Pd/Norbornene-Catalyzed Sequential *ortho* C-H Alkylation and *ipso*-Alkynylation: A 1,1-Dimethyl-2-alkynol Strategy

Supporting Information

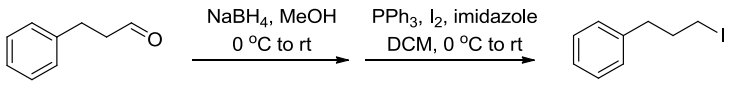
Fenggang Sun, Miao Li and Zhenhua Gu*

Department of Chemistry, University of Science and Technology of China, 96 Jinzhai Road, Hefei, Anhui, China, 230026
zhgu@ustc.edu.cn

General Information

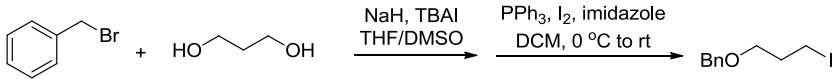
Nuclear magnetic resonance spectrometers were recorded on 400 MHz instruments internally referenced to the residue of $CDCl_3$ (7.26 ppm for ^1H NMR and 77.00 ppm for ^{13}C NMR) signal. All reactions were performed under an inert atmosphere of dry nitrogen in flame-dried glassware unless otherwise stated. Tetrahydrofuran, DME and 1,4-dioxane were distilled over sodium under an atmosphere of nitrogen. Toluene, dichloroethane and DMF were distilled over calcium hydride under an atmosphere of nitrogen. 1,1-dimethyl-2-alkynols[1] were synthesized according to the literature procedures.

Preparation of (3-iodopropyl)benzene



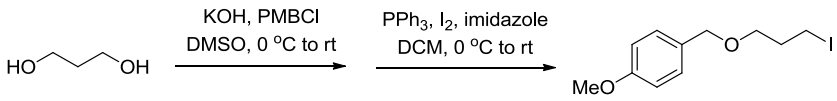
To a solution of phenylpropyl aldehyde (1.34 g, 10.0 mmol) in MeOH (50 mL) at 0 °C was added sodium borohydride (0.57 g, 15.0 mmol) in portions. The resulting mixture was stirred at 0 °C for 1 h before being quenched with water. The mixture was extracted with EtOAc three times and washed with brine (three times). The combined extracts were dried over anhydrous Na_2SO_4, filtrated. The solvent of filtration was evaporated to give a crude colorless oil.

To a solution of the above crude material in DCM (50 mL) was added PPh_3 (3.40 g, 13.0 mmol) and imidazole (0.96 g, 14.0 mmol), followed by the addition of iodine portionwise (3.80 g, 15.0 mmol). The reaction was stirred at rt for 24 h and then quenched with the saturated aqueous Na_2SO_3 solution. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether) to afford the product 1.82g (74%). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.35-7.27 (m, 2 H), 7.25-7.16 (m, 3 H), 3.18 (t, *J* = 6.8 Hz, 2 H), 2.74 (t, *J* = 7.2 Hz, 2 H), 2.22-2.06 (m, 2 H).



To a solution of propane-1,3-diol (7.60 g, 100 mmol) in 200 mL of THF was added NaH (4.40 g, 60% dispersion in mineral oil, 110 mmol) portionwise. The mixture was stirred at rt for 30 min, and then benzyl bromide (18.90 g, 110 mmol) was added dropwise, followed by tetrabutylammonium iodide (7.40 g, 20 mmol) in one portion. The mixture was heated to 60 ℃ and stirred overnight. After cooling, an equal volume of water was added, and the mixture was extracted with diethyl ether. The combined organic layer was washed with brine, dried over Na_2SO_4, and filtrated. The solvent was evaporated, and the resulting residue was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 5:1) to yield 3-(benzyloxy)propan-1-ol 12.08 g (73%).

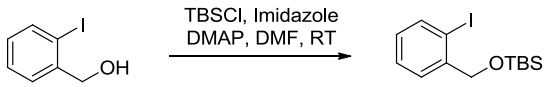
To a solution of the above alcohol in DCM (140 mL) was added PPh_3 (8.40 g, 32.2 mmol) and imidazole (2.50 g, 36.8 mmol), followed by the addition of iodine portionwise (8.54 g, 33.7 mmol). The reaction was stirred at rt for 24 h and then quenched with the aqueous Na_2SO_3 solution. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer was dried with Na_2SO_4 and filtered. The solvent was evaporated, and the resulting residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 30 : 1) to afford the product 5.57 g (66%). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.40-7.25 (m, 5H), 4.53 (s, 2 H), 3.55 (t, *J* = 6.0 Hz, 2 H), 3.31 (t, *J* = 6.8 Hz, 2 H), 2.16-2.03 (m, 2 H).



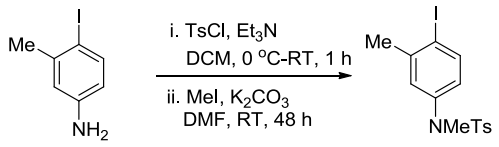
To a solution of propane-1,3-diol (9.24 g, 121.4 mmol) in 60 mL anhydrous DMSO was added KOH (6.8 g, 121.4 mmol) portionwise at 0 °C and the mixture was stirred until the solution turned clear again, then it was treated with PMBCl (9.52 g, 60.8 mmol). The mixture was stirred for 4 h at rt. The mixture was diluted with 30 mL of Et_2O and carefully quenched by the addition of 5 M HCl (24 mL) at 0°C. Afterwards, the mixture was separated, and the aqueous layer was extracted with Et_2O. The combined organic layer was dried over Na_2SO_4 and evaporated. The residue was purified by flash column chromatography on silica gel (petroleum ether/ ethyl acetate = 10/1 → 1/1) to afford 3-((4-methoxybenzyl)oxy)propan-1-ol 7.84 g (66%).

To a solution of the above alcohol (3.0 g, 15.3 mmol) in DCM (70 mL) was added PPh_3 (4.20 g, 16.1 mmol) and imidazole (1.25 g, 18.4 mmol), followed by the addition of iodine portionwise (4.27 g, 16.8 mmol). The reaction was stirred at rt for 24 h and washed with the saturated aqueous Na_2SO_3 solution. The organic layer was dried with Na_2SO_4, filtered and concentrated under reduced pressure to give the crude material which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20: 1) to afford the product 3.51 g (75%). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.28-7.21 (m, 2 H), 6.91-6.83 (m, 2 H), 4.44 (s, 2 H), 3.79 (s, 3 H), 3.50 (t, *J* = 6.0 Hz, 2 H), 3.28 (t, *J* = 6.8 Hz, 2 H), 2.13-2.01 (m, 2 H).

Preparation of *tert*-butyl((2-iodobenzyl)oxy)dimethylsilane



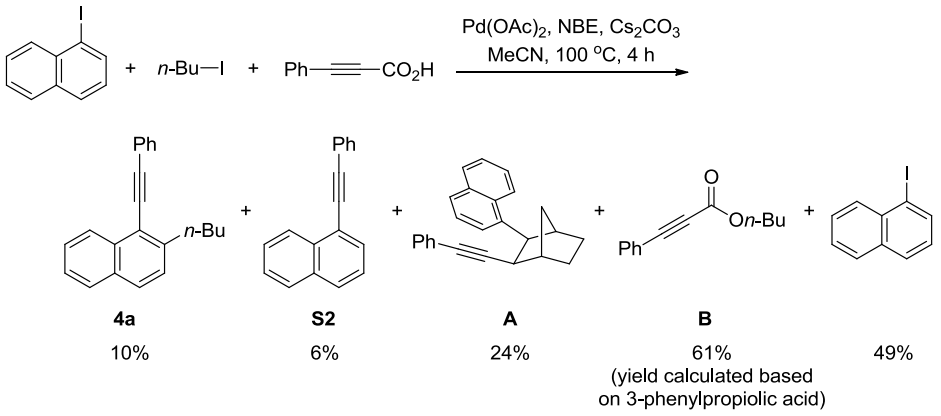
To a solution of 2-iodobenzyl alcohol (0.47 g, 2.0 mmol) in dry DMF (5 mL) was successively added imidazole (0.20 g, 3.0 mmol), DMAP (24.6 mg, 0.20 mmol) and TBSCl (0.47 g, 3.1 mmol). The reaction mixture was stirred at rt overnight before being quenched with H_2O. The resulting mixture was etracted with EtOAc, and the combined organic layer was washed with brine and dried over Na_2SO_4, and filtrated.The solvent was evaporated and the resulting residue was purified by column chromatography on silica gel (petroleum ether / ethyl acetate = 100:1) to afford the product 0.62 g (89%). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.78 (d, *J* = 8. 0 Hz, 1 H), 7.53 (d, *J* = 8.0 Hz, 1 H), 7.38 (t, *J* = 8.0 Hz, 1 H), 6.97 (t, *J* = 8.0 Hz, 1 H), 4.64 (s, 2 H), 0.99 (s, 9 H), 0.15 (s, 6 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.8, 138.6, 128.5, 128.1, 127.3, 95.8, 69.4, 25.9, 18.4, -5.3.



To a stirred solution of 4-iodo-3-methylaniline (0.23 g, 1.00 mmol) and Et_3N (0.21 mL, 0.15 g, 1.50 mmol) in dry DCM at 0 ^{o}C was added *p*-toluenesulfonyl chloride (0.23 g, 1.20 mmol) portion wise and the reaction mixture was stirred at room temperature for 1 h. The reaction mixture was acidified by the addition of 1 M HCl and the aqueous layer was extracted with DCM (2 x 10 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtrated. The solvent was evaporated to afford crude sulfonamide, which was used in next step without further purification.

To a solution of the above crude product in DMF (2 mL), MeI (0.14 g, 0.98 mmol, 2 equiv) and K_2CO_3 (0.21 g, 1.47 mmol, 3 equiv) were added and stirred for 48 h at room temperature. The mixture was diluted with ethyl ether followed by 1 M HCl. The mixture was extracted with diethyl ether, and the combined organic phase was washed with brine, dried over anhydrous Na_2SO_4. After filtration, the solvent was evaporated, and the residue was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 5:1) to yield the desired product 0.15 g (76%). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.69 (d, *J* = 8. 0 Hz, 2 H), 7.46-7.42 (m, 2 H), 7.27-7.24 (m, 2 H), 7.06 (d, *J* = 4.0 Hz, 1 H), 6.55 (dd, *J* = 8.0, 4.0 Hz, 1 H), 3.11 (s, 3 H), 2.42 (s, 3 H), 2.38 (s, 3 H);

The trial for *ortho*-alkylation/*ipso*-alkynylation with 3-phenylpropiolic acid



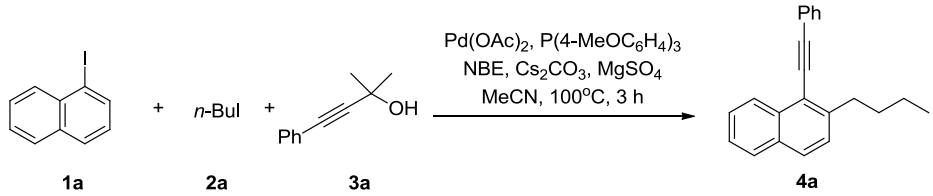
A dried Schlenk tube was charged with 1-iodonaphthalene (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane (73.6 mg, 0.40 mmol, 2.0 equiv), phenylpropiolic acid (43.6 mg, 0.30 mmol, 1.5 equiv), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol, 5.0 mol %), PPh_3 (6.6 mg, 0.025, 12.5 mol %), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv), Cs_2CO_3 (0.196 g, 0.60 mmol, 3.0 equiv) and MeCN (4.0 mL). After stirring at 100 ℃ for 4 h, the reaction mixture was cooled to room temperature and filtered through a pad of celite with EtOAc as the eluent. The filtrate was evaporated under reduced pressure, and the residue was purified by column chromatography on silica gel (petroleum ether / ethyl acetate) to give the corresponding product **A** (15.3 mg, 24%), **B** (37.3 mg, 61%), **4a** (5.7 mg, 10%), **S2** (2.6 mg, 6.0%), as well as the starting material 1-iodonaphthalene (25.1 mg, 49%).

A: **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.07 (d, *J* = 8.4 Hz, 1 H), 7.85 (d, *J* = 8.0 Hz, 1 H), 7.73 (d, *J* = 8.0Hz, 1 H), 7.54-7.40 (m, 4 H), 7.05-6.99 (m, 1 H), 6.96 (t, *J* = 7.6 Hz, 2 H), 6.37 (d, *J* = 8.0 Hz, 2 H), 3.71 (d, *J* = 8.8Hz, 1 H), 3.34 (d, *J* = 8.8 Hz, 1 H), 2.87 (s, 1 H), 2.61 (s, 1 H), 2.21 (d, *J* = 10.0 Hz, 1 H), 1.78 (d, *J* = 8.4Hz, 2 H), 1.57-1.47 (m, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.6, 133.6, 133.0, 131.0, 128.5, 127.6, 126.8, 126.3, 125.5, 125.20, 125.15, 124.1, 123.6, 123.1, 91.4, 83.3, 48.0, 44.4, 42.7, 40.5, 36.4, 30.9, 28.4. HRMS (EI) calcd for $C_{25}H_{22}$ $[M]^+$: 322.1722, found 322.1725.

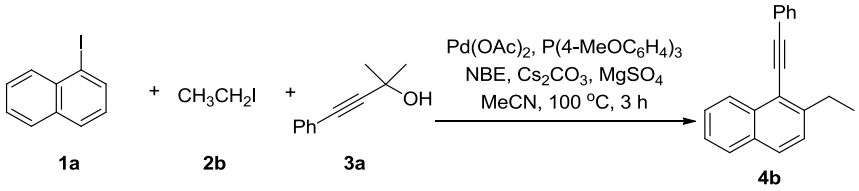
B: **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.65-7.54 (m, 2 H), 7.49-7.42 (m, 1 H), 7.41-7.33 (m, 2 H), 4.24 (t, *J* = 6.8Hz, 2 H), 1.76-1.64 (m, 2 H), 1.51-1.36 (m, 2 H), 0.96 (t, *J* = 7.2Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 154.2, 133.0, 130.6, 128.5, 119.7, 86.0, 80.7, 65.9, 30.5, 19.0, 13.6.

General procedure for the *ortho*-alkylation/*ipso*-alkynylation reaction

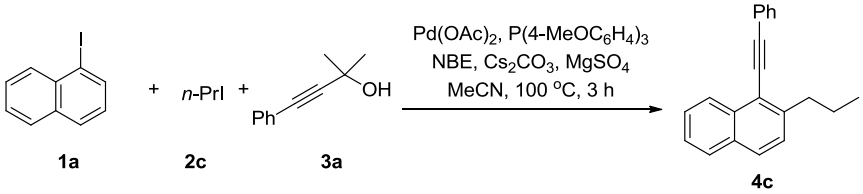
A dried Schlenk tube was charged with aryl iodide **1** (0.20 mmol, 1.0 equiv), alkyl iodide **2** (0.40 mmol, 2.0 equiv), 1,1-dimethyl-2-alkynol **3** (0.30 mmol, 1.5 equiv), $Pd(OAc)_2$ (2.3 mg, 0.010 mmol, 5.0 mol %), P(*p*-$MeOC_6H_4)_3$ (8.8 mg, 0.025 mmol, 12.5 mol %), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv), Cs_2CO_3 (0.196 g, 0.60 mmol, 3.0 equiv), $MgSO_4$ (0.150 g) and MeCN (4.0 mL). The reaction mixture was stirred at 100 ℃ for 3 h then cooled to room temperature and filtered through a pad of celite with EtOAc as the eluent. The filtrate was evaporated under reduced pressure, and the residue was purified by column chromatography on silica gel to give the corresponding product **4**.



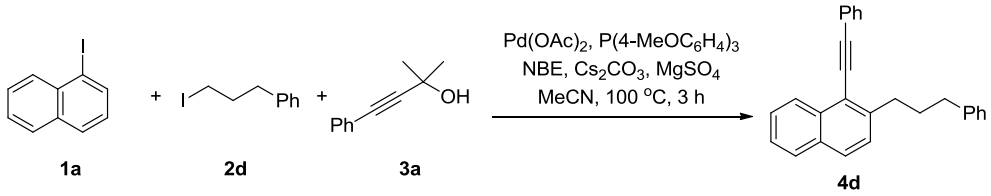
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4a** (50.0 mg, 88%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.49 (d, *J* = 8.4 Hz, 1 H), 7.84 (d, *J* = 8.0 Hz, 1 H), 7.80 (d, *J* = 8.4 Hz, 1 H), 7.68 (d, *J* = 7.6 Hz, 2 H), 7.64-7.57 (m, 1 H), 7.61 (t, *J* = 7.2 Hz, 1 H), 7.46-7.37 (m, 4 H), 3.12 (t, *J* = 8.0 Hz, 2 H), 1.86-1.74 (m, 2 H), 1.54-1.44 (m, 2 H), 1.02 (t, *J* = 8.0 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.1, 133.6, 131.6, 131.5, 128.4, 128.3, 128.2, 128.0, 127.3, 126.7, 126.1, 125.5, 123.8, 118.9, 98.1, 86.4, 35.1, 33.1, 22.7, 14.1. HRMS (EI) calcd for $C_{22}H_{20}$ $[M]^+$: 284.1565, found 284.1567.



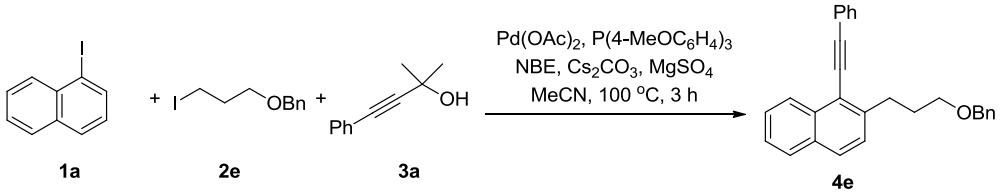
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-iodoethane **2b** (62.4 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4b** (43.4 mg, 85%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.53-8.45 (m, 1 H), 7.83 (dd, *J* = 16.8, 8.4 Hz, 2 H), 7.73-7.66 (m, 2 H), 7.64-7.57 (m, 1 H), 7.54-7.47 (m, 1 H), 7.47-7.37 (m, 4 H), 3.19-3.07 (m, 2 H), 1.47-1.35 (m, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.3, 133.6, 131.7, 131.5, 128.5, 128.4, 128.2, 128.0, 126.72, 126.67, 126.1, 125.5, 123.8, 118.5, 98.2, 86.2, 28.6, 15.1. HRMS (ESI) calcd for $C_{20}H_{17}$ $[M+H]^+$: 257.1327, found 257.1330.



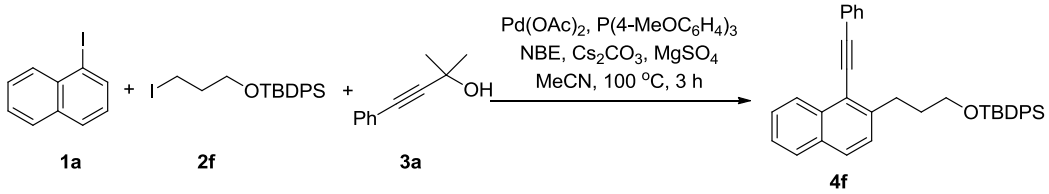
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-iodopropane **2c** (68.0 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4c** (45.6 mg, 84%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.50 (d, *J* = 8.4 Hz, 1 H), 7.85 (d, *J* = 8.0 Hz, 1 H), 7.79 (d, *J* = 8.4 Hz, 1 H), 7.69 (dd, *J* = 8.0, 1.2 Hz, 2 H), 7.65-7.57 (m, 1 H), 7.54-7.47 (m, 1 H), 7.47-7.37 (m, 4 H), 3.10 (t, *J* = 8.0 Hz, 2 H), 1.93-1.79 (m, 2 H), 1.08 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.9, 133.6, 131.7, 131.5, 128.4, 128.23, 128.21, 128.0, 127.4, 126.7, 126.1, 125.5, 123.8, 119.0, 98.2, 86.4, 37.4, 24.1, 14.1. HRMS (ESI) calcd for $C_{21}H_{19}$ $[M+H]^+$: 271.1485, found 271.1487.



The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), (3-iodopropyl)benzene **2d** (98.4 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4d** (64.7 mg, 93%) (petroleum ether/ ethyl acetate =100 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.49 (d, *J* = 8.4 Hz, 1 H), 7.85 (d, *J* = 8.0 Hz, 1 H), 7.80 (d, *J* = 8.8 Hz, 1 H), 7.65-7.57 (m, 3 H), 7.51 (t, *J* = 7.4 Hz, 1 H), 7.46-7.37 (m, 4 H), 7.32-7.17 (m, 5 H), 3.16 (t, *J* = 8.0 Hz, 2 H), 2.80 (t, *J* = 8.0 Hz, 2 H), 2.22-2.09 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.5, 142.2, 133.6, 131.7, 131.5, 128.5, 128.41, 128.35, 128.3, 128.2, 128.0, 127.3, 126.8, 126.1, 125.7, 125.6, 123.7, 119.0, 98.3, 86.2, 35.9, 35.1, 32.4. HRMS (EI) calcd for $C_{27}H_{22}$ $[M]^+$: 346.1722, found 346.1728.

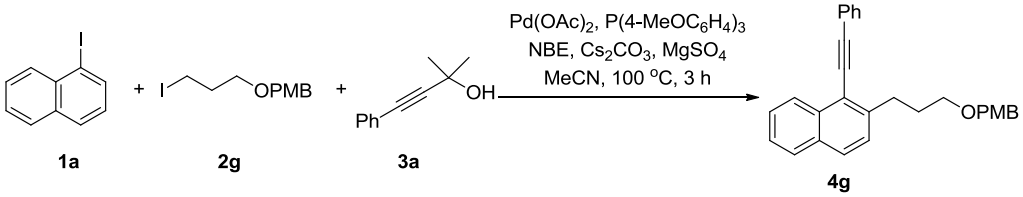


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), [(3-iodopropoxy)methyl]benzene **2e** (0.110 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4e** (64.1 mg, 82%) (petroleum ether/ ethyl acetate = 50 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.48 (dd, *J* = 8.4, 0.8 Hz, 1 H), 7.85 (d, *J* = 8.0 Hz, 1 H), 7.78 (d, *J* = 8.0 Hz, 1 H), 7.67-7.63 (m, 2 H), 7.63-7.58 (m, 1 H), 7.53-7.48 (m, 1 H), 7.43-7.29 (m, 9 H), 4.55 (s, 2 H), 3.61 (t, *J* = 6.4 Hz, 2 H), 3.23 (t, *J* = 7.6 Hz, 2 H), 2.21-2.09 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ.143.2, 138.6, 133.6, 131.7, 131.5, 128.4, 128.33, 128.31, 128.2, 128.0, 127.6, 127.44, 127.38, 126.8, 126.1, 125.6, 123.6, 119.1, 98.4, 86.1, 72.9, 69.8, 32.1, 30.7. HRMS (ESI) calcd for $C_{28}H_{25}O$ $[M+H]^+$: 377.1900, found 377.1905.

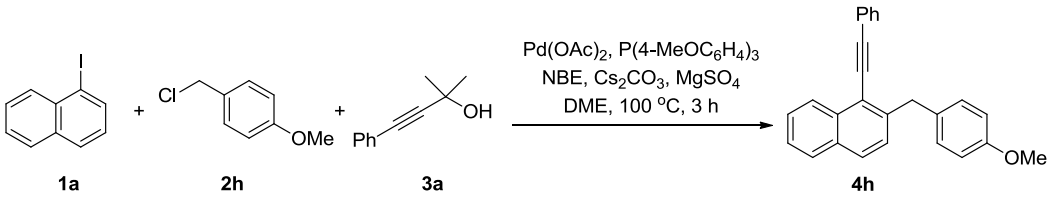


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), O-TBDPS-3-iodopropan-1-ol **2e** (0.170 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4f** (89.4 mg, 91%) (petroleum ether/ ethyl acetate = 50 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.46 (d, *J* = 8.4 Hz, 1 H), 7.84 (d, *J* = 8.0 Hz, 1 H), 7.77 (d, *J* = 8.4 Hz, 1 H), 7.73-7.68 (m, 4 H), 7.64-7.56 (m, 3 H), 7.53-7.47 (m, 1 H), 7.42-7.33 (m, 10 H), 3.82 (t, *J* = 6.4 Hz, 2 H), 3.22 (t, *J* = 7.2 Hz, 2 H), 2.14-2.02 (m, 2 H), 1.12-1.05 (m, 9 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.4, 135.6, 134.0, 133.6, 131.7, 131.5, 129.5, 128.4, 128.3, 128.2, 128.0, 127.6, 127.4,

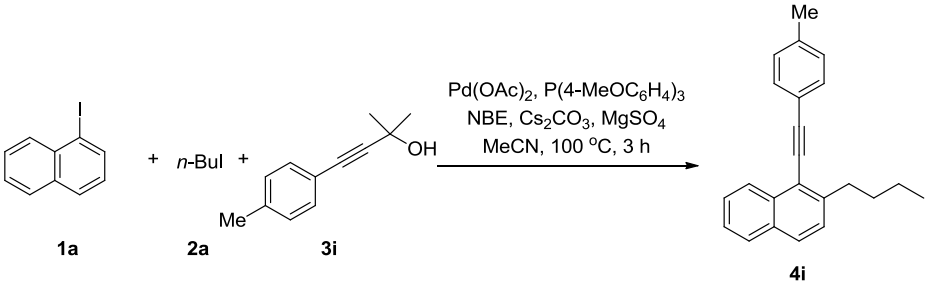
126.7, 126.1, 125.6, 123.7, 119.0, 98.4, 86.1, 63.6, 33.5, 31.9, 26.9, 19.2. HRMS (ESI) calcd for $C_{37}H_{37}OSi$ $[M+H]^+$: 525.2612, found 525.2614.



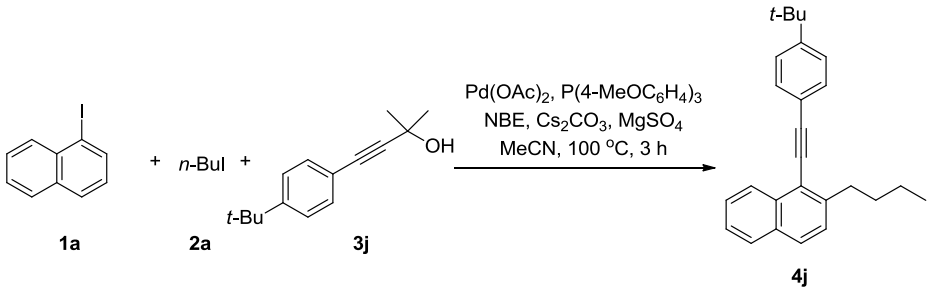
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4g** (68.5 mg, 84%) (petroleum ether/ ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.48 (d, *J* = 7.2 Hz, 1 H), 7.84 (d, *J* = 8.0 Hz, 1 H), 7.77 (d, *J* = 8.4 Hz, 1 H), 7.69-7.57 (m, 3 H), 7.54-7.47 (m, 1 H), 7.43-7.35 (m, 4 H), 7.32-7.26 (m, 2 H), 6.87 (dd, *J* = 8.4, 1.2 Hz, 2 H), 4.47 (s, 2 H), 3.81 (s, 3 H), 3.57 (t, *J* = 6.4 Hz, 2 H), 3.21 (t, *J* = 7.2 Hz, 2 H), 2.19-2.08 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.0, 143.2, 133.6, 131.7, 131.5, 130.7, 129.2, 128.4, 128.3, 128.2, 128.0, 127.4, 126.7, 126.1, 125.6, 123.6, 119.1, 113.7, 98.4, 86.1, 72.5, 69.4, 55.2, 32.0, 30.7. HRMS (EI) calcd for $C_{29}H_{26}O_2$ $[M]^+$: 406.1933, found 406.1939.



The reaction of 1-iodonaphthalene **1a**(50.8 mg, 0.20 mmol, 1.0 equiv), 4-methoxybenzyl chloride **2h** (62.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol, 5 mol %), P(*p*-$MeOC_6H_4)_3$ (8.8 mg, 0.025 mmol, 12.5 mol %), norbornene (56.5 mg, 0.6 mmol, 3.0 equiv), Cs_2CO_3 (0.196 g, 0.6 mmol, 3.0 equiv), $MgSO_4$ (0.150 g) in DME (4.0 mL) at 100 °C for 3 h afforded **4h** (55.8 mg, 80%) (petroleum ether/ dichloromethane = 9 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1 H), 7.81 (d, *J* = 8.0 Hz, 1 H), 7.75 (d, *J* = 8.0 Hz, 1 H), 7.64 (dd, *J* = 8.0, 2.0 Hz, 2 H), 7.59 (t, *J* = 8.0 Hz, 1 H), 7.49 (t, *J* = 7.4 Hz, 1 H), 7.44-7.37 (m, 3 H), 7.34 (d, *J* = 8.0 Hz, 1 H), 7.25 (d, *J* = 8.0 Hz, 2 H), 6.83 (d, *J* = 8.0 Hz, 2 H), 4.42 (s, 2 H), 3.77 (s, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.0, 142.4, 133.6, 132.9, 131.8, 131.6, 129.9, 128.6, 128.5, 128.4, 128.1, 127.4, 126.9, 126.3, 125.8, 123.6, 119.3, 113.9, 98.5, 86.6, 55.2, 40.0. HRMS (ESI) calcd for $C_{26}H_{21}O$ $[M+H]^+$: 349.1586, found 349.1592.

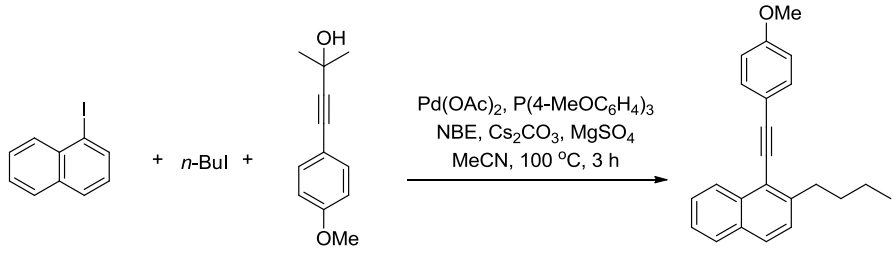


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-(4'-methylphenyl)-2-methylbut-3-yn-2-ol **3i** (52.3 mg, 0.30 mmol, 1.5 equiv) afforded **4i** (49.3 mg, 83%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.47 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.76 (d, *J* = 8.0 Hz, 1 H), 7.61-7.52 (m, 3 H), 7.51-7.45 (m, 1 H), 7.39 (d, *J* = 8.0 Hz, 1 H), 7.24 (t, *J* = 8.0 Hz, 2 H), 3.10 (t, *J* = 8.0 Hz, 2H), 2.42 (s, 3 H), 1.84-1.71 (m, 2 H), 1.54-1.40 (m, 2 H), 1.00 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.9, 138.4, 133.6, 131.7, 131.4, 129.2, 128.1, 128.0, 127.4, 126.6, 126.1, 125.4, 120.8, 119.1, 98.3, 85.7, 35.1, 33.1, 22.7, 21.5, 14.1. HRMS (EI) calcd for $C_{23}H_{22}$ $[M]^+$: 298.1722, found 298.1725.



1a **2a** **3j** **4j**

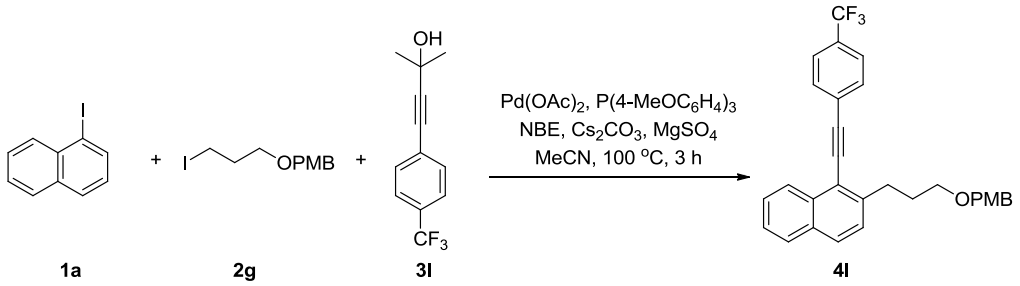
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-(4'-*tert*-butylphenyl)-2-methylbut-3-yn-2-ol **3j** (64.9 mg, 0.30 mmol, 1.5 equiv) afforded **4j** (53.5 mg, 79%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.46 (d, *J* = 8.0 Hz, 1 H), 7.82 (d, *J* = 8.0 Hz, 1 H), 7.76 (d, *J* = 8.0 Hz, 1 H), 7.62-7.54 (m, 3 H), 7.50-7.42 (m, 3 H), 7.39 (d, *J* = 8.0 Hz, 1 H), 3.09 (t, *J* = 8.0 Hz, 2 H), 1.83-1.71 (m, 2 H), 1.53-1.42 (m, 2 H), 1.37 (s, 9 H), 0.99 (d, *J* = 7.4 Hz, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 151.5, 144.0, 133.6, 131.7, 131.2, 128.1, 128.0, 127.4, 126.6, 126.2, 125.5, 120.8, 119.1, 98.3, 85.7, 35.1, 34.8, 33.1, 31.2, 22.7, 14.1. HRMS (EI) calcd for $C_{26}H_{28}$ $[M]^+$: 340.2191, found 340.2194.



1a **2a** **3k** **4k**

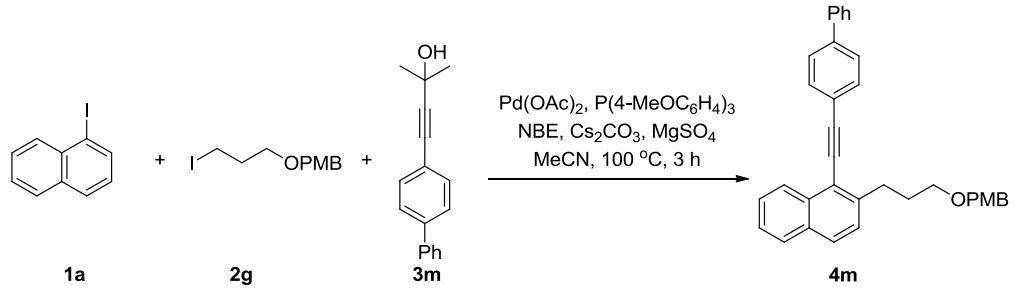
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-(4'-methoxyphenyl)-2-methylbut-3-yn-2-ol **3k** (57.1 mg, 0.30 mmol, 1.5 equiv) afforded **4k** (54.6 mg, 87%) (petroleum ether / ethyl acetate = 100 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.76 (d, *J* = 8.0 Hz, 1 H), 7.65-7.55 (m, 3 H), 7.48 (t, *J* = 8.0 Hz, 1 H), 7.39 (d, *J* = 8.4 Hz, 1 H), 6.98-6.92 (m, 2 H), 3.86 (s, 3 H), 3.10 (t, *J* = 8.0 Hz, 2 H), 1.85-1.72 (m, 2 H), 1.54-1.42 (m, 2 H), 1.01 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.6, 143.7, 133.5, 132.9, 131.6, 128.0, 127.9, 127.3, 126.6, 126.1, 125.4, 119.2, 116.0, 114.1, 98.1, 85.0, 55.3, 35.1, 33.1, 22.7, 14.1. HRMS (EI) calcd for $C_{23}H_{22}O$ $[M]^+$: 314.1671, found

314.1667.



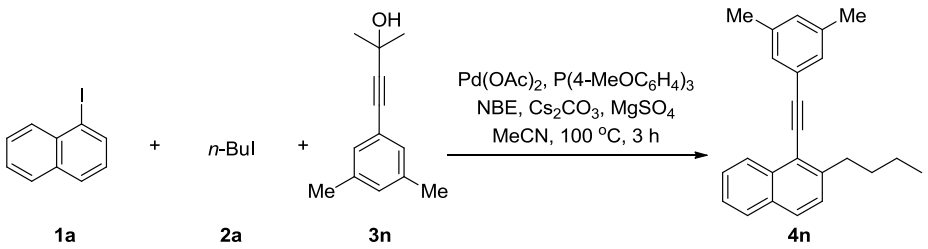
1a **2g** **3l** **4l**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-(4'-trifluoromethylphenyl)-2-methylbut-3-yn-2-ol **3l** (68.5 mg, 0.30 mmol, 1.5 equiv) afforded **4l** (58.5 mg, 62%) (petroleum ether / ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.41 (d, *J* = 8.4 Hz, 1 H), 7.84 (d, *J* = 8.0 Hz, 1 H), 7.80 (d, *J* = 8.4 Hz, 1 H), 7.69 (d, *J* = 8.0 Hz, 2 H), 7.63-7.56 (m, 3 H), 7.53-7.47 (m, 1 H), 7.40 (d, *J* = 8.4 Hz, 1 H), 7.27 (d, *J* = 8.8 Hz, 2 H), 6.85 (d, *J* = 8.8 Hz, 2 H), 4.46 (s, 2 H), 3.80 (s, 3 H), 3.56 (t, *J* = 6.4 Hz, 2 H), 3.20 (t, *J* = 7.8 Hz, 2 H), 2.15-2.03 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 143.9, 133.5, 131.71, 131.69, 130.6, 130.0, 129.7, 129.3, 129.0, 128.2, 127.4, 127.0, 125.9, 125.8, 125.3 (q, *J* = 4.0 Hz), 118.4, 113.7, 96.9, 88.6, 72.7, 69.5, 55.2, 32.2, 30.9. HRMS (ESI) calcd for $C_{30}H_{26}O_2F_3$ $[M+H]^+$: 475.1881, found 475.1885.

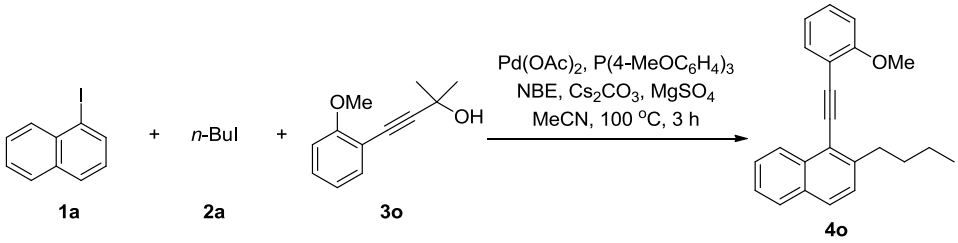


1a **2g** **3m** **4m**

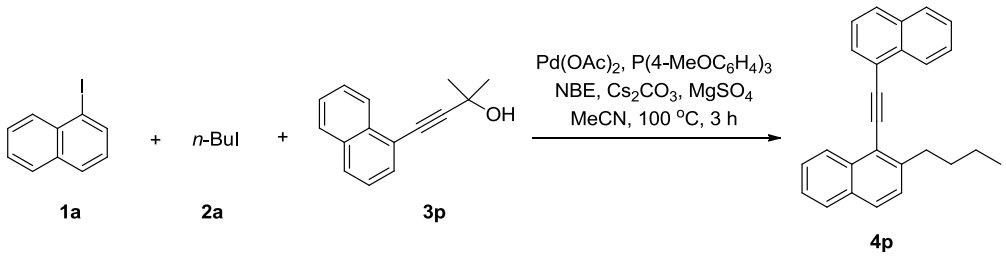
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-([1,1'-biphenyl]-4-yl)-2-methylbut-3-yn-2-ol **3m** (70.9 mg, 0.30 mmol, 1.5 equiv) afforded **4m** (60.4 mg, 63%) (petroleum ether / ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.49 (d, *J* = 8.8 Hz, 1 H), 7.84 (d, *J* = 8.0 Hz, 1 H), 7.77 (d, *J* = 8.4 Hz, 1 H), 7.72-7.67 (m, 2 H), 7.67-7.57 (m, 5 H), 7.53-7.45 (m, 3 H), 7.42-7.36 (m, 2 H), 7.28 (dd, *J* = 8.4, 1.2 Hz, 2 H), 6.86 (dd, *J* = 8.4, 1.6 Hz, 2 H), 4.48 (s, 2 H), 3.77 (s, 3 H), 3.58 (t, *J* = 6.4 Hz, 2 H), 3.22 (t, *J* = 8.0 Hz, 2 H), 2.19-2.06 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.3, 140.9, 140.4, 133.6, 131.9, 131.7, 130.7, 129.3, 128.9, 128.4, 128.1, 127.6, 127.4, 127.1, 127.0, 126.8, 126.1, 125.6, 122.5, 119.1, 113.7, 98.4, 86.9, 72.6, 69.5, 55.2, 32.1, 30.8. HRMS (ESI) calcd for $C_{35}H_{31}O_2$ $[M+H]^+$: 483.2321, found 483.2324.



The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-(3',5'-dimethylphenyl)-2-methylbut-3-yn-2-ol **3n** (56.5 mg, 0.30 mmol, 1.5 equiv) afforded **4n** (46.2 mg, 74%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.48 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.77 (d, *J* = 8.0 Hz, 1 H), 7.62-7.56 (m, 1 H), 7.51-7.46 (m, 1 H), 7.40 (d, *J* = 8.0 Hz, 1 H), 7.31 (d, *J* = 6.0 Hz, 2 H), 7.03 (s, 1 H), 3.10 (t, *J* = 8.0 Hz, 2 H), 2.385 (s, 3 H), 2.383 (s, 3 H), 1.84-1.74 (m, 2 H), 1.54-1.43 (m, 2 H), 1.01 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.0, 138.0, 133.6, 131.6, 130.2, 129.1, 128.1, 128.0, 127.4, 126.6, 126.2, 125.5, 123.4, 119.1, 98.5, 85.7, 35.1, 33.1, 22.7, 21.2, 14.1. HRMS (EI) calcd for $C_{24}H_{24}$ $[M]^+$: 312.1878, found 312.1879.

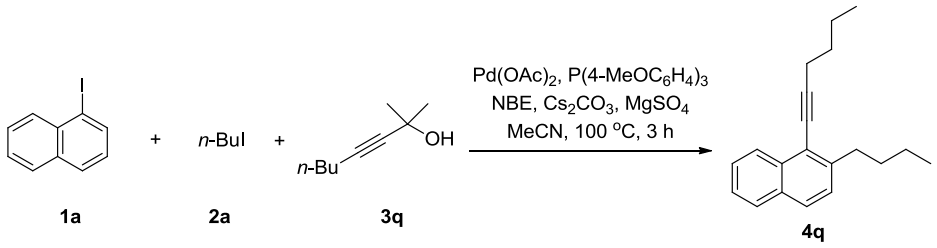


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-(2'-methoyphenyl)-2-methylbut-3-yn-2-ol **3o** (57.1 mg, 0.30 mmol, 1.5 equiv) afforded **4o** (49.3 mg, 78%) (petroleum ether/ ethyl acetate = 100 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.61 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.76 (d, *J* = 8.0 Hz, 1 H), 7.65-7.56 (m, 2 H), 7.52-7.44 (m, 1 H), 7.40 (d, *J* = 8.0 Hz, 1 H), 7.35 (t, *J* = 8.0 Hz, 1 H), 7.01 (t, *J* = 8.0 Hz, 1 H), 6.97 (d, *J* = 8.0 Hz, 1 H), 3.99 (s, 3 H), 3.14 (t, *J* = 8.0 Hz, 2 H), 1.86-1.73 (m, 2 H), 1.55-1.41 (m, 2 H), 1.01 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.1, 143.8, 133.7, 133.0, 131.6, 129.6, 128.0, 127.9, 127.3, 126.6, 126.4, 125.4, 120.5, 119.4, 113.2, 110.7, 94.6, 90.6, 55.7, 35.0, 33.1, 22.7, 14.1. HRMS (ESI) calcd for $C_{23}H_{23}O$ $[M+H]^+$: 315.1745, found 315.1749.

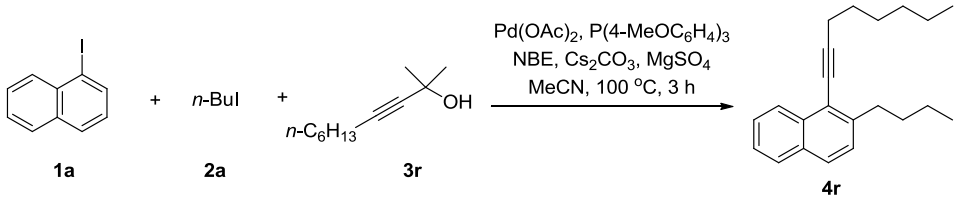


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6

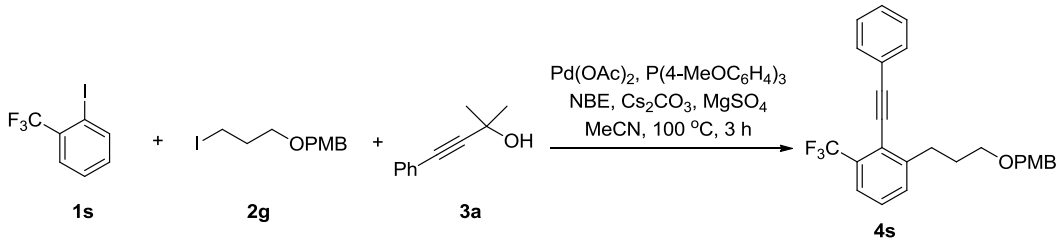
mg, 0.40 mmol, 2.0 equiv), 4-(α-naphthyl)-2-methylbut-3-yn-2-ol **3p** (57.1 mg, 0.30 mmol, 1.5 equiv) afforded **4p** (57.8 mg, 86%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.63 (d, *J* = 8.0 Hz, 1 H), 7.98-7.86 (m, 4 H), 7.83 (d, *J* = 8.0 Hz, 1 H), 7.70-7.50 (m, 5 H), 7.46 (d, *J* = 8.0 Hz, 1 H), 3.23 (t, *J* = 8.0 Hz, 2 H), 1.93-1.83 (m, 2 H), 1.60-1.48 (m, 2 H), 1.03 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.2, 133.7, 133.3, 133.2, 131.7, 130.5, 128.7, 128.43, 128.38, 128.1, 127.4, 126.9, 126.8, 126.5, 126.3, 126.1, 125.6, 125.4, 121.5, 119.0, 96.3, 91.2, 35.5, 33.3, 22.8, 14.1. HRMS (EI) calcd for $C_{26}H_{22}$ $[M]^+$: 334.1722, found 334.1721.



The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 2-methyloct-3-yn-2-ol **3q** (42.1 mg, 0.30 mmol, 1.5 equiv) afforded **4q** (38.7mg, 73%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.35 (d, *J* = 8.4 Hz, 1 H), 7.78 (d, *J* = 8.0 Hz, 1 H), 7.69 (d, *J* = 8.4 Hz, 1 H), 7.56-7.49 (m, 1 H), 7.46-7.40 (m, 1 H), 7.33 (d, *J* = 8.4 Hz, 1 H), 2.98 (t, *J* = 8.0 Hz, 2 H), 2.63 (t, *J* = 6.8 Hz, 2 H), 1.77-1.65 (m, 4 H), 1.65-1.52 (m, 2 H), 1.49-1.36 (m, 2 H), 1.05-0.93 (m, 6 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.4, 133.8, 131.6, 127.9, 127.3, 126.4, 126.2, 125.2, 119.7, 99.3, 35.0, 33.0, 31.1, 22.7, 22.1, 19.6, 14.0, 13.7. HRMS (EI) calcd for $C_{20}H_{24}$ $[M]^+$: 264.1878, found 264.1876.

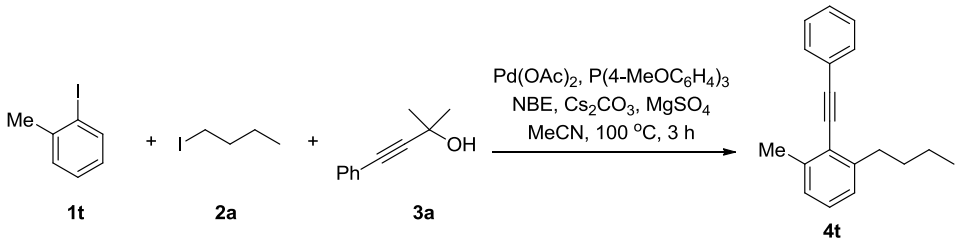


The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 2-methyldec-3-yn-2-ol **3r** (50.5 mg, 0.30 mmol, 1.5 equiv) afforded **4r** (50.5 mg, 81%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 8.37 (d, *J* = 8.4 Hz, 1 H), 7.79 (d, *J* = 8.0 Hz, 1 H), 7.70 (d, *J* = 8.4 Hz, 1 H), 7.56-7.49 (m, 1 H), 7.47-7.39 (m, 1 H), 7.34 (d, *J* = 8.8 Hz, 1 H), 3.00 (t, *J* = 8.0 Hz, 2 H), 2.63 (t, *J* = 7.2 Hz, 2 H), 1.83-1.64 (m, 4 H), 1.64-1.51 (m, 2 H), 1.50-1.31 (m, 6 H), 1.02-0.88 (m, 6 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.4, 133.9, 131.6, 127.9, 127.3, 126.3, 126.2, 125.2, 119.8, 99.3, 35.0, 33.0, 31.4, 29.0, 28.7, 22.64, 22.62, 19.9, 14.1, 14.0. HRMS (ESI) calcd for $C_{22}H_{29}$ $[M+H]^+$: 293.2270, found 293.2269.



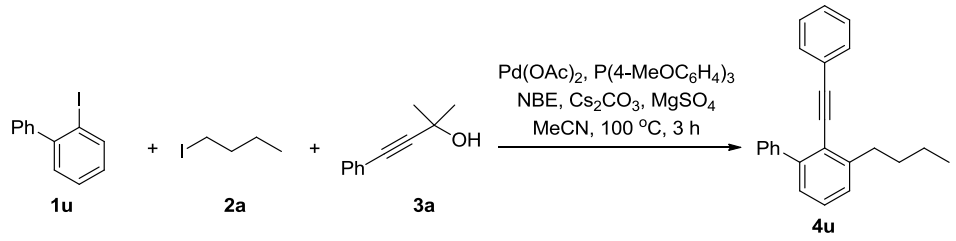
1s **2g** **3a** **4s**

The reaction of 2-iodo-trifluoromethylbenzene **1s** (54.4 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4s** (53.0 mg, 62%) (petroleum ether / ethyl acetate = 50 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.58-7.48 (m, 3 H), 7.40 (d, *J* = 7.6 Hz, 1 H), 7.38-7.25 (m, 6 H), 6.90-6.83 (m, 2 H), 4.46 (s, 2 H), 3.81 (s, 3 H), 3.52 (t, *J* = 6.4 Hz, 2 H), 3.05 (t, *J* = 8.0 Hz, 2 H), 2.10-1.94 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 146.1, 132.2, 132.1, 131.8, 131.5, 130.6, 129.3, 128.7, 128.4, 127.5, 125.1, 123.6 (q, *J* = 5.2 Hz), 122.9, 122.4, 120.7 (q, *J* = 1.9 Hz), 113.7, 99.0, 83.8, 72.6, 69.2, 55.2, 31.5, 30.3. HRMS (EI) calcd for $C_{26}H_{23}F_3O_2$ $[M]^+$: 424.1650, found 424.1655.



1t **2a** **3a** **4t**

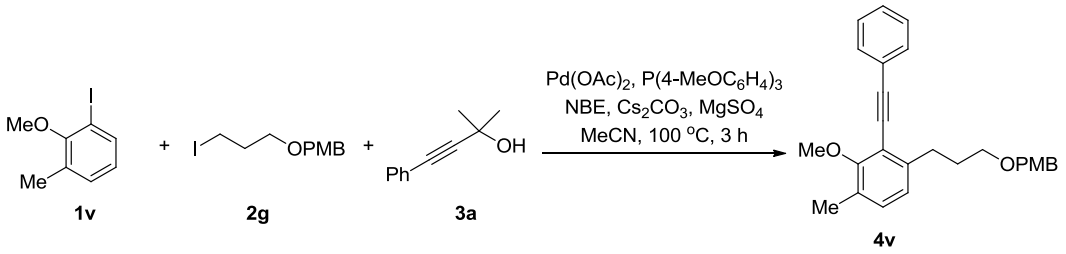
The reaction of 2-iodotoluene **1t** (43.6 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4t** (39.6 mg, 80%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.61-7.48 (m, 2 H), 7.43-7.31 (m, 3 H), 7.16 (t, *J* = 7.6 Hz, 1 H), 7.08 (t, *J* = 6.4 Hz, 2 H), 2.88 (t, *J* = 7.6 Hz, 2 H), 2.53 (s, 3 H), 1.78-1.62 (m, 2 H), 1.51-1.35 (m, 2 H), 0.97 (t, *J* = 7.2 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.1, 140.4, 131.3, 128.4, 128.0, 127.8, 126.8, 126.1, 124.0, 122.5, 97.2, 87.1, 34.8, 32.9, 22.7, 21.2, 14.0. HRMS (ESI) calcd for $C_{19}H_{21}$ $[M+H]^+$: 249.1642, found 249.1643.



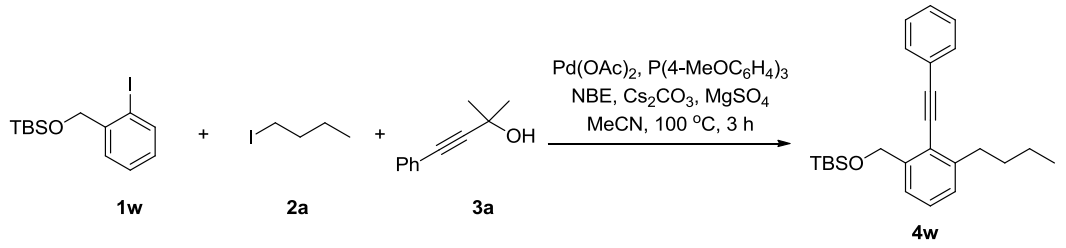
1u **2a** **3a** **4u**

The reaction of 2-phenyliodobenzene **1u** (56.0 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4u** (55.7 mg, 90%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.63 (d, *J* = 7.2 Hz, 2 H), 7.43 (t, *J* = 7.2 Hz, 2 H), 7.40-7.34 (m, 1 H), 7.32-7.19 (m, 8 H), 2.94 (t, *J* = 8.0 H, 2 H), 1.82-1.66 (m, 2 H), 1.51-1.38 (m, 2 H), 0.98 (t, *J* = 7.2 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.7,

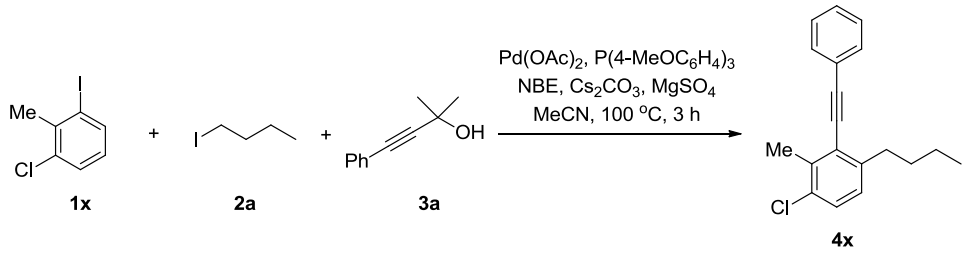
144.6, 141.2, 131.1, 129.6, 128.2, 128.0, 127.9, 127.70, 127.66, 127.2, 126.9, 123.8, 121.0, 96.2, 88.0, 35.0, 33.0, 22.8, 14.1. HRMS (ESI) calcd for $C_{24}H_{23}$ $[M+H]^+$: 311.1796, found 311.1800.



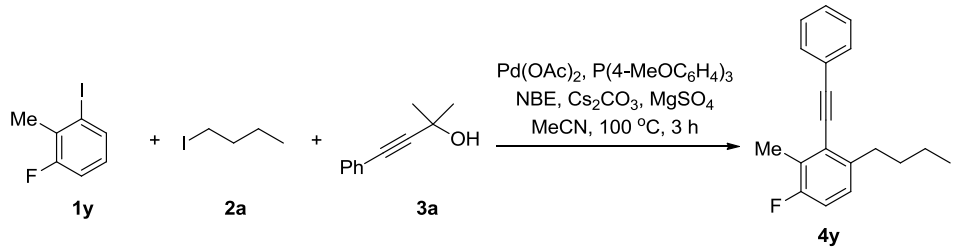
The reaction of 1-iodo-2-methoxy-3-methylbenzene **1v** (49.6 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4v** (64.6 mg, 81%) (petroleum ether / ethyl acetate = 35 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.57-7.49 (m, 2 H), 7.38-7.30 (m, 3 H), 7.27 (d, *J* = 8.4 Hz, 2 H), 7.07 (d, *J* = 8.0 Hz, 1 H), 6.92-6.83 (m, 3 H), 4.46 (s, 2 H), 3.97 (s, 3 H), 3.80 (s, 3 H), 3.53 (t, *J* = 6.4 Hz, 2 H), 2.93 (t, *J* = 7.6 Hz, 2 H), 2.28 (s, 3 H), 2.08-1.95 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.6, 159.1, 143.6, 131.3, 130.8, 130.7, 129.2, 128.4, 128.3, 128.1, 124.2, 123.7, 116.6, 113.7, 97.2, 84.6, 72.5, 69.5, 60.5, 55.2, 31.1, 30.5, 15.8. HRMS (ESI) calcd for $C_{27}H_{28}O_3Na$ $[M+Na]^+$: 423.1933, found 423.1936.



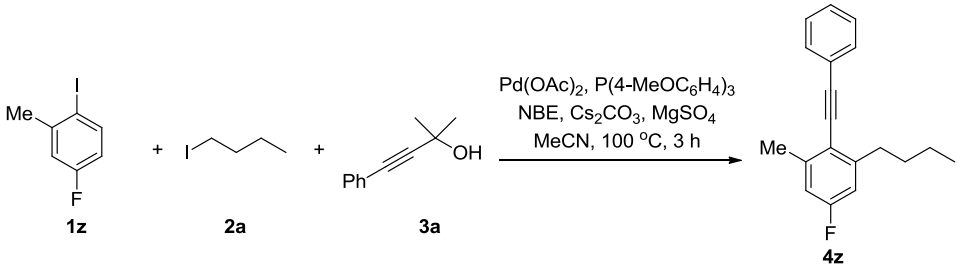
The reaction of O-TBS-(2-iodophenyl)methanol **1w** (69.7 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4w** (46.1 mg, 61%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.55-7.50 (m, 2 H), 7.44 (d, *J* = 7.6 Hz, 1 H), 7.41-7.34 (m, 3 H), 7.30 (t, *J* = 7.6 Hz, 1 H), 7.14 (d, *J* = 7.6 Hz, 1 H), 5.01 (s, 2 H), 2.88 (t, *J* = 8.0 Hz, 2 H), 1.78-1.62 (m, 2 H), 1.51-1.36 (m, 2 H), 1.02-0.93 (m, 12 H), 0.15 (s, 6 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.7, 143.4, 131.3, 128.4, 128.2, 128.1, 127.0, 123.7, 123.1, 119.1, 98.1, 85.5, 63.6, 34.4, 32.9, 26.0, 22.7, 18.5, 14.0, -5.3. HRMS (ESI) calcd for $C_{25}H_{34}OSiNa$ $[M+Na]^+$: 401.2275, found 401.2277.



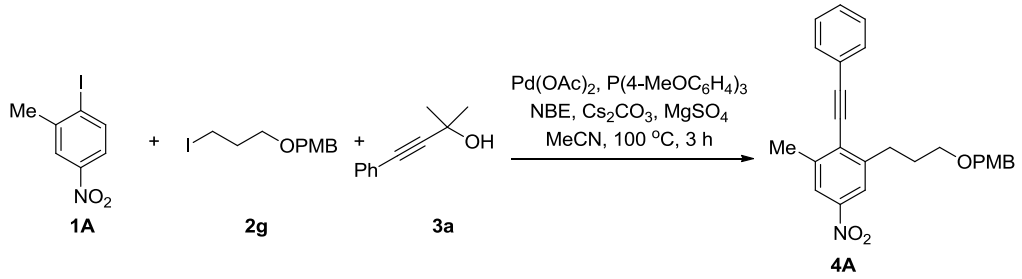
The reaction of 3-chloro-2-methyliodobenzene **1x** (50.5 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4x** (46.0 mg, 81%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.60-7.53 (m, 2 H), 7.46-7.34 (m, 3 H), 7.27 (d, *J* = 9.2 Hz, 1 H), 7.03 (d, *J* = 8.0 Hz, 1 H), 2.87 (t, *J* = 8.0 Hz, 2 H), 2.64 (s, 3 H), 1.77-1.65 (m, 2 H), 1.51-1.37 (m, 2 H), 0.99 (t, *J* = 7.2 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.8, 137.8, 131.7, 131.4, 128.6, 128.43, 128.37, 127.1, 124.3, 123.4, 97.7, 86.6, 34.6, 32.7, 22.6, 18.7, 14.0. HRMS (EI) calcd for $C_{19}H_{19}Cl$ $[M]^+$: 282.1175, found 282.1172.



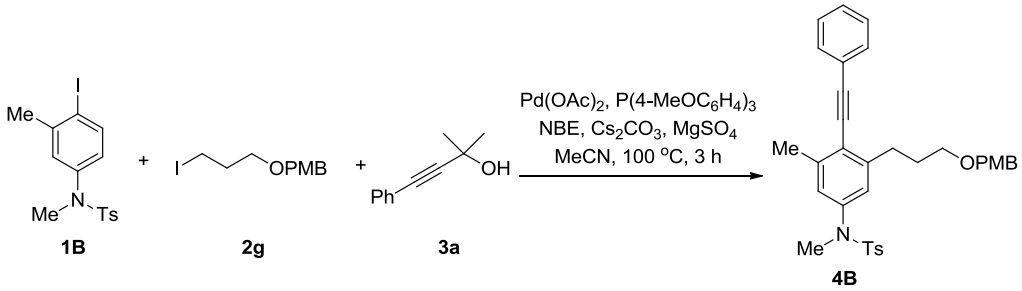
The reaction of 3-fluoro-2-methyliodobenzene **1y** (47.3 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4y** (42.9 mg, 81%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.60-7.50 (m, 2 H), 7.42-7.32 (m, 3 H), 7.00 (dd, *J* = 8.4, 5.6 Hz, 1 H), 6.92 (t, *J* = 9.0 Hz, 1 H), 2.83 (t, *J* = 8.0 Hz, 2 H), 2.45 (d, *J* = 2.4 Hz, 3 H), 1.72-1.60 (m, 2 H), 1.48-1.34 (m, 2 H), 0.96 (t, *J* = 7.4 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) 159.3 (d, *J* = 239.9 Hz), 140.7 (d, *J* = 3.4 Hz), 131.4, 128.4, 128.3, 126.9 (d, *J* = 8.6 Hz), 126.7, 124.3 (d, *J* = 6.0 Hz), 123.5, 114.8 (d, *J* = 22.6 Hz), 97.7, 86.2 (d, *J* = 4.4 Hz), 34.3, 32.9, 22.6, 14.0, 13.0 (d, *J* = 3.9 Hz). HRMS (EI) calcd for $C_{19}H_{19}F$ $[M]^+$: 266.1471, found 266.1476.



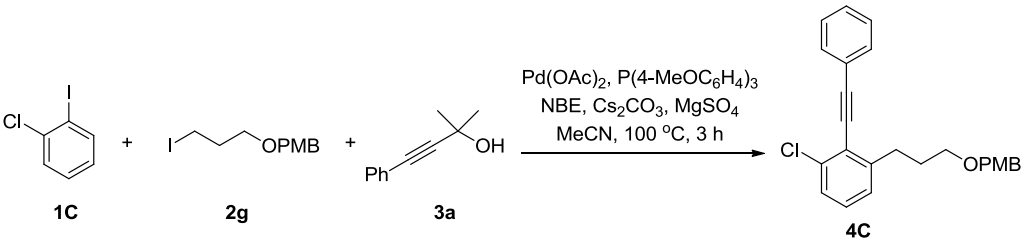
The reaction of 4-fluoro-2-methyliodobenzene **1z** (47.3 mg, 0.20 mmol, 1.0 equiv), *n*-iodobutane **2a** (73.6 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4z** (41.2 mg, 77%) (petroleum ether). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.56-7.45 (m, 2 H), 7.41-7.29 (m, 3 H), 6.83-6.69 (m, 2 H), 2.85 (t, *J* = 8.0 Hz, 2 H), 2.51 (s, 3 H), 1.75-1.60 (m, 2 H), 1.50-1.32 (m, 2 H), 0.96 (t, *J* = 7.2 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) 162.1 (d, *J* = 246.8 Hz), 147.7 (d, *J* = 8.0 Hz), 142.9 (d, *J* = 8.5 Hz), 131.3, 128.4, 128.1, 123.8, 118.5 (d, *J* = 2.7 Hz), 113.8 (d, *J* = 21.7 Hz), 113.0 (d, *J* = 21.4 Hz), 96.8 (d, *J* = 1.8 Hz), 86.2, 34.7 (d, *J* = 1.5 Hz), 32.5, 22.6, 21.3 (d, *J* = 1.6 Hz), 14.0. HRMS (EI) calcd for $C_{19}H_{19}F$ $[M]^+$: 266.1471, found 266.1479.



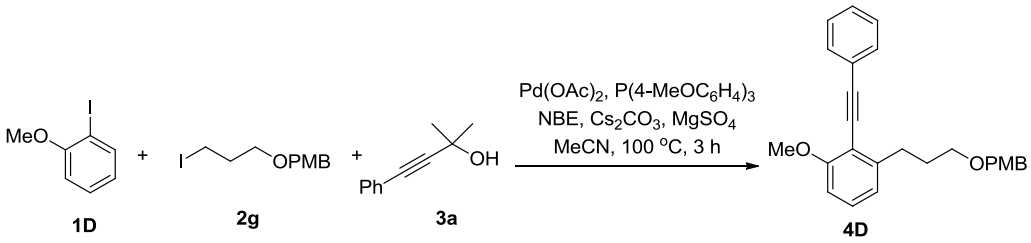
The reaction of 4-nitro-2-methyliodobenzene **1A** (52.6 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4A** (51.8 mg, 62%) (petroleum ether / ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.96 (s, 2 H), 7.56-7.49 (m, 2 H), 7.43-7.32 (m, 3 H), 7.30-7.23 (m, 2 H), 6.89-6.81 (m, 2 H), 4.45 (s, 2 H), 3.80 (s, 3 H), 3.52 (t, *J* = 6.0 Hz, 2 H), 3.06 (t, *J* = 8.0 Hz, 2 H), 2.61 (s, 3 H), 2.11-1.98 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 146.5, 145.7, 141.9, 131.6, 130.5, 129.5, 129.3, 129.1, 128.5, 122.6, 121.8, 121.1, 113.8, 102.4, 85.4, 72.7, 69.0, 55.2, 31.8, 30.0, 21.3. HRMS (ESI) calcd for $C_{26}H_{25}NO_4Na$ $[M+Na]^+$: 438.1683, found 438.1681.



The reaction of aryliodide **1B** (80.3 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4B** (78.3 mg, 71%) (petroleum ether / ethyl acetate = 10 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.53-7.47 (m, 2 H), 7.44 (d, *J* = 8.0 Hz, 2 H), 7.37-7.31 (m, 3 H), 7.27-7.20 (m, 4 H), 6.92 (d, *J* = 1.6 Hz, 1 H), 6.85 (d, *J* = 8.4 Hz, 2 H), 6.76 (d, *J* = 1.6 Hz, 1 H), 4.43 (s, 2 H), 3.80 (s, 3 H), 3.49 (t, *J* = 6.0 Hz, 2 H), 3.13 (s, 3 H), 2.89 (t, *J* = 7.6 Hz, 2 H), 2.48 (s, 3 H), 2.41 (s, 3 H), 1.99-1.83 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) 159.1, 144.8, 143.6, 141.2, 140.8, 133.5, 131.3, 130.6, 129.3, 129.2, 128.4, 128.2, 127.9, 125.2, 123.7, 123.4, 121.5, 113.7, 98.1, 86.2, 72.5, 69.4, 55.2, 37.9, 31.6, 30.4, 21.5, 21.2. HRMS (ESI) calcd for $C_{34}H_{35}NO_4SNa$ $[M+Na]^+$: 576.2184, found 576.2188.

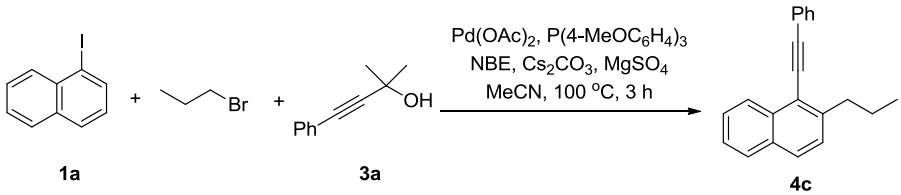


The reaction of 2-chloro-iodobenzene **1C** (47.7 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4C** (44.7 mg, 57%) (petroleum ether / ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.59-7.50 (m, 2 H), 7.39-7.32 (m, 3 H), 7.30-7.23 (m, 3 H), 7.19-7.08 (m, 2 H), 6.89-6.83 (m, 2 H), 4.45 (s, 2 H), 3.80 (s, 3 H), 3.51 (t, *J* = 6.4 Hz, 2 H), 2.98 (t, *J* = 8.0 Hz, 2H), 2.09-1.95 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 146.2, 136.3, 131.6, 130.6, 129.3, 128.6, 128.5, 128.4, 127.1, 126.8, 123.1, 122.7, 113.7, 98.4, 84.8, 72.5, 69.2, 55.3, 31.9, 30.2. HRMS (ESI) calcd for $C_{25}H_{23}O_2ClNa$ $[M+Na]^+$: 413.1281, found 413.1284.



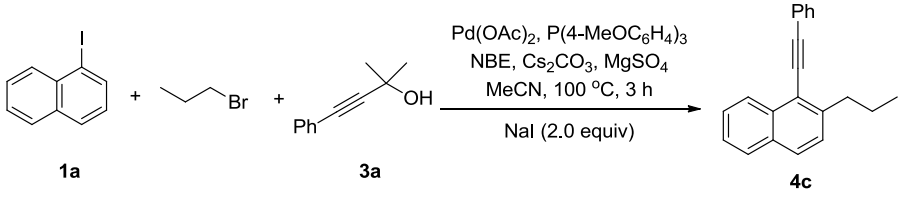
1D **2g** **3a** **4D**

The reaction of 2-methyliodobenzene **1D** (46.8 mg, 0.20 mmol, 1.0 equiv), 1-iodo-O-(4'-methoxyphenylmethyl)-propan-1-ol **2g** (0.122 g, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4D** (57.0 mg, 74%) (petroleum ether / ethyl acetate = 20 : 1). **^{1}H NMR** (400 MHz $CDCl_3$) δ 7.58-7.50 (m, 2 H), 7.37-7.30 (m, 3 H), 7.27 (d, *J* = 8.0 Hz, 2 H), 7.22 (t, *J* = 8.0 Hz, 1 H), 6.90-6.83 (m, 3 H), 6.77 (d, *J* = 8.4 Hz, 1 H), 4.45 (s, 2 H), 3.92 (s, 3 H), 3.80 (s, 3 H), 3.52 (t, *J* = 6.4 Hz, 2 H), 2.97 (t, *J* = 7.6 Hz, 2 H), 2.10-1.98 (m, 2 H); **^{13}C NMR** (100 MHz, $CDCl_3$) 160.2, 159.0, 146.2, 131.5, 130.7, 129.2, 129.0, 128.2, 127.9, 123.8, 121.3, 113.7, 111.9, 108.0, 97.5, 84.2, 72.5, 69.5, 55.9, 55.2, 31.4, 30.3. HRMS (EI) calcd for $C_{26}H_{26}O_3$ $[M]^+$: 386.1882, found 386.1884.



1a **3a** **4c**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-bromopropane (49.2 mg, 0.40 mmol, 2.0 equiv) and 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv) afforded **4c** (22 mg, 40%) (petroleum ether).



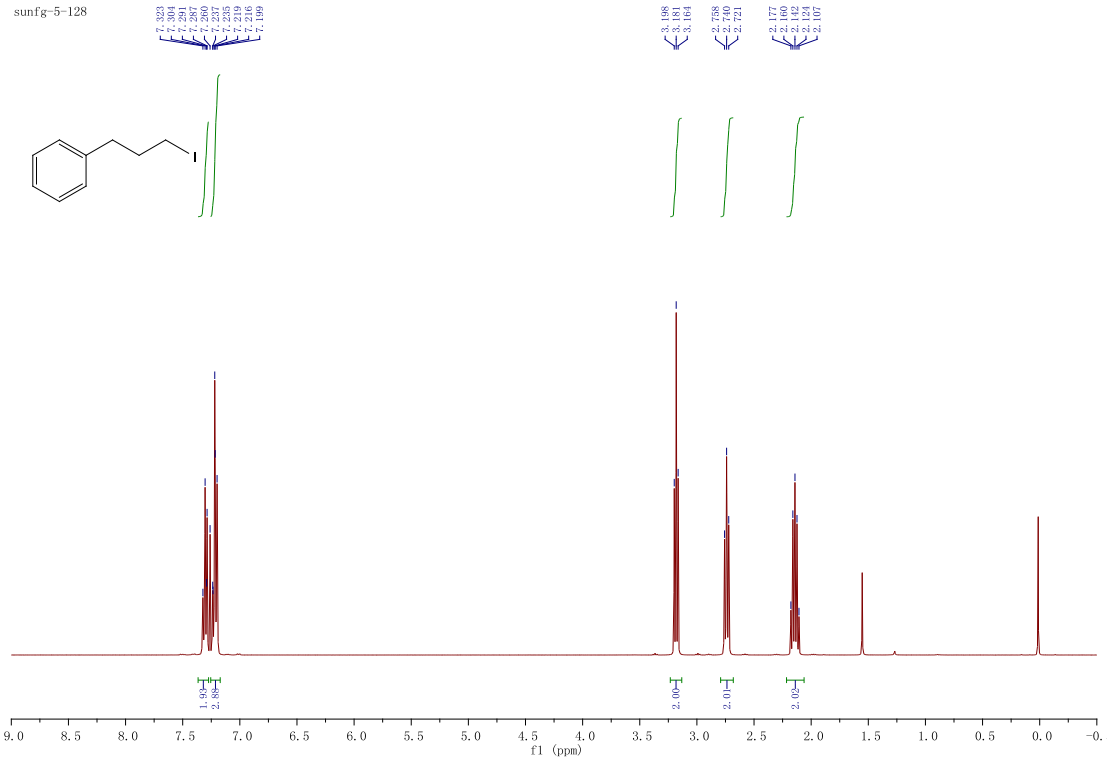
1a **3a** **4c**

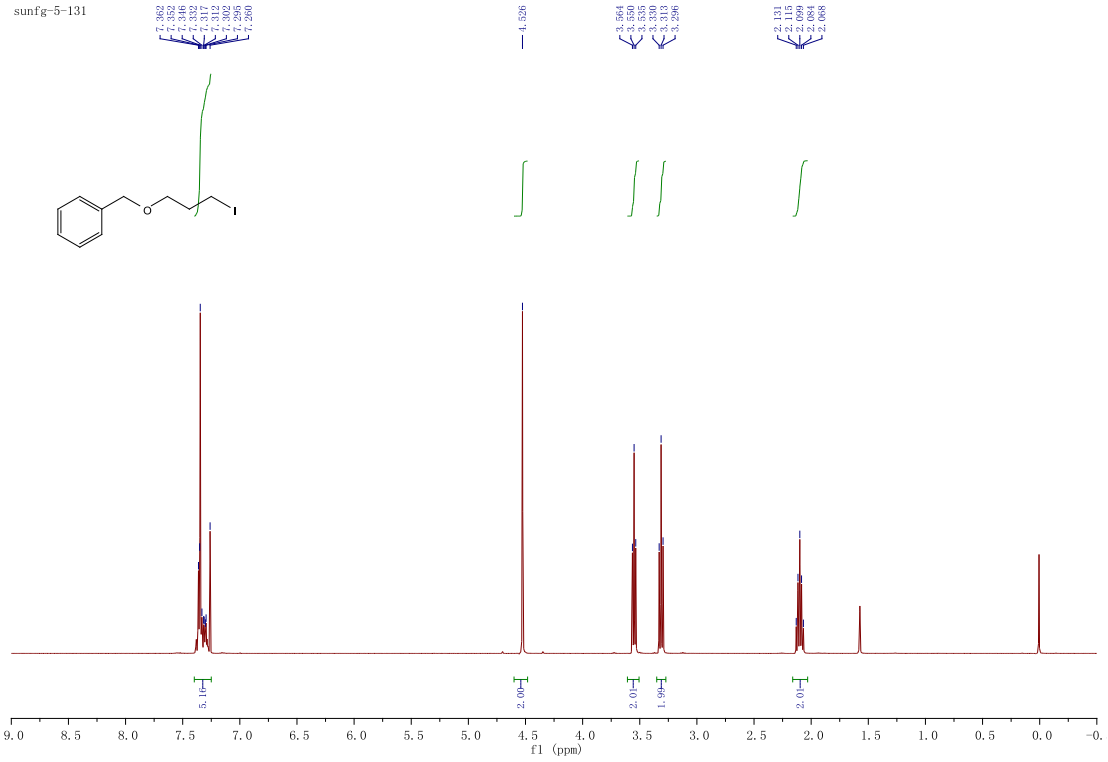
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), 1-bromopropane (49.2 mg, 0.40 mmol, 2.0 equiv), 4-phenyl-2-methylbut-3-yn-2-ol **3a** (48.0 mg, 0.30 mmol, 1.5 equiv)

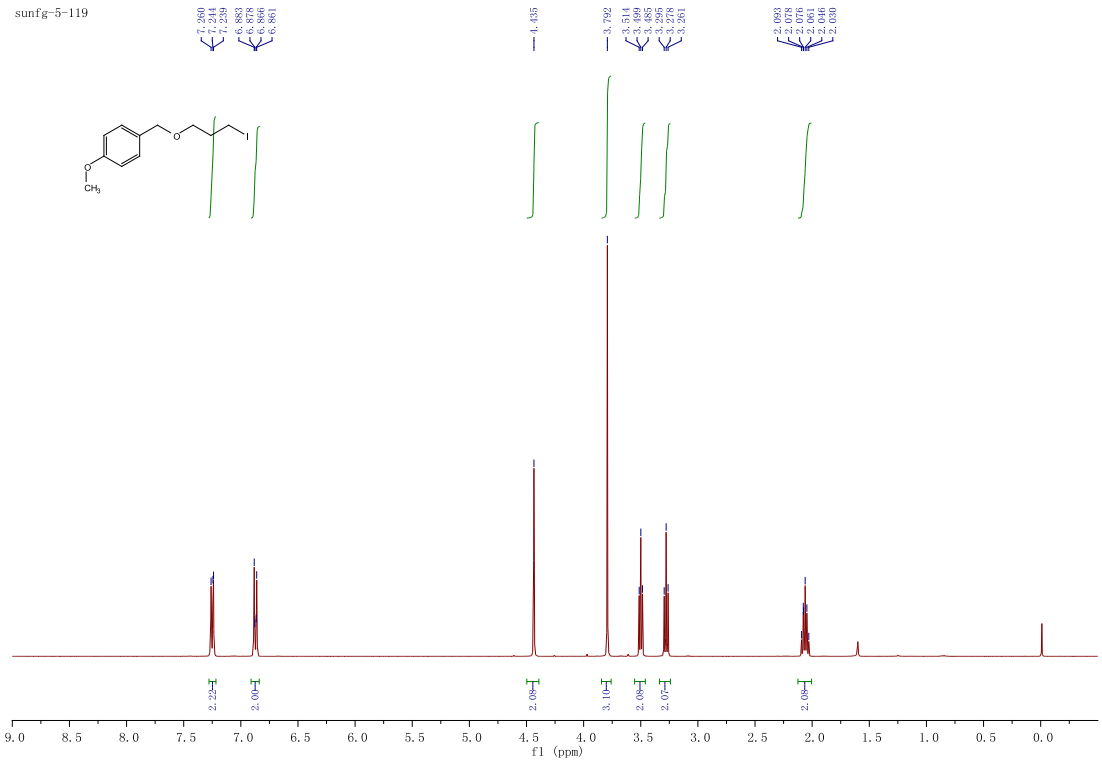
and NaI (60 mg, 0.40 mmol, 2.0 equiv) afforded **4c** (38 mg, 70%) (petroleum ether).

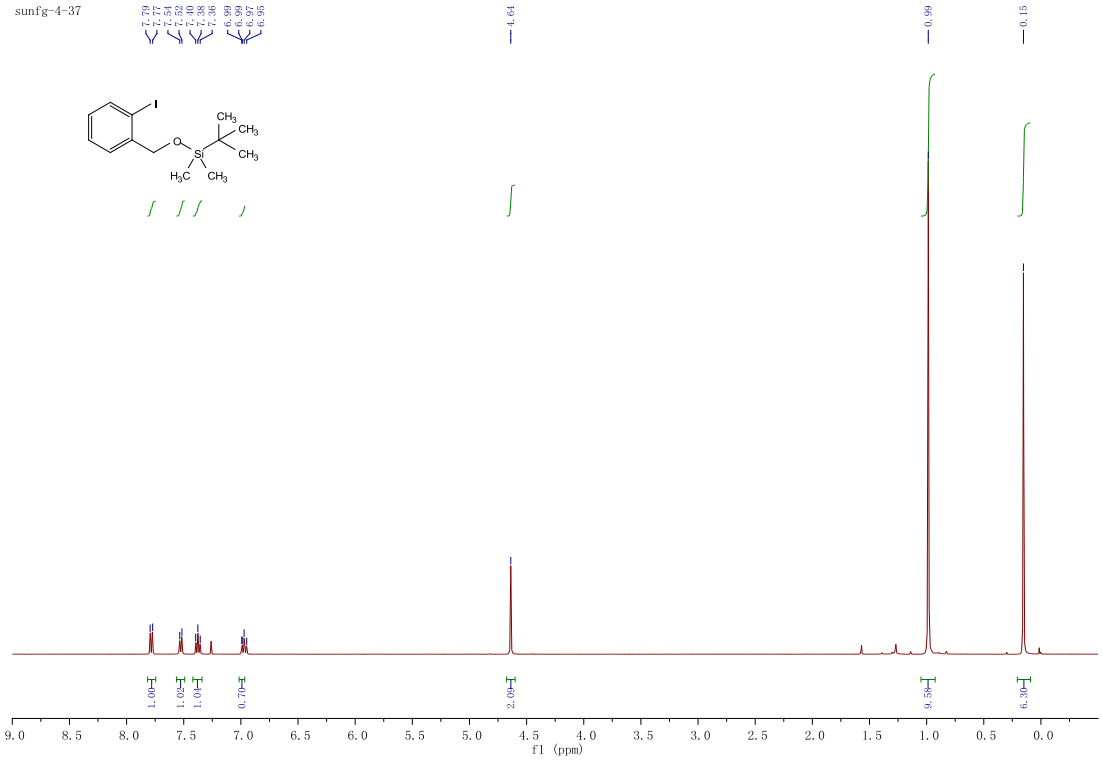
Reference

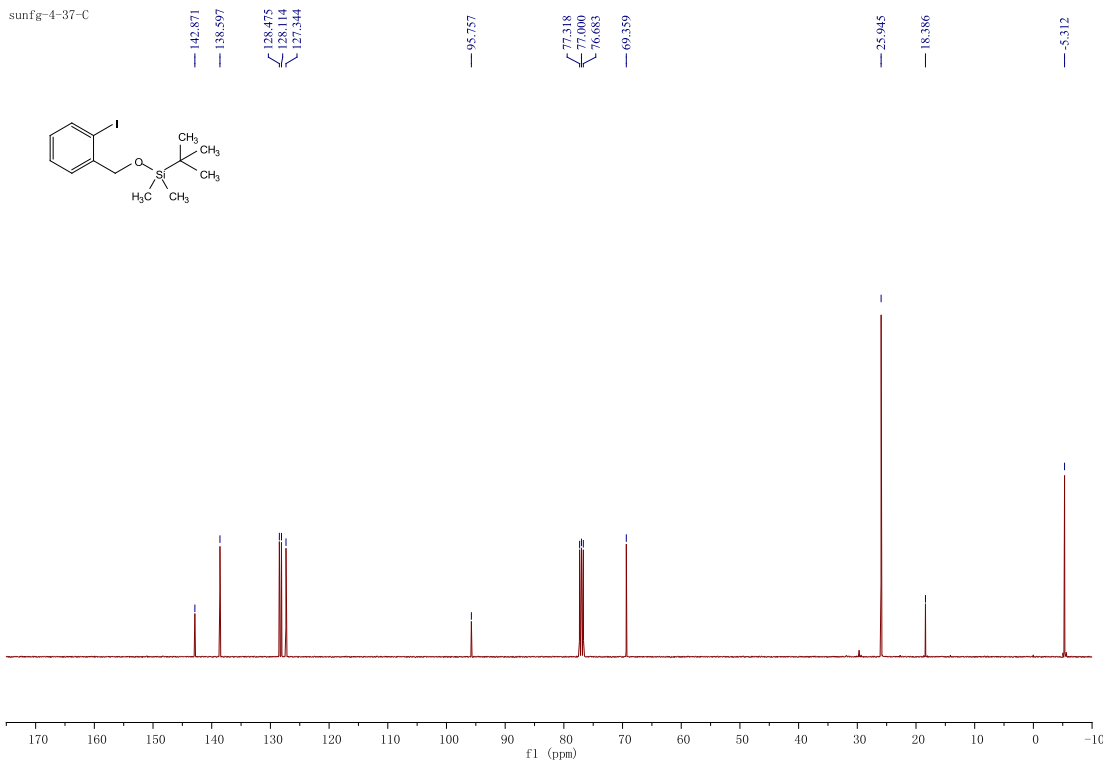
1. T. A. Gschneidtnerand K. Moth-Poulsen, *Tetrahedron Lett.*, **2013**, *54*, 5426.

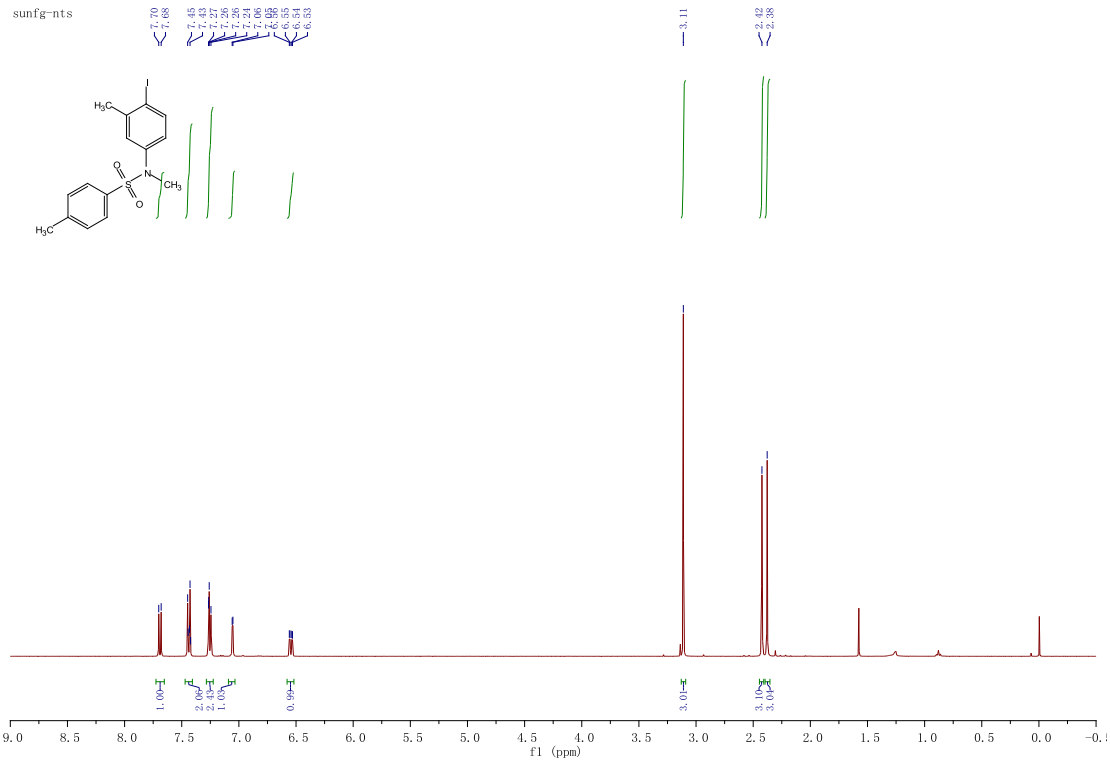

sunfg-5-128
7.323
7.304
7.291
7.283
7.260
7.237
7.235
7.219
7.216
7.199
3.198
3.181
3.164
2.758
2.740
2.721
2.177
2.160
2.142
2.124
2.107
1.89
2.88
2.00
2.01
2.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

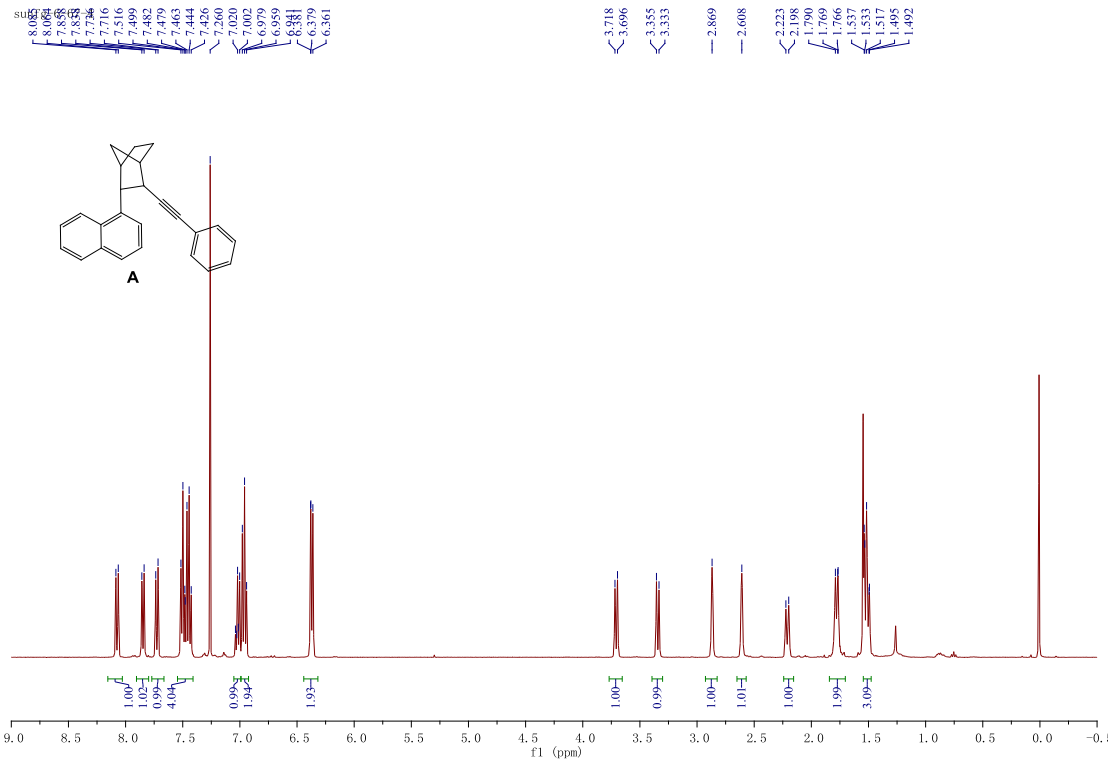

sunfg-5-131
7.362
7.352
7.346
7.332
7.317
7.312
7.302
7.295
7.290
4.526
3.564
3.550
3.535
3.330
3.313
3.296
2.131
2.115
2.099
2.084
2.068
5.16
2.00
2.01
1.99
2.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

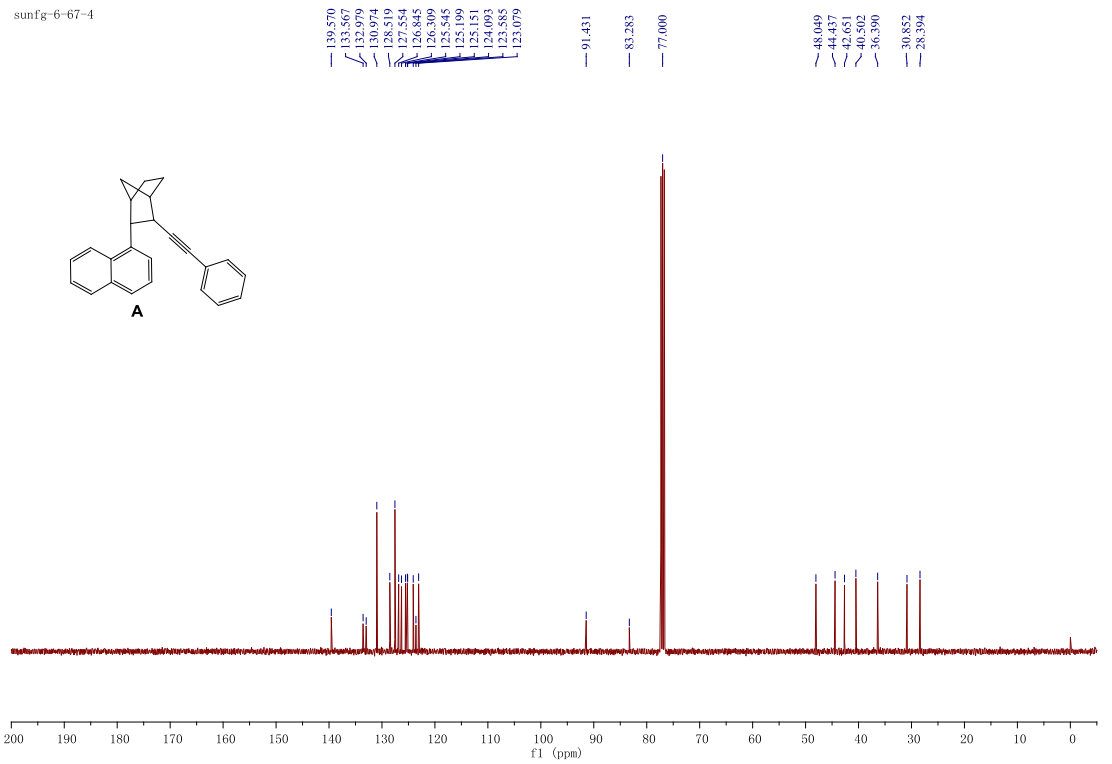

sunfg-5-119
7.260
7.244
7.239
6.878
6.866
6.861
4.435
3.792
3.514
3.499
3.485
3.256
3.238
3.261
2.093
2.078
2.076
2.061
2.046
2.030
O
I
O
CH3
2.22
2.00
2.08
3.10
2.08
2.07
2.08
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

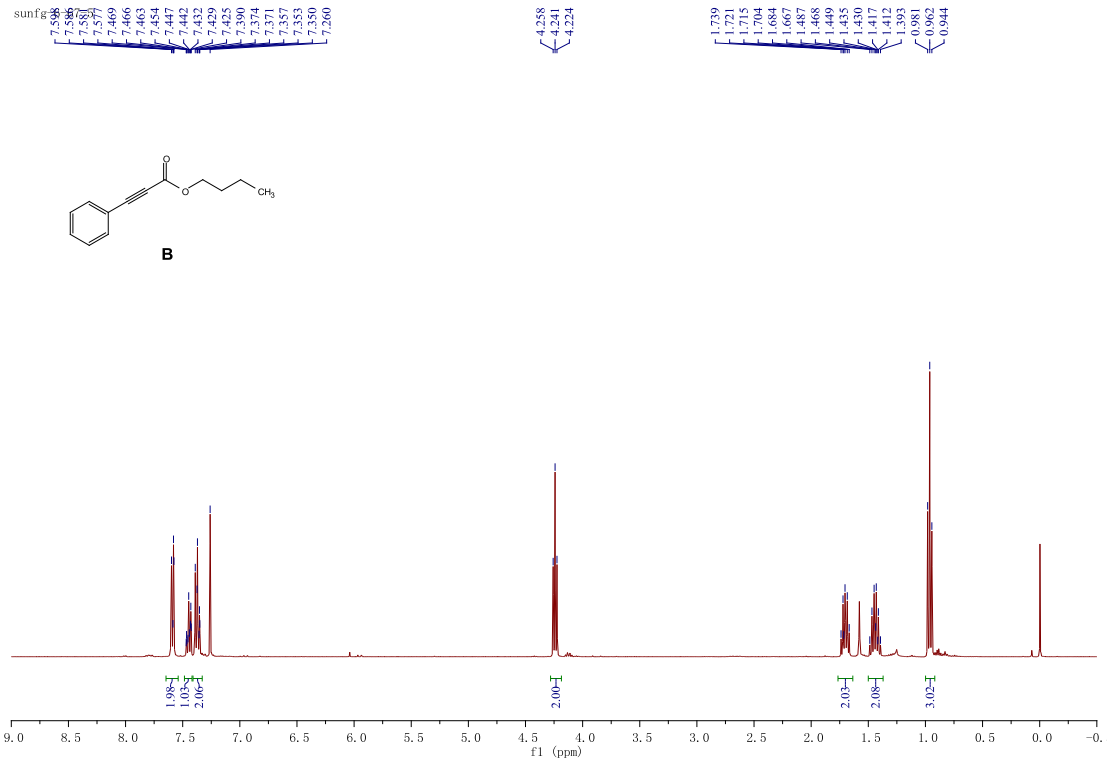

sunfg-4-37
7.79
7.77
7.54
7.52
7.40
7.38
7.36
6.99
6.99
6.97
6.95
4.64
0.99
0.15
I
O
Si
CH3
CH3
CH3
H3C
CH3
1.00
1.02
1.04
0.70
2.09
9.58
6.30
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

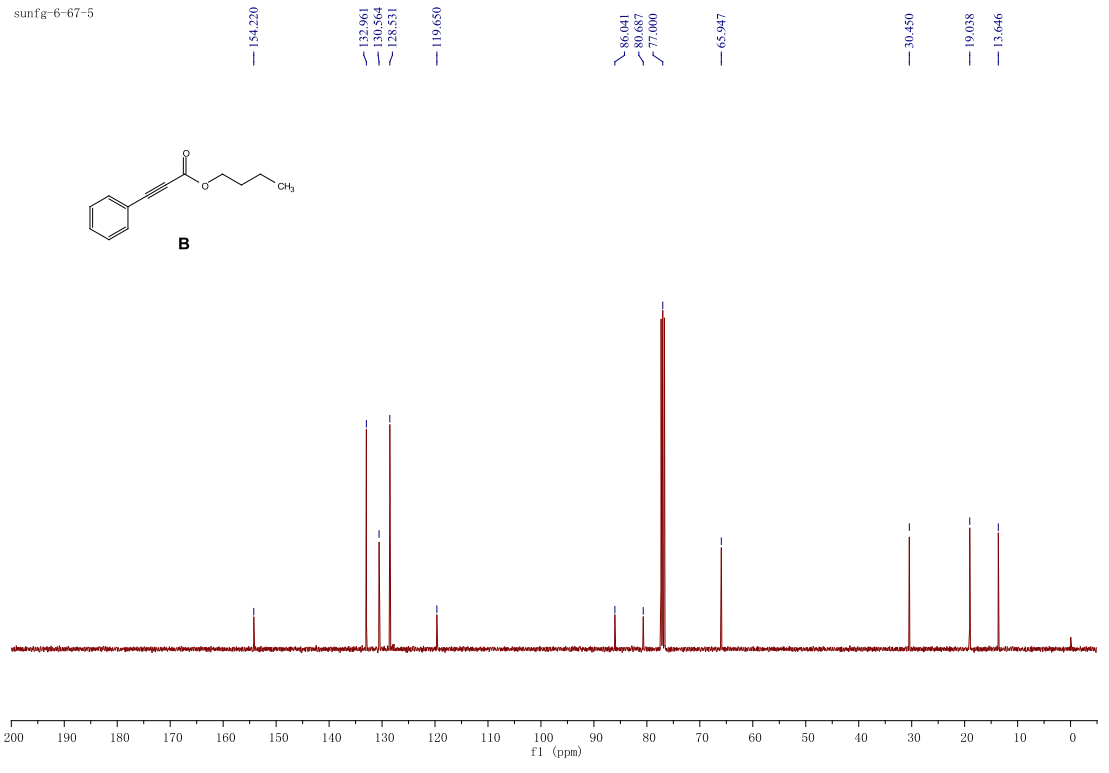

sunfg-4-37-C
142.871
138.597
128.475
128.114
127.344
95.757
77.318
77.000
76.683
69.359
25.945
18.386
-5.312
f1 (ppm)

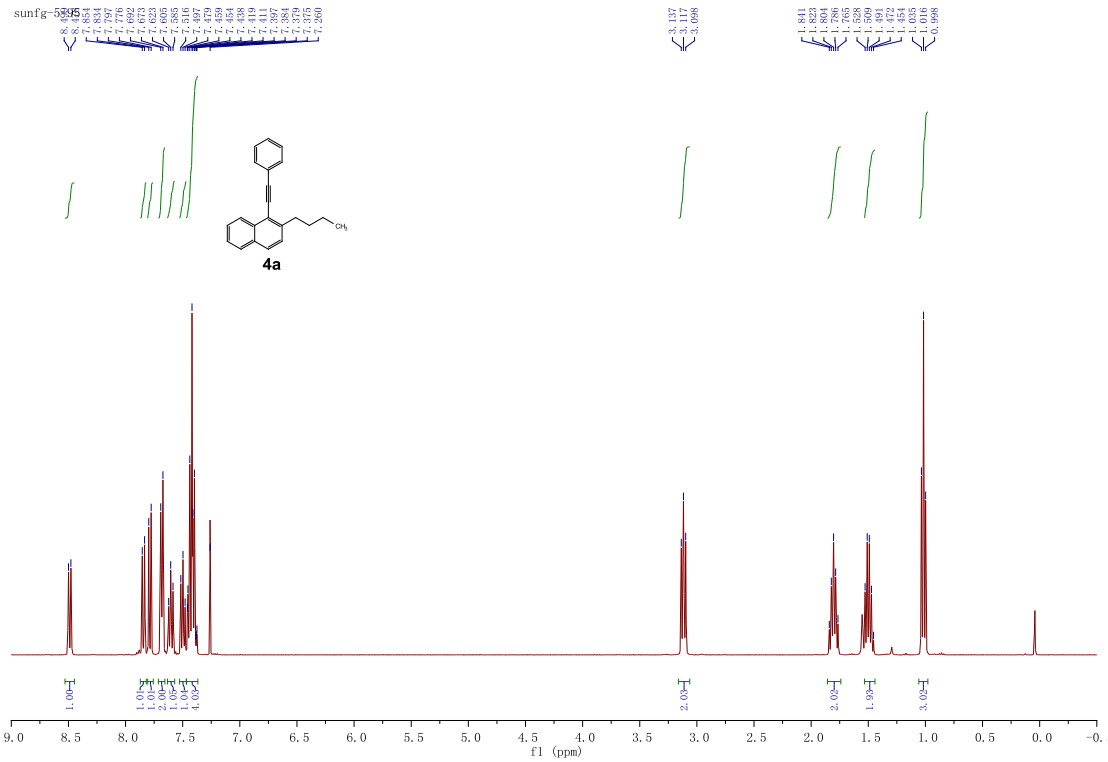

sunfg-nts
7.70
7.68
7.45
7.43
7.27
7.26
7.25
7.24
7.06
6.55
6.54
6.53
3.11
2.42
2.38
1.00
2.05
2.43
1.08
0.99
3.01
3.10
3.01
f1 (ppm)

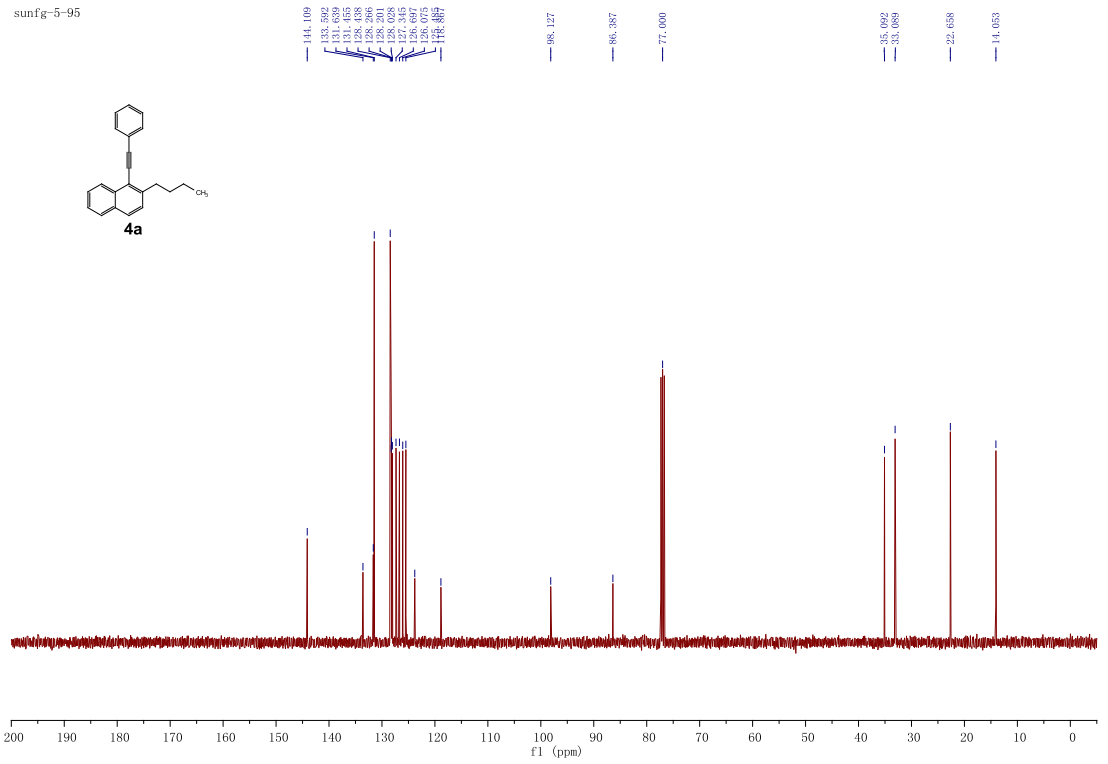

A
f1 (ppm)

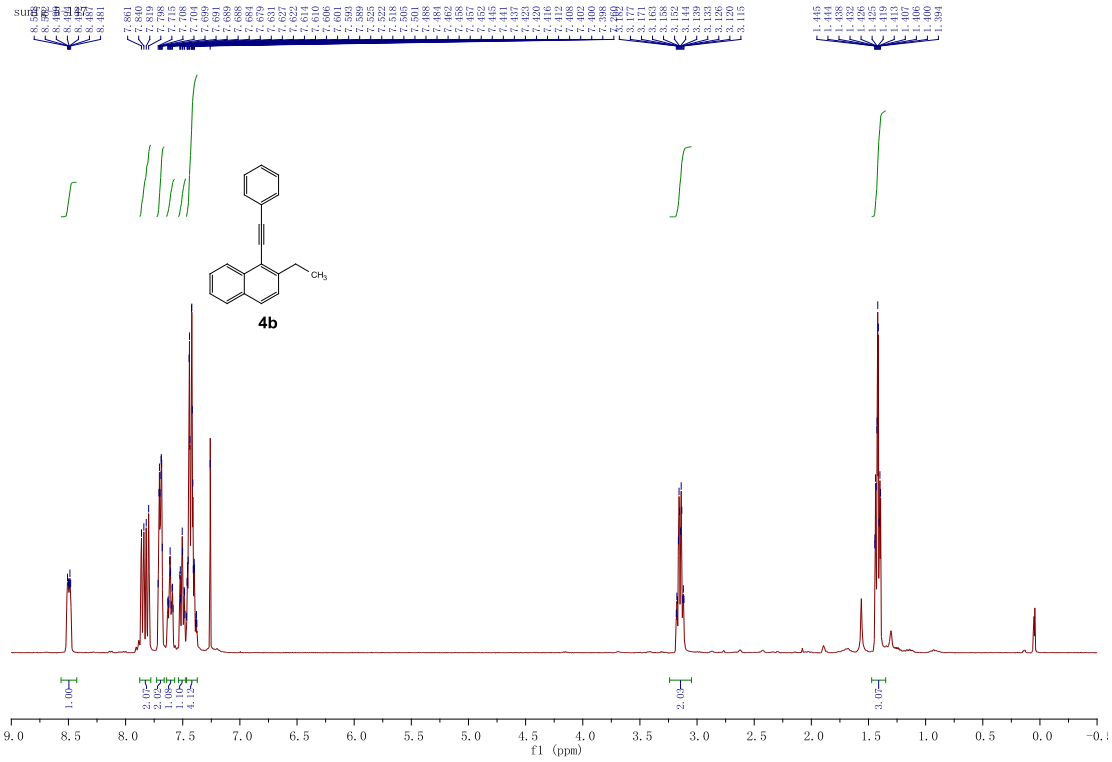

sunfg-6-67-4
A
f1 (ppm)

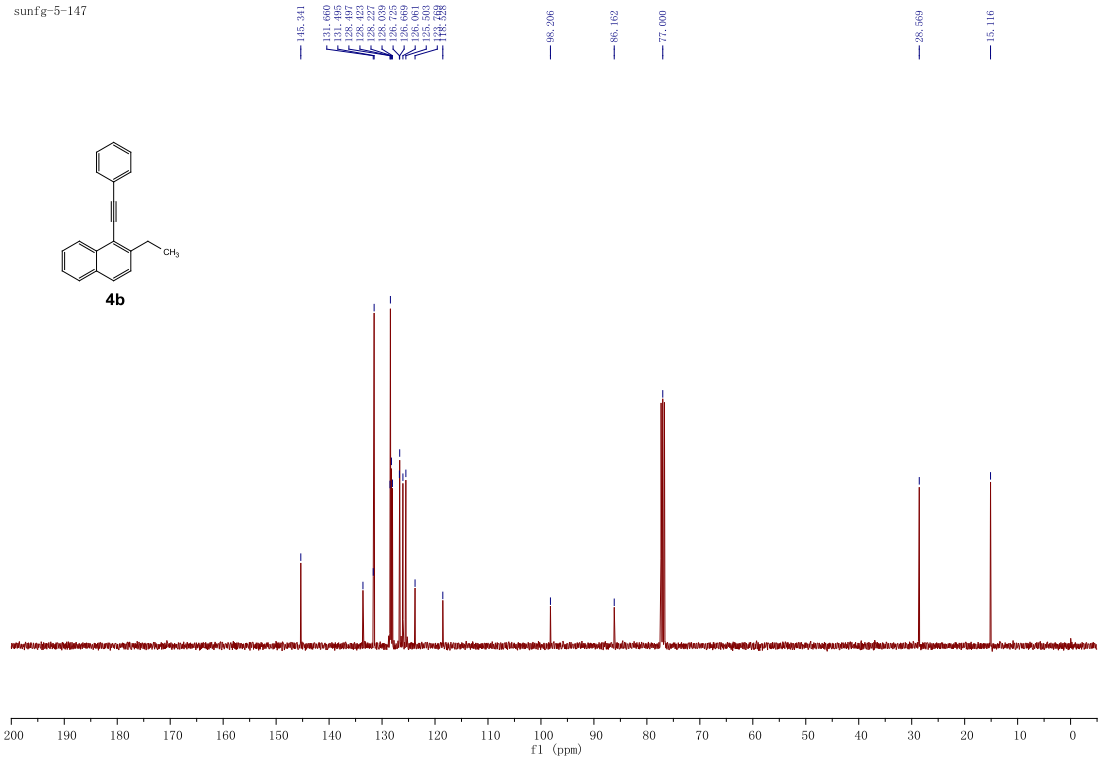

sunfg-6-67-1
7.598
7.595
7.587
7.584
7.577
7.469
7.466
7.463
7.454
7.447
7.442
7.432
7.429
7.425
7.390
7.374
7.371
7.357
7.353
7.350
7.260
4.258
4.241
4.224
1.739
1.721
1.715
1.704
1.684
1.667
1.487
1.468
1.449
1.435
1.430
1.417
1.412
1.393
0.981
0.962
0.944
O
O
CH3
B
1.98
1.05
2.06
2.00
2.03
2.08
3.02
f1 (ppm)

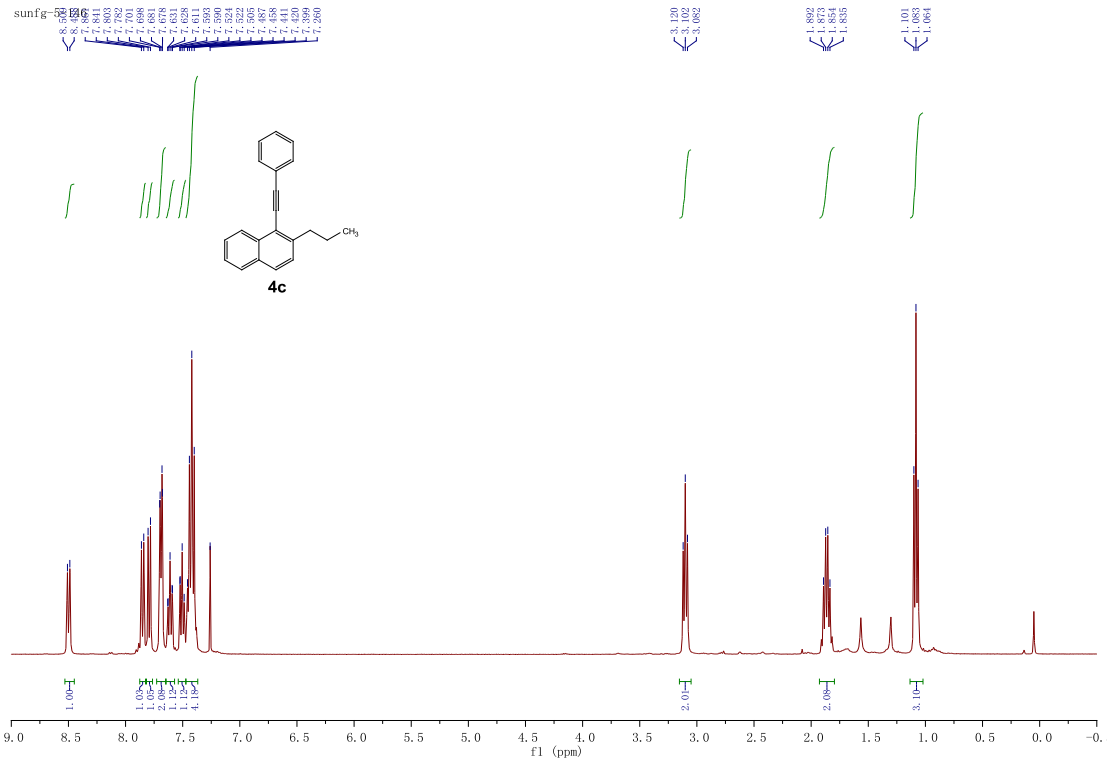

sunfg-6-67-5
154.220
132.961
130.564
128.531
119.650
86.041
80.687
77.000
65.947
30.450
19.038
13.646
O
O
CH3
B
f1 (ppm)

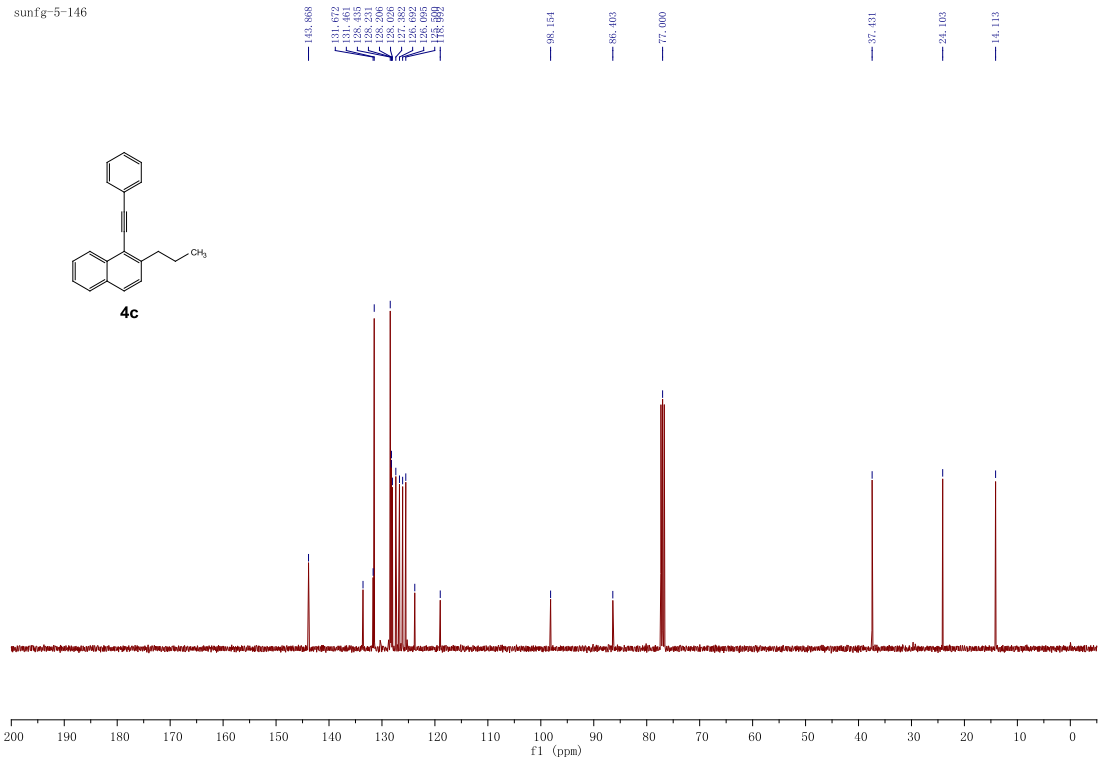

sunfg-5-95
4a
f1 (ppm)

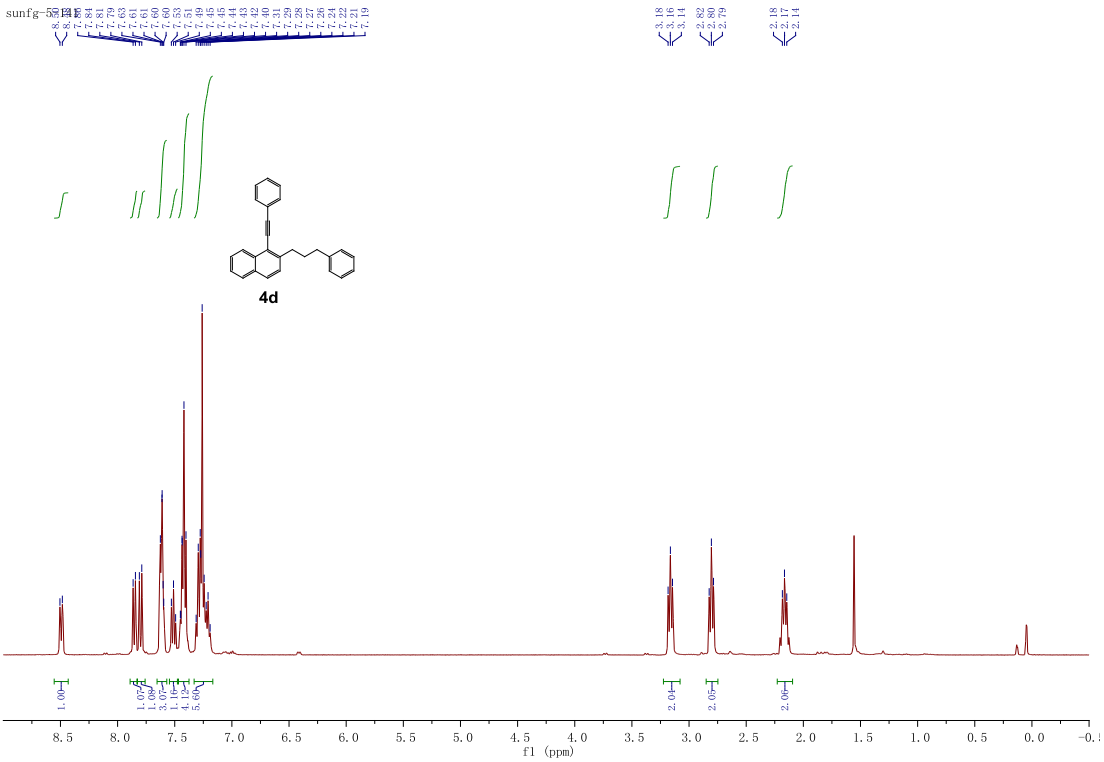

sunfg-5-95
4a
f1 (ppm)

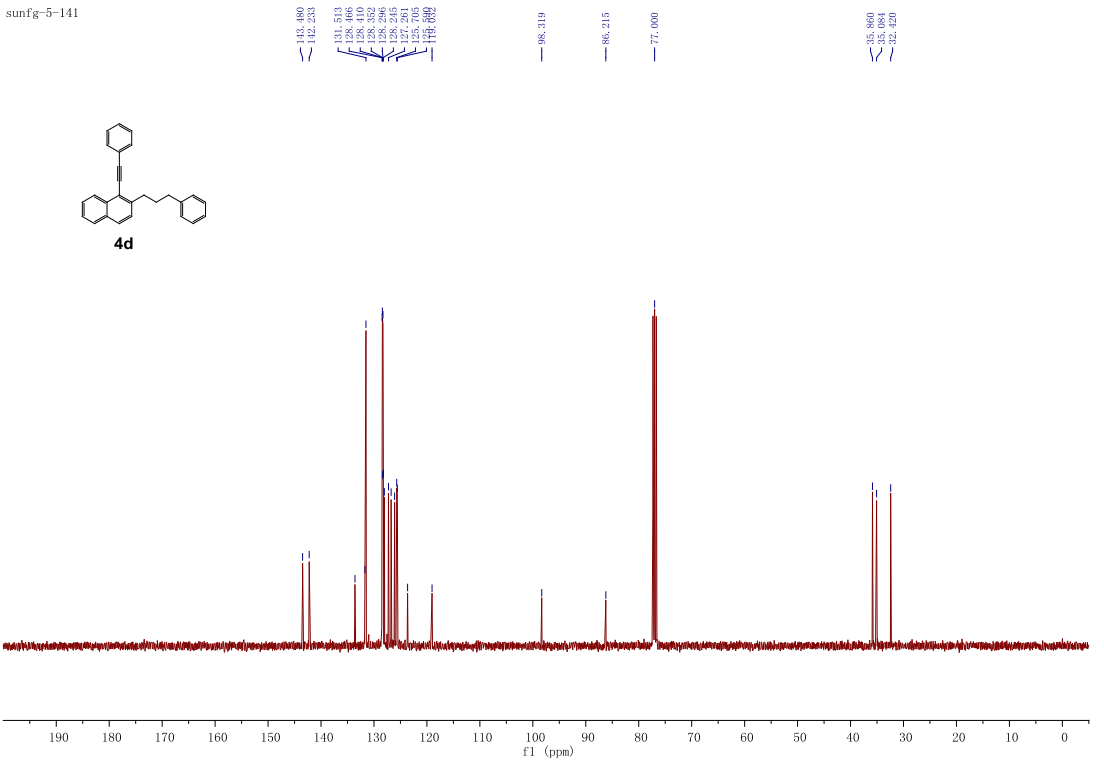

4b
CH3
1.00
2.07
2.02
1.08
1.15
4.12
2.03
3.07
f1 (ppm)

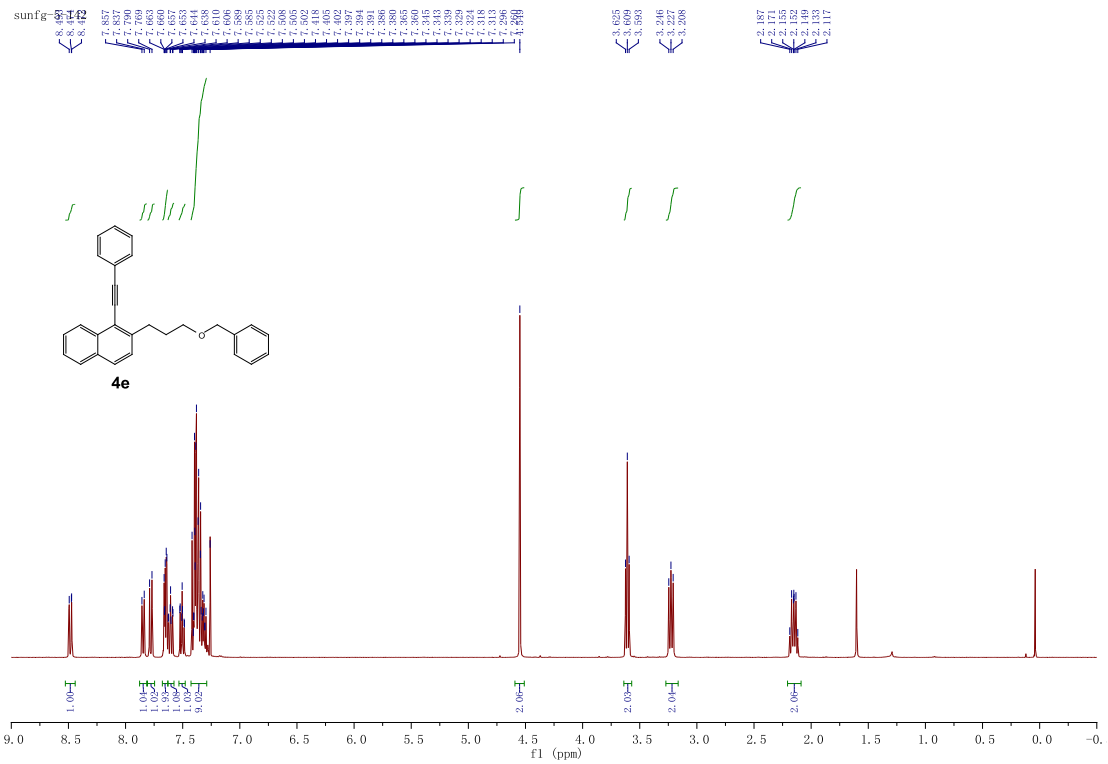

sunfg-5-147
4b
CH3
145.341
131.660
131.495
128.447
128.423
128.227
128.039
126.725
126.669
126.061
125.503
98.206
86.462
77.000
28.569
15.116
f1 (ppm)

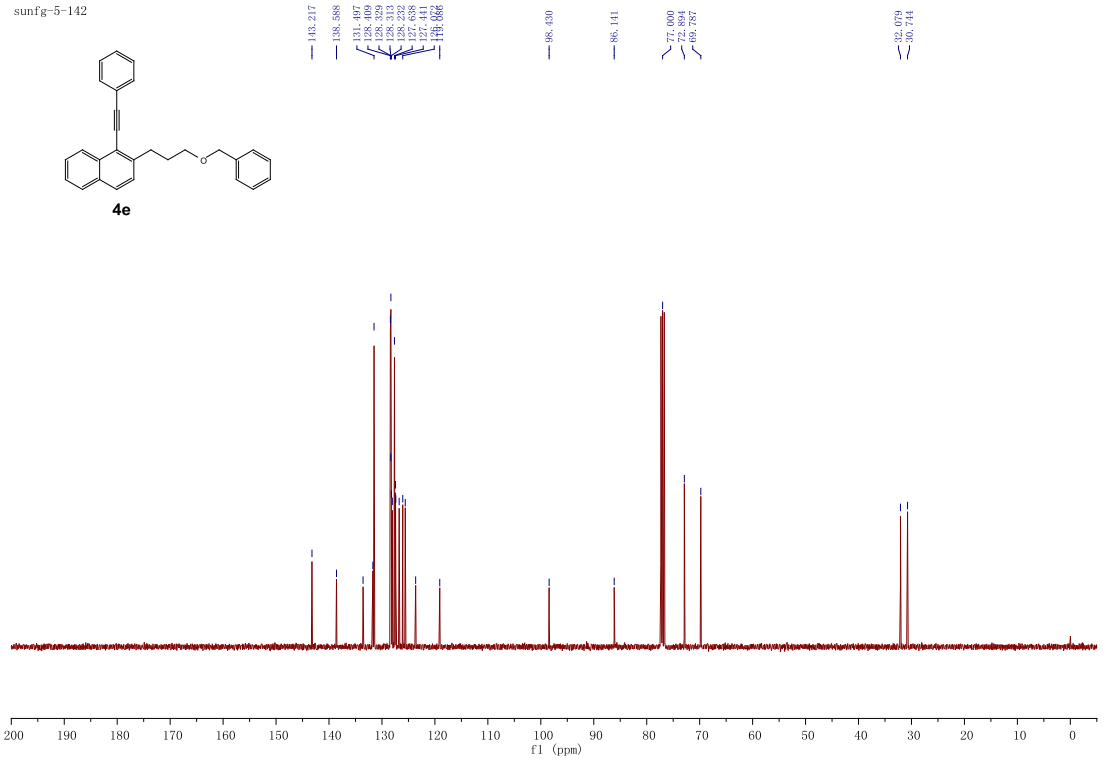

sunfg-5-146
3.120
3.102
3.082
1.892
1.873
1.854
1.835
1.101
1.083
1.064
CH3
4c
1.00
1.03
1.05
2.08
1.12
1.12
1.12
4.16
2.01
2.08
3.10
f1 (ppm)

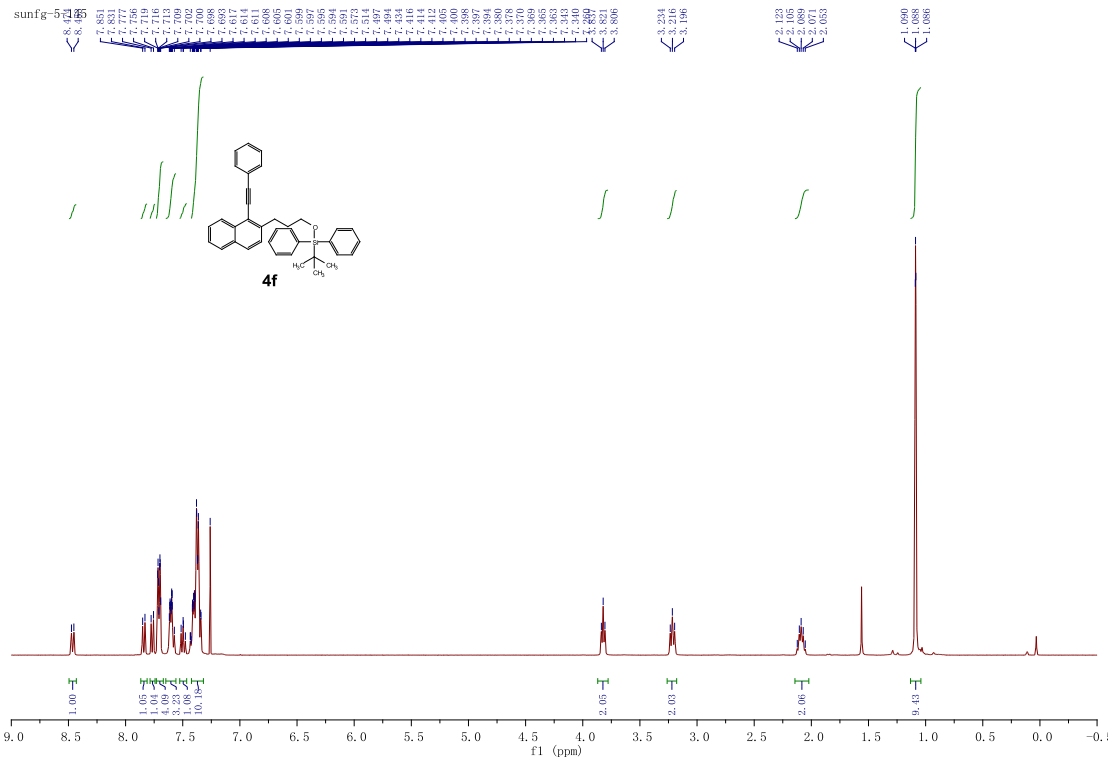

sunfg-5-146
143.868
131.672
131.461
128.435
128.231
128.206
128.095
127.382
126.692
126.095
98.154
86.403
77.000
37.431
24.103
14.113
CH3
4c
f1 (ppm)

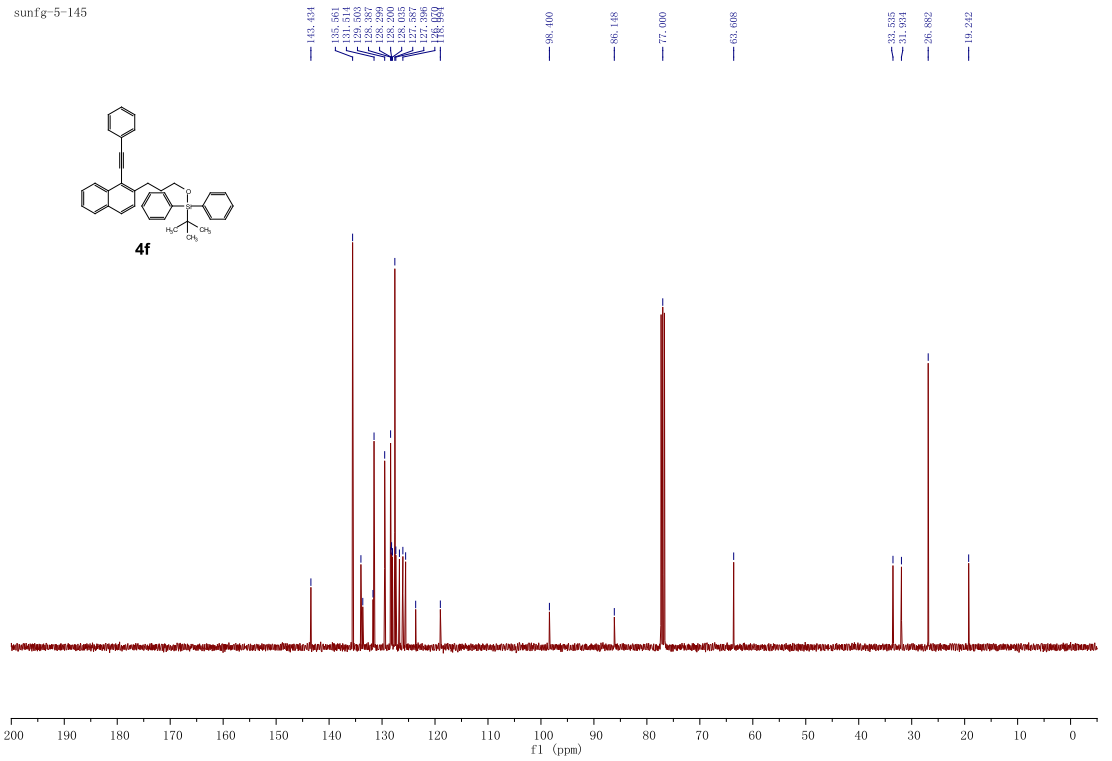

4d
f1 (ppm)

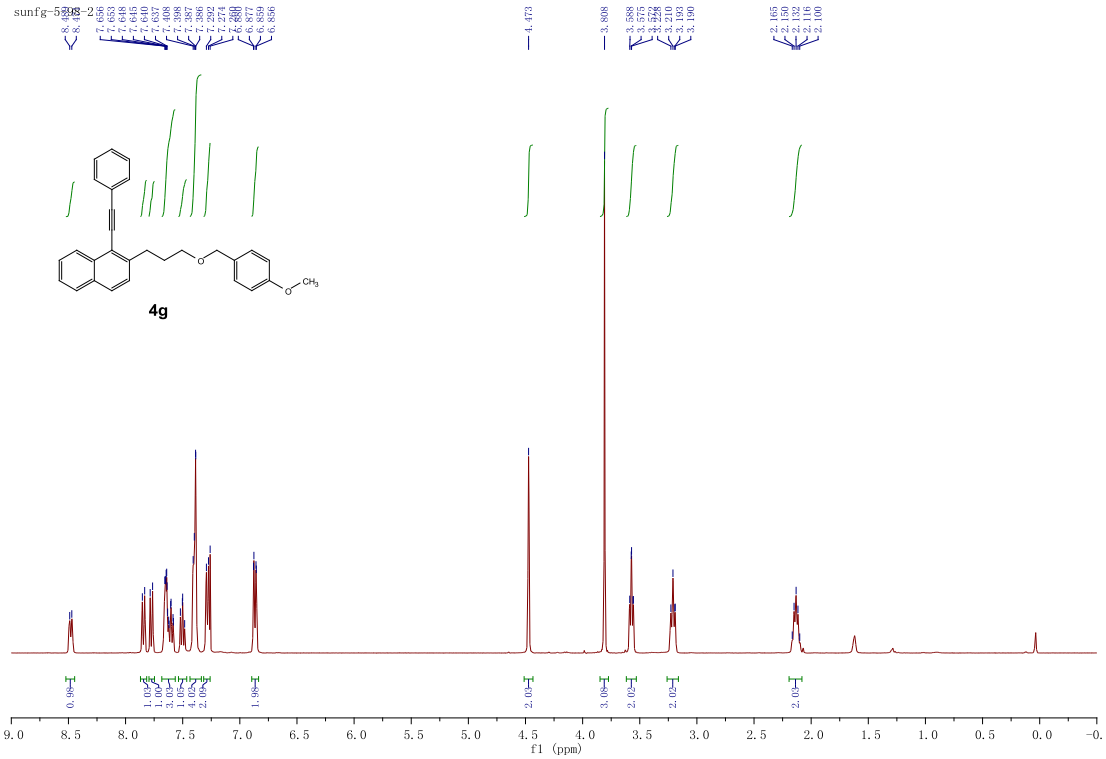

sunfg-5-141
4d
f1 (ppm)

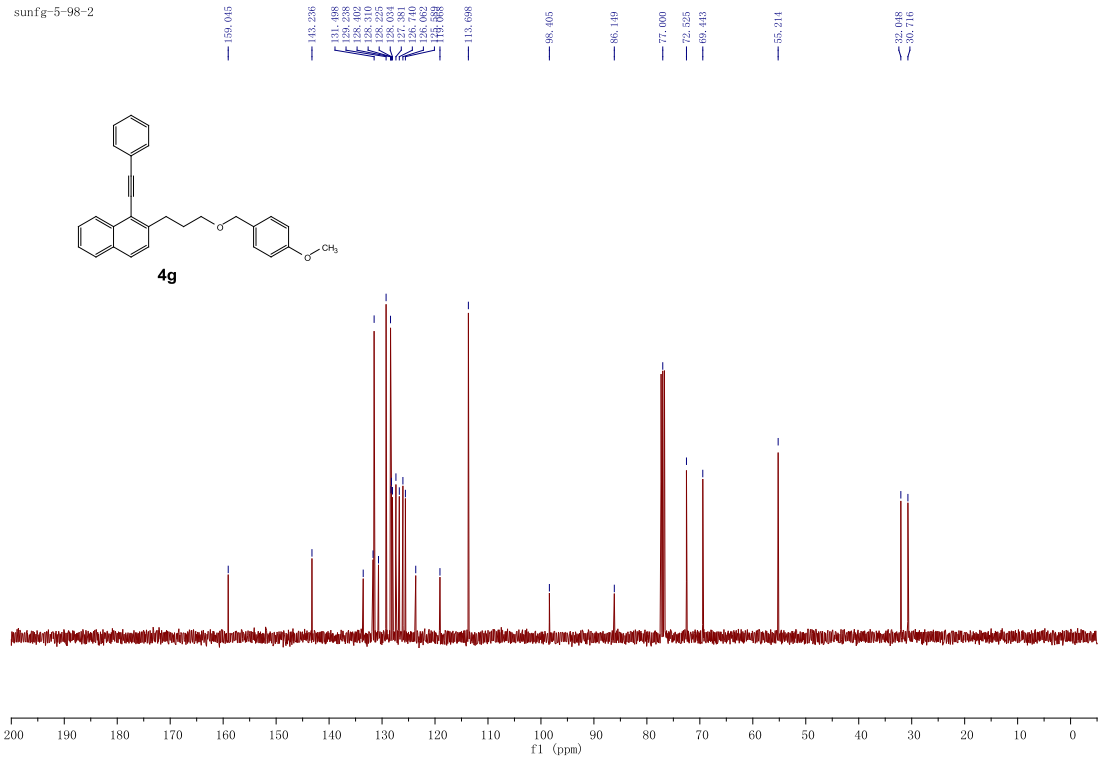

sunfg-5-142
4e
f1 (ppm)

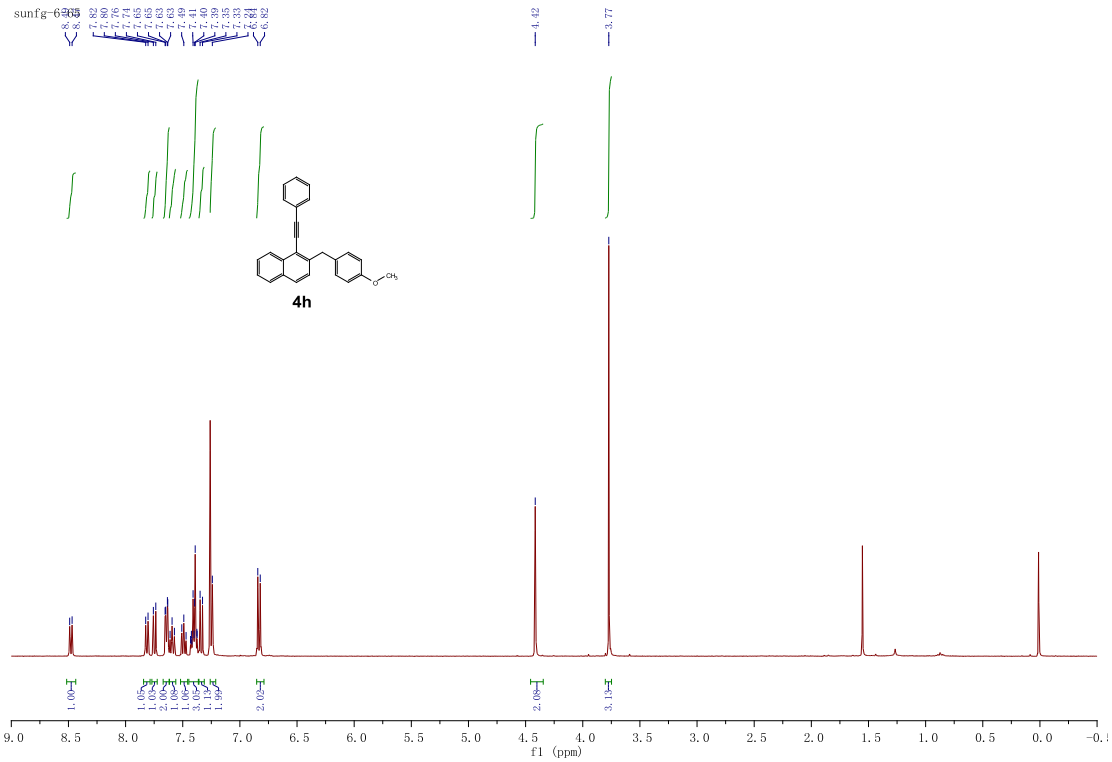

sunfg-5-142
4e
f1 (ppm)

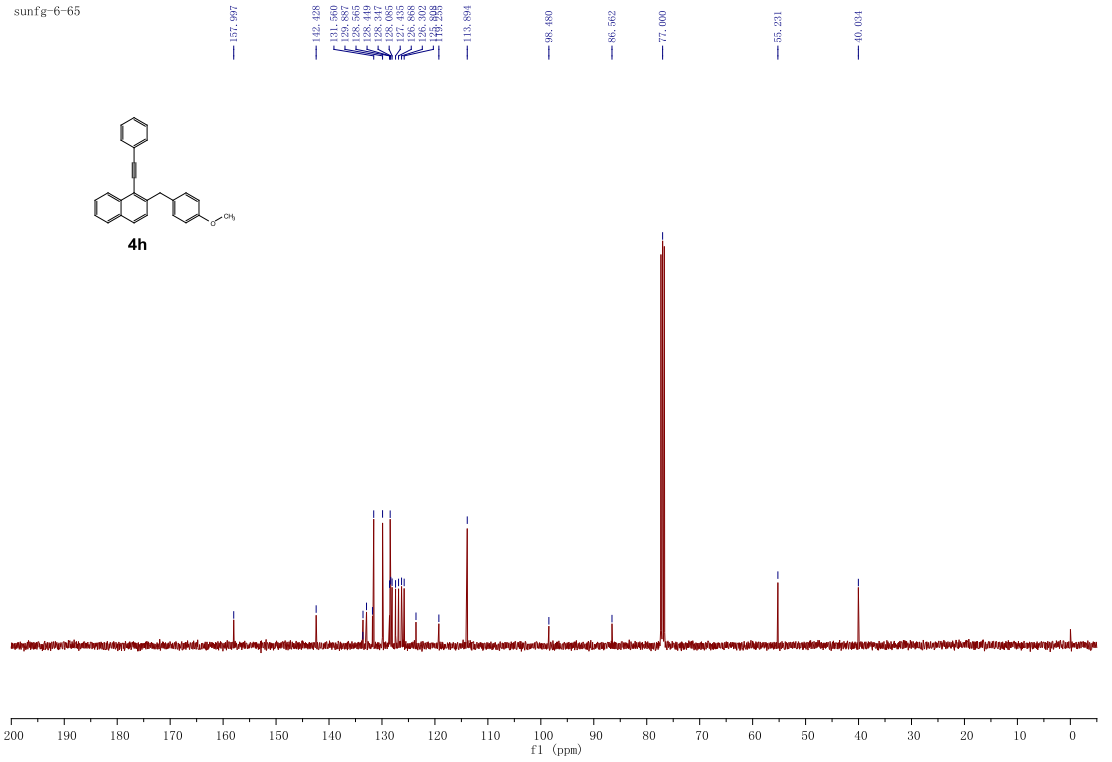

4f
f1 (ppm)

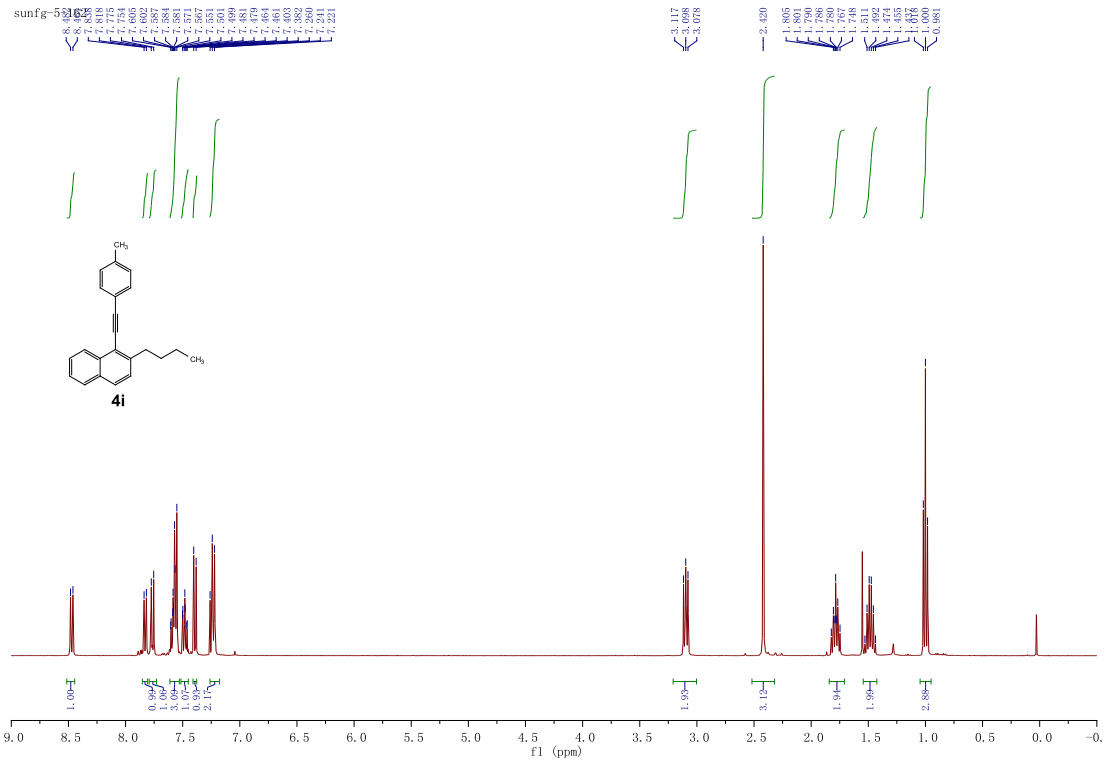

sunfg-5-145
4f
f1 (ppm)

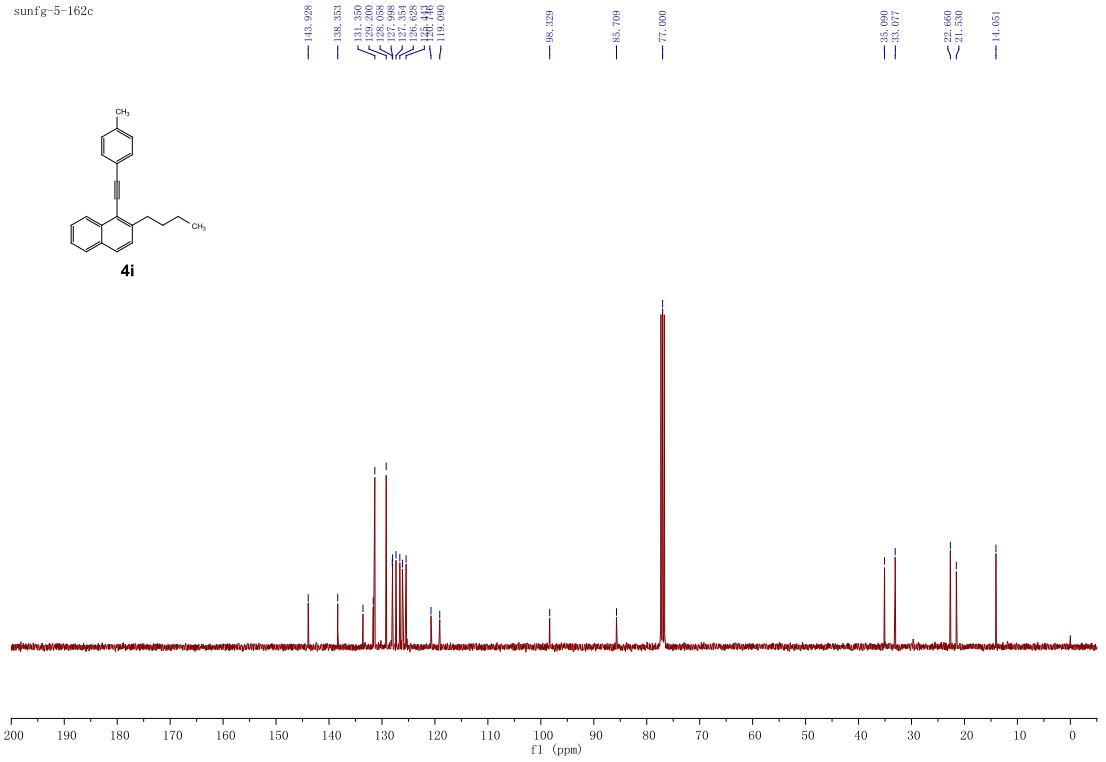

sunfg-5-98-2
4g
f1 (ppm)

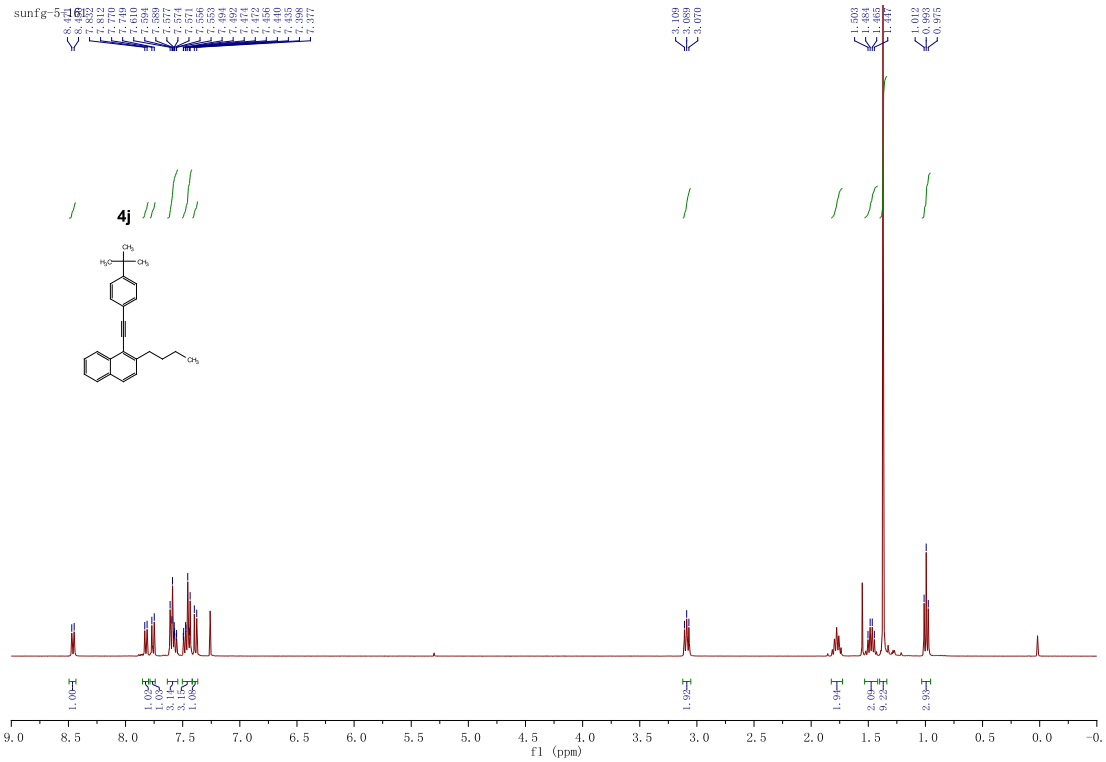

sunfg-5-98-2
4g
f1 (ppm)

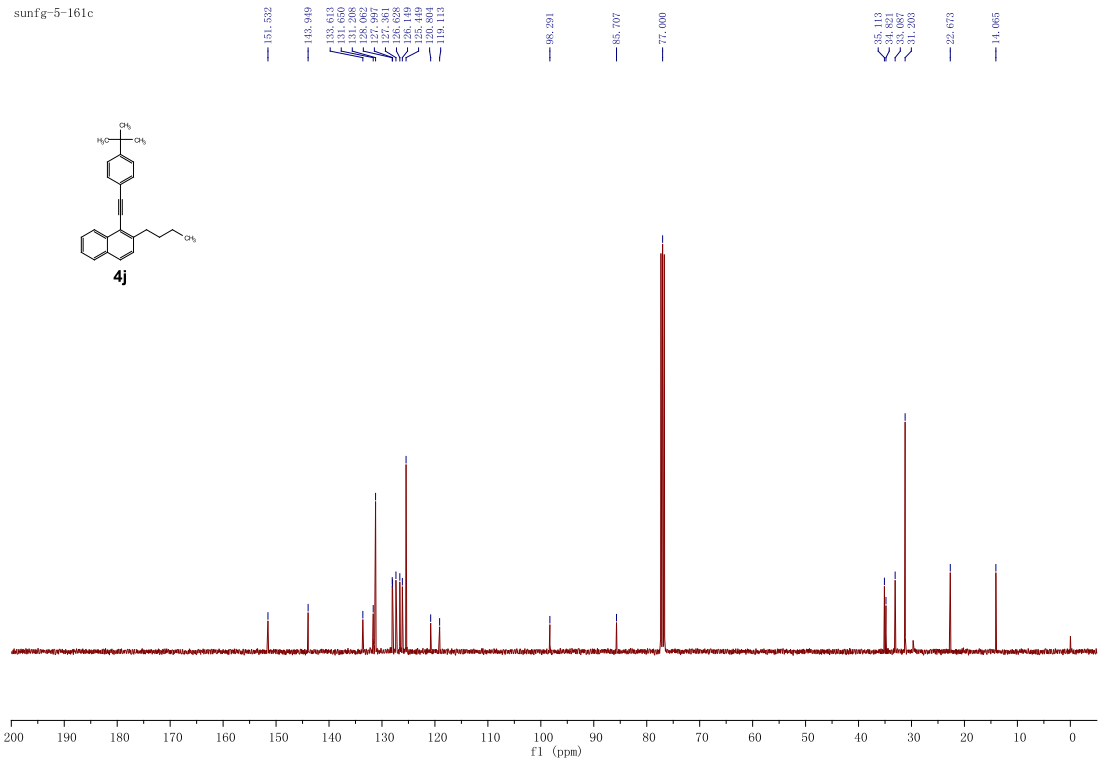

sunfg-6-65
4h
f1 (ppm)

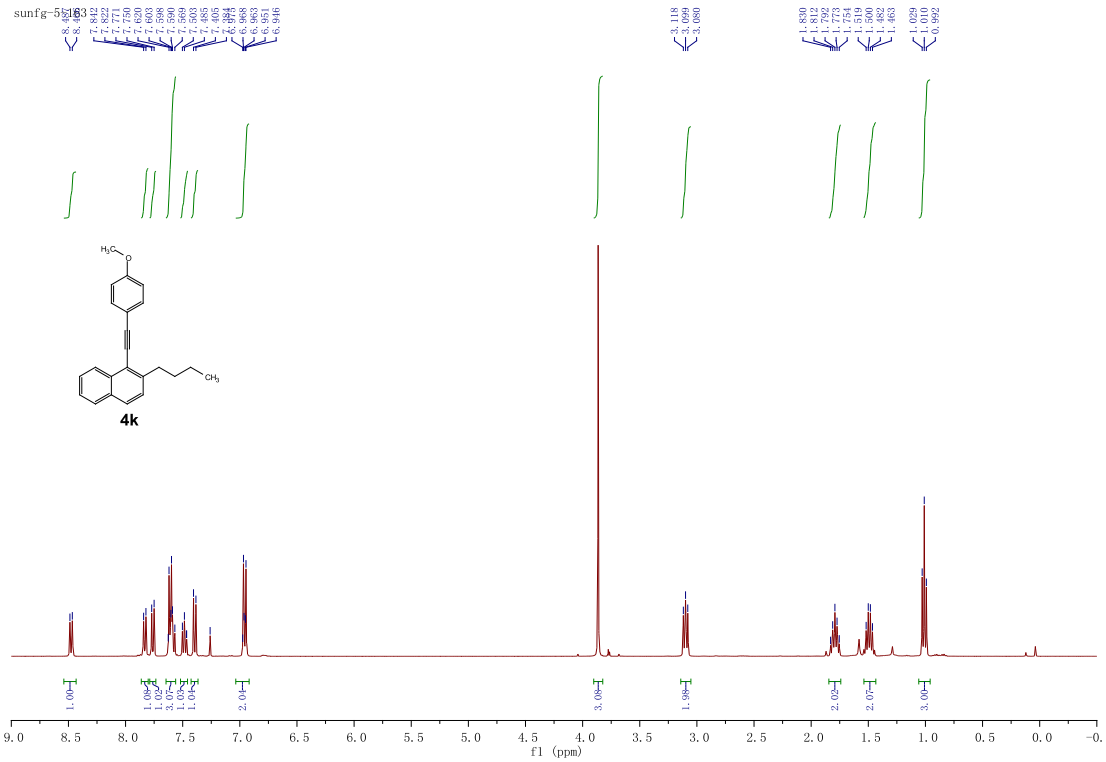

sunfg-6-65
4h
f1 (ppm)

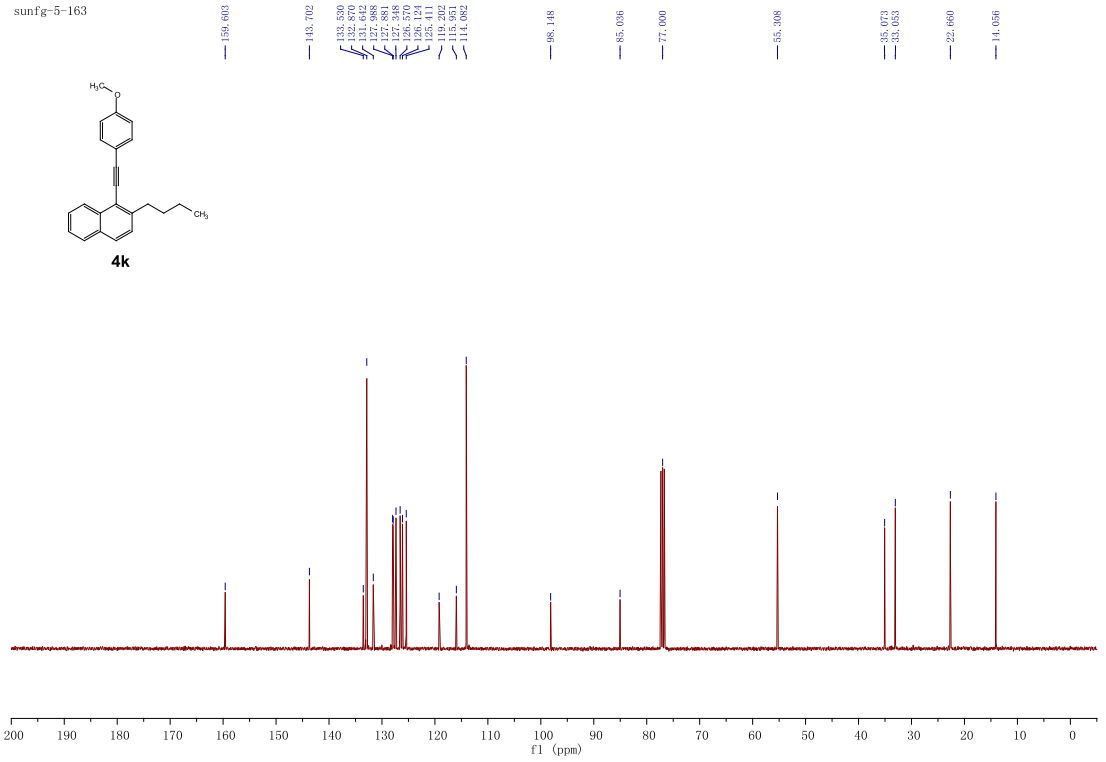

sunfg-5-162
8.493
8.485
7.818
7.775
7.754
7.605
7.602
7.587
7.584
7.581
7.571
7.567
7.551
7.500
7.499
7.481
7.479
7.464
7.463
7.403
7.382
7.269
7.241
7.221
3.117
3.098
3.078
2.420
1.805
1.801
1.790
1.786
1.780
1.767
1.748
1.511
1.492
1.474
1.455
1.437
1.000
0.981
CH3
CH3
4i
1.00
0.99
1.09
3.09
1.07
0.59
2.17
1.93
3.12
1.94
1.99
2.88
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

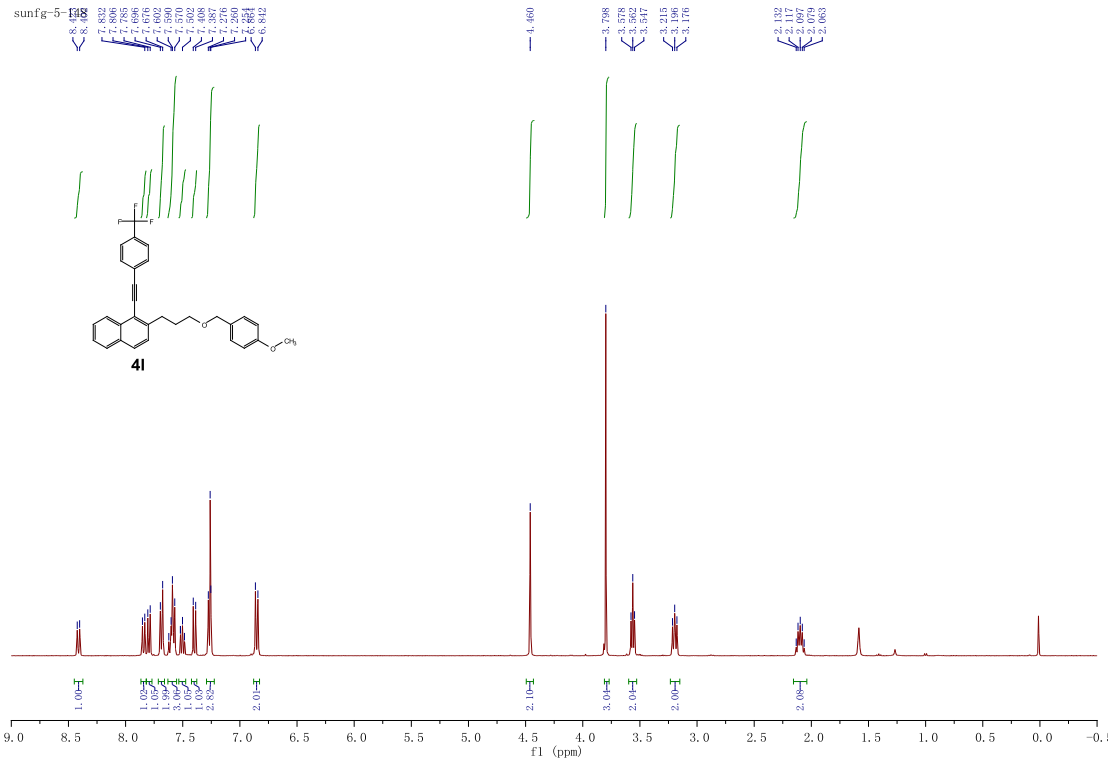

sunfg-5-162c
143.928
138.353
131.350
129.200
128.658
128.048
127.354
126.628
125.443
125.416
119.090
98.329
85.709
77.000
35.090
33.077
22.860
21.530
14.051
CH3
CH3
4i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

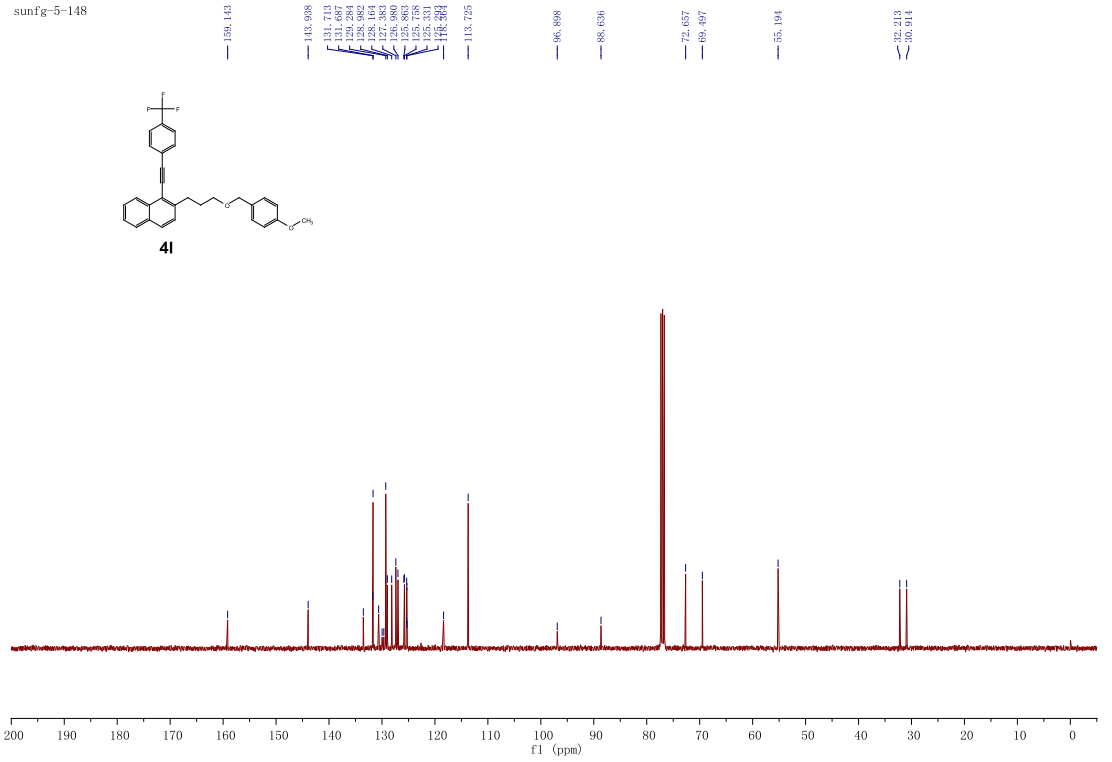

sunfg-5-161
8.477
8.475
7.832
7.812
7.770
7.749
7.610
7.594
7.589
7.577
7.574
7.571
7.556
7.553
7.494
7.492
7.474
7.472
7.456
7.440
7.435
7.398
7.377
3.109
3.089
3.070
1.503
1.484
1.465
1.447
1.012
0.993
0.975
4j
1.00
1.02
1.03
3.14
3.15
3.08
1.92
1.94
2.09
9.22
2.99
f1 (ppm)

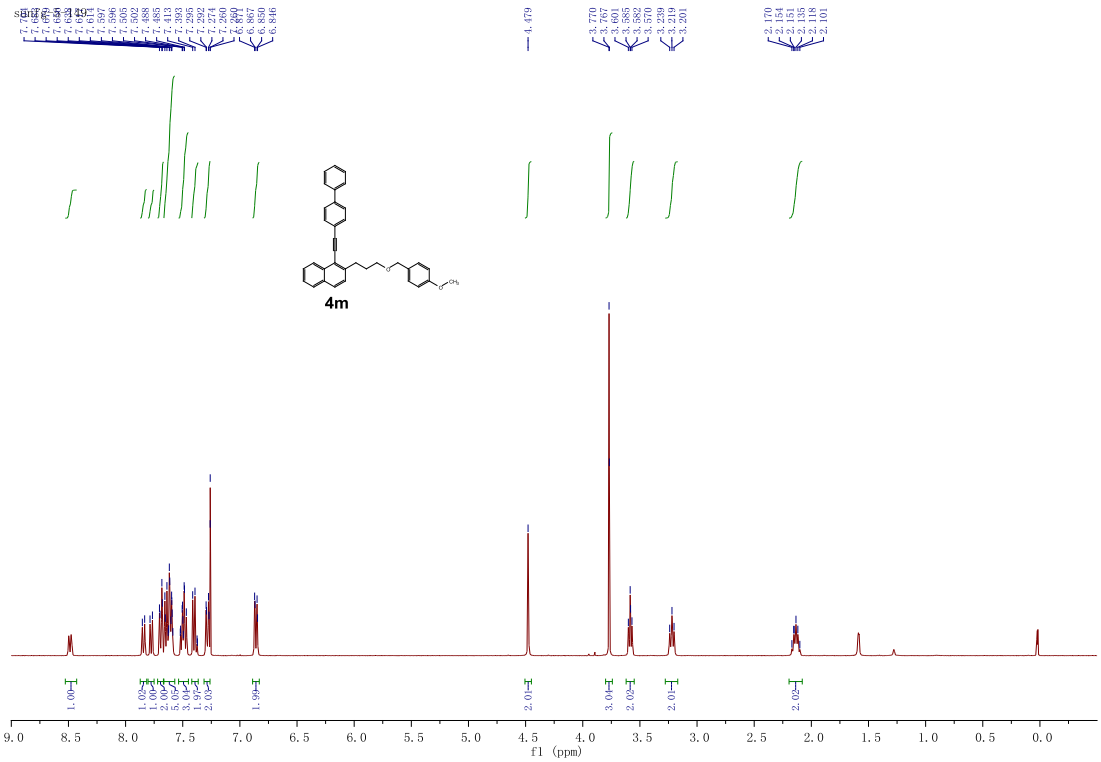

sunfg-5-161c
151.532
143.949
133.613
131.650
131.208
130.662
127.997
127.361
126.628
126.149
125.444
120.804
119.113
98.291
85.707
77.000
35.113
34.821
33.087
31.203
22.673
14.065
4j
f1 (ppm)

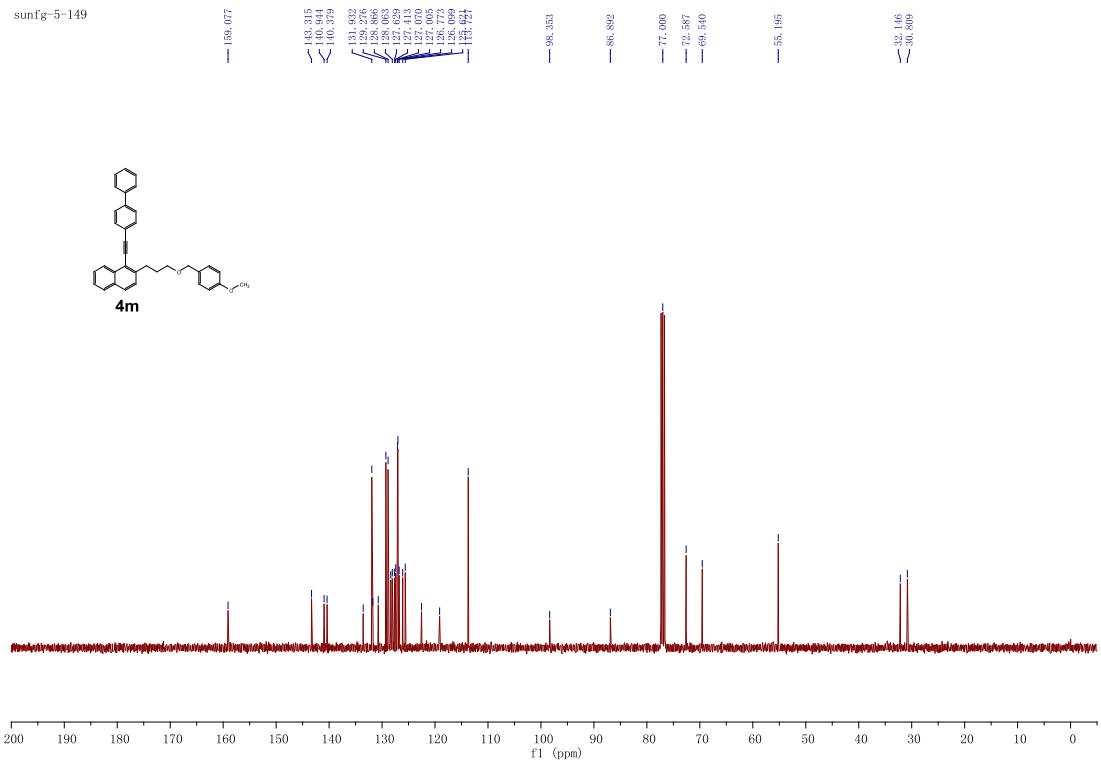

sunfg-5-163
8.482
8.480
7.842
7.822
7.771
7.750
7.730
7.663
7.598
7.590
7.569
7.563
7.485
7.405
6.968
6.963
6.951
6.946
3.118
3.094
3.068
1.830
1.812
1.792
1.773
1.754
1.519
1.500
1.482
1.463
1.029
1.010
0.992
4k
1.00
1.08
1.02
3.07
1.05
1.04
2.04
3.08
1.98
2.02
2.07
3.00
f1 (ppm)

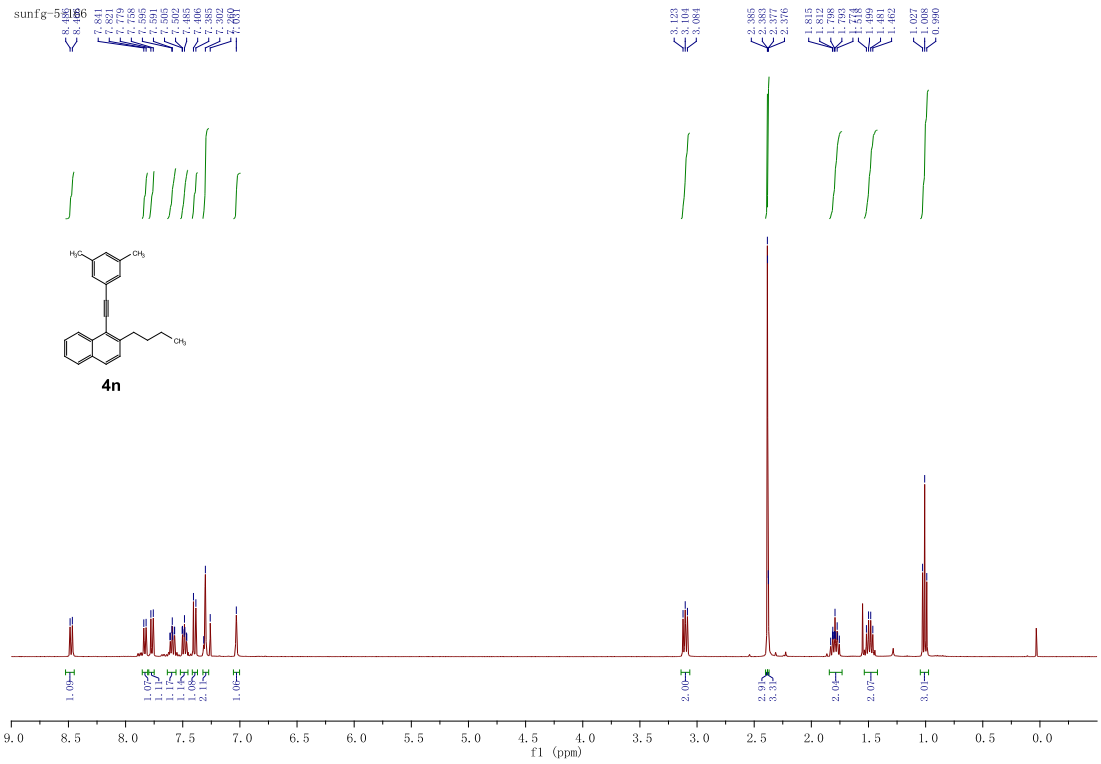

sunfg-5-163
159.603
143.702
133.530
132.870
131.664
127.988
127.881
127.348
126.570
126.114
125.411
119.202
115.951
114.082
98.148
85.036
77.000
55.308
35.073
33.053
22.660
14.056
4k
f1 (ppm)

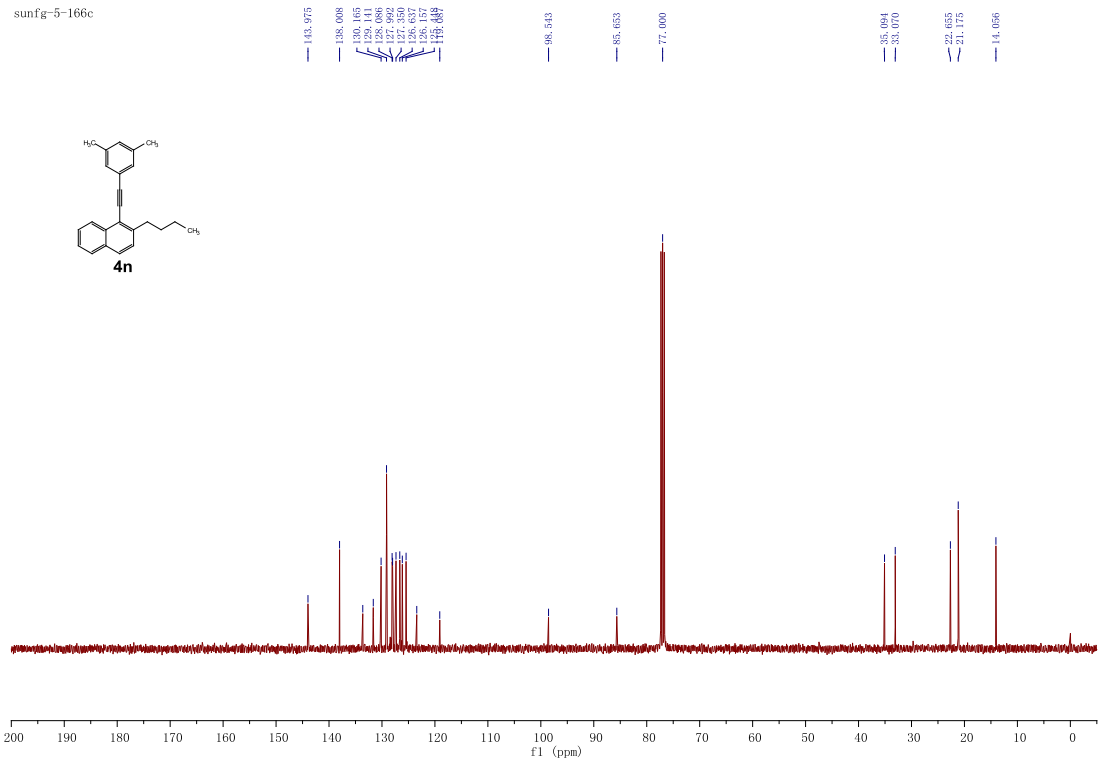

sunfg-5-148
4.460
3.798
3.578
3.562
3.547
3.215
3.196
3.176
2.132
2.117
2.097
2.079
2.063
4l
f1 (ppm)

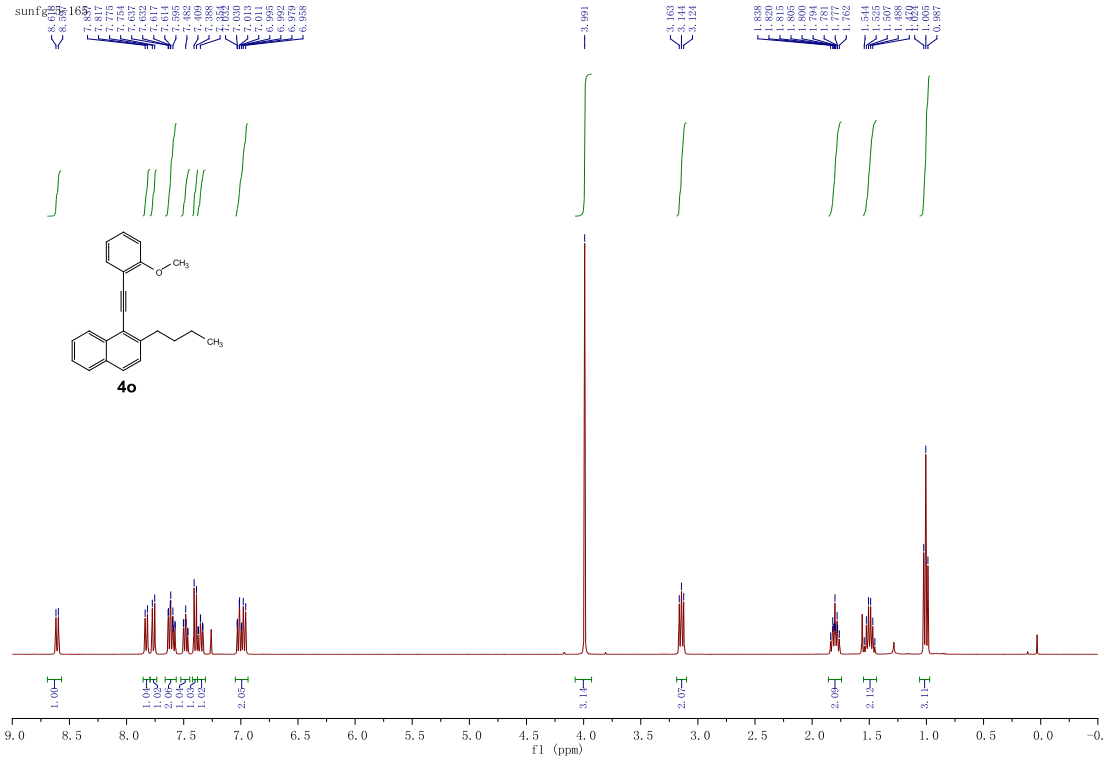

sunfg-5-148
159.143
143.938
131.713
131.687
129.394
128.982
128.164
127.383
126.980
125.963
125.758
125.331
113.725
96.898
88.636
72.657
69.497
55.194
32.213
30.944
4l
f1 (ppm)

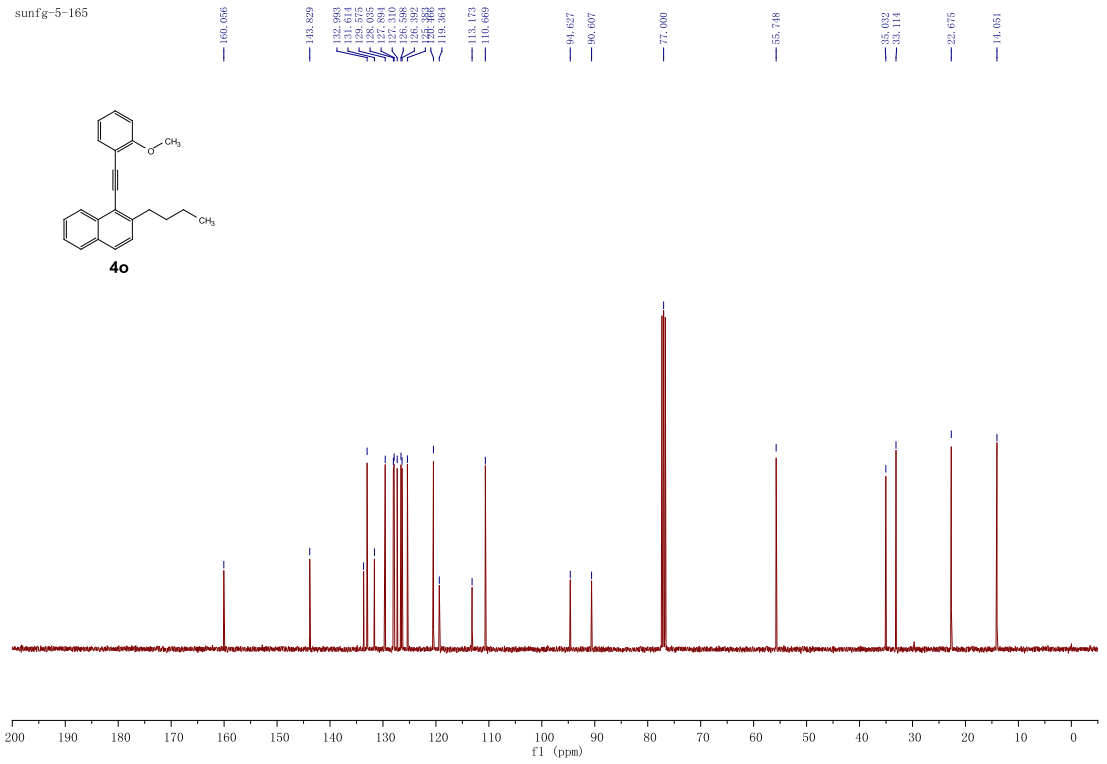

4m
f1 (ppm)

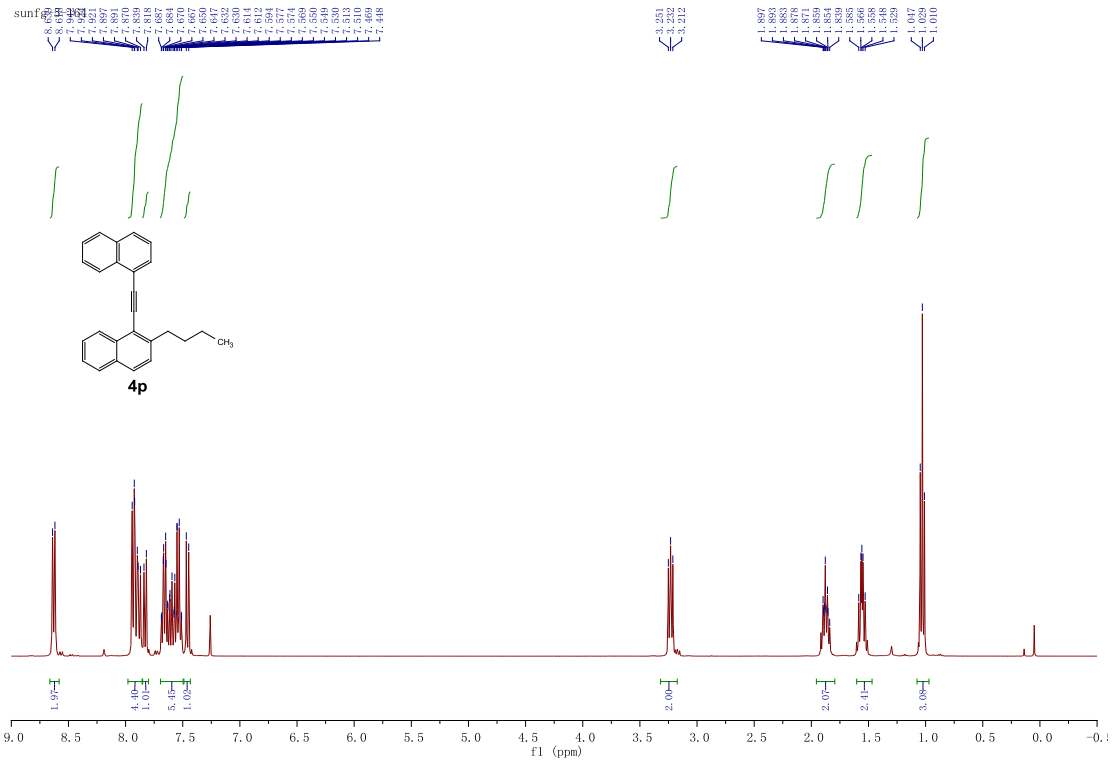

sunfg-5-149
4m
f1 (ppm)

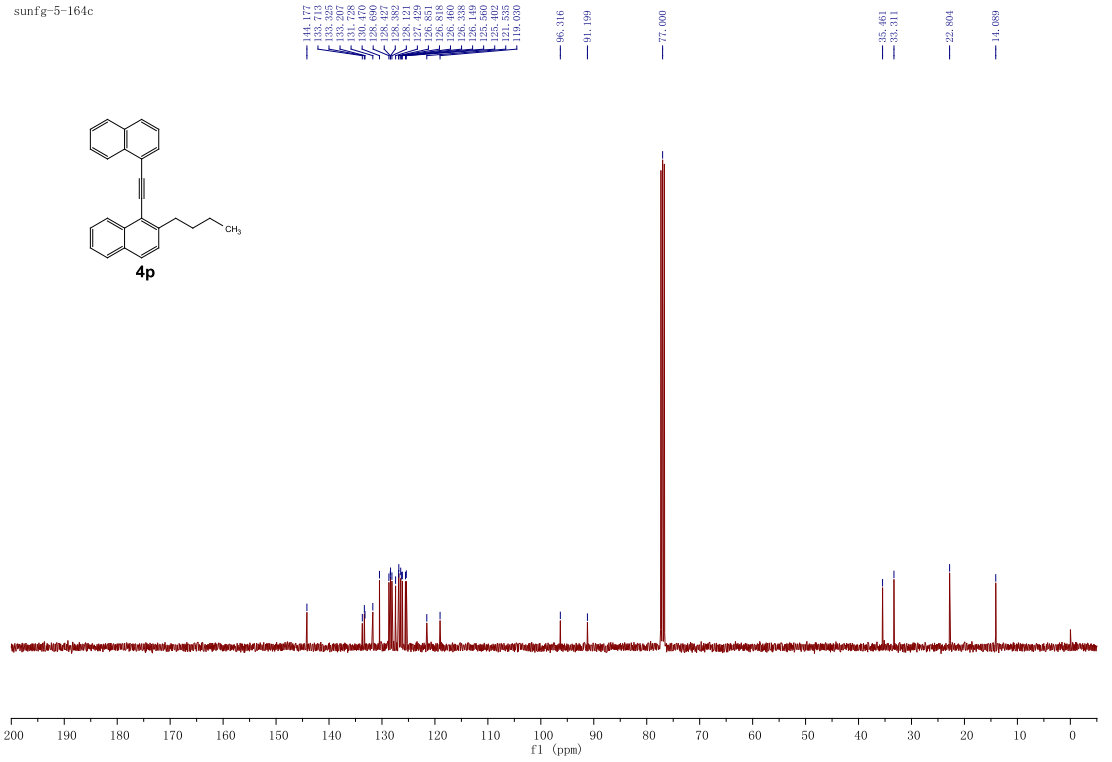

sunfg-5-166c
4n
f1 (ppm)

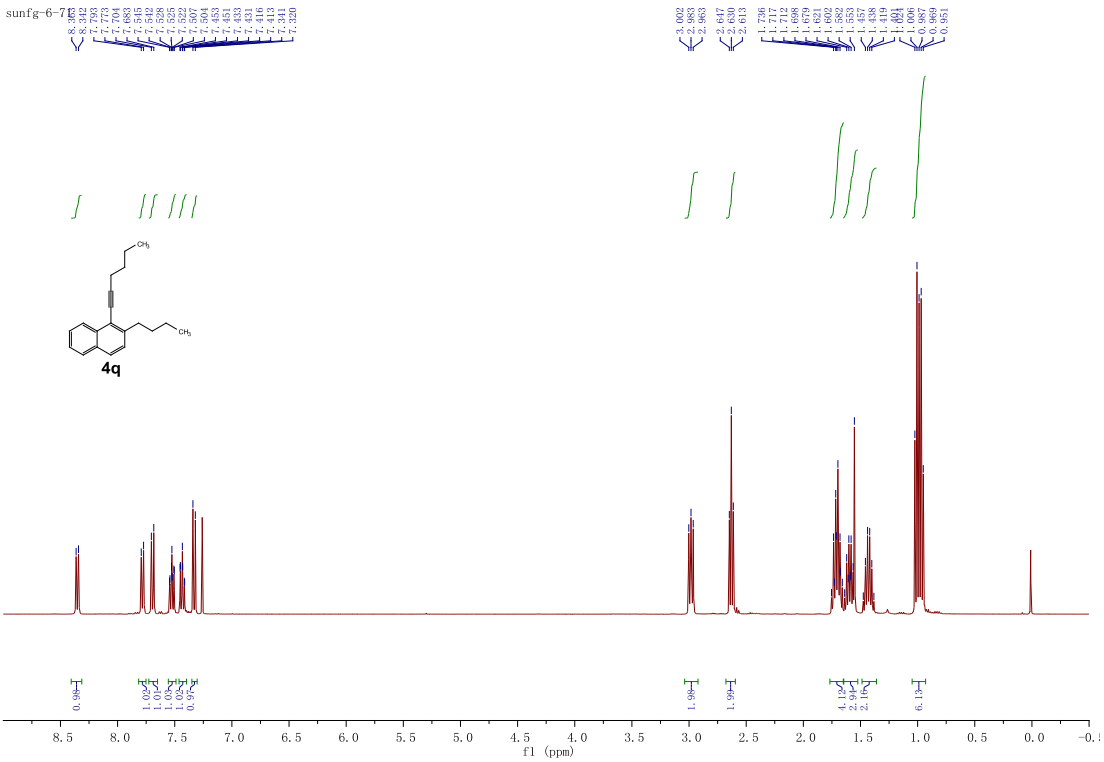

sunfg-5-166c
4n
f1 (ppm)

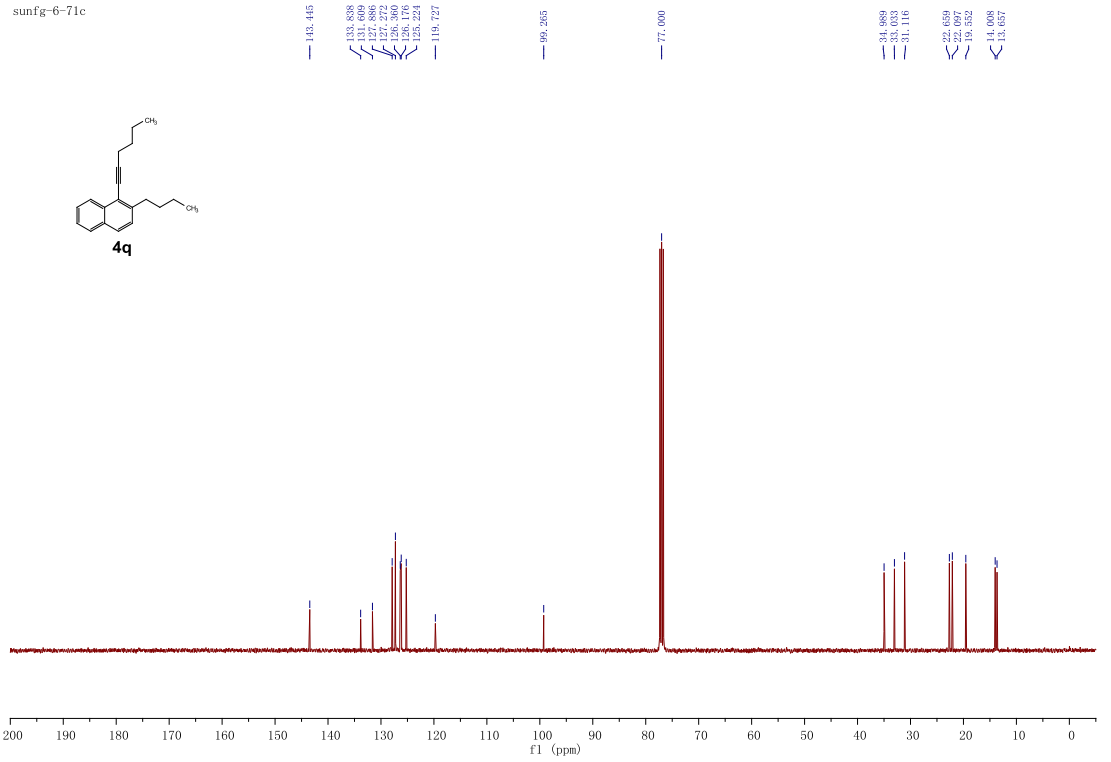

sunfg-5-165
8.637
8.557
7.847
7.775
7.754
7.637
7.632
7.617
7.614
7.595
7.482
7.469
7.398
7.354
7.052
7.050
7.013
7.011
6.995
6.992
6.979
6.958
3.991
3.163
3.144
3.124
1.838
1.830
1.815
1.805
1.800
1.794
1.781
1.777
1.762
1.544
1.525
1.507
1.488
1.470
1.065
0.887
4o
1.06
1.04
1.02
2.08
1.06
1.03
1.02
2.05
3.14
2.07
2.09
2.12
3.14
f1 (ppm)

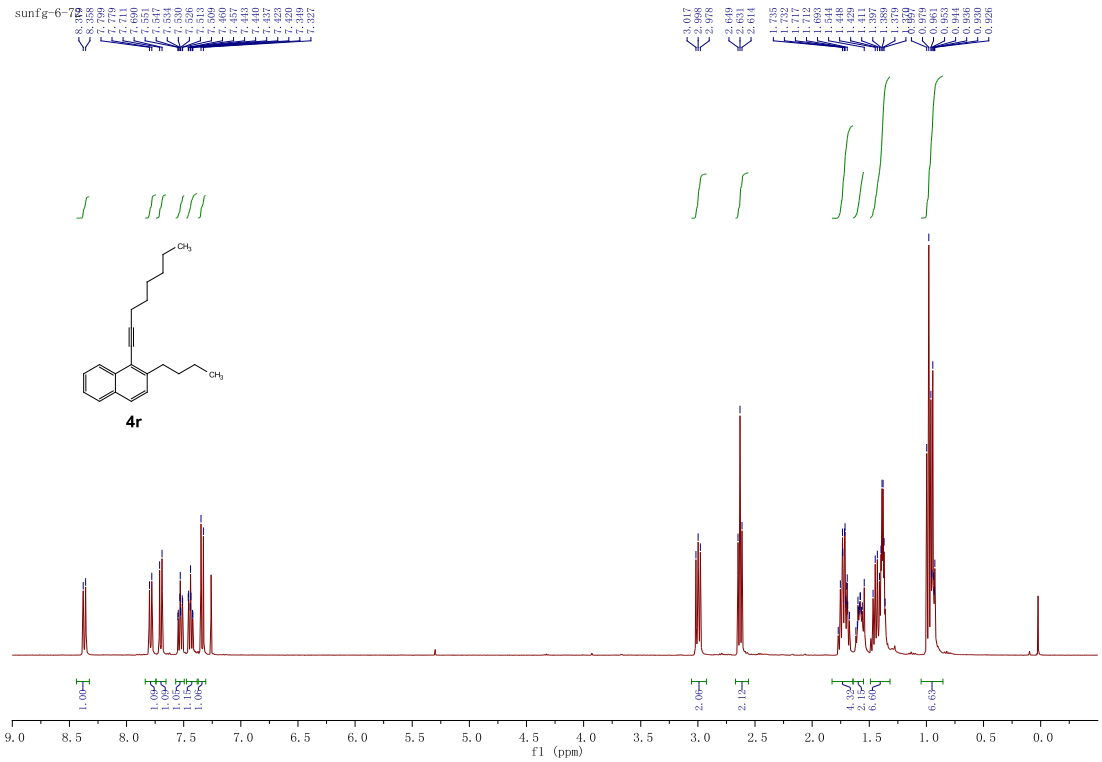

sunfg-5-165
160.056
143.829
132.903
131.614
129.575
128.035
127.894
127.310
126.598
126.392
119.364
113.173
110.669
94.827
90.607
77.000
55.748
35.032
33.114
22.675
14.051
4o
f1 (ppm)

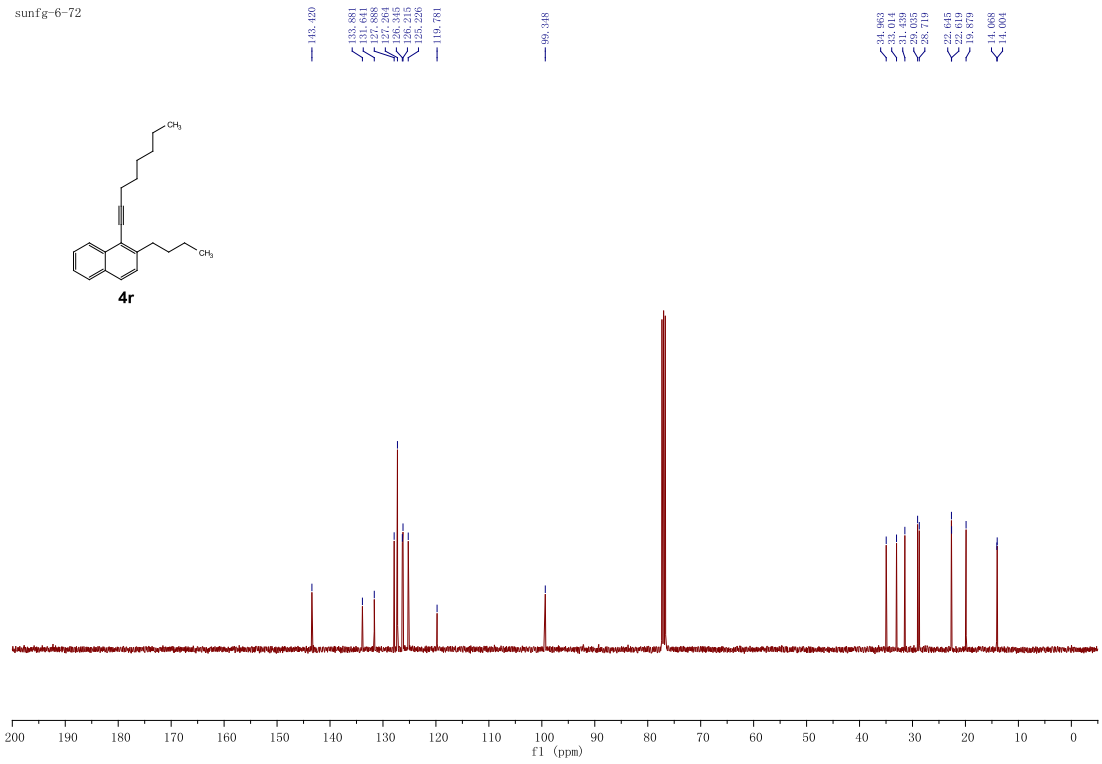

4p
f1 (ppm)

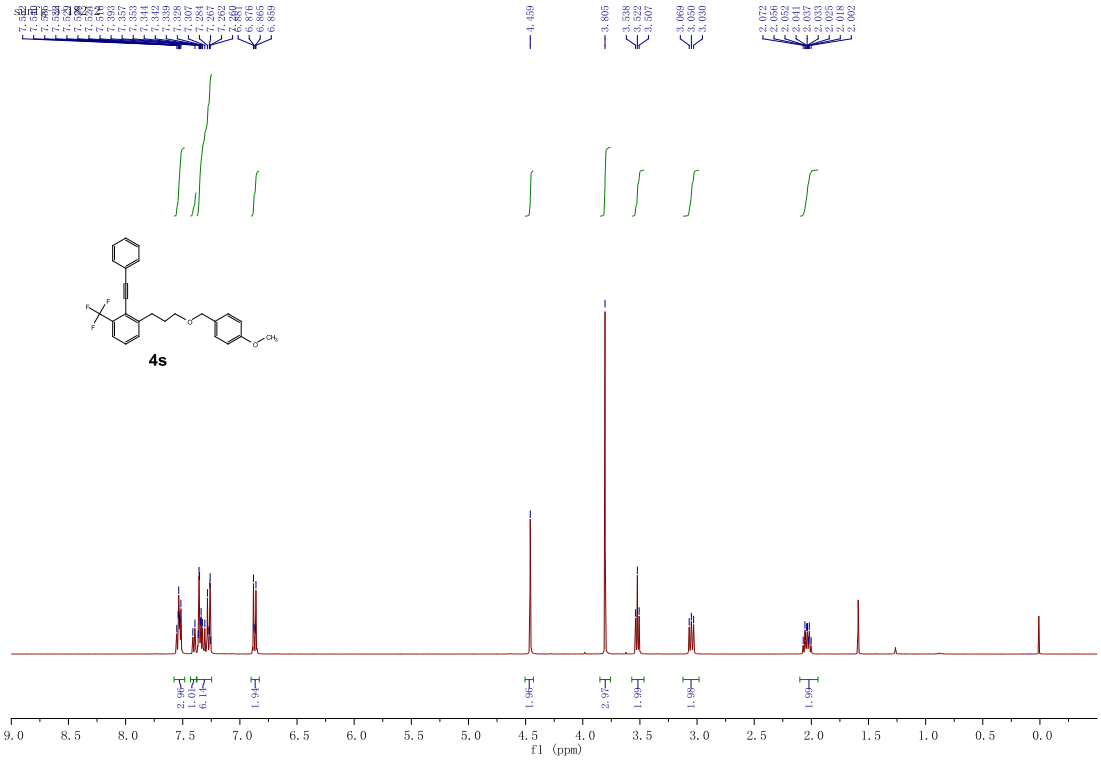

sunfg-5-164c
4p
f1 (ppm)

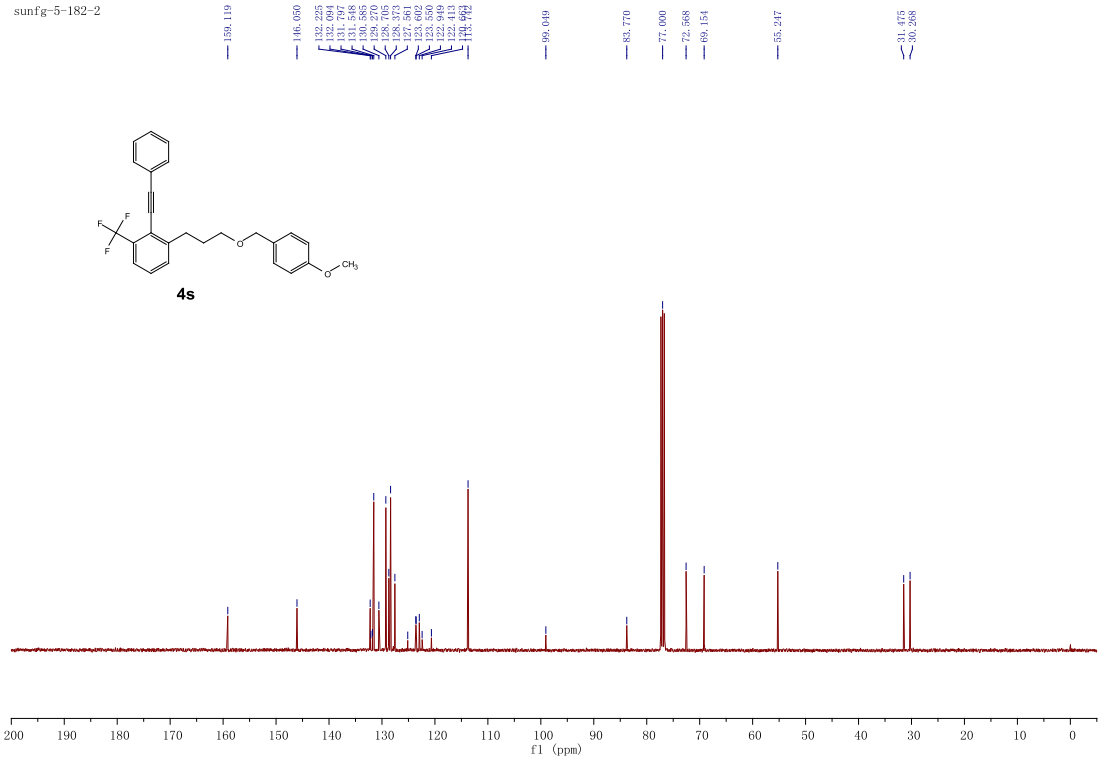

sunfg-6-71
8.345
8.342
7.793
7.773
7.704
7.683
7.545
7.542
7.528
7.525
7.522
7.507
7.504
7.453
7.433
7.431
7.416
7.413
7.341
7.320
3.002
2.983
2.963
2.647
2.630
2.613
1.736
1.717
1.712
1.698
1.679
1.621
1.602
1.582
1.553
1.457
1.438
1.419
1.006
0.987
0.969
0.951
4q
0.98
1.02
1.04
1.03
1.02
0.97
1.98
1.99
4.12
2.94
2.16
6.13
f1 (ppm)

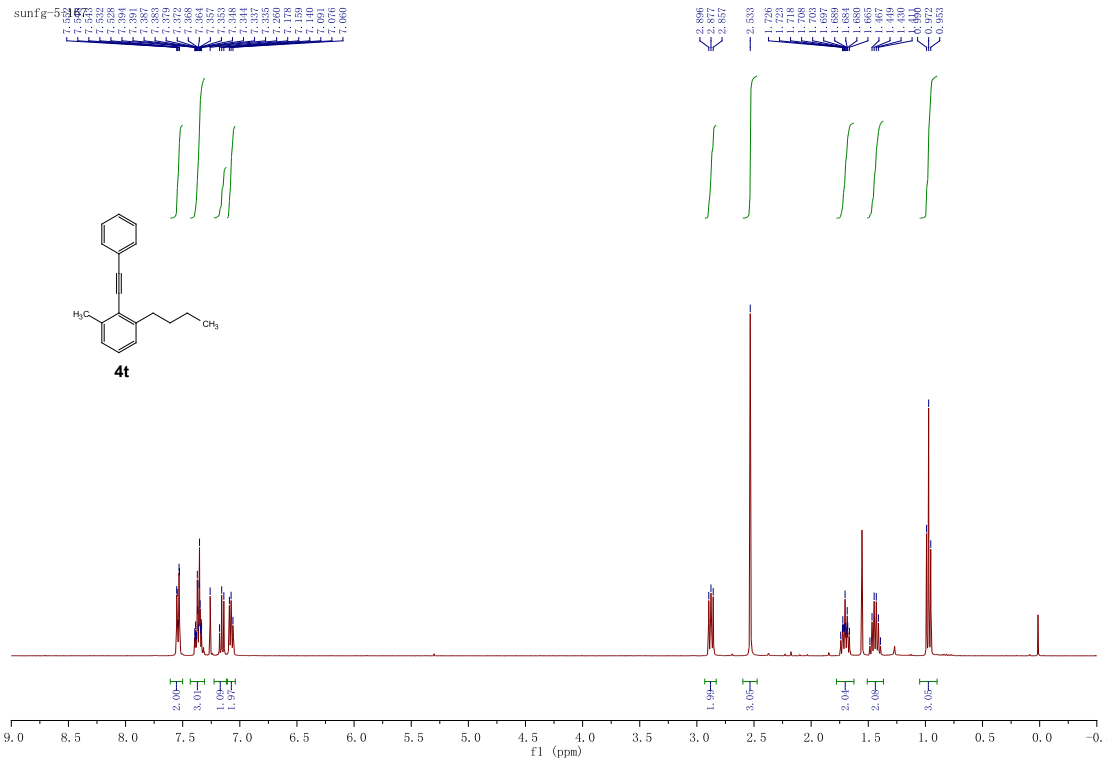

sunfg-6-71c
143.445
133.838
131.604
127.886
127.272
126.360
126.176
125.224
119.727
99.265
77.000
34.884
33.493
31.116
22.659
22.097
19.552
14.008
13.657
4q
f1 (ppm)

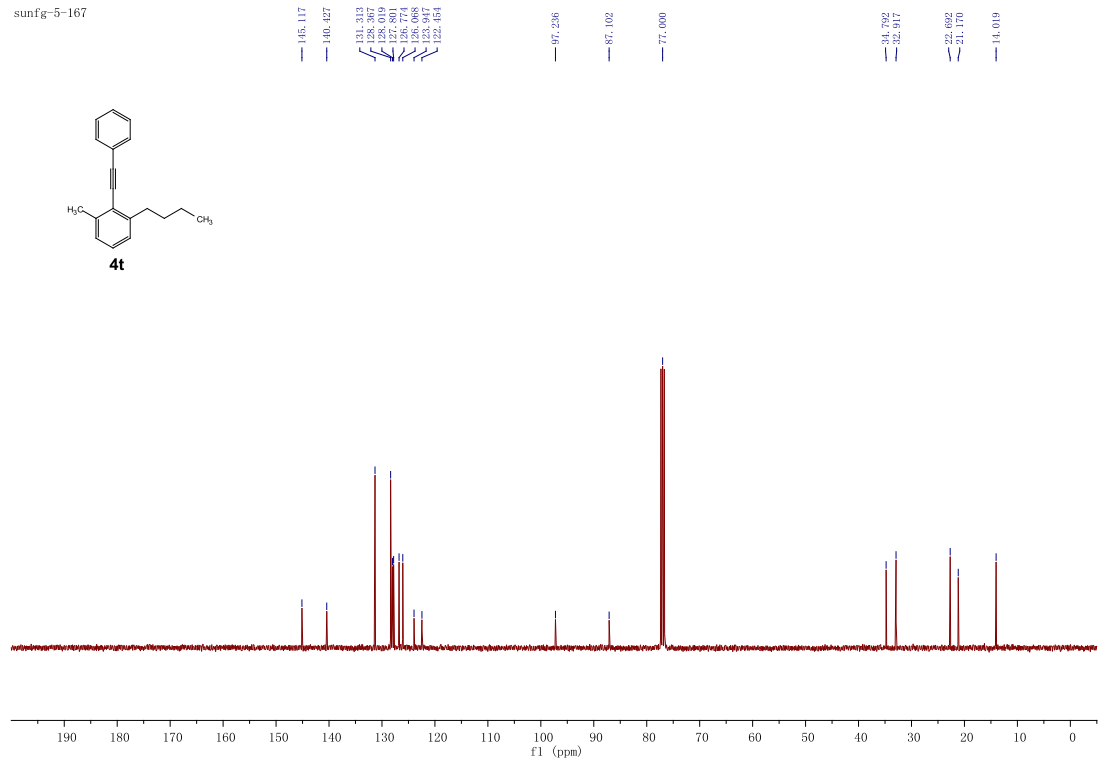

sunfg-6-72
8.358
8.358
7.799
7.779
7.711
7.690
7.551
7.547
7.534
7.530
7.526
7.513
7.469
7.460
7.457
7.440
7.437
7.423
7.420
7.349
7.327
3.017
2.998
2.978
2.649
2.633
2.614
1.735
1.732
1.717
1.712
1.693
1.544
1.448
1.429
1.411
1.397
1.389
1.379
1.370
0.979
0.961
0.953
0.944
0.936
0.930
0.926
CH3
CH3
4r
1.00
1.09
1.09
1.05
1.15
1.06
2.06
2.12
4.32
2.15
6.60
6.63
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

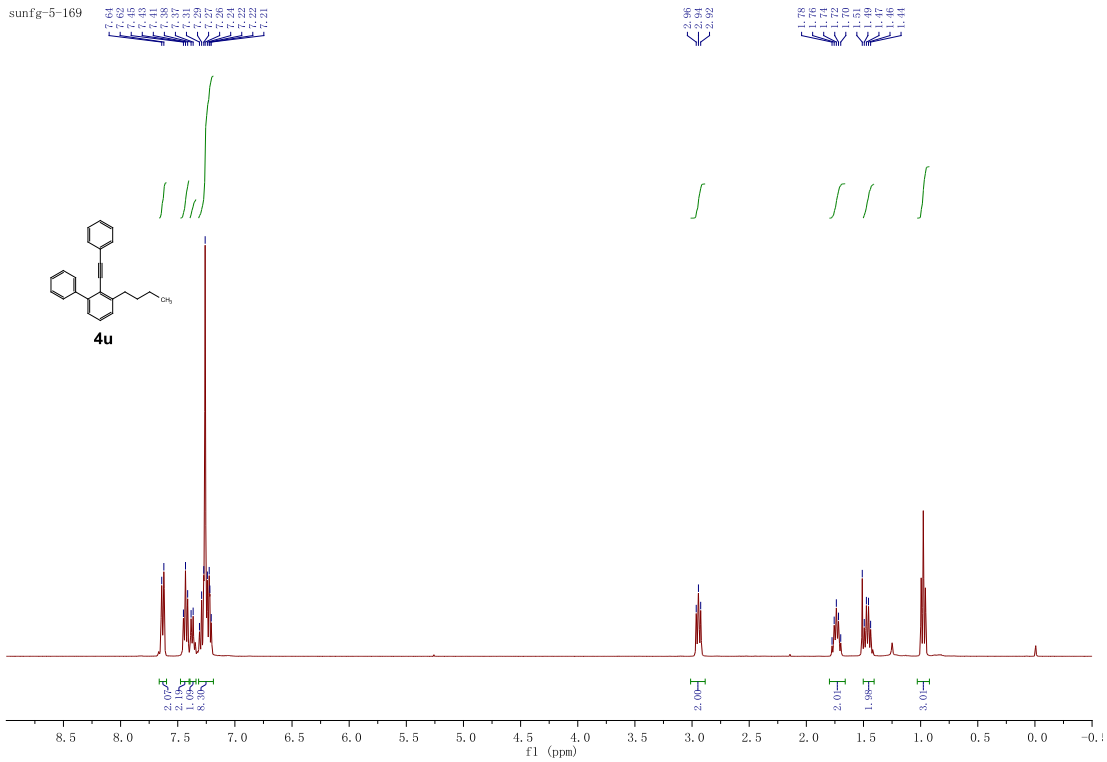

sunfg-6-72
143.420
133.881
131.641
127.888
127.264
126.345
126.215
125.226
119.781
99.348
34.963
33.014
31.439
29.035
28.719
22.645
22.619
19.879
14.068
14.004
CH3
CH3
4r
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

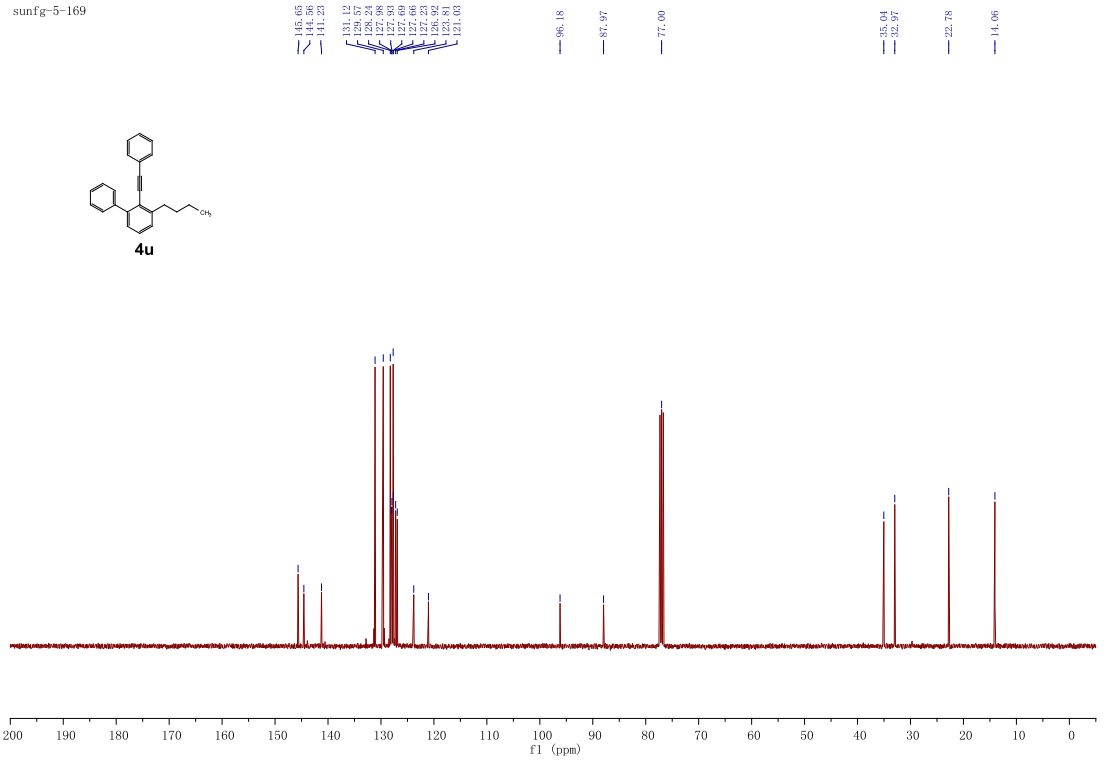

4s
f1 (ppm)

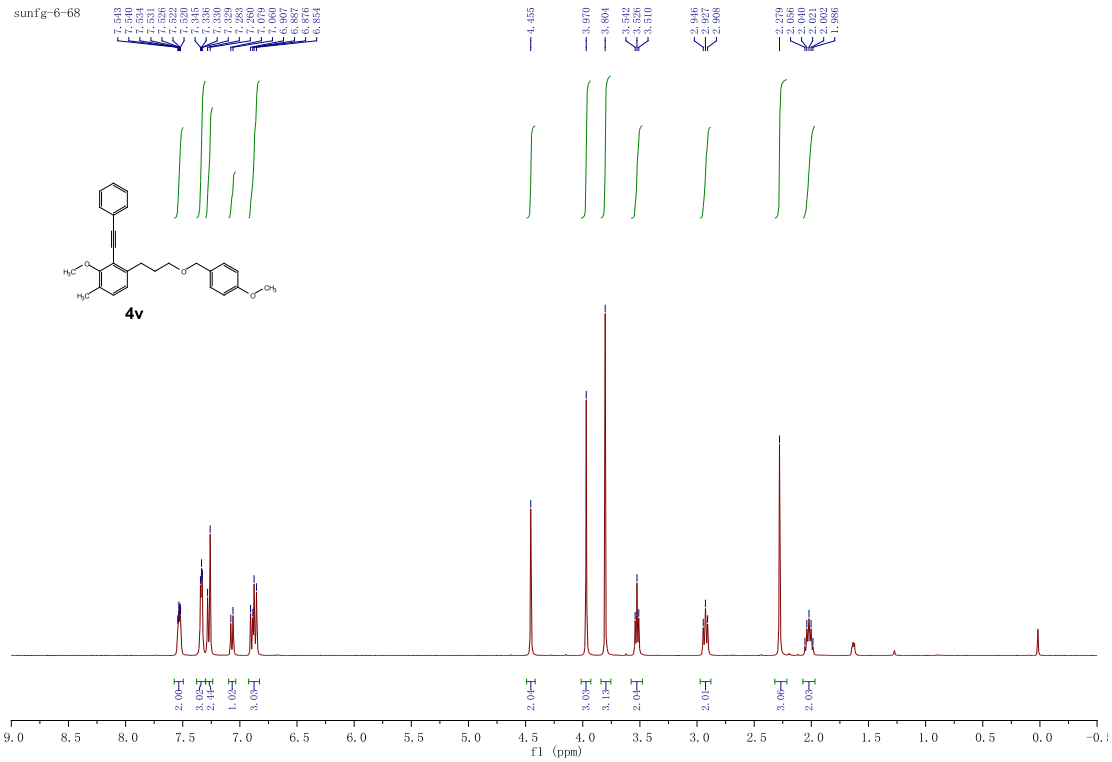

sunfg-5-182-2
4s
f1 (ppm)

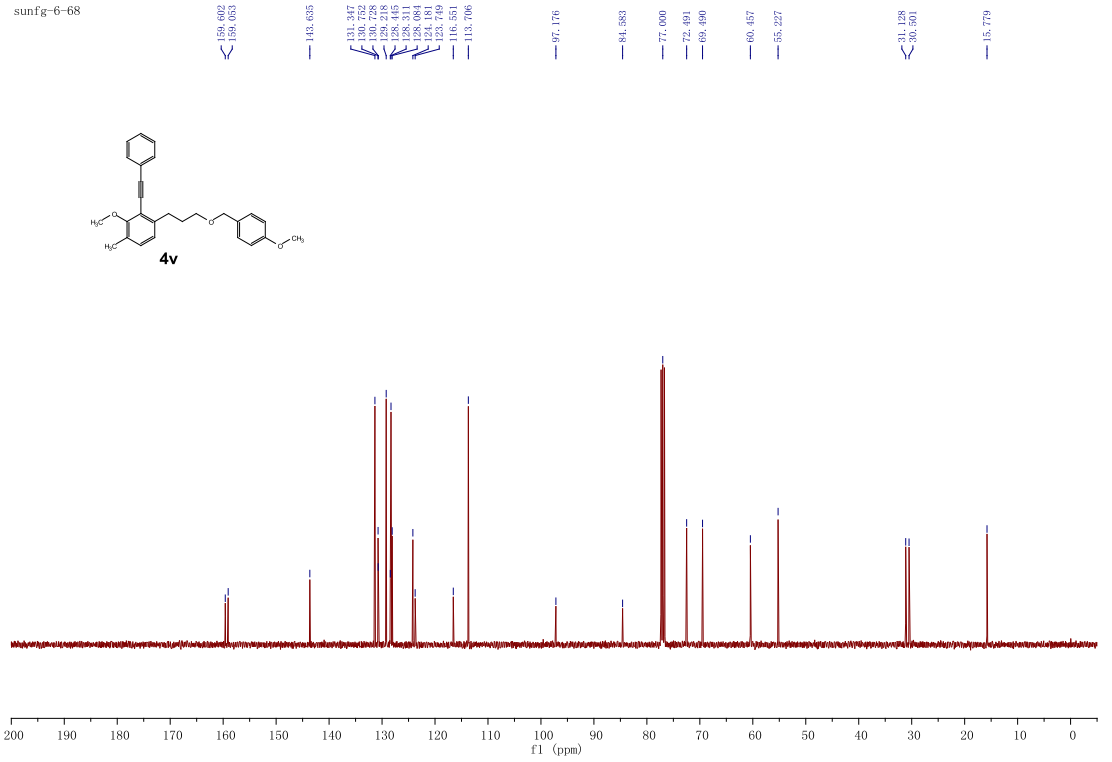

sunfg-5-167
7.592
7.588
7.573
7.532
7.528
7.394
7.391
7.387
7.383
7.379
7.372
7.368
7.364
7.357
7.353
7.348
7.344
7.337
7.335
7.180
7.178
7.159
7.140
7.091
7.076
7.060
3.896
3.877
3.857
2.533
1.726
1.723
1.718
1.708
1.703
1.697
1.689
1.684
1.680
1.465
1.446
1.449
1.430
0.994
0.990
0.972
0.953
H3C
CH3
4t
2.00
3.01
1.09
1.97
1.99
3.05
2.04
2.08
3.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

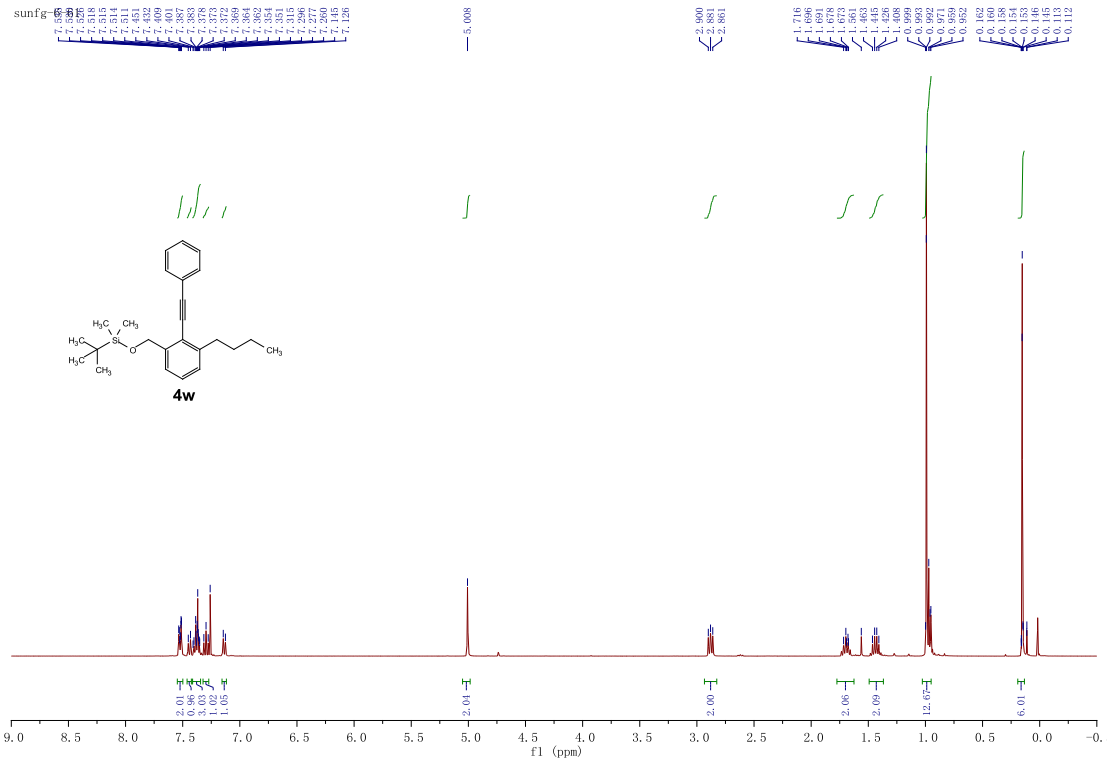

sunfg-5-167
145.117
140.427
131.313
128.367
128.019
127.801
126.774
126.068
123.947
122.454
97.236
87.102
77.000
34.792
32.947
22.692
21.170
14.019
H3C
CH3
4t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

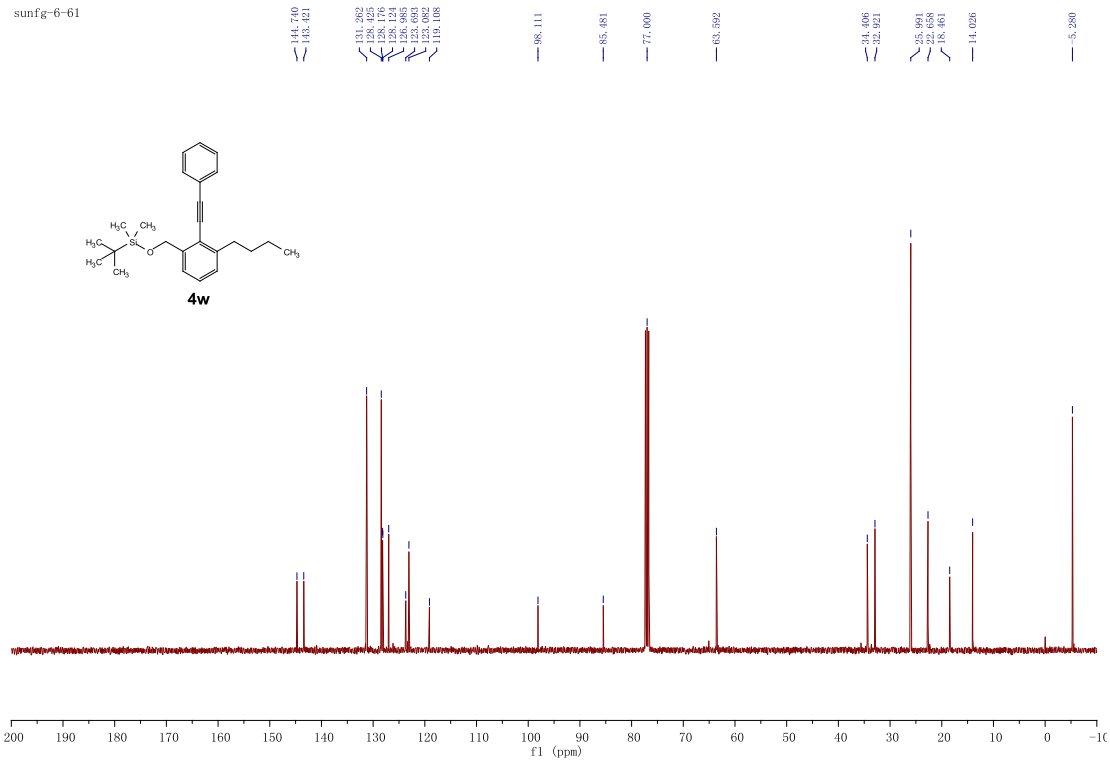

sunfg-5-169
7.64
7.62
7.45
7.43
7.41
7.38
7.37
7.31
7.29
7.27
7.26
7.24
7.22
7.21
2.96
2.94
2.92
1.78
1.76
1.74
1.72
1.70
1.51
1.49
1.47
1.46
1.44
4u
2.07
2.14
1.66
8.30
2.00
2.01
1.98
3.01
f1 (ppm)

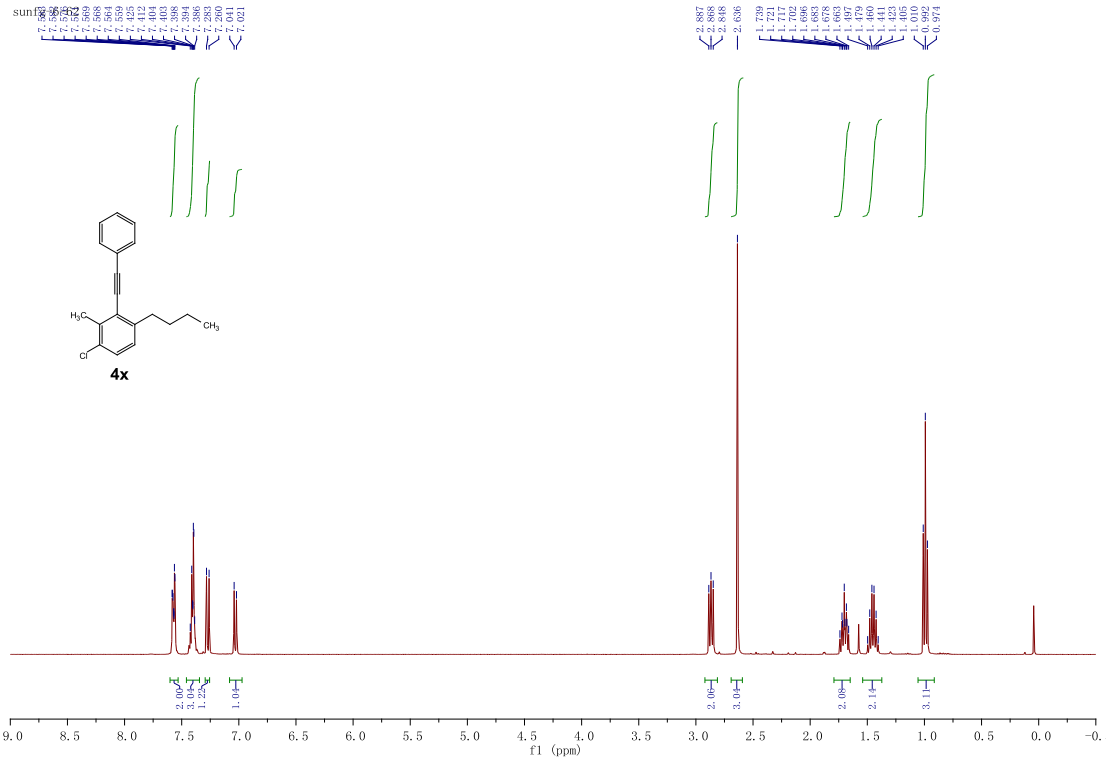

sunfg-5-169
145.65
144.56
141.23
131.12
129.57
128.24
127.98
127.63
127.60
127.66
127.23
126.92
121.81
121.03
96.18
87.97
77.00
35.04
32.97
22.78
14.06
4u
f1 (ppm)

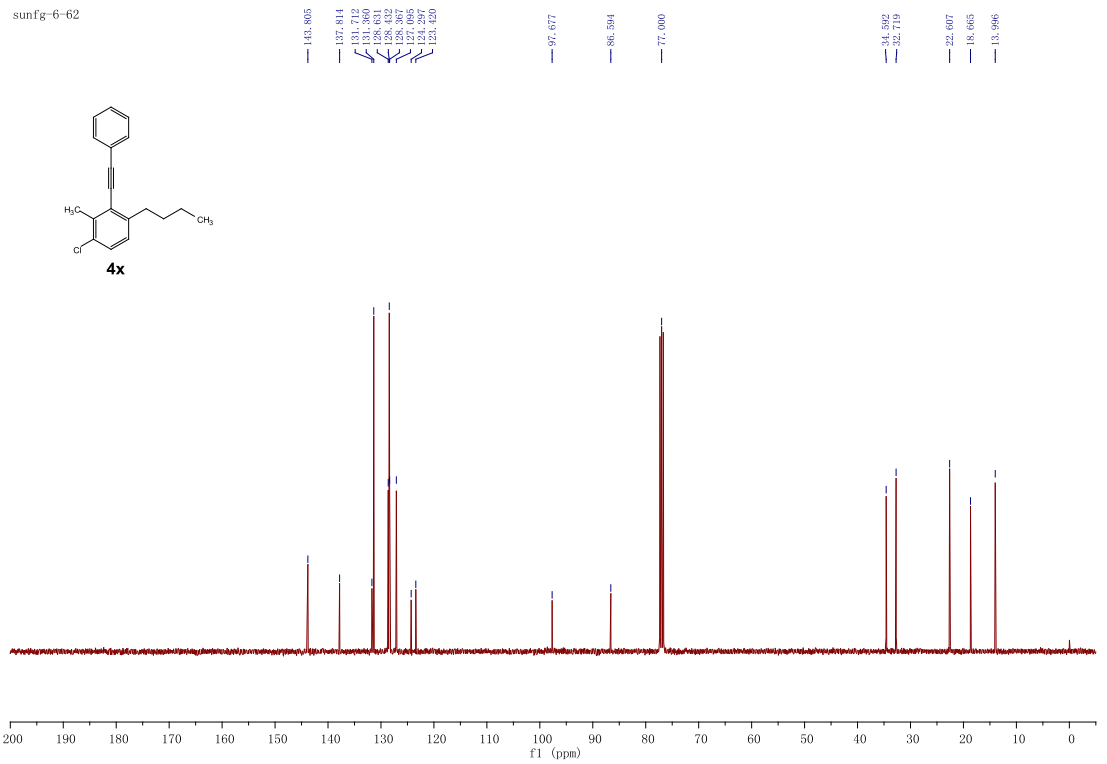

sunfg-6-68
7.543
7.540
7.534
7.531
7.526
7.522
7.520
7.345
7.336
7.330
7.329
7.283
7.269
7.079
7.060
6.907
6.887
6.870
6.854
4.455
3.970
3.804
3.542
3.526
3.510
2.946
2.927
2.908
2.279
2.056
2.040
2.021
2.002
1.986
4v
2.06
3.02
2.44
1.02
3.03
2.04
3.03
3.13
2.04
2.01
3.06
2.03
f1 (ppm)

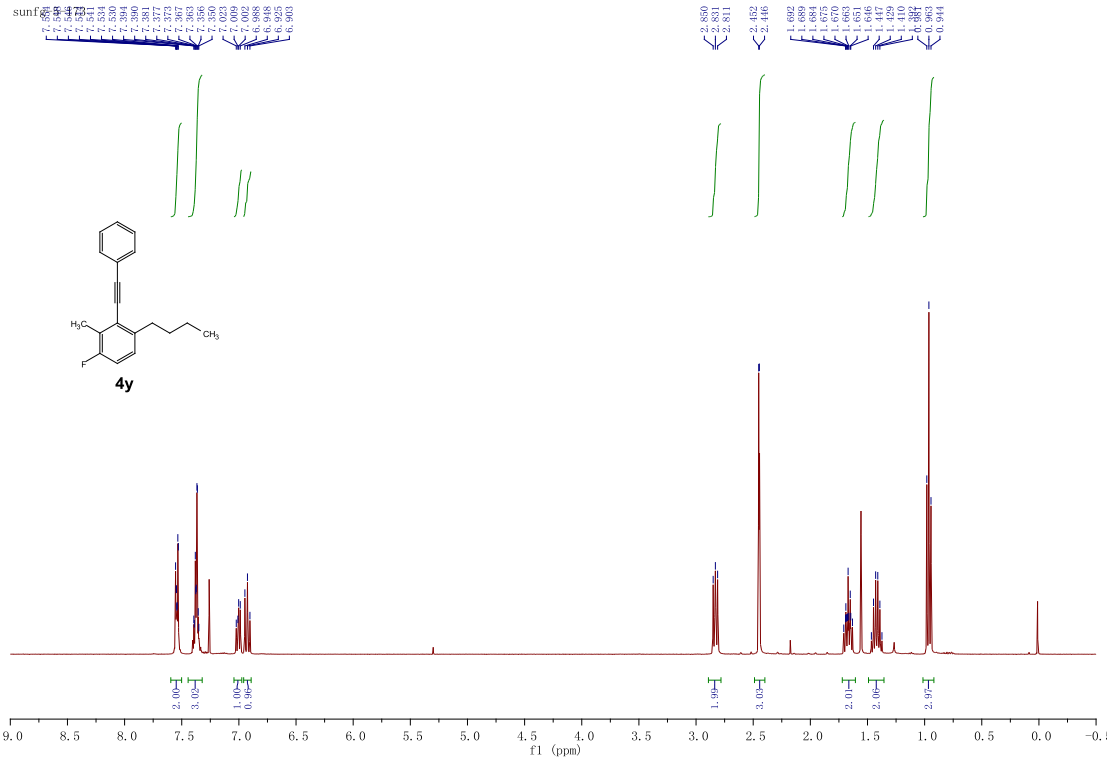

sunfg-6-68
159.602
159.053
143.635
131.347
130.752
130.728
129.218
128.445
128.311
128.084
124.181
123.749
116.551
113.706
97.176
84.883
77.000
72.491
69.490
60.457
55.227
31.128
30.501
15.779
4v
f1 (ppm)

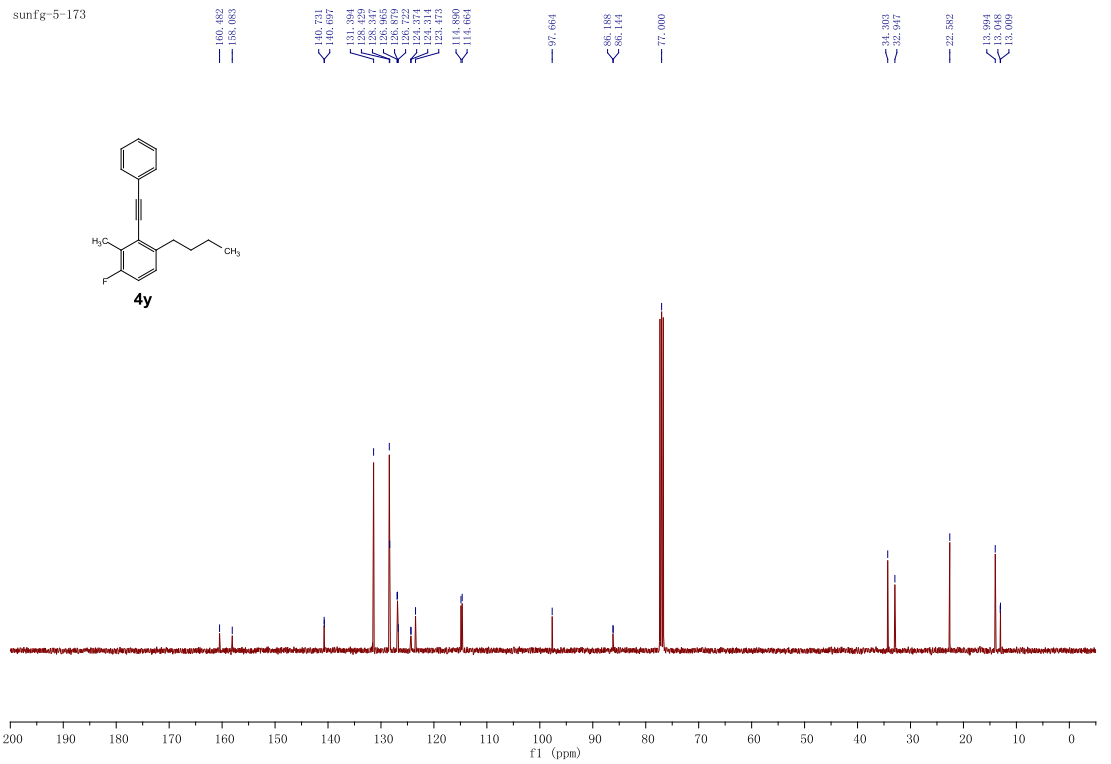

sunfg-6-61
5.008
2.900
2.883
2.861
1.716
1.696
1.691
1.678
1.673
1.581
1.463
1.445
1.426
1.408
1.069
0.993
0.992
0.971
0.959
0.952
0.162
0.160
0.158
0.154
0.153
0.146
0.145
0.113
0.112
2.01
0.96
3.03
1.02
1.05
2.04
2.00
2.06
2.09
12.67
6.01
f1 (ppm)
4w

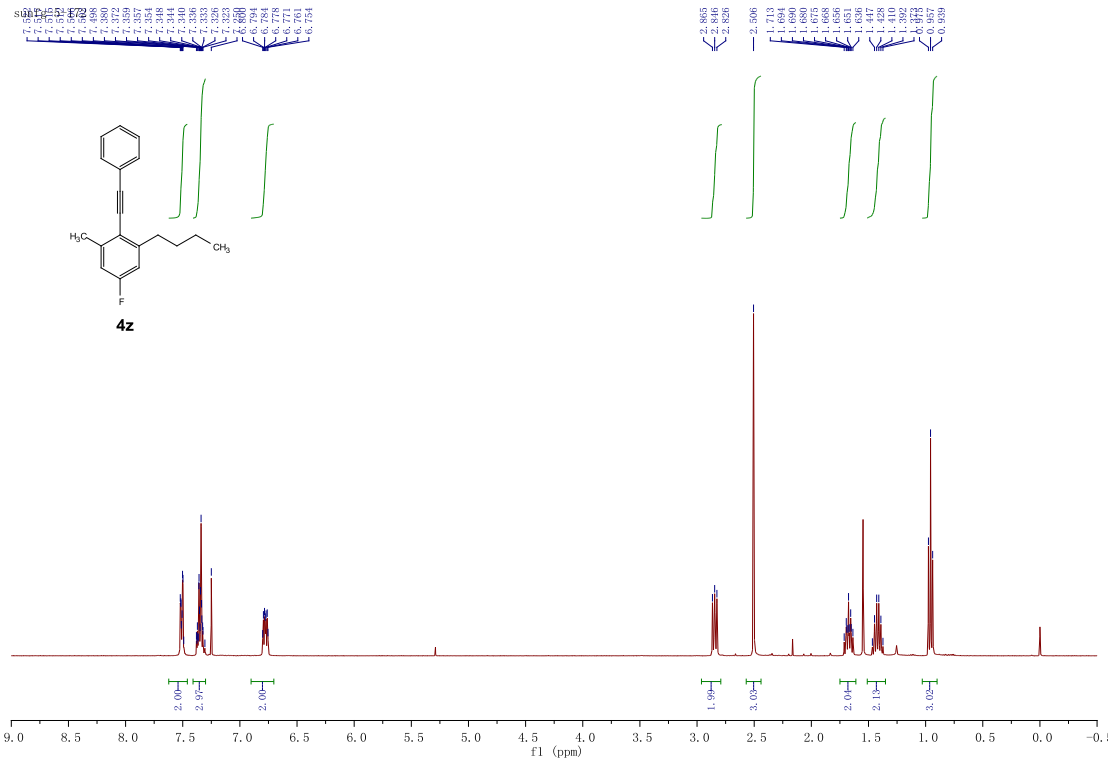

sunfg-6-61
144.740
143.421
131.262
128.425
128.176
128.124
126.985
123.693
123.082
119.108
98.111
85.481
77.000
63.592
34.406
32.921
25.991
22.658
18.461
14.026
5.280
f1 (ppm)
4w

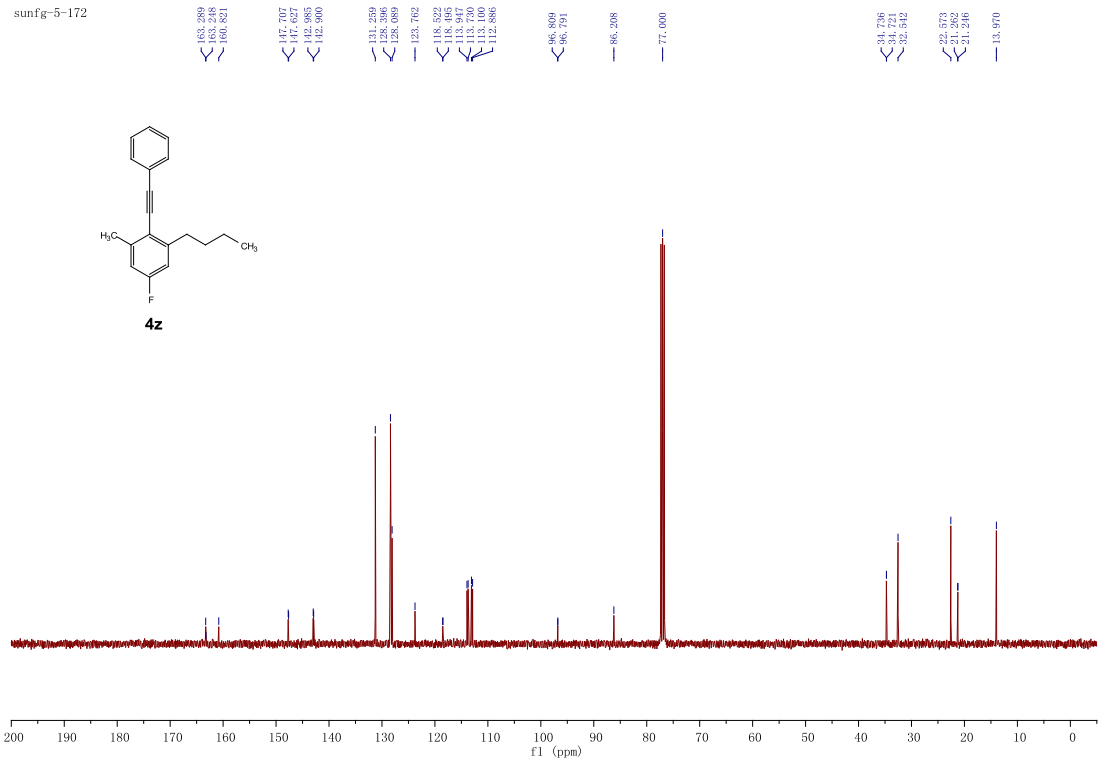

sunfg-6-62
4x
f1 (ppm)

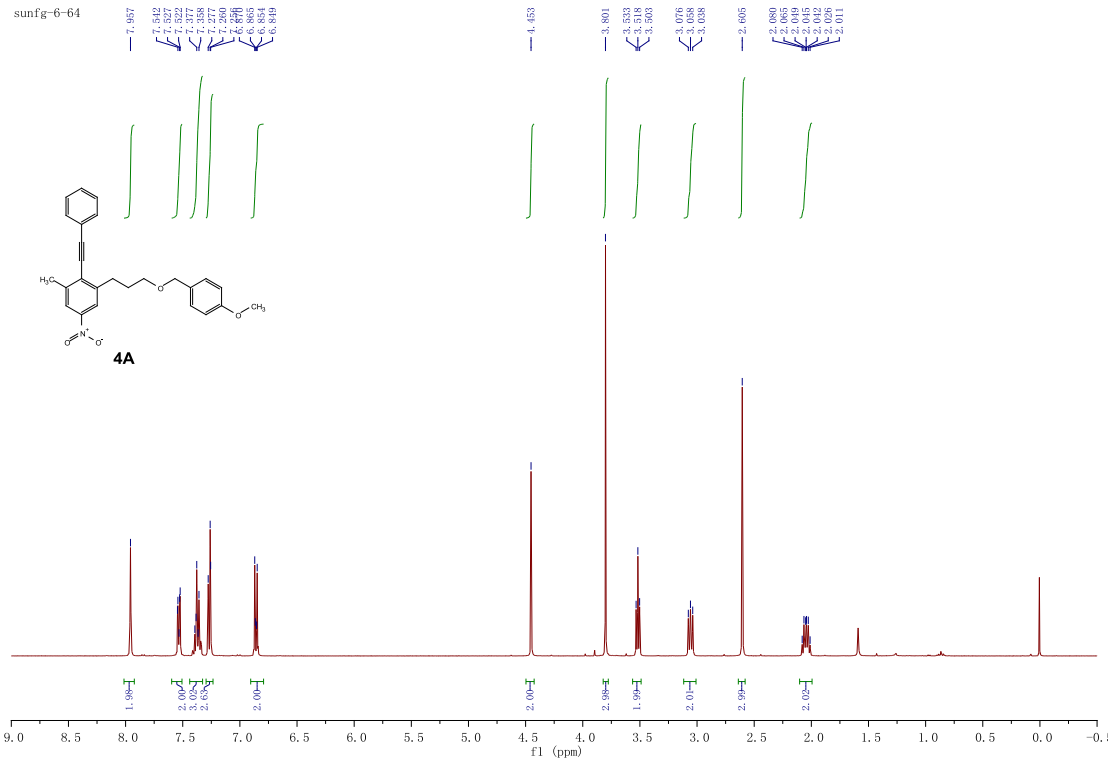

sunfg-6-62
4x
f1 (ppm)

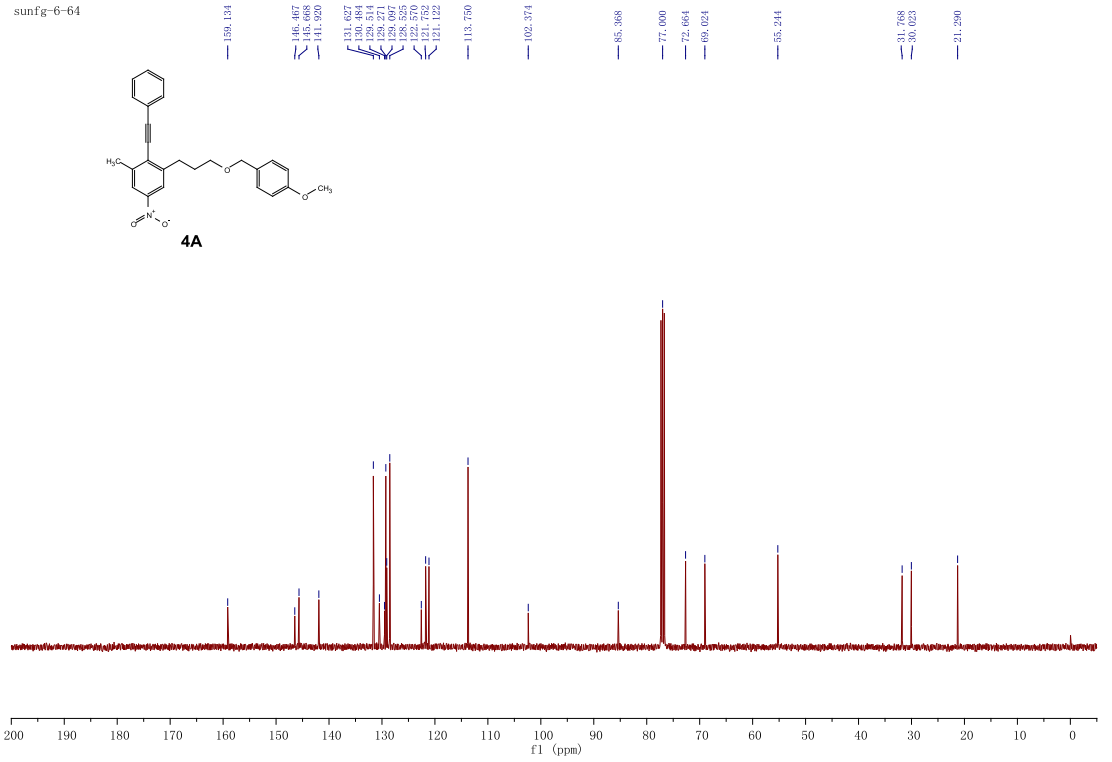

4y
f1 (ppm)

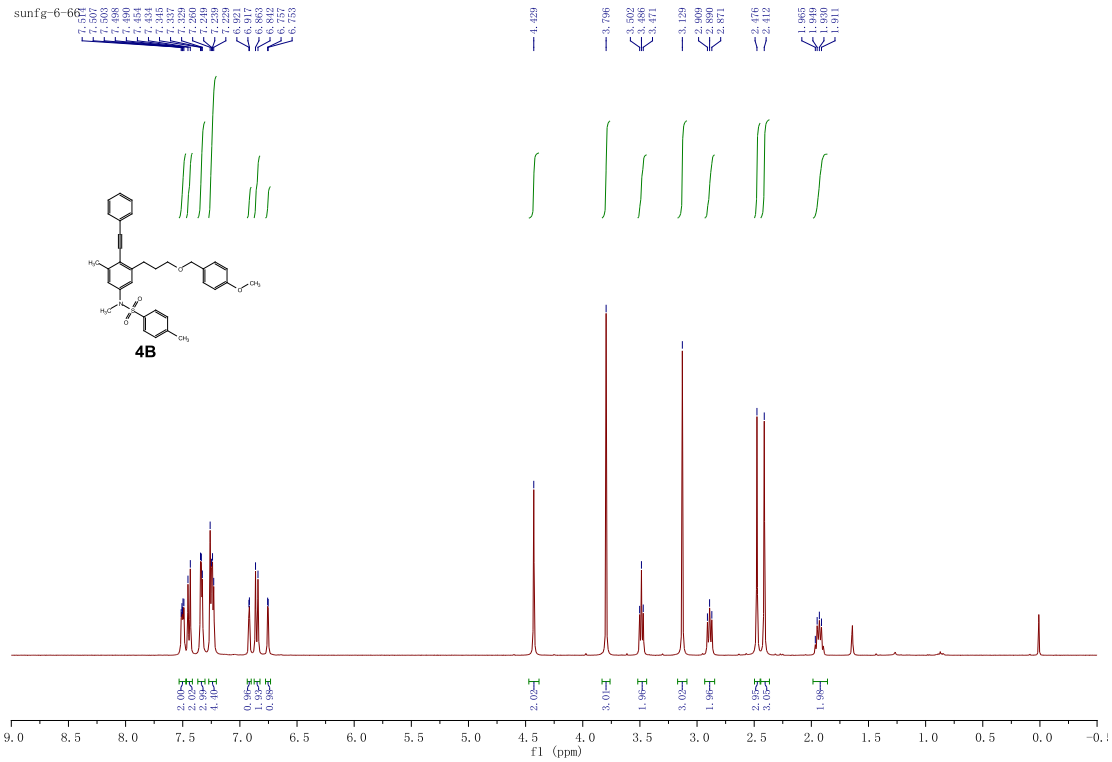

sunfg-5-173
4y
f1 (ppm)

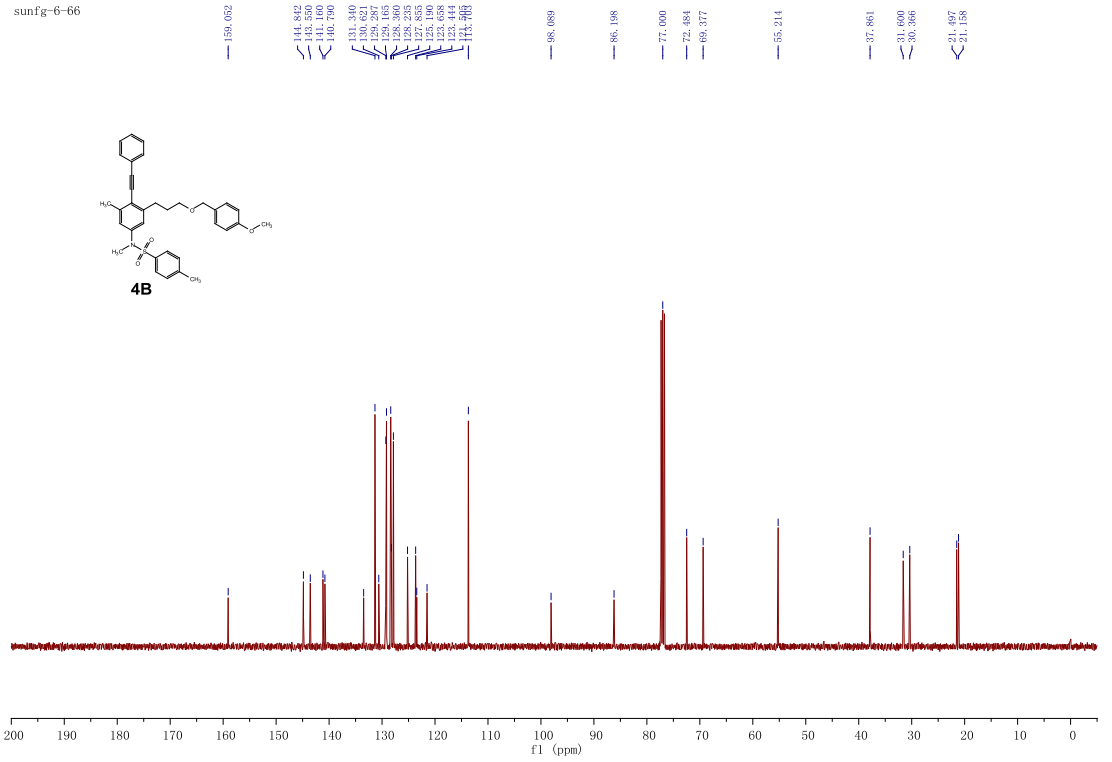

sunfg-5-172
H3C
CH3
F
4z
f1 (ppm)

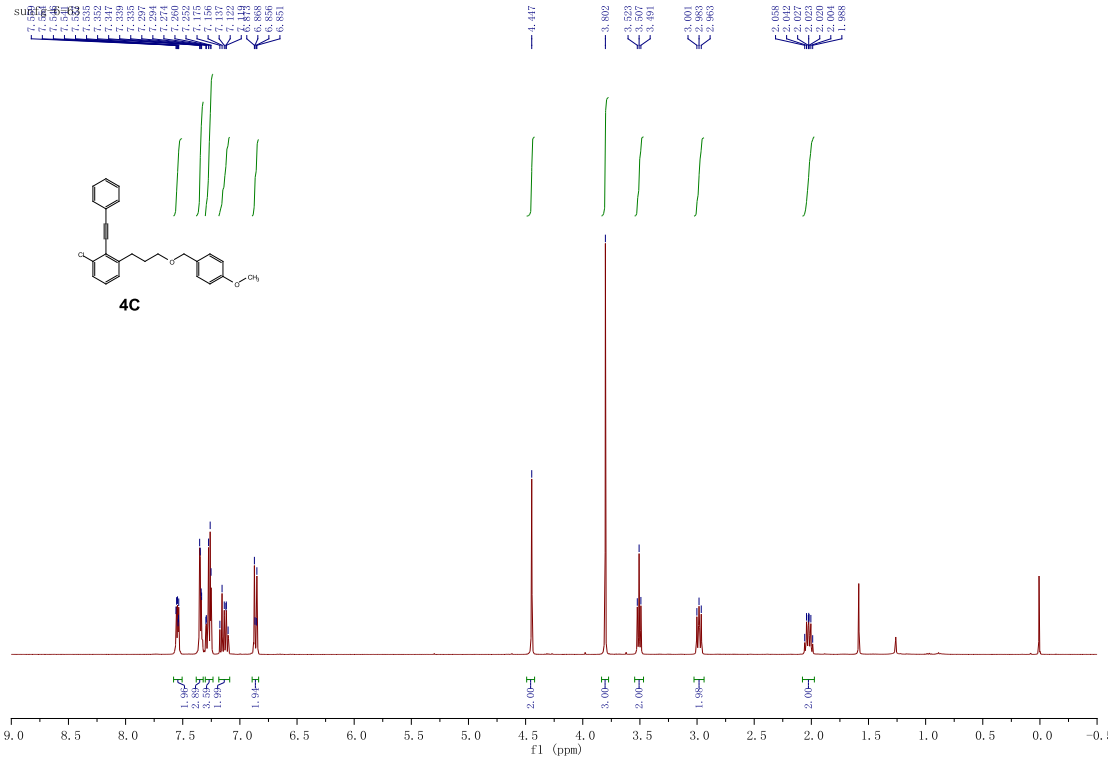

sunfg-5-172
H3C
CH3
F
4z
f1 (ppm)

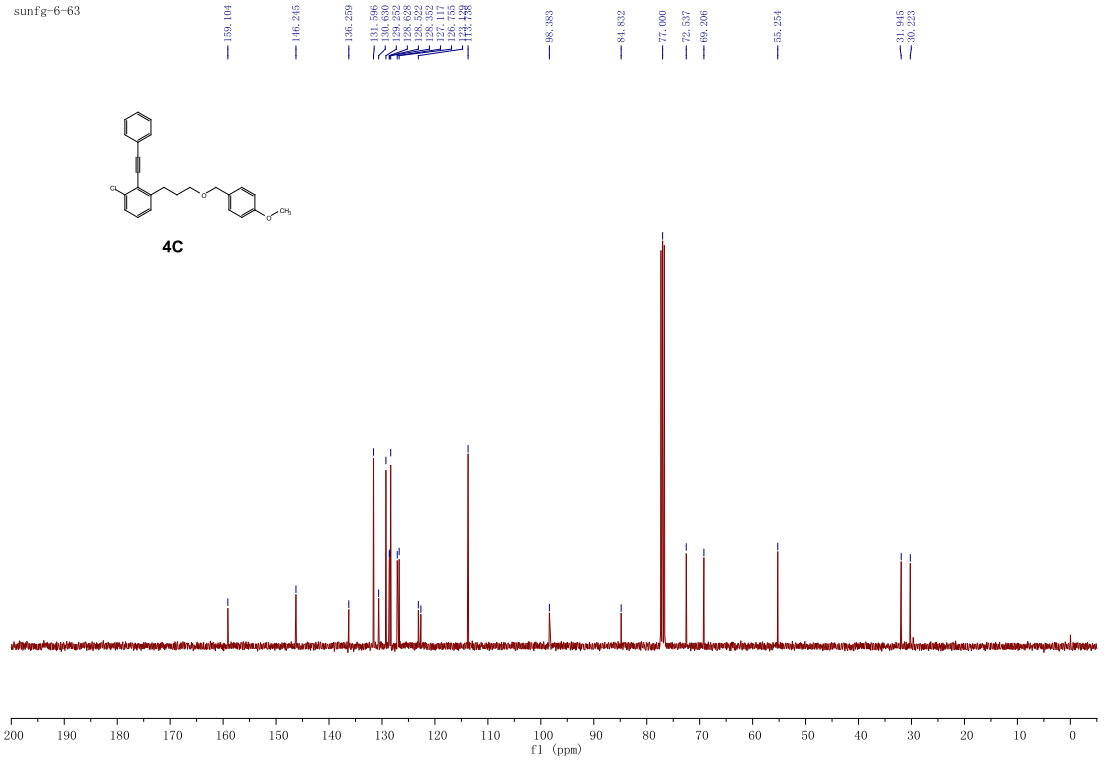

sunfg-6-64
7.957
7.542
7.527
7.522
7.377
7.358
7.277
7.260
6.870
6.865
6.854
6.849
4.453
3.801
3.533
3.518
3.503
3.076
3.058
3.038
2.605
2.080
2.065
2.049
2.045
2.042
2.026
2.011
4A
1.98
2.00
1.05
2.63
2.06
2.04
2.98
1.99
2.01
2.99
2.02
f1 (ppm)

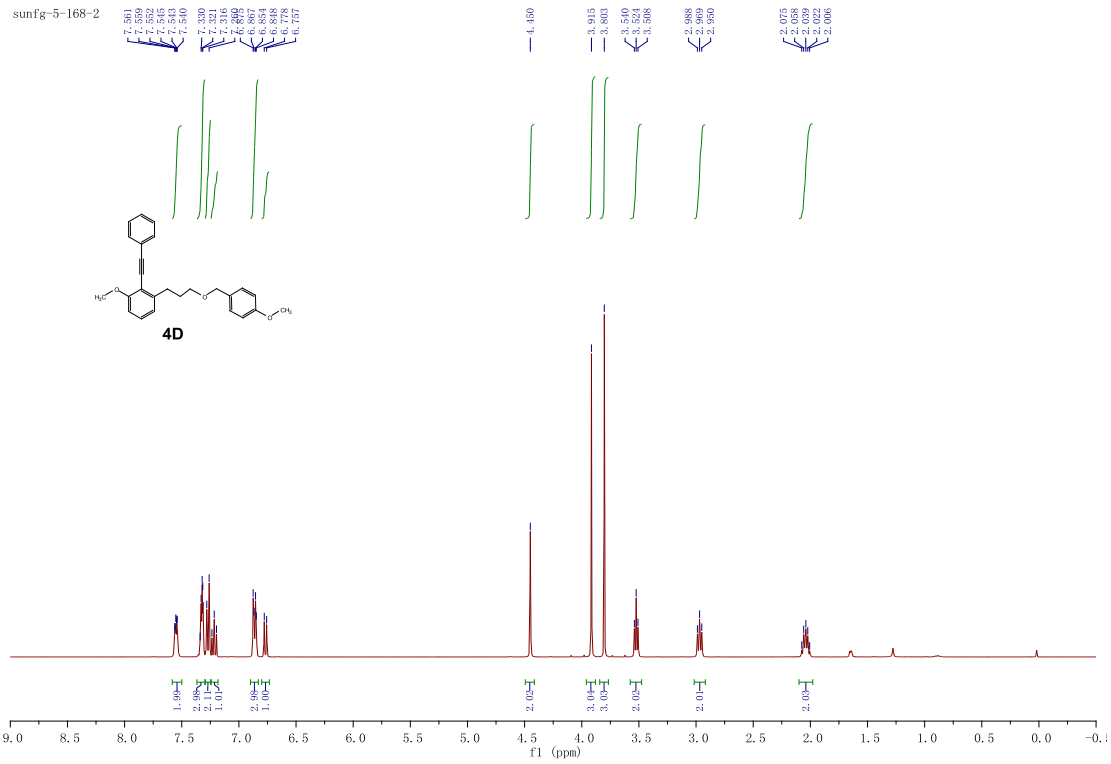

sunfg-6-64
159.134
146.467
145.668
141.920
131.627
130.484
129.514
129.271
128.097
126.525
122.570
121.752
121.122
113.750
102.374
85.368
77.000
72.664
69.024
55.244
31.768
30.023
21.290
4A
f1 (ppm)

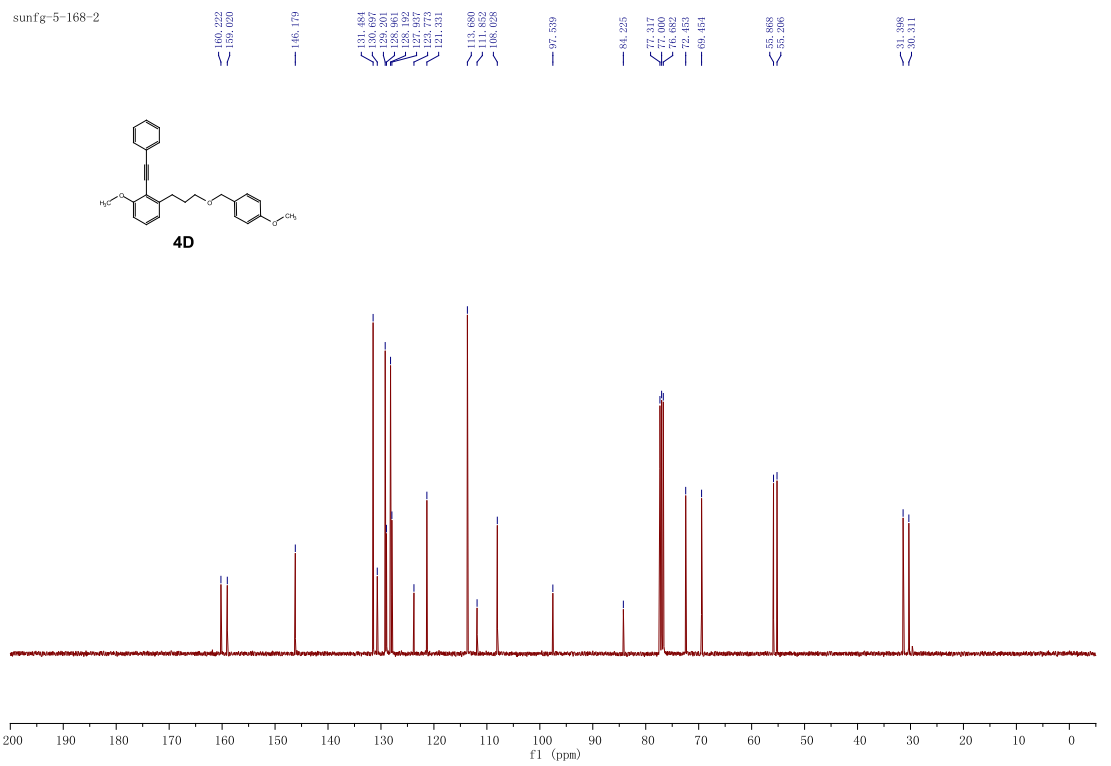

sunfg-6-66
4B
f1 (ppm)


sunfg-6-66
4B
f1 (ppm)


4C
f1 (ppm)


sunfg-6-63
4C
f1 (ppm)


sunfg-5-168-2
7.561
7.559
7.552
7.545
7.543
7.540
7.330
7.321
7.316
7.309
6.875
6.867
6.854
6.848
6.778
6.757
4.450
3.915
3.803
3.540
3.524
3.508
2.988
2.969
2.950
2.075
2.058
2.039
2.022
2.006
4D
1.99
2.98
2.13
1.01
2.98
1.00
2.02
3.04
3.03
2.02
2.01
2.03
f1 (ppm)


sunfg-5-168-2
160.222
159.020
146.179
131.484
130.697
129.201
128.961
128.192
127.943
127.847
123.773
121.331
113.680
111.852
108.028
97.539
84.225
77.317
77.000
76.682
72.453
69.454
55.808
55.306
31.398
30.311
4D
f1 (ppm)