

Supporting Information for

Acid-Catalyzed Acylation Reaction *via* C–C Bond Cleavage: A Facile Approach to 3-Acylindole

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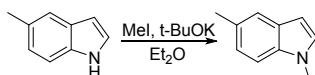
1) General Information

The reagents, free (NH)-indoles, 7-zazindole, and N-methyl indole were purchased and used as received without further purification. NMR spectra of the products were recorded using a Bruker Avance TM spectrometer operating at 400 MHz for ¹H and 100 MHz for ¹³C in CDCl₃ unless otherwise noted. High resolution mass spectra (HRMS) of the products were obtained on a Bruker Daltonics micro TOF-spectrometer. High-performance liquid chromatography (HPLC) analysis (methanol/H₂O = 80/20, 0.8 mL/min, λ = 318 nm) was performed by Agilent 1260 Infinity with an Agilent ZORBAX C18 column using xanthene as inner standard for 3-acylindole. Isolated yield was obtained by column chromatography (300-400 mesh), and Ethyl acetate/ Petroleum ether was used as the eluent.

2) General Procedures for the Synthesis of N-substituted Indoles

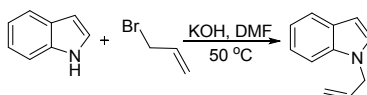
All of the N-substituted indoles were prepared according to literatures with slight modification.

2.1 General procedure for the preparation of N-methyl indoles¹



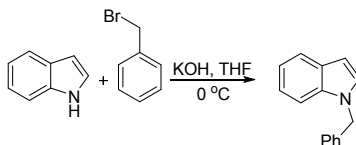
To a stirred solution of 5-methyl indole (1 mol) in ether (10 mL) was slowly added potassium *tert*-butanolate (2 mol) and iodomethane (2 mol) at 0 °C, after 24 hours the reaction mixture was poured into saturated aqueous NaHCO₃ solution (25 mL) and extracted with ether (10 mL) three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The crude product was purified by column chromatography (silica gel, pure hexane). The product was characterized and compared with the previously reported literature. Other N-methyl indoles were synthesized according to the above method.

2.2 General procedure for the preparation of N-allyl indole²



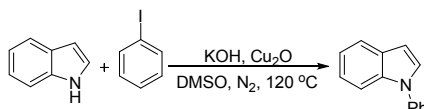
To a stirred solution of 1H-indole (1 mol) in DMF (10 mL) was slowly added potassium hydroxide (1.2 mol) and allyl bromide (1.2 mol). After 12 hours at 50 °C, the reaction mixture was cooled to room temperature, poured into saturated aqueous NaHCO₃ solution (10 mL) and extracted with ether (20 mL) three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The crude product was purified by column chromatography (silica gel, pure hexane). The product was characterized and compared with the previously reported literature. Other N-allyl indoles were synthesized according to the above method.

2.3 General procedure for the preparation of N-benzyl indoles³



To a stirred solution of 1H-indole (1 mol) in tetrahydrofuran (10 mL) was slowly added potassium hydroxide (4 mol) and benzyl bromide (1 mol) at 0 °C. After 24 hours, the mixture was poured into saturated aqueous NaHCO₃ solution (10 mL) and extracted with ether (20 mL) three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The crude product was purified by column chromatography (silica gel, pure hexane). The product was characterized and compared with the previously reported literature. Other N-benzyl indoles were synthesized according to the above method.

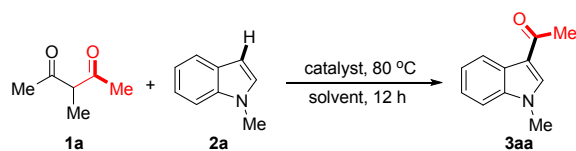
2.4 General procedure for the preparation of N-phenyl indole⁴



A mixture of iodobenzene (1 mol), 1H-Indole (1.5 mol), cuprous oxide (0.1 mol), potassium hydroxide (2 mol) and DMSO (2 mL) was stirred at 120 °C under N₂ atmosphere for 24 hours. Then the mixture was poured into 15 mL H₂O and extracted with ether (20 mL) three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The crude product was purified by column chromatography (silica gel, hexane:ethyl acetate = 50:1). The product was characterized and compared with the previously reported literature.

3) Optimization of the reaction conditions

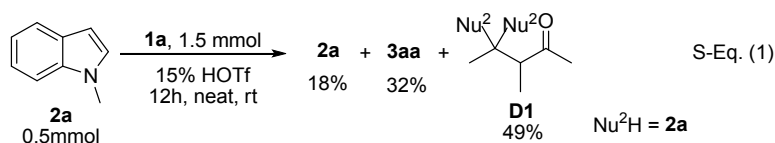
Table S1. Optimization of reaction conditions in the synthesis of **3aa**^[a].



Entry	Catalyst	Solvent	Temp (°C)	Yield (%)
1	Fe(OTf) ₃	neat	80	91
2	Ni(OTf) ₂	neat	80	76
3	Cu(OTf) ₂	neat	80	64
4	AgOTf	neat	80	68
5	Sc(OTf) ₃	neat	80	75
6	Fe(acac) ₃	neat	80	13
7	FeCl ₂	neat	80	29
8	FeCl ₃	neat	80	46
9	Fe(OAc) ₂	neat	80	n.d.
10	HOTf	neat	80	71 ^[b]
11	HOTf	neat	80	93 ^[c]
12	HOTf	neat	80	100 (89) ^{[c], [d]}
13	HOTf	neat	r.t.	32 ^[c]
14	HOTf	H ₂ O	80	91(80) ^[c]
15	HOTf	H ₂ O	80	(72) ^[e]
16	–	neat	80	n.d.

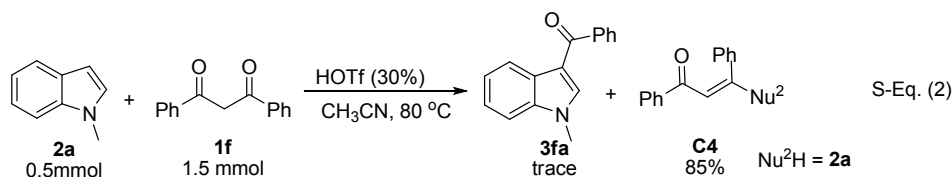
[a] Reaction conditions: **1a** (1.5 mmol), **2a** (0.5 mmol), 80 °C, 5.0 mol % of catalyst, solvent (1 mL), 12 h. The yields were determined by HPLC analysis with xanthene as an internal standard. [b] 5 mol % of HOTf was used as catalyst. [c] 15 mol % of HOTf was used as catalyst. [d] 24 h, isolated yields in the parentheses. [e] 15 mol % of HOTf, isolated yield of the second run.

3.1 Reaction of **2a** and **1a** at room temperature

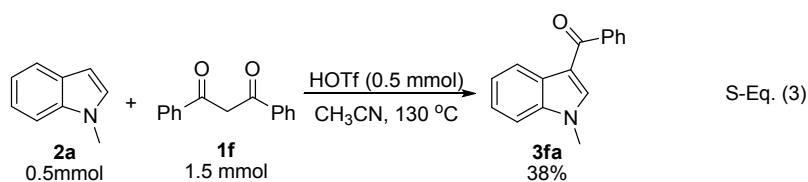


A mixture of **2a** (0.5mmol), **1a** (1.5mmol) and HOTf (15 mol%) was added into a 25 mL Schlenk tube. After stirring for 12h at room temperature, the mixture was detected by HPLC and **3aa** was obtained in 32% yield along with 49% yield of 3-methyl-4,4-bis(1-methyl-1H-indol-3-yl)pentan-2-one.

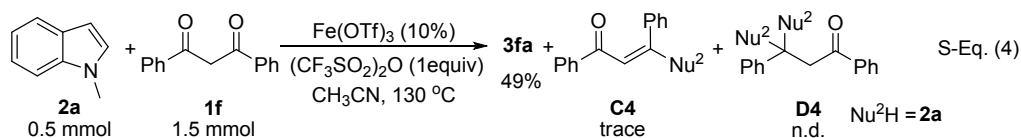
3.2 Optimization of the reaction of **1f** and **2a**



A mixture of **2a** (0.5mmol), **1f** (1.5mmol) and HOTf (30 mol%) in acetonitrile (1mL) was added into a 25 mL Schlenk tube. After stirring for 24 h at 80 °C, the mixture was detected by HPLC. Only trace of **3fa** was detected and 85% yield of **C4** was obtained.

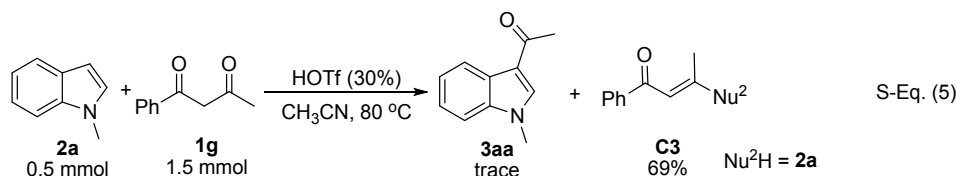


A mixture of **2a** (0.5mmol), **1f** (1.5mmol) and HOTf (0.5 mmol) in acetonitrile (1mL) was added into a 25 mL Schlenk tube. After stirring for 24 h at 130 °C, the mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate (5/1) with 0.5 mL triethylamine as the eluent and 38% yield of **3fa** was obtained.

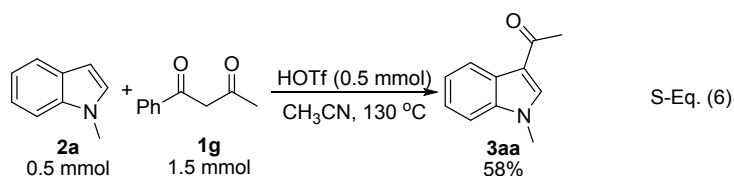


A mixture of **2a** (0.5mmol), **1f** (1.5mmol) and $\text{Fe}(\text{OTf})_3$ (0.05mmol) in acetonitrile (1mL) was added into a 25 mL Schlenk tube, to which $(\text{CF}_3\text{SO}_2)_2\text{O}$ (0.5mmol) was injected quickly. After stirring for 24 h at 130 °C, the mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate (5/1) with 0.5 mL triethylamine as the eluent to afford **3fa** in 49% yield.

3.3 Optimization of the reaction of **1g** and **2a**

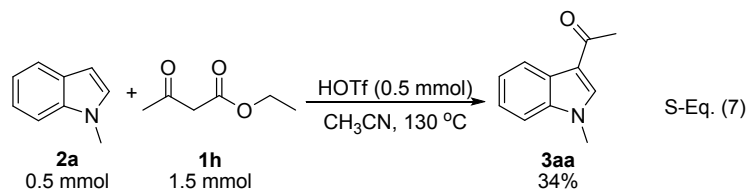


A mixture of **2a** (0.5mmol), **1g** (1.5mmol) and HOTf (30 mol%) in acetonitrile (1mL) was added into a 25 mL Schlenk tube. After stirring for 24 h at 80 °C, the mixture was detected by HPLC and only trace of **3aa** along with 79% yield of **C4** was detected.

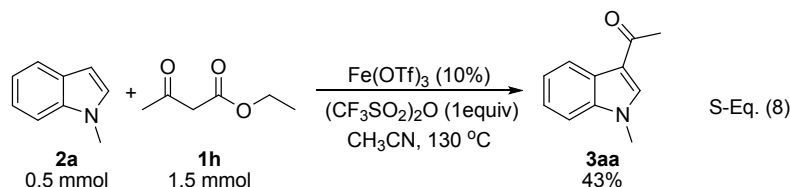


A mixture of **2a** (0.5mmol), **1g** (1.5mmol) and HOTf (0.5 mmol) in acetonitrile (1mL) was added into a 25 mL Schlenk tube. After stirring for 24 h at 130 °C, the mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate (2/1) with 0.5 mL triethylamine as the eluent to afford **3aa** in 58% yield.

3.4 Optimization of the reaction of **1h** and **2a**



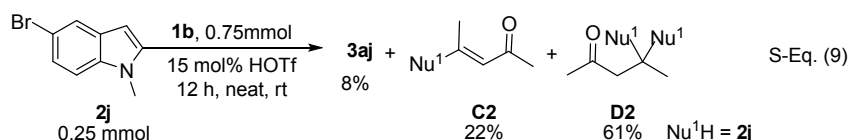
A mixture of **2a** (0.5mmol), **1h** (1.5mmol) and HOTf (0.5 mmol) in acetonitrile (1mL) was added into a 25 mL Schlenk tube. After stirring for 24 h at 130 °C, the mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate (2/1) with 0.5 mL triethylamine as the eluent and 34% yield of **3aa** was obtained.



A mixture of **2a** (0.5mmol), **1h** (1.5mmol) and $\text{Fe}(\text{OTf})_3$ (0.05mmol) in acetonitrile (1mL) was added into a 25 mL Schlenk tube, to which $(\text{CF}_3\text{SO}_2)_2\text{O}$ (0.5mmol) was injected quickly. After stirring for 24 h at 130 °C, the mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate (2/1) with 0.5 mL triethylamine as the eluent to afford **3aa** in 43% yield.

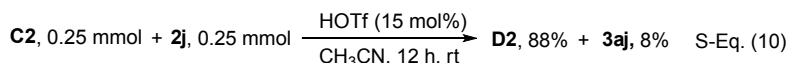
4) Mechanistic studies

4.1 The reaction of **2j** and **1b** at room temperature

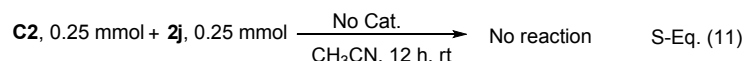


A mixture of **2j** (0.25 mmol), **1b** (0.75 mmol), and HOTf (15 mol%) was introduced into a 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was detected by HPLC to afford **3aj** in 8% yield, **C2** in 22% yield and **D2** in 61% yield.

4.2 The reaction of C1 and 2j under different conditions

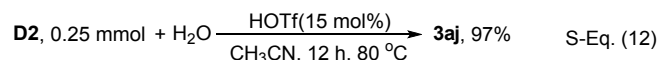


A mixture of **C2** (0.25 mmol), **2j** (0.25 mmol) and HOTf (15 mol%) in CH₃CN (2mL) was introduced into a 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate 10/1 as the eluent to afford **D2** in 88% yield and **3aj** in 8% yield.

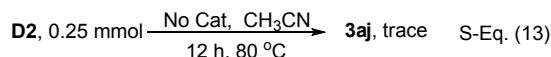


When the reaction was conducted in the absence of HOTf, no desired product **D2** was observed.

4.3 The reaction of D1 under different conditions



A mixture of **D2** (0.25 mmol) and HOTf (15 mol%) in CH₃CN (2mL) was introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 12 h. After cooling to room temperature, the resultant reaction mixture was purified by the silica gel chromatography using petroleum ether and ethyl acetate 10/1, 2/1 as the eluent to afford **3aj** in 97% yield.



When the reaction was conducted in the absence of HOTf, only trace of **3aj** was observed.

4.4 Investigations on whether present acylation proceeds *via* retro-Aldol reaction.

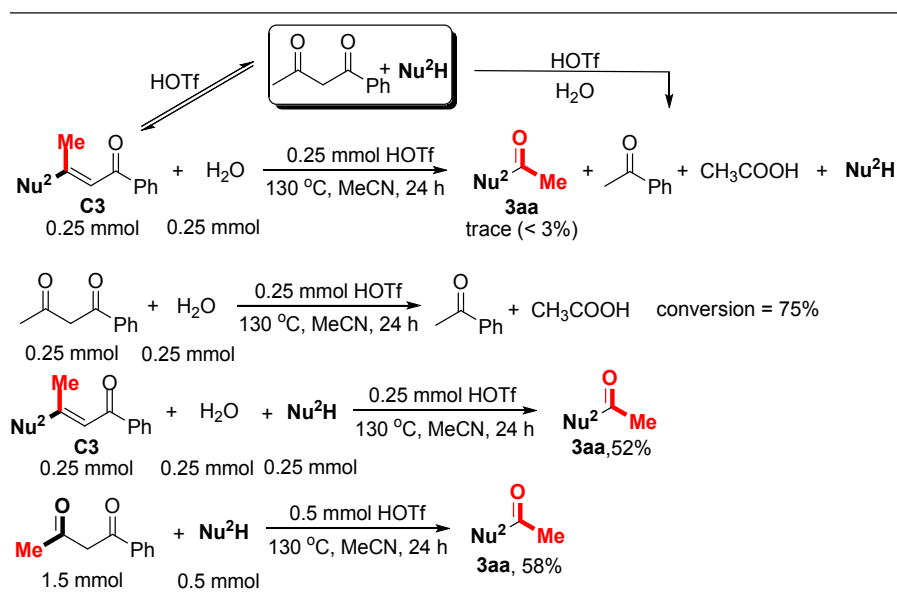


Figure S1. Investigations on whether present acylation proceeds *via* retro-Aldol reaction. **Nu²H** = 1-methyl-1H-indole (**2a**), the conversion of 1-Benzoyl-2-propanone was detected by GC-MS.

4.5 Track of the reaction of 2j and 1b under the optimized reaction conditions

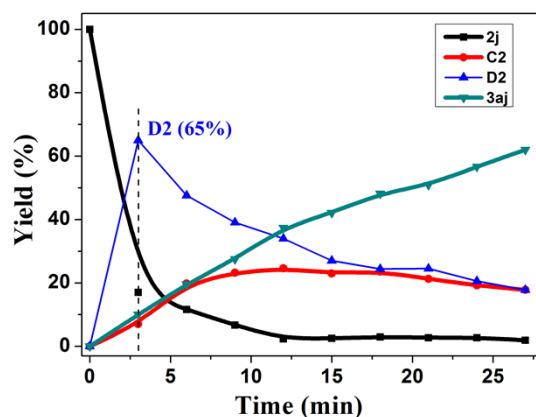
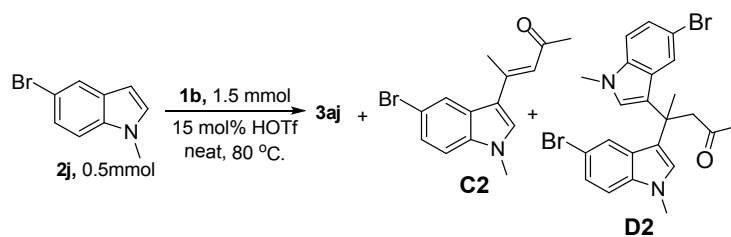
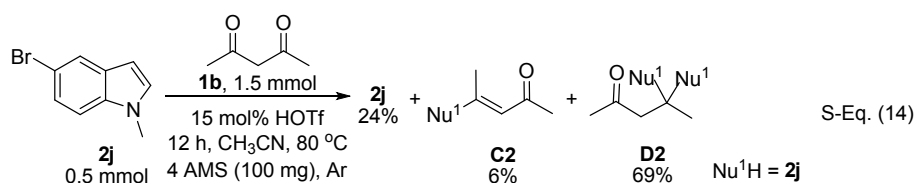
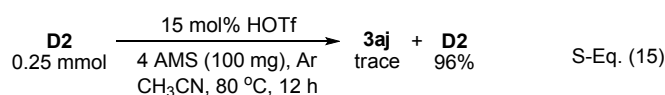


Figure S2. The distribution of substrate, intermediates and product with time in the reaction of **1b** and **2j** under the optimized conditions.

4.6 Control reactions in the absence of water

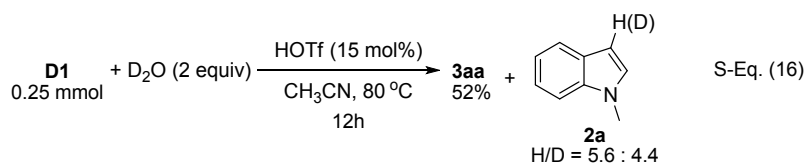


2j (0.5 mmol), **1b** (1.5 mmol), 4 AMS (100 mg) and HOTf (15 mol%) in CH_3CN (1 mL) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred in argon atmosphere at 80 °C for 12 h. After cooling to room temperature, the resultant reaction mixture was detected by HPLC to afford **C2** in 6% yield, **D2** in 69% yield and 24% of **2j** was recovered.



A mixture of **D2** (0.25 mmol), 4AMS (100 mg) and HOTf (15 mol%) in CH_3CN (2 mL) was introduced into a 25 mL Schlenk tube. The formed mixture was stirred in argon atmosphere at 80 °C for 12 h. After detected by HPLC, only trace of **3aj** was obtained and 96% of **D2** was recovered.

4.7 The reaction of **D3** with the addition of D_2O



In a glove-box, a mixture of **D3** (0.25 mmol), D_2O (0.5 mmol) and HOTf (15 mol%) in anhydrous acetonitrile (2 mL) was stirred in a 25 mL Schlenk tube at 80 °C for 12 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1, 2:1 with 0.5 mL triethylamine) to afford **3aa** in 52% yield. And a mixture of **2a** (H/D = 5.6:4.4) was detected by HNMR.

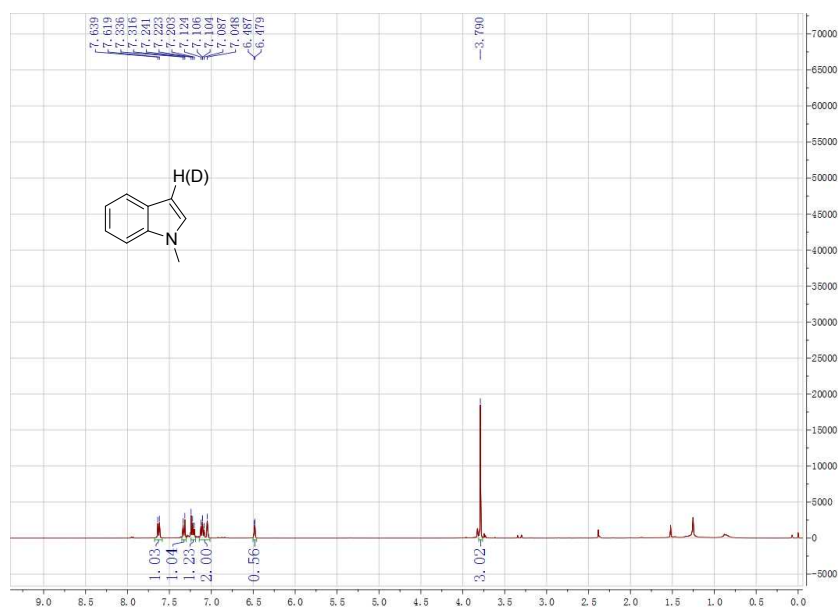
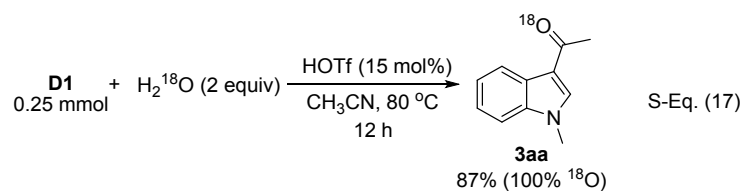
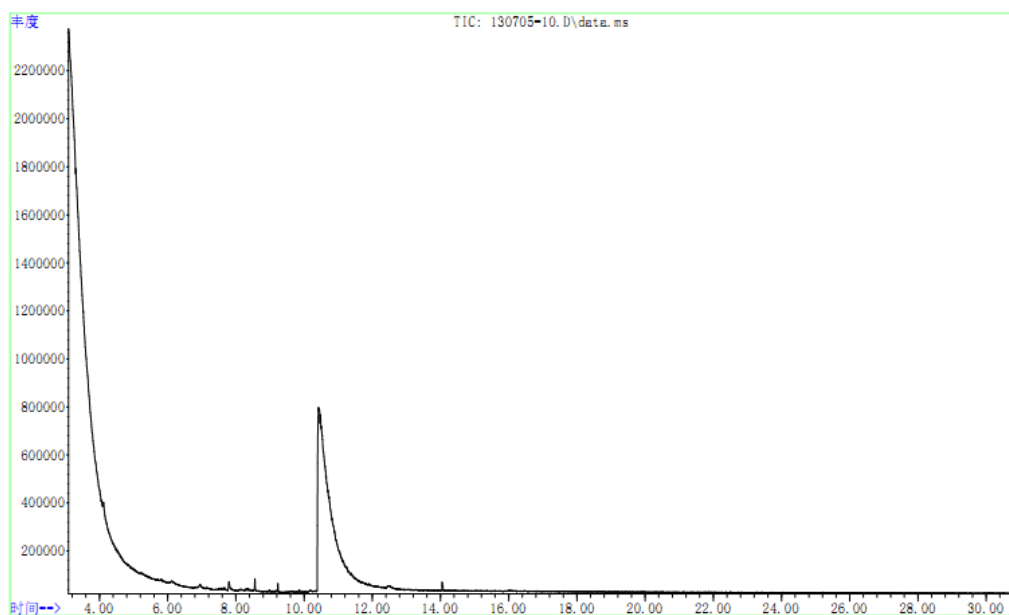


Figure S3. ^1H NMR for the generated indole

4.8 The reaction of D3 with the addition of H_2^{18}O



In a glove-box, a mixture of **D3** (0.25mmol), H_2^{18}O (0.5 mmol) and HOTf (15 mol%) in anhydrous acetonitrile (2mL) was stirred in a 25 mL Schlenk tube at 80 °C for 12 h. After cooling to room temperature, the resultant reaction mixture was purified by flash silica gel chromatography (petroleum ether / ethyl acetate =2:1 with 0.5 mL triethylamine) to afford 100% ^{18}O -labeled **3aa** in 87% yield.



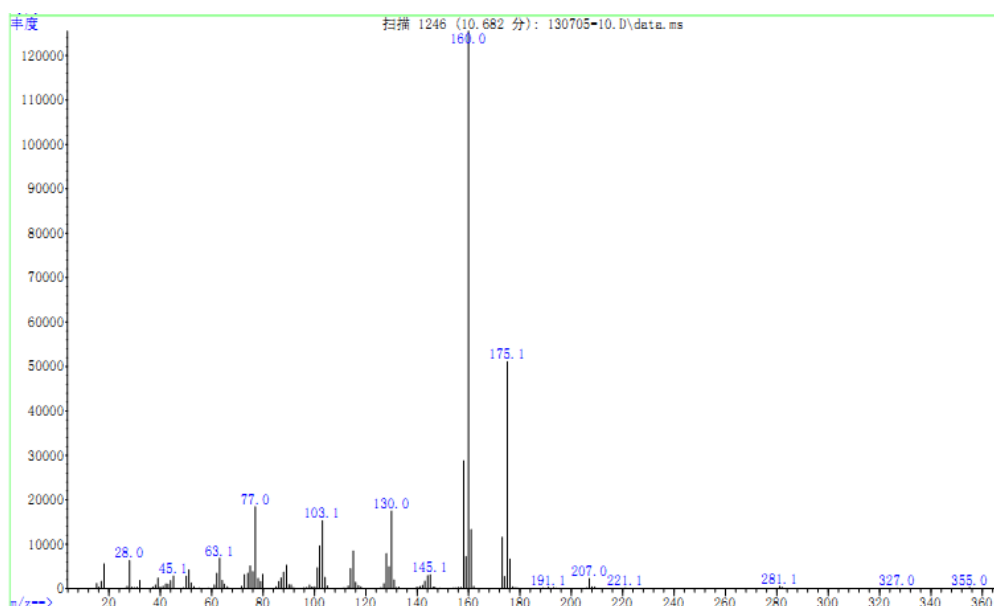


Figure S4. GC-MS for ^{18}O -3-acylindole

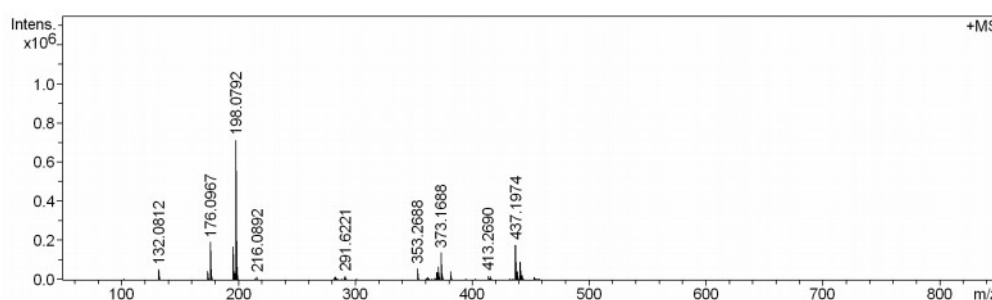
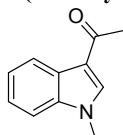


Figure S5. HRMS for ^{18}O -3-acylindole

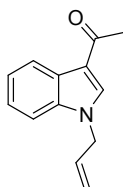
5) Experimental Procedures and Characterization of Products

1-(1-methyl-1H-indol-3-yl)ethanone (**3aa**)



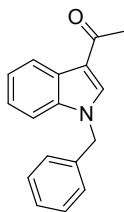
2a (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 89% of **3aa**. ^1H NMR (400 MHz, CDCl_3) δ 8.38 – 8.36 (m, 1H), 7.72 (s, 1H), 7.37 – 7.29 (m, 3H), 3.86 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 193.0, 135.7, 126.2, 123.3, 122.6, 121.8, 117.0, 109.9, 109.6, 33.5, 27.6. HRMS (ESI) Calcd for $\text{C}_{11}\text{H}_{11}\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 196.0733; Found: 196.0743.

1-(1-allyl-1H-indol-3-yl)ethanone (**3ab**)



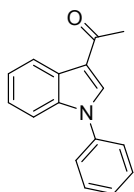
2b (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 71% of **3ab**. ^1H NMR (400 MHz, CDCl_3) δ 8.43 – 8.34 (m, 1H), 7.75 (s, 1H), 7.39 – 7.28 (m, 3H), 6.03 (m, 1H), 5.31 (d, $J = 10.4$ Hz, 1H), 5.18 (d, $J = 17.2$ Hz, 1H), 4.78 (d, $J = 5.2$ Hz, 2H), 2.54 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 193.0, 136.9, 134.7, 132.1, 126.4, 123.3, 122.7, 122.6, 118.7, 117.4, 110.0, 49.4, 27.7. HRMS (ESI) Calcd for $\text{C}_{13}\text{H}_{13}\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 222.0889; Found: 222.0902.

1-(1-benzyl-1H-indol-3-yl)ethanone (3ac)



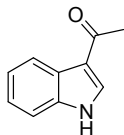
2c (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 54% of **3ac**. ¹H NMR (400 MHz, CDCl₃) δ 8.36 – 8.30 (m, 1H), 7.68 (s, 1H), 7.25 – 7.17 (m, 6H), 7.09 – 7.06 (m, 2H), 5.27 (s, 2H), 2.44 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 137.1, 135.8, 135.0, 129.1, 128.2, 127.0, 126.4, 123.5, 122.7, 122.7, 117.5, 110.2, 50.7, 27.7. HRMS (ESI) Calcd for C₁₇H₁₅NNaO: [M+Na]⁺, 272.1046; Found: 272.1056.

1-(1-phenyl-1H-indol-3-yl)ethanone (3ad)



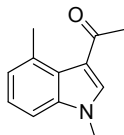
2d (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 58% of **3ad**. ¹H NMR (400 MHz, CDCl₃) δ 8.46 (d, *J* = 7.6 Hz, 1H), 7.94 (s, 1H), 7.51 (m, 6H), 7.33 (m, 2H), 2.59 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.3, 138.4, 137.1, 134.7, 129.9, 128.0, 126.5, 125.0, 124.0, 123.1, 122.8, 118.7, 110.8, 27.7. HRMS (ESI) Calcd for C₁₆H₁₃NNaO: [M+Na]⁺, 258.0889; Found: 258.0879.

1-(1H-indol-3-yl)ethanone (3ae)



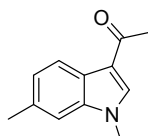
2e (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 64% of **3ae**. ¹H NMR (400 MHz, CDCl₃) δ 8.70 (s, 1H), 8.32 (m, 1H), 7.77 (d, *J* = 2.8 Hz, 1H), 7.34(m, 1H), 7.24(m, 2H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.7, 136.3, 131.5, 125.4, 123.7, 122.7, 122.4, 118.6, 111.3, 27.6. HRMS (ESI) Calcd for C₁₀H₉NNaO: [M+Na]⁺, 182.0576; Found: 182.0580

1-(1,4-dimethyl-1H-indol-3-yl)ethanone(3ag)



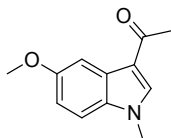
2g (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 48% of **3ag**. ¹H NMR (400 MHz, CDCl₃) δ 7.71 (s, 1H), 7.23 – 7.19 (m, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 7.1 Hz, 1H), 3.81 (s, 3H), 2.85 (s, 3H), 2.53 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 191.9, 138.4, 137.0, 133.9, 124.9, 124.4, 123.6, 118.7, 107.1, 33.6, 28.5, 23.2. HRMS (ESI) Calcd for C₁₂H₁₃NNaO: [M+Na]⁺, 210.0889; Found: 210.0898.

1-(1,6-dimethyl-1H-indol-3-yl)ethanone(3ah)



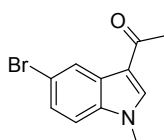
2h (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 61% of **3ah**. ¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 8.4 Hz, 1H), 7.65 (s, 1H), 7.14 (d, *J* = 4.9 Hz, 2H), 3.81 (s, 3H), 2.51 (s, 3H), 2.50 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.9, 137.9, 135.3, 133.3, 124.2, 124.0, 122.2, 116.9, 109.6, 33.3, 27.5, 21.8. HRMS (ESI) Calcd for C₁₂H₁₃NNaO: [M+Na]⁺, 210.0889; Found: 210.0896.

1-(5-methoxy-1-methyl-1H-indol-3-yl)ethanone(3ai)



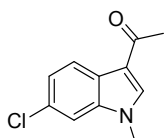
2i (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 86% of **3ai**. ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 2.5 Hz, 1H), 7.63 (s, 1H), 7.21 (d, *J* = 8.9 Hz, 1H), 6.94 (m, 1H), 3.90 (s, 3H), 3.80 (s, 3H), 2.49 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.9, 156.5, 135.8, 132.5, 127.0, 116.5, 113.8, 110.4, 103.8, 55.8, 33.7, 27.4. HRMS (ESI) Calcd for C₁₂H₁₃NNaO₂: [M+Na]⁺, 226.0839; Found: 226.0853.

1-(5-bromo-1-methyl-1H-indol-3-yl)ethanone(3aj)



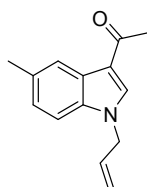
2j (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 76% of **3aj**. ¹H NMR (400 MHz, CDCl₃) δ 8.52 (d, *J* = 1.7 Hz, 1H), 7.66 (s, 1H), 7.38 (m, 1H), 7.18 (d, *J* = 8.7 Hz, 1H), 3.82 (s, 3H), 2.49 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.6, 136.3, 136.1, 127.7, 126.3, 125.2, 116.4, 116.3, 111.0, 33.7, 27.5. HRMS (ESI) Calcd for C₁₁H₁₀BrNNaO: [M+Na]⁺, 273.9838; Found: 273.9846.

1-(6-chloro-1-methyl-1H-indol-3-yl)ethanone(3ak)



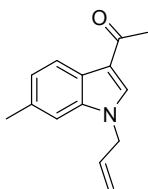
2k (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 67% of **3ak**. ¹H NMR (400 MHz, CDCl₃) δ 8.29 (d, *J* = 8.5 Hz, 1H), 7.67 (s, 1H), 7.32 (d, *J* = 1.5 Hz, 1H), 7.26 – 7.22 (m, 1H), 3.81 (s, 3H), 2.50 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.7, 137.9, 136.1, 129.4, 124.7, 123.6, 123.2, 117.0, 109.7, 33.6, 27.5. HRMS (ESI) Calcd for C₁₁H₁₀ClNNaO: [M+Na]⁺, 230.0343; Found: 230.0345.

1-(1-allyl-5-methyl-1H-indol-3-yl)ethanone (3an)



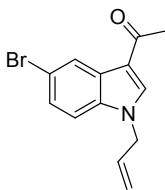
2n (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 72% of **3an**. ¹H NMR (400 MHz, CDCl₃) δ 8.19 (s, 1H), 7.71 (s, 1H), 7.22 (d, *J* = 8.4 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 6.01 (m, 1H), 5.29 (d, *J* = 10.3 Hz, 1H), 5.16 (d, *J* = 17.1 Hz, 1H), 4.74 (d, *J* = 5.4 Hz, 2H), 2.52 (s, 3H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 135.2, 134.8, 132.3, 132.1, 126.6, 124.9, 122.3, 118.6, 116.9, 109.7, 49.4, 27.6, 21.5. HRMS (ESI) Calcd for C₁₄H₁₅NNaO: [M+Na]⁺, 236.1046; Found: 236.1033.

1-(1-allyl-6-methyl-1H-indol-3-yl)ethanone(3ao)



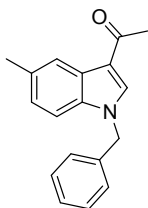
2o (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 76% of **3ao**. ¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 8.6 Hz, 1H), 7.68 (s, 1H), 7.13 (d, *J* = 7.7 Hz, 2H), 6.02 (m, 5.4 Hz, 1H), 5.30 (d, *J* = 10.2 Hz, 1H), 5.16 (d, *J* = 17.0 Hz, 1H), 4.74 (d, *J* = 5.4 Hz, 2H), 2.52 (s, 3H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.0, 137.3, 134.3, 133.3, 132.2, 124.3, 124.1, 122.3, 118.5, 117.3, 109.9, 49.2, 27.6, 21.9. HRMS (ESI) Calcd for C₁₄H₁₅NNaO: [M+Na]⁺, 236.1046; Found: 236.1043.

1-(1-allyl-5-bromo-1H-indol-3-yl)ethanone(3ap)



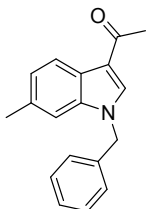
2p (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 65% of **3ap**. ¹H NMR (400 MHz, CDCl₃) δ 8.56 (d, *J* = 1.9 Hz, 1H), 7.72 (s, 1H), 7.38 (m, 1H), 7.20 (d, *J* = 8.7 Hz, 1H), 6.00 (m, 1H), 5.32 (d, *J* = 10.3 Hz, 1H), 5.16 (d, *J* = 17.1 Hz, 1H), 4.75 (m, 2H), 2.51 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.7, 135.5, 135.3, 131.7, 127.9, 126.4, 125.3, 119.0, 116.8, 116.4, 111.5, 49.6, 27.6. HRMS (ESI) Calcd for C₁₃H₁₃BrNO: [M+H]⁺, 278.0175; Found: 278.0178.

1-(1-benzyl-5-methyl-1H-indol-3-yl)ethanone(3aq)



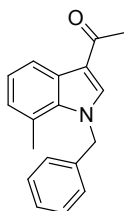
2t (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 73% of **3aq**. ¹H NMR (400 MHz, CDCl₃) δ 8.21 (s, 1H), 7.72 (s, 1H), 7.36 – 7.29 (m, 3H), 7.20 – 7.12 (m, 3H), 7.08 (d, *J* = 8.4 Hz, 1H), 5.33 (s, 2H), 2.51 (s, 3H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 135.9, 135.5, 135.1, 132.4, 129.0, 128.2, 126.9, 126.7, 125.0, 122.4, 117.1, 109.8, 50.8, 27.7, 21.5. HRMS (ESI) Calcd for C₁₈H₁₇NNaO: [M+Na]⁺, 286.1202; Found: 286.1217.

1-(1-benzyl-6-methyl-1H-indol-3-yl)ethanone(3ar)



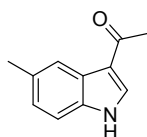
2u (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 63% of **3ar**. ¹H NMR (400 MHz, CDCl₃) δ 8.26 (d, *J* = 8.1 Hz, 1H), 7.69 (s, 1H), 7.38 – 7.30 (m, 3H), 7.20 – 7.07 (m, 4H), 5.32 (s, 2H), 2.51 (s, 3H), 2.44 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 137.5, 136.0, 134.5, 133.5, 129.1, 128.2, 126.9, 124.4, 124.2, 122.3, 117.5, 110.0, 50.5, 27.6, 21.9. HRMS (ESI) Calcd for C₁₈H₁₇NNaO: [M+Na]⁺, 286.1202; Found: 286.1211.

1-(1-benzyl-7-methyl-1H-indol-3-yl)ethanone(3as)



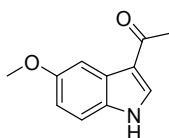
2v (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 56% of **3as**. ¹H NMR (400 MHz, CDCl₃) δ 8.34 (d, *J* = 8.0 Hz, 1H), 7.71 (s, 1H), 7.39 – 7.28 (m, 3H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.97 (m, 3H), 5.63 (s, 2H), 2.52 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 193.1, 137.9, 137.1, 135.9, 129.1, 127.9, 127.5, 126.6, 125.5, 123.0, 121.3, 120.8, 117.2, 53.0, 27.7, 19.4. HRMS (ESI) Calcd for C₁₈H₁₇NNaO: [M+Na]⁺, 286.1202; Found: 286.1200.

1-(5-methyl-1H-indol-3-yl)ethanone(3at)



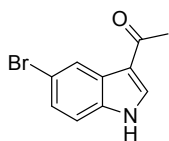
2q (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 58% of **3at**. ¹H NMR (400 MHz, DMSO) δ 11.87 (s, 1H), 8.30 (d, *J* = 3.1 Hz, 1H), 8.05 (s, 1H), 7.40 (d, *J* = 8.3 Hz, 1H), 7.09 (m, 1H), 2.49 (s, 3H), 2.46 (s, 3H). ¹³C NMR (100 MHz, DMSO) δ 192.5, 134.9, 134.3, 130.4, 125.5, 124.1, 121.0, 116.4, 111.6, 27.2, 21.3. HRMS (ESI) Calcd for C₁₁H₁₁NNaO: [M+H]⁺, 196.0733; Found: 196.0738.

1-(5-methoxy-1H-indol-3-yl)ethanone(3au)



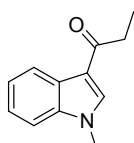
2r (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 76% of **3au**. ¹H NMR (400 MHz, DMSO) δ 11.86 (s, 1H), 8.29 (d, *J* = 3.0 Hz, 1H), 7.74 (d, *J* = 2.3 Hz, 1H), 7.41 (d, *J* = 8.8 Hz, 1H), 6.89 (m, 1H), 3.82 (s, 3H), 2.48 (s, 3H). ¹³C NMR (100 MHz, DMSO) δ 192.5, 155.3, 134.5, 131.5, 126.0, 116.6, 112.7, 112.5, 103.0, 55.2, 27.1. HRMS (ESI) Calcd for C₁₁H₁₁NNaO₂: [M+Na]⁺, 212.0682; Found: 212.0671.

1-(5-bromo-1H-indol-3-yl)ethanone(3av)



2s (0.5 mmol), **1a** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 2:1 with 0.5 mL triethylamine) to afford 62% of **3av**. ¹H NMR (400 MHz, DMSO) δ 12.19 (s, 1H), 8.38 (s, 1H), 8.31 (d, *J* = 1.8 Hz, 1H), 7.46 (d, *J* = 8.6 Hz, 1H), 7.34 (m, 1H), 2.45 (s, 3H). ¹³C NMR (100 MHz, DMSO) δ 192.7, 135.6, 135.4, 127.0, 125.3, 123.4, 116.2, 114.4, 114.2, 27.1. HRMS (ESI) Calcd for C₁₀H₉BrNO: [M+H]⁺, 237.9862; Found: 237.9854.

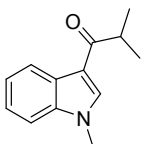
1-(1-methyl-1H-indol-3-yl)propan-1-one(3ca)



2a (0.5 mmol), **1c** (1.5 mmol), and HOTf (15 mol%) were introduced into a 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica

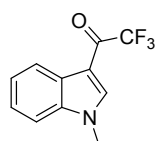
gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 75% of **3ca**. ^1H NMR (400 MHz, CDCl_3) δ 8.38 (m, 1H), 7.69 (s, 1H), 7.30 (m, 3H), 3.81 (s, 3H), 2.86 (q, $J = 7.4$ Hz, 2H), 1.25 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.4, 137.4, 135.1, 126.4, 123.2, 122.6, 122.5, 116.2, 109.6, 33.5, 32.9, 9.0. HRMS (ESI) Calcd for $\text{C}_{12}\text{H}_{13}\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 210.0889; Found: 210.0896.

2-methyl-1-(1-methyl-1H-indol-3-yl)propan-1-one(3da)



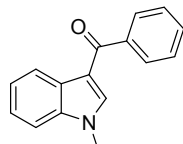
2a (0.5 mmol), **1d** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 74% of **3da**. ^1H NMR (400 MHz, CDCl_3) δ 8.37 – 8.31 (m, 1H), 7.67 (s, 1H), 7.27 – 7.22 (m, 3H), 3.77 (s, 3H), 3.24 (m, 1H), 1.18 (d, $J = 6.8$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 199.0, 136.5, 133.9, 125.6, 122.3, 121.8, 121.5, 114.3, 108.5, 36.1, 32.5, 18.7. HRMS (ESI) Calcd for $\text{C}_{13}\text{H}_{15}\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 224.1046; Found: 224.1058.

2,2,2-trifluoro-1-(1-methyl-1H-indol-3-yl)ethanone(3ea)



2a (0.5 mmol), **1e** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 80 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1 with 0.5 mL triethylamine) to afford 79% of **3ea**. ^1H NMR (400 MHz, CDCl_3) δ 8.50 – 8.34 (m, 1H), 7.91 (d, $J = 1.6$ Hz, 1H), 7.45 – 7.35 (m, 3H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.9, 174.5, 138.3, 138.2, 137.3, 126.9, 124.6, 124.0, 122.6, 110.1, 109.5, 34.0. HRMS (ESI) Calcd for $\text{C}_{11}\text{H}_8\text{F}_3\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 250.0450; Found: 250.0472.

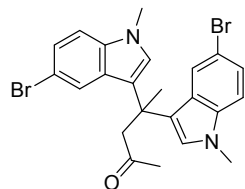
(1-methyl-1H-indol-3-yl)(phenyl)methanone(3fa)



2a (0.5 mmol), **1f** (1.5 mmol), HOTf (0.5 mmol) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 130 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 38% of **3fa**.

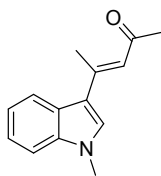
2a (0.5 mmol), **1f** (1.5 mmol), $\text{Fe}(\text{OTf})_3$ (0.05 mmol) and $(\text{CF}_3\text{SO}_2)_2\text{O}$ (0.5 mmol) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at 130 °C for 24 h. After cooling to room temperature, the resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 5:1 with 0.5 mL triethylamine) to afford 49% of **3fa**. ^1H NMR (400 MHz, CDCl_3) δ 8.53 – 8.33 (m, 1H), 7.87 – 7.76 (m, 2H), 7.57 – 7.45 (m, 4H), 7.39 – 7.31 (m, 3H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 190.9, 140.9, 137.9, 137.6, 131.1, 128.7, 128.3, 127.2, 123.7, 122.7, 115.6, 109.6, 33.6. HRMS (ESI) Calcd for $\text{C}_{16}\text{H}_{13}\text{NNaO}$: $[\text{M}+\text{Na}]^+$, 258.0889; Found: 258.0888.

4,4-bis(5-bromo-1-methyl-1H-indol-3-yl)pentan-2-one (D2)



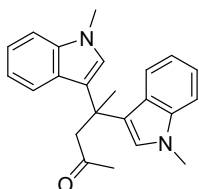
2j (0.25 mmol), **1b** (0.75 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1) to afford 61% of **D2** as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.33 (d, 2H), 7.16 (s, 2H), 7.11 (m, 2H), 6.84 (s, 2H), 3.64 (s, 6H), 3.23 (s, 2H), 1.84 (s, 3H), 1.50 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 207.4, 135.4, 126.8, 126.2, 123.2, 122.0, 119.7, 111.0, 109.9, 52.1, 36.4, 31.9, 31.1, 26.4. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{22}\text{Br}_2\text{N}_2\text{NaO}$: $[\text{M}+\text{Na}]^+$, 524.9983; Found: 524.9993.

(E)-4-(1-methyl-1H-indol-3-yl)pent-3-en-2-one(C1)



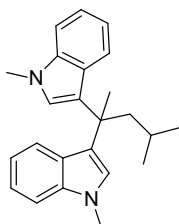
2a (0.5 mmol), **1b** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1) to afford 17% of **C2** as a pale yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 7.8 Hz, 1H), 7.41 – 7.22 (m, 4H), 6.83 (s, 1H), 3.81 (s, 3H), 2.63 (d, *J* = 0.9 Hz, 3H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 198.8, 149.1, 138.1, 131.3, 125.4, 122.7, 121.1, 120.3, 117.7, 110.0, 33.2, 32.4, 18.7. HRMS (ESI) Calcd for C₁₄H₁₅NNaO: [M+Na]⁺, 236.1046; Found: 236.1045.

4,4-bis(1-methyl-1H-indol-3-yl)pentan-2-one(D1)



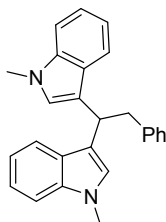
2a (0.5 mmol), **1b** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1) to afford 58% of **D2** as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.39–7.37 (m, 2H), 7.24 – 7.22 (m, 2H), 7.13–7.09 (m, 2H), 6.90–6.86 (m, 4H), 3.72 (s, 6H), 3.49 (s, 2H), 1.97 (s, 3H), 1.51 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 209.5, 137.7, 126.4, 126.3, 121.5, 121.2, 121.1, 118.5, 109.3, 53.6, 37.9, 32.8, 32.1, 27.7. HRMS (ESI) Calcd for C₂₃H₂₄N₂NaO: [M+Na]⁺, 367.1805; Found: 367.1814.

3,3'-(4-methylpentane-2,2-diyl)bis(1-methyl-1H-indole)(F1)



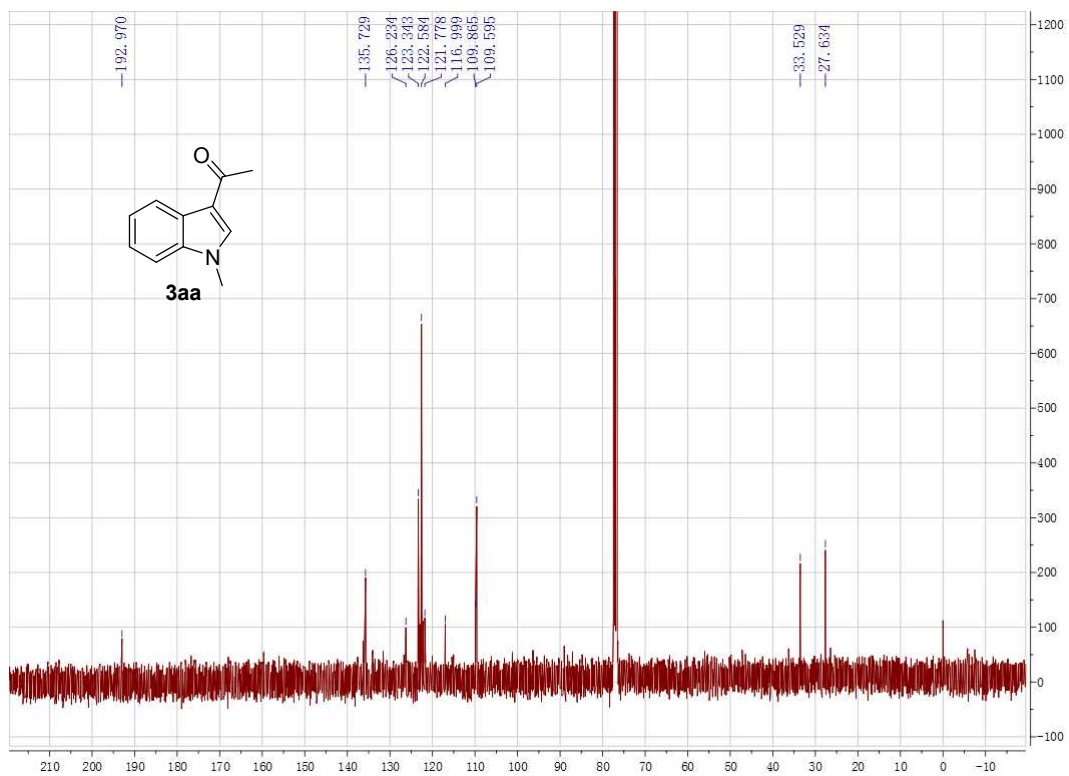
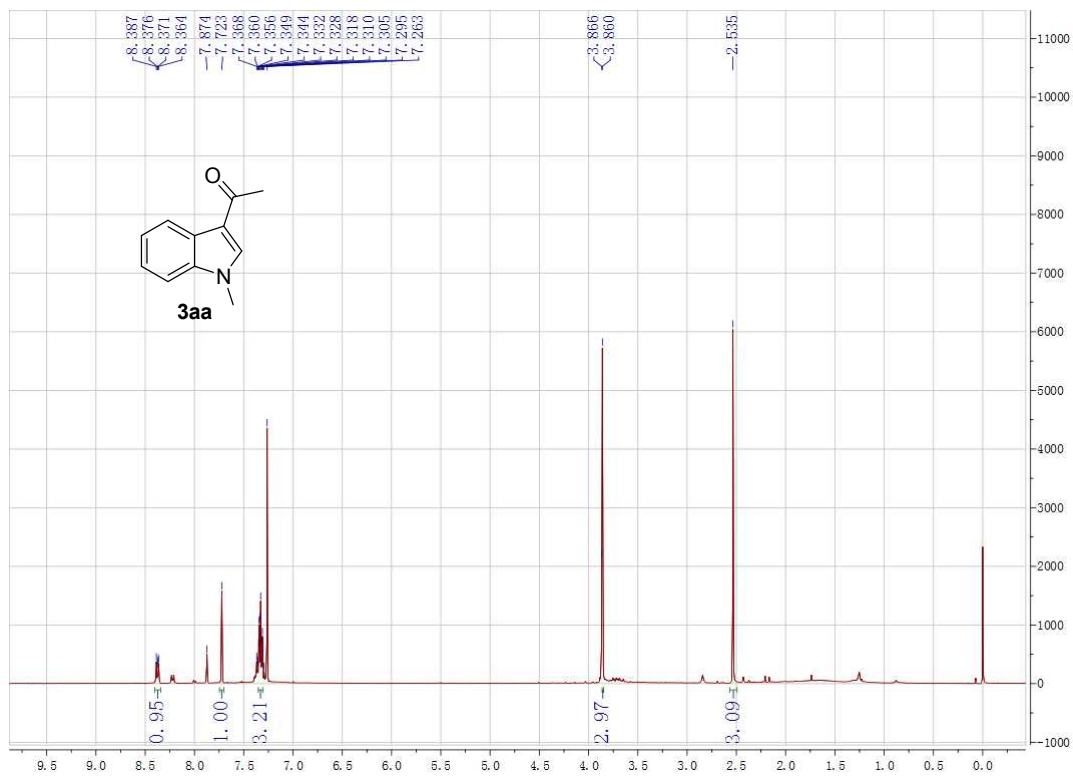
2a (0.5 mmol), **1k** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1) to afford 69% of **F1** as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 7.04 – 6.97 (m, 2H), 6.86 – 6.70 (m, 4H), 3.62 (s, 6H), 2.25 (d, *J* = 5.1 Hz, 2H), 1.77 (s, 3H), 0.63 (d, *J* = 6.7 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 136.1, 125.4, 124.5, 121.7, 120.0, 119.2, 116.4, 107.4, 47.7, 37.3, 31.1, 26.3, 23.7, 23.2.

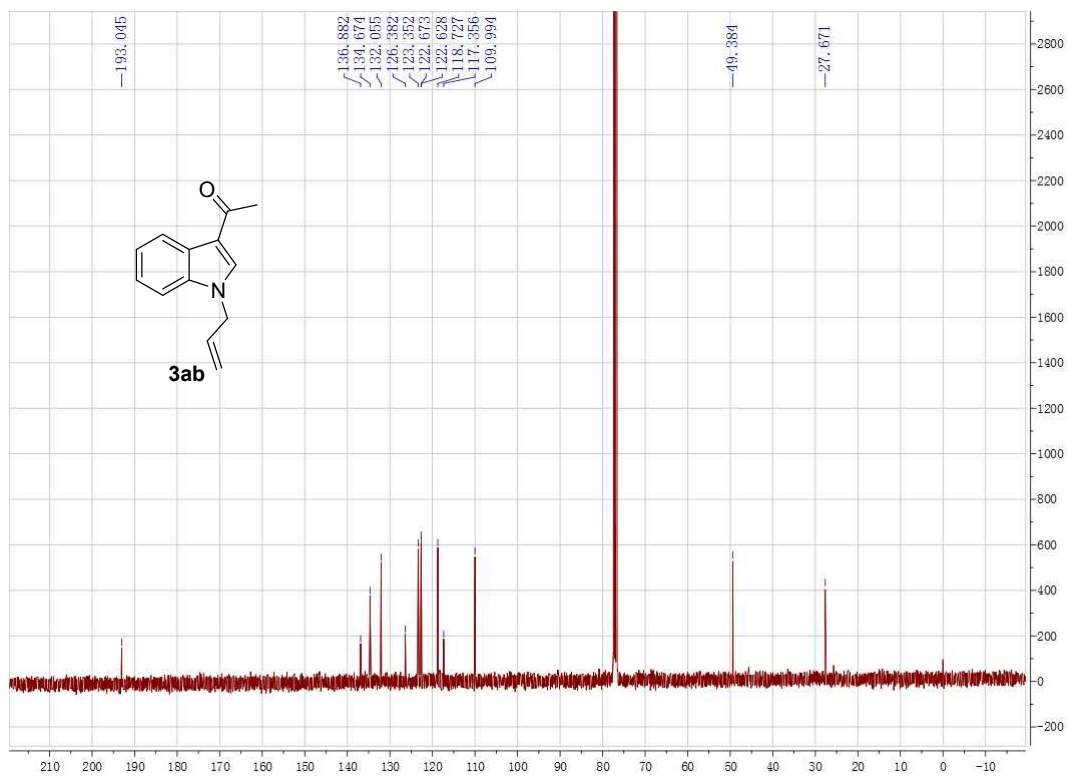
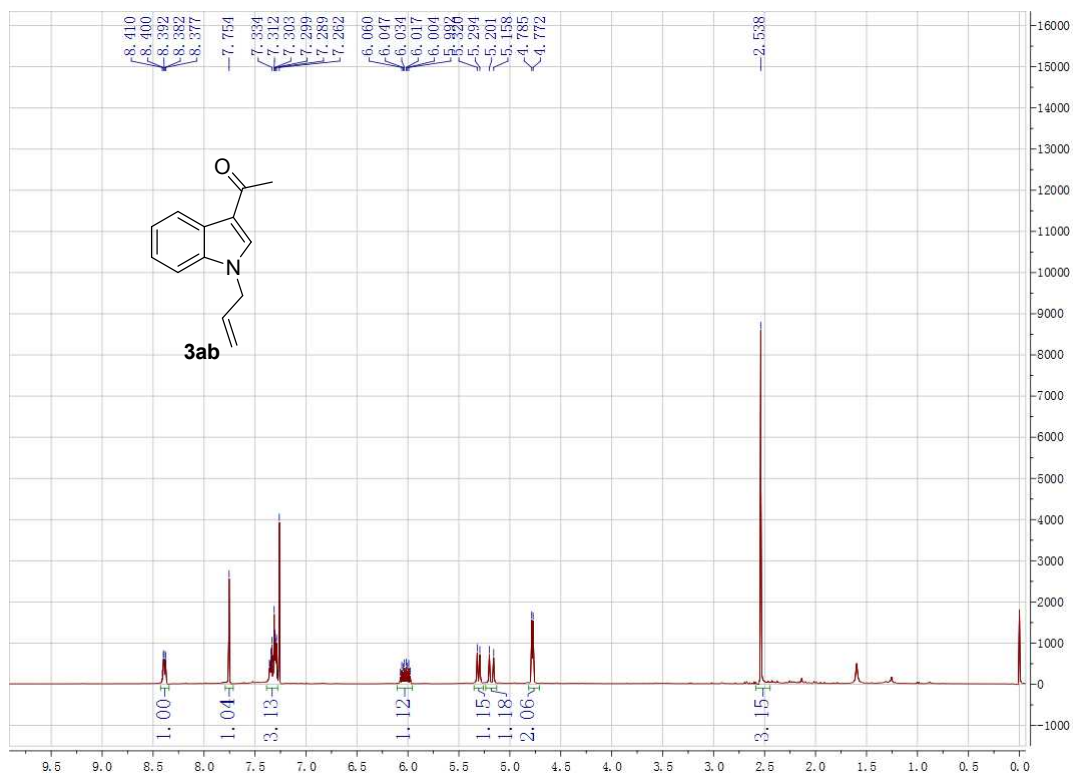
3,3'-(2-phenylethane-1,1-diyl)bis(1-methyl-1H-indole)(F2)

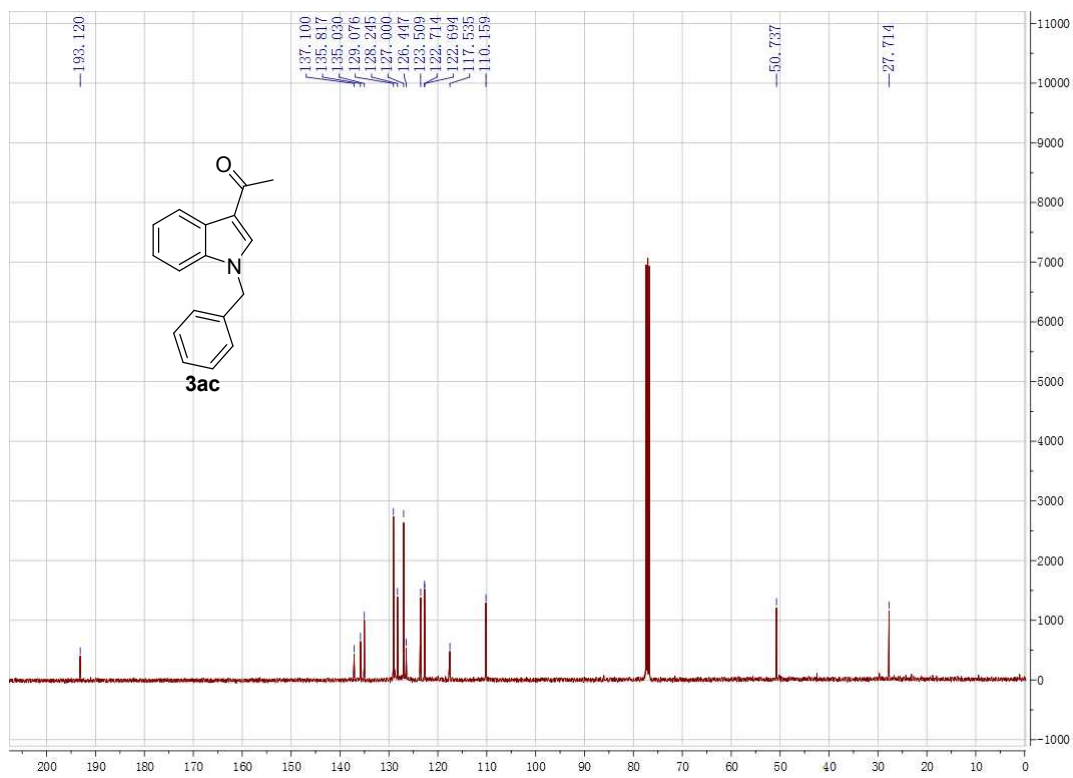
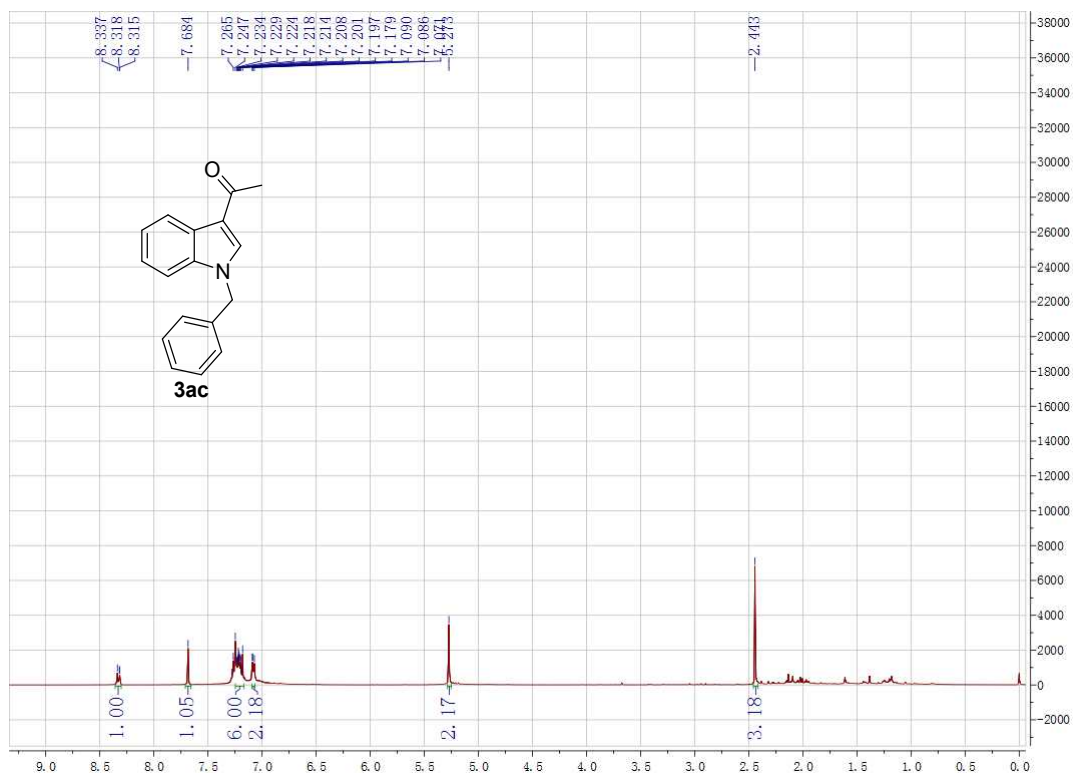


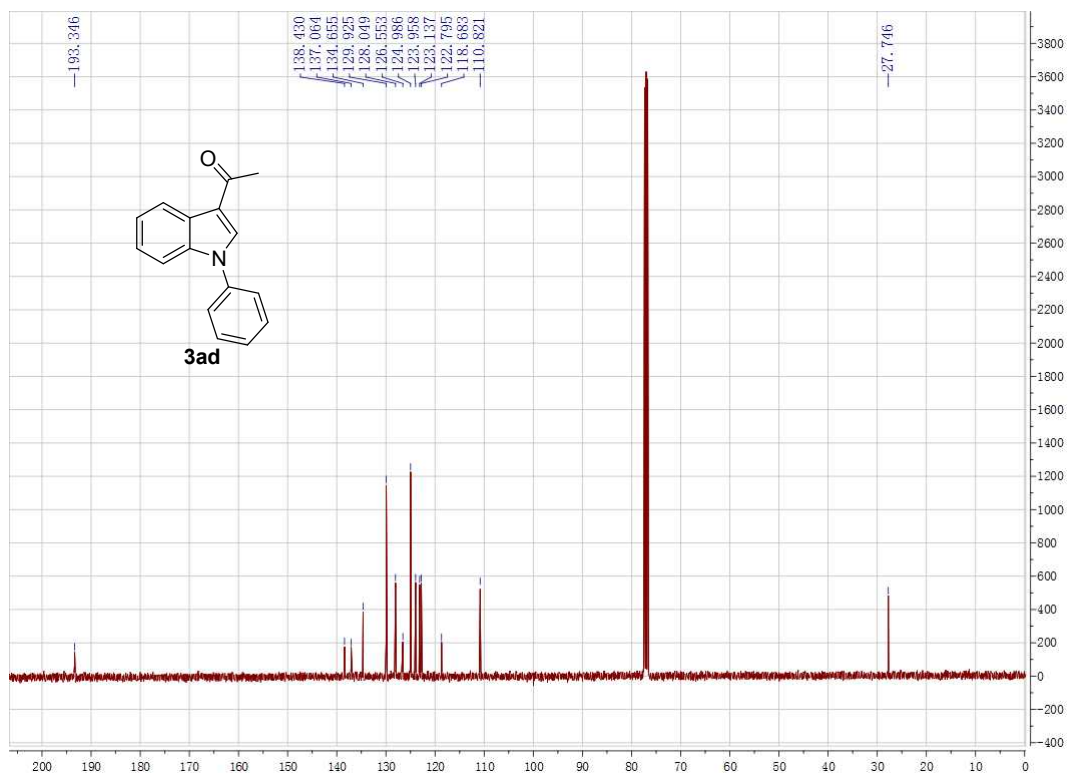
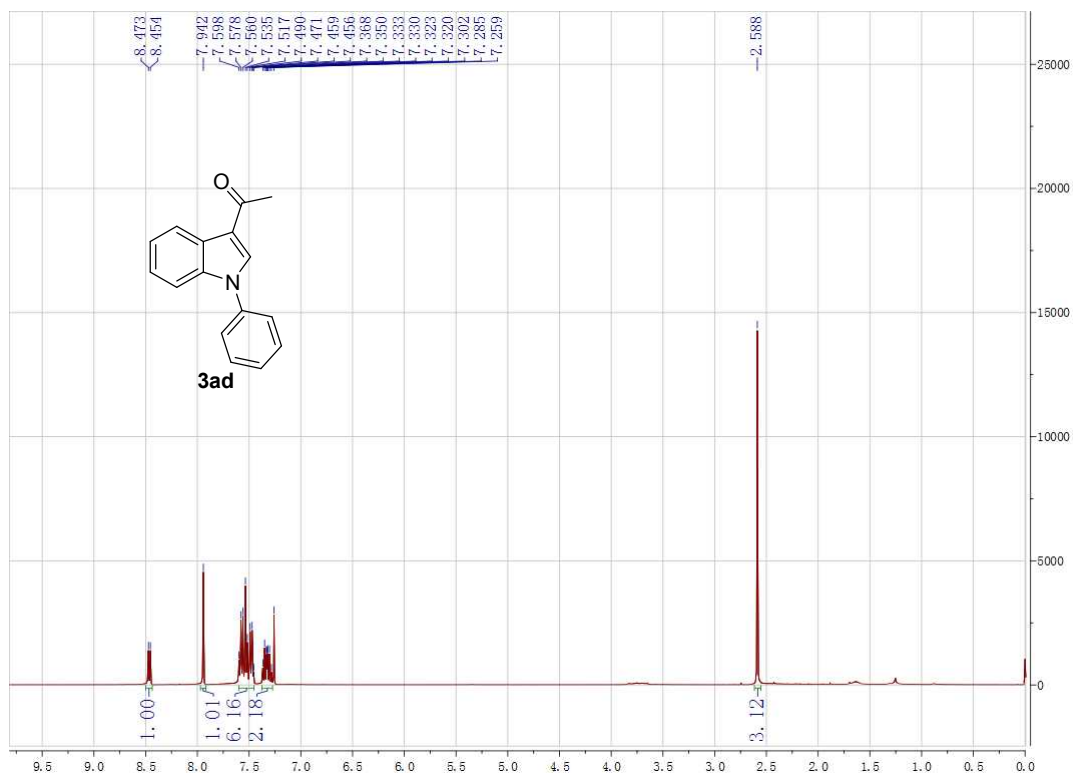
2a (0.5 mmol), **1l** (1.5 mmol), and HOTf (15 mol%) were introduced into an 25 mL Schlenk tube. The formed mixture was stirred at room temperature for 12 h. The resultant reaction mixture was purified by flash chromatography using silica gel (petroleum ether / ethyl acetate = 10:1) to afford 33% of **F2** as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.15 – 6.99 (m, 9H), 6.95 – 6.90 (m, 2H), 6.68 (s, 2H), 4.69 (t, *J* = 7.4 Hz, 1H), 3.53 (s, 6H), 3.42 (d, *J* = 7.6 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 141.5, 137.4, 129.1, 128.1, 126.8, 121.4, 119.8, 118.6, 118.3, 109.2, 42.3, 36.1, 32.7.

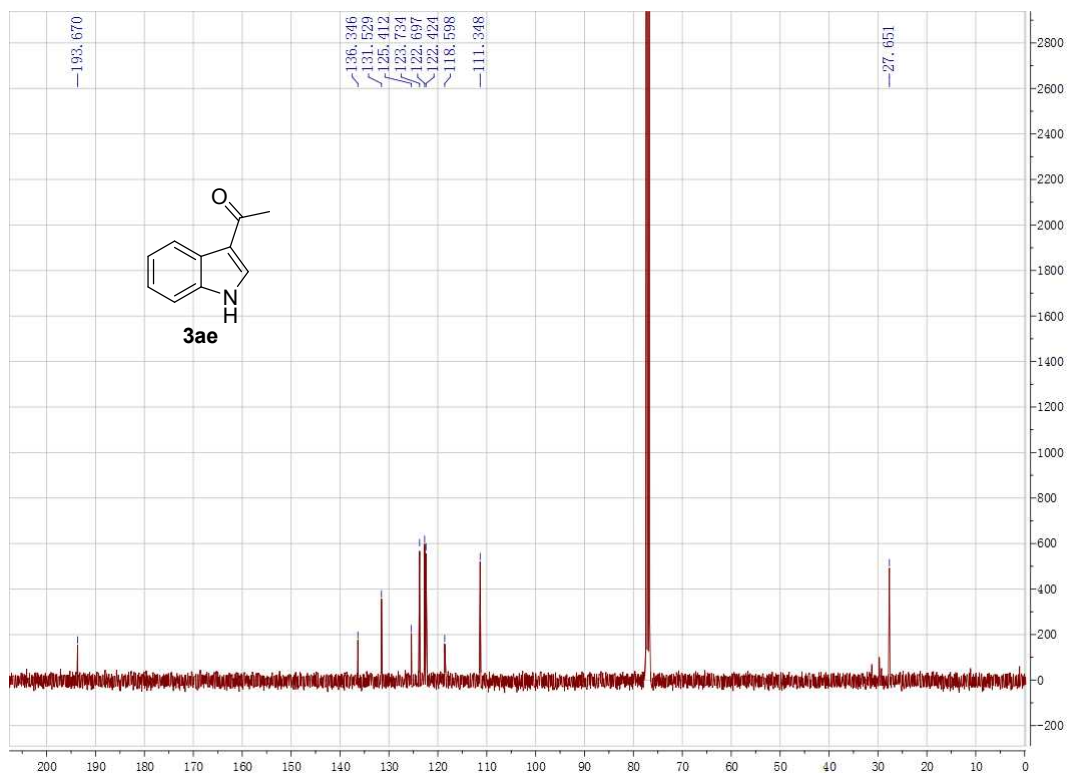
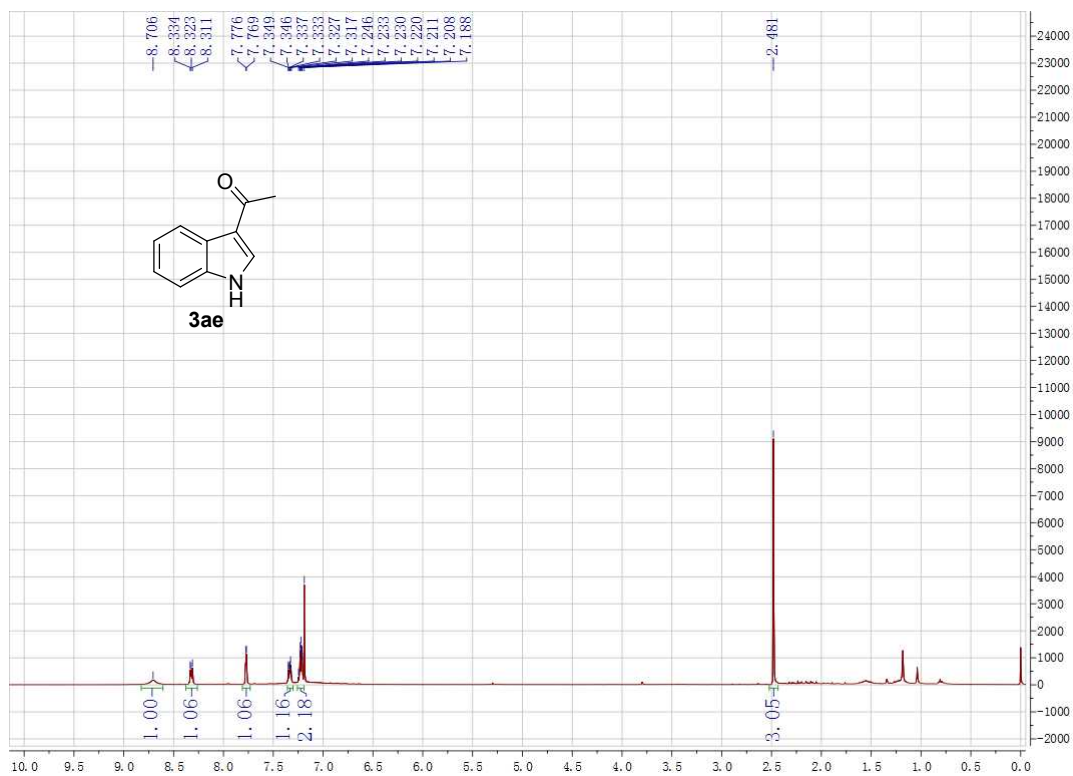
6) ^1H NMR and ^{13}C NMR Copies of Products

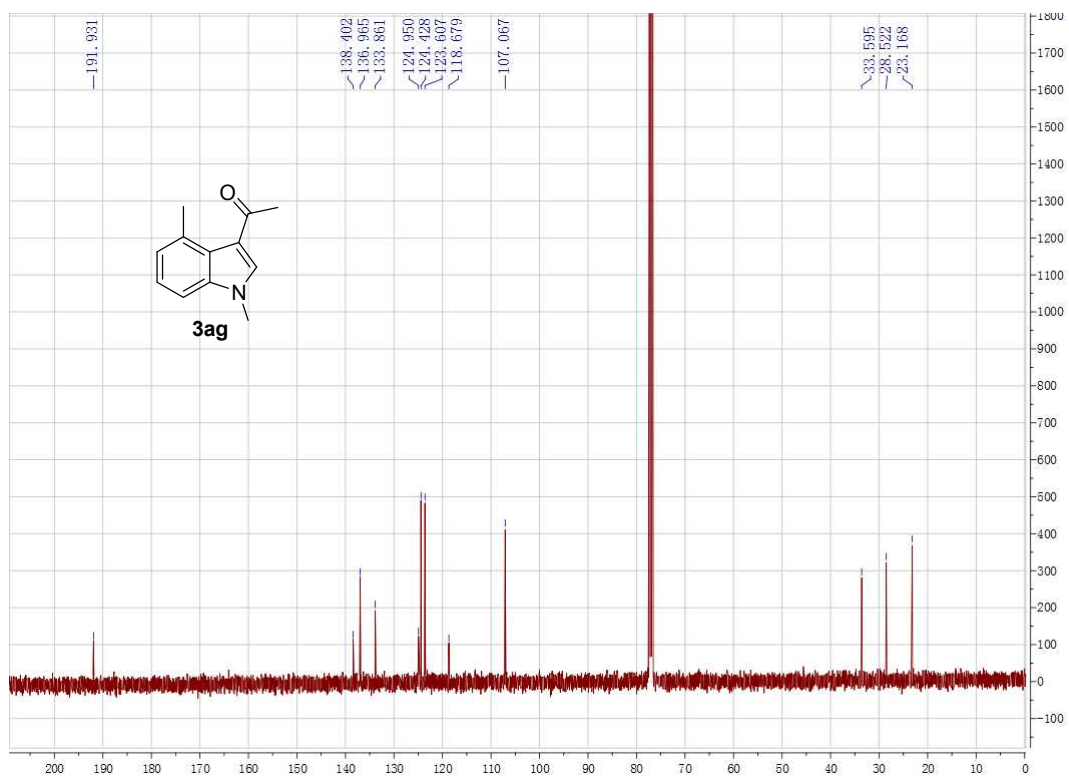
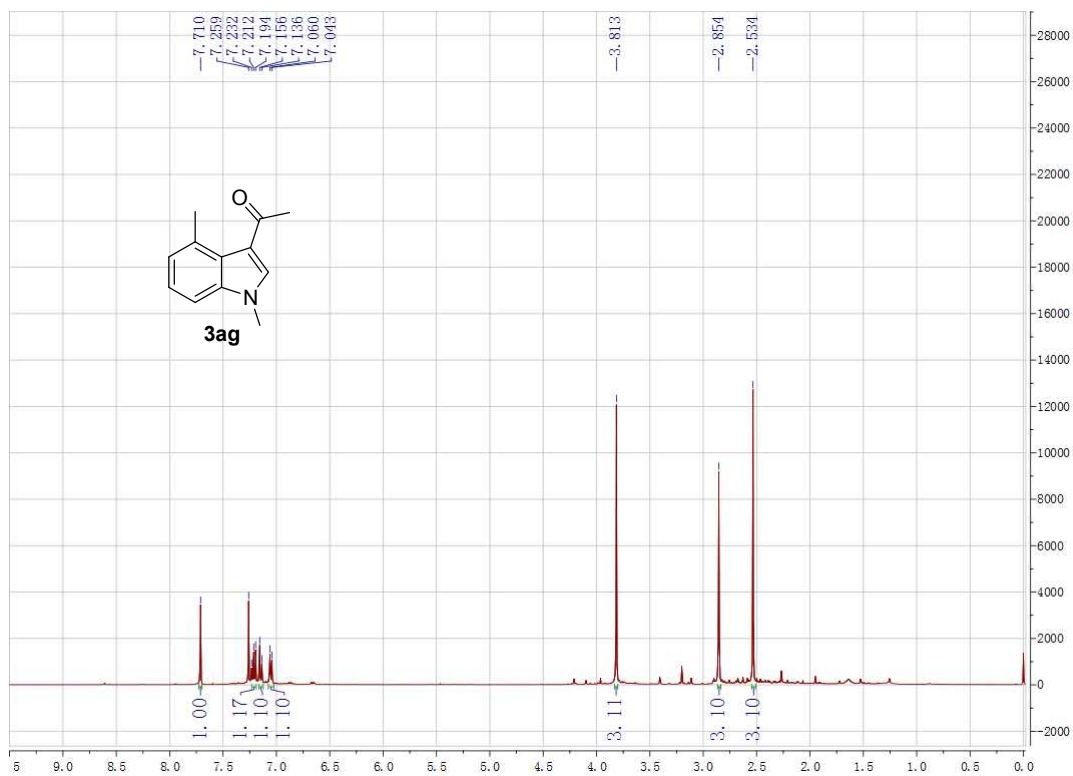


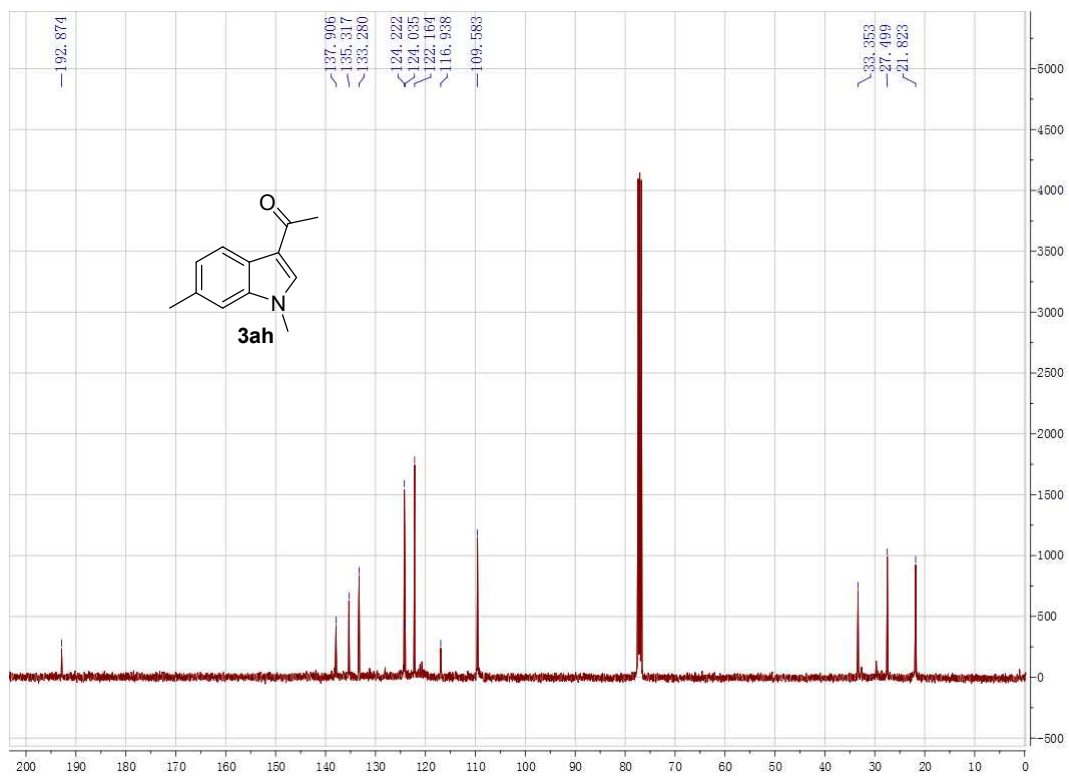
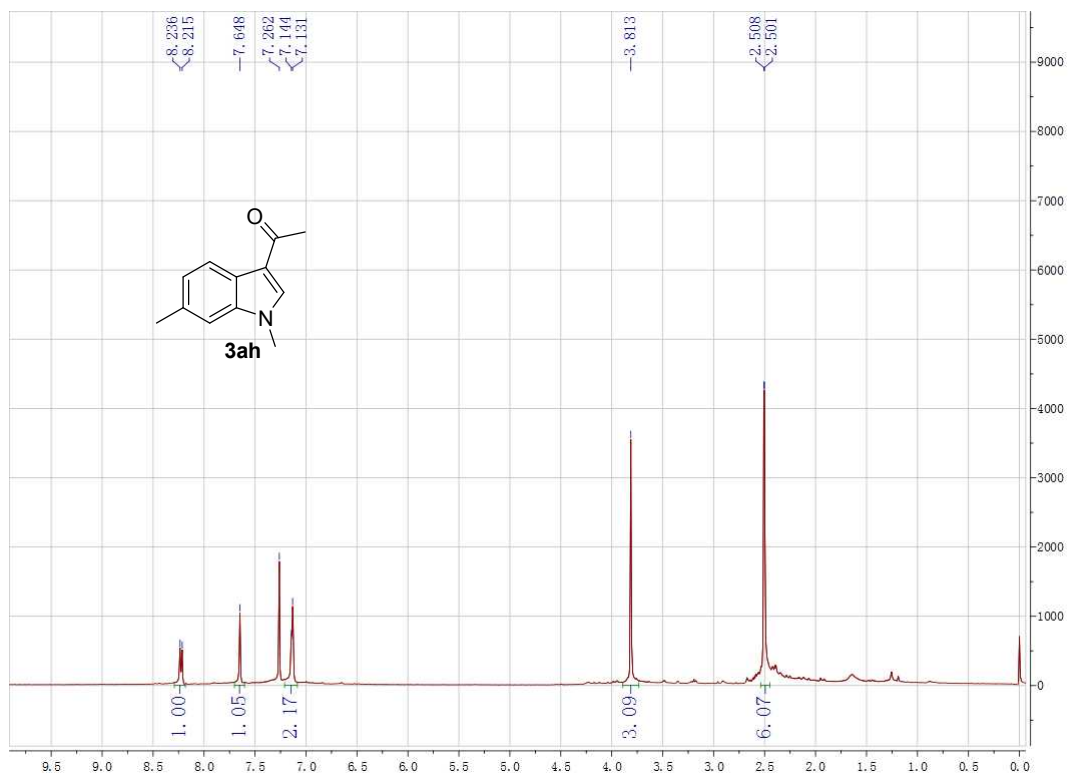


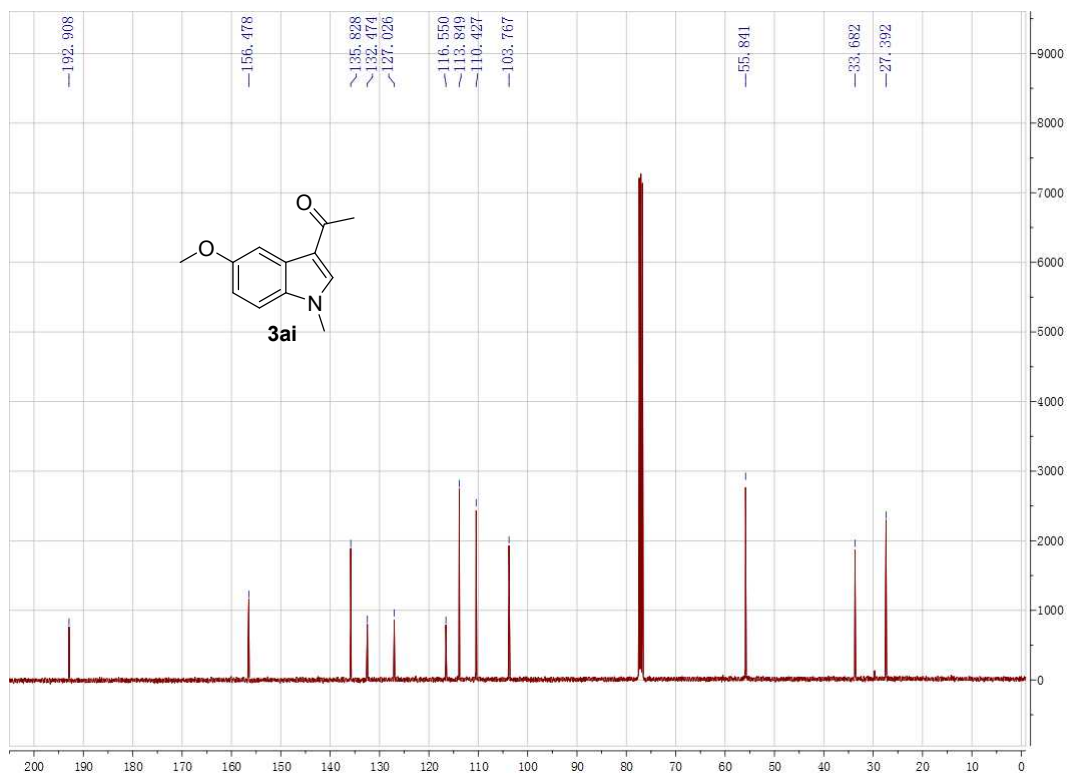
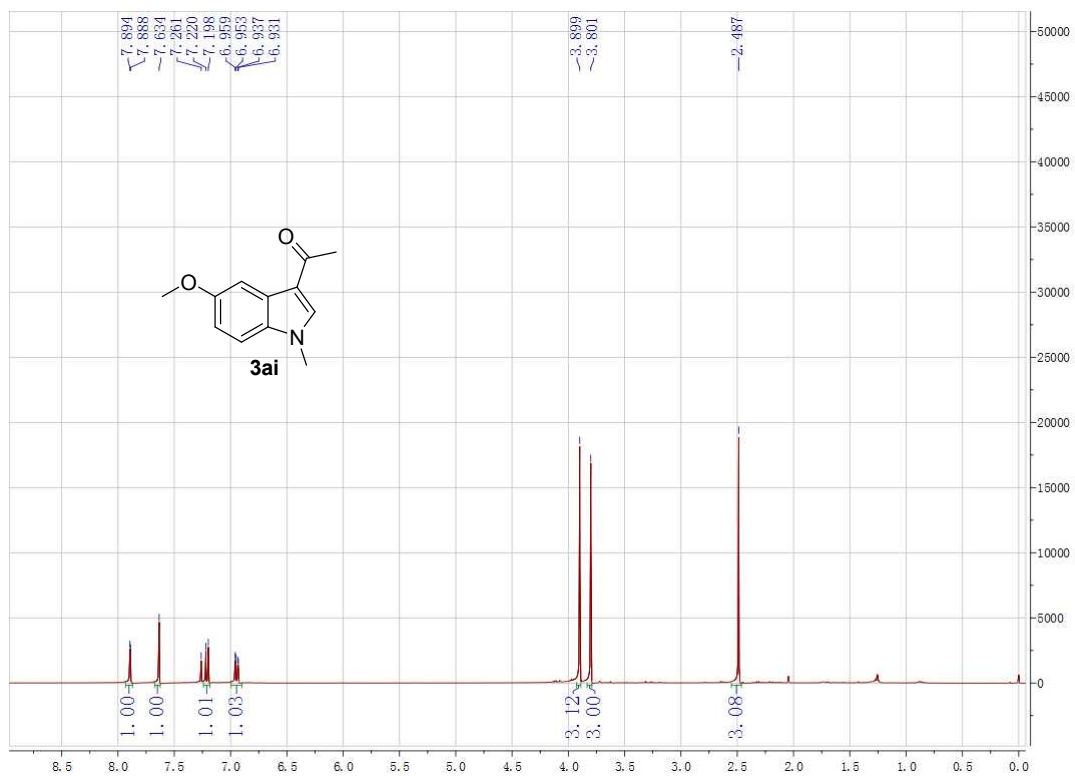


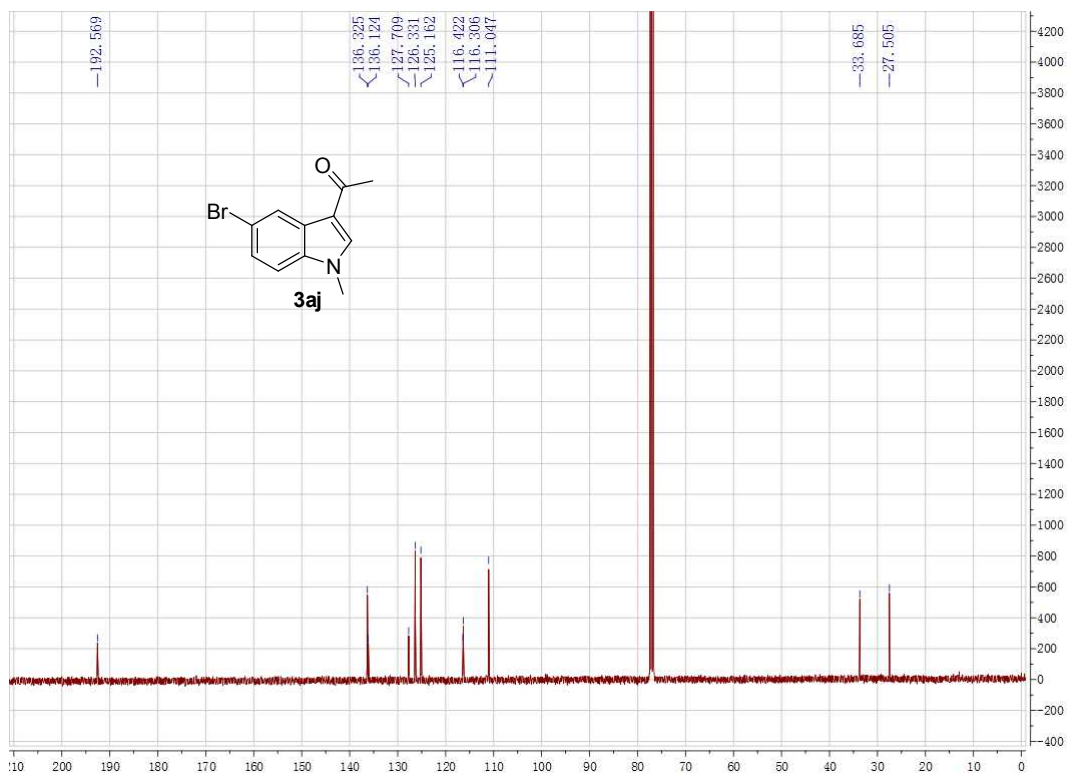
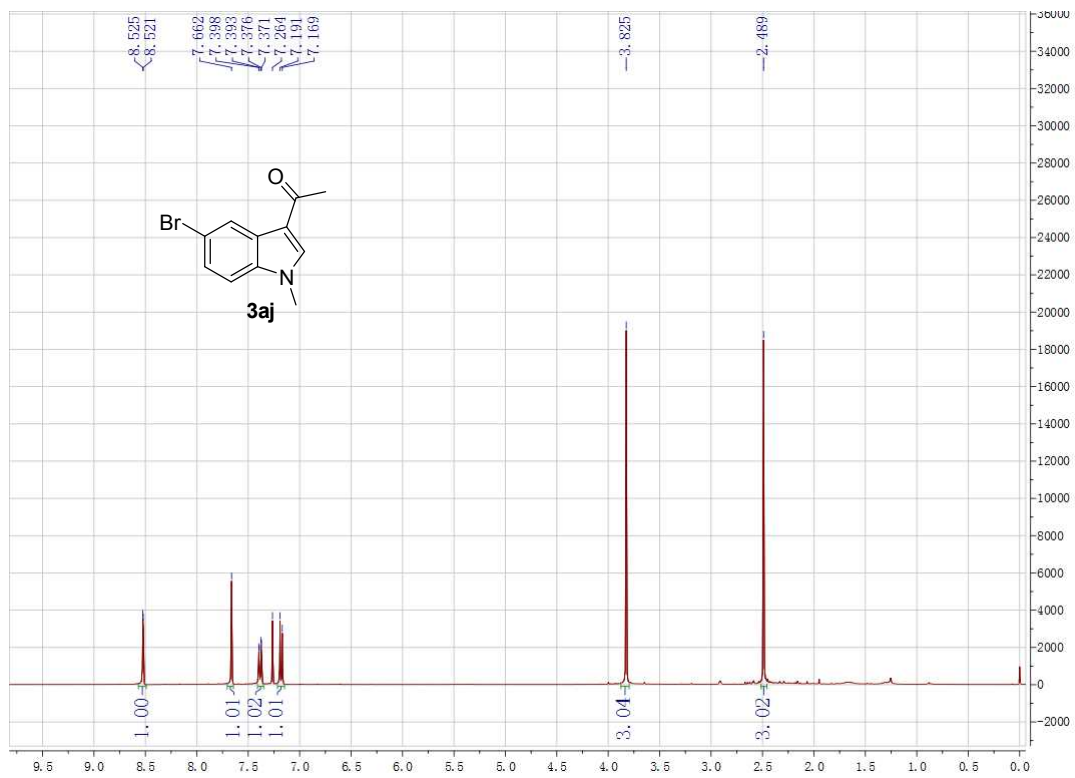


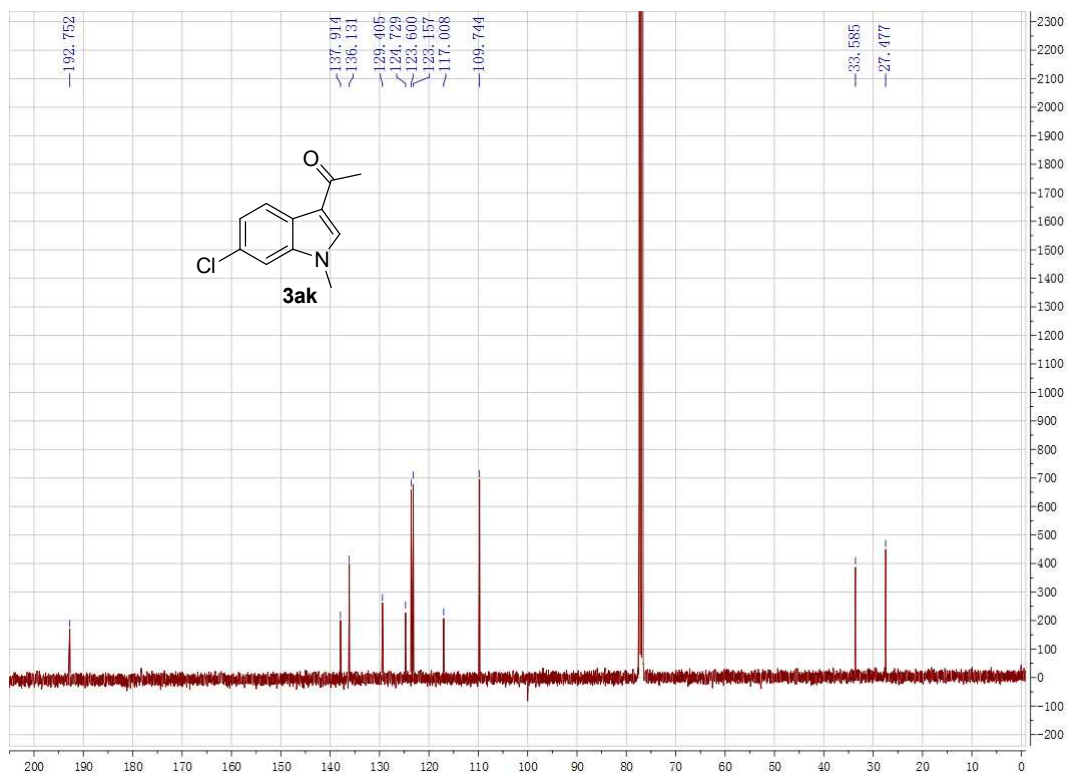
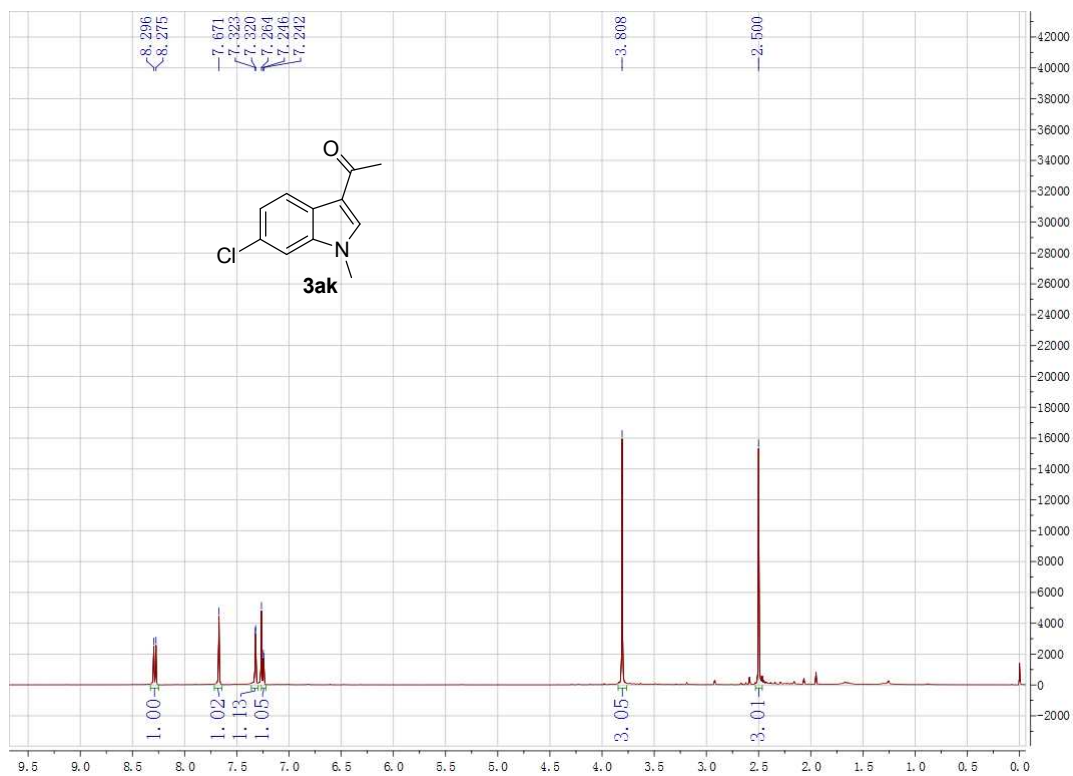


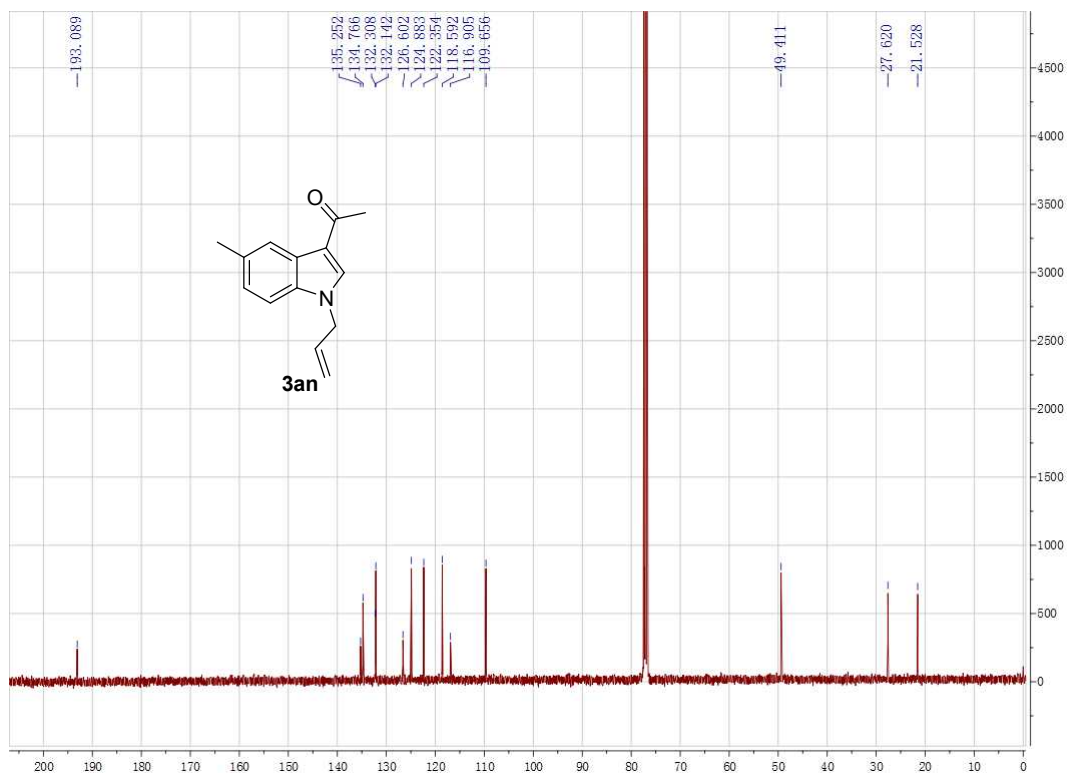
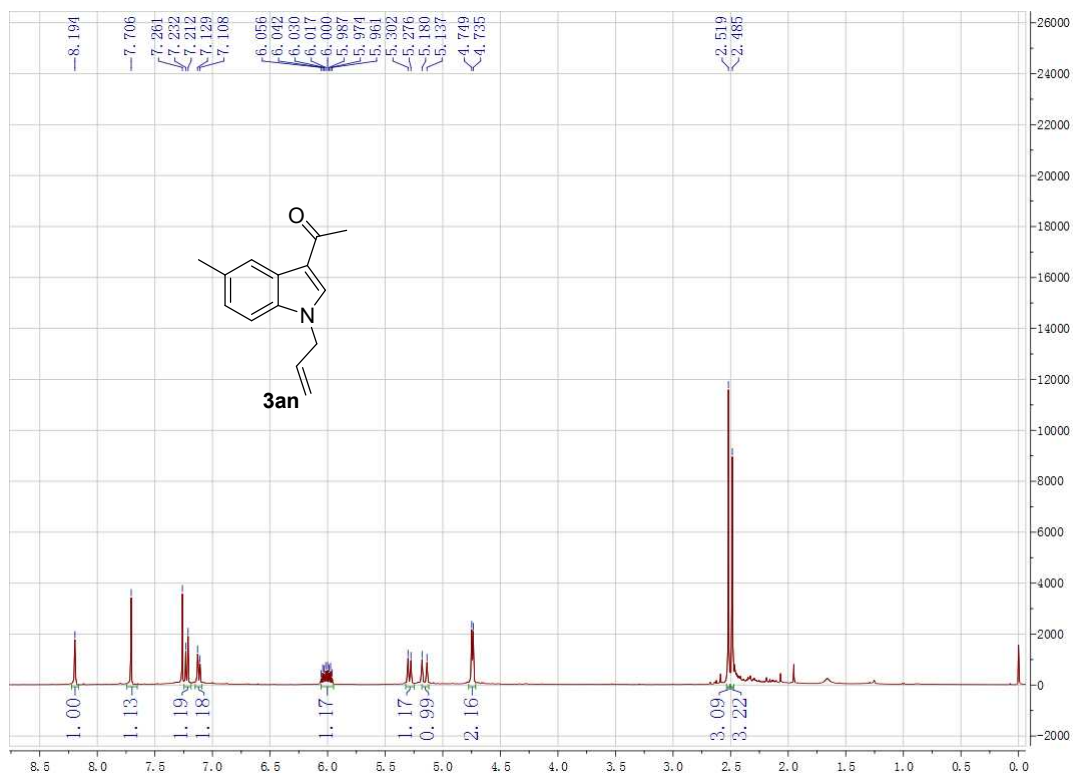


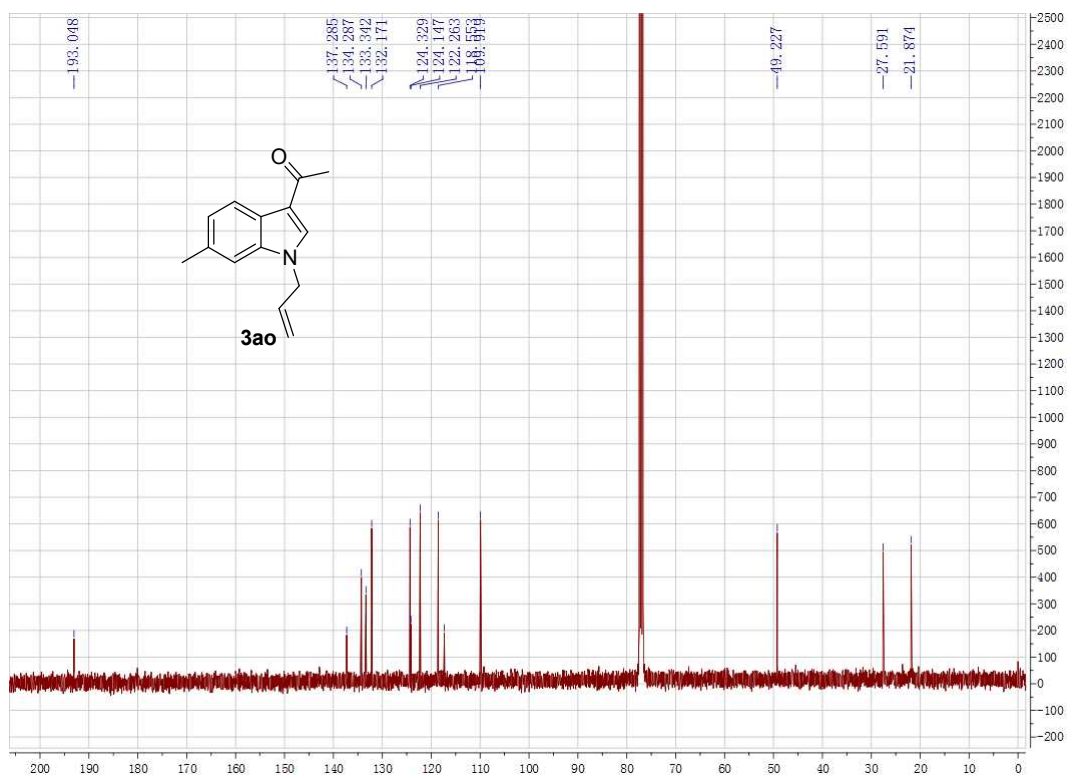
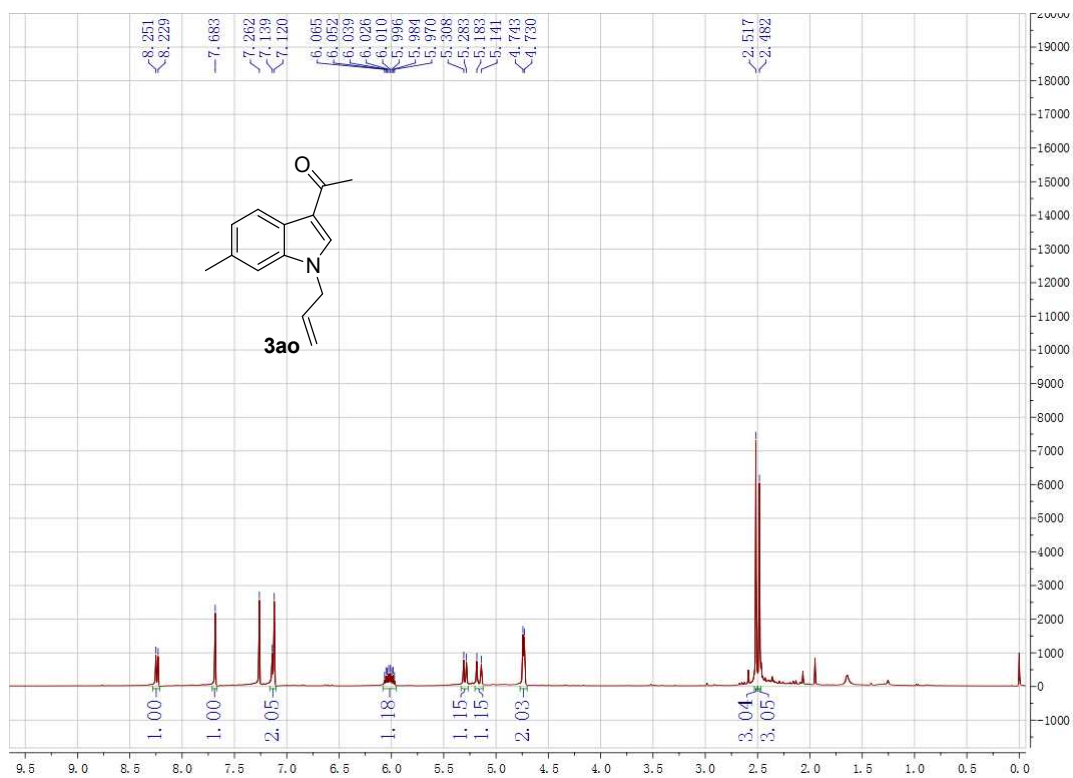


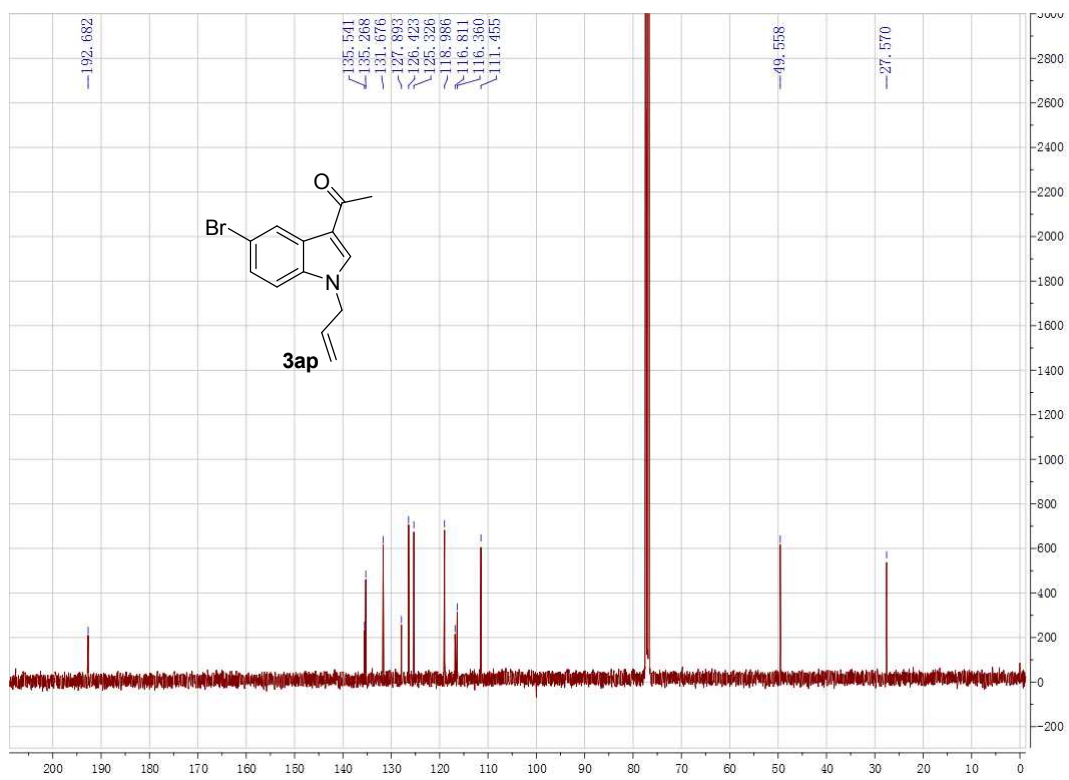
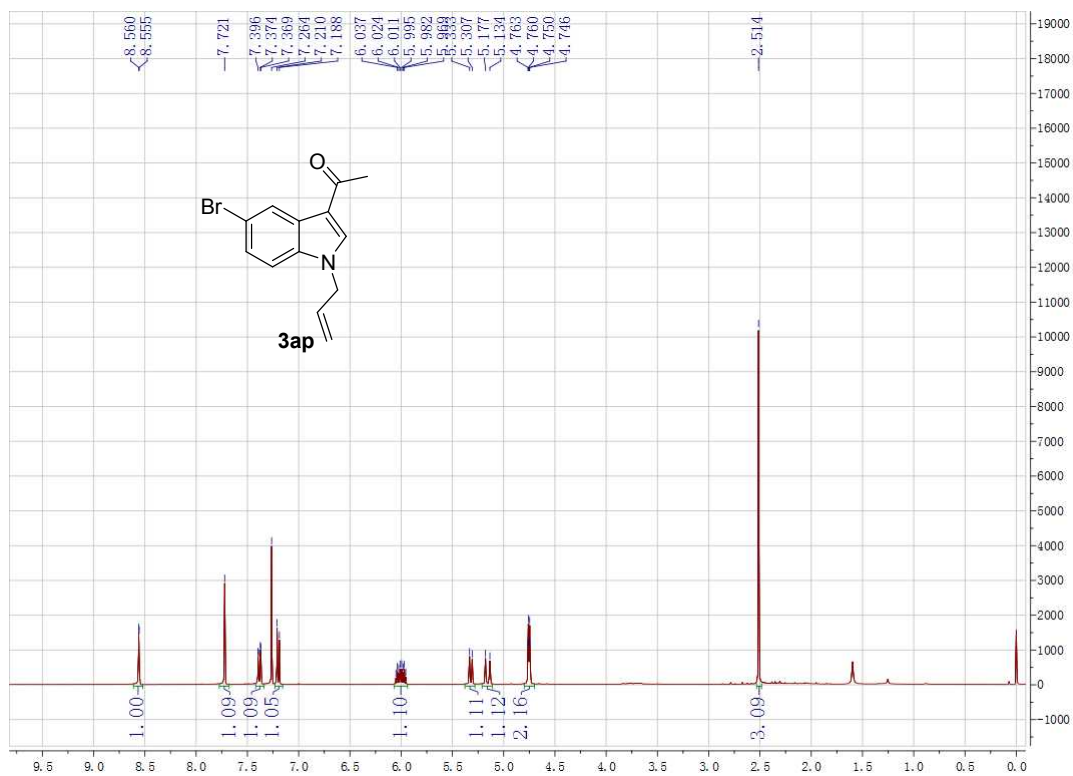


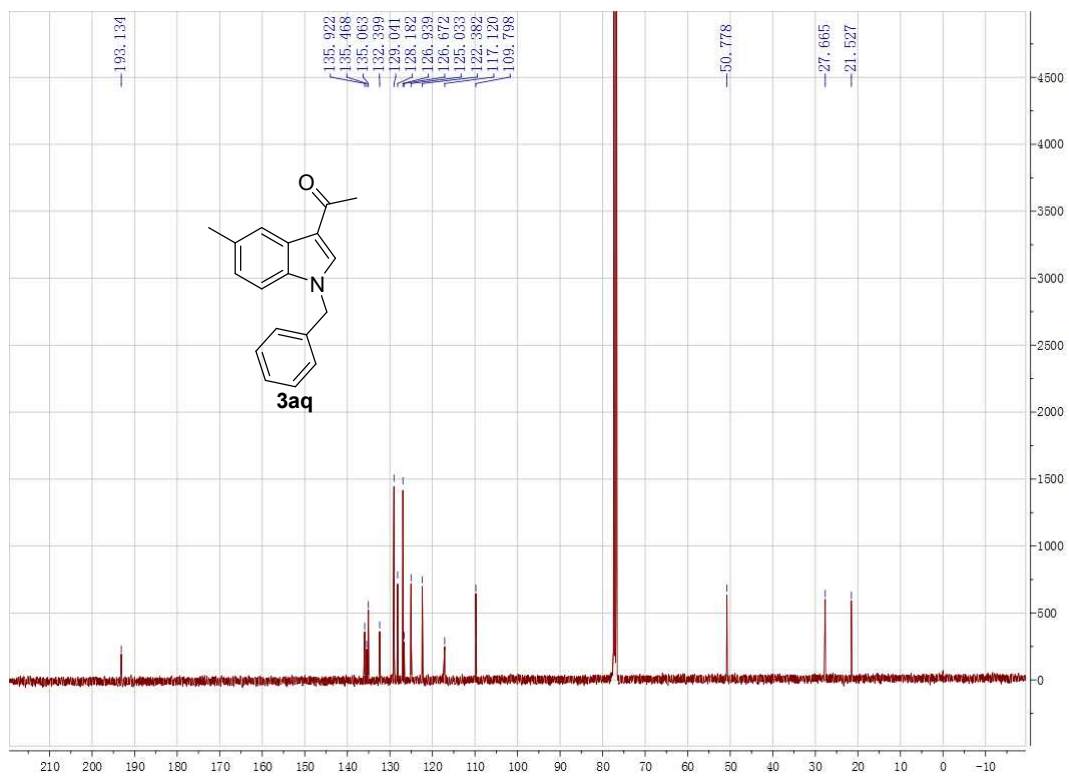
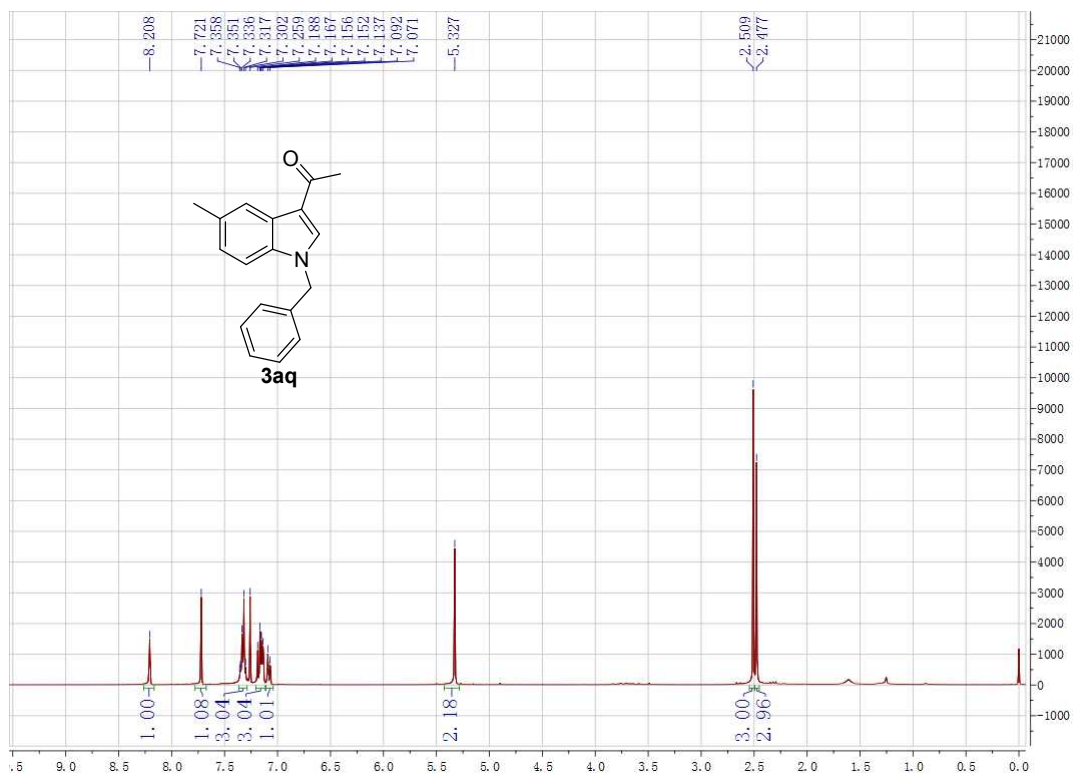


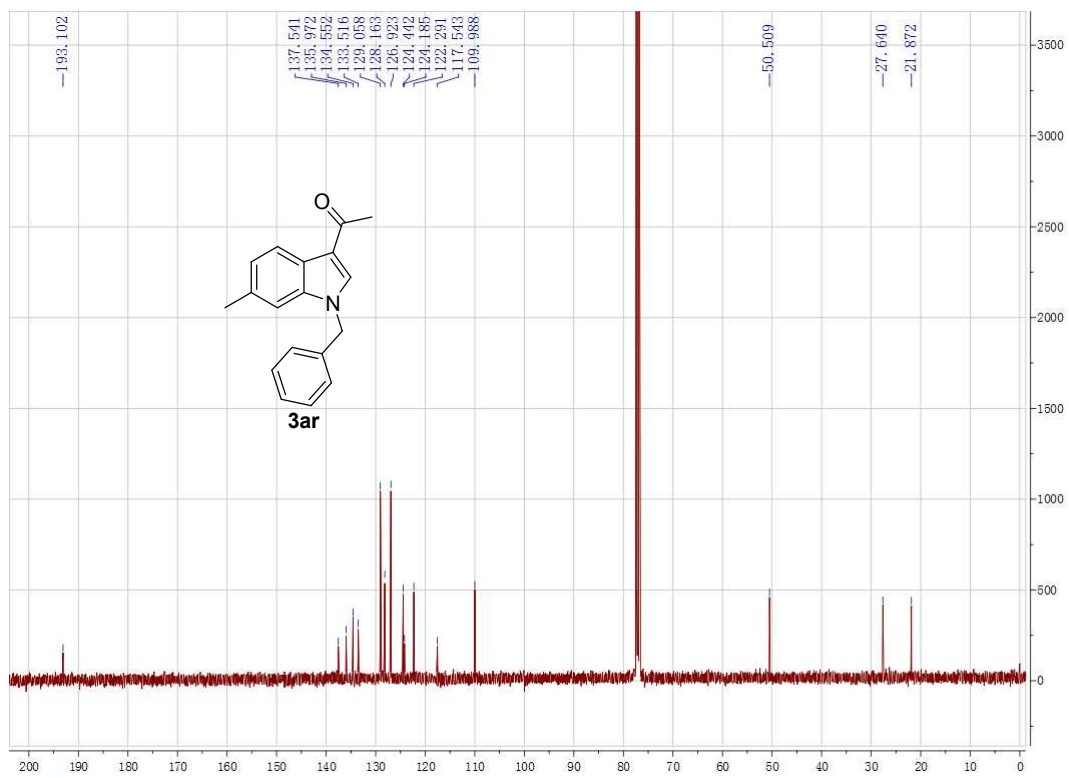
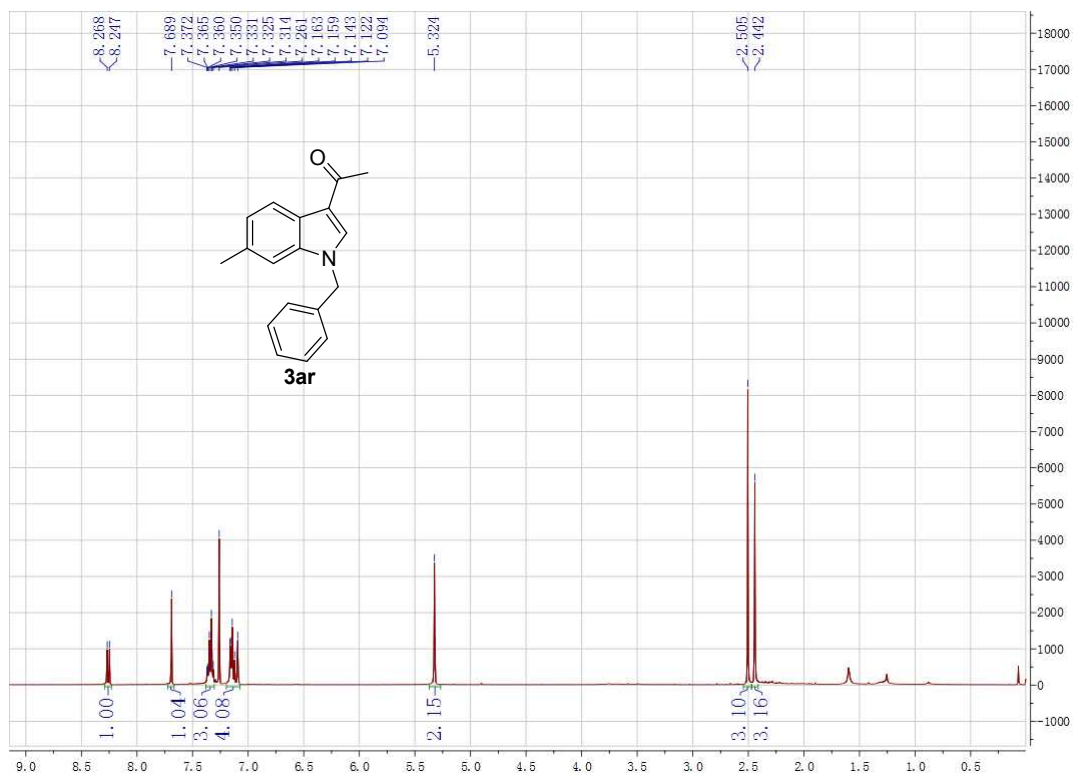


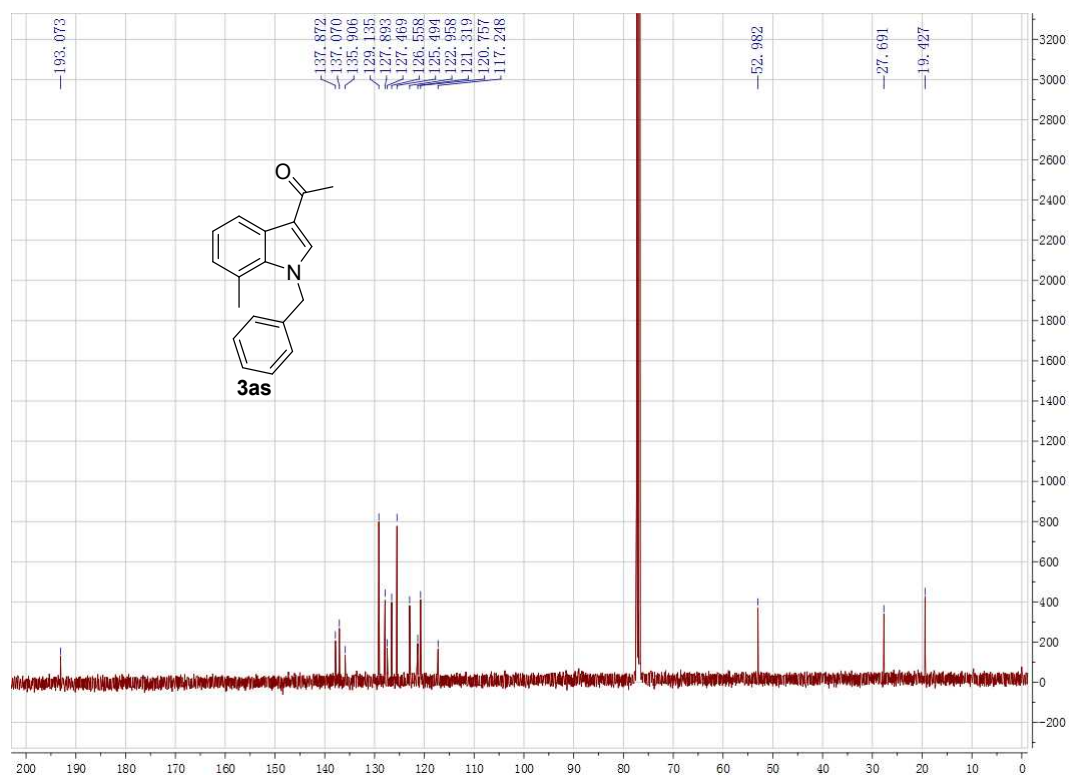
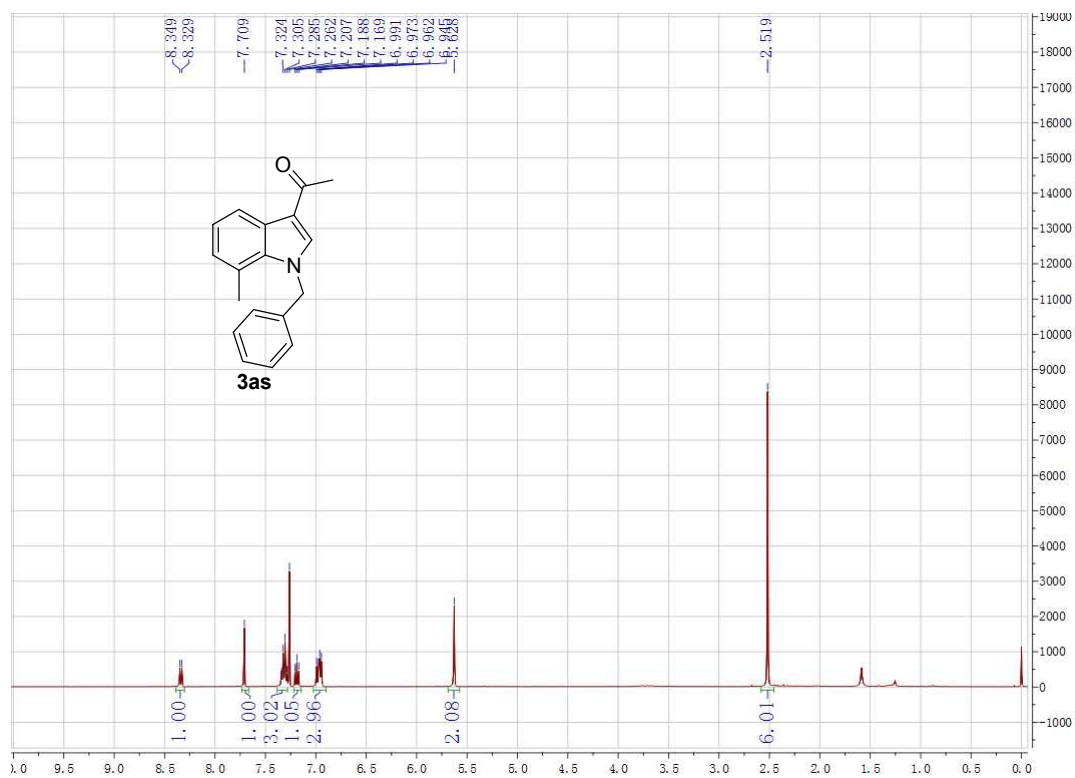


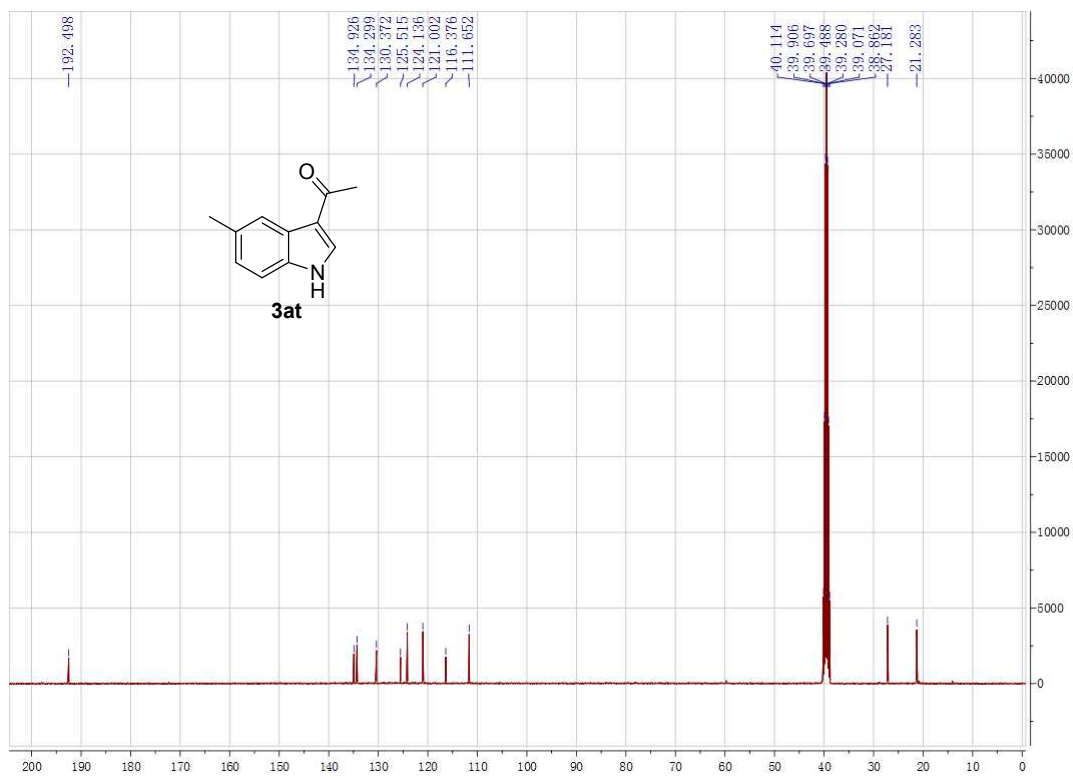
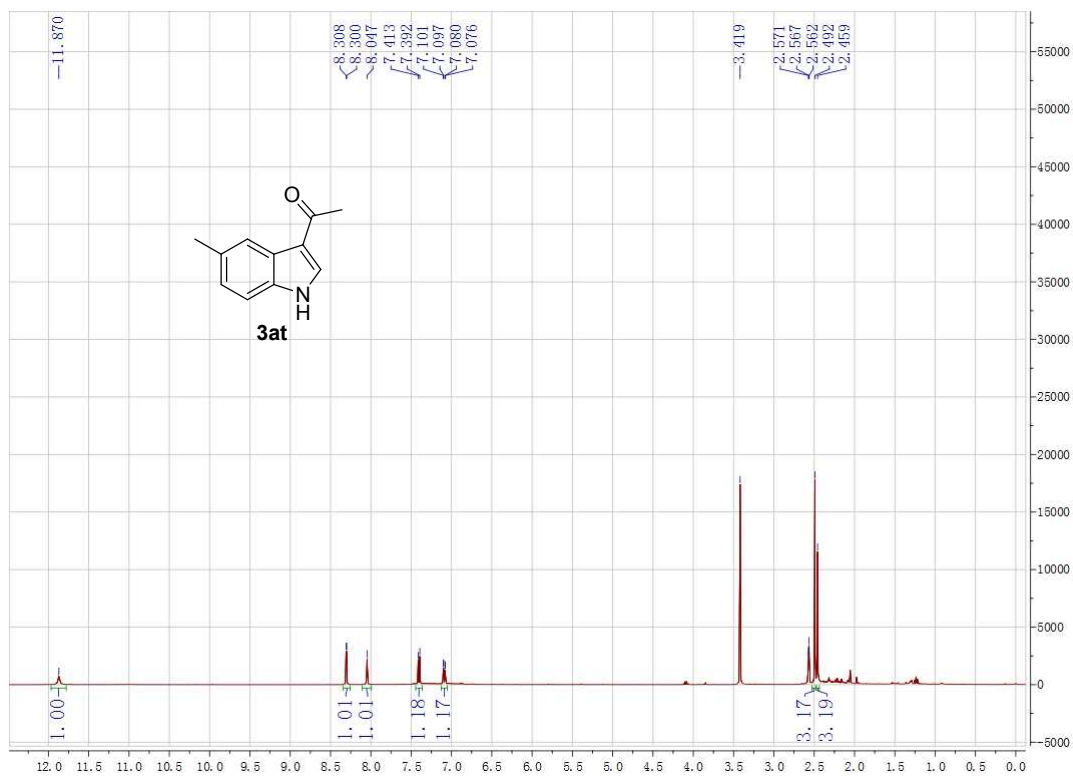


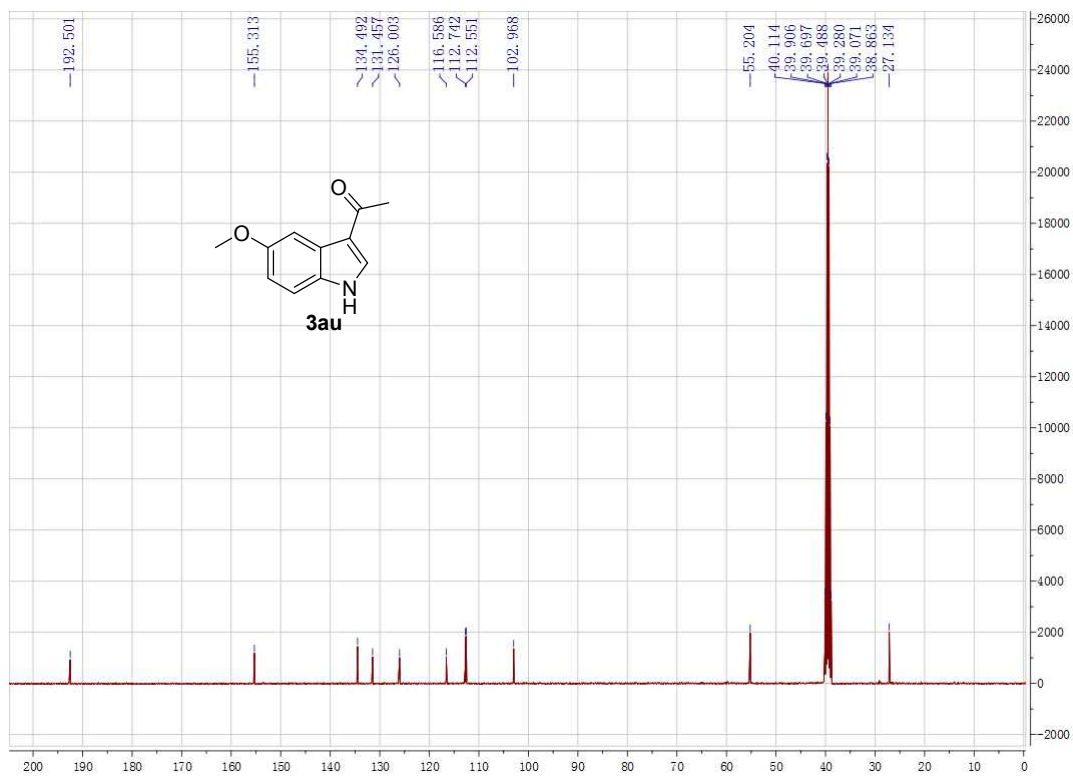
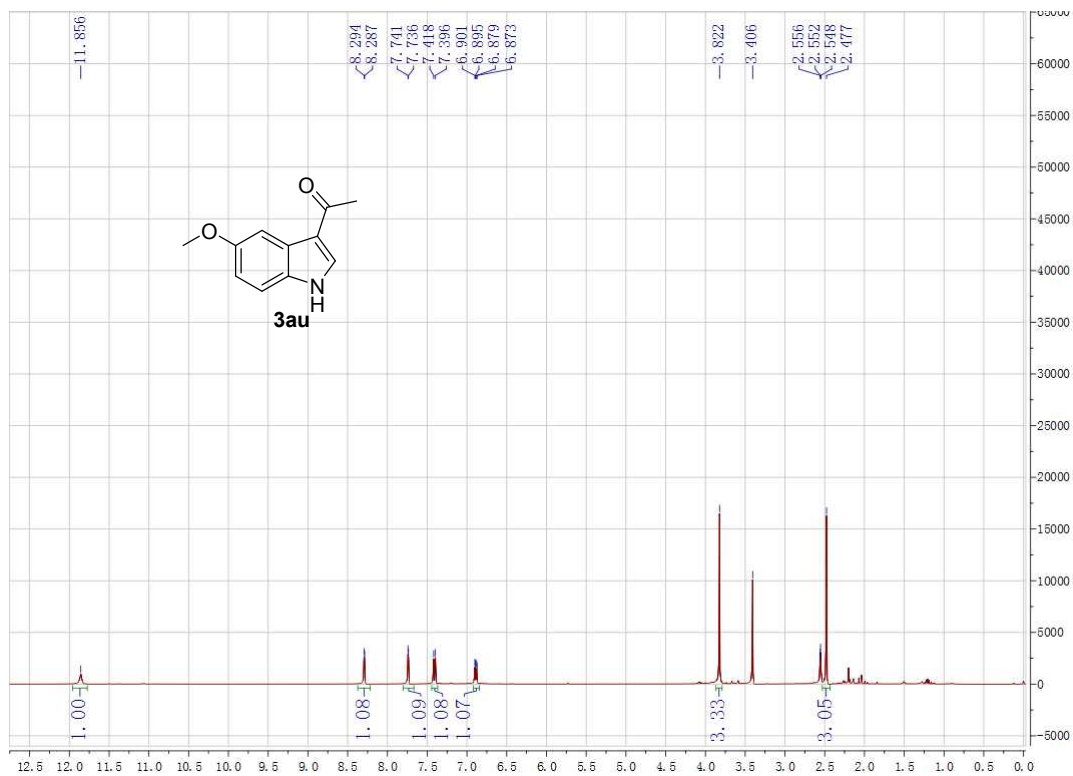


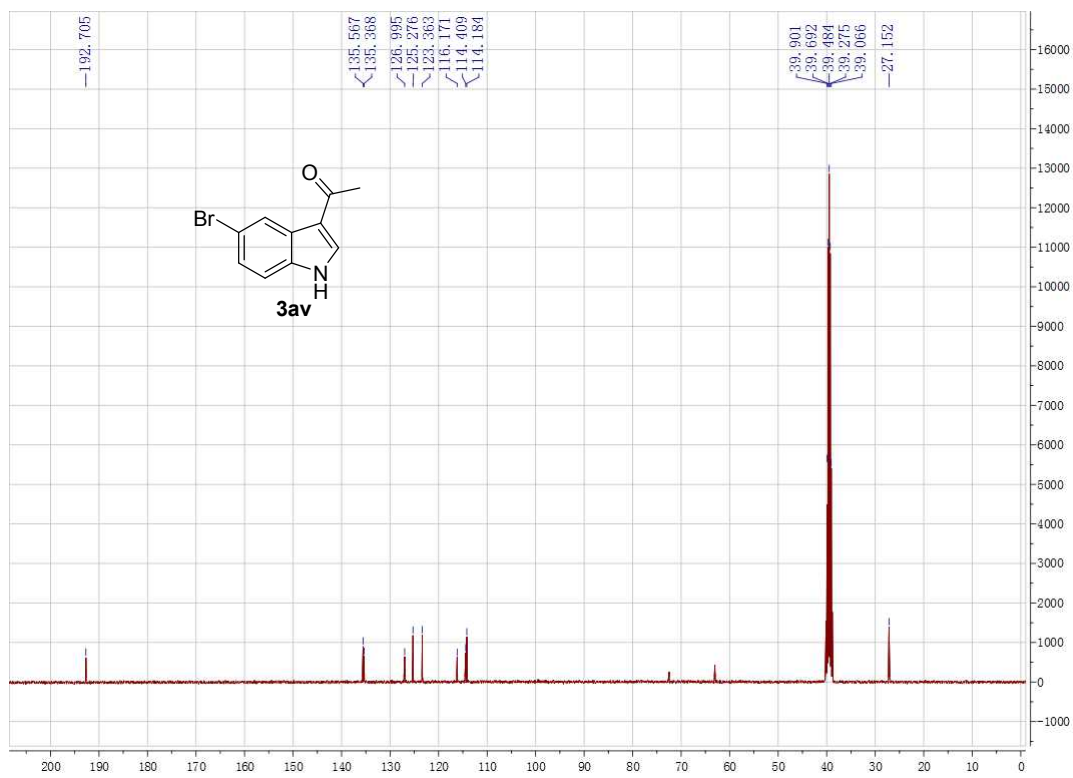
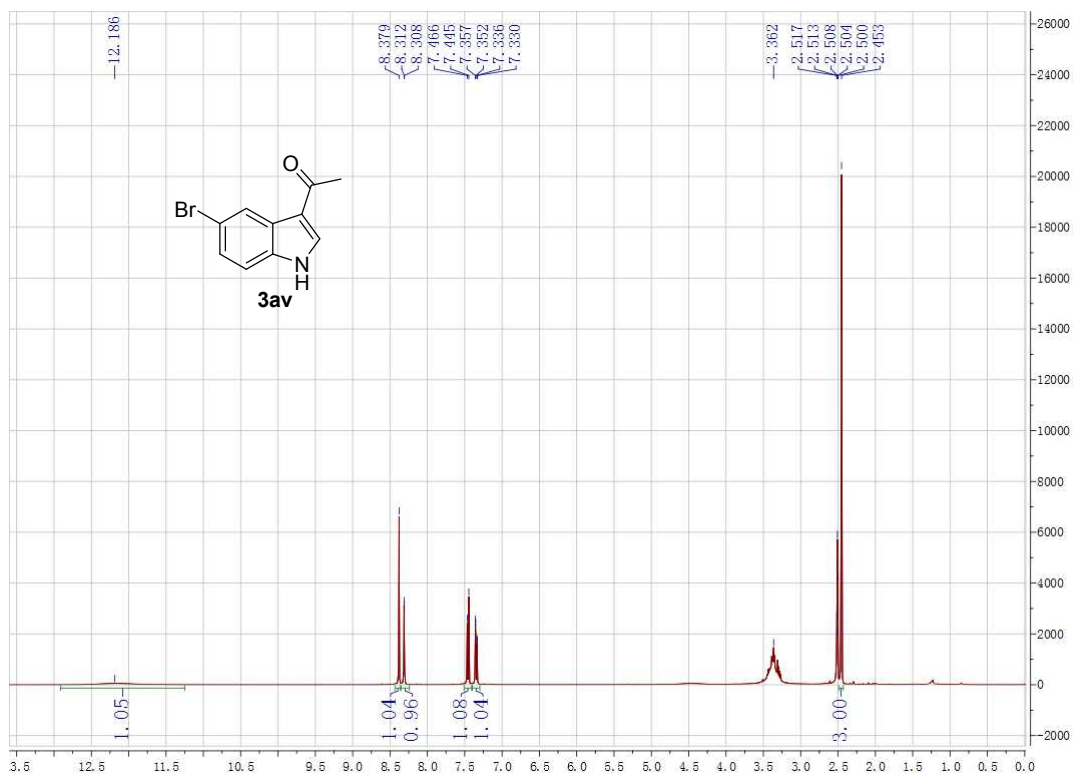


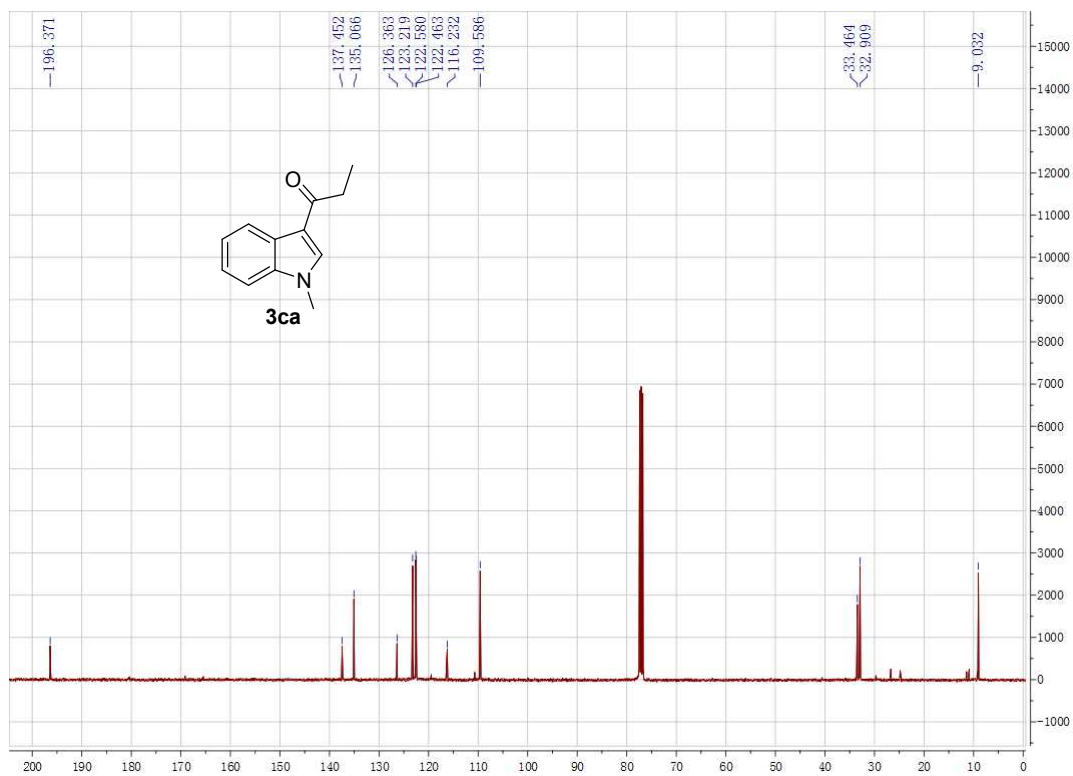
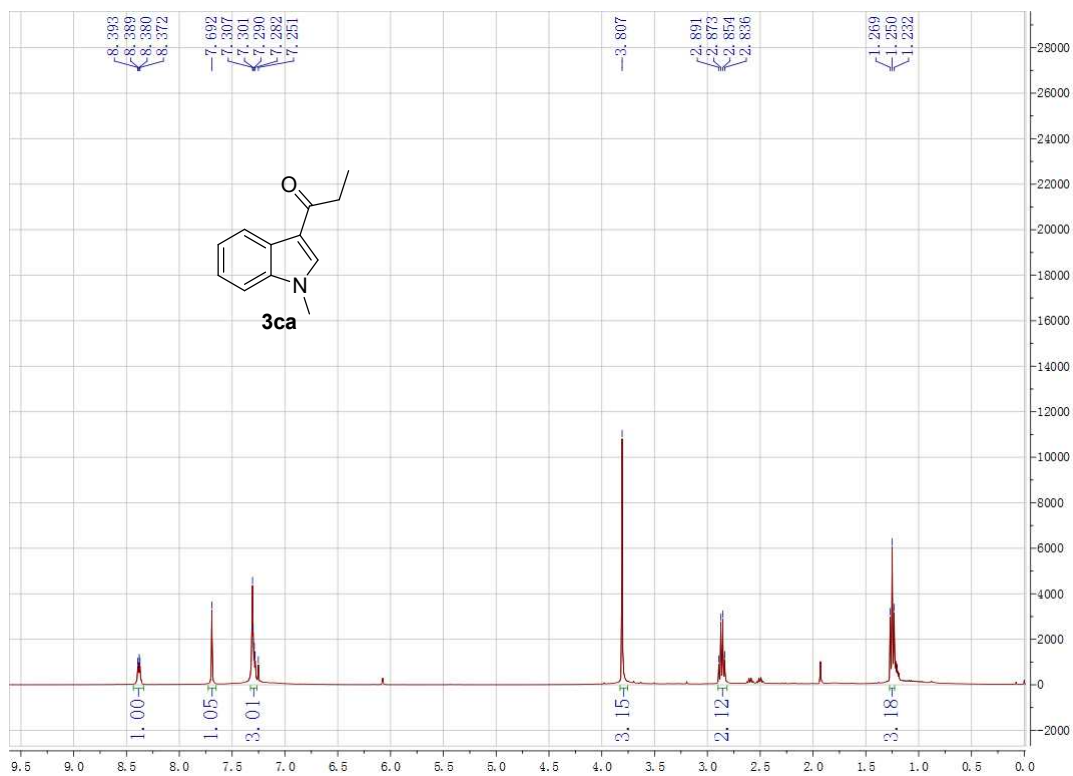


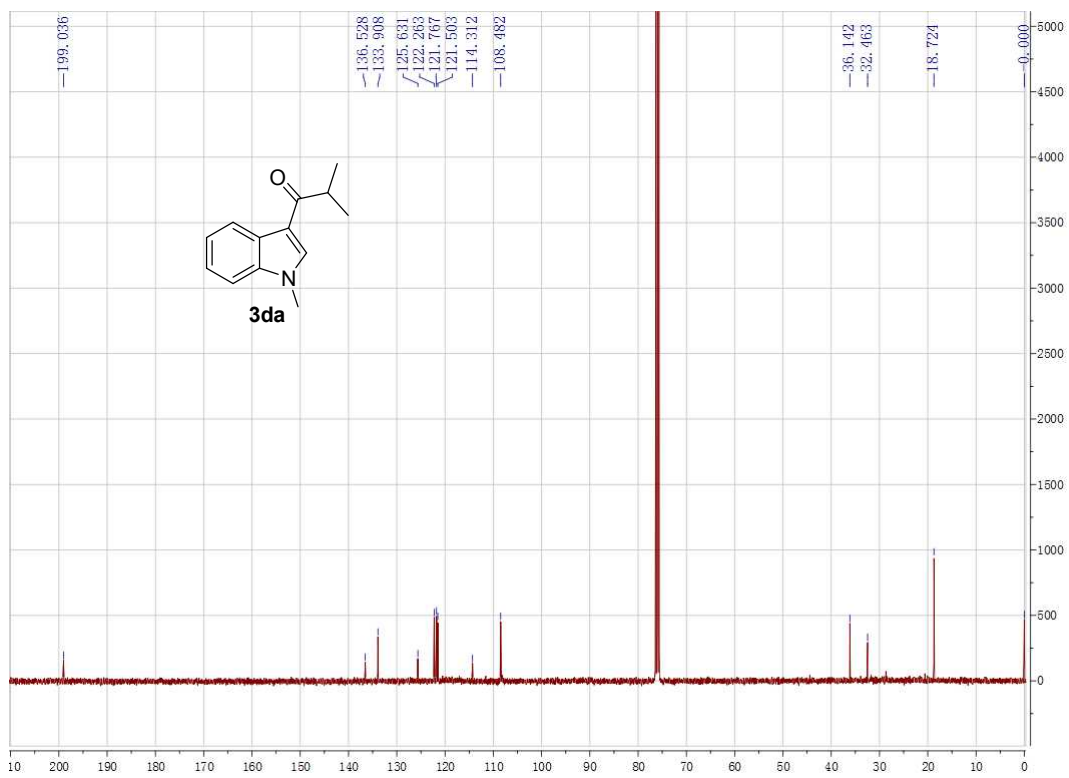
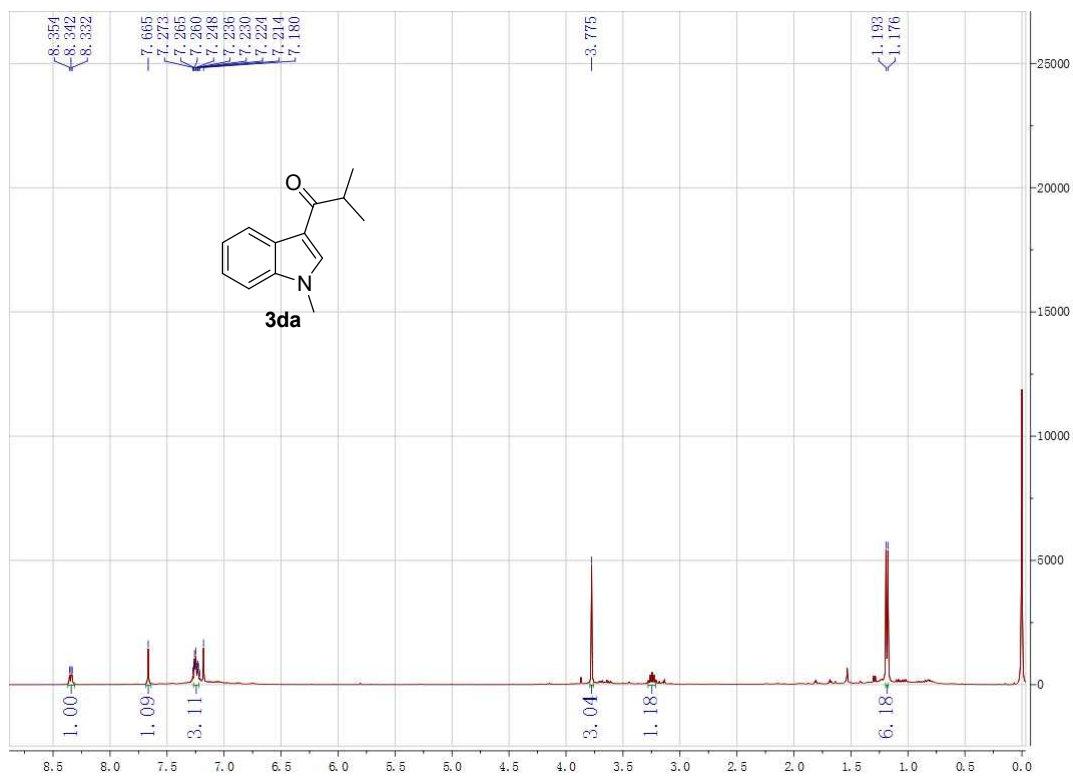


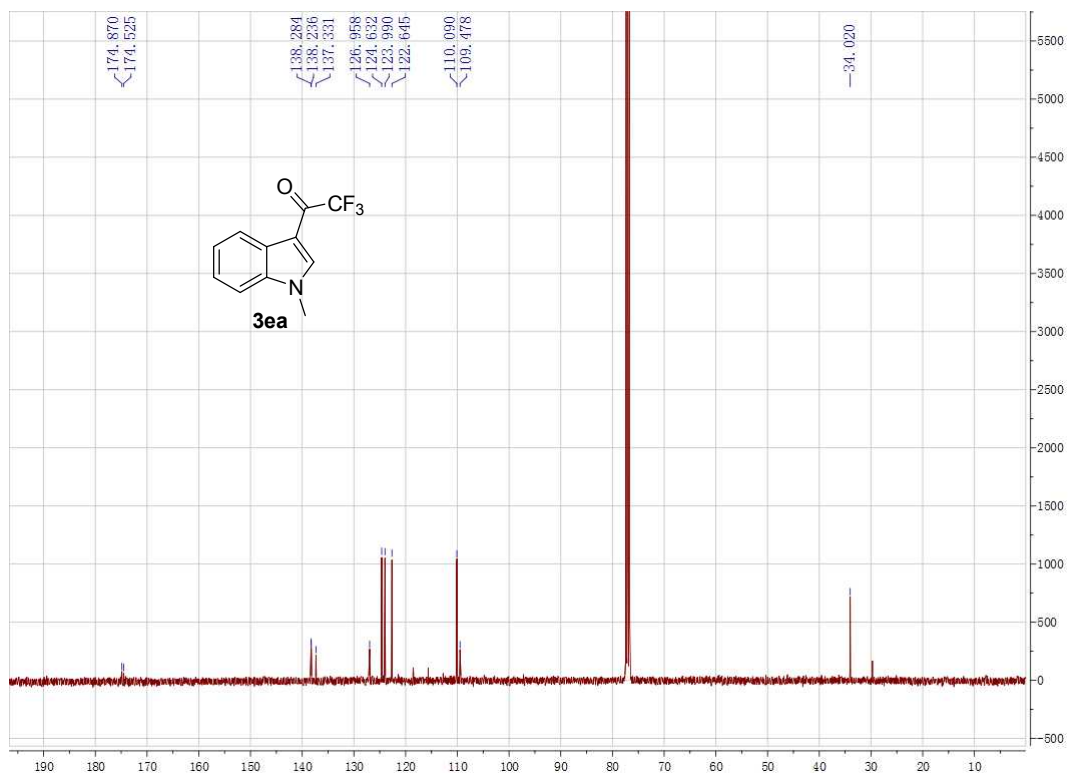
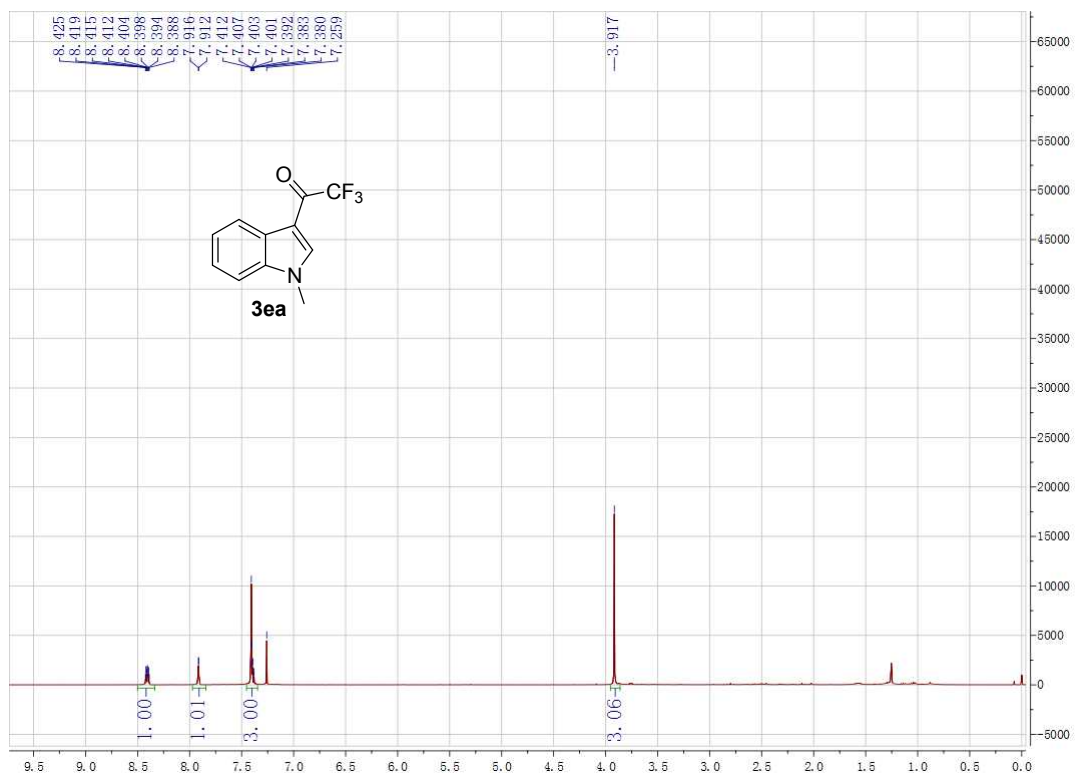


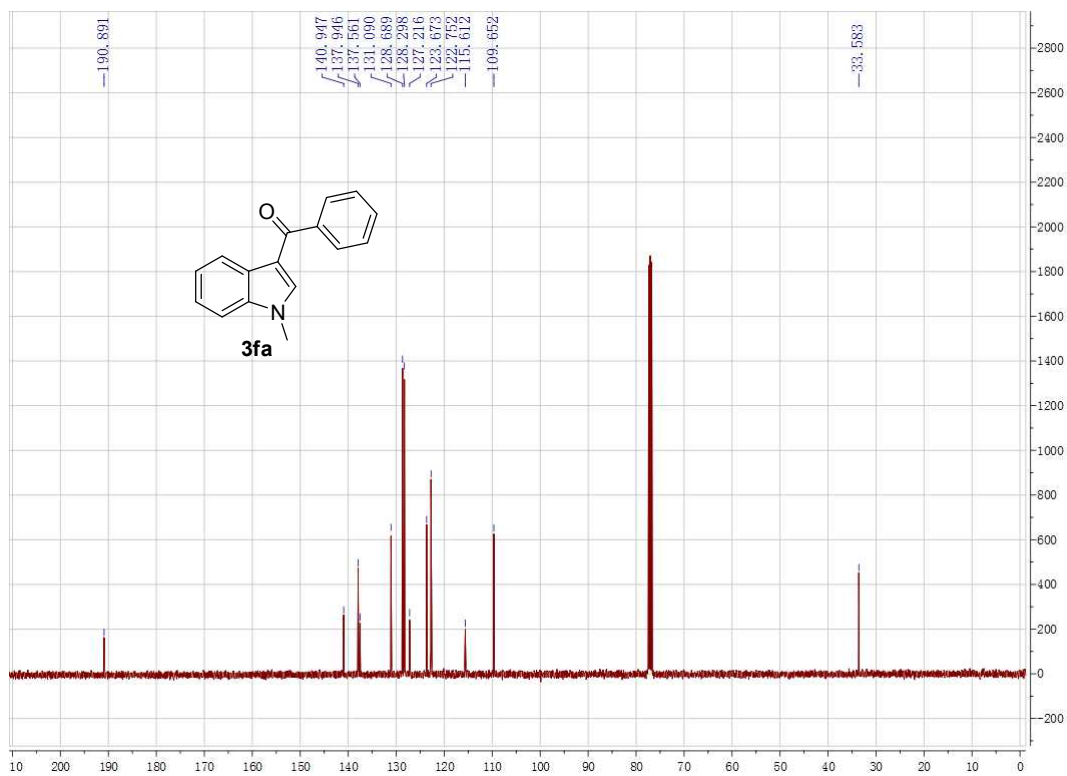
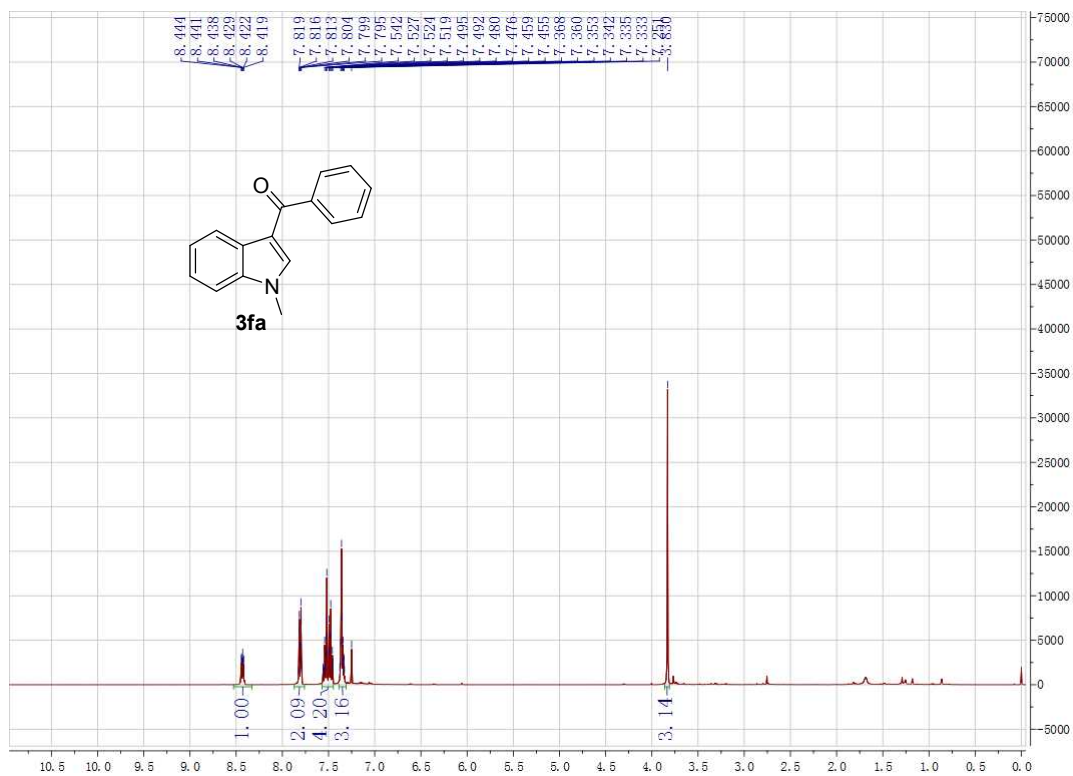


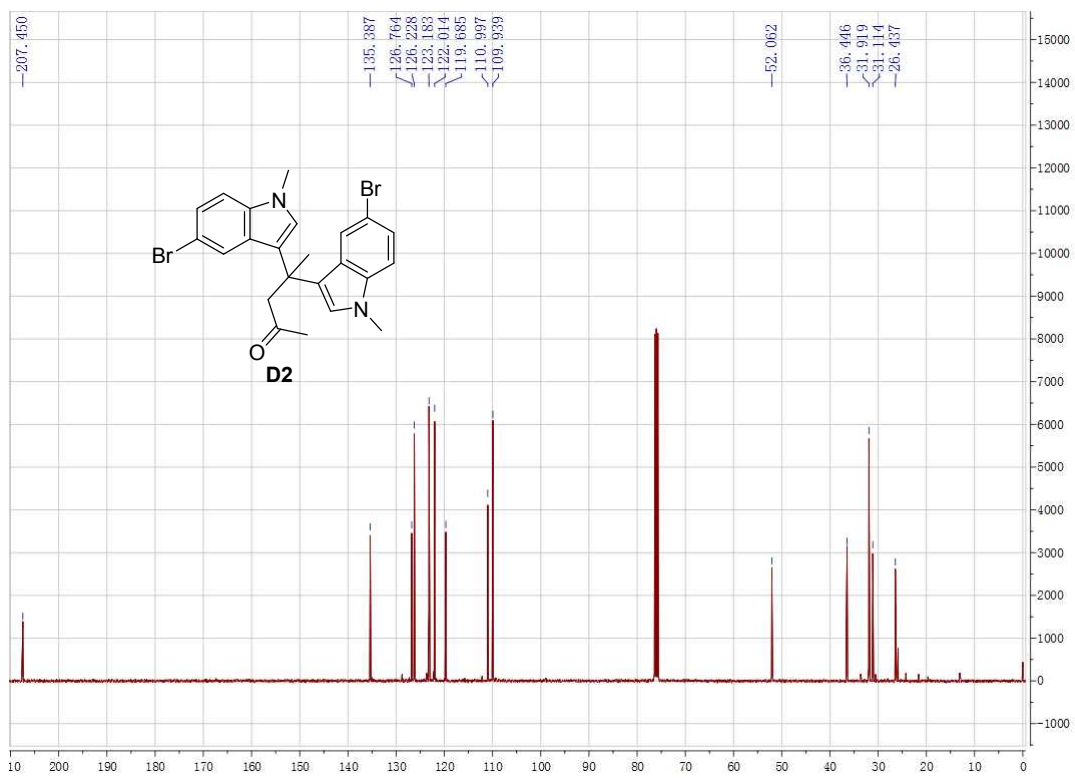
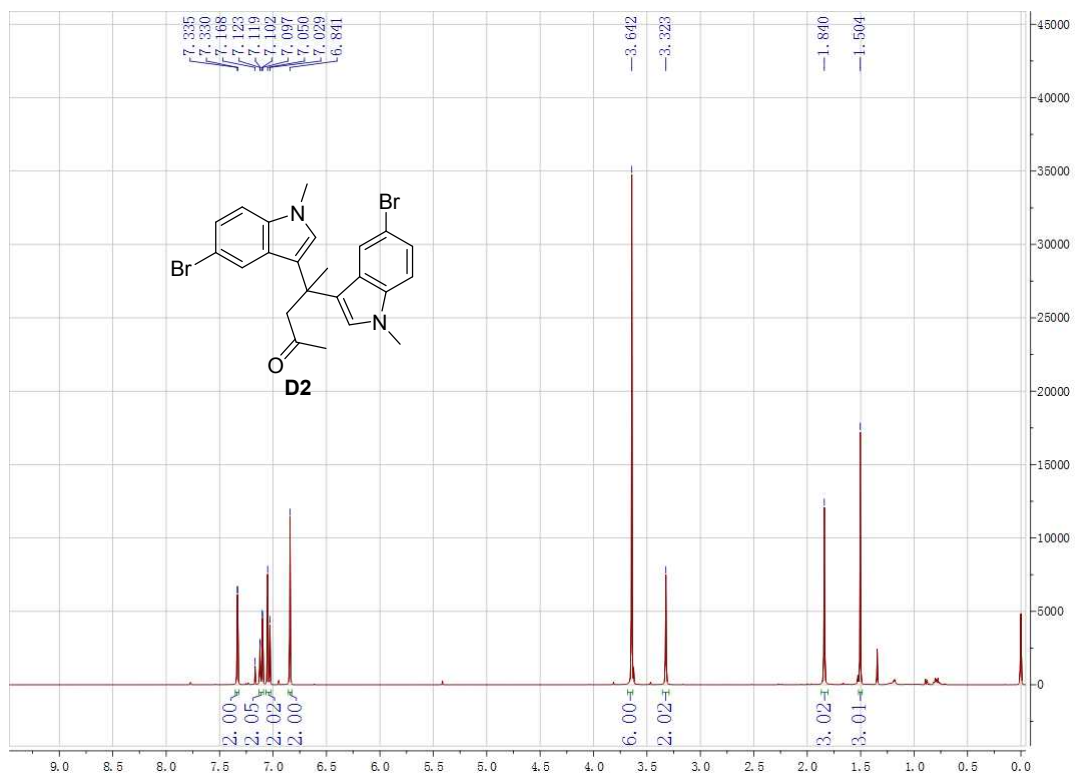




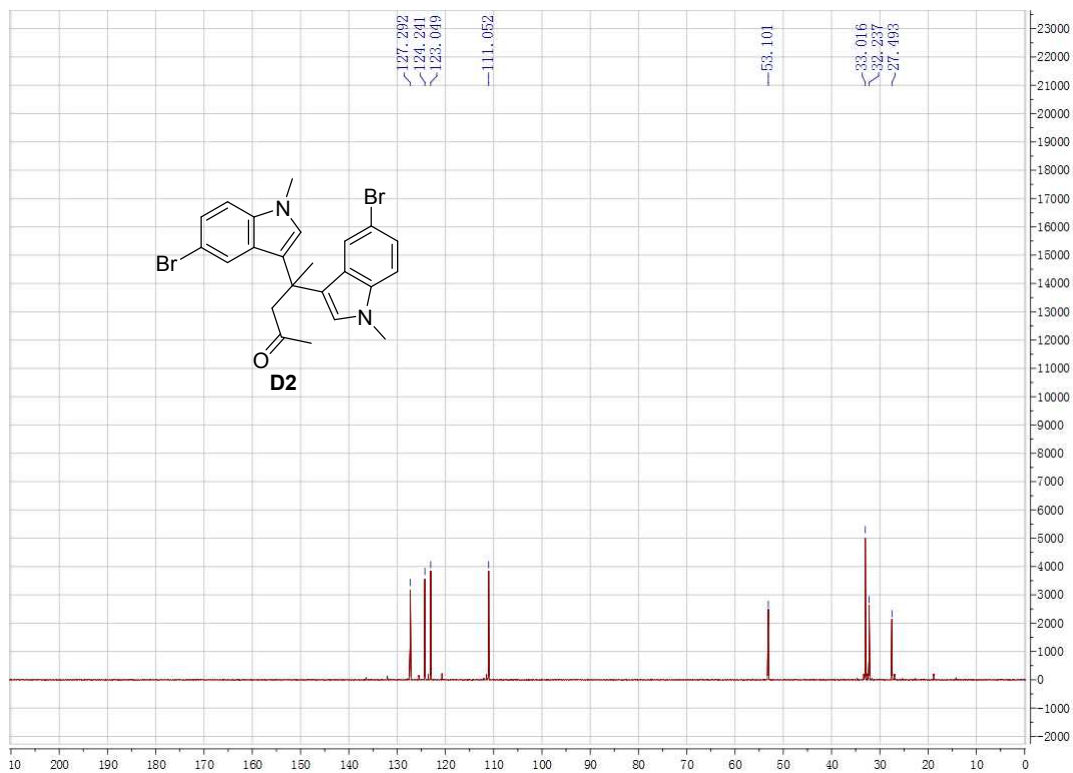




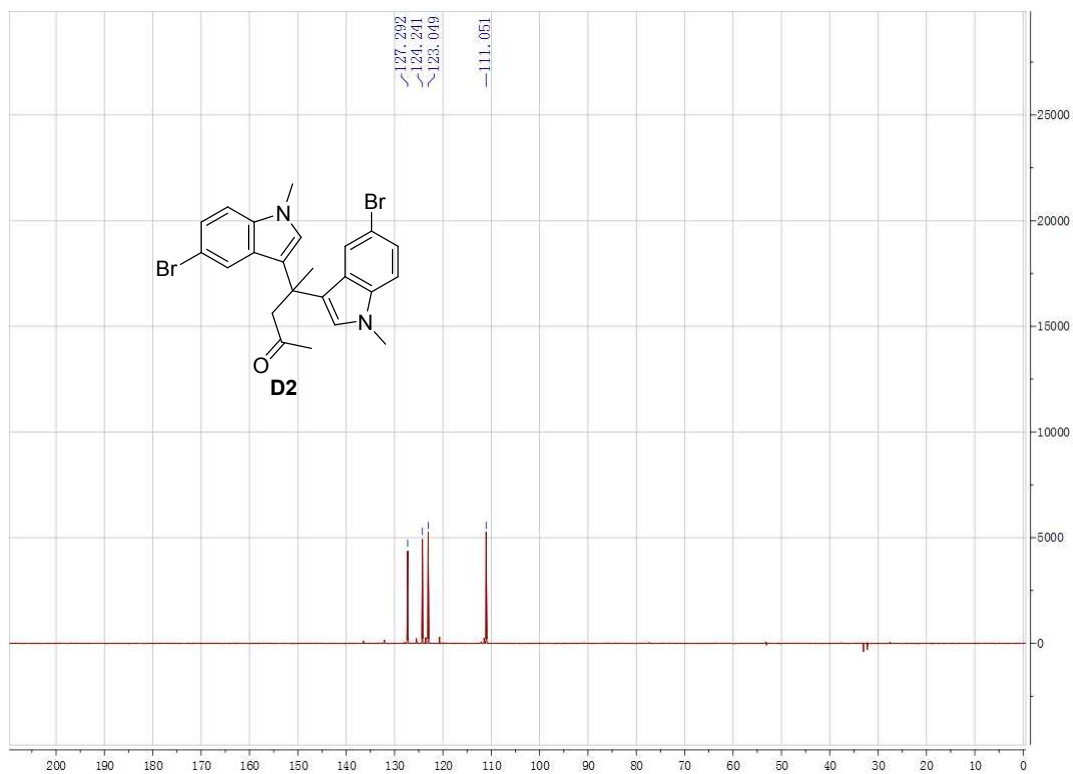




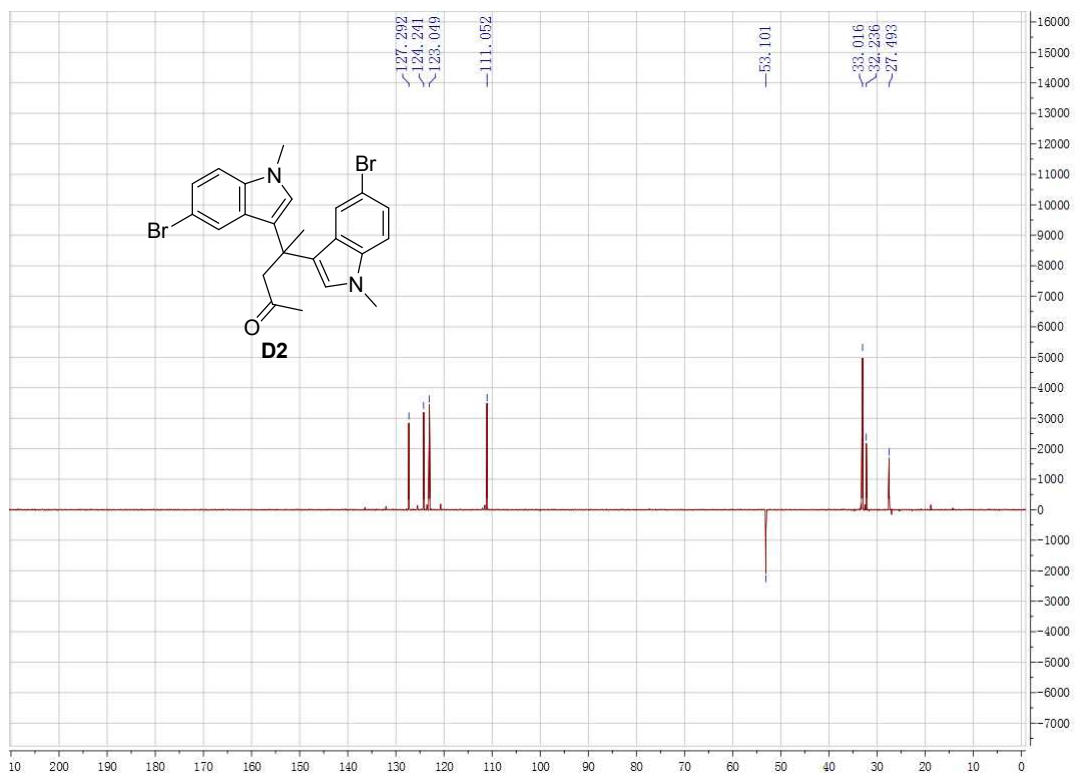
¹³C NMR DEPT45°C

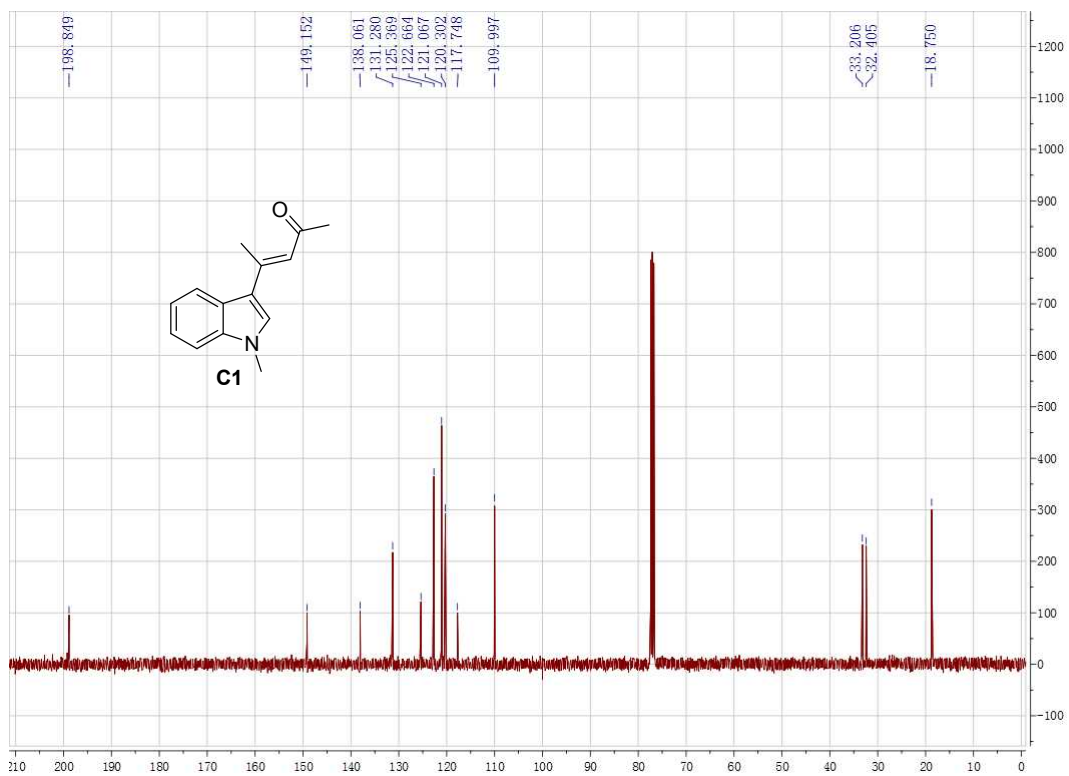
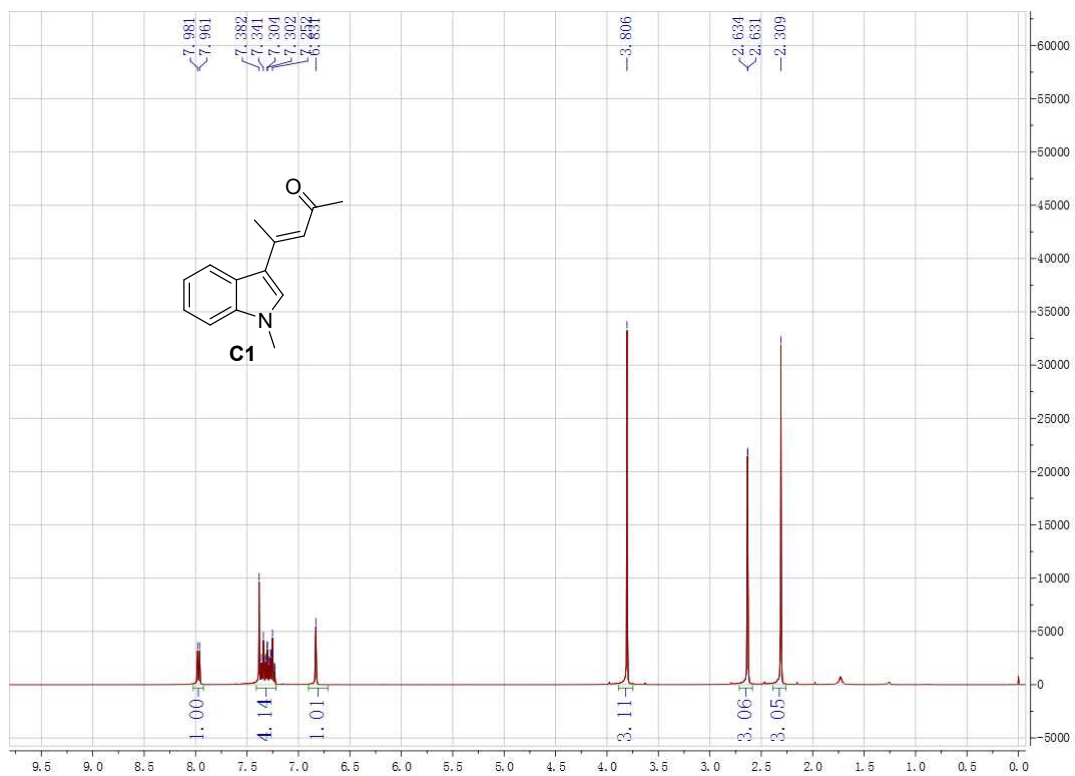


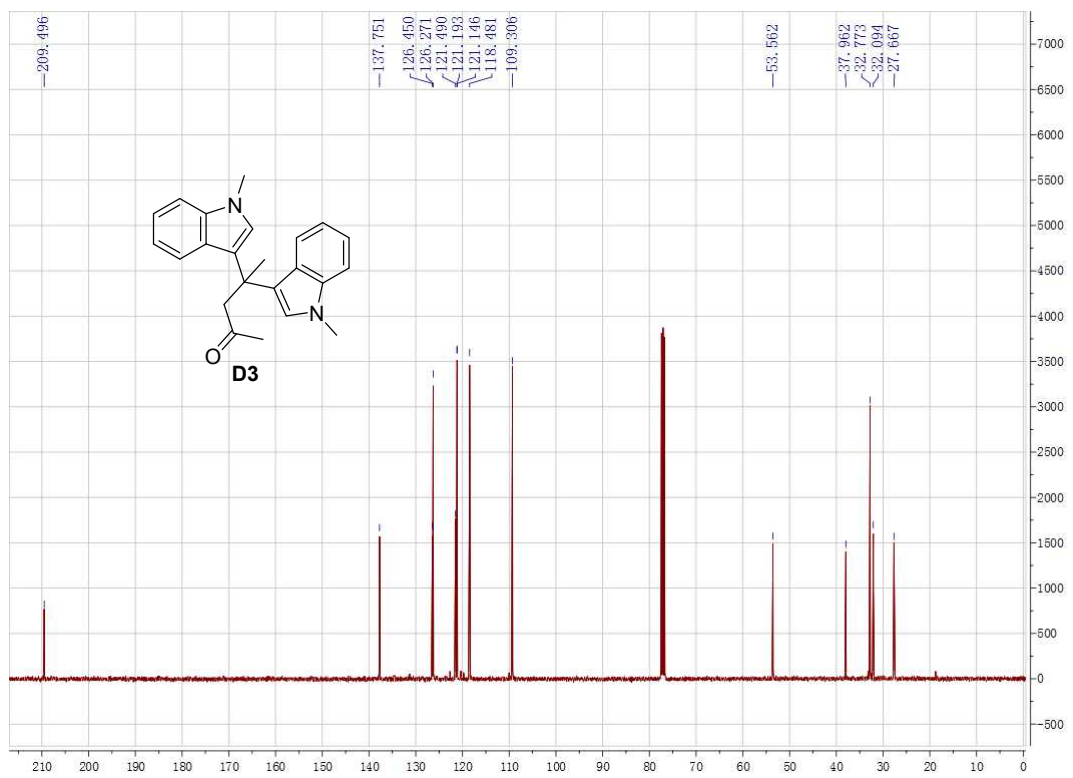
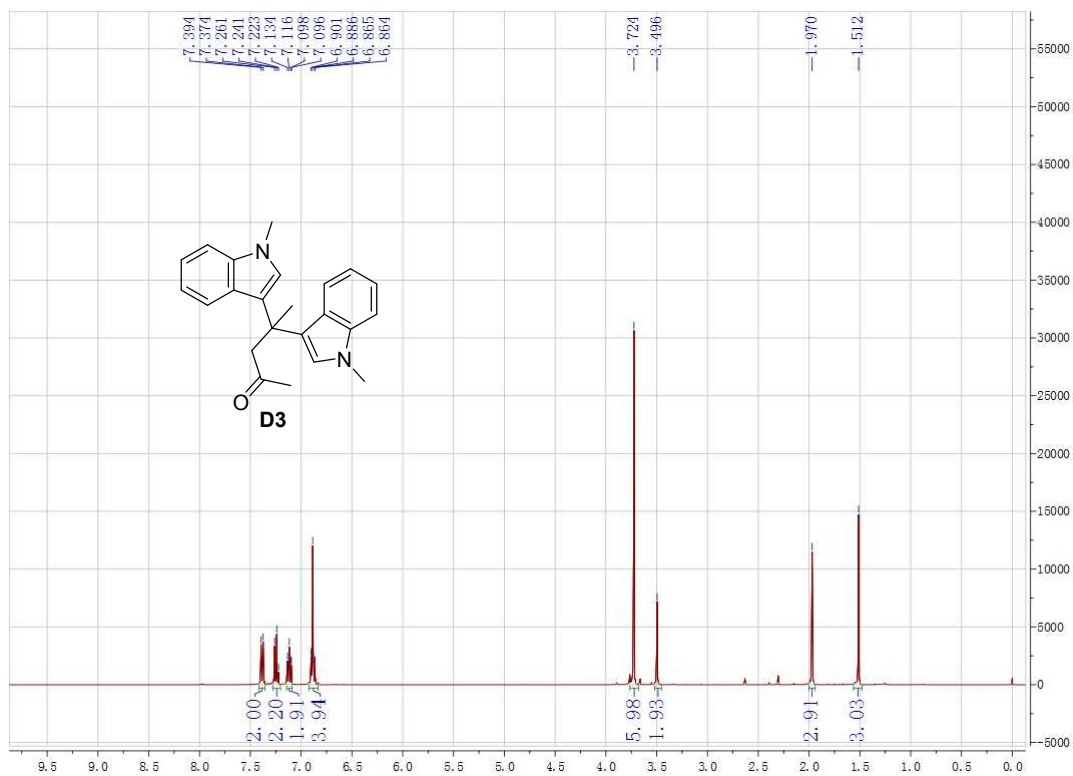
^{13}C NMR DEPT90°C

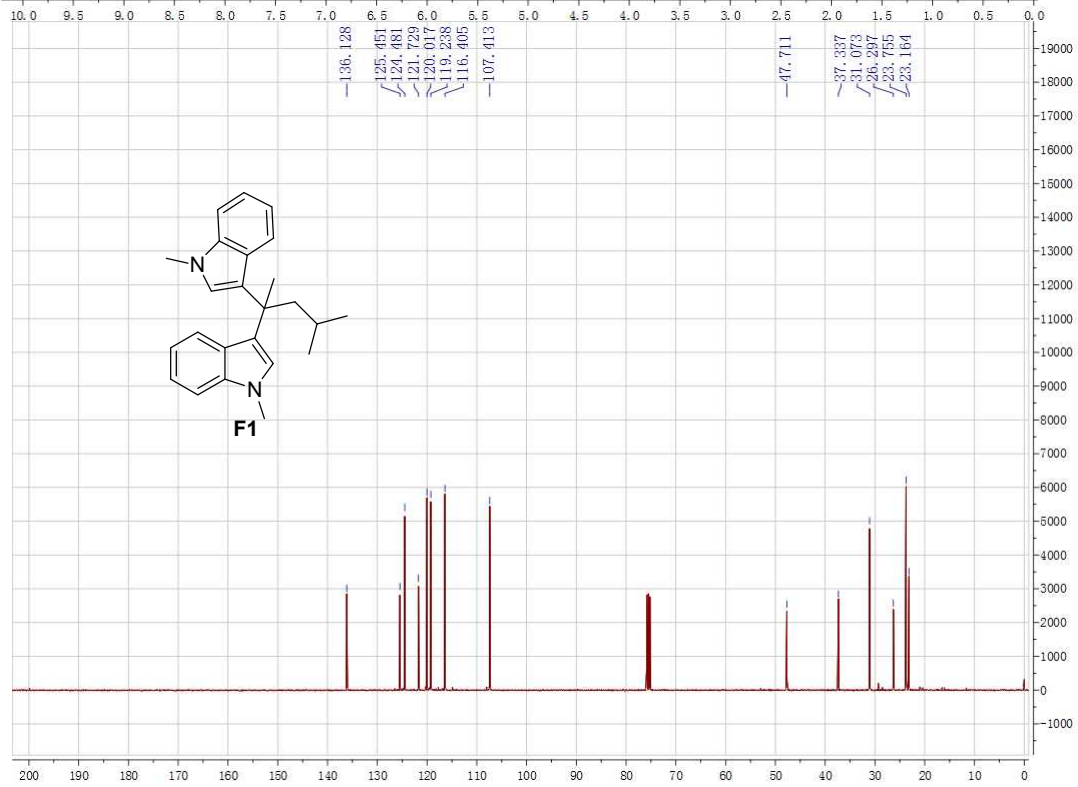
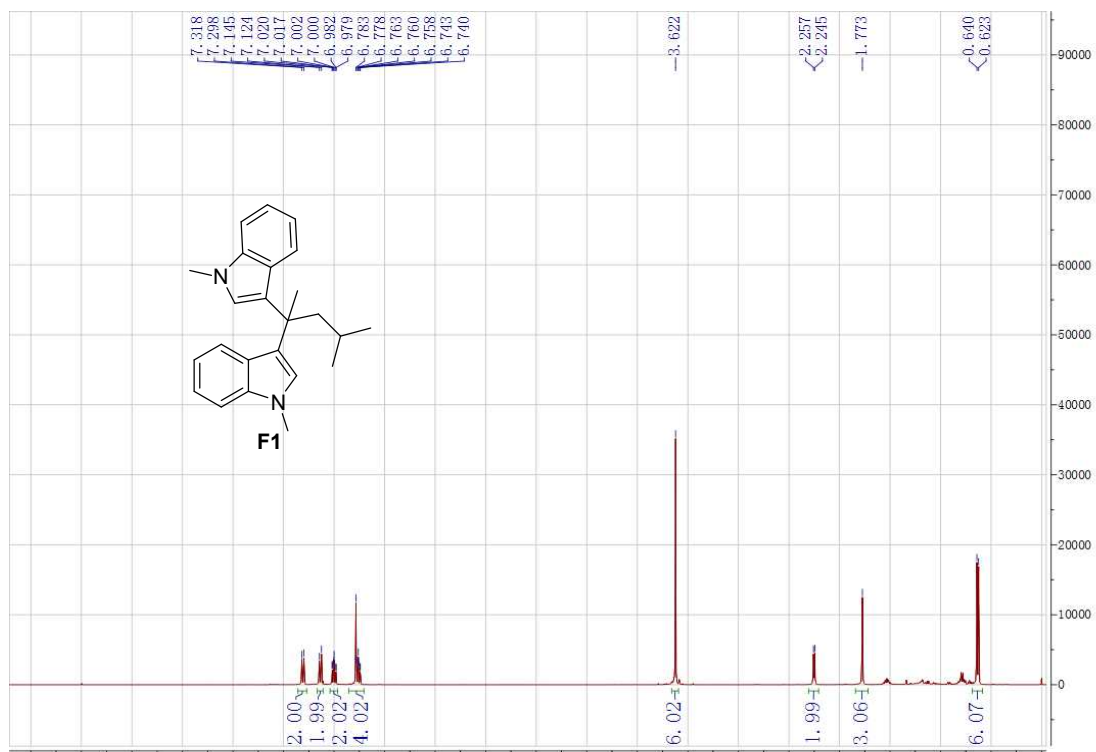


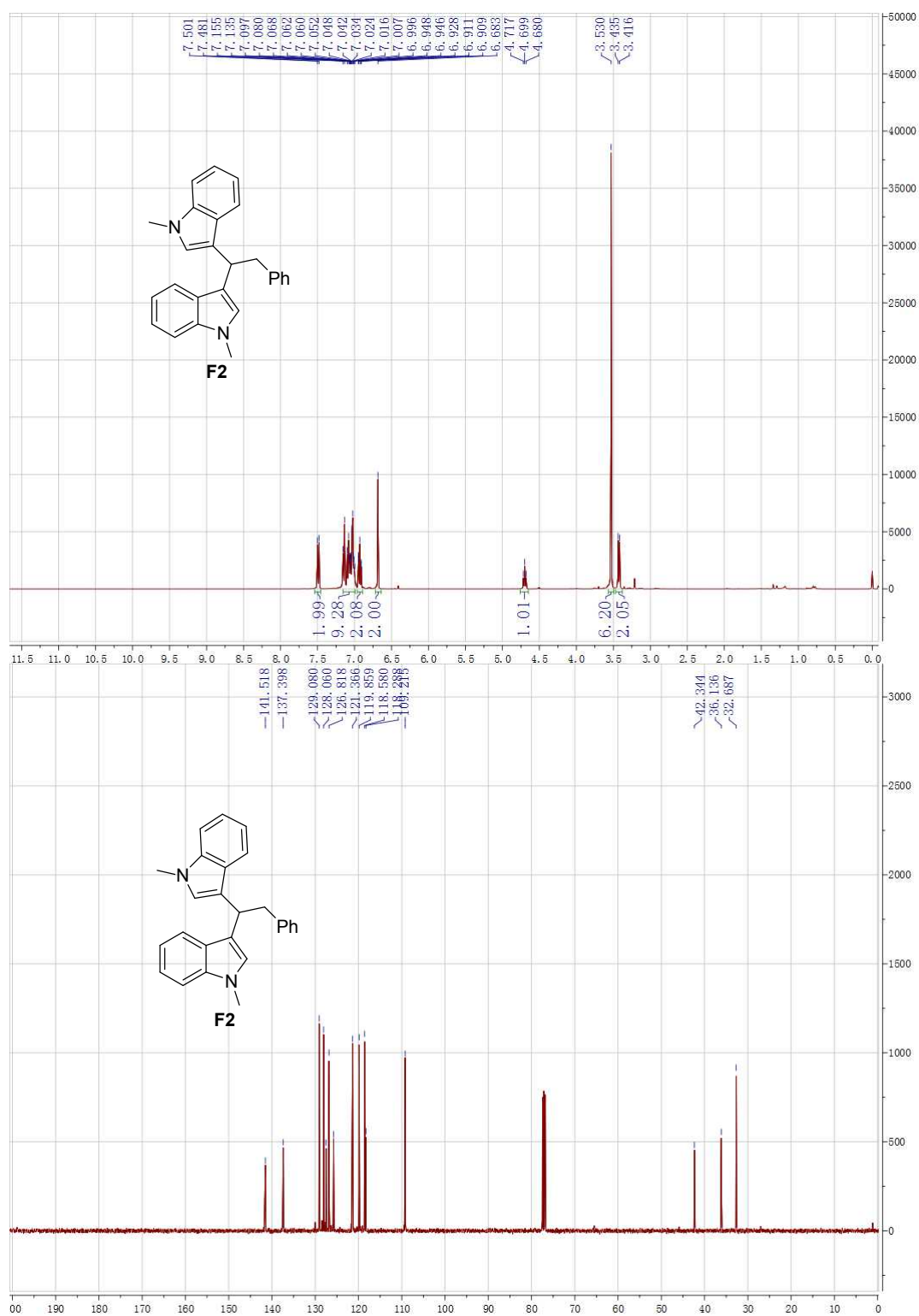
^{13}C NMR DEPT135°C











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