Cu-Photoredox-Catalyzed C(sp)-C(sp^3) Coupling of Redox-Active Esters with Terminal Alkynes

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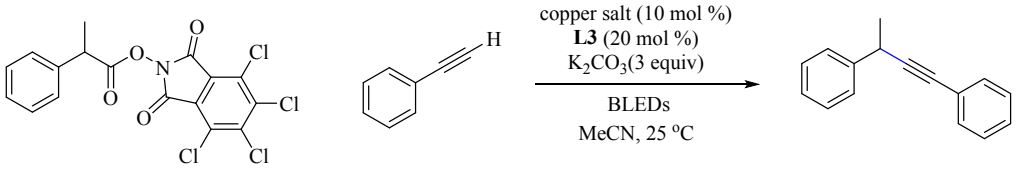
1 General information

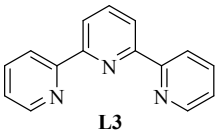
All reagents and solvents were purchased from commercial suppliers, and the reactions were carried out under a nitrogen atmosphere. ^{1}H-NMR, ^{13}C-NMR, and ^{19}F-NMR spectra were recorded with a Bruker (400 MHz), Varian Inova (400 MHz) or Aglient (400 MHz) spectrometer. All chemical shifts (δ) are quoted in parts per million (ppm) and $CDCl_3$ (77.16 ppm for ^{13}C and 7.260 ppm for ^{1}H) was the test solvent unless otherwise noted. The abbreviations were used for an explanation of multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet. Blue LED lamps (40W, Kessil A160WE tuna blue) were used for reactions. Cyclic voltammetry were obtained from Shanghai Chen Hua CHI660. Photolysis experiments were performed on FZ-A Photolysis Spectrometer.

2 Experimental Details

(1) Optimization of the reaction conditions

Copper salt Screen





L3

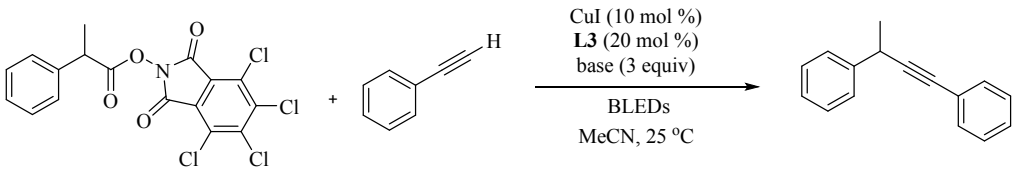
Copper Salt Screen

Entry	Copper salt	Yield (%)[a]
1	CuCl	53
2	CuBr	50
3	$Cu(MeCN)_4PF_6$	38
4	CuCN	40
5	$Cu(ClO_4)_2·6H_2O$	49
6	CuOAc	34
7	$Cu(OTf)_2$	32

[a]Yields determined by^{1}HNMR analysis.

NR: no reaction

Base Screen



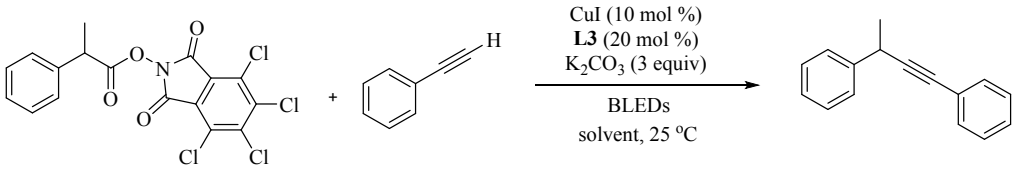
Entry	Base	Yield (%)[a]

1	Li_2CO_3	13
2	Na_2CO_3	21
3	K_2CO_3	81
4	Cs_2CO_3	34
5	K_3PO_4	46
6	Na_2HPO_4	5
7	Et_3N	NR
8	LiOtBu	trace
9	KOtBu	trace

[a]Yields determined by[1]HNMR analysis.

NR: no reaction

Solvent Screen



Entry	Solvent	Yield (%)[a]
1	THF	10
2	DCE	30
3	DMF	48
4	iPrOH	NR
5	DMSO	NR
6	MeCN:MeOH (V:V=3:1)	37

[a]Yields determined by[1]HNMR analysis.

NR: no reaction

(2) Reaction setup

Fig. S1 shows the emission spectra of the light. The vials are borosilicate glass and composed of a 3.0 cm long and 1.0 cm wide. It was placed 5cm away from blue LED lamps, and then place a fan blowing down on the vials. The strength of the air flow is adjusted so that the reaction temperature of the vials never exceeded 25 °C (Fig. 2).

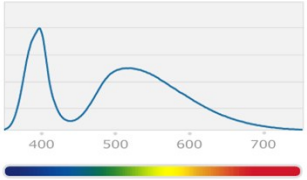


Fig. 1 Emission spectra[1]

Fig. 2 Reaction chamber

(3) General procedure and characterizations of products

General procedure for decarboxylative alkylation

General procedure: Under nitrogen atmosphere, 2, 2′, 6′, 2′′-terpyridine (0.04 mmol, 20 mol %), CuI (0.02 mmol, 10 mol %), K_2CO_3 (0.6 mmol, 3.0 equiv), alkyne (0.2 mmol, 1 equiv), and N-acyloxyl derivatives (0.4 mmol, 2.0 equiv) were added in a dried reaction vessel with 2 mL of MeCN. The reaction mixture was stirred at room temperature under Blue LED for 24h. After completion, the reaction was quenched and the suspension was filtered by CH_2Cl_2 over a pad of silica gel. The volatile solvent was evaporated in vacuum. The crude product was purified by column chromatography.

Note: The electron deficient tetrachloro derivative proved to be unstable[2], carboxylic acids at activated position compounds **a2-a8** were N-hydroxyphthalimide esters. Others substrates are TCNHPI esters

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenylpropanoate **a1**

According to the published procedures[3], **a1** was obtained from 2-phenyllpropionic acid (1.50g, 10mmol) as a white solid (3.4g, 79%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.38 (m, 4H), 7.35-7.31 (m, 1H), 4.12 (q, *J*=7.1Hz, 1H), 1.67 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.4, 141.1, 138.0, 130.5, 129.1, 128.1, 127.6, 124.8, 43.0, 19.0 HRMS(ESI): $C_{17}H_{10}Cl_4NO_4$, calcd $[M+H]^+$: 431.9283 found:431.9288

1,3-dioxoisoindolin-2-yl 2-(2-bromophenyl)acetate **a4**

According to the published procedures[3], **a4** was obtained from 2-bromophenylacetic acid (1.08g, 5mmol) as a white solid (1.48g, 82%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.86(m, 2H), 7.80-7.76 (m, 2H), 7.61(dd, *J* = 8.0, 0.8 Hz, 1H), 7.44 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.33 (td, *J* = 7.6, 1.2 Hz, 1H), 7.19 (dd, *J* = 7.6, 1.6 Hz, 1H), 4.16 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): 167.0, 161.9, 134.9, 133.1, 132.0, 131.4, 129.7, 129.0, 127.9, 125.1, 124.1, 38.5 HRMS(ESI): $C_{16}H_{11}BrNO_4$, calcd $[M+H]^+$: 359.9795 found: 359.9789

1,3-dioxoisoindolin-2-yl 2-(4-(trifluoromethyl)phenyl)acetate **a5**

According to the published procedures[3], **a5** was obtained from 2-(4-(trifluoromethyl)phenyl)acetic acid (1.02g, 5mmol) as a white solid (1.31g, 75%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87-7.84 (m, 2H), 7.78-7.75 (m, 2H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 4.05 (s, 2H). ^{19}F NMR (376 MHz, $CDCl_3$): -62.6 (s, 1F);^{13}C NMR (101 MHz, $CDCl_3$): 167.2, 161.8, 135.6 (q, *J* = 1.2 Hz), 135.0, 129.8, 129.7 (q, *J* = 32.7 Hz), 128.8, 125.9 (q, *J* = 3.8 Hz), 124.11, 124.08 (q, *J* = 273.2 Hz), 37.5 HRMS(ESI): $C_{17}H_{11}F_3NO_4$, calcd $[M+H]^+$: 350.0565 found: 350.05659

1,3-dioxoisoindolin-2-yl 2-methyl-2-phenylpropanoate **a7**

According to the published procedures[3], **a7** was obtained from 2-methyl-2-phenylpropanoic acid (0.82g, 5mmol) as a white solid (0.57g, 37%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.79-7.76 (m, 2H), 7.51-7.50 (m, 2H), 7.44-7.41 (m, 2H), 7.34-7.30 (m, 1H), 1.79 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): 173.3, 162.0, 142.7, 134.8, 129.0, 128.8, 127.5, 125.9, 123.9, 46.4, 26.9. HRMS(ESI): $C_{18}H_{16}NO_4$, calcd $[M+H]^+$: 310.1005 found: 310.1001

1,3-dioxoisoindolin-2-yl 1-(p-tolyl)cyclopropane-1-carboxylate **a8**

According to the published procedures[3], **a8** was obtained from 1-(p-tolyl)cyclopropane-1-carboxylic acid (0.88g, 5mmol) as a white solid (0.63g, 39%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86-7.82 (m, 2H), 7.77-7.73 (m, 2H), 7.43-7.41 (m, 2H), 7.19-7.17 (m, 2H), 2.35 (s, 2H), 1.89 (q, *J* = 3.9 Hz, 2H), 1.47 (q, *J* = 3.9 Hz, 2H); ^{13}C NMR (101 MHz,

$CDCl_3$): 171.3, 162.0, 137.8, 134.7, 134.1, 130.5, 129.3, 129.0, 123.9, 27.0, 21.3, 18.9 HRMS(ESI): $C_{19}H_{15}NO_4Na$, calcd $[M+Na]^+$: 344.1000 found: 344.1004

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 3-cyclohexylpropanoate **a10**

According to the published procedures[3], **a10** was obtained from cyclohexanepropionic acid (0.78g, 5mmol) as a white solid (1.67g, 76%). ^1H NMR (400 MHz, $CDCl_3$): δ 2.67-2.63 (m, 2H), 1.75-1.63 (m, 7H), 1.39-1.09 (m, 4H), 0.97-0.87 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 169.5, 157.6, 140.9, 130.4, 124.8, 37.0, 32.9, 32.0, 28.6, 26.5, 26.2 HRMS(ESI): $C_{17}H_{16}Cl_4NO_4$, calcd $[M+H]^+$: 437.9751 found: 437.9747

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl cyclobutanecarboxylate **a13**

According to the published procedures[3], **a13** was obtained from cyclobutanecarboxylicc acid (0.50g, 5mmol) as a white solid (1.74g, 91%). ^1H NMR (400 MHz, $CDCl_3$): δ 3.55-3.47 (m, 1H), 2.55-2.37 (m, 4H), 2.17-2.01 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 157.8, 141.1, 130.6, 124.9, 35.0, 25.5, 18.9 HRMS(ESI): $C_{13}H_8\ Cl_4NO_4$, calcd $[M+H]^+$: 381.9123 found: 381.9128

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenoxypropanoate **a17**

According to the published procedures[3], **a17** was obtained from 2-phenoxypropionic acid (0.83g, 5mmol) as a white solid (0.76g, 34%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.36-7.32 (m, 2H), 7.06-7.02 (m, 1H), 6.99-6.96 (m, 2H), 5.10 (q, *J*=6.8Hz, 1H), 1.86 (d, *J*=6.8Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 168.5, 157.0, 141.3, 130.7, 129.9, 124.7, 122.6, 115.2, 70.9, 19.1 HRMS(ESI): $C_{17}H_{10}\ Cl_4NO_5$, calcd $[M+H]^+$: 447.9241 found: 447.9242

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenylacetate **a18**

According to the published procedures[3], **a18** was obtained from phenylacetic acid (0.68g, 5mmol) as a white solid (0.32g, 43%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.41-7.31 (m, 5H), 3.99 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 167.3, 157.5, 141.2, 131.3, 130.6, 129.4, 129.1, 128.1, 124.8, 37.8 HRMS(ESI): $C_{16}H_8\ Cl_4NO_4$, calcd $[M+H]^+$: 417.9131 found: 417.9127

but-1-yne-1,3-diyldibenzene **c1**

Following the general procedure, **c1** was obtained after flash column chromatography (PE) as colourless oil[3] (33.3mg, 81%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.50-7.46 (m, 3H), 7.40-7.36 (m, 2H), 7.34- 7.28 (m, 4H), 4.02 (q, *J* = 7.1 Hz, 1H), 1.61 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.5, 131.77, 128.7, 128.3, 127.9, 127.1, 126.8, 123.9, 92.7, 82.6, 32.6, 24.7 HRMS (EI^+, 70eV): $C_{16}H_{14}$ $[M]^+$: calcd: 206.1096, found: 206.1100.

4-(3-phenylprop-2-yn-1-yl)-1,1'-biphenyl **c2**

Following the general procedure, **c2** was obtained after flash column chromatography (PE:EA=100:1) as colourless oil (38.1mg, 71%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.62-7.58 (m, 4H), 7.51-7.43 (m, 6H), 7.37-7.30(m, 4H), 3.89 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.1, 139.8, 136.0, 131.8, 128.9, 128.5, 128.4, 128.0, 127.5, 127.3, 127.2, 123.8, 87.6, 82.9, 25.6 HRMS (EI^+, 70eV): $C_{21}H_{16}$ $[M]^+$: calcd:268.1252, found: 268.1242

2-(3-phenylprop-2-yn-1-yl)naphthalene **c3**

Following the general procedure, **c3** was obtained after flash column chromatography (petroleum ether) as colourless oil[4] (35.8mg, 74%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.88

(s, 1H), 7.84-7.82 (m, 3H), 7.53-7.43(m, 5H), 7.33-7.31 (m, 3H), 4.00 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ134.4, 133.7, 132.5, 131.8, 128.4, 128.3, 128.0, 127.8, 126.7, 126.4, 126.3, 125.7, 123.8, 87.6, 83.0, 26.1 HRMS (EI^+, 70eV): $C_{21}H_{16}$ $[M]^+$: calcd: 242.1096, found: 242.1100

1-bromo-2-(3-phenylprop-2-yn-1-yl)benzene **c4**

Following the general procedure, **c4** was obtained after flash column chromatography (petroleum ether) as colourless oil[4] (38.3mg, 71%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70-7.69 (m, 1H), 7.59-7.56 (m, 1H), 7.49-7.46 (m, 2H), 7.35-7.31(m, 4H), 7.16-7.12 (m, 1H), 3.91 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.3, 132.7, 131.8, 129.9, 128.5, 128.4, 128.1, 127.8, 124.0, 123.6, 86.4, 83.8, 26.9 HRMS (EI^+, 70eV): $C_{15}H_{11}Br$ $[M]^+$: calcd: 270.0044, found: 270.0041

1-(3-phenylprop-2-yn-1-yl)-4-(trifluoromethyl)benzene **c5**

Following the general procedure, **c5** was obtained after flash column chromatography (PE:EA=100:1) as colourless oil[4] (30.7, 59%).^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.60 (m, 2H), 7.56-7.54 (m, 2H), 7.48-7.46 (m, 2H), 7.34-7.31 (m, 3H), 3.90 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.0, 131.8, 129.2 (q, *J*=32.5 Hz), 128.5, 128.2, 125.6 (q, *J*=3.8 Hz), 123.4, 86.4, 83.5, 25.8 ^{19}F-NMR (374 MHz, $CDCl_3$):-62.37 (s, 3F). HRMS (EI^+, 70eV): $C_{16}H_{11}F_3$ $[M]^+$: calcd: 200.1565, found: 200.1573

but-1-yne-1,4-diyldibenzene **c6**

Following the general procedure, **c6** was obtained after flash column chromatography (petroleum ether) as colourless oil[5](21.4mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.40 (m, 2H), 7.39-7.35 (m, 2H), 7.33-7.30(m, 4H), 7.28-7.26 (m, 2H), 2.98 (t, *J*= 7.6Hz, 2H), 2.74 (t, *J*= 7.4Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ140.8, 131.7, 128.7, 128.5, 128.3, 127.6, 126.4, 124.0, 89.6, 81.4, 35.3, 21.8 HRMS (EI^+, 70eV): $C_{16}H_{14}$ $[M]^+$: calcd:206.1096, found: 206. 1097

(4-cyclohexylbut-1-yn-1-yl)benzene **c7**

Following the general procedure, **c7** was obtained after flash column chromatography (petroleum ether) as colourless oil[6](27.6mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.38 (m, 2H), 7.28-7.25 (m, 3H), 2.43-2.38 (m, 2H), 1.77-1.64 (m, 5H), 1.56-1.47 (m, 2H), 1.43-1.36(m, 1H), 1.28-1.16 (m, 3H), 0.95-0.87 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ131.7, 128.3, 127.6, 124.3, 90.8, 80.5, 37.0, 36.4, 33.1, 26.8, 26.4, 17.0 HRMS (EI^+, 70eV): $C_{16}H_{20}$ $[M]^+$: calcd: 212.1565, found: 212.1569

(4-cyclopentylbut-1-yn-1-yl)benzene **c8**

Following the general procedure, **c8** was obtained after flash column chromatography (petroleum ether) as colourless oil (23.3, 59%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.38 (m, 2H), 7.30-7.26 (m, 3H), 2.41 (t, *J*= 7.4Hz, 2H), 1.99-1.89 (m, 1H), 1.85-1.77 (m, 2H), 1.65-1.58 (m, 4H), 1.55-1.49 (m, 2H), 1.17-1.08 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 131.7, 128.3, 127.6, 124.2, 90.8, 80.5, 39.6, 35.3, 32.5, 25.3, 18.8 HRMS (EI^+, 70eV): $C_{15}H_{18}$ $[M]^+$: calcd: 198.1409, found: 198.1400

1-(phenylethynyl)adamantine **c9**

Following the general procedure, **c9** was obtained after flash column chromatography (petroleum ether) as colourless oil (30.1mg, 0.57%). ^{1}H NMR (400 MHz, $CDCl_3$): δ7.42-7.40 (m, 2H), 7.31-7.26 (m, 3H), 2.16 (s, 2H), 2.00 (s, 3H), 1.74-1.64 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 131.7, 128.3, 127.5, 124.4, 88.0, 82.8, 42.3, 37.1, 34.8,

33.2, 28.8 HRMS (EI$^+$, 70eV): $C_{19}H_{22}$ [M]$^+$: calcd: 250.1722, found: 250.1729

(cyclobutylethynyl)benzene **c10**

Following the general procedure, **c10** was obtained after flash column chromatography (petroleum ether) as colourless oil (20.0mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.37 (m, 2H), 7.30-7.25 (m, 3H), 3.27-3.19 (m, 1H), 2.37-2.29 (m, 2H), 2.27-2.17 (m, 2H), 2.02-1.88 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ131.7, 128.3, 127.6, 124.1, 94.1, 81.3, 30.2, 25.7, 19.4 HRMS (EI$^+$, 70eV): $C_{12}H_{12}$ [M]$^+$: calcd: 156.0939, found: 156.0941

(cyclopentylethynyl)benzene **c11**

Following the general procedure, **c11** was obtained after flash column chromatography (petroleum ether) as colourless oil[7](22.4mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.40 (m, 2H), 7.33-7.27 (m, 3H), 2.89-2.82 (m, 1H), 2.06-1.99 (m, 2H), 1.83-1.69 (m, 4H), 1.68-1.61 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.0, 131.8, 129.2 (q, *J*=32.5 Hz), 128.5, 128.2, 125.6 (q, *J*=3.8 Hz), 123.4, 86.4, 83.5, 25.8 HRMS (EI$^+$, 70eV): $C_{13}H_{14}$ [M]$^+$: calcd: 170.1096, found: 170.1094

(cyclohexylethynyl)benzene **c12**

Following the general procedure, **c12** was obtained after flash column chromatography (petroleum ether) as colourless oil[5](23.6mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.38 (m, 2H), 7.30-7.23 (m, 3H), 2.62-2.56(m, 1H), 1.90-1.86 (m, 2H), 1.78-1.73 (m, 2H), 1.58-1.51(m, 4H), 1.36-1.33 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ131.7, 128.3, 127.5, 124.3, 94.6, 80.6, 32.9, 29.8, 26.1, 25.1 HRMS (EI$^+$, 70eV): $C_{14}H_{16}$ [M]$^+$: calcd:184.1247, found: 184.1247

(3-ethylhept-1-yn-1-yl)benzene **c13**

Following the general procedure, **c13** was obtained after flash column chromatography (petroleum ether) as colourless oil (22.4mg, 60%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.38 (m, 2H), 7.30-7.24 (m, 3H), 2.50-2.43 (m, 1H), 1.62-1.51 (m, 5H), 1.46-1.32 (m, 3H), 1.06 (t, *J*= 7.4 Hz, 3H), 0.93 (t, *J*= 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 131.7, 128.3, 127.5, 124.4, 93.9, 81.9, 34.7, 34.2, 29.9, 28.3, 22.8, 14.2, 12.0 HRMS (EI$^+$, 70eV): $C_{15}H_{20}$ [M]$^+$: calcd: 200.1565, found: 200.1573

(3-phenoxybut-1-yn-1-yl)benzene **c14**

Following the general procedure, **c14** was obtained after flash column chromatography (PE:EA=50:1) as colourless oil[8](30.6mg, 69%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.39 (m, 2H), 7.34-7.27 (m, 5H), 7.09-7.08 (m, 2H), 7.01-6.97 (m, 1H), 5.10 (q, *J*= 6.5 Hz, 1H), 1.75 (d, *J*= 6.4 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.7, 131.9, 129.5, 128.6, 128.4, 122.6, 121.4, 116.0, 88.5, 85.8, 64.4, 22.5. HRMS (EI$^+$, 70eV): $C_{16}H_{14}O$ [M]$^+$: calcd: 222.1045, found: 222.1047

4-methoxy-4'-(4-phenylbut-3-yn-2-yl)-1,1'-biphenyl **c15**

Following the general procedure, **c15** was obtained after flash column chromatography (PE:EA=50:1) as colourless oil (40.0mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.74-7.72 (m, 2H), 7.55-7.53 (m, 1H), 7.48-7.46 (m, 2H), 7.32-7.29 (m, 3H), 7.16-7.13 (m, 2H), 4.12 (q, *J*= 7.2Hz, 1H), 3.92 (s, 3H), 1.65 (d, *J*= 7.2Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 157.6, 138.6, 133.6, 131.8, 129.4, 129.1, 128.4, 127.9, 127.3, 126.2, 125.1, 123.9, 119.0, 105.8, 92.9, 82.7, 55.4, 32.6, 24.5 HRMS (EI$^+$, 70eV): $C_{21}H_{18}O$ [M]$^+$: calcd: 286.1358, found: 286.1362

4-(3-methyl-3-phenylbut-1-yn-1-yl)-1,1'-biphenyl **c16**

Following the general procedure, **c16** was obtained after flash column chromatography (PE:EA=100:1) as colourless oil(45.0mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68-7.66 (m, 2H), 7.63-7.61 (m, 2H), 7.59-7.54 (m, 4H), 7.49-7.45(m, 2H), 7.41-7.36 (m, 3H), 7.30-7.26 (m, 1H), 1.73 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ147.1, 140.64, 140.56, 132.2, 129.0, 128.4, 127.6, 127.1, 127.0, 126.6, 125.8, 122.9, 97.4, 82.0, 36.6, 31.9 HRMS (EI^+, 70eV): $C_{23}H_{20}$ $[M]^+$: calcd: 296.1565, found: 296.1564

4-((1-(p-tolyl)cyclopropyl)ethynyl)-1,1'-biphenyl **c17**

Following the general procedure, **c17** was obtained after flash column chromatography (petroleum ether) as colourless oil (38.5mg, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.59 (m, 2H), 7.55-7.50 (m, 4H), 7.47-7.43 (m, 2H), 7.38-7.31 (m, 3H), 7.15-7.13 (m, 2H), 2.35 (m, 3H), 1.56-1.54 (m, 2H), 1.35-1.33 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.6, 140.6, 139.0, 135.8, 132.2, 129.2, 129.0, 127.6, 127.1, 127.0, 125.8, 122.9, 95.0, 78.1, 21.1, 20.4, 16.2 HRMS (EI^+, 70eV): $C_{24}H_{20}$ $[M]^+$: calcd: 308.1565, found: 308.1570

1-chloro-2-(3-phenylbut-1-yn-1-yl)benzene **c18**

Following the general procedure, **c18** was obtained after flash column chromatography (petroleum ether) as colourless oil(31.7mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.46 (m, 3H), δ 7.41-7.34 (m, 3H), 7.28-7.17 (m, 3H), 4.05 (q, J = 7.07 Hz, 1H), 1.62 (d, J = 7.20 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.1, 136.1, 133.4, 129.3, 128.9, 128.7, 127.1, 126.9, 126.5, 123.7, 98.2, 79.6, 32.9, 24.7 HRMS (EI^+, 70eV): $C_{13}H_{16}Cl$ $[M]^+$: calcd: 240.0700, found: 240.0702

1-methyl-3-(3-phenylbut-1-yn-1-yl)benzene **c19**

Following the general procedure, **c19** was obtained after flash column chromatography (petroleum ether) as colourless oil[4](35.6mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.45 (m, 2H), 7.37-7.33 (m, 2H), 7.28-7.23 (m, 3H), 7.19 (t, J = 7.6 Hz, 1H), 7.11-7.09 (m, 1H), 3.98 (q, J = 7.1 Hz, 1H), 2.32 (s, 3H), 1.58 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.5, 138.0, 132.4, 128.8 (d, J = 3.5 Hz), 128.7, 128.2, 127.1, 126.8, 123.7, 92.4, 82.7, 32.6, 24.7, 21.3 HRMS (EI^+, 70eV): $C_{17}H_{16}$ $[M]^+$: calcd: 220.1247, found: 220.1246

4-(3-phenylbut-1-yn-1-yl)-1,1'-biphenyl **c20**

Following the general procedure, **c20** was obtained after flash column chromatography (petroleum ether) as colourless oil (44.6mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.58 (m, 2H), 7.55-7.50 (m, 4H), 7.49-7.42 (m, 4H), 7.38-7.34 (m, 2H), 7.28-7.24 (m, 2H), 4.01 (q, J = 7.1 Hz, 1H), 1.60 (d, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.5, 140.6, 132.2, 129.0, 128.7, 127.7, 127.14, 127.09, 127.04, 126.8, 122.8, 93.5, 82.4, 32.7, 24.7 HRMS (EI^+, 70eV): $C_{17}H_{16}$ $[M]^+$: calcd: 220.1247, found: 220.1246

1-chloro-4-(3-phenylprop-1-yn-1-yl)benzene **c21**

Following the general procedure, **c21** was obtained after flash column chromatography (petroleum ether) as colourless oil[9](31.6mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.35 (m, 6H), 7.30-7.28 (m, 3H), 3.84(s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.6, 133.9, 133.0, 128.75, 128.70, 128.1, 126.9, 122.3, 88.8, 81.7, 25.9. HRMS (EI^+, 70eV): $C_{15}H_{11}Cl$ $[M]^+$: calcd: 226.0549, found: 226.0546

1-bromo-4-(3-phenylprop-1-yn-1-yl)benzene **c22**

Following the general procedure, **c22** was obtained after flash column chromatography (petroleum ether) as colourless oil[10] (34.6mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.35 (m, 6H), 7.30-7.28 (m, 3H), 3.84(s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.6, 133.2, 131.6, 128.7, 128.1, 126.9, 122.7, 122.1, 89.0, 81.7, 25.9 HRMS (EI^+, 70eV): $C_{15}H_{11}Br$ $[M]^+$: calcd: 270.0044, found: 270.0042

6-methoxy-1-(3-phenylbut-1-yn-1-yl) naphthalene **c23**

Following the general procedure, **c23** was obtained after flash column chromatography (PE:EA=50:1) as colourless oil (48.0mg, 84%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89(s, 1H), 7.67 (t, *J*=8.6Hz, 2H), 7.51-7.46 (m, 3H), 7.39-7.35 (m, 2H), 7.28-7.24 (m, 1H), 7.16-7.10 (m, 2H), 4.03(q, *J*=7.1Hz, 1H), 1.62 (d, *J*=7.2Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 158.2, 143.6, 134.0, 131.2, 129.4, 129.3, 128.7, 128.6, 127.1, 126.801, 126.785, 119.4, 118.8, 105.9, 92.3, 82.9, 55.5, 32.7, 24.7 HRMS (EI^+, 70eV): $C_{22}H_{18}O$ $[M]^+$: calcd: 286.1352, found: 286.1361

4-(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)benzonitrile **c24**

Following the general procedure, **c24** was obtained after flash column chromatography (PE:EA=30:1) as colourless oil (20.5mg, 35%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.56 (m, 6H), 7.54-7.52 (m, 2H), 7.47-7.40 (m, 5H), 7.37-7.33 (m, 1H), 3.90 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.9, 140.1, 135.1, 132.4, 132.1, 129.0, 128.8, 128.5, 127.6, 127.5, 127.2, 118.7, 111.4, 92.7, 81.5, 25.7. HRMS (EI^+, 70eV): $C_{22}H_{15}N$ $[M]^+$: calcd: 239.1204 found: 239.1209

4-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1,1'-biphenyl **c25**

Following the general procedure, **c25** was obtained after flash column chromatography (petroleum ether) as colourless oil(53.8mg, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.56 (m, 4H), 7.50-7.40 (m, 6H), 7.37-7.32 (m, 3H), 3.88 (s, 2H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 151.2, 141.1, 139.8, 136.2, 131.5, 128.9, 128.5, 127.4, 127.3, 127.2, 125.4, 120.7, 86.8, 82.9, 34.9, 31.3, 25.6 HRMS (EI^+, 70eV): $C_{25}H_{24}$ $[M]^+$: calcd: 324.1878, found: 324.1800

2-(3-phenylbut-1-yn-1-yl)thiophene **c26**

Following the general procedure, **c26** was obtained after flash column chromatography (PE:EA=40:1) as colourless oil[11] (27.3mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.42 (m, 2H), 7.37-7.33 (m, 2H), 7.27-7.24 (m, 1H), 7.21-7.17 (m, 2H), 6.97-6.94 (m, 1H), 4.09 (q, *J* = 7.1Hz, 1H), 1.58 (d, *J* = 7.2Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.1, 131.4, 128.7, 127.1, 126.95, 126.90, 126.4, 123.9, 96.6, 75.6, 32.9, 24.4 HRMS (EI^+, 70eV): $C_{14}H_{12}S$ $[M]^+$: calcd: 212.0660, found: 212.0665

3-(3-phenylbut-1-yn-1-yl)pyridine **c27**

Following the general procedure, **c27** was obtained after flash column chromatography (PE:EA=20:1) as colourless oil[12](29.8mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.57-8.56 (m, 2H), 7.62 (td, *J* = 7.6, 1.6Hz, 1H), 7.47-7.40 (m, 3H), 7.36-7.33 (m, 2H), 7.27-7.18 (m, 2H), 4.02 (q, *J* = 7.1Hz, 1H), 1.62 (d, *J* = 7.2Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 150.0, 143.8, 142.7, 136.2, 128.8, 127.2, 127.1, 126.9, 122.6, 93.1, 82.1, 32.5, 24.2 HRMS (EI^+, 70eV): $C_{15}H_{13}N$ $[M]^+$: calcd: 207.1048, found: 207.1039

4-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-1,1'-biphenyl **c28**

Following the general procedure, **c28** was obtained after flash column chromatography (PE:EA=50:1) as colourless oil (46.5mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-

7.55(m, 4H), 7.53-7.40 (m, 5H), 7.36-7.33 (m, 1H), 6.85-6.83 (m, 2H), 3.87 (s, 2H), 3.81(s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.4, 141.1, 139.8, 136.2, 133.2, 128.9, 128.5, 127.4, 127.3, 127.2, 115.9, 114.0, 86.0, 82.6, 55.4, 25.6 HRMS (EI$^+$, 70eV): $C_{22}H_{18}O$ $[M]^+$: calcd: 298.1358, found: 298.1361.

(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)trimethylsilane **c29**

Following the general procedure, **c29** was obtained after flash column chromatography (petroleum ether) as colourless oil (36.9mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.55 (m, 4H), 7.46-7.41 (m, 4H), 7.36-7.33 (m, 1H), 3.70 (s, 1H), 0.21 (m, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.0, 139.8, 135.6, 128.9, 128.4, 127.39, 127.33, 127.2, 104.3, 87.1, 26.0, 0.3 HRMS (EI$^+$, 70eV): $C_{18}H_{20}Si$ $[M]^+$: calcd:263.1326, found: 263.1334

(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)triethylsilane **c30**

Following the general procedure, **c30** was obtained after flash column chromatography (petroleum ether) as colourless oil (45.2mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.57 (m, 4H), 7.47-7.43 (m, 4H), 7.37-7.33 (m, 1H), 3.75 (s, 1H), 1.05 (t, *J*= 8.0Hz, 9H), 0.66 (t, *J* = 7.9Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.0, 139.7, 135.8, 128.9, 128.4, 127.33, 127.30, 127.2, 105.3, 84.5, 26.0, 7.7, 4.7 HRMS (EI$^+$, 70eV): $C_{21}H_{26}Si$ $[M]^+$: calcd:306.1809, found: 306.1804

(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)triisopropylsilane **c31**

Following the general procedure, **c31** was obtained after flash column chromatography (petroleum ether) as colourless oil (52.2mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.57 (m, 4H), 7.48-7.43 (m, 4H), 7.37-7.33 (m, 1H), 3.76 (s, 1H), 1.13-1.12 (m, 21H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.0, 139.6, 136.0, 128.9, 128.4, 127.3, 127.2, 105.7, 83.2, 26.1, 18.8, 11.5 HRMS (EI$^+$, 70eV): $C_{24}H_{32}Si$ $[M]^+$: calcd:348.2273, found: 348.2280

4-(5-methylhex-2-yn-1-yl)-1,1'-biphenyl **c32**

Following the general procedure, **c32** was obtained after flash column chromatography (petroleum ether) as colourless oil (31.2mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.54 (m, 4H), 7.46-7.42 (m, 4H), 7.36-7.32 (m, 1H), 3.64 (t, *J*=2.0 Hz, 1H), 2.16-2.13 (m, 2H), 1.89-1.79 (m, 1H), 1.01 (d, *J* = 6.4Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.1, 139.5, 136.9, 128.9, 128.4, 127.31, 127.26, 127.2, 81.8, 78.4, 28.4, 28.3, 25.0, 22.2 HRMS (EI$^+$, 70eV): $C_{19}H_{20}$ $[M]^+$: calcd: 248.1565, found: 248.1564

4-(hex-2-yn-1-yl)-1,1'-biphenyl **c33**

Following the general procedure, **c33** was obtained after flash column chromatography (petroleum ether) as colourless oil (32.8mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61-7.55 (m, 4H), 7.47-7.43 (m, 4H), 7.37-7.33(m, 1H), 3.65 (t, *J*= 2.0Hz, 1H), 2.26-2.22 (m, 2H), 1.64-1.55 (m, 2H), 1.03 (t, *J*= 7.4Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.1, 139.5, 136.9, 128.9, 128.4, 127.31, 127.25, 127.2, 82.8, 77.7, 25.0, 22.6, 21.0, 13.7 HRMS (EI$^+$, 70eV): $C_{18}H_{18}$ $[M]^+$: calcd:234.1409, found: 234.1414

4-(hept-2-yn-1-yl)-1,1'-biphenyl **c34**

Following the general procedure, **c34** was obtained after flash column chromatography (petroleum ether) as colourless oil (33.7mg, 68%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.54 (m, 4H), 7.46-7.42 (m, 4H), 7.36-7.32 (m, 1H), 3.63 (t, *J*= 2.0Hz, 1H), 2.28-2.23 (m, 2H), 1.58-1.43 (m, 4H), 0.94 (t, *J*= 7.2Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$):

δ141.1, 139.6, 136.9, 128.8, 128.4, 127.32, 127.26, 127.2, 82.9, 77.5, 31.3, 25.0, 22.2, 18.7, 13.8 HRMS (EI$^+$, 70eV): $C_{19}H_{20}$ [M]$^+$: calcd:248.1565, found: 248.1570

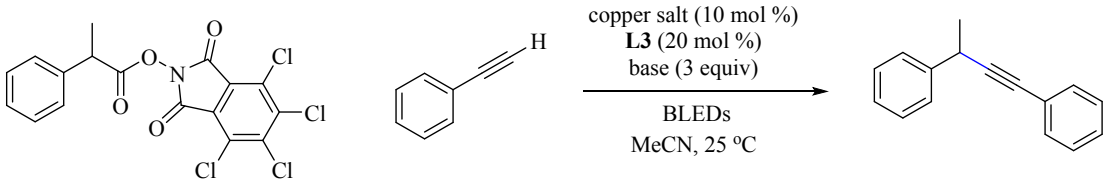
4-(oct-2-yn-1-yl)-1,1'-biphenyl **c35**

Following the general procedure, **c35** was obtained after flash column chromatography (petroleum ether) as colourless oil (39.3mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.54 (m, 4H), 7.45-7.41 (m, 4H), 7.35-7.32 (m, 1H), 3.63 (t, *J*=2.2 Hz, 1H), 2.26-2.21 (m, 2H), 1.59-1.52 (m, 2H), 1.44-1.34 (m, 4H), 0.91 (t, *J* = 7.0Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.2, 139.6, 136.9, 128.9, 128.4, 127.32, 127.27, 127.2, 83.0, 77.6, 31.3, 28.9, 25.0, 22.4, 19.0, 14.2 HRMS (EI$^+$, 70eV): $C_{20}H_{22}$ [M]$^+$: calcd: 262.1722, found: 262.1724

4-(pentadec-2-yn-1-yl)-1,1'-biphenyl **c36**

Following the general procedure, **c36** was obtained after flash column chromatography (PE:EA=50:1) as colourless oil (53.3mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.54 (m, 4H), 7.46-7.42 (m, 4H), 7.36-7.32(m, 1H), 3.63 (t, *J*= 2.0Hz, 1H), 2.26-2.22 (m, 2H), 1.58-1.51 (m, 2H), 1.43-1.38(m, 2H), 1.29-1.27 (m, 16H), 0.88 (t, *J*= 6.8Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ141.1, 139.5, 136.9, 128.9, 128.4, 127.31, 127.26, 127.2, 83.0, 77.5, 32.1, 29.85, 29.81, 29.8, 29.7, 29.5, 29.3, 29.1, 25.0, 22.8, 19.0, 14.3 HRMS (EI$^+$, 70eV): $C_{27}H_{36}$ [M]$^+$: calcd:360.2817, found: 360.2820

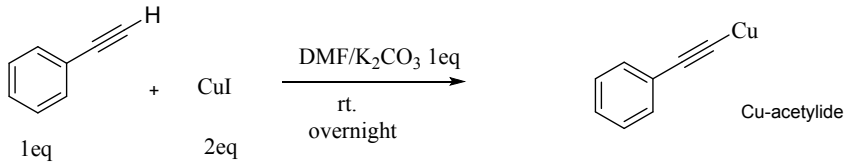
(4)Gram-Scale Reaction



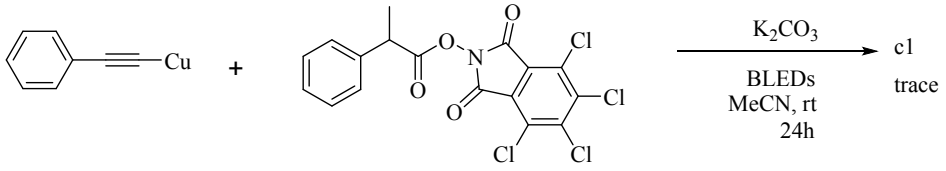
Under nitrogen atmosphere, 2, 2′, 6′, 2′′-terpyridine (1.6 mmol, 20 mol %), CuI (0.8 mmol, 10 mol %), K_2CO_3 (24 mmol, 3.0 equiv), alkyne (8.0 mmol, 1 equiv), and N-acyloxyl derivatives (16mmol, 2.0 equiv) were added in a dried reaction vessel with 30 mL of MeCN. After 48h, the reaction was quenched with ice water and extracted with DCM, then dried over anhydrous Na_2SO_4 and concentrated. The crude product was purified by column chromatography. (1.2g, 73%)

3 Mechanistic Investigations

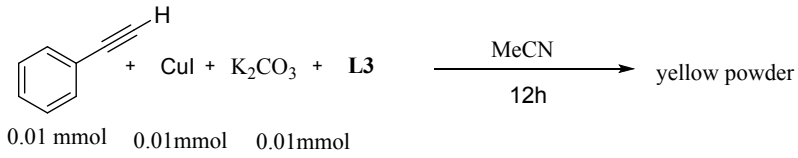
(1) Considerations of the reaction's active intermediate[13]



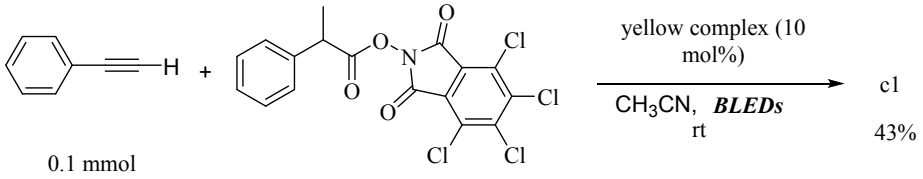
(1)



(2)



(3)



(4)

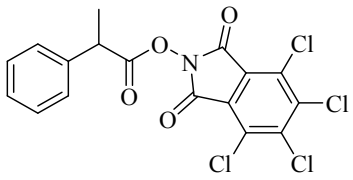
phenylethynylcopper was obtained according to literature (eq 1).

Control experiment (eq 2): under our standard conditions, stoichiometry (phenylethynyl)copper or catalytic amount (phenylethynyl)copper, ethynylbenzene (0 mmol or 0.09 mmol), **a1** (0.2 mmol, 2.0 equiv) and K_2CO_3 (0.3 mmol, 3.0 equiv) were added sequentially for 24h. After completion, Trace product was obtained.

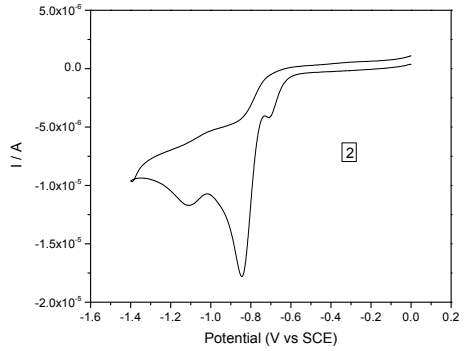
We prepared the Cu-tpy-alkyne complex and isolated it as a crude yellow powder (eq 3). The yellow complex as the catalyst catalyzed the reaction in 43% yield (eq 4). Thus, Cu-tpy-alkyne complex played a role of catalyst for this reaction.

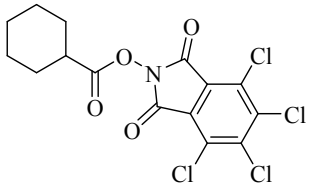
(2) Cyclic Woltammetry analysis[13]

Cyclic Woltammetry analysis was performed by Shanghai Chen Hua CHI660D. Pt electrode(area=0.03 cm^2), and Pt sheet were working electrode and auxiliary electrode, respectively. A saturated calomel eleetrode (SCE) was reference electrode. Tetrabutylammonium hexafluorophosphata (nBu_4NPF_6) was supporting electrolyte and electrolysis was conducted at room temperature. 0.1mol substrate was added in 0.1M solution of nBu_4NPF_6 (10mL MeCN). The reduction potentials of selected TCNHPI esters of primary, secondary, tertiary and acids were exhibited. However, the reduction potential of ligand-Cu-acetylide complex in suite genernated is difficult to test. Based on the report[13], ligand-Cu-acetylide complex have higher reduction potential.

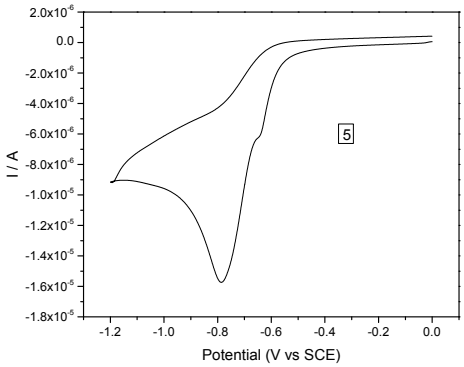


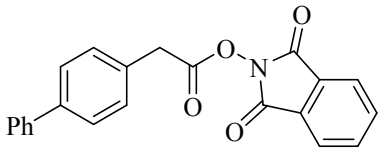
$E_{1/2}$ = –0.79 V vs SCE



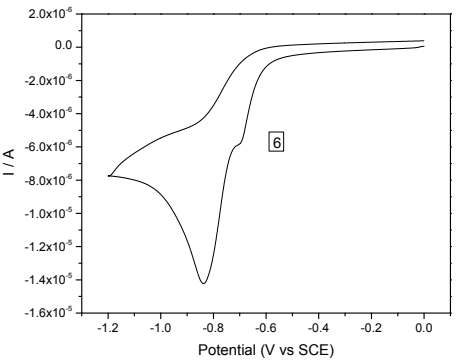


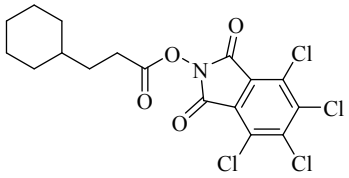
$E_{1/2}$ = –0.73 V vs SCE



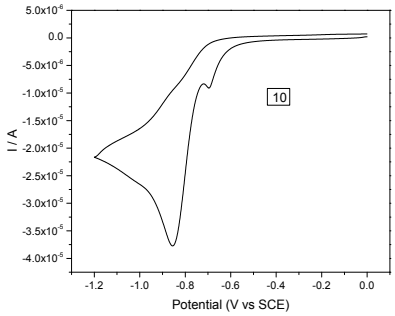


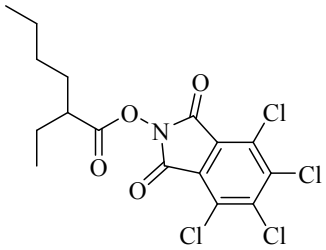
$E_{1/2}$ = –0. 77 V vs SCE



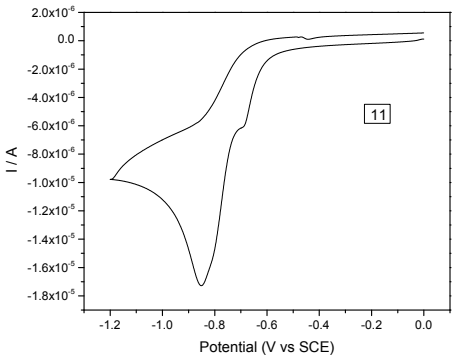


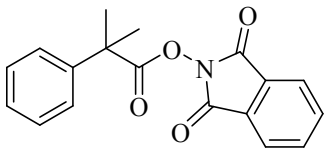
$E_{1/2}$ = –0.79 V vs SCE



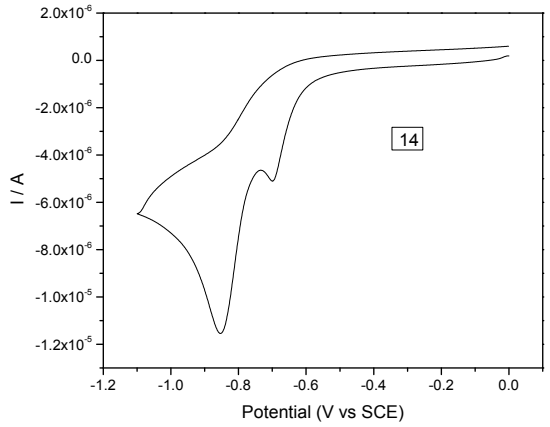


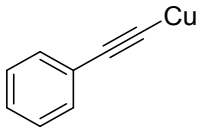
$E_{1/2}$ = –0.78 V vs SCE



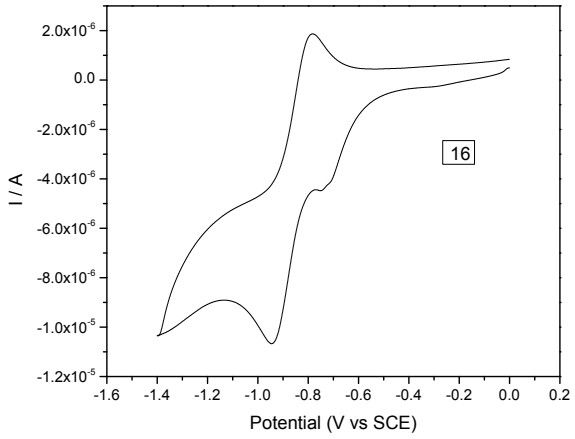


$E_{1/2}$ = –0.80 V vs SCE



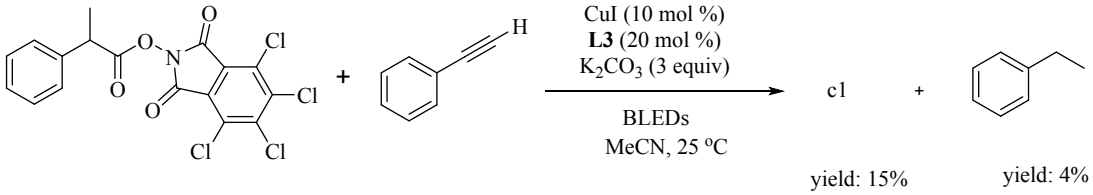


$E_{1/2}$ = –0.86 V vs SCE



(3) **Quantum Yield Measurement**[14]

The quantum yield measurement of the photoinduced reaction was performed based on our previous publication. The cuvette placed 5cm away from blue LED lamps and the incident area was 1 cm×1 cm[4].



Prepared following the general procedure showed above and the reaction mixture was stirred under Blue LED for 30min. After completion, the reaction was quenched and the yield of crud product was determined by ^{1}H NMR. Diethyl phthalate was internal standard.

The photon flux was 379 mW (average of three experiments).

Photon flux= $\frac{P}{N_A \cdot hc/\lambda} = \frac{379.0 \times 10^{-3}}{6.02 \times 10^{23} \times 6.63 \times 10^{-34} \times 3 \times 10^{8} / (400 \times 10^{-9})} = 1.3 \times 10^{-6}$ einstein • s^{-1} (1)

QY calculated by the equation (2). T is the reaction time (30~60s).QY was Φ=0.8%.

$$\Phi = \frac{mol\ product}{photon\ flux \cdot t \cdot f} = \frac{1.9 \times 10^{-5}}{1.3 \times 10^{-6} \times 30 \times 60} = 0.8\ \%. \quad (2)$$

4 References

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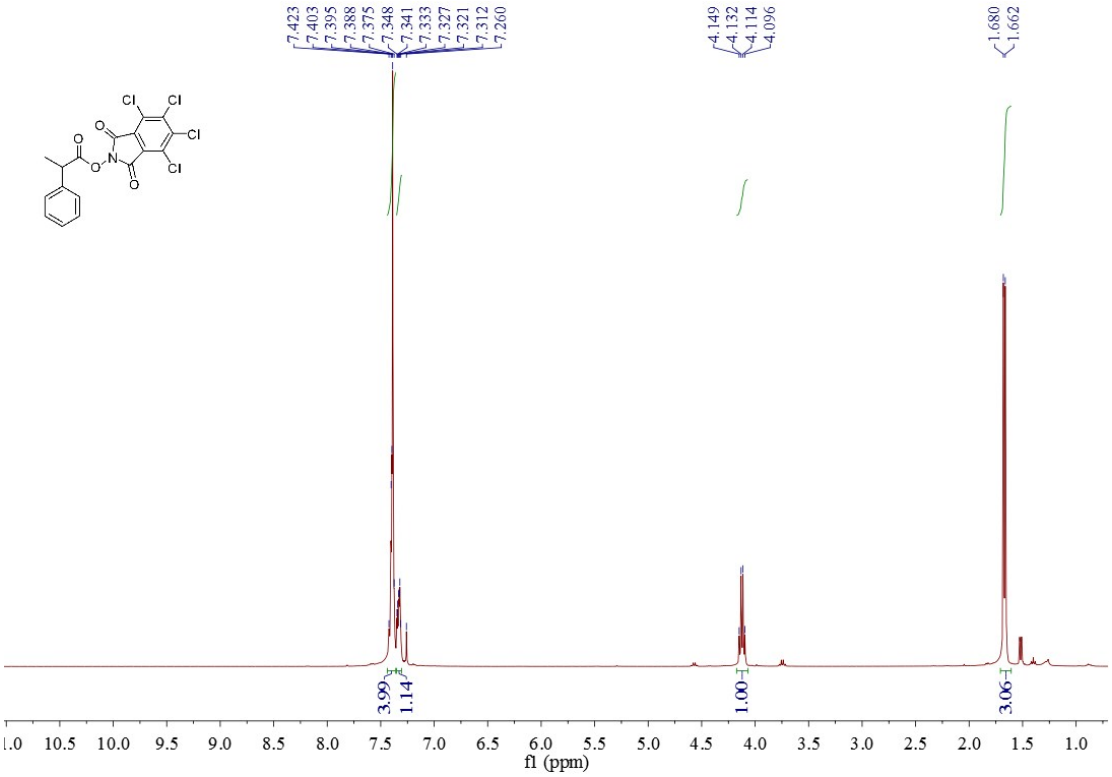
[13] See references 16a, 16b of manuscript

[14] L. Zhang, L. Jiao, Visible-Light-Induced Organocatalytic Borylation of Aryl Chlorides *J.Am.Chem.Soc*. **2019**, 141, 9124-9128.

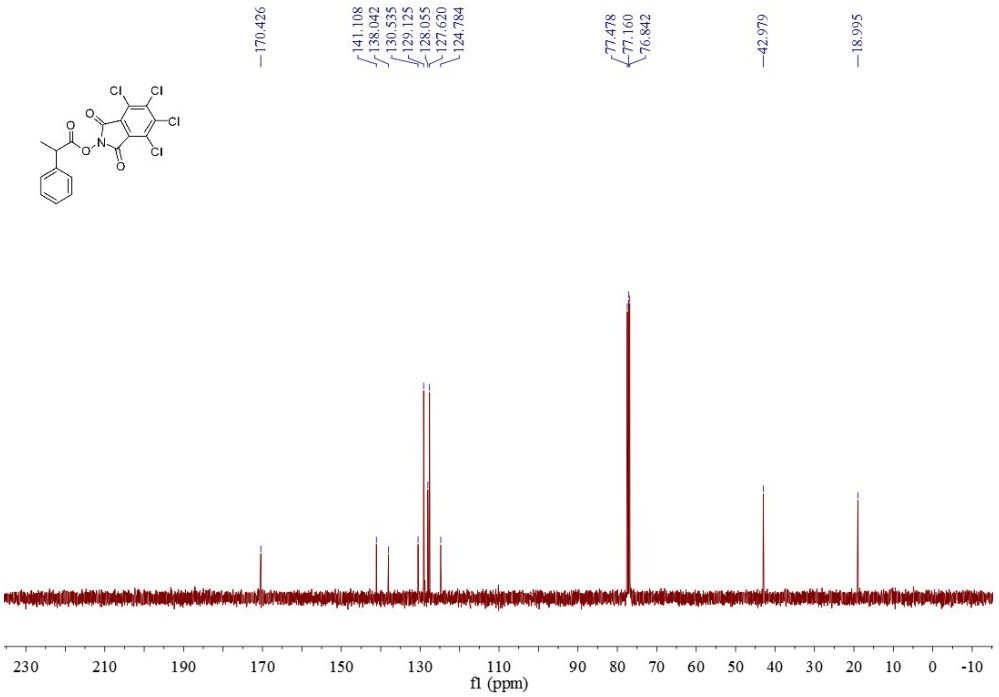
5 Copies of NMR Spectra

4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenylpropanoate **a1**

^{1}H-NMR

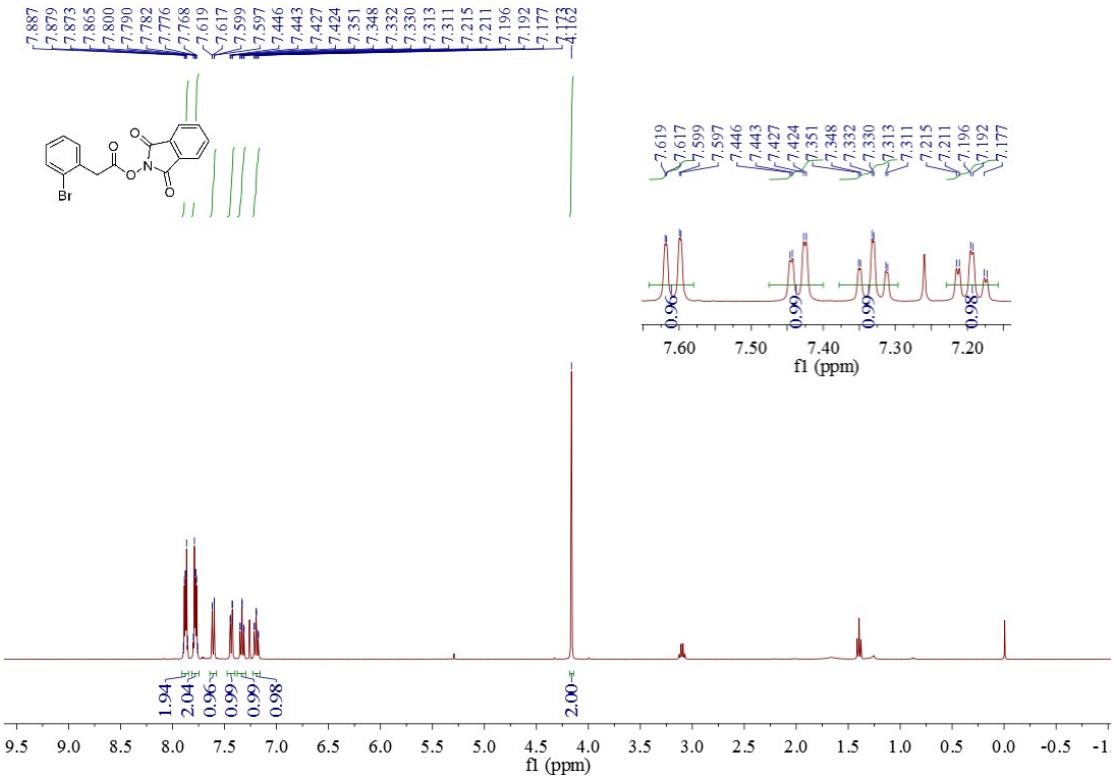


^{13}C-NMR



1,3-dioxoisoindolin-2-yl 2-(2-bromophenyl)acetate **a4**

[1]H-NMR



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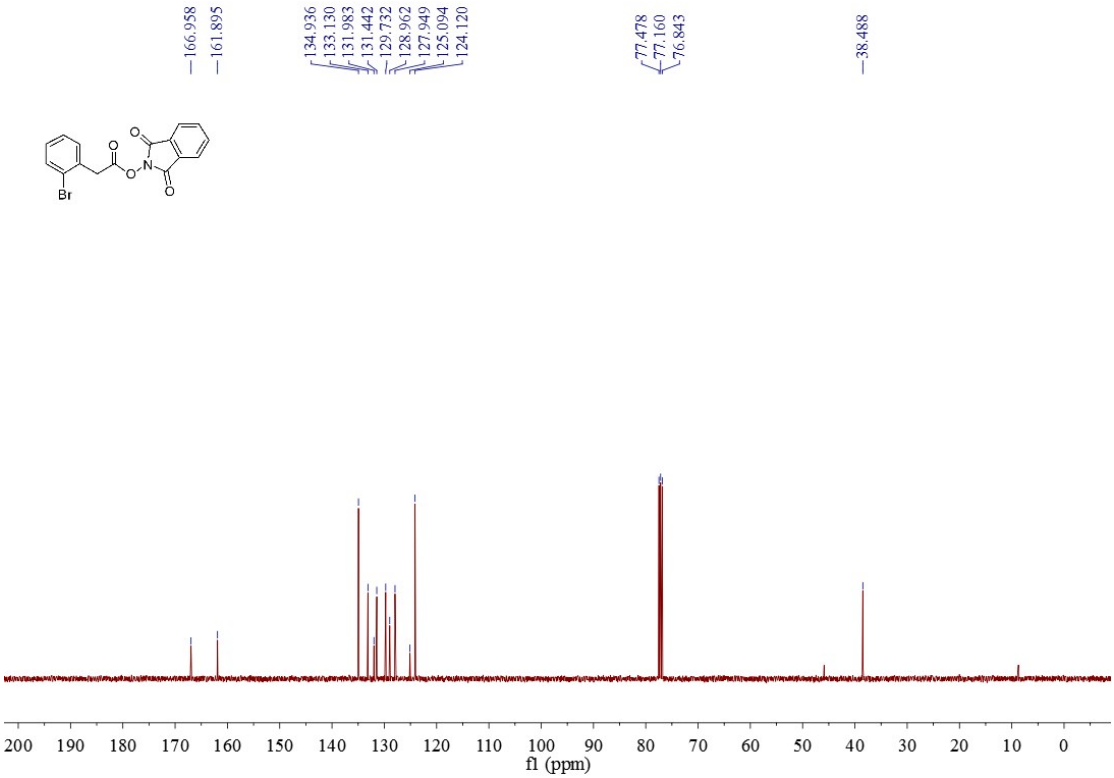
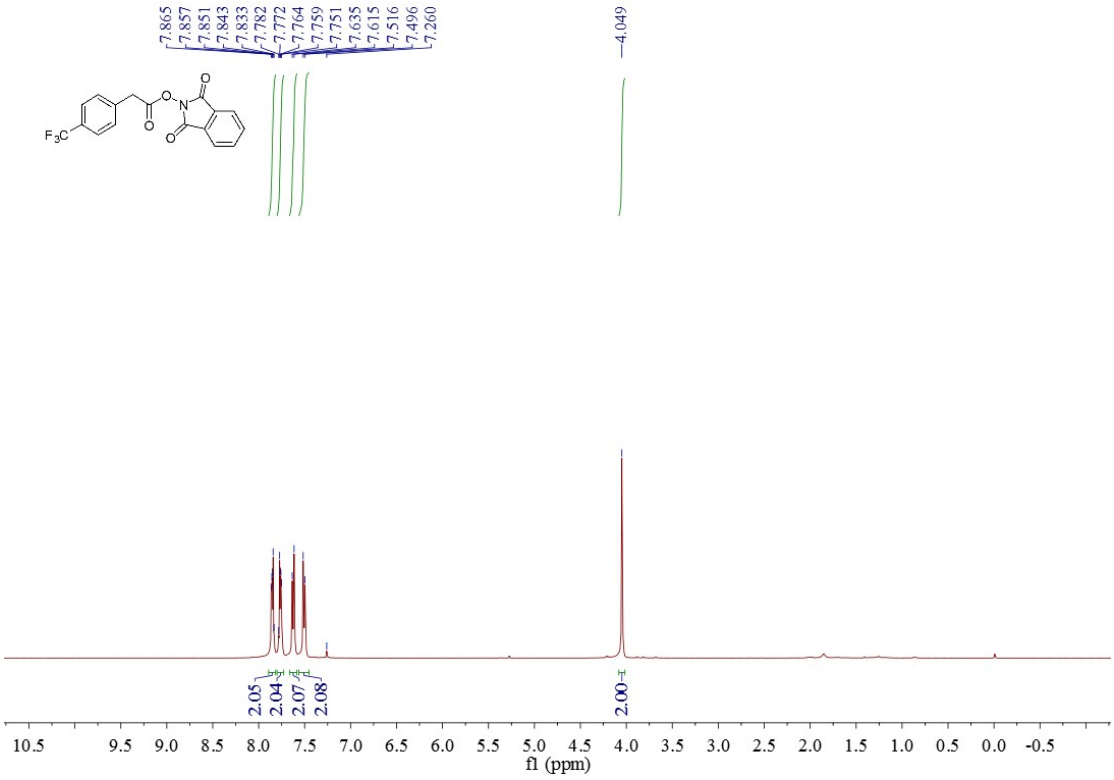


1,3-dioxoisoindolin-2-yl 2-(4-(trifluoromethyl)phenyl)acetate **a5**

^{1}H-NMR



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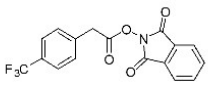

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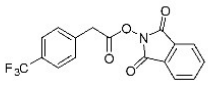
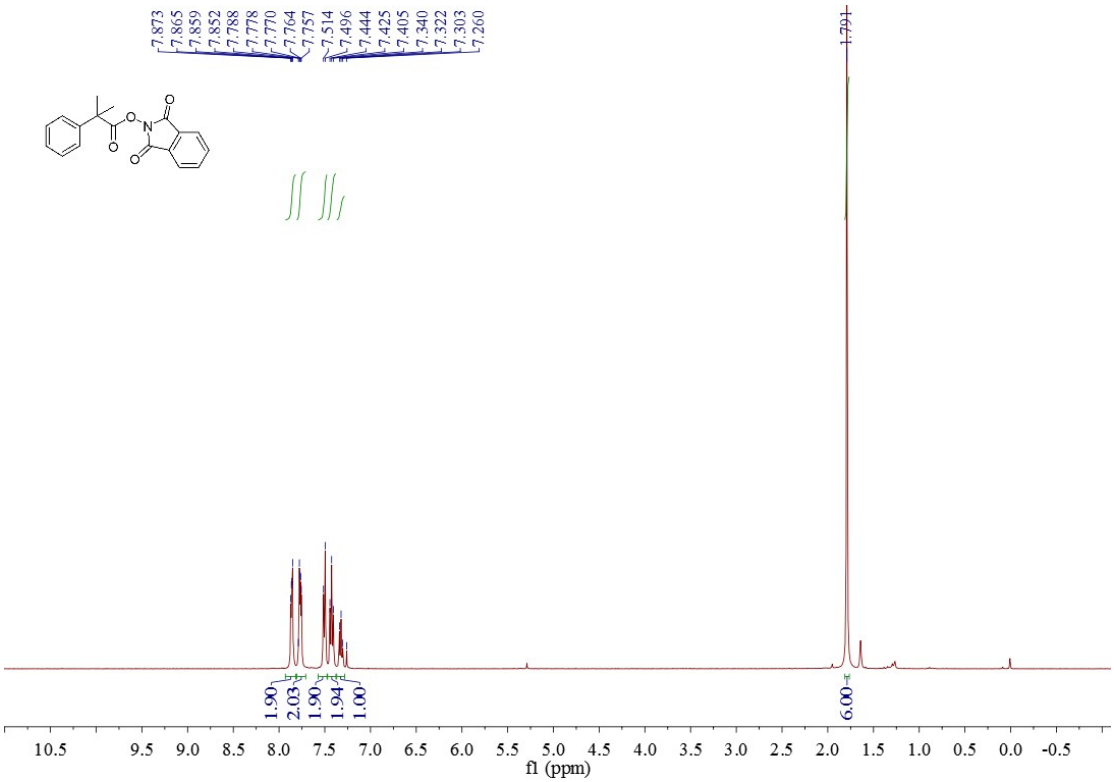


1,3-dioxoisoindolin-2-yl 2-methyl-2-phenylpropanoate **a7**

^{1}H-NMR



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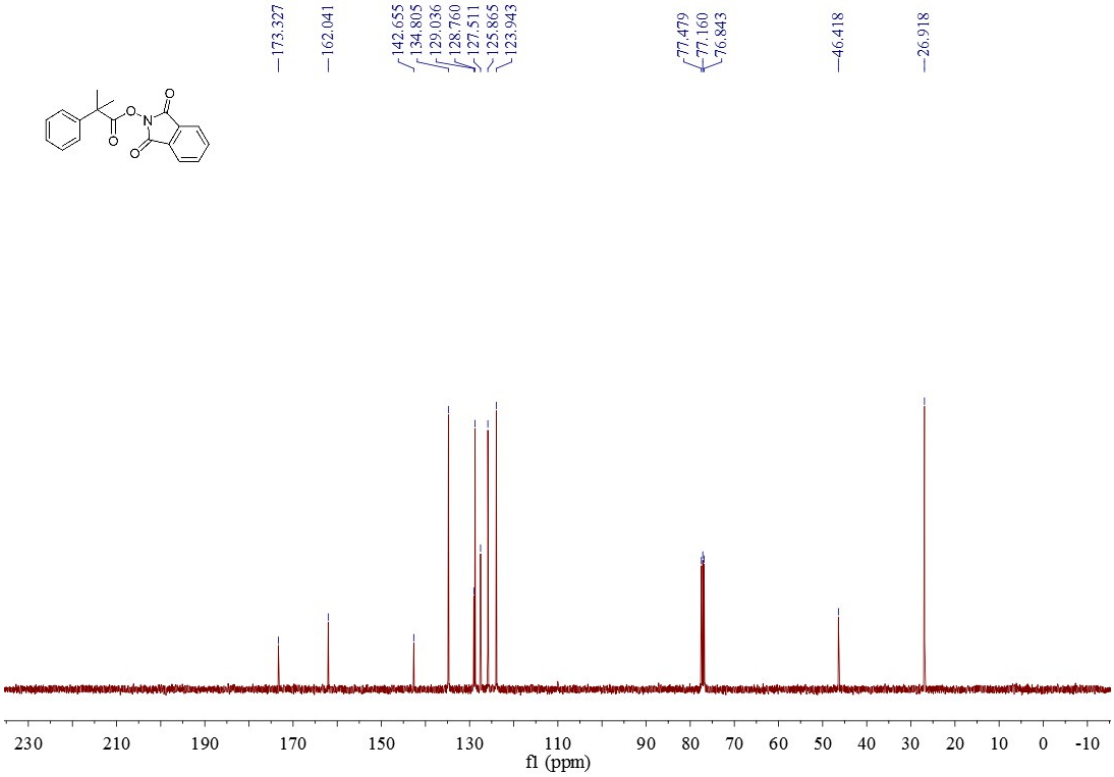
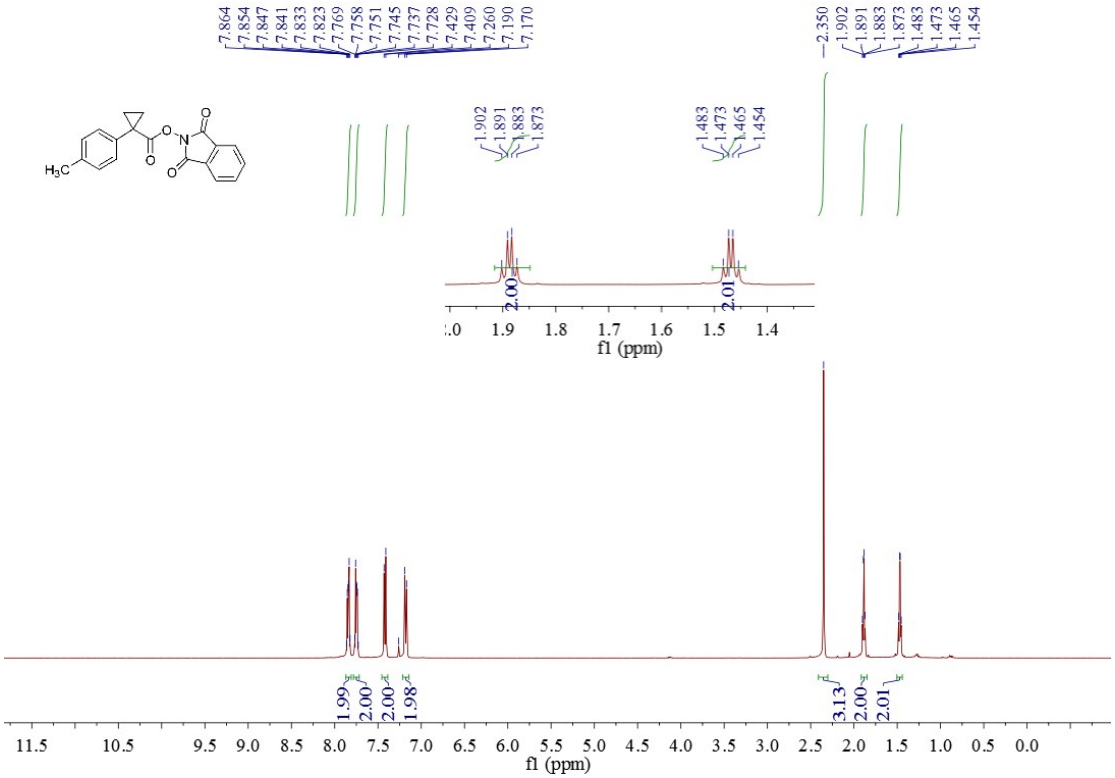


1,3-dioxoisoindolin-2-yl 1-(p-tolyl)cyclopropane-1-carboxylate **a8**

^{1}H-NMR



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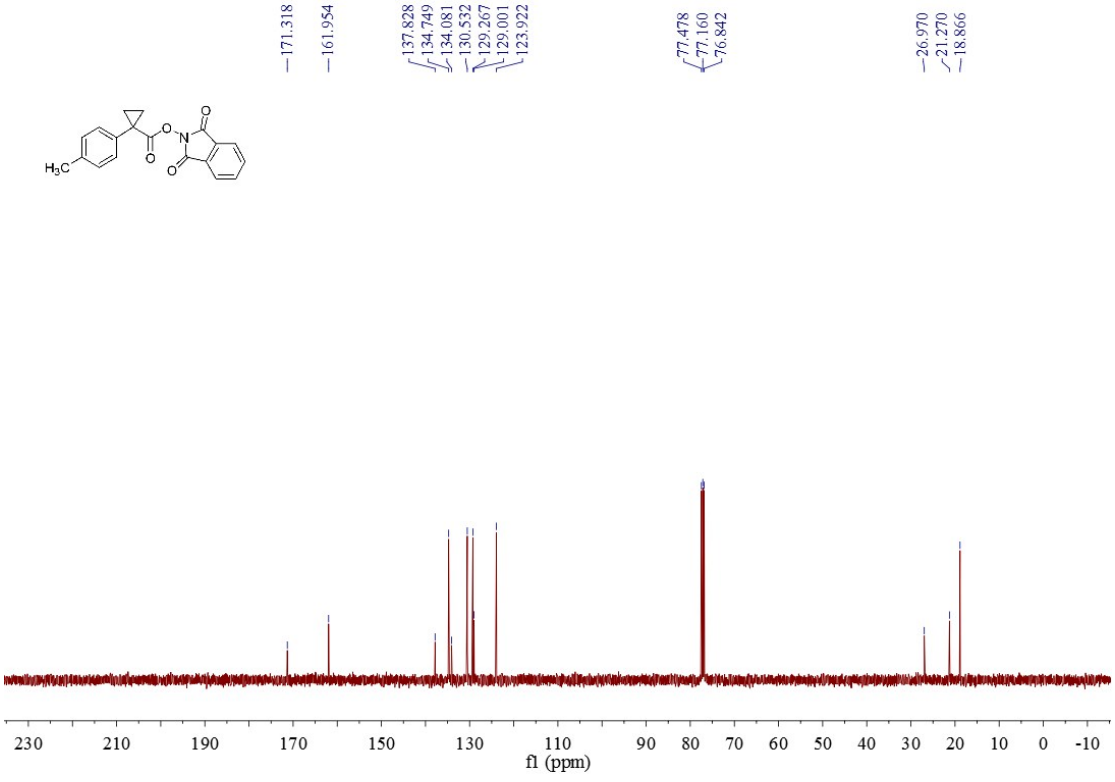
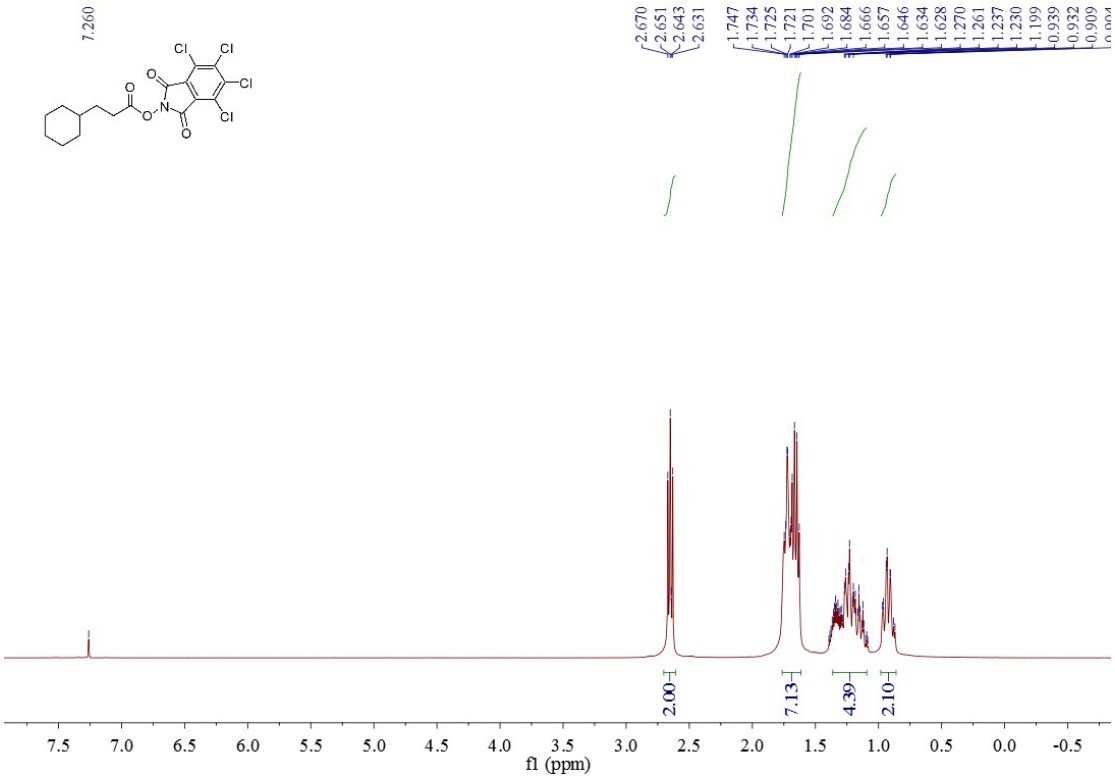


4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 3-cyclohexylpropanoate **a10**

^{1}H-NMR



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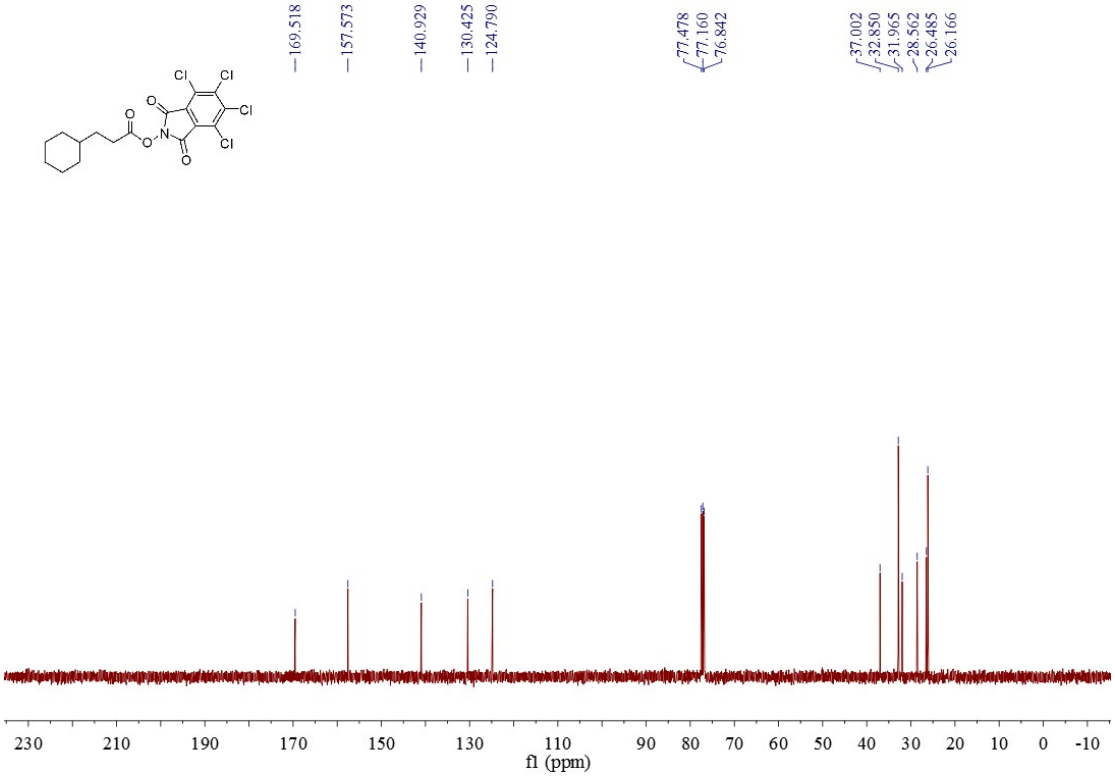
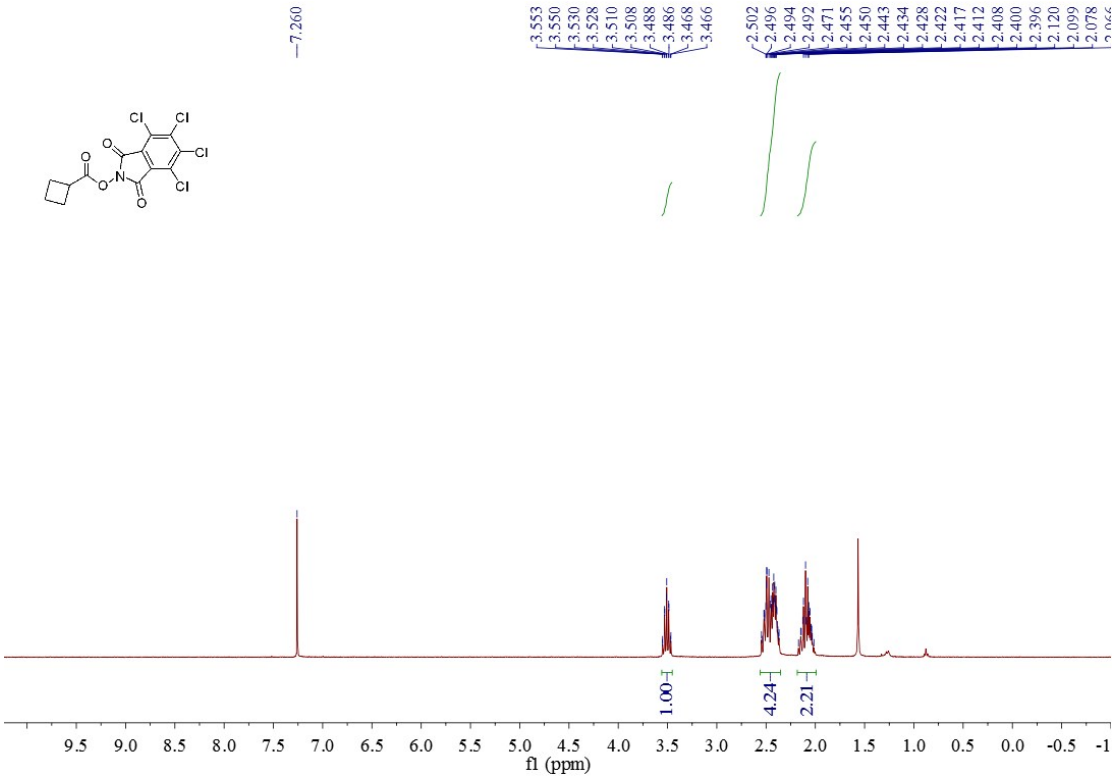


4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl cyclobutanecarboxylate **a13**

^{1}H-NMR



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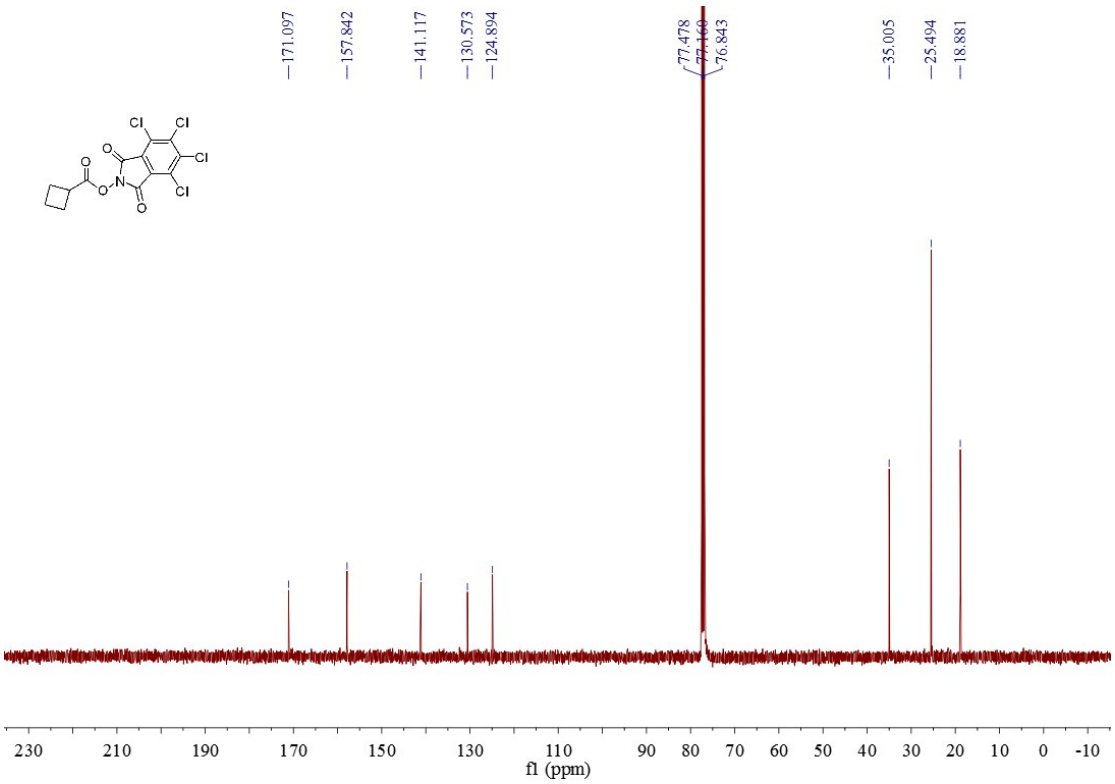
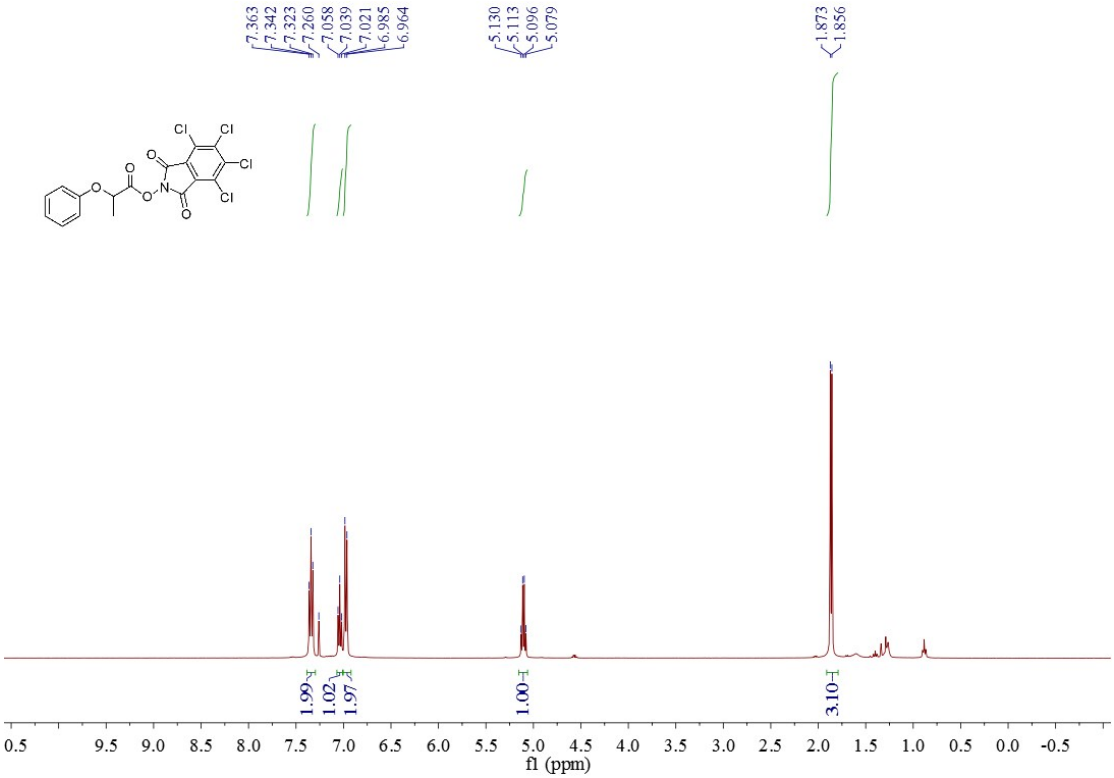


4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenoxypropanoate **a17**

^{1}H-NMR



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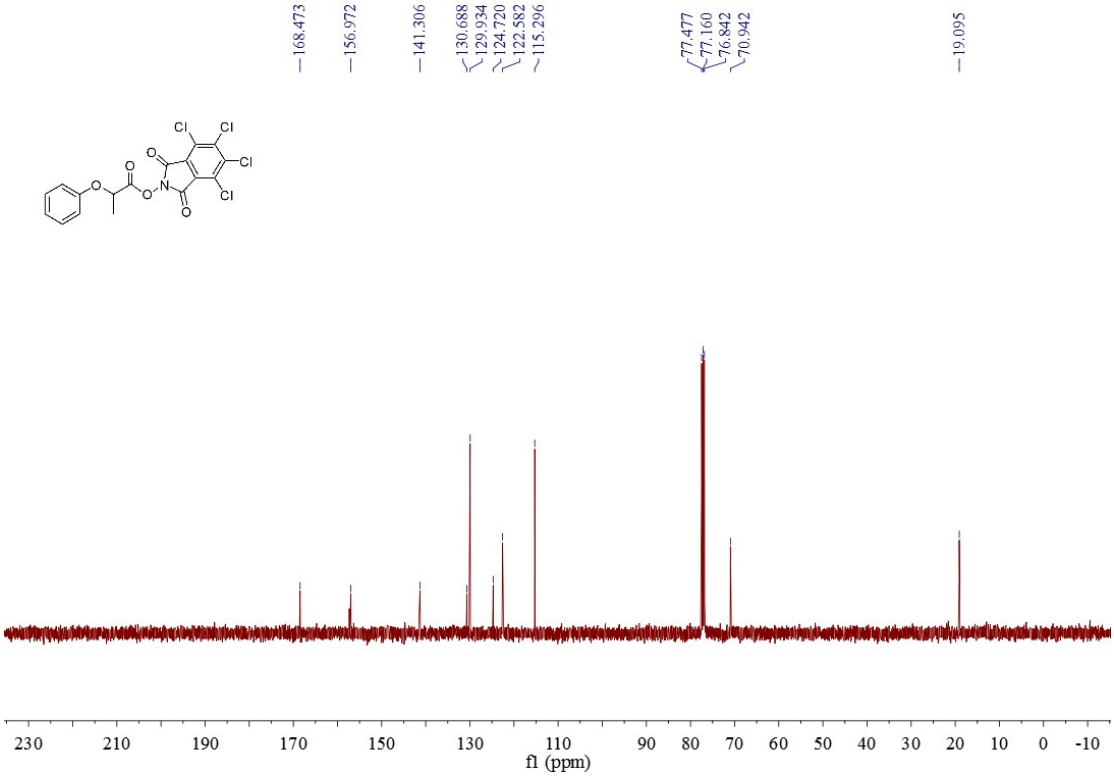
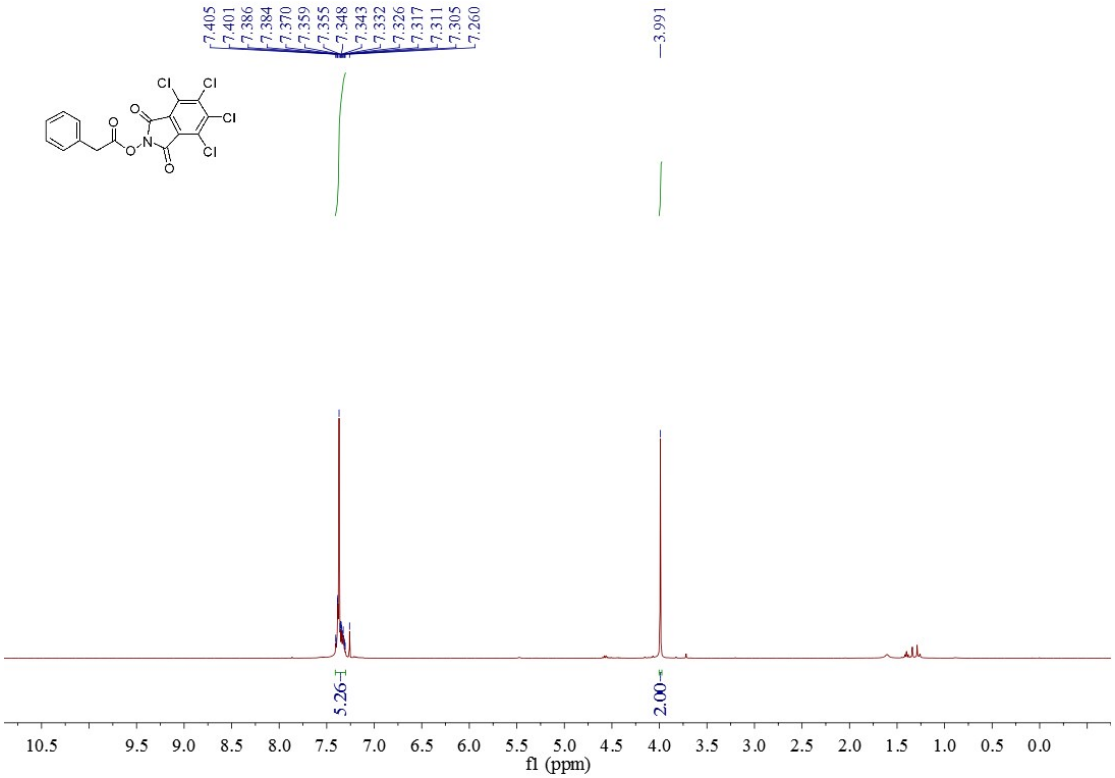


4,5,6,7-tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenylacetate **a18**

^{1}H-NMR



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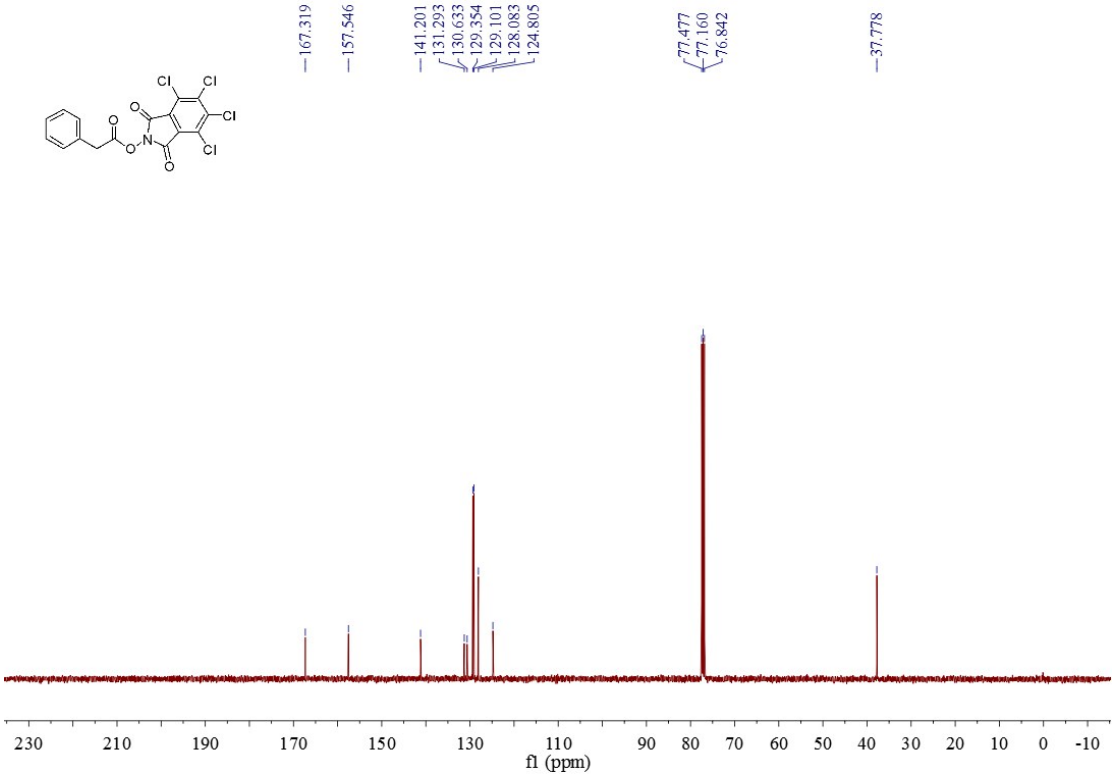
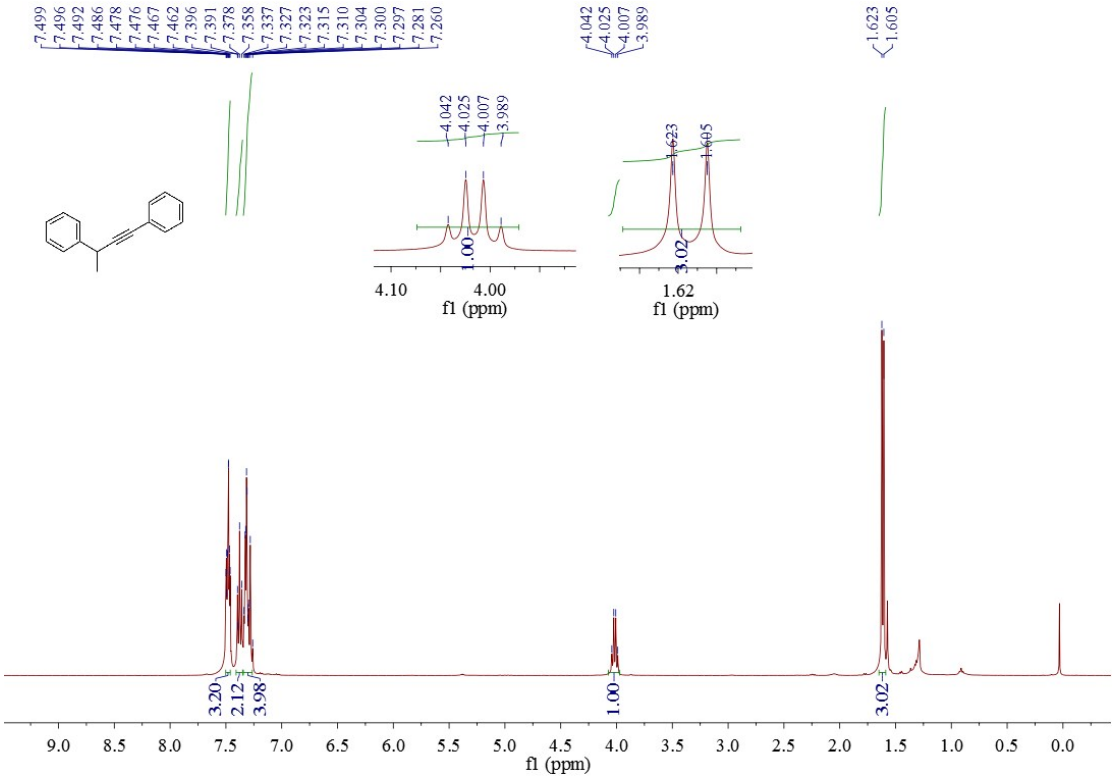


but-1-yne-1,3-diyldibenzene **c1**

^{1}H-NMR



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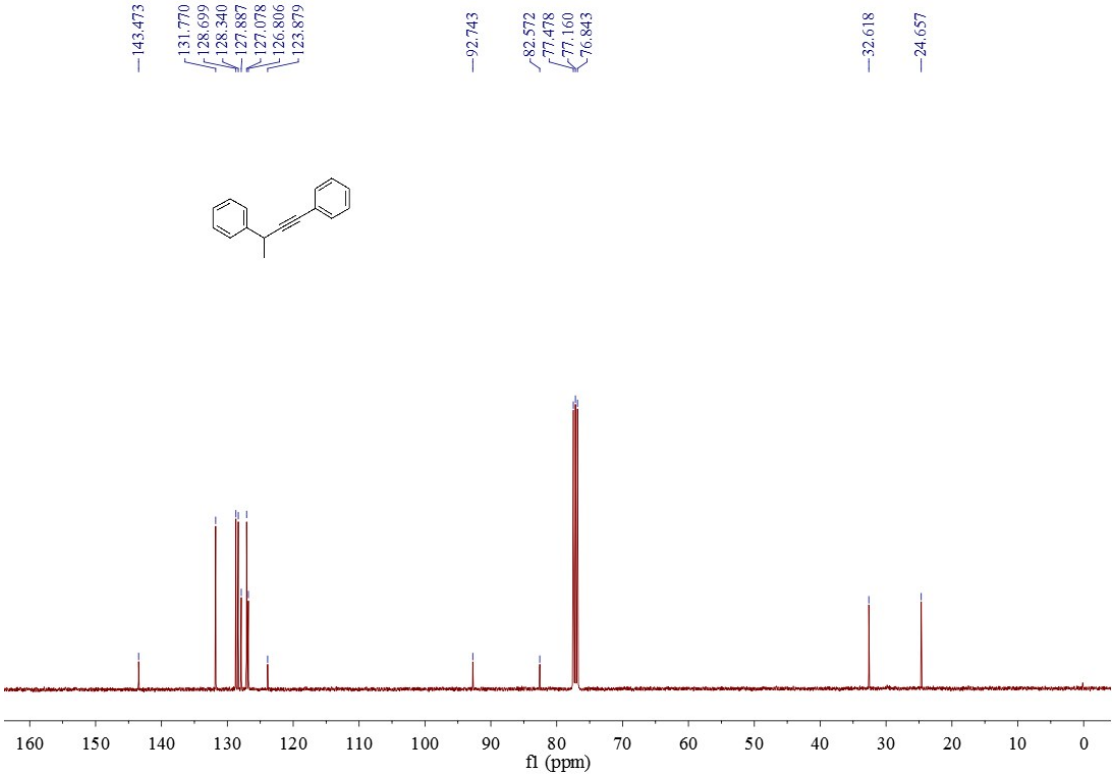
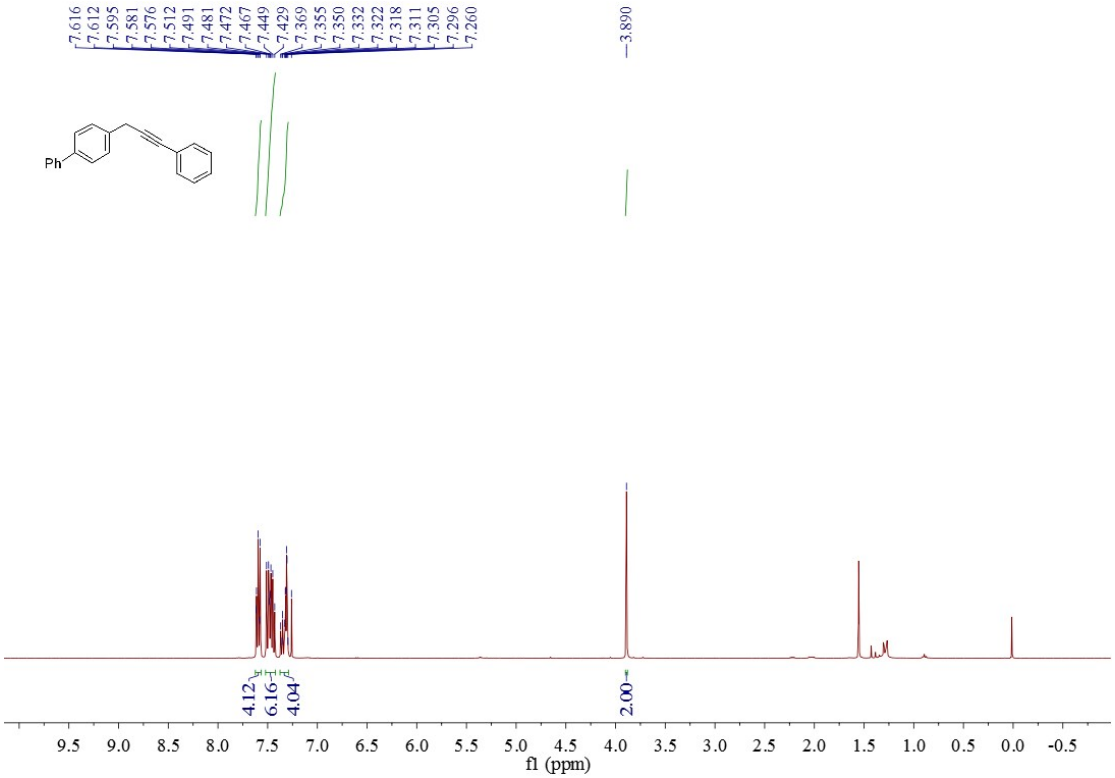
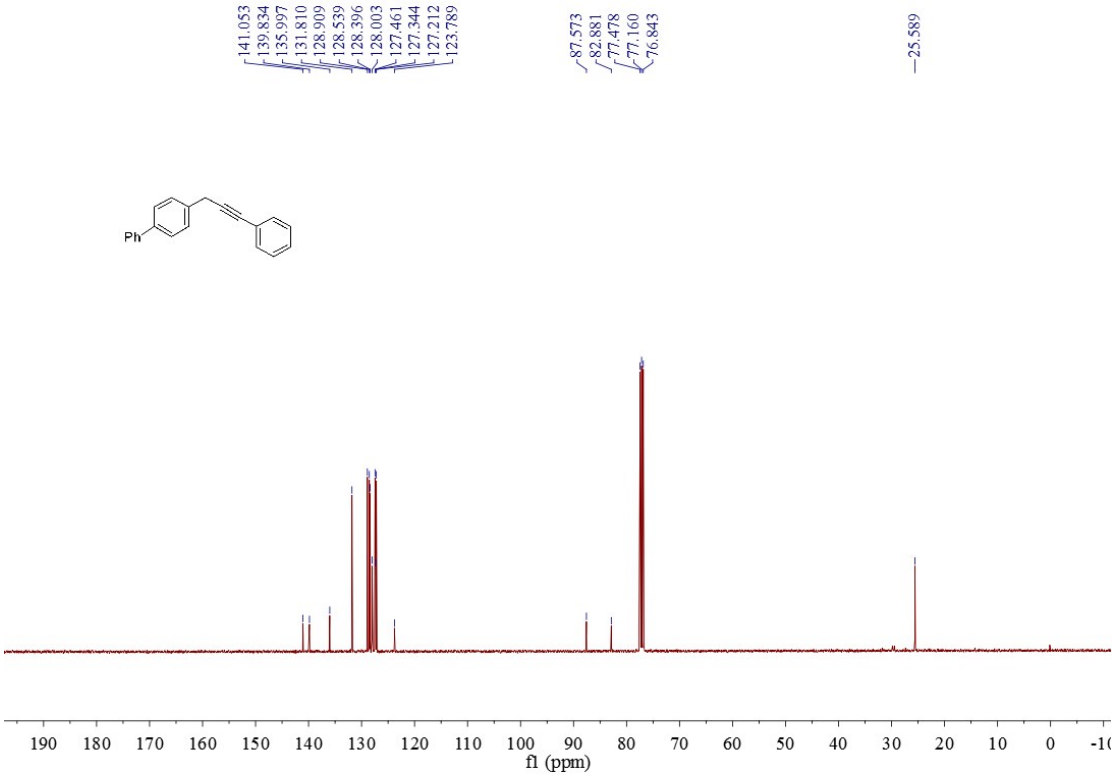


4-(3-phenylprop-2-yn-1-yl)-1,1'-biphenyl **c2**

^{1}H NMR

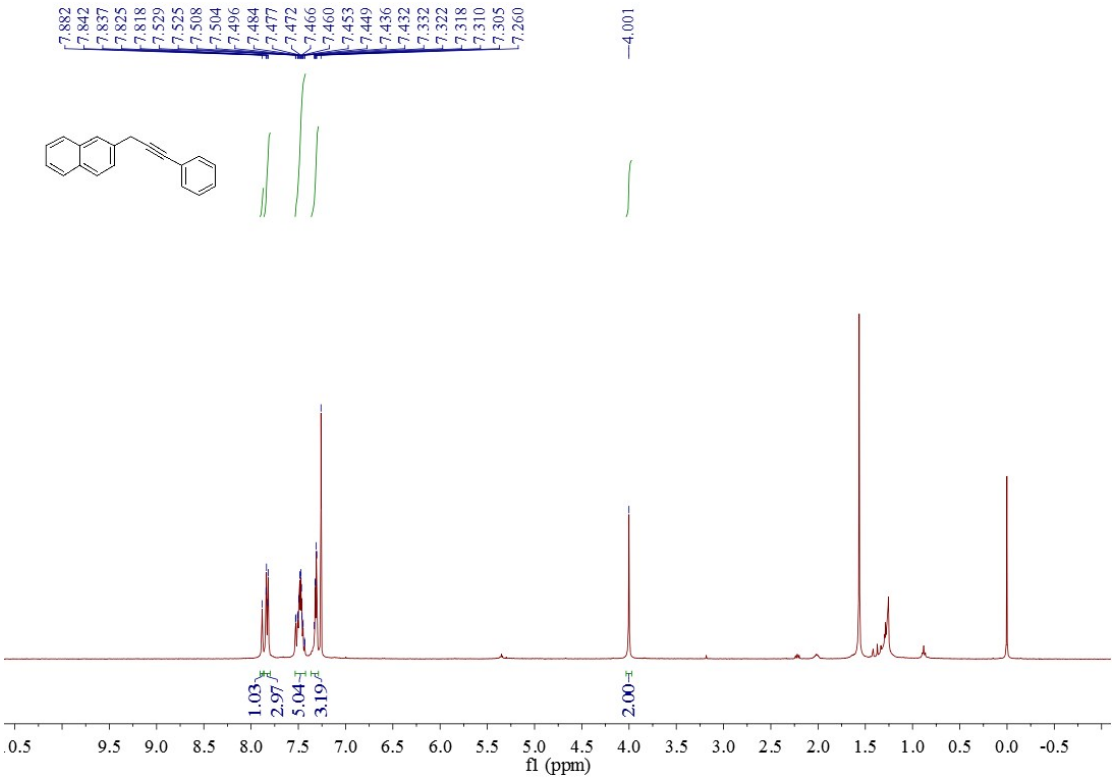


^{13}C NMR



2-(3-phenylprop-2-yn-1-yl)naphthalene **c3**

^{1}H NMR



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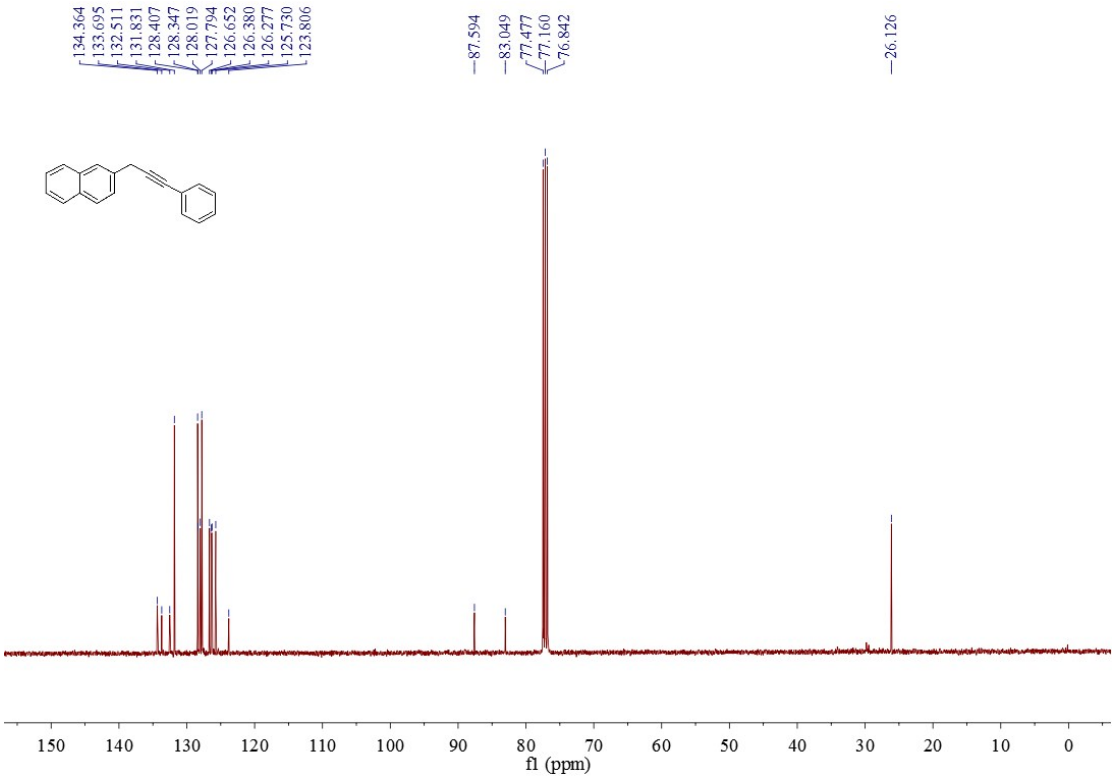
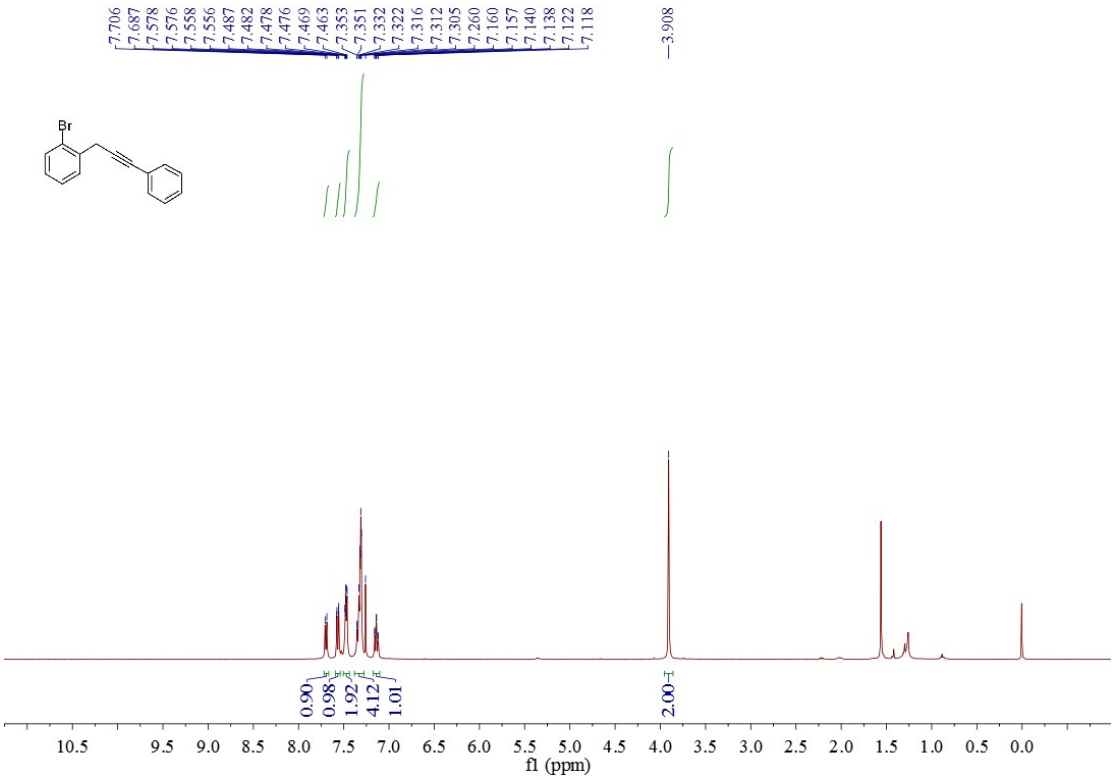


1-bromo-2-(3-phenylprop-2-yn-1-yl)benzene **c4**

^{1}H NMR



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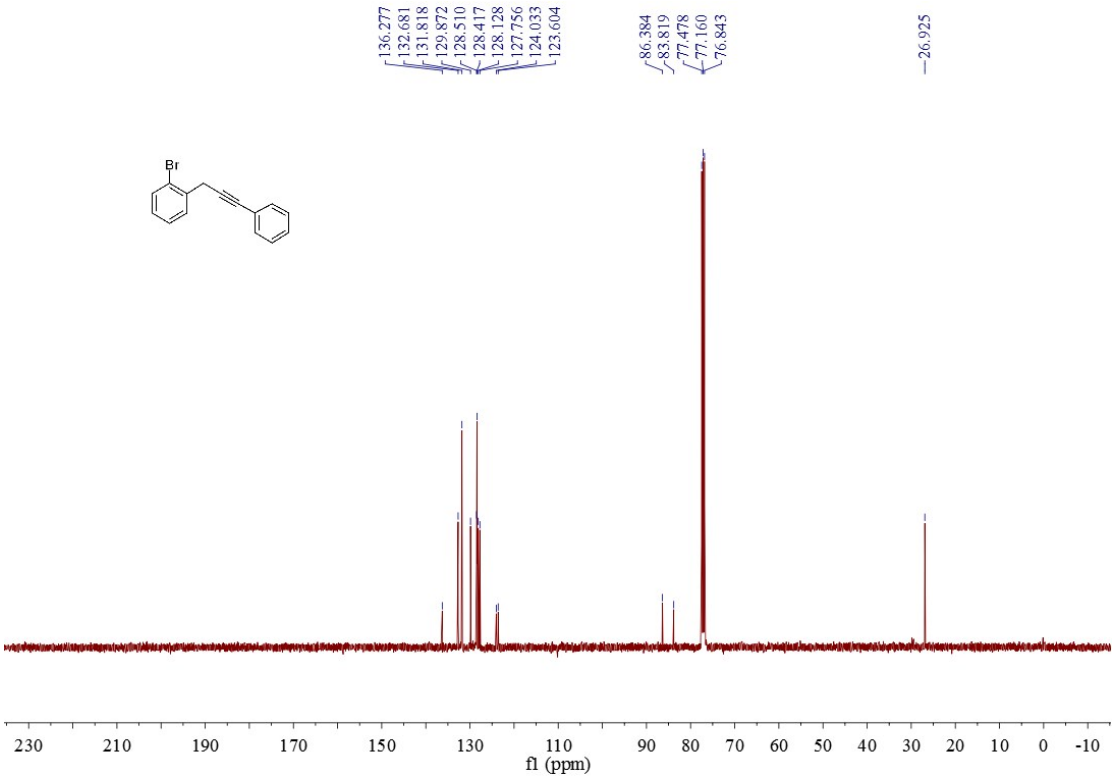
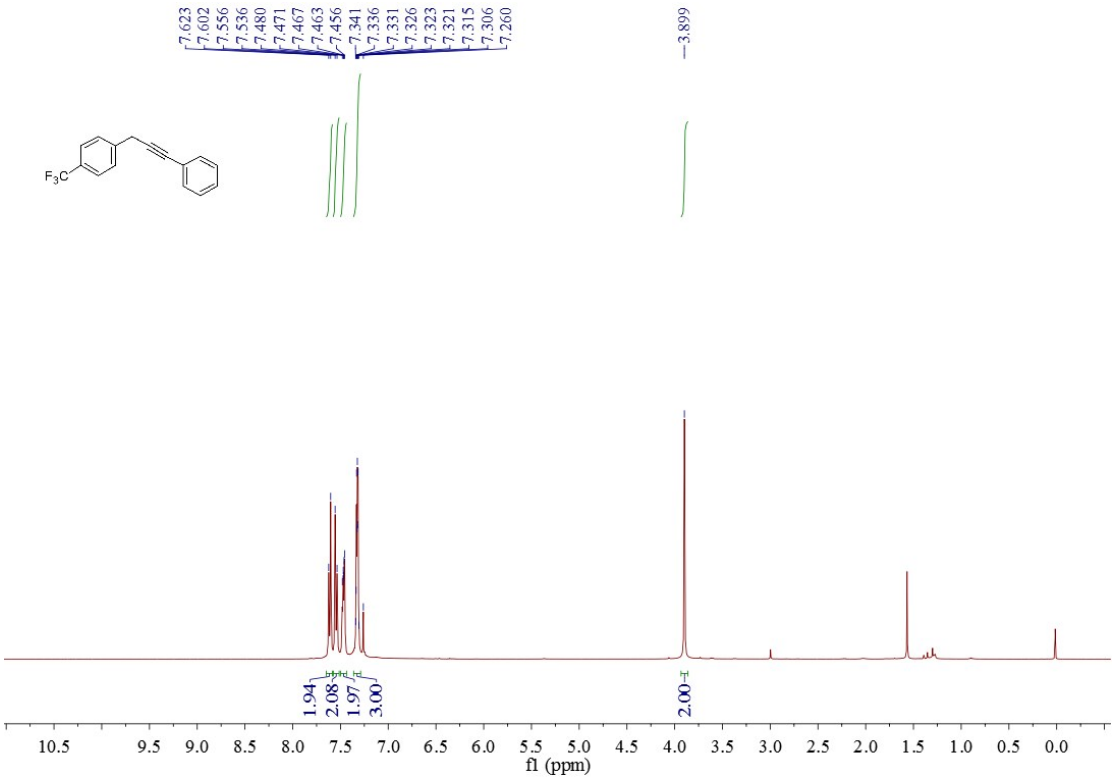


1-(3-phenylprop-2-yn-1-yl)-4-(trifluoromethyl)benzene **c5**

^{1}H NMR



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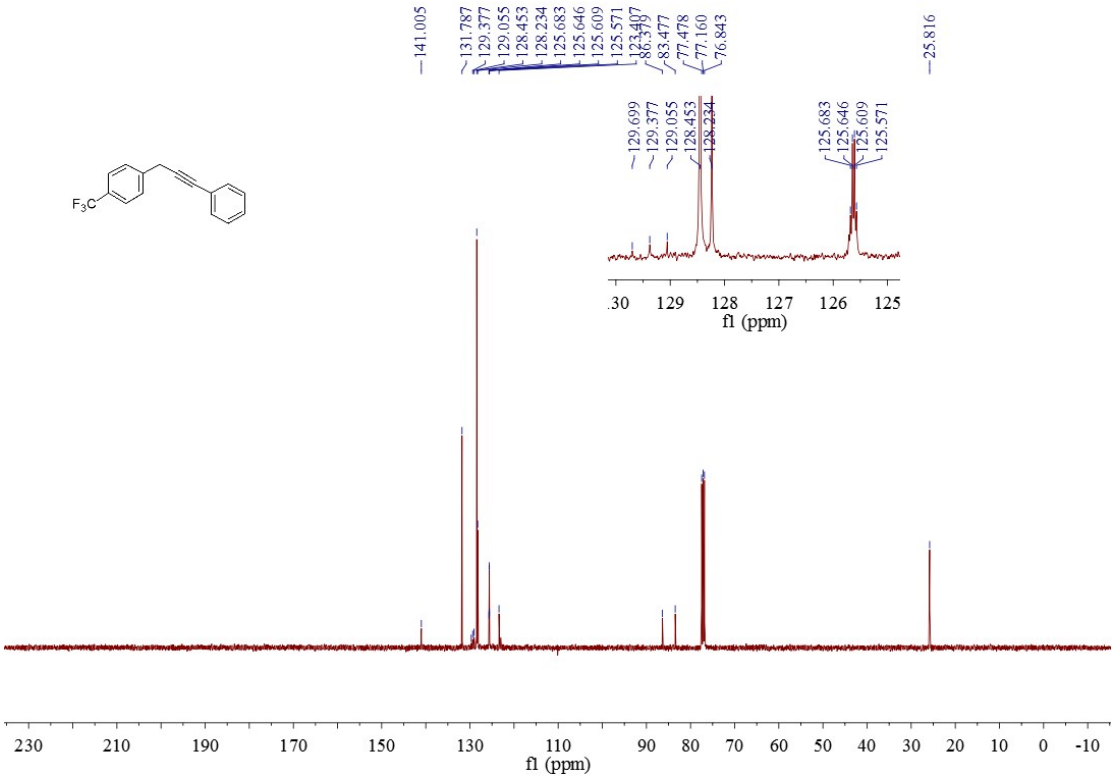
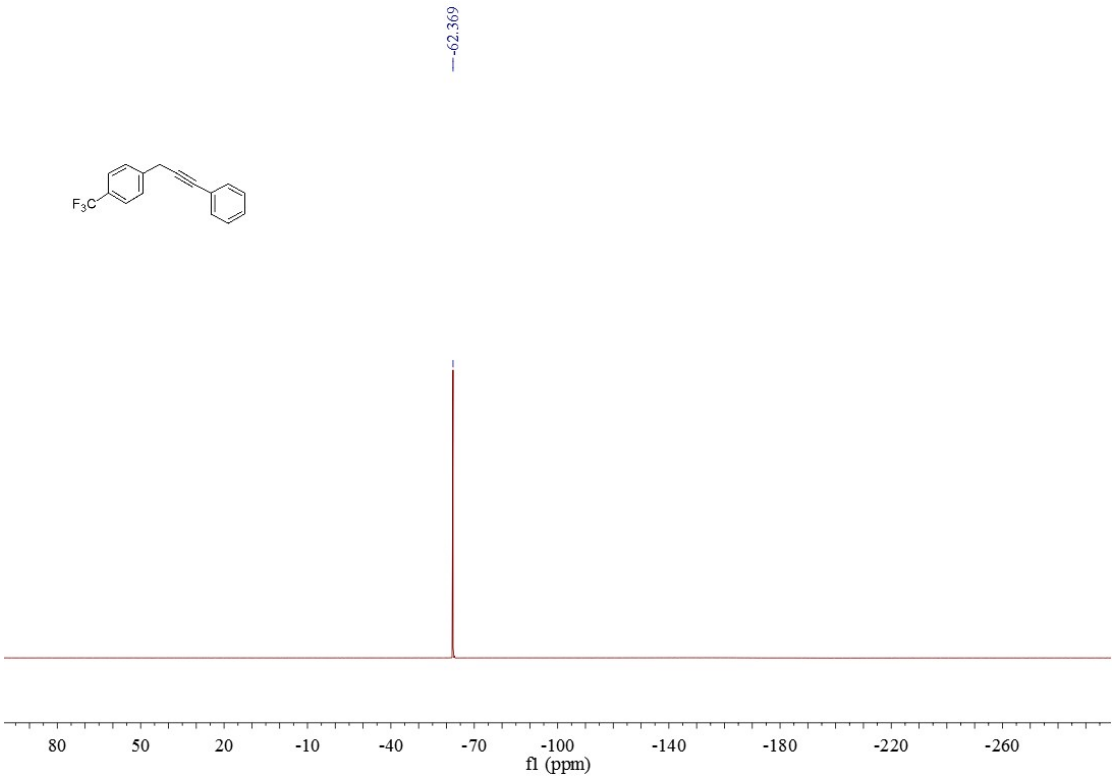
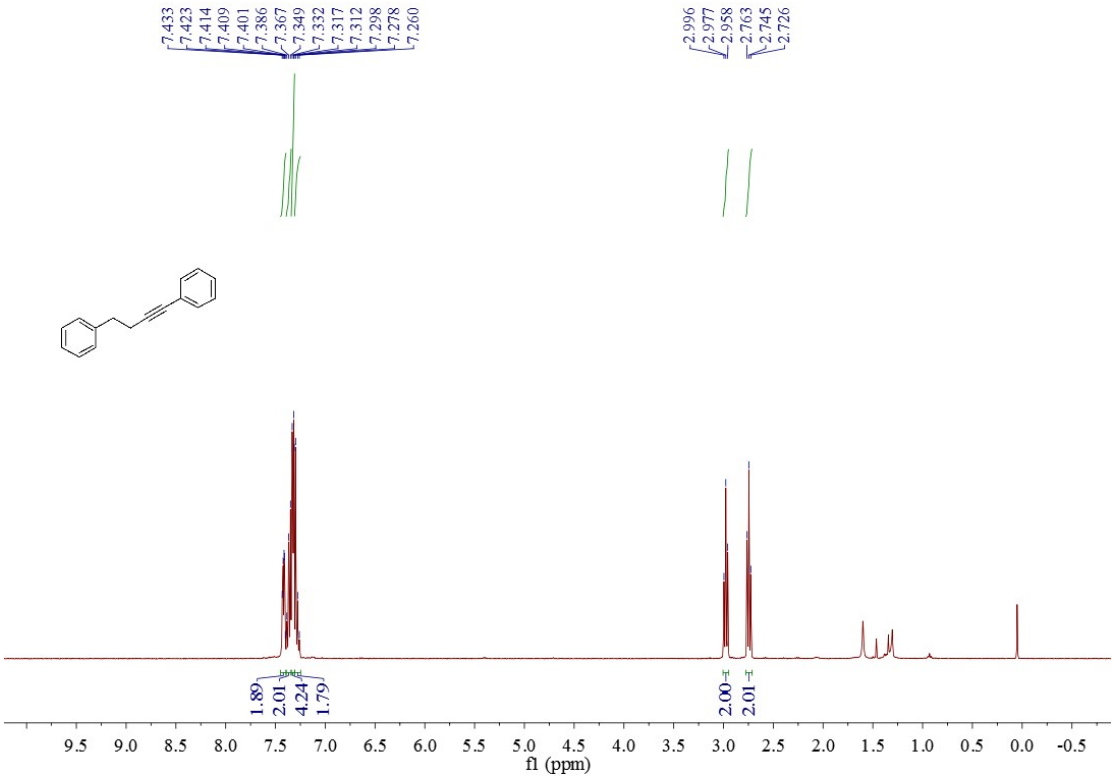


^{19}F-NMR



but-1-yne-1,4-diyldibenzene **c6**

^{1}H NMR



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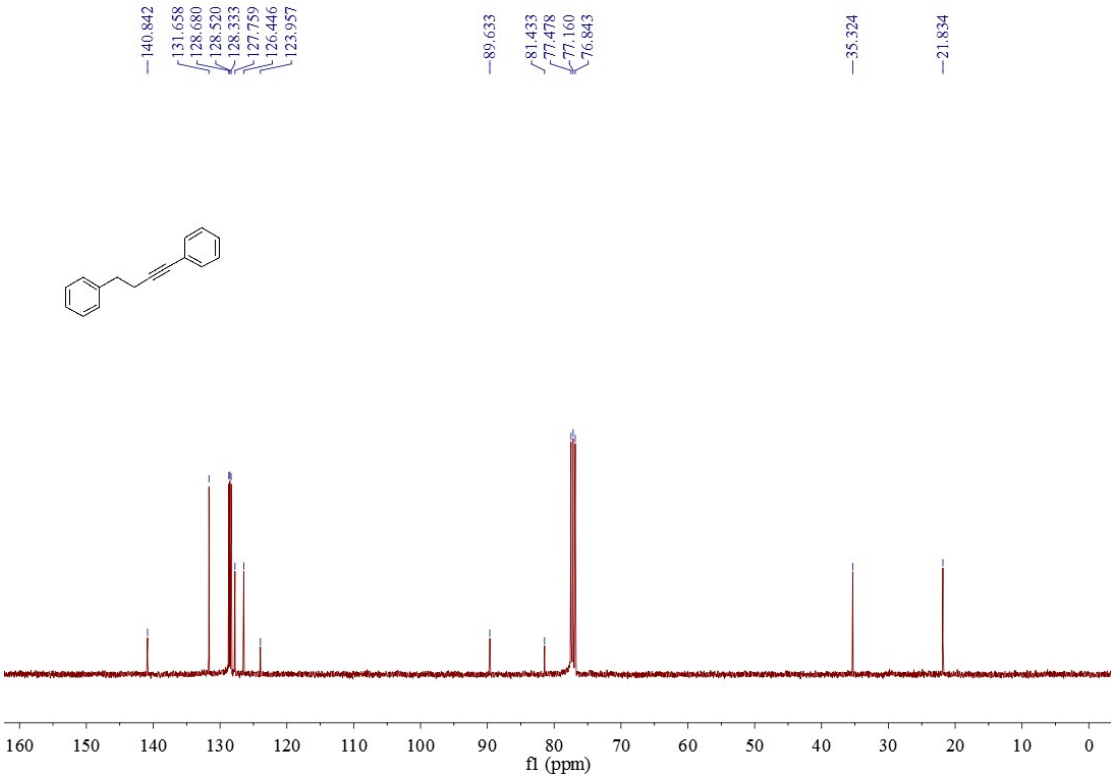
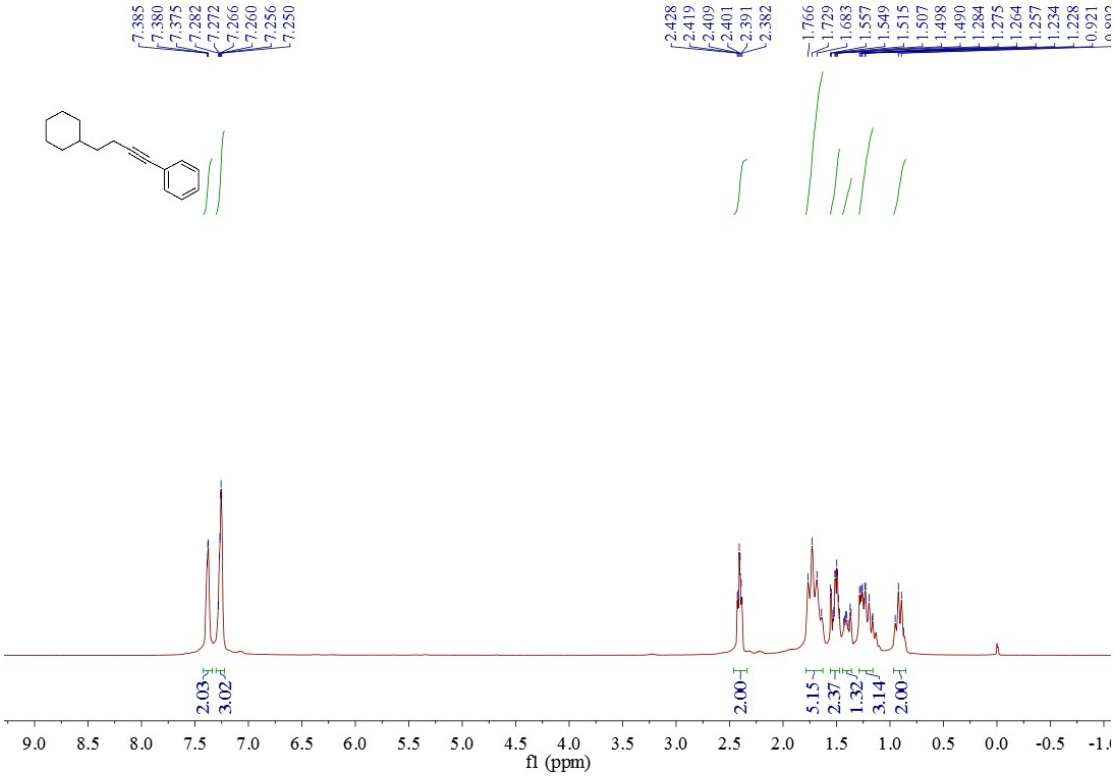


(4-cyclohexylbut-1-yn-1-yl)benzene **c7**

^{1}H NMR



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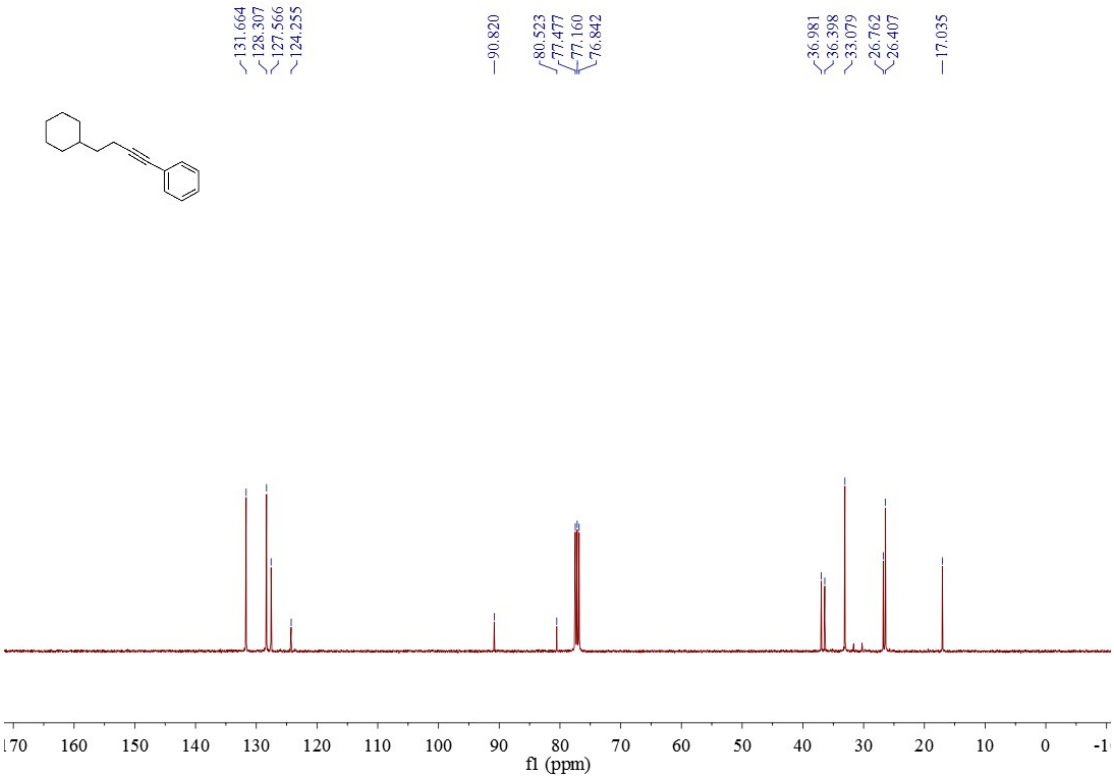
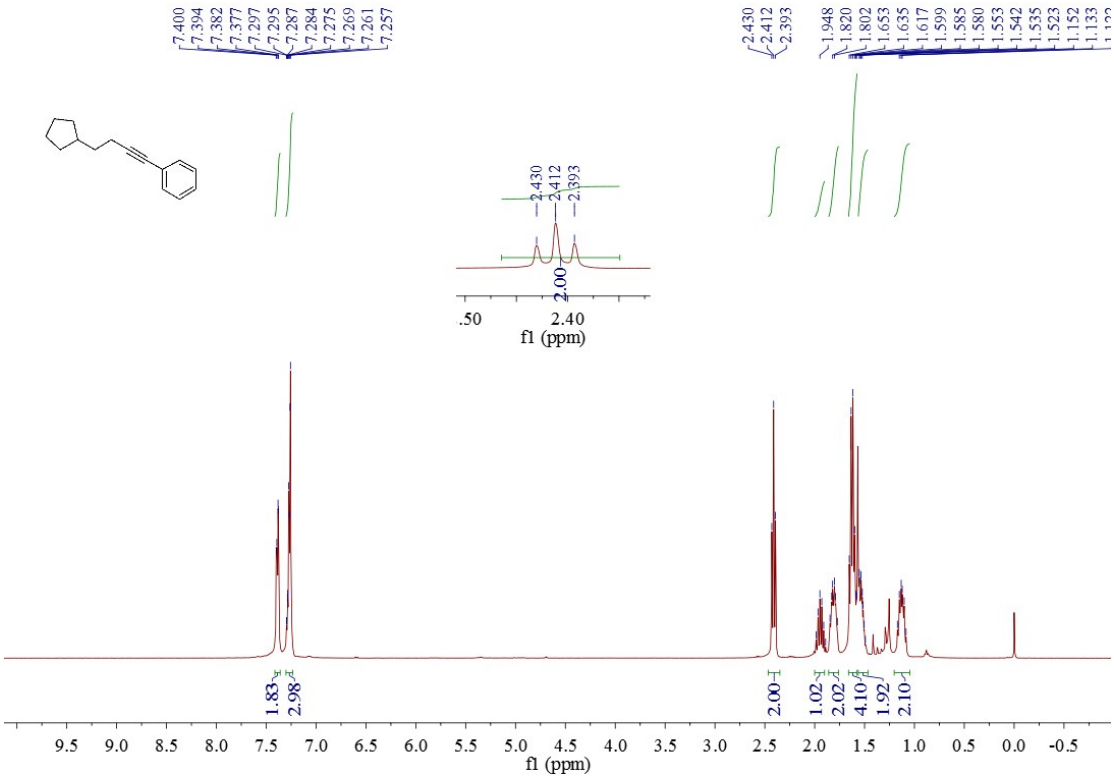
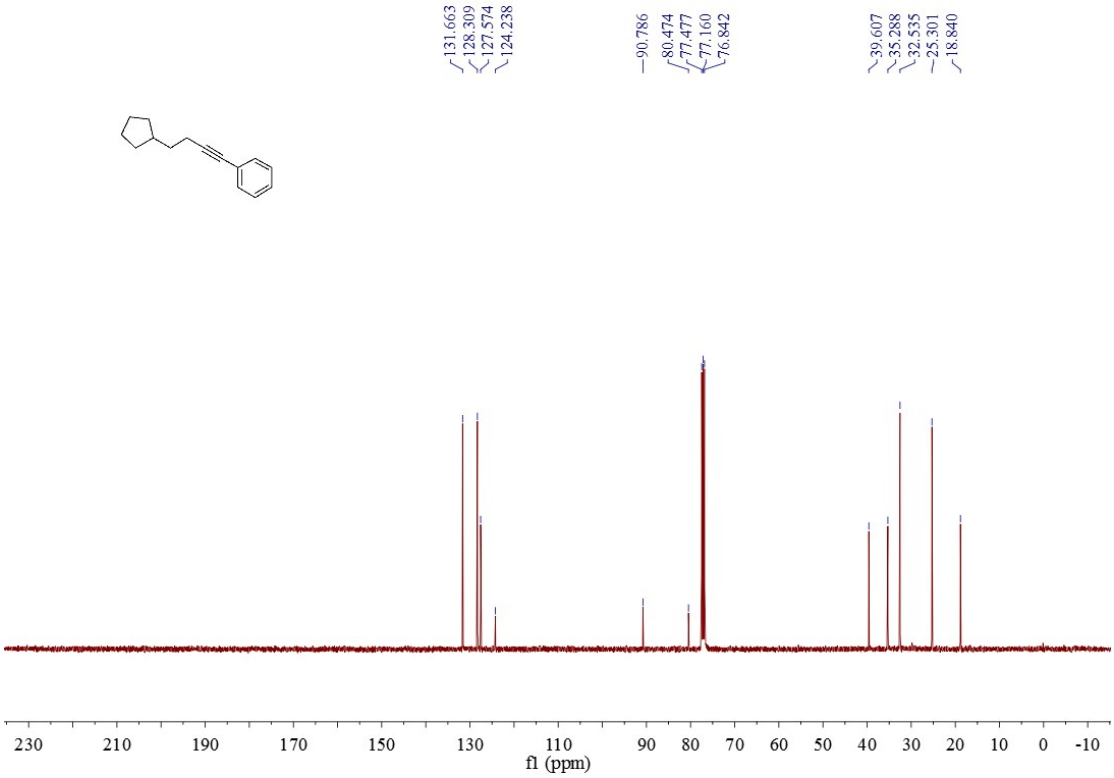


(4-cyclopentylbut-1-yn-1-yl)benzene **c8**

^{1}H NMR

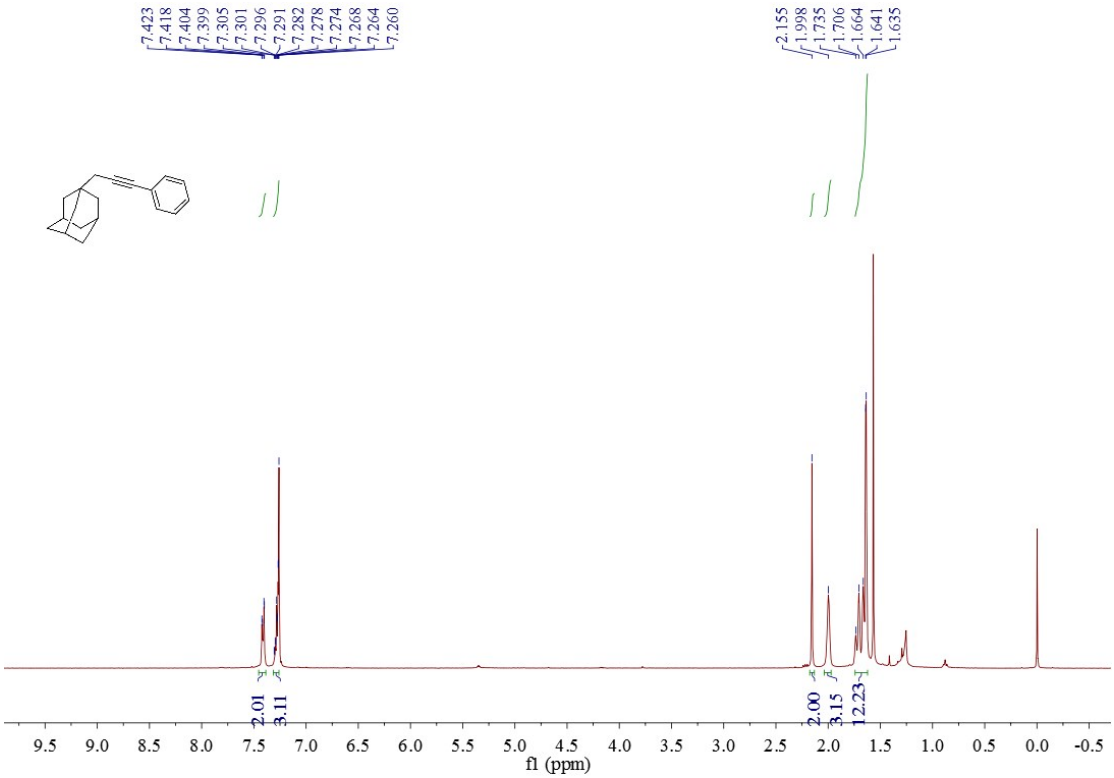


^{13}C NMR

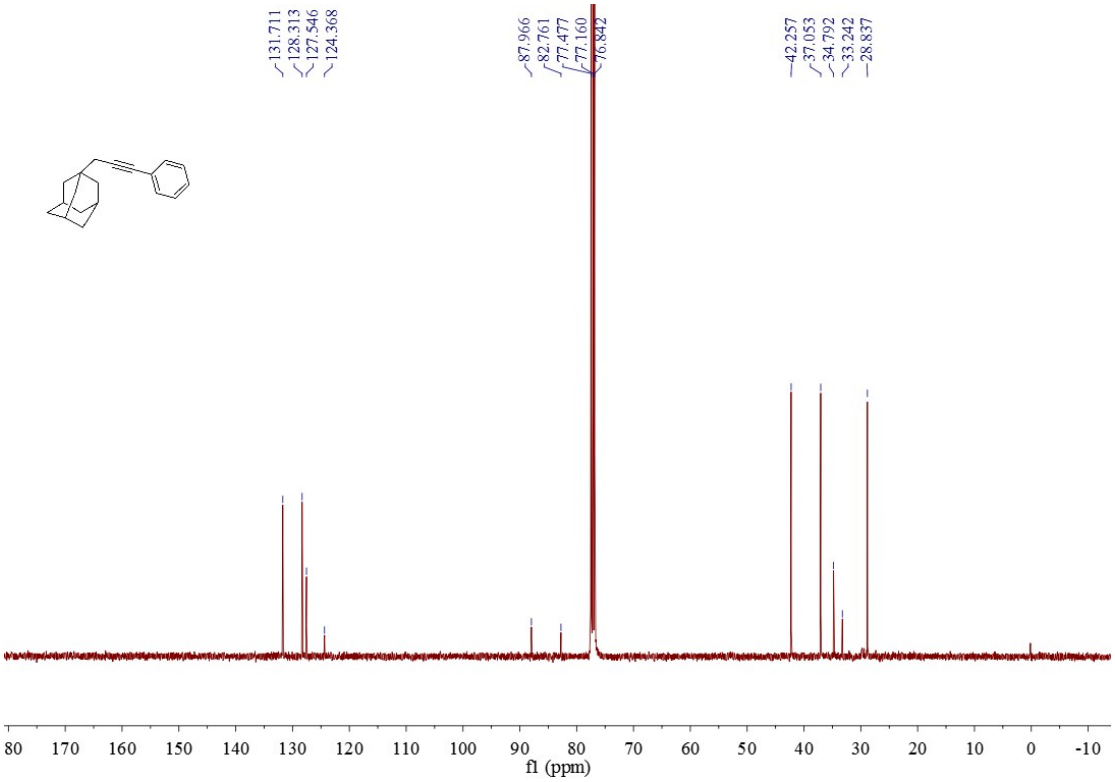


1-(phenylethynyl)adamantine **c9**

^{1}H NMR

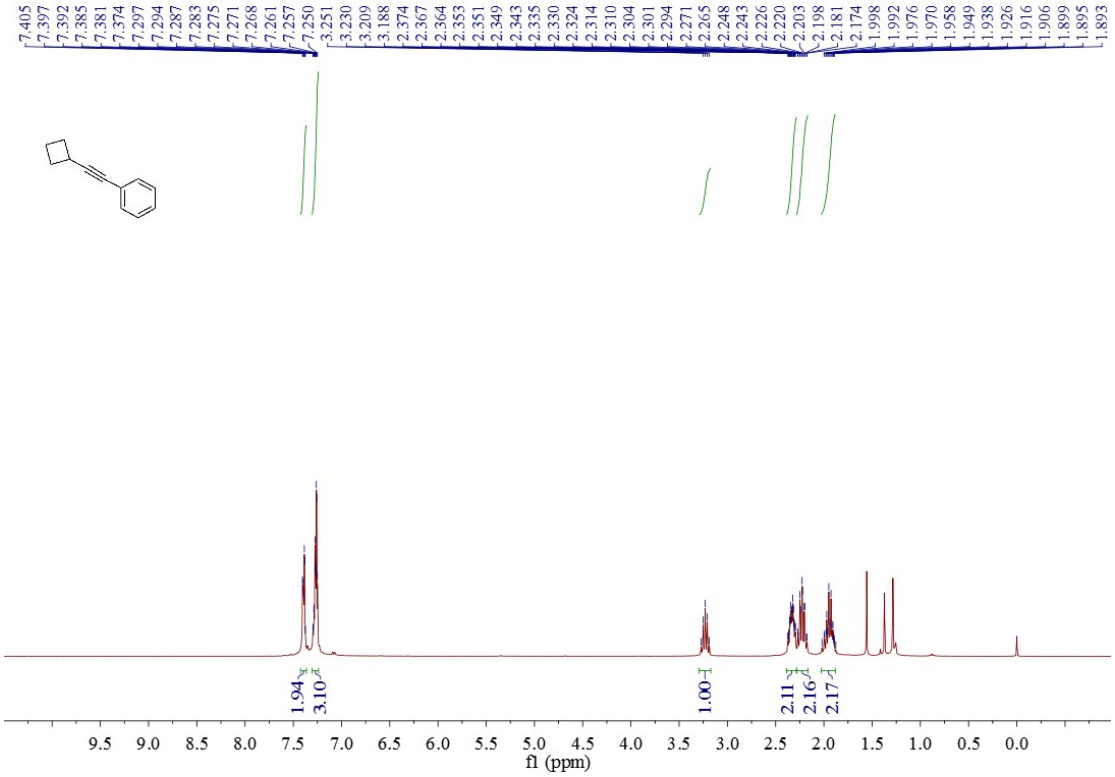


^{13}C NMR

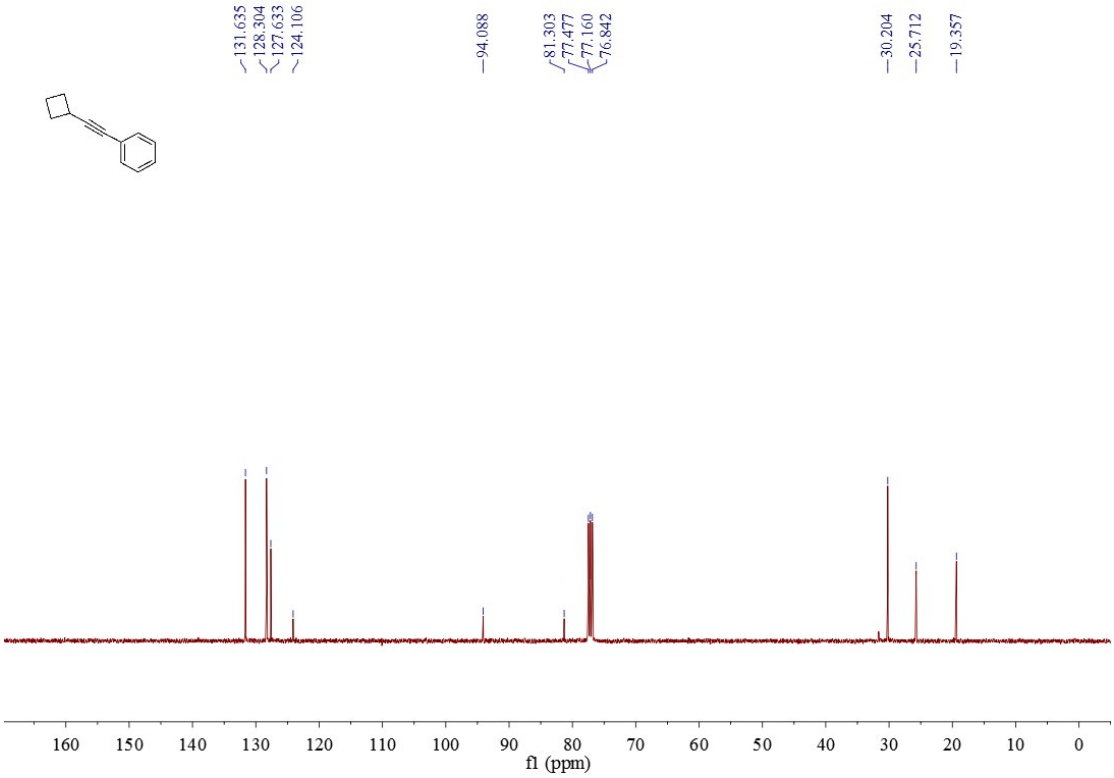


(cyclobutylethynyl)benzene **c10**

^{1}H NMR

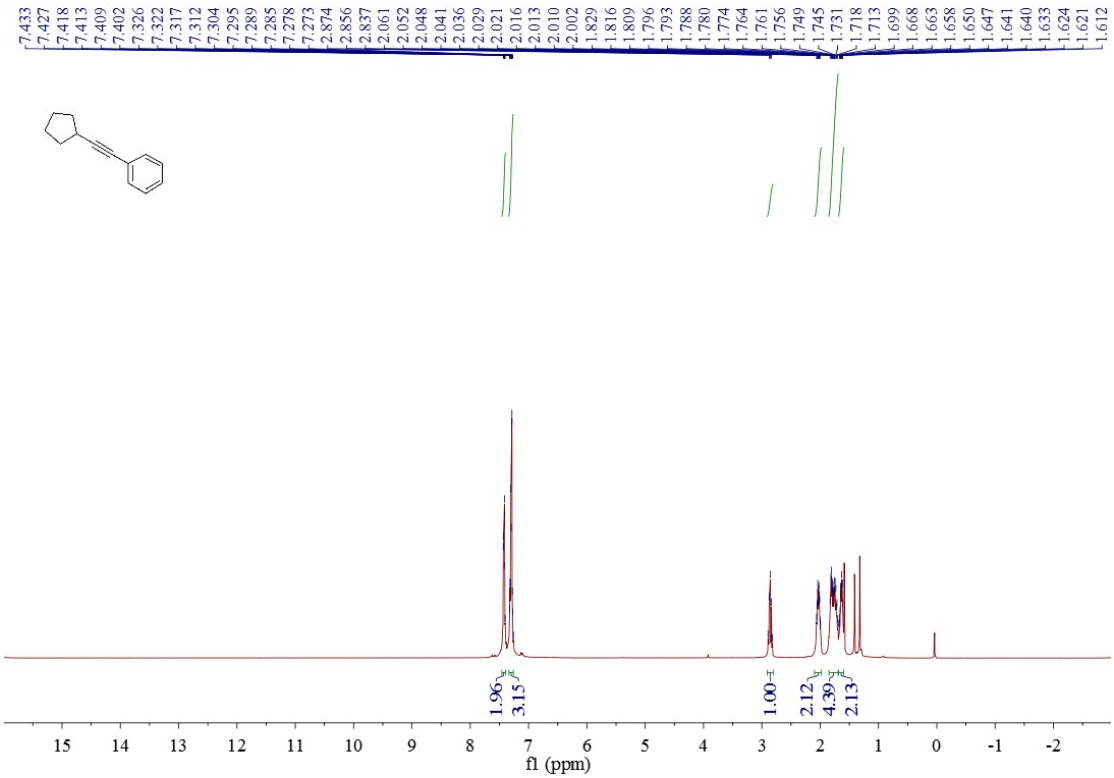


^{13}C NMR

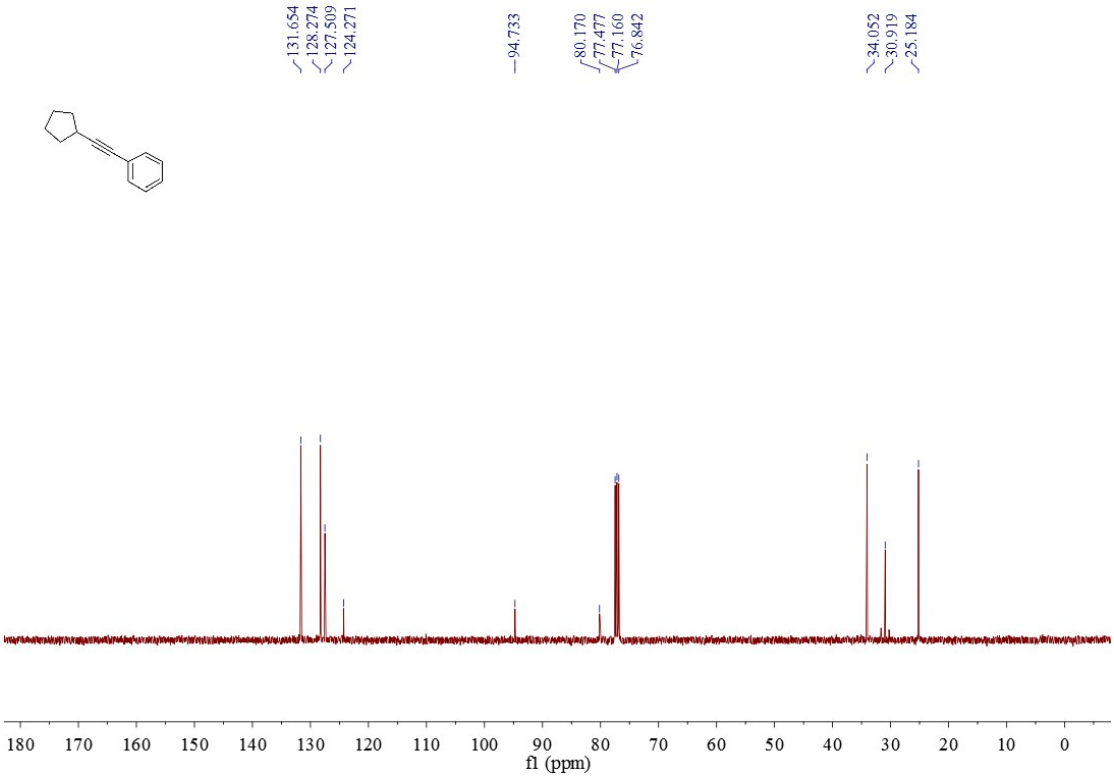


(cyclopentylethynyl)benzene **c11**

^{1}H-NMR

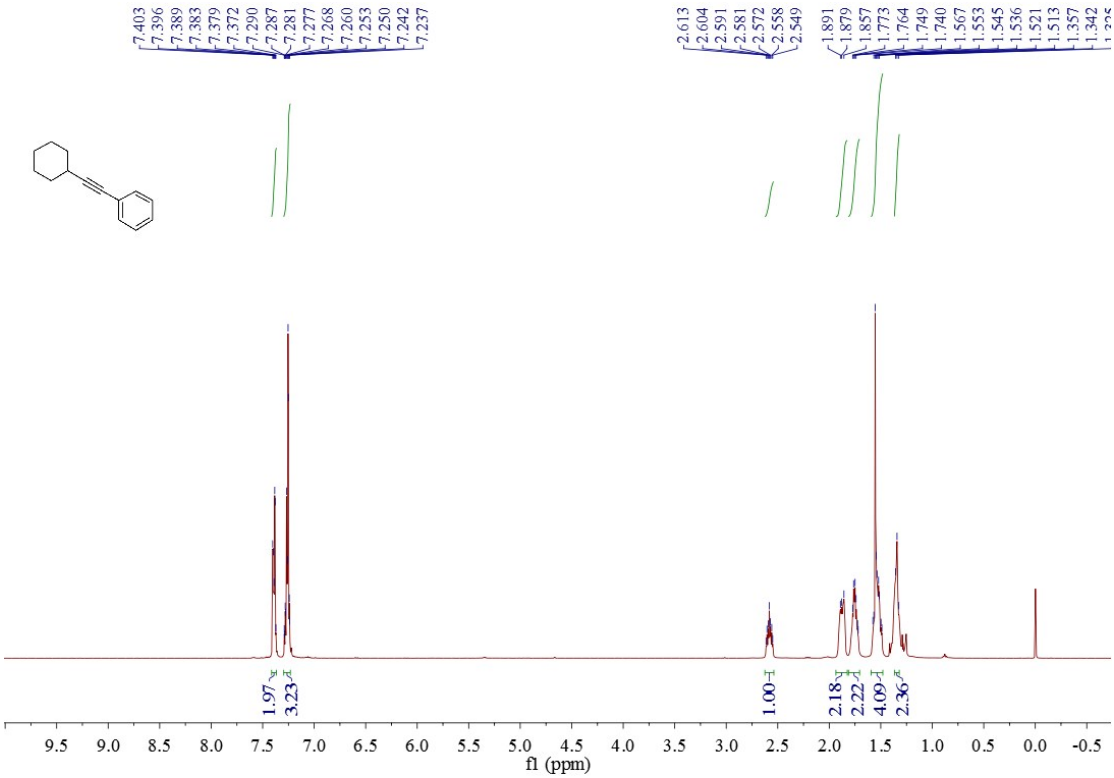


^{13}C-NMR

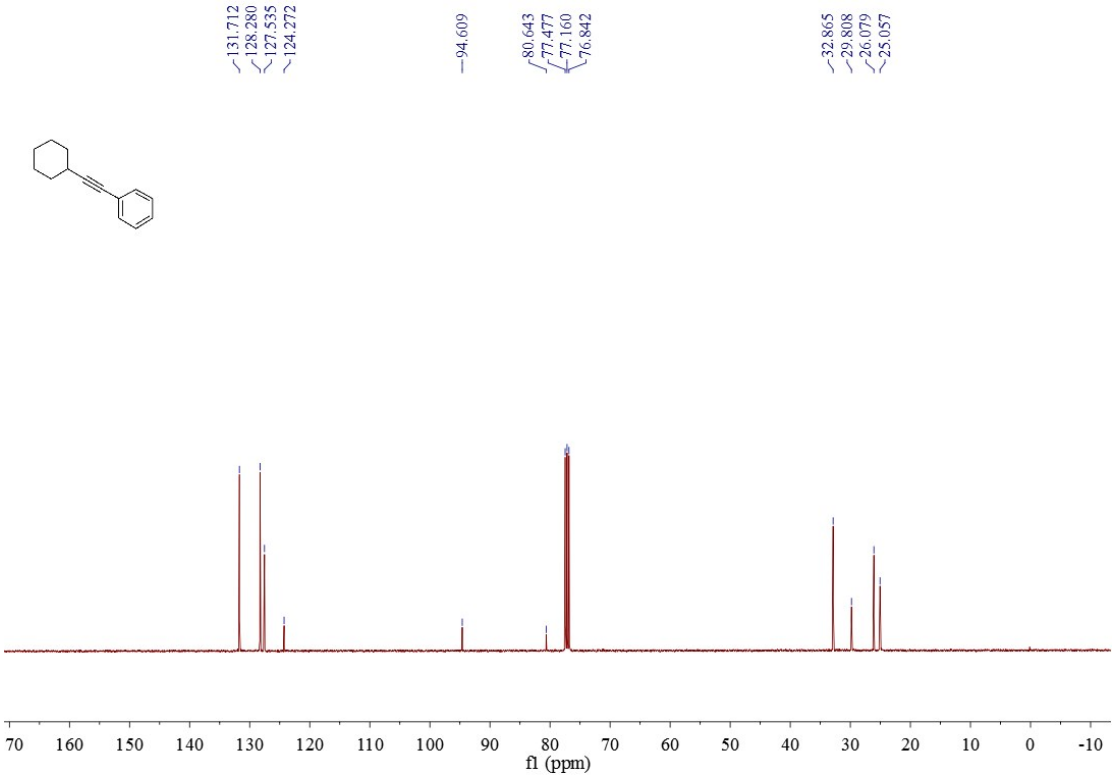


(cyclohexylethynyl)benzene **c12**

^{1}H NMR

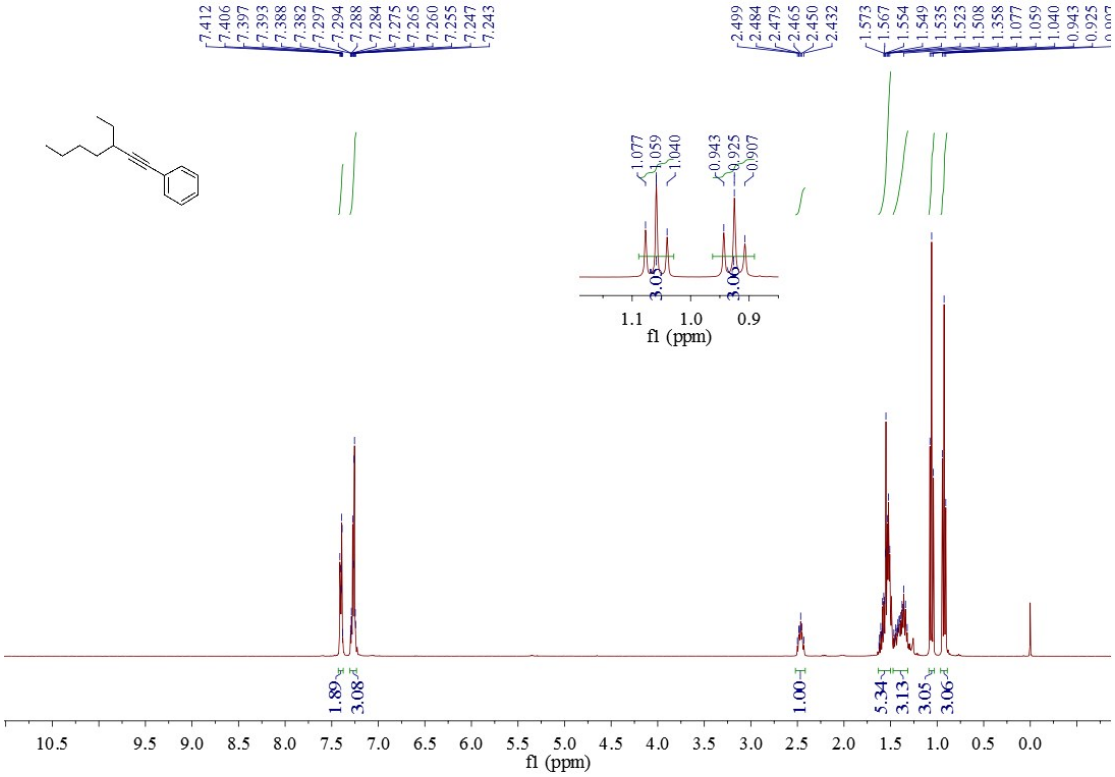


^{13}C NMR

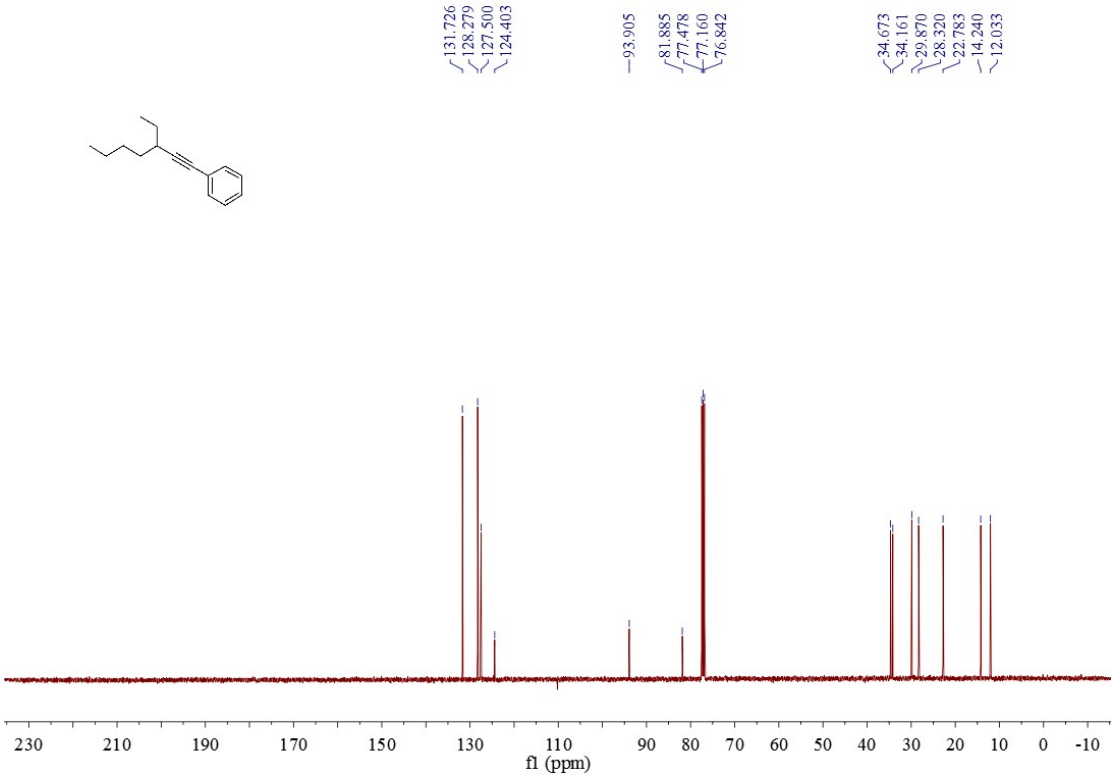


(3-ethylhept-1-yn-1-yl)benzene **c13**

^{1}H NMR

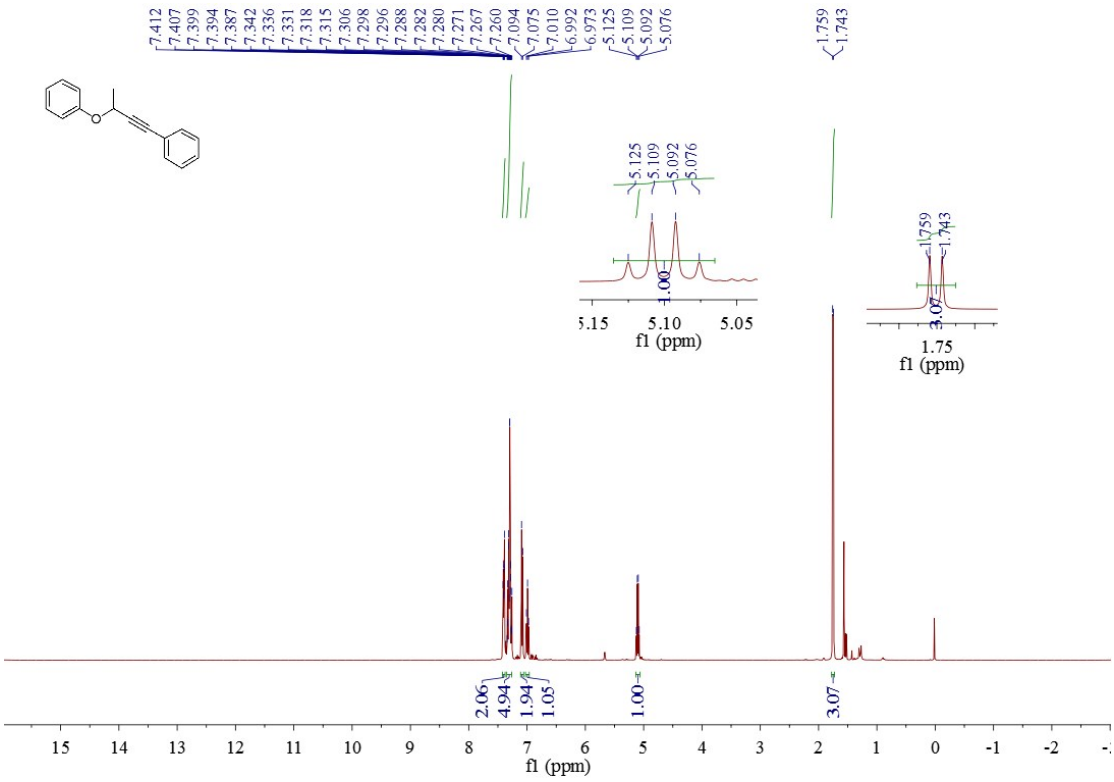


^{13}C NMR

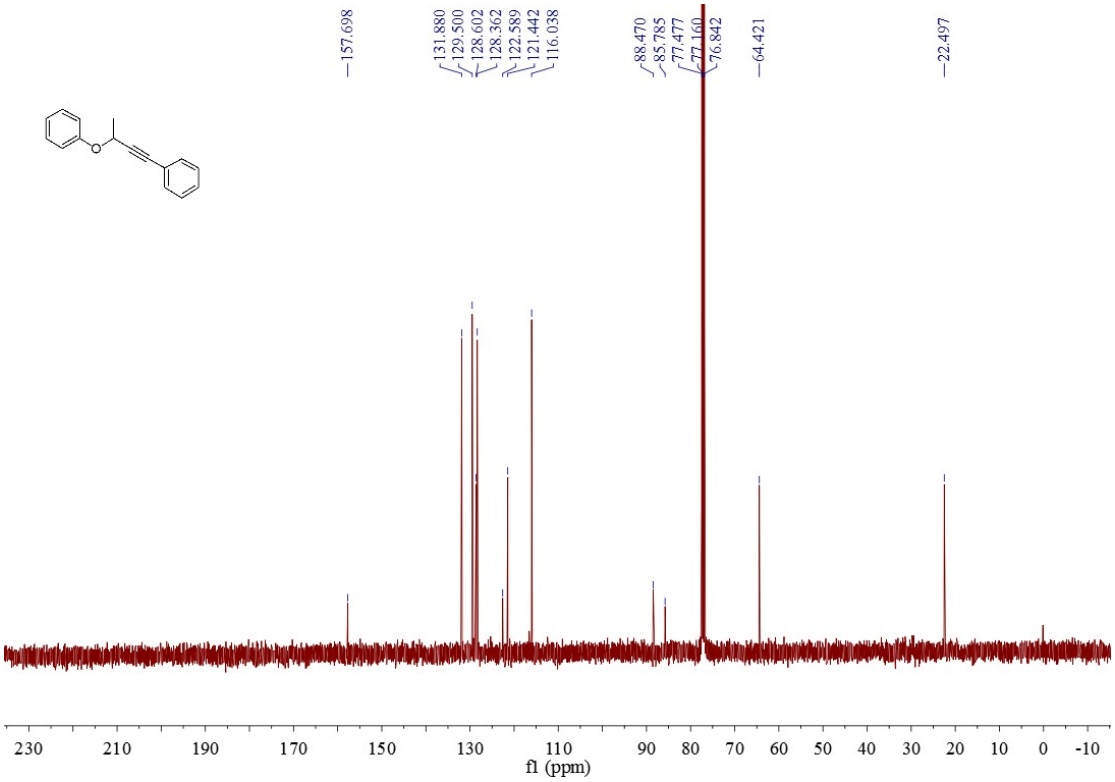


(3-phenoxybut-1-yn-1-yl)benzene **c14**

^{1}H NMR

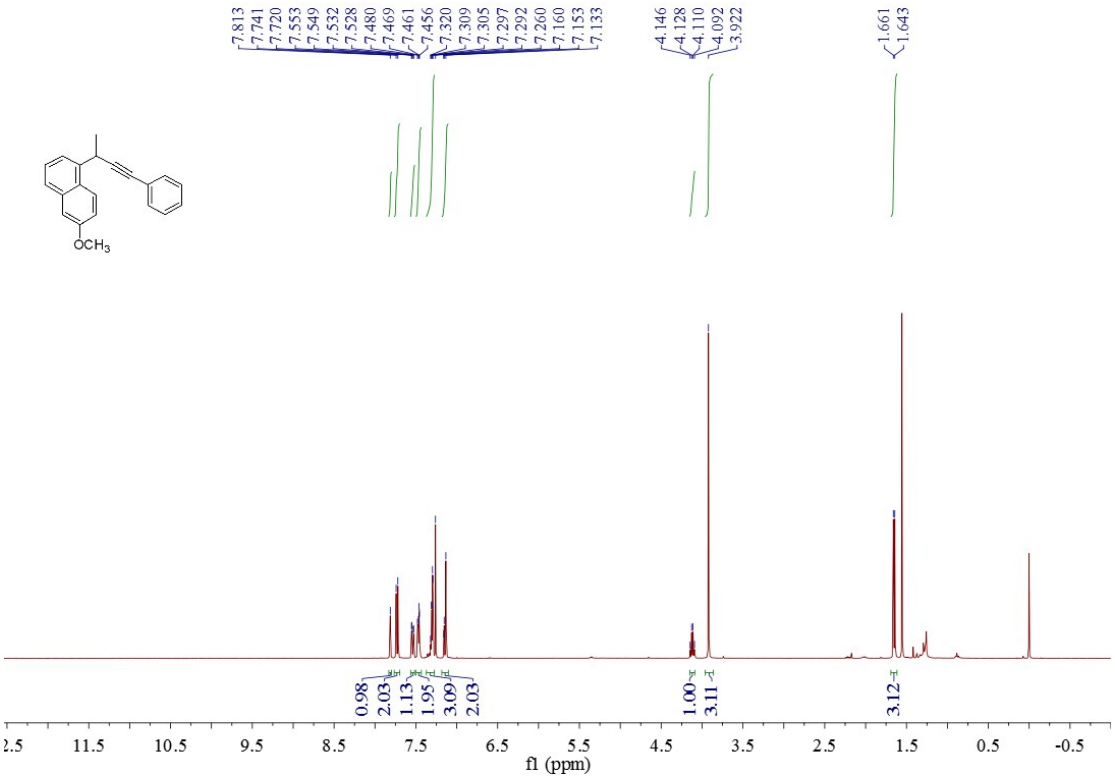


^{13}C NMR

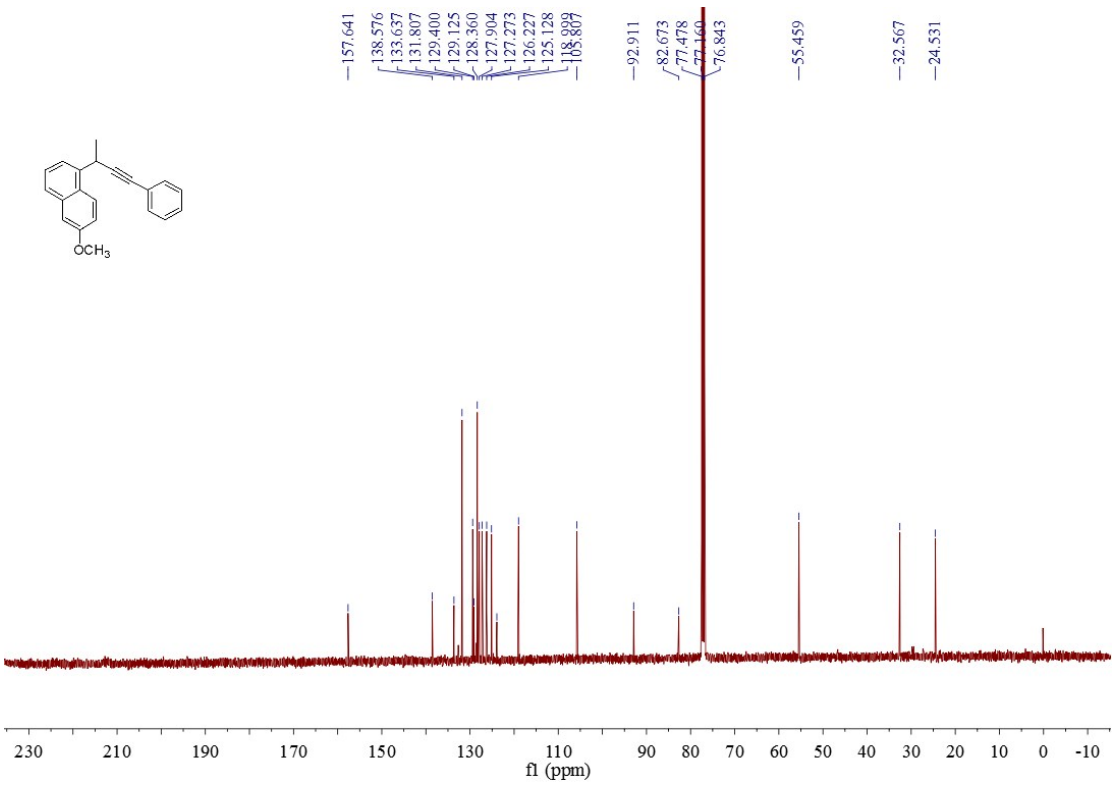


4-methoxy-4'-(4-phenylbut-3-yn-2-yl)-1,1'-biphenyl **c15**

^{1}H-NMR

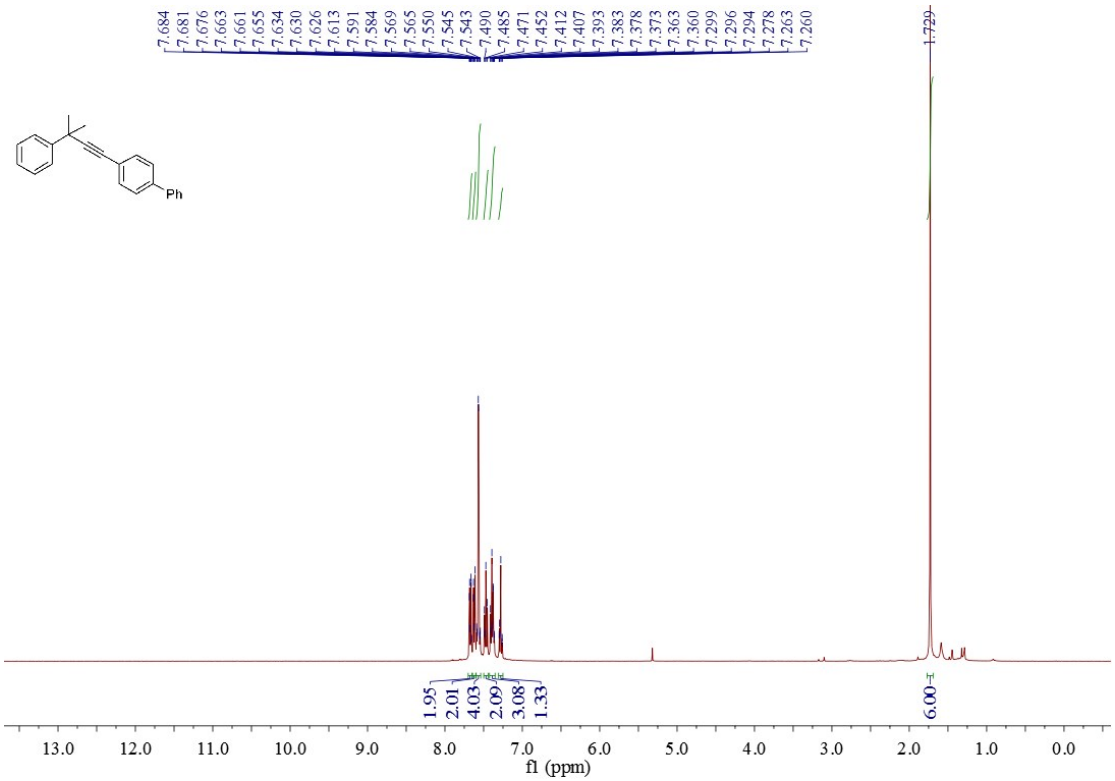


^{13}C-NMR

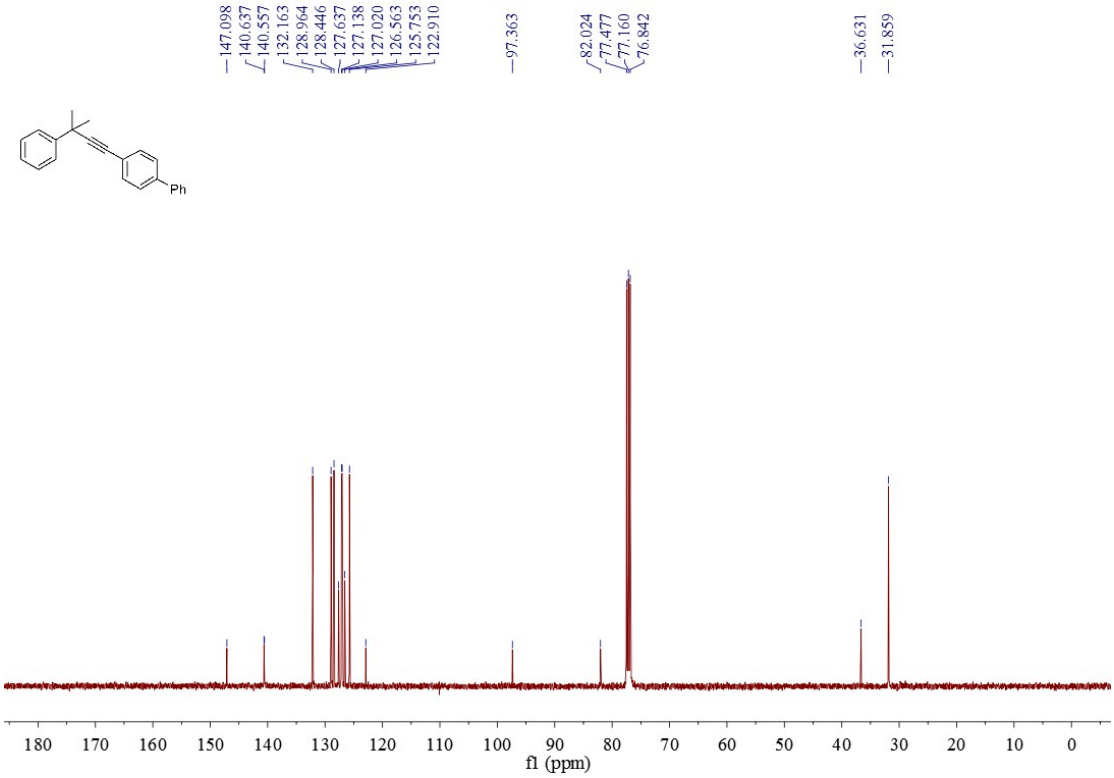


4-(3-methyl-3-phenylbut-1-yn-1-yl)-1,1'-biphenyl **c16**

^{1}H NMR

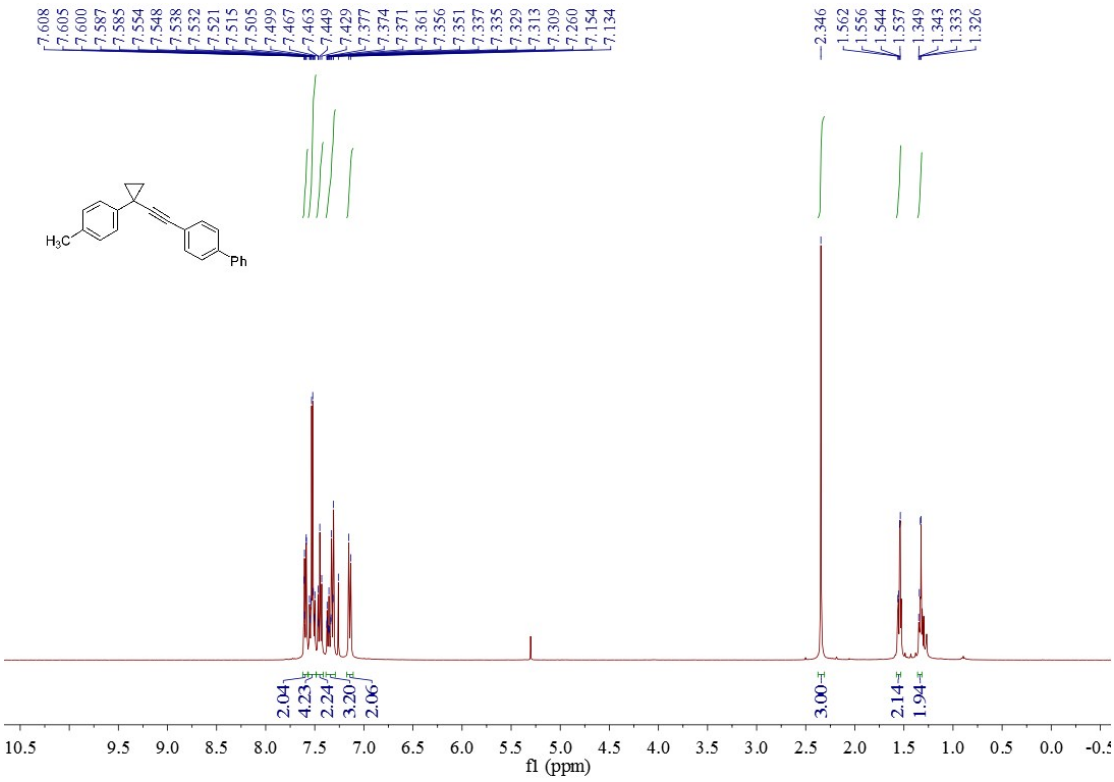


^{13}C NMR

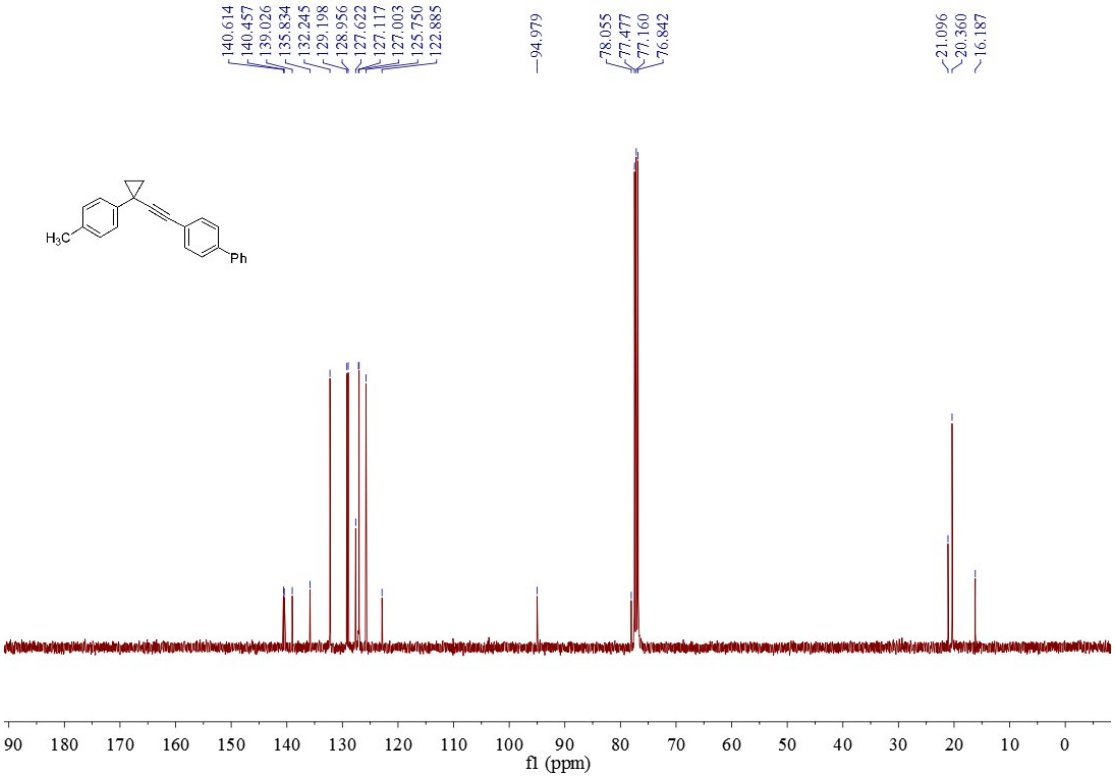


4-((1-(p-tolyl)cyclopropyl)ethynyl)-1,1'-biphenyl **c17**

^{1}H-NMR

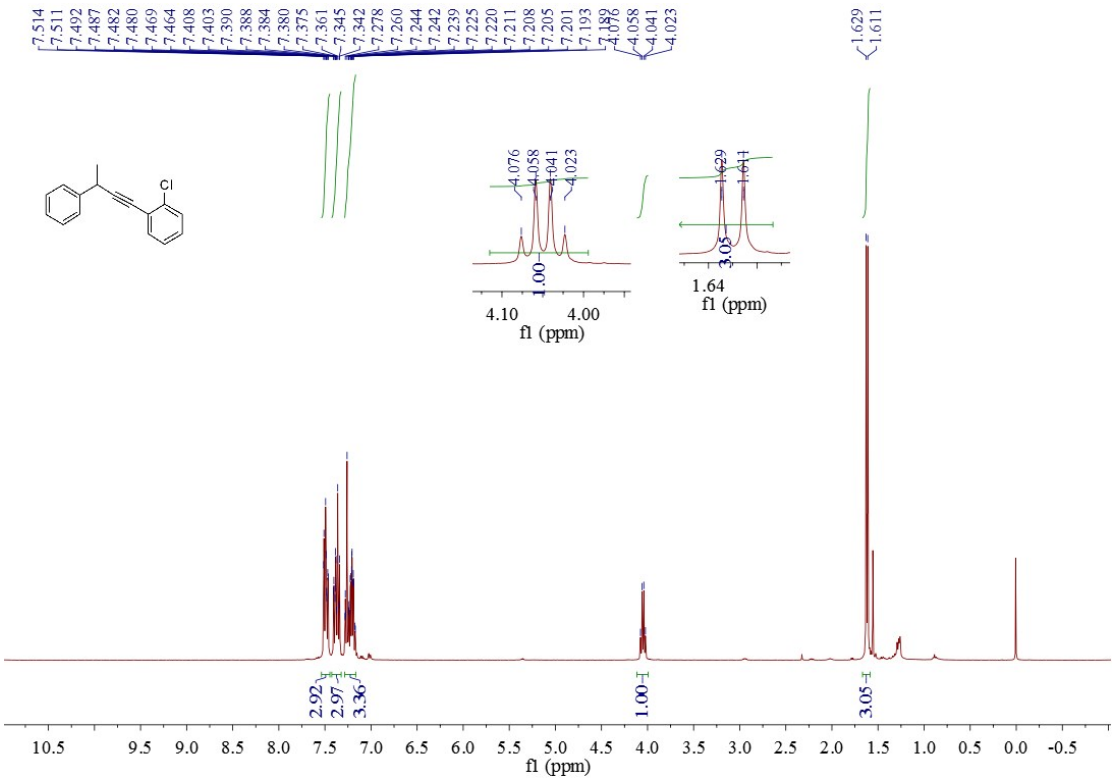


^{13}C-NMR

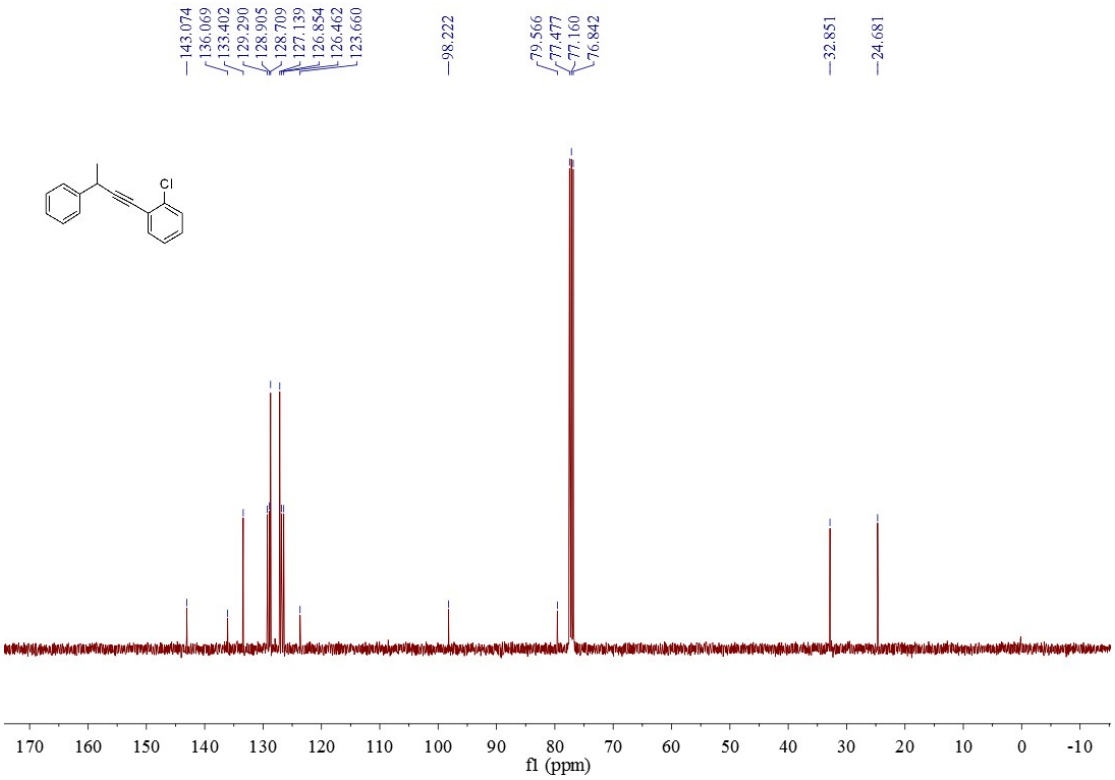


1-chloro-2-(3-phenylbut-1-yn-1-yl)benzene **(c18)**

^{1}H NMR

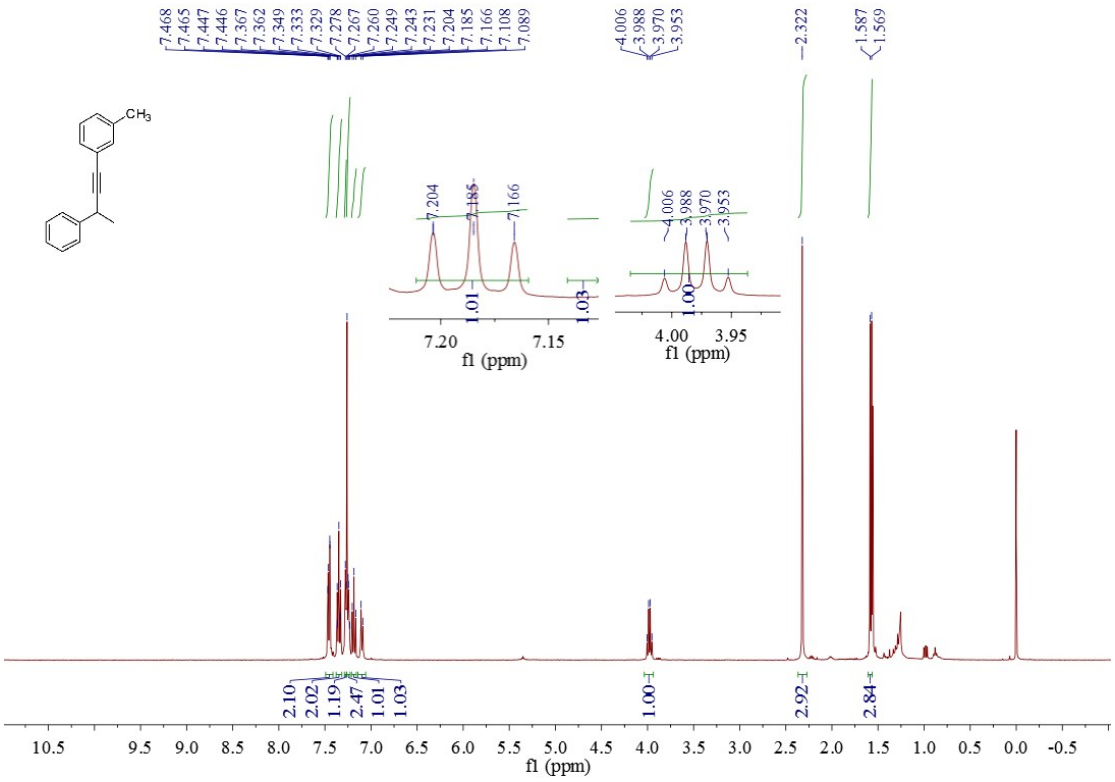


^{13}C NMR

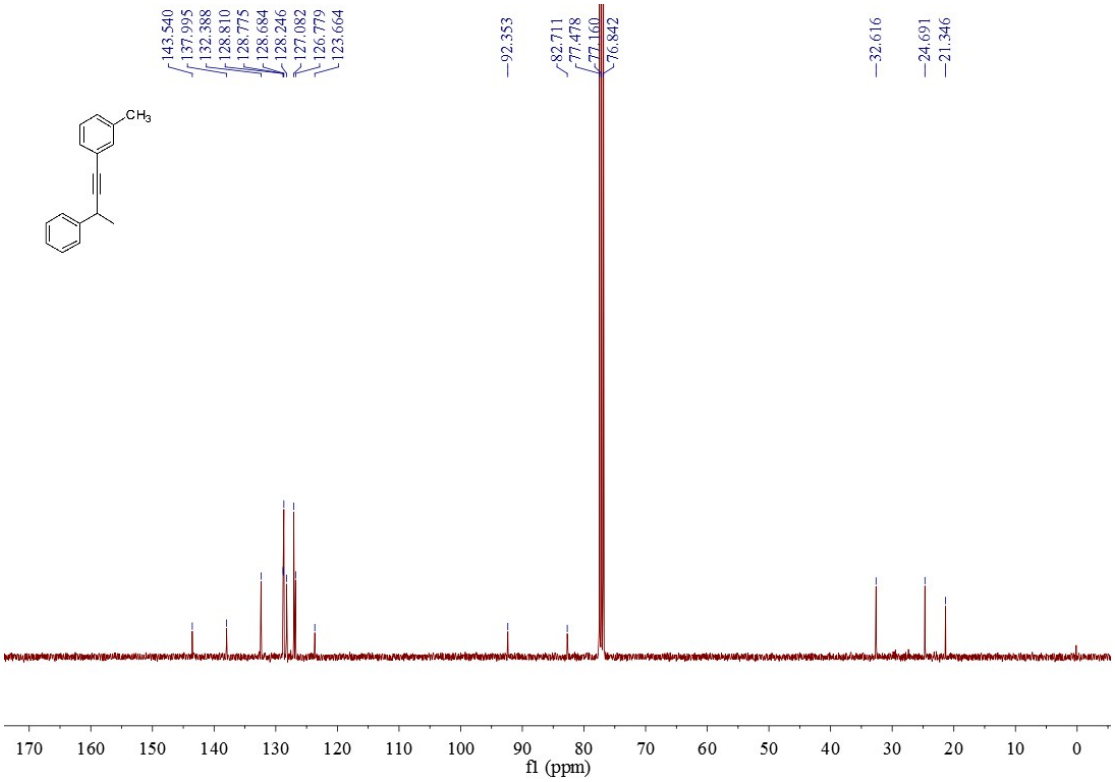


1-methyl-3-(3-phenylbut-1-yn-1-yl)benzene **c19**

^{1}H NMR

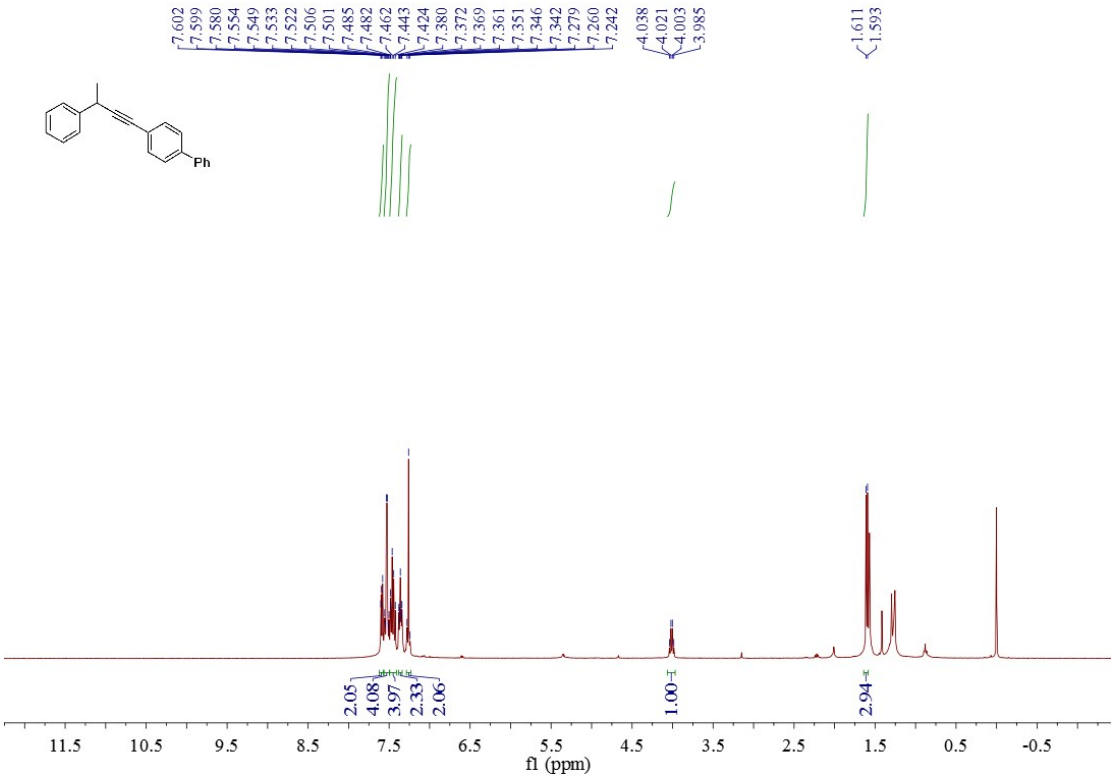


^{13}C NMR

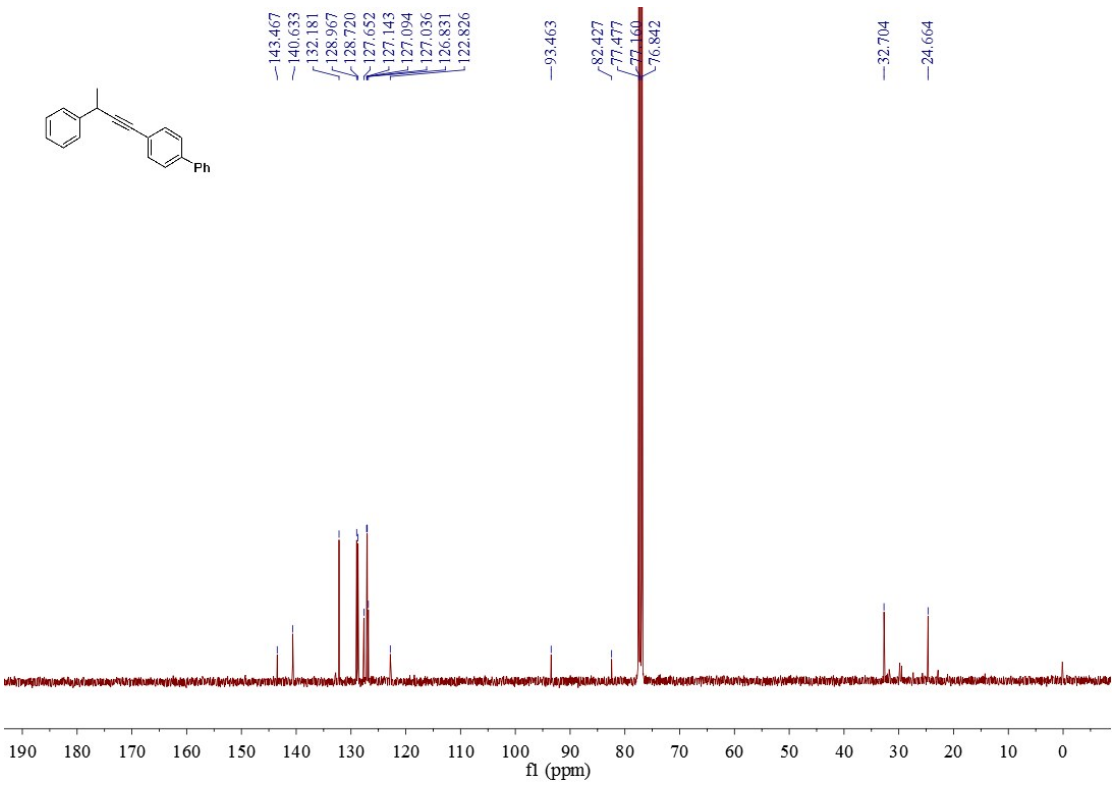


4-(3-phenylbut-1-yn-1-yl)-1,1'-biphenyl **c20**

^{1}H NMR

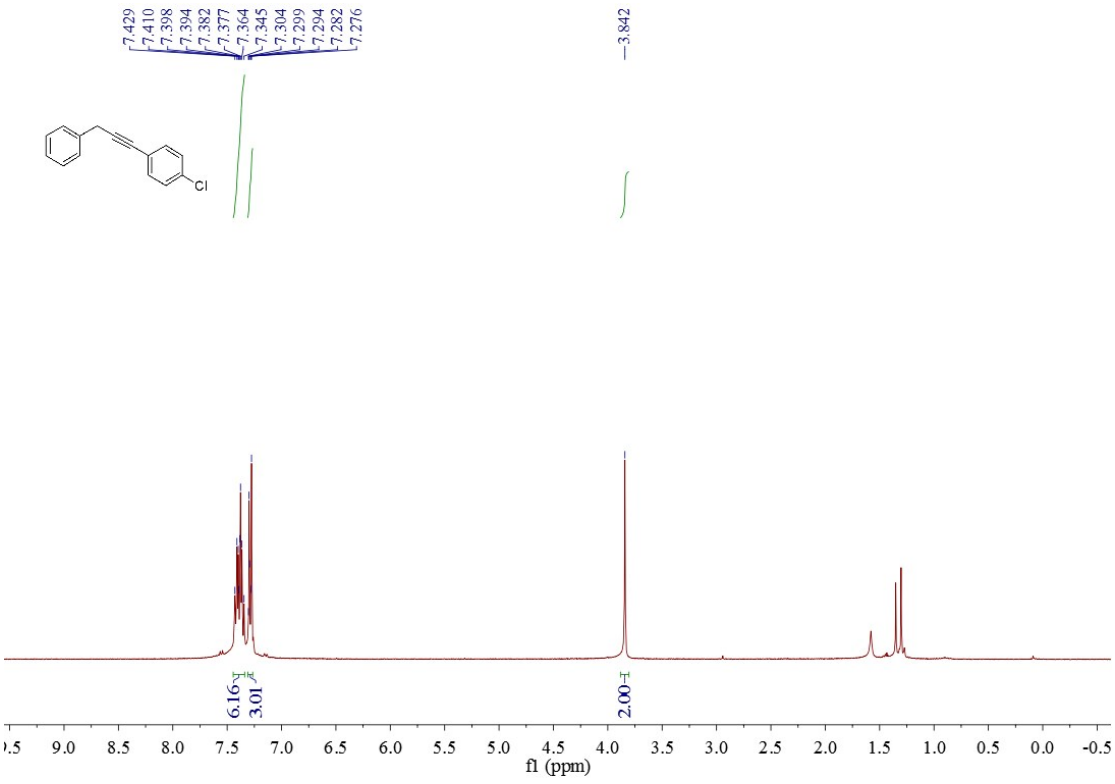


^{13}C NMR

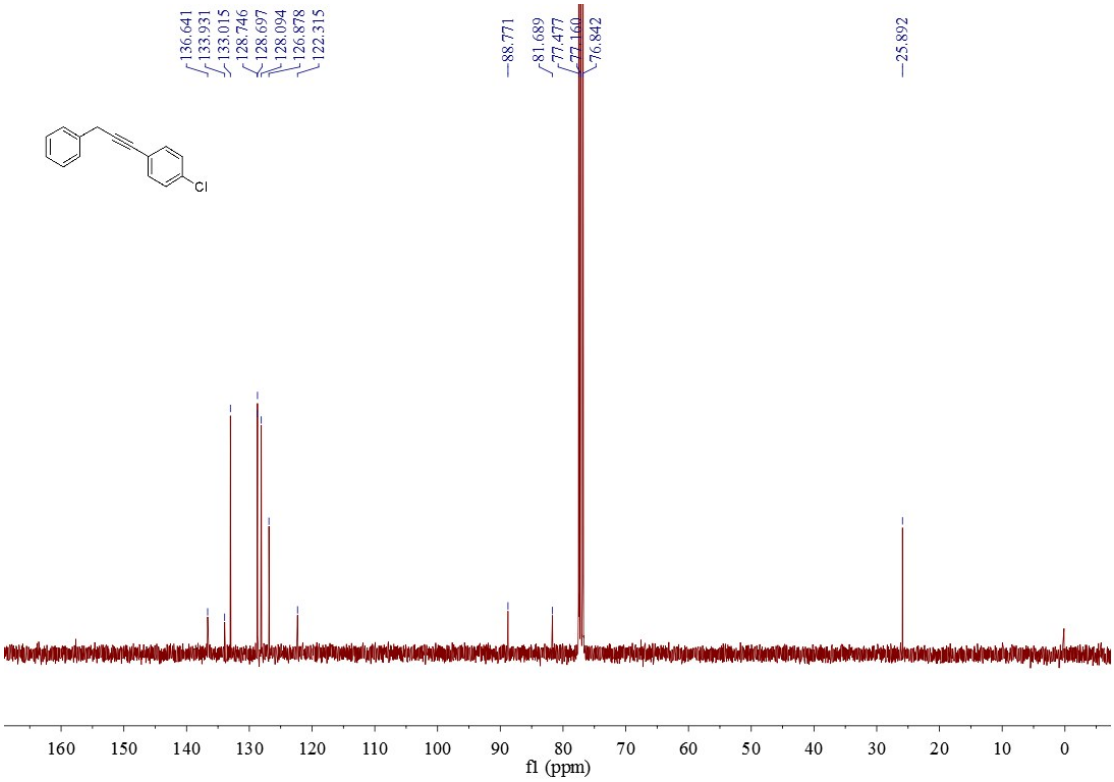


1-chloro-4-(3-phenylprop-1-yn-1-yl)benzene **c21**

^{1}H NMR

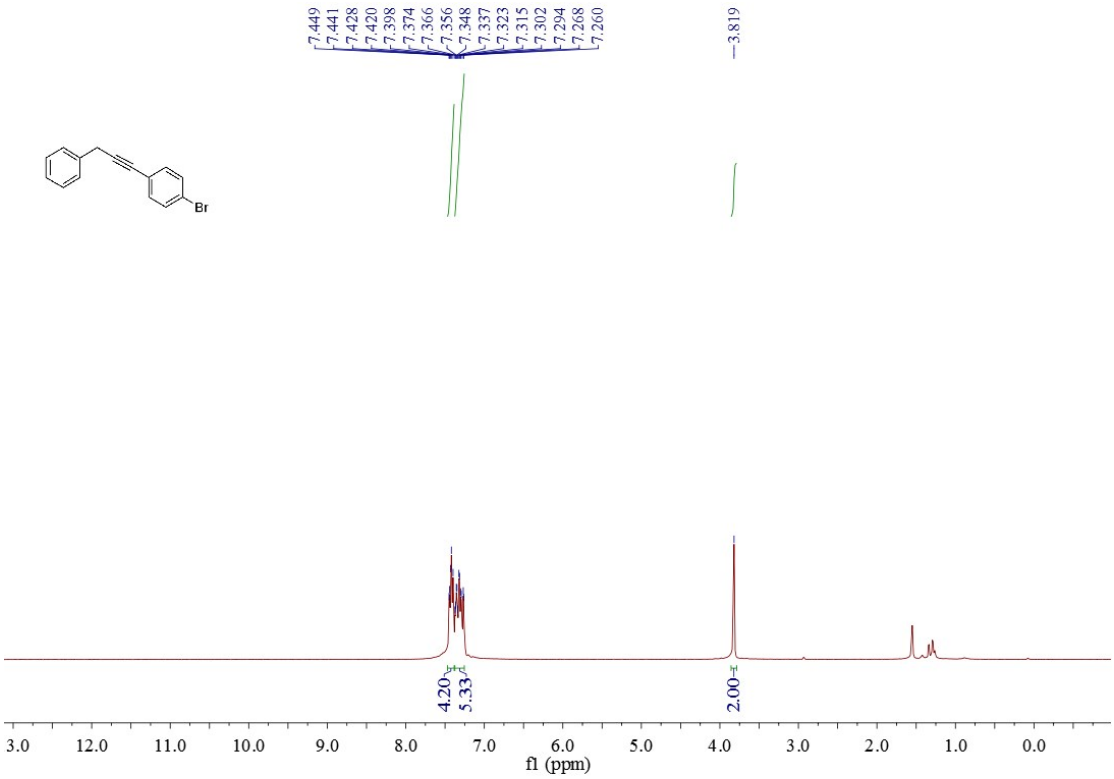


^{13}C NMR

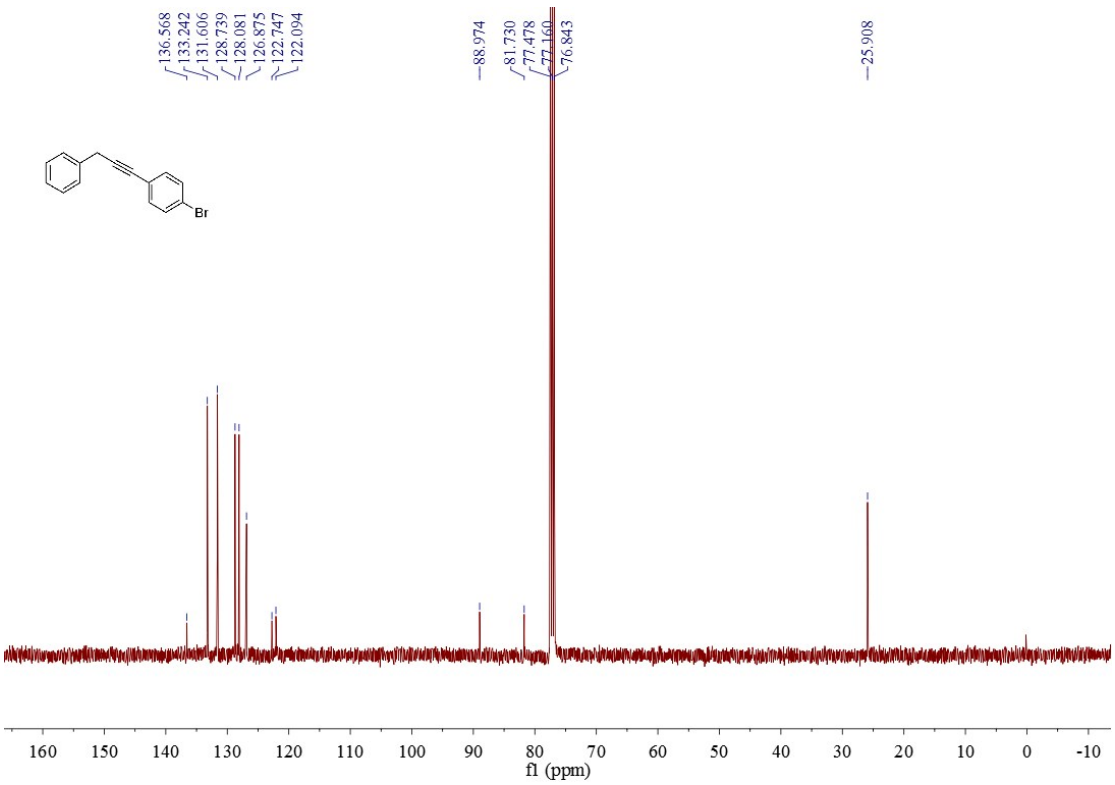


1-bromo-4-(3-phenylprop-1-yn-1-yl)benzene **c22**

^{1}H NMR

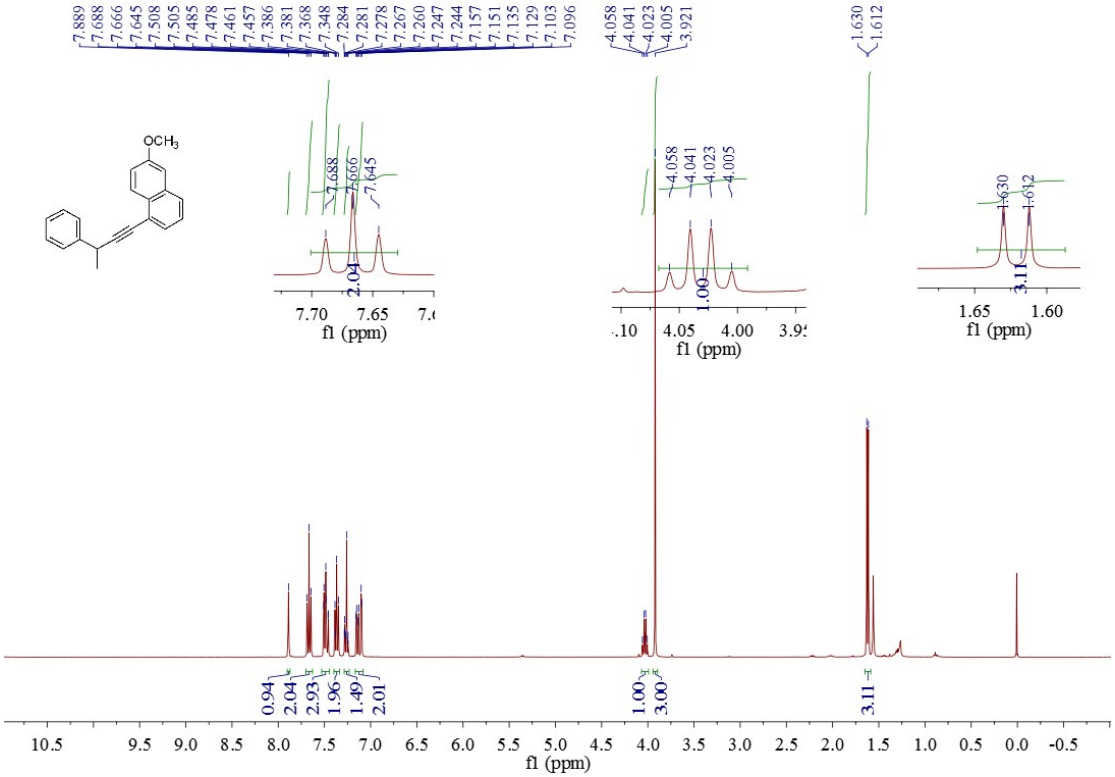


^{13}C NMR

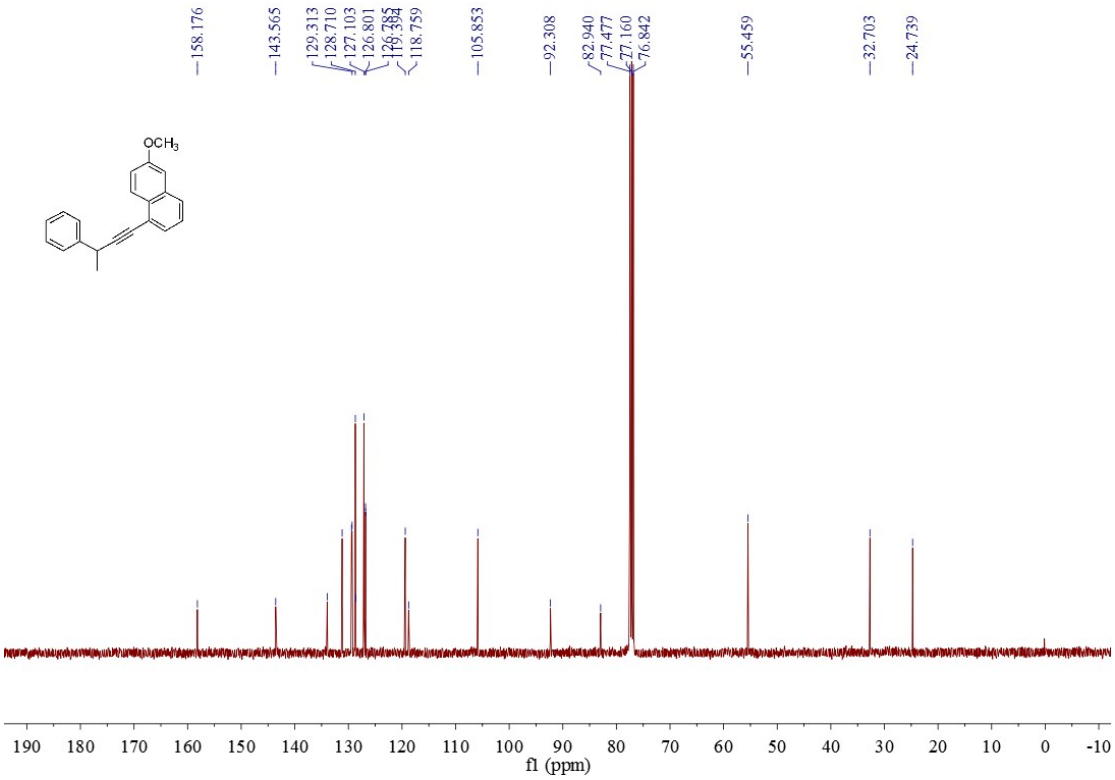


6-methoxy-1-(3-phenylbut-1-yn-1-yl)naphthalene **c23**

^{1}H NMR

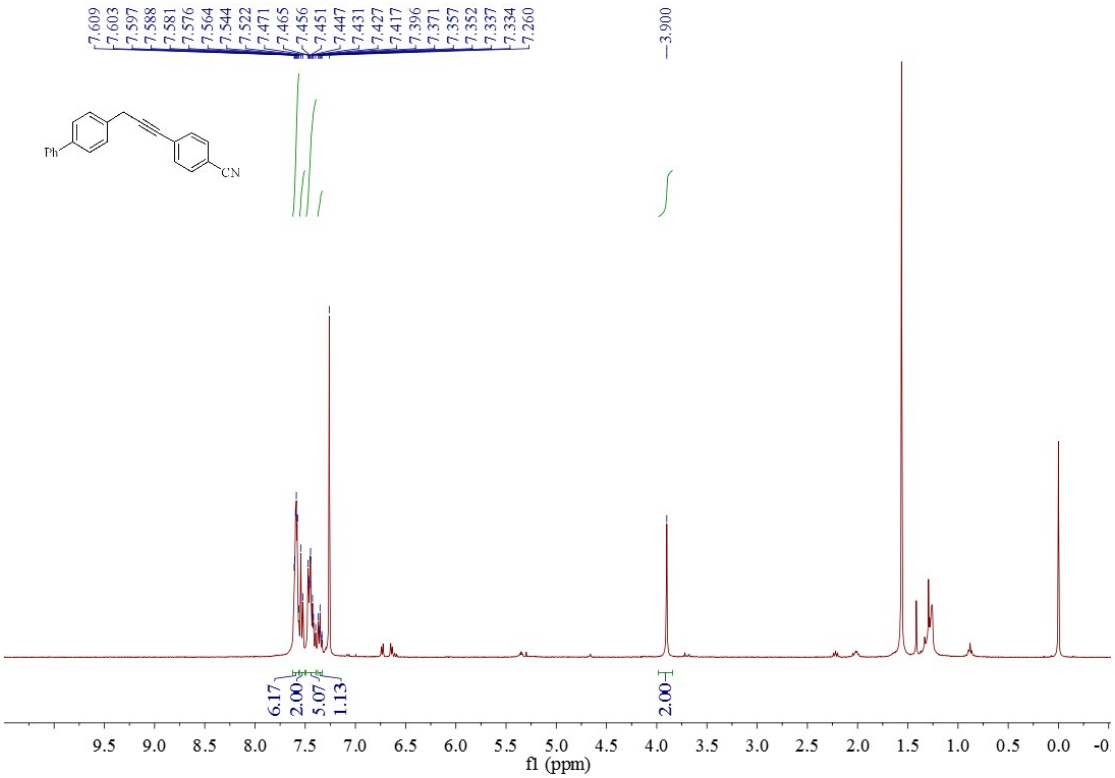


^{13}C NMR

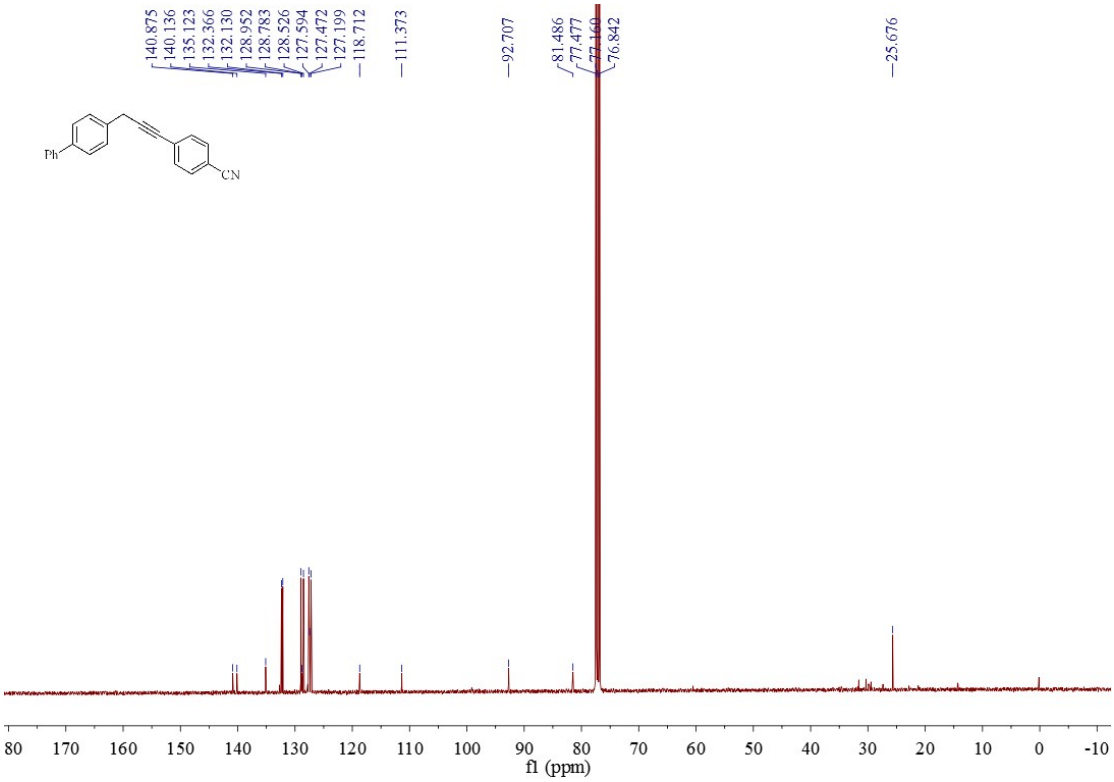


4-(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)benzonitrile **c24**

^{1}H NMR

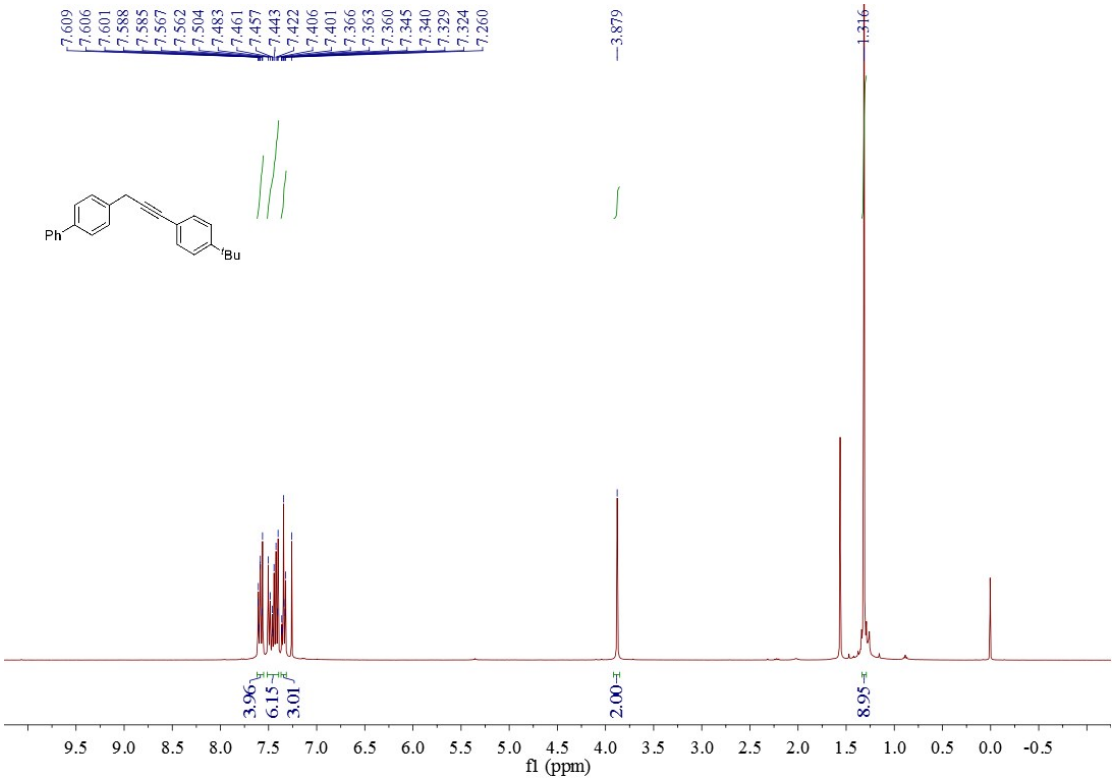


^{13}C NMR

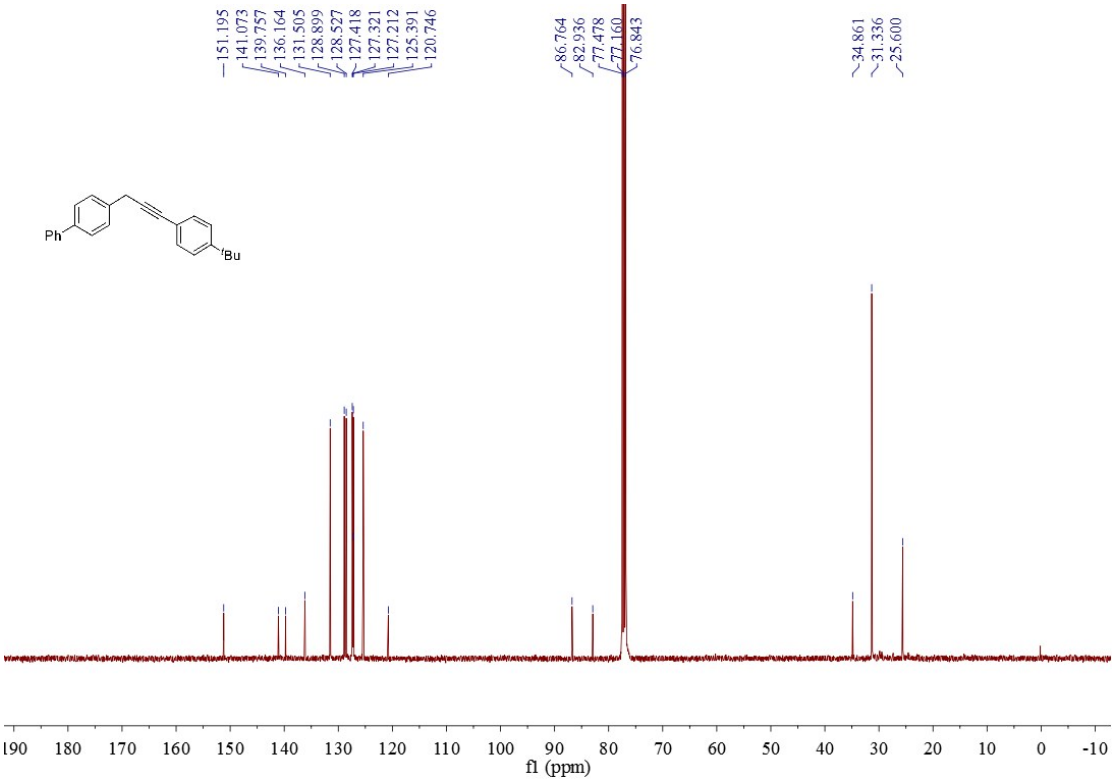


4-(3-(4-(tert-butyl)phenyl)prop-2-yn-1-yl)-1,1'-biphenyl **c25**

^{1}H NMR

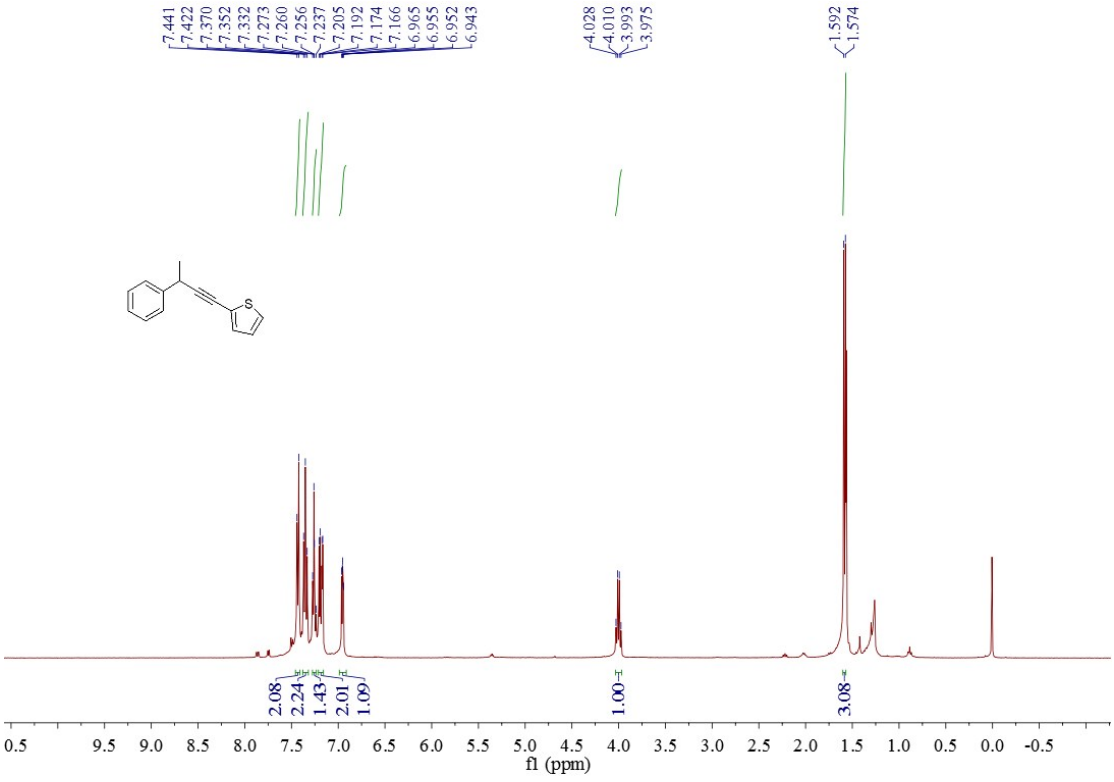


^{13}C NMR

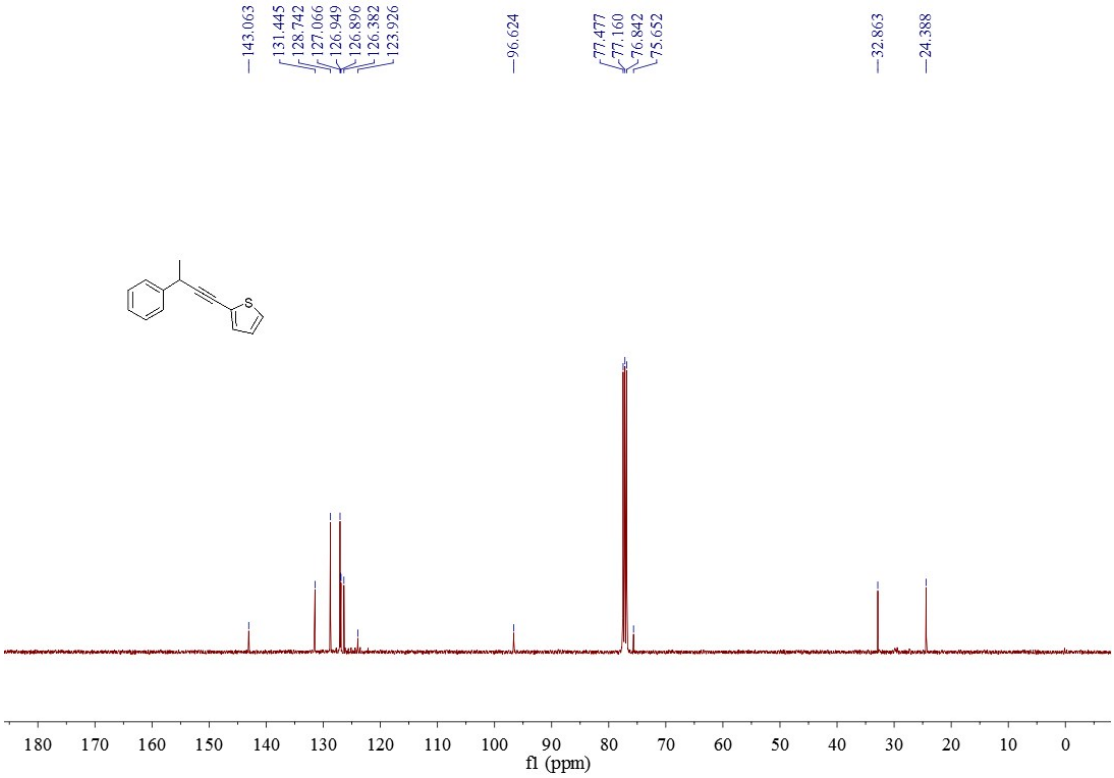


2-(3-phenylbut-1-yn-1-yl)thiophene **c26**

^{1}H NMR

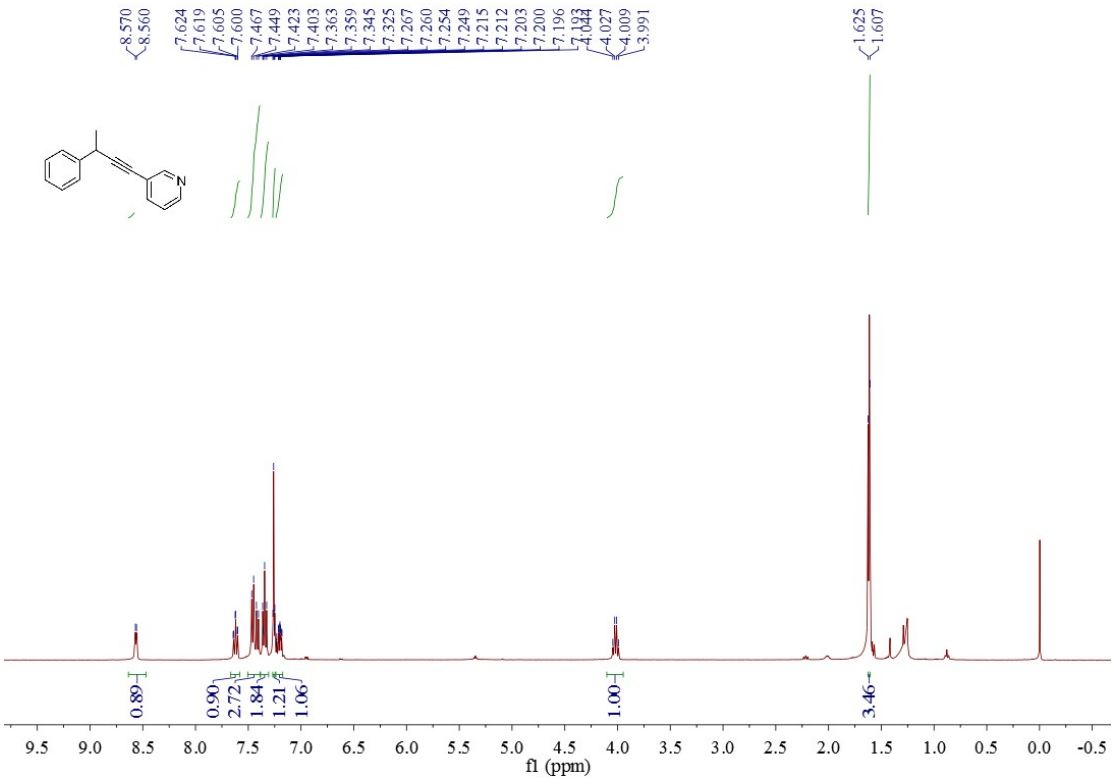


^{13}C NMR

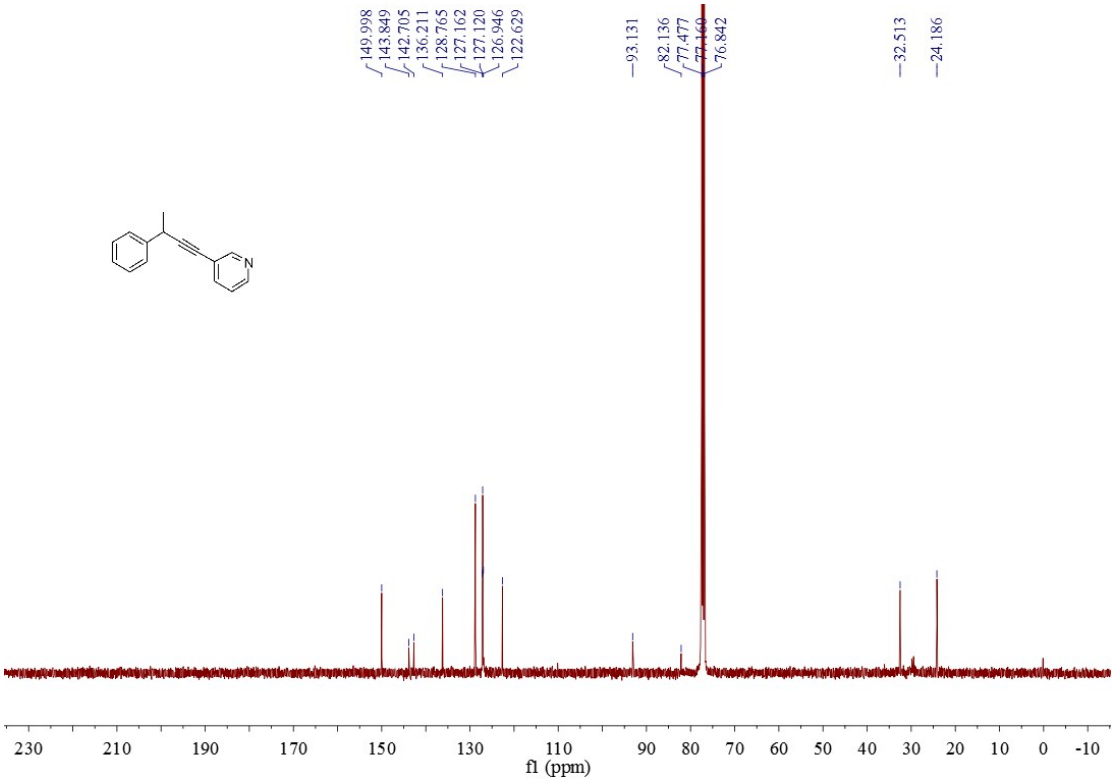


3-(3-phenylbut-1-yn-1-yl)pyridine **c27**

^{1}H NMR

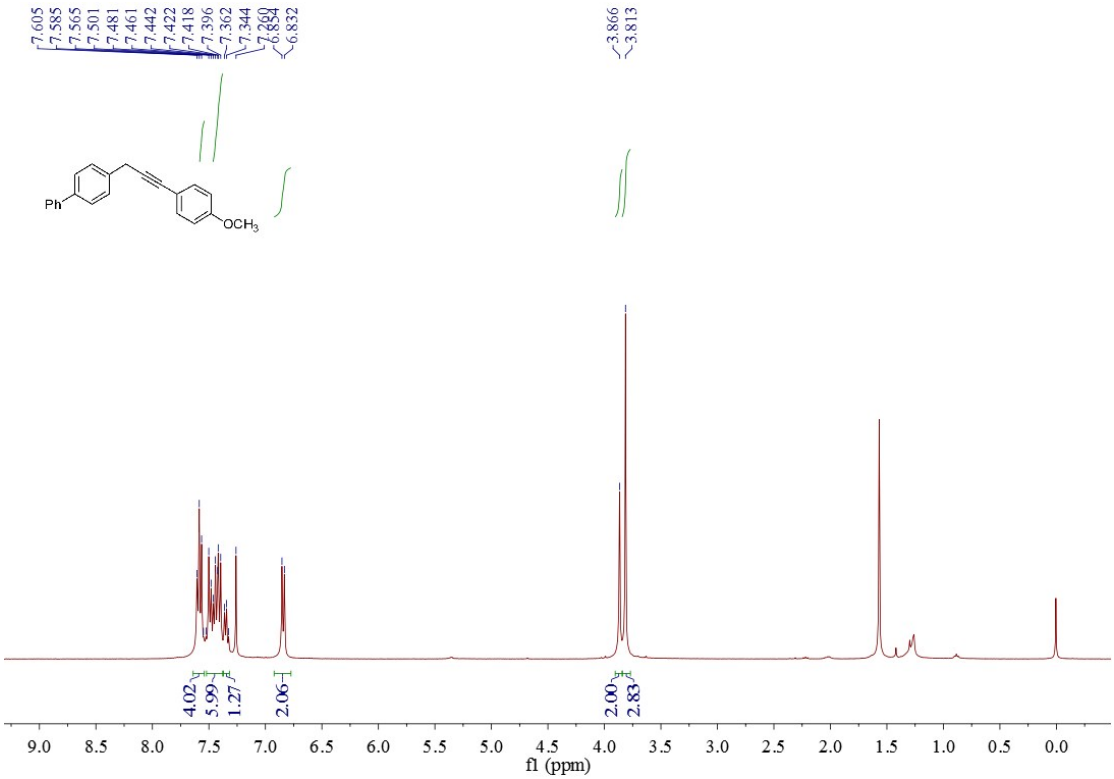


^{13}C NMR

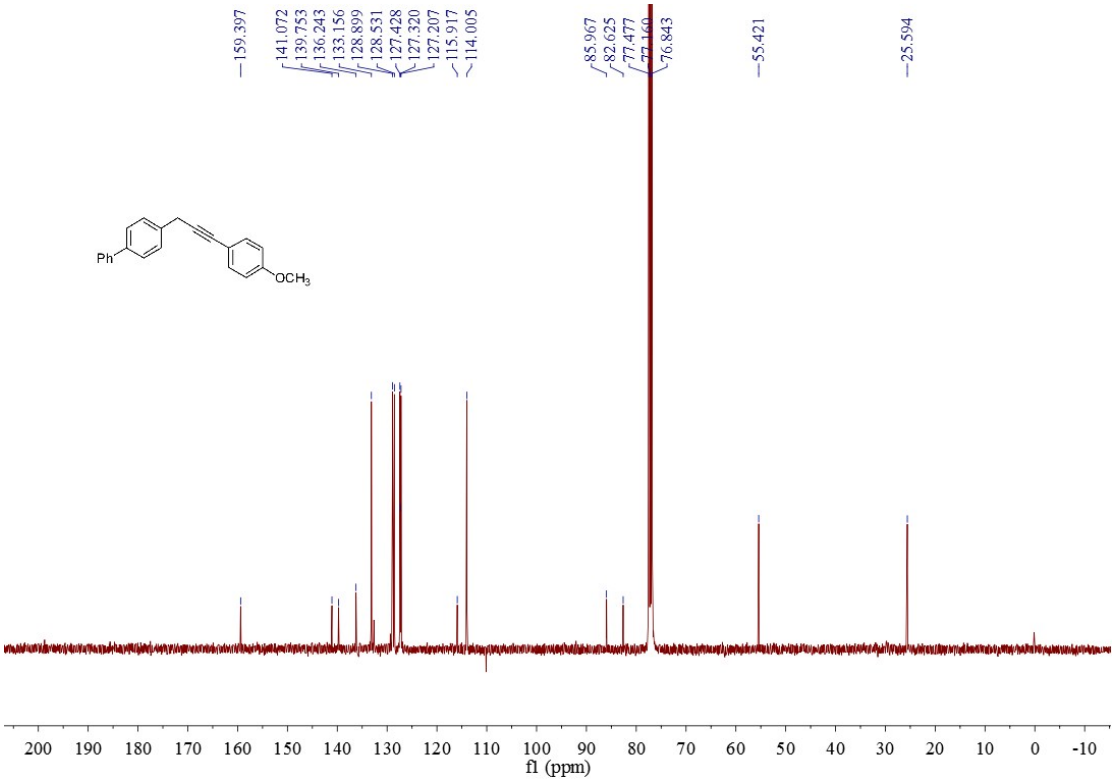


4-(3-(4-methoxyphenyl)prop-2-yn-1-yl)-1,1'-biphenyl **c28**

^{1}H NMR

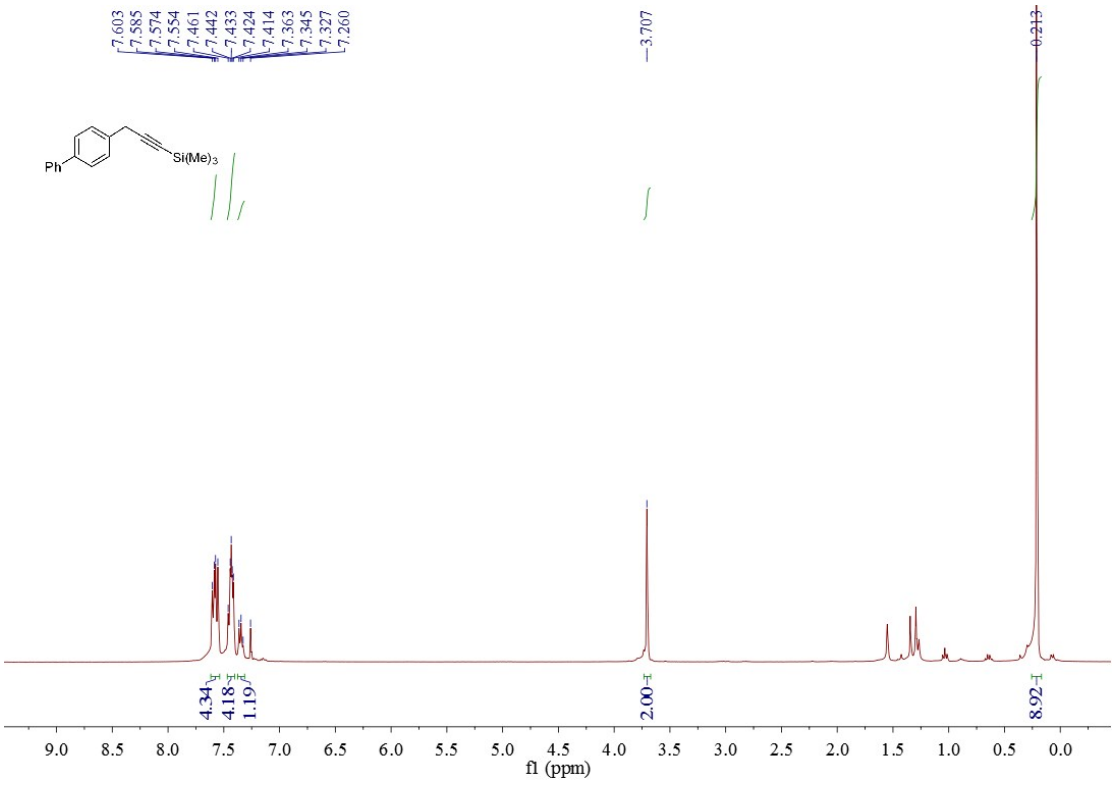


^{13}C NMR

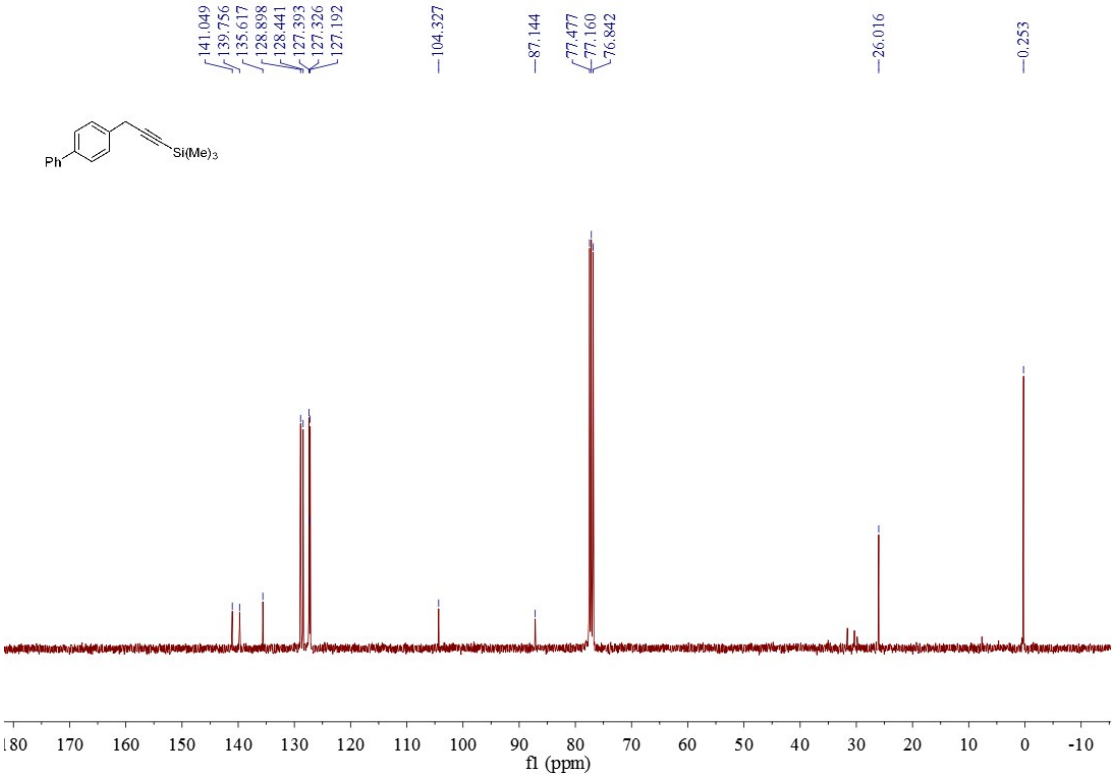


(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)trimethylsilane **c29**

^{1}H NMR

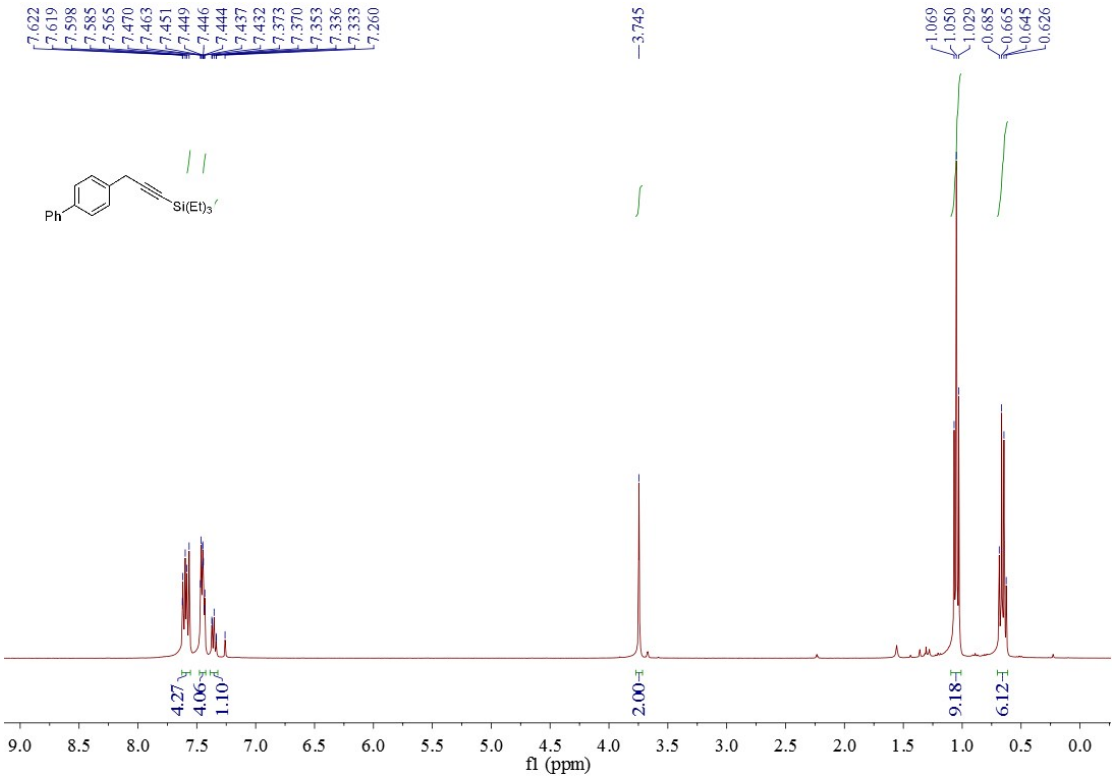


^{13}C NMR

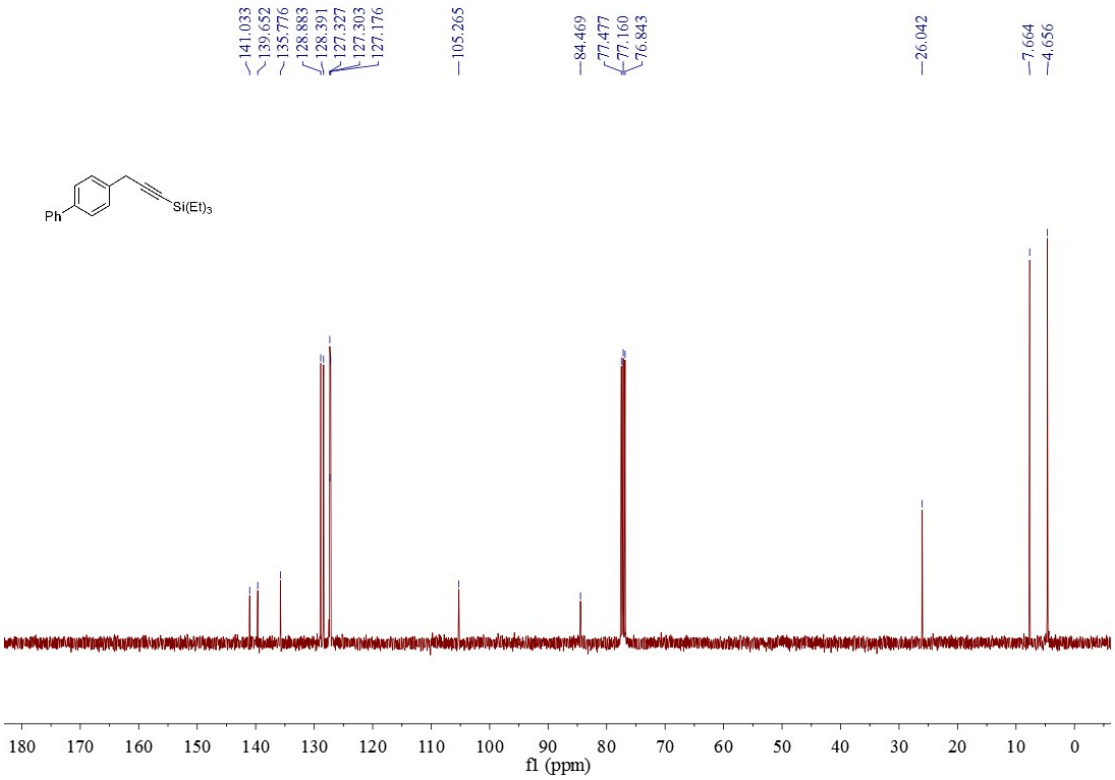


3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)triethylsilane **c30**

^{1}H NMR

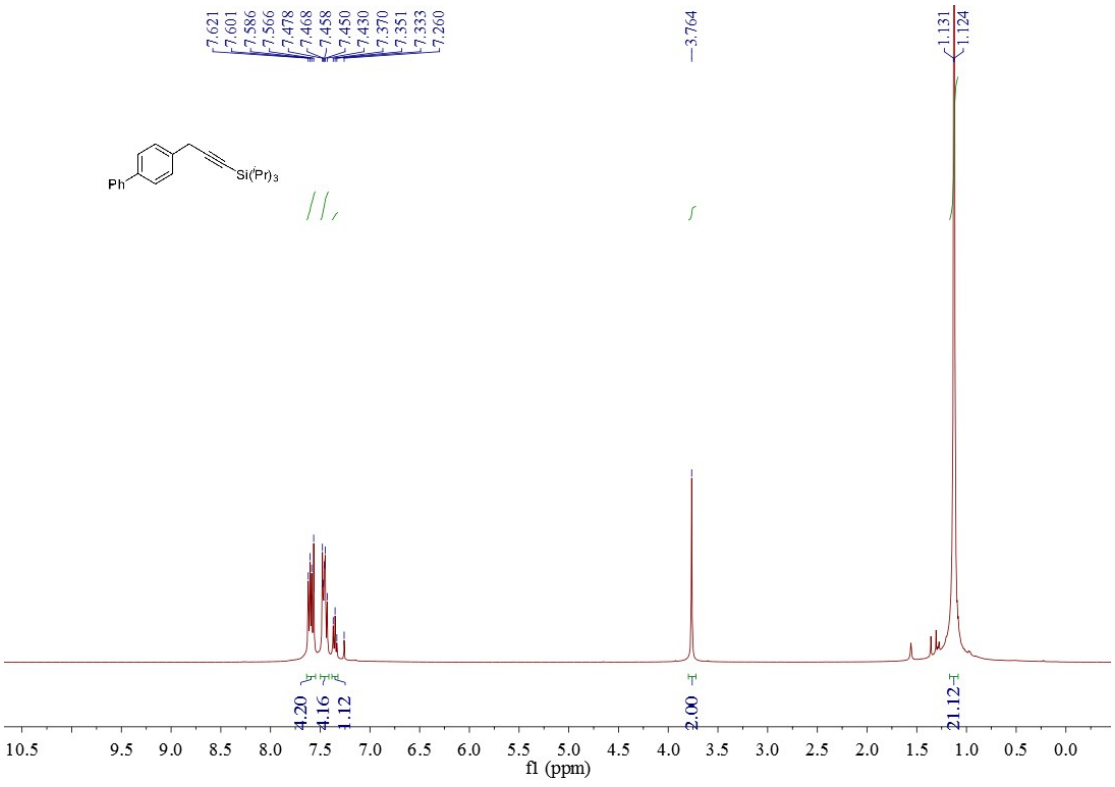


^{13}C NMR

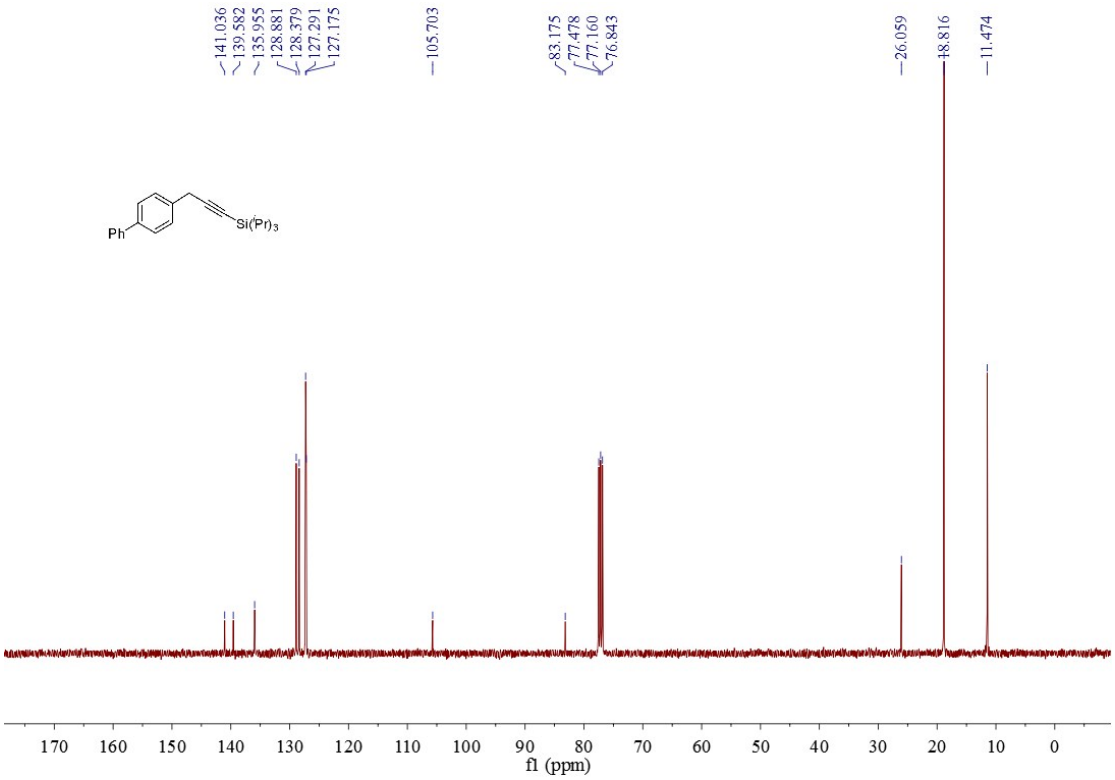


(3-([1,1'-biphenyl]-4-yl)prop-1-yn-1-yl)triisopropylsilane **c31**

^{1}H NMR

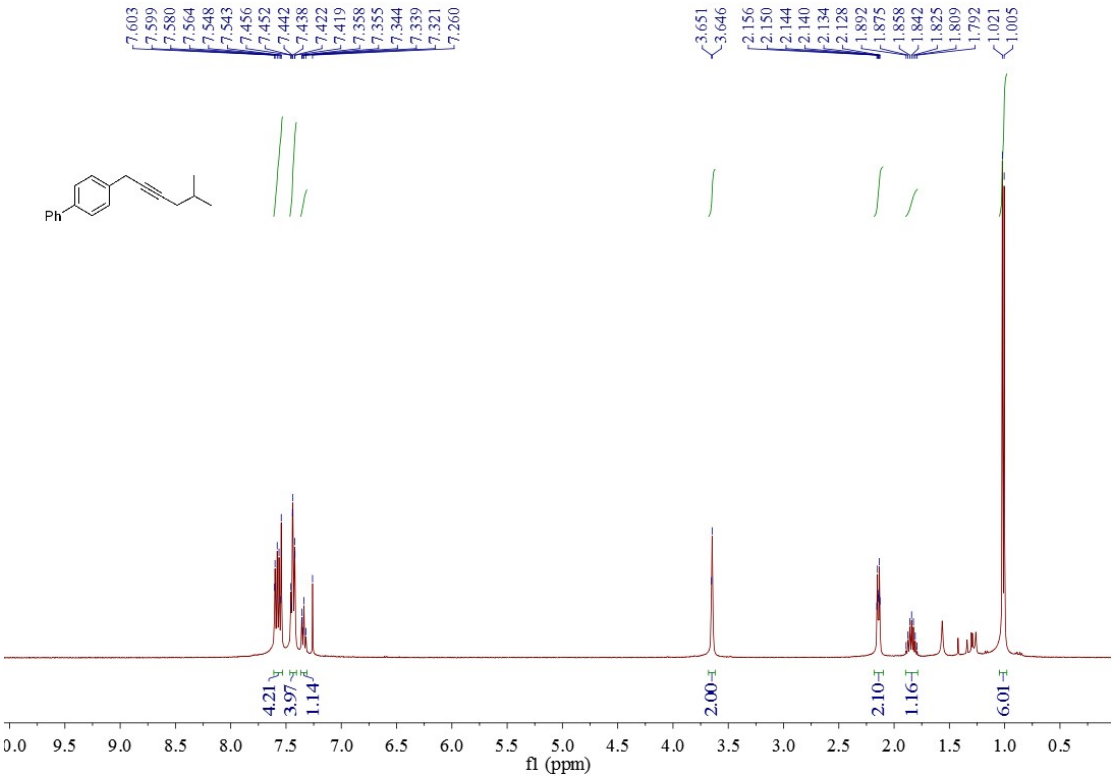


^{13}C NMR

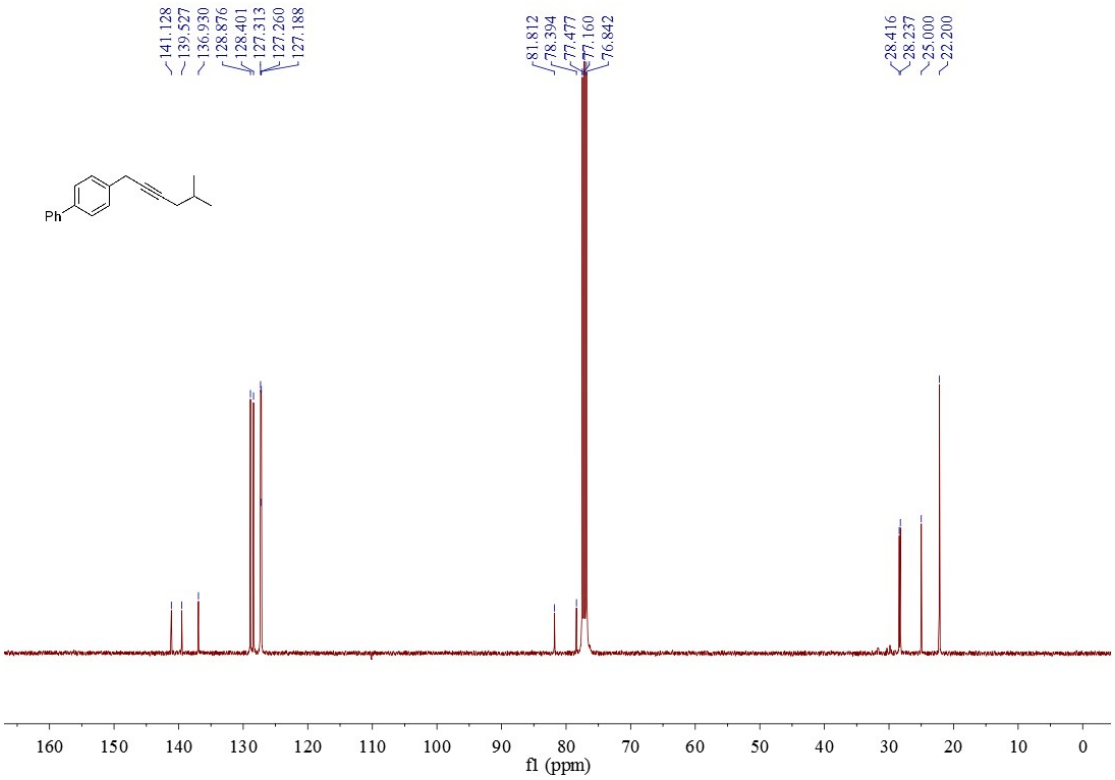


4-(5-methylhex-2-yn-1-yl)-1,1'-biphenyl **c32**

^{1}H NMR

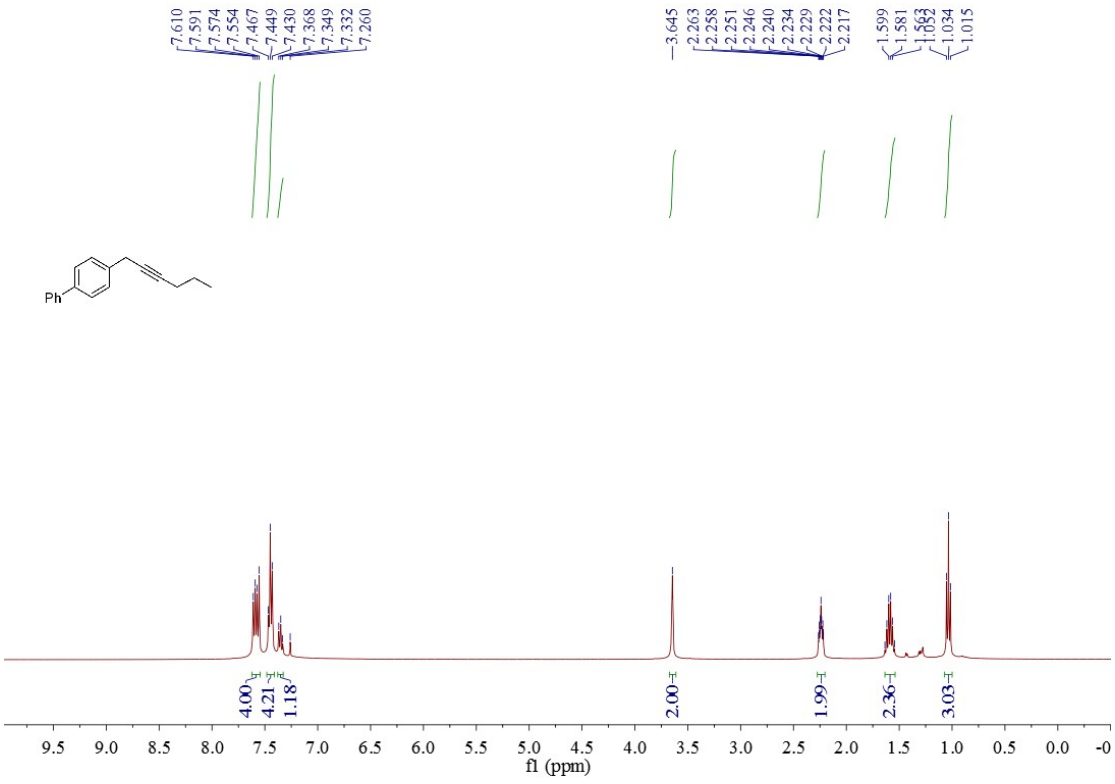


^{13}C NMR

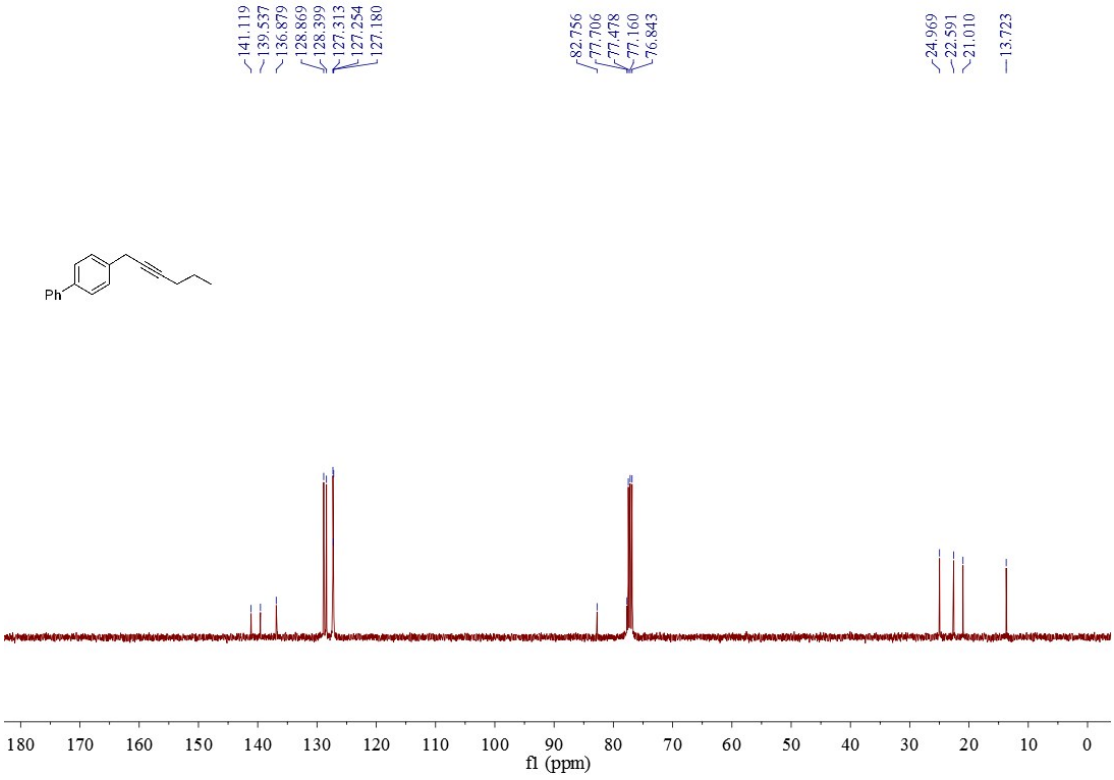


4-(hex-2-yn-1-yl)-1,1'-biphenyl **c33**

^{1}H NMR

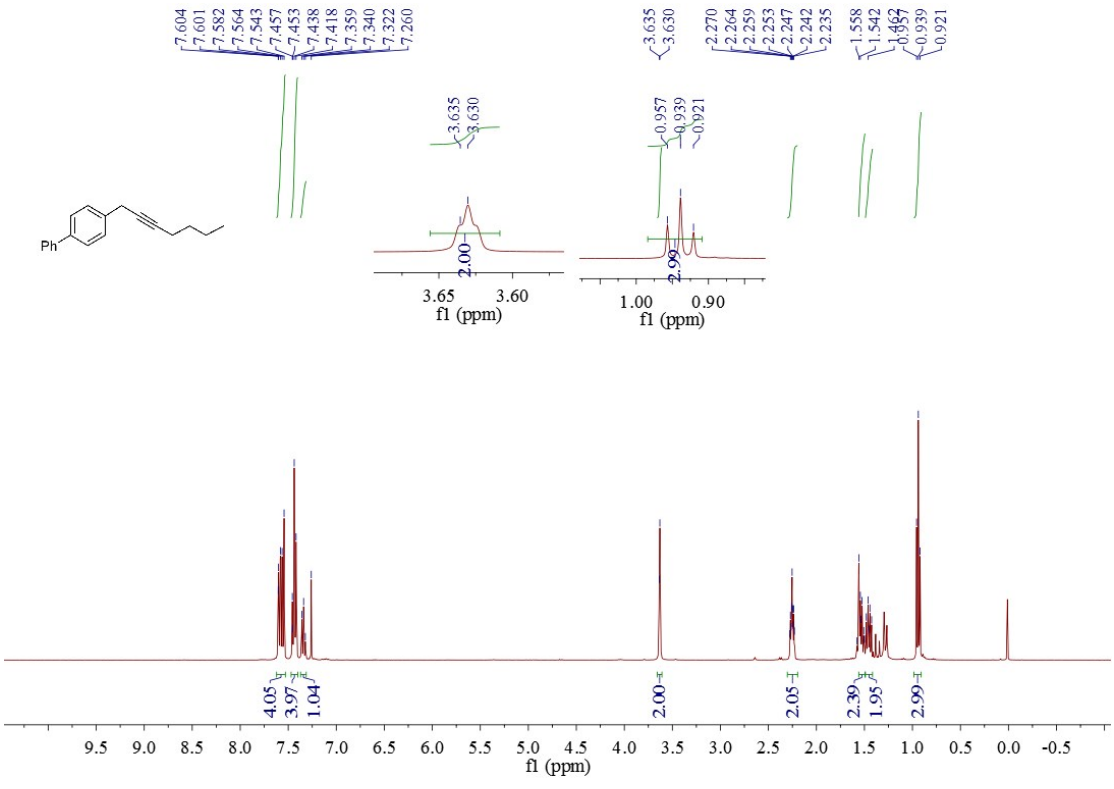


^{13}C NMR

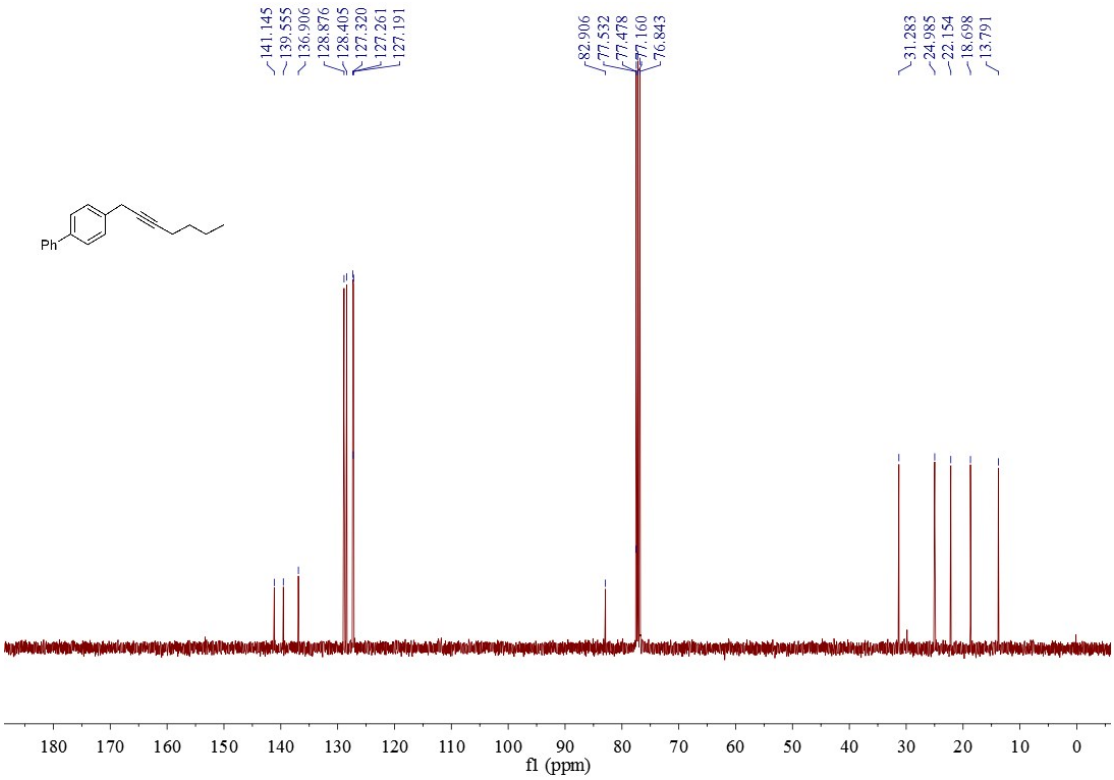


4-(hept-2-yn-1-yl)-1,1'-biphenyl **c34**

^{1}H NMR

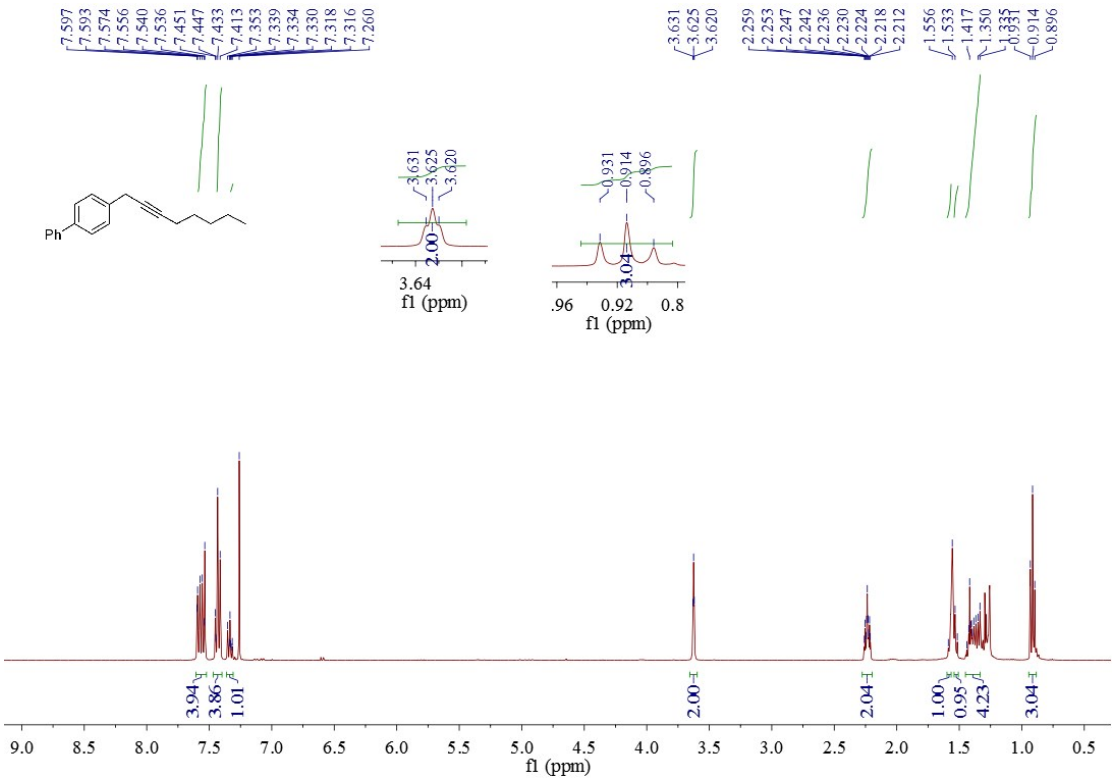


^{13}C NMR

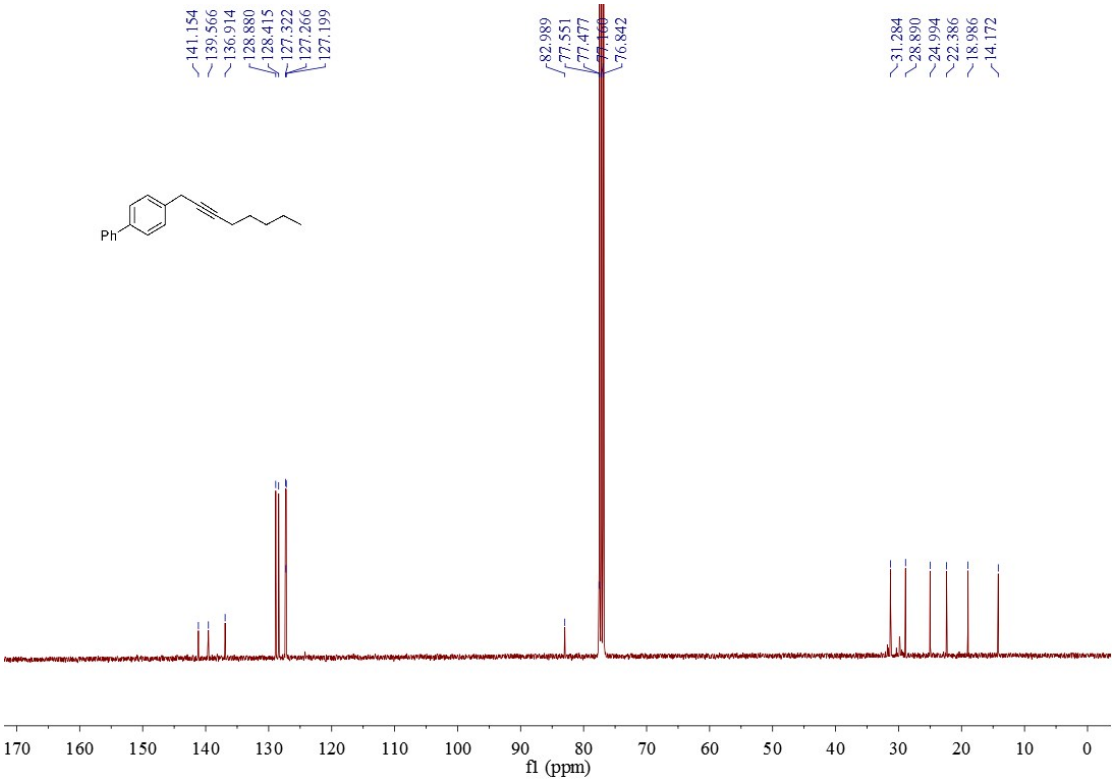


4-(oct-2-yn-1-yl)-1,1'-biphenyl **c35**

^{1}H NMR

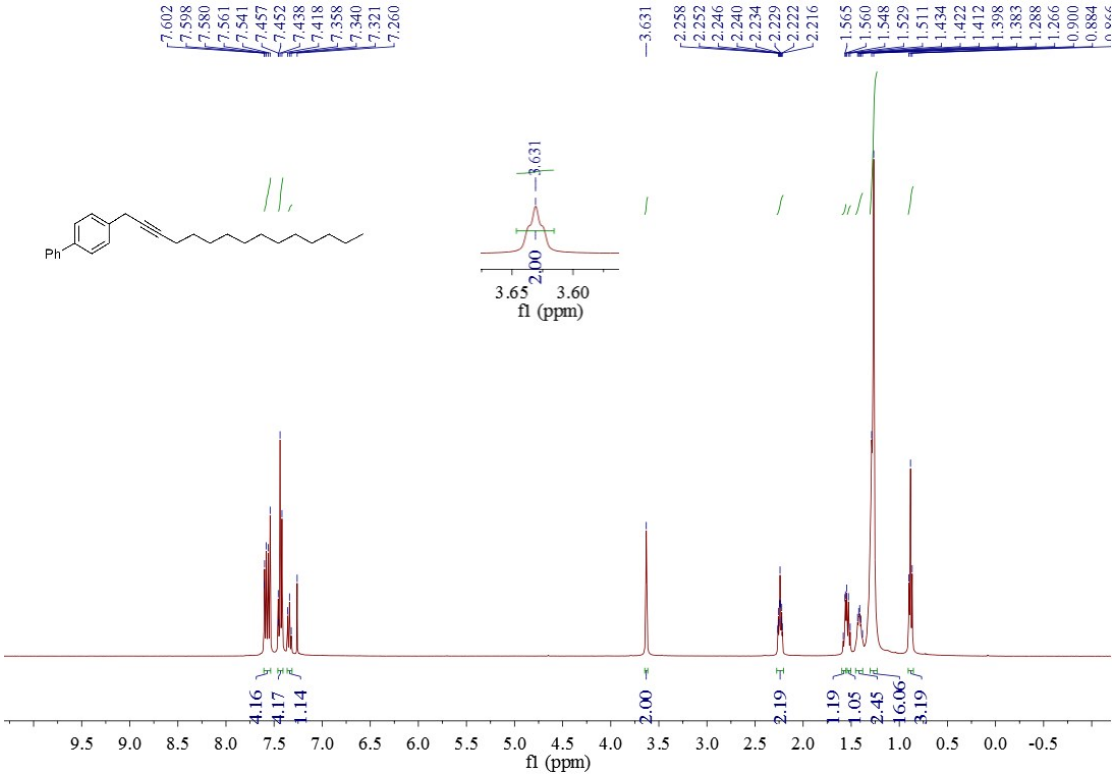


^{13}C NMR

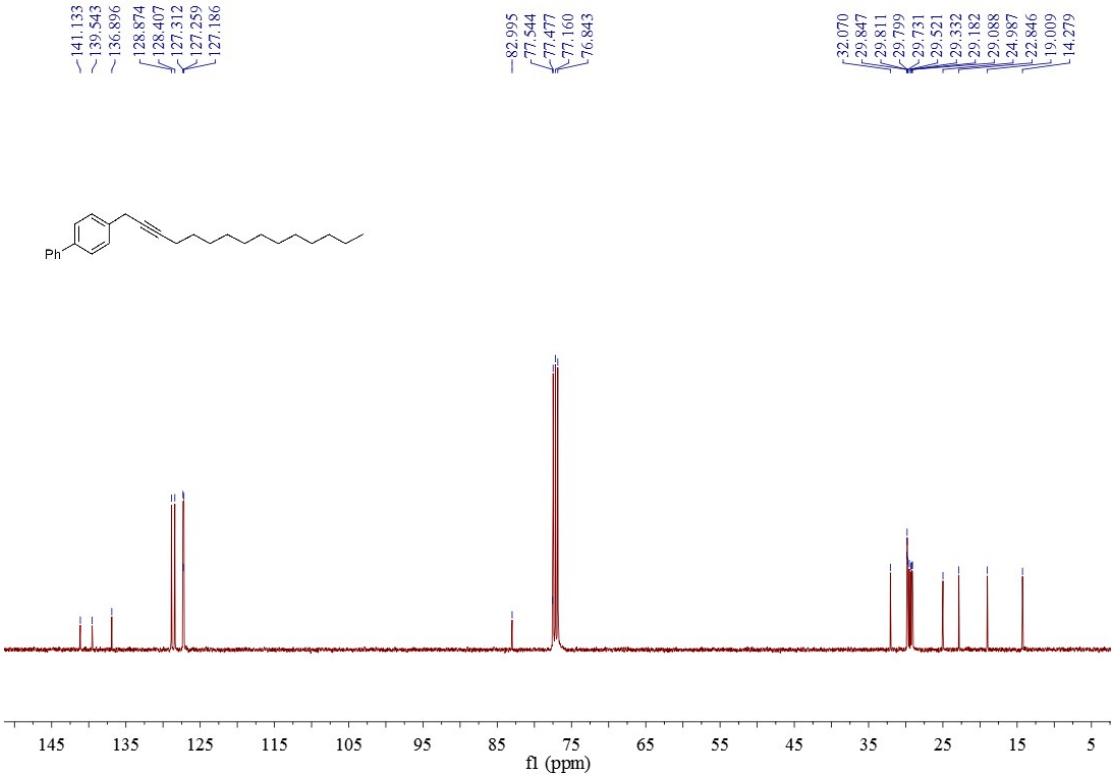


4-(pentadec-2-yn-1-yl)-1,1'-biphenyl **c36**

^{1}H NMR



^{13}C NMR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139.543
136.896
128.874
128.407
127.312
127.259
127.186
82.995
77.544
77.477
77.160
76.843
32.070
29.847
29.811
29.799
29.731
29.521
29.332
29.182
29.088
24.987
22.846
19.009
14.279
Ph
145
135
125
115
105
95
85
75
65
55
45
35
25
15
5
f1 (ppm)