

Supporting Information

Preactivation-free direct decarboxylative alkylation of indoleacetic acids: access to alkynyl-functionalized indoles

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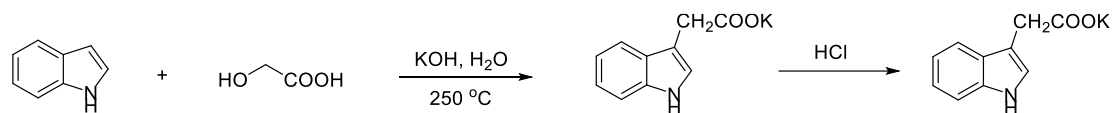
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1. General Information

^1H and ^{13}C NMR were recorded on a Biotage 400 spectrometer. ^1H NMR data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constant (Hz), relative intensity. ^{13}C NMR data are reported as follows: chemical shift in ppm (δ). HPLC-MS analyses were performed on a Shimadzu-2020 LC-MS instrument using the following conditions: Shim-pack VP-ODS C18 column (reverse phase, 50 x 4.6 mm); a linear gradient from 10% water and 90% acetonitrile to 95% acetonitrile and 5% water over 4.0 min; flow rate of 1 mL/min; UV photodiode array detection from 200 to 300 nm. The products were purified by Biotage IsoleraTM Spektra Systems and Petroleum Ether/EtOAc solvent systems. All reagents and solvents were obtained from commercial sources and used without further purification. Melting points (m.p.) were determined on a micro melting point apparatus (WRX-4) and were uncorrected. All indoleacetic acid derivatives used in this work were commercially available and used as received without further purification. For reference, a representative synthetic procedure for indole-3-acetic acid is provided below (adapted from *Org. Synth.* **1964**, *44*, 64; DOI: 10.15227/orgsyn.044.0064):

Procedure:



A 3-l. stainless steel, rocking autoclave (Note 1) is charged with 270 g. (4.1 moles) of 85% potassium hydroxide and 351 g. (3.00 moles) of indole (Note 2), and then 360 g. (3.3 moles) of 70% aqueous glycolic acid is added gradually (Note 3). The autoclave is closed and rocked at 250° for about 18 hours (Note 4). The reaction mixture is cooled to below 50°, 500 ml. of water is added, and the autoclave is rocked at 100° for 30 minutes to dissolve the potassium indole-3-acetate. The aqueous solution is cooled to 25° and removed from the autoclave, the autoclave is rinsed out well with water, and water is added until the total volume of solution is 3 l. The solution is extracted with 500 ml. of ether (Note 5). The aqueous phase is acidified at 20–30° with 12*N* hydrochloric acid and then is cooled to 10° (Note 6). The indole-3-acetic acid that precipitates is collected on a Büchner funnel, washed with copious amounts of cold water, and dried in air or a vacuum desiccator out of direct light (Note 7); weight 455–490 g. (87–93%); m.p. 163–165° (dec.).

The indole-3-acetic acid, which is cream-colored, is of high purity. If further purification is desired, it may be done conveniently by recrystallization from water. One liter of water is used for 30 g. of acid, with 10 g. of decolorizing carbon added. Recovery is about 22 g. of a nearly colorless product, m.p. 164–166° (dec.).

Notes:

1. A stirred autoclave is just as satisfactory. The scale is not critical, for the checkers got equally good results on one-third the scale; they used a 1-l. rocking autoclave.
2. Indole from the Union Carbide Olefins Company, Institute, West Virginia, is satisfactory.
3. If the reactants are added in this order, with the glycolic acid being introduced over a 5–10 minute period, there is no violent heating because the heat of neutralization is used to melt the indole. An equivalent amount of anhydrous glycolic acid may be used, but this offers no special advantage.
4. These limits are not critical, but they are probably optimum. Reaction times of 24–30 hours are not particularly detrimental, and high yields of product can be obtained within 12 hours. The temperature can range from 230° to 270° with but slight effect on the yield of product.

5. This extraction may be omitted. It does, however, remove traces of neutral material and consequently gives a product with greater color stability.
6. This operation is most conveniently conducted in a flask equipped with a stirrer.
7. The product dries slowly, and several days in air or 24 hours in a vacuum desiccator is usually required. Considerable coloration will result if this is done in direct light. Drying in a heated oven or removing the water as a benzene azeotrope is not satisfactory because of some decarboxylation to skatole. The product should be stored in a dark bottle away from direct sunlight.

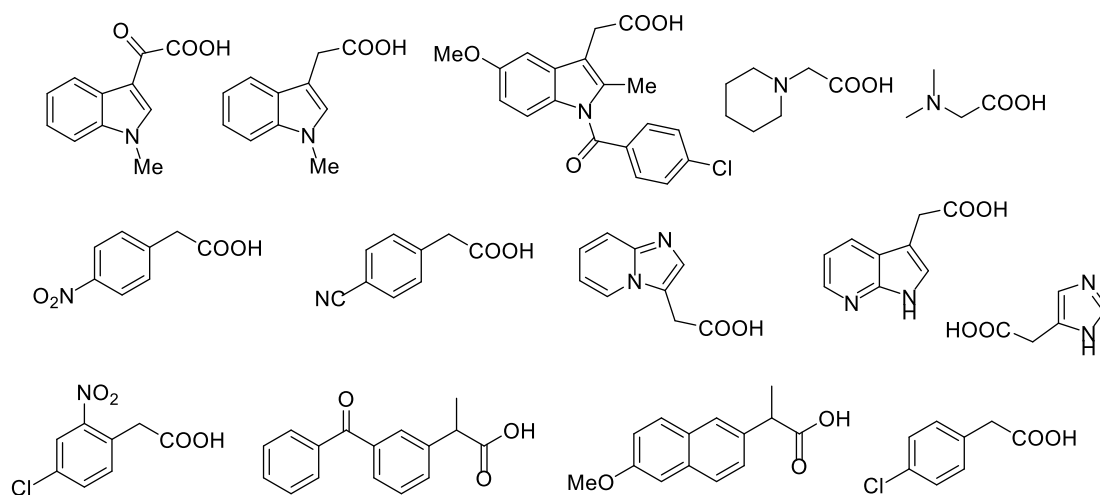
2. Microwave Irradiation Experiments

All microwave irradiation experiments were carried out in a Biotage® Initiator Classic microwave apparatus with continuous irradiation power from 0 to 400W with utilization of the standard absorbance level of 250W maximum power. The reactions were carried out in 10 mL glass tubes, sealed with microwave cavity. The reaction was irradiated at a required ceiling temperature using maximum power for the stipulated time. Then it was cooled to 50 °C with gas jet cooling.

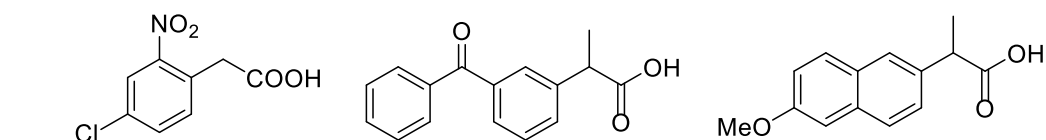


3. Unsuccessful substrates and comparison with conventional thermal heating conditions

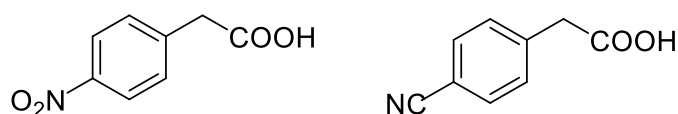
Several carboxylic acids failed to deliver the desired products under the optimized conditions.



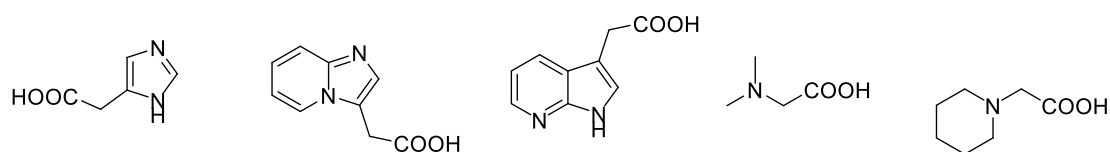
Steric hindrance likely accounts for the inactivity of ortho-substituted and bulky substrates, such as 2-nitro-5-chlorophenylacetic acid and 2-(3-benzoylphenyl)propanoic acid, which may impede coordination to copper or subsequent decarboxylation.



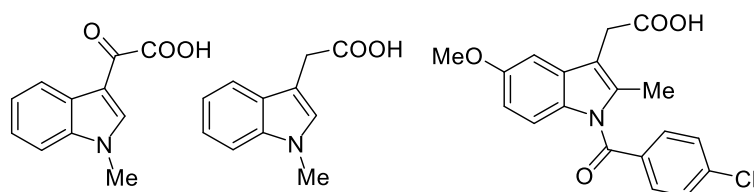
Strong electron-withdrawing groups (e.g., NO₂, CN) on the aryl ring destabilize the copper carboxylate intermediate and/or excessively stabilize the resulting radical, preventing productive coupling, as observed for p-nitrophenylacetic acid and p-cyanophenylacetic acid.



Heterocyclic and aliphatic carboxylic acids (e.g., imidazolylacetic acid, piperidinylacetic acid) showed poor reactivity, probably due to competing coordination of the nitrogen atoms to copper, catalyst deactivation, or inefficient decarboxylation of these skeletons.



Slight structural modifications of the indole core (e.g., N-methyl substitution or replacement of the 3-acetic acid moiety with a 3-glyoxylic acid (α -keto acid) group) also abolished reactivity, indicating that the indol-3-ylacetic acid scaffold is critical for efficient decarboxylation and subsequent coupling.



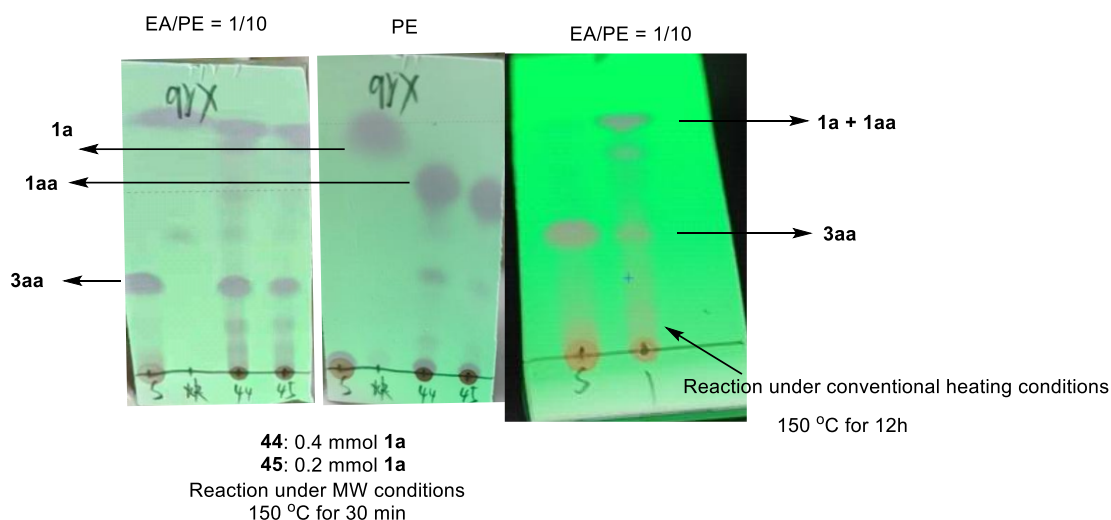
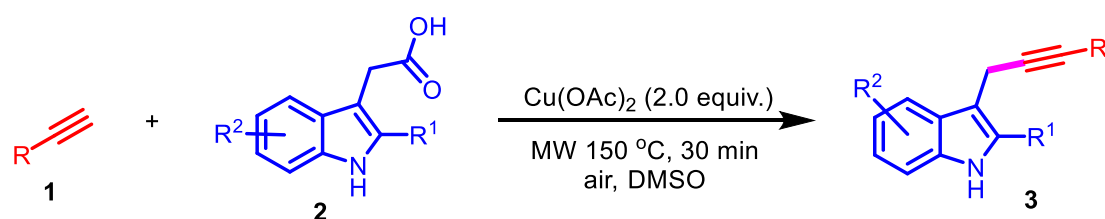


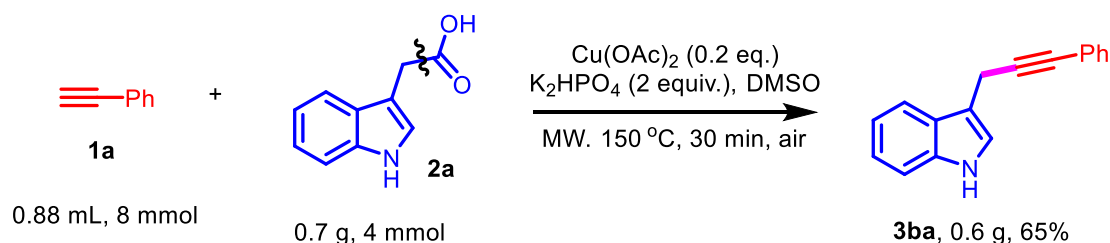
Figure S1. TLC analysis comparing the reaction under microwave irradiation (150 °C, 30 min) and conventional thermal heating (150 °C, 12 h). The desired product 3aa is formed in significantly higher yield under microwave conditions, while only trace product is observed under conventional heating.

4. General Procedure for Synthesis of Indolyated Alkynes



In an oven-dried glass tubes alkynes **1** (0.4 mmol, 2.0 eq.), 3-indoleacetic acids **2** (0.2 mmol, 1.0 eq.), and Cu(OAc)_2 (80 mg, 0.4 mmol, 2.0 eq.) were dissolved in DMSO (2 mL) and the reaction mixture was stirred at **MW** 150 °C for 30 min by microwave irradiation and monitored by TLC. Then the reaction mixture was extracted using EtOAc/ H_2O and concentrated under reduced pressure followed by column chromatography over silica gel using petroleum/EtOAc = 10/1 to 5/1 as eluent to afford the desired product **3**.

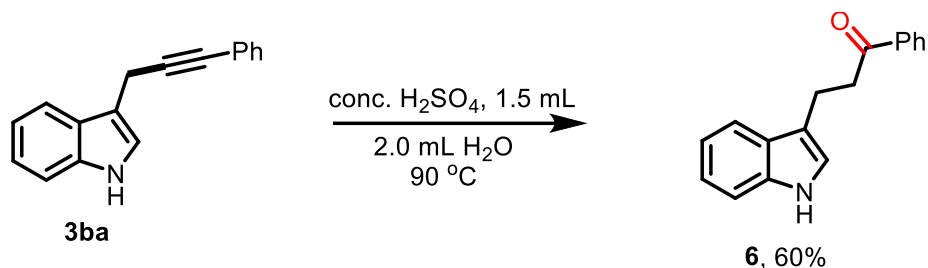
5. Gram Scale-up Experiment



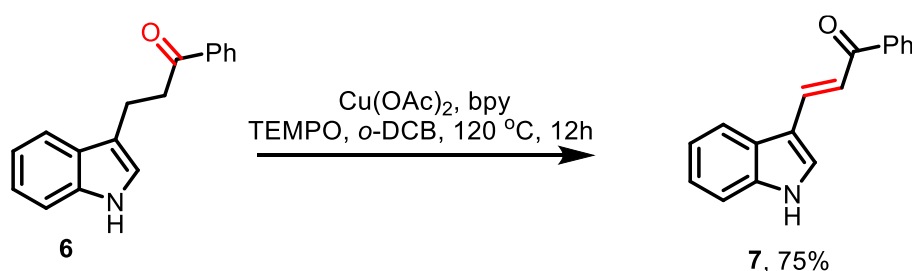
In an oven-dried glass tubes alkyne **1a** (0.88 mL, 8 mmol, 2.0 eq.), 3-indoleacetic acid **2a** (0.7 g, 4 mmol, 1.0 eq.), and Cu(OAc)_2 (1.6 g, 8 mmol, 2.0 eq.) were dissolved in DMSO (20 mL) and the reaction mixture was stirred at **MW** 150 °C for 30 min by microwave irradiation and monitored by

TLC. Then the reaction mixture was extracted using EtOAc/H₂O and concentrated under reduced pressure followed by column chromatography over silica gel using petroleum/EtOAc = 10/1 as eluent to afford the desired product **3ba** (0.6 g, 65%).

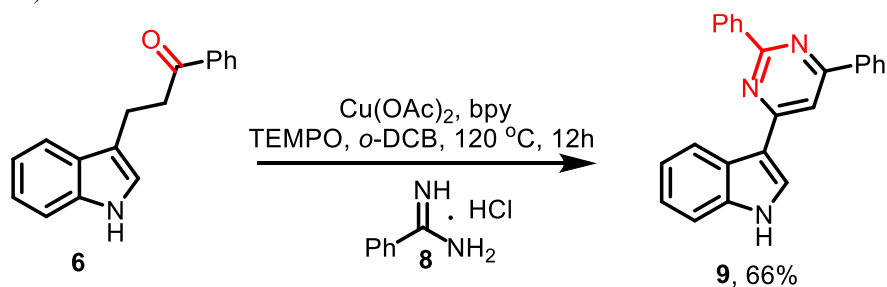
6. Synthetic applications of **3ba**



Compound **6** was prepared according to the literature.¹ In an oven-dried glass tubes alkyne **3ba** (185 mg, 0.8 mmol, 1.0 eq.) and conc. H₂SO₄ (1.5 mL) were dissolved in H₂O (2 mL) and the reaction mixture was stirred at 90 °C for 24 h. Then the reaction mixture was extracted using EtOAc/Saturated aqueous sodium bicarbonate and concentrated under reduced pressure followed by column chromatography over silica gel using petroleum/EtOAc = 10/1 as eluent to afford the desired product **6** (120 mg, 60%).



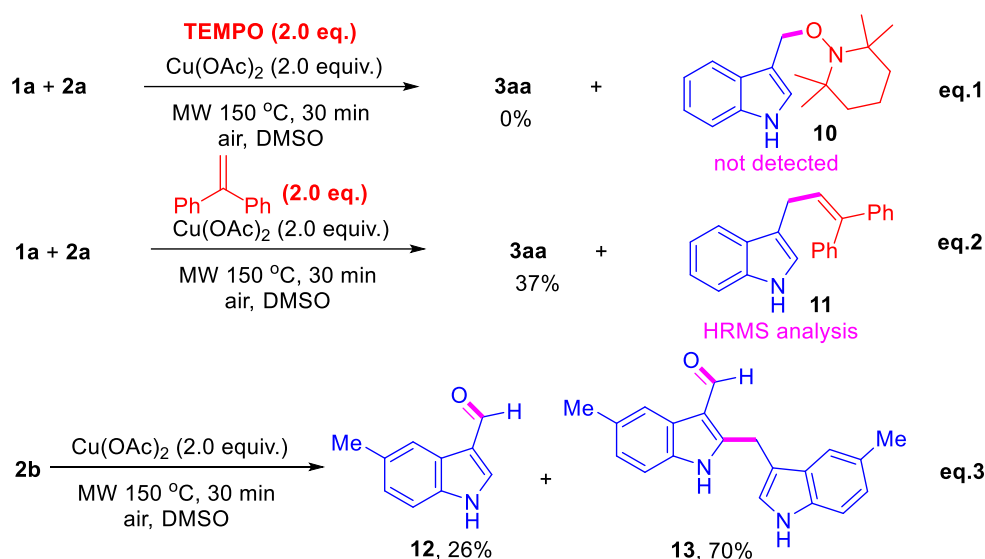
Compound **7** was prepared according to the literature.² To a solution of ketone **6** (50 mg, 0.2 mmol, 1.0 eq.) and TEMPO (156 mg, 1.0 mmol, 1.0 eq.) in dry 1,2 dichlorobenzene (2.0 mL) was added Cu(OAc)₂ (8 mg, 0.04 mmol, 20 mol%) and bpy (13 mg, 0.08 mmol, 40 mol%) at room temperature. The temperature of oil bath was increased up to 120 °C and mixture was stirred at same temperature for another 12 hrs. The reaction mixture was then allowed to attain room temperature and diluted with ethyl acetate (3 mL). The organic layer was washed with water (3 mL), sodium bicarbonate (3 mL) and brine (3 mL). The organic layer was further dried over anhydrous sodium sulphate and solvent was evaporated under reduced pressure to get crude product which was purified by column chromatography over silica gel using petroleum/EtOAc = 10/1 as eluent to afford the desired product **7** (37 mg, 75%).



Compound **9** was prepared according to the literature.³ To a solution of ketone **6** (50 mg, 0.2 mmol,

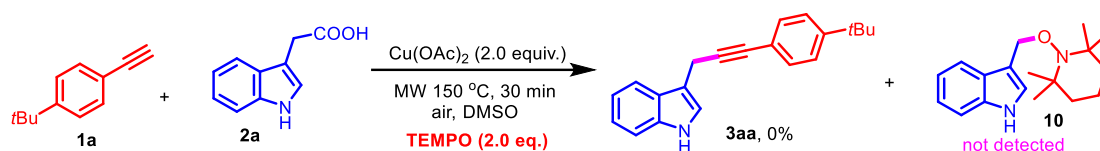
1.0 eq.), benzimidamide hydrochloride **8** (47 mg, 0.3 mmol, 1.5 eq.) and TEMPO (156 mg, 1.0 mmol, 1.0 eq.) in dry 1,2 dichlorobenzene (2.0 mL) was added Cu(OAc)₂ (8 mg, 0.04 mmol, 20 mol%) and bpy (13 mg, 0.08 mmol, 40 mol%) at room temperature. The temperature of oil bath was increased up to 120 °C and mixture was stirred at same temperature for another 12 hrs. The reaction mixture was then allowed to attain room temperature and diluted with ethyl acetate (3 mL). The organic layer was washed with water (3 mL), sodium bicarbonate (3 mL) and brine (3 mL). The organic layer was further dried over anhydrous sodium sulphate and solvent was evaporated under reduced pressure to get crude product which was purified by column chromatography over silica gel using petroleum/EtOAc = 10/1 as eluent to afford the desired product **9** (46 mg, 66%).

7. Experimental Studies on the Reaction Mechanism



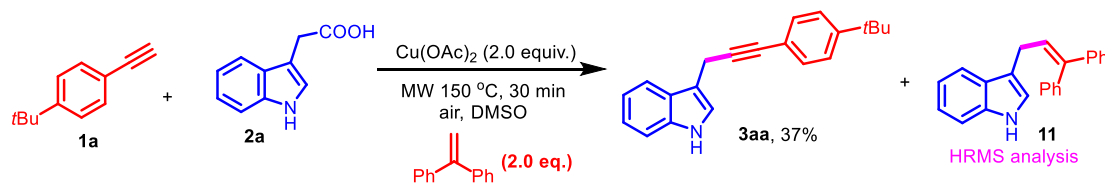
Scheme S1 Mechanism studies.

7.1 Control experiments



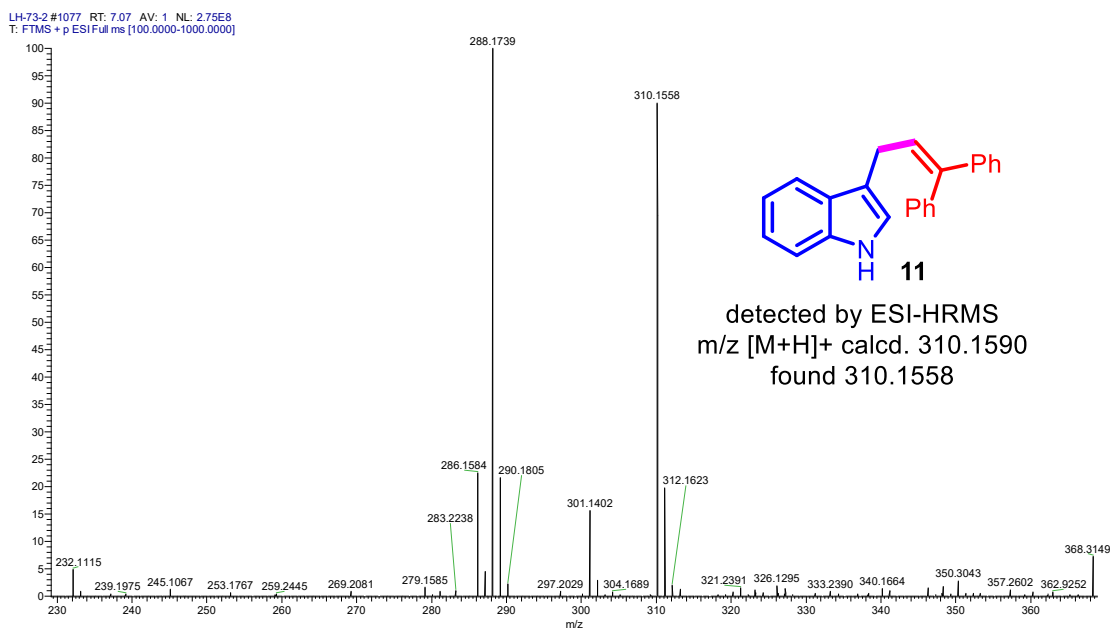
Scheme S2

In an oven-dried glass tubes alkyne **1a** (70 μ L, 0.4 mmol, 2.0 eq.), 3-indoleacetic acid **2a** (35 mg, 0.2 mmol, 1.0 eq.), TEMPO (63 mg, 0.4 mmol, 2.0 eq.), and Cu(OAc)₂ (80 mg, 0.4 mmol, 2.0 equiv.) were dissolved in DMSO (2 mL) and the reaction mixture was stirred at MW 150 °C for 30 min. After which, the target product **3aa** was not detected by TLC. Then, the ESI-HRMS analysis of the crude reaction mixture was carried out. The mass for indole-3-methyl radical trapping product **10** could not be detected.

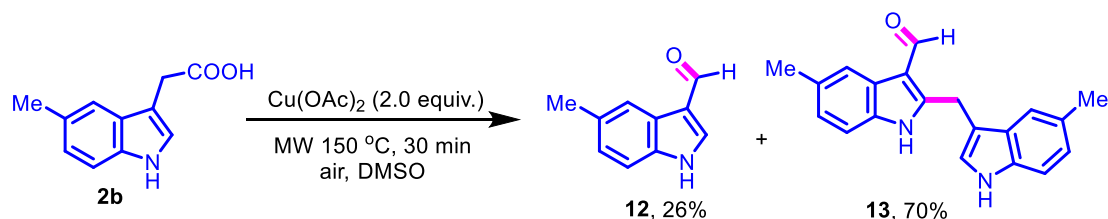


Scheme S3

In an oven-dried glass tubes alkyne **1a** (70 μ L, 0.4 mmol, 2.0 eq.), 3-indoleacetic acid **2a** (35 mg, 0.2 mmol, 1.0 eq.), 1,1-diphenylethylene (71 μ L, 0.4 mmol, 2 eq.), and Cu(OAc)₂ (80 mg, 0.4 mmol, 2.0 equiv.) were dissolved in DMSO (2 mL) and the reaction mixture was stirred at MW 150 °C for 30 min. After which, the ESI-HRMS analysis of the crude reaction mixture was carried out. The mass for indole-3-methyl radical trapping product **11** could be detected. The mass for **11** is C₂₃H₂₀N⁺ [M+H]⁺ calcd. 310.1590, found 310.1558. Then the reaction residue was extracted using EtOAc/H₂O and concentrated under reduced pressure followed by column chromatography over silica gel using petroleum / EtOAc = 10/1 as eluent to afford the desired product **3aa** (21 mg, 37% yield).



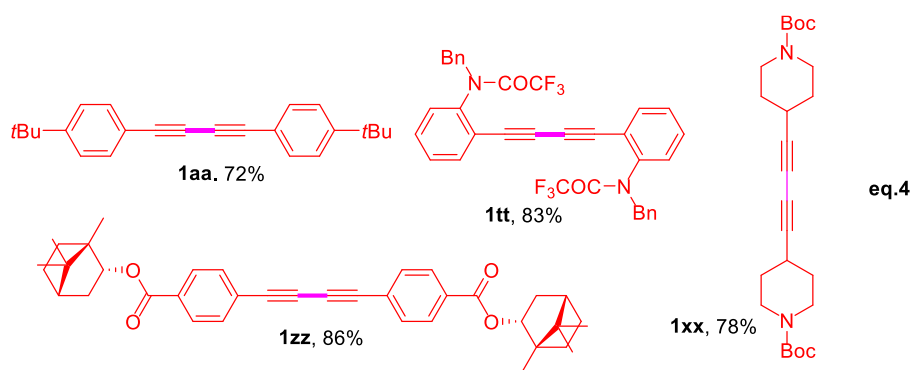
7.2 Trapping the indole-3-methyl radical with O₂



Scheme S4

In an oven-dried glass tubes 3-indoleacetic acid **2b** (76 mg, 0.4 mmol, 1.0 eq.) and Cu(OAc)₂ (160 mg, 0.8 mmol, 2.0 equiv.) were dissolved in DMSO (4 mL) and the reaction mixture was stirred at MW 150 °C for 30 min by microwave irradiation and monitored by TLC. Then the reaction mixture was concentrated under reduced pressure followed by column chromatography over silica gel using petroleum/EtOAc = 10/4 as eluent to afford the desired product **12** (17 mg, 26% yield) and **13** (85 mg, 70%).

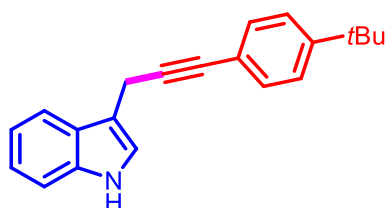
7.3 1,3-Diynes byproducts formation during the evaluation the substrate scope



Scheme S5

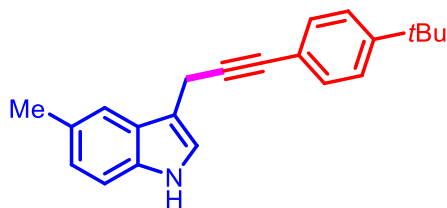
8. Characterization data of compounds

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1H-indole (3aa)



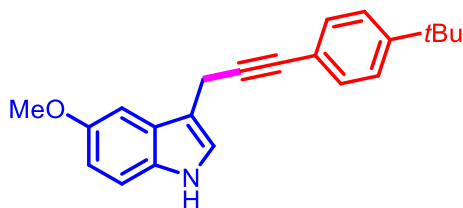
Brown solid, **MP**: 123-125 °C; 44 mg, 76% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.92 (s, 1H), 7.63 (d, $J = 7.7$ Hz, 1H), 7.30 (dd, $J = 8.1, 6.1$ Hz, 3H), 7.24 (d, $J = 8.3$ Hz, 2H), 7.14 (t, $J = 7.1$ Hz, 2H), 7.07 (t, $J = 7.3$ Hz, 1H), 3.84 (s, 3H), 1.23 (s, 9H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 150.9, 136.5, 131.3, 126.8, 125.2, 122.2, 122.0, 120.9, 119.5, 118.8, 111.9, 111.2, 87.1, 81.3, 34.7, 31.2, 16.3 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{21}\text{H}_{22}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 288.1747, found m/z 288.1750.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methyl-1H-indole (3ab)



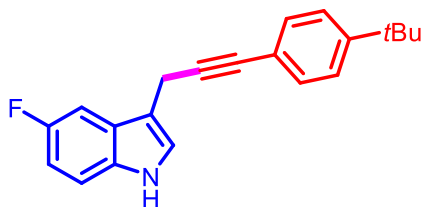
Brown solid, **MP**: 117-119 °C; 49 mg, 82% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.82 (s, 1H), 7.39 (s, 1H), 7.30 (ddd, $J = 11.0, 5.3, 3.2$ Hz, 2H), 7.26 – 7.20 (m, 2H), 7.17 (dd, $J = 8.0, 3.9$ Hz, 1H), 7.09 (s, 1H), 6.95 (dd, $J = 8.2, 2.2$ Hz, 1H), 3.80 (s, 2H), 2.39 (s, 3H), 1.23 (s, 9H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 150.8, 134.8, 132.3, 131.3, 128.7, 127.0, 125.5, 125.2, 123.8, 122.2, 120.9, 118.5, 111.3, 110.8, 87.3, 81.3, 34.7, 31.2, 21.6, 16.3 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{22}\text{H}_{24}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 302.1903, found m/z 302.1905.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methoxy-1H-indole (3ac)



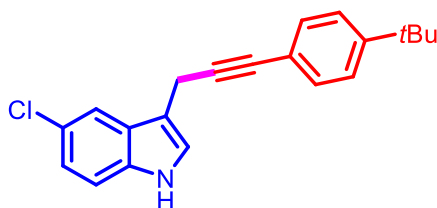
Yellow solid, **MP**: 134-136 °C; 52 mg, 82% yield, $R_f = 0.2$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.84 (s, 1H), 7.31 (d, $J = 8.3$ Hz, 2H), 7.24 (d, $J = 8.4$ Hz, 2H), 7.19 (t, $J = 4.4$ Hz, 1H), 7.12 (s, 1H), 7.07 (d, $J = 2.2$ Hz, 1H), 6.80 (dd, $J = 8.7, 2.3$ Hz, 1H), 3.80 (s, 3H), 3.80 (s, 2H), 1.23 (s, 9H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 154.0, 150.9, 131.6, 131.3, 127.2, 125.2, 122.8, 120.8, 112.5, 111.9, 111.5, 100.6, 87.1, 81.3, 55.9, 34.7, 31.2, 16.3 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{22}\text{H}_{24}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 318.1852, found m/z 318.1852.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-fluoro-1H-indole (3ad)



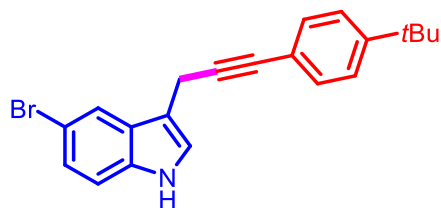
Colorless oil, 45 mg, 74% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.94 (s, 1H), 7.43 – 7.28 (m, 3H), 7.27–7.20 (m, 3H), 7.19 (d, $J = 4.8$ Hz, 1H), 6.88 (td, $J = 9.0, 2.3$ Hz, 1H), 3.78 (s, 2H), 1.23 (s, 9H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 158.9, 156.6, 152.6, 151.0, 133.0, 132.3, 131.3, 127.2, 127.1, 125.5, 125.2, 123.8, 120.7, 111.8, 111.7, 110.7, 110.5, 104.0, 103.8, 86.7, 81.5, 34.7, 31.2, 16.3 ppm; **$^{19}\text{F NMR}$** (376 MHz, CDCl_3) δ -124.42 (td, $J = 9.5, 4.1$ Hz) ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{21}\text{H}_{21}\text{FN}^+$ ($\text{M}+\text{H}$) $^+$ 306.1653, found m/z 306.1652.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-chloro-1H-indole (3ae)



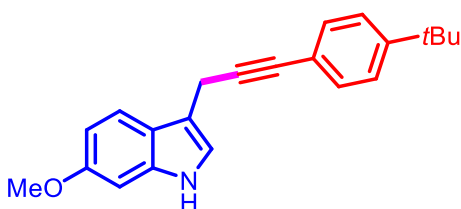
Yellow solid, **MP**: 150-152 °C; 50 mg, 77% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.97 (s, 1H), 7.60 (s, 1H), 7.31 (d, $J = 8.3$ Hz, 2H), 7.23 (d, $J = 6.6$ Hz, 1H), 7.19 (d, $J = 5.1$ Hz, 2H), 7.17 (s, 1H), 7.09 (d, $J = 8.6$ Hz, 1H), 3.79 (s, 2H), 1.23 (s, 9H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 151.0, 134.8, 131.3, 127.9, 125.2, 123.4, 122.5, 120.7, 118.4, 112.2, 111.7, 86.6, 81.6, 34.7, 31.2, 16.2 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{21}\text{H}_{21}\text{ClN}^+$ ($\text{M}+\text{H}$) $^+$ 322.1357, found m/z 322.1354.

5-bromo-3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1H-indole (3af)



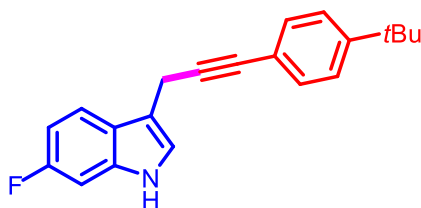
Yellow solid, **MP**: 165-167 °C; 55 mg, 75% yield, *R_f* = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.95 (s, 1H), 7.63 (d, *J* = 7.9 Hz, 1H), 7.34–7.28 (m, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.17 – 7.12 (m, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 3.85 (s, 2H), 1.23 (s, 9H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 150.9, 136.5, 131.3, 126.8, 125.2, 122.2, 122.0, 120.9, 119.5, 118.8, 111.9, 111.2, 87.1, 81.3, 34.7, 31.2, 16.3 ppm; **HRMS (ESI)** *m/z* calcd for C₂₁H₂₁BrN⁺ (M+H)⁺ 366.0852, found *m/z* 366.0853.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-6-methoxy-1H-indole (3ag)



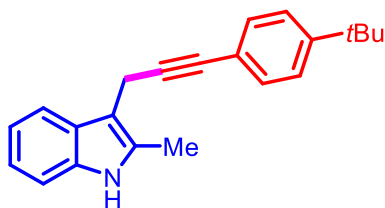
Yellow solid, **MP**: 143-145 °C; 55 mg, 86% yield, *R_f* = 0.2 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.80 (s, 1H), 7.50 (d, *J* = 8.7 Hz, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.08 – 6.98 (m, 1H), 6.79 (d, *J* = 2.1 Hz, 1H), 6.74 (dd, *J* = 8.6, 2.2 Hz, 1H), 3.80 (s, 2H), 3.78 (s, 3H), 1.23 (s, 9H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 156.7, 150.8, 137.3, 131.3, 125.2, 121.3, 120.9, 120.8, 119.5, 111.8, 109.5, 94.7, 87.2, 81.2, 55.7, 34.7, 31.2, 16.4 ppm; **HRMS (ESI)** *m/z* calcd for C₂₂H₂₄NO⁺ (M+H)⁺ 318.1852, found *m/z* 318.1849.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-6-fluoro-1H-indole (3ah)



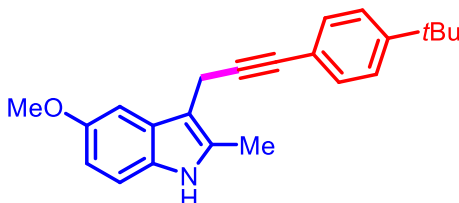
Colorless oil, 49 mg, 80% yield, *R_f* = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.93 (s, 1H), 7.54 (dd, *J* = 8.7, 5.4 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.27–7.23 (m, 2H), 7.13–7.09 (m, 1H), 6.97 (dd, *J* = 9.6, 2.3 Hz, 1H), 6.87–6.79 (m, 1H), 3.81 (s, 2H), 1.23 (s, 9H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 161.3, 158.9, 151.0, 136.4 (d, *J* = 12.5 Hz), 132.3, 131.3, 125.5, 125.2, 123.5, 122.3 (d, *J* = 3.7 Hz), 120.7, 119.6 (d, *J* = 10.3 Hz), 112.0, 108.3 (d, *J* = 24.6 Hz), 97.5 (d, *J* = 26.0 Hz) 86.8, 81.4, 34.7, 31.2, 16.3 ppm; **¹⁹F NMR** (376 MHz, CDCl₃) δ -121.1 (d, *J* = 5.4 Hz) ppm; **HRMS (ESI)** *m/z* calcd for C₂₁H₂₁FN⁺ (M+H)⁺ 306.1653, found *m/z* 306.1649.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-2-methyl-1H-indole (3ai)



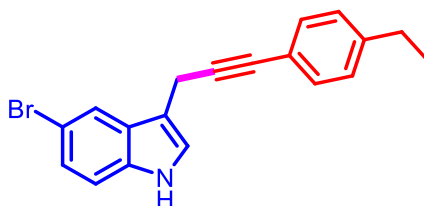
Yellow solid, **MP**: 107-109 °C; 32 mg, 53% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.79 (s, 1H), 7.73 – 7.67 (m, 1H), 7.46 (dd, $J = 14.7, 6.7$ Hz, 1H), 7.32 (d, $J = 8.5$ Hz, 2H), 7.28 (s, 2H), 7.16 – 7.07 (m, 2H), 3.83 (s, 2H), 2.46 (s, 3H), 1.28 (s, 9H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 150.7, 135.1, 131.3, 131.2, 128.3, 125.5, 125.1, 121.2, 121.0, 119.4, 118.3, 110.1, 106.8, 87.6, 80.4, 34.6, 31.2, 31.1, 14.9, 11.8 ppm; **HRMS (ESI)** m/z calcd for C₂₂H₂₄N⁺ (M+H)⁺ 302.1903, found m/z 302.1908.

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methoxy-2-methyl-1H-indole (3aj)



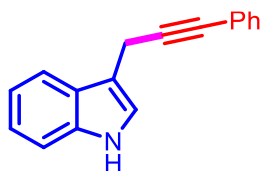
Yellow solid, **MP**: 123-125 °C; 53 mg, 80% yield, $R_f = 0.2$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.70 (s, 1H), 7.30 (dd, $J = 20.6, 8.4$ Hz, 4H), 7.18 (d, $J = 2.1$ Hz, 1H), 7.15 (d, $J = 8.6$ Hz, 1H), 6.78 (dd, $J = 8.7, 2.4$ Hz, 1H), 3.85 (s, 3H), 3.80 (s, 2H), 2.43 (s, 3H), 1.28 (s, 9H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 153.9, 150.7, 132.1, 131.3, 130.2, 128.6, 125.1, 120.9, 111.0, 110.9, 106.6, 100.6, 87.6, 80.4, 55.9, 34.7, 31.2, 15.0, 11.9 ppm; **HRMS (ESI)** m/z calcd for C₂₃H₂₆NO⁺ (M+H)⁺ 332.2009, found m/z 332.2011.

5-bromo-3-(3-(4-ethylphenyl)prop-2-yn-1-yl)-1H-indole (3ak)



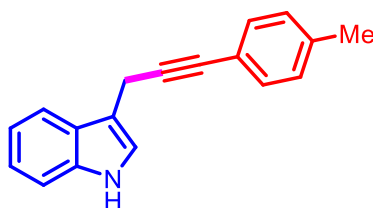
Yellow solid, **MP**: 144-146 °C; 40 mg, 60% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 8.05 (s, 1H), 7.84 (d, $J = 1.7$ Hz, 1H), 7.37 (d, $J = 8.1$ Hz, 2H), 7.29 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.24 (d, $J = 8.5$ Hz, 2H), 7.13 (d, $J = 8.1$ Hz, 2H), 3.86 (s, 2H), 2.64 (q, $J = 7.6$ Hz, 2H), 1.22 (t, $J = 7.6$ Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 144.1, 135.1, 131.6, 128.6, 127.8, 125.1, 123.3, 121.5, 120.9, 112.8, 112.6, 111.7, 86.5, 81.7, 28.8, 16.1, 15.3 ppm; **HRMS (ESI)** m/z calcd for C₁₉H₁₇BrN⁺ (M+H)⁺ 338.0539, found m/z 338.0534.

3-(3-phenylprop-2-yn-1-yl)-1H-indole (3ba)



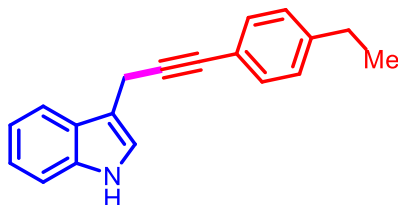
Yellow solid, **MP:** 122-124 °C; 34 mg, 73% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.97 (s, 1H), 7.70 (d, $J = 7.8$ Hz, 1H), 7.44 (dd, $J = 6.5, 2.8$ Hz, 2H), 7.35 (d, $J = 7.9$ Hz, 1H), 7.28 (dd, $J = 6.8, 3.5$ Hz, 3H), 7.24 – 7.19 (m, 2H), 7.15 (t, $J = 7.5$ Hz, 1H), 3.91 (s, 2H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 136.5, 131.7, 128.2, 127.7, 126.8, 123.9, 122.3, 122.0, 119.5, 118.8, 111.7, 111.2, 88.0, 81.3, 16.3 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{17}\text{H}_{14}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 232.1121, found m/z 232.1124.

3-(3-(*p*-tolyl)prop-2-yn-1-yl)-1H-indole (3ca)



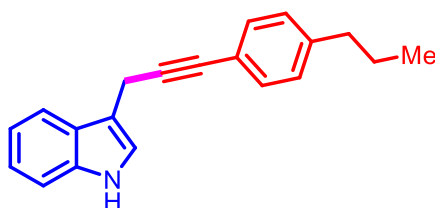
Yellow solid, **MP:** 111-113 °C; 40 mg, 81% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.92 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.28 (dd, $J = 12.0, 8.1$ Hz, 3H), 7.17–7.12 (m, 2H), 7.10–7.05 (m, 1H), 7.02 (d, $J = 7.9$ Hz, 2H), 3.84 (s, 2H), 2.26 (s, 3H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 137.7, 136.5, 131.5, 129.0, 126.8, 122.2, 122.0, 120.8, 119.5, 118.8, 111.9, 111.2, 87.1, 81.3, 21.4, 16.3 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{18}\text{H}_{16}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 246.1277, found m/z 246.1285.

3-(3-(4-ethylphenyl)prop-2-yn-1-yl)-1H-indole (3da)



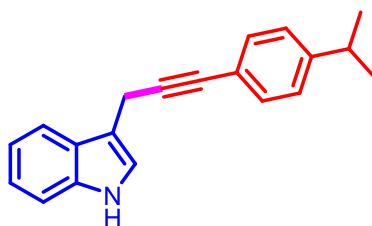
Yellow solid, **MP:** 104-106 °C; 43 mg, 84% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.91 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.29 (d, $J = 8.1$ Hz, 3H), 7.14 (t, $J = 7.5$ Hz, 2H), 7.06 (dd, $J = 13.4, 7.9$ Hz, 3H), 3.84 (s, 2H), 2.55 (q, $J = 7.6$ Hz, 2H), 1.14 (t, $J = 7.6$ Hz, 3H) ppm; **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ 144.0, 136.5, 131.6, 127.8, 126.8, 122.2, 122.1, 121.0, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 28.8, 16.3, 15.4 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{18}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 260.1434, found m/z 260.1431.

3-(3-(4-propylphenyl)prop-2-yn-1-yl)-1H-indole (3ea)



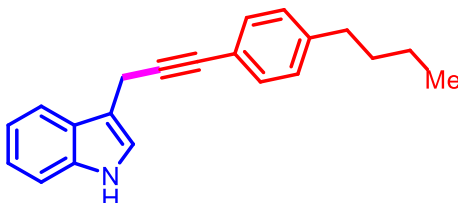
Yellow solid, **MP**: 99-101 °C; 48 mg, 87% yield, R_f = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.90 (s, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 7.9 Hz, 3H), 7.14 (t, *J* = 7.4 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 7.02 (d, *J* = 8.1 Hz, 2H), 3.83 (s, 2H), 2.69–2.32 (m, 2H), 1.54 (dd, *J* = 15.0, 7.5 Hz, 2H), 0.85 (t, *J* = 7.3 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 142.5, 136.5, 131.5, 128.4, 126.8, 122.2, 122.1, 121.0, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 37.9, 24.4, 16.3, 13.8 ppm; **HRMS (ESI)** *m/z* calcd for C₂₀H₂₀N⁺ (M+H)⁺ 274.1590, found *m/z* 274.1596.

3-(3-(4-isopropylphenyl)prop-2-yn-1-yl)-1H-indole (3fa)



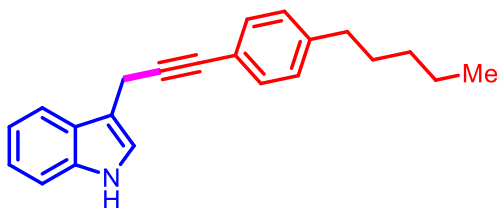
Yellow solid, **MP**: 119-121 °C; 45 mg, 82% yield, R_f = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.99 (s, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.37 (dd, *J* = 8.1, 4.6 Hz, 3H), 7.24 – 7.19 (m, 2H), 7.13 (dd, *J* = 10.5, 4.4 Hz, 3H), 3.91 (s, 2H), 2.88 (dt, *J* = 13.8, 6.9 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 6H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 148.6, 136.5, 131.6, 126.8, 126.4, 122.2, 122.0, 121.2, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 34.0, 23.8, 16.3 ppm; **HRMS (ESI)** *m/z* calcd for C₂₀H₂₀N⁺ (M+H)⁺ 274.1590, found *m/z* 274.1591.

3-(3-(4-butylphenyl)prop-2-yn-1-yl)-1H-indole (3ga)



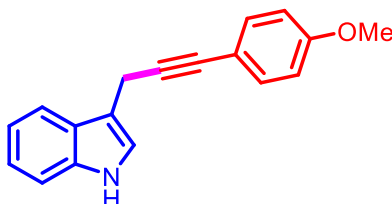
Brown solid, **MP**: 134-136 °C; 45 mg, 79% yield, R_f = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.92 (s, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.29 (dd, *J* = 7.9, 4.4 Hz, 3H), 7.14 (t, *J* = 7.2 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 7.03 (d, *J* = 8.1 Hz, 2H), 3.84 (s, 2H), 2.62 – 2.25 (m, 2H), 1.50 (dd, *J* = 8.8, 6.4 Hz, 2H), 1.33 – 1.21 (m, 2H), 0.84 (t, *J* = 7.3 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 142.7, 136.5, 131.5, 128.3, 126.8, 122.2, 122.0, 121.0, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 35.5, 33.4, 22.3, 16.3, 13.9 ppm; **HRMS (ESI)** *m/z* calcd for C₂₁H₂₂N⁺ (M+H)⁺ 288.1747, found *m/z* 288.1752.

3-(3-(4-pentylphenyl)prop-2-yn-1-yl)-1H-indole (3ha)



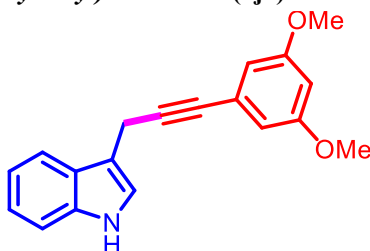
White solid, **MP**:127-129 °C; 53 mg, 88% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.91 (s, 1H), 7.63 (d, $J = 7.9$ Hz, 1H), 7.31 – 7.25 (m, 3H), 7.16–7.11 (m, 2H), 7.10–7.04 (m, 1H), 7.02 (d, $J = 8.2$ Hz, 2H), 3.84 (d, $J = 0.7$ Hz, 2H), 2.58 – 2.30 (m, 2H), 1.65 – 1.40 (m, 2H), 1.22 (ddd, $J = 15.5, 10.1, 7.4$ Hz, 4H), 0.81 (t, $J = 6.9$ Hz, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 142.8, 136.5, 131.5, 128.3, 126.8, 122.2, 122.1, 121.0, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 35.8, 31.5, 31.0, 22.5, 16.3, 14.0 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{22}\text{H}_{24}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 302.1903, found m/z 302.1900.

3-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ia)



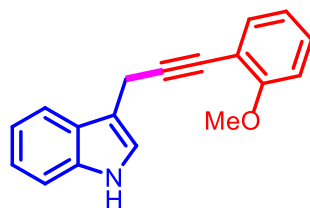
Yellow solid, **MP**:136-138 °C; 43 mg, 83% yield, $R_f = 0.2$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.92 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.30 (dd, $J = 8.0, 5.9$ Hz, 3H), 7.14 (t, $J = 7.2$ Hz, 2H), 7.07 (t, $J = 7.4$ Hz, 1H), 6.74 (d, $J = 8.7$ Hz, 2H), 3.83 (s, 2H), 3.72 (s, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 159.2, 136.5, 133.0, 126.8, 122.2, 122.0, 119.5, 118.8, 116.1, 113.8, 111.9, 111.2, 86.3, 81.0, 55.3, 16.2 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 262.1226, found m/z 262.1233.

3-(3-(3,5-dimethoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ja)



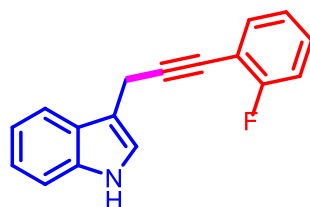
Yellow solid, **MP**:152-153 °C; 45 mg, 78% yield, $R_f = 0.3$ (ethyl acetate/hexane = 20%); $^1\text{H NMR}$ (400 MHz, CDCl_3) 7.96 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.30 (d, $J = 8.2$ Hz, 1H), 7.14 (dd, $J = 11.1, 3.9$ Hz, 2H), 7.08 (dd, $J = 10.9, 3.9$ Hz, 1H), 6.53 (d, $J = 2.2$ Hz, 2H), 6.34 (t, $J = 2.3$ Hz, 1H), 3.84 (s, 2H), 3.69 (s, 6H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) 160.5, 136.5, 126.8, 125.2, 122.3, 122.1, 119.5, 118.8, 111.6, 111.2, 109.5, 101.2, 87.7, 81.2, 55.4, 16.2 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{18}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$ 292.1332, found m/z 292.1331.

3-(3-(2-methoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ka)



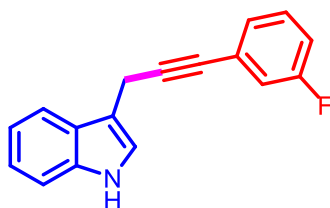
Yellow solid, **MP**:113-115 °C; 41 mg, 79% yield, $R_f = 0.2$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 8.01 (s, 1H), 7.72 (d, $J = 7.7$ Hz, 1H), 7.41 (dd, $J = 7.5, 1.5$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.29 – 7.17 (m, 3H), 7.14 (t, $J = 7.4$ Hz, 1H), 6.96 – 6.61 (m, 2H), 3.96 (s, 2H), 3.88 (s, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 160.0, 136.5, 133.7, 129.1, 126.8, 122.3, 122.1, 120.4, 119.4, 118.9, 113.0, 111.76, 111.2, 110.6, 92.2, 55.8, 16.7 ppm; **HRMS (ESI)** m/z calcd for C₁₈H₁₆NO⁺ (M+H)⁺ 262.1226, found m/z 262.1232.

3-(3-(2-fluorophenyl)prop-2-yn-1-yl)-1H-indole (3la)



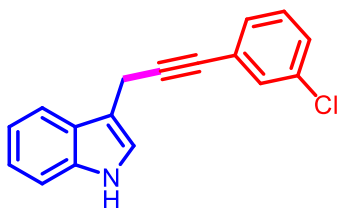
Yellow oil, 37 mg, 74% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.94 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.35 (dd, $J = 10.7, 4.2$ Hz, 1H), 7.30 (d, $J = 8.1$ Hz, 1H), 7.21–7.12 (m, 3H), 7.08 (t, $J = 7.5$ Hz, 1H), 6.98 (dd, $J = 12.5, 5.0$ Hz, 2H), 3.89 (s, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 164.2, 161.7, 136.5, 133.6, 133.6, 129.3 (d, $J = 8.1$ Hz), 126.7, 123.8 (d, $J = 3.7$ Hz), 122.3, 122.2, 119.5, 118.8, 115.4 (d, $J = 21.3$ Hz), 112.4 (d, $J = 15.8$ Hz), 111.3, 111.2, 93.4, 93.4, 74.7, 16.5 ppm; **¹⁹F NMR** (376 MHz, CDCl₃) δ -107.68 – -111.95 (m) ppm; **HRMS (ESI)** m/z calcd for C₁₇H₁₃FN⁺ (M+H)⁺ 250.1027, found m/z 250.1023.

3-(3-(3-fluorophenyl)prop-2-yn-1-yl)-1H-indole (3ma)



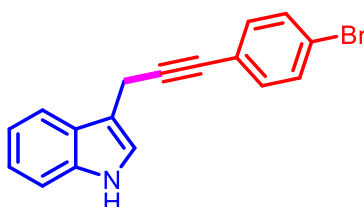
Yellow solid, **MP**:88-89 °C; 35 mg, 70% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) 7.95 (s, 1H), 7.62 (d, $J = 7.8$ Hz, 1H), 7.31 (d, $J = 8.1$ Hz, 1H), 7.17 – 7.12 (m, 4H), 7.07 (dd, $J = 16.0, 8.5$ Hz, 2H), 6.91 (td, $J = 7.1, 2.0$ Hz, 1H), 3.84 (s, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 163.6, 161.2, 136.5, 130.9, 129.7 (d, $J = 8.4$ Hz), 128.9, 127.5 (d, $J = 3.3$ Hz), 126.7, 125.7 (d, $J = 9.5$ Hz), 122.3, 122.0, 119.6, 118.8, 118.5 (d, $J = 22.4$ Hz), 115.0 (d, $J = 21.3$ Hz), 111.3, 111.2, 89.1, 80.2, 80.1, 65.6, 16.2 ppm; **HRMS (ESI)** m/z calcd for C₁₇H₁₃FN⁺ (M+H)⁺ 250.1027, found m/z 250.1033.

3-(3-(3-chlorophenyl)prop-2-yn-1-yl)-1H-indole (3na)



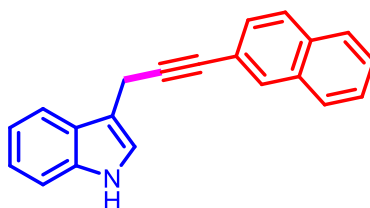
White solid, **MP**:123-125 °C; 38 mg, 72% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.94 (s, 1H), 7.65 – 7.59 (m, 1H), 7.35 (t, $J = 1.6$ Hz, 1H), 7.33 – 7.28 (m, 1H), 7.23 (dt, $J = 7.3, 1.4$ Hz, 1H), 7.21 – 7.12 (m, 4H), 7.11 – 7.03 (m, 1H), 3.84 (d, $J = 1.0$ Hz, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 136.5, 134.0, 131.6, 129.8, 129.4, 128.0, 126.7, 125.6, 122.3, 122.0, 119.6, 118.8, 111.3, 111.3, 89.4, 80.0, 16.2 ppm; **HRMS (ESI)** m/z calcd for C₁₇H₁₃ClN⁺ (M+H)⁺ 266.0731, found m/z 266.0734.

3-(3-(4-bromophenyl)prop-2-yn-1-yl)-1H-indole (3oa)



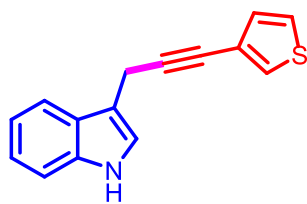
Yellow solid, **MP**:133-135 °C; 47 mg, 76% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.93 (s, 1H), 7.64 (d, $J = 7.8$ Hz, 1H), 7.37 (dd, $J = 6.5, 3.2$ Hz, 2H), 7.30 (d, $J = 8.1$ Hz, 1H), 7.21 (dd, $J = 5.0, 1.9$ Hz, 2H), 7.17 – 7.12 (m, 2H), 7.11 – 7.03 (m, 1H), 3.85 (d, $J = 0.7$ Hz, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 136.5, 131.7, 128.2, 127.7, 126.8, 123.9, 122.3, 122.0, 119.5, 118.8, 111.7, 111.2, 88.0, 81.3, 16.3 ppm; **HRMS (ESI)** m/z calcd for C₁₇H₁₃BrN⁺ (M+H)⁺ 310.0226, found m/z 310.0227.

3-(3-(naphthalen-2-yl)prop-2-yn-1-yl)-1H-indole (3pa)



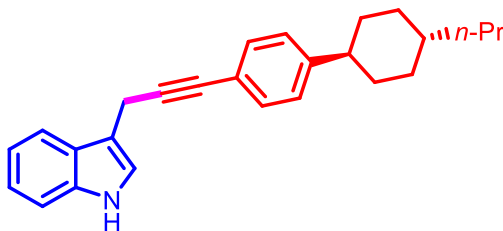
Slight yellow solid, **MP**:130-132 °C; 48 mg, 85% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.93 (s, 1H), 7.88 (s, 1H), 7.77 – 7.59 (m, 4H), 7.47 – 7.36 (m, 3H), 7.30 (d, $J = 7.9$ Hz, 1H), 7.16 (dd, $J = 10.7, 5.3$ Hz, 2H), 7.09 (t, $J = 7.4$ Hz, 1H), 3.90 (s, 2H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 136.5, 133.0, 132.6, 131.2, 128.7, 127.8, 127.7, 127.6, 126.8, 126.4, 126.4, 122.3, 122.1, 121.2, 119.6, 118.9, 111.7, 111.2, 88.4, 81.6, 16.4 ppm; **HRMS (ESI)** m/z calcd for C₂₁H₁₆N⁺ (M+H)⁺ 282.1277, found m/z 282.1273.

3-(3-(thiophen-3-yl)prop-2-yn-1-yl)-1H-indole (3qa)



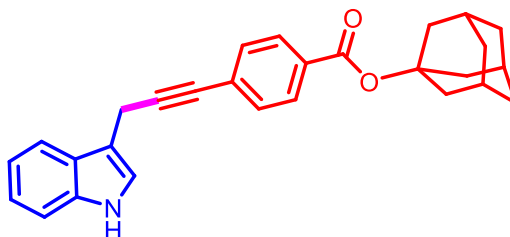
Yellow oil, 34 mg, 71% yield, Rf = 0.4 (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 (s, 1H), 7.61 (d, $J = 7.8$ Hz, 1H), 7.30 (d, $J = 8.1$ Hz, 1H), 7.17 – 7.05 (m, 5H), 6.87 (dd, $J = 5.1$, 3.7 Hz, 1H), 3.87 (s, 2H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.4, 131.2, 126.8, 126.7, 126.2, 124.0, 122.3, 122.1, 119.6, 118.8, 111.2, 92.0, 74.4, 16.5 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{12}\text{NS}^+$ ($\text{M}+\text{H}$) $^+$ 238.0685, found m/z 238.0685.

3-(3-(4-((1*s*,4*r*)-4-propylcyclohexyl)phenyl)prop-2-yn-1-yl)-1H-indole (3ra)



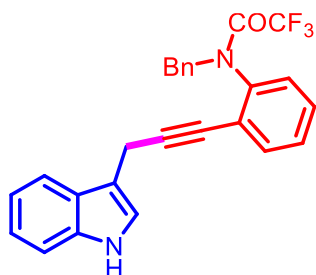
Yellow solid, **MP**:127-128 °C; 62 mg, 87% yield, Rf = 0.4 (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.91 (s, 1H), 7.62 (d, $J = 7.8$ Hz, 1H), 7.47 – 7.23 (m, 4H), 7.12 (dd, $J = 14.1$, 7.2 Hz, 2H), 7.06 (s, 2H), 3.83 (s, 2H), 2.36 (ddd, $J = 12.1$, 9.1, 3.1 Hz, 1H), 1.78 (d, $J = 10.8$ Hz, 4H), 1.36 (dd, $J = 12.7$, 10.1 Hz, 2H), 1.27 (d, $J = 6.9$ Hz, 3H), 1.16 – 1.12 (m, 2H), 0.96 (d, $J = 11.5$ Hz, 2H), 0.82 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 147.7, 136.5, 132.5, 131.6, 127.0, 126.8, 126.8, 122.2, 122.1, 121.2, 119.5, 118.8, 111.9, 111.2, 87.1, 81.4, 44.5, 39.7, 37.0, 34.2, 33.5, 20.0, 16.3, 14.4 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{26}\text{H}_{30}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 356.2373, found m/z 356.23736.

adamantan-1-yl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (3sa)



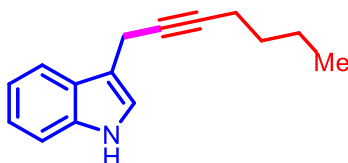
Yellow solid, **MP**:153-155 °C; 63 mg, 77% yield, Rf = 0.2 (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.05 (s, 1H), 7.90 (d, $J = 8.3$ Hz, 2H), 7.71 (d, $J = 7.8$ Hz, 1H), 7.46 (d, $J = 8.3$ Hz, 2H), 7.38 (d, $J = 8.1$ Hz, 1H), 7.22 (dd, $J = 8.2$, 5.9 Hz, 2H), 7.16 (t, $J = 7.1$ Hz, 1H), 3.94 (s, 2H), 2.23 (d, $J = 11.4$ Hz, 9H), 1.78 – 1.64 (m, 6H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.0, 136.5, 131.4, 131.0, 129.3, 128.1, 126.7, 122.3, 122.0, 119.6, 118.8, 111.3, 91.0, 81.3, 80.9, 41.4, 36.2, 30.9, 16.4 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{28}\text{H}_{28}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$ 410.2115, found m/z 410.2114.

N-(2-(3-(1H-indol-3-yl)prop-1-yn-1-yl)phenyl)-N-benzyl-2,2,2-trifluoroacetamide (3ta)



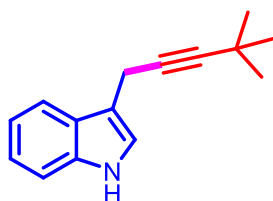
Yellow oil, 33 mg, 39% yield, $R_f = 0.3$ (ethyl acetate/hexane = 20%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.10 (s, 1H), 7.66 (d, $J = 7.8$ Hz, 1H), 7.57 – 7.50 (m, 1H), 7.37 (dd, $J = 11.7, 5.3$ Hz, 2H), 7.32 – 7.27 (m, 2H), 7.25 – 7.23 (m, 2H), 7.19 – 7.15 (m, 2H), 7.14 – 7.08 (m, 3H), 6.71 (d, $J = 7.9$ Hz, 1H), 5.53 (d, $J = 14.1$ Hz, 1H), 4.19 (d, $J = 14.1$ Hz, 1H), 3.94 (s, 2H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 157.2 (q, $J = 36.0$ Hz), 139.5, 136.4, 135.2, 133.2, 130.0, 129.4, 129.0, 128.5, 128.0, 127.9, 126.6, 123.7, 122.4, 122.0, 119.6, 118.6, 116.4 (q, $J = 288.6$ Hz) 111.3, 110.9, 94.8, 53.9, 16.4 ppm; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -68.07 (d, $J = 7.5$ Hz) ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{26}\text{H}_{20}\text{F}_3\text{N}_2\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 433.1522, found m/z 433.1525.

3-(hept-2-yn-1-yl)-1H-indole (3ua)



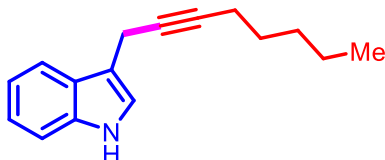
Colorless oil, 31 mg, 73% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.87 (s, 1H), 7.57 (d, $J = 7.9$ Hz, 1H), 7.27 (d, $J = 8.1$ Hz, 1H), 7.12 (t, $J = 7.5$ Hz, 1H), 7.09 – 7.00 (m, 2H), 3.59 (s, 2H), 2.27 – 1.93 (m, 2H), 1.43 (dd, $J = 14.9, 7.5$ Hz, 2H), 1.36 (dd, $J = 15.0, 7.3$ Hz, 2H), 0.84 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.5, 126.8, 122.1, 121.9, 119.4, 118.8, 112.7, 111.1, 81.2, 77.8, 31.2, 22.0, 18.6, 15.5, 13.7 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{18}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 212.1434, found m/z 212.1431.

3-(4,4-dimethylpent-2-yn-1-yl)-1H-indole (3va)



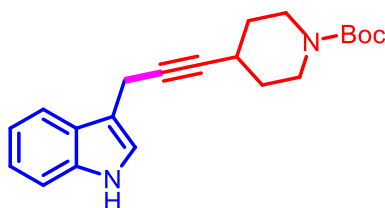
Colorless oil, 30 mg, 71% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.93 (s, 1H), 7.64 (d, $J = 7.9$ Hz, 1H), 7.34 (d, $J = 8.1$ Hz, 1H), 7.19 (t, $J = 7.2$ Hz, 1H), 7.16 – 7.03 (m, 2H), 3.65 (s, 2H), 1.25 (s, 9H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.5, 126.9, 122.1, 121.9, 119.3, 118.9, 112.8, 111.1, 89.9, 76.2, 31.4, 27.5, 15.6 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{18}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 212.1434, found m/z 212.1438.

3-(oct-2-yn-1-yl)-1H-indole (3wa)



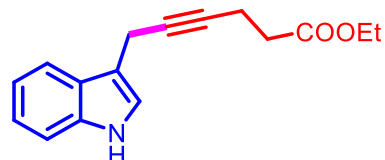
Colorless oil, 34 mg, 76% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.89 (s, 1H), 7.57 (d, $J = 7.8$ Hz, 1H), 7.28 (d, $J = 8.1$ Hz, 1H), 7.13 (t, $J = 7.5$ Hz, 1H), 7.09 – 6.98 (m, 2H), 3.59 (d, $J = 0.9$ Hz, 2H), 2.14 (tt, $J = 7.1, 2.3$ Hz, 2H), 1.45 (dd, $J = 14.9, 7.5$ Hz, 2H), 1.37 – 1.20 (m, 4H), 0.83 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 136.5, 126.8, 122.1, 121.8, 119.4, 118.9, 112.7, 111.1, 81.2, 77.9, 31.2, 28.8, 22.3, 18.8, 15.5, 14.0 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{16}\text{H}_{20}\text{N}^+$ ($\text{M}+\text{H}$) $^+$ 226.1590, found m/z 226.1587.

adamantan-1-yl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (3xa)



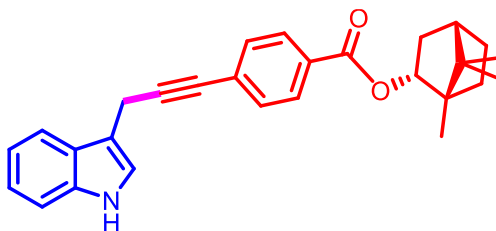
Yellow solid, **MP**:151-153 °C; 49 mg, 73% yield, $R_f = 0.5$ (ethyl acetate/hexane = 20%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.06 (s, 1H), 7.63 (d, $J = 7.8$ Hz, 1H), 7.36 (d, $J = 8.1$ Hz, 1H), 7.23 – 7.17 (m, 1H), 7.12 (td, $J = 7.6, 0.9$ Hz, 2H), 3.76 – 3.63 (m, 4H), 3.18 (ddd, $J = 13.2, 8.5, 3.4$ Hz, 2H), 2.61 (tdd, $J = 6.0, 4.1, 2.0$ Hz, 1H), 1.84 – 1.74 (m, 2H), 1.64 – 1.56 (m, 2H), 1.46 (s, 9H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 154.87, 136.54, 126.77, 122.17, 121.80, 119.38, 118.77, 112.36, 111.18, 82.98, 79.49, 79.45, 42.27, 31.75, 28.47, 27.16, 15.54 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 339.2067, found m/z 339.2063.

ethyl 6-(1H-indol-3-yl)hex-4-ynoate (3ya)



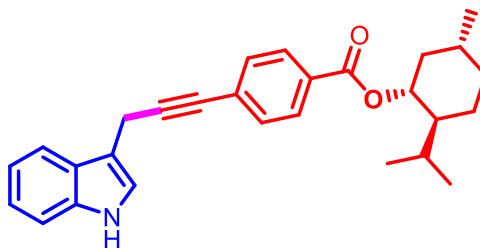
Colorless oil, 40 mg, 79% yield, $R_f = 0.2$ (ethyl acetate/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 (s, 1H), 7.62 (d, $J = 7.7$ Hz, 1H), 7.40 – 7.29 (m, 1H), 7.20 (t, $J = 7.5$ Hz, 1H), 7.15 – 7.06 (m, 2H), 4.14 (ddd, $J = 9.9, 7.2, 3.7$ Hz, 2H), 3.64 (s, 2H), 2.54 (s, 4H), 1.24 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 172.2, 136.5, 126.8, 122.2, 121.9, 119.4, 118.8, 112.3, 111.1, 79.0, 78.8, 60.5, 34.1, 15.5, 14.9, 14.2 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{16}\text{H}_{18}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$ 256.1332, found m/z 256.1330.

(1S,2R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (4)



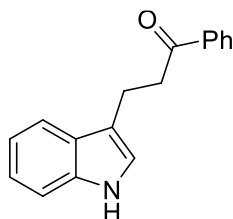
Yellow solid, **MP**:132-134 °C; 66 mg, 80% yield, R_f = 0.2 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 8.01 (s, 1H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 1H), 7.15 (dd, *J* = 3.1, 2.1 Hz, 2H), 7.08 (t, *J* = 7.5 Hz, 1H), 5.15 – 4.93 (m, 1H), 3.88 (s, 2H), 2.52 – 2.30 (m, 1H), 2.11 – 1.94 (m, 1H), 1.82 – 1.68 (m, 1H), 1.66 (t, *J* = 4.5 Hz, 1H), 1.38 – 1.28 (m, 2H), 1.04 (dd, *J* = 13.8, 3.3 Hz, 1H), 0.89 (s, 3H), 0.84 (d, *J* = 2.4 Hz, 6H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 166.4, 136.5, 131.6, 129.8, 129.3, 128.5, 126.7, 122.3, 122.1, 119.6, 118.8, 111.3, 111.2, 91.3, 80.8, 80.7, 49.1, 47.9, 45.0, 36.9, 28.1, 27.4, 19.7, 18.9, 16.4, 13.6 ppm; **HRMS (ESI)** *m/z* calcd for C₂₈H₃₀NO₂⁺ (M+H)⁺ 412.2271, found *m/z* 412.2278.

(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (5)



White solid, **MP**:141-143 °C; 60 mg, 73% yield, R_f = 0.2 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 8.03 (s, 1H), 7.88 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 1H), 7.13 (dd, *J* = 12.9, 4.7 Hz, 2H), 7.10 – 7.05 (m, 1H), 4.84 (td, *J* = 10.8, 4.3 Hz, 1H), 3.86 (s, 2H), 2.04 (dd, *J* = 6.8, 5.0 Hz, 1H), 1.86 (dtd, *J* = 13.9, 7.0, 2.7 Hz, 1H), 1.64 (dd, *J* = 14.1, 2.4 Hz, 2H), 1.54 – 1.39 (m, 2H), 1.09 – 0.97 (m, 2H), 0.84 (dd, *J* = 6.8, 2.6 Hz, 6H), 0.71 (d, *J* = 7.0 Hz, 3H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 165.8, 136.5, 131.5, 129.7, 129.4, 128.5, 126.7, 122.3, 122.1, 119.6, 118.8, 111.3, 111.1, 91.3, 80.8, 75.1, 47.3, 41.0, 34.3, 31.5, 26.5, 23.7, 22.1, 20.8, 16.6, 16.4 ppm; **HRMS (ESI)** *m/z* calcd for C₂₈H₃₂NO₂⁺ (M+H)⁺ 414.2428, found *m/z* 414.2425.

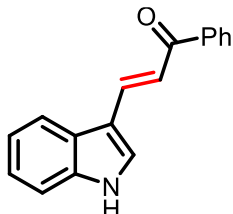
3-(1H-indol-3-yl)-1-phenylpropan-1-one (6)



Yellow solid, 120 mg, 60% yield, R_f = 0.4 (ethyl acetate/hexane = 10%); **¹H NMR** (400 MHz, CDCl₃) δ 7.96 (t, *J* = 7.2 Hz, 3H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.56-7.53=0.03(m, 1H), 7.46-7.42 (m, 2H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.22-7.18 (m, 1H), 7.15-7.11 (m, 1H), 7.04 (d, *J* = 2.0 Hz, 1H), 3.38 (t, *J* = 8.0 Hz,

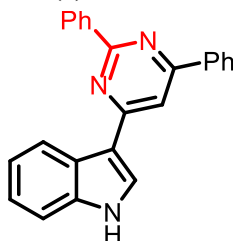
2H), 3.23 (t, $J = 8.0$ Hz, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 200.0, 137.0, 136.3, 133.0, 128.6, 128.1, 127.3, 122.1, 121.6, 119.3, 118.7, 115.5, 111.2, 39.3, 19.1 ppm; HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{16}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 250.1226, found m/z 250.1228.

(E)-3-(1H-indol-3-yl)-1-phenylprop-2-en-1-one (7)



Yellow solid, 37 mg, 75% yield, $R_f = 0.4$ (ethyl acetate/hexane = 10%); ^1H NMR (400 MHz, CDCl_3) δ 8.82 (s, 1H), 8.16 – 7.99 (m, 4H), 7.65 – 7.55 (m, 3H), 7.52 (t, $J = 7.3$ Hz, 2H), 7.45 (dt, $J = 7.2, 3.3$ Hz, 1H), 7.35 – 7.28 (m, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 191.1, 139.0, 137.3, 132.3, 130.4, 128.6, 128.4, 125.4, 123.6, 121.8, 120.7, 117.9, 114.5, 112.0 ppm; HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{14}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 248.1070, found m/z 248.1071.

3-(2,6-diphenylpyrimidin-4-yl)-1H-indole (9)



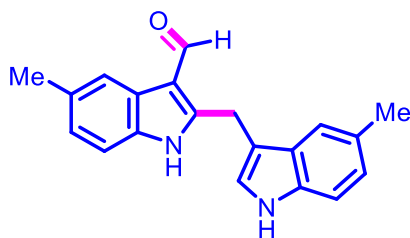
Yellow solid, MP: 155–157 °C; 46 mg, 66% yield, $R_f = 0.4$ (ethyl acetate/hexane = 20%); ^1H NMR (400 MHz, CDCl_3) δ 8.74 (dd, $J = 8.1, 1.4$ Hz, 2H), 8.64 (d, $J = 7.9$ Hz, 1H), 8.62 (s, 1H), 8.28 (dd, $J = 7.9, 1.5$ Hz, 2H), 8.04 (d, $J = 2.8$ Hz, 1H), 7.90 (s, 1H), 7.59 – 7.53 (m, 5H), 7.41 (d, $J = 8.0$ Hz, 1H), 7.37 – 7.26 (m, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 164.5, 163.7, 162.6, 138.7, 138.0, 137.0, 130.5, 130.4, 128.9, 128.5, 127.2, 126.6, 125.5, 124.4, 123.5, 123.2, 121.9, 121.7, 116.2, 111.7, 109.9 ppm; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{18}\text{N}_3^+$ ($\text{M}+\text{H}$) $^+$ 348.1495, found m/z 348.1491.

5-methyl-1H-indole-3-carbaldehyde (12)



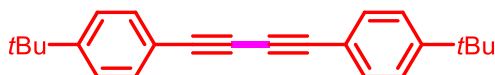
Yellow oil, 17 mg, 26% yield, $R_f = 0.5$ (ethyl acetate/hexane = 40%); ^1H NMR (400 MHz, CDCl_3) δ 9.97 (s, 1H), 8.68 (s, 1H), 8.06 (s, 1H), 7.73 (d, $J = 3.1$ Hz, 1H), 7.25 (d, $J = 8.3$ Hz, 1H), 7.08 (d, $J = 8.1$ Hz, 1H), 2.41 (s, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 185.1, 135.3, 134.9, 132.8, 128.8, 126.0, 124.7, 121.7, 119.5, 111.1, 21.4 ppm; HRMS (ESI) m/z calcd for $\text{C}_{10}\text{H}_{10}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 160.0757, found m/z 160.0759.

5-methyl-2-((5-methyl-1H-indol-3-yl)methyl)-1H-indole-3-carbaldehyde (13)



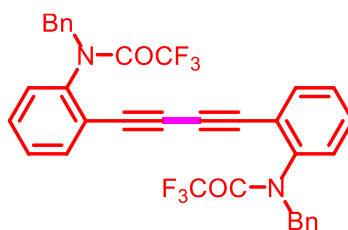
Yellow oil, 85 mg, 70% yield, $R_f = 0.3$ (ethyl acetate/hexane = 40%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.77 (s, 1H), 8.18 (s, 1H), 8.06 (s, 1H), 7.51 (s, 1H), 7.35 (d, $J = 8.4$ Hz, 1H), 7.25 (d, $J = 8.3$ Hz, 1H), 7.12 (dd, $J = 8.4, 7.2$ Hz, 3H), 7.01 (d, $J = 8.3$ Hz, 1H), 5.36 (s, 2H), 2.42 (s, 3H), 2.32 (s, 3H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 184.7, 138.2, 135.9, 134.8, 132.8, 130.0, 126.6, 126.0, 125.4, 124.7, 124.3, 121.9, 118.0, 117.6, 111.3, 109.8, 109.0, 42.5, 21.5, 21.4 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{20}\text{H}_{19}\text{N}_2\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 303.1492, found m/z 303.1498.

1,4-bis(4-(tert-butyl)phenyl)buta-1,3-diyne (1aa)



White solid, **MP**: 113-115 °C; 23 mg, 72% yield, $R_f = 0.4$ (hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 (d, $J = 8.0$ Hz, 4H), 7.27 (d, $J = 8.0$ Hz, 4H), 1.23 (s, 18H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 153.6, 132.3, 125.5, 118.8, 81.5, 73.5, 34.9, 31.1 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{24}\text{H}_{27}^+$ ($\text{M}+\text{H}$) $^+$ 315.2107, found m/z 315.2109.

***N,N'*-(buta-1,3-diyne-1,4-diylbis(2,1-phenylene))bis(*N*-benzyl-2,2,2-trifluoroacetamide) (1tt)**



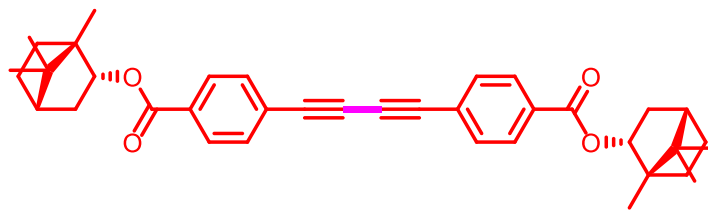
Colorless oil, 50 mg, 83% yield, $R_f = 0.4$ (EtOAc/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.68 (d, $J = 7.7$ Hz, 2H), 7.38 (t, $J = 7.6$ Hz, 2H), 7.31 – 7.24 (m, 8H), 7.22 – 7.16 (m, 4H), 6.78 (d, $J = 7.8$ Hz, 2H), 5.65 (t, $J = 13.5$ Hz, 2H), 4.36 (dd, $J = 14.1, 5.7$ Hz, 2H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 157.01 (q, $J = 36.1$ Hz), 140.48, 134.89, 134.86, 134.46, 134.43, 130.42, 129.80, 129.61, 129.32, 128.65, 128.32, 128.30, 121.61, 116.33 (q, $J = 288.5$ Hz), 78.71, 78.40, 78.34, 54.35 ppm; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -68.07 (d, $J = 7.5$ Hz) ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{34}\text{H}_{23}\text{F}_6\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 605.1658, found m/z 605.1663.

***di*-tert-butyl 4,4'-(buta-1,3-diyne-1,4-diyl)bis(piperidine-1-carboxylate) (1xx)**



Colorless oil, 32 mg, 78% yield, $R_f = 0.4$ (EtOAc/hexane = 10%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 3.68 (ddd, $J = 13.0, 6.2, 3.5$ Hz, 4H), 3.24 – 3.12 (m, 4H), 2.65 (dq, $J = 12.0, 3.9$ Hz, 2H), 1.83 – 1.72 (m, 4H), 1.63 – 1.56 (m, 4H), 1.45 (s, 18H) ppm; $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 154.7, 80.0, 79.6, 66.3, 31.0, 28.4, 27.5 ppm; **HRMS (ESI)** m/z calcd for $\text{C}_{24}\text{H}_{37}\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}$) $^+$ 417.2748, found m/z 417.2743.

bis((1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl) 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoate
(1zz)



White Solid, **MP**:143-145 °C; 48 mg, 86% yield, $R_f = 0.4$ (EtOAc/hexane = 5%); **¹H NMR** (400 MHz, CDCl₃) δ 7.96 (d, $J = 8.0$ Hz, 4H), 7.53 (d, $J = 7.9$ Hz, 4H), 5.05 (d, $J = 9.2$ Hz, 2H), 2.41 (dd, $J = 13.0, 10.0$ Hz, 2H), 2.10 – 1.99 (m, 2H), 1.80 – 1.65 (m, 4H), 1.34 (t, $J = 12.0$ Hz, 3H), 1.05 (d, $J = 11.5$ Hz, 3H), 0.90 (s, 6H), 0.85 (s, 12H) ppm; **¹³C NMR** (100 MHz, CDCl₃) δ 166.0, 132.4, 131.3, 129.5, 125.9, 81.9, 81.0, 76.2, 49.1, 47.9, 45.0, 36.9, 28.1, 27.4, 19.7, 18.9, 13.6 ppm; **HRMS (ESI)** m/z calcd for C₃₈H₄₃O₄⁺ (M+H)⁺ 563.3156, found m/z 563.3159.

9. Reference

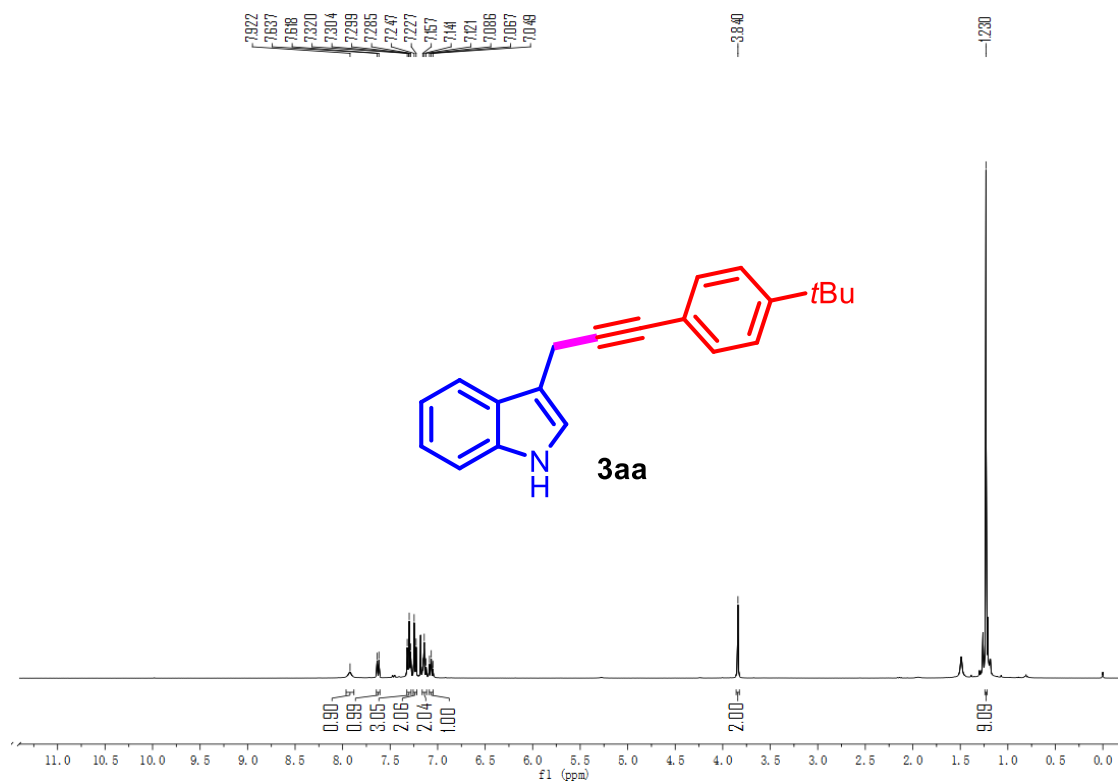
1. X. Wu, A. Liu, S. Xu, J. He, W. Sun, S. Chen, *Org. Lett.* 2018, **20**, 1538-1541.
2. M. Zhang, J. Xie, C. Zhu, *Nat. Commun.* 2018, **9**, 3517.
3. X. Jie, Y. Shang, X. Zhang, W. Su, *J. Am. Soc. Chem.* 2016, **138**, 5623-5633.

NOTE: Resonances from hydrocarbon grease at $\delta=1.25$ ppm are a result of contamination in the NMR facility. This hydrocarbon grease resonance can be seen in NMR spectra taken of only solvent. Residual solvent may remain for products that were volatile.

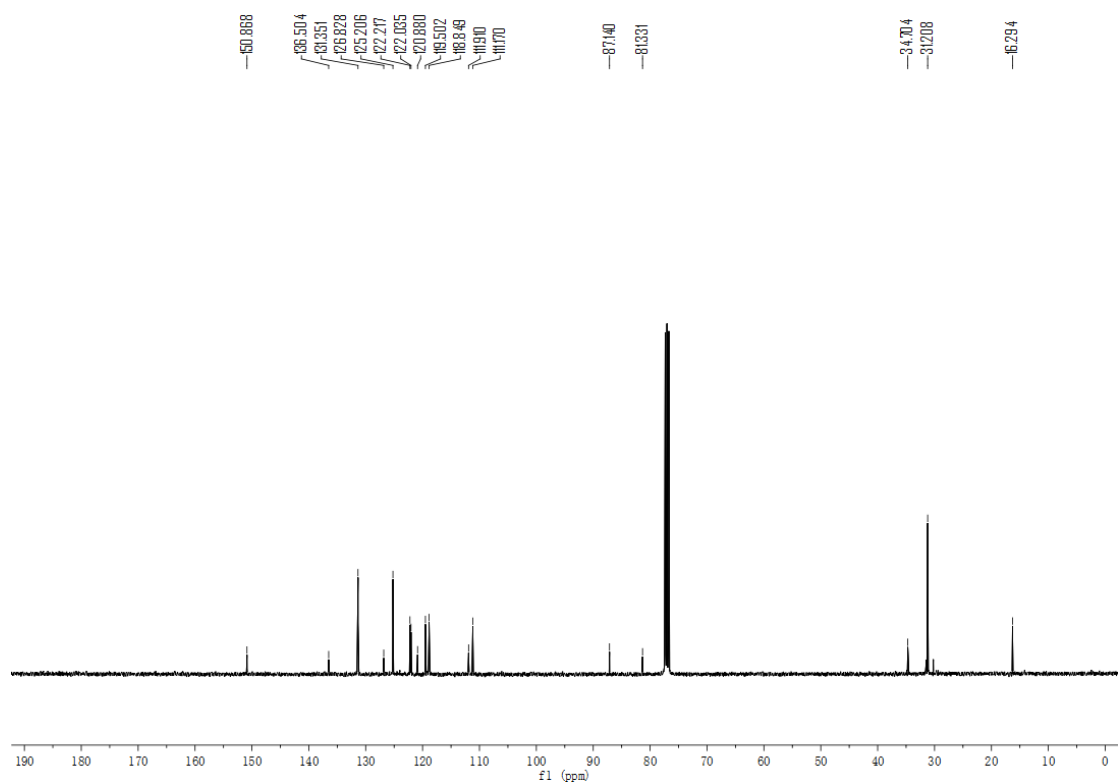
10. Copies ¹H NMR, ¹³C NMR, ¹⁹F NMR

3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1H-indole (3aa)

¹H NMR (400 MHz, CDCl₃):

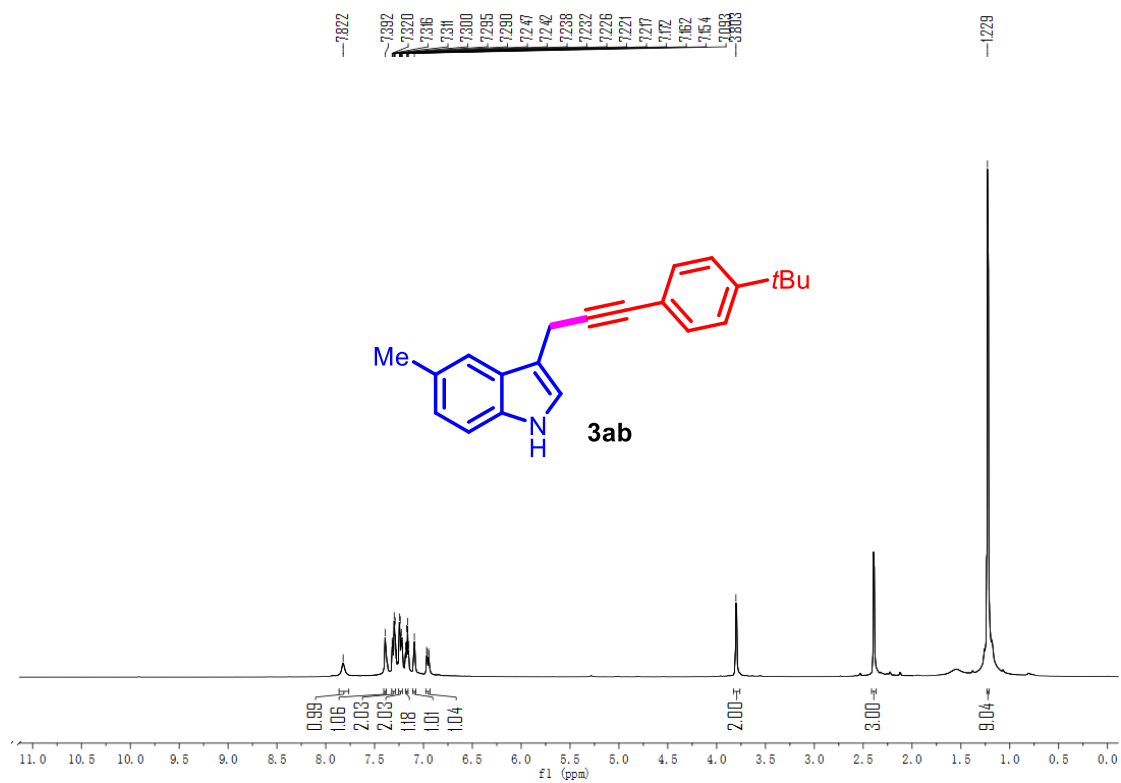


¹³C NMR (100 MHz, CDCl₃):

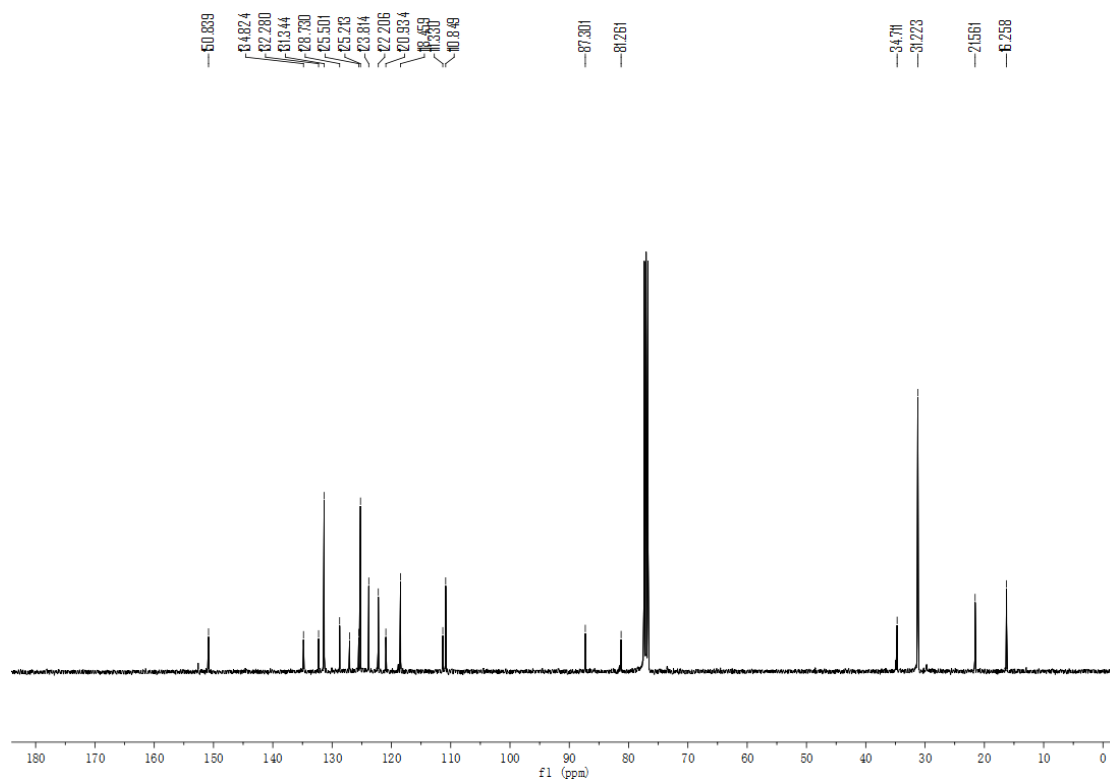


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methyl-1H-indole (3ab)

^1H NMR (400 MHz, CDCl_3):

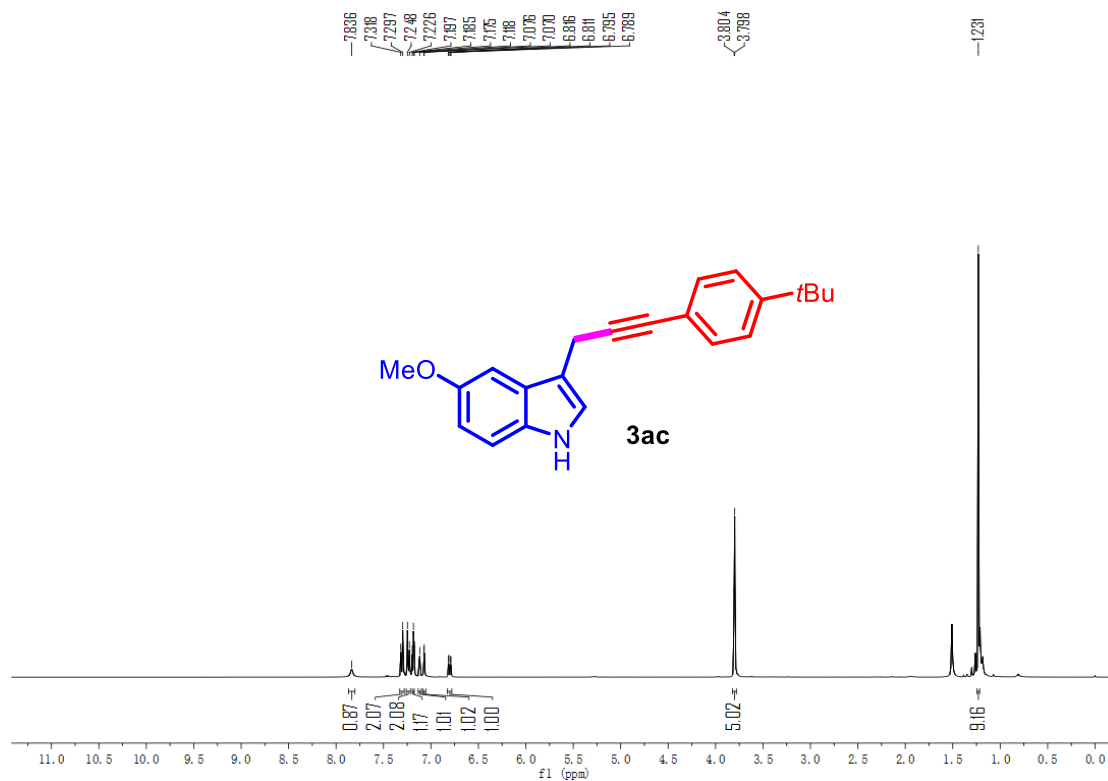


^{13}C NMR (100 MHz, CDCl_3):

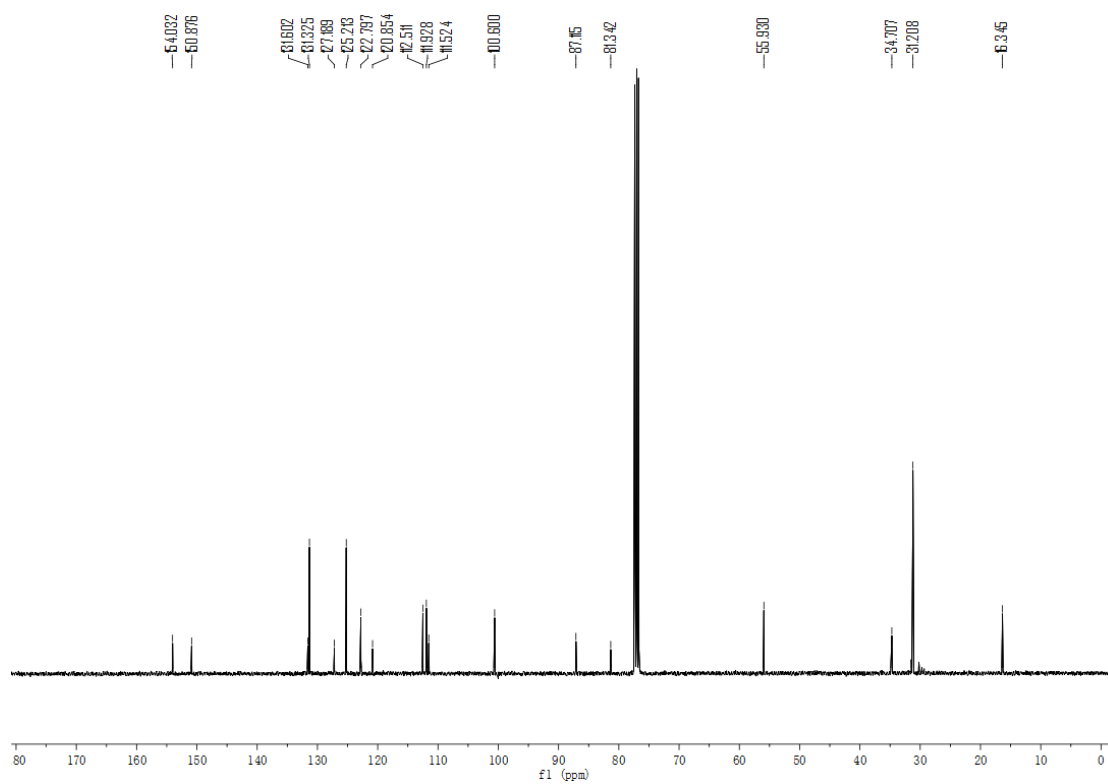


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methoxy-1H-indole (3ac)

^1H NMR (400 MHz, CDCl_3):

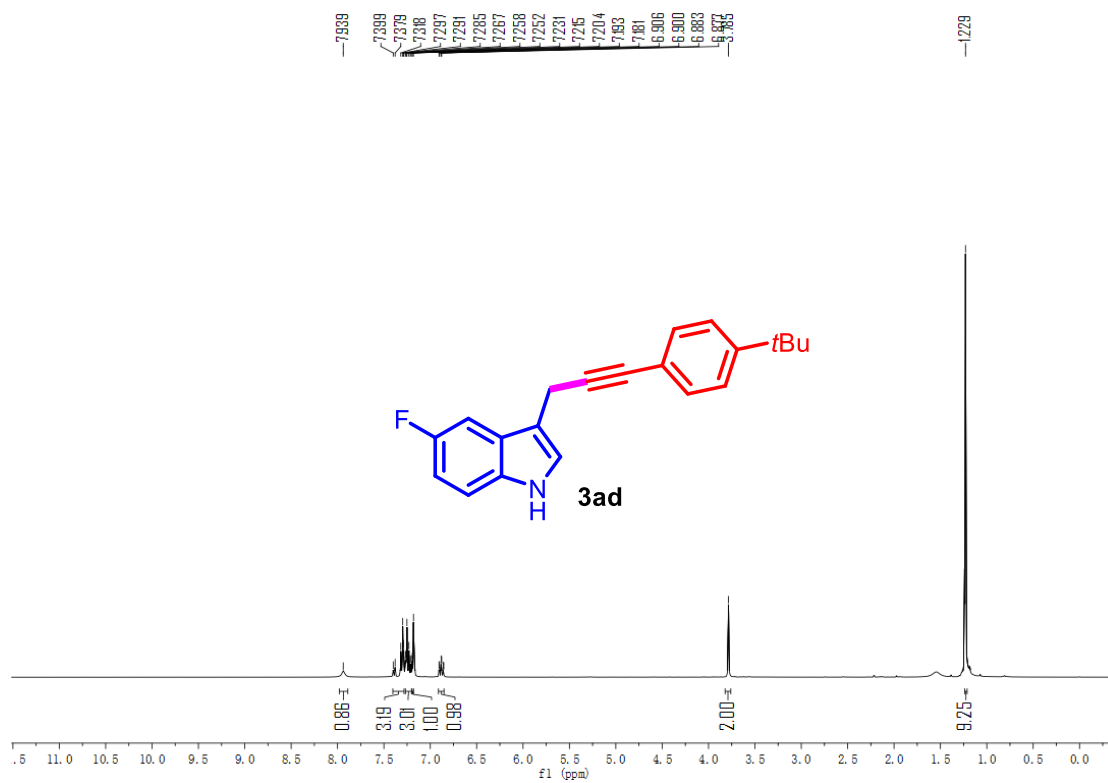


^{13}C NMR (100 MHz, CDCl_3):

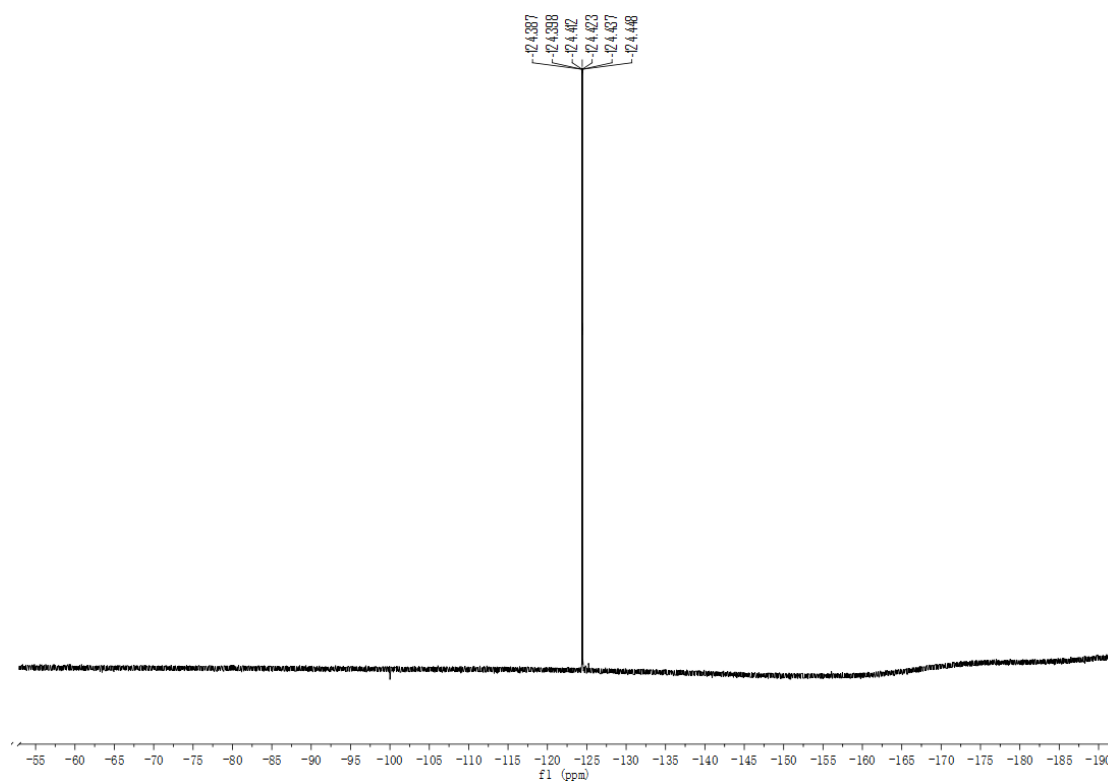


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-fluoro-1H-indole (3ad)

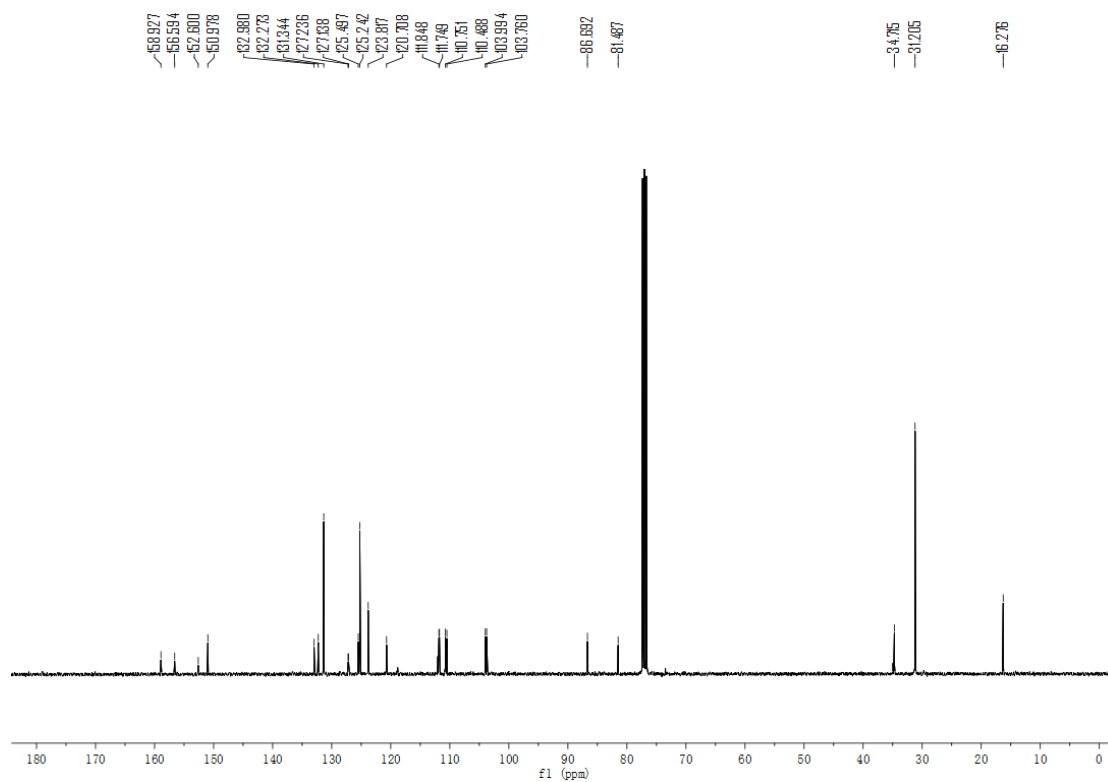
^1H NMR (400 MHz, CDCl_3):



^{19}F NMR (376 MHz, CDCl_3):

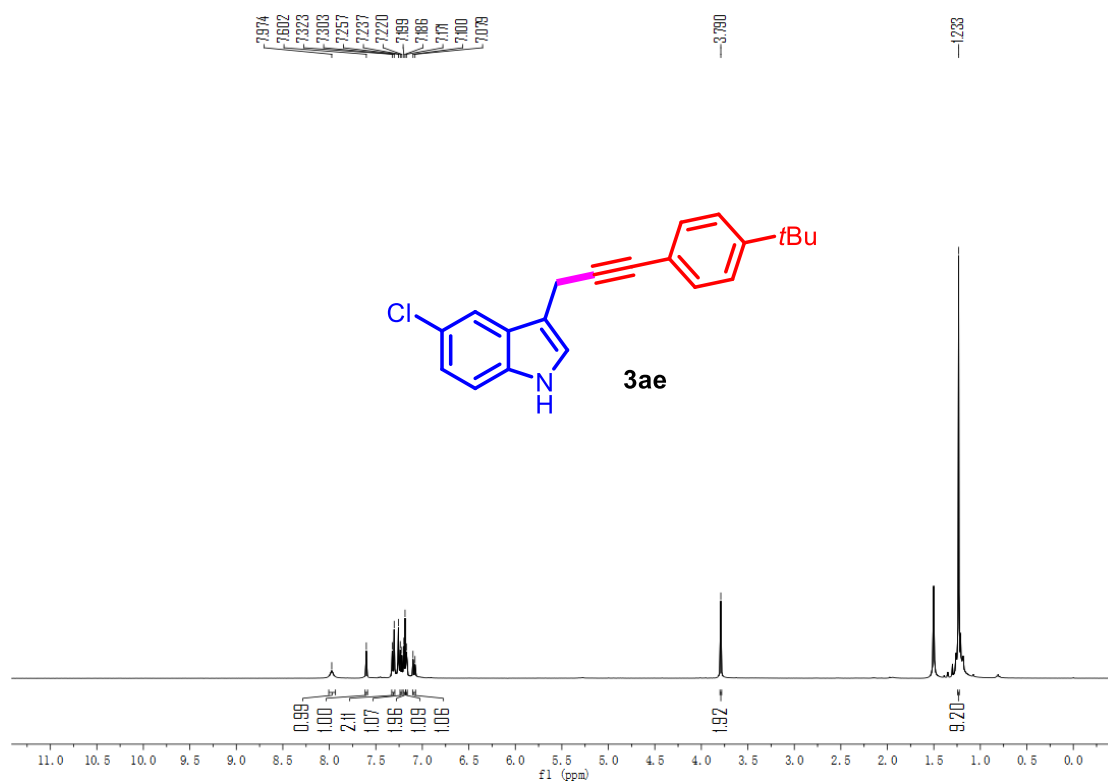


^{13}C NMR (100 MHz, CDCl_3):

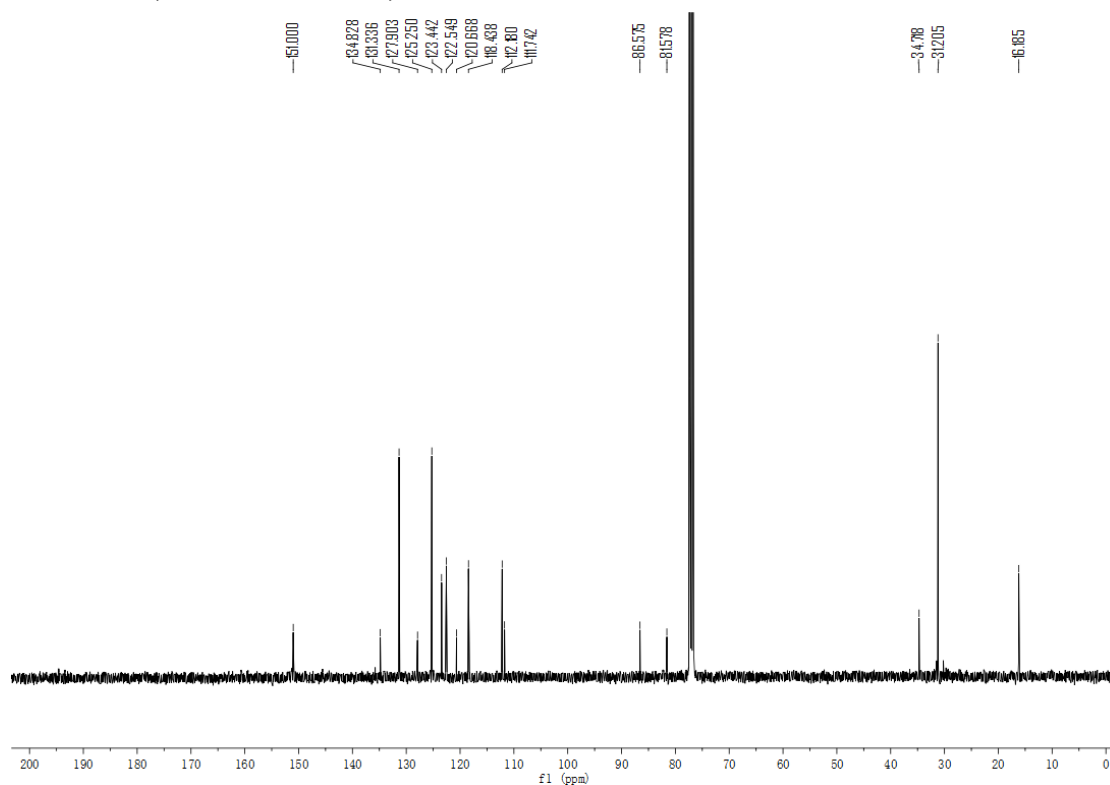


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-chloro-1H-indole (3ae)

^1H NMR (400 MHz, CDCl_3):

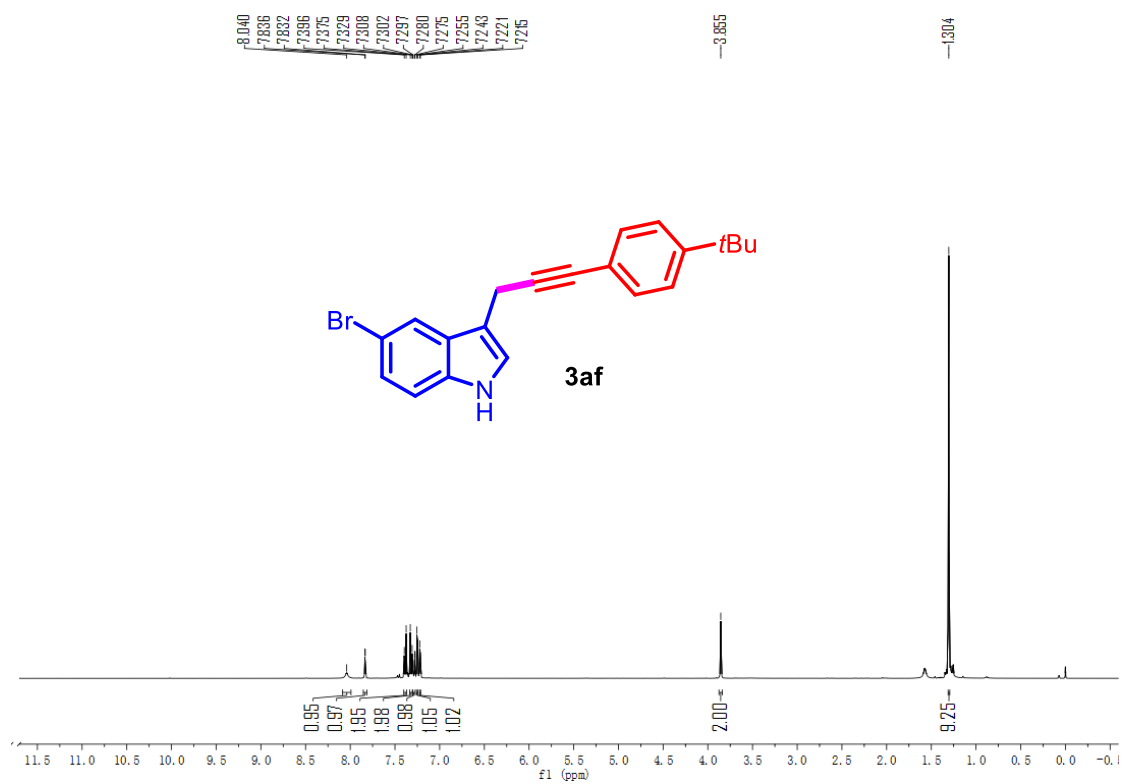


^{13}C NMR (100 MHz, CDCl_3):

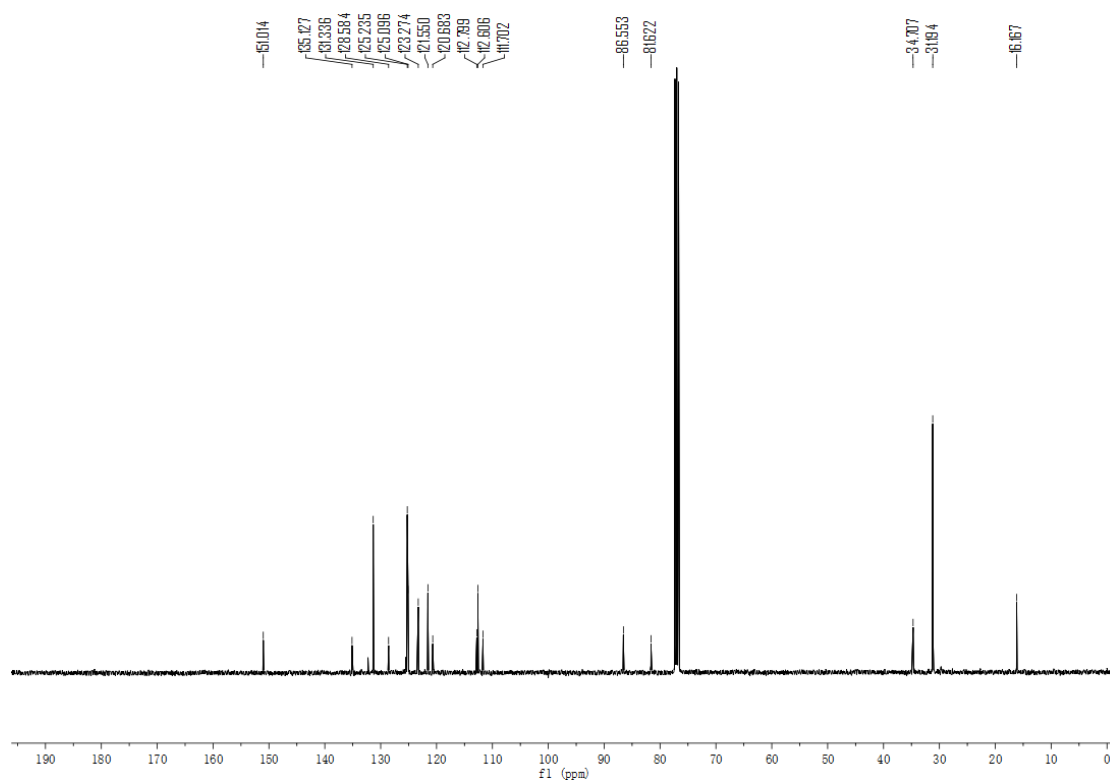


5-bromo-3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1H-indole (3af)

^1H NMR (400 MHz, CDCl_3):

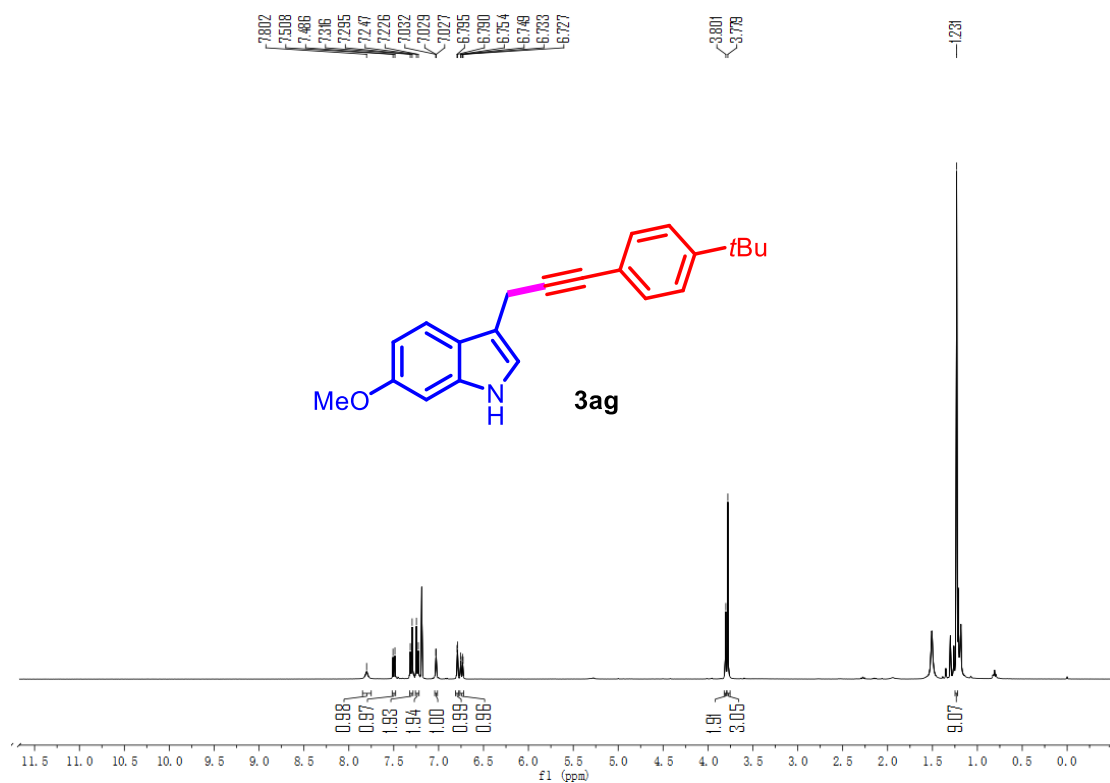


¹³C NMR (100 MHz, CDCl₃):

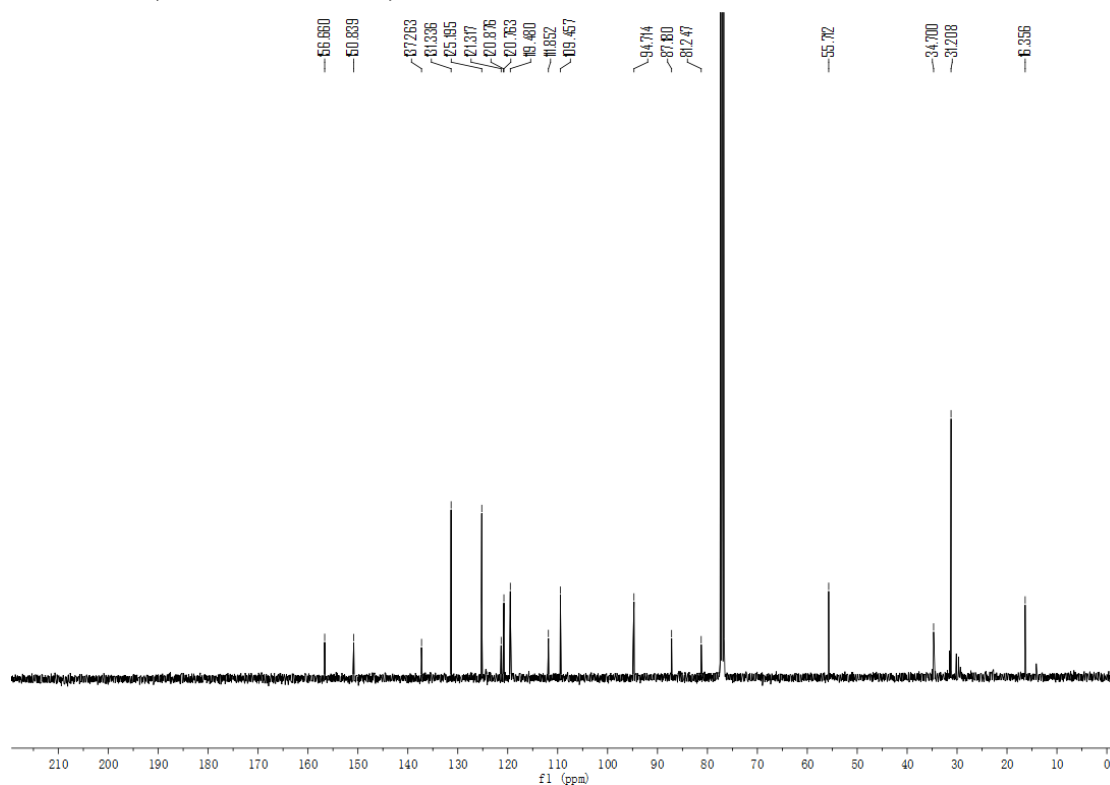


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-6-methoxy-1H-indole (3ag)

¹H NMR (400 MHz, CDCl₃):

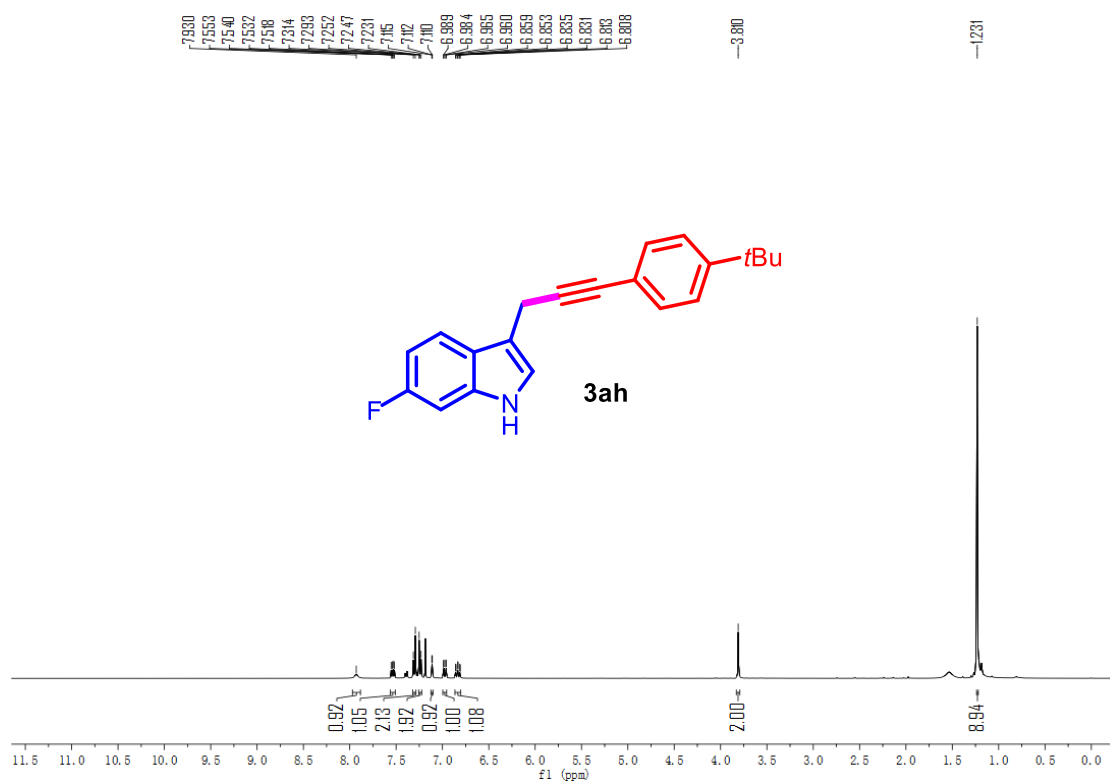


¹³C NMR (100 MHz, CDCl₃):

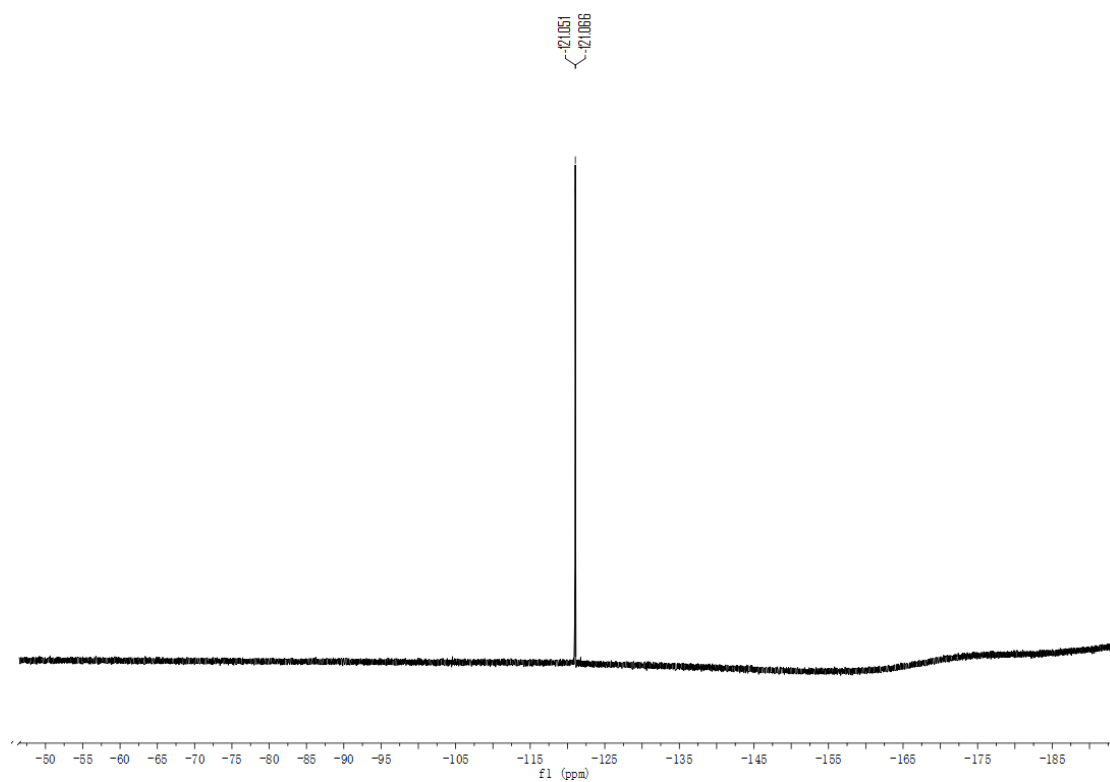


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-6-fluoro-1H-indole (3ah)

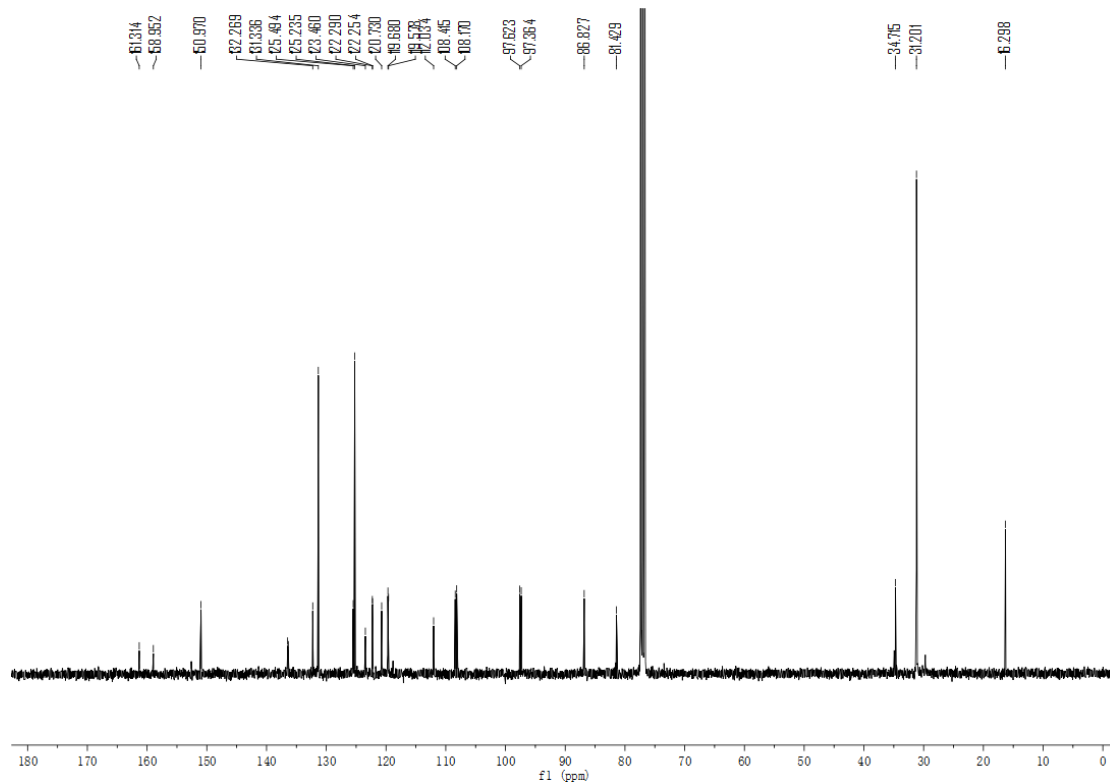
¹H NMR (400 MHz, CDCl₃):



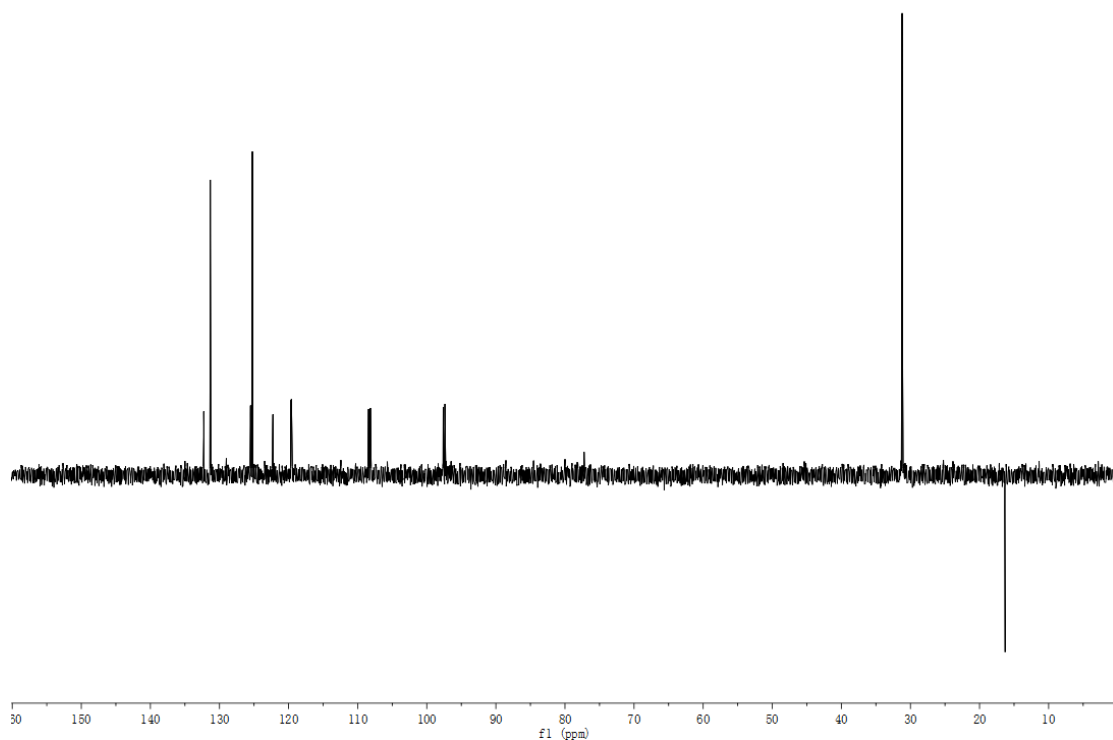
¹⁹F NMR (376 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

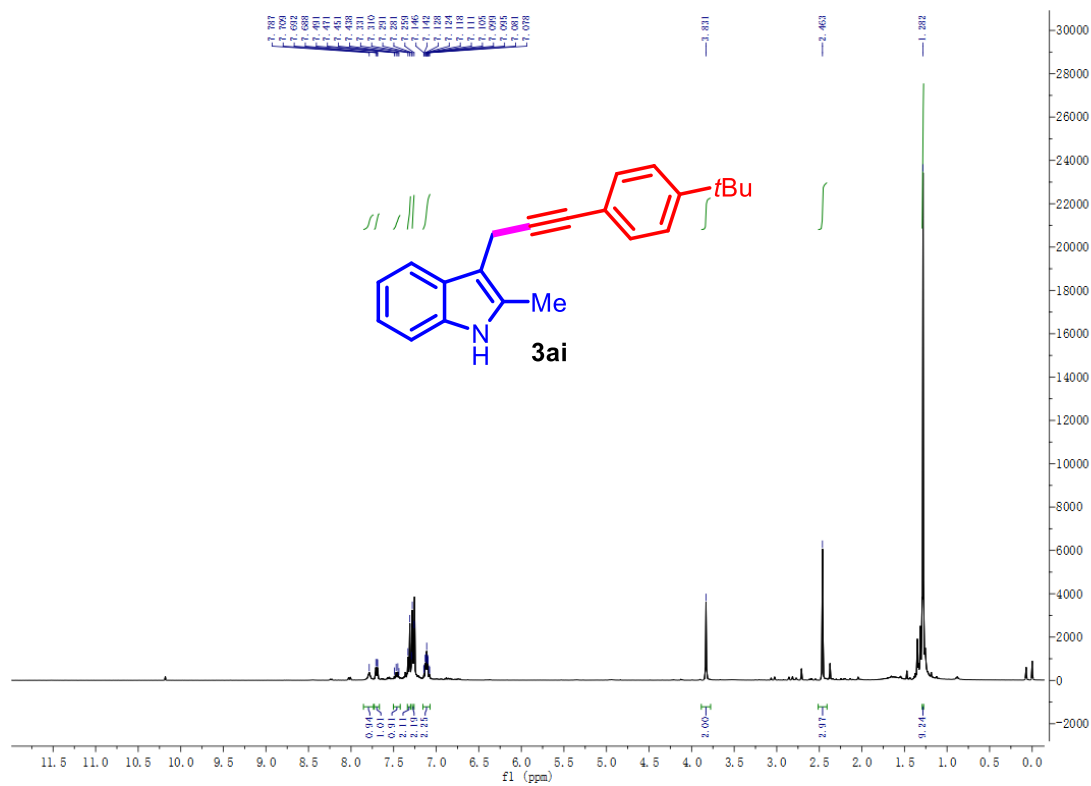


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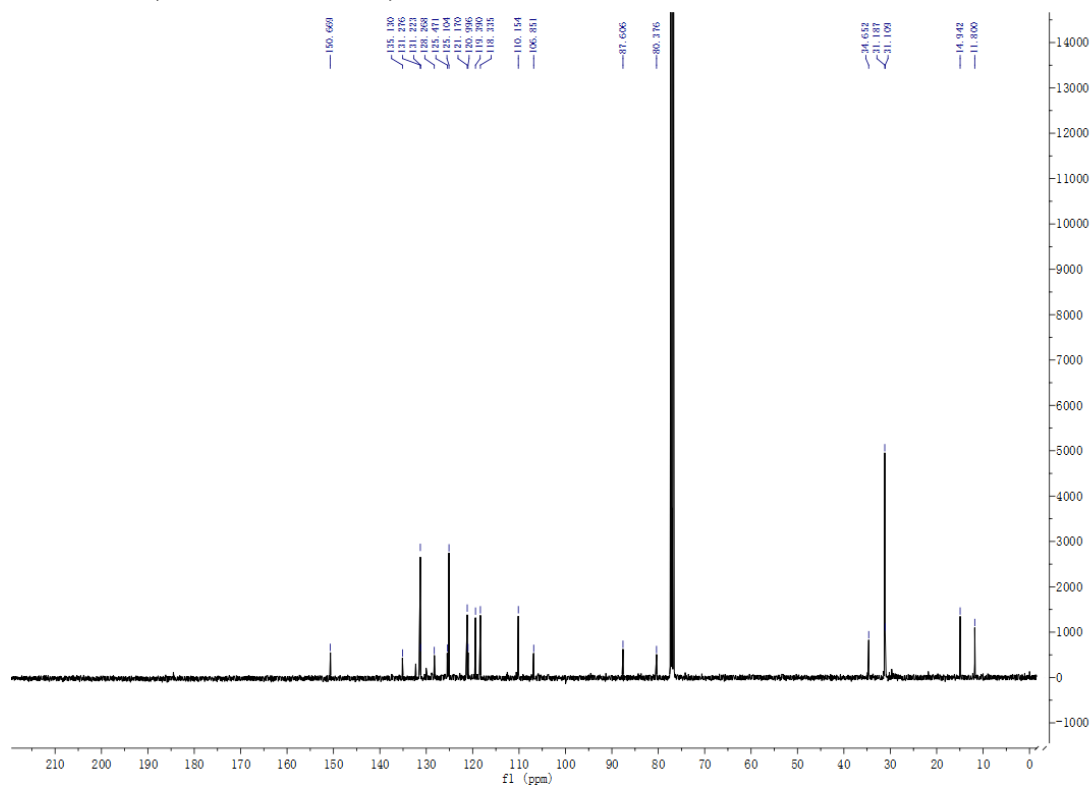


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-2-methyl-1H-indole (3ai)

¹H NMR (400 MHz, CDCl₃):

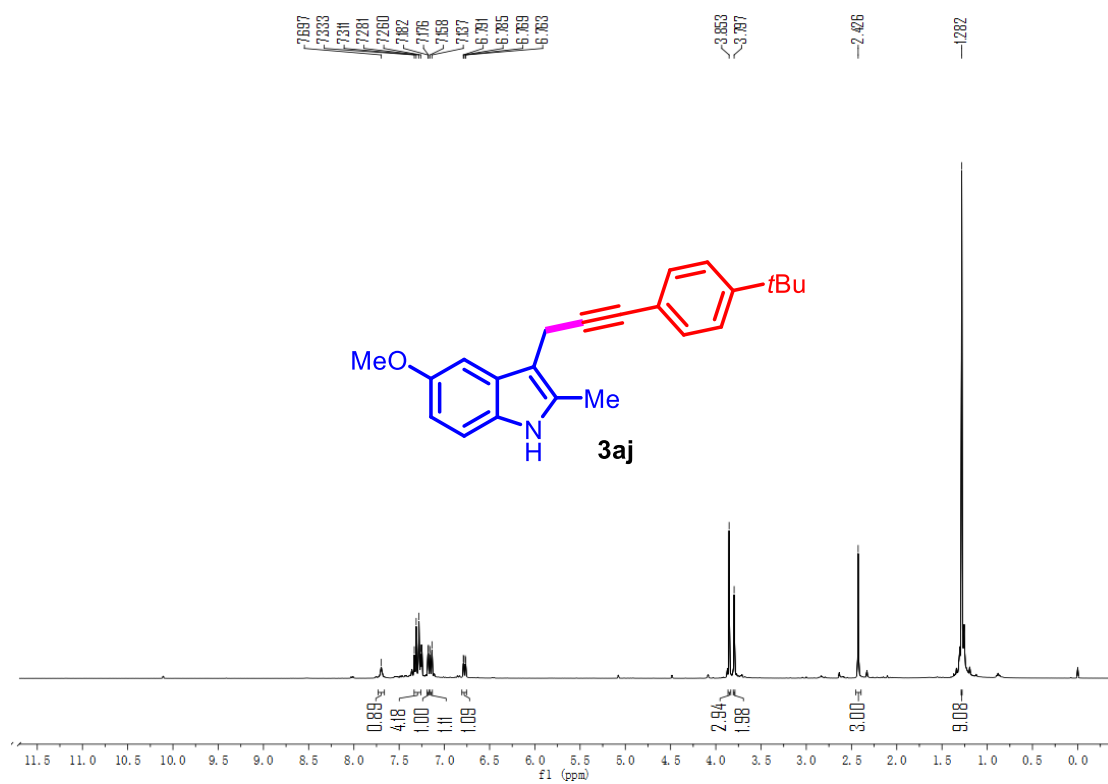


¹³C NMR (100 MHz, CDCl₃):

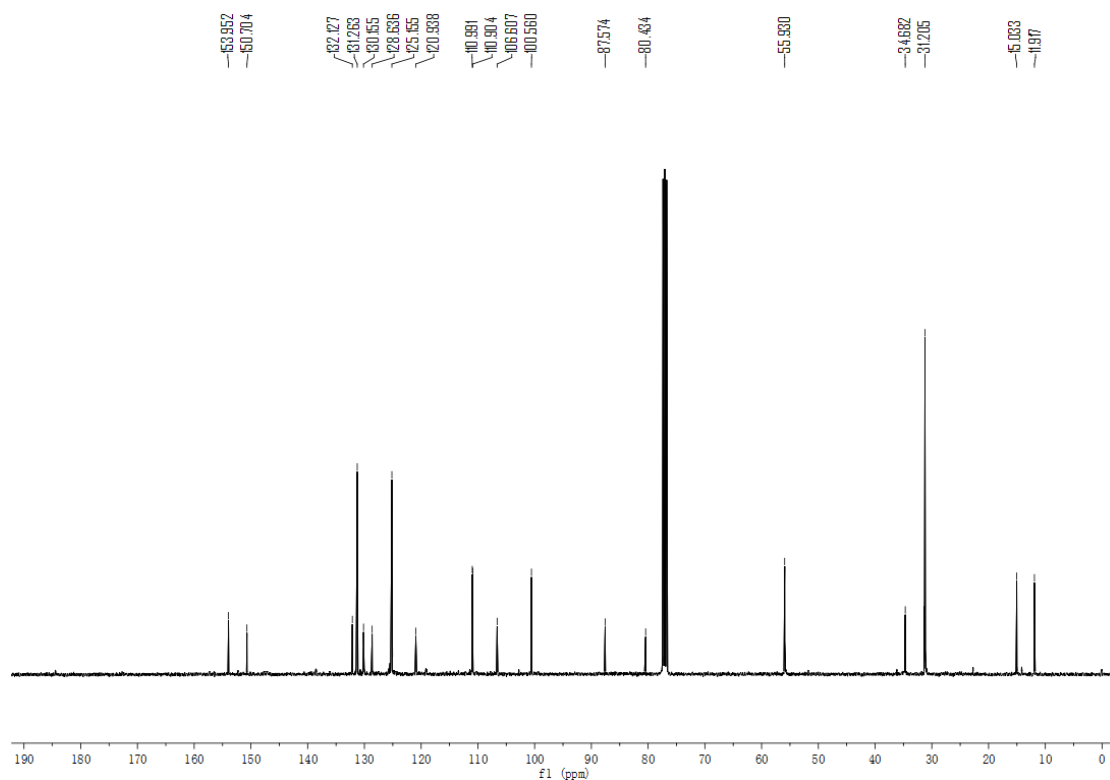


3-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-5-methoxy-2-methyl-1H-indole (3aj)

¹H NMR (400 MHz, CDCl₃):

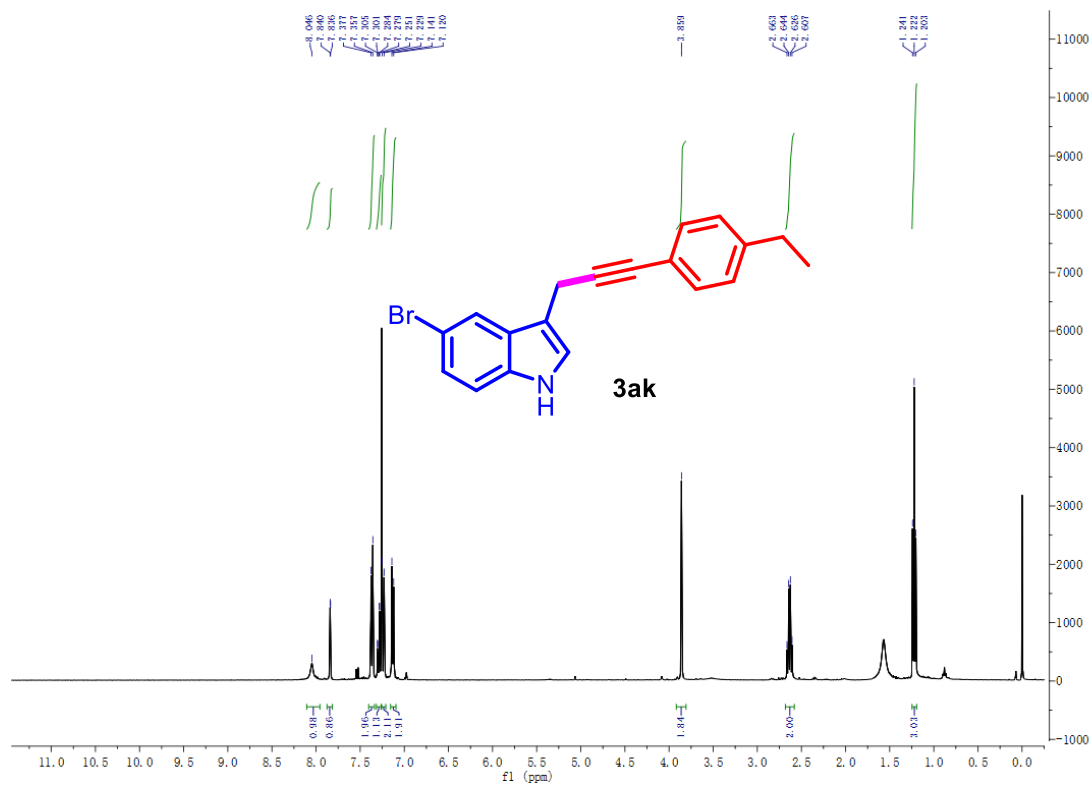


¹³C NMR (100 MHz, CDCl₃):

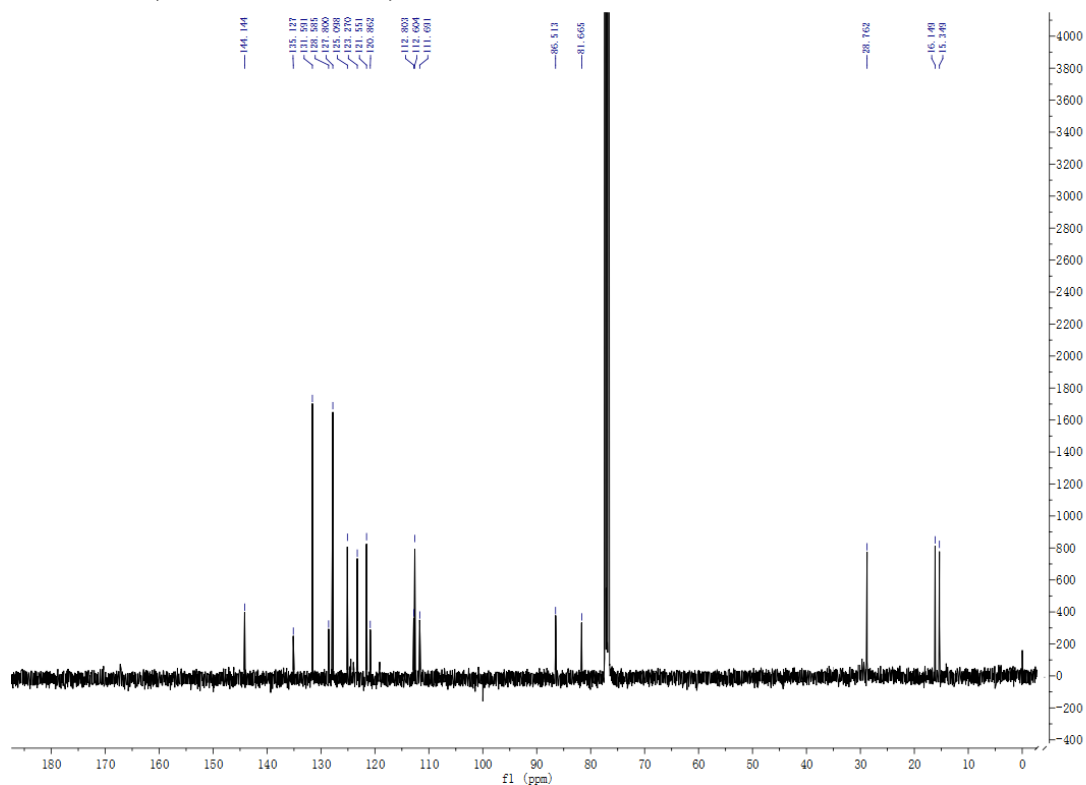


5-bromo-3-(3-(4-ethylphenyl)prop-2-yn-1-yl)-1H-indole (3ak)

¹H NMR (400 MHz, CDCl₃):

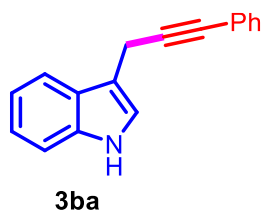
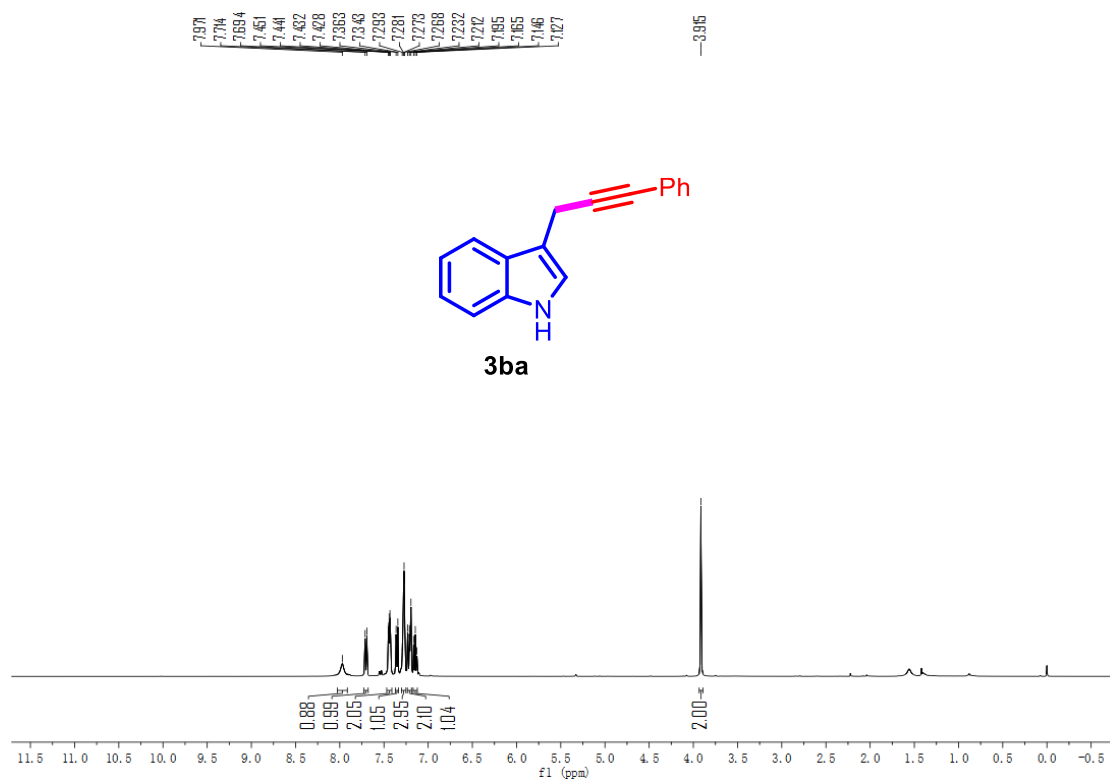


¹³C NMR (100 MHz, CDCl₃):

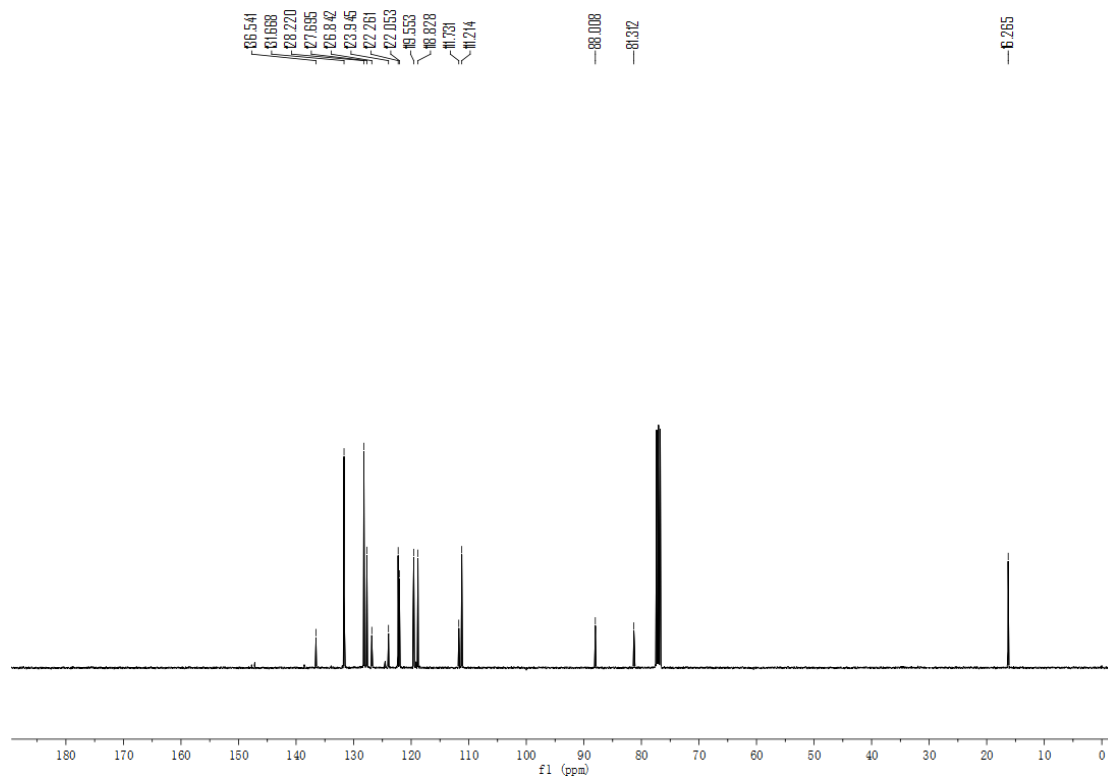


3-(3-phenylprop-2-yn-1-yl)-1H-indole (3ba)

¹H NMR (400 MHz, CDCl₃):

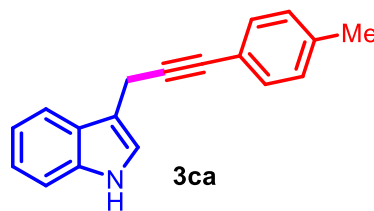
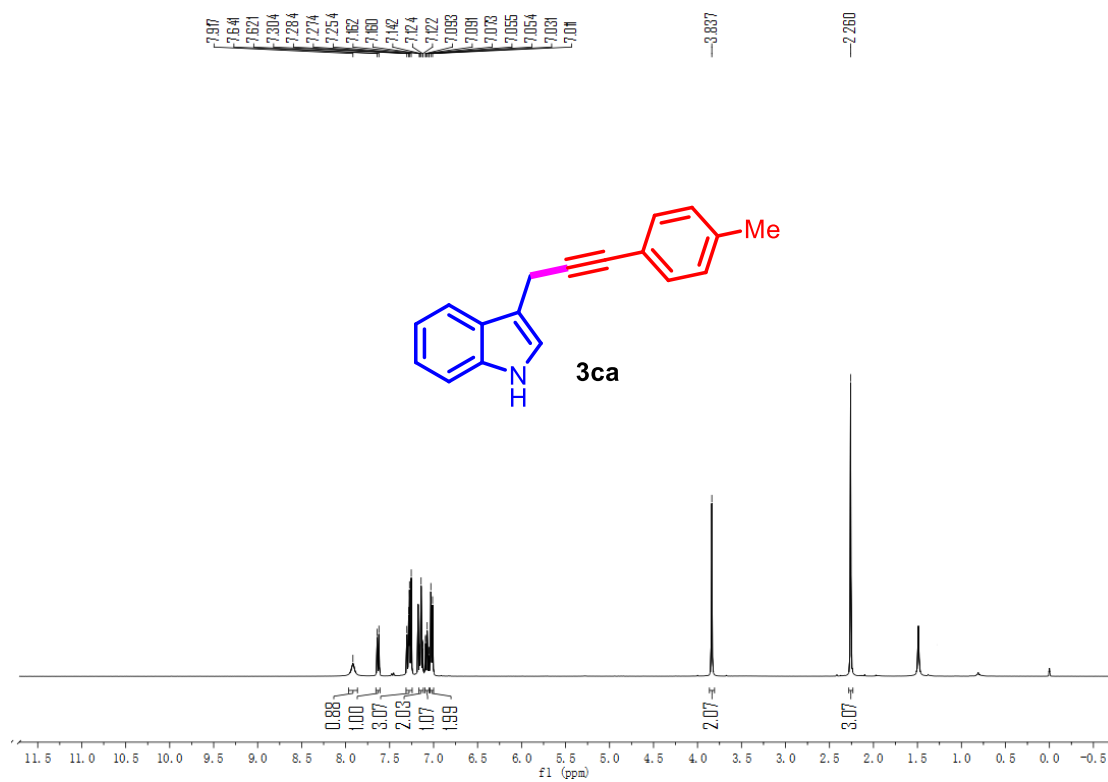


^{13}C NMR (100 MHz, CDCl_3):

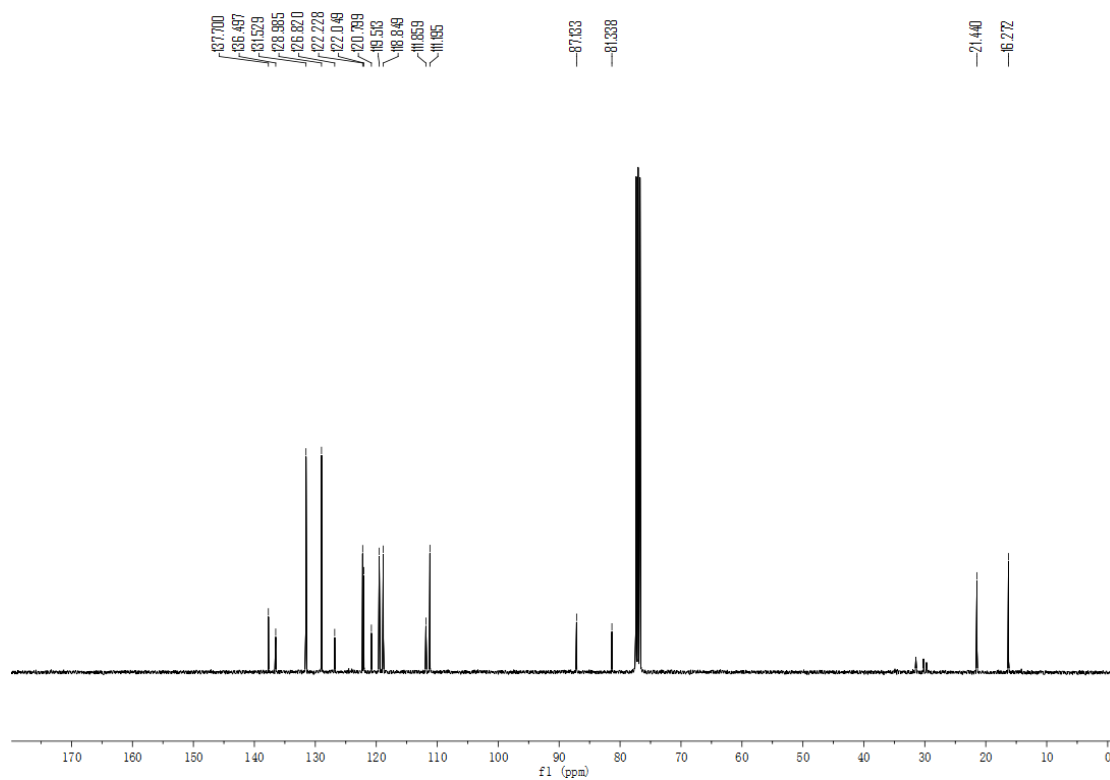


3-(3-(p-tolyl)prop-2-yn-1-yl)-1H-indole (3ca)

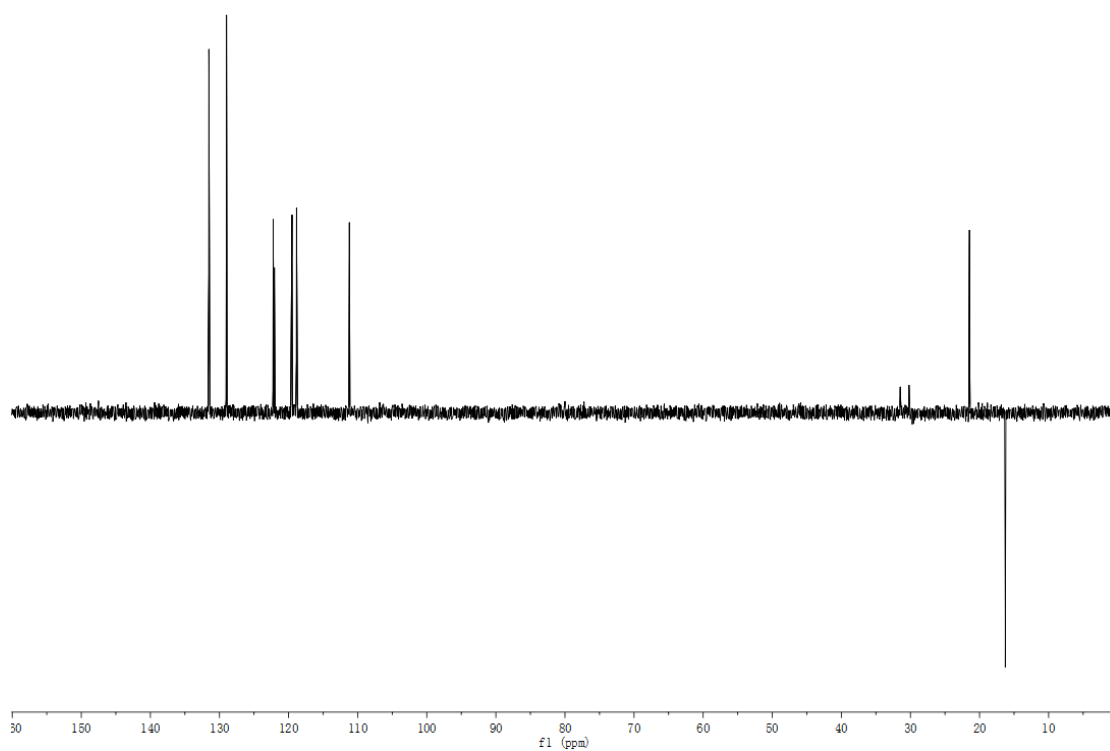
^1H NMR (400 MHz, CDCl_3):



¹³C NMR (100 MHz, CDCl₃):

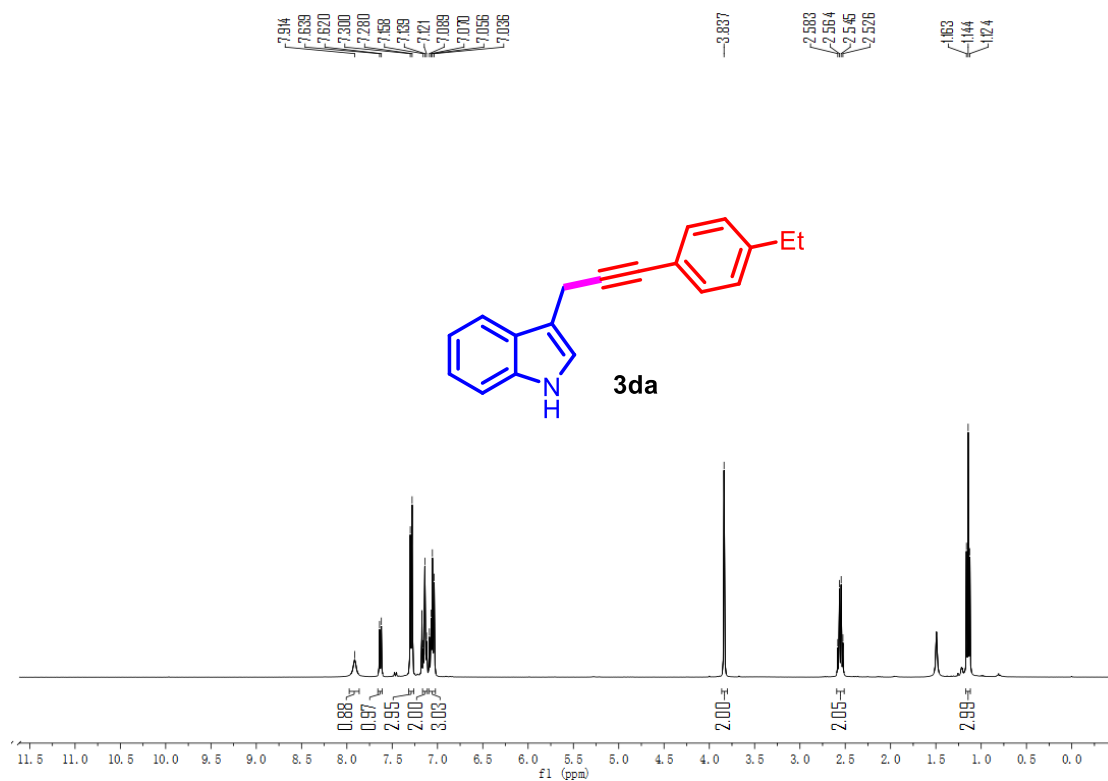


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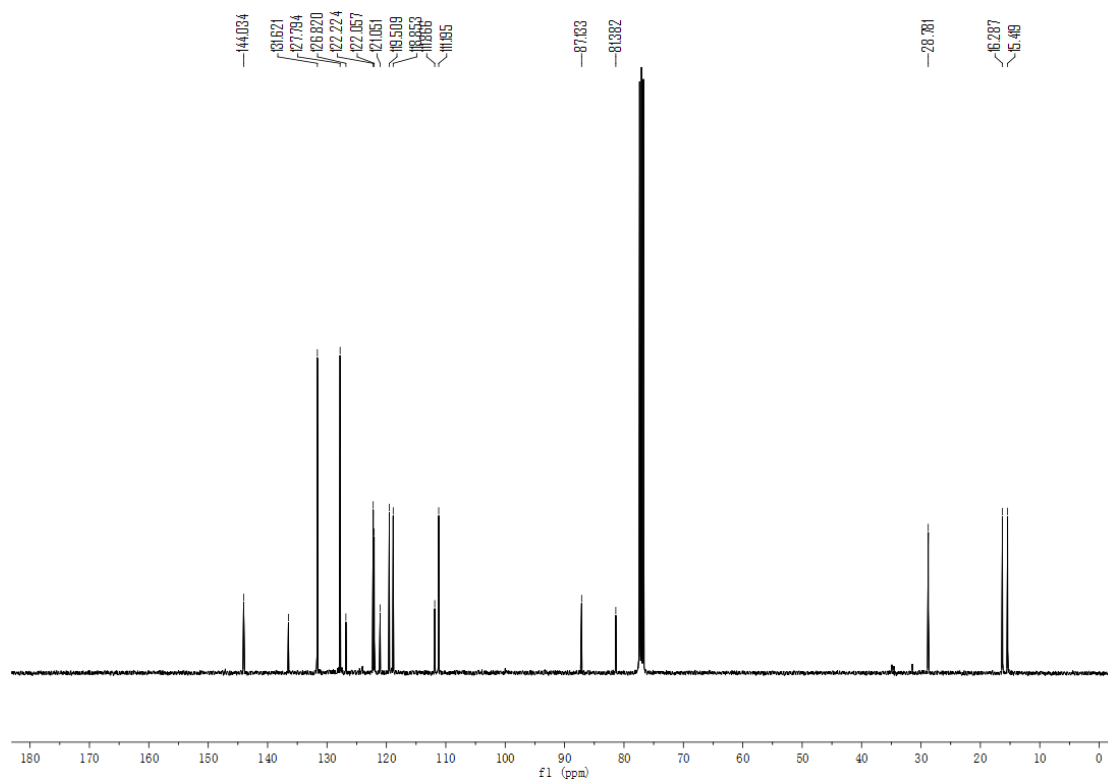


3-(3-(4-ethylphenyl)prop-2-yn-1-yl)-1H-indole (3da)

^1H NMR (400 MHz, CDCl_3):

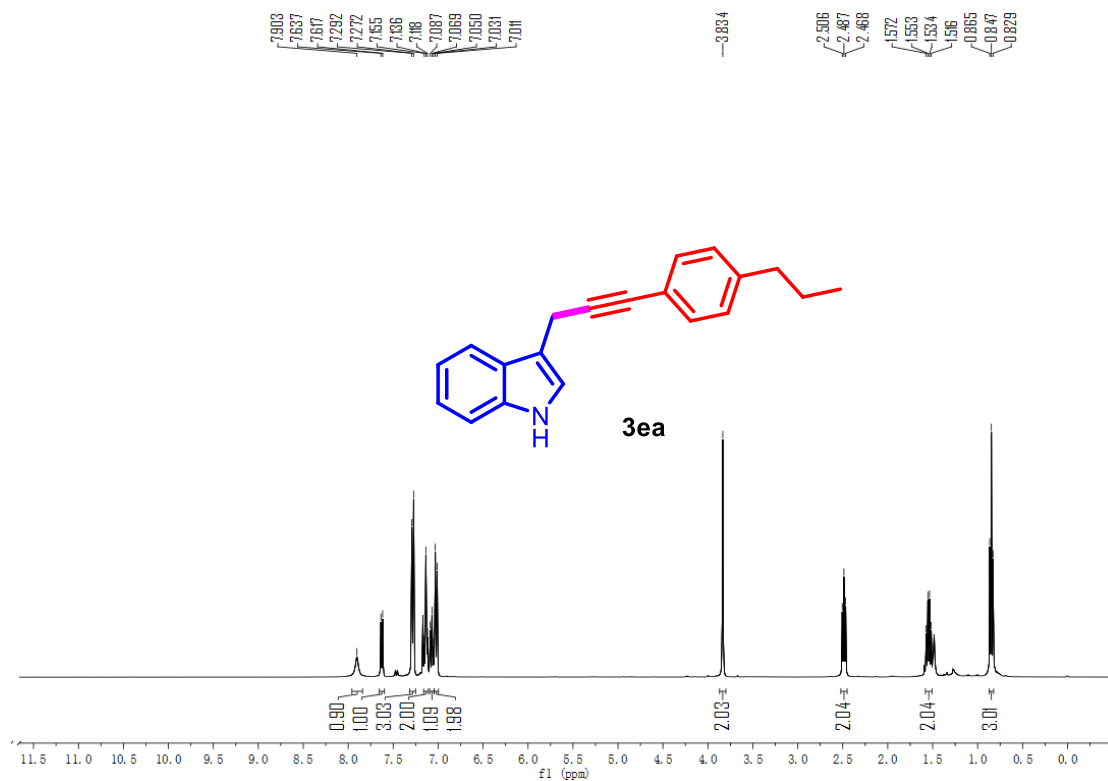


^{13}C NMR (100 MHz, CDCl_3):

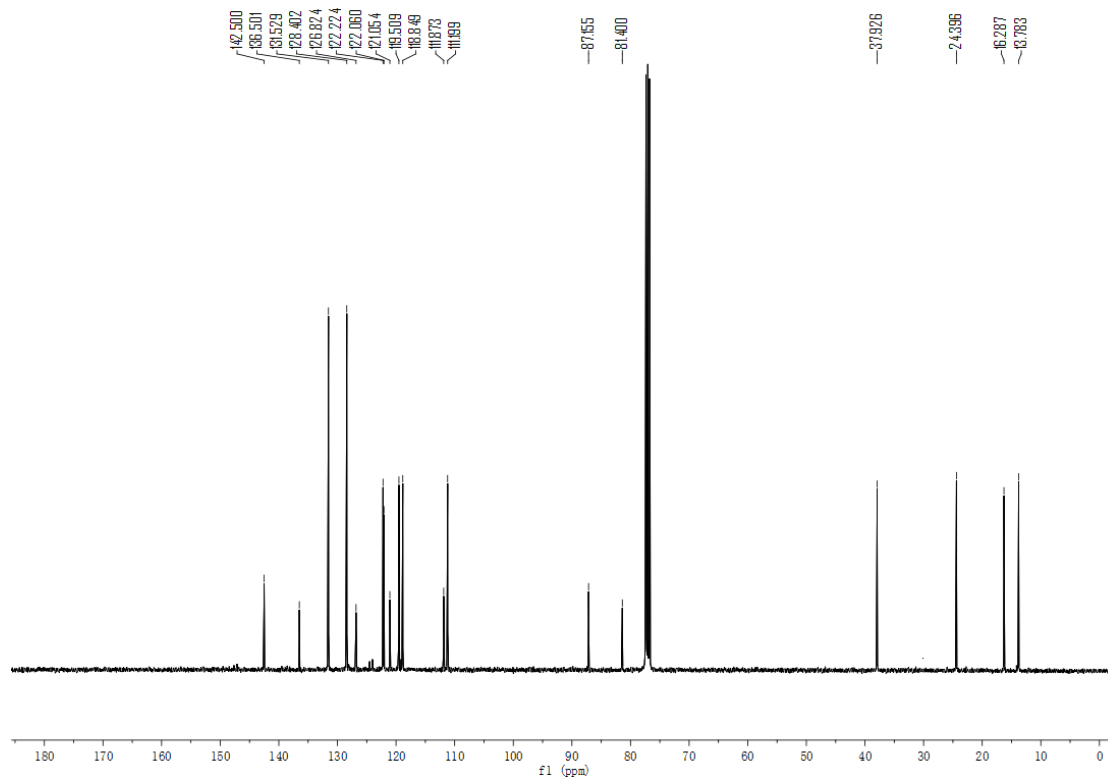


3-(3-(4-propylphenyl)prop-2-yn-1-yl)-1H-indole (3ea)

¹H NMR (400 MHz, CDCl₃):

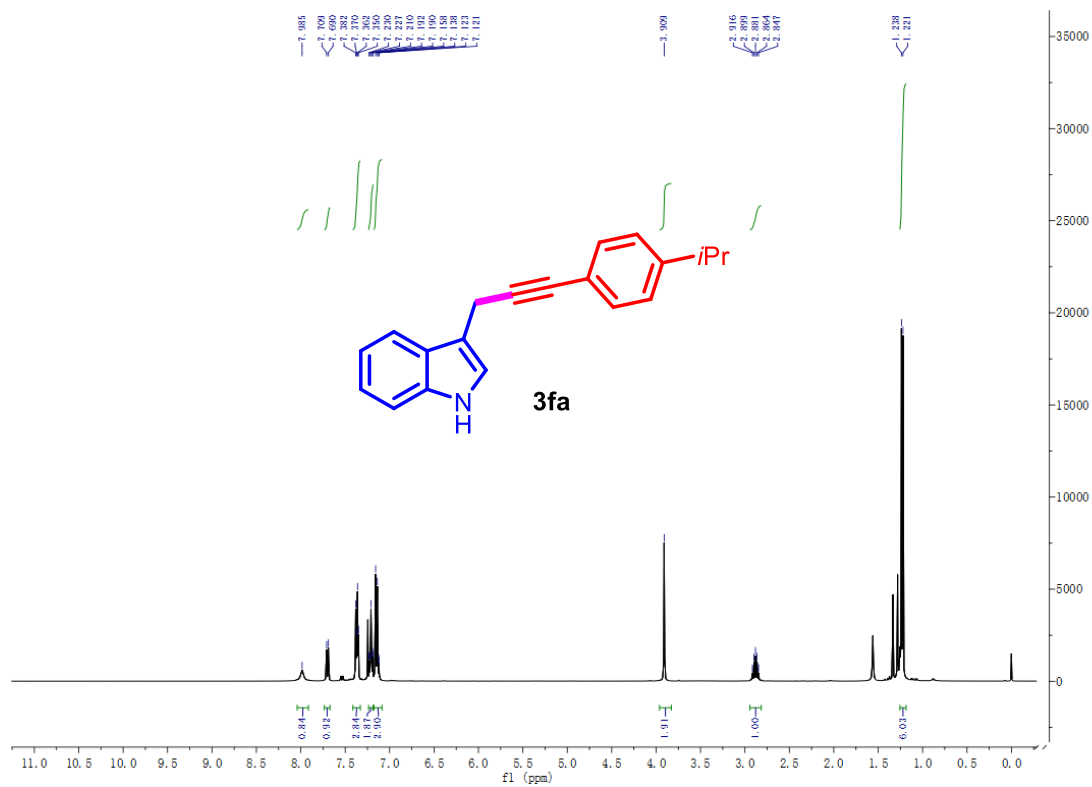


¹³C NMR (100 MHz, CDCl₃):

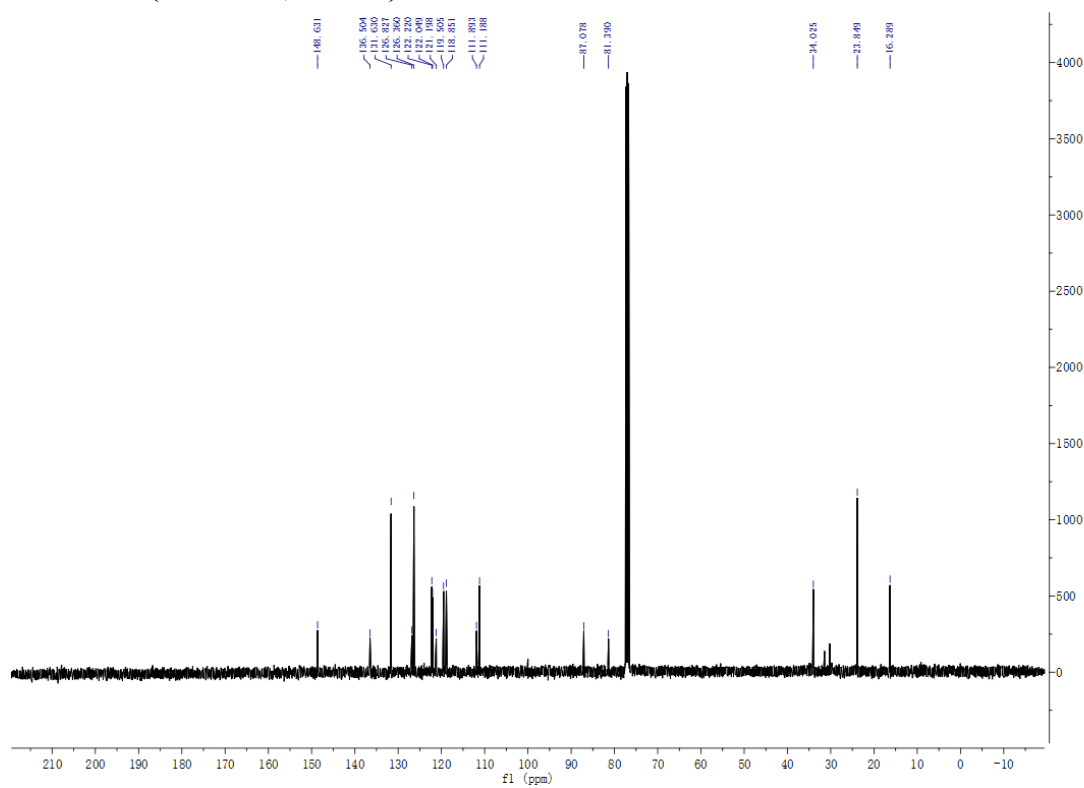


3-(3-(4-isopropylphenyl)prop-2-yn-1-yl)-1H-indole (3fa)

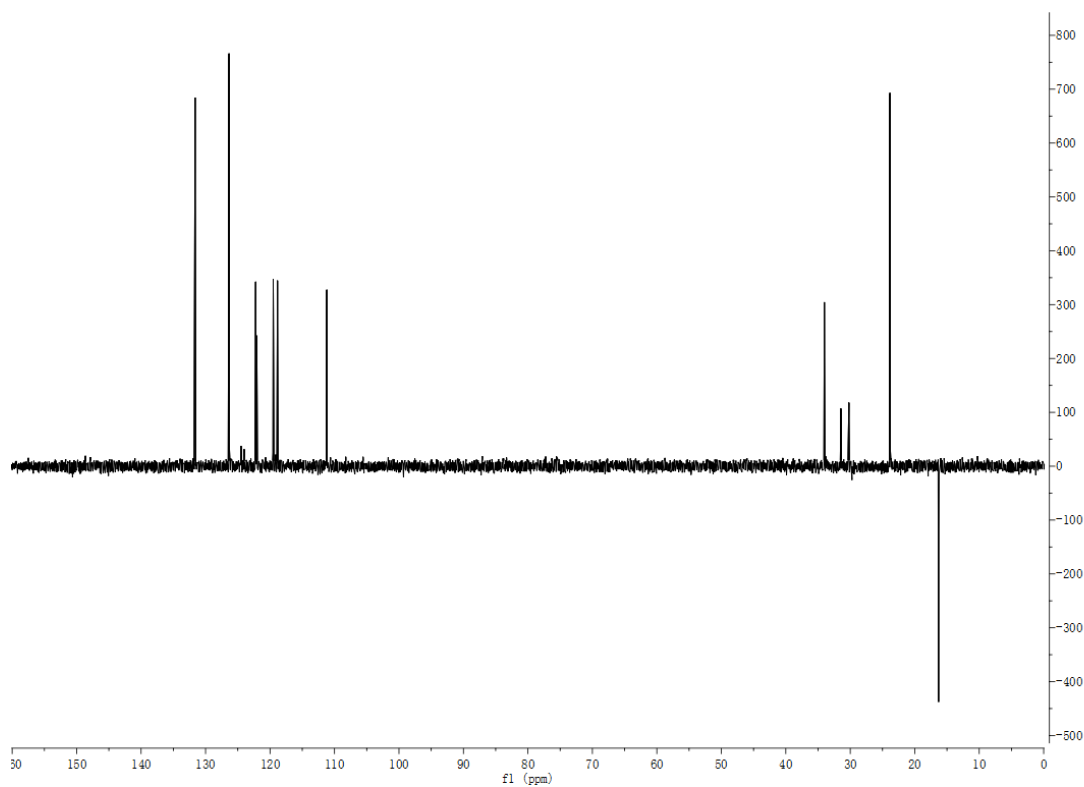
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

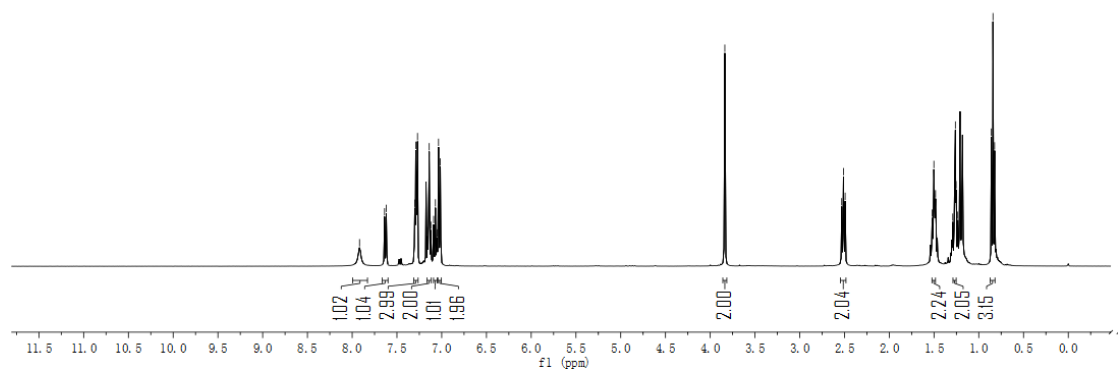
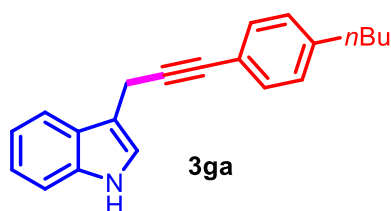


DEPT

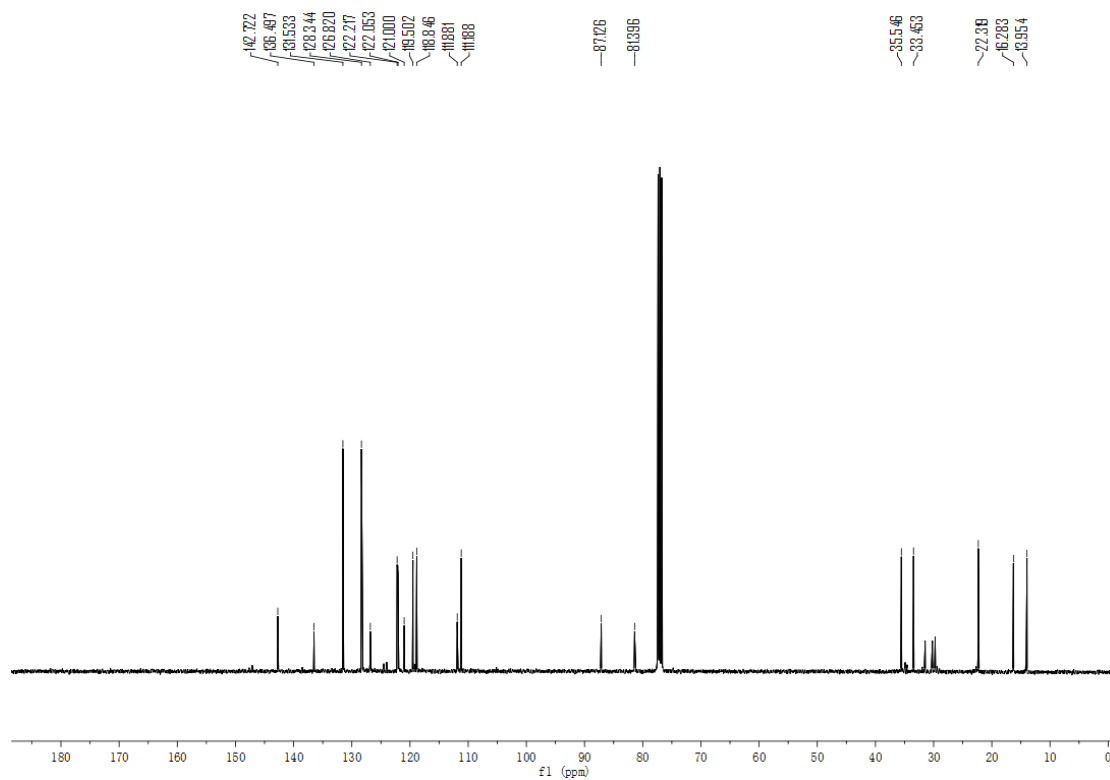


3-(3-(4-butylphenyl)prop-2-yn-1-yl)-1H-indole (3ga)

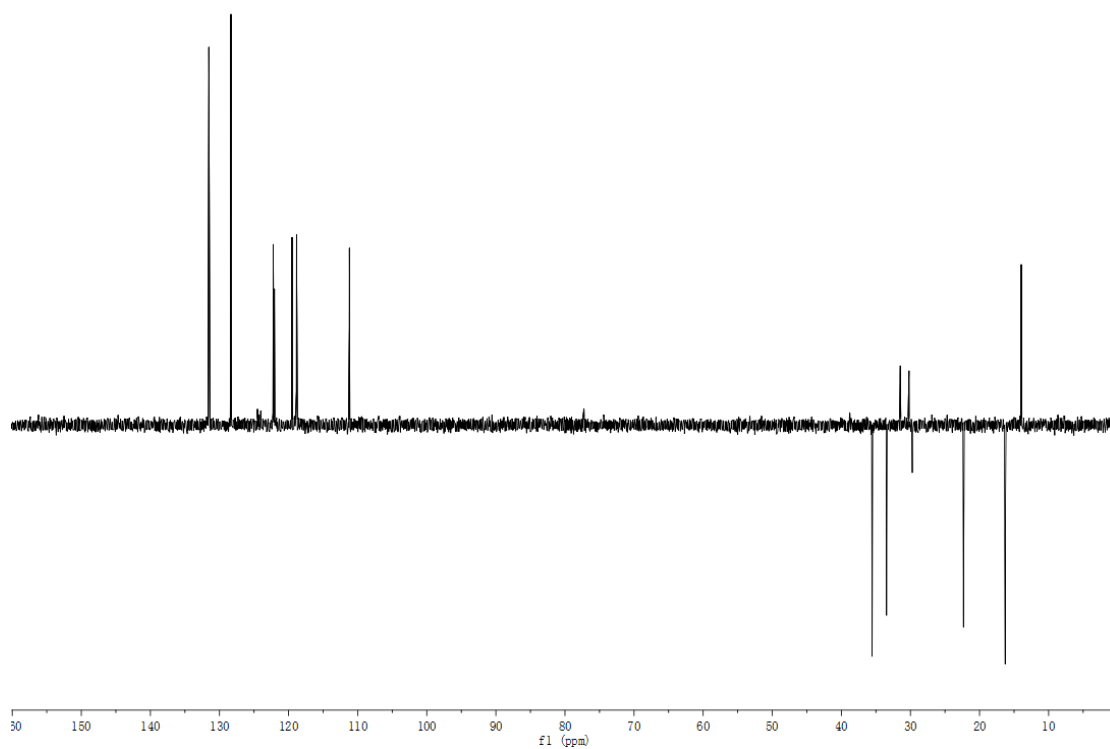
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

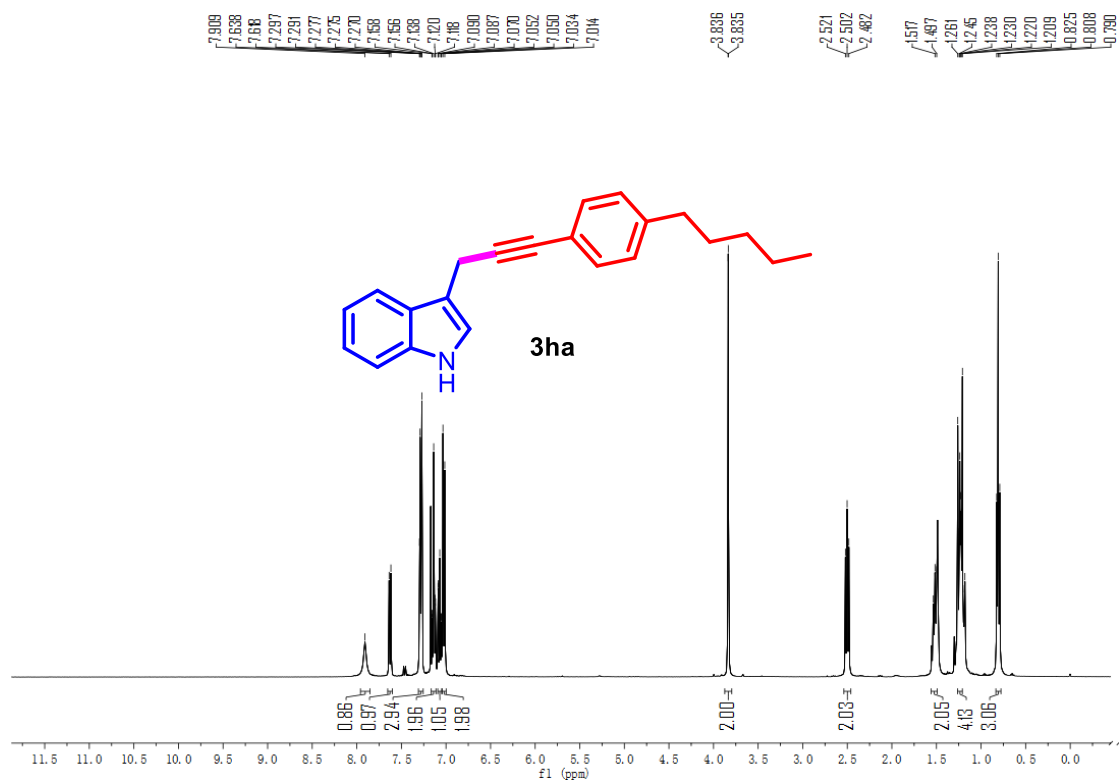


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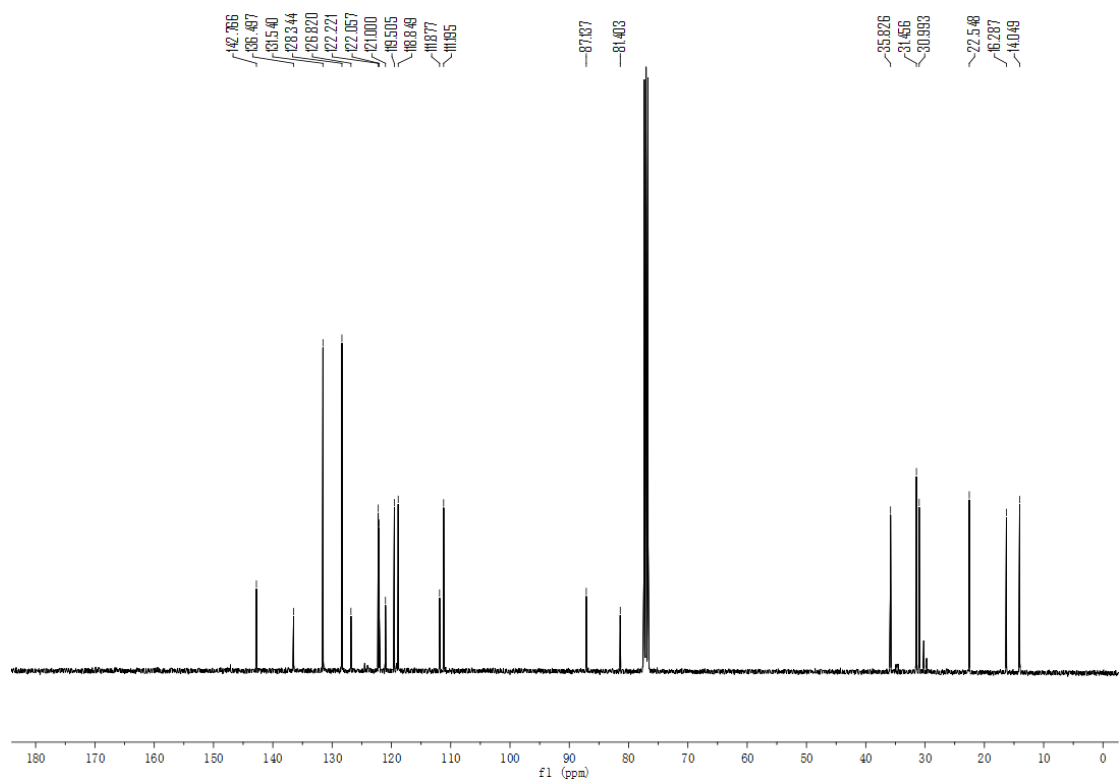


3-(3-(4-pentylphenyl)prop-2-yn-1-yl)-1H-indole (3ha)

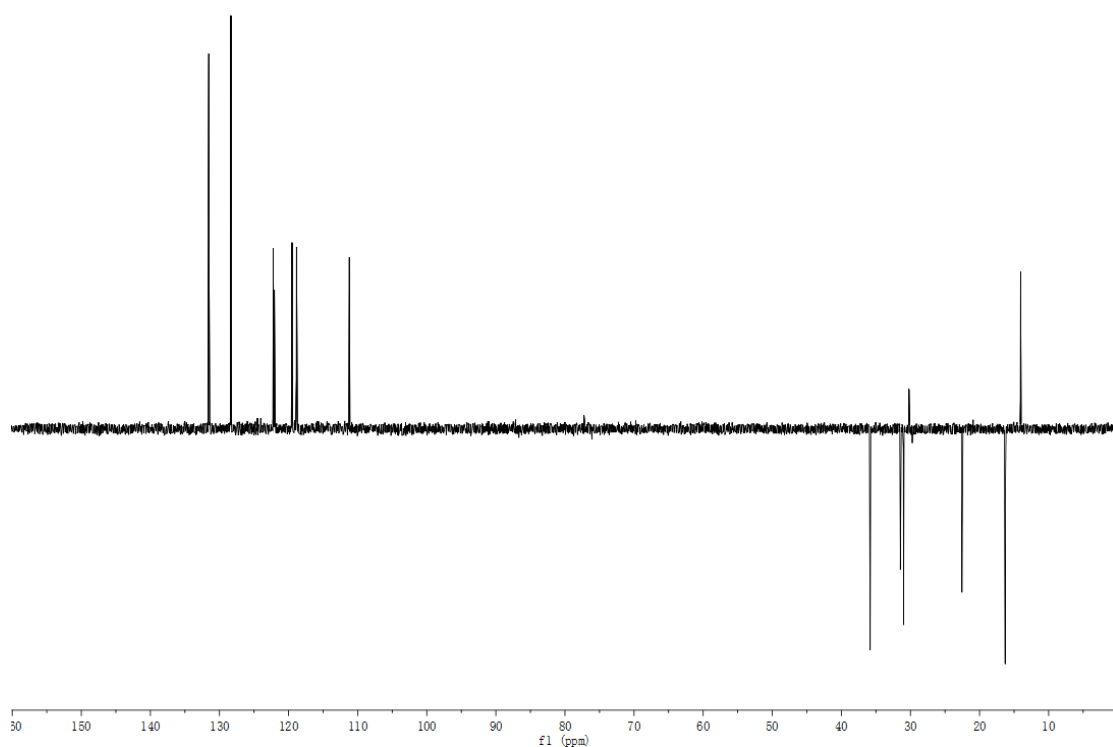
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

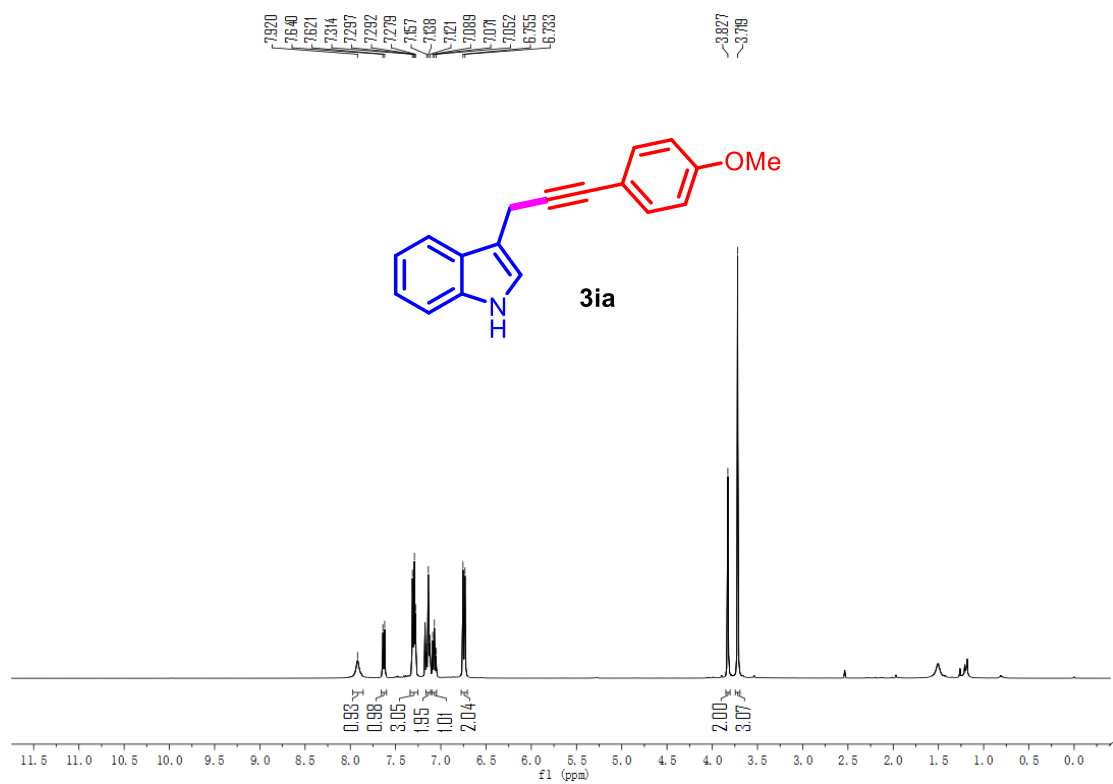


DEPT

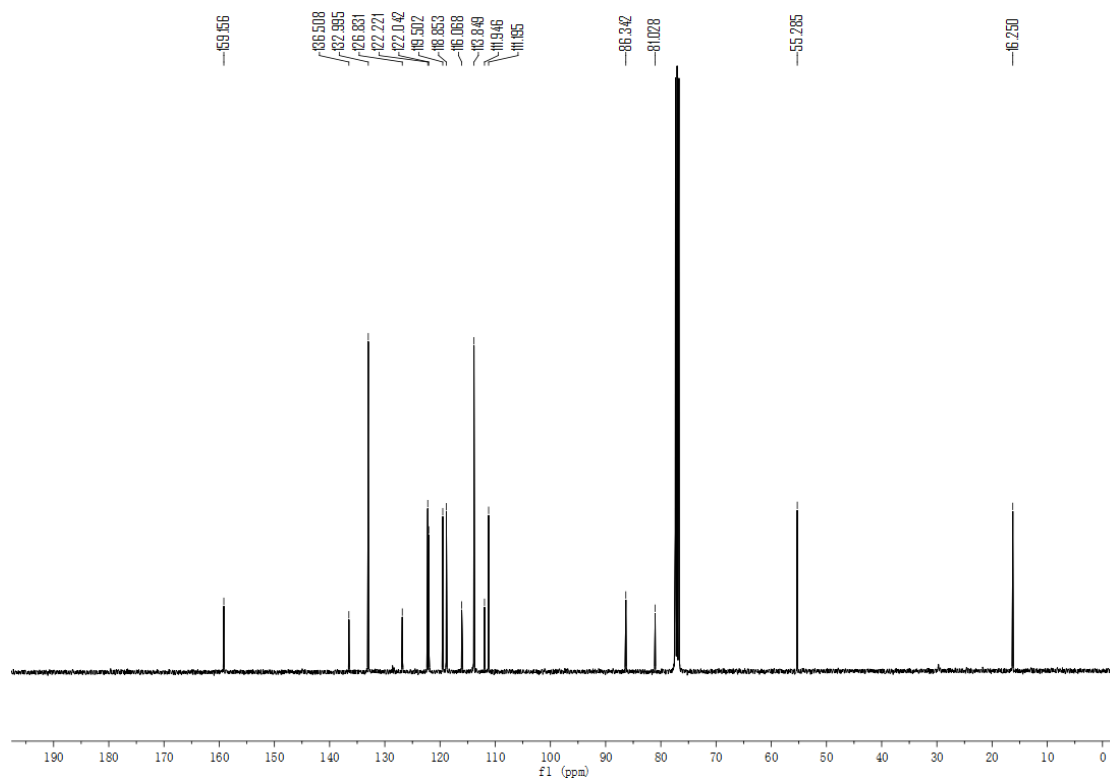


3-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ia)

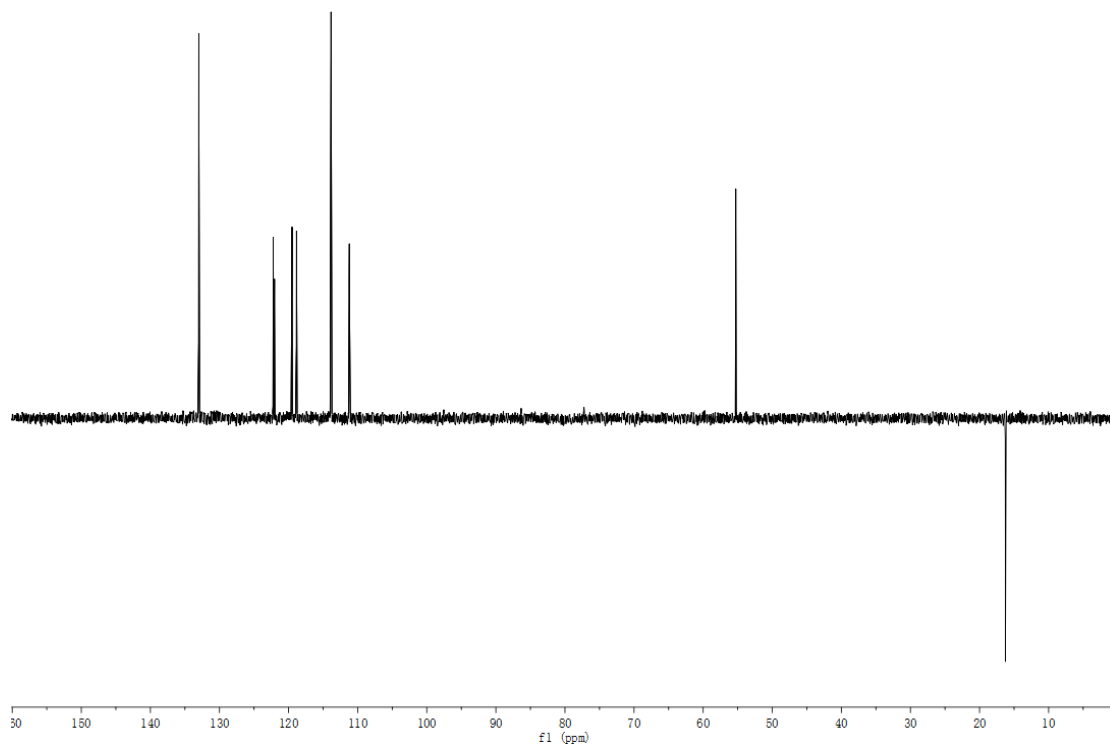
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

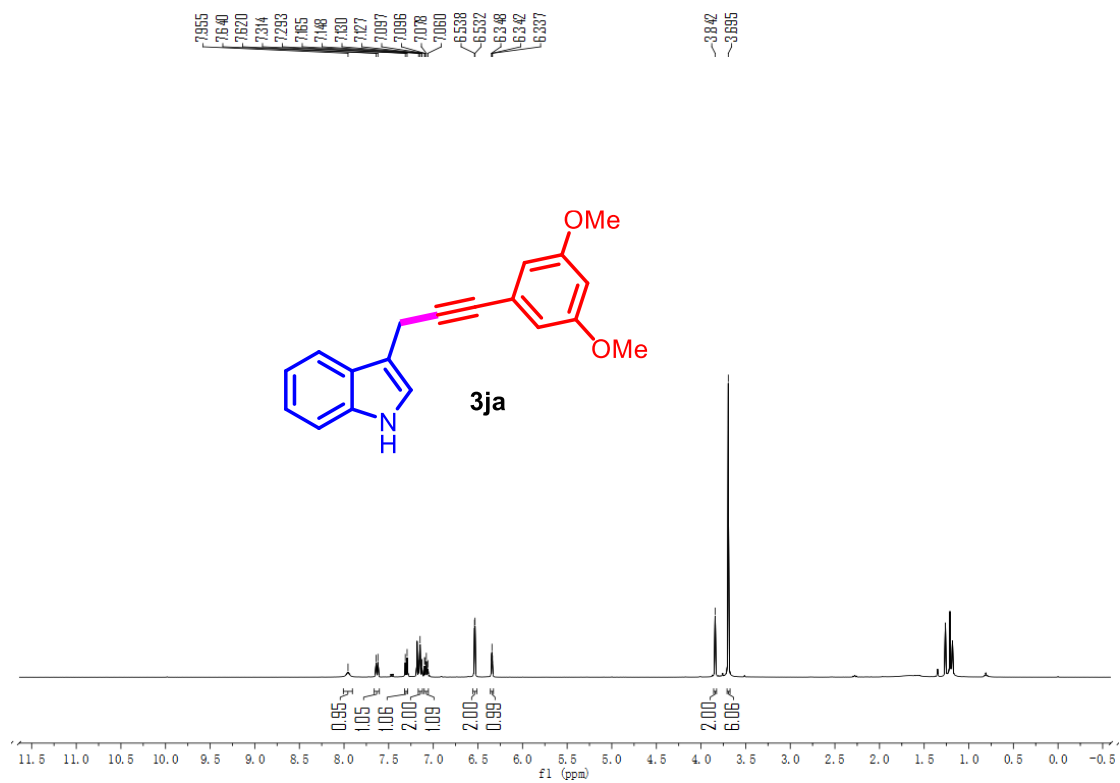


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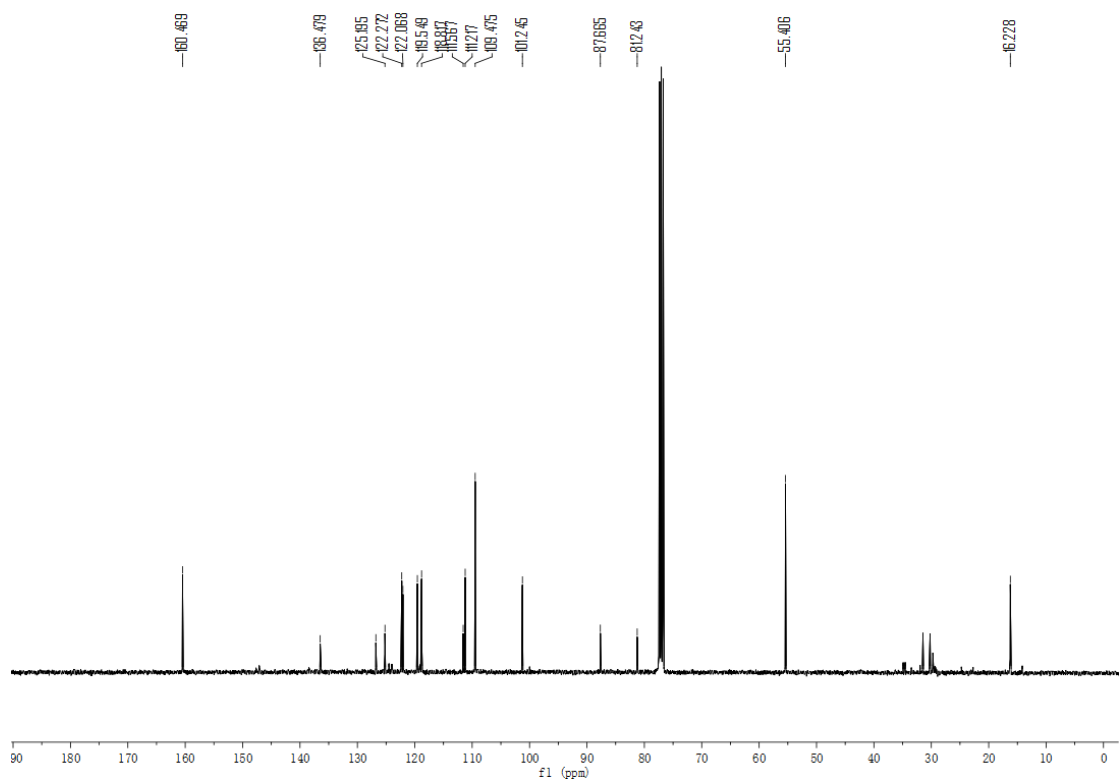


3-(3-(3,5-dimethoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ja)

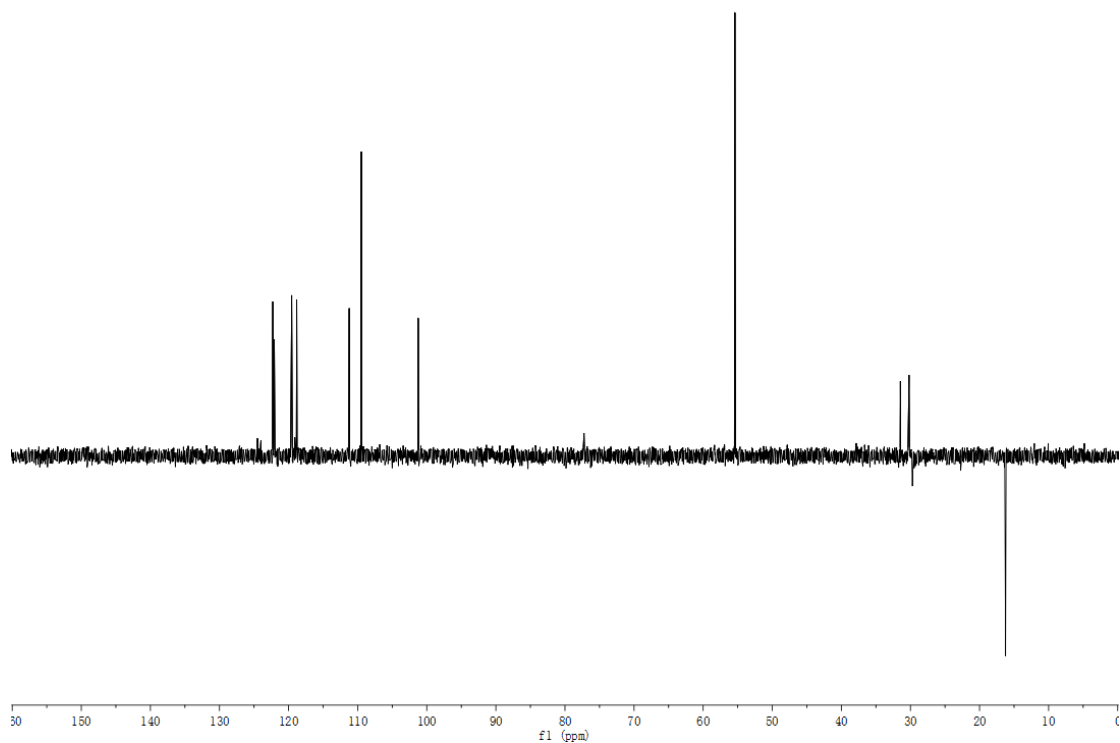
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):



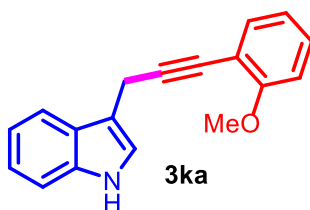
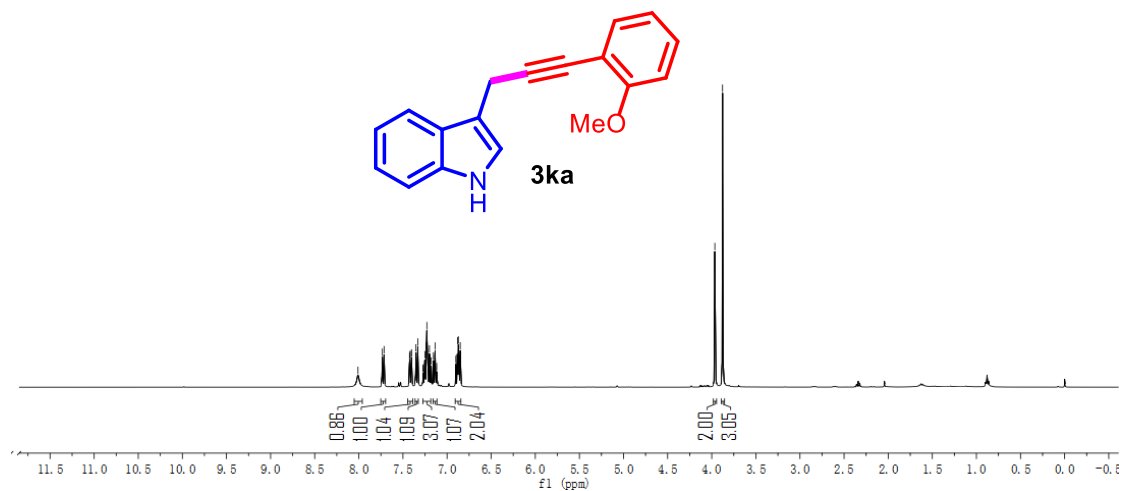
DEPT



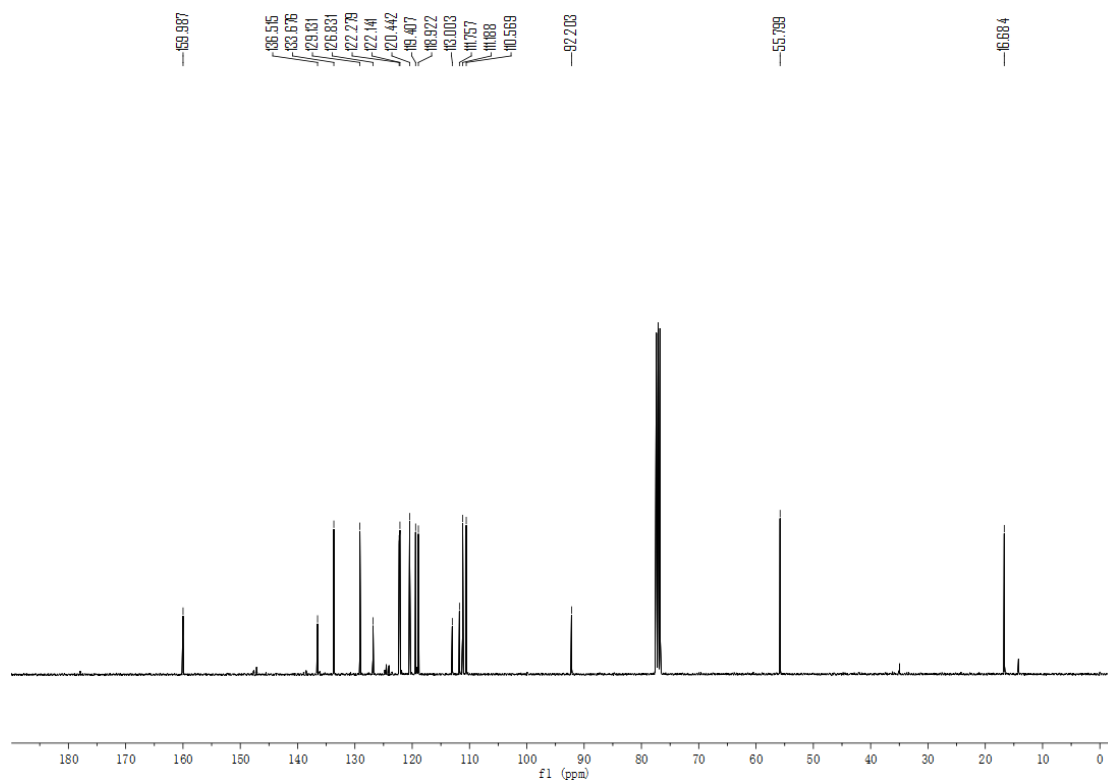
3-(3-(2-methoxyphenyl)prop-2-yn-1-yl)-1H-indole (3ka)

¹H NMR (400 MHz, CDCl₃):

8.009, 7.733, 7.704, 7.426, 7.422, 7.407, 7.403, 7.332, 7.278, 7.269, 7.251, 7.238, 7.229, 7.200, 7.180, 7.154, 7.106, 7.107, 6.901, 6.882, 6.872, 6.863, 6.851, 3.965, 3.877

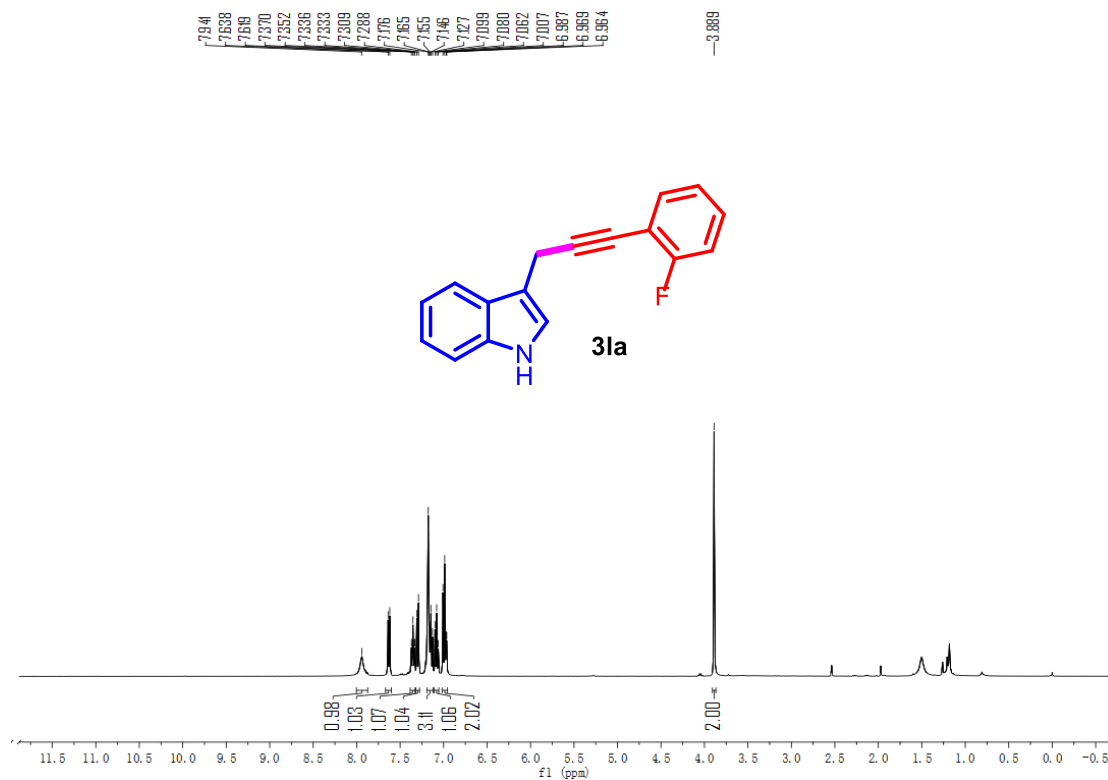


^{13}C NMR (100 MHz, CDCl_3):

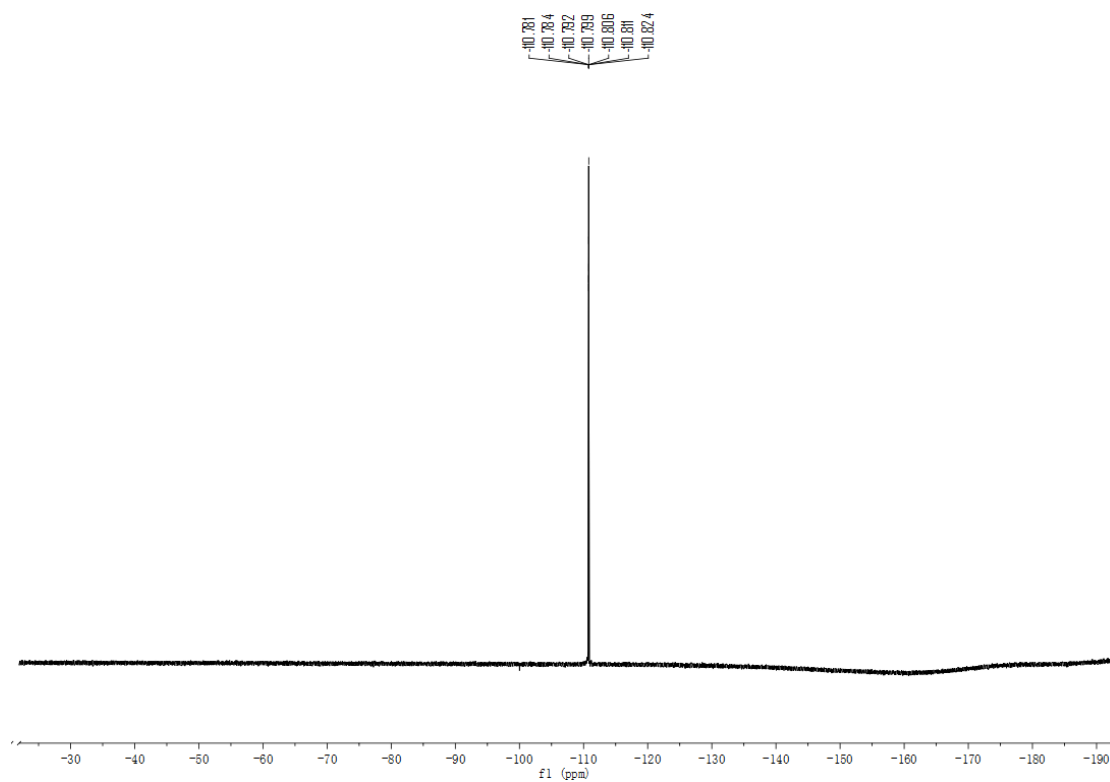


3-(3-(2-fluorophenyl)prop-2-yn-1-yl)-1H-indole (3la)

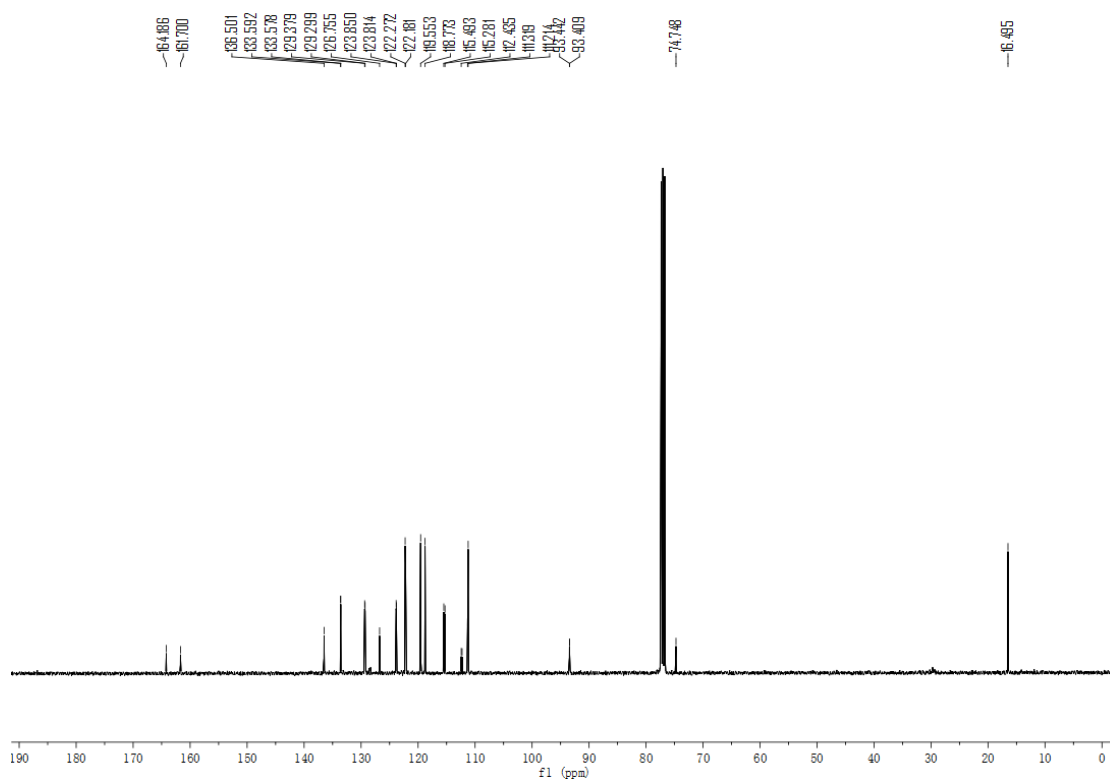
^1H NMR (400 MHz, CDCl_3):



¹⁹F NMR (376 MHz, CDCl₃):

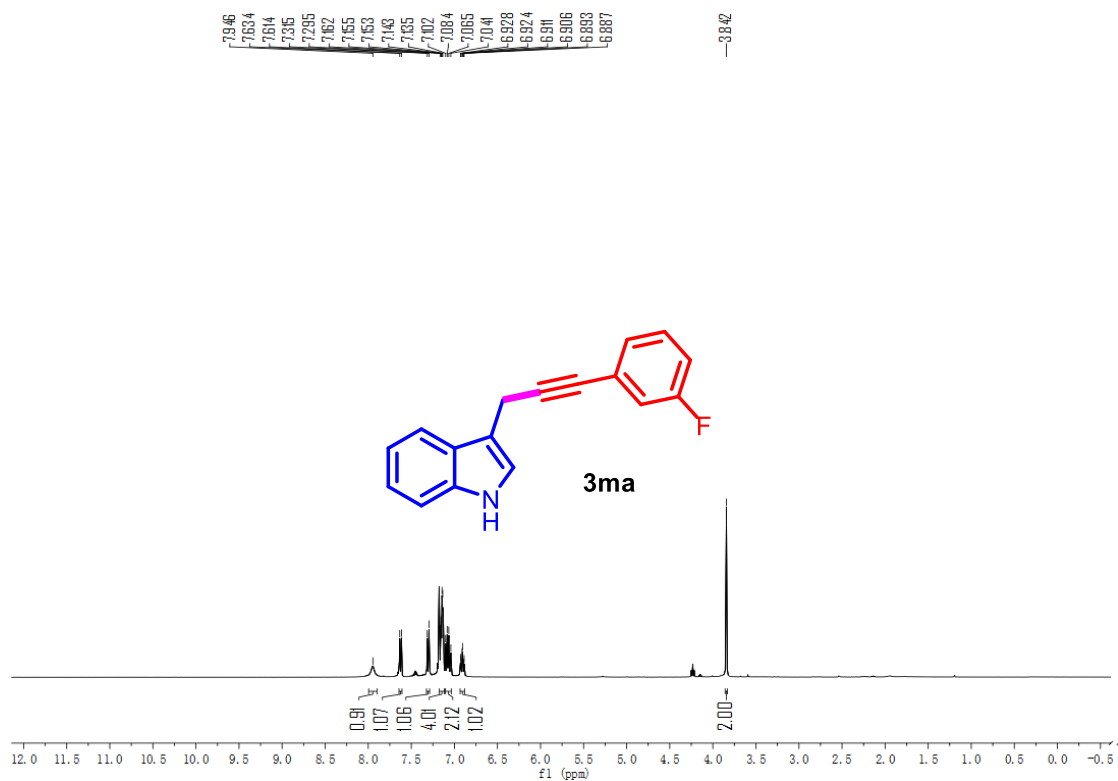


¹³C NMR (100 MHz, CDCl₃):

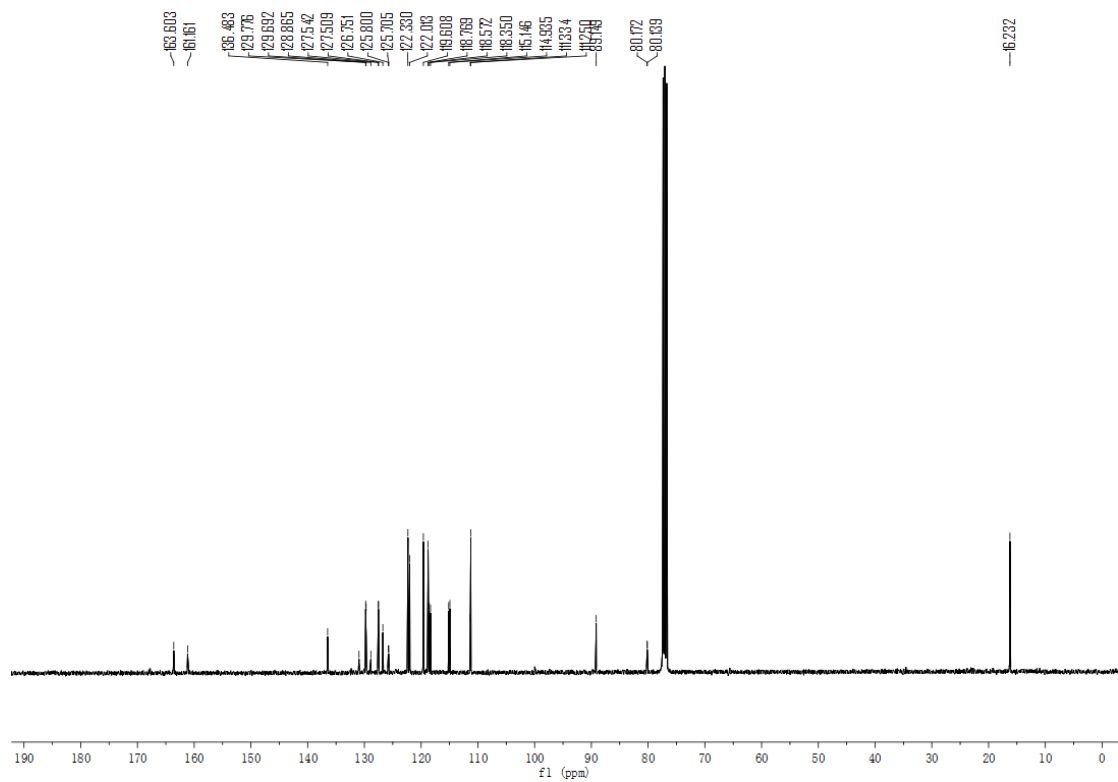


3-(3-(3-fluorophenyl)prop-2-yn-1-yl)-1H-indole (3ma)

¹H NMR (400 MHz, CDCl₃):

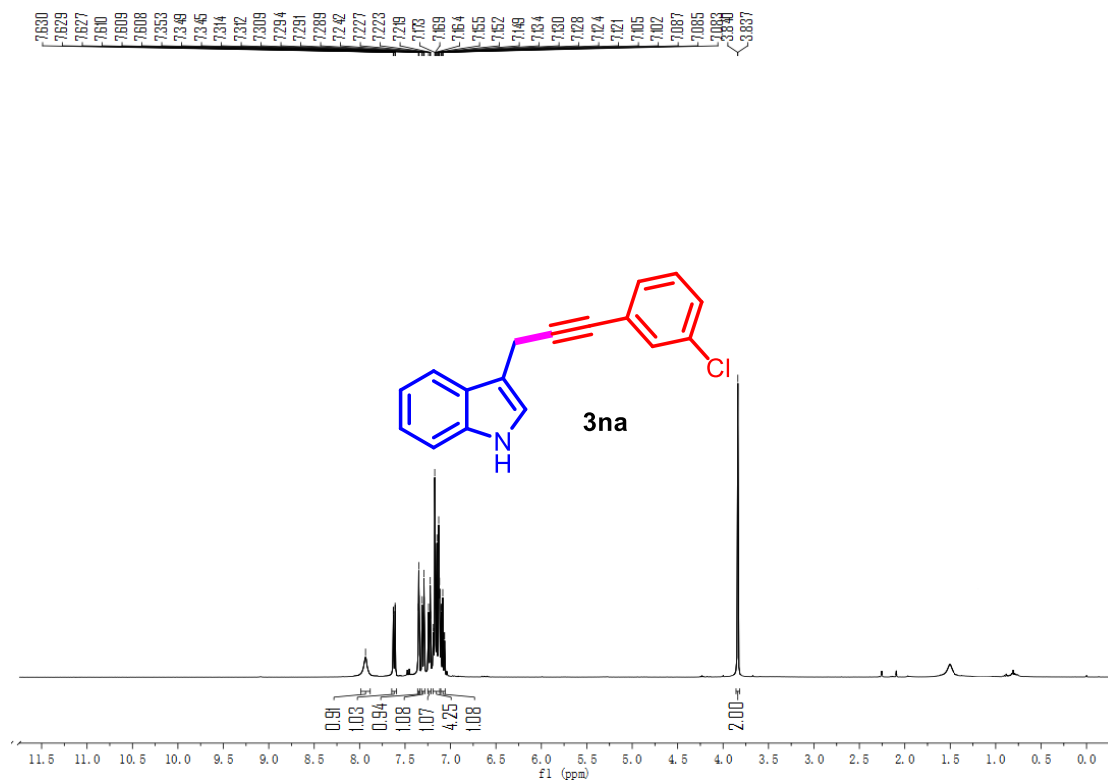


¹³C NMR (100 MHz, CDCl₃):

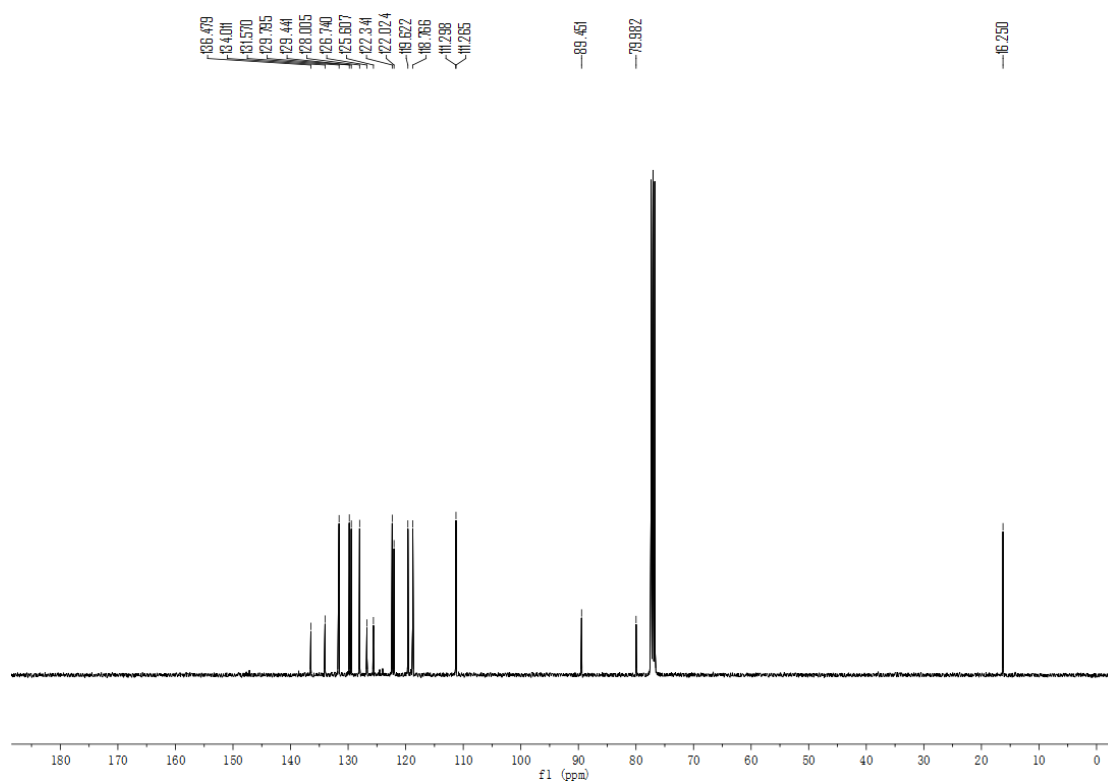


3-(3-(3-chlorophenyl)prop-2-yn-1-yl)-1H-indole (3na)

¹H NMR (400 MHz, CDCl₃):

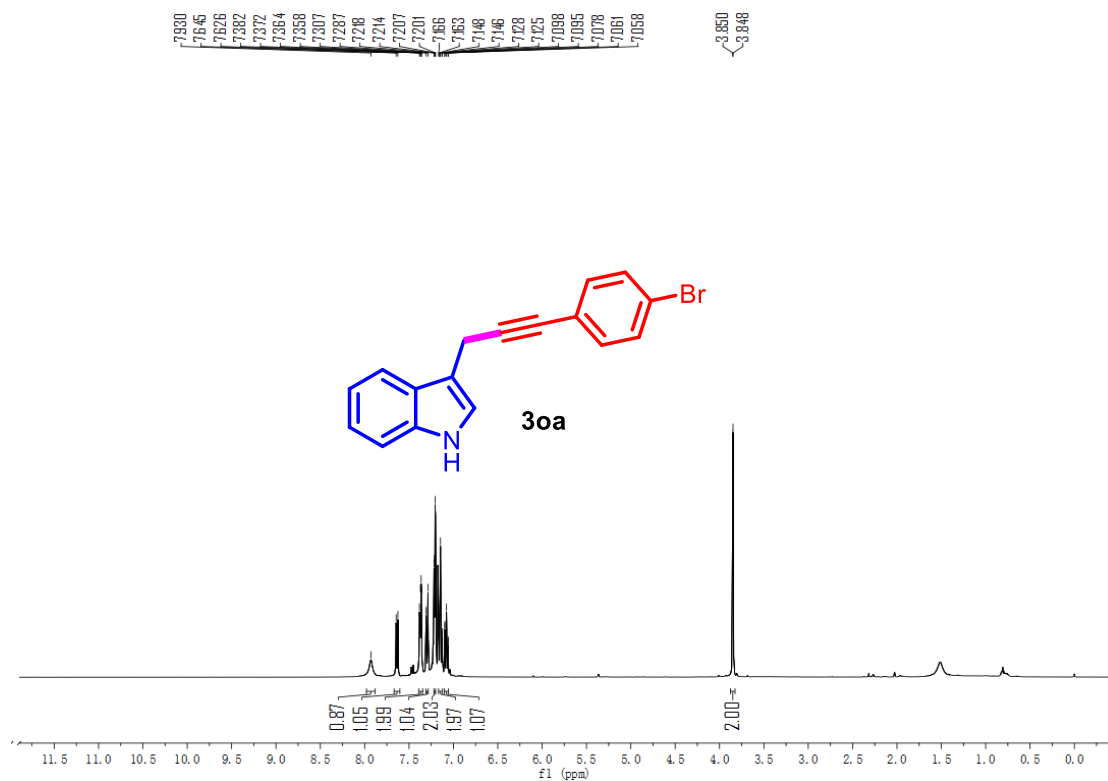


¹³C NMR (100 MHz, CDCl₃):

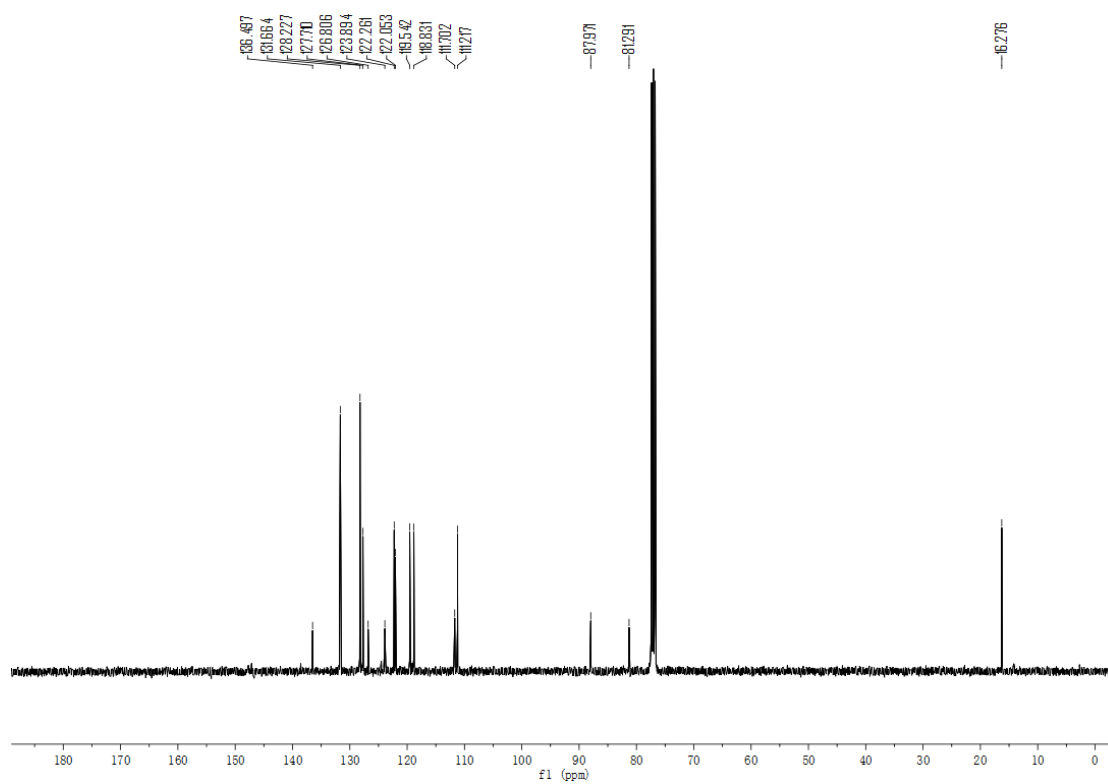


3-(3-(4-bromophenyl)prop-2-yn-1-yl)-1H-indole (3oa)

¹H NMR (400 MHz, CDCl₃):

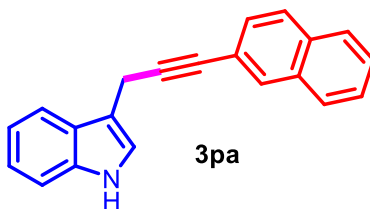
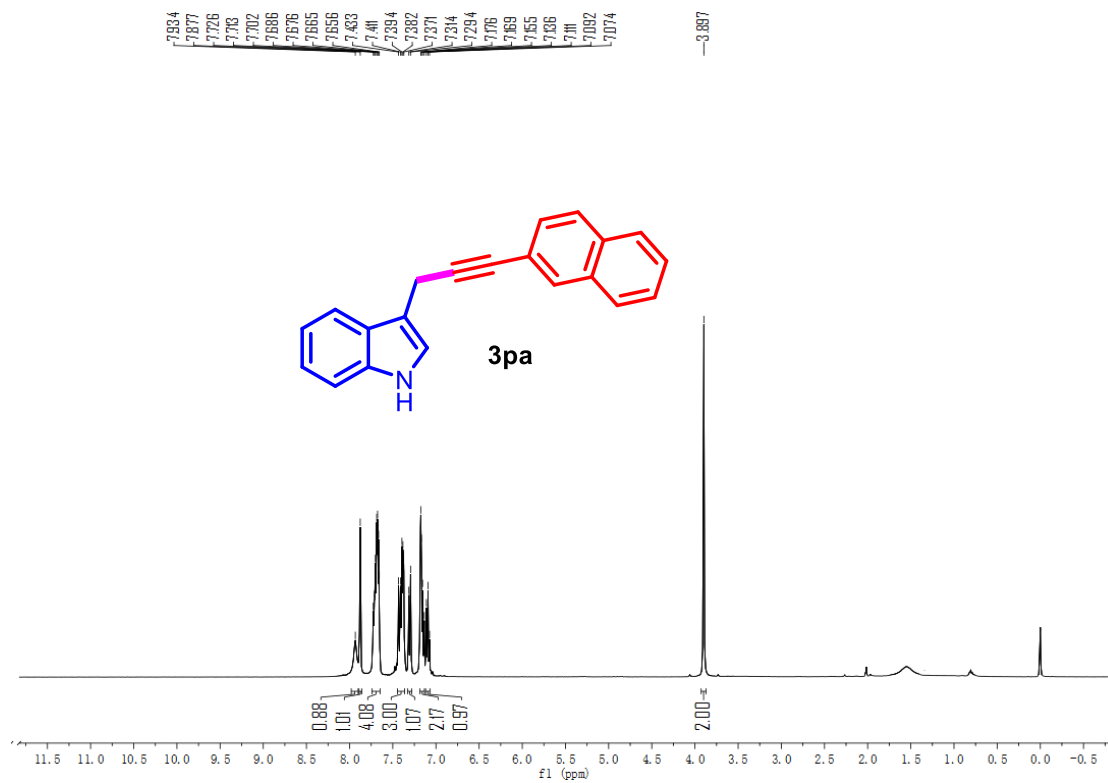


¹³C NMR (100 MHz, CDCl₃):

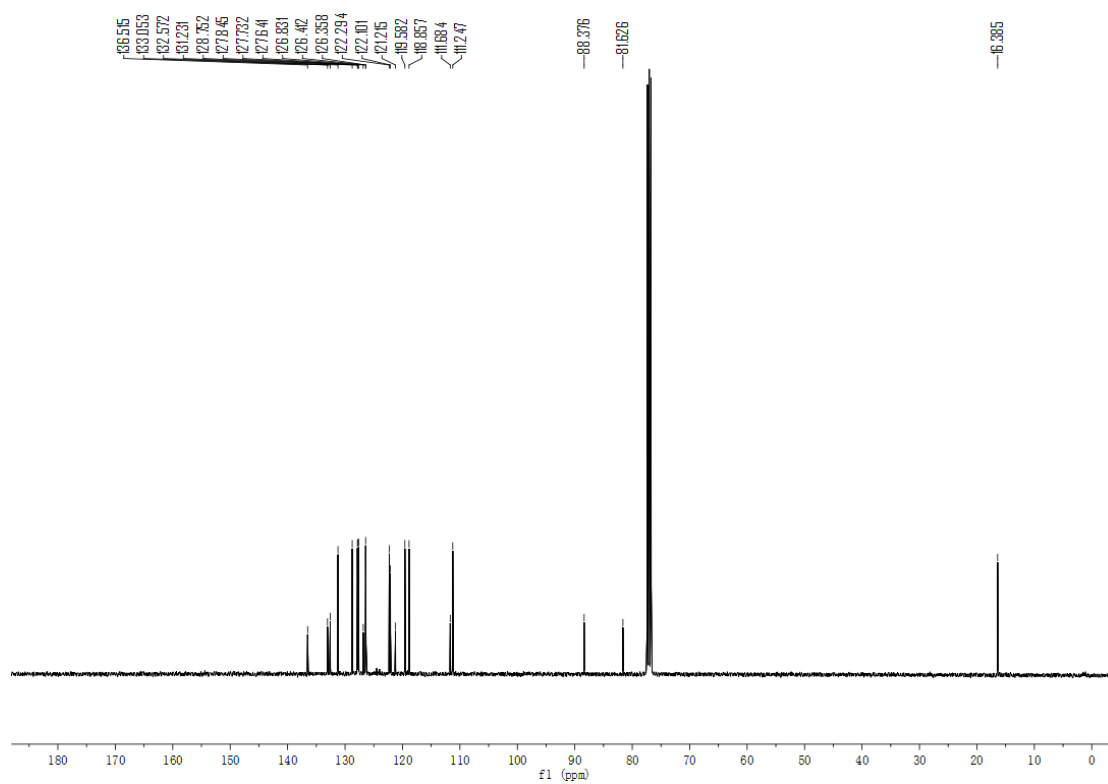


3-(3-(naphthalen-2-yl)prop-2-yn-1-yl)-1H-indole (3pa)

¹H NMR (400 MHz, CDCl₃):

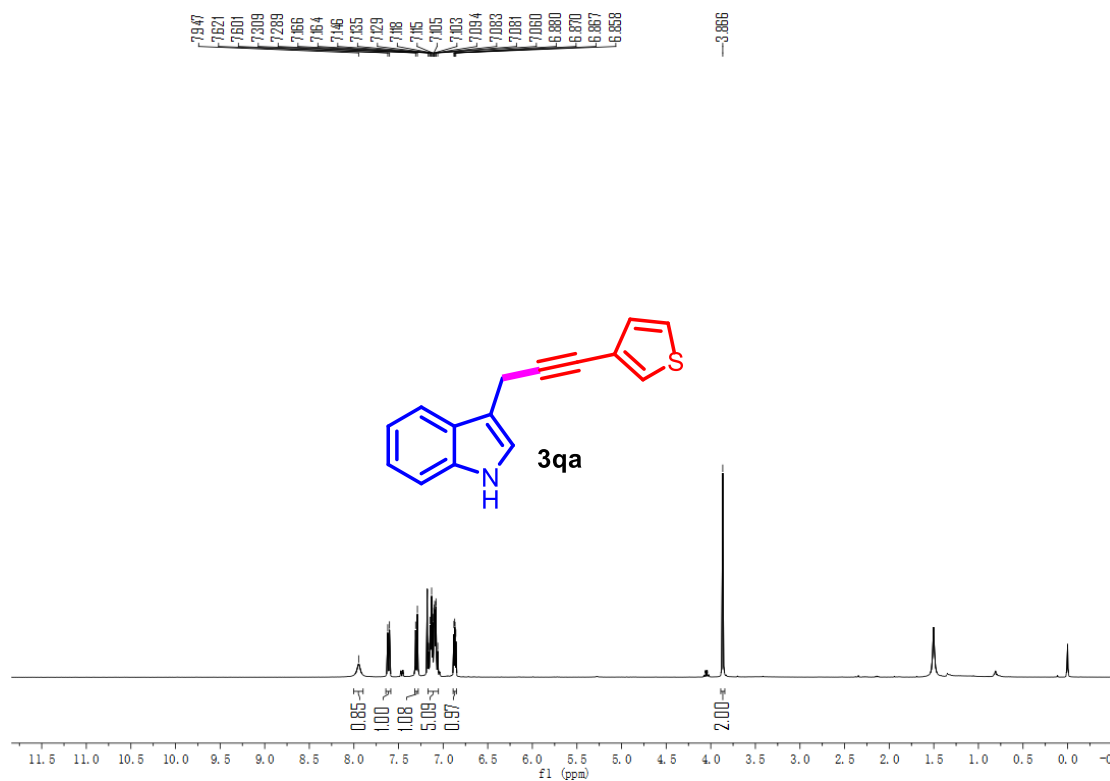


¹³C NMR (100 MHz, CDCl₃):

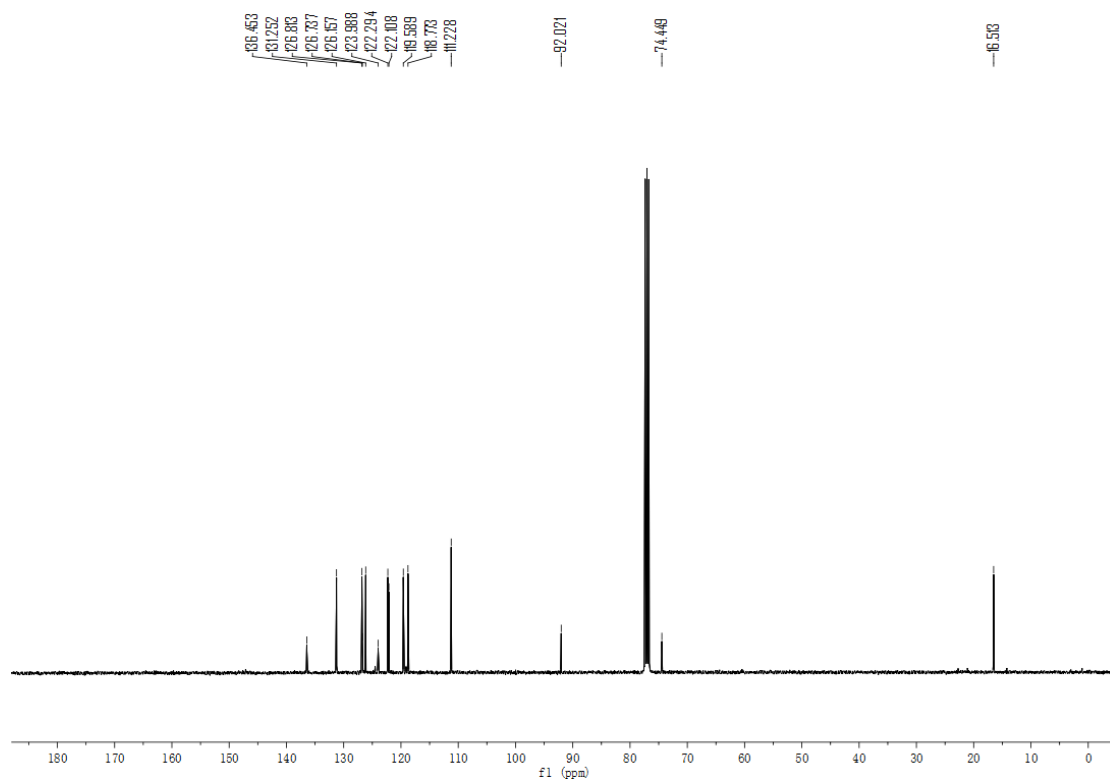


3-(3-(thiophen-3-yl)prop-2-yn-1-yl)-1H-indole (3qa)

^1H NMR (400 MHz, CDCl_3):

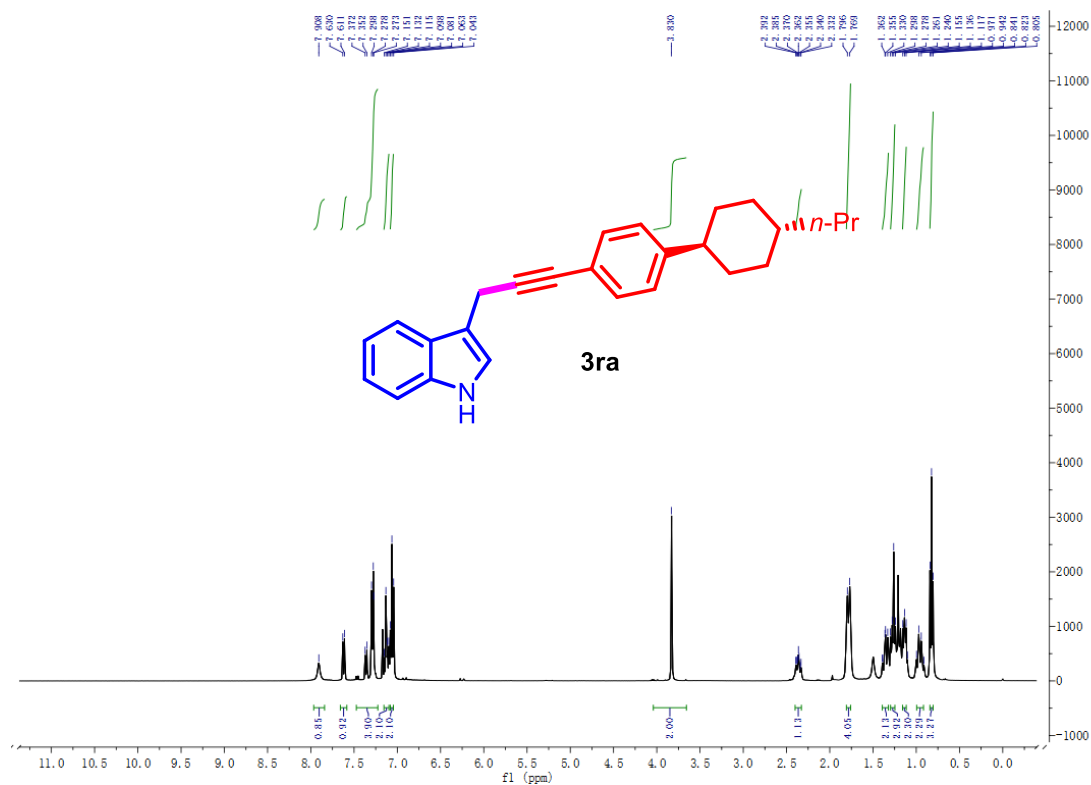


^{13}C NMR (100 MHz, CDCl_3):

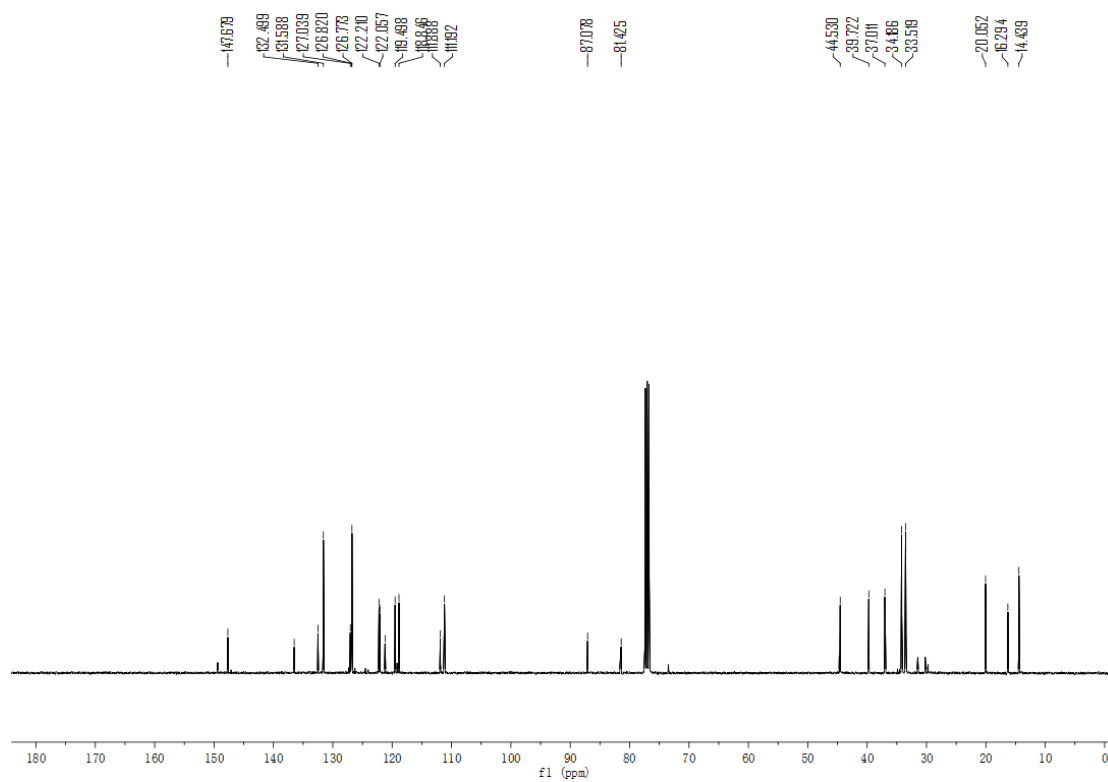


3-(3-(4-((1s,4r)-4-propylcyclohexyl)phenyl)prop-2-yn-1-yl)-1H-indole (3ra)

¹H NMR (400 MHz, CDCl₃):

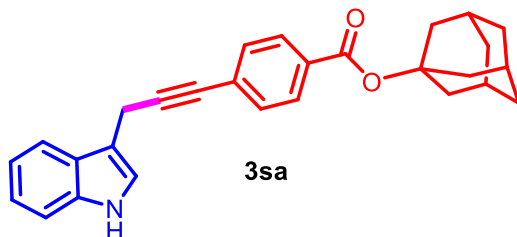


¹³C NMR (100 MHz, CDCl₃):

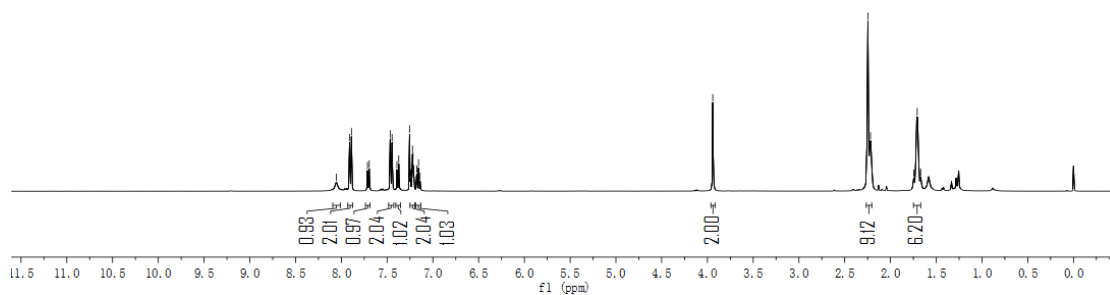


(3s,5s,7s)-adamantan-1-yl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (3sa)

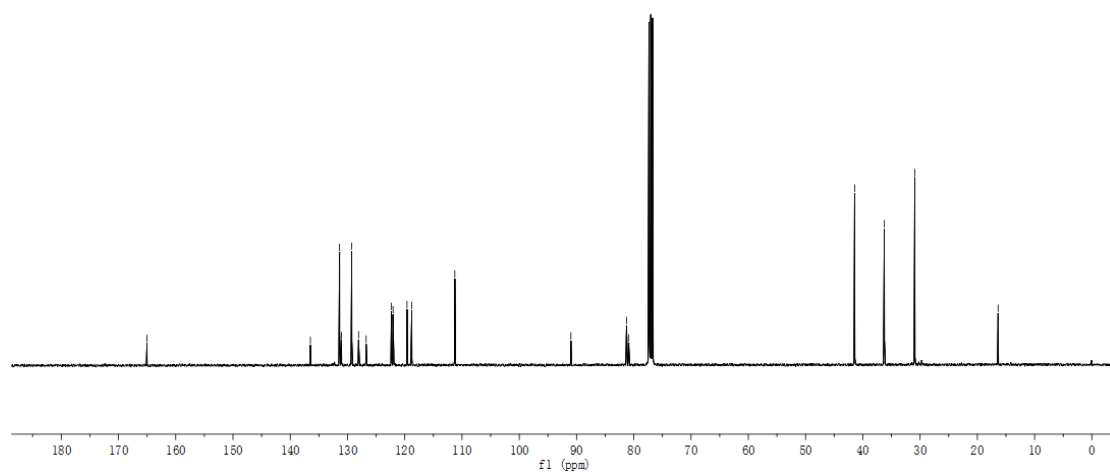
¹H NMR (400 MHz, CDCl₃):



3sa

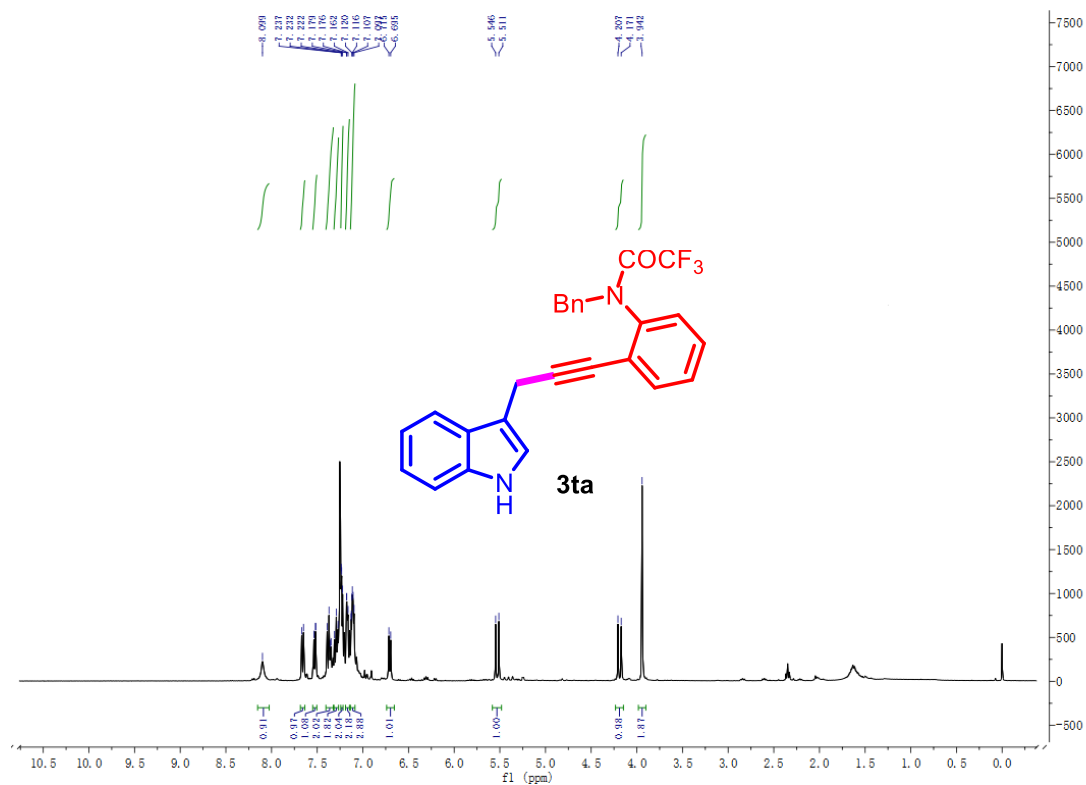


¹³C NMR (100 MHz, CDCl₃):

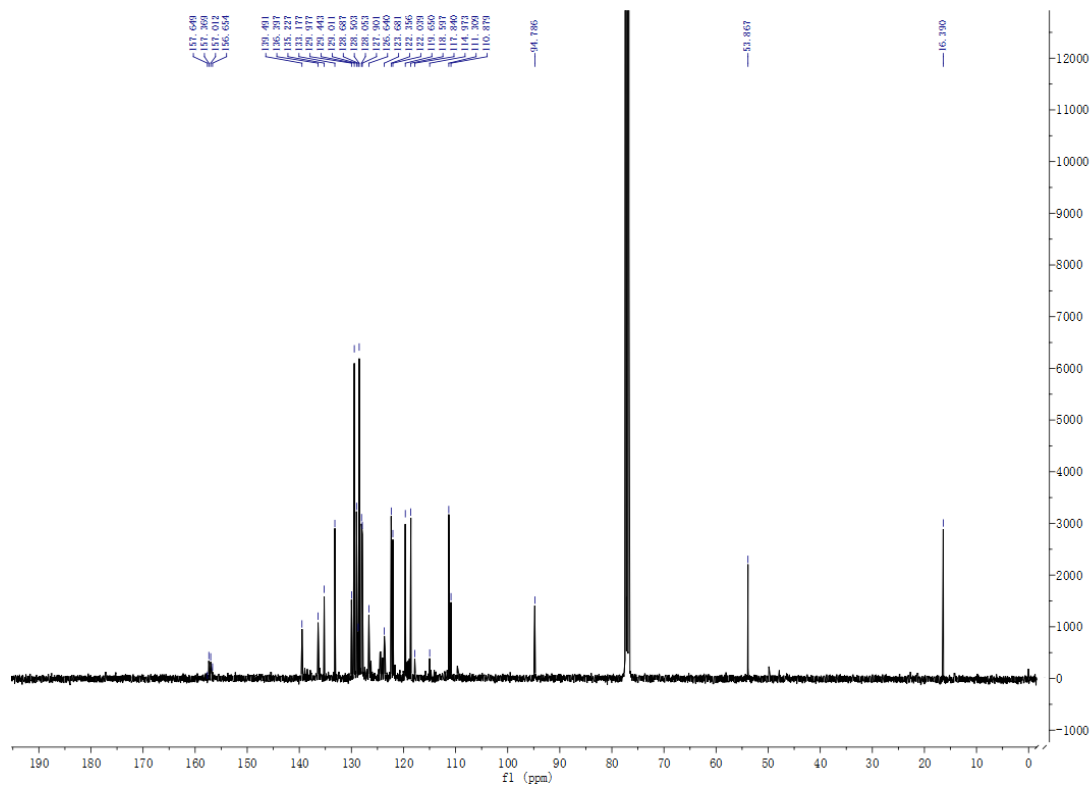


N-(2-(3-(1H-indol-3-yl)prop-1-yn-1-yl)phenyl)-N-benzyl-2,2,2-trifluoroacetamide (3ta)

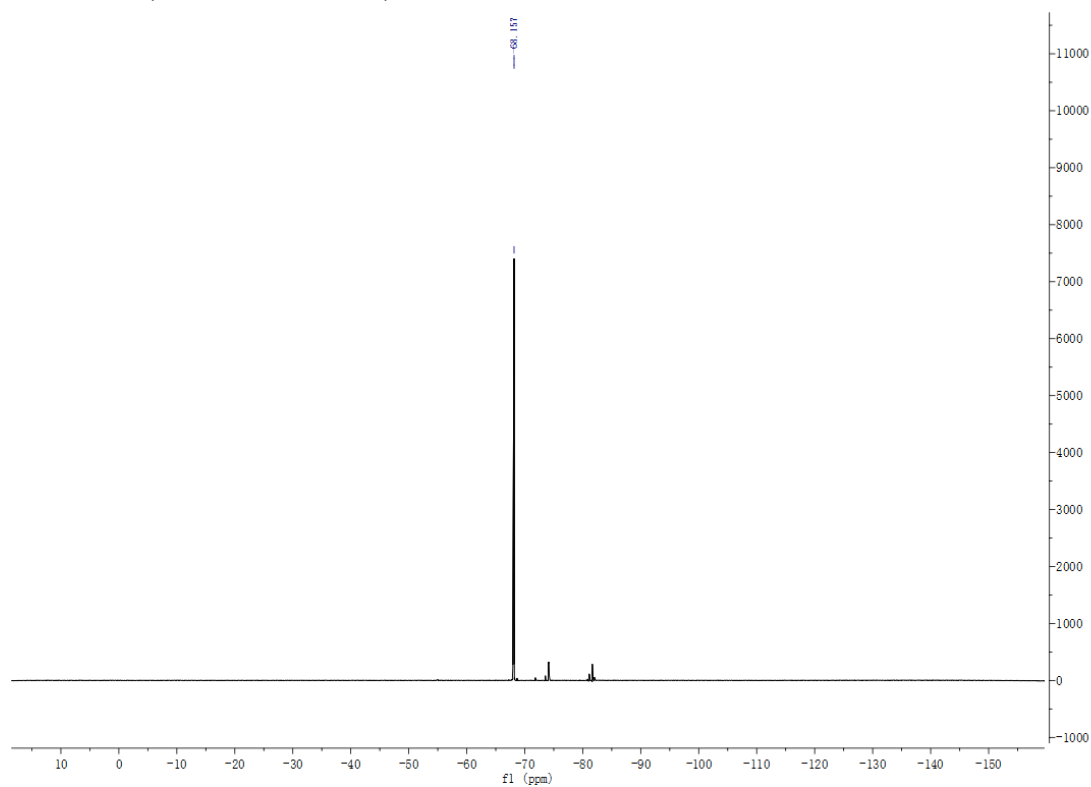
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

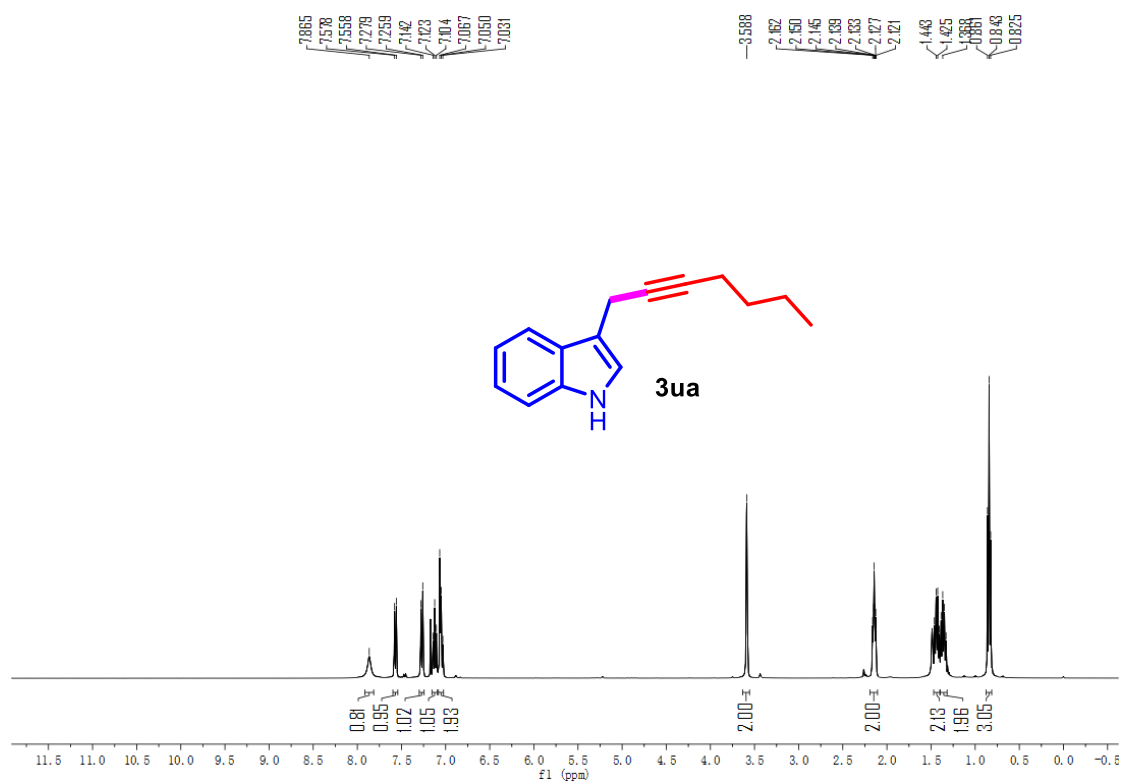


¹⁹F NMR (376 MHz, CDCl₃):

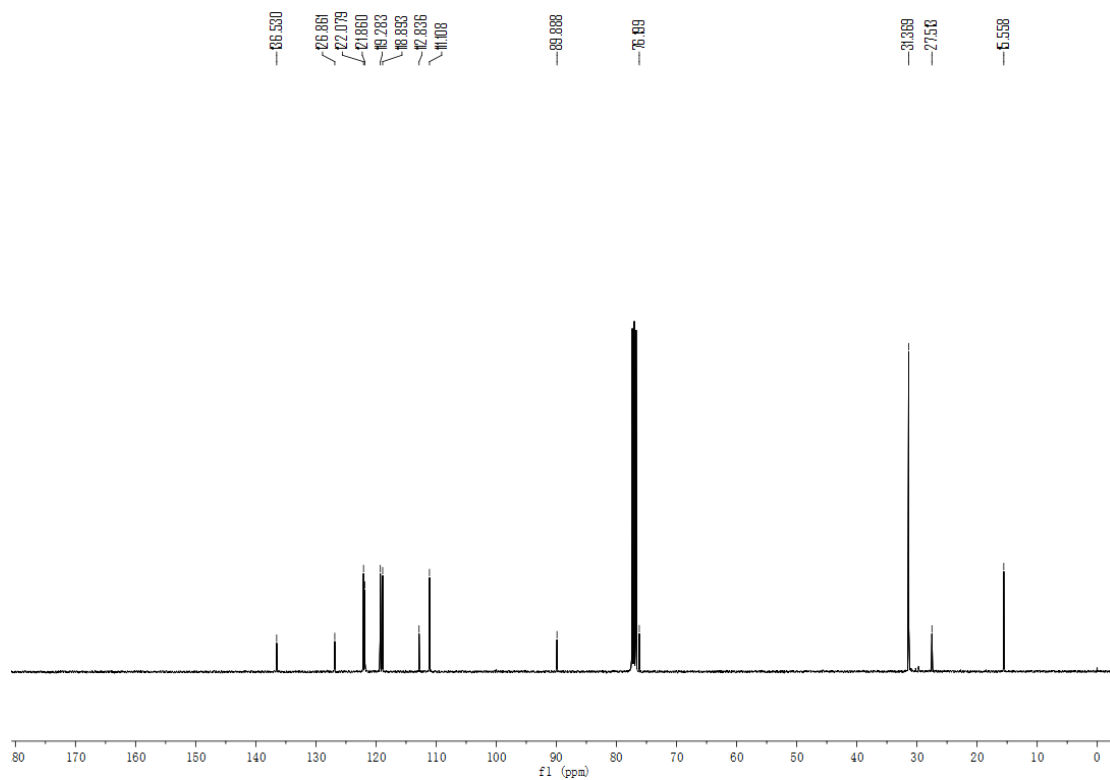


3-(hept-2-yn-1-yl)-1H-indole (3ua)

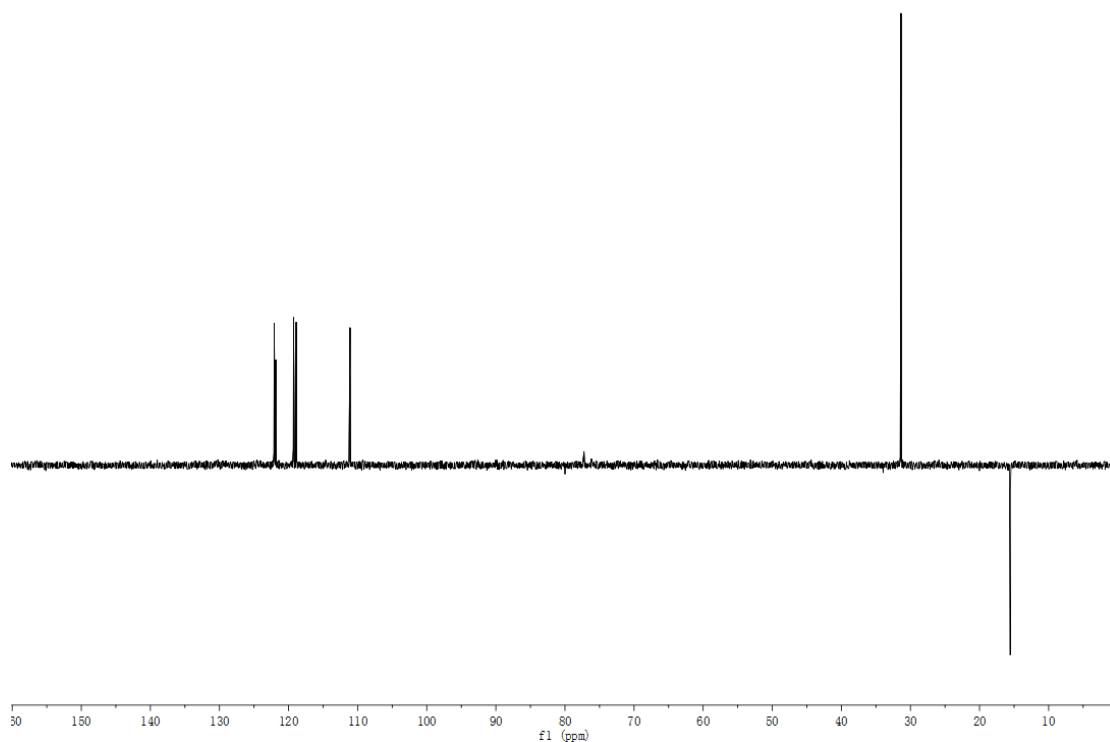
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

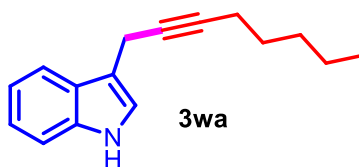
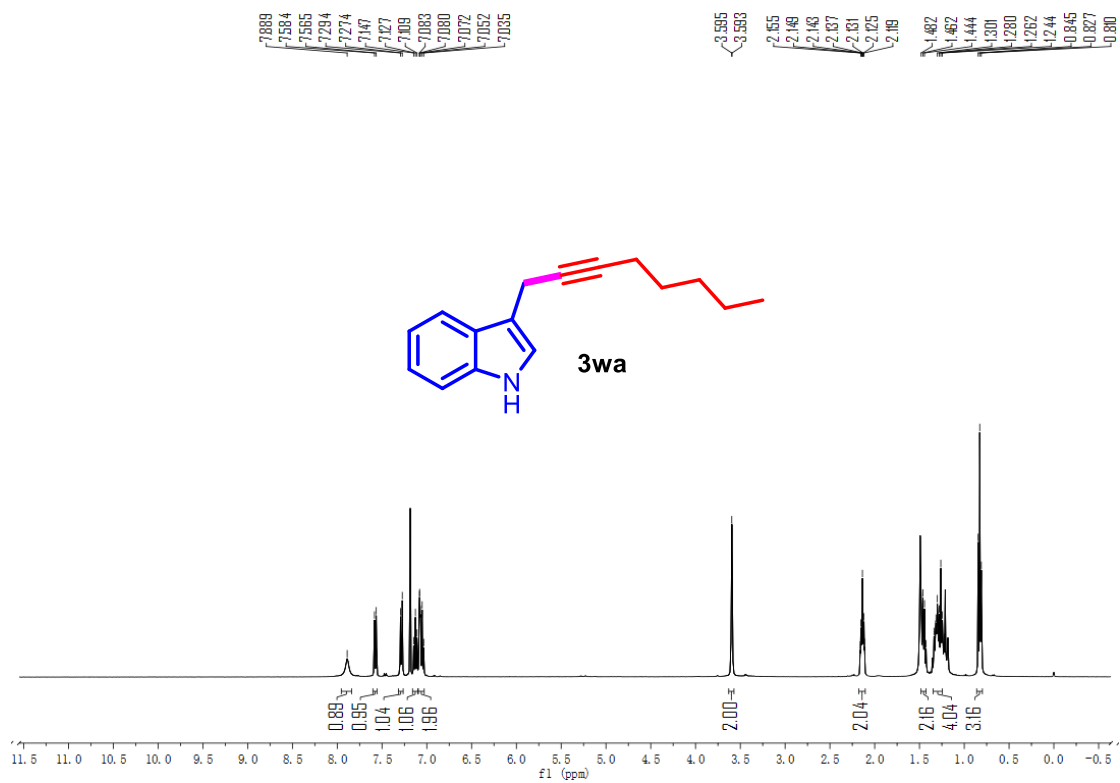


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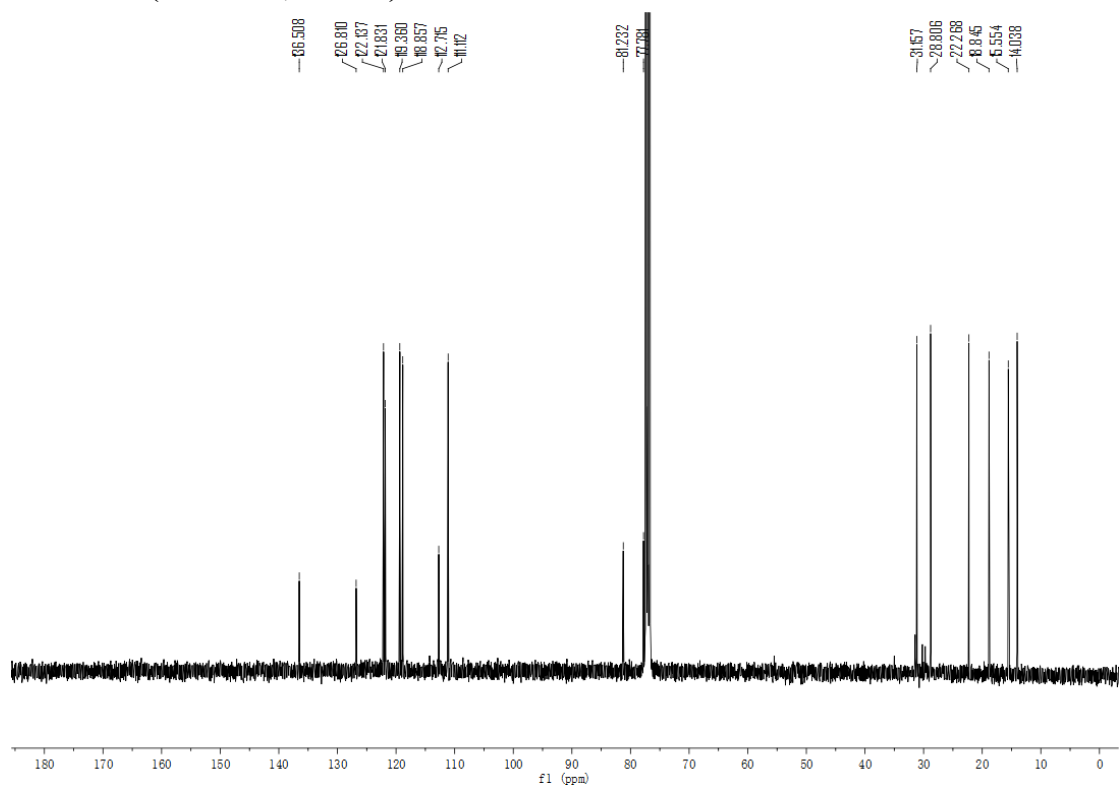


3-(oct-2-yn-1-yl)-1H-indole (3wa)

¹H NMR (400 MHz, CDCl₃):

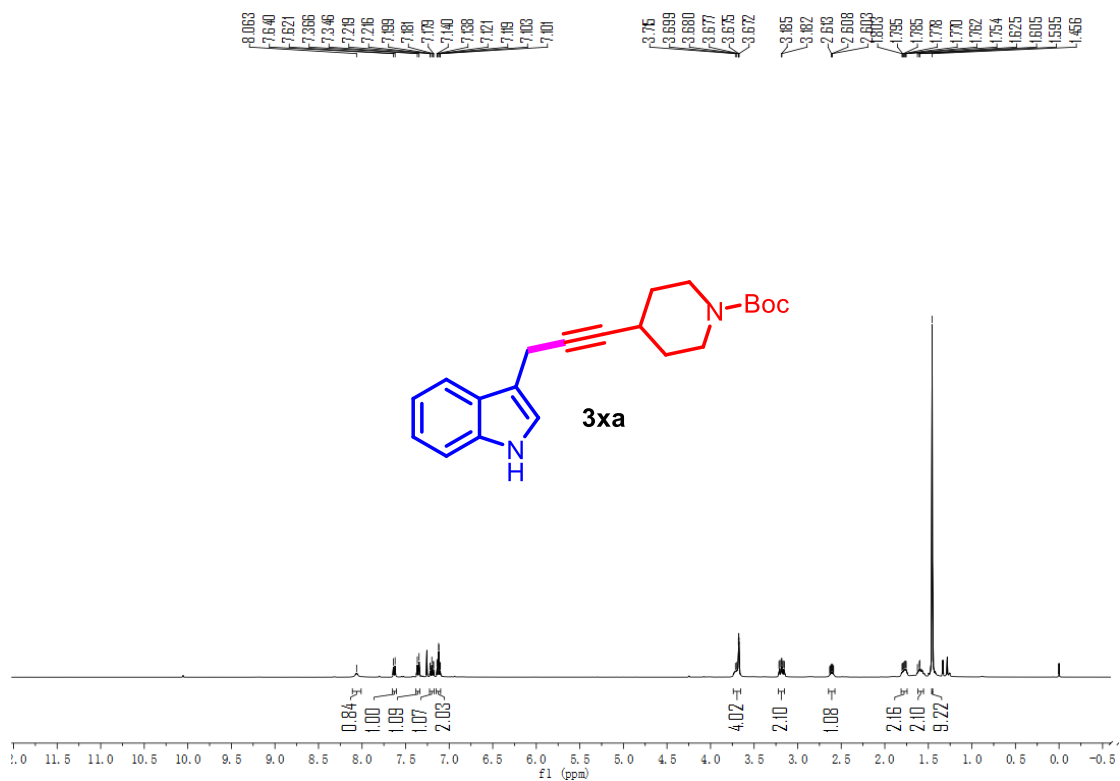


¹³C NMR (100 MHz, CDCl₃):

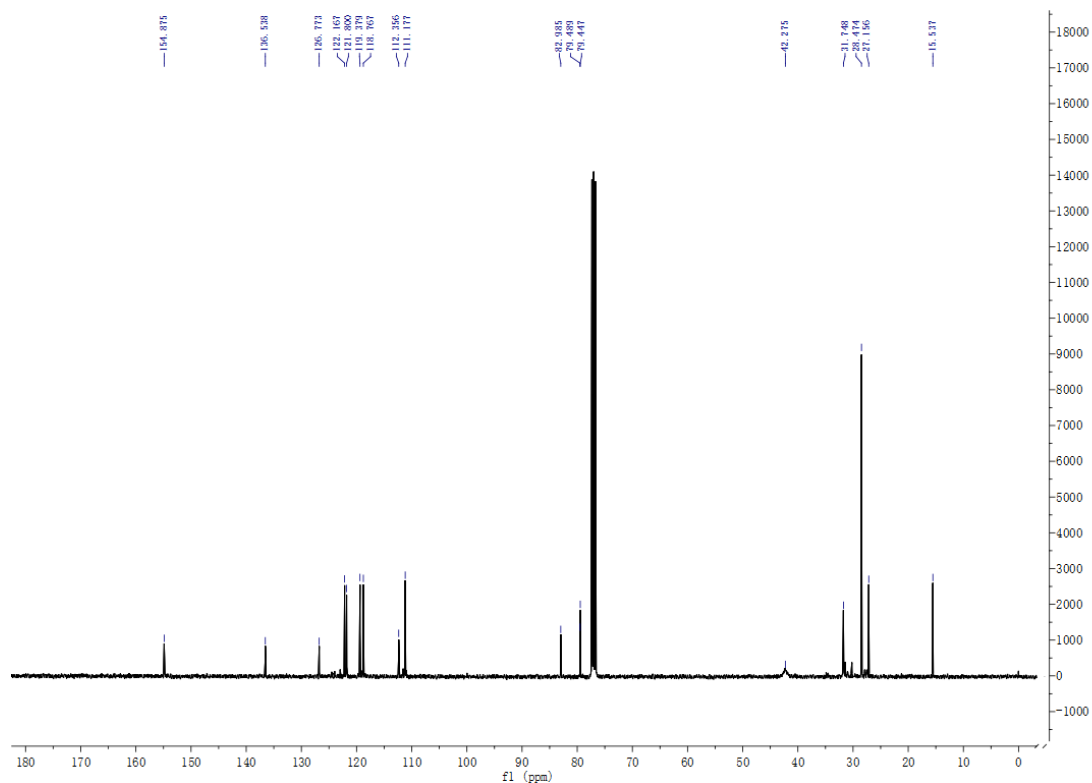


tert-butyl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)piperidine-1-carboxylate (3xa)

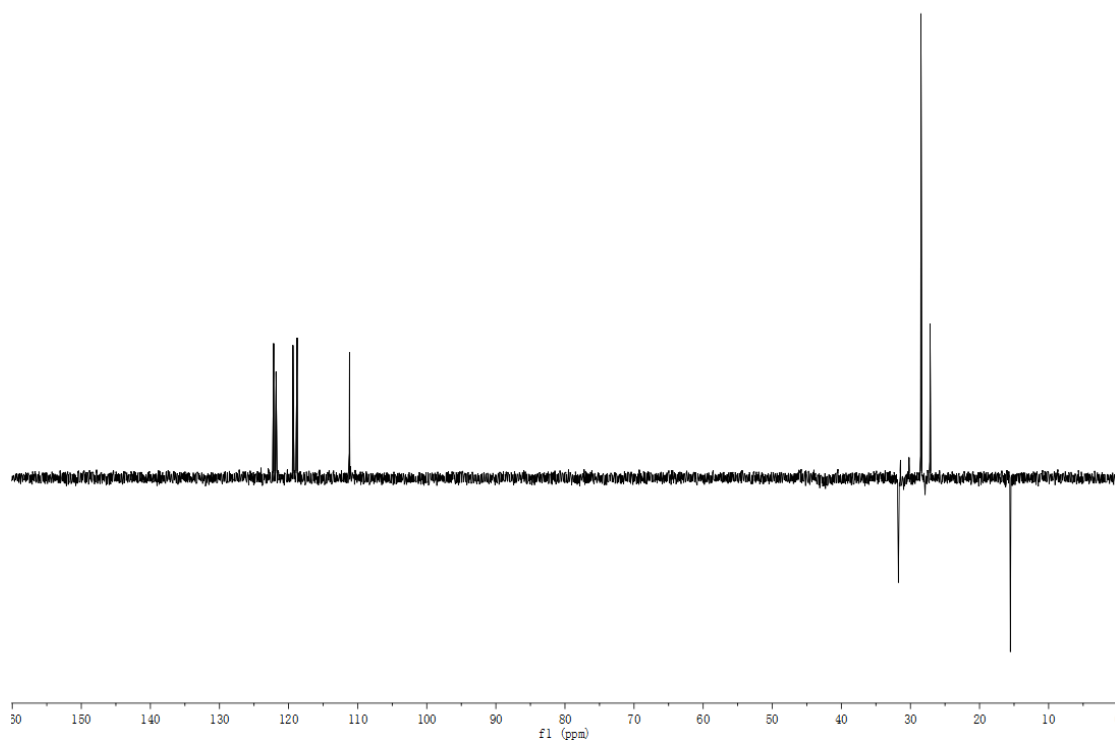
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

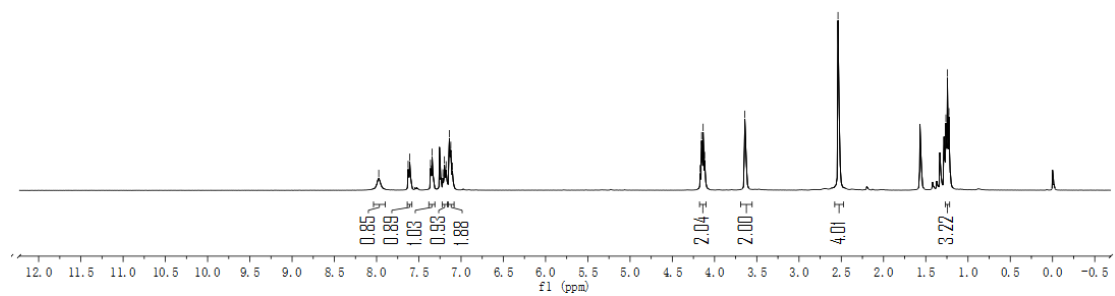
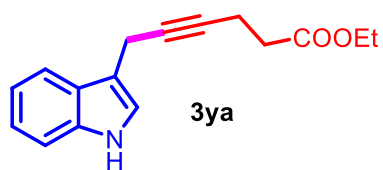
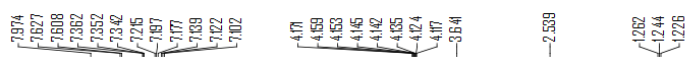


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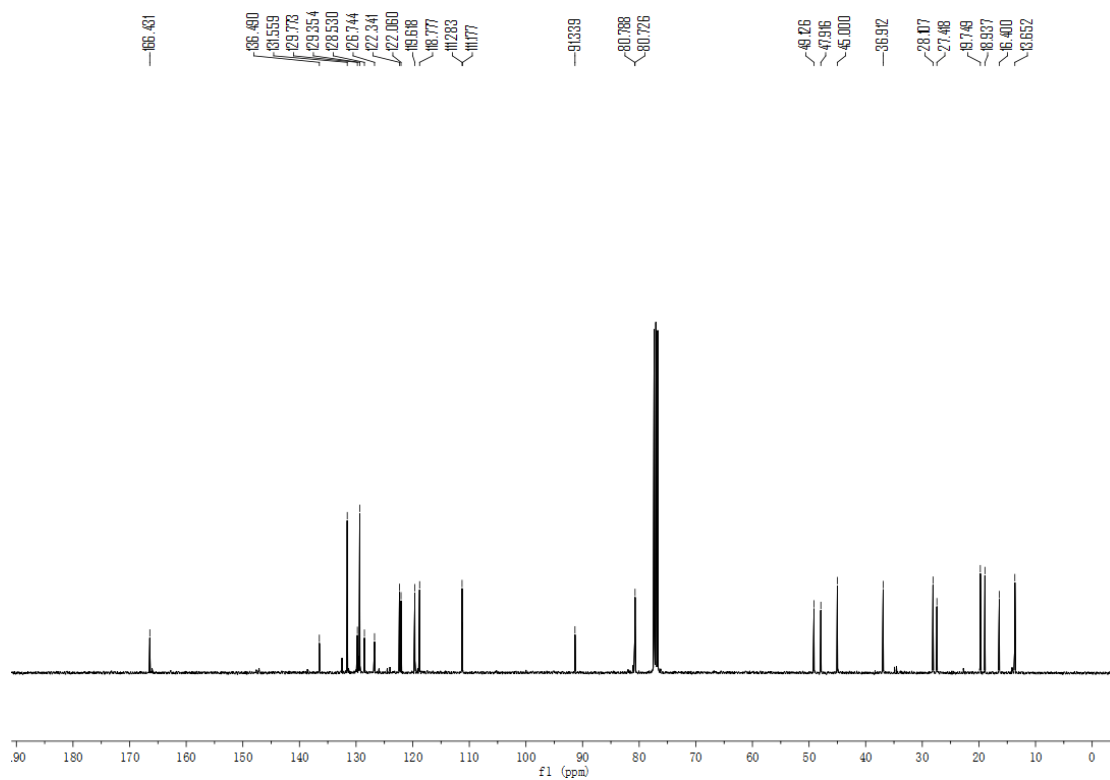


ethyl 6-(1H-indol-3-yl)hex-4-ynoate (3ya)

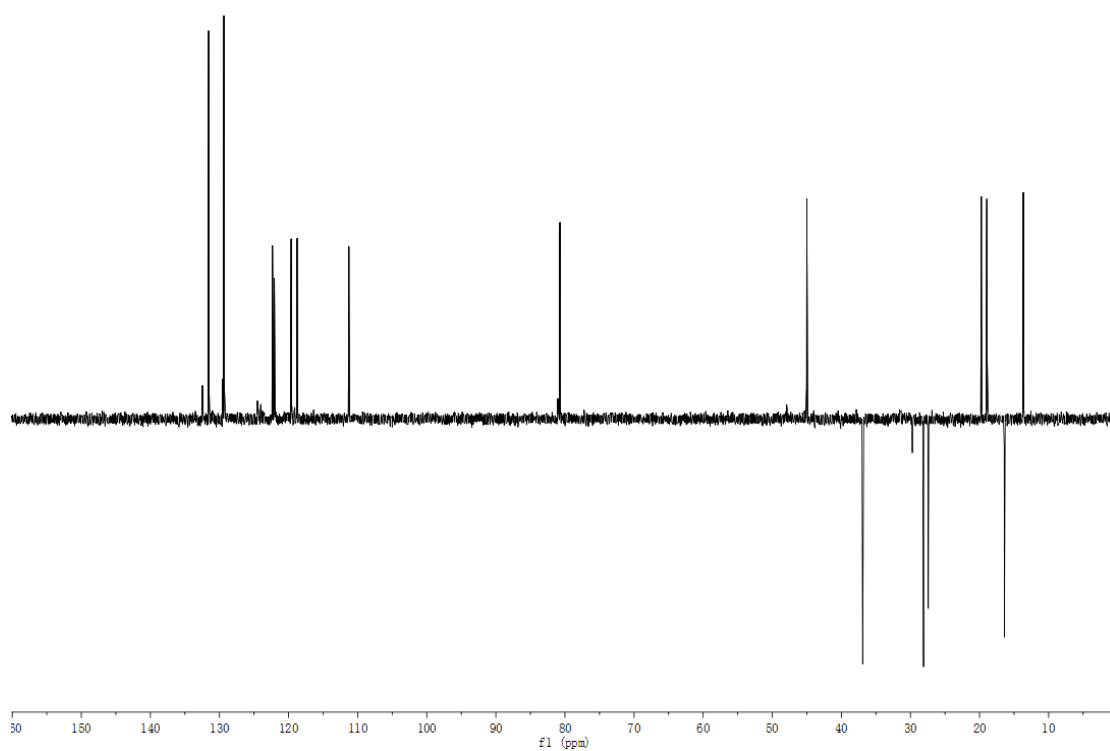
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

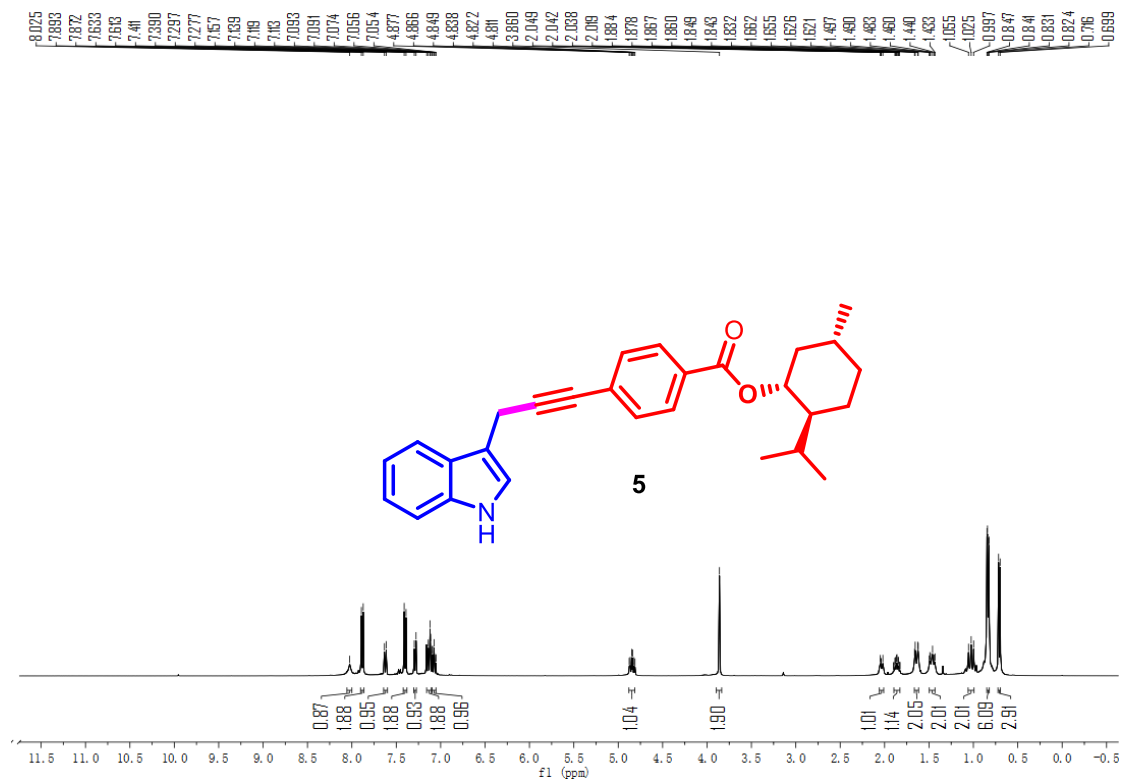


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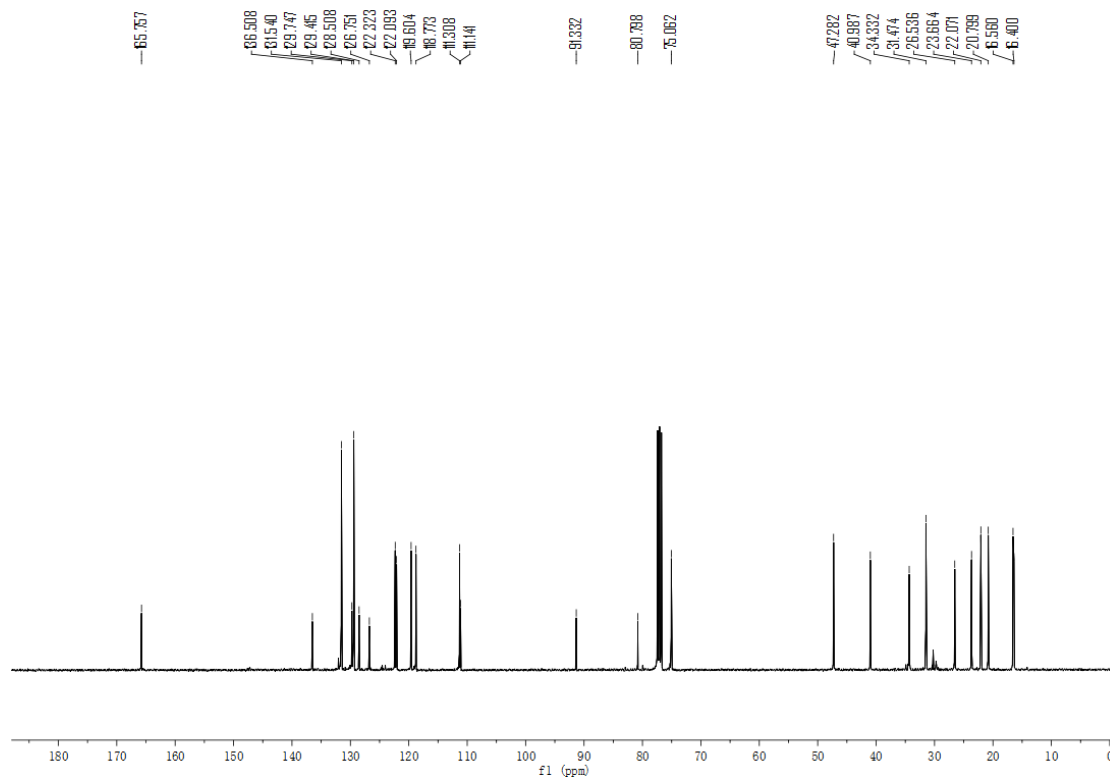


(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-(3-(1H-indol-3-yl)prop-1-yn-1-yl)benzoate (5)

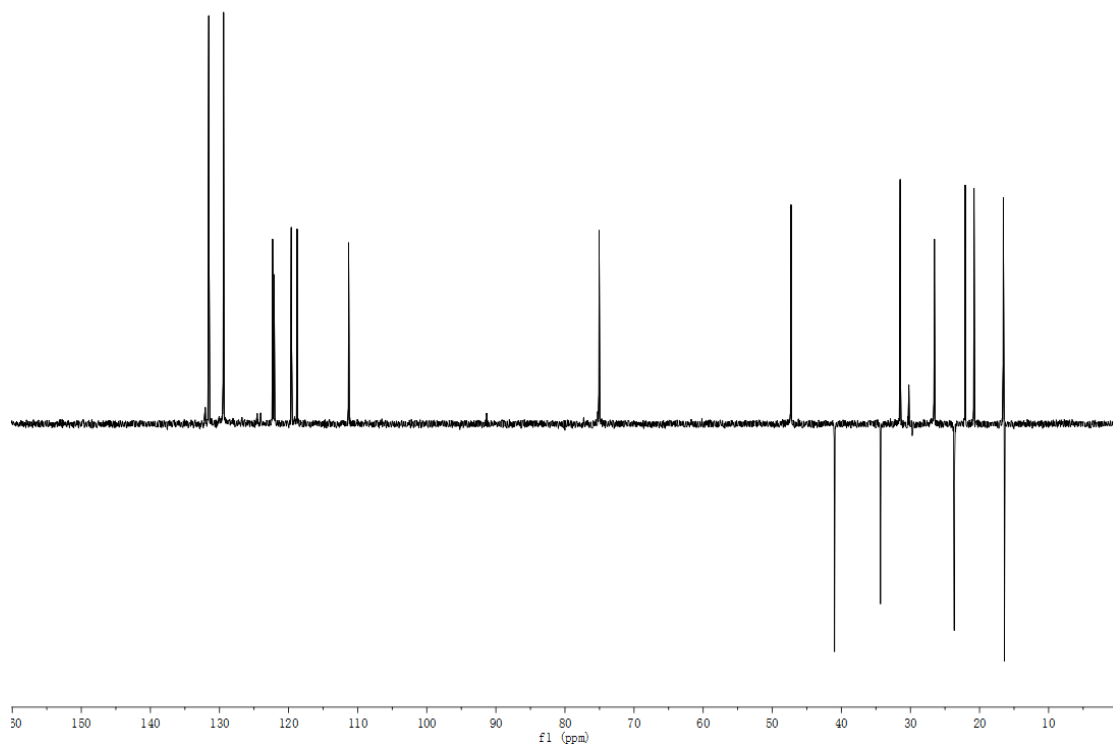
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

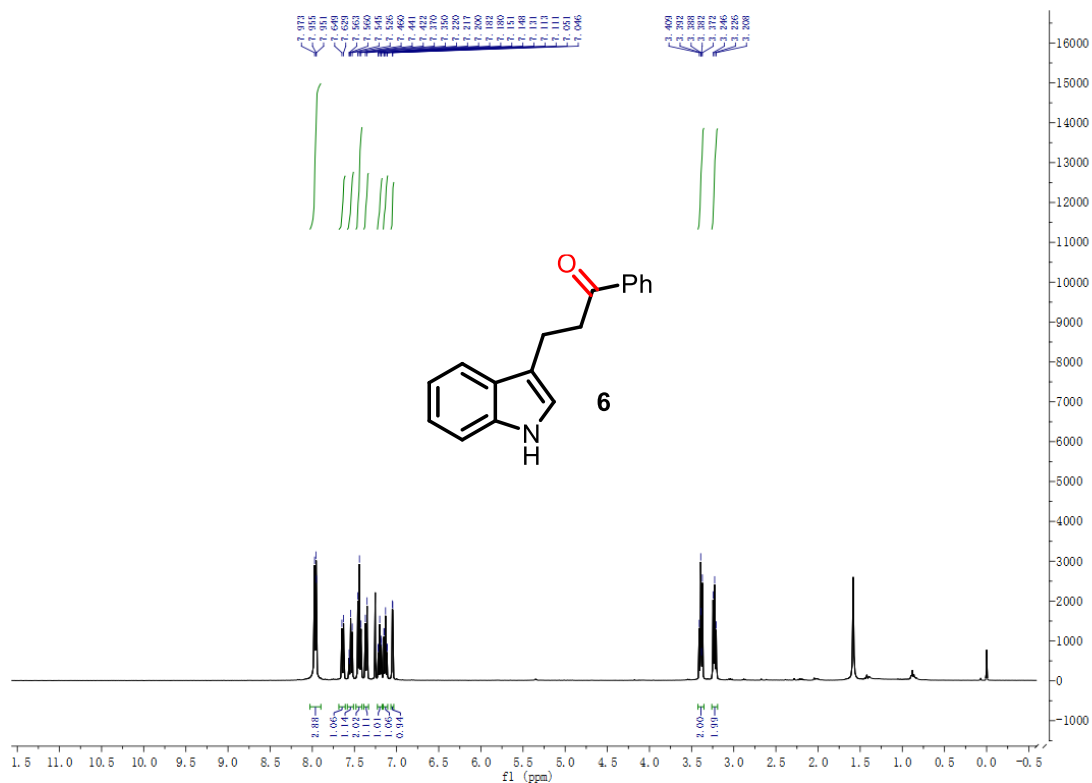


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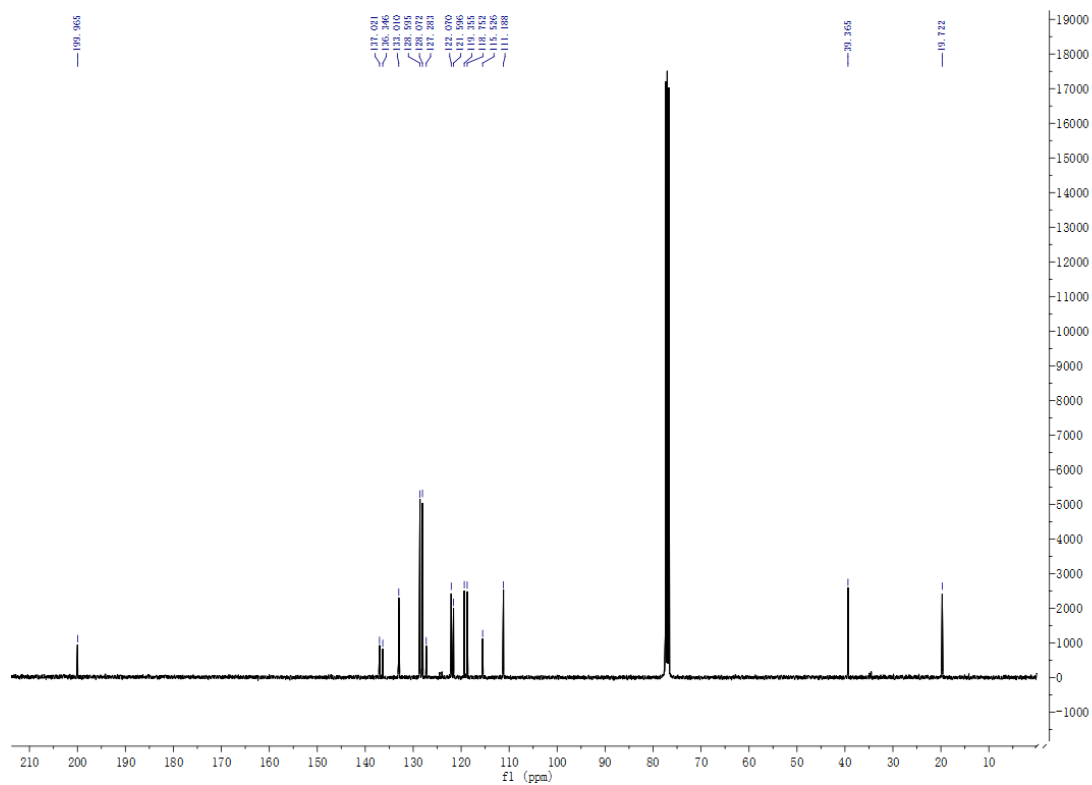


3-(1H-indol-3-yl)-1-phenylpropan-1-one (6)

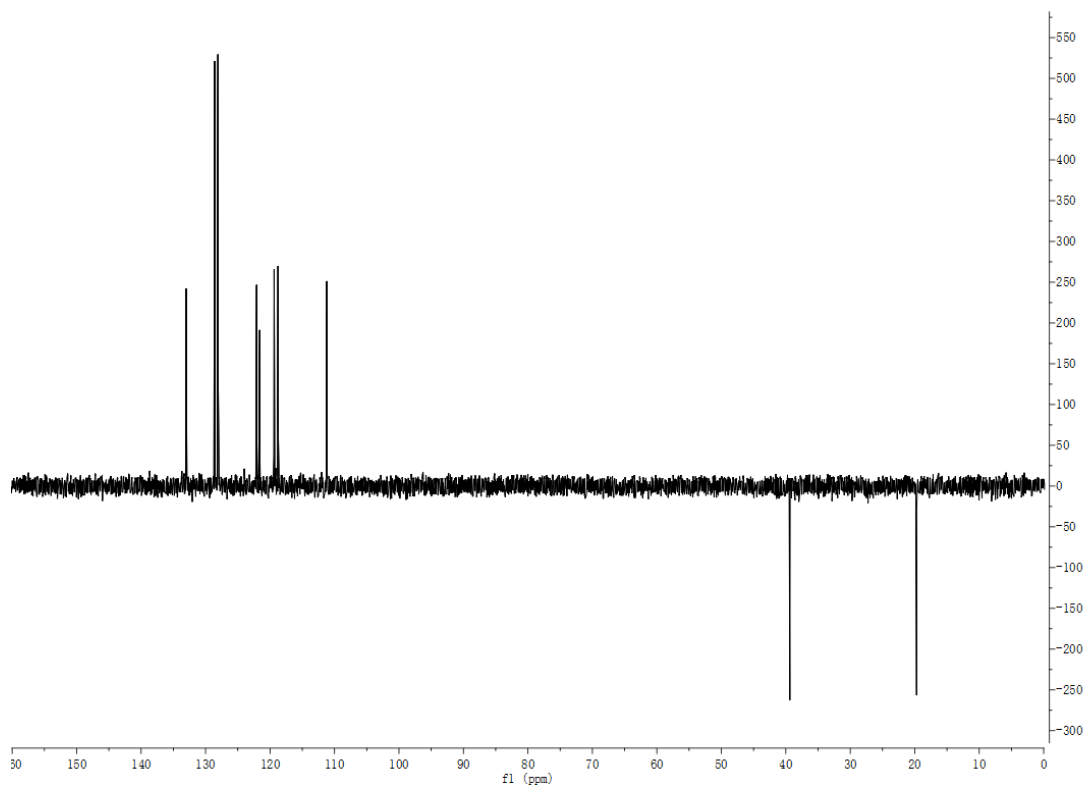
¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):

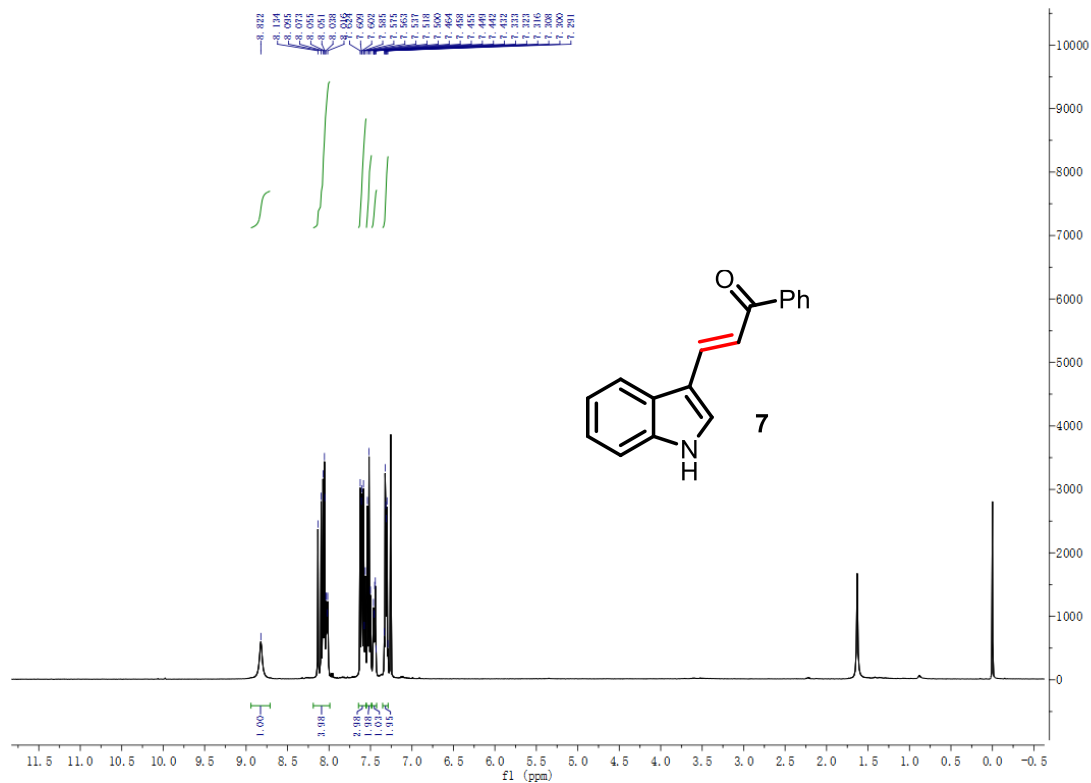


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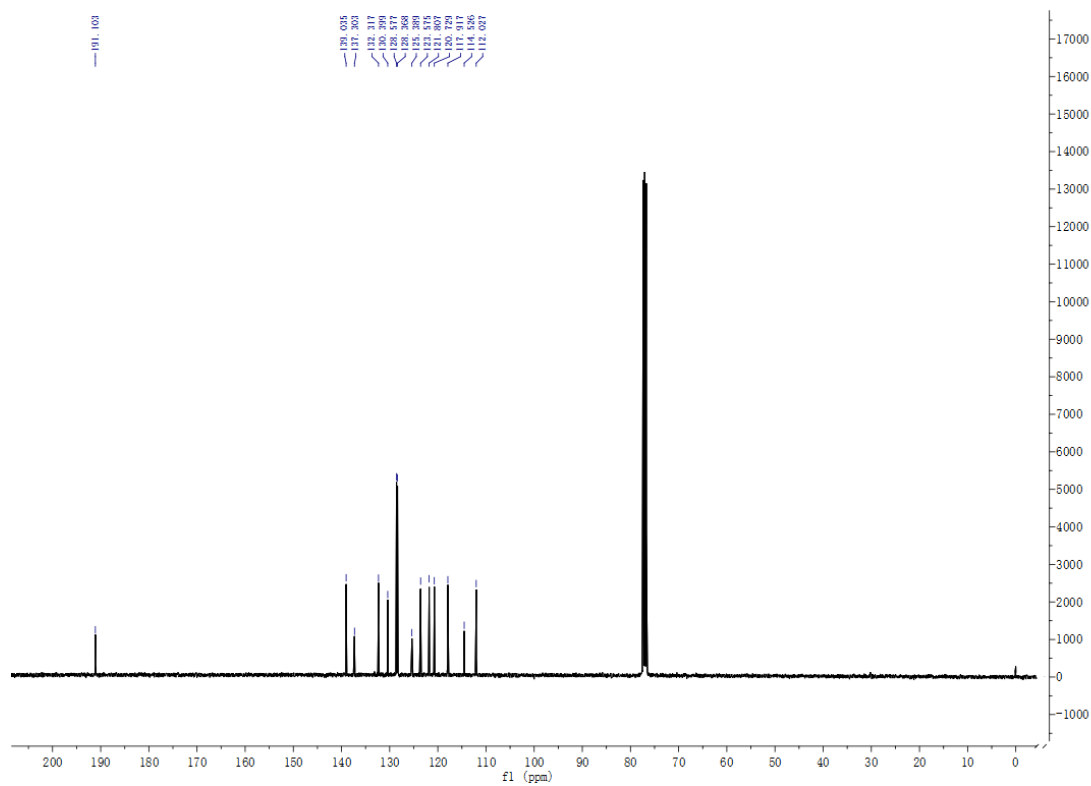


(E)-3-(1H-indol-3-yl)-1-phenylprop-2-en-1-one (7)

¹H NMR (400 MHz, CDCl₃):

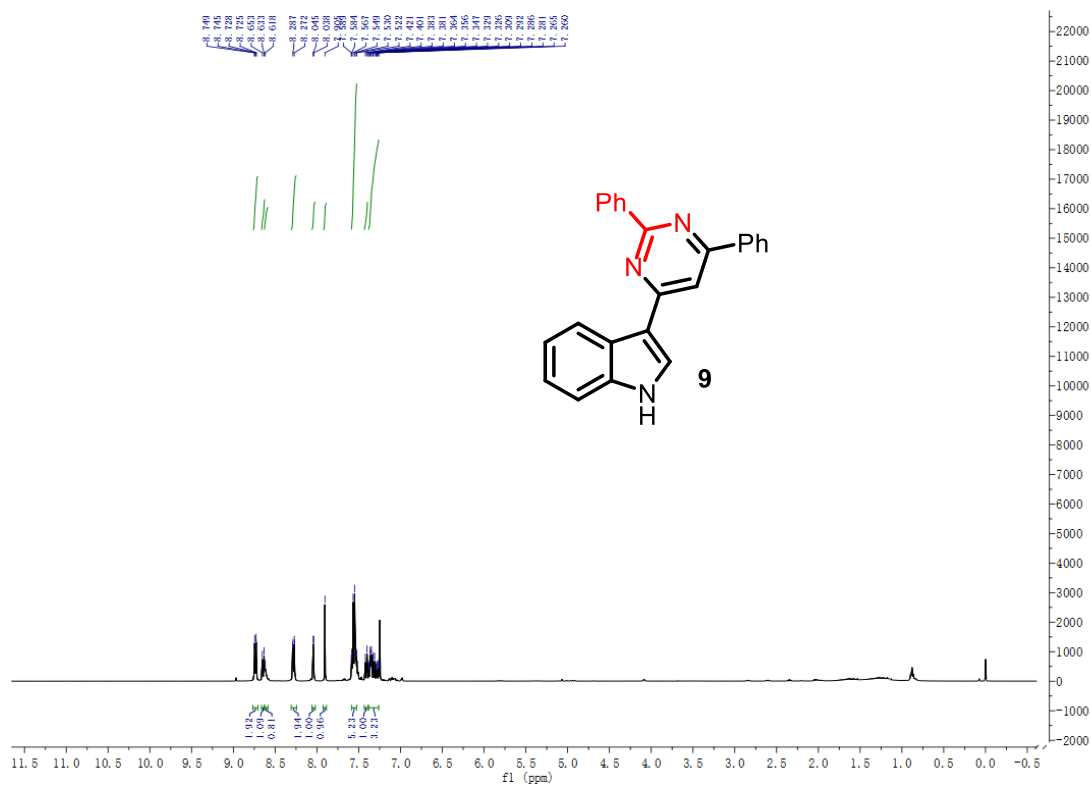


¹³C NMR (100 MHz, CDCl₃):

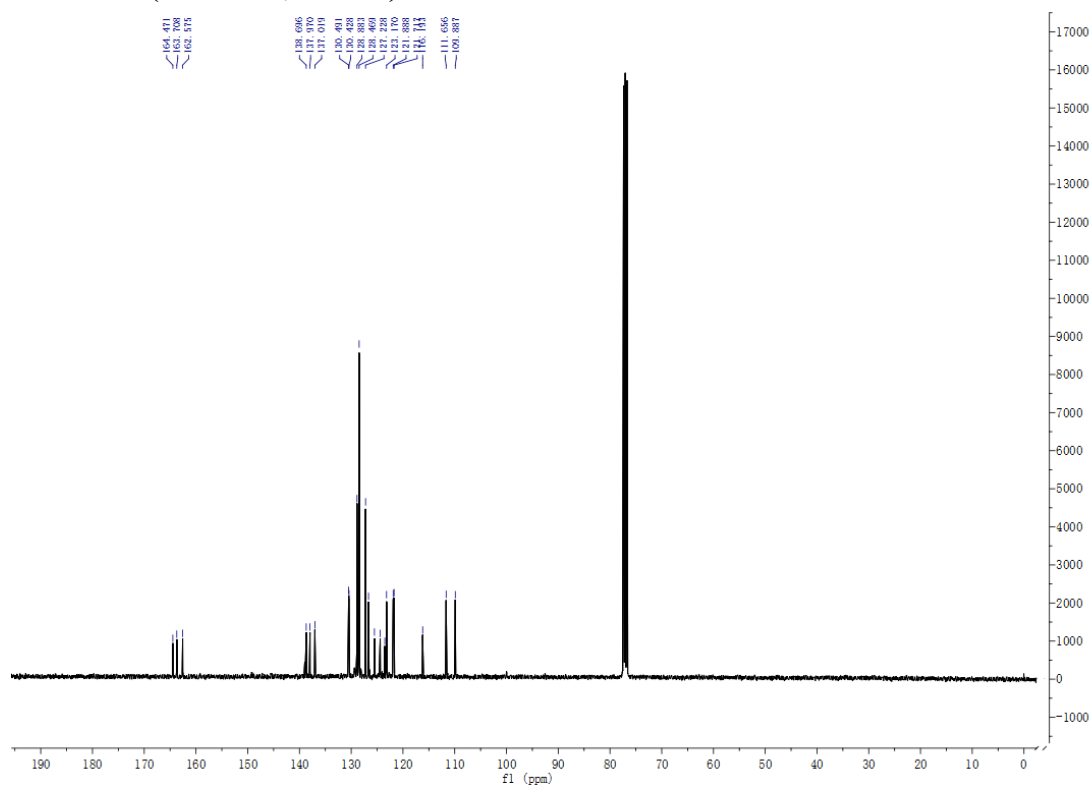


3-(2,6-diphenylpyrimidin-4-yl)-1H-indole (9)

¹H NMR (400 MHz, CDCl₃):

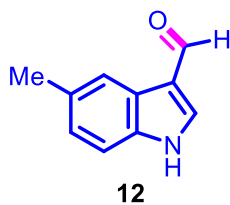
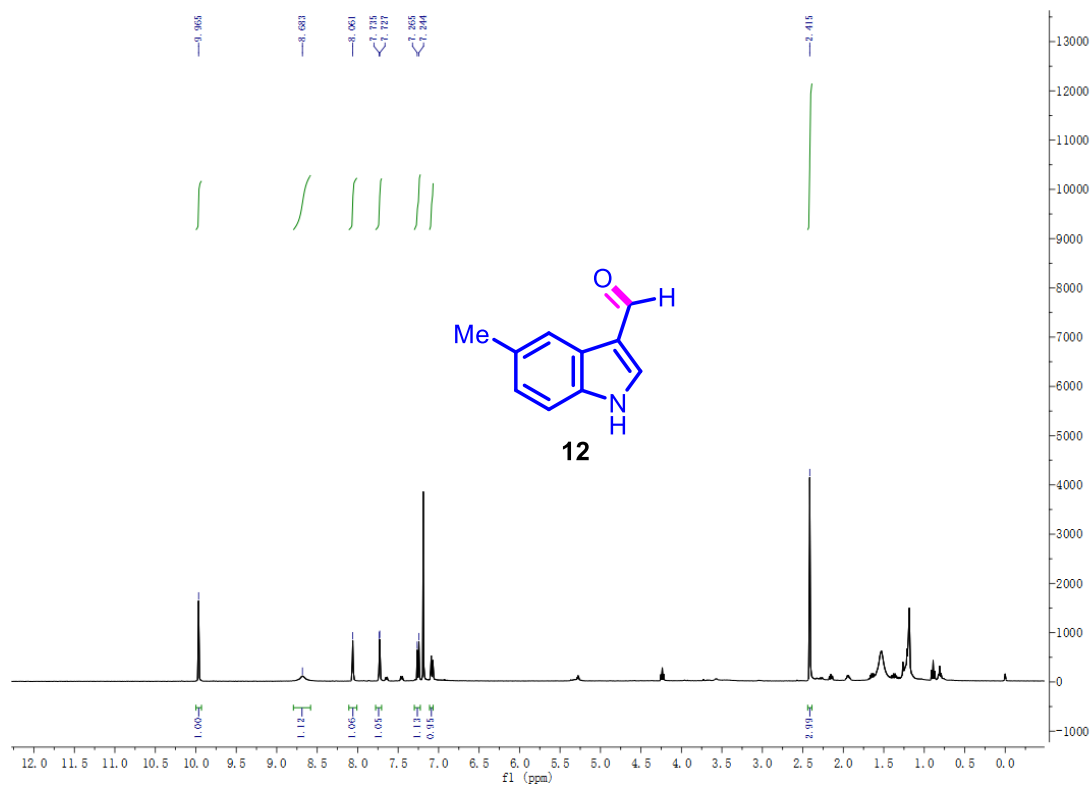


¹³C NMR (100 MHz, CDCl₃):

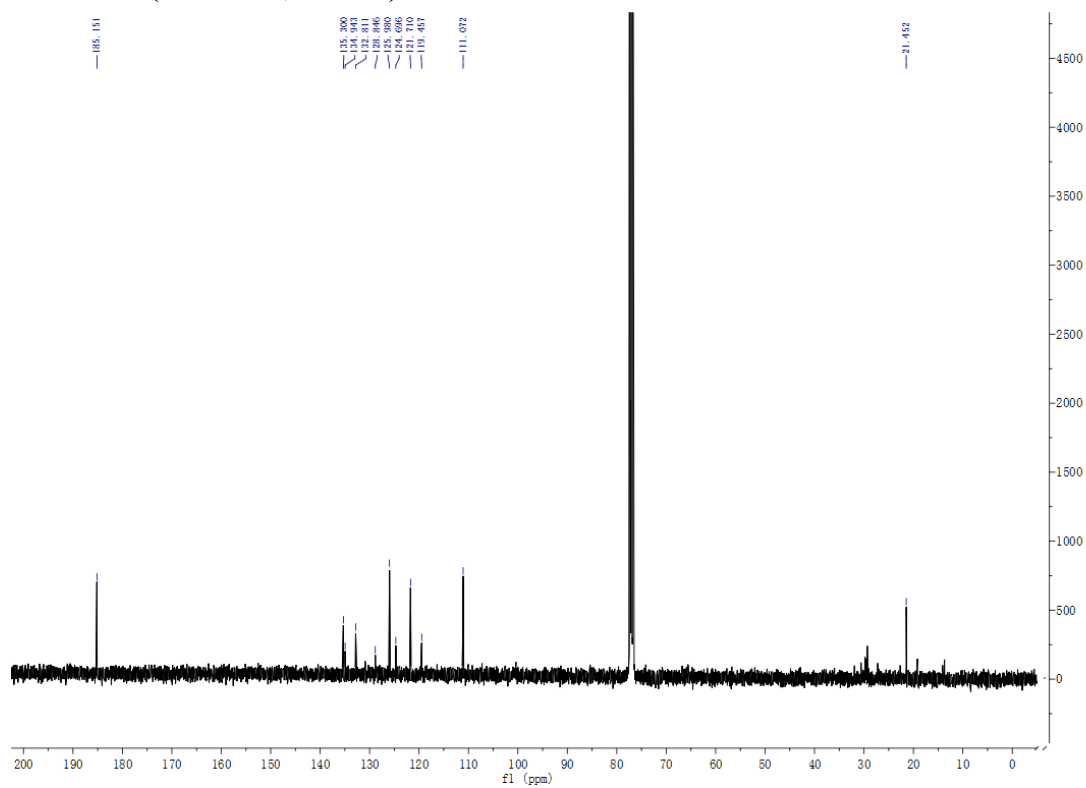


5-methyl-1H-indole-3-carbaldehyde (12)

¹H NMR (400 MHz, CDCl₃):

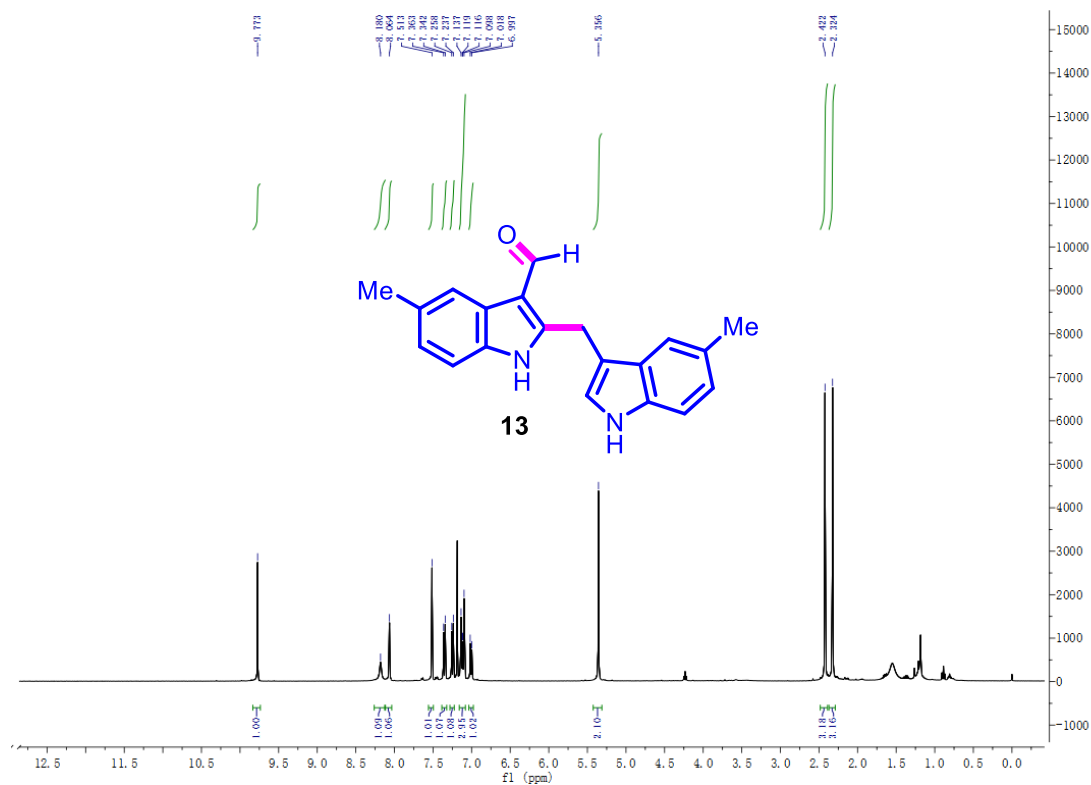


¹³C NMR (100 MHz, CDCl₃):

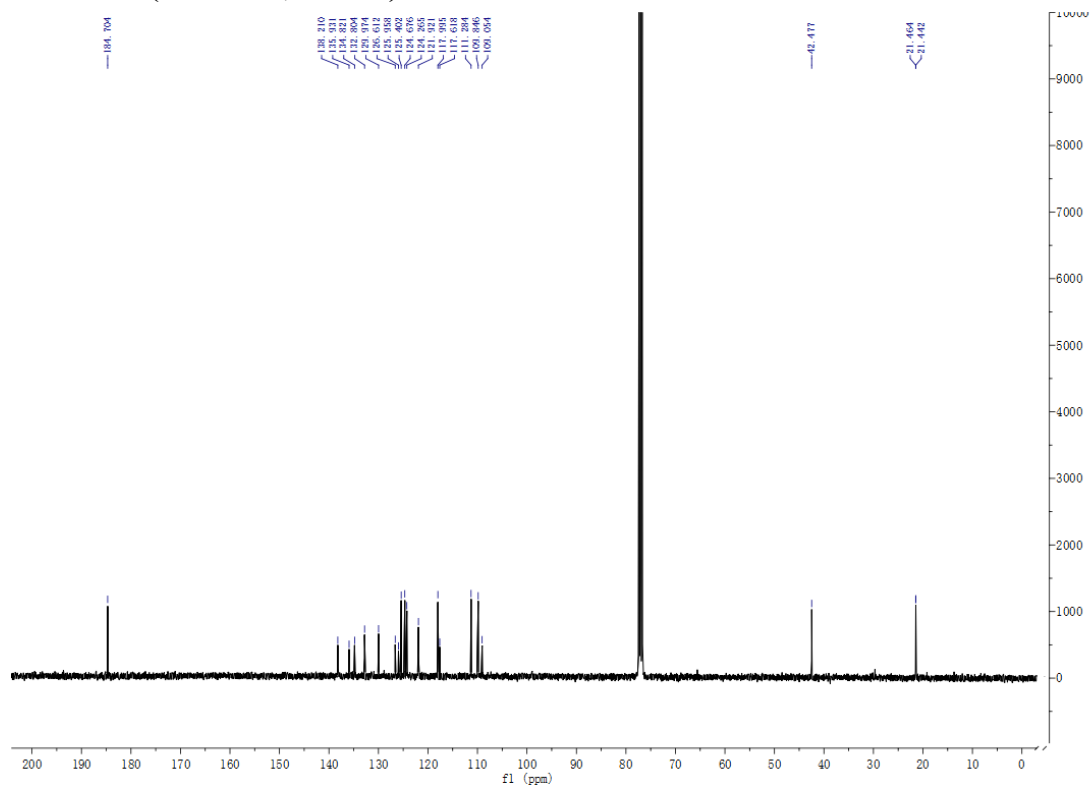


5-methyl-2-((5-methyl-1H-indol-3-yl)methyl)-1H-indole-3-carbaldehyde (13)

¹H NMR (400 MHz, CDCl₃):

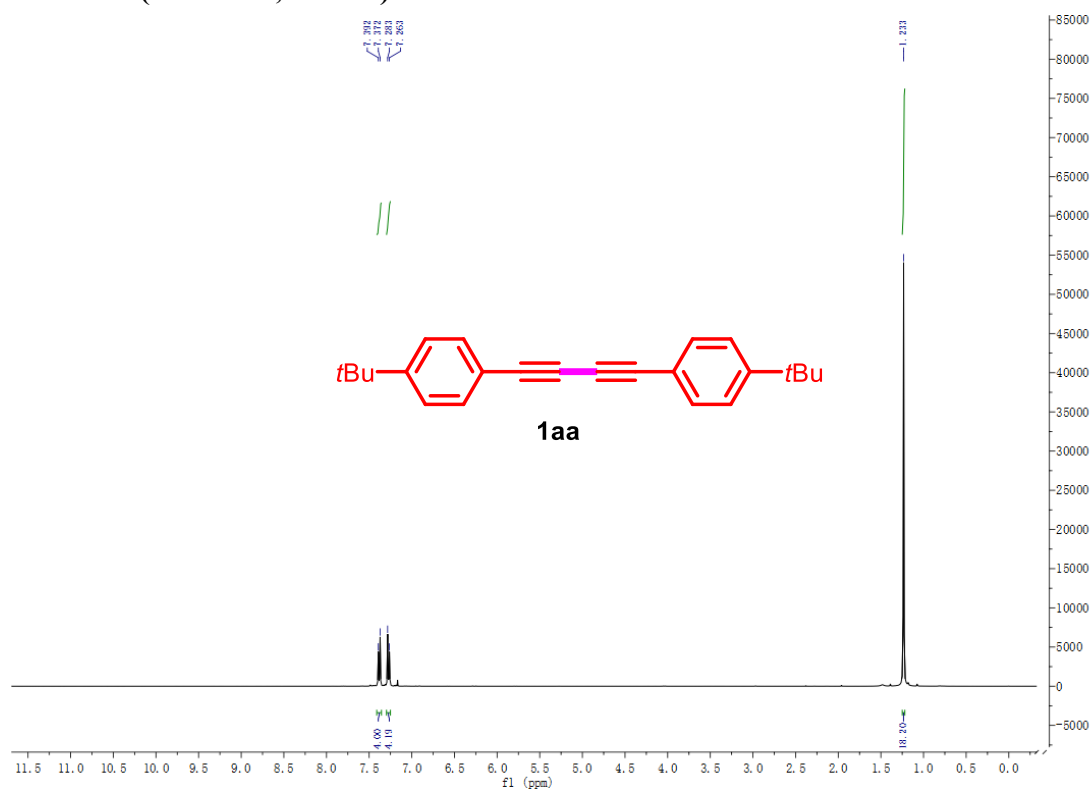


¹³C NMR (100 MHz, CDCl₃):

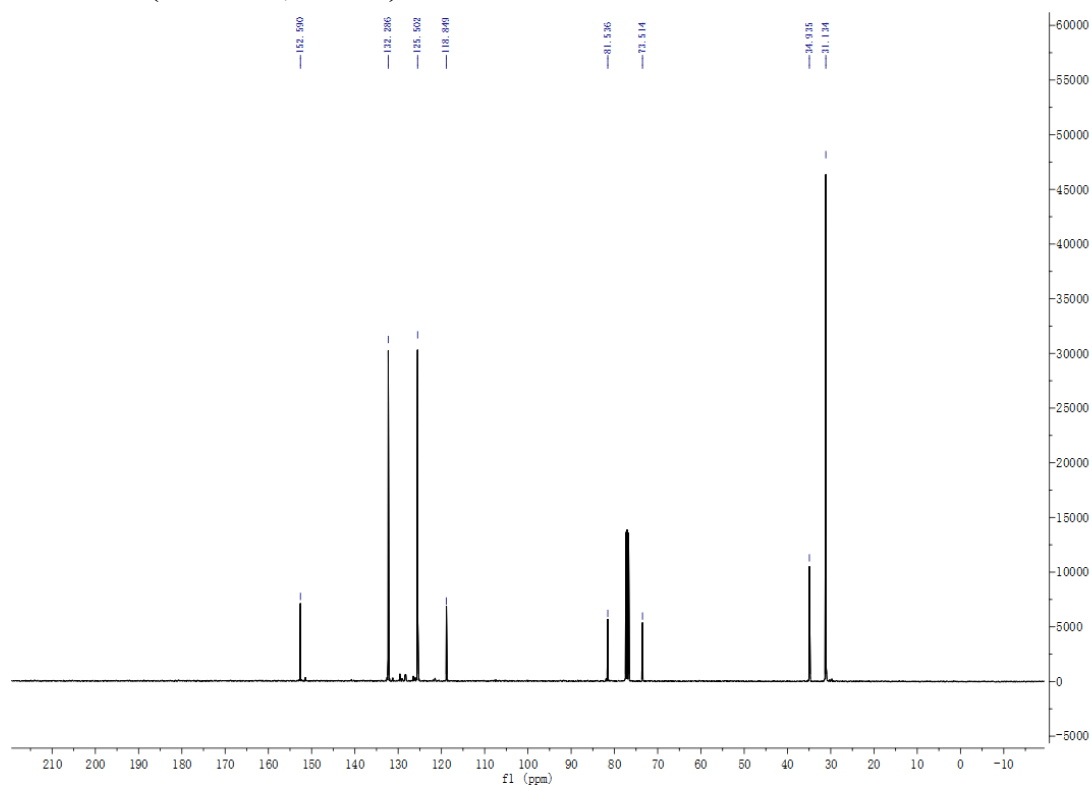


1,4-bis(4-(tert-butyl)phenyl)buta-1,3-diyne (1aa)

^1H NMR (400 MHz, CDCl_3):

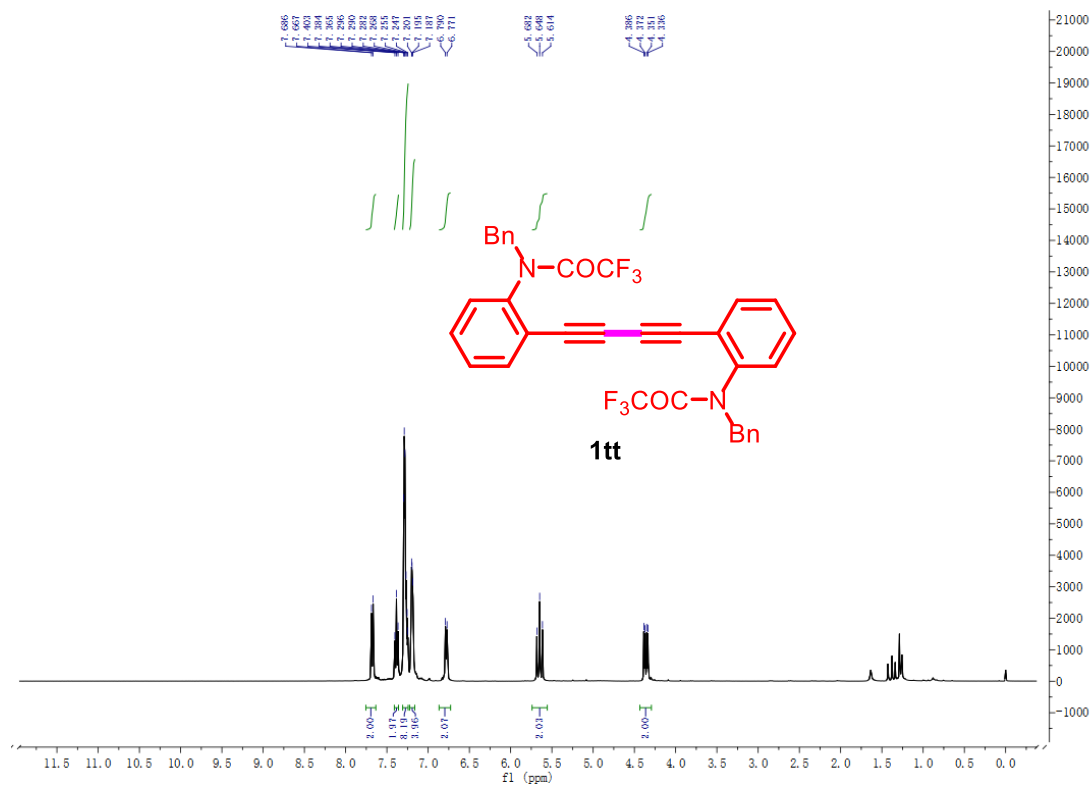


^{13}C NMR (100 MHz, CDCl_3):

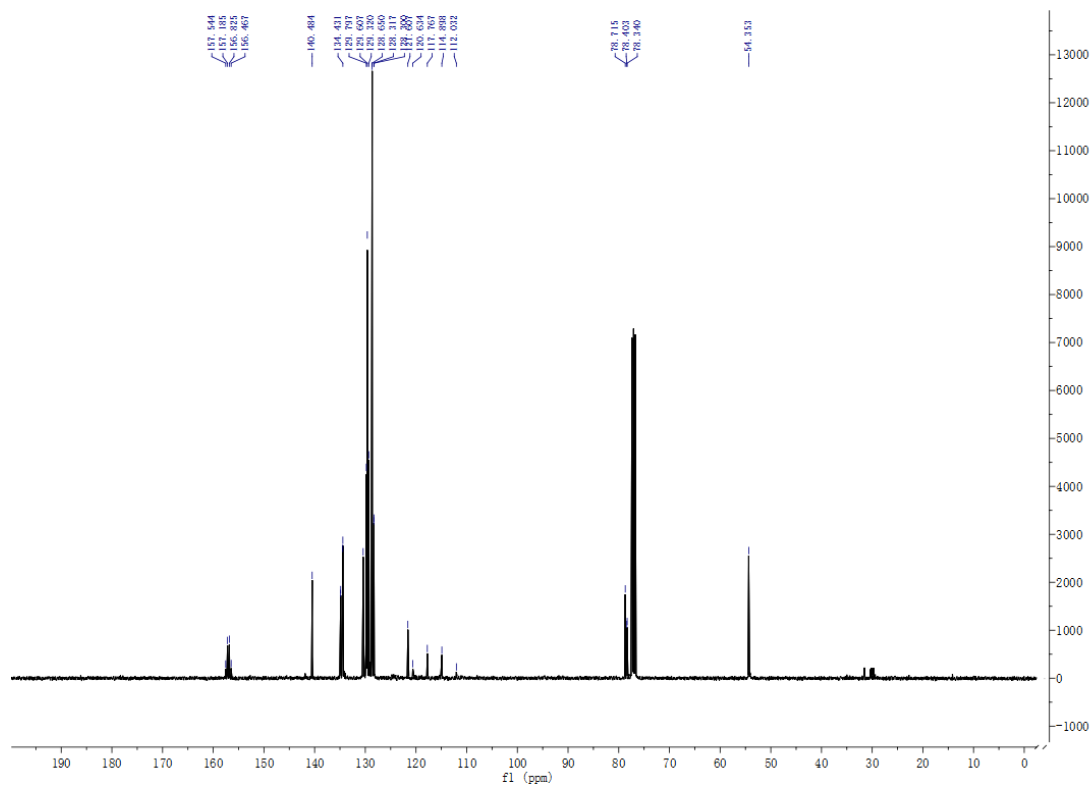


N,N'-(buta-1,3-diyne-1,4-diylbis(2,1-phenylene))bis(N-benzyl-2,2,2-trifluoroacetamide) (1tt)

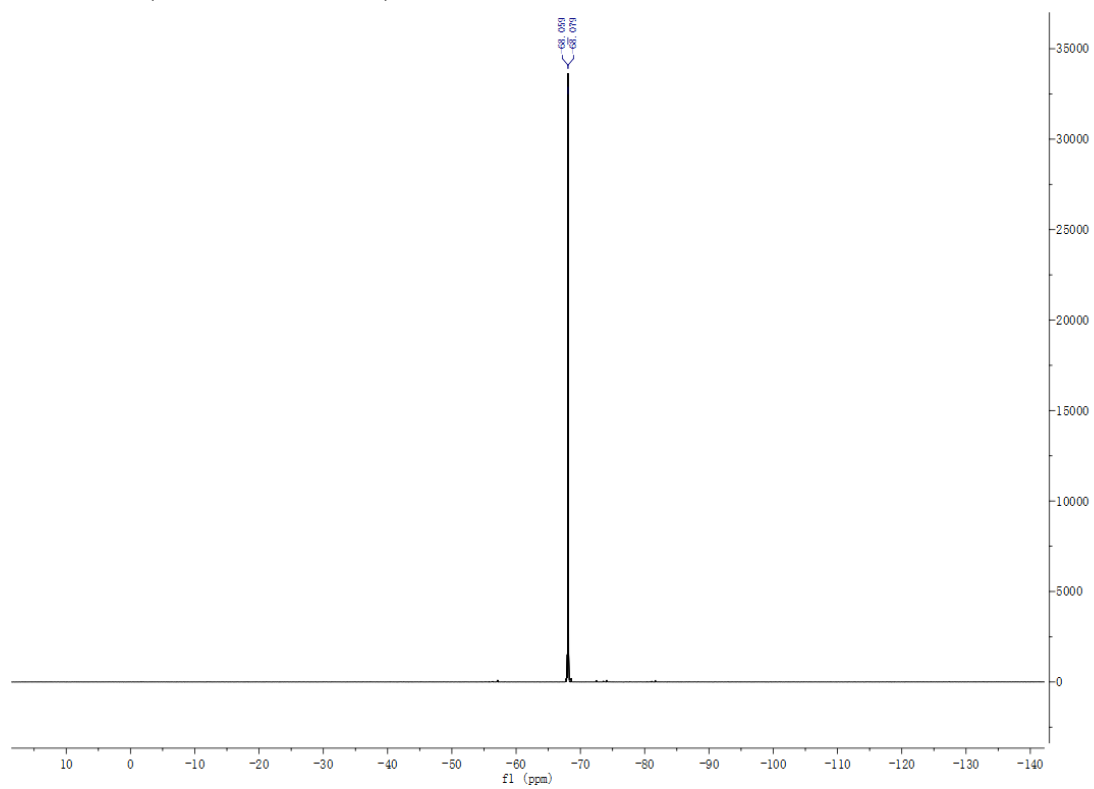
¹H NMR (400 MHz, CDCl₃):



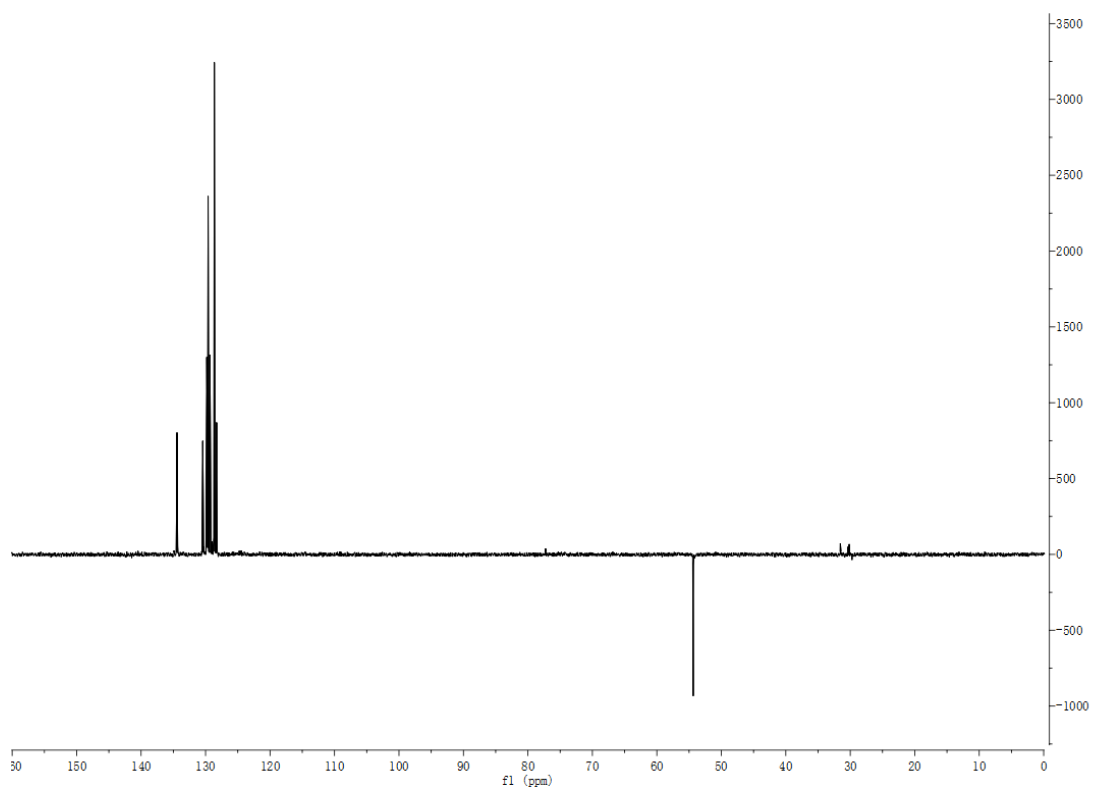
¹³C NMR (100 MHz, CDCl₃):



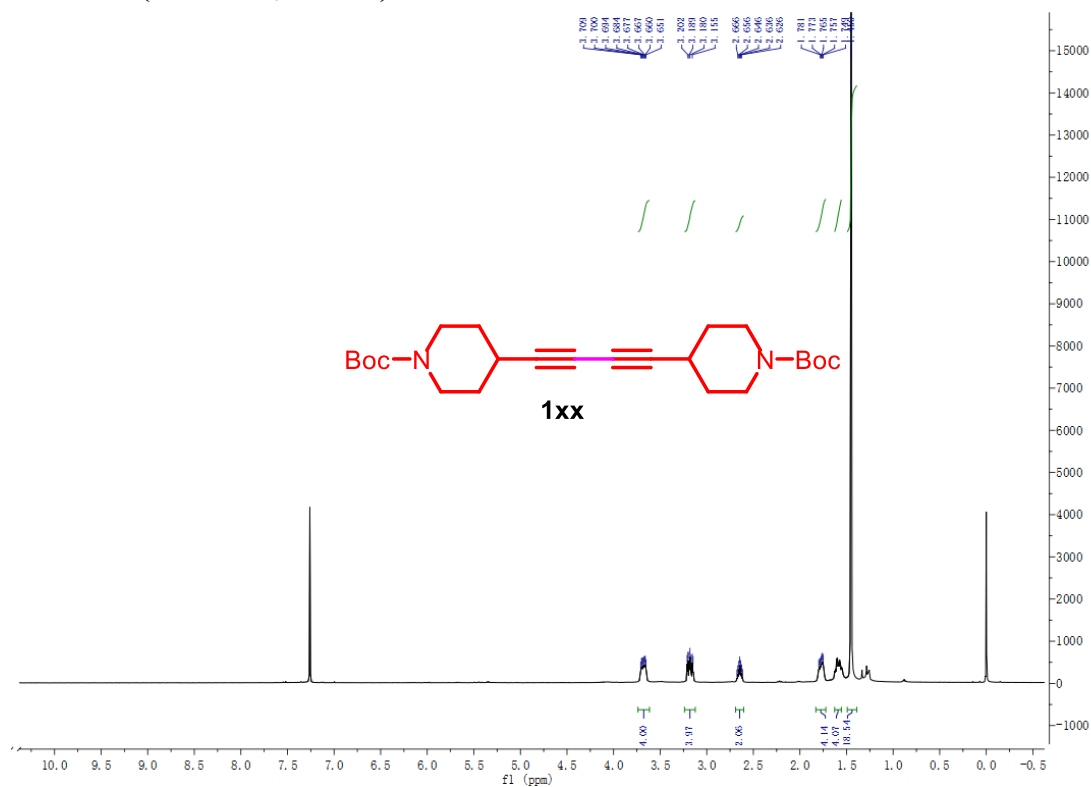
^{19}F NMR (376 MHz, CDCl_3):



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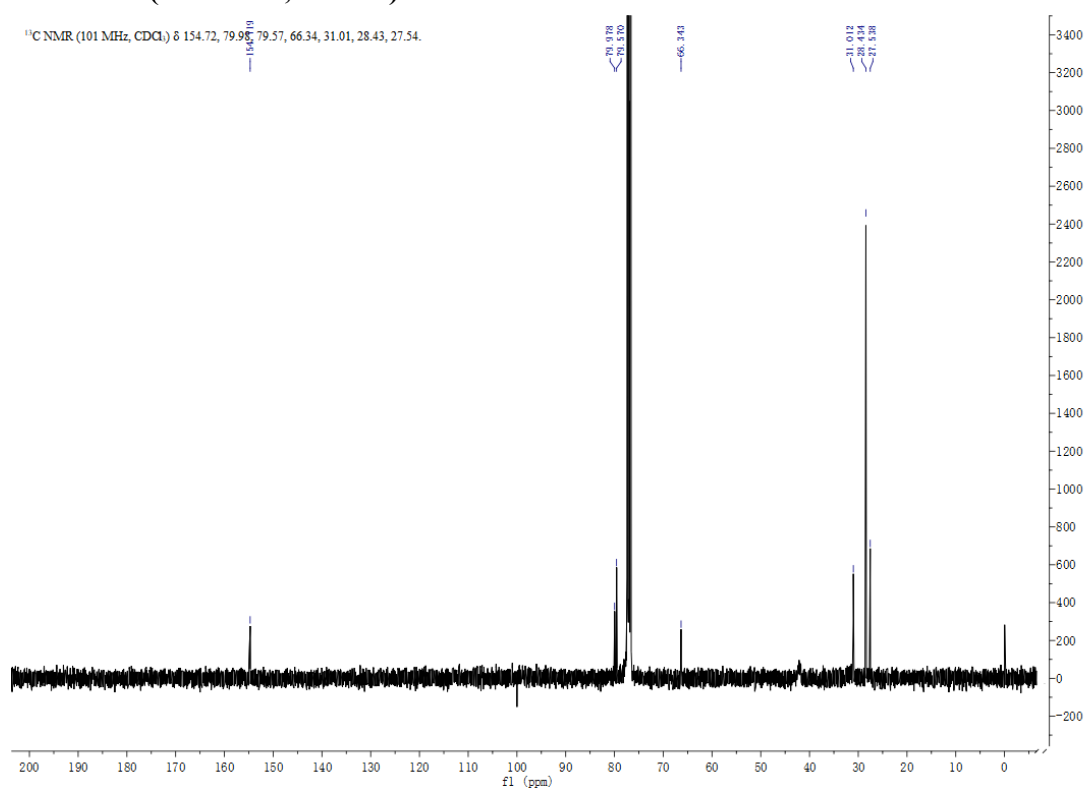


di-tert-butyl 4,4'-(buta-1,3-diyne-1,4-diyl)bis(piperidine-1-carboxylate) (1xx)
¹H NMR (400 MHz, CDCl₃):



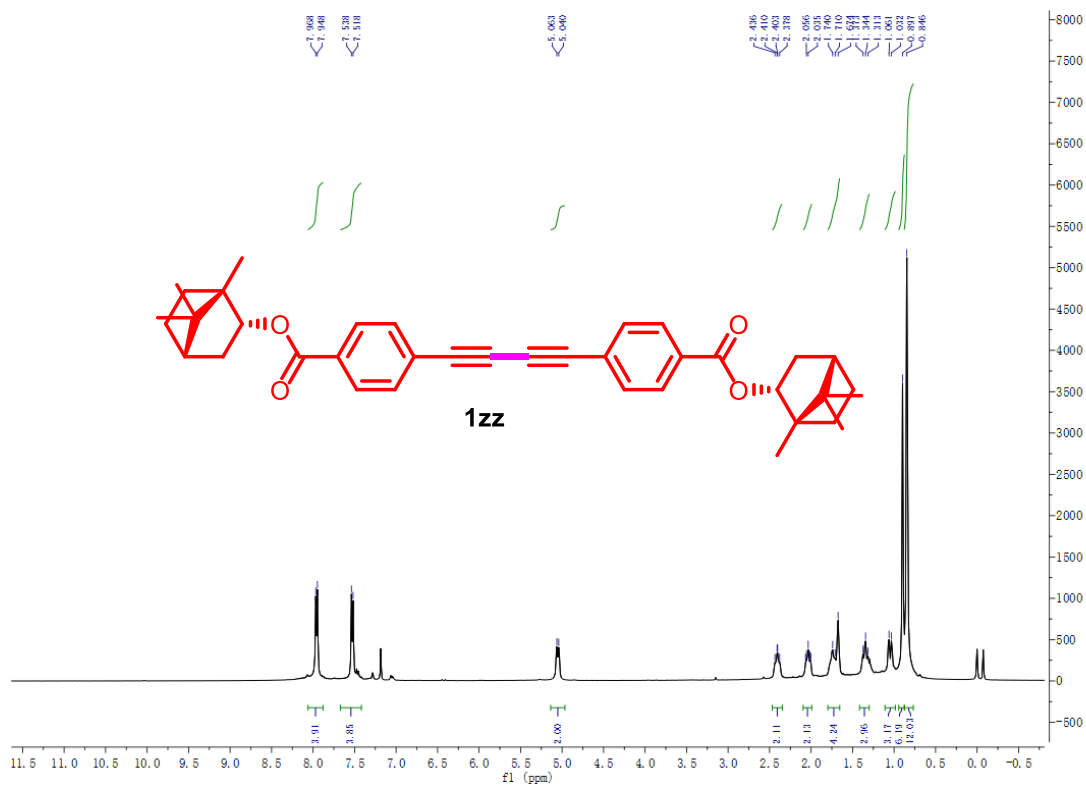
¹³C NMR (100 MHz, CDCl₃):

¹³C NMR (101 MHz, CDCl₃) δ 154.72, 79.95, 79.57, 66.34, 31.01, 28.43, 27.54.

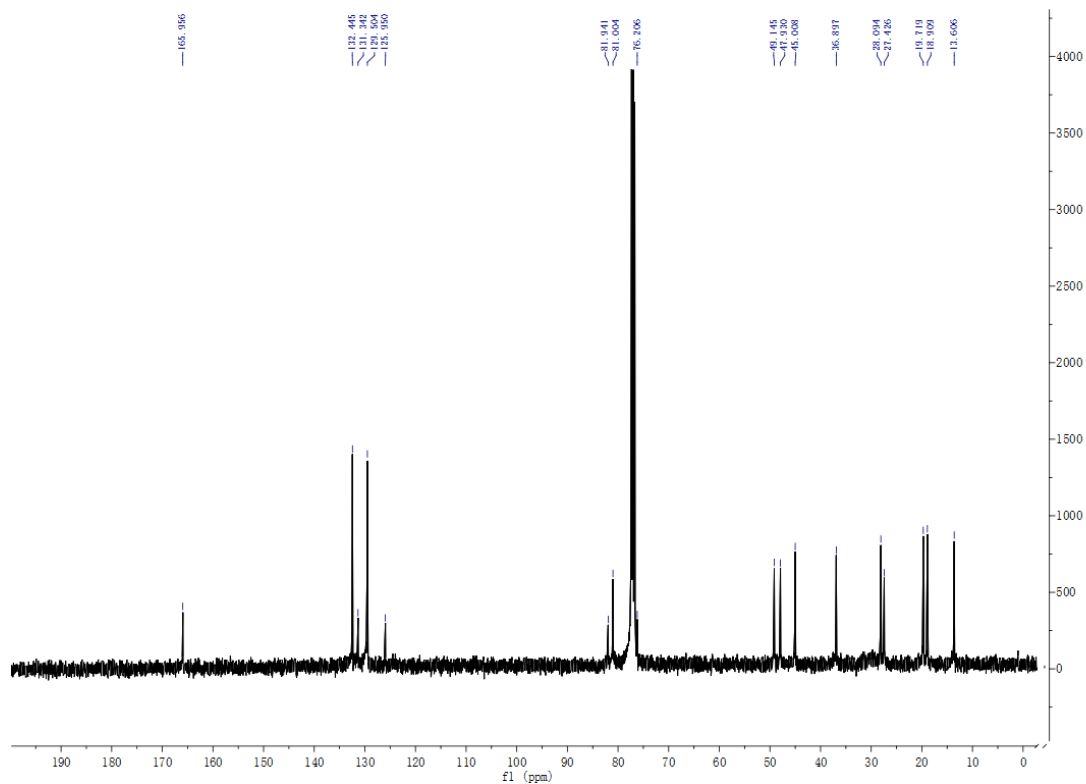


bis((1S,2R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl) 4,4'-(buta-1,3-diyne-1,4-diyl)dibenzoate (1zz)

¹H NMR (400 MHz, CDCl₃):



¹³C NMR (100 MHz, CDCl₃):



DEPT

