

**Rh(III)-catalyzed regioselective C–H activation
dialkenylation/annulation cascade for the rapid access to
6H-isoindolo[2,1-a]indole**

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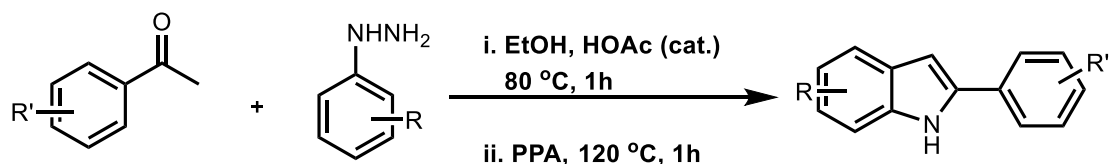
General Methods and Materials:

Unless specified, all reactions were carried out under air using commercially available solvents as received. Dichloro(η^5 -pentamethylcyclopentadienyl)rhodium(III) dimer was synthesized according to a previous literature.¹ 2-phenyl indole was purchased from Alfa Aesar and used directly. All other reagents were purchased and used without further purification unless specified otherwise. Solvents for chromatography were technical grade and distilled prior to use. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 – 0.2 mm). Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator. Visualization of the developed chromatogram was performed by UV absorbance (254 nm). ^1H NMR and ^{13}C NMR data were recorded on Varian VNMR 600, Varian VNMR 400 or Mercury 300 spectrometer. Chemical shifts (δ) in ppm are reported as quoted relative to the residual signals of chloroform (^1H 7.26 ppm or ^{13}C 77.16 ppm). Multiplicities are described as: s (singlet), bs (broad singlet), d (doublet), t (triplet), q (quartet), m (multiplet); and coupling constants (J) are reported in Hertz (Hz). ^{13}C NMR spectra were recorded with total proton decoupling. Mass spectra (MS-EI, 70 eV) were conducted on Finnigan MAT SSQ 700, unless otherwise specified, respectively. IR spectra were recorded on a Perkin Elmer Spectrum 100 spectrometer and are reported in terms of frequency of absorption (cm^{-1}).

Synthesis and Characterization of Starting Materials

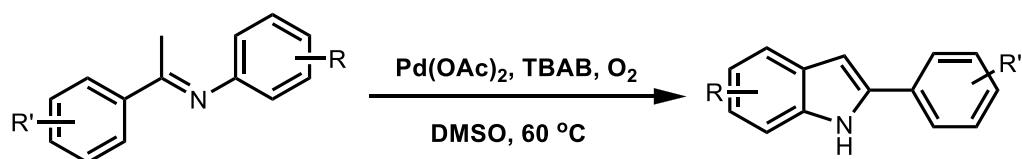
General procedure for preparation of 2-aryl indole substrates

Method A ²



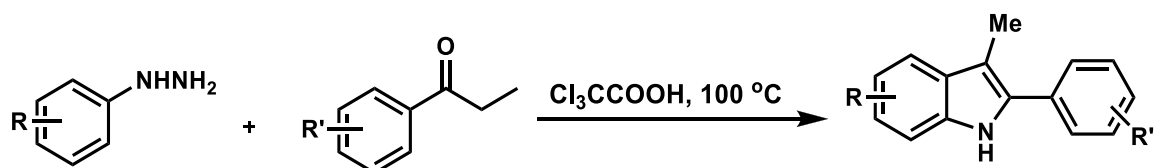
Substituted acetophenone (10 mmol) was mixed with related phenylhydrazine (10 mmol) in ethanol (25 mL) with a few drops of glacial acetic acid. The reaction was heated to 80 °C and stirred for 1 hour. Solvent was evaporated to yield the phenylhydrazone intermediate, which was added to polyphosphoric acid (20 g). Exothermic reaction was observed and the reaction mixture was slowly heated to 120 °C (keep for 1 hour). The mixtures were poured into crashed ice cube and then neutralized with 2M NaOH and extracted with EtOAc. The combined organic extracts were washed with water, dried over anhydrous Na₂SO₄, and purified by column to give the desired 2-aryl indole product.

Method B ³



A Schlenk tube equipped with a stirrer bar was charged with *N*-aryl imine (2 mmol), Pd(OAc)₂ (45 mg, 0.2 mmol, 10 mol%) and *n*-Bu₄NBr (1.29 g, 4 mmol), followed by addition of DMSO (10 mL). The Schlenk tube was quickly evacuated, closed under vacuum, and then refilled with oxygen using an oxygen balloon. The resulting mixture was stirred at 60 °C for 24 h. Upon cooling to room temperature, the reaction mixture was diluted with 50 mL of ethyl acetate, followed by filtration through a pad of silica gel. The filtrate was concentrated under reduced pressure, and the residue was purified by flash chromatography on silica gel to afford the indole product.

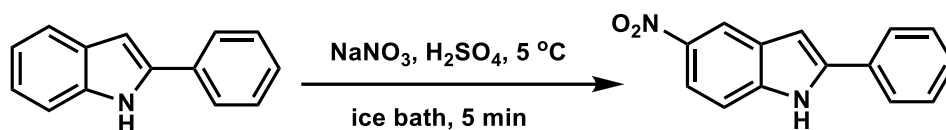
Method C ⁴



A mixture of phenylhydrazine (10 mmol), ketones (10 mmol) trichloroacetic acid (30 mmol) was heated, with swirling, in test tube at 100 °C for 5 min. Water was added to the cooled

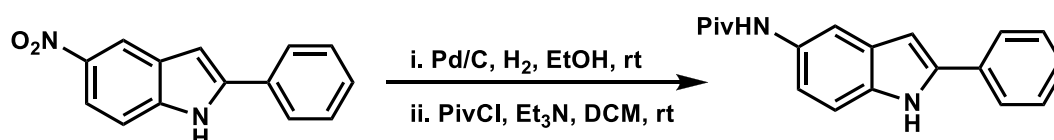
mixture which was filtered. The retentate was washed with water and dried in vacuum to give the corresponding analytically pure indoles.

Method D ⁵



A solution of 2-phenyl indole in conc. H_2SO_4 (100 mL) was cooled to $5\text{ }^\circ\text{C}$ in an ice bath. A solution of sodium nitrate in H_2SO_4 (50 mL), also cooled, was added, with stirring. The resulting mixture was kept for 5 min, then poured into crushed ice. The flocculent yellow precipitate was filtered and dried, crystallization by DCM-petroleum ether.

Method E

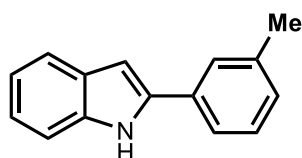


Step 1⁶

2-phenyl-5-nitro-1H-indole (1.63g, 6.84 mmol) and Pd/C (30% wt, 0.28 g) were added into a flask, then 50 ml EtOH was added via syringe. The vial was capped and evacuated/flushed with H_2 three times, then stirred under a hydrogen balloon for 18 h. Palladium catalyst was removed by filtration through a plug of Celite and washed thoroughly with ethyl acetate. The filtrate was concentrated under vacuum and the residue was pure enough for the next step use.

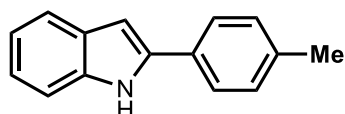
Step 2

The above obtained indole material (229 mg, 1.1 mmol) and Et_3N (318 μL , 2.18 mmol) were added into a vial containing 5 mL DCM. Then PivCl (109 μL , 1.31 mmol) was slowly added via a microsyringe at $0\text{ }^\circ\text{C}$, after addition, the mixture was stirred at rt overnight. After reaction, the product was purified by column.

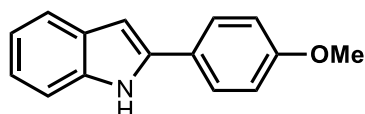


2-m-tolyl-1H-indole (**1b**)⁷: method A, yield: 72%, pale white solid. ^1H NMR (d_6 -DMSO, 600 MHz): δ ; 11.50 (s, 1H), 7.71 (s, 1H), 7.67 (d, $J = 7.8\text{ Hz}$, 1H), 7.53 (d, $J = 7.8\text{ Hz}$, 1H), 7.42 (d, $J = 7.8\text{ Hz}$, 1H), 7.34 (t, $J = 7.8\text{ Hz}$, 1H), 7.13 (d, $J = 7.2\text{ Hz}$, 1H), 7.10 (t, $J = 7.8\text{ Hz}$, 1H), 7.00 (t, $J = 7.2\text{ Hz}$, 1H), 6.87 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (d_6 -DMSO, 150 MHz): δ 138.0, 137.7, 137.1,

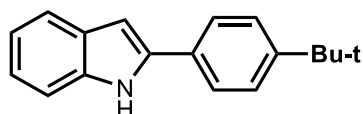
132.1, 128.7, 128.6, 128.0, 125.5, 122.2, 121.4, 120.0, 119.3, 111.3, 98.5, 21.1.



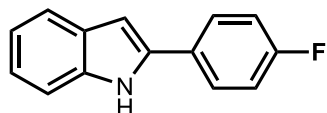
2-(4-methylphenyl)-1H-indole (**1c**)⁷: method A, yield: 93%, white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.45 (s, 1H), 7.75 (d, *J* = 7.8 Hz, 2H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 7.8 Hz, 2H), 7.08 (t, *J* = 7.8 Hz, 1H), 6.98 (t, *J* = 7.2 Hz, 1H), 6.82 (s, 1H), 2.33 (s, 3H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 137.8, 137.0, 136.7, 129.4, 128.7, 124.9, 121.3, 119.8, 119.3, 111.2, 98.0, 20.8.



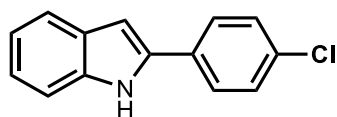
2-(4-methoxyphenyl)-1H-indole (**1d**)⁷: method B, yield: 87%, brown solid. ¹H NMR (d₆-DMSO, 400 MHz): δ 11.41 (s, 1H), 7.79 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.08-6.96 (m, 4H), 6.75 (s, 1H), 3.80 (s, 3H); ¹³C NMR (d₆-DMSO, 100 MHz): δ 158.8, 137.8, 136.9, 128.8, 126.3, 124.9, 121.0, 119.7, 119.2, 114.3, 111.1, 97.3, 55.2.



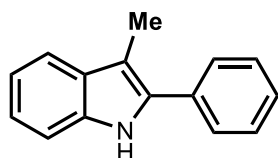
2-(4-tert-butylphenyl)-1H-indole (**1e**)⁷: method A, yield: 62%, white solid. ¹H NMR (CDCl₃, 600 MHz): δ 8.28 (s, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 8.4, 2H), 7.50 (d, *J* = 8.4, 2H), 7.41 (d, *J* = 8.4, 1H), 7.24 (td, *J* = 7.2, 1.2, 1H), 7.18 (td, *J* = 7.8, 1.2, 1H), 6.84 (d, *J* = 1.2, 1H), 1.41 (s, 9H); ¹³C NMR (CDCl₃, 150 MHz): δ 151.0, 138.1, 136.8, 129.6, 129.5, 126.1, 125.0, 122.2, 120.7, 120.3, 111.0, 99.6, 34.8, 31.4.



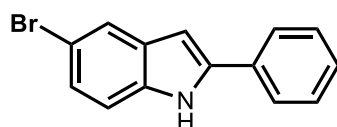
2-(4-fluorophenyl)-1H-indole (**1f**)⁷: method A, yield: 77%, white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.53 (s, 1H), 7.90 (t, *J* = 5.4, 2H), 7.53 (d, *J* = 7.8, 1H), 7.41 (d, *J* = 7.8, 1H), 7.31 (t, *J* = 7.8, 2H), 7.11 (t, *J* = 7.8, 1H), 7.01 (t, *J* = 7.8, 1H), 6.86 (s, 1H); ¹⁹F NMR (d₆-DMSO, 576 MHz) δ -114.7 (s); ¹³C NMR (d₆-DMSO, 150 MHz): δ 161.5 (d, *J*_F=243.0), 136.9 (d, *J*_F=60.0), 128.9, 128.6, 126.9 (d, *J*_F=9.0), 121.5, 120.0, 119.4, 115.8 (d, *J*_F=21.0), 111.3, 98.6.



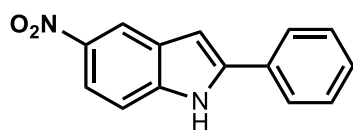
2-(4-chlorophenyl)-1H-indole (**1g**)⁷: method A, yield: 67%, brownish white solid ¹H NMR (d₆-DMSO, 400 MHz): δ 11.58 (s, 1H), 7.88 (d, *J* = 8.4, 2H), 7.53 (t, *J* = 8.0, 3H), 7.41 (d, *J* = 8.0, 1H), 7.12 (t, *J* = 7.2, 1H), 7.01 (t, *J* = 7.2, 1H), 6.93 (s, 1H); ¹³C NMR (d₆-DMSO, 100 MHz): δ 137.2, 136.3, 131.7, 131.1, 128.9, 128.5, 126.6, 121.9, 120.2, 119.5, 111.3, 99.3.



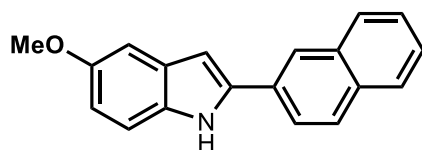
3-methyl-2-phenyl-1H-indole (**1h**)⁴: method C, yield: 82%, yellow solid. ¹H NMR (d₆-DMSO, 400 MHz): δ 11.16 (s, 1H), 7.69 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.54-7.49 (m, 3H), 7.39-7.33 (m, 2H), 7.12 (td, *J* = 7.2, 0.8 Hz, 1H), 7.02 (td, *J* = 7.2, 0.8 Hz, 1H), 2.42 (s, 3H); ¹³C NMR (d₆-DMSO, 100 MHz): δ 135.9, 133.7, 133.1, 129.4, 128.7, 127.5, 126.9, 121.5, 118.6, 118.4, 110.0, 106.8, 9.84.



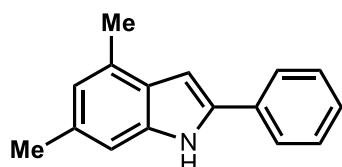
5-bromo-2-phenyl-1H-indole (**1i**)⁷: method B, yield: 93%, pale yellow white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.76 (s, 1H), 7.86 (d, *J* = 7.2 Hz, 2H), 7.71 (s, 1H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.38-7.33 (m, 2H), 7.21 (d, *J* = 8.4 Hz, 1H), 6.89 (s, 1H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 139.2, 135.8, 131.6, 130.5, 129.0, 127.9, 125.2, 124.0, 122.1, 113.2, 111.9, 98.3.



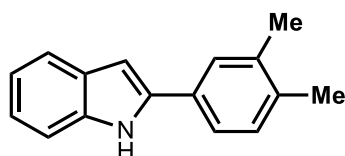
5-nitro-2-phenyl-1H-indole (**1j**)⁵: method D, yield: 95%, red solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 12.29 (s, 1H), 8.53 (d, *J* = 2.4 Hz, 1H), 8.01 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.90 (d, *J* = 7.2 Hz, 2H), 7.56 (d, *J* = 9.0 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.16 (d, *J* = 1.2 Hz, 1H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 141.4, 141.0, 140.3, 131.0, 129.1, 128.5, 127.9, 125.4, 117.0, 111.6, 100.8.



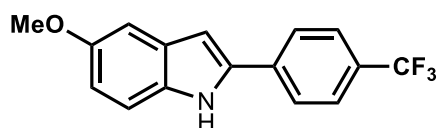
5-methoxy-2-(naphthalen-2-yl)-1H-indole (**1k**)³: method B, yield: 83%, pale yellow white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.54 (s, 1H), 8.35 (s, 1H), 7.98 (q, *J* = 8.4 Hz, 2H), 7.92 (t, *J* = 9.0 Hz, 2H), 7.55 (t, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.06 (s, 1H), 6.96 (s, 1H), 6.78 (dd, *J* = 8.4, 1.2 Hz, 1H), 3.78 (s, 3H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 153.7, 138.0, 132.5, 132.2, 129.8, 128.3, 127.8, 127.7, 126.6, 125.9, 123.7, 122.6, 112.1, 112.0, 101.6, 99.4, 55.3.



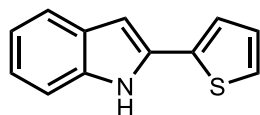
4,6-dimethyl-2-phenyl-1H-indole (**1l**)⁸: method B, yield: 61%, pale grey solid. ¹H NMR (CDCl₃, 600 MHz): δ 8.27 (s, 1H), 7.67 (d, *J* = 7.8 Hz, 2H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.02 (s, 1H), 6.84 (d, *J* = 1.2 Hz, 1H), 6.82 (s, 1H), 2.60 (s, 3H), 2.47 (s, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 137.1, 136.7, 132.7, 132.4, 129.9, 129.0, 127.4, 127.1, 125.0, 122.4, 108.6, 98.5, 21.9, 18.8.



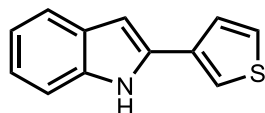
2-(3,4-dimethylphenyl)-1H-indole (**1m**)⁹: method A, yield: 57%, pale white solid. ¹H NMR (CDCl₃, 400 MHz): δ 8.29 (s, 1H), 7.65 (dd, *J* = 7.6, 0.4 Hz, 1H), 7.47 (s, 1H), 7.42-7.39 (m, 2H), 7.23-7.19 (m, 2H), 7.15 (td, *J* = 7.6, 0.8 Hz, 1H), 6.81 (d, *J* = 1.6 Hz, 1H), 2.36 (s, 3H), 2.33 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 138.3, 137.3, 136.8, 136.5, 130.4, 130.1, 129.5, 126.6, 122.7, 122.1, 120.6, 120.3, 110.9, 99.4, 20.1, 19.7.



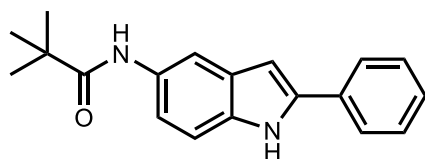
5-methoxy-2-(4-(trifluoromethyl)phenyl)-1H-indole (**1n**)³: method B, yield: 71%, white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.58 (s, 1H), 8.03 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 9.0 Hz, 2H), 7.33 (d, *J* = 9.0 Hz, 1H), 7.05 (d, *J* = 2.4 Hz, 1H), 6.97 (d, *J* = 1.2 Hz, 1H), 6.80 (dd, *J* = 9.0, 2.4 Hz, 1H), 3.77 (s, 3H); ¹⁹F NMR (d₆-DMSO, 576 MHz) δ -60.9 (s); ¹³C NMR (d₆-DMSO, 150 MHz): δ 153.8, 136.2, 136.2, 132.7, 128.8, 127.1 (q, *J*_F=31.8), 126.6 (q, *J*_F=270.0), 125.8 (q, *J*_F=3.6), 125.1, 113.0, 112.3, 101.7, 100.5, 55.2.



2-(thiophen-2-yl)-1H-indole (**1o**)¹⁰: method A, yield: 51%, pale grey solid. ¹H NMR (d₆-DMSO, 400 MHz): δ 11.55 (s, 1H), 7.53-7.49 (m, 3H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.15 (t, *J* = 4.0 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.67 (d, *J* = 1.2 Hz, 1H); ¹³C NMR (d₆-DMSO, 100 MHz): δ 136.8, 135.5, 132.4, 128.5, 128.1, 125.1, 123.5, 121.7, 119.9, 119.5, 111.1, 98.7.

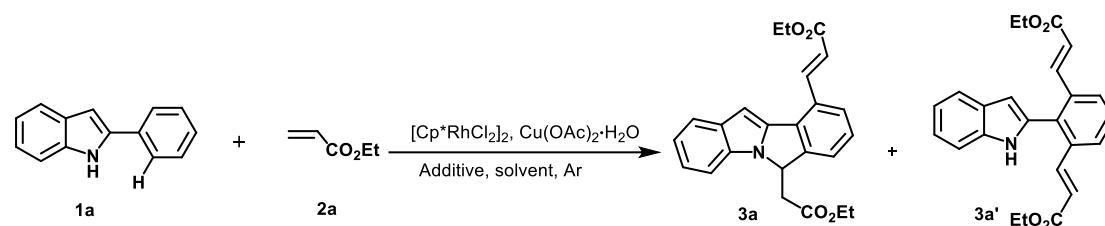


2-(thiophen-3-yl)-1H-indole (**1p**)¹¹: method A, yield: 49%, white solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.45 (s, 1H), 7.87 (d, *J* = 1.2 Hz, 1H), 7.64-7.61 (m, 2H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.2 Hz, 1H), 6.76 (s, 1H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 136.6, 134.1, 134.1, 128.5, 127.0, 125.9, 121.4, 119.9, 119.6, 119.3, 110.0, 98.6.



N-(2-phenyl-1H-indol-5-yl)pivalamide (**1q**)¹²: method E, yield: 88%, red brown solid. ¹H NMR (d₆-DMSO, 600 MHz): δ 11.42 (s, 1H), 9.03 (s, 1H), 7.85 (d, *J* = 7.8 Hz, 2H), 7.82 (s, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.32-7.26 (s, 3H), 6.86 (s, 1H), 1.25 (s, 9H); ¹³C NMR (d₆-DMSO, 150 MHz): δ 176.0, 138.0, 134.0, 132.2, 131.6, 128.8, 128.3, 127.3, 124.9, 117.0, 112.2, 110.7, 98.7, 27.4.

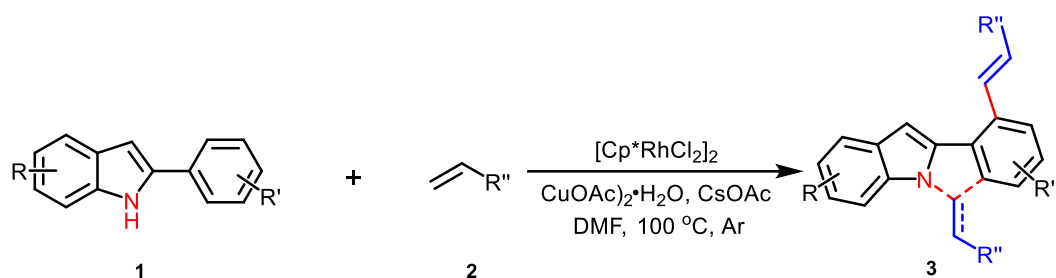
Condition Screening:



Entry	Temperature (°C)	Solvent	Additive	Yield ^a of 3a (%)
1	100	DMF	–	54 (3a')
2	100	<i>o</i> -xylene	Na_2CO_3	10
3	100	DMF	CsOAc	55
4	100	DMF	<i>t</i> -BuOK	–
5	100	DMF	$\text{K}_3\text{PO}_4 \cdot 3\text{H}_2\text{O}$	23
6	100	DMF	NaOH	41
7 ^b	100	DMF	CsOAc	trace
8 ^c	100	DMF	CsOAc	<5
9 ^d	100	DMF	CsOAc	–
10 ^e	100	DMF	CsOAc	trace
11 ^f	100	DMF	CsOAc	–
12	100	DMF	CsOAc	75
13	rt	DMF	CsOAc	trace
14	60	DMF	–	50 (3a')
15 ^h	80	DMF	CsOAc	40
16 ^h	100	DMF	CsOAc	77
17 ^{h-i}	100	DMF	CsOAc	67 (45h)
18 ^{h-j}	100	DMF	CsOAc	81 (45h)
19^{g,j}	100	DMF	CsOAc	86 (41h)
20 ^{i-k}	100	DMF	CsOAc	78 (54h)
21	100	<i>t</i> -AmylOH	AgSbF_6 (0.15 equiv.)	trace

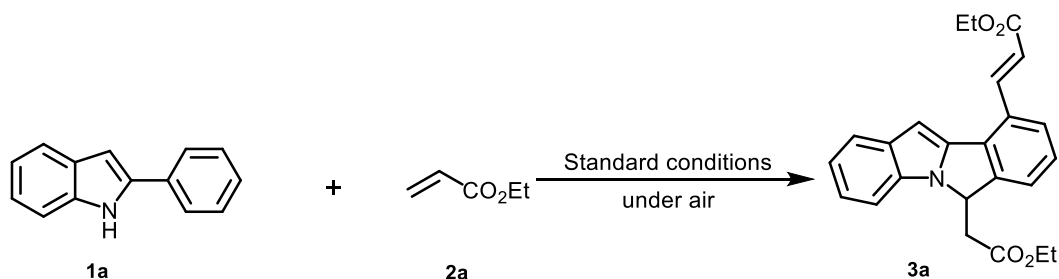
- Reaction on a 0.2 mmol scale, using **1a** (1.0 equiv.), **2a** (1.5 equiv.), $[\text{Cp}^*\text{RhCl}_2]_2$ (3 mol%), additive (2 equiv.), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (2 equiv.), solvent (1.5 mL), under argon, 21 h, isolated yield.
- Without $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$.
- 10 mol% $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$, under air.
- Without $[\text{Cp}^*\text{RhCl}_2]_2$.
- $[\text{RuCl}_2(p\text{-cymene})]_2$ as catalyst.
- $[\text{Rh}(\text{PPh}_3)_3\text{Cl}]$ as catalyst.
- 2.5 equiv. ethyl acrylate was used.
- 1.1 equiv. CsOAc was used.
- 4 equiv. ethyl acrylate was used.
- 5 mol% $[\text{Cp}^*\text{RhCl}_2]_2$ was used.
- 4 equiv. $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ was used.

General Procedure for Rh(III)-Catalyzed C–H Activation



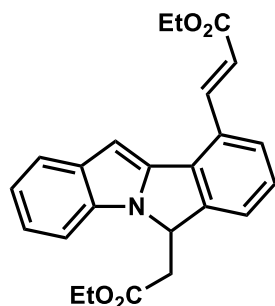
$[\text{Cp}^*\text{RhCl}_2]_2$ (0.01 mmol, 6.2 mg, 5 mol%), CsOAc (0.4 mmol, 76.8 mg, 2.0 equiv.), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (0.4 mmol, 79.9 mg, 2.0 equiv.) and 2-aryl indole substrate **1** (0.2 mmol, 1.0 equiv.) were weighed into a Schlenk tube. The reaction vessel was capped and subjected to three vacuum-purge/argon-flush cycles. A solution of olefin **2** (0.5 mmol, 2.5 equiv.) in DMF (1.5 mL) was then added through the side-arm by syringe. The reaction was stirred under argon at 100 °C and the progress of the olefination annulation was monitored by TLC. Upon complete consumption of **1**, the reaction was cooled to room temperature. Volatile solvent and reagents were removed by rotary evaporation and the residue was purified by silica gel flash chromatography using *n*-Hexane/EtOAc (20:1 to 10:1) to afford product **3**.

Reaction under air

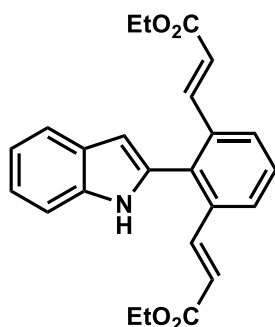


Using a similar procedure as above: $[\text{Cp}^*\text{RhCl}_2]_2$ (0.01 mmol, 6.2 mg, 5 mol%), CsOAc (0.4 mmol, 76.8 mg, 2.0 equiv.), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (0.4 mmol, 79.9 mg, 2.0 equiv.) and 2-phenyl indole **1a** (0.2 mmol, 38.6 mg, 1.0 equiv.) were weighed into a Schlenk tube. A solution of acrylate **2a** (0.5 mmol, 2.5 equiv.) in DMF (1.5 mL) was then added through the side-arm by syringe. The vessel was sealed with a TFE cap and the reaction was stirred at 100 °C. The progress of the olefination annulation was monitored by TLC. Upon complete consumption of **1a**, the reaction was cooled to room temperature. Volatile solvent and reagents were removed by rotary evaporation and the residue was purified by silica gel flash chromatography using *n*-Hexane/EtOAc (20:1 to 10:1) to afford product **3a**.

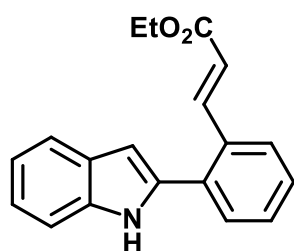
Characterization of Products:



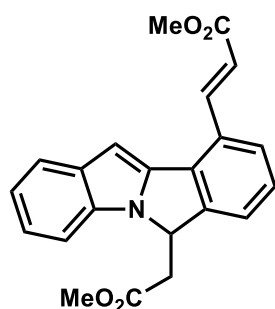
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3a**)¹³: yield: 86%, yellow solid, melting point: 102-104 °C. ¹H NMR (CDCl₃, 600 MHz): δ 8.32 (d, *J* = 16.2 Hz, 1H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 1H), 7.14 (t, *J* = 7.8 Hz, 1H), 6.80 (s, 1H), 6.56 (d, *J* = 16.2 Hz, 1H), 5.73 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.35 (q, *J* = 7.2 Hz, 2H), 4.25-4.20 (m, 2H), 3.30 (ABq, *J* = 16.8, 4.8 Hz, 1H), 2.76 (ABq, *J* = 16.2, 8.4 Hz, 1H), 1.41 (t, *J* = 7.2 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 170.7, 166.9, 146.6, 141.3, 140.7, 133.5, 133.2, 132.2, 129.0, 127.6, 125.9, 124.7, 122.5, 122.3, 120.5, 120.2, 109.7, 95.9, 61.3, 60.8, 56.5, 39.6, 14.5, 14.2.



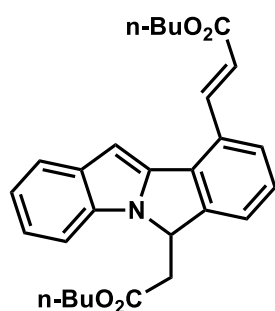
diethyl 3,3'-(2-(1H-indol-2-yl)-1,3-phenylene)(2*E*,2'*E*)-diacrylate (**3a'**)¹³: yield: 61%, yellow solid, melting point: 158-160 °C. ¹H NMR (CDCl₃, 300 MHz): δ 8.33 (s, 1H), 7.72-7.67 (m, 3H), 7.61 (d, *J* = 16.0 Hz, 2H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 7.9 Hz, 1H), 7.26-7.15 (m, 2H), 6.53 (s, 1H), 6.36 (d, *J* = 16.0 Hz, 2H), 4.14 (q, *J* = 7.1 Hz, 4H), 1.22 (t, *J* = 7.1 Hz, 6H); ¹³C NMR (CDCl₃, 75 MHz): δ 166.6, 142.7, 136.8, 135.8, 131.6, 129.0, 128.5, 128.0, 122.8, 121.0, 120.7, 120.4, 111.1, 107.3, 60.7, 14.3.



ethyl (*E*)-3-(2-(1H-indol-2-yl)phenyl)acrylate (**3a''**)¹³: yield: 16%, yellow oil. ¹H NMR (CDCl₃, 300 MHz): δ 8.29 (s, 1H), 8.07 (d, *J* = 16.0 Hz, 1H), 7.67 (d, *J* = 7.4 Hz, 2H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.47-7.36 (m, 3H), 7.24 (d, *J* = 6.6 Hz, 1H), 7.20-7.12 (m, 1H), 6.60 (s, 3H), 6.44 (d, *J* = 16.0 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 166.9, 143.6, 136.9, 135.7, 133.5, 133.3, 130.0, 129.4, 128.9, 128.2, 127.7, 122.7, 121.0, 120.3, 111.1, 105.3, 60.7, 14.4.

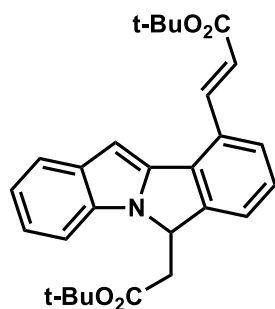


(*E*)-methyl 3-(6-(2-methoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3b**): yield: 77%, yellow solid, melting point: 108-110 °C. ¹H NMR (CDCl₃, 400 MHz): δ 8.32 (d, *J* = 16.0 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.38 (d, *J* = 9.6 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.23 (td, *J* = 7.2, 1.2 Hz, 1H), 7.14 (td, *J* = 8.0, 1.2 Hz, 1H), 6.80 (s, 1H), 6.57 (d, *J* = 16.0 Hz, 1H), 5.75 (dd, *J* = 8.4, 4.4 Hz, 1H), 3.89 (s, 3H), 3.79 (s, 3H), 3.32 (ABq, *J* = 16.4, 4.8 Hz, 1H), 2.73 (ABq, *J* = 16.4, 8.4 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz): δ 171.3, 167.3, 146.6, 141.2, 140.9, 133.6, 133.2, 132.2, 129.0, 127.7, 126.0, 124.8, 122.6, 122.4, 120.2, 120.1, 109.6, 96.0, 56.5, 52.4, 52.1, 39.5. IR (ATR): 2950, 1720, 1431, 1164, 978, 744 cm⁻¹. EI-MS *m/z* (%): 361.2 (92) [M]⁺, 288.1 (100) [M-CH₂CO₂Me]⁺. HRMS (ESI): found: 362.1387, calcd. for C₂₂H₂₀NO₄ ([M+H]⁺): 362.1392. Anal. Calcd. for C₂₂H₁₉NO₄: C, 73.12; H, 5.30; N, 3.88; found: C, 74.29; H, 5.16; N, 3.61.

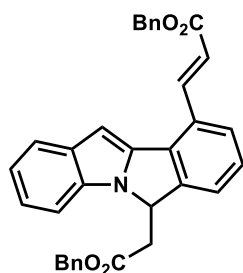


(*E*)-butyl 3-(6-(2-butoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3c**)¹³: yield: 78%, yellow semi-solid. ¹H NMR (CDCl₃, 300 MHz): δ 8.23 (d, *J* = 15.9 Hz, 1H), 7.61 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 6.9 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.18-7.12 (m, 1H), 7.08-7.03 (m, 1H), 6.70 (s, 1H), 6.48 (dd, *J* = 15.9, 1.2 Hz, 1H), 5.64 (dd, *J* = 7.8, 4.2 Hz, 1H), 4.21 (td, *J* = 6.3, 0.9 Hz, 2H), 4.11-4.03 (m, 2H), 3.22 (ABq, *J* = 16.2, 4.5 Hz, 1H), 2.69 (ABq, *J* = 16.2, 8.1 Hz, 1H), 1.73-1.63 (m, 2H), 1.50-1.37 (m, 4H), 1.28-1.16 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H), 0.82 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 170.7,

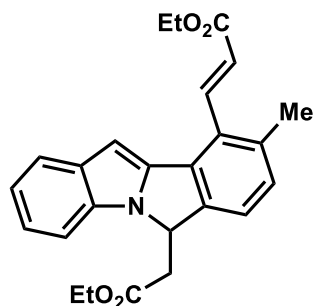
166.9, 146.6, 141.3, 140.6, 133.5, 133.2, 132.2, 129.0, 127.6, 125.9, 124.7, 122.5, 122.3, 120.4, 120.2, 109.7, 95.9, 65.2, 64.7, 56.6, 39.6, 31.0, 30.6, 19.4, 19.2, 13.9, 13.8.



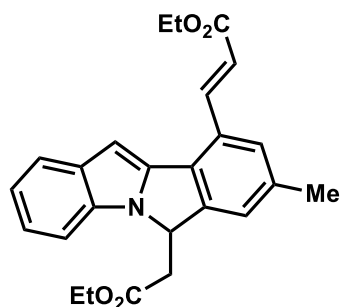
(*E*)-*tert*-butyl 3-(6-(2-*tert*-butoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3d**)¹³: yield: 73%, yellow solid, melting point: 124-126 °C. ¹H NMR (CDCl₃, 300 MHz): δ 8.25 (d, *J* = 15.9 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.22 (td, *J* = 7.2, 1.2 Hz, 1H), 7.13 (td, *J* = 7.8, 0.9 Hz, 1H), 6.80 (s, 1H), 6.50 (d, *J* = 15.9 Hz, 1H), 5.69 (dd, *J* = 7.5, 4.2 Hz, 1H), 3.21 (ABq, *J* = 16.2, 4.5 Hz, 1H), 2.79 (ABq, *J* = 16.2, 7.8 Hz, 1H), 1.61 (s, 9H), 1.36 (s, 9H); ¹³C NMR (CDCl₃, 150 MHz): δ 169.7, 166.3, 146.7, 141.5, 139.7, 133.5, 133.3, 132.3, 129.1, 127.5, 125.7, 124.5, 122.4, 122.2, 122.1, 120.1, 109.8, 95.7, 81.7, 80.9, 56.7, 40.5, 28.4, 28.0.



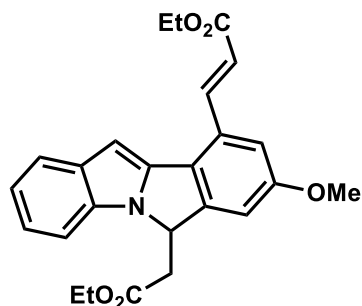
(*E*)-benzyl 3-(6-(2-(benzyloxy)-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3e**): yield: 57%, pale yellow solid, melting point: 120-122 °C. ¹H NMR (CDCl₃, 300 MHz): δ 8.30 (d, *J* = 16.0 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 7.47-7.24 (m, 12H), 7.19-7.08 (m, 3H), 6.69 (s, 1H), 6.55 (d, *J* = 15.9 Hz, 1H), 5.62 (dd, *J* = 7.8, 4.2 Hz, 1H), 5.30 (s, 2H), 5.14 (dd, *J* = 15.1, 12.2 Hz, 2H), 3.28 (ABq, *J* = 16.2, 4.4 Hz, 1H), 2.75 (ABq, *J* = 16.2, 8.1 Hz, 1H); ¹³C NMR (CDCl₃, 100 MHz): δ 170.4, 166.7, 146.4, 141.3, 141.2, 128.8, 128.7, 128.6, 128.6, 128.5, 128.4, 127.6, 125.9, 124.8, 122.6, 122.3, 120.2, 119.9, 109.6, 96.0, 67.2, 66.7, 56.5, 39.5. IR (ATR): 3055, 2937, 1715, 1638, 1308, 1174, 973, 736 cm⁻¹. EI-MS *m/z* (%): 513.3 (4) [M]⁺, 364.2 (9) [M-CH₂CO₂Bn]⁺. HRMS (ESI): found: 514.2012, calcd. for C₃₄H₂₈NO₄ ([M+H]⁺): 514.2018. Anal. Calcd. for C₃₄H₂₇NO₄: C, 79.51; H, 5.30; N, 2.73; found: C, 80.02; H, 5.05; N, 2.91.



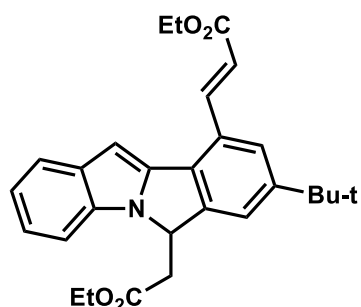
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-9-methyl-6H-indolo[2,1-a]indol-10-yl)acrylate (**3f**):
 yield: 62%, yellow solid, melting point: 86-88 °C. ¹H NMR (CDCl₃, 300 MHz): δ 8.25 (d, *J* = 15.9 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.20 (t, *J* = 7.0 Hz, 1H), 7.13 (t, *J* = 7.1 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.71 (s, 1H), 6.48 (d, *J* = 15.9 Hz, 1H), 5.58-5.55 (m, 1H), 4.35 (q, *J* = 7.1 Hz, 2H), 4.05-3.98 (m, 2H), 3.13 (ABq, *J* = 16.2, 3.5 Hz, 1H), 2.81 (ABq, *J* = 16.2, 5.7 Hz, 1H), 2.37 (s, 3H), 1.42 (t, *J* = 7.1 Hz, 3H), 1.03 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 170.5, 167.0, 144.1, 141.5, 140.6, 134.7, 133.4, 133.0, 132.4, 129.5, 126.5, 125.8, 122.3, 122.0, 120.0, 119.2, 109.9, 95.7, 61.1, 60.7, 56.2, 38.2, 18.8, 14.5, 13.9. IR (ATR): 2980, 1710, 1633, 1451, 1159, 1033, 738 cm⁻¹. EI-MS *m/z* (%): 403.5 (1) [M]⁺, 316.2 (3) [M-CH₂CO₂Et]⁺. HRMS (ESI): found: 404.1856, calcd. for C₂₅H₂₆NO₄ ([M+H]⁺): 404.1862. Anal. Calcd. for C₂₅H₂₅NO₄: C, 74.42; H, 6.25; N, 3.47; found: C, 74.82; H, 6.05; N, 3.67.



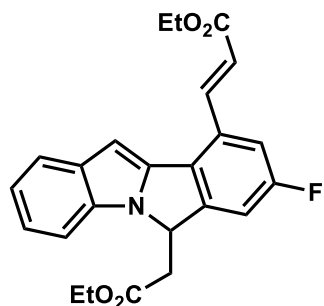
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-8-methyl-6H-indolo[2,1-a]indol-10-yl)acrylate (**3g**):
 yield: 88%, yellow solid, melting point: 96-98 °C. ¹H NMR (CDCl₃, 600 MHz): δ 8.27 (d, *J* = 15.6 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.42 (s, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.26 (s, 1H), 7.20 (td, *J* = 7.2, 0.6 Hz, 1H), 7.12 (td, *J* = 7.2, 0.6 Hz, 1H), 6.73 (s, 1H), 6.54 (d, *J* = 15.6 Hz, 1H), 5.65 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.34 (q, *J* = 7.2 Hz, 2H), 4.26-4.18 (m, 2H), 3.26 (ABq, *J* = 16.2, 4.8 Hz, 1H), 2.73 (ABq, *J* = 16.8, 8.4 Hz, 1H), 2.41 (s, 3H), 1.40 (t, *J* = 7.2 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 170.8, 166.9, 146.9, 141.4, 140.7, 137.8, 133.6, 133.1, 129.8, 128.7, 126.4, 125.7, 122.2, 122.1, 120.1, 120.0, 109.5, 95.1, 61.3, 60.8, 56.4, 40.0, 21.7, 14.5, 14.2. IR (ATR): 2981, 1712, 1637, 1454, 1170, 1035, 741 cm⁻¹. EI-MS *m/z* (%): 403.3 (57) [M]⁺, 316.1 (100) [M-CH₂CO₂Et]⁺. HRMS (ESI): found: 426.1676, calcd. for C₂₅H₂₅NO₄Na ([M+Na]⁺): 426.1681. Anal. Calcd. for C₂₅H₂₅NO₄: C, 74.42; H, 6.25; N, 3.47; found: C, 75.07; H, 5.85; N, 3.02.



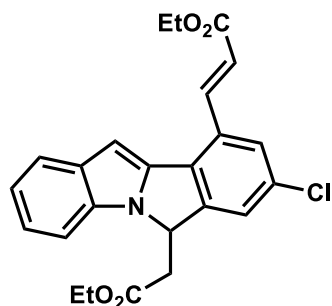
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-8-methoxy-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3h**): yield: 80%, yellow solid, melting point: 103-105 °C. ^1H NMR (CDCl_3 , 600 MHz): δ 8.25 (dd, J = 15.6, 3.0 Hz, 1H), 7.67 (d, J = 8.4 Hz, 1H), 7.34 (d, J = 8.4 Hz, 1H), 7.19 (t, J = 7.2 Hz, 1H), 7.12 (d, J = 7.8 Hz, 1H), 7.10 (s, 1H), 7.06 (s, 1H), 6.67 (s, 1H), 6.52 (dd, J = 16.2, 2.4 Hz, 1H), 5.67-5.66 (m, 1H), 4.34 (q, J = 7.2 Hz, 2H), 4.23 (qd, J = 7.2, 1.2 Hz, 2H), 3.86 (d, J = 3.0 Hz, 3H), 3.31-3.27 (m, 1H), 2.73 (ABq, J = 16.8, 8.4 Hz, 1H), 1.41 (t, J = 7.2 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 170.7, 166.8, 159.6, 148.5, 141.3, 140.6, 133.6, 133.2, 129.7, 125.5, 122.0, 122.0, 112.2, 110.1, 109.4, 94.4, 61.3, 60.9, 56.5, 55.8, 39.7, 14.5, 14.2. IR (ATR): 2980, 1710, 1635, 1466, 1172, 1032, 736 cm^{-1} . EI-MS m/z (%): 419.4 (100) $[\text{M}]^+$, 332.3 (37) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 442.1625, calcd. for $\text{C}_{25}\text{H}_{25}\text{NO}_5\text{Na}$ ($[\text{M}+\text{Na}]^+$): 442.1630. Anal. Calcd. for $\text{C}_{25}\text{H}_{25}\text{NO}_5$: C, 71.58; H, 6.01; N, 3.34; found: C, 72.12; H, 5.73; N, 3.05.



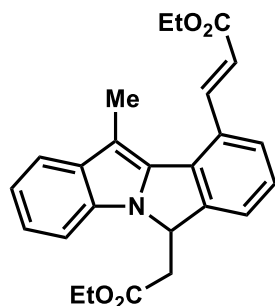
(*E*)-ethyl 3-(8-tert-butyl-6-(2-ethoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3i**): yield: 90%, yellow solid, melting point: 142-144 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.32 (d, J = 16.0 Hz, 1H), 7.70 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 1.6 Hz, 1H), 7.52 (s, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.22 (td, J = 7.2, 1.2 Hz, 1H), 7.14 (td, J = 8.0, 0.8 Hz, 1H), 6.76 (s, 1H), 6.60 (d, J = 16.0 Hz, 1H), 5.72 (dd, J = 8.0, 4.8 Hz, 1H), 4.37 (q, J = 7.2 Hz, 2H), 4.30-4.22 (m, 2H), 3.32 (ABq, J = 16.0, 4.8 Hz, 1H), 2.73 (ABq, J = 16.0, 8.0 Hz, 1H), 1.43 (t, J = 7.2 Hz, 3H), 1.39 (s, 9H), 1.27 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 170.9, 166.9, 151.2, 146.7, 141.3, 141.2, 133.6, 133.2, 129.7, 128.3, 122.7, 122.2, 122.2, 122.1, 120.0, 120.0, 109.6, 95.2, 61.3, 60.8, 56.6, 39.9, 35.2, 31.5, 14.5, 14.3; IR (ATR): 2963, 1731, 1636, 1452, 1308, 1181, 1034, 857, 737 cm^{-1} . EI-MS m/z (%): 445.5 (100) $[\text{M}]^+$, 388.4 (21) $[\text{M}-(\text{CH}_3)_3\text{C}]^+$, 358.4 (93) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 468.2145, calcd. for $\text{C}_{28}\text{H}_{31}\text{NO}_4\text{Na}$ ($[\text{M}+\text{Na}]^+$): 468.2151. Anal. Calcd. for $\text{C}_{28}\text{H}_{31}\text{NO}_4$: C, 75.48; H, 7.01; N, 3.14; found: C, 76.06; H, 6.76; N, 3.37.



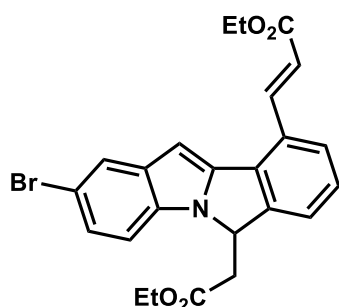
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-8-fluoro-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3j**): yield: 57%, yellow solid, melting point: 100-102 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.24 (dd, J = 16.0, 1.2 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.36 (dd, J = 8.0, 0.8 Hz, 1H), 7.31 (dd, J = 9.6, 2.0 Hz, 1H), 7.25-7.22 (m, 2H), 7.13 (td, J = 8.0, 0.8 Hz, 1H), 6.74 (s, 1H), 6.52 (d, J = 16.0 Hz, 1H), 5.71 (dd, J = 8.4, 4.0 Hz, 1H), 4.34 (q, J = 7.2 Hz, 2H), 4.23 (qd, J = 7.2, 1.2 Hz, 2H), 3.33 (ABq, J = 16.4, 4.0 Hz, 1H), 2.72 (ABq, J = 16.4, 8.4 Hz, 1H), 1.40 (t, J = 7.2 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H); ^{19}F NMR (CDCl_3 , 376 MHz) δ -113.1 (m); ^{13}C NMR (CDCl_3 , 100 MHz): δ 170.5, 166.5, 162.3 (J_{F} = 247.0), 148.7 (J_{F} = 9.0), 140.4, 139.5, 133.5, 133.2, 130.3, 130.2, 128.6, 122.5 (J_{F} = 24.0), 121.6, 120.3, 113.1 (J_{F} = 15.0), 112.4 (J_{F} = 23.0), 109.5, 95.4, 61.5, 61.0, 56.5, 39.4, 14.5, 14.2; IR (ATR): 2982, 1715, 1638, 1465, 1260, 1172, 1033, 862, 741 cm^{-1} . EI-MS m/z (%): 407.4 (100) $[\text{M}]^+$, 320.3 (82) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 430.1425, calcd. for $\text{C}_{24}\text{H}_{22}\text{NO}_4\text{NaF}$ ($[\text{M}+\text{Na}]^+$): 430.1431. Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{FNO}_4$: C, 70.75; H, 5.44; N, 3.44; found: C, 70.01; H, 4.86; N, 3.81.



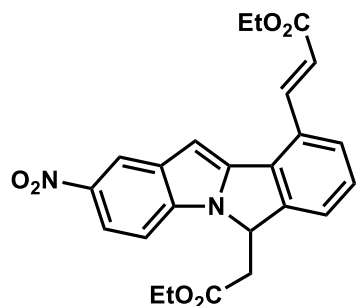
(*E*)-ethyl 3-(8-chloro-6-(2-ethoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3k**): yield: 55%, yellow solid, melting point: 127-129 °C. ^1H NMR (CDCl_3 , 300 MHz): δ 8.18 (d, J = 16.0 Hz, 1H), 7.68 (d, J = 7.8 Hz, 1H), 7.56 (s, 1H), 7.44 (s, 1H), 7.34 (d, J = 8.1 Hz, 1H), 7.22 (t, J = 7.2 Hz, 1H), 7.15 (t, J = 7.5 Hz, 1H), 6.74 (s, 1H), 6.52 (d, J = 16.0 Hz, 1H), 5.64 (dd, J = 8.1, 3.9 Hz, 1H), 4.33 (q, J = 7.2 Hz, 2H), 4.25-4.18 (m, 2H), 3.28 (ABq, J = 16.5, 3.9 Hz, 1H), 2.71 (ABq, J = 16.5, 8.4 Hz, 1H), 1.39 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 170.4, 166.5, 148.1, 140.2, 139.3, 133.4, 133.2, 130.8, 129.9, 125.9, 125.1, 122.8, 122.4, 121.6, 120.4, 109.6, 96.1, 61.5, 61.0, 56.4, 39.3, 14.5, 14.2; IR (ATR): 2982, 1716, 1638, 1449, 1310, 1172, 1032, 860, 739 cm^{-1} . EI-MS m/z (%): 423.2 (97) $[\text{M}]^+$, 336.1 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 446.1130, calcd. for $\text{C}_{24}\text{H}_{22}\text{NO}_4\text{NaCl}$ ($[\text{M}+\text{Na}]^+$): 446.1135. Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{ClNO}_4$: C, 68.00; H, 5.23; N, 3.30; found: C, 68.75; H, 4.61; N, 3.72.



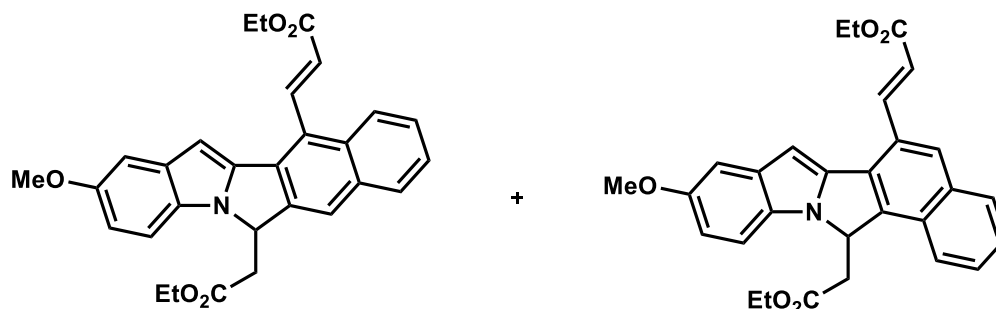
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-11-methyl-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3l**): yield: 43%, yellow solid, melting point: 112-114 °C. ^1H NMR (CDCl_3 , 300 MHz): δ 8.58 (d, J = 15.9 Hz, 1H), 7.67 (d, J = 8.1, Hz, 1H), 7.56 (d, J = 7.8 Hz, 1H), 7.44 (d, J = 7.5 Hz, 1H), 7.34 (d, J = 8.1 Hz, 1H), 7.29 (d, J = 7.8 Hz, 1H), 7.23 (t, J = 8.1 Hz, 1H), 7.14 (t, J = 7.2 Hz, 1H), 6.45 (d, J = 15.9 Hz, 1H), 5.67 (dd, J = 7.8, 4.2 Hz, 1H), 4.32 (q, J = 7.2 Hz, 2H), 4.21 (q, J = 6.9 Hz, 2H), 3.26 (ABq, J = 16.2, 4.2 Hz, 1H), 2.73-2.65 (m, 4H), 1.38 (t, J = 7.2 Hz, 3H), 1.23 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 170.8, 166.9, 146.9, 144.3, 138.4, 134.3, 133.4, 133.0, 128.8, 127.3, 126.8, 124.5, 122.8, 120.4, 120.1, 119.5, 109.4, 104.7, 61.3, 60.8, 55.7, 40.1, 14.5, 14.2, 11.5; IR (ATR): 2980, 1709, 1633, 1463, 1164, 1032, 865, 738 cm^{-1} . EI-MS m/z (%): 403.5 (44) $[\text{M}]^+$, 316.3 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 426.1676, calcd. for $\text{C}_{25}\text{H}_{25}\text{NO}_4\text{Na}$ ($[\text{M}+\text{Na}]^+$): 426.1681. Anal. Calcd. for $\text{C}_{25}\text{H}_{25}\text{NO}_4$: C, 74.42; H, 6.25; N, 3.27; found: C, 75.15; H, 5.66; N, 3.54.



(*E*)-ethyl 3-(2-bromo-6-(2-ethoxy-2-oxoethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3m**): yield: 45%, yellow semi-solid. ^1H NMR (CDCl_3 , 300 MHz): δ 8.23 (d, J = 15.9 Hz, 1H), 7.78 (s, 1H), 7.61 (d, J = 7.8 Hz, 1H), 7.45 (d, J = 7.5 Hz, 1H), 7.33-7.21 (m, 3H), 6.68 (s, 1H), 6.54 (d, J = 15.9 Hz, 1H), 5.65 (dd, J = 7.2, 4.7 Hz, 1H), 4.34 (q, J = 7.1 Hz, 2H), 4.19 (q, J = 6.9 Hz, 2H), 3.18 (ABq, J = 16.4, 4.6 Hz, 1H), 2.77 (ABq, J = 16.3, 7.7 Hz, 1H), 1.40 (t, J = 7.1 Hz, 3H), 1.21 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 170.5, 166.8, 146.5, 142.4, 140.3, 135.1, 131.8, 131.7, 129.2, 128.1, 126.0, 125.2, 124.7, 124.5, 120.7, 113.4, 111.0, 95.2, 61.4, 60.9, 56.7, 39.6, 14.5, 14.2; IR (ATR): 2979, 1723, 1635, 1462, 1166, 1030, 861, 752 cm^{-1} . EI-MS m/z (%): 467.3 (55) $[\text{M}]^+$, 380.2 (66) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$, 302.2 (32) $[\text{M}-\text{CH}_3\text{CO}_2\text{Et}-\text{Br}]^+$. HRMS (ESI): found: 490.0624, calcd. for $\text{C}_{24}\text{H}_{22}\text{BrNO}_4\text{Na}$ ($[\text{M}+\text{Na}]^+$): 490.0630. Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{BrNO}_4$: C, 61.55; H, 4.73; N, 2.99; found: C, 62.21; H, 4.31; N, 3.43.



(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-2-nitro-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3n**): yield: 36%, yellow semi-solid. ^1H NMR (CDCl_3 , 300 MHz): δ 8.60 (d, J = 2.0 Hz, 1H), 8.22 (d, J = 15.9 Hz, 1H), 8.11 (dd, J = 9.0, 2.1 Hz, 1H), 7.66 (d, J = 7.7 Hz, 1H), 7.50 (d, J = 7.5 Hz, 1H), 7.41-7.36 (m, 2H), 6.89 (s, 1H), 6.57 (d, J = 15.9 Hz, 1H), 5.76 (dd, J = 7.0, 5.1 Hz, 1H), 4.35 (q, J = 7.1 Hz, 2H), 4.19 (q, J = 7.1 Hz, 2H), 3.21 (ABq, J = 16.5, 4.7 Hz, 1H), 2.88 (ABq, J = 16.5, 7.4 Hz, 1H), 1.40 (t, J = 7.1 Hz, 3H), 1.20 (t, J = 7.1 Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 170.1, 166.5, 146.1, 144.4, 141.9, 139.7, 135.8, 132.6, 130.6, 129.7, 128.8, 126.2, 124.7, 121.2, 118.9, 118.0, 109.3, 97.6, 61.5, 61.0, 56.9, 39.4, 14.4, 14.1; IR (ATR): 2981, 1718, 1516, 1319, 1167, 1029, 805, 746 cm^{-1} . EI-MS m/z (%): 434.4 (68) $[\text{M}]^+$, 347.2 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 457.1370, calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_6\text{Na}$ ($[\text{M}+\text{Na}]^+$): 457.1376. Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_6$: C, 66.35; H, 5.10; N, 6.45; found: C, 67.12; H, 4.74; N, 6.81.

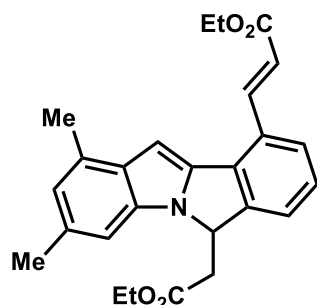


Ethyl

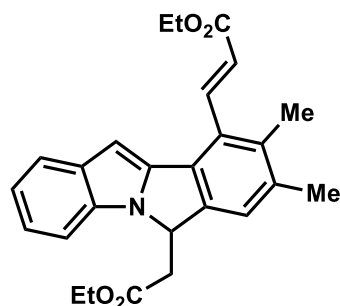
(*E*)-3-(6-(2-ethoxy-2-oxoethyl)-2-methoxy-6H-benzo[5,6]isoindolo[2,1-a]indol-12-yl)acrylate (**3o**) and ethyl

(*E*)-3-(13-(2-ethoxy-2-oxoethyl)-9-methoxy-13H-benzo[4,5]isoindolo[2,1-a]indol-6-yl)acrylate (**3o'**): isolated as an inseparable mixture (ratio = 1 : 1.7), yield: 53%, brown semi-solid. ^1H NMR (CDCl_3 , 400 MHz): δ 8.41 (d, J = 16.4 Hz, 1H), 8.35 (d, J = 16.0 Hz, 1.7H), 8.19 (d, J = 8.4 Hz, 1H), 8.06 (s, 1.6H), 7.89 (d, J = 8.0 Hz, 1.7H), 7.83 (d, J = 8.4 Hz, 2H), 7.78 (d, J = 8.4 Hz, 1.8H), 7.58-7.46 (m, 6H), 7.28 (d, J = 8.8 Hz, 2.5H), 7.15 (d, J = 2.0 Hz, 2.5H), 6.91-6.86 (m, 2.7H), 6.70 (s, 2.3H), 6.66 (d, J = 10.8 Hz, 1.7H), 6.63 (d, J = 16.4 Hz, 0.8H), 6.07 (dd, J = 6.8, 3.2 Hz, 1.7H), 5.83 (dd, J = 8.4, 4.8 Hz, 1H), 4.44-4.34 (m, 5.4H), 4.24 (qd, J = 7.2, 2.4 Hz, 2.5H), 4.16 (qd, J = 7.2, 2.4 Hz, 3.5H), 3.88-3.87 (m, 6.9H), 3.35-3.24 (m, 3H), 2.84-2.76 (m, 2.9H), 1.47-1.41 (m, 7.7H), 1.24 (t, J = 7.2 Hz, 3.6H), 1.14 (t, J = 7.2 Hz, 5H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 171.4, 170.8, 166.8, 154.6, 154.4, 143.5, 142.8, 141.7, 141.0, 140.6, 132.9, 130.1, 128.4, 127.4, 127.3, 127.0, 126.4, 126.3, 122.8, 120.9, 113.3, 112.6, 110.7, 110.5, 103.8, 103.5, 96.5, 95.4, 61.5, 61.3, 61.0, 60.9, 56.9, 56.1, 56.0, 40.7, 40.2, 14.6, 14.3, 14.1; IR (ATR):

2982, 1714, 1479, 1166, 1031, 856, 751 cm^{-1} . EI-MS m/z (%): 469.3 (63) $[\text{M}]^+$, 382.2 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 470.1962, calcd. for $\text{C}_{29}\text{H}_{28}\text{NO}_5$ ($[\text{M}+\text{H}]^+$): 470.1967. Anal. Calcd. for $\text{C}_{29}\text{H}_{27}\text{NO}_5$: C, 74.18; H, 5.80; N, 2.98; found: C, 74.86; H, 5.24; N, 3.45.

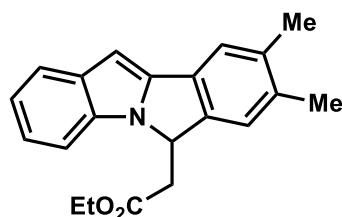


(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-1,3-dimethyl-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3p**): yield: 90%, yellow solid, melting point: 94-96 $^{\circ}\text{C}$. ^1H NMR (CDCl_3 , 600 MHz): δ 8.34 (d, J = 16.2 Hz, 1H), 7.61 (d, J = 7.8 Hz, 1H), 7.44 (d, J = 7.8 Hz, 1H), 7.26 (t, J = 7.2 Hz, 1H), 7.01 (s, 1H), 6.80 (s, 1H), 6.72 (s, 1H), 6.56 (d, J = 15.6 Hz, 1H), 5.66 (dd, J = 8.4, 4.2 Hz, 1H), 4.35 (q, J = 7.2 Hz, 2H), 4.26-4.21 (m, 2H), 3.30 (ABq, J = 16.2, 4.2 Hz, 1H), 2.70 (ABq, J = 16.8, 8.4 Hz, 1H), 2.58 (s, 3H), 2.47 (s, 3H), 1.42 (t, J = 7.2 Hz, 3H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 170.8, 167.0, 146.4, 140.9, 140.0, 133.3, 132.7, 132.6, 131.5, 131.2, 128.6, 127.2, 125.8, 124.7, 122.4, 120.1, 107.2, 94.3, 61.2, 60.8, 56.4, 39.5, 22.0, 18.9, 14.5, 14.2; IR (ATR): 2979, 1714, 1632, 1421, 1166, 1031, 975, 754 cm^{-1} . EI-MS m/z (%): 417.2 (100) $[\text{M}]^+$, 330.1 (71) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 418.2013, calcd. for $\text{C}_{26}\text{H}_{28}\text{NO}_4$ ($[\text{M}+\text{H}]^+$): 418.2018. Anal. Calcd. for $\text{C}_{26}\text{H}_{27}\text{NO}_4$: C, 74.80; H, 6.52; N, 3.35; found: C, 75.23; H, 5.94; N, 6.96.

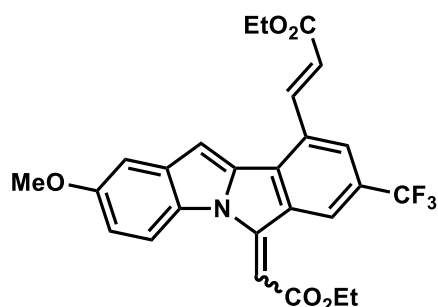


(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-8,9-dimethyl-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3q**): yield: 60%, yellowish-brown oil. ^1H NMR (CDCl_3 , 300 MHz): δ 8.16 (d, J = 15.9 Hz, 1H), 7.67 (d, J = 7.8 Hz, 1H), 7.31-7.26 (m, 2H), 7.18-7.12 (m, 2H), 6.62 (s, 1H), 6.44 (d, J = 15.9 Hz, 1H), 5.45 (dd, J = 5.4, 3.3 Hz, 1H), 4.35 (q, J = 7.2 Hz, 2H), 4.04 (q, J = 7.2 Hz, 2H), 3.04 (ABq, J = 16.5, 3.0 Hz, 1H), 2.69 (ABq, J = 16.5, 6.0 Hz, 1H), 2.25 (s, 3H), 2.19 (s, 3H), 1.43 (t, J = 7.2 Hz, 3H), 1.06 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 170.9, 167.1, 144.9, 141.8, 140.8, 136.9, 133.8, 133.5, 133.1, 130.4, 127.1, 126.3, 122.1, 120.0, 119.1, 109.9, 95.2, 61.2, 60.7, 56.5, 39.1, 20.0, 16.1, 14.5, 14.0; IR (ATR): 2979, 1710, 1634, 1452, 1164, 1036, 860, 741 cm^{-1} . EI-MS m/z (%): 417.1 (59) $[\text{M}]^+$, 330.1 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 440.1832,

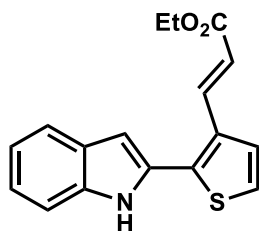
calcd. for $C_{26}H_{27}NO_4Na$ ($[M+Na]^+$): 440.1838. Anal. Calcd. for $C_{26}H_{27}NO_4$: C, 74.80; H, 6.52; N, 3.35; found: C, 75.31; H, 6.12; N, 6.83.



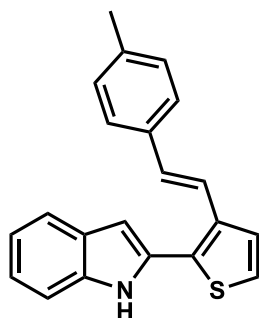
ethyl 2-(8,9-dimethyl-6H-isoindolo[2,1-a]indol-6-yl)acetate (**3q'**): yield: 25%, yellow oil. 1H NMR ($CDCl_3$, 600 MHz): δ 7.66-7.63 (m, 1H), 7.48 (s, 1H), 7.35 (d, J = 8.1 Hz, 1H), 7.23 (s, 1H), 7.19-7.14 (m, 1H), 7.12-7.07 (m, 1H), 6.55 (s, 1H), 5.68 (dd, J = 8.1, 1.5 Hz, 1H), 4.30-4.20 (m, 2H), 3.27 (ABq, J = 16.2, 4.8 Hz, 1H), 2.72 (ABq, J = 16.2, 8.1 Hz, 1H), 2.35 (s, 3H), 2.32 (s, 3H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 171.1, 143.7, 143.4, 137.3, 136.3, 133.5, 133.3, 130.1, 124.6, 122.0, 121.7, 121.5, 119.7, 109.5, 61.2, 56.7, 39.9, 20.4, 20.2, 14.3; IR (ATR): 2923, 1725, 1612, 1454, 1153, 1014, 868, 739 cm^{-1} . EI-MS m/z (%): 319.1 (60) $[M]^+$, 231.9 (100) $[M-CH_2CO_2Et]^+$. HRMS (ESI): found: 342.1465, calcd. for $C_{21}H_{21}NO_2Na$ ($[M+Na]^+$): 342.1470. Anal. Calcd. for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.63; N, 4.39; found: C, 79.37; H, 6.32; N, 4.81.



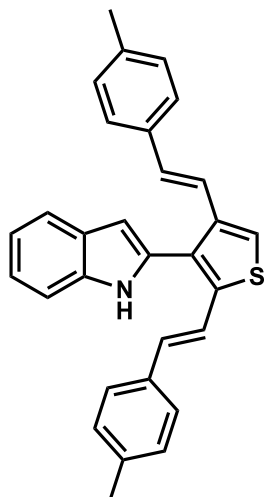
(2E)-ethyl 3-(6-(2-ethoxy-2-oxoethylidene)-2-methoxy-8-(trifluoromethyl)-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3r**): yield: 58%, yellow solid, melting point: 154-156 $^{\circ}C$. 1H NMR ($CDCl_3$, 600 MHz): δ 9.38 (s, 1H), 7.90 (d, J = 16.2 Hz, 1H), 7.73 (s, 1H), 7.38 (d, J = 9.0 Hz, 1H), 6.95 (d, J = 2.4 Hz, 1H), 6.86 (dd, J = 9.0, 2.4 Hz, 1H), 6.58 (s, 1H), 6.50 (d, J = 15.6 Hz, 1H), 6.20 (s, 1H), 4.33 (q, J = 7.2 Hz, 2H), 4.28 (q, J = 7.2 Hz, 2H), 3.84 (s, 3H), 1.39 (q, J = 7.2 Hz, 6H); ^{19}F NMR ($CDCl_3$, 576 MHz) δ -62.5 (s); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 166.2, 166.2, 156.1, 144.9, 139.1, 138.3, 136.9, 136.0, 134.3, 130.4 (q, J_F = 32.4), 128.4, 127.3 (q, J_F = 3.6), 125.0 (q, J_F = 3.6), 123.9 (q, J_F = 271.2), 122.0, 114.8, 112.8, 106.8, 103.5, 99.4, 61.1, 60.7, 55.7, 14.5; IR (ATR): 2926, 1708, 1631, 1477, 1296, 1034, 966, 816 cm^{-1} . EI-MS m/z (%): 485.5 (4) $[M]^+$. HRMS (ESI): found: 486.1523, calcd. for $C_{26}H_{23}F_3NO_5$ ($[M+H]^+$): 486.1528. Anal. Calcd. for $C_{26}H_{22}F_3NO_5$: C, 64.33; H, 4.57; N, 2.89; found: C, 64.86; H, 4.35; N, 3.24.



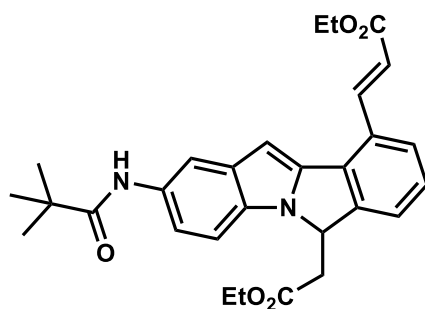
(*E*)-ethyl 3-(2-(1H-indol-2-yl)thiophen-3-yl)acrylate (**3s**): yield: 76%, yellow semi-solid. ^1H NMR (CDCl_3 , 600 MHz): δ 8.32 (s, 1H), 7.97 (d, J = 16.2 Hz, 1H), 7.66 (d, J = 7.8 Hz, 1H), 7.41 (dd, J = 8.4, 0.6 Hz, 1H), 7.33 (d, J = 5.4 Hz, 1H), 7.30 (dd, J = 5.4, 0.6 Hz, 1H), 7.25 (td, J = 7.2, 1.2 Hz, 1H), 7.16 (td, J = 7.8, 0.6 Hz, 1H), 6.73 (d, J = 1.8 Hz, 1H), 6.37 (d, J = 15.6 Hz, 1H), 4.26 (q, J = 7.2 Hz, 2H), 1.33 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 167.4, 137.1, 136.8, 133.8, 129.9, 129.0, 126.6, 125.6, 123.3, 121.1, 120.8, 119.5, 111.2, 105.1, 60.7, 14.5; IR (ATR): 3327, 2981, 1691, 1620, 1303, 1173, 1035, 862, 746 cm^{-1} . EI-MS m/z (%): 297.1 (25) $[\text{M}]^+$, 224.0 (100) $[\text{M}-\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 298.0896, calcd. for $\text{C}_{17}\text{H}_{16}\text{SNO}_2$ ($[\text{M}+\text{H}]^+$): 298.0902. Anal. Calcd. for $\text{C}_{17}\text{H}_{15}\text{SNO}_2$: C, 68.66; H, 5.08; N, 4.71; found: C, 69.15; H, 4.75; N, 4.54.



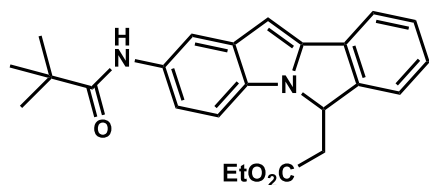
(*E*)-2-(3-(4-methylstyryl)thiophen-2-yl)-1H-indole (**3t**): yield: 54%, yellow-brown solid, melting point: 92-94 $^{\circ}\text{C}$. ^1H NMR (CDCl_3 , 600 MHz): δ 8.21 (s, 1H), 7.68 (d, J = 7.8 Hz, 1H), 7.42-7.39 (m, 4H), 7.34 (d, J = 16.2 Hz, 1H), 7.31 (d, J = 5.4 Hz, 1H), 7.25 (td, J = 7.8, 1.2 Hz, 1H), 7.19-7.17 (m, 3H), 7.05 (d, J = 16.2 Hz, 1H), 6.73-6.72 (m, 1H), 2.38 (s, 3H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 137.8, 136.9, 136.8, 134.6, 131.3, 130.7, 130.6, 129.6, 129.1, 126.6, 125.1, 122.8, 121.1, 120.9, 120.6, 111.1, 103.9, 21.4; IR (ATR): 3388, 2921, 1606, 1513, 1453, 1304, 1092, 800, 745 cm^{-1} . EI-MS m/z (%): 315.1 (100) $[\text{M}]^+$, 224.0 (54) $[\text{M}-\text{PhCH}_2]^+$. HRMS (ESI): found: 316.1155, calcd. for $\text{C}_{21}\text{H}_{18}\text{NS}$ ($[\text{M}+\text{H}]^+$): 316.1160. Anal. Calcd. for $\text{C}_{21}\text{H}_{17}\text{NS}$: C, 79.96; H, 5.43; N, 4.44; found: C, 80.52; H, 4.82; N, 4.84.



2-(2,4-bis(4-methylstyryl)thiophen-3-yl)-1H-indole (**3u**): yield: 56%, yellow solid, melting point: 212-214 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.09 (s, 1H), 7.71 (d, J = 7.6 Hz, 1H), 7.37 (d, J = 7.2 Hz, 1H), 7.29-7.17 (m, 8H), 7.09 (dd, J = 7.6, 2.8 Hz, 4H), 6.98 (d, J = 16.0 Hz, 1H), 6.91 (s, 2H), 6.61 (dd, J = 6.0, 0.8 Hz, 1H), 2.32 (s, 6H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 141.6, 140.3, 138.0, 137.9, 136.5, 134.4, 134.1, 131.7, 130.5, 129.9, 129.6, 129.5, 128.8, 126.6, 126.6, 122.4, 121.1, 120.9, 120.3, 120.2, 111.1, 105.2, 21.4, 21.4; IR (ATR): 3359, 2920, 1603, 1509, 1450, 1300, 1104, 957, 801, 713 cm^{-1} . EI-MS m/z (%): 431.2 (100) $[\text{M}]^+$, 340.1 (73) $[\text{M}-\text{PhCH}_2]^+$. HRMS (ESI): found: 432.1781, calcd. for $\text{C}_{30}\text{H}_{26}\text{NS}$ ($[\text{M}+\text{H}]^+$): 432.1786. Anal. Calcd. for $\text{C}_{30}\text{H}_{25}\text{NS}$: C, 83.49; H, 5.84; N, 3.25; found: C, 84.04; H, 5.35; N, 3.70.



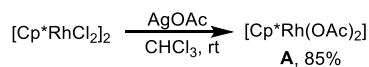
(*E*)-ethyl 3-(6-(2-ethoxy-2-oxoethyl)-2-pivalamido-6H-isoindolo[2,1-a]indol-10-yl)acrylate (**3v**): yield: 74%, yellow semi-solid. ^1H NMR (CDCl_3 , 400 MHz): δ 8.24 (d, J = 16.0 Hz, 1H), 7.80 (d, J = 2.0 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H), 7.44 (s, 1H), 7.42 (d, J = 7.6 Hz, 1H), 7.33 (dd, J = 8.8, 2.0 Hz, 1H), 7.29-7.25 (m, 2H), 6.67 (s, 1H), 6.53 (d, J = 15.6 Hz, 1H), 5.63 (dd, J = 8.0, 4.8 Hz, 1H), 4.33 (q, J = 7.2 Hz, 2H), 4.22-4.14 (m, 2H), 3.19 (ABq, J = 16.4, 4.8 Hz, 1H), 2.73 (ABq, J = 16.4, 8.0 Hz, 1H), 1.40 (t, J = 7.2 Hz, 3H), 1.35 (s, 9H), 1.21 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.7, 170.6, 166.8, 146.5, 142.0, 140.5, 133.6, 132.1, 131.1, 130.6, 128.9, 127.6, 125.8, 124.6, 120.4, 117.1, 114.0, 109.6, 95.8, 61.3, 60.8, 56.6, 39.6, 39.6, 27.9, 14.5, 14.2; IR (ATR): 3346, 2967, 1714, 1657, 1476, 1165, 1030, 866, 727 cm^{-1} . ESI-MS m/z (%): 489.2 (100) $[\text{M}+\text{H}]^+$. HRMS (ESI): found: 511.2203, calcd. for $\text{C}_{29}\text{H}_{32}\text{N}_2\text{O}_5\text{Na}$ ($[\text{M}+\text{Na}]^+$): 511.2209. Anal. Calcd. for $\text{C}_{29}\text{H}_{32}\text{N}_2\text{O}_5$: C, 71.29; H, 6.60; N, 5.73; found: C, 72.01; H, 6.36; N, 5.99.



ethyl 2-(2-pivalamido-6H-isoindolo[2,1-a]indol-6-yl)acetate (**3v'**): yield: 20%, yellow oil. ^1H NMR (CDCl_3 , 400 MHz): δ 7.89 (d, J = 2.0 Hz, 1H), 7.67 (d, J = 7.6 Hz, 1H), 7.45 (dd, J = 7.6, 0.8 Hz, 1H), 7.40 (t, J = 7.6 Hz, 1H), 7.36 (s, 1H), 7.31-7.27 (m, 2H), 7.22 (dd, J = 8.4, 2.0 Hz, 1H), 6.57 (s, 1H), 5.72 (dd, J = 7.6, 4.8 Hz, 1H), 4.26-4.18 (m, 2H), 3.22 (ABq, J = 16.4, 4.8 Hz, 1H), 2.76 (ABq, J = 16.4, 7.6 Hz, 1H), 1.35 (s, 9H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.6, 170.9, 145.7, 143.9, 133.6, 132.3, 130.8, 128.8, 127.5, 123.5, 121.0, 116.1, 113.9, 109.6, 92.1, 61.3, 57.0, 39.8, 27.9, 14.3; IR (ATR): 3341, 2964, 1728, 1656, 1476, 1178, 1023, 870, 752 cm^{-1} . EI-MS m/z (%): 390.4 (95) $[\text{M}]^+$, 303.3 (100) $[\text{M}-\text{CH}_2\text{CO}_2\text{Et}]^+$. HRMS (ESI): found: 413.1836, calcd. for $\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_3\text{Na}$ ($[\text{M}+\text{Na}]^+$): 413.1841. Anal. Calcd. for $\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_3$: C, 73.82; H, 6.71; N, 7.17; found: C, 74.35; H, 6.31; N, 6.87.

Mechanism study

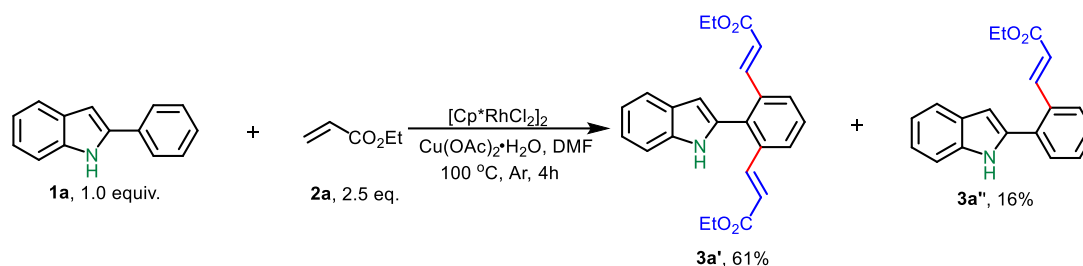
(a) Synthesis of active Rh catalyst species



Active catalytic Rh species **A** was synthesized according to a previous reported literature.¹⁴ $[\text{Cp}^*\text{RhCl}_2]_2$ (750 mg, 1.0 equiv.) and AgOAc (810 mg, 4.0 equiv.) were weighed into an oven dried Schlenk tube. The reaction vessel was capped and vacuum/backfilled with argon three times. To this vessel, 20 mL CHCl_3 was added. The reaction was stirred at rt for two hours. The reaction mixture was filtered through a plug of celite and washed with *n*-hexane. The filtrate was concentrated by rotary evaporation and the residue yellow solid was collected as the desired catalyst (in case there is residual solvents, drying under high vacuum pump overnight might be necessary).

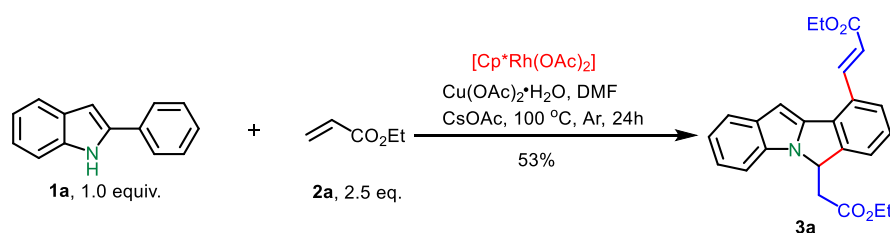
(b) Catalytic reactions

(i) In the absence of base



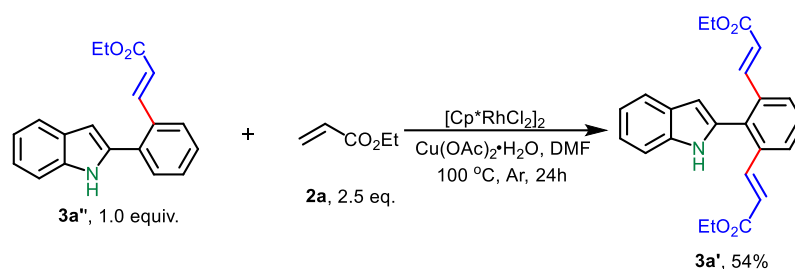
Following the **General Procedure for Rh(III)-Catalyzed C–H Activation**, reaction is conducted on a 0.2 mmol scale, using **1a** (1.0 equiv.), ethyl acrylate **2a** (2.5 equiv.), $[\text{Cp}^*\text{RhCl}_2]_2$ (5 mol%), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (2.0 equiv.), DMF (1.5 mL).

(ii) $[\text{Cp}^*\text{Rh}(\text{OAc})_2]$ -catalyzed reaction



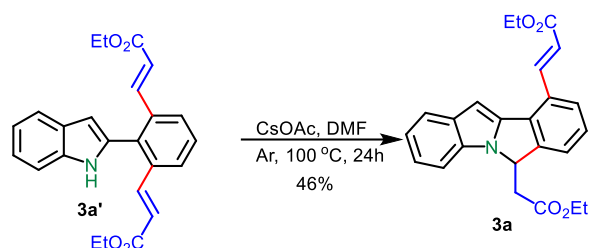
Following the **General Procedure for Rh(III)-Catalyzed C–H Activation**, reaction is conducted on a 0.2 mmol scale, using **1a** (1.0 equiv.), ethyl acrylate **2a** (2.5 equiv.), $[\text{Cp}^*\text{Rh}(\text{OAc})_2]$ (5 mol%), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (2.0 equiv.), DMF (1.5 mL).

(iii) Conversion of mono-alkenylation product to dialkenylation product



Following the **General Procedure for Rh(III)-Catalyzed C–H Activation**, reaction is conducted on a 0.2 mmol scale, using **1a** (1.0 equiv.), ethyl acrylate **2a** (2.5 equiv.), $[\text{Cp}^*\text{RhCl}_2]_2$ (5 mol%), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (2.0 equiv.), DMF (1.5 mL).

(c) Cyclization reactions



3a' (0.2 mmol, 77.9 mg, 1.0 equiv.) and CsOAc (0.4 mmol, 76.8 mg, 2.0 equiv.) were weighed into a Schlenk tube. The reaction vessel was capped and subjected to three vacuum-purge/argon-flush cycles. DMF (1.5 mL) was then added through the side-arm by syringe. The reaction was stirred under argon at 100°C for 24h. Volatile solvent and reagents were removed by rotary evaporation and the residue was purified by silica gel flash chromatography using *n*-Hexane/EtOAc (20:1 to 10:1) to afford product **3a**.

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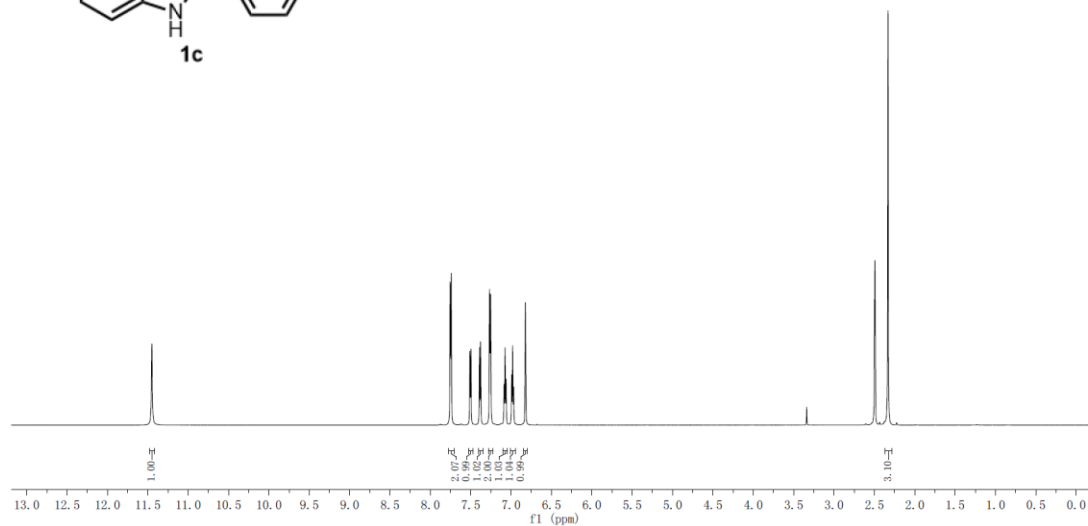
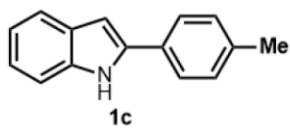


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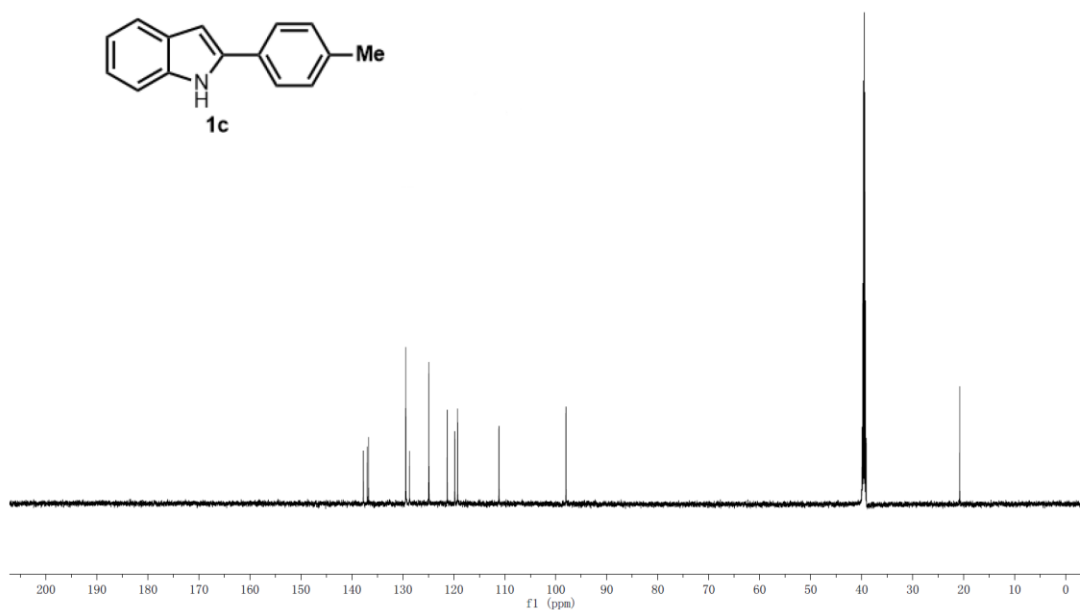
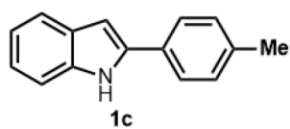


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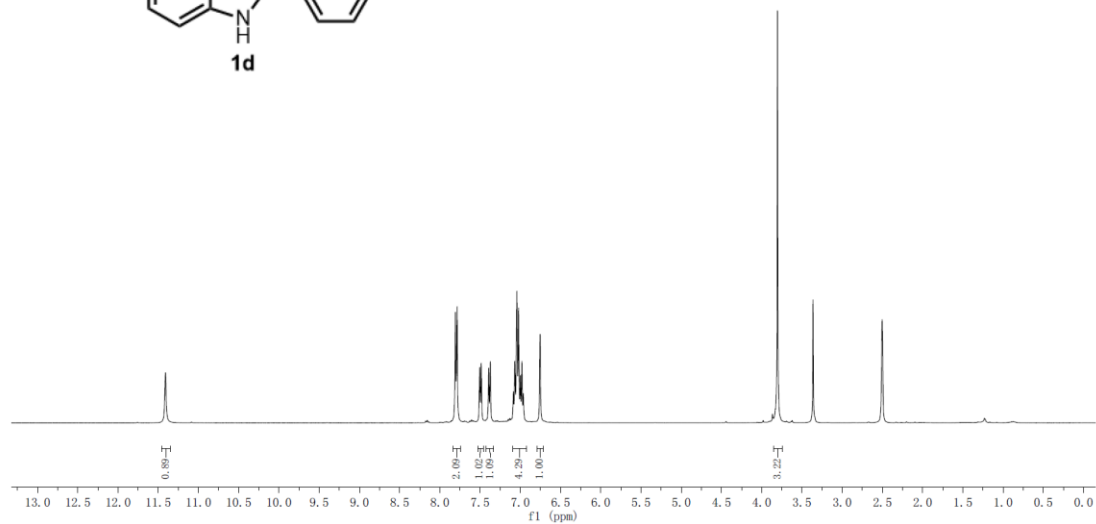
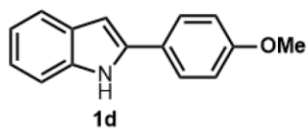


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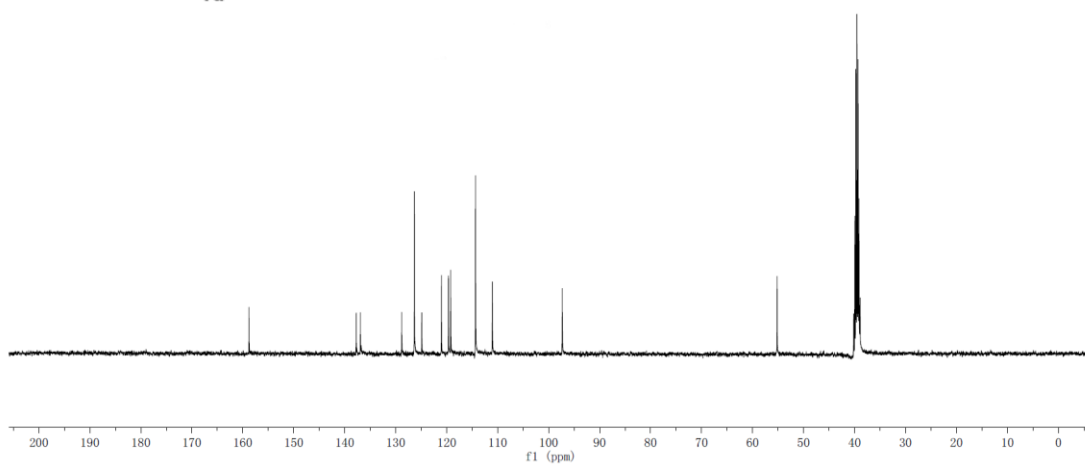
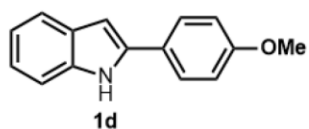
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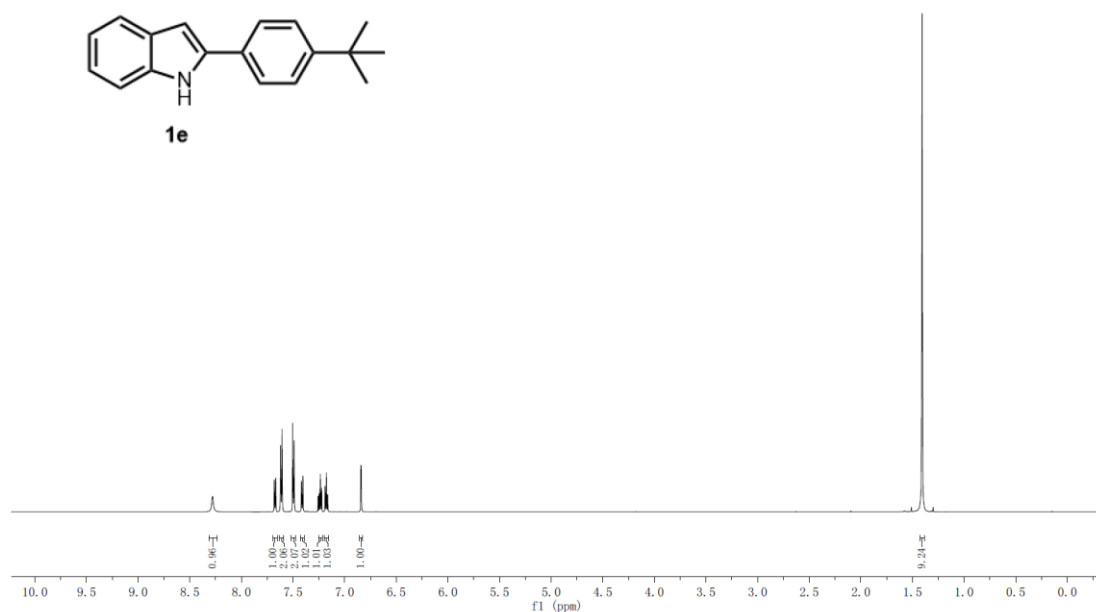
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r/Wang_CMW143

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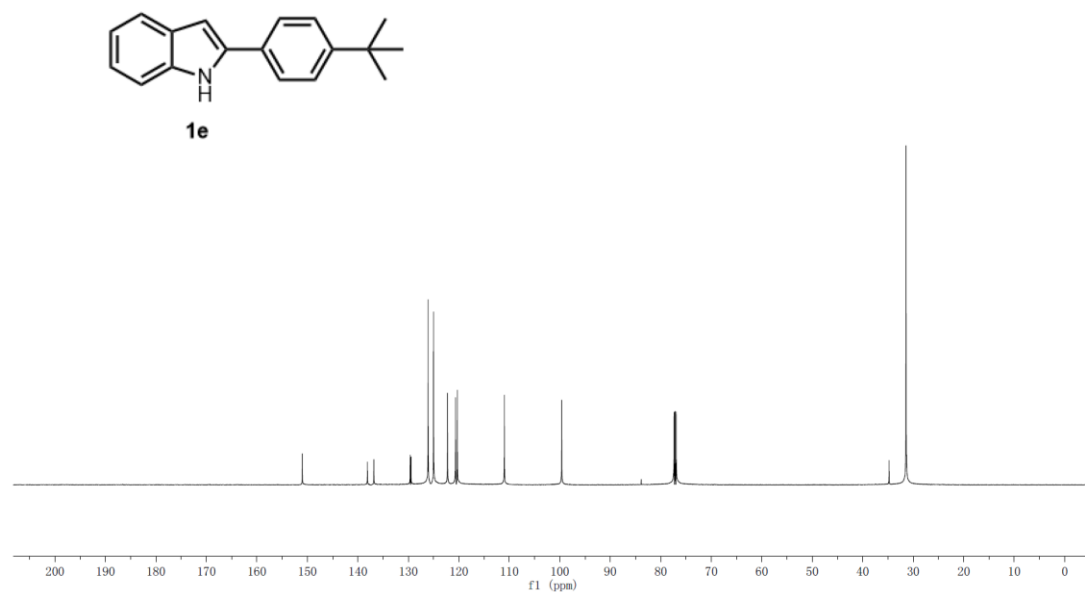
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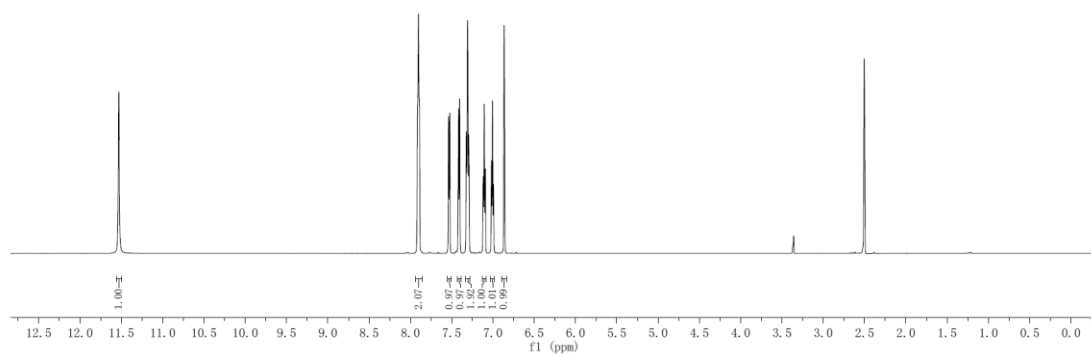
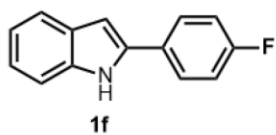
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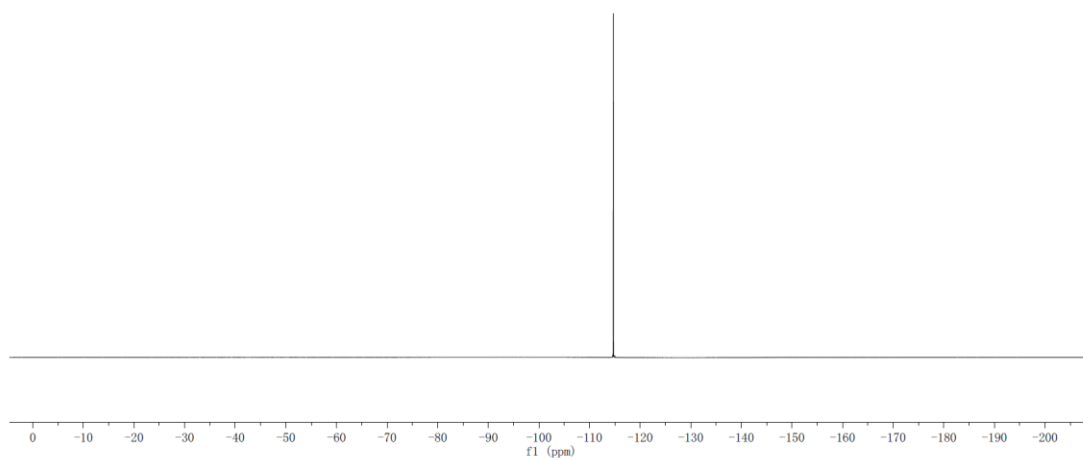
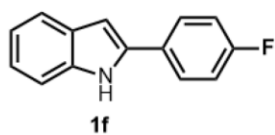
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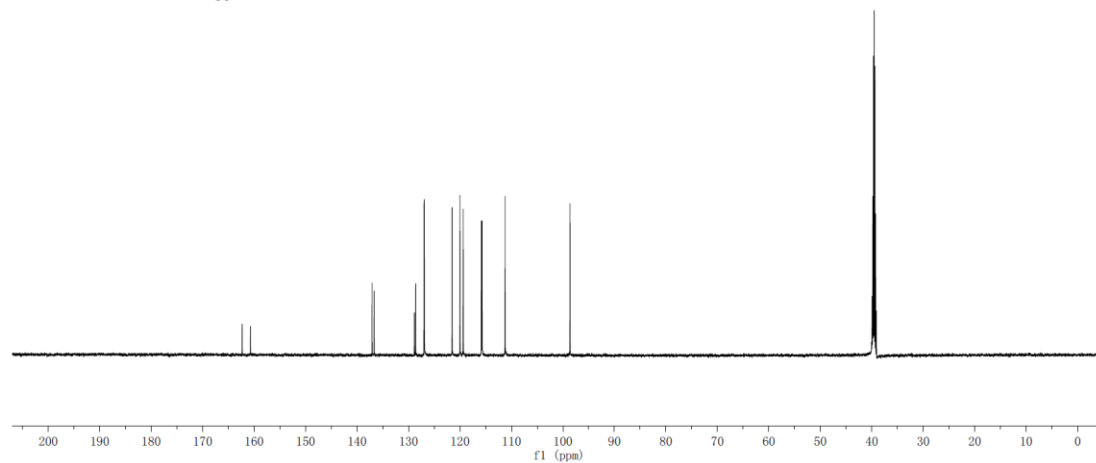
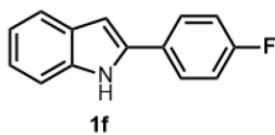
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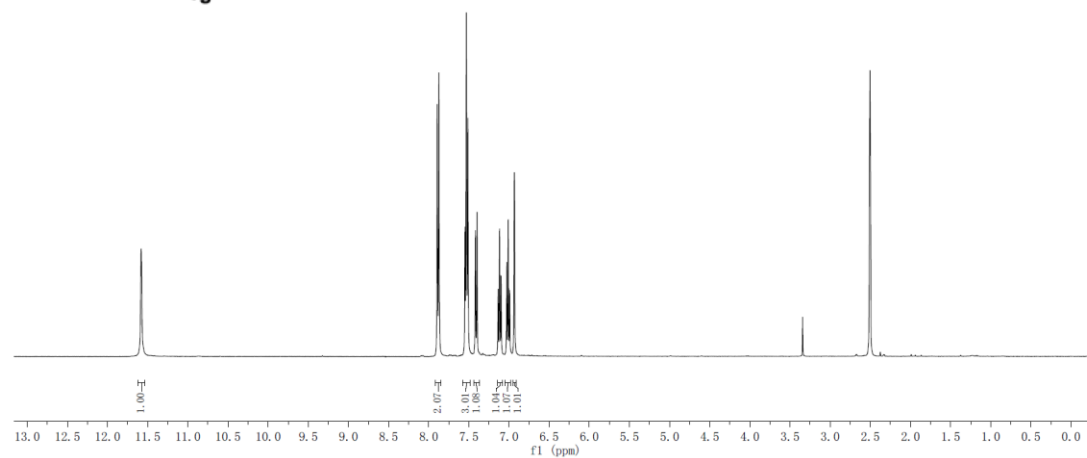
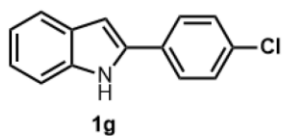
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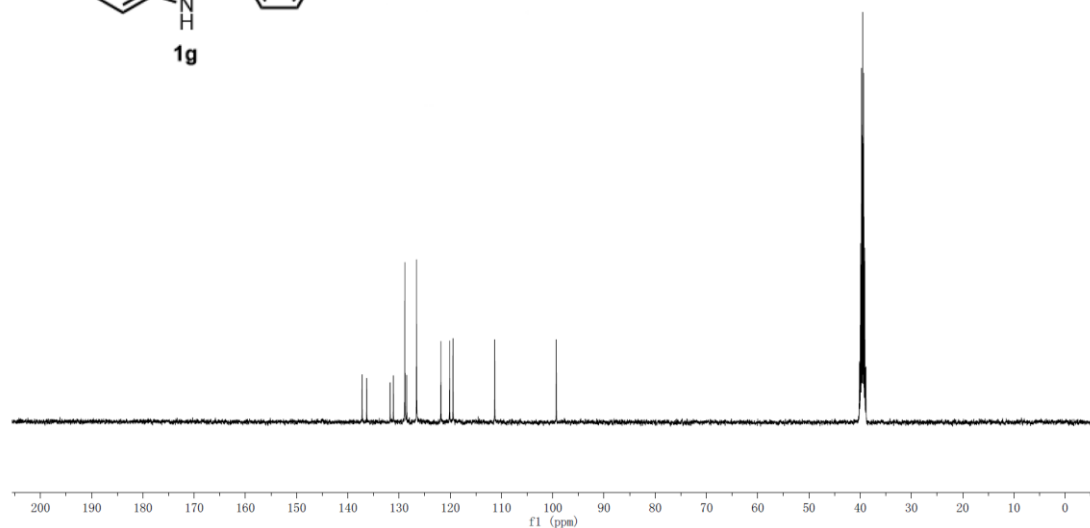
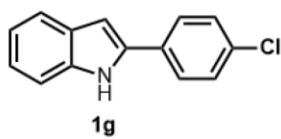
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CARBON_01
r/wang_cmw203

137.20
136.33
131.74
128.88
128.62
127.85
123.15
119.49
111.33
96.31

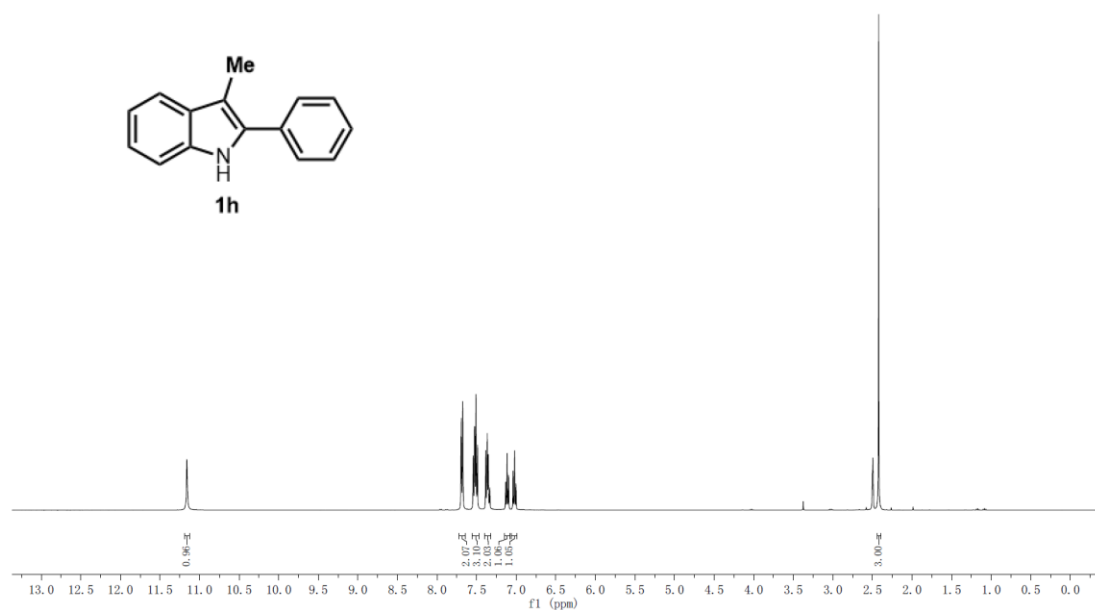
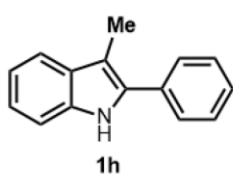


PROTON_01
r/Wang_CMW168

11.160

7.697
7.674
7.674
7.542
7.523
7.509
7.385
7.371
7.353
7.334
7.117
7.117
7.097
7.095
7.044
7.039
7.022
7.004
7.002

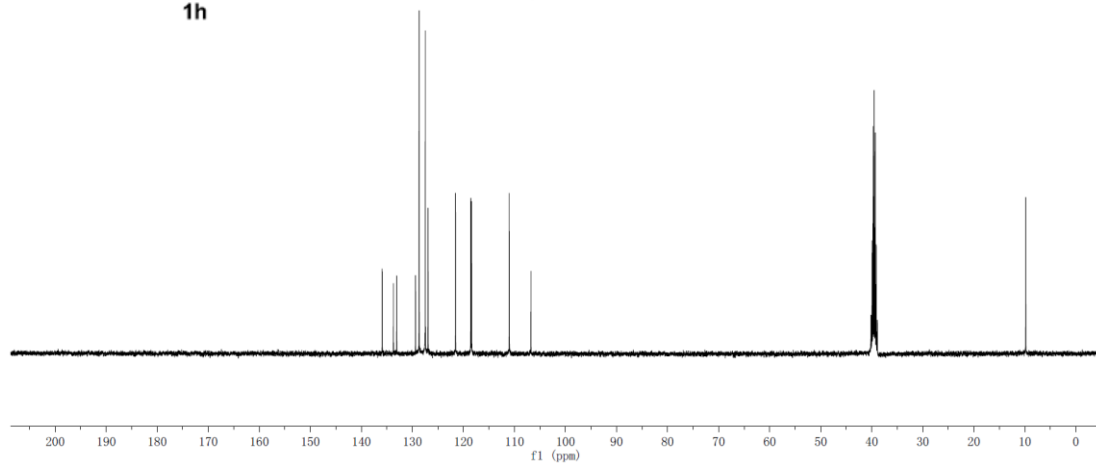
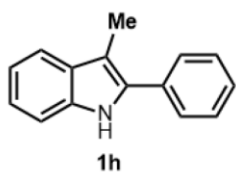
2.422



CARBON_01
r/Wang_CM#168

135.50
133.72
133.09
129.89
129.72
127.46
126.94
121.53
118.41
—111.01
—106.76

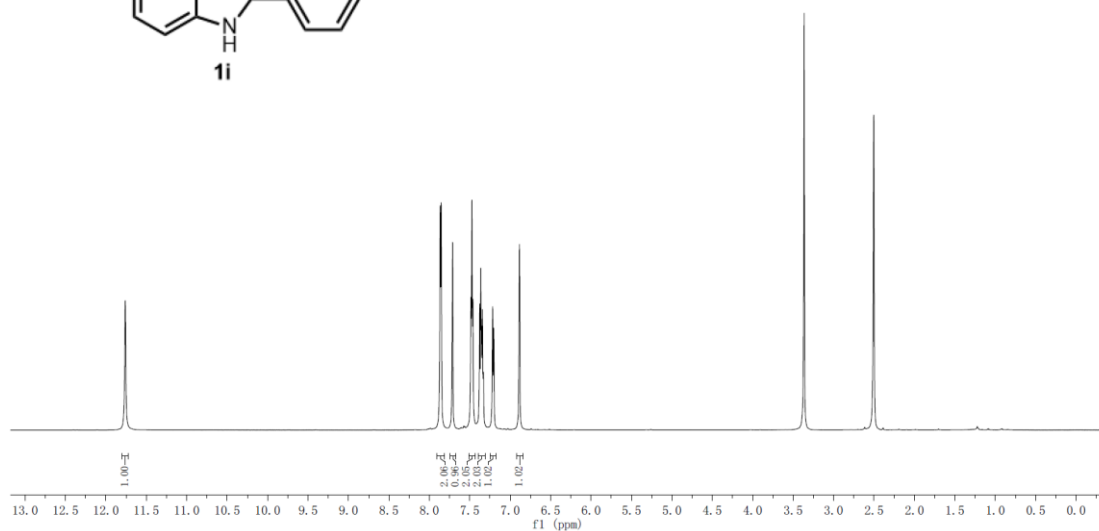
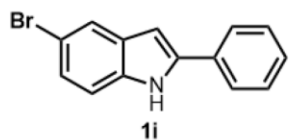
—9.84



PROTON_01
r/Wang_CM#151

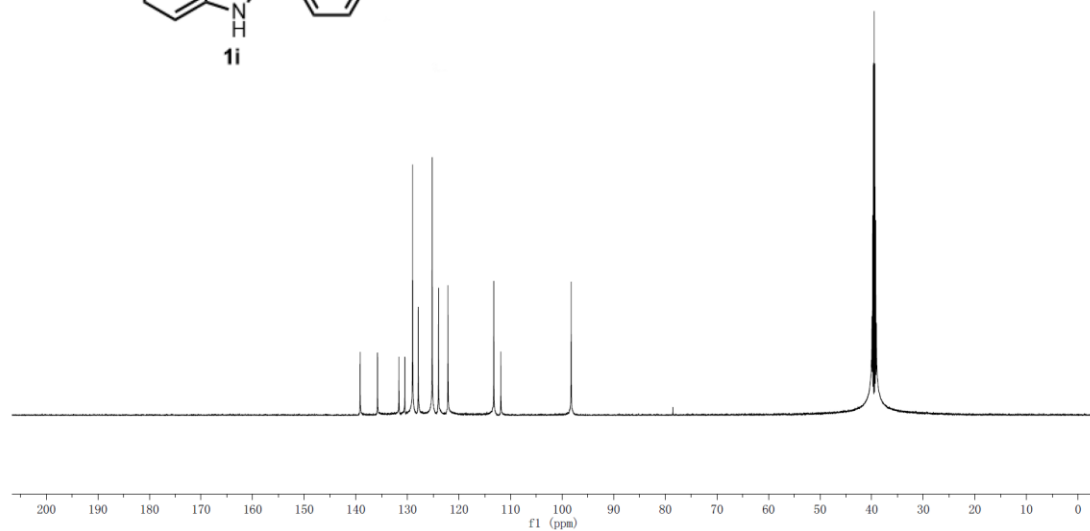
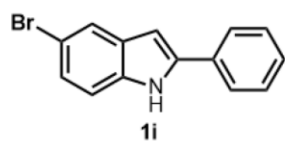
—11.760

7.665
7.653
7.713
7.645
7.693
7.461
7.377
7.356
7.343
7.331
7.203
—6.885



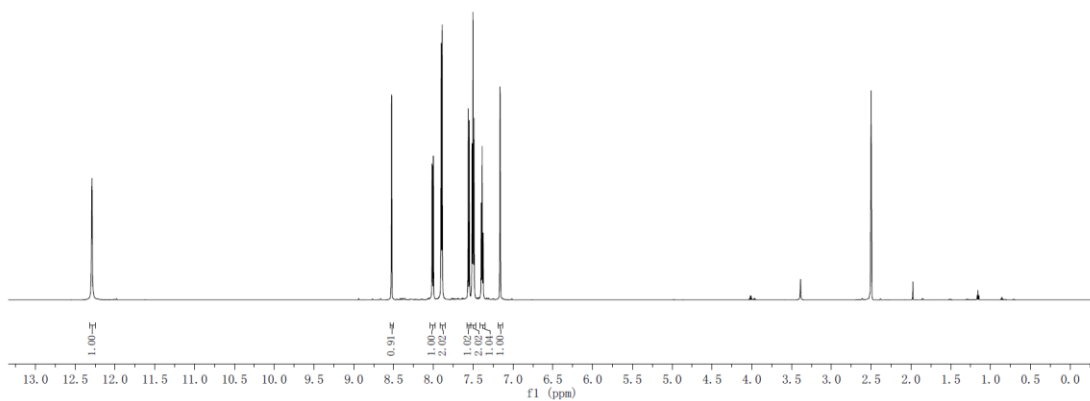
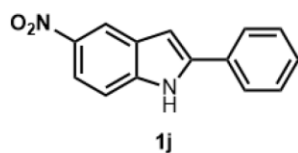
CARBON_01
r/Wang_CMW151

130.15
135.78
131.63
132.42
128.98
127.88
131.13
123.48
122.13
113.24
111.07
98.25



PROTON_01
r/Wang_CMW151

8.527
8.523
8.002
7.903
7.902
7.898
7.815
7.815
7.802
7.899
7.387
7.383
7.160



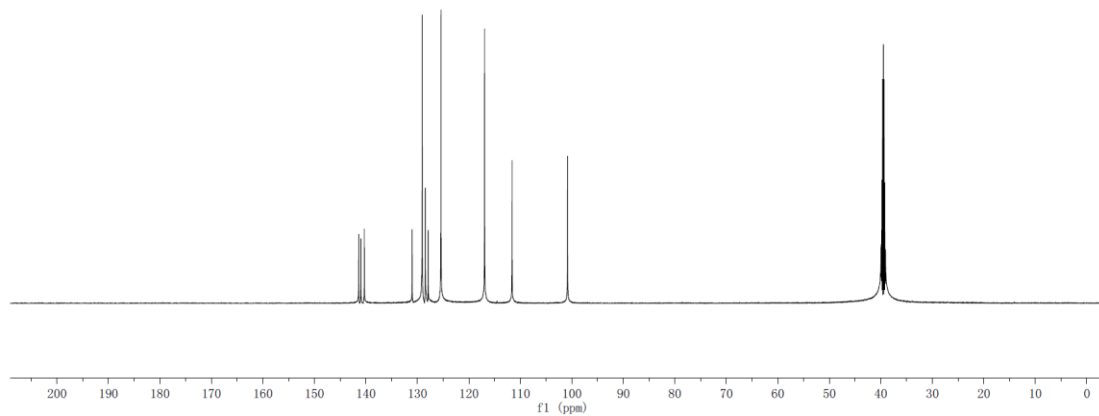
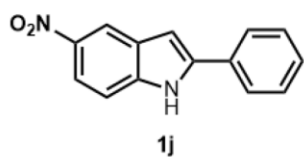
141.41
140.99
140.31

131.01
129.08
128.45
127.93
125.43

— 116.96

— 111.62

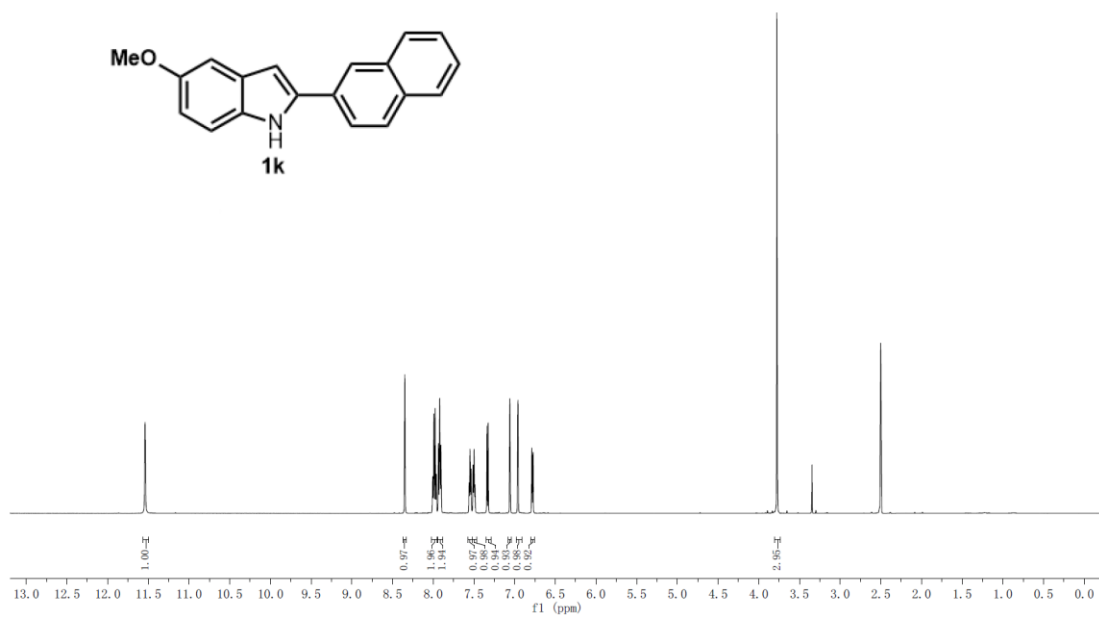
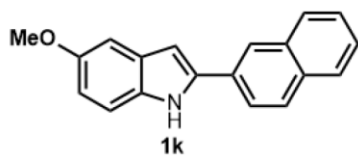
— 100.84



—11.540

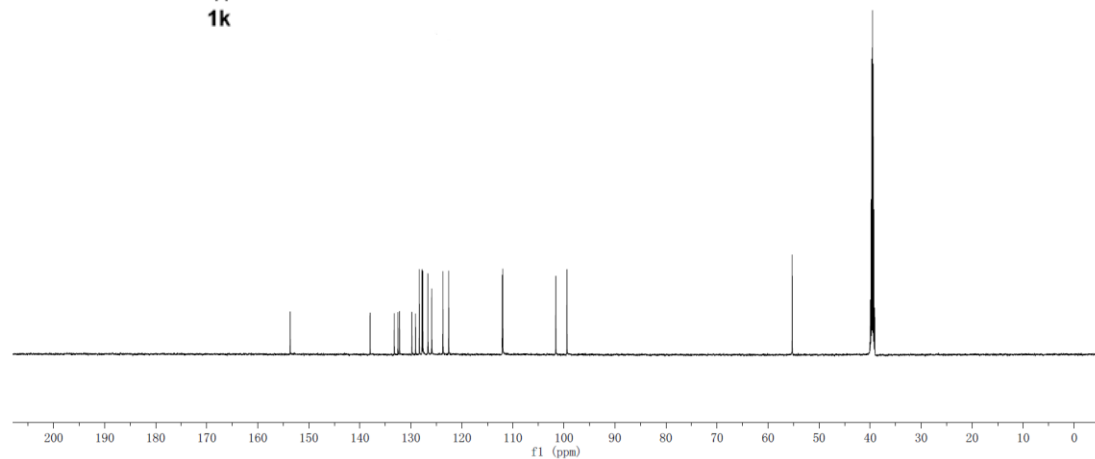
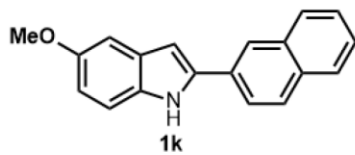
—8.349
7.992
7.976
7.936
7.921
7.939
7.548
7.534
7.511
7.498
7.486
7.339
7.325
7.057
6.959
6.789
6.787
6.775
6.772

—3.777



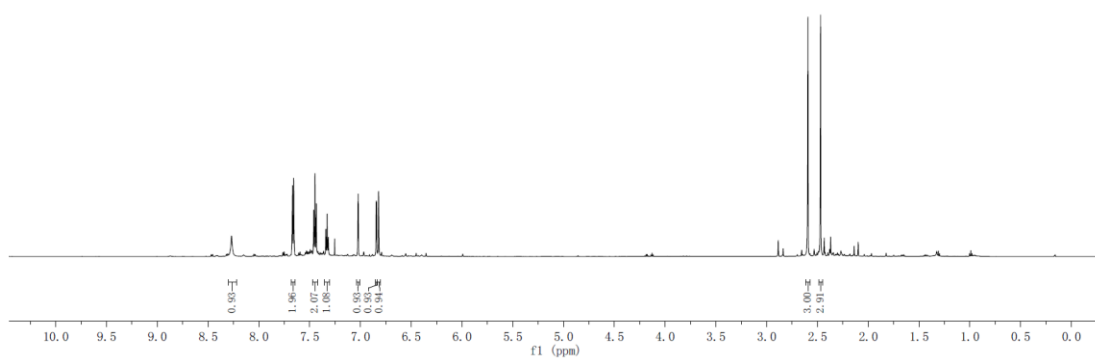
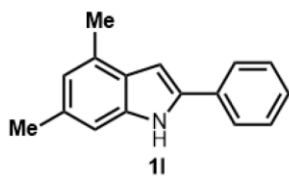
CARBON_01
r/Wang_CMW166

151.65
138.00
132.53
132.19
129.75
129.34
127.81
127.62
126.64
125.91
122.72
122.69
111.58
101.69
98.40
55.26



PROTON_01
r/Wang_CMW156

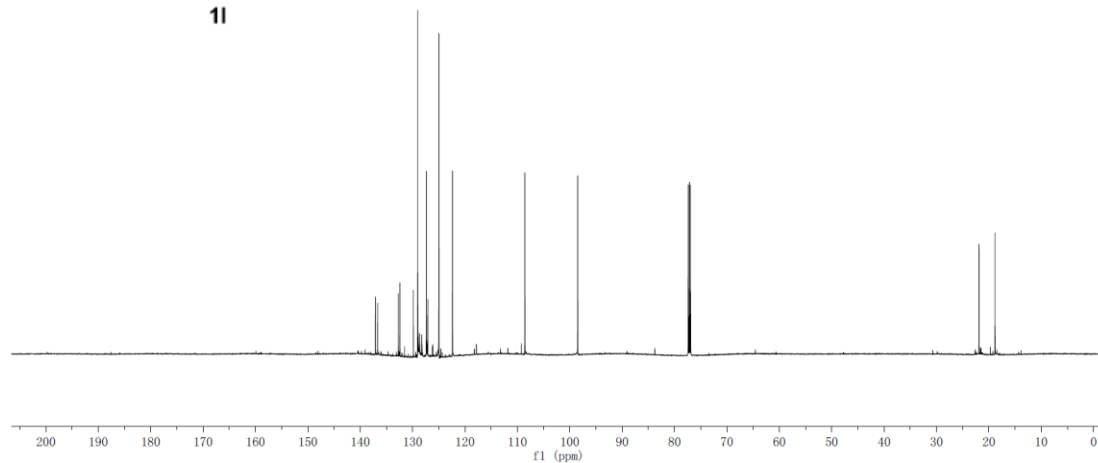
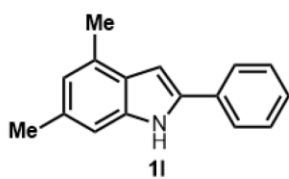
8.270
7.671
7.670
7.638
7.448
7.435
7.329
7.317
7.225
6.843
6.822
2.695
2.470



CARBON_01
r/Wang_CMW156

137.10
136.69
132.72
132.42
129.86
129.53
127.38
127.09
124.98
122.38
108.57
98.47

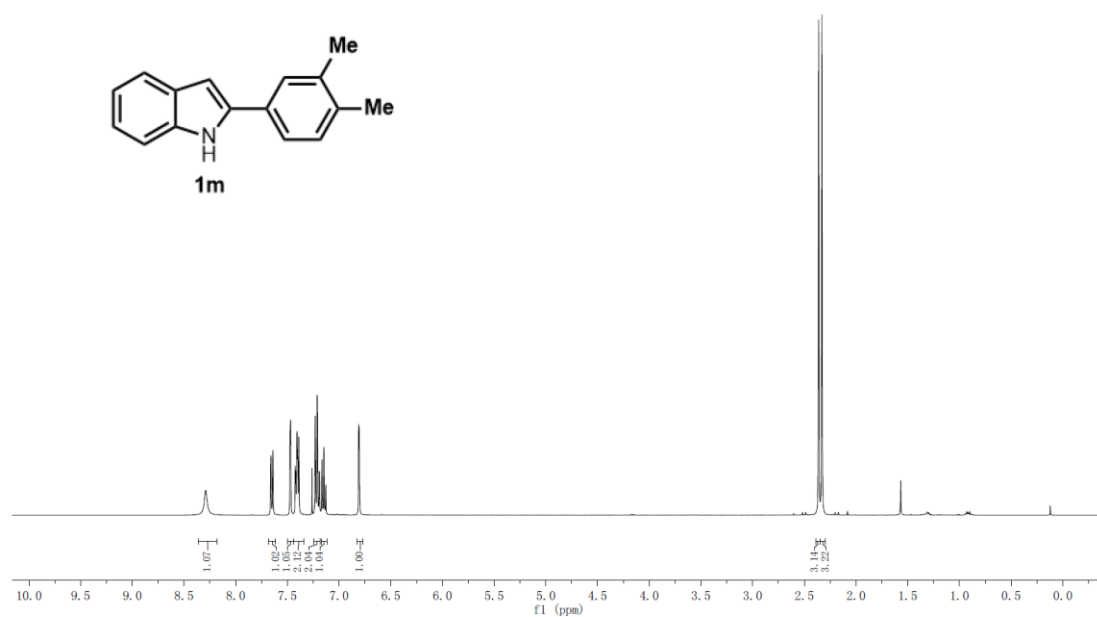
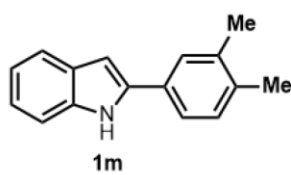
21.85
18.81



PROTON_01
r/Wang_CMW-265

8.290
8.111
7.703
7.470
7.410
7.385
7.365
7.360
7.358
7.322
7.312
7.188
6.880

2.361
2.330

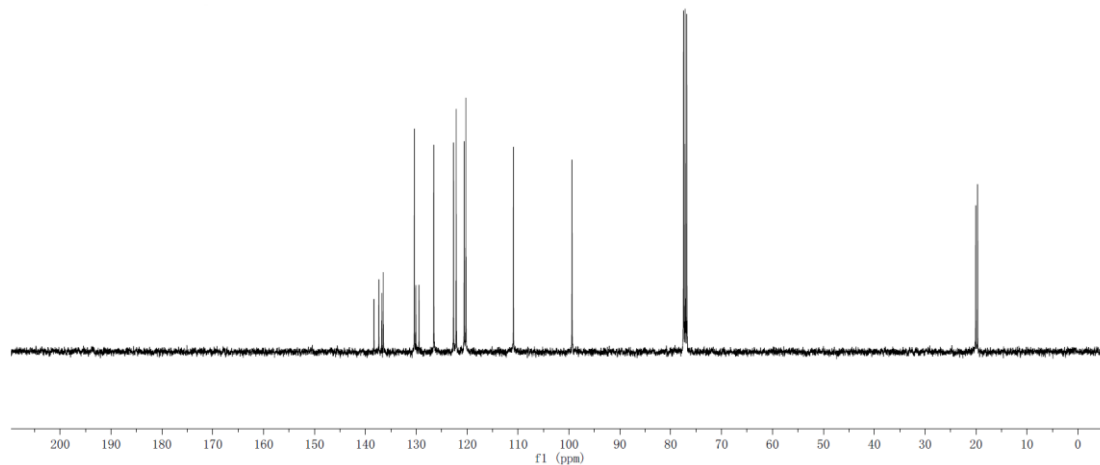
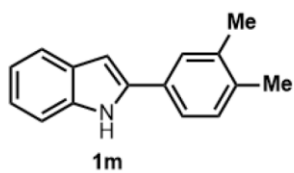


CARBON_01
r/Wang_CMW-265

138.31
137.84
136.78
136.50
135.72
130.08
129.46
128.95
122.14
120.60
120.26
—110.91

—59.40

20.06
19.71

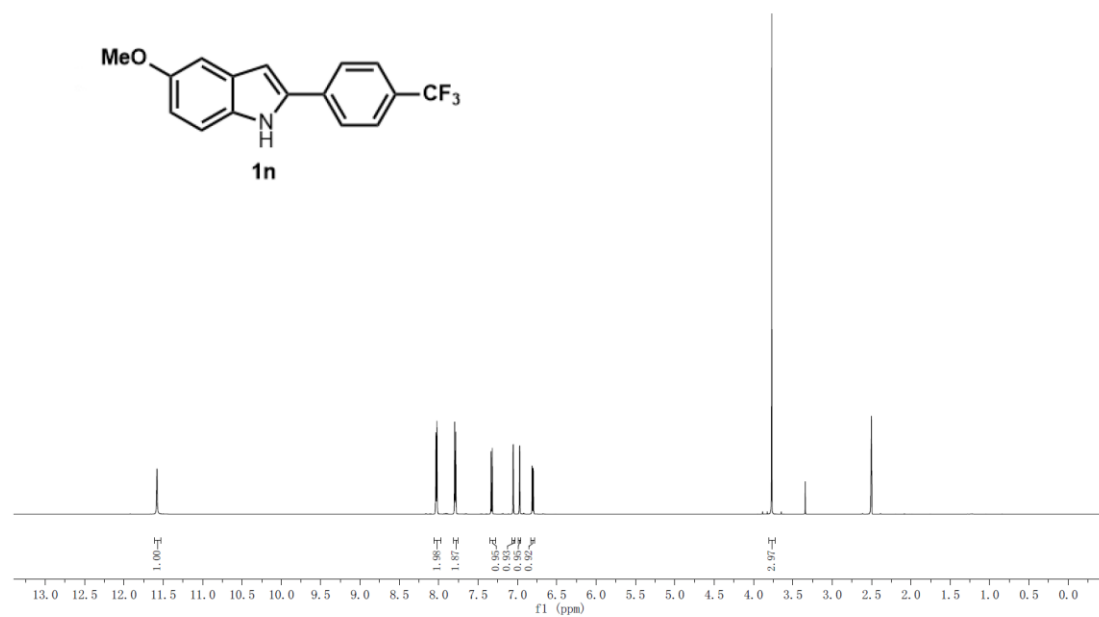
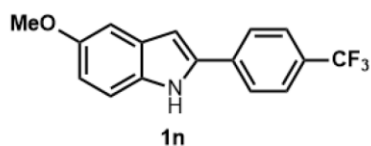


PROTON_01
r/Wang_CMW167

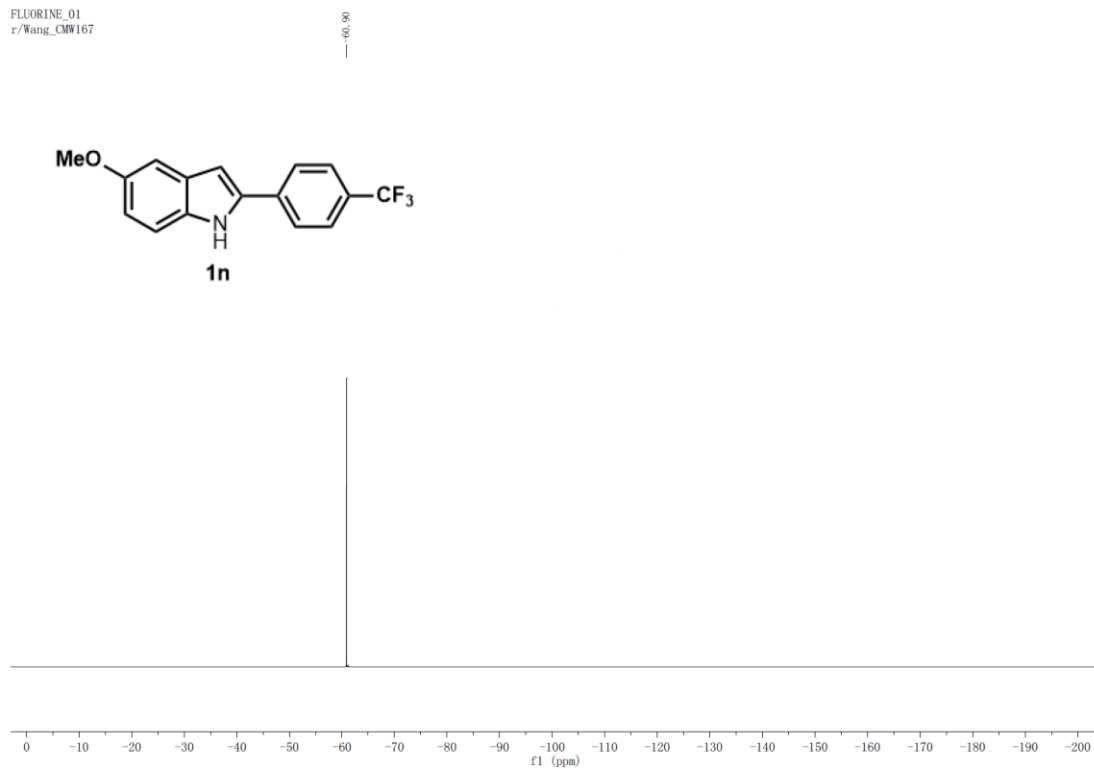
—11.380

8.036
8.022
7.785
7.781
7.335
7.335
7.035
7.035
6.814
6.810
6.795

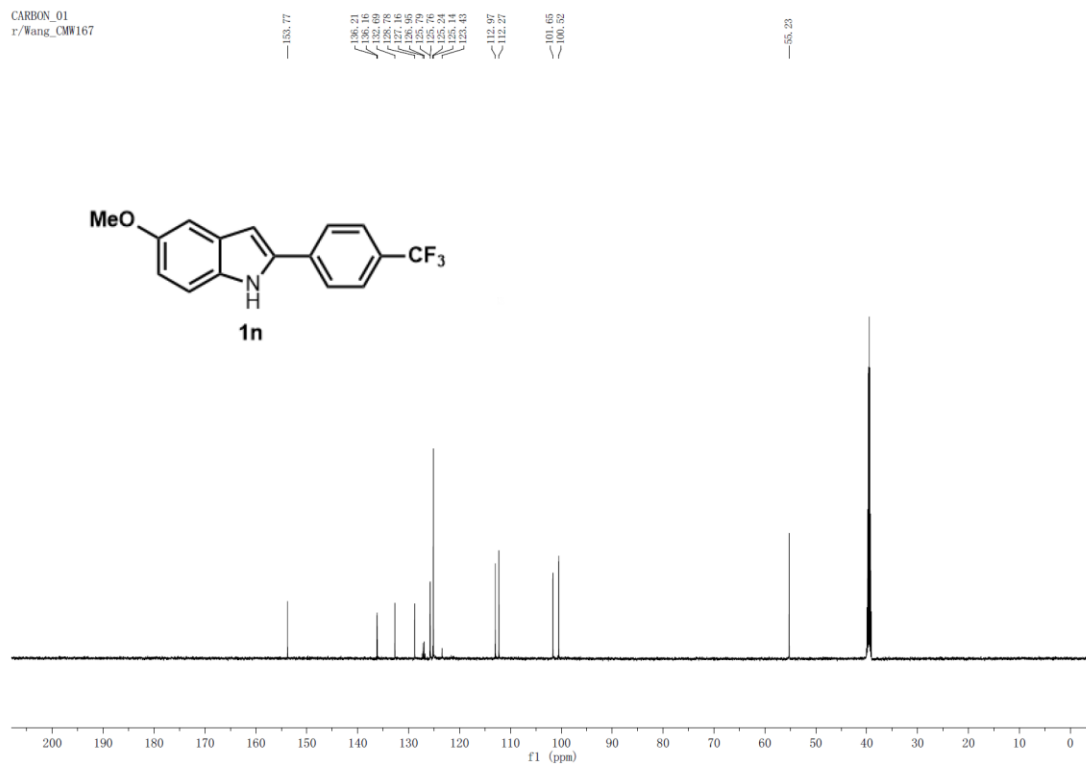
—3.769



FLUORINE_01
r/Wang_CMW167



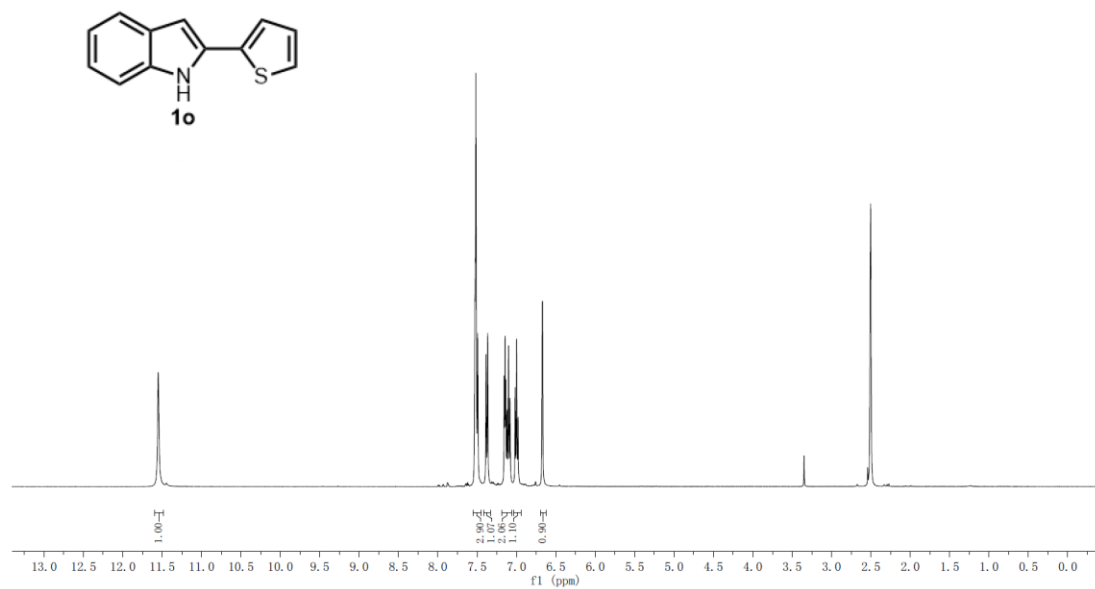
CARBON_01
r/Wang_CMW167



PROTON_01
r/wang_cmw201

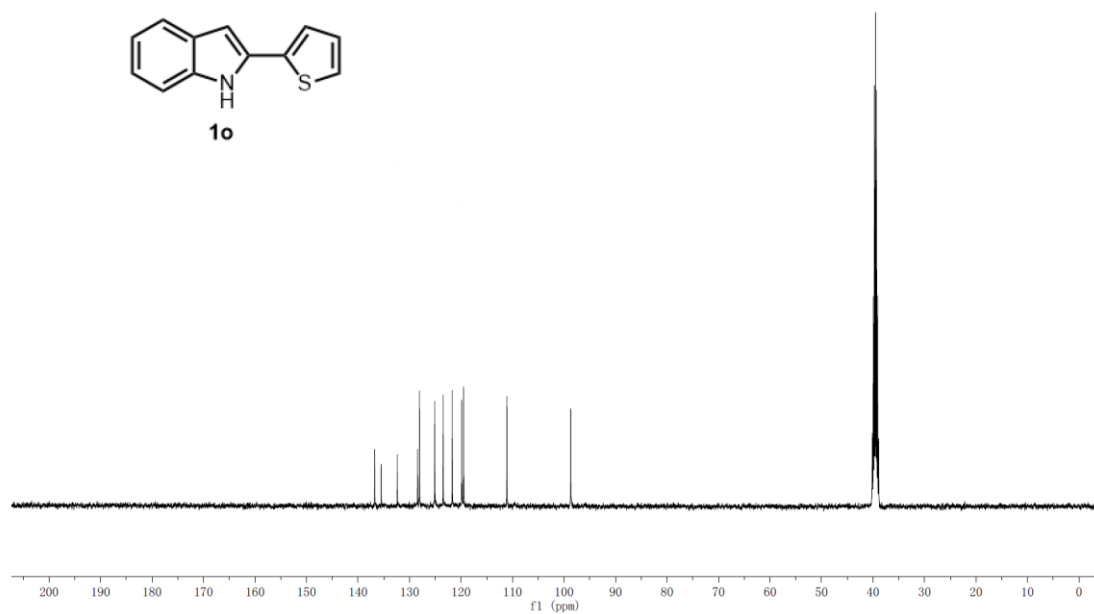
—11.550

7.529
7.526
7.516
7.494
7.485
7.387
7.357
7.337
7.317
7.313
7.302
7.293
7.017
6.988
6.973
6.670



CARBON_01
r/wang_cmw201

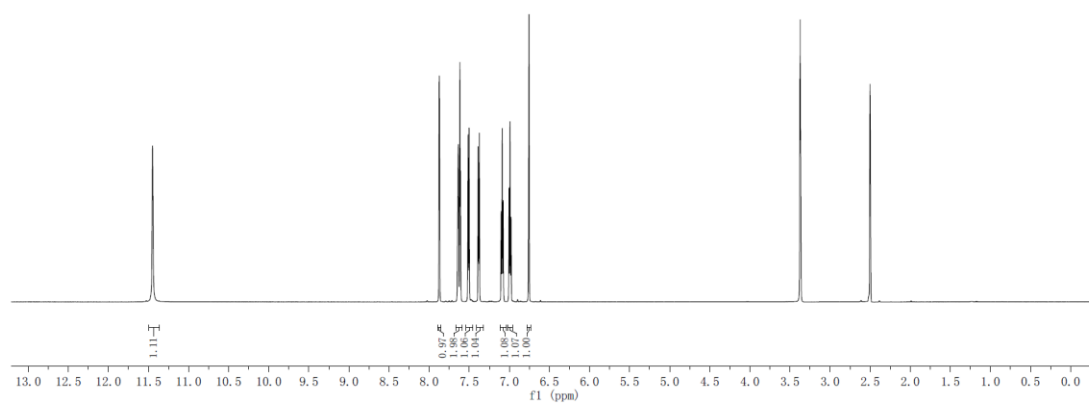
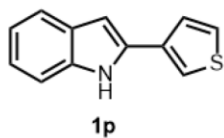
136.76
135.47
135.45
128.45
128.08
127.03
123.48
121.70
119.50
—111.10
—98.69



PROTON_01
r/Wang_CM#226

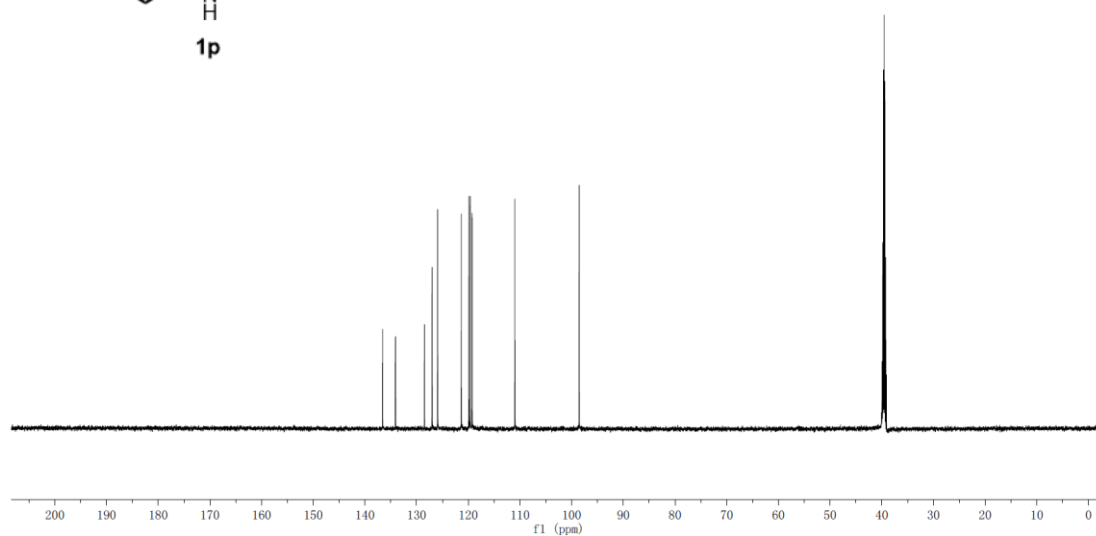
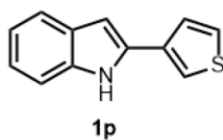
11.449

7.875
7.873
7.838
7.835
7.833
7.699
7.514
7.512
7.388
7.375
7.000
7.000
6.997
6.993
6.978
6.756

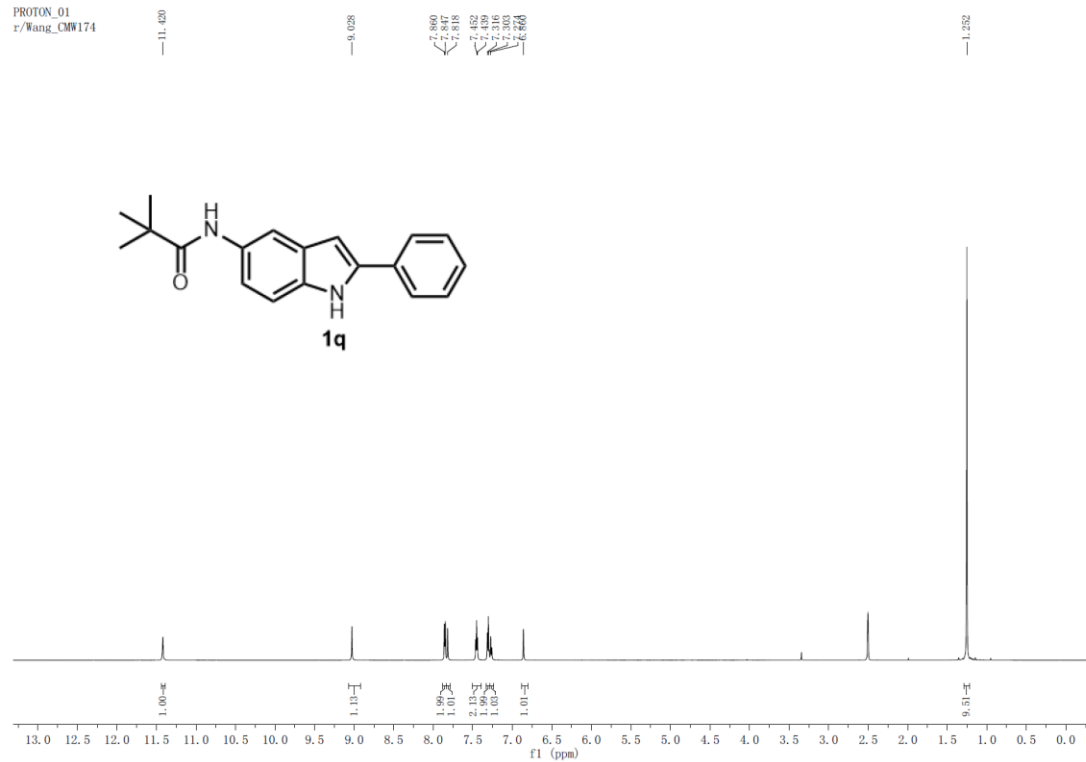


CARBON_01
r/Wang_CM#226

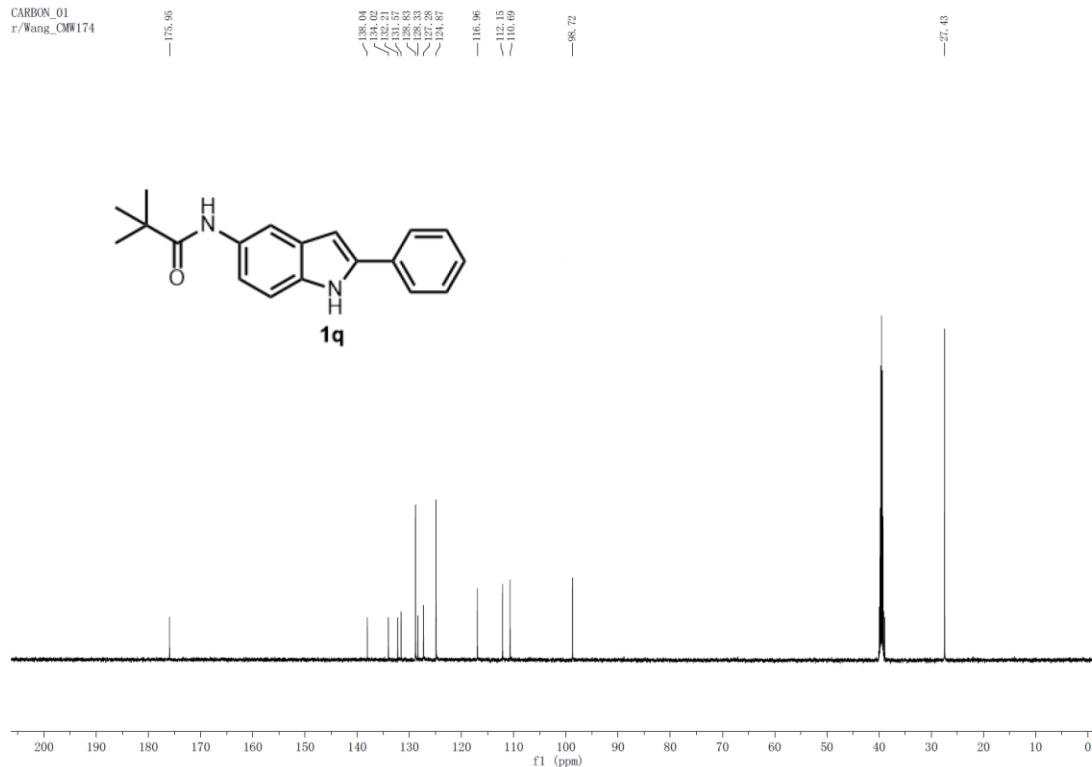
136.59
134.13
134.06
128.00
127.44
125.94
121.35
119.26
119.26
111.00
98.55



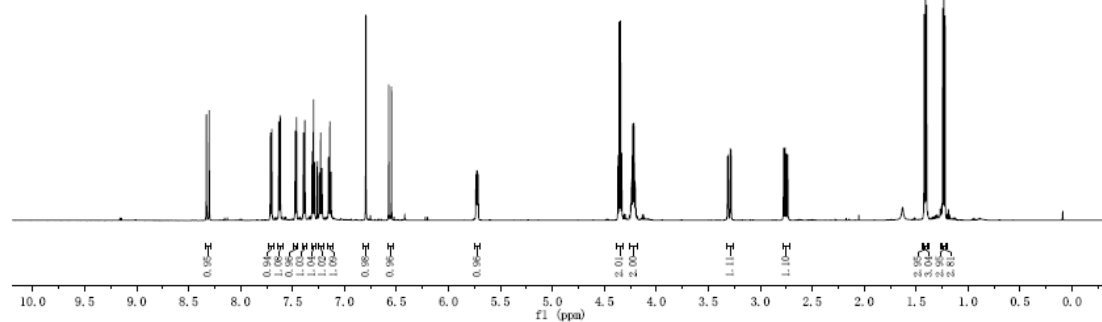
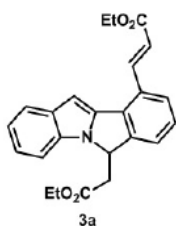
PROTON_01
r/Wang_CMW174



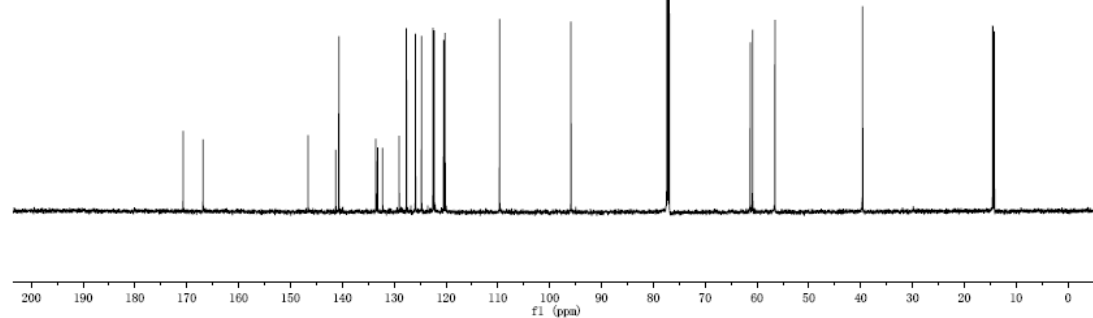
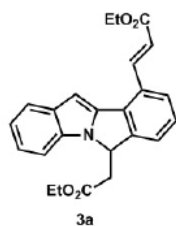
CARBON_01
r/Wang_CMW174



PROTON_01
r/Wang_CMW178



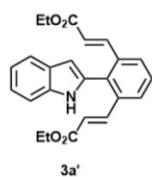
CARBON_01
r/Wang_CMW178



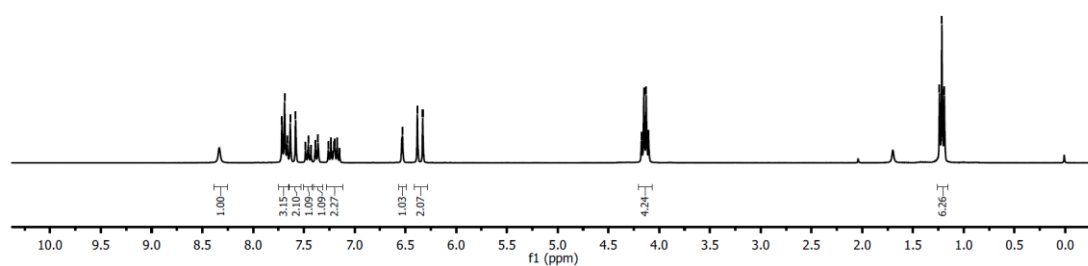
ZFM-I-56-B

7.716
7.690
7.685
7.634
7.581
7.483
7.457
7.431
7.397
7.360
7.233
7.205
7.198
7.171
7.149
5.529
4.175
4.152
4.128
4.104

1.239
1.215
1.191



3a'



ZFM-I-56-B-C

166.57

142.71

135.75

135.75

131.63

129.02

128.48

125.96

120.69

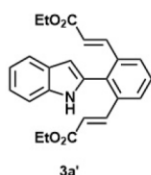
120.40

111.13

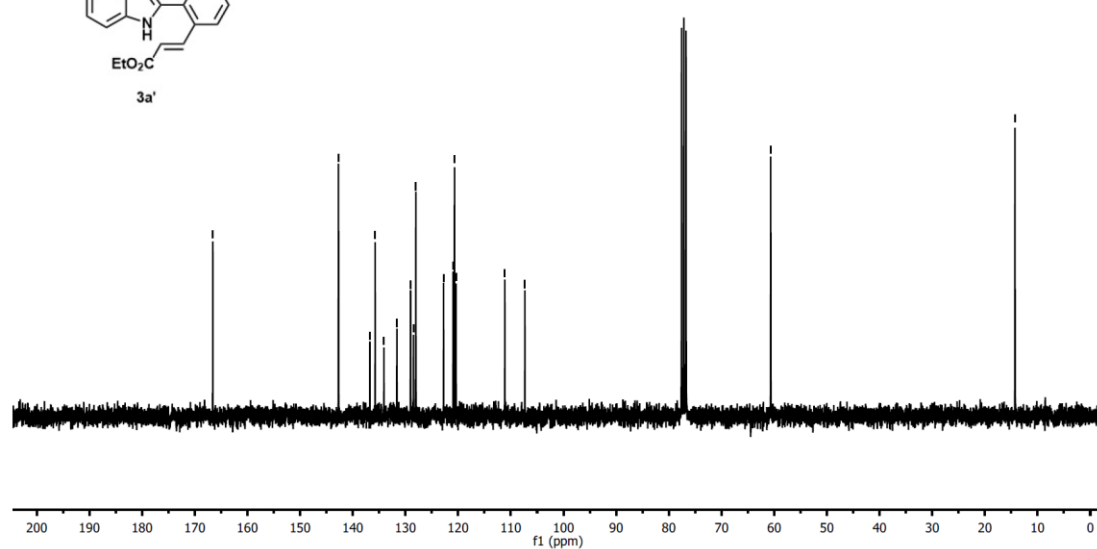
107.33

60.66

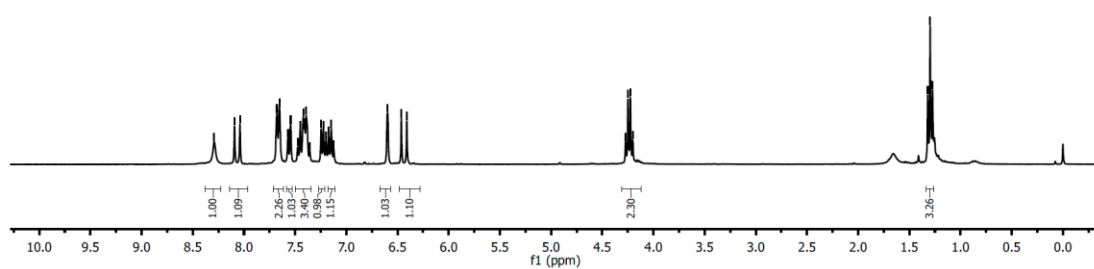
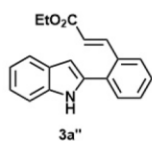
14.29



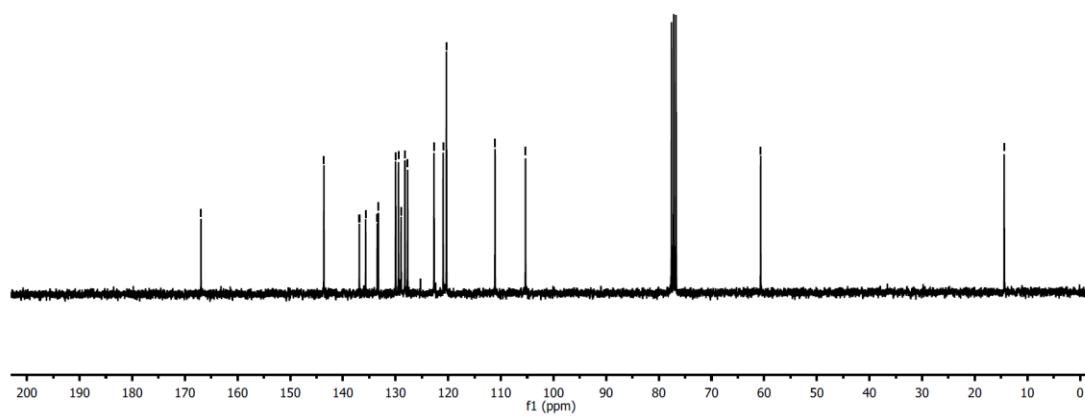
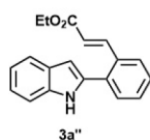
3a'



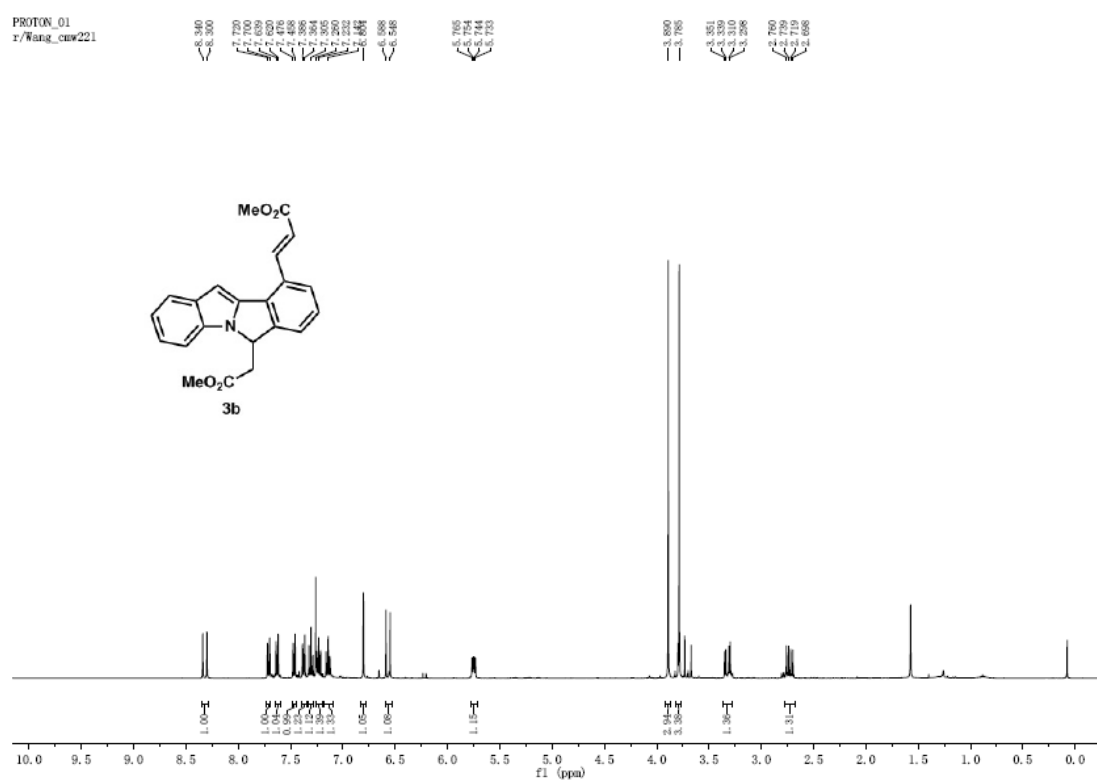
ZFM-I-56-D



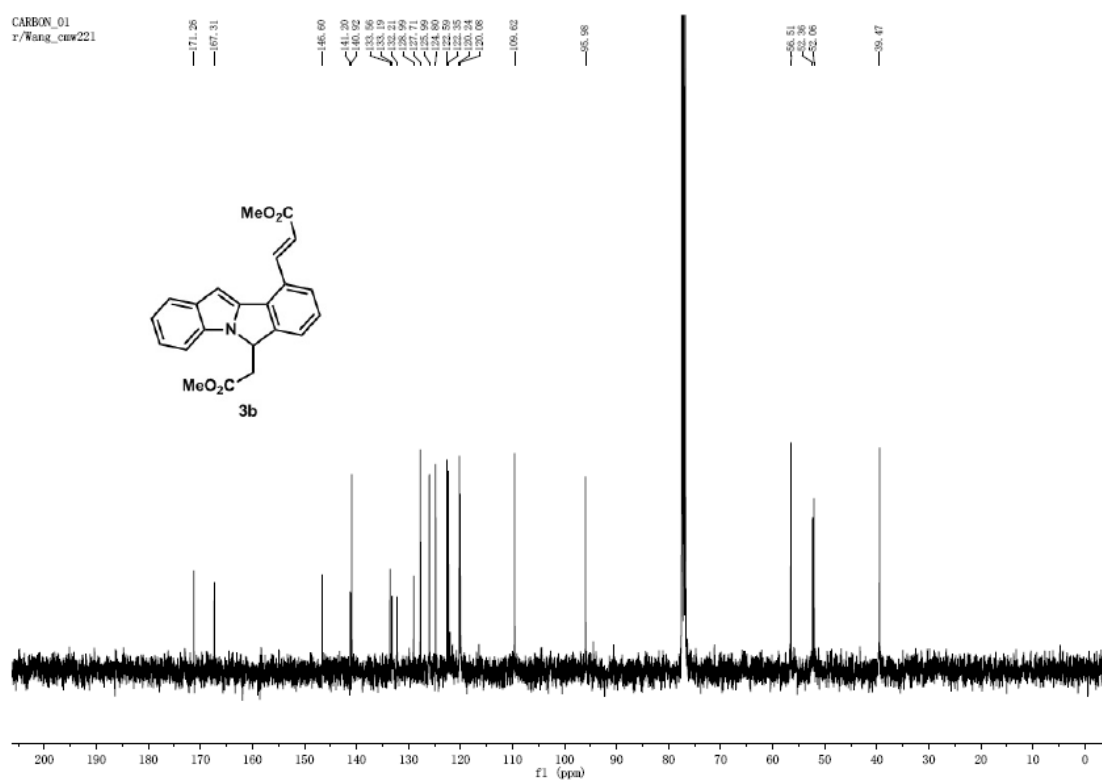
ZFM-I-56-D-C
ZFM-I-56-D-C



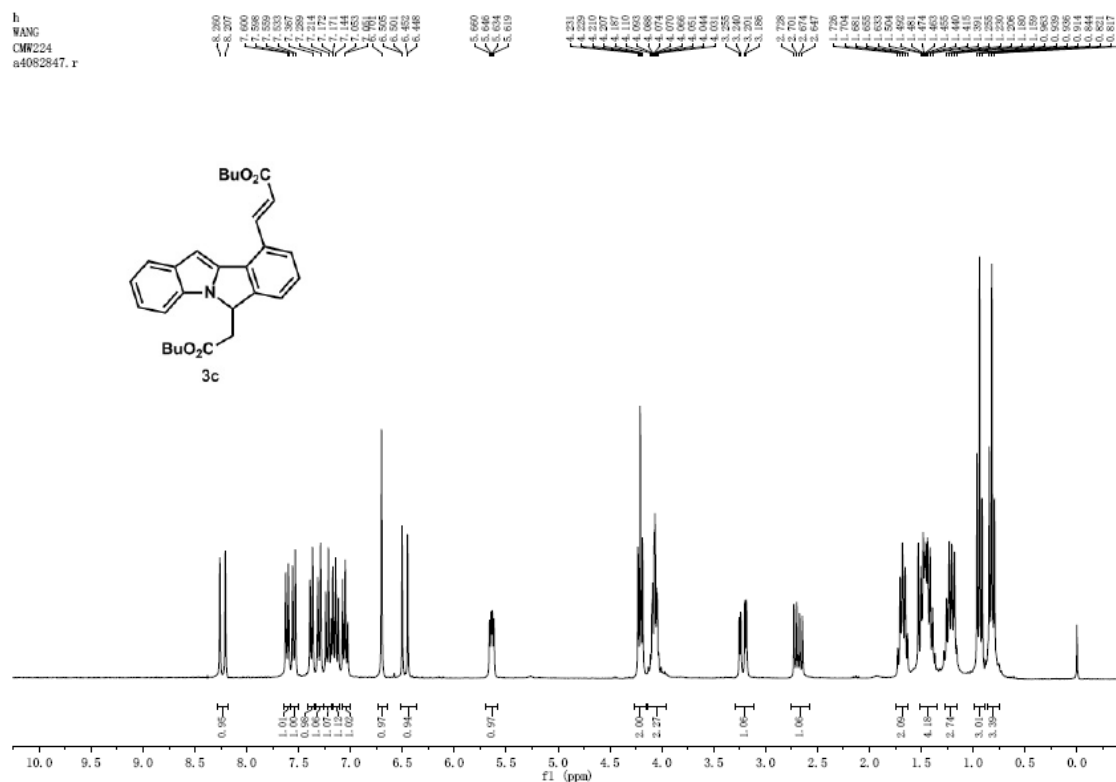
PROTON_01
r/Wang_cwv221



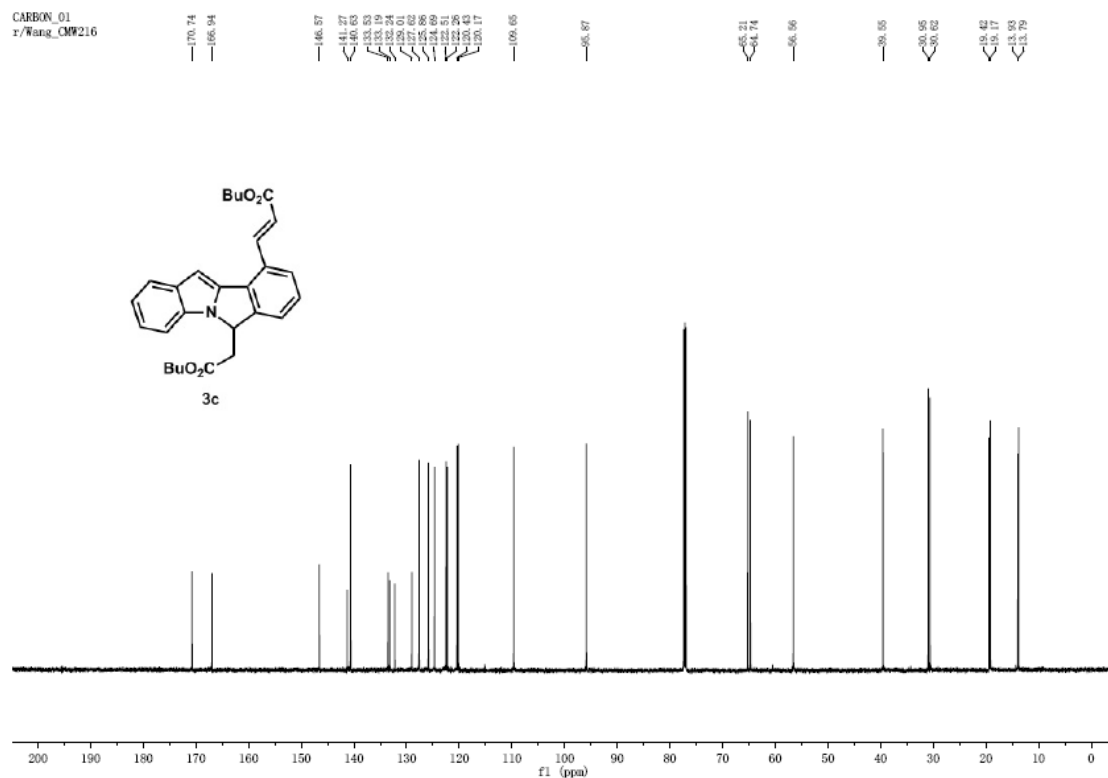
CARBON_01
r/Wang_cwv221



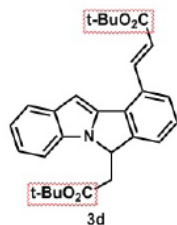
h
WANG
CMW224
a4062847.r



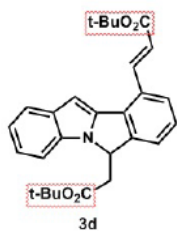
CARBON_01
r/Wang_CMW216



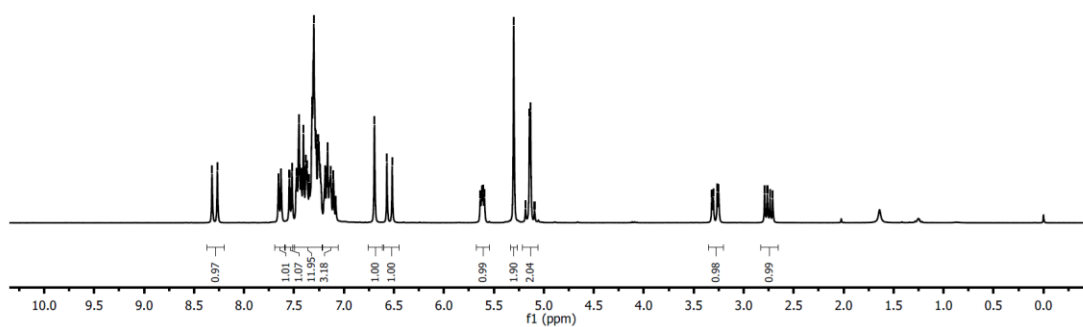
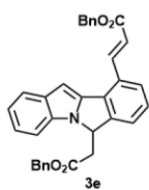
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8.227
7.705
7.679
7.641
7.615
7.489
7.464
7.413
7.396
7.361
7.222
6.199
6.528
6.475
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5.691
5.680
5.666
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3.233
3.194
3.180
2.830
2.804
2.776
2.751



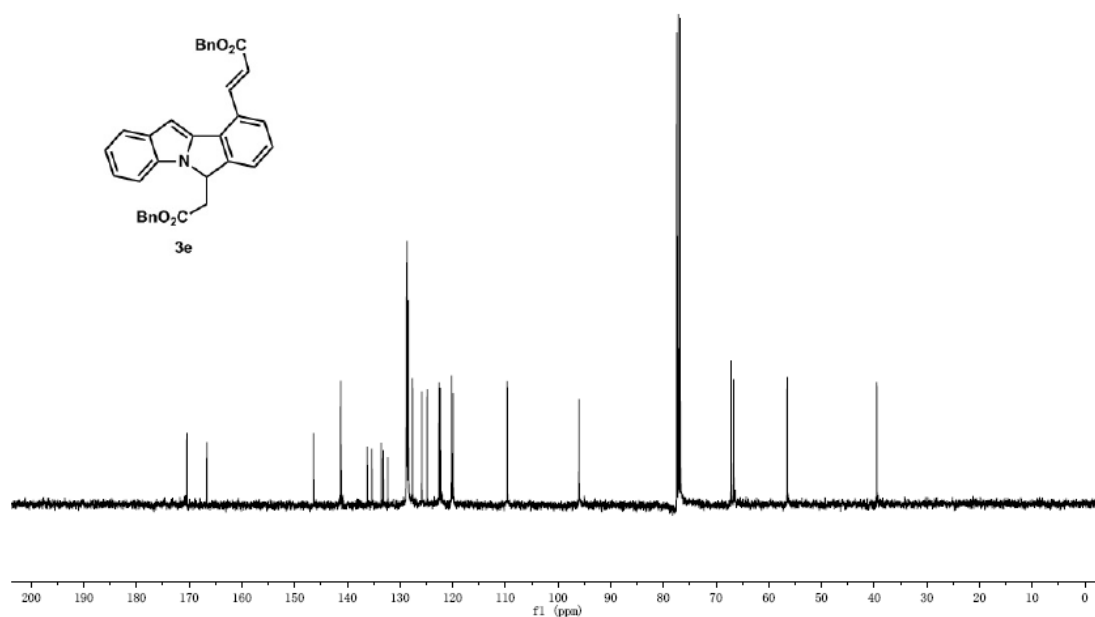
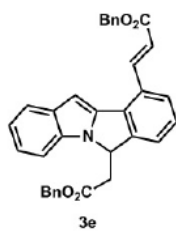
—169.73
—166.25
—146.65
—141.47
—139.72
—133.51
—133.25
—132.31
—129.12
—127.54
—125.67
—124.94
—123.96
—122.19
—122.15
—120.07
—109.83
—95.74
—81.72
—80.93
—56.74
—40.54
—38.41
—38.02



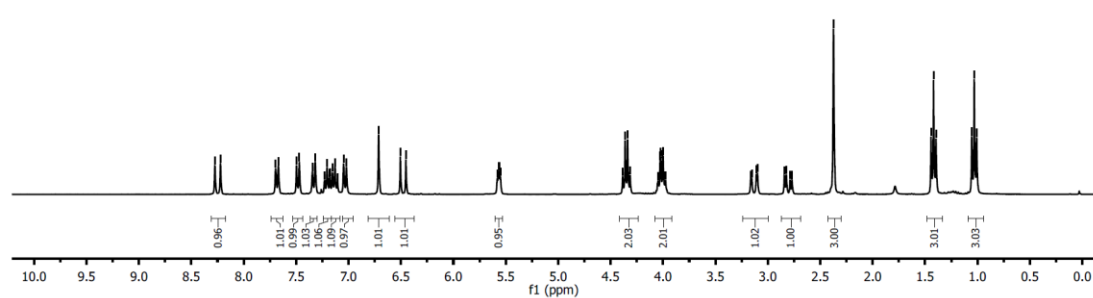
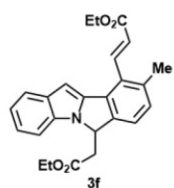
8.3199
8.2667
7.4493
7.4055
7.3796
7.3701
7.3182
7.3011
7.2787
7.2596
7.2481
7.1639
6.5702
6.5172
5.6351
5.6210
5.6091
5.5945
5.5012
5.1834
5.1429
5.1331
5.0923
3.3184
3.3039
3.2643
3.2498
2.7903
2.7634
2.7362
2.7092



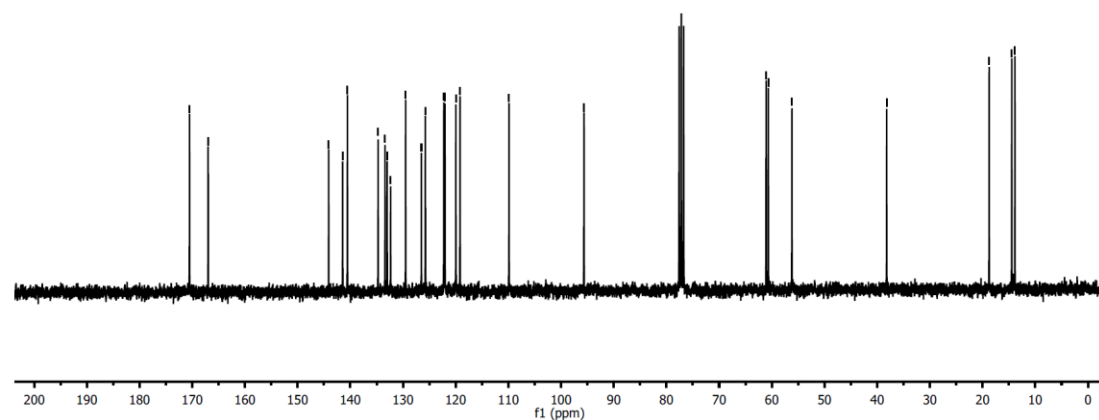
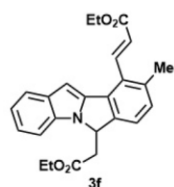
CARBON_01
r/wang_ciw204



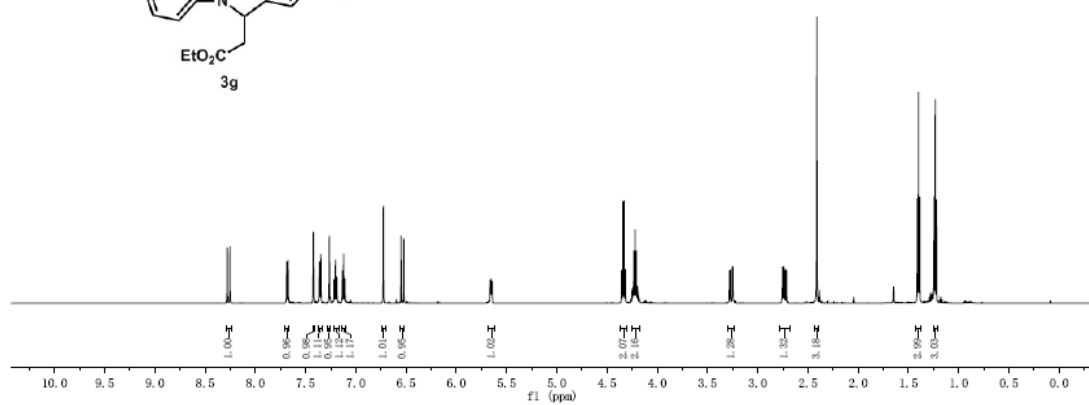
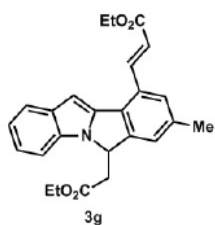
ZFM-I-105-1
ZFM-I-105-1-H



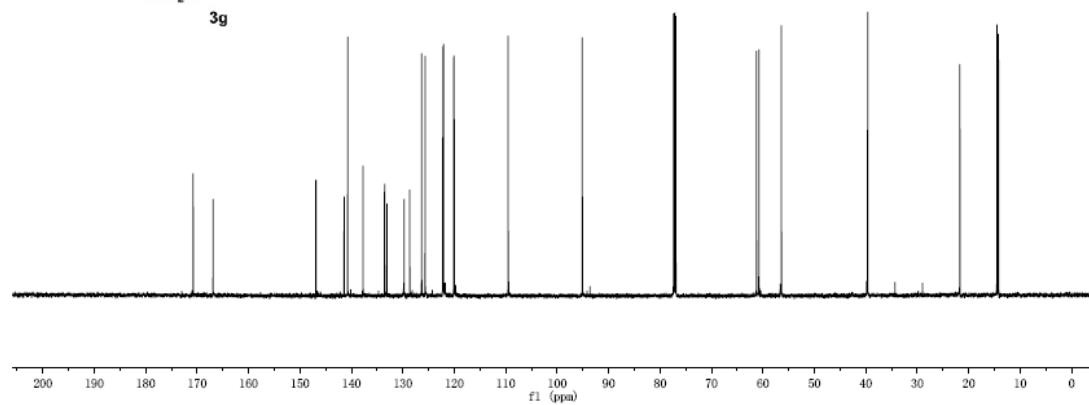
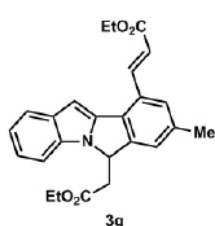
ZFM-I-105-1-C
ZFM-I-105-1-C



PROTON_01
r/Wang_CM9215

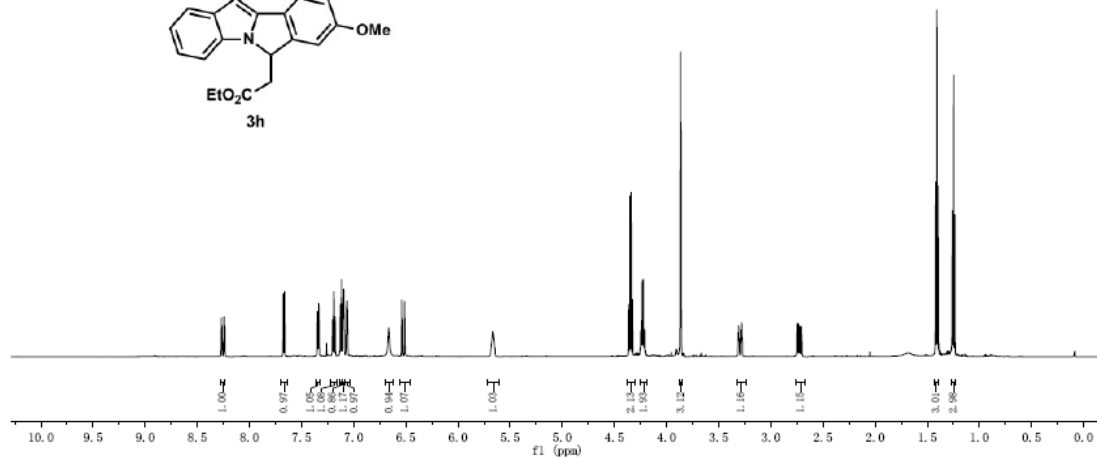
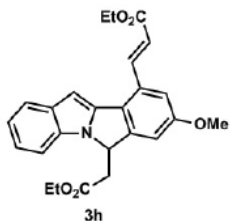


CARBON_01
r/Wang_CM9215



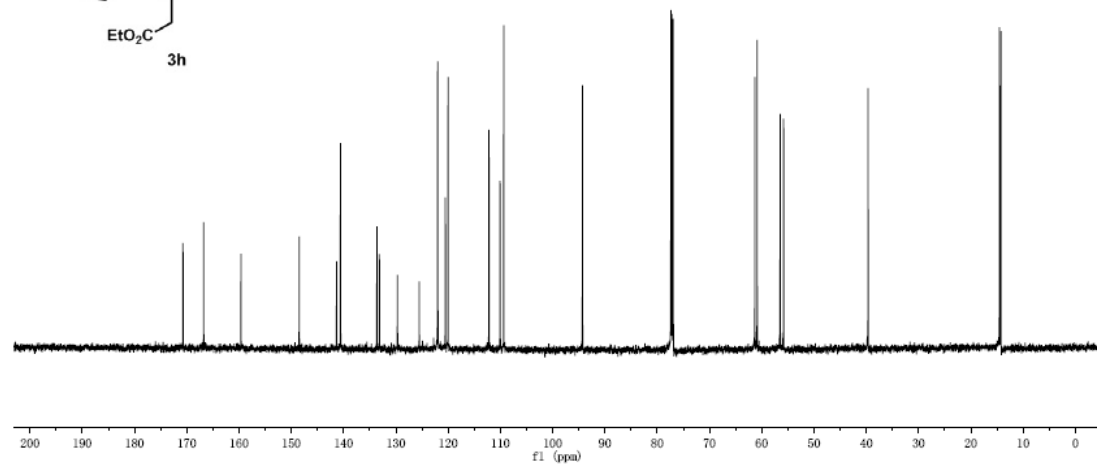
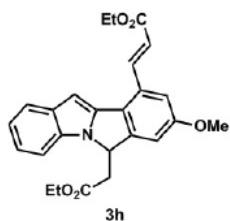
PROTON_01
r/Wang_CMW186

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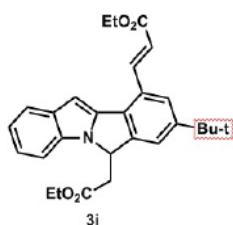


CARBON_01
r/Wang_CMW186

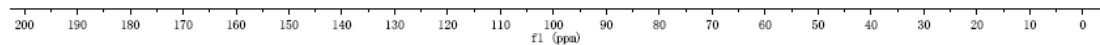
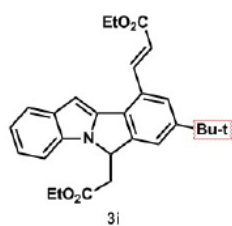
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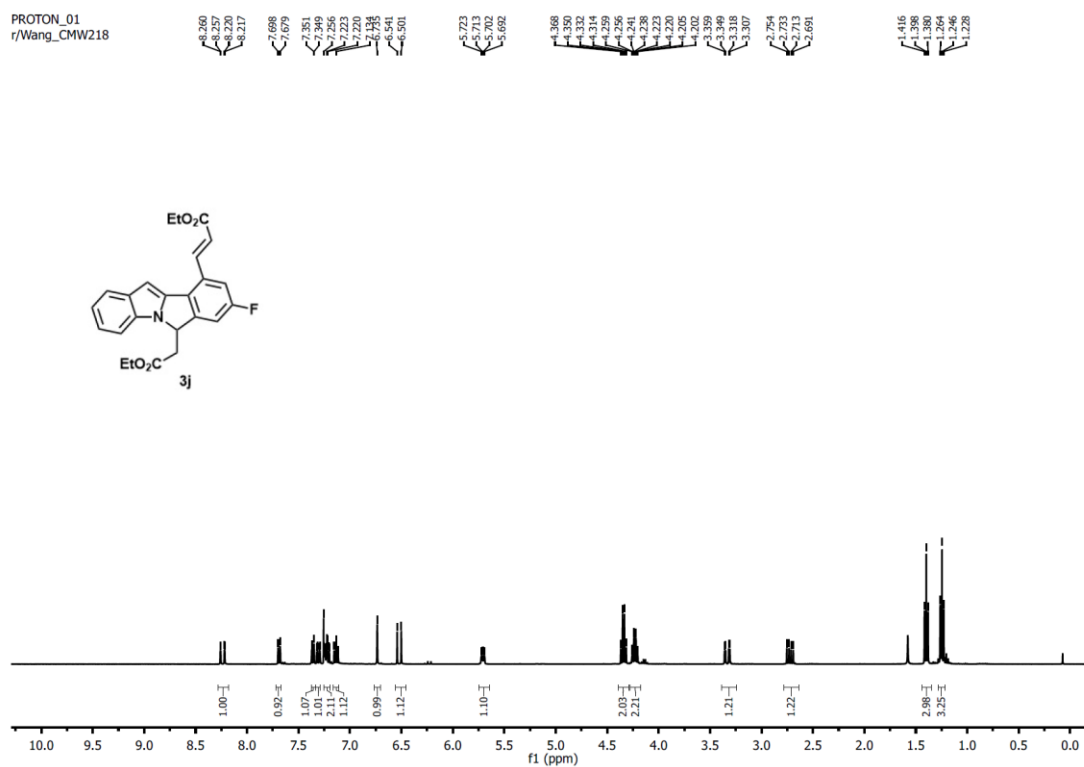
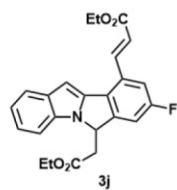
PROTON_01
r/Wang_CMW181



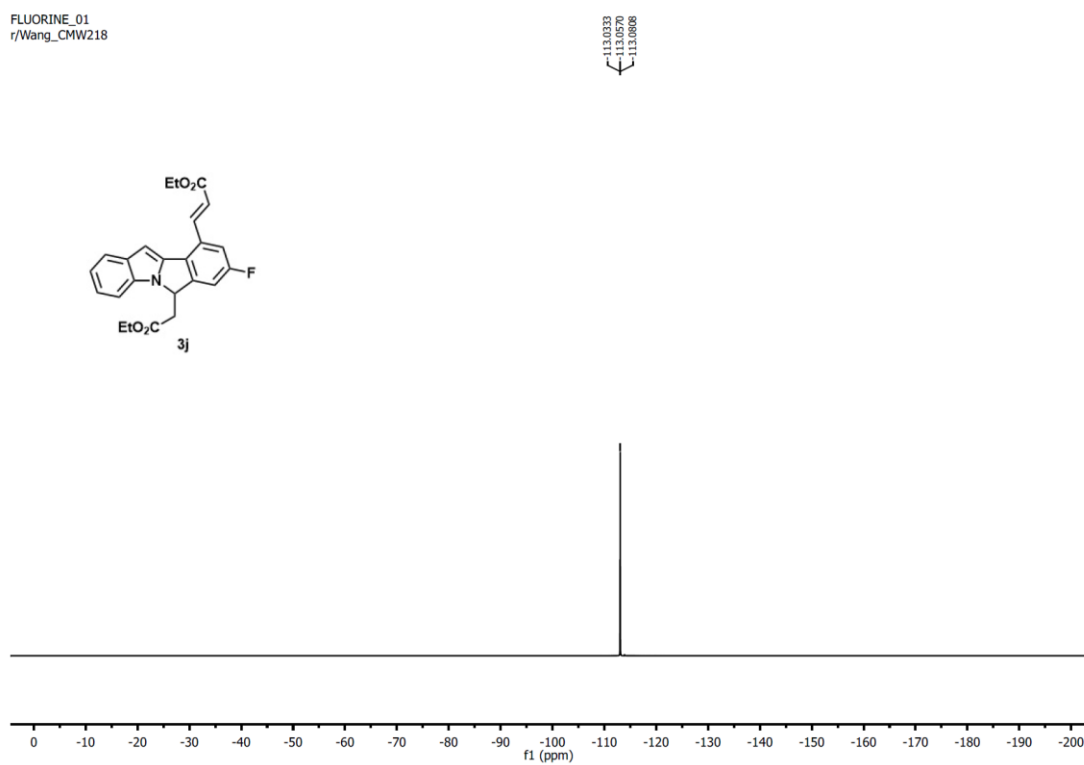
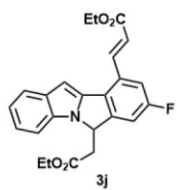
CARBON_01
r/Wang_CMW181

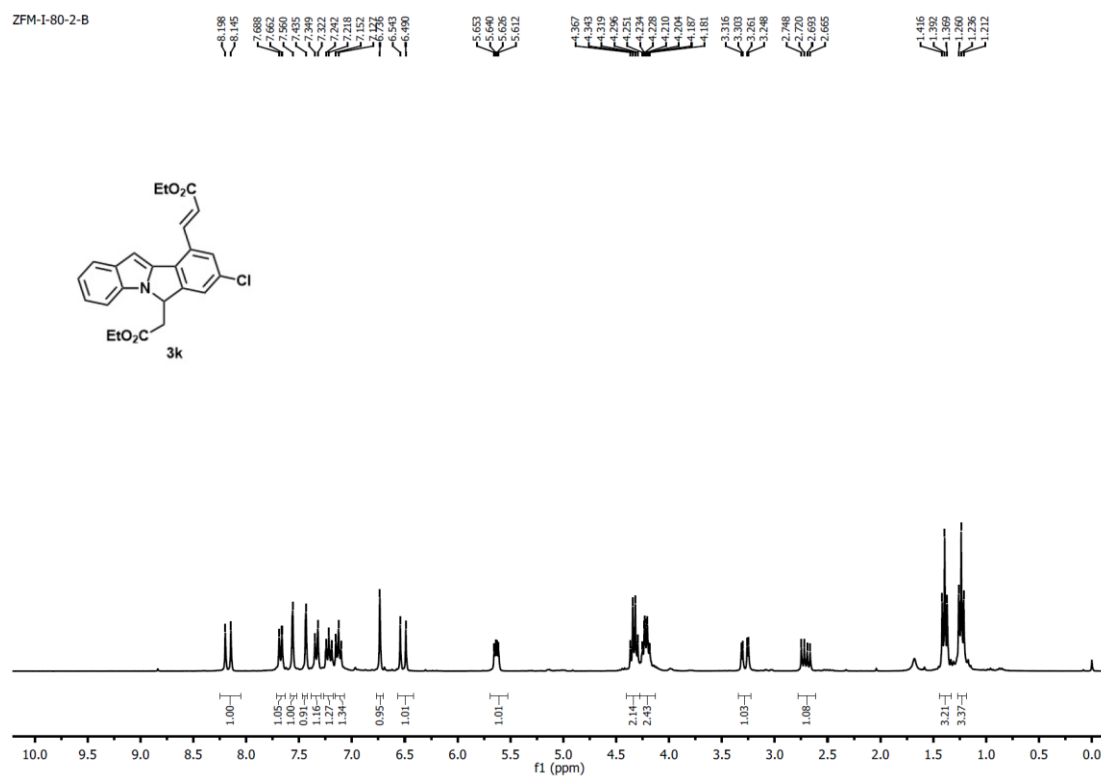
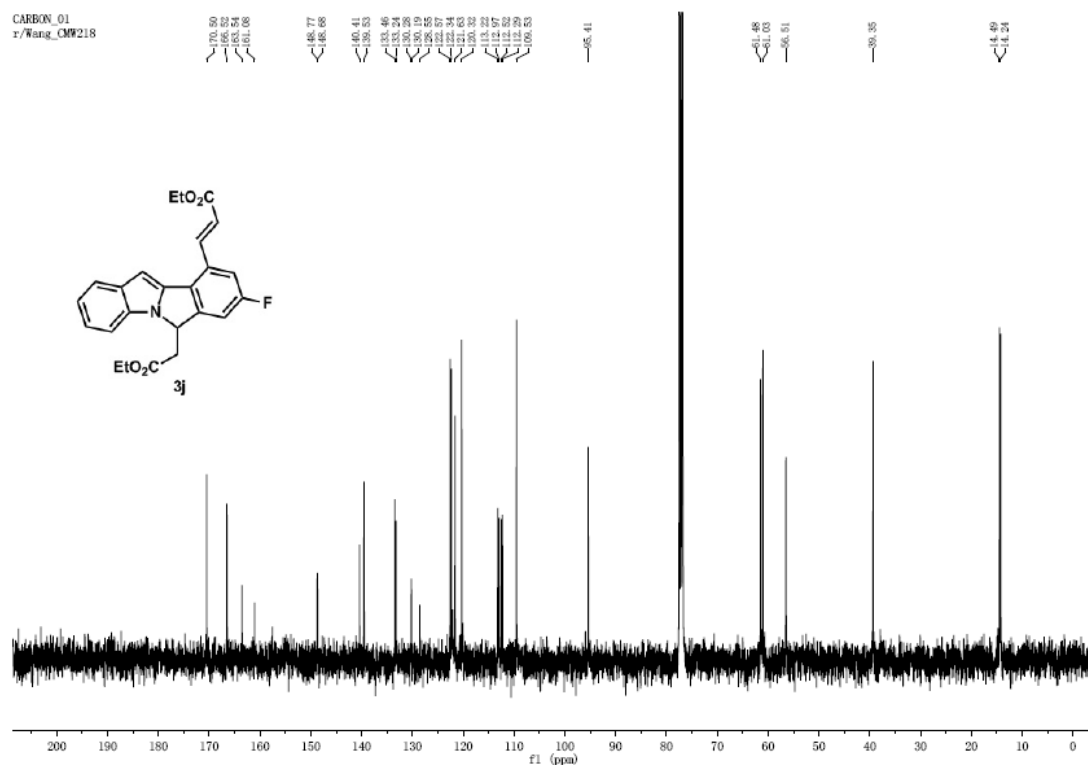


PROTON_01
r/Wang_CMW218



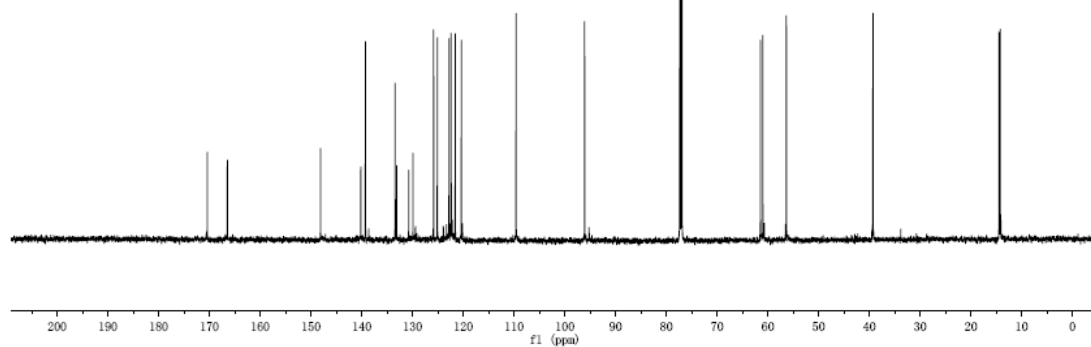
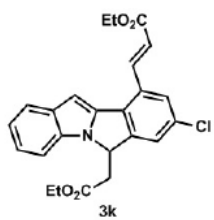
FLUORINE_01
r/Wang_CMW218





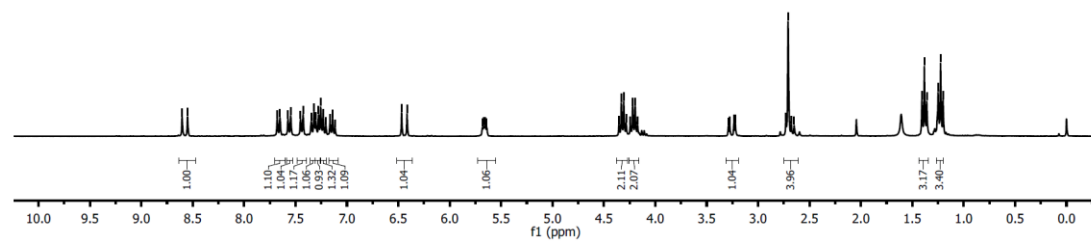
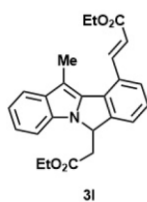
CARBON_01
r/wang_cw207

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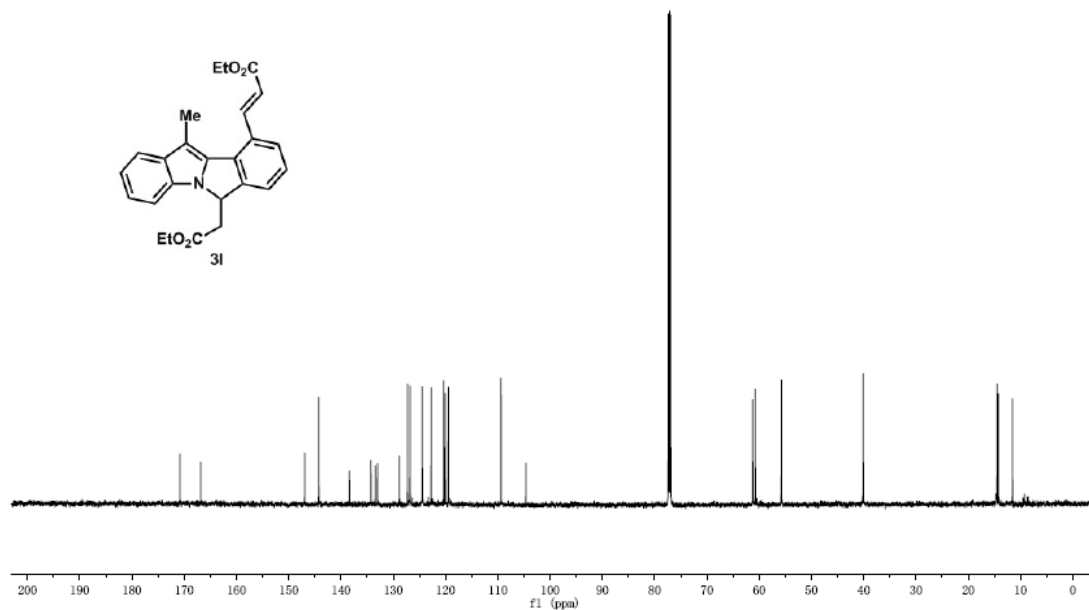
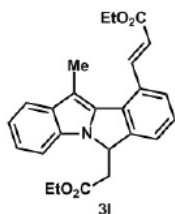
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ZFM-I-87-H

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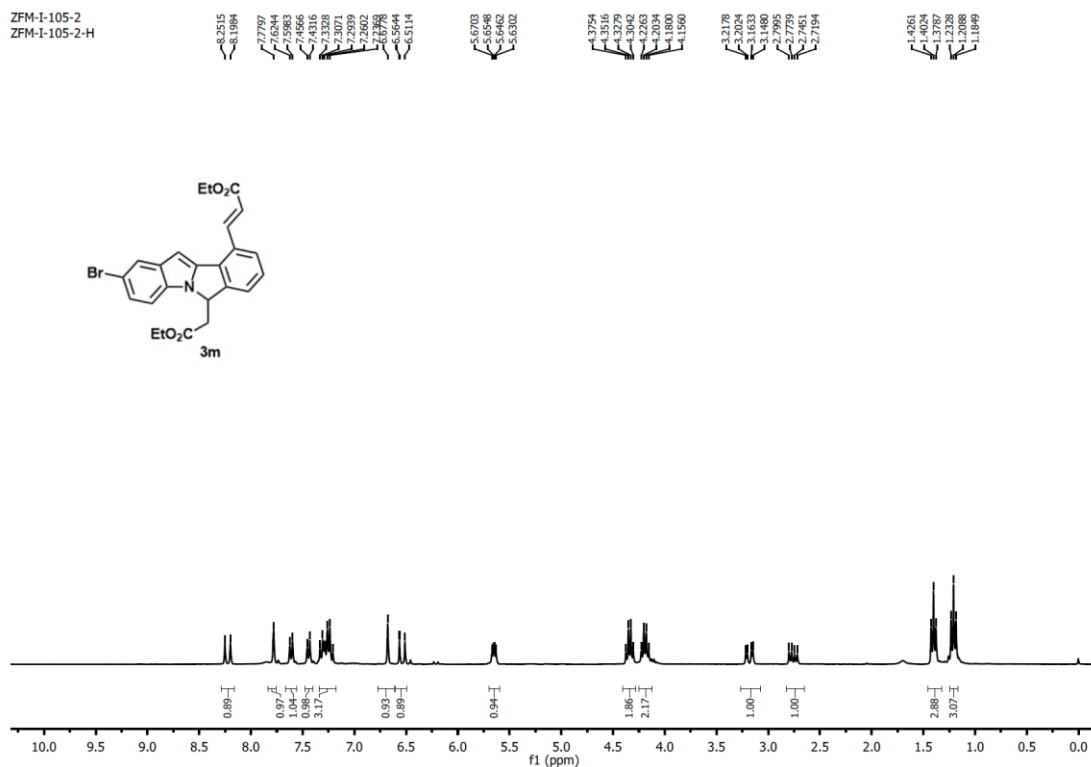
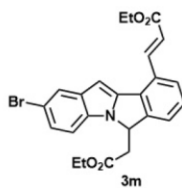
CARBON_01
r/Wang_CM9192

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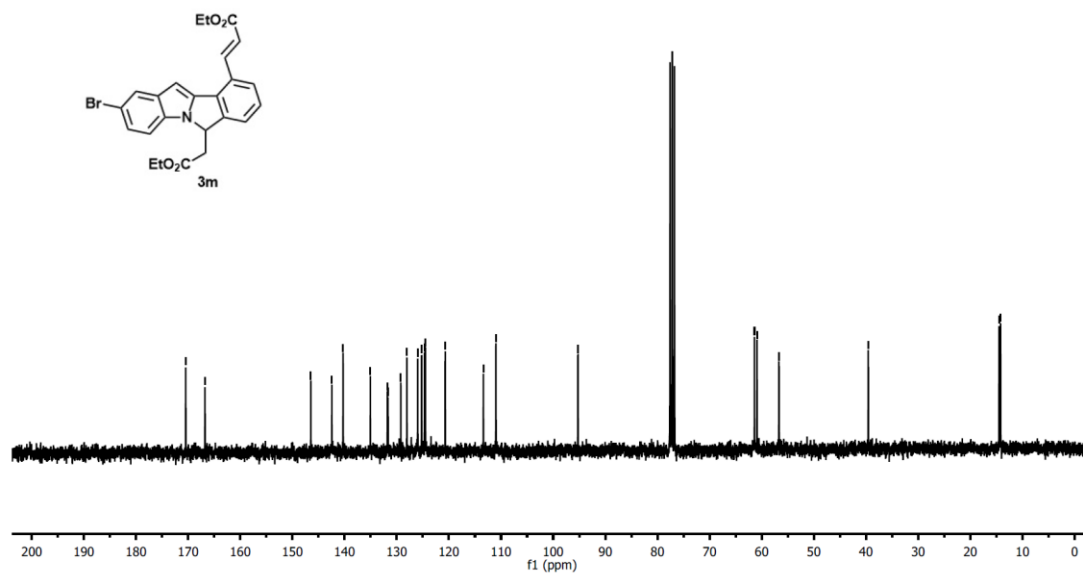
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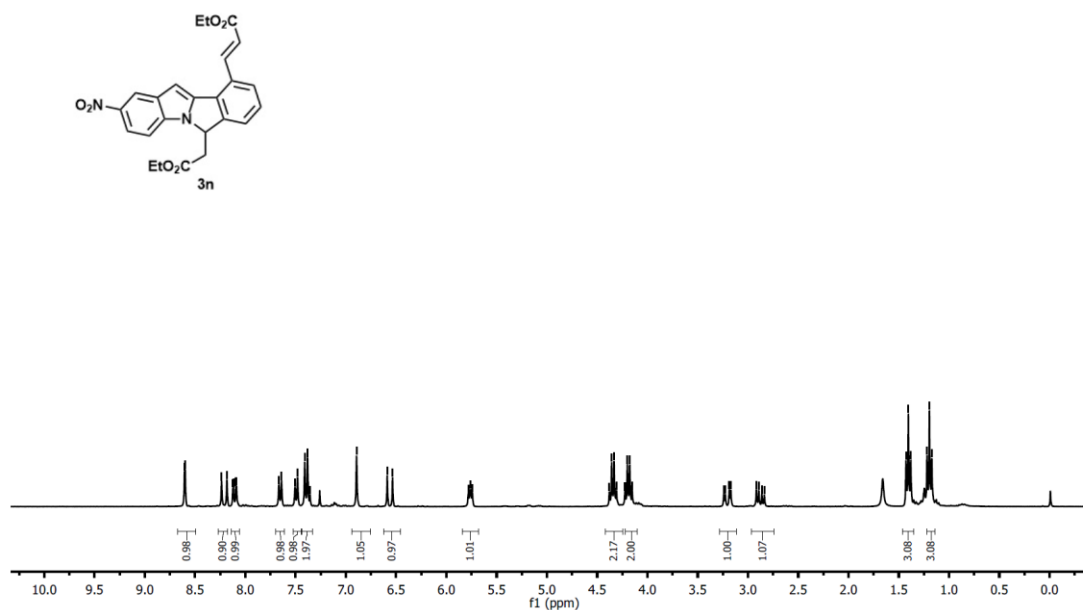
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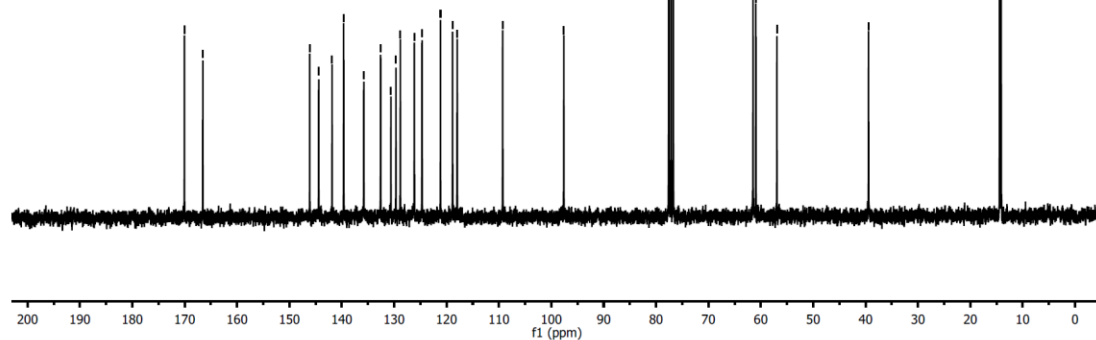
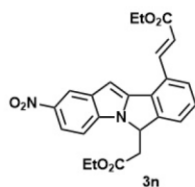
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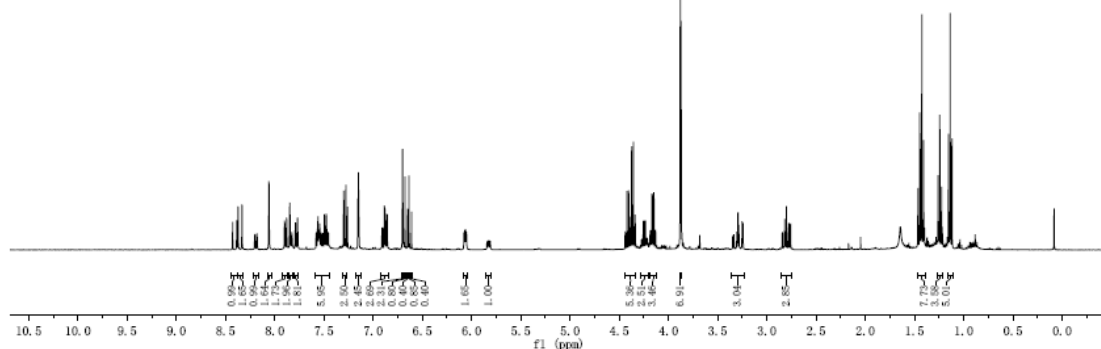
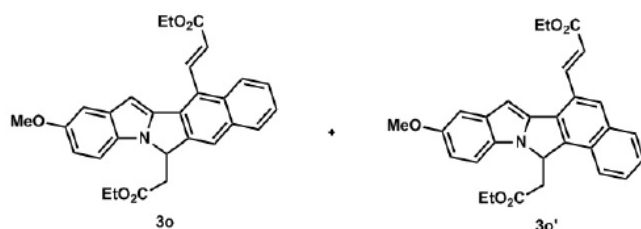


ZFM-1-79-C
ZFM-1-79-C

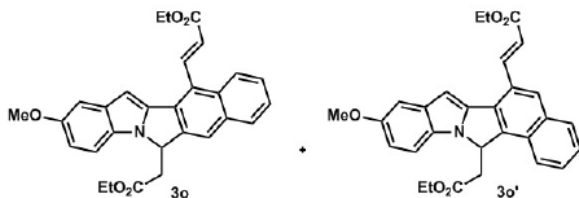
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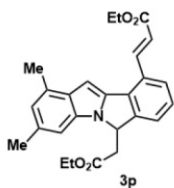
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7.125
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7.123
7.122
7.121
7.120



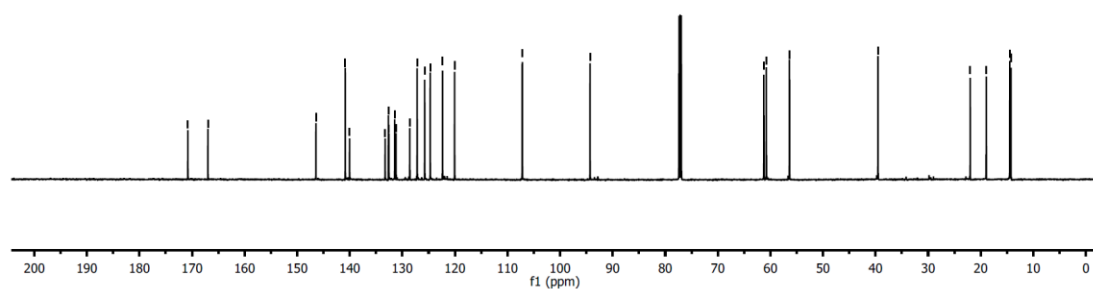
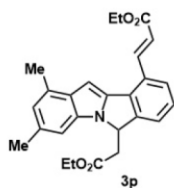
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 $\begin{array}{c} 184.59 \\ 184.37 \\ 180.81 \end{array}$
 $\begin{array}{c} 143.52 \\ 142.75 \\ 141.68 \\ 140.96 \\ 140.56 \end{array}$
 $\begin{array}{c} 132.91 \\ 130.06 \\ 129.06 \\ 127.37 \\ 127.26 \\ 126.97 \\ 126.40 \\ 125.76 \\ 123.93 \\ 120.50 \end{array}$
 $\begin{array}{c} 103.79 \\ 103.54 \\ 98.51 \\ 98.44 \end{array}$
 $\begin{array}{c} 61.47 \\ 61.33 \\ 61.01 \\ 60.87 \\ 58.16 \\ 55.13 \\ 55.05 \end{array}$
 $\begin{array}{c} 40.67 \\ 40.18 \end{array}$
 $\begin{array}{c} 14.55 \\ 14.27 \\ 14.10 \end{array}$



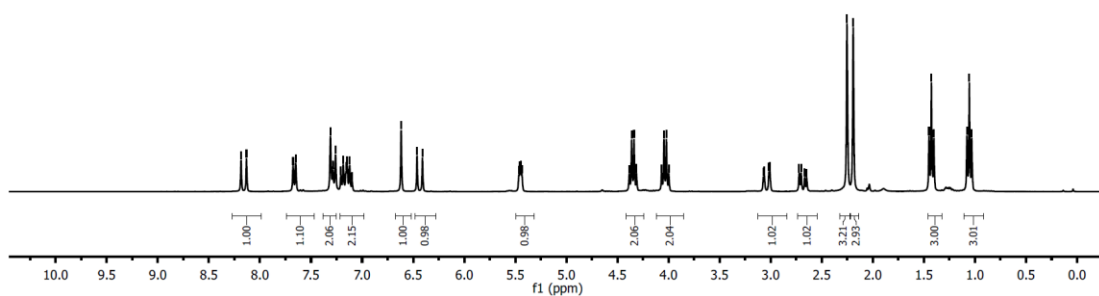
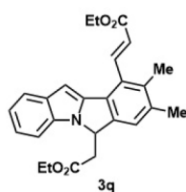
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7.263
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7.250
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6.715
6.575
6.547
5.669
5.662
5.655
5.646
4.369
4.357
4.345
4.333
4.258
4.255
4.246
4.243
4.234
4.231
4.222
4.219
3.320
3.313
3.292
3.285
2.717
2.717
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2.675
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1.416
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1.246
1.246



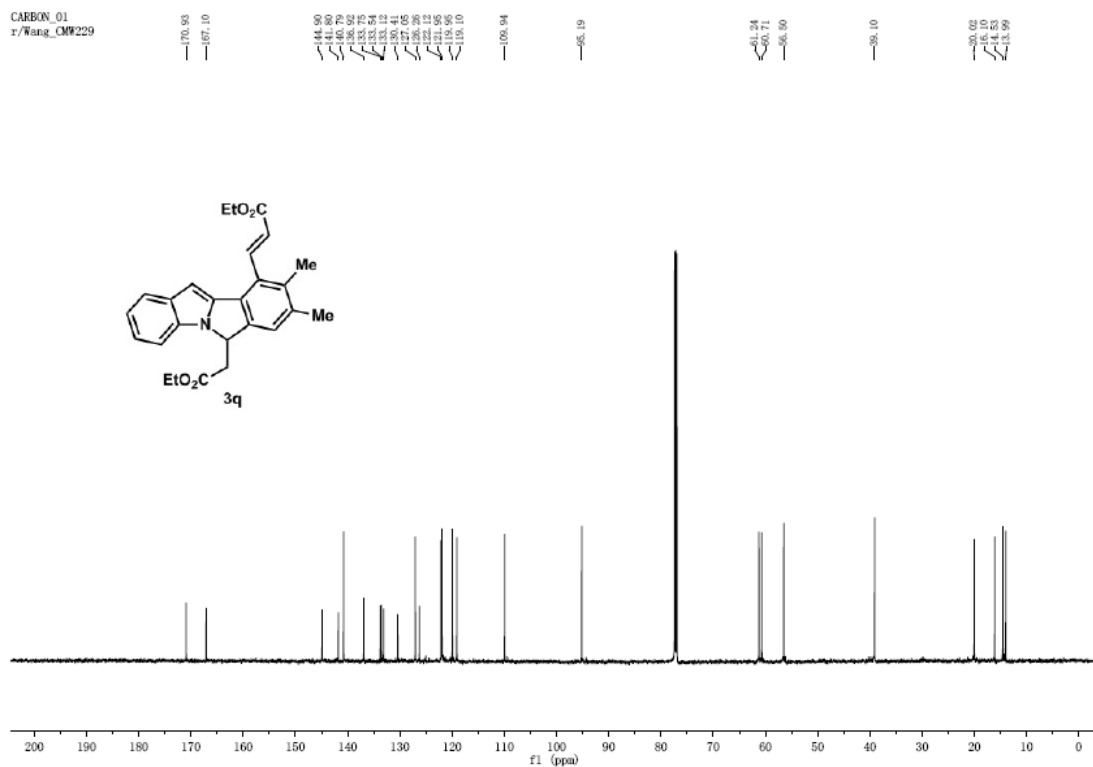
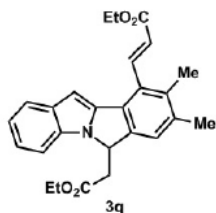
CARBON_01
r/wang_cmw210



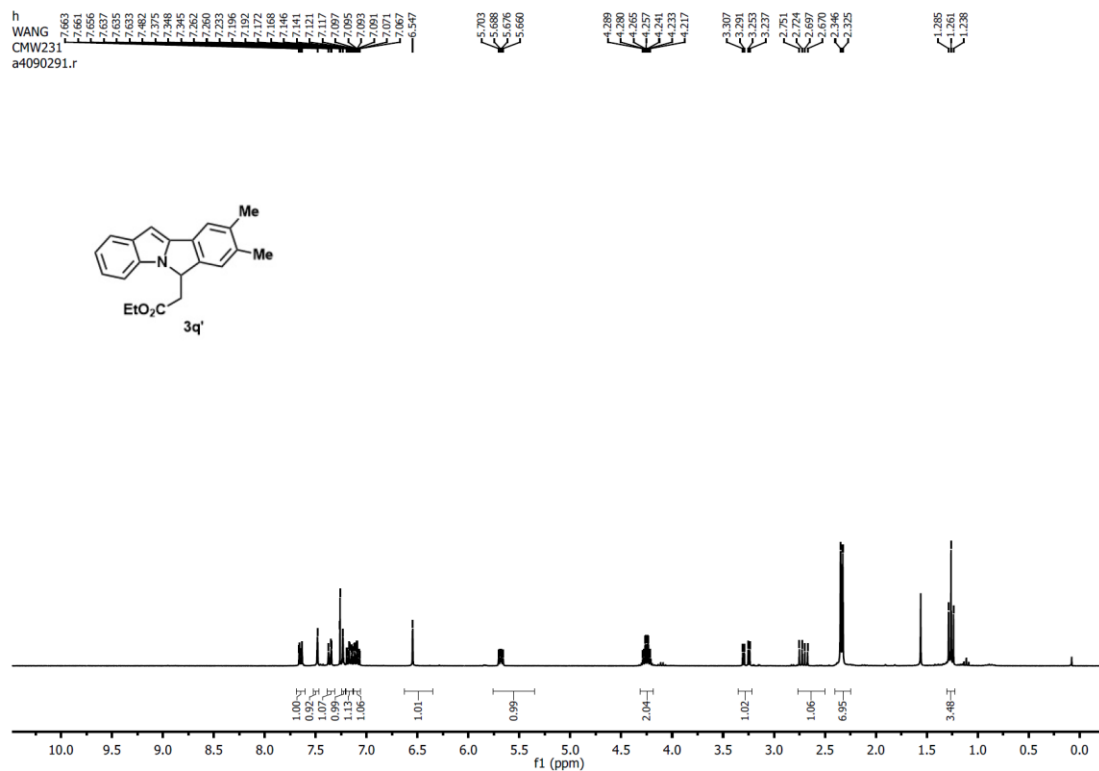
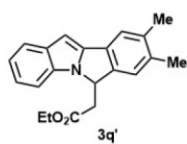
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CARBON_01
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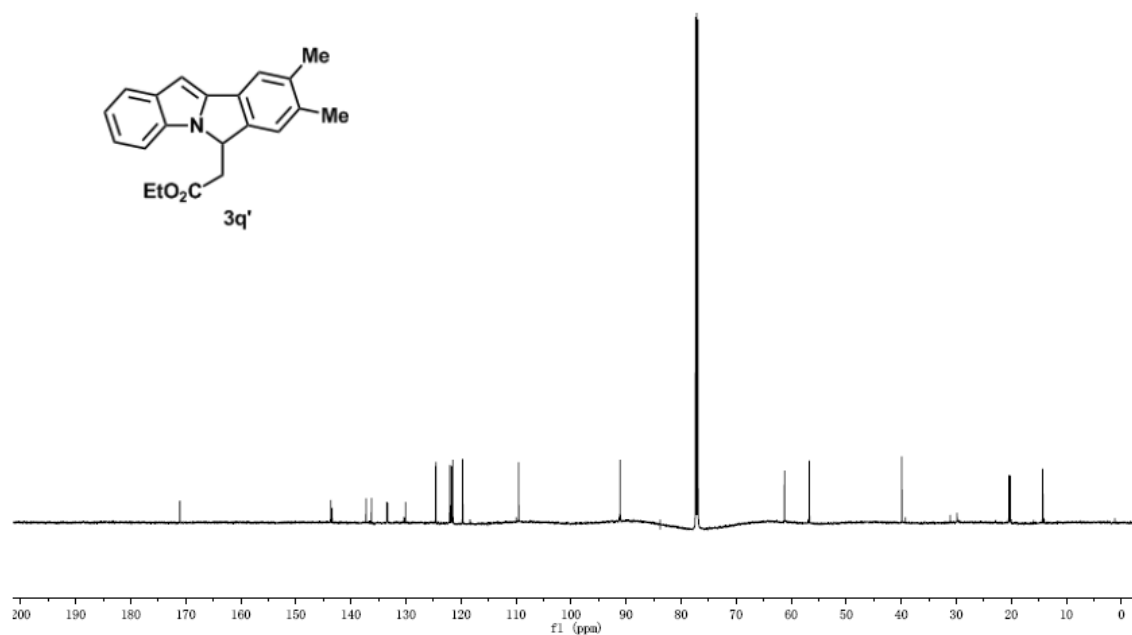
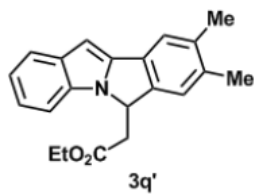


h
WANG
CMW231
a4090291.r



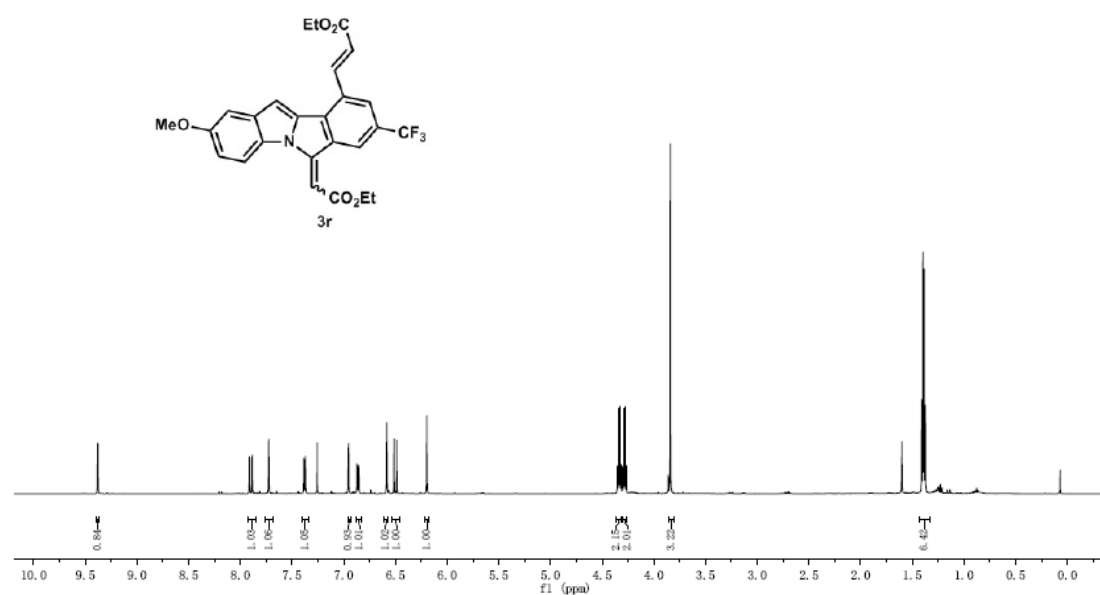
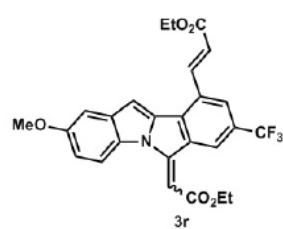
CARBON_01
r/Wang_CMW249

71.06
143.07
142.49
137.25
136.27
133.35
133.33
130.07
124.57
122.04
121.48
121.49
119.69
106.02
91.09
61.21
56.71
39.90
20.43
20.20
14.29



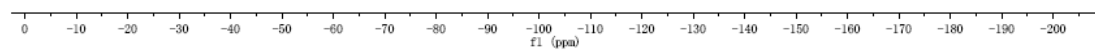
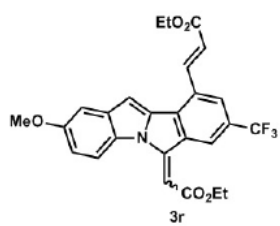
PROTON_01
r/Wang_CMW229

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7.729
7.386
7.371
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6.933
6.923
6.889
6.884
6.814
6.805
6.198
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4.329
4.316
4.312
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1.385
1.373



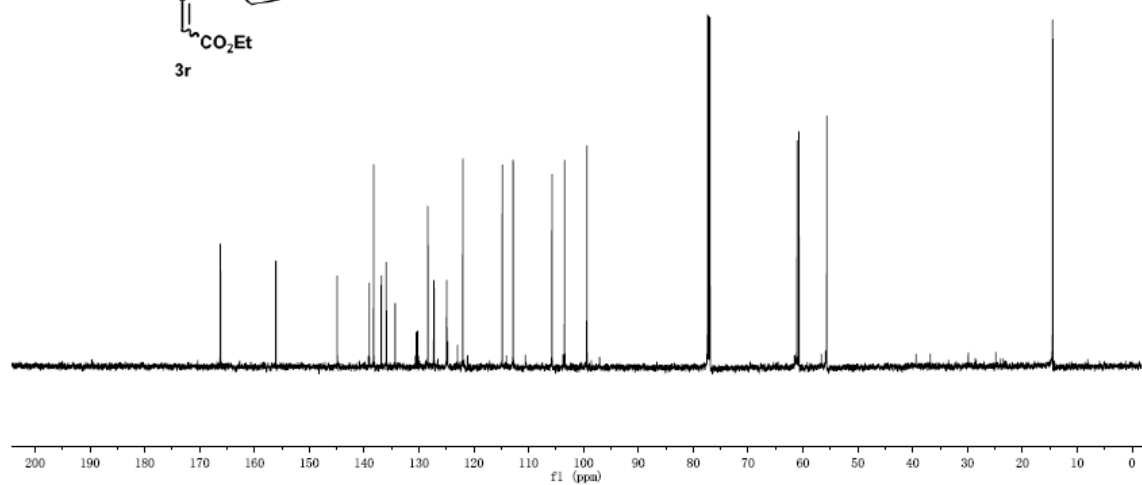
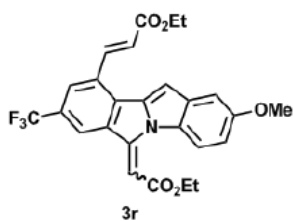
FLUORINE_01
r/Wang_cwv220

62.51



CARBON_01
r/Wang_CMW184

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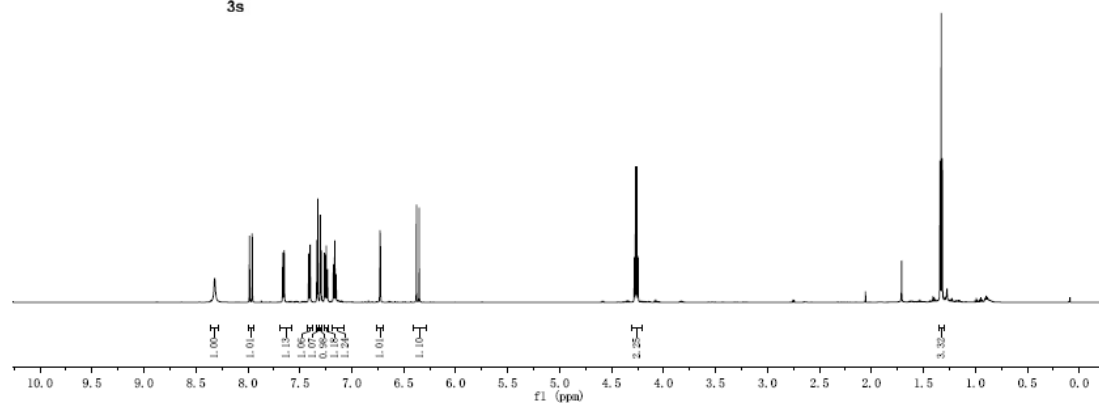
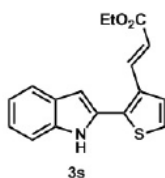


PROTON_01
r/wang_czw213

8.320
7.983
7.968
7.664
7.661
7.401
7.339
7.335
7.334
7.303
7.299
6.778
6.378
6.352

4.281
4.270
4.258
4.246

1.344
1.332
1.317

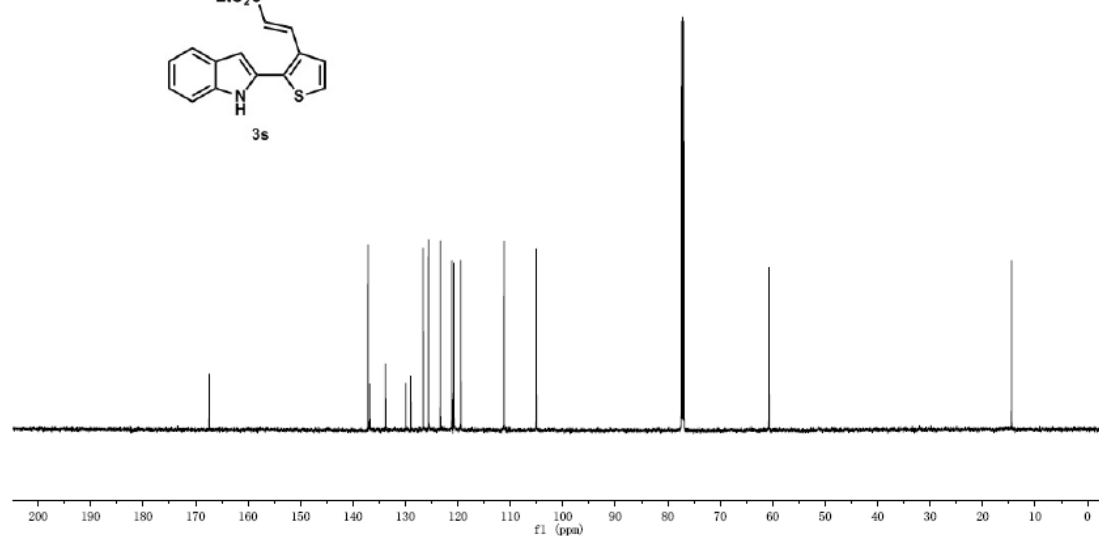
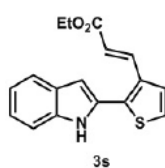


CARBON_01
r/wang_czw213

157.40
137.13
136.78
136.50
136.05
135.96
135.60
135.34
135.15
135.05
134.75
111.21
105.06

60.69

14.48

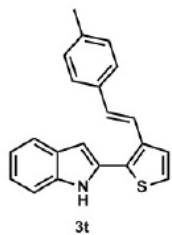


—8.210

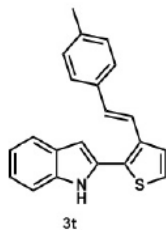
7.684
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7.417
7.408
7.399
7.385
7.182
7.158
6.958

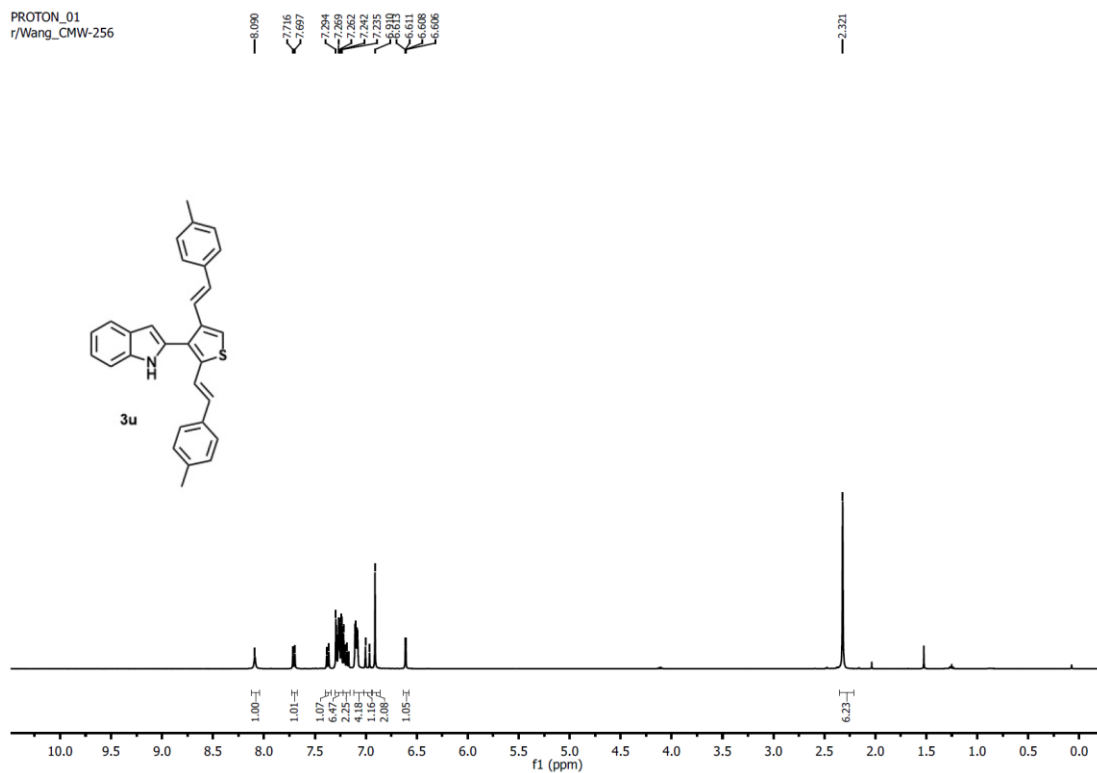
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6.727



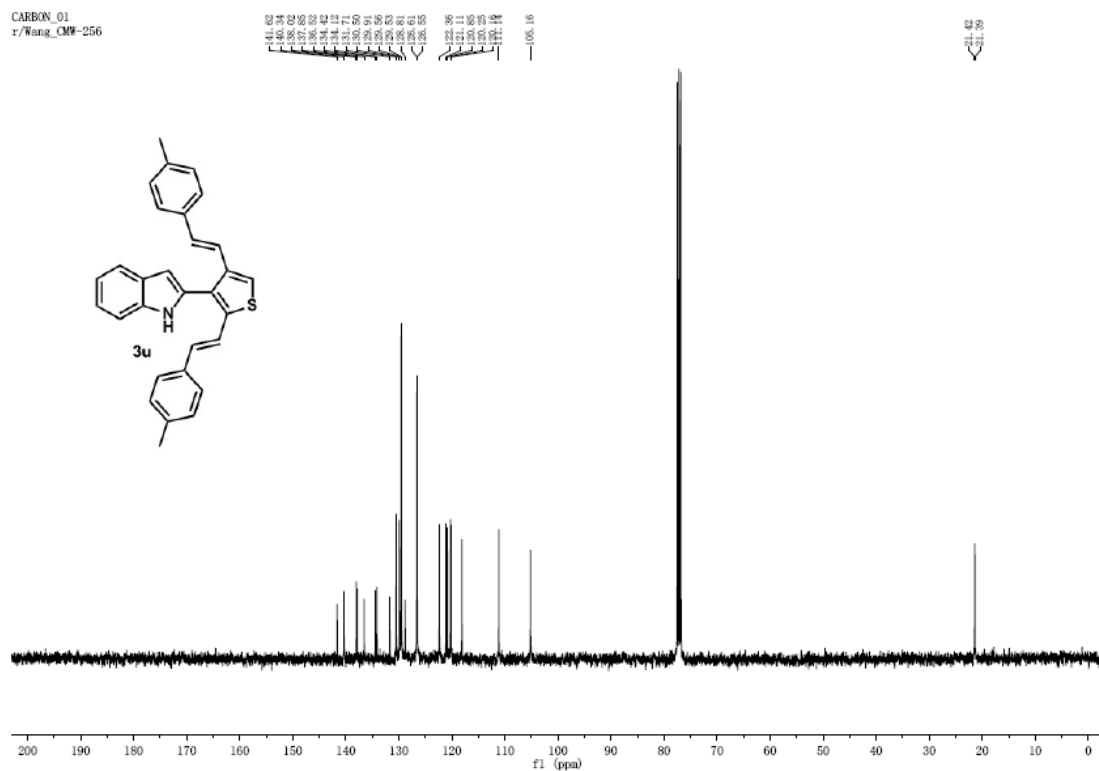
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136.89
136.79
134.64
131.27
130.65
130.60
129.60
129.11
126.56
125.13
122.82
121.14
120.86
120.61



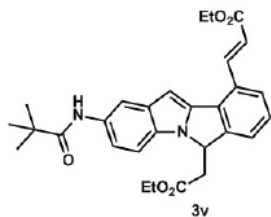
PROTON_01
r/Wang_CMW-256



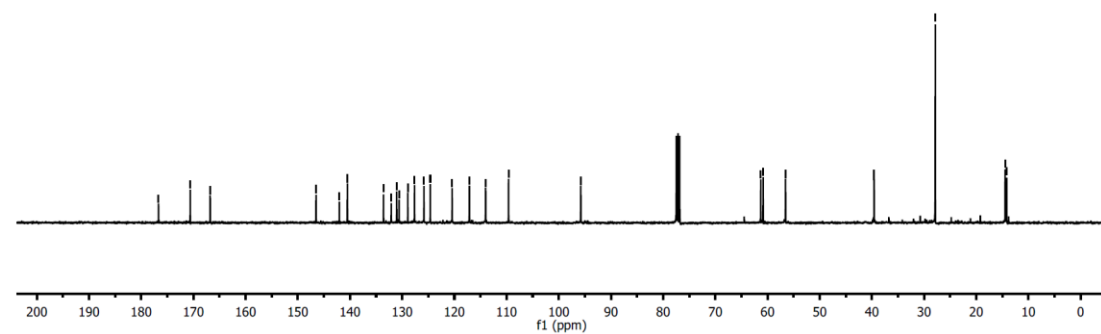
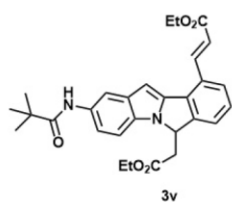
CARBON_01
r/Wang_CMW-256



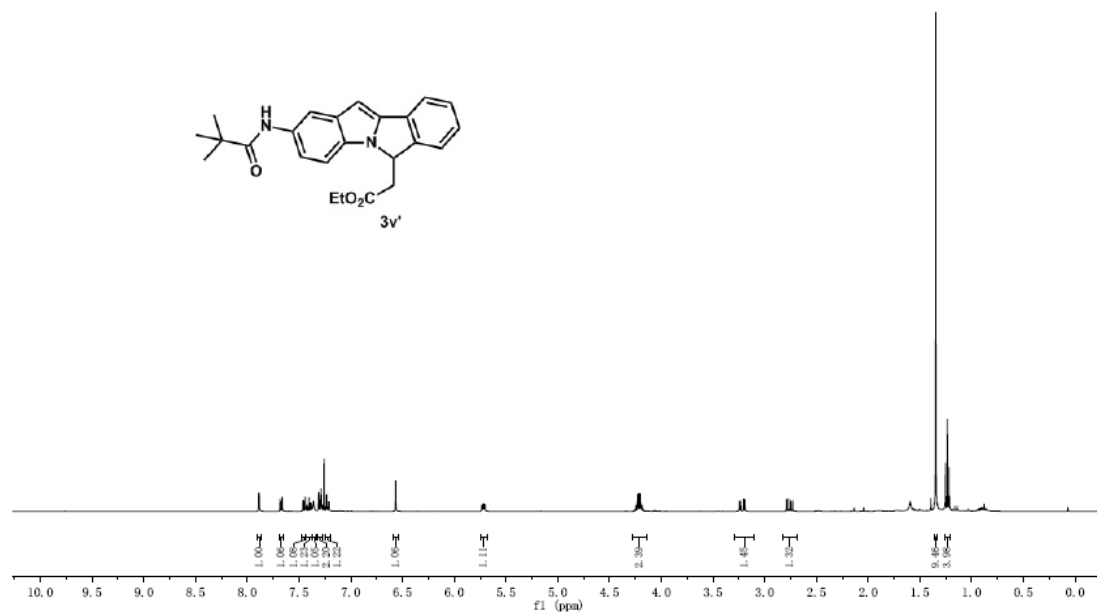
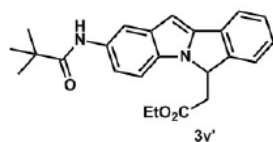
PROTON_01
r/wang_cmw198



CARBON_01
r/wang_cmw198



PROTON_01
r/wang_cw197



CARBON_01
r/wang_cw197

