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Supporting Information

Charge Transfer Complex Enabled Photoreduction of Wittig Phosphonium Salts

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1. General experimental details

Chemicals were purchased from HEOWNS or Bidepharm and used without further purification unless otherwise noted. Solvents were purified using a solvent-purification system (VSPS-8, Vigor) that contained activated alumina and molecular sieves.

Analytical thin layer chromatography was carried out with silica gel pre-coated glass plates (TLC-Silica gel GF254, coating thickness: 0.25 mm) purchased from Xinnuo Chemical (Yantai, China). Chromatographic purification of the products was performed on silica gel 200-300 mesh. Visualization of the developed TLC plates was performed with ultraviolet irradiation (254 nm) or by staining with basic potassium permanganate solution.

High-resolution mass spectra (HRMS) were obtained with the mass analyzer of an orbitrap. The calculated values are based on the most abundant isotope.

IR spectra were taken on a Vertex 70 spectrophotometer and reported as wave numbers (cm⁻).

The SGW X-4 was used to measure the melting point of solids.

The GC-MS TQ8040 was used in the detection of the reaction mixture.

UV-vis absorption spectra were acquired on Mettler Toledo UV-5 spectrophotometer.

¹H- and ¹³C- NMR spectra were recorded at ambient temperature on a Shimadzu Avance 400/500 Spectrometer. The chemical shifts are reported in ppm downfield of tetramethylsilane (TMS) and referenced to residual solvent peaks resonance as the internal standard. The order of citation in parentheses is a) multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd= doublet of doublet, ddd= doublet of doublet of doublet, td = triplet of doublet, m = multiplet), b) coupling constants, c) number of protons. Coupling constants (J) are reported in Hertz (Hz).

All photochemical experiments were performed magnetically stirred in 10 mL glass tubes under a nitrogen atmosphere. Photochemical experiments were performed magnetically stirred in 10 mL glass tubes, sealed with a rubber septum. The tubes were irradiated with blue light (460 nm) using a LED lamp with a power output of 100 W. The distance from the light source to the irradiation vessel is 2 cm to keep the reaction temperature at 45 ± 5 °C.



Figure S1. Blue LEDs employed in the reactions

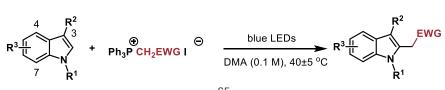
2. General procedures

2.1. General procedure for the synthesis of (alkoxycarbonyl)methylated heteroarenes

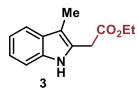
Me NH +	Ph ₃ P CH ₂ CO ₂ Et I —	additive (20 mol%) vent (0.1 M), 40±5 °C	e CO ₂ Et
1 (1.0 equiv.)	2 (2.0 equiv.)	blue LEDs 3	
Entry	Solvent	Additive	Yield (%) ^{a}
1	DMF	/	71
2	DMA	/	81
3	CH ₃ CN	/	NR
4	DMSO	/	trace
5	DCE	/	NR
6	THF	/	39
7	DCM	/	trace
8	toluene	/	NR
9	Xylenes	/	NR
10^{b}	DMA	/	NR
11	DMA	TMEDA	76
12 ^c	DMA	TMEDA	42
13	DMA	DBU	56
14	DMA	K ₂ CO ₃	78

Table S1. Optimization of the reaction conditions

^{*a*}Yield of isolated products after chromatography. ^{*b*}no irradiated with blue light. ^{*c*}100 mol% of TMEDA was used.

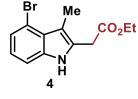


General procedures I: In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with heteroarene (0.15 mmol) and triphenylphosphonium salt (0.3 mmol, 2.0 equiv.) and DMA (1.5 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 $^{\circ}$ C under blue LED (100 W) irradiation for 24 hours. Upon completion, solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product.



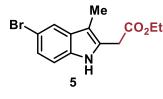
Ethyl 2-(3-methyl-1H-indol-2-yl)acetate (3): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **3** as a light yellow oil (0.122 mmol, 25.6 mg, 81% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.50 (s, 1H),

7.52 (d, J = 7.7 Hz, 1H), 7.33 – 7.28 (m, 1H), 7.20 – 7.06 (m, 2H), 4.20 (q, J = 7.1 Hz, 2H), 3.77 (s, 2H), 2.27 (s, 3H), 1.30 (t, J = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 170.9, 135.7, 126.5, 121.8, 119.2, 118.5, 118.5, 110.7, 109.0, 61.4, 32.0, 14.3, 8.5. **HRMS (ESI)**: m/z [M-H]⁻ calcd for C₁₃H₁₄O₂N⁻: 210.1030; found 210.1028. Analytical data for compound **3** was consistent with the literature.¹



Ethyl 2-(4-bromo-3-methyl-1H-indol-2-yl)acetate (4): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **4** as a yellow solid (0.083 mmol, 24.3 mg, 55% yield). **Melting Point:** 83-85

°C. ¹H NMR (400 MHz, CDCl₃) δ 8.69 (s, 1H), 7.24 – 7.19 (m, 2H), 6.95 – 6.91 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.75 (s, 2H), 2.49 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.7, 136.9, 127.9, 126.6, 123.8, 122.5, 114.3, 110.1, 109.9, 61.6, 31.6, 14.2, 10.9. IR (ATR): 3338, 2918, 1712, 1609, 1451, 1374, 1312, 1175, 1077, 1014, 922 cm⁻¹. HRMS (ESI): *m/z* [M-H]⁻ calcd for C₁₃H₁₃O₂NBr⁻: 294.0135; found 294.0140.



Ethyl 2-(5-bromo-3-methyl-1H-indol-2-yl)acetate (5): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **5** as a yellow solid (0.090 mmol, 26.6 mg, 60%)

yield). **Melting Point:** 73-75 °C. ¹**H NMR (400 MHz, CDCl₃)** δ 8.61 (s, 1H), 7.61 (d, *J* = 1.9 Hz, 1H), 7.21 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.15 (dd, *J* = 8.5, 0.5 Hz, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.75 (s, 2H), 2.20 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 170.7, 134.3, 130.6, 127.9, 124.5, 121.2, 112.5, 112.2, 108.6, 61.6, 31.7, 14.3, 8.4. **IR (ATR)**: 1976, 1706, 1609, 1459, 1378, 1285, 1166, 1056, 894 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₃H₁₃O₂NBr⁻: 294.0135; found 294.0139.

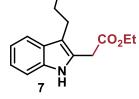
F N H G Me F CO₂Et H CO₂Et

Ethyl 2-(5-fluoro-3-methyl-1H-indol-2-yl)acetate (6)

Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded $\bf 6$ as a yellow wax (0.089 mmol, 20.8 mg, 59%)

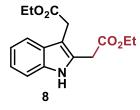
yield). ¹**H NMR (400 MHz, CDCl₃)** δ 8.54 (s, 1H), 7.20 (dd, *J* = 8.8, 4.4 Hz, 1H), 7.12 (dd, *J* = 9.4, 2.3 Hz, 1H), 6.90 – 6.85 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.76 (s, 2H), 2.20 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 170.7, 157.8 (d, *J* = 235.3 Hz), 129.3 (d, *J* = 9.6 Hz), 111.3, 111.2, 109.9 (d, *J* = 26.3 Hz), 109.2 (d, *J* = 4.7 Hz), 103.6, 103.3, 61.5, 31.9, 14.2, 8.5. **IR** (**ATR**): 3378, 2920, 1720, 1661, 1919, 1480, 1283, 1162, 1022, 936, 796 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₃H₁₃O₂F⁻: 234.0936; found 234.0935.

Ethyl 2-(3-(2-bromoethyl)-1H-indol-2-yl)acetate (7)



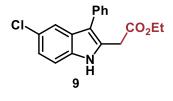
Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **7** as a light yellow oil (0.068 mmol, 21.0 mg, 45% yield). ¹H NMR (500 MHz,

CDCl₃) δ 8.68 (s, 1H), 7.52 (d, *J* = 8.7 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.20 – 7.15 (m, 1H), 7.12 -7.10 (m, 1H), 3.81 (d, *J* = 6.9 Hz, 2H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.71 (t, *J* = 7.5 Hz, 1H), 3.38 – 3.30 (m, 2H), 3.22 – 3.16 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 170.5, 135.7, 128.0, 127.6, 122.2, 119.8, 118.1, 111.1, 110.8, 61.6, 32.9, 32.0, 28.4, 14.3. **IR (ATR)**: 3391, 2974, 1720, 1600, 1451, 1372, 1304, 1163, 1023, 932, 739 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₄H₁₇BrNO₂: 310.0437; found 310.0432.



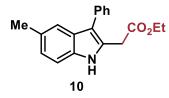
Diethyl 2,2'-(1H-indole-2,3-diyl)diacetate (8): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **8** as a colorless oil (0.123 mmol, 35.5 mg, 82% yield). ¹H NMR (400 MHz,

CDCl₃) δ 8.78 (s, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.31 (d, *J* = 7.9 Hz, 1H), 7.18 – 7.09 (m, 2H), 4.20 q, *J* = 7.1 Hz, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.85 (s, 2H), 3.70 (s, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 171.1, 170.7, 135.5, 128.4, 128.0, 122.1, 119.8, 118.6, 111.0, 106.2, 61.5, 60.9, 31.9, 30.4, 14.3, 14.3. **IR (ATR)**: 3340, 2980, 1716, 1453, 1373, 1306, 1158, 1024, 950, 735 cm⁻¹ **HRMS (ESI)**: *m*/*z* [M-H]⁻ calcd for C₁₆H₁₈O₄N⁻: 288.1241; found 288.1245.



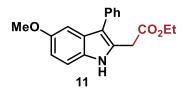
Ethyl 2-(5-chloro-3-phenyl-1H-indol-2-yl)acetate (9): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **9** as a light yellow oil (0.075 mmol, 23.5 mg, 50%)

yield). ¹**H NMR (400 MHz, CDCl₃)** δ 8.98 (s, 1H), 7.59 (d, *J* = 2.0 Hz, 1H), 7.49 – 7.43 (m, 4H), 7.36 – 7.32 (m, 1H), 7.29 (d, *J* = 8.6 Hz, 1H), 7.15 (dd, *J* = 8.6, 2.0 Hz, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.88 (s, 2H), 1.29 (t, *J* = 7.2 Hz, 3H). ¹³**C NMR (126 MHz, CDCl₃)** δ 170.9, 134.1, 134.0, 129.9, 129.7, 128.9, 128.5, 126.8, 126.0, 122.7, 118.8, 116.1, 112.1, 61.7, 32.1, 14.3. **IR (ATR)**: 3346, 2981, 1721, 1603, 1446, 1370, 1290, 1200, 1062, 1024, 799 cm⁻¹ **HRMS (ESI)**: *m/z* [M+Na]⁺ calcd for C₁₈H₁₆ClNO₂Na⁺: 336.0762.; found 336.0747.



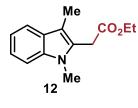
Ethyl 2-(5-methyl-3-phenyl-1H-indol-2-yl)acetate (10): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **10** as a colorless oil (0.102 mmol, 29.9 mg, 68%

yield). ¹**H NMR (400 MHz, CDCl₃)** δ 8.79 (s, 1H), 7.51 – 7.44 (m, 5H), 7.36 – 7.27 (m, 2H), 7.08 – 7.00 (m, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 3.88 (s, 2H), 2.43 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 171.0, 134.8, 134.1, 129.8, 129.5, 128.7, 127.6, 127.1, 126.3, 123.9, 118.9, 115.9, 110.7, 61.5, 32.3, 21.6, 14.3. **IR (ATR)**: 3373, 2918, 1720, 1602, 1448, 1374, 1300, 1187, 1022, 796 cm⁻¹ **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₉H₁₈O₂N⁻: 292.1343; found 292.1349.



Ethyl 2-(5-methoxy-3-phenyl-1H-indol-2-yl)acetate (11): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **11** as a yellow oil (0.084 mmol, 26.0 mg, 56%

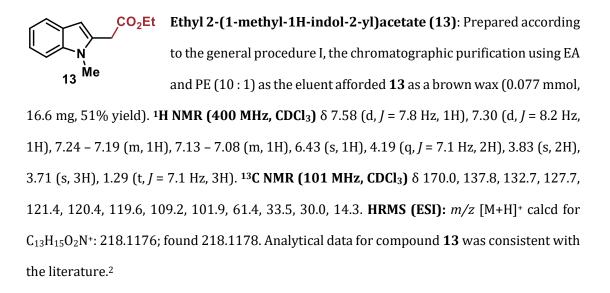
yield). ¹**H NMR (500 MHz, CDCl₃)** δ 8.80 (s, 1H), 7.49 – 7.46 (m, 4H), 7.36 – 7.30 (m, 1H), 7.27 – 7.23 (m, 1H), 7.10 (s, 1H), 6.86 (d, *J* = 8.8 Hz, 1H), 4.20 (q, *J* = 7.0 Hz, 2H), 3.86 (s, 2H), 3.80 (s, 3H), 1.28 (t, *J* = 6.9 Hz, 3H). ¹³**C NMR (126 MHz, CDCl₃)** δ 171.0, 154.7, 134.8, 130.9, 129.7, 128.8, 127.8, 126.4, 116.2, 112.5, 111.8, 101.2, 61.6, 56.1, 32.4, 14.3. **IR (ATR)**: 2979, 1721, 1594, 1447, 1402, 1205, 1145, 1068, 799 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₉H₁₈O₃N⁻: 308.1292; found 308.1296.



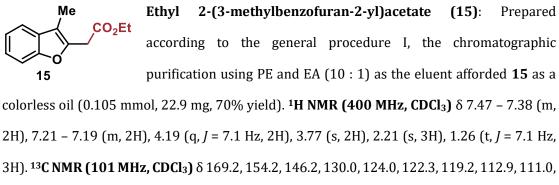
Ethyl 2-(1,3-dimethyl-1H-indol-2-yl)acetate (12): Prepared according to the general procedure I, the chromatographic purification using EA and PE (10 : 1) as the eluent afforded **12** as a colorless oil (0.128 mmol, 29.5 mg, 85% yield). ¹H NMR (400 MHz,

CDCl₃) δ 7.56 – 7.54 (m, 1H), 7.29 – 7.27 (m, 1H), 7.24 – 7.19 (m, 1H), 7.13 – 7.09 (m, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 2H), 3.72 (s, 3H), 2.32 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 170.2, 137.0, 128.9, 128.1, 121.5, 118.9, 118.7, 109.1, 109.0, 61.3, 31.2, 30.0,

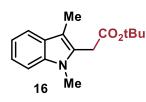
14.3, 9.0. **HRMS (ESI)**: m/z [M+H]⁺ calcd for C₁₄H₁₈O₂N⁺: 232.1332; found 232.1335. Analytical data for compound **12** was consistent with the literature.²



brown wax (0.078 mmol, 17.9 mg, 52% yield). ¹**H NMR (400 MHz, CDCl**₃) δ 9.00 (s, 1H), 7.48 – 7.43 (m, 2H), 7.37 – 7.31 (m, 2H), 7.22 – 7.15 (m, 1H), 6.43 – 6.39 (m, 1H), 6.08 – 6.03 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.70 (s, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 171.2, 132.8, 132.2, 128.9, 126.1, 124.6, 123.7, 109.2, 105.9, 61.3, 33.4, 14.3. **IR (ATR)**: 3344, 2918, 1714, 1598, 1506, 1459, 1369, 1221, 1160, 1028, 752 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₄H₁₄O₂N⁻: 228.1030; found 228.1030.

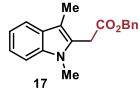


61.4, 32.9, 14.3, 8.1. **IR (ATR)**: 2922, 1730, 1605, 1512, 1462, 1368, 1242, 1161, 1025, 930, 741 cm⁻¹. **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₁₃H₁₃O₃⁻: 217.0870; found 217.0865.



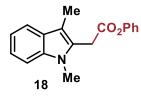
Tert-butyl2-(1,3-dimethyl-1H-indol-2-yl)acetate(16):Prepared according to the general procedure I, the chromatographicpurification using PE and EA (10 : 1) as the eluent afforded 16 as acolorless oil (0.096 mmol, 24.9 mg, 64% yield). 1H NMR (400 MHz,

CDCl₃) δ 7.55 (d, *J* = 7.8 Hz, 1H), 7.31 – 7.18 (m, 2H), 7.14 – 7.08 (m, 1H), 3.73 – 3.71 (m, 5H), 2.33 (s, 3H), 1.45 (s, 9H). ¹³**C NMR (101 MHz, CDCl₃)** δ 169.4, 137.0, 129.5, 128.2, 121.3, 118.8, 118.6, 108.9, 108.8, 81.5, 32.5, 30.0, 28.1, 8.9. **IR (ATR)**: 2973, 1783, 1465, 1366, 1321, 1251, 1203, 1143, 1070, 727 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₁₆H₂₂O₂N⁺: 260.1645; found 260.1649.



Benzyl 2-(1,3-dimethyl-1H-indol-2-yl)acetate (17): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **17** as a colorless oil (0.119 mmol, 34.7 mg, 79% yield). **¹H NMR (400 MHz**,

CDCl₃) δ 7.57 (d, *J* = 8.5 Hz, 1H), 7.36 – 7.22 (m, 7H), 7.16 – 7.11 (m, 1H), 5.16 (s, 2H), 3.87 (s, 2H), 3.68 (s, 3H), 2.34 (s, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 169.9, 137.0, 135.8, 128.7, 128.6, 128.4, 128.2, 128.1, 121.6, 118.9, 118.7, 109.2, 109.0, 67.0, 31.2, 30.0, 9.0. **IR (ATR)**, 2977, 1732, 1463, 1375, 1235, 1152, 1066, 898 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₁₉H₂₀O₂N⁺: 294.1489; found 294.1492.



Phenyl 2-(1,3-dimethyl-1H-indol-2-yl)acetate (18): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **18** as a colorless oil (0.117 mmol, 32.6 mg, 78% yield). **¹H NMR (400 MHz,**

CDCl₃) δ 7.59 – 7.54 (m, 1H), 7.38 – 7.28 (m, 3H), 7.25 – 7.19 (m, 2H), 7.16 – 7.02 (m, 3H), 4.04 (s, 2H), 3.79 (s, 3H), 2.39 (s, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 168.5, 150.7, 137.1, 129.5,

128.1, 128.1, 126.1, 121.8, 121.5, 119.0, 118.8, 109.6, 109.0, 31.3, 30.1, 9.0. **IR (ATR)**: 2918, 1745, 1465, 1372, 1250, 1071, 1017 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₁₈H₁₈O₂N⁺: 280.1332; found 280.1335.

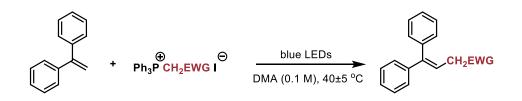
Me
 2-(1,3-Dimethyl-1H-indol-2-yl)acetonitrile (19): Prepared according to the general procedure I, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded 19 as a colorless oil (0.092 mmol, 16.8 mg, 61% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.57 – 7.53 (m, 1H), 7.31 – 7.24 (m, 2H), 7.17 – 7.12 (m, 1H), 3.83 (s, 2H), 3.76 (s, 3H), 2.31 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 137.2, 127.8, 123.6, 122.6, 119.5, 119.1, 116.2, 109.8, 109.1, 30.0, 13.9, 8.9. IR (ATR): 2964, 1643, 1464, 1399, 1256, 1069, 796 cm⁻¹. HRMS (ESI): m/z [M+H]⁺ calcd for C₁₂H₁₃N₂⁺: 185.1073; found 185.1077.

2.2. One-pot synthesis of (alkoxycarbonyl)methylated 3-methylindole

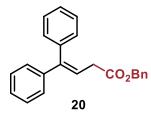
$$\begin{array}{c} & & & \\ & & & & \\ & & & \\ &$$

In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with 3-methylindole (0.15 mmol), triphenylphosphine (0.3 mmol, 2.0 equiv.), ethyl bromoacetate (0.3 mmol, 2.0 equiv.), NaI (0.03 mmol, 0.2 equiv.), and DMA (1.5 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 $^{\circ}$ C under blue LED (100 W) irradiation for 24 hours. Upon completion, solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate PE and EA (10 : 1) as eluent to afford the desired product **3** (17.9 mg, 0.083 mmol, 55%).

2.3. General procedure for the synthesis of (alkoxycarbonyl)methylated 1,1diphenylethylenes

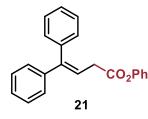


General procedures II: In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with 1,1-diphenylethylene (0.15 mmol), triphenylphosphonium salt (0.3 mmol, 2.0 equiv.) and DMF (1.5 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 °C under blue LED (100 W) irradiation for overnight as monitored by TLC. Upon completion, solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product.



Benzyl 4,4-diphenylbut-3-enoate (20): Prepared according to the general procedure II, the chromatographic purification using PE and EA (50 : 1) as the eluent afforded **20** as a white solid. (0.098 mmol, 32.0 mg, 65% yield). **Melting Point:** 63-65 °C. ¹H NMR (400

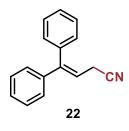
MHz, CDCl₃) δ 7.40 – 7.31 (m, 8H), 7.30 – 7.24 (m, 5H), 7.20 – 7.17 (m, 2H), 6.30 (t, *J* = 7.4 Hz, 1H), 5.16 (s, 2H), 3.23 (d, *J* = 7.4 Hz, 2H). ¹³**C NMR (101 MHz, CDCl₃)** δ 171.8, 145.0, 142.0, 139.3, 136.0, 129.9, 128.7, 128.5, 128.4, 128.4, 128.3, 127.6, 127.6, 120.3, 66.6, 35.6. **IR (ATR)**: 3668, 2975, 2912, 1719, 1381, 1325, 1186, 1069, 808, 757 cm⁻¹ **HRMS (ESI)**: *m/z* [M-H]⁻ calcd for C₂₃H₁₉O₂⁻: 327.1391; found 327.1397.



Phenyl 4,4-diphenylbut-3-enoate (21): Prepared according to the general procedure II, the chromatographic purification using PE and EA (50 : 1) as the eluent afforded 21 as a colorless oil (0.111 mmol, 34.9 mg, 74% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.34

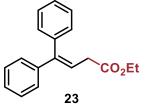
(m, 5H), 7.31 – 7.27 (m, 5H), 7.27 – 7.21 (m, 3H), 7.12 – 7.07 (m, 2H), 6.36 (t, J = 7.4 Hz, 1H),

3.42 (d, J = 7.4 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 170.5, 150.8, 145.5, 141.9, 139.3, 129.9, 129.5, 128.6, 128.3, 127.7, 127.7, 127.6, 126.0, 121.6, 119.7, 35.6. IR (ATR): 2969, 1729, 1643, 1592, 1449, 1381, 1314, 1261, 1167, 1078, 1019, 754 cm⁻¹. HRMS (ESI): m/z [M-H]⁻ calcd for $C_{22}H_{17}O_2$: 313.1234; found 213.1240.



4,4-Diphenylbut-3-enenitrile (22): Prepared according to the general procedure II, the chromatographic purification using PE and EA (50 : 1) as the eluent afforded **22** as a white solid (0.090 mmol, 19.7 mg, 60% yield). **Melting Point:** 94-96 °C. ¹**H NMR (400 MHz, CDCl₃)** δ 7.46 – 7.35 (m, 3H), 7.30 – 7.16 (m, 7H), 6.03 (t, *J* = 7.4 Hz, 1H), 3.14 (d, *J* =

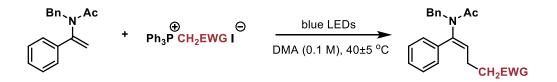
7.9 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 147.7, 140.8, 138.1, 129.5, 128.9, 128.5, 128.3, 128.2, 127.5, 118.2, 115.6, 18.5. HRMS (ESI): *m/z* [M-H]⁻ calcd for C₁₆H₁₂: 218.0975; found 218.0975.
Analytical data for compound 22 was consistent with the literature.³



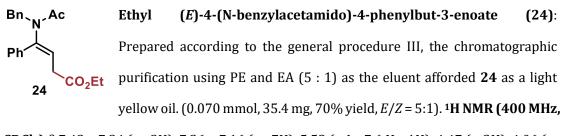
Ethyl 4,4-diphenylbut-3-enoate (23): Prepared according to the general procedure II, the chromatographic purification using PE and EA (50 : 1) as the eluent afforded 23 as a colorless oil (0.111 mmol, 29.5 mg, 74% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.32 (m,

3H), 7.28 – 7.24 (m, 5H), 7.21 – 7.16 (m, 2H), 6.26 (t, *J* = 7.4 Hz, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.15 (d, *J* = 7.4 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 171.9, 144.6, 142.0, 139.2, 129.8, 128.3, 128.1, 127.4, 127.4, 120.5, 60.7, 35.5, 14.2. Analytical data for compound **23** was consistent with the literature.³

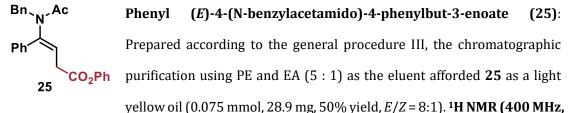
2.4. General procedure for synthesis of (alkoxycarbonyl)methylated enamides



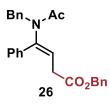
General procedures III: In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with enamide (0.15 mmol), triphenylphosphonium salt (0.3 mmol, 2.0 equiv.) and DMA (1.5 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 $^{\circ}$ C under blue LED (100 W) irradiation for overnight as monitored by TLC. Upon completion, solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product.



CDCl₃) δ 7.42 – 7.34 (m, 3H), 7.26 – 7.16 (m, 7H), 5.52 (t, *J* = 7.6 Hz, 1H), 4.47 (s, 2H), 4.06 (q, *J* = 7.1 Hz, 2H), 3.24 – 3.13 (m, 2H), 2.26 (s, 3H), 1.18 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 171.0, 170.7, 141.5, 137.4, 134.3, 129.2, 129.0, 128.9, 128.8, 128.3, 127.3, 123.3, 61.0, 49.0, 34.2, 22.4, 14.2. **IR (ATR)**: 2979, 1731, 1649, 1384, 1257, 1162, 1066, 864 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₂₁H₂₄O₃N⁺: 338.1751; found 338.1750.

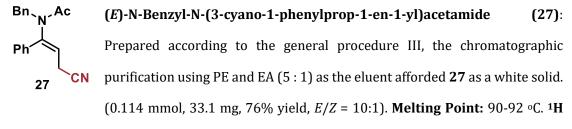


CDCl₃) δ 7.47 – 7.41 (m, 3H), 7.39 – 7.18 (m, 10H), 7.00 – 6.93 (m, 2H), 5.62 (t, *J* = 7.6 Hz, 1H), 4.51 (s, 2H), 3.45 (d, *J* = 7.6 Hz, 2H), 2.30 (s, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 171.0, 169.4, 150.5, 142.4, 137.4, 134.2, 129.6, 129.4, 129.0, 129.0, 128.9, 128.4, 127.4, 126.2, 122.4, 121.4, 49.1, 34.2, 22.5. **IR (ATR)**: 2975, 1754, 1648, 1488, 1440, 1387, 1316, 1193, 1131, 1068, 978 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₂₅H₂₄O₃N⁺: 386.1751; found 386.1750.



Benzyl (*E*)-4-(N-benzylacetamido)-4-phenylbut-3-enoate (26): Prepared according to the general procedure III, the chromatographic purification using PE and EA (5 : 1) as the eluent afforded 26 as a light yellow oil (0.102 mmol, 40.7 mg, 68% yield, E/Z = 5:1). ¹H NMR (400

MHz, **CDCl**₃) δ 7.40 – 7.31 (m, 6H), 7.29 – 7.15 (m, 9H), 5.54 (t, *J* = 7.6 Hz, 1H), 5.06 (s, 2H), 4.48 (s, 2H), 3.25 (d, *J* = 7.6 Hz, 2H), 2.21 (s, 3H). ¹³**C NMR (101 MHz, CDCl**₃) δ 171.0, 170.6, 141.8, 137.5, 135.5, 134.3, 129.3, 129.0, 128.9, 128.9, 128.7, 128.6, 128.5, 128.4, 123.0, 100.0, 66.9, 49.1, 34.2, 22.4. **IR (ATR)**: 2977, 1732, 1648, 1493, 1384, 1260, 1154, 1069, 979 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₂₆H₂₆O₃N⁺: 400.1907; found 400.1907.



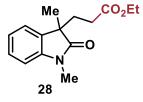
NMR (400 MHz, CDCl₃) δ 7.44 (s, 3H), 7.33 – 7.09 (m, 7H), 5.33 – 5.29 (m, 1H), 4.50 (s, 2H), 3.16 (d, *J* = 7.7 Hz, 2H), 2.21 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.4, 144.5, 137.0, 133.3, 130.1, 129.3, 128.9, 128.7, 128.6, 127.7, 117.9, 116.8, 49.3, 22.5, 17.5. IR (ATR): 2971, 1731, 1651, 1384, 1264, 1066, 922 cm⁻¹. HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₉H₁₉O₂N⁺: 291.1492; found 291.1493.

2.5. General procedure for the synthesis of (alkoxycarbonyl)methylated oxindoles



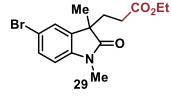
General procedures IV: In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with N-phenyl acrylamides (0.15 mmol), triphenylphosphonium salt (0.3 mmol, 2.0 equiv.) and DMA (1.5 mL). The vial was closed and

removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 °C under blue LED (100 W) irradiation for overnight as monitored by TLC. Upon completion, solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired product.



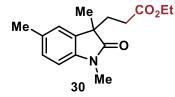
Ethyl 3-(1,3-dimethyl-2-oxoindolin-3-yl)propanoate (28): Prepared according to the general procedure IV, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **28** as a colorless oil (0.055 mmol, 14.4 mg, 55% yield). ¹H

NMR (400 MHz, CDCl₃) δ 7.27 – 7.22 (m, 1H), 7.16 (d, *J* = 7.8 Hz, 1H), 7.07 – 7.02 (m, 1H), 6.82 (d, *J* = 7.7 Hz, 1H), 4.04 – 3.91 (m, 2H), 3.19 (s, 3H), 2.25 – 2.00 (m, 3H), 1.90 – 1.77 (m, 1H), 1.36 (s, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 180.0, 172.8, 143.3, 133.0, 128.1, 122.8, 122.7, 108.1, 60.5, 47.7, 33.1, 29.7, 26.2, 23.7, 14.2. **IR (ATR)**: 2974, 1706, 1609, 1460, 1376, 1302, 1249, 1175, 1103, 1027, 750 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₁₅H₂₀O₃N⁺: 262.1438; found 262.1440.



Ethyl3-(5-bromo-1,3-dimethyl-2-oxoindolin-3-yl)propanoate(29):Preparedaccordingtothegeneralprocedure IV, the chromatographic purification using PE and EA(10:1) as the eluent afforded 29 as a colorless oil (0.113 mmol,

38.1 mg, 75% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.40 – 7.37 (m, 1H), 7.28 – 7.26 (m, 1H), 6.72 – 6.68 (m, 1H), 4.02 – 3.97 (m, 2H), 3.17 (s, 3H), 2.25 – 1.98 (m, 3H), 1.92 – 1.80 (m, 1H), 1.36 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 179.3, 172.5, 142.3, 135.1, 131.0, 126.1, 115.4, 109.6, 60.5, 47.9, 32.9, 29.6, 26.3, 23.5, 14.1. IR (ATR): 2974, 1712, 1604, 1479, 1345, 1248, 1177, 1109, 1030, 810 cm⁻¹. HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₅H₁₉O₃NBr⁺: 340.1543; found 340.0544.



Ethyl 3-(1,3,5-trimethyl-2-oxoindolin-3-yl)propanoate
(30): Prepared according to the general procedure IV, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded 30 as a colorless oil (0.102 mmol, 28.1 mg, 68%)

yield). ¹**H NMR (400 MHz, CDCl₃)** δ 7.04 (d, *J* = 7.9 Hz, 1H), 6.97 (s, 1H), 6.70 (d, *J* = 7.9 Hz, 1H), 4.04 – 3.91 (m, 2H), 3.16 (s, 3H), 2.31 (s, 3H), 2.25 – 1.97 (m, 3H), 1.88 – 1.79 (m, 1H), 1.34 (s, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 180.0, 172.9, 141.0, 133.1, 132.3, 128.3, 123.6, 107.9, 60.4, 47.7, 33.1, 29.8, 26.3, 23.7, 21.2, 14.2. **IR (ATR)**: 2977, 1704, 1611, 1484, 1377, 1249, 1180, 1060, 889 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]⁺ calcd for C₁₆H₂₂O₃N⁺: 276.1594; found 276.1596.



Ethyl 3-(3-benzyl-1,5-dimethyl-2-oxoindolin-3-yl)propanoate (31): Prepared according to the general procedure IV, the chromatographic purification using PE and EA (10 : 1) as the eluent afforded **31** as a colorless oil (0.089 mmol, 29.8 mg, 59% yield). ¹H

NMR (400 MHz, CDCl₃) δ 7.19 – 7.11 (m, 2H), 7.06 – 6.97 (m, 4H), 6.79 (d, *J* = 7.8 Hz, 2H), 6.56 (d, *J* = 7.5 Hz, 1H), 4.04 – 3.91 (m, 2H), 3.14 (d, *J* = 12.9 Hz, 1H), 3.01 (d, *J* = 12.9 Hz, 1H), 2.93 (s, 3H), 2.44 – 2.20 (m, 2H), 2.10 – 1.99 (m, 1H), 1.87 – 1.76 (m, 1H), 1.14 (t, *J* = 7.2 Hz, 3H). ¹³**C NMR (101 MHz, CDCl₃)** δ 177.4, 171.7, 142.8, 134.5, 129.0, 128.8, 127.1, 126.5, 125.5, 122.7, 121.2, 106.8, 59.4, 53.0, 43.2, 30.6, 28.6, 24.8, 13.1. **IR (ATR)**: 2979, 1705, 1608, 1459, 1379, 1250, 1169, 1056, 872 cm⁻¹. **HRMS (ESI)**: *m/z* [M+H]+ calcd for C₂₁H₂₄O₃N+: 338.1751; found 338.1751.

3. Mechanistic studies

3.1. UV/vis absorption spectrometry.

UV/vis absorption spectra were recorded using DMA as solvent in 1 cm path quartz cuvettes using a Mettler Toledo UV-5 UV/Vis spectrometer.

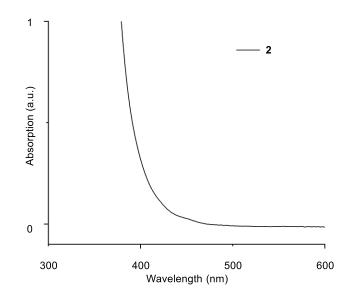
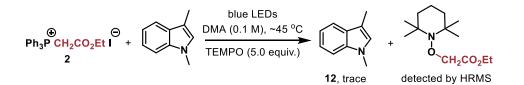
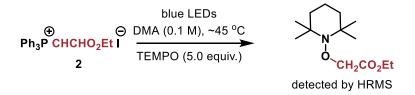


Figure S2: UV/vis spectrum of 2 (recorded 0.2 mmol in 1.0 mL DMA)

3.2 Control experiments

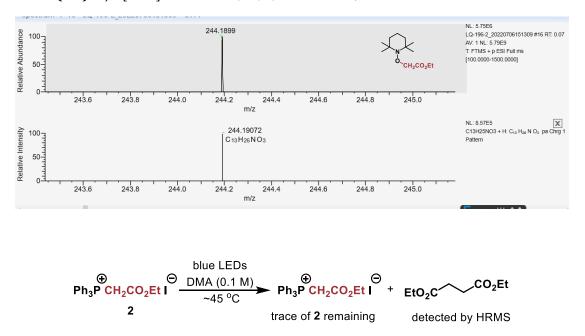


In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with 1,3-dimethylindole (0.15 mmol, 1.0 equiv.), triphenylphosphonium salt **2** (0.3 mmol, 2.0 equiv.), TEMPO (0.75 mmol, 5.0 equiv.) and DMA (1.5 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 $^{\circ}$ C under blue LED (100 W) irradiation for overnight. The desired product was observed with trace yield and the trapped radical species was detected by HRMS.



In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with triphenylphosphonium salt **2** (0.1 mmol, 1.0 equiv.), TEMPO (0. 5 mmol, 5.0 S19

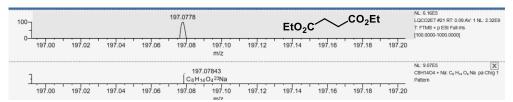
equiv.) and DMA (1.0 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 \pm 5 $^{\circ}$ C under blue LED (100 W) irradiation for overnight. The trapped radical species was detected by HRMS.



HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₁₃H₂₆O₃N⁺: 244.1907; found 244.1899.

In a nitrogen-filled glove box, a 10-mL vial equipped with a magnetic stirring bar was charged sequentially with triphenylphosphonium salt **2** (0.1 mmol) and DMA (1.0 mL). The vial was closed and removed from the glove box. The resulting mixture was allowed to stir at 40 ± 5 $^{\circ}$ C under blue LED (100 W) irradiation for overnight. Trace of **2** was observed from the reaction mixture, and the radical dimerization product (diethyl succinate) was detected by HRMS analysis.

HRMS (ESI): *m*/*z* [M+H]⁺ calcd for C₈H₁₄O₄Na⁺: 197.0784; found 197.0778.



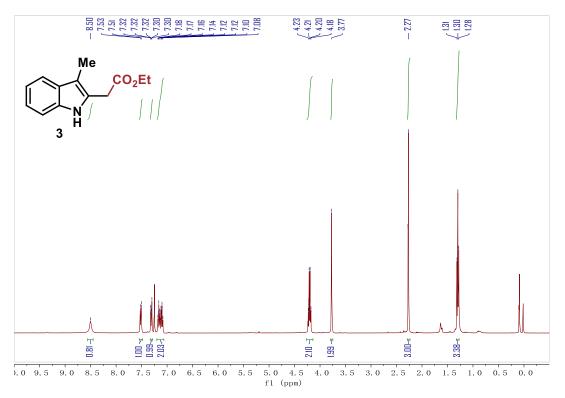
4. References

- (1) J. Huang, Zhao, Y. Liu, W. Cao, X. Wu, Org. Lett. 2013, 15, 4338–4341.
- (2) H.-L. Jang, H. T. Kim, E. J. Cho, J. M. Joo, Asian J. Org. Chem, 2015, 4, 1386-1391.

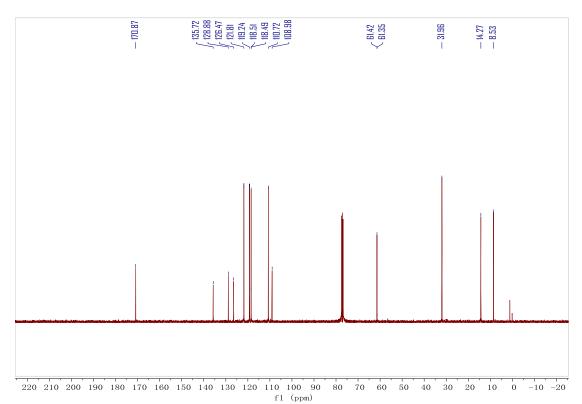
(3) N. Kato, T. Nanjo, Y. Takemoto, *ACS Catal.* 2022, **12**, 7843–7849.

5. NMR Spectra

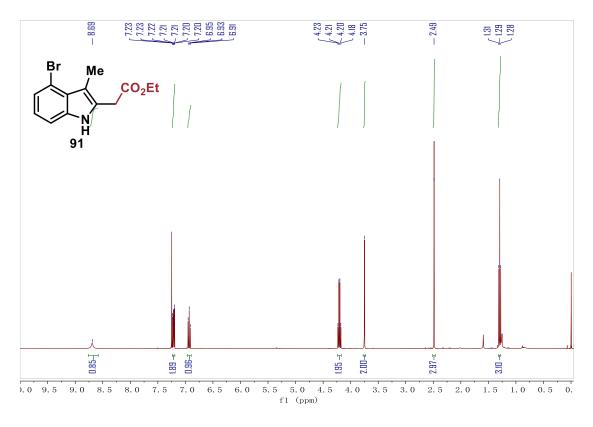
¹H NMR of compound 3 (400 MHz in CDCl₃)



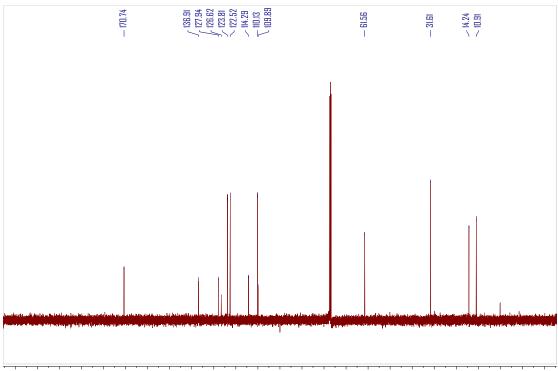
¹³C NMR of compound 3 (101 MHz in CDCl₃)



¹H NMR of compound 4 (400 MHz in CDCl₃)

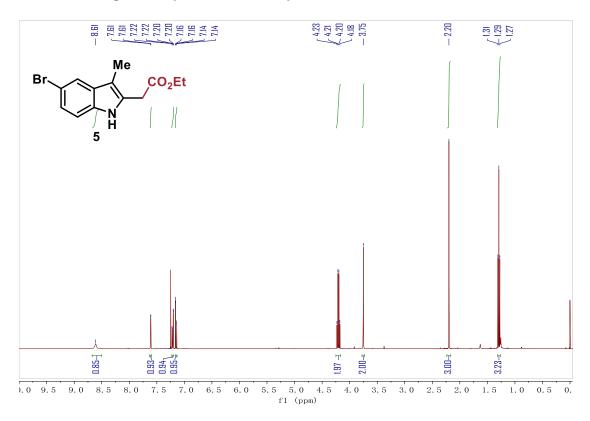


¹³C NMR of compound 4 (101 MHz in CDCl₃)

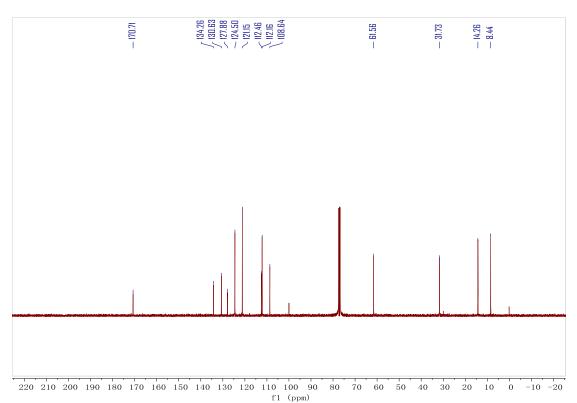


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 f1 (ppm)

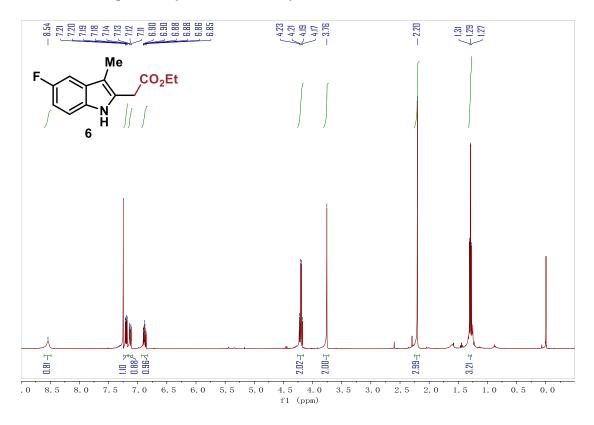
¹H NMR of compound 5 (400 MHz in CDCl₃)



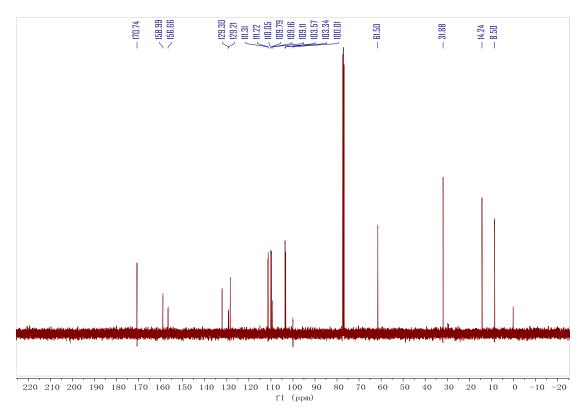
¹³C NMR of compound 5 (101 MHz in CDCl₃)



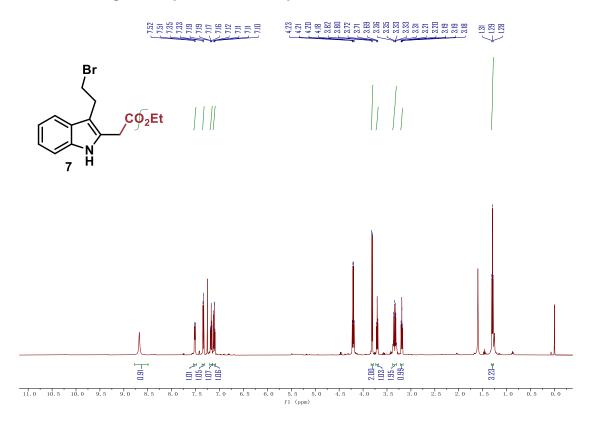
¹H NMR of compound 6 (400 MHz in CDCl₃)



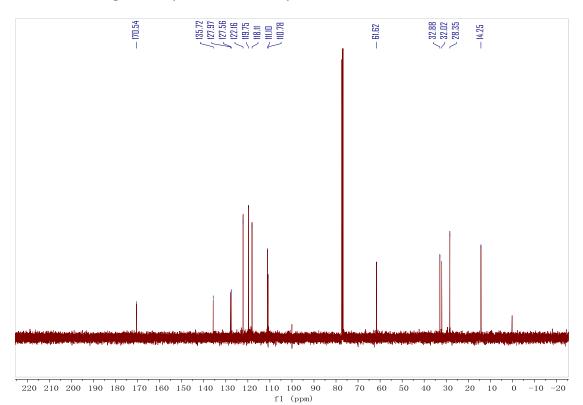
¹³C NMR of compound 6 (101 MHz in CDCl₃)



¹H NMR of compound 7 (400 MHz in CDCl₃)

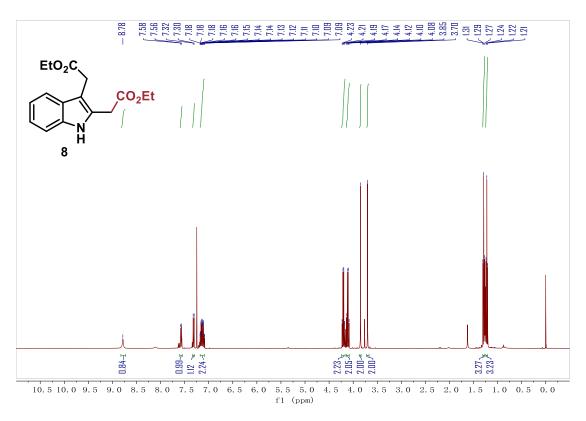


¹³C NMR of compound 7 (101 MHz in CDCl₃)

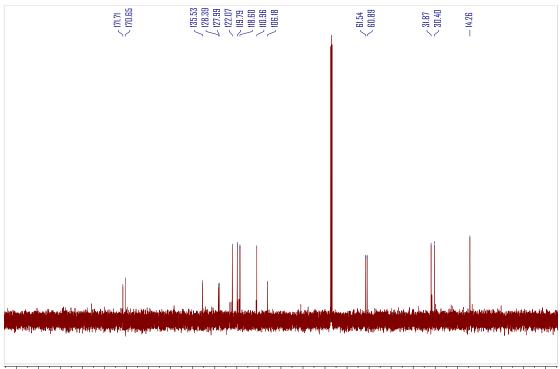


S26

¹H NMR of compound 8 (400 MHz in CDCl₃)

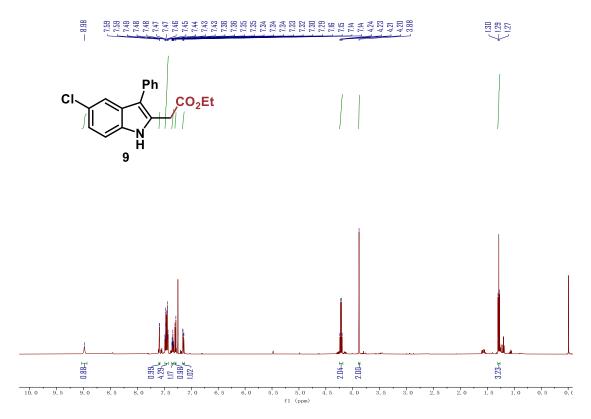


¹³C NMR of compound 8 (101 MHz in CDCl₃)

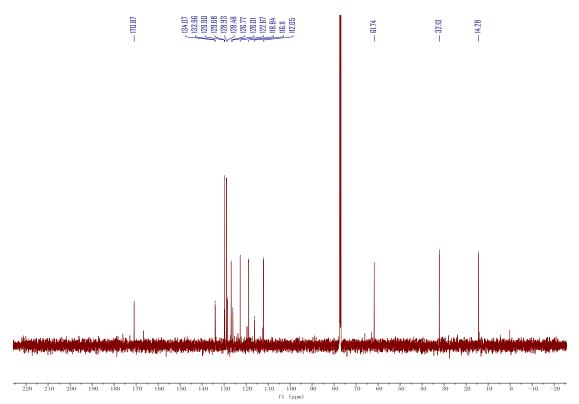


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 f1 (ppm)

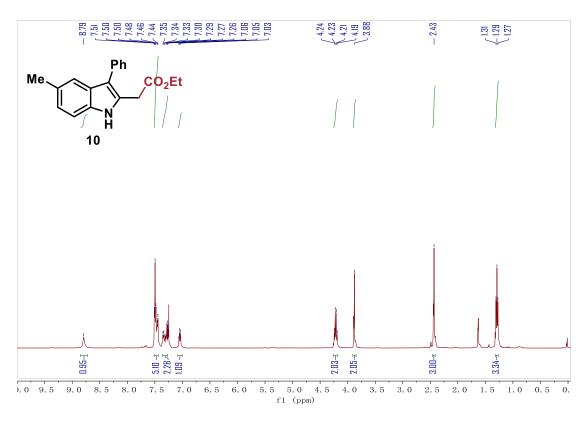
¹H NMR of compound 9 (500 MHz in CDCl₃)



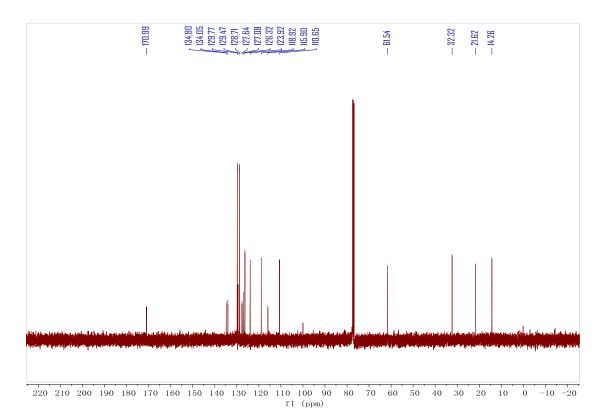
¹³C NMR of compound 9 (126 MHz in CDCl₃)



¹H NMR of compound 10 (400 MHz in CDCl₃)

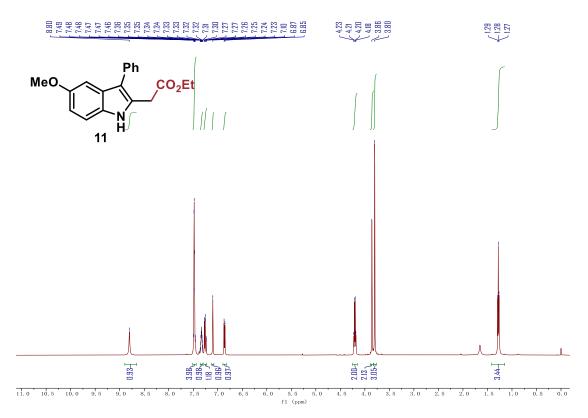


¹³C NMR of compound 10 (101 MHz in CDCl₃)

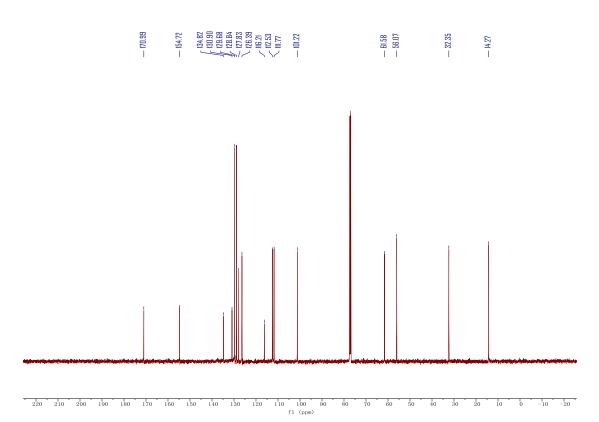


S29

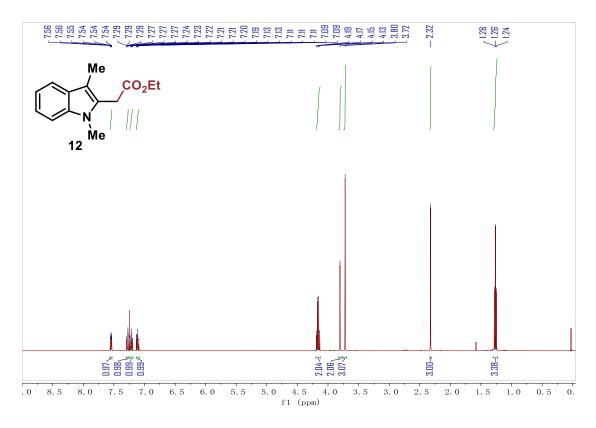
¹H NMR of compound 11 (400 MHz in CDCl₃)



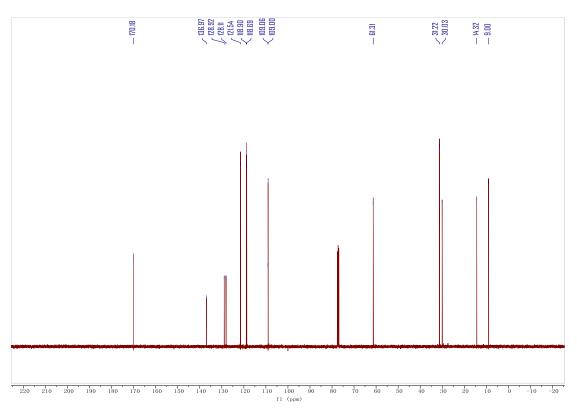
¹³C NMR of compound 11 (101 MHz in CDCl₃)



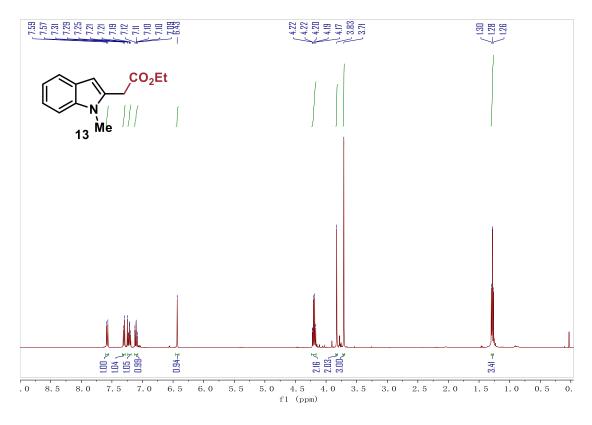
¹H NMR of compound 12 (400 MHz in CDCl₃)



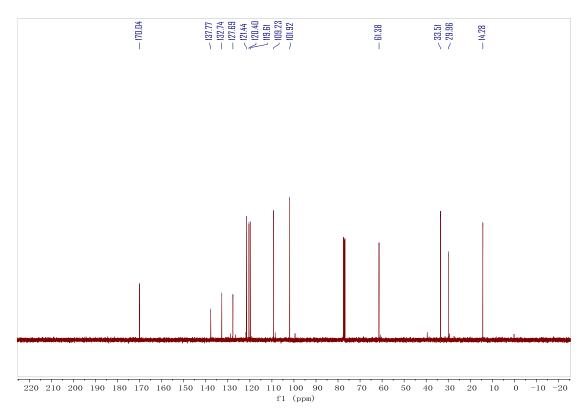
¹³C NMR of compound 12 (101 MHz in CDCl₃)



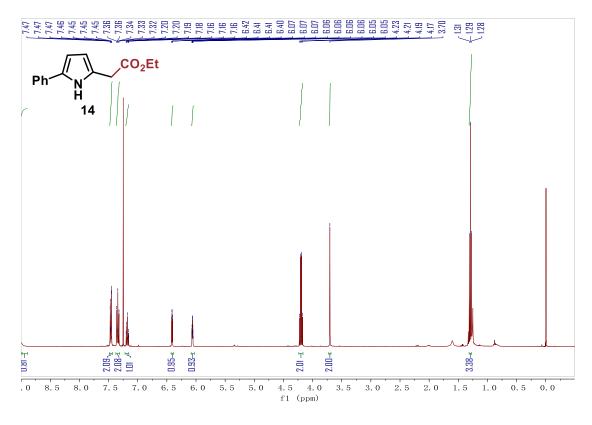
¹H NMR of compound 13 (400 MHz in CDCl₃)



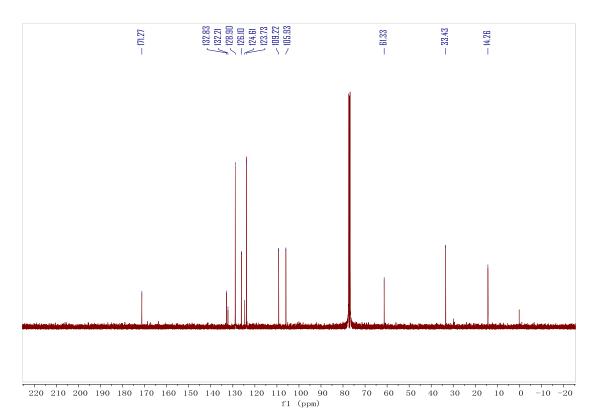
¹³C NMR of compound 13 (101 MHz in CDCl₃)



¹H NMR of compound 14 (400 MHz in CDCl₃)

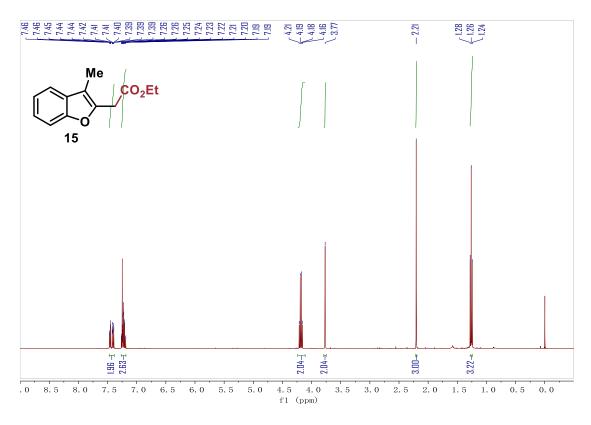


¹³C NMR of compound 14 (101 MHz in CDCl₃)

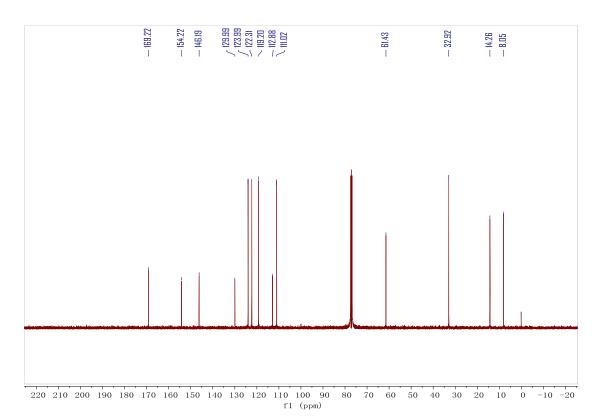


S33

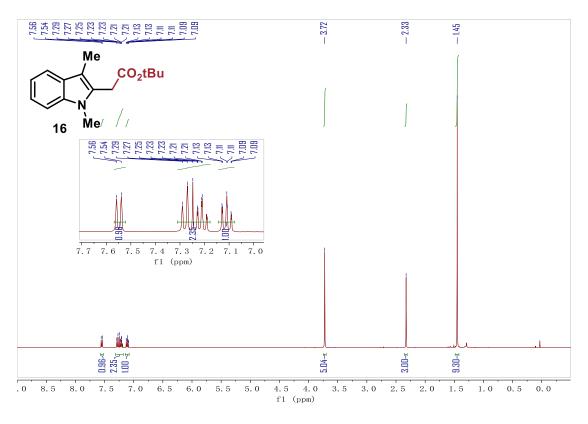
¹H NMR of compound 15 (400 MHz in CDCl₃)



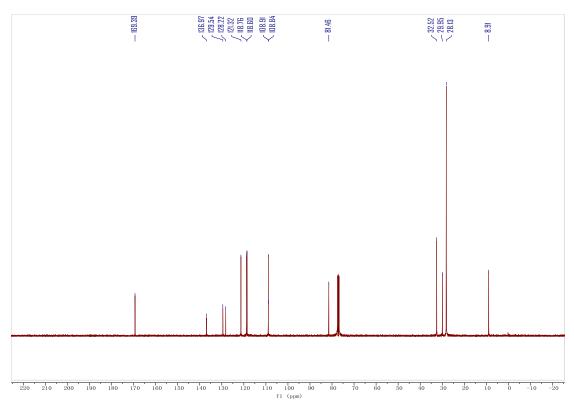
¹³C NMR of compound 15 (101 MHz in CDCl₃)



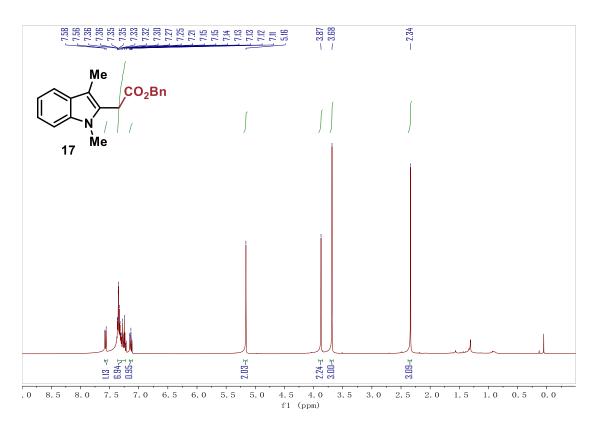
¹H NMR of compound 16 (400 MHz in CDCl₃)



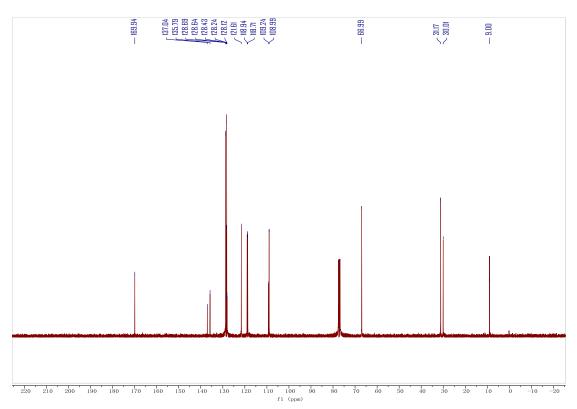
¹³C NMR of compound 16 (101 MHz in CDCl₃)



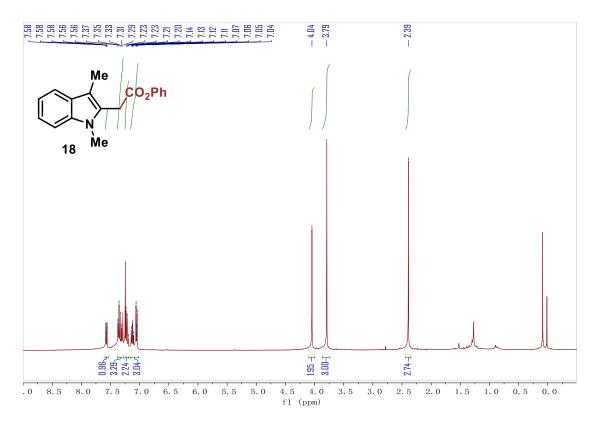
¹H NMR of compound 17 (400 MHz in CDCl₃)



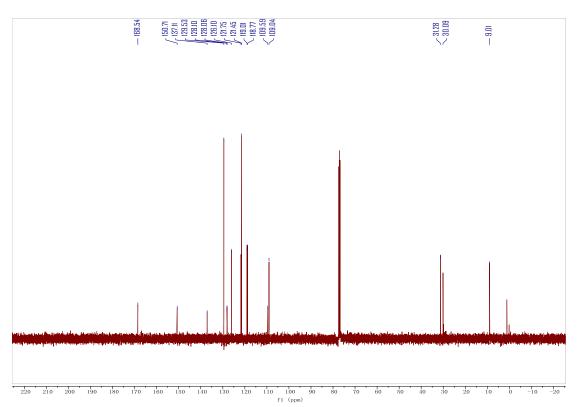
¹³C NMR of compound 17 (101 MHz in CDCl₃)

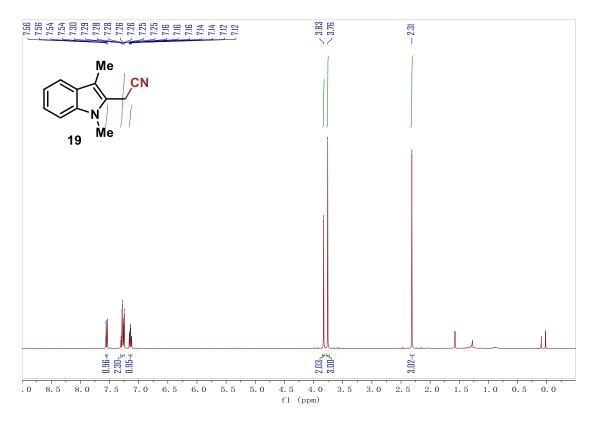


¹H NMR of compound 18 (400 MHz in CDCl₃)



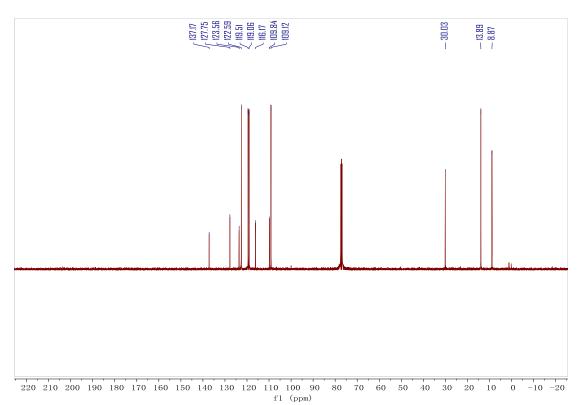
¹³C NMR of compound 18 (101 MHz in CDCl₃)



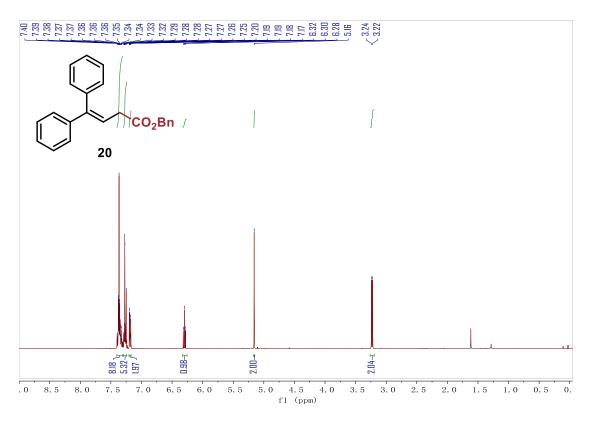


¹H NMR of compound 19 (400 MHz in CDCl₃)

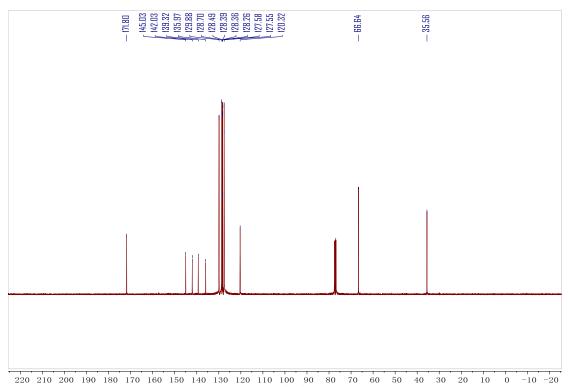
¹³C NMR of compound 19 (101 MHz in CDCl₃)



¹H NMR of compound 20 (400 MHz in CDCl₃)

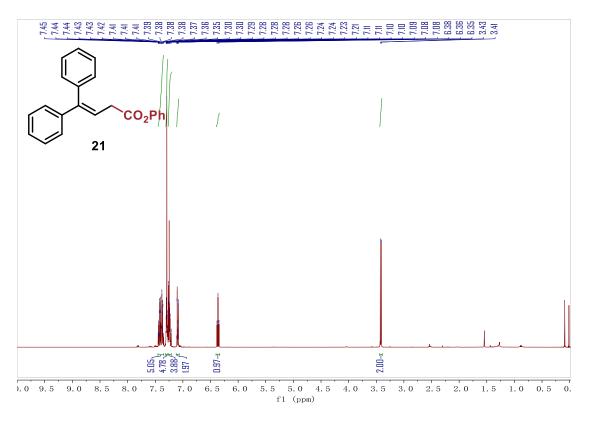


^{13}C NMR of compound 20 (101 MHz in CDCl_3)

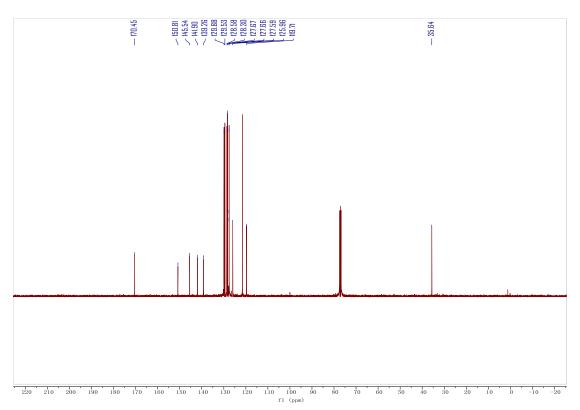


fl (ppm)

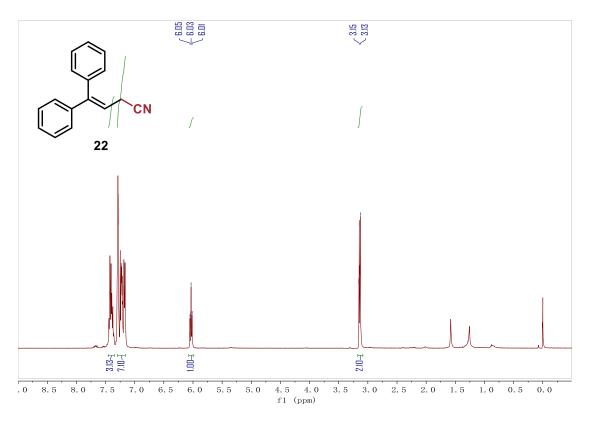
¹H NMR of compound 21 (400 MHz in CDCl₃)



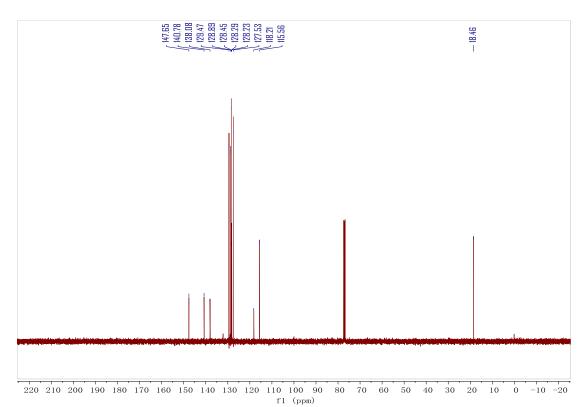
¹³C NMR of compound 21 (101 MHz in CDCl₃)



¹H NMR of compound 22 (400 MHz in CDCl₃)

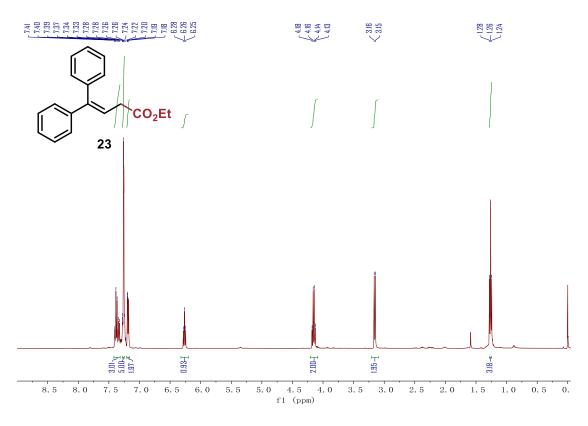


¹³C NMR of compound 22 (101 MHz in CDCl₃)

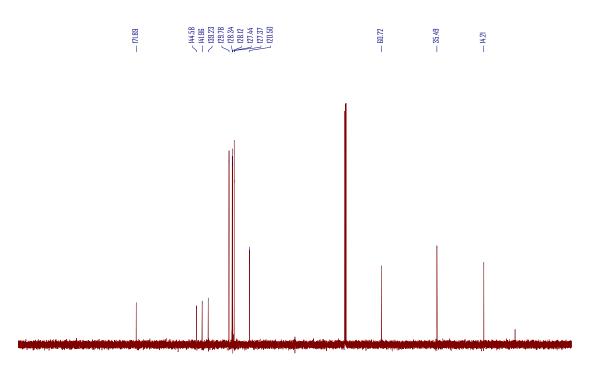


S41

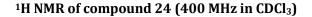
¹H NMR of compound 23 (400 MHz in CDCl₃)

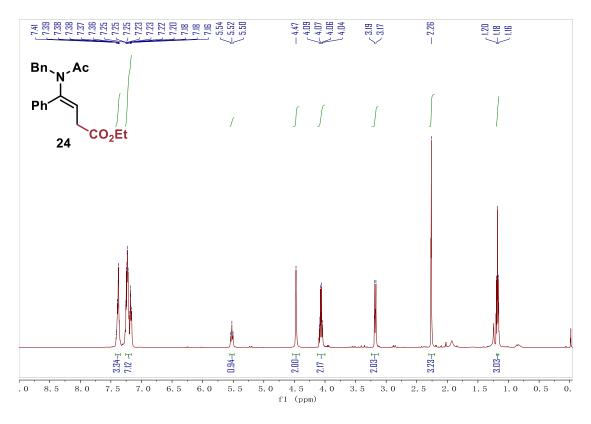


¹³C NMR of compound 23 (101 MHz in CDCl₃)

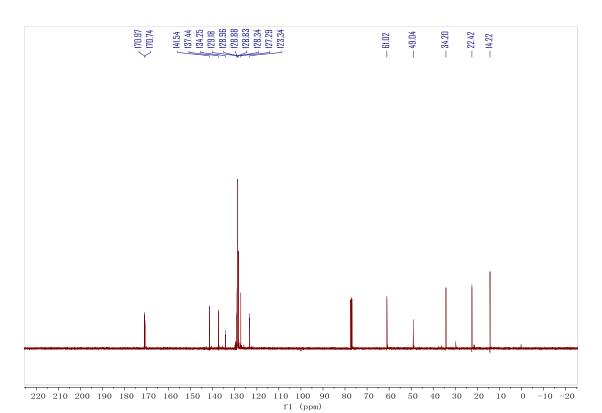


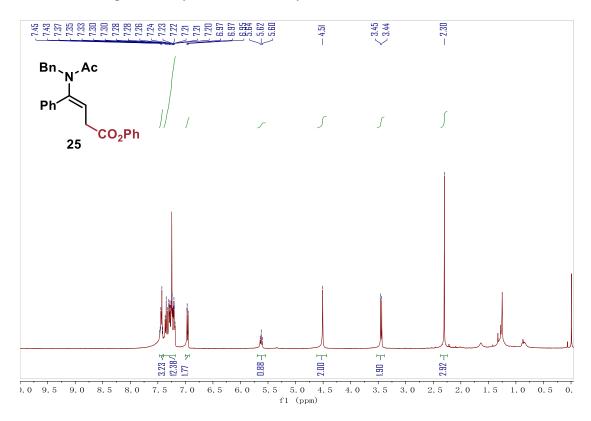
^{220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20} f1 (ppm)





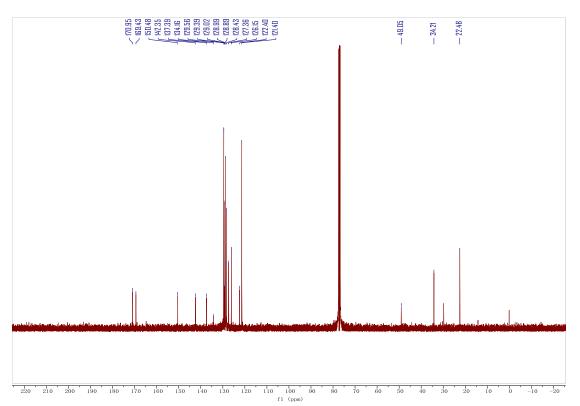
¹³C NMR of compound 24 (101 MHz in CDCl₃)





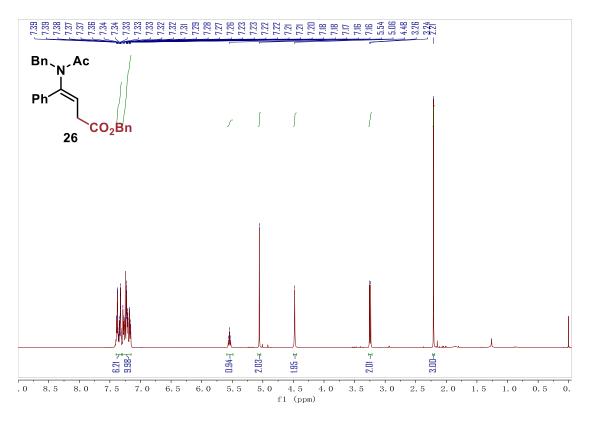
¹H NMR of compound 25 (400 MHz in CDCl₃)

¹³C NMR of compound 25 (101 MHz in CDCl₃)

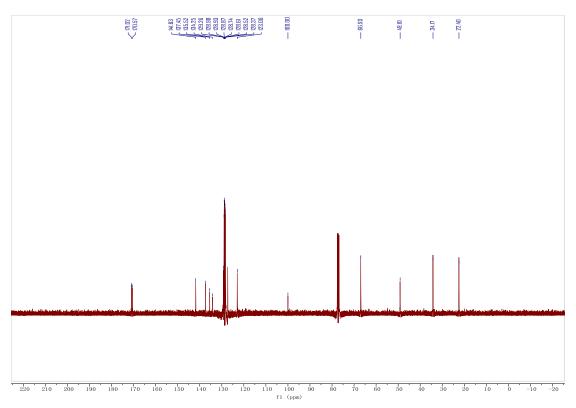


S44

¹H NMR of compound 26 (400 MHz in CDCl₃)

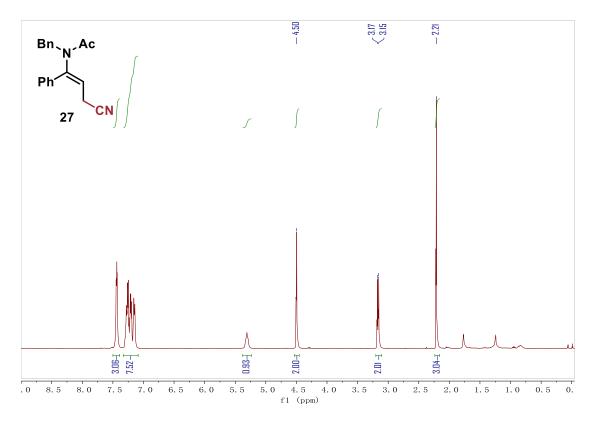


¹³C NMR of compound 26 (101 MHz in CDCl₃)

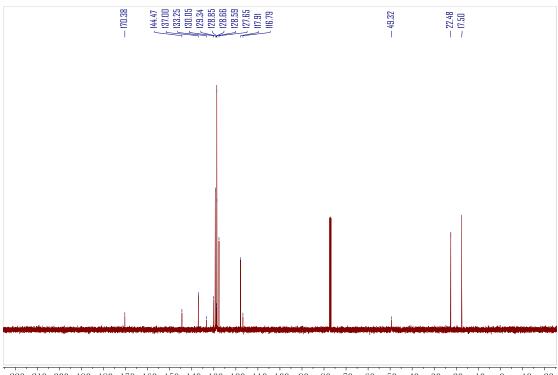


S45

¹H NMR of compound 27 (400 MHz in CDCl₃)

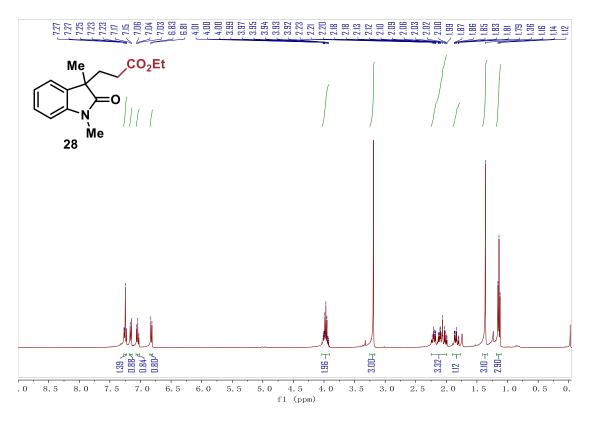


¹³C NMR of compound 27 (101 MHz in CDCl₃)

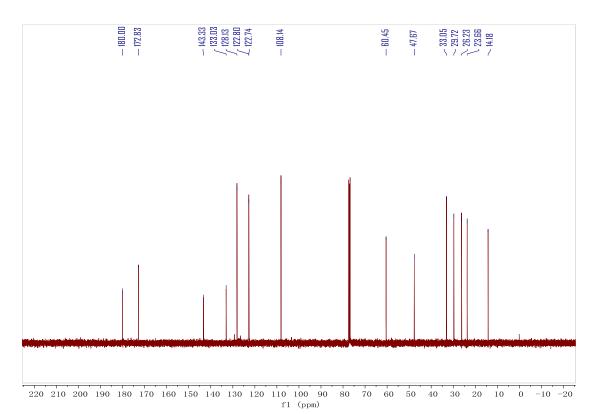


220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 f1 (ppm)

¹H NMR of compound 28 (400 MHz in CDCl₃)

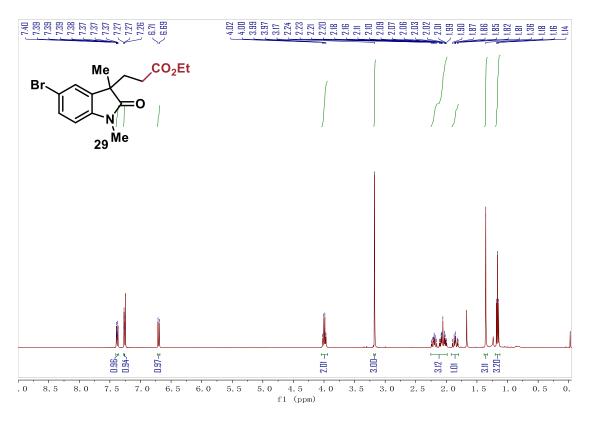


¹³C NMR of compound 28 (101 MHz in CDCl₃)

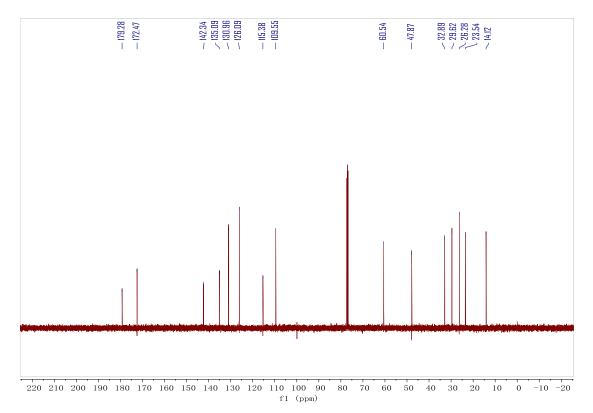


S47

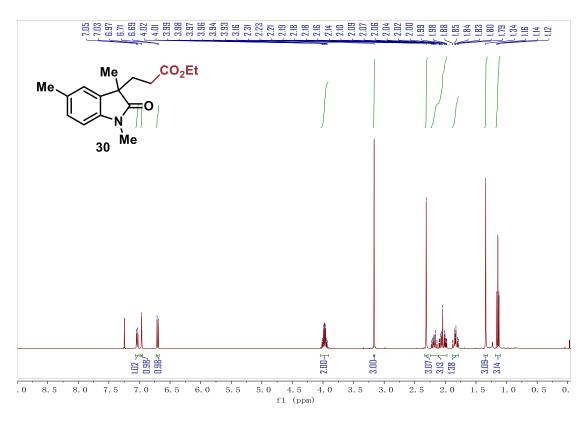
¹H NMR of compound 29 (400 MHz in CDCl₃)



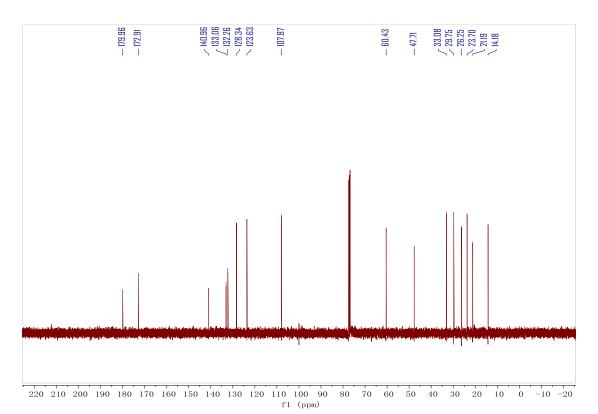
¹³C NMR of compound 29 (101 MHz in CDCl₃)



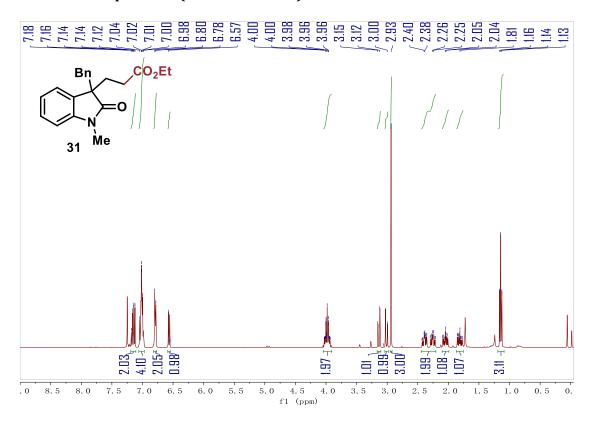
¹H NMR of compound 30 (400 MHz in CDCl₃)



¹³C NMR of compound 30 (101 MHz in CDCl₃)



S49



¹H NMR of compound 31 (400 MHz in CDCl₃)

¹³C NMR of compound 31 (101 MHz in CDCl₃)

