Supporting Information

N-Chlorosulfonyl carbamate-enabled, photoinduced amidation of quinoxalin-2(1*H*)-ones

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1. General experimental information

Unless otherwise noted, all the reagents were purchased from commercial suppliers and used without further purification. The light source used for illuminating the reaction vessel consists of 452 nm LEDs purchased from Taobao (https://gpiled.taobao.com/, manufacture: Shenzhen Star Sources Lighting Technology Co., Ltd.). A clip fan was placed over the reaction vials to cool down the reaction system during the whole process of the reaction. ^{1}H NMR spectra were recorded at 400 MHz. The chemical shifts were recorded in *ppm* relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = quintet, m = multiplet), coupling constants (Hz), integration. ^{13}C NMR data were collected at 100 MHz with complete proton decoupling. ^{19}F NMR data were collected at 376 MHz with complete proton decoupling. Infrared spectra (IR) were measured by FT-IR apparatus. High resolution mass spectroscopy (HRMS) was recorded on TOF MS ES^{+} mass spectrometer and acetonitrile were used to dissolve the sample. Column chromatography was carried out on silica gel (200-300 mesh).

2. Experimental procedures

Reaction set-up

Figure S1. The set-up for the reaction (photographed by C.-P. Yuan)

The light source used for illuminating the reaction tube was purchased from Taobao (https://gpiled.taobao.com/, manufacture: Shenzhen Star Sources Lighting Technology Co., Ltd). The quartz tube was screw-capped and stirred at room temperature under side irradiation of 452 nm LEDs (30 W blue LEDs, distance app. 5 cm, without filters) for 12 h. A clip fan was placed over the reaction vials to cool down the reaction system, and the measured reaction temperature varied between 30-40 °C.

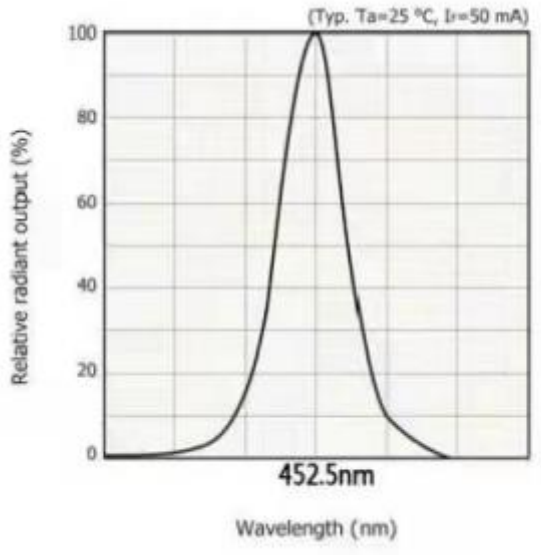
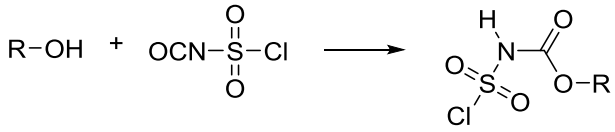


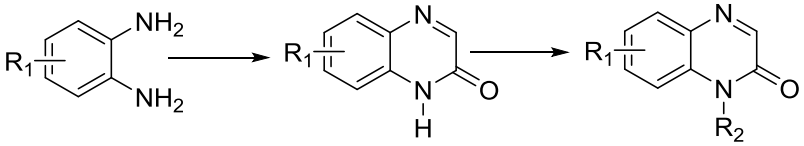
Figure S2. The emission spectra of the LEDs used in this study.

2.1 General procedure for the preparation of *N*-chlorosulfonyl carbamates



To a mixture of chlorosulfonyl isocyanate (20 mmol) in dry CH_2Cl_2 (5 mL) was added solution of alcohol (23 mmol) in dry CH_2Cl_2 (5 mL) dropwise at 0 ℃. The reaction mixture was stirred for 2 h at 0 ºC and concentrated under reduced pressure. The resultant solid was dried *in vacuo* to give chlorosulfonylcarbamate (98%) as a white solid, which was used next step without any further purification.[1]

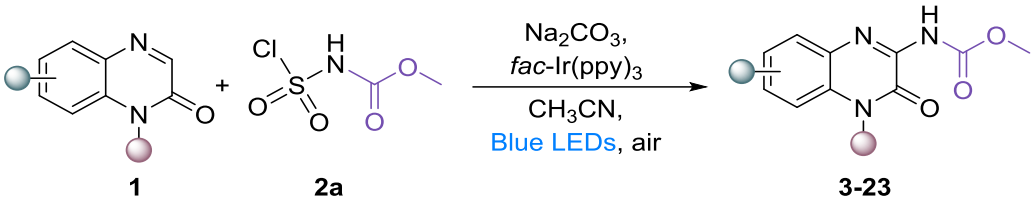
2.2 General procedure for the preparation of quinoxalin-2(1*H*)-ones



1' **1**

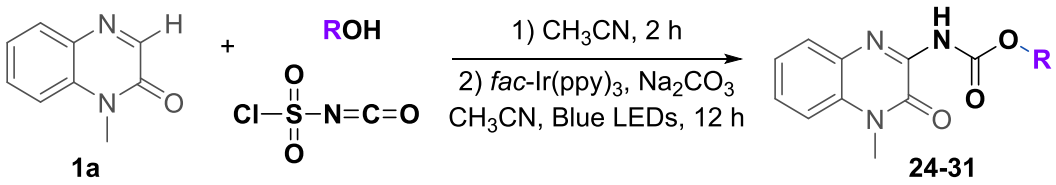
A mixture of *o*-phenylenediamine (5 mmol), ethyl 2-oxoacetate (6 mmol) and ethanol (20 mL) in a dried 50 mL round-bottom flask was stirred at reflux temperature for 1 hour in oil bath. After the completion (as monitoredby TLC), the reaction mixture was filtered, washed with ethanol, and then dried to give quinoxalinone **1'**. Subsequently, a mixture of quinoxalinone **1'**, K_2CO_3 (1.2 equiv.), corresponding halogenoalkane (1.6 equiv.), and DMF (20 mL) in a dried 50 mL round-bottom flask was stirred at room temperature overnight. After the completion (as monitored by TLC), themixture was then extracted with ethyl acetate and the collected organic layer was washed with brine, dried with $MgSO_4$. The solvent was removed under reduced pressure, and the crude product was further purified by silica gel column chromatography (200-300 mesh silica gel, PE/EtOAc = 5:1) to afford desired substrates **1**. All the substrates **1** are known compounds.[2−6]

2.3 General procedure for the preparation of compounds 3-23



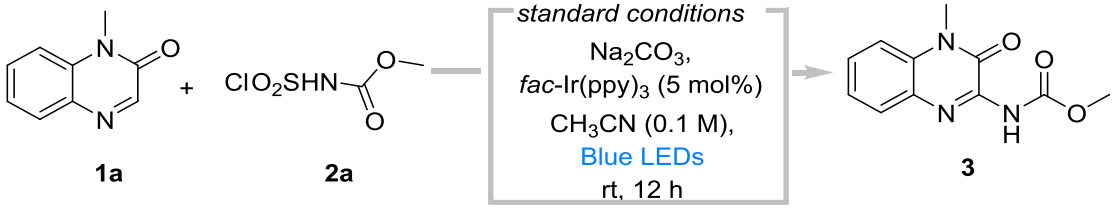
To a 20 mL reaction tube equipped with a magnetic stirring bar, quinoxalin-2(*1H*)-ones **1** (0.1mmol, 1.0 equiv), **2a** (0.2mmol, 2.0 equiv), Na_2CO_3(0.2mmol, 2.0 equiv), *fac*-Ir(ppy)$_3$ (5 mol%)and CH_3CN (1.0mL) were added. The tube was screw-capped under irradiation of 30 W blue LEDs (distance app. 5 cm, without filters) for 12 h. The solvent was removed under reduced pressure, and then the residue was purified by flash column chromatography (EtOAc/PE = 1:9 to 1:3) to afford the desired product **3-23**.

2.4 General procedure for one-pot synthesis of compounds 24-31



To a 20 mL reaction tube equipped with a magnetic stirring bar, dissolve the alcohol (0.576 mmol, 2.88 equiv.) in 2 mL acetonitrile solution, then add chlorosulfonyl isocyanate (0.48 mmol, 2.4 equiv.) under argon protection at 0 ° C and react at room temperature for 2 hours, **1a** (0.2 mmol, 1.0 equiv.), Na_2CO_3 (0.4 mmol, 2.0 equiv.), *fac*-Ir(ppy)$_3$ (5 mol%) were added. The tube was screw-capped under irradiation of 30 W blue LEDs (distance app. 5 cm, without filters) for 12 h. The solvent was removed under reduced pressure, and then the residue was purified by flash column chromatography (EtOAc/PE = 1:19 to 1:3) to afford the desired product **24-31**.

3. Optimization of reaction conditions



3.1 Screening of base

Entry	Photocatalyst	Base	[a]Yield(%)
1	*fac*-Ir(ppy)$_3$	DIPEA	58
2	*fac*-Ir(ppy)$_3$	DMAP	29
3	*fac*-Ir(ppy)$_3$	DABCO	11
4	*fac*-Ir(ppy)$_3$	K_2CO_3	80
5	*fac*-Ir(ppy)$_3$	Na_2CO_3	95

[a]The reaction was carried out on a 0.1 mmol scale in CH_3CN (1 mL) with photocatalyst (5 mol%), base (0.2 mmol), irradiating with 30 W blue LEDs for specified time, under air atmosphere.

3.2 Screening of solvent

Entry	Solvent	Base	[a]Yield (%)
1	DCM	Na_2CO_3	n.d.
2	Acetone	Na_2CO_3	n.d.
3	THF	Na_2CO_3	trace
4	DMF	Na_2CO_3	51

[a]The reaction was carried out on a 0.1 mmol scale in CH_3CN (1 mL) with *fac*-Ir(ppy)$_3$ (5 mol%), base (0.2 mmol), irradiating with 30 W blue LEDs for specified time, under air atmosphere.

4. Scale up reaction

To an oven-dried 120 mL reaction tube equipped with a magnetic stir bar, quinoxalin-2(*1H*)-one (**1a**, 2.0 mmol, 1.0 equiv., 320 mg), **2a** (4.0 mmol, 2.0 equiv., 700 mg), Na_2CO_3 (4 mmol, 2.0 equiv., 424 mg), *fac*-Ir(ppy)$_3$ (5 mol%, 0.1 mmol, 65 mg) and CH_3CN (20 mL) were added. The tube was screw-capped and stirred at room temperature under irradiation of blue LEDs (30 W, home-made set-up) for 24 h. The solvent was removed under reduced pressure, and then the residue was purified by flash column chromatography (PE/EtOAc = 75:25) to afford the product **3** (336 mg, 72% yield).

initial state (scale: 2 mmol) | The reaction process 24 h | end state (336mg, 72% yield)

Figure S3. Scale-up reaction (photographed by C.-P. Yuan)

5. Mechanistic studies

5.1 Trapping experiment

Radical trapping experiments between **1a** and **2a** were conducted under standard conditions with two trapping agents (BHT and TEMPO) to catch the putative radicals. Interestingly, no desired product **3** was detected when TEMPO or BHT were employed as theradical scavenger, indicating that the reaction was completely inhibited. ESI-MS analysis of the crude reaction mixture was performed and the corresponding radical captured products were successfully detected by HRMS (**Figure S2**). HRMS (EI): $C_{17}H_{27}NNaO_3^+$, $[M+Na]^+$calcd: 316.1883, found: 316.1883 (BHT-adduct); HRMS (EI): $C_{20}H_{31}N_4O_4^+$, $[M+H]^+$ calcd: 391.2340, found: 391.2349 (TEMPO-adduct).

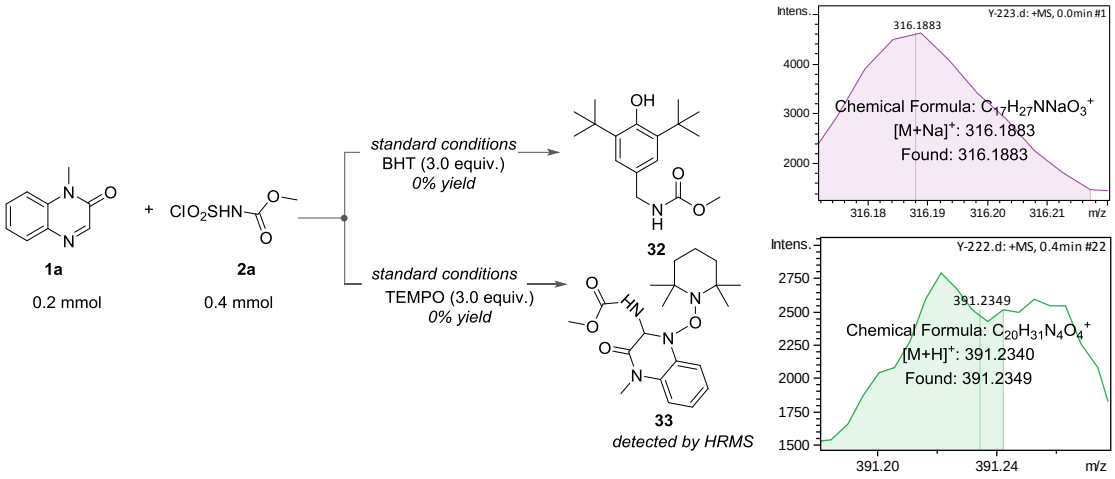


Figure S4. Crude ESI-MS analysis of the BHT-trapping and TEMPO-trapping experiments

5.2 Cyclic voltammetry study

Cyclic voltammetry experiments were performed at room temperature by using a CHI650D electrochemical workstation with a 3 mm glassy carbon disk electrode as the working electrode (polished with cloth polishing pad in a water-alumina slurry), the $Ag/AgNO_3$ (0.1 M $AgNO_3$) electrode as the reference electrode, and a platinum electrode as the counter electrode. The testing solution of **2a** was prepared by dissolving the sample (0.20 mmol) into MeCN (10 mL) with 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$). Solutions were degassed with argon prior to measurement and experiments were performed under an atmosphere of argon. The scanned potential range was typically -2.0 V to 2.0 V at a scan rate of 100 mV/s.The measurement was performed by first scanning the potential in positive direction and then in negative direction. Substrate **2a** was irreversibly reduced at a peak potential of -0.806 V *vs*. $Ag/AgNO_3$. This gives the irreversible reduction potential of -0.686 V *vs* SCE for **2a** in DMF (using the conversion $E^0_{1/2}$ ($Ag/AgNO_3$) = +0.12 V *vs* SCE). Figure S4 displays the cyclic voltammogram. IUPAC convention was used to report CV data.

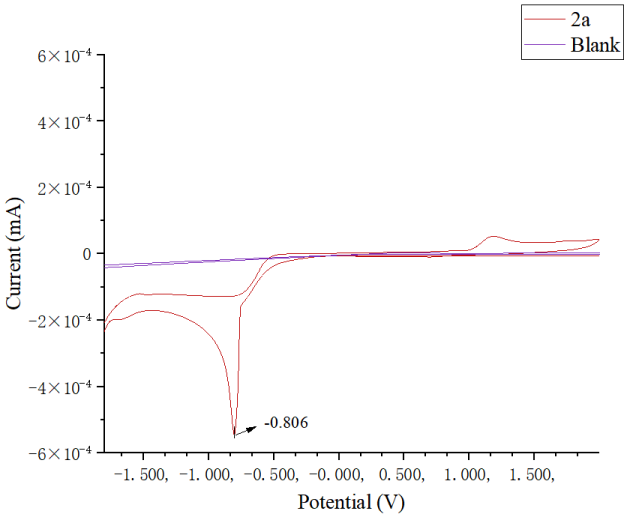


Figure S5. Cyclic voltammetry of **2a** in MeCN (0.1 M $TBAPF_6$), using glassy carbon plate working electrode, platinum electrode as counter electrode and $Ag/AgNO_3$ as reference electrode at 100 mV/s scan rate. Initial potential was -2.0 V to 2.0 V. The data is plotted using the IUPAC convention.

5.3 Time profile of the transformation with the light on/off over time

Standard reactions were set up parallelly on a 0.20 mmol scale on the basis of the general procedure and additional 1.0 equiv of $PhCF_3$ (0.20 mmol) was added as the interior standard. After being irradiated for 2 hours, an aliquot (100 μL) from the reaction mixture was transferred into a nuclear magnetic tube charged with 0.4 mL of $CDCl_3$. The yield of product **22** was determined by ^1H NMR. Then, the reaction mixture was stirred for 2 hours with light-off. All of the following yields were analyzed in the identical way after 2 hours light on or off. It seems that induction period was observed from light on-off experiment. The increase of reaction rate should be explained by a higher temperature after light illumination.

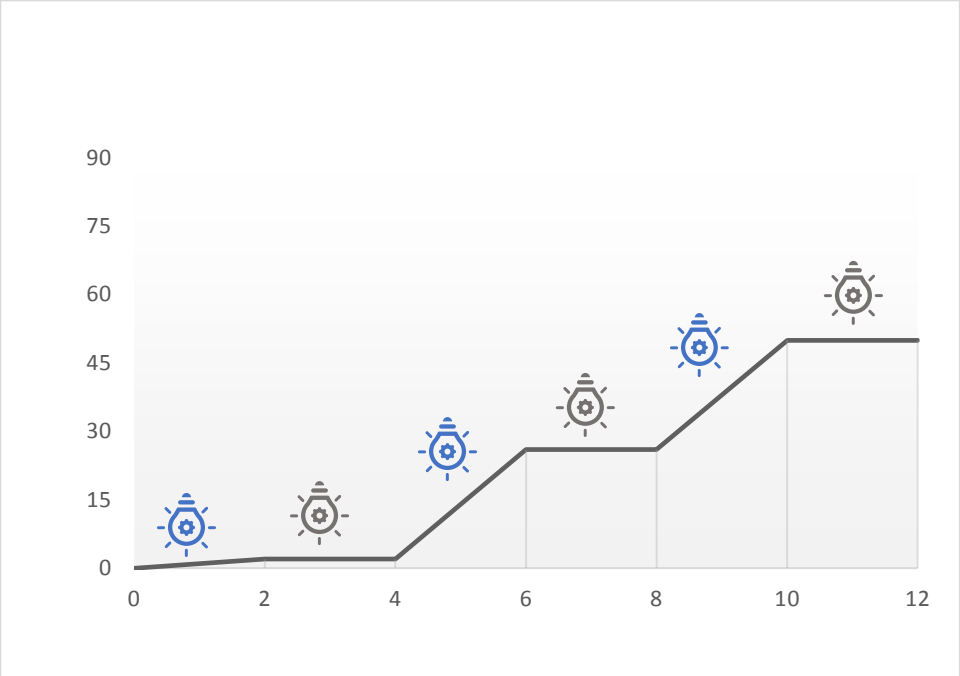


Figure S6. Time profile of the NMR yield with the light on/off over time

5.4 Emission Quenching Experiments

All fluorescence measurements were recorded using a Hitachi FL-7000 Fluorometer. Quenching studies were conducted in CH_3CN. All *fac*-Ir(ppy)$_3$ solutions (concentration of 5 μM) were excited at 260 nm and the emission intensity was collected at 605 nm. Measurements using corresponding quenchers were taken in triplicate at different concentrations.

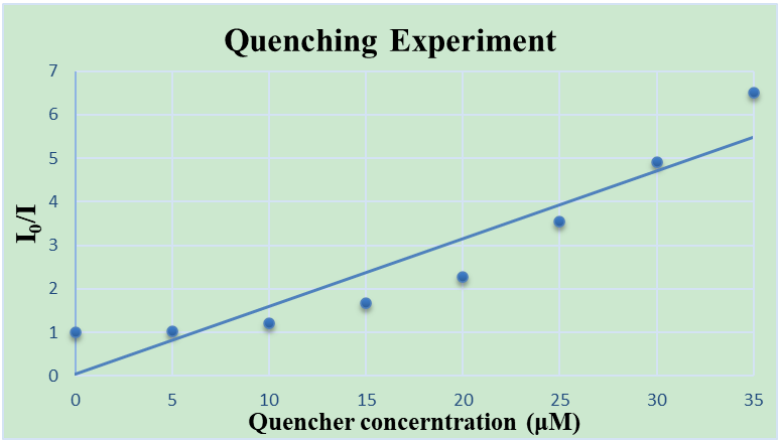
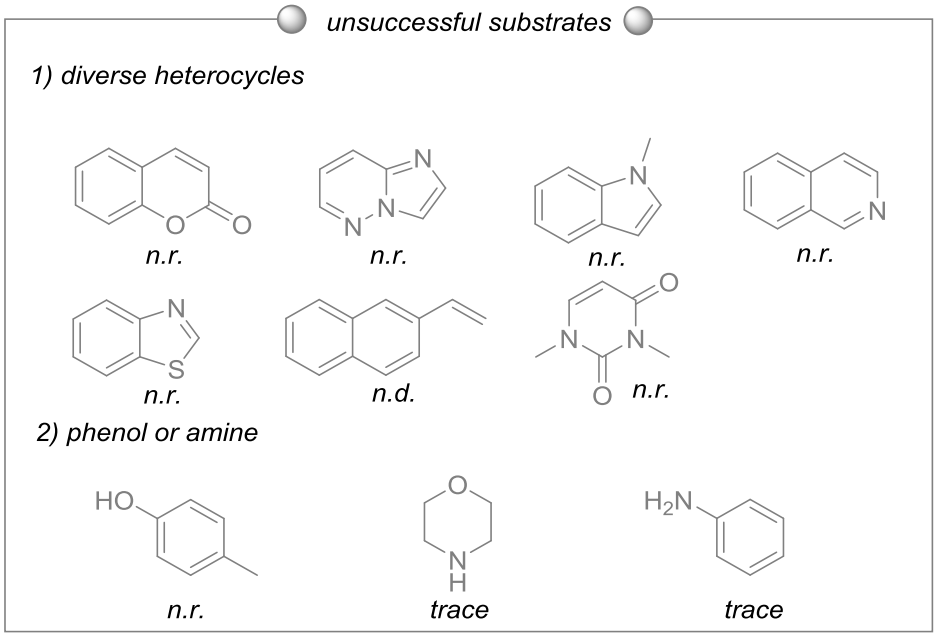


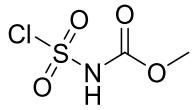
Figure S7. Quenching of the *fac*-Ir(ppy)$_3$ emission (5×10^{-5} M in CH_3CN) in the presence of increasing amounts of **2a**.

5.5 Unsuccessful substrates

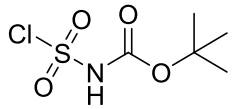


6. Characterization data of compounds

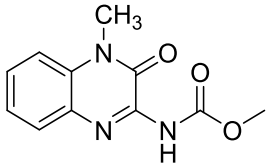
All quinoxalin-2(*1H*)-one substrates were known compounds, and their data were in agreement with previous reports.[7,8,9]



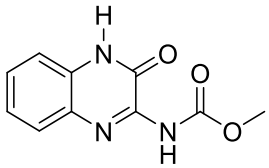
2a: white solid (3.39g, yield: 98%) using the general procedure **2.1.** This is a known compound.[1,10]
^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.17 (s, 1H), 3.96 (s, 3H).



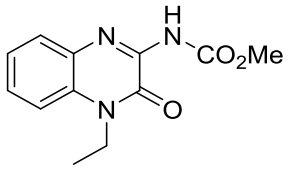
2b: white solid (4.21g, yield: 98%) using the general procedure **2.1.** This is a known compound.[1,10]
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.70 (s, 1H), 1.57 (s, 9H).



3: 21.9 mg, (PE/EtOAc = 75:25), white solid, yield: 95%;
IR (neat) *v* 3282, 2918, 1768, 1516, 1359, 1182, 751, 546 cm $^{-1}$;
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.69 (s, 1H), 7.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.48 – 7.44 (m, 1H), 7.39 – 7.34 (m, 1H), 7.29 (dd, *J* = 8.4, 1.4 Hz, 1H), 3.87 (s, 3H), 3.76 (s, 3H).
^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.5, 150.9, 144.1, 132.0, 130.7, 129.1, 128.0, 124.6 113.6, 52.9, 29.9.
HRMS (ESI) m/z: [M+K]$^+$Calcd for $C_{11}H_{11}KN_3O_3^+$272.0432, Found 272.0435.

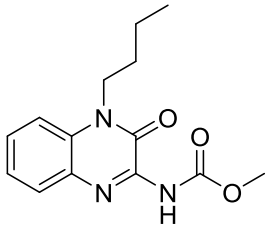


4: 14.0 mg, (PE/EtOAc = 70:30), white solid, yield: 64%;
IR (neat) *v* 3397, 2918, 1774, 1523, 1370, 1177, 746, 582cm $^{-1}$;
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.32 (*br*s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 2H), 3.73 (s, 3H).
^{13}C NMR (100 MHz, DMSO-d_6) δ 151.9, 151.2, 146.0, 131.6, 130.2, 128.1, 127.4, 124.1, 115.8, 52.8.
HRMS (ESI) m/z: [M+Na]$^+$Calcd for $C_{10}H_9N_3NaO_3^+$242.0536, Found 242.0557.

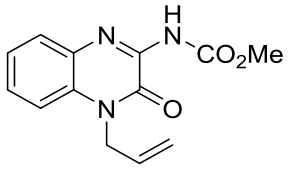


5: 19.5 mg, (PE/EtOAc = 75:25), white solid, yield: 79%;

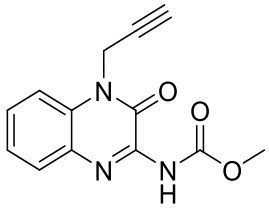
IR (neat) *v* 3253, 2918, 1756, 1569, 1338, 1185, 714, 560cm^{-1};
^{1}H NMR (400 MHz, Chloroform-*d*) *δ*8.67 (s, 1H), 7.87 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.46 (ddd, *J* = 8.5, 7.3, 1.6 Hz, 1H), 7.38 – 7.29 (m, 2H), 4.36 (q, *J* = 7.2 Hz, 2H), 3.86 (s, 3H), 1.40 (t, *J* = 7.2 Hz, 3H);
^{13}C NMR (100 MHz, Chloroform-*d*) *δ* 151.5, 150.4, 144.1, 132.4, 129.6, 129.4, 128.0, 124.3, 113.4, 52.9, 38.1, 12.4.
HRMS (ESI) m/z: [M+K]$^+$Calcd for $C_{12}H_{13}KN_3O_3^+$ 286.0588, Found 286.0597.



6: 20.9 mg, (PE/EtOAc = 80:20), yellow solid, yield: 76%;
IR (neat) *v* 3282, 2922, 1698, 1514, 1315, 1176, 742, 561cm^{-1};
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 8.67 (s, 1H), 7.87 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.45 (ddd, *J* = 8.6, 7.3, 1.6 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.29 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.32 – 4.26 (m, 2H), 3.86 (s, 3H), 1.79 – 1.75 (m, 2H), 1.48 (q, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (100 MHz, Chloroform-*d*) *δ* 151.6, 150.6, 144.1, 132.3, 129.8, 129.3, 127.9, 124.3, 113.6, 52.9, 42.9, 29.3, 20.2, 13.7.
HRMS (ESI) m/z: [M+Na]$^+$Calcd for $C_{14}H_{17}N_3NaO_3^+$298.1162, Found 298.1163.



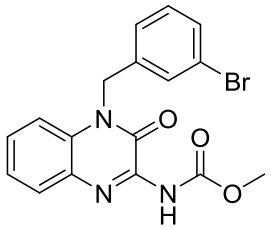
7: 15.5 mg, (PE/EtOAc = 75:25), yellow solid, yield: 60%;
IR (neat) *v* 3282, 2848, 1762, 1510, 1360, 1175, 750, 556cm^{-1};
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 8.67 (s, 1H), 7.87 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.45 – 7.39 (m, 1H), 7.37 – 7.32 (m, 1H), 7.28 – 7.27 (m, 1H), 6.01 – 5.88 (m, 1H), 5.33 – 5.26 (m, 1H), 5.16 (dd, *J* = 17.2, 1.9 Hz, 1H), 4.94 (dt, *J* = 5.1, 1.8 Hz, 2H), 3.87 (s, 3H);
^{13}C NMR (100 MHz, Chloroform-*d*) *δ* 151.5, 150.6, 144.1, 132.2, 130.1, 129.9, 129.2, 128.0,124.6, 118.4, 114.2, 52.9, 45.2.
HRMS (ESI) m/z: [M+K]$^+$Calcd for $C_{13}H_{13}KN_3O_3^+$298.0588, Found 298.0596.



8: 10.0 mg, (PE/EtOAc = 75:25), white solid, yield: 39%;
IR (neat) *v* 3264, 2360, 1761, 1521, 1363, 1180, 758, 566cm^{-1};
^{1}H NMR (400 MHz, Chloroform-*d*) *δ* 8.61 (s, 1H), 7.88 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.53 – 7.44 (m, 2H), 7.41 – 7.37 (m, 1H), 5.08 (d, *J* = 2.5 Hz, 2H), 3.87 (s, 3H), 2.33 (t, *J* = 2.5 Hz, 1H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.4, 150.2, 143.8, 132.2, 129.24, 129.22, 128.1, 124.9, 114.1, 73.8, 53.0, 32.3, 29.7.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{13}H_{11}N_3NaO_3^+$280.0693, Found 280.0689.



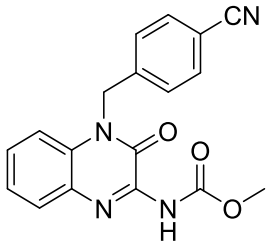
9: 15.5 mg, (PE/EtOAc = 75:25), yellow solid, yield: 40%;

IR (neat) *v* 3255, 2919, 1748, 1540, 1303, 1188, 735, 557cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.69 (s, 1H), 7.90 – 7.87 (m, 1H), 7.43 – 7.37 (m, 2H), 7.36 – 7.33(m, 2H), 7.22 – 7.16 (m, 2H), 7.13 (dt, *J* = 7.9, 1.3 Hz, 1H), 5.49 (s, 2H), 3.88 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.5, 151.1, 144.1, 136.9, 132.3, 131.2, 130.6, 129.81, 129.79, 129.3, 128.2, 125.3, 124.8, 123.2, 114.2, 53.0, 46.0.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{17}H_{14}BrN_3NaO_3^+$410.0111, Found 410.0121.



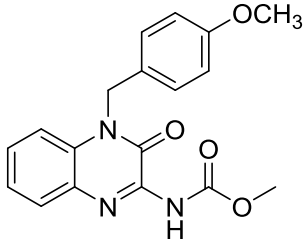
10: 28.0 mg, (PE/EtOAc = 65:35), white solid, yield: 84%;

IR (neat) *v* 3385, 2920, 1754, 1520, 1361, 1181, 747, 550cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.65 (s, 1H), 7.90 (dd, *J* = 6.1, 3.4 Hz, 1H), 7.65 – 7.62 (m, 2H), 7.36 (d, *J* = 3.5 Hz, 1H), 7.35 – 7.33 (m, 2H), 7.32 (s, 1H), 7.10 (dd, *J* = 6.1, 3.5 Hz, 1H), 5.58 (s, 2H), 3.89 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.4, 151.1, 144.0, 139.9, 132.9, 132.3, 129.6, 129.5, 128.2, 127.5, 125.0, 118.2, 113.9, 112.1, 53.0, 46.2.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{18}H_{14}N_4NaO_3^+$357.0958, Found 357.0976.



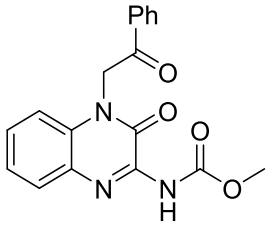
11: 18.9 mg, (PE/EtOAc = 75:25), white solid, yield: 56%;

IR (neat) *v* 3281, 2834, 1756, 1522, 1315, 1176, 739, 561cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.73 (s, 1H), 7.90 – 7.82 (m, 1H), 7.32 (ddd, *J* = 6.8, 4.1, 2.1 Hz, 2H), 7.23 (ddd, *J* = 7.9, 5.1, 3.1 Hz, 2H), 6.82 – 6.77 (m, 2H), 6.75 (t, *J* = 2.0 Hz, 1H), 5.49 (s, 2H), 3.87 (s, 3H), 3.74 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 160.1, 151.6, 151.1, 144.1, 136.2, 132.3, 130.1, 130.0, 129.1, 128.0, 124.6, 118.9, 114.5, 112.9, 112.8, 55.2, 52.9, 46.6.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{18}H_{17}N_3NaO_4^+$362.1111, Found 362.1126.



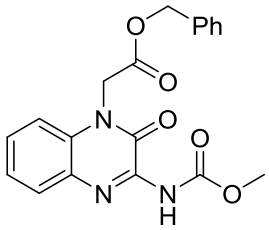
12: 24.0 mg, (PE/EtOAc = 75:25), white solid, yield: 71%;

IR (neat) *v* 3263, 2919, 1760, 1517, 1311, 1175, 748, 561cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.60 (s, 1H), 8.08 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.92 – 7.87 (m, 1H), 7.69 (d, *J* = 7.5 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.32 (m, 2H), 6.95 – 6.91 (m, 1H), 5.76 (s, 2H), 3.87 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 190.3, 151.5, 151.0, 143.8, 134.5, 134.3, 132.2, 130.1, 129.3, 129.2, 128.2, 128.1, 124.7, 113.4, 52.9, 49.1.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{18}H_{15}N_3NaO_4^+$360.0955, Found 360.0962.



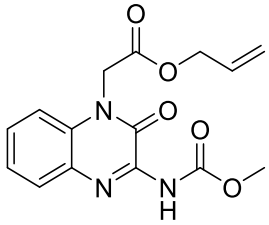
13: 20.9 mg, (PE/EtOAc = 75:25), white solid, yield: 57%;

IR (neat) *v* 3285, 2359, 1737, 1507, 1360, 1174, 738, 583cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.60 (s, 1H), 7.91 – 7.85 (m, 1H), 7.38 – 7.32 (m, 5H), 7.27 (d, *J* = 3.9 Hz, 2H), 7.01 – 6.98 (m, 1H), 5.21 (s, 2H), 5.09 (s, 2H), 3.86 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 166.5, 151.4, 150.8, 143.8, 134.7, 132.1, 129.8, 129.4, 128.70, 128.69, 128.4, 128.2, 124.9, 113.0, 67.9 , 53.0 , 44.2.

HRMS (ESI) m/z: $[M+K]^+$Calcd for $C_{19}H_{17}KN_3O_5^+$406.0800, Found 406.0801.



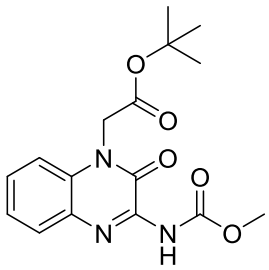
14: 20.0 mg, (PE/EtOAc = 75:25), white solid, yield: 63%;

IR (neat) *v* 3272, 2955, 1748, 1521, 1361, 1185, 758, 562cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.60 (s, 1H), 7.88 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.36 (td, *J* = 7.6, 1.3 Hz, 1H), 7.06 (dd, *J* = 8.3, 1.3 Hz, 1H), 5.94 – 5.80 (m, 1H), 5.34 – 5.22 (m, 2H), 5.09 (s, 2H), 4.68 (dd, *J* = 5.8, 1.4 Hz, 2H), 3.87 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 166.3, 151.4, 150.8, 143.8, 132.1, 131.0, 129.8, 129.4,128.2, 124.9, 119.4, 113.0, 66.6, 52.9, 44.1.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{15}H_{15}N_3NaO_5^+$340.0904, Found 340.0908.



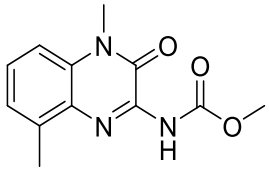
15: 19.3 mg, (PE/EtOAc = 75:25), white solid, yield: 58%;

IR (neat) *v* 3274, 2959, 1761, 1525, 1363, 1153, 750, 577cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.61 (s, 1H), 7.88 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.36 (td, *J* = 7.7, 1.3 Hz, 1H), 7.06 (dd, *J* = 8.3, 1.3 Hz, 1H), 4.96 (s, 2H), 3.87 (s, 3H), 1.45 (s, 9H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 165.6, 151.5, 150.8, 143.8, 132.1, 129.9, 129.4, 128.1, 124.7, 113.1, 83.5, 52.9, 44.9, 27.9.

HRMS (ESI) m/z: [M+Na]$^{+}$Calcd for $C_{16}H_{19}N_3NaO_5^{+}$356.1217, Found 356.1235.



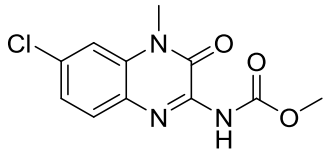
16: 22.2 mg, (PE/EtOAc = 85:15), white solid, yield: 90%;

IR (neat) *v* 3273, 2919, 1752, 1519, 1373, 1173, 764, 547cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.62 (s, 1H), 7.34 (dd, *J* = 8.3, 7.5 Hz, 1H), 7.26 – 7.20 (m, 1H), 7.13 (dt, *J* = 8.1, 0.9 Hz, 1H), 3.87 (s, 3H), 3.74 (s, 3H), 2.69 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.7, 150.8, 142.5, 137.7, 130.9, 130.5, 127.6, 125.8, 111.4, 52.7, 29.9, 17.4.

HRMS (ESI) m/z: [M+Na]$^{+}$Calcd for $C_{12}H_{13}N_3NaO_3^{+}$270.0849, Found 270.0858.



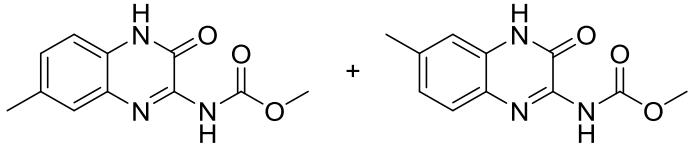
17: 20.0 mg, (PE/EtOAc = 75:25), white solid, yield: 75%;

IR (neat) *v* 3259, 2918, 1755, 1506, 1277, 1178, 747, 572cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.68 (s, 1H), 7.85 (d, *J* = 2.4 Hz, 1H), 7.40 (dd, *J* = 8.9, 2.4 Hz, 1H), 7.21 (d, *J* = 8.9 Hz, 1H), 3.87 (s, 3H), 3.73 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.3, 150.6, 144.9, 132.8, 130.0, 129.4, 128.4, 127.9, 114.7, 53.0, 30.0.

HRMS (ESI) m/z: [M+Na]$^{+}$Calcd for $C_{11}H_{10}ClN_3NaO_3^{+}$ 290.0303, Found 290.0312.



18: 18.6 mg, (PE/EtOAc = 65:35), white solid, yield: 80%;

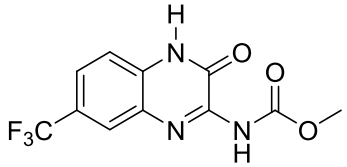
It is a mixture of 6-methylquinoxalinone and 7-methylquinoxalinone (the ratio is about 2:1).

IR (neat) *v* 3382, 2848, 1742, 1491, 1386, 1142, 737, 564cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 12.05 (s, 1H), 8.55 (s, 1H), 7.69 – 7.59 (m, 1H), 7.23 – 7.07 (m, 2H), 3.88 (s, 3H), 2.42 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 152.0, 151.7 – 151.6 (m), 144.3, 143.6, 138.8, 134.9, 131.9, 130.0, 129.2, 128.3, 127.8, 127.6, 126.4, 126.2, 115.5, 115.4, 52.9, 21.4, 21.0.

HRMS (ESI) m/z: [M+K]$^{+}$Calcd for $C_{11}H_{11}KN_3O_3^{+}$ 272.0432, Found 272.0420.



19: 17.0 mg, (PE/EtOAc = 70:30), white solid, yield: 59%;

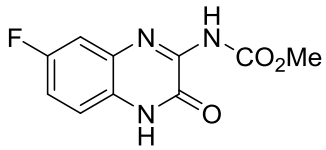
IR (neat) *v* 3255, 2956, 1738, 1528, 1324, 1161, 765, 567cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.92 (s, 1H), 9.52 (s, 1H), 7.80 (d, *J* = 2.0 Hz, 1H), 7.68 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.40 (d, *J* = 8.5 Hz, 1H), 3.73 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 151.7, 151.3, 147.3, 133.1, 131.2, 124.6 (q, $^{1}J_{C\text{-}F}$ = 270.0 Hz), 124.5 (q, $^{2}J_{C\text{-}F}$ = 32.0 Hz), 124.2 – 124.0 (m), 116.9 , 52.9.

^{19}F NMR (376 MHz, DMSO-d_6) δ -60.27 .

HRMS (ESI) m/z: [M+K]$^{+}$Calcd for $C_{11}H_8F_3KN_3O_3^{+}$ 326.0149, Found 326.0153.



20:12.6 mg, (PE/EtOAc = 65:35), white solid, yield: 53%;

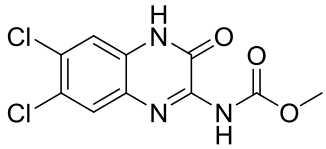
IR (neat) *v* 3355, 2957, 1736, 1516, 1355, 1148, 741, 590cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.68 (s, 1H), 9.38 (s, 1H), 7.40 – 7.34 (m, 1H), 7.33 – 7.24 (m, 2H), 3.73 (s, 3H);

^{13}C NMR (100 MHz, DMSO-d_6) δ158.7 (d, $^{1}J_{C\text{-}F}$ = 237.6 Hz), 151.7, 150.8, 147.0, 132.3 (d, $^{3}J_{C\text{-}F}$= 12.1 Hz), 127.0, 117.0 (d, $^{3}J_{C\text{-}F}$= 9.5 Hz), 115.6 (d, $^{2}J_{C\text{-}F}$ = 24.1 Hz), 112.7 (d, $^{2}J_{C\text{-}F}$= 22.9 Hz), 52.9;

^{19}F NMR (376 MHz, DMSO-d_6) δ-118.9;

HRMS (ESI) m/z: [M+Na]$^{+}$Calcd for $C_{10}H_8FN_3NaO_3^{+}$260.0442, Found 260.0451.



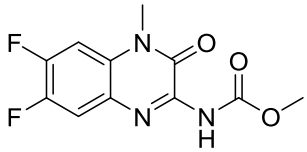
21: 11.4 mg, (PE/EtOAc = 80:20), white solid, yield: 40%;

IR (neat) *v* 2955, 2849, 1695, 1490, 1312, 1076, 786, 564cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.66 (s, 1H), 7.36 (s, 1H), 3.72 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6) δ 151.8, 146.5, 136.1, 132.4, 130.1, 128.5, 127.5, 123.4, 119.9, 52.8.

HRMS (ESI) m/z: [M+Na]$^{+}$Calcd for $C_{10}H_7Cl_2N_3NaO_3^{+}$309.9757, Found 309.9766.



22: 22.1 mg, (PE/EtOAc = 80:20), white solid, yield: 82%;

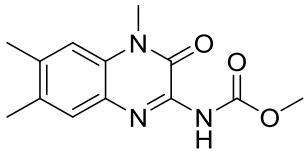
IR (neat) *v* 3253,2359, 1742, 1507, 1372, 1181, 748, 563cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.64 (s, 1H), 7.70 – 7.65 (m, 1H), 7.13 – 7.08 (m, 1H), 3.87 (s, 3H), 3.71 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.3, 150.5, 149.8 (dd, $^{1}J_{C\text{-}F}$ = 249.2Hz, $^{2}J_{C\text{-}F}$ =14.2 Hz), 147.2 (dd, $^{1}J_{C\text{-}F}$ = 245.5 Hz, $^{2}J_{C\text{-}F}$ =13.6 Hz), 144.3, 128.4 (dd, $^{3}J_{C\text{-}F}$ = 9.8 Hz, $^{4}J_{C\text{-}F}$ =3.0 Hz), 127.5 (dd, $^{3}J_{C\text{-}F}$ = 8.4, $^{4}J_{C\text{-}F}$ =1.7 Hz), 116.7 (dd, $^{2}J_{C\text{-}F}$ = 18.6, $^{3}J_{C\text{-}F}$ =1.8 Hz), 102.4 (d, $^{2}J_{C\text{-}F}$ = 23.3 Hz), 53.0, 30.3.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -134.2 (d, *J* = 22.5 Hz), -140.9 (d, *J* = 22.4 Hz).

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{11}H_9F_2N_3NaO_3^+$ 292.0504, Found 292.0511.



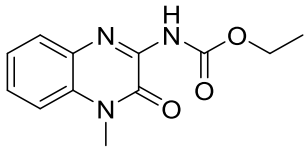
23: 15.7 mg, (PE/EtOAc = 75:25), white solid, yield: 60%;

IR (neat) *v* 3237, 2918, 1739, 1506, 1360, 1190, 765, 565cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.61 (s, 1H), 7.63 (s, 1H), 7.04 (s, 1H), 3.85 (s, 3H), 3.72 (s, 3H), 2.39 (s, 3H), 2.33 (s, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.7, 150.9, 143.4, 137.5, 133.5, 130.1, 129.3, 128.6, 114.2, 52.8, 29.7, 20.3, 19.2.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{13}H_{15}N_3NaO_3^+$284.1006, Found 284.1016.



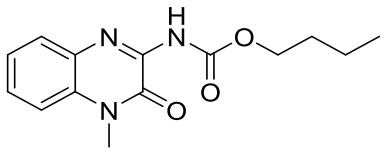
24:35.0 mg, (PE/EtOAc = 80:20), yellow solid, yield: 71%;

IR (neat) *v* 3270, 2976, 1748, 1524, 1365, 1186, 763, 556cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.64 (s, 1H), 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.48 – 7.42 (m, 1H), 7.38 – 7.33 (m, 1H), 7.30 – 7.27 (m, 1H), 4.32 (q, *J* = 7.1 Hz, 2H), 3.75 (s, 3H), 1.36 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.02, 150.96, 144.2, 132.1, 130.7, 129.1, 127.9, 124.5, 113.6, 62.0, 29.8, 14.4.

HRMS (ESI) m/z: $[M+K]^+$Calcd for $C_{12}H_{13}KN_3O_3^+$ 286.0588, Found 286.0596.



25:42.4 mg, (PE/EtOAc = 85:15), white solid, yield: 77%;

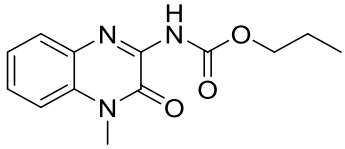
IR (neat) *v* 3269, 2953, 1749, 1506, 1353, 1179, 716, 557cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.63 (s, 1H), 7.86 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.48 – 7.43 (m,

1H), 7.36 (t, *J* = 7.4 Hz, 1H), 7.29 (dd, *J* = 8.3, 1.2 Hz, 1H), 4.26 (t, *J* = 6.6 Hz, 2H), 3.76 (s, 3H), 1.73 – 1.69 (m, 2H), 1.48 – 1.42 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.2, 151.0, 144.2, 132.1, 130.7, 129.1, 127.9, 124.6, 113.6, 65.8, 30.7, 29.7, 19.0, 13.7.

HRMS (ESI) m/z: $[M+K]^+$Calcd for $C_{14}H_{17}KN_3O_3^+$ 314.0901, Found 314.0910.



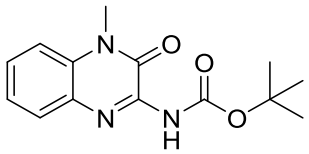
26:36.5 mg, (PE/EtOAc = 85:15), white solid, yield: 70%;

IR (neat) *v* 3249, 2964, 1750, 1516, 1360, 1179, 744, 557cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.65 (s, 1H), 7.86 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.45 (ddd, *J* = 8.7, 7.4, 1.5 Hz, 1H), 7.35 (m, 1H), 7.29 (dd, *J* = 8.9, 1.7 Hz, 1H), 4.22 (t, *J* = 6.7 Hz, 2H), 3.76 (s, 3H), 1.78 – 1.73 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.2, 151.0, 144.2, 132.1, 130.7, 129.1, 127.9, 124.6, 113.6, 67.6, 29.8, 22.1, 10.3.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{13}H_{15}N_3NaO_3^+$ 284.1006, Found 284.0979.

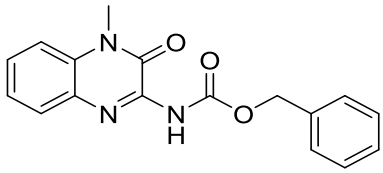


27: 25.8 mg, (PE/EtOAc = 85:15), yellow solid, yield: 94%;This is a known compound.[11]

IR (neat) *v*3388, 2919, 1750, 1508, 1364, 1137, 753, 553 cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.57 (s, 1H), 7.88 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.44 (m, 1H), 7.34 (m, 1H), 7.30 – 7.27 (m, 1H), 3.76 (s, 3H), 1.57 (s, 9H).

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{14}H_{17}N_3NaO_3^+$ 298.1162, Found 298.1173.



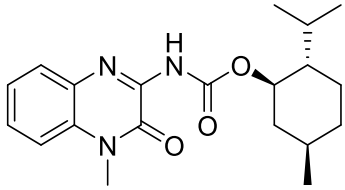
28:42.0 mg, (PE/EtOAc = 75:25), white solid, yield: 68%;

IR (neat) *v* 3269,3022, 1759, 1514, 1369, 1176, 743, 597cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.75 (s, 1H), 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.50 – 7.42 (m, 3H), 7.41 – 7.32 (m, 4H), 7.29 – 7.26 (m, 1H), 5.28 (s, 2H), 3.73 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 150.89, 150.87, 144.1, 135.5, 132.0, 130.7, 129.1, 128.6, 128.5, 128.0 , 124.6, 113.6, 67.6, 29.8.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{17}H_{15}N_3NaO_3^+$332.1006, Found 332.1012.



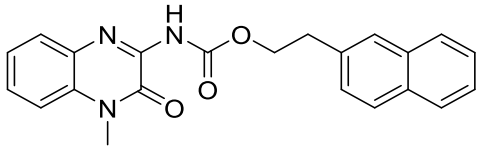
29:60.7mg, (PE/EtOAc = 95:5), white solid, yield: 85%;

IR (neat) *v* 3244, 2919, 1744, 1514, 1311, 1154, 737, 595cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.62 (s, 1H), 7.86 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.44 (ddd, *J* = 8.6, 7.4, 1.5 Hz, 1H), 7.35 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.29 (d, *J* = 1.3 Hz, 1H), 4.75 (td, *J* = 10.9, 4.4 Hz, 1H), 3.76 (s, 3H), 2.23 – 2.15 (m, 1H), 2.02 (qd, *J* = 6.9, 2.7 Hz, 1H), 1.75 – 1.67 (m, 2H), 1.53 (m, 1H), 1.43 (m, 1H), 1.26 (d, *J* = 3.8 Hz, 1H), 1.12 – 1.03 (m, 2H), 0.92 (dd, *J* = 6.8, 2.5 Hz, 6H), 0.81 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.0, 150.8, 144.3, 132.1, 130.6, 129.1, 127.8, 124.5, 113.5, 47.1, 41.0, 34.2, 31.4, 29.8, 26.2, 23.4, 22.0, 20.8, 16.4.

HRMS (ESI) m/z: $[M+K]^+$Calcd for $C_{20}H_{27}KN_3O_3^+$396.1684, Found 396.1683.



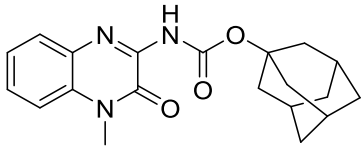
30:35.0 mg, (PE/EtOAc = 85:15), yellow solid, yield: 47%;

IR (neat) *v* 3282, 2848, 1749, 1507, 1356, 1176, 743, 576cm^{-1};

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.68 (s, 1H), 8.19 (d, *J* = 8.4 Hz, 1H), 7.88 (ddd, *J* = 12.1, 8.0, 1.5 Hz, 2H), 7.77 (q, *J* = 4.9 Hz, 1H), 7.56 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.52 – 7.45 (m, 2H), 7.44 – 7.41 (m, 2H), 7.37 (td, *J* = 7.7, 1.3 Hz, 1H), 7.30 (dd, *J* = 8.2, 1.3 Hz, 1H), 4.59 (t, *J* = 7.6 Hz, 2H), 3.76 (s, 3H), 3.54 (t, *J* = 7.6 Hz, 2H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 151.03, 150.94, 144.1, 133.9, 133.2, 132.06, 132.05, 130.7, 129.1, 128.8, 128.0, 127.6, 127.2, 126.3, 125.7, 125.5, 124.6, 123.7, 113.6, 65.8, 32.5, 29.8.

HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{22}H_{19}N_3NaO_3^+$396.1319, Found 396.1324.



31:50.8 mg, (PE/EtOAc = 93:7), white solid, yield: 72%;

IR (neat) *v* 3381, 2910, 1755, 1514, 1352, 1172, 735, 633cm^{-1};

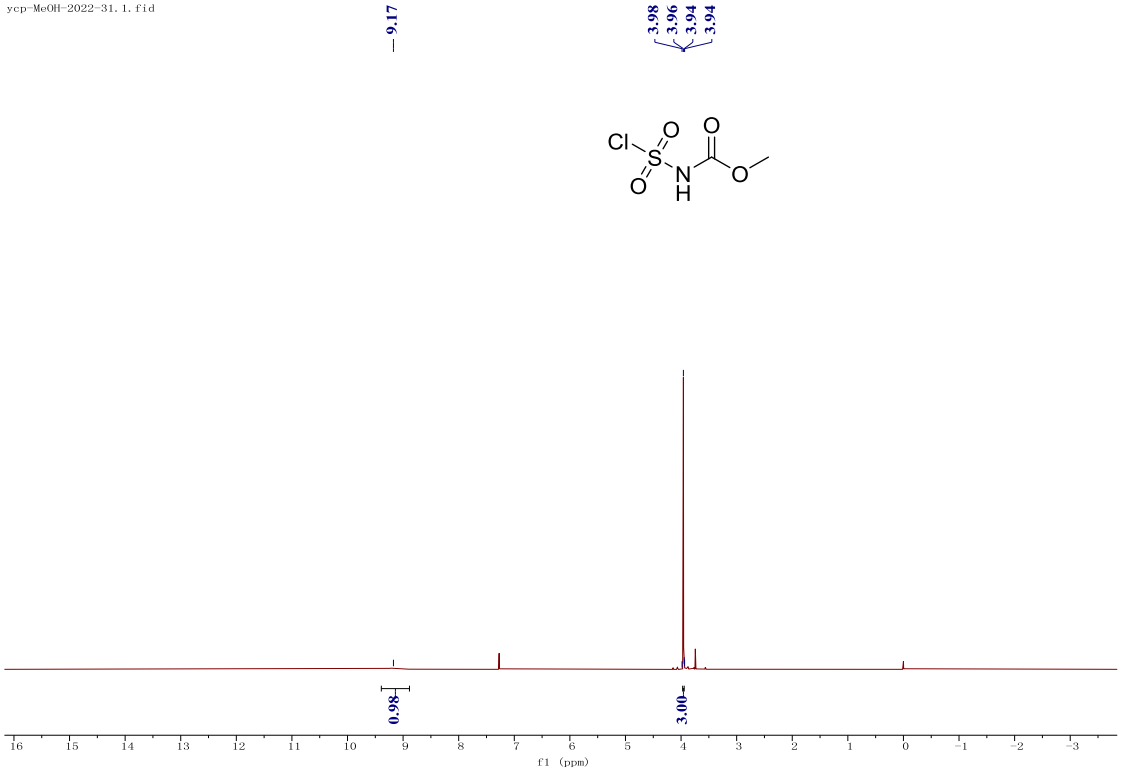
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.55 (s, 1H), 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.46 – 7.41(m, 1H), 7.34 (td, *J* = 7.7, 1.3 Hz, 1H), 7.29 – 7.28 (m, 1H), 3.75 (s, 3H), 2.21 (s, 9H), 1.68 (d, *J* = 3.3 Hz, 6H).

^{13}C NMR (100MHz, Chloroform-*d*) δ 151.0, 149.4, 144.5, 132.2, 130.6, 129.0, 127.6, 124.5, 113.5, 81.9, 41.4, 36.1, 30.9, 29.8.

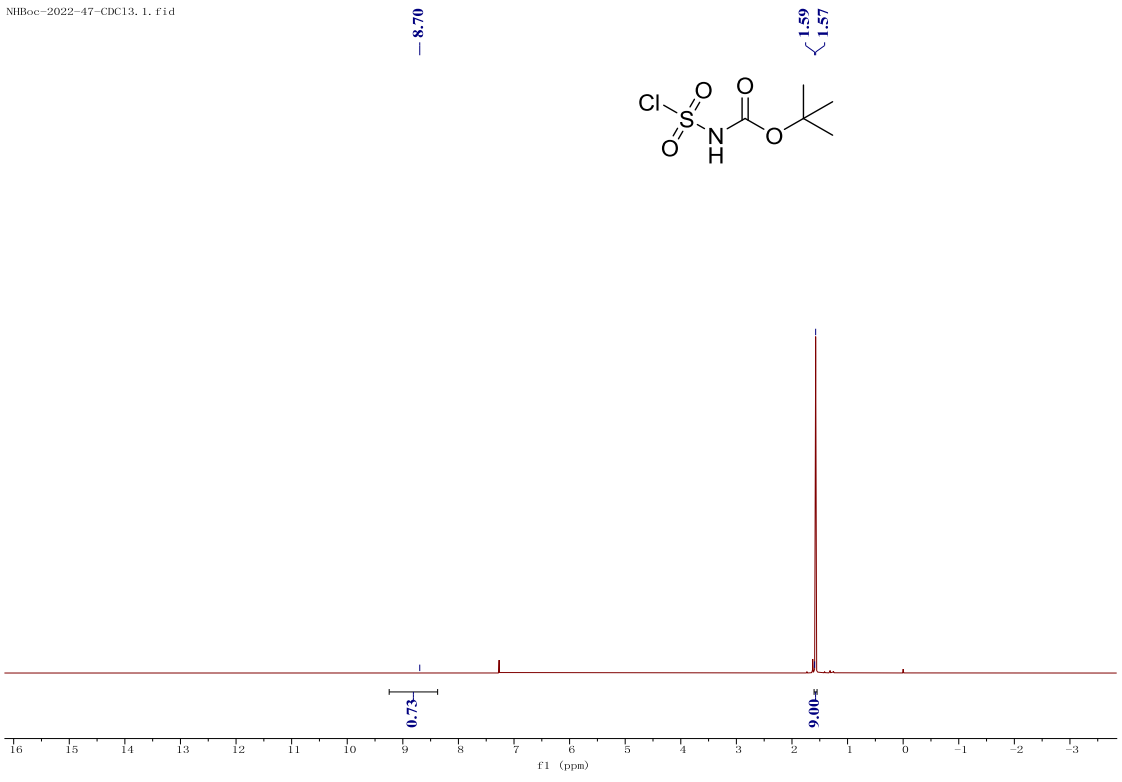
HRMS (ESI) m/z: $[M+Na]^+$Calcd for $C_{20}H_{23}N_3NaO_3^+$376.1632, Found 376.1634.

7. Copies of NMR spectra

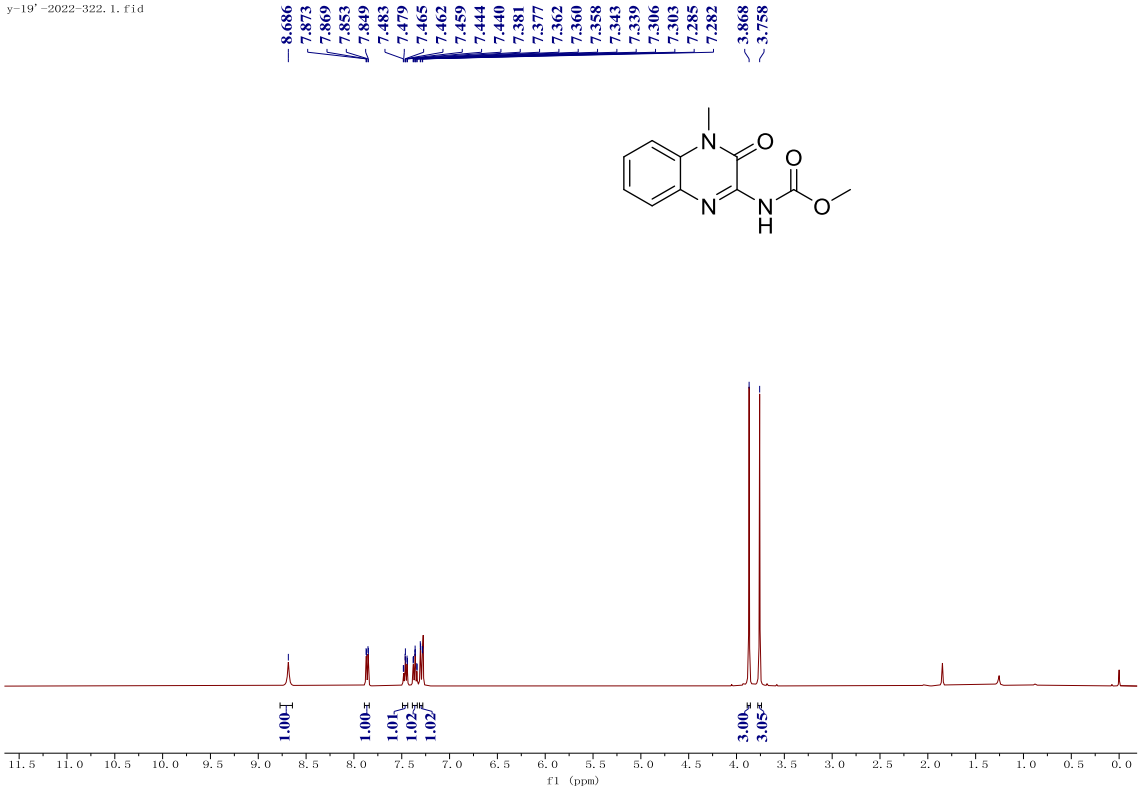
Substrate **2a**:^{1}H NMR ($CDCl_3$, 400 MHz)

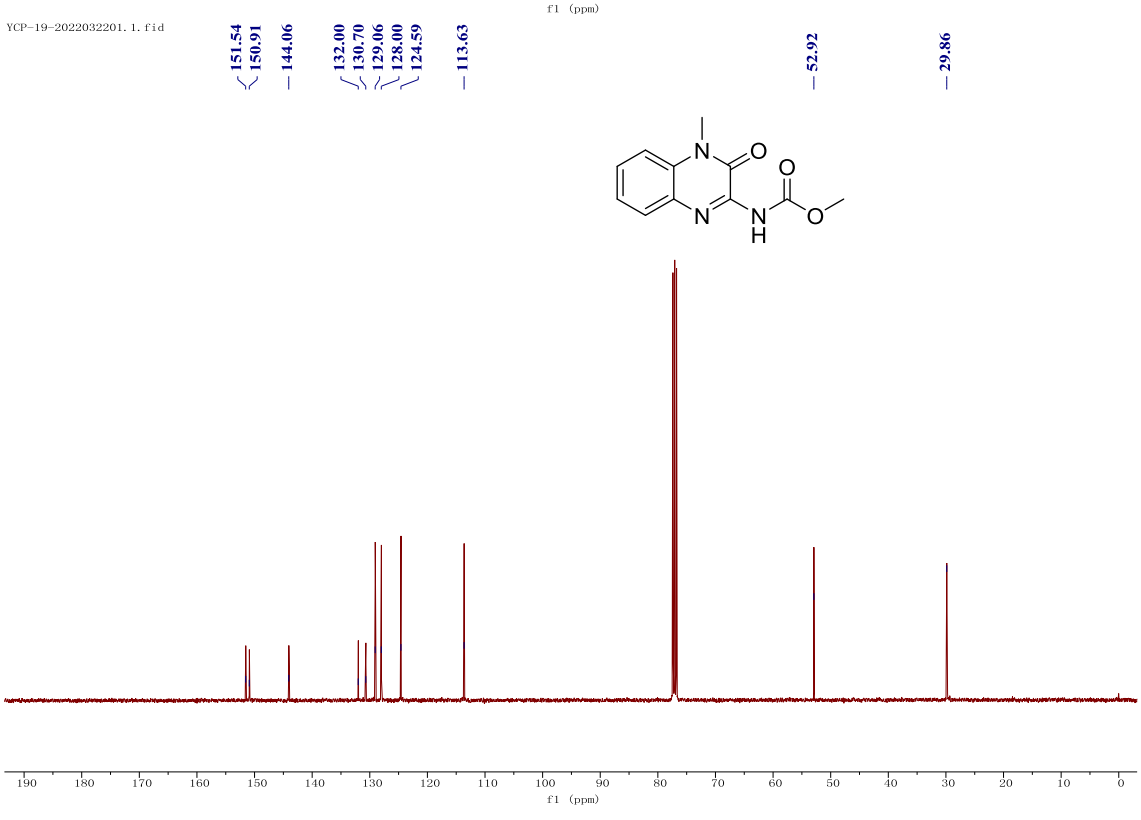


Substrate **2b**:^{1}H NMR ($CDCl_3$, 400 MHz)

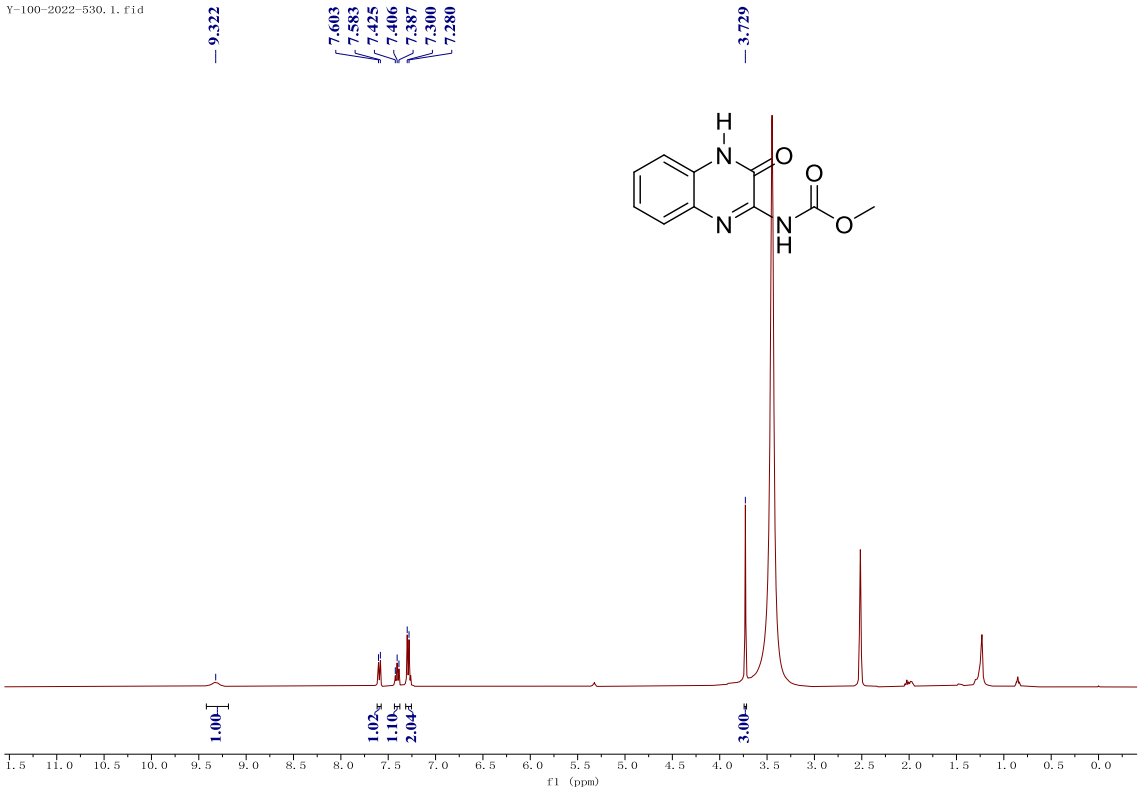


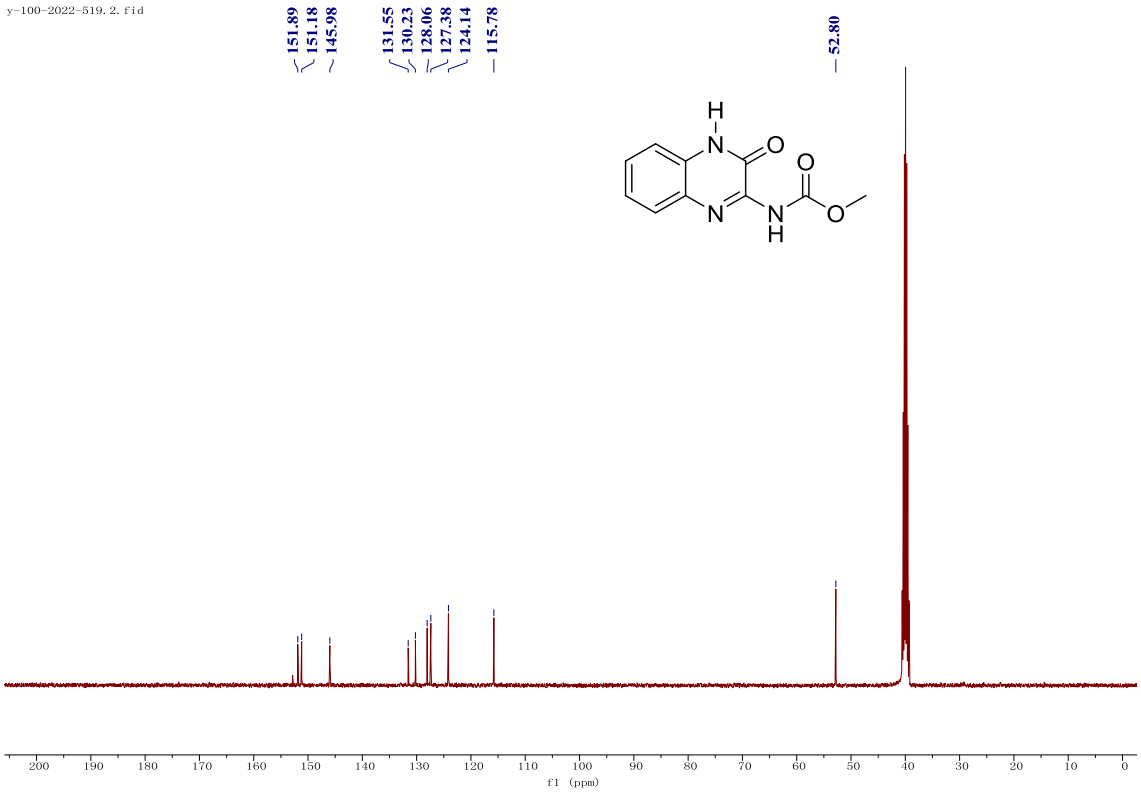
Product **3**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



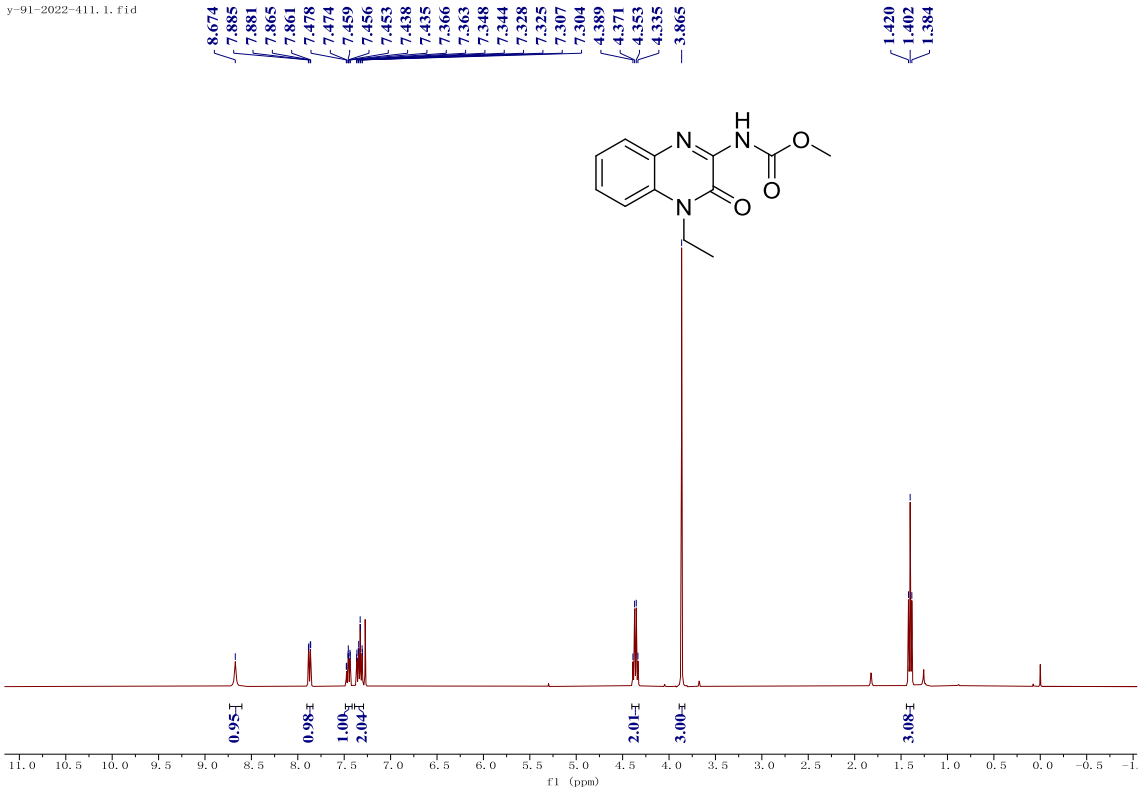


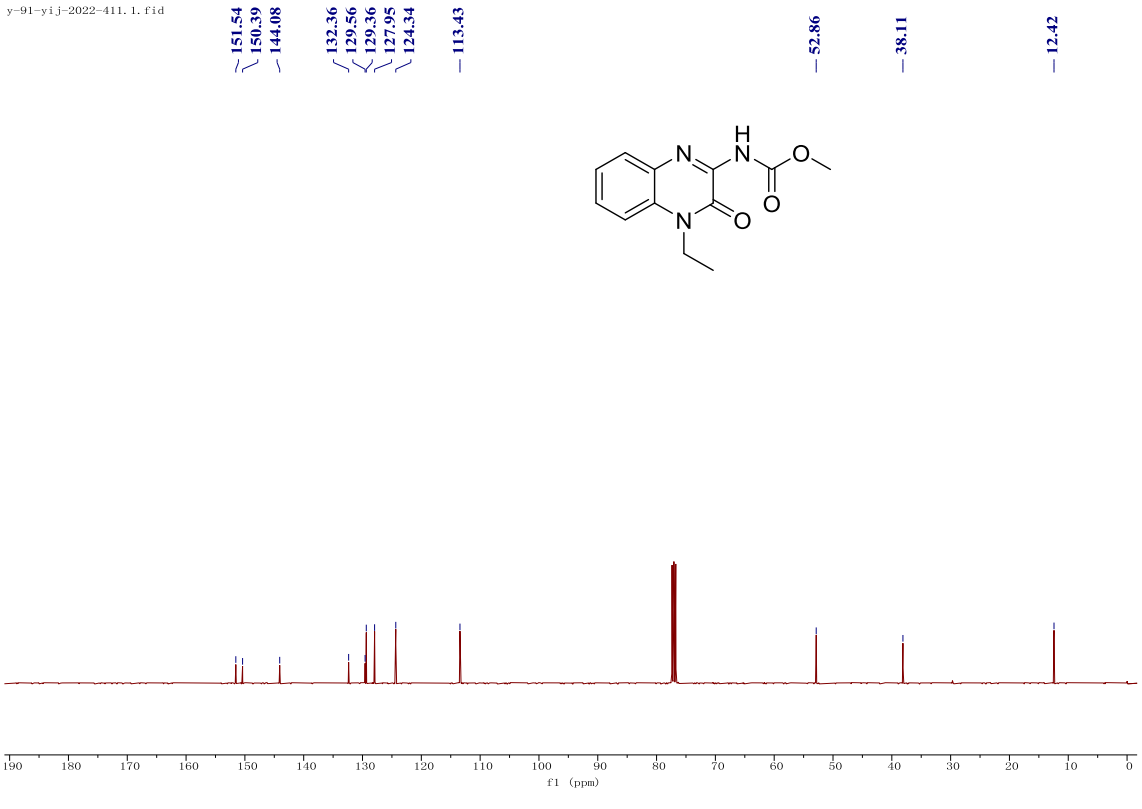
Product **4**: ^{1}H NMR (DMSO-d_6, 400 MHz), ^{13}C{^{1}H} NMR (DMSO-d_6, 100 MHz)



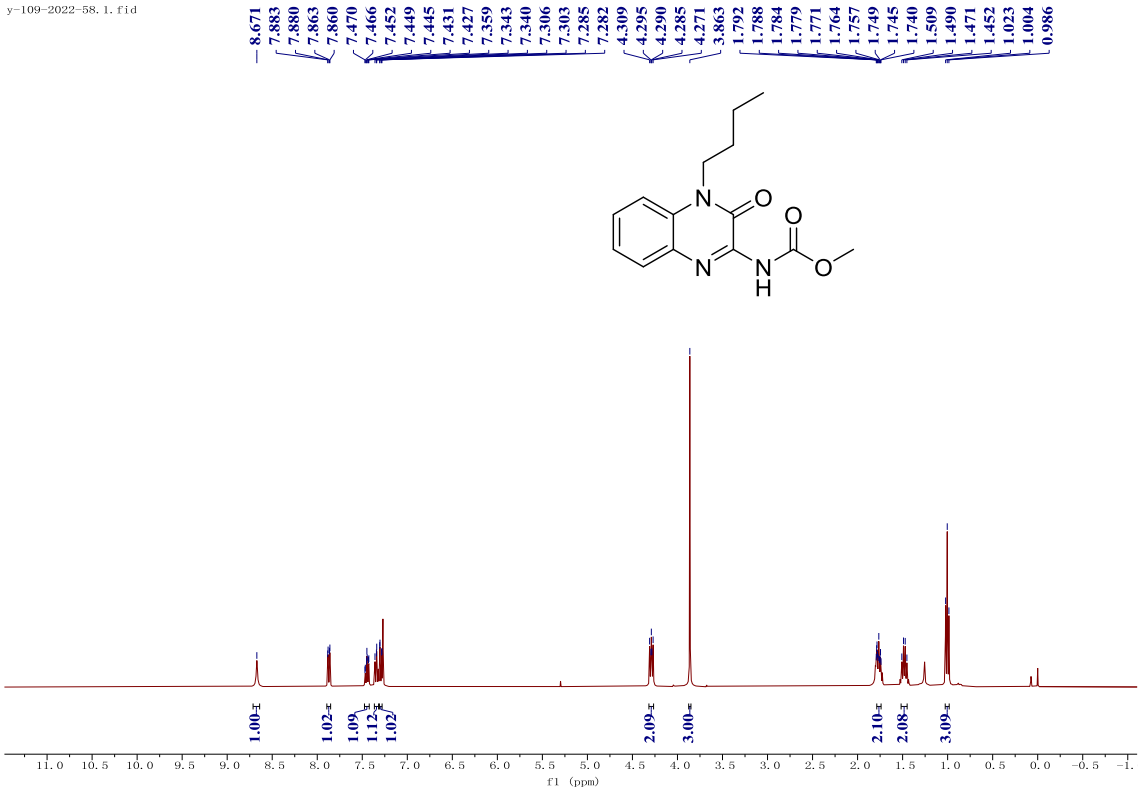


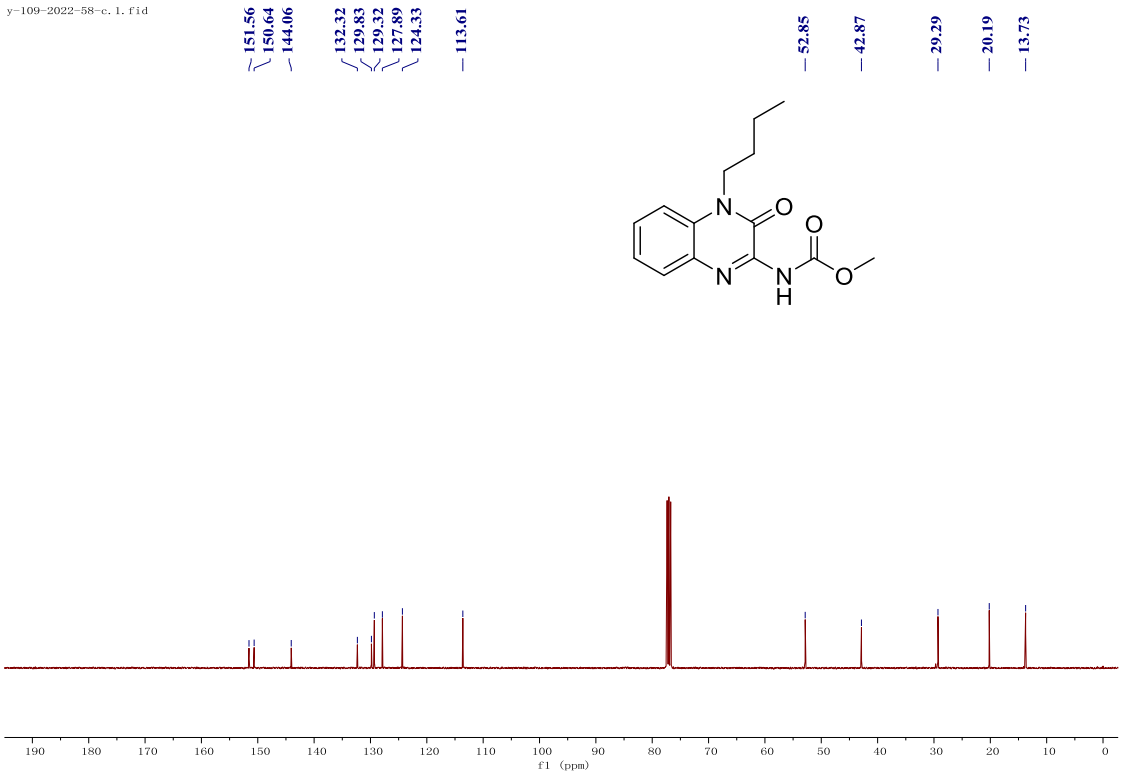
Product **5**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



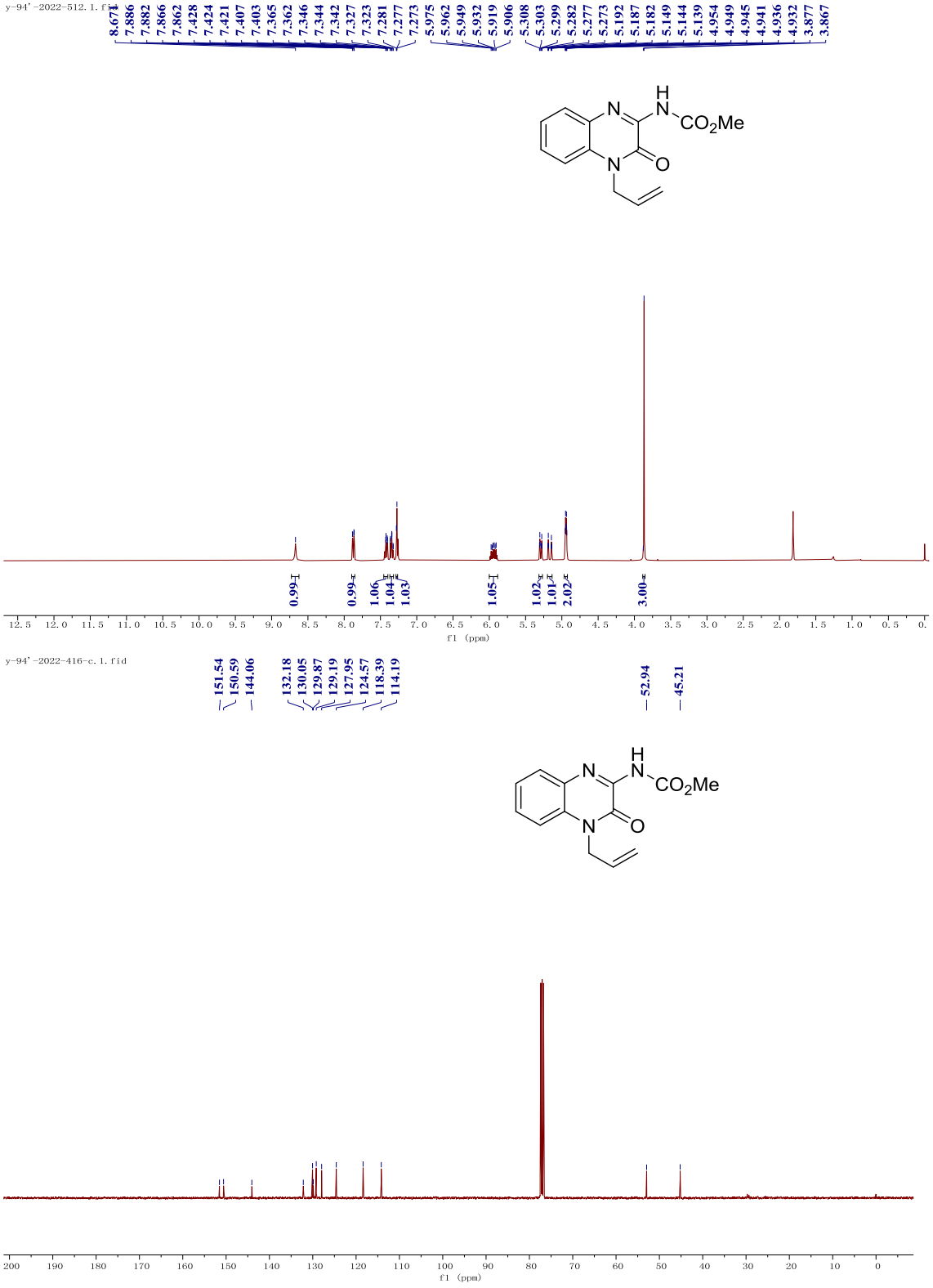


Product **6**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

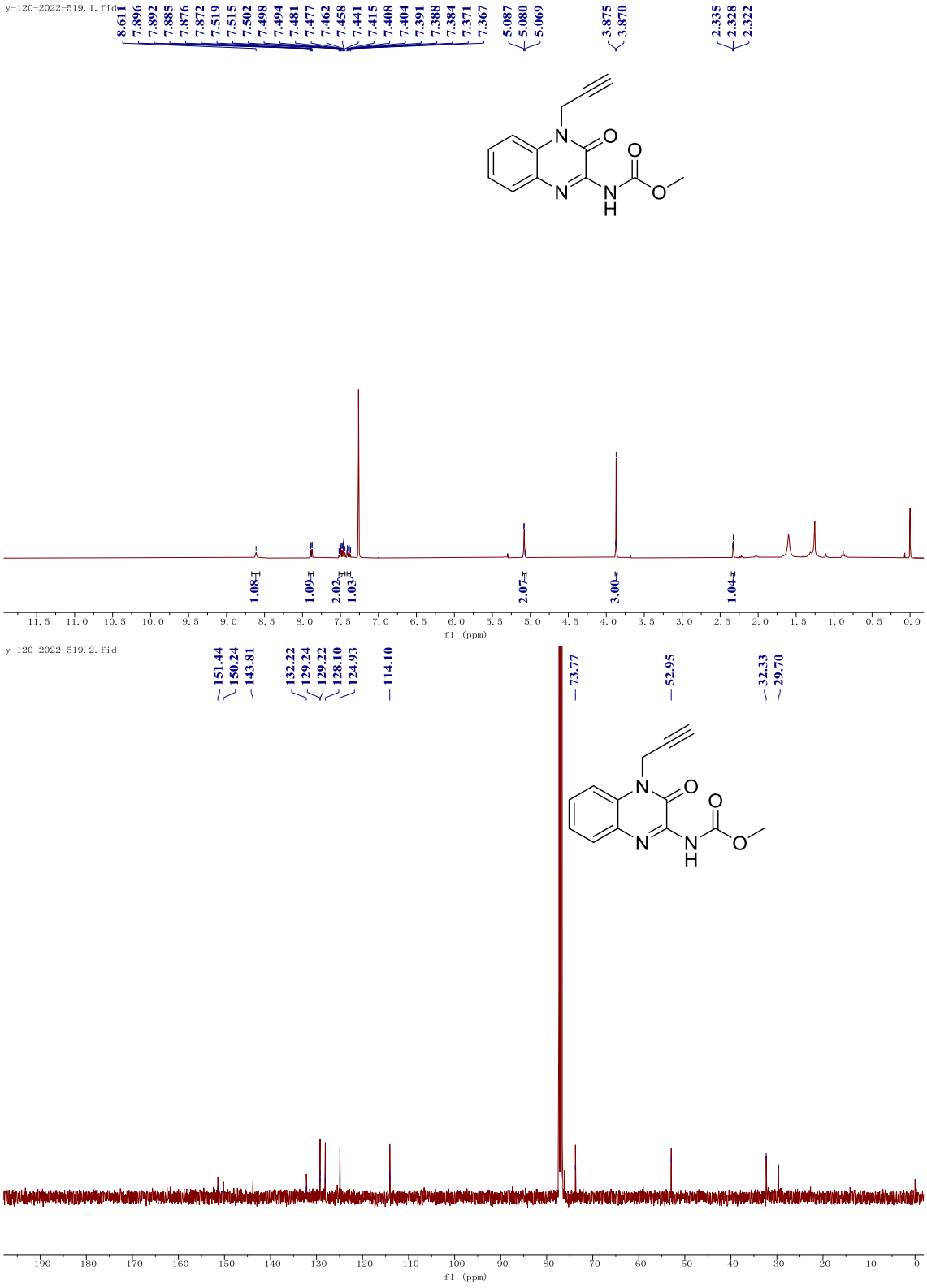




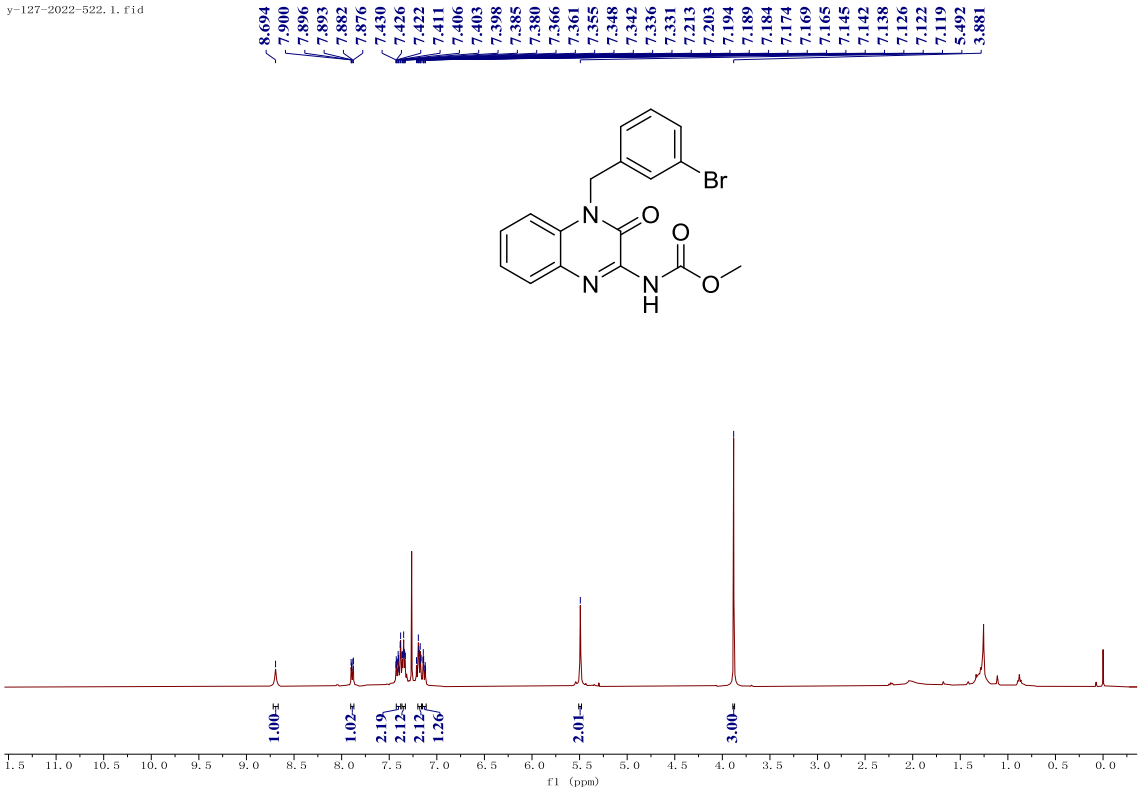
Product **7**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

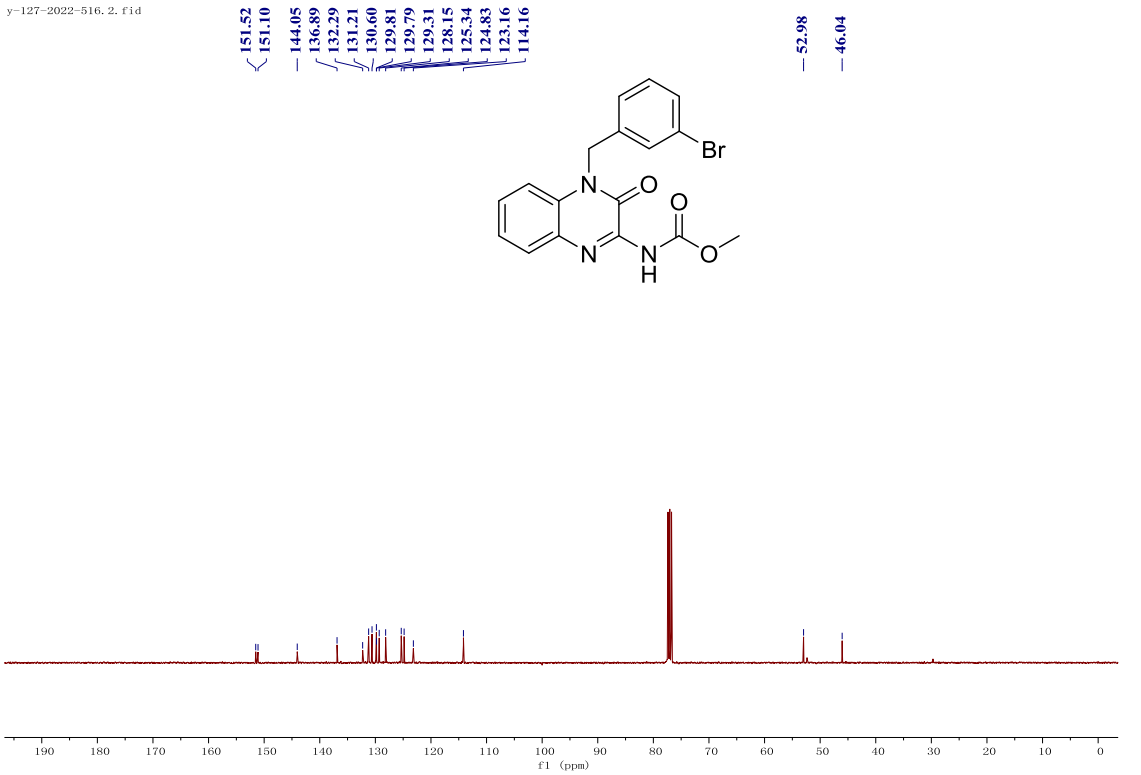


Product **8**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

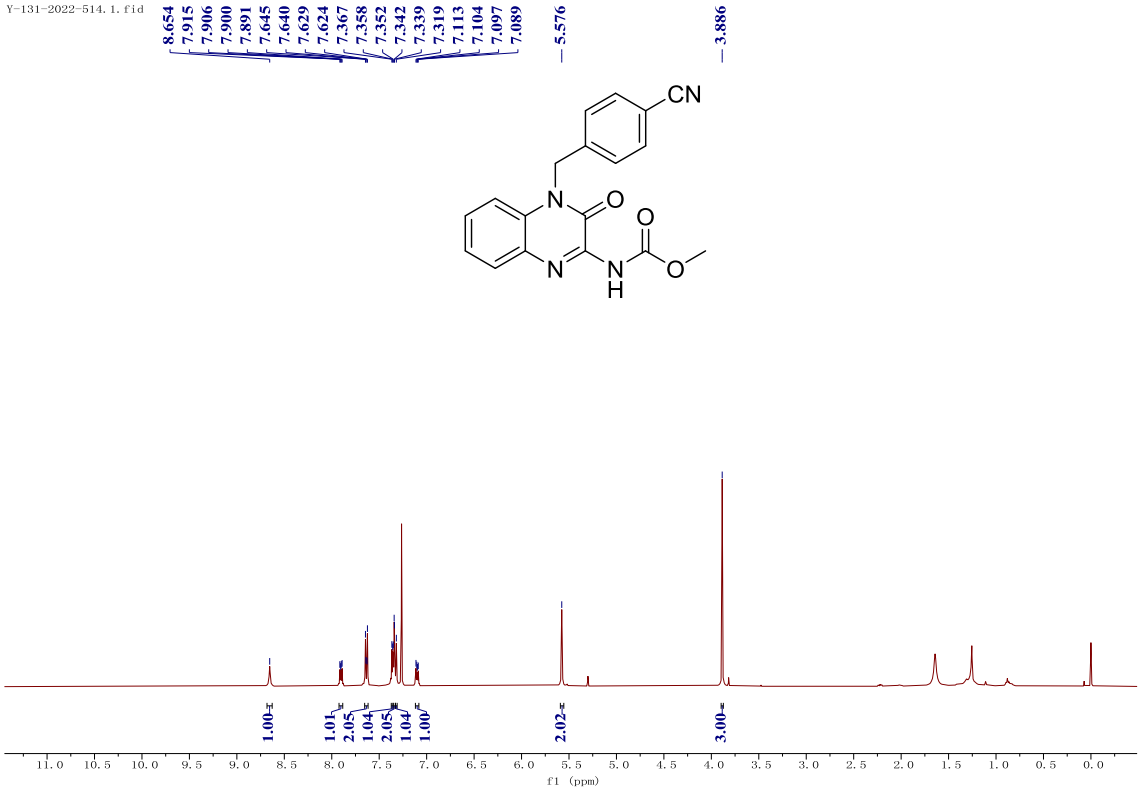


Product **9**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)





Product **10**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)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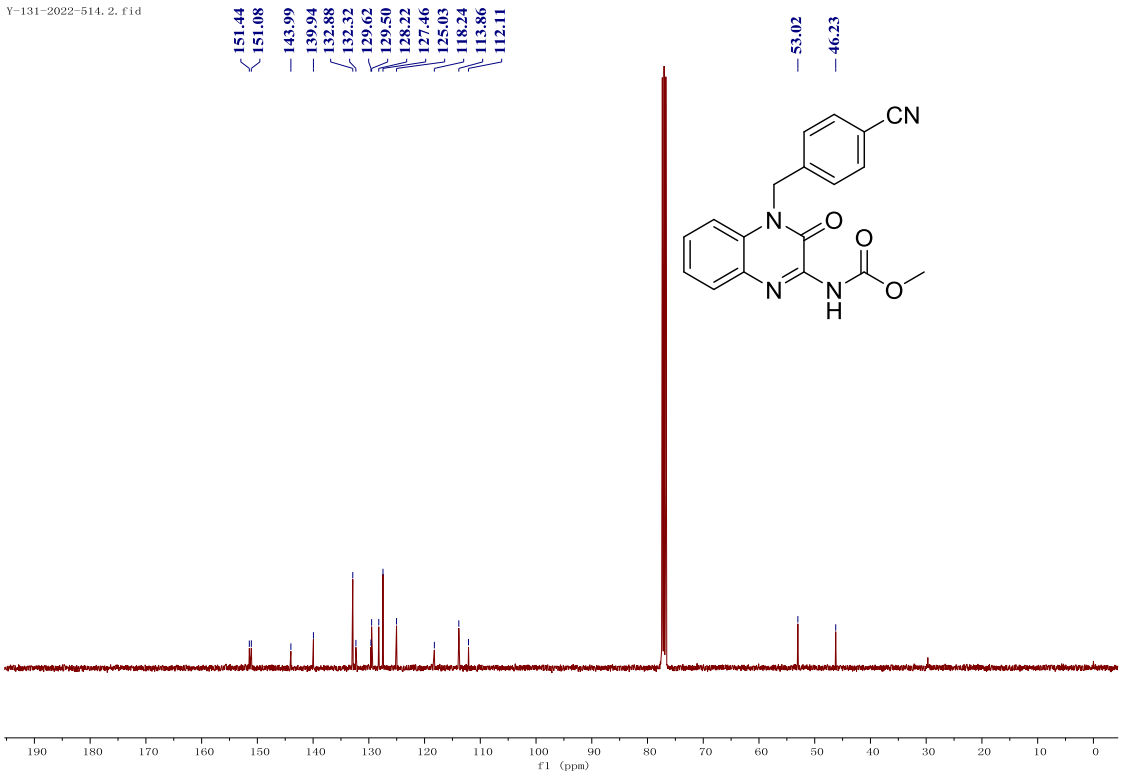
7.342
7.339
7.319
7.113
7.104
7.097
7.089
5.576
3.886
CN
N
O
O
N
N
H
O
1.00
1.01
2.05
1.04
2.05
1.04
1.00
2.02
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Y-131-2022-514.2.fid
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132.32
129.62
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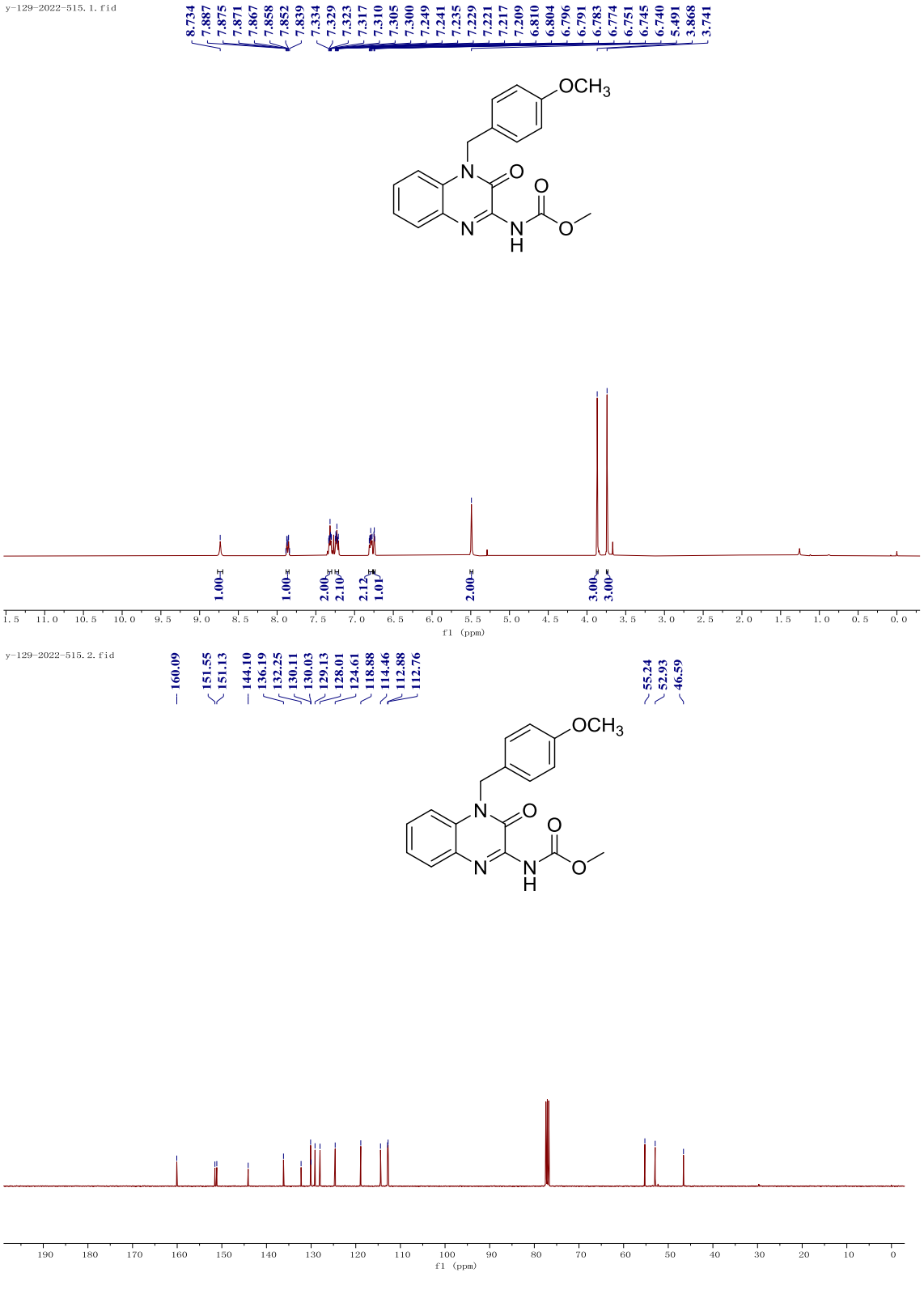
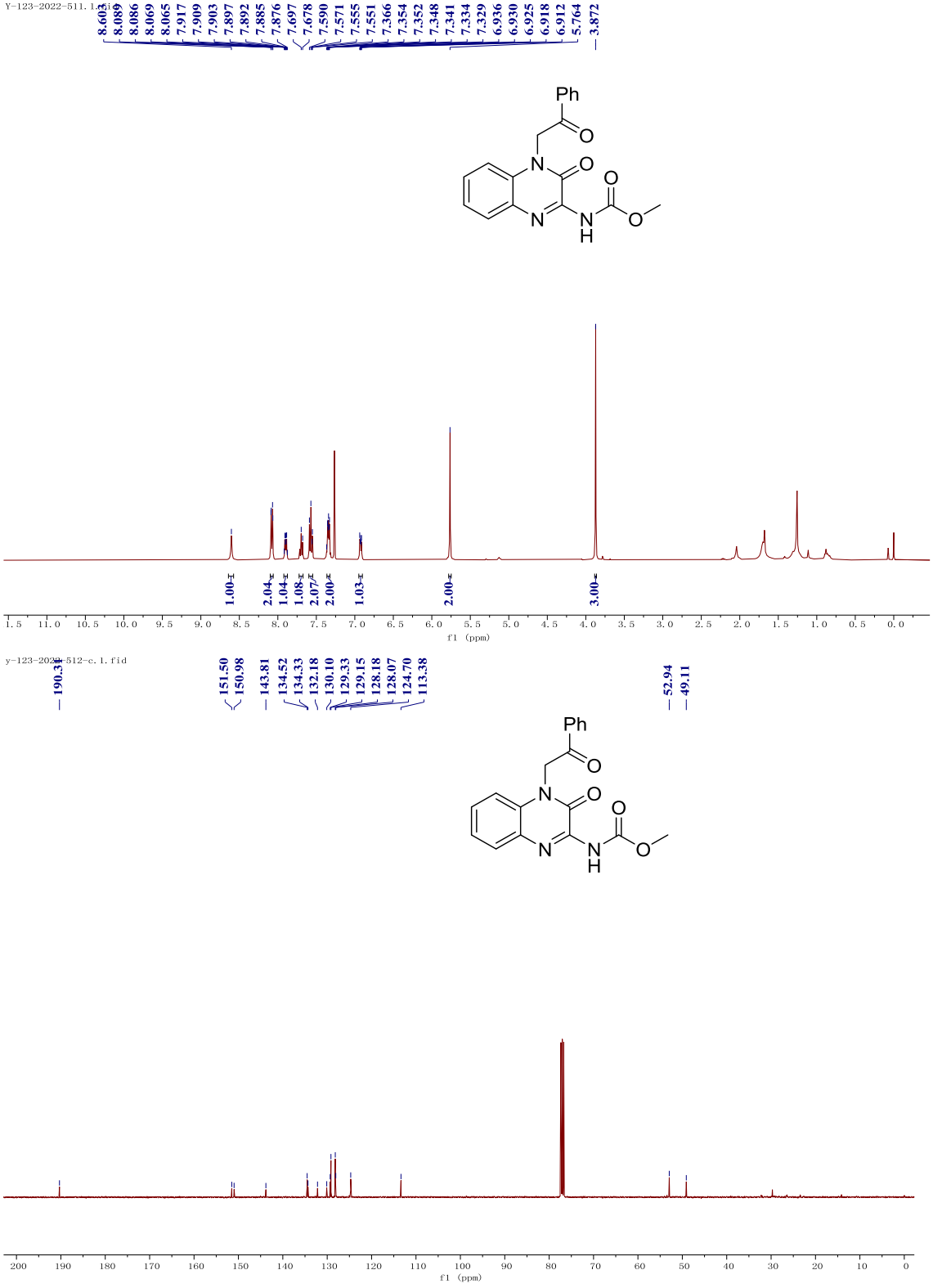
112.11
53.02
46.23
CN
N
O
O
N
N
H
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

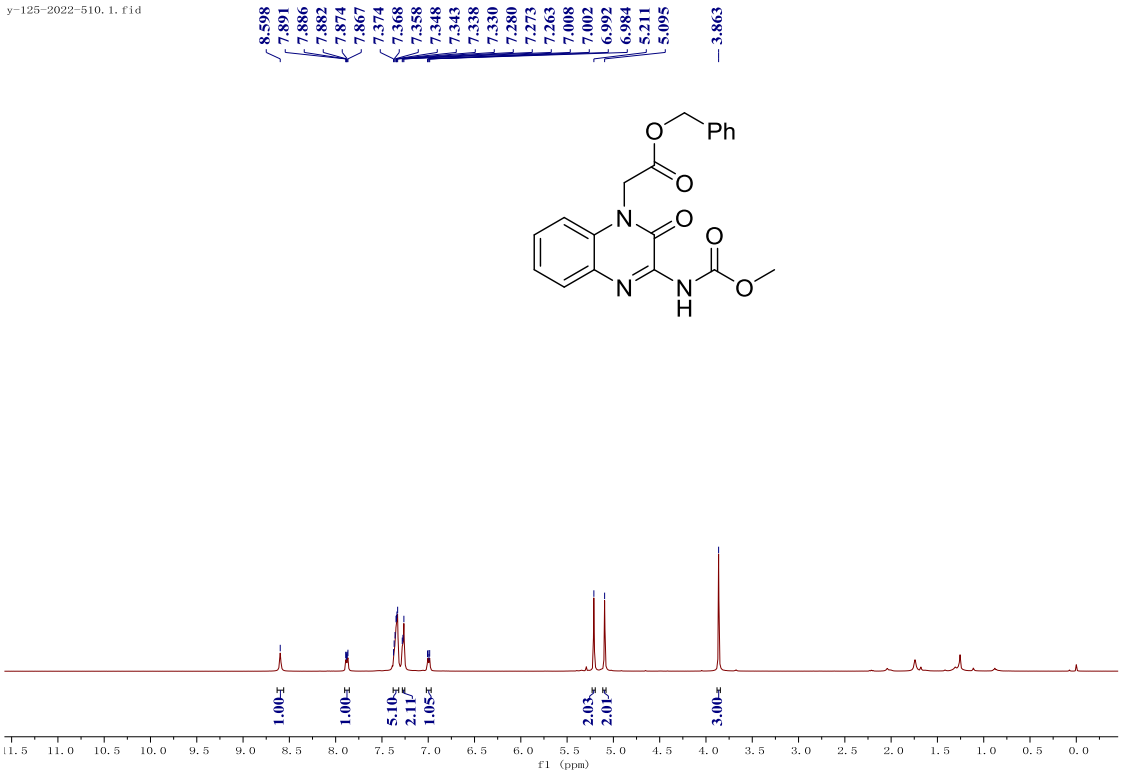
Product **11**: ^{1}H NMR ($CDCl_3$, 400 MHz), ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz)

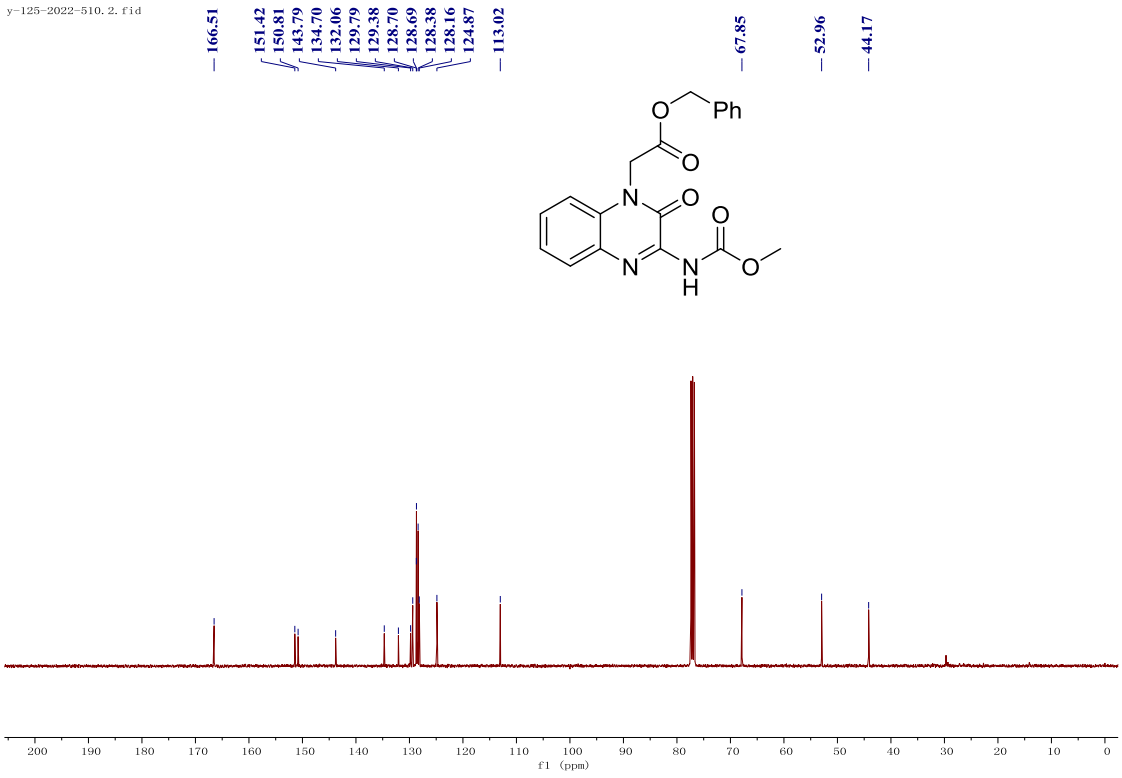


Product **12**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

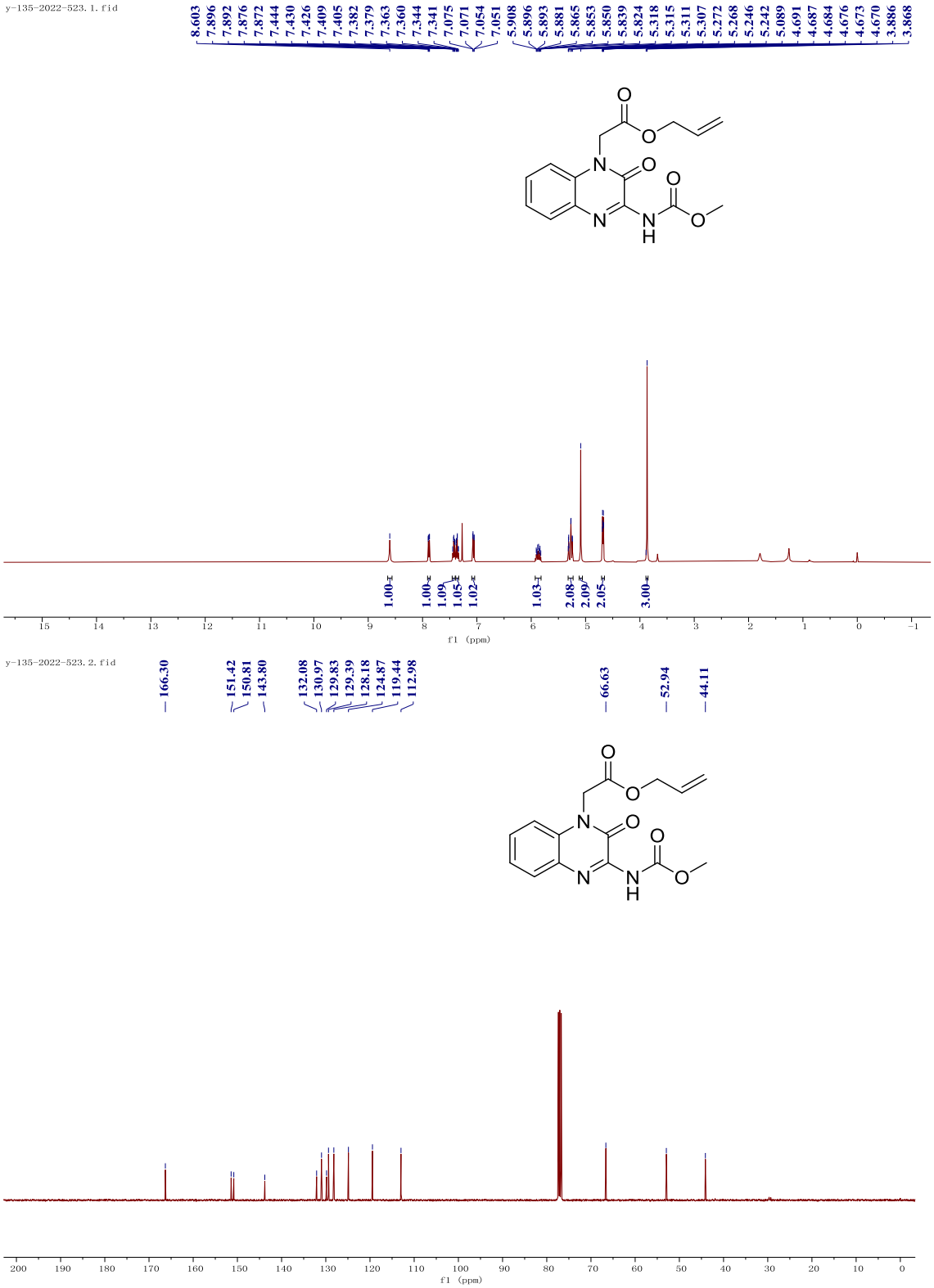


Product **13**: ^{1}H NMR ($CDCl_3$, 400 MHz), ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz)

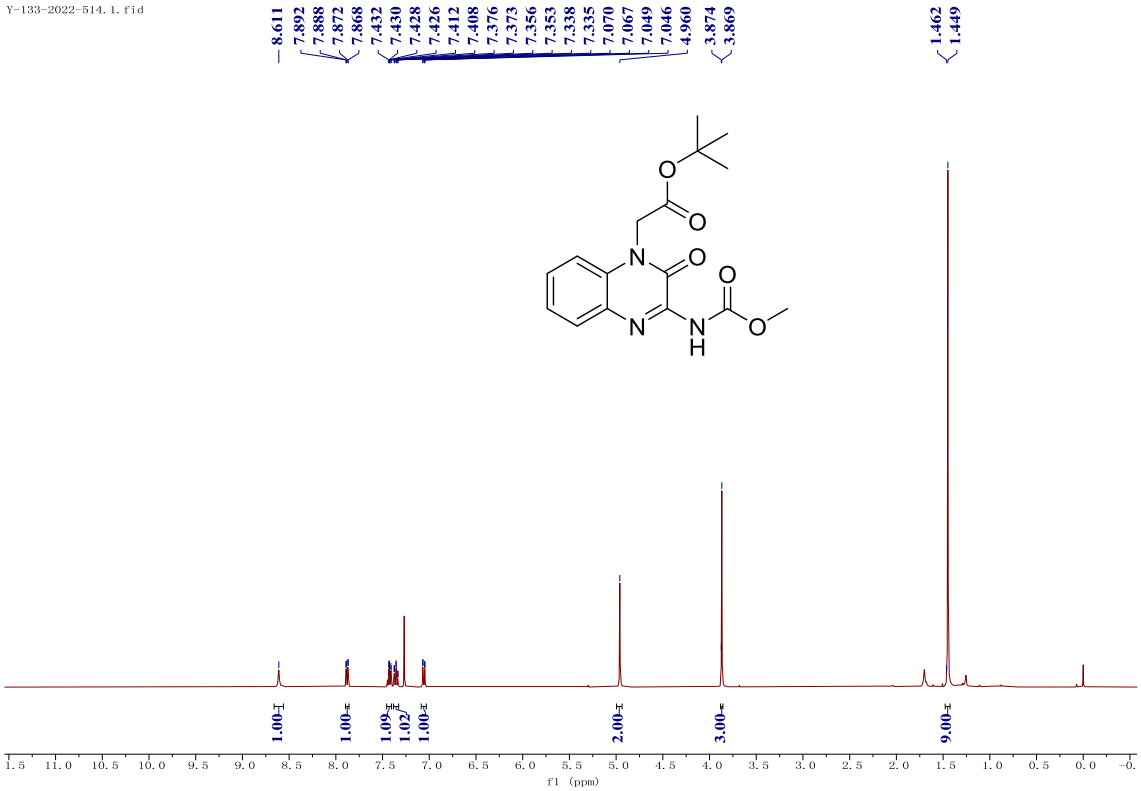


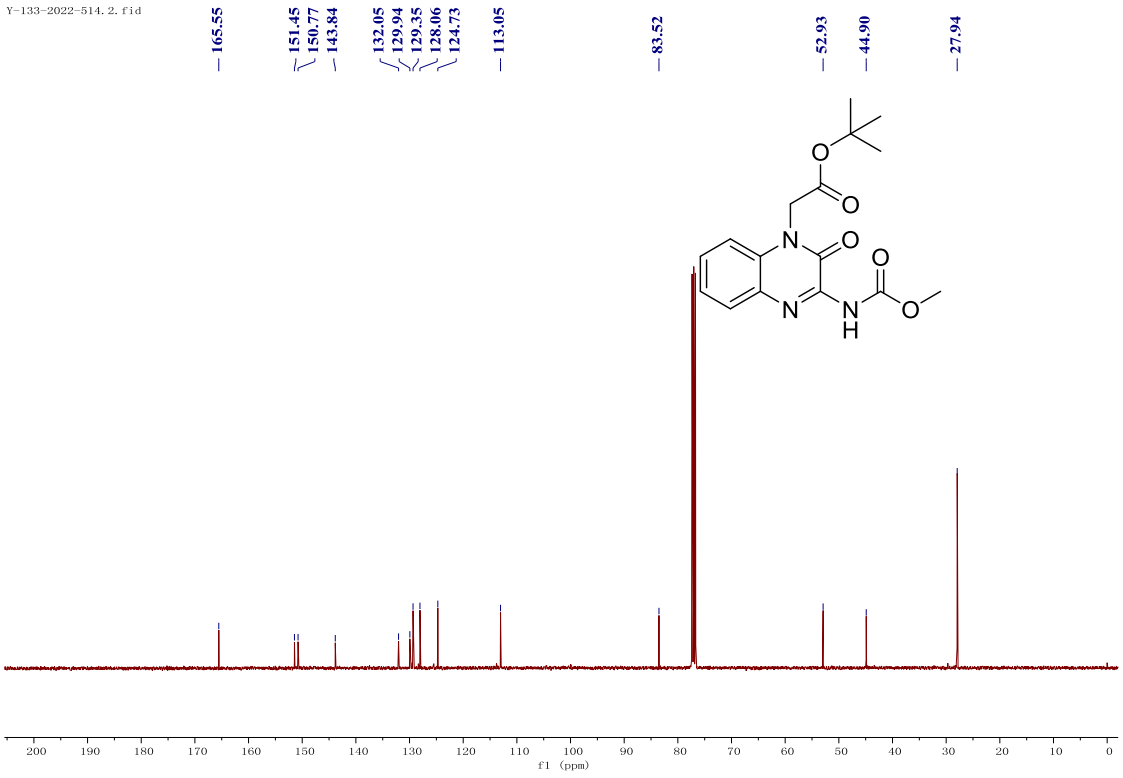


Product **14**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

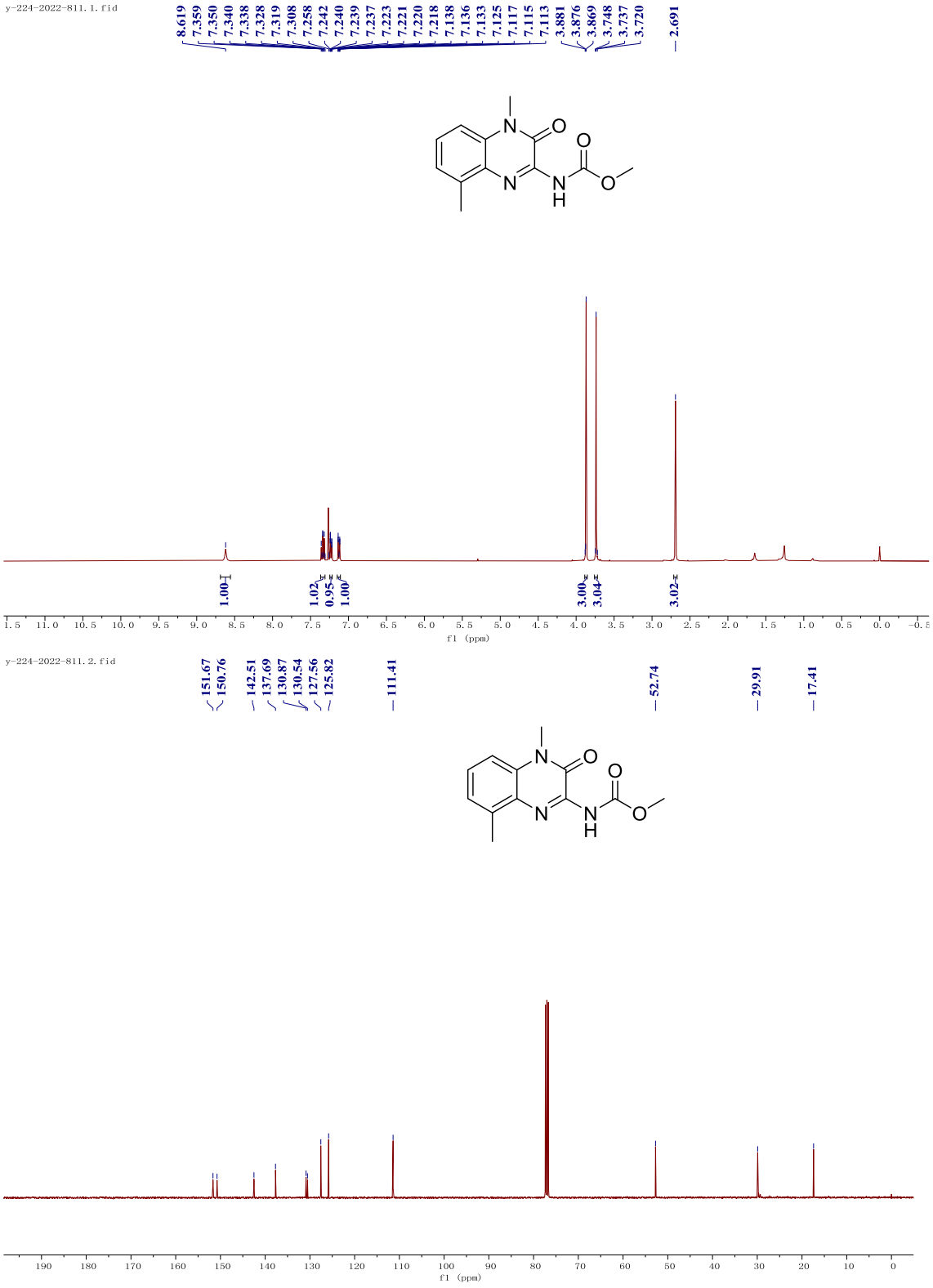


Product **15**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

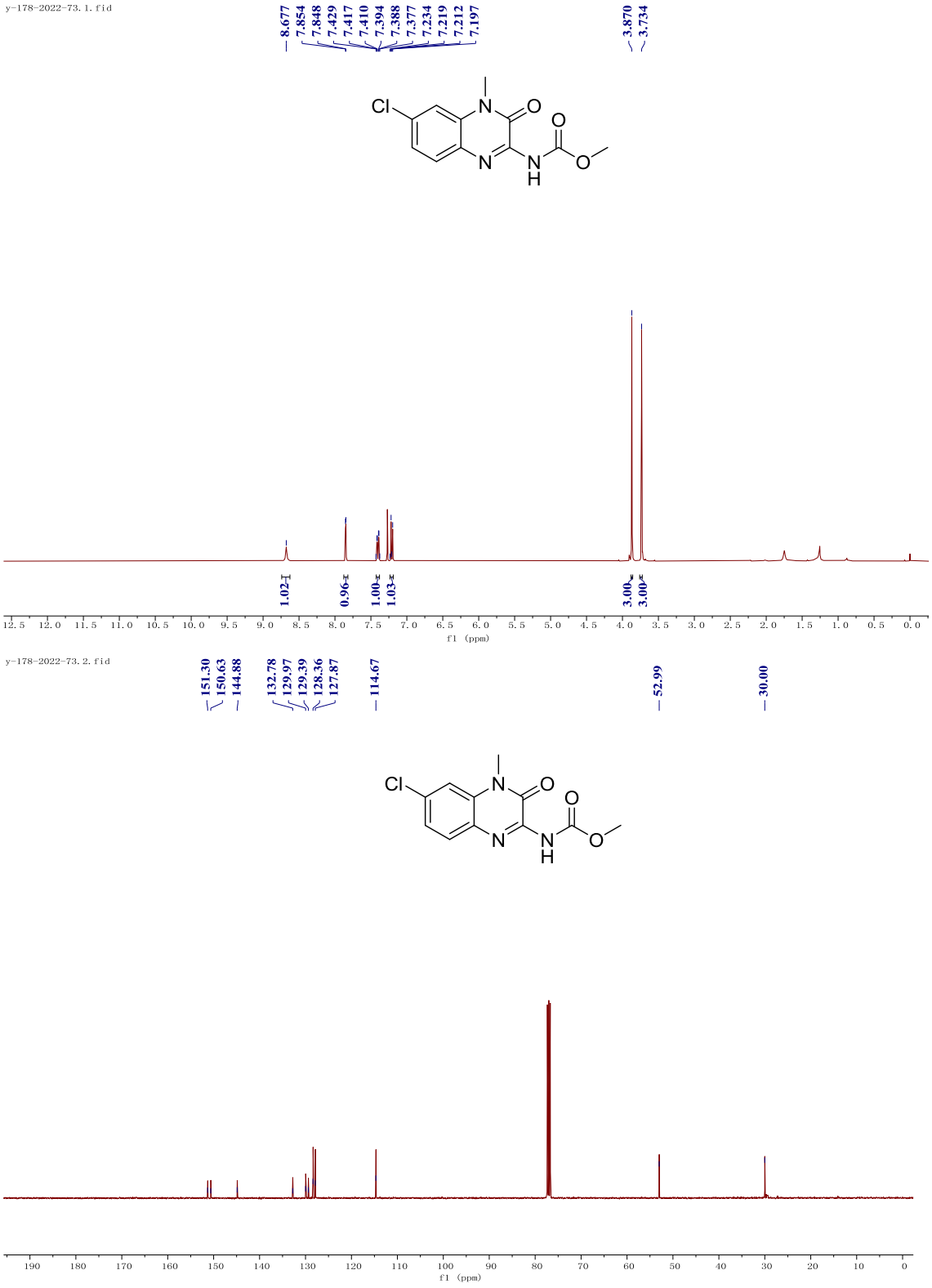




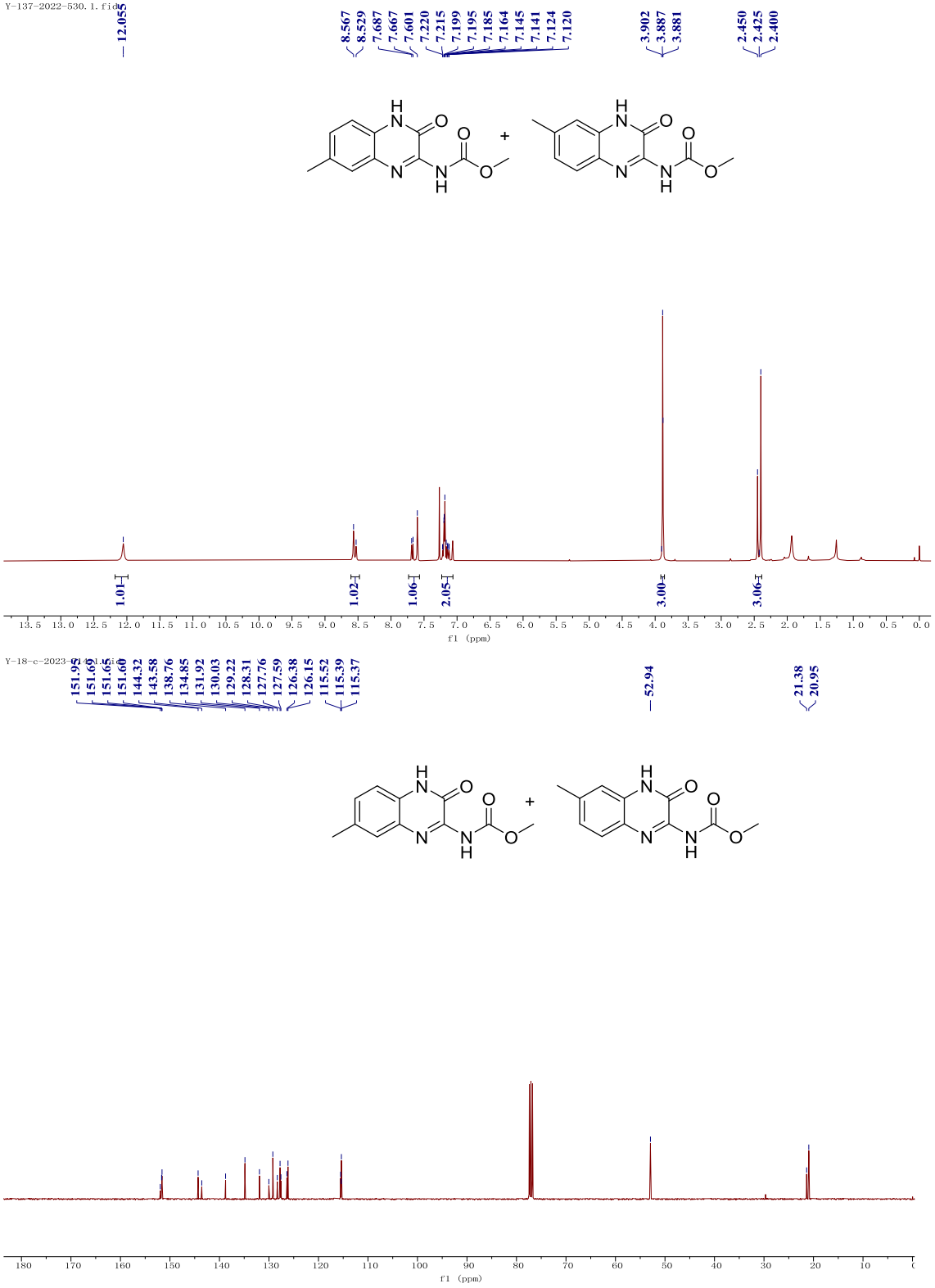
Product **16**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



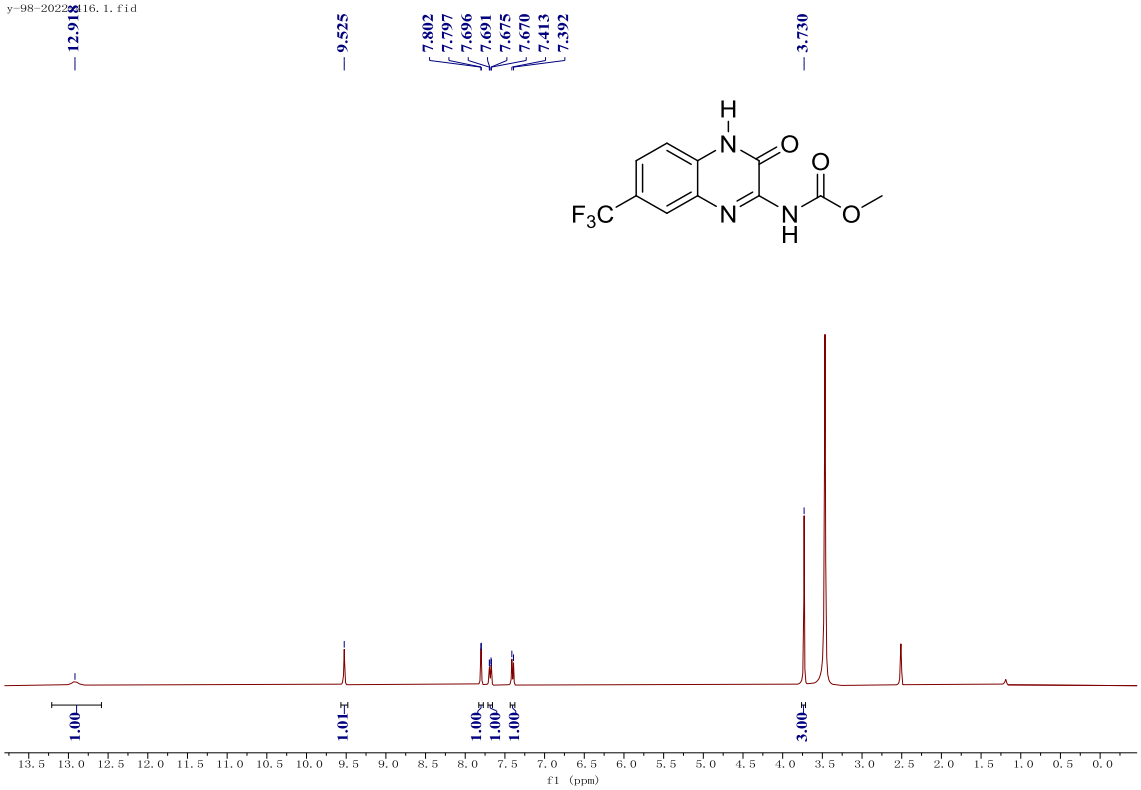
Product **17**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

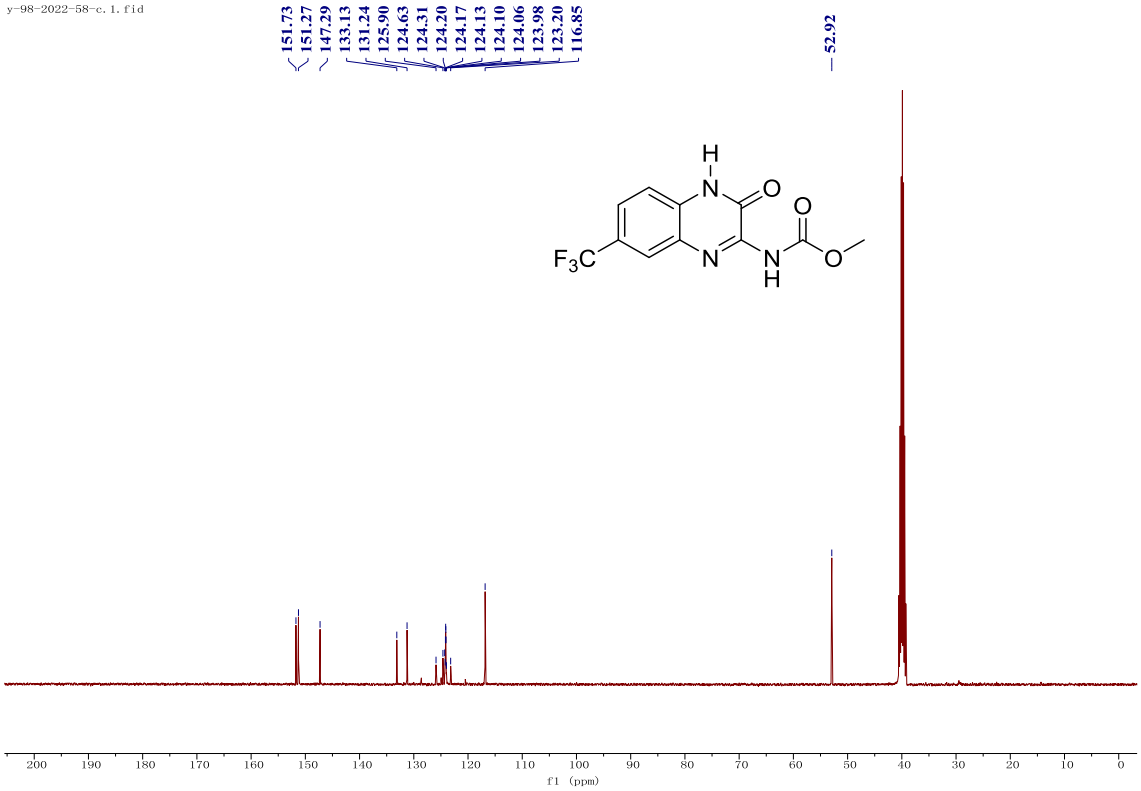


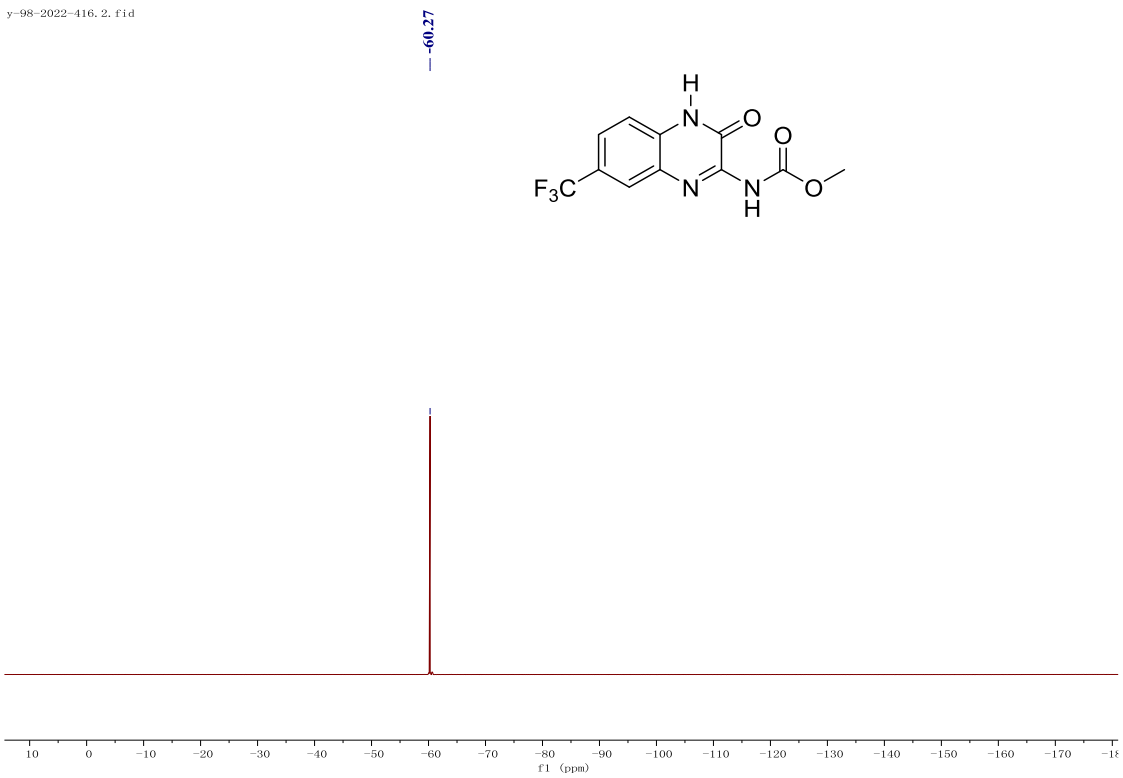
Product **18**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



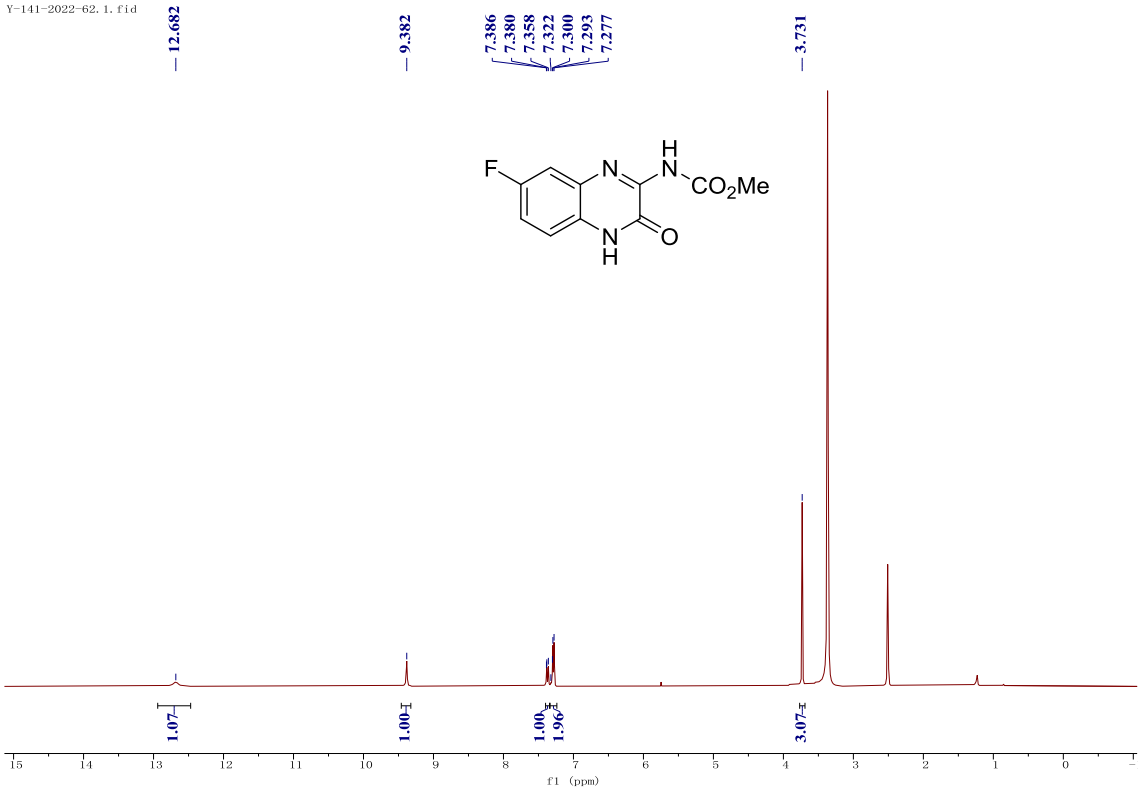
Product **19**: ^{1}H NMR (DMSO-d_6, 400 MHz), ^{13}C{^{1}H} NMR (DMSO-d_6, 100 MHz), ^{19}F NMR (DMSO-d_6, 376 MHz)

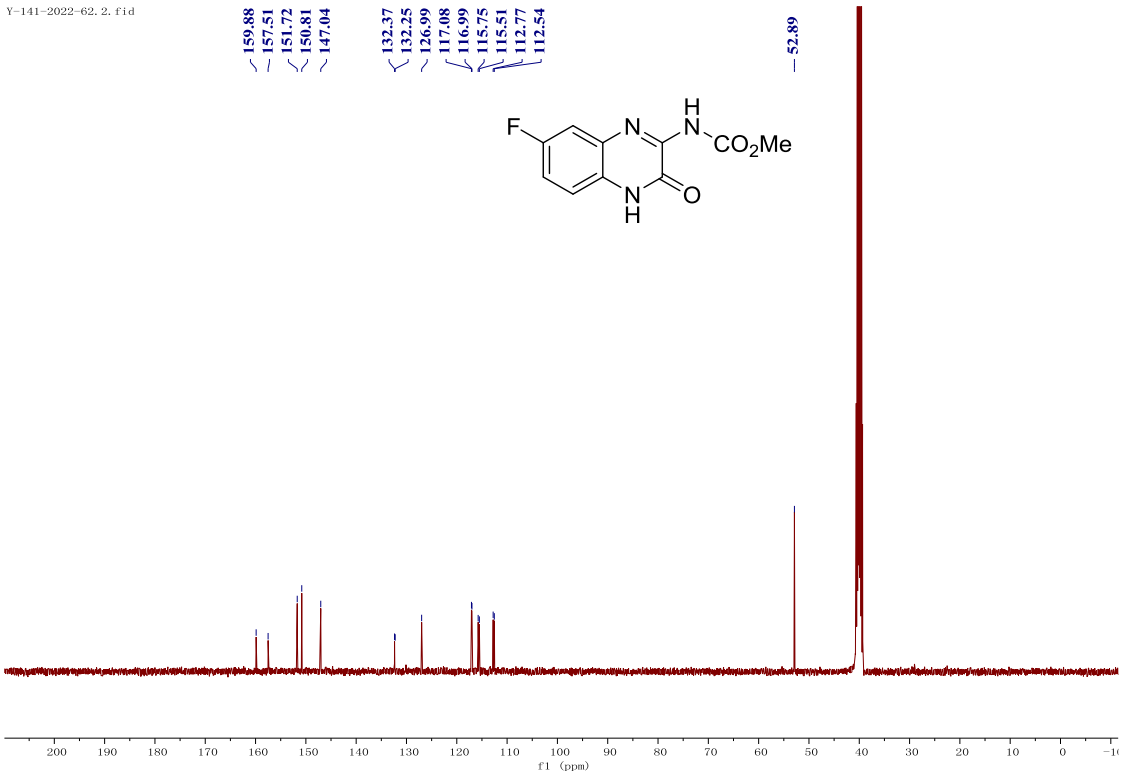






Product **20**: ^{1}H NMR (DMSO-d_6, 400 MHz), ^{13}C{^{1}H} NMR (DMSO-d_6, 100 MHz), ^{19}F NMR (DMSO-d_6, 376 MHz)

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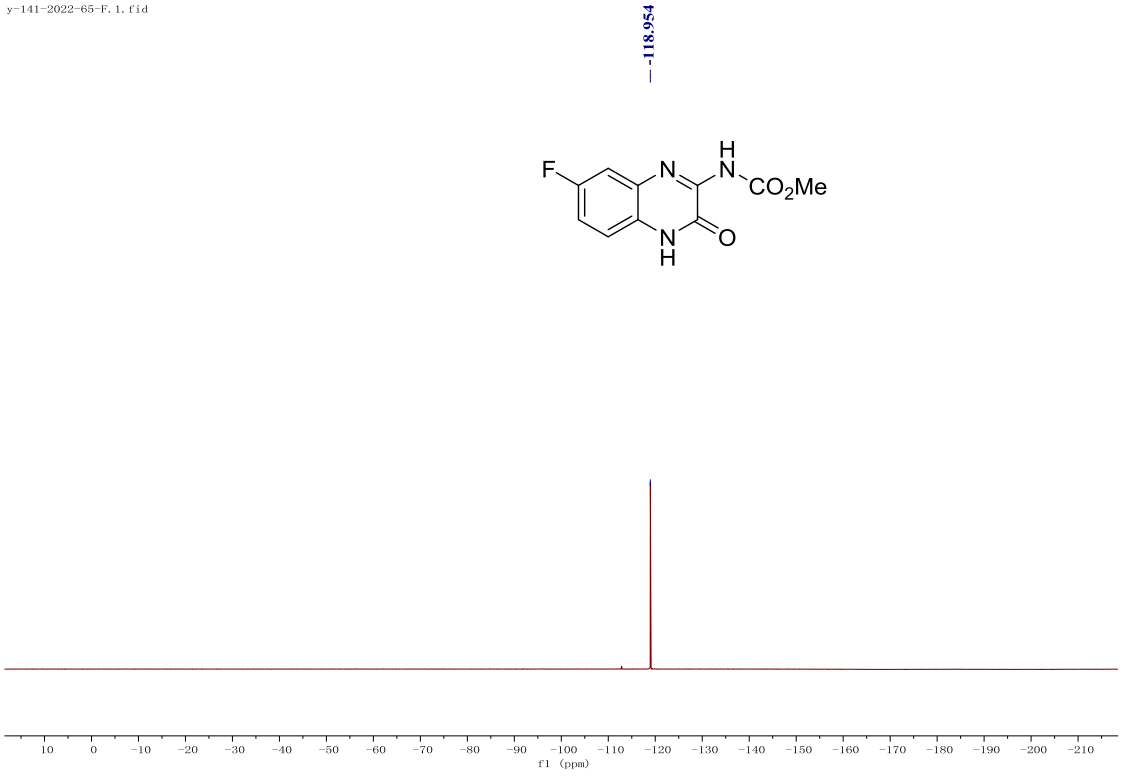
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


y-141-2022-65-F.1.fid
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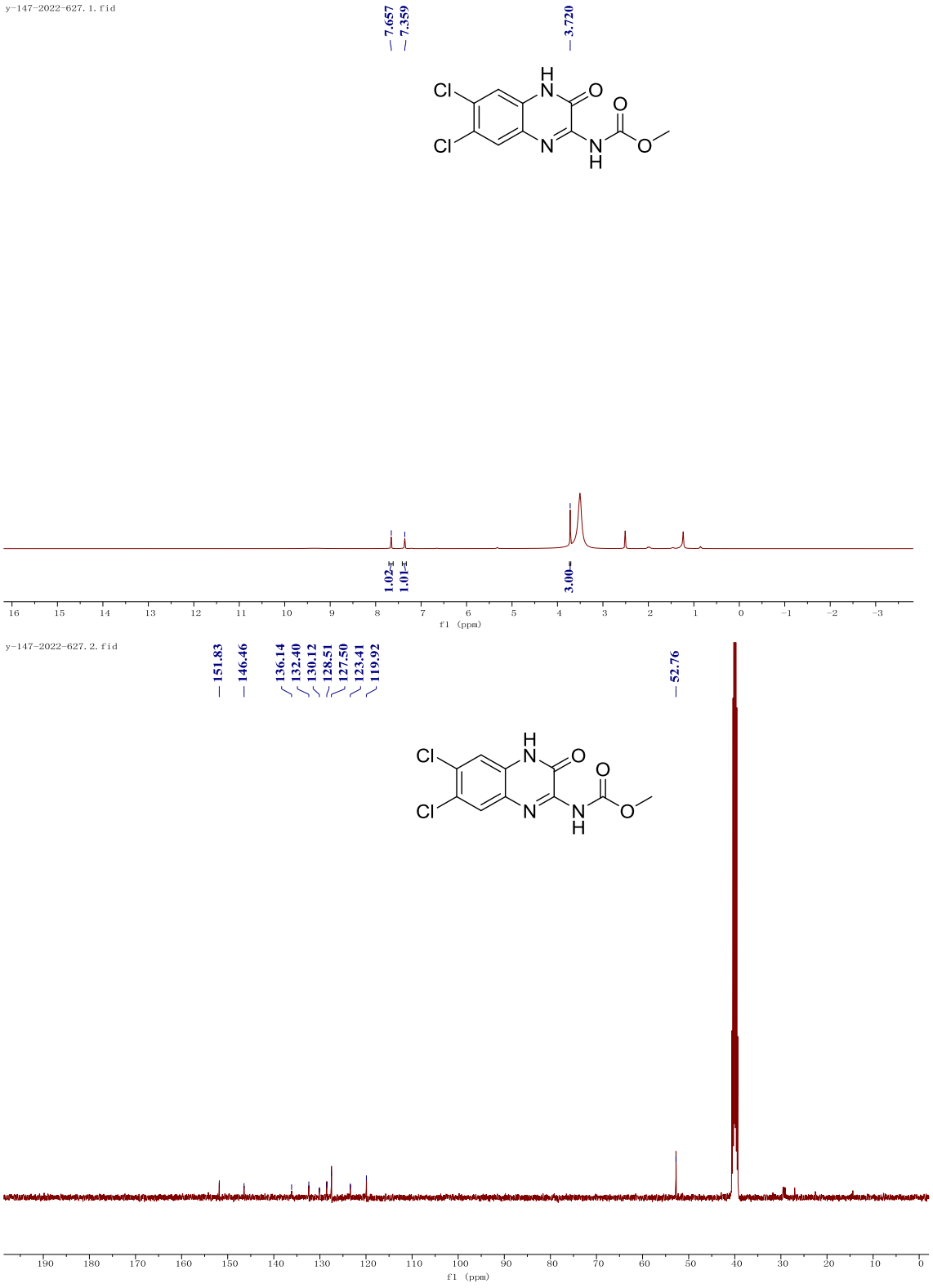
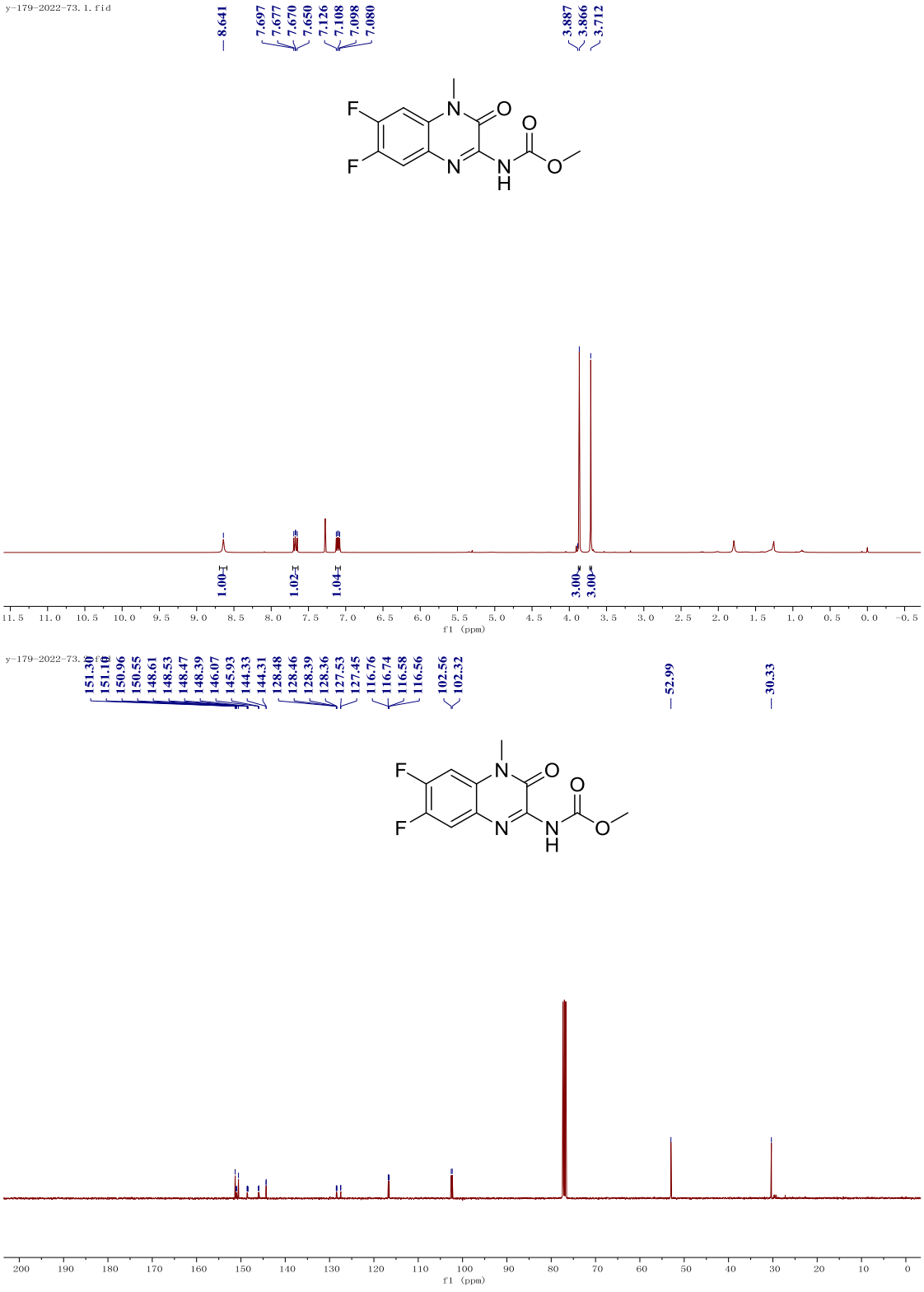
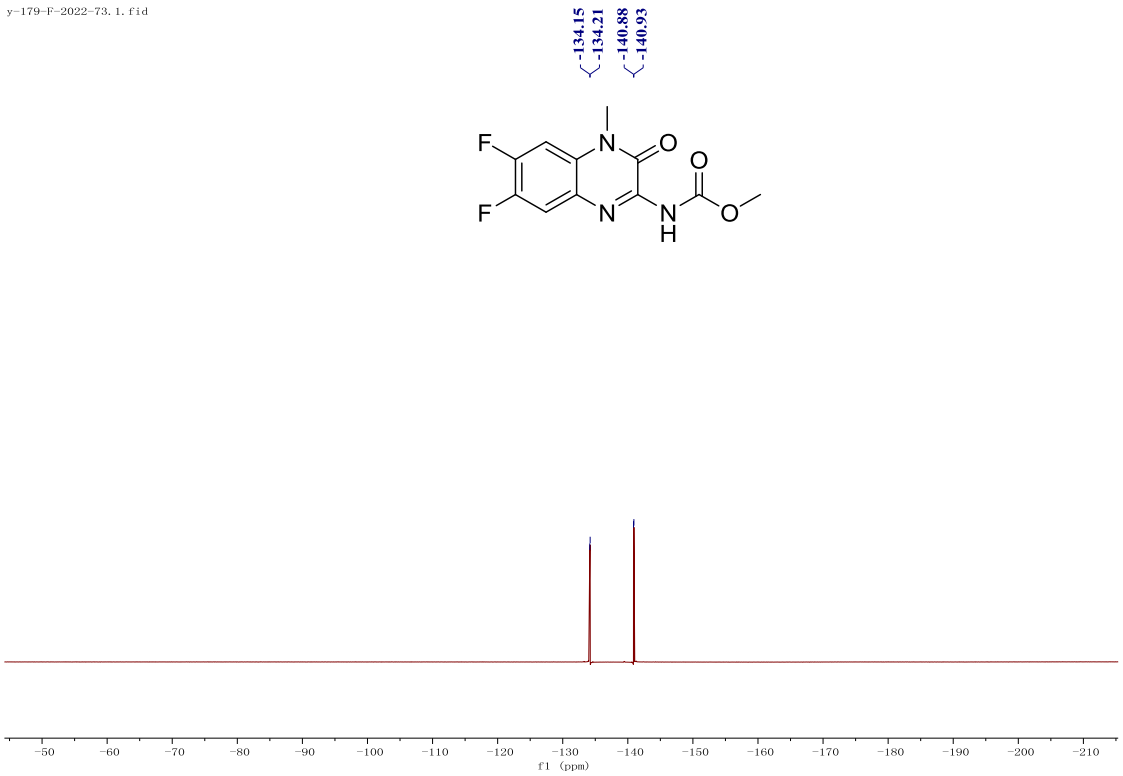
N
H
N
CO2Me
N
H
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

Product **21**: ^{1}H NMR (DMSO-d_6, 400 MHz), ^{13}C{^{1}H} NMR (DMSO-d_6, 100 MHz)

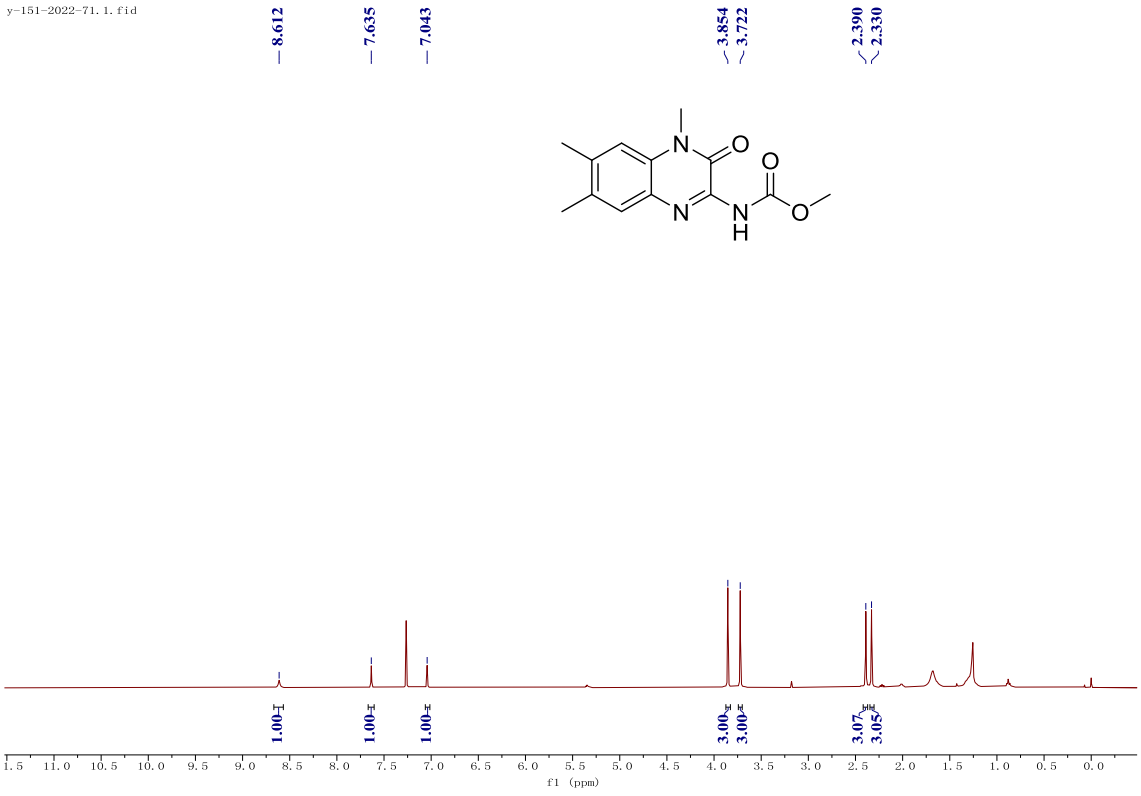


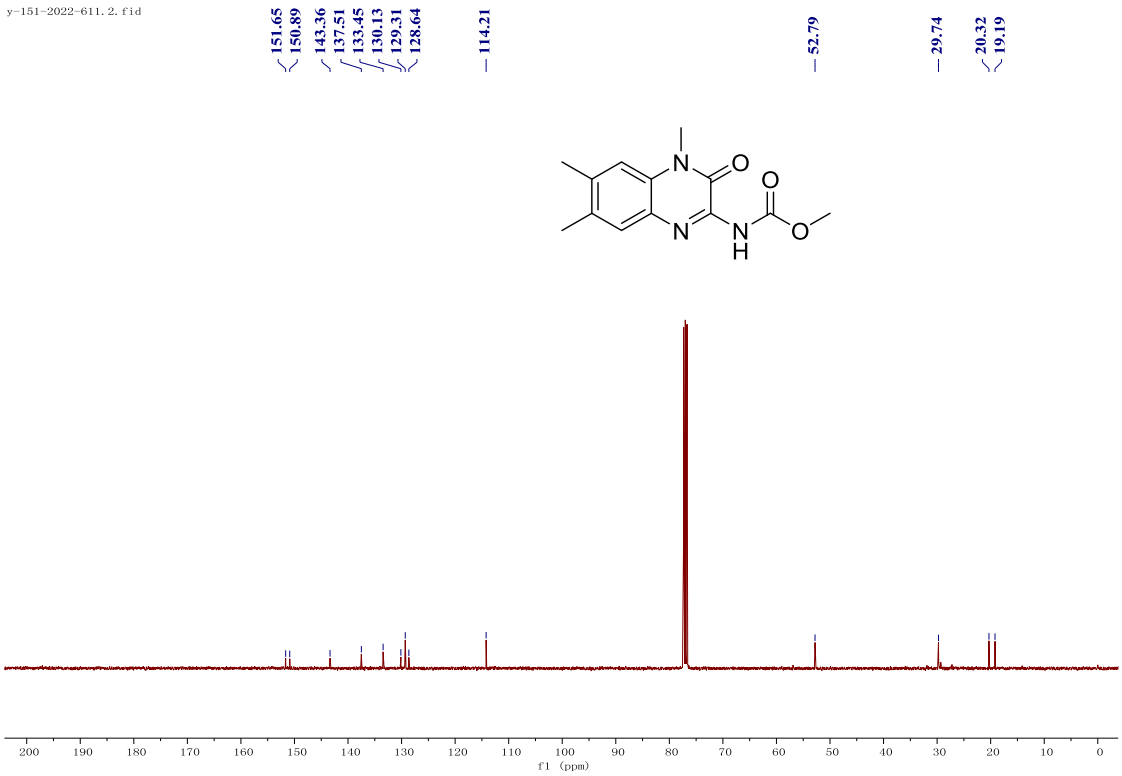
Product **22**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz), ^{19}F NMR ($CDCl_3$, 376 MHz)



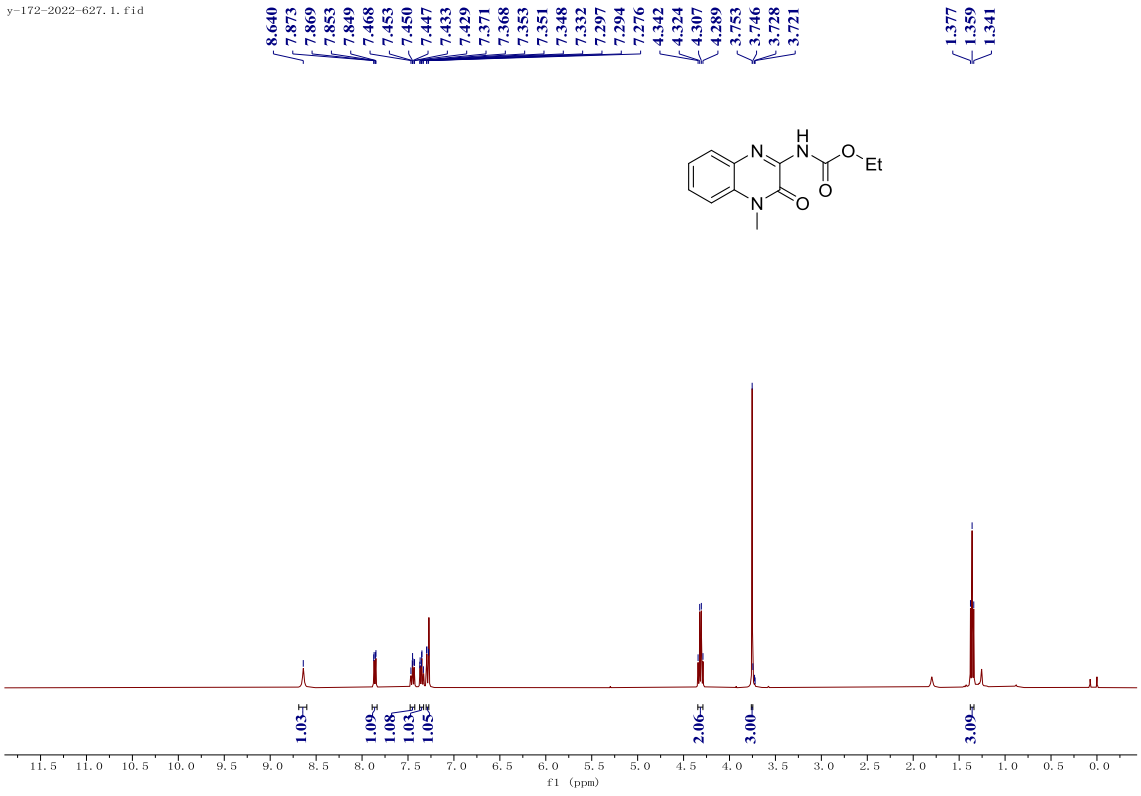


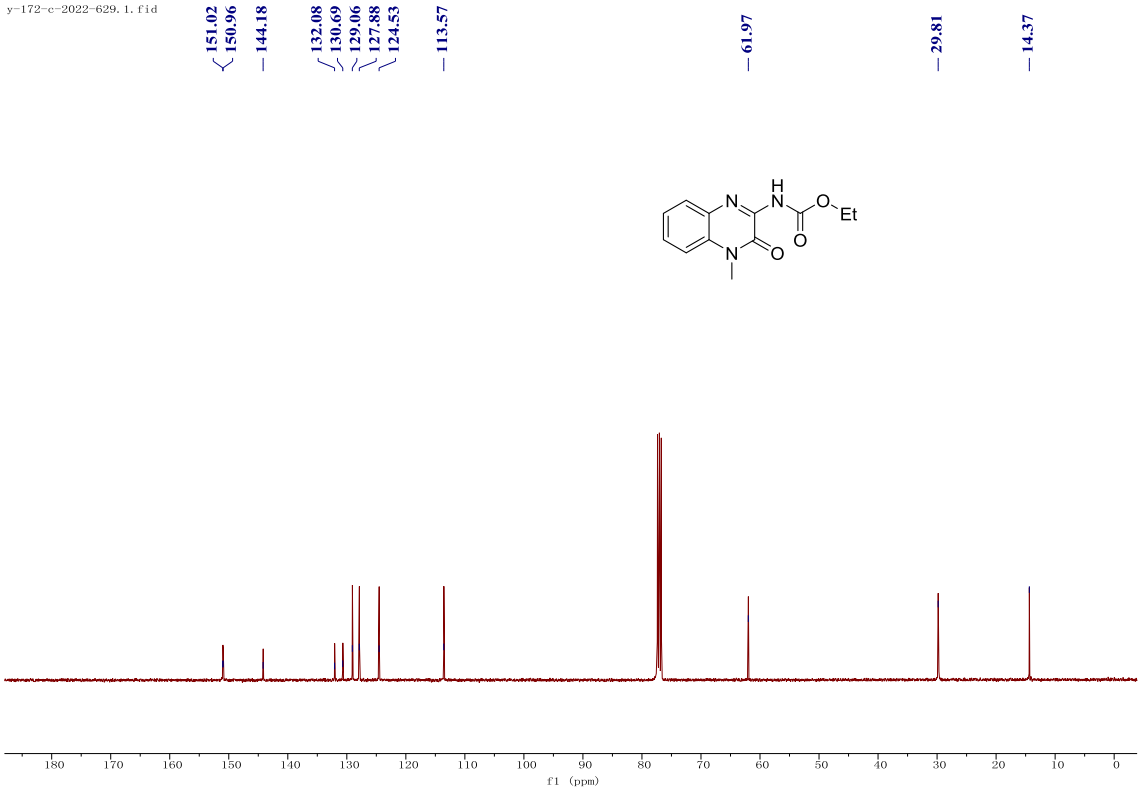
Product **23**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



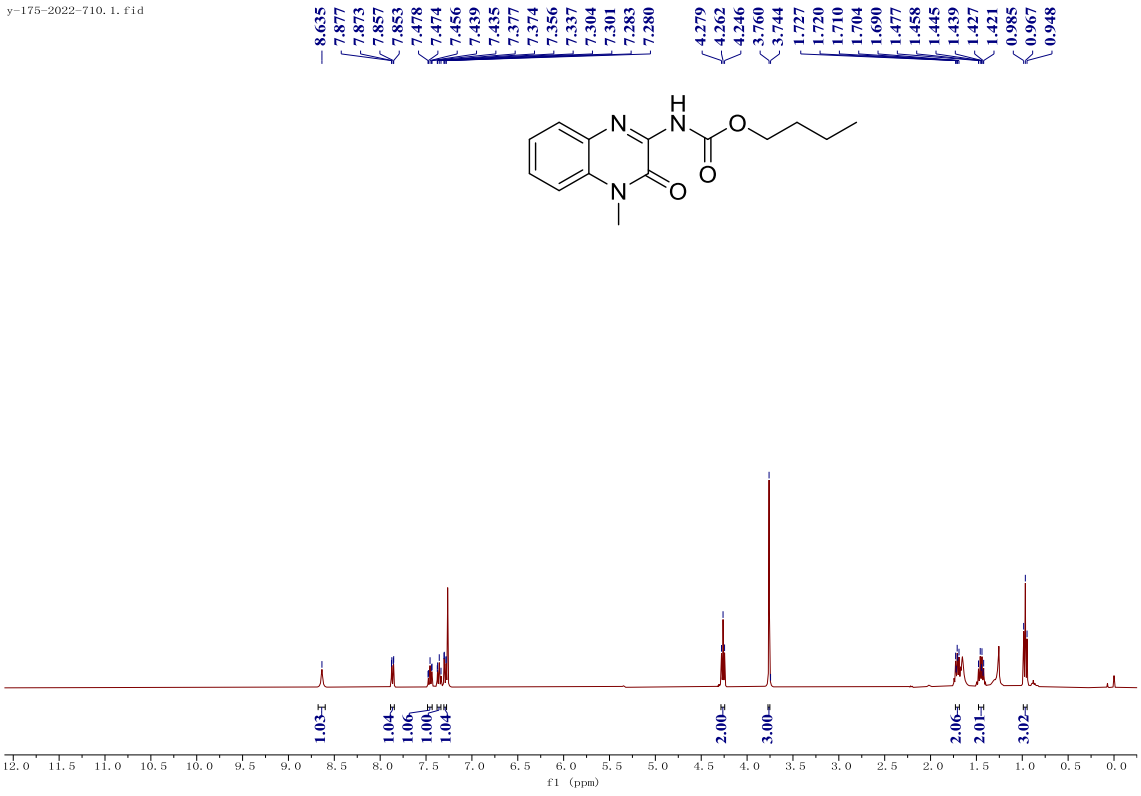


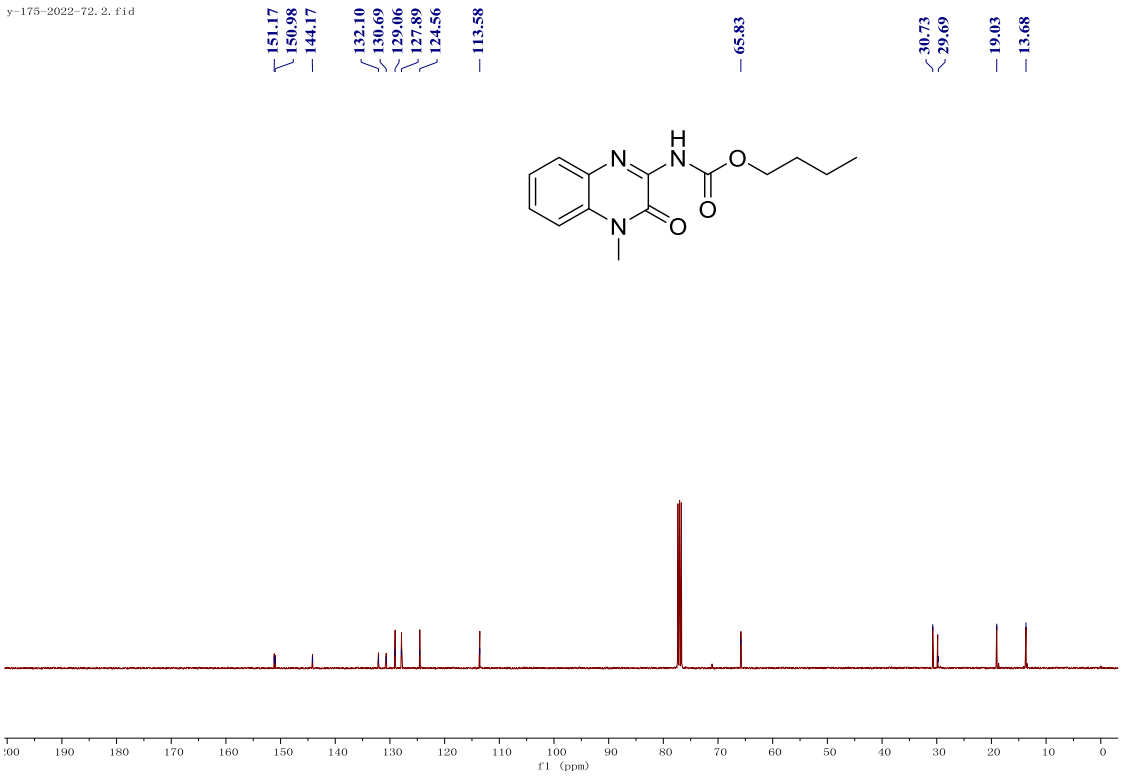
Product **24**: ^{1}H NMR ($CDCl_3$, 400 MHz), ^{13}C{^{1}H} NMR ($CDCl_3$, 100 MHz)



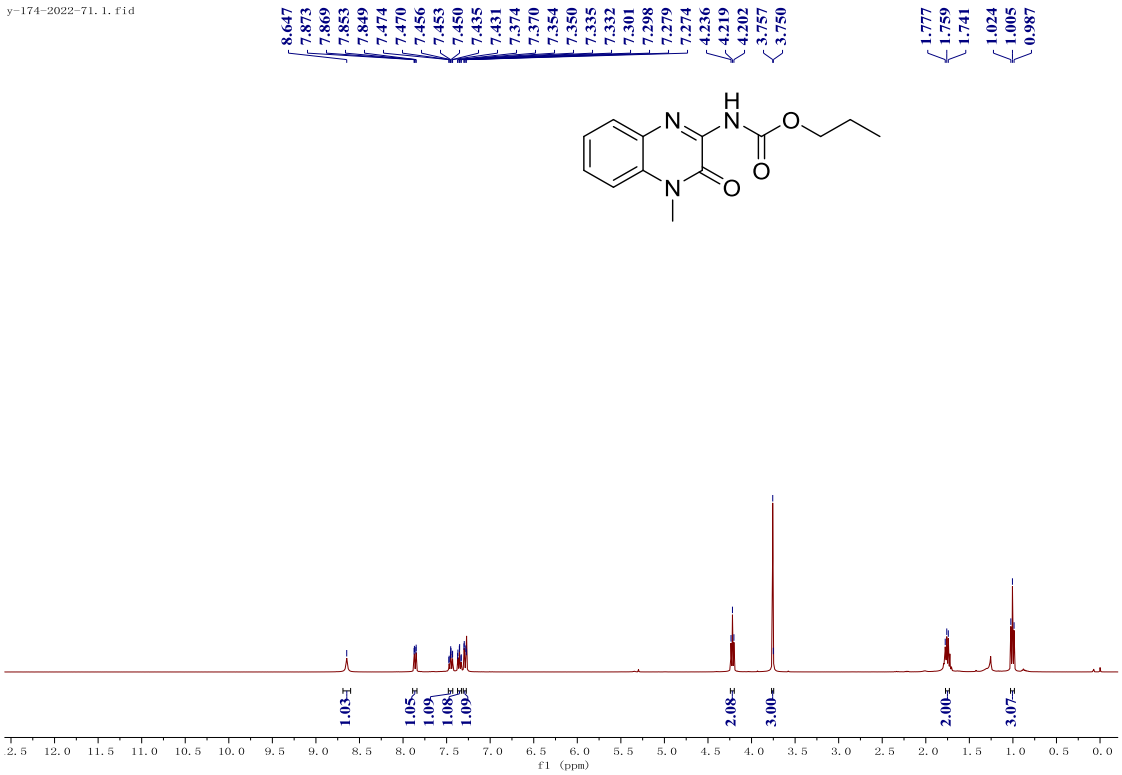


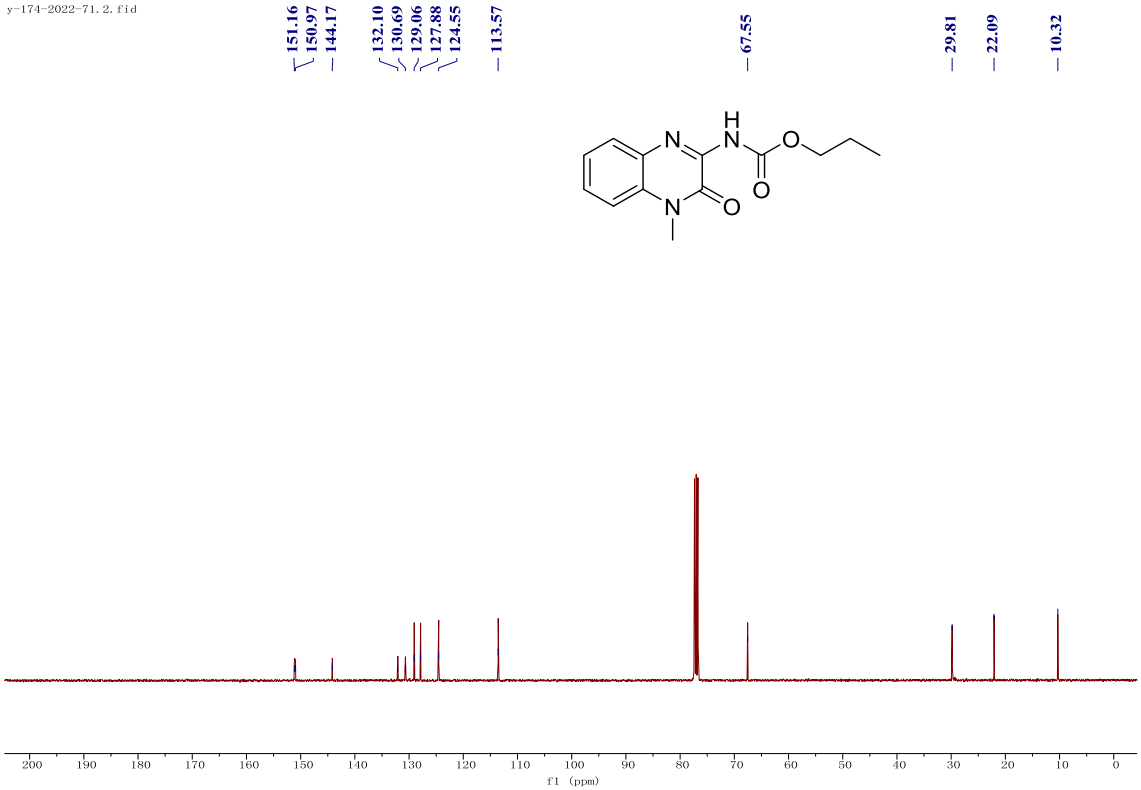
Product **25**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



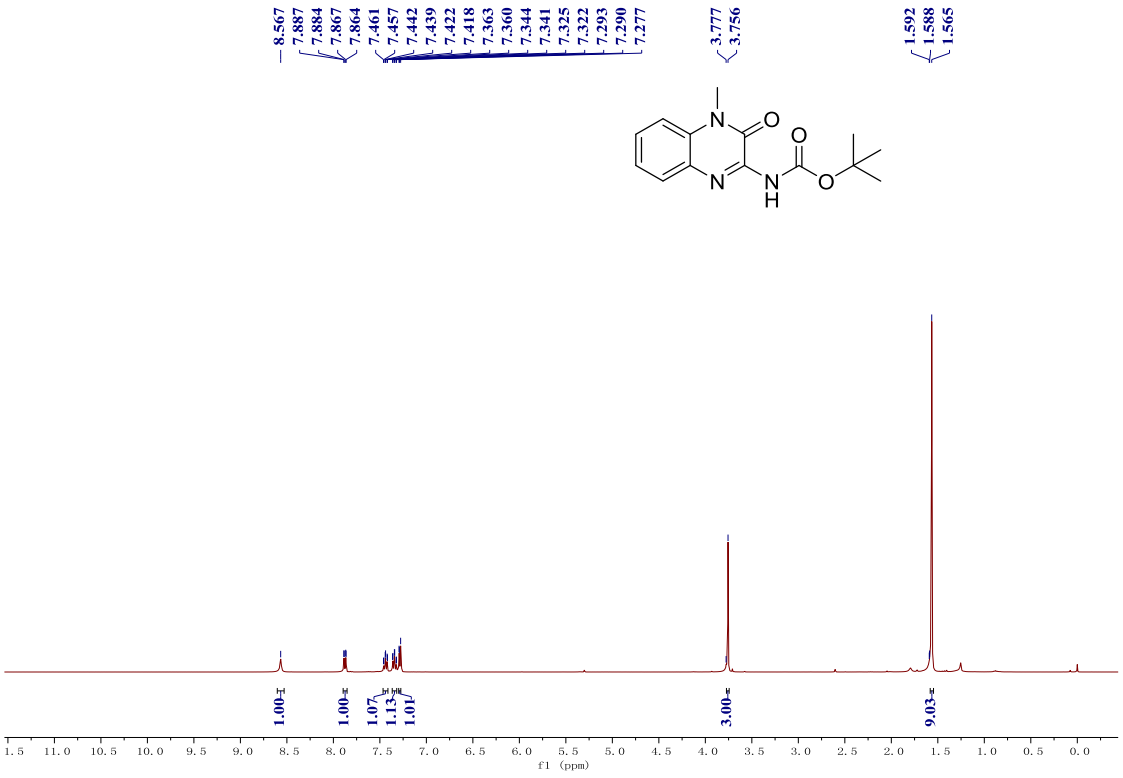


Product **26**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

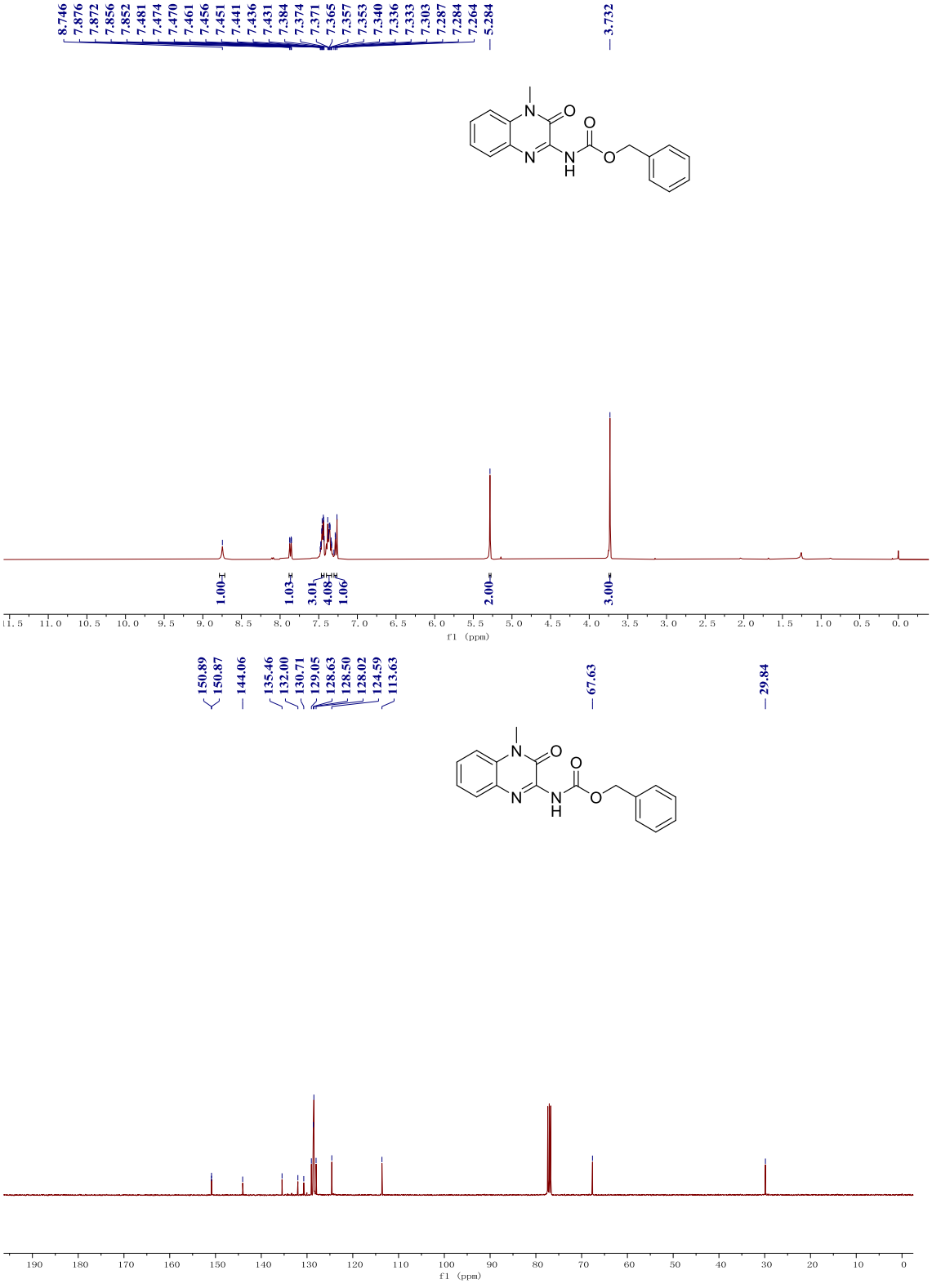




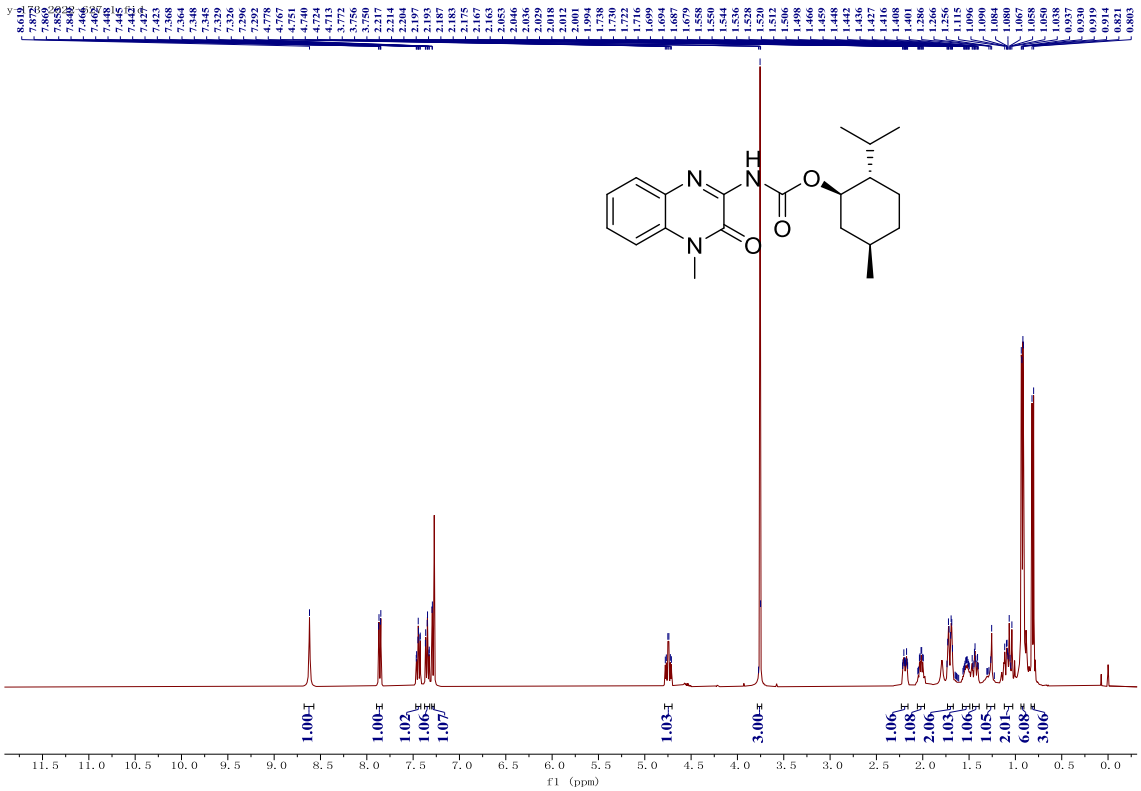
Product **27**: ^{1}H NMR ($CDCl_3$, 400 MHz)

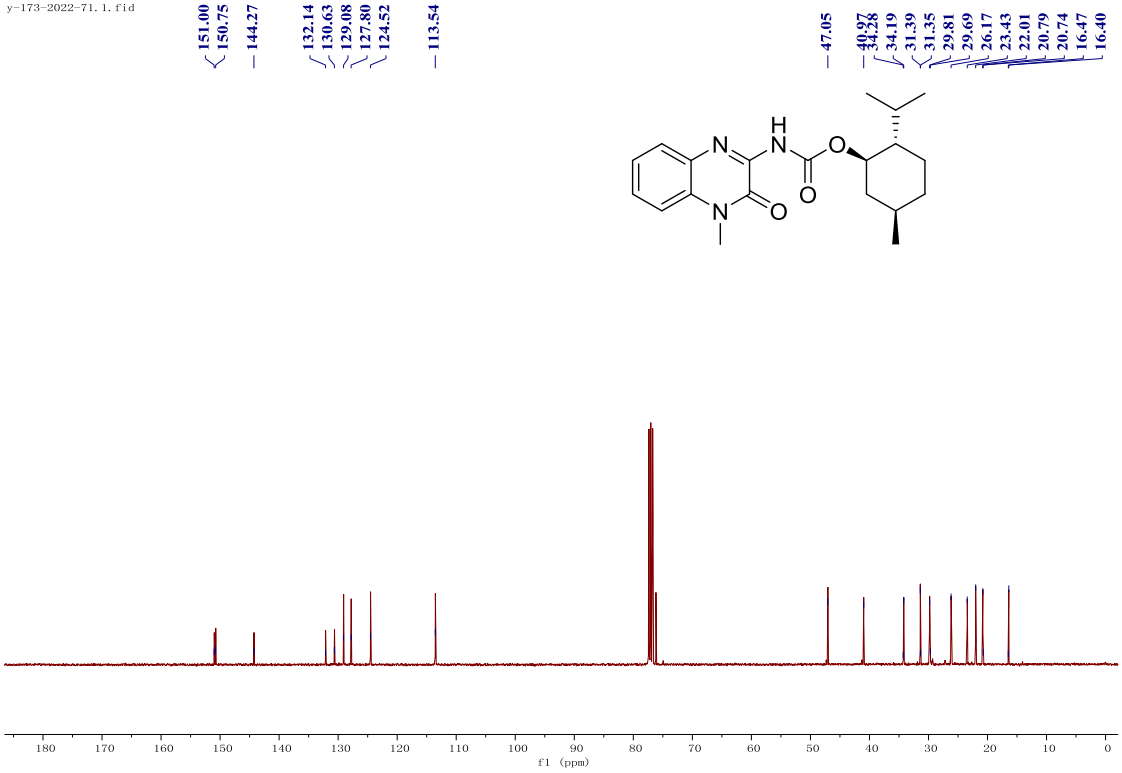


Product **28**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

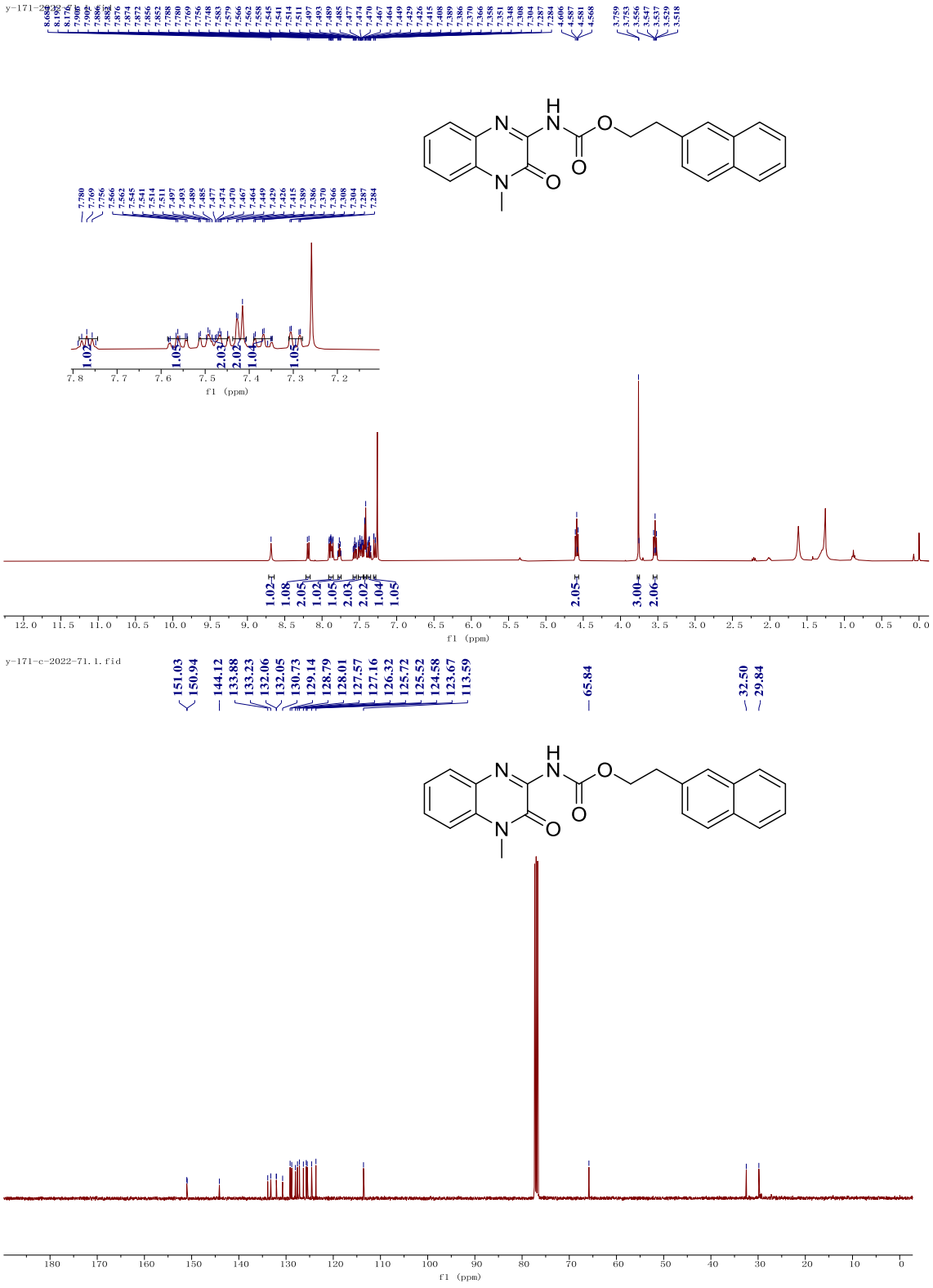


Product **29**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)





Product **30**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)



Product **31**: ^{1}H NMR ($CDCl_3$, 400 MHz), $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 100 MHz)

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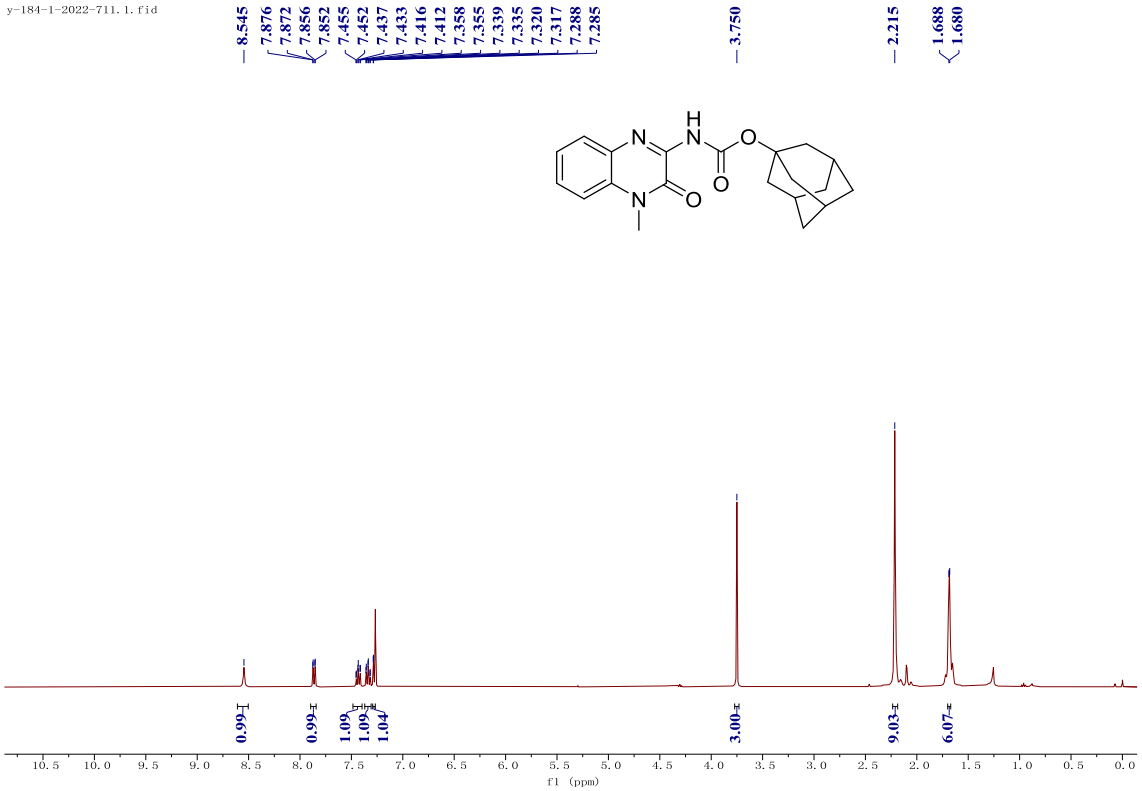

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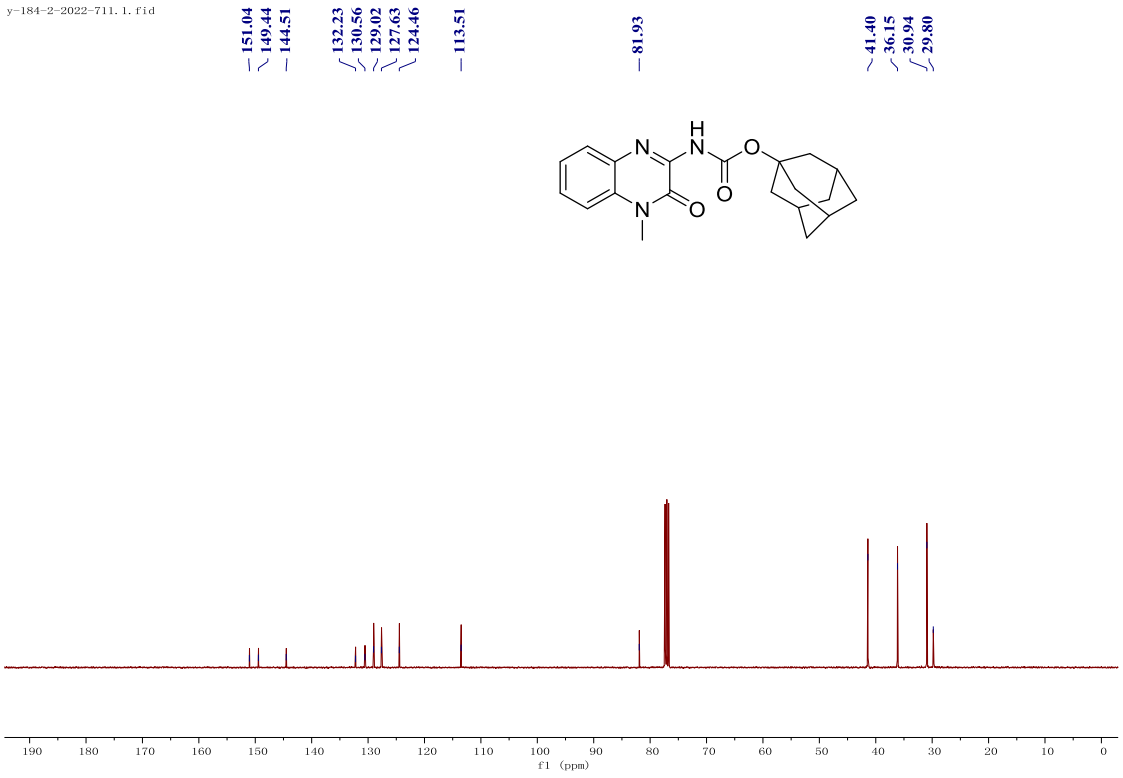

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