Supporting Information

Visible-light-promoted catalyst-/additive-free synthesis of aroylated heterocycles in sustainable solvent

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1. General Information

1.1 Materials and instruments

All commercially available reagents were used directly without further purification. All reactions were monitored by Thin Layer Chromatography (TLC) using silica gel F254 plates. Products were purified by column chromatography using 200-300 mesh silica gel as the stationary phase. All the ^{1}H, ^{13}C, and ^{19}F NMR spectra were recorded on Bruker Avance 400 or 600 spectrometers. All NMR spectra were recorded in $CDCl_3$ at room temperature (20 ± 2 °C). Proton chemical shifts δ were given in ppm using TMS as the internal standard. High-resolution mass spectra (HRMS) were obtained with a 3000-mass spectrometer, using Waters Q-Tof MS/MS system with the ESI technique. The reactants involved are prepared by references.[8]

1.2 The spectrum of our lamp and the visible-light irradiation instrument.

The photochemical reaction was carried out under visible light irradiation by a blue LED at 25 ºC. RLH-18 8-position Photo Reaction System manufactured by Beijing Roger Tech Ltd. was used in this system. Eight 10 W blue LEDs were equipped in this Photo reactor. The blue LED'S energy peak wavelength is 458 nm, peak width at half-height is 23.4 nm, Iirradiance@10 W is 188.65 mW/cm². The reaction vessel is a borosilicate glass tube with 1.5 cm from the lamp, and no filter is applied.

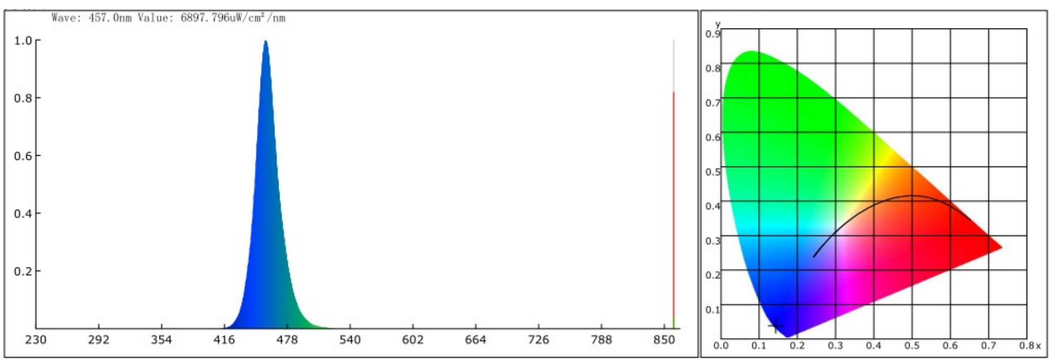


Figure S1a. The spectrum of our lamp (blue LED)

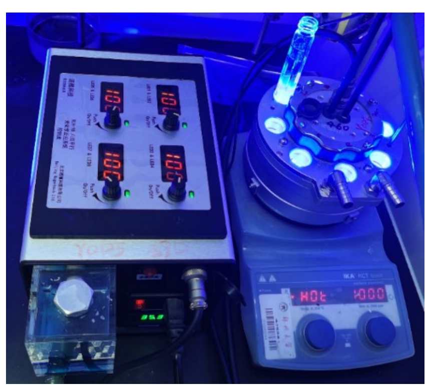
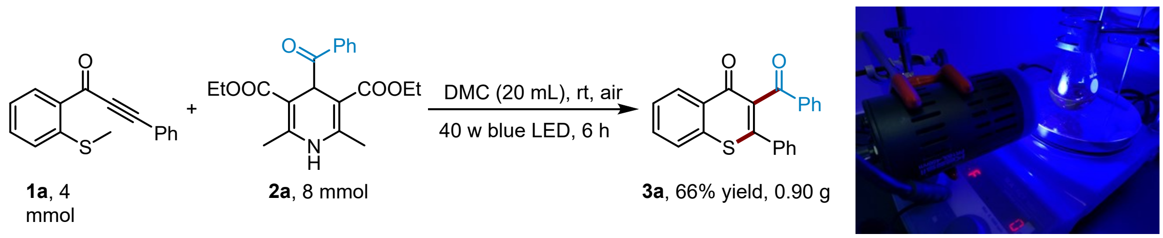


Figure S1b. The visible-light irradiation instrument

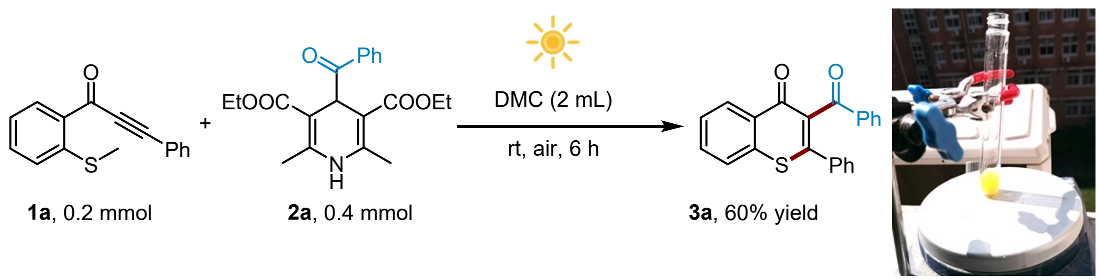
2. Experimental procedures

2.1 The gram-scale synthesis and application under the sunlight



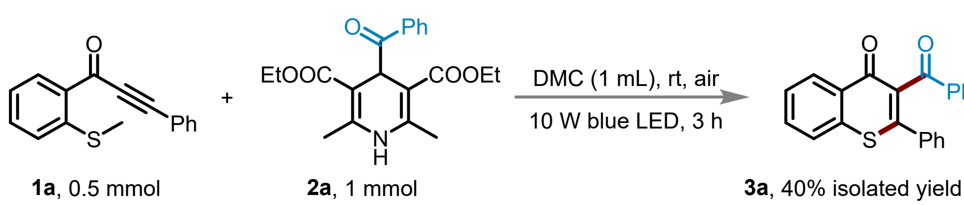
Scheme S1. The gram-scale synthesis of **3a**

The gram-scale synthesis of **3a** with 40 W Kessil blue lamp (456 nm) irradiation in air atmosphere: **1a** (4 mmol), **2a** (8 mmol) in DMC (20 mL) at room temperature for 6 h. The isolated yield of **3a** (66%, 0.90 g) was given. The reaction vessel was a borosilicate glass bottle 5 cm away from the lamp.



Scheme S2. The synthesis of **3a** under the sunlight

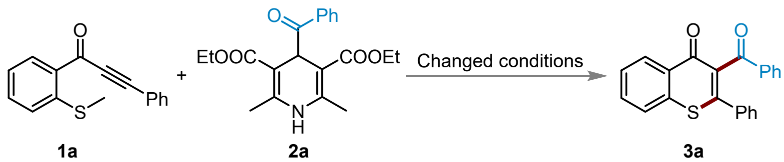
The synthesis under the sunlight: A 25 mL reaction tube was charged with **1a** (0.2 mmol), **2a** (0.4 mmol) in DMC (2 mL) at room temperature under air, then the reaction system was irradiated under sunlight for 6 h (from 10:00 to 16:00; 2021/09/20. Zhengzhou, Henan province, China. Temperature: 19 ^{o}C – 29 ^{o}C). The isolated yield of **3a** (60%) was given.



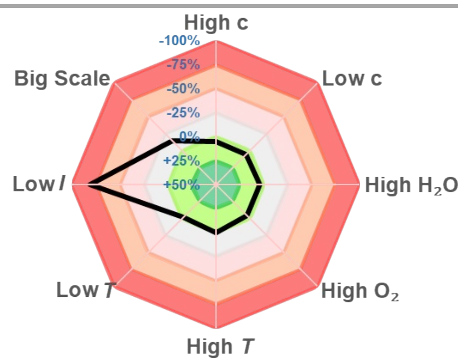
Scheme S3. The reaction at 0.5 M concentration.

2.2 Sensitivity assessment of this reaction.

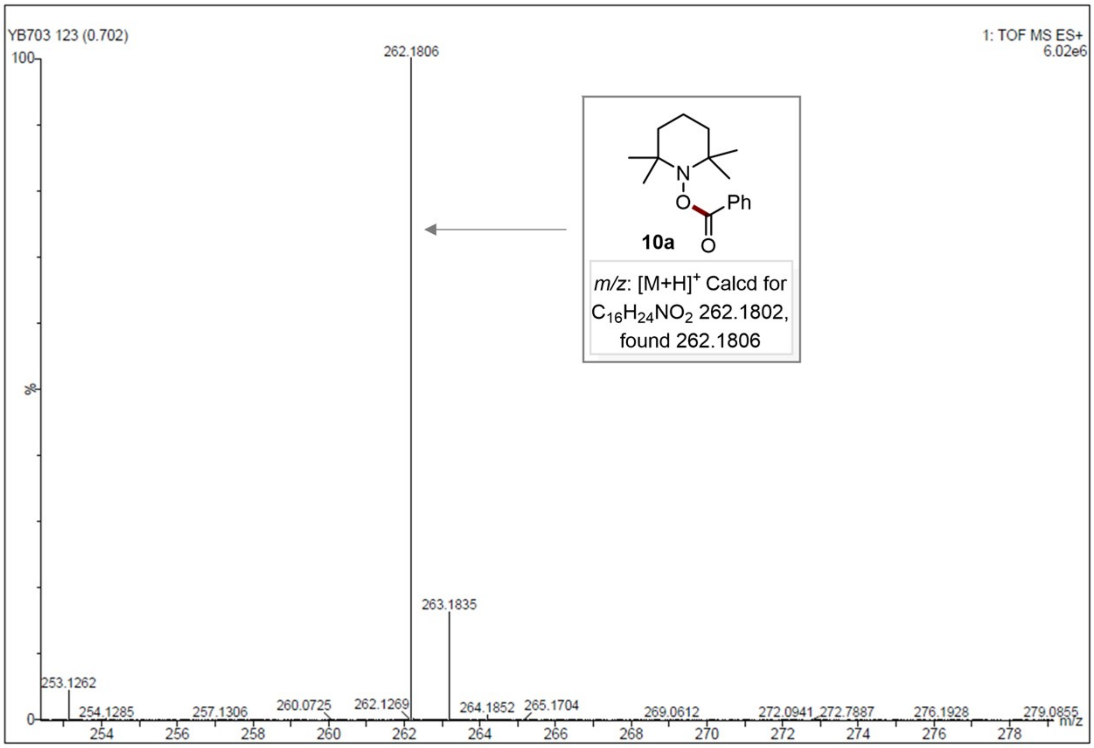
Table S1. Sensitivity assessment of this reaction



Variation of	Lower Point	Center Point	Higher Point
Concentration (*c*)	*c* - 10% *c*	Standard conditions	*c* + 10% *c*
H_2O level	DMC (2 mL)		$+H_2O$; $V_{H_2O} = 1\%V_{rxn}$
O_2 level	air		O_2 balloon
Temperature (*T*)	*T* – 10 ºC		*T* + 10 ºC
Light intensity (*I*)	0.5 W		10 W
Scale	n = 0.2 mmol		n*20 (4 mmol)



2.3 HRMS data analysis



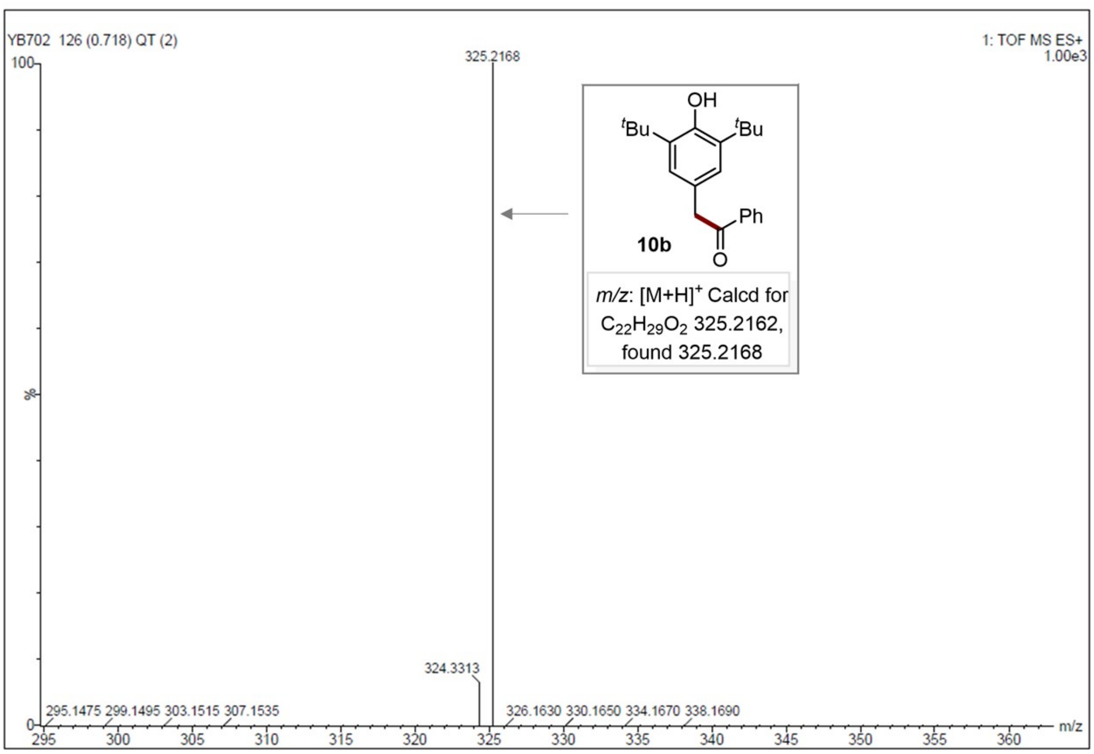
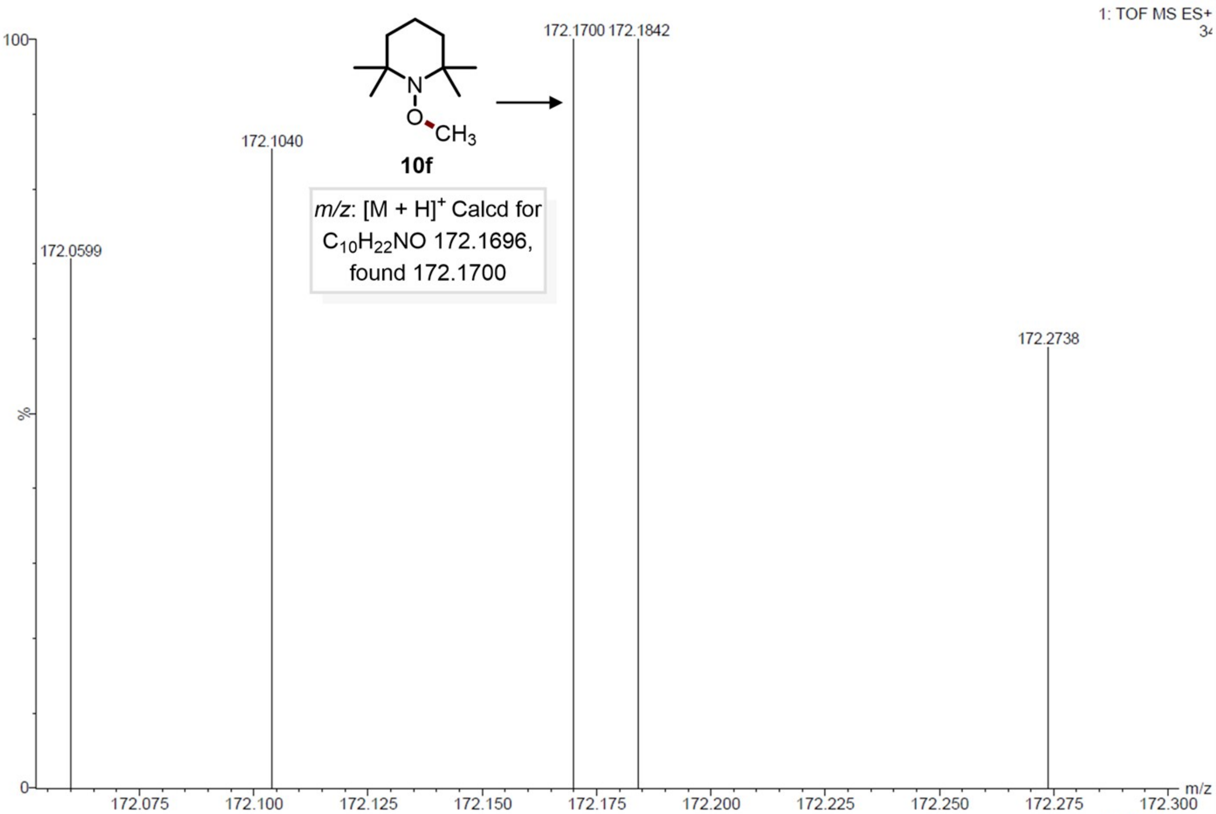


Figure S2i. HRMS data analysis of **10a-b**



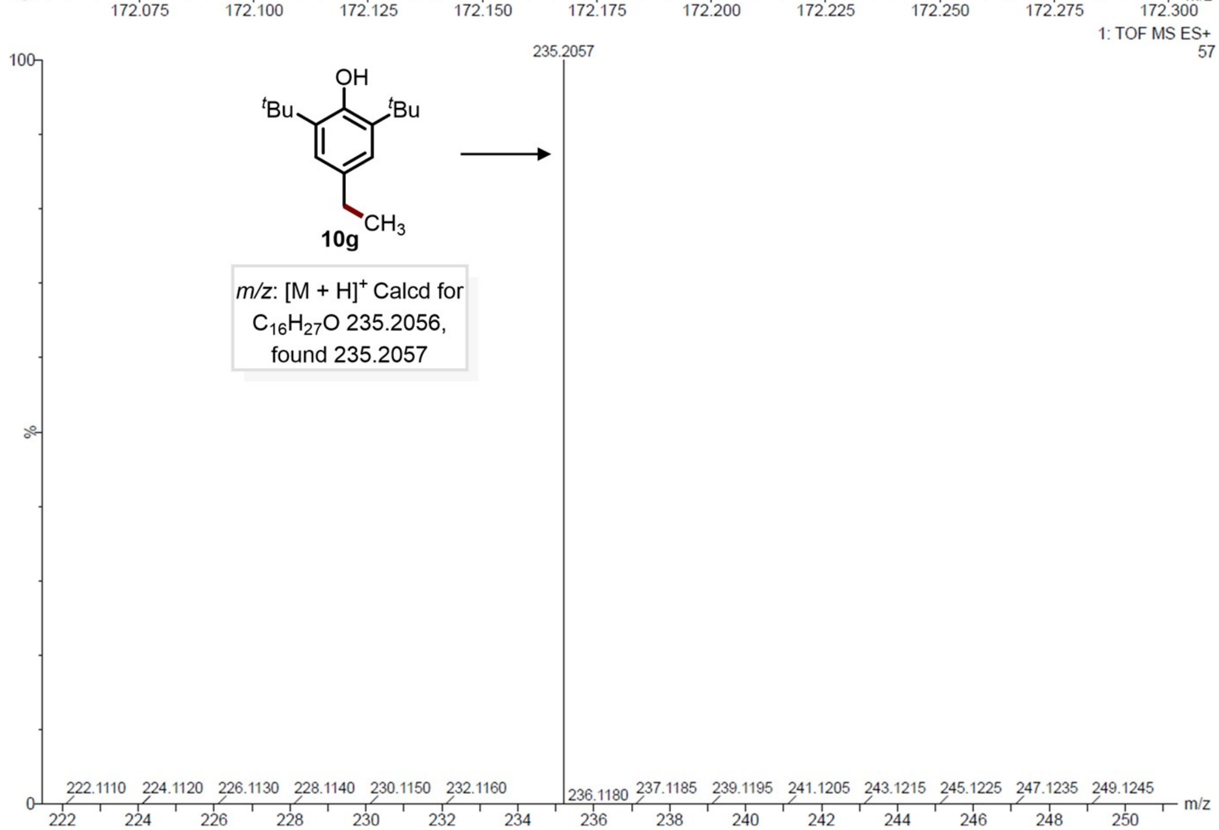


Figure S2ii. HRMS data analysis of **10f-g**

2.4 The UV-Vis and Fluorescence quenching experiments

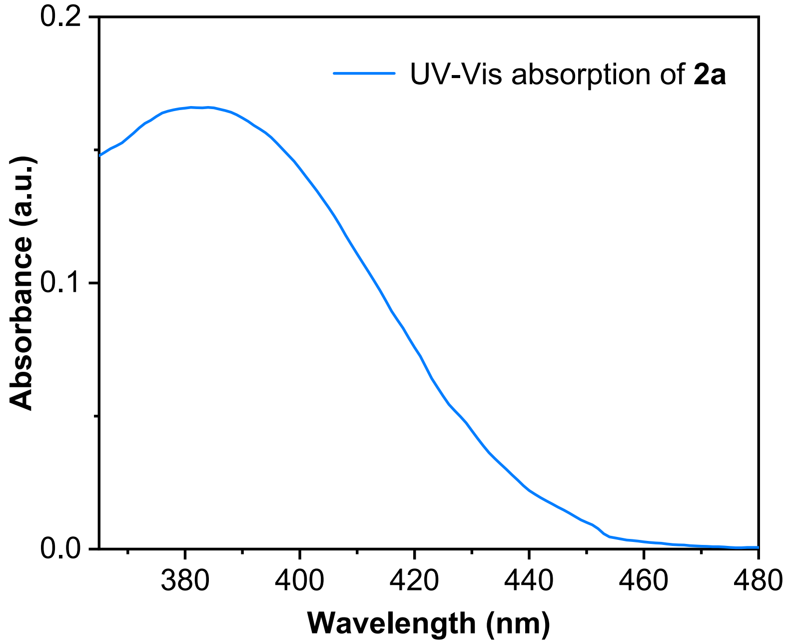


Figure S3. The UV-Vis experiment of **2a** (10^{-4} M) in MeCN.

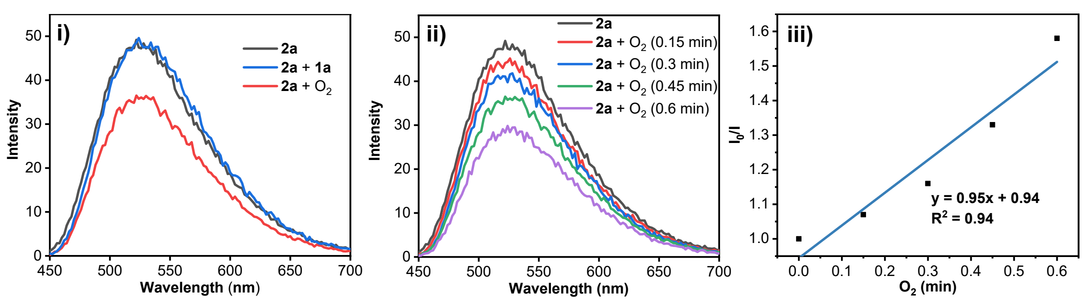
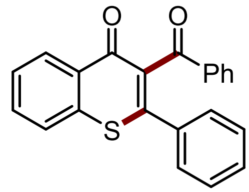


Figure S4. Fluorescence quenching experiments: i) The fluorescence quenching of **1a** and O_2; ii) The fluorescence quenching of a 12.5 mM solution of **2a** with the change of the time of O_2 bubbling (O_2 is bubbled through a balloon with a needle without external pressure.) in degassed anhydrous DMC excited at 380 nm; iii) The linear relationship between I_0/I (I_0 and I are the fluorescence intensities before and after the increasing the concentration of O_2, respectively) and the time for O_2 to be bubbled into the solvent.

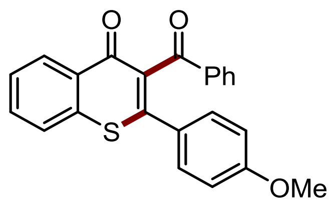
3. **Characterization Data for Products**

3-benzoyl-2-phenyl-4H-thiochromen-4-one (3a)[1]



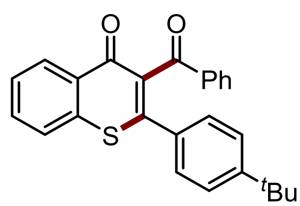
White solid (58.8 mg, 86% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 (d, *J* = 8.0 Hz, 1H), 7.83 – 7.76 (m, 2H), 7.72 – 7.65 (m, 2H), 7.61 – 7.57 (m, 1H), 7.50 – 7.41 (m, 3H), 7.38 – 7.27 (m, 5H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.8, 178.9, 152.1, 137.5, 137.0, 135.2, 134.7, 133.4, 132.1, 130.7, 130.3, 129.2, 129.1, 128.8, 128.59, 128.57, 128.1, 126.1.

3-benzoyl-2-(4-methoxyphenyl)-4H-thiochromen-4-one (3b)[1]



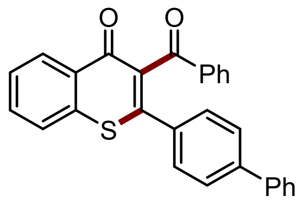
White solid (55.0 mg, 74% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.86 – 7.78 (m, 2H), 7.71 – 7.64 (m, 2H), 7.59 – 7.55 (m, 1H), 7.48 – 7.46 (m, 1H), 7.42 – 7.32 (m, 4H), 6.81 (d, *J* = 8.8 Hz, 2H), 3.76 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.1, 178.9, 161.2, 152.0, 137.6, 137.0, 134.8, 133.4, 132.0, 130.6, 130.1, 129.2, 129.1, 128.6, 128.0, 127.0, 126.0, 114.3, 55.3.

3-benzoyl-2-(4-(tert-butyl)phenyl)-4H-thiochromen-4-one (3c)[2]



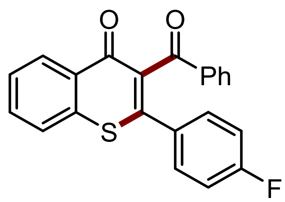
Yellow solid (47.8 mg, 60% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 – 8.52 (m, 1H), 7.84 – 7.78 (m, 2H), 7.70 – 7.64 (m, 2H), 7.60 – 7.56 (m, 1H), 7.50 – 7.46 (m, 1H), 7.38 – 7.33 (m, 4H), 7.32 – 7.28 (m, 2H), 1.25 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.0, 179.0, 153.7, 152.5, 137.7, 137.3, 135.0, 133.3, 132.0, 131.9, 130.7, 129.14, 129.06, 128.5, 128.3, 128.0, 126.0, 125.8, 34.8, 31.1.

2-([1,1'-biphenyl]-4-yl)-3-benzoyl-4H-thiochromen-4-one (3d)[4]



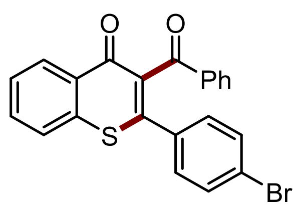
White solid (79.4 mg, 95% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 – 8.53 (m, 1H), 7.84 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.61 – 7.58 (m, 1H), 7.54 – 7.45 (m, 7H), 7.43 – 7.32 (m, 5H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.9, 178.9, 151.9, 143.1, 139.6, 137.5, 137.1, 135.2, 133.6, 133.5, 132.1, 130.7, 129.2, 129.10, 129.06, 128.9, 128.6, 128.2, 128.0, 127.4, 127.1, 126.1.

3-benzoyl-2-(4-fluorophenyl)-4H-thiochromen-4-one (3e)[2]



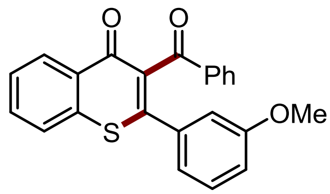
Yellow solid (48.2 mg, 67% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 (d, *J* = 7.6 Hz, 1H), 7.83 – 7.75 (m, 2H), 7.72 – 7.65 (m, 2H), 7.61 – 7.57 (m, 1H), 7.52 – 7.46 (m, 1H), 7.45 – 7.39 (m, 2H), 7.38 – 7.34 (m, 2H), 7.03 – 6.94 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.7, 178.8, 163.7 (d, $^{1}J_{C-F}$ = 251.8 Hz), 150.8, 137.3, 136.9, 135.5, 133.6, 132.2, 130.80, 130.75, 130.7, 130.6, 129.1 (d, $^{3}J_{C-F}$ = 1.8 Hz), 128.7, 128.2, 126.1, 116.0 (d, $^{2}J_{C-F}$ = 22.1 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -109.38.

3-benzoyl-2-(4-bromophenyl)-4H-thiochromen-4-one (3f)



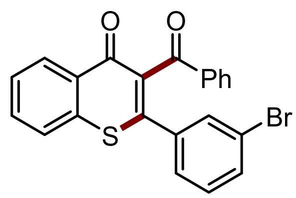
Yellow solid (47.0 mg, 56% yield), mp 148.4 – 150.3 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 7.9 Hz, 1H), 7.82 – 7.78 (m, 2H), 7.72 – 7.65 (m, 2H), 7.61 – 7.57 (m, 1H), 7.52 – 7.47 (m, 1H), 7.46 – 7.42 (m, 2H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.29 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.5, 178.8, 150.6, 137.2, 136.8, 135.5, 133.7, 133.6, 132.2, 132.1, 130.6, 130.1, 129.1, 128.7, 128.3, 126.1, 125.0. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{22}H_{14}BrO_2S$, 420.9892, Found: 420.9897.

3-benzoyl-2-(3-methoxyphenyl)-4H-thiochromen-4-one (3g)



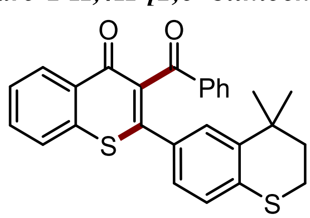
Colorless oil (53.5 mg, 72% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 1.1 Hz, 1H), 7.90 – 7.77 (m, 2H), 7.72 – 7.63 (m, 2H), 7.61 – 7.56 (m, 1H), 7.52 – 7.45 (m, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 8.0 Hz, 1H), 7.03 –7.00 (m, 1H), 6.94 (t, *J* = 2.0 Hz, 1H), 6.88 – 6.85 (m, 1H), 3.63 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.8, 178.9, 159.5, 151.9, 137.5, 137.1, 135.9, 135.2, 133.4, 132.1, 130.7, 129.9, 129.2, 129.1, 128.6, 128.1, 126.1, 120.9, 116.7, 113.6, 55.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{23}H_{17}O_3S$, 373.0893, Found: 373.0897.

3-benzoyl-2-(3-bromophenyl)-4H-thiochromen-4-one (3h)



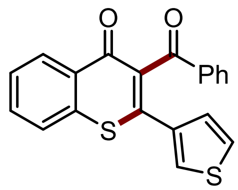
White solid (58.5 mg, 70% yield), mp 139.8 – 141.7 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 6.7 Hz, 1H), 7.83 – 7.74 (m, 2H), 7.73 – 7.64 (m, 2H), 7.63 – 7.55 (m, 2H), 7.52 – 7.43 (m, 2H), 7.39 – 7.33 (m, 3H), 7.15 (t, *J* = 7.9 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.3, 178.7, 150.0, 137.2, 136.9, 136.5, 135.7, 133.6, 133.3, 132.3, 131.4, 130.6, 130.2, 129.13, 129.11, 128.7, 128.3, 127.3, 126.1, 122.7. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{22}H_{14}BrO_2S$, 420.9892, Found: 420.9897.

3-benzoyl-4',4'-dimethyl-3',4'-dihydro-2'H,4H-[2,6'-bithiochromen]-4-one (3i)



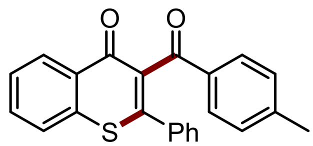
Yellow solid (50.6 mg, 60% yield), mp 163.2 – 165.2 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.52 (d, *J* = 8.1 Hz, 1H), 7.88 – 7.83 (m, 2H), 7.70 – 7.64 (m, 2H), 7.58 – 7.54 (m, 1H), 7.51 – 7.47 (m, 1H), 7.40 – 7.34 (m, 3H), 7.13 (dd, *J* = 8.2, 2.1 Hz, 1H), 7.02 (d, *J* = 8.2 Hz, 1H), 2.98 – 2.92 (m, 2H), 1.87 – 1.80 (m, 2H), 1.07 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.0, 179.0, 152.4, 142.3, 137.5, 137.1, 135.9, 134.7, 133.4, 132.0, 130.6, 130.4, 129.3, 129.0, 128.6, 128.0, 127.0, 126.9, 126.1, 125.6, 36.9, 32.9, 29.6, 23.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{27}H_{23}O_2S_2$, 443.1134, Found: 443.1141.

3-benzoyl-2-(thiophen-3-yl)-4H-thiochromen-4-one (3j)[2]



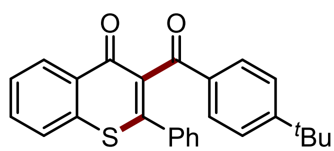
White solid (33.4 mg, 48% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 – 8.48 (m, 1H), 7.88 – 7.82 (m, 2H), 7.70 – 7.64 (m, 2H), 7.60 – 7.53 (m, 2H), 7.52 – 7.46 (m, 1H), 7.41 – 7.34 (m, 2H), 7.28 – 7.26 (m, 1H), 7.17 (dd, *J* = 5.1, 1.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.4, 179.1, 146.0, 137.2, 136.7, 135.0, 134.3, 133.6, 132.1, 130.6, 129.1, 129.0, 128.7, 128.1, 127.7, 127.4, 127.2, 126.0.

***3-(4-methylbenzoyl)-2-phenyl-4H-thiochromen-4-one (3k)*[1]**



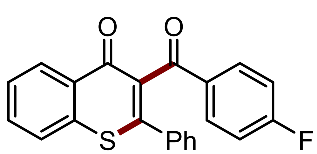
White solid (58.4 mg, 82% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.57 – 8.49 (m, 1H), 7.73 – 7.64 (m, 4H), 7.59 – 7.55 (m, 1H), 7.46 – 7.41 (m, 2H), 7.36 – 7.27 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.2, 178.8, 151.7, 144.3, 137.5, 135.5, 134.9, 134.7, 132.0, 130.7, 130.2, 129.3, 129.1, 128.7, 128.6, 128.0, 126.0, 21.7.

3-(4-(tert-butyl)benzoyl)-2-phenyl-4H-thiochromen-4-one (3l)



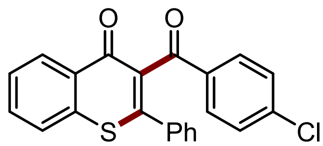
White solid (59.7 mg, 75% yield), mp 179.1 – 180.8 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 (dd, *J* = 8.3, 4.5 Hz, 1H), 7.81 – 7.64 (m, 4H), 7.61 – 7.56 (m, 1H), 7.48 – 7.44 (m, 2H), 7.39 – 7.27 (m, 5H), 1.28 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.3, 178.9, 157.1, 151.8, 137.6, 135.5, 134.9, 134.5, 132.0, 130.7, 130.2, 129.1, 128.7, 128.6, 128.0, 126.0, 125.6, 35.1, 31.0. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{26}H_{23}O_2S$, 399.1413, Found: 399.1415.

***3-(4-fluorobenzoyl)-2-phenyl-4H-thiochromen-4-one (3m)*[1]**



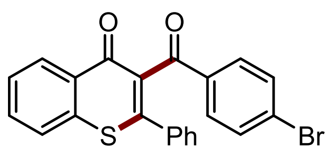
White solid (40.3 mg, 56% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 (d, *J* = 8.1 Hz, 1H), 7.86 – 7.78 (m, 2H), 7.69 (d, *J* = 7.2 Hz, 2H), 7.62 – 7.57 (m, 1H), 7.43 – 7.28 (m, 5H), 7.01 (t, *J* = 8.1 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.2, 178.8, 165.9 (d, $^{1}J_{C-F}$ = 255.5 Hz), 152.3, 137.5, 134.9, 134.6, 133.5 (d, $^{3}J_{C-F}$ = 2.8 Hz), 132.2, 131.8 (d, $^{3}J_{C-F}$ = 9.6 Hz), 130.6, 130.4, 129.1, 128.8, 128.5, 128.2, 126.1, 115.8 (d, $^{2}J_{C\text{-}F}$ = 22.1 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -104.34.

***3-(4-chlorobenzoyl)-2-phenyl-4H-thiochromen-4-one (3n)*[1]**



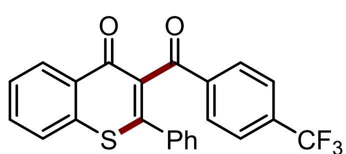
White solid (50.4 mg, 67% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 7.5 Hz, 1H), 7.76 – 7.71 (m, 2H), 7.71 – 7.65 (m, 2H), 7.60 – 7.58 (m, 1H), 7.44 – 7.39 (m, 2H), 7.36 – 7.29 (m, 5H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.6, 178.8, 152.6, 139.9, 137.5, 135.4, 134.7, 134.6, 132.2, 130.6, 130.5, 129.1, 129.0, 128.9, 128.5, 128.3, 126.1.

3-(4-bromobenzoyl)-2-phenyl-4H-thiochromen-4-one (3o)



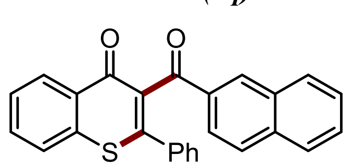
Yellow solid (50.4 mg, 60% yield), mp 159.6 – 161.6 ℃, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 8.8 Hz, 1H), 7.73 – 7.63 (m, 4H), 7.61 – 7.57 (m, 1H), 7.49 (d, *J* = 8.6 Hz, 2H), 7.43 – 7.38 (m, 2H), 7.33 – 7.29 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.8, 178.8, 152.7, 137.5, 135.8, 134.7, 134.6, 132.2, 131.9, 130.59, 130.57, 130.5, 129.1, 128.9, 128.7, 128.5, 128.3, 126.1. HRMS (ESI-TOF) *m/z*: [M + H]$^+$ Calcd for $C_{22}H_{14}BrO_2S$, 420.9892, Found: 420.9898.

2-phenyl-3-(4-(trifluoromethyl)benzoyl)-4H-thiochromen-4-one (3p)



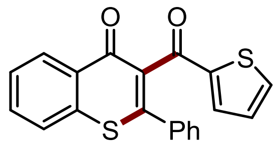
Colorless oil (46.7 mg, 57% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, *J* = 7.4 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 2H), 7.74 – 7.67 (m, 2H), 7.65 – 7.57 (m, 3H), 7.44 – 7.30 (m, 5H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 193.8, 178.9, 153.3, 139.7, 137.5, 134.5 (q, $^{2}J_{C\text{-}F}$ = 32.7 Hz), 134.4, 132.3, 130.59, 130.57, 129.3, 129.0, 128.9, 128.5, 128.4, 126.2, 125.7 (q, $^{2}J_{C\text{-}F}$ = 3.9 Hz), 124.4 (q, $^{1}J_{C\text{-}F}$ = 273.31 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -63.15. HRMS (ESI-TOF) *m/z*: [M + H]$^+$ Calcd for $C_{23}H_{14}F_3O_2S$, 411.0661, Found: 411.0668.

3-(2-naphthoyl)-2-phenyl-4H-thiochromen-4-one (3q)



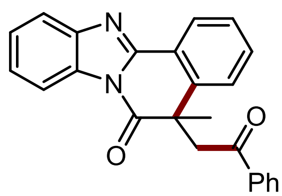
Yellow solid (54.9 mg, 70% yield), mp 162.8 – 164.8 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 (d, *J* = 8.5 Hz, 1H), 8.28 (s, 1H), 7.91 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.88 – 7.77 (m, 3H), 7.71 (d, *J* = 3.8 Hz, 2H), 7.63 – 7.53 (m, 2H), 7.50 – 7.45 (m, 3H), 7.32 – 7.26 (m, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 194.7, 178.9, 152.2, 137.6, 135.8, 135.3, 134.8, 134.5, 132.5, 132.1, 131.5, 130.7, 130.3, 129.7, 129.2, 128.8, 128.6, 128.54, 128.52, 128.2, 127.7, 126.6, 126.1, 124.3. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{26}H_{17}O_2S$, 393.0944, Found: 393.0945.

2-phenyl-3-(thiophene-2-carbonyl)-4H-thiochromen-4-one (3r)



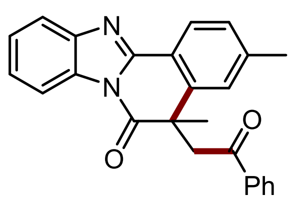
Yellow solid (62.6 mg, 90% yield), mp 149.9 – 151.8 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 (d, *J* = 7.6 Hz, 1H), 7.72 – 7.64 (m, 2H), 7.63 – 7.54 (m, 2H), 7.52 – 7.43 (m, 3H), 7.40 – 7.30 (m, 3H), 6.97 (dd, *J* = 4.9, 3.8 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 186.5, 178.5, 152.3, 144.3, 137.3, 135.0, 134.7, 134.2, 132.1, 130.7, 130.4, 129.2, 128.8, 128.6, 128.2, 128.0, 126.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{13}O_2S_2$, 349.0351, Found: 349.0353.

5-methyl-5-(2-oxo-2-phenylethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5a)[2]



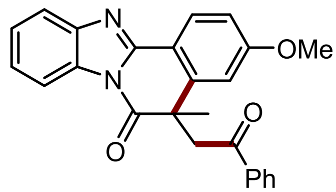
White solid (57.8 mg, 79% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.58 – 8.53 (m, 1H), 8.34 (dd, *J* = 7.5, 2.4 Hz, 1H), 7.97 – 7.73 (m, 3H), 7.57 – 7.50 (m, 1H), 7.48 – 7.37 (m, 6H), 7.35 – 7.30 (m, 1H), 4.30 (d, *J* = 18.3, 1H), 4.15 (d, *J* = 18.2, 1H), 1.72 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.1, 173.3, 150.1, 144.1, 142.0, 135.7, 133.6, 131.7, 131.6, 128.7, 128.1, 127.6, 126.4, 125.6, 125.3, 124.4, 123.1, 119.8, 115.6, 49.3, 46.2, 30.2.

3,5-dimethyl-5-(2-oxo-2-phenylethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5b)[2]



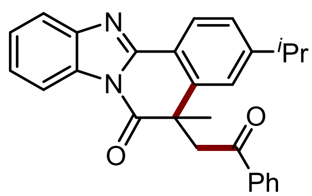
Yellow solid (59.2 mg, 78% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.43 (d, *J* = 8.0 Hz, 1H), 8.34 – 8.28 (m, 1H), 7.89 – 7.83 (m, 3H), 7.58 – 7.53 (m, 1H), 7.46 – 7.36 (m, 4H), 7.25 – 7.23 (m, 1H), 7.11 (s, 1H), 4.31 (d, *J* = 18.3 Hz, 1H), 4.12 (d, *J* = 18.3 Hz, 1H), 2.37 (s, 3H), 1.71 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.0, 173.4, 150.3, 144.2, 142.1, 142.0, 135.8, 133.6, 131.7, 128.7, 128.6, 128.1, 126.4, 125.5, 125.1, 124.9, 120.4, 119.6, 115.6, 49.3, 46.1, 30.3, 22.0.

3-methoxy-5-methyl-5-(2-oxo-2-phenylethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5c)[2]



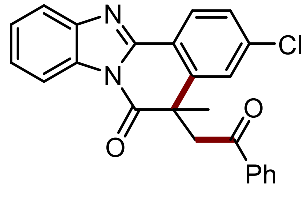
Yellow solid (59.4 mg, 75% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.50 (d, *J* = 8.7 Hz, 1H), 8.30 (d, *J* = 7.2 Hz, 1H), 7.88 – 7.80 (m, 3H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.43 – 7.34 (m, 4H), 6.97 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.81 (d, *J* = 2.4 Hz, 1H), 4.29 (d, *J* = 18.3 Hz, 1H), 4.08 (d, *J* = 18.2 Hz, 1H), 3.80 (s, 3H), 1.70 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.0, 173.3, 162.3, 150.2, 144.2, 144.1, 135.7, 133.6, 131.6, 128.7, 128.4, 128.1, 125.5, 124.9, 119.4, 116.0, 115.5, 112.4, 111.1, 55.5, 49.3, 46.3, 30.4.

3-isopropyl-5-methyl-5-(2-oxo-2-phenylethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5d)[2]



Yellow solid (62.8 mg, 77% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.45 (d, *J* = 8.1 Hz, 1H), 8.33 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.87 – 7.87 (m, 3H), 7.57 – 7.52 (m, 1H), 7.45 – 7.37 (m, 4H), 7.31 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.14 (d, *J* = 1.6 Hz, 1H), 4.28 (d, *J* = 18.1 Hz, 1H), 4.15 (d, *J* = 18.1 Hz, 1H), 2.94 – 2.84 (m, 1H), 1.72 (s, 3H), 1.20 (dd, *J* = 6.9, 1.8 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.3, 173.5, 152.9, 150.3, 144.2, 141.9, 135.9, 133.6, 131.7, 128.6, 128.0, 126.5, 125.8, 125.5, 125.1, 122.5, 120.8, 119.6, 115.6, 49.2, 46.3, 34.4, 30.4, 23.8, 23.7.

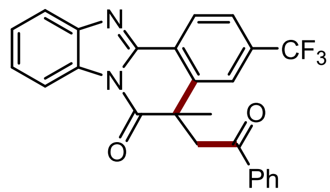
3-chloro-5-methyl-5-(2-oxo-2-phenylethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5e)[2]



Yellow solid (52 mg, 65% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.49 (d, *J* = 8.4 Hz, 1H), 8.35 – 8.28 (m, 1H), 7.85 (dd, *J* = 8.3, 1.3 Hz, 3H), 7.58 – 7.51 (m, 1H), 7.48 – 7.39 (m, 5H), 7.30 (d, *J* = 2.0 Hz, 1H), 4.33 (d, *J* = 18.4 Hz, 1H), 4.05 (d, *J* = 18.3 Hz, 1H), 1.71 (s, 3H). ^{13}C

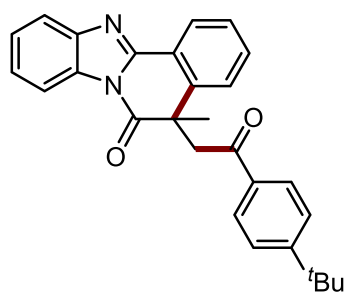
NMR (101 MHz, Chloroform-*d*) δ 195.9, 172.6, 149.2, 144.1, 143.7, 137.7, 135.5, 133.8, 131.6, 128.7, 128.2, 128.1, 127.8, 125.8, 125.6, 124.8, 121.8, 119.9, 115.6, 49.4, 46.2, 30.0.

5-methyl-5-(2-oxo-2-phenylethyl)-3-(trifluoromethyl)benzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5f)[2]



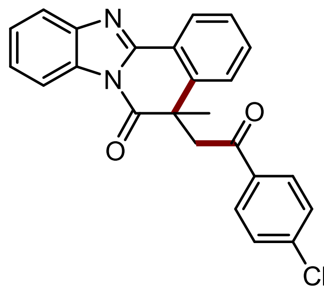
White solid (52.0 mg, 60% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.69 (d, J = 8.2 Hz, 1H), 8.40 – 8.28 (m, 1H), 7.95 – 7.88 (m, 1H), 7.88 – 7.80 (m, 2H), 7.70 (d, J = 8.3 Hz, 1H), 7.60 – 7.52 (m, 2H), 7.51 – 7.39 (m, 4H), 4.38 (d, J = 18.4 Hz, 1H), 4.13 (d, J = 18.4 Hz, 1H), 1.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.0, 172.4, 148.6, 144.0, 142.6, 135.4, 133.9, 133.0 (q, $^2J_{C\text{-}F}$ = 32.7 Hz), 131.7, 128.7, 128.1, 127.0, 126.4, 126.1, 126.0, 124.52 (q, $^3J_{C\text{-}F}$ = 3.7 Hz), 123.6 (q, $^1J_{C\text{-}F}$ = 272.8 Hz), 121.45 (q, $^3J_{C\text{-}F}$ = 3.9 Hz), 120.2, 115.8, 49.5, 46.4, 30.0. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.75.

5-(2-(4-(tert-butyl)phenyl)-2-oxoethyl)-5-methylbenzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5g)



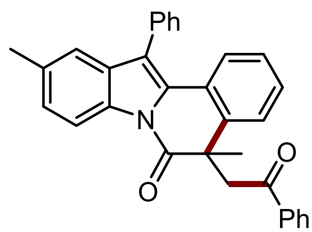
White solid (53.1 mg, 63% yield), mp 83.2 – 85.1 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.58 – 8.50 (m, 1H), 8.38 – 8.30 (m, 1H), 7.90 – 7.83 (m, 1H), 7.82 – 7.76 (m, 2H), 7.46 – 7.37 (m, 6H), 7.36 – 7.28 (m, 1H), 4.28 (d, J = 18.2 Hz, 1H), 4.13 (d, J = 18.1 Hz, 1H), 1.71 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.7, 173.3, 157.5, 150.1, 144.1, 142.1, 133.2, 131.7, 131.5, 128.0, 127.5, 126.4, 125.59, 125.57, 125.3, 124.4, 123.1, 119.8, 115.7, 49.3, 46.2, 35.2, 31.0, 30.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{28}H_{27}N_2O_2$, 423.2067, Found: 423.2071.

5-(2-(4-chlorophenyl)-2-oxoethyl)-5-methylbenzo[4,5]imidazo[2,1-a]isoquinolin-6(5H)-one (5h)[3]



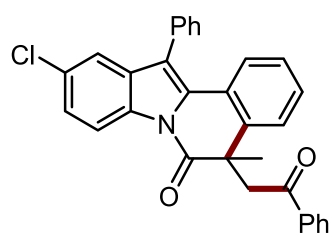
White solid (40 mg, 50% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.59 – 8.51 (m, 1H), 8.38 – 8.28 (m, 1H), 7.89 – 7.84 (m, 1H), 7.81 – 7.75 (m, 2H), 7.48 – 7.36 (m, 6H), 7.34 – 7.28 (m, 1H), 4.27 (d, *J* = 18.2 Hz, 1H), 4.08 (d, *J* = 18.2 Hz, 1H), 1.72 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.9, 173.2, 150.0, 144.1, 141.8, 140.2, 134.0, 131.7, 131.6, 129.5, 129.0, 127.6, 126.4, 125.7, 125.4, 124.3, 123.1, 119.8, 115.6, 49.2, 46.2, 30.2.

5,10-dimethyl-5-(2-oxo-2-phenylethyl)-12-phenylindolo[2,1-a]isoquinolin-6(5H)-one (5i)[2]



White solid (70.9 mg, 78% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.51 (d, *J* = 8.3 Hz, 1H), 7.97 – 7.90 (m, 2H), 7.73 – 7.36 (m, 9H), 7.23 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.17 – 7.09 (m, 2H), 6.99 – 6.95 (m, 1H), 4.36 (d, *J* = 18.2 Hz, 1H), 4.10 (d, *J* = 18.1 Hz, 1H), 2.42 (s, 3H), 1.73 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.4, 173.1, 139.3, 136.3, 134.6, 134.0, 133.3, 132.6, 132.5, 130.4, 129.9, 129.2, 128.6, 128.2, 128.1, 127.9, 127.0, 126.4, 125.7, 125.4, 124.2, 120.0, 119.2, 116.3, 48.3, 45.8, 30.7, 21.5.

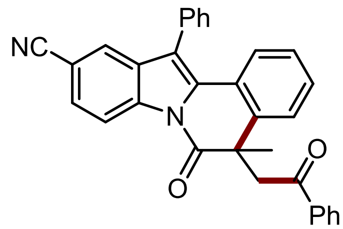
10-chloro-5-methyl-5-(2-oxo-2-phenylethyl)-12-phenylindolo[2,1-a]isoquinolin-6(5H)-one (5j)[2]



White solid (61.7 mg, 65% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.55 (d, *J* = 8.7 Hz, 1H), 7.95 – 7.85 (m, 2H), 7.67 – 7.37 (m, 10H), 7.34 (dd, *J* = 8.7, 2.1 Hz, 1H), 7.29 (d, *J* = 2.1 Hz, 1H), 7.24 (d, *J* = 6.8 Hz, 1H), 7.20 – 7.14 (m, 1H), 6.98 (td, *J* = 7.7, 7.3, 1.3 Hz, 1H), 4.34 (d, *J* = 18.1 Hz, 1H), 4.11 (d, *J* = 18.1 Hz, 1H), 1.73 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.4, 173.3, 139.5, 136.1, 133.73, 133.71, 133.5, 132.7, 131.1, 130.3, 130.0, 129.4, 128.7, 128.6, 128.3, 128.1, 126.6, 125.9, 125.7, 124.9, 124.3, 119.3, 118.9, 117.7, 48.6, 45.8, 30.6.

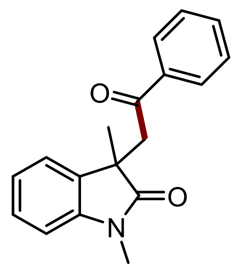
10-isocyano-5-methyl-5-(2-oxo-2-phenylethyl)-12-phenylindolo[2,1-a]isoquinolin-6(5H)-one

***(5k)*[2]**



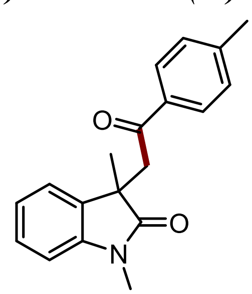
White solid (51.2 mg, 55% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.71 (d, *J* = 8.5 Hz, 1H), 7.94 – 7.86 (m, 2H), 7.77 – 7.49 (m, 10H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.29 – 7.18 (m, 2H), 7.03 – 6.99 (m, 1H), 4.34 (d, *J* = 18.2 Hz, 1H), 4.15 (d, *J* = 18.2 Hz, 1H), 1.74 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.4, 173.7, 139.4, 136.2, 135.9, 133.6, 133.1, 132.5, 132.0, 130.2, 129.5, 129.2, 128.7, 128.6, 128.1, 126.8, 126.1, 124.4, 124.1, 119.7, 119.2, 117.4, 107.7, 48.8, 45.9, 30.5.

***1,3-dimethyl-3-(2-oxo-2-phenylethyl)indolin-2-one (7a)*[4]**



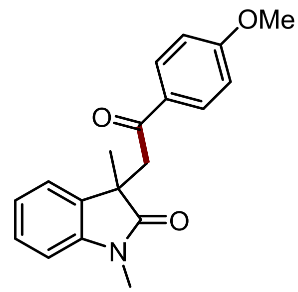
Colorless oil (34.1 mg, 61% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.76 (m, 2H), 7.55 – 7.49 (m, 1H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.29 – 7.22 (m, 1H), 7.14 (d, *J* = 7.4 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 3.68 (q, *J* = 16.0 Hz, 2H), 3.31 (s, 3H), 1.44 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.1, 180.6, 143.8, 136.4, 133.7, 133.2, 128.5, 128.0, 127.8, 122.2, 121.8, 108.2, 46.0, 45.3, 26.5, 24.9.

***1,3-dimethyl-3-(2-oxo-2-(p-tolyl)ethyl)indolin-2-one (7b)*[4]**



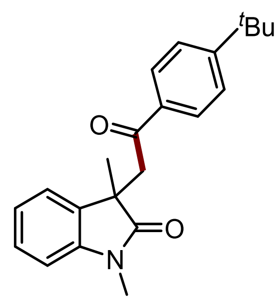
Colorless oil (29.2 mg, 50% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 8.2 Hz, 2H), 7.24 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.4 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 7.7 Hz, 1H), 3.65 (q, *J* = 16.2 Hz, 2H), 3.31 (s, 3H), 2.37 (s, 3H), 1.43 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 195.7, 180.7, 144.0, 143.9, 134.0, 133.8, 129.2, 128.1, 127.8, 122.1, 121.8, 108.1, 45.9, 45.3, 26.5, 24.9, 21.6.

***3-(2-(4-methoxyphenyl)-2-oxoethyl)-1,3-dimethylindolin-2-one (7c)*[4]**



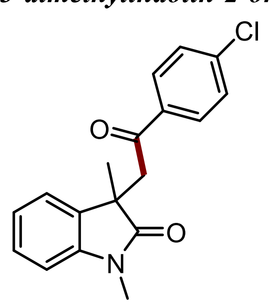
Colorless oil (37.3 mg, 60% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.8 Hz, 2H), 7.23 (d, *J* = 7.7 Hz, 1H), 7.13 (d, *J* = 7.3 Hz, 1H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.93 – 6.82 (m, 3H), 3.83 (s, 3H), 3.63 (q, *J* = 16.1 Hz, 2H), 3.31 (s, 3H), 1.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.6, 180.7, 163.5, 143.8, 133.9, 130.2, 129.6, 127.8, 122.1, 121.8, 113.6, 108.1, 55.4, 45.7, 45.4, 26.4, 24.9.

3-(2-(4-(tert-butyl)phenyl)-2-oxoethyl)-1,3-dimethylindolin-2-one (7d)[4]



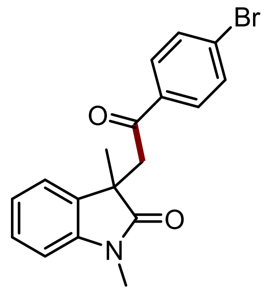
Colorless oil (40.1 mg, 60% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 – 7.73 (m, 2H), 7.43 – 7.35 (m, 2H), 7.24 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.13 – 7.11 (m, 1H), 6.96 (td, *J* = 7.5, 1.0 Hz, 1H), 6.89 (d, *J* = 7.7 Hz, 1H), 3.67 (q, *J* = 16.3 Hz, 2H), 3.31 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.8, 180.7, 156.9, 143.8, 133.84, 133.83, 127.9, 127.8, 125.4, 122.1, 121.7, 108.1, 45.9, 45.3, 35.1, 31.0, 26.5, 24.9.

3-(2-(4-chlorophenyl)-2-oxoethyl)-1,3-dimethylindolin-2-one (7e)[4]



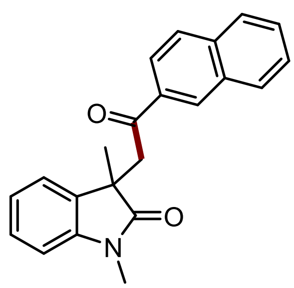
Colorless oil (24.8 mg, 40% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.70 (m, 2H), 7.40 – 7.32 (m, 2H), 7.28 – 7.23 (m, 1H), 7.13 (dd, *J* = 7.5, 1.2 Hz, 1H), 6.98 (td, *J* = 7.5, 1.0 Hz, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 3.63 (q, *J* = 16.0 Hz, 2H), 3.30 (s, 3H), 1.44 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.9, 180.4, 143.8, 139.7, 134.7, 133.5, 129.4, 128.8, 127.9, 122.2, 121.8, 108.2, 46.0, 45.3, 26.5, 24.9.

***3-(2-(4-bromophenyl)-2-oxoethyl)-1,3-dimethylindolin-2-one (7f)*[5]**



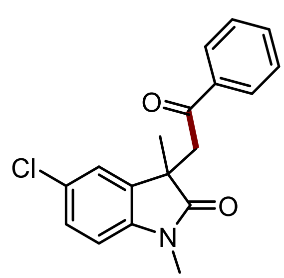
Colorless oil (30.0 mg, 42% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.66 (m, 2H), 7.56 – 7.51 (m, 2H), 7.29 – 7.23 (m, 1H), 7.14 – 7.12 (m, 1H), 6.98 (td, *J* = 7.5, 1.0 Hz, 1H), 6.90 (d, *J* = 7.8 Hz, 1H), 3.63 (q, *J* = 16.0 Hz, 2H), 3.30 (s, 3H), 1.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.1, 180.4, 143.8, 135.1, 133.5, 131.8, 129.5, 128.4, 127.9, 122.2, 121.8, 108.2, 45.9, 45.3, 26.5, 24.9.

***1,3-dimethyl-3-(2-(naphthalen-2-yl)-2-oxoethyl)indolin-2-one (7g)*[5]**



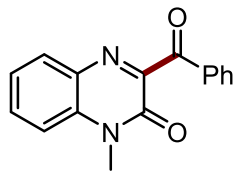
Colorless oil (26.3 mg, 53% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.39 (s, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.88 – 7.78 (m, 3H), 7.60 – 7.51 (m, 2H), 7.29 – 7.23 (m, 1H), 7.19 – 7.17 (m, 1H), 6.98 (td, *J* = 7.5, 1.0 Hz, 1H), 6.91 (d, *J* = 7.8 Hz, 1H), 3.82 (q, *J* = 16.3 Hz, 2H), 3.32 (s, 3H), 1.49 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.1, 180.7, 143.9, 135.6, 133.8, 133.7, 132.4, 129.7, 129.5, 128.5, 128.3, 127.9, 127.7, 126.8, 123.7, 122.2, 121.8, 108.2, 46.1, 45.4, 26.5, 24.9.

***5-chloro-1,3-dimethyl-3-(2-oxo-2-phenylethyl)indolin-2-one (7h)*[4]**



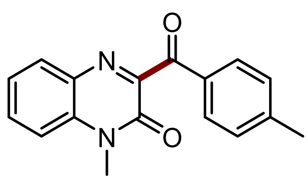
Colorless oil (36.0 mg, 58% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.80 (m, 2H), 7.59 – 7.50 (m, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.23 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.10 (d, *J* = 2.1 Hz, 1H), 6.82 (d, *J* = 8.3 Hz, 1H), 3.69 (s, 2H), 3.30 (s, 3H), 1.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.8, 180.1, 142.5, 136.1, 135.6, 133.4, 128.6, 128.0, 127.7, 127.5, 122.3, 109.0, 46.1, 45.4, 26.6, 24.8.

3-benzoyl-1-methylquinoxalin-2(1H)-one (9a)[6]



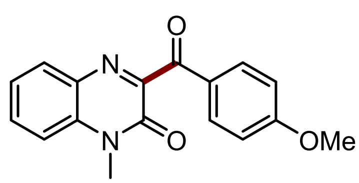
White solid (36.9 mg, 70% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.96 (m, 2H), 7.93 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.70 – 7.65 (m, 1H), 7.63 – 7.60 (m, 1H), 7.51 – 7.45 (m, 2H), 7.44 – 7.38 (m, 2H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.8, 154.7, 153.3, 134.9, 134.2, 133.9, 132.2, 132.1, 131.0, 130.0, 128.7, 124.2, 114.0, 29.1.

1-methyl-3-(4-methylbenzoyl)quinoxalin-2(1H)-one (9b)[6]



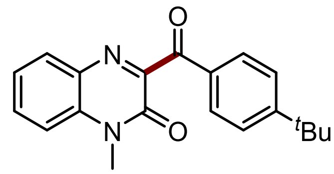
White solid (31.1 mg, 56% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.91 – 7.83 (m, 2H), 7.70 – 7.65 (m, 1H), 7.43 – 7.39 (m, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 3.75 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.4, 155.0, 153.4, 145.3, 133.9, 132.5, 132.3, 131.9, 131.0, 130.1, 129.4, 124.1, 113.9, 29.0, 21.9.

3-(4-methoxybenzoyl)-1-methylquinoxalin-2(1H)-one (9c)[6]



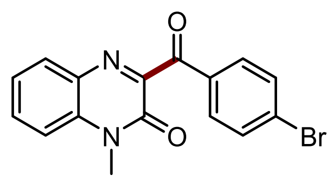
White solid (35.2 mg, 60% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.94 (m, 2H), 7.93 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.69 – 7.65 (m, 1H), 7.46 – 7.36 (m, 2H), 6.99 – 6.91 (m, 2H), 3.88 (s, 3H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.2, 164.5, 155.0, 153.4, 133.9, 132.5, 132.2, 131.9, 130.9, 128.0, 124.1, 114.0, 113.9, 55.6, 29.1.

3-(4-(tert-butyl)benzoyl)-1-methylquinoxalin-2(1H)-one (9d)



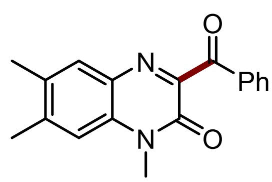
Yellow solid (35.2 mg, 55% yield), mp 170.3 – 172.1 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.91 (m, 1H), 7.89 – 7.82 (m, 2H), 7.72 – 7.66 (m, 1H), 7.65 – 7.60 (m, 2H), 7.45 – 7.40 (m, 2H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.6, 153.9, 153.3, 134.0, 133.7, 132.3, 132.2, 132.0, 131.8, 131.6, 131.4, 131.1, 129.6, 124.3, 114.0, 29.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{21}N_2O_2$, 321.1598, Found: 321.1603.

3-(4-bromobenzoyl)-1-methylquinoxalin-2(1H)-one (9e)[6]



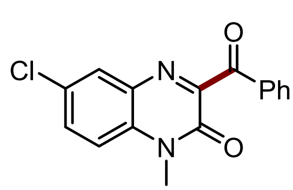
White solid (30.7 mg, 45% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.91 (m, 1H), 7.88 – 7.82 (m, 2H), 7.72 – 7.66 (m, 1H), 7.66 – 7.60 (m, 2H), 7.45 – 7.40 (m, 2H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.6, 153.9, 153.3, 134.0, 133.7, 132.3, 132.2, 132.0, 131.4, 131.1, 129.6, 124.3, 114.0, 29.1.

3-benzoyl-1,6,7-trimethylquinoxalin-2(1H)-one (9f)



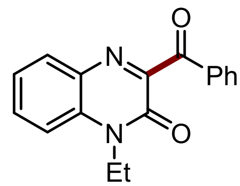
Reddish brown oil (35.5 mg, 61% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.91 (m, 2H), 7.66 (s, 1H), 7.63 – 7.57 (m, 1H), 7.49 – 7.43 (m, 2H), 7.16 (s, 1H), 3.72 (s, 3H), 2.46 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.0, 153.4, 153.3, 142.5, 135.1, 134.0, 133.3, 132.0, 130.9, 130.7, 130.0, 128.6, 114.5, 29.0, 20.8, 19.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{18}H_{17}N_2O_2$, 293.1285, Found: 293.1289.

3-benzoyl-6-chloro-1-methylquinoxalin-2(1H)-one (9g)[6]



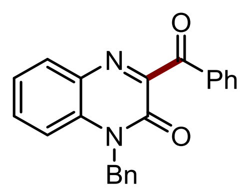
White solid (44.1 mg, 74% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.95 (m, 2H), 7.92 (d, *J* = 2.4 Hz, 1H), 7.67 – 7.60 (m, 2H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.35 (d, *J* = 9.0 Hz, 1H), 3.74 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.2, 155.9, 153.0, 134.6, 134.4, 132.7, 132.6, 132.0, 130.2, 130.0, 129.6, 128.7, 115.1, 29.3.

3-benzoyl-1-ethylquinoxalin-2(1H)-one (9h)[6]



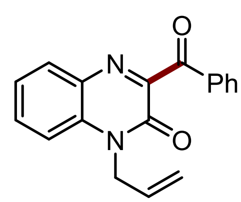
White solid (40.0 mg, 72% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.97 (m, 2H), 7.94 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.70 – 7.59 (m, 2H), 7.44 – 7.38 (m, 2H), 7.44 – 7.38 (m, 2H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.8, 154.7, 152.8, 134.9, 134.2, 132.9, 132.5, 132.0, 131.3, 130.0, 128.7, 124.0, 113.8, 37.4, 12.5.

3-benzoyl-1-benzylquinoxalin-2(1H)-one (9i)[6]



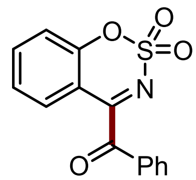
White solid (42.8 mg, 63% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.94 – 7.92 (m, 1H), 7.68 – 7.59 (m, 1H), 7.57 – 7.47 (m, 3H), 7.39 – 7.27 (m, 7H), 5.53 (s, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.7, 154.8, 153.5, 134.90, 134.85, 134.3, 133.3, 132.5, 132.0, 131.1, 130.0, 129.0, 128.7, 127.9, 127.1, 124.2, 114.8, 45.9.

1-allyl-3-benzoylquinoxalin-2(1H)-one (9j)[6]



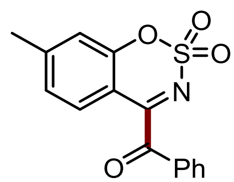
Pale yellow solid (37.5 mg, 64% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.89 (m, 3H), 7.69 – 7.57 (m, 2H), 7.49 (dd, *J* = 8.4, 7.2 Hz, 2H), 7.43 – 7.35 (m, 2H), 6.00 – 5.91 (m, 1H), 5.36 – 5.20 (m, 2H), 4.95 (dt, *J* = 5.4, 1.7 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.7, 154.7, 152.9, 134.9, 134.2, 133.2, 132.4, 131.9, 131.1, 130.3, 130.0, 128.7, 124.2, 118.8, 114.5, 44.5.

(2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)(phenyl)methanone (9k)



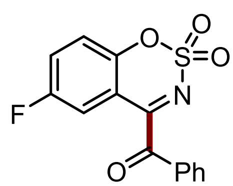
Colorless oil (30.4 mg, 53% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.09 – 8.01 (m, 2H), 7.82 – 7.71 (m, 2H), 7.63 – 7.54 (m, 3H), 7.42 – 7.33 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 187.8, 171.6, 155.0, 138.1, 135.9, 133.1, 130.5, 129.9, 129.3, 126.3, 119.4, 114.1. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{14}H_{10}NO_4S$, 288.0325, Found: 288.0327.

(7-methyl-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)(phenyl)methanone (9l)



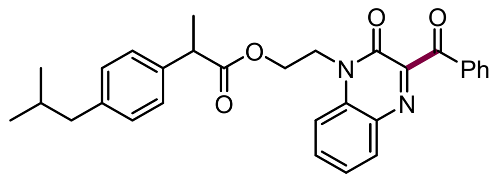
White solid (30.7 mg, 51% yield), mp 138.3 – 140.2 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 8.01 (m, 2H), 7.76 – 7.69 (m, 1H), 7.56 (t, *J* = 7.9 Hz, 2H), 7.47 (d, *J* = 8.1 Hz, 1H), 7.20 (s, 1H), 7.17 – 7.12 (m, 1H), 2.51 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 187.9, 171.5, 155.1, 151.1, 135.7, 133.2, 130.5, 129.7, 129.3, 127.3, 119.5, 111.8, 22.4. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_{12}NO_4S$, 302.0482, Found: 302.0485.

(6-fluoro-2,2-dioxidobenzo[e][1,2,3]oxathiazin-4-yl)(phenyl)methanone (9m)



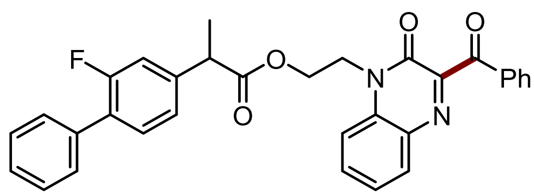
Yellow oil (30.7 mg, 51% yield), mp 112.2 – 114.1 °C, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.79 – 7.72 (m, 1H), 7.62 – 7.56 (m, 2H), 7.53 – 7.49 (m, 1H), 7.41 (dd, *J* = 9.1, 4.2 Hz, 1H), 7.35 (dd, *J* = 7.6, 2.9 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 187.3, 170.1, 159.0 (d, $^{1}J_{C-F}$ = 249.7 Hz), 151.0, 136.0, 132.8, 130.6, 129.4, 125.4 (d, $^{2}J_{C-F}$ = 24.3 Hz), 121.2 (d, $^{1}J_{C-F}$ = 8.1 Hz), 115.9 (d, $^{2}J_{C-F}$ = 25.3 Hz), 114.6 (d, $^{3}J_{C-F}$ = 8.3 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.15. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $C_{14}H_9FNO_4S$, 306.0231, Found: 306.0233.

2-(3-benzoyl-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(4-isobutylphenyl)propanoate (9n)



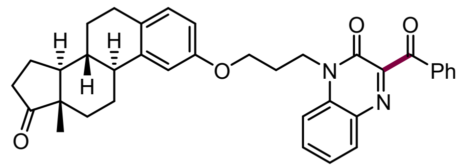
Reddish brown oil (62.3 mg, 65% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.94 (m, 2H), 7.92 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.65 – 7.55 (m, 2H), 7.51 – 7.44 (m, 3H), 7.41 – 7.37 (m, 1H), 7.11 – 7.01 (m, 4H), 4.61 – 4.36 (m, 4H), 3.59 (q, *J* = 7.2 Hz, 1H), 2.43 (d, *J* = 7.1 Hz, 2H), 1.89 – 1.76 (m, 1H), 1.40 (d, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.6 Hz, 7H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.6, 174.7, 154.4, 153.2, 140.7, 137.1, 134.8, 134.3, 133.5, 132.3, 132.1, 131.2, 130.0, 129.4, 128.7, 127.1, 124.2, 114.2, 61.0, 45.03, 44.97, 41.0, 30.2, 22.4, 18.4. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{30}H_{30}N_2NaO_4$, 505.2098, Found: 505.2097.

2-(3-benzoyl-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (9o)



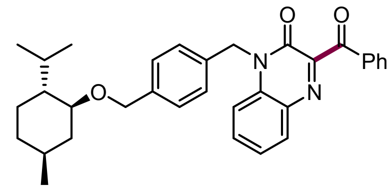
Yellow oil (52 mg, 50% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 – 7.95 (m, 2H), 7.91 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.64 – 7.57 (m, 2H), 7.53 – 7.30 (m, 10H), 7.04 – 6.94 (m, 2H), 4.63 – 4.43 (m, 4H), 3.64 (q, *J* = 7.2 Hz, 1H), 1.45 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.5, 174.0, 154.4, 153.2, 141.1 (d, $^3J_{C-F}$ = 7.5 Hz), 135.3, 134.8, 133.4, 133.2 (d, $^1J_{C-F}$ = 229.9 Hz), 132.3, 130.9 (d, $^3J_{C-F}$ = 4.0 Hz), 130.1, 128.9 (d, $^3J_{C-F}$ = 3.0 Hz), 128.7, 128.5, 127.7, 128.0, 127.9, 115.2 (d, $^2J_{C-F}$ = 23.8 Hz), 114.0, 61.3, 44.9, 41.0, 18.2. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.28. HRMS (ESI-TOF) *m/z*: [M + Na]$^+$ Calcd for $C_{32}H_{25}FN_2NaO_4$, 543.1691, Found: 543.1693.

3-benzoyl-1-(3-(((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)oxy)propyl)quinoxalin-2(1H)-one (9p)



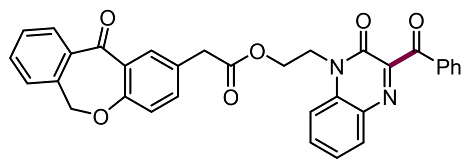
Yellow oil (61.6 mg, 55% yield), ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.91 (m, 3H), 7.66 – 7.56 (m, 3H), 7.49 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 7.21 (d, *J* = 8.6 Hz, 1H), 6.73 (dd, *J* = 8.6, 2.8 Hz, 1H), 6.65 (d, *J* = 2.7 Hz, 1H), 4.59 – 4.46 (m, 2H), 4.09 (t, *J* = 5.7 Hz, 2H), 2.96 – 2.83 (m, 2H), 2.51 (dd, *J* = 18.8, 8.6 Hz, 1H), 2.43 – 2.37 (m, 1H), 2.32 – 2.25 (m, 3H), 2.20 – 2.11 (m, 1H), 2.09 – 1.93 (m, 3H), 1.67 – 1.40 (m, 6H), 0.91 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.8, 156.5, 154.6, 153.2, 137.9, 134.9, 134.2, 133.2, 132.54, 132.46, 132.1, 131.2, 130.0, 128.7, 126.5, 124.1, 114.5, 114.1, 112.2, 65.2, 50.4, 48.0, 44.0, 39.9, 38.4, 35.9, 31.6, 29.7, 27.3, 26.5, 25.9, 21.6, 13.9. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{36}H_{36}N_2NaO_4$, 583.2567, Found: 583.2566.

3-benzoyl-1-(4-((((1S,2R,5S)-2-isopropyl-5-methylcyclohexyl)oxy)methyl)benzyl)quinoxalin-2(1H)-one (9q)



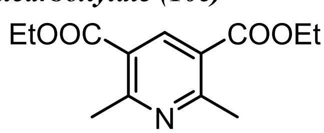
Yellow oil (56.9 mg, 56% yield), ^1H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 7.97 (m, 2H), 7.92 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.67 – 7.61 (m, 1H), 7.54 – 7.48 (m, 3H), 7.40 – 7.26 (m, 6H), 5.52 (s, 2H), 4.62 (d, *J* = 11.5 Hz, 1H), 4.35 (d, *J* = 11.5 Hz, 1H), 3.14 (td, *J* = 10.5, 4.1 Hz, 1H), 2.29 – 2.21 (m, 1H), 2.18 – 2.14 (m, 1H), 1.67 – 1.58 (m, 2H), 1.30 – 1.23 (m, 2H), 0.97 – 0.82 (m, 9H), 0.68 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.7, 154.8, 153.5, 139.0, 134.9, 134.3, 134.0, 133.2, 132.5, 132.0, 131.1, 130.0, 128.7, 128.5, 127.1, 124.2, 114.9, 78.9, 70.0, 48.3, 45.7, 40.3, 34.5, 31.6, 25.5, 23.2, 22.4, 21.0, 16.1. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{33}H_{36}N_2NaO_3$, 531.2618, Found: 531.2626.

2-(3-benzoyl-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl)acetate (9r)



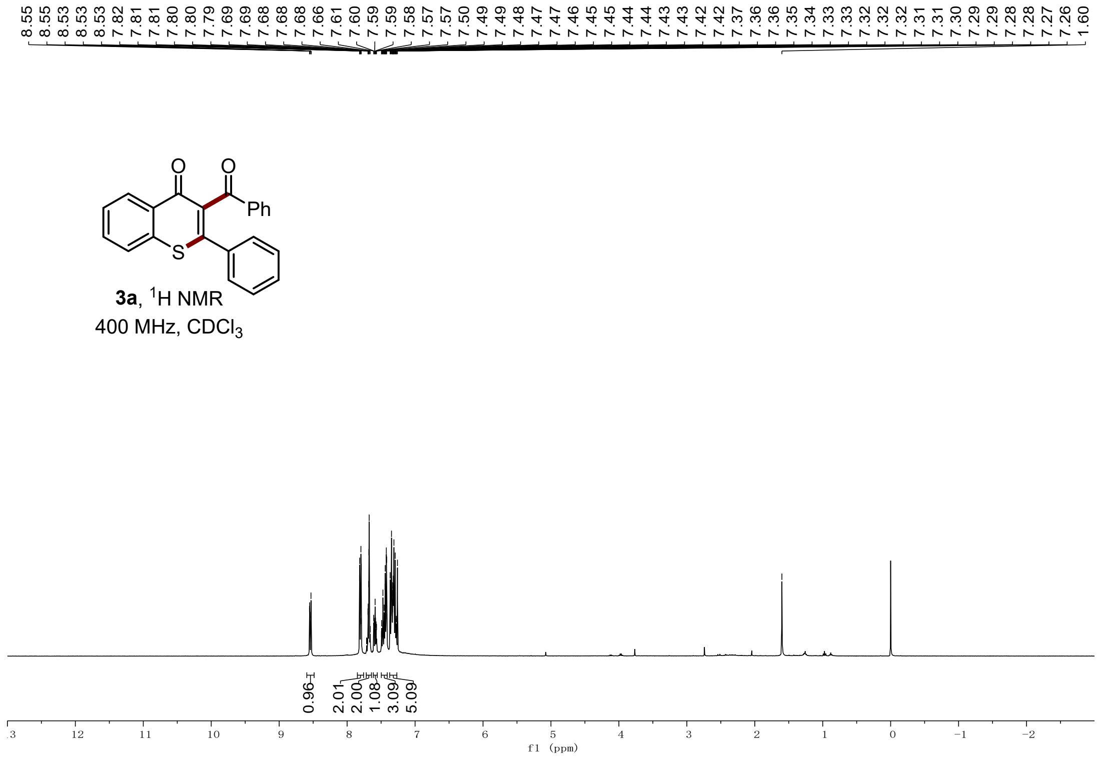
Yellow oil (48.9 mg, 45% yield), ^1H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.97 (m, 3H), 7.90 (td, *J* = 8.2, 1.4 Hz, 2H), 7.67 – 7.55 (m, 3H), 7.52 – 7.44 (m, 4H), 7.40 – 7.34 (m, 2H), 7.30 (dd, *J* = 8.5, 2.4 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 1H), 5.18 (s, 2H), 4.62 – 4.48 (m, 4H), 3.56 (s, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.6, 190.8, 171.4, 160.6, 154.4, 153.3, 140.4, 136.3, 135.5, 134.8, 134.3, 133.4, 132.9, 132.44, 132.36, 132.2, 131.3, 130.1, 129.5, 129.3, 128.7, 127.9, 127.1, 125.1, 124.3, 121.2, 113.9, 73.6, 61.1, 41.1, 39.9. HRMS (ESI-TOF) *m/z*: $[M + Na]^+$ Calcd for $C_{33}H_{24}N_2NaO_6$, 567.1527, Found: 567.1531.

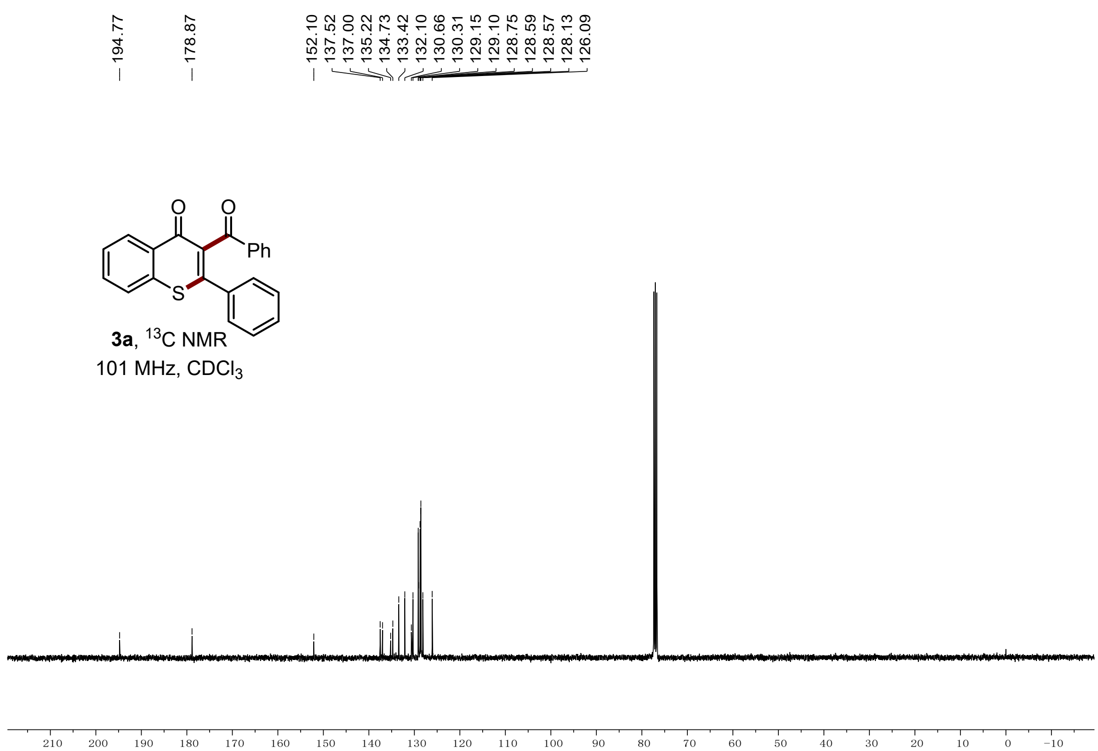
Diethyl 2,6-dimethylpyridine-3,5-dicarboxylate (10c)[7]

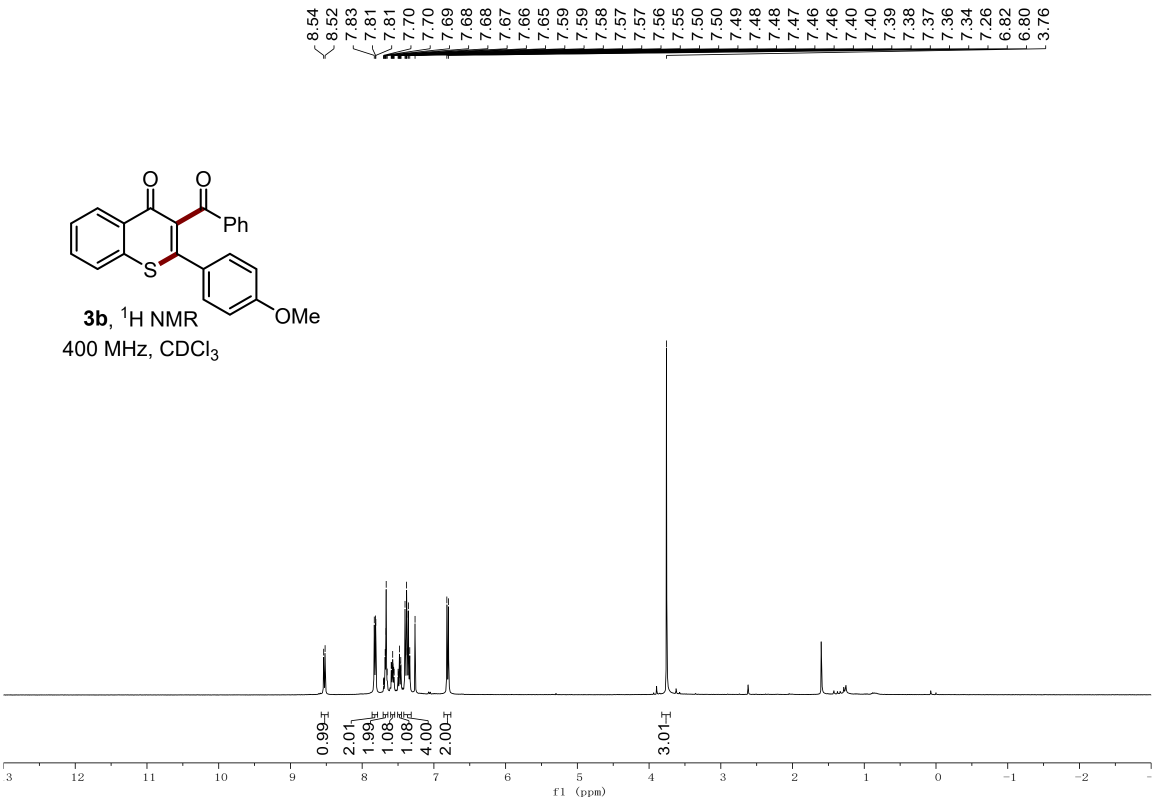


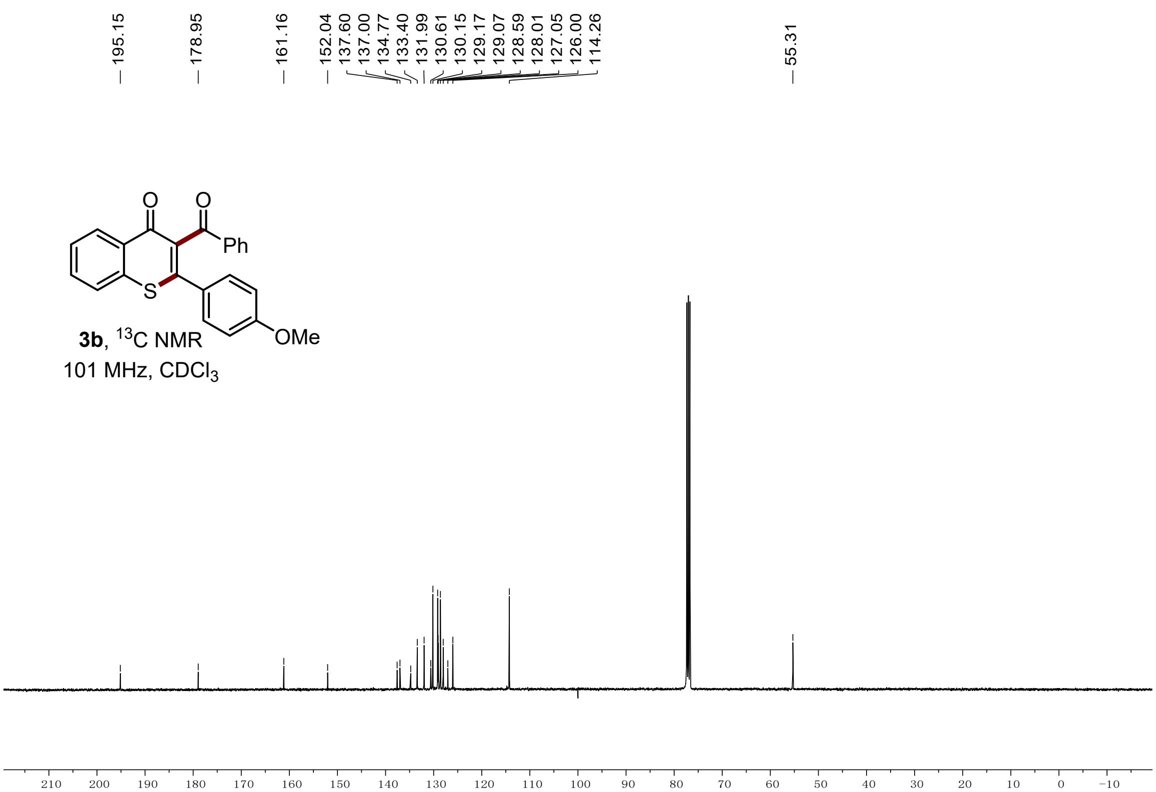
Colorless oil, ^1H NMR (400 MHz, Chloroform-*d*) δ 8.68 (s, 1H), 4.40 (q, *J* = 7.1 Hz, 4H), 2.85 (s, 6H), 1.42 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 165.9, 162.2, 140.9, 123.0, 61.4, 24.9, 14.3.

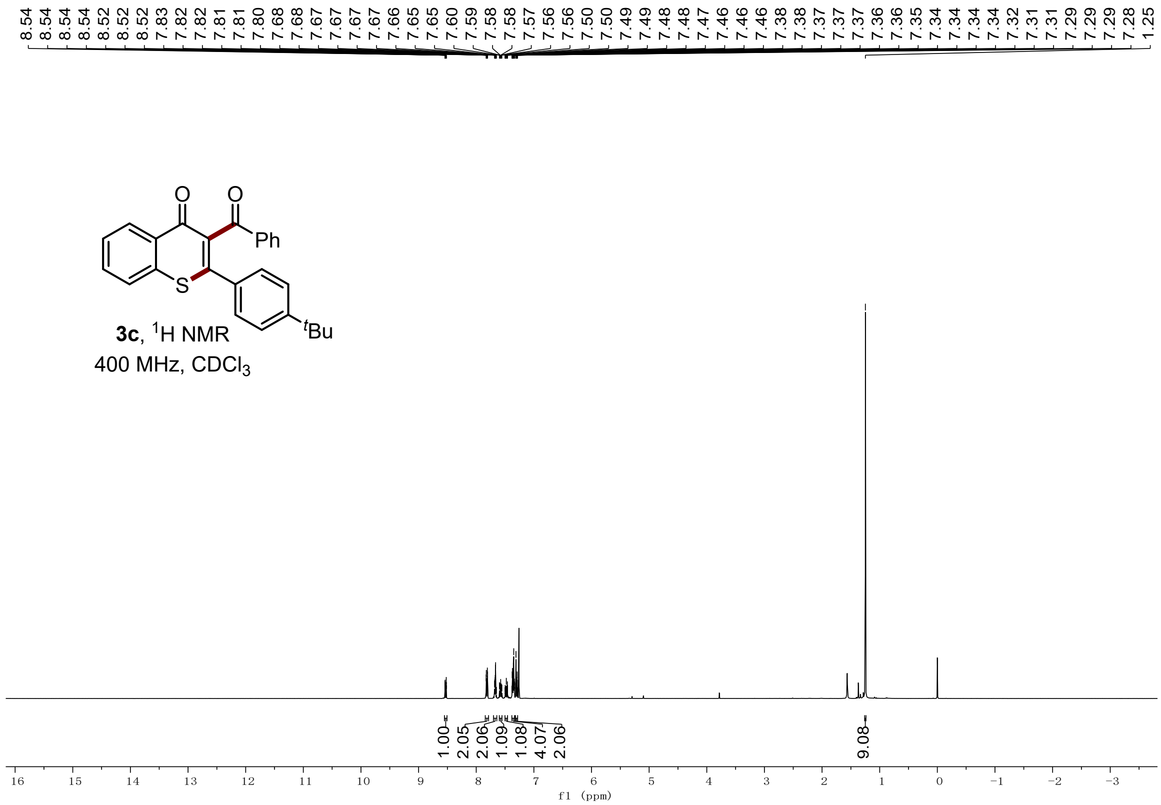
4. NMR Copies of Products

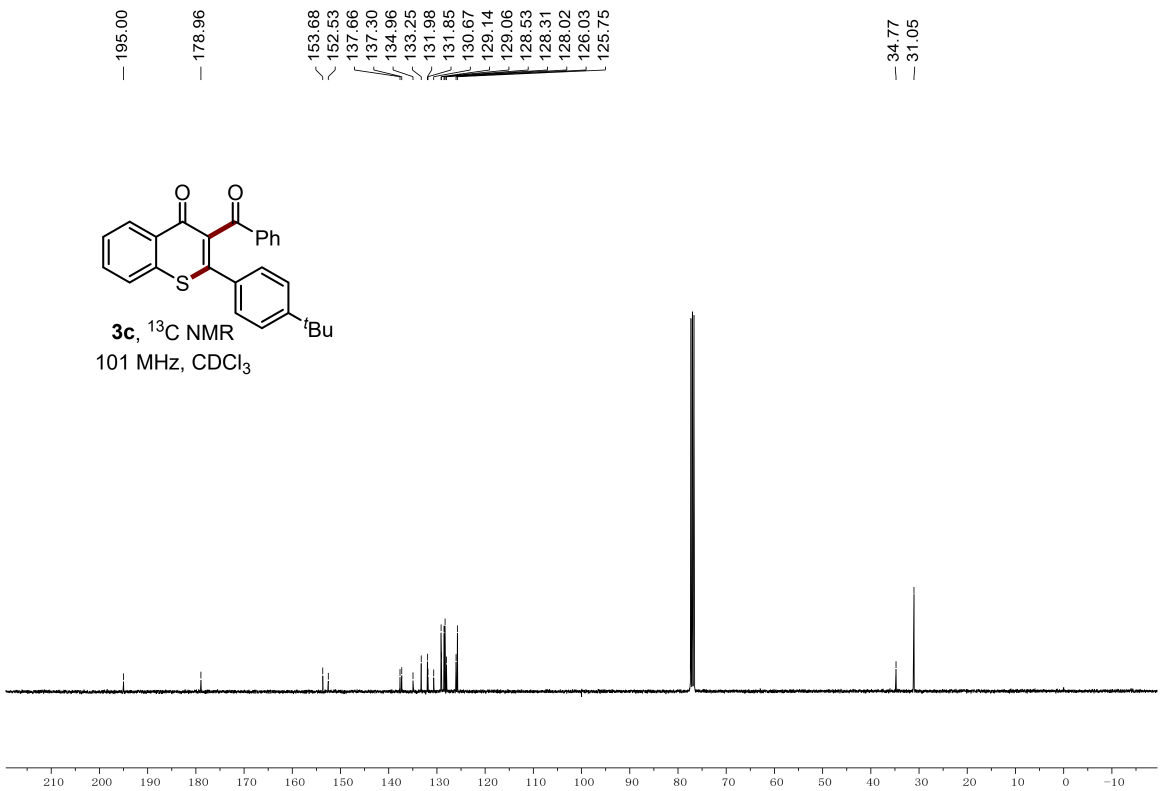


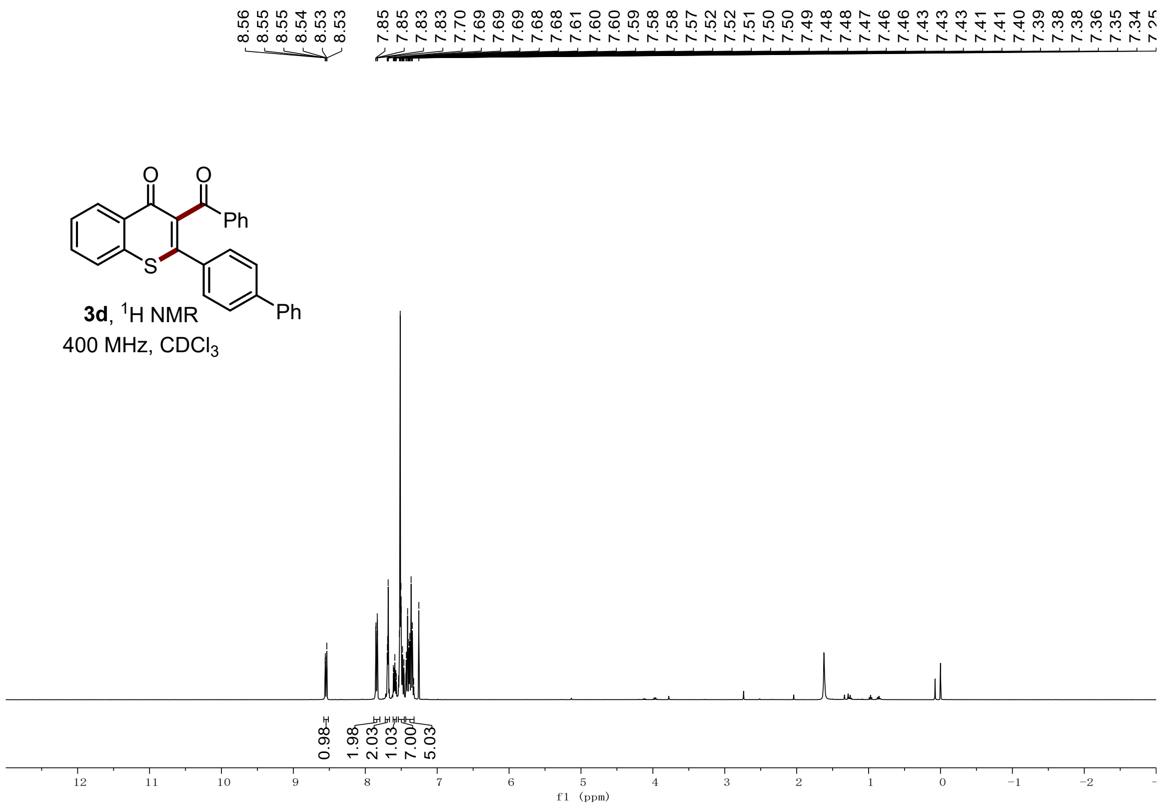



8.54 8.52 7.83 7.81 7.81 7.70 7.69 7.68 7.68 7.67 7.66 7.65 7.59 7.59 7.58 7.57 7.57 7.56 7.55 7.50 7.50 7.49 7.48 7.48 7.47 7.46 7.46 7.40 7.40 7.39 7.38 7.37 7.36 7.34 7.26 6.82 6.80 3.76
O
O
Ph
S
OMe
3b, ^{1}H NMR
400 MHz, $CDCl_3$
f1 (ppm)


195.15
178.95
161.16
152.04 137.60 137.00 134.77 133.40 131.99 130.61 130.15 129.17 129.07 128.59 128.01 127.05 126.00 114.26
55.31
O
O
Ph
S
OMe
3b, ^{13}C NMR
101 MHz, $CDCl_3$
f1 (ppm)


8.54 8.54 8.54 8.52 8.52 8.52 7.83 7.82 7.82 7.81 7.81 7.80 7.68 7.67 7.67 7.67 7.66 7.65 7.65 7.60 7.59 7.58 7.58 7.57 7.56 7.56 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.46 7.46 7.46 7.38 7.38 7.37 7.37 7.37 7.36 7.36 7.35 7.34 7.34 7.34 7.34 7.32 7.31 7.29 7.29 7.29 7.28 1.25
O
O
Ph
S
tBu
3c, 1H NMR
400 MHz, CDCl3
1.00
2.05
2.06
1.09
1.08
4.07
2.06
9.08
f1 (ppm)


195.00
178.96
153.68 152.53 137.66 137.30 134.96 133.25 131.98 131.85 130.67 129.14 129.06 128.53 128.31 128.02 126.03 125.75
34.77
31.05
O
O
Ph
S
tBu
3c, 13C NMR
101 MHz, CDCl3
f1 (ppm)


8.56
8.55
8.55
8.54
8.53
8.53
7.85
7.85
7.83
7.83
7.70
7.69
7.69
7.69
7.68
7.68
7.61
7.60
7.60
7.59
7.58
7.58
7.57
7.52
7.51
7.50
7.50
7.49
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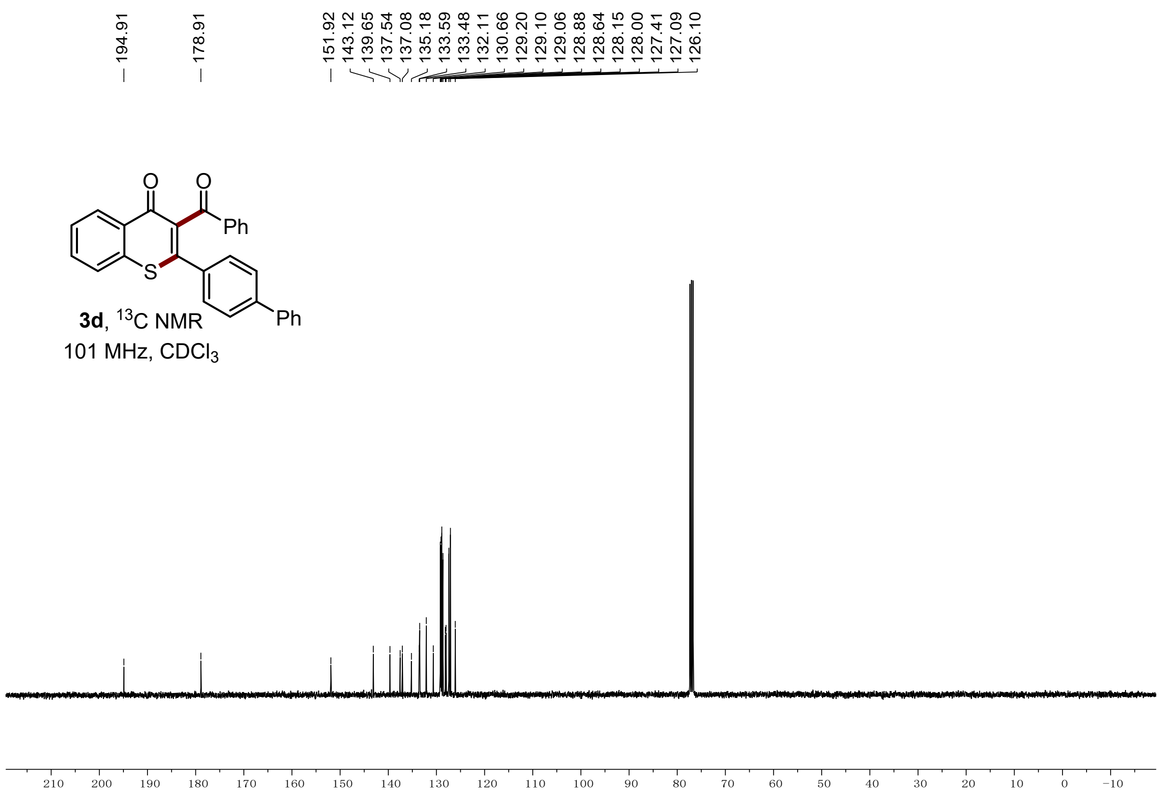
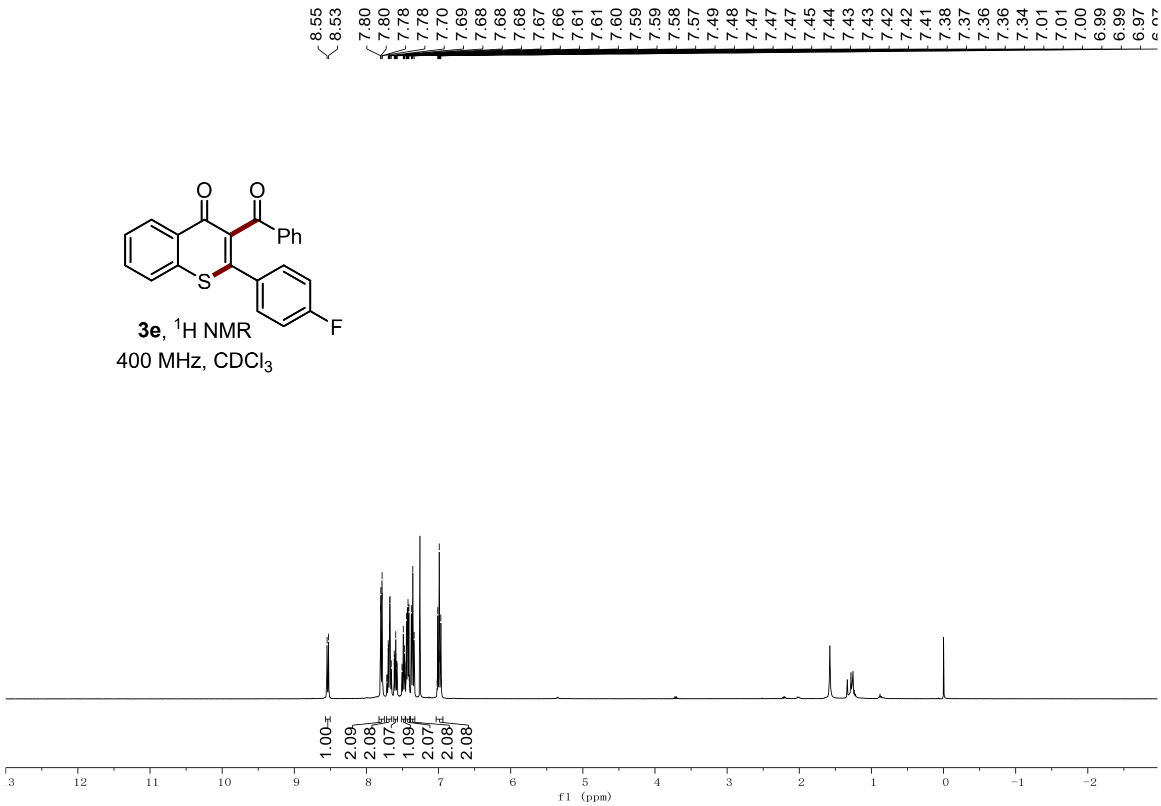
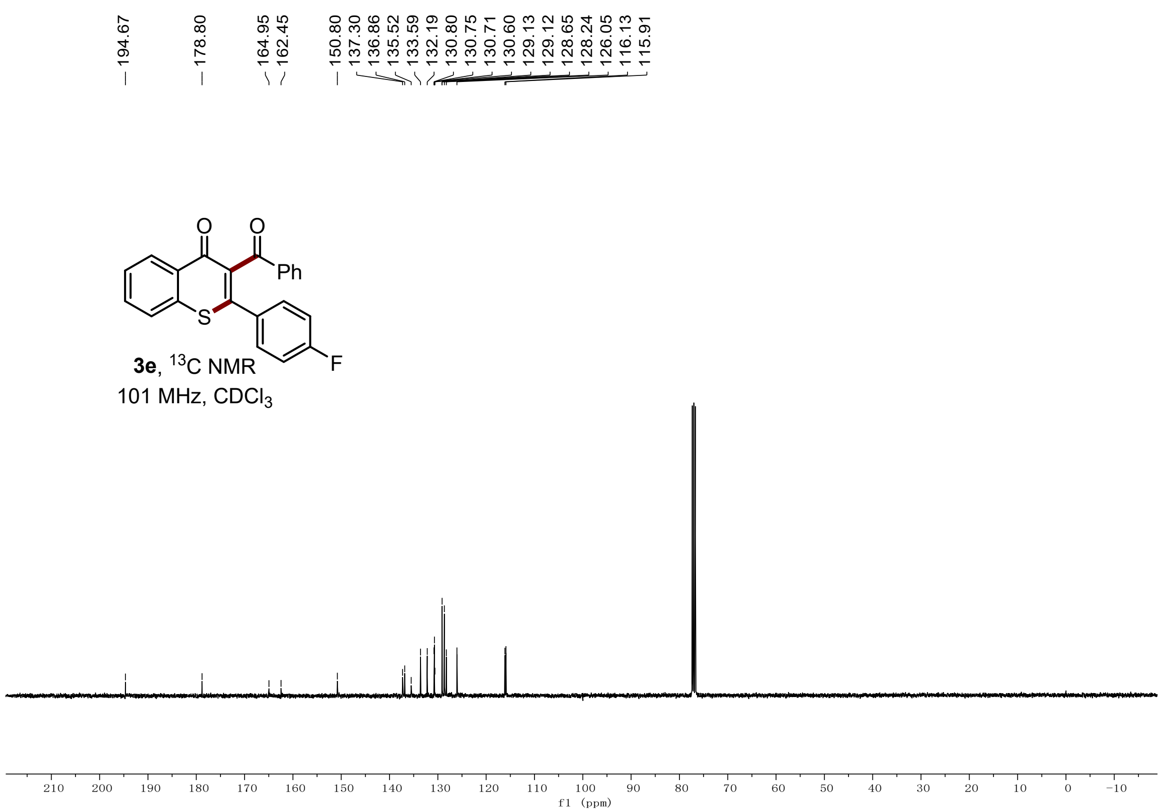
7.48
7.47
7.46
7.46
7.43
7.43
7.43
7.41
7.41
7.40
7.39
7.38
7.36
7.35
7.34
O
O
Ph
S
Ph
3d, 1H NMR
400 MHz, CDCl3
0.98
1.98
2.03
1.03
7.00
5.03
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


194.91
178.91
151.92
143.12
139.65
137.54
137.08
135.18
133.59
133.48
132.11
130.66
129.20
129.10
129.06
128.88
128.64
128.15
128.00
127.41
127.09
126.10
O
O
Ph
S
Ph
3d, 13C NMR
101 MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

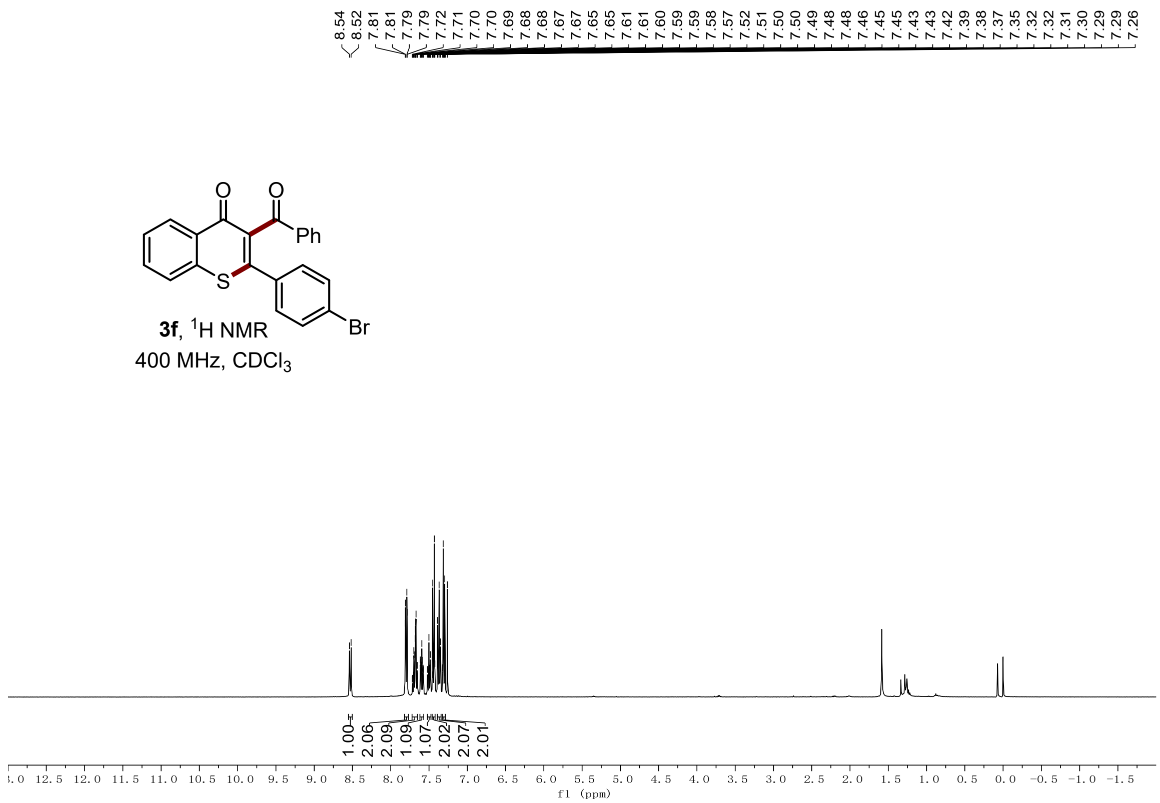
3e, ^{1}H NMR
400 MHz, $CDCl_3$

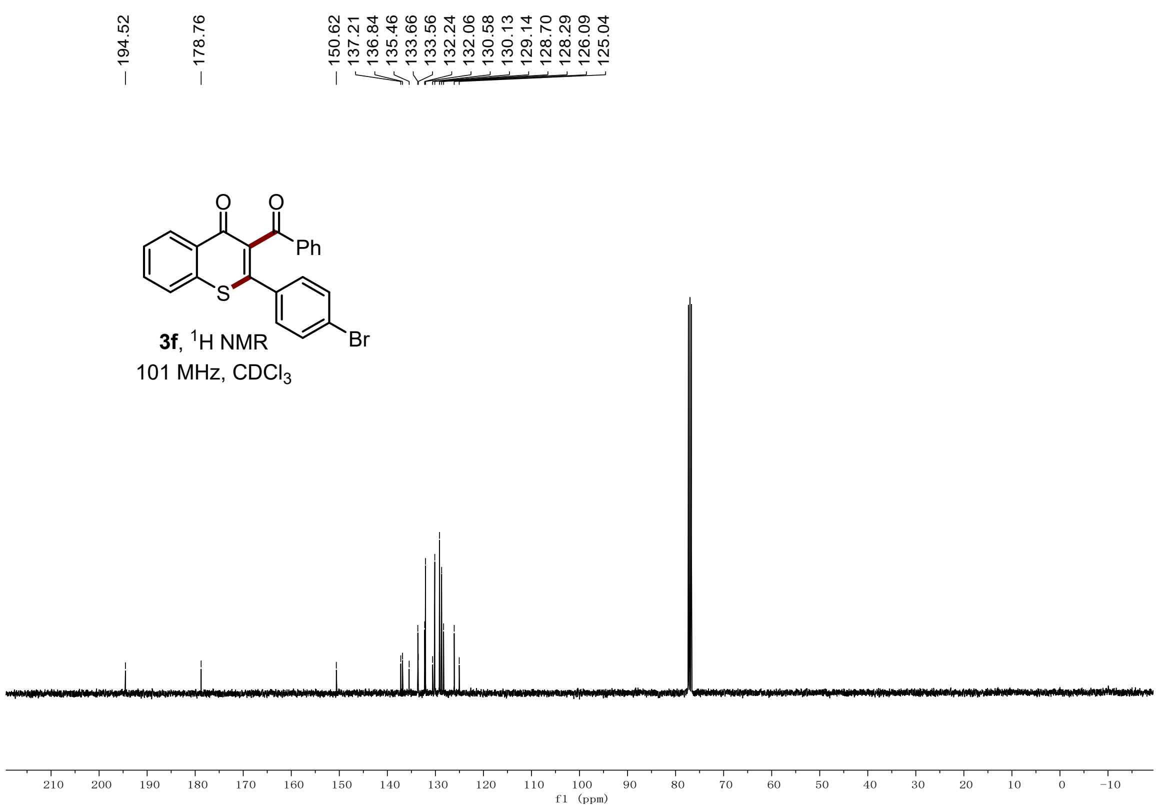


3e, ^{13}C NMR
101 MHz, $CDCl_3$




-109.38
O
O
Ph
S
F
3e, 19F NMR
376 MHz, CDCl3
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)


8.54
8.52
7.81
7.81
7.79
7.79
7.72
7.71
7.70
7.69
7.68
7.68
7.67
7.67
7.65
7.65
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.52
7.51
7.50
7.50
7.49
7.48
7.48
7.46
7.45
7.45
7.43
7.43
7.42
7.39
7.38
7.37
7.35
7.32
7.32
7.31
7.30
7.29
7.29
7.26
O
O
Ph
S
Br
3f, 1H NMR
400 MHz, CDCl3
1.00
2.06
2.09
1.09
1.07
2.02
2.07
2.01
f1 (ppm)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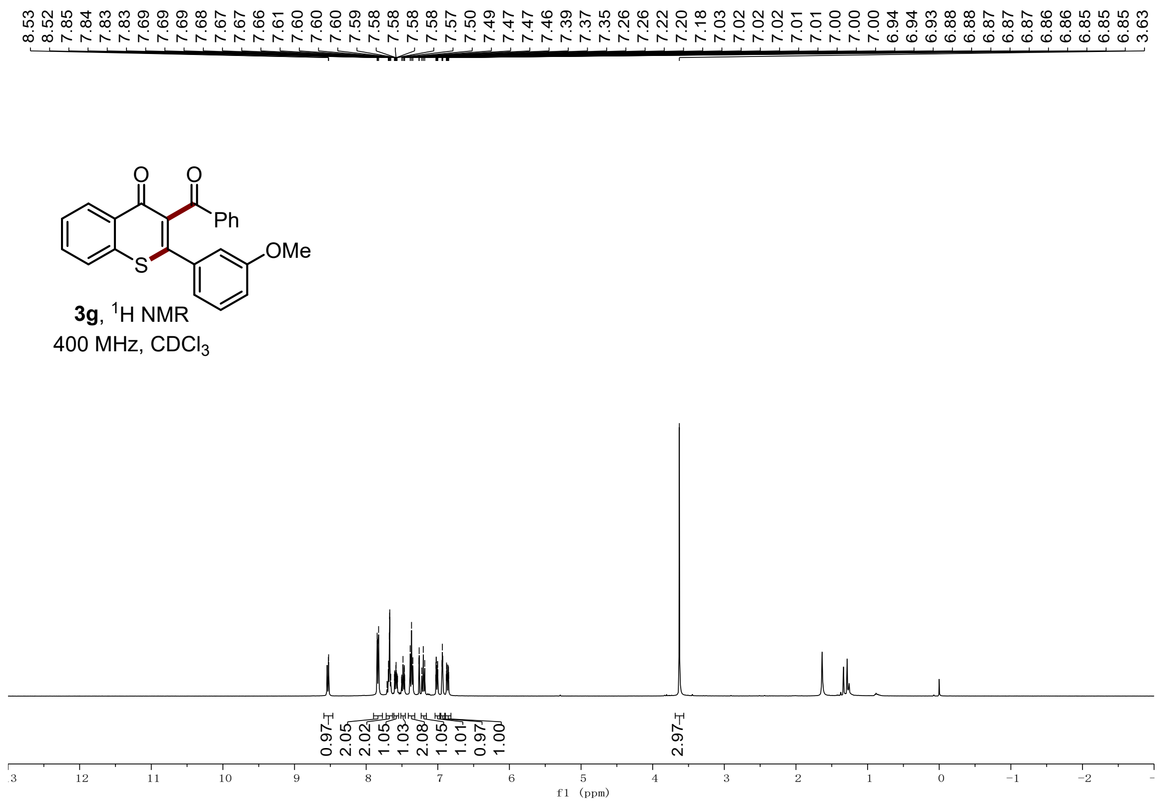
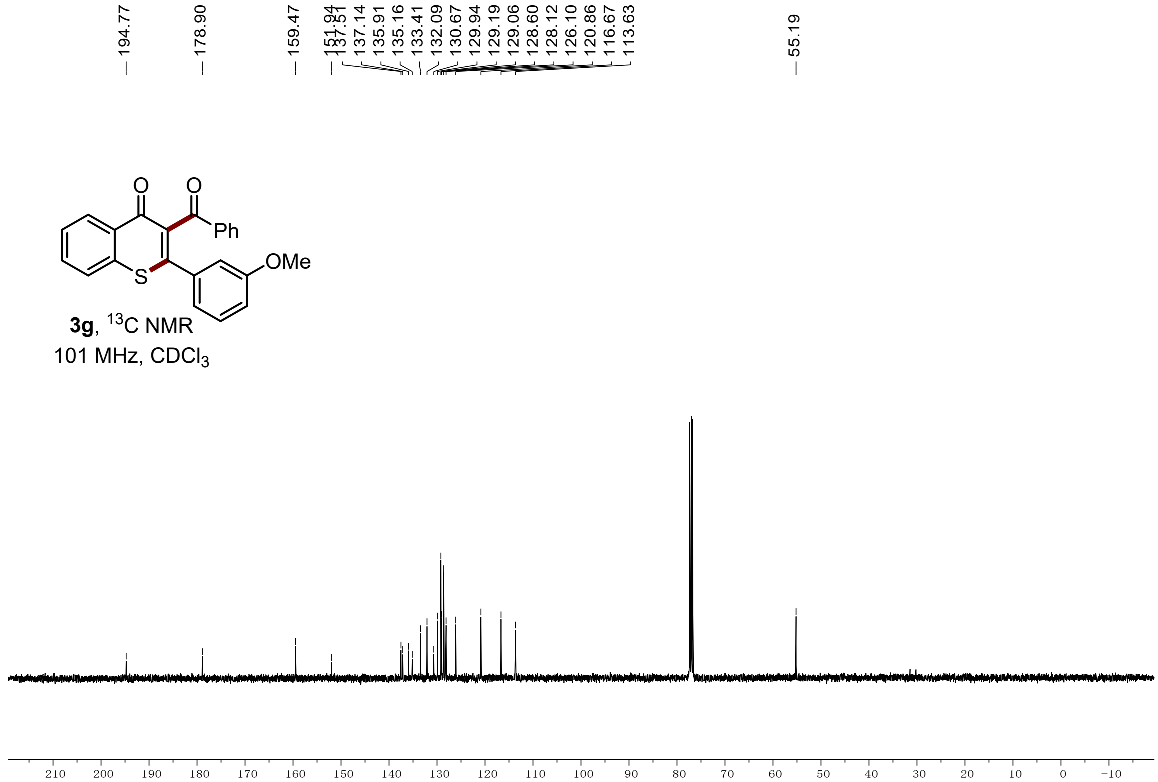
178.76
150.62
137.21
136.84
135.46
133.66
133.56
132.24
132.06
130.58
130.13
129.14
128.70
128.29
126.09
125.04
O
O
Ph
S
Br
3f, 1H NMR
101 MHz, CDCl3
f1 (ppm)



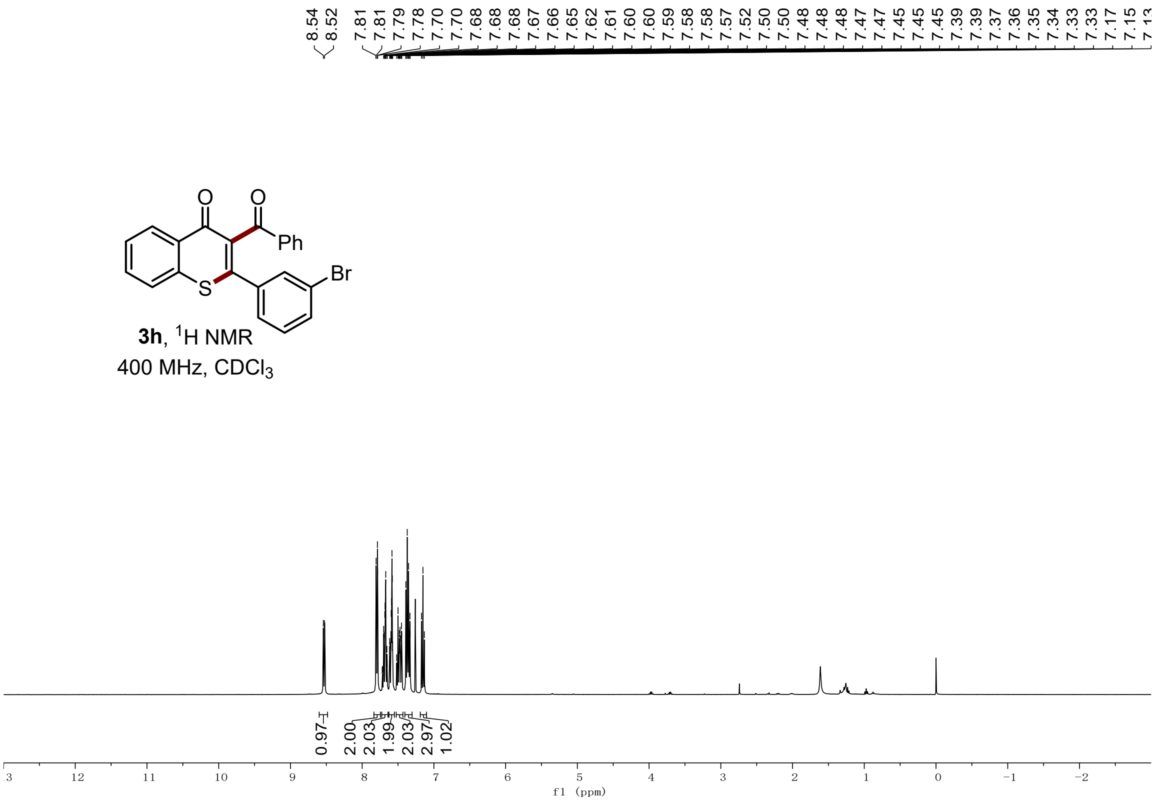
3g, ^{1}H NMR

400 MHz, $CDCl_3$

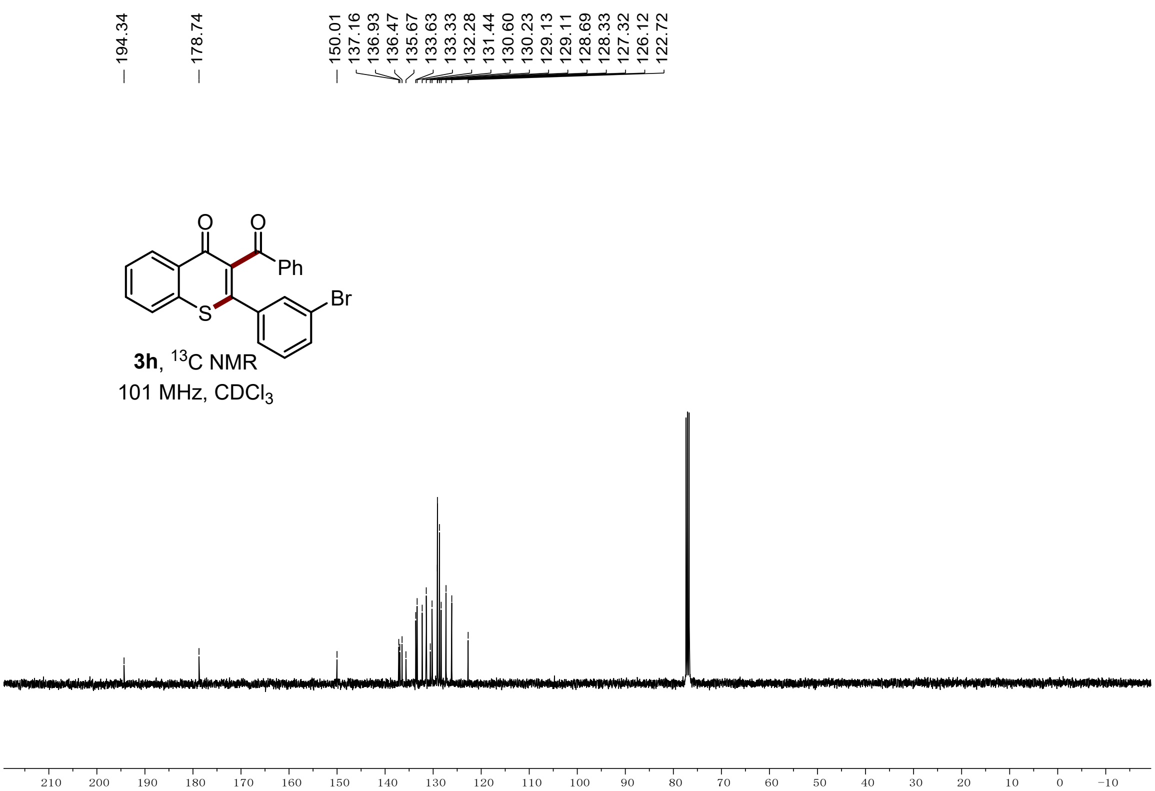


3g, ^{13}C NMR

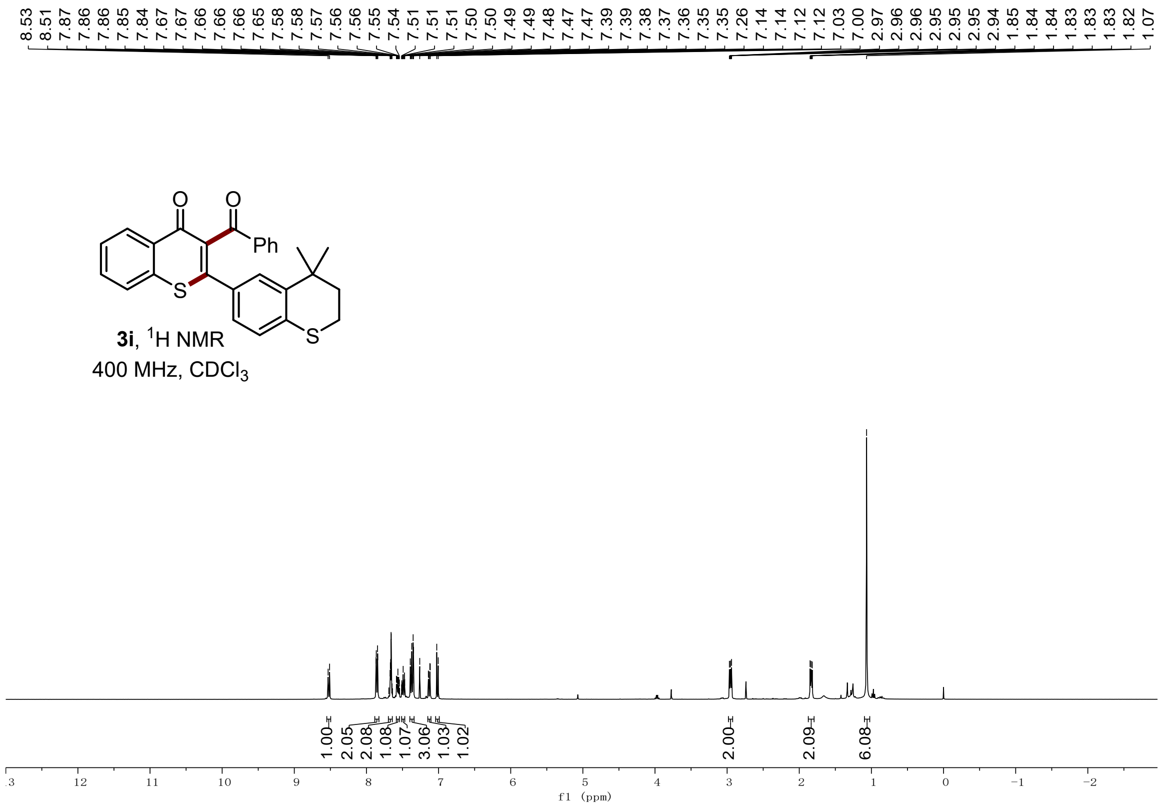
101 MHz, $CDCl_3$

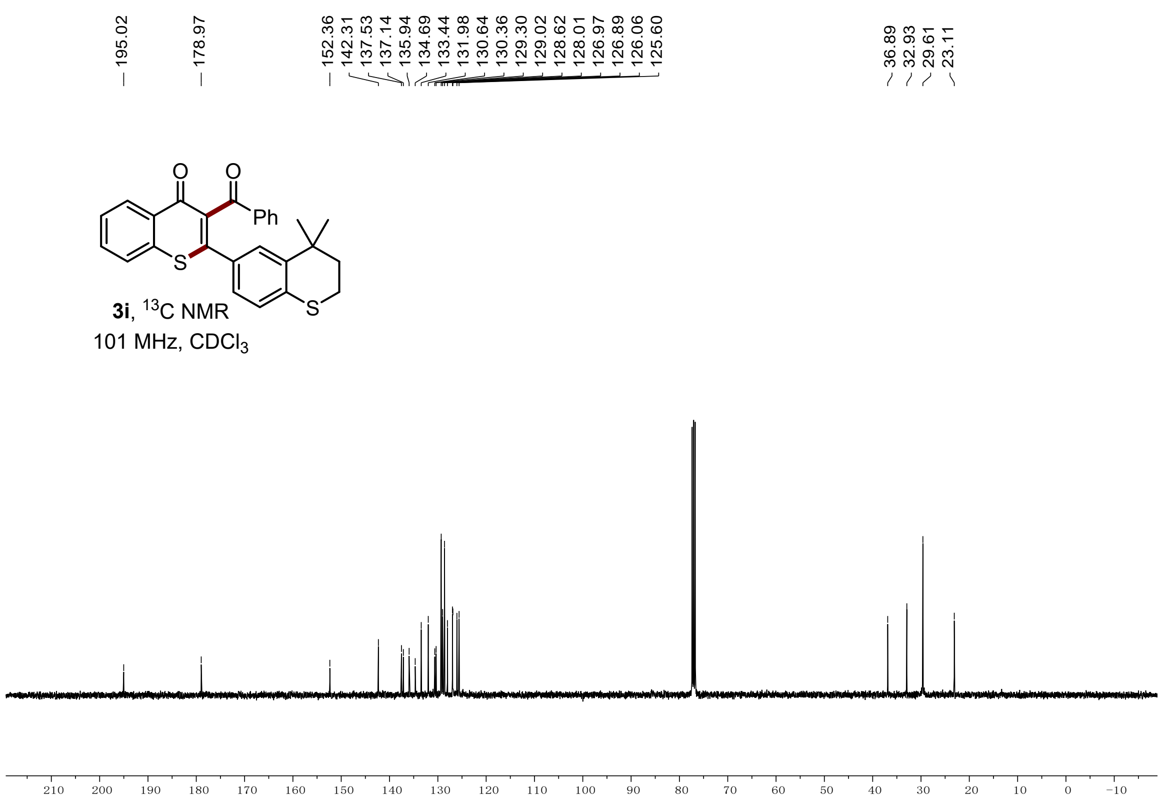


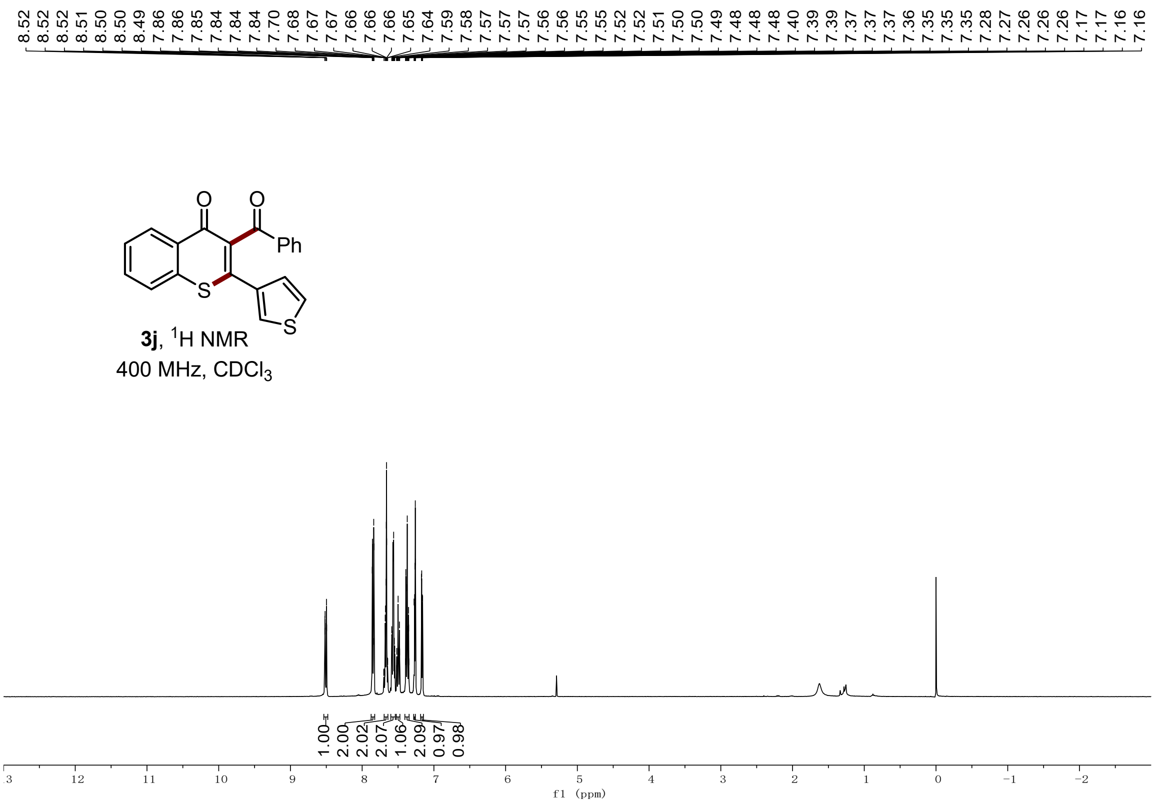
3h, ^{1}H NMR
400 MHz, $CDCl_3$

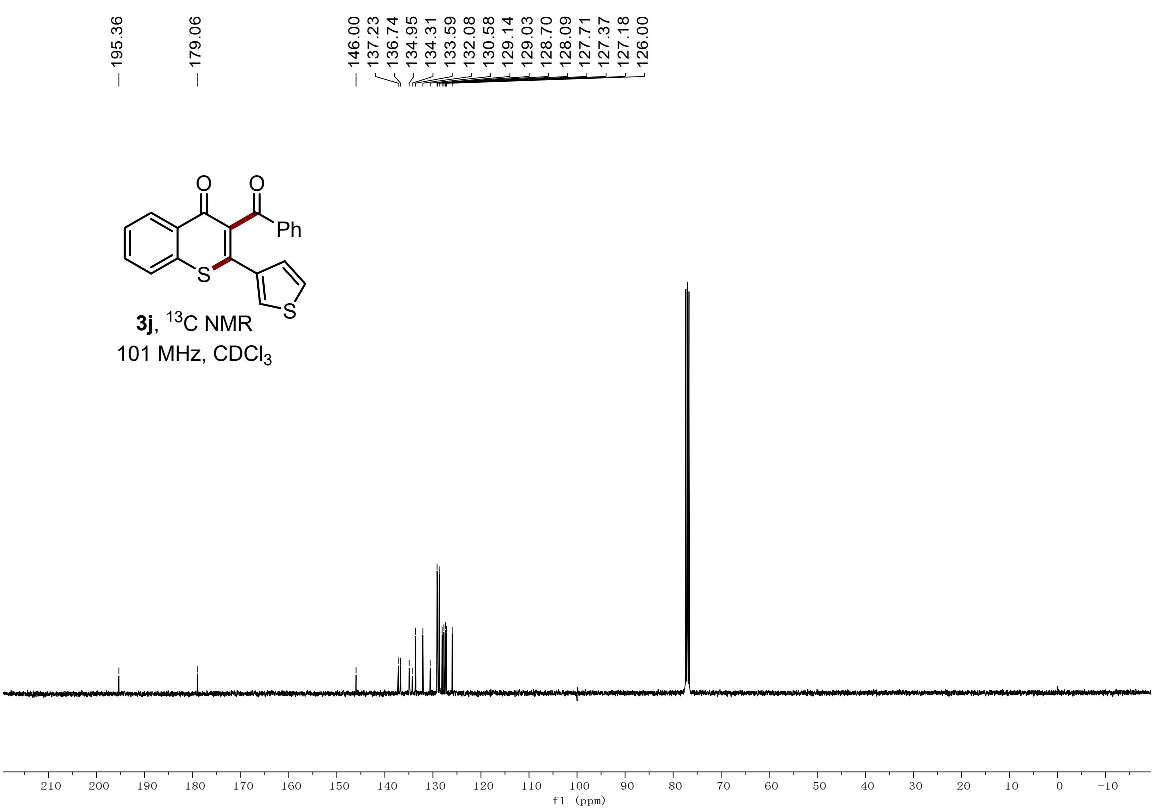


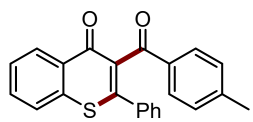
3h, ^{13}C NMR
101 MHz, $CDCl_3$


8.53 8.51 7.87 7.86 7.85 7.84 7.67 7.67 7.66 7.66 7.65 7.58 7.58 7.57 7.56 7.56 7.55 7.54 7.51 7.51 7.50 7.49 7.49 7.48 7.47 7.47 7.39 7.38 7.37 7.36 7.35 7.35 7.26 7.14 7.12 7.12 7.03 7.00 2.97 2.96 2.96 2.95 2.95 2.94 1.85 1.84 1.84 1.83 1.83 1.83 1.82 1.07
O
O
Ph
S
S
3i, 1H NMR
400 MHz, CDCl3
1.00 2.05 2.08 1.08 1.07 3.06 1.03 1.02
2.00
2.09
6.08
f1 (ppm)

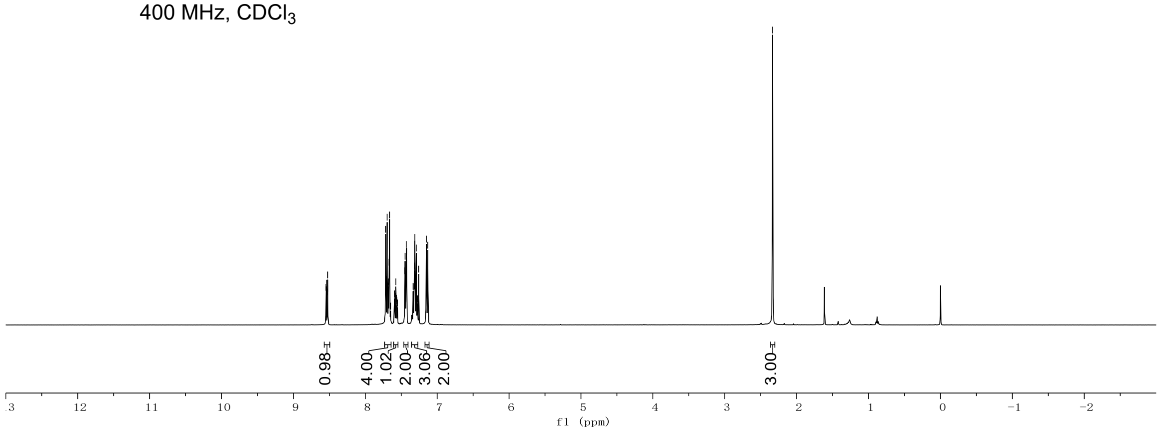

195.02
178.97
152.36 142.31 137.53 137.14 135.94 134.69 133.44 131.98 130.64 130.36 129.30 129.02 128.62 128.01 126.97 126.89 126.06 125.60
36.89 32.93 29.61 23.11
O
O
Ph
S
S
3i, 13C NMR
101 MHz, CDCl3
f1 (ppm)

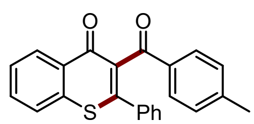

8.52 8.52 8.52 8.51 8.50 8.50 8.49 7.86 7.86 7.85 7.84 7.84 7.84 7.70 7.68 7.67 7.67 7.66 7.66 7.65 7.64 7.59 7.58 7.57 7.57 7.56 7.56 7.55 7.55 7.52 7.52 7.51 7.50 7.50 7.49 7.48 7.48 7.48 7.40 7.39 7.39 7.37 7.37 7.37 7.36 7.35 7.35 7.35 7.28 7.27 7.27 7.26 7.26 7.17 7.17 7.16 7.16
O
O
Ph
S
S
3j, ^{1}H NMR
400 MHz, $CDCl_3$
1.00 2.00 2.02 2.07 1.06 2.09 0.97 0.98
f1 (ppm)


195.36
179.06
146.00 137.23 136.74 134.95 134.31 133.59 132.08 130.58 129.14 129.03 128.70 128.09 127.71 127.37 127.18 126.00
O
O
Ph
S
S
3j, ^{13}C NMR
101 MHz, $CDCl_3$
f1 (ppm)

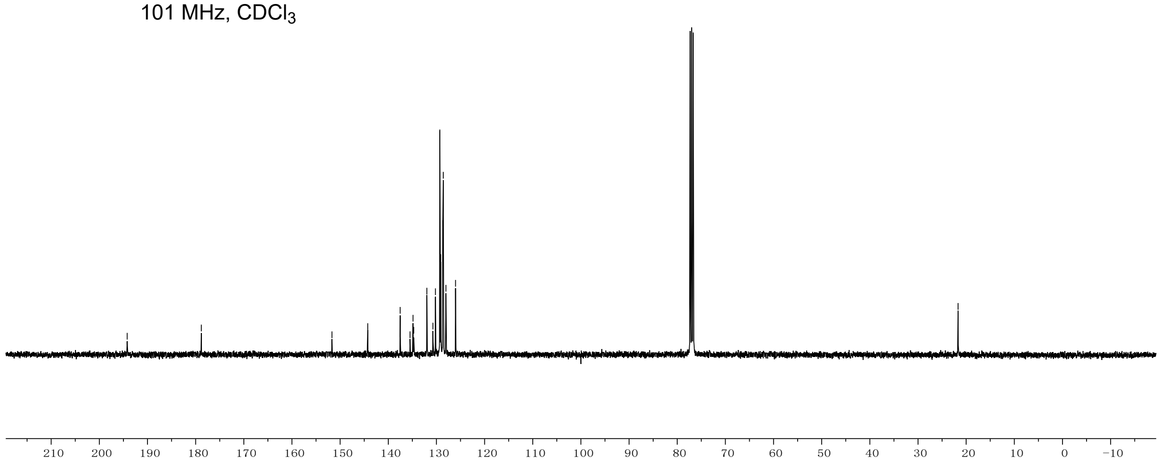


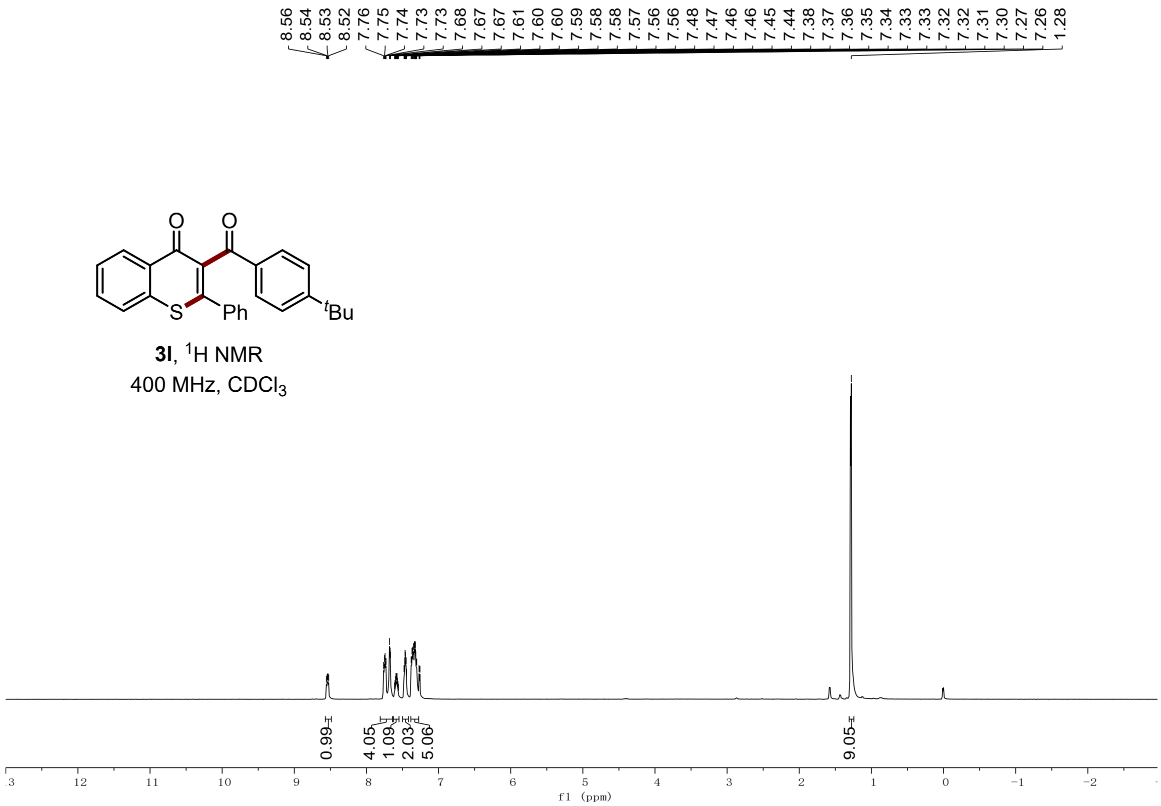
3k, ¹H NMR
400 MHz, $CDCl_3$



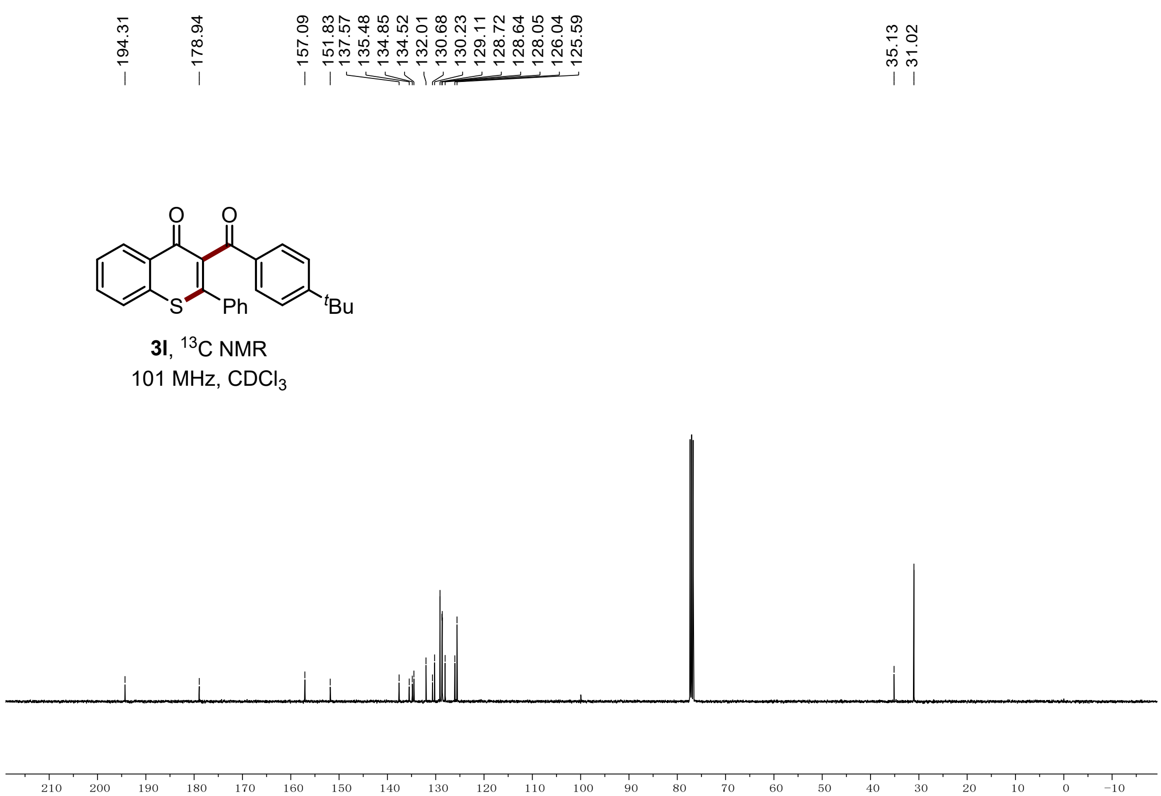


3k, ¹³C NMR
101 MHz, $CDCl_3$

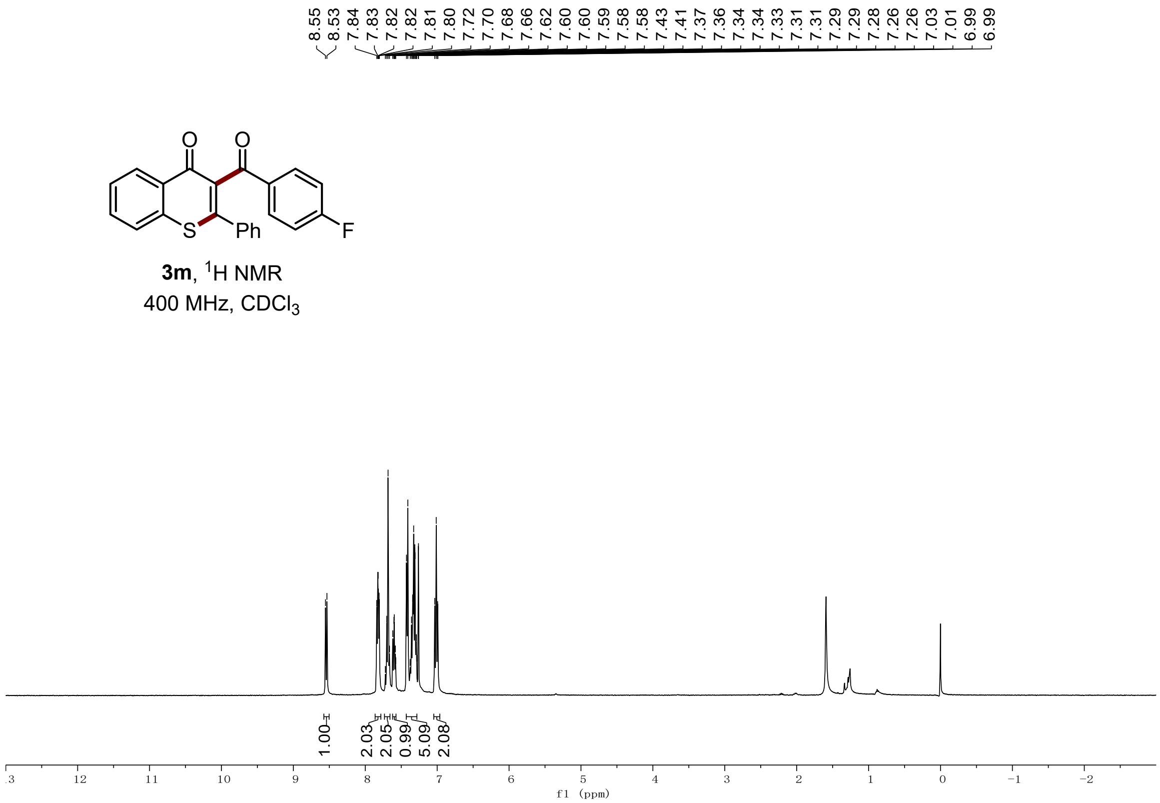




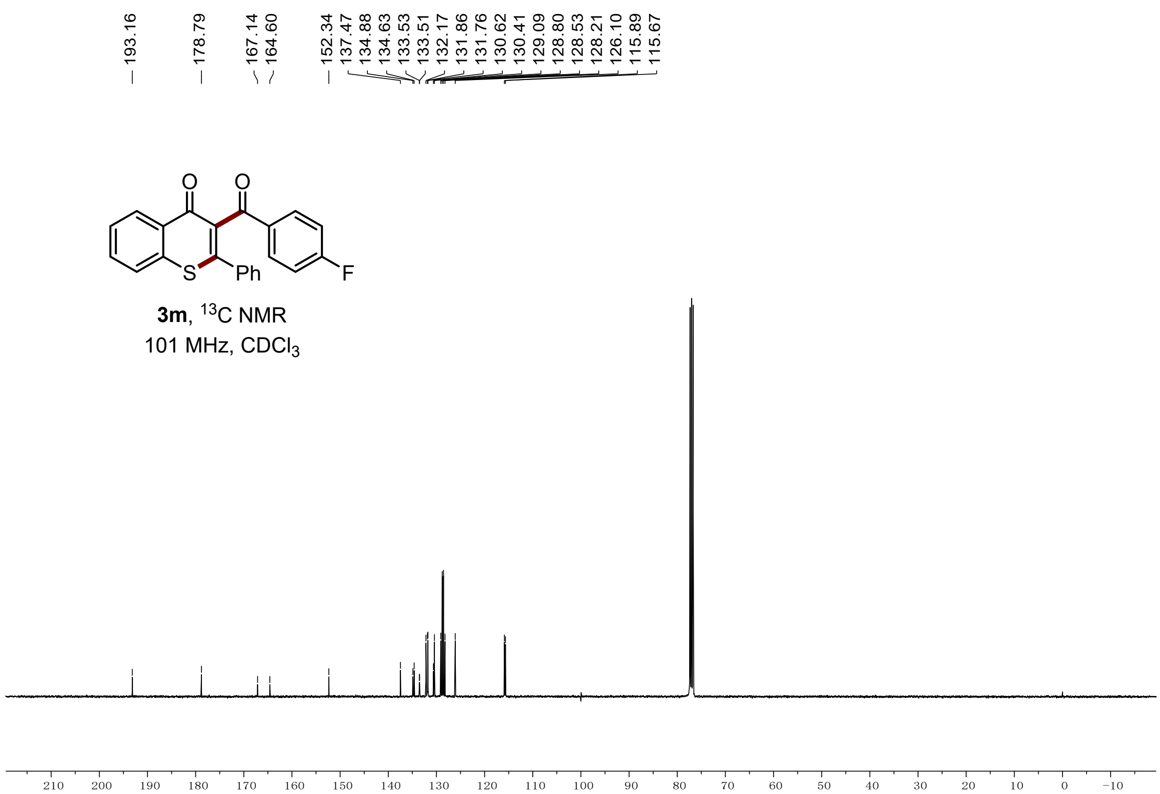
3l, 1H NMR
400 MHz, CDCl3
8.56 8.54 8.53 8.52 7.76 7.75 7.74 7.73 7.73 7.68 7.67 7.67 7.61 7.60 7.60 7.59 7.58 7.58 7.57 7.56 7.56 7.48 7.47 7.46 7.46 7.45 7.44 7.38 7.37 7.36 7.35 7.34 7.33 7.33 7.32 7.32 7.31 7.30 7.27 7.26 1.28
0.99 4.05 1.09 2.03 5.06 9.05
f1 (ppm)



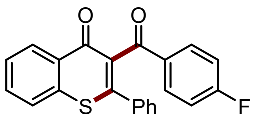
3l, 13C NMR
101 MHz, CDCl3
194.31 178.94 157.09 151.83 137.57 135.48 134.85 134.52 132.01 130.68 130.23 129.11 128.72 128.64 128.05 126.04 125.59 35.13 31.02
f1 (ppm)



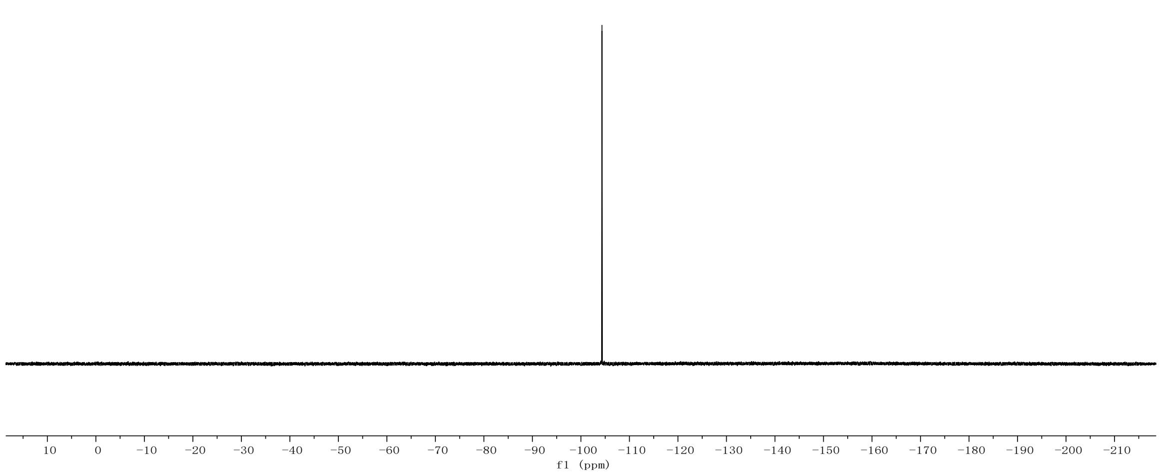
3m, ^{1}H NMR
400 MHz, $CDCl_3$

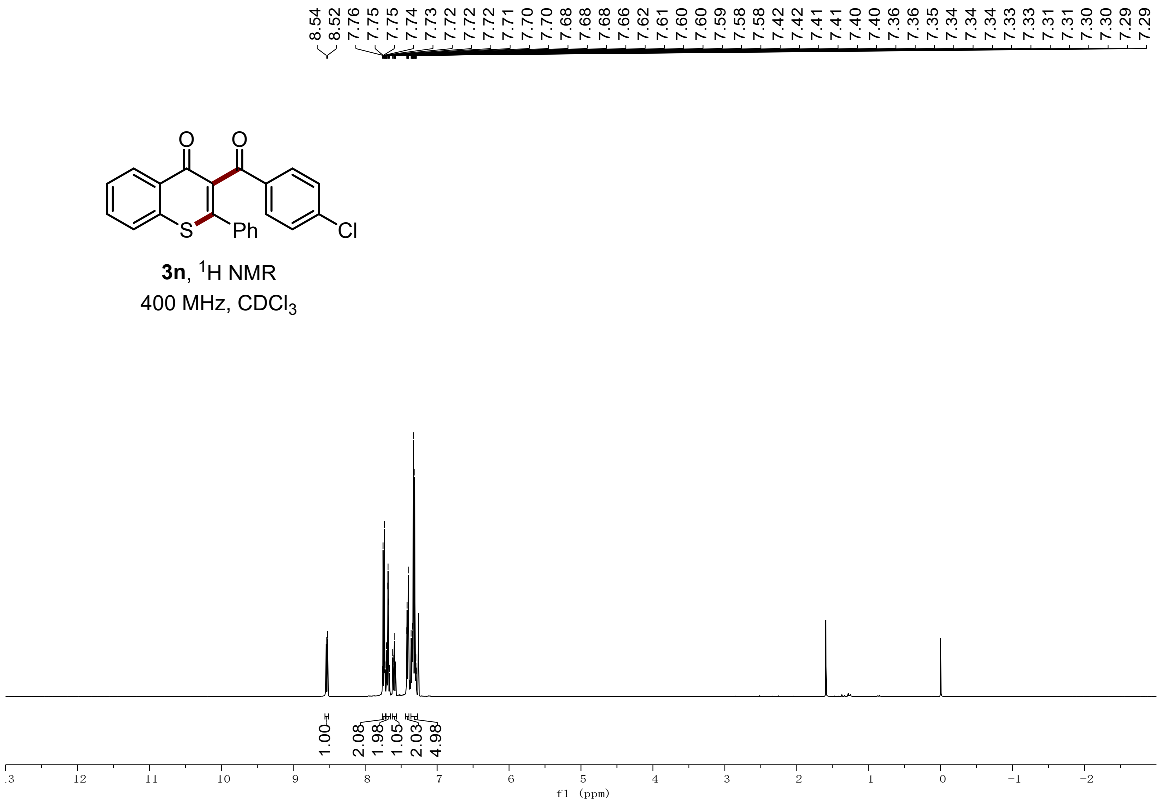


3m, ^{13}C NMR
101 MHz, $CDCl_3$



3m, ^{19}F NMR
376 MHz, $CDCl_3$




8.54
8.52
7.76
7.75
7.75
7.74
7.73
7.72
7.72
7.72
7.71
7.70
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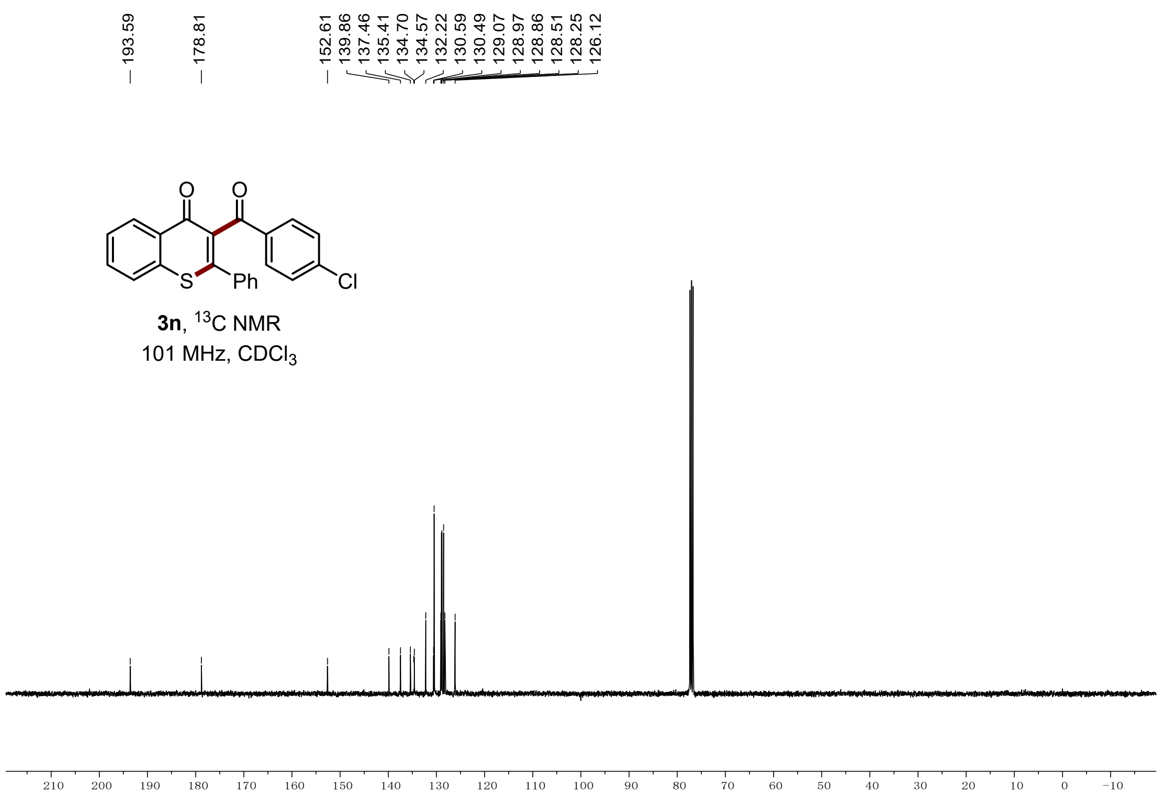
7.68
7.66
7.62
7.61
7.60
7.60
7.59
7.58
7.58
7.42
7.42
7.41
7.41
7.40
7.40
7.36
7.36
7.35
7.34
7.34
7.33
7.33
7.31
7.31
7.30
7.29
7.29
O
O
S
Ph
Cl
3n, 1H NMR
400 MHz, CDCl3
1.00
2.08
1.98
1.05
2.03
4.98
f1 (ppm)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178.81
152.61
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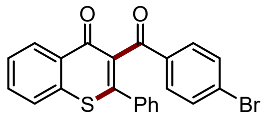
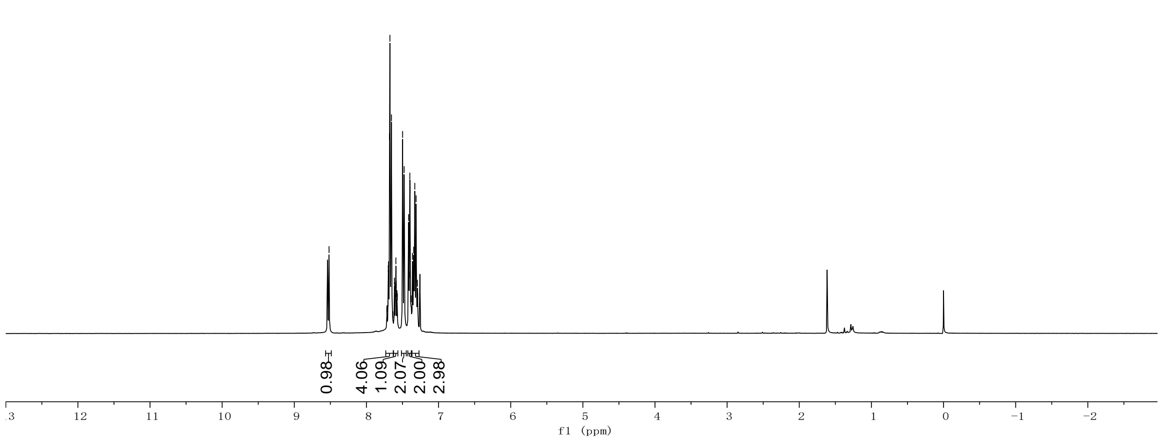
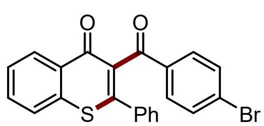
137.46
135.41
134.70
134.57
132.22
130.59
130.49
129.07
128.97
128.86
128.51
128.25
126.12
O
O
S
Ph
Cl
3n, 13C NMR
101 MHz, CDCl3
f1 (ppm)

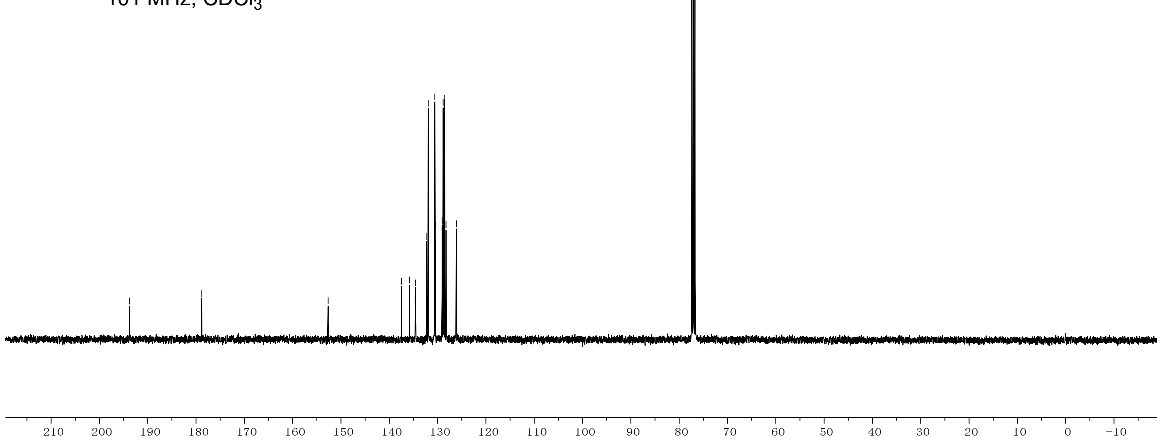


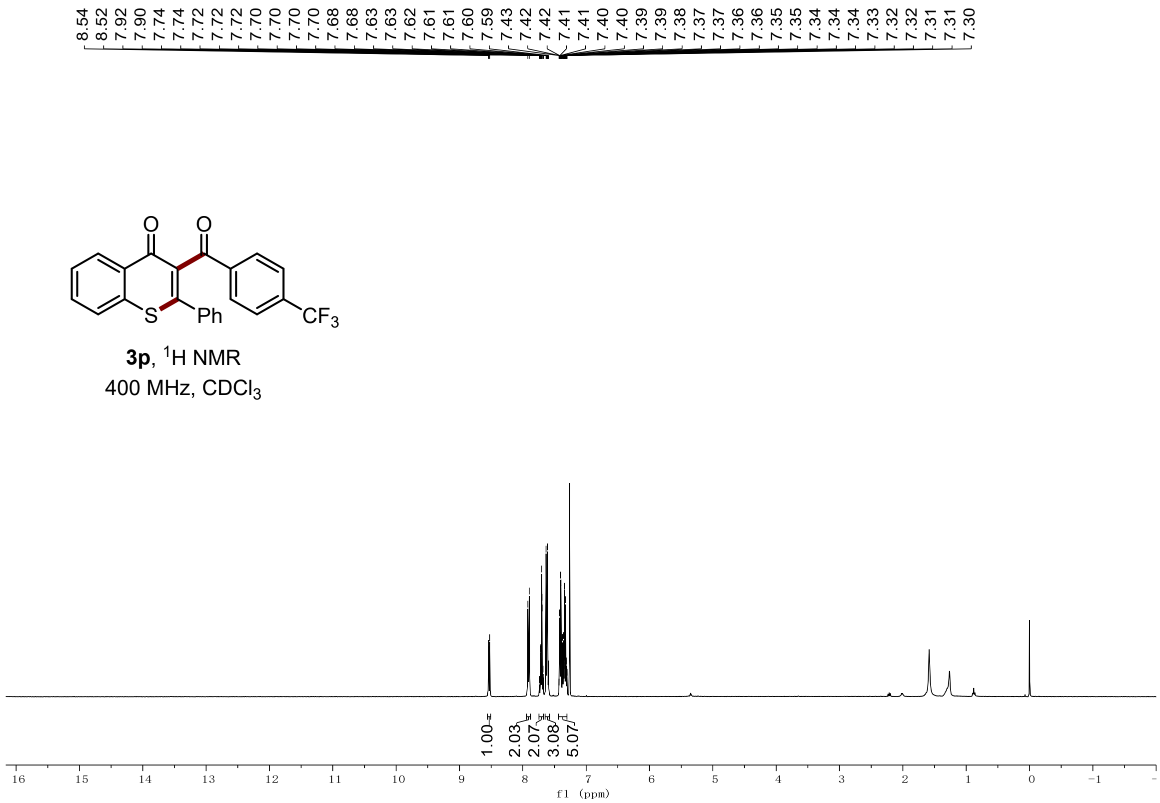
3o, ^{1}H NMR
400 MHz, $CDCl_3$

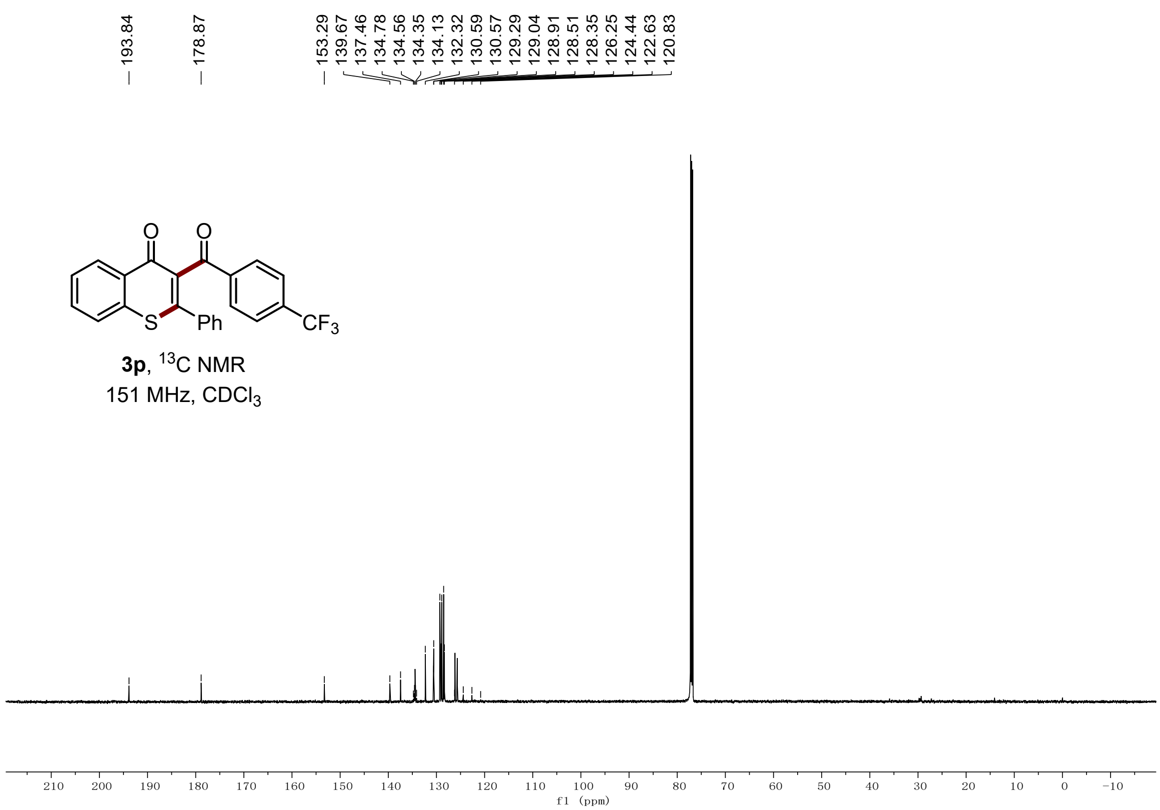




3o, ^{13}C NMR
101 MHz, $CDCl_3$




8.54 8.52 7.92 7.90 7.74 7.74 7.72 7.72 7.72 7.70 7.70 7.70 7.70 7.68 7.68 7.63 7.63 7.62 7.61 7.61 7.60 7.59 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.34 7.33 7.32 7.32 7.31 7.31 7.30
O
O
S
Ph
CF3
3p, 1H NMR
400 MHz, CDCl3
1.00
2.03
2.07
3.08
5.07
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)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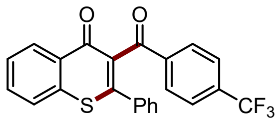
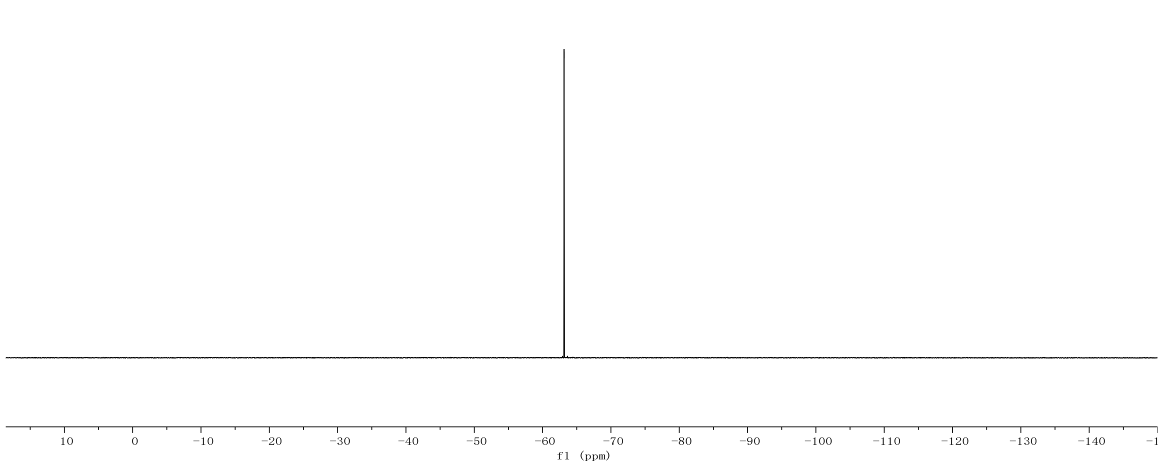
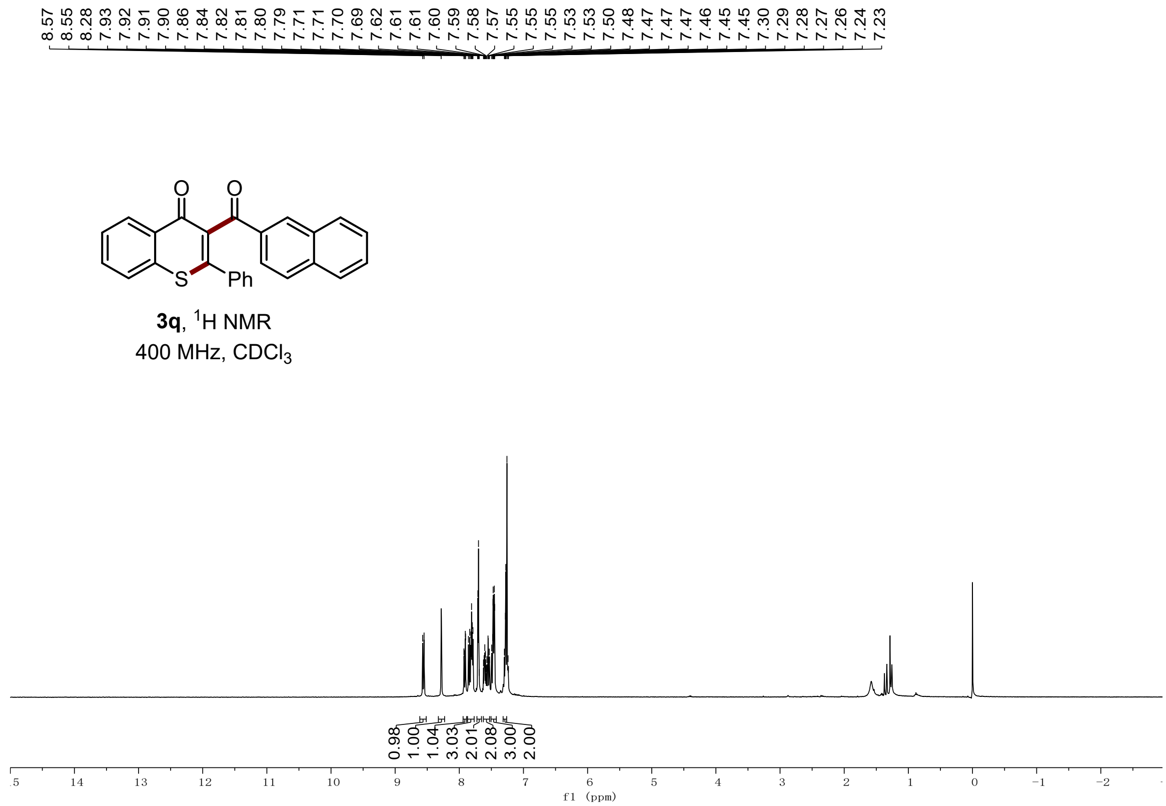
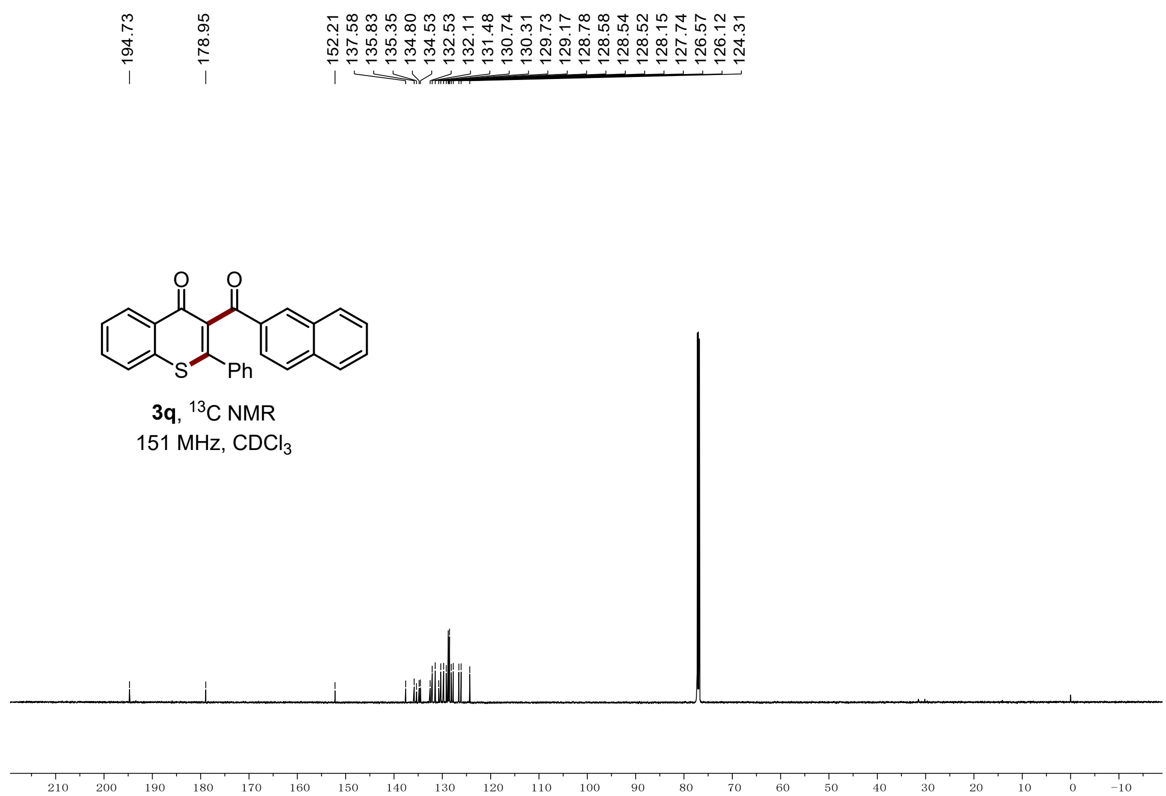
178.87
153.29 139.67 137.46 134.78 134.56 134.35 134.13 132.32 130.59 130.57 129.29 129.04 128.91 128.51 128.35 126.25 124.44 122.63 120.83
O
O
S
Ph
CF3
3p, 13C NMR
151 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

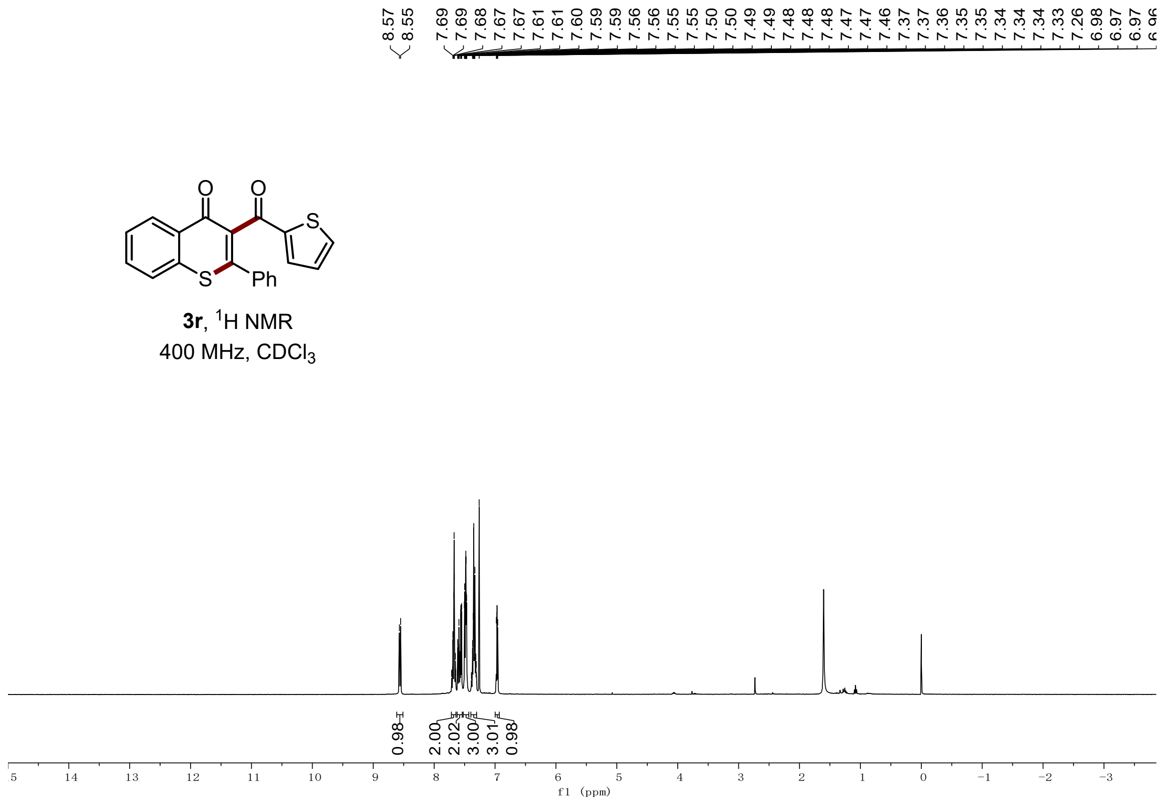


3p, ^{13}F NMR
376 MHz, $CDCl_3$

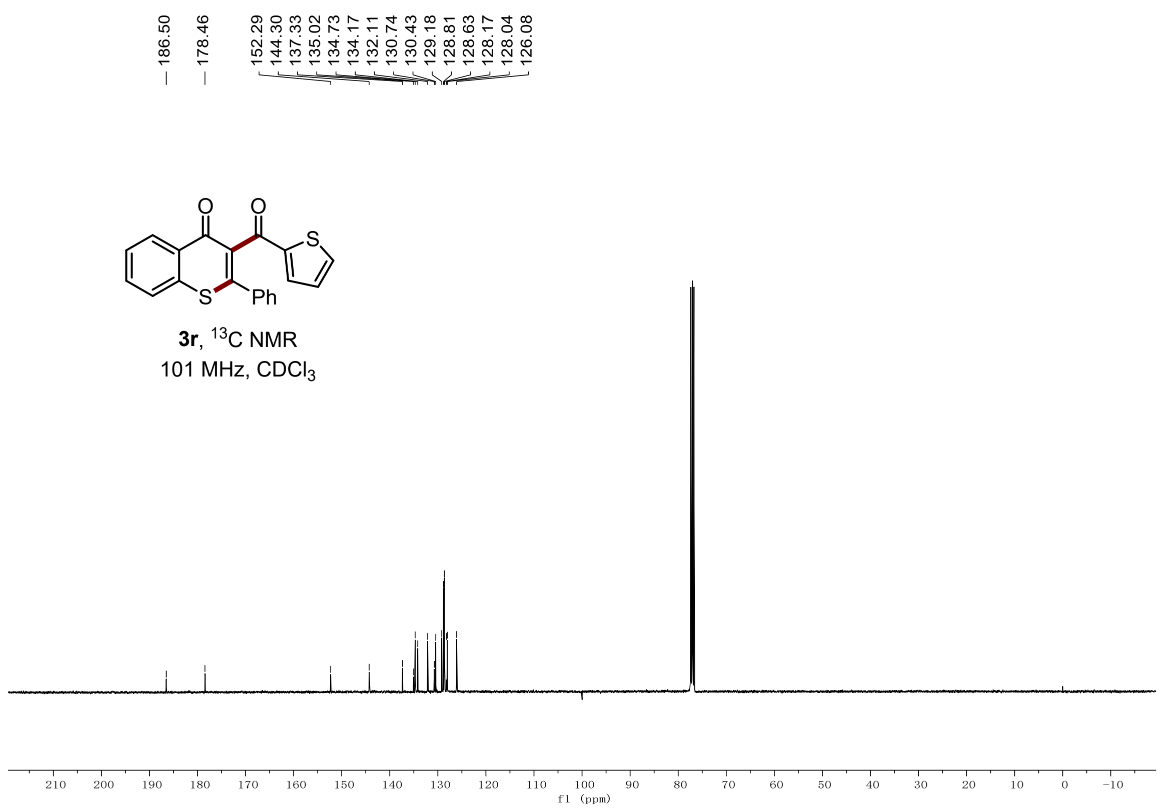



8.57 8.55 8.28 7.93 7.92 7.91 7.90 7.86 7.84 7.82 7.81 7.80 7.79 7.71 7.71 7.70 7.69 7.62 7.61 7.61 7.60 7.59 7.58 7.57 7.55 7.55 7.55 7.53 7.53 7.50 7.48 7.47 7.47 7.46 7.45 7.45 7.30 7.29 7.28 7.27 7.26 7.24 7.23
O
O
S
Ph
3q, 1H NMR
400 MHz, CDCl3
0.98 1.00 1.04 3.03 2.01 2.08 3.00 2.00
f1 (ppm)

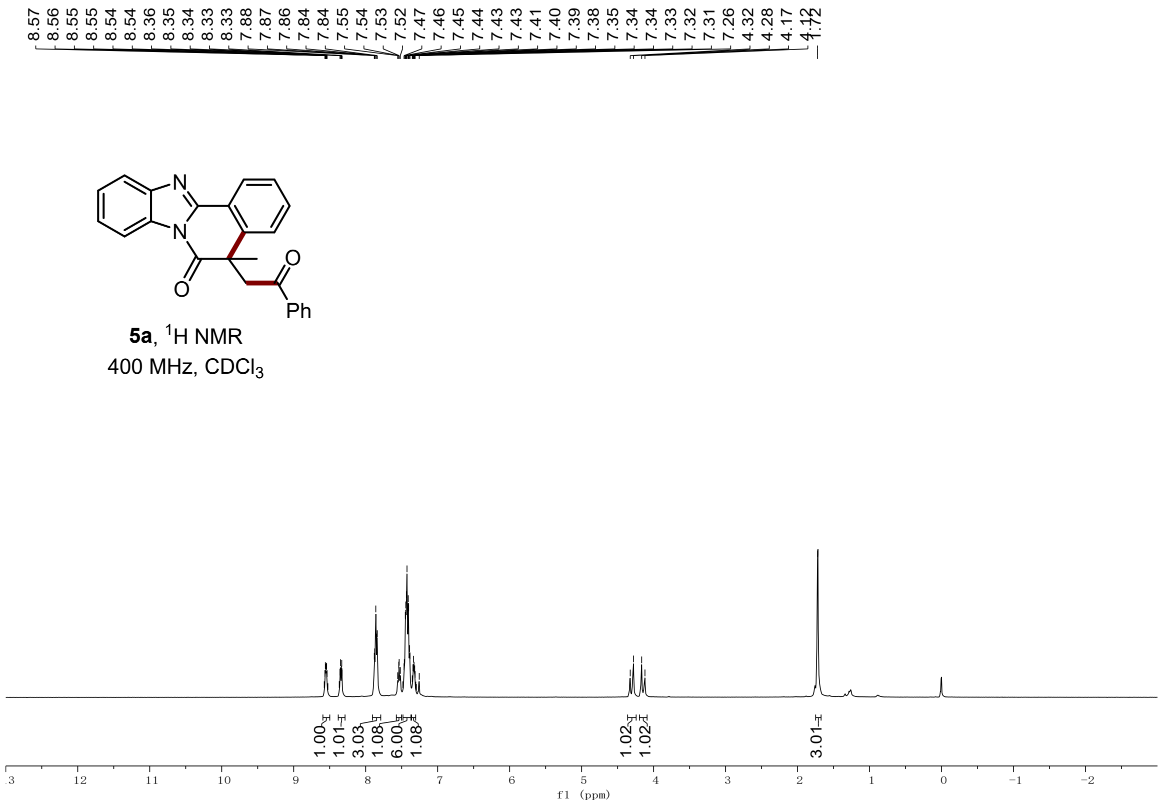

194.73
178.95
152.21 137.58 135.83 135.35 134.80 134.53 132.53 132.11 131.48 130.74 130.31 129.73 129.17 128.78 128.58 128.54 128.52 128.15 127.74 126.57 126.12 124.31
O
O
S
Ph
3q, 13C NMR
151 MHz, CDCl3
f1 (ppm)



3r, ^{1}H NMR
400 MHz, $CDCl_3$



3r, ^{13}C NMR
101 MHz, $CDCl_3$


8.57
8.56
8.55
8.55
8.54
8.54
8.36
8.35
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8.33
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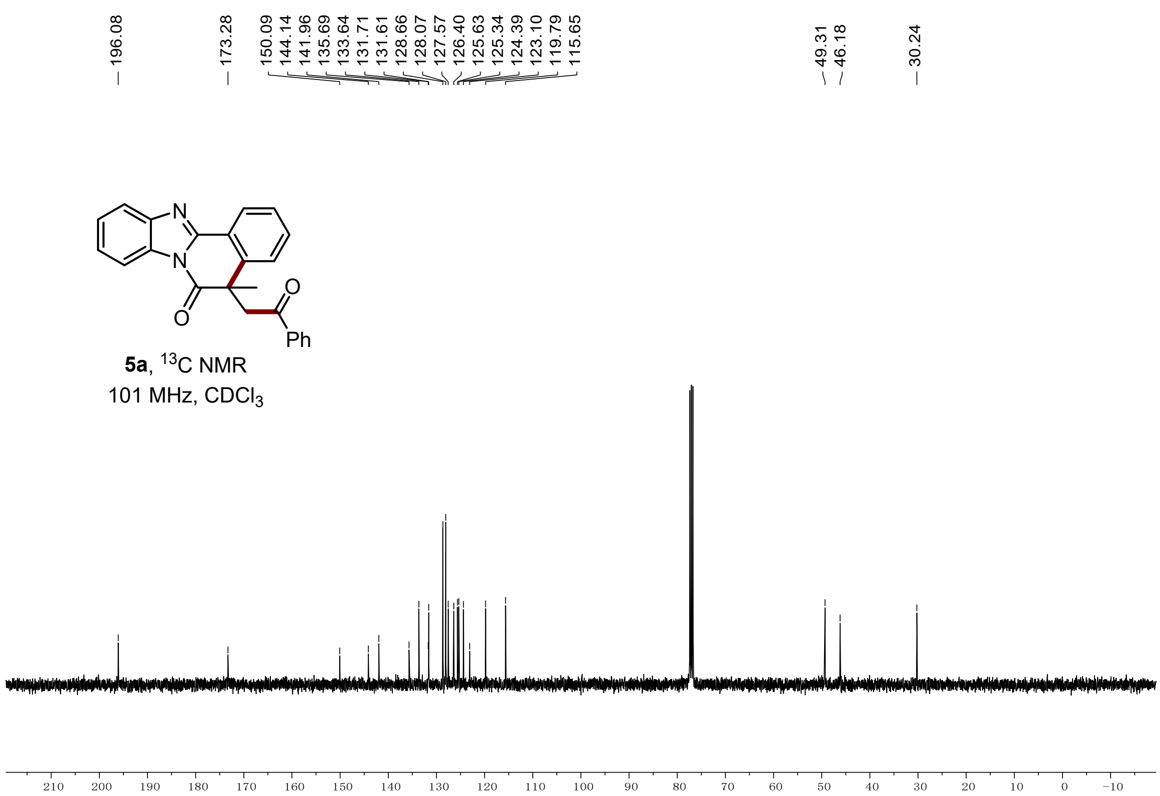
7.88
7.87
7.86
7.84
7.84
7.55
7.54
7.53
7.52
7.47
7.46
7.45
7.44
7.43
7.43
7.41
7.40
7.39
7.38
7.35
7.34
7.34
7.33
7.32
7.31
7.26
4.32
4.28
4.17
4.12
1.72
N
N
O
O
Ph
5a, 1H NMR
400 MHz, CDCl3
1.00
1.01
3.03
1.08
6.00
1.08
1.02
1.02
3.01
f1 (ppm)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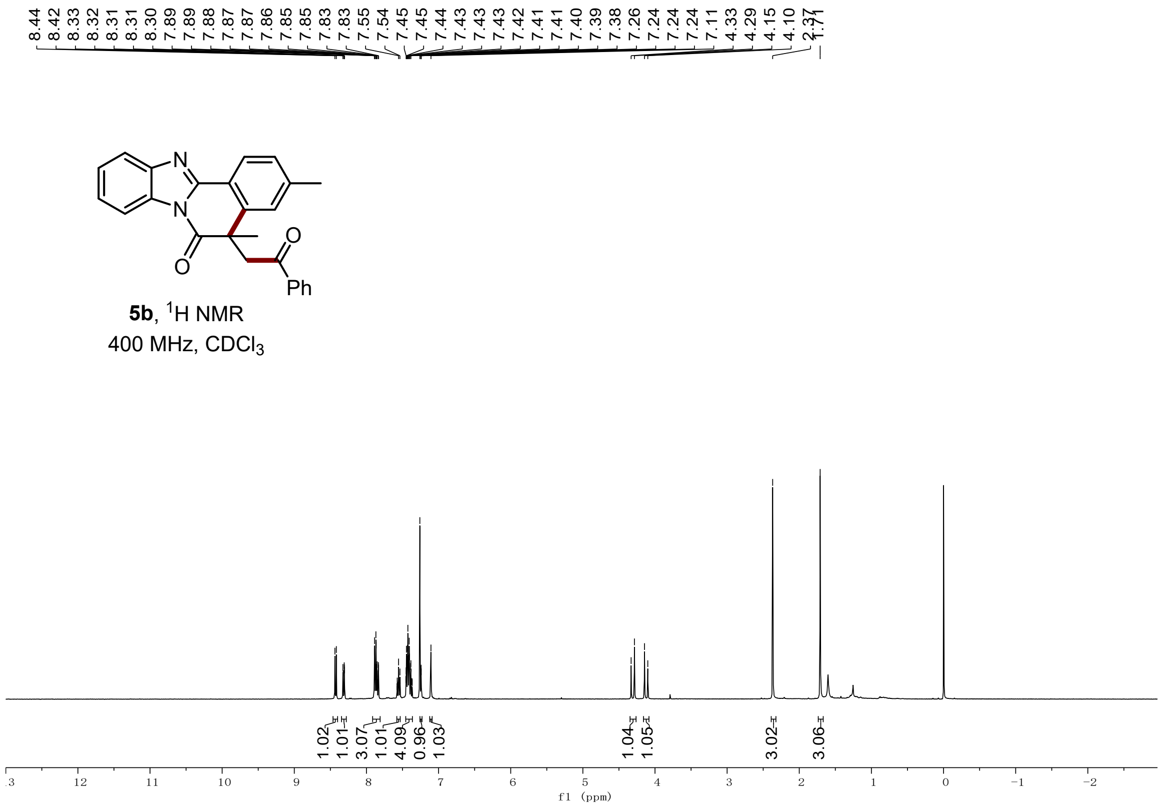
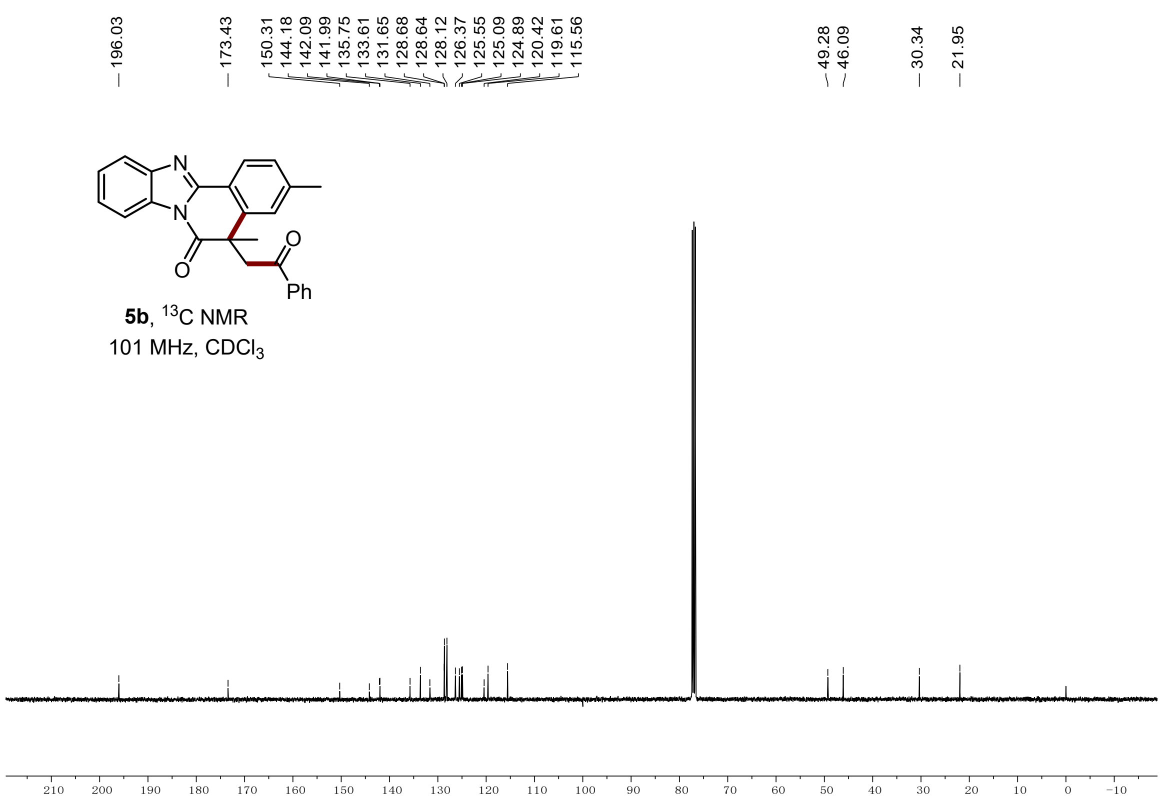
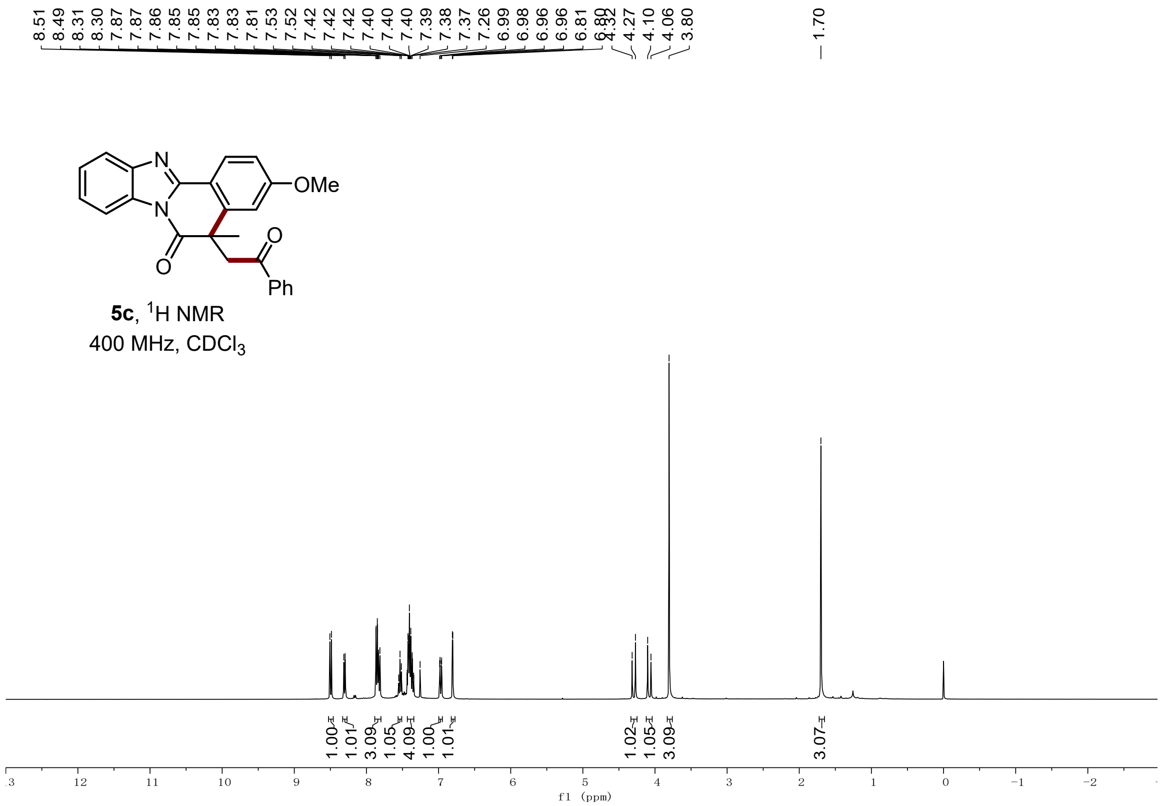
173.28
150.09
144.14
141.96
135.69
133.64
131.71
131.61
128.66
128.07
127.57
126.40
125.63
125.34
124.39
123.10
119.79
115.65
49.31
46.18
30.24
N
N
O
O
Ph
5a, 13C NMR
101 MHz, CDCl3
f1 (ppm)



8.44 8.42 8.33 8.32 8.31 8.31 8.30 7.89 7.88 7.87 7.86 7.85 7.85 7.83 7.83 7.55 7.54 7.45 7.45 7.44 7.43 7.43 7.43 7.42 7.41 7.41 7.40 7.39 7.38 7.26 7.24 7.24 7.24 7.11 4.33 4.29 4.15 4.10 2.37 1.71
N
O
O
Ph
5b, 1H NMR
400 MHz, CDCl3
1.02 1.01 3.07 1.01 4.09 0.96 1.03 1.04 1.05 3.02 3.06
f1 (ppm)



196.03 173.43 150.31 144.18 142.09 141.99 135.75 133.61 131.65 128.68 128.64 128.12 126.37 125.55 125.09 124.89 120.42 119.61 115.56 49.28 46.09 30.34 21.95
N
O
O
Ph
5b, 13C NMR
101 MHz, CDCl3
f1 (ppm)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7.85
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7.83
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7.81
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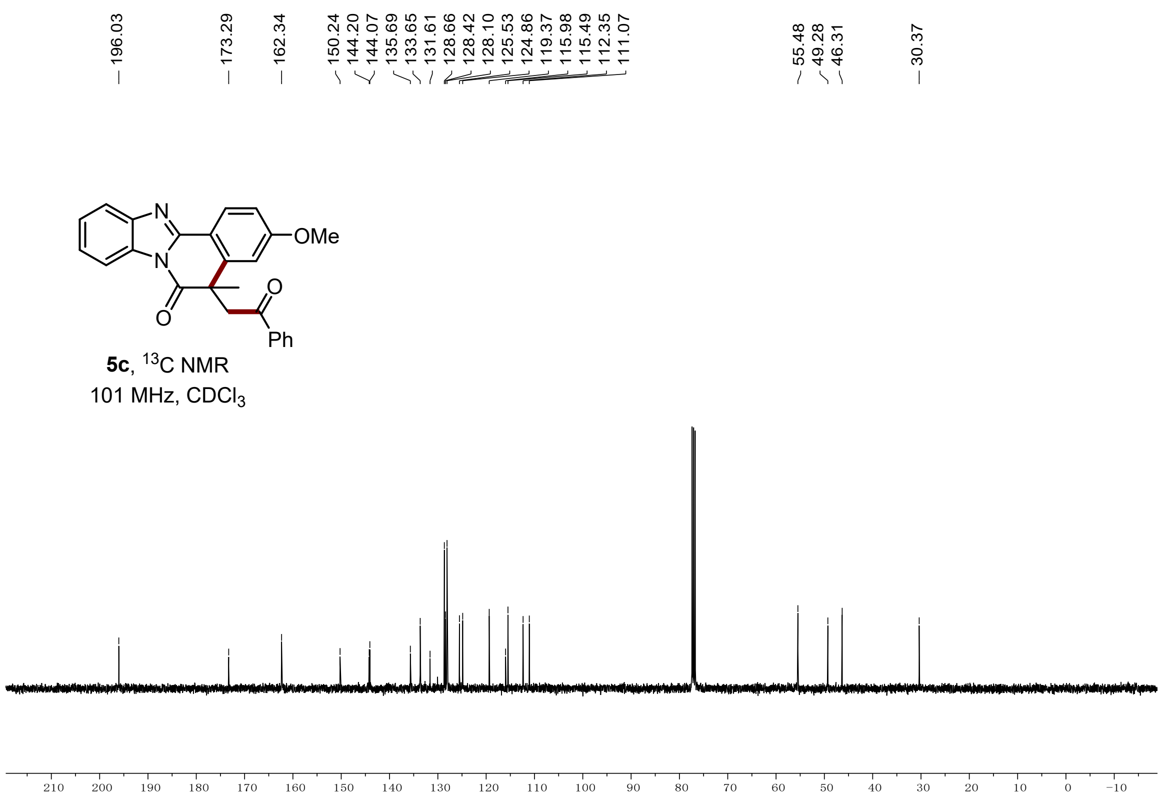
7.42
7.42
7.42
7.40
7.40
7.40
7.39
7.38
7.37
7.26
6.99
6.98
6.96
6.96
6.81
6.80
4.32
4.27
4.10
4.06
3.80
1.70
N
OMe
N
O
O
Ph
5c, 1H NMR
400 MHz, CDCl3
1.00
1.01
3.09
1.05
4.09
1.00
1.01
1.02
1.05
3.09
3.07
f1 (ppm)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173.29
162.34
150.24
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144.07
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133.65
131.61
128.66
128.42
128.10
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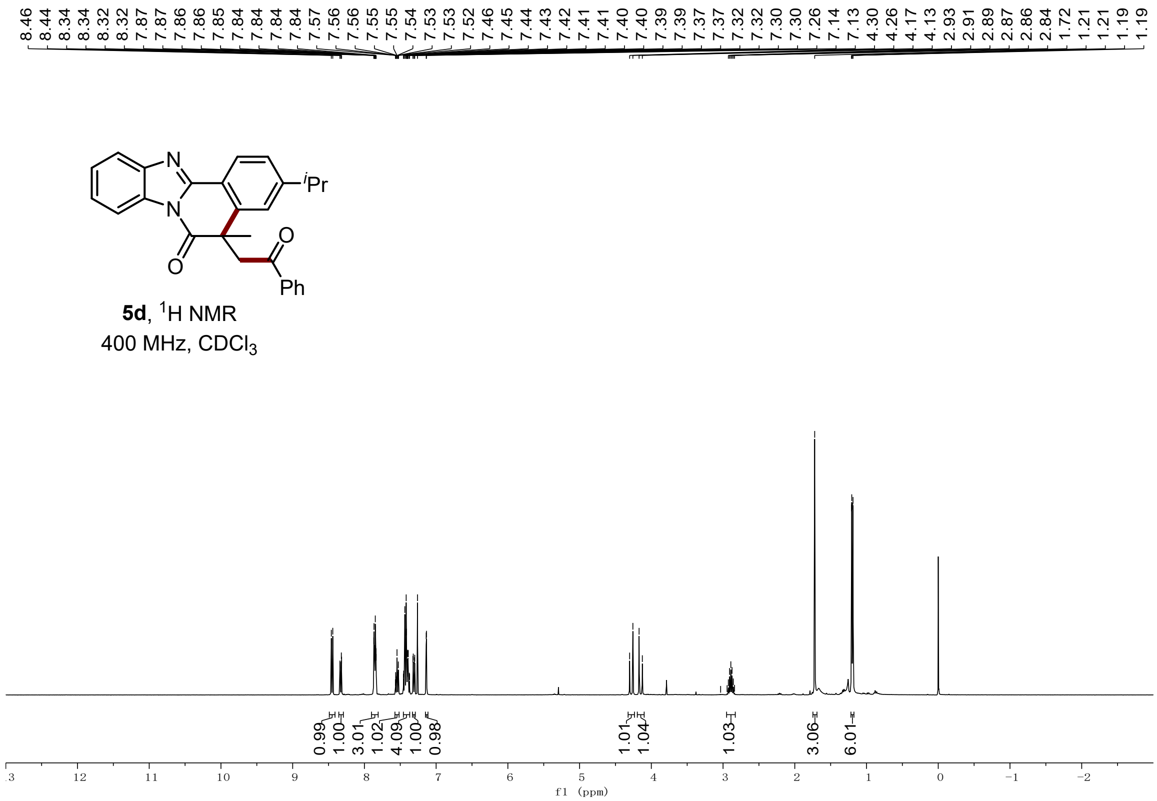
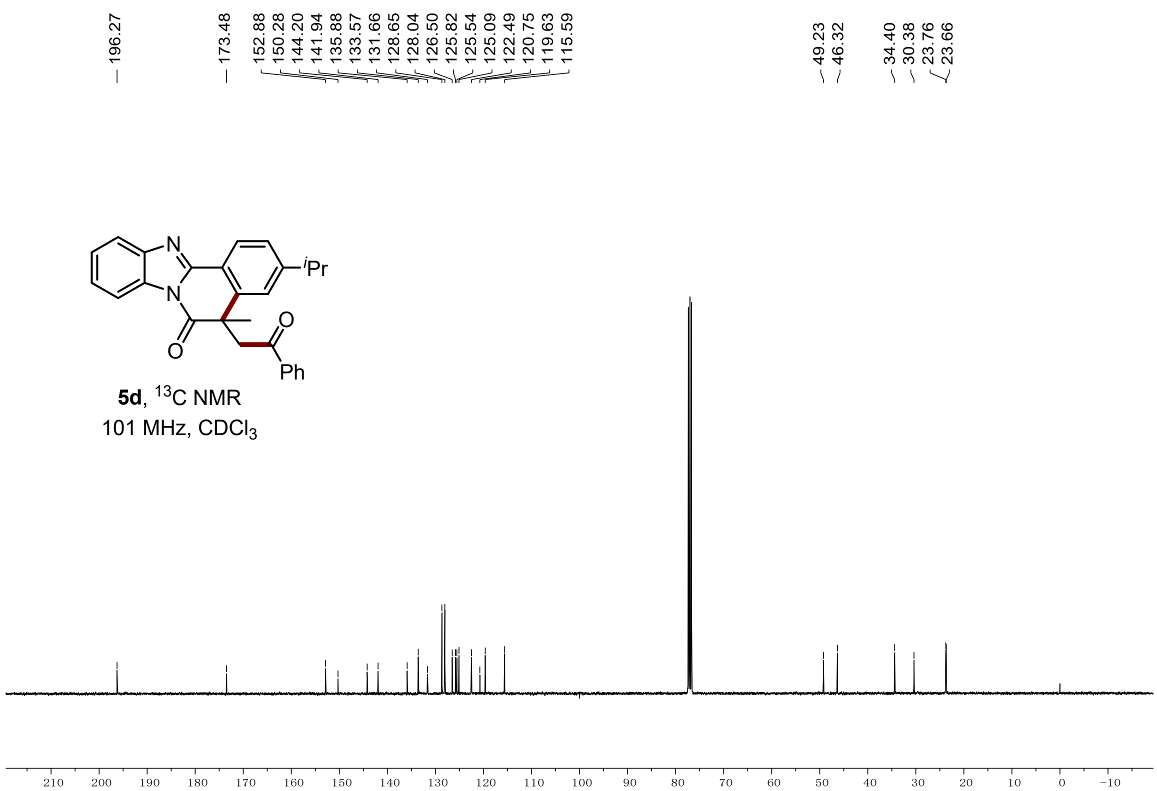
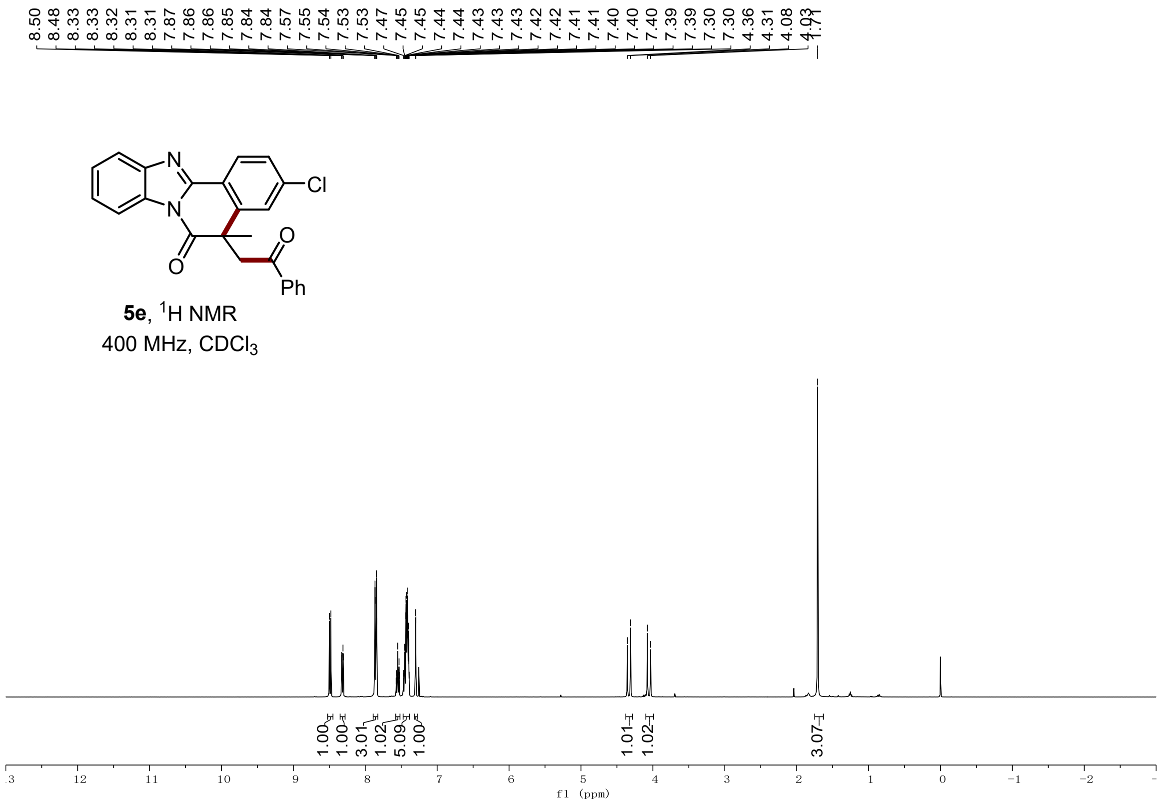
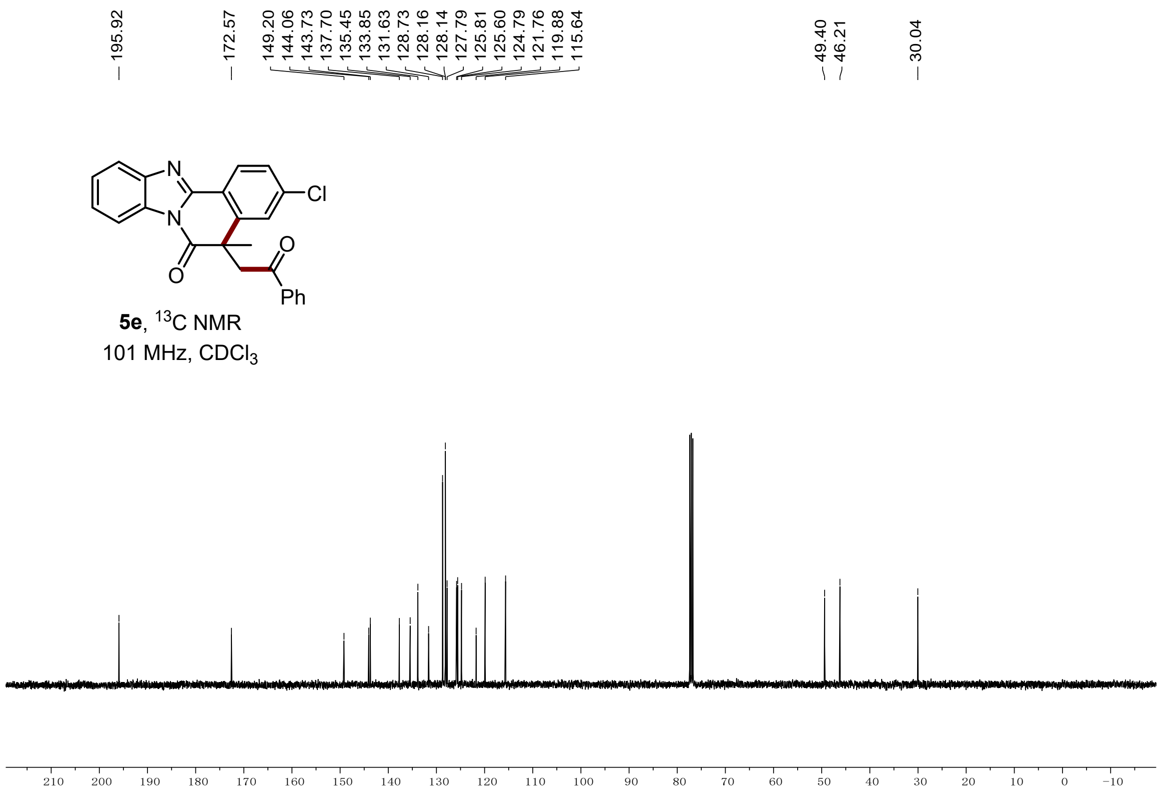
124.86
119.37
115.98
115.49
112.35
111.07
55.48
49.28
46.31
30.37
N
OMe
N
O
O
Ph
5c, 13C NMR
101 MHz, CDCl3
f1 (ppm)


8.46 8.44 8.34 8.34 8.32 8.32 7.87 7.87 7.86 7.85 7.84 7.84 7.84 7.84 7.57 7.56 7.55 7.55 7.54 7.53 7.53 7.52 7.46 7.45 7.44 7.43 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.37 7.37 7.32 7.32 7.30 7.26 7.14 7.13 4.30 4.26 4.17 4.13 2.93 2.91 2.89 2.87 2.86 2.84 1.72 1.21 1.21 1.19 1.19
N
N
O
O
Ph
^{i}Pr
5d, ^{1}H NMR
400 MHz, $CDCl_3$
0.99 1.00 3.01 1.02 4.09 1.00 0.98 1.01 1.04 1.03 3.06 6.01
f1 (ppm)

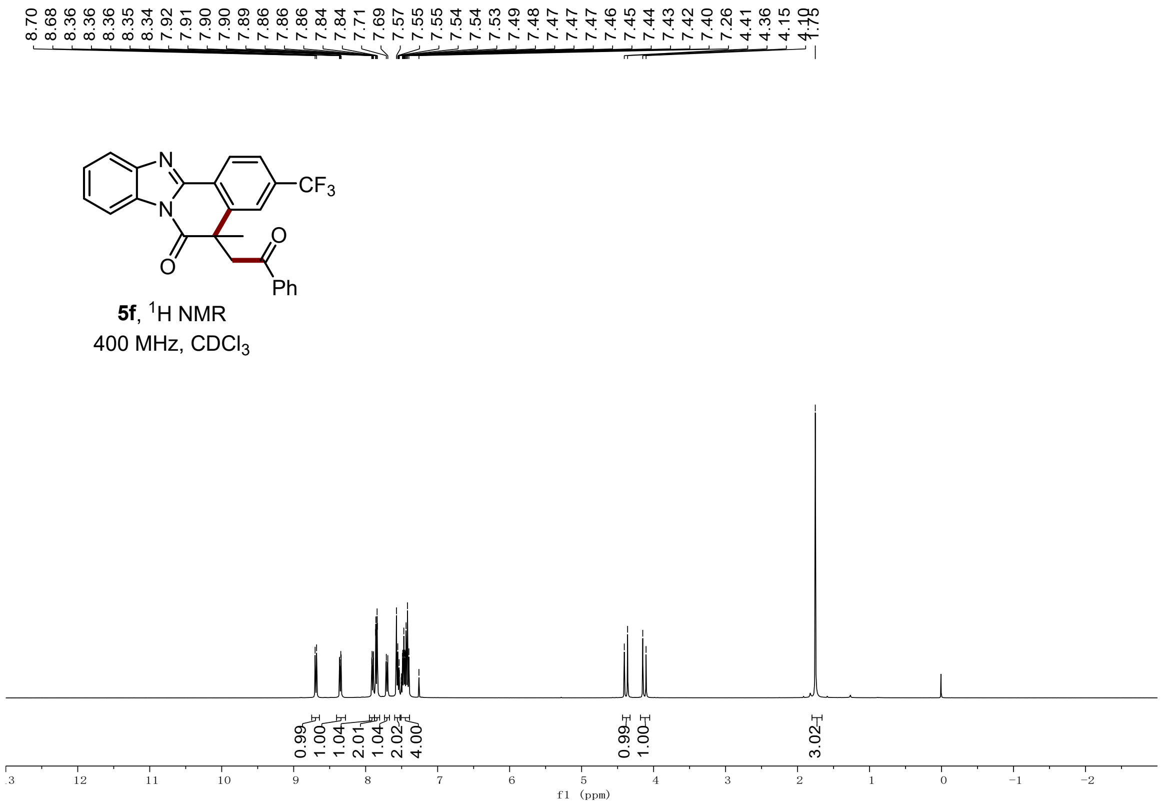

196.27 173.48 152.88 150.28 144.20 141.94 135.88 133.57 131.66 128.65 128.04 126.50 125.82 125.54 125.09 122.49 120.75 119.63 115.59 49.23 46.32 34.40 30.38 23.76 23.66
N
N
O
O
Ph
^{i}Pr
5d, ^{13}C NMR
101 MHz, $CDCl_3$
f1 (ppm)



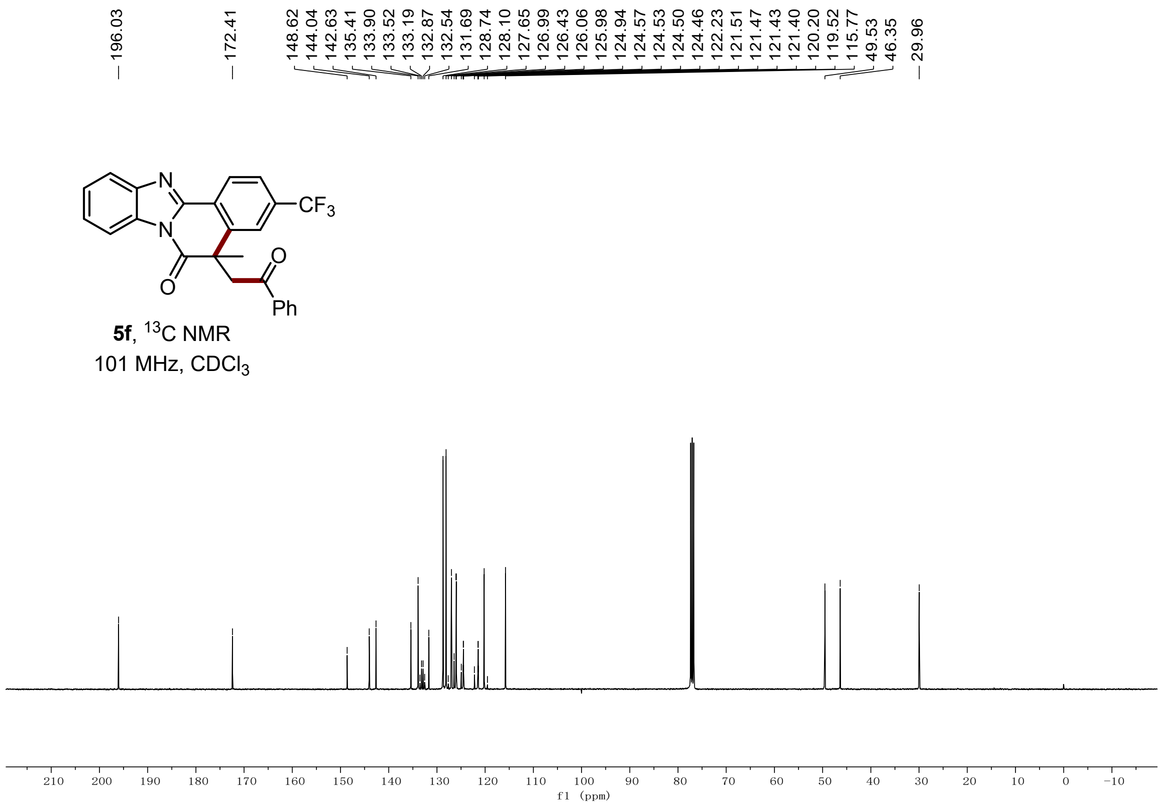
5e, ^{1}H NMR
400 MHz, $CDCl_3$



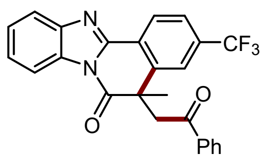
5e, ^{13}C NMR
101 MHz, $CDCl_3$



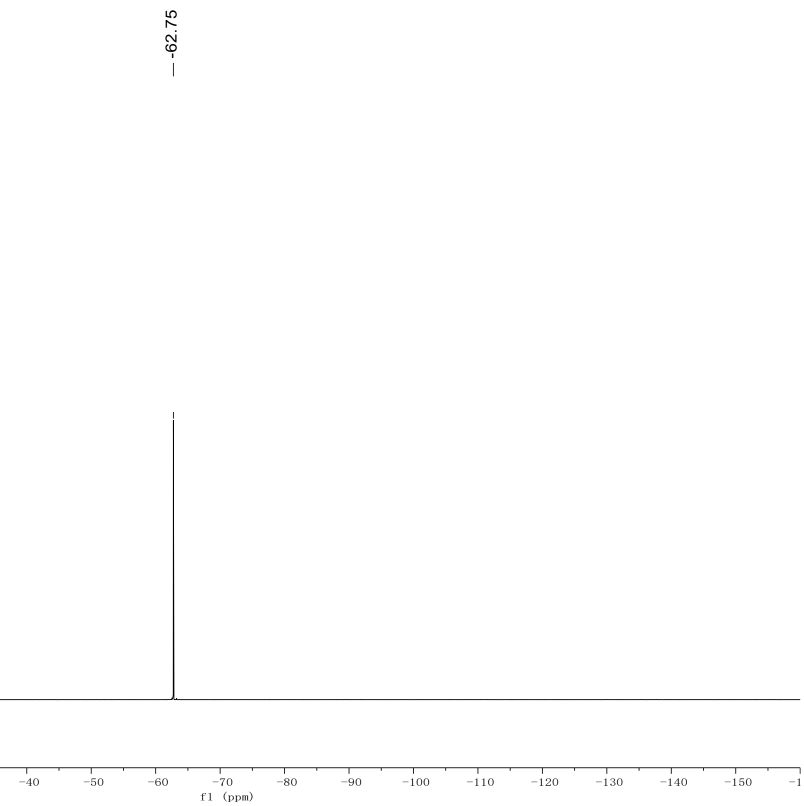
5f, ¹H NMR
400 MHz, $CDCl_3$

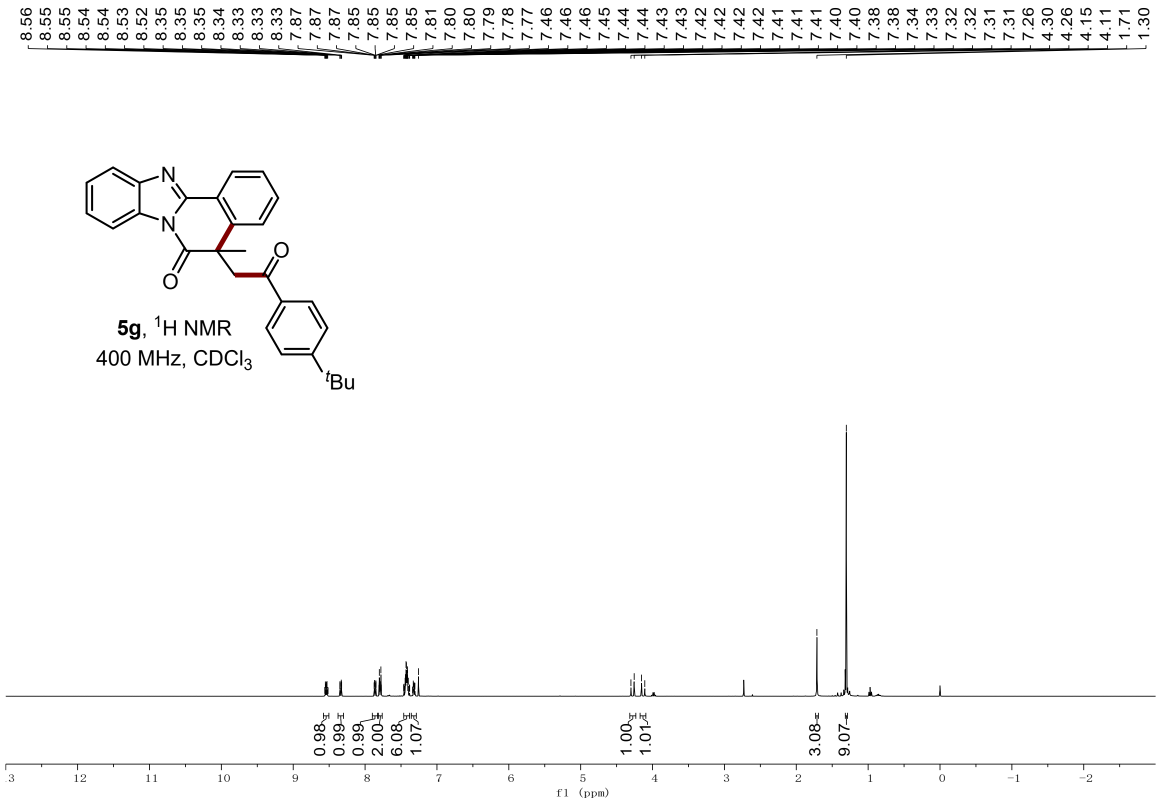


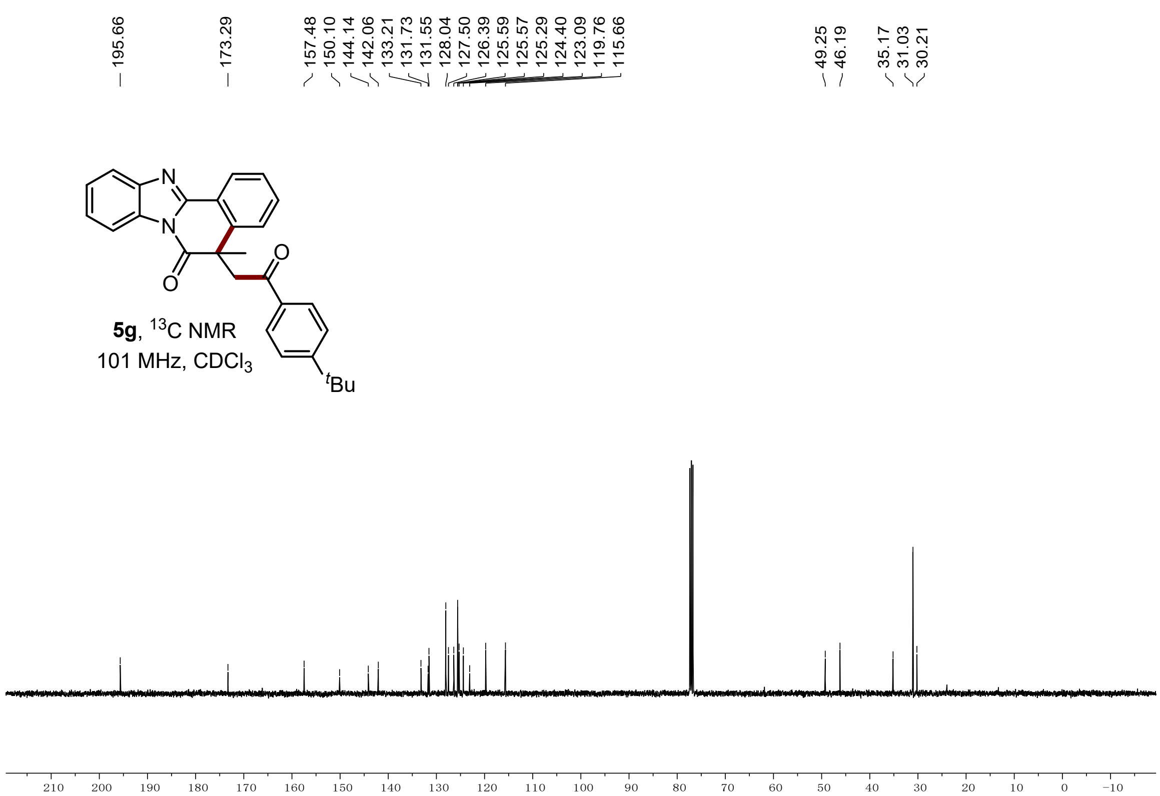
5f, ¹³C NMR
101 MHz, $CDCl_3$



5f, ^{19}F NMR
376 MHz, $CDCl_3$




8.56 8.55 8.55 8.54 8.53 8.52 8.35 8.35 8.35 8.34 8.33 8.33 8.33 7.87 7.87 7.87 7.85 7.85 7.85 7.85 7.81 7.80 7.80 7.79 7.78 7.77 7.46 7.46 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.42 7.42 7.41 7.41 7.41 7.40 7.40 7.38 7.38 7.34 7.33 7.32 7.32 7.31 7.31 7.26 4.30 4.26 4.15 4.11 1.71 1.30
N
N
O
O
tBu
5g, 1H NMR
400 MHz, CDCl3
0.98 0.99 0.99 2.00 6.08 1.07 1.00 1.01 3.08 9.07
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)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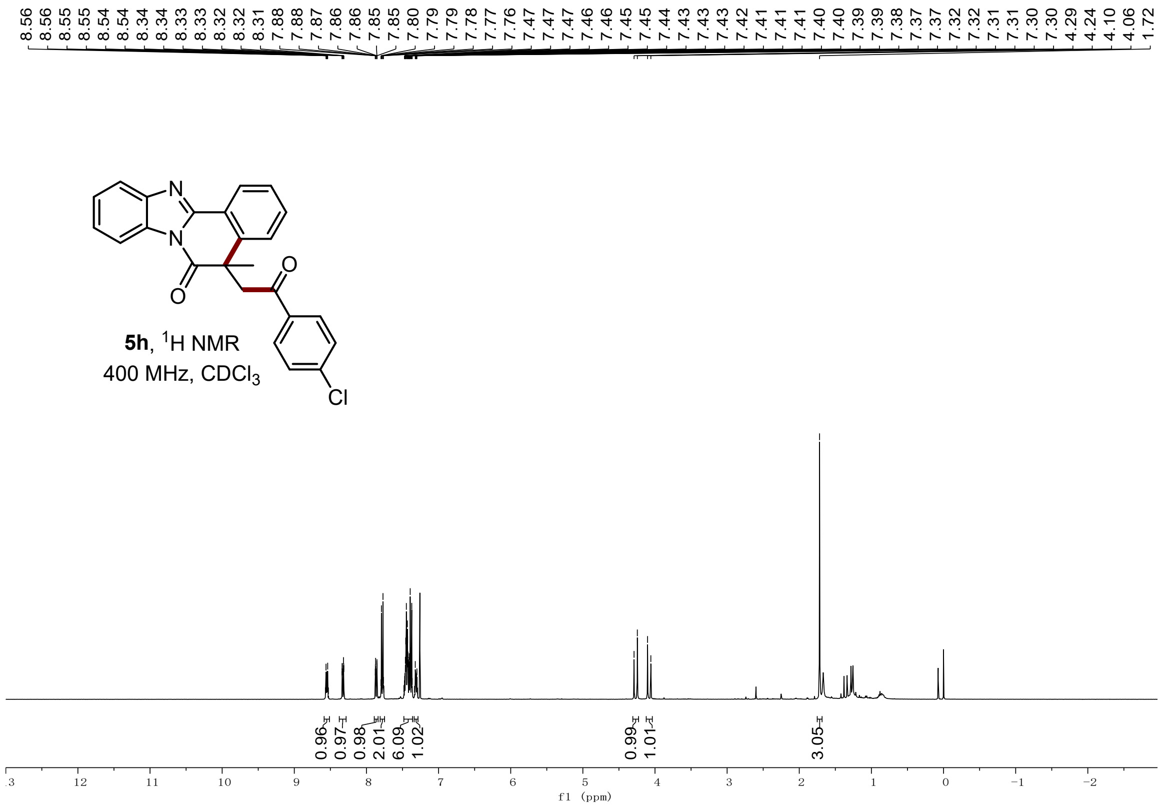
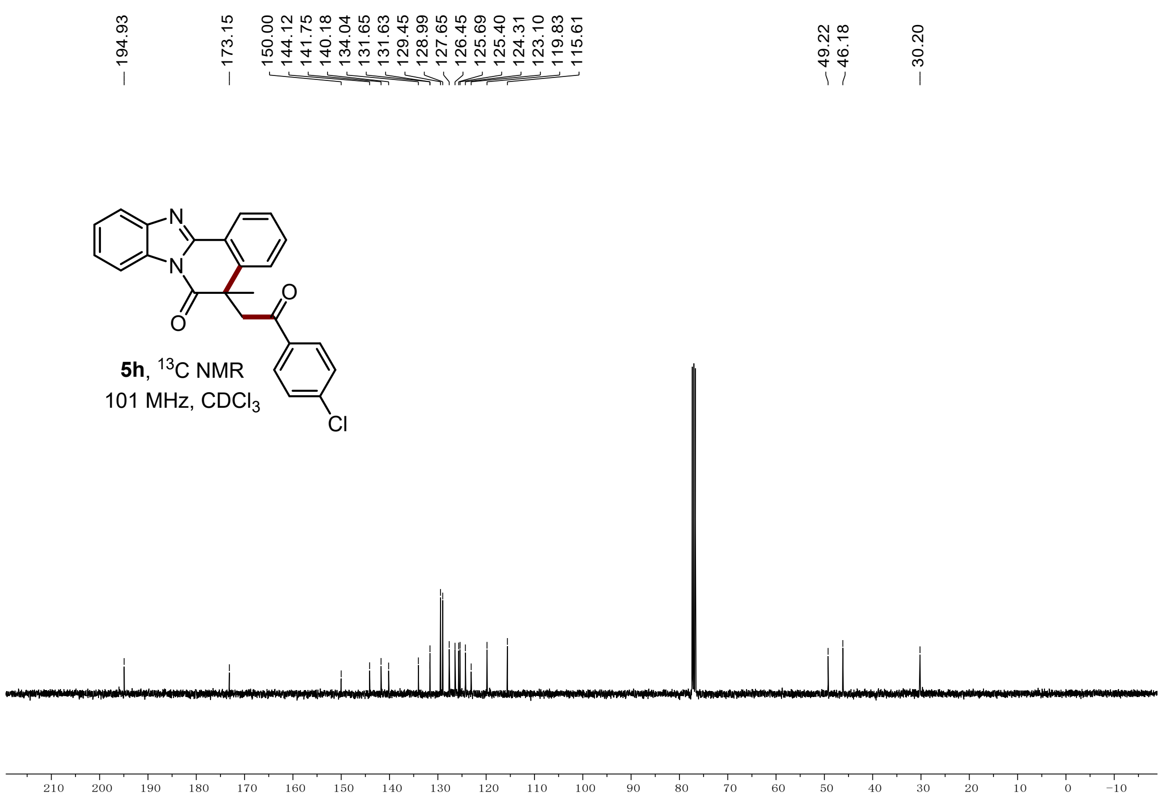
N
N
O
O
tBu
5g, 13C NMR
101 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.56 8.56 8.55 8.55 8.54 8.54 8.34 8.34 8.33 8.33 8.32 8.32 8.31 7.88 7.88 7.87 7.86 7.85 7.85 7.80 7.79 7.79 7.78 7.77 7.76 7.47 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.43 7.43 7.43 7.42 7.41 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.32 7.32 7.31 7.31 7.30 4.29 4.24 4.10 4.06 1.72
N
N
O
O
Cl
5h, 1H NMR
400 MHz, CDCl3
0.96 0.97 0.98 2.01 6.09 1.02
0.99 1.01
3.05
f1 (ppm)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173.15
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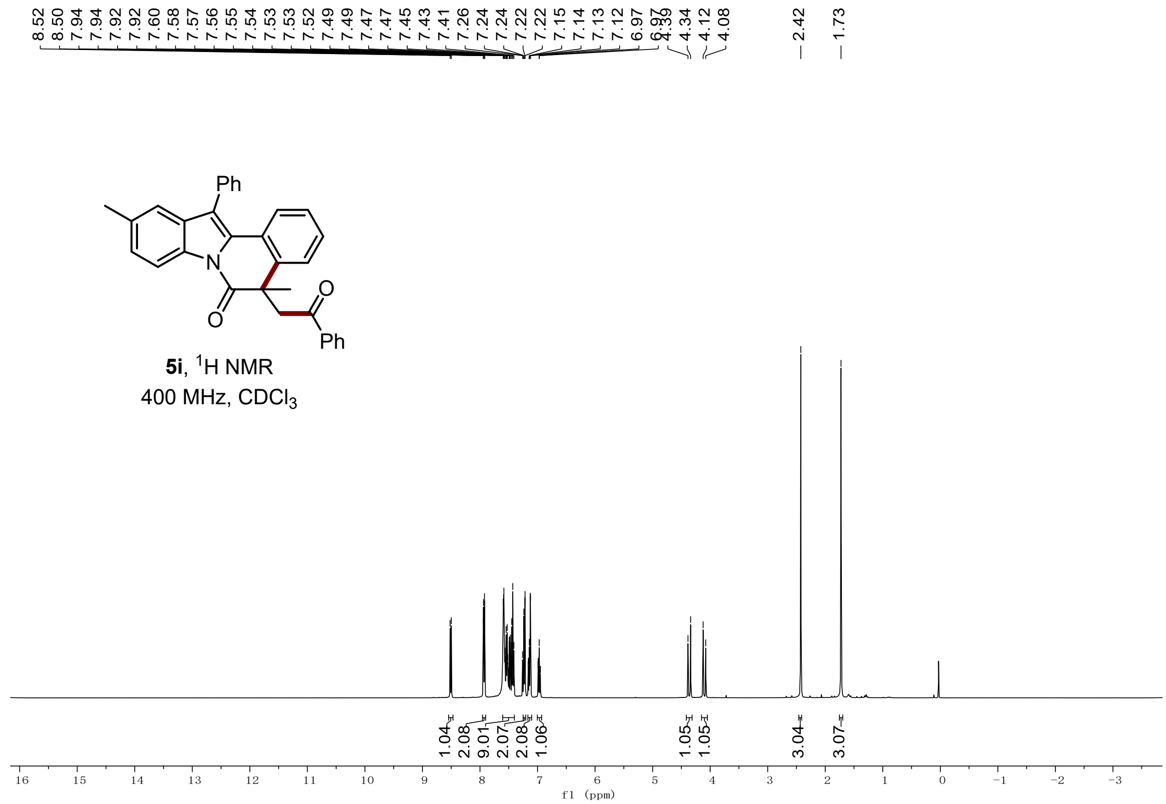
49.22 46.18
30.20
N
N
O
O
Cl
5h, 13C NMR
101 MHz, CDCl3
f1 (ppm)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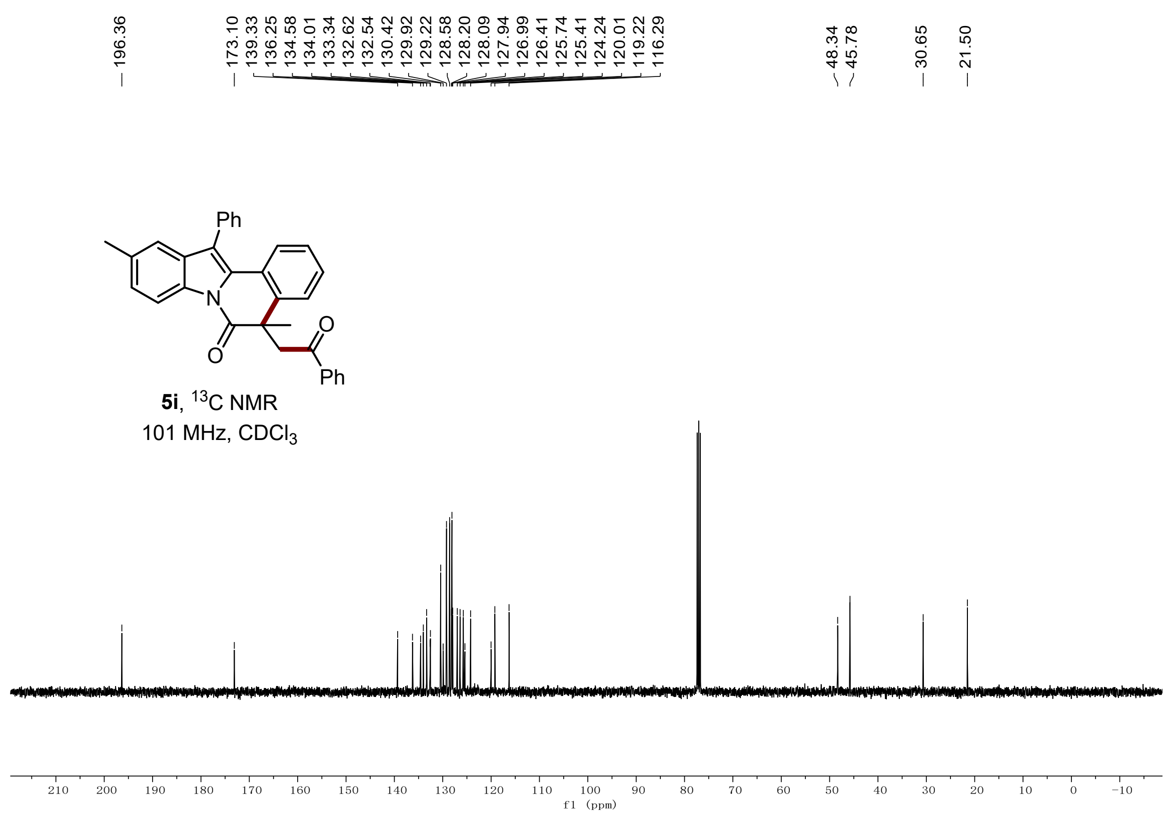
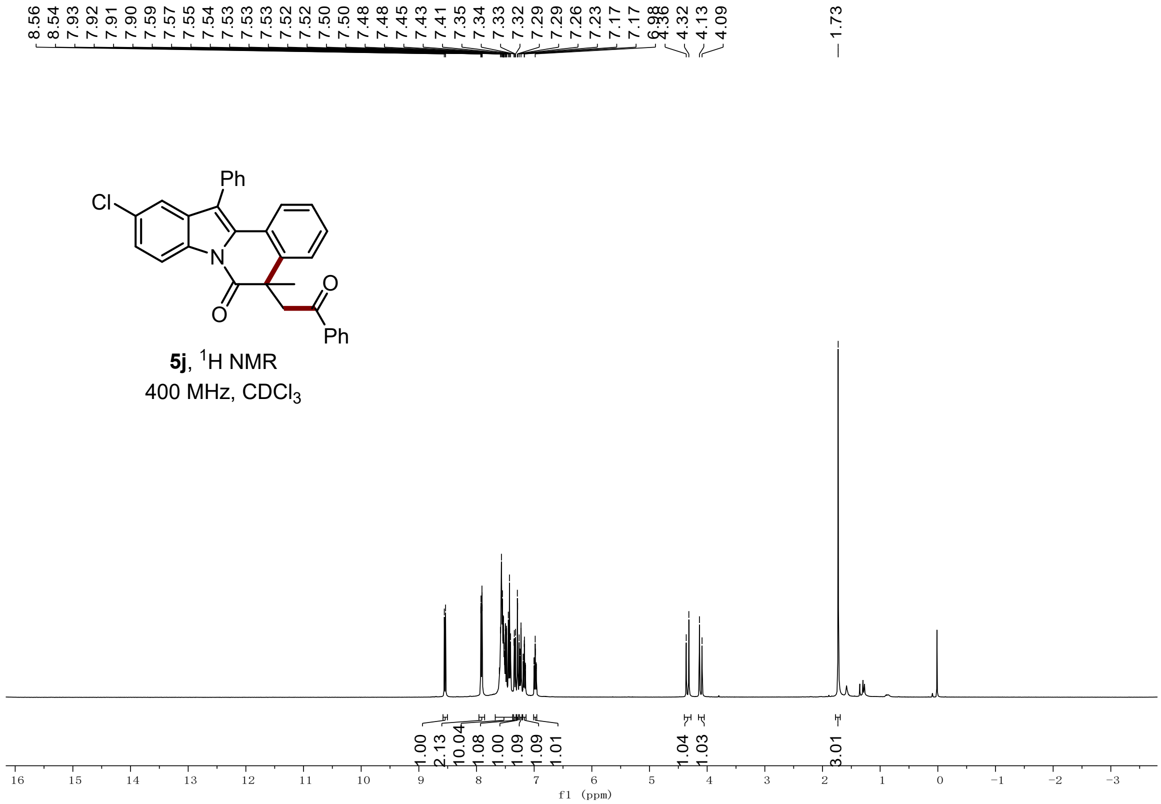
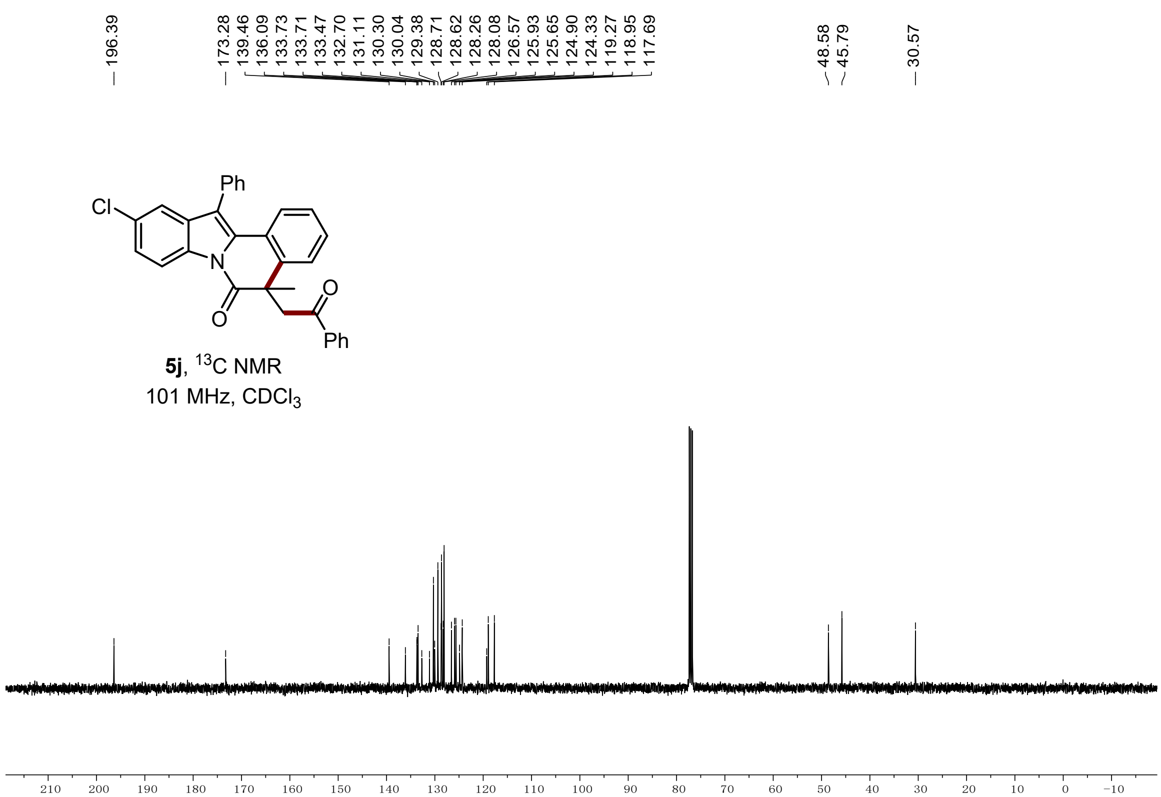
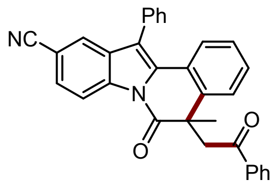
2.42
1.73
Ph
N
O
O
Ph
5i, 1H NMR
400 MHz, CDCl3
1.04 2.08 9.01 2.07 2.08 1.06
1.05 1.05
3.04
3.07
f1 (ppm)

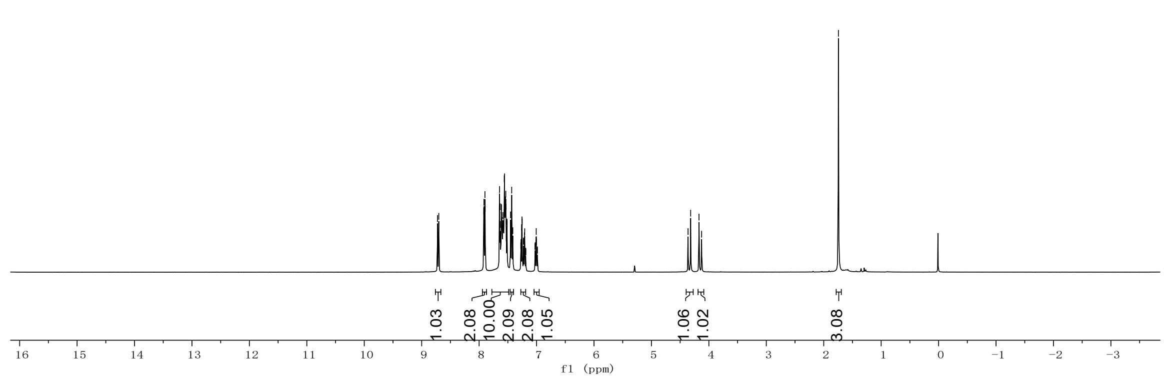

196.36
173.10 139.33 136.25 134.58 134.01 133.34 132.62 132.54 130.42 129.92 129.22 128.58 128.20 128.09 127.94 126.99 126.41 125.74 125.41 124.24 120.01 119.22 116.29
48.34 45.78
30.65
21.50
Ph
N
O
O
Ph
5i, 13C NMR
101 MHz, CDCl3
f1 (ppm)

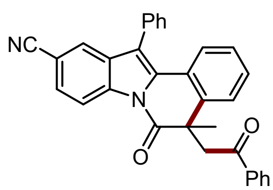

8.56 8.54 7.93 7.92 7.91 7.90 7.59 7.57 7.55 7.54 7.53 7.53 7.53 7.52 7.52 7.50 7.50 7.48 7.48 7.45 7.43 7.41 7.35 7.34 7.33 7.32 7.29 7.29 7.26 7.23 7.17 7.17 6.98 4.36 4.32 4.13 4.09
1.73
Ph
Cl
N
O
O
Ph
5j, ^{1}H NMR
400 MHz, $CDCl_3$
1.00 2.13 10.04 1.08 1.00 1.09 1.09 1.01
1.04 1.03
3.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


196.39
173.28 139.46 136.09 133.73 133.71 133.47 132.70 131.11 130.30 130.04 129.38 128.71 128.62 128.26 128.08 126.57 125.93 125.65 124.90 124.33 119.27 118.95 117.69
48.58 45.79
30.57
Ph
Cl
N
O
O
Ph
5j, ^{13}C NMR
101 MHz, $CDCl_3$
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

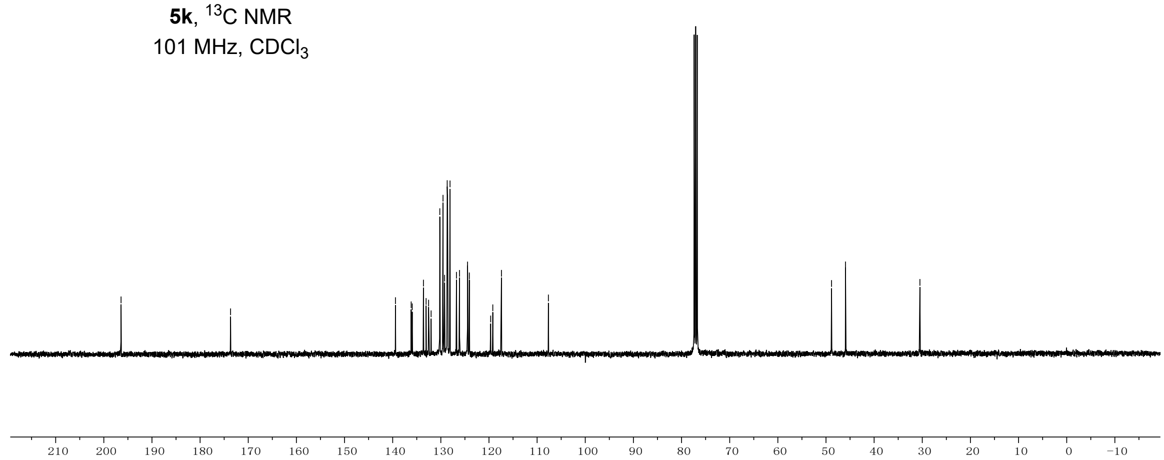


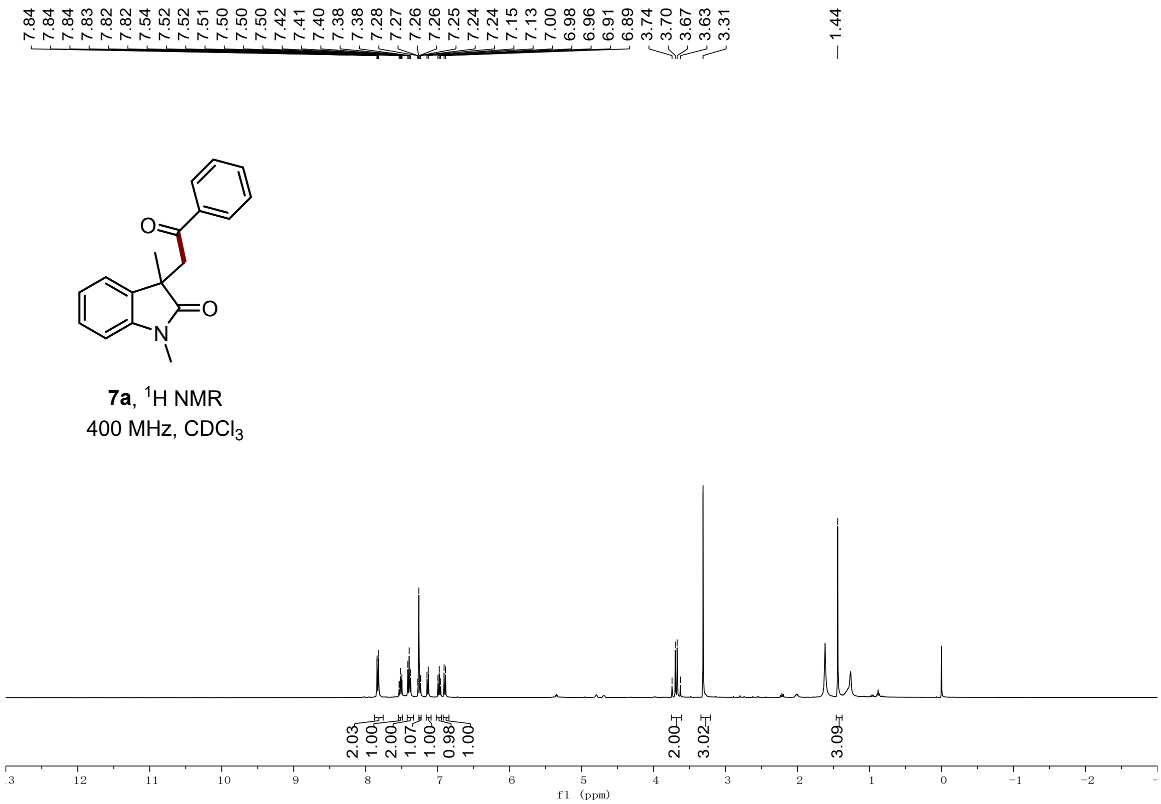
5k, ^{1}H NMR
400 MHz, $CDCl_3$



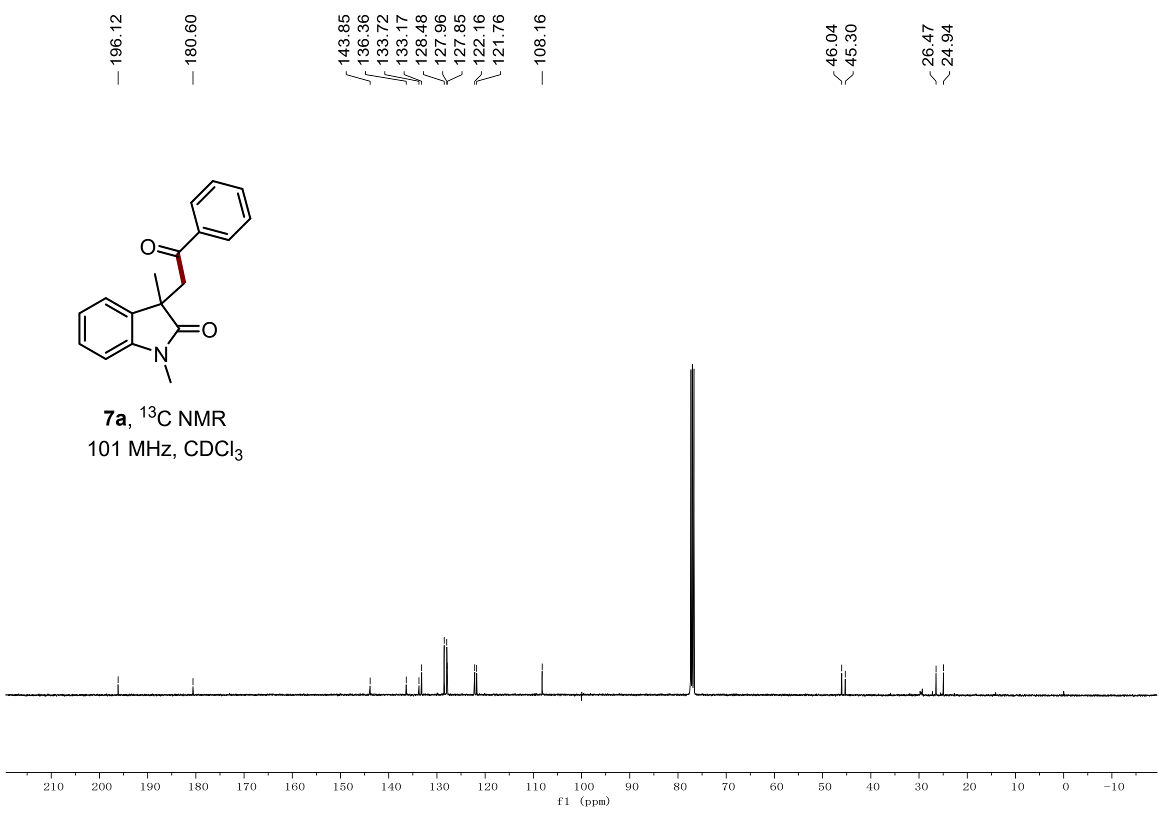


5k, ^{13}C NMR
101 MHz, $CDCl_3$

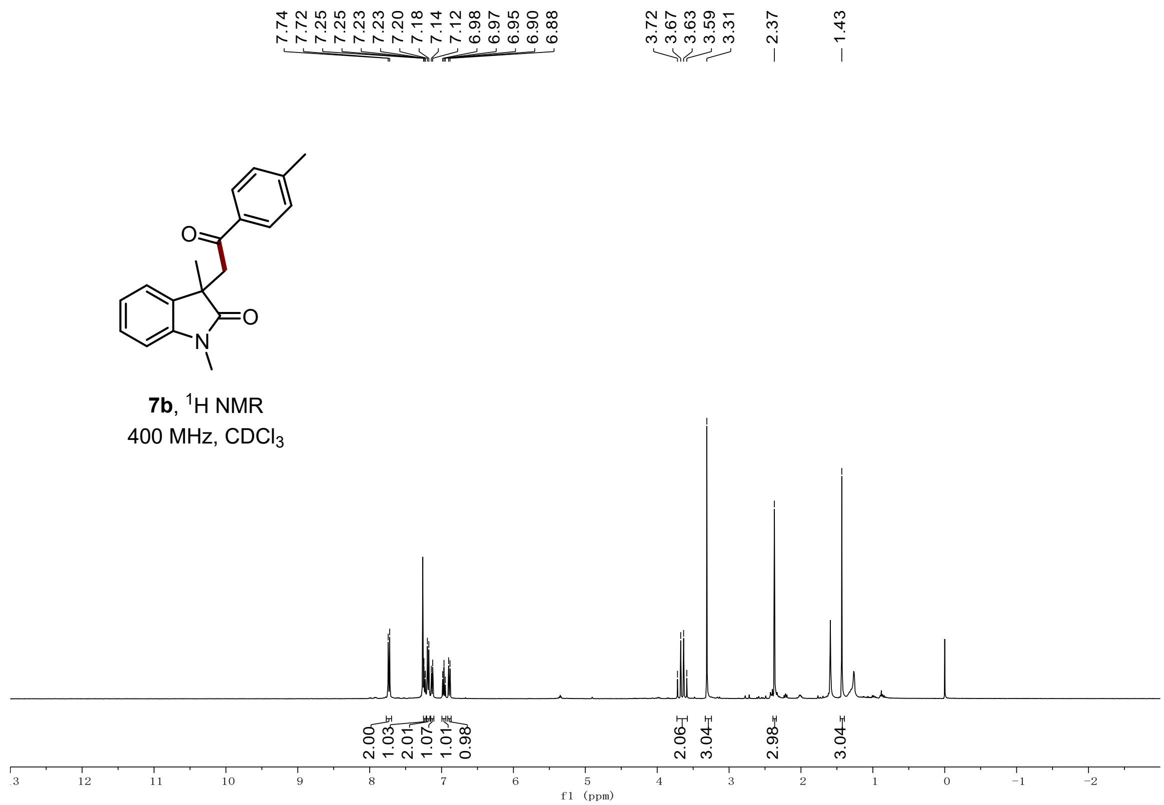




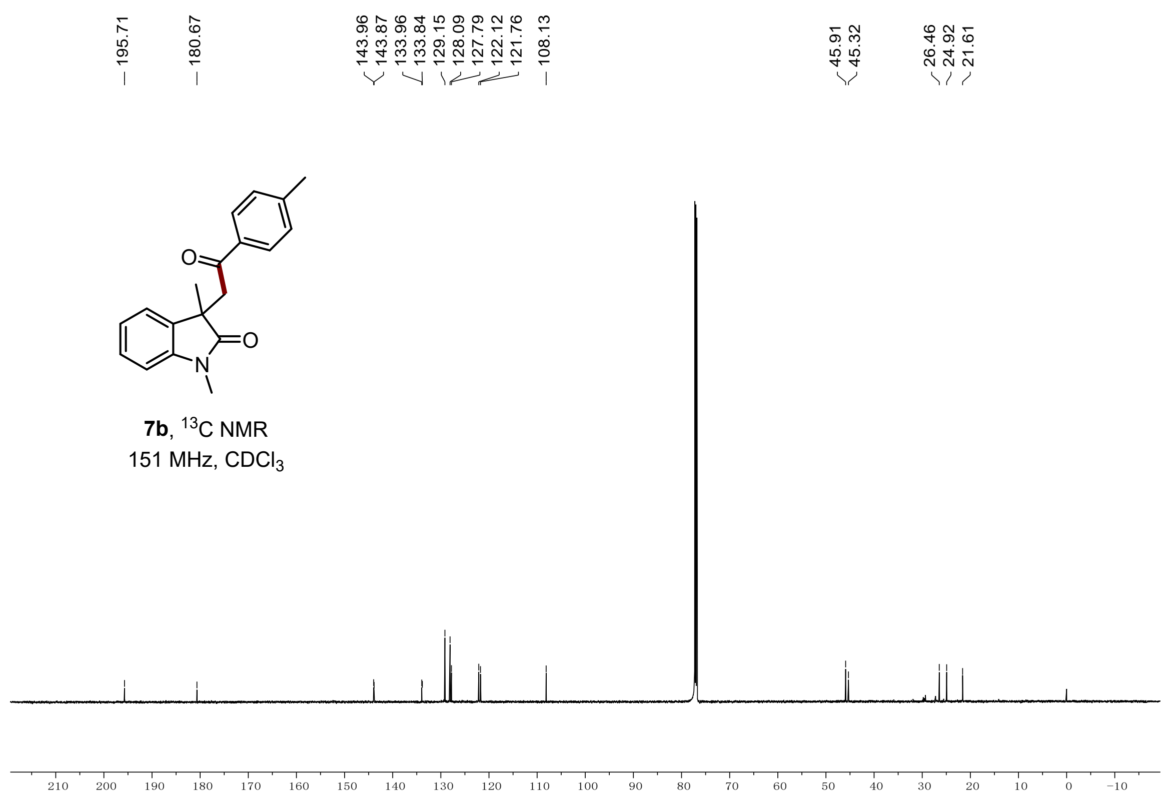
7a, ^{1}H NMR
400 MHz, $CDCl_3$



7a, ^{13}C NMR
101 MHz, $CDCl_3$



7.74
7.72
7.25
7.25
7.23
7.23
7.20
7.18
7.14
7.12
6.98
6.97
6.95
6.90
6.88
3.72
3.67
3.63
3.59
3.31
2.37
1.43
O
O
N
7b, ¹H NMR
400 MHz, CDCl3
2.00
1.03
2.01
1.07
1.01
0.98
2.06
3.04
2.98
3.04
f1 (ppm)



195.71
180.67
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143.87
133.96
133.84
129.15
128.09
127.79
122.12
121.76
108.13
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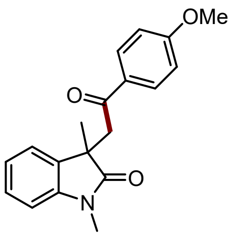
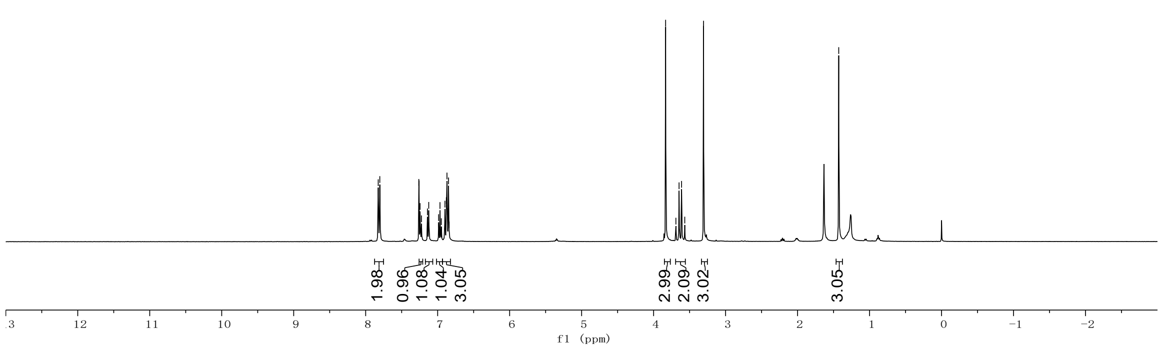
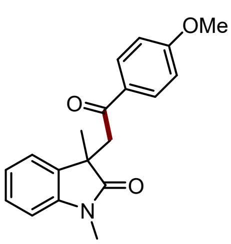
45.32
26.46
24.92
21.61
O
O
N
7b, ¹³C NMR
151 MHz, CDCl3
f1 (ppm)

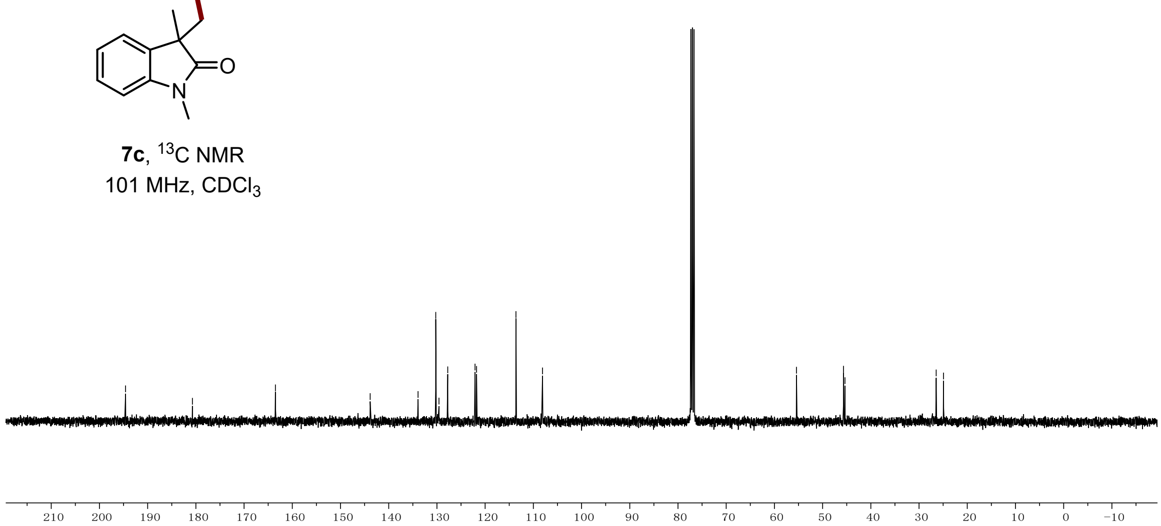


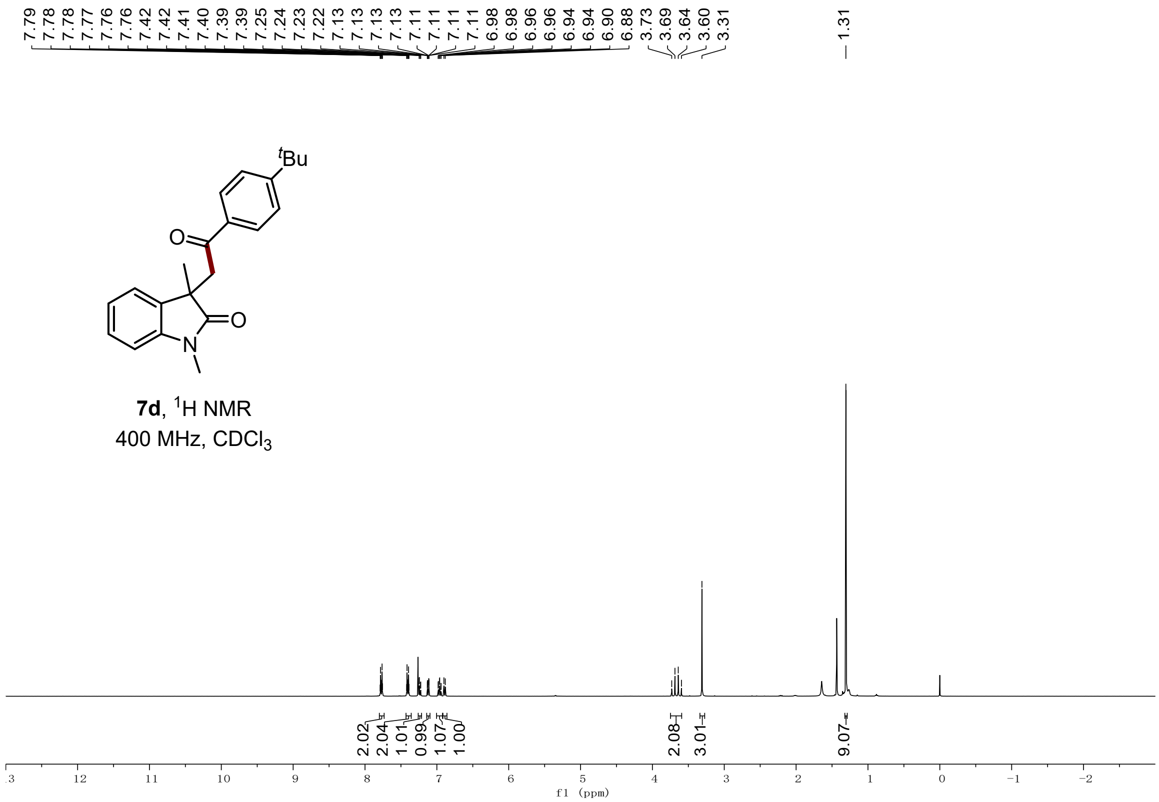
7c, ^{1}H NMR
400 MHz, $CDCl_3$



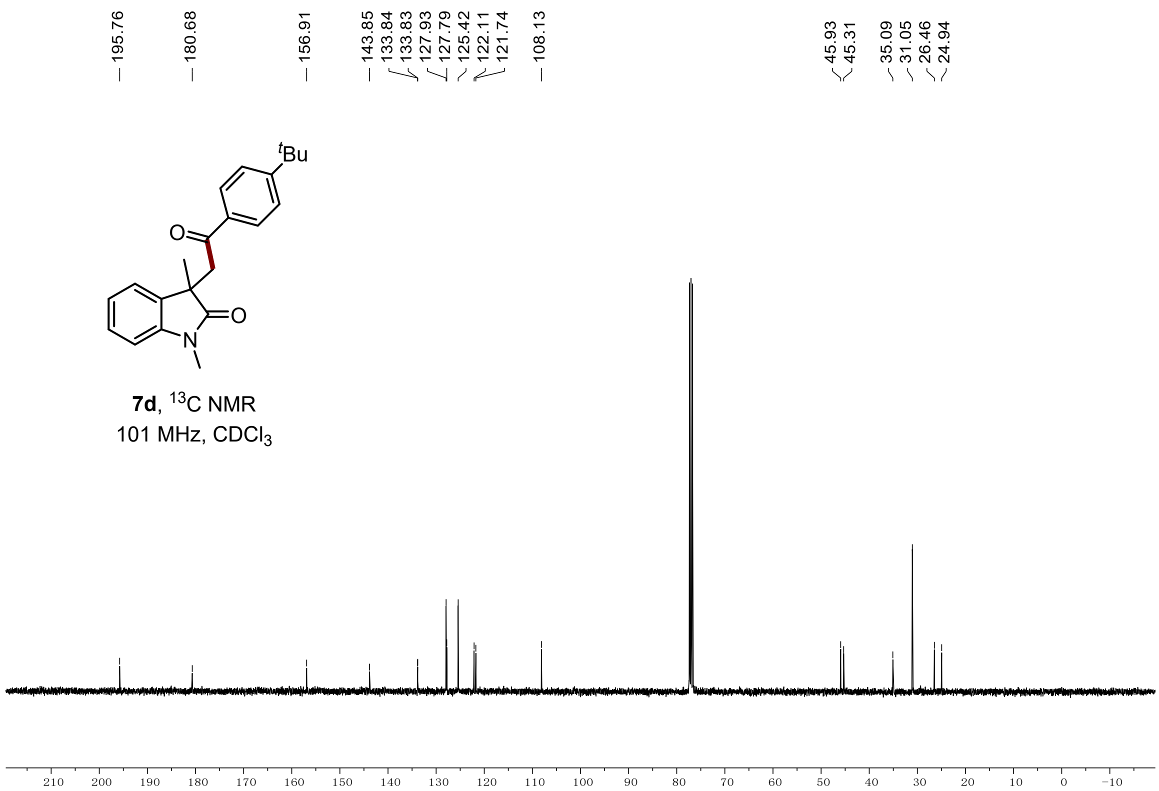


7c, ^{13}C NMR
101 MHz, $CDCl_3$

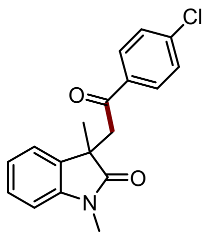




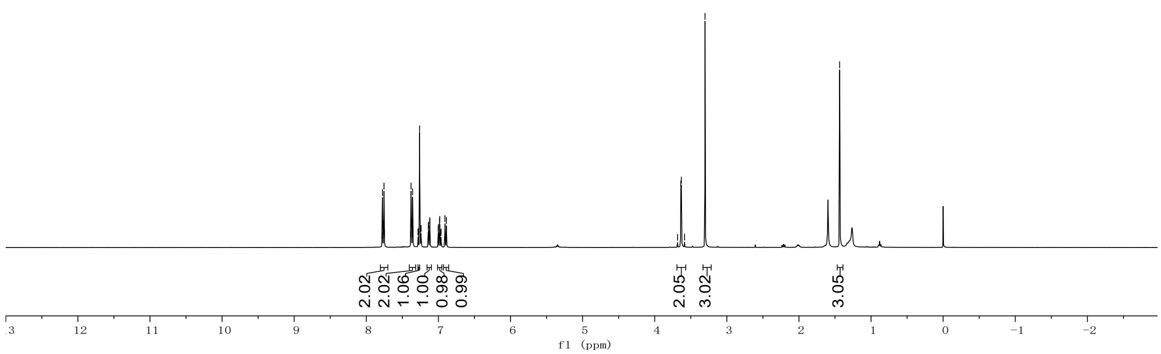
7d, ^{1}H NMR
400 MHz, $CDCl_3$

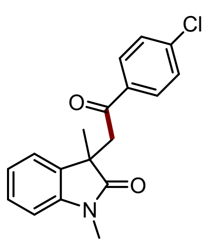


7d, ^{13}C NMR
101 MHz, $CDCl_3$



7e, ^{1}H NMR
400 MHz, $CDCl_3$





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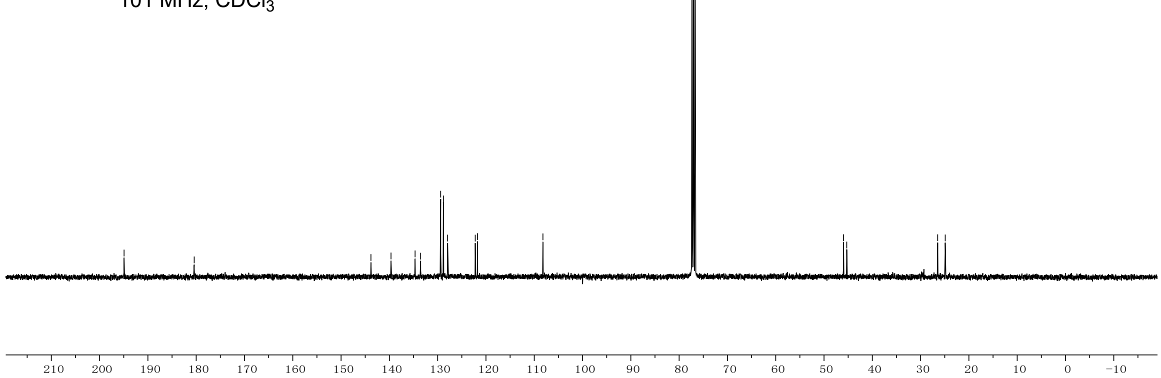
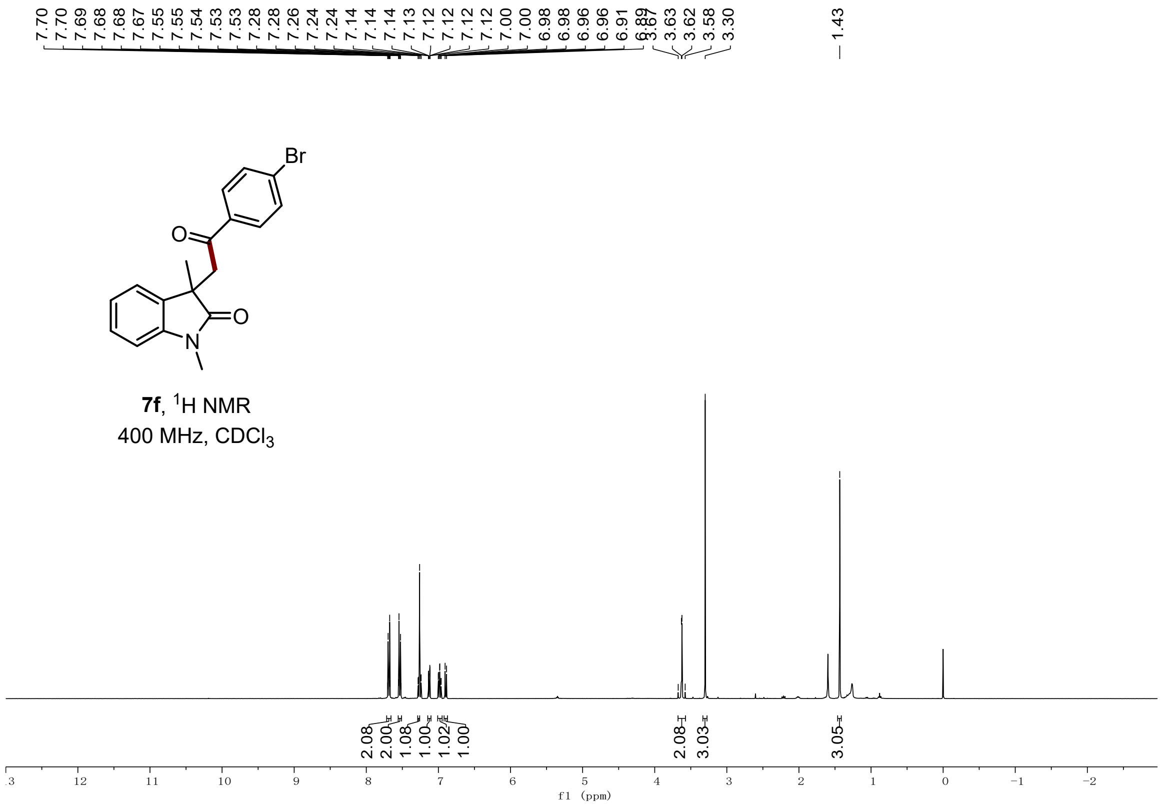
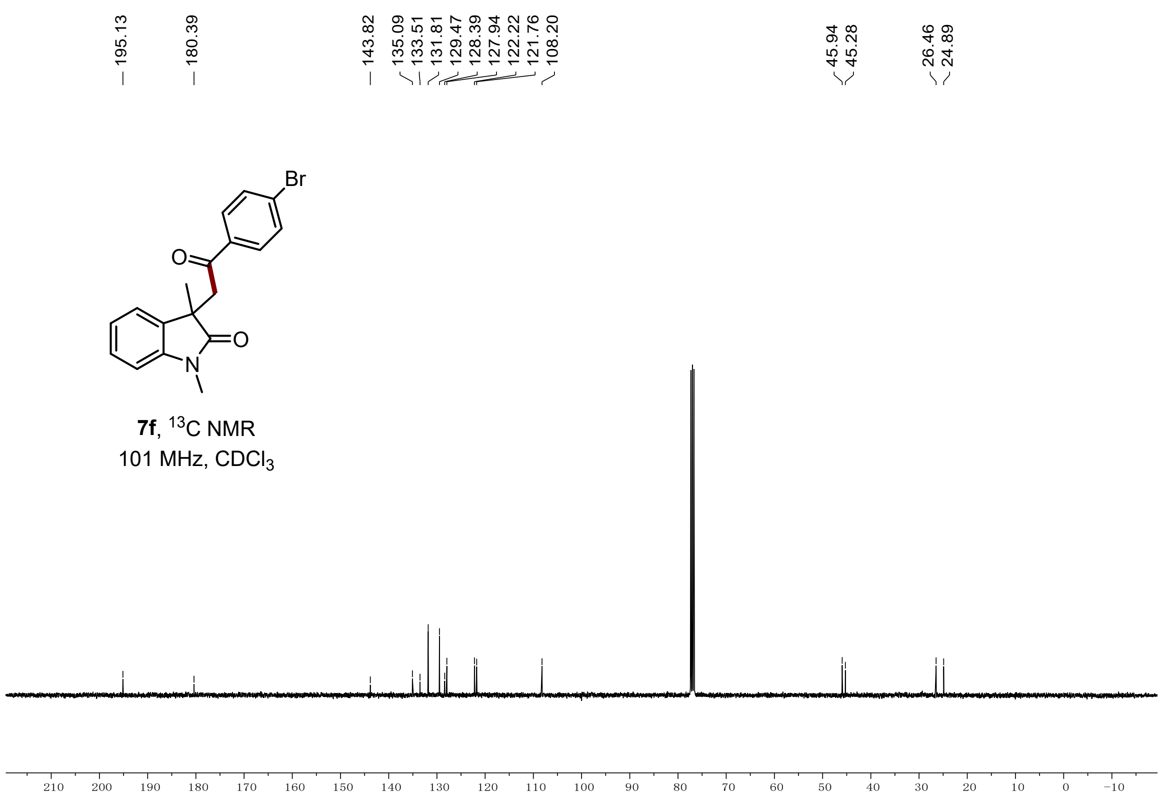
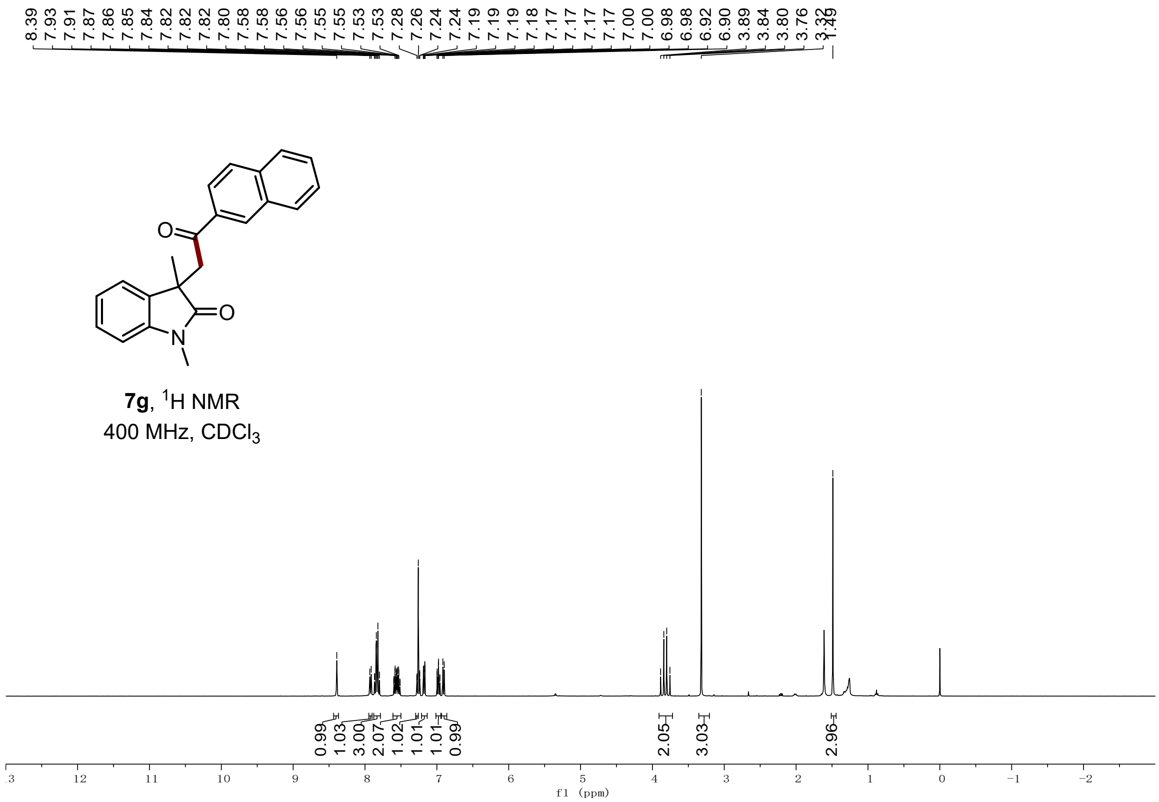




7f, ^{1}H NMR
400 MHz, $CDCl_3$



7f, ^{13}C NMR
101 MHz, $CDCl_3$



7g, ^{1}H NMR
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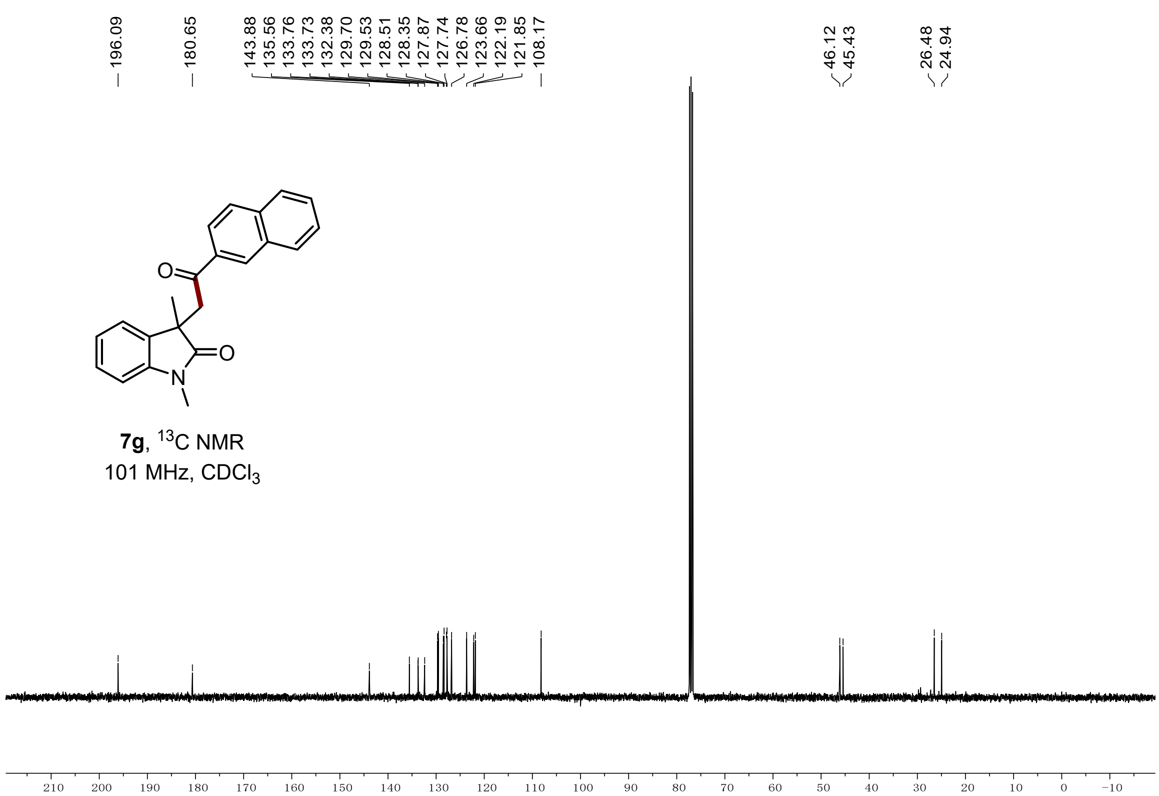
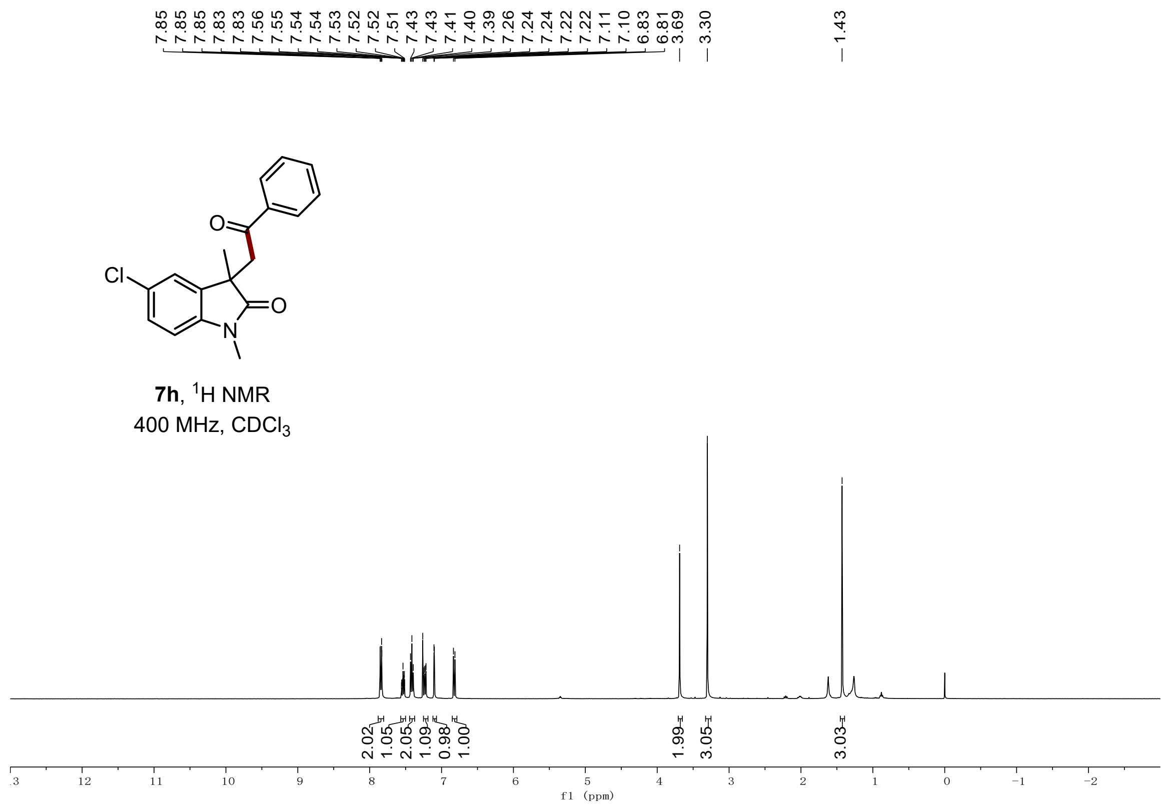
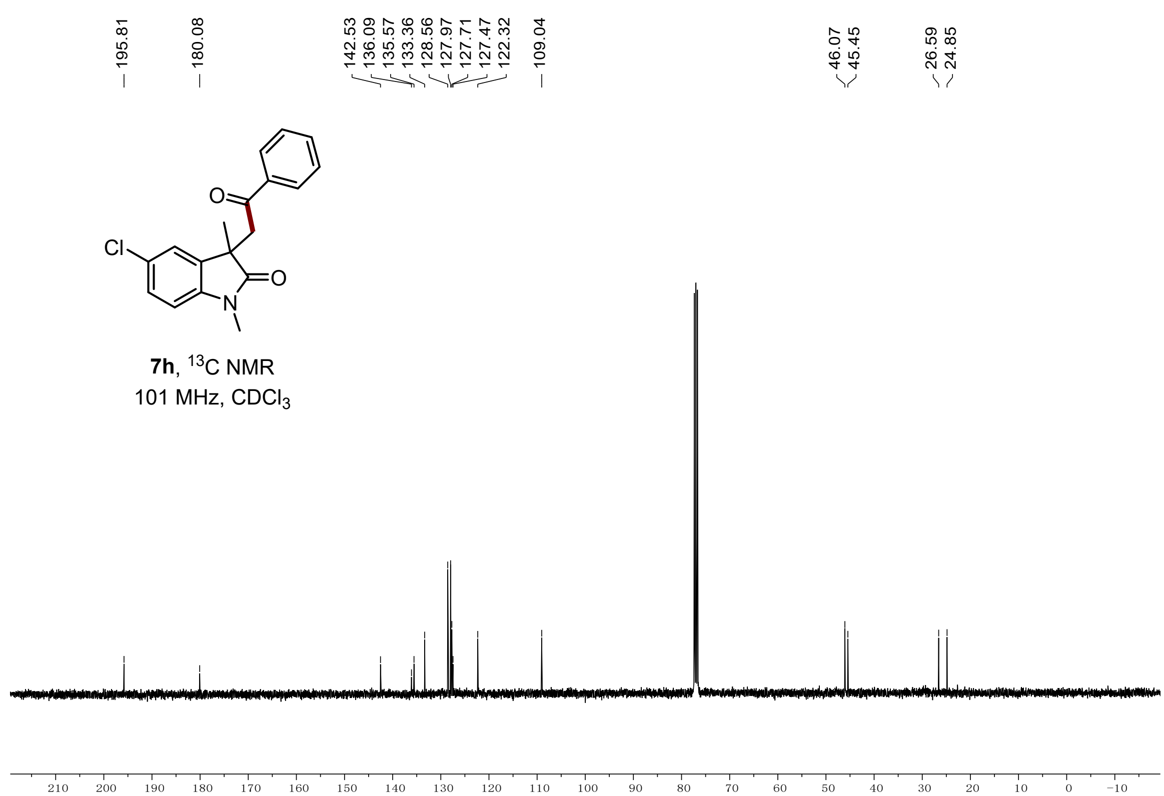
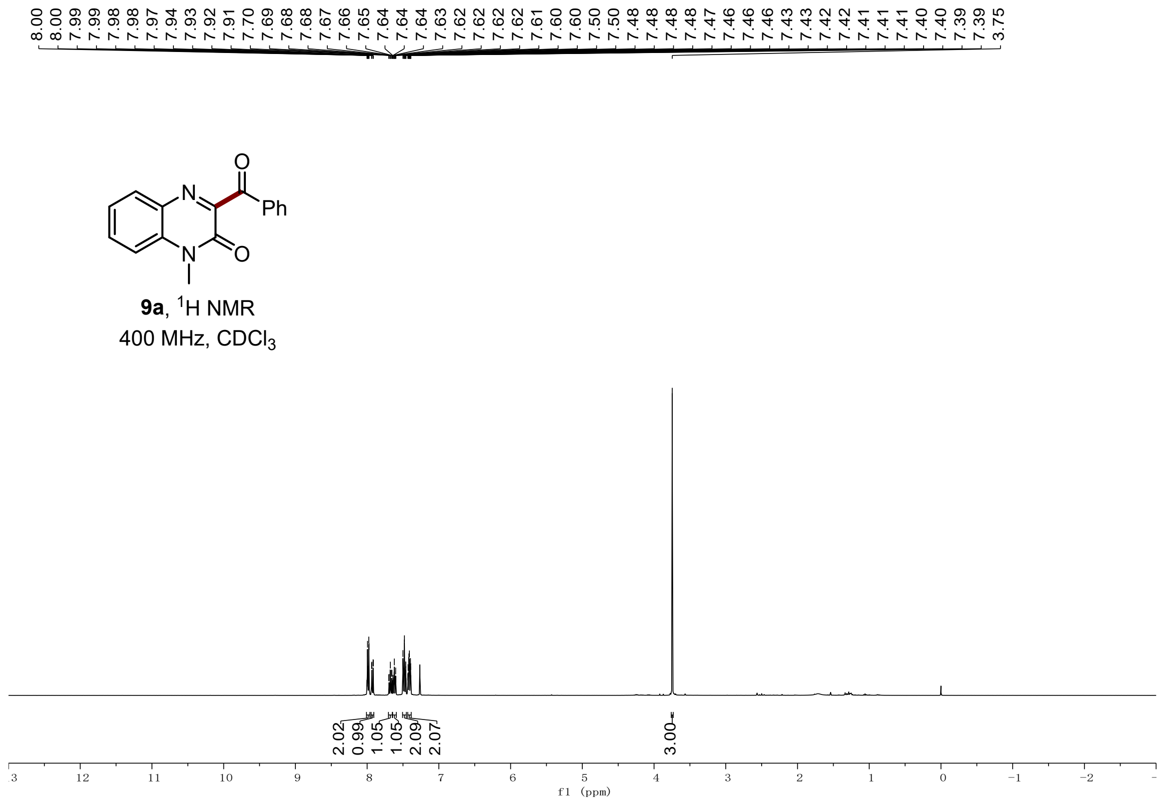


7g, ^{13}C NMR
101 MHz, $CDCl_3$



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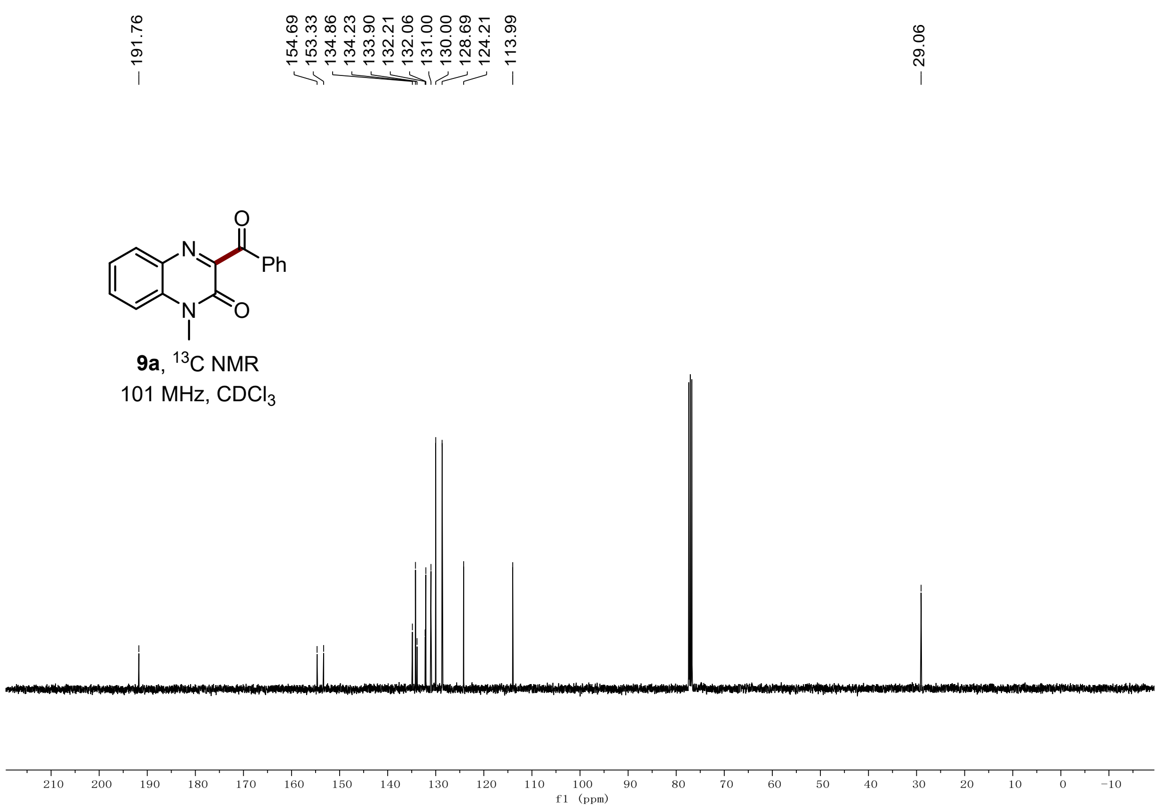
N
O
Ph
N
O
9a, 1H NMR
400 MHz, CDCl3
2.02 0.99 1.05 1.05 2.09 2.07
3.00
f1 (ppm)


191.76
154.69 153.33 134.86 134.23 133.90 132.21 132.06 131.00 130.00 128.69 124.21 113.99
29.06
N
O
Ph
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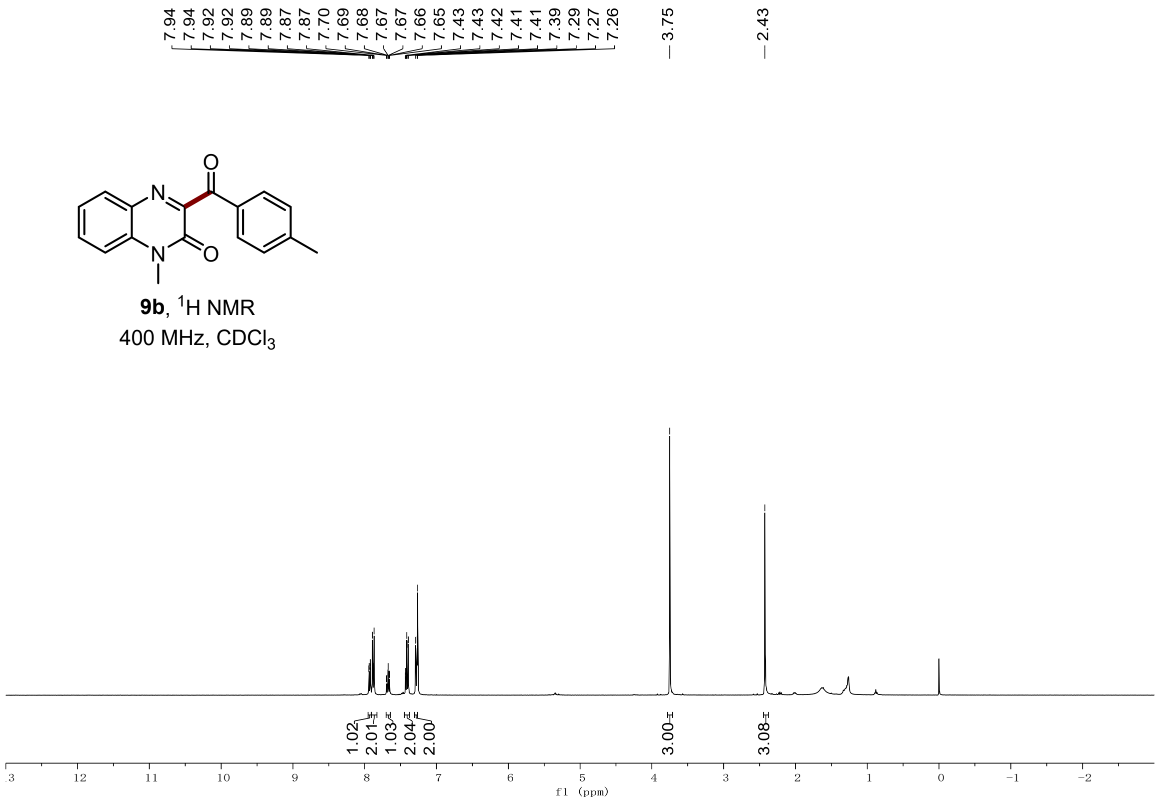
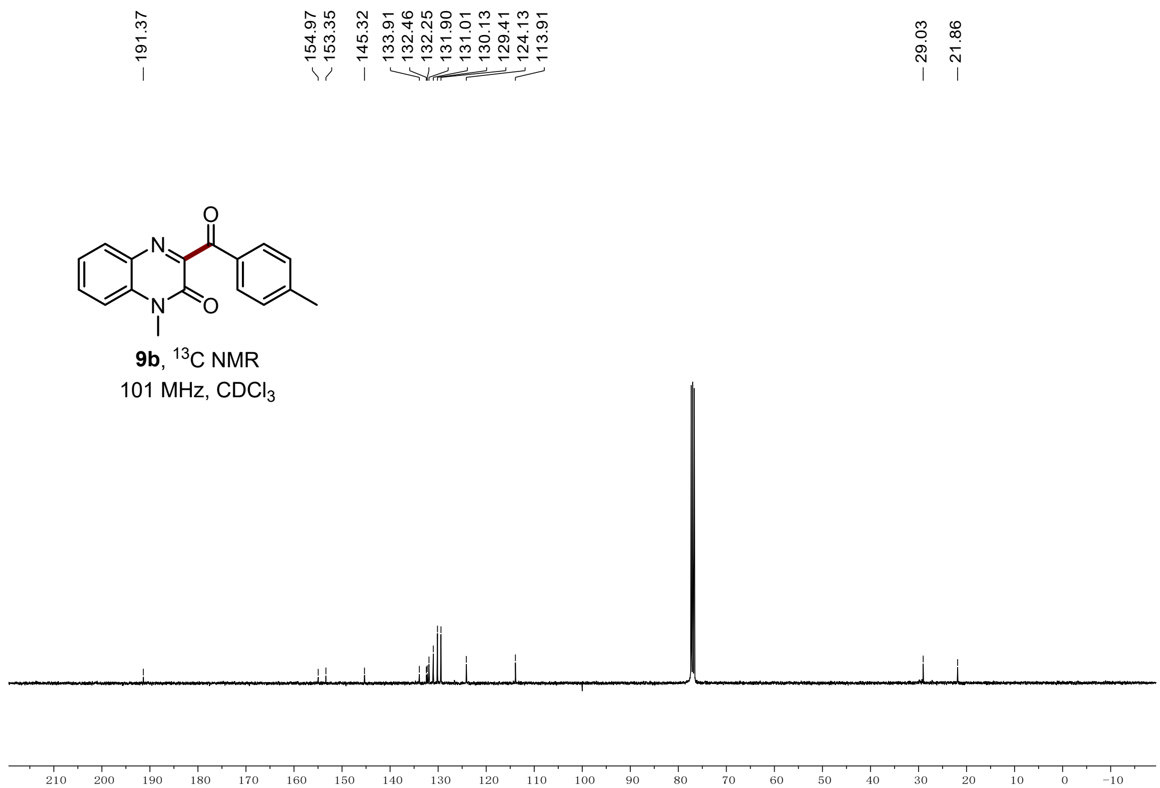
101 MHz, CDCl3
f1 (ppm)


7.94 7.94 7.92 7.92 7.89 7.89 7.87 7.87 7.70 7.69 7.68 7.67 7.67 7.66 7.65 7.43 7.43 7.42 7.41 7.41 7.39 7.29 7.27 7.26
3.75
2.43
O
N
N
O
9b, 1H NMR
400 MHz, CDCl3
1.02 2.01 1.03 2.04 2.00
3.00
3.08
f1 (ppm)


191.37
154.97 153.35
145.32
133.91 132.46 132.25 131.90 131.01 130.13 129.41 124.13 113.91
29.03
21.86
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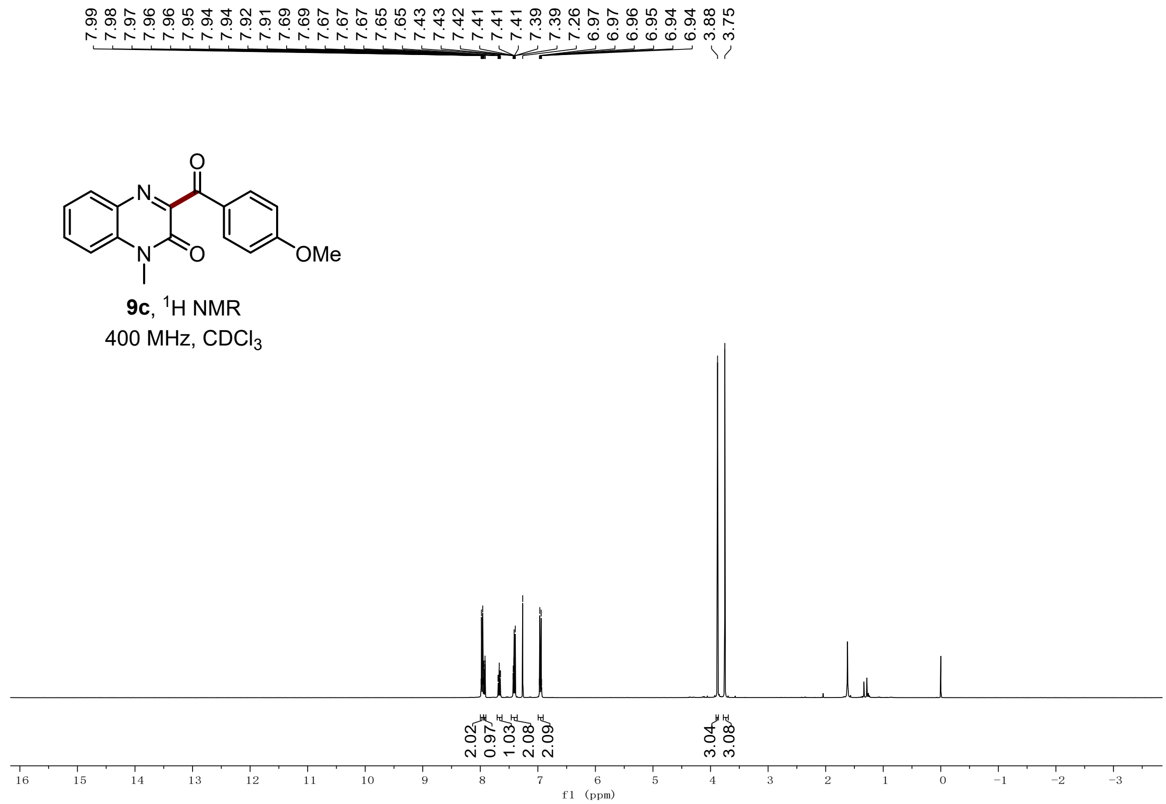
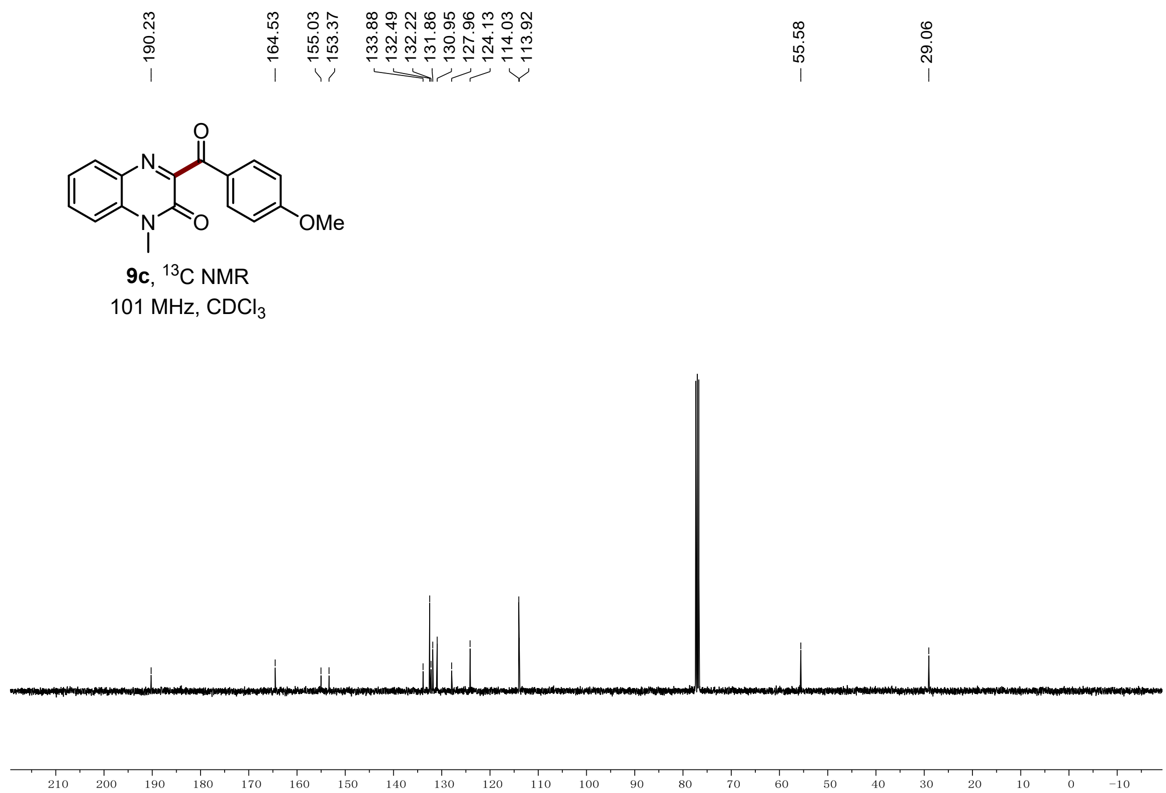
9b, 13C NMR
101 MHz, CDCl3
f1 (ppm)


7.99 7.98 7.97 7.96 7.96 7.95 7.94 7.94 7.92 7.91 7.69 7.69 7.67 7.67 7.65 7.65 7.43 7.42 7.41 7.41 7.41 7.39 7.39 7.26 6.97 6.97 6.96 6.95 6.94 6.94 3.88 3.75
N
O
O
N
OMe
9c, 1H NMR
400 MHz, CDCl3
2.02 0.97 1.03 2.08 2.09 3.04 3.08
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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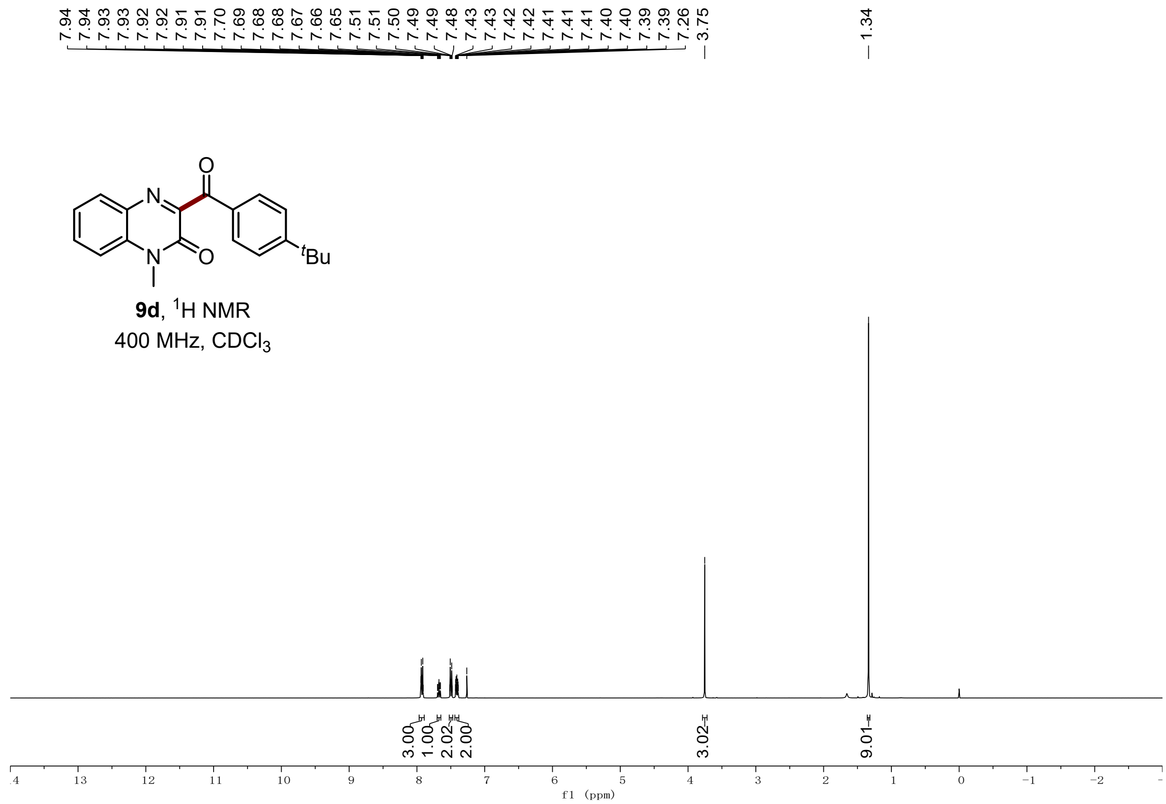
164.53 155.03 153.37
133.88 132.49 132.22 131.86 130.95 127.96 124.13 114.03 113.92
55.58
29.06
N
O
O
N
OMe
9c, 13C NMR
101 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



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7.93
7.93
7.92
7.92
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7.91
7.70
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7.68
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7.66
7.65
7.51
7.51
7.50
7.49
7.49
7.48
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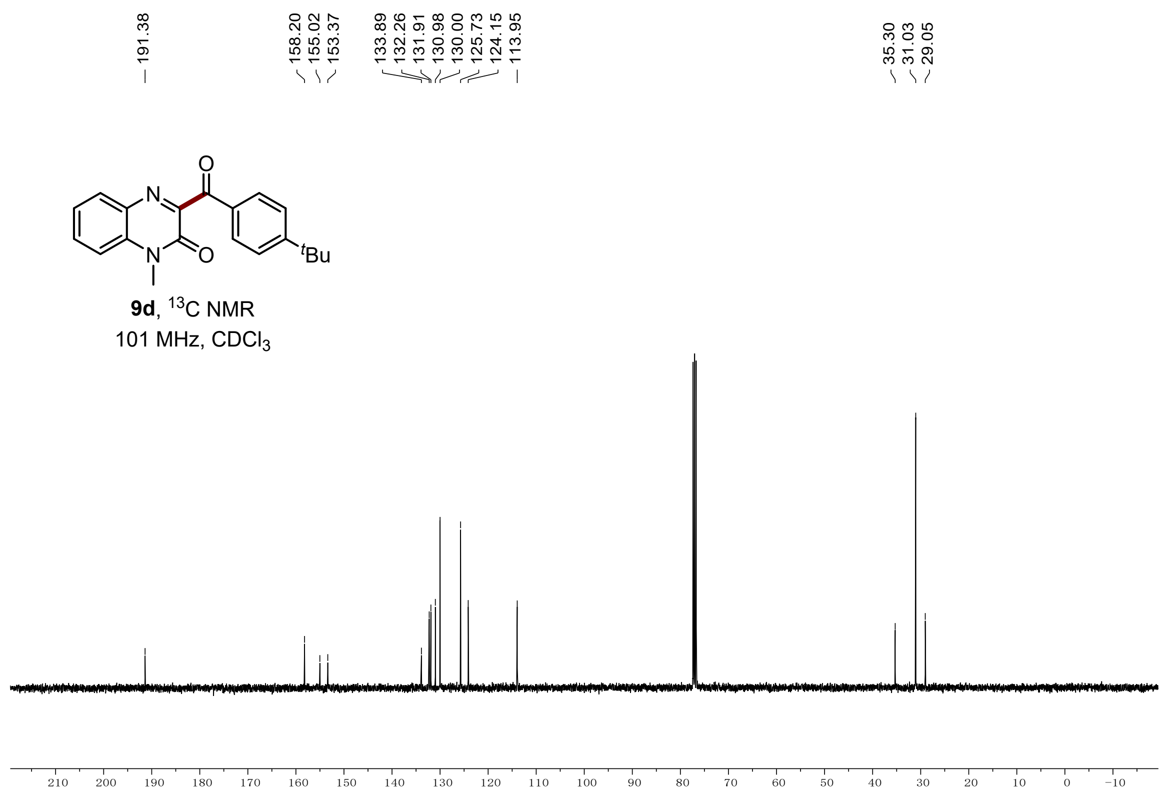
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.39
7.39
7.26
3.75
1.34
O
N
N
O
tBu
9d, 1H NMR
400 MHz, CDCl3
3.00
1.00
2.02
2.00
3.02
9.01
f1 (ppm)



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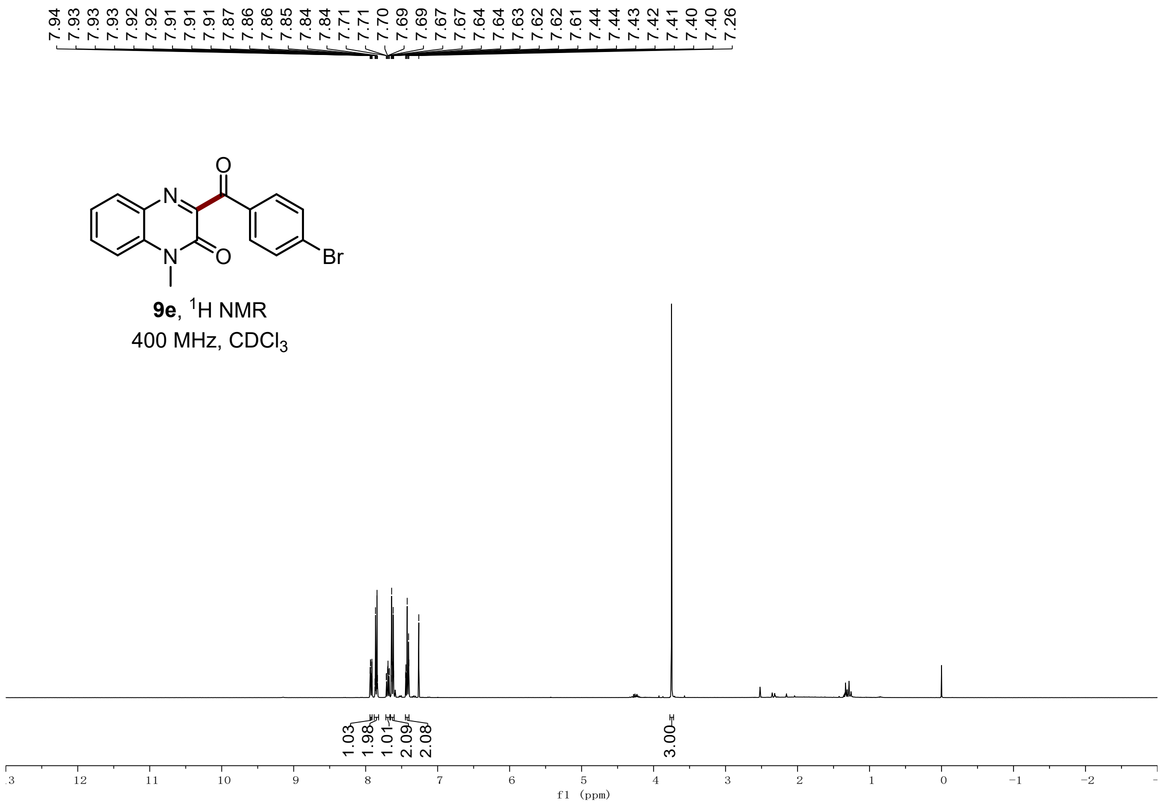
31.03
29.05
O
N
N
O
tBu
9d, 13C NMR
101 MHz, CDCl3
f1 (ppm)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7.71
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7.69
7.67
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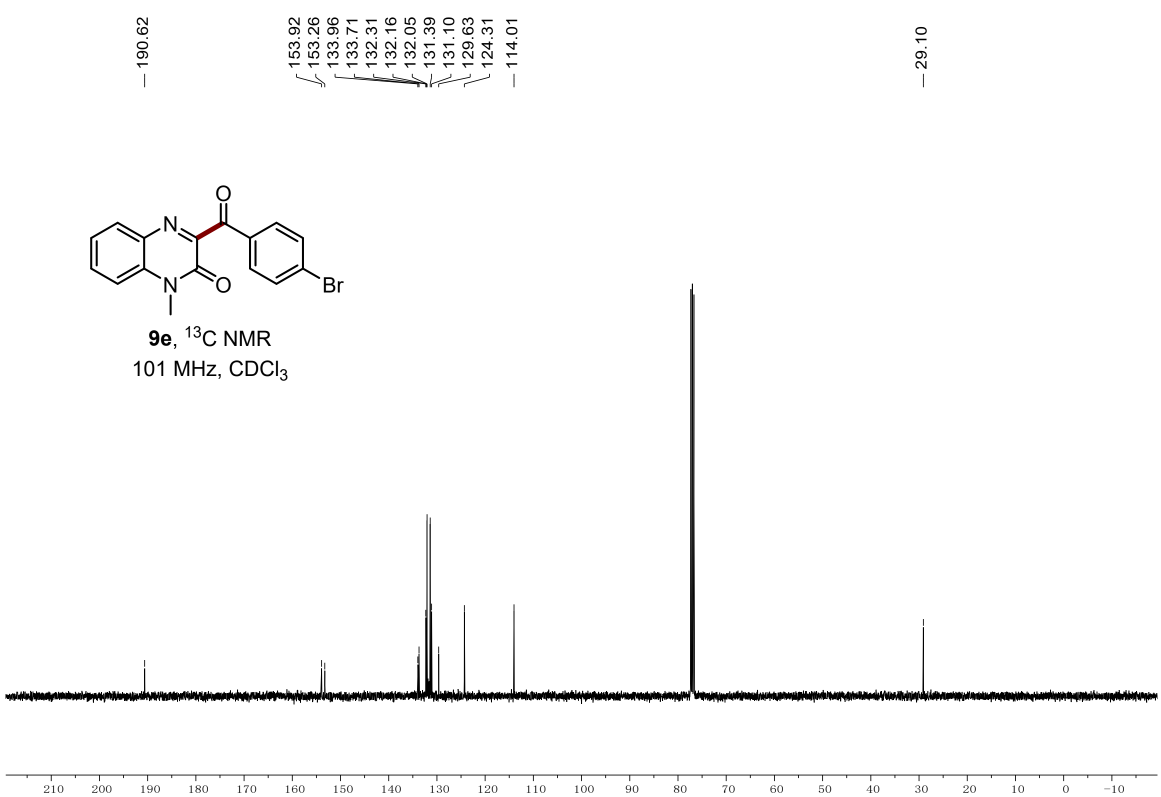
7.64
7.63
7.62
7.62
7.61
7.44
7.44
7.43
7.42
7.41
7.40
7.40
7.26
N
O
O
N
Br
9e, 1H NMR
400 MHz, CDCl3
1.03
1.98
1.01
2.09
2.08
3.00
f1 (ppm)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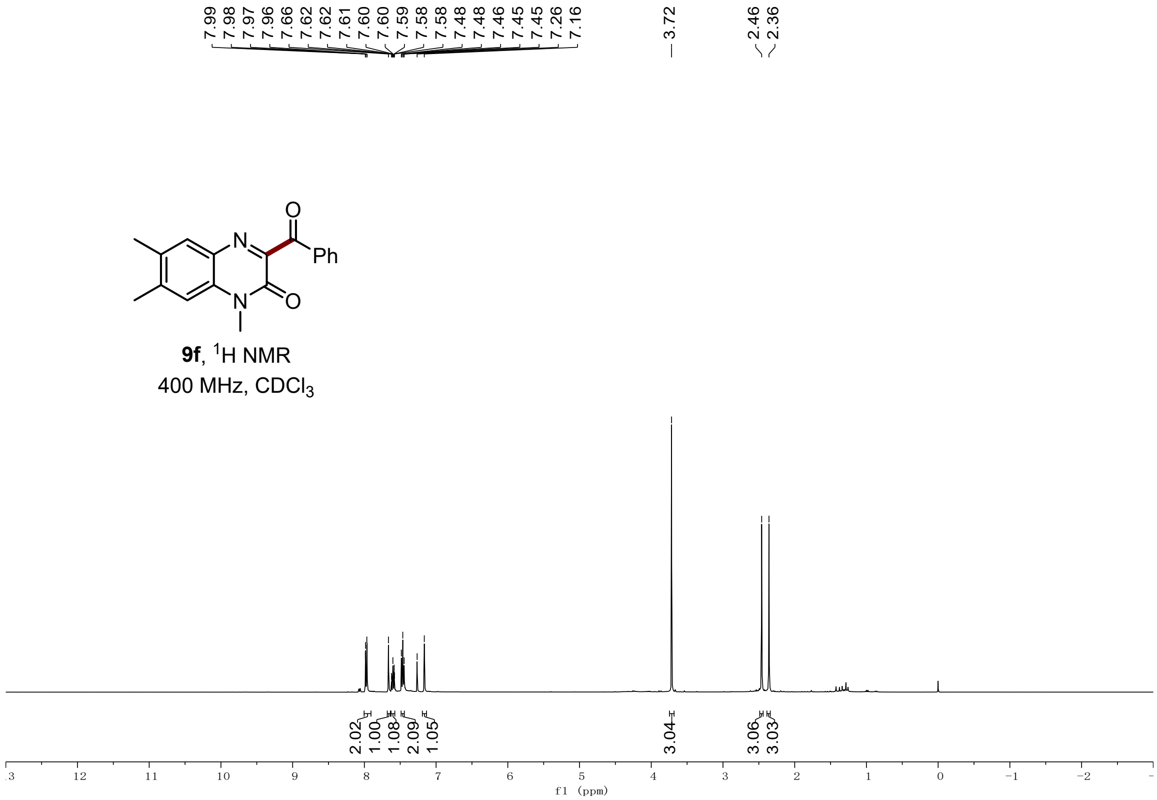
29.10
N
O
O
N
Br
9e, 13C NMR
101 MHz, CDCl3
f1 (ppm)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7.48
7.48
7.46
7.45
7.45
7.26
7.16
3.72
2.46
2.36
N
O
Ph
N
O
9f, 1H NMR
400 MHz, CDCl3
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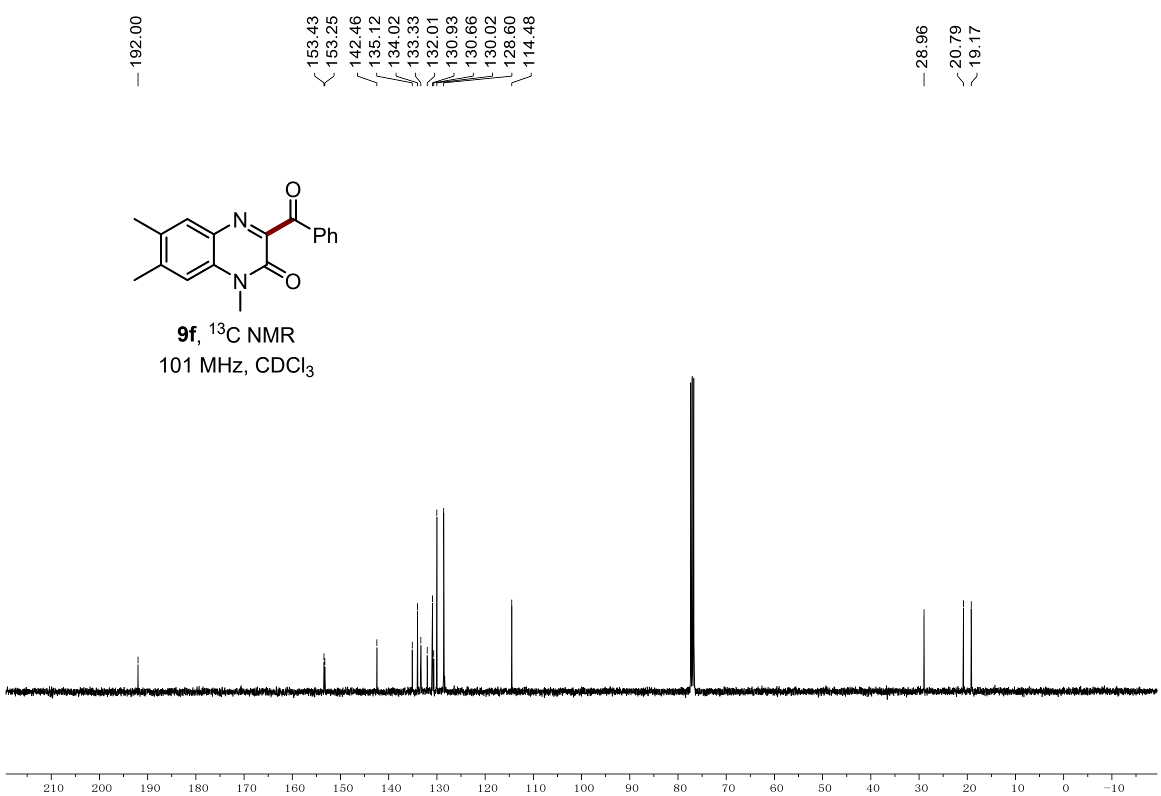
2.09
1.05
3.04
3.06
3.03
f1 (ppm)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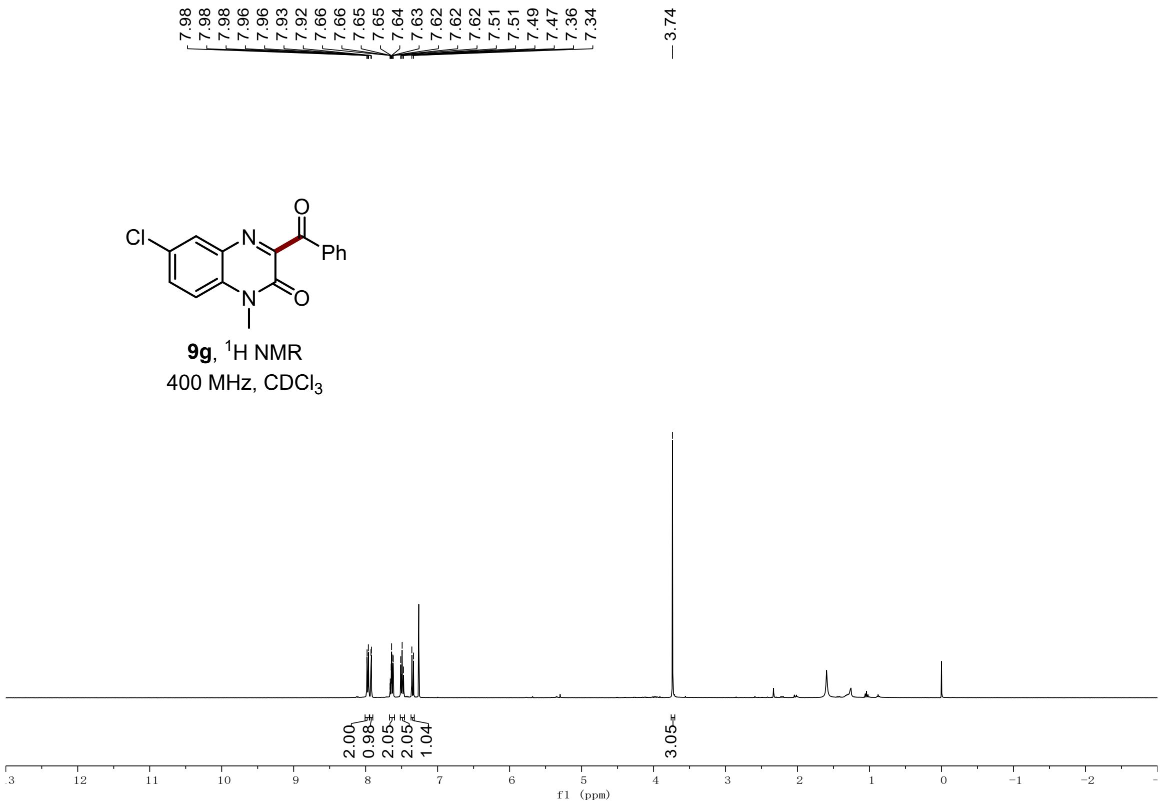
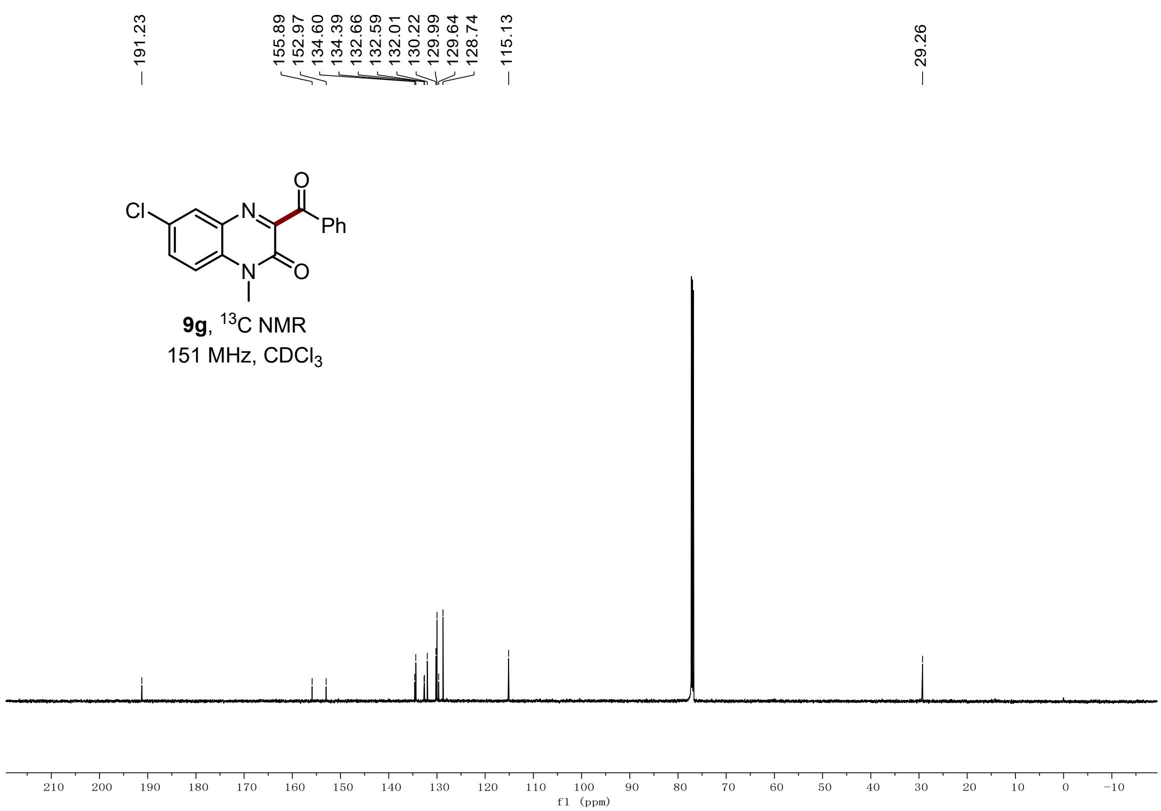
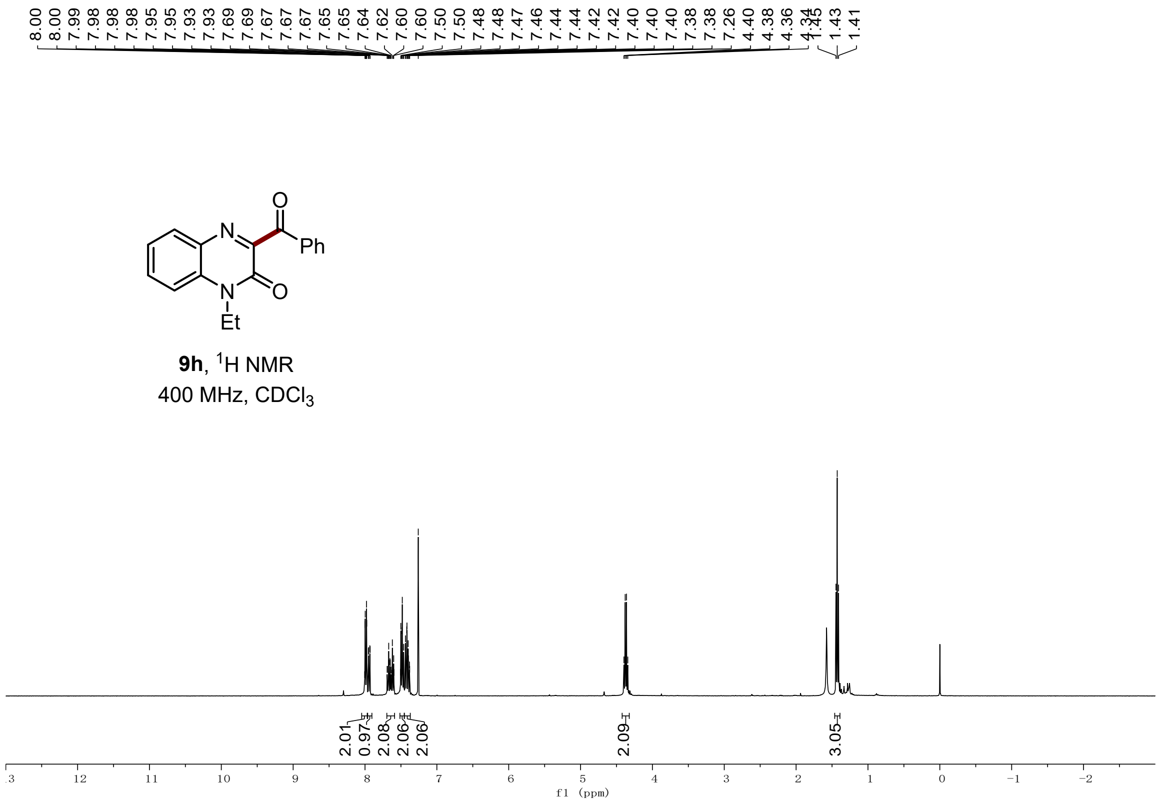
9f, 13C NMR
101 MHz, CDCl3
f1 (ppm)



9g, ^{1}H NMR
400 MHz, $CDCl_3$



9g, ^{13}C NMR
151 MHz, $CDCl_3$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7.40
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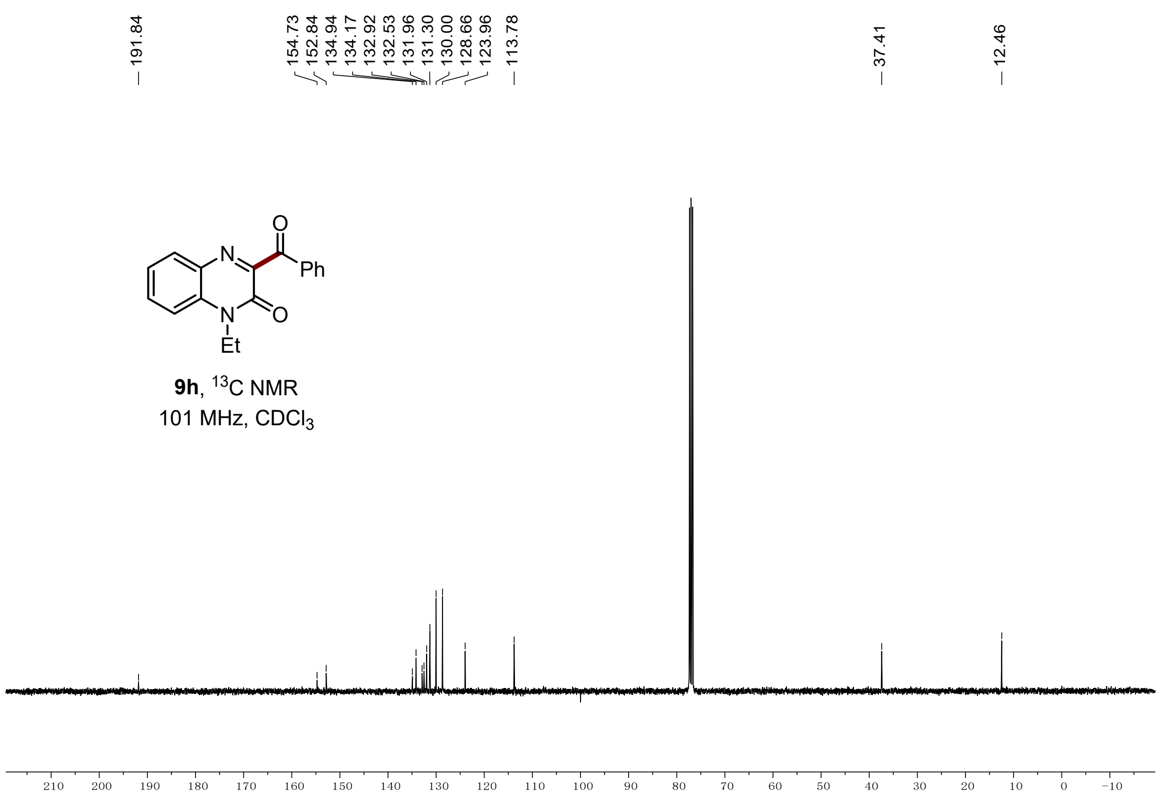
7.26
4.40
4.38
4.36
4.34
1.45
1.43
1.41
N
O
Ph
N
O
Et
9h, ^{1}H NMR
400 MHz, $CDCl_3$
2.01
0.97
2.08
2.06
2.06
2.09
3.05
f1 (ppm)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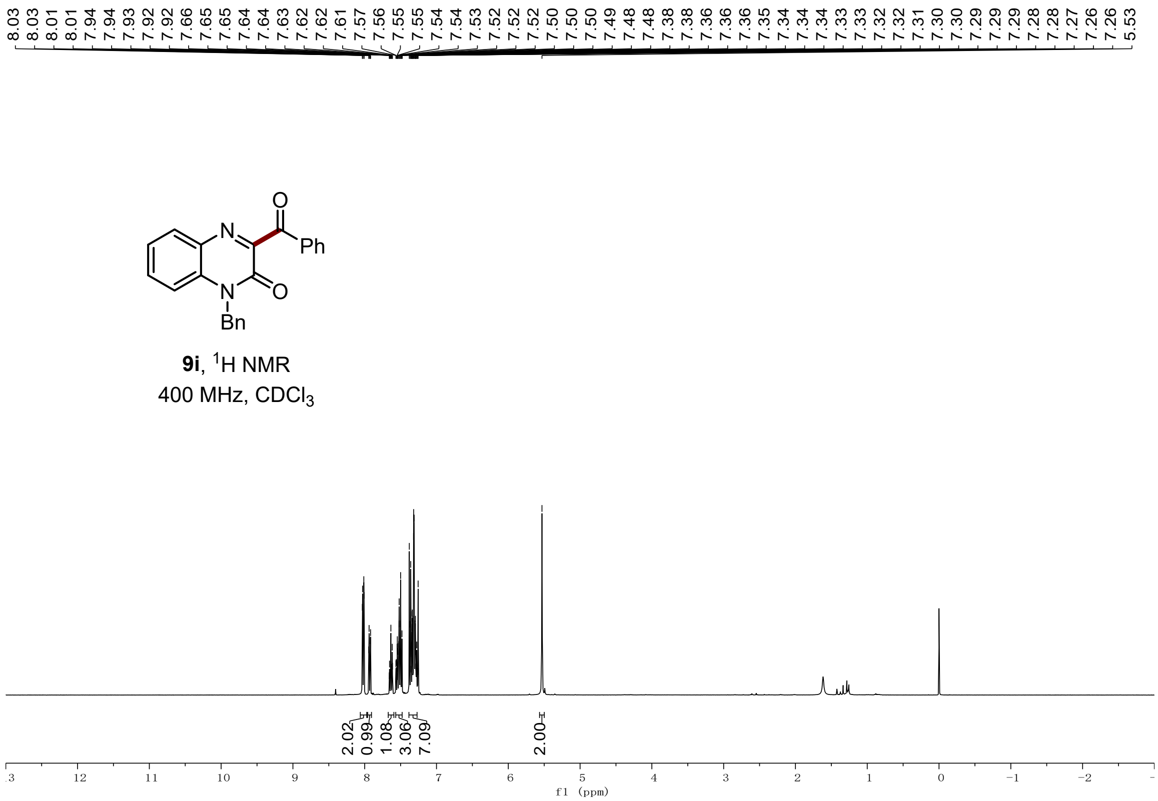
113.78
37.41
12.46
N
O
Ph
N
O
Et
9h, ^{13}C NMR
101 MHz, $CDCl_3$
f1 (ppm)



9i, ^{1}H NMR
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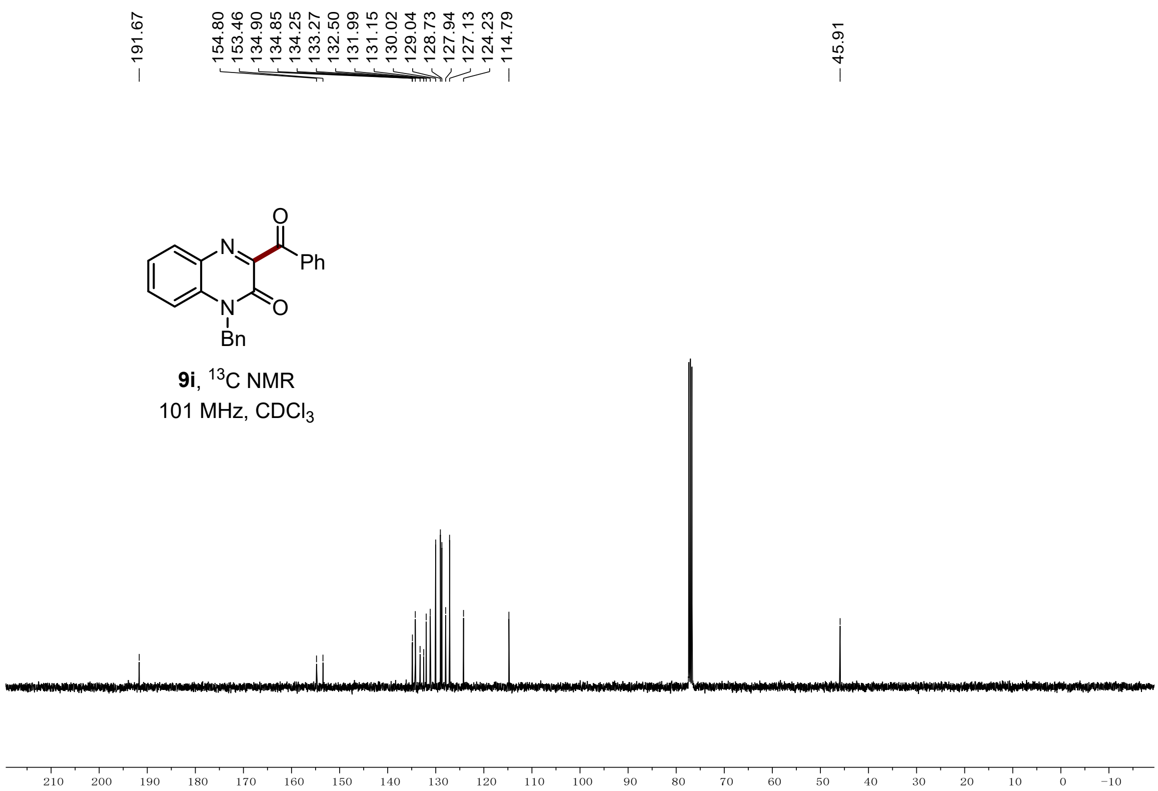
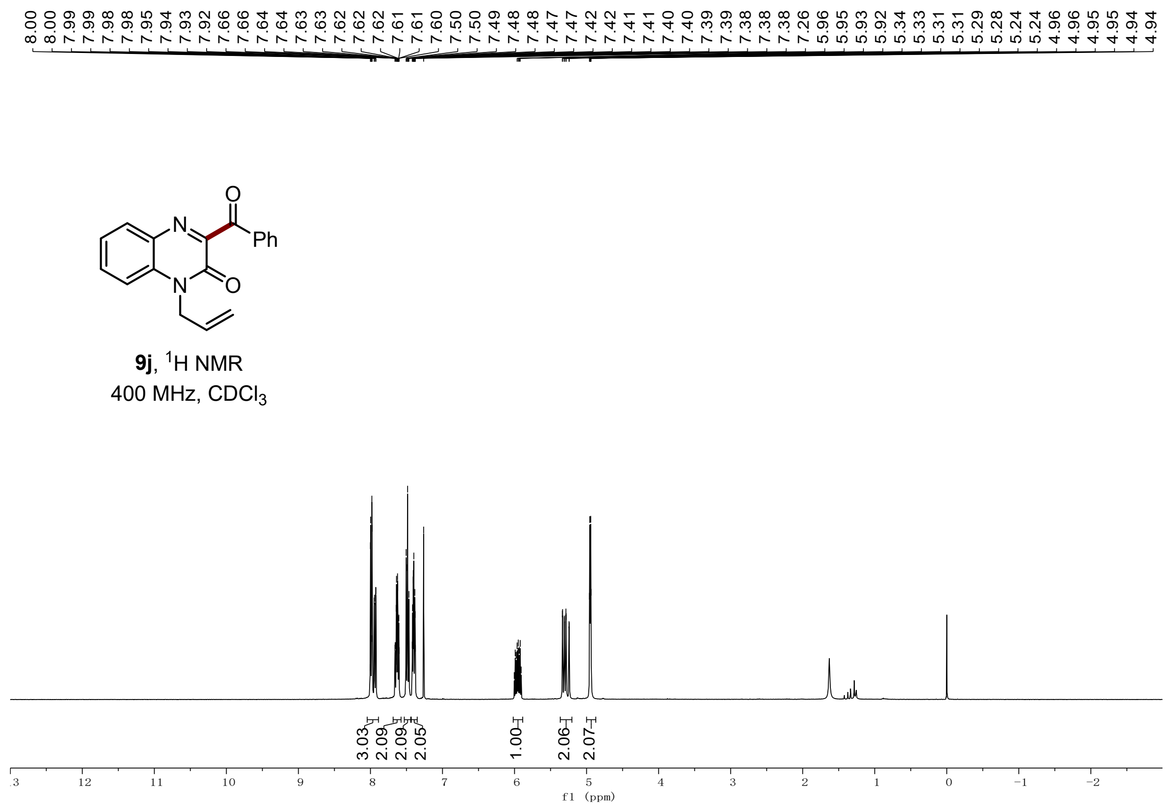
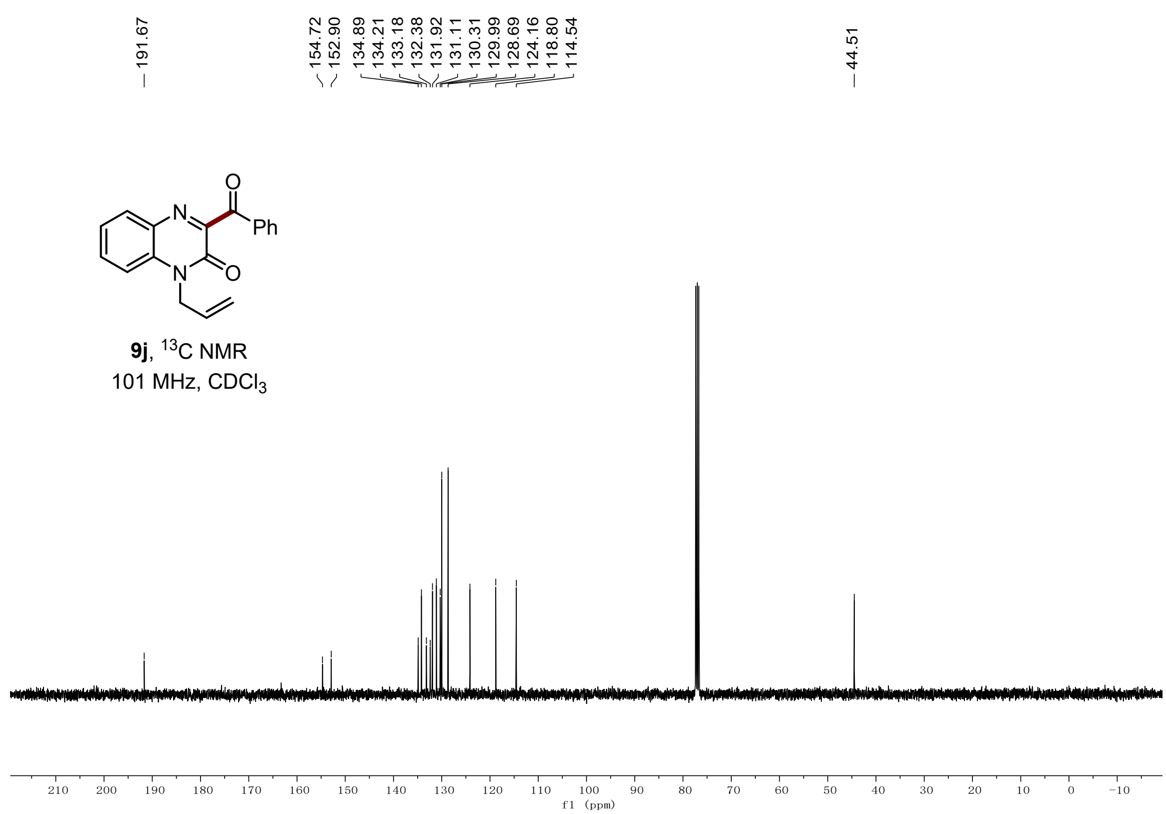


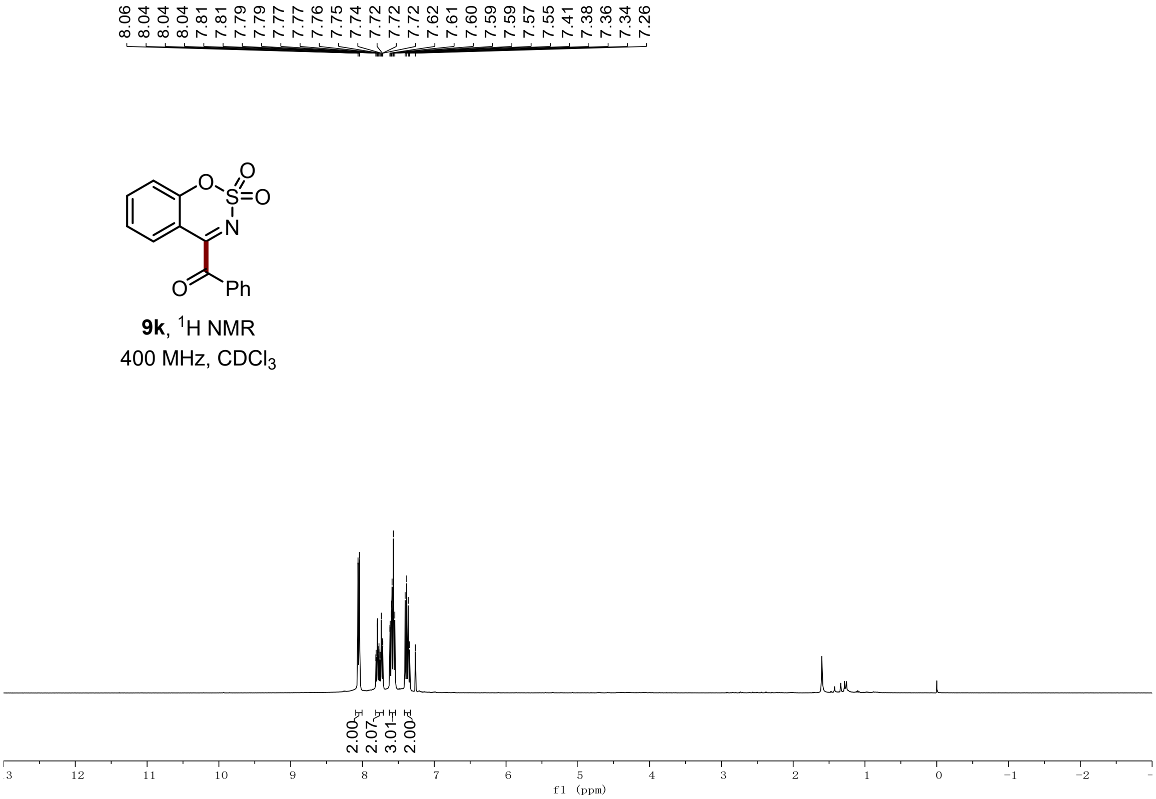
9i, ^{13}C NMR
101 MHz, $CDCl_3$



9j, ^{1}H NMR
400 MHz, $CDCl_3$



9j, ^{13}C NMR
101 MHz, $CDCl_3$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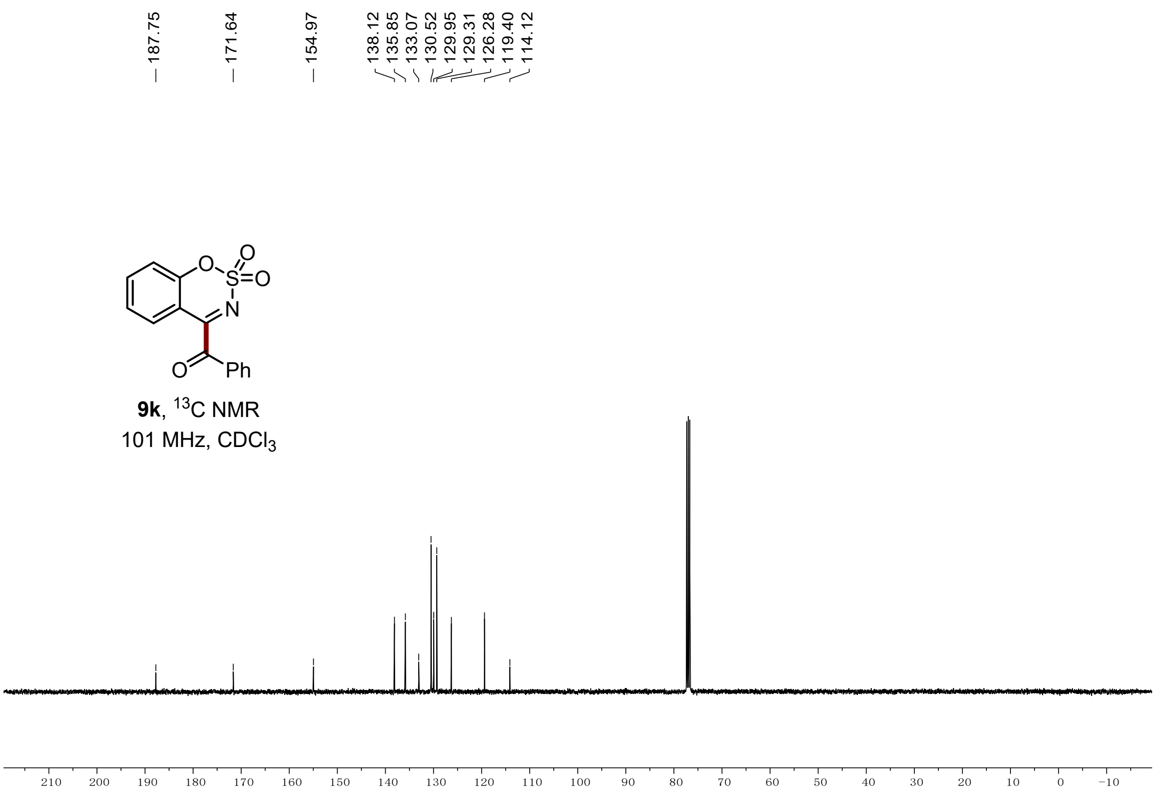
O
S
O
O
N
O
Ph
9k, ^{1}H NMR
400 MHz, $CDCl_3$
2.00
2.07
3.01
2.00
f1 (ppm)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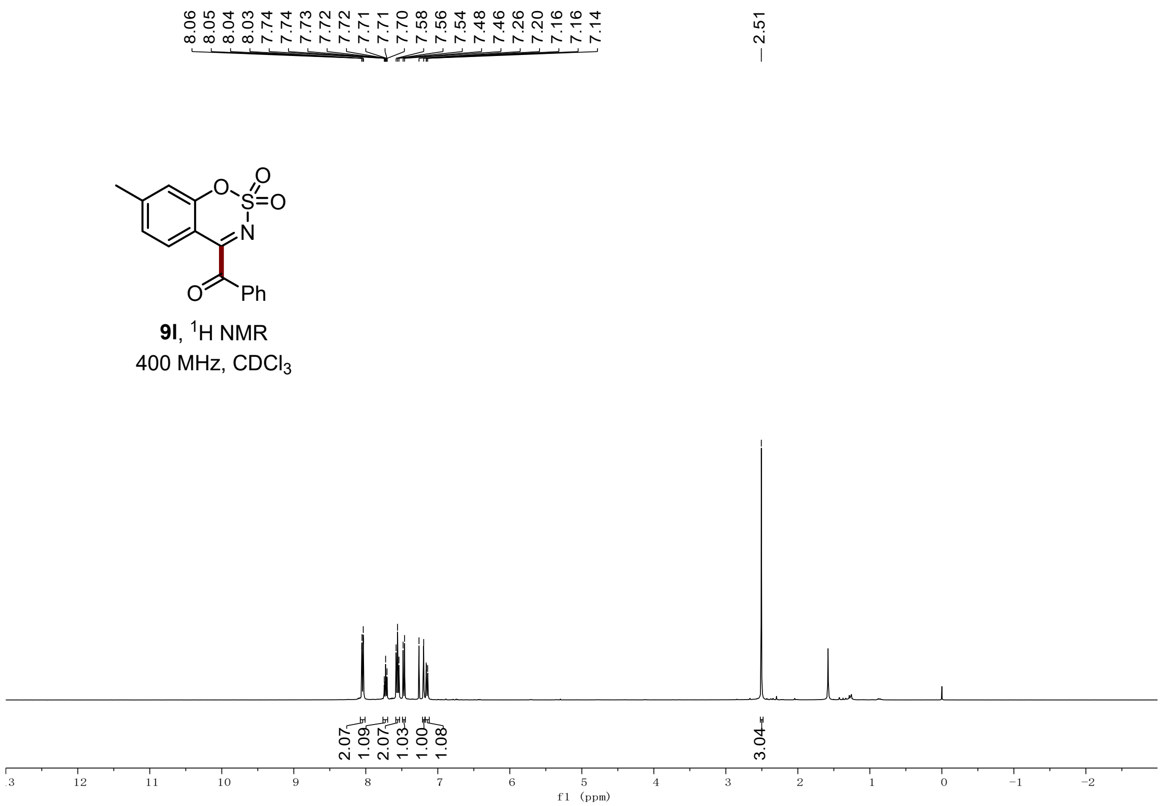
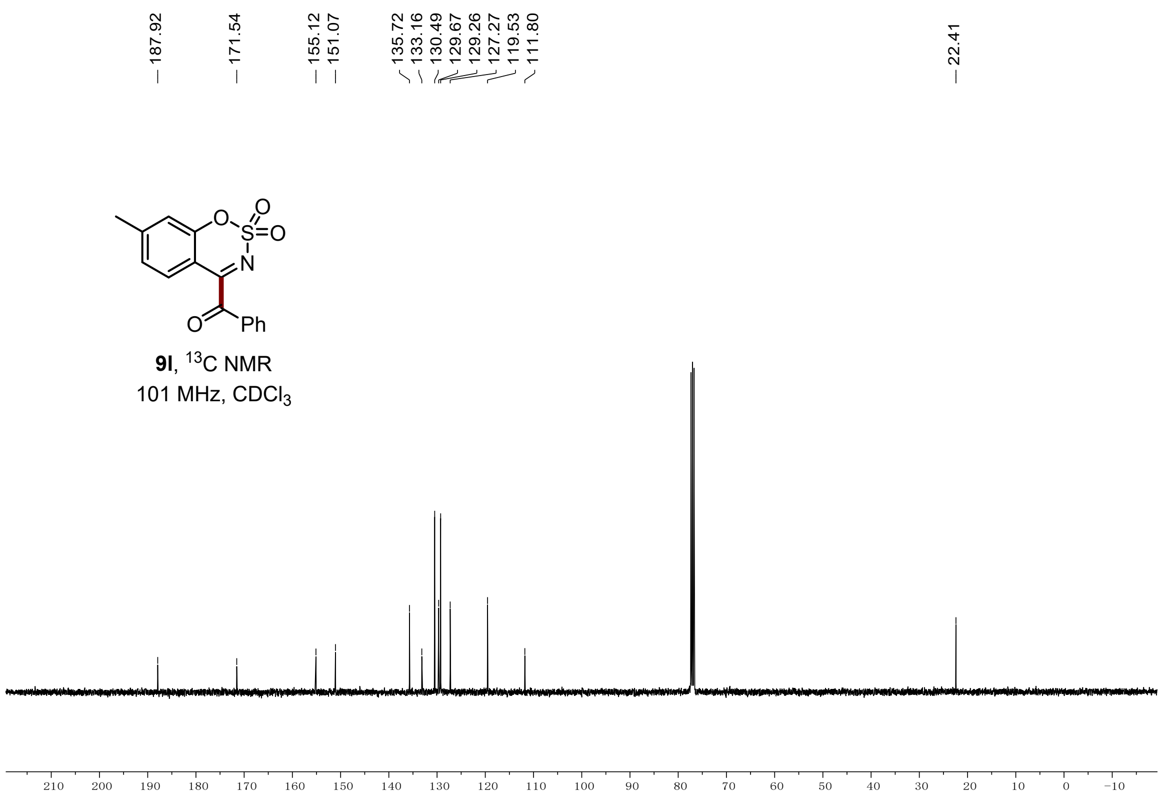
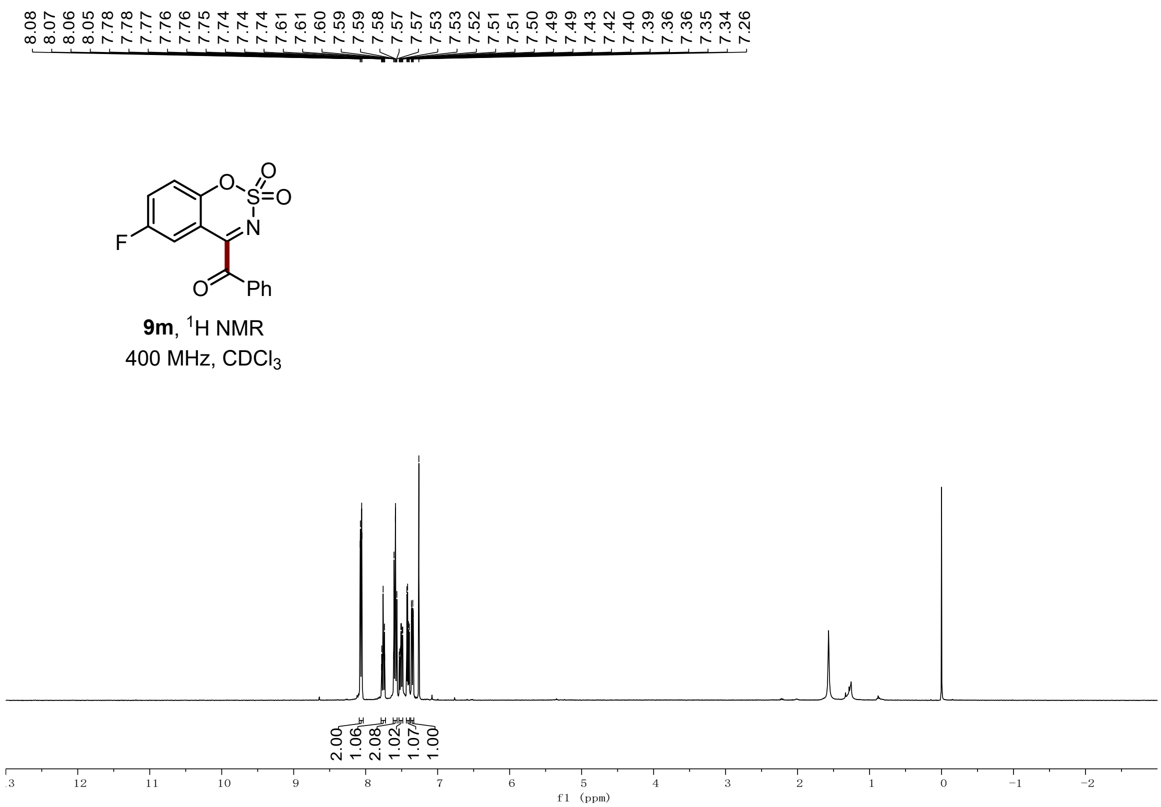
171.64
154.97
138.12 135.85 133.07 130.52 129.95 129.31 126.28 119.40 114.12
O
S
O
O
N
O
Ph
9k, ^{13}C NMR
101 MHz, $CDCl_3$
f1 (ppm)



9l, ^{1}H NMR
400 MHz, $CDCl_3$



9l, ^{13}C NMR
101 MHz, $CDCl_3$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8.06
8.05
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7.76
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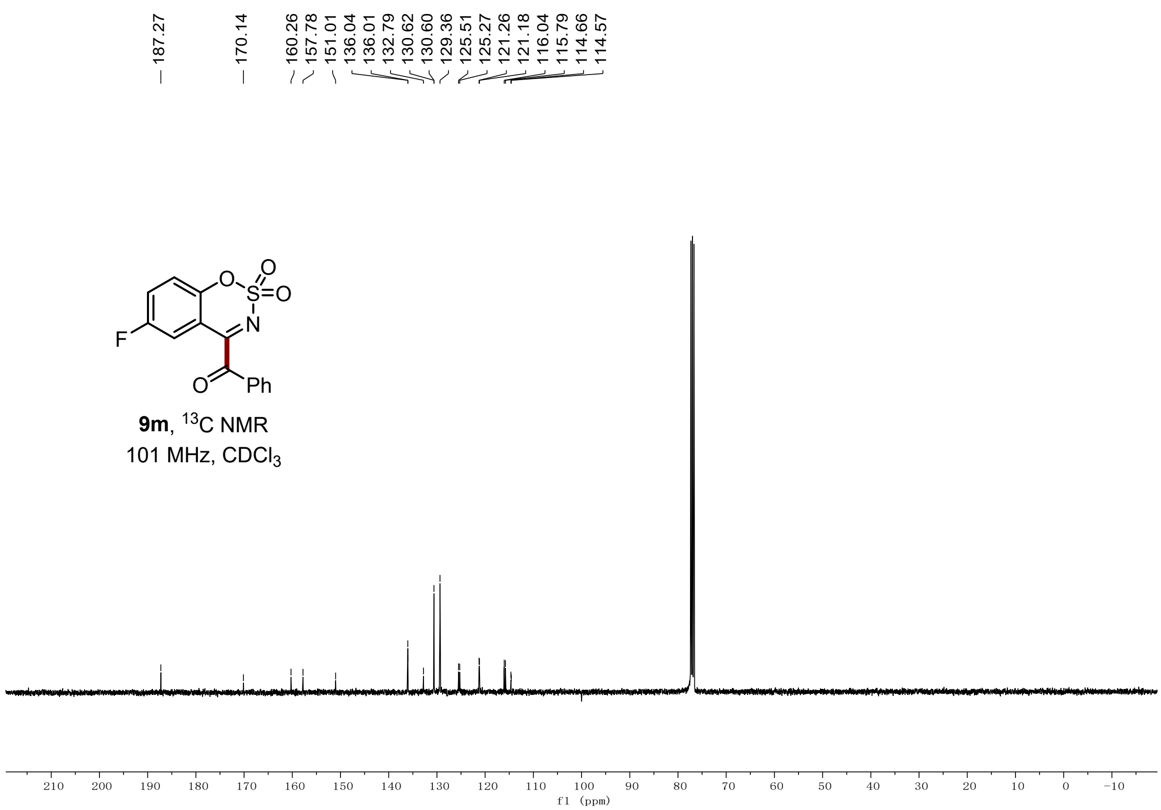
7.74
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.49
7.43
7.42
7.40
7.39
7.36
7.36
7.35
7.34
7.26
O
S
O
O
N
F
O
Ph
9m, 1H NMR
400 MHz, CDCl3
2.00
1.06
2.08
1.02
1.07
1.00
f1 (ppm)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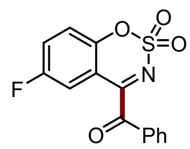
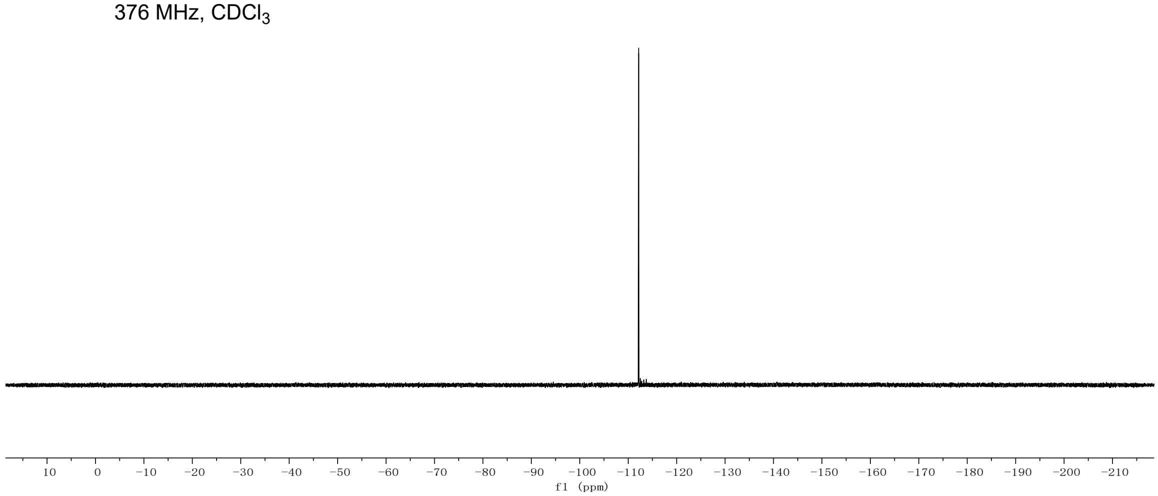
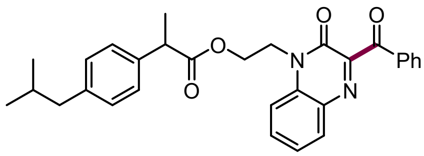
151.01
136.04
136.01
132.79
130.62
130.60
129.36
125.51
125.27
121.26
121.18
116.04
115.79
114.66
114.57
O
S
O
O
N
F
O
Ph
9m, 13C NMR
101 MHz, CDCl3
f1 (ppm)

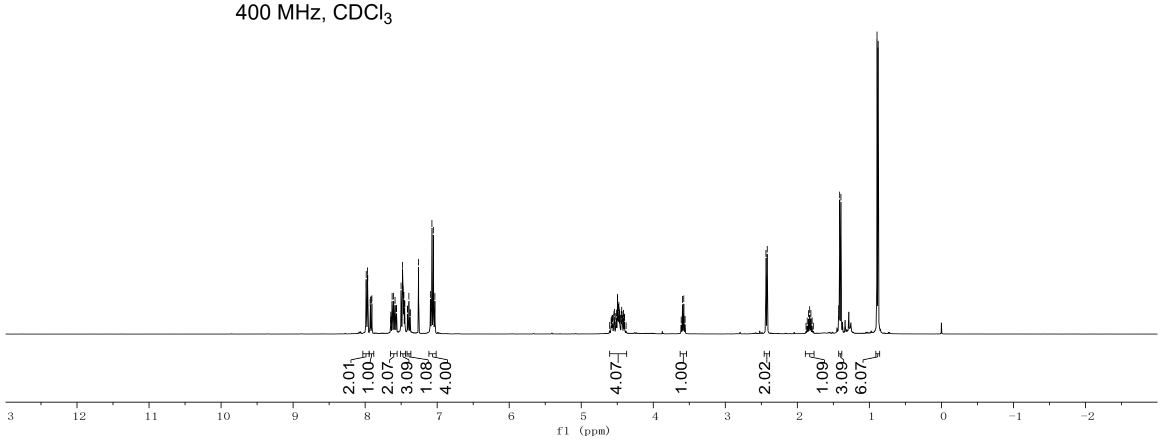


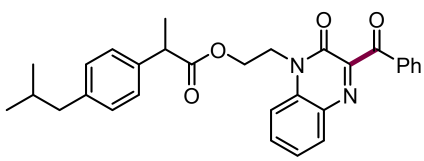
9m, ^{19}F NMR
376 MHz, $CDCl_3$



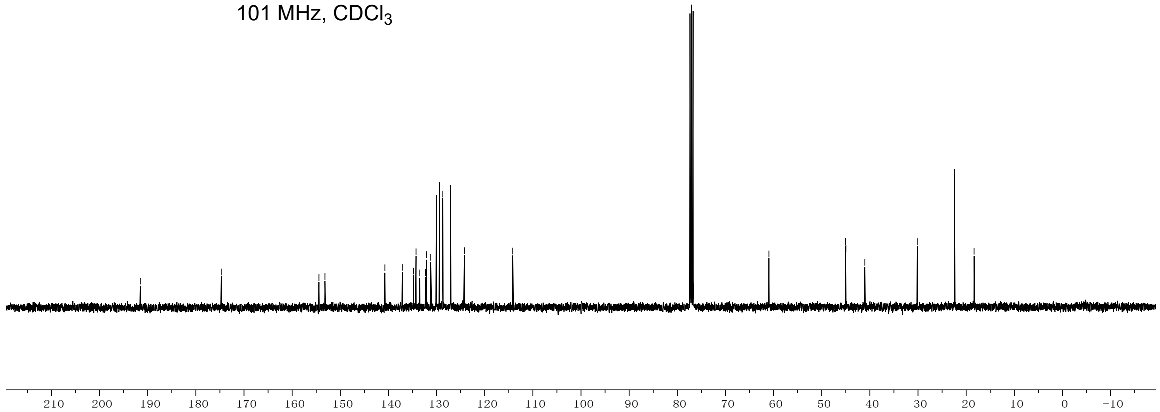


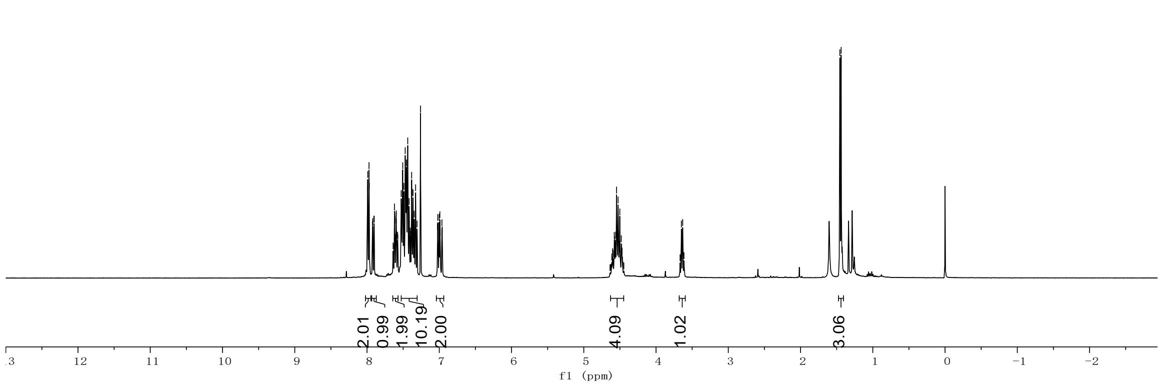
9n, ^{1}H NMR
400 MHz, $CDCl_3$

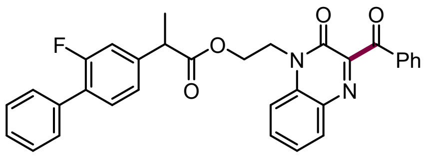




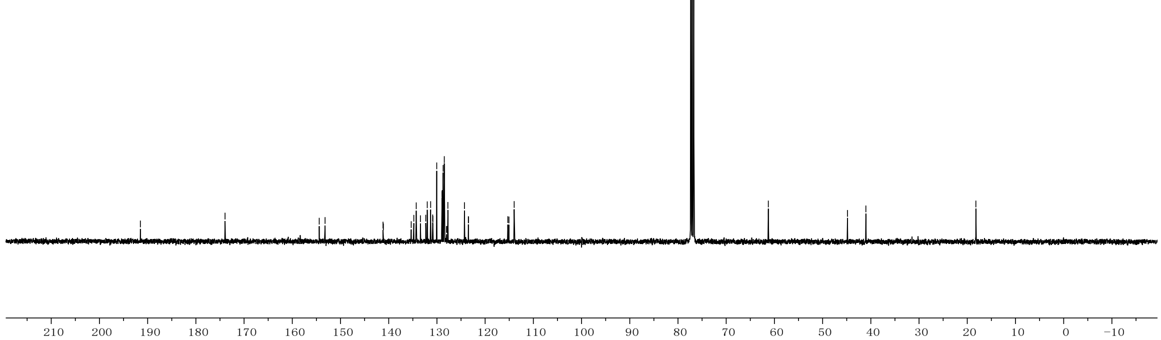
9n, ^{13}C NMR
101 MHz, $CDCl_3$

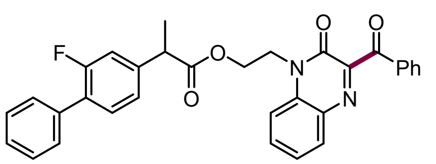




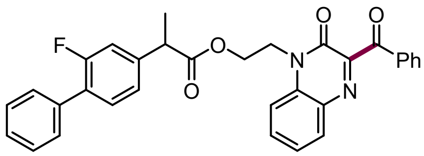


9o, ^{1}H NMR
400 MHz, $CDCl_3$

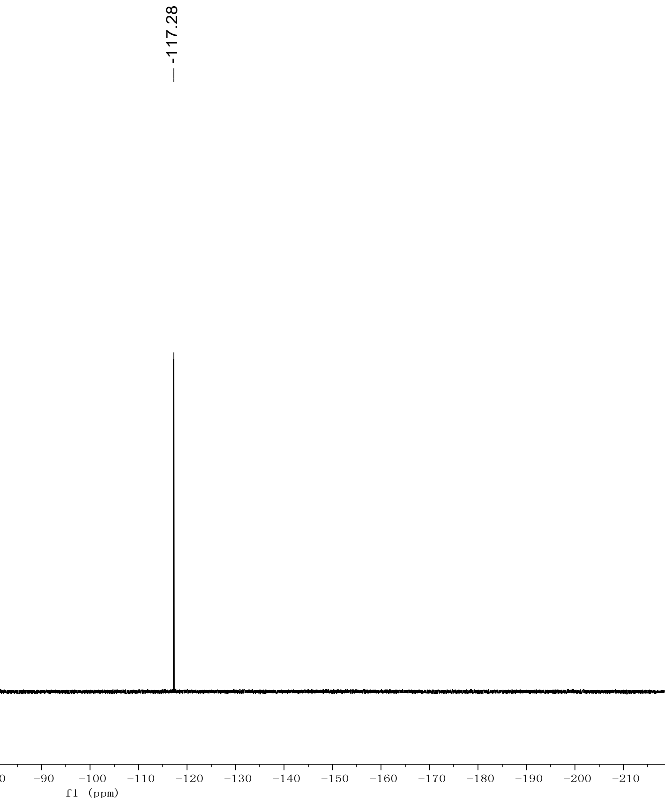


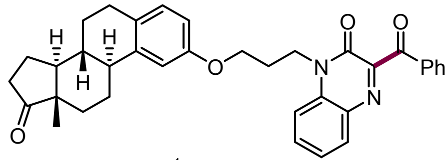


9o, ^{13}C NMR
101 MHz, $CDCl_3$

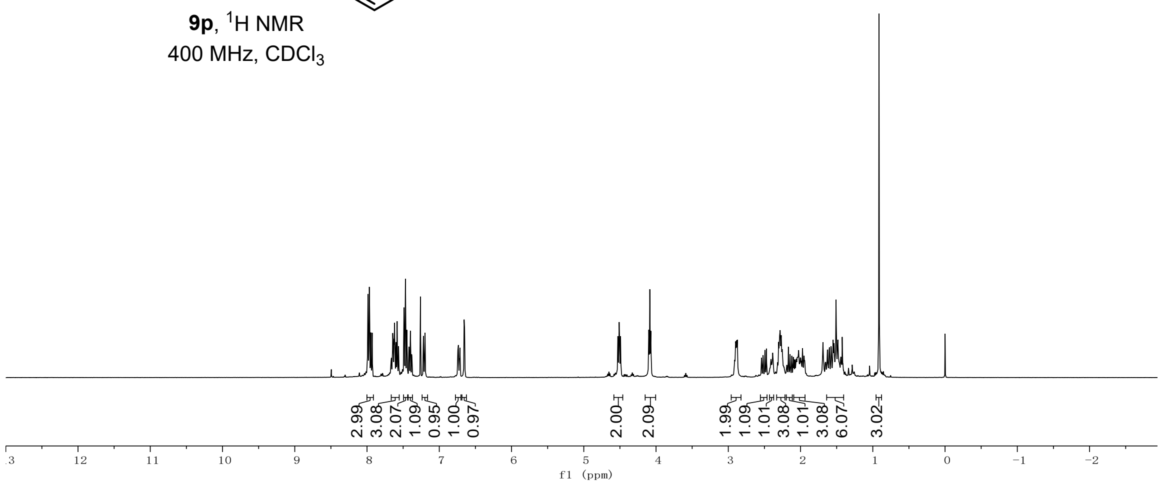


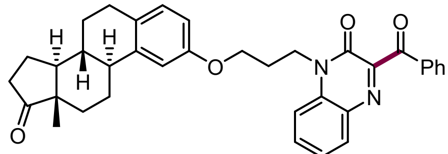
9o, ^{19}F NMR
376 MHz, $CDCl_3$



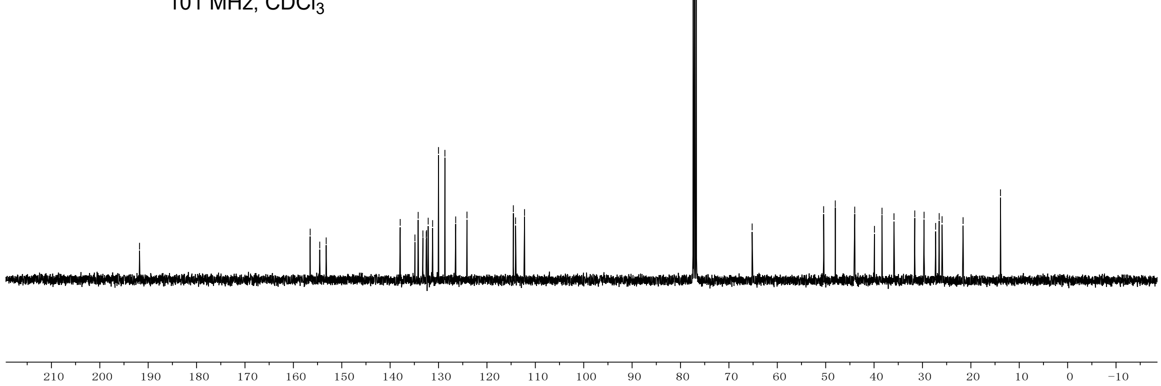


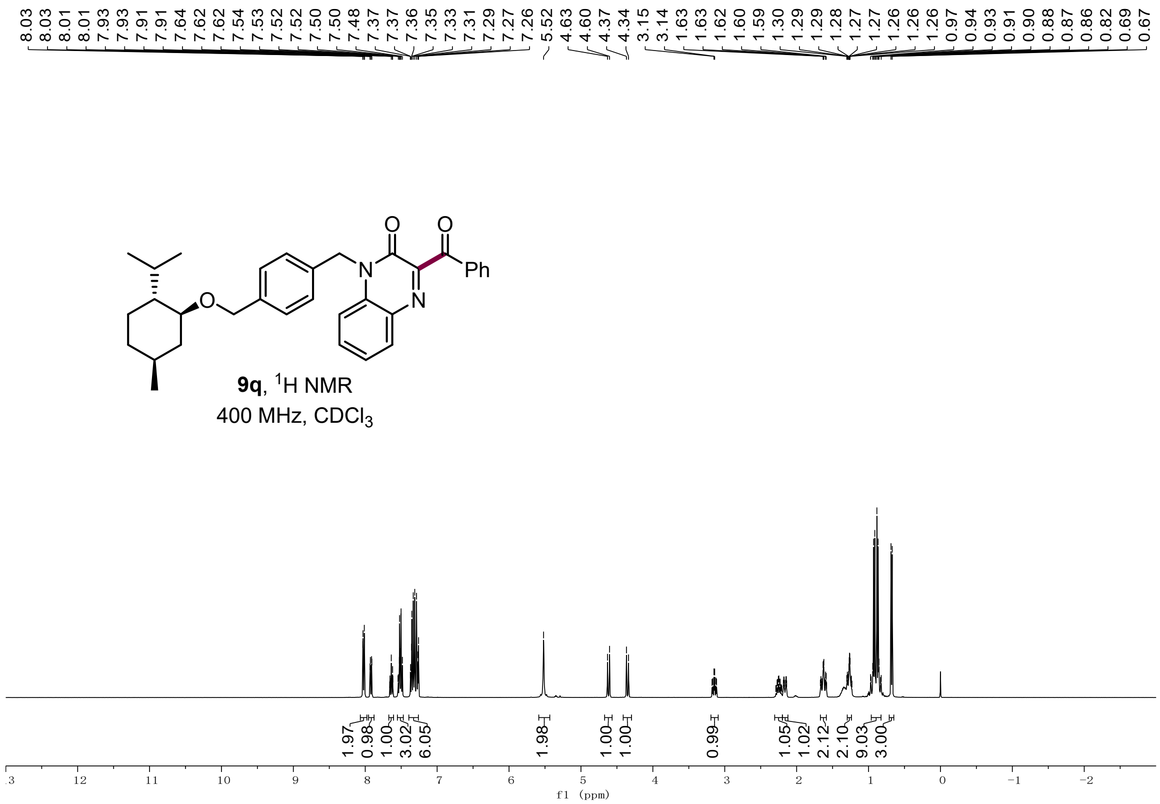
9p, ^{1}H NMR
400 MHz, $CDCl_3$





9p, ^{13}C NMR
101 MHz, $CDCl_3$

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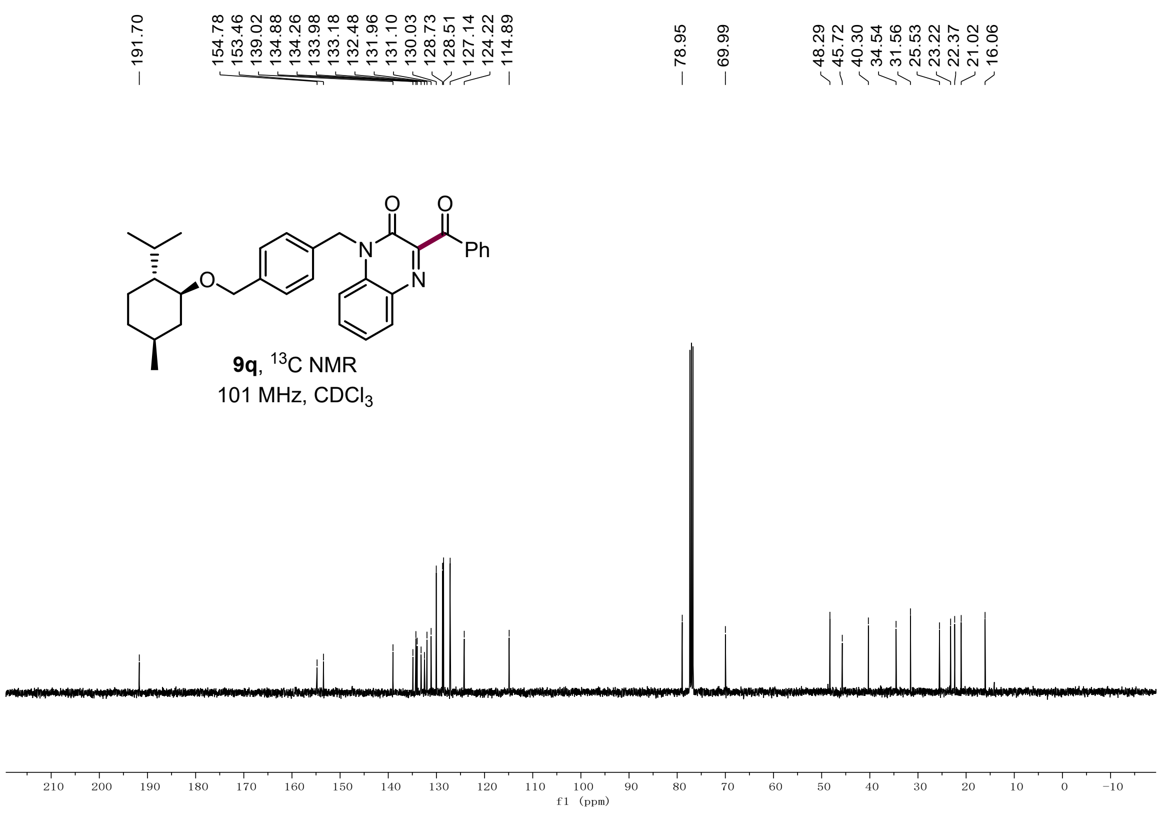
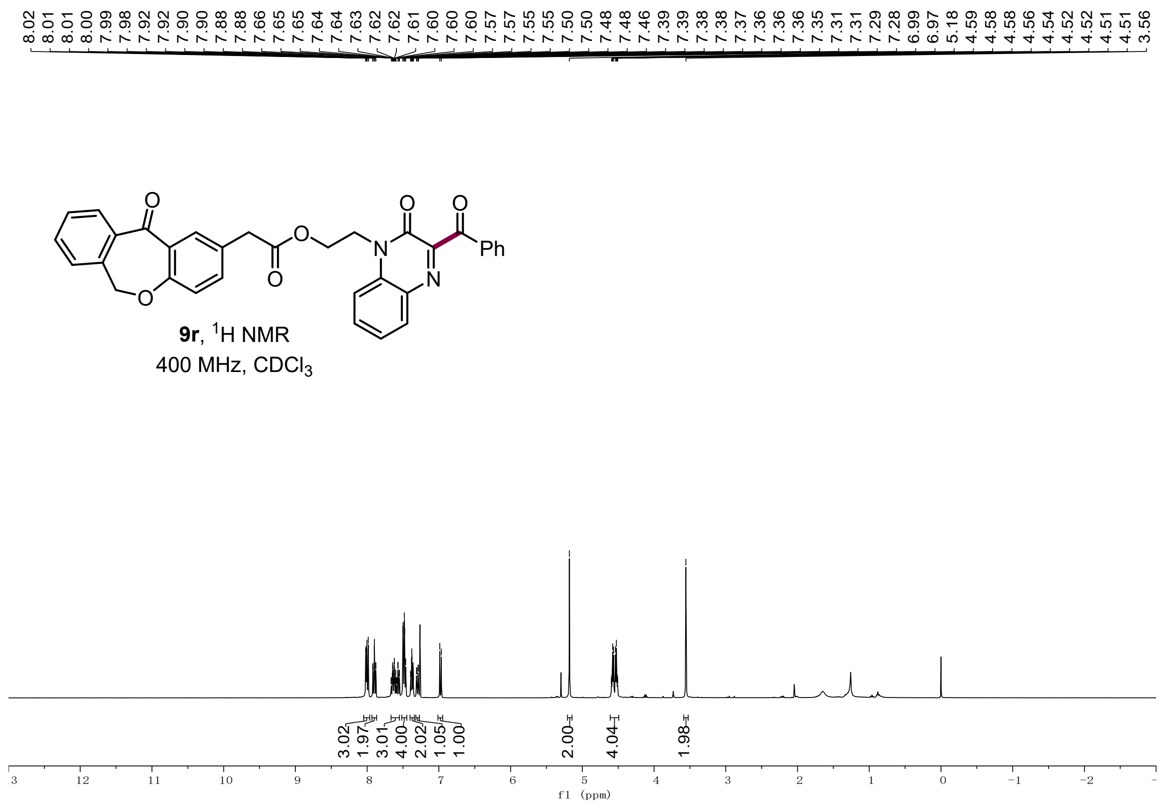
Ph
O
N
9q, ¹H NMR
400 MHz, CDCl3
1.97 0.98 1.00 3.02 6.05 1.98 1.00 1.00 0.99 1.05 1.02 2.12 2.10 9.03 3.00
f1 (ppm)


191.70
154.78 153.46 139.02 134.88 134.26 133.98 133.18 132.48 131.96 131.10 130.03 128.73 128.51 127.14 124.22 114.89
78.95 69.99
48.29 45.72 40.30 34.54 31.56 25.53 23.22 22.37 21.02 16.06
Ph
O
N
9q, ¹³C NMR
101 MHz, CDCl3
f1 (ppm)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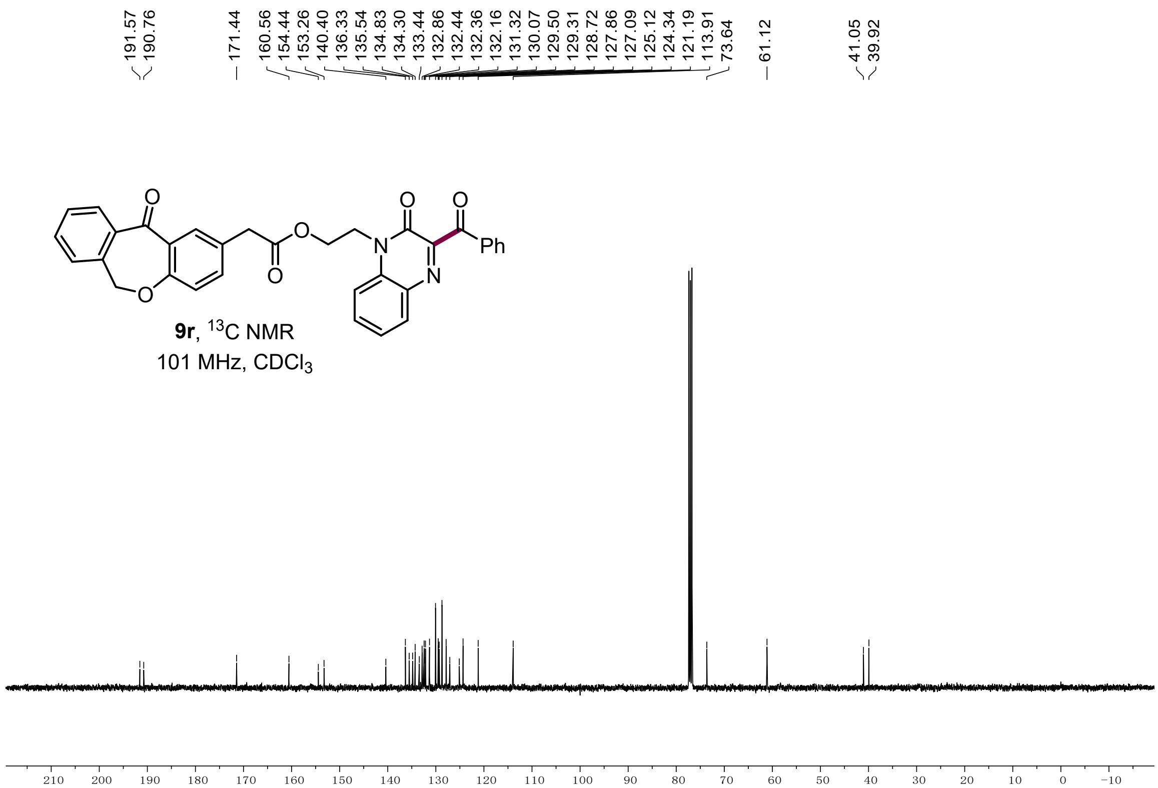
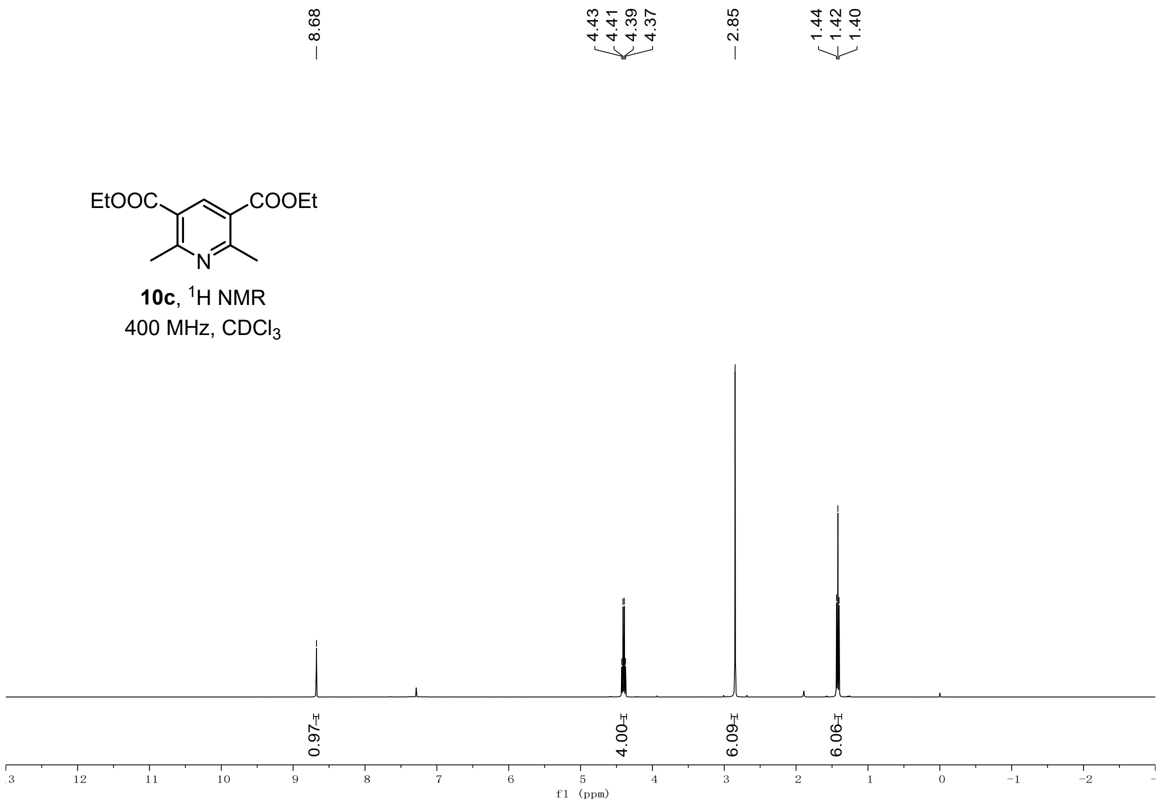
O
O
O
O
O
N
O
N
Ph
9r, 1H NMR
400 MHz, CDCl3
3.02 1.97 3.01 4.00 2.02 1.05 1.00
2.00
4.04
1.98
f1 (ppm)


191.57 190.76
171.44
160.56 154.44 153.26 140.40 136.33 135.54 134.83 134.30 133.44 132.86 132.44 132.36 132.16 131.32 130.07 129.50 129.31 128.72 127.86 127.09 125.12 124.34 121.19 113.91 73.64
61.12
41.05 39.92
9r, 13C NMR
101 MHz, CDCl3
f1 (ppm)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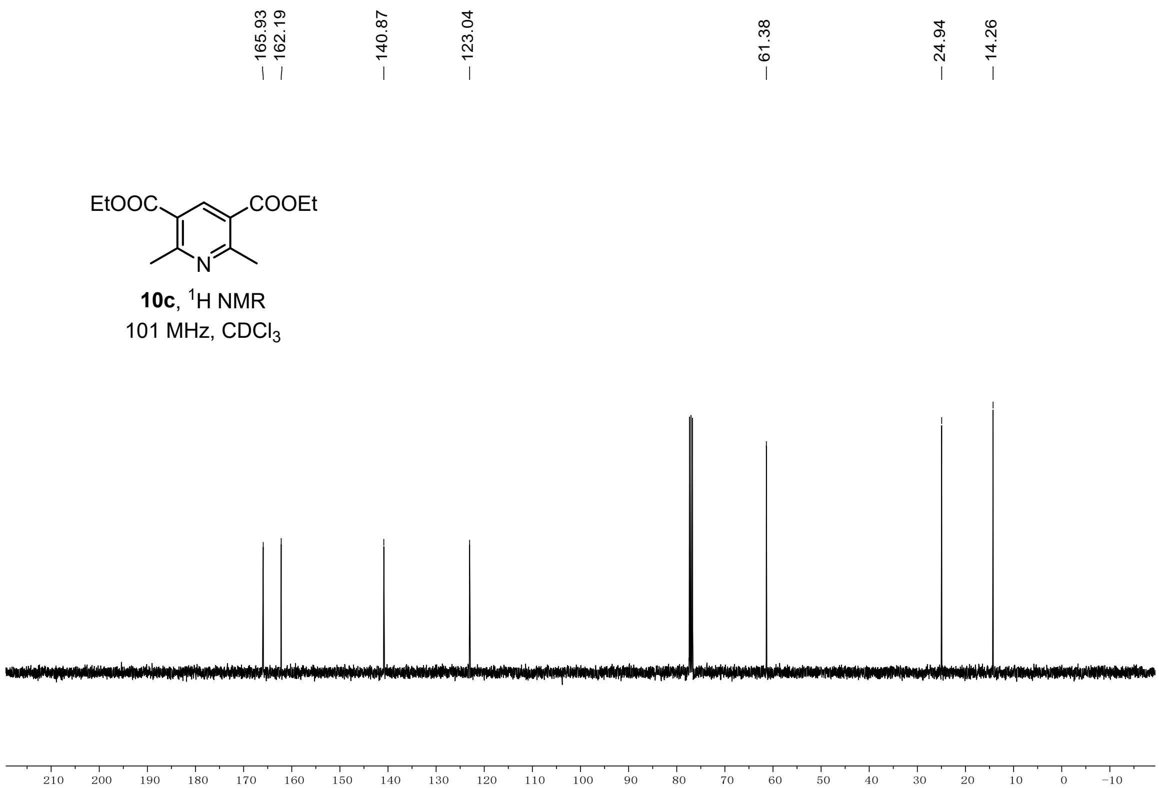
2.85
1.44
1.42
1.40
EtOOC
COOEt
N
10c, ¹H NMR
400 MHz, CDCl₃
0.97
4.00
6.09
6.06
f1 (ppm)


165.93
162.19
140.87
123.04
61.38
24.94
14.26
EtOOC
COOEt
N
10c, ¹H NMR
101 MHz, CDCl₃
f1 (ppm)

5. **Reference**

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