Electronic Supplementary Information

for

Electrochemical Minisci reaction via HAT-driven α-C(sp^3)−H functionalization of alcohols

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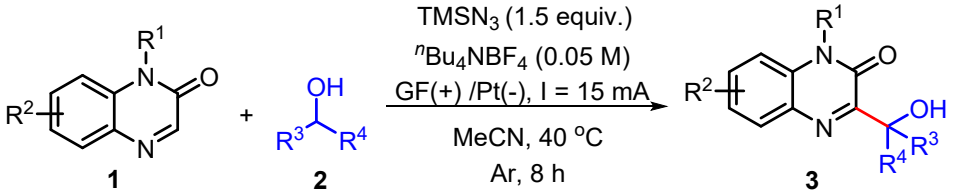
Contents

1 General information

All reagents were obtained from commercial suppliers and used without further purification. The reactions were monitored by TLC (thin layer chromatography). Column chromatography was performed using silica gel (300−400 mesh). The NMR spectra were recorded on a Bruker Avance 400 spectrometer at 400 MHz (^{1}H) and 100 MHz (^{13}C) in $CDCl_3$ or DMSO-d_6 using tetramethylsilane as the internal standard. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, m = multiplet, q = quartet. High-resolution mass spectra were obtained with an AB Triple 5600 mass spectrometer by ESI on a TOF mass analyzer. Melting points are uncorrected.

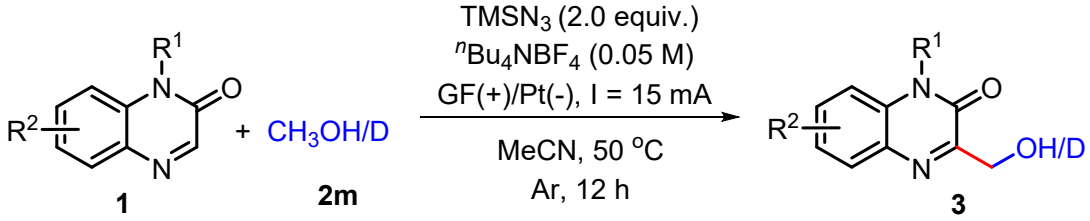
2 Experimental procedures

2.1 The general procedure for electrochemical hydrohydroxyalkylation reaction of 3a-3ff.



To an undivided three-necked flask (25 mL) were added quinoxalin-2(1*H*)-ones **1** (0.5 mmol), alcohols **2** (1.0 mL), $TMSN_3$ (86.4 mg, 99.0 μL, 0.75 mmol), $^n Bu_4NBF_4$ (0.5 mmol, 164.6 mg) and CH_3CN (10 mL). The flask was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (15 mA) under Ar at 40 °C for 8 h. After the reaction was completed, the mixture was diluted with water (20 mL) and then extracted by CH_2Cl_2 (30 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. The pure product was obtained by silica gel chromatography using petroleum ether/ethyl acetate (10:1, v/v) as the eluent.

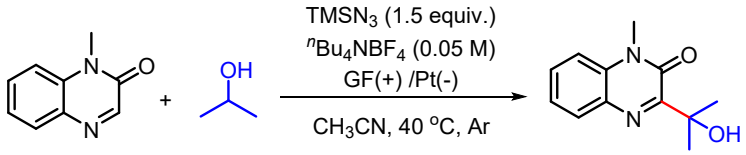
2.2 The general procedure for electrochemical hydrohydroxyalkylation reaction of 3gg–3pp.



To an undivided three-necked flask (25 mL) were added quinoxalin-2(1*H*)-one (**1**, 80.1 mg, 0.5

mmol), methanol (**2m**, 1.0 mL**)** or CH_3OD (**2m-*d*$_1$**, 1.0 mL), $TMSN_3$ (115.2 mg, 133.0 μL, 1.0 mmol), $^nBu_4NBF_4$ (164.6 mg, 0.5 mmol) and CH_3CN (10 mL). The flask was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (15 mA) under Ar at 50 °C for 12 h. After the reaction was completed, the mixture was diluted with water (20 mL) and then extracted by CH_2Cl_2 (30 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. The pure product was obtained by silica gel chromatography using petroleum ether/ethyl acetate (2:1, v/v) as the eluent.

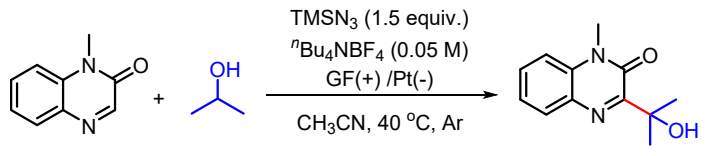
2.3 5 mmol-scale synthesis of 3a.



1a, 5 mmol **2a** (2 mL) **3a,** 0.97 g, 89%, c.e 37.2% (15 mA, 48 h)

To an undivided three-necked flask (100 mL) were added 1-methylquinoxalin-2(1*H*)-one (**1a**, 800.4 mg, 5 mmol), isopropanol (**2a**, 2.0 mL), $TMSN_3$ (864.2 mg, 0.99 mL, 7.5 mmol, 1.5 equiv.) and CH_3CN (50 mL). The flask was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (15 mA) under air at 40 °C for 48 h. After the reaction was completed, the mixture was diluted with water (50 mL) and then extracted by CH_2Cl_2 (80 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. The pure product **3a** was obtained by silica gel chromatography using petroleum ether/ethyl acetate (4:1, v/v) as the eluent.

2.4 20 mmol-scale synthesis of 3a.

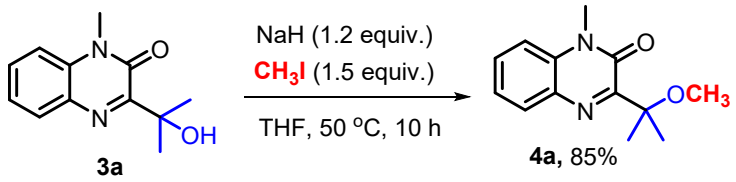


1a, 20 mmol **2a** (8 mL) **3a,** 3.53 g, 81%, c.e 59.6% (25 mA, 72 h)

To an undivided three-necked flask (250 mL) were added 1-methylquinoxalin-2(1*H*)-one (**1a**, 3.20 g, 20 mmol), isopropanol (**2a**, 8.0 mL), $TMSN_3$ (3.46 g, 3.96 mL, 30.0 mmol, 1.5 equiv.) and CH_3CN (100 mL). The flask was equipped with graphite felt as anode and platinum plate

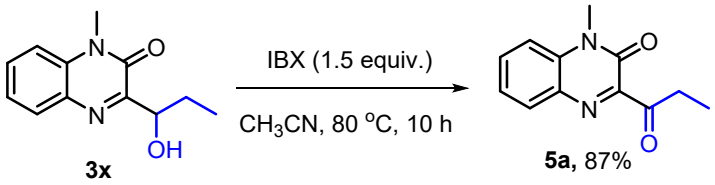
electrode (10 mm × 15 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (25 mA) under air at 40 °C for 72 h. After the reaction was completed, the mixture was diluted with water (50 mL) and then extracted by CH_2Cl_2 (80 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo and the crude product was obtained. The pure product **3a** was obtained by silica gel chromatography using petroleum ether/ethyl acetate (3:1, v/v) as the eluent.

2.5 The etherification of 3a.



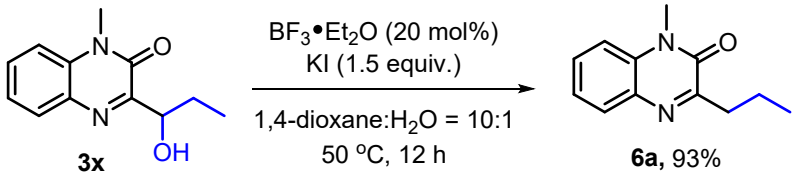
To a dry Schlenk tube (25 mL) were added 3-(2-hydroxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (**3a**, 109.1 mg, 0.5 mmol), NaH (14.4 mg, 0.6 mmol), tetrahydrofuran (5 mL) and CH_3I (106.5 mg, 47 μL, 0.75 mmol). The resulting solution was stirred at 50 °C for 10 h. After completion of the reaction, the reaction mixture was diluted with H_2O (15 mL) and CH_2Cl_2 (15 mL). The organic layer was collected, dried over anhydrous $MgSO_4$, and evaporated under vacuum. The residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (4:1, v/v) as the eluent to afford the desired product **4a** in 85% yield.

2.6 The oxidation of 3x.



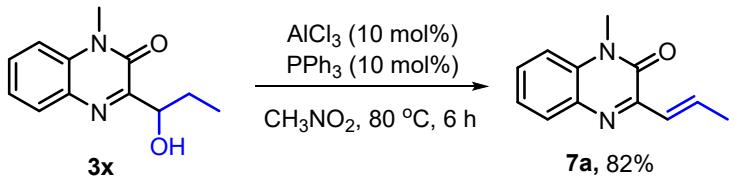
To a dry Schlenk tube (25 mL) were added 3-(1-hydroxypropyl)-1-methylquinoxalin-2(1*H*)-one (**3x**, 208.1 mg, 1.0 mmol), IBX (420.2 mg, 1.5 mmol) and CH_3CN (5 mL). The resulting solution was stirred at 80 °C for 10 h. After completion of the reaction, the reaction mixture was diluted with H_2O (15 mL) and CH_2Cl_2 (15 mL). The organic layer was collected, dried over anhydrous $MgSO_4$, and evaporated under vacuum. The residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (4:1, v/v) as the eluent to afford the desired product **5a** in 87% yield.

2.7 The reduction of 3x.



To a dry Schlenk tube (25 mL) were added 3-(1-hydroxypropyl)-1-methylquinoxalin-2(1*H*)-one (**3x**, 109.1 mg, 0.5 mmol), KI (124.5 mg, 0.75 mmol), $BF_3{\cdot}Et_2O$ (14.2 mg, 13 μL, 0.1 mmol), 1,4-dioxane (5 mL) and H_2O (0.5 mL). The resulting solution was stirred at 50 °C for 12 h. After completion of the reaction, the reaction mixture was diluted with H_2O (15 mL) and CH_2Cl_2 (15 mL). The organic layer was collected, dried over anhydrous $MgSO_4$, and evaporated under vacuum. The residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (4:1, v/v) as the eluent to afford the desired product **6a** in 93% yield.

2.8 Elimination of H_2O from 3x.



To a dry Schlenk tube (25 mL) were added 3-(1-hydroxypropyl)-1-methylquinoxalin-2(1*H*)-one (**3x**, 65.4 mg, 0.3 mmol), $AlCl_3$ (4.4 mg, 0.03 mmol), PPh_3 (8.7 mg, 0.03 mmol) and CH_3NO_2 (5 mL). The resulting solution was stirred at 80 °C for 6 h. After completion of the reaction, the reaction mixture was diluted with H_2O (15 mL) and CH_2Cl_2 (15 mL). The organic layer was collected, dried over anhydrous $MgSO_4$, and evaporated under vacuum. The residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (4:1, v/v) as the eluent to afford the desired product **7a** in 82%.

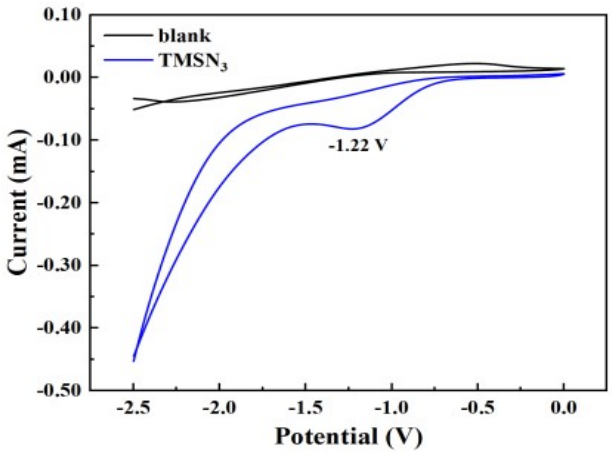
3 Mechanistic investigations

3.1 Cyclic voltammetry studies.

Cyclic voltammetry (CV) experiments were performed to detect the oxidation potential of two substrates involving the process. As shown in Figure S1-a, a reduction peak of $TMSN_3$ can be observed at -1.22 V vs SCE (green curve), which reveals that $TMSN_3$ may be reduced at cathode. By using $^nBu_4NN_3$ as the azide anion source, an oxidation peak at 1.02 V vs SCE can be found, which indicated that the azide anion is preferentially oxidized than 1-methylquinoxalin-2(1*H*)-one (**1a**, 2.29 V vs SCE, red curve) and $TMSN_3$ (2.36 V vs SCE, blue curve) in this electrochemical

reaction process (Figure S1-b).

(a)



(b)

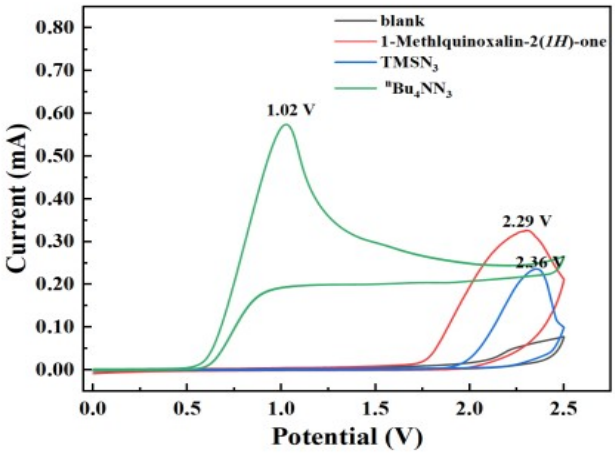
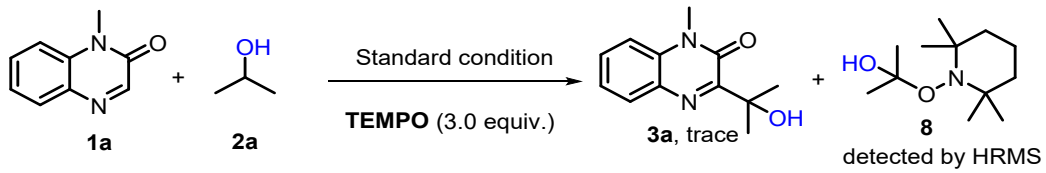


Figure S1 CV scans (scan rate 100 mv·s^{-1}) of substrates: (a) Blank (^{n}Bu$_4$NBF$_4$ (0.01 M) in MeCN); $TMSN_3$ (0.02 M, blue curve). (b) Blank (^{n}Bu$_4$NBF$_4$ (0.02 M) in MeCN); 1-Methylquinoxalin-2(1*H*)-one (**1a**, 0.02 M, red curve); $TMSN_3$ (0.02 M, blue curve); ^{n}Bu$_4$NN$_3$ (0.02 M, green curve).

3.2 Radical capture experiment.

3.2.1 Reaction with TEMPO.



To an undivided three-necked flask (25 mL) were added 1-methylquinoxalin-2(1*H*)-one (**1a**, 80.1 mg, 0.5 mmol), isopropyl alcohol (**2a**, 1.0 mL), $TMSN_3$ (86.4 mg, 99.0 μL, 0.75 mmol), ^{n}Bu$_4$NBF$_4$ (164.6 mg, 0.5 mmol), TEMPO (234.4 mg, 1.5 mmol), and CH_3CN (10 mL). The flask

was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (15 mA) under Ar at 40 °C for 8 h. After the reaction was stopped, no desired product **3a** was detected by TLC, indicating that the reaction was completely inhibited. Meanwhile, an adduct product **8** of **2a** with TEMPO was observed through the HRMS analysis from the reaction solution.

8, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_{12}H_{26}NO_2$, 216.1963; found 216.1968.

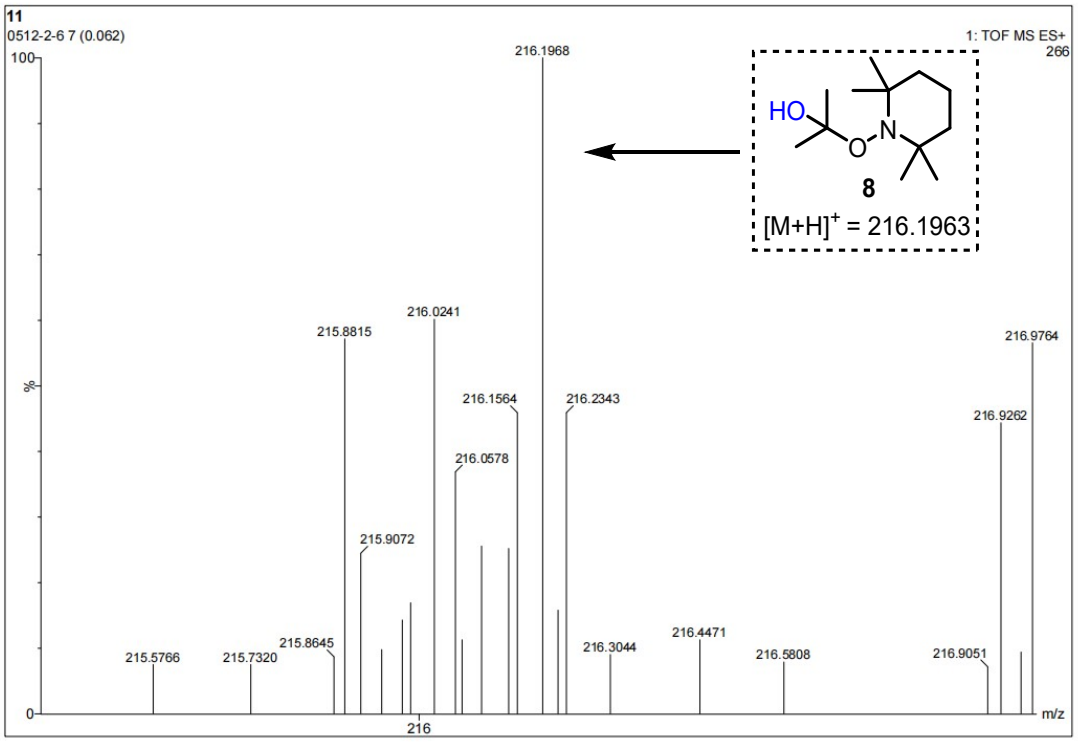
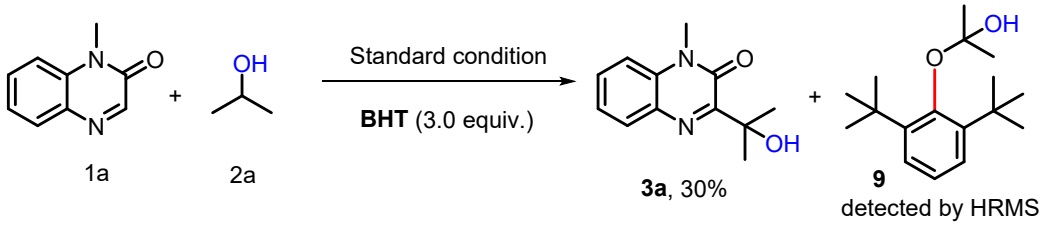


Figure S2 HRMS analysis of the adduct product **8**.

3.2.2 Reaction with BHT.



To an undivided three-necked flask (25 mL) were added 1-methylquinoxalin-2(1*H*)-one (**1a**, 80.1 mg, 0.5 mmol), isopropyl alcohol (**2a**, 1.0 mL), $TMSN_3$ (86.4 mg, 99.0 μL, 0.75 mmol), $^nBu_4NBF_4$ (164.6 mg, 0.5 mmol), 2,6-di-*t*-butyl-4-methylphenol (BHT, 330.3 mg, 1.5 mmol) and CH_3CN (10 mL). The flask was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant

current (15 mA) under Ar at 40 °C for 8 h. After the reaction was stopped, only a small amount of target product **3a** was observed by TLC, indicating that the reaction was inhibited. Meanwhile, an adduct product **9** was observed through the HRMS analysis from the reaction solution.

9, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_{17}H_{29}O_2$, 265.2161; found 265.2167.

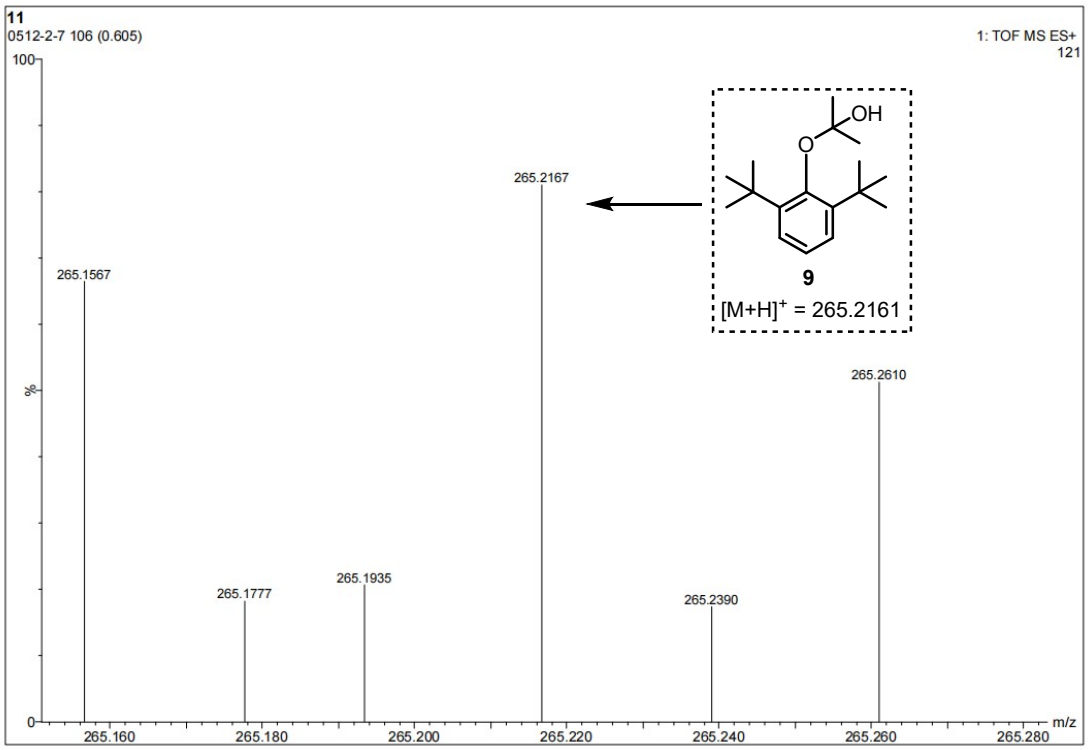
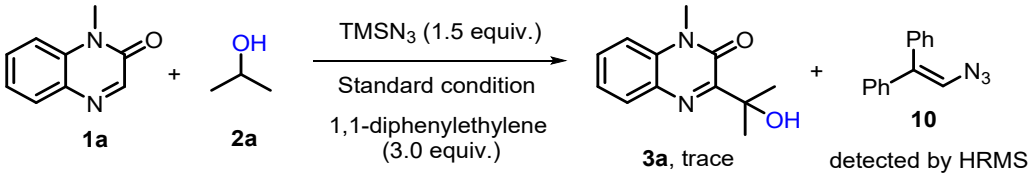


Figure S3 HRMS analysis of the adduct product **9**.

3.2.3 Reaction with 1,1-diphenylethylene.



To an undivided three-necked flask (25 mL) were added 1-methylquinoxalin-2(1*H*)-one (**1a**, 80.1 mg, 0.5 mmol), isopropyl alcohol (**2a**, 1.0 mL), $TMSN_3$ (86.4 mg, 99.0 μL, 0.75 mmol), $^nBu_4NBF_4$ (164.6 mg 0.5 mmol), 1,1-diphenylethylene (270.4 mg, 265 μL, 1.5 mmol), and CH_3CN (10 mL). The flask was equipped with graphite felt as anode and platinum plate electrode (10 mm × 10 mm) as cathode. The reaction mixture was stirred and electrolyzed at a constant current (15 mA) under Ar at 40 °C for 8 h. After the reaction was stopped, no desired product **3a** was detected by TLC, indicating that the reaction was completely inhibited. Meanwhile, some intermediates, such as **N_3-1,1-diphenylethylene** (**10**), **TMS-1,1-diphenylethylene** (**11**), **N_3-2a**,

and **TMS-2a**, were observed through the HRMS analysis from the reaction solution.

10, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_{14}H_{12}N_3$, 222.1025; found 222.1028.

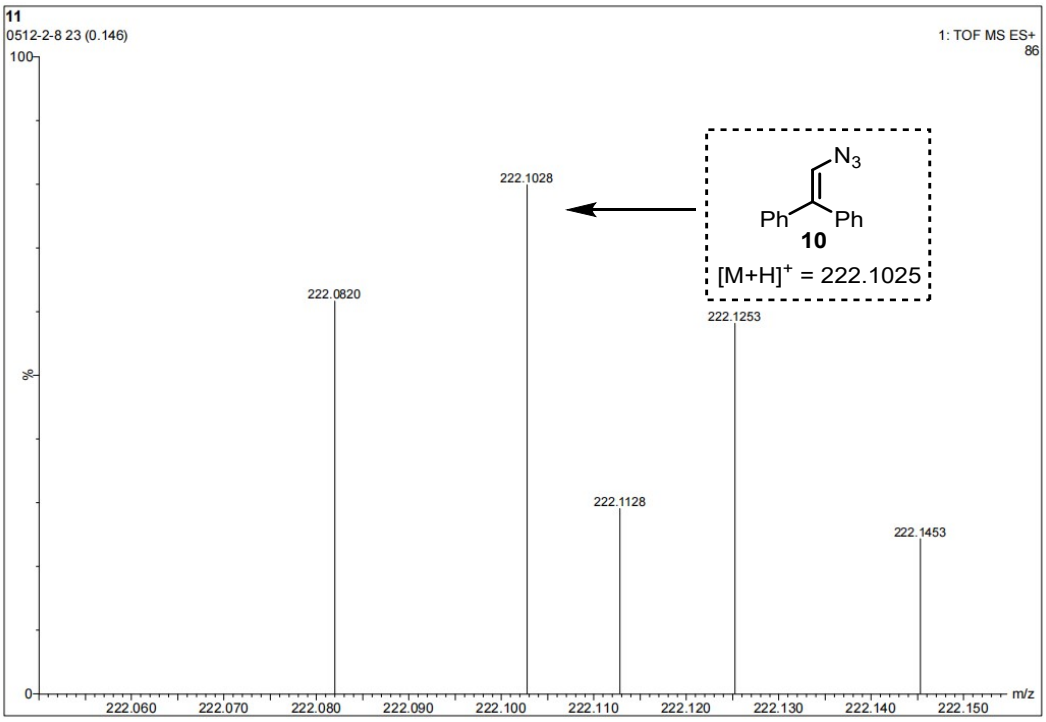


Figure S4 HRMS analysis of the adduct product **10**.

11, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_{17}H_{21}Si^+$, 253.1407; found 253.1409.

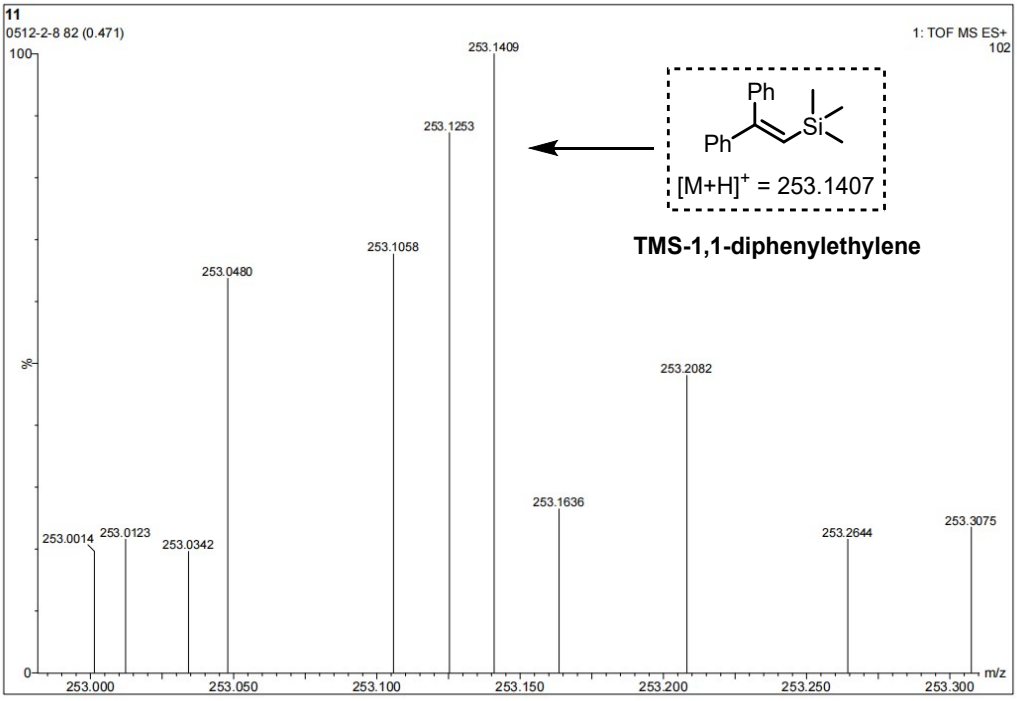


Figure S5 HRMS analysis of the adduct product **11**.

N_3-2a, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_3H_8N_3O$, 102.0661; found 102.0663.

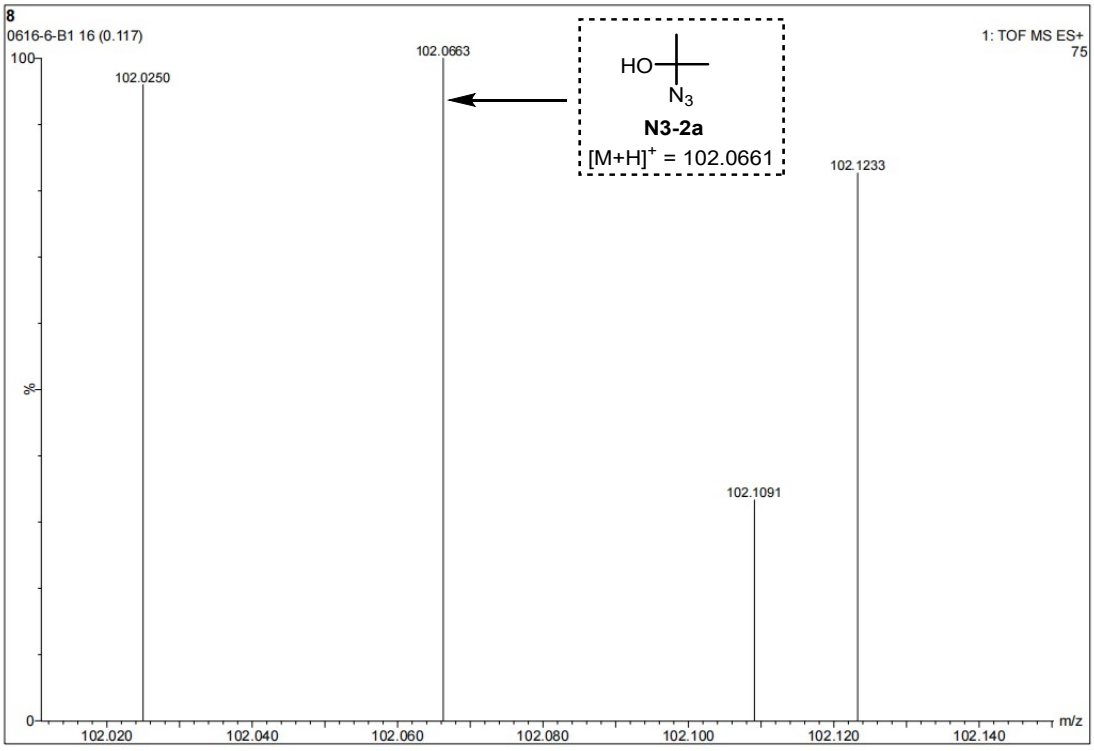


Figure S6 HRMS analysis of the adduct product **N_3-2a**.

TMS-2a, HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for $C_6H_{17}OSi$, 133.1048; found 133.1042.

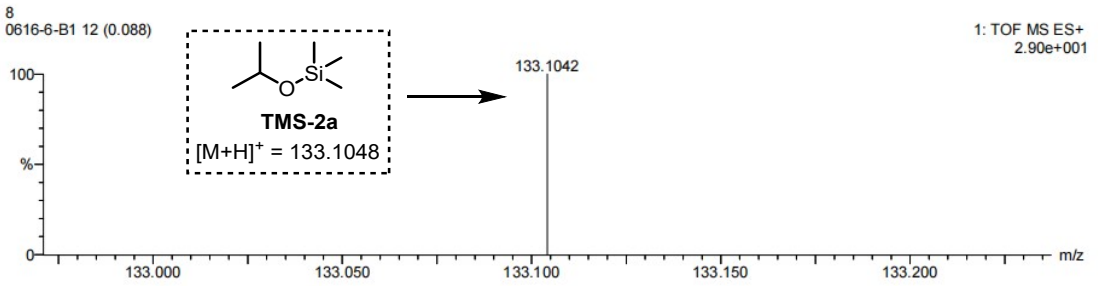
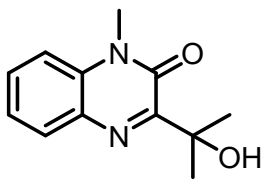


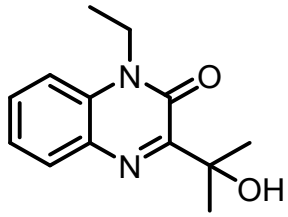
Figure S7 HRMS analysis of the adduct product **TMS-2a**.

4 Experimental data for the products 3, 4a, 5a, 6a and 7a

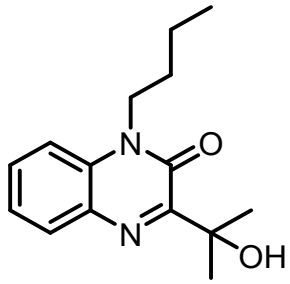


3-(2-Hydroxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (3a).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 102.5 mg, total 94%; mp 118−120 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.85–7.83 (m, 1H), 7.68–7.64 (m, 1H), 7.61–7.58 (m, 1H), 7.43–7.39 (m, 1H),

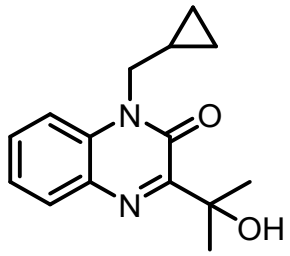
5.37 (s, 1H), 3.66 (s, 3H), 1.56 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 161.9, 153.8, 133.7, 131.3, 131.0, 129.7, 124.2, 115.3, 73.5, 29.4, 28.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{15}N_2O_2$, 219.1128; found 219.1125.



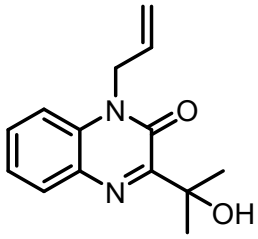
1-Ethyl-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3b). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 106.7 mg, total 92%; mp 101−103 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.86 (d, J = 8.0 Hz, 1H), 7.67–7.66 (m, 2H), 7.44–7.40 (m, 1H), 5.38 (s, 1H), 4.33–4.27(m, 2H), 1.56 (s, 6H), 1.27–1.24 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 161.9, 153.3, 132.5, 131.6, 131.1, 130.1, 124.2, 115.0, 73.6, 37.3, 28.1, 12.8; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1285; found 233.1295.



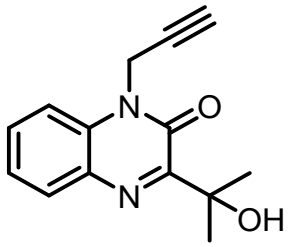
1-Butyl-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3c). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 105.3 mg, total 81%; mp 82−84 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.89–7.87 (m, 1H), 7.60–7.56 (m, 1H), 7.39–7.34 (m, 2H), 5.55 (s, 1H), 4.29–4.25 (m, 2H), 1.80–1.74 (m, 2H), 1.70 (s, 6H), 1.56–1.46 (m, 2H), 1.04–1.00 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.7, 153.8, 132.5, 131.9, 130.4, 130.4, 123.8, 113.7, 73.8, 42.1, 29.3, 27.6, 20.3, 13.8; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{15}H_{21}N_2O_2^+$, 261.1598; found 261.1608.



1-(Cyclopropylmethyl)-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3d).[2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 117.4 mg, total 91%; mp 93−95 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91–7.88 (m, 1H), 7.62–7.57 (m, 1H), 7.49–7.46 (m, 1H), 7.40–7.36 (m, 1H), 5.57 (s, 1H), 4.24 (d, J = 7.0 Hz, 2H), 1.70 (s, 6H), 1.32–1.27 (m, 1H), 0.59–0.57 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.9, 154.1, 132.7, 131.8, 130.3, 123.8, 114.0, 73.9, 46.0, 27.5, 9.7, 4.2; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{15}H_{19}N_2O_2^+$, 259.1441; found 259.1449.

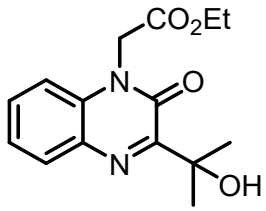


1-Allyl-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3e).[2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 114.7 mg, total 94%; mp 89−91 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90–7.88 (m, 1H), 7.58–7.54 (m, 1H), 7.40–7.32 (m, 2H), 6.00–5.93 (m, 1H), 5.47 (s, 1H), 5.31–5.28 (m, 1H), 5.21–5.16 (m, 1H), 4.94–4.92 (m, 2H), 1.71 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.8, 153.6, 132.6, 131.8, 130.4, 130.2, 124.0, 118.3, 114.3, 73.8, 44.4, 27.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{14}H_{17}N_2O_2^+$, 245.1284; found 245.1291.

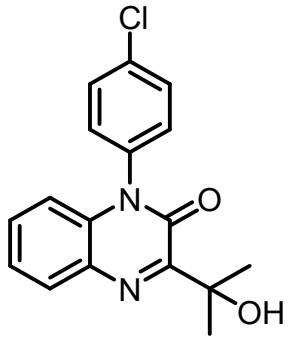


3-(2-Hydroxypropan-2-yl)-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one (3f).[2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 112.5 mg, total 93%; mp 101−103 °C; ^{1}H NMR (400 MHz,

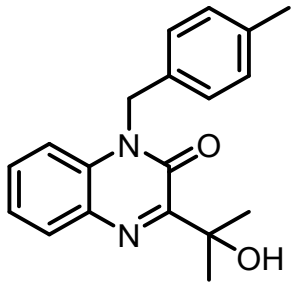
$CDCl_3$) δ (ppm) 7.90–7.88 (m, 1H), 7.65–7.61 (m, 1H), 7.52–7.50 (m, 1H), 7.43–7.39 (m, 1H), 5.33 (s, 1H), 5.07 (d, J = 2.5 Hz, 2H), 2.35–2.34 (m, 1H), 1.70 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.7, 153.0, 131.9, 131.7, 130.6, 130.2, 124.3, 114.2, 76.6, 73.8, 73.5, 31.4, 27.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{14}H_{15}N_2O_2^+$, 243.1127; found 243.1137.



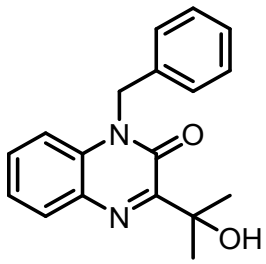
Ethyl 2-(3-(2-hydroxypropan-2-yl)-2-oxoquinoxalin-1(2*H*)-yl)acetate (3g). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 124.7 mg, total 86%; mp 91−93 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.89–7.86 (m, 1H), 7.56–7.51 (m, 1H), 7.39–7.35 (m, 1H), 7.12–7.10 (m, 1H), 5.32 (s, 1H), 5.03 (s, 2H), 4.28–4.22 (m, 2H), 1.68 (s, 6H), 1.29–1.26 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.9, 161.6, 153.5, 132.6, 131.6, 130.7, 130.3, 124.2, 113.2, 73.7, 62.2, 43.4, 27.5, 14.1; HRMS (ESI-TOF) m/z $[M+Na]^+$ calcd for: $C_{15}H_{18}N_2O_4Na$, 313.1164; found 313.1159.



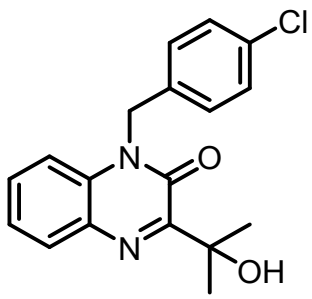
1-(4-Chlorophenyl)-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3h). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 130.3 mg, total 83%; mp 173−175 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.96–7.93 (m, 1H), 7.66–7.62 (m, 2H), 7.44–7.37 (m, 2H), 7.31–7.28 (m, 2H), 6.75–6.72 (m, 1H), 1.73 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 162.5, 153.7, 135.8, 133.9, 133.8, 131.5, 130.7, 130.3, 129.9, 129.7, 124.4, 115.3, 73.9, 27.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{17}H_{16}ClN_2O_2^+$, 315.0894; found 315.0903.



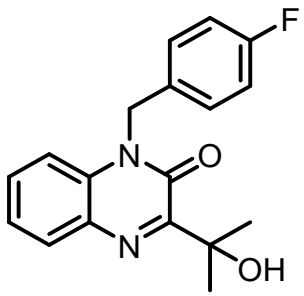
3-(2-Hydroxypropan-2-yl)-1-(4-methylbenzyl)quinoxalin-2(1*H*)-one (3i). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 138.6 mg, total 90%; mp 87−89 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91–7.88 (m, 1H), 7.49–7.45 (m, 1H), 7.36–7.32 (m, 2H), 7.18–7.13 (m, 4H), 5.49 (s, 2H), 5.43 (s, 1H), 2.32 (s, 3H), 1.77 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.9, 154.2, 137.6, 132.7, 132.0, 131.9, 130.5, 130.2, 129.7, 126.8, 124.0, 114.6, 74.0, 45.6, 27.7, 21.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{19}H_{21}N_2O_2^+$, 309.1597; found 309.1607.



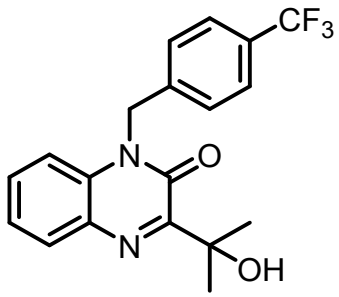
1-Benzyl-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3j).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 127.9 mg, total 87%; mp 85−87 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91–7.89 (m, 1H), 7.49–7.45 (m, 1H), 7.37–7.31 (m, 4H), 7.28–7.24 (m, 3H), 5.53 (s, 2H), 1.77 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.9, 154.2, 135.0, 132.7, 131.9, 130.5, 130.3, 129.0, 127.8, 126.8, 124.1, 114.5, 74.0, 45.8, 27.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{18}H_{19}N_2O_2$, 295.1440; found 295.1448.



1-(4-Chlorobenzyl)-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3k). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 157.4 mg, total 96%; mp 82−84 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.87–7.85 (m, 1H), 7.57–7.52 (m, 1H), 7.46 (d, J = 7.8 Hz, 1H), 7.38–7.34 (m, 3H), 7.30 (d, J = 8.6 Hz, 2H), 5.53 (s, 2H), 5.35 (s, 1H), 1.61 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 162.3, 153.8, 135.2, 132.8, 132.5, 131.7, 131.0, 130.1, 129.2, 129.1, 124.4, 115.5, 73.6, 44.7, 28.1; HRMS (ESI-TOF) m/z [M+H]$^+$ calcd for: $C_{18}H_{18}ClN_2O_2^+$, 329.1051; found 329.1055.

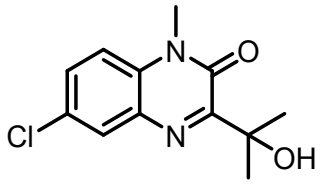


1-(4-Fluorobenzyl)-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3l). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 145.1 mg, total 93%; mp 86−88 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.88–7.86 (m, 1H), 7.49–7.44 (m, 1H), 7.35–7.31 (m, 1H), 7.28–7.21 (m, 3H), 7.01–6.96 (m, 2H), 5.49 (s, 1H), 5.46 (s, 2H), 1.74 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.4 (d, J = 245.1 Hz), 161.8, 154.1, 132.5, 131.9, 130.8 (d, J = 3.2 Hz), 130.6, 130.3, 128.8 (d, J = 8.1 Hz), 124.2, 116.1 (d, J = 21.5 Hz), 114.3, 73.9, 45.1, 27.6; HRMS (ESI-TOF) m/z [M+H]$^+$ calcd for: $C_{18}H_{18}FN_2O_2^+$, 313.1346; found 313.1352.

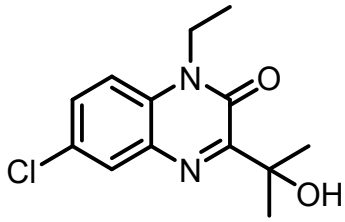


3-(2-Hydroxypropan-2-yl)-1-(4-(trifluoromethyl)benzyl)quinoxalin-2(1*H*)-one (3m). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 166.5 mg, total 92%; mp 103−105 °C;

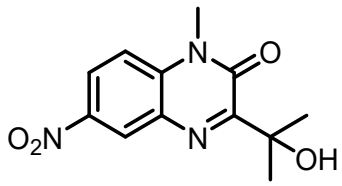
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.94–7.92 (m, 1H), 7.61 (d, J = 8.2 Hz, 2H), 7.52–7.48 (m, 1H), 7.41–7.36 (m, 3H), 7.23–7.21 (m, 1H), 5.59 (s, 2H), 1.75 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.9, 154.0, 139.0, 132.5, 131.9, 130.7, 130.5, 130.4 (q, J = 32.5 Hz), 127.1, 126.1 (q, J = 3.8 Hz), 125.2 (q, J = 270.4 Hz), 124.3, 114.1, 73.9, 45.4, 27.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{19}H_{18}F_3N_2O_2^+$, 363.1314; found 363.1317.



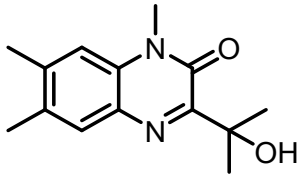
6-Chloro-3-(2-hydroxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (3n).[2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 109.6 mg, total 91%; mp 214−216 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.87 (d, J = 2.4 Hz, 1H), 7.55–7.53 (m, 1H), 7.30–7.28 (m, 1H), 5.31 (s, 1H), 3.71 (s, 3H), 1.68 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.2, 153.7, 132.1, 132.0, 130.5, 129.4, 129.3, 114.9, 74.0, 29.2, 27.5. HRMS (ESI-TOF) m/z $[M+Na]^+$ calcd for: $C_{12}H_{13}ClN_2O_2Na$, 275.0563; found 275.0570.



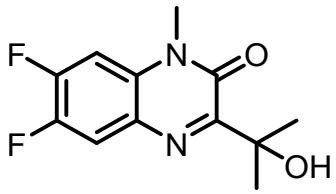
6-Chloro-1-ethyl-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3o). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 115.7 mg, total 87%; mp 184−186 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90 (d, J = 2.4 Hz, 1H), 7.56–7.53 (m, 1H), 7.31 (d, J = 9.0 Hz, 1H), 5.39 (s, 1H), 4.35–7.30 (m, 2H), 1.69 (s, 6H), 1.42–1.39 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.1, 153.3, 132.5, 130.9, 130.5, 129.7, 129.1, 114.7, 74.0, 37.6, 27.5, 12.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{16}ClN_2O_2^+$, 267.0894; found 267.0900.



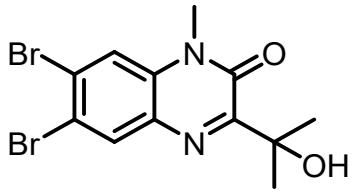
3-(2-Hydroxypropan-2-yl)-1-methyl-6-nitroquinoxalin-2(1*H*)-one (3p).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 103.9 mg, total 79%; mp 174−176 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.58 (d, J = 2.5 Hz, 1H), 7.35 (d, J = 8.9 Hz, 1H), 7.26–7.24 (m, 1H), 5.40 (s, 1H), 3.73 (s, 3H), 1.70 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.2, 153.7, 136.4, 132.4, 130.6, 121.8, 119.2, 115.1, 74.0, 29.2, 27.5. HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{14}N_3O_4$, 264.0978; found 264.0980.



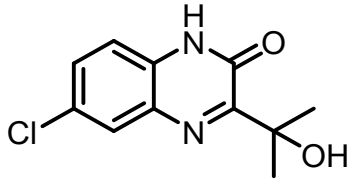
3-(2-Hydroxypropan-2-yl)-1,6,7-trimethylquinoxalin-2(1*H*)-one (3q).[1,2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 103.3 mg, total 84%; mp 132−134 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.64 (s, 1H), 7.12 (s, 1H), 5.59 (s, 1H), 3.71 (s, 3H), 2.45 (s, 3H), 2.38 (s, 3H), 1.69 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 154.1, 140.4, 133.0, 131.4, 130.1, 130.0, 114.2, 73.7, 28.8, 27.6, 20.6, 19.2; HRMS (ESI-TOF) m/z $[M+Na]^+$ calcd for: $C_{14}H_{18}N_2O_2Na$, 269.1266; found 269.1268.



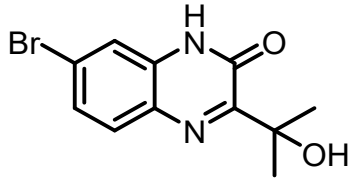
6,7-Difluoro-3-(2-hydroxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (3r). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 116.8 mg, total 92%; mp 145−147 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.71−7.66 (m, 1H), 7.19−7.14(m, 1H), 5.23 (s, 1H), 3.68 (s, 3H), 1.66 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 162.3 (d, J = 3.6 Hz), 153.7, 152.8 (dd, J = 252.5 Hz, 14.4 Hz), 148.1 (dd, J = 246.3 Hz, 14.0 Hz), 130.5 (d, J = 9.0 Hz), 127.9 (dd, J = 9.4, 2.9 Hz), 117.7 (dd, J = 18.0, 2.2 Hz), 102.5, 74.0, 29.5, 27.4. HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{13}F_2N_2O_2^+$, 255.0939; found 255.0944.



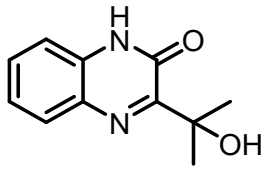
6,7-Dibromo-3-(2-hydroxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (3s). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 170.6 mg, total 91%; mp 172−174 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.13 (s, 1H), 7.62 (s, 1H), 3.69 (s, 3H), 1.68 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.5, 153.5, 134.0, 133.2, 131.3, 126.9, 119.2, 118.4, 74.1, 29.2, 27.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{13}Br_2N_2O_2^+$, 376.9317; found 376.9319.



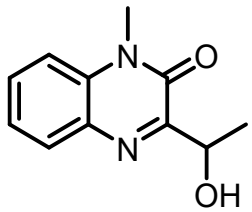
6-Chloro-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3t).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 71.4 mg, total 60%; mp 182−184 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 12.55 (s, 1H), 7.92 (d, J = 2.2 Hz, 1H), 7.57−7.54 (m, 1H), 7.33 (d, J = 8.7 Hz, 1H), 1.75 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.6, 155.8, 132.2, 131.0, 130.1, 129.7, 128.7, 116.6, 73.9, 27.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{12}ClN_2O_2^+$, 239.0582; found 239.0593.



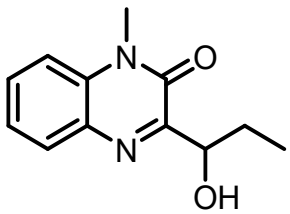
7-Bromo-3-(2-hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3u). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 91.7 mg, total 65%; mp 149−151 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 12.37 (s, 1H), 7.76 (d, J = 8.4 Hz, 1H), 7.54–7.50 (m, 2H), 1.76 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 162.6, 155.7, 132.1, 130.6, 130.5, 128.3, 124.7, 118.2, 73.8, 27.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{12}BrN_2O_2^+$, 283.0078; found 283.0082.



3-(2-Hydroxypropan-2-yl)quinoxalin-2(1*H*)-one (3v).[1,2] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 71.4 mg, total 70%; mp 177−179 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 12.74 (s, 1H), 7.92−7.90 (m, 1H), 7.62–7.58 (m, 1H), 7.45–7.39 (m, 2H), 1.78 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 162.2, 156.1, 131.7, 131.3, 130.7, 129.2, 124.8, 115.6, 73.7, 27.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{13}N_2O_2^+$, 205.0971; found 205.0978.

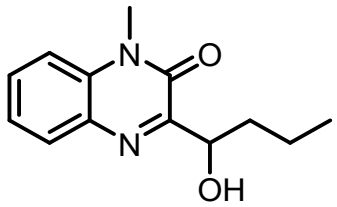


3-(1-Hydroxyethyl)-1-methylquinoxalin-2(1*H*)-one (3w). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 3:1); 88.7 mg, total 87%; mp 97−99 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.84–7.82 (m, 1H), 7.65–7.61 (m, 1H), 7.58–7.55 (m, 1H), 7.41–7.37 (m, 1H), 5.11–5.07 (m, 1H), 5.06–5.03 (m, 1H), 3.63 (s, 3H), 1.42–1.38 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 161.7, 153.8, 133.6, 132.0, 130.7, 129.5, 124.0, 115.2, 65.7, 29.3, 21.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{13}N_2O_2^+$, 205.0971; found 205.0981.

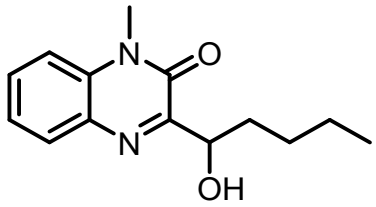


3-(1-Hydroxypropyl)-1-methylquinoxalin-2(1*H*)-one (3x).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 5:1); 91.6 mg, total 84%; mp 127−129 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.84 (d, J = 7.8 Hz, 1H), 7.66–7.56 (m, 2H), 7.41–7.38 (m, 1H), 4.98 (d, J = 6.5 Hz, 1H), 4.87–4.83 (m, 1H), 3.64 (s, 3H), 1.93–1.83 (m, 1H), 1.70–1.59 (m, 1H), 0.94–0.90 (m, 3H); ^{13}C NMR

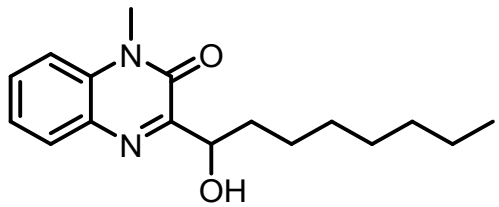
(100 MHz, DMSO-d_6) δ (ppm) 161.1, 153.9, 133.5, 132.0, 130.7, 129.5, 123.9, 115.2, 70.7, 29.3, 28.1, 10.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{15}N_2O_2^+$, 219.1127; found 219.1125.



3-(1-Hydroxybutyl)-1-methylquinoxalin-2(1*H*)-one (3y). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 5:1); 98.6 mg, total 85%; mp 111−113 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.83 (d, J = 7.8 Hz, 1H), 7.65–7.56 (m, 2H), 7.41–7.38 (m, 1H), 4.97 (d, J = 6.5 Hz, 1H), 4.94–4.91 (m, 1H), 3.64 (s, 3H), 1.83–1.75 (m, 1H), 1.65–1.56 (m, 1H), 1.46–1.37 (m, 2H), 0.92–0.88 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 161.4, 153.8, 133.5, 132.0, 130.7, 129.5, 124.0, 115.2, 69.1, 37.2, 29.3, 19.1, 14.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1285; found 233.1295.

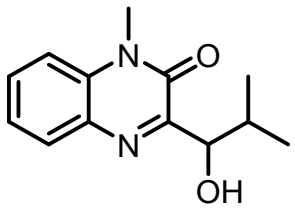


3-(1-Hydroxypentyl)-1-methylquinoxalin-2(1*H*)-one (3z).[1] According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 5:1); 93.5 mg, total 76%; mp 105−107 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91–7.88 (m, 1H), 7.62–7.58 (m, 1H), 7.42–7.36 (m, 2H), 5.02–5.00 (m, 1H), 4.17 (s, 1H), 3.74 (s, 3H), 2.11–2.02 (m, 1H), 1.72–1.65 (m, 1H), 1.57–1.48 (m, 2H), 1.43–1.35 (m, 2H), 0.94–0.91 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.2, 153.9, 133.2, 131.8, 130.4, 129.8, 123.9, 113.8, 71.2, 35.3, 28.9, 27.7, 22.6, 14.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{14}H_{19}N_2O_2^+$, 247.1440; found 247.1442.

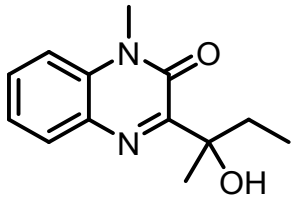


3-(1-Hydroxyoctyl)-1-methylquinoxalin-2(1*H*)-one (3aa). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum

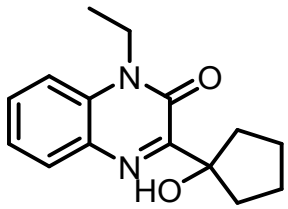
ether/EtOAc = 5:1); 122.4 mg, total 85%; mp 102−104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90 (d, J = 7.8 Hz, 1H), 7.62–7.59 (m, 1H), 7.42–7.36 (m, 2H), 5.02–5.00 (m, 1H), 4.15 (s, 1H), 3.74 (s, 3H), 2.10–2.02 (m, 1H), 1.73–1.64 (m, 1H), 1.55–1.51 (m, 2H), 1.39–1.28 (m, 8H), 0.89–0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.2, 153.9, 133.2, 131.8, 130.4, 129.8, 123.9, 113.8, 71.3, 35.6, 31.8, 29.5, 29.3, 28.9, 25.6, 22.7, 14.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{17}H_{25}N_2O_2^+$, 289.1910; found 289.1912.



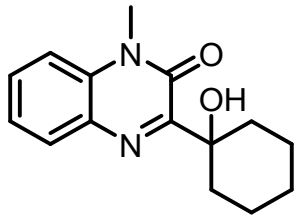
1-(1-Hydroxy-2-methylpropyl)-1-methylquinoxalin-2(1*H*)-one (3bb). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 5:1); 88.2 mg, total 76%; mp 111−113 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.86–7.84 (m, 1H), 7.66–7.57 (m, 2H), 7.42–7.38 (m, 1H), 4.88 (d, J = 6.7 Hz, 1H), 4.71–4.68 (m, 1H), 3.64 (s, 3H), 2.29–2.25 (m, 1H), 0.90 (d, J = 6.8 Hz, 3H), 0.84 (d, J = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 160.8, 154.0, 133.5, 131.9, 130.8, 129.6, 124.0, 115.3, 74.2, 31.7, 29.4, 20.2, 17.6; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1284; found 233.1291.



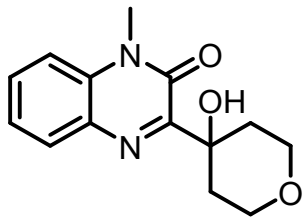
3-(2-Hydroxybutan-2-yl)-1-methylquinoxalin-2(1*H*)-one (3cc). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 83.5 mg, total 72%; mp 102−104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.92–7.90 (m, 1H), 7.63–7.59 (m, 1H), 7.42–7.36 (m, 2H), 5.42 (s, 1H), 3.74 (s, 3H), 2.35–2.26 (m, 1H), 2.04–1.95 (m, 1H), 1.67 (s, 3H), 0.86–0.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.5, 153.8, 133.5, 131.4, 130.5, 130.0, 123.9, 113.7, 76.0, 32.6, 29.0, 25.7, 8.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1284; found 233.1291.



1-Ethyl-3-(1-hydroxycyclopentyl)quinoxalin-2(1*H*)-one (3dd). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 121.3 mg, total 94%; mp 104−106 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90–7.87 (m, 1H), 7.60–7.56 (m, 1H), 7.39–7.35 (m, 2H), 5.29 (s, 1H), 4.37–4.32 (m, 2H), 2.46 (d, J = 5.8 Hz, 2H), 2.03–1.85 (m, 6H), 1.42–1.39 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.7, 153.9, 132.1, 132.0, 130.4, 130.3, 123.8, 113.5, 84.2, 38.9, 37.3, 25.1, 12.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{15}H_{19}N_2O_2^+$, 259.1440; found 259.1443.

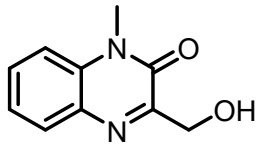


3-(1-Hydroxycyclohexyl)-1-methylquinoxalin-2(1*H*)-one (3ee). According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 116.1 mg, total 90%; mp 123−125 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.85–7.83 (m, 1H), 7.68–7.59 (m, 2H), 7.44–7.40 (m, 1H), 3.66 (s, 3H), 2.14–2.06 (m, 2H), 1.79–1.54 (m, 7H), 1.29–1.22 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 161.7, 154.1, 133.4, 131.5, 131.0, 129.8, 124.3, 115.4, 74.8, 34.7, 29.4, 25.7, 21.7; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{15}H_{19}N_2O_2^+$, 259.1440; found 259.1443.

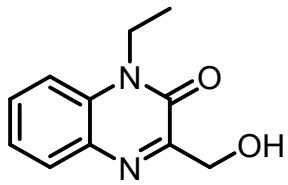


3-**(4-Hydroxytetrahydro-2*H*-pyran-4-yl)-1-methylquinoxalin-2(1*H*)-one (3ff).** According to general procedure for 8 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 5:1); 107.9 mg, total 83%; mp 164−166 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.89–7.86 (m, 1H), 7.62–7.57 (m, 1H), 7.41–7.34 (m, 2H), 5.39 (s, 1H), 4.04–

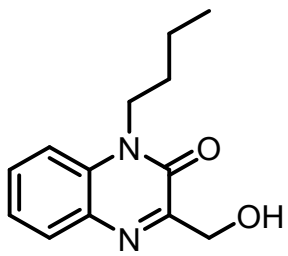
3.98 (m, 2H), 3.91–3.86 (m, 2H), 3.72 (s, 3H), 2.50–2.42 (m, 2H), 1.83–1.80 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.7, 154.5, 132.9, 131.9, 130.7, 130.4, 124.2, 113.8, 72.9, 63.6, 35.0, 28.9; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{14}H_{17}N_2O_3^+$, 261.1233; found 261.1239.



3-(Hydroxymethyl)-1-methylquinoxalin-2(1*H*)-one (3gg). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 72.2 mg, total 76%; mp 131−133 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.84 (d, J = 7.9 Hz, 1H), 7.63–7.56 (m, 2H), 7.42–7.38 (m, 1H), 5.09 (s, 1H), 4.63 (s, 2H), 3.62 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 159.1, 153.8, 133.5, 132.2, 130.5, 129.4, 123.9, 115.3, 61.7, 29.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{10}H_{11}N_2O_2^+$, 191.0814; found 191.0816.

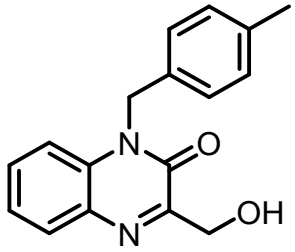


1-Ethyl-3-(hydroxymethyl)quinoxalin-2(1*H*)-one (3hh). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 71.4 mg, total 70%; mp 106−108 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.94–7.92 (m, 1H), 7.64–7.59 (m, 1H), 7.43–7.38 (m, 2H), 4.90 (s, 2H), 4.39–4.34 (m, 2H), 1.43–1.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.4, 153.4, 132.2, 132.0, 130.4, 129.9, 123.8, 113.7, 62.1, 37.1, 12.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{13}N_2O_2^+$, 205.0971; found 205.0981.

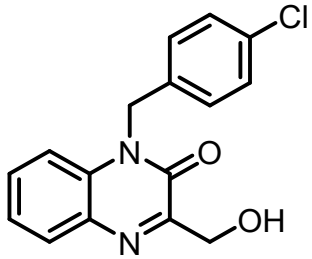


1-Butyl-3-(hydroxymethyl)quinoxalin-2(1*H*)-one (3ii). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum

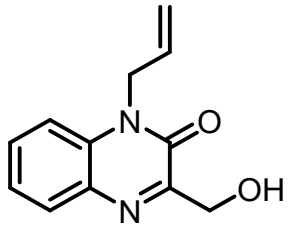
ether/EtOAc = 2:1); 78.9 mg, total 68%; mp 97−99 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.92–7.90 (m, 1H), 7.62–7.57 (m, 1H), 7.40–7.37 (m, 2H), 4.88 (s, 2H), 4.30–4.26 (m, 2H), 1.80–1.72 (m, 2H), 1.55–1.45 (m, 2H), 1.03–1.00 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.3, 153.6, 132.2, 132.1, 130.3, 129.8, 123.8, 113.9, 62.1, 41.9, 29.3, 20.3, 13.8; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1284; found 233.1291.



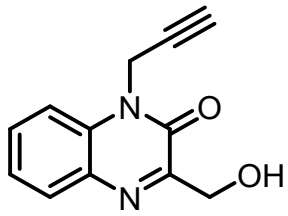
3-(Hydroxymethyl)-1-(4-methylbenzyl)quinoxalin-2(1*H*)-one (3jj). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 88.2 mg, total 63%; mp 119−121 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.93–7.90 (m, 1H), 7.51–7.47 (m, 1H), 7.38–7.34 (m, 2H), 7.18–7.13 (m, 4H), 5.49 (s, 2H), 4.96 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.5, 154.0, 137.7, 132.4, 132.1, 131.9, 130.4, 129.7, 129.7, 126.9, 124.0, 114.7, 62.2, 45.5, 21.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{17}H_{17}N_2O_2^+$, 281.1284; found 281.1296.



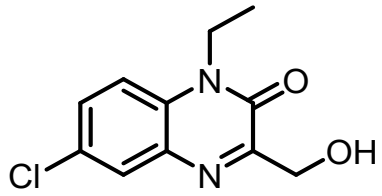
1-(4-Chlorobenzyl)-3-(hydroxymethyl)quinoxalin-2(1*H*)-one (3kk). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 100.5 mg, total 67%; mp 114−116 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm) 7.86 (d, J = 7.7 Hz, 1H), 7.54–7.51 (m, 1H), 7.43 (d, J = 8.4 Hz, 1H), 7.39–7.30 (m, 5H), 5.49 (s, 2H), 5.17–5.14 (m, 1H), 4.70 (d, J = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 159.4, 154.0, 135.3, 132.5, 132.5, 132.4, 130.5, 129.7, 129.3, 129.1, 124.2, 115.5, 61.7, 44.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{16}H_{14}ClN_2O_2^+$, 301.0738; found 301.0746.



1-Allyl-3-(hydroxymethyl)quinoxalin-2(1*H*)-one (3ll). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 65.9 mg, total 61%; mp 117−119 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.93–7.91 (m, 1H), 7.59–7.55 (m, 1H), 7.42–7.35 (m, 2H), 6.00–5.90 (m, 1H), 5.32–5.29 (m, 1H), 5.19 (d, J = 17.2 Hz, 1H), 4.95–4.93 (m, 2H), 4.90 (d, J = 3.1 Hz, 2H), 3.86 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.4, 153.4, 132.3, 132.0, 130.3, 129.7, 124.0, 118.4, 114.5, 62.1, 44.2; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{13}N_2O_2^+$, 217.0971; found 217.0972.

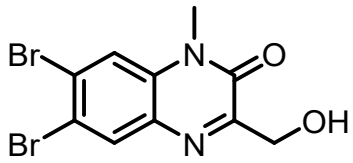


3-(Hydroxymethyl)-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one (3mm). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 77.0 mg, total 72%; mp 144−146 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.95–7.92 (m, 1H), 7.67–7.63 (m, 1H), 7.56–7.54 (m, 1H), 7.47–7.42 (m, 1H), 5.09 (d, J = 2.5 Hz, 2H), 4.90 (s, 2H), 2.34–2.33 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.4, 152.8, 132.0, 131.5, 130.5, 129.8, 124.4, 114.4, 76.4, 73.5, 62.1, 31.2; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{11}N_2O_2^+$, 215.0814; found 215.0824.

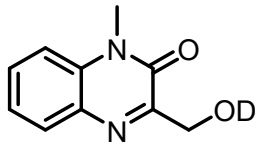


7-Chloro-1-ethyl-3-(hydroxymethyl)quinoxalin-2(1*H*)-one (3nn). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 92.8 mg, total 78%; mp 112−114 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.86 (d, J = 2.4 Hz, 1H), 7.54–7.51 (m, 1H), 7.32 (d, J = 9.0 Hz, 1H), 4.86 (s, 2H), 4.33–

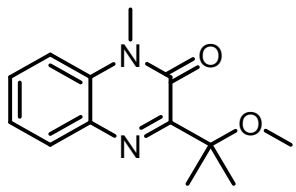
4.28 (m, 2H), 1.39–1.35 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.0, 153.0, 132.7, 130.7, 130.4, 129.1, 129.0, 114.9, 62.1, 37.4, 12.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{11}H_{12}ClN_2O_2^+$, 239.0581; found 239.0578.



6,7-Dibromo-3-(hydroxymethyl)-1-methylquinoxalin-2(1*H*)-one (3oo). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 147.5 mg, total 85%; mp 162−164 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.16 (s, 1H), 7.66 (s, 1H), 4.87 (s, 2H), 3.70 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.3, 153.2, 133.5, 133.0, 131.5, 126.8, 119.3, 118.6, 62.2, 29.1; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{10}H_9Br_2N_2O_2^+$, 348.9004; found 348.9012.

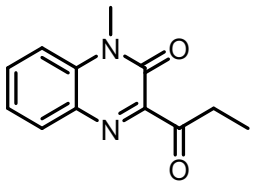


3-((Hydroxy-*d*)methyl)-1-methylquinoxalin-2(1*H*)-one (3pp). According to general procedure for 12 hours; a pale-yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 2:1); 147.5 mg, total 74%; mp 127−129 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.92 (d, J = 8.1 Hz, 1H), 7.64–7.60 (m, 1H), 7.43–7.38 (m, 2H), 4.89 (s, 2H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.4, 153.8, 133.1, 131.9, 130.4, 129.6, 124.1, 113.9, 62.1, 28.8; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{10}H_{10}DN_2O_2^+$, 192.0877; found 192.0883.

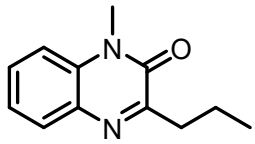


3-(2-Methoxypropan-2-yl)-1-methylquinoxalin-2(1*H*)-one (4a). According to general procedure for 10 hours; a yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 10:1); 98.7 mg, total 85%; mp 108−110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91–7.89 (m, 1H), 7.56–7.52 (m, 1H), 7.34–7.27 (m, 2H), 3.68 (s, 3H), 3.33 (s, 3H), 1.73 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.4, 153.2, 133.6, 131.8, 130.6, 130.4, 123.4, 113.4, 78.9,

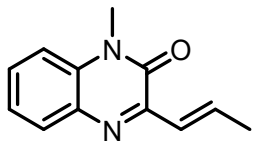
51.4, 28.9, 24.4; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{13}H_{17}N_2O_2^+$, 233.1284; found 233.1295.



1-Methyl-3-propionylquinoxalin-2(1*H*)-one (5a). According to general procedure for 10 hours; a yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 3:1); 198.7 mg, total 92%; mp 175−177 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90 (d, J = 8.0 Hz, 1H), 7.67–7.64 (m, 1H), 7.40–7.34 (m, 2H), 3.71 (s, 3H), 3.12–3.07 (m, 2H), 1.25–1.21 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 201.4, 152.9, 152.8, 134.2, 132.5, 131.9, 131.2, 124.1, 113.9, 34.2, 29.0, 7.5; HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{13}N_2O_2^+$, 217.0971; found 217.0970.



1-Methyl-3-propylquinoxalin-2(1*H*)-one (6a). According to general procedure for 12 hours; a yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 20:1); 104.2 mg, total 93%; mp: 90–92°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.85–7.82 (m, 1H), 7.55–7.50 (m, 1H), 7.36–7.28 (m, 2H), 3.71 (s, 3H), 2.95–2.91 (m, 2H), 1.86–1.81 (m, 2H), 1.08–1.04 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 161.2, 154.9, 133.1, 132.7, 129.6, 129.5, 123.5, 113.6, 36.3, 29.0, 20.3, 14.1. HRMS (ESI-TOF) m/z $[M+Na]^+$ calcd for: $C_{12}H_{14}N_2ONa^+$, 225.0998; found 225.1009.



(*E*)-1-Methyl-3-(prop-1-en-1-yl)quinoxalin-2(1*H*)-one (7a). According to general procedure for 6 hours; a yellow solid has been obtained after purification on silica gel (petroleum ether/EtOAc = 20:1); 49.2 mg, total 82%; mp: 105–107°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.85–7.83 (m, 1H), 7.54–7.50 (m, 1H), 7.37–7.28 (m, 3H), 7.10–7.06 (m, 1H), 3.73 (s, 3H), 2.05–2.03 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.8, 152.6, 138.0, 133.1, 132.9, 129.8, 129.5, 125.9,

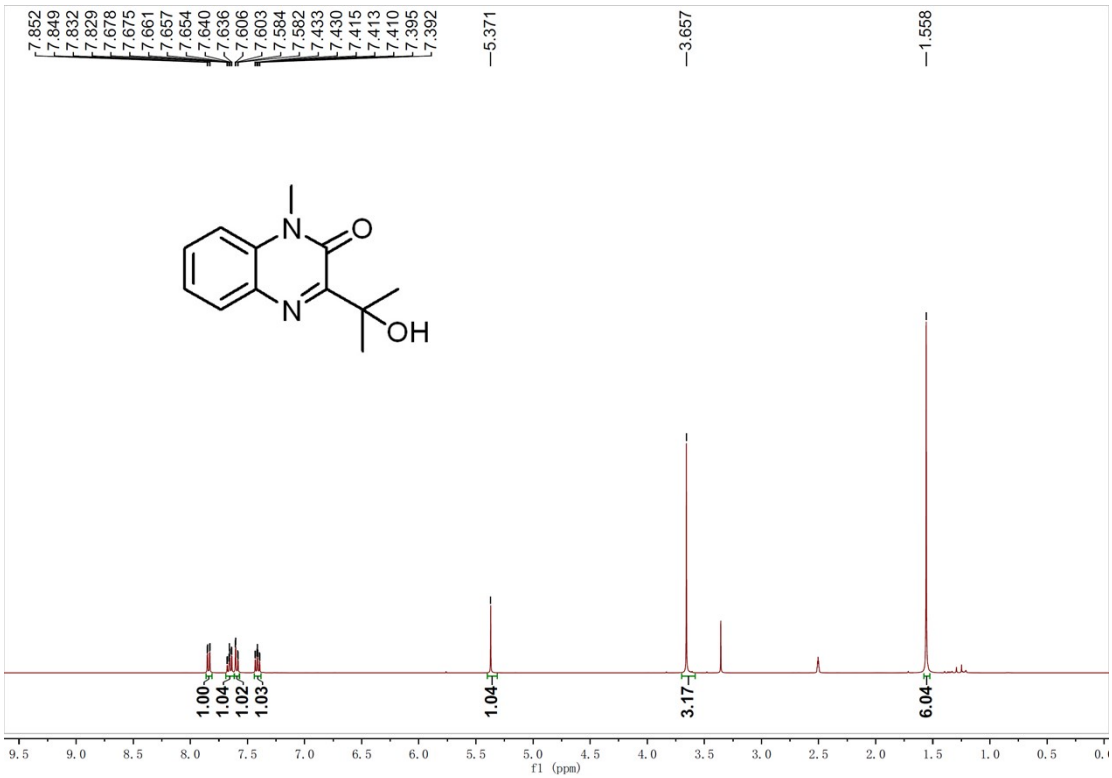
123.7, 113.5, 19.2. HRMS (ESI-TOF) m/z $[M+H]^+$ calcd for: $C_{12}H_{13}N_2O^+$, 201.1022; found 201.1026.

5 References

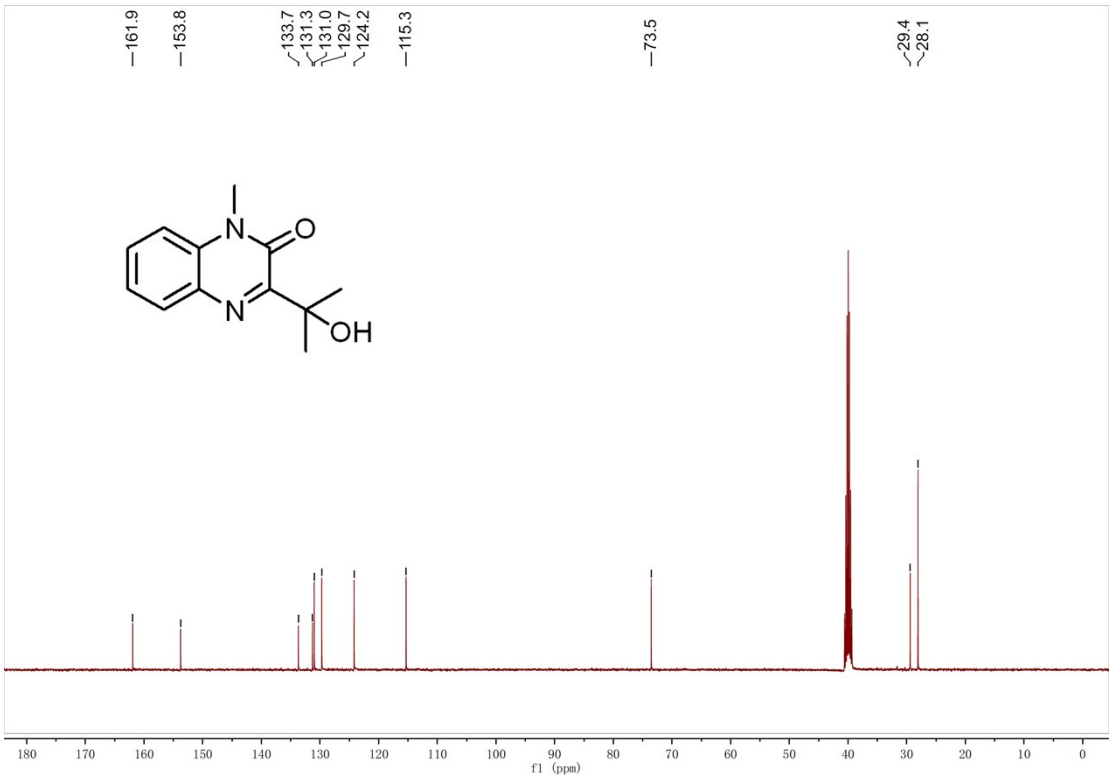
1 J. Fu, J. Yuan, Y. Zhang, Y. Xiao, P. Mao, X. Diao and L. Qu, *Org. Chem. Front*., 2018, **5**, 3382–3390.

2. H. Zhang, J. Xu, Y. Ouyang, X. Yue, C. Zhou, Z. Ni and W. Li, *Chin. Chem. Lett*., 2022, **33**, 2036–2040.

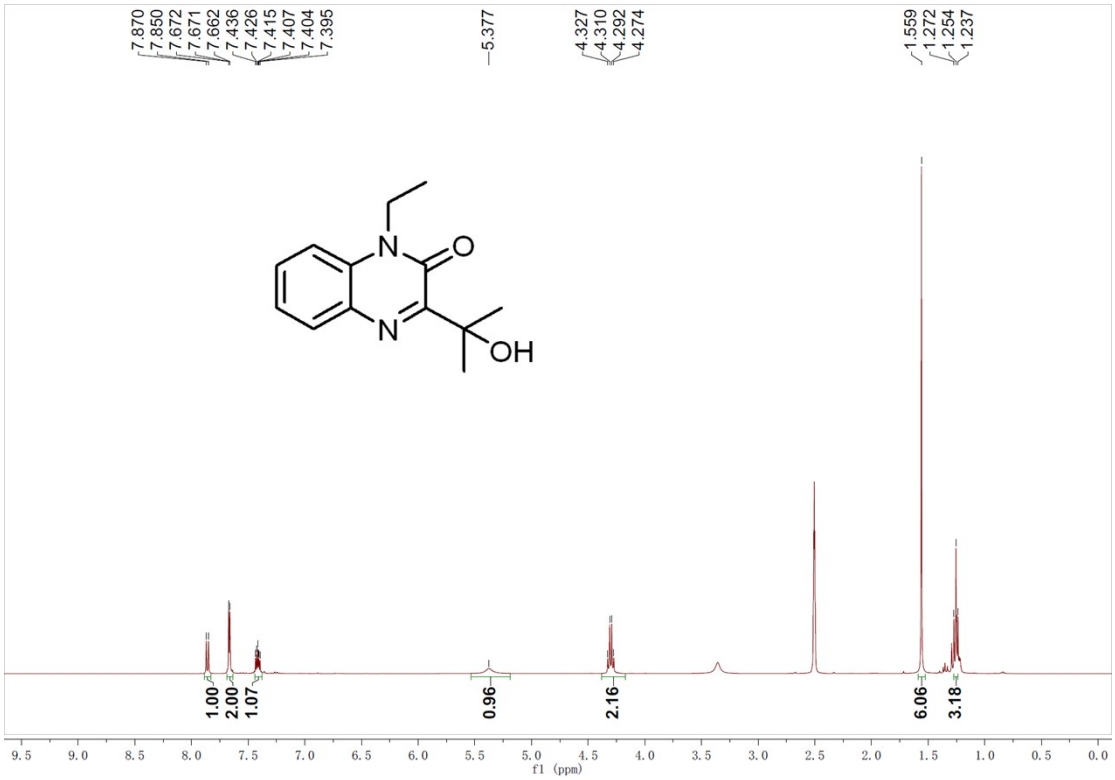
6 Copies of ^{1}H and ^{13}C NMR spectra of products 3, 4a, 5a, 6a and 7a



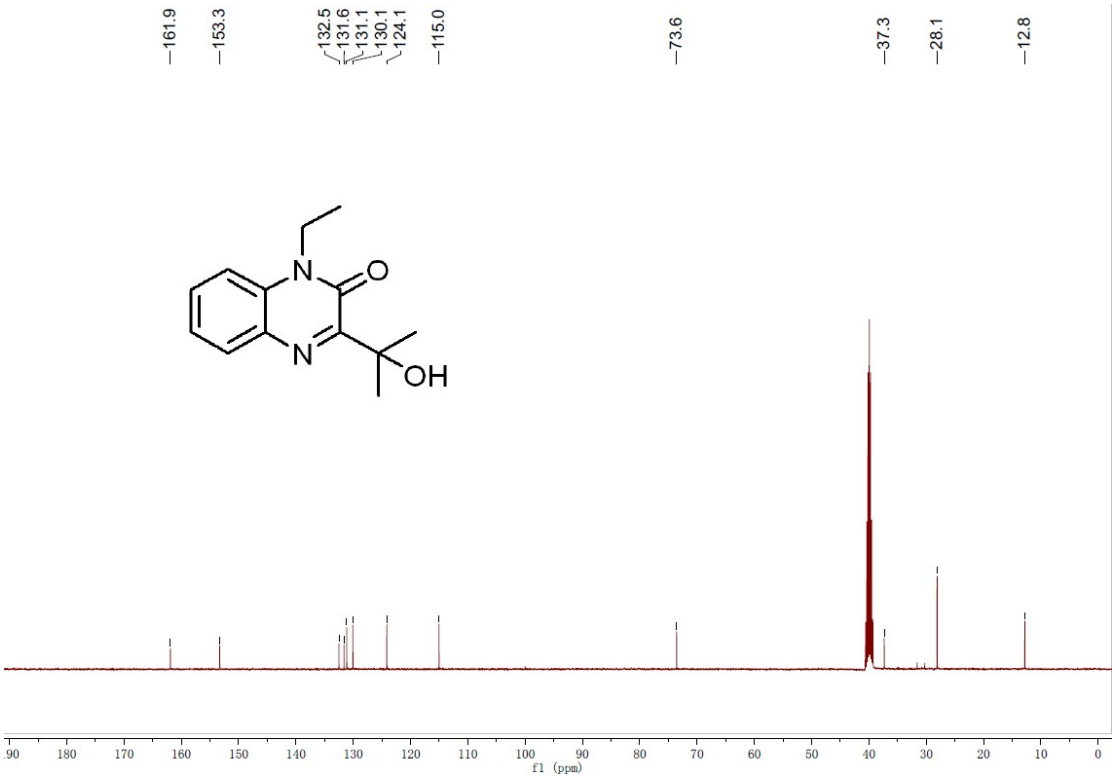
^{1}H NMR Spectrum of Compound 3a



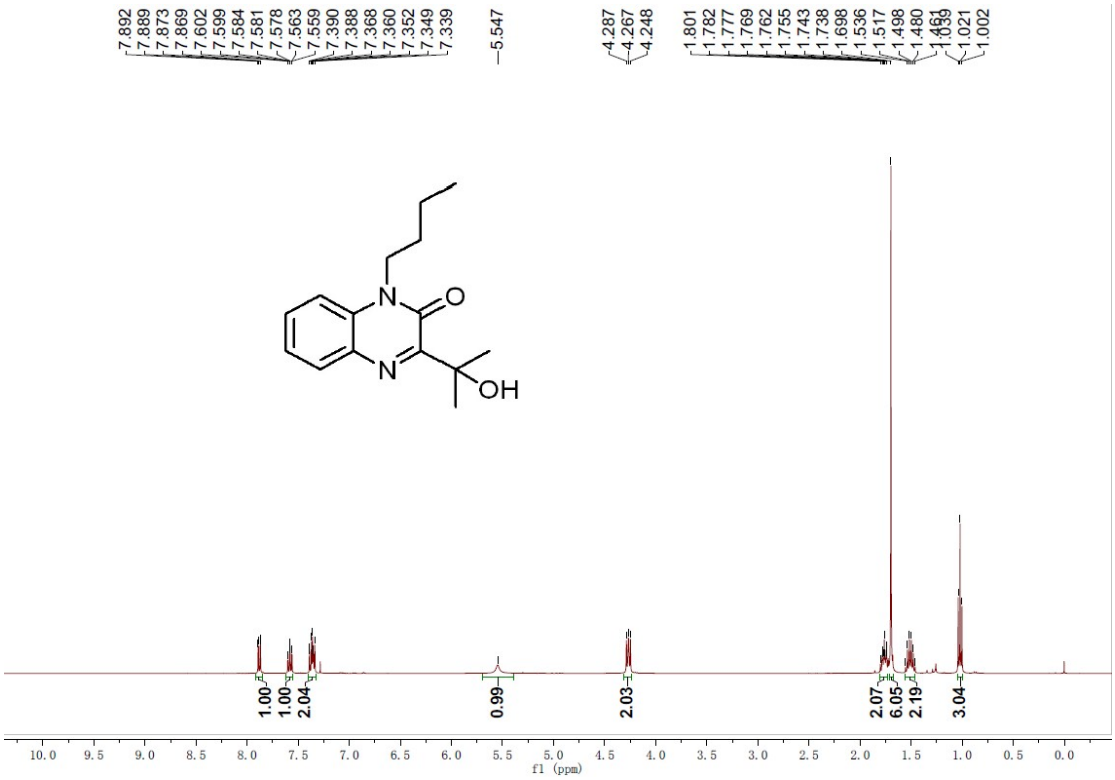
^{13}C NMR Spectrum of Compound 3a



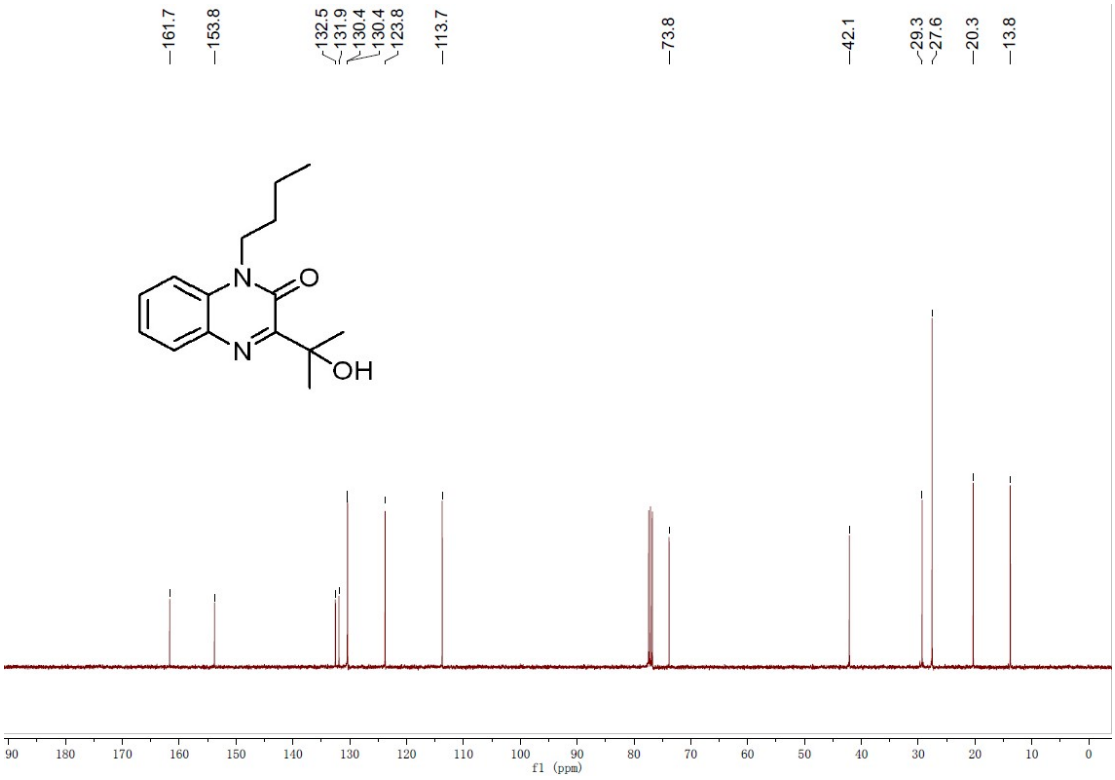
^{1}H NMR Spectrum of Compound 3b



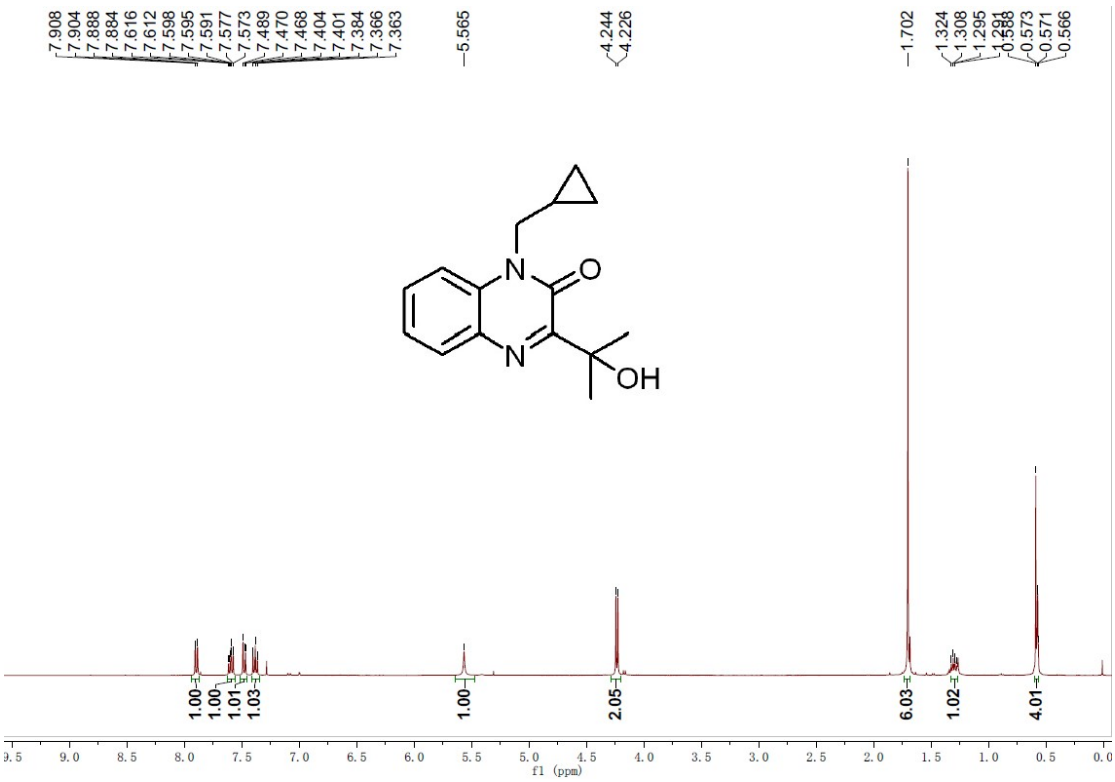
^{13}C NMR Spectrum of Compound 3b



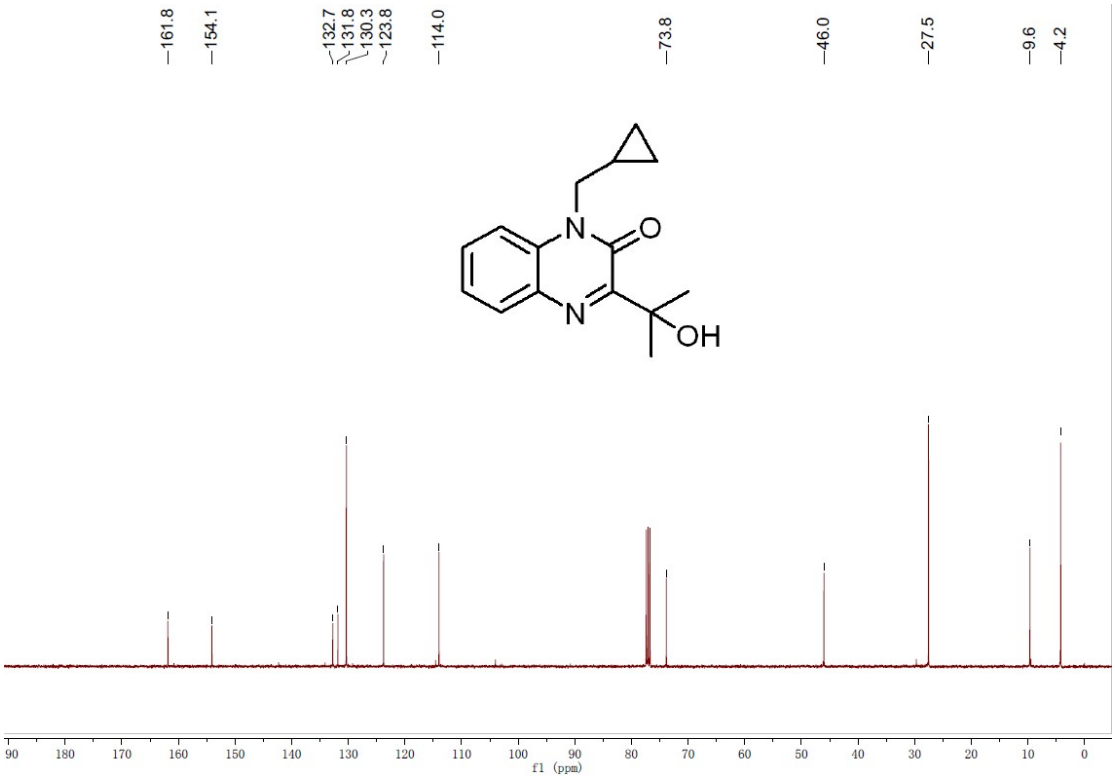
^{1}H NMR Spectrum of Compound 3c



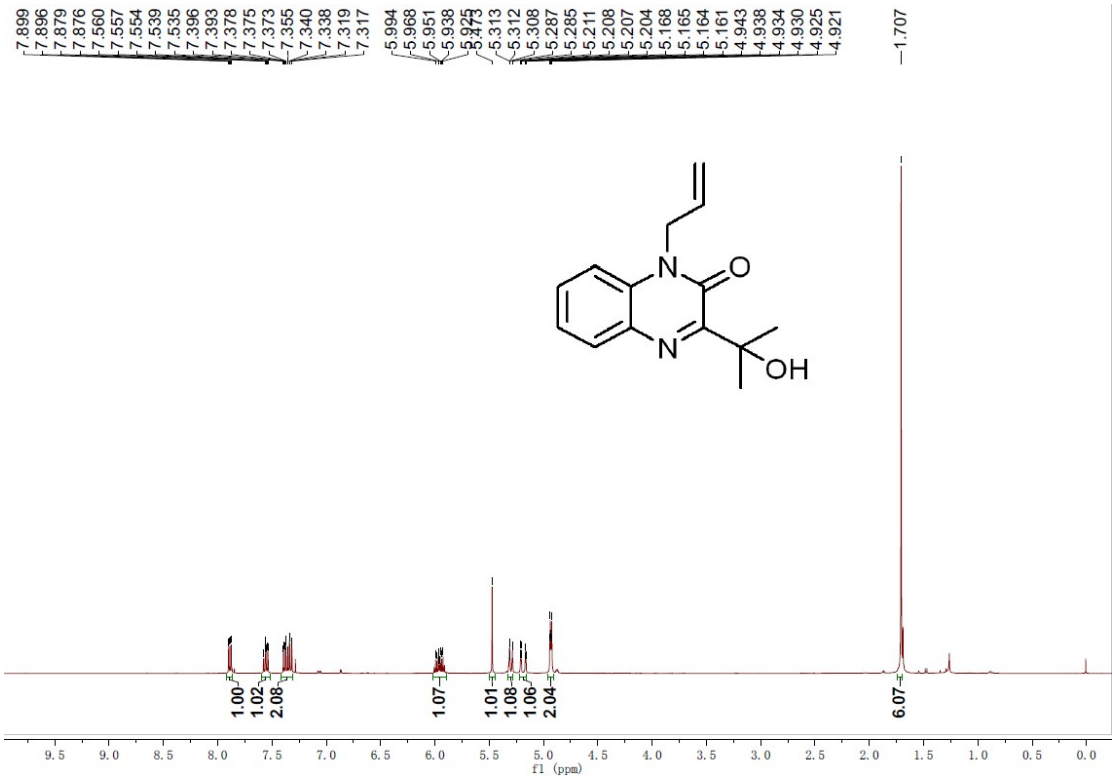
^{13}C NMR Spectrum of Compound 3c



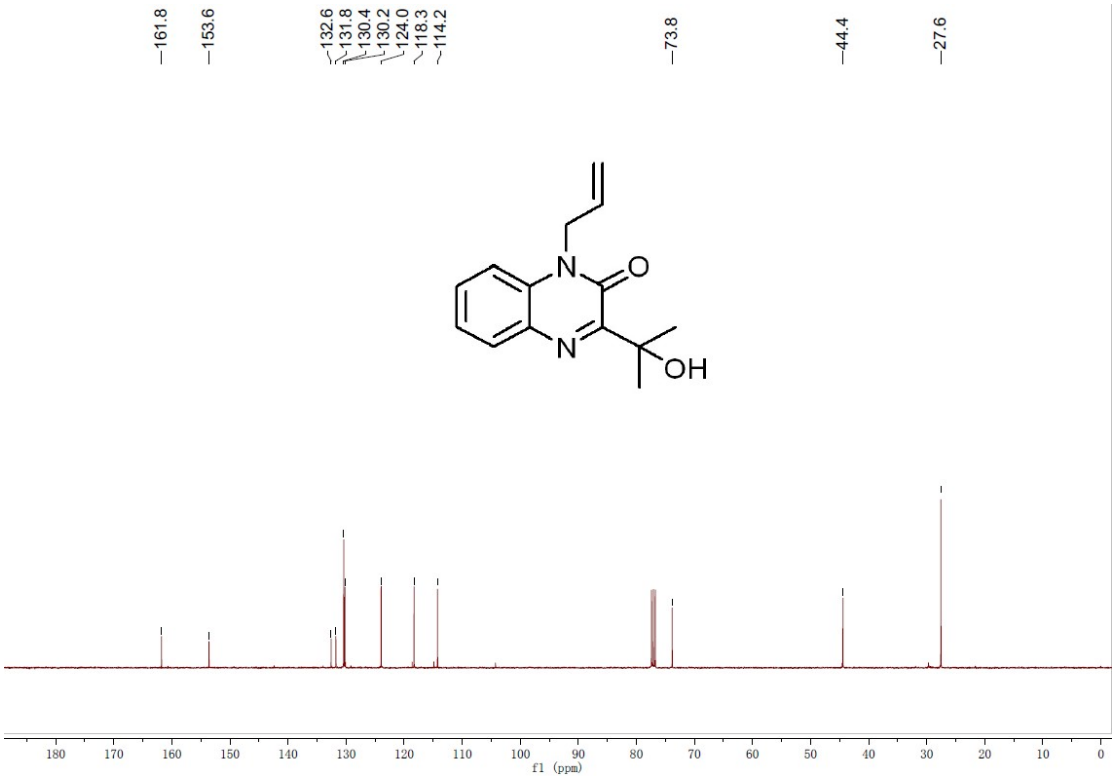
^{1}H NMR Spectrum of Compound 3d



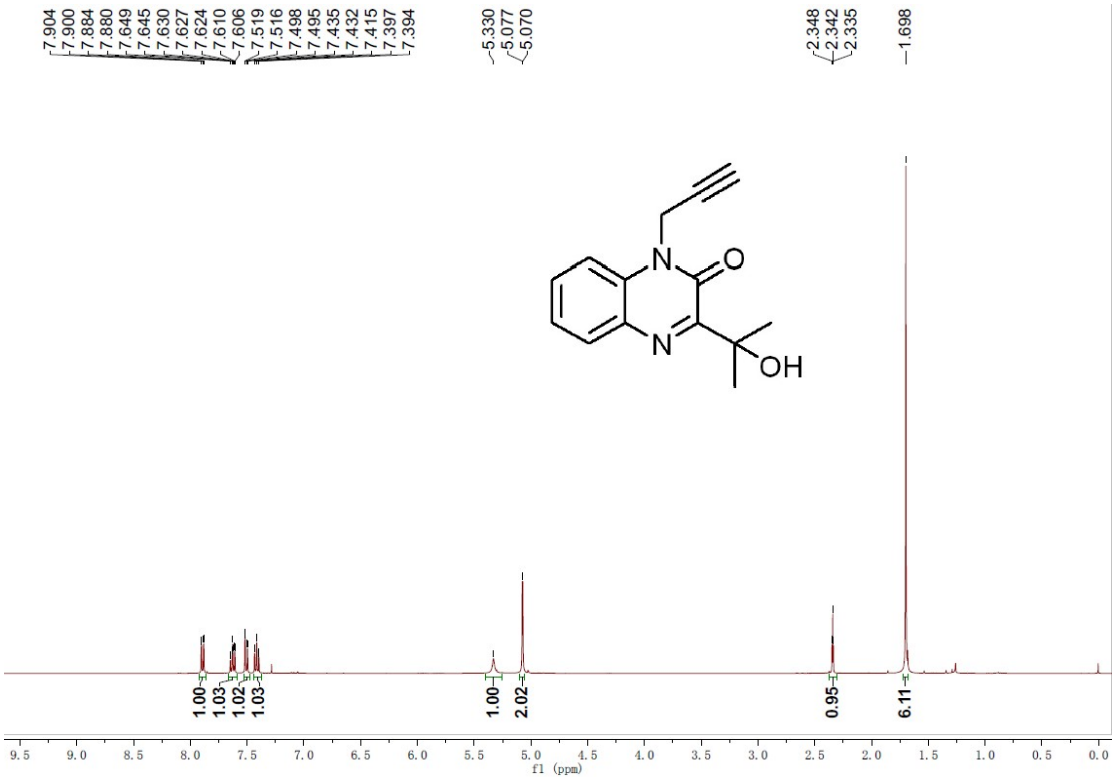
^{13}C NMR Spectrum of Compound 3d



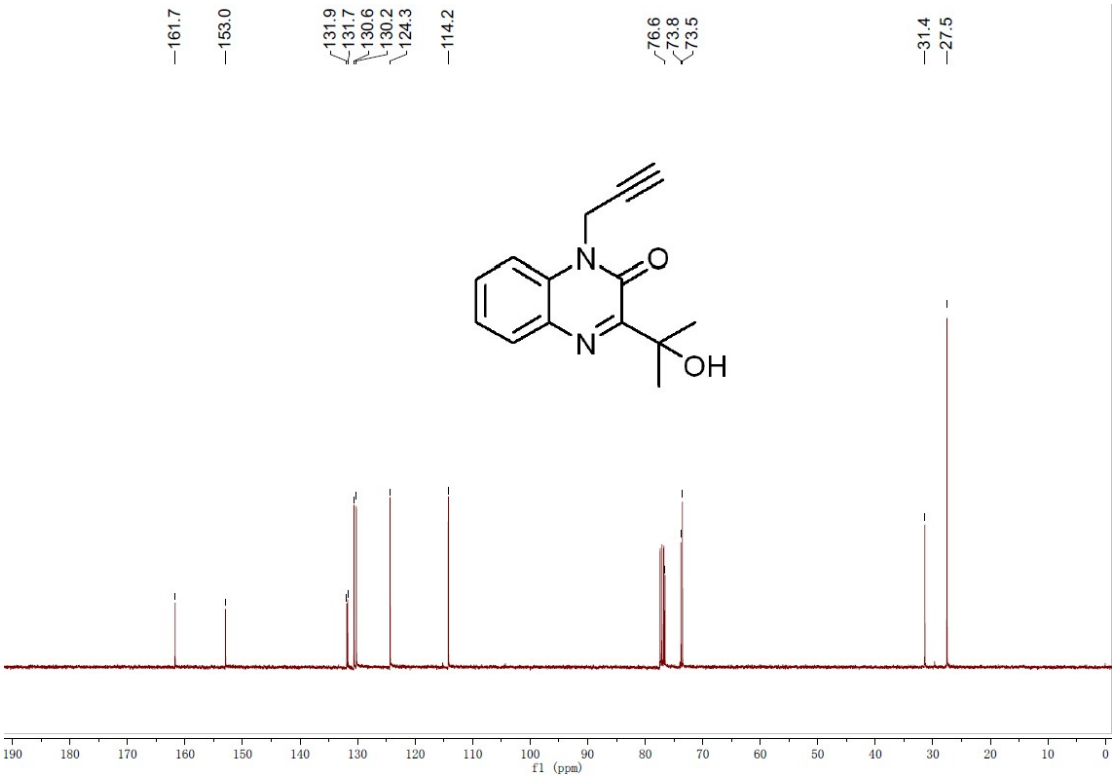
^{1}H NMR Spectrum of Compound 3e



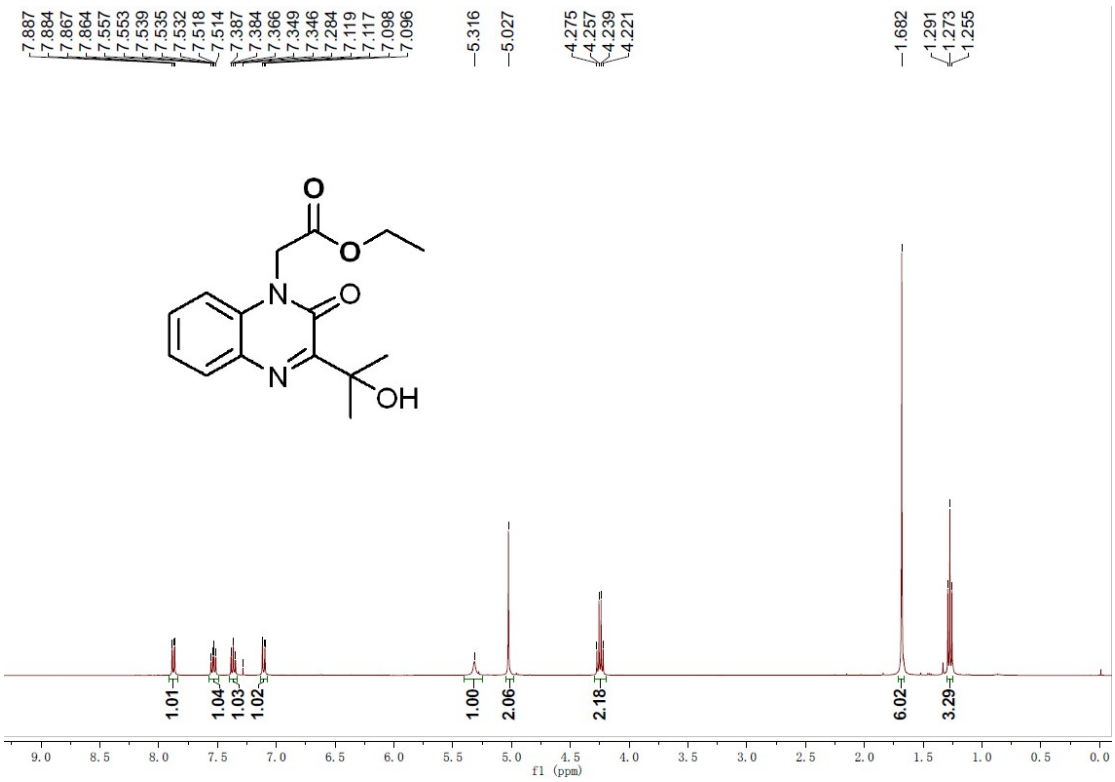
^{13}C NMR Spectrum of Compound 3e



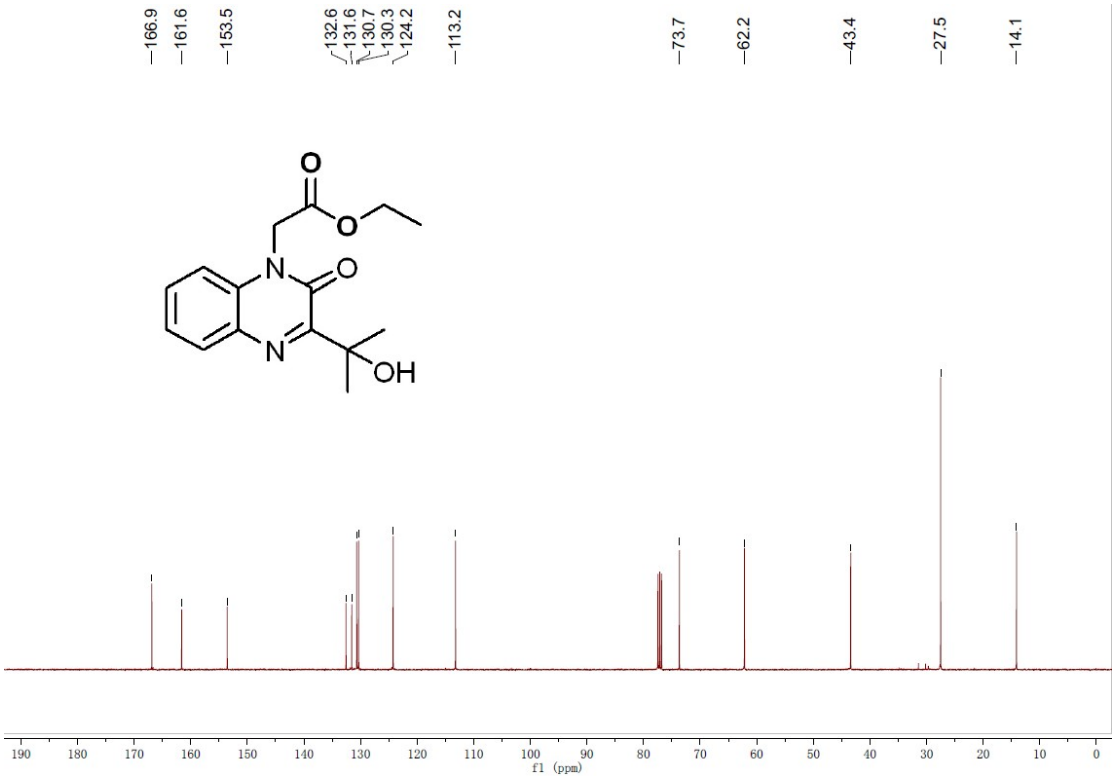
^{1}H NMR Spectrum of Compound 3f



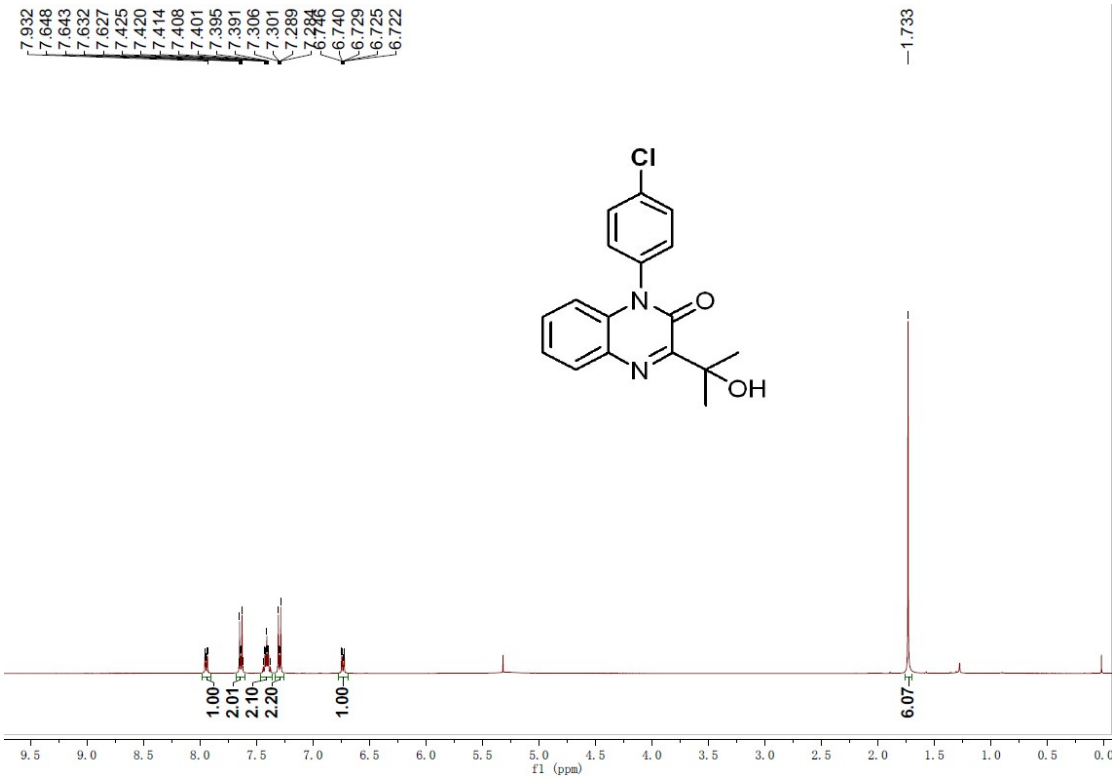
^{13}C NMR Spectrum of Compound 3f



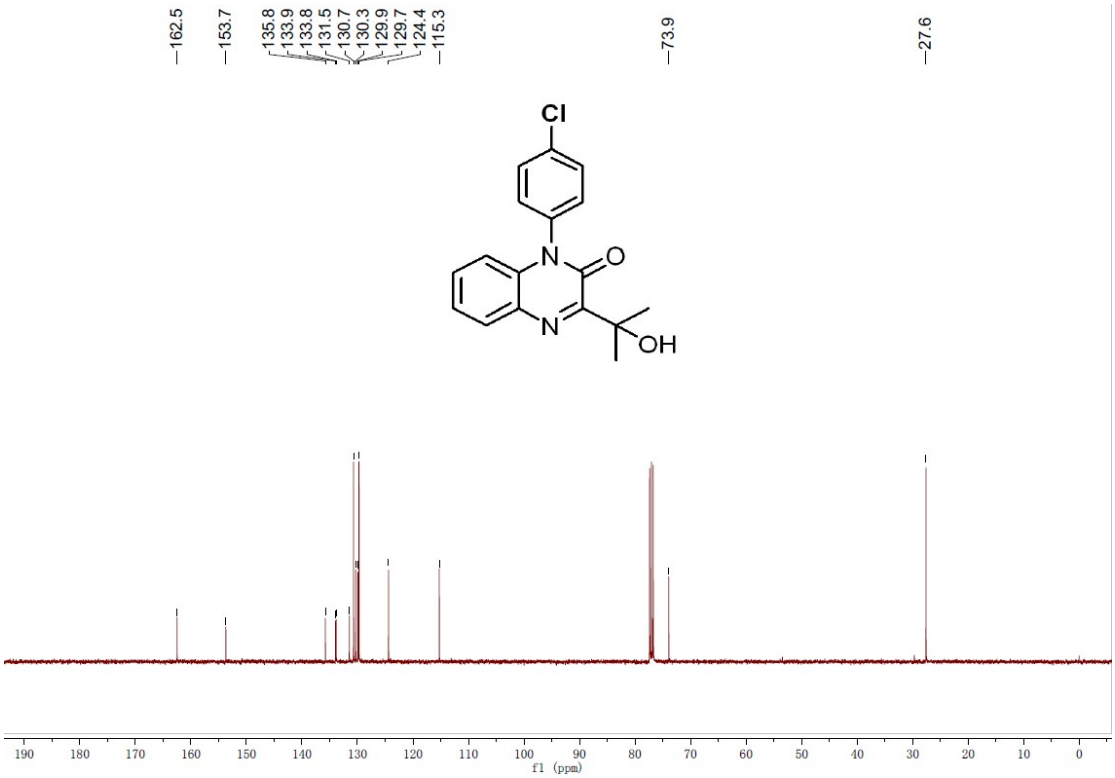
^{1}H NMR Spectrum of Compound 3g



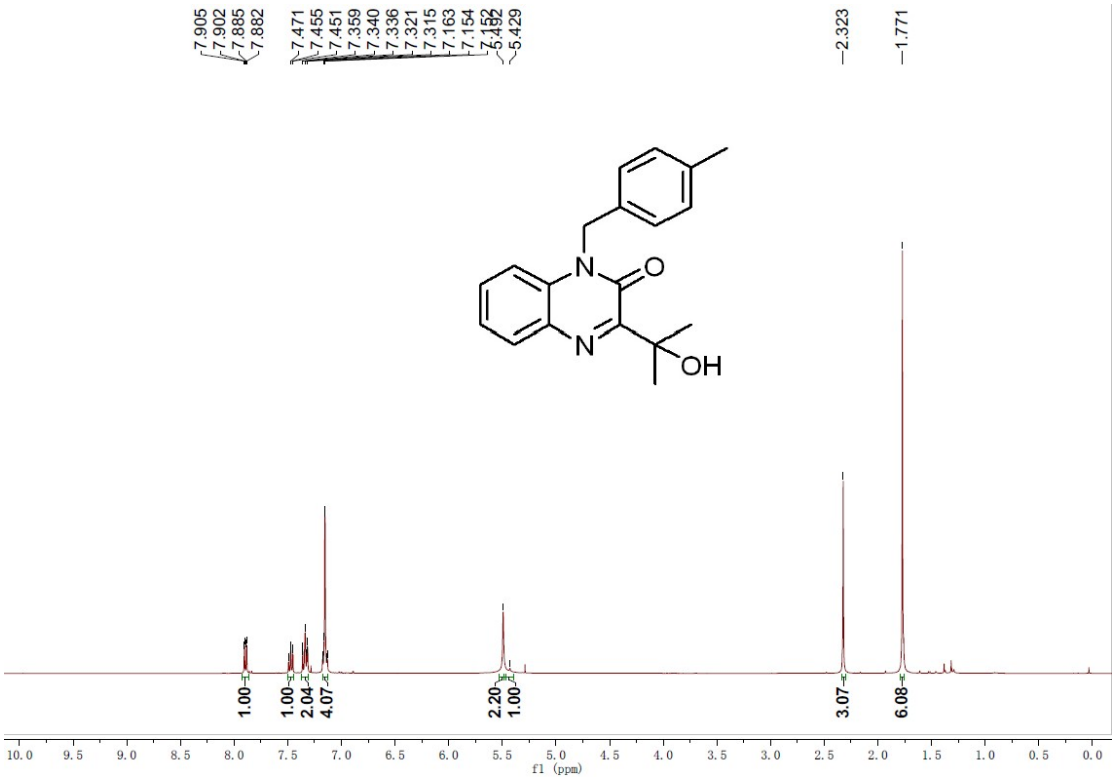
^{13}C NMR Spectrum of Compound 3g



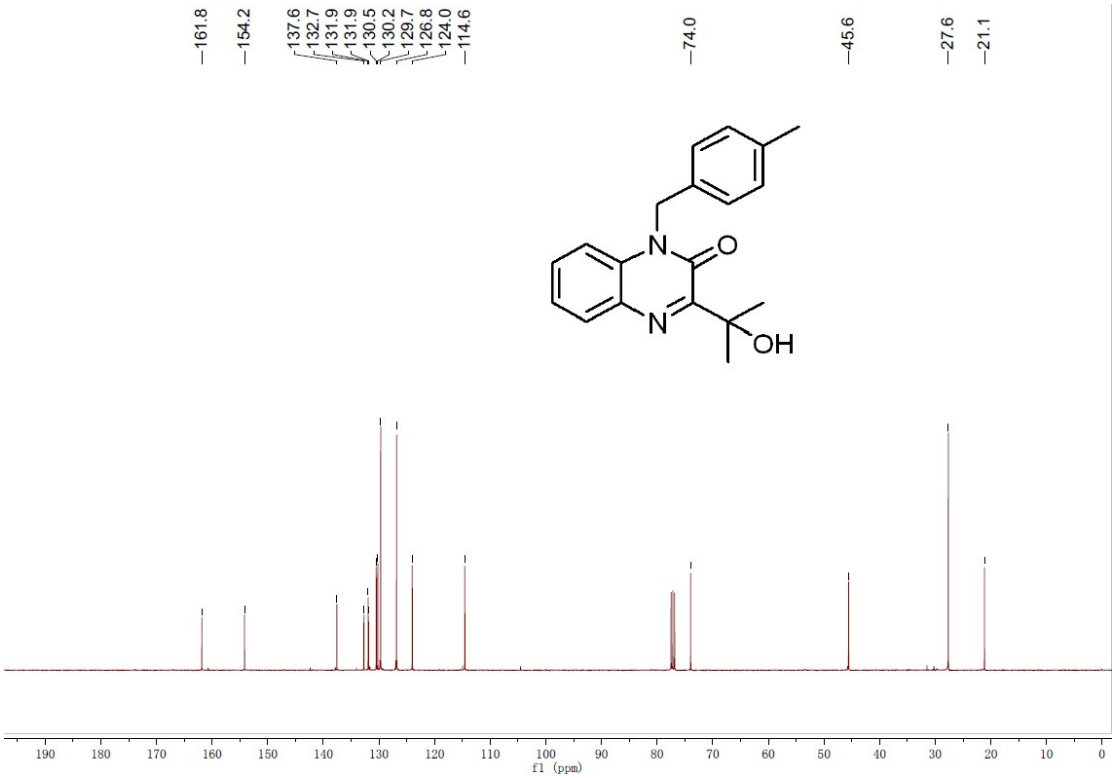
^{1}H NMR Spectrum of Compound 3h



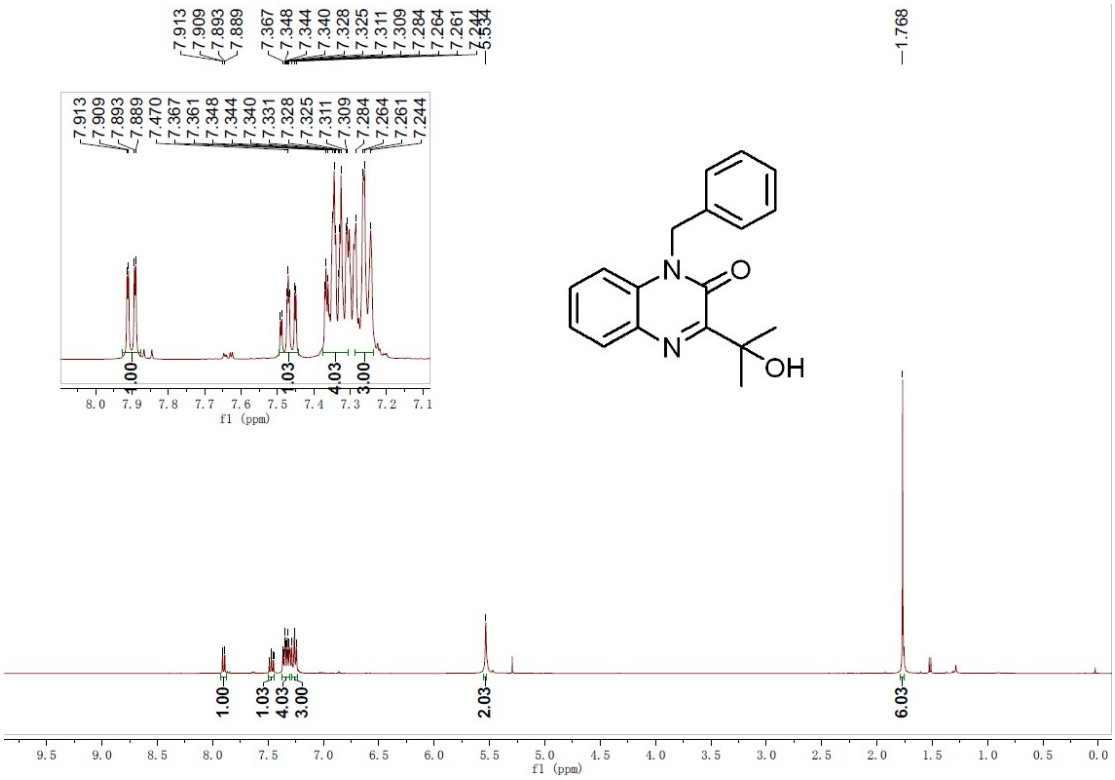
^{13}C NMR Spectrum of Compound 3h



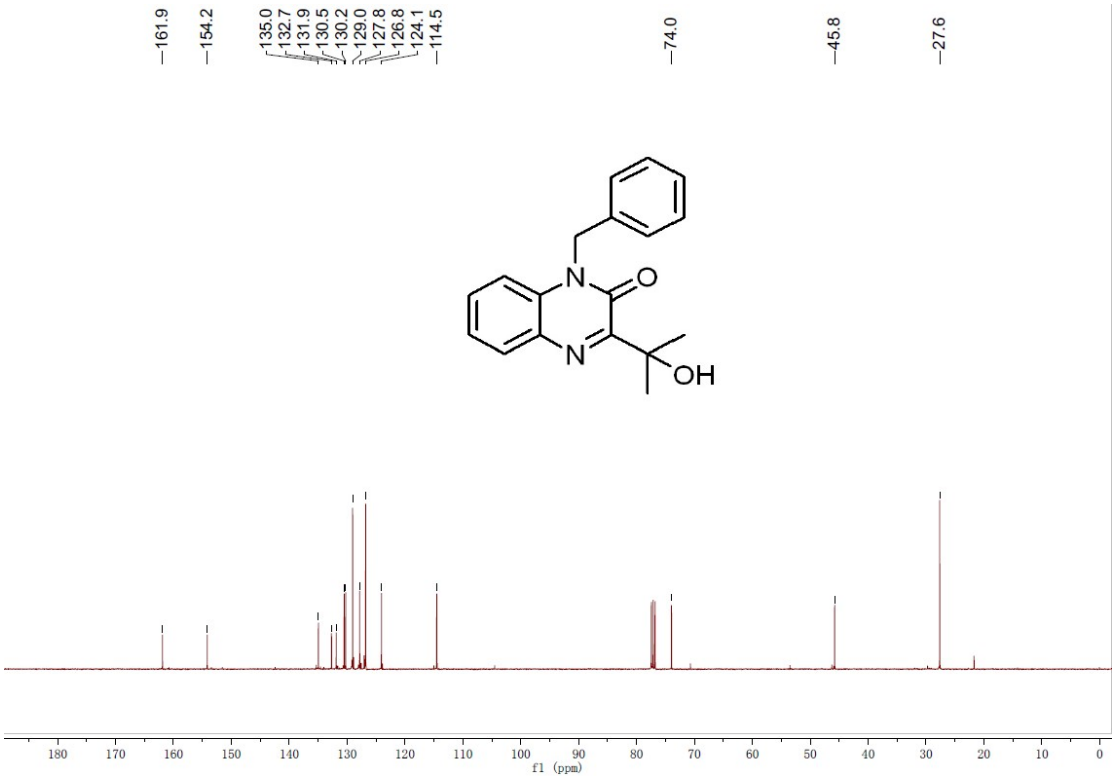
^{1}H NMR Spectrum of Compound 3i



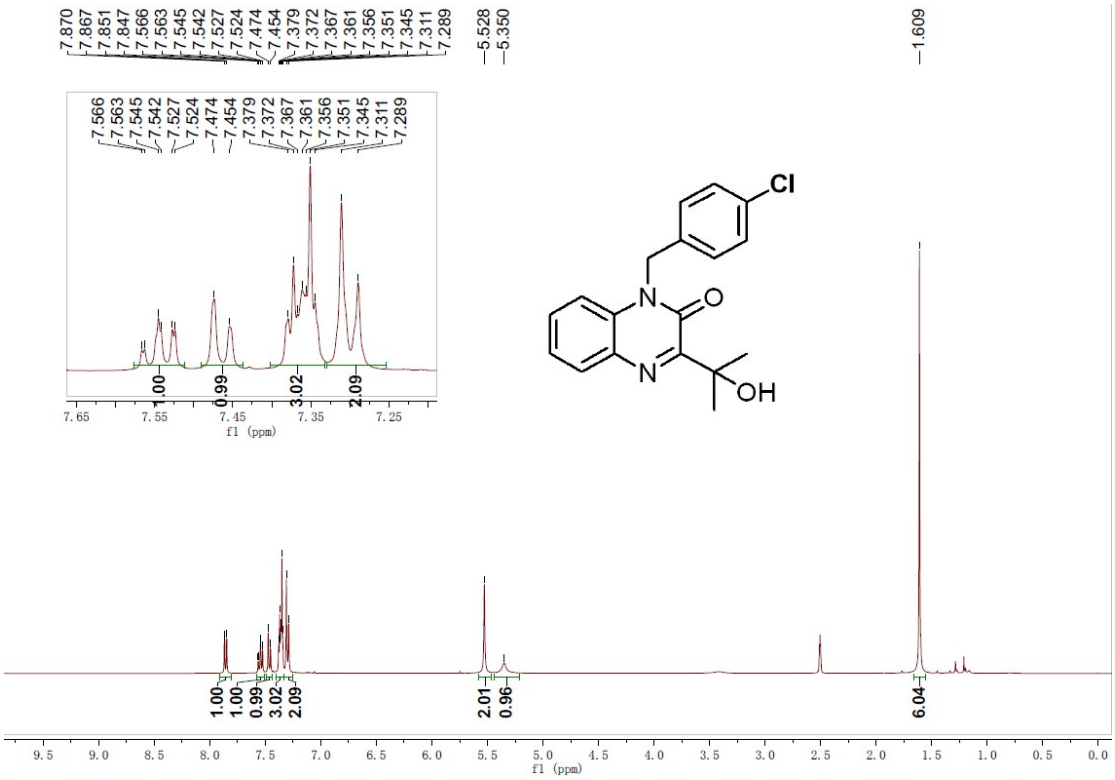
^{13}C NMR Spectrum of Compound 3i



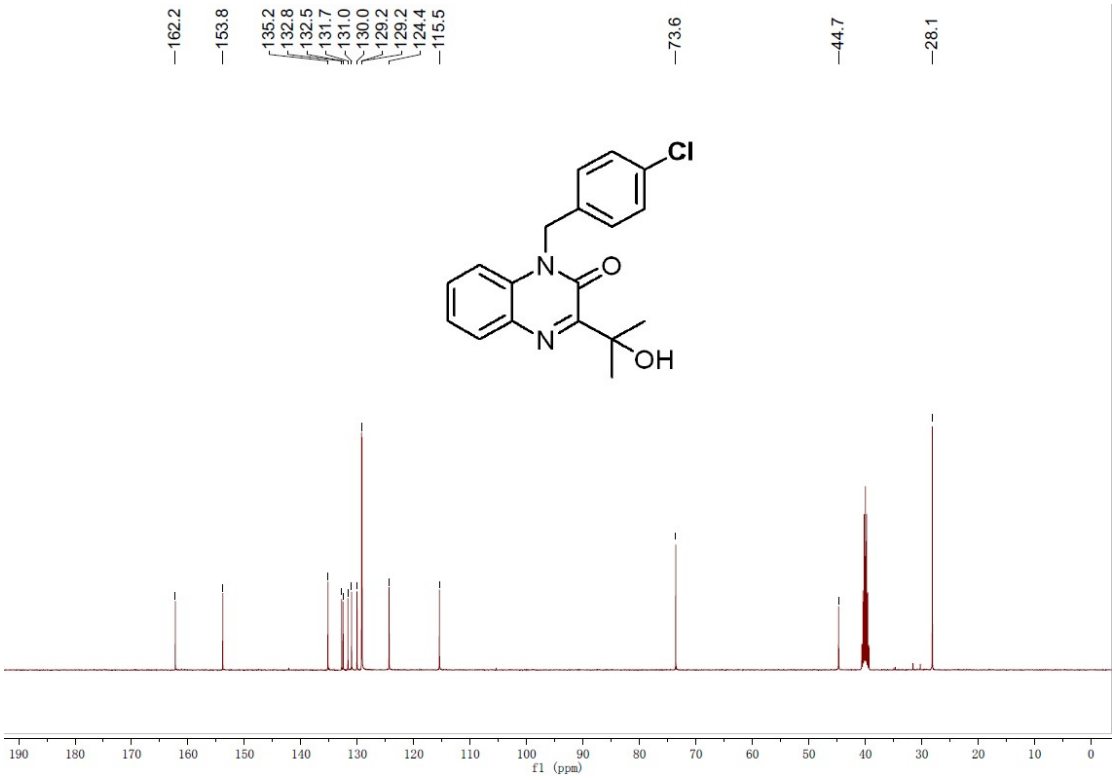
^{1}H NMR Spectrum of Compound 3j



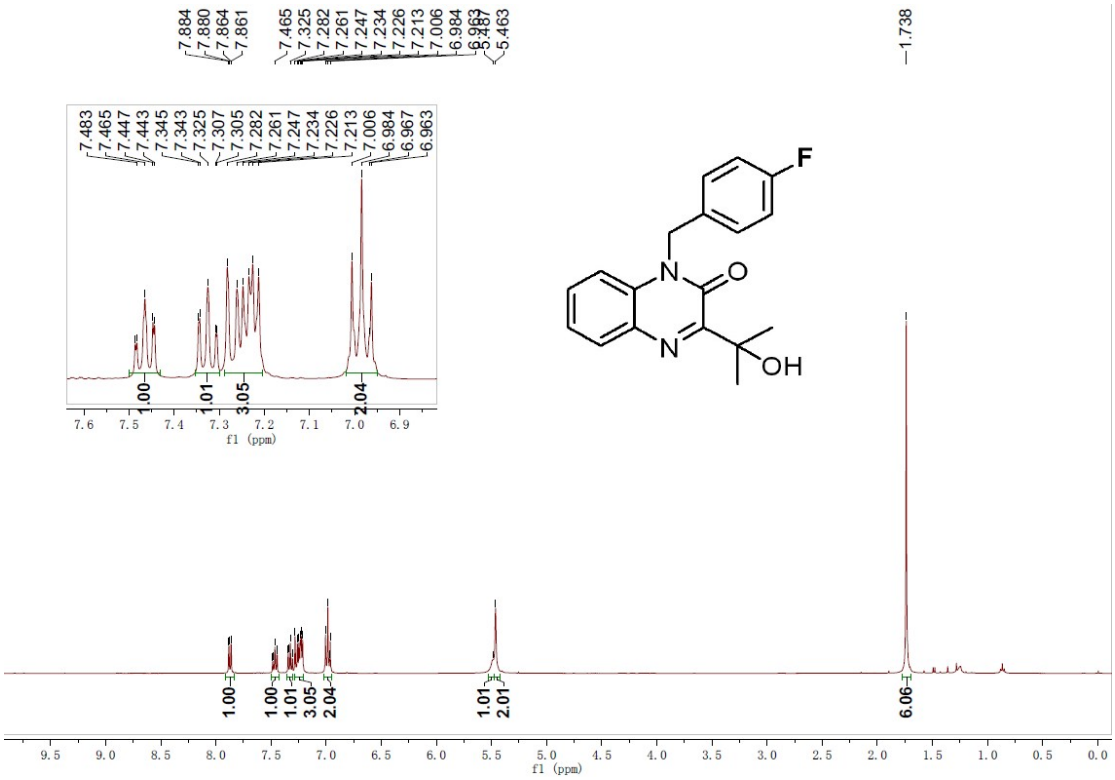
^{13}C NMR Spectrum of Compound 3j



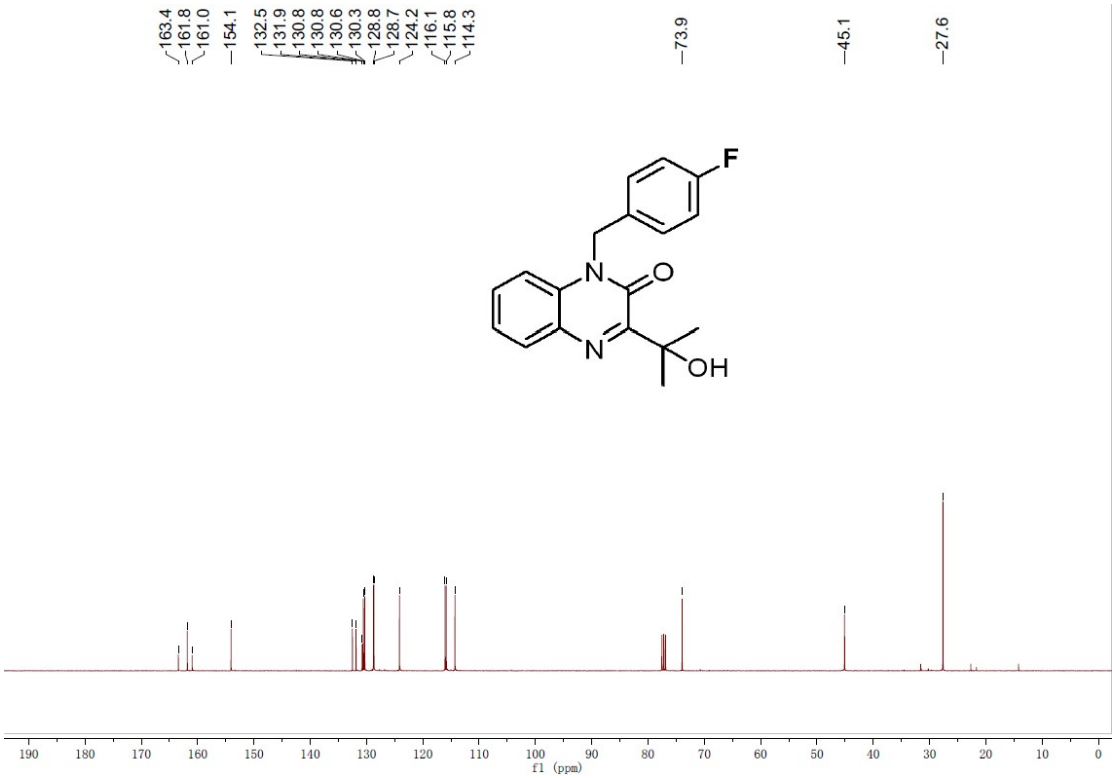
^{1}H NMR Spectrum of Compound 3k



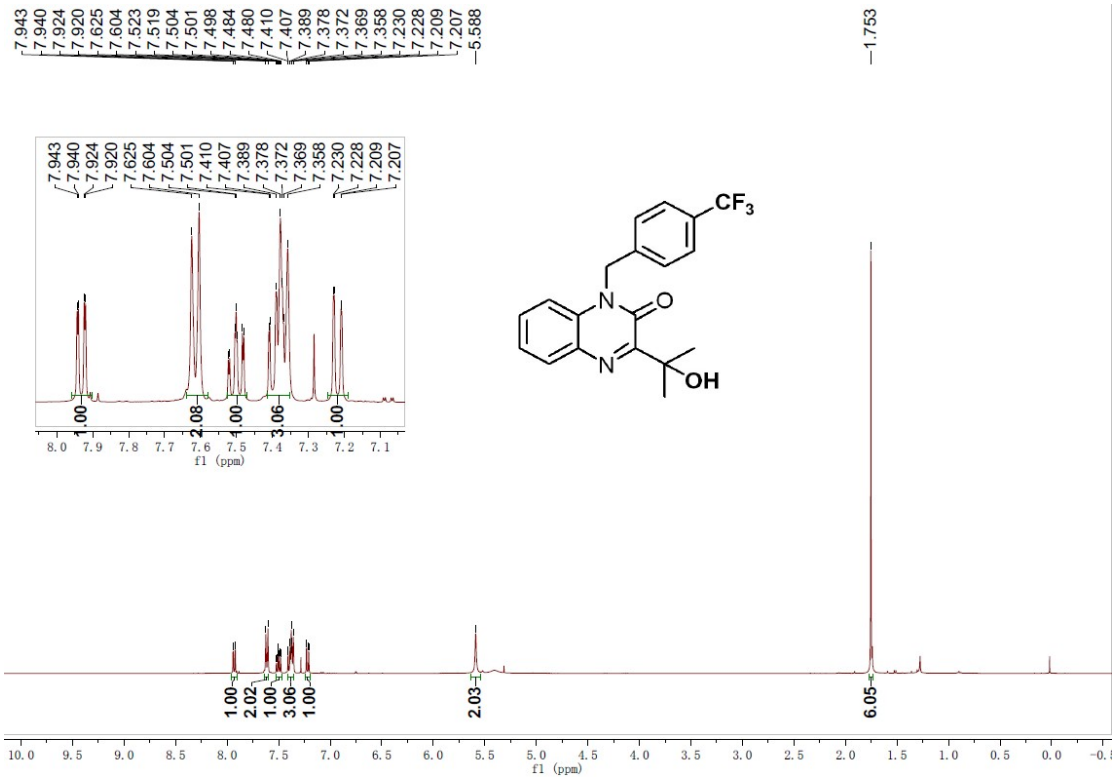
^{13}C NMR Spectrum of Compound 3k



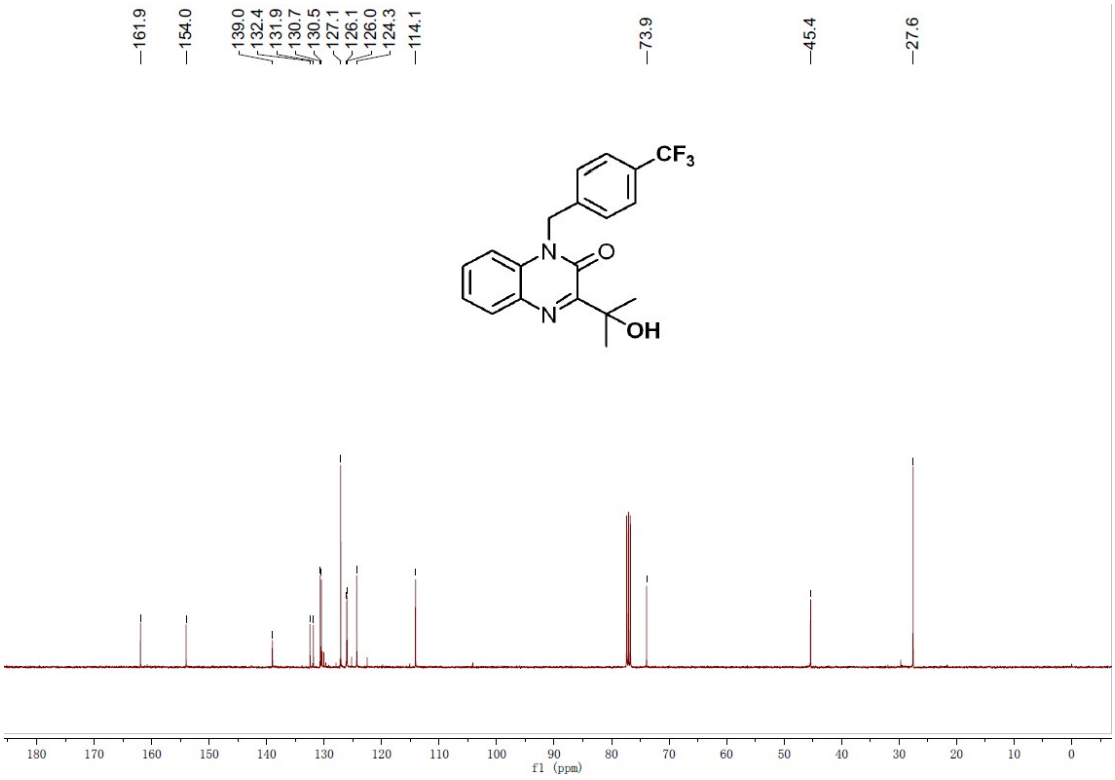
^{1}H NMR Spectrum of Compound 3l



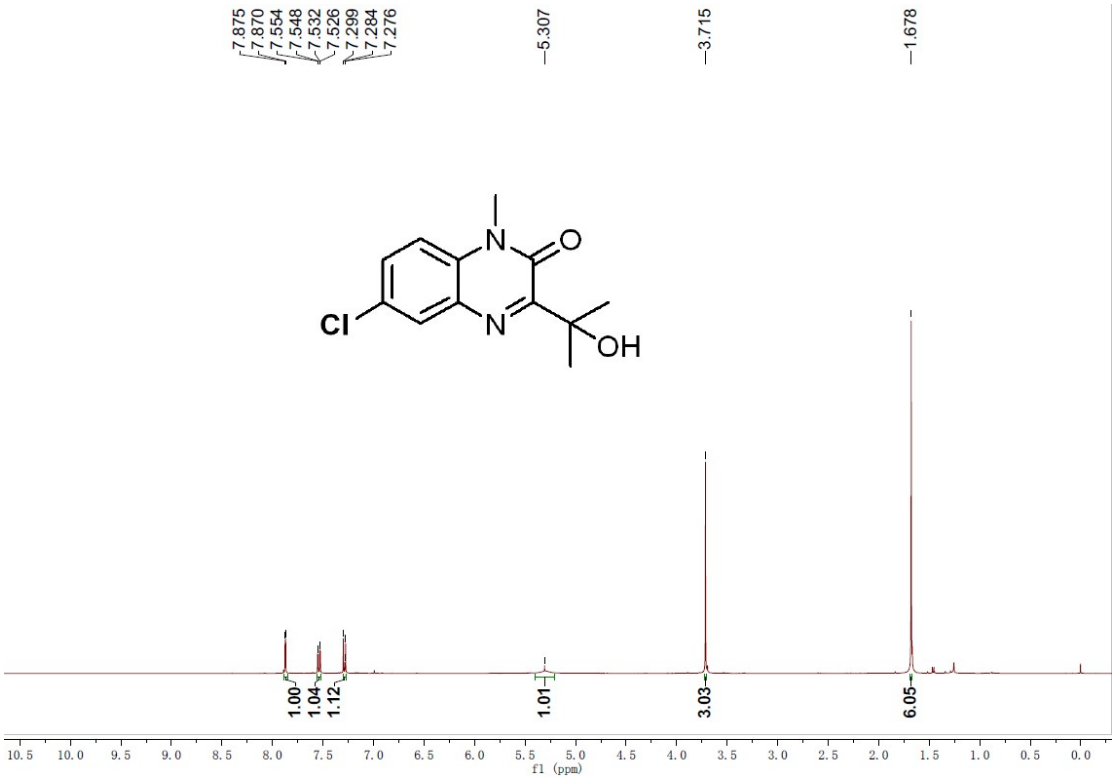
^{13}C NMR Spectrum of Compound 3l



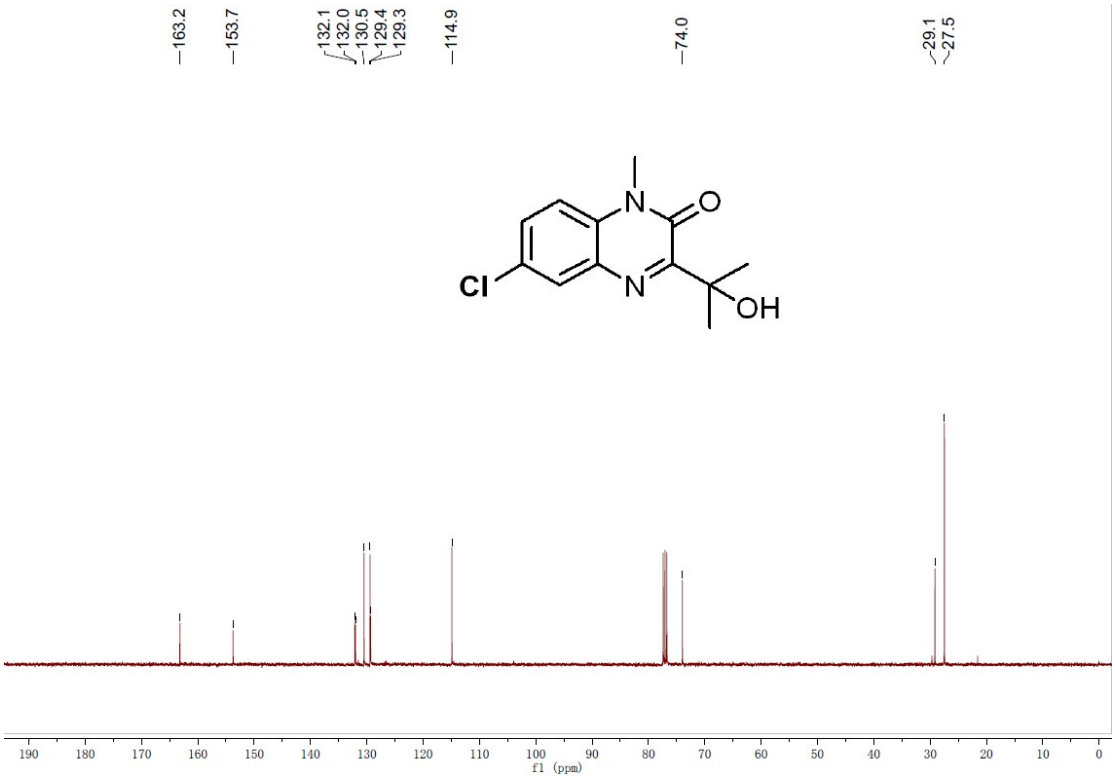
^{1}H NMR Spectrum of Compound 3m



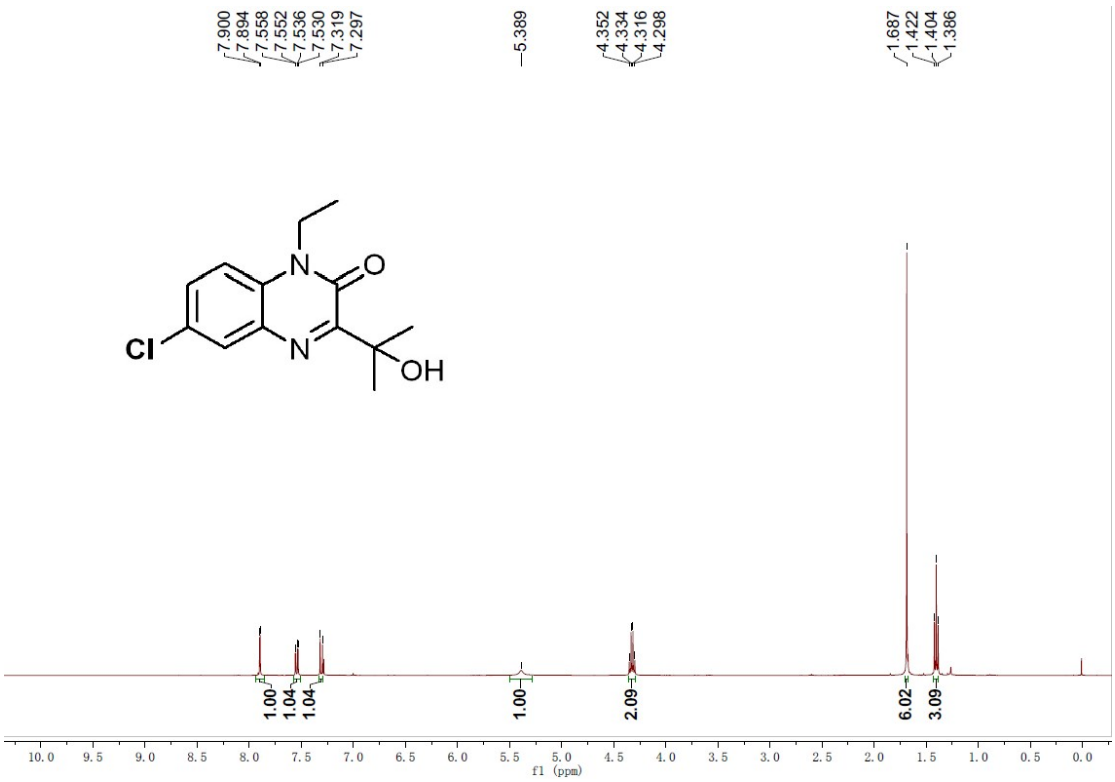
^{13}C NMR Spectrum of Compound 3m



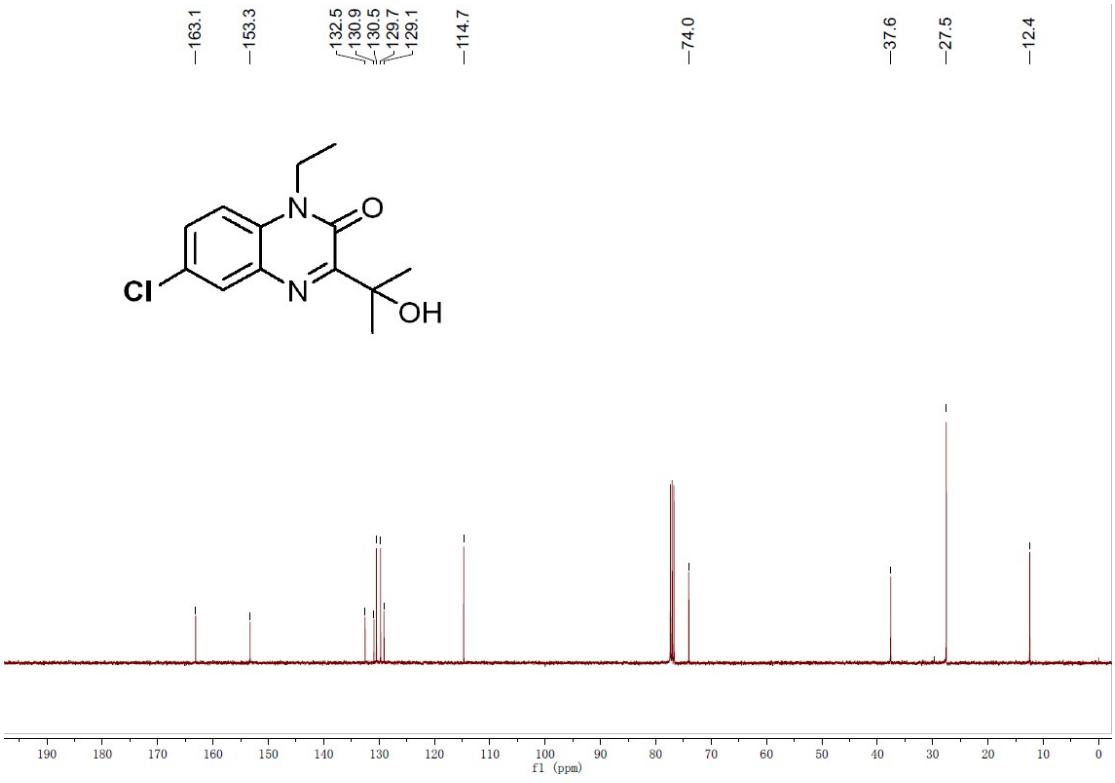
^{1}H NMR Spectrum of Compound 3n



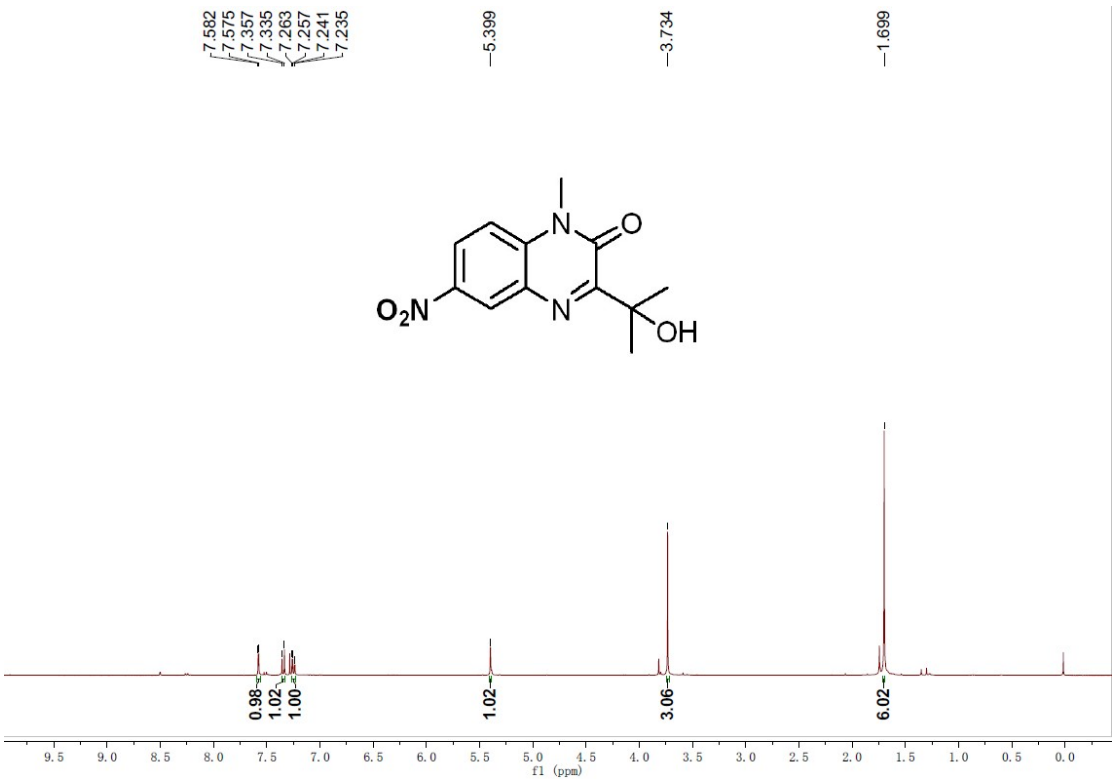
^{13}C NMR Spectrum of Compound 3n



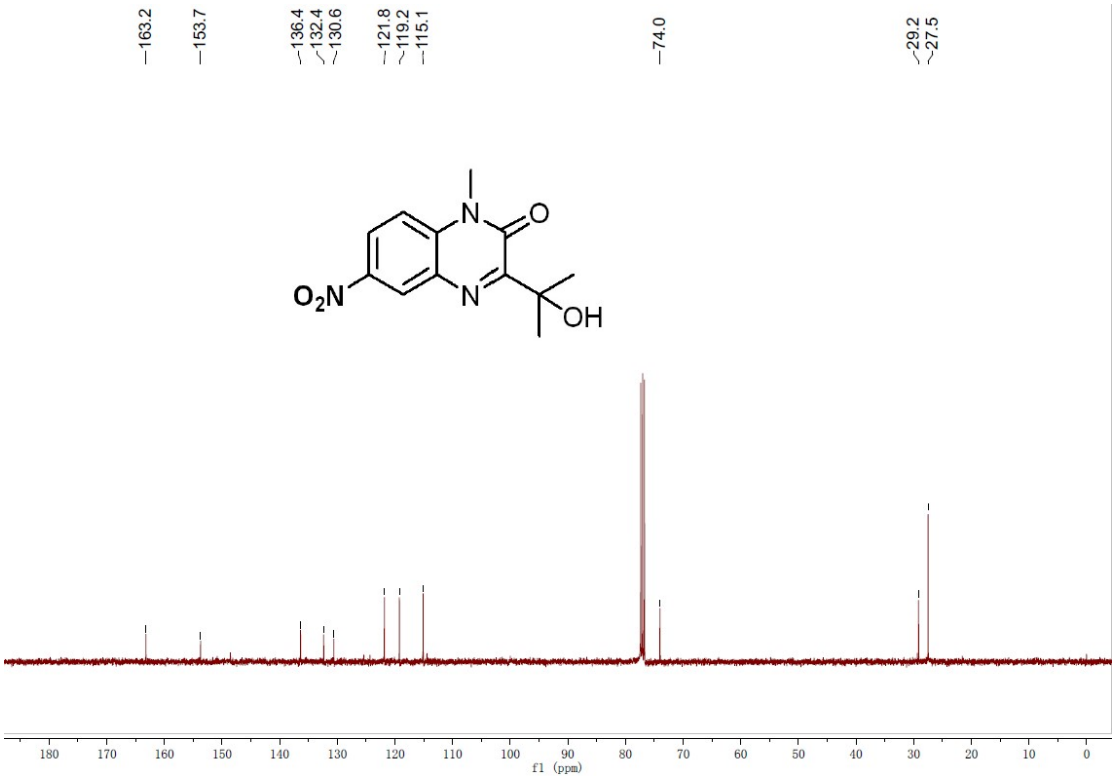
^{1}H NMR Spectrum of Compound 3o



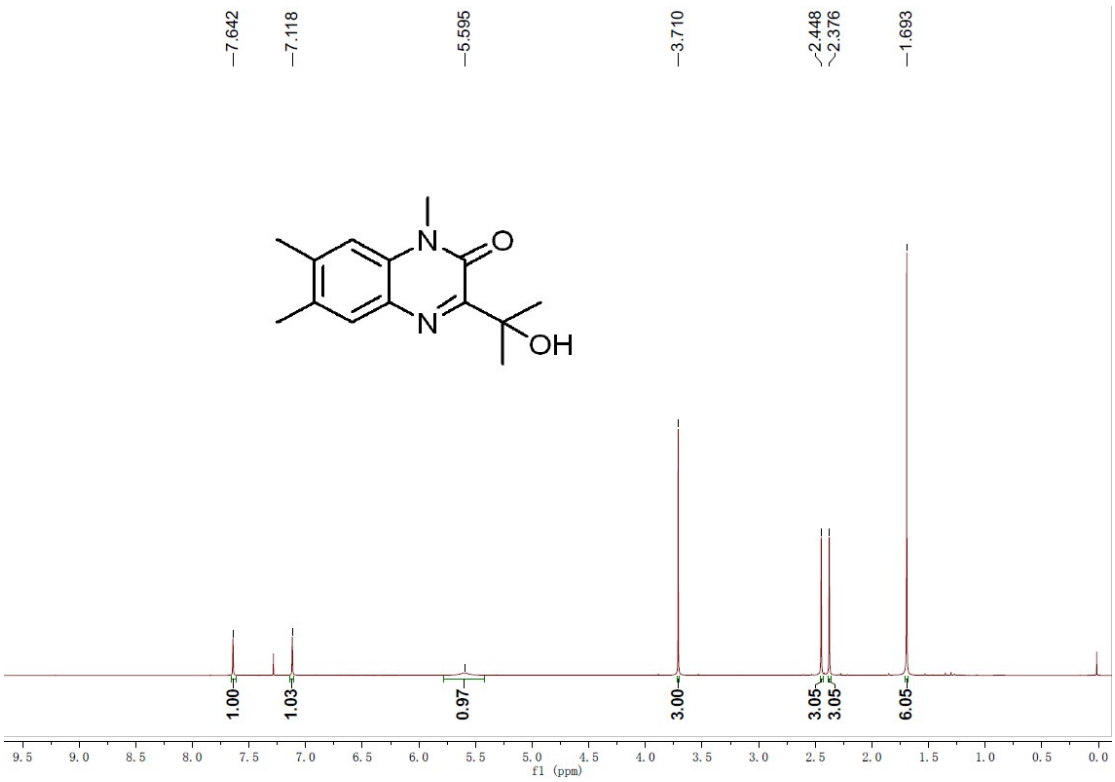
^{13}C NMR Spectrum of Compound 3o



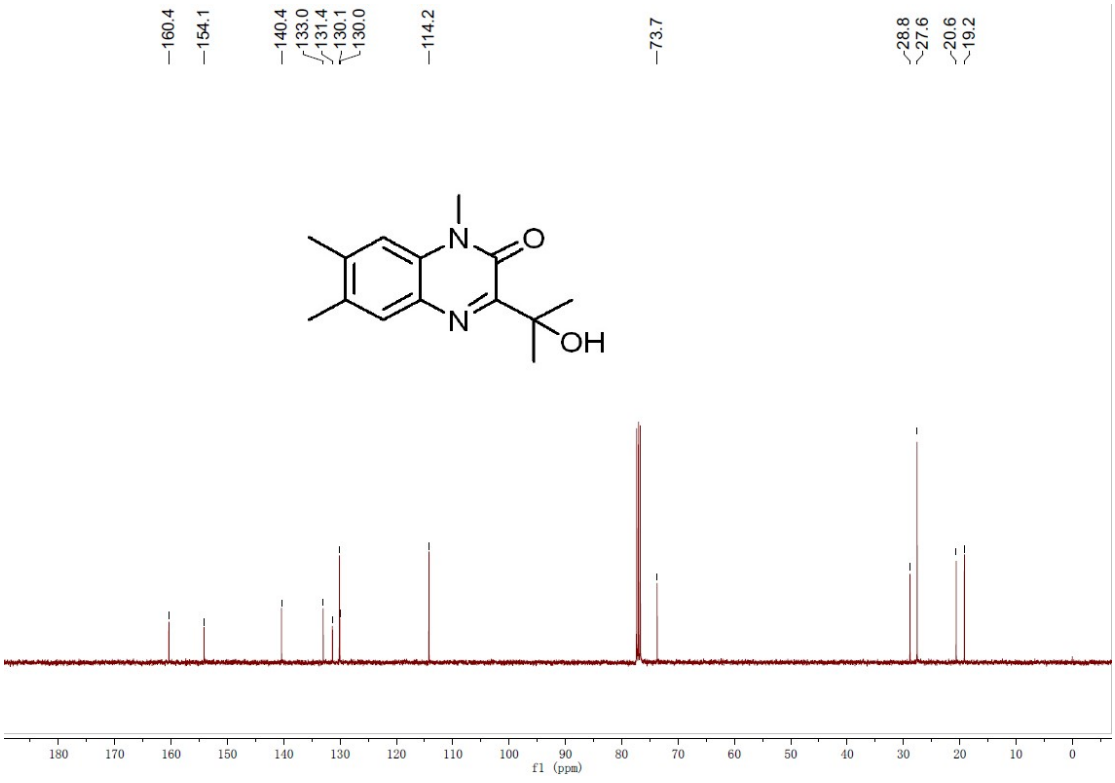
^{1}H NMR Spectrum of Compound 3p



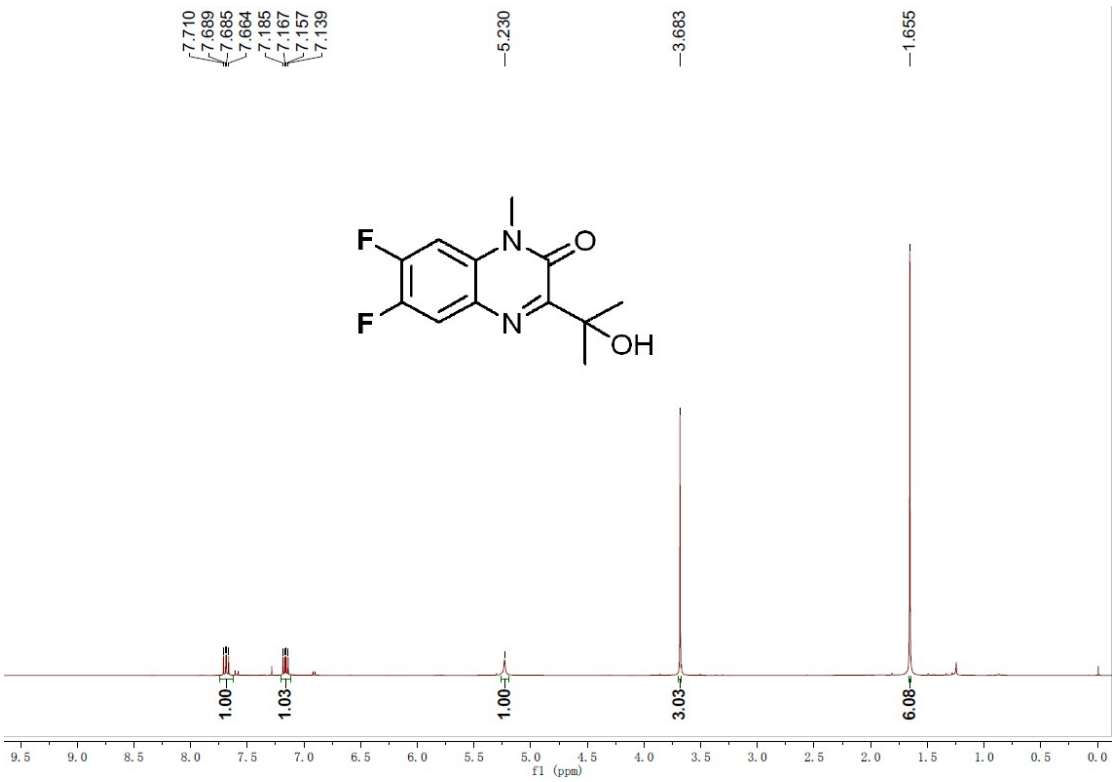
^{13}C NMR Spectrum of Compound 3p



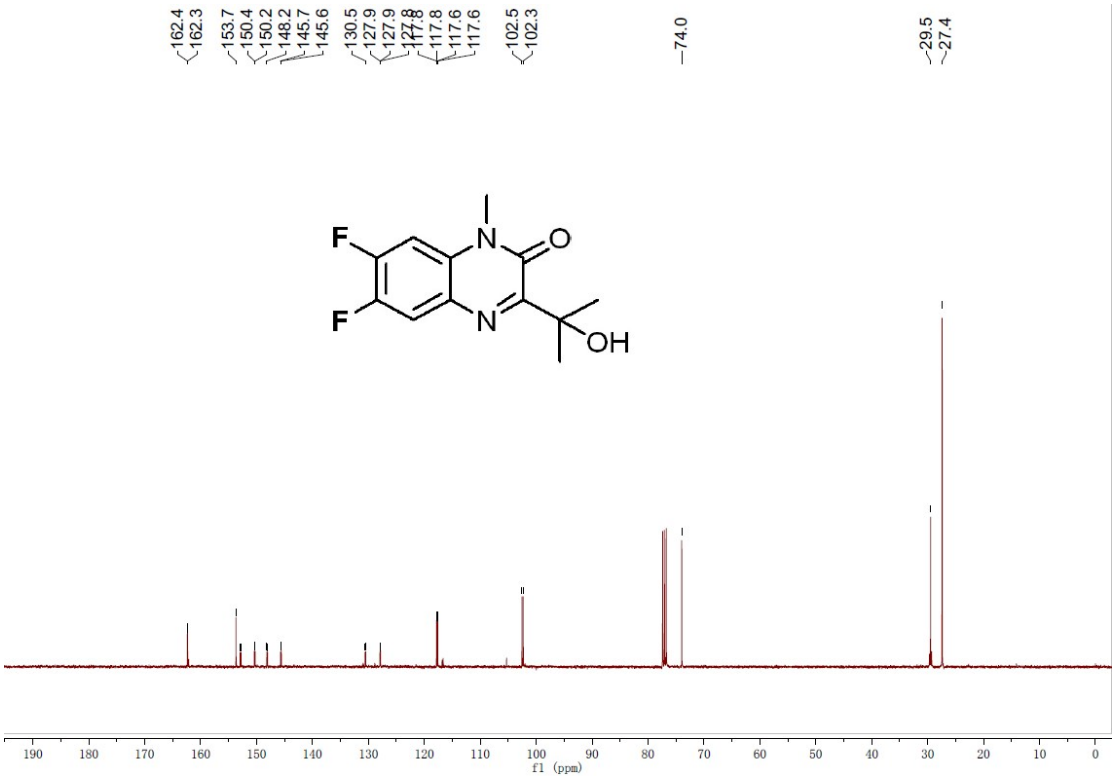
^{1}H NMR Spectrum of Compound 3q



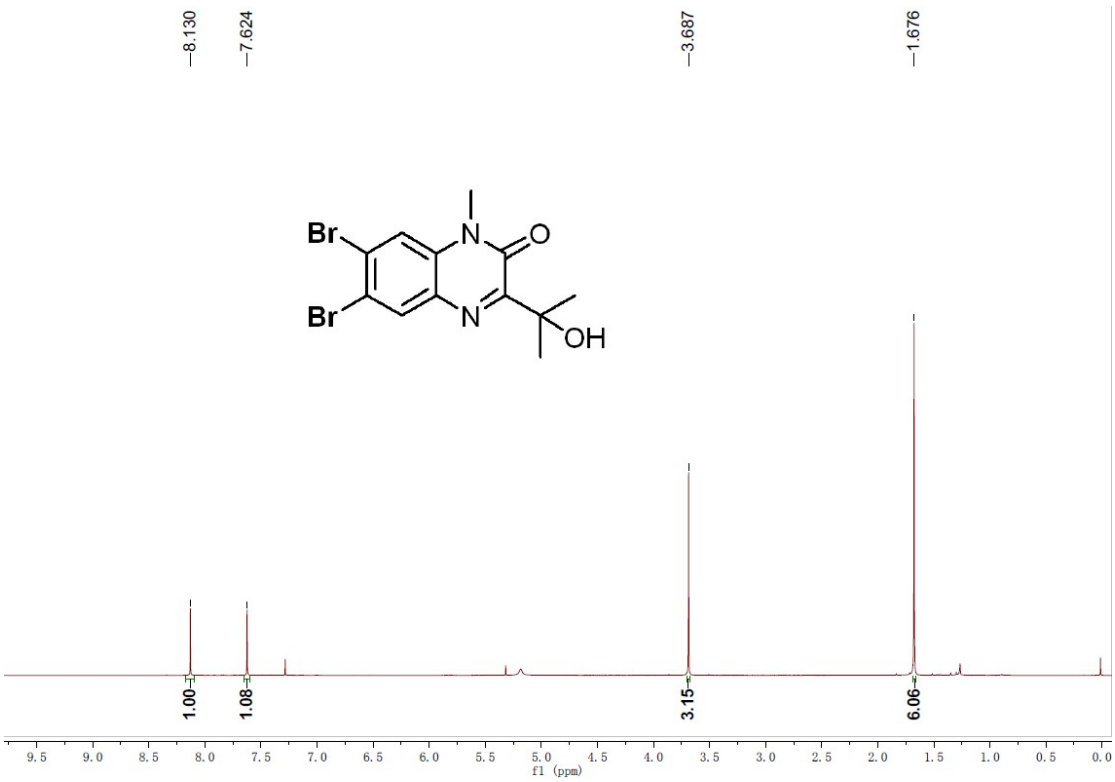
^{13}C NMR Spectrum of Compound 3q



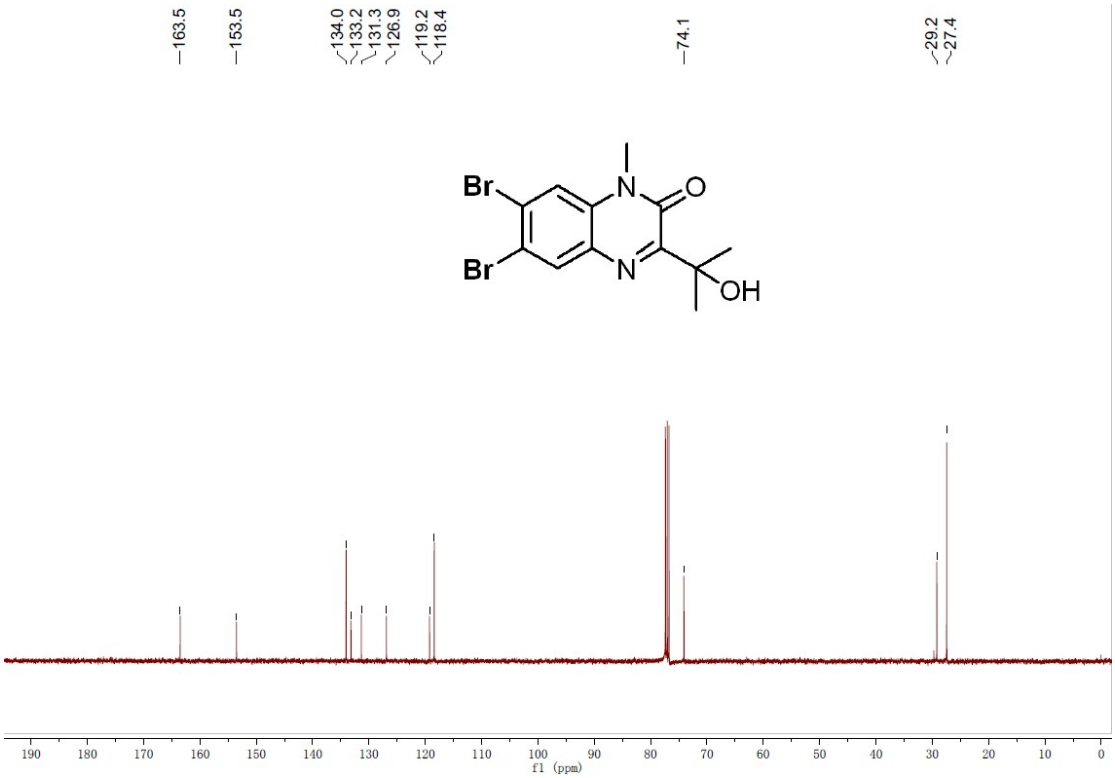
^{1}H NMR Spectrum of Compound 3r



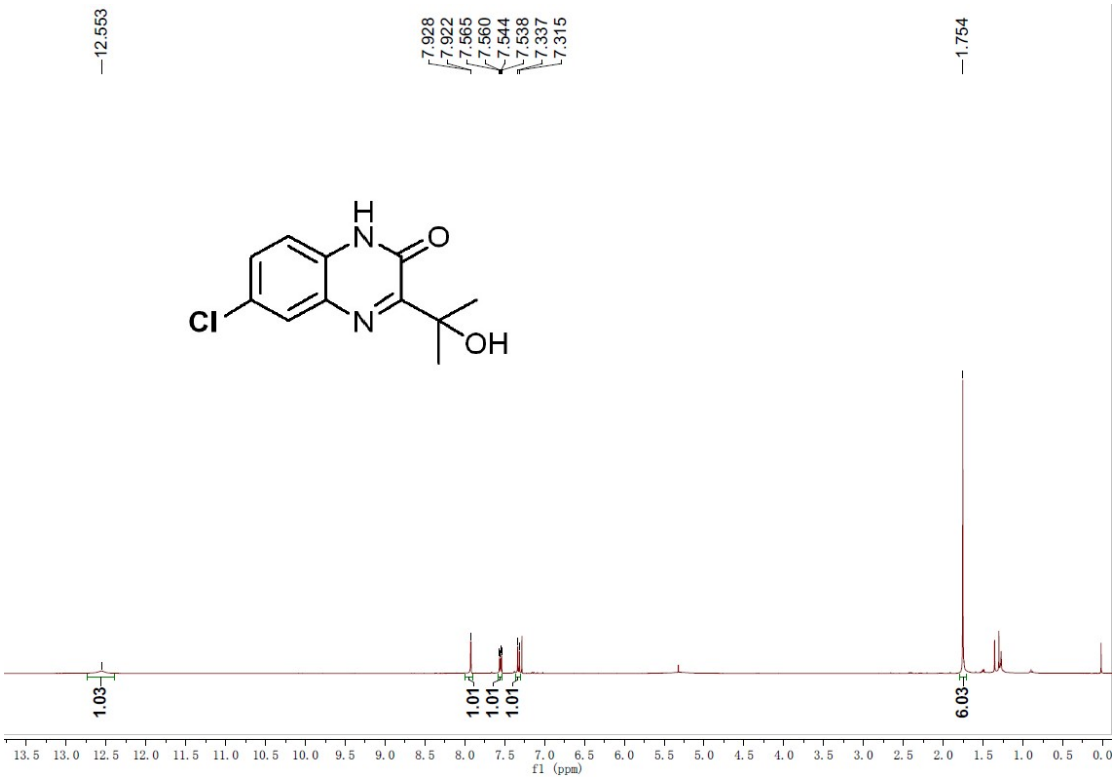
^{13}C NMR Spectrum of Compound 3r



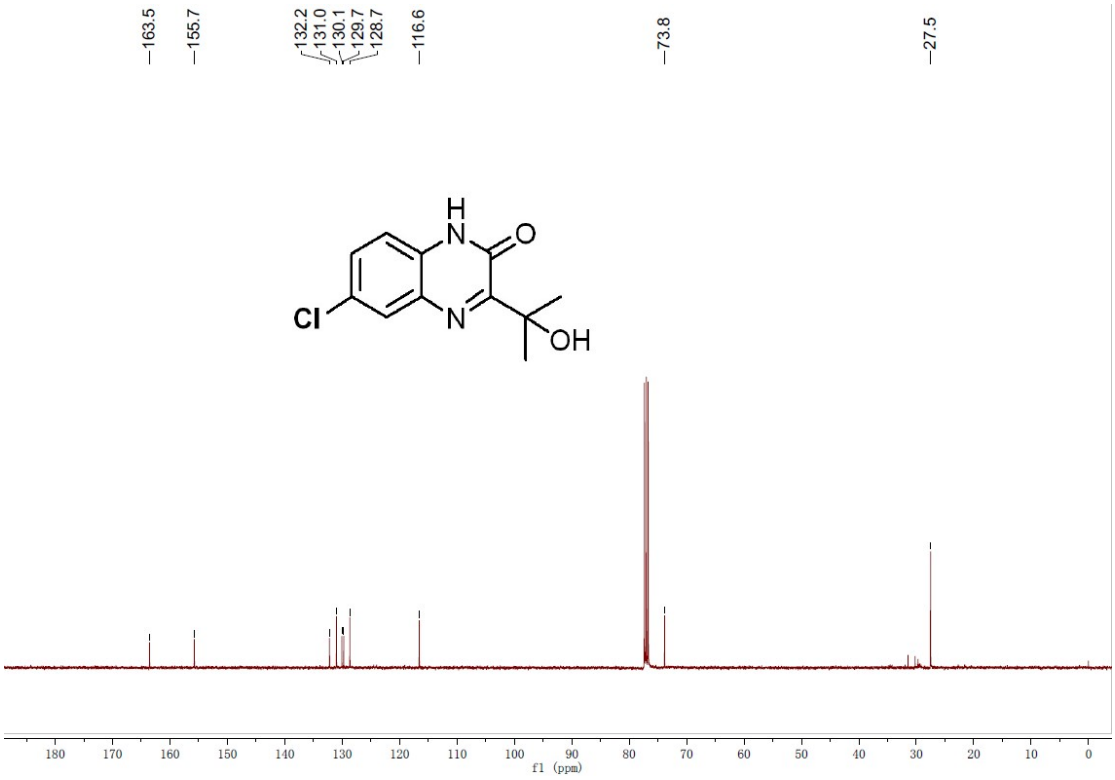
^{1}H NMR Spectrum of Compound 3s



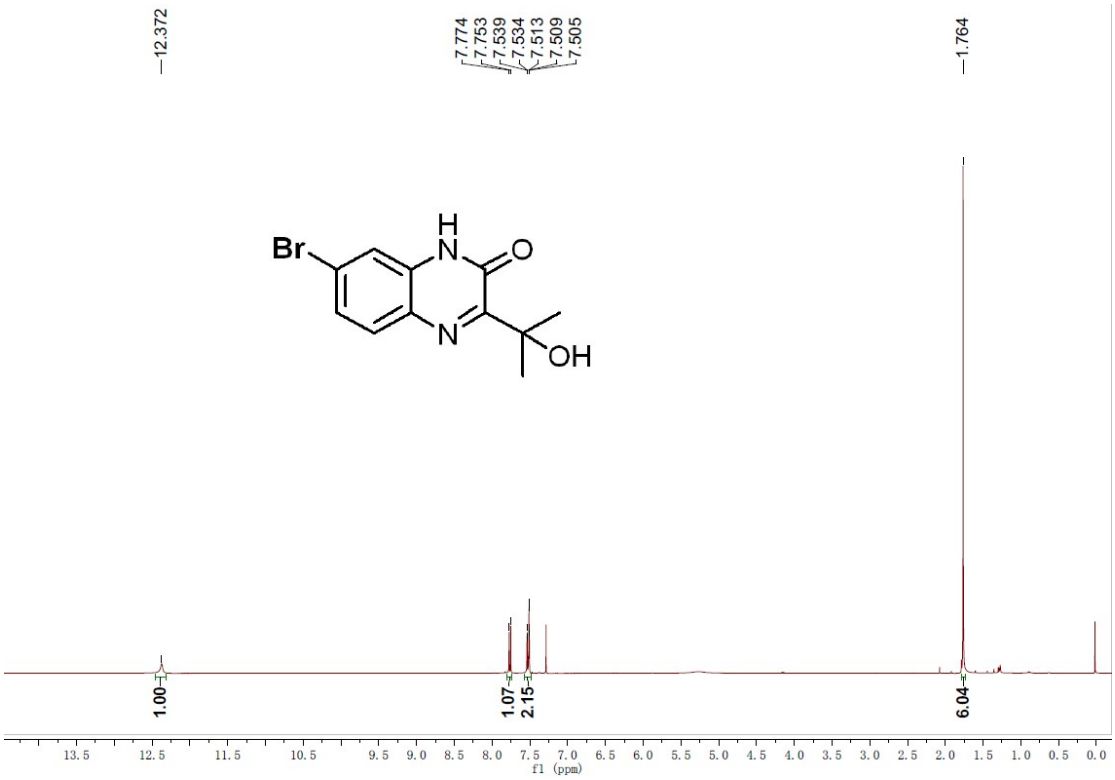
^{13}C NMR Spectrum of Compound 3s



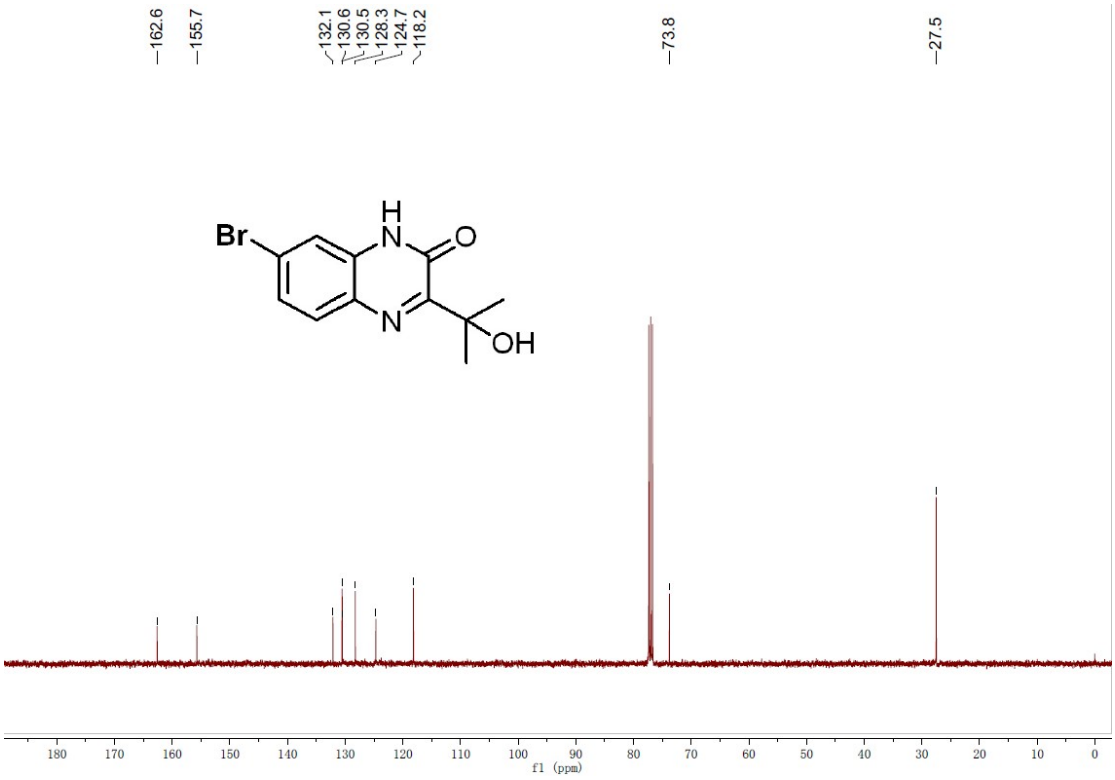
^{1}H NMR Spectrum of Compound 3t



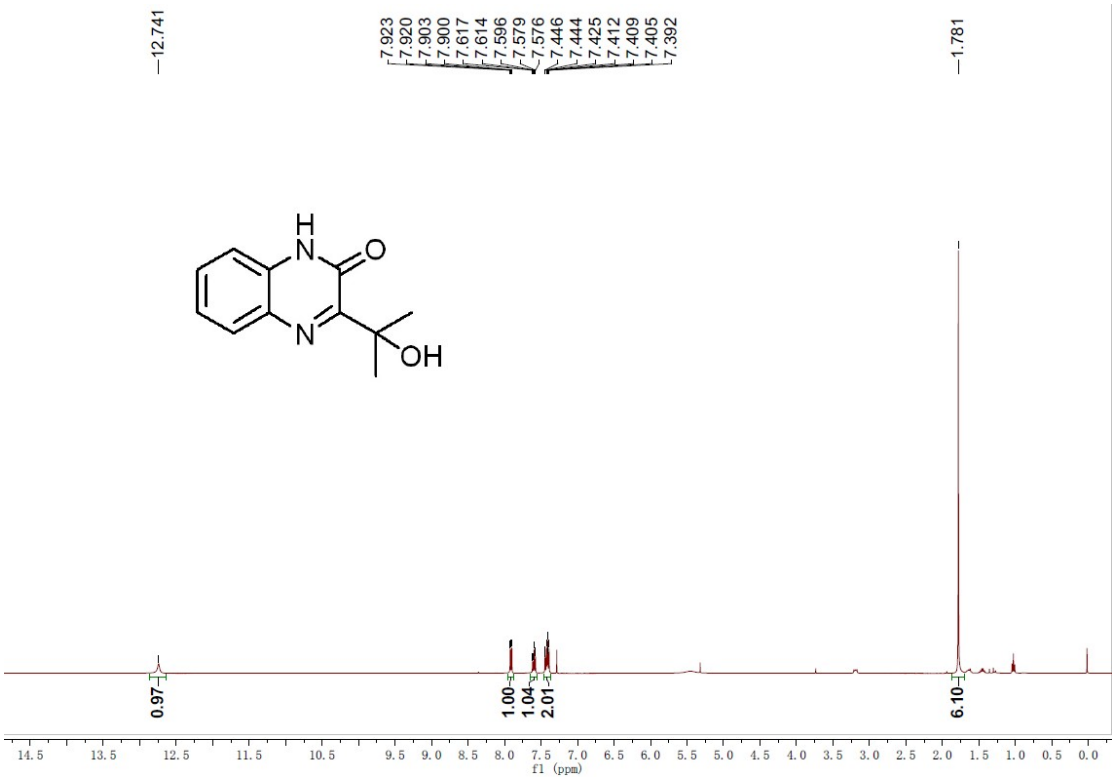
^{13}C NMR Spectrum of Compound 3t



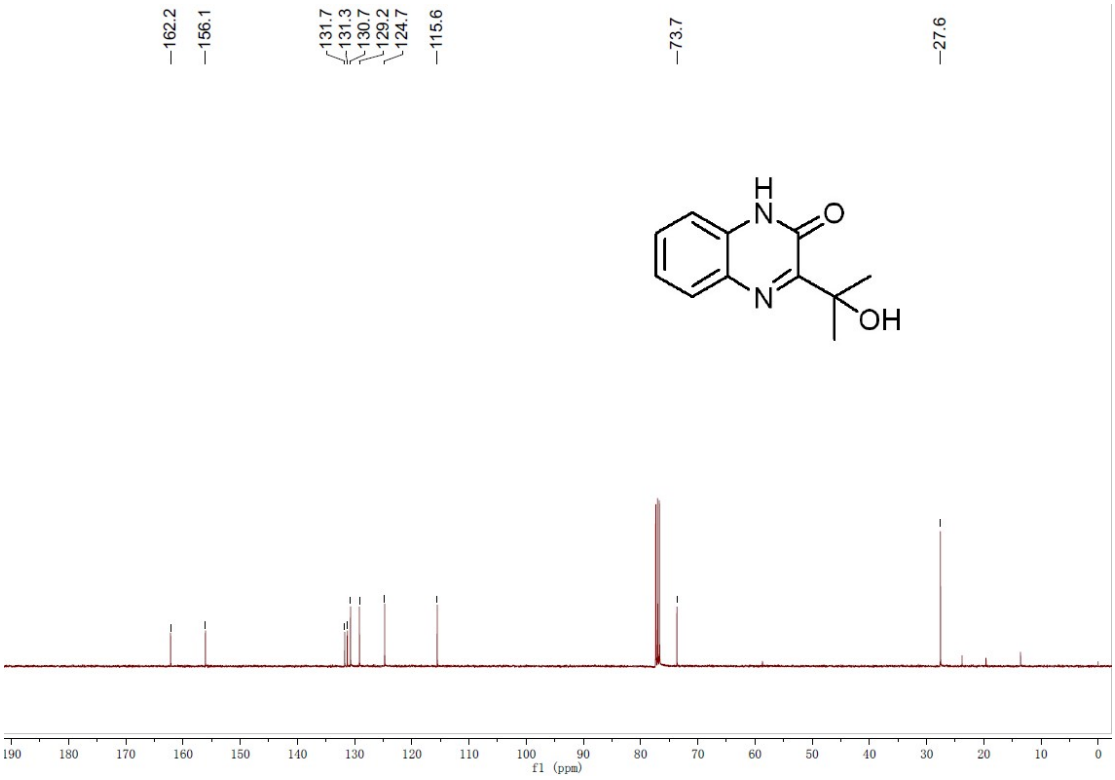
^{1}H NMR Spectrum of Compound 3u



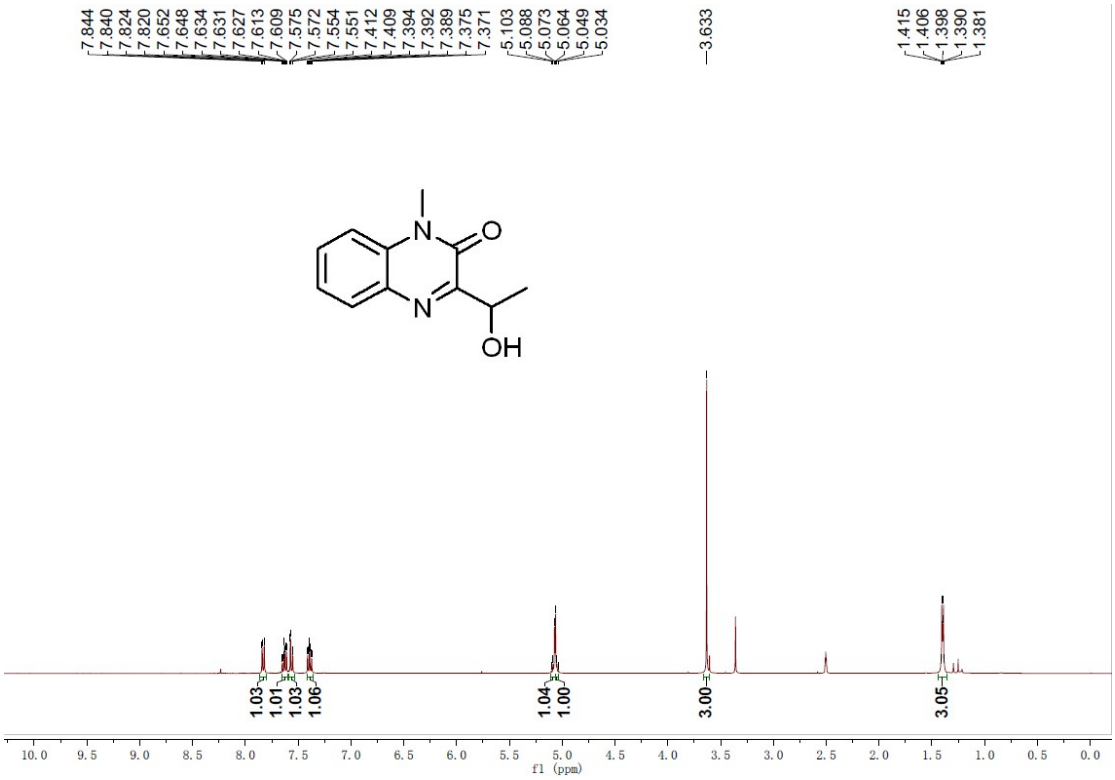
^{13}C NMR Spectrum of Compound 3u



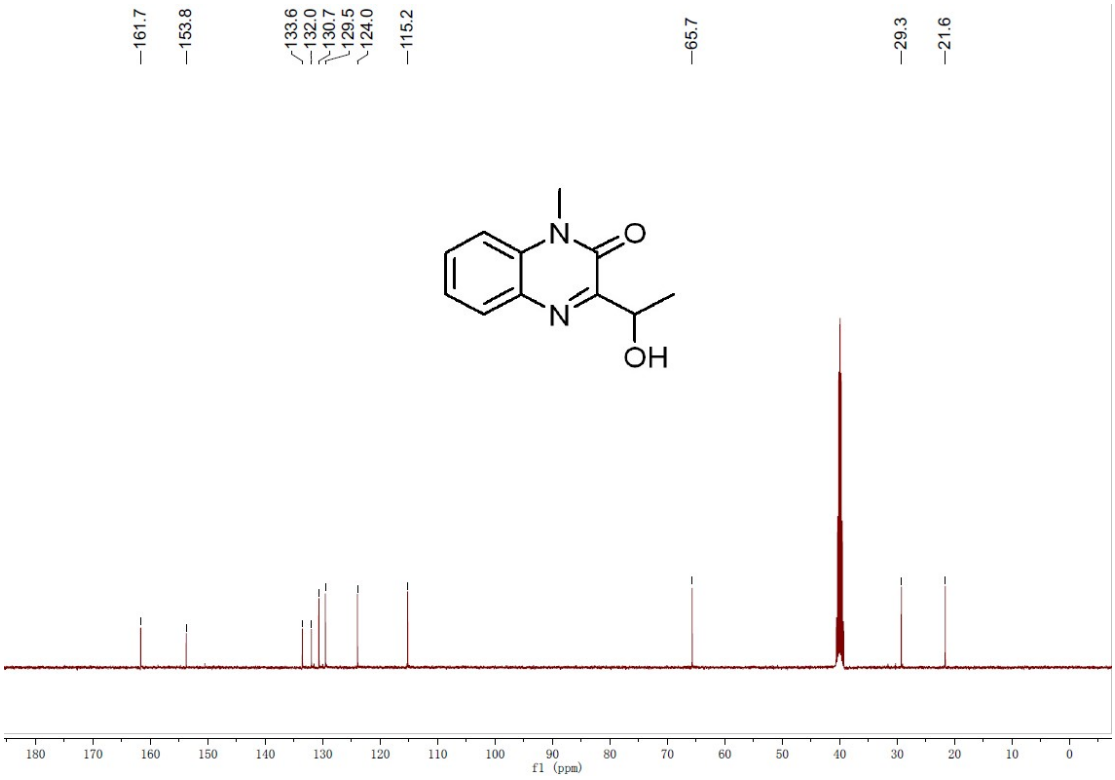
^{1}H NMR Spectrum of Compound 3v



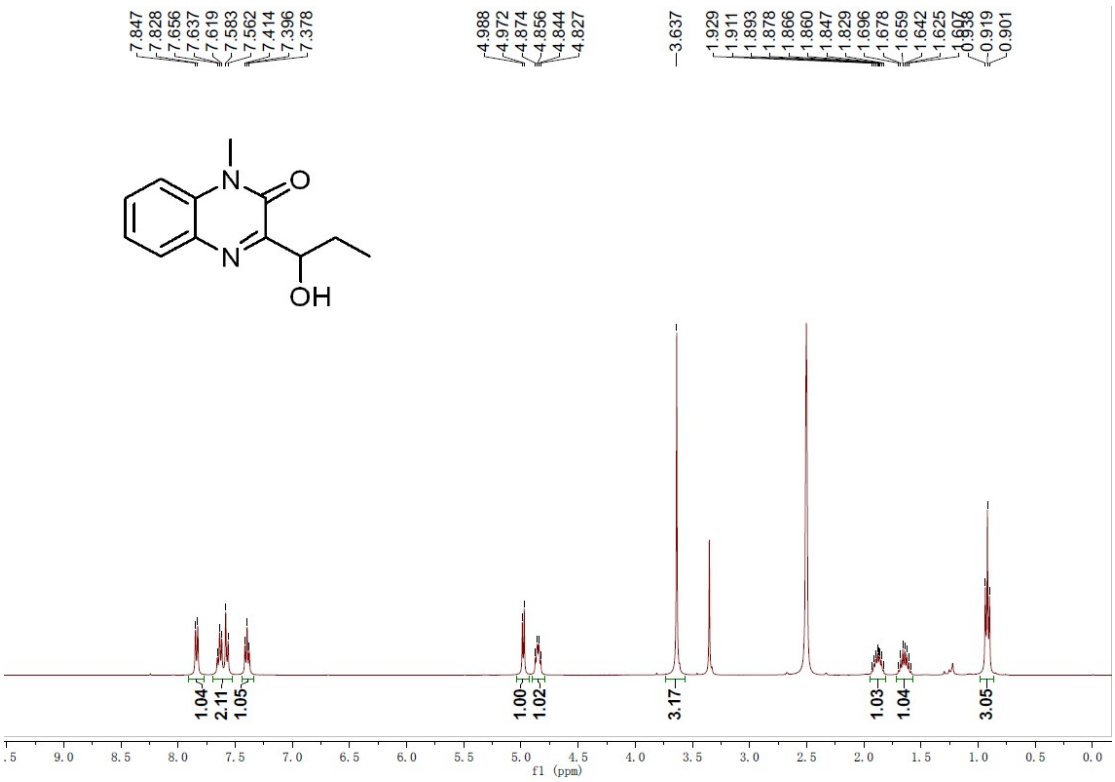
^{13}C NMR Spectrum of Compound 3v



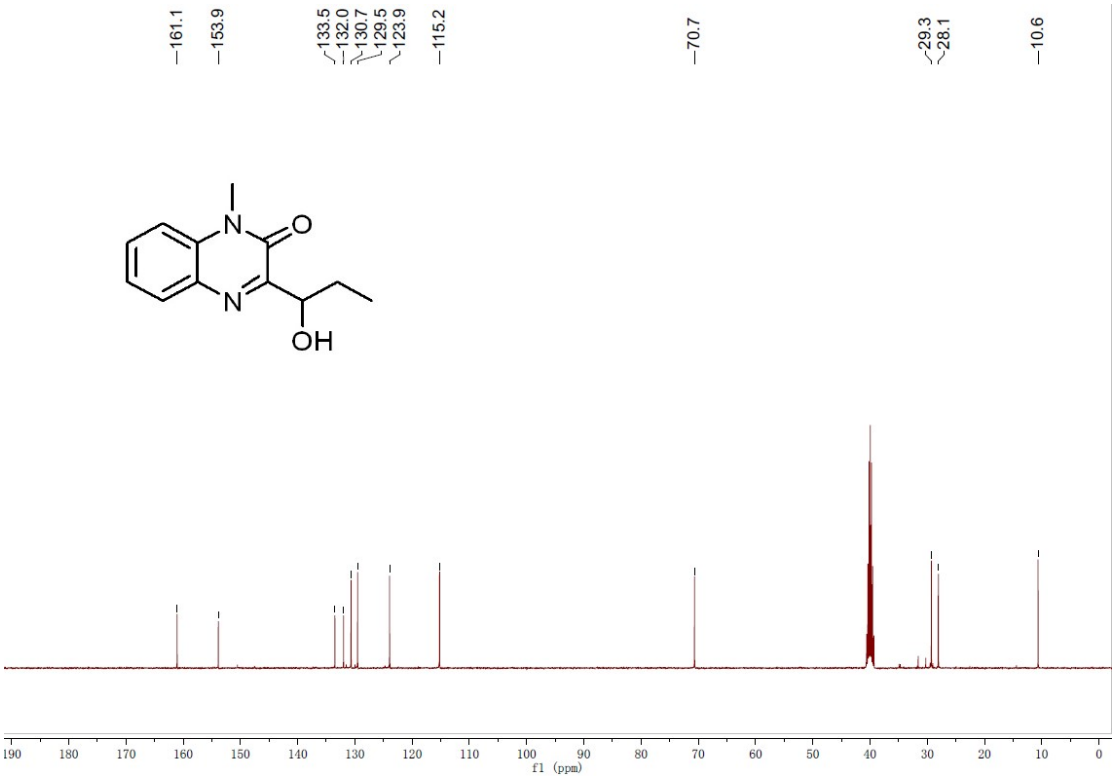
^{1}H NMR Spectrum of Compound 3w



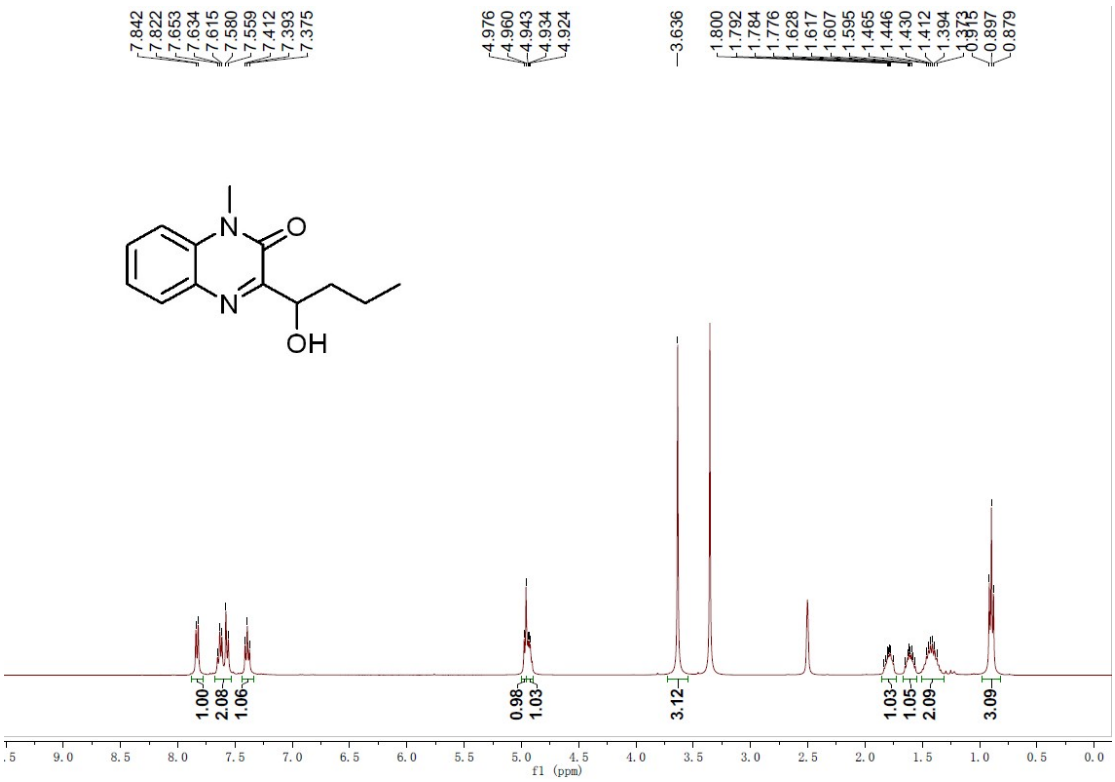
^{13}C NMR Spectrum of Compound 3w



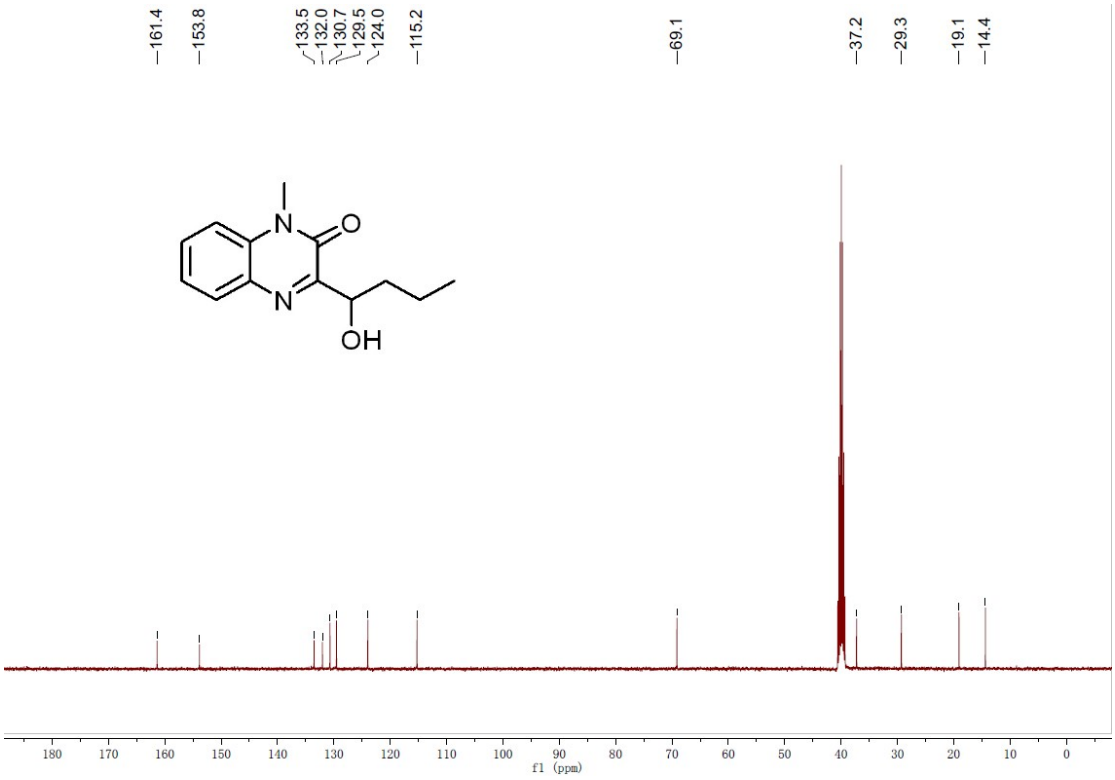
^{1}H NMR Spectrum of Compound 3x



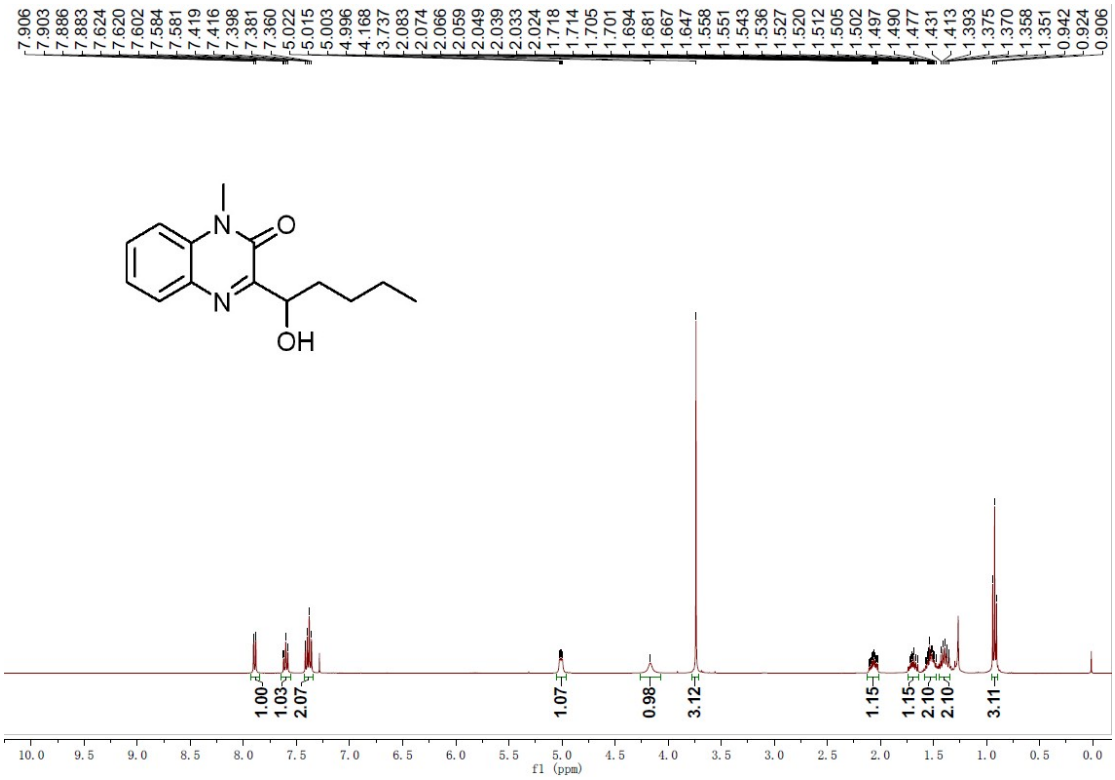
^{13}C NMR Spectrum of Compound 3x



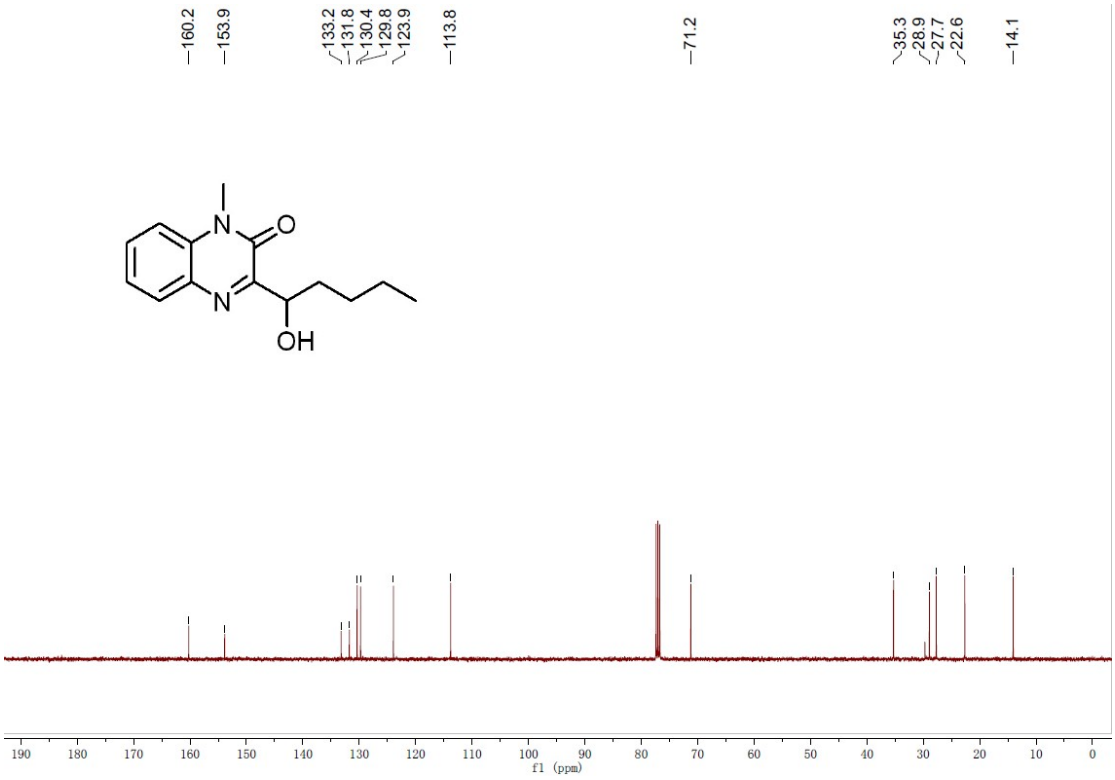
^{1}H NMR Spectrum of Compound 3y



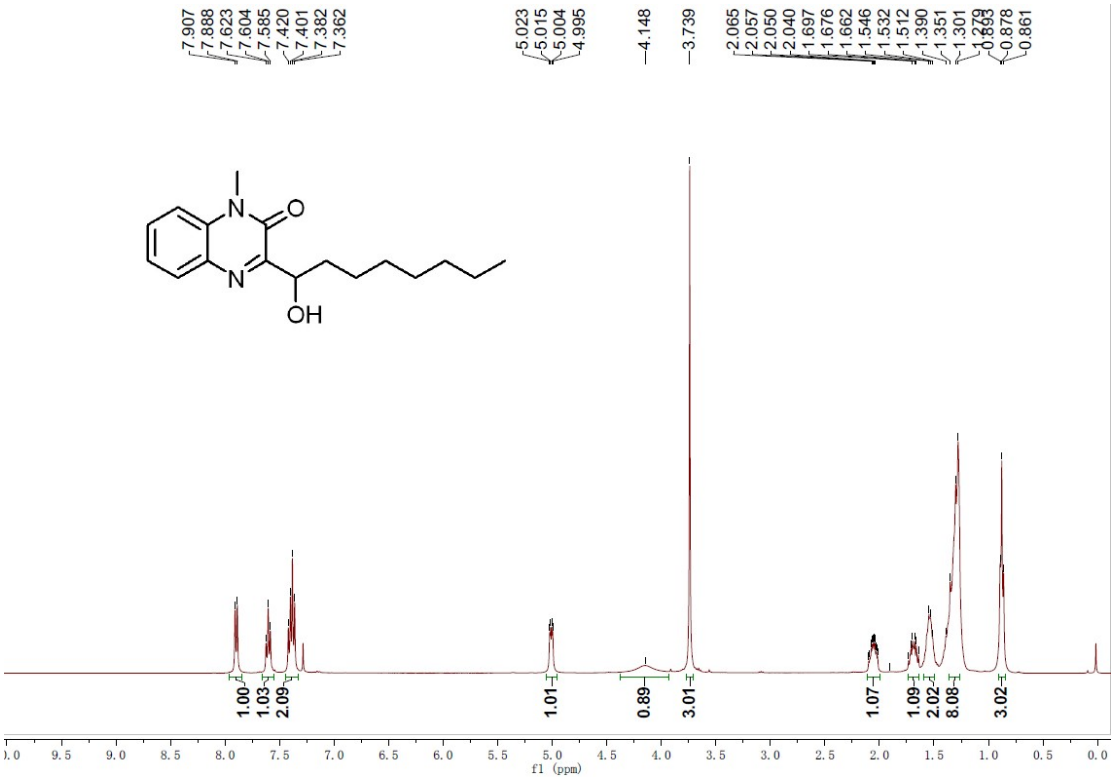
^{13}C NMR Spectrum of Compound 3y



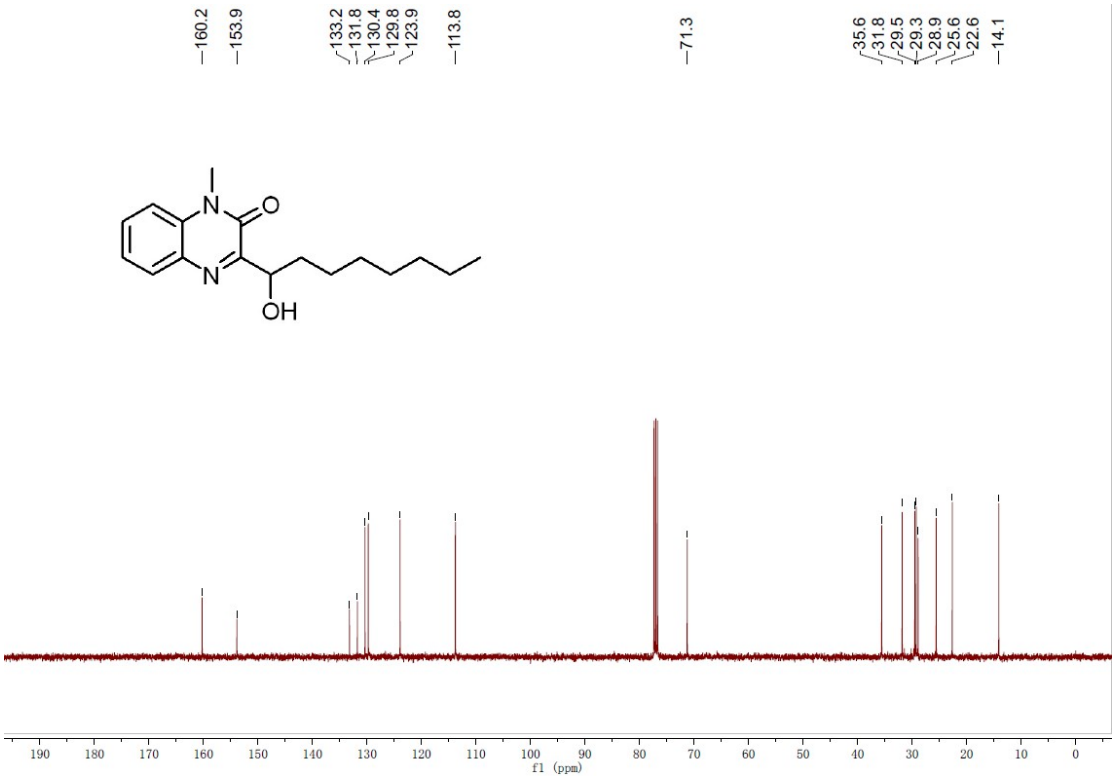
^{1}H NMR Spectrum of Compound 3z



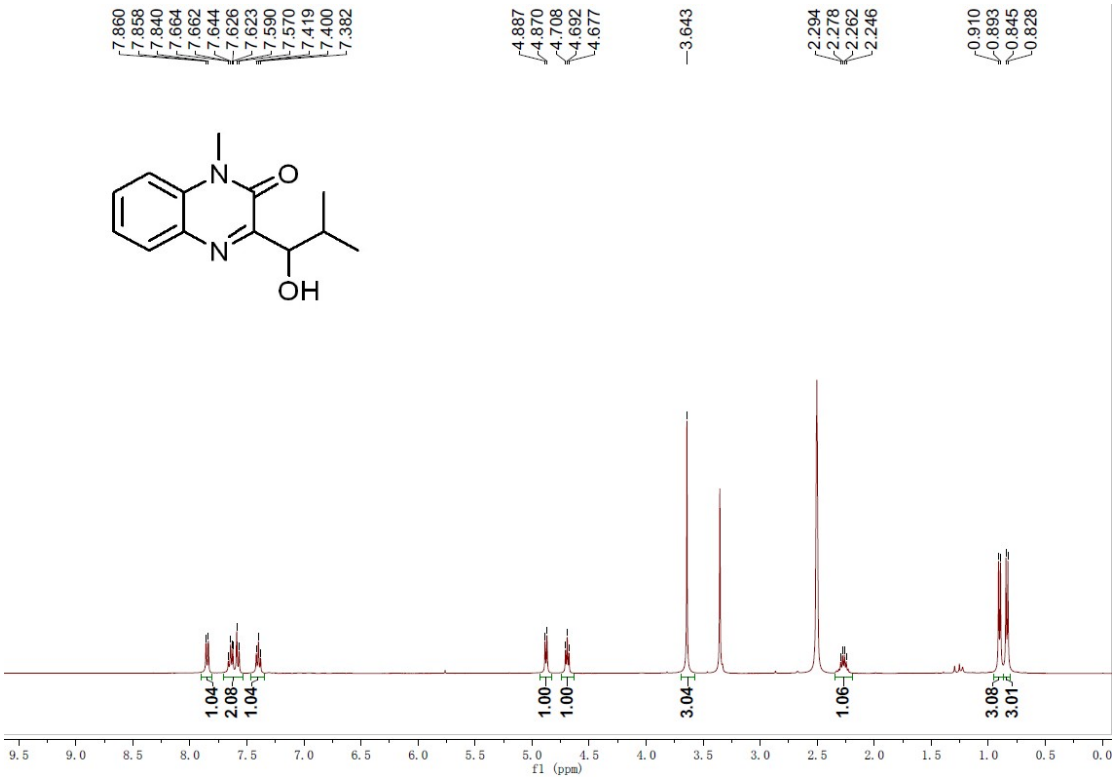
^{13}C NMR Spectrum of Compound 3z



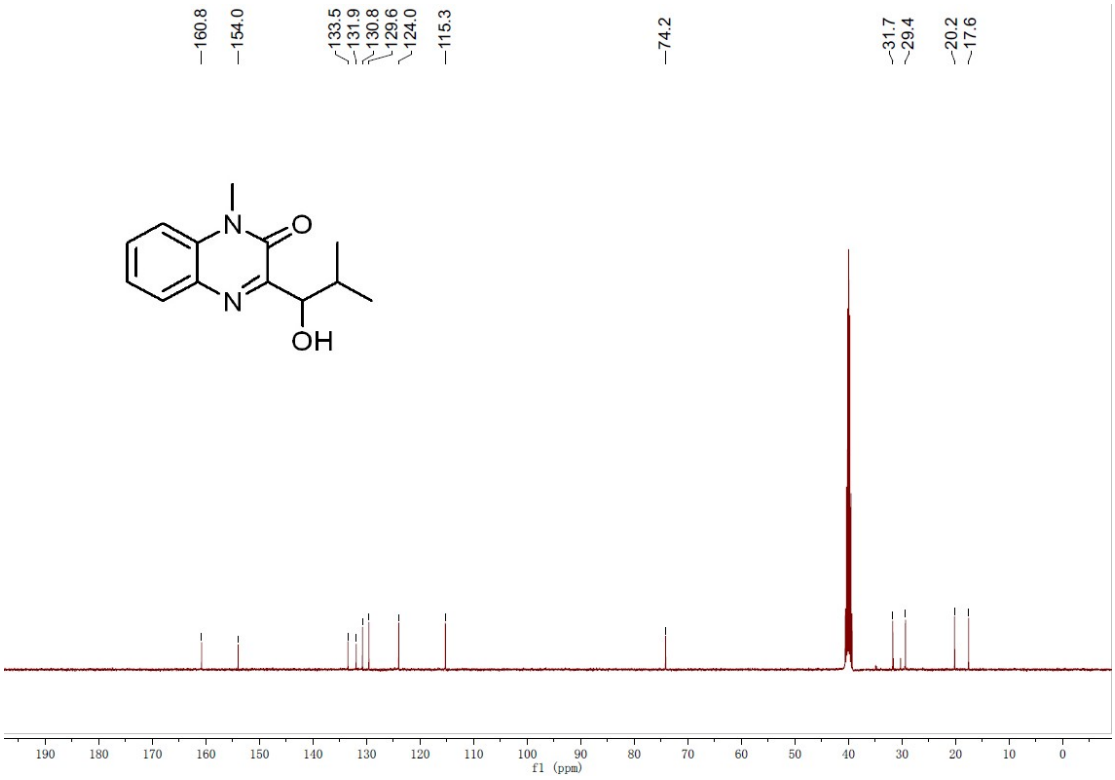
^{1}H NMR Spectrum of Compound 3aa



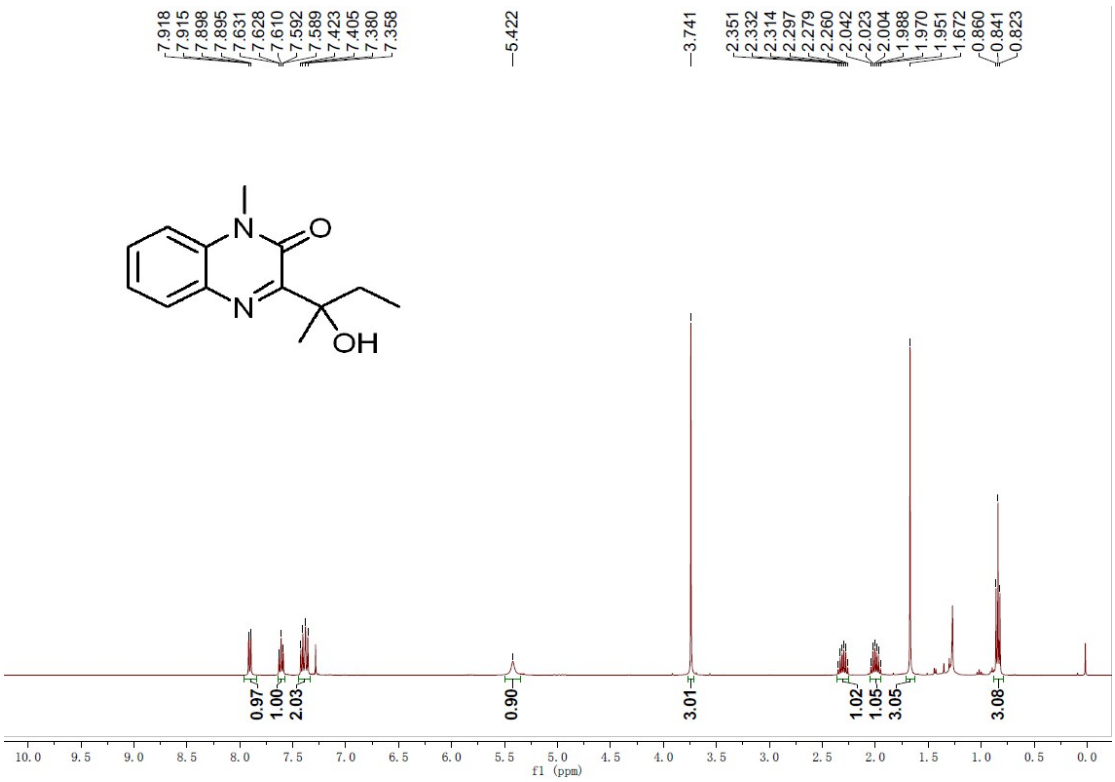
^{13}C NMR Spectrum of Compound 3aa



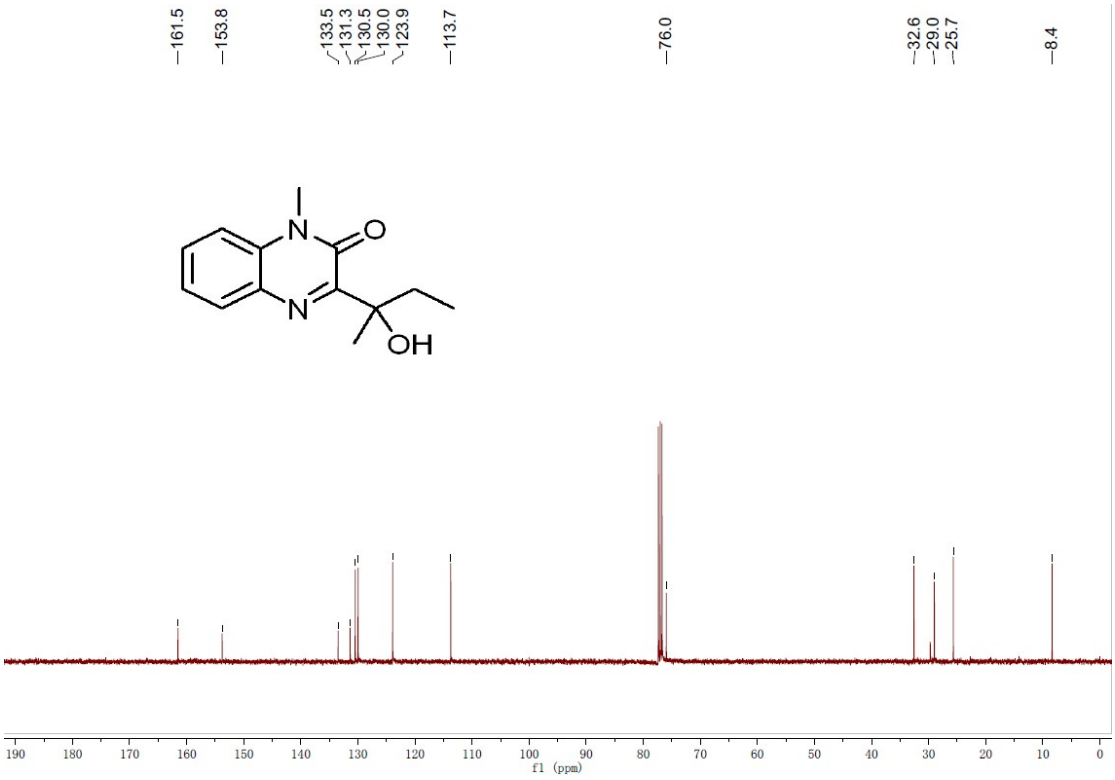
^{1}H NMR Spectrum of Compound 3bb



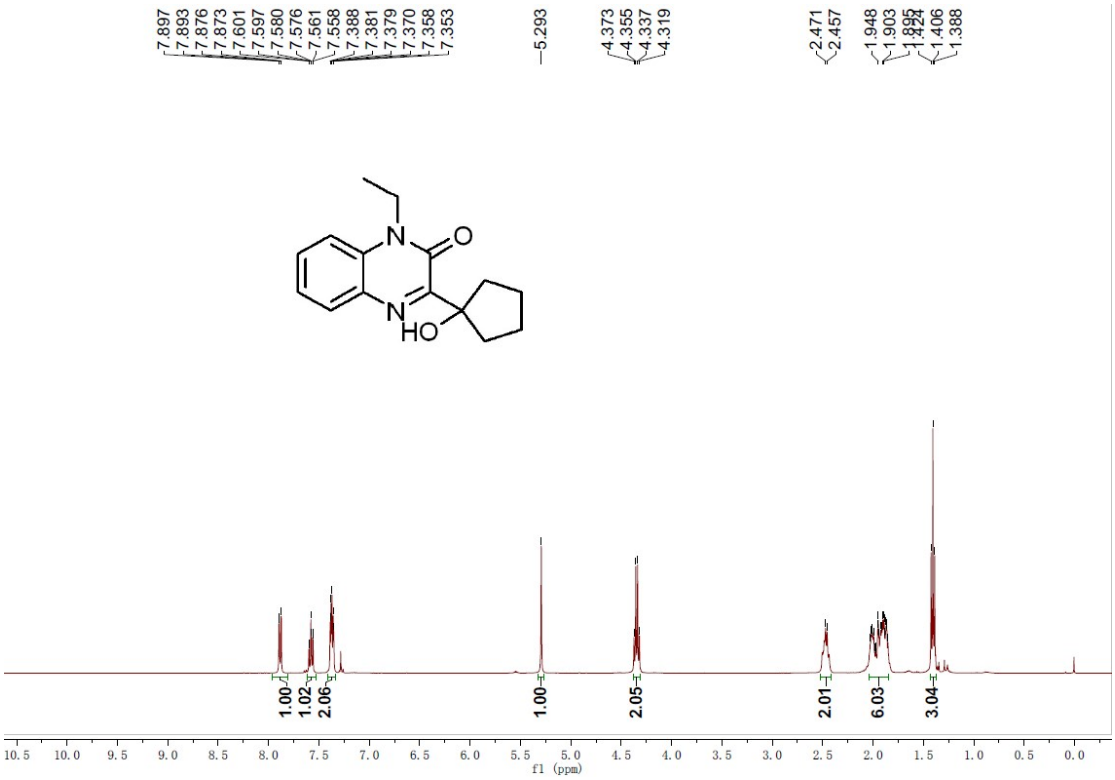
^{13}C NMR Spectrum of Compound 3bb



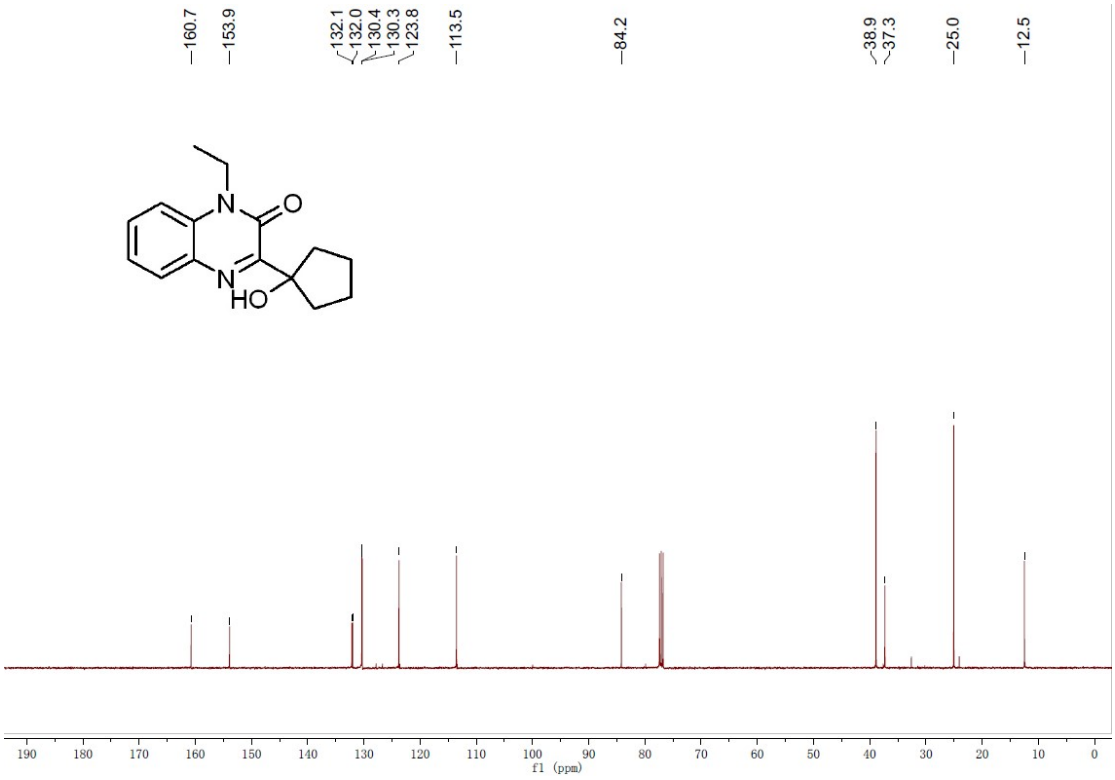
^{1}H NMR Spectrum of Compound 3cc



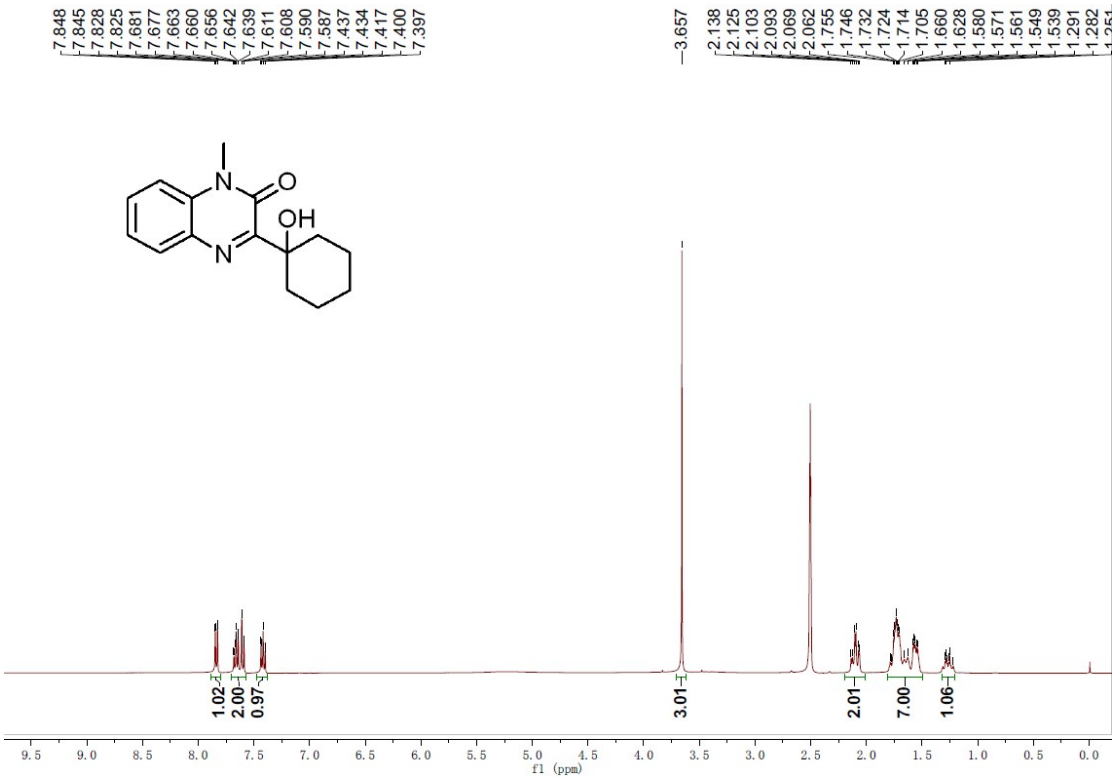
^{13}C NMR Spectrum of Compound 3cc



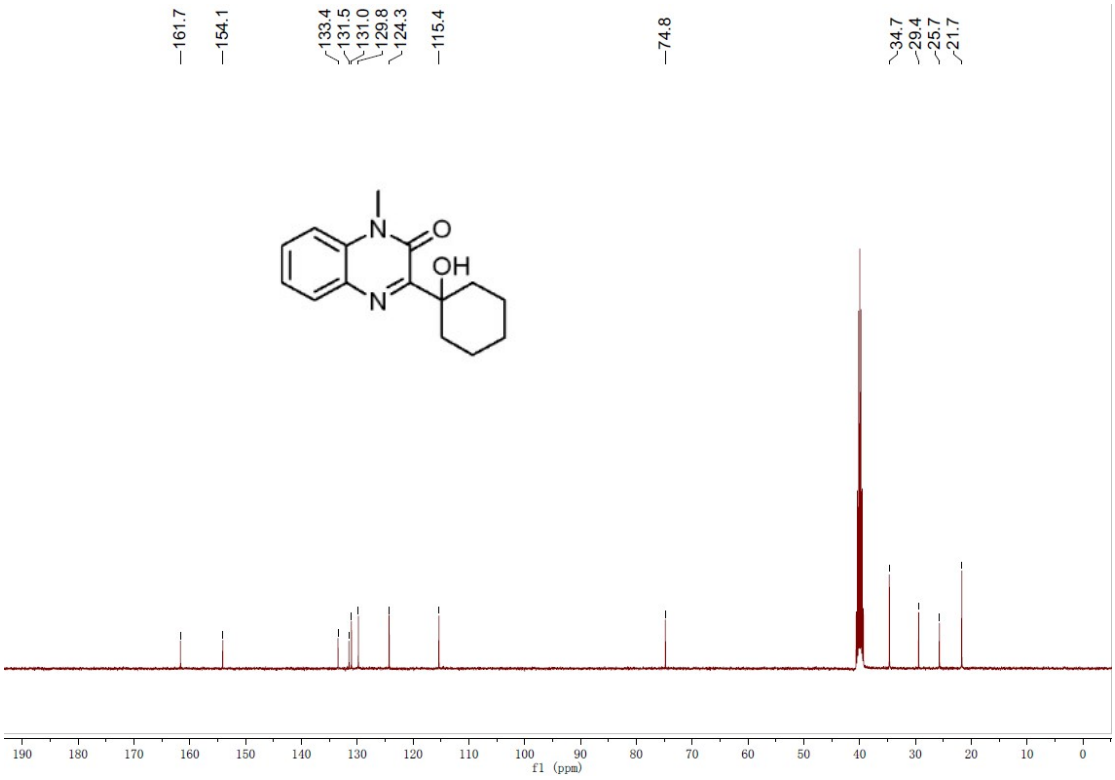
^{1}H NMR Spectrum of Compound 3dd



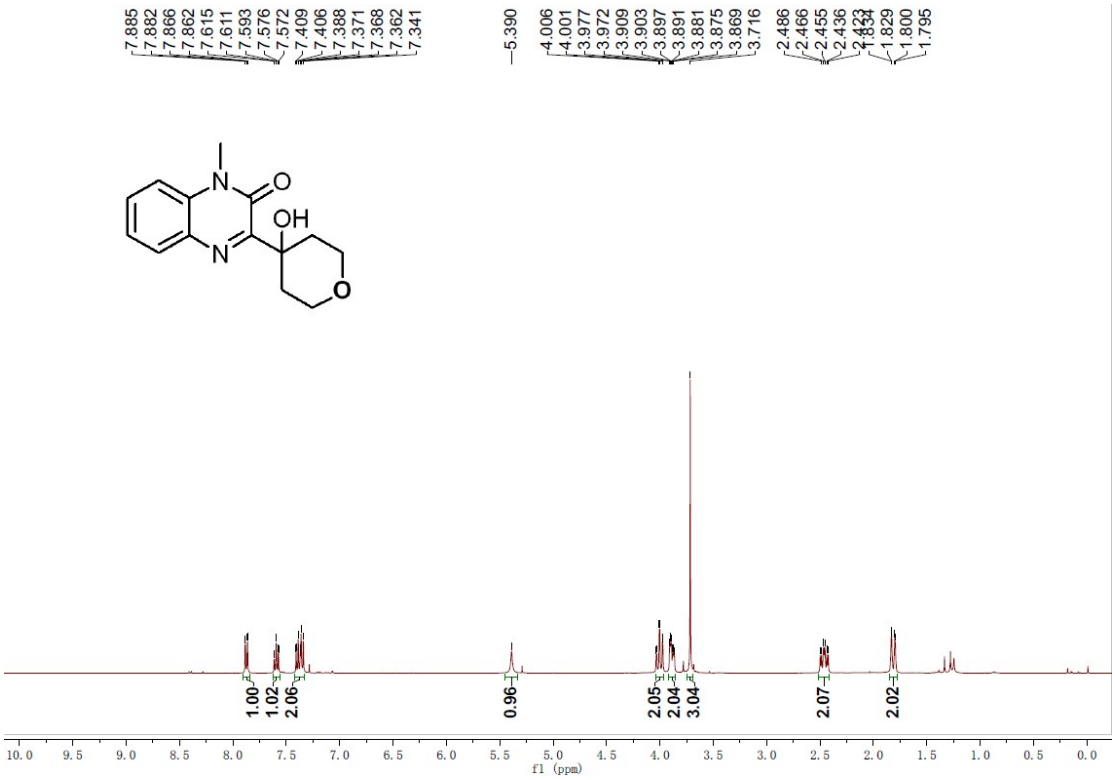
^{13}C NMR Spectrum of Compound 3dd



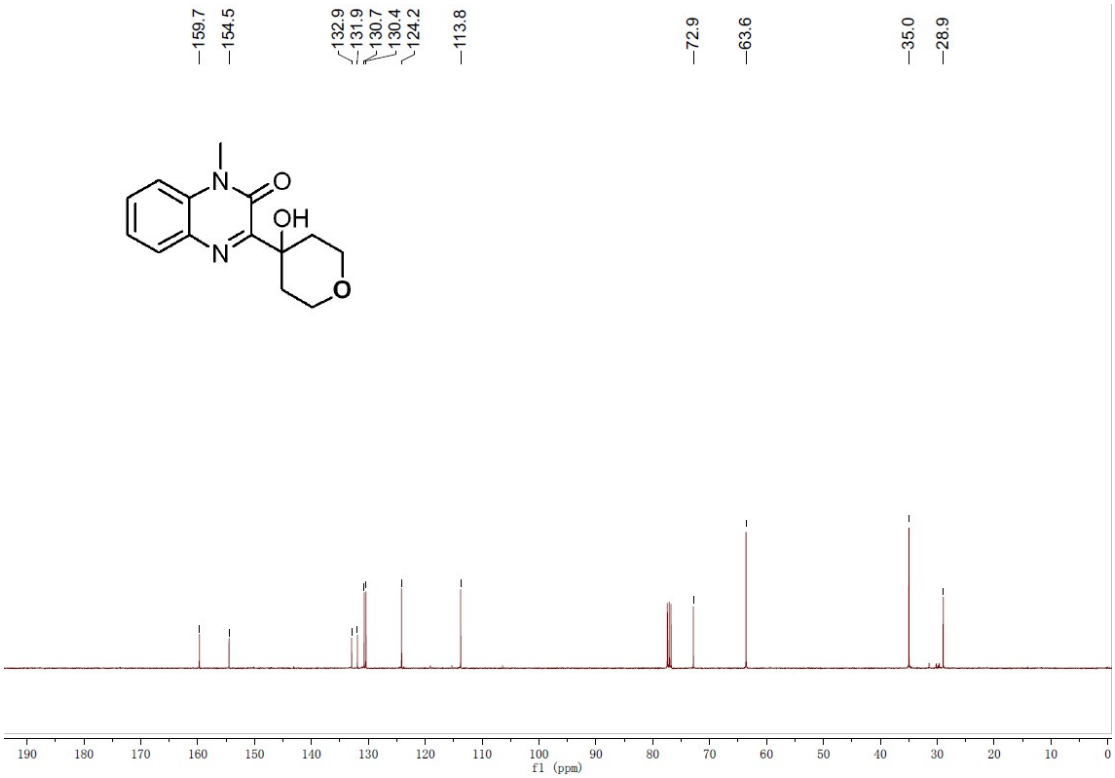
^{1}H NMR Spectrum of Compound 3ee



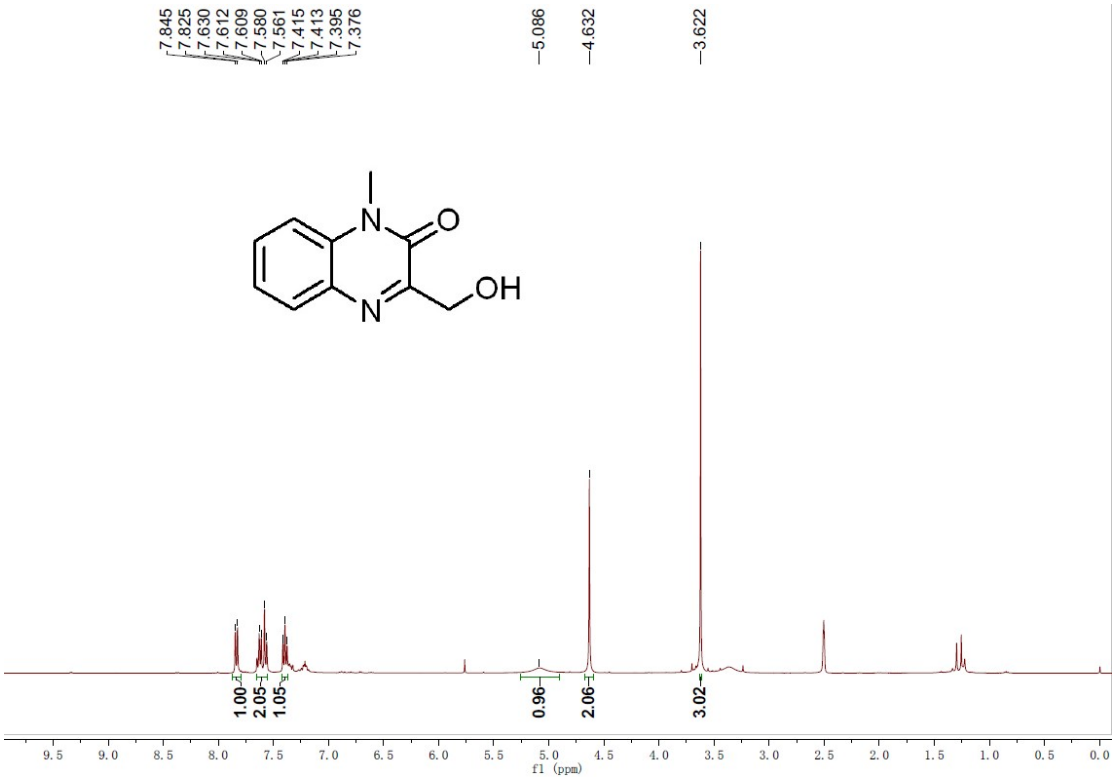
^{13}C NMR Spectrum of Compound 3ee



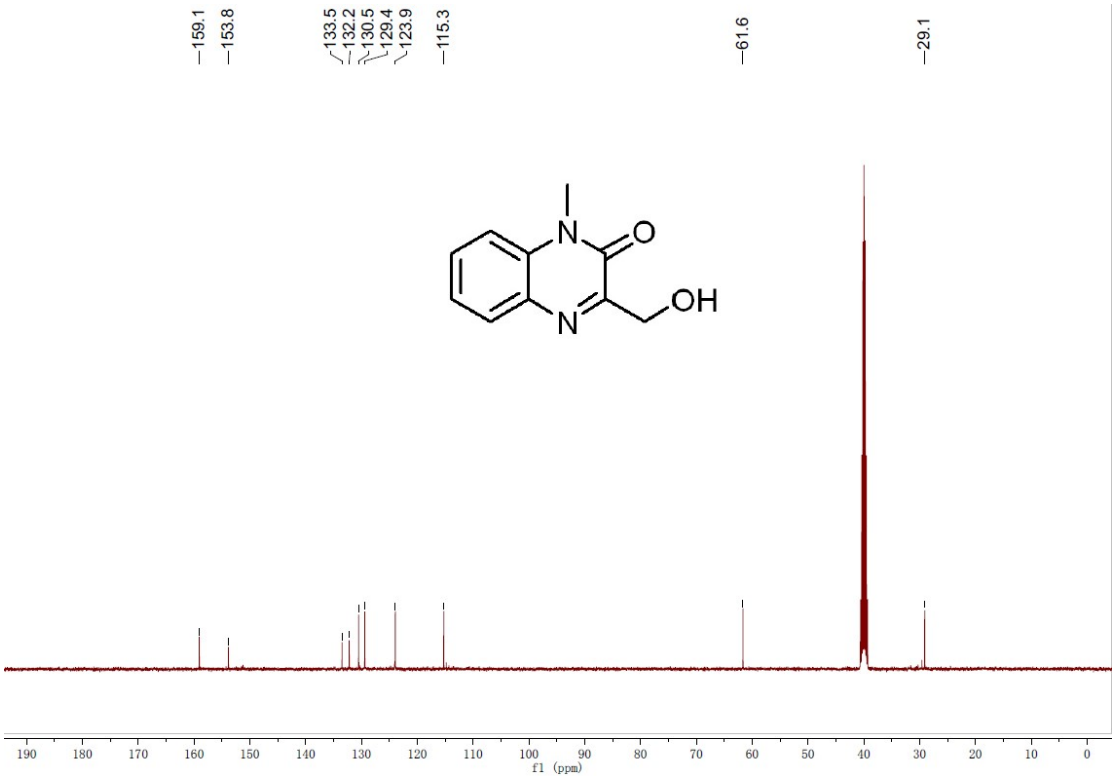
^{1}H NMR Spectrum of Compound 3ff



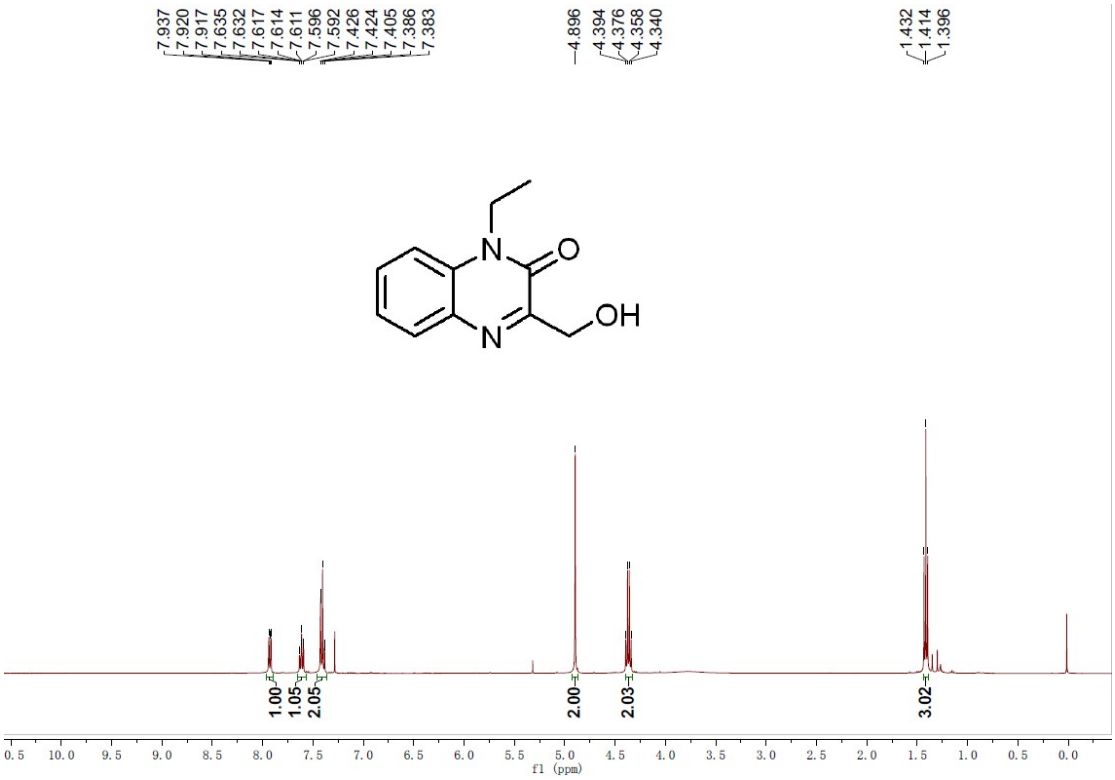
^{13}C NMR Spectrum of Compound 3ff



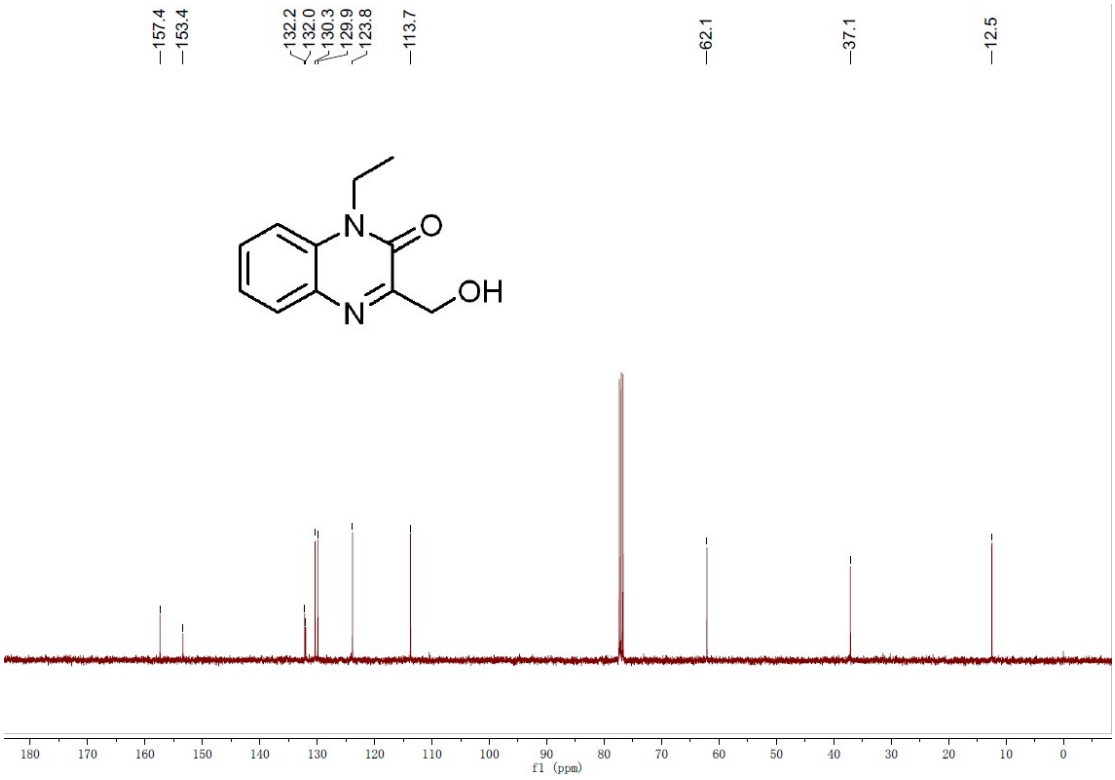
^{1}H NMR Spectrum of Compound 3gg



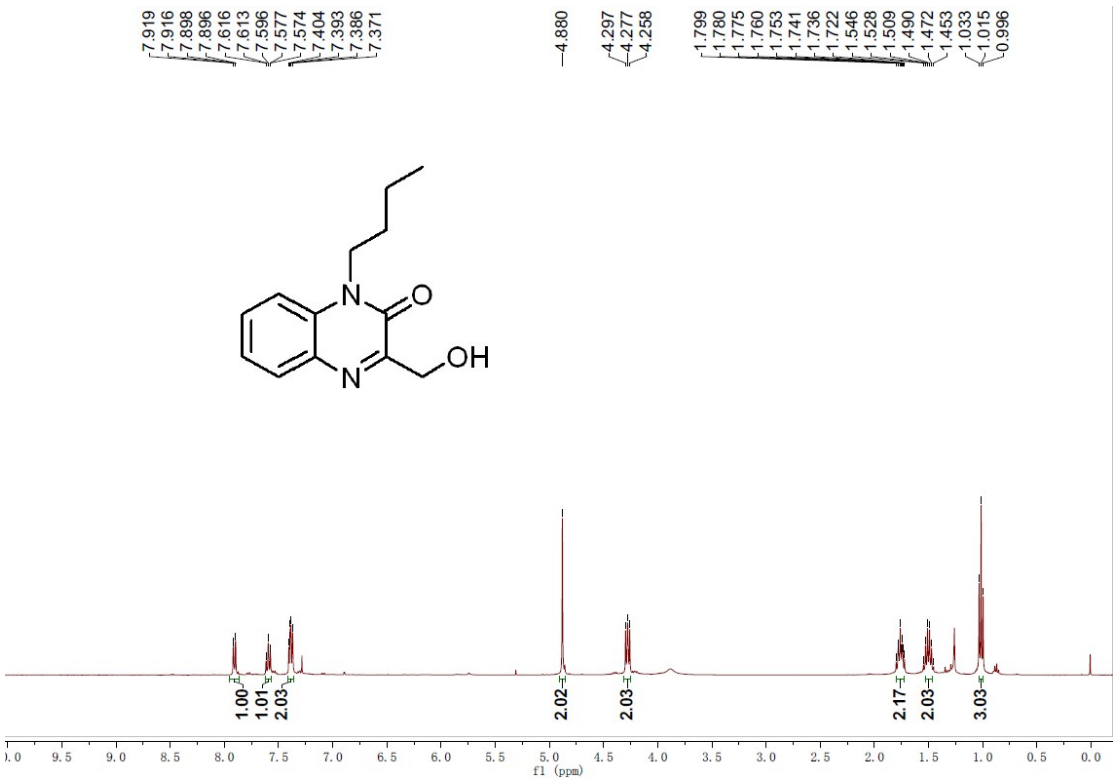
^{13}C NMR Spectrum of Compound 3gg



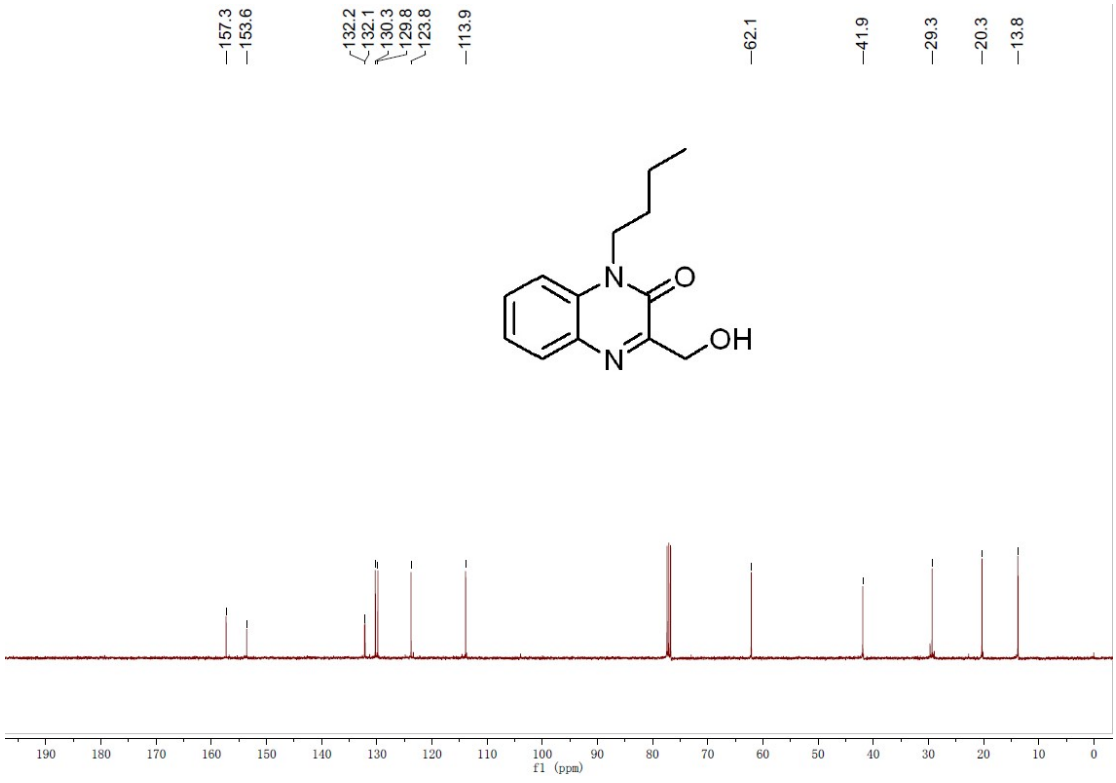
^{1}H NMR Spectrum of Compound 3hh



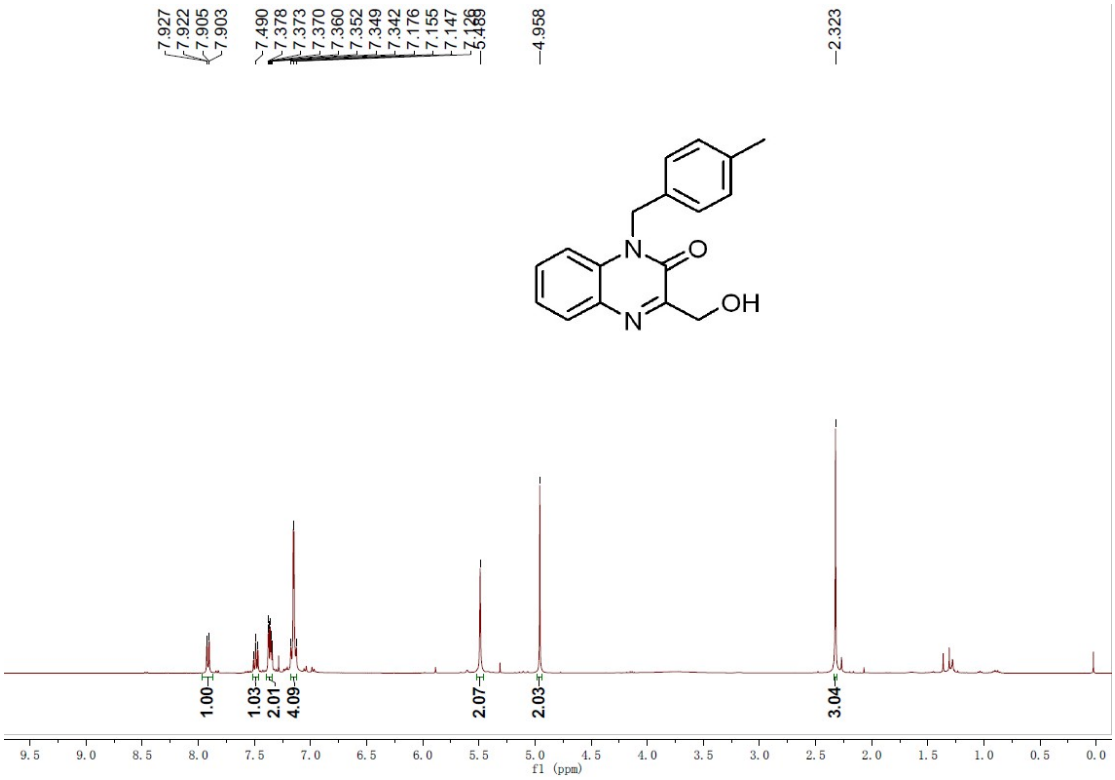
^{13}C NMR Spectrum of Compound 3hh



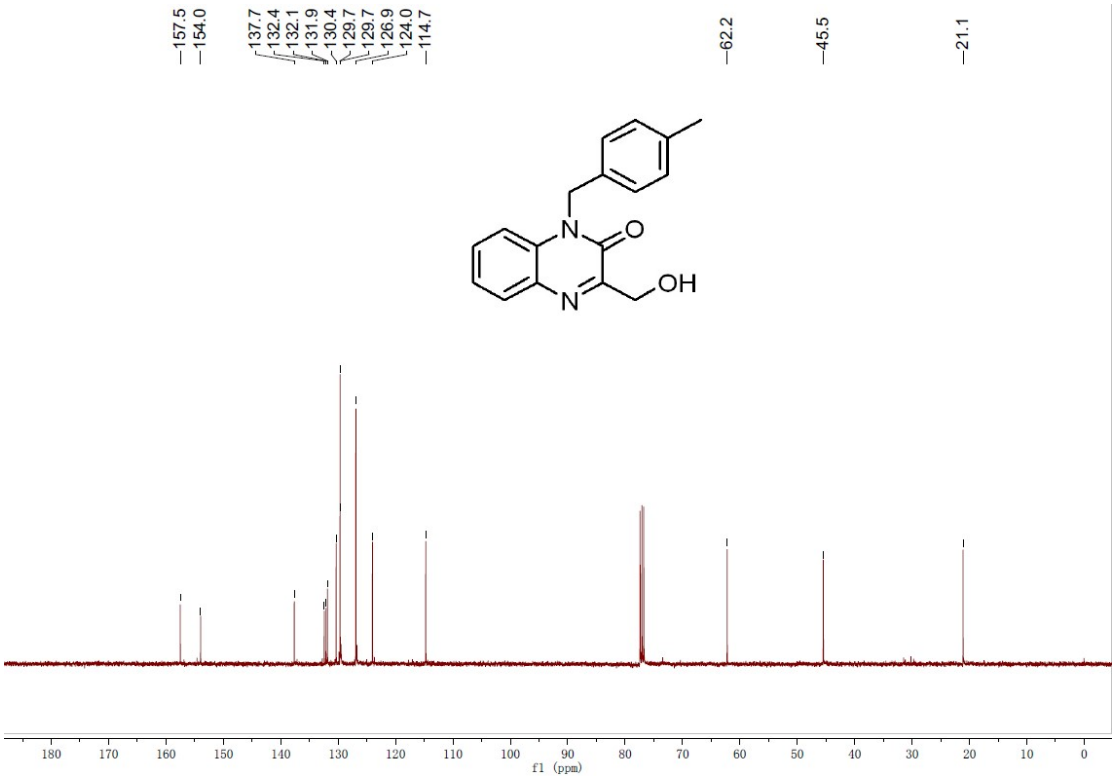
^{1}H NMR Spectrum of Compound 3ii



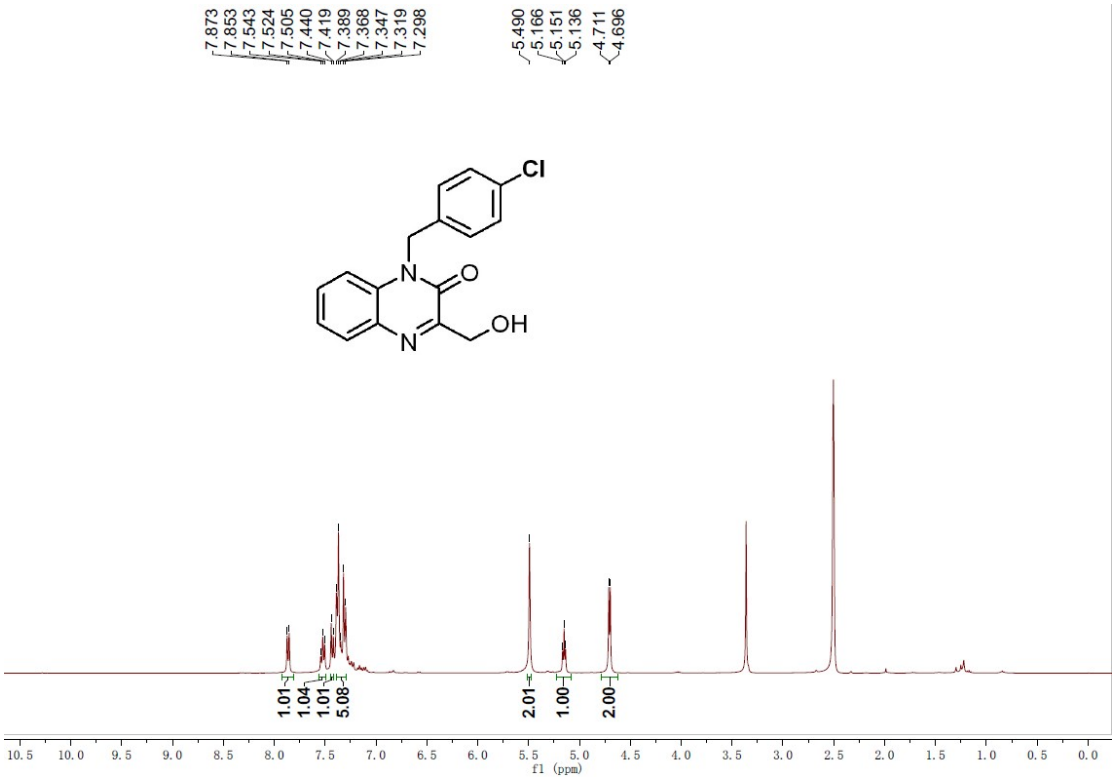
^{13}C NMR Spectrum of Compound 3ii



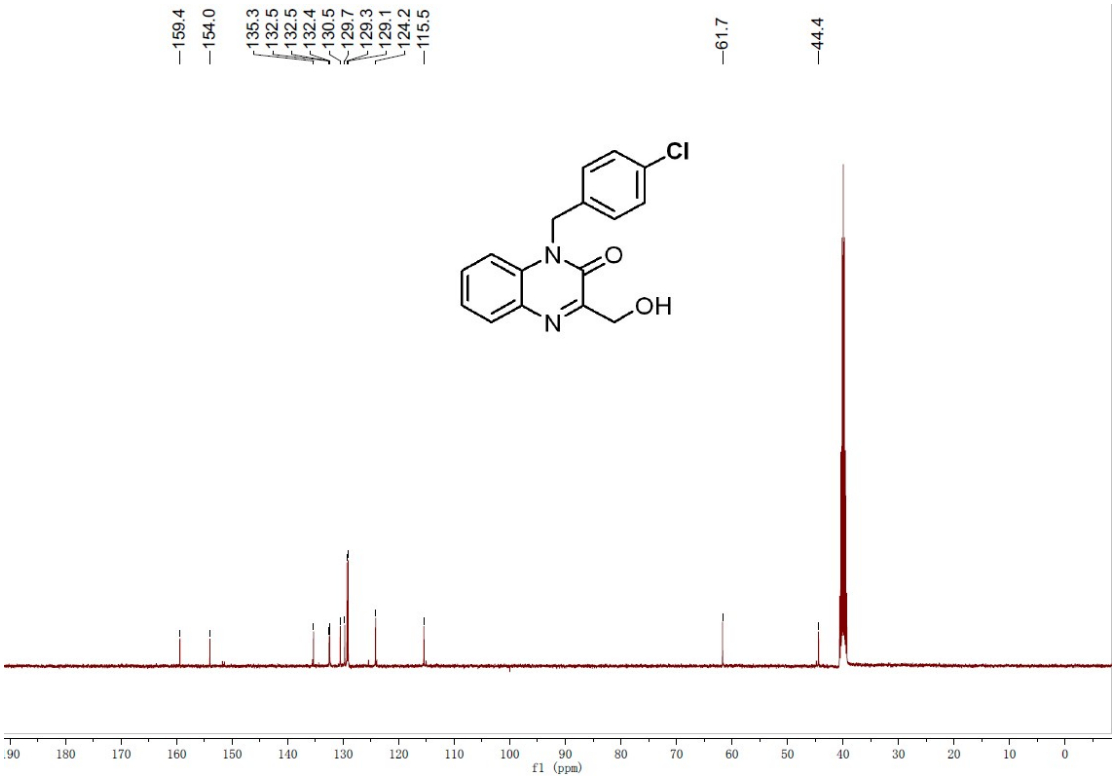
^{1}H NMR Spectrum of Compound 3jj



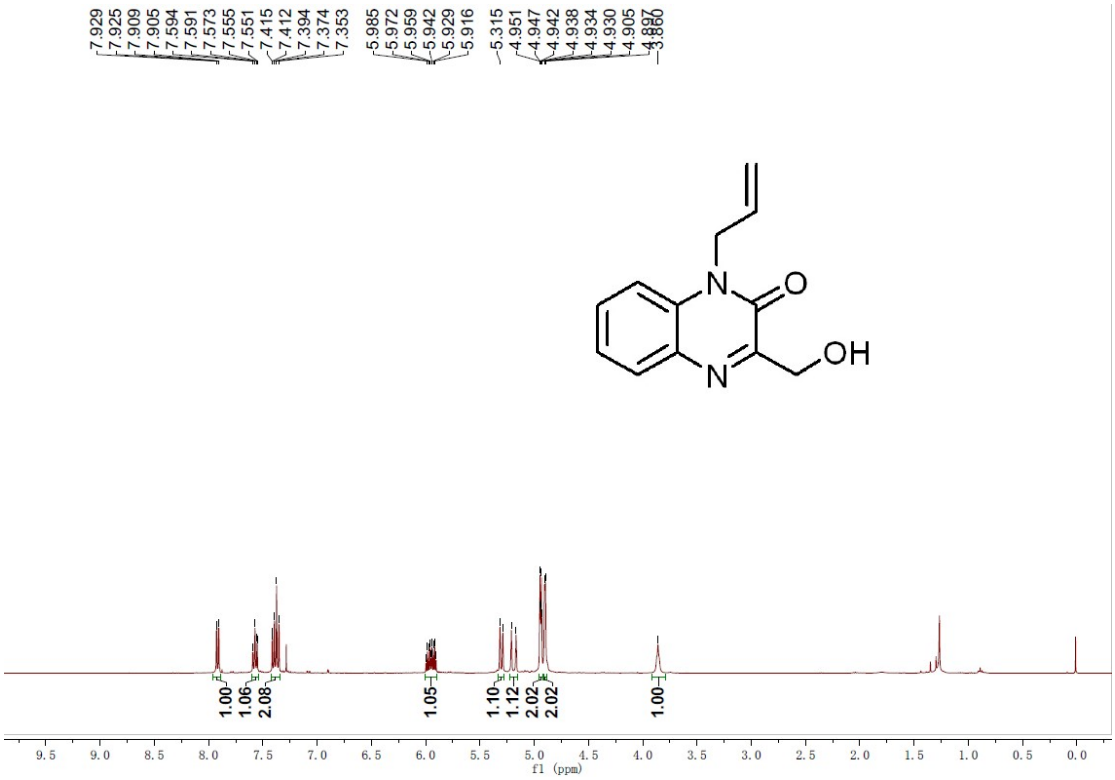
^{13}C NMR Spectrum of Compound 3jj



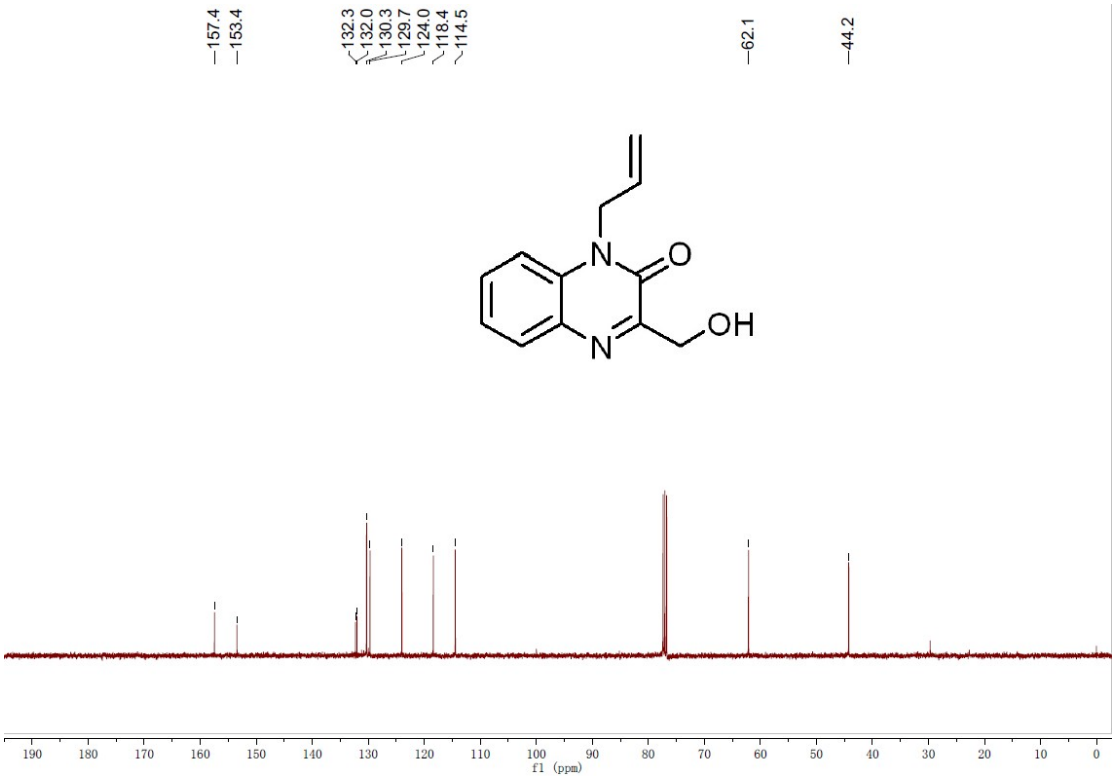
^{1}H NMR Spectrum of Compound 3kk



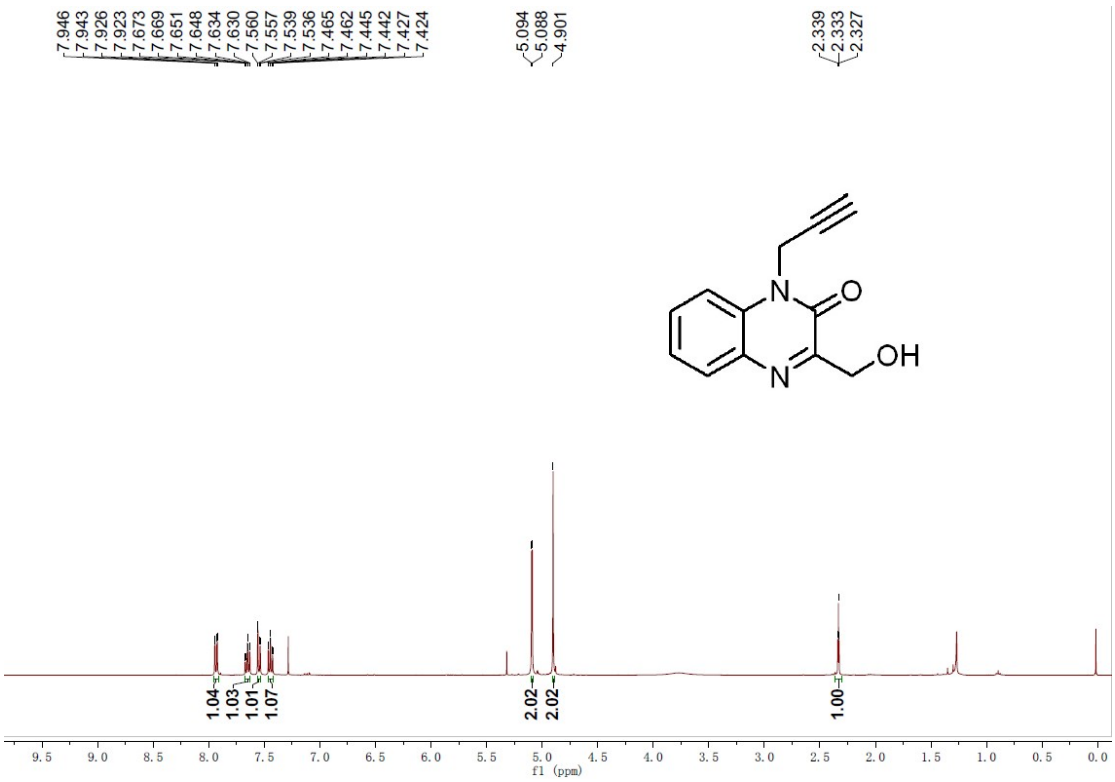
^{13}C NMR Spectrum of Compound 3kk



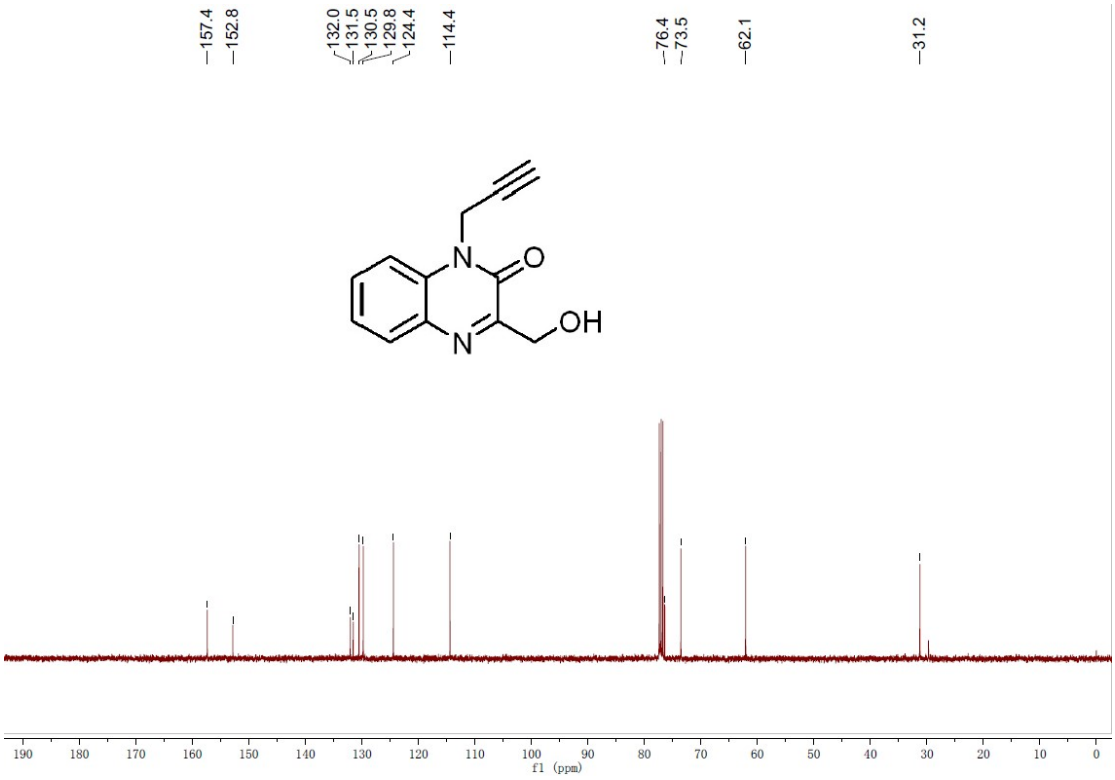
^{1}H NMR Spectrum of Compound 3ll



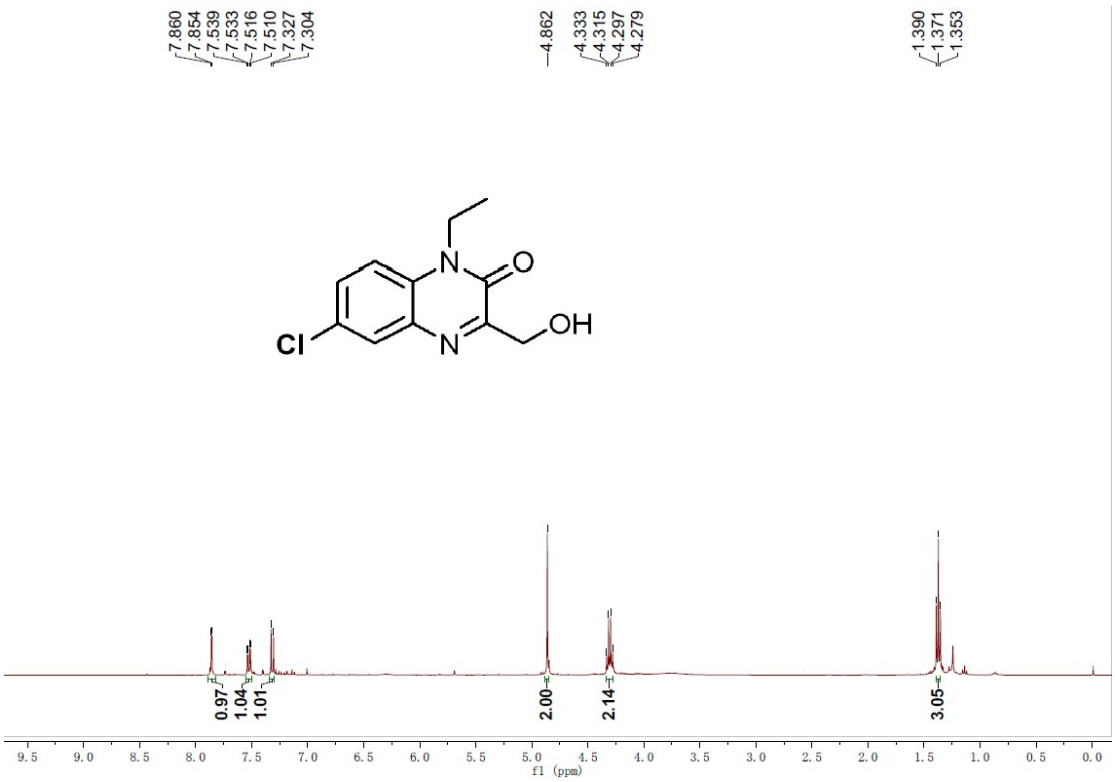
^{13}C NMR Spectrum of Compound 3ll



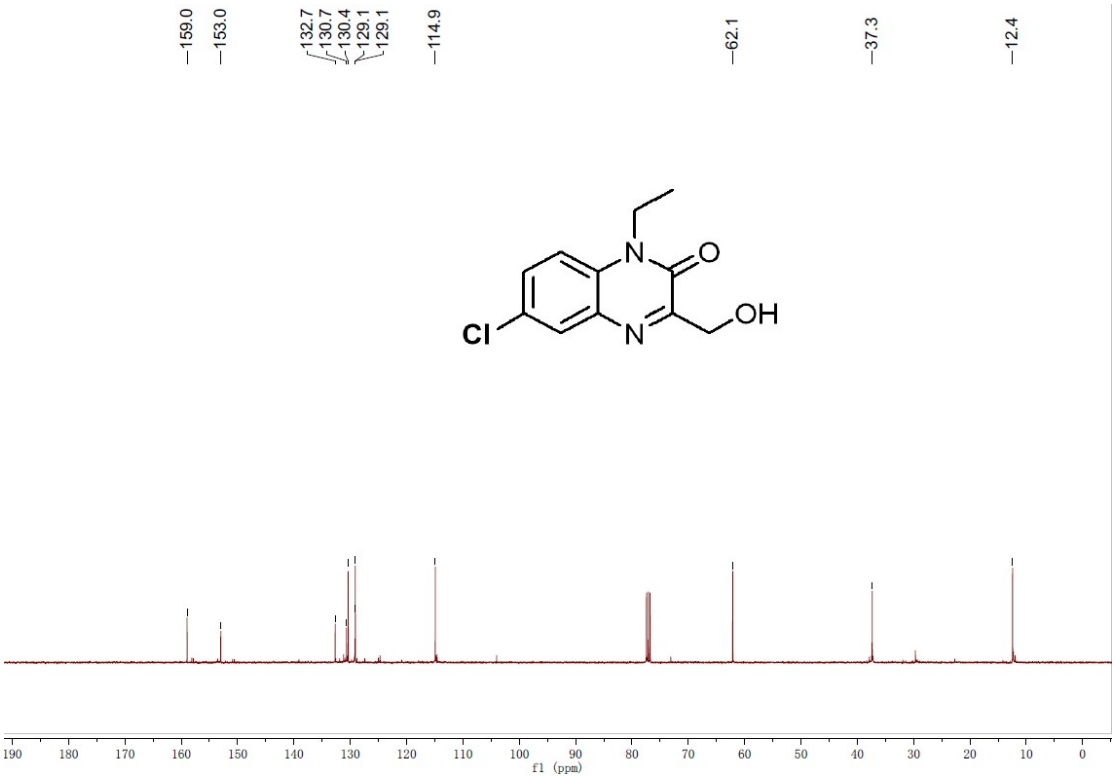
^{1}H NMR Spectrum of Compound 3mm



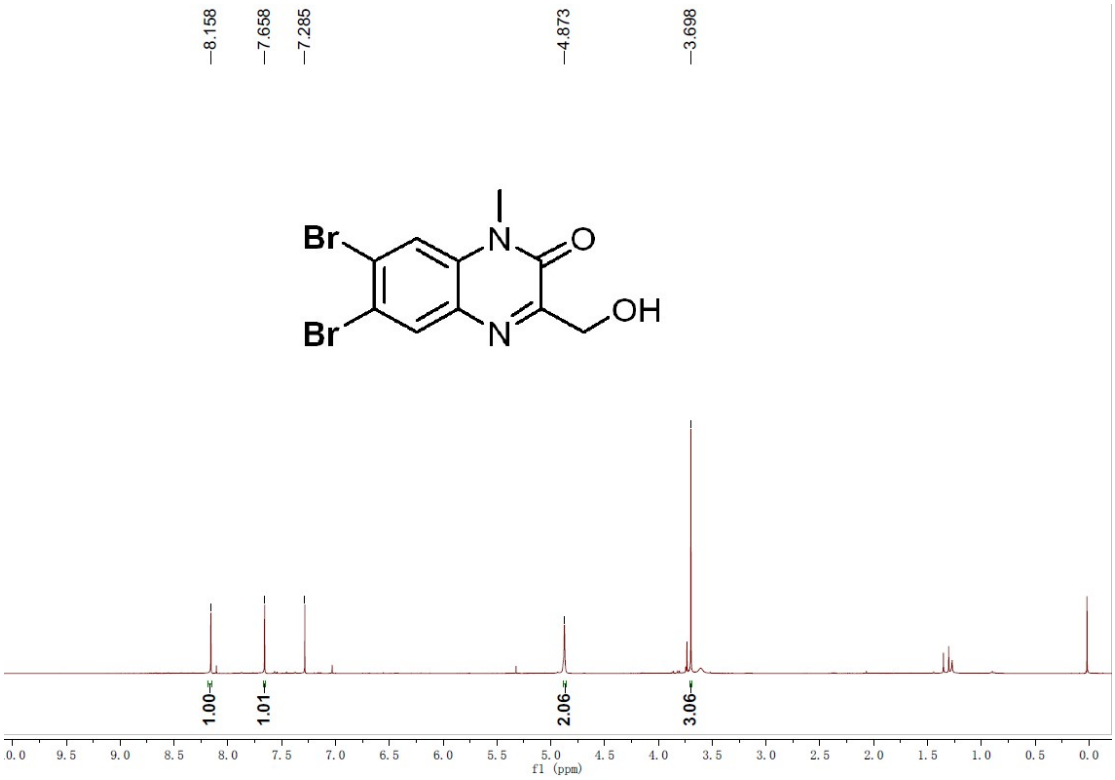
^{13}C NMR Spectrum of Compound 3mm



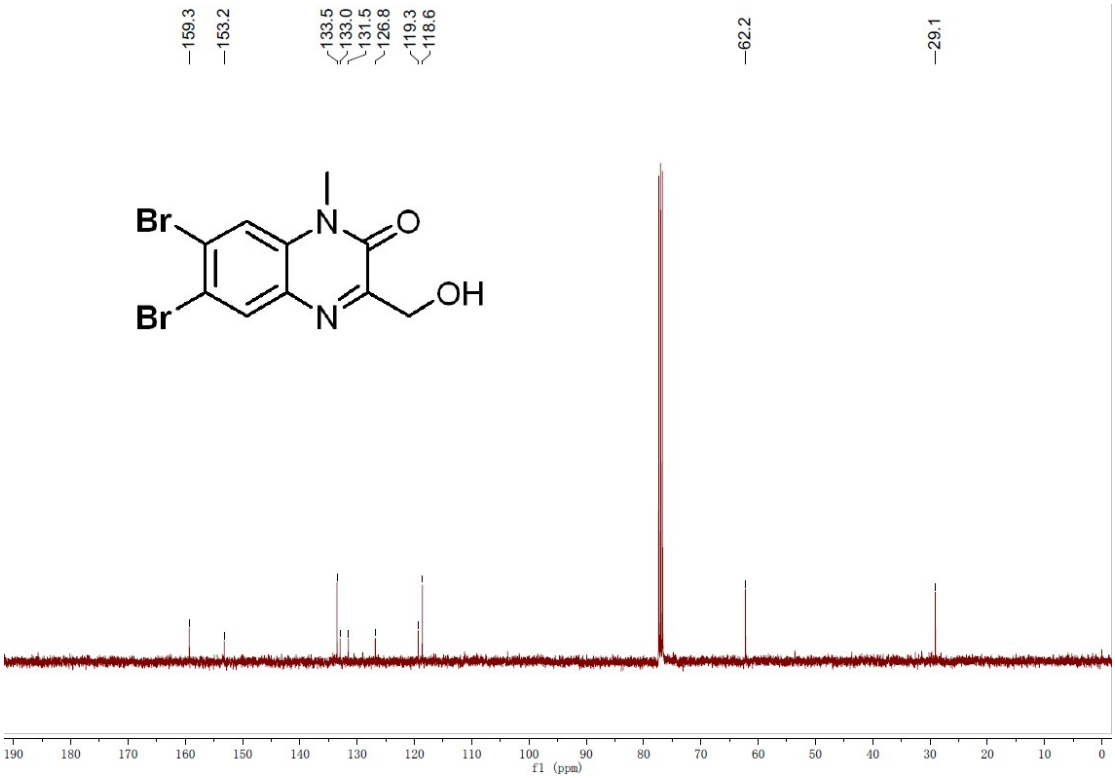
^{1}H NMR Spectrum of Compound 3nn



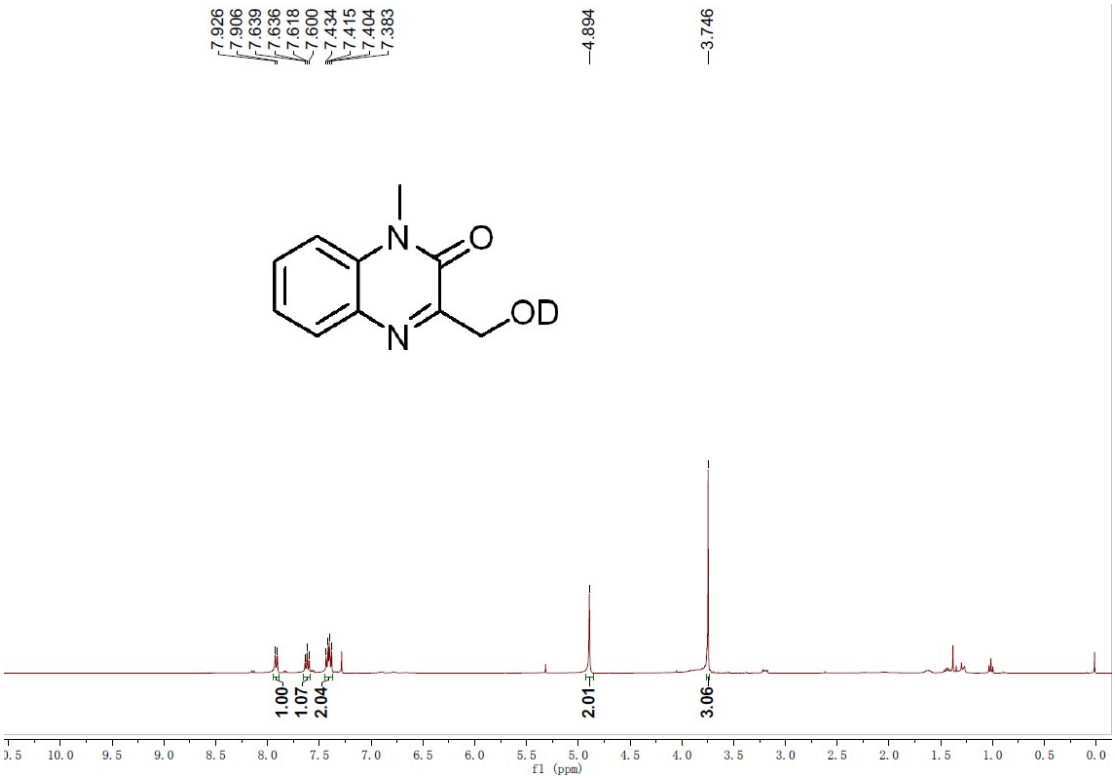
^{13}C NMR Spectrum of Compound 3nn



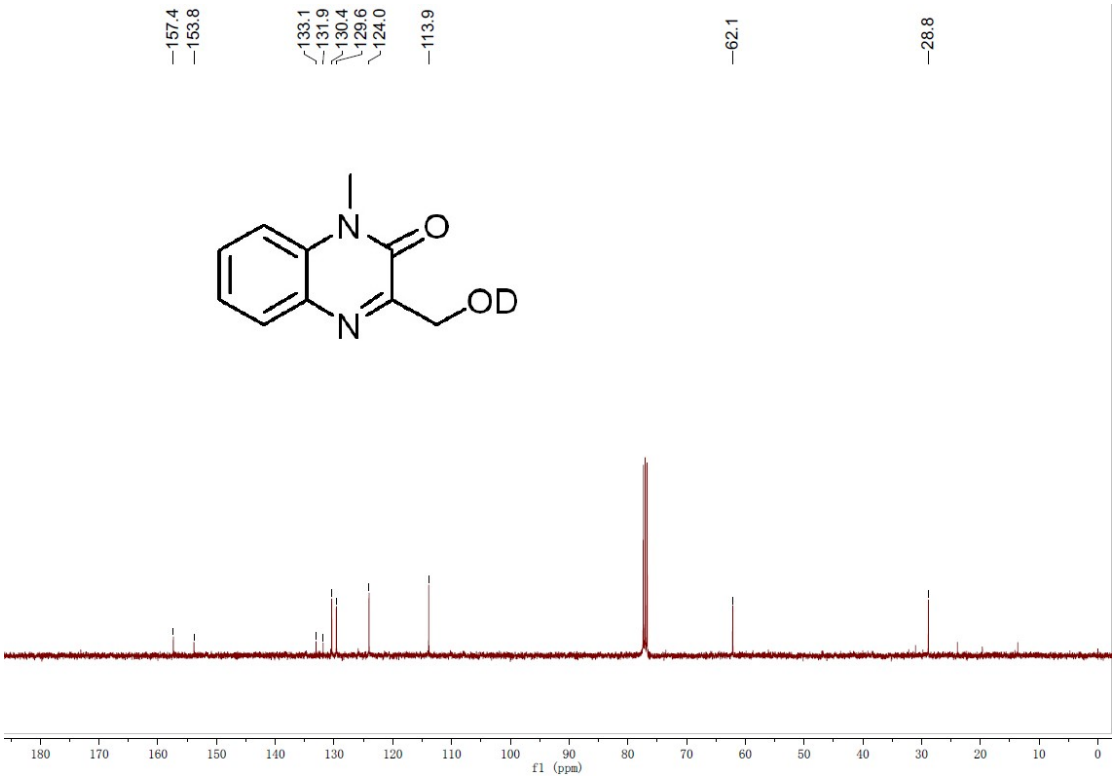
^{1}H NMR Spectrum of Compound 3oo



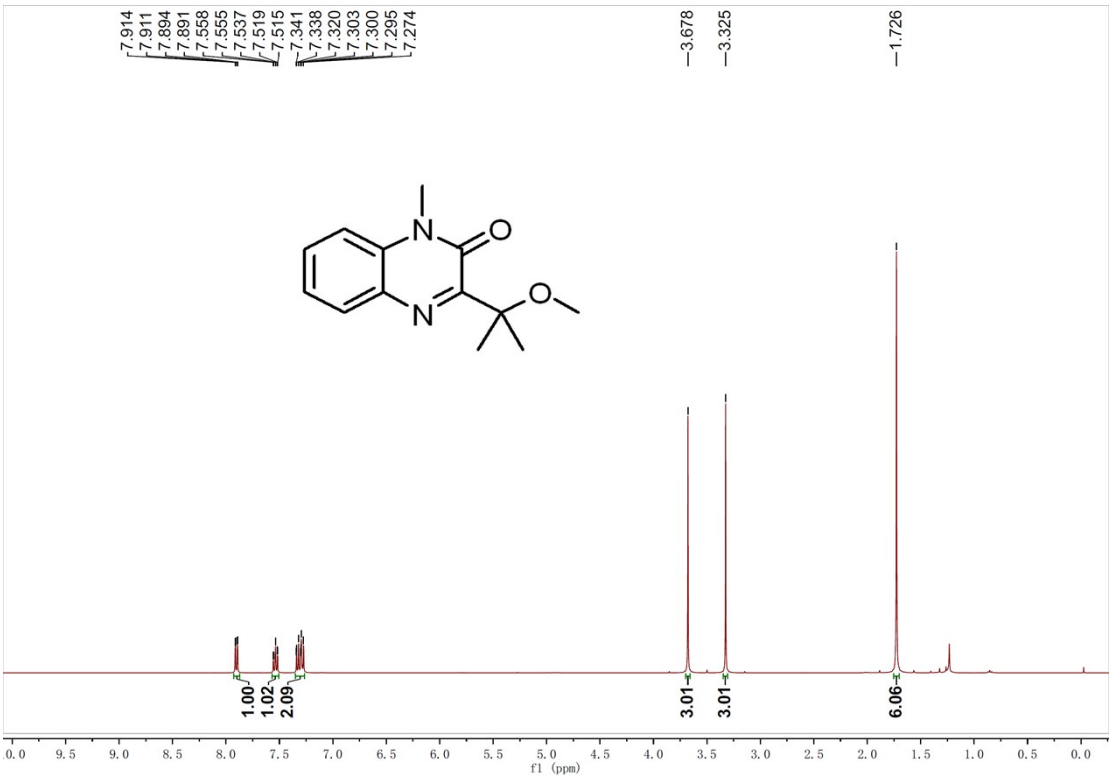
^{13}C NMR Spectrum of Compound 3oo



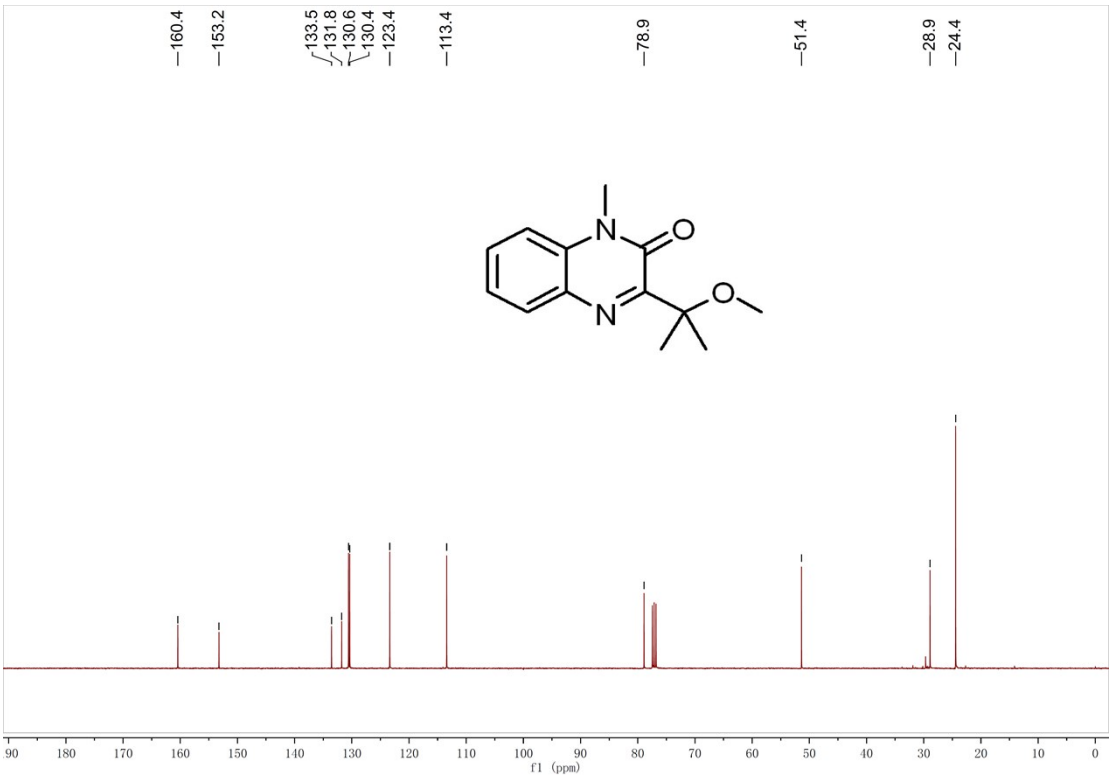
^{1}H NMR Spectrum of Compound 3pp



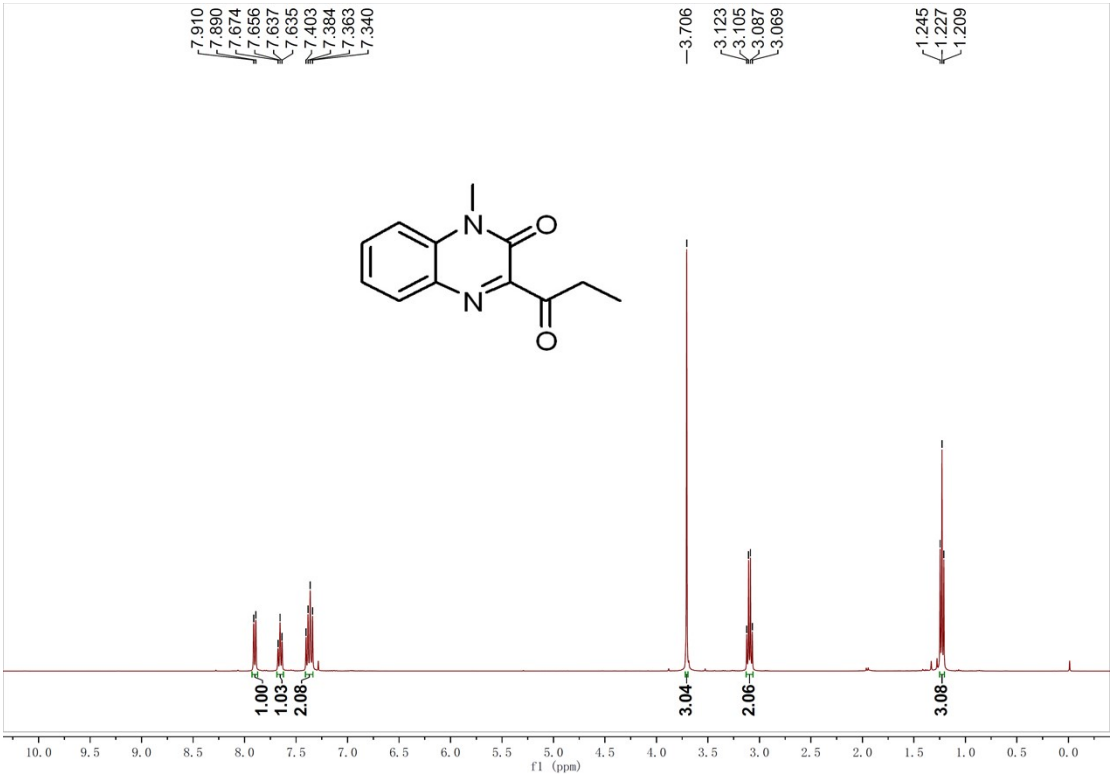
^{13}C NMR Spectrum of Compound 3pp



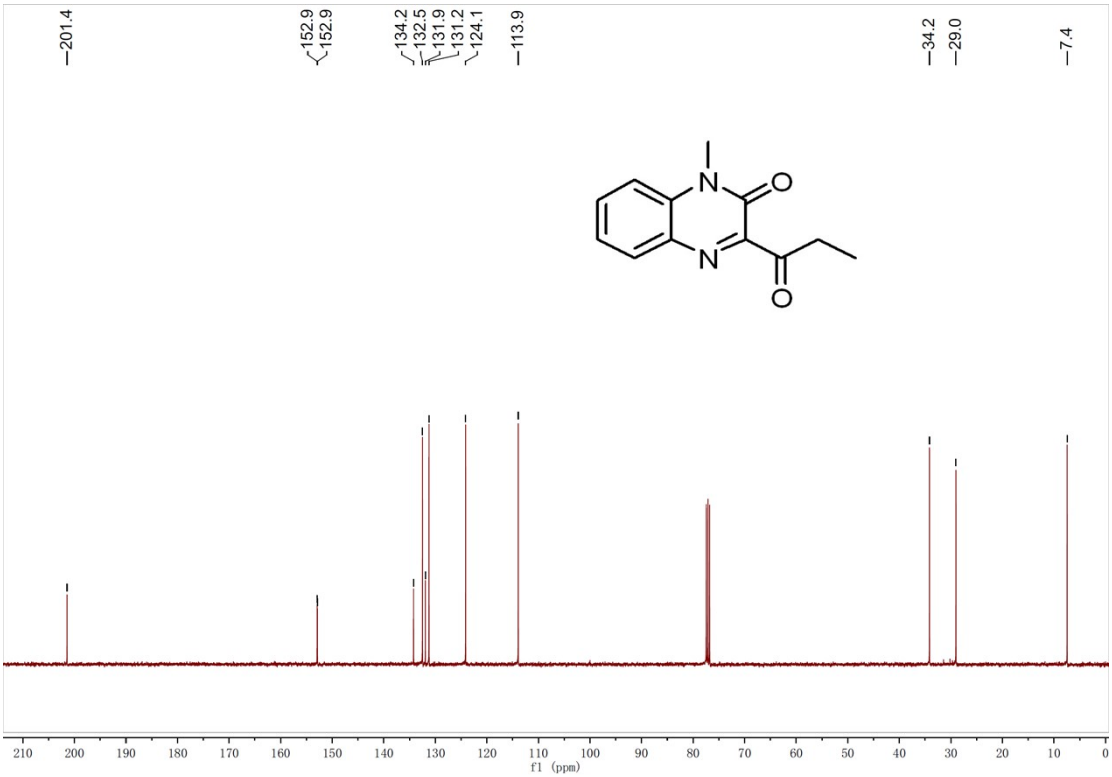
^{1}H NMR Spectrum of Compound 4a



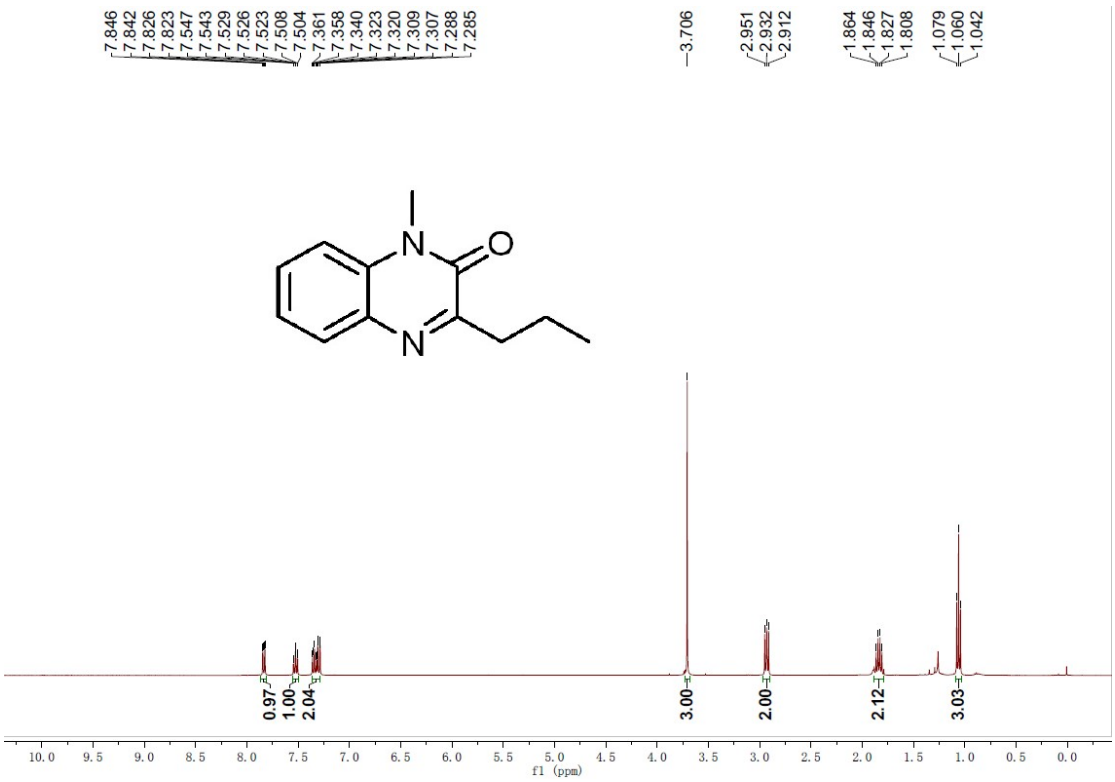
^{13}C NMR Spectrum of Compound 4a



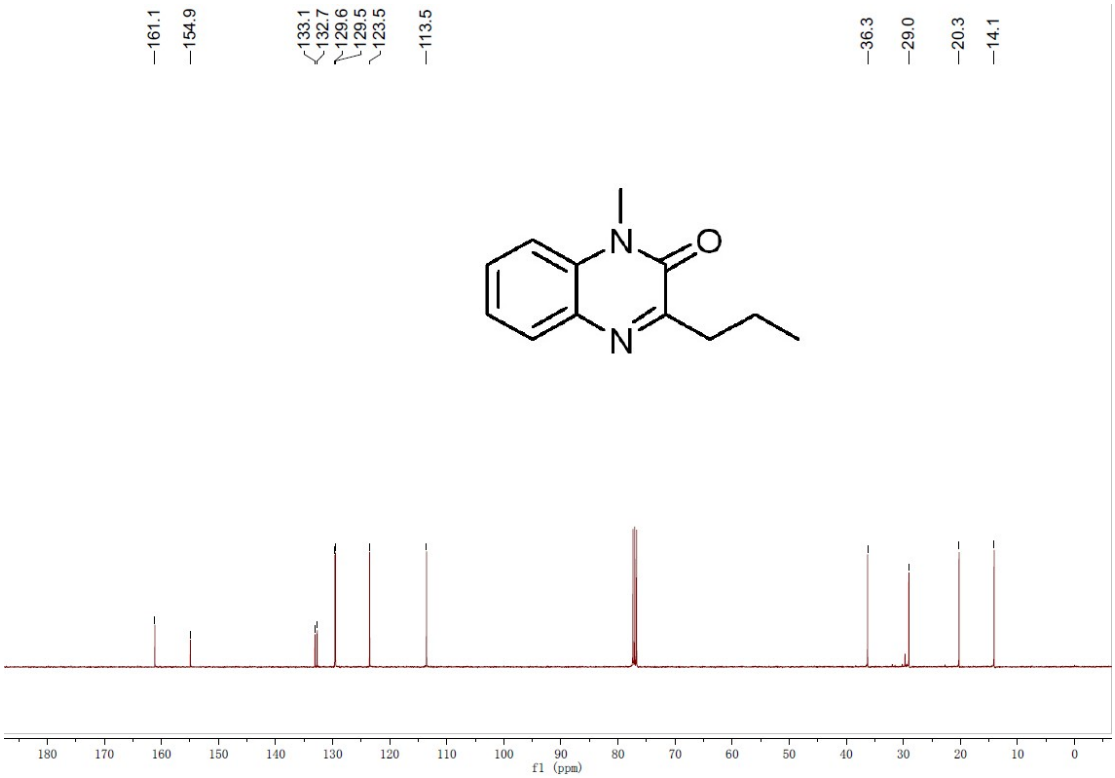
^{1}H NMR Spectrum of Compound 5a



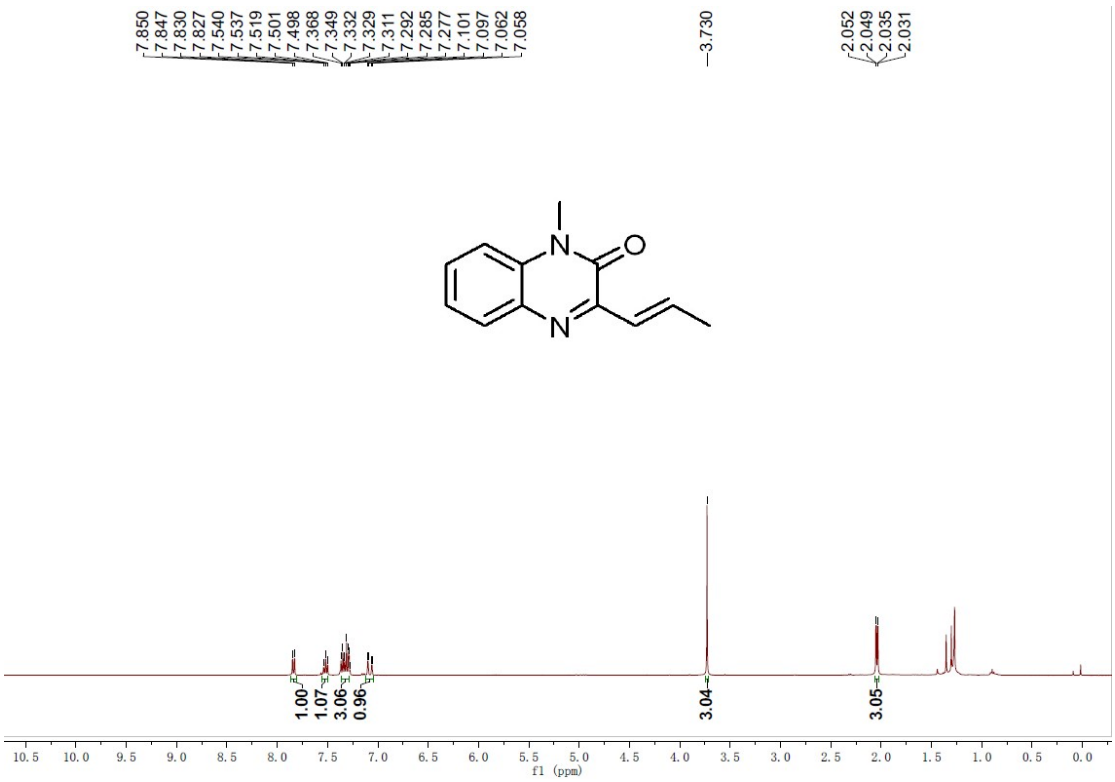
^{13}C NMR Spectrum of Compound 5a



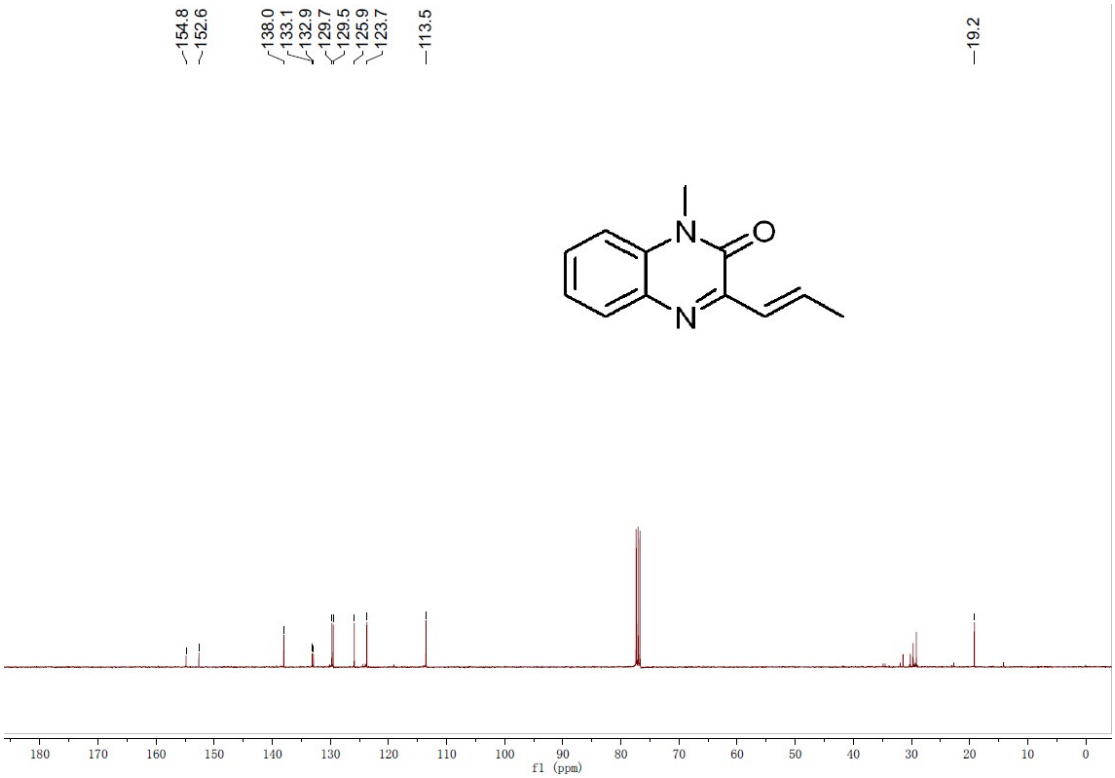
^{1}H NMR Spectrum of Compound 6a



^{13}C NMR Spectrum of Compound 6a



^{1}H NMR Spectrum of Compound 7a



^{13}C NMR Spectrum of Compound 7a