Electronic Supplementary Material (ESI) for Catalysis Science & Technology

Pd/Tetraphosphine Catalytic System for Cu-Free Sonogashira Reaction "on Water"

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Supporting Information

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Materials and Methods

All chemicals were purchased from commercial suppliers. Except for some liquid reagents being sensitive to light and moisture (DMA, toluene, methanol and alcohol) was redistilled prior to use, there is no further treatment. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded on a Bruker AV II-400 MHz. Mass spectrosopy data of the products were collected with a MS-EI instrument. All products were isolated by short chromatography on a silica gel (300-400 mesh) using petroleum ether (60-90 ℃), unless otherwise noted. Compouds described in the literature were characterized by ^{1}H NMR spectroscopy and compared to the reported data.

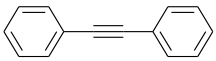
Synthesis of N,N,N',N'-tetra(diphenylphosphinomethy) pyridine-2,6-diamine **1**

A solution of pyridine-2,6-diamine (110 mg, 1 mmol) in 5mL alcohol and triethylamine of 8 mL was added slowly to a stirred suspension of $[Ph_2P(CH_2OH)_2]Cl$[17] (1.4 g, 5 mmol) in 15 mL toluene and 10 mL alcohol under nitrogen, then the mixture was refluxed for 40 h. At the end of the reaction, the mixture solution was washed with degassed water and organic layer was dried over $MgSO_4$. The organic layer was filtered and the solvent was removed under vacuum. The residue was recrystallized in 1.5 mL dichloromethane and 15 mL methanol. After the resulting crude product was refluxed in 10 mL methanol for 1 h and slowly cooled to room temperature, the light yellow solid product was obtained. Yield: 0.76 g (84%). Compared with the literature,[18] we used a new and easy method to synthesize this ligand, and got a higher productivity. The product was characterized by ^{1}H NMR and ^{31}P NMR, and the results were in accordance with the reported results.[18] ^{1}H NMR (400 MHz, DMSO, 25 ℃): δ = 7.15-7.35 (m, 41H, Ph, Py^{4}), 5.88 (d, $^{3}J_{H,H}$ = 8.1 Hz, 2H, Py^{3,5}), 4.14 (d, $^{2}J_{H,H}$ = 3.4 Hz, 8H, NCH_2P) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ = 155.49 (s, Py^{2,6}), 137.93 (S, Py^{4}), 137.40 (d, *J* = 15.5 Hz , Ar^{1}), 132.89 (d, *J* = 19.3 Hz, Ar^{2,6}), 128.38 – 127.78 (m, Ar^{3,4,5}), 95.08 (s, Py^{3,5}), 49.75 (s, NCH_2P). ^{31}P NMR (162 MHz, $CDCl_3$) δ = -24.62 ppm. MS (ESI) $[C_{58}H_{52}N_2P_4]$: m/z $(M+H)^+$: 902.

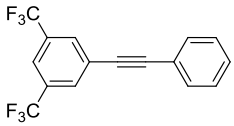
General Procedure for the Sonogashira Reaction of Aryl Halides with Terminal Alkynes

K_3PO_4 (212 mg, 1 mmol), aryl halides (0.5 mmol), terminal alkynes (0.6 mmol) and degassed H_2O (3 mL) were added successively into a dried Schlenk tube with a magnetic bar under nitrogen. Then DMA (N, N-dimethylacetamide 0.05 mL) solution of tetraphosphine 1 (0.0005 mmol) and $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (0.00025 mmol), which was reacted at 100 ℃ for 30 min prior to use, were added into the mixture. The reaction was performed at 100 ℃. At the end of reaction, the solution was cooled to room temperature and was extracted with ethyl acetate (3×3 mL). The organic layer was dried over $MgSO_4$, filtered and purified with silica gel chromatography (petroleum ether) to give a corresponding product.

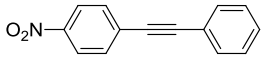
Characterization Data



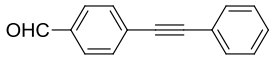
3[1]:[1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.58 – 7.47 (m, 4H, Ar-*H*), 7.35 (d, *J* = 5.5 Hz, 6H, Ar-*H*) ppm.



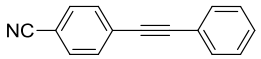
4[10]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.96 (s, 2H, Ar-*H*), 7.82 (s, 1H, Ar-*H*), 7.62 – 7.50 (m, 2H, Ar-*H*), 7.40 (d, *J* = 4.3 Hz, 3H, Ar-*H*) ppm.



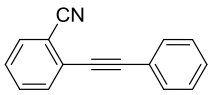
5[2]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 8.23 (d, *J* = 9.0 Hz, 2H, Ar-*H*), 7.67 (d, *J* = 9.0 Hz, 2H, Ar-*H*), 7.60 – 7.54 (m, 2H, Ar-*H*), 7.40 (d, *J* = 5.0 Hz, 3H, Ar-*H*) ppm.



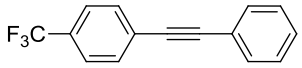
6[5]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 10.03 (s, 1H, C*H*O), 7.87 (d, *J* = 7.9 Hz, 2H, Ar-*H*), 7.68 (d, *J* = 7.6 Hz, 2H, Ar-*H*), 7.56 (dd, *J* = 5.4, 1.9 Hz, 2H, Ar-*H*), 7.43 – 7.34 (m, 3H, Ar-*H*) ppm.



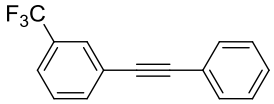
7[2]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.63 (m, 4H, Ar-*H*), 7.55 (m, 2H, Ar-*H*), 7.44 – 7.36 (m, 3H, Ar-*H*) ppm.



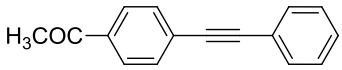
8[7]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.68 (d, *J* = 7.8 Hz, 1H, Ar-*H*), 7.63 (t, *J* = 6.2 Hz, 3H, Ar-*H*), 7.57 (t, *J* = 7.7 Hz, 1H, Ar-*H*), 7.40 (dd, *J* = 15.9, 6.3 Hz, 4H, Ar-*H*) ppm.



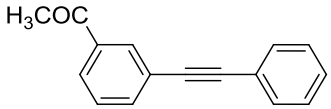
9[1]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.73 – 7.57 (m, 4H, Ar-*H*), 7.57 – 7.51 (m, 2H, Ar-*H*), 7.44 – 7.33 (m, 3H, Ar-*H*).



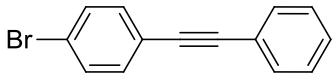
10[11]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.80 (s, 1H, Ar-*H*), 7.69 (d, *J* = 7.7 Hz, 1H, Ar-*H*), 7.63 – 7.51 (m, 3H, Ar-*H*), 7.47 (t, *J* = 7.8 Hz, 1H, Ar-*H*), 7.37 (d, *J* = 2.6 Hz, 3H, Ar-*H*) ppm.



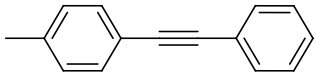
11[2]: [1]H NMR (400 MHz, $CDCl_3$, TMS): δ 7.92-7.95 (m, 2H, Ar-*H*), 7.59-7.63 (m, 2H, Ar-*H*), 7.54-7.57 (m, 2H, Ar-*H*), 7.36-7.38 (m, 3H, Ar-*H*), 2.61 (s, 3H, CH_3) ppm.



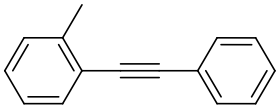
12[5]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.12 (s, 1H, Ar-*H*), 7.92 (d, *J* = 7.8 Hz, 1H, Ar-*H*), 7.79 (d, *J* = 8.2 Hz, 1H, Ar-*H*), 7.72 (d, *J* = 7.6 Hz, 1H, Ar-*H*), 7.55 (dd, *J* = 6.4, 2.8 Hz, 2H, Ar-*H*), 7.46 (t, *J* = 7.8 Hz, 1H, Ar-*H*), 7.36 (dd, *J* = 10.0, 7.4 Hz, 2H, Ar-*H*), 2.63 (s, 3H, C*H*$_3$) ppm.



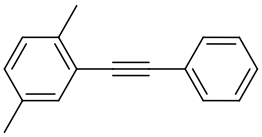
13[1]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.55 – 7.46 (m, 4H, Ar-*H*), 7.43 – 7.33 (m, 5H, Ar-*H*) ppm.



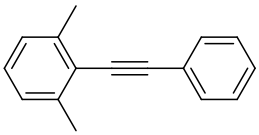
14[1]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.52 (d, *J* = 7.3 Hz, 2H, Ar-*H*), 7.43 (d, *J* = 7.6 Hz, 2H, Ar-*H*), 7.37 – 7.28 (m, 3H, Ar-*H*), 7.16 (d, *J* = 7.7 Hz, 2H, Ar-*H*), 2.37 (s, 3H, C*H*$_3$) ppm.



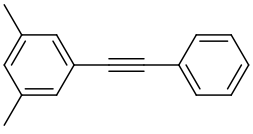
15[3]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.54 (d, *J* = 7.3 Hz, 2H, Ar-*H*), 7.50 (d, *J* = 7.6 Hz, 1H, Ar-*H*), 7.39 – 7.30 (m, 3H, Ar-*H*), 7.26 – 7.21 (m, 2H, Ar-*H*), 7.17 (dd, *J* = 6.9, 4.2 Hz, 1H, Ar-*H*), 2.52 (s, 3H, C*H*$_3$) ppm.



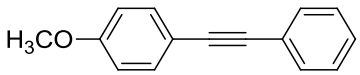
16[12]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.53 (d, *J* = 7.6 Hz, 2H, Ar-*H*), 7.40 – 7.28 (m, 4H, Ar-*H*), 7.12 (d, *J* = 7.7 Hz, 1H, Ar-*H*), 7.05 (d, *J* = 7.8 Hz, 1H, Ar-*H*), 2.47 (s, 3H, C*H*$_3$), 2.31 (s, 3H, C*H*$_3$) ppm.



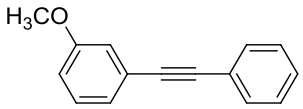
17[3]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.56 (d, *J* = 7.0 Hz, 2H, Ar-*H*), 7.40 – 7.32 (m, 3H, Ar-*H*), 7.17 – 7.11 (m, 1H, Ar-*H*), 7.08 (d, *J* = 7.4 Hz, 2H, Ar-*H*), 2.53 (s, 6H, C*H*$_3$) ppm.



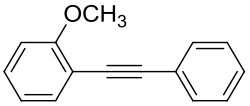
18[4]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.52 (d, *J* = 7.5 Hz, 2H, Ar-*H*), 7.33 (d, *J* = 6.1 Hz, 3H, Ar-*H*), 7.17 (s, 2H, Ar-*H*), 6.97 (s, 1H, Ar-*H*), 2.31 (s, 6H, C*H*$_3$) ppm.



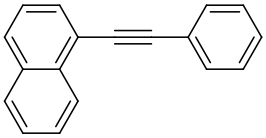
19[2]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.56 – 7.44 (m, 4H, Ar-*H*), 7.40 – 7.30 (m, 3H, Ar-*H*), 6.88 (d, *J* = 8.8 Hz, 2H, Ar-*H*), 3.80 (s, 3H, C*H*$_3$) ppm.



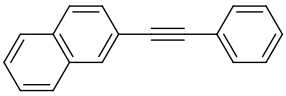
20[8]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.59 – 7.50 (m, 2H, Ar-*H*), 7.35 (d, *J* = 5.1 Hz, 3H, Ar-*H*), 7.29 – 7.22 (m, 2H, Ar-*H*), 7.13 (d, *J* = 7.6 Hz, 1H, Ar-*H*), 6.90 (d, *J* = 9.1 Hz, 1H, Ar-*H*), 3.83 (s, 3H, C*H*$_3$) ppm.



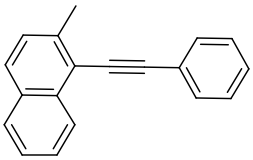
21[7]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.61 – 7.47 (m, 3H, Ar-*H*), 7.41 – 7.27 (m, 4H, Ar-*H*), 6.93 (m, 2H, Ar-*H*), 3.92 (s, 3H, C*H*$_3$) ppm.



22[1]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.47 (d, *J* = 7.9 Hz, 1H, Naphth-*H*), 7.87 (t, *J* = 8.0 Hz, 2H, Naphth-*H*), 7.79 (d, *J* = 8.0 Hz, 1H, Naphth-*H*), 7.68 (d, *J* = 6.3 Hz, 2H, Naphth-*H*), 7.65 – 7.58 (t, 1H, Naphth-*H*), 7.58 – 7.53 (t, 1H, Ar-*H*), 7.48 (t, *J* = 7.6 Hz, 1H, Ar-*H*), 7.41 (d, *J* = 6.3 Hz, 3H, Ar-*H*) ppm.



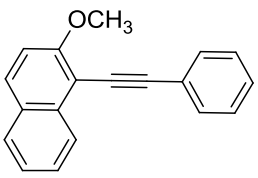
23[13]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.06 (s, 1H, Naphth-*H*), 7.82 (dd, *J* = 8.0, 4.2 Hz, 3H, Naphth-*H*), 7.58 (d, *J* = 8.0 Hz, 3H, Naphth-*H*), 7.53 – 7.46 (m, 2H, Ar-*H*), 7.37 (d, *J* = 6.1 Hz, 3H, Ar-*H*) ppm.



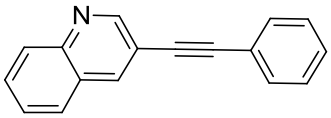
24: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.44 (d, *J* = 8.2 Hz, 1H, Naphth-*H*), 7.83 (d, *J* = 8.0 Hz, 1H, Naphth-*H*), 7.75 (d, *J* = 8.3 Hz, 1H, Naphth-*H*), 7.68 (d, *J* = 6.7 Hz, 2H, Naphth-*H*), 7.63 – 7.55 (t, 1H, Naphth-*H*), 7.53 – 7.45 (t, 1H, Ar-*H*), 7.40 (t, *J* = 7.7 Hz, 4H, Ar-*H*), 2.74 (s, 3H, C*H*$_3$) ppm.

^{13}C NMR (101 MHz, $CDCl_3$): δ 139.23 (s), 133.54 (s), 131.58 (s), 128.59 – 127.92 (m), 126.77 (s), 125.93 (s), 125.46 (s), 123.74 (s), 119.31 (s), 98.79 (s), 86.47 (s), 77.02 (s), 21.44 (s) ppm.

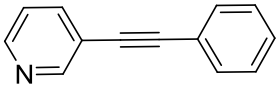
MS (ESI) [$C_{19}H_{14}$]: m/z $(M)^+$: 242.2621.



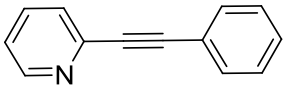
25[7]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.94 – 7.76 (m, 3H, Naphth-*H*), 7.68 (d, *J* = 7.7 Hz, 2H, Naphth-*H*), 7.63 – 7.51 (m, 1H, Naphth-*H*), 7.51 – 7.31 (m, 5H, Ar-*H*), 4.07 (s, 3H, C*H*$_3$) ppm.



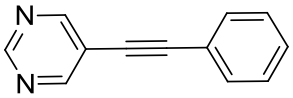
26[9]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 9.01 (s, 1H, Quin-*H*), 8.33 (s, 1H, Quin-*H*), 8.12 (d, *J* = 8.2 Hz, 1H, Quin-*H*), 7.82 (d, *J* = 8.0 Hz, 1H, Quin-*H*), 7.78 – 7.69 (t, 1H, Quin-*H*), 7.60 (d, *J* = 5.7 Hz, 3H, Quin-*H*, Ar-*H*), 7.39 (s, 3H, Ar-*H*) ppm.



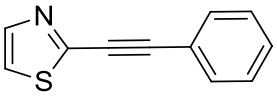
27[1]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.78 (s, 1H, Py-*H*), 8.56 (d, *J* = 4.9 Hz, 1H, Py-*H*), 7.90 – 7.74 (m, 1H, Py-*H*), 7.56 (dd, *J* = 6.6, 2.9 Hz, 2H, , Py-*H*, Ar-*H*), 7.41 – 7.35 (m, 3H, Ar-*H*), 7.31 (dd, *J* = 8.3, 4.5 Hz,1H, Ar-*H*) ppm.



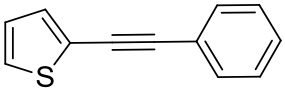
28[6]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.64 (d, *J* = 4.4 Hz, 1H, Py-*H*), 7.73 (t, *J* = 7.6 Hz, 1H, Py-*H*), 7.62 (d, *J* = 5.1 Hz, 2H, Py-*H*), 7.56 (d, *J* = 7.8 Hz, 1H, Ar-*H*), 7.37 (d, *J* = 5.0 Hz, 3H, Ar-*H*), 7.32 – 7.23 (m, 1H, Ar-*H*) ppm.



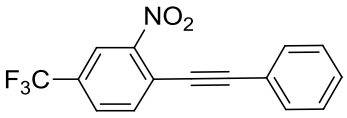
29[9]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 9.15 (s, 1H, Pyrim-*H*), 8.87 (s, 2H, Pyrim-*H*), 7.65 – 7.47 (m, 2H , Ar-*H*), 7.46 – 7.36 (m, 3H Ar-*H*) ppm.



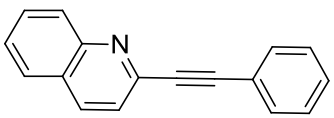
30[9]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.57 – 7.50 (m, 3H,Th-*H*, Ar-*H*), 7.42 – 7.31 (m, 4H, Ar-*H*) ppm.



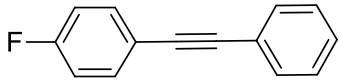
31[16]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.51 (dd, *J* = 7.4, 2.2 Hz, 2H,Th-*H*), 7.36 – 7.32 (m, 3H, Ar-*H*), 7.28 (dd, *J* = 4.2, 3.4 Hz, 2H, Ar-*H*), 7.01 (dd, *J* = 5.0, 3.8 Hz, 1H,Th-*H*) ppm.



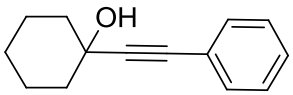
32[14]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.37 (s, 1H), 7.86 (s, 2H, Ar-*H*), 7.63 (dd, *J* = 7.5, 1.8 Hz, 2H, Ar-*H*), 7.42 (m, *J* = 5.9 Hz, 3H, Ar-*H*) ppm.



33[9]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.14 (t, *J* = 7.5 Hz, 2H, Quin-*H*), 7.81 (d, *J* = 8.1 Hz, 1H, Quin-*H*), 7.78 – 7.70 (m, 1H, Quin-*H*), 7.67 (dd, *J* = 6.5, 3.2 Hz, 2H, Quin-*H*), 7.62 (d, *J* = 8.5 Hz, 1H, Ar-*H*), 7.59 – 7.51 (m, 1H, Ar-*H*), 7.45 – 7.34 (m, 3H, Ar-*H*) ppm.



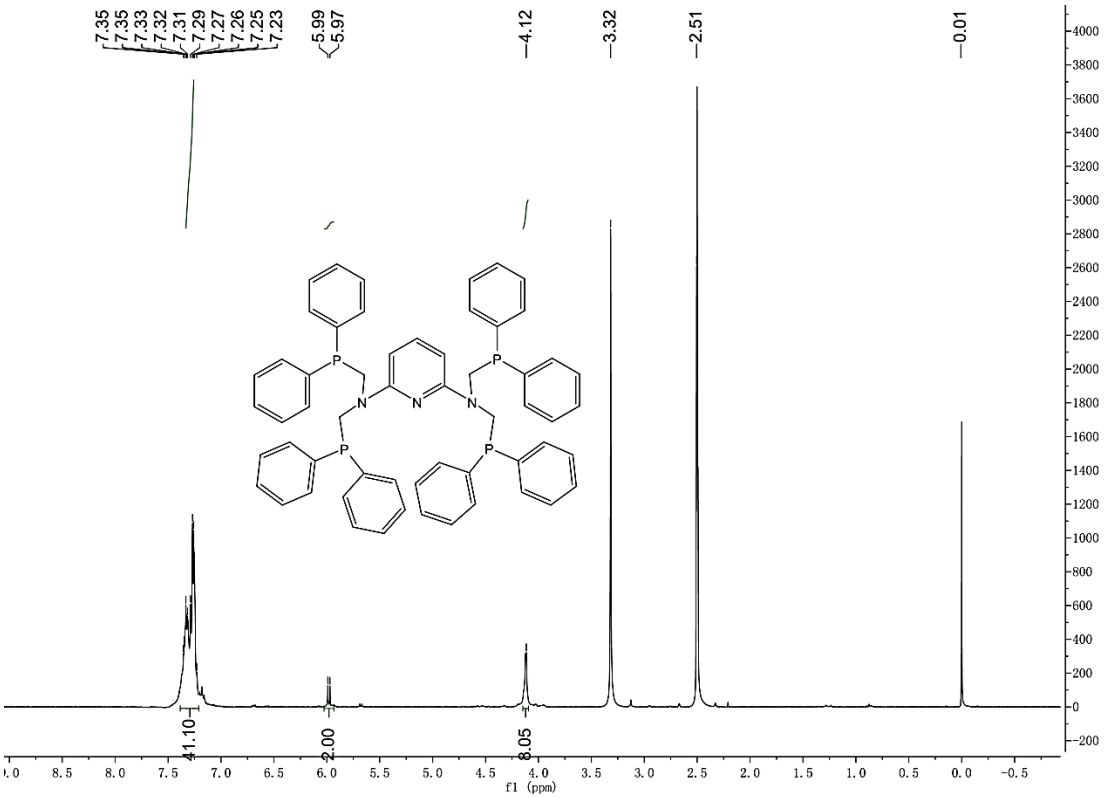
34[13]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.54 – 7.46 (m, 4H, Ar-*H*), 7.39 – 7.31 (m, 3H, Ar-*H*), 7.04 (t, *J* = 8.7 Hz, 2H, Ar-*H*) ppm.



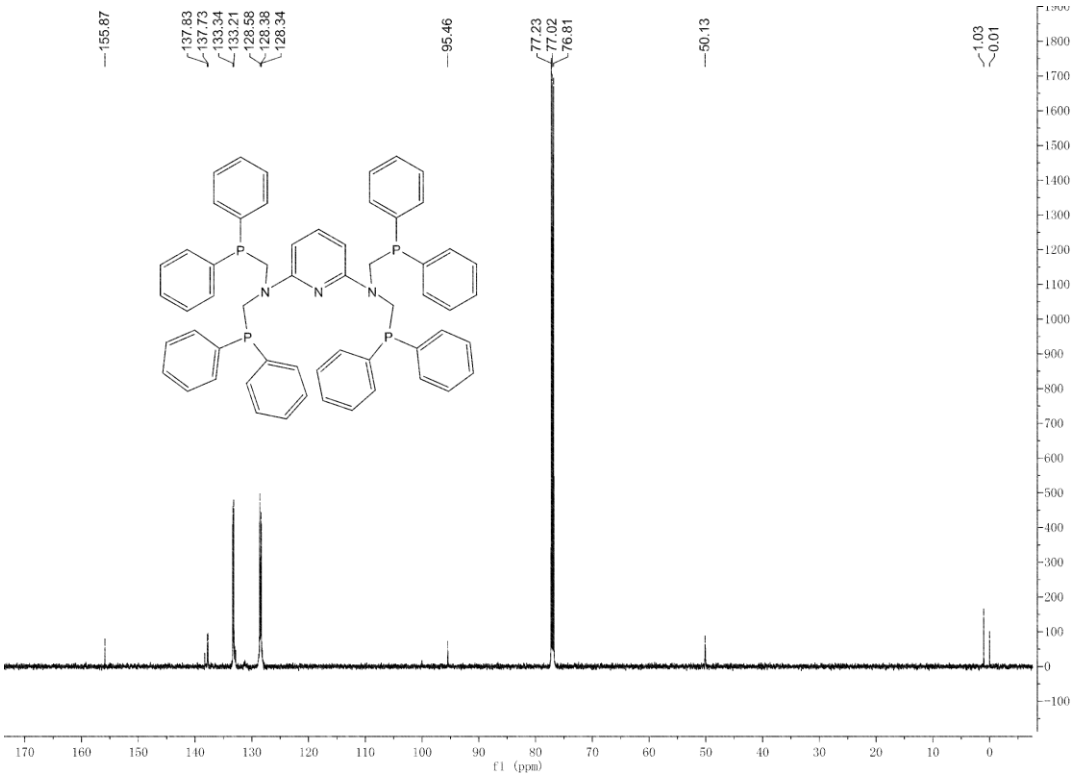
35[14]: ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 7.43 (dd, *J* = 6.5, 3.1 Hz, 2H, Ar-*H*), 7.31 (dd, *J* = 6.4, 3.5 Hz, 3H, Ar-*H*), 2.10 – 1.92 (m, 3H, Cyclohexan-*H*), 1.82 – 1.53 (m, 8H, Cyclohexan-*H*, O*H*) ppm.

NMR spectra of compounds

^{1}H NMR of ligand **1:**

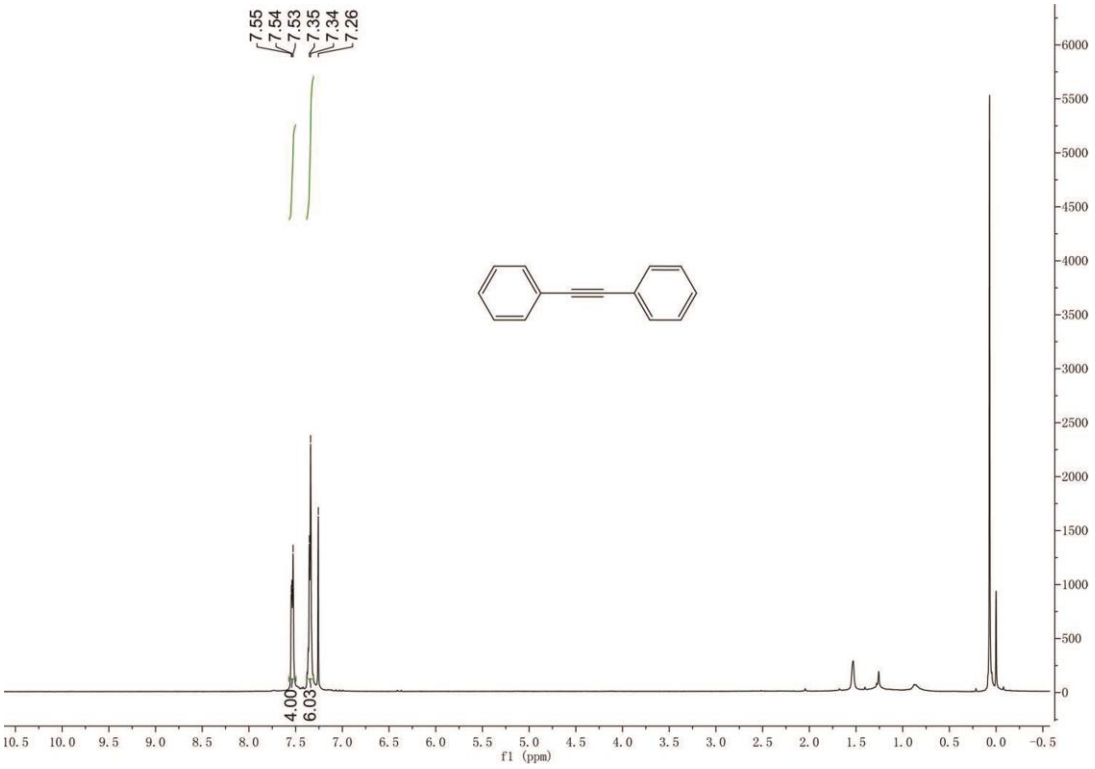


^{13}C NMR of ligand **1**

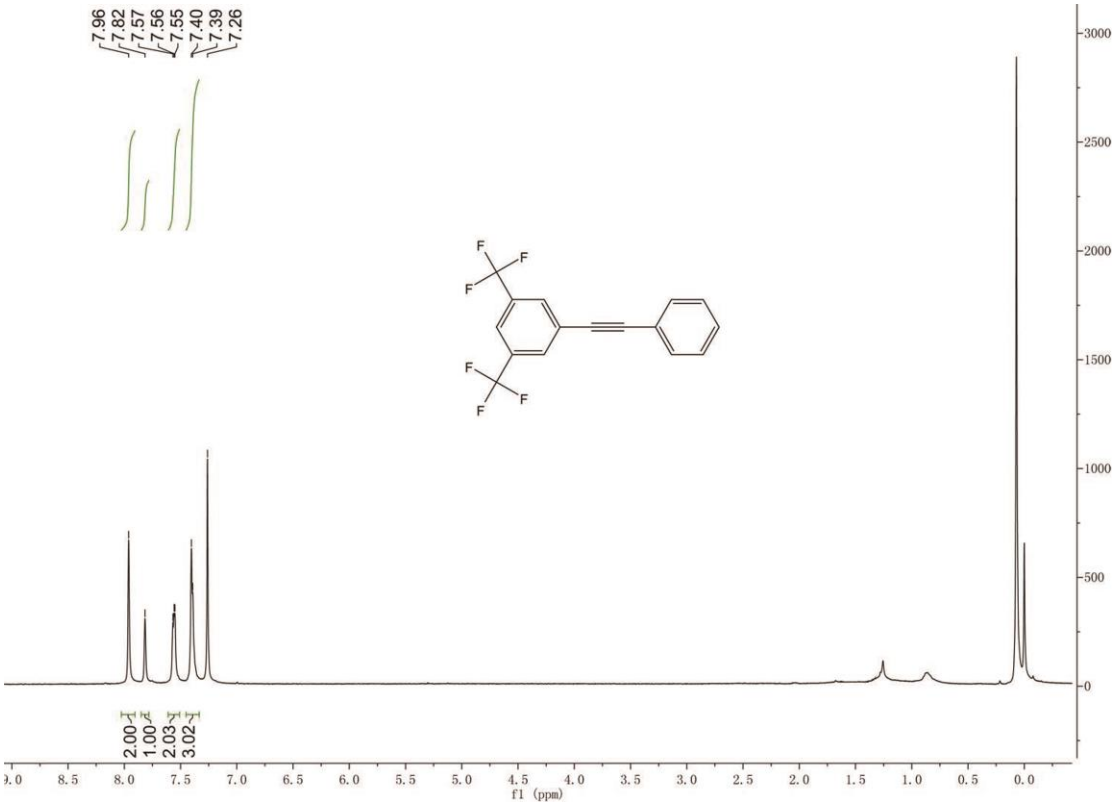


^{31}P NMR of ligand **1:**

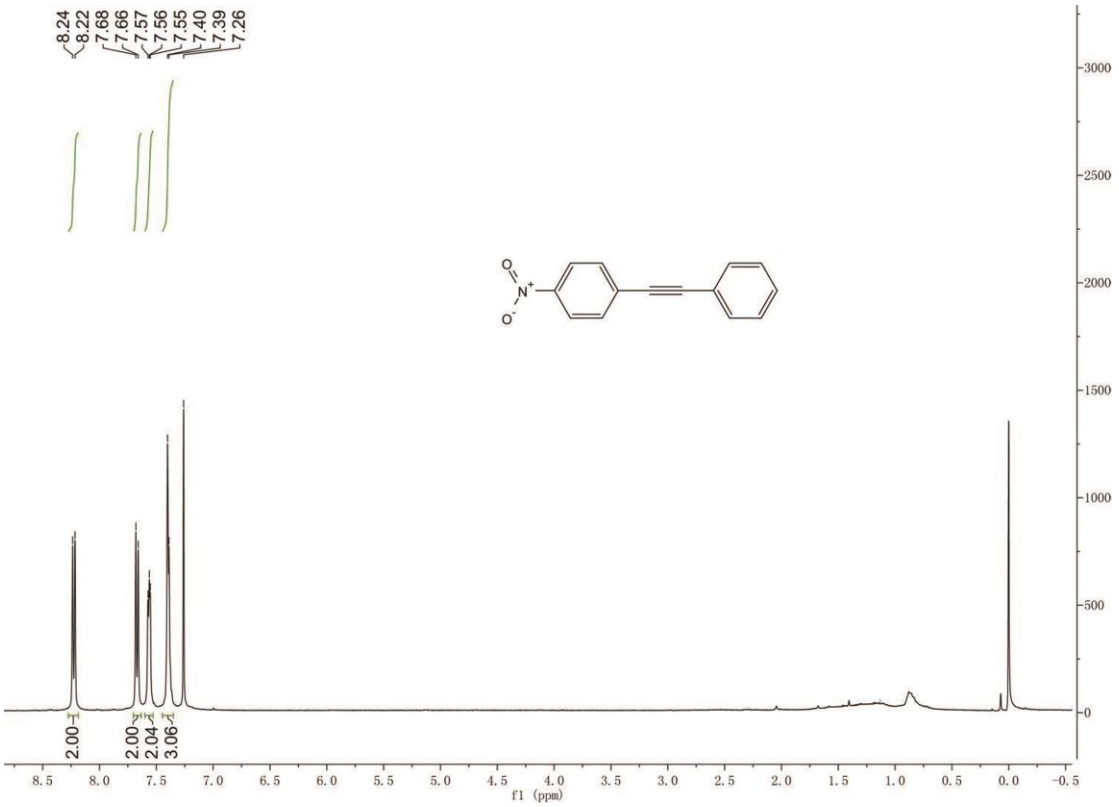
^{1}H NMR of **3**



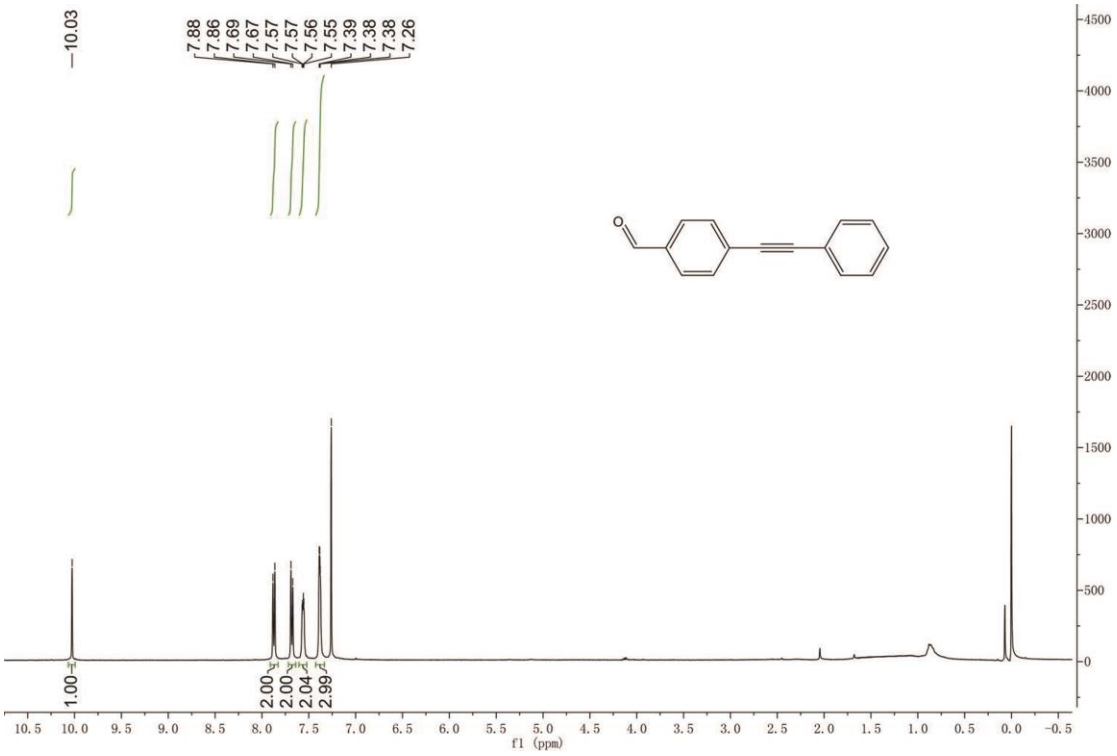
^{1}H NMR of **4**



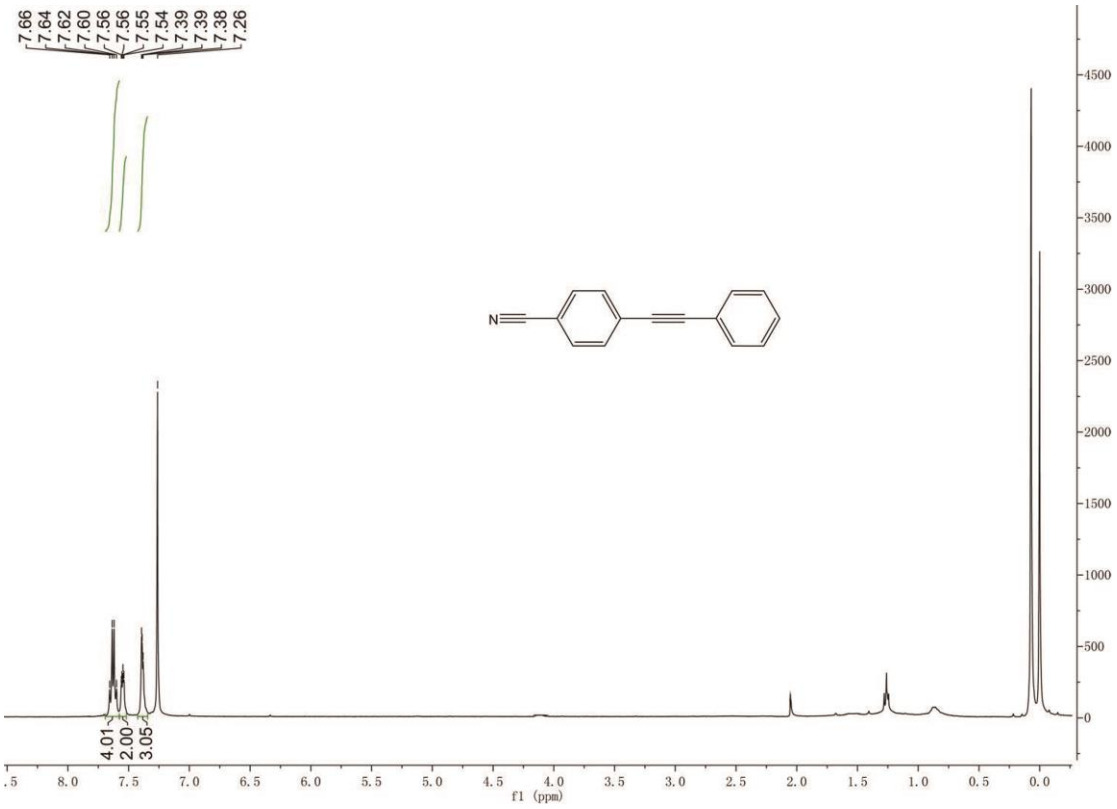
^{1}H NMR of **5**



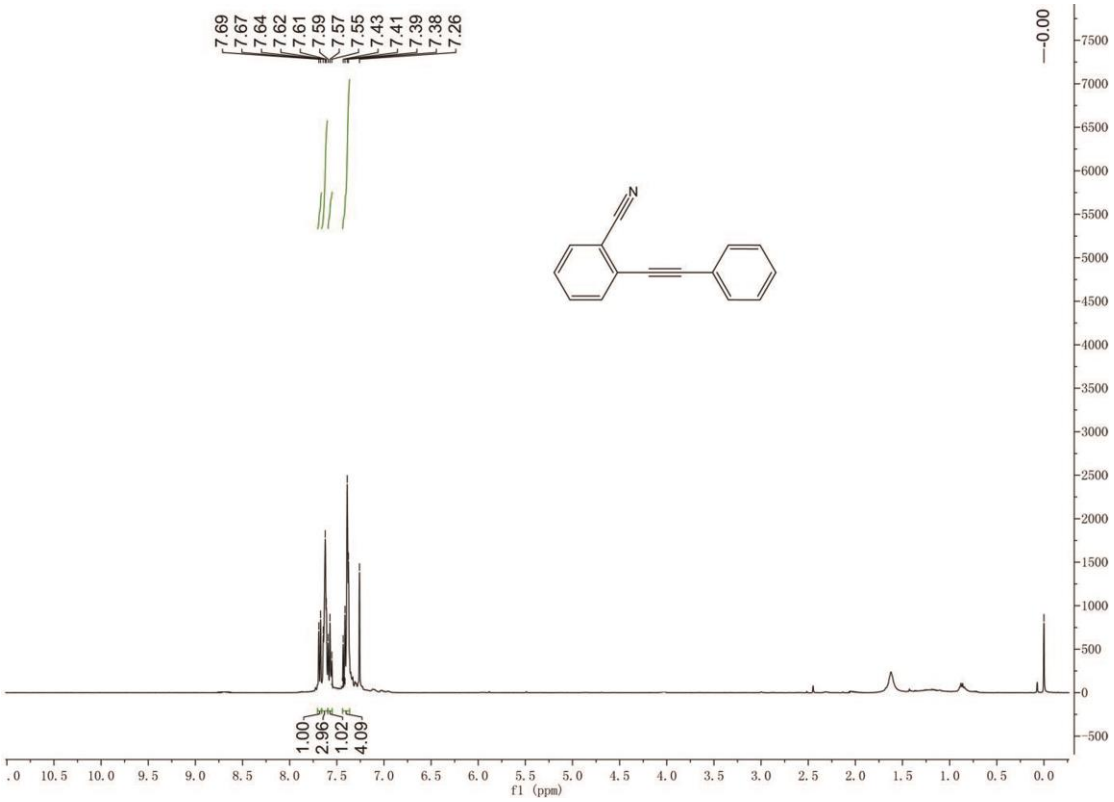
^{1}H NMR of **6**



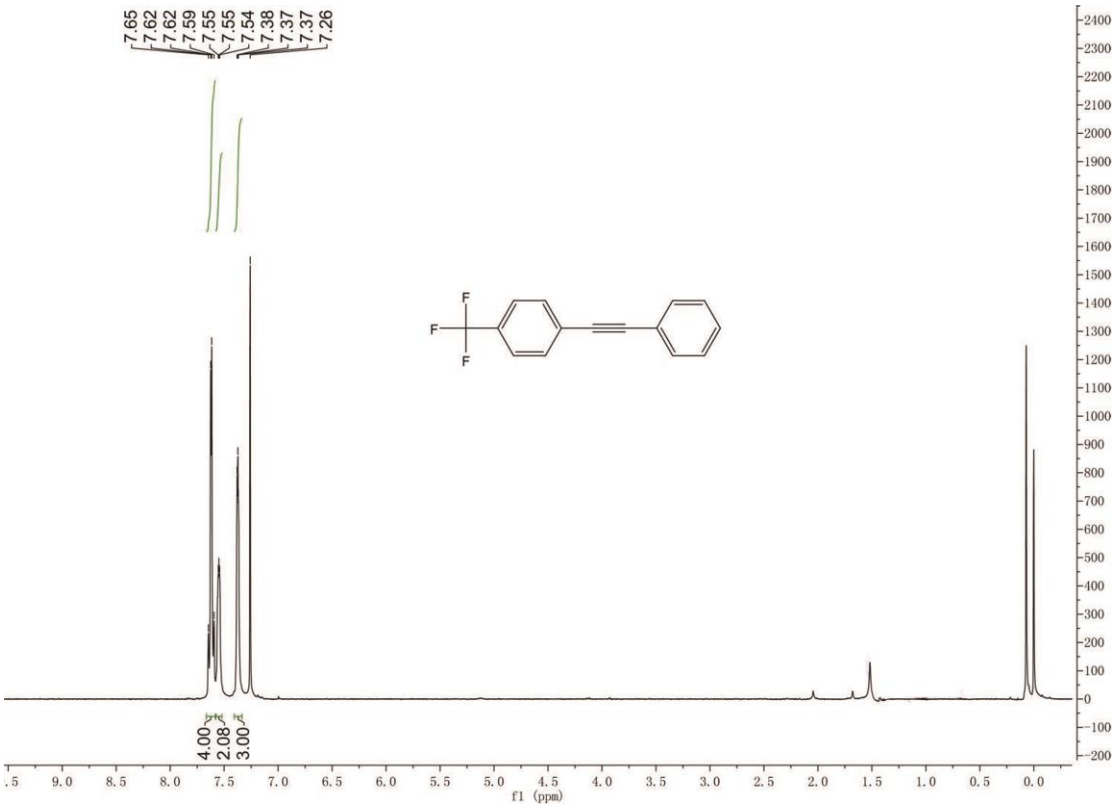
^{1}H NMR of **7**



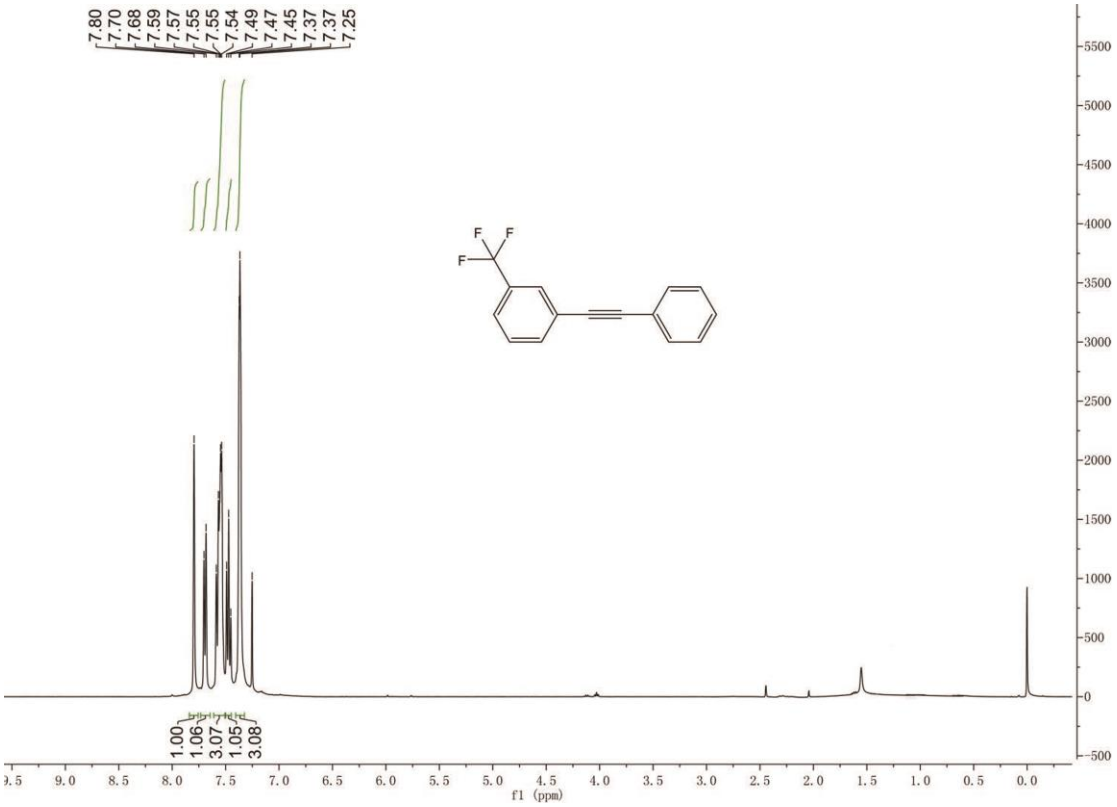
^{1}H NMR of **8**



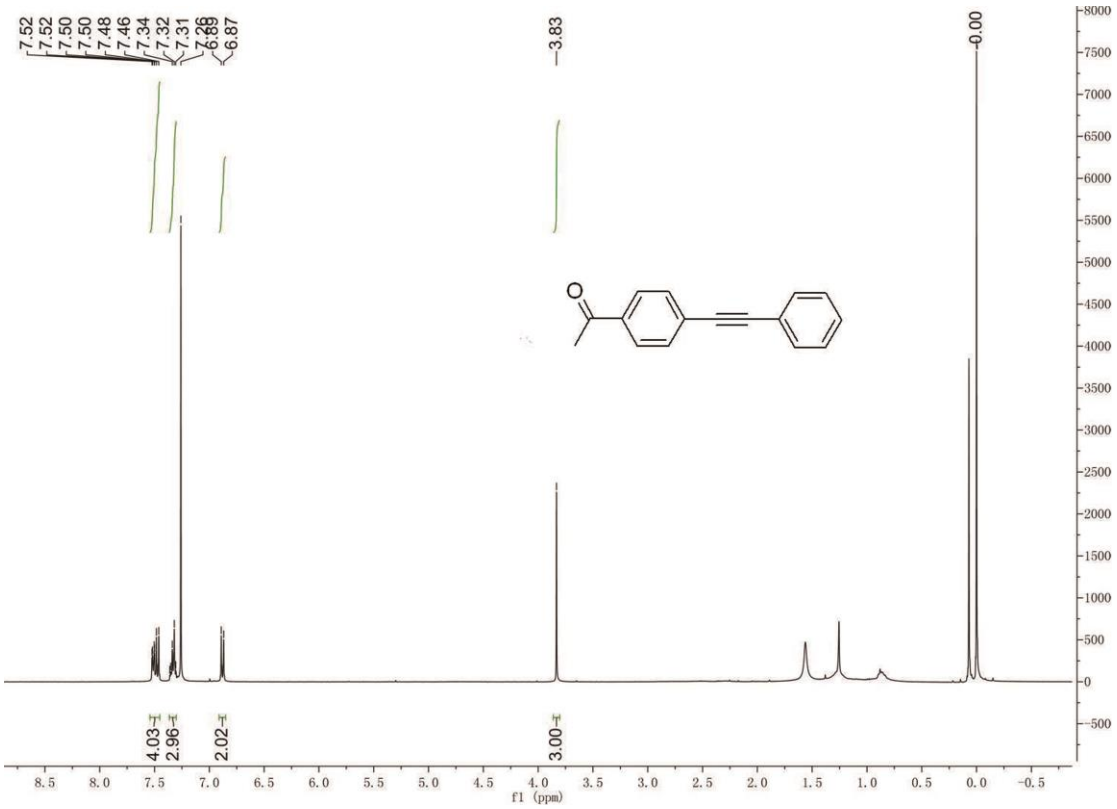
^{1}H NMR of **9**



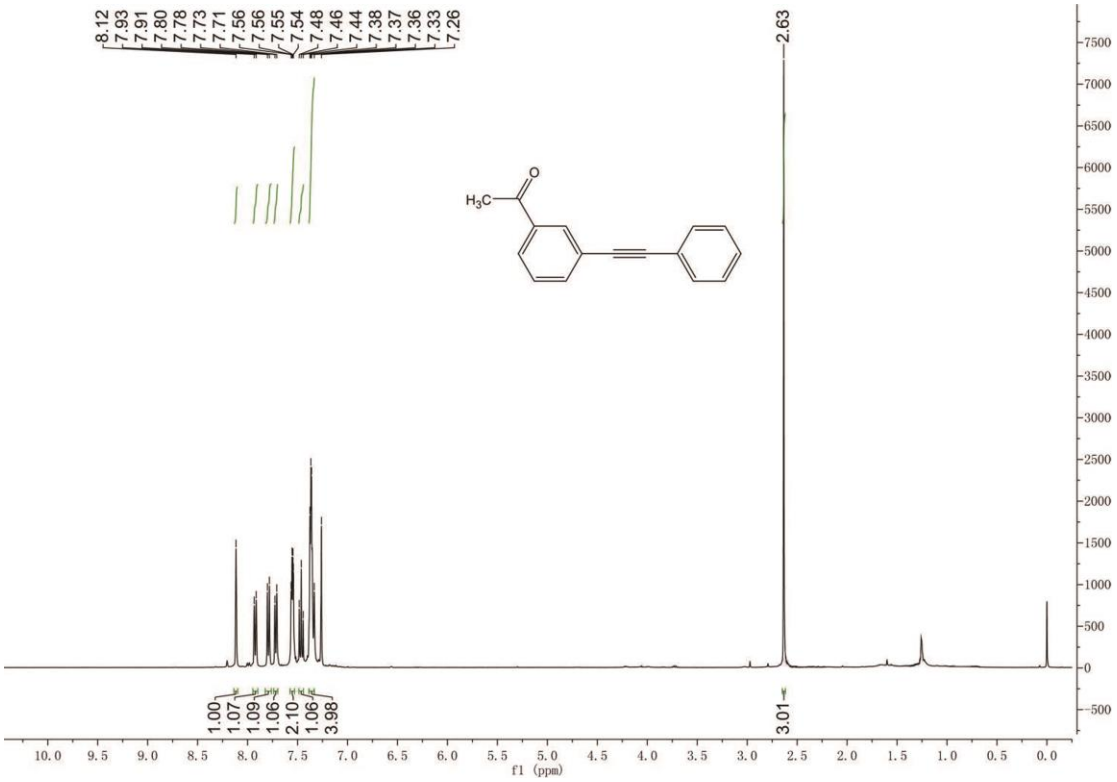
^{1}H NMR of **10**



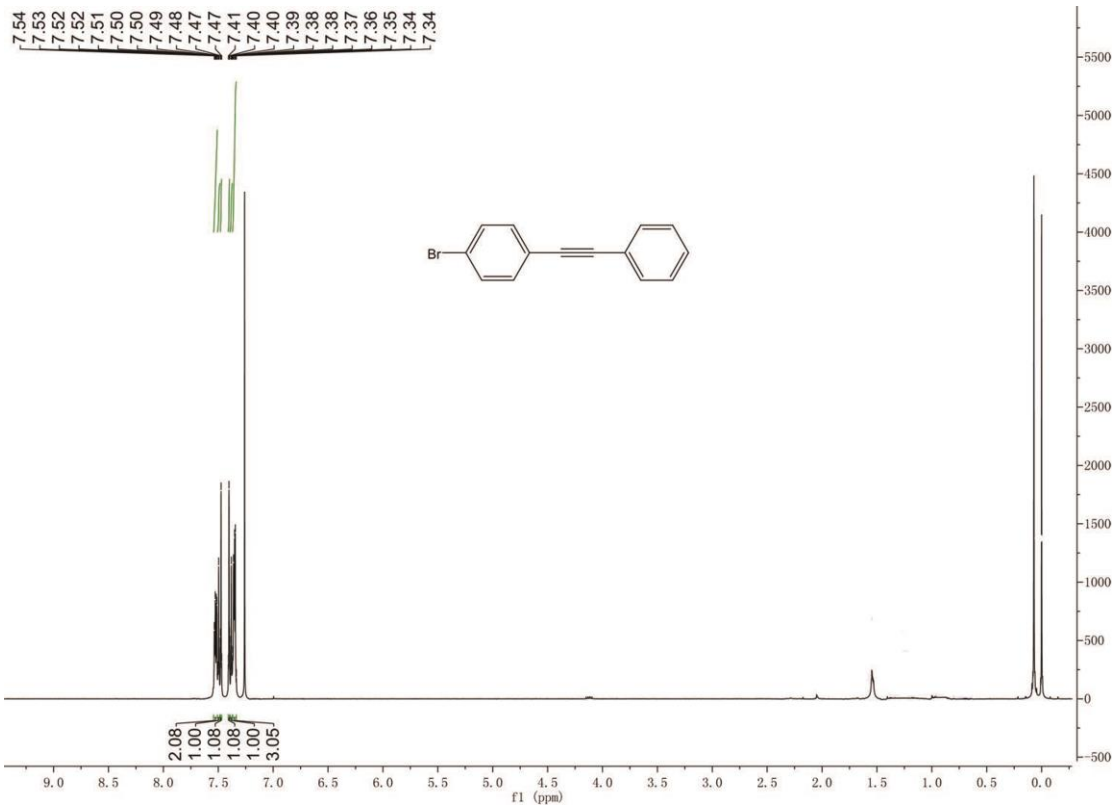
^{1}H NMR of **11**



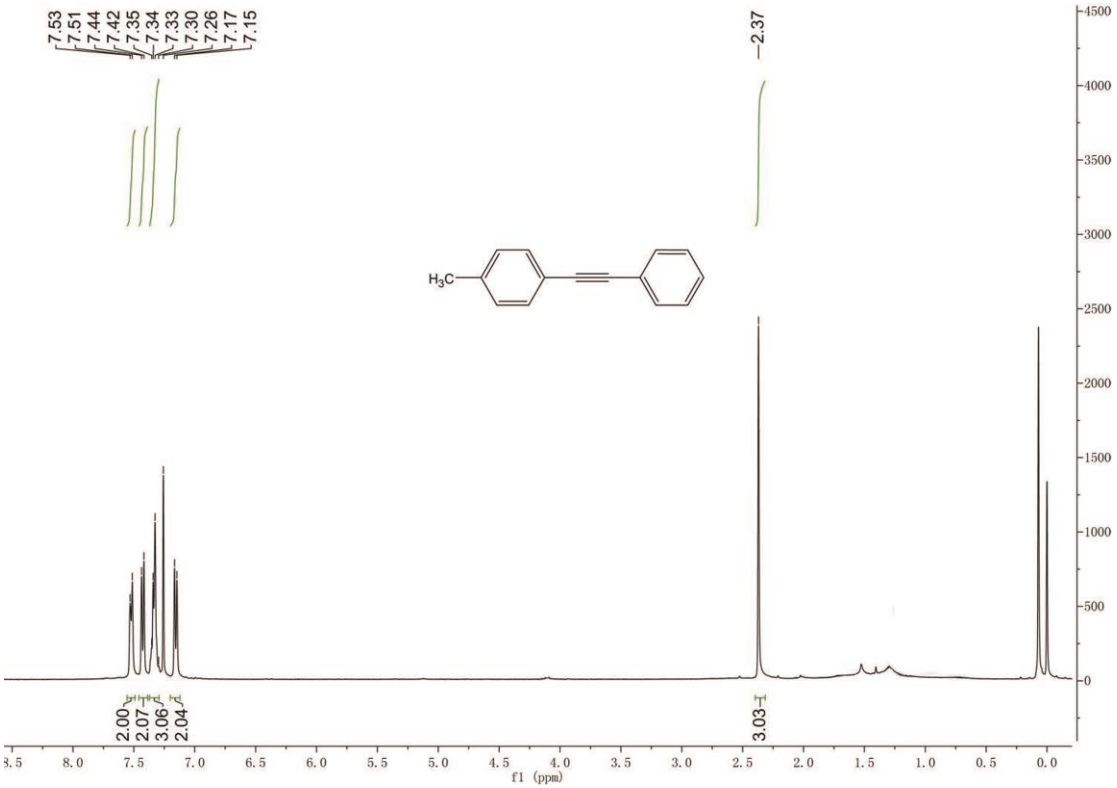
^{1}H NMR of **12**



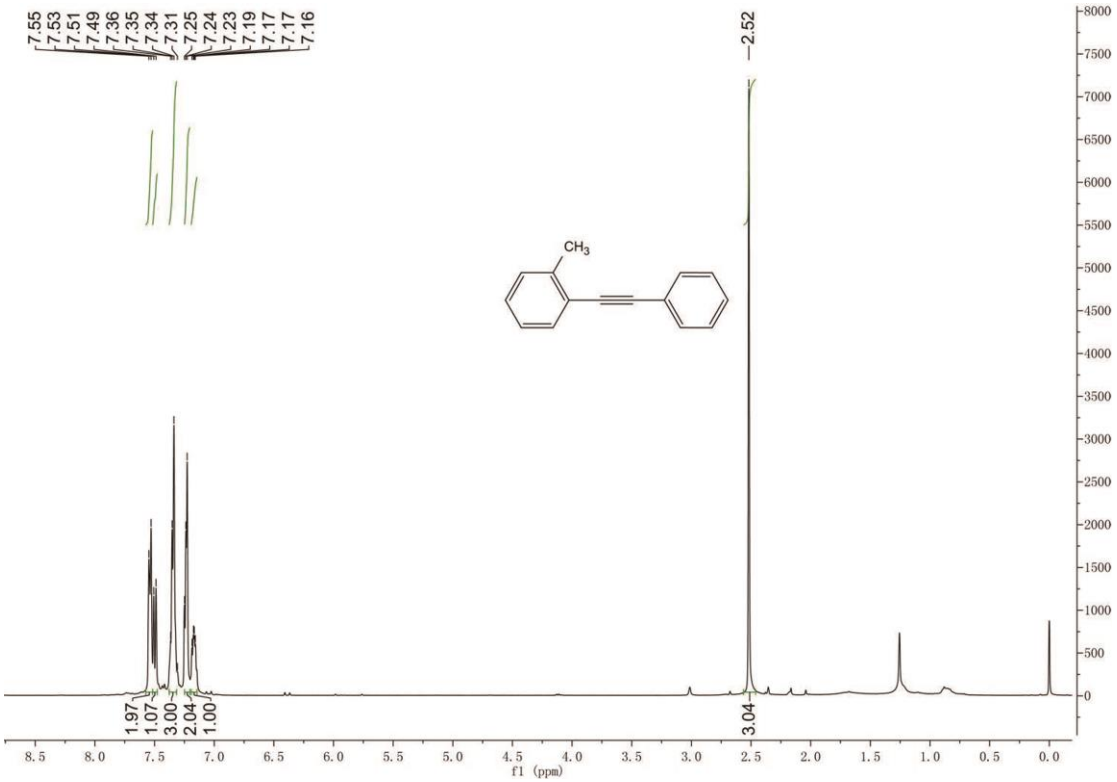
^{1}H NMR of **13**



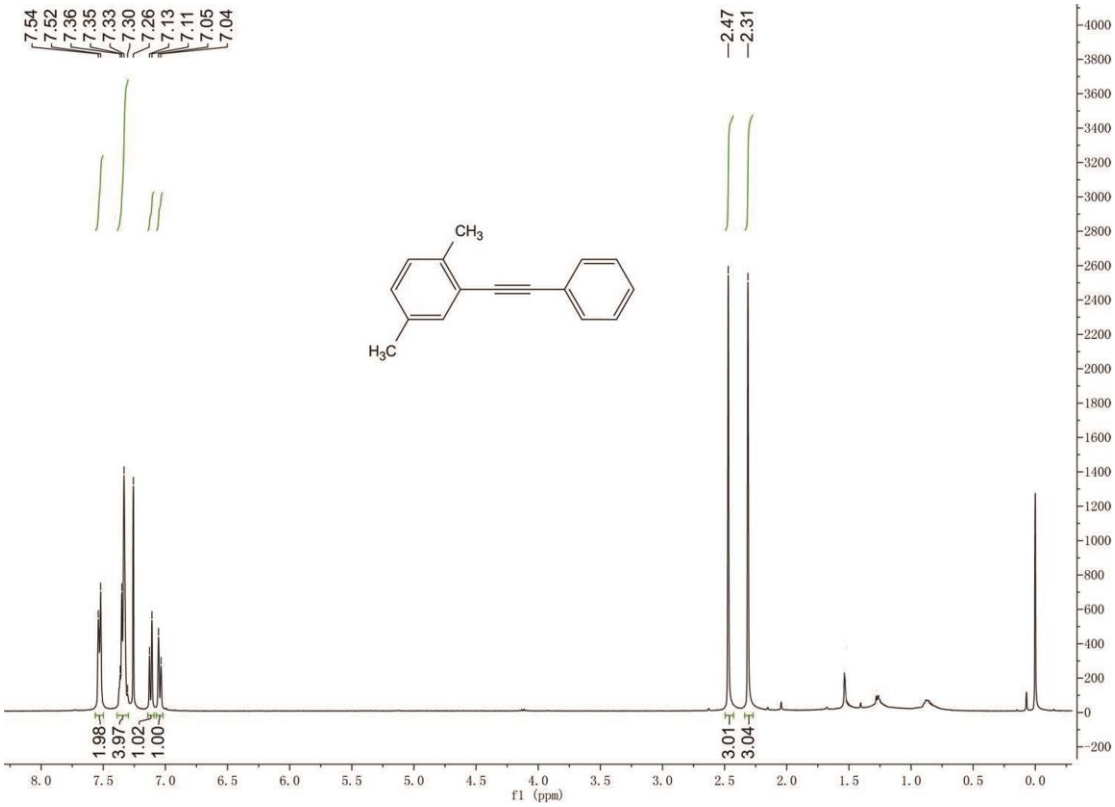
^{1}H NMR of **14**



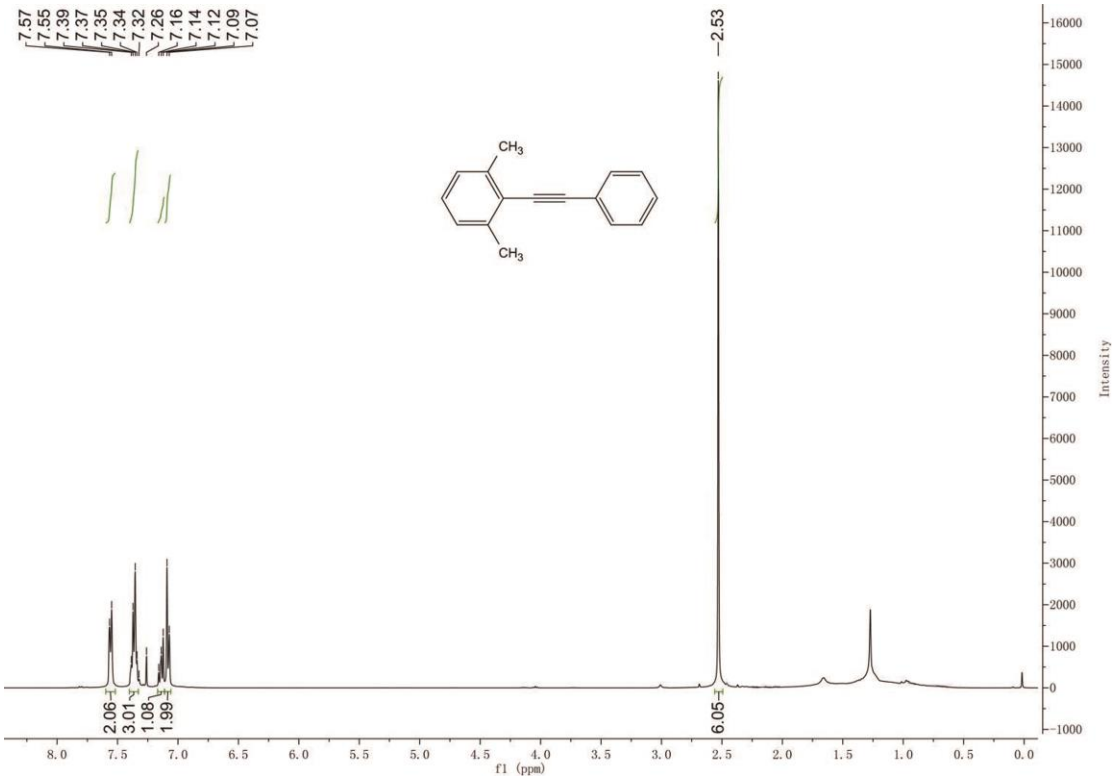
^{1}H NMR of **15**



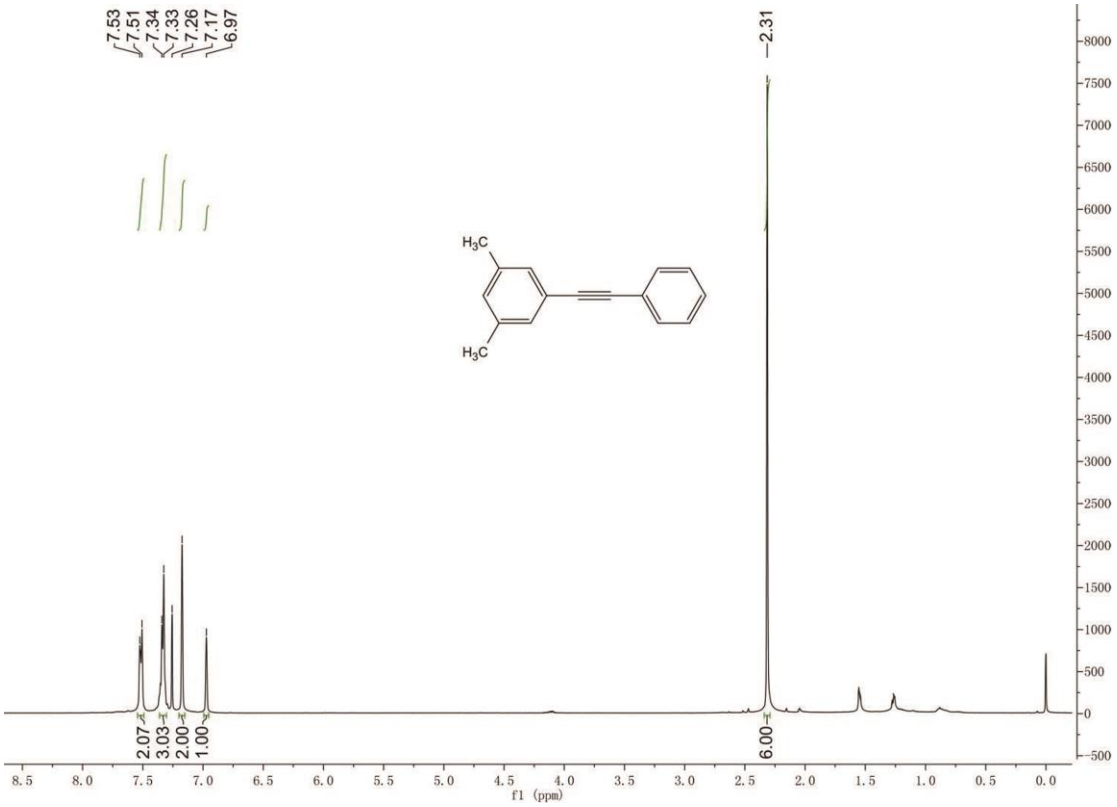
^{1}H NMR of **16**



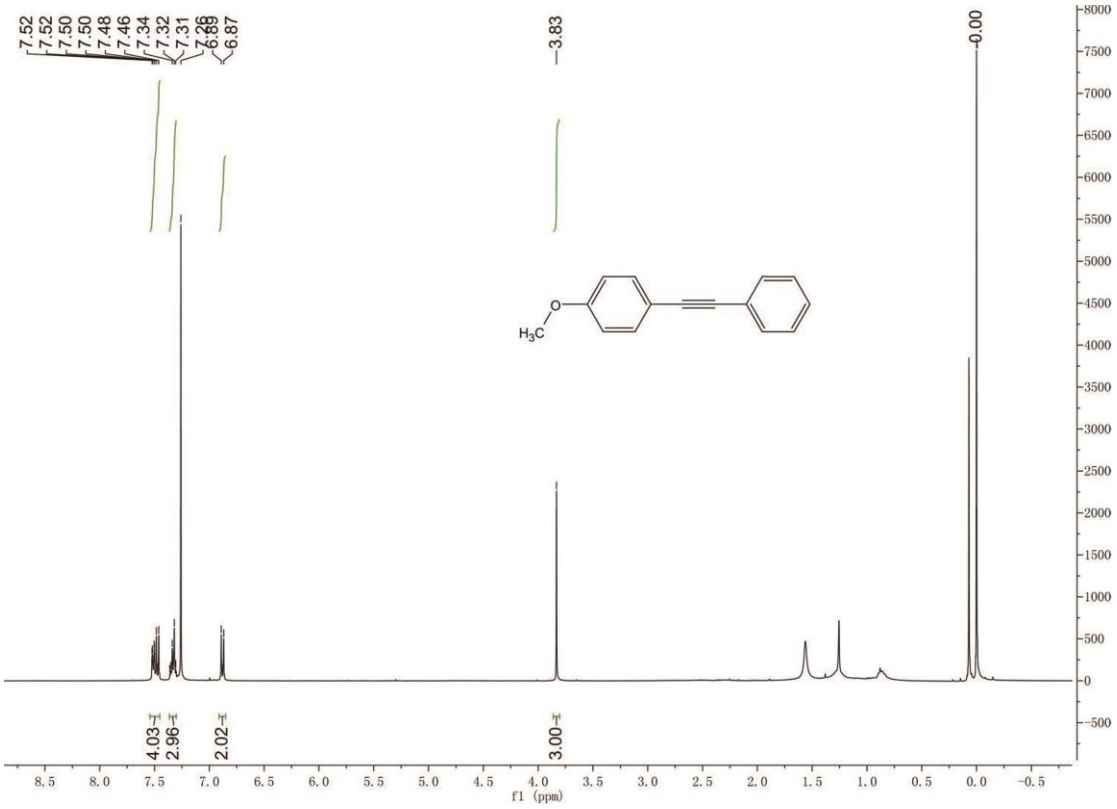
^{1}H NMR of **17**



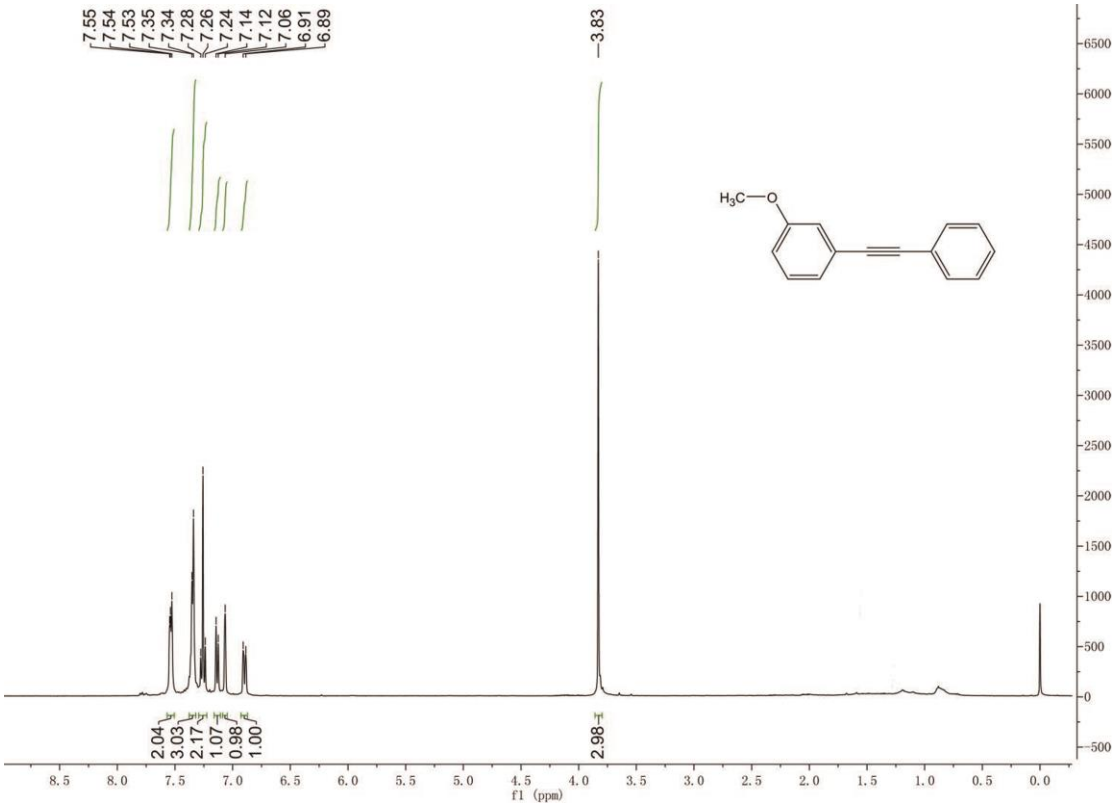
^{1}H NMR of **18**



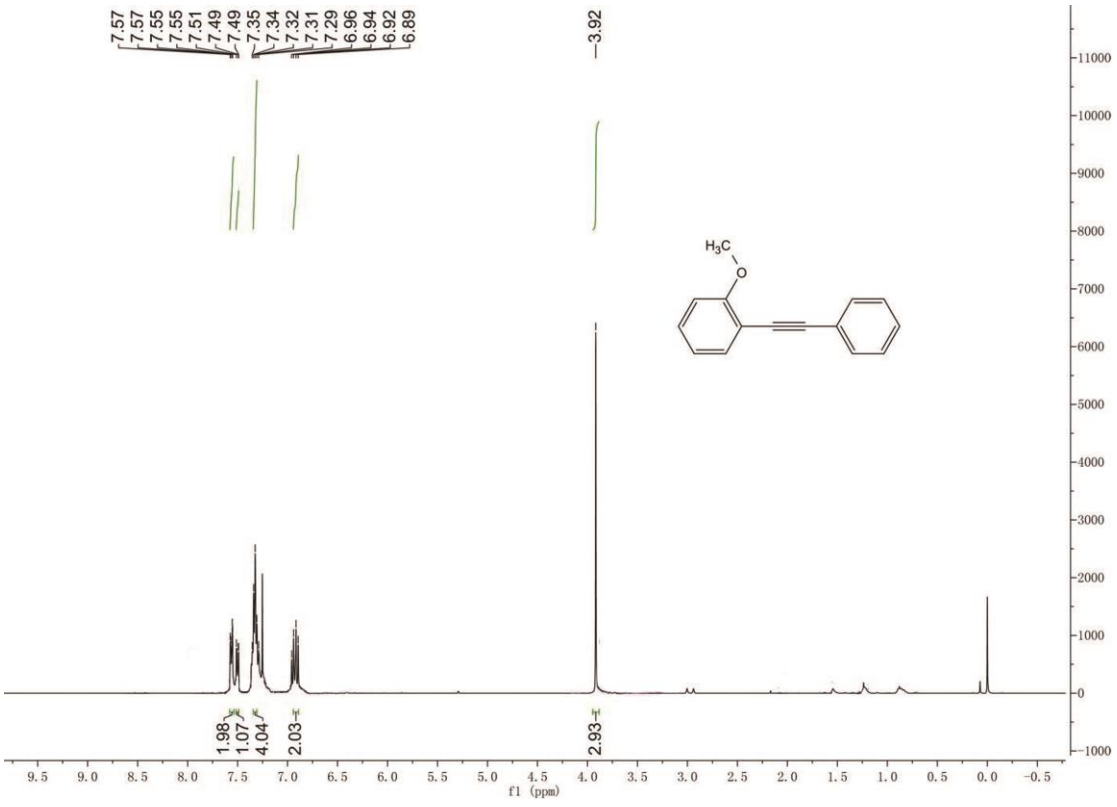
^{1}H NMR of **19**



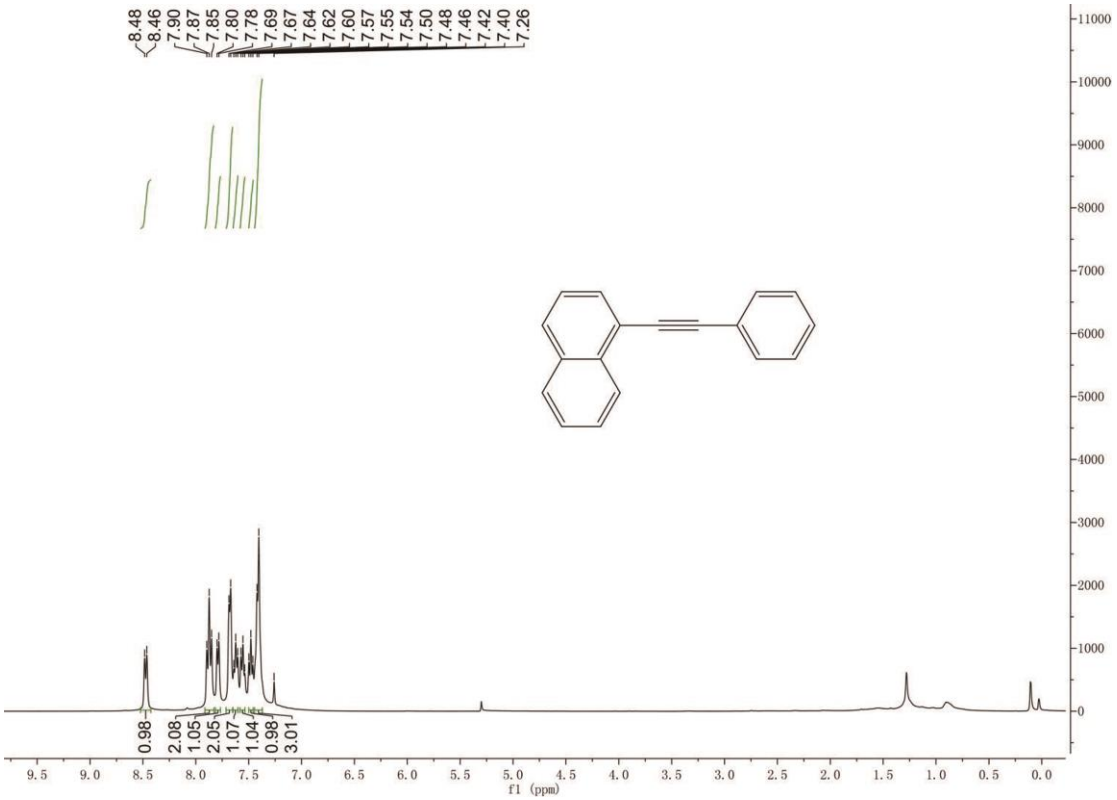
^{1}H NMR of **20**



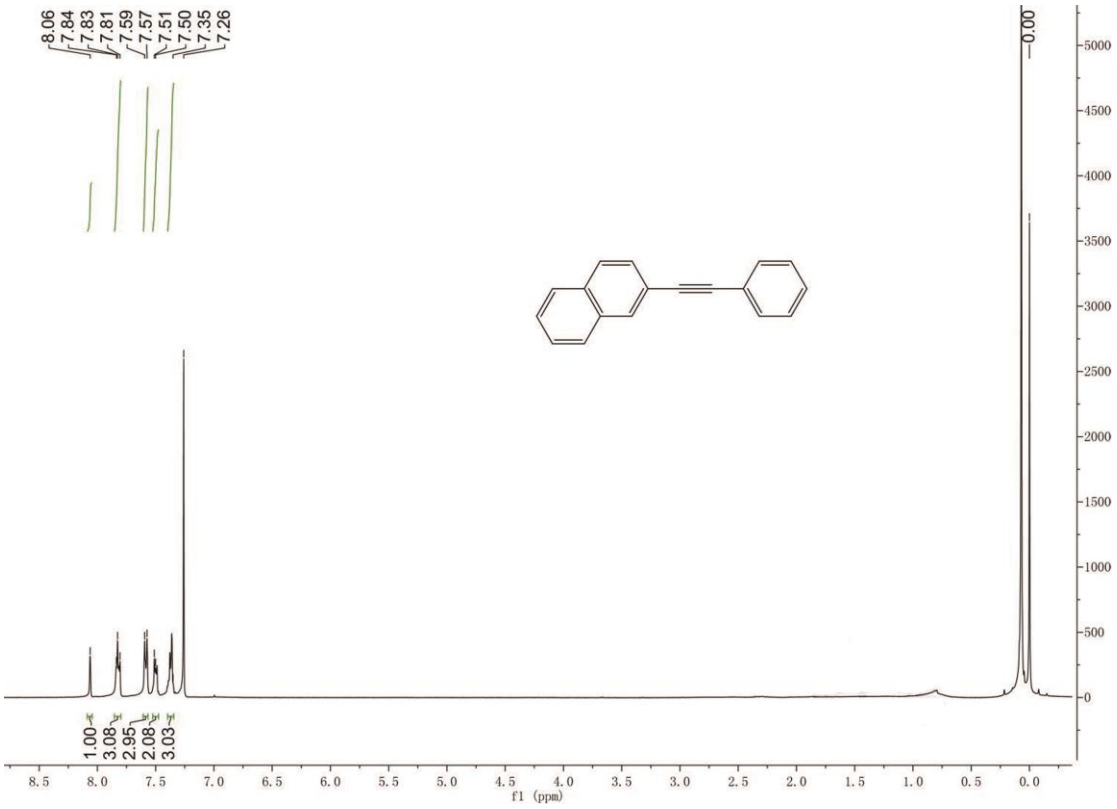
^{1}H NMR of **21**



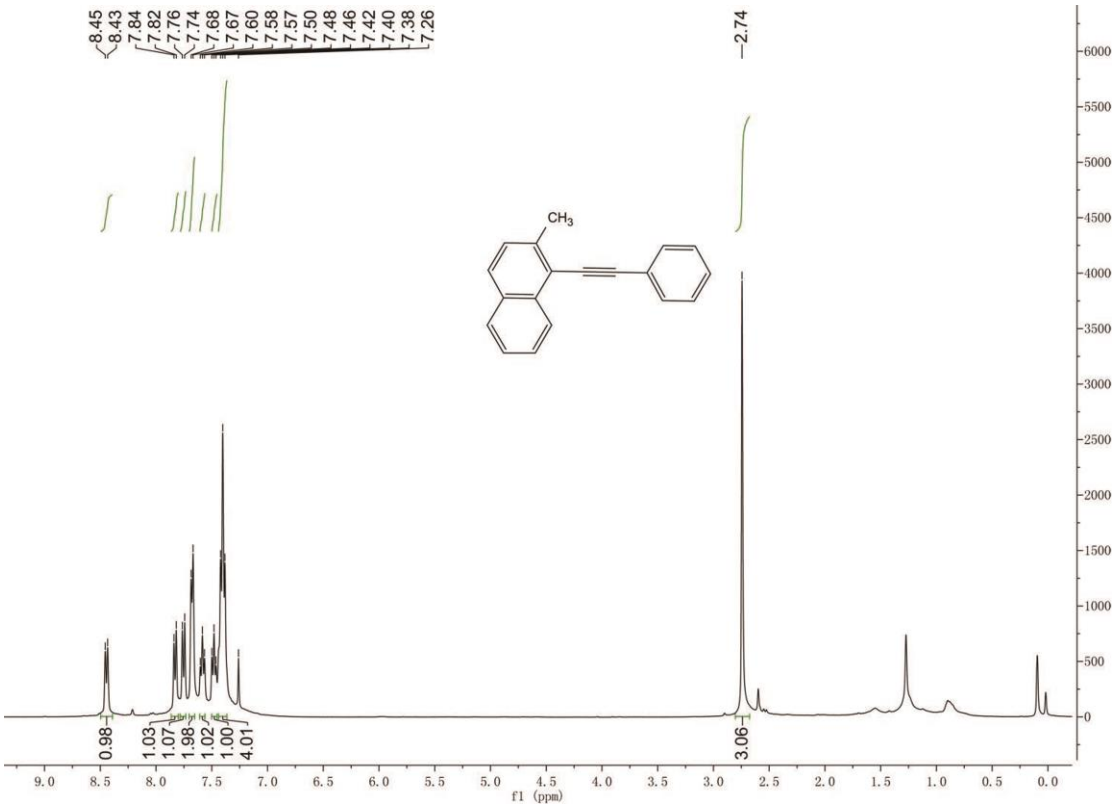
^{1}H NMR of **22**



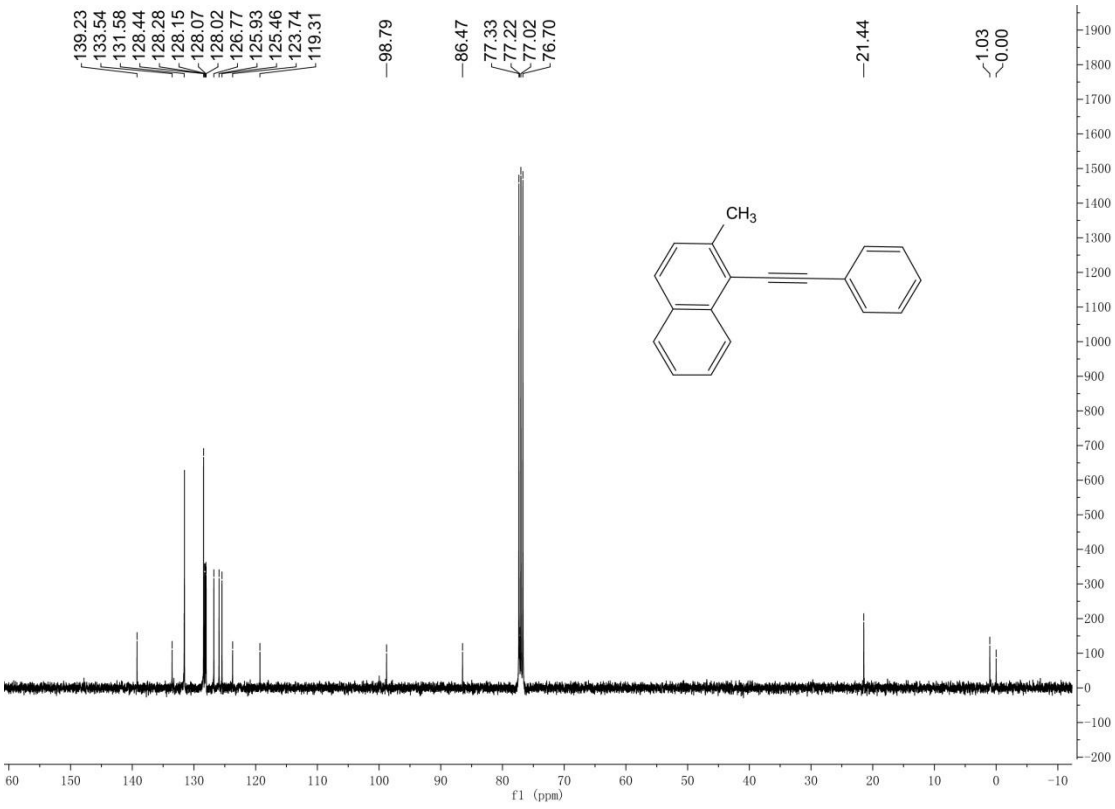
^{1}H NMR of **23**



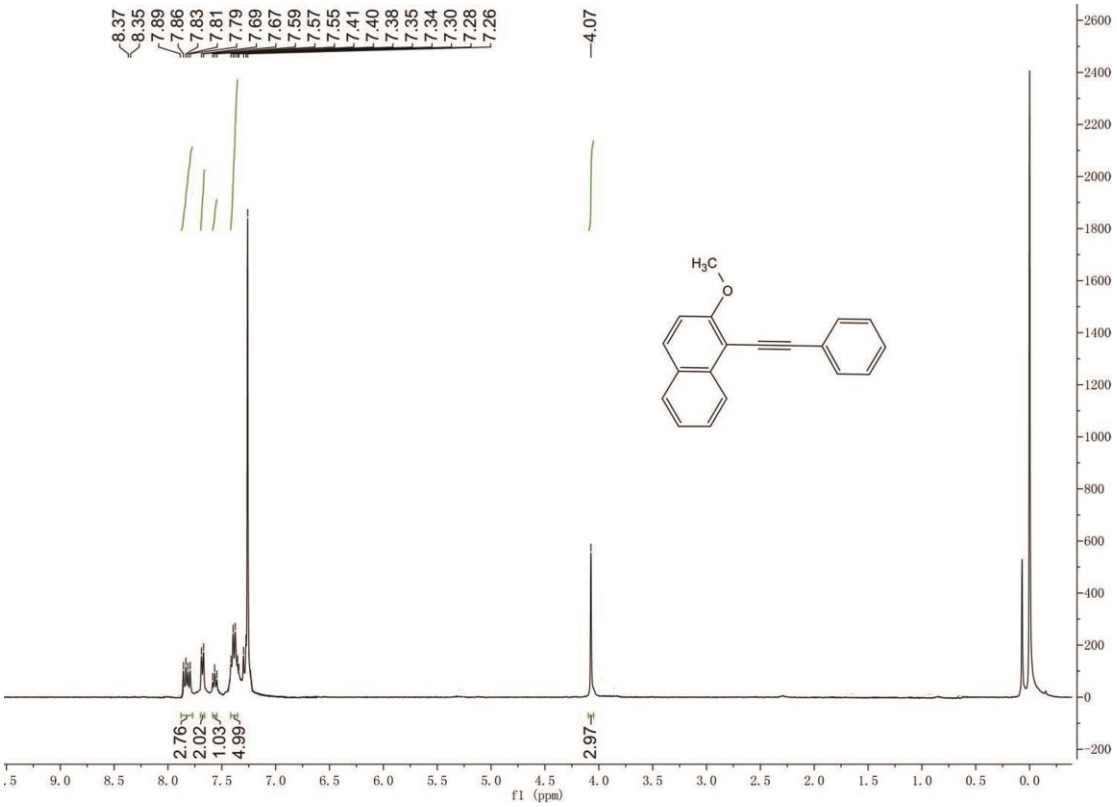
^{1}H NMR of **24**



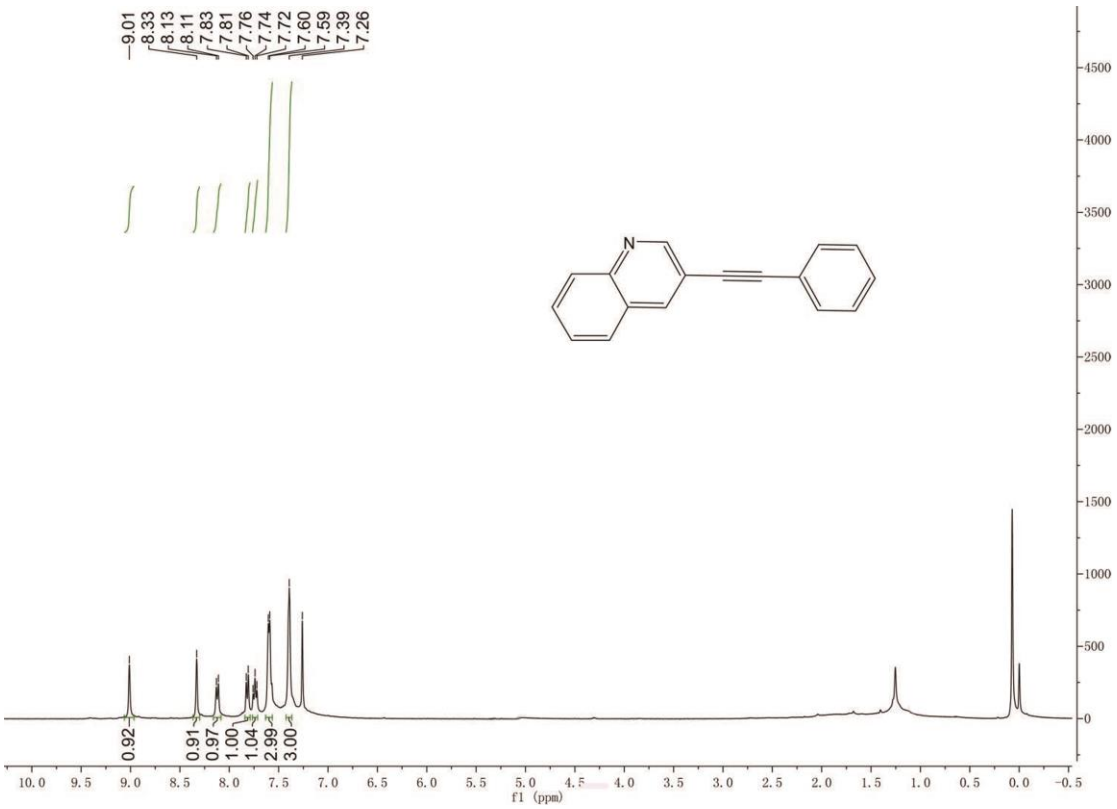
^{13}C NMR of **24**



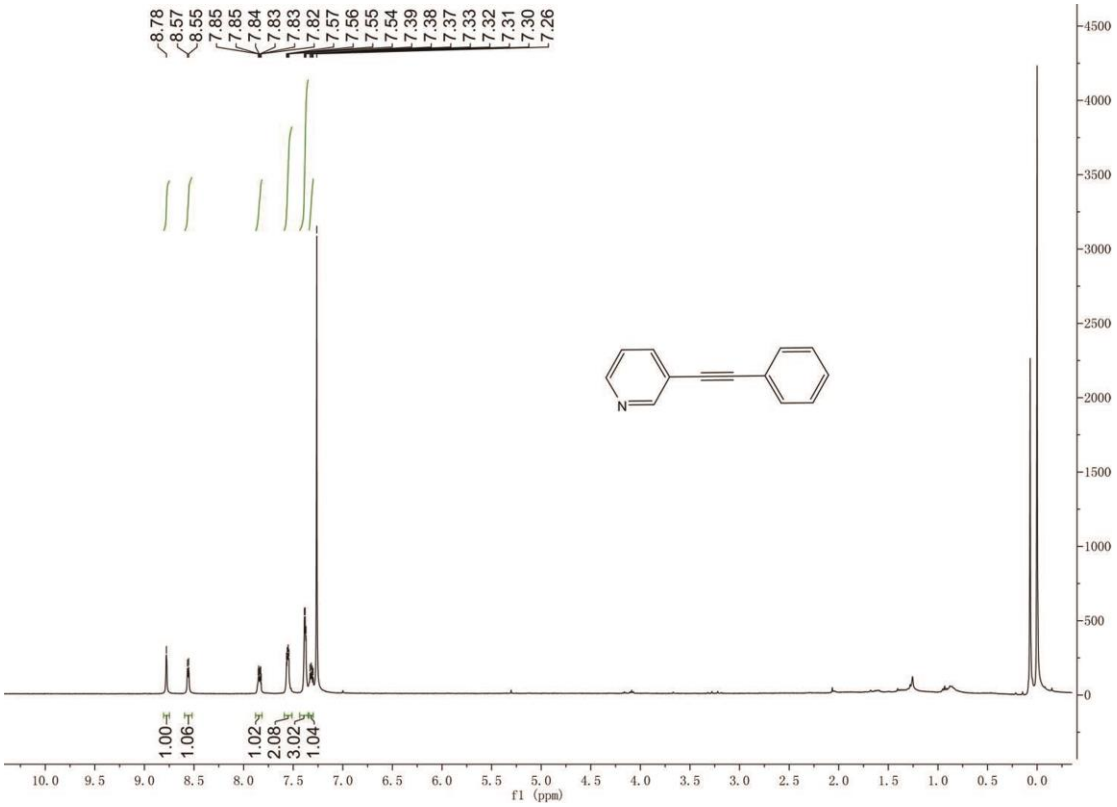
^{1}H NMR of **25**



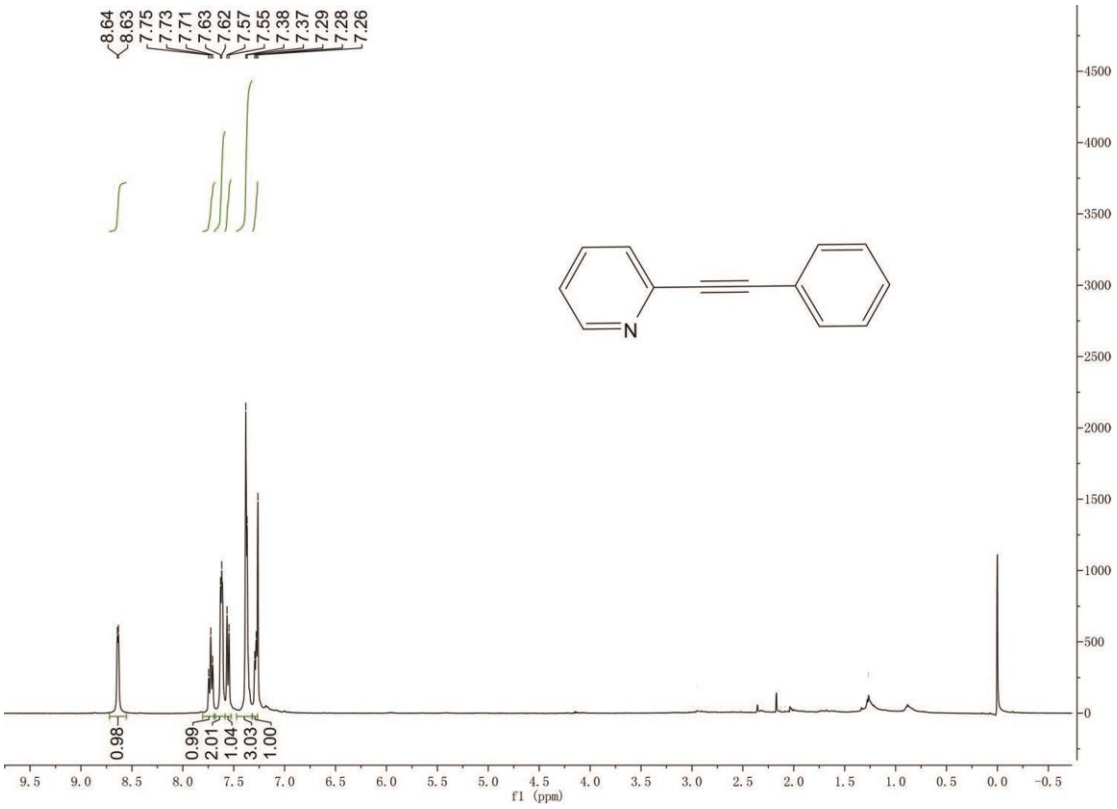
^{1}H NMR of **26**



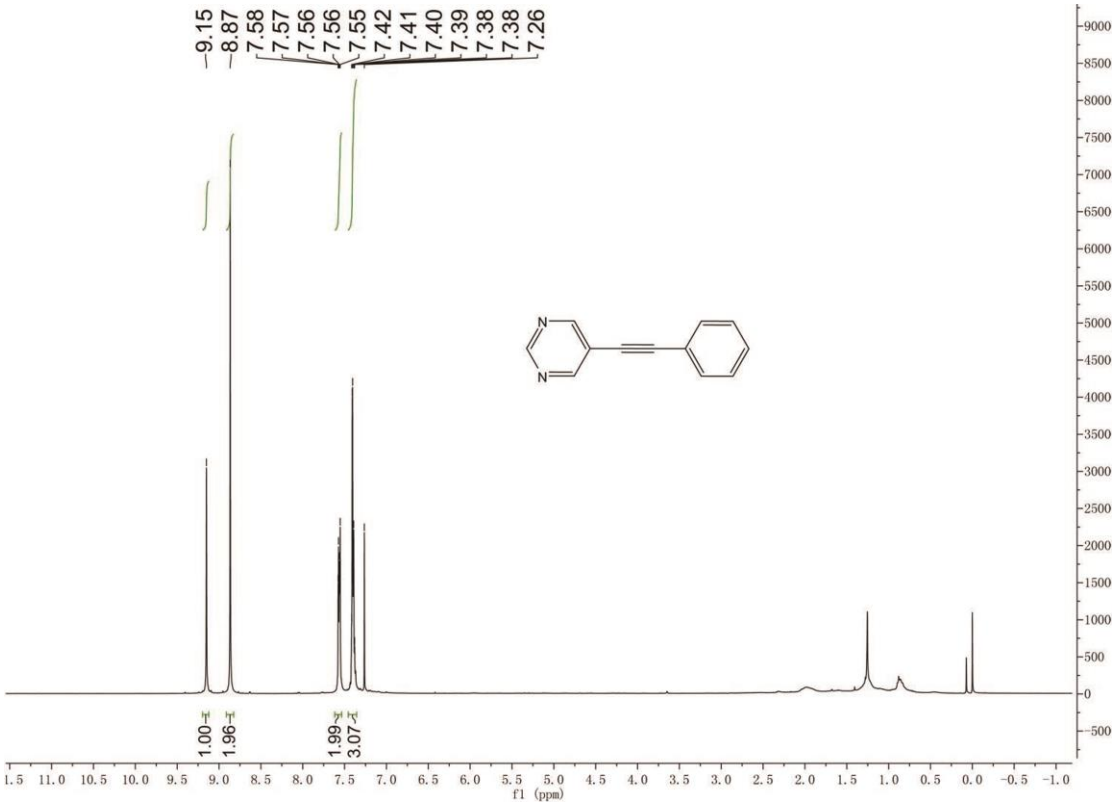
^{1}H NMR of **27**



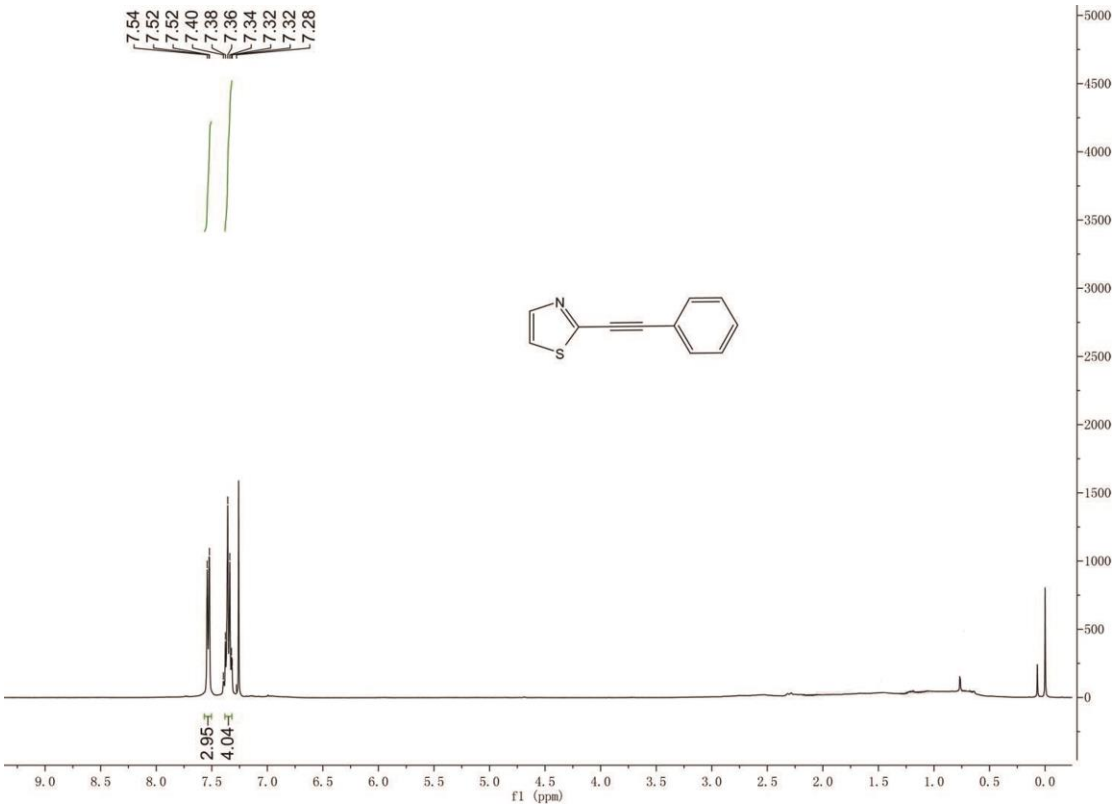
^{1}H NMR of **28**



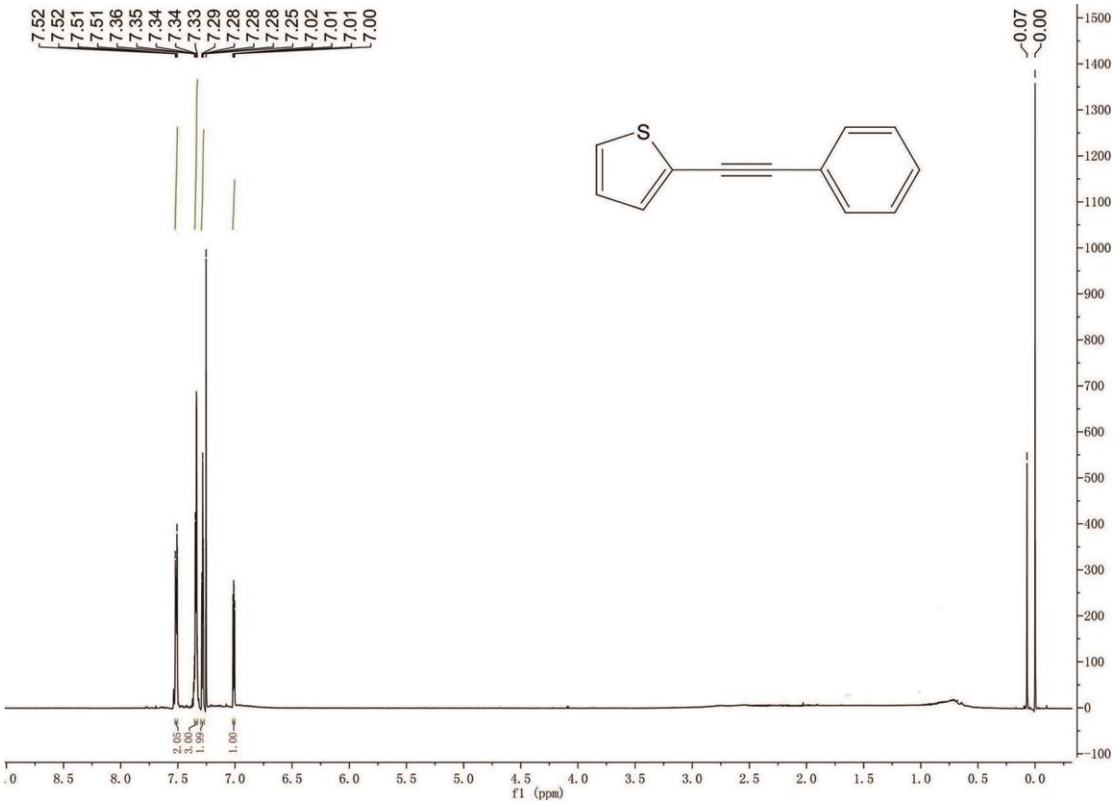
^{1}H NMR of **29**



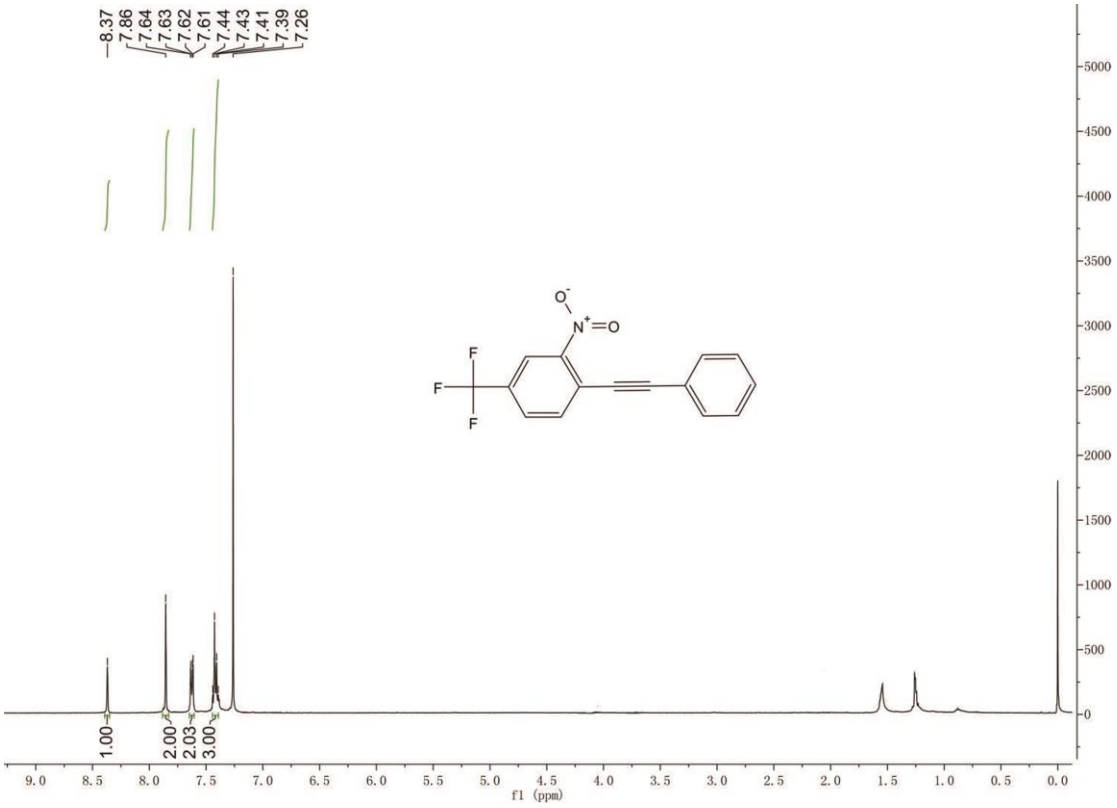
^{1}H NMR of **30**



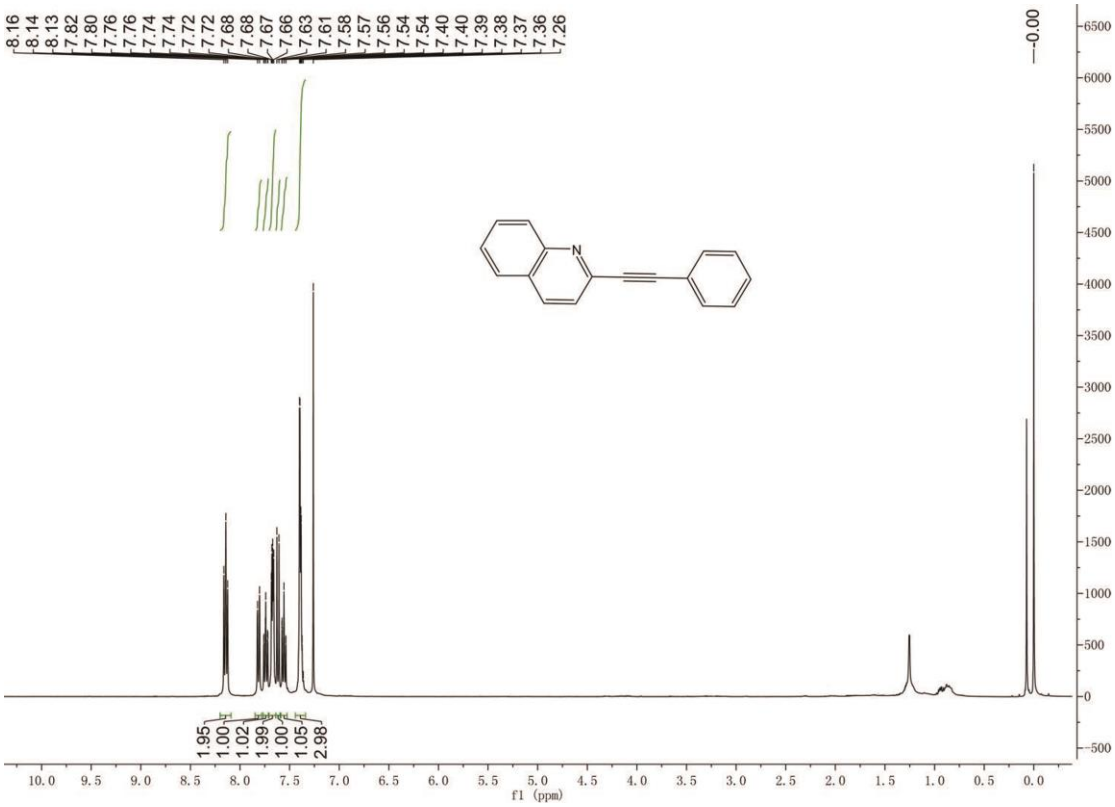
^{1}H NMR of **31**



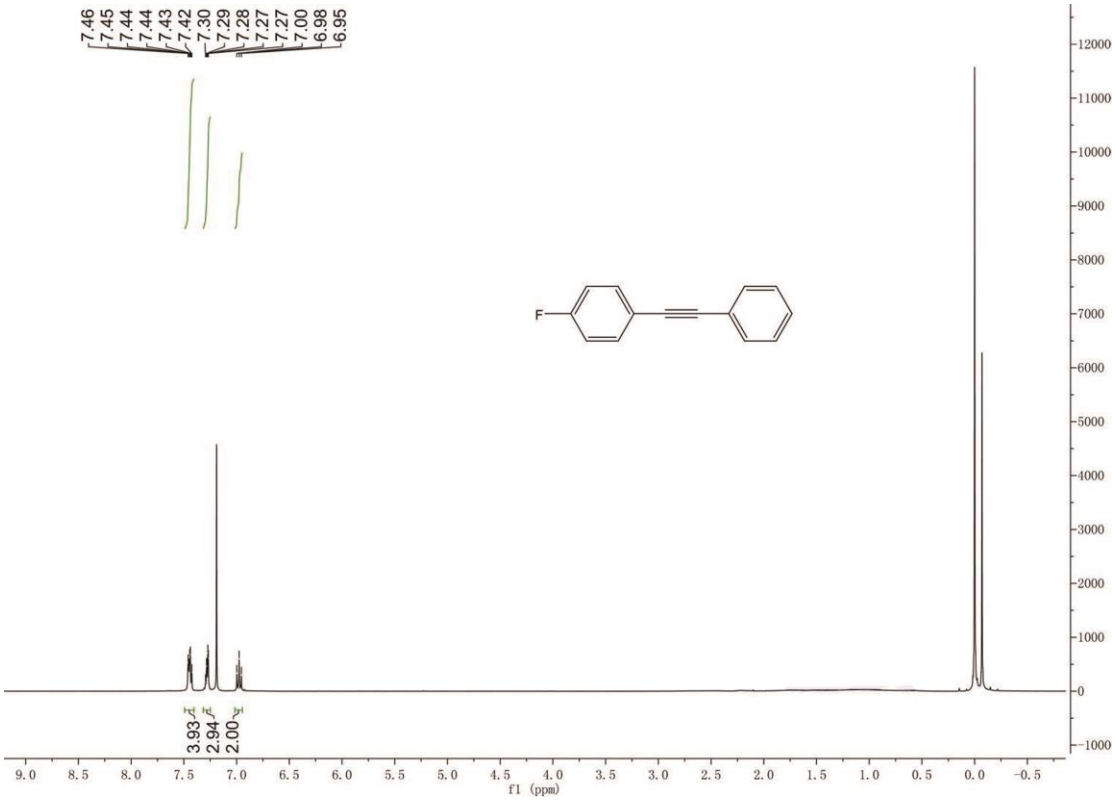
^{1}H NMR of **32**



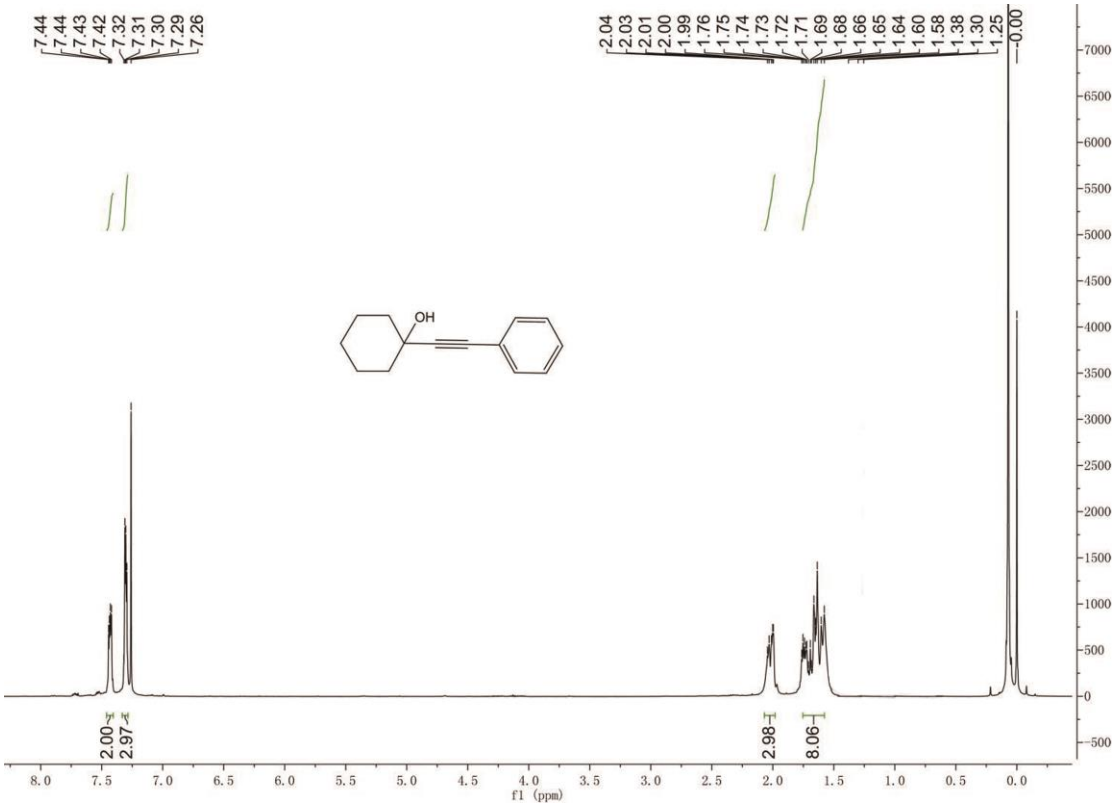
^{1}H NMR of **33**



^{1}H NMR of **34**



^{1}H NMR of **35**



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