Supporting Information

Nickel-Catalyzed Sonogashira Coupling Reactions of Nonactivated Alkyl Chlorides under Mild Conditions

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1 NMR Spectra of Complexes 1 and 2

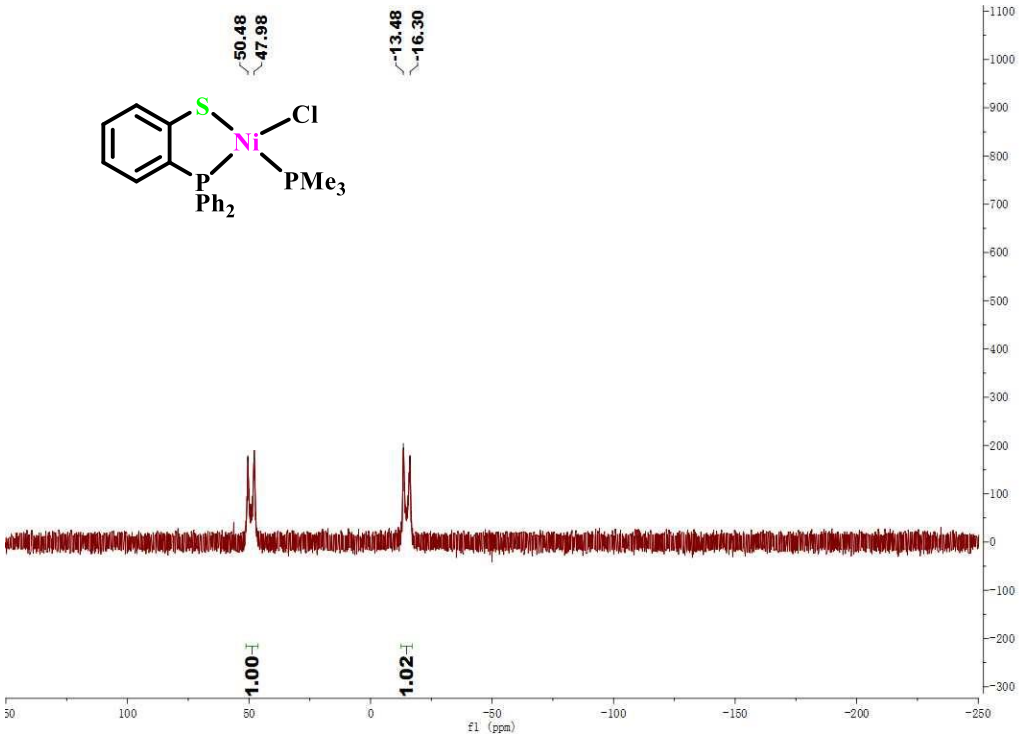


Figure S1. ^{31}P NMR spectrum of Complex **1** (C_6D_6)

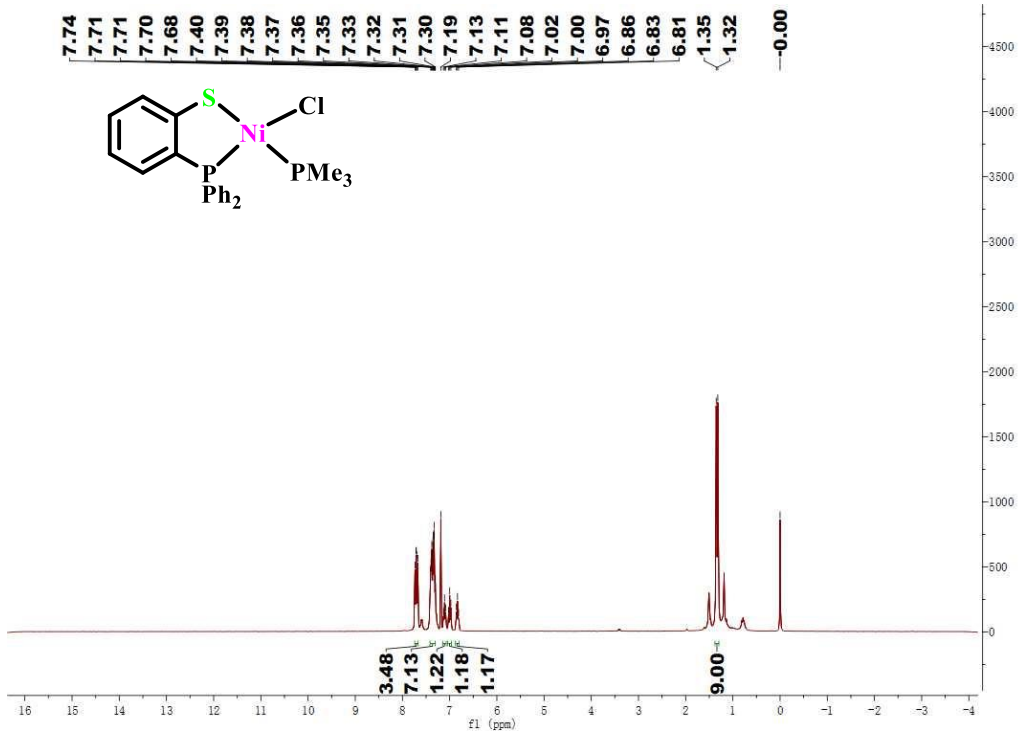


Figure S2. ^{1}H NMR spectrum of Complex **1** (C_6D_6)

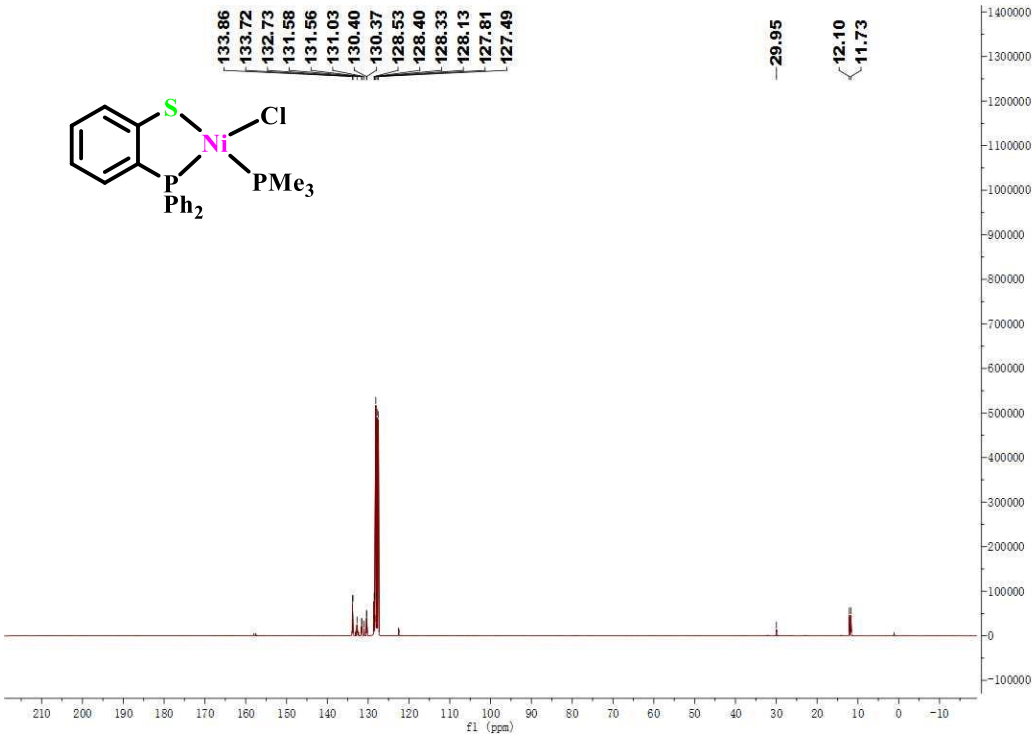


Figure S3. ^{13}C NMR spectrum of Complex **1** (C_6D_6)

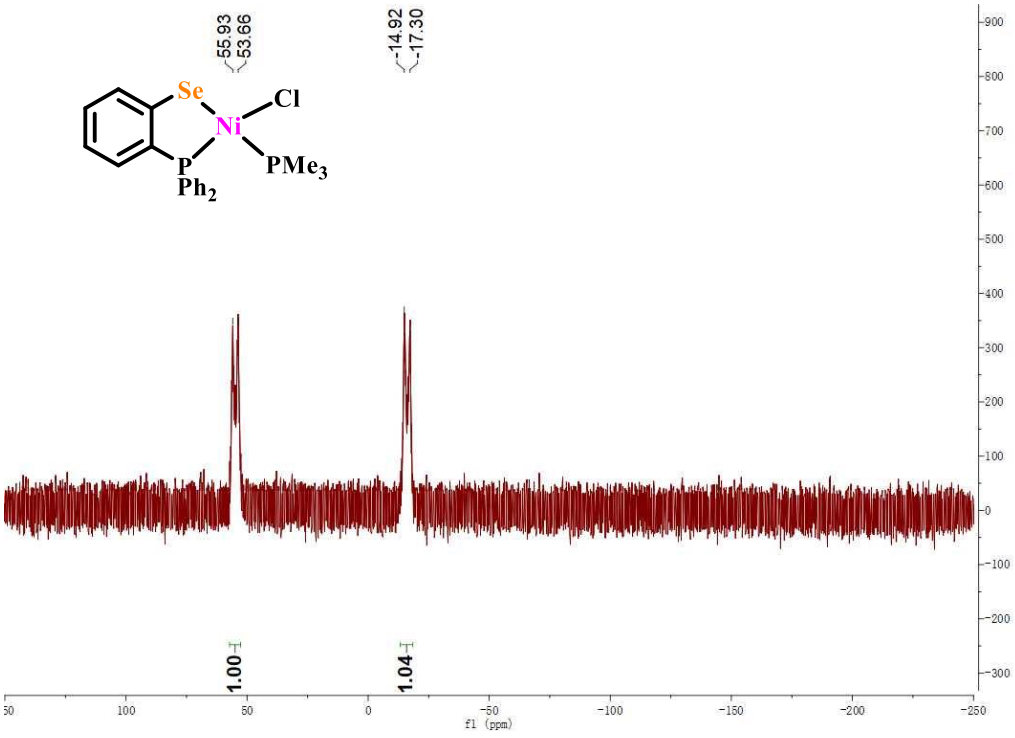


Figure S4. ^{31}P NMR spectrum of Complex **2** (C_6D_6)

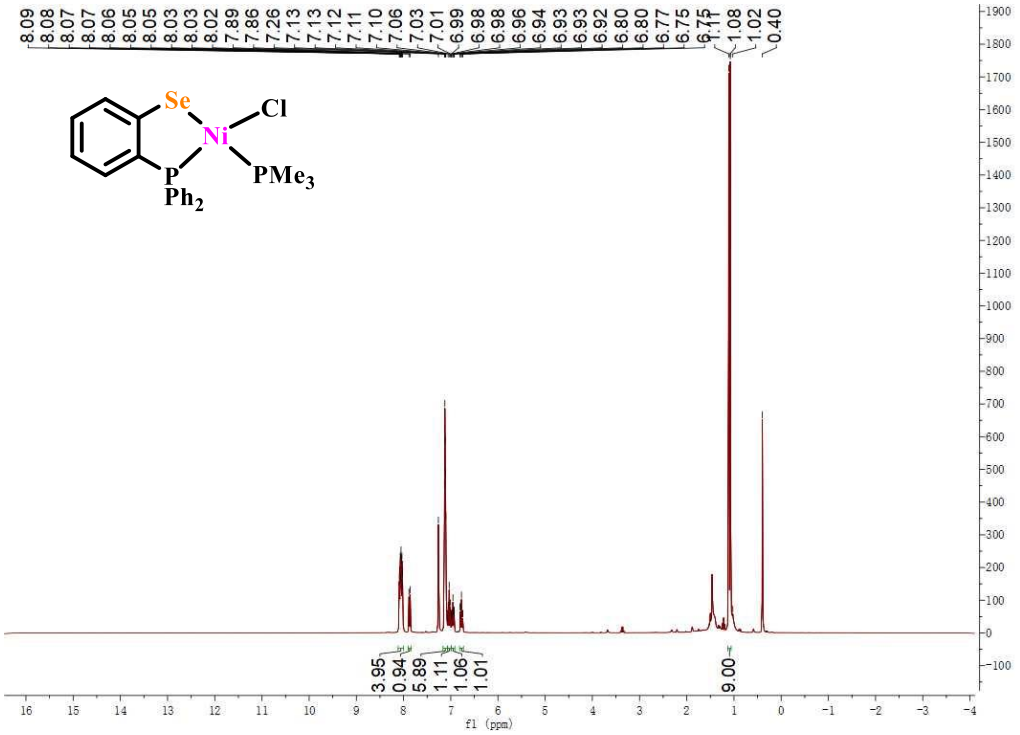


Figure S5. ^{1}H NMR spectrum of Complex **2** (C_6D_6)

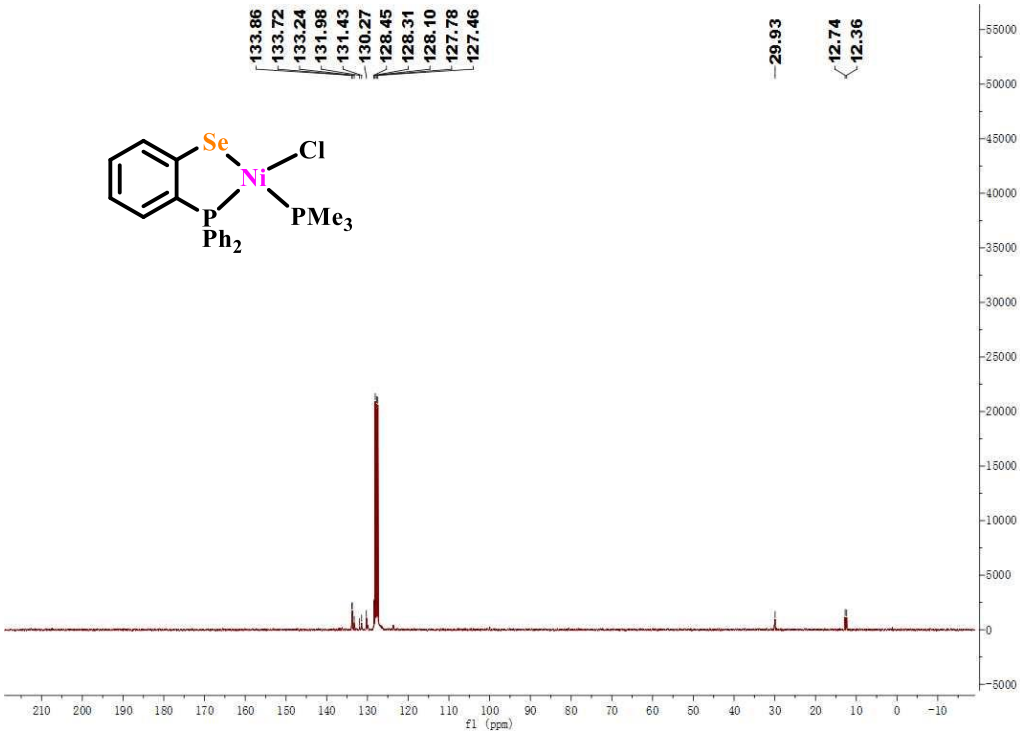
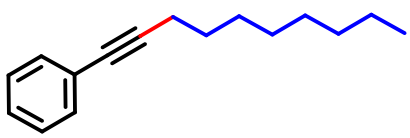


Figure S6. ^{13}C NMR spectrum of Complex **2** (C_6D_6)

2 ^{1}H and ^{13}C NMR of Sonongashira Coupling Products

(1) dec-1-yn-1-ylbenzene (2a)[1]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.32 – 7.29 (m, 2H), 7.18 – 7.16 (m, 3H), 2.31 (t, J = 7.0 Hz, 2H), 1.54 – 1.49 (m, 2H), 1.39 – 1.36 (m, 2H), 1.21 (m, 8H), 0.81 (d, J = 5.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 130.52, 127.12, 126.38, 123.15, 89.42, 79.56, 76.41, 75.59, 30.93, 30.85, 28.71, 28.21, 27.94, 21.66, 18.40, 13.08.

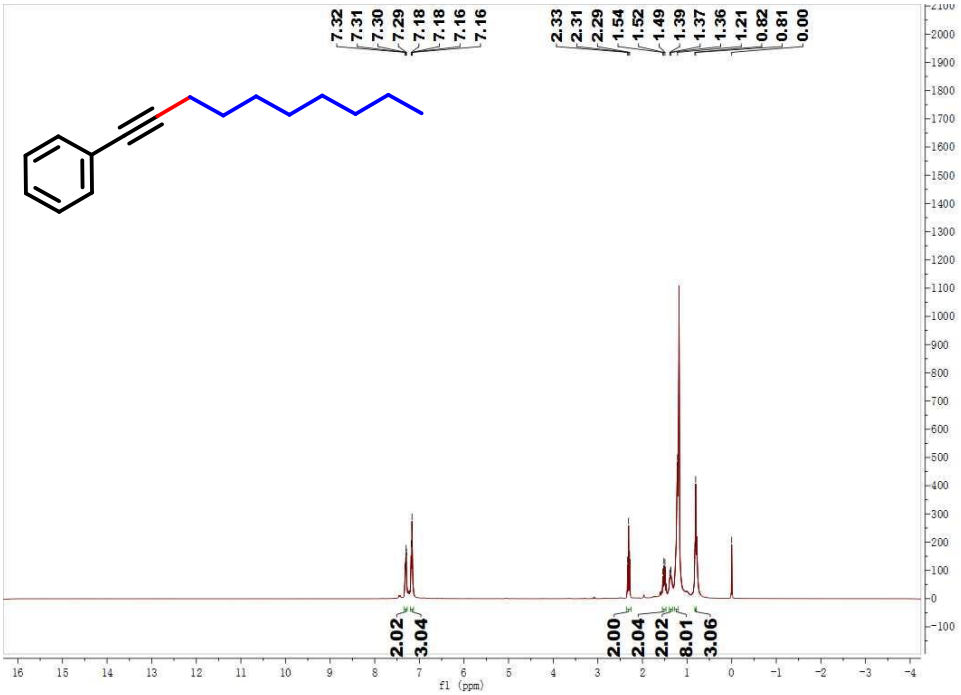


Figure S7. ^{1}H NMR spectrum of **2a** ($CDCl_3$)

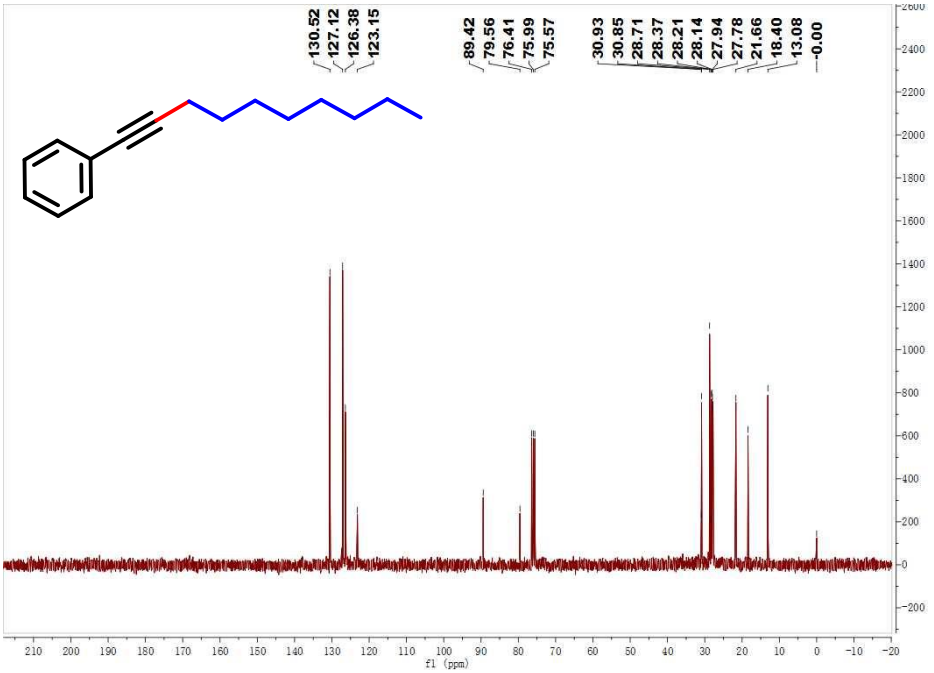
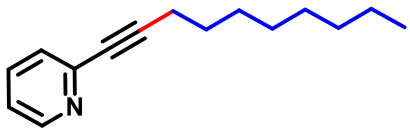


Figure S8. ^{13}C NMR spectrum of **2a** ($CDCl_3$)

(2) 2-(dec-1-yn-1-yl)pyridine (2b)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.46 (d, J = 4.4 Hz, 1H), 7.52 (td, J = 7.7, 1.1 Hz, 1H), 7.28 (d, J = 7.8 Hz, 1H), 7.15 - 7.04 (m, 1H), 2.36 (t, J = 7.1 Hz, 2H), 1.61 – 1.51 (m, 2H), 1.40 – 1.21 (m, 10H), 0.86 – 0.80 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.00, 143.30, 134.96, 125.95, 121.19, 90.14, 79.09, 76.14, 75.65, 30.84, 28.69, 27.40, 21.64, 18.34, 13.08

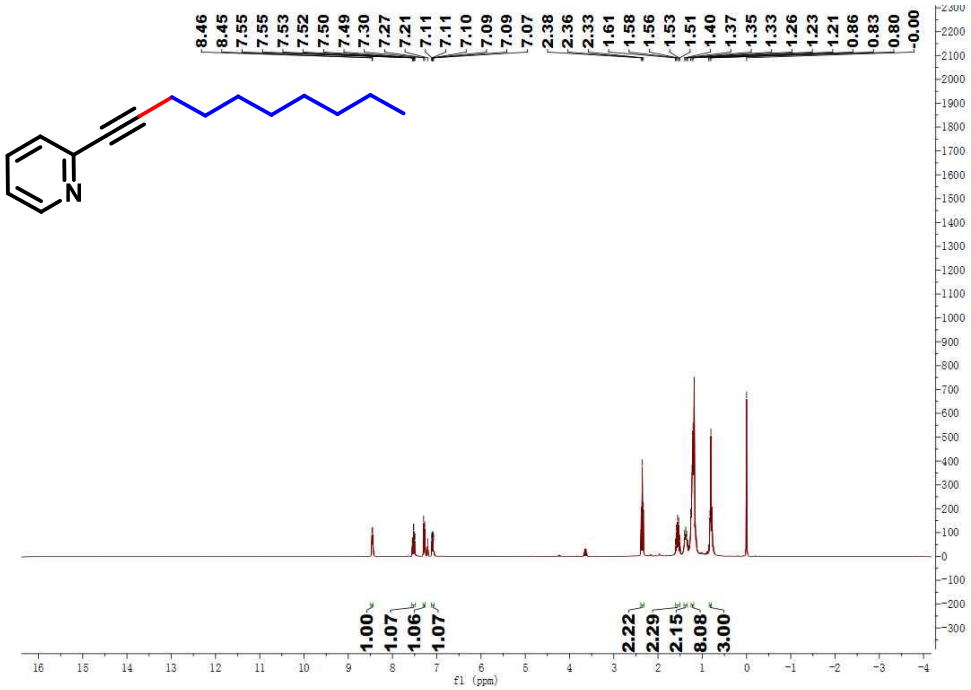


Figure S9. ^{1}H NMR spectrum of **2b** ($CDCl_3$)

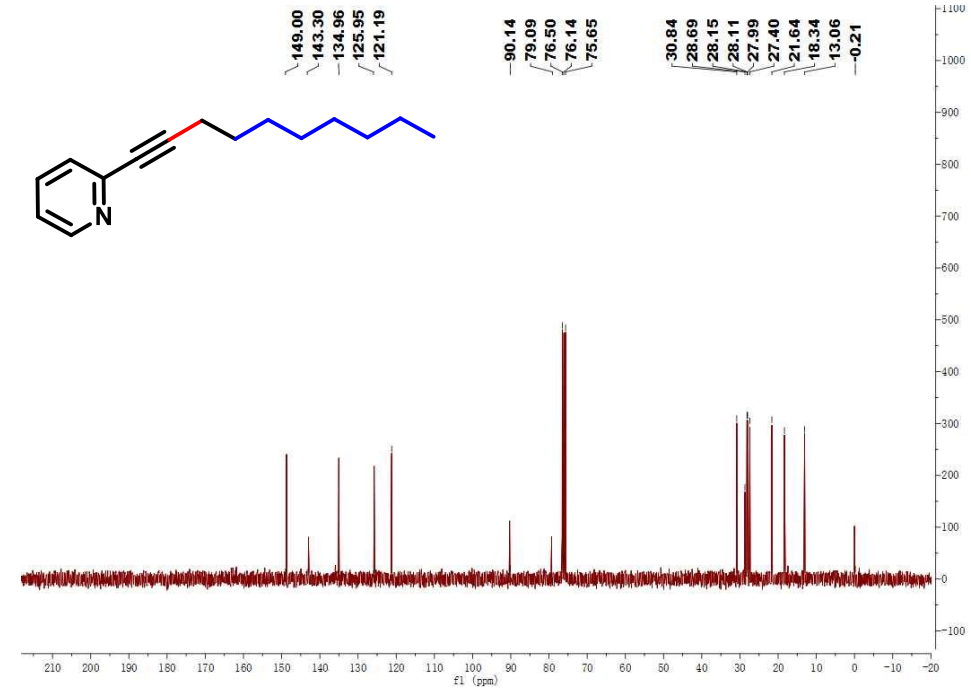
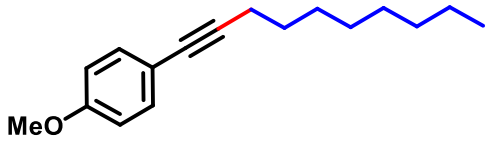


Figure S10. ^{13}C NMR spectrum of **2b** ($CDCl_3$)

(3) 1-(dec-1-yn-1-yl)-4-methoxybenzene (2c)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 – 7.22 (m, 2H), 6.73 – 6.70 (m, 2H), 3.69 (s, 3H), 2.29 (t, J = 7.0 Hz, 2H), 1.48 – 1.46 (m, 2H), 1.48 – 1.21 (m, 10H), 0.82 – 0.81 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.97, 132.21, 115.15, 112.83, 87.75, 79.24, 76.01, 54.25, 30.86, 28.22, 27.90, 21.82, 18.40, 13.14.

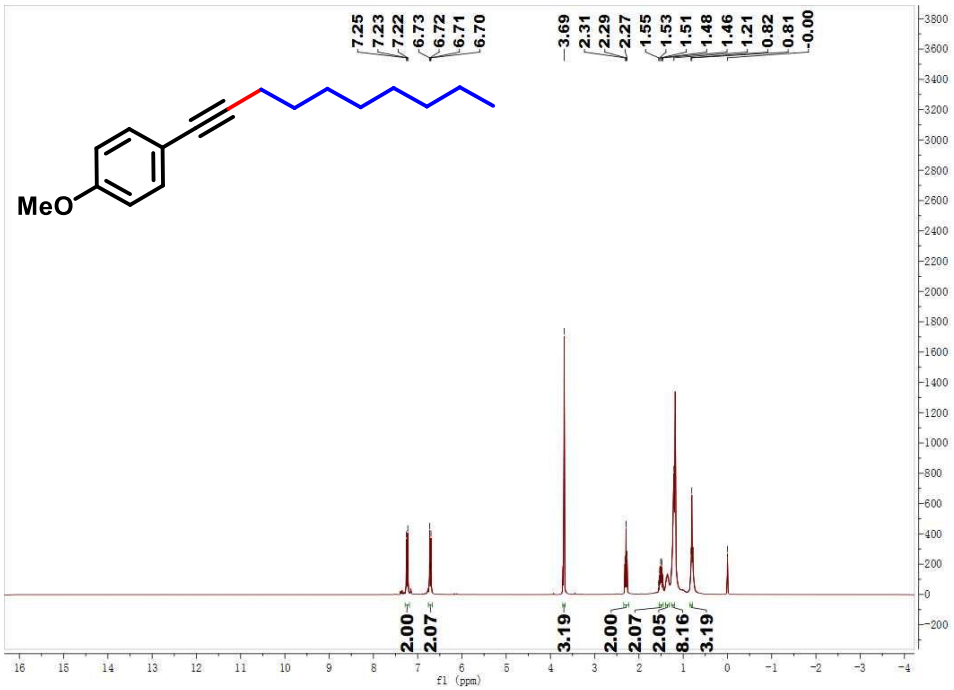


Figure S11. ^{1}H NMR spectrum of **2c** ($CDCl_3$)

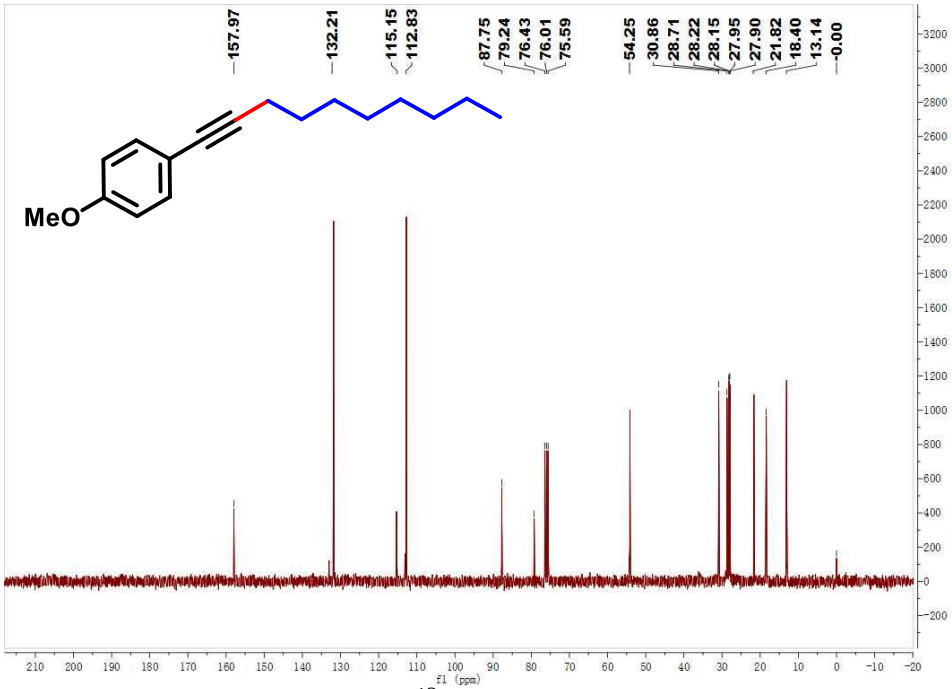
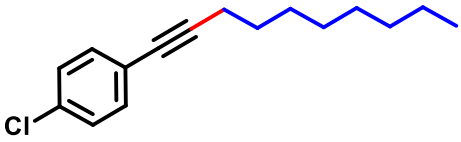


Figure S12. ^{13}C NMR spectrum of **2c** ($CDCl_3$)

(4) 1-chloro-4-(dec-1-yn-1-yl)benzene (2d)[2]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.20 (m, 2H), 7.15 – 7.12 (m, 2H), 2.29 (t, J = 7.0 Hz, 2H), 1.53 – 1.50 (m, 2H), 1.48 – 1.20 (m, 10H), 0.80 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.38, 131.72, 129.99, 128.10, 127.42, 121.70, 90.52, 78.46, 75.99, 30.84, 28.37, 28.19, 27.67, 21.69, 18.45, 13.08.

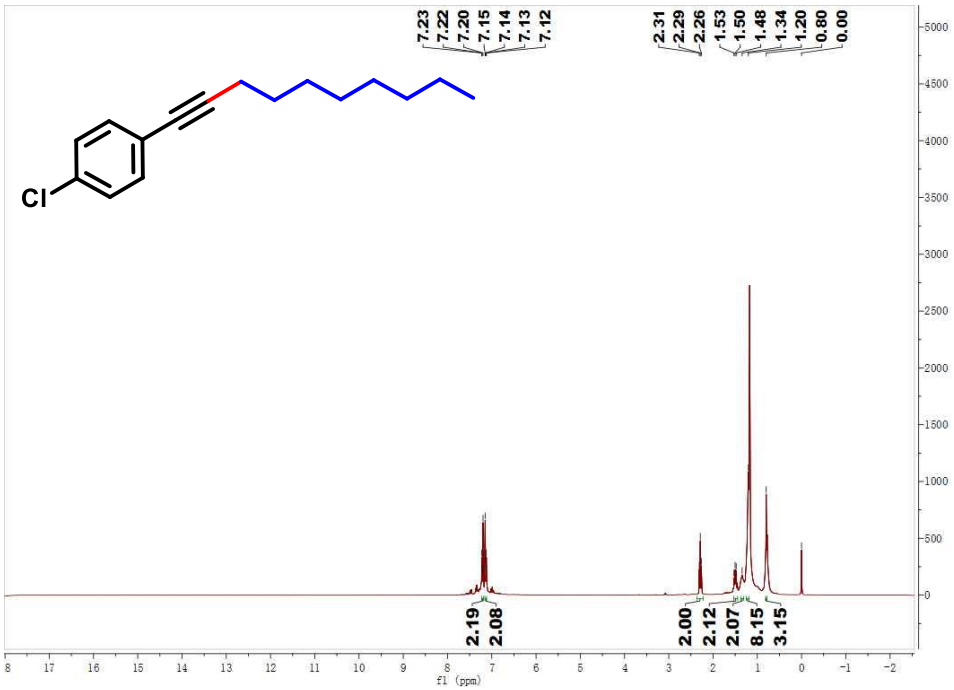


Figure S13. ^{1}H NMR spectrum of **2d** ($CDCl_3$)

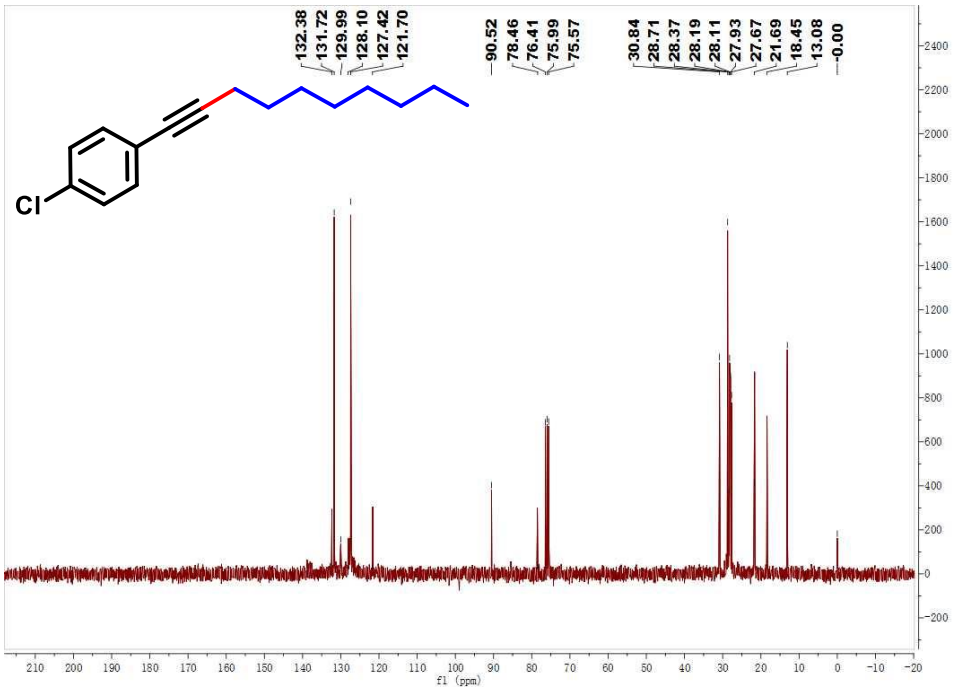
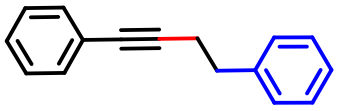


Figure S14. ^{13}C NMR spectrum of **2d** ($CDCl_3$)

(5) but-1-yne-1,4-diyldibenzene (2e)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50- 7.44 (m, 5H), 7.44 – 7.40 (m, 3H), 7.38- 7.29 (m, 2H), 3.64 (t, J = 7.6 Hz, 2H), 3.25 (t, J = 7.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.89, 132.19, 128.84, 128.71, 128.67, 128.37, 126.97, 122.21, 83.73, 77.55, 77.27, 77.12, 76.70, 39.47, 32.95.

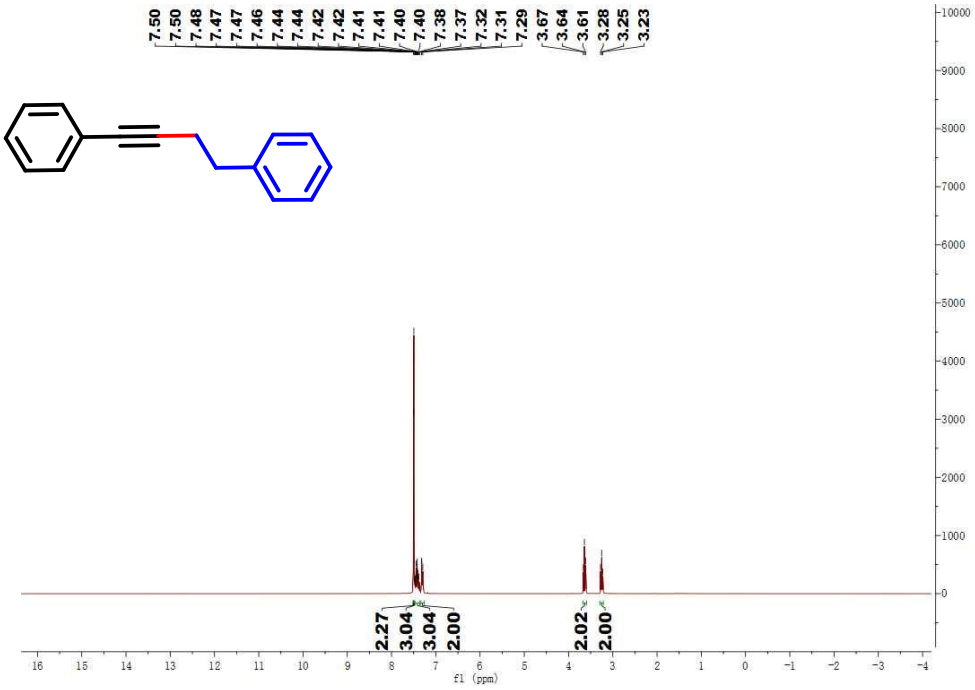


Figure S15. ^{1}H NMR spectrum of **2e** ($CDCl_3$)

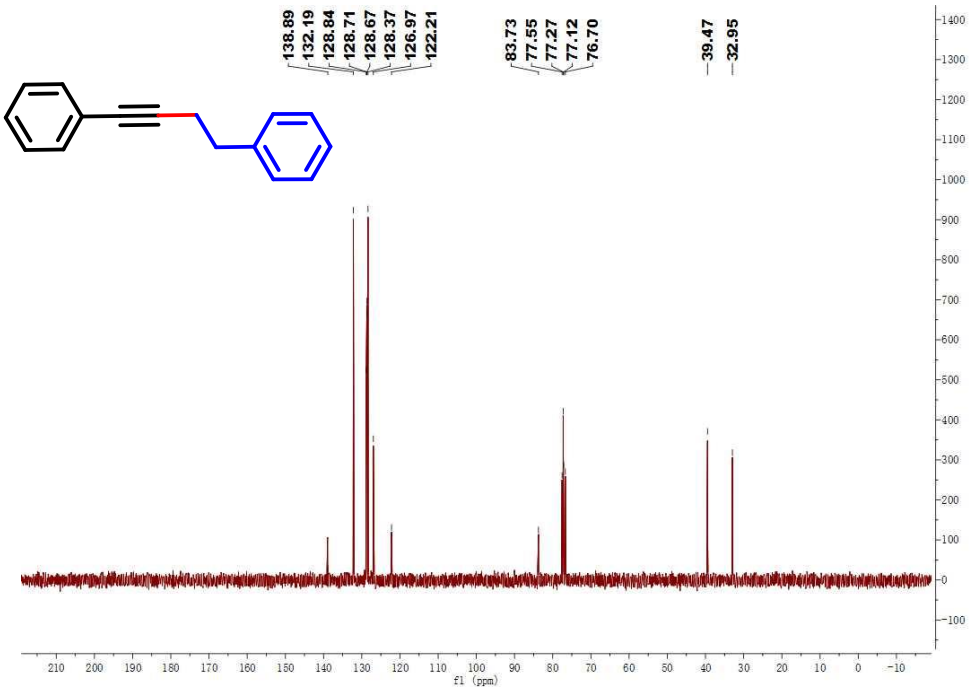
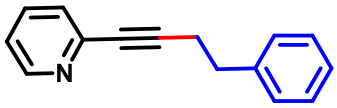


Figure S16. ^{13}C NMR spectrum of **2e** ($CDCl_3$)

(6) 2-(4-phenylbut-1-yn-1-yl)pyridine (2f)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 – 8.16 (m, 1H), 7.34 – 7.28 (m, 1H), 7.28 – 7.25 (m, 1H), 7.17 – 7.14 (m, 1H), 7.14 – 7.10 (m, 1H), 7.10 – 7.06 (m, 1H), 7.06 – 7.04 (m, 1H), 7.04 – 7.02 (m, 1H), 7.02 – 7.01 (m, 1H), 3.38 (t, J = 7.6 Hz, 2H), 2.97 (t, J = 7.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.01, 142.33, 138.85, 136.16, 128.63, 128.58, 127.46, 126.88, 123.41, 82.76, 77.60, 77.18, 76.75, 39.36, 32.94.

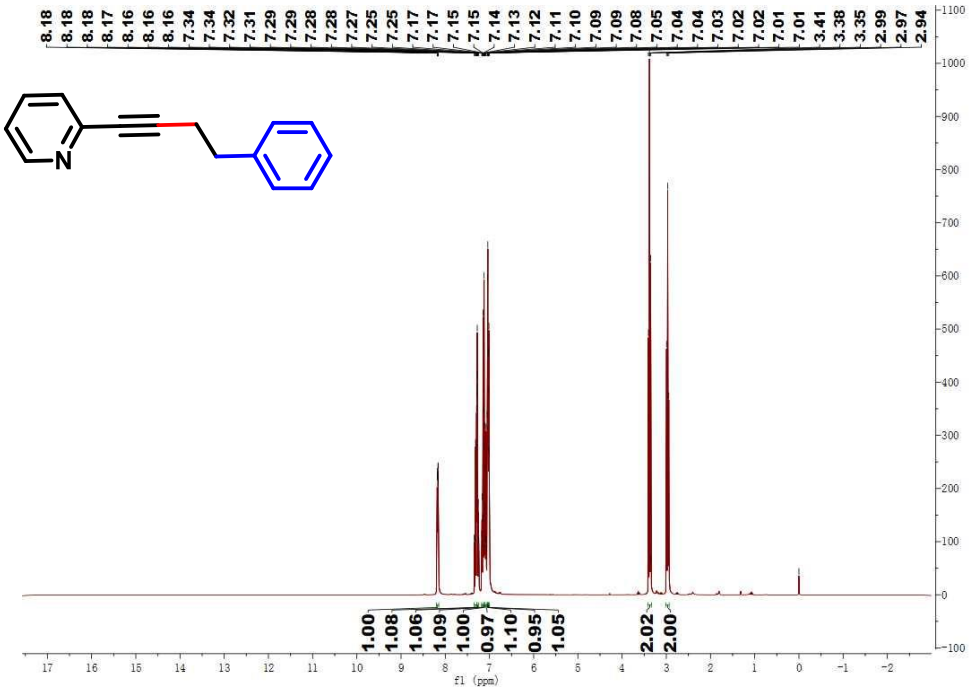


Figure S17. ^{1}H NMR spectrum of **2f** ($CDCl_3$)

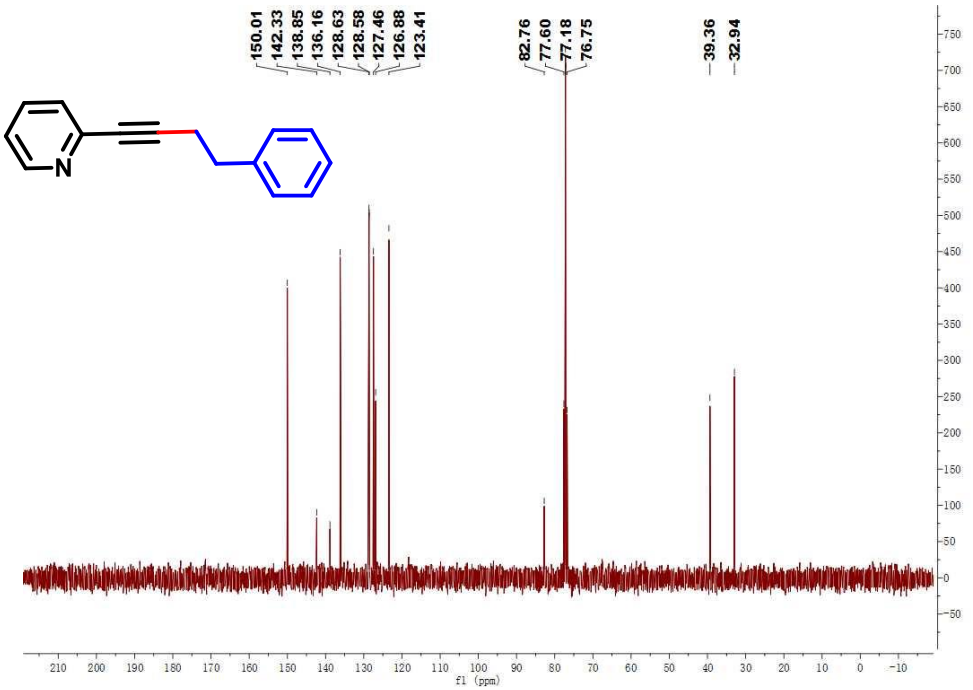
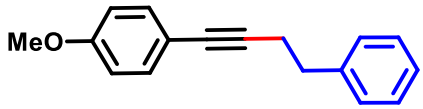


Figure S18. ^{13}C NMR spectrum of **2f** ($CDCl_3$)

(7) 1-methoxy-4-(4-phenylbut-1-yn-1-yl)benzene (2g)[3]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.28 (m, 2H), 7.25 – 7.23 (m, 2H), 7.23 – 7.21 (m, 1H), 7.13 – 7.11 (m, 2H), 6.70 – 6.67 (m, 2H), 3.64 (s, 3H), 3.46 (t, J = 7.6 Hz, 2H), 3.06 (t, J = 7.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.98, 138.93, 133.62, 128.68, 128.64, 126.95, 114.22, 113.98, 83.71, 77.53, 77.10, 76.68, 75.86, 55.30, 39.45, 32.94.

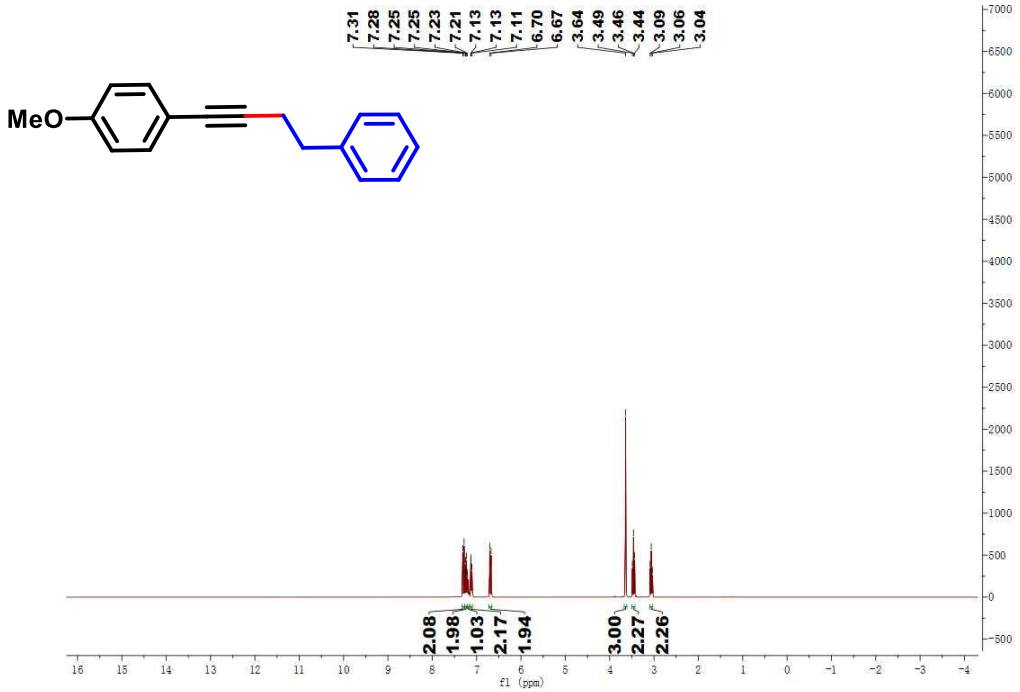


Figure S19. ^{1}H NMR spectrum of **2g** ($CDCl_3$)

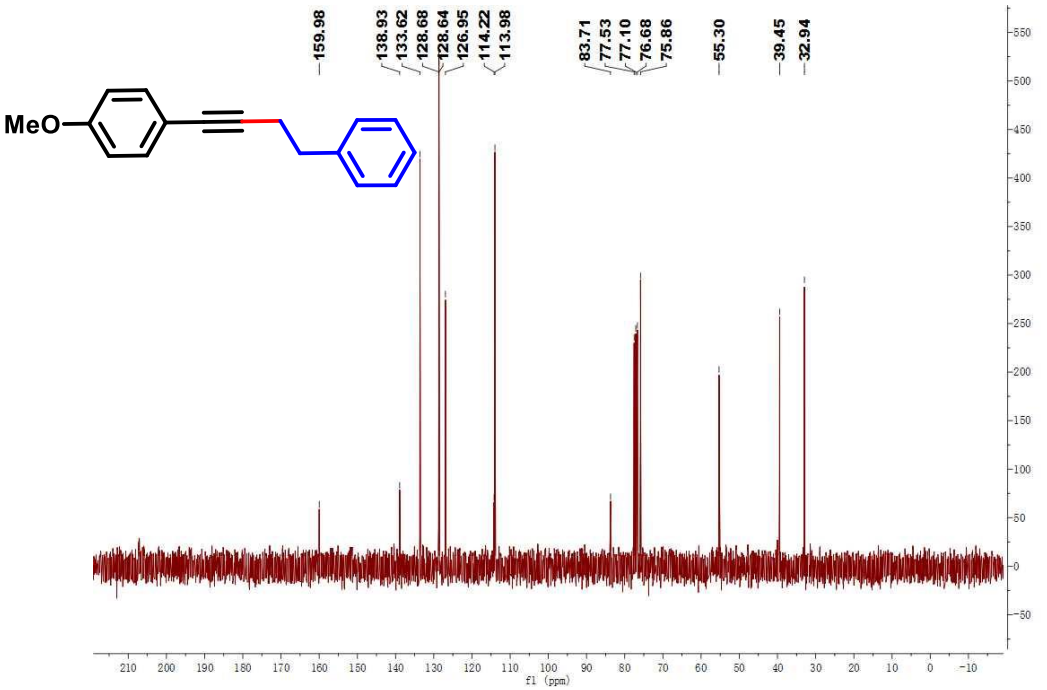
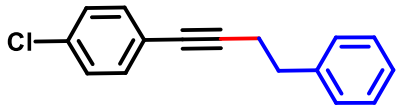


Figure S20. ^{13}C NMR spectrum of **2g** ($CDCl_3$)

(8) 1-chloro-4-(4-phenylbut-1-yn-1-yl)benzene (2h)[3]



^{1}H NMR (300 MHz, $CDCl_3$) 7.50- 7.48 (m, 4H), 7.48 – 7.37 (m, 3H), 7.31- 7.29 (m, 2H), 3.64 (t, J = 7.60 Hz, 2H), 3.25 (t, J = 7.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.95, 134.95, 133.41, 128.71, 128.67, 126.97, 120.74, 119.87, 82.58, 78.31, 77.56, 77.14, 76.71, 39.47, 32.96.

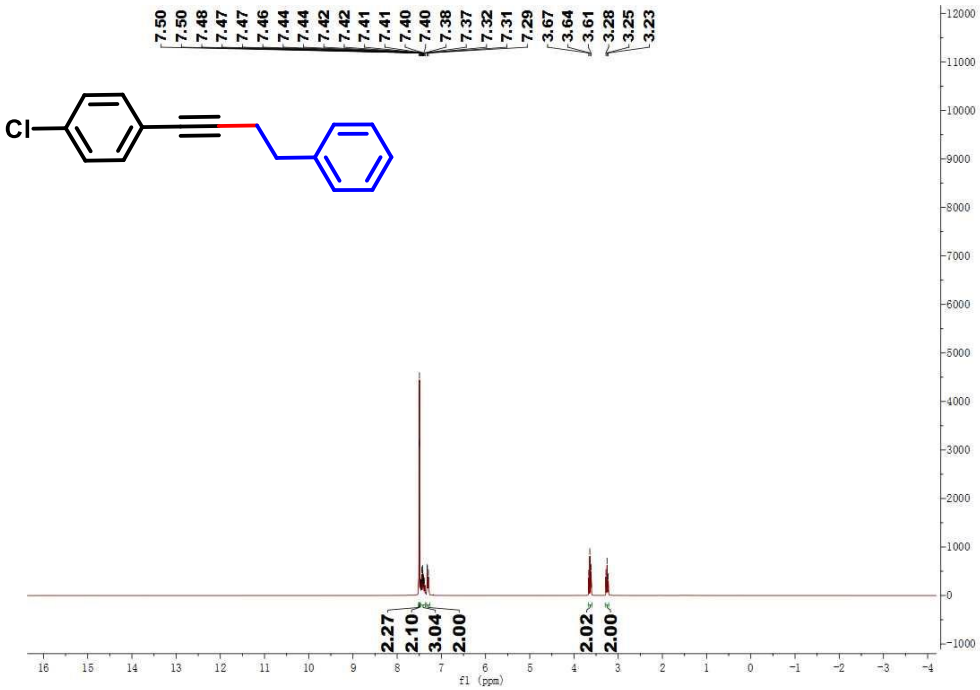


Figure S21. ^{1}H NMR spectrum of **2h** ($CDCl_3$)

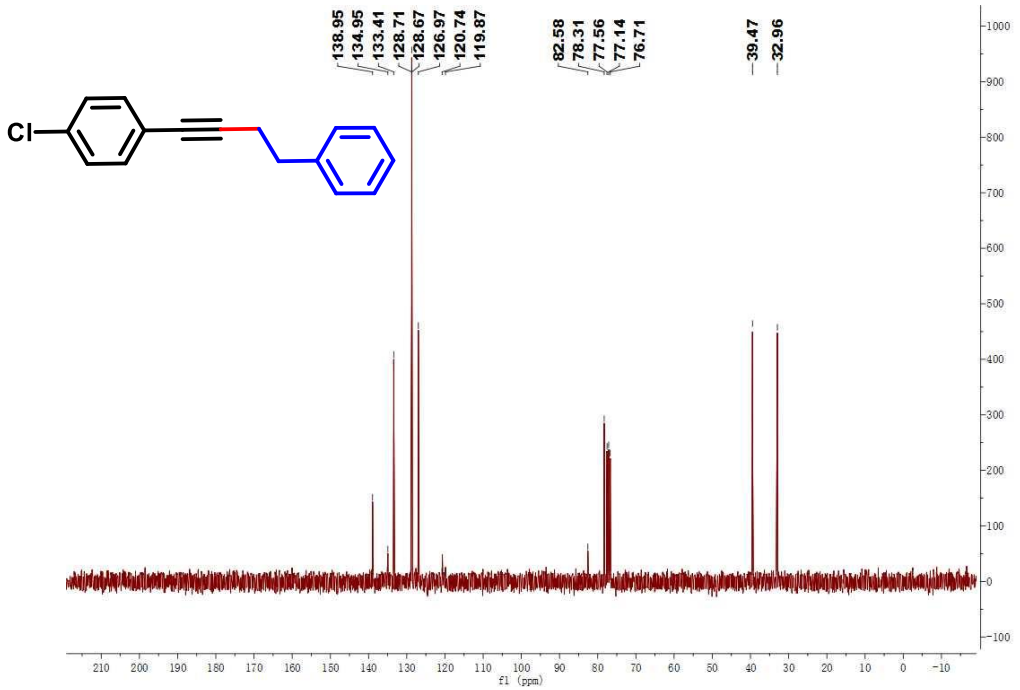
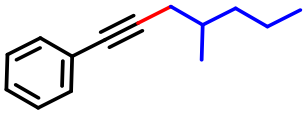


Figure S22. ^{13}C NMR spectrum of **2h** ($CDCl_3$)

(9) (4-methylhept-1-yn-1-yl)benzene (2i)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (s, 2H), 7.39 (s, 3H), 3.46 – 3.41 (m, 2H), 1.86 – 1.74 (m, 3H), 1.36 (s, 1H), 0.99 – 0.95 (m, 7H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 130.66, 127.31, 126.84, 120.69, 82.19, 76.01, 75.70, 75.58, 75.16, 40.24, 30.70, 25.37, 20.44.

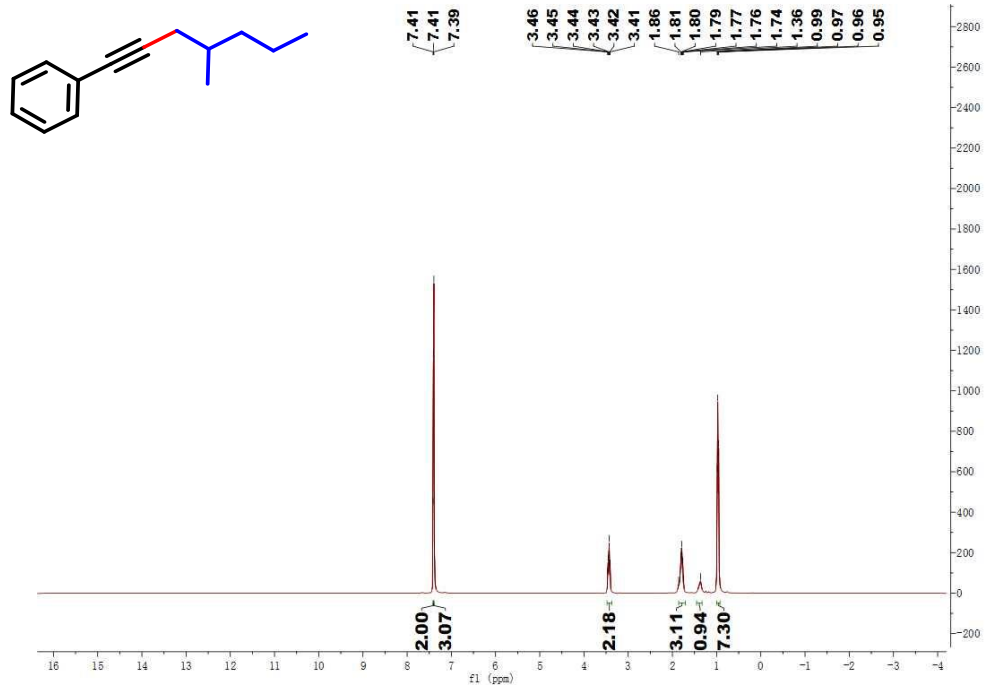


Figure S23. ^{1}H NMR spectrum of **2i** ($CDCl_3$)

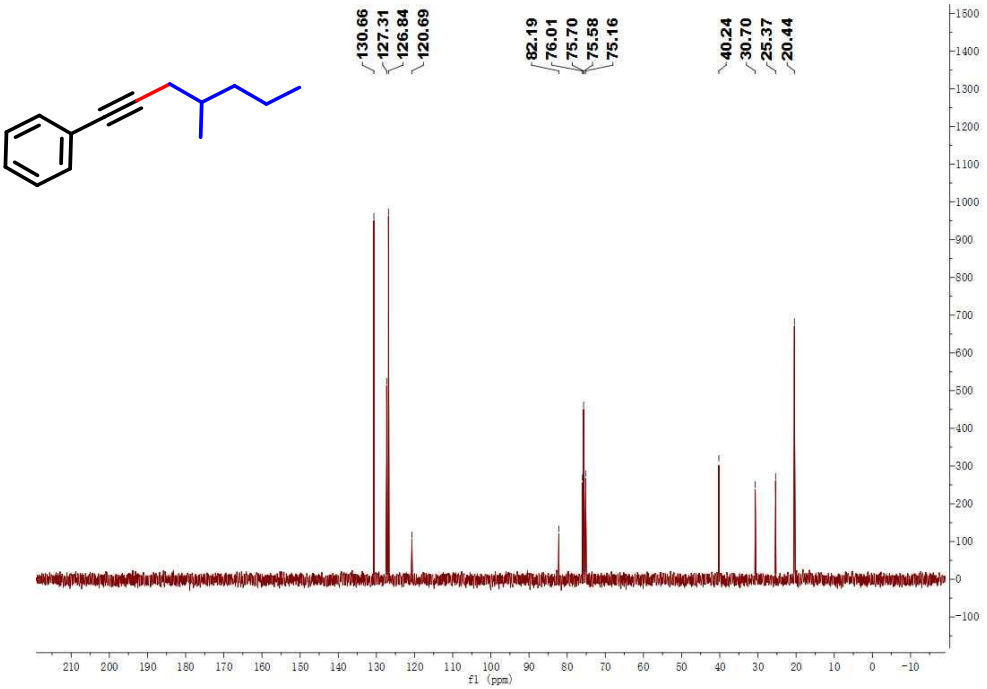
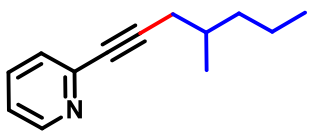


Figure S24. ^{13}C NMR spectrum of **2i** ($CDCl_3$)

(10) 2-(4-methylhept-1-yn-1-yl)pyridine (2j)



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.23 (m, 1H), 7.46 – 7.43 (m, 1H), 7.41 – 7.34 (m, 1H), 7.16 – 7.14 (m, 1H), 3.31 – 3.27 (m, 2H), 1.64 – 1.59 (m, 4H), 0.79 – 0.77 (m, 7H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.98, 142.32, 136.13, 127.42, 123.37, 82.70, 77.53, 77.11, 76.68, 41.63, 32.14, 26.76, 21.84.

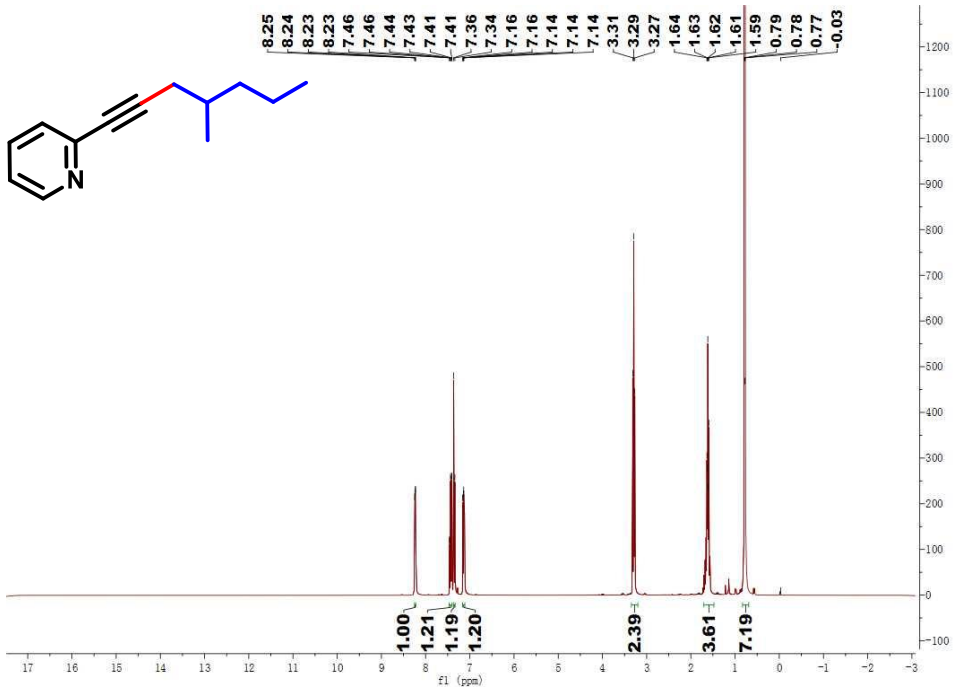


Figure S25. ^{1}H NMR spectrum of **2j** ($CDCl_3$)

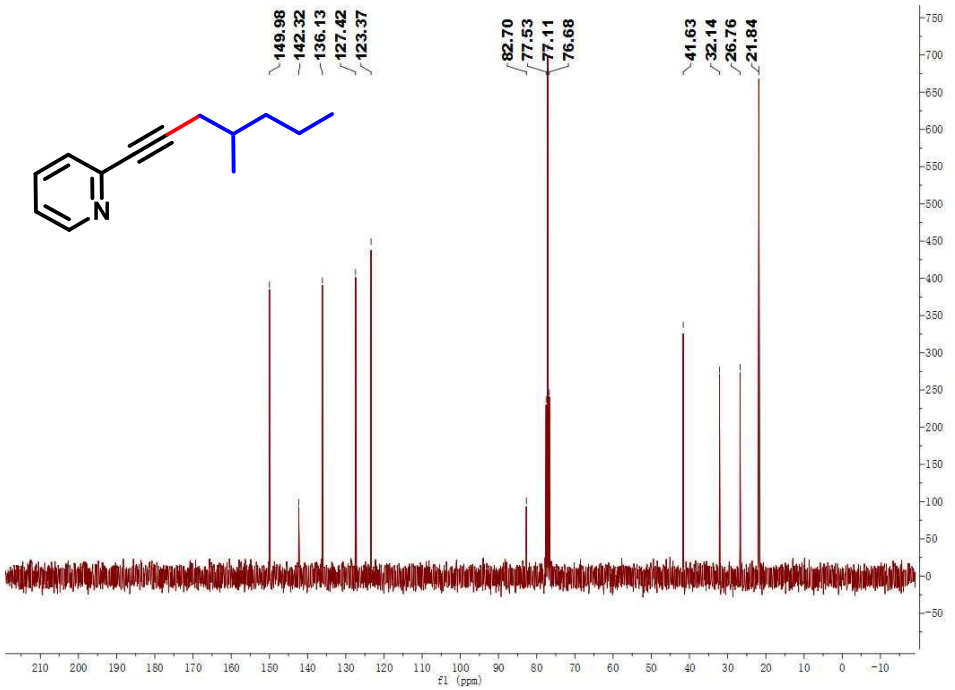
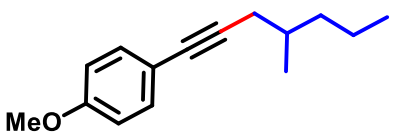


Figure S26. ^{13}C NMR spectrum of **2j** ($CDCl_3$)

(11) 1-methoxy-4-(4-methylhept-1-yn-1-yl)benzene (2k)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 - 7.22 (m, 2H), 6.73 – 6.70 (m, 2H), 3.70 (s, 3H), 2.33 – 2.28 (t, 2H), 1.67 – 1.65 (m, 1H), 1.42 – 1.40 (m, 2H), 1.20 – 1.17 (m, 2H), 0.86 – 0.83 (m, 3H), 0.83 – 0.77 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.95, 133.57, 114.20, 113.94, 83.66, 77.50, 77.08, 76.66, 75.80, 55.23, 41.70, 32.15, 26.83, 21.90.

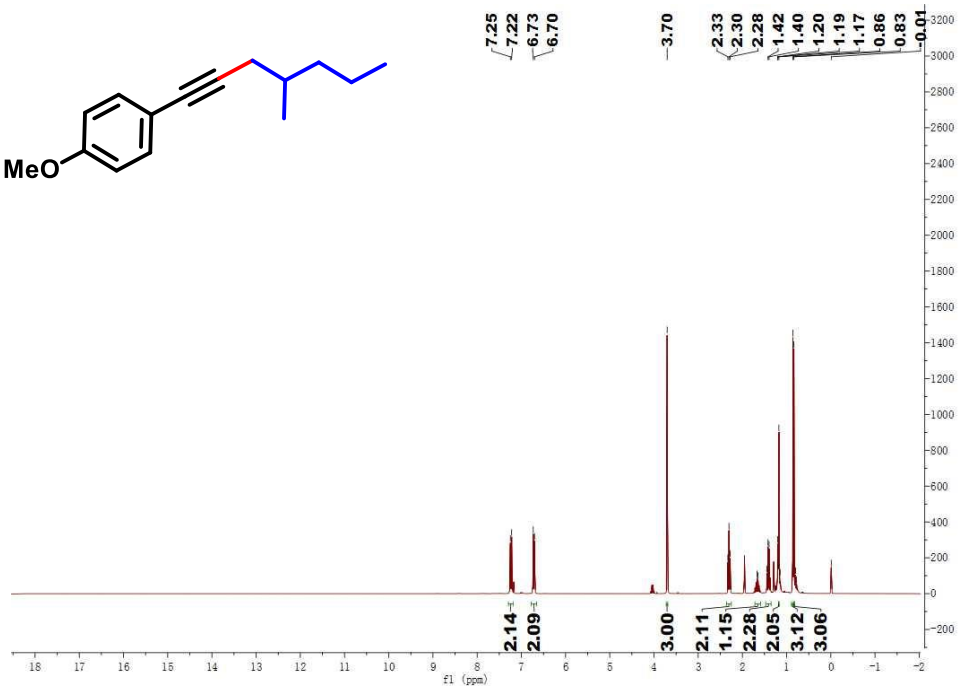


Figure S27. ^{1}H NMR spectrum of **2k** ($CDCl_3$)

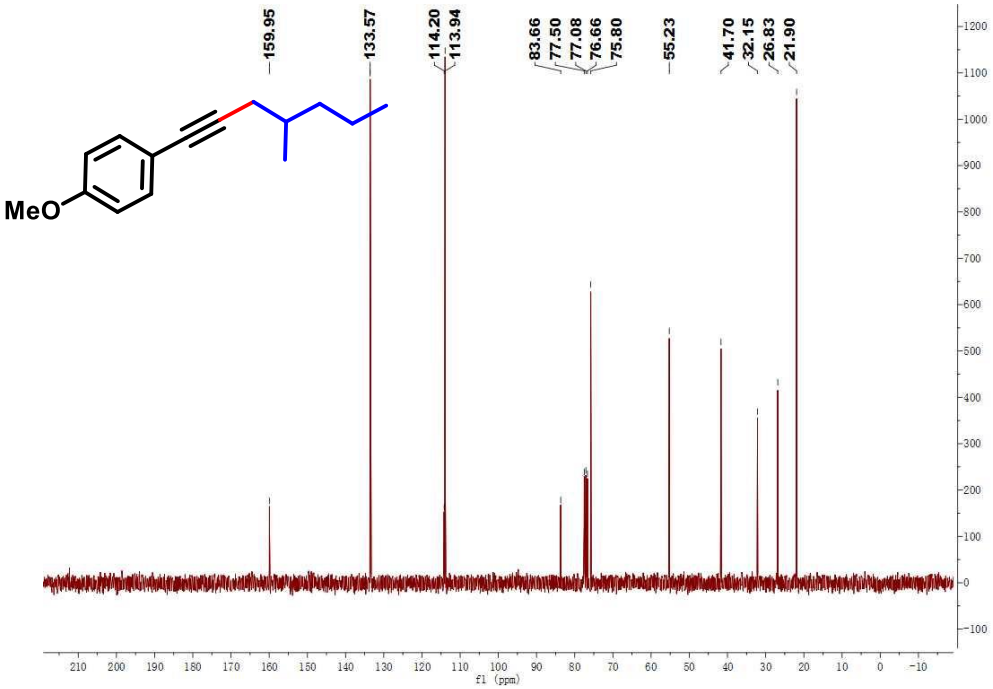
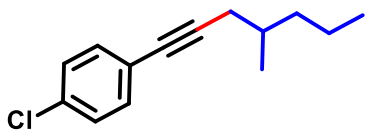


Figure S28. ^{13}C NMR spectrum of **2k** ($CDCl_3$)

(12) 1-chloro-4-(4-methylhept-1-yn-1-yl)benzene (2l)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (s, 2H)，7.39 (s, 2H), 3.46 – 3.41 (m, 2H), 1.86 – 1.74 (m, 3H), 1.36 (s, 1H), 0.99 – 0.95 (m, 7H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.89, 133.34, 128.66, 120.62, 82.50, 78.17, 77.47, 77.05, 76.62, 41.70, 32.12, 26.83, 21.89.

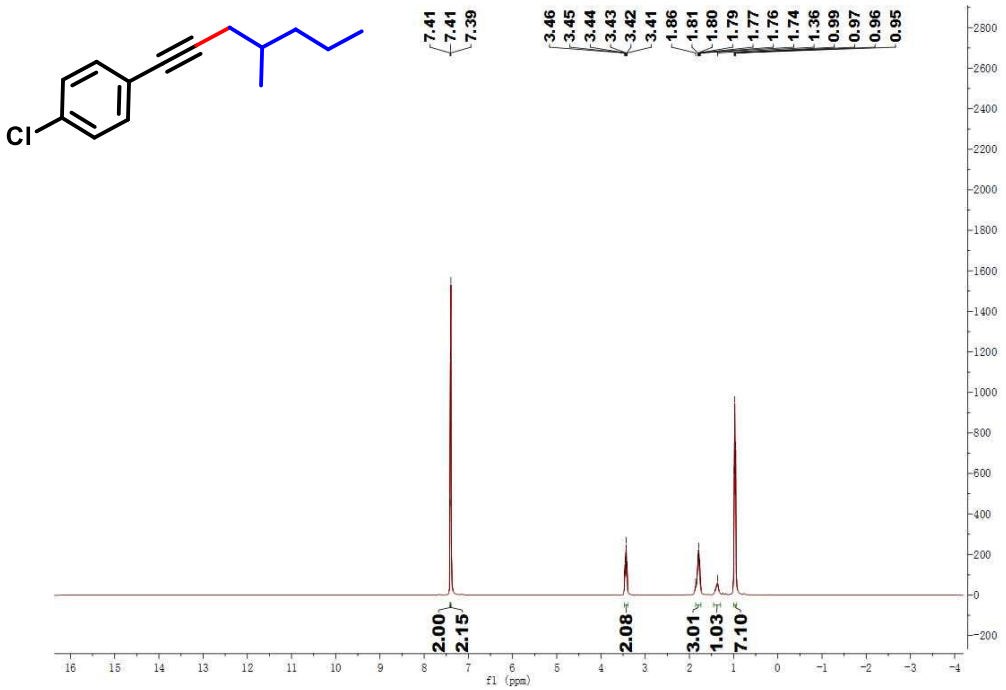


Figure S29. ^{1}H NMR spectrum of **2l** ($CDCl_3$)

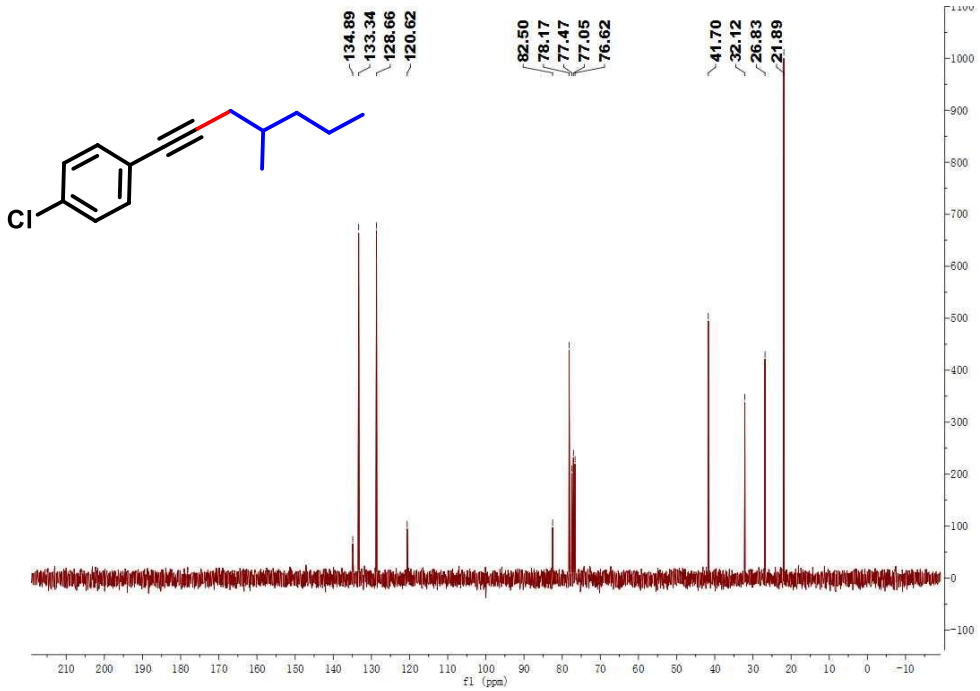
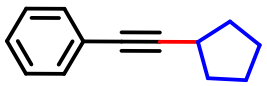


Figure S30. ^{13}C NMR spectrum of **2l** ($CDCl_3$)

(13)(cyclopentylethynyl)benzene(2m)[4]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (m, 5H), 4.30 – 4.27 (m, 1H), 1.94 – 1.90 (m, 3H), 1.78 – 1.66 (m, 2H), 1.60 – 0.97 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.13, 128.78, 128.31, 122.16, 83.66, 77.49, 77.17, 77.06, 76.64, 53.68, 37.87, 23.24.

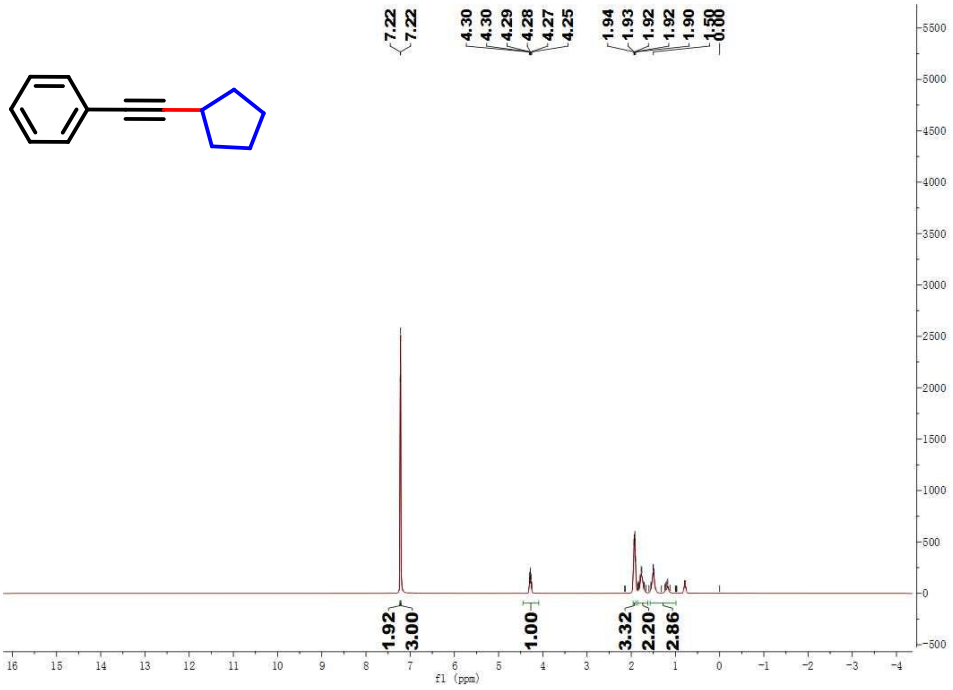


Figure S31. ^{1}H NMR spectrum of **2m** ($CDCl_3$)

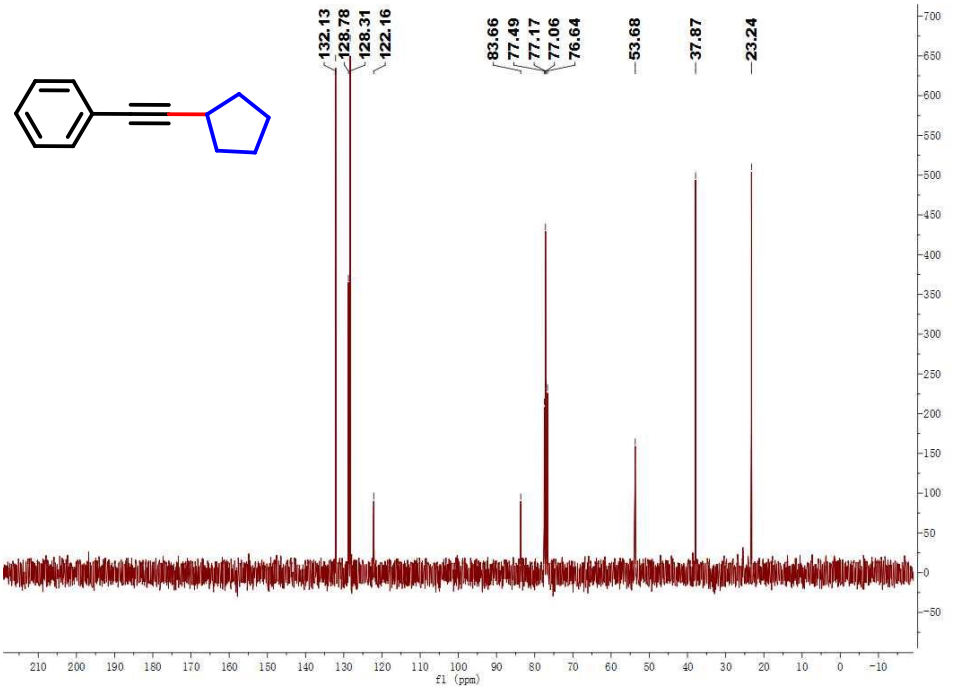


Figure S32. ^{13}C NMR spectrum of **2m** ($CDCl_3$)

(14) 2-(cyclopentylethynyl)pyridine (2n)[5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27 – 8.25 (m, 1H), 7.49 – 7.46 (m, 1H), 7.44 – 7.37 (m, 1H), 7.19 – 7.14 (m, 1H), 4.36 – 4.32 (m, 1H), 2.02 – 1.90 (m, 4H), 1.84 – 1.74 (m, 2H), 1.58 – 1.50 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.98, 142.30, 136.12, 127.41, 123.37, 82.71, 77.57, 77.14, 77.10, 76.72, 53.68, 37.79, 23.15.

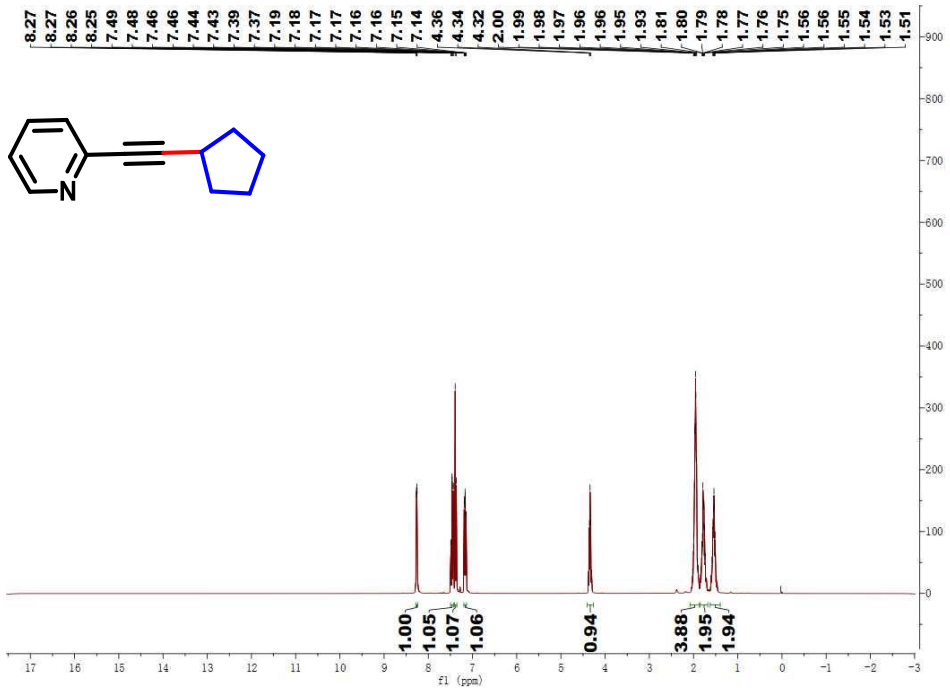


Figure S33. ^{1}H NMR spectrum of **2n** ($CDCl_3$)

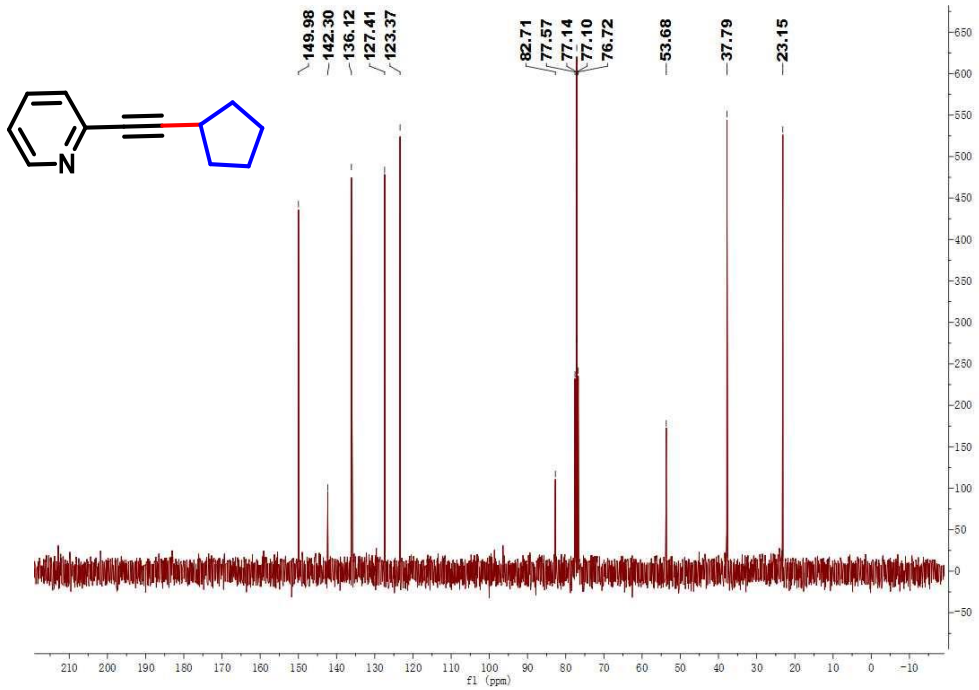
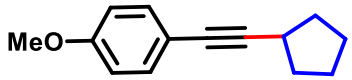


Figure S34. ^{13}C NMR spectrum of **2n** ($CDCl_3$)

(15) 1-(cyclopentylethynyl)-4-methoxybenzene (2o)[6]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 – 7.22 (m, 2H), 6.73 – 6.70 (m, 2H), 3.70 (s, 3H), 1.71 – 1.62 (m, 1H), 1.45 – 1.37 (m, 1H), 1.19 – 1.17 (m, 2H), 0.86 (s, 2H), 0.83 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.95, 133.56, 114.19, 113.94, 83.66, 77.53, 77.10, 76.68, 75.82, 55.24, 53.69, 37.86, 23.23.

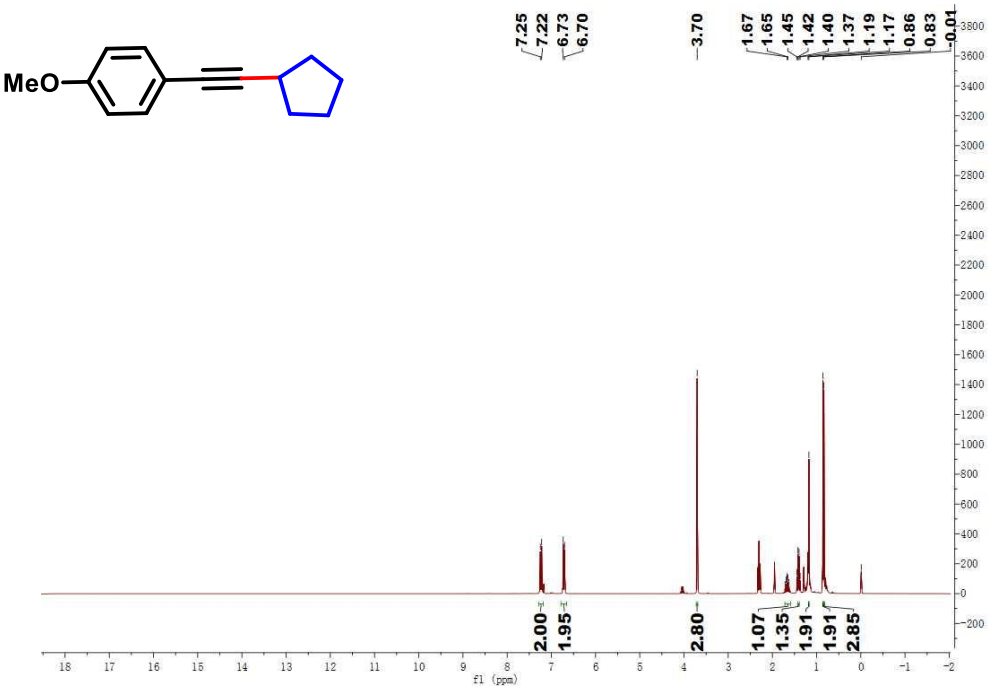


Figure S35. ^{1}H NMR spectrum of **2o** ($CDCl_3$)

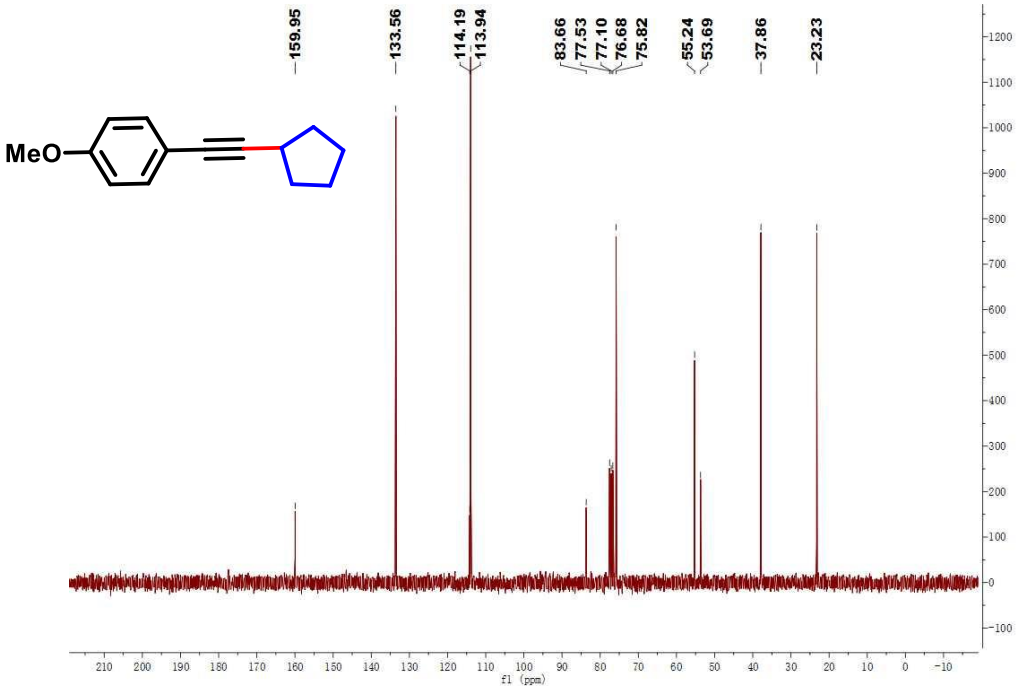
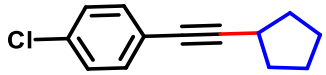


Figure S36. ^{13}C NMR spectrum of **2o** ($CDCl_3$)

(16) 1-chloro-4-(cyclopentylethynyl)benzene (2p)



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (s, 2H), 7.38 (s, 2H), 4.47 – 4.41 (m, 1H), 2.10 – 2.06 (m, 4H), 1.98 – 1.87 (m, 2H), 1.72 – 1.65 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.90, 133.34, 128.67, 120.61, 82.51, 78.18, 77.46, 77.04, 76.61, 53.67, 37.86, 23.23.

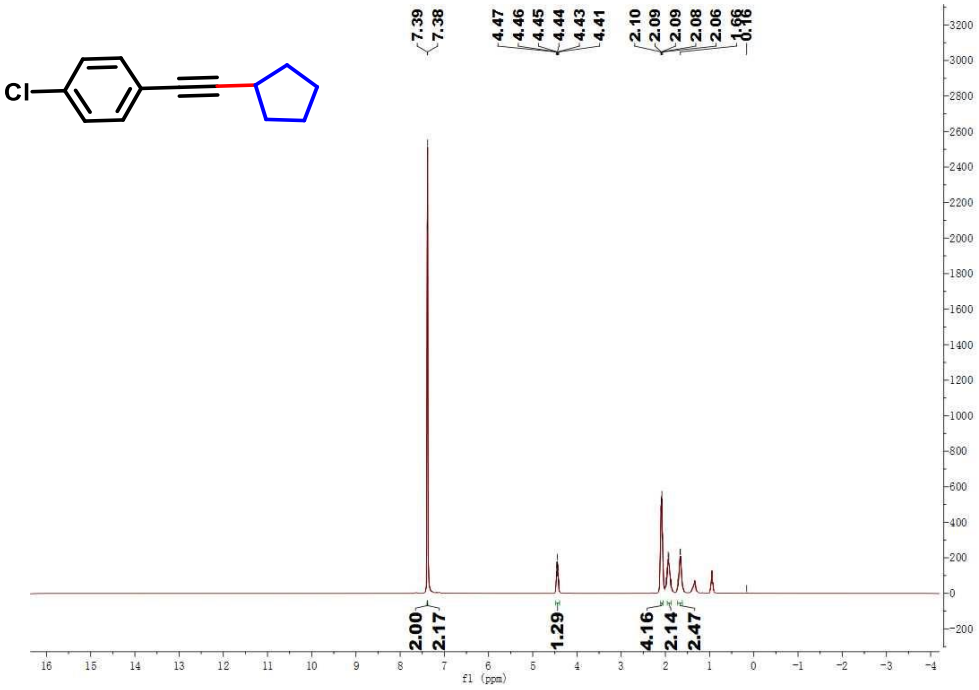


Figure S37. ^{1}H NMR spectrum of **2p** ($CDCl_3$)

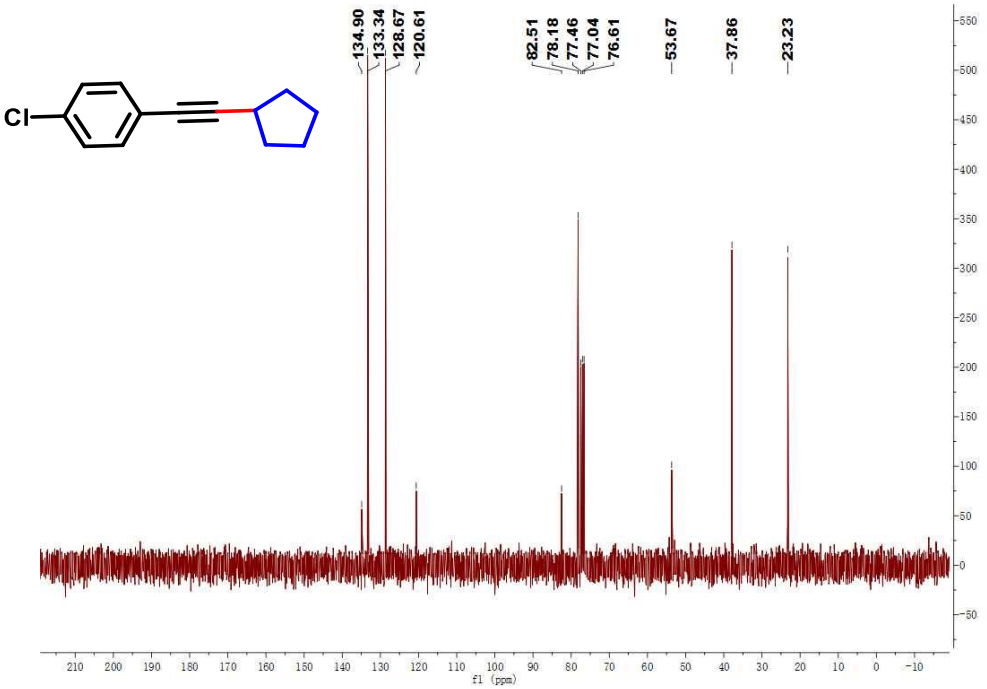
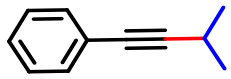


Figure S38. ^{13}C NMR spectrum of **2p** ($CDCl_3$)

(17)(3-methylbut-1-yn-1-yl)benzene (2q)[4]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.44 (s, 2H), 6.43 (s, 3H), 3.38 – 3.26 (m, 1H), 0.78 (d, J = 2.8 Hz, 3H), 0.76 (d, J = 2.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.13, 128.79, 128.32, 122.11, 83.67, 77.52, 77.21, 77.09, 76.67, 45.56, 28.49.

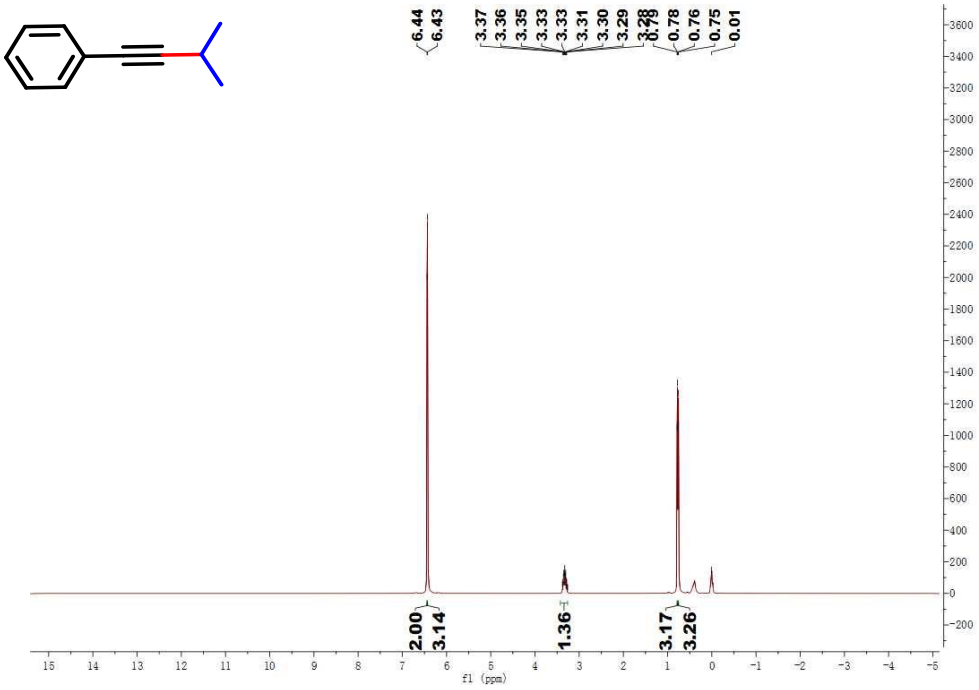


Figure S39. ^{1}H NMR spectrum of **2q** ($CDCl_3$)

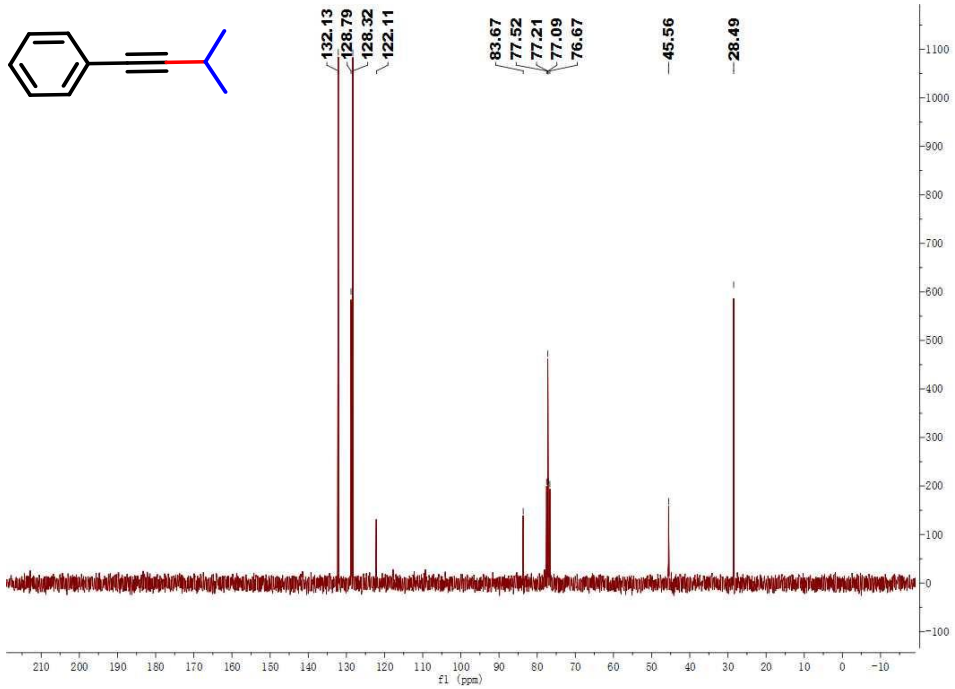
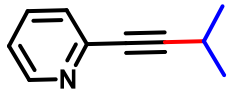


Figure S40. ^{13}C NMR spectrum of **2q** ($CDCl_3$)

(18) 2-(3-methylbut-1-yn-1-yl)pyridine (2r)[7]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.28 – 8.25 (m, 1H), 7.49 – 7.43 (m, 1H), 7.39 – 7.36 (m, 1H), 7.19 – 7.15 (m, 1H), 4.25 – 4.14 (m, 1H), 1.61 (d, J = 0.8 Hz, 3H), 1.58 (d, J = 0.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.96, 142.28, 136.11, 127.40, 123.36, 82.70, 77.17, 76.74, 45.53, 28.41.

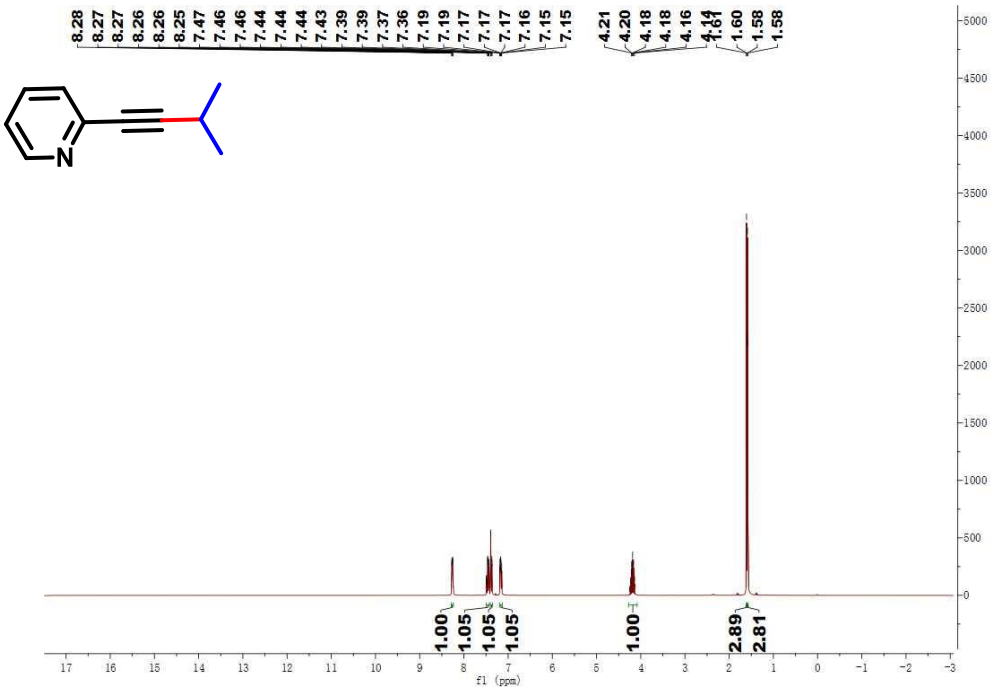


Figure S41. ^{1}H NMR spectrum of **2r** ($CDCl_3$)

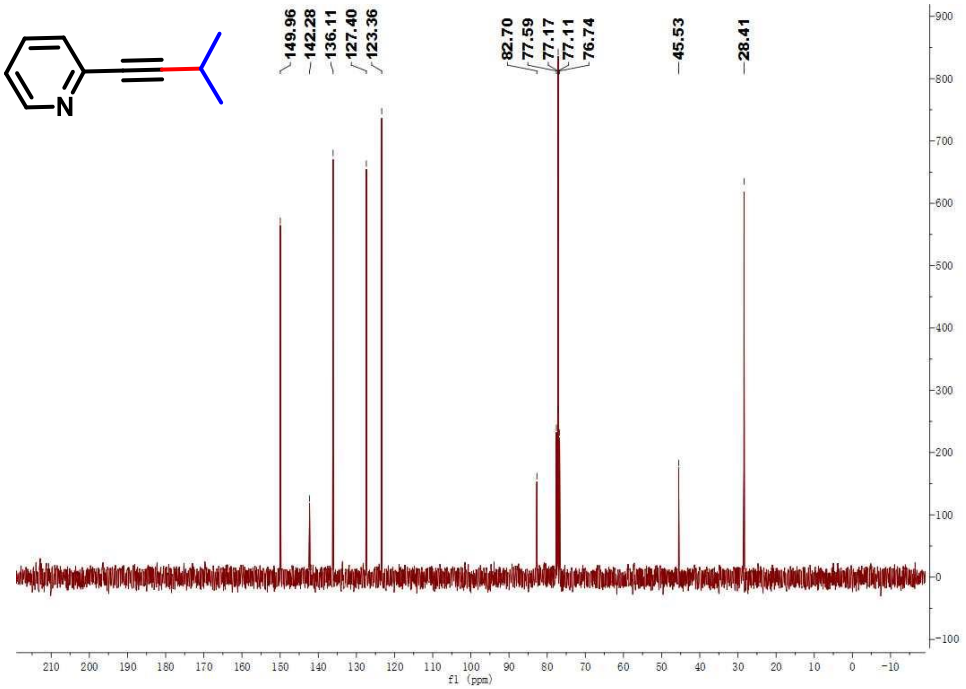
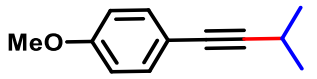


Figure S42. ^{13}C NMR spectrum of **2r** ($CDCl_3$)

(19) 1-methoxy-4-(3-methylbut-1-yn-1-yl)-benzene (2s)[8]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.23 (m, 2H), 6.67 – 6.64 (m, 2H), 4.07 (m, J = 6.5 Hz，1H), 3.64 (s, 3H), 1.41 (s, 3H), 1.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.95, 133.56, 114.18, 113.94, 83.65, 77.49, 77.07, 76.64, 75.79, 55.25, 45.53, 28.48.

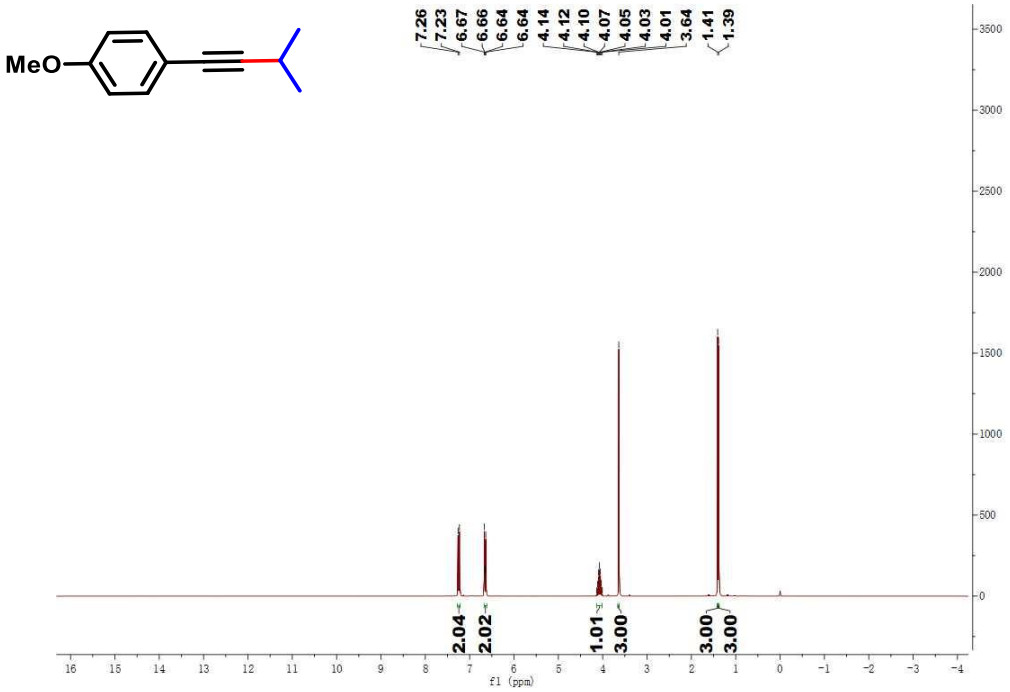


Figure S43. ^{1}H NMR spectrum of **2s** ($CDCl_3$)

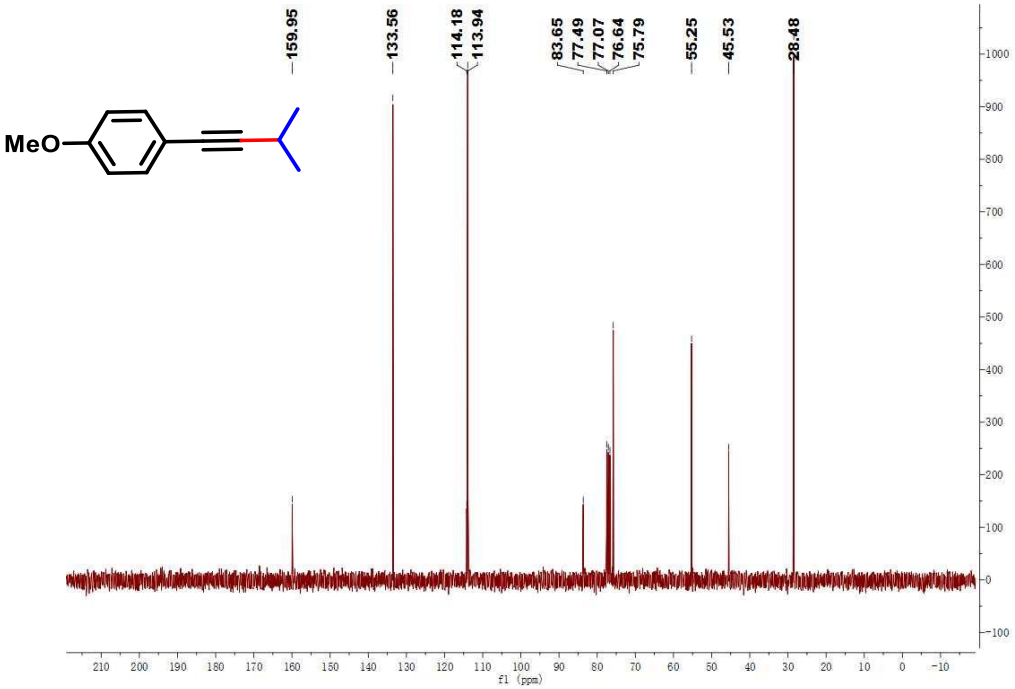
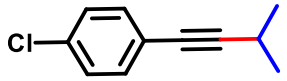


Figure S44. ^{13}C NMR spectrum of **2s** ($CDCl_3$)

(20) 1-chloro-4-(3-methylbut-1-yn-1-yl)benzene (2t)[9]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.43 (s, 2H), 6.42 (s, 2H), 3.39 – 3.25 (m, 1H), 0.77 (d, J = 2.8 Hz, 3H), 0.75 (d, J = 2.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.89, 133.34, 128.66, 120.61, 82.49, 78.17, 77.45, 77.03, 76.61, 45.51, 28.48.

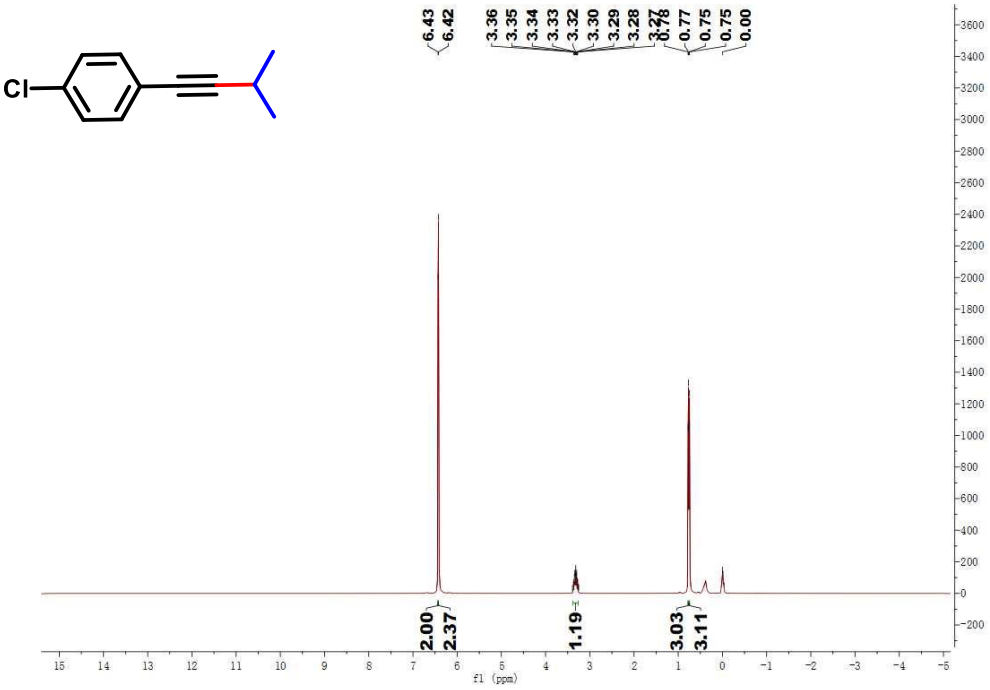


Figure S45. ^{1}H NMR spectrum of **2t** ($CDCl_3$)

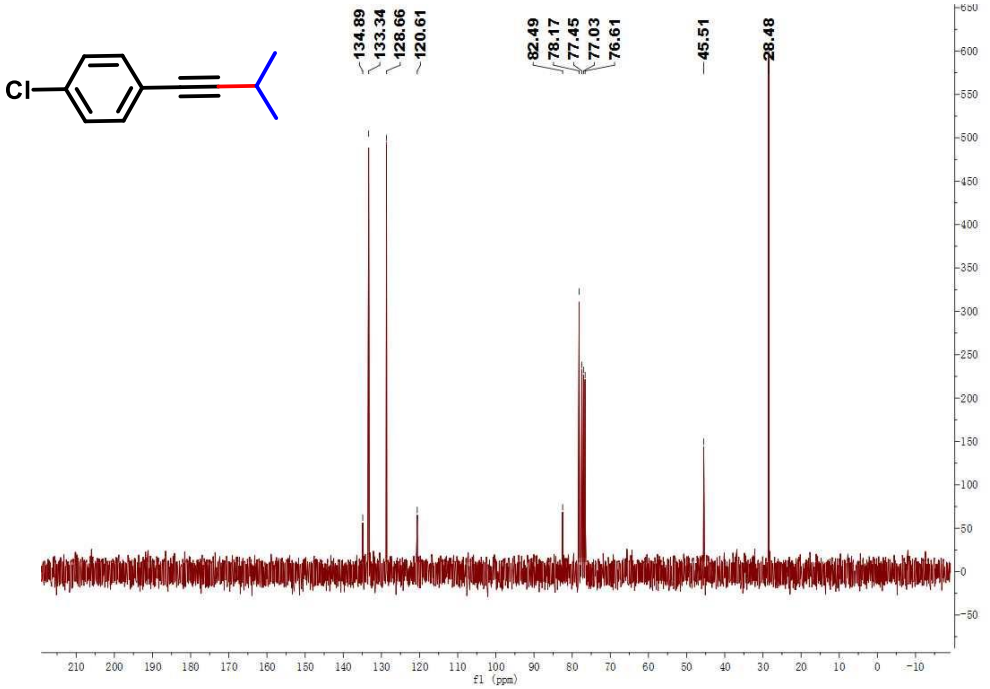
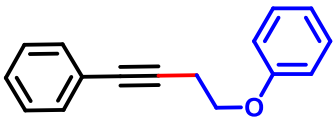


Figure S46. ^{13}C NMR spectrum of **2t** ($CDCl_3$)

(21)(4-phenoxybut-1-yn-1-yl)benzene (2u)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.42 – 6.41 (m, 5H), 6.37 – 6.31 (m, 2H), 6.06 – 5.94 (m, 3H), 3.19 – 3.15 (m, 2H), 2.79 – 2.74 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.24, 132.17, 129.62, 128.82, 128.35, 122.18, 121.45, 114.78, 83.70, 77.52, 77.24, 77.09, 76.67, 67.99，41.94.

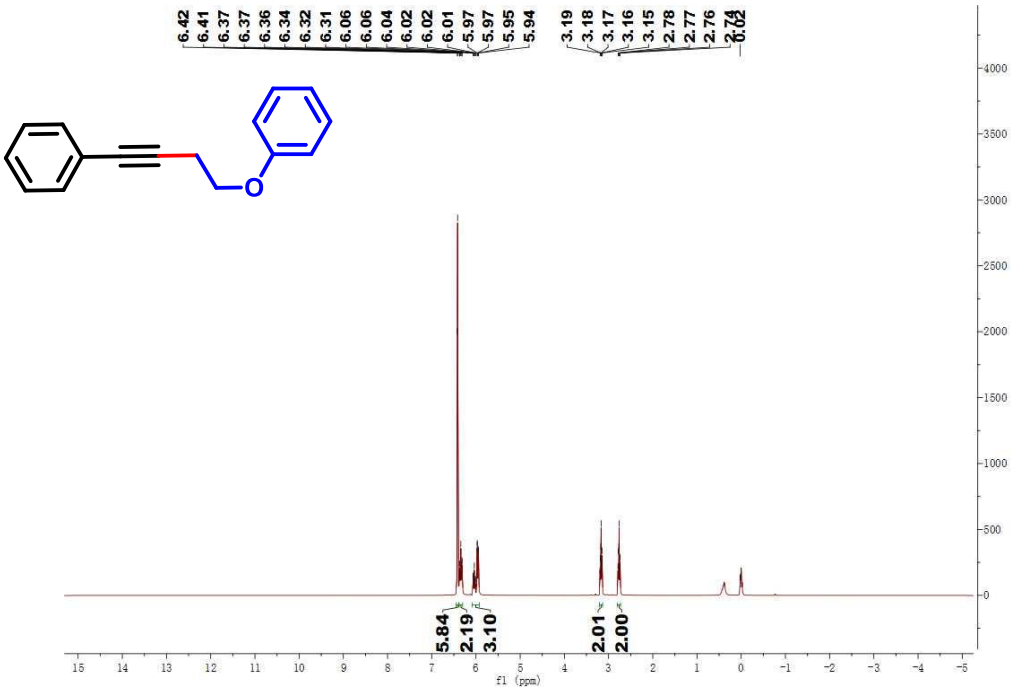


Figure S47. ^{1}H NMR spectrum of **2u** ($CDCl_3$)

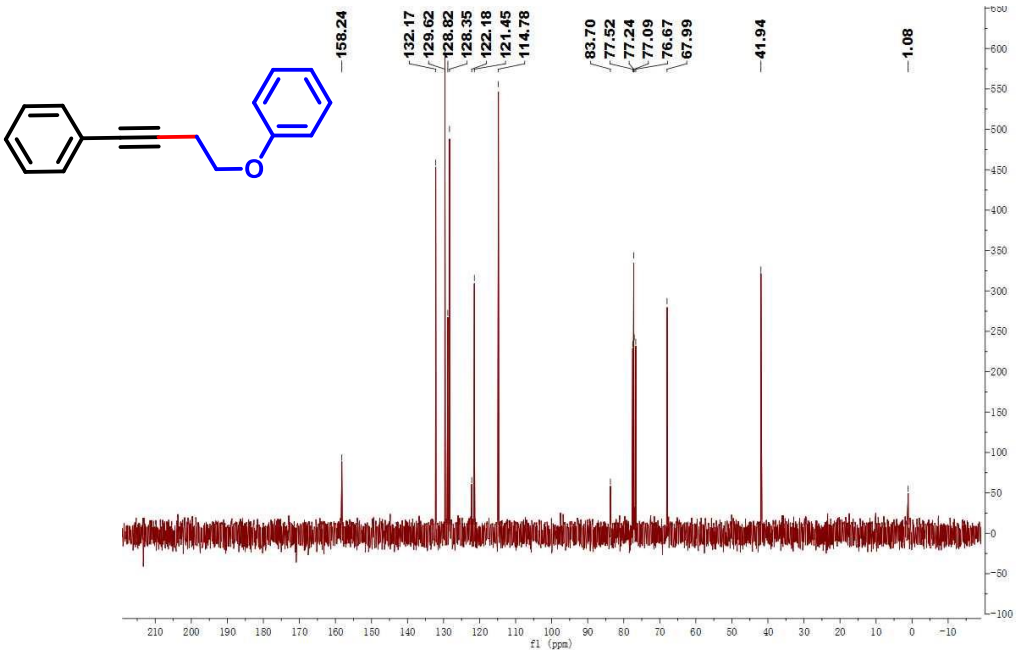
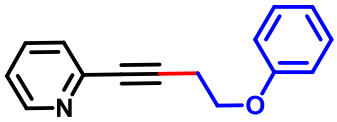


Figure S48. ^{13}C NMR spectrum of **2u** ($CDCl_3$)

(22) 2-(4-phenoxybut-1-yn-1-yl)pyridine (2v)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 – 8.19 (m, 1H), 7.39 – 7.32 (m, 1H), 7.31 – 7.28 (m, 1H), 7.16 – 7.14 (m, 1H), 7.09 – 7.05 (m, 2H), 6.95 – 6.74 (m, 3H), 4.07 – 4.02 (m, 2H), 3.66 – 3.62 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.17, 150.00, 142.31, 136.16, 129.55, 127.46, 123.42, 121.37, 114.71, 82.79, 77.67, 77.22, 76.82, 67.94, 42.01.

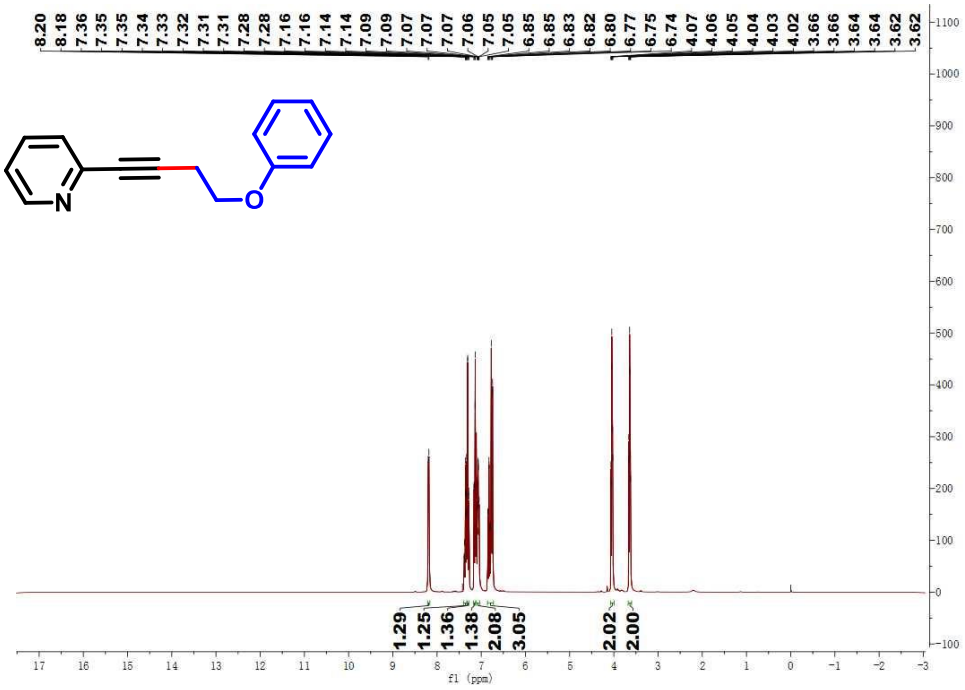


Figure S49. ^{1}H NMR spectrum of **2v** ($CDCl_3$)

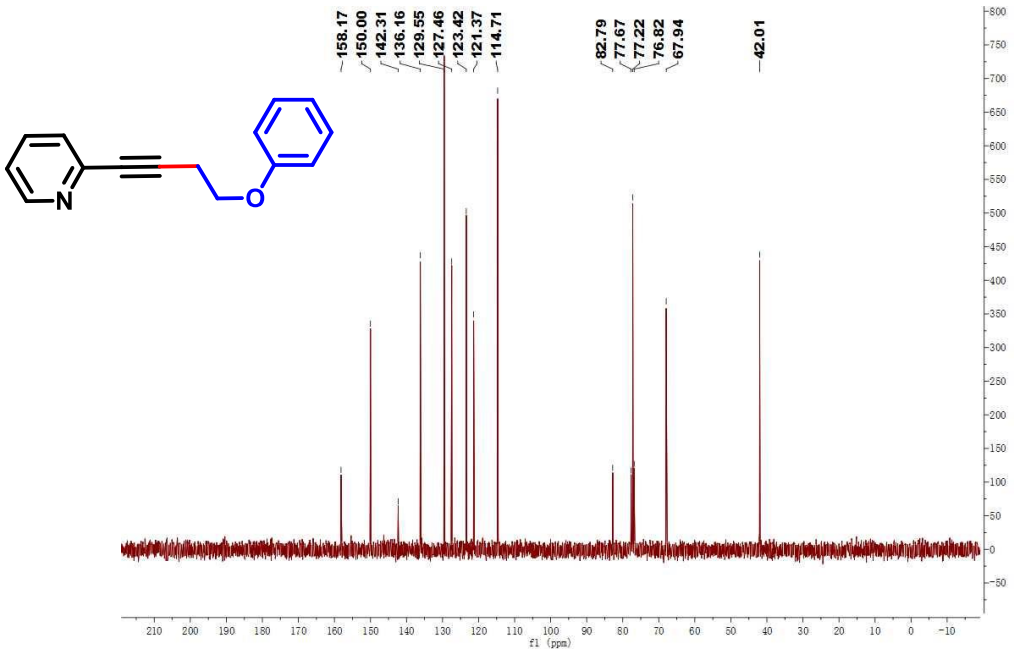
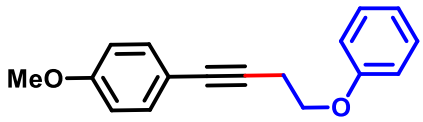


Figure S50. ^{13}C NMR spectrum of **2v** ($CDCl_3$)

(23) 1-methoxy-4-(4-phenoxybut-1-yn-1-yl)benzene (2w)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.19 (m, 2H), 7.14 - 7.11 (m, 2H), 6.86 - 6.81 (m, 1H), 6.77 - 6.74 (m, 2H), 6.62 – 6.59 (m, 2H), 4.01 (t, J = 5.8 Hz, 2H), 3.61 (t, J = 5.8 Hz, 2H), 3.57 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.02, 158.27, 133.64, 129.64, 121.46, 114.80, 114.24, 114.02, 83.75, 77.60, 77.18, 76.75, 75.97, 68.00, 55.29, 42.01.

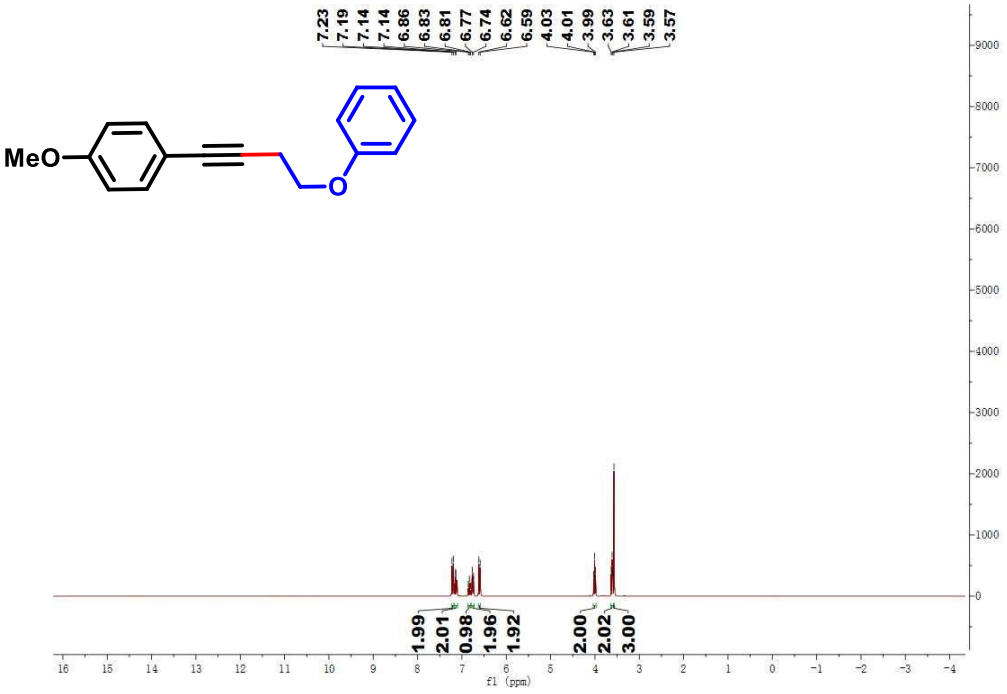


Figure S51. ^{1}H NMR spectrum of **2w** ($CDCl_3$)

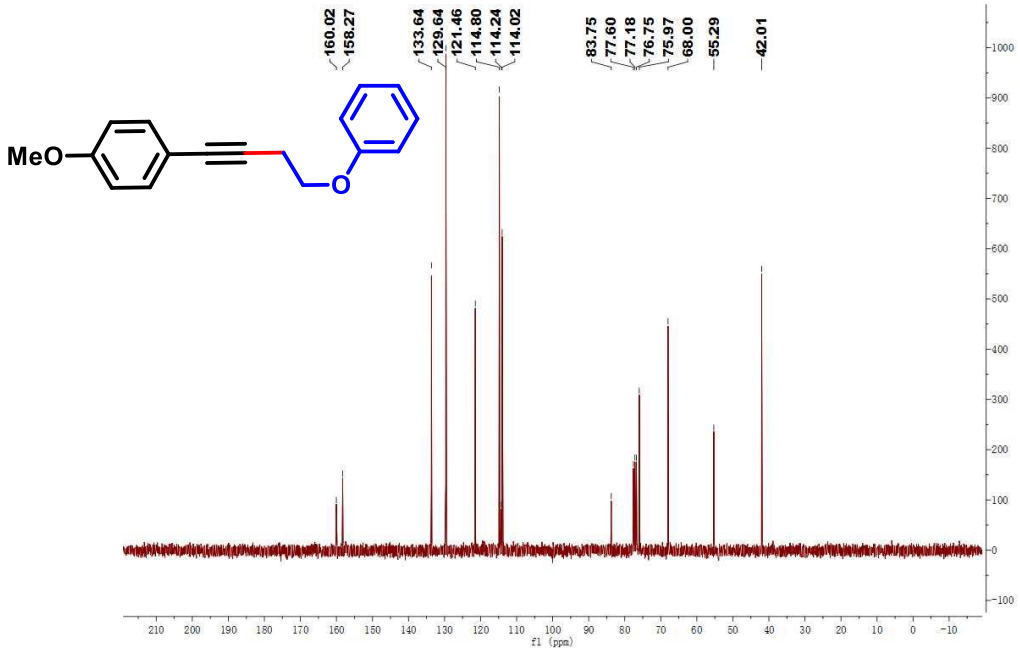
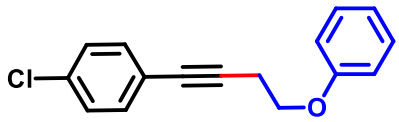


Figure S52. ^{13}C NMR spectrum of **2w** ($CDCl_3$)

(24) 1-chloro-4-(4-phenoxybut-1-yn-1-yl)benzene (2x)



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.45(s, 2H), 6.44 (s, 3H), 6.40 – 6.38 (m, 1H), 6.37 – 6.34 (m, 1H), 6.09 – 6.04 (m, 1H), 6.00 – 5.97 (m, 1H), 3.22 – 3.17 (m, 2H), 2.81 – 2.77 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.22, 134.93, 133.38, 129.61, 128.71, 121.45, 120.65, 114.77, 82.56, 78.27, 77.51, 77.09, 76.67, 67.97, 41.93.

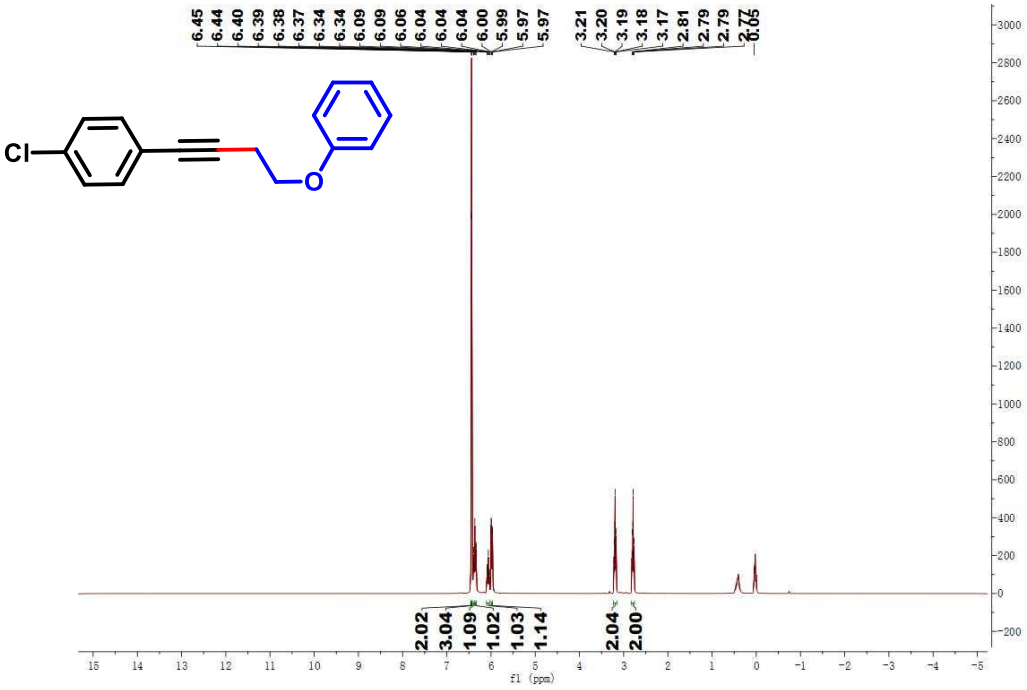


Figure S53. ^{1}H NMR spectrum of **2x** ($CDCl_3$)

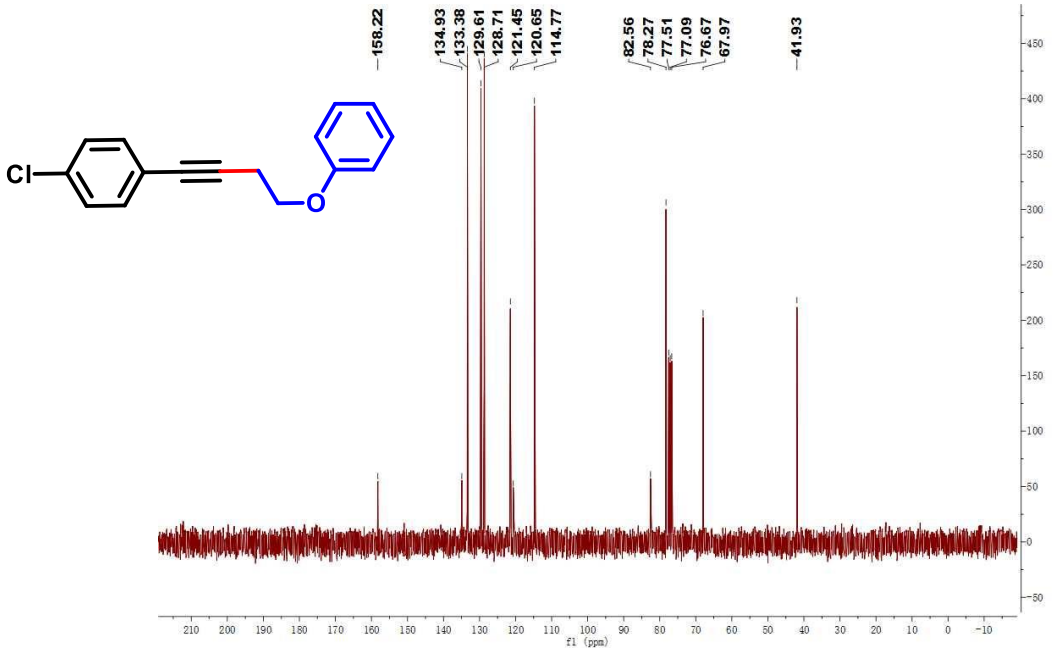
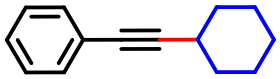


Figure S54. ^{13}C NMR spectrum of **2x** ($CDCl_3$)

(25)(cyclohexylethynyl)benzene (2y)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.42 (s, 2H), 6.41 (s, 3H), 3.08 – 3.01 (m, 1H), 1.13 – 1.10 (m,2H), 0.85 – 0.84 (m, 2H), 0.78 – 0.68 (m, 2H), 0.59 – 0.54 (m, 2H), 0.43- 0.36 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.12, 128.77, 128.30, 122.16, 83.64, 77.49, 77.15, 77.06, 76.64, 60.26, 36.70, 25.14, 24.88.

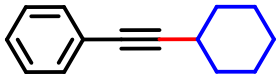


Figure S55. ^{1}H NMR spectrum of **2y** ($CDCl_3$)

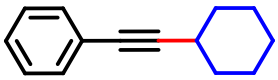
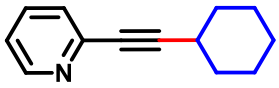


Figure S56. ^{13}C NMR spectrum of **2y** ($CDCl_3$)

(26)(2-cyclohexylethynyl)pyridine (2z)[1]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27 – 8.25 (m, 1H), 7.49 – 7.43 (m, 1H), 7.39 – 7.36 (m, 1H), 7.18 – 7.10 (m, 1H), 3.94 – 3.85 (m, 1H), 2.02 – 1.92 (m, 2H), 1.72 – 1.63 (m, 2H), 1.60 – 1.48 (m, 2H), 1.44 – 1.25 (m, 2H), 1.25 – 1.16 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.98, 142.32, 136.11, 127.40, 123.36, 82.70, 77.54, 77.12, 77.07, 76.70, 60.21, 36.62, 25.06, 24.79.

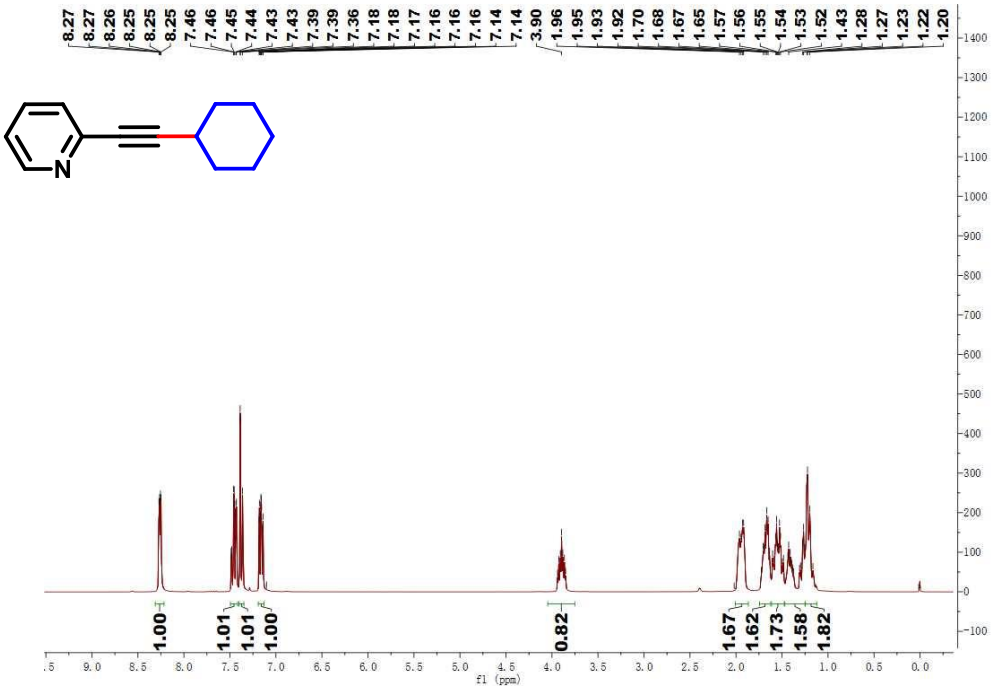


Figure S57. ^{1}H NMR spectrum of **2z** ($CDCl_3$)

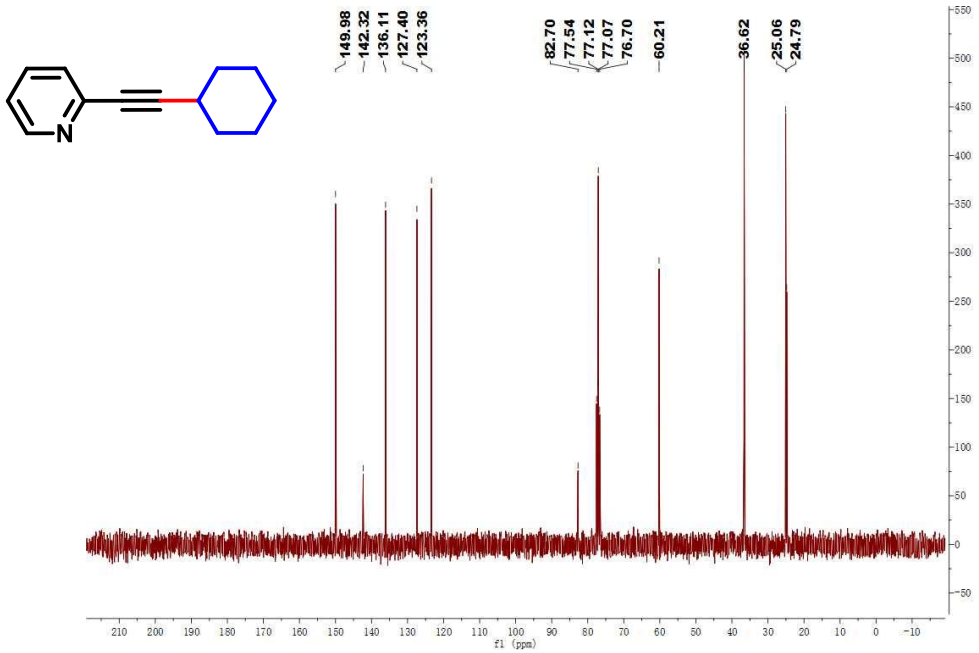
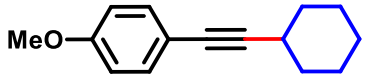


Figure S58. ^{13}C NMR spectrum of **2z** ($CDCl_3$)

(27) 1-(cyclohexylethynyl)-4-methoxybenzene (2a')[4]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.24 (m, 1H), 7.23 – 7.21 (m, 1H), 6.74 – 6.72(m, 1H), 6.71 – 6.69 (m, 1H), 3.70 (s, 3H), 1.76 – 1.60 (m, 1H), 1.45 – 1.37 (m, 2H), 1.20 – 1.15 (m, 4H), 0.90 – 0.86 (s, 2H), 0.83 – 0.80 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.95, 133.56, 114.19, 113.93, 83.65, 77.50, 77.07, 76.65, 75.78, 60.26, 55.24, 36.69, 25.13, 24.87.

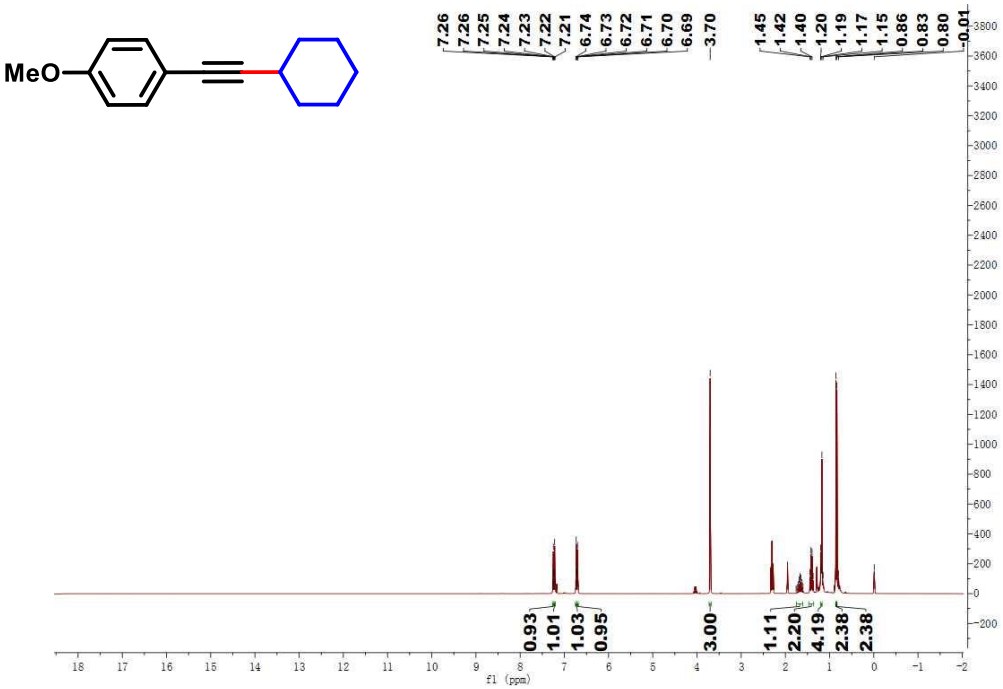


Figure S59. ^{1}H NMR spectrum of **2a'** ($CDCl_3$)

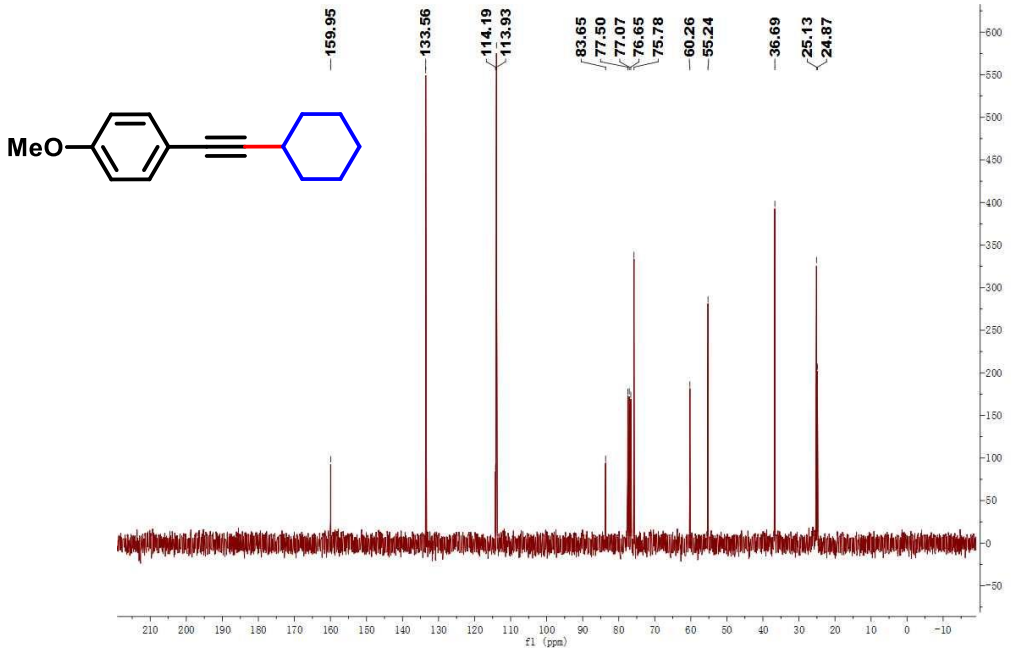
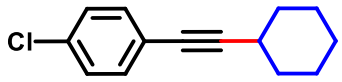


Figure S60. ^{13}C NMR spectrum of **2a'** ($CDCl_3$)

(28) 1-chloro-4-(cyclohexylethynyl)benzene (2b')[10]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.42 (s, 2H), 6.41 (s, 2H), 3.08 – 3.01 (m, 1H), 1.13 – 1.10 (m, 2H), 0.85 – 0.78 (m, 2H), 0.78 – 0.68 (m, 2H), 0.59 – 0.42 (m, 2H), 0.42 – 0.36 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.89, 133.34, 128.66, 120.62, 82.49, 78.17, 77.47, 77.05, 76.62, 60.25, 36.68, 25.13, 24.86.

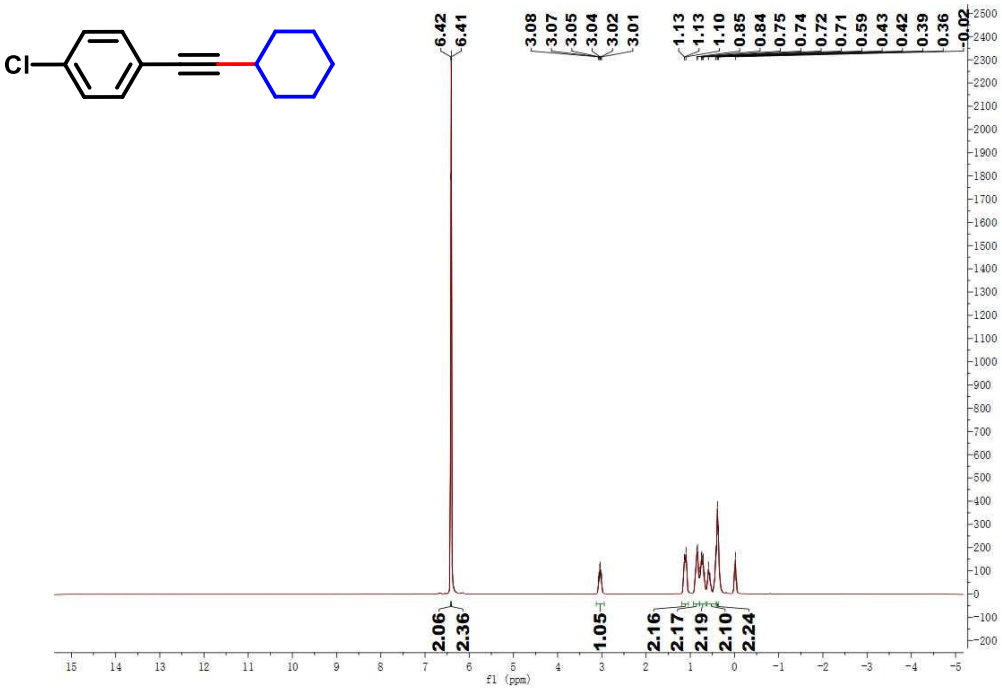


Figure S61. ^{1}H NMR spectrum of **2b'** ($CDCl_3$)

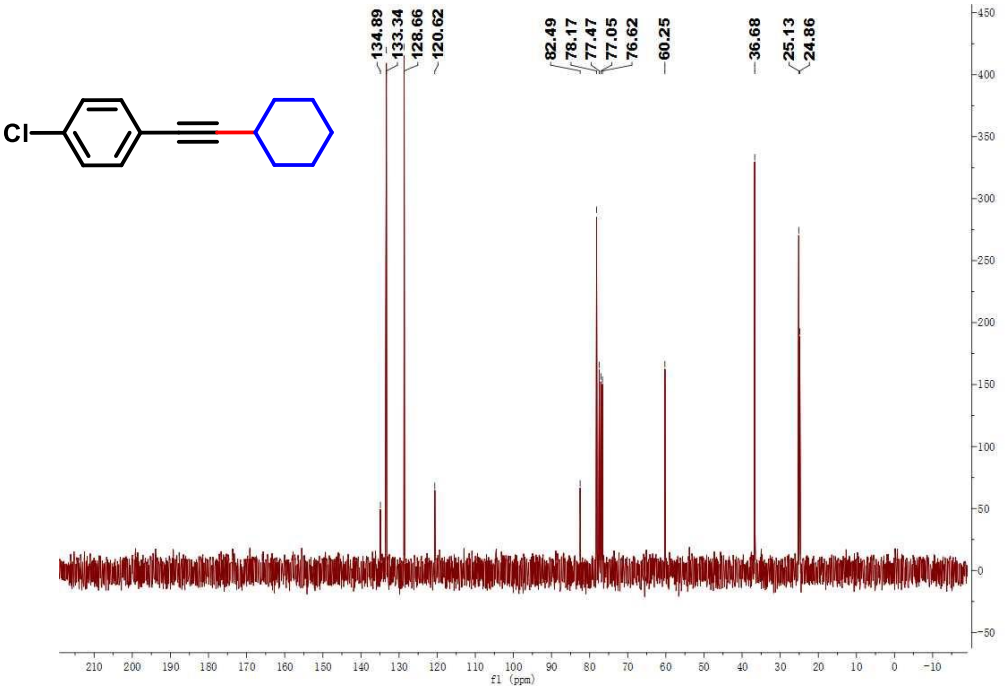
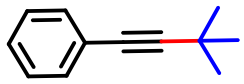


Figure S62. ^{13}C NMR spectrum of **2b'** ($CDCl_3$)

(29)(3,3-dimethylbut-1-yn-1-yl)benzene(2c')[10]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.43 (s, 2H), 6.42 (s, 3H), 0.71 – 0.70 (s, 3H), 0,70 – 0.69 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.12, 128.78, 128.31, 122.16, 83.65, 77.47, 77.16, 77.04, 76.62, 67.39, 34.45.

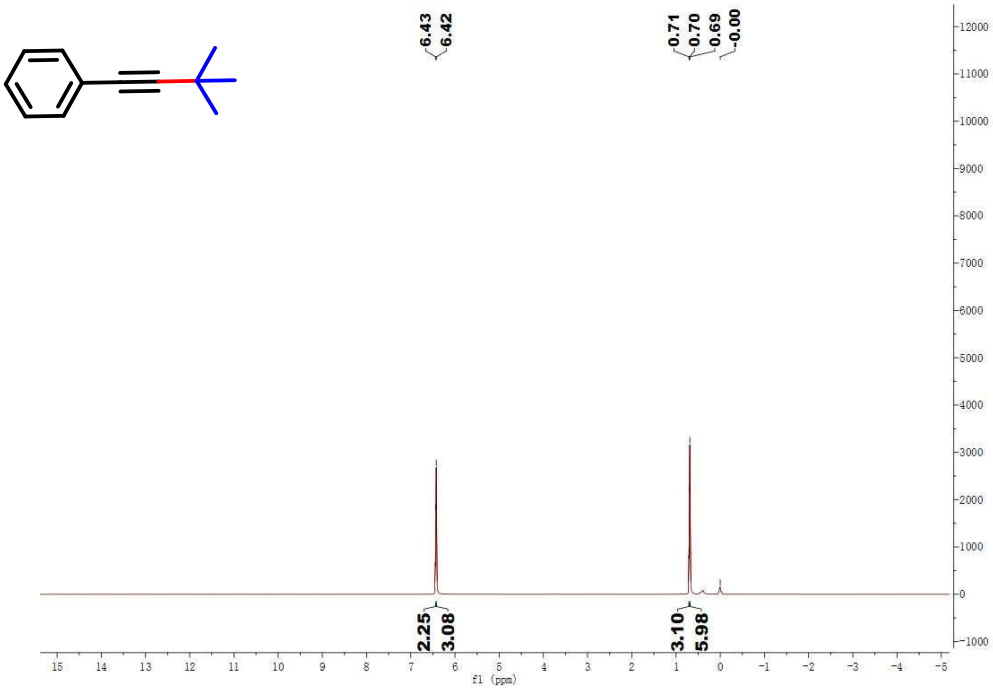


Figure S63. ^{1}H NMR spectrum of **2c'** ($CDCl_3$)

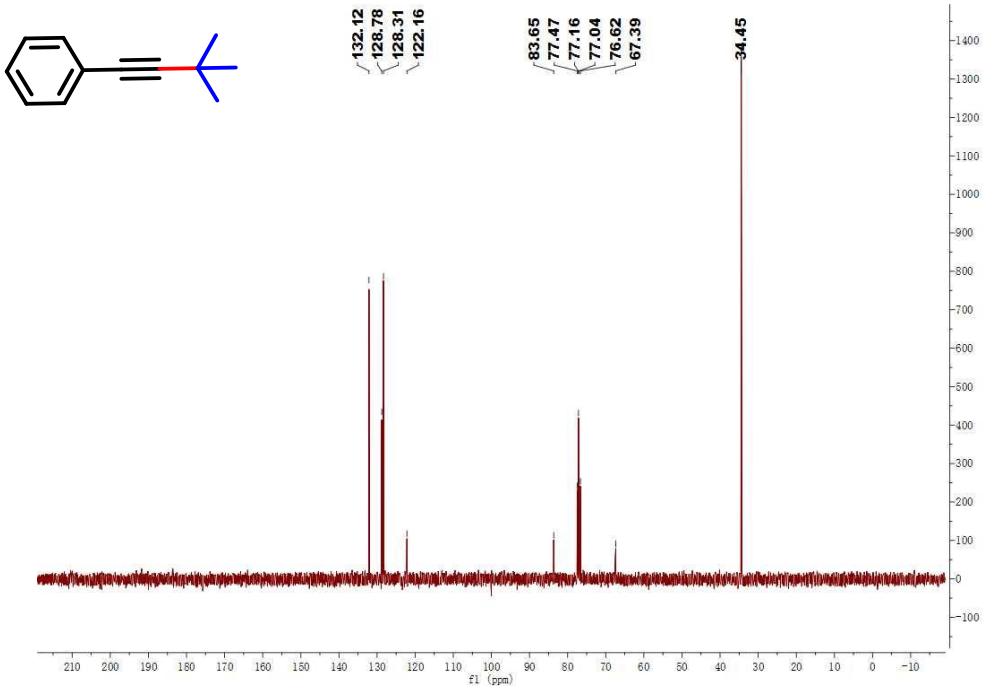
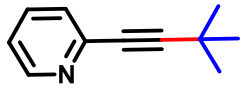


Figure S64. ^{13}C NMR spectrum of **2c'** ($CDCl_3$)

(30) 2-(3,3-dimethylbut-1-yn-1-yl)pyridine (2d')[5]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27 – 8.25 (m, 1H), 7.48 – 7.42 (m, 1H), 7.39 – 7.36 (m, 1H), 7.18 – 7.14 (m, 1H), 1.51 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.95, 142.28, 136.11, 127.39, 123.36, 82.70, 77.58, 77.16, 77.10, 76.73, 67.38, 34.36.

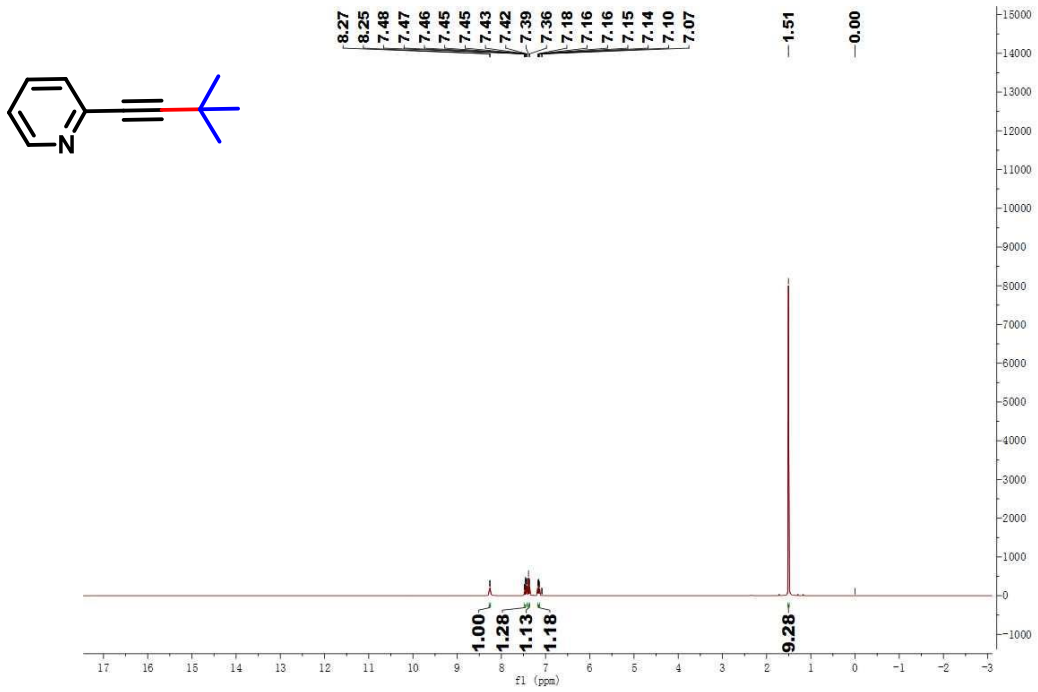


Figure S65. ^{1}H NMR spectrum of **2d'** ($CDCl_3$)

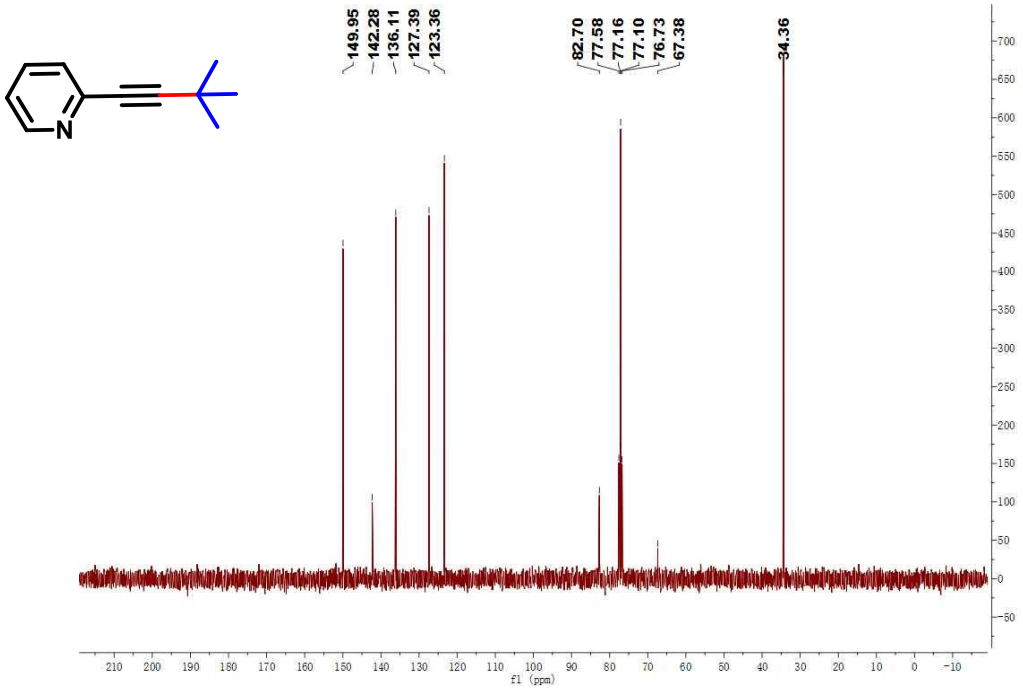
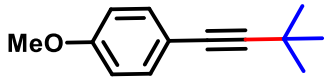


Figure S66. ^{13}C NMR spectrum of **2d'** ($CDCl_3$)

(31) 1-(3,3-dimethylbut-1-yn-1-yl)-4-methoxybenzene (2e')[10]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26– 7.23 (m, 2H), 6.68 – 6.64 (m, 2H), 3.64 (s, 3H), 1.51 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.95, 133.56, 114.19, 113.93, 83.65, 77.46, 77.04, 76.62, 75.78, 67.39, 55.24, 34.44.

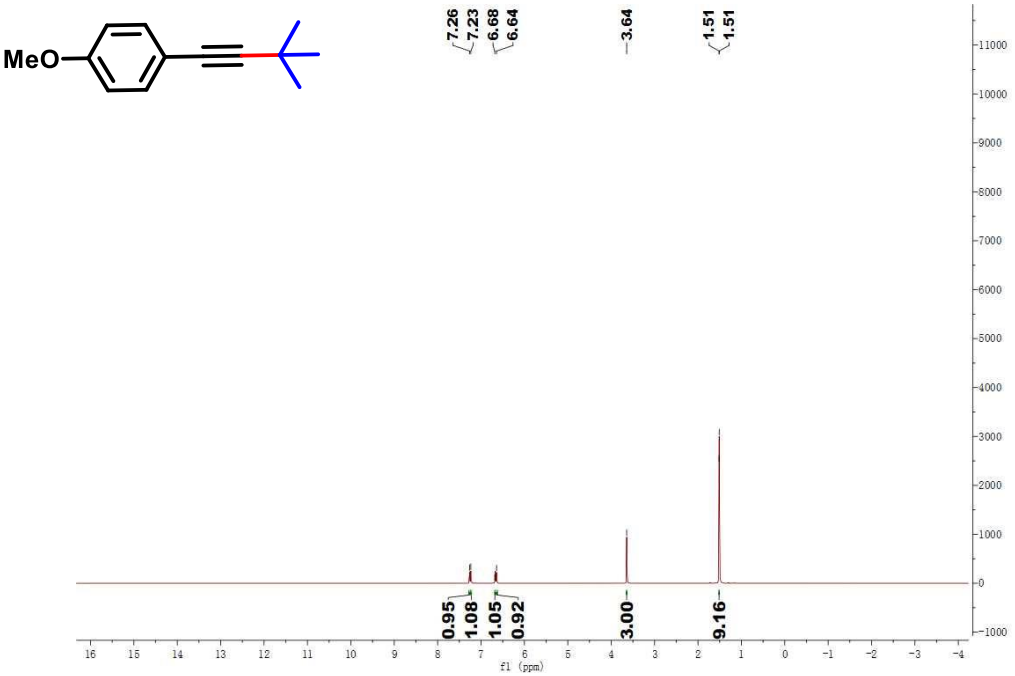


Figure S67. ^{1}H NMR spectrum of **2e'** ($CDCl_3$)

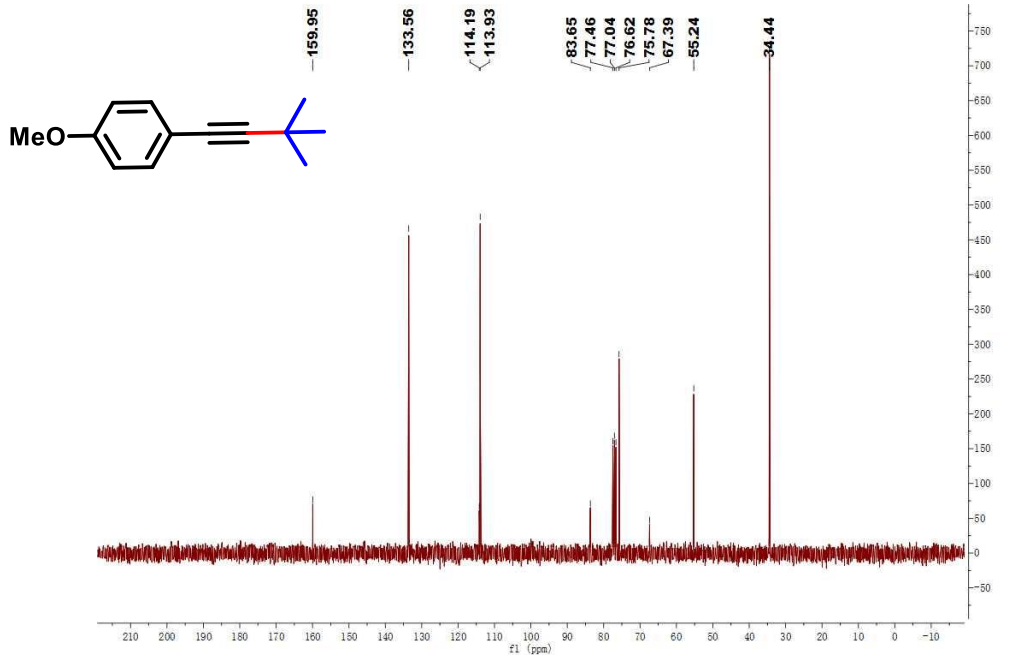
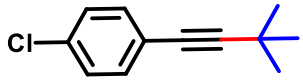


Figure S68. ^{13}C NMR spectrum of **2e'** ($CDCl_3$)

(32) 1-chloro-4-(3,3-dimethylbut-1-yn-1-yl)benzene (2f')[2]



^{1}H NMR (300 MHz, $CDCl_3$) δ 6.43 (s, 2H), 6.42 (s, 2H), 0,70 (s, 3H), 0.69 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.89, 133.34, 128.66, 120.63, 82.50, 78.18, 77.45, 77.02, 76.60, 67.36, 34.43.

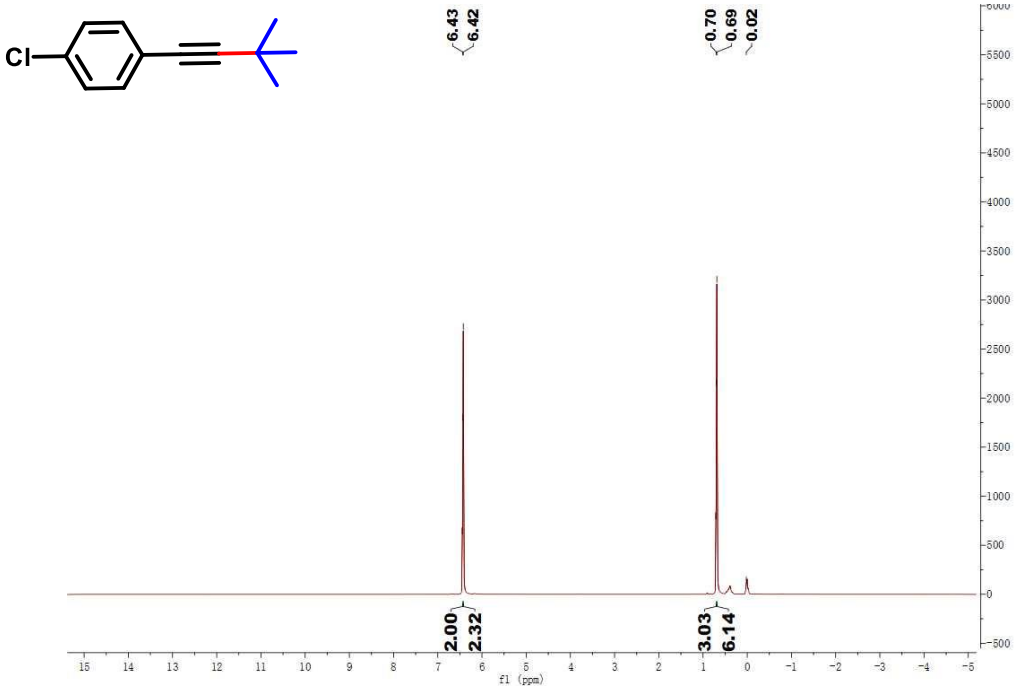


Figure S69. ^{1}H NMR spectrum of **2f'** ($CDCl_3$)

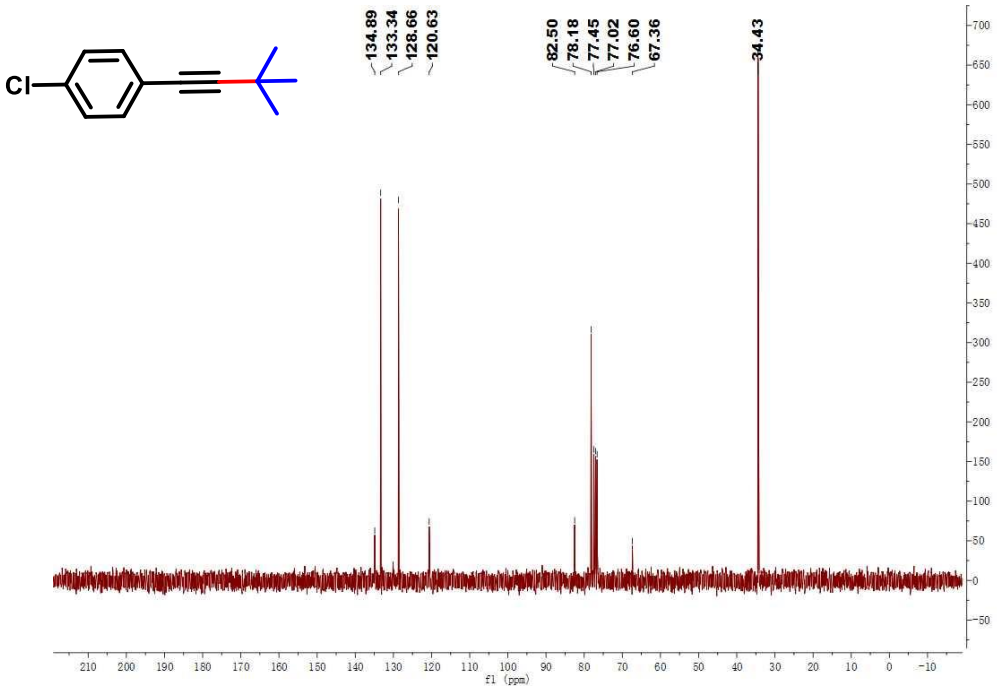
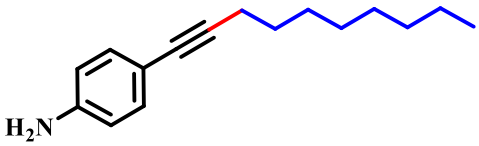


Figure S70. ^{13}C NMR spectrum of **2f'** ($CDCl_3$)

(33) 4-(dec-1-yn-1-yl)aniline (2g')[11]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.00 (s, 1H), 6.98 (s, 1H), 6.50 (s, 1H), 6.47 (s, 1H), 3.52 (s, 2H), 3.43 (t, J = 6.7 Hz, 2H), 1.69 – 1.62 (m, 2H), 1.32 (s, 2H), 1.20 (s, 8H), 0.80 – 0.78 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.09, 133.46, 114.58, 111.27, 84.44, 77.53, 77.11, 76.68, 74.93, 45.24, 32.68, 31.70, 29.17, 28.89, 26.92, 22.67, 14.12.

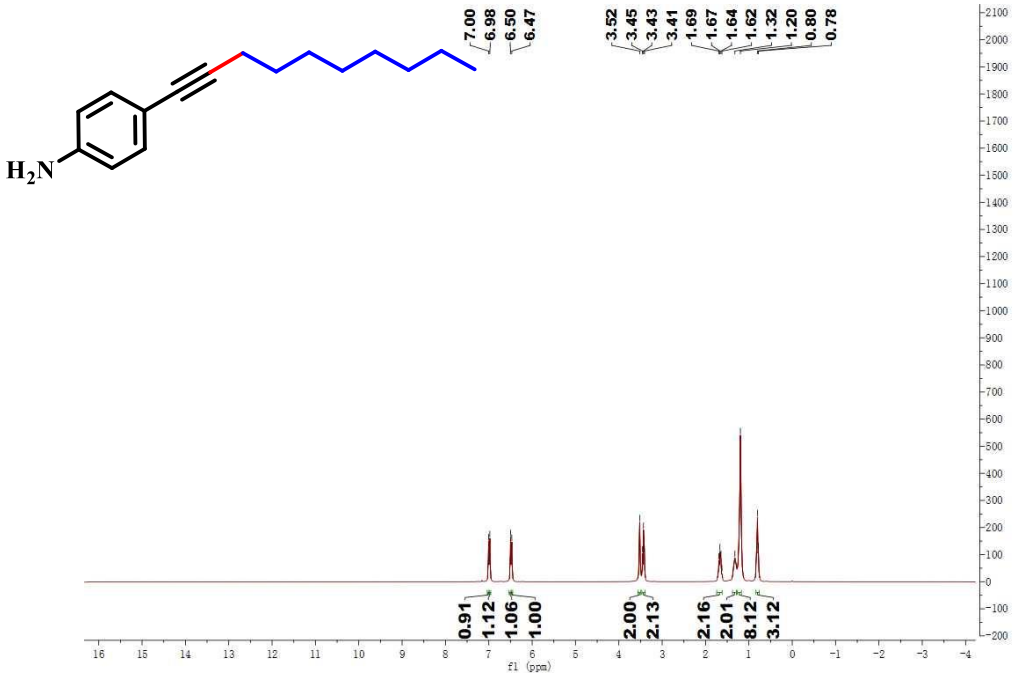


Figure S71. ^{1}H NMR spectrum of **2g'** ($CDCl_3$)

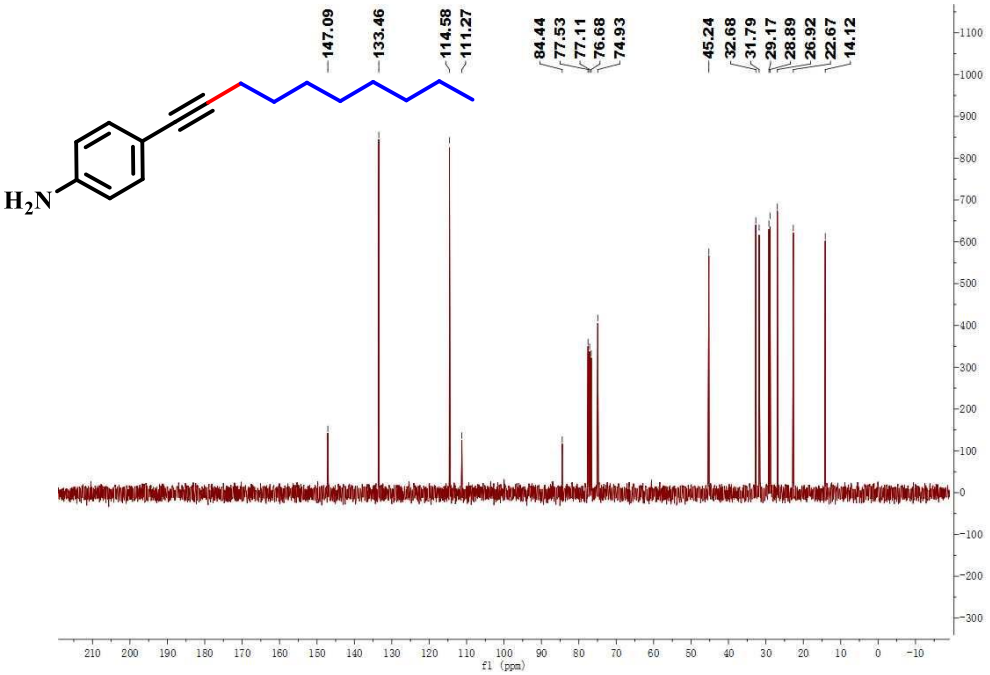
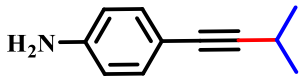


Figure S72. ^{13}C NMR spectrum of **2g'** ($CDCl_3$)

(34) 4-(3-methylbut-1-yn-1-yl)aniline (2h')



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.02 (s, 1H), 6.99 (s, 1H), 6.53 (s, 1H), 6.50 (s, 1H), 4.19 – 4.08 (m, 1H), 3.43 (s, 2H), 1.45 (s, 3H), 1.42 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.13, 133.45, 114.59, 111.24, 84.46, 77.55, 77.13, 76.70, 74.96, 54.16, 27.24.

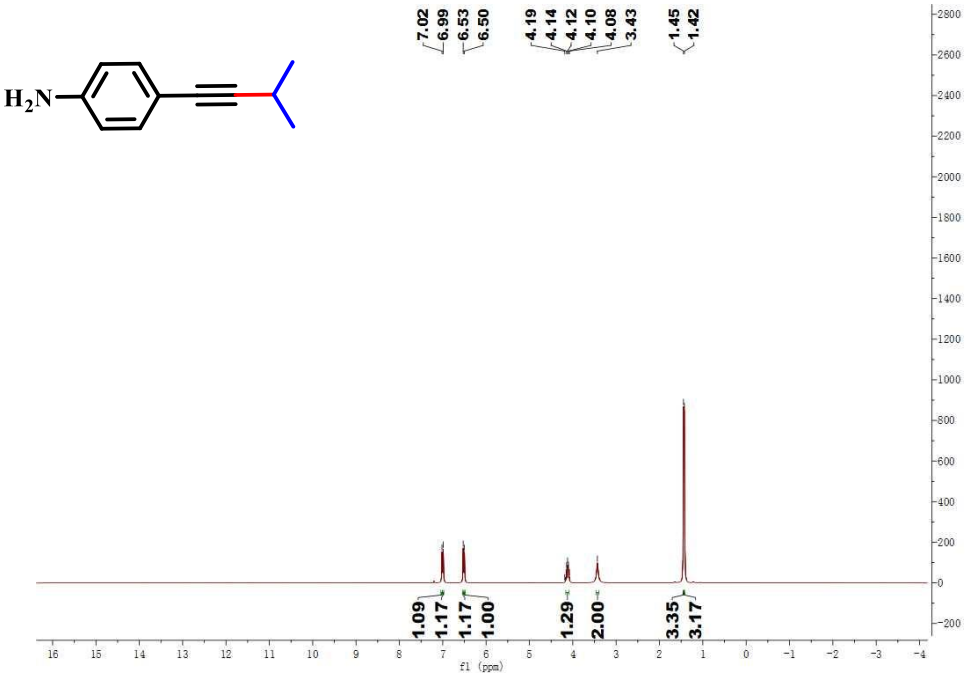


Figure S73. ^{1}H NMR spectrum of **2h'** ($CDCl_3$)

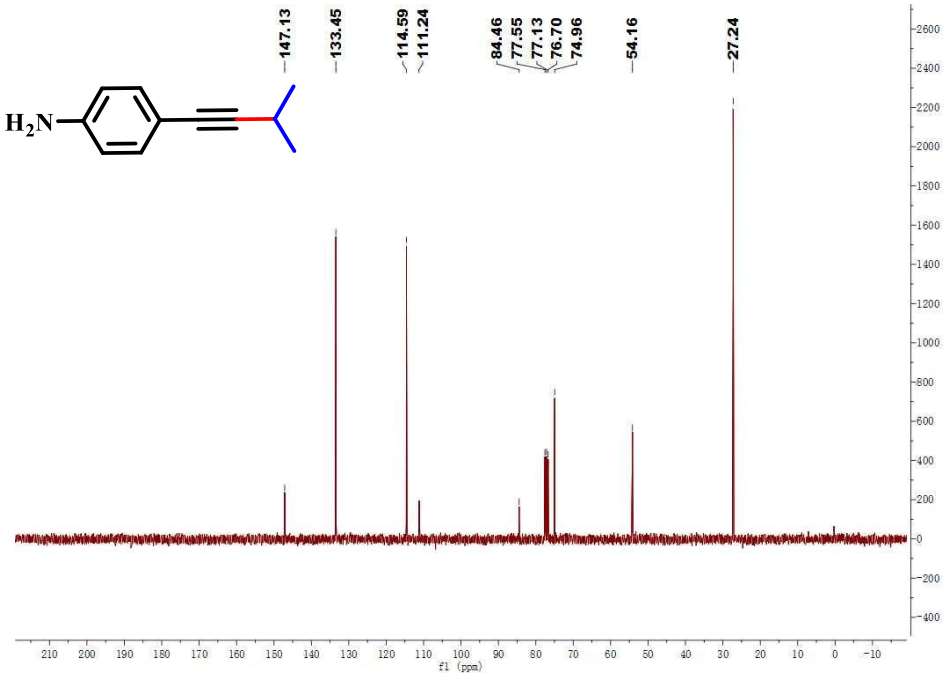
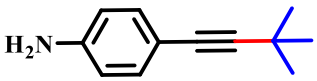


Figure S74. ^{13}C NMR spectrum of **2h'** ($CDCl_3$)

(35) 4-(3,3-dimethylbut-1-yn-1-yl)aniline (2i')



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.12 (s, 1H), 7.10 (s, 1H), 6.62 (s, 1H), 6.60 (s, 1H), 3.66 (s, 2H), 1.65 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.09, 133.46, 114.59, 111.25, 84.52, 77.51, 77.09, 76.67, 74.94, 67.62, 34.47.

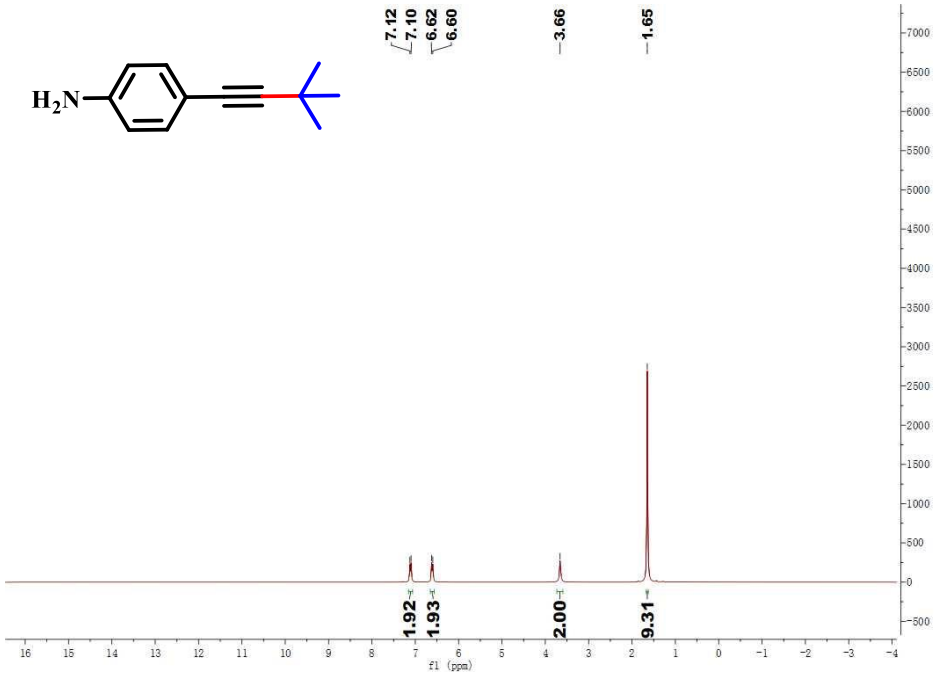


Figure S75. ^{1}H NMR spectrum of **2i'** ($CDCl_3$)

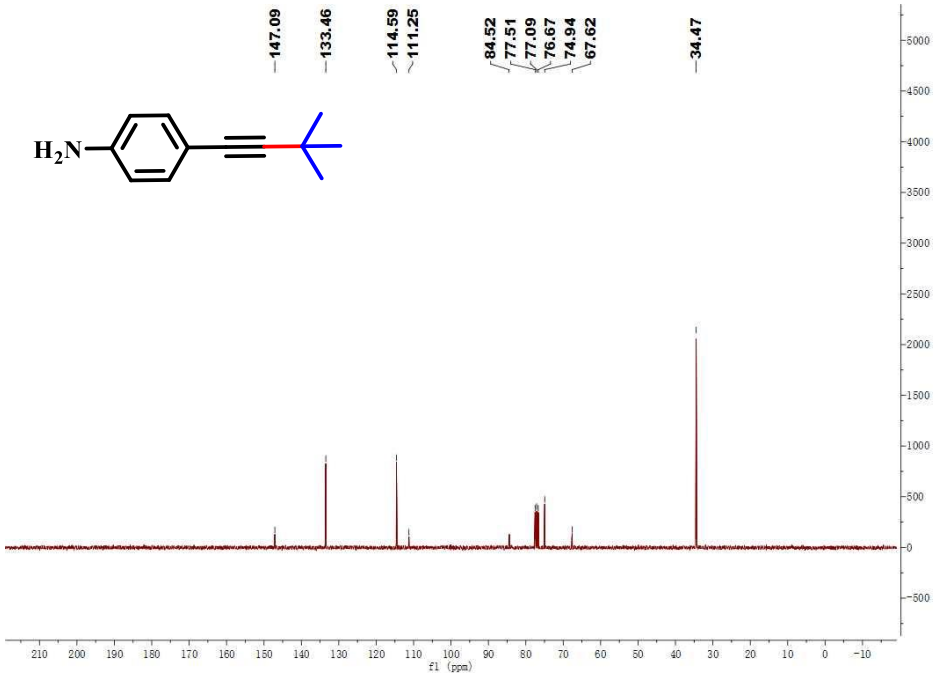
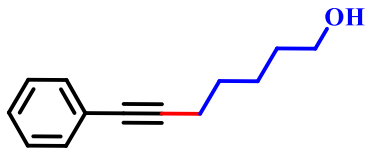


Figure S76. ^{13}C NMR spectrum of **2i'** ($CDCl_3$)

(36) 7-phenylhept-6-yn-1-ol (2j')[12]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39 – 7.36 (m, 2H), 7.17 – 7.08 (m, 3H), 6.52 (s, 1H), 3.59 – 3.54 (m, 2H), 3.32 – 3.28 (m, 2H), 1.79 – 1.74 (m, 2H), 1.53 – 1.36 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.09, 133.46, 114.59, 111.25, 84.52, 77.51, 77.09, 76.67, 74.94, 67.62, 34.47.

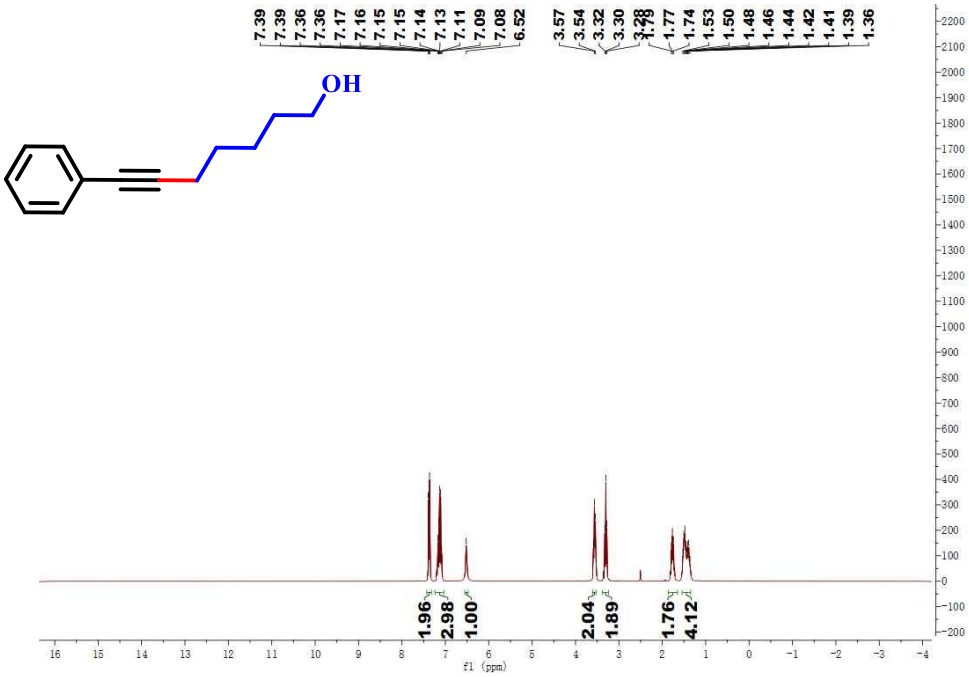


Figure S77. ^{1}H NMR spectrum of **2j'** ($CDCl_3$)

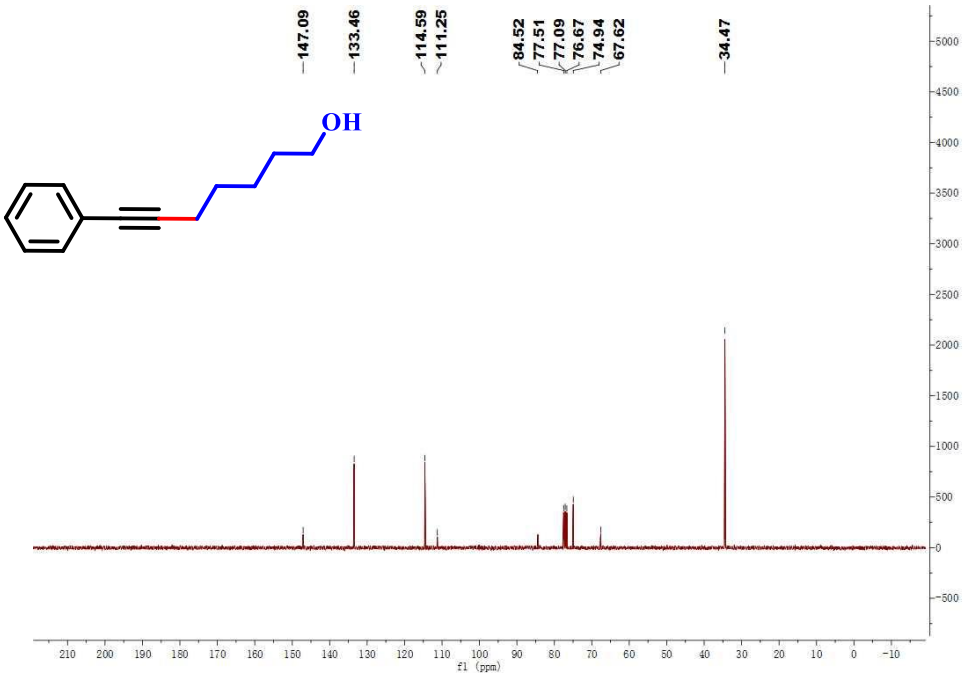
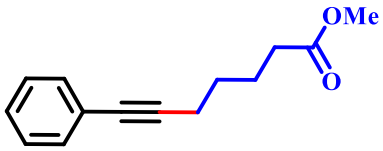


Figure S78. ^{13}C NMR spectrum of **2j'** ($CDCl_3$)

(37) methyl 7-phenylhept-6-ynoate (2k')[13]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38 (s, 1H), 7.36 (s, 1H), 7.16 – 7.13 (m, 1H), 7.13 – 7.10 (m, 2H), 3.55 (s, 3H), 3.42 – 3.38 (m, 2H), 2.22 – 2.20 (m, 2H), 1.68 – 1.65 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 173.44, 132.05, 128.75, 128.20, 122.12, 83.57, 77.68, 77.35, 77.26, 76.83, 51.45, 44.40, 33.05, 31.80, 22.19.

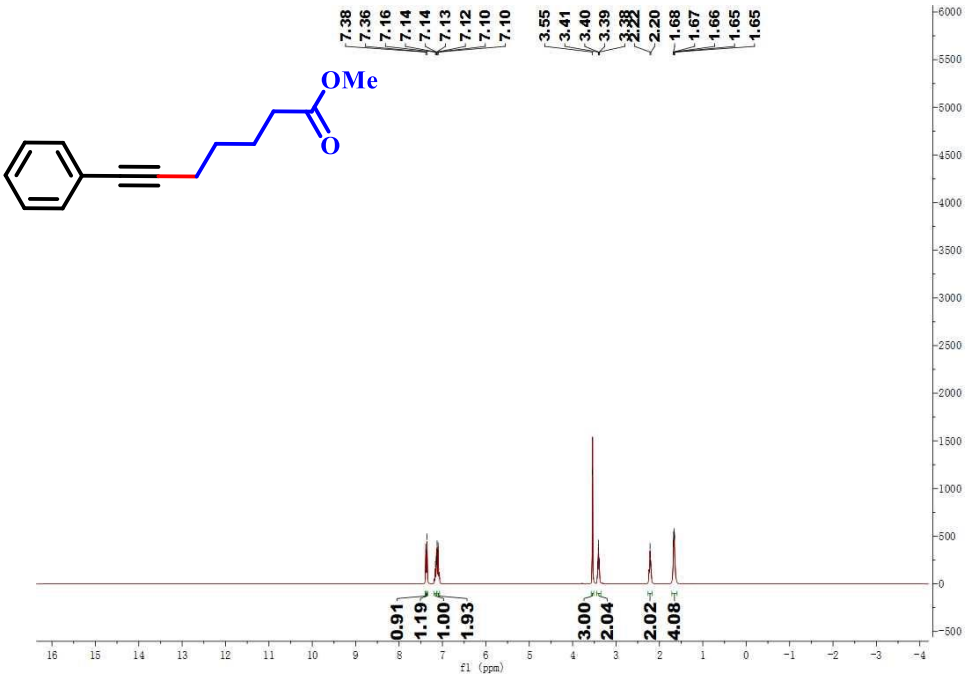


Figure S79. ^{1}H NMR spectrum of **2k'** ($CDCl_3$)

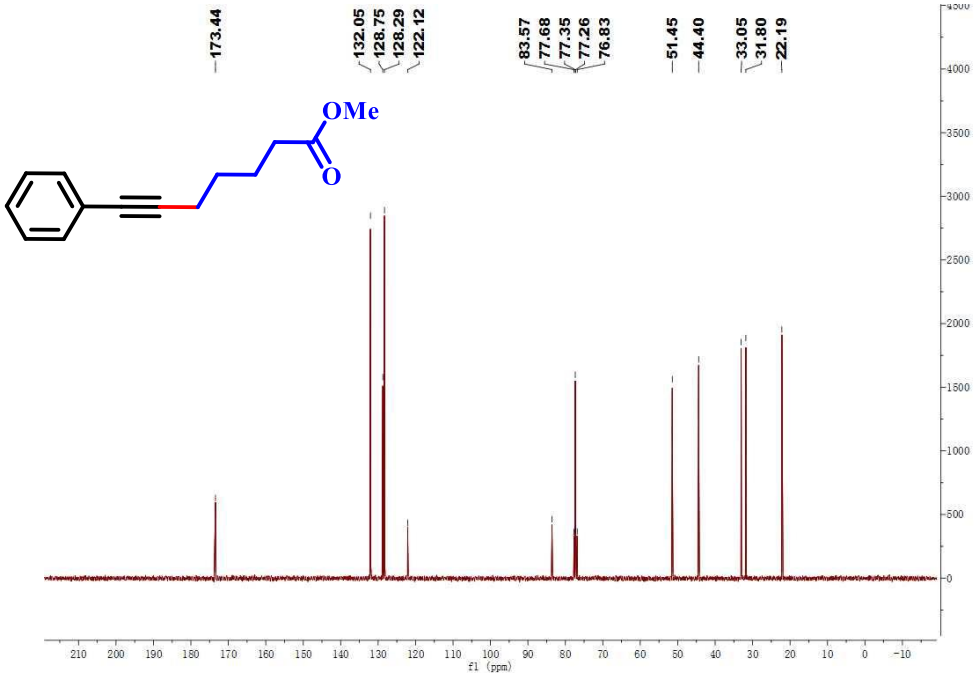
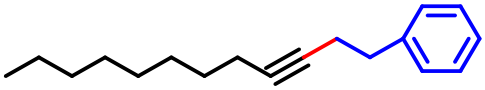


Figure S80. ^{13}C NMR spectrum of **2k'** ($CDCl_3$)

(38)(dodec-3-yn-1-ylbenzene (2l')



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.43 – 7.35 (m, 2H), 7.35 – 7.27 (m, 3H), 3.63 (t, J = 7.7 Hz, 2H), 3.27 – 3.21 (m, 4H), 1.92 – 1.85 (m, 2H), 1.49 – 1.45 (m, 2H), 1.38 (s, 8H), 1.02 – 0.97(m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.99, 128.67, 126.97, 84.64, 77.70, 77.27, 76.85, 68.28, 39.56, 32.84, 32.00, 29.36, 29.25, 28.92, 28.66, 22.82, 18.52, 14.23.

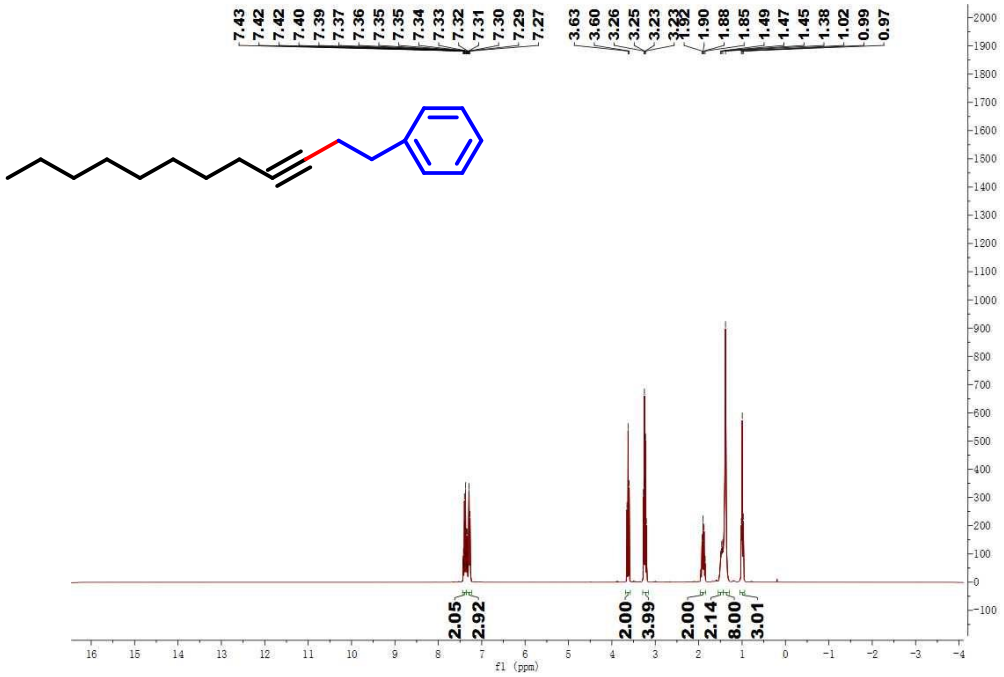


Figure S81. ^{1}H NMR spectrum of **2l'** ($CDCl_3$)

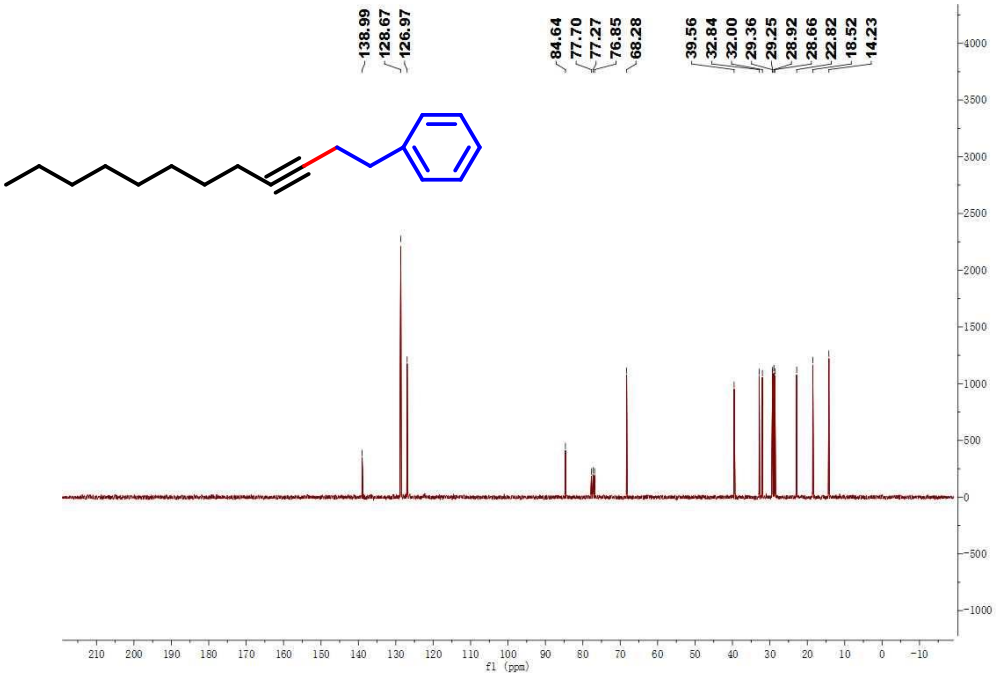
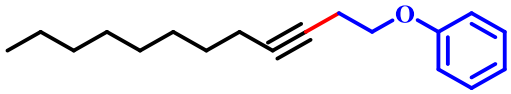


Figure S82. ^{13}C NMR spectrum of **2l'** ($CDCl_3$)

(39)(dodec-3-yn-1-yloxy)benzene (2m')



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.15 – 7.12 (m, 2H), 6.84 – 6.75 (m, 3H), 4.03 (t, J = 5.9 Hz, 2H), 3.63 (t, J = 5.9 Hz, 2H), 3.03 (t, J = 7.1 Hz, 2H), 1.72 – 1.63 (m, 2H), 1.20 – 1.17 (m, 10H), 0.81 – 0.77 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 158.27, 129.61, 121.41, 114.72, 84.68, 77.65, 77.23, 76.80, 68.23, 67.93, 41.92, 31.95, 29.30, 29.20, 28.87, 28.61, 22.77, 18.46, 14.17.

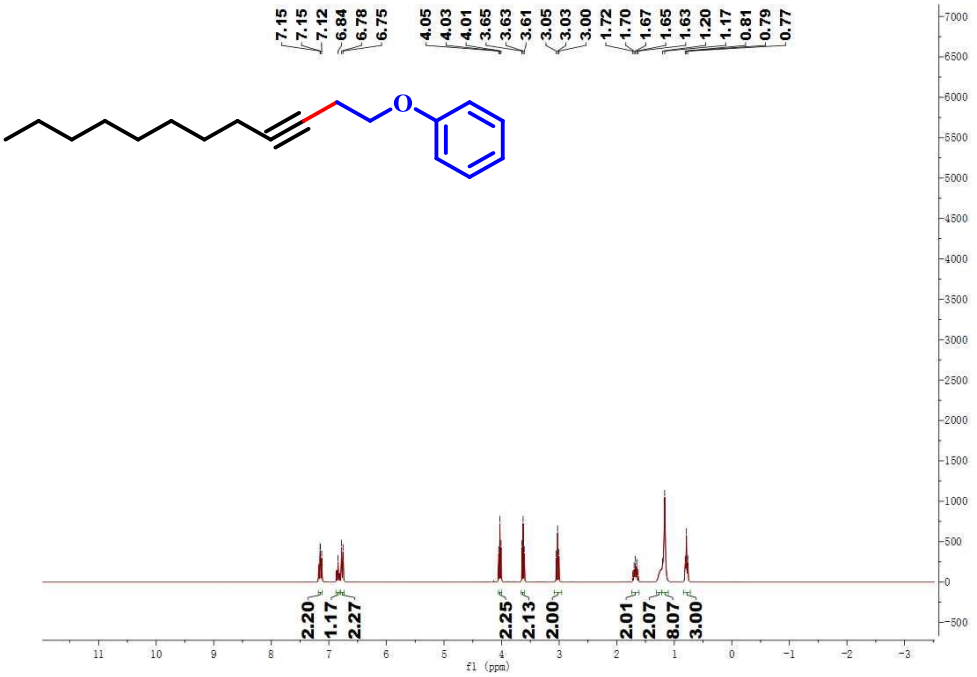


Figure S83. ^{1}H NMR spectrum of **2m'** ($CDCl_3$)

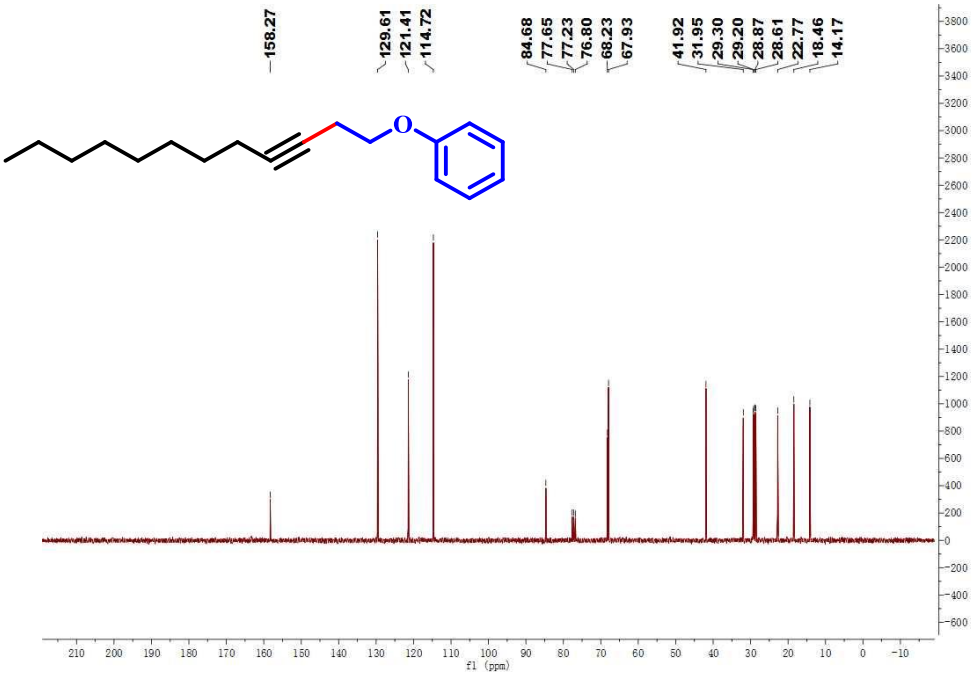
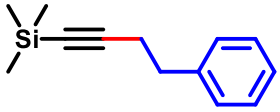


Figure S84. ^{13}C NMR spectrum of **2m'** ($CDCl_3$)

(40)(trimethyl(4-phenylbut-1-yn-1-yl)silane (2n')[14]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.44 – 7.38 (m, 2H), 7.36 – 7.28 (m, 3H), 3.63 (t, J = 7.6 Hz, 2H), 3.23 (t, J = 7.6 Hz, 2H), 1.72 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.40, 129.18, 129.13, 127.43, 93.76, 90.53, 78.09, 77.66, 77.23, 39.94, 33.41, 0.27.

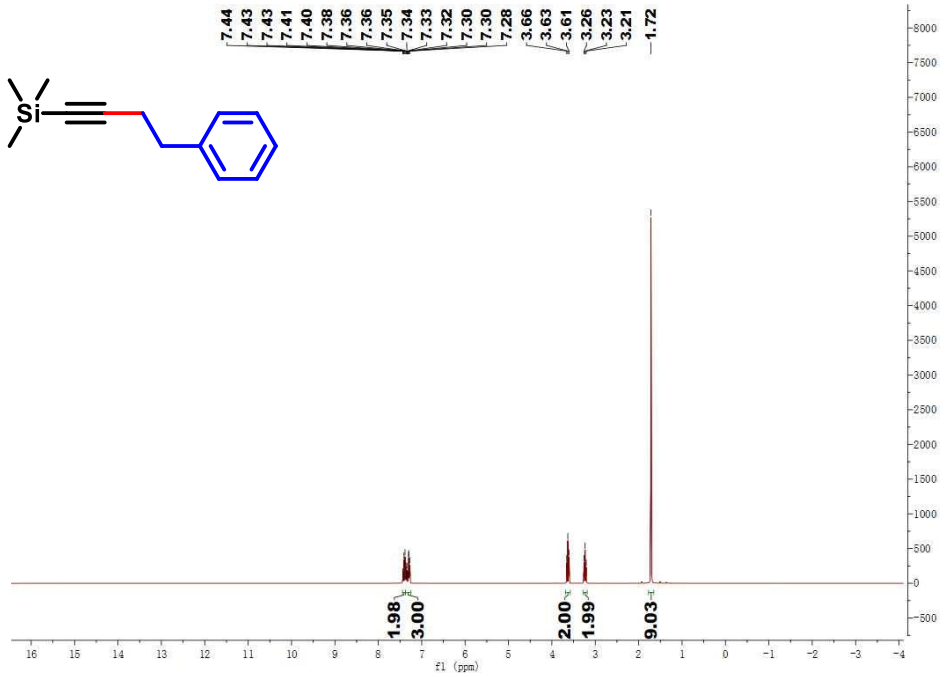


Figure S85. ^{1}H NMR spectrum of **2n'** ($CDCl_3$)

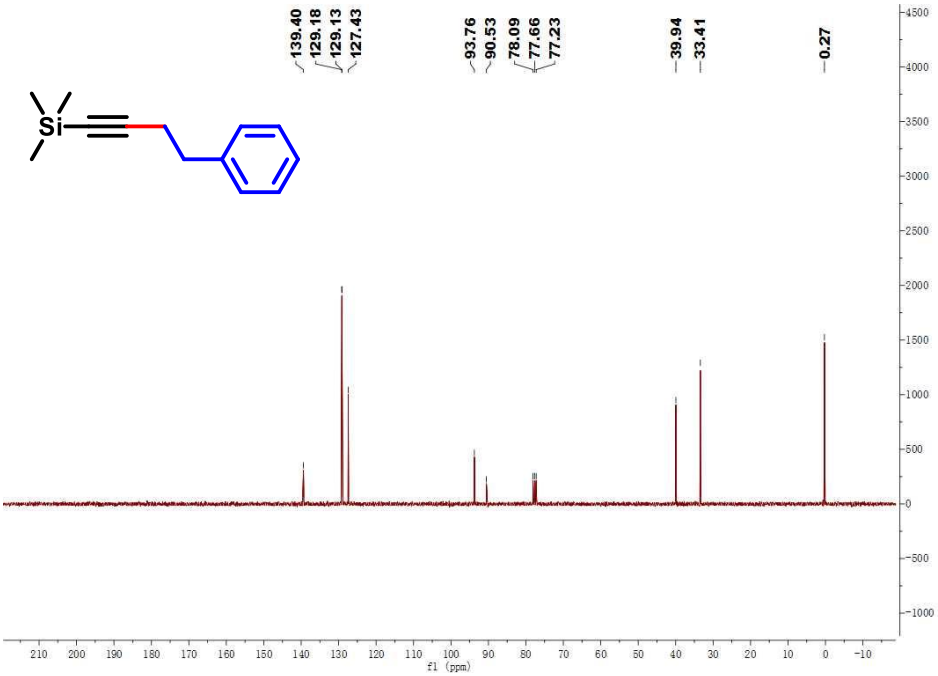
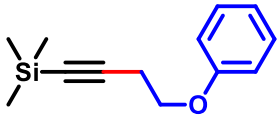


Figure S86. ^{13}C NMR spectrum of **2n'** ($CDCl_3$)

(41)(trimethyl(4-phenoxybut-1-yn-1-yl)silane (2o')[15]



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.18– 7.13 (m, 2H), 6.87 – 6.76 (m, 3H), 4.06 (t, J = 5.8 Hz, 2H), 3.63 (t, J = 5.8 Hz, 2H), 1.49 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 158.75, 130.15, 121.95, 115.26, 93.81, 90.62, 78.12, 77.70, 77.21, 68.45, 42.51, 0.28.

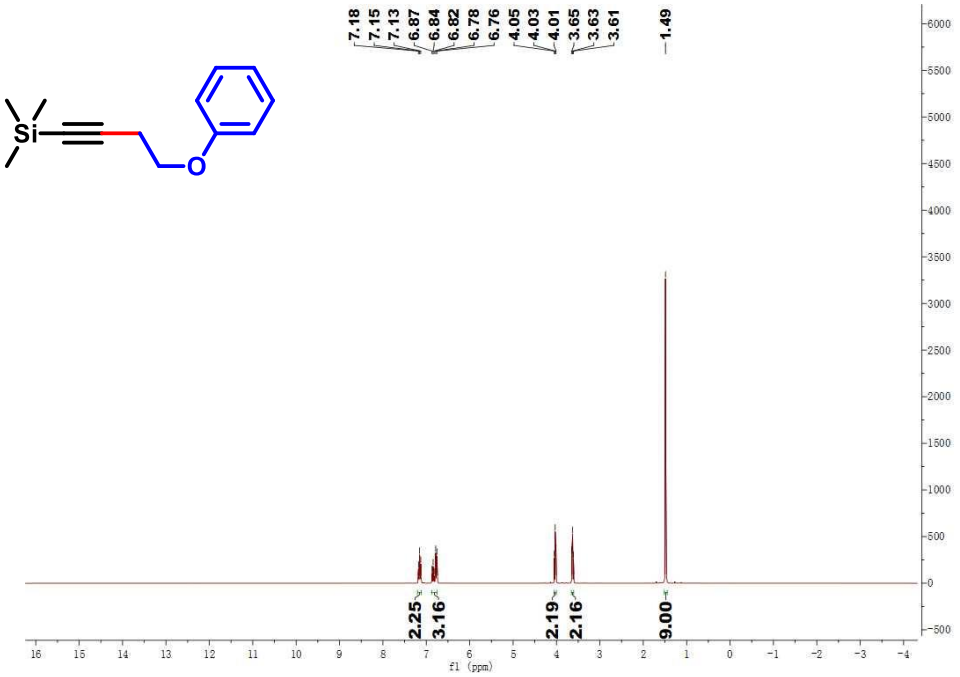


Figure S87. ^{1}H NMR spectrum of **2o'** ($CDCl_3$)

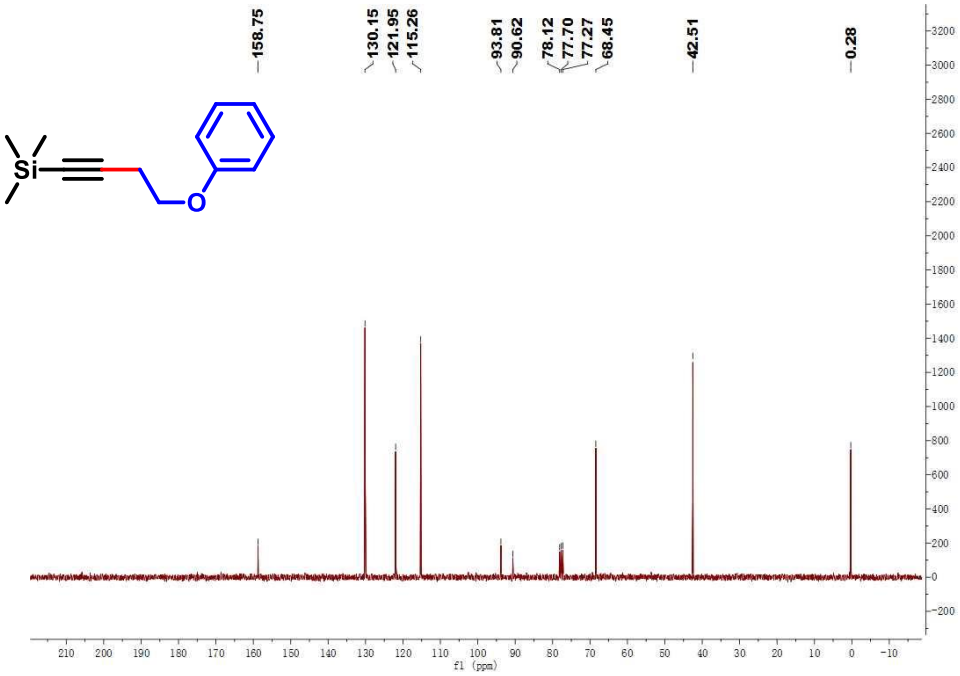


Figure S88. ^{13}C NMR spectrum of **2o'** ($CDCl_3$)

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