Supporting Information

Cross-Dehydrogenative Radical Coupling Enabled by $K_2S_2O_8$: Efficient Synthesis of 2,3-Dicarbonyl Quinolines from Enaminones and Glycine Derivatives

Chunxiao Nong [a], Kun He [a], Yingguo Jiang [a], Fan Zhu [a], Mingquan Yuan*[a], Jingbo Chen*[a], Yi Jin*[a]

[a] *Key Laboratory of Medicinal Chemistry for Natural Resource, Ministry of Education; Yunnan Key Laboratory of Research & Development for Natural Products; School of Pharmacy, Yunnan University, Kunming, 650091, P. R. China.*

Corresponding author. Tel./fax: +86-871-65031633. E-mail:

chenjb@ynu.edu.cn (J. Chen); jinyi@ynu.edu.cn (Y. Jin).

Contents

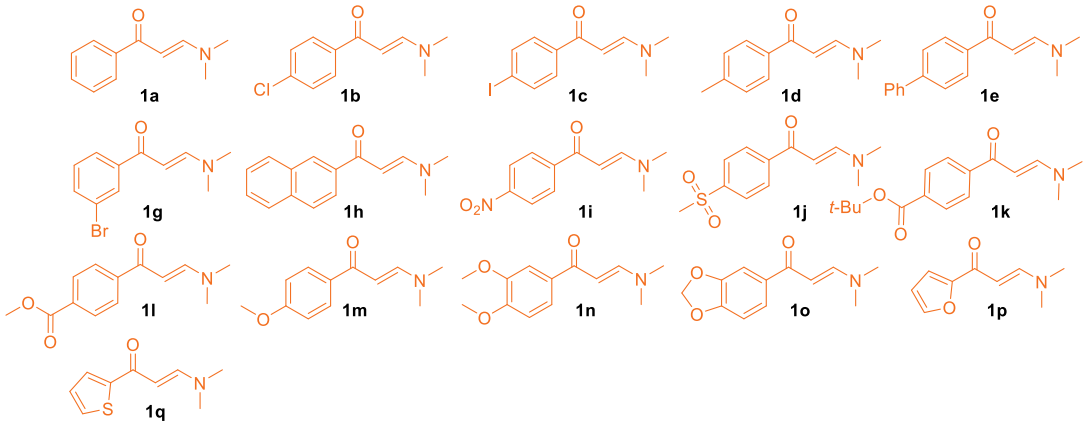
1. General considerations

Dry each component of the laboratory glassware before using them in general experimental procedures. Measure ^{1}H NMR and ^{13}C NMR spectra on a JEOL ECS-400 spectrometer operating at 400 MHz for both ^{1}H and ^{13}C nuclei, utilizing $CDCl_3$ and DMSO-d_6 as solvents for sample preparation. All chemical shifts are reported as δ values (ppm) with tetramethylsilane (TMS) as the internal standard. Peak shapes are designated as follows: s for singlet; d for doublet; t for triplet; m for multiplet; q for quartet. Coupling constants, *J*, are reported in Hertz (Hz). High-resolution mass spectrometry data for the products were acquired using an Agilent 6540 Ultra High Resolution Accurate Mass Q-TOF LC-MS (ESI). Crystallographic data for product **3a** were collected using a Bruker SMART APEX II (Mo Kα radiation, 50 KV voltage, 30 mA current). Chemicals and solvents were purchased from commercial suppliers, including Aldrich (USA) or Shanghai Chemical Reagent Company (China). The products were purified by flash chromatography on 200-300 mesh silica gel (SiO_2).

2. General procedures for the synthesis of substrates and products

2.1 Preparation of substrates 1 (enaminones)

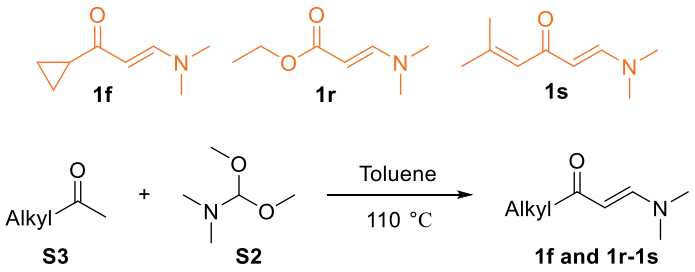
2.1.1 The typical synthetic procedure for aryl enaminones [1]



To a round-bottomed flask equipped with a stirring bar, add ketone **S1** (10.0 mmol,

1.0 equiv.) and 1,1-dimethoxy-N, N-dimethylmethanamine **S2** (20.0 mmol, 2 equiv.), using toluene (20 mL) as the solvent. Stir the mixture in an oil bath at 110 °C overnight. Upon completion of the reaction, extract the reaction mixture with ethyl acetate and dry it over anhydrous sodium sulfate (Na_2SO_4). Subsequently, concentrate the reaction mixture under reduced pressure and purify the residue by column chromatography using a mixture of petroleum ether and ethyl acetate (1:1, V/V) to obtain the corresponding aryl enaminones **1a-1q**.

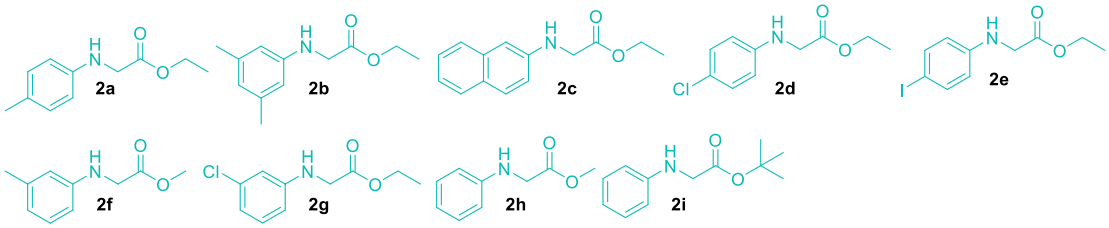
2.1.2 Typical synthesis steps of aralkyl enaminones [2]

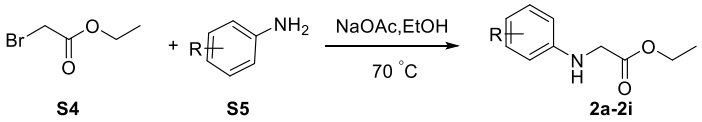


To a round-bottomed flask equipped with a stirring bar, add ketone **S3** (5.0 mmol, 1.0 equiv.) and 1,1-dimethoxy-N, N-dimethylmethanamine **S2** (50.0 mmol, 10.0 equiv.). Stir the mixture in an oil bath at 110 °C overnight. Upon completion of the reaction, remove the solvent under vacuum, and purify the residue by column chromatography using a mixture of petroleum ether and ethyl acetate (1:1, V/V) to obtain the corresponding alkyl enaminones **1f** and **1r-1s**.

2.2 Preparation of glycine derivatives

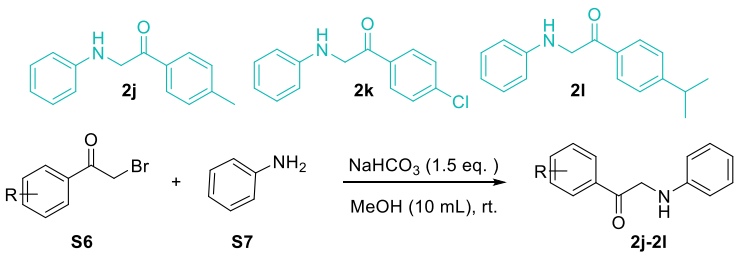
2.2.1 Preparation of *N*-glycine ethyl ester [3]





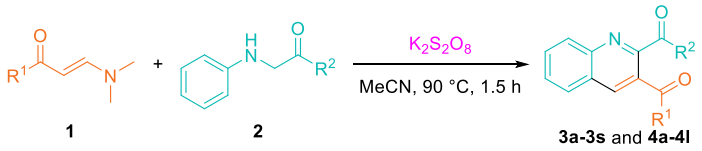
In a 100 mL round-bottomed flask, dissolve the appropriate aniline **S5** (10 mmol, 1.0 equiv.) in anhydrous ethanol (50 mL). Subsequently, add ethyl bromoacetate **S4** (10 mmol, 1.0 equiv.) dropwise. Heat the mixture to 70 °C and reflux for 10 hours. Monitor the reaction progress by TLC. Upon completion of the reaction, extract the mixture with ethyl acetate and filter the precipitate. Concentrate the filtrate under vacuum to obtain the product **2a-2i**.

2.2.2 Preparation of α-Aminoacetophenone [4]



Dissolve a mixture of aniline **S7** (1 mmol, 1.0 equiv.) and 2-bromoacetophenone compounds **S6** (1.2 mmol, 1.2 equiv.) in 10 mL of methanol. Then, add sodium bicarbonate ($NaHCO_3$, 1.5 mmol) to the solution and stir at room temperature. Upon completion of the reaction (monitored by thin-layer chromatography (TLC)), extract the mixture with ethyl acetate (15 mL×2). Wash the organic layer with water (15 mL×2) and finally dry it over anhydrous sodium sulfate (Na_2SO_4). Concentrate the organic layer using a rotary evaporator, and purify the crude product by column chromatography on silica gel (200–300 mesh). The yellow solid product, 1-phenyl-2-(phenylamino)acetone **2j-2l**.

2.3 General preparation methods for compounds 3a-3s and 4a-4l



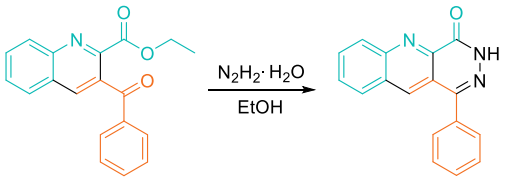
Enaminone derivatives **1** (0.2 mmol, 1.0 equiv.), glycine derivatives **2** (0.2 mmol,

1.0 equiv.), and potassium persulfate ($K_2S_2O_8$, 0.6 mmol, 3.0 equiv.) in MeCN (2.0 mL, 0.1 M) were added to 10.0 mL reaction tube. The mixture was stirred in an oil bath maintained at 90 °C for 1.5 hours, and monitored by TLC. Then the reaction system was cooled to room temperature and quenched with saturated sodium chloride solution (10 mL) and extracted with 20.0 mL EtOAc for three times. The organic layers were combined, dried over Na_2SO_4, filtered and evaporated under reduced pressure. The residues were purified by flash column chromatography on silica gel to provide the products **3a-3s** and **4a-4l**. The products were further identified by NMR spectroscopy and HRMS.

2.4 A gram-scale method for the preparation of 2,3-dicarbonylquinoline 3a.

Under an air atmosphere, enaminone **1a** (10 mmol), N-ethyl glycine **2a** (10 mmol, 1.0 equiv.), $K_2S_2O_8$(3 equiv.) and acetonitrile (15 mL) were added to a 25 mL round-bottom flask. The mixture was stirred at 90 °C in an oil bath for 48 hours. After cooling to room temperature, the mixture was extracted three times with a combination of saturated sodium chloride solution and 200 mL of ethyl acetate. The organic layers were combined, dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography to afford the product **3a** in 76% yield (2.3 g).

2.5 General preparation methods for compounds 5

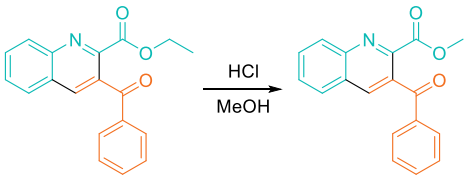


3a **5**, 62%

A 25 mL round-bottom flask was charged with 2,3-dicarbonyl quinoline **3a** (0.5 mmol), $N_2H_4 \cdot H_2O$ (2 equiv.), and EtOH (2 mL). The mixture was refluxed in an oil bath at 70 °C for 12 h. After cooling to room temperature, the mixture was extracted three times with a combination of saturated sodium chloride solution and 200 mL of ethyl acetate. The organic layers were combined, dried over anhydrous sodium sulfate,

filtered, and concentrated under reduced pressure to afford the product **5** with a yield of 62%.

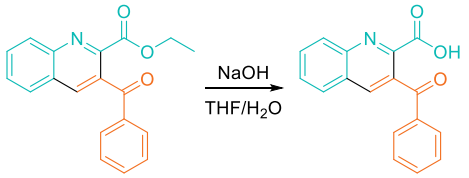
2.6 General preparation methods for compounds 4h



3a **4h**, 58%

A 25 mL round-bottom flask was charged with 2,3-dicarbonyl quinoline **3a** (0.5 mmol), HCl (1 equiv.), and MeOH (2 mL). The mixture was then reacted in an oil bath at 70 °C for 12 h. After cooling to room temperature, the mixture was extracted three times with a combination of saturated sodium chloride solution and 200 mL of ethyl acetate. The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to afford product **4h** with a yield of 58%.

2.7 General preparation methods for compounds 6



3a **6**, 45%

A 25 mL round-bottom flask was charged with 2,3-dicarbonyl quinoline **3a** (0.5 mmol), NaOH (2 equiv.), and a mixture of THF/H_2O (1:1, 1 mL each). The mixture was allowed to react in an oil bath at 50 °C for 4 h. After cooling to room temperature, the pH of the mixture was adjusted to 2 or 3 using dilute hydrochloric acid. The mixture was then quenched with saturated sodium chloride solution and extracted three times with 200 mL of ethyl acetate. The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure to afford product **6** with a yield of 45%.

2.8 To verify the Povarov reaction

To verify the possibility of a Povarov reaction occurring in our standard reaction system, we conducted a series of meticulous control experiments. Initially, we replaced compound **2a** with imine **10** (0.2 mmol) and allowed it to react with **1a** (0.2 mmol) under standard reaction conditions. Notably, no formation of product **3a** was observed in this reaction. Subsequently, we maintained all other reaction conditions constant and carried out three additional sets of experiments: 1、In the first set of experiments, we substituted $K_2S_2O_8$ (3 eq.) with $Yb(OTf)_3$ (3 eq.). No product **3a** was detected. 2、In the second experiment, we replaced $K_2S_2O_8$ (3 eq.) by sequentially adding CF_3SO_3H (1.5 eq.) in 1 mL of acetonitrile and $Mn(OAc)_3$ (1.5 eq.) in 1 mL of acetic acid to the reaction system. Again, the expected product **3a** was not observed. 3、In the final set of experiments, we exchanged $K_2S_2O_8$ (3 eq.) for a sequential addition of $BF_3{\cdot}OEt_2$ (1.5 eq.) and *p*-TsOH (1.5 eq.) to the reaction system. Despite these modifications, the reaction still did not yield any detectable amounts of **3a**.

3. X-ray diffraction structure and data of 3a

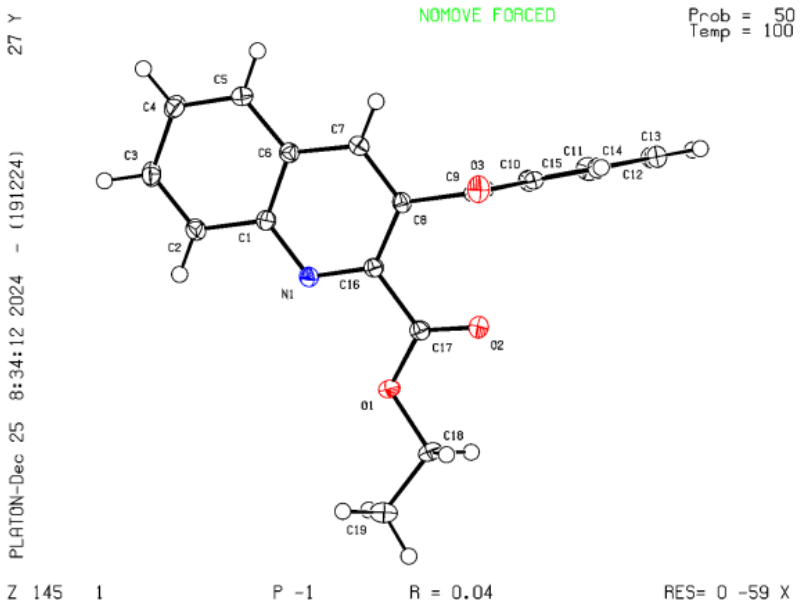


Figure S1. X-Ray crystal structure of **3a**, ellipsoid was drawn at the 50% probability level.

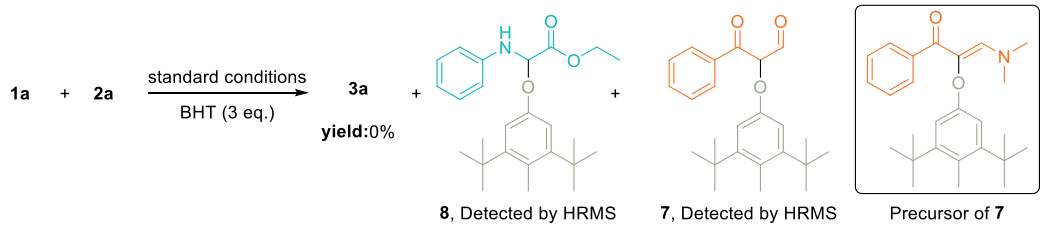
Table S1 Crystal data and structure refinement for **3a**

```
Bond precision:   C-C = 0.0018 A          Wavelength=0.71073

Cell:           a=9.0995(8)       b=9.5130(9)       c=9.7237(9)
                alpha=77.734(3)   beta=66.596(3)    gamma=77.558(3)
Temperature:    100 K

                Calculated                Reported
Volume          746.73(12)                746.73(12)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C19 H15 N O3              C19 H15 N O3
Sum formula     C19 H15 N O3              C19 H15 N O3
Mr              305.32                    305.32
Dx,g cm-3       1.358                     1.358
Z               2                         2
Mu (mm-1)       0.092                     0.092
F000            320.0                     320.0
F000'           320.15
h,k,lmax        12,12,12                  12,12,12
Nref            3712                      3701
Tmin,Tmax       0.978,0.984               0.717,0.746
Tmin'           0.978

Correction method= # Reported T Limits: Tmin=0.717 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 28.321

R(reflections)= 0.0406( 3242)                wR2(reflections)=
                                             0.1056( 3701)
S = 1.090               Npar= 209
```

Compound **3a** (20 mg) was added to a 5 mL sample vial, followed by the addition of dichloromethane (1 mL) and *n*-hexane (2.5 mL). The vial was then sealed with a septum and 16 small holes were punched in the septum. The sample vial was placed in a safe location to allow for evaporation and the isolation of single crystals. The single crystals were removed and sent for single crystal diffraction testing to obtain relevant data. Instrumentation: A BRUKER SMART APEX II CCD detector was used in conjunction with graphite-monochromatized Mo Kα radiation (k = 0.071073 nm) to collect single crystal intensity data for each complex. The structure was solved using the SHELXS-97 program by direct methods and subsequent Fourier difference techniques, and refined anisotropically by full-matrix least-squares on F2 using the SHELXL-97 program.

4 Mechanism studies

4.1 Radical Trapping Experiment



Enaminone **1a** (0.2 mmol, 1.0 equiv.), glycine ester **2a** (0.2 mmol, 1.0 equiv.), and potassium persulfate ($K_2S_2O_8$, 0.6 mmol, 3.0 equiv.) in MeCN (2.0 mL, 0.1 M) were added to 10.0 mL reaction tube. The mixture was stirred in an oil bath maintained at 90 °C, and monitored by TLC. After stirring for 1.5 h and directly detected by HRMS.

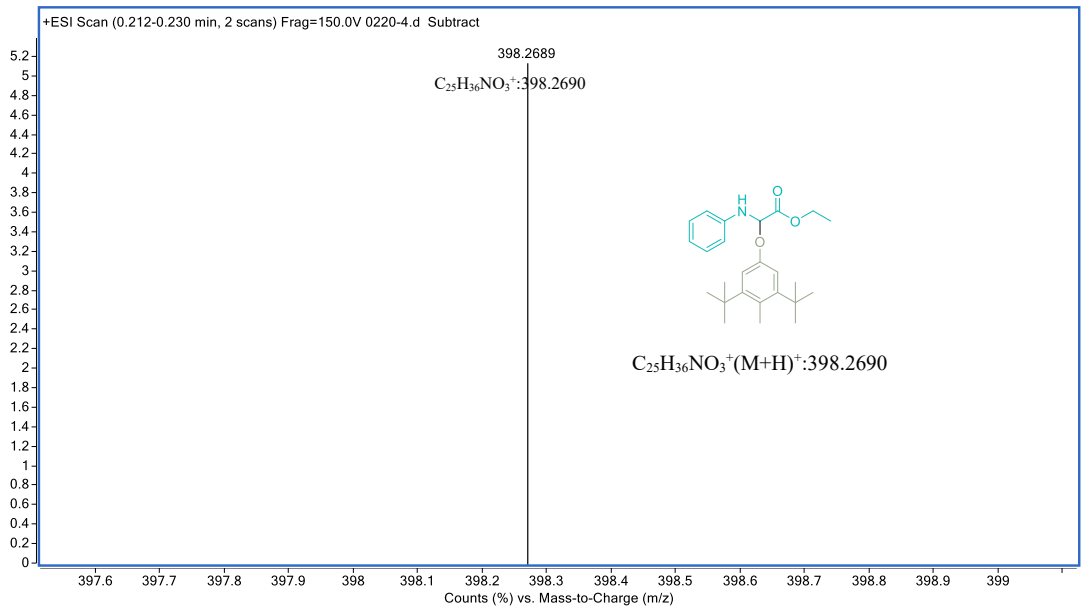


Figure S2. HRMS spectrum of **8**

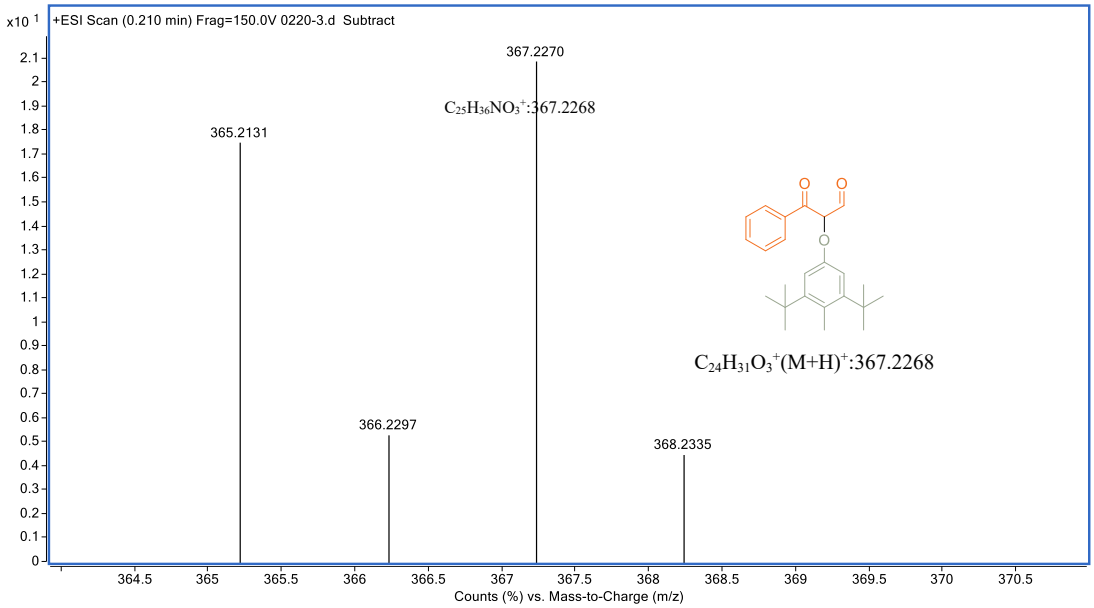


Figure S3. HRMS spectrum of **7**

4.2 Time progression experiment

Add enaminone **1a** (0.0355 g, 0.2 mmol, 1.0 equiv.), ethyl N-glycinate **2a** (0.0338 g, 0.2 mmol, 1.0 equiv.), $K_2S_2O_8$ (0.1623 g, 3.0 equiv.), and dry MeCN (2 mL) in a 10 mL reaction tube that had been dried in a high-temperature oven. Subsequently, heat the reaction mixture in an oil bath at 90 °C. Concurrently, conduct 11 identical reactions, using dibromomethane as an internal standard to determine the yield via ^{1}H NMR of the crude mixture. Plot the reaction times at 5 min, 10 min, 15 min, 20 min, 30 min, 40 min, 50 min, 60 min, 70 min, 80 min, and 90 min to illustrate the increase in product **3a** over time, the decrease in the two substrates **1a** and **2a** over time, and the changes in intermediate **Int-4** over time. (**Figure S4**）

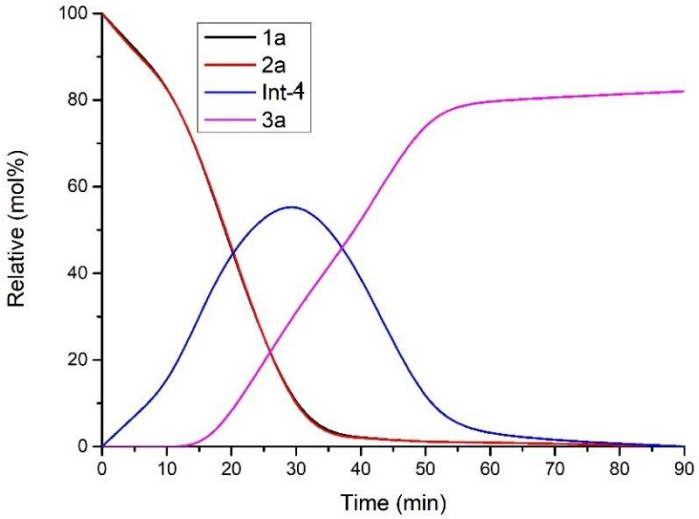


Figure S4. Time course experiments

4.3 Effect of N substituents on reaction rate

Enaminones (0.2 mmol, 1.0 equiv.), ethyl glycinate (0.0338 g, 0.2 mmol, 1.0 equiv.), $K_2S_2O_8$ (0.1623 g, 3.0 equiv.), and dry acetonitrile (2 mL) were added to a 10 mL reaction tube that had been dried in a high-temperature oven. Subsequently, the reaction mixture was heated in an oil bath at 90 °C. Four enaminones with different N-substituents were tested, and each enaminone underwent 11 identical reactions. Dibromomethane was used as an internal standard to determine the yield via ^{1}H NMR of the crude mixture. The reactions were monitored at time points of 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, and 90 minutes. The yields of product **3a** after the reactions indicated that compound **C** exhibited the fastest rate of product formation, followed by **A**, then **B**, with **D** being the slowest. The experimental observations confirmed this order of reaction rates. (**Figure S5**）

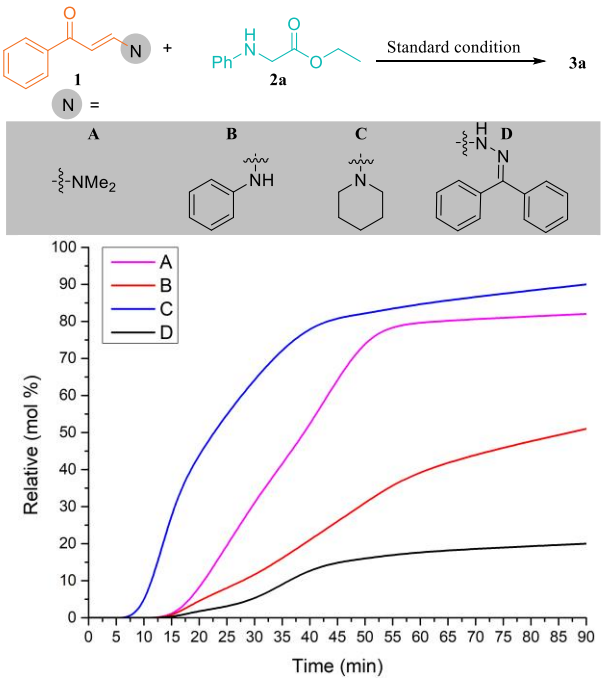
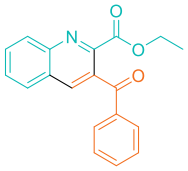


Figure S5. Effect of N substituents on reaction rate

5.Spectroscopic Data of 3a-3s, 4a-4l



5.1 Spectroscopic Data of 3a

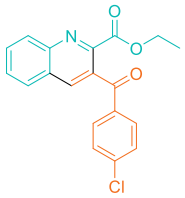
Ethyl 3-benzoylquinoline-2-carboxylate

50.0 mg, 82% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 8.8 Hz, 1H), 8.33 (s, 1H), 7.94-7.86 (m, 2H), 7.82 (d, *J* = 7.2 Hz, 2H), 7.72 (t, *J* = 7.0 Hz, 1H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 164.2, 146.8, 146.4, 136.3, 136.0, 132.5, 132.1, 130.6, 129.5, 128.6, 128.3, 127.7, 127.1, 126.5, 61.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{16}NO_3^+$ $[M+H]^+$: 306.1125; found: 306.1124.



5.2 Spectroscopic Data of 3b

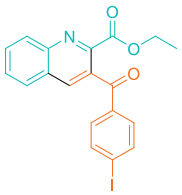
Ethyl 3-(4-chlorobenzoyl)quinoline-2-carboxylate

39.3 mg, 58% yield. White solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.6 Hz, 1H), 8.31 (s, 1H), 7.93 (d, *J* = 8.8 Hz, 1H), 7.91-7.88 (m, 1H), 7.82 (t, *J* = 1.7 Hz, 1H), 7.74 (t, *J* = 7.0 Hz, 1H), 7.67-7.65 (m, 1H), 7.58-7.55 (m, 1H), 7.41 (t, *J* = 7.8 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 1.25 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 164.1, 146.5, 146.4, 137.7, 136.2, 134.1, 132.4, 131.6, 130.8, 129.5, 129.0, 128.5, 128.4, 127.1, 126.7, 61.7, 12.9.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}ClNO_3^+$ $[M+H]^+$: 340.0735; found: 340.0733.



5.3 Spectroscopic Data of 3c

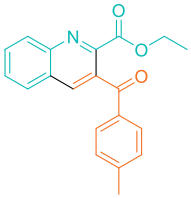
Ethyl 3-(4-iodobenzoyl)quinoline-2-carboxylate

44.0 mg, 51% yield. White solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 8.5 Hz, 1H), 8.30 (s, 1H), 7.94-7.87 (m, 2H), 7.84 (d, *J* = 8.5 Hz, 2H), 7.73 (t, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 8.5 Hz, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.0, 164.1, 146.5, 146.4, 137.0, 136.1, 135.4, 131.7, 130.8, 129.8, 129.5, 128.5, 127.1, 126.5, 61.7, 12.9.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}INO_3^+$ $[M+H]^+$: 432.0091; found: 432.0090.



5.4 Spectroscopic Data of 3d

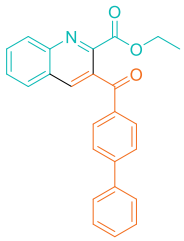
Ethyl 3-(4-methylbenzoyl)quinoline-2-carboxylate

42.0 mg, 65% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.34 (d, *J* = 8.0 Hz, 1H), 8.31 (s, 1H), 7.91 (d, *J* = 8 Hz, 2H), 7.89-7.85 (m, 1H), 7.73 (d, *J* = 8.0 Hz, 3H), 7.28 (s, 1H), 4.28 (q, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 164.2, 146.4, 143.5, 136.1, 133.5, 132.3, 130.5, 129.5, 128.8, 128.4, 128.2, 127.1, 126.6, 61.5, 20.7, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{18}NO_3^+$ $[M+H]^+$: 320.1281; found: 320.1280.



5.5 Spectroscopic Data of 3e

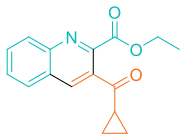
Ethyl 3-([1,1'-biphenyl]-4-carbonyl)quinoline-2-carboxylate

54.9 mg, 72% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.37-8.36 (m, 2H), 7.95-7.87 (m, 4H), 7.76-7.72 (m, 1H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.65-7.62 (m, 2H), δ 7.48 (t, *J* = 8.0 Hz, 2H), 7.41 (t, *J* = 8.0 Hz, 1H)., 4.32 (q, *J* = 7.2 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 164.3, 146.8, 146.4, 145.2, 138.7, 136.2, 134.7, 132.3, 130.6, 129.5, 129.2, 128.3, 128.0, 127.4, 127.1, 126.33, 126.29, 61.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{25}H_{20}NO_3^+$ $[M+H]^+$: 382.1438; found: 382.1436.



5.6 Spectroscopic Data of 3f

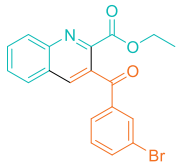
Ethyl 3-(cyclopropanecarbonyl)quinoline-2-carboxylate

30.1 mg, 57% yield. Yellow oil, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.57 (s, 1H), 8.25 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.2 Hz, 1H), 7.88-7.84 (m, 1H), 7.69 (t, *J* = 7.5 Hz, 1H), 4.51 (q, *J* = 7.2 Hz, 2H), 2.57-2.51 (m, 1H), 1.43 (t, *J* = 7.2 Hz, 3H), 1.38-1.34 (m, 2H), 1.18-1.37 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 165.6, 148.1, 146.6, 135.8, 131.5, 130.9, 129.1, 127.8, 127.4, 126.2, 61.5, 19.1, 13.1, 11.7.

HRMS (ESI) *m/z*: Calcd for $C_{16}H_{16}NO_3^+$ $[M+H]^+$: 270.1125; found: 270.1124.



5.7 Spectroscopic Data of 3g

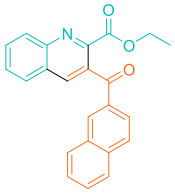
Ethyl 3-(3-bromobenzoyl)quinoline-2-carboxylate

27.6 mg, 36% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.6 Hz, 1H), 8.31 (s, 1H), 7.97 (t, *J* = 1.8 Hz, 1H), 7.94 (d, *J* = 7.2 Hz, 1H), 7.91-7.88 (m, 1H), 7.76-7.69 (m, 3H), 7.35 (t, *J* = 7.8 Hz, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 165.1, 147.5, 147.4, 138.9, 137.2, 136.3, 132.6, 132.3, 131.8, 130.6, 130.3, 129.5, 128.2, 127.6, 123.1, 62.8, 13.9.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}BrNO_3^+$ $[M+H]^+$: 384.0230; found: 384.0229.



5.8 Spectroscopic Data of 3h

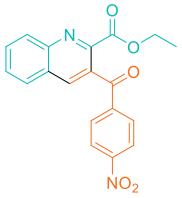
Ethyl 3-(2-naphthoyl)quinoline-2-carboxylate

50.4 mg, 71% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.40 (s, 1H), 8.39 (d, *J* = 9.0 Hz, 1H), 8.13 (s, 1H), 8.08-8.06 (m, 1H), 7.97-7.89 (m, 4H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.14 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 164.2, 146.9, 146.5, 136.4, 134.7, 133.5, 132.3, 131.3, 131.0, 130.6, 129.5, 128.6, 128.3, 127.84, 127.81, 127.1, 126.8, 126.6, 126.0, 123.5, 61.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{23}H_{18}NO_3^+$ $[M+H]^+$: 356.1281; found: 356.1280.



5.9 Spectroscopic Data of 3i

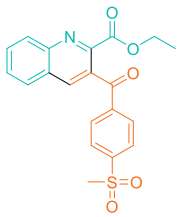
Ethyl 3-(4-nitrobenzoyl)quinoline-2-carboxylate

32.2 mg, 46% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.63-8.62 (m, 1H), 8.47-8.44 (m, 1H), 8.39 (d, *J* = 9.0 Hz, 1H), 8.32 (s, 1H), 8.16-8.14 (m, 1H), 7.94 (t, *J* = 7.2 Hz, 2H), 7.77 (t, *J* = 6.6 Hz, 1H), 7.69 (t, *J* = 5.8 Hz, 1H), 4.35 (q, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 191.5, 164.1, 147.4, 146.6, 145.9, 137.7, 136.0, 133.8, 131.3, 131.0, 129.7, 129.0, 128.8, 127.1, 126.6, 126.5, 123.1, 61.9, 13.0.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}N_2O_5^+$ $[M+H]^+$: 351.0975; found: 351.0974.



5.10 Spectroscopic Data of 3j

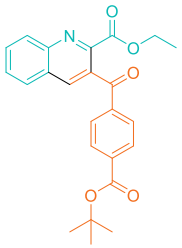
Ethyl 3-(4-(methylsulfonyl)benzoyl)quinoline-2-carboxylate

34.4 mg, 45% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.23 (d, *J* = 9.1 Hz, 1H), 8.20 (s, 1H), 8.10 (d, *J* = 2.0 Hz, 1H), 8.07-8.04 (m, 3H), 7.99-7.97 (m, 3H), 4.33 (q, *J* = 7.2 Hz, 2H), 3.09 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.9, 164.9, 147.2, 146.1, 144.5, 140.9, 135.9, 135.7, 133.4, 132.1, 130.2, 130.1, 129.2, 128.6, 127.9, 124.3, 118.3, 63.7, 44.3, 14.0.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{18}NO_5S^+$ $[M+H]^+$: 384.0900; found: 384.0901.



5.11 Spectroscopic Data of 3k

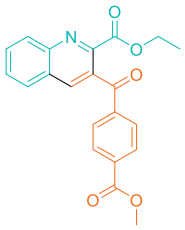
Ethyl 3-(4-(*tert*-butoxycarbonyl)benzoyl)quinoline-2-carboxylate

51.0 mg, 63% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.6 Hz, 1H), 8.32 (s, 1H), 8.07 (d, *J* = 8.5 Hz, 2H), 7.94-7.91 (m, 1H), 7.91-7.88 (m, 1H), 7.85 (d, *J* = 8.5 Hz, 2H), 7.74 (t, *J* = 7.2 Hz, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 1.60 (s, 9H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.1, 164.1, 163.7, 146.5, 138.9, 136.3, 135.0, 131.8, 130.8, 129.5, 128.7, 128.5, 128.3, 127.1, 126.5, 80.9, 61.7, 27.1, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{24}H_{24}NO_5$ $[M+H]^+$: 406.1649; found: 406.1648.



5.12 Spectroscopic Data of 3l

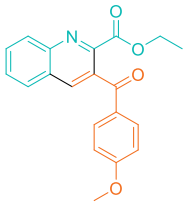
Ethyl 3-(4-(methoxycarbonyl)benzoyl)quinoline-2-carboxylate

38.5 mg, 53% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.37 (d, *J* = 8.5 Hz, 1H), 8.34 (s, 1H), 8.13 (d, *J* = 8.2 Hz, 2H), 7.93 (t, *J* = 7.2 Hz, 2H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 4.29 (q, *J* = 7.2 Hz, 2H), 3.96 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.1, 165.1, 164.1, 146.5, 146.4, 139.3, 136.3, 133.1, 131.7, 130.8, 129.5, 128.9, 128.5, 128.4, 127.1, 126.5, 61.7, 51.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{21}H_{18}NO_5^+$ $[M+H]^+$: 364.1179; found: 364.1179.



5.13 Spectroscopic Data of 3m

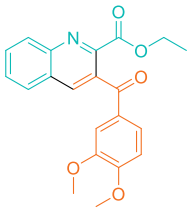
Ethyl 3-(4-methoxybenzoyl)quinoline-2-carboxylate

40.9 mg, 61 % yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.34 (d, *J* = 8.5 Hz, 1H), 8.30 (s, 1H), 7.90 (d, *J* = 8.6 Hz, 1H), 7.88-7.84 (m, 1H), 7.81 (d, *J* = 8.8 Hz, 2H), 7.71 (t, *J* = 7.2 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.88 (s, 3H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 164.3, 162.9, 146.8, 146.3, 135.9, 132.5, 131.0, 130.4, 129.5, 129.0, 128.2, 127.0, 126.6, 112.9, 61.5, 54.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{18}NO_4$ $[M+H]^+$: 336.1230; found: 336.1228.



5.14 Spectroscopic Data of 3n

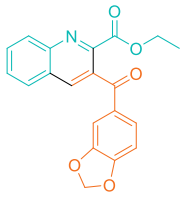
Ethyl 3-(3,4-dimethoxybenzoyl)quinoline-2-carboxylate

41.6 mg, 57% yield. Yellow oil, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 8.5 Hz, 1H), 8.32 (s, 1H), 7.92-7.86 (m, 2H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.62 (d, *J* = 1.9 Hz, 1H), 7.19-7.17 (m, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.96 (s, 3H), 3.94 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 164.3, 152.7, 148.3, 146.8, 146.3, 136.1, 132.3, 130.5, 129.4, 129.2, 128.3, 127.0, 126.6, 124.5, 123.5, 109.6, 108.9, 61.6, 55.1, 12.9.

HRMS (ESI) *m/z*: Calcd for $C_{21}H_{20}NO_5^+$ $[M+H]^+$: 366.1336; found: 366.1336.



5.15 Spectroscopic Data of 3o

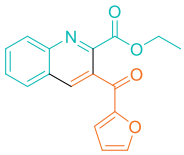
Ethyl 3-(benzo[*d*][1,3]dioxole-5-carbonyl)quinoline-2-carboxylate

37.0 mg, 53% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.33 (d, *J* = 8.5 Hz, 1H), 8.28 (s, 1H), 7.90 (d, *J* = 8.7 Hz, 1H), 7.88-7.84 (m, 1H), 7.71 (t, *J* = 7 Hz, 1H), 7.44 (d, *J* = 1.7 Hz, 1H), 7.27-7.23 (m, 1H), 6.81 (d, *J* = 8.1 Hz, 1H), 6.07 (s, 2H), 4.33 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.0, 164.2, 151.3, 147.4, 146.6, 146.3, 135.9, 132.4, 130.9, 130.5, 129.5, 128.3, 127.0, 126.5, 125.9, 107.8, 106.9, 101.1, 61.6, 12.9.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{16}NO_5^+$ $[M+H]^+$: 350.1023; found: 350.1021.



5.16 Spectroscopic Data of 3p

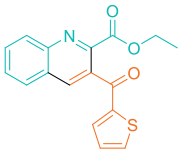
Ethyl 3-(furan-2-carbonyl)quinoline-2-carboxylate

30.7 mg, 52% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.47 (s, 1H), 8.33 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.91-7.86 (m, 1H), 7.77-7.69 (m, 1H), 7.66-7.64 (m, 1H), 7.19-7.17 (m, 1H), 6.61-6.59 (m, 1H), 4.36 (q, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 180.7, 164.3, 146.3, 136.7, 130.9, 130.8, 129.44, 129.38, 128.3, 128.2, 127.3, 127.2, 126.5, 118.5, 111.7, 61.6, 12.9.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{14}NO_4^+$ $[M+H]^+$: 296.0917; found: 296.0915.



5.17 Spectroscopic Data of 3q

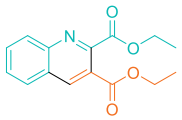
Ethyl 3-(thiophene-2-carbonyl)quinoline-2-carboxylate

41.7 mg, 67% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.41 (s, 1H), 8.34 (d, *J* = 8.4 Hz, 1H), 7.93 (t, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.2 Hz, 1H), 7.77 (d, *J* = 4.9 Hz, 1H), 7.75 (t, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 3.8 Hz, 1H), 7.13 (t, *J* = 4.7 Hz, 1H), 4.35 (q, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 185.7, 164.2, 146.5, 143.2, 136.0, 134.1, 133.8, 131.7, 130.7, 129.5, 128.3, 127.3, 127.1, 126.4, 61.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{14}NO_3S^+$ $[M+H]^+$: 312.0689; found: 312.0687.



5.18 Spectroscopic Data of 3r[5]

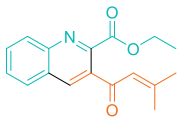
Diethyl quinoline-2,3-dicarboxylate

29.6 mg, 54% yield. Yellow oil, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.79 (s, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.2 Hz, 1H), 7.86 (t, *J* = 7.6 Hz, 1H), 7.69-7.66 (m, 1H), 4.53 (q, *J* = 7.2 Hz, 2H), 4.44 (q, *J* = 7.2 Hz, 2H), 1.45 (t, *J* = 7.2 Hz, 3H), 1.42 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 164.1, 150.0, 147.0, 138.6, 131.3, 128.8, 127.6, 127.6, 126.0, 121.4, 61.4, 61.0, 13.2, 13.1.

HRMS (ESI) *m/z*: Calcd for $C_{15}H_{16}NO_4^+$ $[M+H]^+$: 274.1074; found: 274.1076.



5.19 Spectroscopic Data of 3s

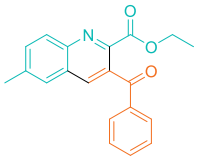
Ethyl 3-(3-methylbut-2-enoyl)quinoline-2-carboxylate

42.0 mg, 65% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.41 (s, 1H), 8.25-8.23 (m, 1H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.85-7.81 (m, 1H), 7.69-7.65 (m, 1H), 6.59 (s, 1H), 4.49 (q, *J* = 7.2 Hz, 2H), 2.25 (s, 3H), 2.04 (s, 3H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 Hz, $CDCl_3$) δ 190.1, 165.7, 157.9, 148.5, 146.4, 135.4, 132.8, 130.6, 129.1, 127.7, 127.3, 126.3, 121.4, 61.4, 27.2, 20.3, 13.0.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{18}NO_3^+$ $[M+H]^+$: 284.1281; found: 284.1283.



5.20 Spectroscopic Data of 4a[5]

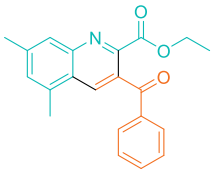
Ethyl 3-benzoyl-6-methylquinoline-2-carboxylate

41.5 mg, 65% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.24-8.22 (m, 2H), 7.81 (d, *J* = 7.2 Hz, 2H), 7.72-7.69 (m, 1H), 7.67 (s, 1H), 7.61-7.57 (m, 1H), 7.46 (t, *J* = 7.9 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.59 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 164.2, 145.7, 145.0, 138.8, 136.1, 135.4, 133.0, 132.4, 132.3, 129.1, 128.6, 127.7, 126.7, 125.8, 61.5, 20.8, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{18}NO_3^+$ $[M+H]^+$: 320.1281; found: 320.1280.



5.21 Spectroscopic Data of 4b

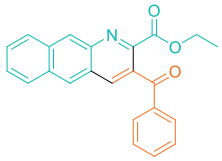
Ethyl 3-benzoyl-5,7-dimethylquinoline-2-carboxylate

47.3 mg, 71% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.42 (s, 1H), 7.96 (s, 1H), 7.82-7.79 (m, 2H), 7.60-7.56 (m, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.37 (s, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 2.65 (s, 3H), 2.56 (s, 3H), 1.17 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 194.1, 164.3, 147.2, 146.1, 141.0, 136.3, 133.8, 132.7, 132.3, 131.1, 130.9, 128.5, 127.6, 126.5, 124.3, 61.4, 21.0, 17.5, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{21}H_{20}NO_3^+$ $[M+H]^+$: 334.1438; found: 334.1439.



5.22 Spectroscopic Data of 4c

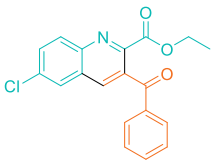
Ethyl 3-benzoylbenzo[*g*]quinoline-2-carboxylate

42.6 mg, 60% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 30/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.08 (s, 1H), 8.63-8.58 (m, 1H), 8.20 (d, *J* = 9.2 Hz, 1H), 8.13 (d, *J* = 9.2 Hz, 1H), 8.01-7.98 (m, 1H), 7.86-7.83 (m, 2H), 7.75-7.72 (m, 2H), 7.64-7.58 (m, 1H), 7.48 (t, *J* = 7.9 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 194.1, 164.1, 147.0, 145.6, 136.2, 132.7, 132.5, 132.2, 131.6, 130.8, 128.6, 128.0, 127.9, 127.8, 127.7, 127.0, 126.9, 125.0, 122.4, 61.6, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{23}H_{18}NO_3^+$ $[M+H]^+$: 356.1281; found: 356.1283.



5.23 Spectroscopic Data of 4d

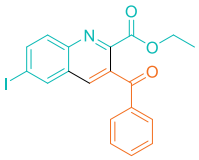
Ethyl 3-benzoyl-6-chloroquinoline-2-carboxylate

39.3 mg, 58% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 9.0 Hz, 1H), 8.23 (s, 1H), 7.90 (d, *J* = 2.2 Hz, 1H), 7.82-7.79 (m, 3H), 7.63-7.60 (m, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.2, 163.9, 146.9, 144.7, 135.1, 134.4, 133.1, 132.6, 131.6, 131.0, 128.6, 128.3, 127.8, 127.2, 125.7, 61.7, 13.1.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}ClNO_3^+$ $[M+H]^+$: 340.0735; found: 340.0732.



5.24 Spectroscopic Data of 4e

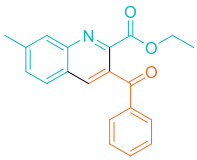
Ethyl 3-benzoyl-6-iodoquinoline-2-carboxylate

56.0 mg, 65% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.31 (d, *J* = 1.9 Hz, 1H), 8.19 (s, 1H), 8.12-8.09 (m, 1H), 8.05 (d, *J* = 8.9 Hz, 1H), 7.80-7.78 (m, 2H), 7.64-7.59 (m, 1H), 7.47 (t, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 193.2, 163.9, 147.1, 145.3, 139.4, 137.2, 135.8, 135.7, 134.8, 132.9, 132.6, 130.8, 128.6, 128.0, 127.8, 61.7, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}INO_3^+$ $[M+H]^+$: 432.0091; found: 432.0090.



5.25 Spectroscopic Data of 4f

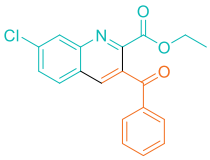
Ethyl 3-benzoyl-7-methylquinoline-2-carboxylate

46.6 mg, 73% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (s, 1H), 8.12 (s, 1H), 7.84-7.78 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.54 (d, *J* = 7.0 Hz, 2H), 7.47 (t, *J* = 7.8 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.62 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 164.4, 146.9, 146.7, 136.1, 136.0, 132.4, 131.3, 130.6, 128.6, 128.7, 128.4, 127.68, 127.65, 126.7, 61.5, 21.1, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{20}H_{18}NO_3^+$ $[M+H]^+$: 320.1281; found: 320.1280.



5.26 Spectroscopic Data of 4g

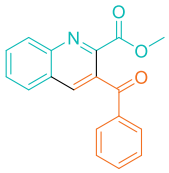
Ethyl 3-benzoyl-7-chloroquinoline-2-carboxylate

35.9 mg, 53% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.36 (s, 1H), 8.32 (s, 1H), 7.86 (d, *J* = 8.7 Hz, 1H), 7.81 (d, *J* = 7.2 Hz, 2H), 7.69-7.66 (m, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 163.9, 148.0, 146.7, 136.8, 136.1, 132.6, 132.2, 129.4, 129.2, 128.6, 128.4, 128.2, 127.8, 127.5, 61.7, 12.8.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{15}ClNO_3^+$ $[M+H]^+$: 340.0735; found: 340.0737.



5.27 Spectroscopic Data of 4h

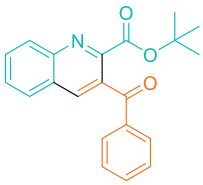
Methyl 3-benzoylquinoline-2-carboxylate

43.7 mg, 75% yield. Yellow oil, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.22 (s, 1H), 8.21 (d, *J* = 9.2 Hz, 1H), 8.09 (d, *J* = 2.0 Hz, 1H), 7.96-7.94 (m, 1H), 7.81-7.79 (m, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 3H), 3.84 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 193.2, 164.3, 146.6, 144.9, 135.7, 135.0, 134.3, 133.2, 132.7, 130.96, 129.1, 128.6, 127.8, 127.7, 122.9, 52.3.

HRMS (ESI) *m/z*: Calcd for $C_{18}H_{14}NO_3^+$ $[M+H]^+$: 292.0968; found: 292.0967.



5.28 Spectroscopic Data of 4i

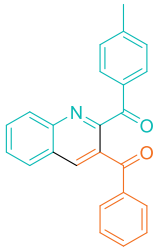
Tert-butyl 3-benzoylquinoline-2-carboxylate

27.3 mg, 41% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.6 Hz, 1H), 8.30 (s, 1H), 7.90 (d, *J* = 8.5 Hz, 1H), 7.87-7.83 (m, 3H), 7.70 (t, *J* = 8.0 Hz, 1H), 7.64-7.59 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 1.30 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.6, 162.9, 147.8, 146.5, 136.2, 136.1, 132.5, 131.8, 130.4, 129.5, 128.8, 128.0, 127.7, 127.0, 126.5, 83.0, 26.4.

HRMS (ESI) *m/z*: Calcd for $C_{21}H_{20}NO_3^+$ $[M+H]^+$: 334.1438; found: 334.1437.



5.29 Spectroscopic Data of 4j

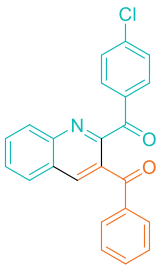
(3-benzoylquinolin-2-yl)(*p*-tolyl)methanone

44.2 mg, 63% yield. Yellow solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.40 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.2 Hz, 2H), 7.93 (d, *J* = 8.2 Hz, 1H), 7.87 (t, *J* = 7.4 Hz, 3H), 7.72 (t, *J* = 7.5 Hz, 1H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 2.43 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 193.4, 156.6, 146.8, 144.5, 137.9, 136.8, 133.4, 133.2, 133.0, 131.8, 131.1, 130.2, 130.1, 129.1, 128.9, 128.6, 128.4, 126.6, 21.9.

HRMS (ESI) *m/z*: Calcd for $C_{24}H_{18}NO_2^+$ $[M+H]^+$: 352.1332; found: 352.1331.



5.30 Spectroscopic Data of 4k

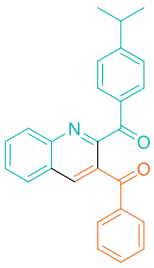
(3-benzoylquinolin-2-yl)(4-chlorophenyl)methanone

30.4 mg, 41% yield. White solid, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.41 (s, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 8.6 Hz, 2H), 7.96-7.91 (m, 1H), 7.88 (t, *J* = 7.0 Hz, 3H), 7.81 (d, *J* = 8.5 Hz, 1H), 7.74 (t, *J* = 7.5 Hz, 1H), 7.61 (t, *J* = 6.8 Hz, 1H), 7.50-7.42 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 191.4, 154.9, 145.7, 139.0, 137.1, 135.6, 133.0, 132.6, 131.9, 131.3, 130.9, 129.1, 129.0, 128.11, 128.08, 128.0, 127.68, 127.65.

HRMS (ESI) *m/z*: Calcd for $C_{23}H_{15}ClNO_2^+$ $[M+H]^+$: 372.0786; found: 372.0785.



5.31 Spectroscopic Data of 4l

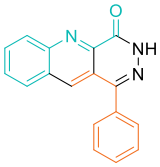
(6-isopropylquinoline-2,3-diyl)bis(phenylmethanone)

46.2 mg, 61% yield. Yellow oil, (Flash column chromatography eluent, petroleum ether/ethyl acetate = 20/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (s, 1H), 8.16 (d, *J* = 8.6 Hz, 1H), 8.10-8.06 (m, 2H), 7.89-7.86 (m, 2H), 7.81-7.78 (m, 1H), 7.72 (d, *J* = 1.6 Hz, 1H), 7.61-7.55 (m, 2H), 7.48-7.43 (m, 4H), 3.19-3.12 (m, 1H), 1.38 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 192.7, 154.5, 149.0, 144.7, 136.6, 135.8, 134.7, 132.41, 132.35, 131.9, 130.9, 129.9, 129.0, 128.9, 127.7, 127.6, 127.3, 123.5, 33.2, 22.7.

HRMS (ESI) *m/z*: Calcd for $C_{26}H_{22}NO_2^+$ $[M+H]^+$: 380.1645; found: 380.1646.



5.32 Spectroscopic Data of 5

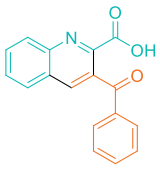
1-phenylpyridazino[4,5-*b*]quinolin-4(3*H*)-one

79.2 mg, 58% yield. Yellow solid, (Direct extraction).

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.97 (s, 1H), 8.79 (s, 1H), 8.30 (t, *J* = 7.2 Hz, 2H), 8.03 (t, *J* = 7.6 Hz, 1H), 7.80 (t, *J* = 7.6 Hz, 1H), 7.71-7.69 (m, 2H), 7.63-7.57 (m, 3H).

^{13}C NMR (100 MHz, DMSO) δ 158.8, 149.4, 146.9, 143.7, 137.0, 135.2, 133.3, 130.1, 130.0, 129.9, 129.7, 129.5, 129.21, 129.16, 123.2.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{12}N_3O_1^+$ $[M+H]^+$: 274.0975; found: 274.0974.



5.33 Spectroscopic Data of 6

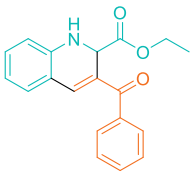
3-benzoylquinoline-2-carboxylic acid

76.1 mg, 55% yield. Yellow solid, (Direct extraction).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.34 (s, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 8.00-7.91 (m, 1H), 7.79 (m, 3H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.2, 162.1, 144.8, 142.5, 136.5, 135.9, 132.7, 132.6, 131.1, 129.2, 128.5, 128.5, 127.9, 127.7, 127.1.

HRMS (ESI) *m/z*: Calcd for $C_{17}H_{12}NO_3^+$ $[M+H]^+$: 278.0812; found: 278.0812



5.34 Spectroscopic Data of Int-4

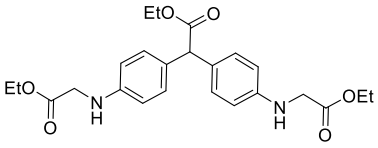
Ethyl 3-benzoyl-1,2-dihydroquinoline-2-carboxylate

37.5 mg, 61% yield. yellow solid. (Flash column chromatography eluent, petroleum ether/ethyl acetate = 10/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72-7.70 (m, 3H), 7.51 (d, *J* = 7.3 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 3H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 7.6 Hz, 2H), 5.47 (s, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 193.4, 171.3, 141.9, 138.7, 137.6, 130.5, 129.0, 127.7, 127.4, 126.0, 120.3, 114.7, 60.4, 37.2, 13.6.

HRMS (ESI) *m/z*: Calcd for $C_{19}H_{18}NO_3^+$ $[M+H]^+$: 308.1281; found:308.1282.



5.35 Spectroscopic Data of 9

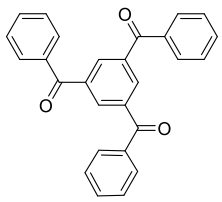
diethyl 2,2'-(((2-ethoxy-2-oxoethane-1,1-diyl)bis(4,1-phenylene))bis(azanediyl)) diacetate

63.6 mg, 72% yield. yellow oil. (Flash column chromatography eluent, petroleum ether/ethyl acetate = 5/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.12 (d, *J* = 8.5 Hz, 4H), 6.55 (d, *J* = 8.5 Hz, 4H), 4.79 (s, 1H), 4.24 (q, *J* = 7.2 Hz, 4H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.87 (s, 4H), 1.29 (t, *J* = 7.2 Hz, 6H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 170.1, 144.9, 128.4, 127.9, 112.0, 60.3, 59.9, 54.4, 44.9, 28.7, 13.2.

HRMS (ESI) *m/z*: Calcd for $C_{24}H_{31}N_2O_6^+$ $[M+H]^+$: 443.2177; found: 443.2174.



5.36 Spectroscopic Data of 11[6]

Benzene-1,3,5-triyltris(phenylmethanone)

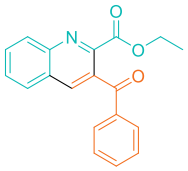
35.9 mg, 46% yield. white solid. (Flash column chromatography eluent, petroleum ether/ethyl acetate = 15/1, V/V).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.40 (s, 3H), 7.86-7.84 (m, 6H), 7.54-7.50 (m, 9H).

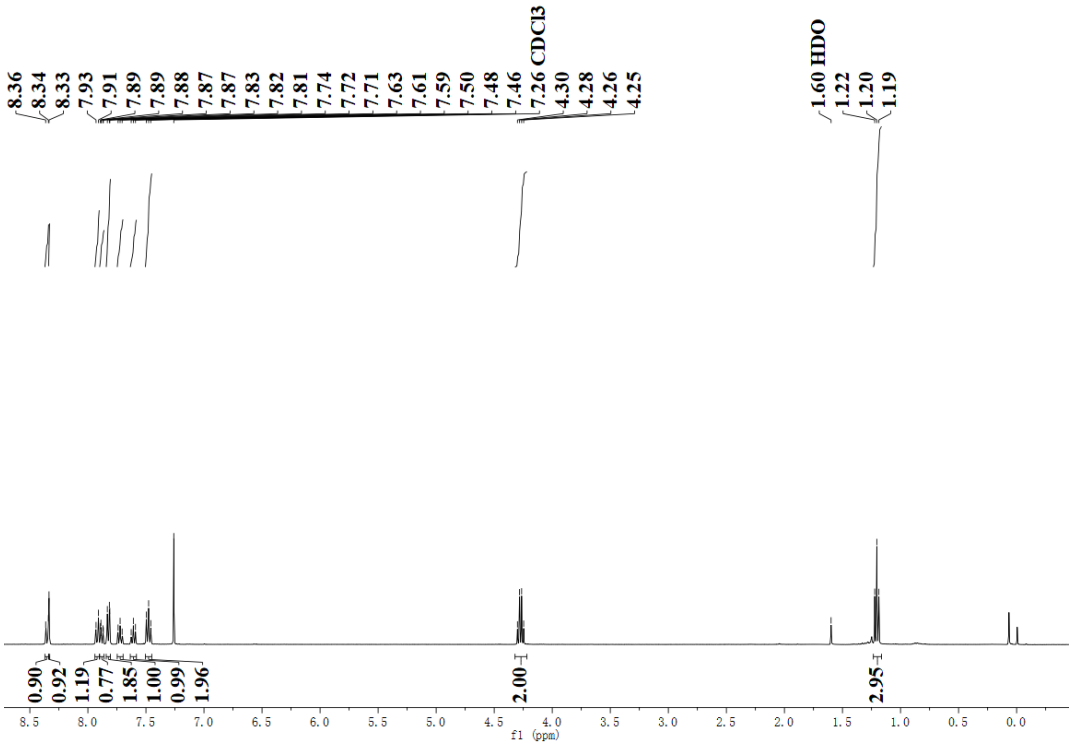
^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 137.2, 135.4, 133.1, 132.3, 129.1, 127.7.

HRMS (ESI) *m/z*: Calcd for $C_{27}H_{19}O_3^+$ $[M+H]^+$: 391.1329; found: 391.1326.

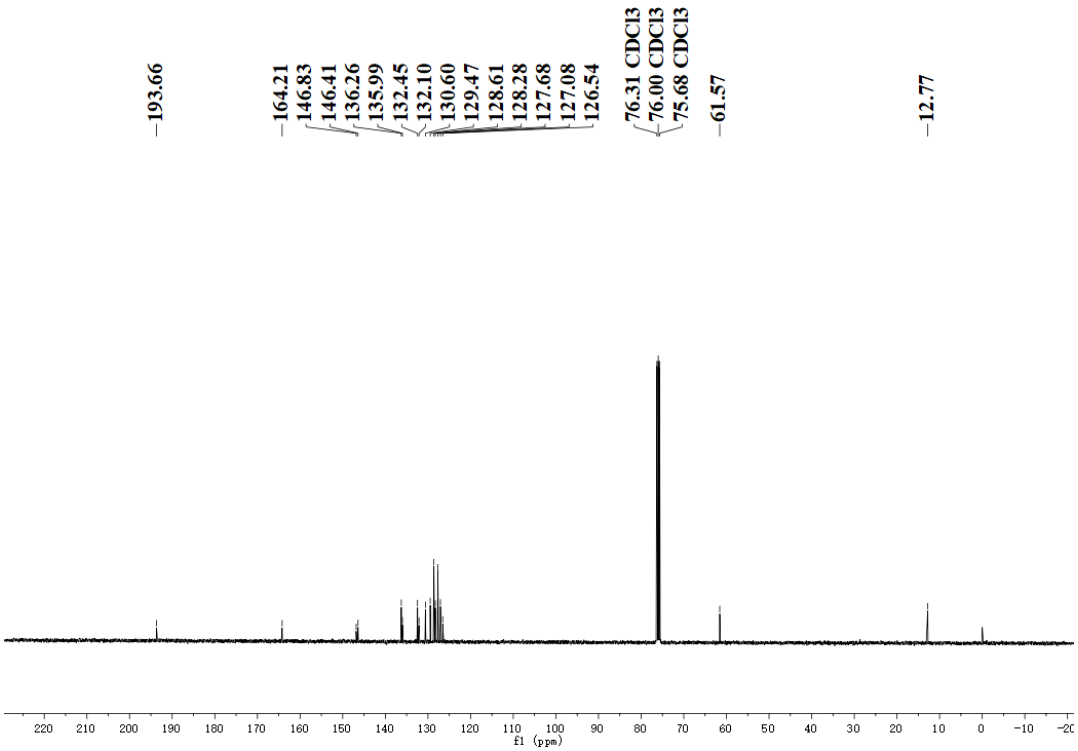
6. ^{1}H NMR and ^{13}C NMR spectra of 3a-3s and 4a-4l

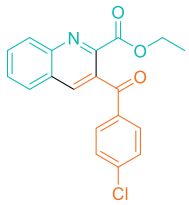


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3a

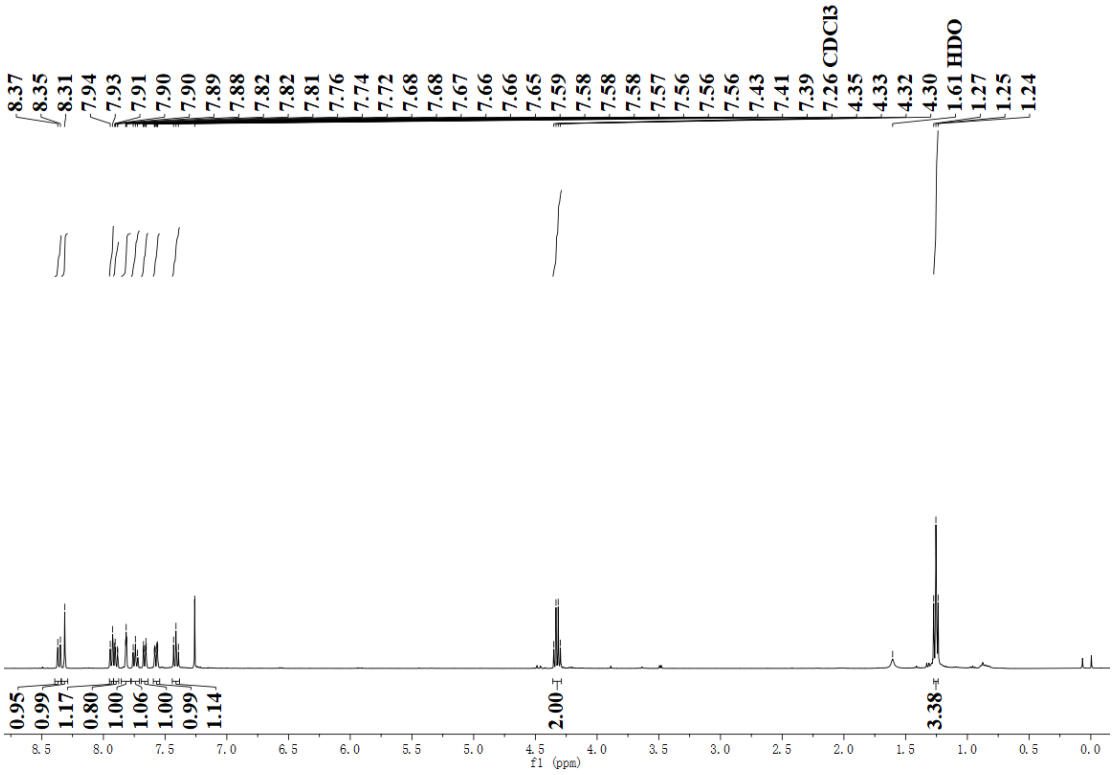


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3a

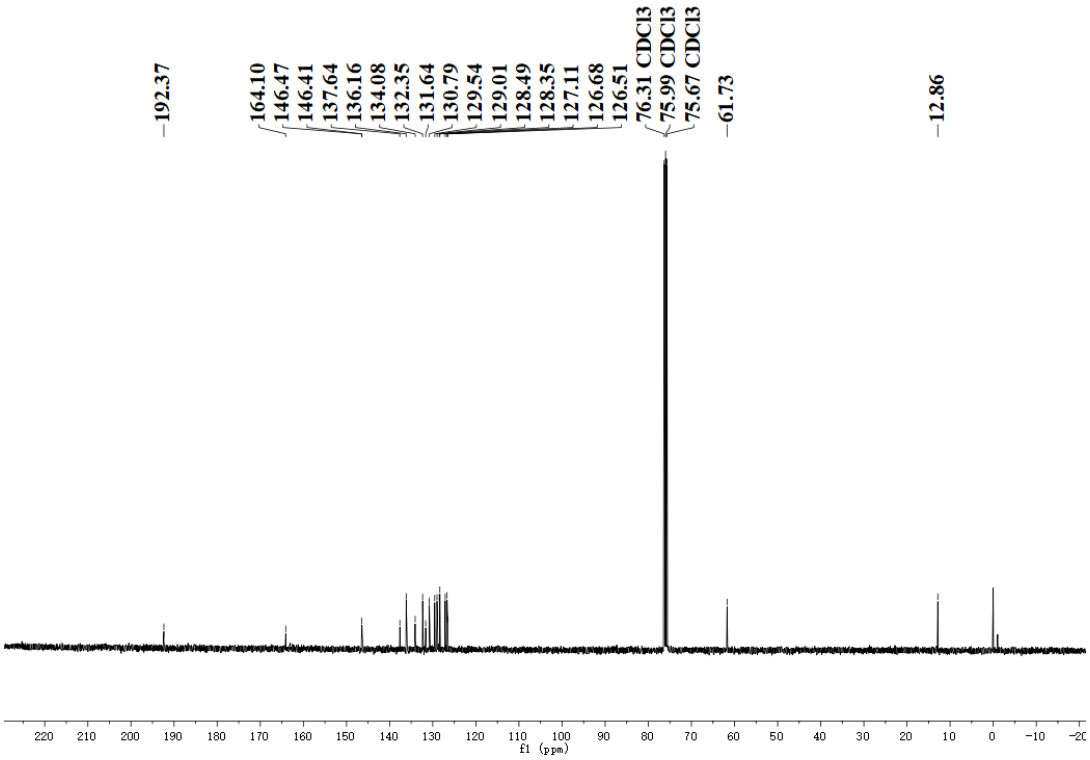


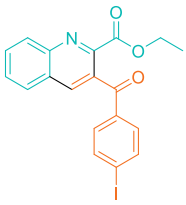


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3b

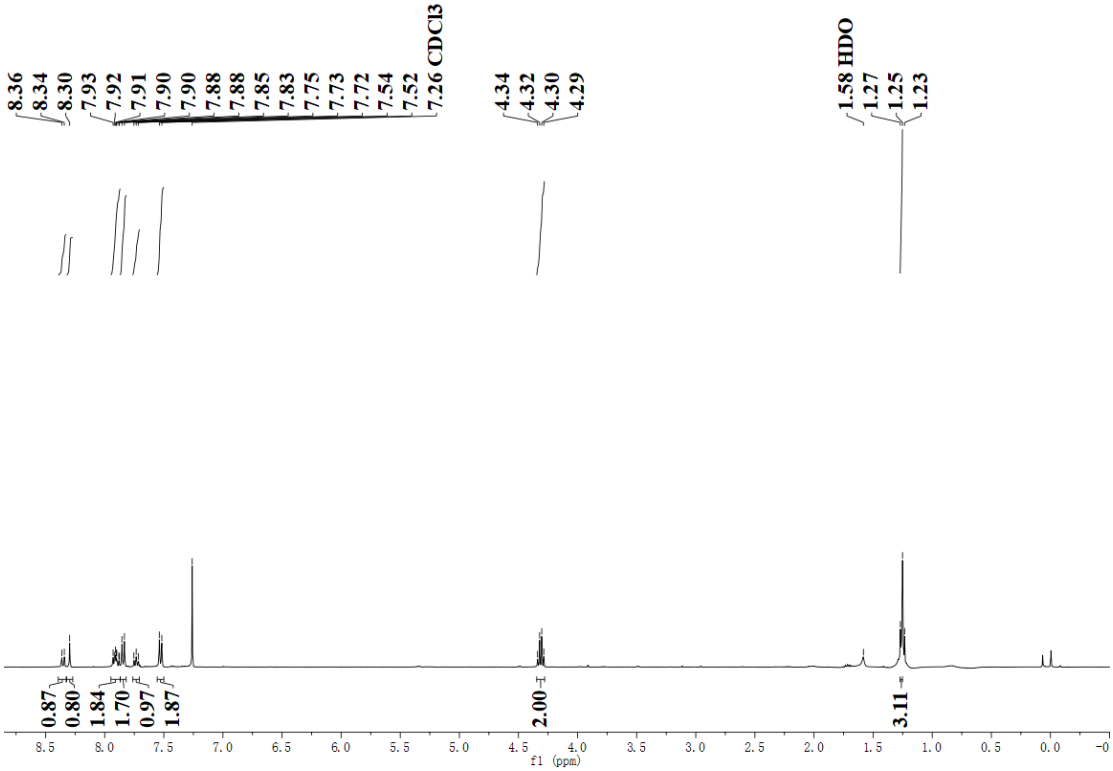


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3b

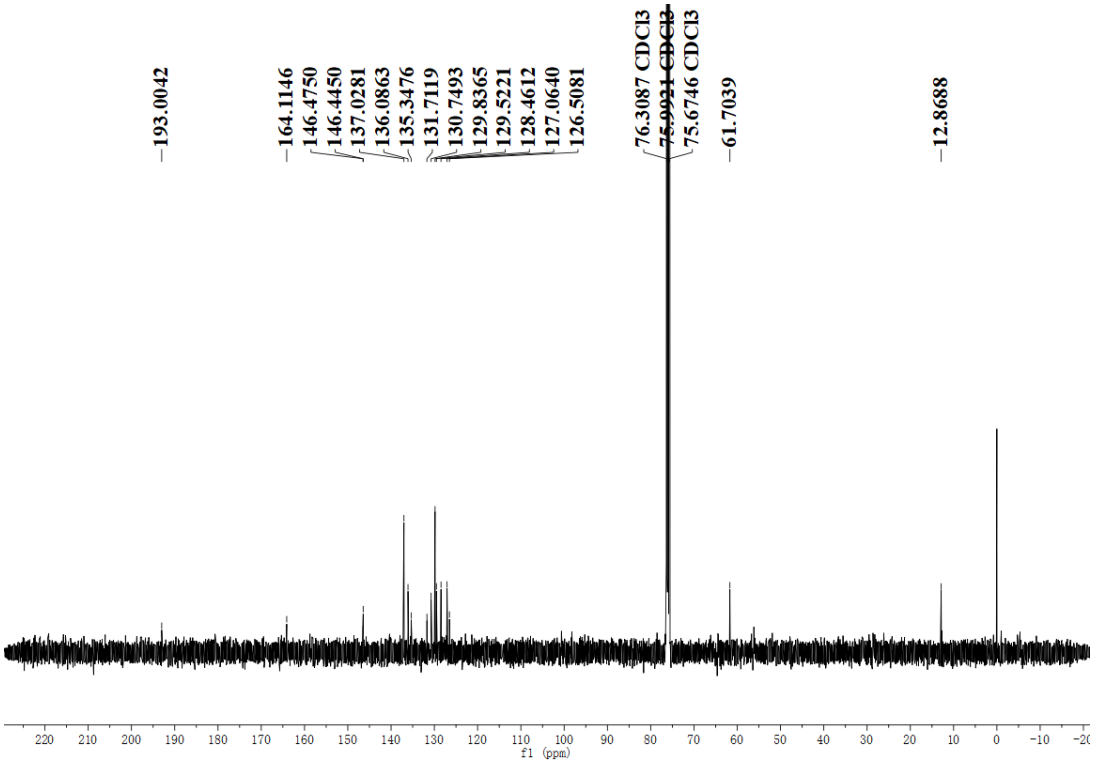


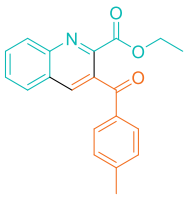


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3c

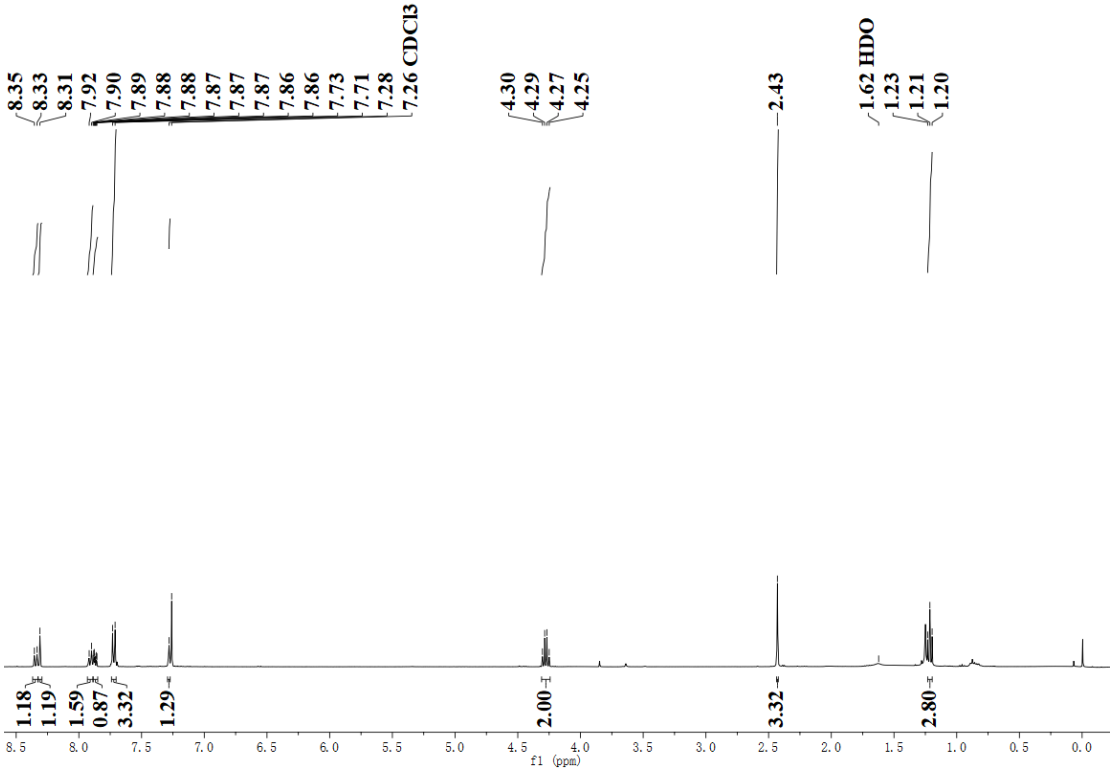


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3c

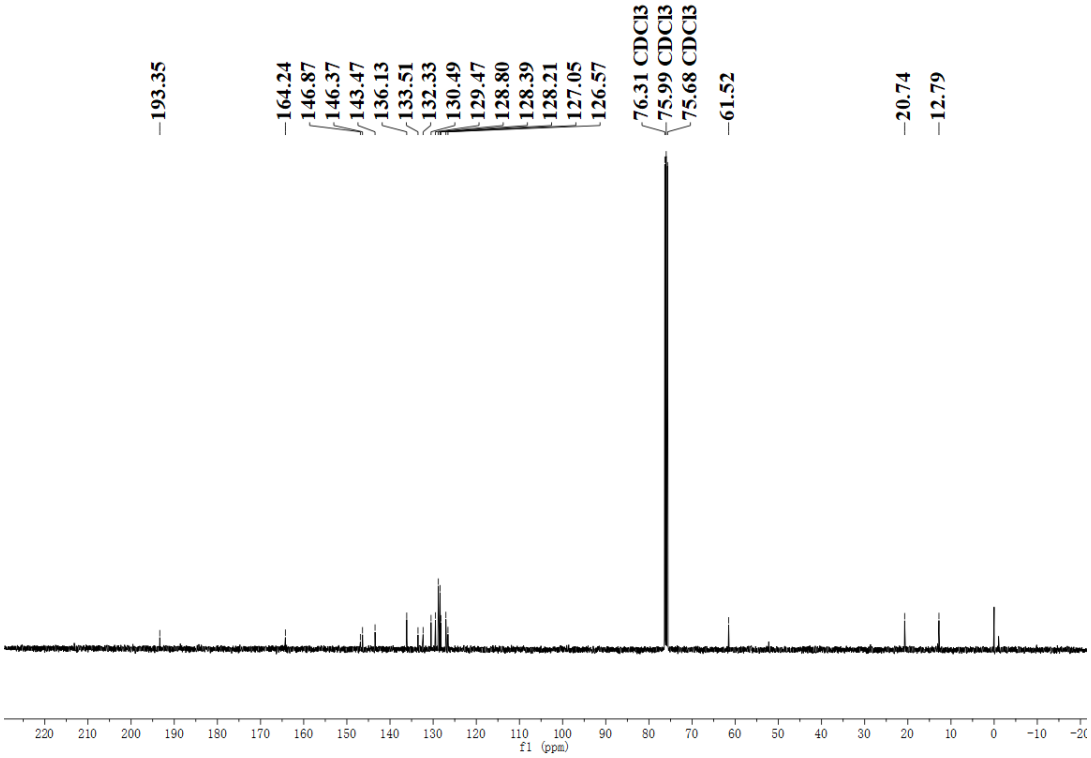


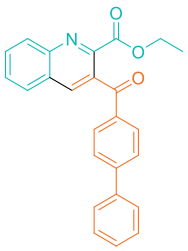


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3d

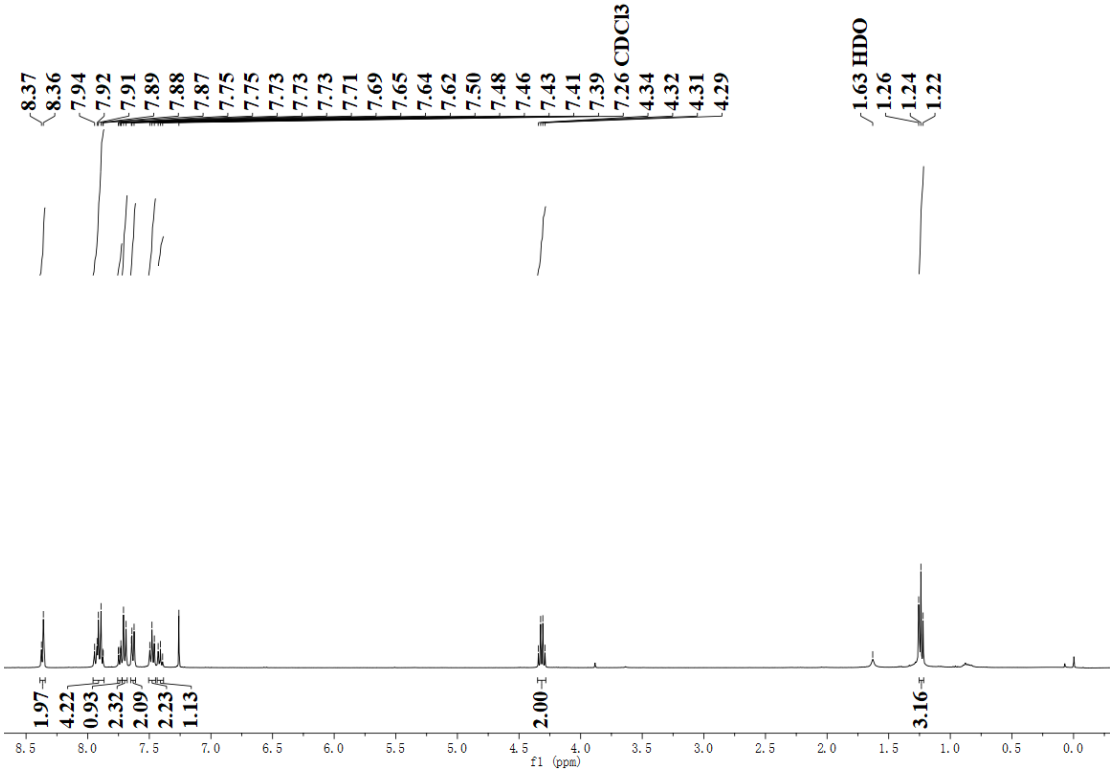


^{13}C-NMR (100MHz, $CDCl^3$) spectrum of compound 3d

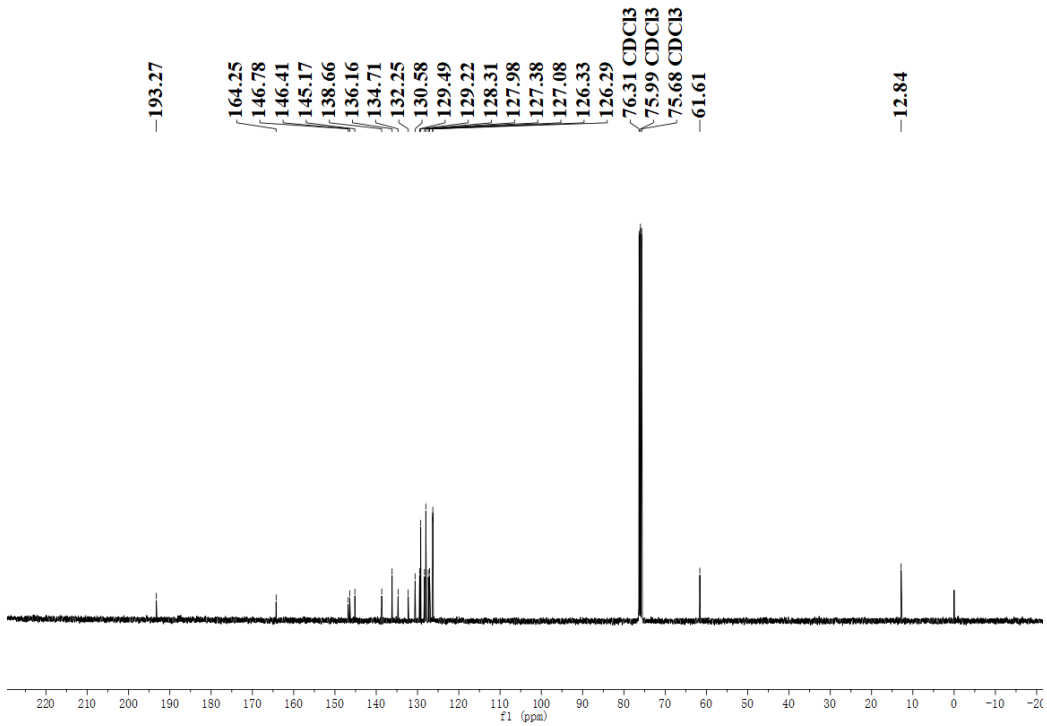


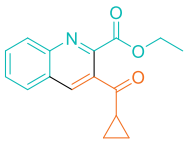


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3e

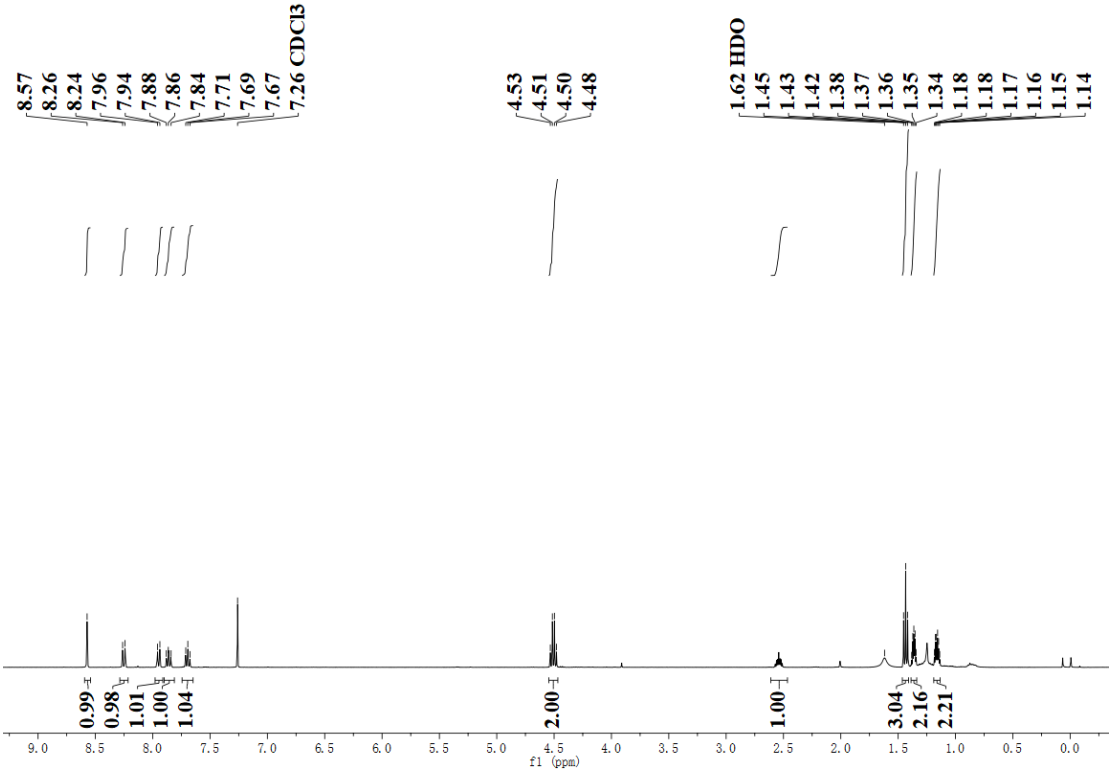


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3e

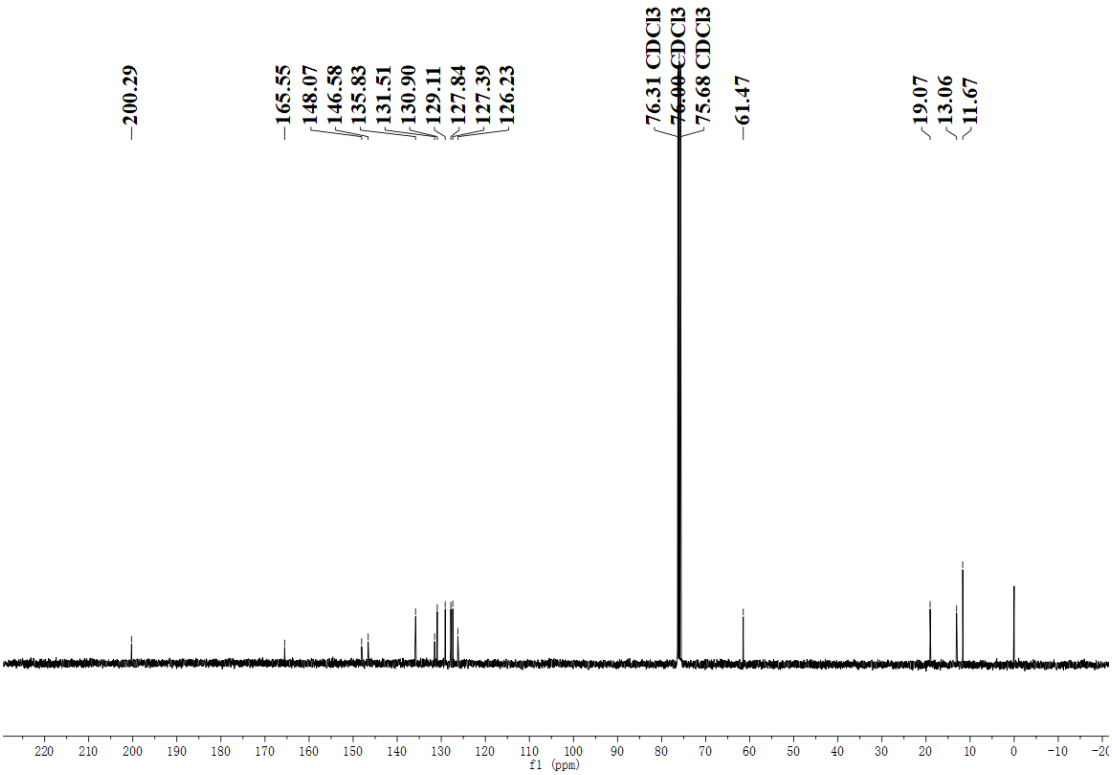


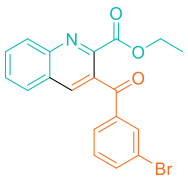


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3f

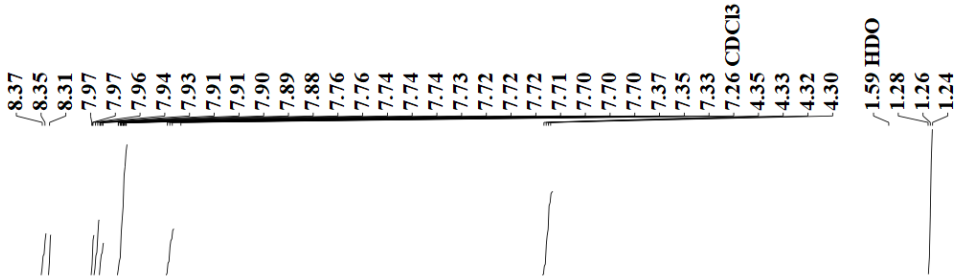


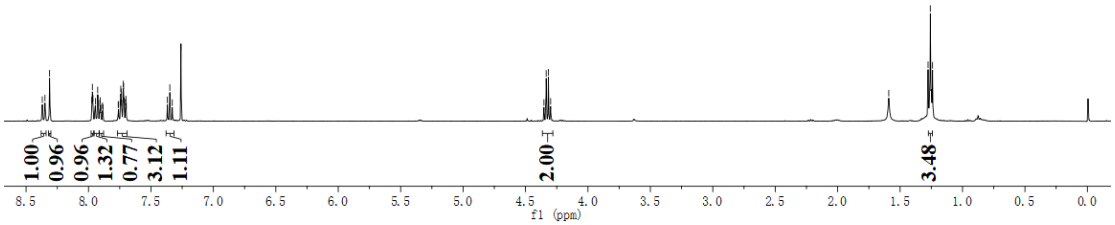
^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3f



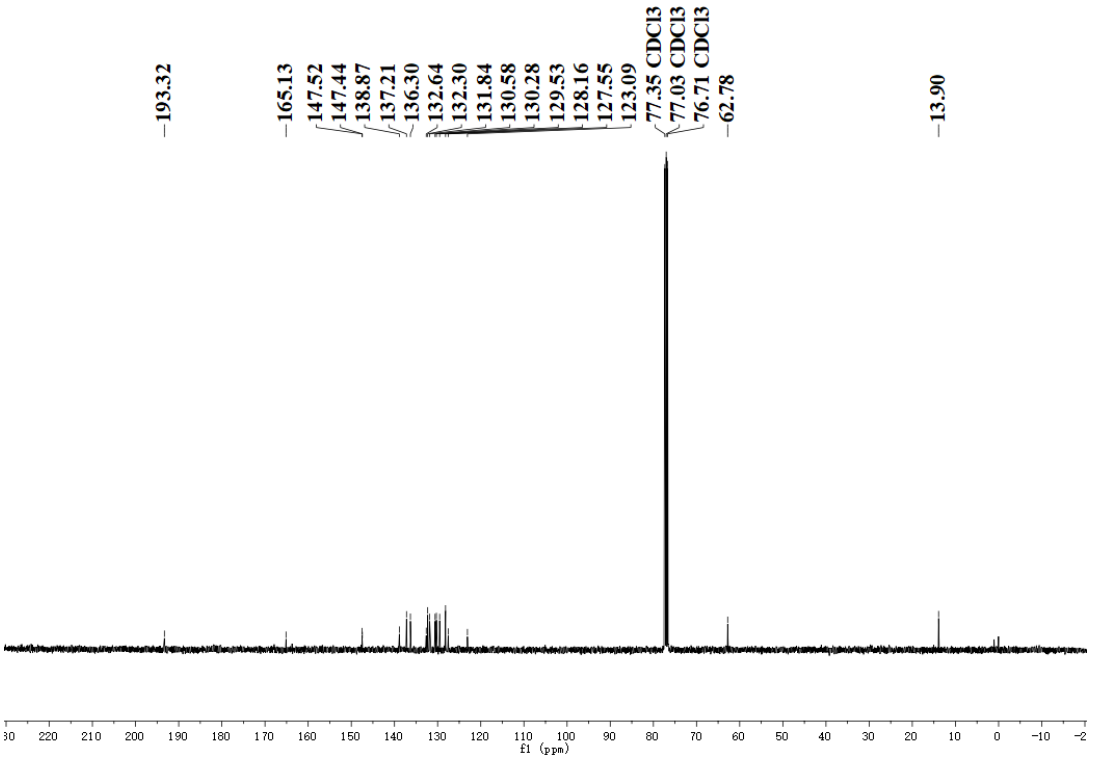


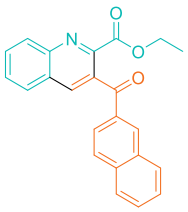
^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3g



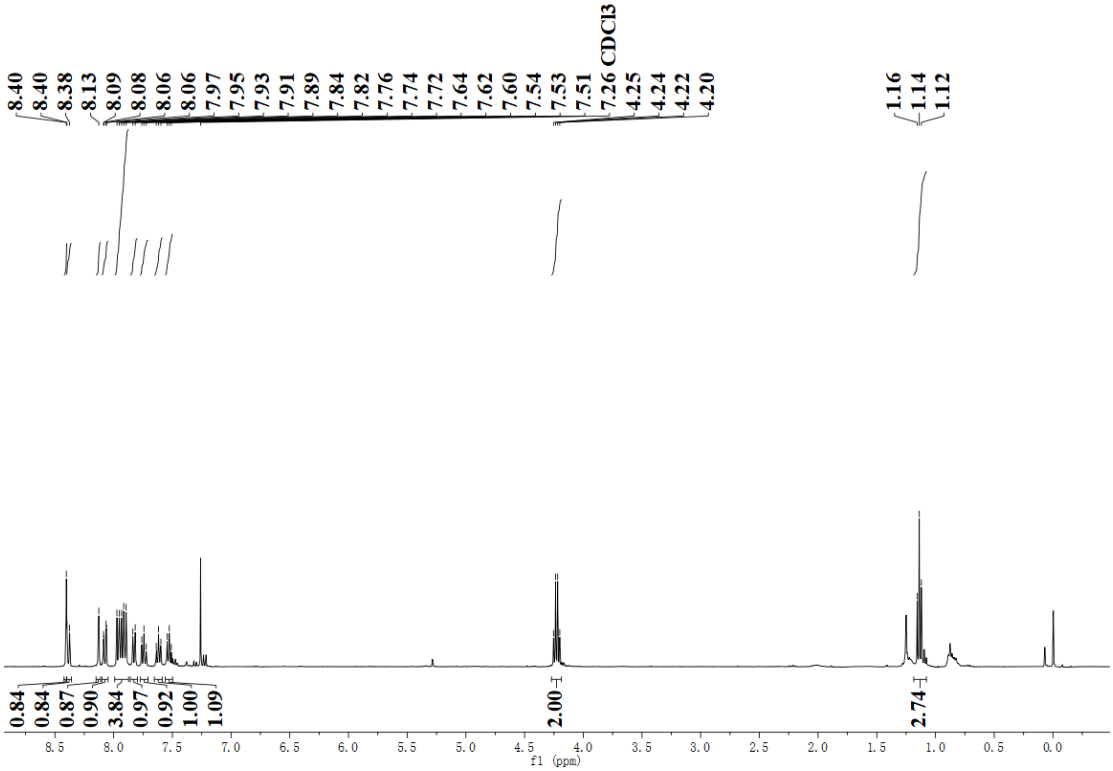


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3g

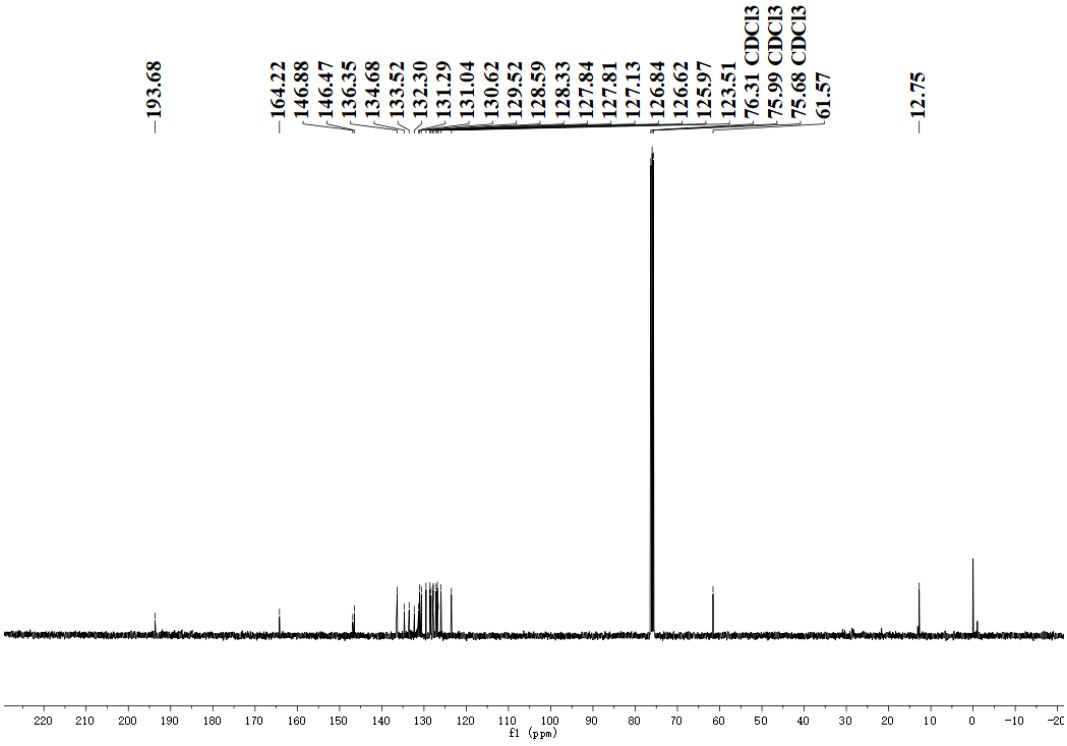


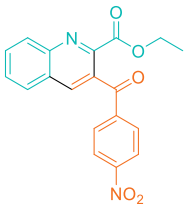


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3h

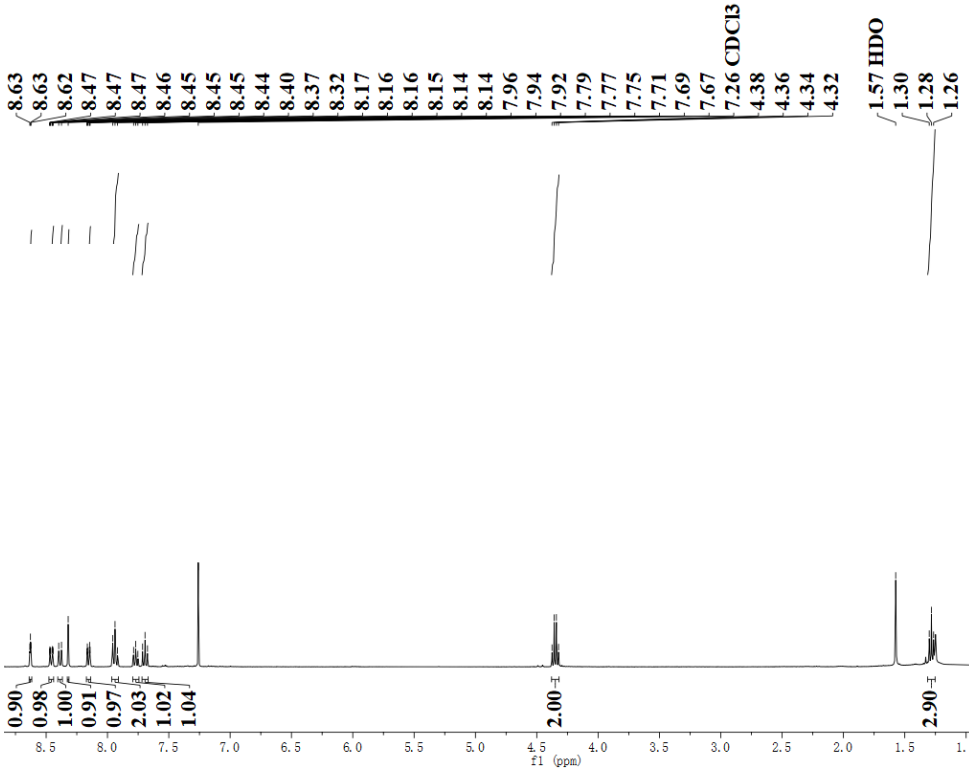


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3h

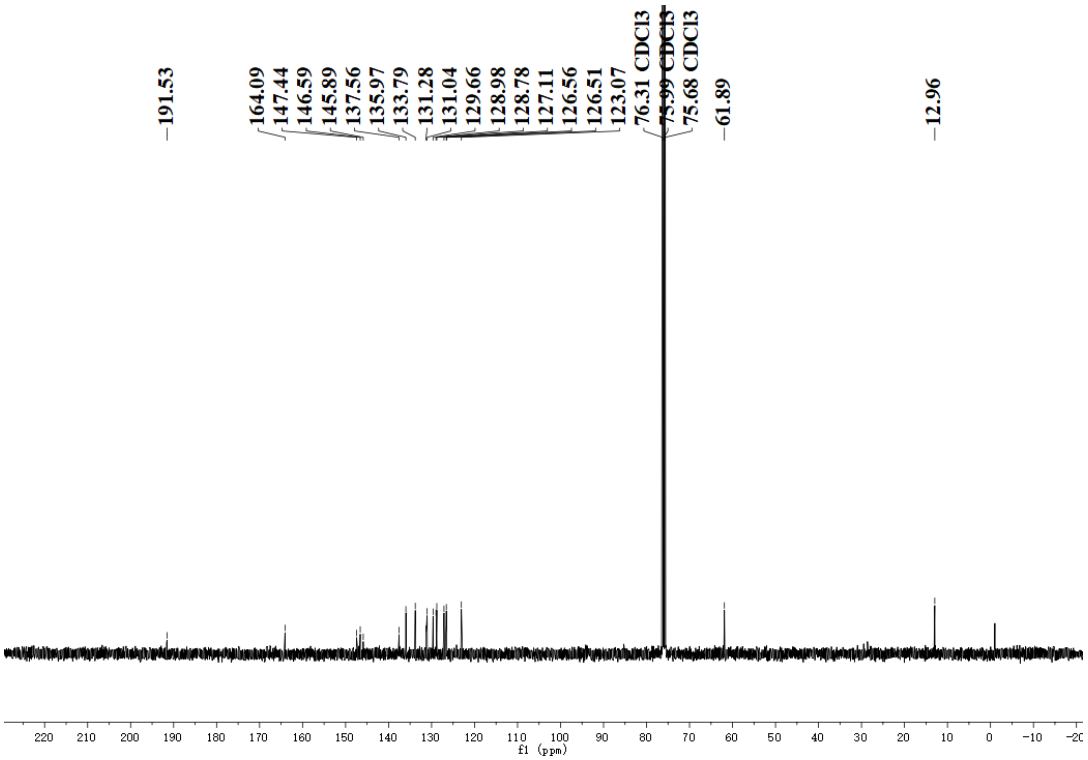


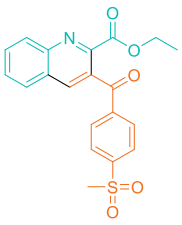


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3i

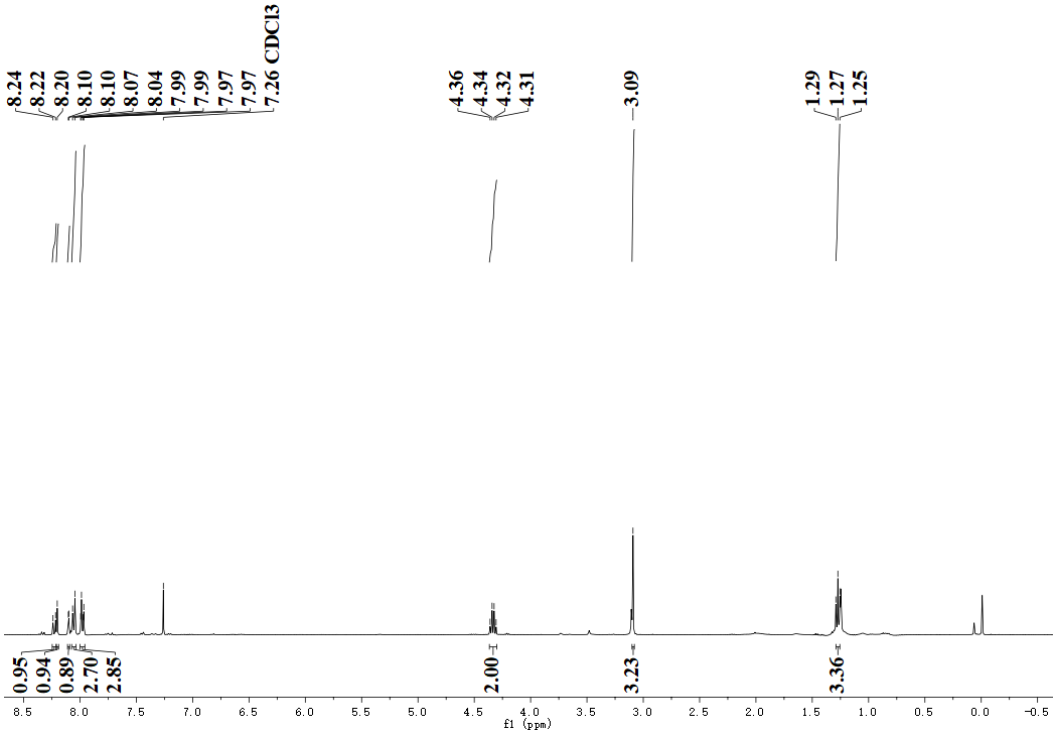


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3i

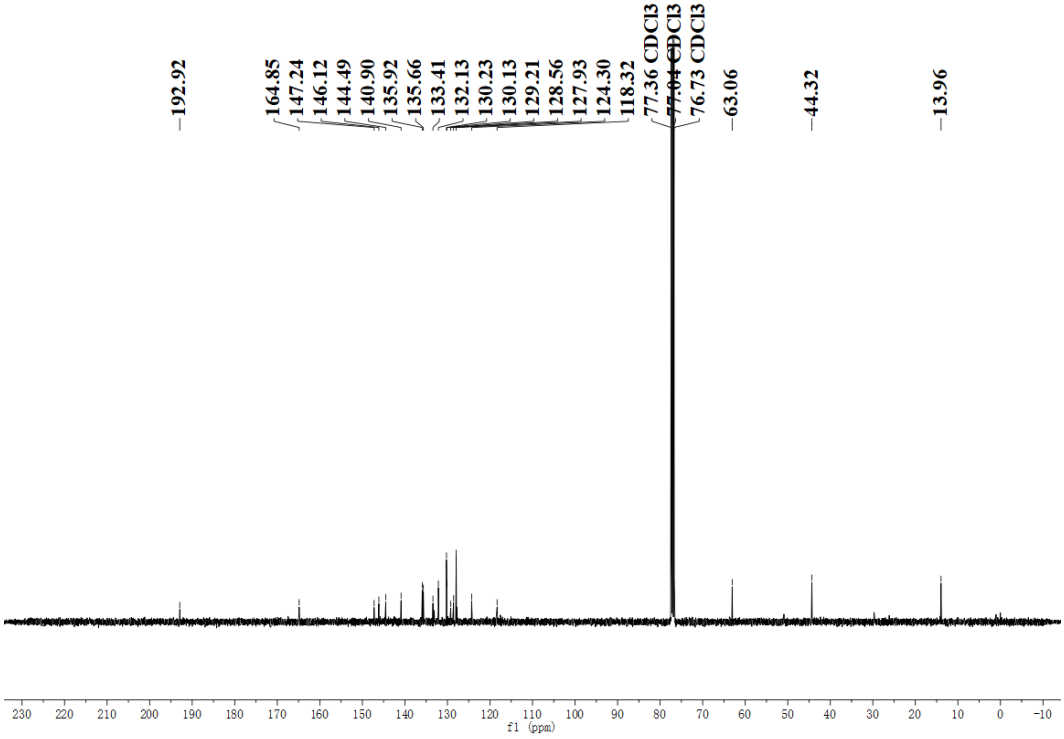


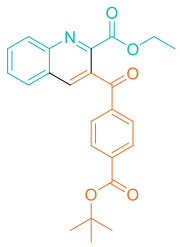


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3j

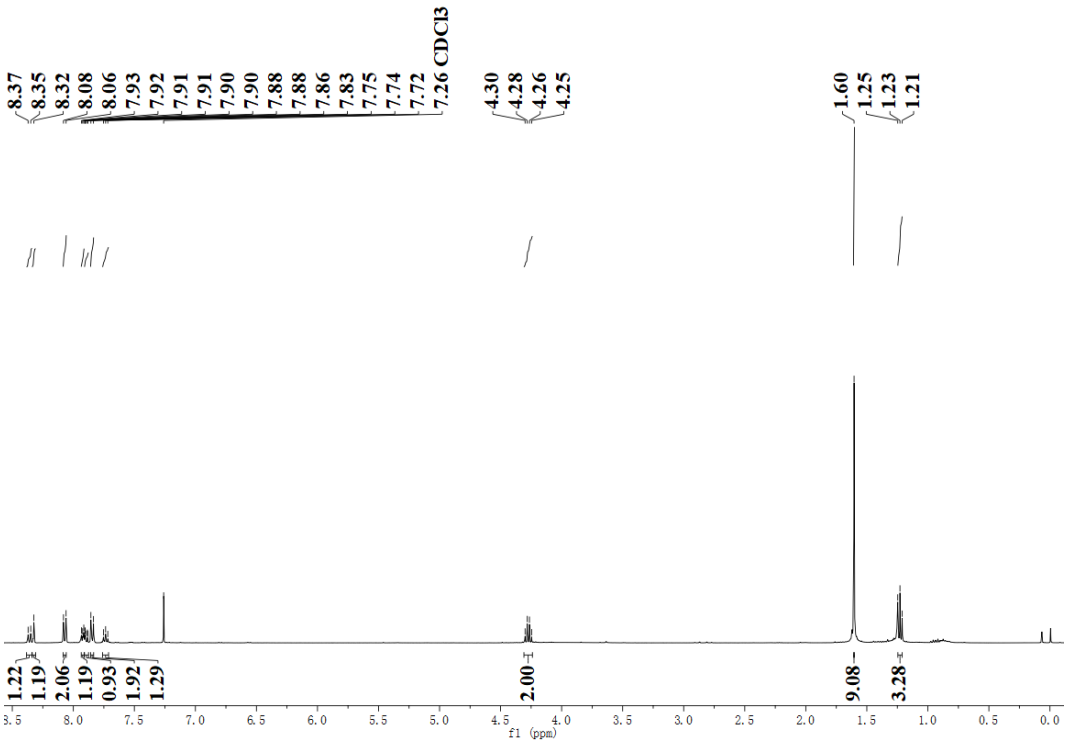


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3j

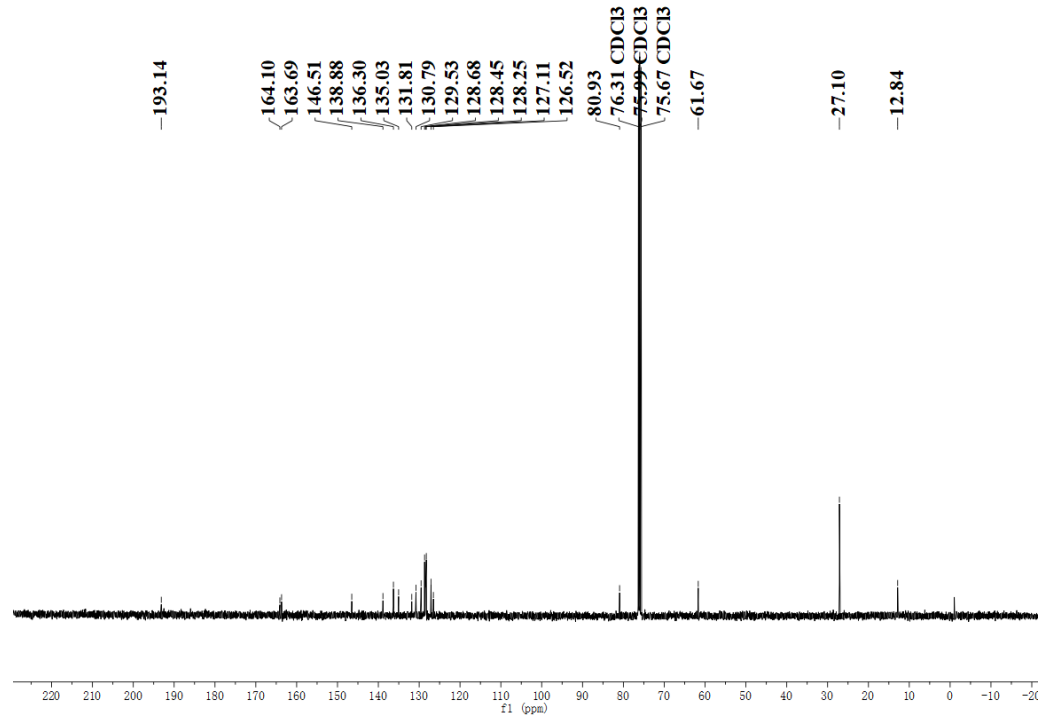


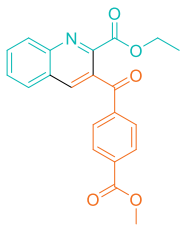


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3k

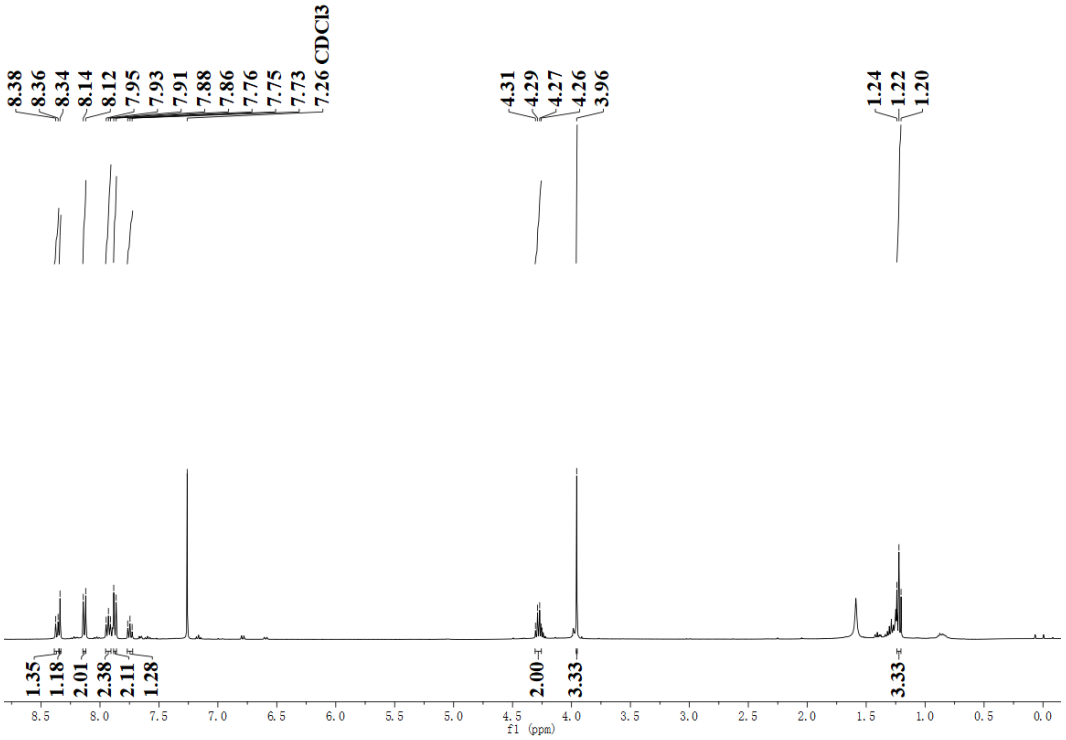


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3k

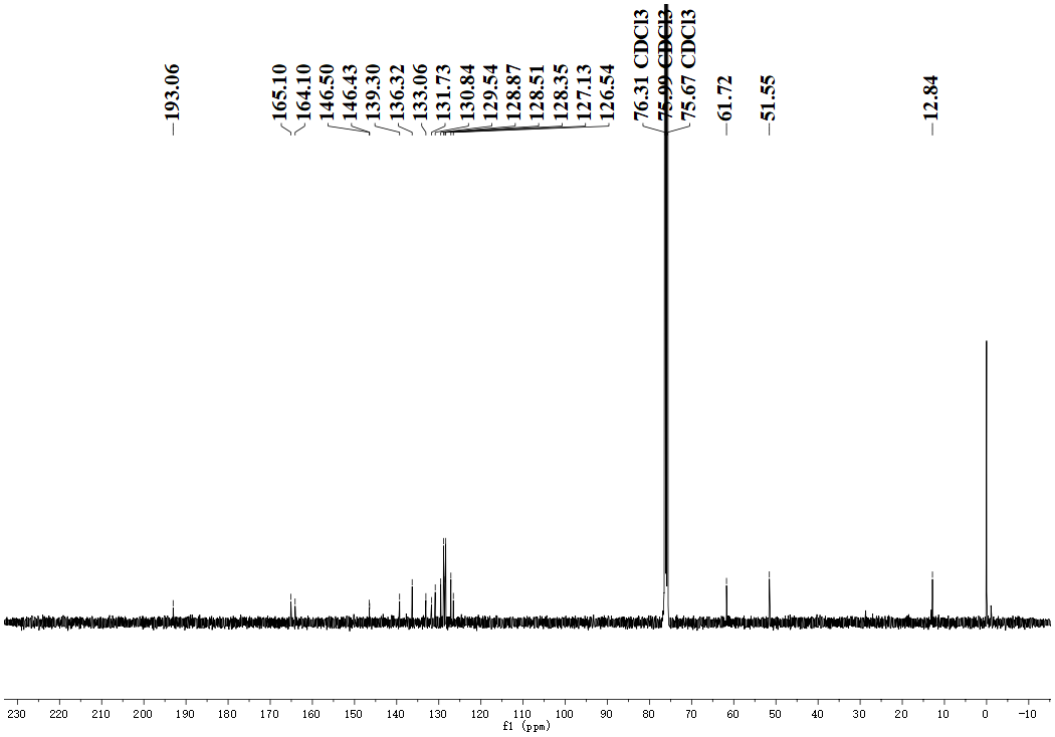


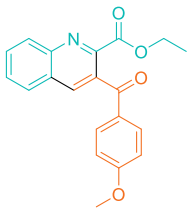


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3l

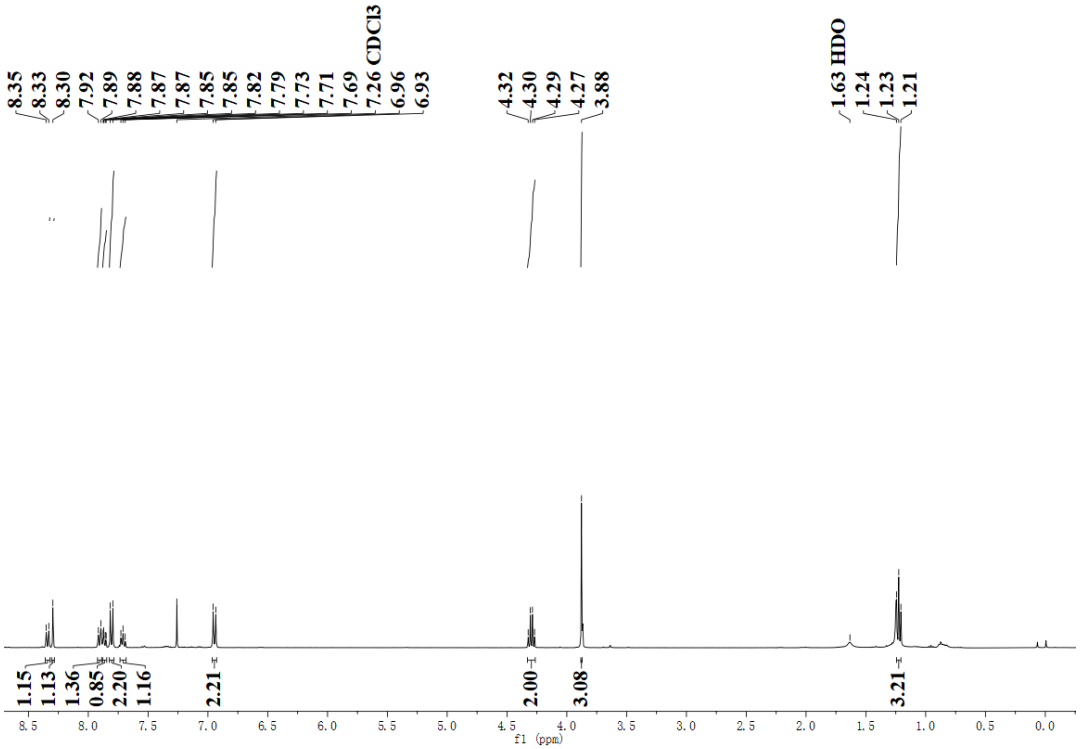


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3l

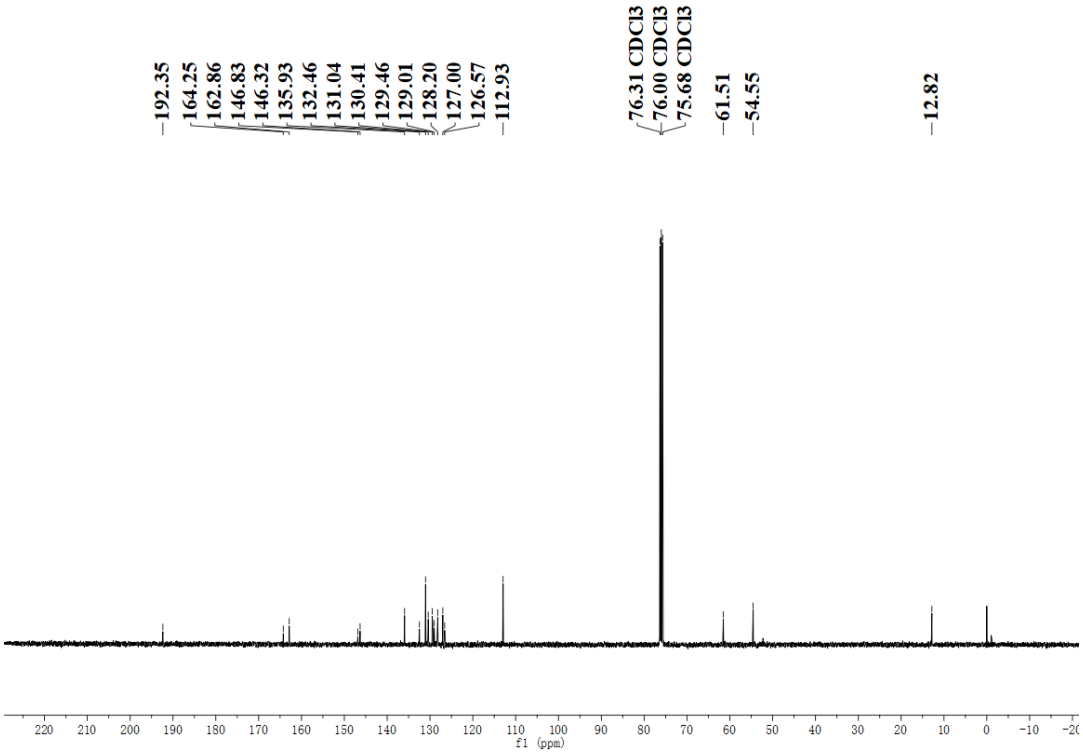


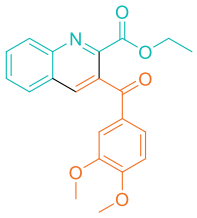


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3m

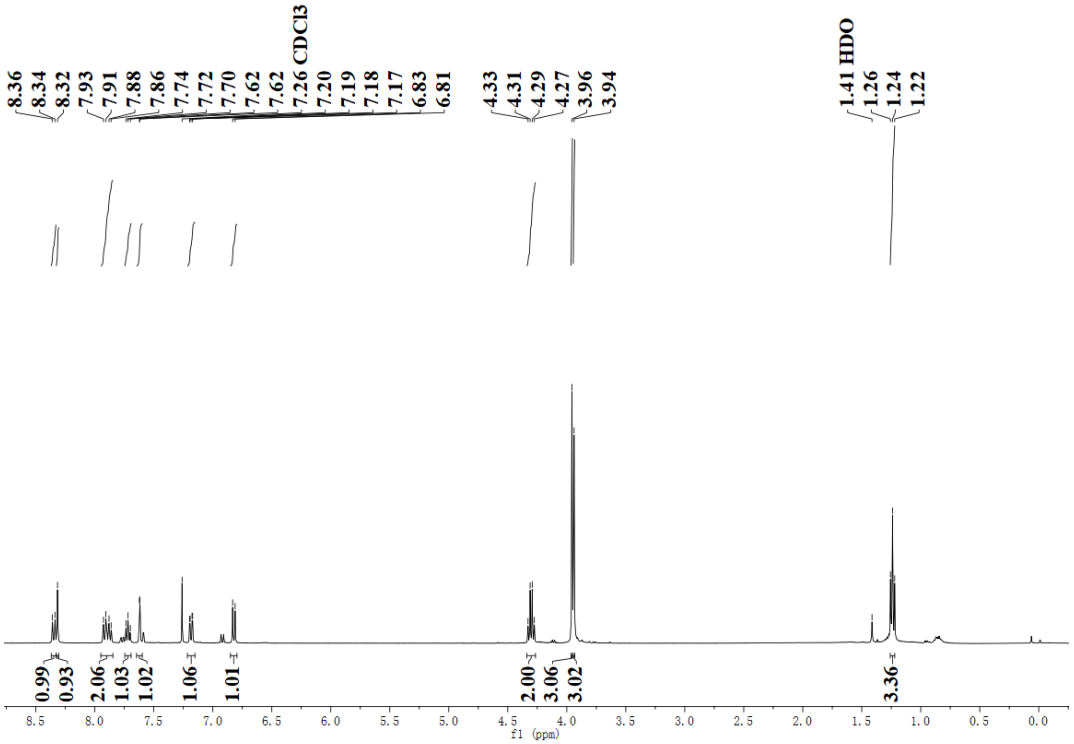


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3m

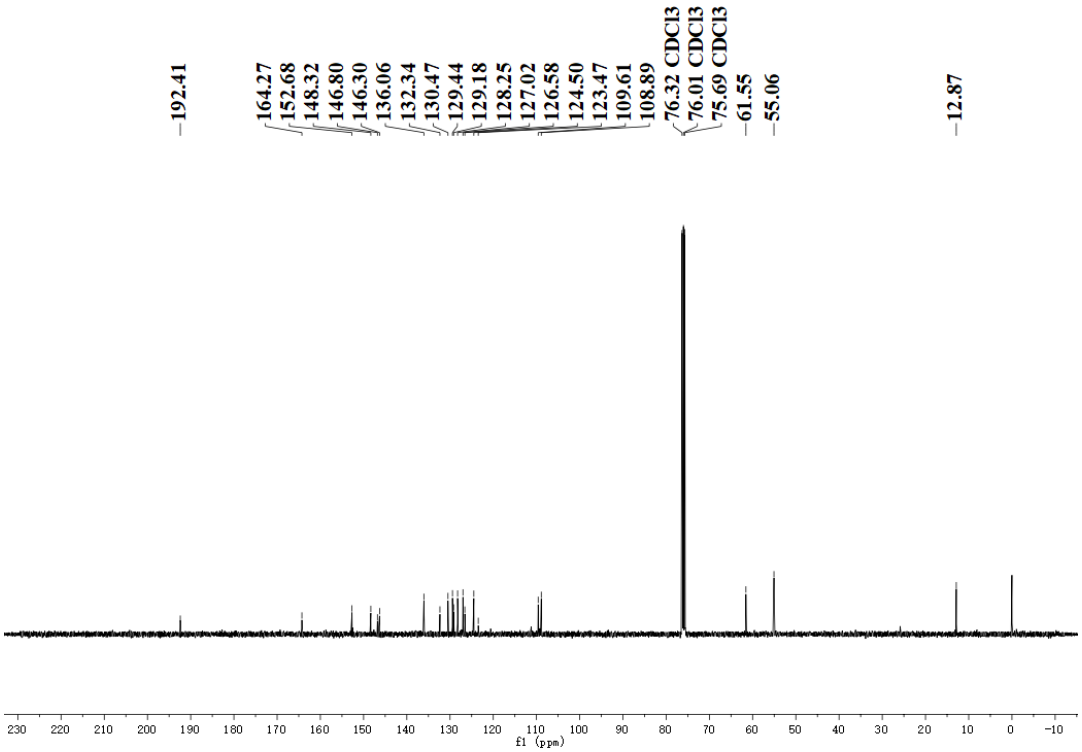


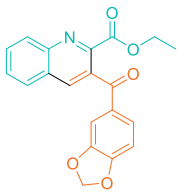


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3n

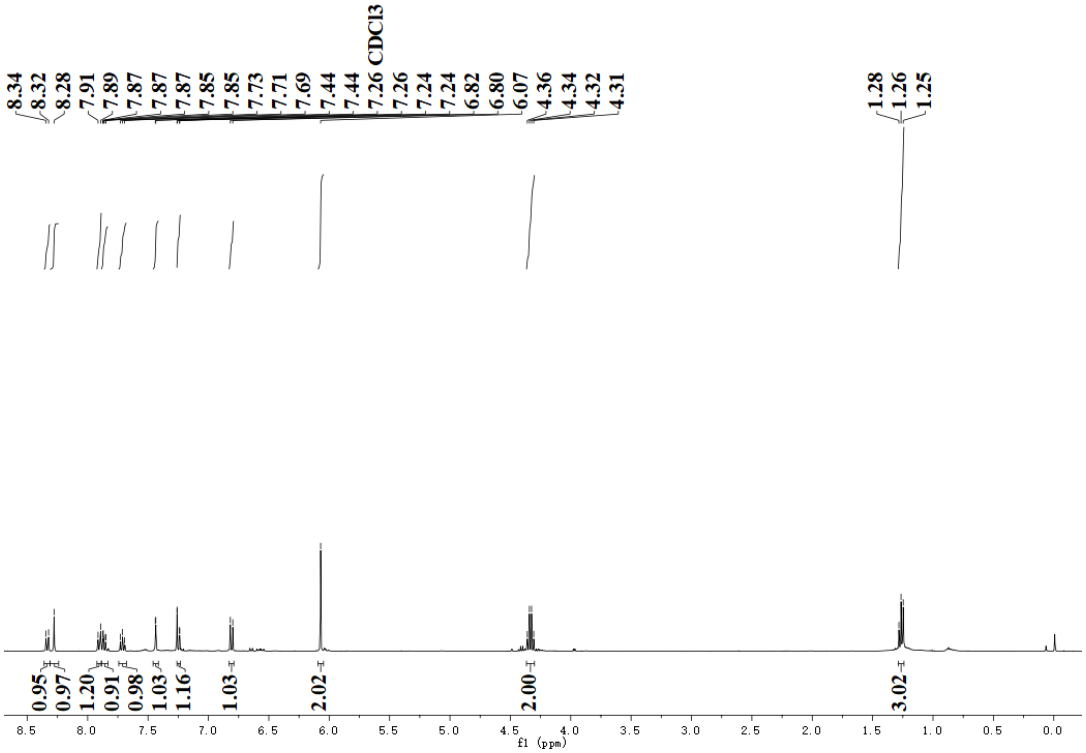


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3n

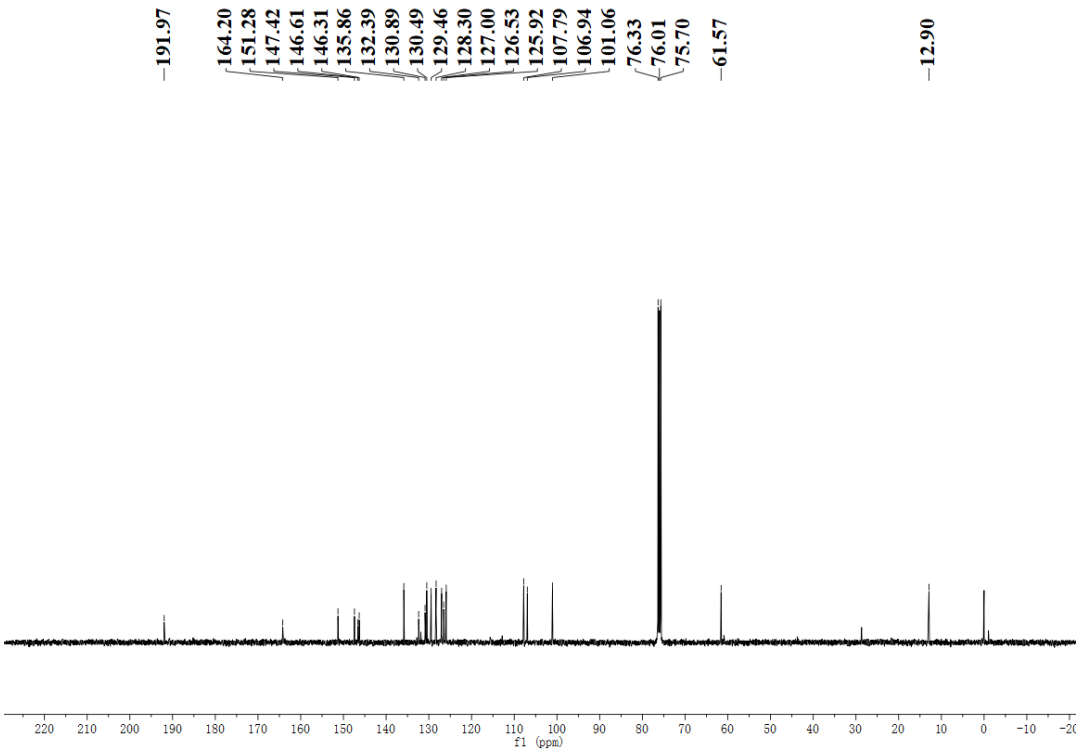


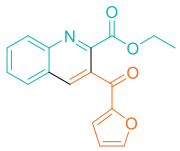


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3o

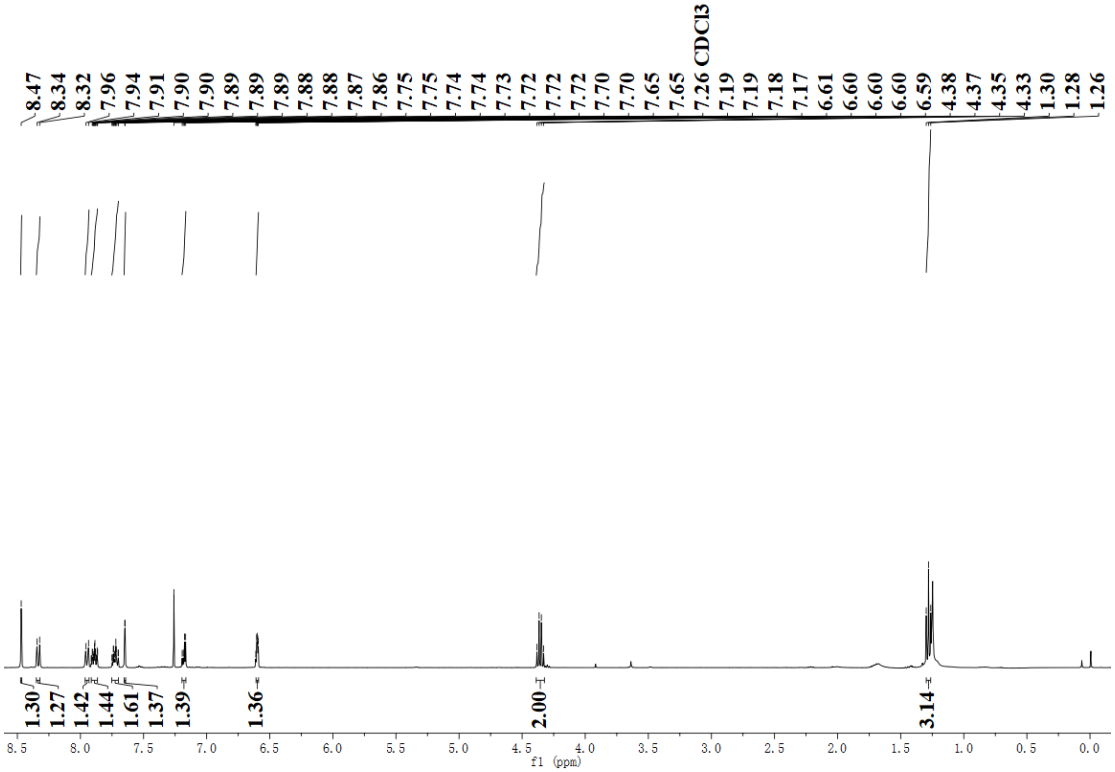


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3o

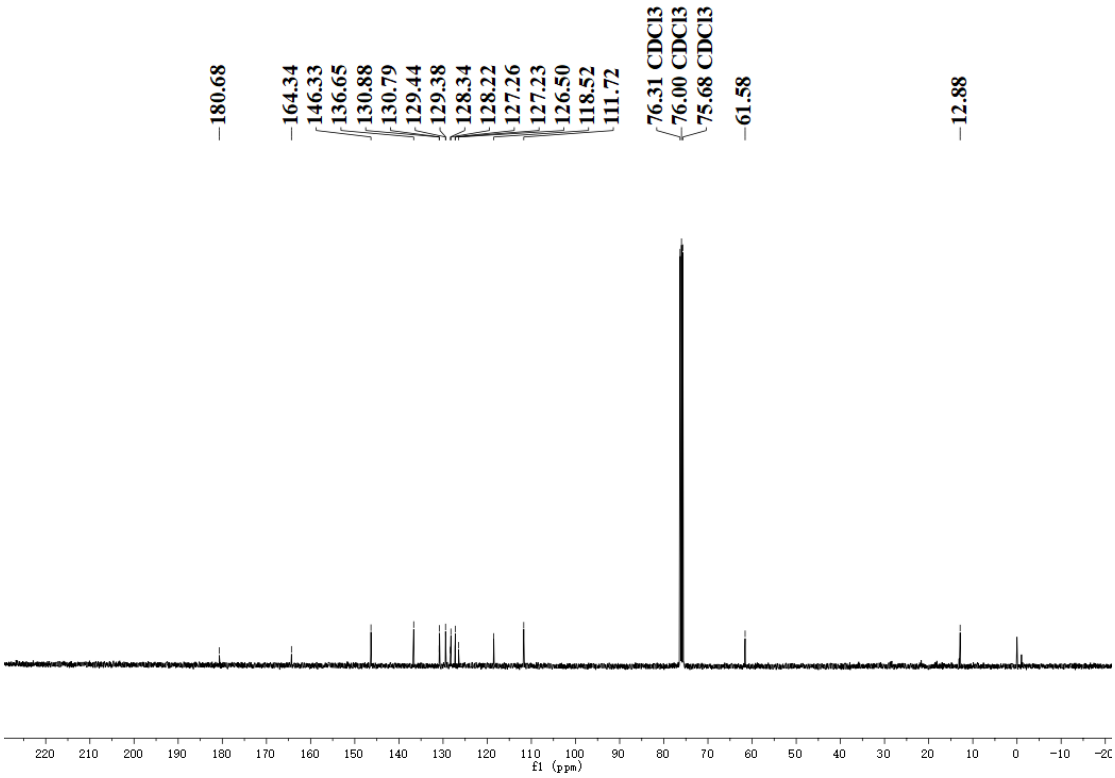


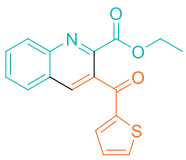


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3p

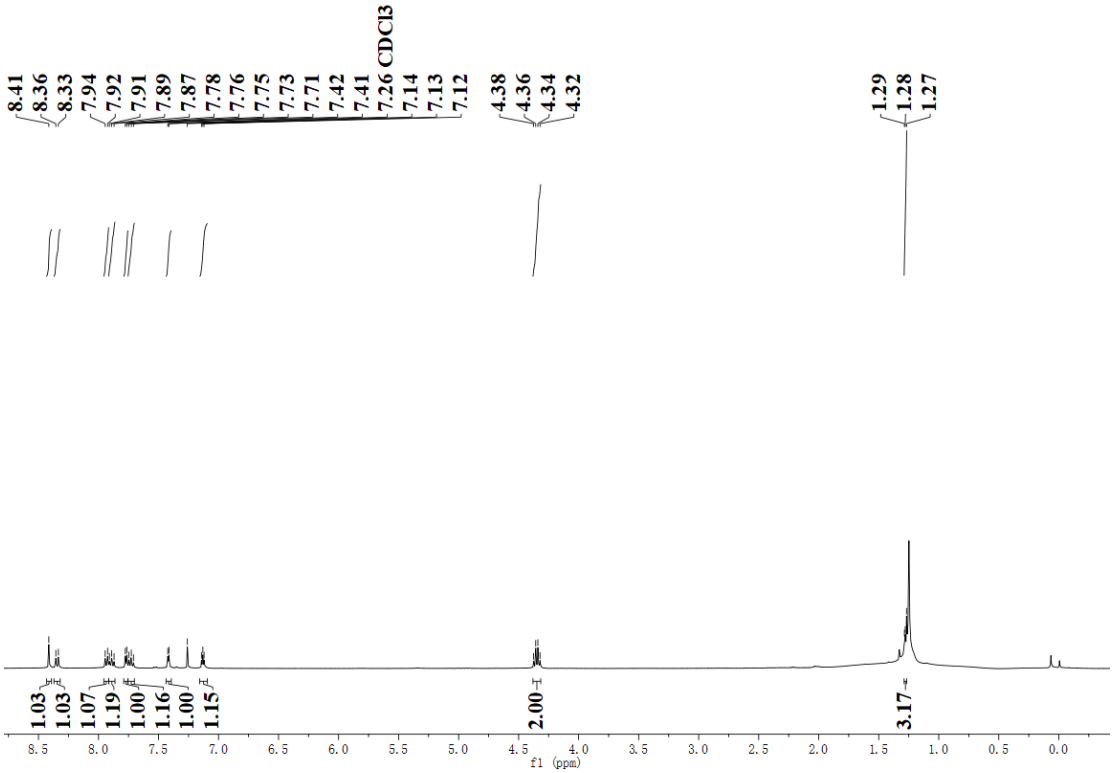


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3p

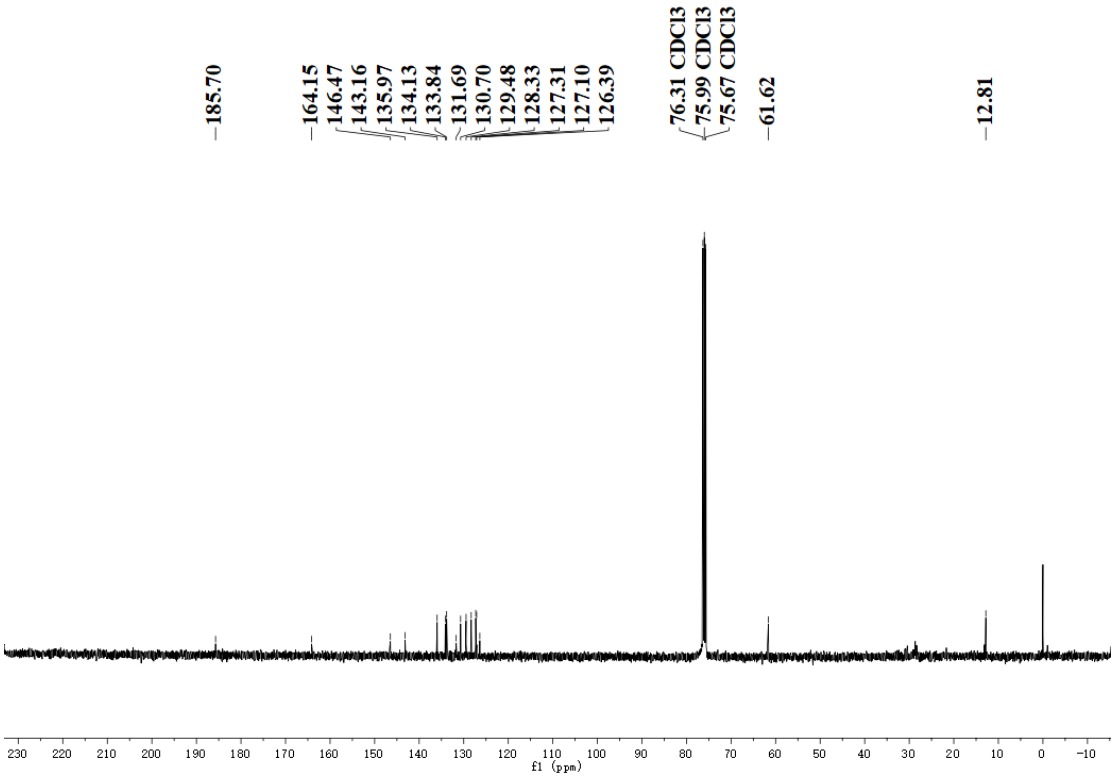


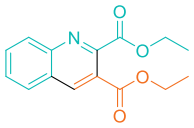


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3q

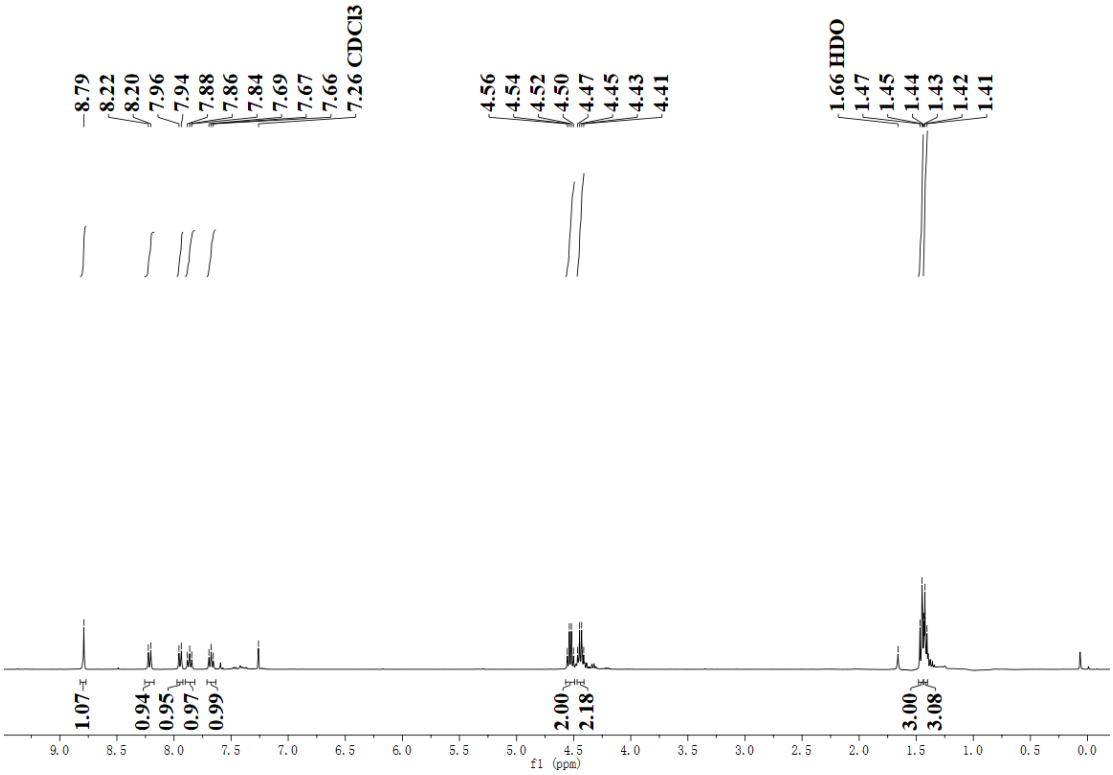


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3q

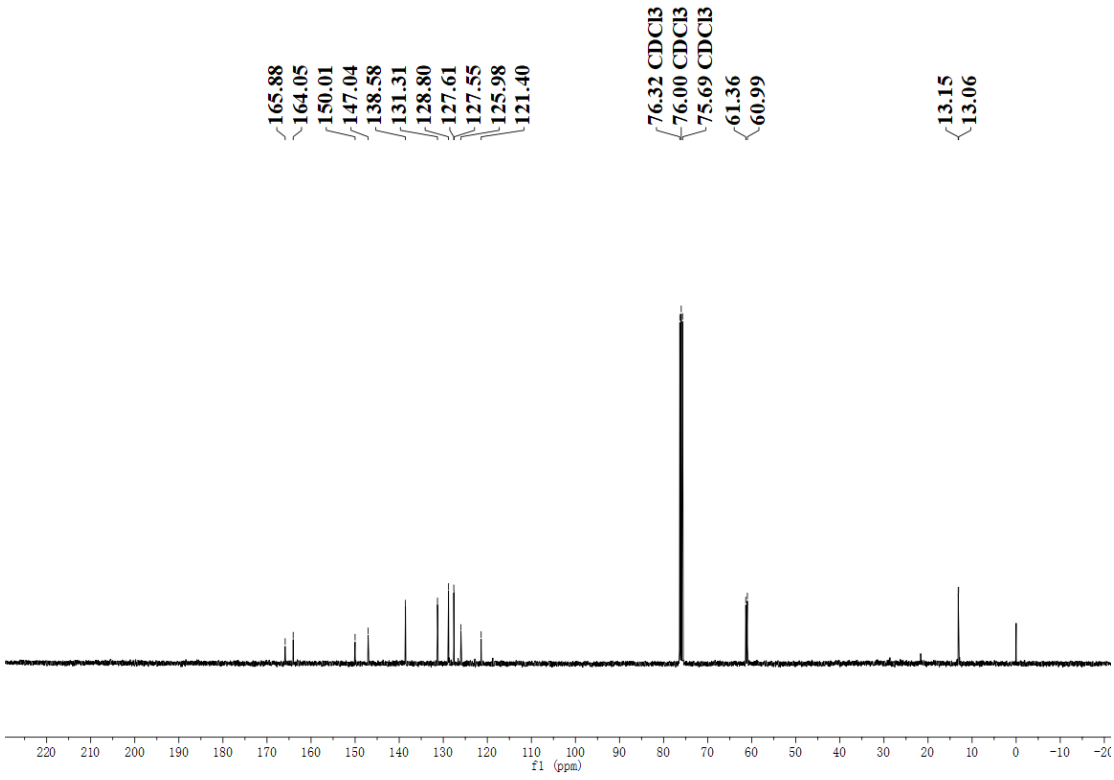


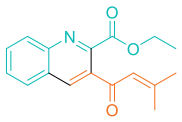


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3r

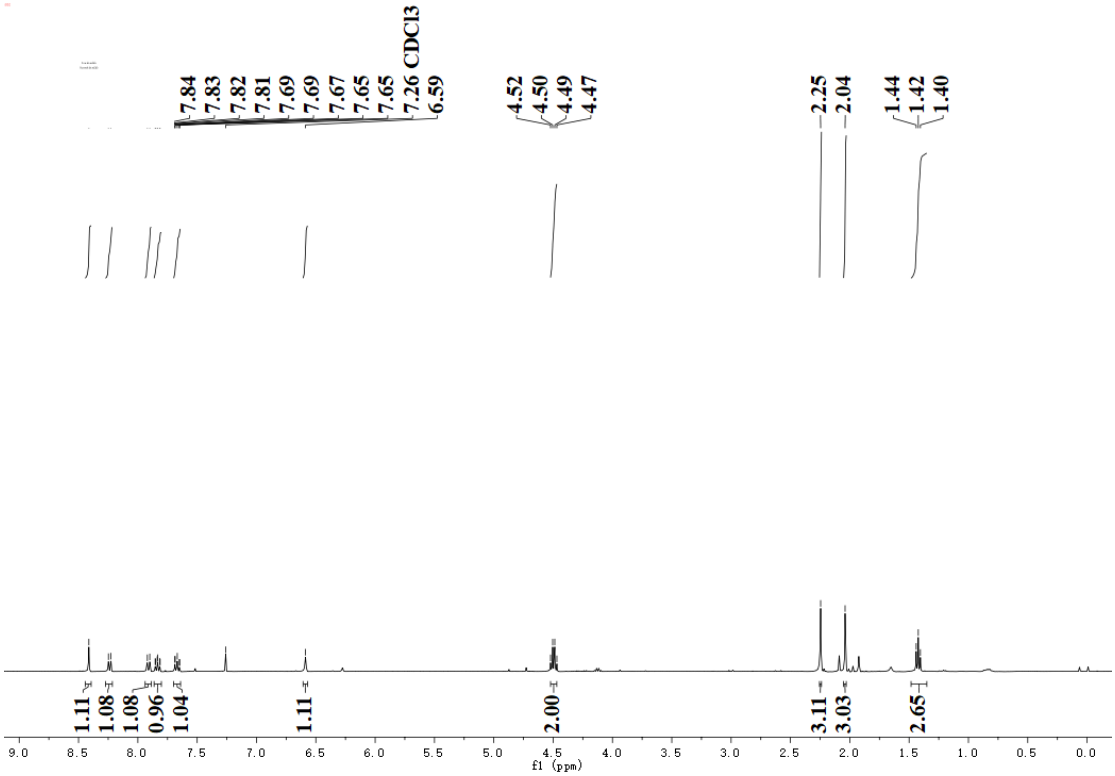


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3r

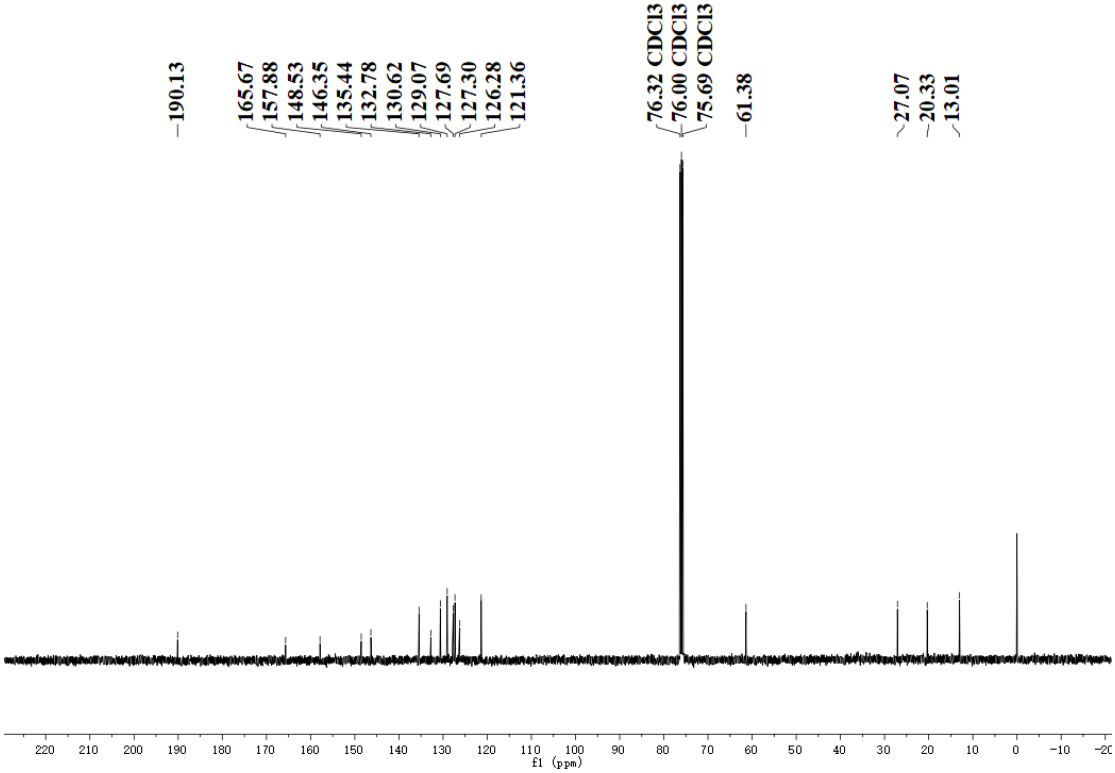


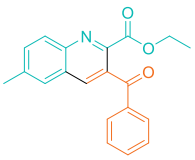


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 3s

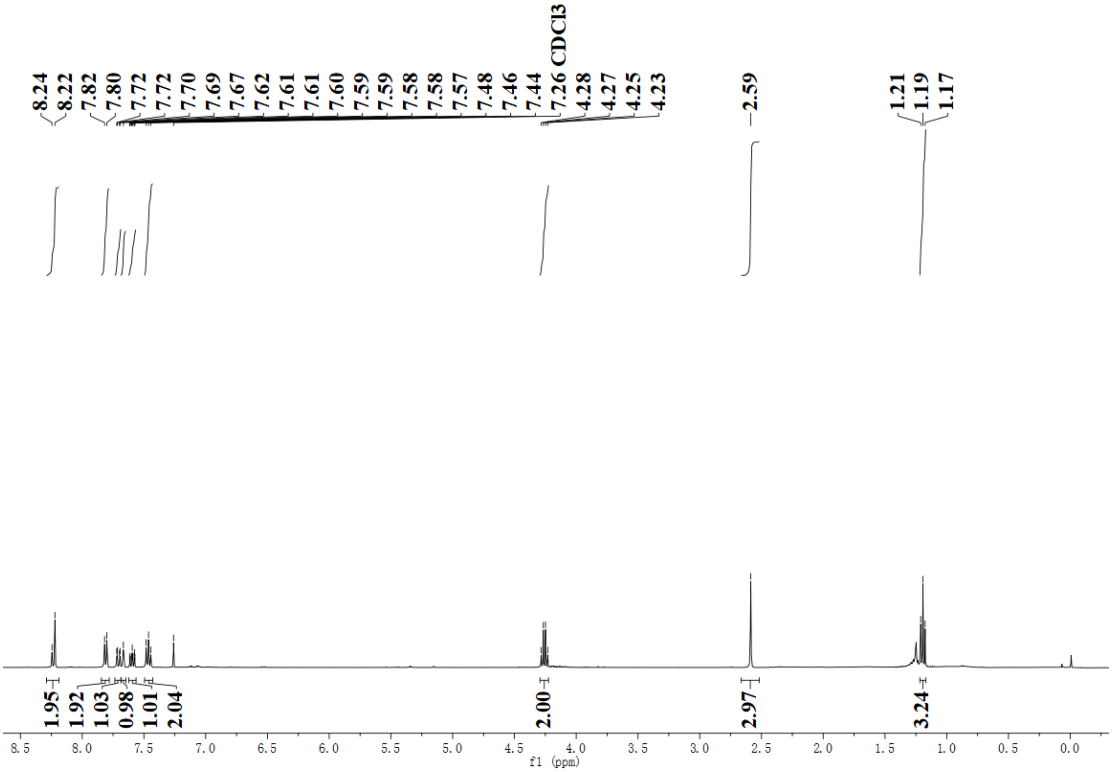


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 3s

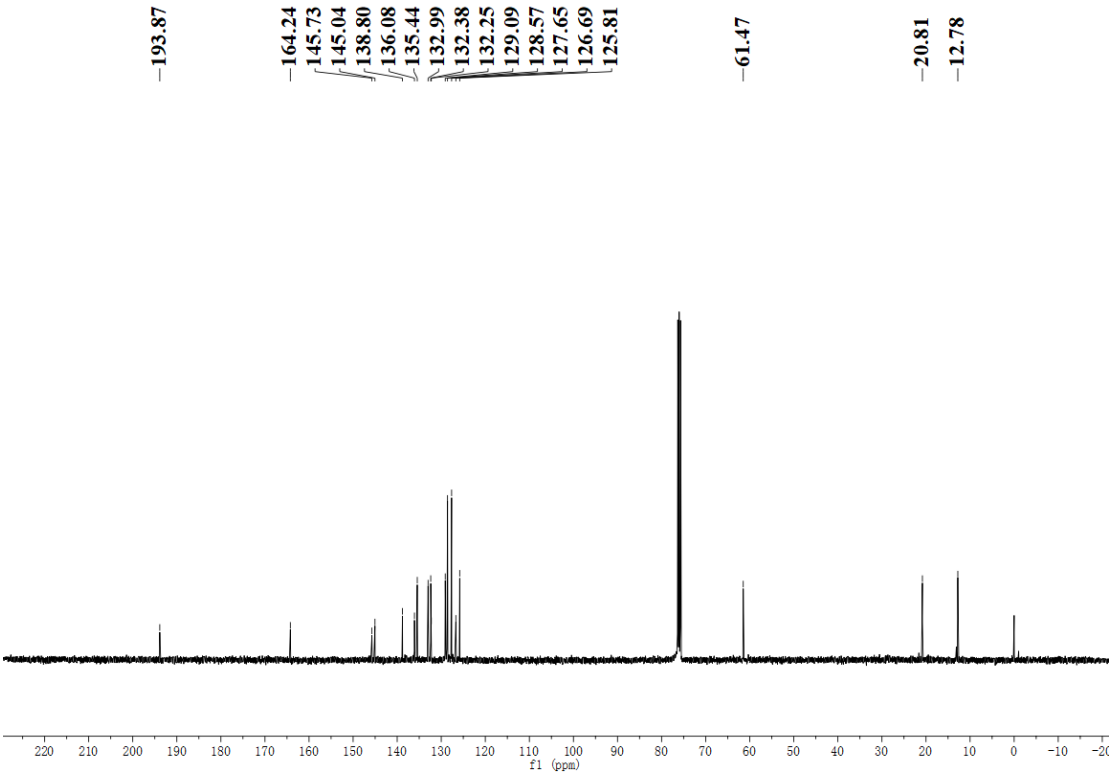


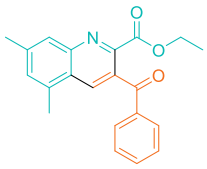


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4a

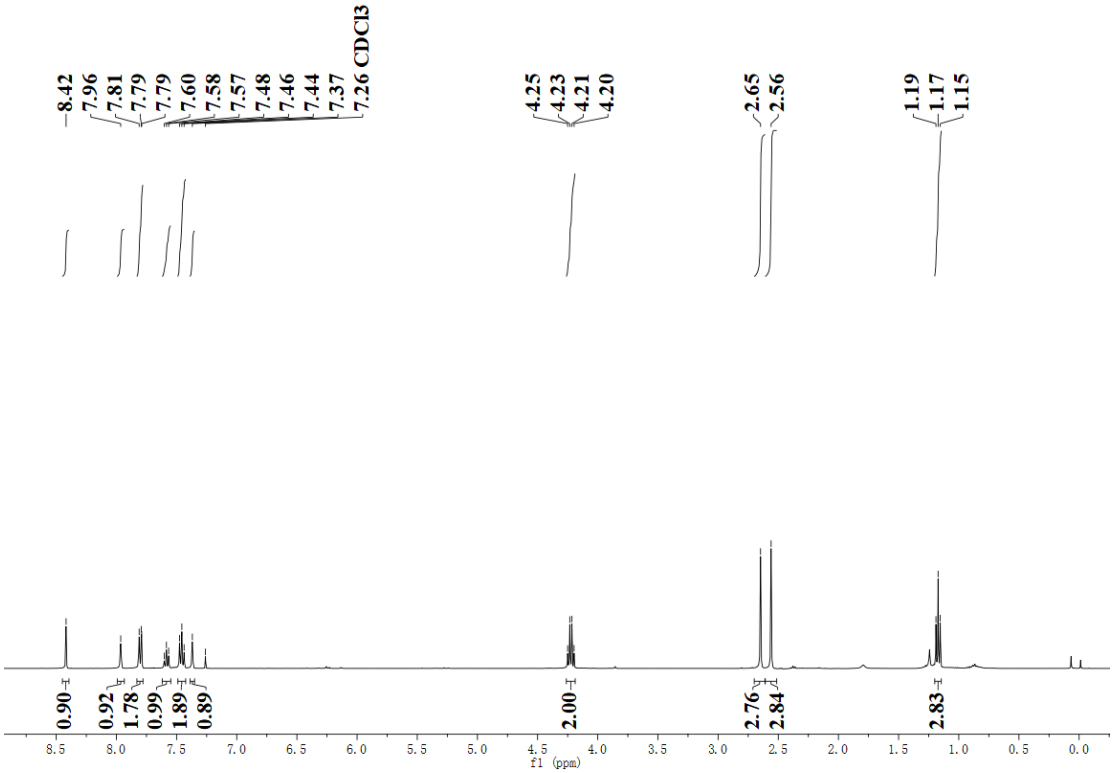


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4a

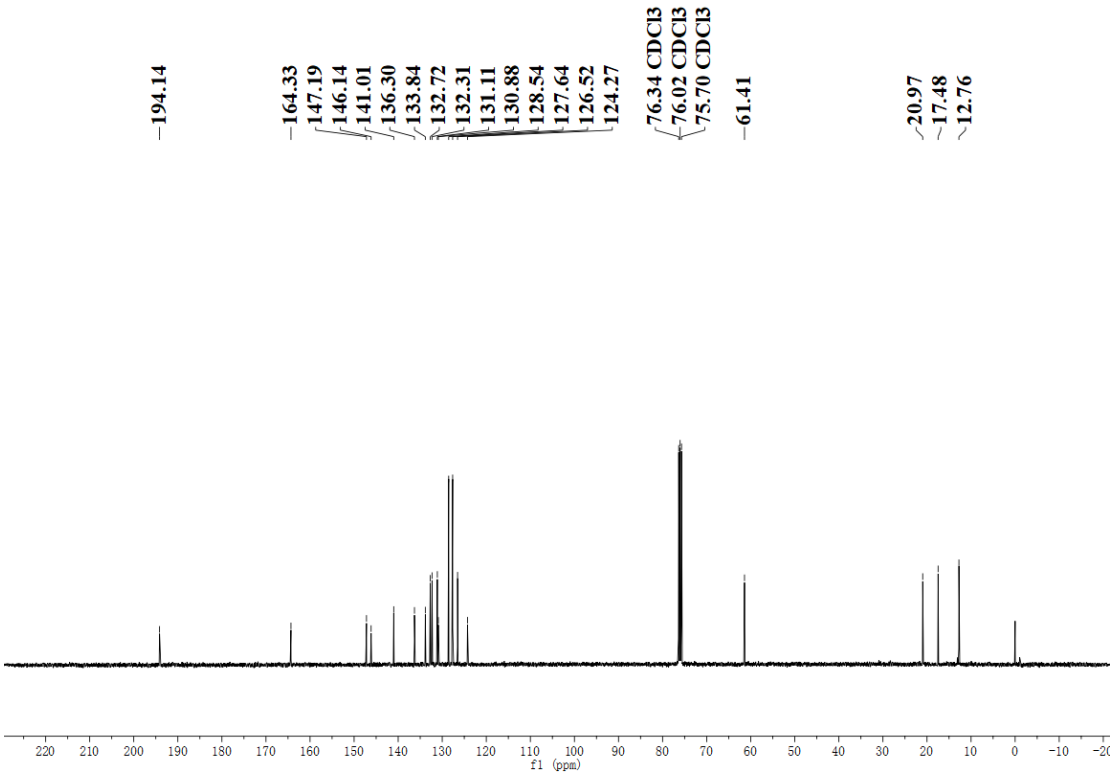


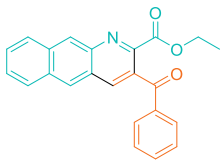


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4b

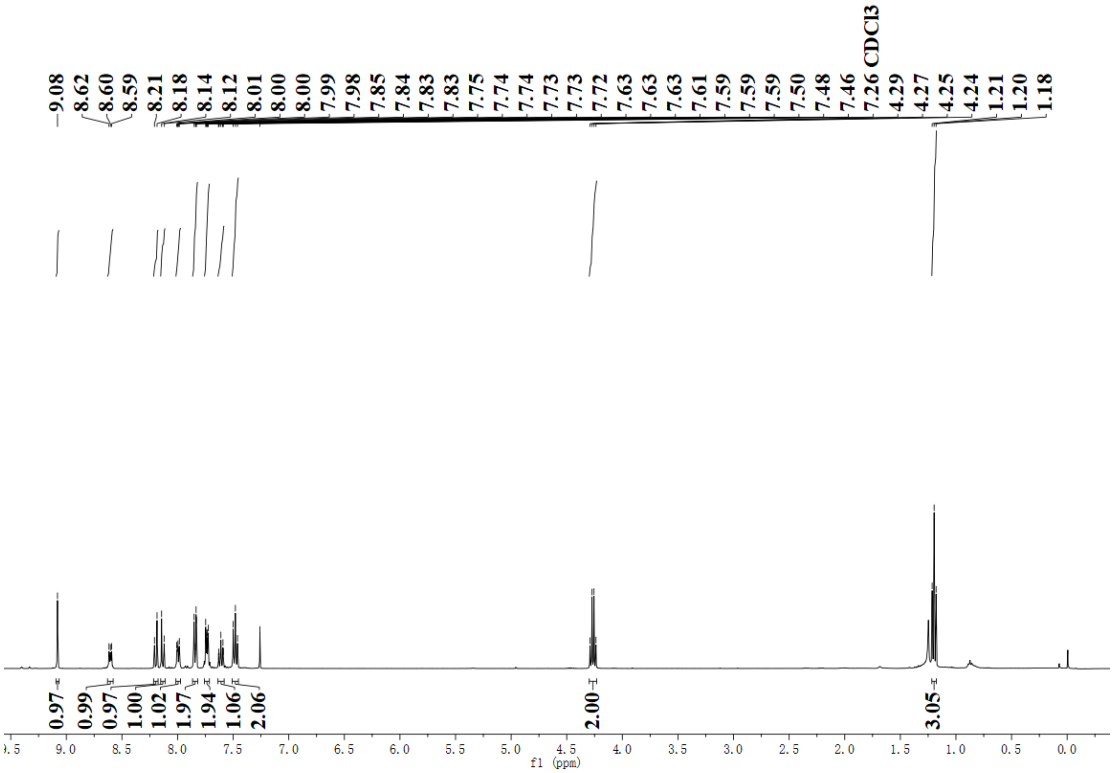


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4b

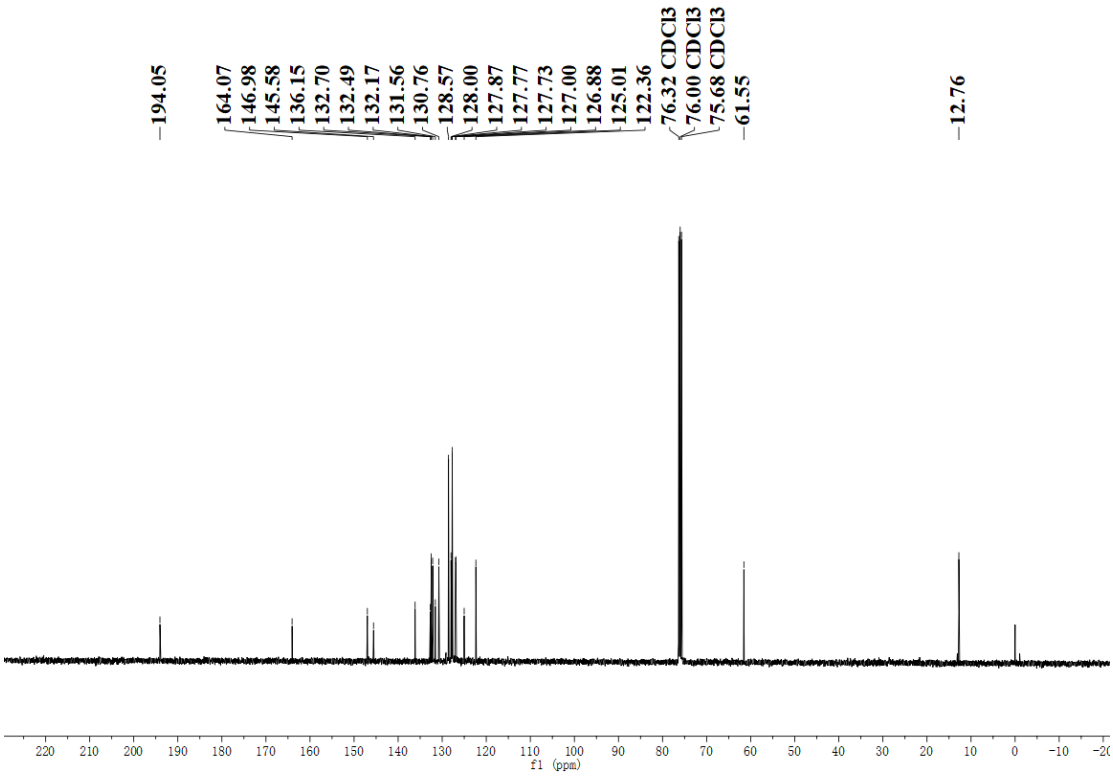


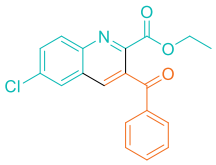


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4c

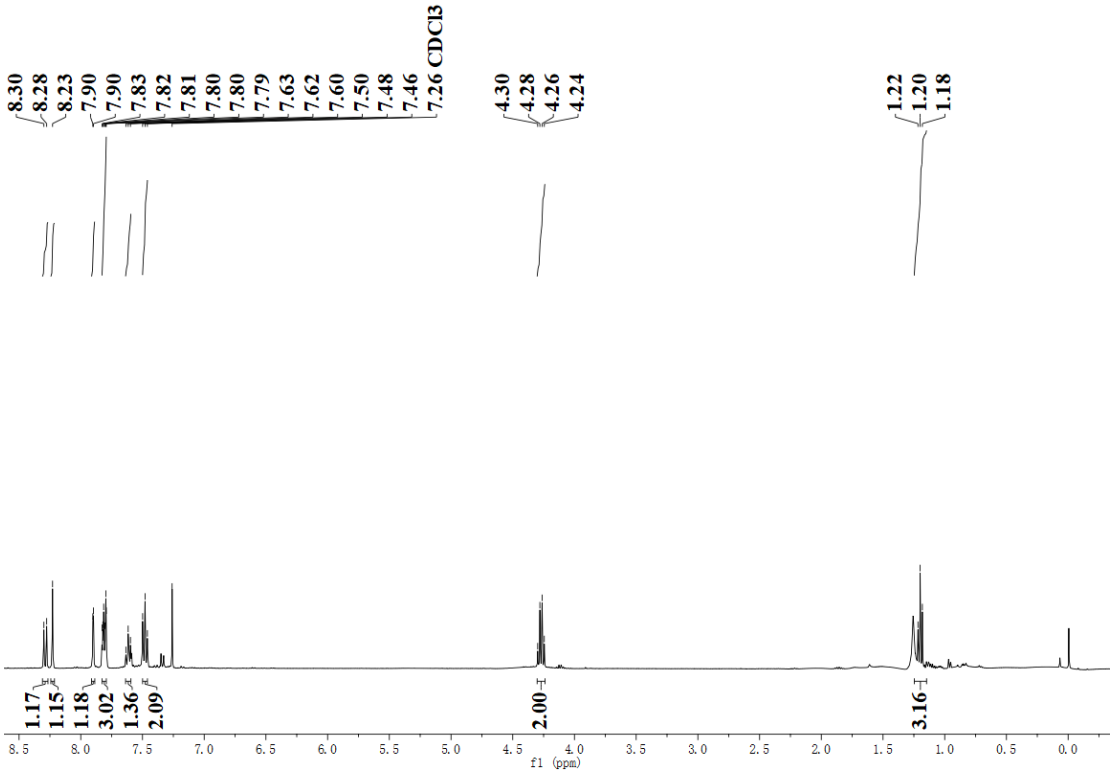


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4c

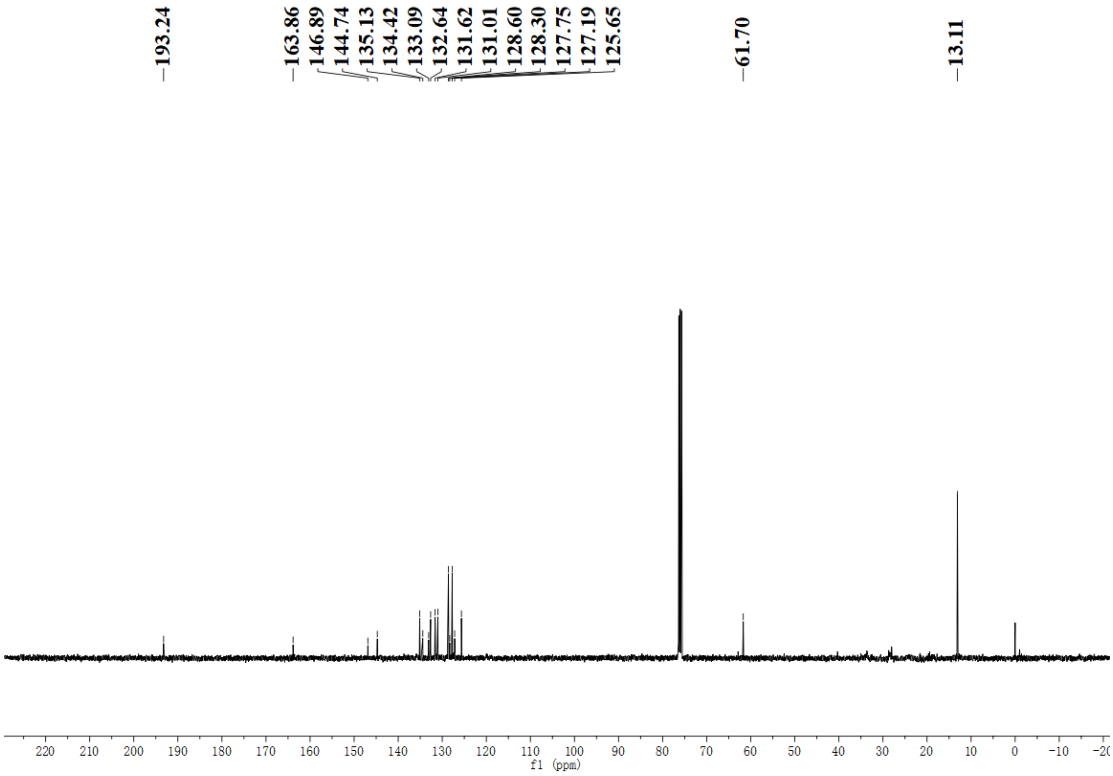


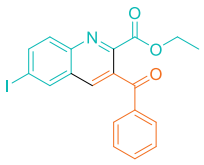


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4d

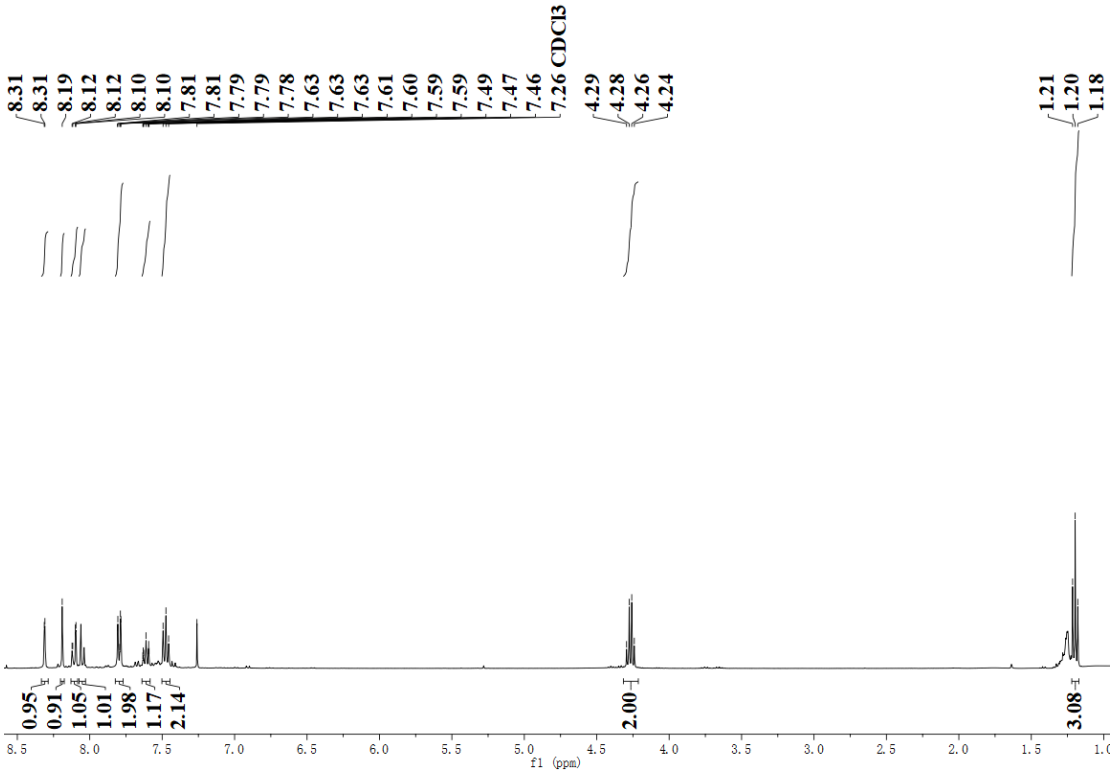


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4d

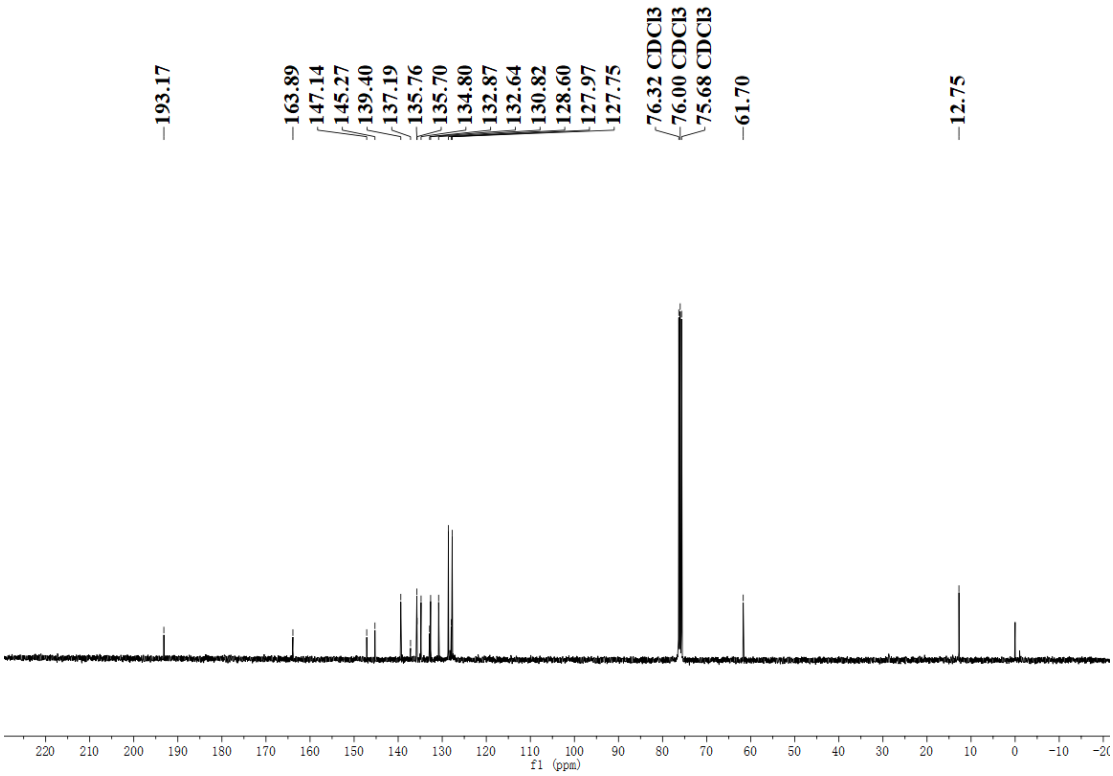


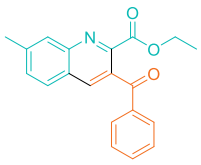


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4e

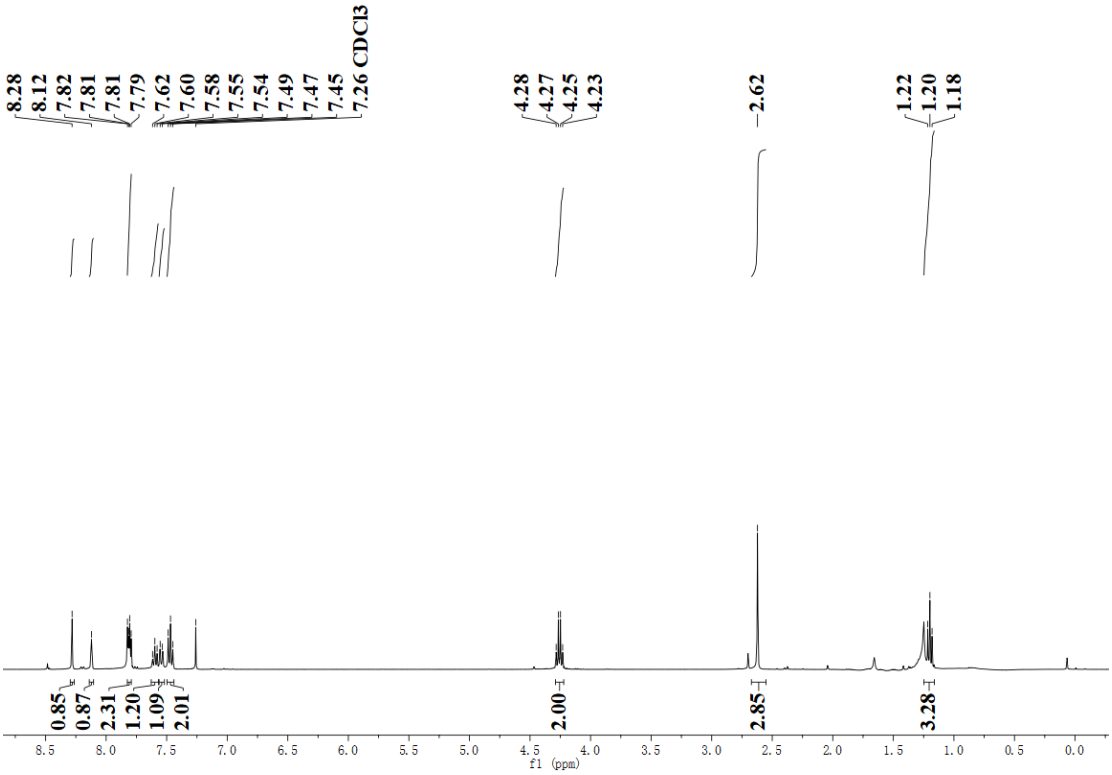


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4e

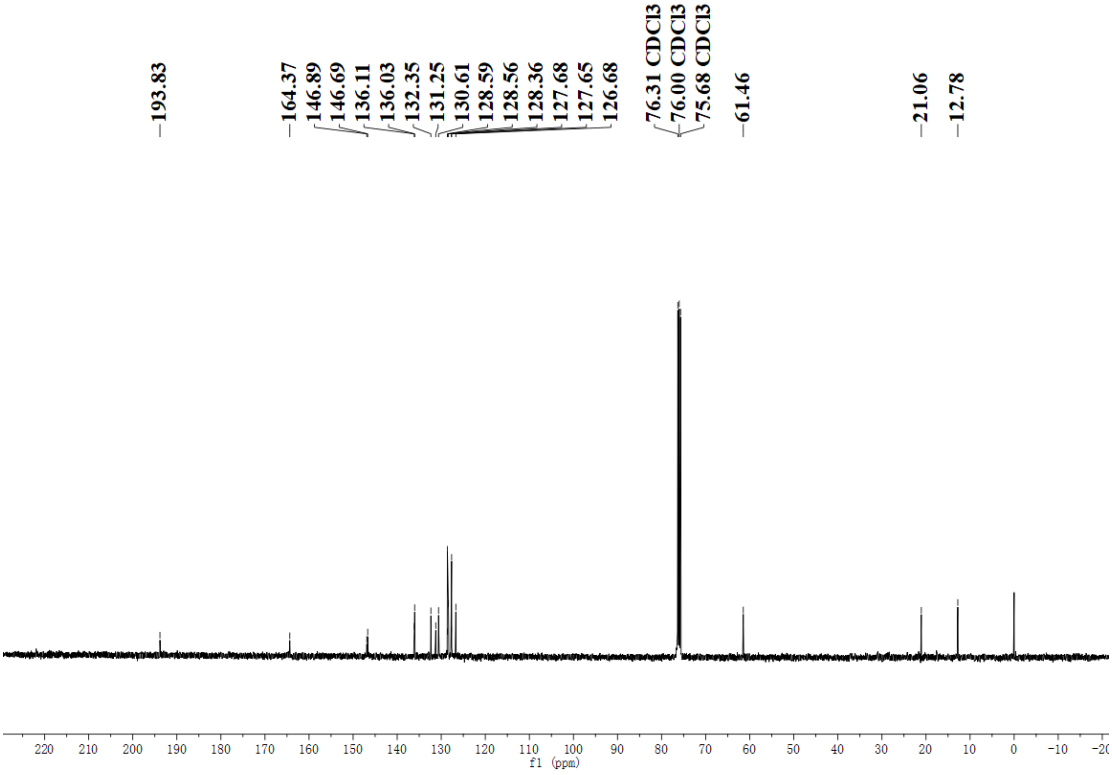


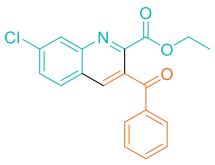


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4f

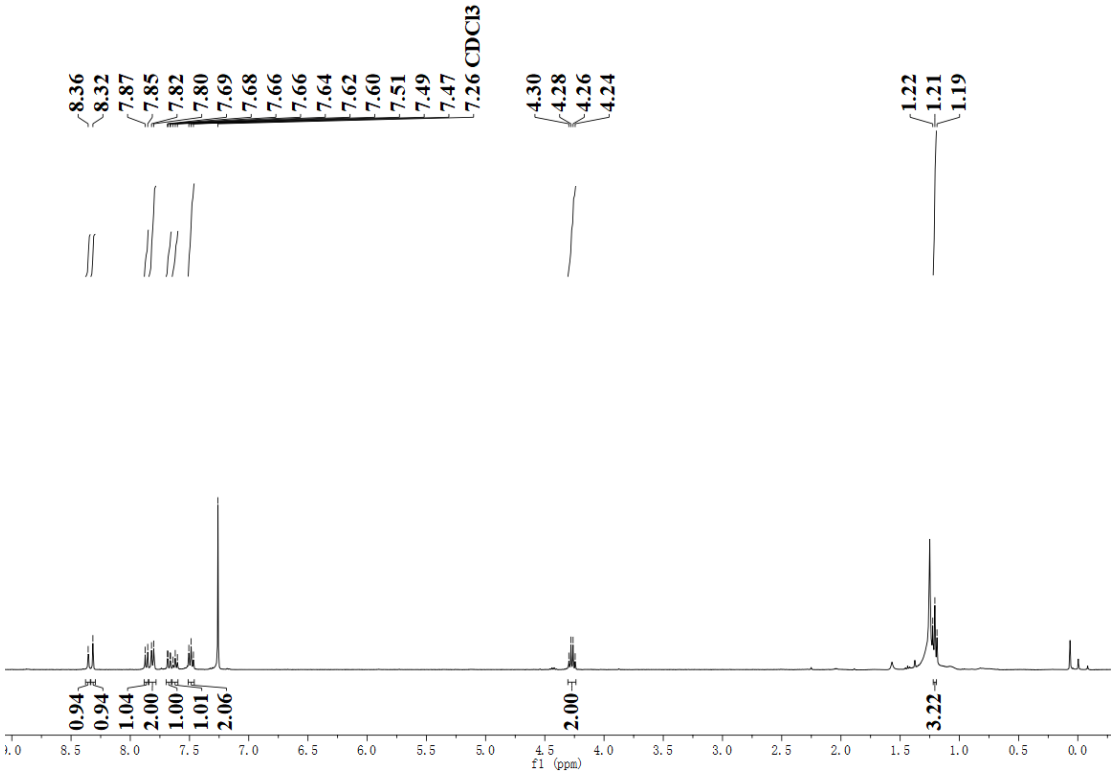


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4f

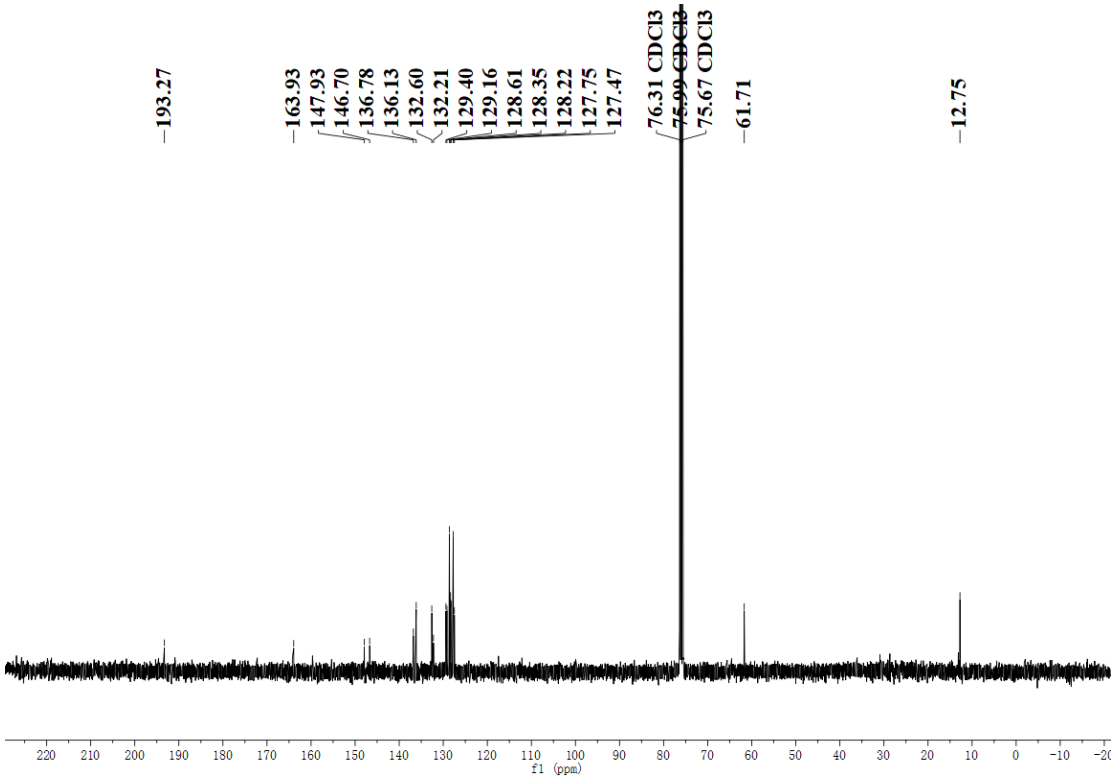


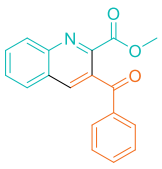


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4g

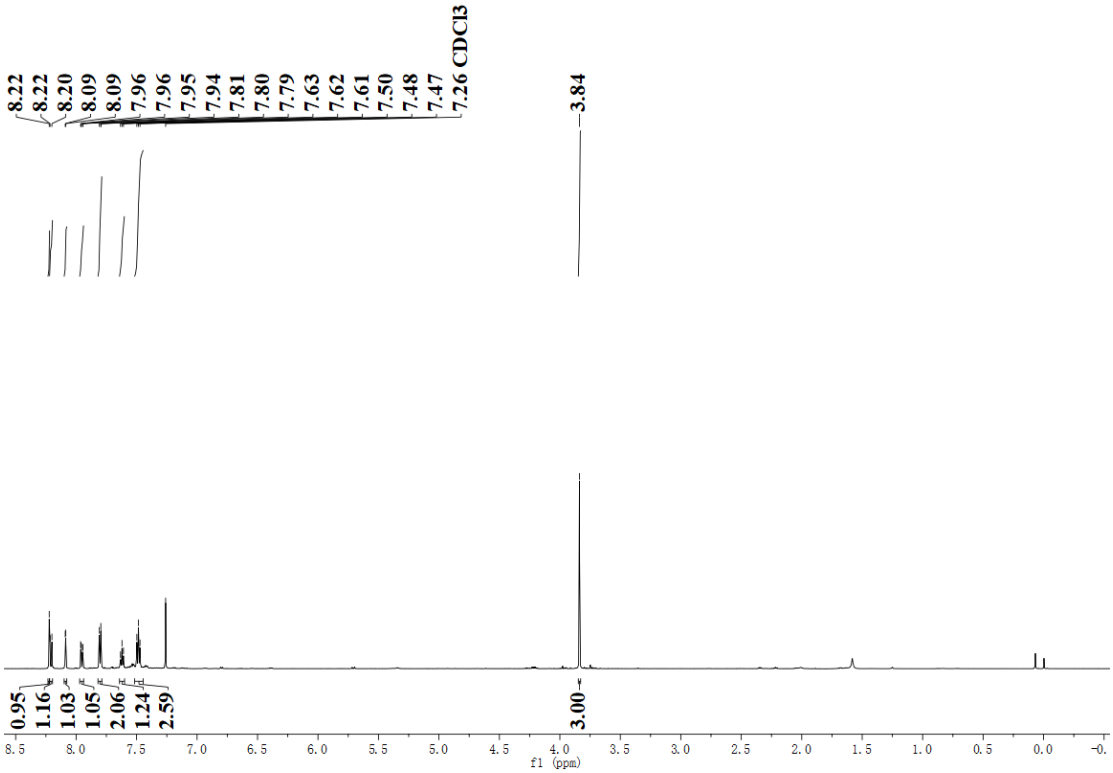


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4g

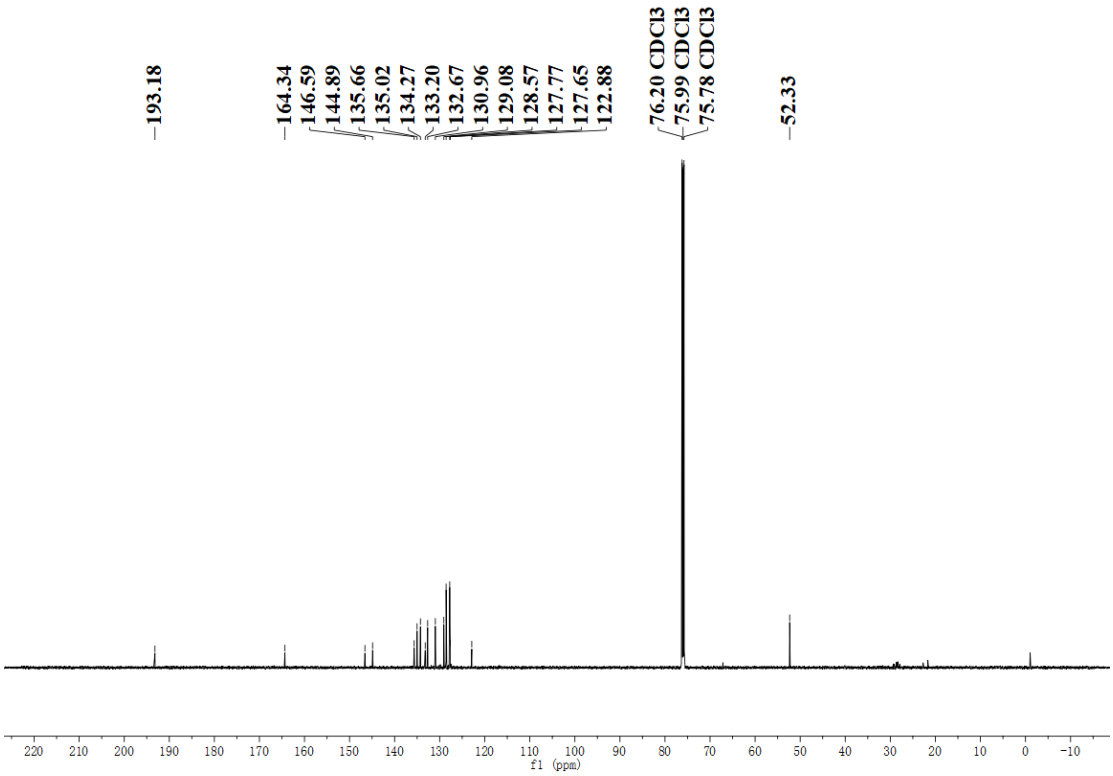


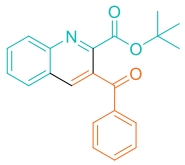


^{1}H-NMR (600MHz, $CDCl_3$) spectrum of compound 4h

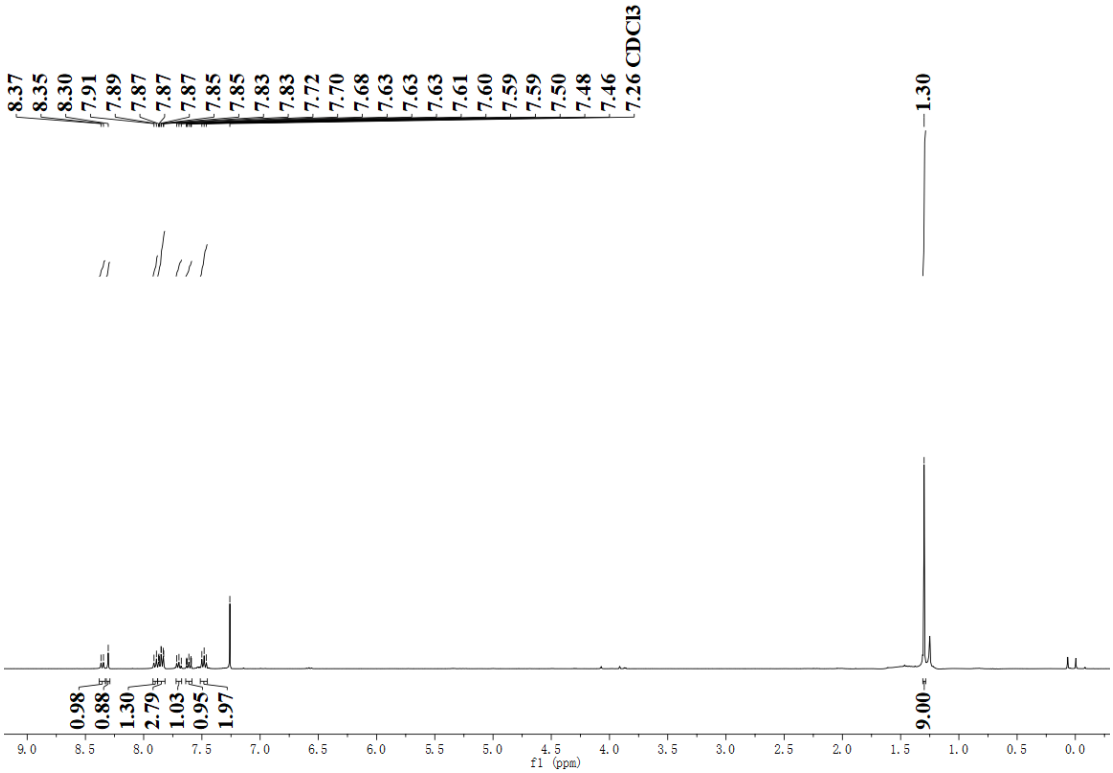


^{13}C-NMR (150MHz, $CDCl_3$) spectrum of compound 4h

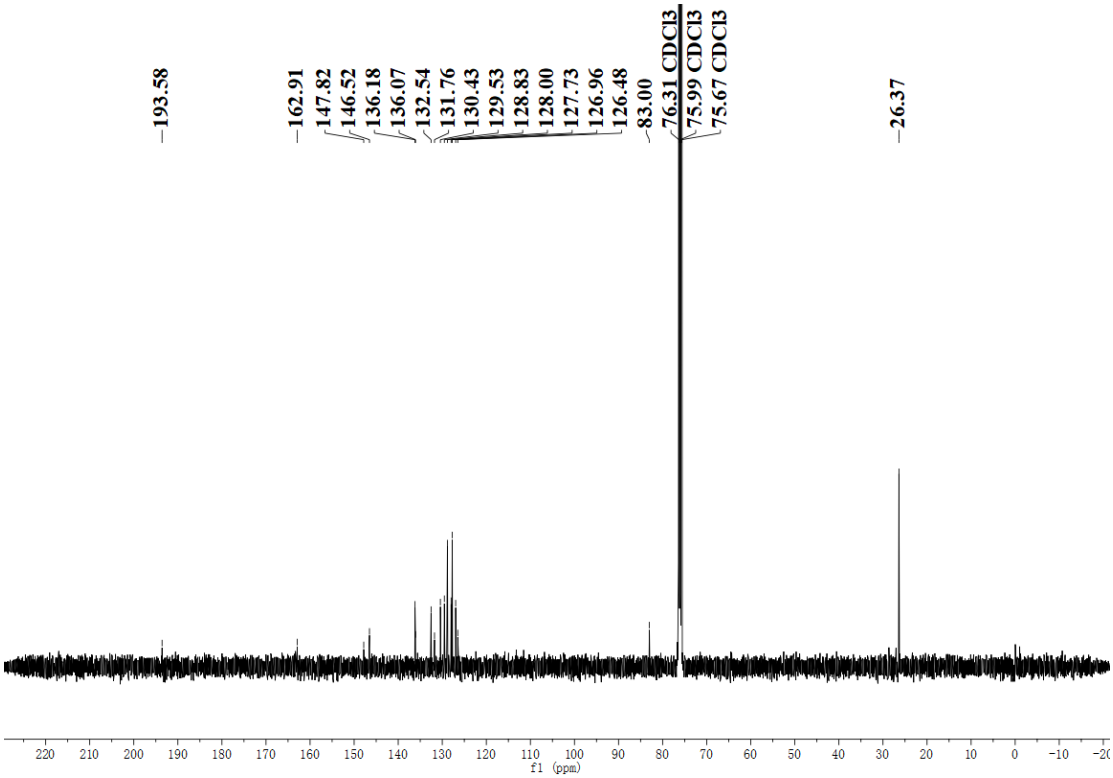


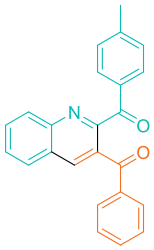


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4i

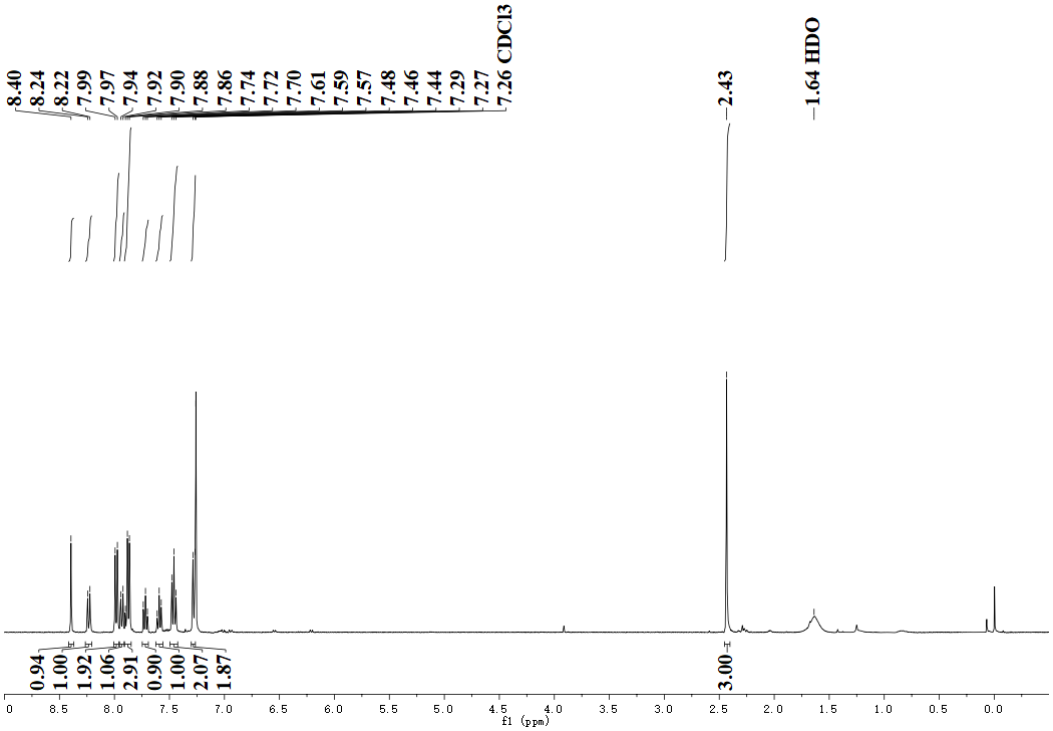


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4i

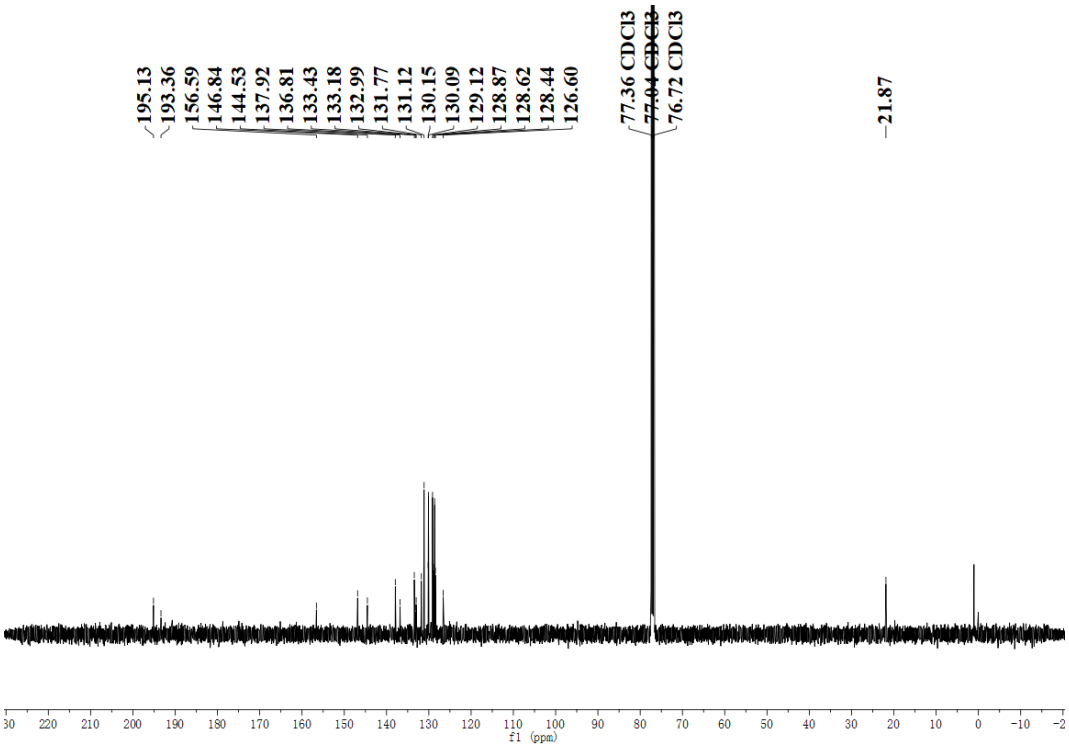


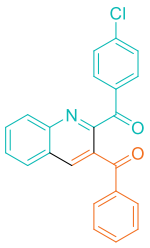


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4j

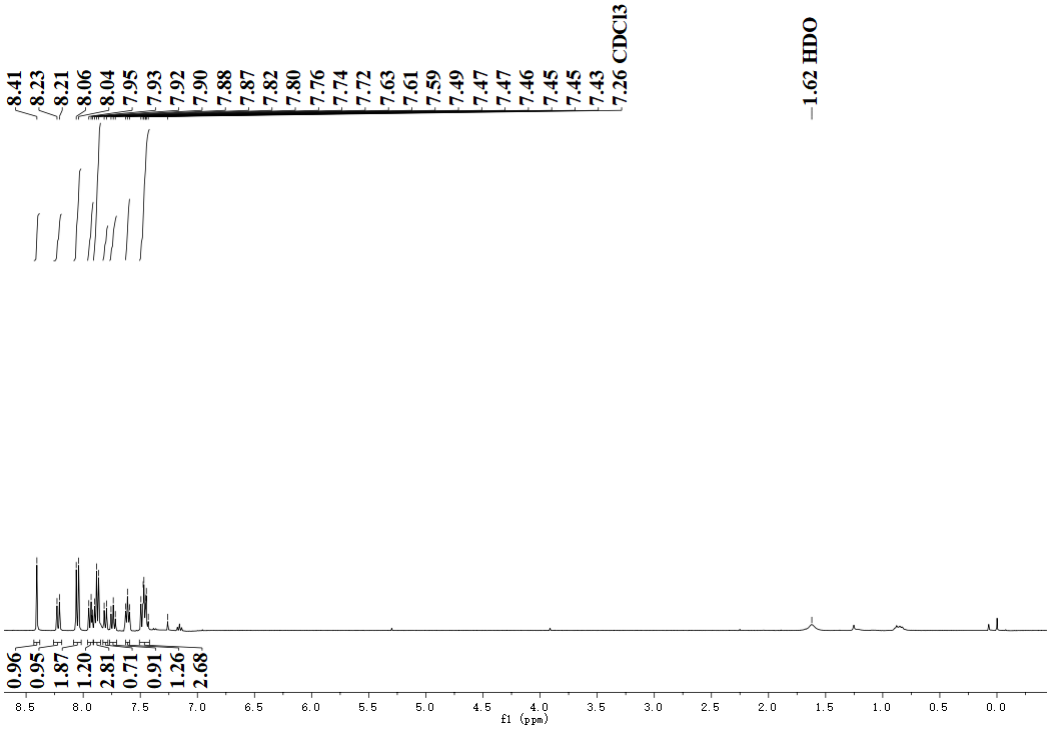


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4j

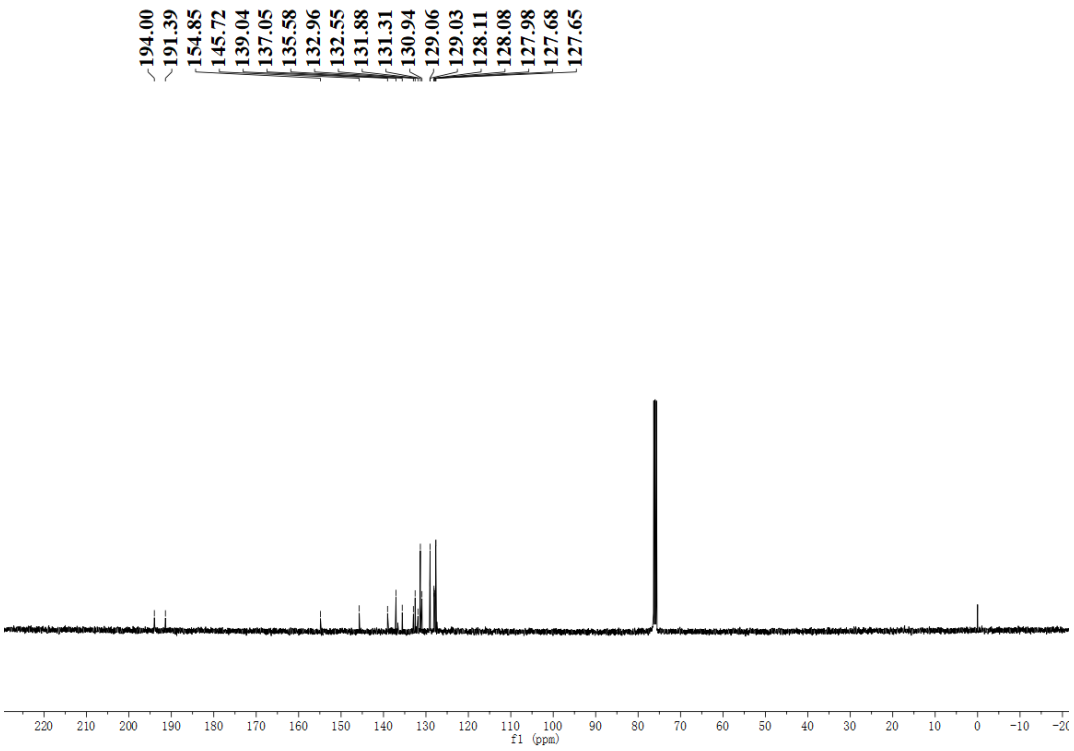


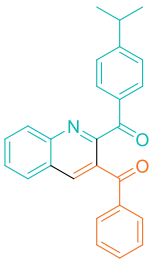


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4k

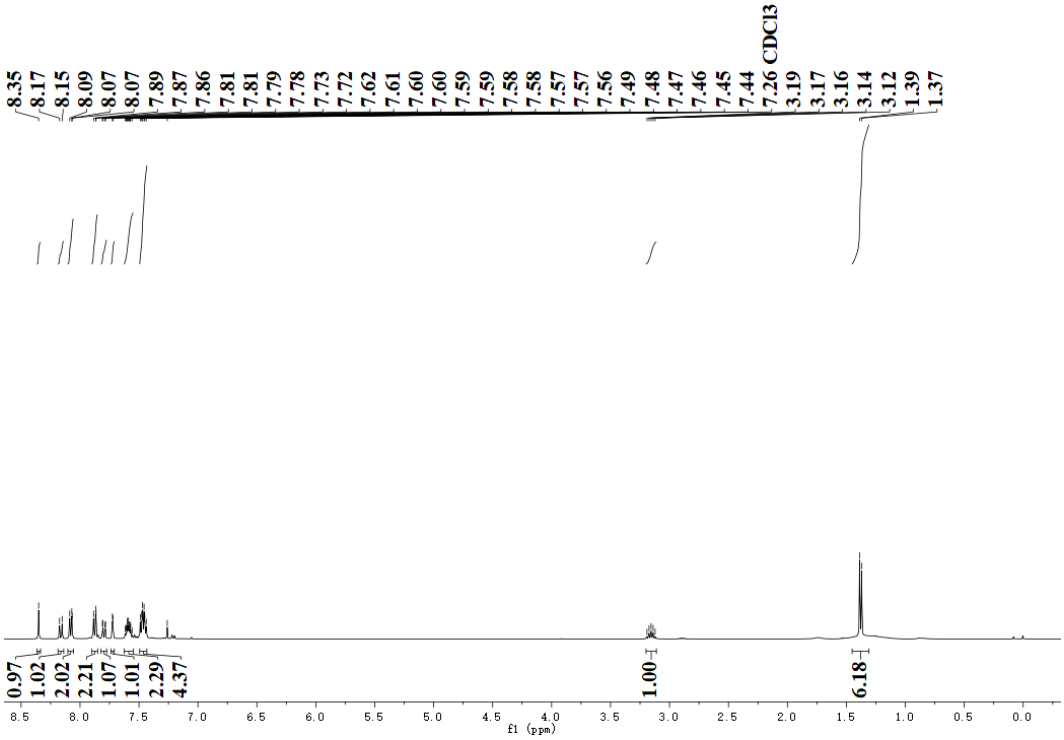


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4k

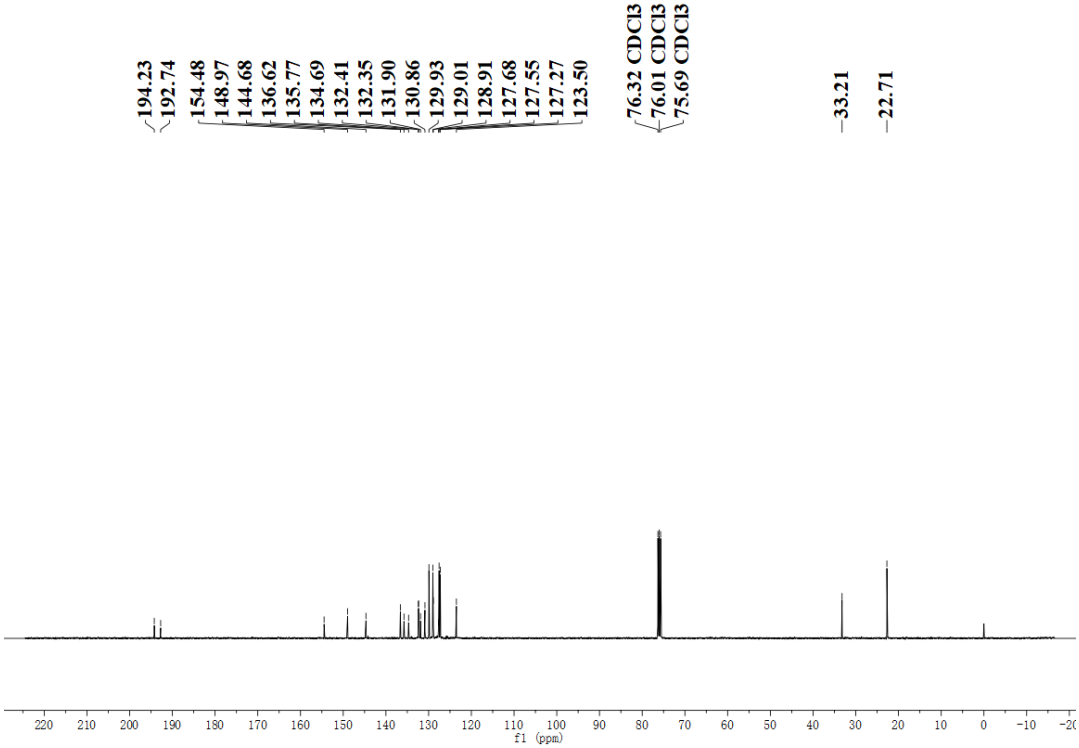


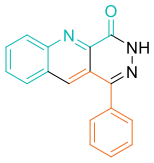


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 4l

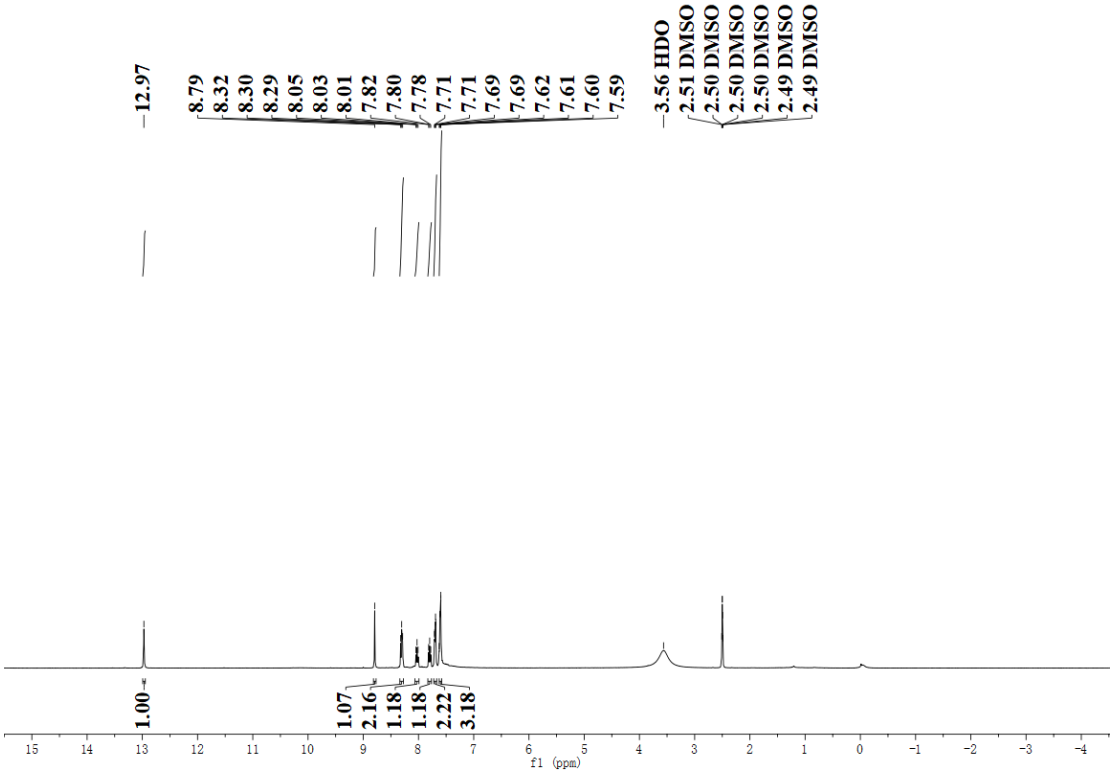


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 4l

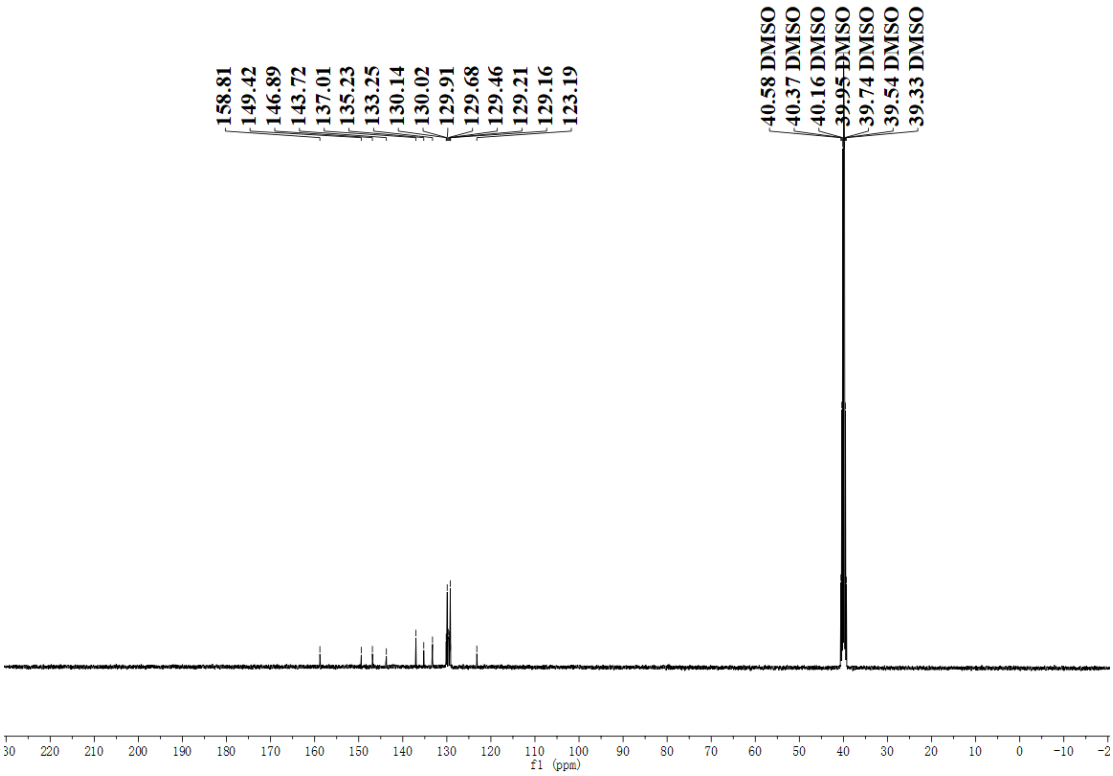


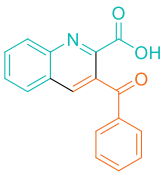


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 5

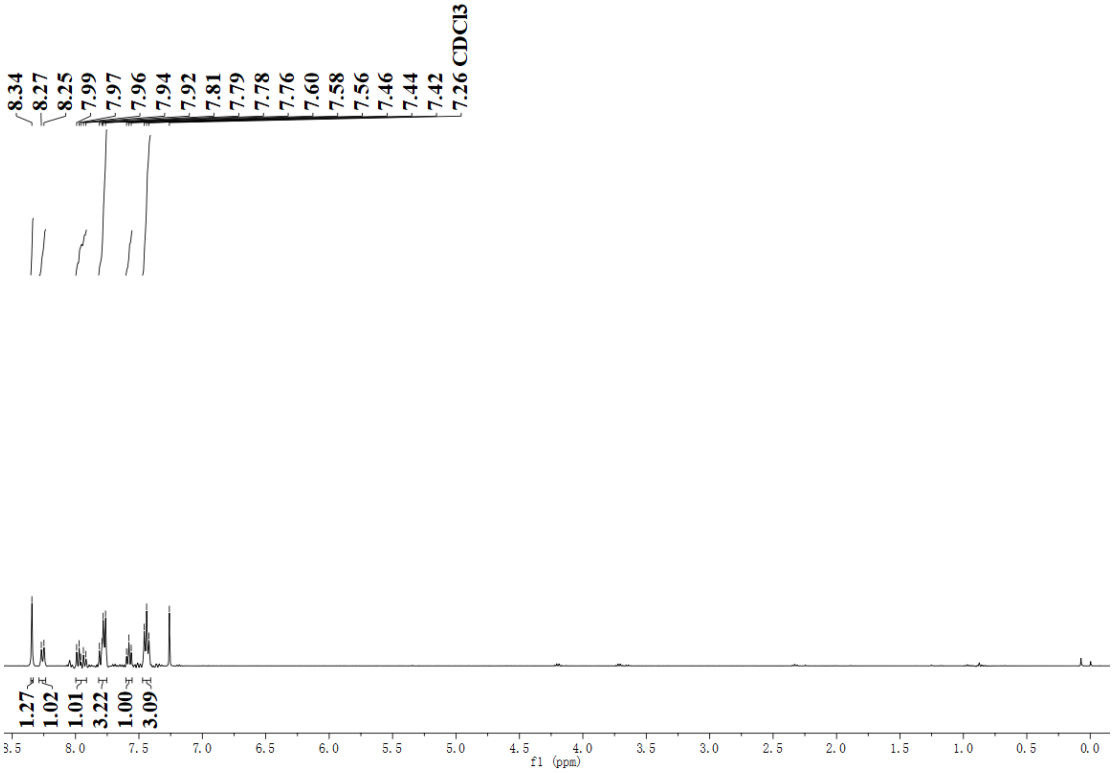


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 5

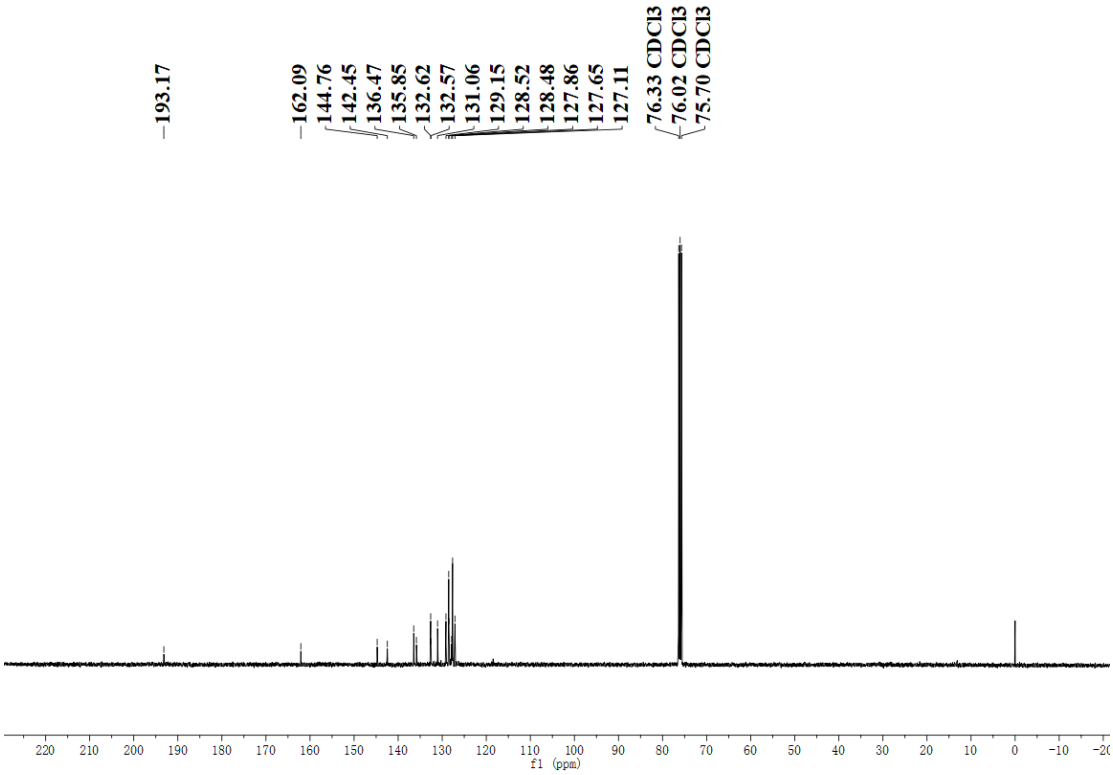


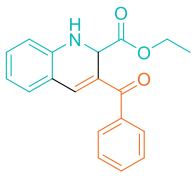


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 6

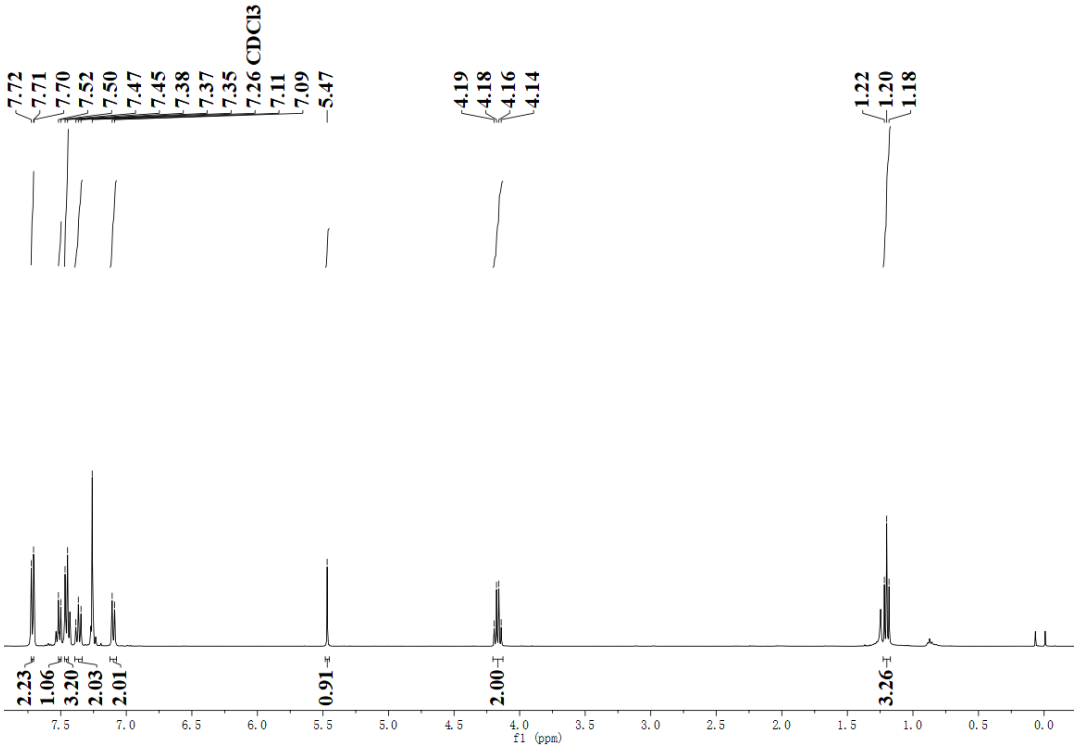


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 6

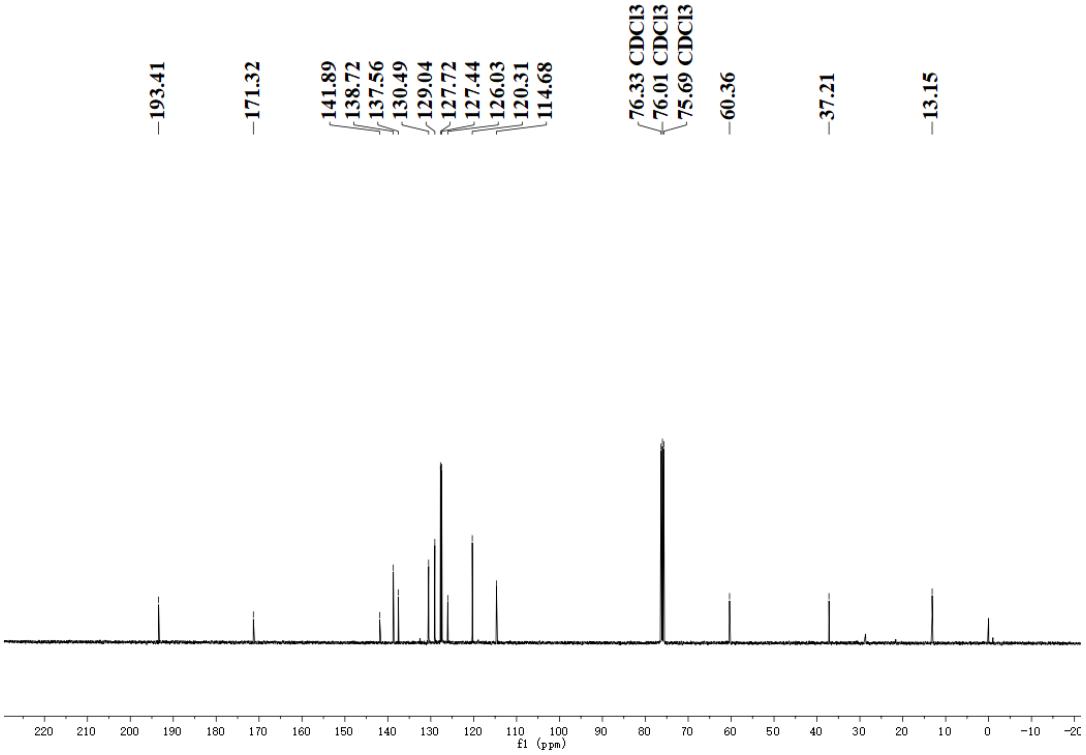


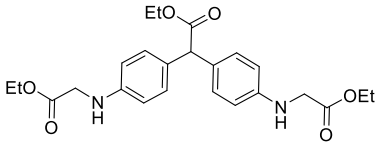


^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound Int-4

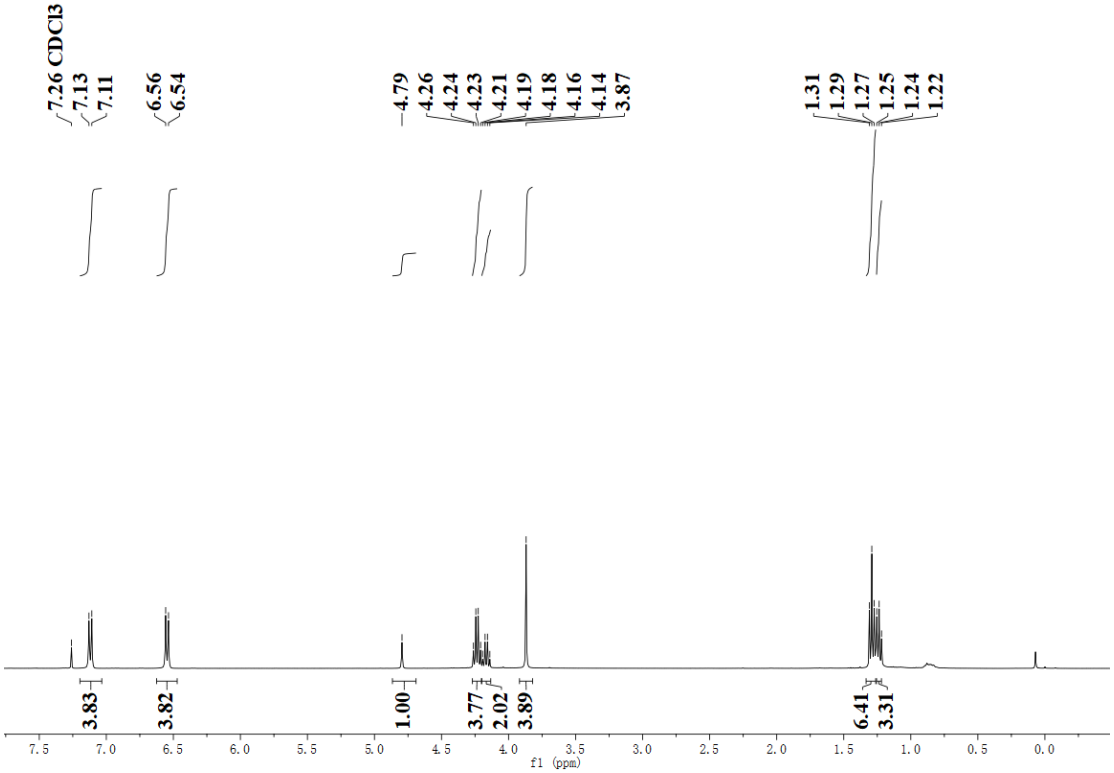


^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound Int-4

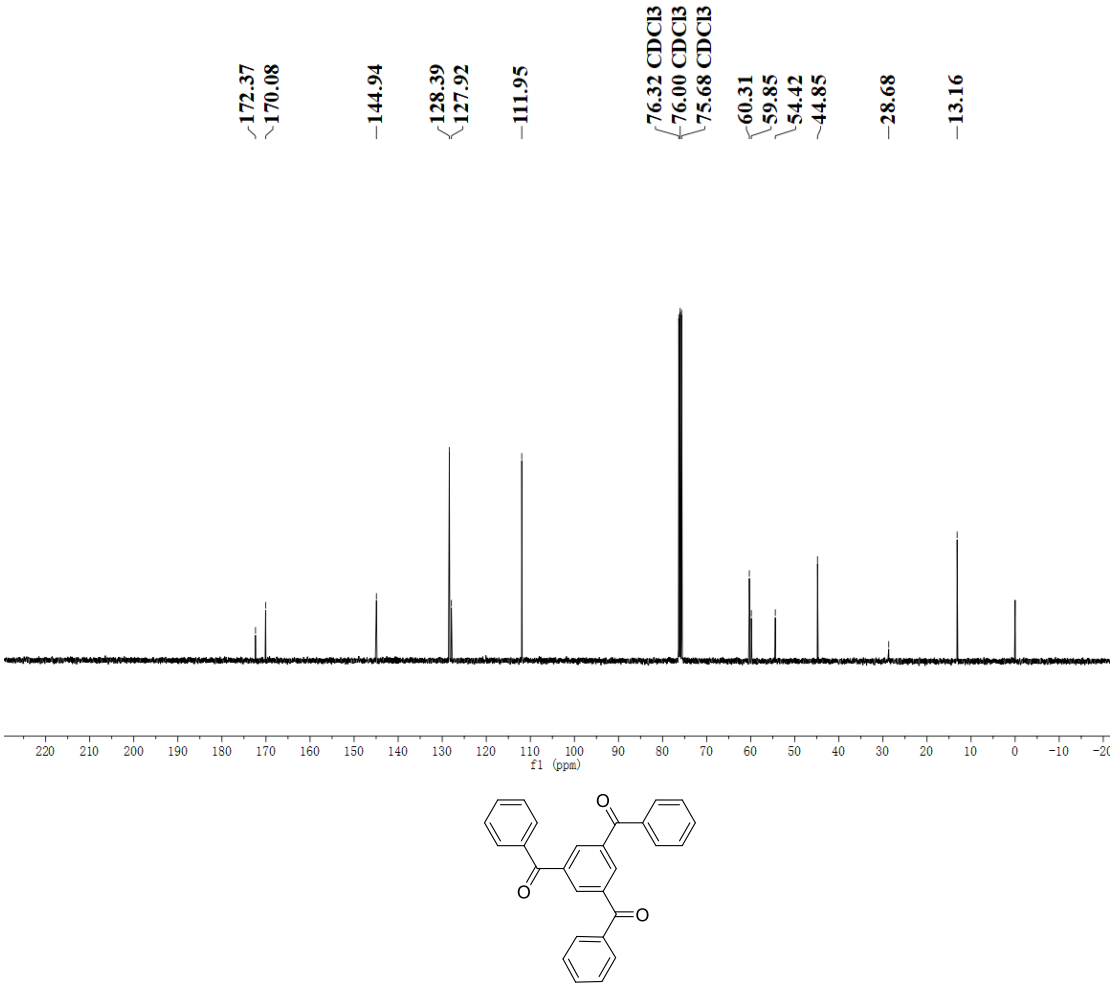




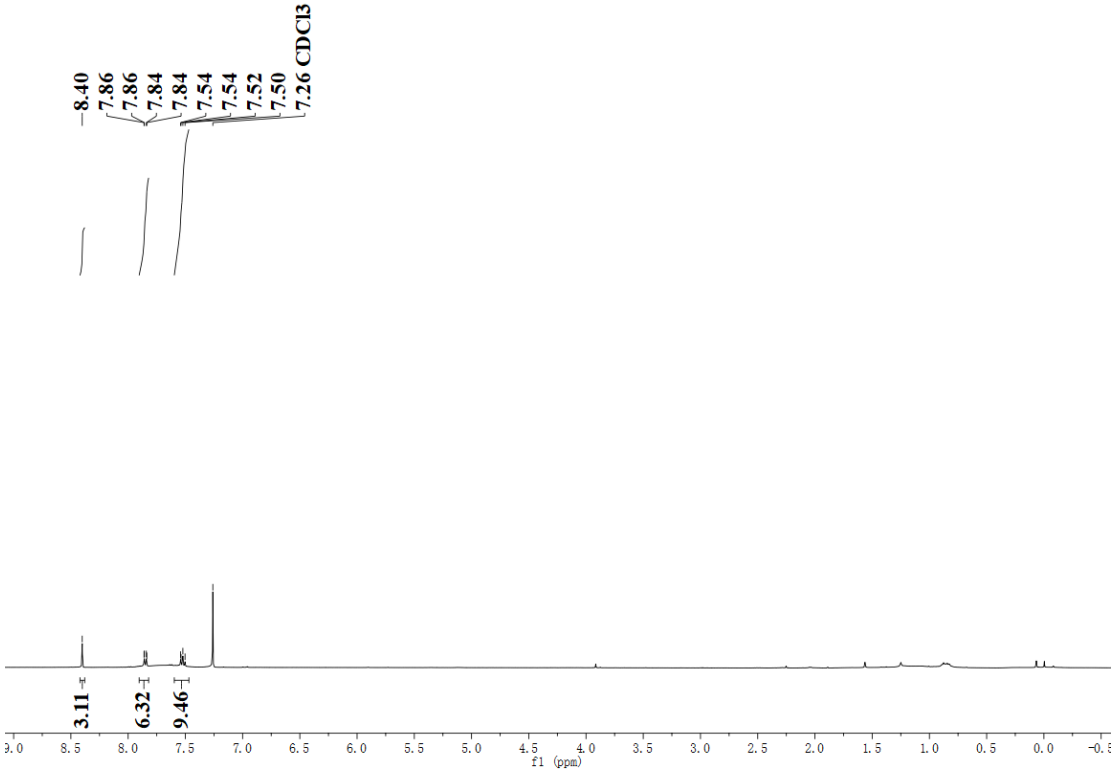
^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 9



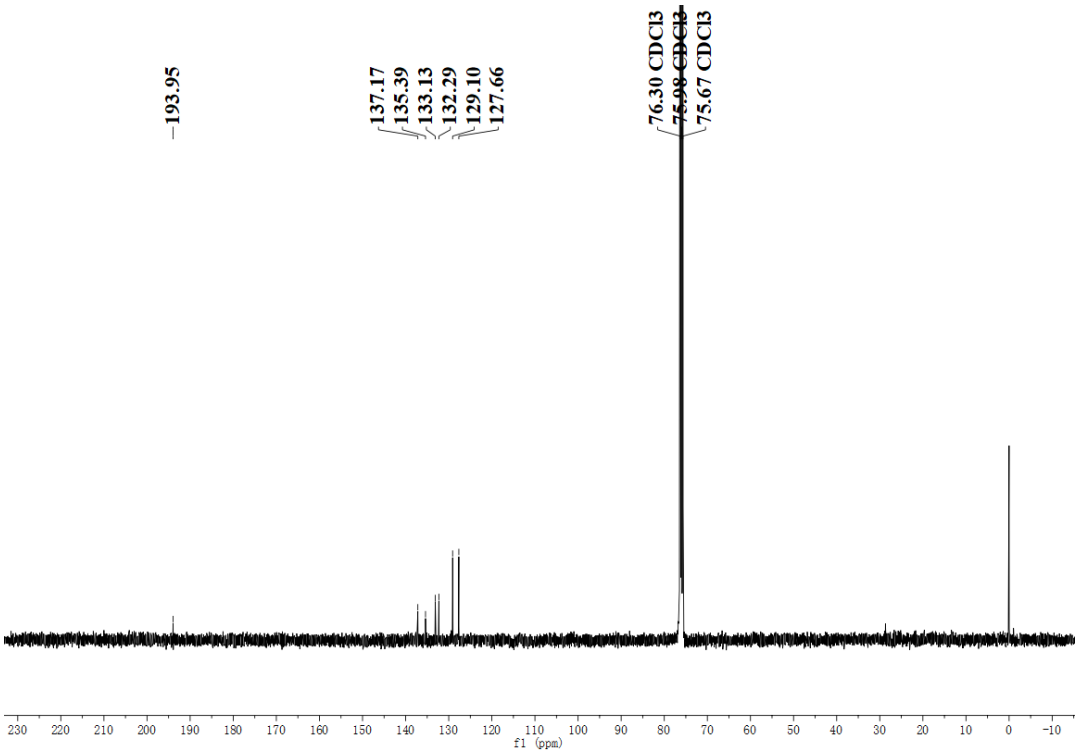
^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 9



^{1}H-NMR (400MHz, $CDCl_3$) spectrum of compound 11



^{13}C-NMR (100MHz, $CDCl_3$) spectrum of compound 11



7.References

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