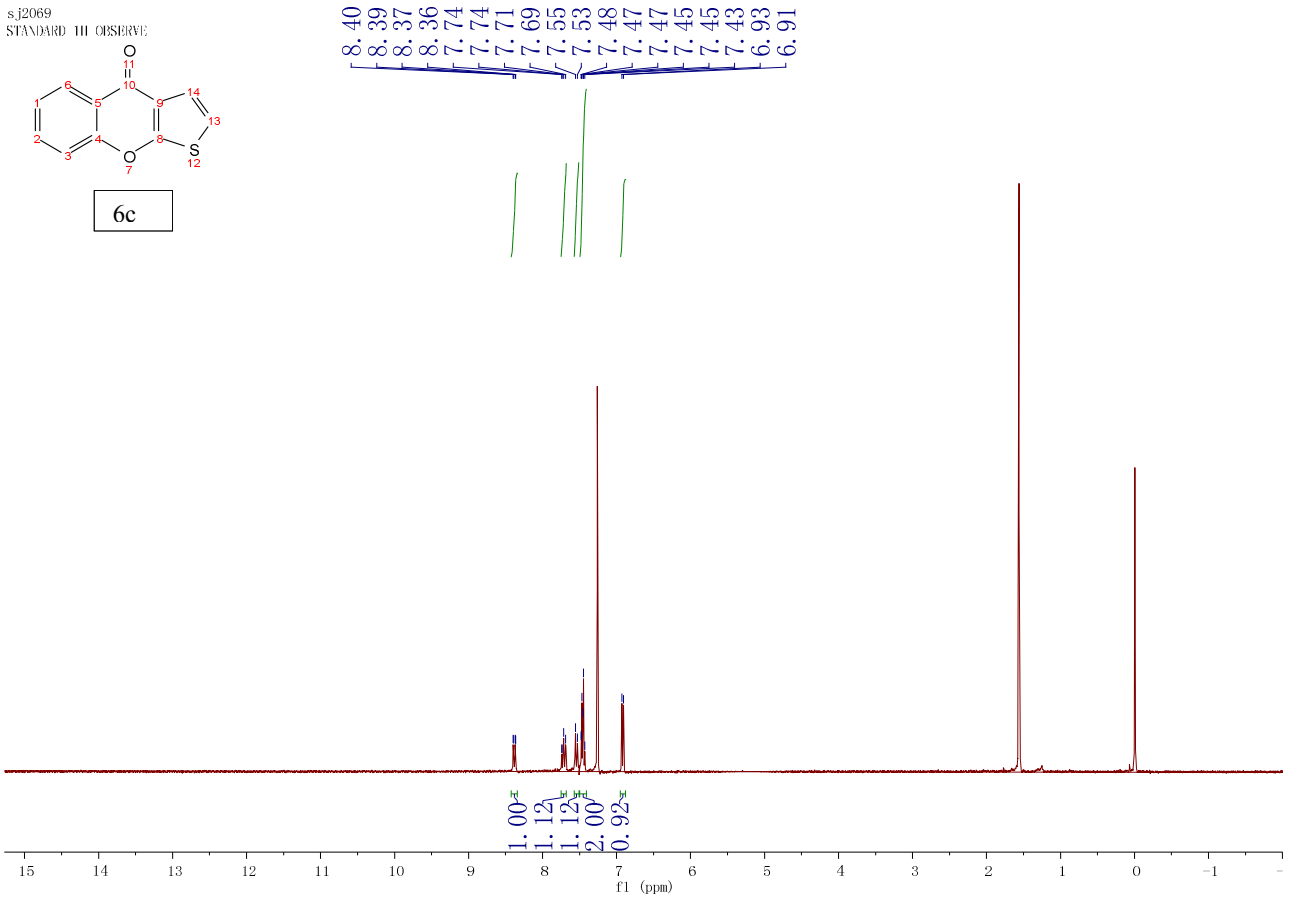


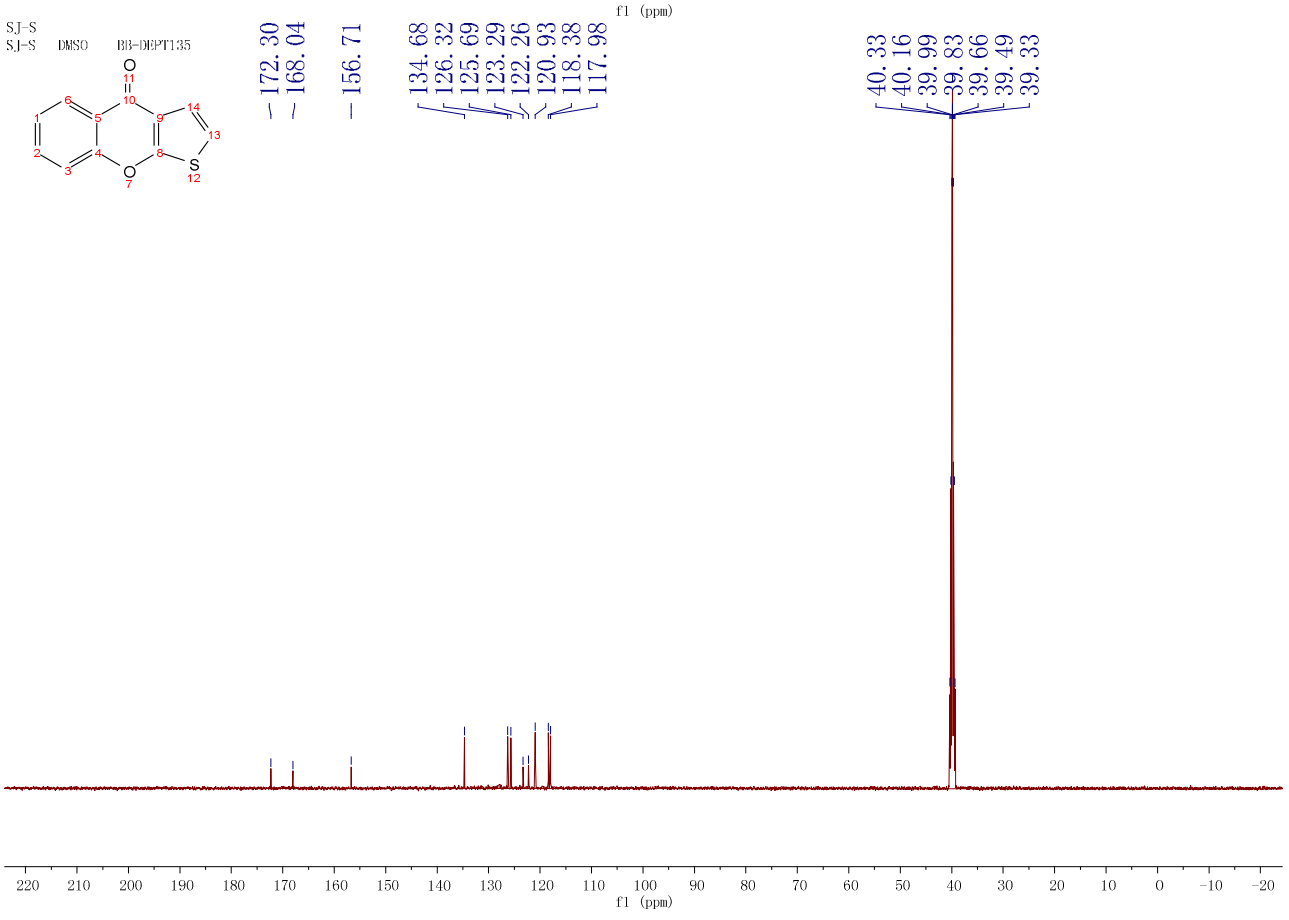


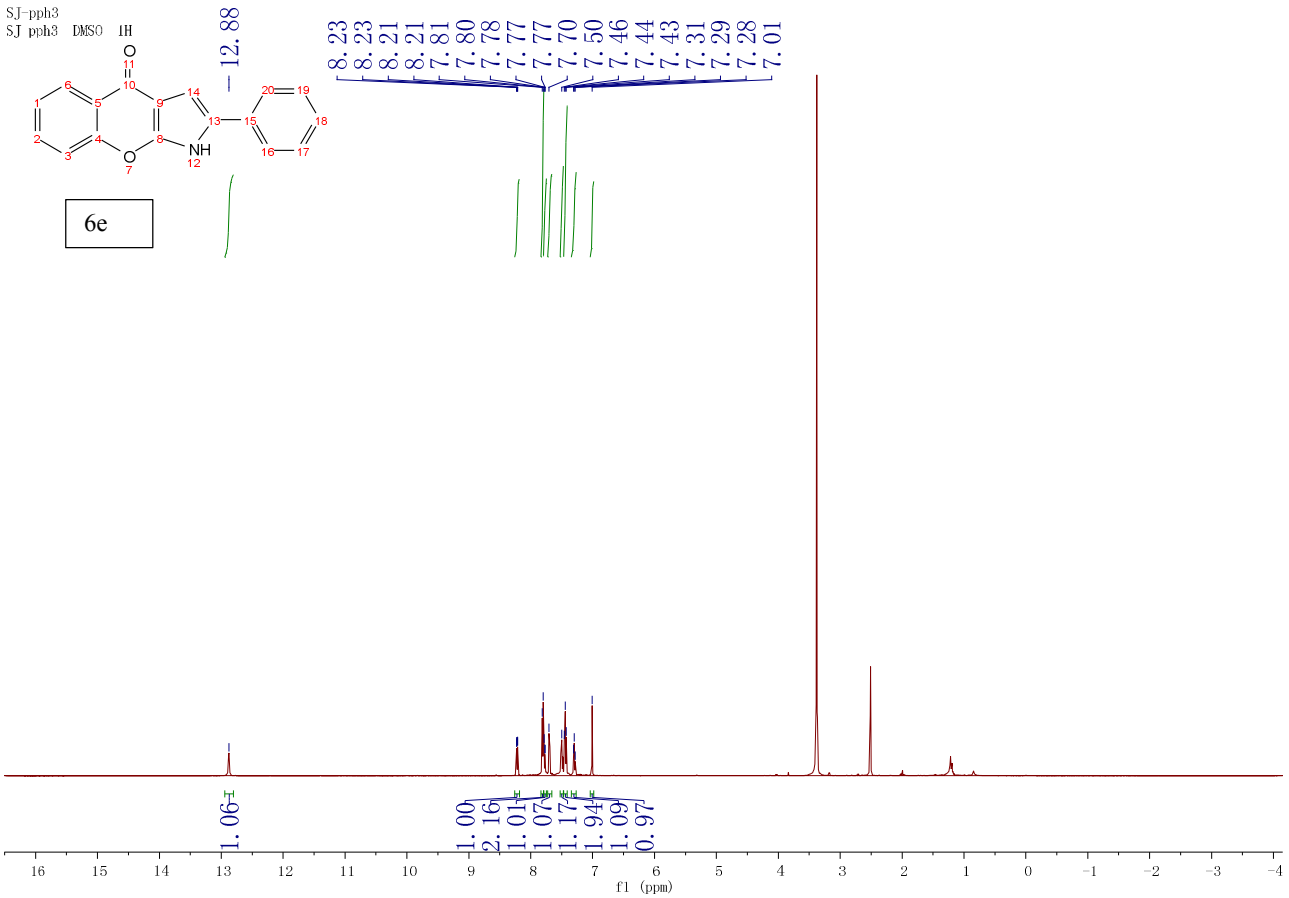
SJ-PyYi_bb130225
SJ-PyYi TFA BB+DEPT-135

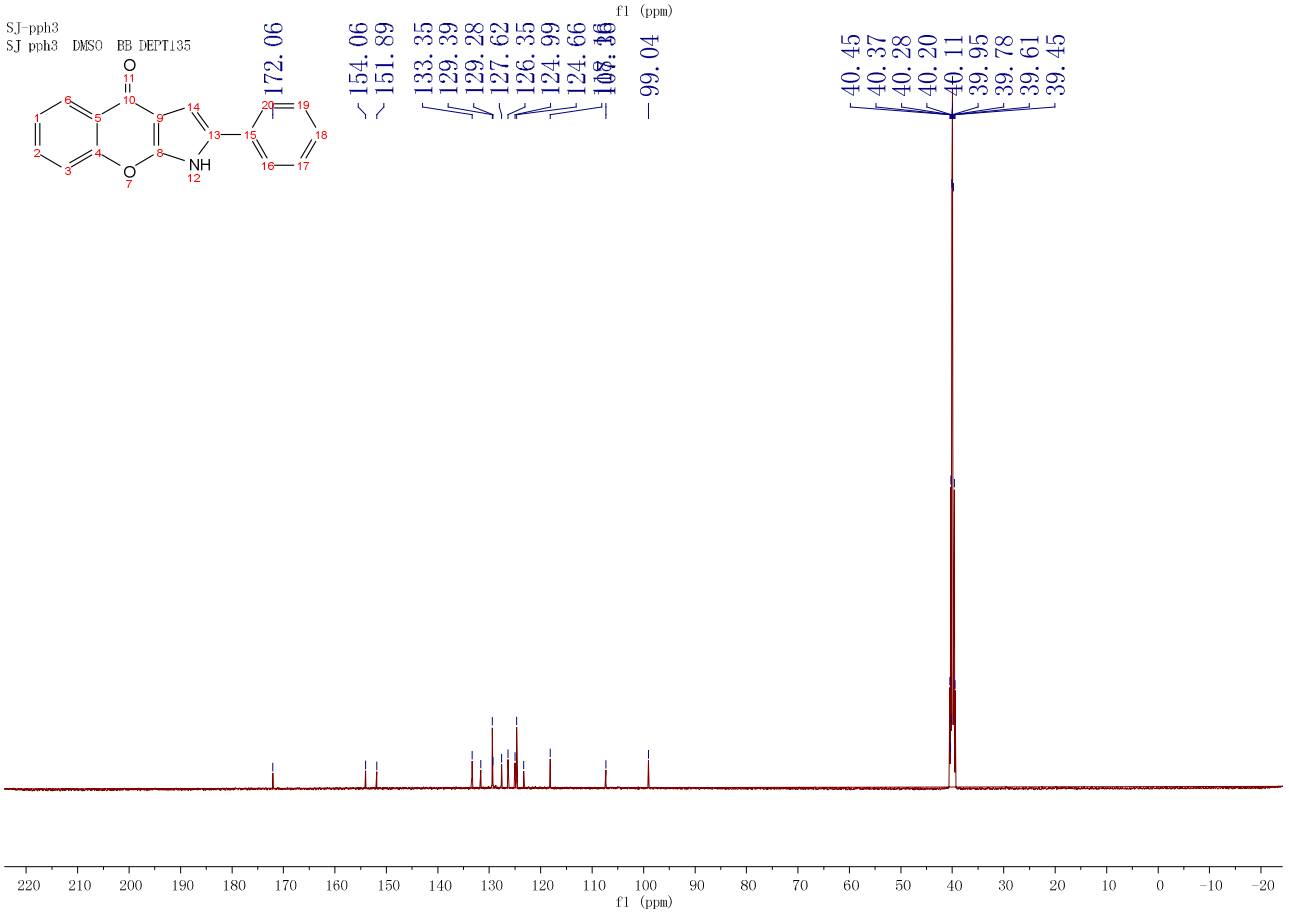



sj240
STANDARD 1H OBSERVE
8.31
8.31
8.29
8.28
7.75
7.75
7.72
7.63
7.60
7.49
7.47
6.83
6.82
6.62
6.61
6a
1.00
1.18
1.09
1.19
1.00
0.99
f1 (ppm)
SJ-P
SJ-P DMSO BB-DEPT135
172.32
153.94
150.36
133.22
126.21
124.70
123.01
117.95
116.23
105.94
102.07
40.26
40.09
39.92
39.76
39.59
39.42
39.26
f1 (ppm)

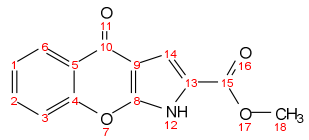

sjj2069
STANDARD 1H OBSERVE
6c
8.40
8.39
8.37
8.36
7.74
7.74
7.71
7.69
7.55
7.53
7.48
7.47
7.47
7.45
7.45
7.43
6.93
6.91
1.00
1.12
1.12
2.00
0.92
f1 (ppm)


SJ-S
SJ-S DMSO BB-DEPT135
172.30
168.04
156.71
134.68
126.32
125.69
123.29
122.26
120.93
118.38
117.98
40.33
40.16
39.99
39.83
39.66
39.49
39.33
f1 (ppm)


SJ-pph3
SJ pph3 DMSO 1H
12.88
8.23
8.23
8.21
8.21
7.81
7.80
7.78
7.77
7.77
7.70
7.50
7.46
7.44
7.43
7.31
7.29
7.28
7.01
6e
1.06
1.00
2.16
1.01
1.07
1.17
1.94
1.09
0.97
f1 (ppm)


SJ-pph3
SJ pph3 DMSO BB DEPT135
172.06
154.06
151.89
133.35
129.39
129.28
127.62
126.35
124.99
124.66
99.04
40.45
40.37
40.28
40.20
40.11
39.95
39.78
39.61
39.45
f1 (ppm)

sj-2111z
STANDARD 1H OBSERVE



6f

SJ-PCO2Me
SJ-PCO2Me DMSO BB+DEPT135