Supporting Information

Metal-free nitroxyl radical-mediated β-C(sp^3)−H amination of saturated ketones with heteroaryl halides: multiple roles of TEMPO

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1. General information

Derivatives of heteroaryl halides (**4b, 4c, 4d, 4e**)[1] and phentylvinylketone (**2a-1**)[2] were synthesized according to previous literatures. The other reagents were obtained from commercial suppliers and used without further purification. The reactions were conducted under an atmosphere of argon and were monitored by TLC unless otherwise noted. Solvents were dried and distilled prior to use. Flash chromatography was performed using silica gel 60 (300–400 mesh). ^{1}H, ^{19}F and ^{13}C NMR spectra were recorded on a Bruker AVANCE400M spectrometer. Melting points were uncorrected. Infrared spectra were obtained on Agilent Cary 630 instrument on a diamond plate by way of technology Attenuated Total Reflection (ATR). HRMS were conducted on an Agilent 6540Q-TOF LC/MS equipped with an electrospray ionization (ESI) probe operating in positive ion mode.

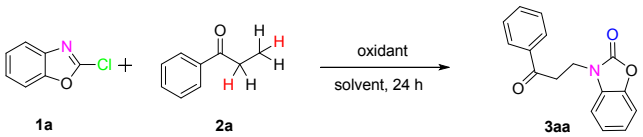
Reference

1. Ognyanov, V. I.; Balan, C.; Bannon, A. W.; Bo, Y.; Dominguez, C.; Fotsch, C.; Gore, V. K.; Klionsky, L.; Ma, V. V.; Qian, Y. X.; Tamir, R.; Wang, X.; Xi, N.; Xu, S.; Zhu, D.; Gavva, N. R.; Treanor, J. J. S.; Norman, M. H. *J. Med. Chem.* **2006**, *49*, 3719 – 3742.
2. Chanthamath, S.; Takaki, S.; Shibatomi, K.; Iwasa, S. *Angew. Chem. Int. Ed.* **2013**, *52*, 5818 – 5821.

2. General procedure for the nitroxyl radical-mediated amination of saturated ketones

Into an oven-dried reaction vial flushed with argon was added 2-chlorobenzo[*d*]oxazole **1a** (0.2 mmol), propiophenone **2a** (0.6 mmol), NaOAc (0.2 mmol), TEMPO (0.6 mmol), *o*-DCB (2 mL). Then the reaction mixture was stirred for 24 hours at 160 ^{o}C under argon atmosphere. After the reaction was complete, the mixture was poured into H_2O (20 mL) and extracted with EtOAc (30 mL) three times. The combined organic layer was dried with anhydrous Na_2SO_4 and evaporated under vacuum. The crude mixture was charged onto silica gel and purified by flash chromatography to furnish the corresponding product **3aa**.

3. Table S1. Optimization of Reaction Conditions[a]



1a 2a 3aa

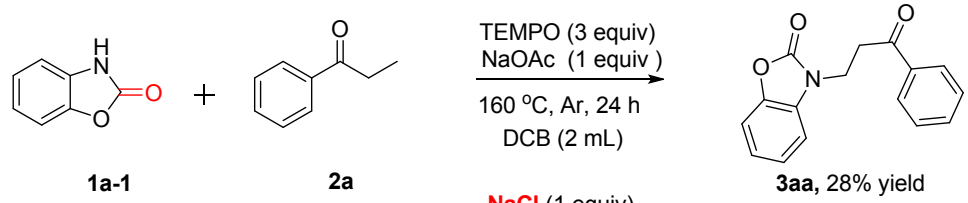
entry	additives (equiv)	oxidant	solvent	Yield (%)[b]
1	$Cu(OAc)_2$/bpy (0.3)	TEMPO	*o*-DCB	52
2	$FeCl_3$/bpy (0.3)	TEMPO	*o*-DCB	37
3	$Mn(OAc)_3.2H_2O$/bpy (0.3)	TEMPO	*o*-DCB	81
4	$Co(OAc)_2.4H_2O$/bpy (0.3)	TEMPO	*o*-DCB	79
5	$Ni(OAc)_2$/bpy (0.3)	TEMPO	*o*-DCB	83
6	NaOAc (0.3)	TEMPO	*o*-DCB	85
7	NaOAc (1)	TEMPO	*o*-DCB	97
8	None	TEMPO	*o*-DCB	78
9	HCOONa (1)	TEMPO	*o*-DCB	93
10	NaOH (1)	TEMPO	*o*-DCB	59
11[c]	NaOAc (1)	TEMPO	*o*-DCB	81
12[d]	NaOAc (1)	TEMPO	*o*-DCB	77
13	NaOAc(1)	TEMPO	*o*-DCB	27[e]
14	NaOAc (1)	TEMPO	*o*-DCB	66[f]
16[g]	NaOAc (1)	NHPI/O_2	*o*-DCB	0
17	NaOAc (1)	TEMPO	PhCl	78
18	NaOAc (1)	TEMPO	$PhCF_3$	83
19	NaOAc (1)	TEMPO	toluene	84
20	H_2O (1.0)	TEMPO	*o*-DCB	88
21	H_2O (2.0)	TEMPO	*o*-DCB	85
22	H_2O (3.0)	TEMPO	*o*-DCB	84

[a]Reaction condition: **1a** (0.2 mmol), **2a** (0.6 mmol), additives, oxidant (3.0 equiv), solvent (2 mL) and stirred for 24 h under argon atmosphere. [b]Isolated yield. [c]TEMPO (2.0 equiv). [d]**2a** (0.4 mmol). [e]120 °C. [f]140 °C. [g]NHPI = *N*-hydroxyphthalimide.

4. Control experiments

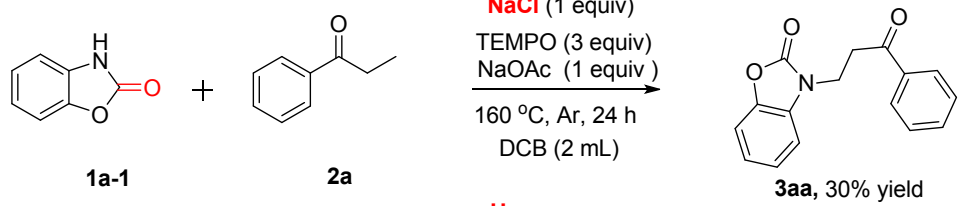
Scheme S1. Control experiments

a)



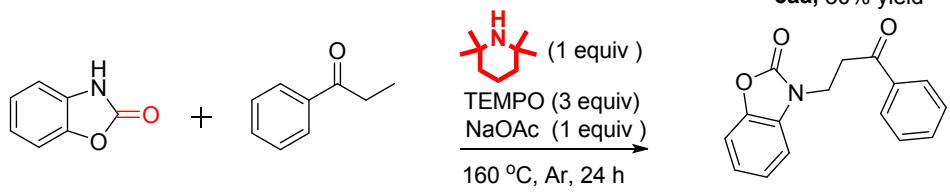
1a-1 **2a** **3aa,** 28% yield

b)



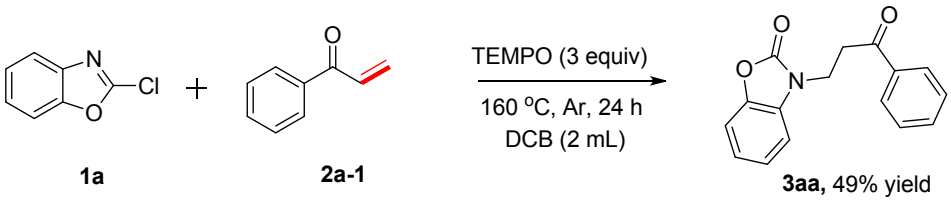
1a-1 **2a** **3aa,** 30% yield

c)



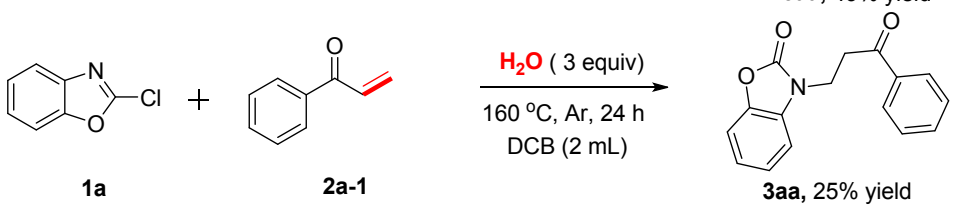
1a-1 **2a** **3aa,** 71% yield

d)



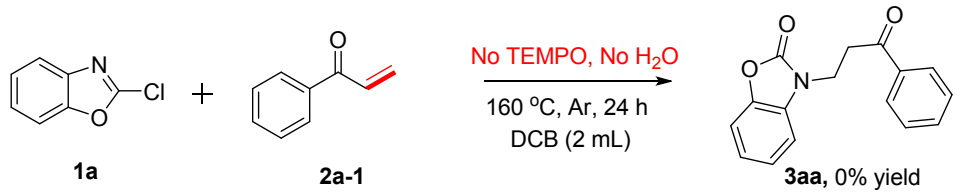
1a **2a-1** **3aa,** 49% yield

e)



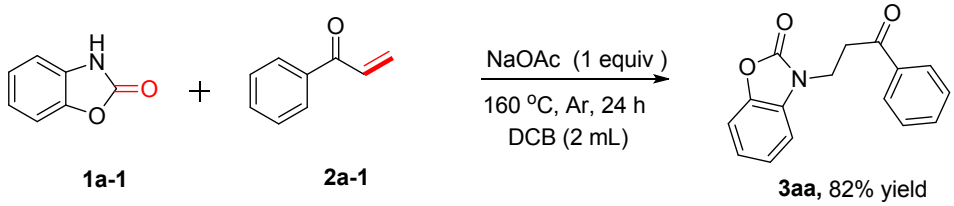
1a **2a-1** **3aa,** 25% yield

f)



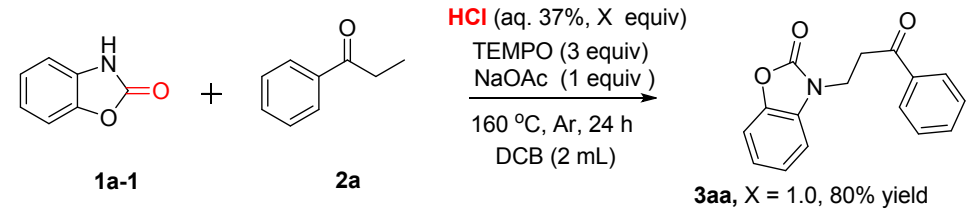
1a **2a-1** **3aa,** 0% yield

g)



1a-1 **2a-1** **3aa,** 82% yield

h)



1a-1 **2a**

3aa, X = 1.0, 80% yield
3aa, X = 2.0, 64% yield

Control experiment a):

Benzo[d]oxazol-2(3*H*)-one **1a-1**, instead of 2-chlorobenzo[*d*]oxazole **1a** was used to react with propiophenone **2a**, NaOAc (0.2 mmol), TEMPO (0.6 mmol), *o*-DCB (2 mL), under argon atmosphere for 24 h. The target product **3aa** was obtained with 28% yield.

Control experiment b):

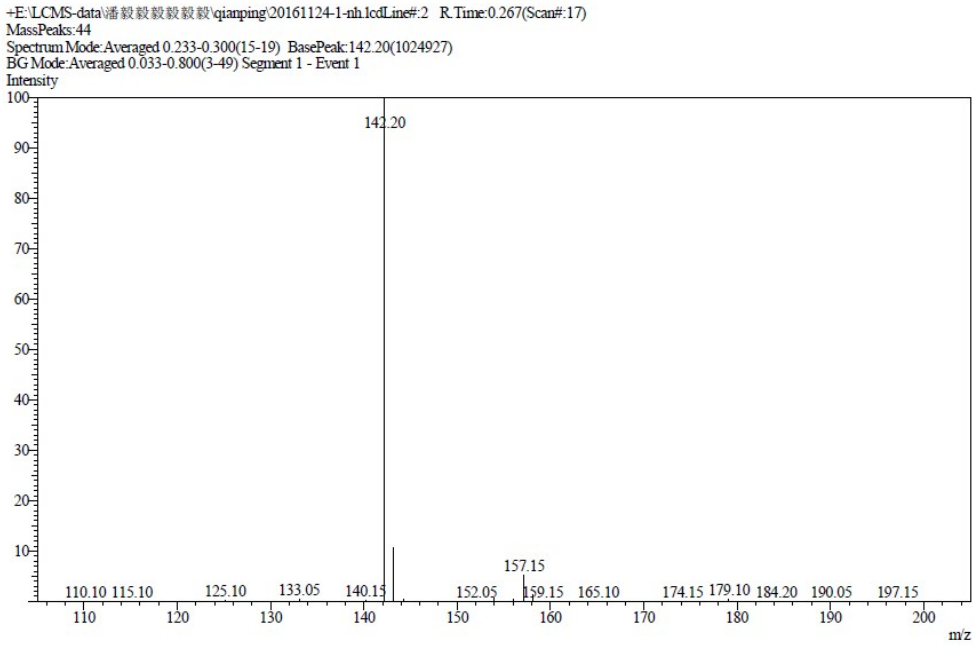
Benzo[d]oxazol-2(3*H*)-one **1a-1** was used to react with propiophenone **2a**, NaOAc (0.2 mmol), TEMPO (0.6 mmol), *o*-DCB (2 mL), with the addition of NaCl (0.2 mmol) under argon atmosphere for 24 h. The target product **3aa** was obtained with 30% yield.

Control experiment c):

This reaction was carried out in the condition of **1a-1** (0.2 mmol), **2a** (0.6 mmol), TEMPO (0.6 mmol), *o*-DCB (2 mL), under argon atmosphere for 24 h. Product **3aa** was obtained with 71% yield.

The 2,2,6,6-tetramethylpiperidine was detected in the crude reaction system by the technology of LC-MASS (**Figure 1**). LC-MASS (TOF MS ESI): calcd for $C_9H_{20}N^+$ $[M+H]^+$142.16, found 142.20.

Figure 1. LC-MASS of 2,2,6,6-tetramethylpiperidine



Control experiment d):

This reaction of **1a** (0.2 mmol) and **2a-1** (0.2 mmol) was operated under the condition of TEMPO (0.6 mmol), *o*-DCB (2 mL), argon atmosphere for 24 h. The reaction gave desired product **3aa** with 49% yield.

Control experiment e):

This reaction of **1a** (0.2 mmol) and **2a-1** (0.2 mmol) was operated in *o*-DCB (2 mL), argon atmosphere for 24 h with the addition of H_2O (0.6 mmol, 3 equiv). The reaction gave desired product **3aa** with 25% yield.

Control experiment f):

This reaction of **1a** (0.2 mmol) and **2a-1** (0.2 mmol) was operated in *o*-DCB (2 mL), argon atmosphere for 24 h without use of H_2O or TEMPO. No desired product was detected in these reactions.

Control experiment g):

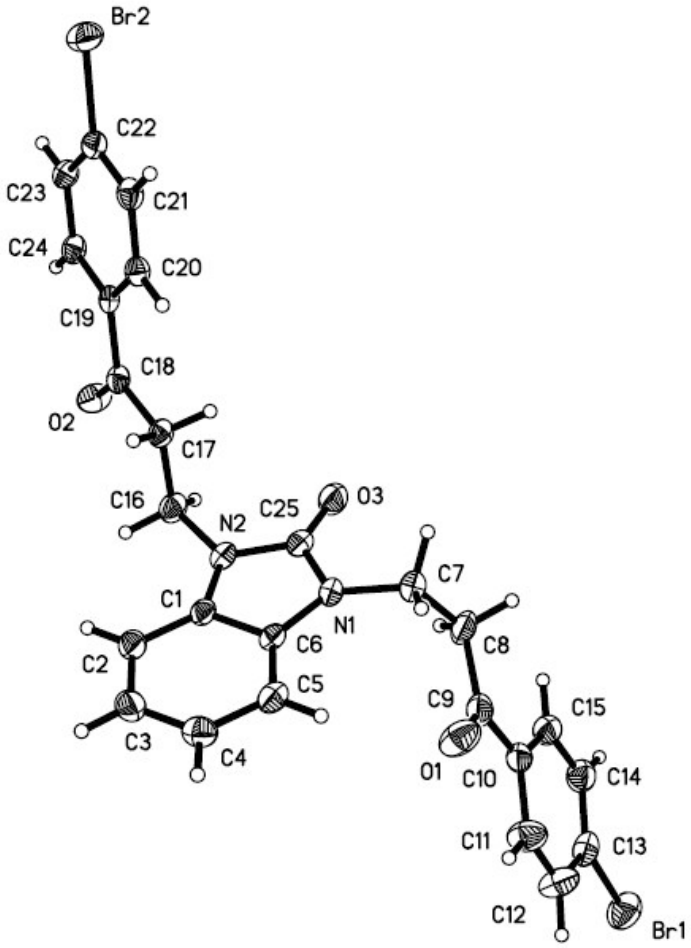
The reaction of **1a-1** (0.2 mmol, 1 equiv) with **2a-1** (0.2 mmol, 1 equiv) was conducted under the

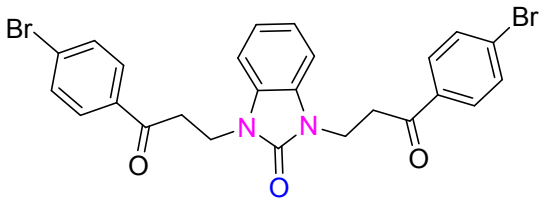
condition of NaOAc (0.2 mmol, 1 equiv), *o*-DCB (2 mL), argon atmosphere, for 24 h. This aza-Michael addition proceeded smoothly to afford 82% yield of **3aa**.

Control experiment h):

This reaction was carried out in the condition of **1a-1** (0.2 mmol), **2a** (0.6 mmol), TEMPO (0.6 mmol), NaOAc (0.2 mmol), *o*-DCB (2 mL), under argon atmosphere for 24 h. When HCl (aq. 37%, 0.2 mmol) was added into the reaction system, **3aa** was obtained with 80% yield. More HCl (aq. 37%, 0.4 mmol) was added into the reaction system, the yield of **3aa** was 64%.

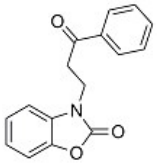
5. X-ray analysis of 5ae



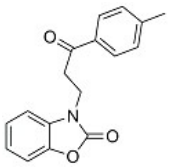


CCDC number: 1511465

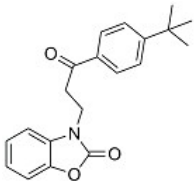
6. Characterization data of compounds 3 and 5



*3-(3-oxo-3-phenylpropyl)benzo[d]oxazol-2(3H)-one (**3aa**)*: Colorless solid (51.8 mg, 97% yield), mp 102-103 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-7.89 (m, 2H), 7.63-7.53 (m, 1H), 7.46 (t, J = 7.7 Hz, 2H), 7.25-7.06 (m, 4H), 4.28 (t, J = 6.7 Hz, 2H), 3.53 (t, J = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.3, 154.7, 142.8, 136.2, 133.8, 131.2, 128.9, 128.2, 124.1, 122.6, 110.1, 109.1, 37.5, 36.4. IR (cm^{-1}): v 3068, 2999, 2924, 1754, 1675, 1617, 1597, 1485, 1450, 1396, 1327, 1258, 1213, 1038, 954, 749. HRMS (TOF MS ESI): calcd for $C_{16}H_{14}NO_3^+$ $[M+H]^+$ 268.0974, found 268.0971.

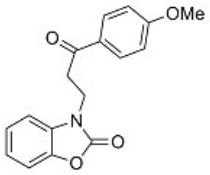


*3-(3-oxo-3-(p-tolyl)propyl)benzo[d]oxazol-2(3H)-one (**3ab**)*: Yellow solid (52.3 mg, 93% yield), mp 108-109 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.2 Hz, 2H),7.28-7.15 (m, 5H), 7.13-7.05 (m, 1H), 4.25 (t, J = 6.7 Hz, 2H), 3.49 (t, J = 6.7 Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.9, 154.6, 144.7, 142.7, 133.8, 131.2, 129.5, 128.2, 124.0, 122.4, 110.0, 109.1, 37.5, 36.2, 21.7. IR (cm^{-1}): v 3062, 2920, 2853, 1756, 1672, 1606, 1487, 1366, 1325, 1259, 1220, 1205, 1185, 1042, 941, 775. HRMS (TOF MS ESI): calcd for $C_{17}H_{15}NO_3Na^+$ $[M+Na]^+$ 304.0950, found 304.0948.

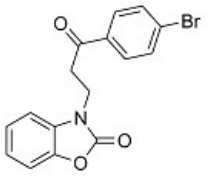


*3-(3-(4-(tert-butyl)phenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3ac**)*: Light yellow solid (58.2 mg, 90% yield), mp 109-110 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.5 Hz, 2H), 7.46 (d, J = 8.5 Hz, 2H), 7.23-7.06 (m, 4H), 4.27 (t, J = 6.7 Hz, 2H), 3.50 (t, J = 6.7 Hz, 2H), 1.33 (s, 9H).

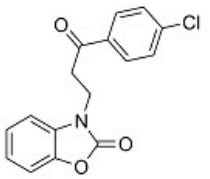
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 157.5, 154.5, 142.7, 133.6, 131.1, 128.0, 125.7, 123.9, 122.4, 109.9, 109.0, 37.5, 36.1, 35.1, 31.2, 31.0. IR (cm^{-1}): ν 2965, 2920, 2868, 1784, 1689, 1608, 1489, 1392, 1374, 1254, 1224, 1193, 997, 747, 554. HRMS (TOF MS ESI): calcd for $C_{20}H_{21}NO_3Na^+$ $[M+Na]^+$ 346.1419, found 346.1417.



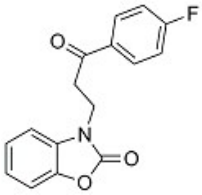
*3-(3-(4-methoxyphenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3ad**)*: Yellow solid (52.3 mg, 88% yield), mp 119-120 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.88 (m, 2H), 7.23-7.15 (m, 3H), 7.13-7.06 (m, 1H), 6.93-6.86 (m, 2H), 4.25 (t, J = 6.7 Hz, 2H), 3.85 (s, 3H), 3.46 (t, J = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.7, 164.0, 154.6, 142.7, 131.2, 130.44 129.3, 124.0, 122.4, 114.0, 110.0, 109.1, 55.6, 37.6, 35.9. IR (cm^{-1}): ν 3064, 2917, 2850, 1754, 1664, 1599, 1572, 1485, 1420, 1366, 1254, 1215, 1174, 1114, 837, 792, 680. HRMS (TOF MS ESI): calcd for $C_{17}H_{15}NO_4Na^+$ $[M+Na]^+$ 320.0899, found 320.0897.



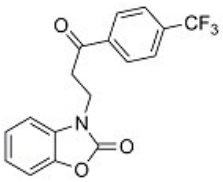
*3-(3-(4-bromophenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3ae**)*: Light yellow solid (63.0 mg, 91% yield), mp 101-102 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.77 (m, 2H), 7.63-7.57 (m, 2H), 7.23-7.08 (m, 4H), 4.26 (t, J = 6.7 Hz, 2H), 3.49 (t, J = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.3, 154.6, 142.8, 134.9, 132.2, 131.1, 129.7, 129.1, 124.1, 122.6, 110.2, 109.1, 37.4, 36.3. IR (cm^{-1}): ν 3068, 2954, 2345, 1742, 1671, 1578, 1485, 1362, 1318, 1260, 1213, 1043, 1008, 982, 775, 697. HRMS (TOF MS ESI): calcd for $C_{16}H_{12}BrNO_3Na^+$ $[M+Na]^+$ 367.9898, found 367.9891.



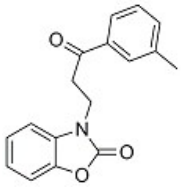
*3-(3-(4-chlorophenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3af**)*: Yellow solid (56.7 mg, 94% yield), mp 156-157 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.84 (m, 2H), 7.47-7.39 (m, 2H), 7.24-7.16 (m, 3H), 7.15-7.07 (m, 1H), 4.26 (t, J = 6.7 Hz, 2H), 3.50 (t, J = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 154.6, 142.8, 140.3, 134.5, 131.1, 129.6, 129.2, 124.1, 122.6, 110.2, 109.1, 37.4, 36.3. IR (cm^{-1}): ν 3069, 2952, 2920, 2851, 1744, 1672, 1618, 1586, 1541, 1485, 1442, 1405, 1362, 1282, 1213, 1087, 939, 874, 624. HRMS (TOF MS ESI): calcd for $C_{16}H_{12}ClNO_3Na^+$ $[M+Na]^+$ 324.0403, found 324.0397.



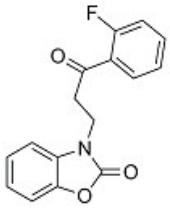
*3-(3-(4-fluorophenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3ag**)*: Light yellow solid (42.2 mg, 74% yield), mp 149-150 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01-7.93 (m,2H), 7.25-7.17 (m, 3H), 7.16-7.06 (m, 3H), 4.26 (t, J = 6.7 Hz, 2H), 3.50 (t, J = 6.7 Hz, 2H).^{13}C NMR (101 MHz, $CDCl_3$) δ 195.7, 167.4, 164.9, 154.7, 142.8, 132.7, 131.2, 130.9, 130.8, 124.1, 122.6, 116.1, 115.9, 110.1, 109.1, 37.5, 36.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -103.96. IR (cm^{-1}): ν 3073, 2989, 2961, 2920, 1746, 1672, 1597, 1487, 1383, 1321, 1362, 1259, 1164, 1114, 1071, 848, 751. HRMS (TOF MS ESI): calcd for $C_{16}H_{12}FNO_3Na^+$ $[M+Na]^+$ 308.0699, found 308.0696.



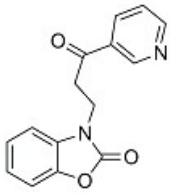
*3-(3-oxo-3-(4-(trifluoromethyl)phenyl)propyl)benzo[d]oxazol-2(3H)-one (**3ah**)*: Light yellow solid (49.6 mg, 74% yield), mp 172-173 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.2 Hz, 2H), 7.65 (d, J = 8.3 Hz, 2H), 7.18-7.01 (m, 4H), 4.21 (t, J = 6.6 Hz, 2H), 3.49 (t, J = 6.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.3, 153.5, 141.7, 137.6, 134.1, 133.8, 130.0, 127.4, 124.9, 124.8, 123.8, 123.0, 121.5, 121.1, 109.1, 107.9, 36.2, 35.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.20. IR (cm^{-1}): ν 2965, 2922, 2854, 1746, 1684, 1610, 1485, 1366, 1317, 1258, 1116, 1064, 1013, 985, 849, 602. HRMS (TOF MS ESI): calcd for $C_{17}H_{12}F_3NO_3Na^+$ $[M+Na]^+$ 358.0667, found 358.0668.



*3-(3-oxo-3-(m-tolyl)propyl)benzo[d]oxazol-2(3H)-one (**3ai**)*: Light yellow solid (51.8 mg, 92% yield), mp 67-68 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.68 (m, 2H), 7.41-7.30 (m, 2H), 7.25-7.06 (m, 4H), 4.26 (t, J = 6.7 Hz, 2H), 3.51 (t, J = 6.7 Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 154.7, 142.8, 138.7, 136.2, 134.5, 131.2, 128.7, 128.7, 125.4, 124.0, 122.5，110.1, 109.1, 37.5, 36.4, 21.4. IR (cm^{-1}): v 3066, 3042, 2960, 2919, 1755, 1675, 1615, 1586, 1487, 1382, 1362, 1321, 1224, 1187, 1039, 945, 751, 685. HRMS (TOF MS ESI): calcd for $C_{17}H_{15}NO_3Na^+$ $[M+Na]^+$ 304.0950, found 304.0947.

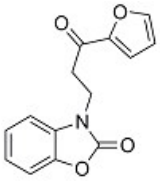


*3-(3-(2-fluorophenyl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3aj**)*: Yellow solid (53.1 mg, 93% yield), mp 121-122 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (td, J = 7.7, 1.8 Hz, 1H), 7.58-7.50 (m, 1H), 7.40-6.89 (m, 6H), 4.26 (t, J = 6.7 Hz, 2H), 3.52 (td, J = 6.7, 3.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 195.4, 163.6, 161.1, 154.6, 142.8, 135.5, 135.4, 131.2, 130.7, 130.7, 124.8, 124.7, 124.7, 124.0, 122.5, 117.1, 116.8, 110.1, 109.0, 41.2, 41.2, 37.4, 37.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.8. IR (cm^{-1}): v 3107, 3076, 1764, 1677, 1603, 1580, 1482, 1398, 1366, 1331, 1316, 1286, 1256, 1142, 945, 745. HRMS (TOF MS ESI): calcd for $C_{16}H_{12}FNO_3Na^+$ $[M+Na]^+$ 308.0699, found 308.0691.

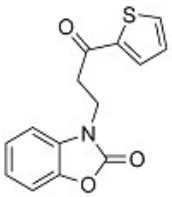


*3-(3-oxo-3-(pyridin-3-yl)propyl)benzo[d]oxazol-2(3H)-one (**3ak**)*: Colorless solid (24.1 mg, 45%

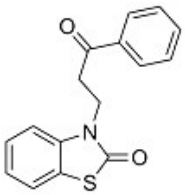
yield), mp 122-123 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.15 (dd, J = 2.3, 0.9 Hz, 1H), 8.79 (dd, J = 4.8, 1.7 Hz, 1H), 8.22 (dt, J = 8.0, 2.0 Hz, 1H), 7.46-7.40 (m, 1H), 7.24-7.16 (m, 3H), 7.16-7.07 (m, 1H), 4.29 (t, J = 6.6 Hz, 2H), 3.56 (t, J = 6.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 154.6, 154.1, 149.7, 142.8, 135.5, 131.5, 131.1, 124.1, 123.9, 122.7, 110.2, 109.0, 37.1, 36.6. IR (cm^{-1}): v 3056, 3028, 2924, 1772, 1686, 1610, 1587, 1487, 1412, 1364, 1321, 1224, 1040, 937, 753, 665. HRMS (TOF MS ESI): calcd for $C_{15}H_{12}N_2O_3Na^+$ $[M+Na]^+$ 291.0746, found 291.0742.



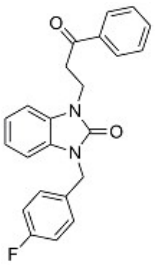
*3-(3-(furan-2-yl)-3-oxopropyl)benzo[d]oxazol-2(3H)-one (**3al**)*: Light yellow solid (49.4 mg, 96% yield), mp 91-92 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.25-7.14 (m, 4H), 7.13-7.05 (m, 1H), 6.54 (dd, J = 3.5, 1.5 Hz, 1H), 4.24 (t, J = 6.6 Hz, 2H), 3.38 (t, J = 6.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.1, 154.6, 152.2, 147.0, 142.7, 131.0, 124.0, 122.5, 118.0, 112.6, 110.0, 109.1, 37.1, 36.3. IR (cm^{-1}): v 3127, 3096, 1746, 1671, 1614, 1587, 1474, 1388, 1223, 1168, 1144, 1038, 917, 955, 514. HRMS (TOF MS ESI): calcd for $C_{14}H_{11}NO_4Na^+$ $[M+Na]^+$ 280.0586, found 280.0587.



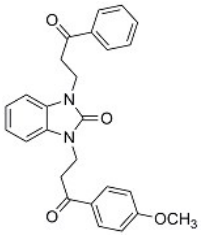
*3-(3-oxo-3-(thiophen-2-yl)propyl)benzo[d]oxazol-2(3H)-one (**3am**)*: Light yellow solid (48.1 mg, 88% yield), mp 108-109 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.69 (m, 1H), 7.69-7.62 (m, 1H), 7.25-7.14 (m, 3H), 7.14-7.05 (m, 1H),4.26 (t, J = 6.6 Hz, 2H), 3.46 (t, J = 6.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.0, 154.6, 143.4, 142.7, 134.6, 132.7, 131.1, 128.4, 124.1, 122.5, 110.0, 109.2, 37.5, 37.0. IR (cm^{-1}): v 3090, 2942, 2918, 2851, 1780, 1653, 1519, 1487, 1451, 1398, 1321, 1262, 1220, 1038, 947, 744. HRMS (TOF MS ESI): calcd for $C_{14}H_{11}NO_3SNa^+$ $[M+Na]^+$ 296.0357, found 296.0353.



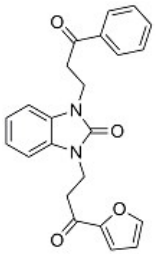
*3-(3-oxo-3-phenylpropyl)benzo[d]thiazol-2(3H)-one (**3ba**)*: White solid (40.2 mg, 71% yield), mp 101-102 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-7.88 (m, 2H), 7.67-7.52 (m, 1H), 7.51-7.39 (m, 3H), 7.38-7.28 (m, 1H), 7.24-7.12 (m, 2H), 4.40 (t, J = 7.1 Hz, 2H), 3.46 (t, J = 7.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 170.2, 136.9, 136.3, 133.7, 128.9, 128.2, 126.6, 123.3, 122.9, 122.8, 110.9, 38.3, 36.3. IR (cm^{-1}): v 3328, 2919, 1670, 1636, 1579, 1541, 1523, 1437, 1418, 1388, 1362, 1314, 1239, 1167, 1012, 975, 740, 688. HRMS (TOF MS ESI): calcd for $C_{16}H_{14}NO_2S^+$ $[M+H]^+$ 284.0745, found 284.0741.



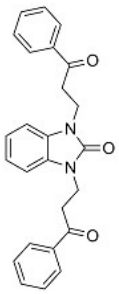
*1-(4-fluorobenzyl)-3-(3-oxo-3-phenylpropyl)-1,3-dihydro-2H-benzo[d]imidazol-2-one (**3ca**)*: Light yellow solid (57.7 mg, 77% yield), mp 112-113 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.5 Hz, 2H), 7.55 (t, J = 7.3 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 7.29 (dd, J = 8.1, 5.5 Hz, 2H), 7.18 (d, J = 7.7 Hz, 1H), 7.09 (t, J = 7.6 Hz, 1H),6.99 (q, J = 8.3 Hz, 3H), 6.86 (d, J = 7.7 Hz, 1H), 5.02 (s, 2H), 4.37 (t, J = 7.0 Hz, 2H), 3.51 (t, J = 7.0 Hz, 2H).^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 163.6, 161.2, 154.4, 136.5, 133.6, 132.2, 132.2, 129.4, 129.4, 129.3, 129.2, 128.8, 128.2, 121.7, 121.4, 115.9, 115.7, 108.3, 108.3, 44.2, 37.1, 36.8. ^{19}F NMR (377 MHz, $CDCl_3$) δ -114.6. IR (cm^{-1}): v 3060, 2924, 2849, 1677, 1618, 1599, 1582, 1508, 1495, 1444, 1411, 1381, 1349, 1327, 1213, 1161, 1098, 975, 704. HRMS (TOF MS ESI): calcd for $C_{23}H_{20}FN_2O_2^+$ $[M+H]^+$ 375.1509, found 375.1507.



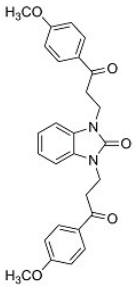
*1-(3-(4-methoxyphenyl)-3-oxopropyl)-3-(3-oxo-3-phenylpropyl)-1,3-dihydro-2H-benzo[d]imidazol-2-one (**3da**)*: Colorless solid (26.6 mg, 31% yield), mp 122-123 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (t, J = 7.8 Hz, 4H), 7.56 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.14-7.07 (m, 2H), 7.21-7.14 (m, 2H),6.91 (d, J = 8.9 Hz, 2H), 4.33 (q, J = 7.0 Hz, 4H), 3.86 (s, 3H), 3.49 (t, J = 7.1 Hz, 2H), 3.43 (t, J = 7.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 196.4, 194.8, 163.9, 154.3, 136.5, 133.6, 130.5, 129.6, 129.3, 128.8, 128.2, 121.5, 113.9, 108.3, 108.2, 55.6, 37.2, 36.8, 36.8, 36.6. IR (cm^{-1}): v 3060, 2952, 2920,2850, 1694, 1671, 1601, 1575, 1495, 1418, 1448, 1375, 1325, 1312, 1254, 1211, 1174, 1028, 978, 729. HRMS (TOF MS ESI): calcd for $C_{26}H_{25}N_2O_4^+$ $[M+H]^+$ 429.1814, found 429.1810.



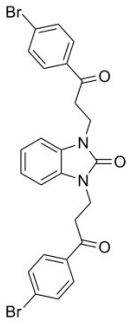
*1-(3-(furan-2-yl)-3-oxopropyl)-3-(3-oxo-3-phenylpropyl)-1,3-dihydro-2H-benzo[d]imidazol-2-one(**3dl**)*: Yellow solid (25.6 mg, 33% yield), mp 133-134 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.3 Hz, 2H), 7.60-7.52 (m, 2H), 7.44 (t, J = 7.7 Hz, 2H), 7.20 (d, J = 3.5 Hz, 1H), 7.18-7.13 (m, 2H), 7.13-7.06 (m, 2H), 6.50 (dd, J = 3.5, 1.6 Hz, 1H), 4.38-4.24 (m, 4H), 3.48 (t, J = 7.0 Hz, 2H), 3.33 (t, J = 7.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ197.9, 186.8, 154.2, 152.4, 146.9, 136.5, 133.6, 129.3, 129.2, 128.8, 128.2, 121.6, 117.8, 112.5, 108.3, 108.2, 37.15, 36.62, 36.29. IR (cm^{-1}): v 3060, 3030, 2947, 1677, 1618, 1579, 1493, 1474, 1448, 1420, 1210, 1163, 982, 751. HRMS (TOF MS ESI): calcd for $C_{23}H_{21}N_2O_4^+$ $[M+H]^+$ 389.1501, found 389.1497.



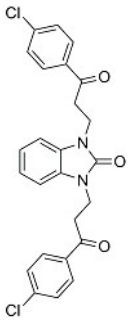
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-phenylpropan-1-one) (***5aa***): Yellow solid (43.8 mg, 55% yield), mp 133-134 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.4 Hz, 4H), 7.55 (t, J = 7.4 Hz, 2H), 7.43 (t, J = 7.7 Hz, 4H), 7.22-7.04 (m, 4H), 4.33 (t, J = 7.0 Hz, 4H), 3.48 (t, J = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 154.2, 136.4, 133.6, 129.2, 128.8, 128.5, 128.2, 121.5, 108.2, 37.1, 36.6. IR (cm^{-1}): ν 3058, 2958, 1700, 1675, 1619, 1580, 1448, 1415, 1370, 1327, 1211, 1157, 1077, 976, 743, 687. HRMS (TOF MS ESI): calcd for $C_{25}H_{23}N_2O_3^+$ $[M+H]^+$ 399.1709, found 399.1706.



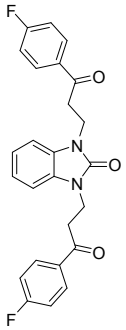
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(4-methoxyphenyl)propan-1-one) (***5ad***): White solid (59.6 mg, 65% yield), mp 193-194 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11-7.67 (m, 4H), 7.12-6.98 (m, 4H), 6.92-6.66 (m, 4H), 4.24 (t, J = 7.1 Hz, 4H), 3.77 (s, 6H), 3.34 (t, J = 7.1 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 163.9, 154.3, 130.5, 129.7, 129.3, 121.5, 113.9, 108.2, 55.6, 36.8, 36.8. IR (cm^{-1}): ν 2948, 2842, 1703, 1661, 1602, 1577, 1493, 1444, 1411, 1372, 1217, 1254, 1233, 1019, 980, 883, 759, 567. HRMS (TOF MS ESI): calcd for $C_{27}H_{27}N_2O_5^+$ $[M+H]^+$ 459.1920, found 459.1913.



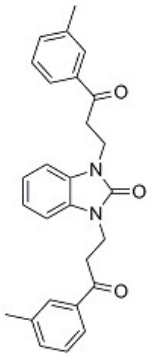
*3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(4-bromophenyl)propan-1-one) (**5ae**)*: Light yellow solid (47.8 mg, 43% yield), mp 180-181 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.62 (m, 4H), 7.62-7.41 (m, 4H), 7.12-6.89 (m, 4H), 4.24 (t, J = 7.0 Hz, 4H), 3.36 (t, J = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 153.1, 134.0, 131.0, 128.6, 128.1, 127.7, 120.5, 107.1, 35.9, 35.4. IR (cm^{-1}): v 2958, 2904, 1696, 1679, 1615, 1582, 1489, 1444, 1411, 1373, 1337, 1259, 1068, 1006, 980, 816, 744. HRMS (TOF MS ESI): calcd for $C_{25}H_{21}Br_2N_2O_3^+$ $[M+H]^+$ 554.9919, found 554.9915.



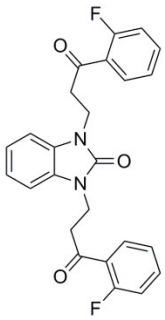
*3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(4-chlorophenyl)propan-1-one) (**5af**)*: White solid (47.7 mg, 51% yield), mp 149-150 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.74 (m, 4H), 7.40-7.28 (m, 4H), 7.14-7.00 (m, 4H), 4.23 (t, J = 7.0 Hz, 4H), 3.37 (t, J = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.7, 154.2, 140.1, 134.8, 129.6, 129.2, 129.1, 121.6, 108.2, 37.1, 36.5. IR (cm^{-1}): v 3060, 2956, 2909, 1702, 1676, 1619, 1588, 1495, 1440, 1400, 1375, 1209, 1090, 1012, 771, 730. HRMS (TOF MS ESI): calcd for $C_{25}H_{21}Cl_2N_2O_3^+$ $[M+H]^+$ 467.0929, found 467.0941.



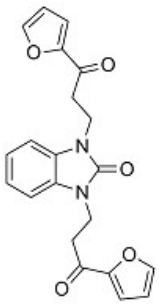
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(4-fluorophenyl)propan-1-one) (***5ag***): Yellow solid (43.4 mg, 50% yield), mp 128-129 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05-7.86 (m, 4H), 7.24-7.00 (m, 8H), 4.32 (t, J = 6.9 Hz, 4H), 3.45 (t, J = 6.9 Hz, 4H).^{13}C NMR (101 MHz, $CDCl_3$) δ 196.2, 167.3, 164.7, 154.2, 132.9, 130.9, 130.8, 129.2, 121.5, 116.0, 115.8, 108.2, 37.0, 36.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.4. IR (cm^{-1}): v 3084, 2928, 1694, 1679, 1595, 1495, 1411, 1375, 1321, 1228, 1209, 1154, 984, 732. HRMS (TOF MS ESI): calcd for $C_{25}H_{21}F_2N_2O_3^+$ $[M+H]^+$ 435.1520, found 435.1516.



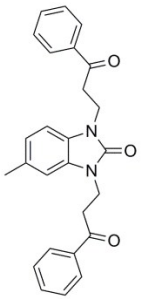
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(m-tolyl)propan-1-one) (***5ai***): White solid (38.4 mg, 45% yield), mp 130-131 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.68 (m, 4H), 7.39-7.28 (m, 4H), 7.21-7.04 (m, 4H), 4.32 (t, J = 7.0 Hz, 4H), 3.47 (t, J = 7.0 Hz, 4H), 2.37 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 154.2, 138.6, 136.5, 134.3, 129.3, 128.7, 128.6, 125.4, 121.5, 108.2, 37.2, 36.5, 21.4. IR (cm^{-1}): v 3058, 2950, 2918, 2229, 1672, 1605, 1586, 1497, 1413, 1373, 1241, 1174, 1157, 1041, 917, 688. HRMS (TOF MS ESI): calcd for $C_{27}H_{27}N_2O_3^+$ $[M+H]^+$ 427.2022, found 427.2017.



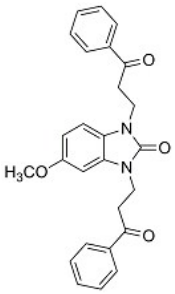
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(2-fluorophenyl)propan-1-one) ***(5aj)***: Yellow solid (37.4 mg, 43% yield), mp 108-109 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (td, J = 7.7, 1.7 Hz, 2H), 7.58-7.45 (m, 2H), 7.24-7.18 (m, 2H), 7.17-7.06 (m, 6H), 4.32 (t, J = 7.0 Hz, 4H), 3.55-3.41 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 196.0, 163.5, 161.0, 154.1, 135.2, 135.1, 130.7, 130.7, 129.3, 125.2, 125.0, 124.6, 124.6, 121.4, 117.0, 116.7, 108.1, 42.0, 41.9, 36.3, 36.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.9. IR (cm^{-1}): v 3060, 2950, 2920, 2365, 1679, 1608, 1541, 1493, 1478, 1396, 1375, 1360, 1330, 1280, 1105, 950, 766. HRMS (TOF MS ESI): calcd for $C_{25}H_{20}F_2N_2O_3Na^+$ $[M+Na]^+$ 457.1340, found457.1341.



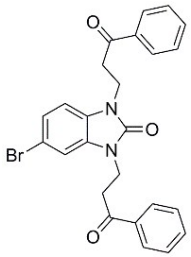
3,3'-(2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-(furan-2-yl)propan-1-one) ***(5am)***: Yellow solid (39.4 mg, 52% yield), mp 134-135 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.52 (m, 2H), 7.21 (d, J = 3.5 Hz, 2H), 7.17-7.05 (m, 4H), 6.51 (dd, J = 3.6, 1.7 Hz, 2H), 4.29 (t, J = 6.9 Hz, 4H), 3.32 (t, J = 6.9 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.7, 154.2, 152.4, 146.9, 129.2, 121.6, 117.8, 112.5, 108.2, 37.1, 36.3. IR (cm^{-1}): v 3114, 2920, 2853, 1679, 1664, 1619, 1561, 1467, 1396, 1370, 1163, 1030, 786,732. HRMS (TOF MS ESI): calcd for $C_{21}H_{19}N_2O_5^+$ $[M+H]^+$ 379.1294, found 379.1293.



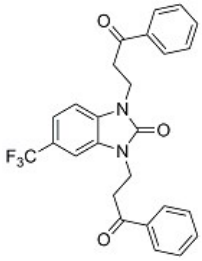
3,3'-(5-methyl-2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-phenylpropan-1-one) (***5ba***): Yellow solid (37.1 mg, 45% yield), mp 111-112 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.87 (m, 4H), 7.59-7.51 (m, 2H), 7.44 (td, J = 7.8, 2.3 Hz, 4H), 7.04 (d, J = 7.9 Hz, 1H), 6.98-6.87 (m, 2H), 4.30 (t, J = 6.9 Hz, 4H), 3.46 (t, J = 7.1 Hz, 4H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 197.9, 154.4, 136.5, 133.5, 131.3, 129.4, 128.8, 128.8, 128.2, 127.2, 122.0, 108.7, 107.9, 37.2, 36.6, 36.6, 21.6. IR (cm^{-1}): v 3060, 2952, 2920, 2853, 1672, 1627, 1597, 1502, 1374, 1325, 1211, 1168, 1142, 1000, 977, 688. HRMS (TOF MS ESI): calcd for $C_{26}H_{25}N_2O_3^+$ $[M+H]^+$ 413.1865, found 413.1860.



3,3'-(5-methoxy-2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-phenylpropan-1-one) (***5ca***): Yellow solid (41.1 mg, 48% yield), mp 122-123 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05-7.83 (m, 4H), 7.55 (t, J = 7.0 Hz, 2H), 7.44 (t, J = 7.6 Hz, 4H), 7.05 (d, J = 8.6 Hz, 1H), 6.77 (d, J = 2.3 Hz, 1H), 6.67 (dd, J = 8.6, 2.3 Hz, 1H), 4.29 (t, J = 7.0 Hz, 4H), 3.83 (s, 3H), 3.46 (t, J = 7.0 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 197.9, 155.7, 154.6, 136.5, 136.5, 133.6, 133.6, 130.1, 128.8, 128.2, 123.4, 108.5, 107.0, 95.5, 56.2, 37.2, 37.2, 36.6. IR (cm^{-1}): v 3062, 3000, 2903, 2833, 1672, 1610, 1579, 1578, 1500, 1446, 1420, 1373, 1325, 1208, 1150, 977, 740, 688. HRMS (TOF MS ESI): calcd for $C_{26}H_{25}N_2O_4^+$ $[M+H]^+$ 429.1814, found 429.1811.



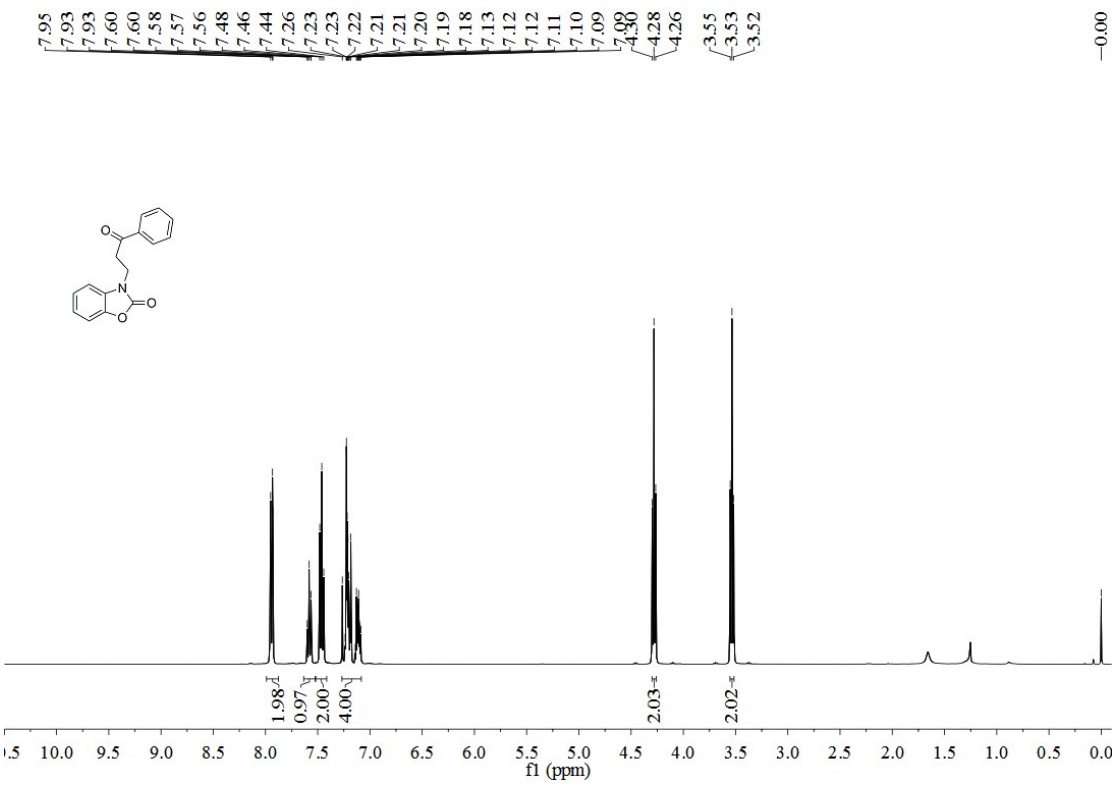
3,3'-(5-bromo-2-oxo-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-phenylpropan-1-one) ***(5da)***: Yellow solid (47.7 mg, 50% yield), mp 115-116 ^{o}C, ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.93 (dd, J = 7.0, 4.7 Hz, 4H), 7.56 (t, J= 7.2 Hz, 2H), 7.44 (t, J = 6.8 Hz, 4H), 7.30 (d, J = 1.2 Hz, 1H), 7.24-7.19 (m, 1H), 7.07 (d, J = 8.3 Hz, 1H), 4.28 (m, 4H), 3.47 (t, J = 6.5 Hz, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 197.6, 154.1, 136.4, 133.7, 133.7, 130.5, 128.8, 128.5, 128.2, 124.3, 114.2, 111.4, 109.6, 37.0, 37.0, 36.8, 36.7. IR (cm^{-1}): v 3058, 2954, 2917, 2850, 1692, 1619, 1672, 1493, 1446, 1413, 1372, 1325, 1238, 1057, 975, 954, 738. HRMS (TOF MS ESI): calcd for $C_{25}H_{22}BrN_2O_3^+$ $[M+H]^+$ 477.0814, found 477.0816.



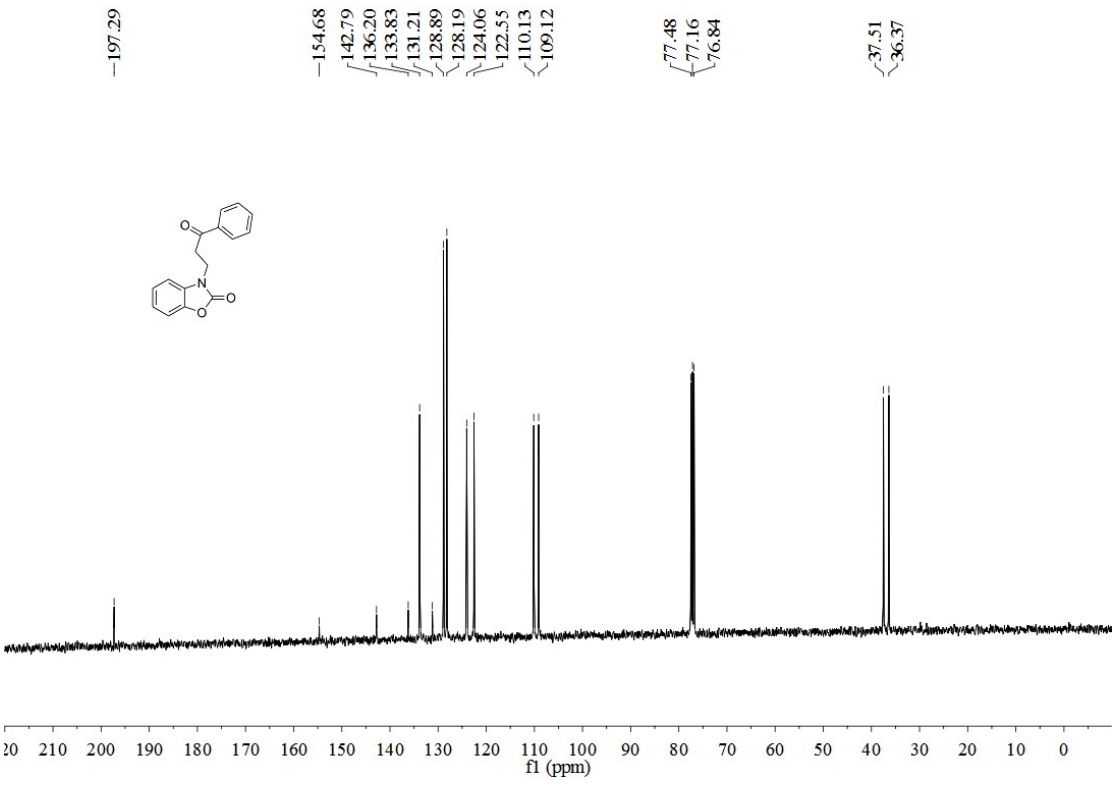
3,3'-(2-oxo-5-(trifluoromethyl)-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(1-phenylpropan-1-one) ***(5ea)***: Yellow solid (50.4mg, 54% yield), mp 107-108 ^{o}C, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 7.8 Hz, 4H), 7.56 (t, J = 7.0 Hz, 2H), 7.48-7.37 (m, 6H), 7.30 (d, J = 8.6 Hz, 1H), 4.46-4.25 (m, 4H), 3.50 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 197.4, 154.3, 136.3, 133.6, 133.6, 131.9, 129.3, 128.7, 128.1, 126.0, 123.9, 123.6, 123.3, 118.9, 108.2, 105.4, 36.9, 36.8, 36.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -60.8. IR (cm^{-1}): v 3060, 2919, 1702, 1677, 1597, 1508, 1480, 1448, 1407, 1370, 1320, 1267, 1211, 1154, 1109, 1059, 863, 740. HRMS (TOF MS ESI): calcd for $C_{26}H_{22}F_3N_2O_3^+$ $[M+H]^+$ 467.1583, found 467.1576.

7. ^{1}H and ^{13}C NMR spectra for compound 3 and 5

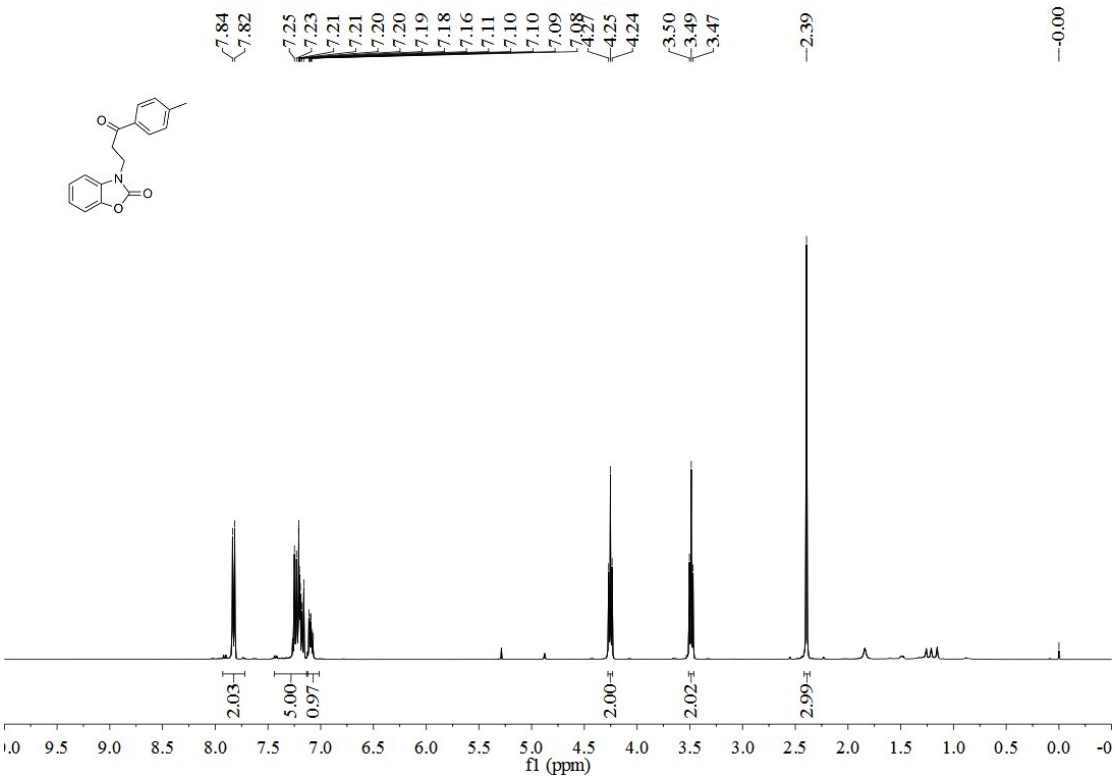
^{1}H NMR spectrum of ***3aa***



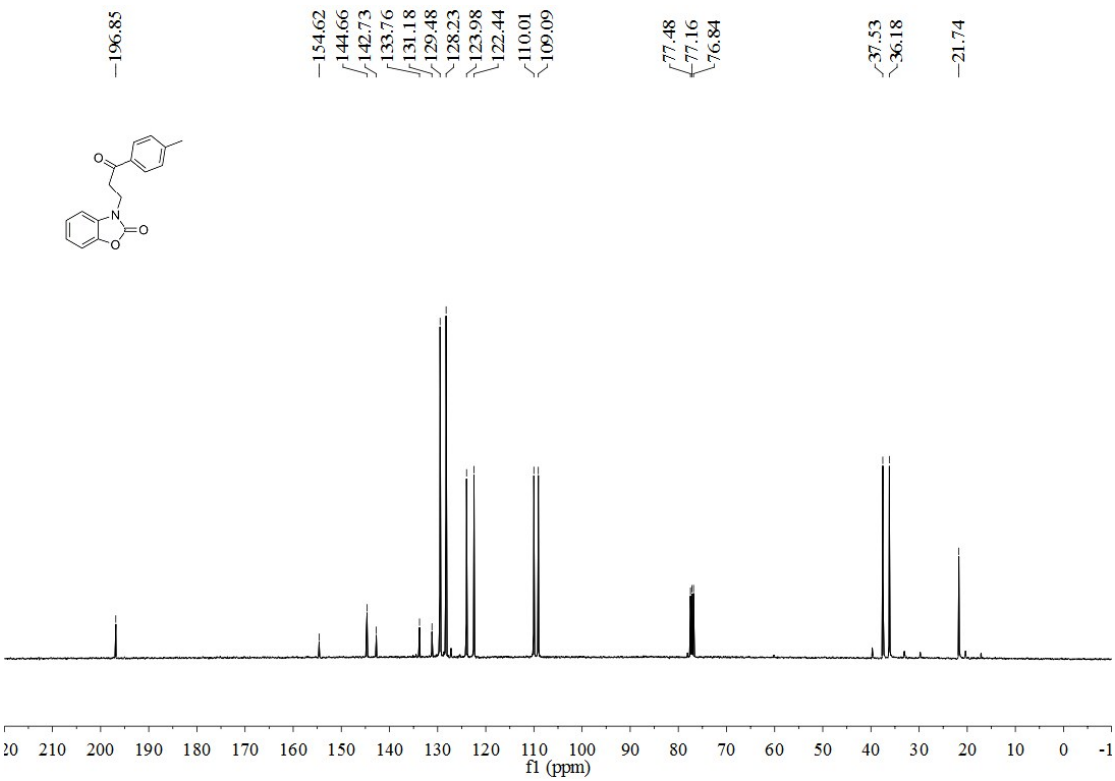
^{13}C NMR spectrum of ***3aa***



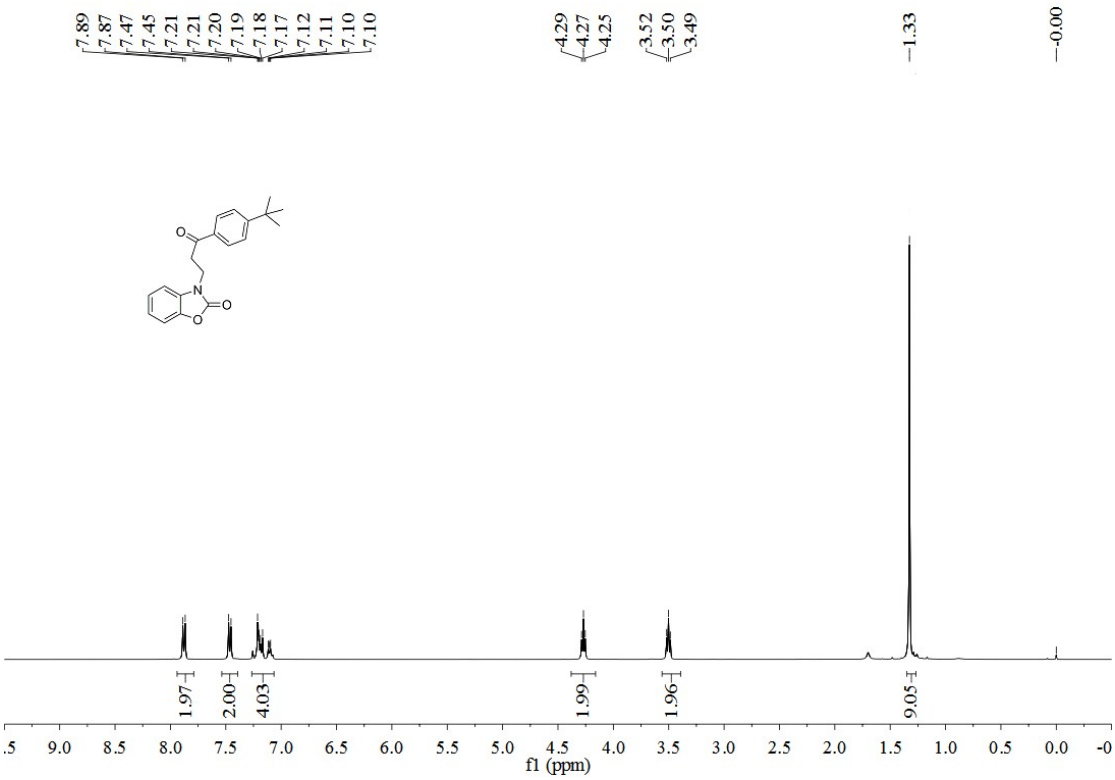
^{1}H NMR spectrum of ***3ab***



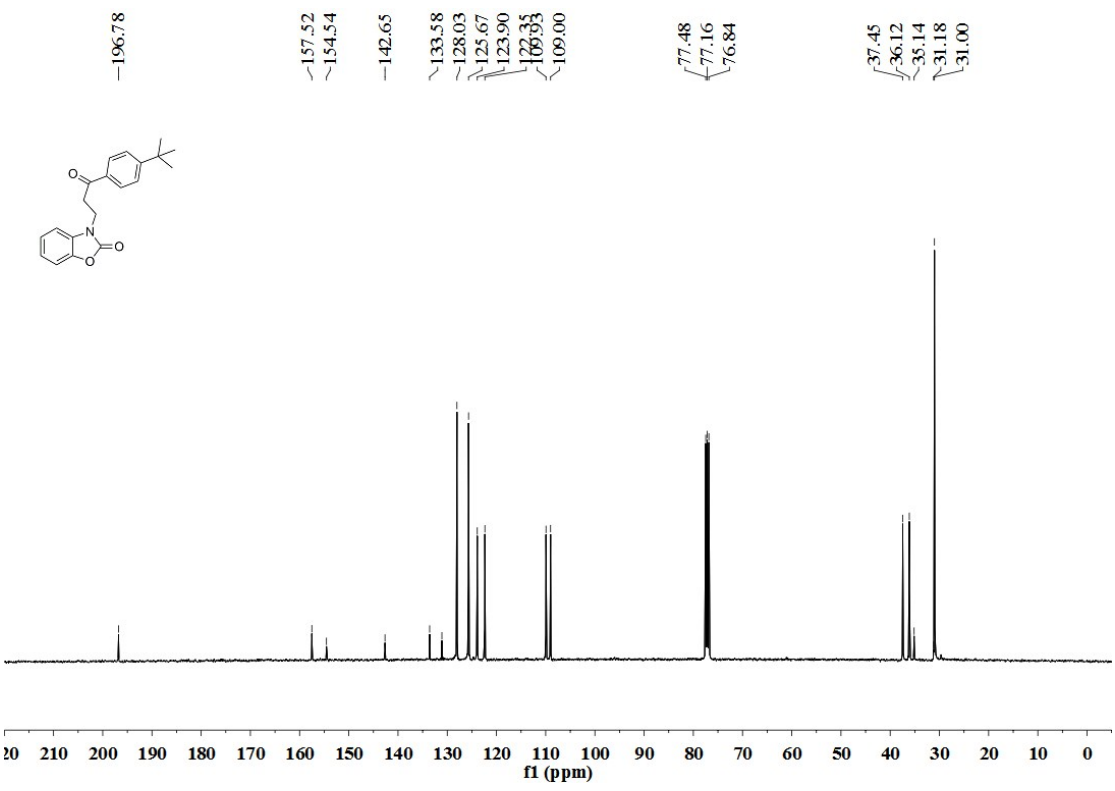
^{13}C NMR spectrum of***3ab***



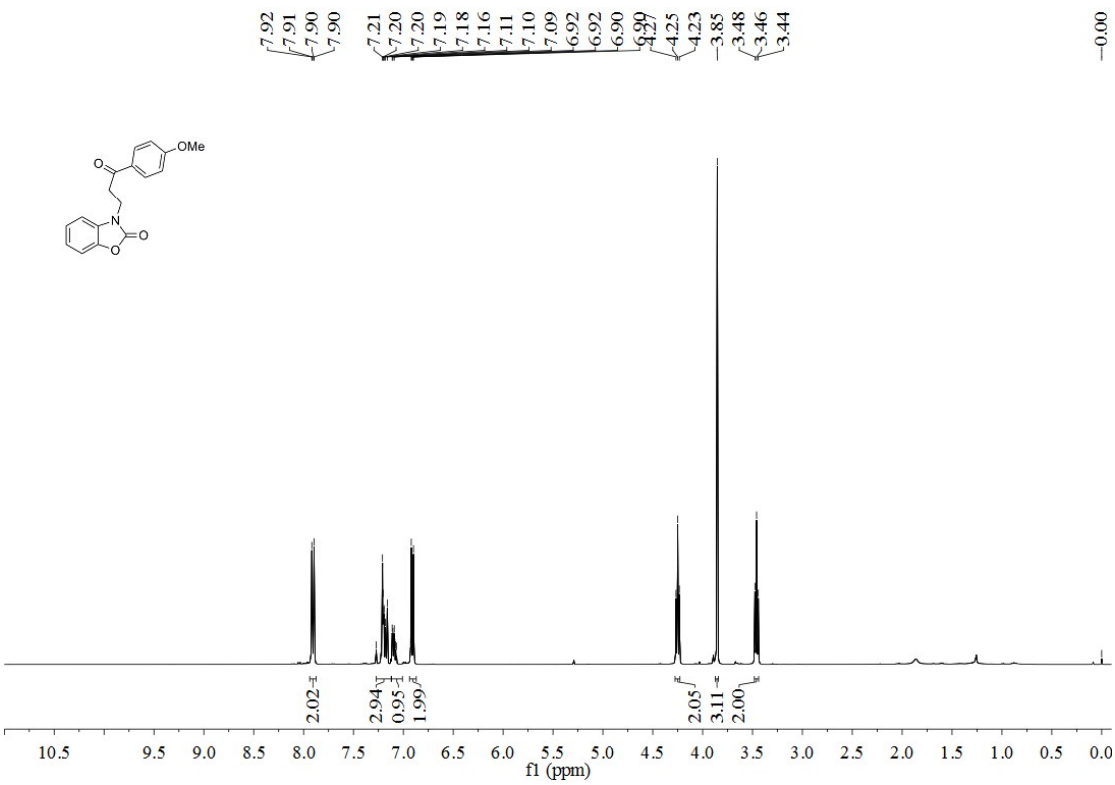
^{1}H NMR spectrum of ***3ac***



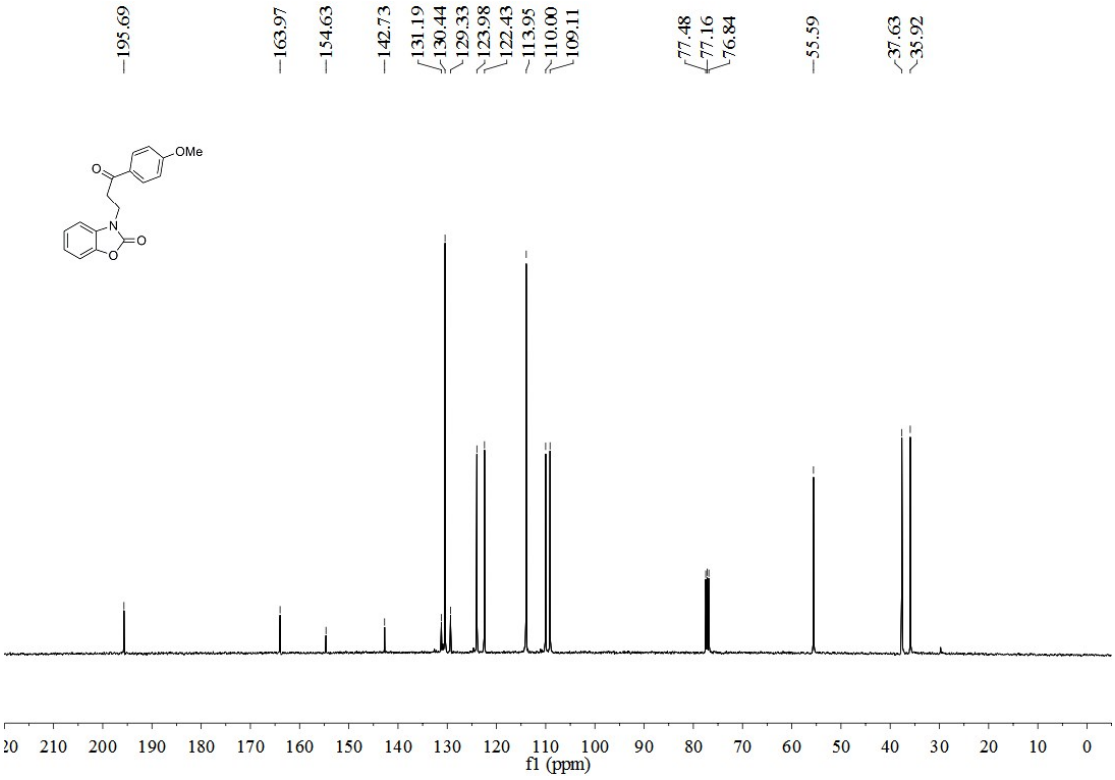
^{13}C NMR spectrum of***3ac***



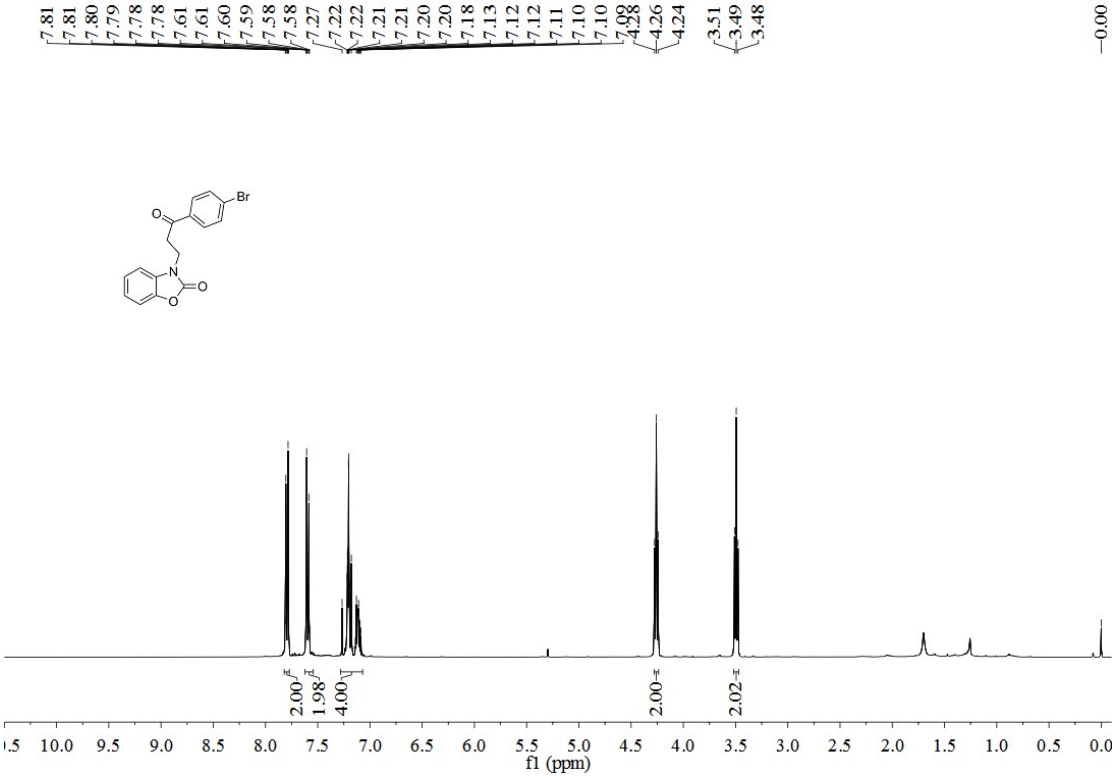
^{1}H NMR spectrum of ***3ad***



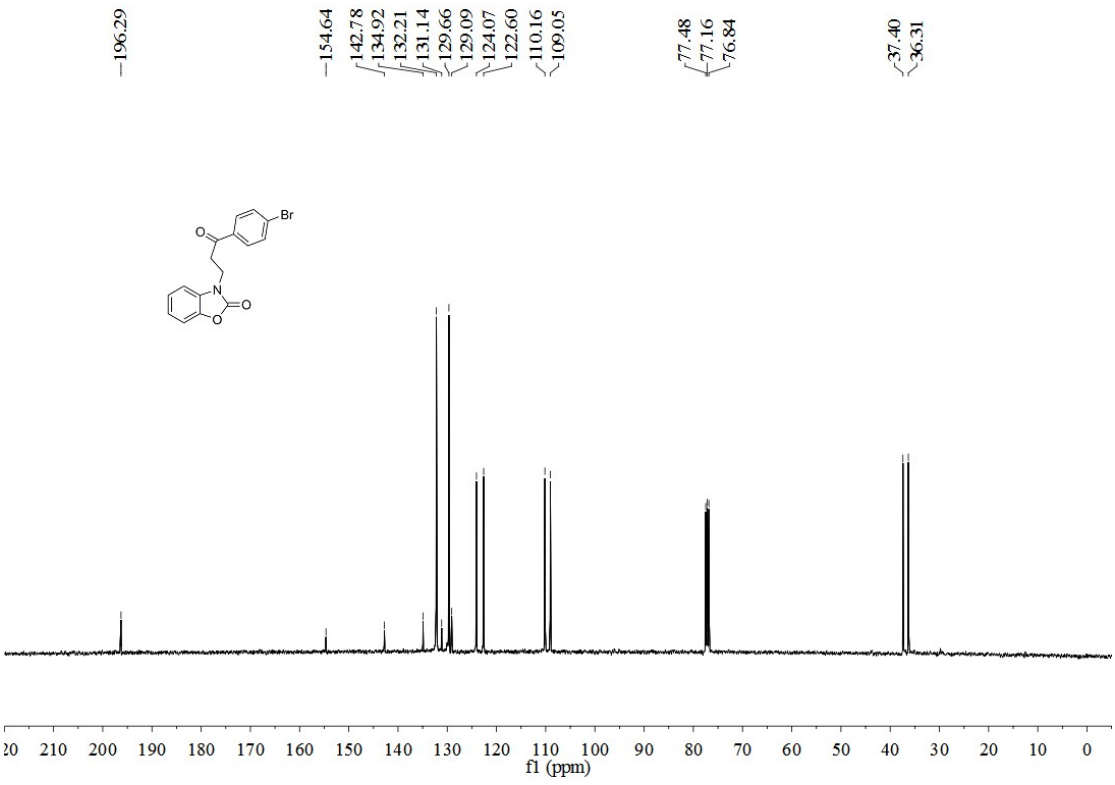
^{13}C NMR spectrum of ***3ad***



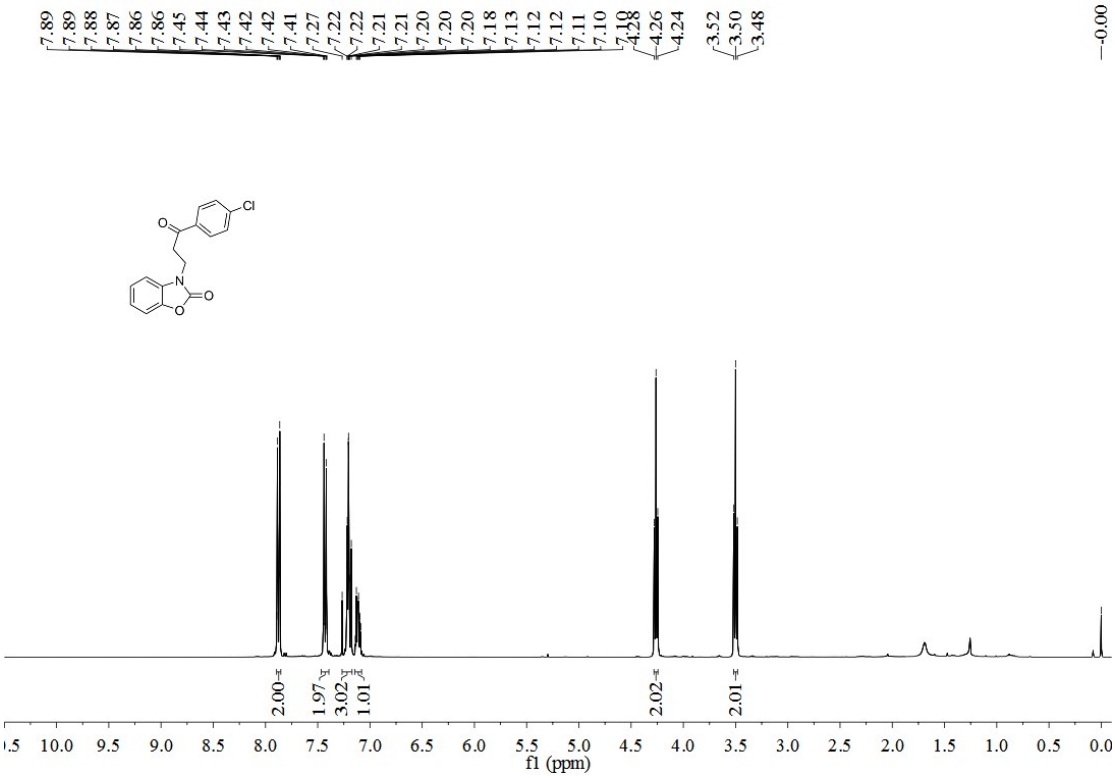
^{1}H NMR spectrum of ***3ae***



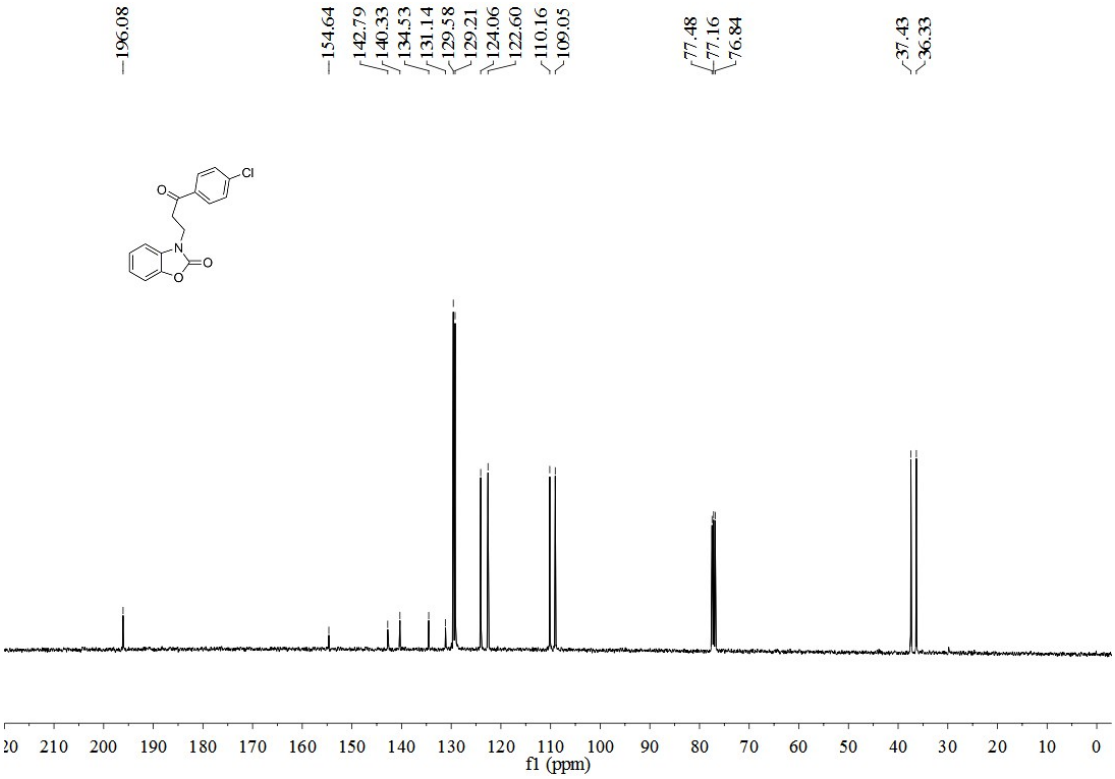
^{13}C NMR spectrum of ***3ae***



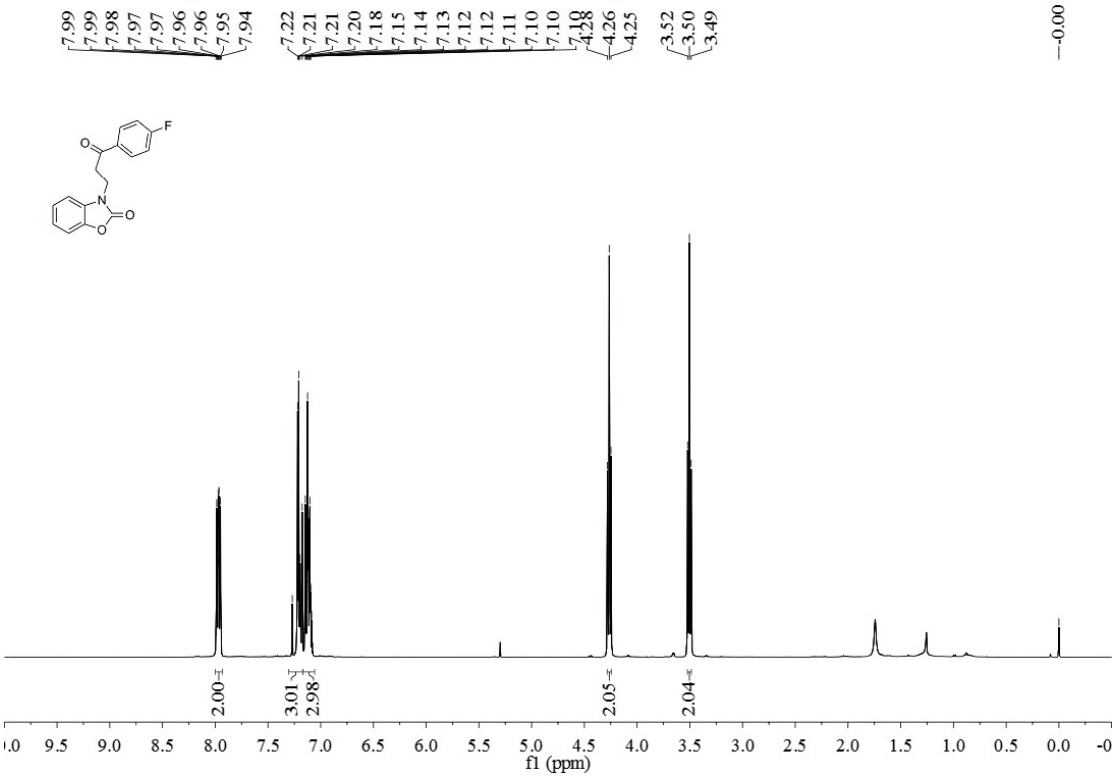
^{1}H NMR spectrum of ***3af***



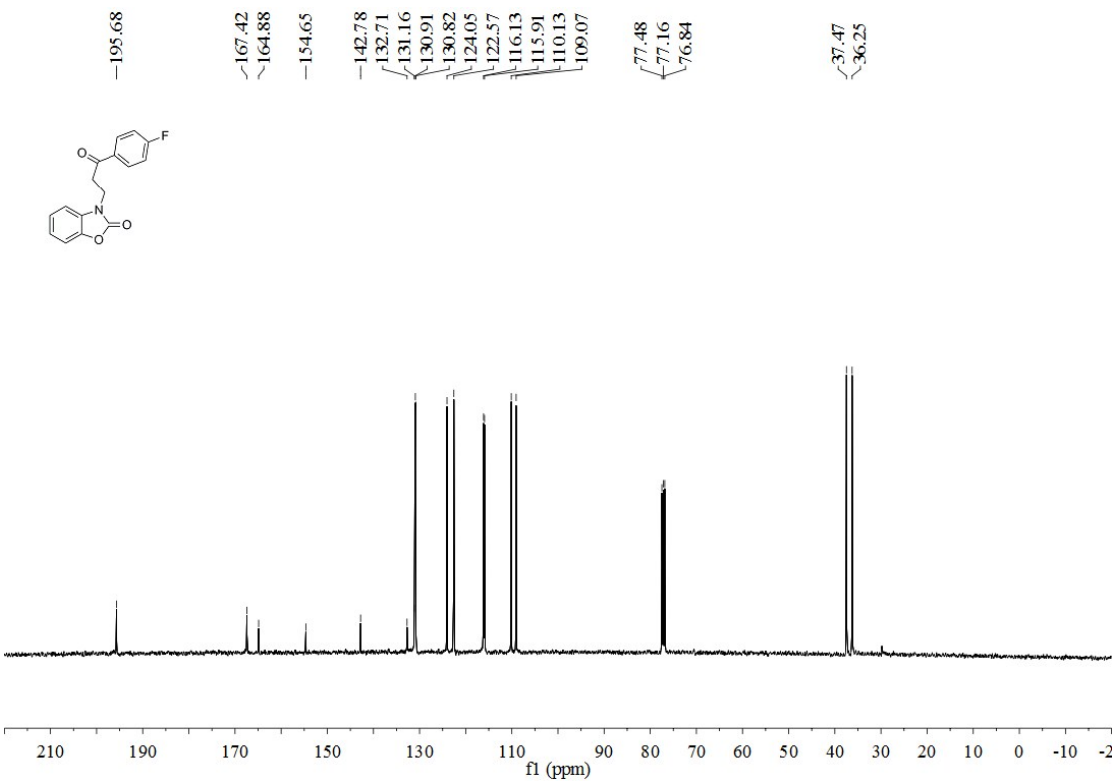
^{13}C NMR spectrum of ***3af***



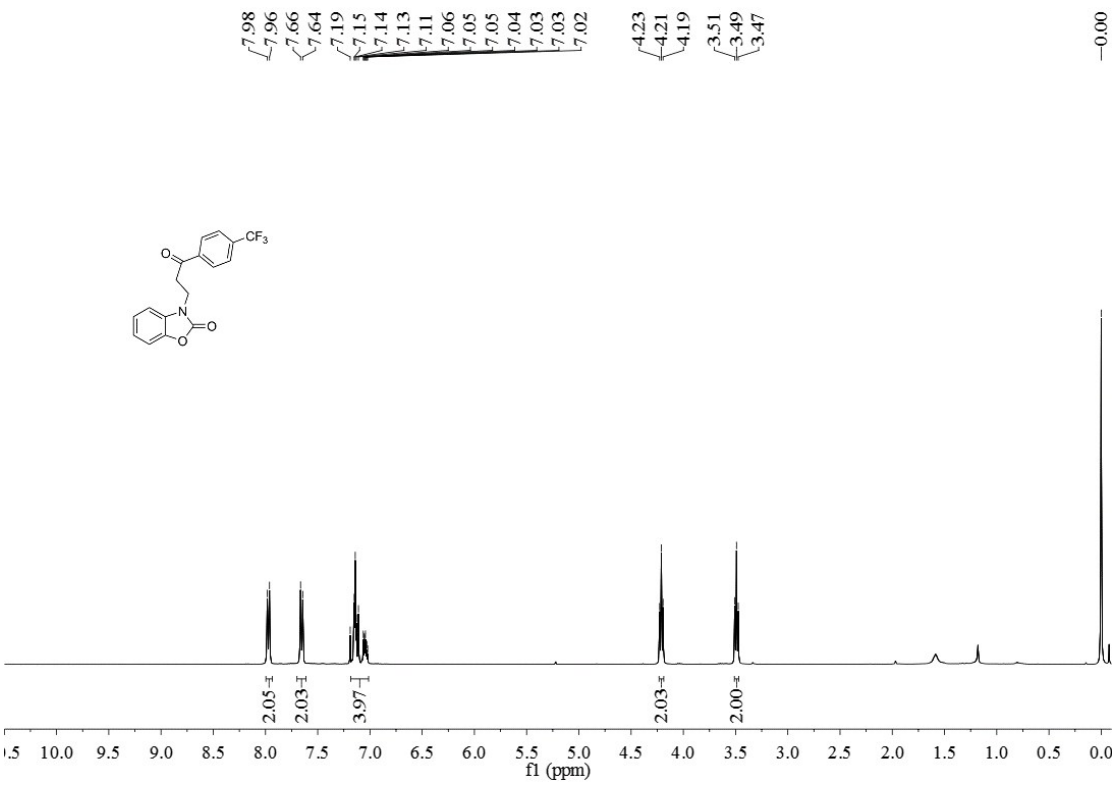
^{1}H NMR spectrum of ***3ag***



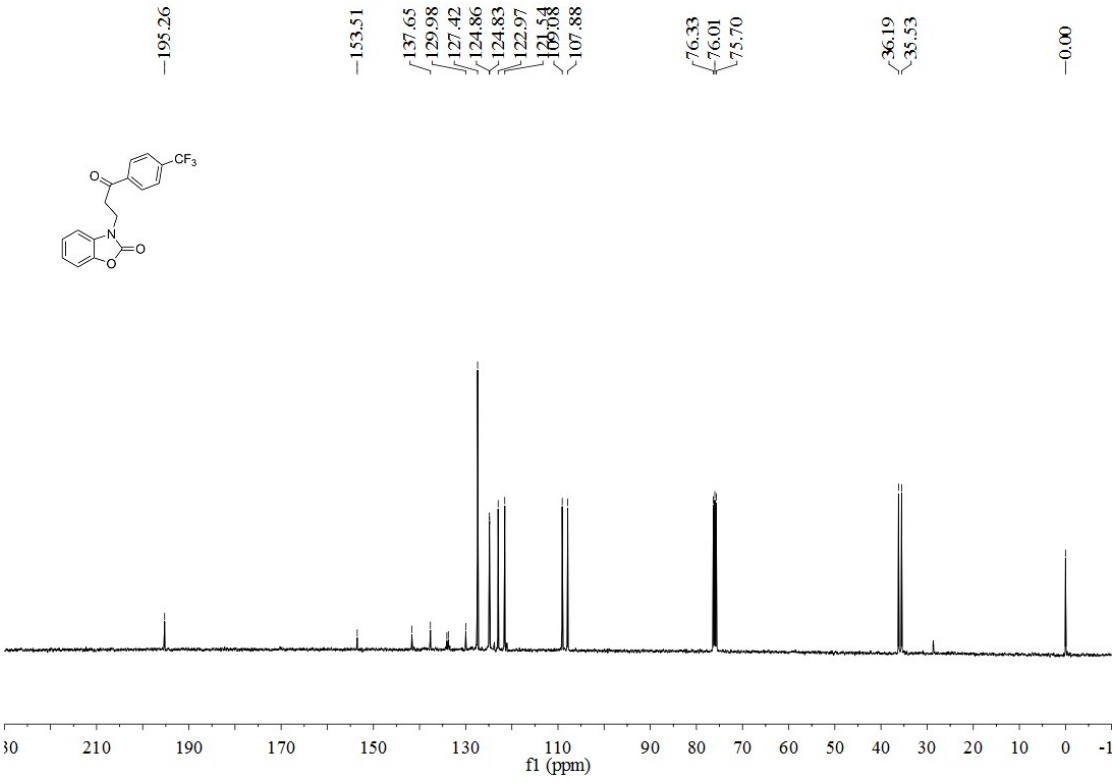
^{13}C NMR spectrum of ***3ag***



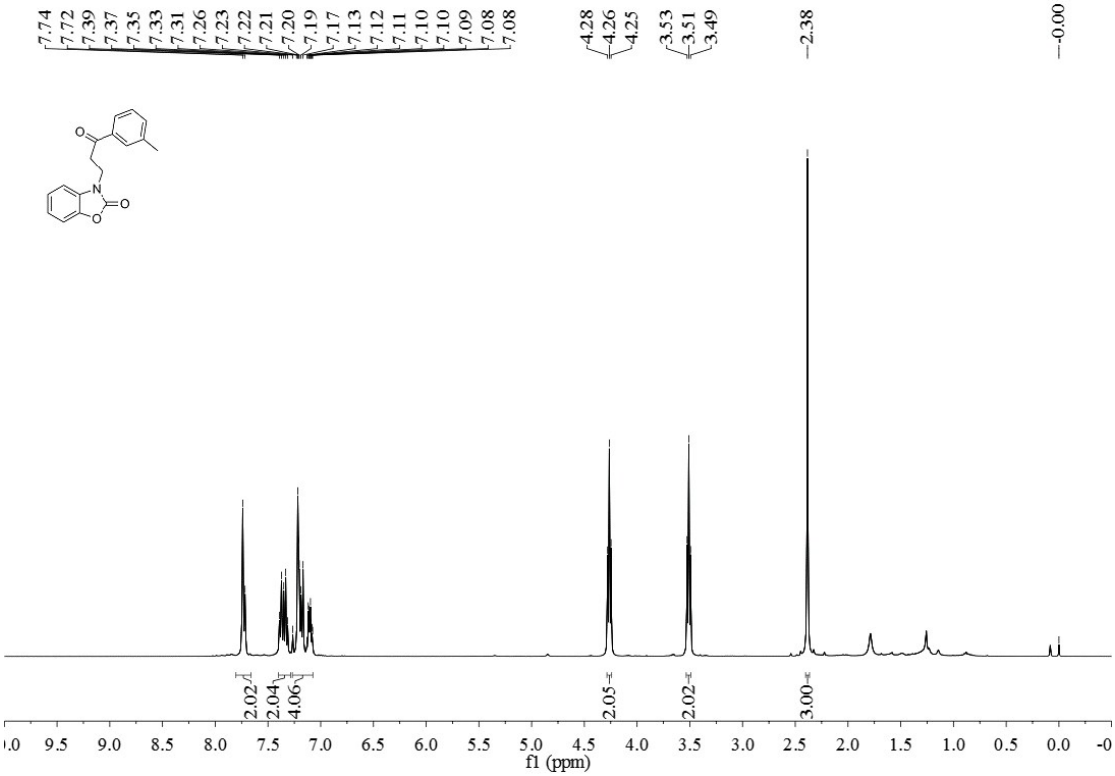
^{1}H NMR spectrum of***3ah***



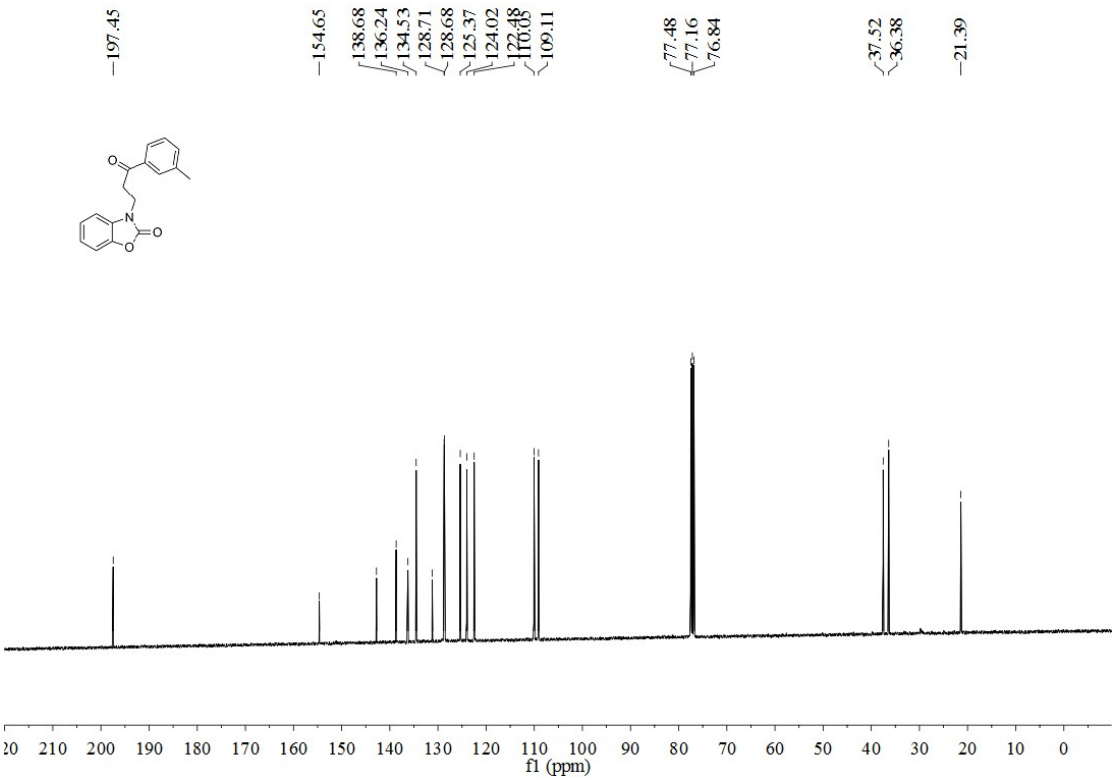
^{13}C NMR spectrum of ***3ah***



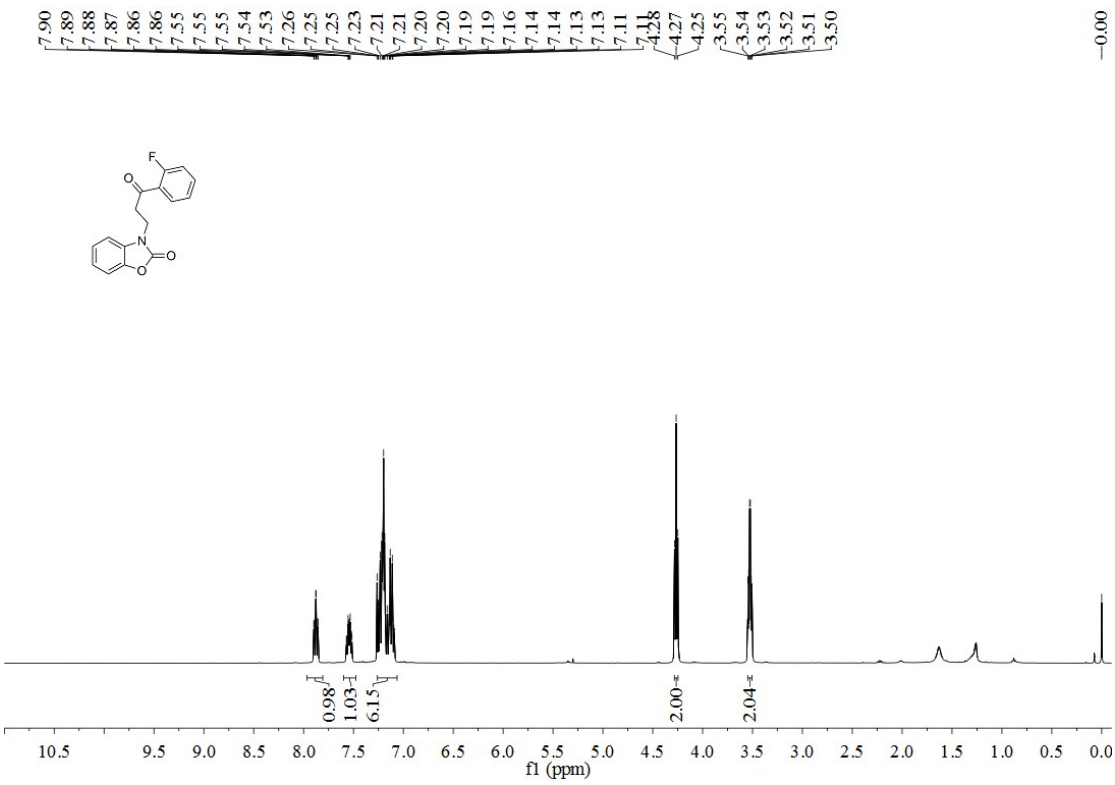
^{1}H NMR spectrum of ***3ai***



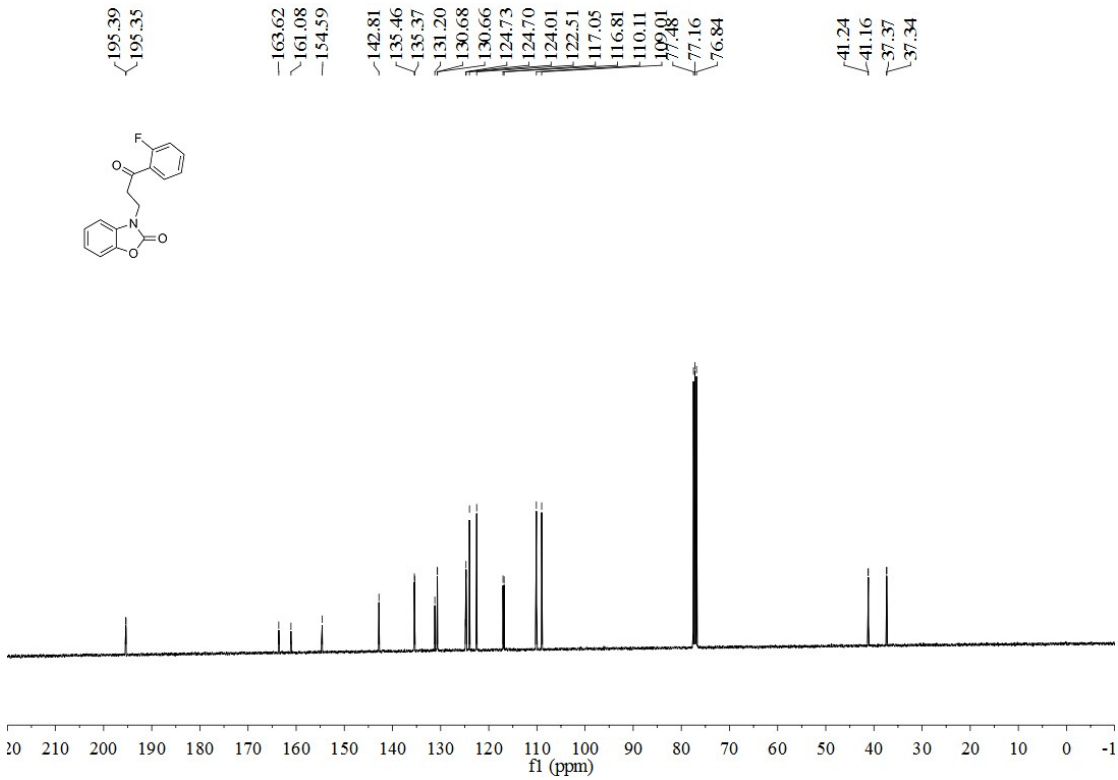
^{13}C NMR spectrum of ***3ai***



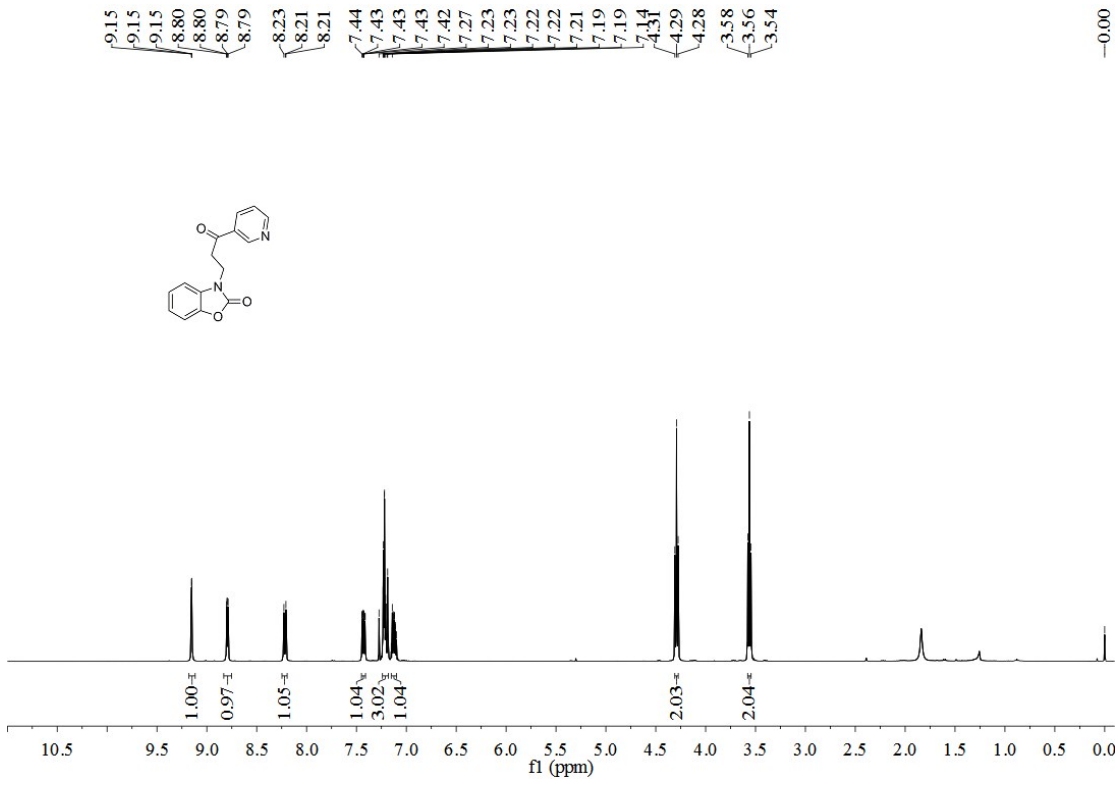
^{1}H NMR spectrum of ***3aj***



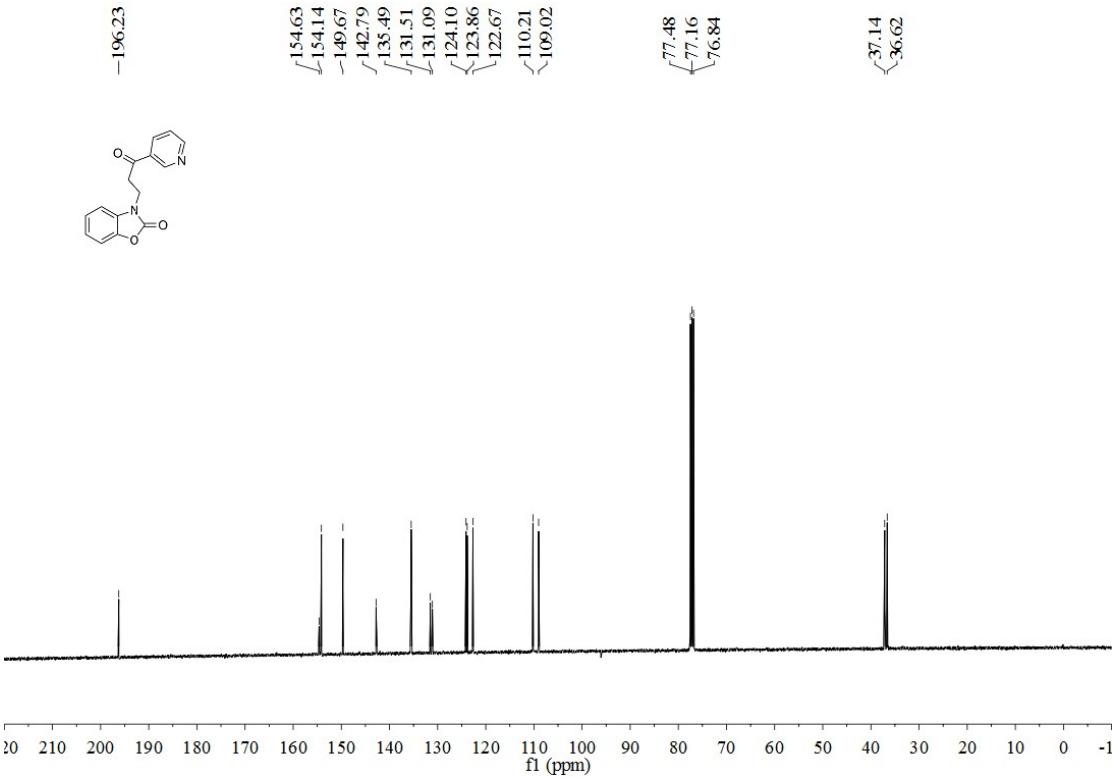
^{13}C NMR spectrum of***3aj***



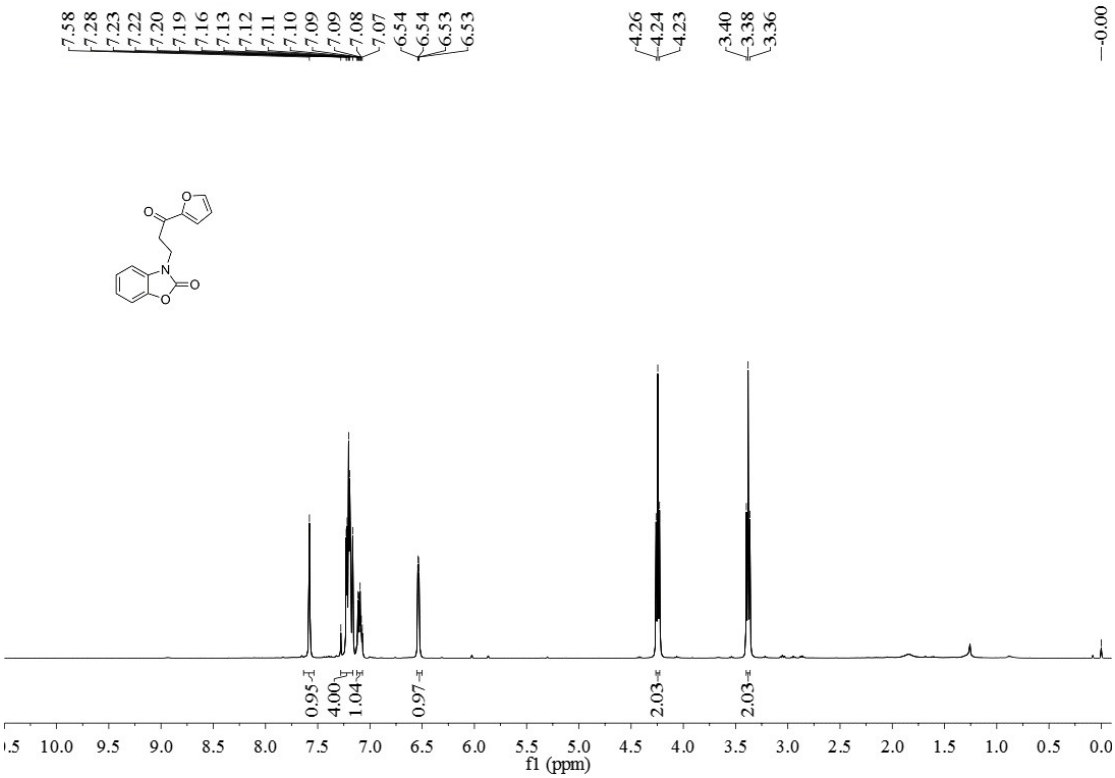
^{1}H NMR spectrum of ***3ak***



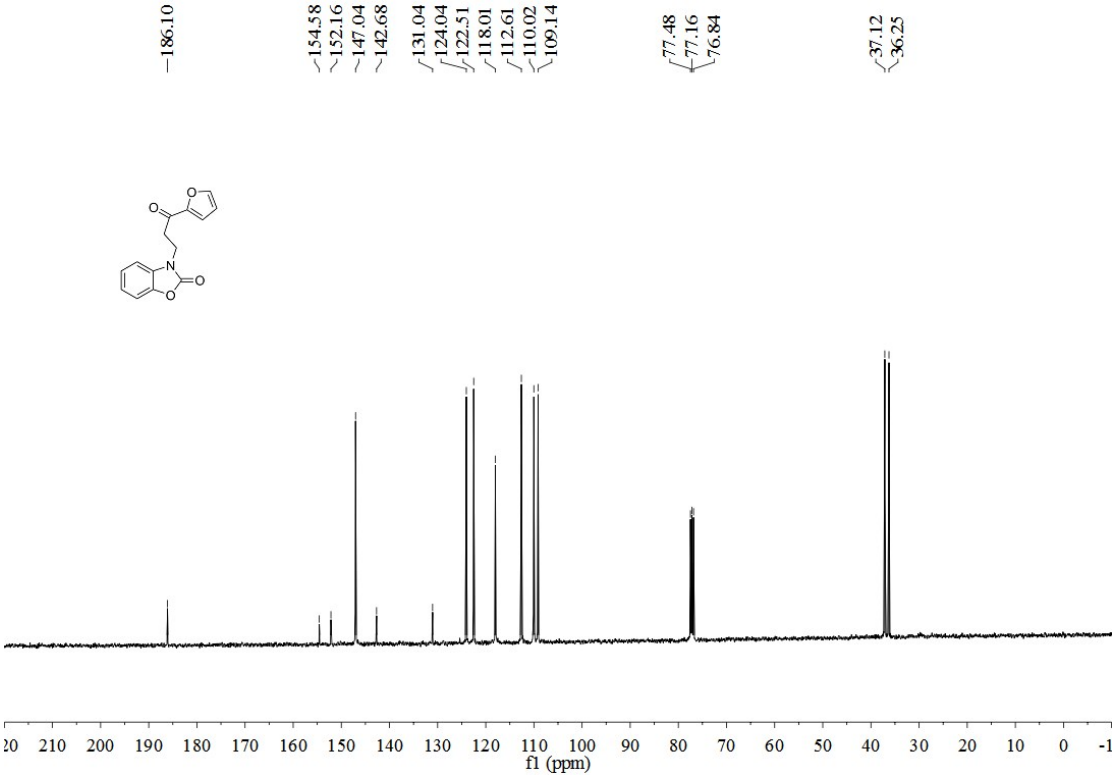
^{13}C NMR spectrum of ***3ak***



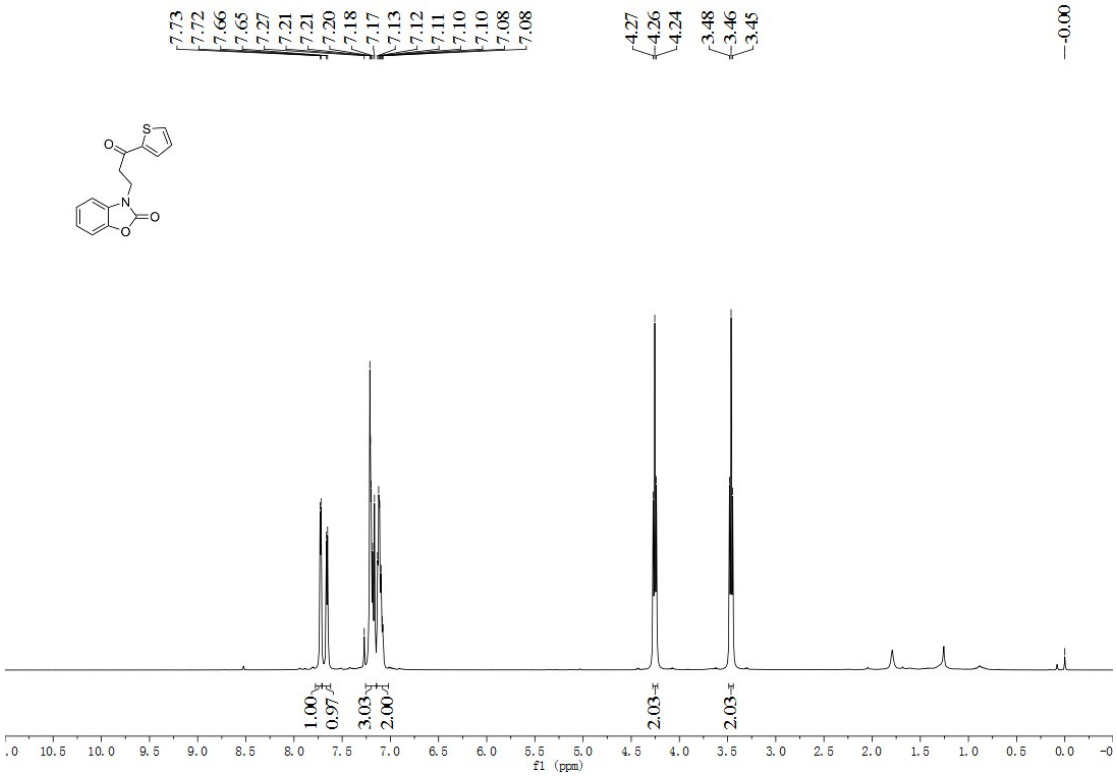
^{1}H NMR spectrum of ***3al***



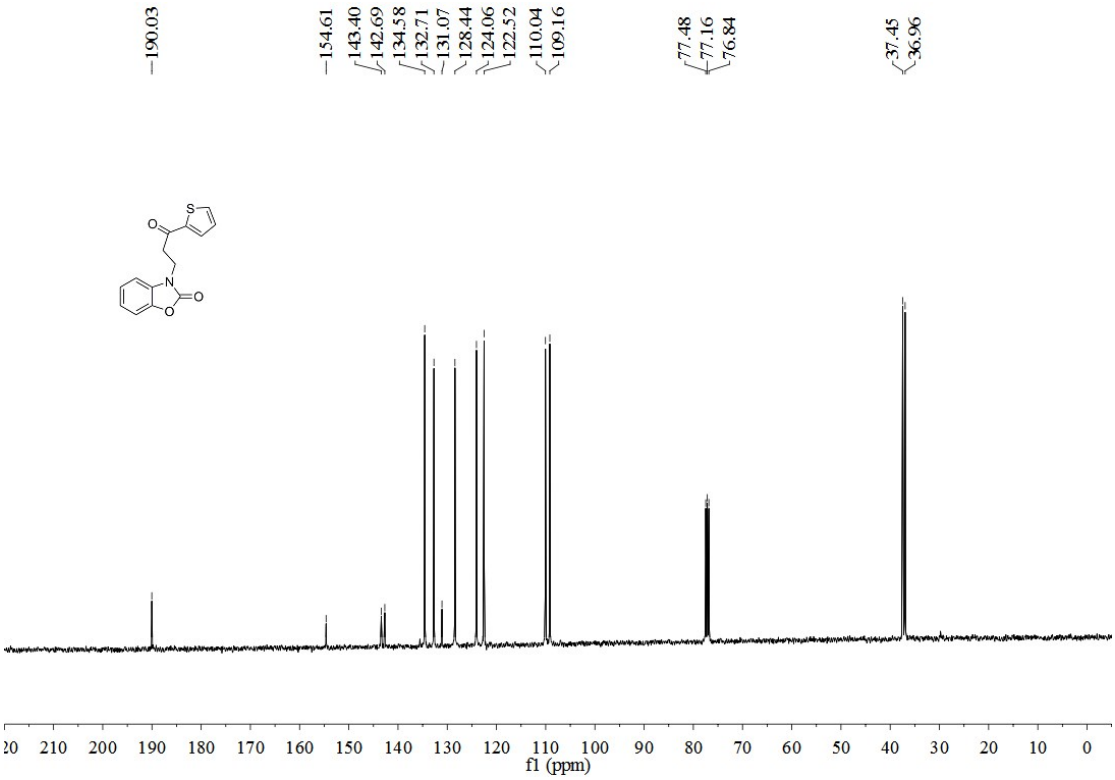
^{13}C NMR spectrum of ***3al***



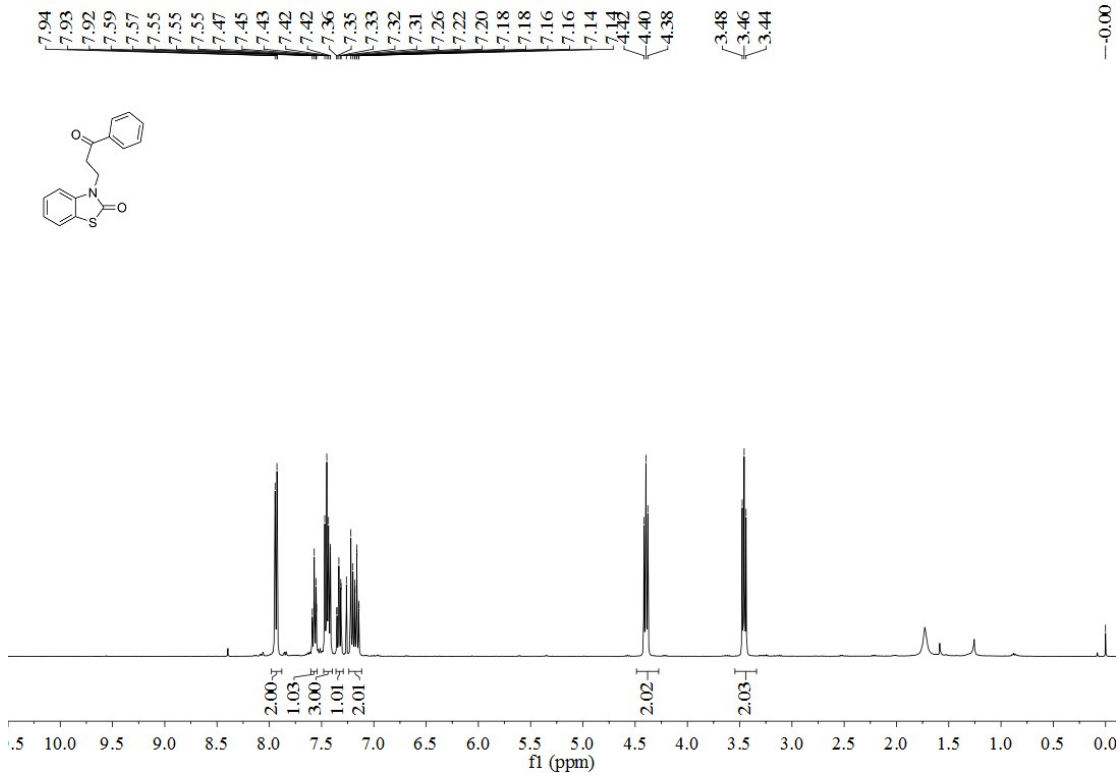
^{1}H NMR spectrum of ***3am***



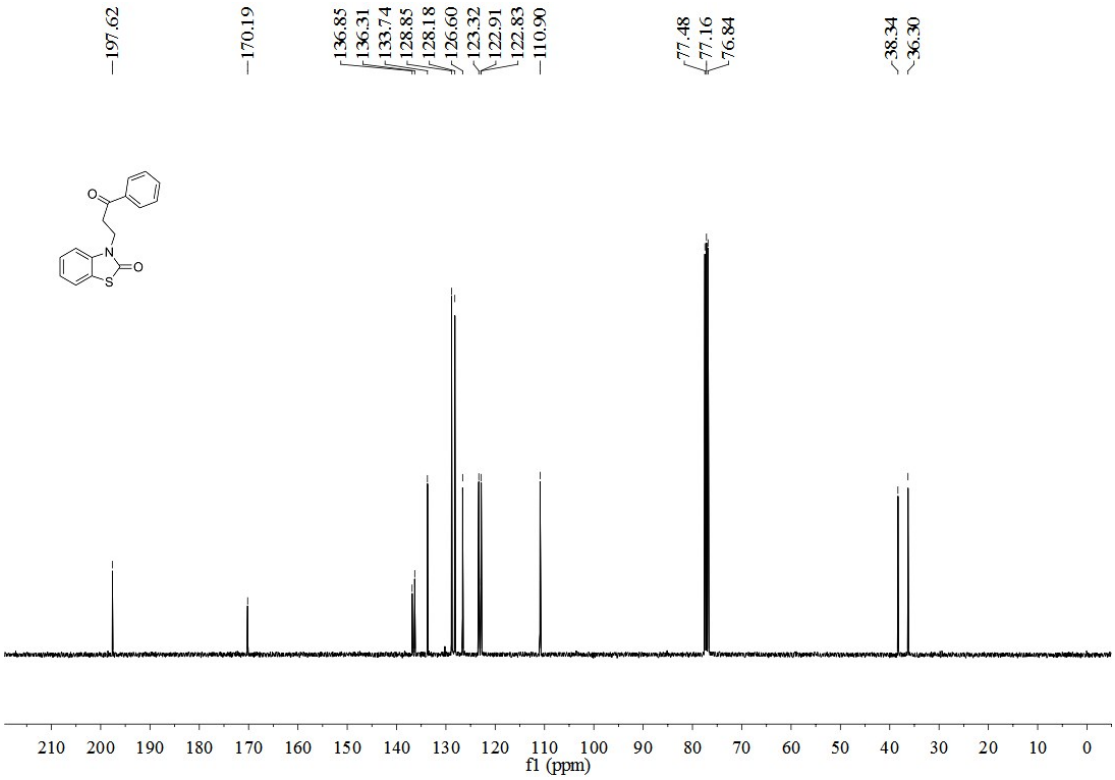
^{13}C NMR spectrum of ***3am***



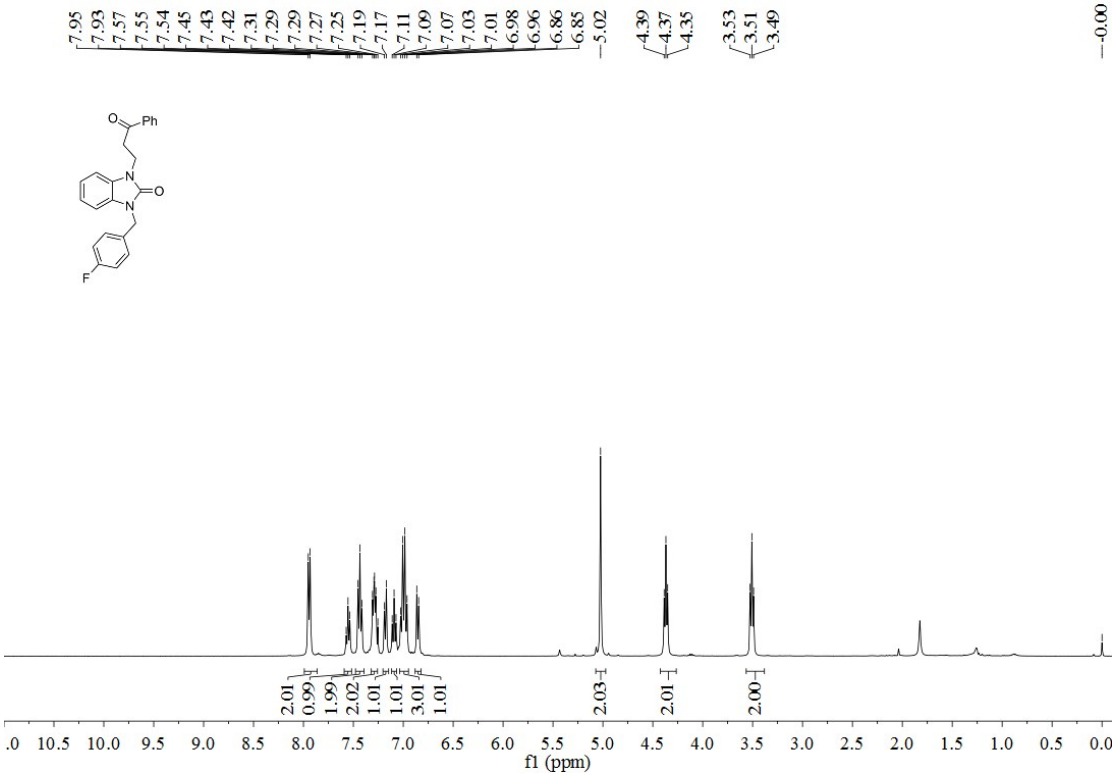
^{1}H NMR spectrum of ***3ba***



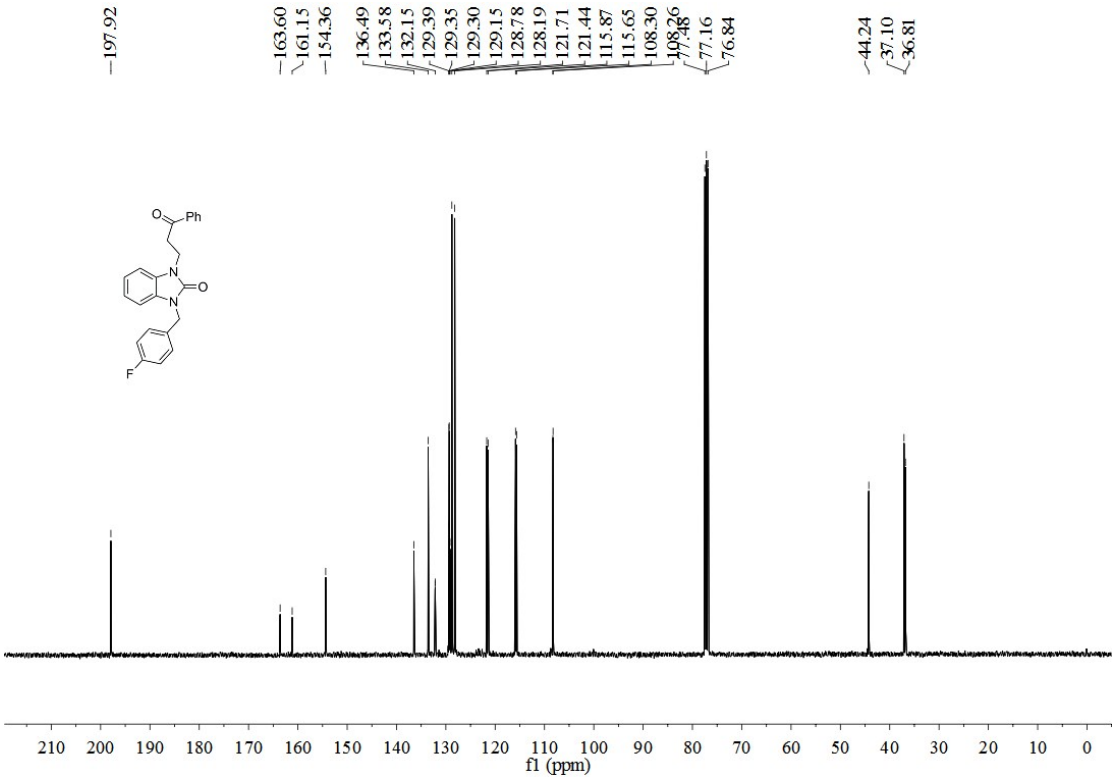
^{13}C NMR spectrum of ***3ba***



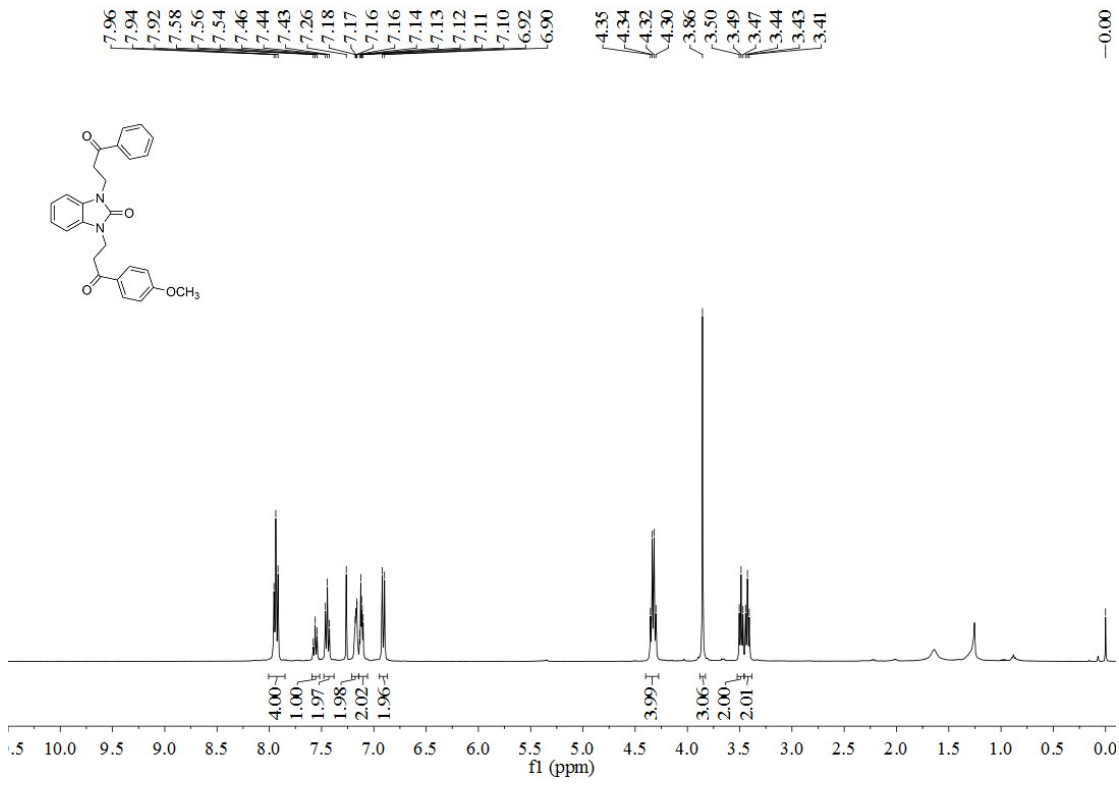
^{1}H NMR spectrum of ***3ca***



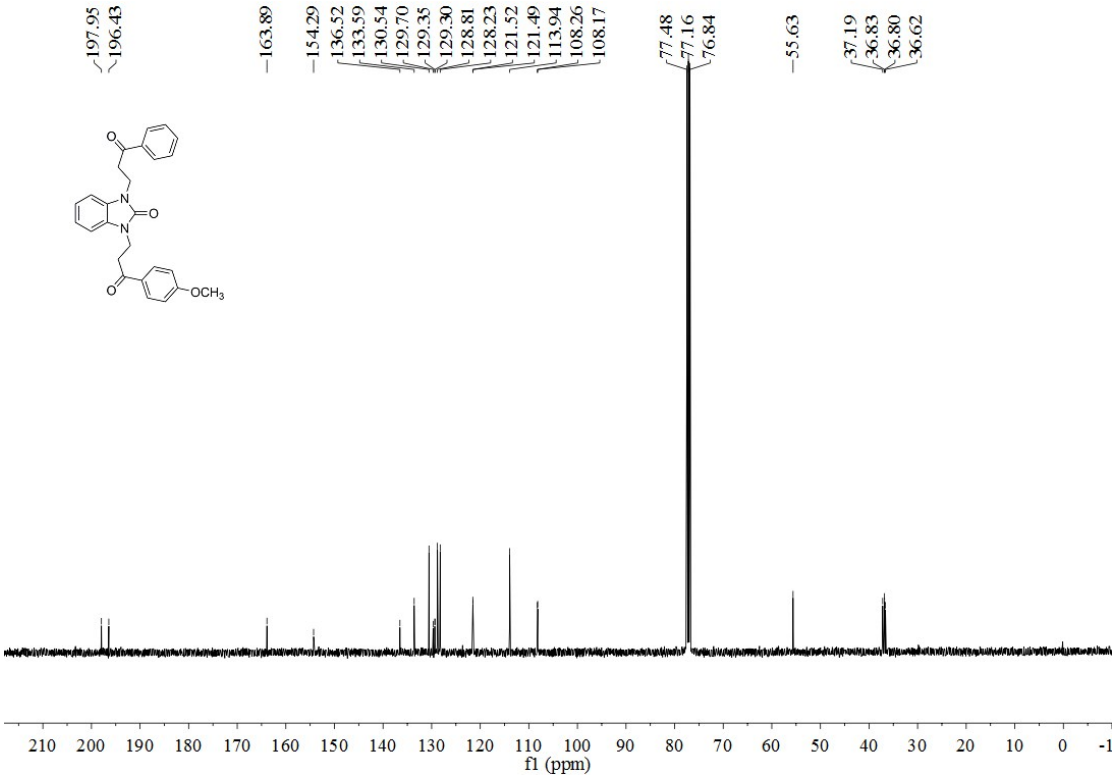
^{13}C NMR spectrum of **3ca**



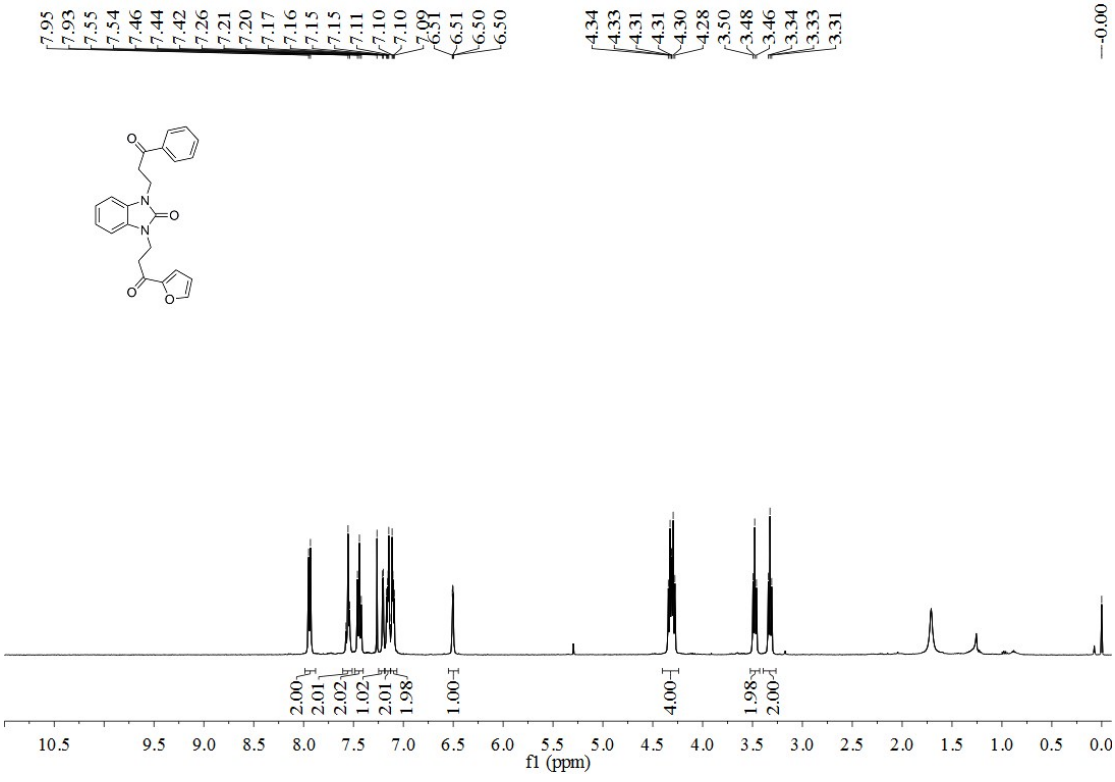
^{1}H NMR spectrum of ***3da***



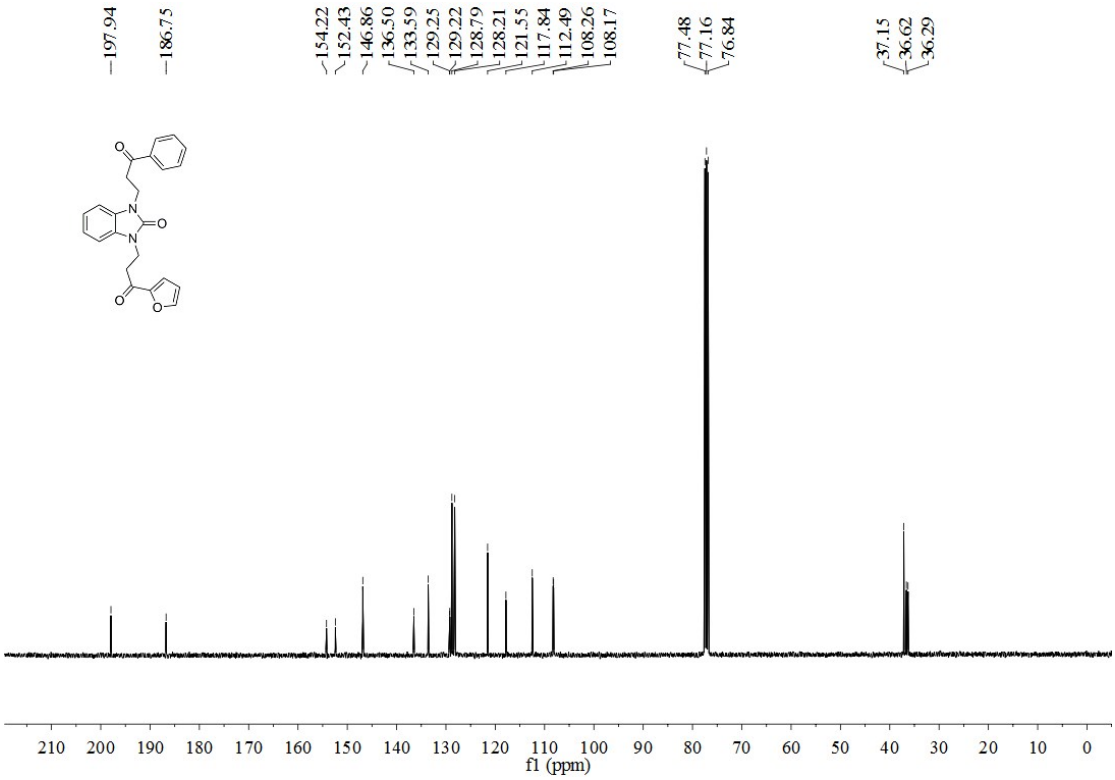
^{13}C NMR spectrum of ***3da***



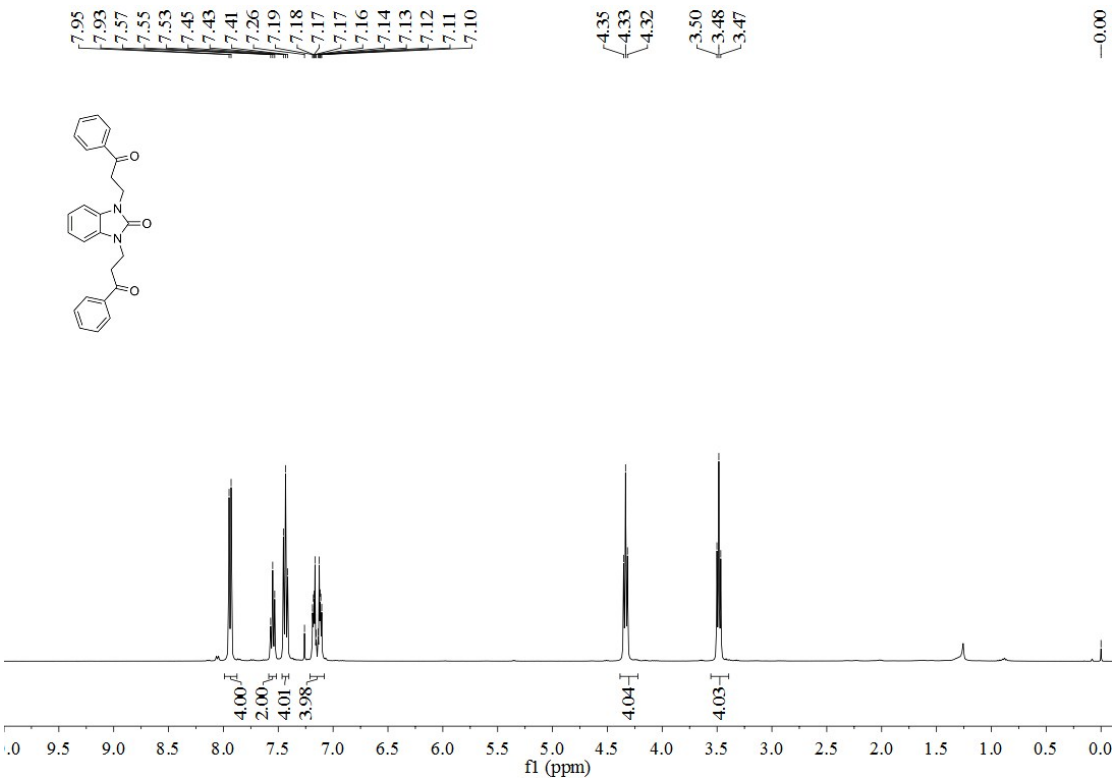
^{1}H NMR spectrum of ***3dl***



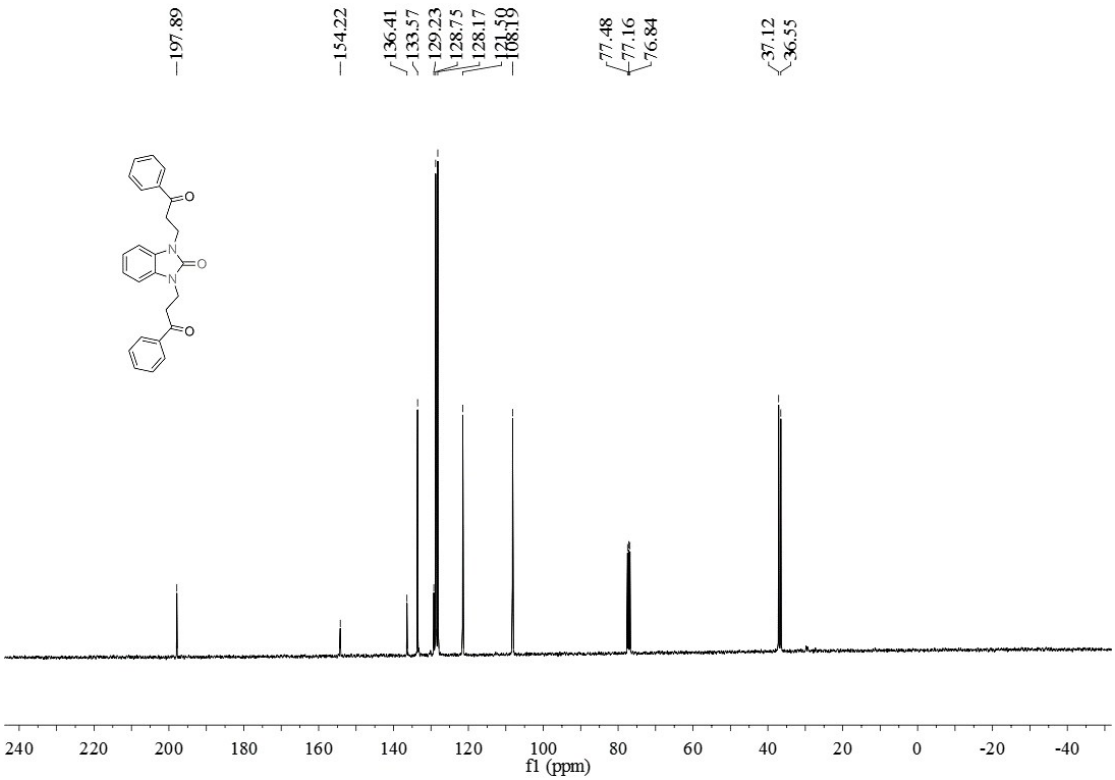
^{13}C NMR spectrum of ***3dl***



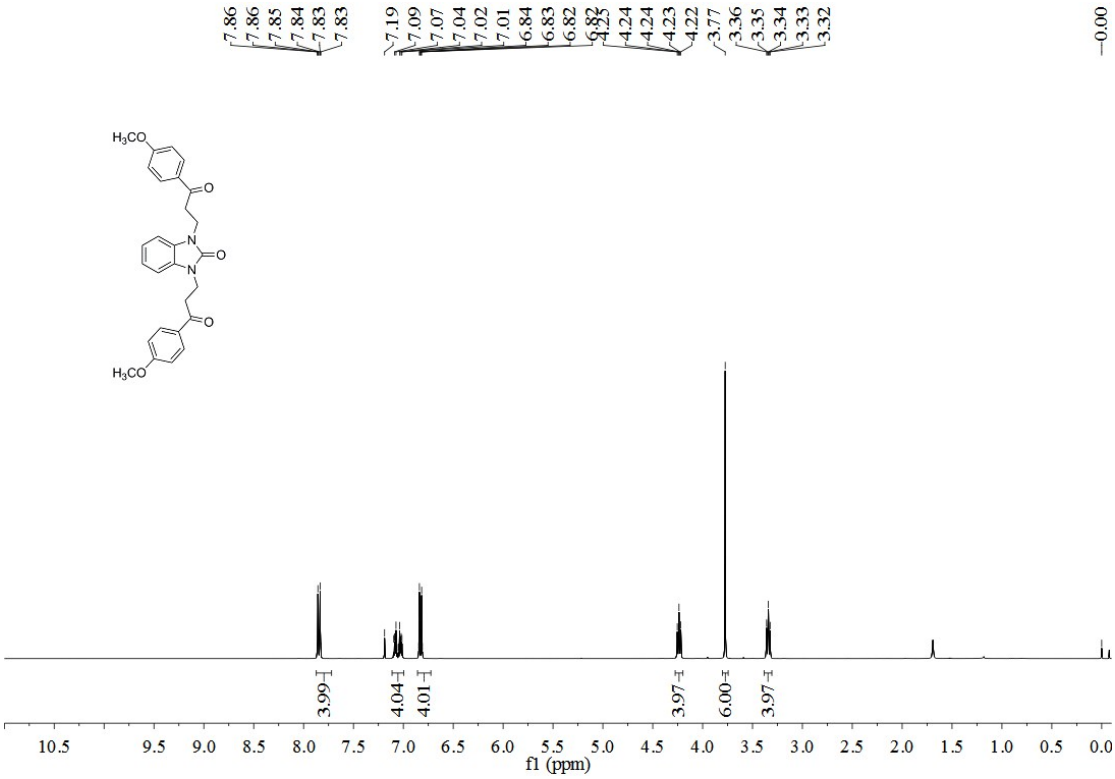
^{1}H NMR spectrum of ***5aa***



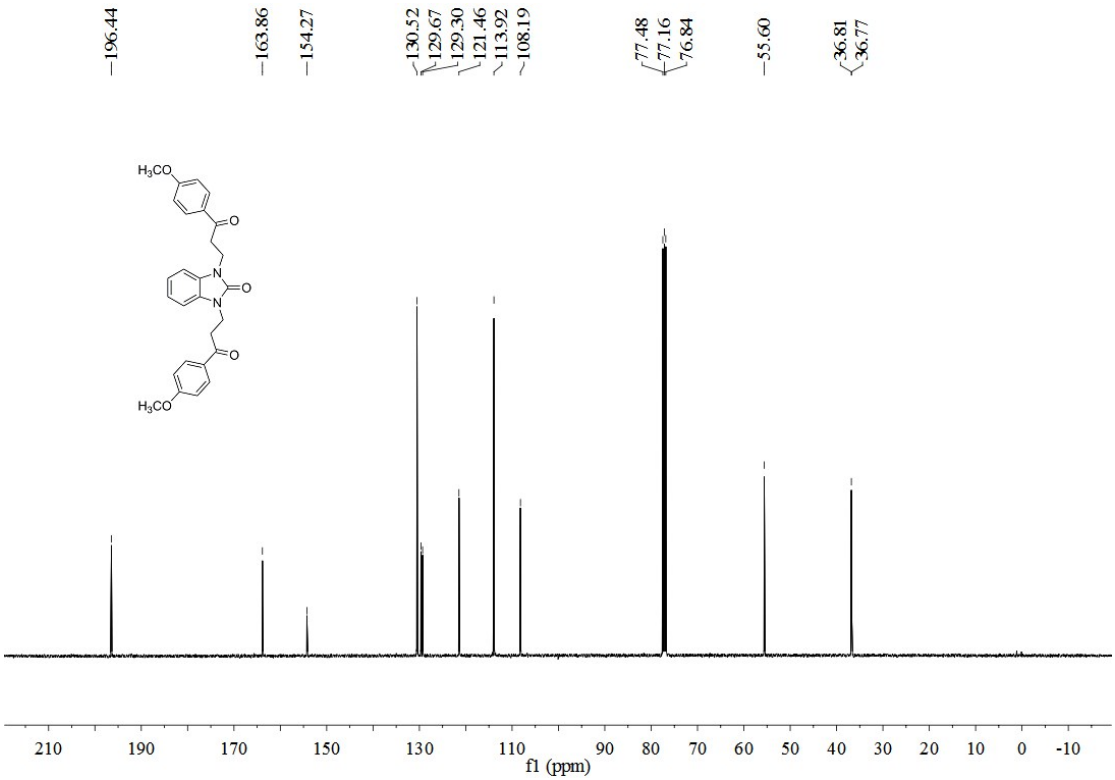
^{13}C NMR spectrum of ***5aa***



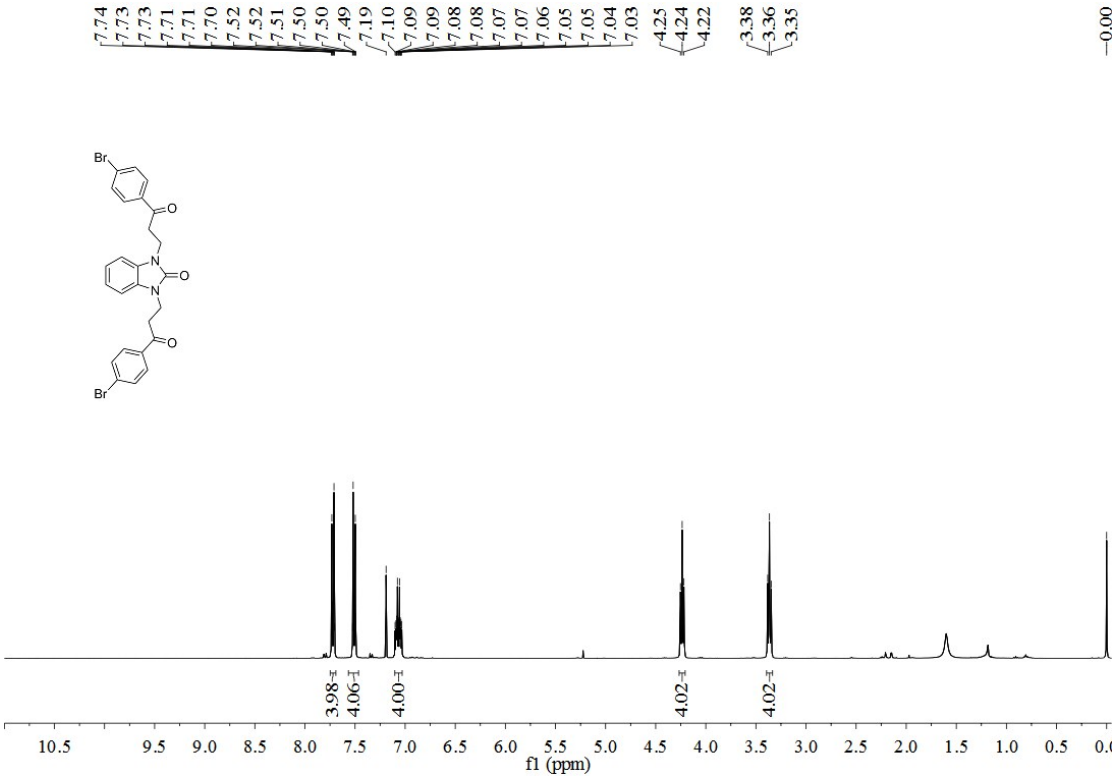
^{1}H NMR spectrum of***5ad***



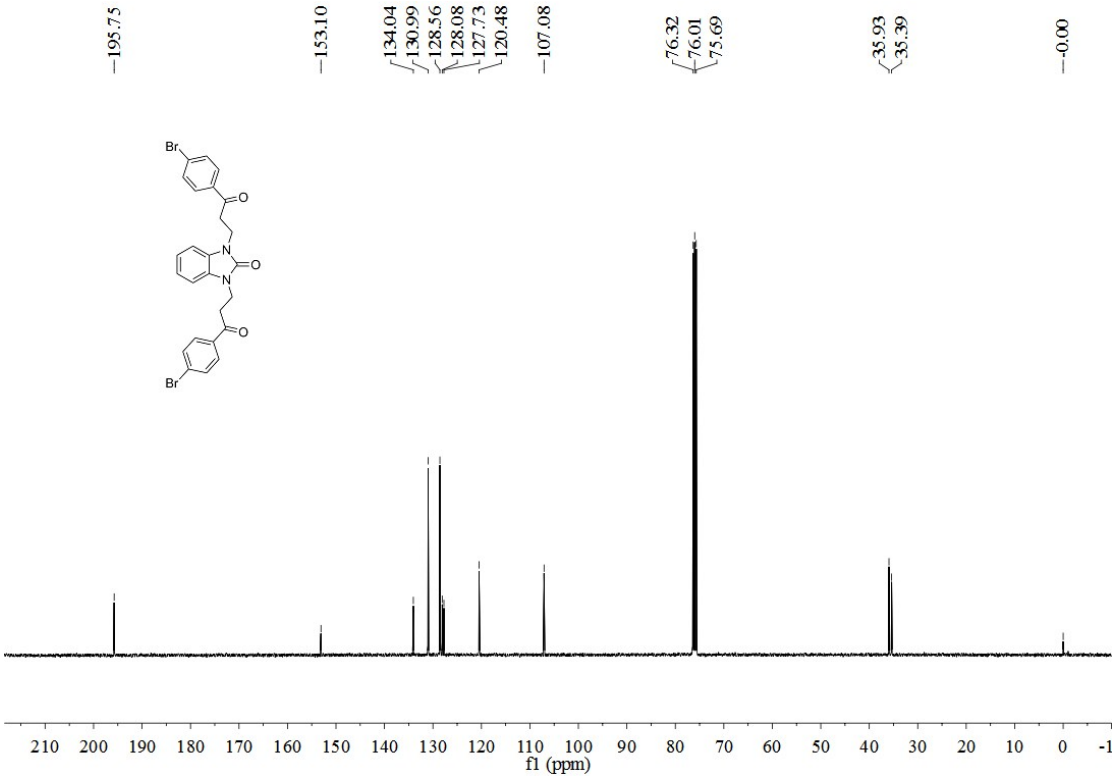
^{13}C NMR spectrum of ***5ad***



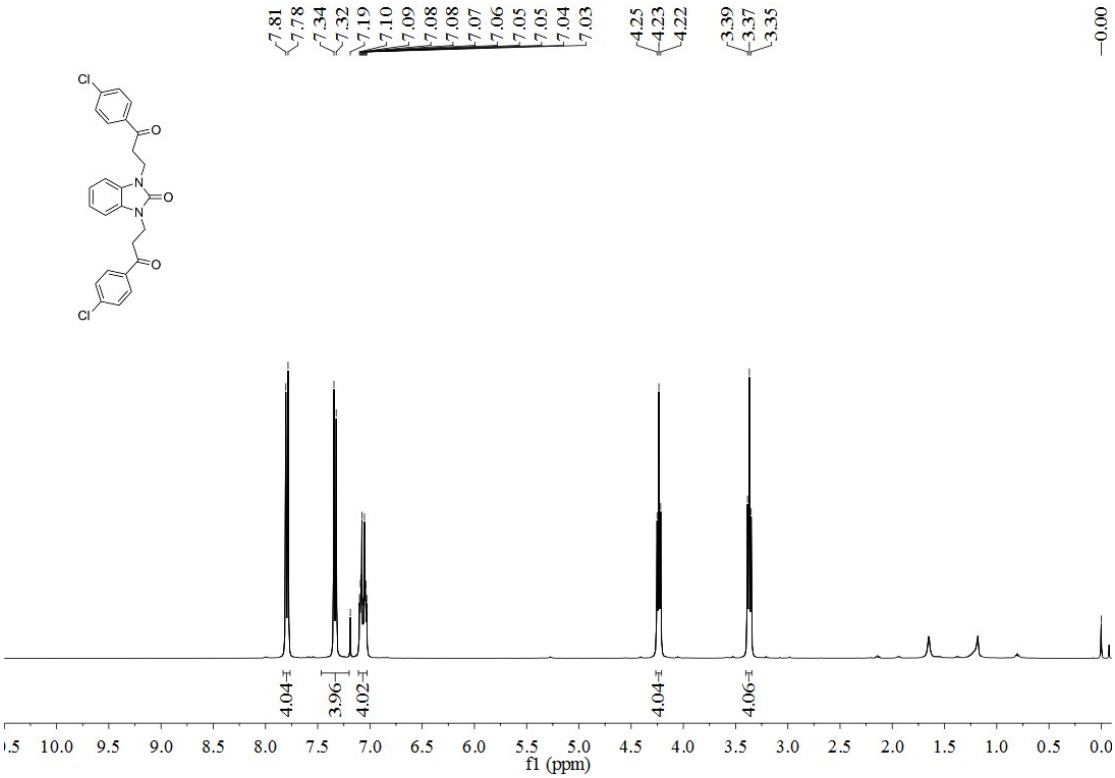
^{1}H NMR spectrum of ***5ae***



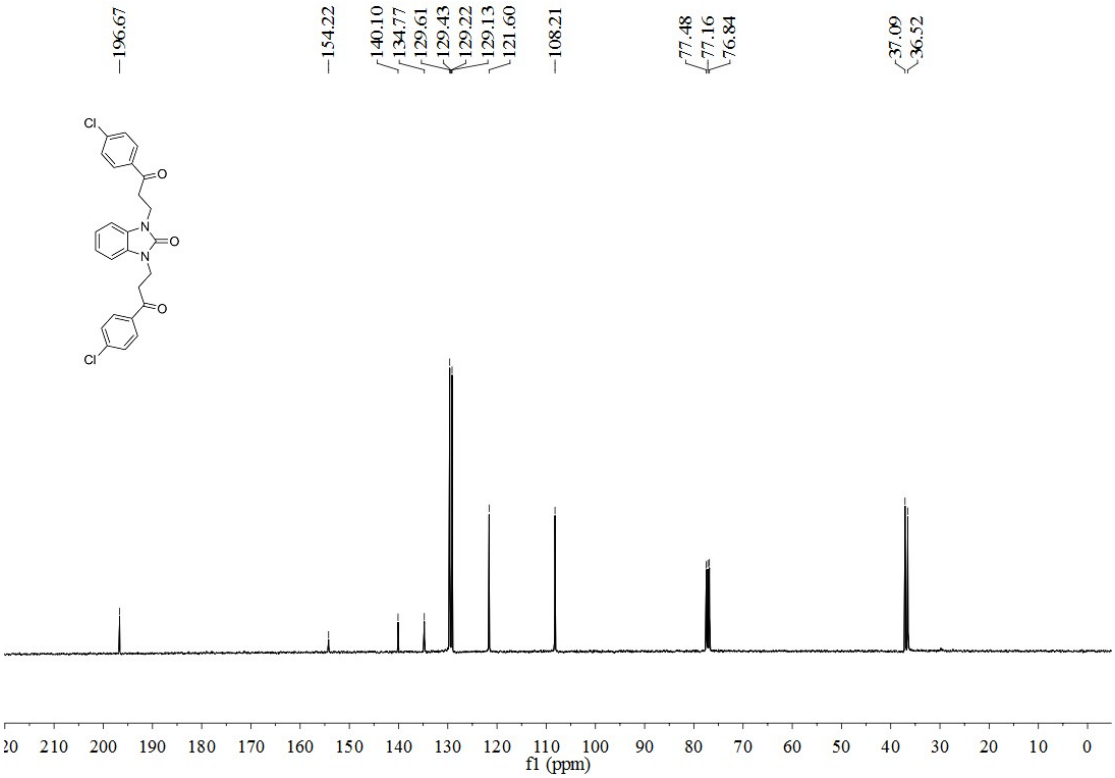
^{13}C NMR spectrum of ***5ae***



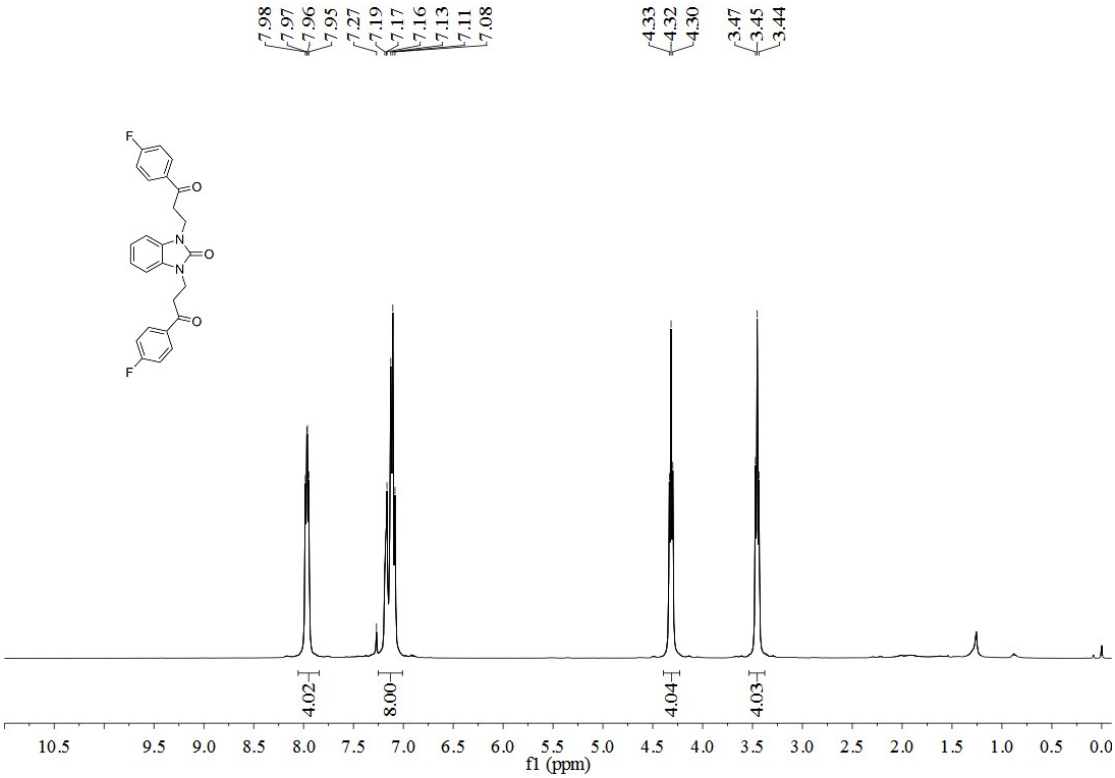
^{1}H NMR spectrum of ***5af***



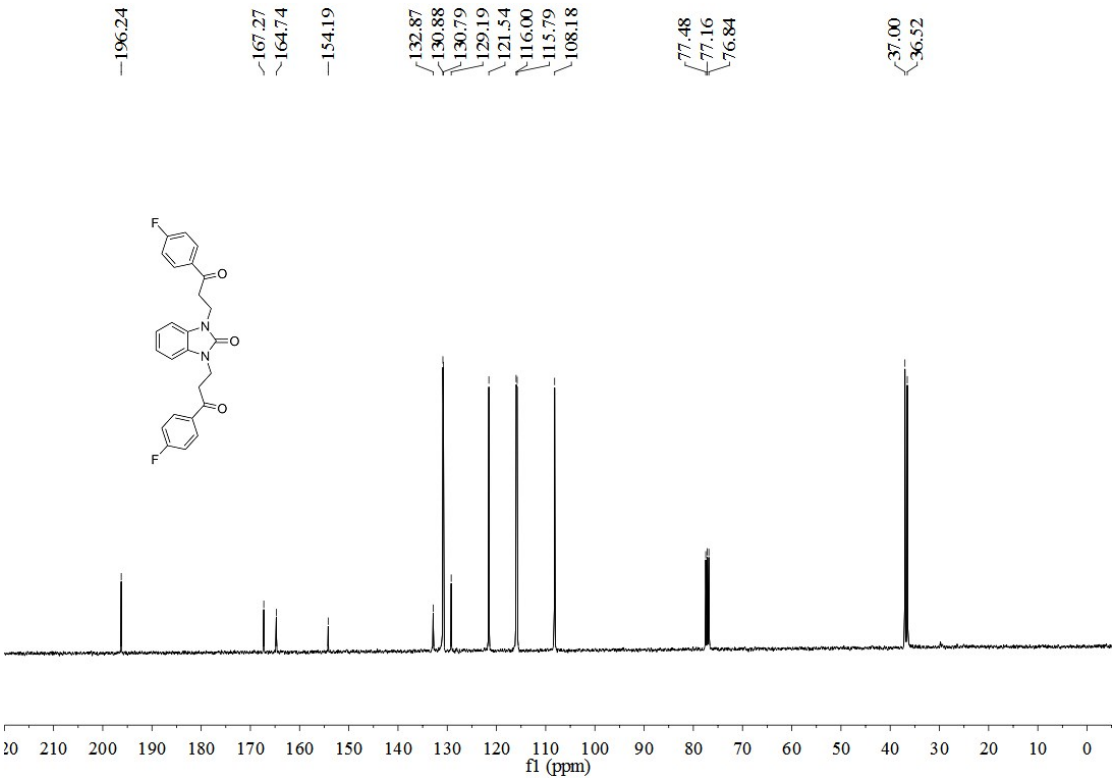
^{13}C NMR spectrum of ***5af***



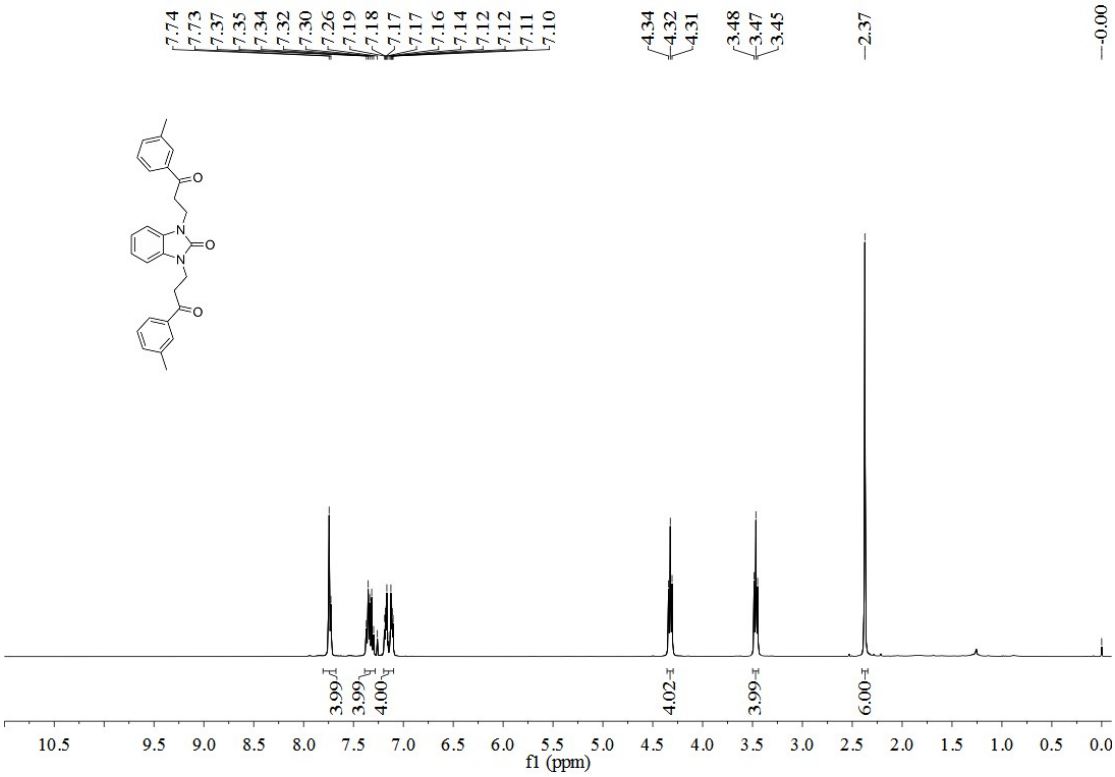
^{1}H NMR spectrum of ***5ag***



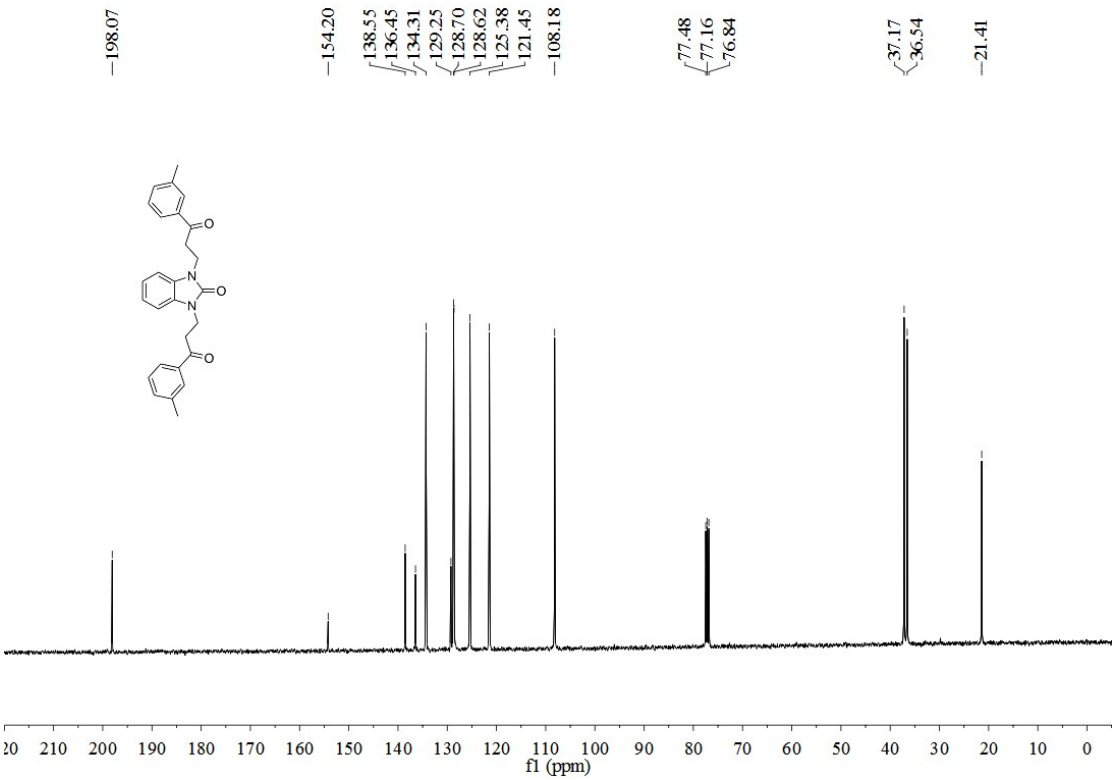
^{13}C NMR spectrum of ***5ag***



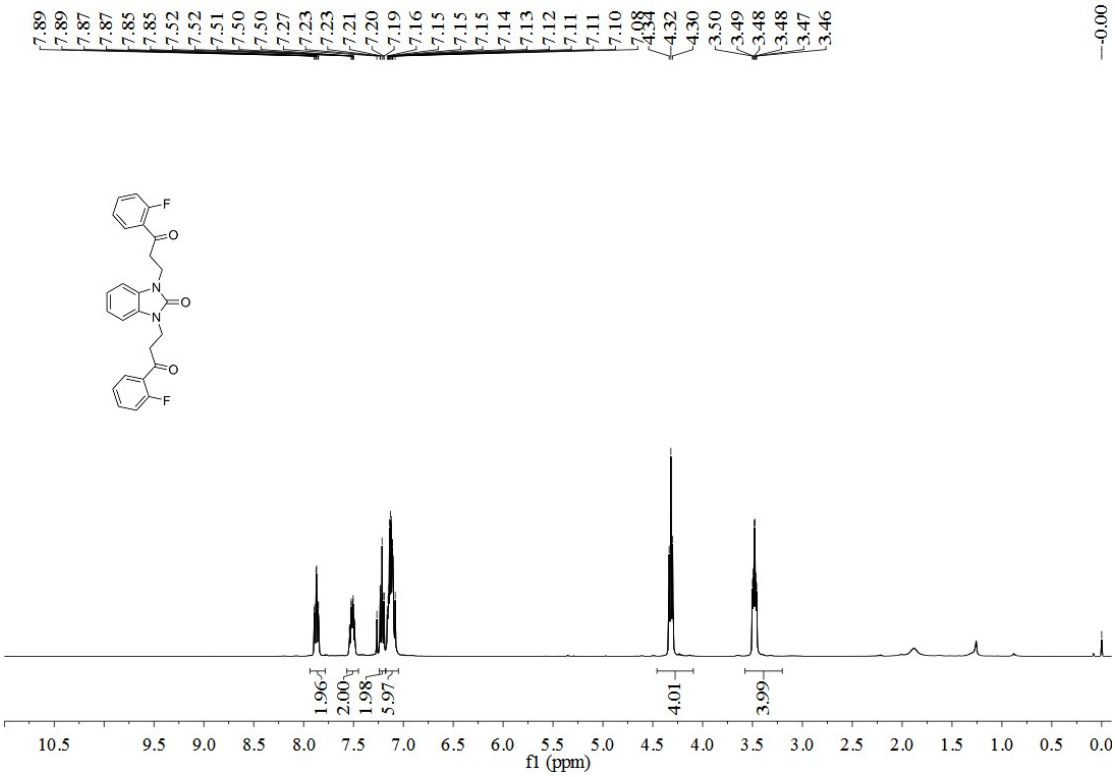
^{1}H NMR spectrum of ***5ai***



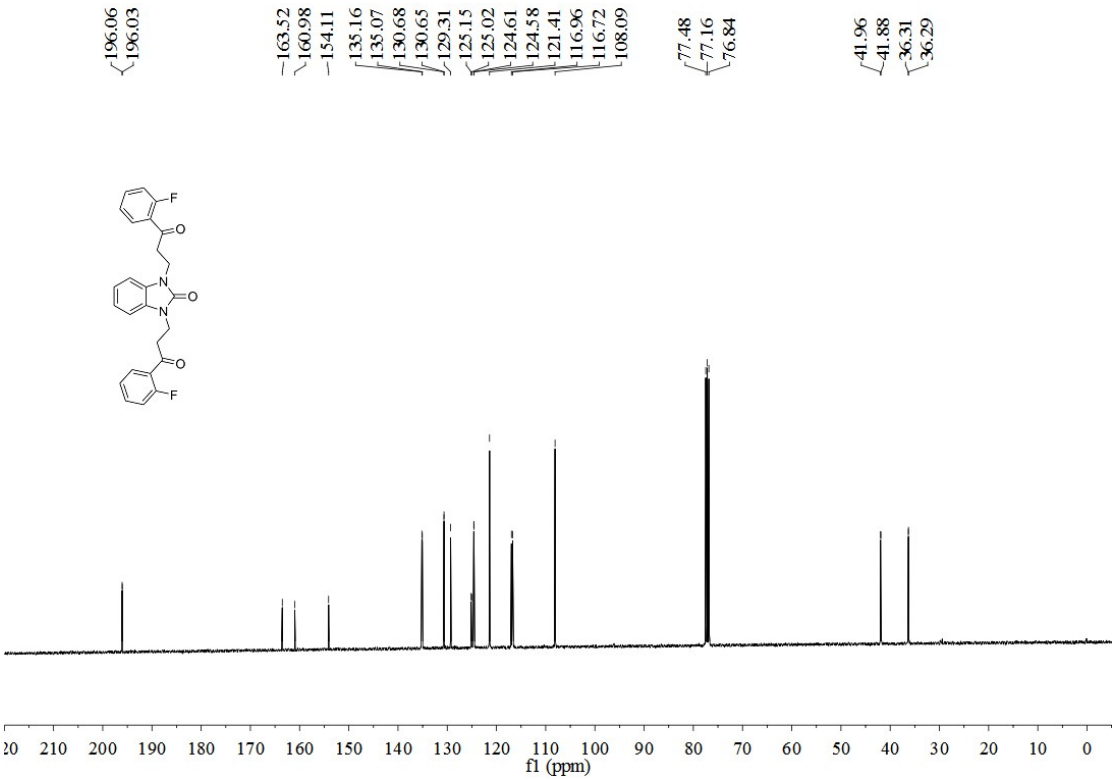
^{13}C NMR spectrum of ***5ai***



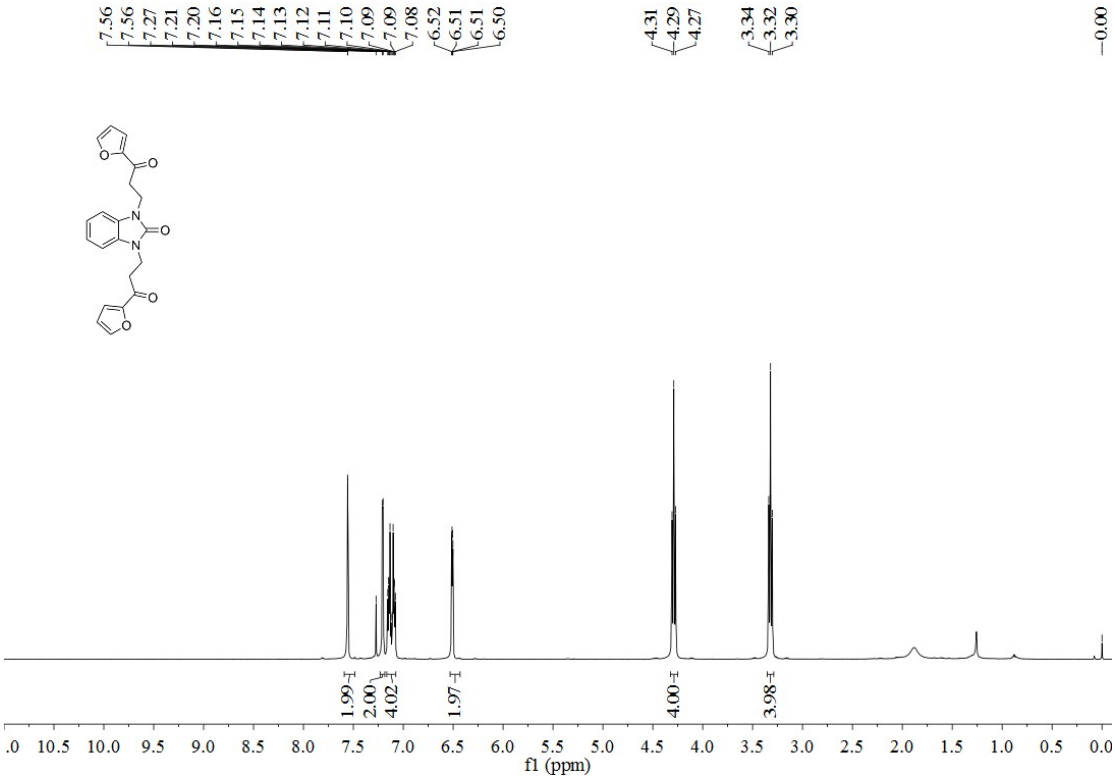
^{1}H NMR spectrum of ***5aj***



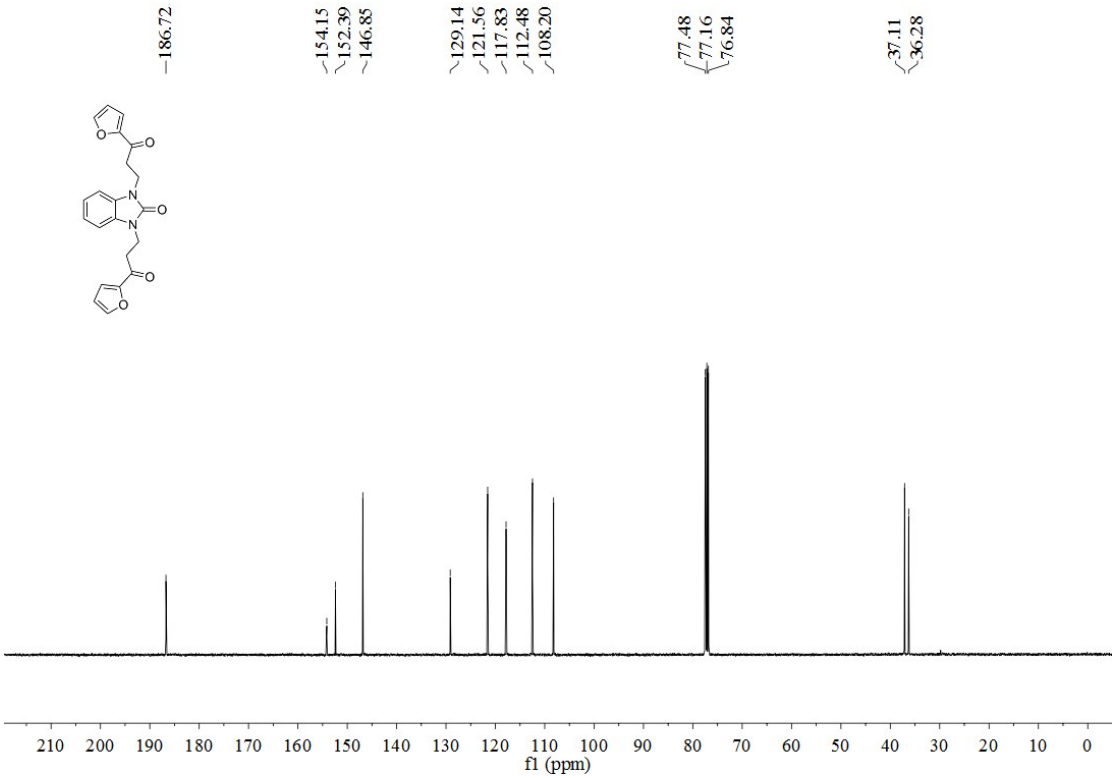
^{13}C NMR spectrum of***5aj***



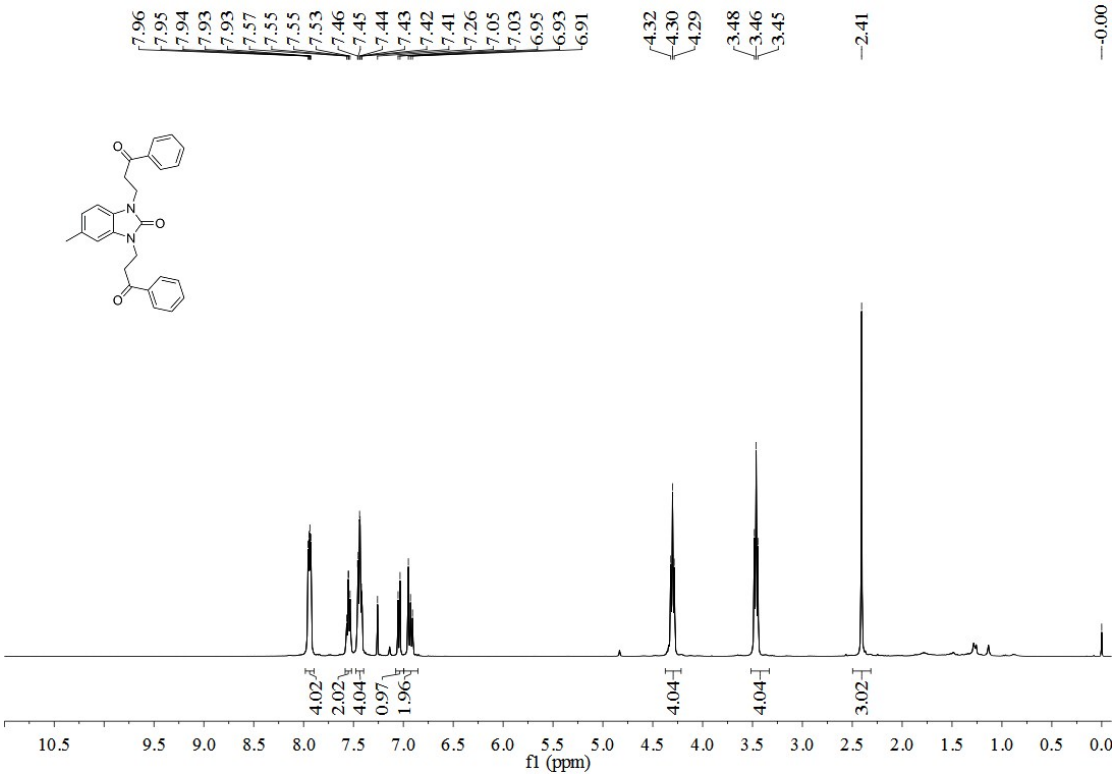
^{1}H NMR spectrum of ***5am***



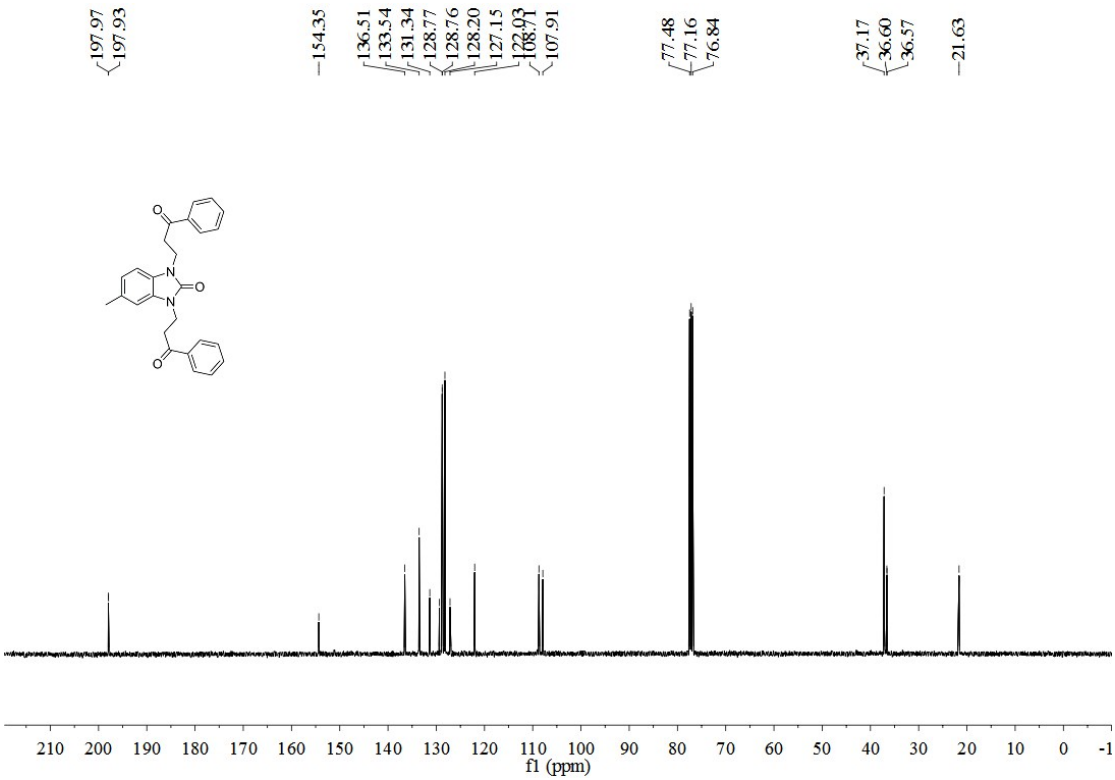
^{13}C NMR spectrum of **5am**



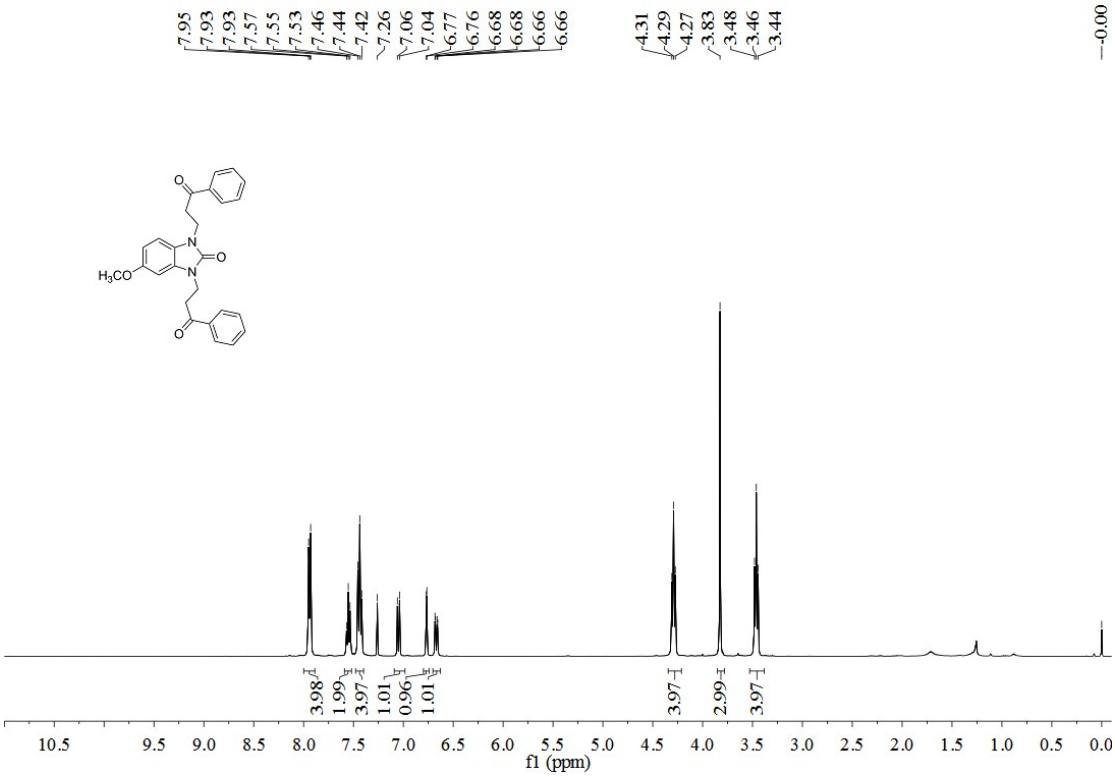
^{1}H NMR spectrum of ***5ba***



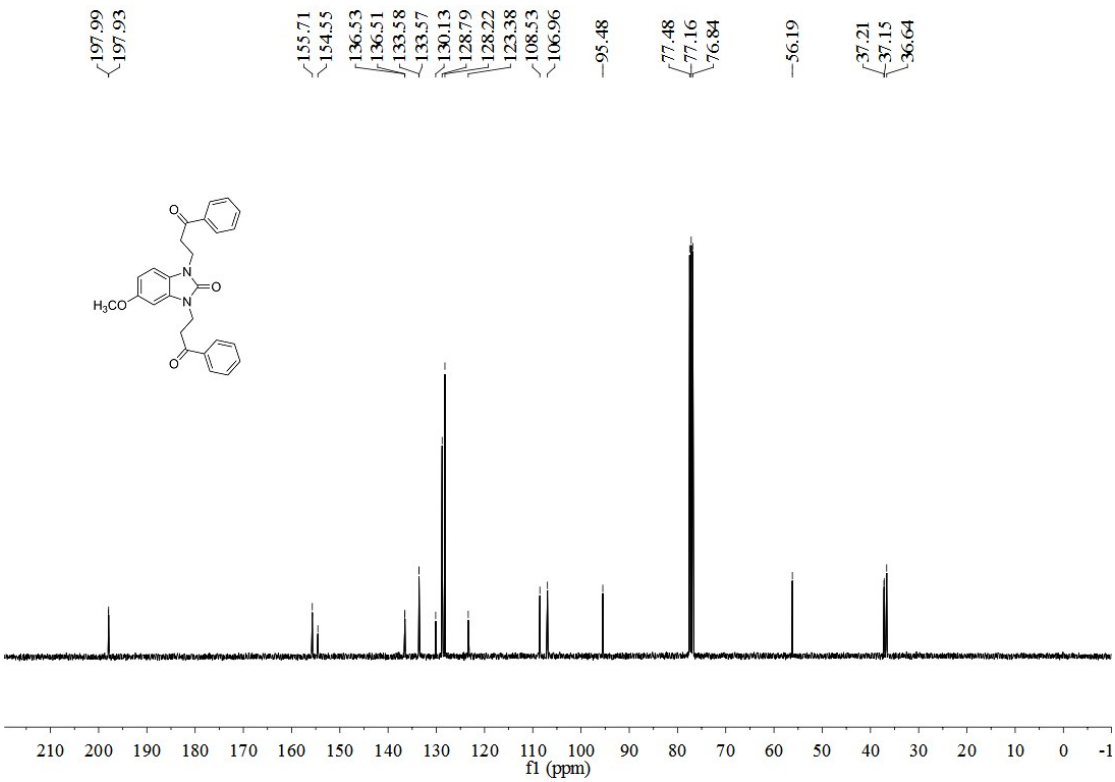
^{13}C NMR spectrum of ***5ba***



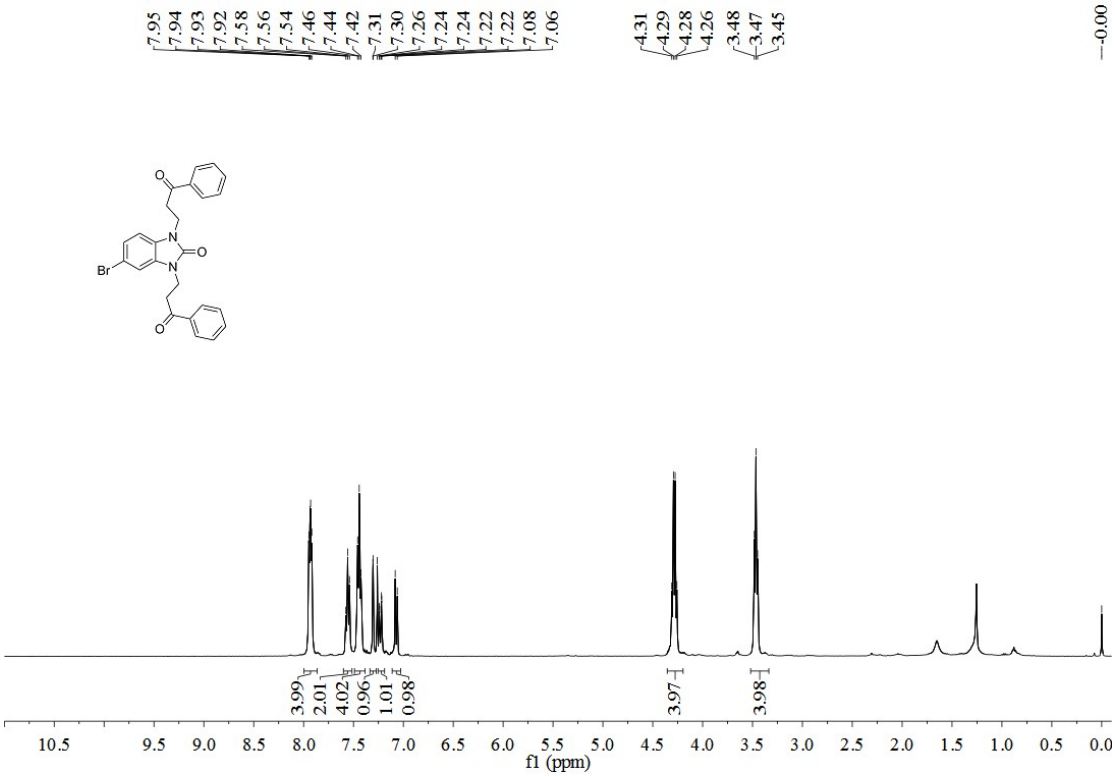
^{1}H NMR spectrum of ***5ca***



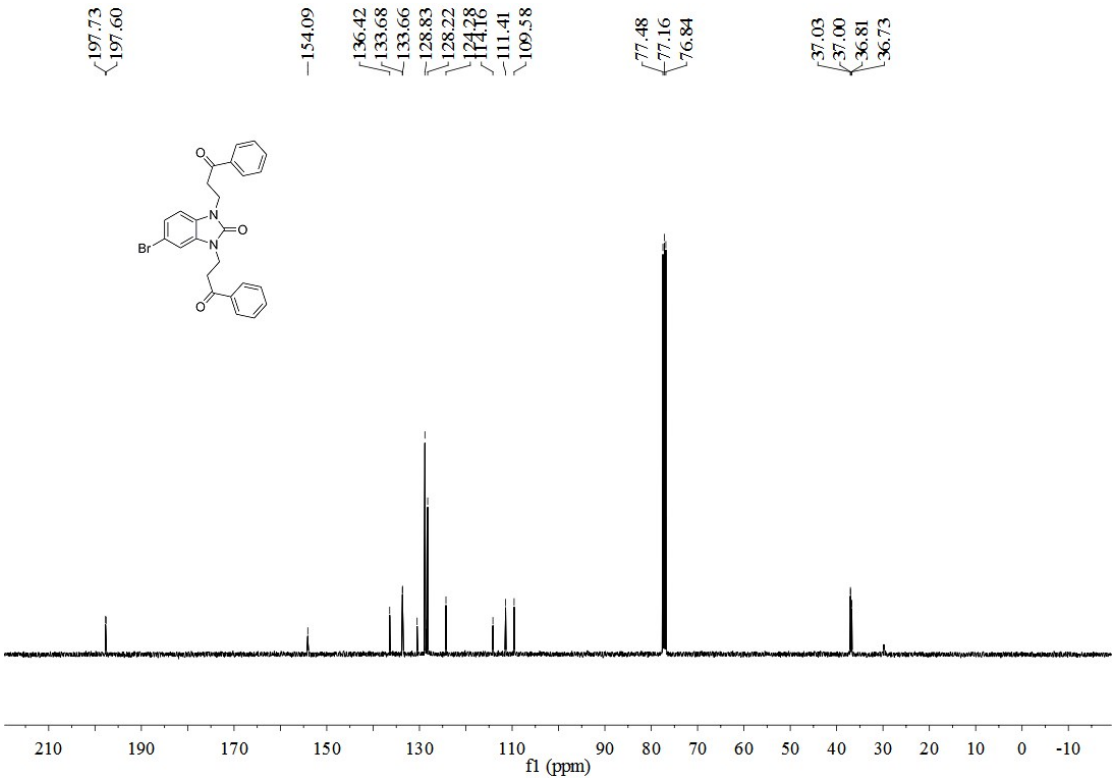
^{13}C NMR spectrum of ***5ca***



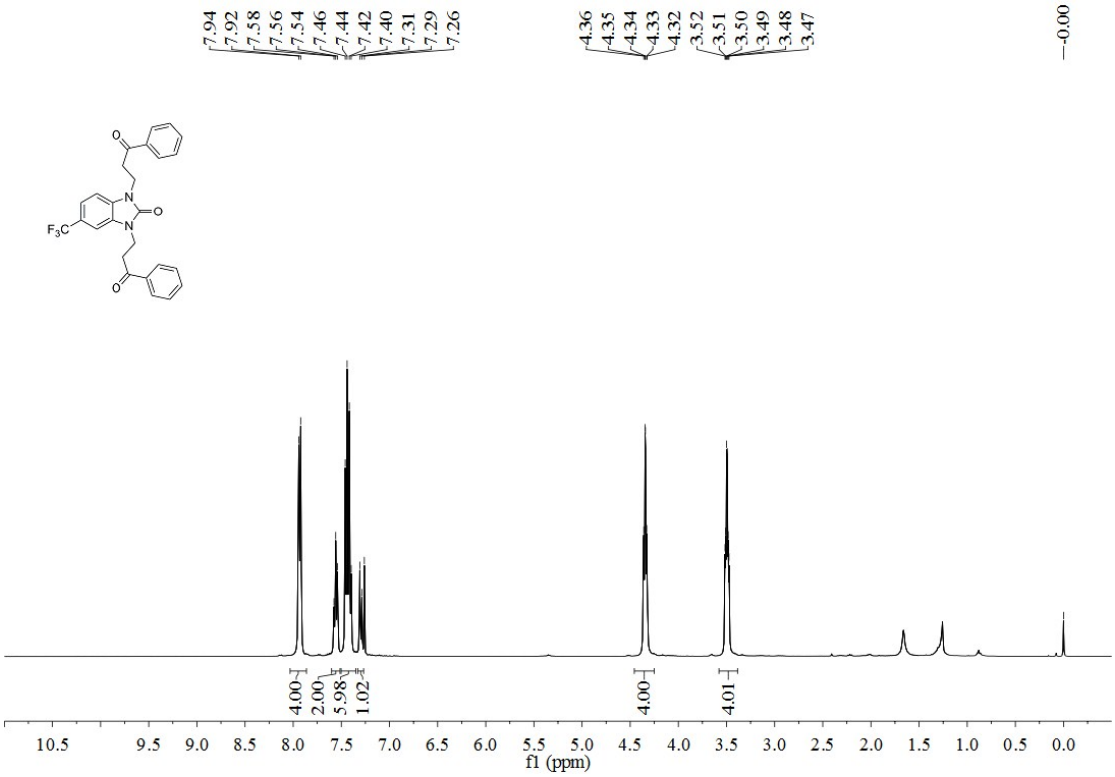
^{1}H NMR spectrum of ***5da***



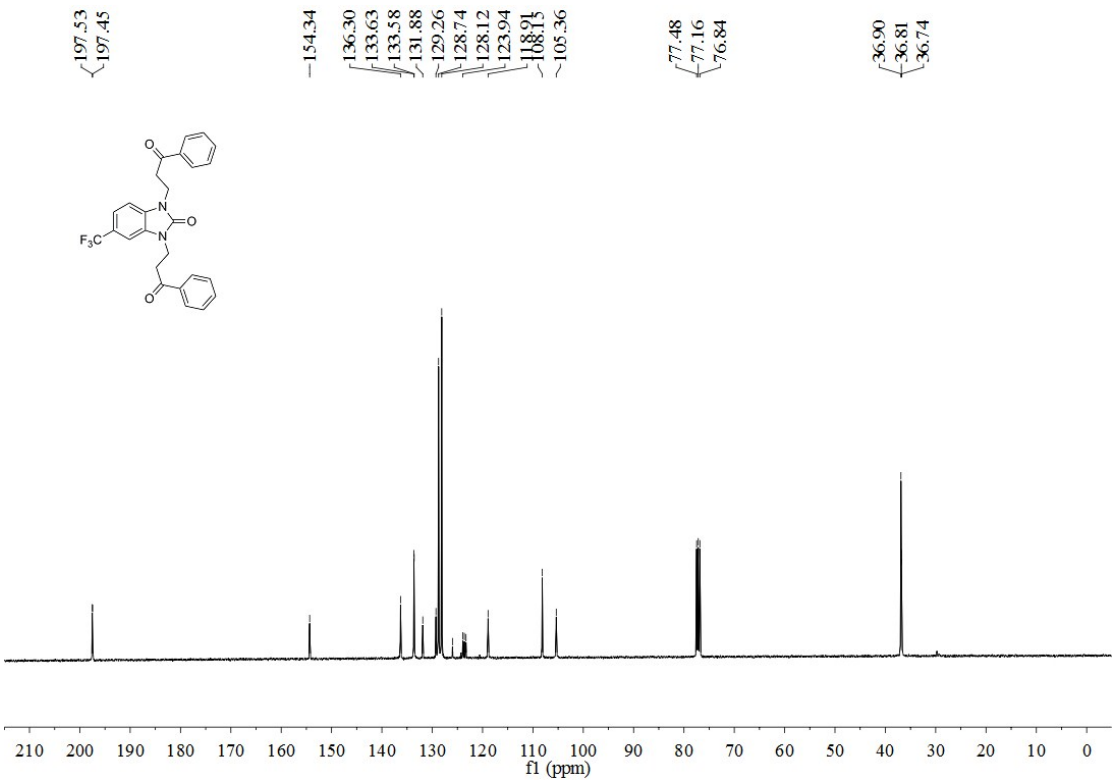
^{13}C NMR spectrum of ***5da***



^{1}H NMR spectrum of ***5ea***



^{13}C NMR spectrum of ***5ea***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197.45
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136.30
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131.88
129.26
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128.12
123.94
118.91
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105.36
77.48
77.16
76.84
36.90
36.81
36.74
F3C
O
N
O
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)