Silver-Catalyzed C2-Selective Direct Alkylation of Heteroarenes with Tertiary Cycloalkanols

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1. General Information

All reagents were used as received from commercial source (Aladdin) unless specified otherwise. Anhydrous THF was purchased from J&K and used without any purification. Silver catalysts were purchased from Aladdin or Alfa-Aesar. All the cycloalcohols were prepared as described in the literature. Melting points were determined on an X-4 binocular microscope and were uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance 400 spectrometer at the ambient temperature in $CDCl_3$. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity, integration, and coupling constant (Hz). Data for ^{13}C NMR are reported in terms of chemical shift (δ ppm). The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, m = multiplet. High-resolution mass spectra (HRMS) was carried out by LC/MSD TOF using a column of C18 (rapid resolution, 3.5 μm, 2.1 mm × 30 mm) at a flow of 0.40 mL/min. Column chromatography was performed using 200-300 mesh silica gel. Organic solutions were concentrated under reduced pressure on a BUCHI rotary evaporator.

2. Starting Materials

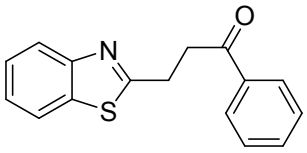
Tertiary cyclopropanols were prepared by the addition of Grignard reagent to the precursor according to the reported procedure.[1] Tertiary cyclobutanols and cyclopentanols were prepared by the addition of Grignard reagent to the corresponding cycloketones according to the reported procedure.[2]

3. General Procedure for the Preparation of the Target Compounds

To a solution of the substrate (0.2 mmol) and cycloalcohol (0.4 mmol) in 0.5 mL of 1,2-dichloroethane (DCE) and 0.5 mL of H_2O was successively added $K_2S_2O_8$ (0.8 mmol), $AgBF_4$ (0.04 mmol) at room temperature. The resulting heterogeneous mixture was charged three times with aron, and then stirred for 12 h. After completion of the reaction, the mixture was added 10 mL of H_2O, and then extracted with ethyl acetate (10 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was puried by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the target compound.

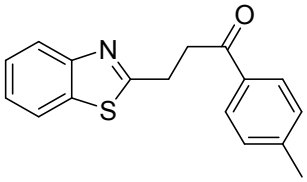
4. Analytical Data of the Target Compounds

3-(Benzo[*d*]thiazol-2-yl)-1-phenylpropan-1-one (3a)



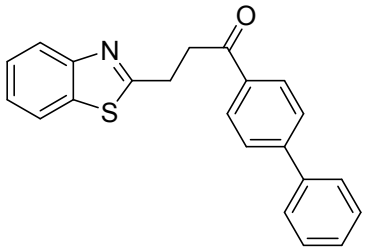
Colorless oil, 40.6 mg (76%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.01-8.03 (m, 2H), 7.95 (d, J = 8.0 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.56-7.60 (m, 1H), 7.42-7.49 (m, 3H), 7.33-7.36 (m, 1H), 3.66 (t, J = 6.8 Hz, 2H), 3.57 (t, J = 6.8 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 198.0, 170.6, 153.2, 136.6, 135.3, 133.4, 128.8, 128.2, 126.0, 124.9, 122.6, 121.6, 37.5, 28.3; HRMS (ESI): Exact mass calcd for $C_{16}H_{14}NOS$ $[M+H]^+$ 268.0796, found 268.0786.

3-(Benzo[*d*]thiazol-2-yl)-1-p-tolylpropan-1-one (3b)



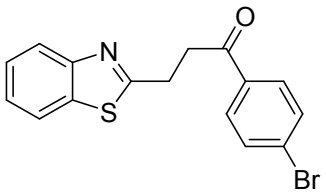
Colorless oil, 40.5 mg (72%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95 (ddd, J = 8.0 Hz, 1.2 Hz, 0.4 Hz, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.83 (ddd, J = 8.0 Hz, 1.2 Hz, 0.4 Hz, 1H), 7.42-7.46 (m, 1H), 7.33-7.37 (m, 1H), 7.27 (d, J = 7.6 Hz, 2H), 3.61-3.65 (m, 2H), 3.54-3.58 (m, 2H), 2.42 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.6, 170.8, 153.3, 144.3, 135.4, 134.1, 129.4, 128.3, 126.0, 124.9, 122.6, 121.6, 37.5, 28.4, 21.8; HRMS (ESI): Exact mass calcd for $C_{17}H_{16}NOS$ $[M+H]^+$ 282.0953, found 282.0943.

3-(Benzo[*d*]thiazol-2-yl)-1-biphenylpropan-1-one (3c)



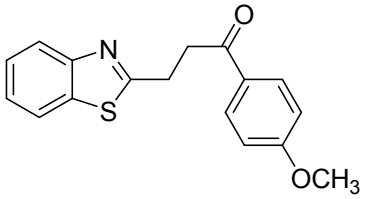
Colorless oil, 46.0 mg (67%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.10 (d, J = 8.8 Hz, 2H), 7.96 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.70 (d, J = 8.8 Hz, 2H), 7.62-7.64 (m, 2H), 7.33-7.50 (m, 5H), 3.69 (t, J = 6.8 Hz, 2H), 3.60 (t, J = 6.4 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.6, 170.6, 153.2, 146.1, 139.9, 135.34, 135.27, 129.1, 128.8, 128.4, 127.39, 127.36, 126.0, 124.9, 122.6, 121.6, 37.6, 28.4; HRMS (ESI): Exact mass calcd for $C_{22}H_{18}NOS$ $[M+H]^+$ 344.1109, found 344.1096.

3-(Benzo[*d*]thiazol-2-yl)-1-(4-bromophenyl)propan-1-one (3d)



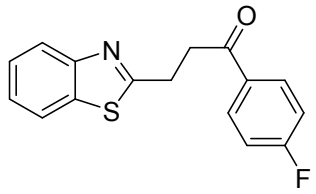
Colorless oil, 44.3 mg (64%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95 (d, J = 8.4 Hz, 1H), 7.88 (d, J = 8.8 Hz, 2H), 7.83 (d, J = 8.0 Hz, 1H), 7.61 (d, J = 8.4 Hz, 2H), 7.42-7.47 (m, 1H), 7.33-7.37 (m, 1H), 3.61-3.65 (m, 2H), 3.54-3.58 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.0, 170.4, 153.1, 135.29, 135.25, 132.1, 131.9, 131.7, 129.7, 128.6, 126.1, 125.0, 122.6, 121.6, 37.4, 28.1; HRMS (ESI): Exact mass calcd for $C_{16}H_{13}BrNOS$ $[M+H]^+$ 345.9901, found 345.9892.

3-(Benzo[*d*]thiazol-2-yl)-1-(4-methoxyphenyl)propan-1-one (3e)



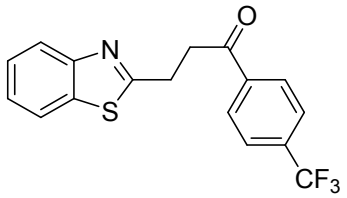
Colorless oil, 39.8 mg (67%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.00 (d, J = 8.8 Hz, 2H), 7.95 (ddd, J = 8.4 Hz, 1.2 Hz, 0.8 Hz, 1H), 7.83 (ddd, J = 8.0 Hz, 1.2 Hz, 0.8 Hz, 1H), 7.42-7.46 (m, 1H), 7.33-7.37 (m, 1H), 6.94 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 3.54-3.63 (m, 4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 196.5, 170.8, 163.8, 153.3, 135.4, 130.5, 129.7, 126.0, 124.9, 122.6, 121.6, 113.9, 55.6, 37.2, 28.5; HRMS (ESI): Exact mass calcd for $C_{17}H_{16}NO_2S$ $[M+H]^+$ 298.0902, found 298.0892.

3-(Benzo[*d*]thiazol-2-yl)-1-(4-fluorophenyl)propan-1-one (3f)



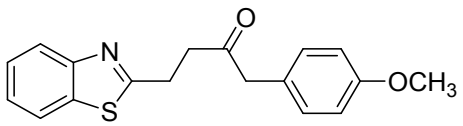
Colorless oil, 33.1 mg (58%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.03-8.06 (m, 2H), 7.94 (d, J = 8.0 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.42-7.47 (m, 1H), 7.33-7.37 (m, 1H), 7.14 (t, J = 8.8 Hz, 2H), 3.62-3.66 (m, 2H), 3.54-3.58 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 196.4, 170.4, 166.0 (d, J = 253.5 Hz), 153.2, 135.3, 133.0 (d, J = 3.0 Hz), 130.9 (d, J = 9.2 Hz), 126.0, 124.9, 122.6, 121.6, 115.9 (d, J = 21.8 Hz), 37.4, 28.2; HRMS (ESI): Exact mass calcd for $C_{16}H_{13}FNOS$ $[M+H]^+$ 286.0702, found 286.0692.

3-(Benzo[*d*]thiazol-2-yl)-1-(4-(trifluoromethyl)phenyl)propan-1-one (3g)



Colorless oil, 31.5 mg (47%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.13 (d, J = 8.0 Hz, 2H), 7.94 (ddd, J = 8.0 Hz, 1.2 Hz, 0.4 Hz, 1H), 7.84 (ddd, J = 8.0 Hz, 1.2 Hz, 0.4 Hz, 1H), 7.75 (d, J = 8.0 Hz, 2H), 7.43-7.47 (m, 1H), 7.34-7.38 (m, 1H), 3.57-3.61 (m, 2H), 3.67-3.71 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.1, 170.0, 153.2, 139.3, 135.3, 134.7 (q, J = 32.6 Hz), 128.6, 126.1, 125.9 (q, J = 3.7 Hz), 125.0, 122.6, 123.6 (q, J = 270.8 Hz), 121.6, 37.6, 28.1; HRMS (ESI): Exact mass calcd for $C_{17}H_{13}F_3NOS$ $[M+H]^+$ 336.0670, found 336.0662.

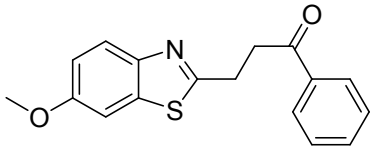
4-(Benzo[*d*]thiazol-2-yl)-1-(4-methoxyphenyl)butan-2-one (3h)



Colorless oil, 41.1 mg (66%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.92 (d, J = 8.0 Hz, 1H), 7.81 (ddd, J = 8.0 Hz, 1.2 Hz, 0.4 Hz, 1H), 7.42-7.46 (m, 1H), 7.32-7.36 (m, 1H),

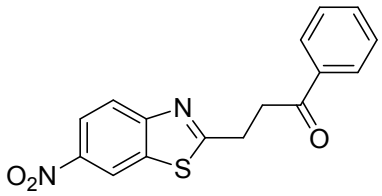
7.12 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 8.8 Hz, 2H), 3.78 (s, 3H), 3.71 (s, 2H), 3.35 (t, J = 7.2 Hz, 2H), 3.09 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 206.7, 170.3, 158.8, 153.1, 135.3, 130.5, 126.0, 125.9, 124.9, 122.6, 121.6, 124.3, 55.3, 49.4, 40.3, 28.1; HRMS (ESI): Exact mass calcd for $C_{18}H_{18}NO_2S$ $[M+H]^+$ 312.1058, found 312.1046.

3-(6-Methoxybenzo[*d*]thiazol-2-yl)-1-phenylpropan-1-one (3i)



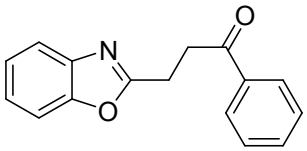
White solid, 48.8 mg (82%), mp 56−58 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.01-8.04 (m, 2H), 7.82 (d, J = 9.2 Hz, 1H), 7.57-7.60 (m, 1H), 7.45-7.50 (m, 2H), 7.29 (d, J = 2.4 Hz, 1H), 7.04 (dd, J = 9.2 Hz, 2.4 Hz, 1H), 3.86 (s, 3H), 3.62-3.66 (m, 2H), 3.50-3.54 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 198.0, 167.9, 157.5, 147.8, 136.6, 133.4, 128.7, 128.2, 123.0, 116.2, 104.3, 55.9, 37.6, 28.2; HRMS (ESI): Exact mass calcd for $C_{17}H_{16}NO_2S$ $[M+H]^+$ 298.0902, found 298.0889.

3-(6-Nitrobenzo[*d*]thiazol-2-yl)-1-phenylpropan-1-one (3j)



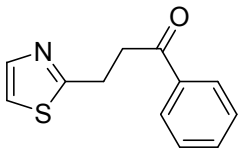
Yellow solid, 21.2 mg (34%), mp 111−113 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.78 (d, J = 2.4 Hz, 1H), 8.32 (dd, J = 8.8 Hz, 2.4 Hz, 1H), 8.00-8.03 (m, 3H), 7.58-7.63 (m, 1H), 7.48-7.51 (m, 2H), 3.69-3.73 (m, 2H), 3.62-3.65 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.5, 177.0, 157.0, 144.9, 136.4, 135.8, 133.6, 128.8, 128.2, 122.9, 121.6, 118.2, 37.0, 29.8; HRMS (ESI): Exact mass calcd for $C_{16}H_{13}N_2O_3S$ $[M+H]^+$ 313.0647, found 313.0634.

3-(Benzo[*d*]oxazol-2-yl)-1-phenylpropan-1-one (3k)



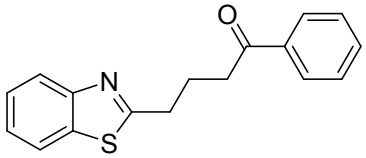
Colorless oil, 26.1 mg (52%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.02-8.06 (m, 2H), 7.64-7.68 (m, 1H), 7.57-7.61 (m, 1H), 7.47-7.51 (m, 3H), 7.28-7.32 (m, 2H), 3,67 (t, J = 7.6 Hz, 2H), 3,40 (t, J = 8.0 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.6, 166.3, 150.9, 141.4, 136.5, 133.5, 128.8, 128.2, 124.7, 124.2, 119.6, 110.5, 35.1, 22.9; HRMS (ESI): Exact mass calcd for $C_{16}H_{14}NO_2$ $[M+H]^+$ 252.1025, found 252.1014.

1-Phenyl-3-(thiazol-2-yl)propan-1-one (3l)



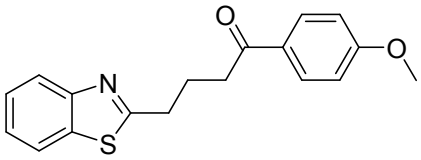
Colorless oil, 20.0 mg (46%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.99-8.02 (m, 2H), 6.80 (d, J = 7.6 Hz, 1H), 7.55-7.60 (m, 1H), 7.45-7.49 (m, 2H), 7.20 (d, J = 3.2 Hz, 1H), 3.55-3.59 (m, 2H), 3.47-3.50 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 198.2, 169.6, 142.3, 136.6, 133.4, 128.7, 128.2, 118.5, 37.9, 27.3; HRMS (ESI): Exact mass calcd for $C_{12}H_{12}NOS$ $[M+H]^+$ 218.0640, found 218.0631.

4-(Benzo[*d*]thiazol-2-yl)-1-phenylbutan-1-one (3m)



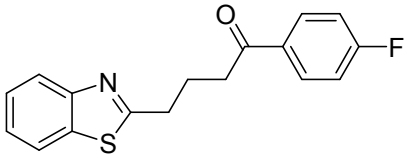
White solid, 38.3 mg (68%), mp 68−70 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 7.94-7.98 (m, 3H), 7.85 (d, J = 8.0 Hz, 1H), 7.55 (t, J = 7.6 Hz, 1H), 7.43-7.48 (m, 3H), 7.34-7.38 (m, 1H), 3.25 (t, J = 7.2 Hz, 2H), 3.14 (t, J = 7.2 Hz, 2H), 2.32-2.39 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.4, 171.4, 153.3, 136.9, 135.2, 133.2, 128.7, 128.1, 126.0, 124.9, 122.7, 121.6, 37.4, 33.5, 23.8; HRMS (ESI): Exact mass calcd for $C_{17}H_{16}NOS$ $[M+H]^+$ 282.0953, found 282.0942.

4-(Benzo[*d*]thiazol-2-yl)-1-(4-methoxyphenyl)butan-1-one (3n)



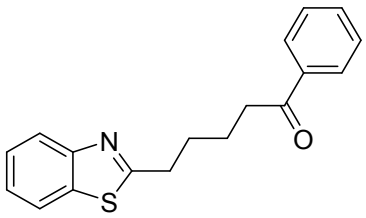
White solid, 43.6 mg (70%), mp 65−67 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 7.97 (d, J = 8.0 Hz, 1H), 7.93 (d, J = 8.8 Hz, 2H), 7.84 (d, J = 8.0 Hz, 1H), 7.43-7.47 (m, 1H), 7.33-7.37 (m, 1H), 6.91 (d, J = 8.8 Hz, 2H), 3.86 (s, 3H), 3.24 (t, J = 7.2 Hz, 2H), 3.08 (t, J = 7.2 Hz, 2H), 2.30-2.37 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.9, 171.5, 163.5, 153.2, 135.2, 130.4, 129.9, 126.0, 124.8, 122.6, 121.6, 113.8, 55.5, 37.1, 33.6, 24.0; HRMS (ESI): Exact mass calcd for $C_{18}H_{18}NO_2S$ $[M+H]^+$ 312.1058, found 312.1046.

4-(Benzo[*d*]thiazol-2-yl)-1-(4-fluorophenyl)butan-1-one (3o)



White solid, 37.1 mg (62%), mp 74−76 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 7.96-8.00 (m, 3H), 7.84-7.86 (m, 1H), 7.44-7.49 (m, 1H), 7.34-7.38 (m, 1H), 7.09-7.15 (m, 2H), 3.25 (t, J = 7.2 Hz, 2H), 3.11 (t, J = 7.2 Hz, 2H), 2.31-2.39 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 197.8, 171.2, 165.8 (d, J = 253.1 Hz), 153.3, 135.3, 133.3 (d, J = 3.0 Hz), 130.8 (d, J = 9.2 Hz), 126.1, 124.9, 122.7, 121.7, 115.8 (d, J = 21.7 Hz), 37.3, 33.5, 23.8; HRMS (ESI): Exact mass calcd for $C_{17}H_{15}FNOS$ $[M+H]^+$ 300.0858, found 300.0848.

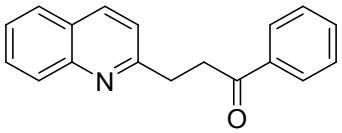
5-(benzo[*d*]thiazol-2-yl)-1-phenylpentan-1-one (3p)



Colorless oil, 25.5 mg (43%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.94-7.97 (m, 3H), 7.84 (dd, J = 8.0 Hz, 0.8 Hz, 1H), 7.53-7.58 (m, 1H), 7.43-7.47 (m, 3H), 7.33-7.37 (m, 1H),

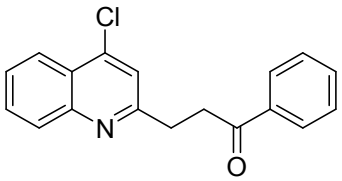
3.19 (t, J = 7.2 Hz, 2H), 3.06 (t, J = 7.2 Hz, 2H), 1.87-2.04 (m, 4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.9, 171.8, 153.3, 137.0, 136.2, 133.1, 128.7, 128.1, 126.0, 124.8, 122.6, 121.6, 38.2, 34.2, 29.3, 23.7; HRMS (ESI): Exact mass calcd for $C_{18}H_{18}NOS$ $[M+H]^+$ 296.1109, found 296.1099.

1-Phenyl-3-(quinolin-2-yl)propan-1-one (5a)



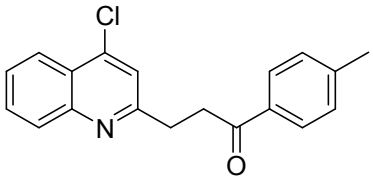
Colorless oil, 22.5 mg (43%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.02-8.08 (m, 3H), 7.97 (d, J = 8.4 Hz, 1H), 7.77 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.64-7.68 (m, 1H), 7.54-7.58 (m, 1H), 7.44-7.50 (m, 3H), 7.40 (d, J = 8.4 Hz, 1H), 3.64 (t, J = 7.2 Hz, 2H), 3.45 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.5, 161.3, 147.9, 137.1, 136.4, 133.1, 129.4, 128.8, 128.6, 128.2, 127.6, 126.9, 125.9, 122.0, 37.6, 32.8; HRMS (ESI): Exact mass calcd for $C_{18}H_{16}NO$ $[M+H]^+$ 262.1232, found 262.1226.

3-(4-Chloroquinolin-2-yl)-1-phenylpropan-1-one (5b)



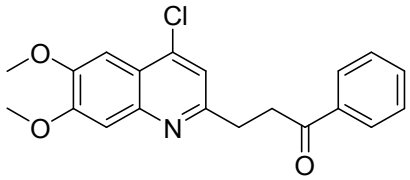
White solid, 40.2 mg (68%), mp 82−84 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.17 (dd, J = 8.4 Hz, 0.8 Hz, 1H), 8.02-8.04 (m, 2H), 7.96 (dd, J = 8.4 Hz, 0.4 Hz, 1H), 7.68-7.72 (m, 1H), 7.54-7.58 (m, 2H), 7.51 (s, 1H), 7.45-7.48 (m, 2H), 3.63 (t, J = 7.2 Hz, 2H), 3.41 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.2, 161.2, 148.7, 142.6, 137.0, 133.1, 130.3, 129.2, 128.7, 128.2, 126.8, 125.1, 124.0, 122.0, 37.1, 32.5; HRMS (ESI): Exact mass calcd for $C_{18}H_{15}ClNO$ $[M+H]^+$ 296.0842, found 296.0835.

3-(4-Chloroquinolin-2-yl)-1-p-tolylpropan-1-one (5c)



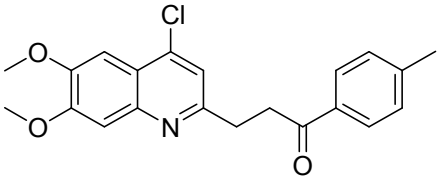
White solid, 52.7 mg (85%), mp 92−94 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.18 (dd, J = 8.4 Hz, 0.8 Hz, 1H), 7.97 (d, J = 7.6 Hz, 1H), 7.92-7.94 (m, 2H), 7.68-7.73 (m, 1H), 7.55-7.59 (m, 1H),7.51 (s, 1H), 7.26 (d, J = 8.0 Hz, 2H), 3.60 (t, J = 7.2 Hz, 2H), 3.40 (t, J = 7.2 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 198.8, 161.4, 148.8, 143.9, 142.6, 134.6, 130.3, 129.3, 129.2, 128.3, 126.8, 125.1, 124.0, 122.0, 37.2, 32.6, 21.7; HRMS (ESI): Exact mass calcd for $C_{19}H_{17}ClNO$ $[M+H]^+$ 310.0999, found 310.0997.

3-(4-Chloro-6,7-dimethoxyquinolin-2-yl)-1-phenylpropan-1-one (5d)



Colorless oil, 51.2 mg (72%); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.01-8.03 (m, 2H), 7.54-7.59 (m, 1H), 7.45-7.48 (m, 2H), 7.37 (s, 2H), 7.30 (s, 1H), 4.04 (s, 3H), 4.01 (s, 3H), 3.59 (t, J = 7.2 Hz, 2H), 3.35 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.1, 159.0, 153.0, 150.2, 146.8, 140.7, 137.1, 133.1, 128.6, 128.2, 130.4, 130.1, 107.9, 101.8, 56.3, 56.2, 37.7, 32.4; HRMS (ESI): Exact mass calcd for $C_{20}H_{19}ClNO_3$ $[M+H]^+$ 356.1053, found 356.1049.

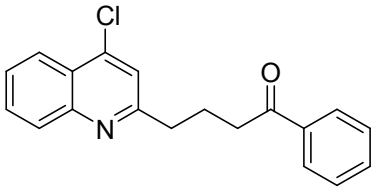
3-(4-Chloro-6,7-dimethoxyquinolin-2-yl)-1-p-tolylpropan-1-one (5e)



Colorless oil, 54.7 mg (74%); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.92 (d, J = 8.4 Hz, 2H), 7.37 (s, 2H), 7.32 (s, 1H), 7.25 (d, J = 8.4 Hz, 2H), 4.04 (s, 3H), 4.01 (s, 3H), 3.56 (t, J = 7.2 Hz, 2H), 3.34 (t, J = 7.2 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ

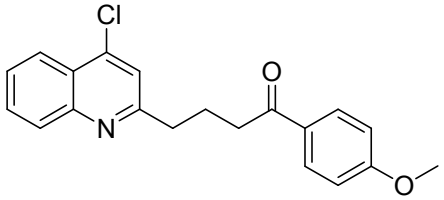
198.7, 159.1, 153.1, 150.1, 145.7, 143.9, 140.7, 134.5, 129.3, 128.3, 130.4, 130.1, 107.9, 101.8, 56.3, 56.2, 37.7, 32.4, 21.7; HRMS (ESI): Exact mass calcd for $C_{21}H_{21}ClNO_3$ $[M+H]^+$ 370.1210, found 370.1205.

4-(4-Chloroquinolin-2-yl)-1-phenylbutan-1-one (5f)



White solid, 43.4 mg (70%), mp 74−76 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.19 (d, J = 8.4 Hz, 1H), 8.06 (d, J = 8.4 Hz, 1H), 7.94-7.96 (m, 2H), 7.72-7.76 (m, 1H), 7.53-7.61 (m, 2H), 7.43-7.46 (m, 3H), 3.10 (t, J = 7.2 Hz, 2H), 3.07 (t, J = 7.2 Hz, 2H), 2.27-2.34 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 199.9, 162.0, 148.6, 143.0, 137.0, 133.1, 130.5, 129.1, 128.6, 128.1, 126.9, 125.1, 124.1, 121.5, 38.0, 37.8, 23.9; HRMS (ESI): Exact mass calcd for $C_{19}H_{17}ClNO$ $[M+H]^+$ 310.0999, found 310.0992.

4-(4-Chloroquinolin-2-yl)-1-(4-methoxyphenyl)butan-1-one (5g)

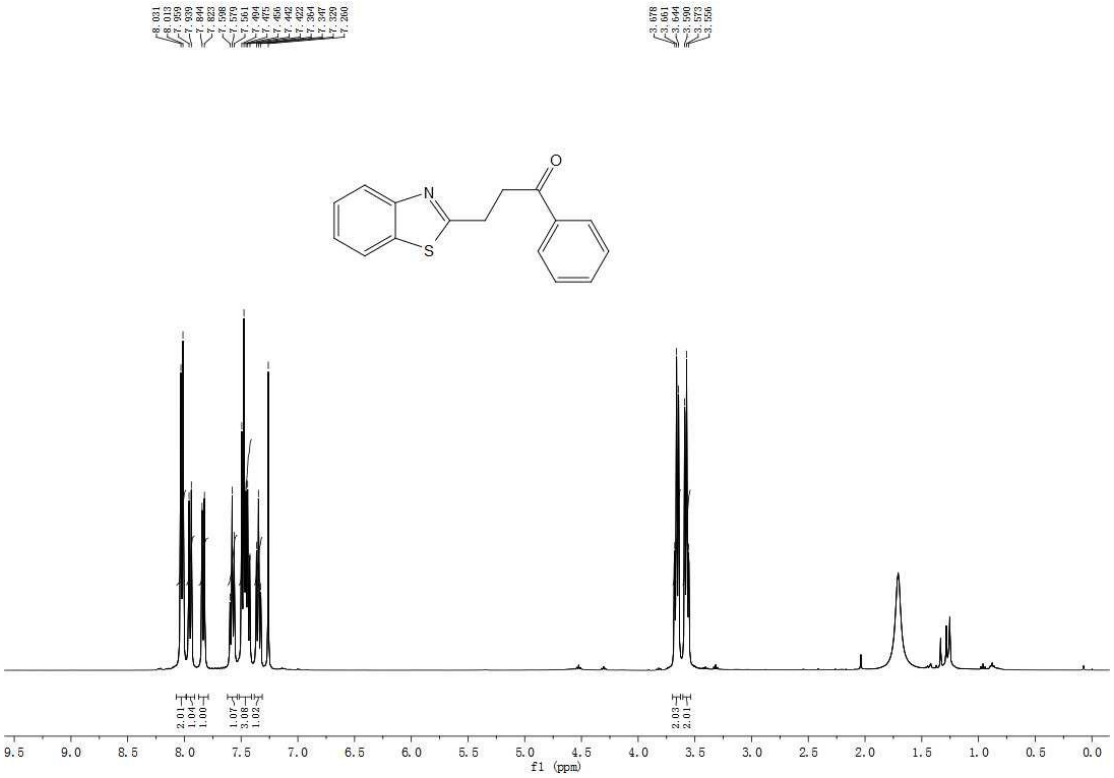


White solid, 53.7 mg (79%), mp 76−78 °C; ^{1}H NMR (400MHz, $CDCl_3$) δ 8.19 (dd, J = 8.4 Hz, 0.8 Hz, 1H), 8.05 (d, J = 8.4 Hz, 1H), 7.92-7.96 (m, 2H), 7.72-7.76 (m, 1H), 7.57-7.61 (m, 1H), 7.47 (s, 1H), 6.90-6.94 (m, 2H), 3.86 (s, 3H), 3.06 (t, J = 7.2 Hz, 2H), 3.04 (t, J = 7.2 Hz, 2H), 2.25-2.32 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 198.5, 163.5, 162.1, 148.7, 142.8, 130.4, 130.1, 129.2, 126.9, 125.1, 124.1, 121.6, 113.8, 55.5, 38.2, 37.5, 24.2; HRMS (ESI): Exact mass calcd for $C_{20}H_{19}ClNO_2$ $[M+H]^+$ 340.1104, found 340.1097.

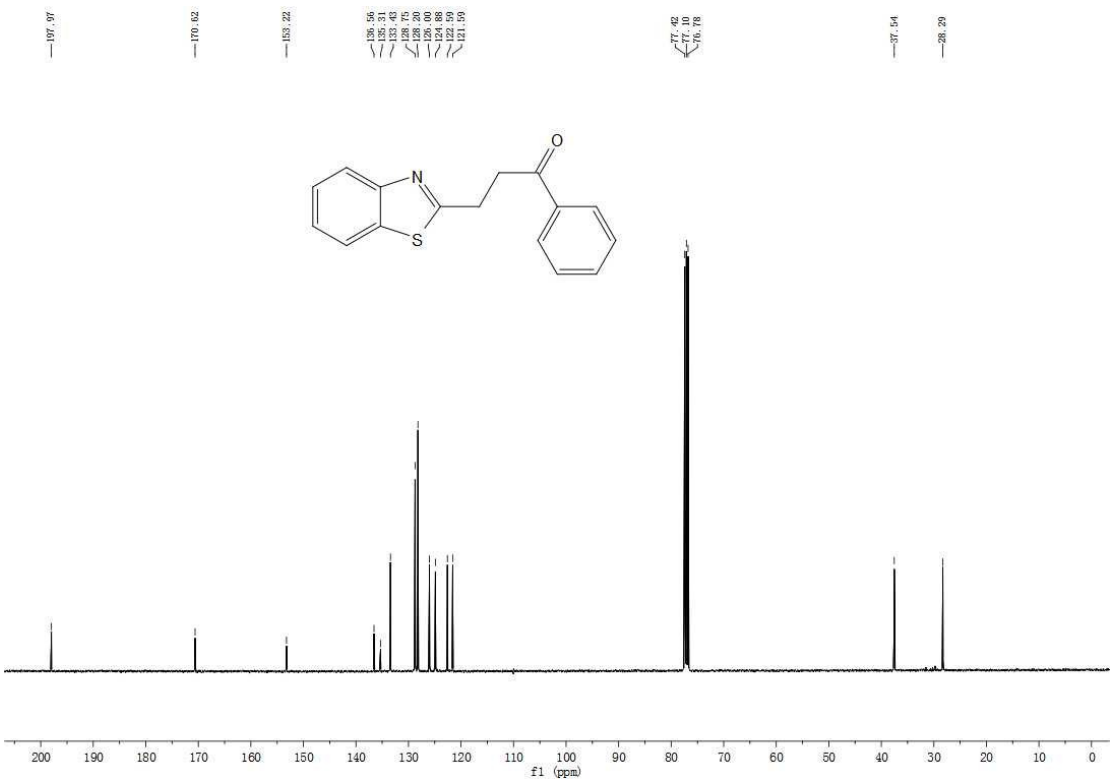
5. References

(1) (a) Ilangovan, A.; Saravanakumar, S.; Malayappasamy, S. Org. Lett. 2013, 15, 4968. (b) Jiao, J.; Nguyen, L. X.; Patterson, D. R.; Flowers II, R. A. Org. Lett. 2007, 9, 1323.
(2) (a) Casey, B. M.; Eakin, C. A.; Flowers II, R. A. Tetrahedron Lett. 2009, 50, 1264. (b) Xu, H.-J.; Zhu, F.-F.; Shen, Y.-Y.; Wan, X.; Feng, Y.-S. Tetrahedron 2012, 68, 4145.

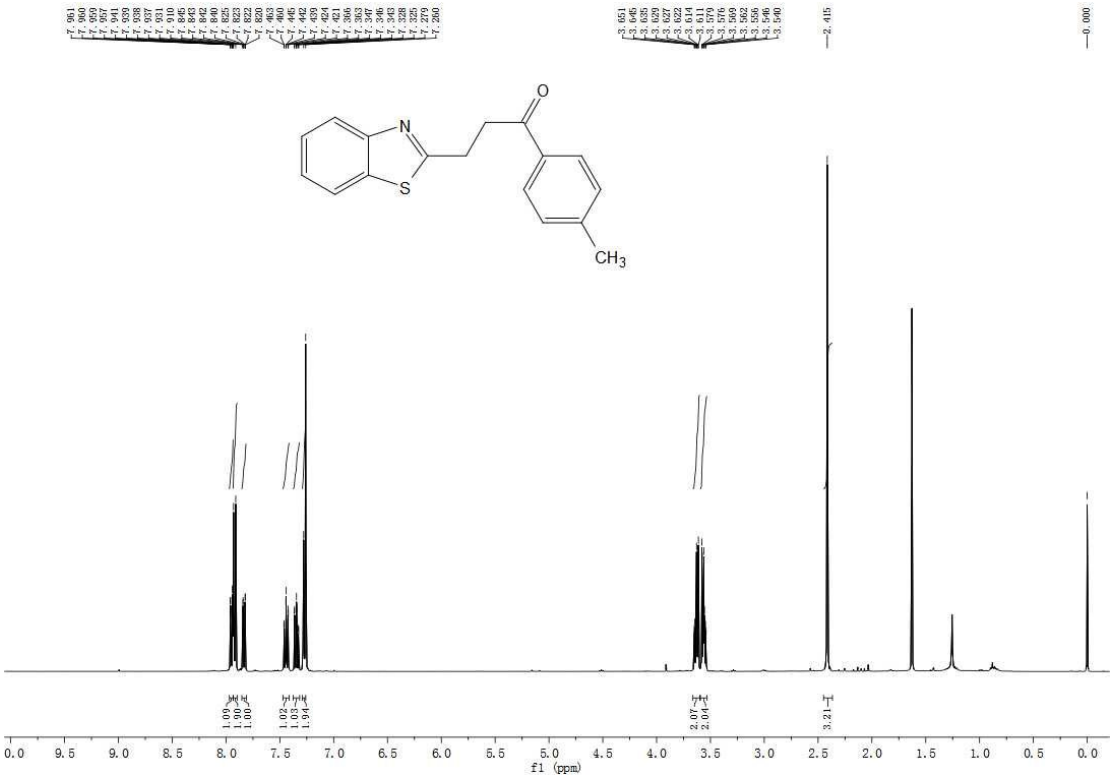
6. ^{1}H NMR and ^{13}C NMR Spectra of the Target Compounds



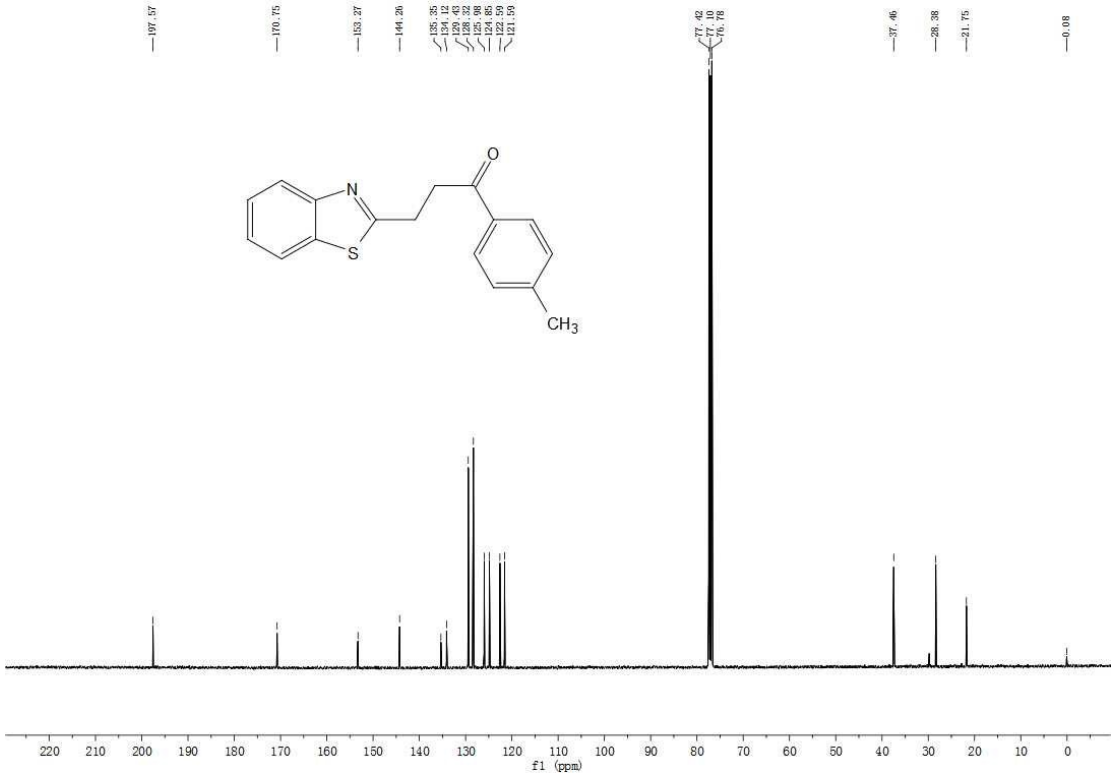
^{1}H NMR spectrum of 3a



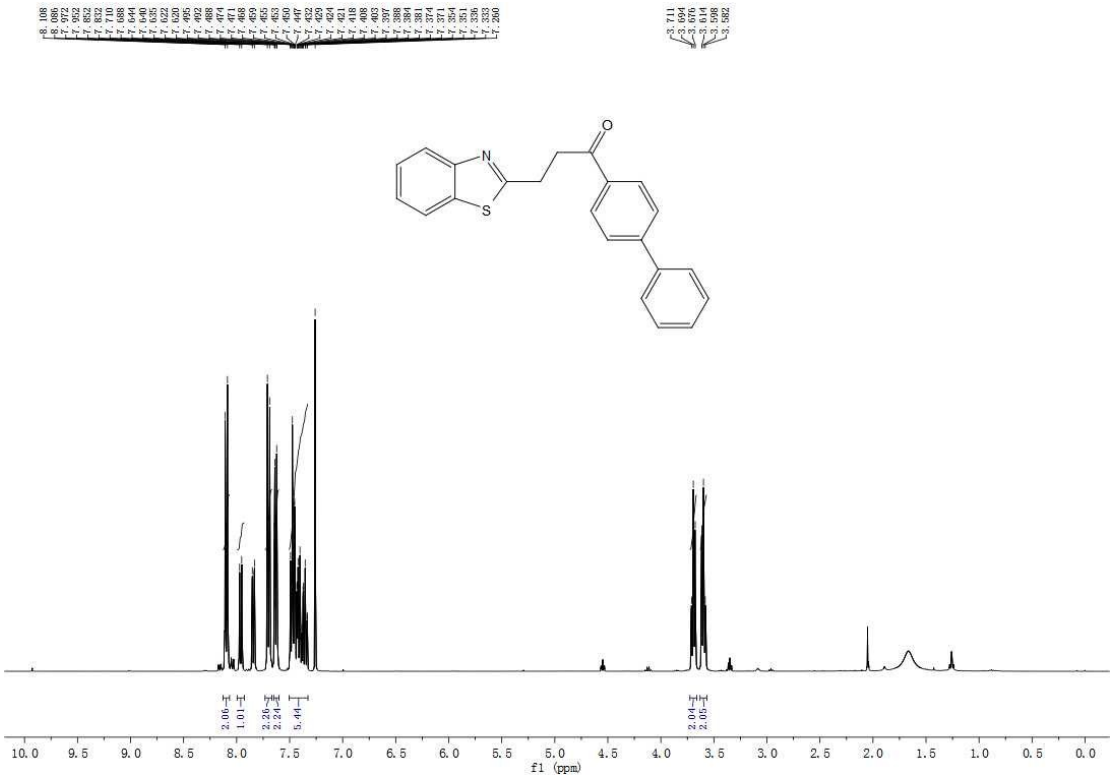
^{13}C NMR spectrum of 3a



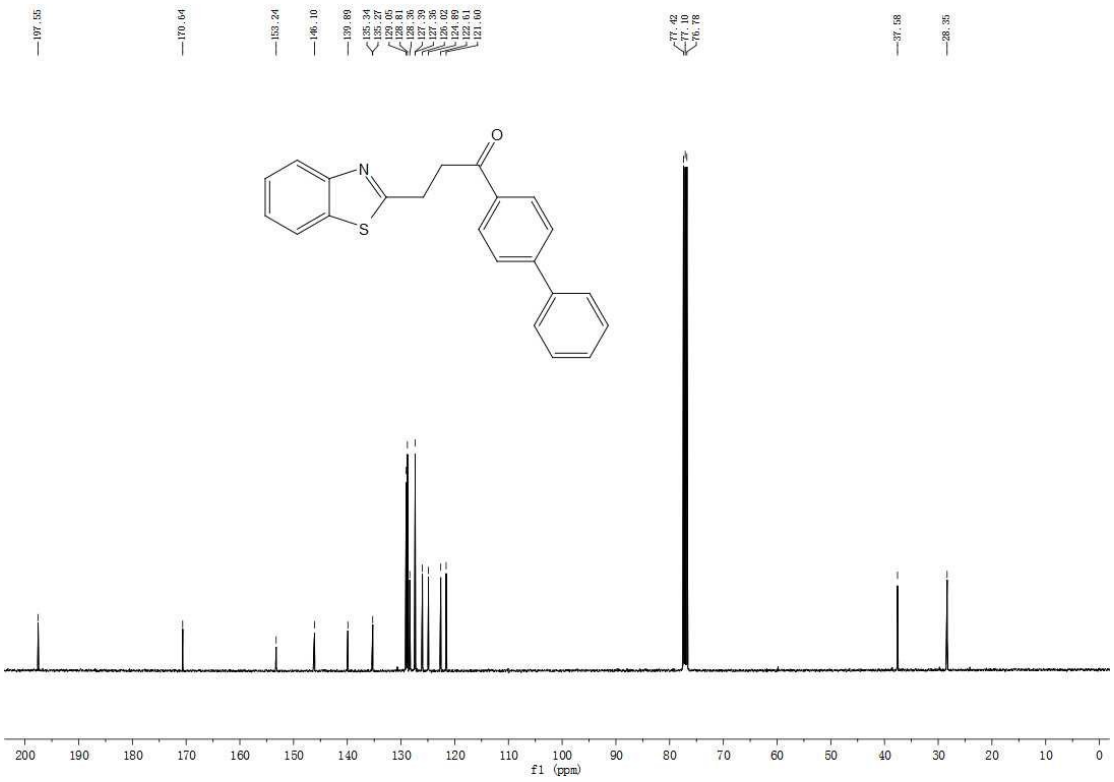
^{1}H NMR spectrum of 3b



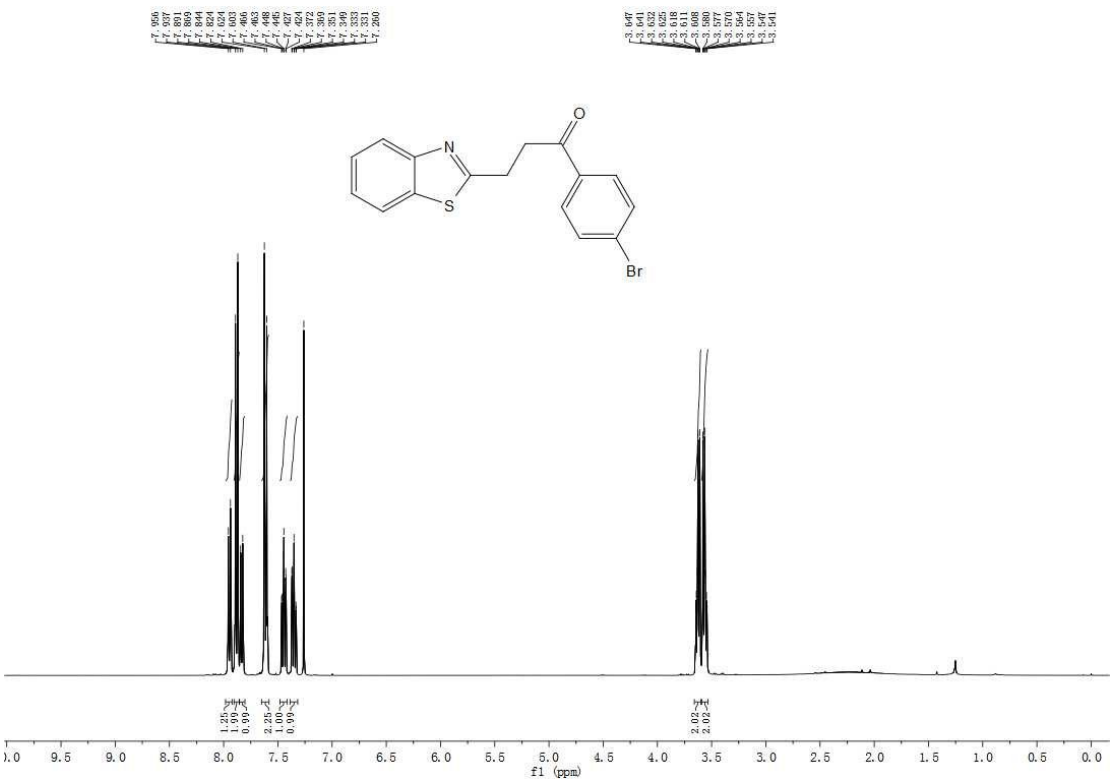
^{13}C NMR spectrum of 3b



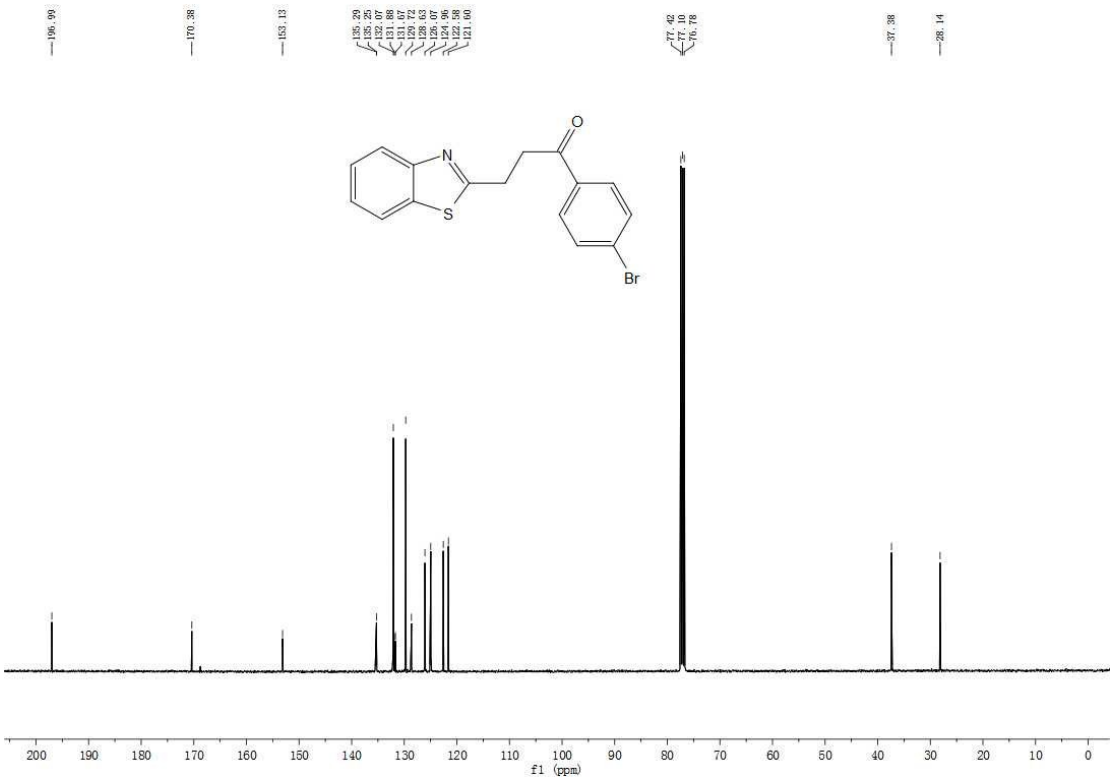
^{1}H NMR spectrum of 3c



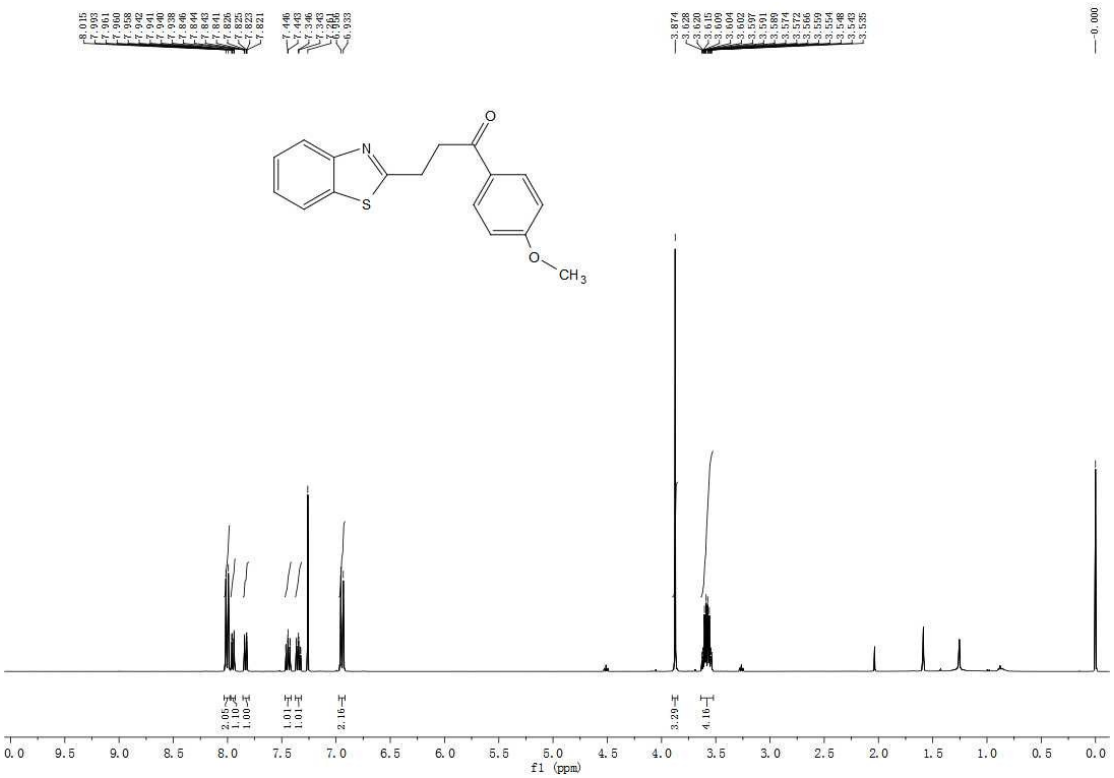
^{13}C NMR spectrum of 3c



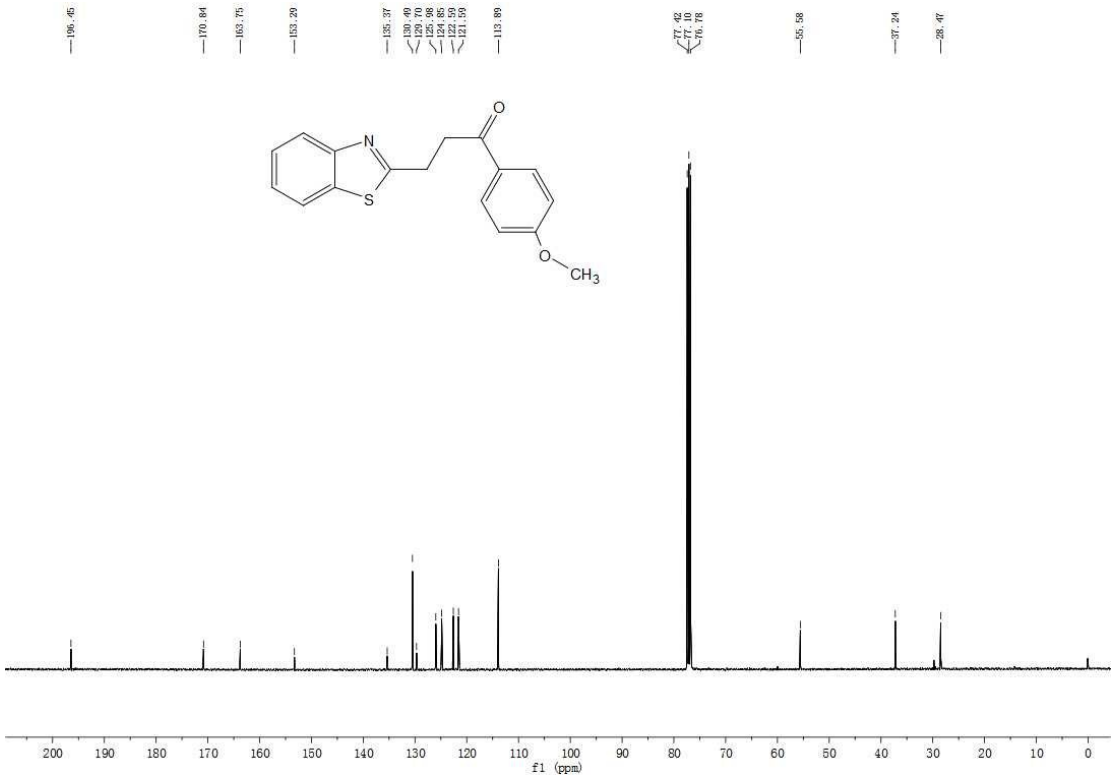
^{1}H NMR spectrum of 3d



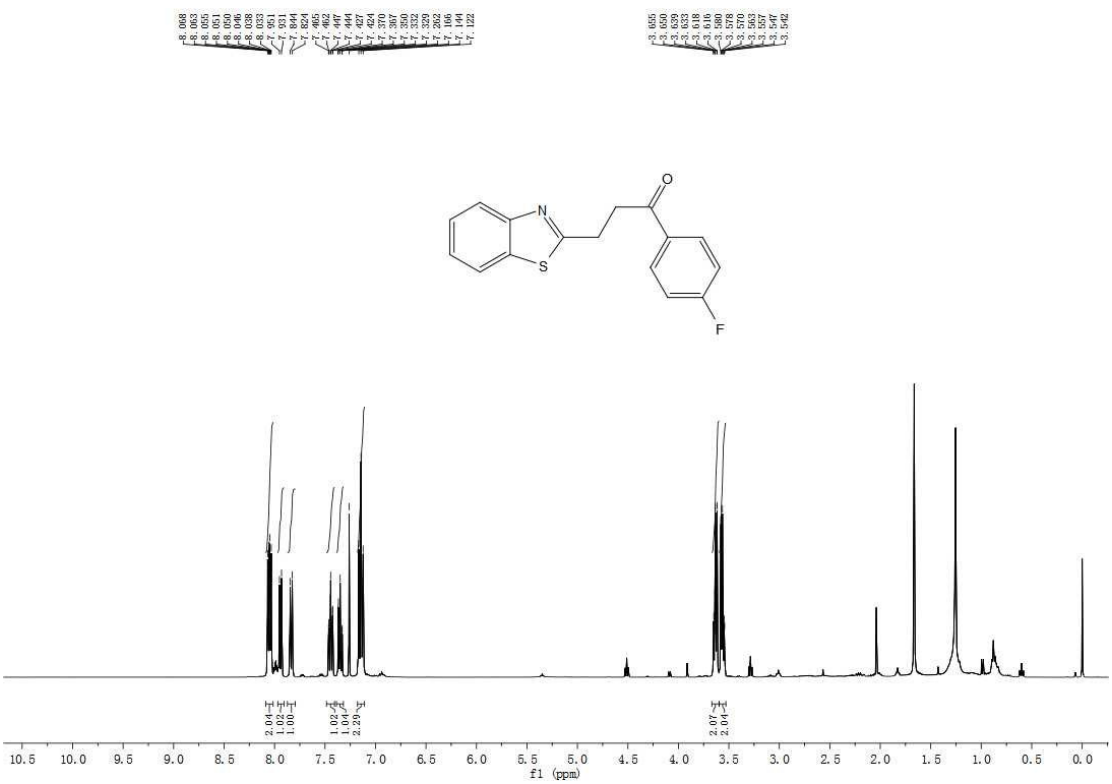
^{13}C NMR spectrum of 3d



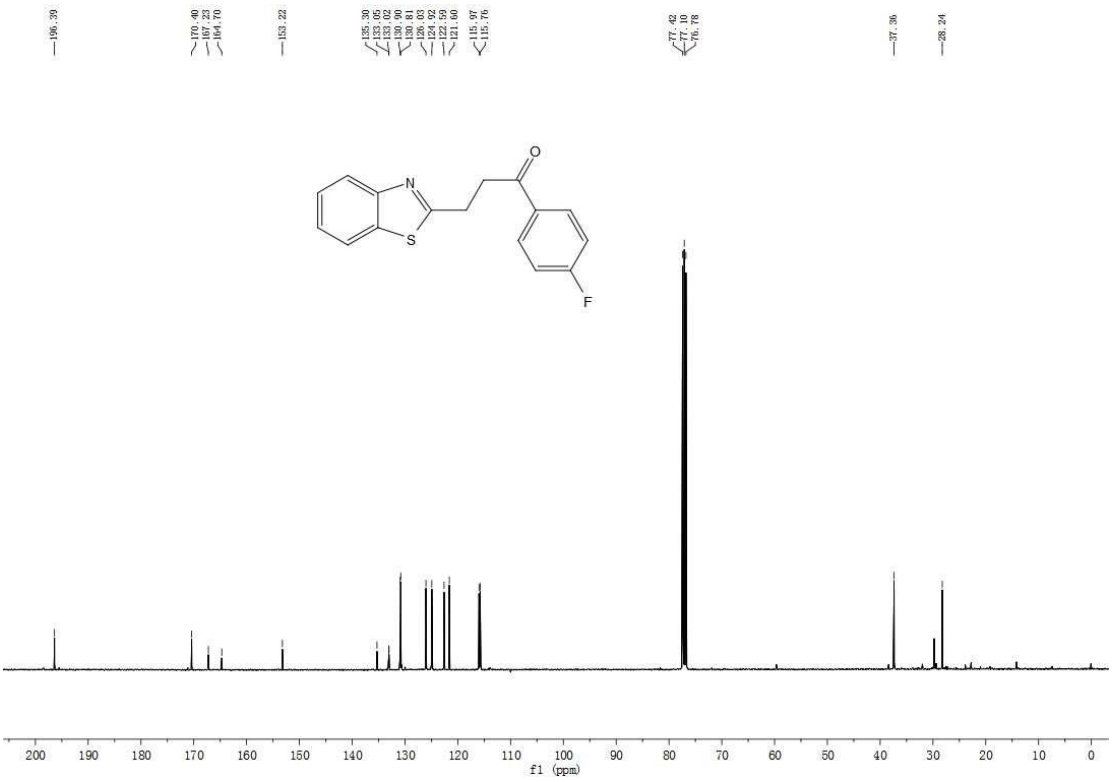
^{1}H NMR spectrum of 3e



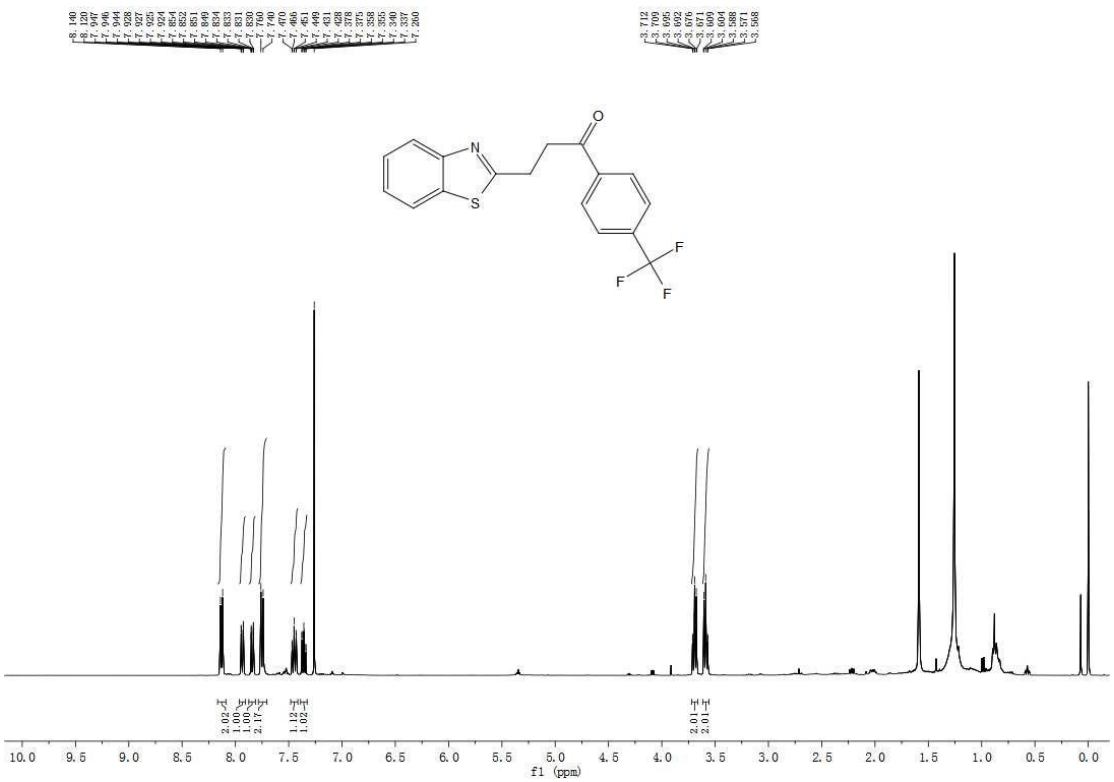
^{13}C NMR spectrum of 3e



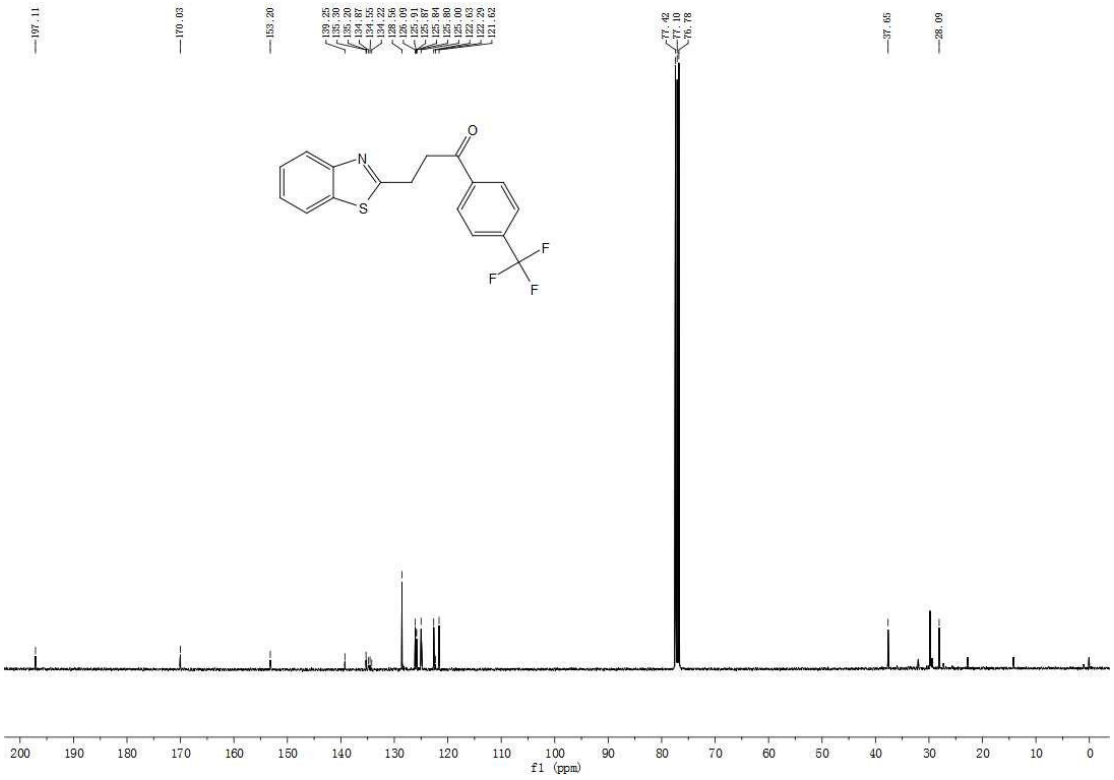
^{1}H NMR spectrum of 3f



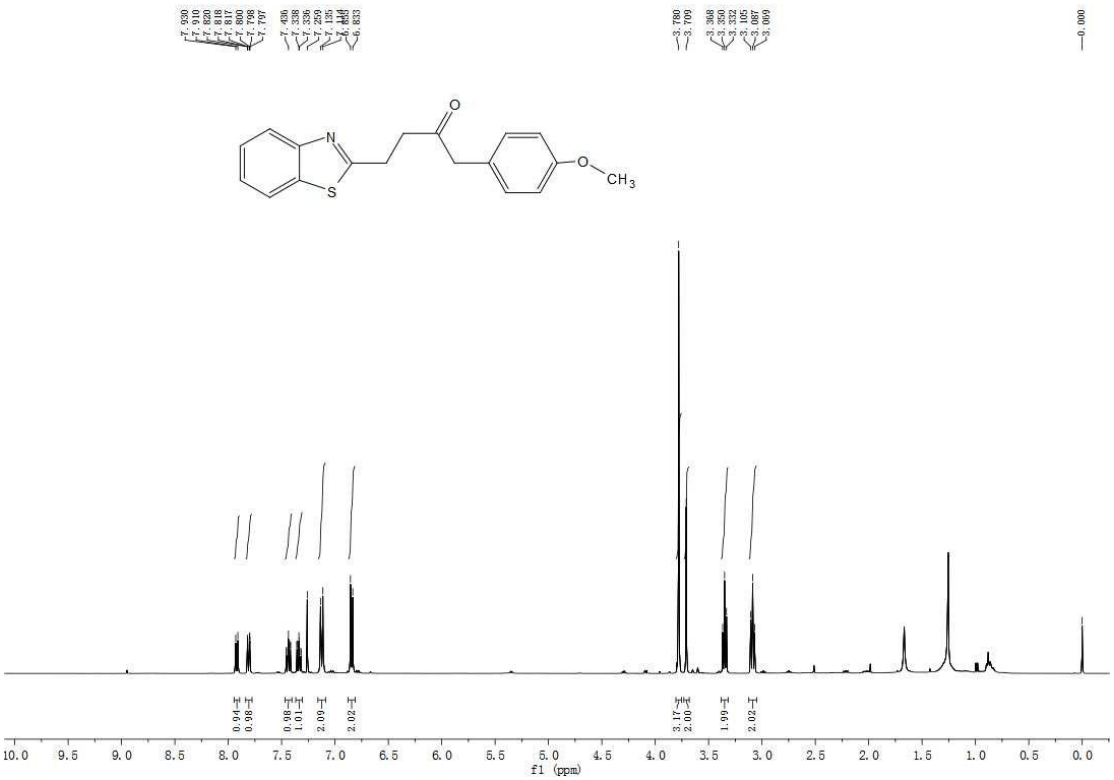
^{13}C NMR spectrum of 3f



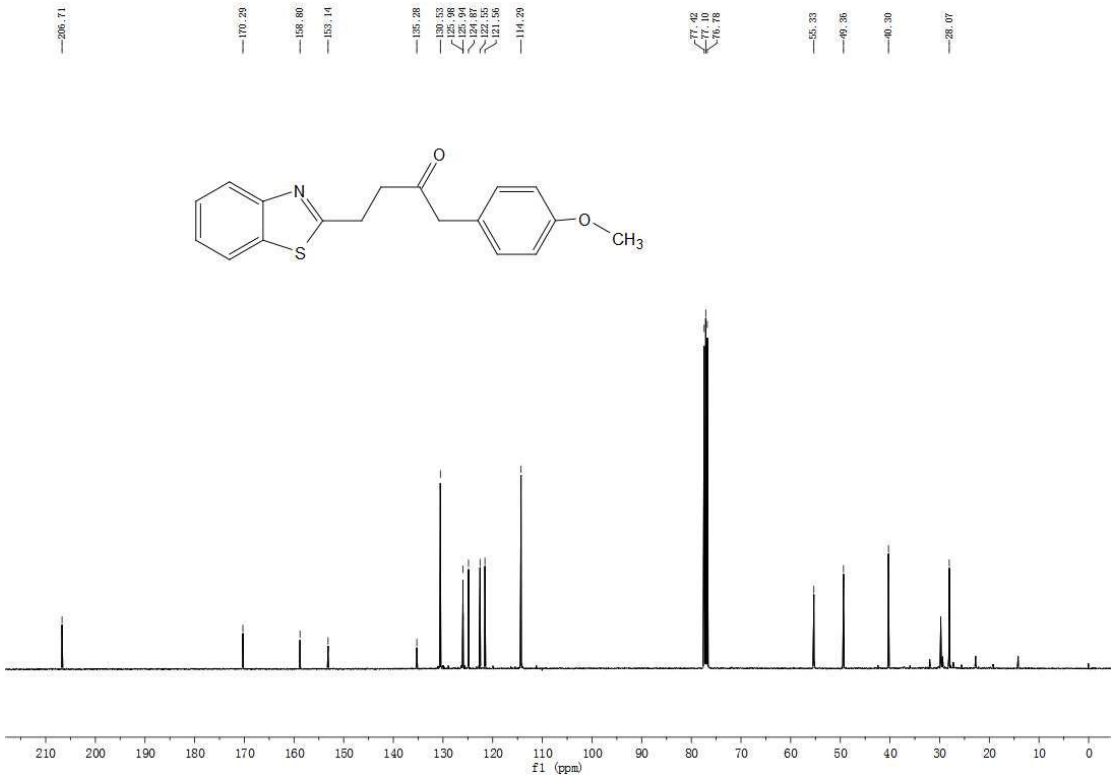
^{1}H NMR spectrum of 3g



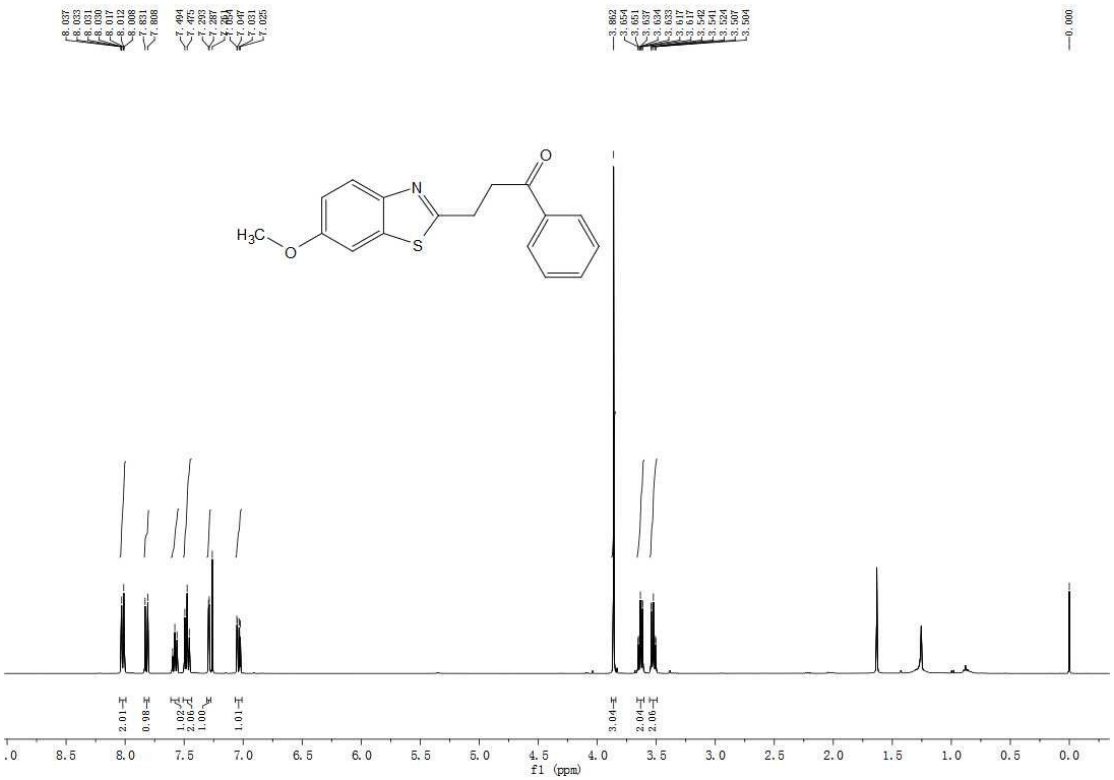
^{13}C NMR spectrum of 3g



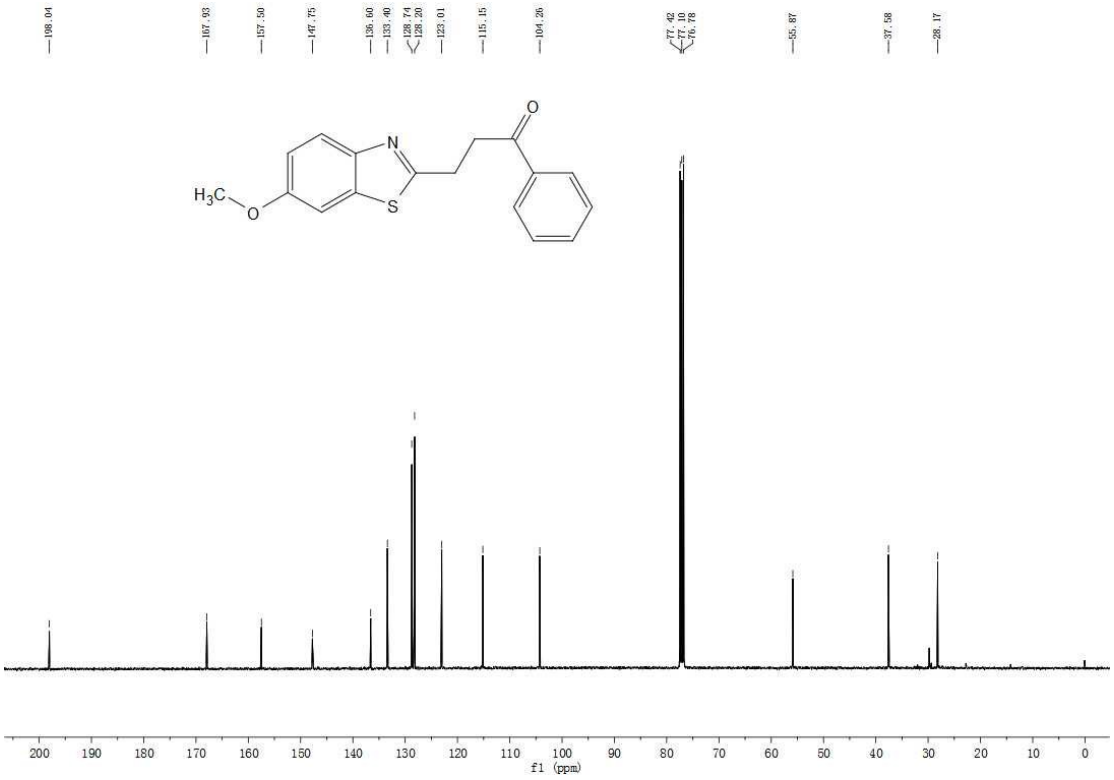
^{1}H NMR spectrum of 3h



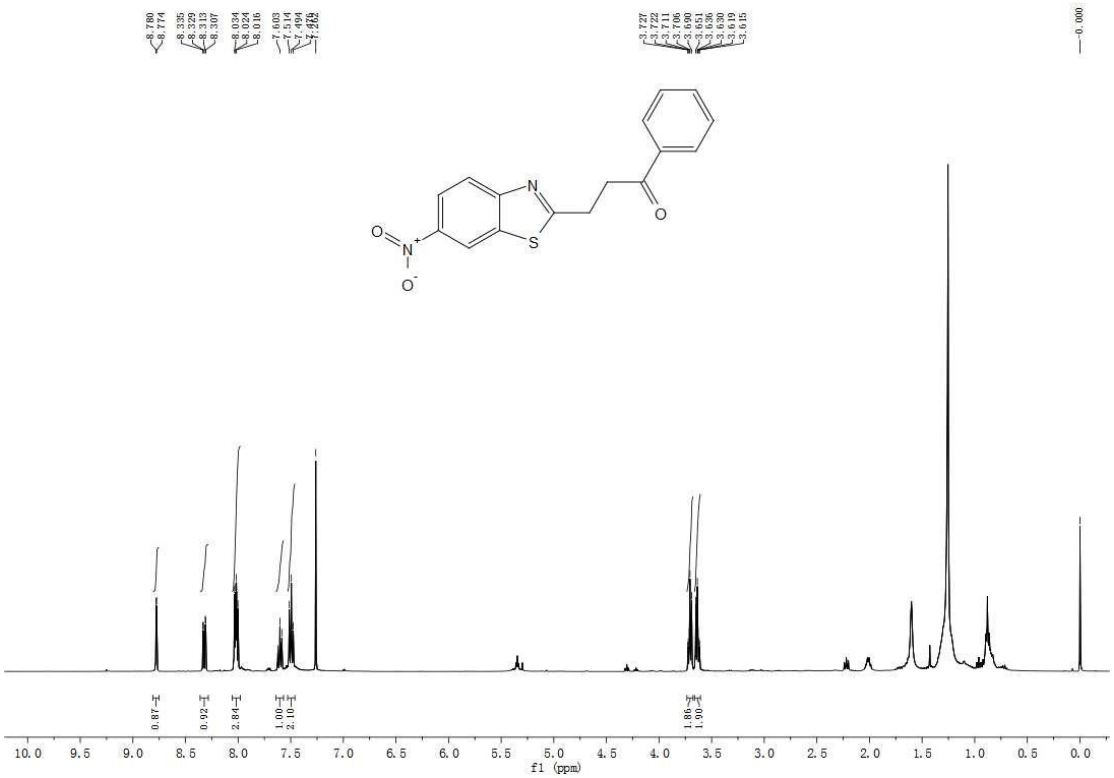
^{13}C NMR spectrum of 3h



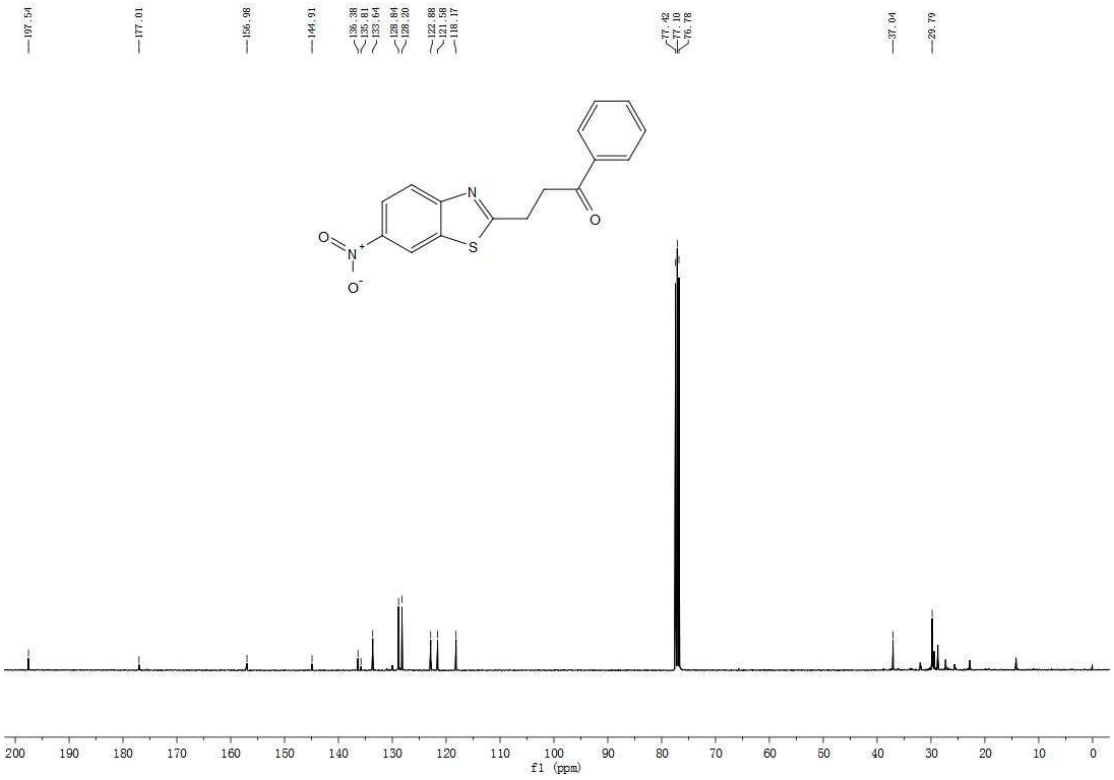
^{1}H NMR spectrum of 3i



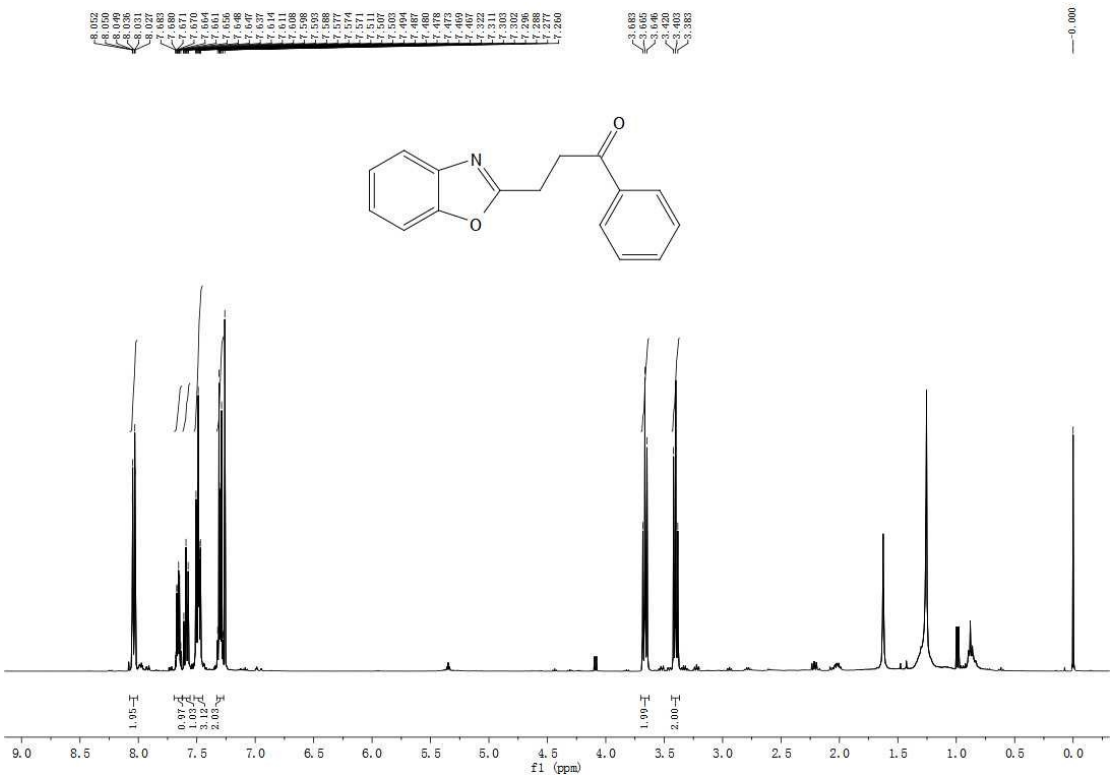
^{13}C NMR spectrum of 3i



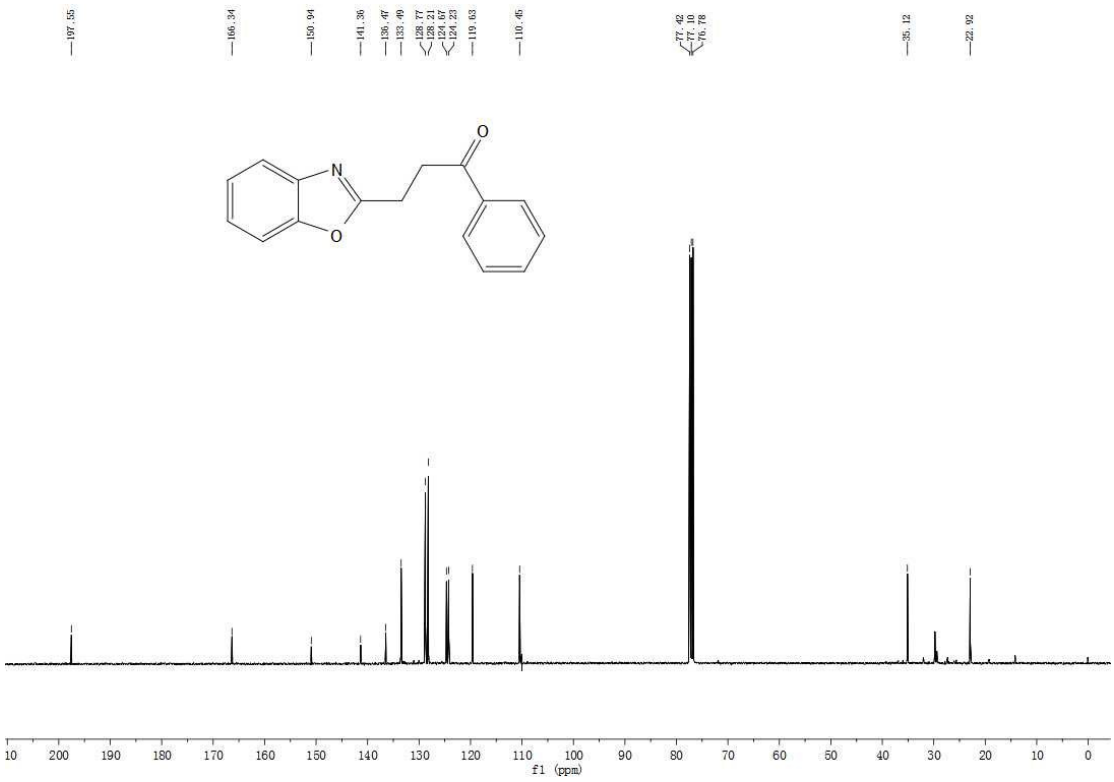
^{1}H NMR spectrum of 3j



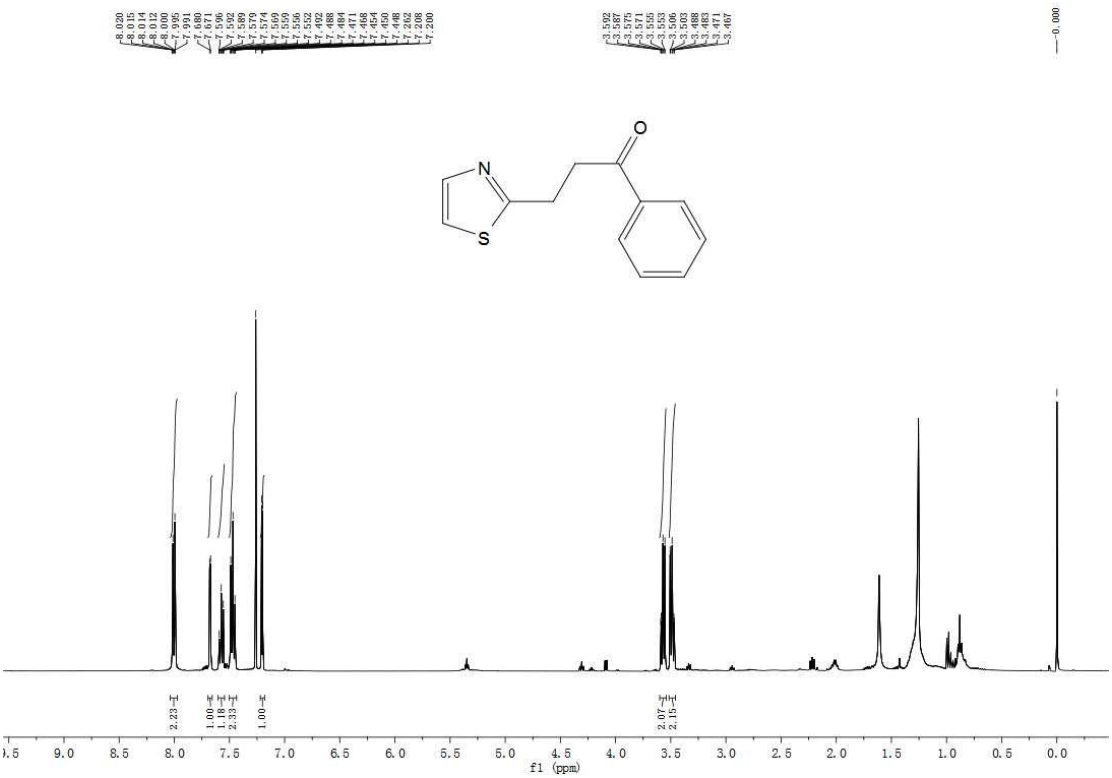
^{13}C NMR spectrum of 3j



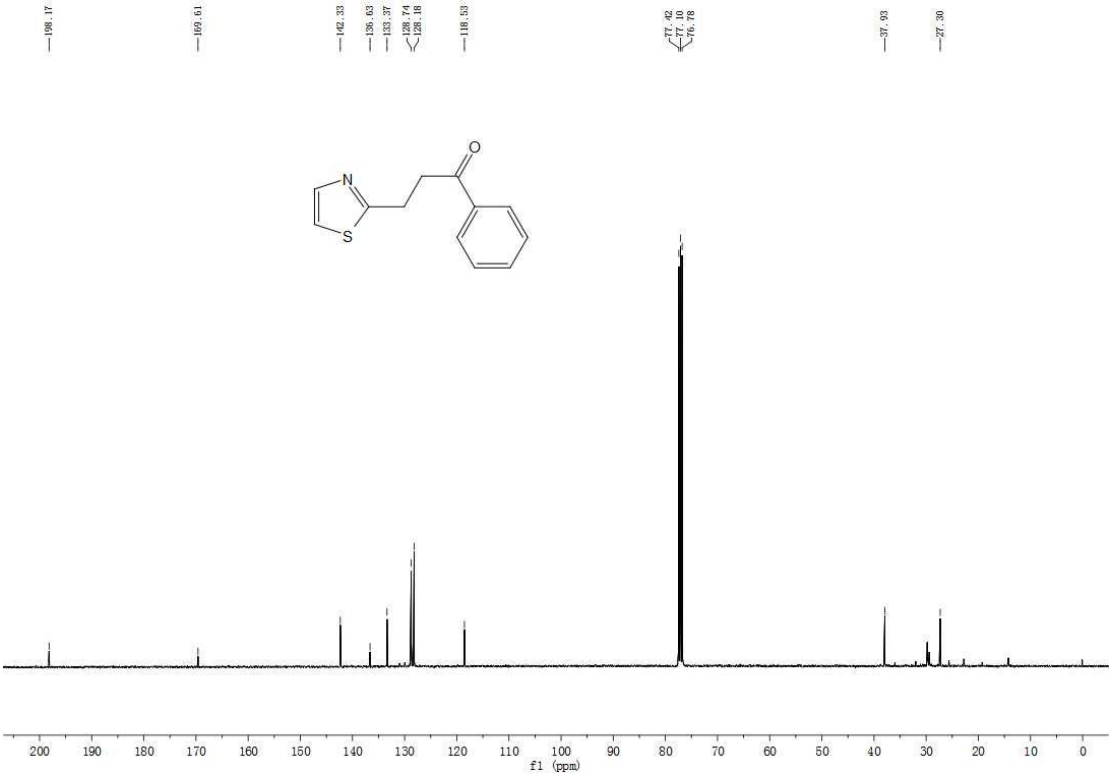
^{1}H NMR spectrum of 3k



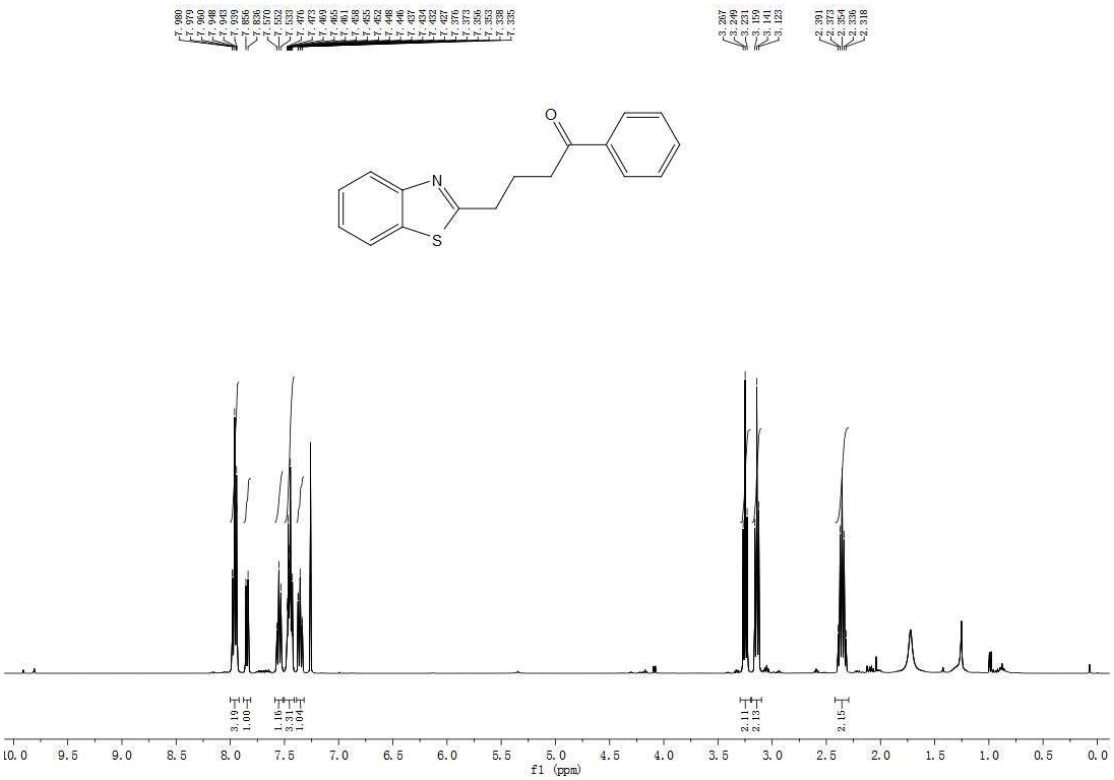
^{13}C NMR spectrum of 3k



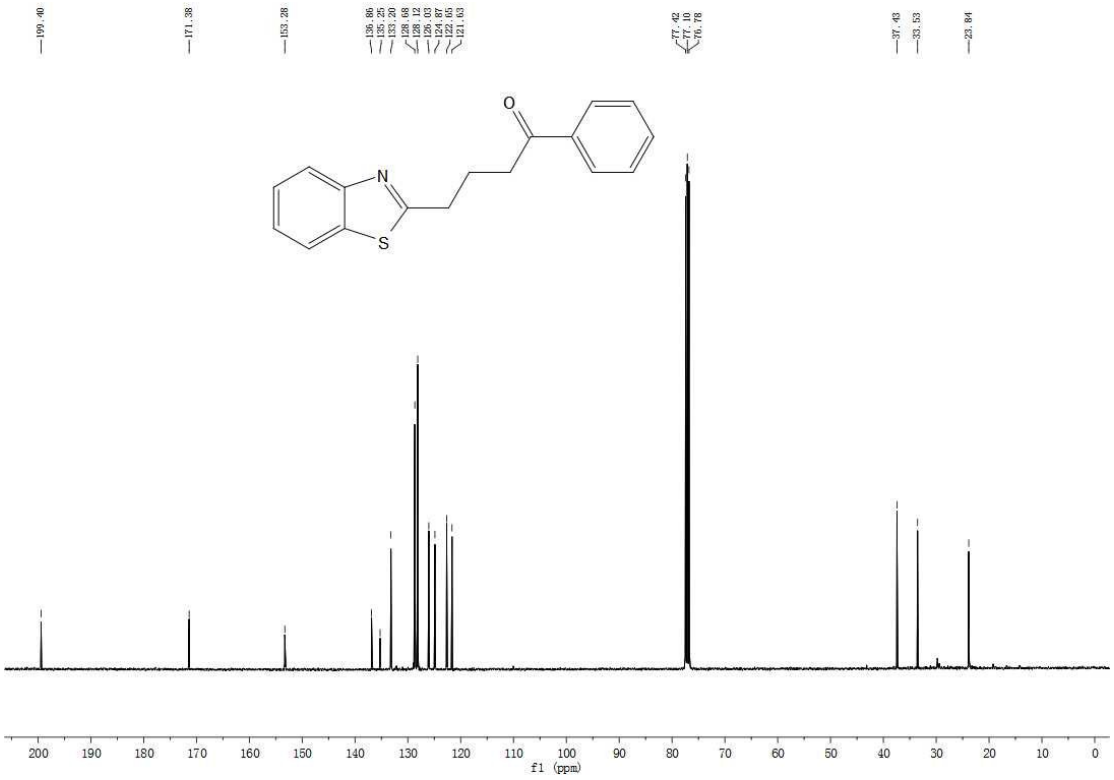
^{1}H NMR spectrum of 3l



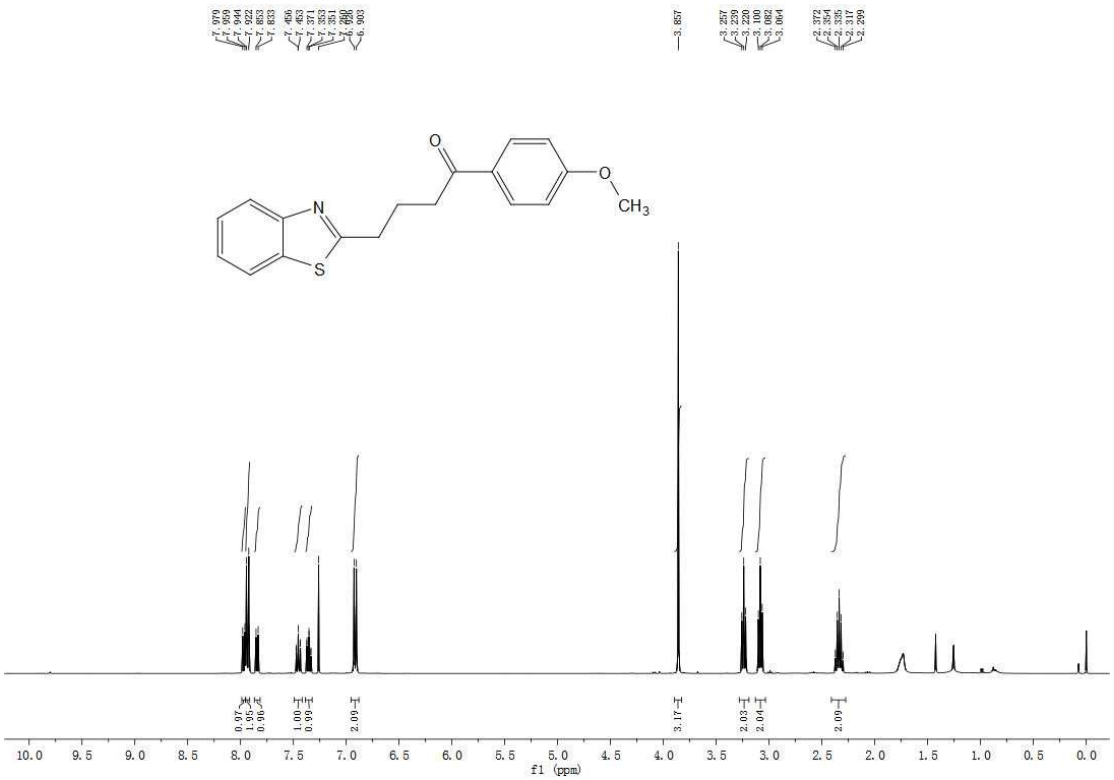
^{13}C NMR spectrum of 3l



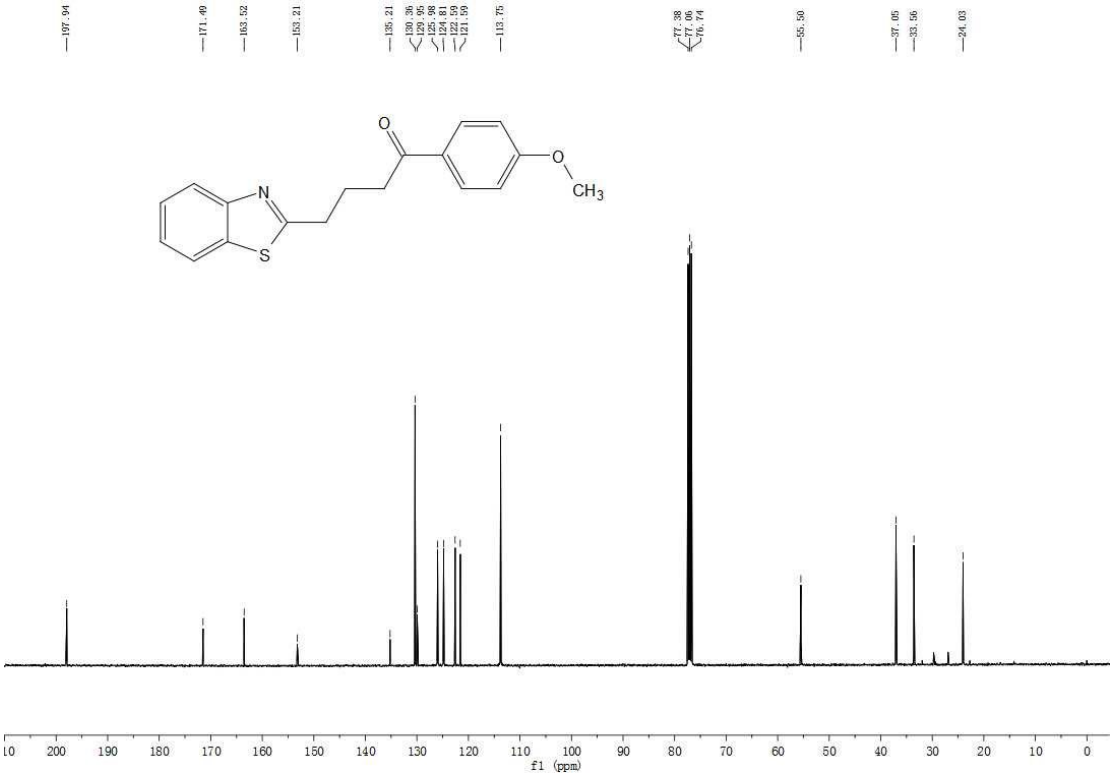
^{1}H NMR spectrum of 3m



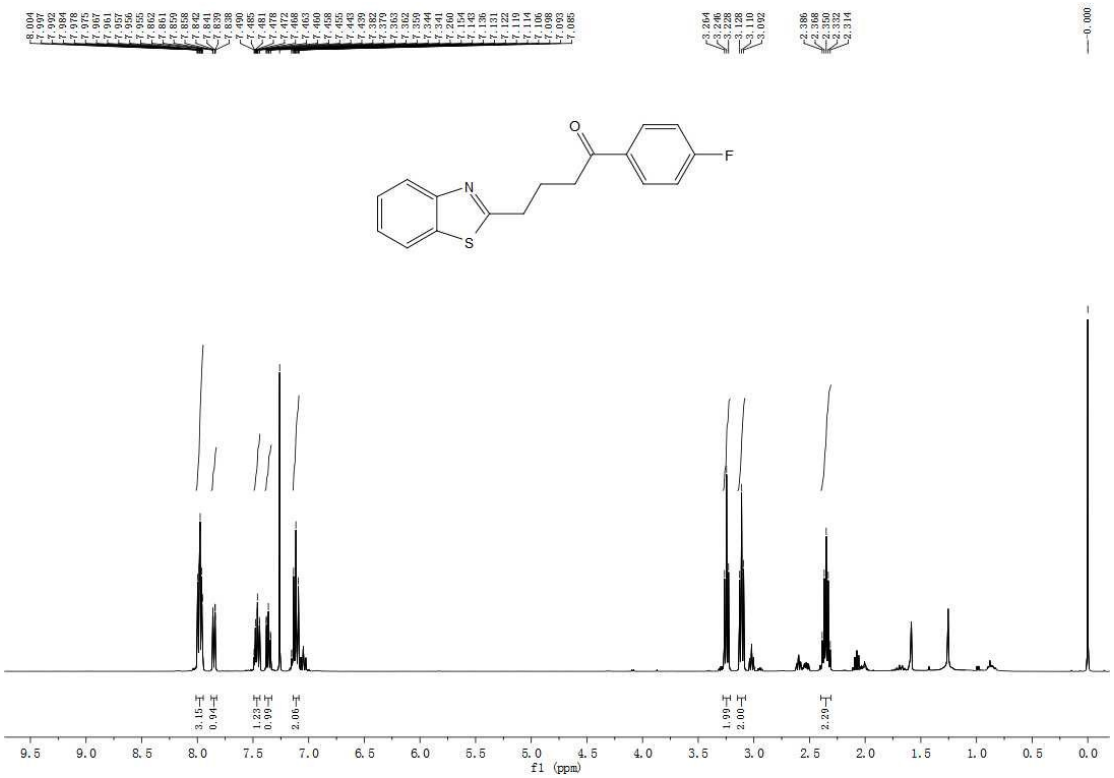
^{13}C NMR spectrum of 3m



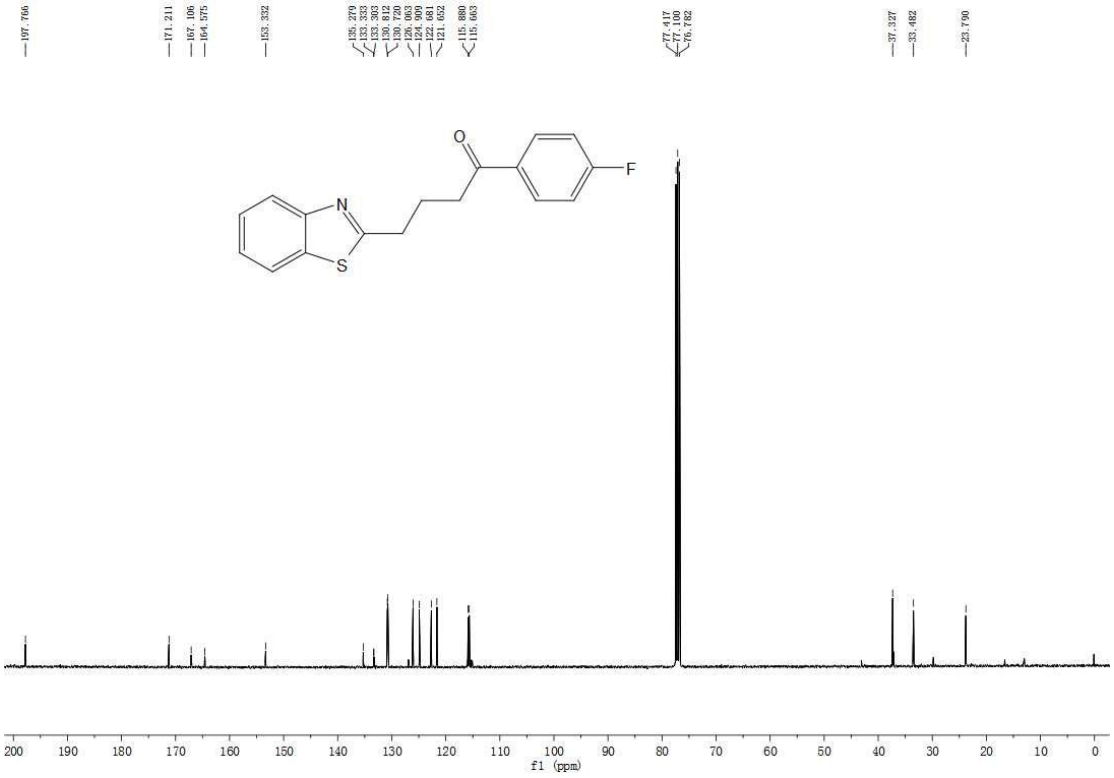
^{1}H NMR spectrum of 3n



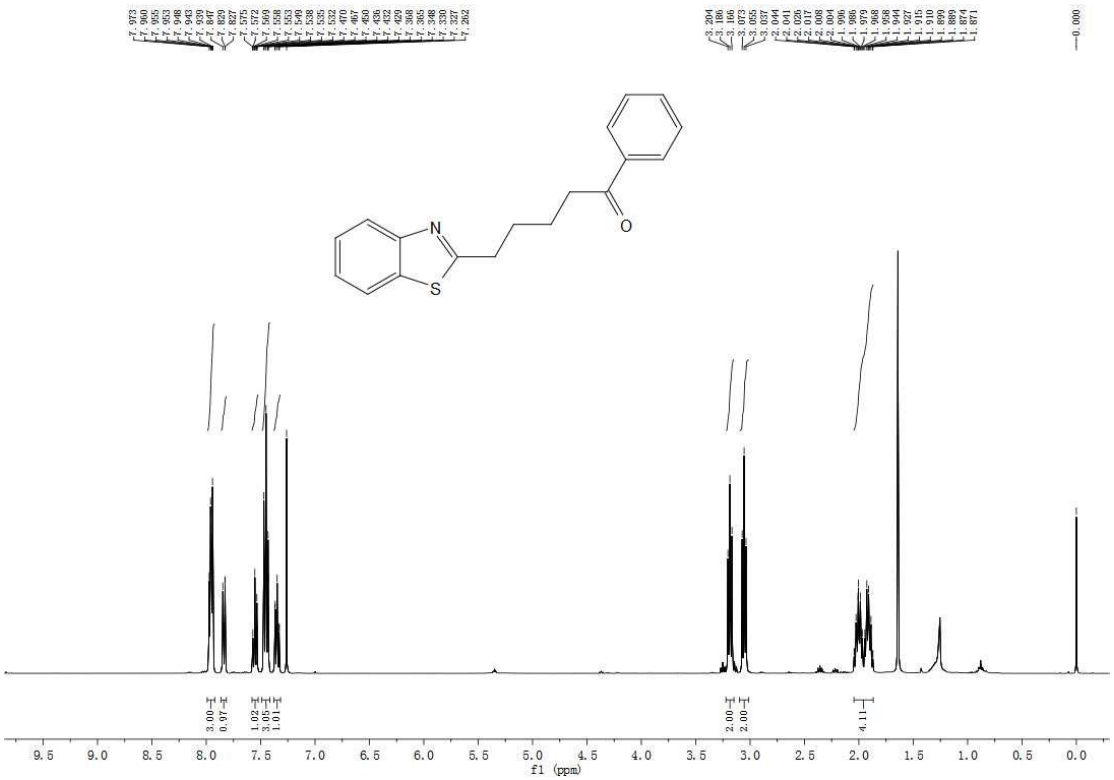
^{13}C NMR spectrum of 3n



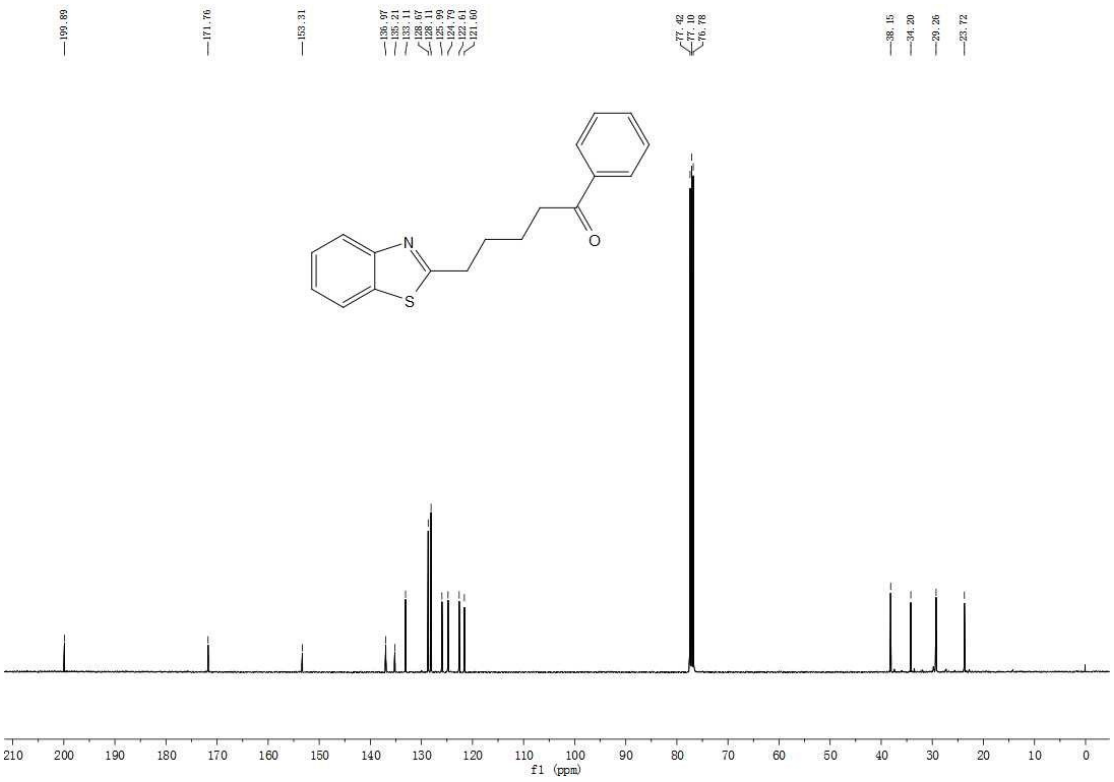
^{1}H NMR spectrum of 3o



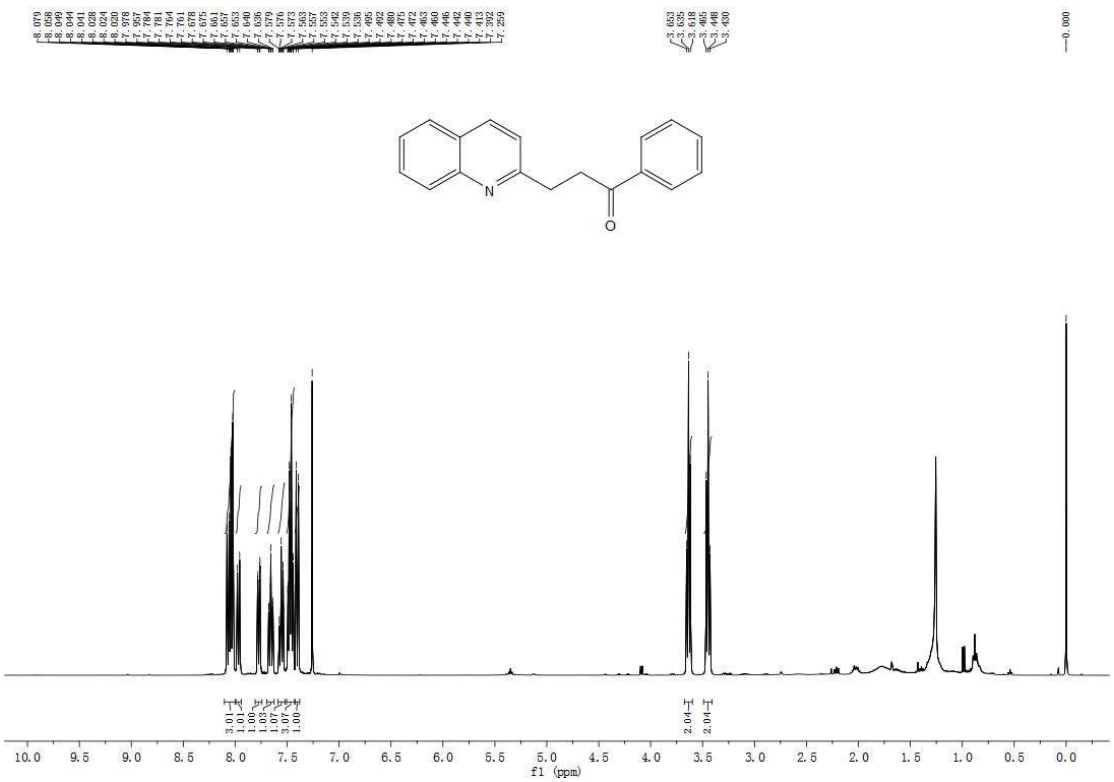
^{13}C NMR spectrum of 3o



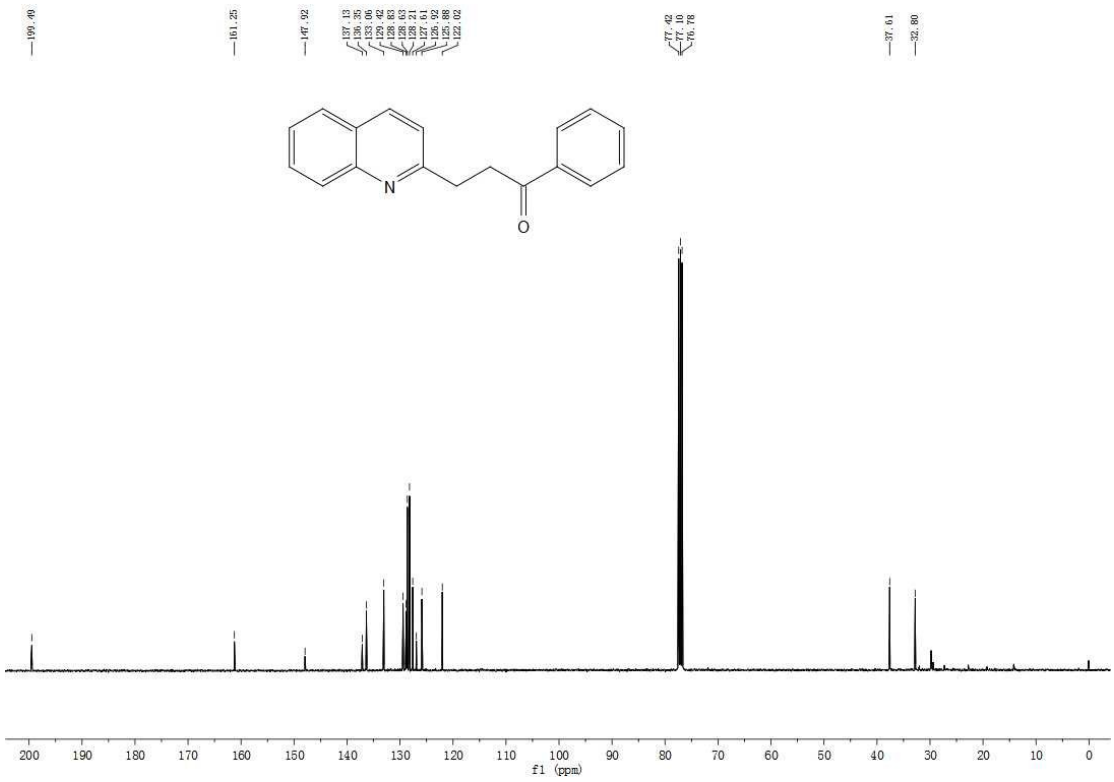
^{1}H NMR spectrum of 3p



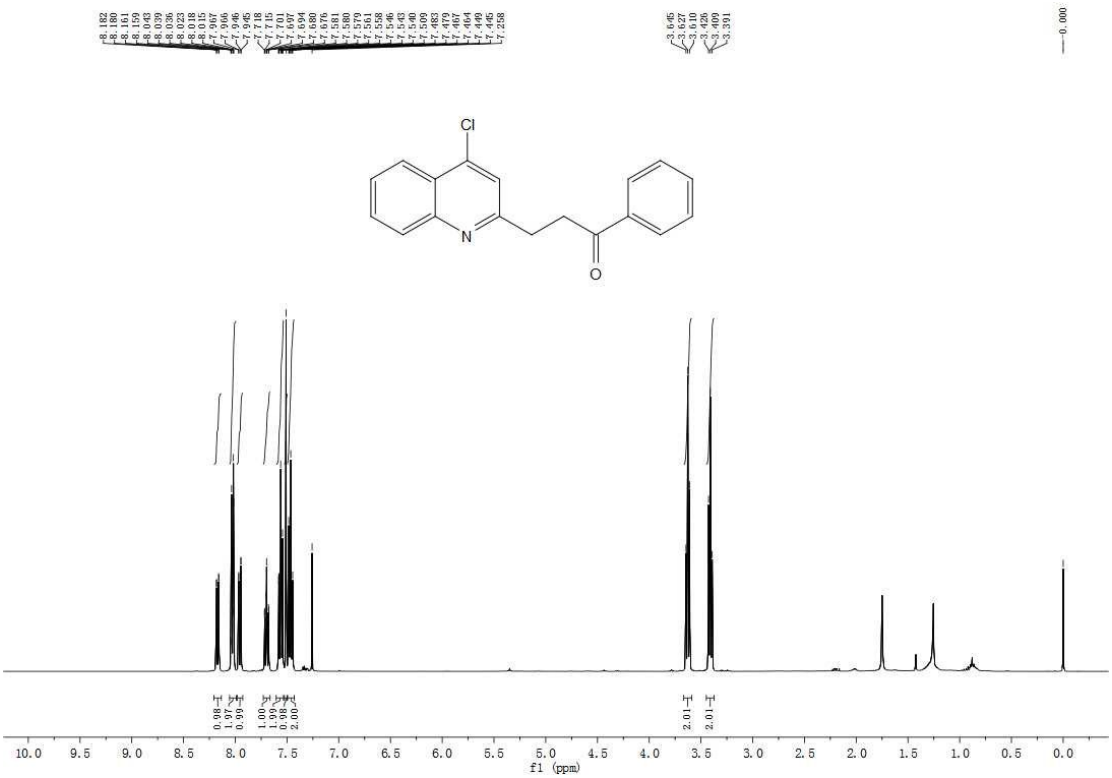
^{13}C NMR spectrum of 3p



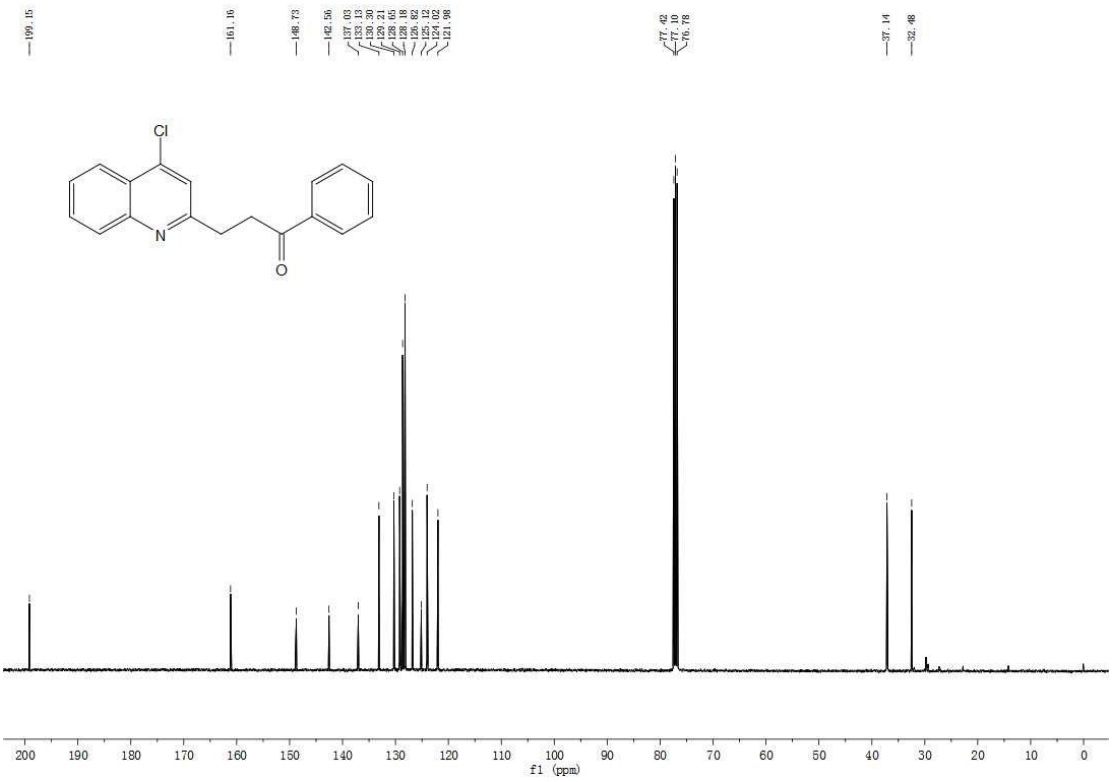
^{1}H NMR spectrum of 5a



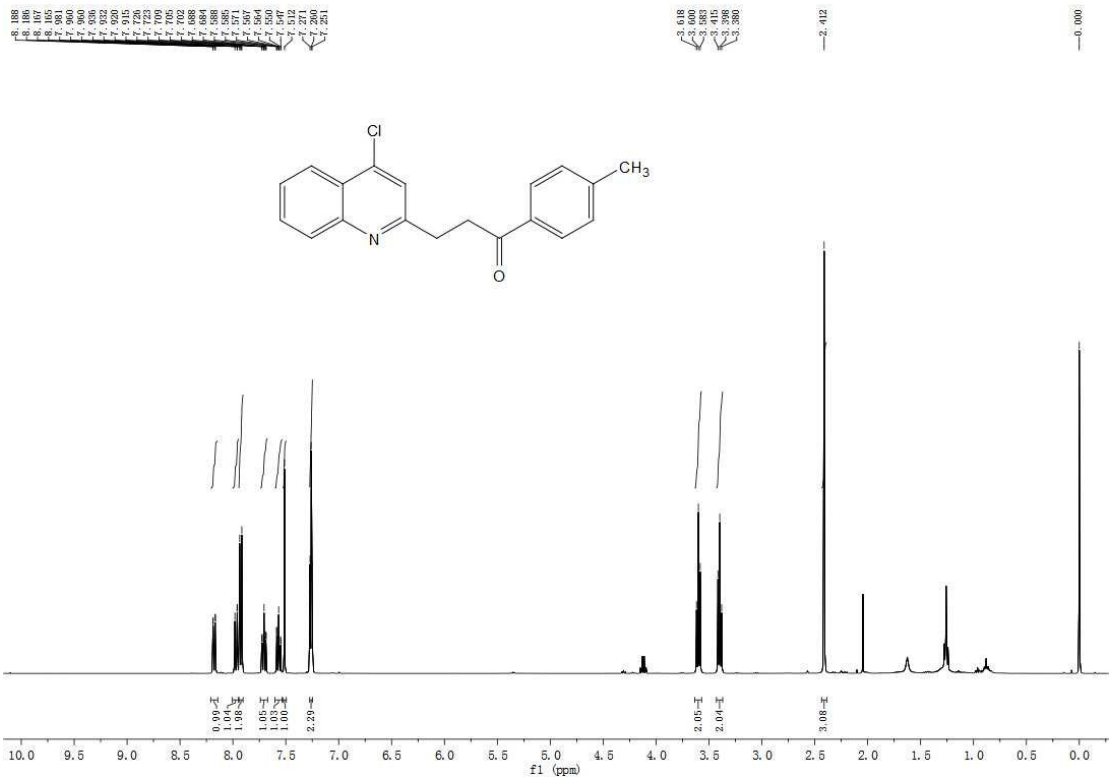
^{13}C NMR spectrum of 5a



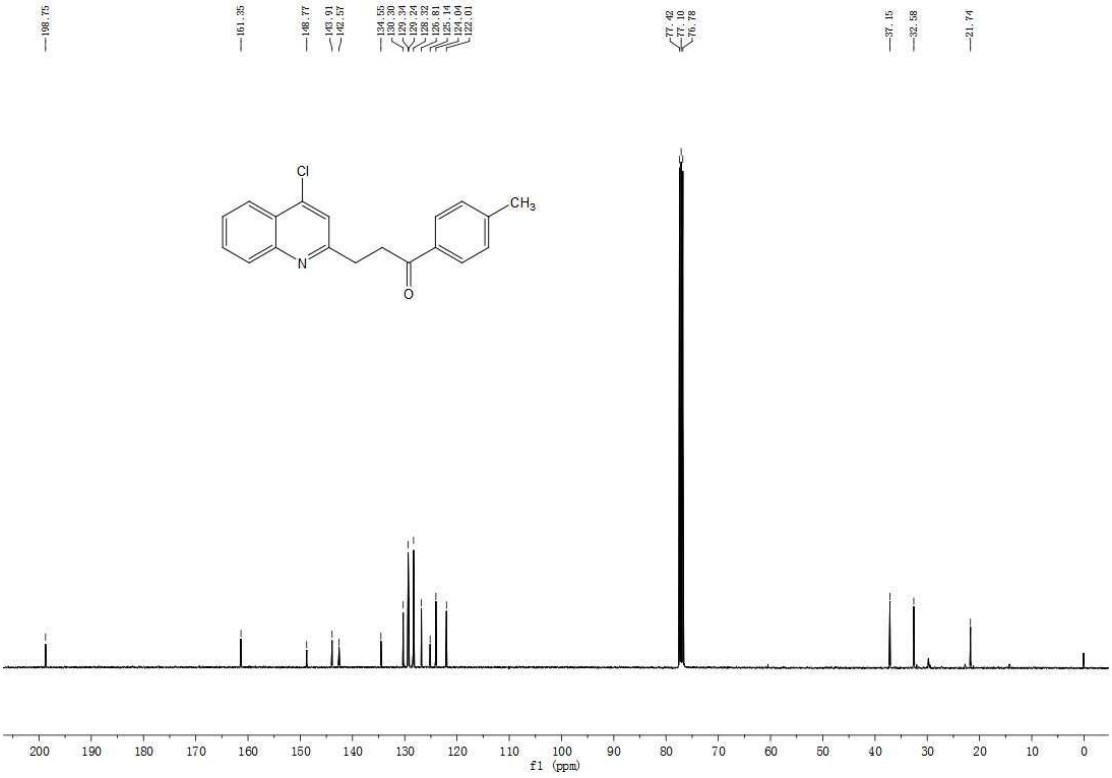
^{1}H NMR spectrum of 5b



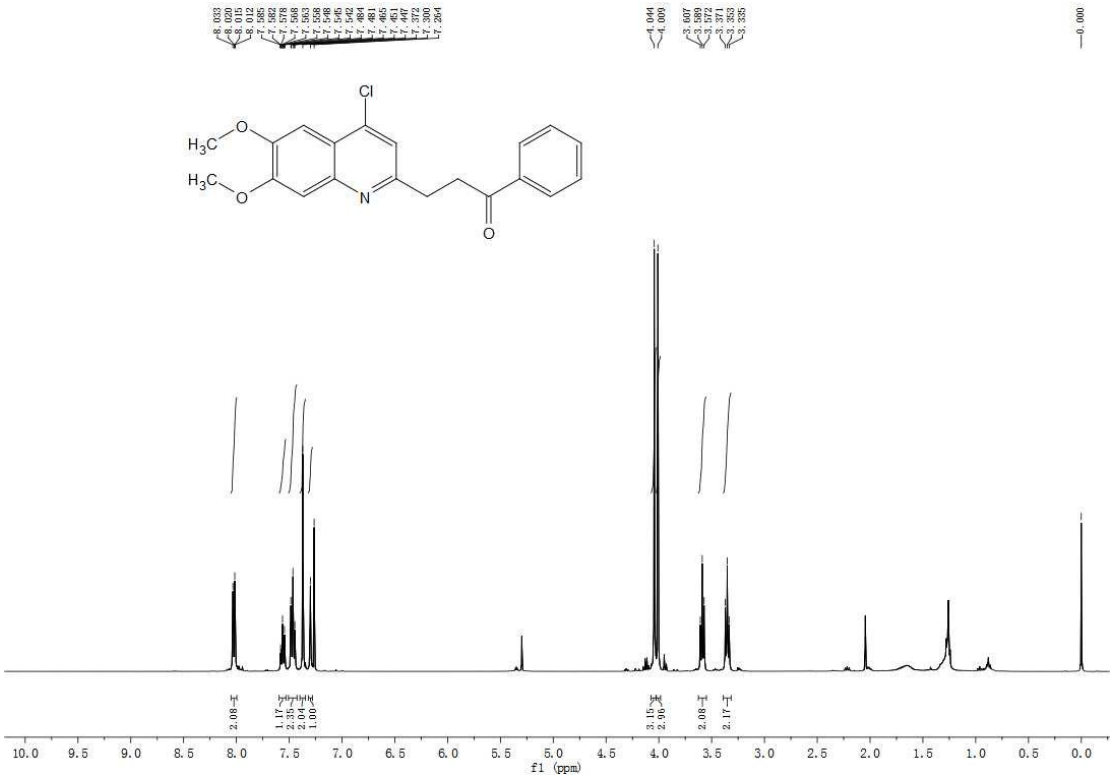
^{13}C NMR spectrum of 5b



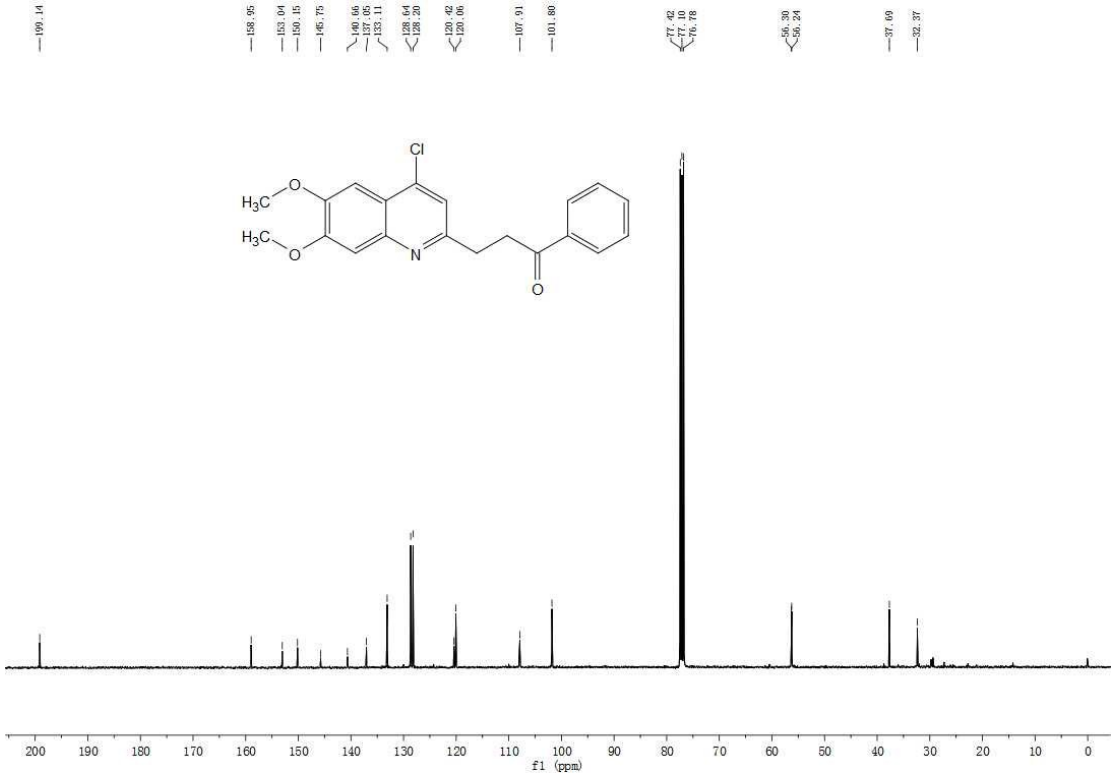
^{1}H NMR spectrum of 5c



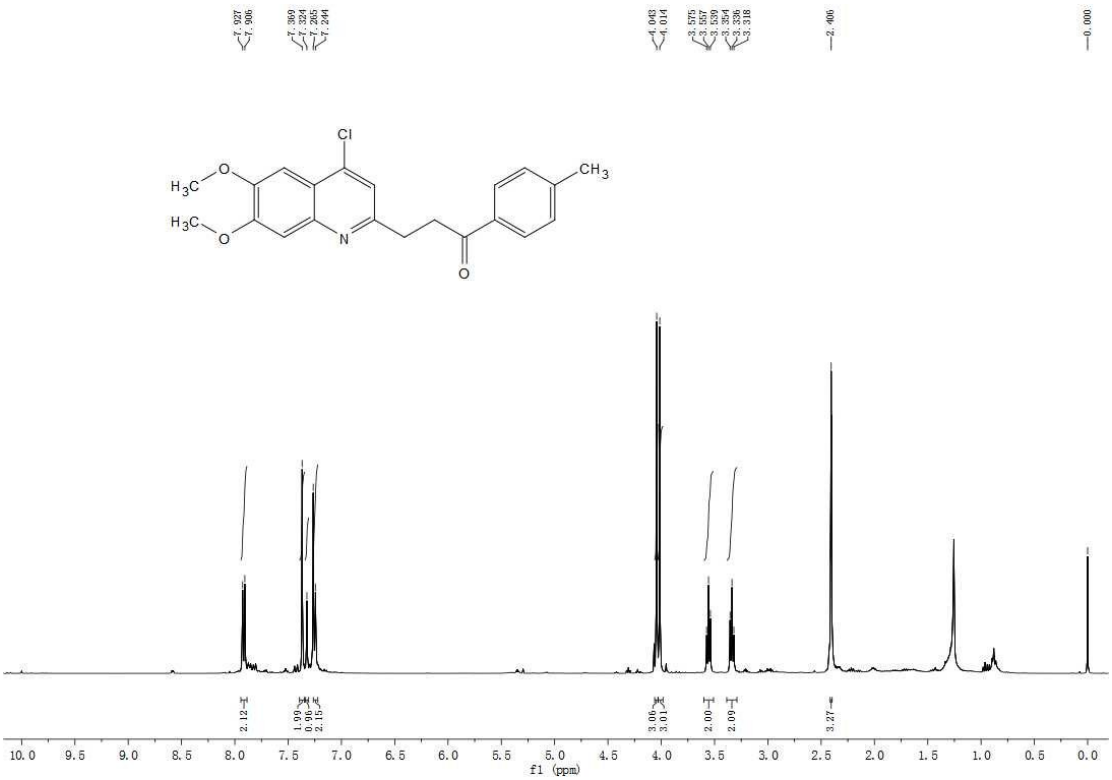
^{13}C NMR spectrum of 5c



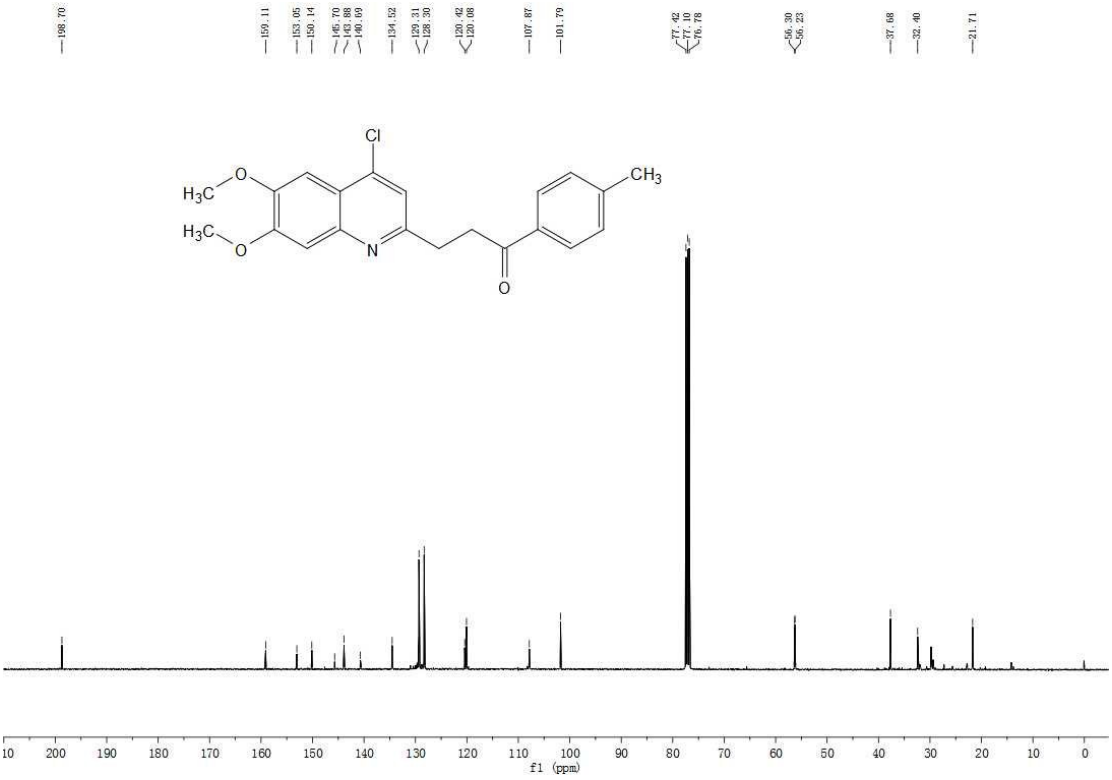
^{1}H NMR spectrum of 5d



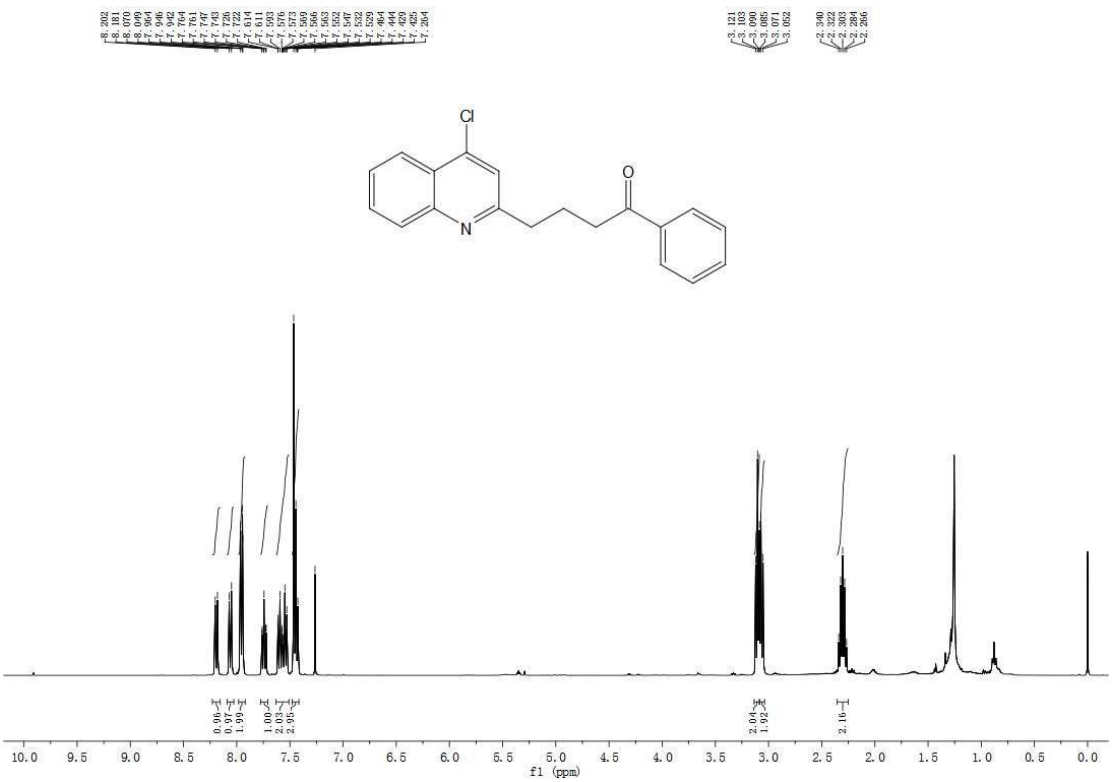
^{13}C NMR spectrum of 5d



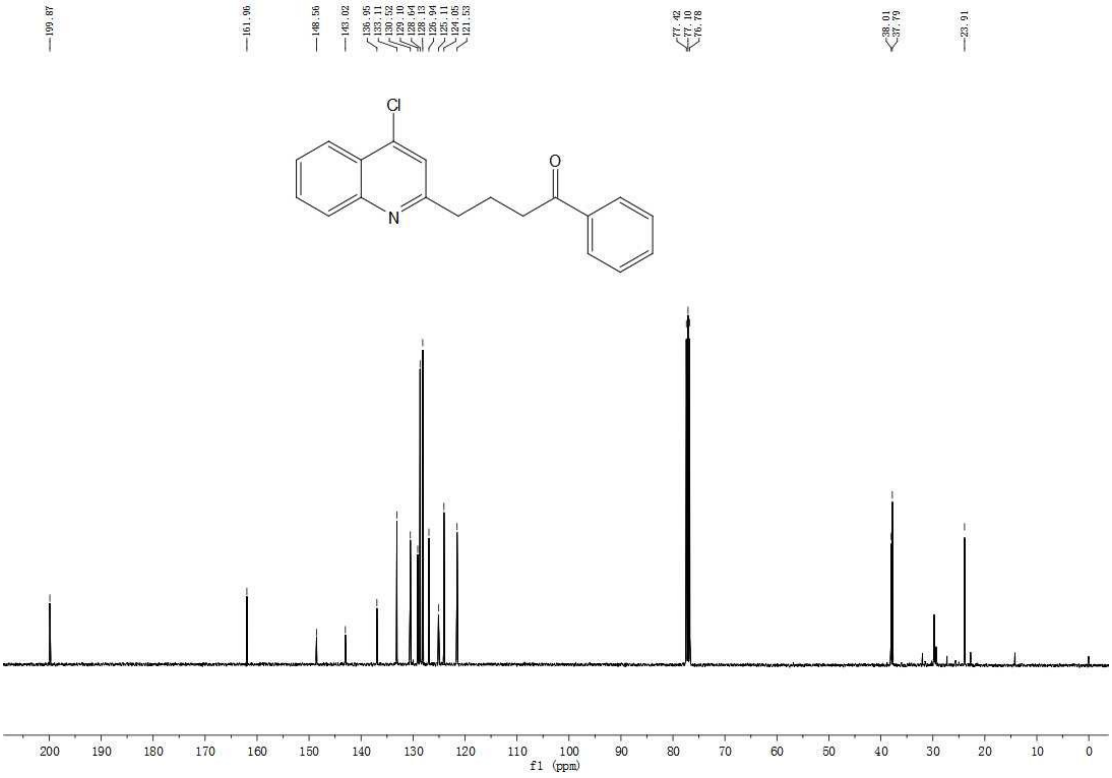
^{1}H NMR spectrum of 5e



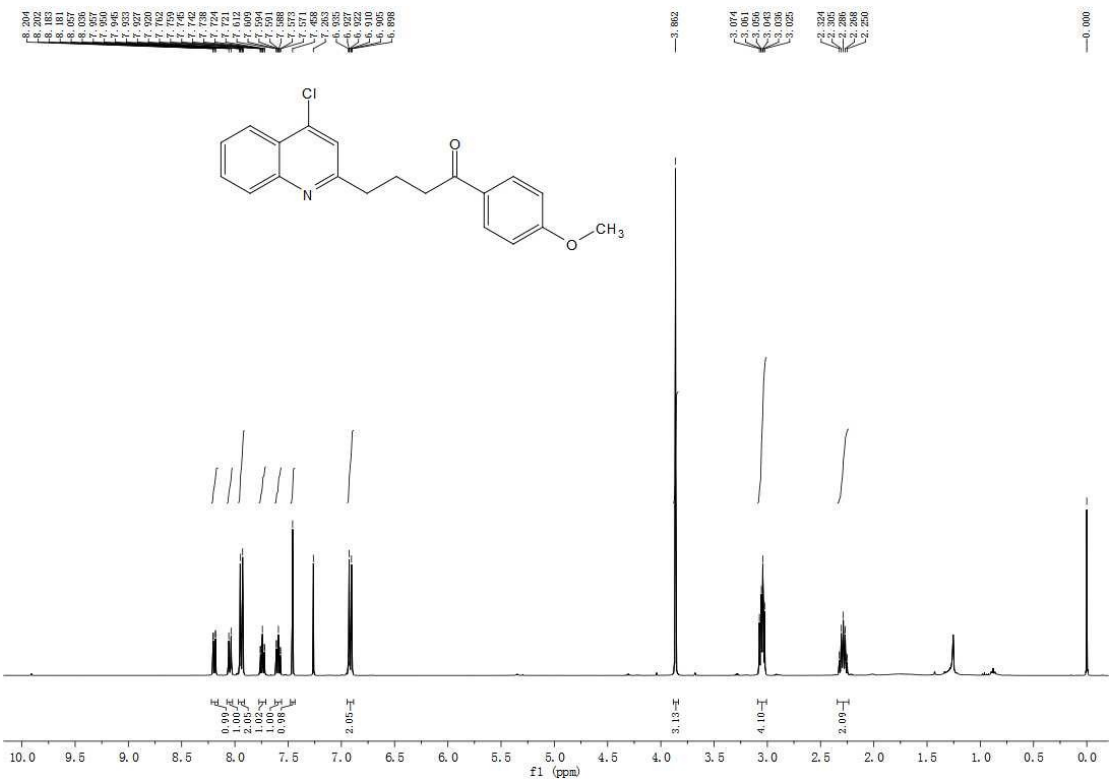
^{13}C NMR spectrum of 5e



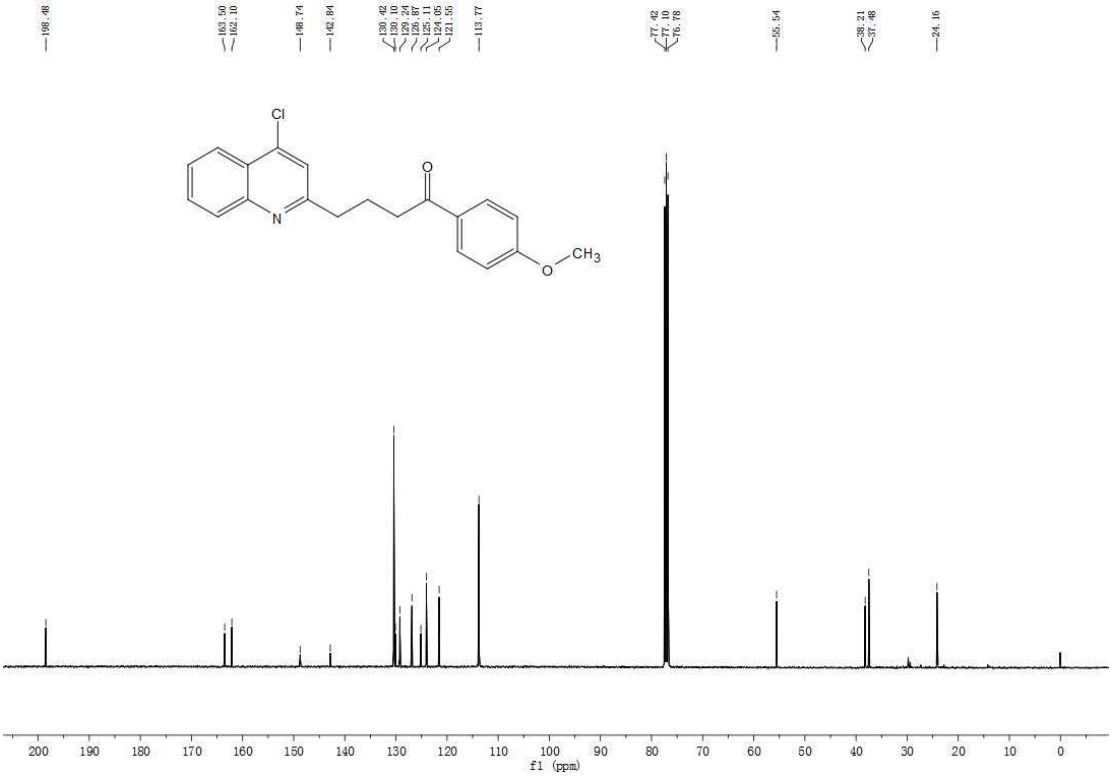
^{1}H NMR spectrum of 5f



^{13}C NMR spectrum of 5f



^{1}H NMR spectrum of 5g



^{13}C NMR spectrum of 5g