Copper-catalyzed tandem direct-monodesilylation/ CuAAC reaction to synthesize 4-(TMS-ethynyl)-1,2,3-triazoles from 1,4-Bis(trimethylsilyl)Butadiyne with azides

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1. General methods

1. General Information

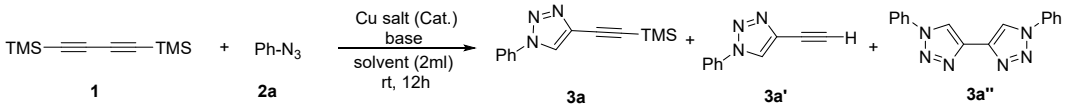
Melting points were measured with a Beijing-Taike X-4 apparatus without corrected.

NMR spectra were recorded on a Bruker 400 MHz spectrometer (^{1}H NMR at 400 MHz, ^{13}C{^{1}H} NMR at 101 MHz, ^{19}F NMR at 377 MHz) or a JEOL 600 MHz spectrometer (^{1}H NMR at 600 MHz, ^{13}C{^{1}H} NMR at 151 MHz, ^{19}F NMR at 565 MHz), with $CDCl_3$ or DMSO-d_6 as solvents unless noted otherwise. Chemical shifts are reported in ppm with TMS as an internal standard. HRMS analyses were performed on Thermo Scientific Q Exactive Focus Orbitrap. Column chromatography was carried out on silica gel (200-300 mesh). All chemicals used were of reagent grade. Solvents were commercial grade and dried via standard procedures. Reactions were performed in a closed system.

CAUTION: Organic azides are potentially explosive compounds with the sensitivity towards shock, temperature, light and other influences. Whilst no problems were encountered by the authors, caution must be exercised when handling.

2. Optimization of Reaction Conditions

Table S1. Optimization of Reaction Conditions[a]



Entry	alkyne	Cat./ligand	Solvent	Yield [%][b]		
				3a	**3a'**	**3a''**
1	1.25	CuI/Et_3N	DMF	52	4	8
2	1.4	CuI/Et_3N	DMF	37	4	9
3	1	CuI/Et_3N	DMF	43	11	8
4	0.8	CuI/Et_3N	DMF	40	10	10
5	1.25	CuI/Et_2NH	DMF	51	4	10
6	1.25	CuI/TMEDA	DMF	60	7	12
7	1.25	CuI/Pyridine	DMF	70	8	11
8	1.25	CuI/Pyridine[c]	DMF	n.d.	n.d.	trace
9	1.25	CuI/DMAP	DMF	56	7	9
10	1.25	CuI/DIPEA	DMF	45	2	12
11	1.25	CuI/DBU	DMF	29	16	5
12	1.25	CuI/K_2CO_3	DMF	49	14	10

13	1.25	CuI/1,10-phen	DMF	51	12	17
14	1.25	CuI/4,4’-dimethyl-2,2’-bipy	DMF	36	28	17
15	1.25	CuI/TABF	DMF	n.d.	n.d.	n.d.
16	1.25	CuI/*t*-BuOK	DMF	n.d.	n.d.	n.d.
17	1.25	CuCl/Pyridine	DMF	57	13	17
18	1.25	CuBr/Pyridine	DMF	45	12	17
19	1.25	$Cu(phen)_2I$/Pyridine	DMF	57	10	23
20	1.25	$Cu(phen)_2Br$/Pyridine	DMF	46	20	21
21	1.25	$Cu(phen)_2Cl$/Pyridine	DMF	51	19	14
22	1.25	$Cu(PPh_3)_2Cl$/Pyridine	DMF	44	11	19
23	1.25	$Cu(PPh_3)_2Br$/Pyridine	DMF	46	5	14
24	1.25	$Cu(phen)(PPh_3)_2NO_3$/Pyridine	DMF	37	20	13
25	1.25	$Cu(PPh_3)_2NO_3$/Pyridine	DMF	51	10	6
26	1.25	CuI/Pyridine	THF	n.d. [d]	n.d.	Trace
27	1.25	CuI/Pyridine	CH_3CN	n.d.	n.d.	Trace
28	1.25	CuI/Pyridine	$CHCl_2$	n.d.	n.d.	Trace
29	1.25	CuI/Pyridine	PhMe	n.d.	n.d.	Trace
30	1.25	CuI/Pyridine	DMSO	Trace	Trace	Trace
31	1.25	CuI/Pyridine	DMA	66	6	13
32	1.25	CuI/Pyridine	NMP	53	7	17
33[e]	1.25	$CuSO_4·5H_2O$/sodium ascorbate/without Pyridine	DMF/H_2O[f]	n.d.	n.d.	5
34[e]	1.25	$CuSO_4·5H_2O$/sodium ascorbate/Pyridine	DMF/H_2O[f]	n.d.	n.d.	5
35[e]	1.25	$CuSO_4·5H_2O$/sodium ascorbate/without Pyridine	THF/H_2O[f]	n.d.	n.d.	trace[e]

36[e]	1.25	$CuSO_4·5H_2O$/sodium ascorbate/Pyridine	THF/H_2O[f]	22	9	5[e]

[a] A 10 mL vial containing BTMSBD (0.5 mmol, 1.25 equiv), azides (0.4 mmol, 1.0 equiv), Cu salt (0.03 mmol, 0.07 equiv) dissolved in solvent (2.0 mL) was treated with base (0.4 mmol, 1.0 equiv), purged with argon, and stirred at room temperature for 12 h. [b] Isolated yields. [c] Ice-water bath. [d] no detection. [e] 7.5 mol% of $CuSO_4·5H_2O$ and 15 mol% of sodium ascorbate. [f] V : V = 1 : 1.

3. General procedure for the synthesis of 1,2,3-triazoles

3.1 General procedure for the synthesis of 4-TMS-ethynyl-1,2,3-triazole 3

To a 10 mL vial were added BTMSBD (0.5 mmol, 1.25 equiv), azide (0.4 mmol, 1.0 equiv), CuI (0.03 mmol, 0.07 equiv), DMF (2.0 mL), and pyridine (0.4 mmol, 1.0 equiv) was added. The vial was then purged with argon. The mixture was stirred at room temperature for 12 h, with TLC monitoring to confirm reaction completion. Next, it was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (*V*: *V* = 30:1 to 10:1) as the eluent to afford 4-TMS-ethynyl-1,2,3-triazole **3**.

3.2 General procedure for the synthesis of compound 4

To a 10 mL vial were added BTMSBD (0.5 mmol, 1.25 equiv), azide (0.4 mmol, 1.0 equiv), CuI (0.4 mmol, 1.0 equiv) and THF (2.0 mL). Subsequently, DIPEA (0.4 mmol, 1.0 equiv) and H_2O_2 (30%, 1.0 equiv) were added. The vial was then purged with argon. The mixture was stirred at room temperature for 48 h, with TLC monitoring to confirm reaction completion. Next, it was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (*V: V* = 8:1) as the eluent to afford compound **4**.

3.3 General procedure for the synthesis of compound 5

To a 10 mL vial were added **3a** (0.5 mmol, 1 equiv), 1-azido-2-fluorobenzene (0.5 mmol, 1 equiv), CuI (0.05 mmol, 0.1 equiv) and DMF (2.0 mL). Subsequently, Et_3N (0.2 mmol, 0.4 equiv) and H_2O (0.5 mmol, 1 equiv) were added. The vial was then

purged with argon. The mixture was stirred at room temperature for 24 h, with TLC monitoring to confirm reaction completion. Next, it was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using dichloromethane as the eluent to afford compound **5**.

3.4 General procedure for the synthesis of compound 6

To a 10 mL vial were added **3a** (0.5 mmol, 1 equiv), BnN_3 (0.5 mmol, 1 equiv), $CuSO_2 \cdot 5H_2O$ (0.05 mmol, 0.1 equiv), sodium ascorbate (0.12 mmol, 0.2 equiv), and CH_3OH/H_2O (1:1, 3 mL). The mixture was stirred at room temperature for 24 h, with TLC monitoring to confirm reaction completion. Next, it was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using dichloromethane as the eluent to afford compound **6**.

3.5 General procedure for the synthesis of compound 7

To a 10 mL vial were added **3a** (0.5 mmol, 1 equiv) and PhN_3 (0.5 mmol, 1.0 equiv). The mixture was dissolved in THF (2.0 mL). Subsequently, *t*-BuOK (0.5 mmol, 1.0 equiv) was added. The mixture was stirred at room temperature for 12 h, with TLC monitoring to confirm reaction completion. Then, the mixture was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (V:V = 2:1) as the eluent to afford compound **7**.

3.6 General procedure for the synthesis of compound 8

To a 10 mL vial were added **3a** (0.3 mmol, 1 equiv) and CuCl (0.3 mmol, 1 equiv). They were dissolved in DMF (0.5 mL). The vial was then left open to air. The mixture was stirred in an oil bath at 60 °C for 6 h, with TLC monitoring to confirm reaction completion. Subsequently, the reaction mixture was quenched with 1 M hydrochloric acid. Next, it was diluted with dichloromethane (30 mL) and washed successively with

H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using dichloromethane as the eluent to afford compound 8.

3.7 General procedure for the synthesis of compound 9

To a 10 mL vial were added compound **3a** (0.1 mmol, 1 equiv) and phenylethynyl iodide (0.1 mmol, 1 equiv). They were dissolved in THF (0.5 mL). Subsequently, tetrabutylammonium fluoride (TBAF, 1M in THF, 0.5 equiv) was added. Next, $Pd(PPh_3)_2Cl_2$ (0.03 equiv) and CuI (0.03 equiv) were added, followed by *N,N*-diisopropylethylamine (DIPEA, 2 equiv). The mixture was stirred at room temperature for 12 hours under argon atmosphere, with TLC monitoring to confirm reaction completion. After the reaction, the mixture was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (V:V = 6:1) as the eluent to afford compound **9**.

3.8 General procedure for the synthesis of compound 10

To a 10 mL vial were added compound **3a** (0.1 mmol, 1 equiv) and PhI (0.1 mmol, 1 equiv). They were dissolved in THF (0.5 mL). Subsequently, TBAF (1M in THF, 0.5 equiv) was added. Next, $Pd(PPh_3)_2Cl_2$ (0.03 equiv) and CuI (0.03 equiv) were added, followed by DIPEA (2 equiv). The mixture was stirred at room temperature for 12 hours under argon atmosphere, with TLC monitoring to confirm reaction completion. After the reaction, the mixture was diluted with EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (V:V = 6:1) as the eluent to afford compound 10.

3.9 General procedure for the synthesis of compound 11

To a 10 mL vial was added compound **3a** (0.2 mmol, 1 equiv), which was then dissolved in THF (1.0 mL). Subsequently, TBAF (1M in THF, 1.0 equiv) was added. The mixture was stirred at room temperature for 3 hours, with TLC monitoring to confirm the reaction completion. After the reaction, the mixture was diluted with

EtOAc (30 mL) and washed successively with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (V:V = 6:1) as the eluent to afford compound 11.

3.10 General procedure for the synthesis of compound 12a

To a 10 mL vial were added compound **3a** (0.25 mmol, 1.0 equiv) and acetophenone (0.5 mmol, 2.0 equiv). Subsequently, *t*-BuOK (0.25 mmol, 1.0 equiv) was added. The reaction mixture was stirred at room temperature for 50 minutes. After that, K_2CO_3 (3.0 equiv) and MeOH (1.0 mL) were added, and the mixture was stirred at room temperature until the reaction was complete, with TLC monitoring. Then, the mixture was diluted with ethyl acetate (EtOAc, 30 mL) and washed successively with water (H_2O, 10 mL × 2) and saturated brine. The organic phase was dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and concentrated. The residue was purified by silica gel column chromatography using petroleum ether/ethyl acetate (PE/EtOAc, V:V = 3:1) as the eluent to afford compound **12a**.

3.11 General procedure for the synthesis of compound 12b

To a 10 mL vial were added compound **3a** (0.25 mmol, 1.0 equiv) and acetone (1.0 mmol, 4.0 equiv). Subsequently, *t*-BuOK (0.25 mmol, 1.0 equiv) was added. The reaction mixture was stirred at room temperature for 50 minutes. After that, K_2CO_3 (3.0 equiv) and MeOH (1.0 mL) were added, and the mixture was stirred at room temperature until the reaction was complete, with TLC monitoring. Then, the mixture was diluted with ethyl acetate (EtOAc, 30 mL) and washed successively with water (H_2O, 10 mL × 2) and saturated brine. The organic phase was dried over anhydrous sodium sulfate (Na_2SO_4), filtered, and concentrated. The residue was purified by silica gel column chromatography using petroleum ether/ethyl acetate (PE/EtOAc, V:V = 2:1) as the eluent to afford compound **12b**.

3.12 General procedure for the synthesis of compound 13.

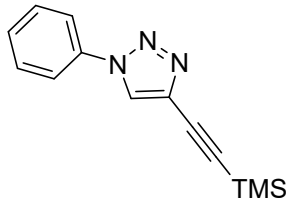
To a 5 mL vial were added **3a** (0.3 mmol, 1 equiv), pyrrolidine (0.3 mmol, 1 equiv), benzaldehyde (0.48 mmol, 1.6 equiv.) and Ag_2CO_3 (3 mol%). The mixture was stirred in an oil bath at 110 °C for 15 min. Then, the mixture was diluted with

EtOAc (30 mL) and washed with H_2O (10 mL × 2) and saturated brine. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography using PE/EtOAc (V:V = 4:1) as the eluent to afford compound **13**.

4. Characterization data of 1,2,3-triazoles

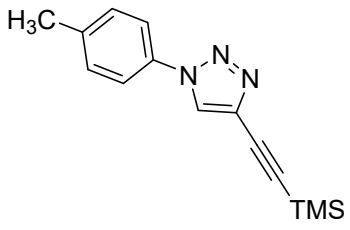
4.1 Characterization data of 4-TMS-ethynyl-1,2,3-triazole 3(3a-3o)

1-phenyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3a)



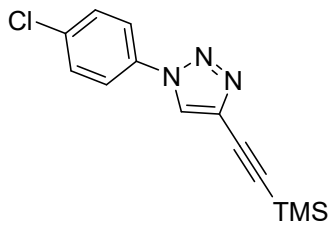
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 84 mg; mp: 132–135 °C; 70% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 (s, 1H), 7.71 (dd, J = 7.8, 2.0 Hz, 2H), 7.57 – 7.50 (m, 2H), 7.49 – 7.43 (m, 1H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 136.69, 131.89, 130.02, 129.26, 124.39, 120.74, 99.77, 93.28, -0.18. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + H]^+$ Calcd for $C_{13}H_{15}N_3Si$: 242.1113, found: 242.1103.

1-(p-tolyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3b)



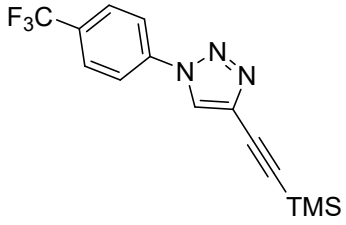
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 80 mg; mp: 115–126 °C; 63% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (s, 1H), 7.57 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.2 Hz, 2H), 2.42 (s, 3H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 139.45, 134.37, 131.70, 130.48, 124.40, 120.62, 99.59, 93.38, 21.24, -0.18. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + Na]^+$ Calcd for $C_{14}H_{17}N_3Si$: 278.1089, found: 278.1077.

1-(4-chlorophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3c)



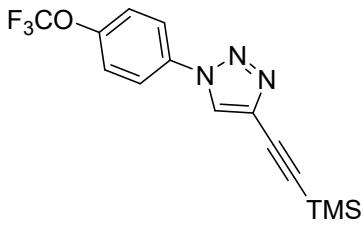
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 83 mg; mp: 142–160°C; 61% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 (s, 1H), 7.69 – 7.63 (m, 2H), 7.53 – 7.49 (m, 2H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 135.11, 132.14, 130.22, 127.17, 124.23, 121.87, 100.13, 92.97, -0.20. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{13}H_{14}ClN_3Si$: 298.0543, found: 298.0532.

1-(4-(trifluoromethyl)phenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3d)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 111 mg; mp: 115–124 °C; 72% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.15 (s, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 8.4 Hz, 2H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 139.00, 132.39, 131.18 (q, *J* = 33.1 Hz), 127.36 (q, *J* = 3.9 Hz), 124.18, 123.56 (q, *J* = 271.6 Hz), 120.63, 100.46, 92.72, -0.26. ^{19}F NMR (377 MHz, Chloroform-*d*) δ -62.68. HRMS (ESI-Q-Orbitrap) *m/z*: [M + H]$^{+}$ Calcd for $C_{14}H_{14}F_3N_3Si$: ; 310.0987, found: 310.0970.

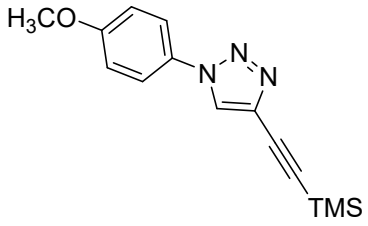
1-(4-(trifluoromethoxy)phenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3e)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid;119 mg; mp: 132–138 °C; 73% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (s, 1H), 7.80 – 7.73 (m, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 0.28 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 149.39, 135.01, 132.24, 124.31, 122.56, 122.18, 120.48 (q, *J* = 258.6 Hz), 100.24, 92.90, -0.20. ^{19}F NMR (377 MHz, Chloroform-*d*) δ -57.98. HRMS (ESI-Q-Orbitrap)

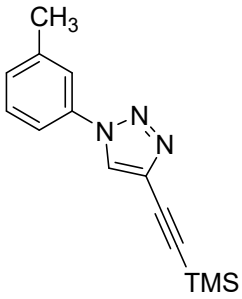
m/z: [M + Na]$^+$ Calcd for $C_{14}H_{14}F_3N_3OSi$: 348.0756, found: 348.0743.

1-(4-methoxyphenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3f)



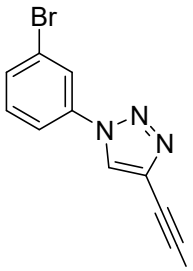
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 92 mg; mp: 125–128 °C; 68% yield; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (s, 1H), 7.60 (d, J = 8.8 Hz, 2H), 7.02 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*) δ 160.22, 131.66, 130.08, 124.57, 122.40, 115.03, 99.55, 93.41, 55.80, -0.17. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^+$ Calcd for $C_{14}H_{17}N_3OSi$: 294.1039, found: 294.1025.

1-(m-tolyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3g)



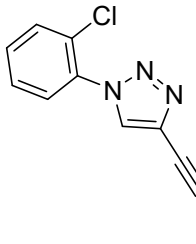
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 93 mg; mp: 99–103 °C; 65% yield; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.05 (s, 1H), 7.56 (s, 1H), 7.47 (d, J = 8.1 Hz, 1H), 7.40 (t, J = 7.8 Hz, 1H), 7.26 (d, J = 7.3 Hz, 1H), 2.44 (s, 3H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*) δ 140.34, 136.62, 131.77, 130.01, 129.78, 124.45, 121.44, 117.74, 99.69, 93.33, 21.55, -0.17. HRMS (ESI-Q-Orbitrap) *m/z*: [M + K]$^+$ Calcd for $C_{14}H_{17}N_3Si$: 294.0829, found: 294.0816.

1-(3-bromophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3h)



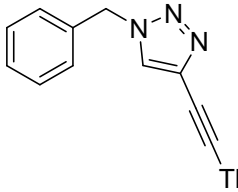
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 92 mg; mp: 141–150 °C; 58% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (s, 1H), 7.92 (t, J = 2.0 Hz, 1H), 7.66 (ddd, J = 8.1, 2.2, 1.0 Hz, 1H), 7.59 (ddd, J = 8.1, 1.9, 1.0 Hz, 1H), 7.41 (t, J = 8.1 Hz, 1H), 0.27 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 137.53, 132.27, 132.17, 131.33, 124.24, 123.83, 123.59, 119.10, 100.24, 92.88, -0.21. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{13}H_{14}BrN_3Si$: 342.0038, found: 342.0025.

1-(2-chlorophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3i)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish oil; 92 mg; 67% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (s, 1H), 7.59 – 7.51 (m, 2H), 7.48 – 7.39 (m, 2H), 0.24 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 134.28, 131.17, 130.95, 130.88, 128.54, 128.26, 128.11, 127.73, 99.55, 93.09, -0.27. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{13}H_{14}ClN_3Si$: 298.0543, found: 298.0530.

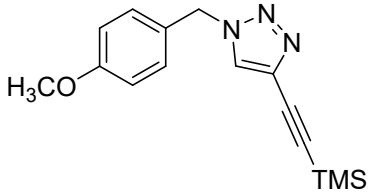
1-benzyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3j)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 8:1) as yellowish oil; 71mg; 56% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (s, 1H), 7.25 (dd, J = 5.1, 1.9 Hz, 3H), 7.15 – 7.11 (m, 2H), 5.39 (s, 2H), 0.10 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 134.18, 131.50,

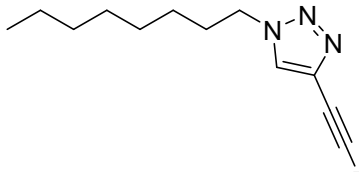
129.34, 129.10, 128.27, 126.32, 98.94, 93.62, 54.43, -0.21. HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{14}H_{17}N_3Si$ found 256.1254, calculated 256.1265.[1]

1-(4-methoxybenzyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3k)



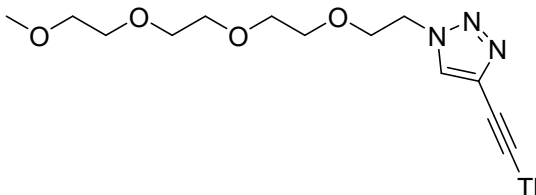
Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 6:1) as yellowish solid; 94 mg; mp: 94–96 °C; 66% yield; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.49 (s, 1H), 7.22 – 7.19 (m, 2H), 6.90 – 6.87 (m, 2H), 5.44 (s, 2H), 3.80 (s, 3H), 0.22 (s, 9H). ^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*) δ 160.22, 131.39, 129.91, 126.14, 126.09, 114.70, 98.85, 93.68, 55.50, 54.01, -0.21. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + Na]^+$ Calcd for $C_{15}H_{19}N_3OSi$: 308.1195, found: 308.1181.

1-octyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3l)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish oil; 66 mg; 48% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 (s, 1H), 4.29 (t, J = 7.1 Hz, 2H), 1.83 (p, J = 6.6, 5.9 Hz, 2H), 1.28 – 1.16 (m, 10H), 0.82 (t, J = 6.8 Hz, 3H), 0.20 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 130.86, 126.18, 98.53, 93.77, 50.53, 31.69, 30.19, 29.03, 28.92, 26.36, 22.60, 14.07, 0.27. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + Na]^+$ Calcd for $C_{15}H_{27}N_3Si$: 300.1872, found: 300.1858.

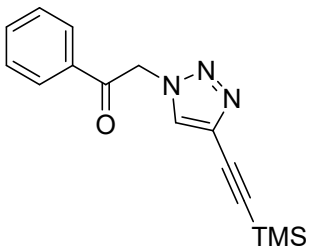
1-(2,5,8,11-tetraoxatridecan-13-yl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3m)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 6:1) as brown oil; 85 mg, 48%

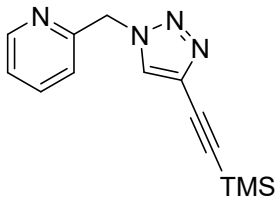
yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (s, 1H), 4.54 – 4.50 (m, 2H), 3.85 – 3.80 (m, 2H), 3.64 – 3.58 (m, 10H), 3.53 – 3.50 (m, 2H), 3.34 (s, 3H), 0.23 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 130.89, 127.79, 98.41, 94.01, 71.98, 70.68, 70.66, 70.62, 70.59, 70.48, 69.31, 59.09, 50.51, -0.18. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^+$ Calcd for $C_{16}H_{29}N_3O_4Si$: 378.1825, found: 378.1810.

1-phenyl-2-(4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazol-1-yl)ethan-1-one (3n)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 15:1) as yellowish solid; 58 mg; mp: 123–129 °C; 41% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.97 (m, 2H), 7.82 (s, 1H), 7.70 – 7.65 (m, 1H), 7.55 (t, *J* = 7.8 Hz, 2H), 5.84 (s, 2H), 0.26 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 189.81, 134.86, 133.90, 131.56, 129.35, 128.32, 128.29, 99.07, 93.58, 55.56, -0.18. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^+$ Calcd for $C_{15}H_{17}N_3OSi$: 306.1039, found: 306.1025.

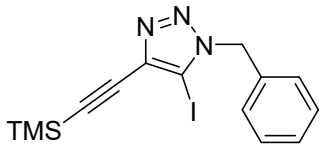
2-((4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazol-1-yl)methyl)pyridine (3o)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 2:1) as brown oil; 83 mg; 65% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.59 (d, *J* = 4.9 Hz, 1H), 7.84 (s, 1H), 7.69 (td, *J* = 7.7, 1.8 Hz, 1H), 7.29 – 7.25 (m, 1H), 7.20 (dd, *J* = 7.8, 1.1 Hz, 1H), 5.64 (s, 2H), 0.24 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 153.96, 149.94, 137.50, 131.46, 127.02, 123.67, 122.58, 98.93, 93.60, 55.76, -0.24. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^+$ Calcd for $C_{13}H_{16}N_4Si$: 279.1042, found: 279.1031.

4.2 Characterization data of compound 4

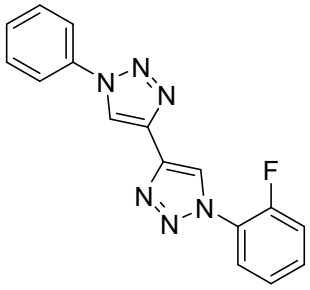
1-benzyl-5-iodo-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (4)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 8:1) as yellowish solid; 67 mg; mp: 124–130 °C; 35% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.31 (m, 3H), 7.24 (dd, *J* = 7.3, 2.4 Hz, 2H), 5.57 (s, 2H), 0.26 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 138.67, 133.93, 129.07, 128.79, 127.92, 101.73, 93.32, 84.76, 54.80, -0.18. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{14}H_{16}IN_3Si$: 404.0056, found: 404.0038.

4.3 Characterization data of compound 5

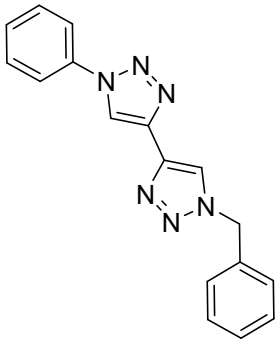
1-(2-fluorophenyl)-1'-phenyl-1*H*,1'*H*-4,4'-bi(1,2,3-triazole) (5)



Purified by flash chromatography on silica gel (eluted with dichloromethane) as yellowish solid; 77 mg; mp: 183–190 °C; 84% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.35 (s, 1H), 9.07 (d, *J* = 1.9 Hz, 1H), 8.05 – 8.00 (m, 2H), 7.93 (td, *J* = 7.8, 1.7 Hz, 1H), 7.69 – 7.58 (m, 4H), 7.55 – 7.45 (m, 2H). ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) δ 154.03 (d, *J* = 250.9 Hz), 139.49, 139.22, 136.51, 131.56 (d, *J* = 7.8 Hz), 129.96, 128.90, 126.17, 125.61 (d, *J* = 3.8 Hz), 124.65 (d, *J* = 11.0 Hz), 123.49 (d, *J* = 4.2 Hz), 120.33, 120.20, 117.22 (d, *J* = 19.4 Hz). ^{19}F NMR (377 MHz, DMSO-d_6) δ -123.77. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{16}H_{11}FN_6$: 329.0927, found: 329.0912.

4.4 Characterization data of compound 6

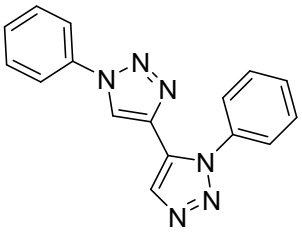
1-benzyl-1'-phenyl-1*H*,1'*H*-4,4'-bi(1,2,3-triazole) (6)



Purified by flash chromatography on silica gel (eluted with dichloromethane) as yellowish solid; 115 mg; mp: 223–226 °C; 76% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.23 (s, 1H), 8.66 (s, 1H), 8.02 – 7.96 (m, 2H), 7.61 (dd, J = 8.6, 7.1 Hz, 2H), 7.53 – 7.48 (m, 1H), 7.43 – 7.32 (m, 5H), 5.70 (s, 2H). ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) δ 140.10, 138.88, 136.53, 135.96, 129.95, 128.86, 128.83, 128.27, 128.05, 122.28, 120.13, 119.78, 53.05. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{17}H_{14}N_6$: 325.1178, found: 325.1162.

4.5 Characterization data of compound 7

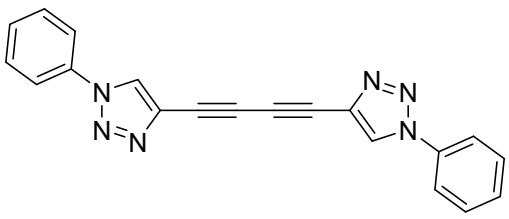
1,3'-diphenyl-1*H*,3'*H*-4,4'-bi(1,2,3-triazole) (7)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 2:1) as yellowish solid; 73 mg; mp: 61–65 °C; 85% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.97 (d, J = 1.7 Hz, 1H), 8.29 (d, J = 1.8 Hz, 1H), 7.86 (dd, J = 7.8, 1.9 Hz, 2H), 7.68 – 7.47 (m, 8H). ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) δ 136.19, 136.09, 134.84, 133.70, 130.00, 129.86, 129.45, 129.21, 128.86, 125.66, 122.91, 120.31. HRMS (ESI-Q-Orbitrap) *m/z*: [M + Na]$^{+}$ Calcd for $C_{16}H_{12}N_6$: 311.1021, found: 311.1008.

4.6 Characterization data of compound 8

1,4-bis(1-phenyl-1*H*-1,2,3-triazol-4-yl)buta-1,3-diyne (8)



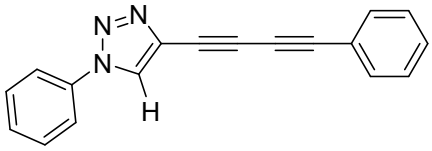
Purified by flash chromatography on silica gel

(eluted with dichloromethane) as yellowish solid; 34 mg; mp: 251–255 °C; 67% yield;

^{1}H NMR (400 MHz, DMSO-d_6) δ 9.41 (s, 2H), 7.95 – 7.90 (m, 4H), 7.67 – 7.61 (m, 4H), 7.58 – 7.53 (m, 2H). ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) δ 135.98, 130.04, 129.42, 128.63, 124.66, 120.58, 76.12, 72.61. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + H]^+$ Calcd for $C_{20}H_{12}N_6$: 359.1021, found: 359.1011.

4.7 Characterization data of compound 9

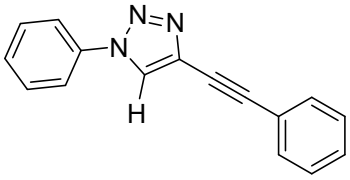
1-phenyl-4-(phenylbuta-1,3-diyn-1-yl)-1*H*-1,2,3-triazole (9)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 6:1) as yellowish solid; 13 mg; mp: 165–170 °C; 49% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.15 (s, 1H), 7.75 – 7.70 (m, 2H), 7.55 (dtt, *J* = 8.8, 3.7, 1.8 Hz, 4H), 7.51 – 7.45 (m, 1H), 7.42 – 7.32 (m, 3H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 136.53, 132.79, 130.99, 130.08, 129.74, 129.45, 128.64, 125.49, 121.35, 120.81, 83.00, 78.19, 73.40, 70.10. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + H]^+$ Calcd for $C_{18}H_{11}N_3$: 270.1031, found: 270.1019.

4.8 Characterization data of compound 10

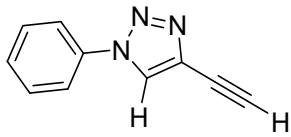
1-phenyl-4-(phenylethynyl)-1*H*-1,2,3-triazole (10)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 6:1) as tan solid; 22 mg; mp: 117–126 °C; 90% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.77 – 7.72 (m, 2H), 7.60 – 7.51 (m, 4H), 7.49 – 7.44 (m, 1H), 7.40 – 7.33 (m, 3H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 136.69, 131.97, 131.81, 130.01, 129.24, 129.05, 128.55, 123.96, 122.31, 120.70, 93.34, 78.29. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}N_3$: 246.1031, found: 246.1018.

4.9 Characterization data of compound 11

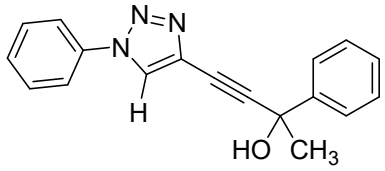
4-ethynyl-1-phenyl-1*H*-1,2,3-triazole (11)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 6:1) as yellowish solid; 30 mg; mp: 89–92 °C; 88% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.11 (s, 1H), 7.72 (dd, *J* = 7.5, 1.8 Hz, 2H), 7.54 (dd, *J* = 8.7, 6.9 Hz, 2H), 7.50 – 7.43 (m, 1H), 3.31 (s, 1H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 136.60, 130.90, 130.04, 129.36, 124.84, 120.79, 81.91, 72.87. HRMS (ESI-Q-Orbitrap) *m/z*: [M + H]$^{+}$ Calcd for $C_{10}H_7N_3$: 170.0718, found: 170.0706.

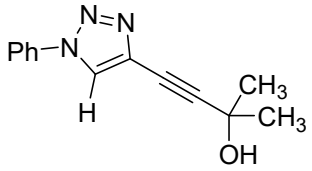
4.10 Characterization data of compound 12

2-phenyl-4-(1-phenyl-1*H*-1,2,3-triazol-4-yl)but-3-yn-2-ol (12a)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 3:1) as yellowish oil; 64 mg; 88% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.08 (s, 1H), 7.72 (ddt, *J* = 9.9, 8.1, 1.4 Hz, 4H), 7.53 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.49 – 7.43 (m, 1H), 7.38 (dd, *J* = 8.3, 6.7 Hz, 2H), 7.34 – 7.28 (m, 1H), 1.90 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 145.06, 136.60, 131.27, 130.01, 129.30, 128.53, 127.98, 125.09, 124.33, 120.72, 96.90, 74.05, 70.44, 33.15. HRMS (ESI-Q-Orbitrap) *m/z*: [M + H]$^{+}$ Calcd for $C_{18}H_{15}N_3O$: 290.1293, found: 290.1281.

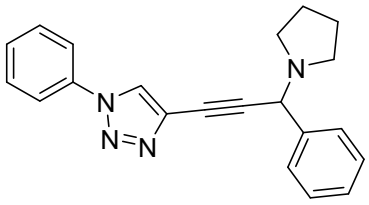
2-methyl-4-(1-phenyl-1*H*-1,2,3-triazol-4-yl)but-3-yn-2-ol (12b)



Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 2:1) as yellowish oil; 46 mg; 81% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 (s, 1H), 7.72 – 7.67 (m, 2H), 7.55 – 7.49 (m, 2H), 7.48 – 7.42 (m, 1H), 1.64 (s, 6H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 136.63, 131.43, 130.00, 129.27, 124.06, 120.70, 98.20, 71.39, 65.65, 31.29. HRMS (ESI-Q-Orbitrap) *m/z*: [M + H]$^{+}$ Calcd for $C_{13}H_{13}N_3O$: 228.1137, found: 228.1123.

4.12 Characterization data of compound 13

1-phenyl-4-(3-phenyl-3-(pyrrolidin-1-yl)prop-1-yn-1-yl)-1H-1,2,3-triazole (13)

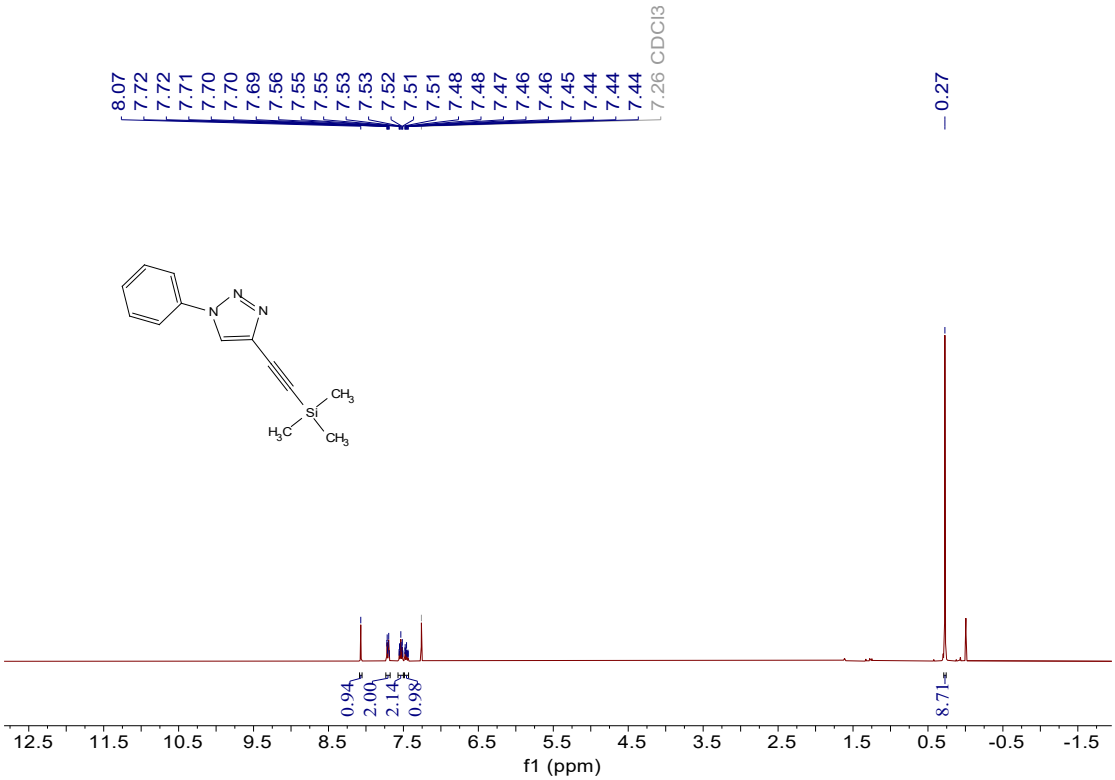


Purified by flash chromatography on silica gel (eluted with petroleum ether/ethyl acetate = 4:1) as tan oil; 138 mg; 87% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.09 (s, 1H), 7.72 (dd, *J* = 7.7, 2.0 Hz, 2H), 7.62 (dd, *J* = 6.9, 1.6 Hz, 2H), 7.56 – 7.51 (m, 2H), 7.49 – 7.44 (m, 1H), 7.39 – 7.34 (m, 2H), 7.33 – 7.28 (m, 1H), 5.01 (s, 1H), 2.76 (dt, *J* = 6.7, 4.5 Hz, 4H), 1.82 (td, *J* = 5.8, 3.2 Hz, 4H). ^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*) δ 138.42, 136.72, 131.59, 130.01, 129.25, 128.53, 128.47, 128.04, 124.24, 120.77, 90.89, 76.29, 59.22, 50.45, 23.57. HRMS (ESI-Q-Orbitrap) *m/z*: $[M + H]^+$ Calcd for $C_{21}H_{20}N_4$: 329.1766, found: 329.1757.

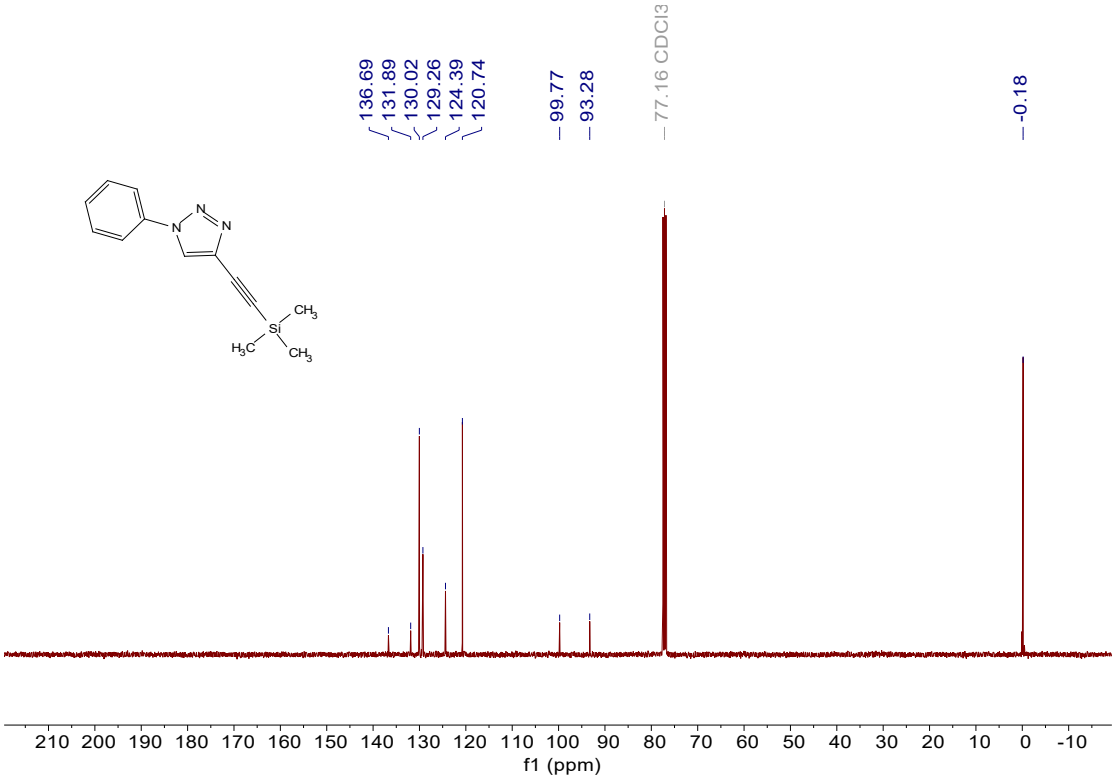
5. Copies of NMR spectra

5.1 Copies of NMR spectra of 4-TMS-ethynyl-1,2,3-triazole 3(3a-3o)

1-phenyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3a)

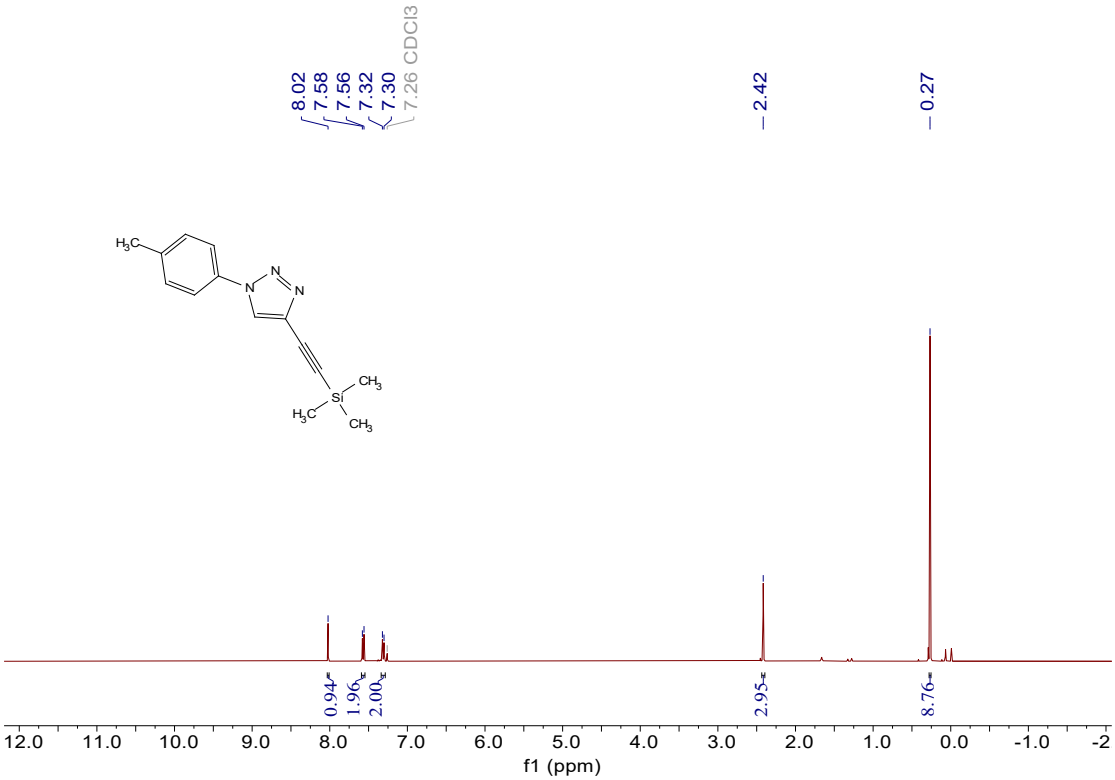


^{1}H NMR (400 MHz, Chloroform-*d*)

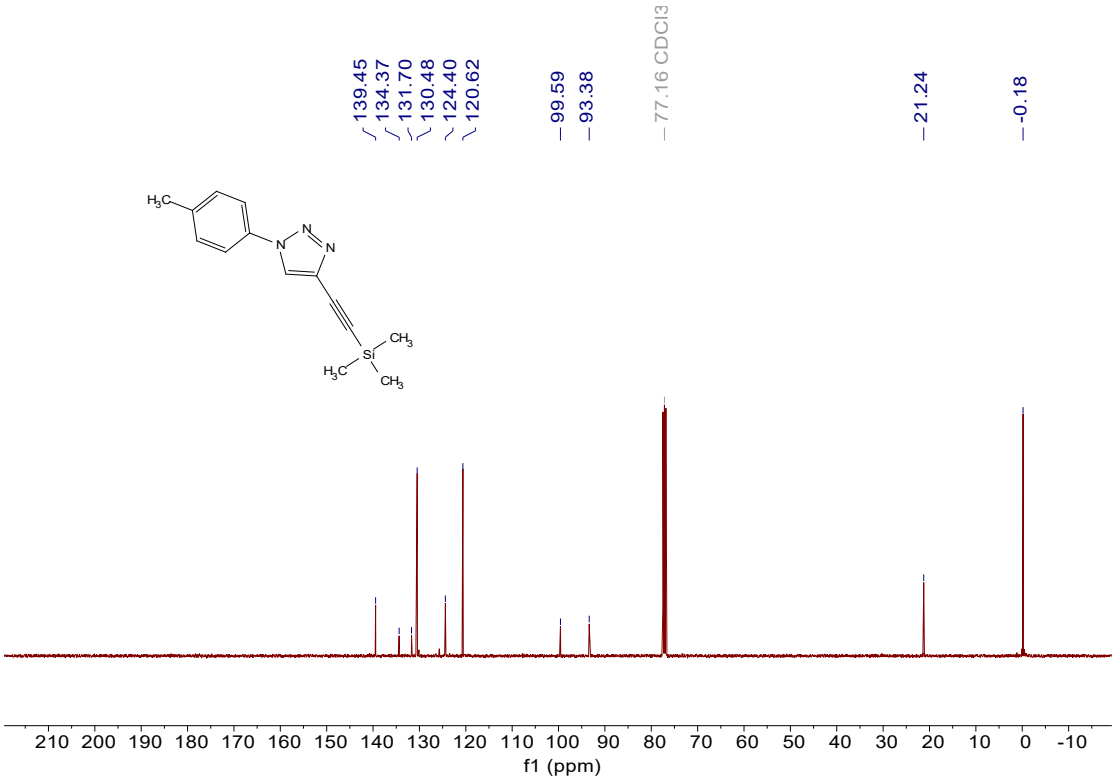


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-(p-tolyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3b)

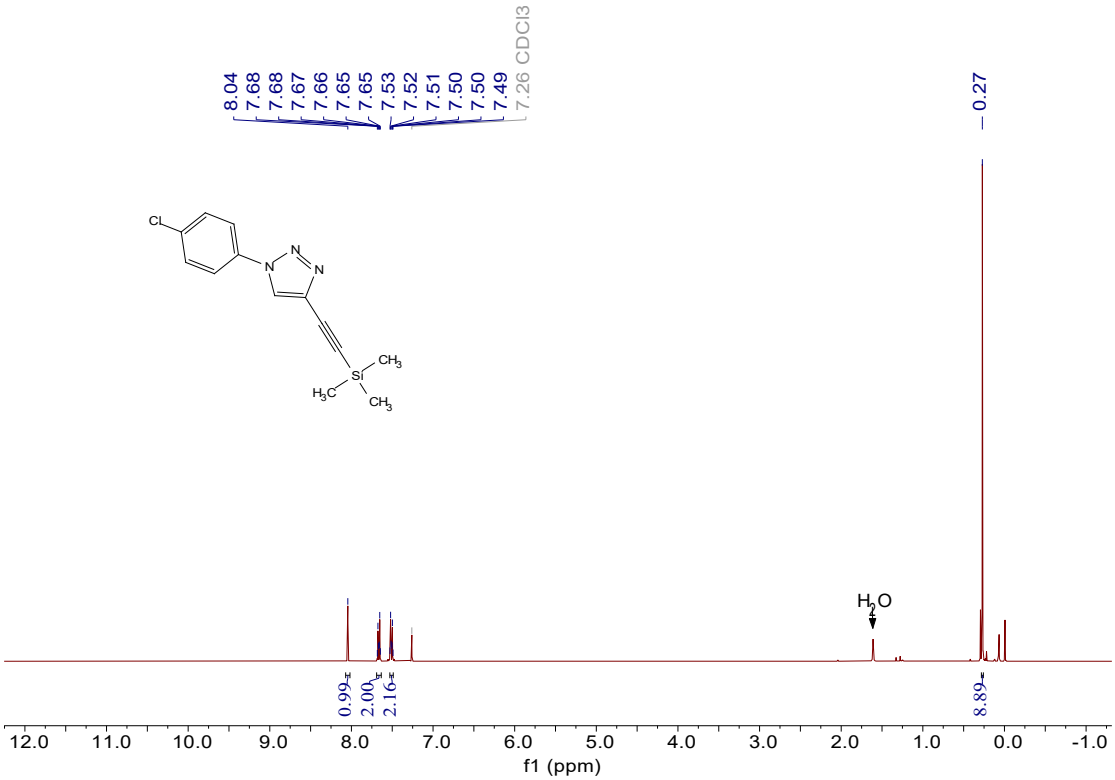


^{1}H NMR (400 MHz, Chloroform-*d*)

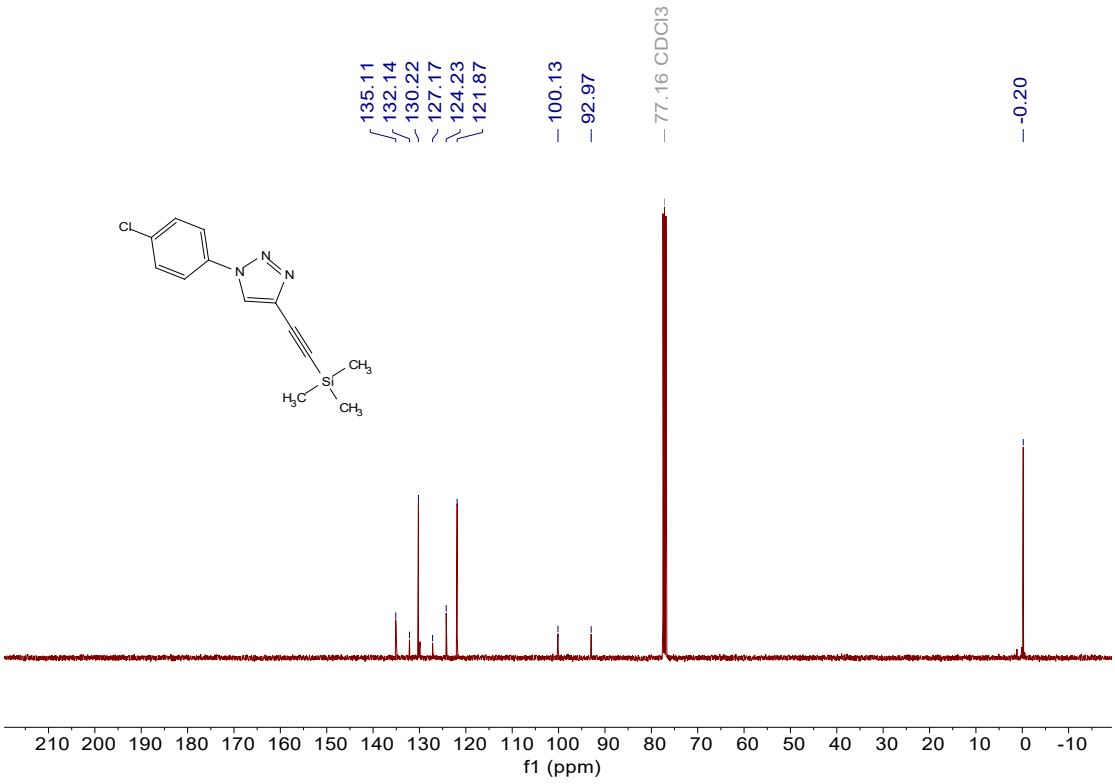


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-(4-chlorophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole(3c)

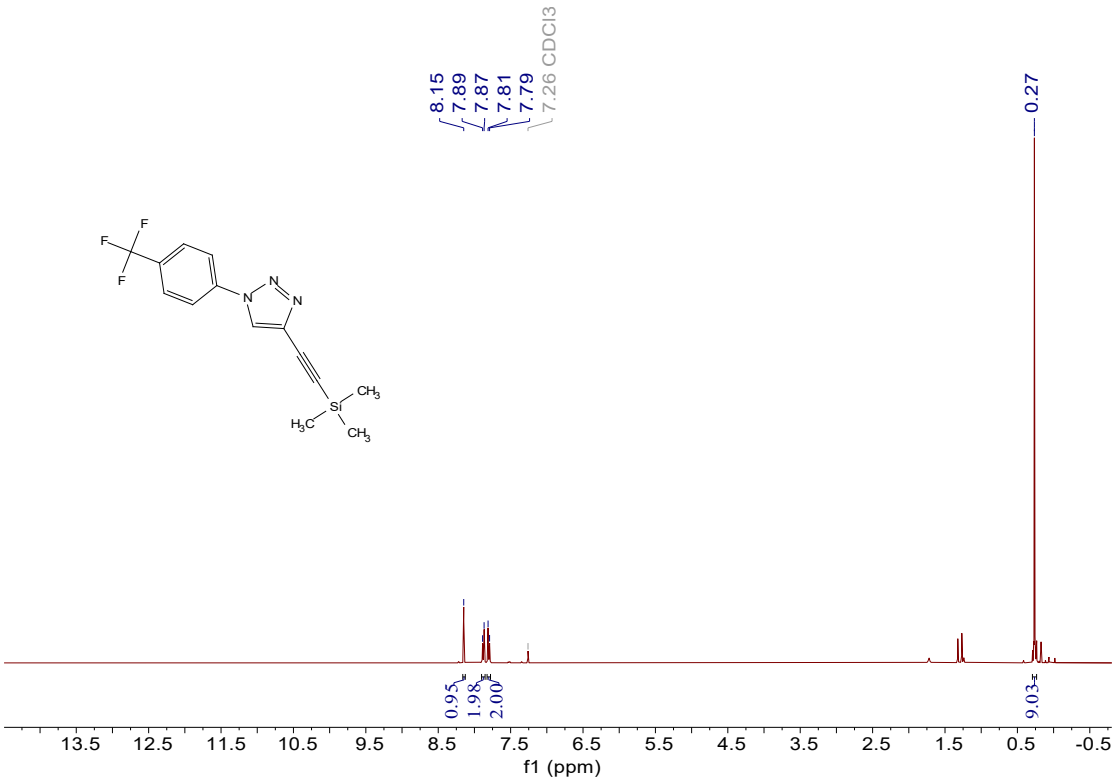


^{1}H NMR (400 MHz, Chloroform-*d*)

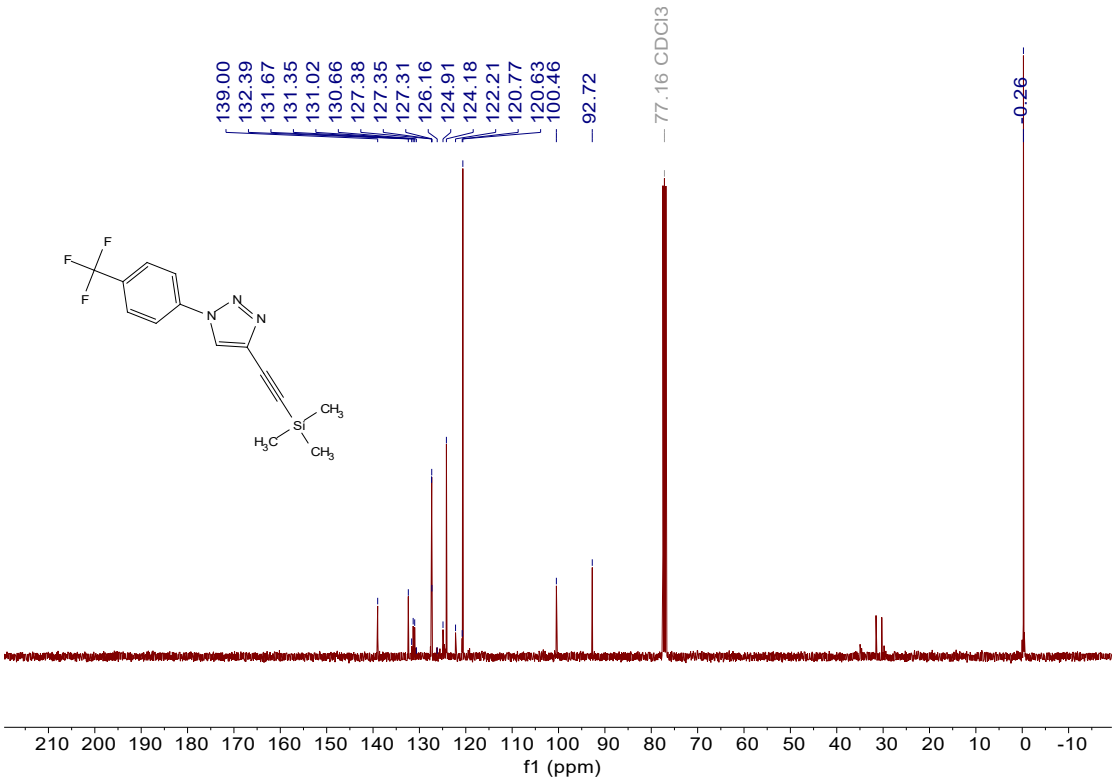


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

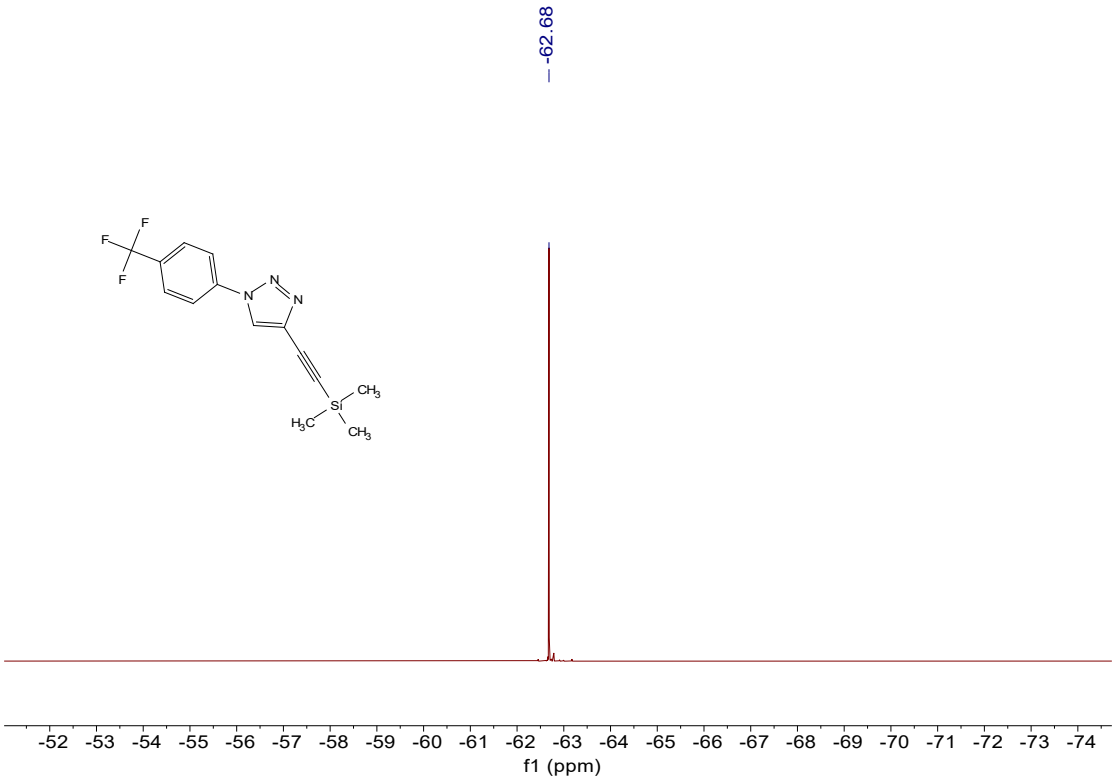
1-(4-(trifluoromethyl)phenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3d)



^{1}H NMR (400 MHz, Chloroform-*d*)

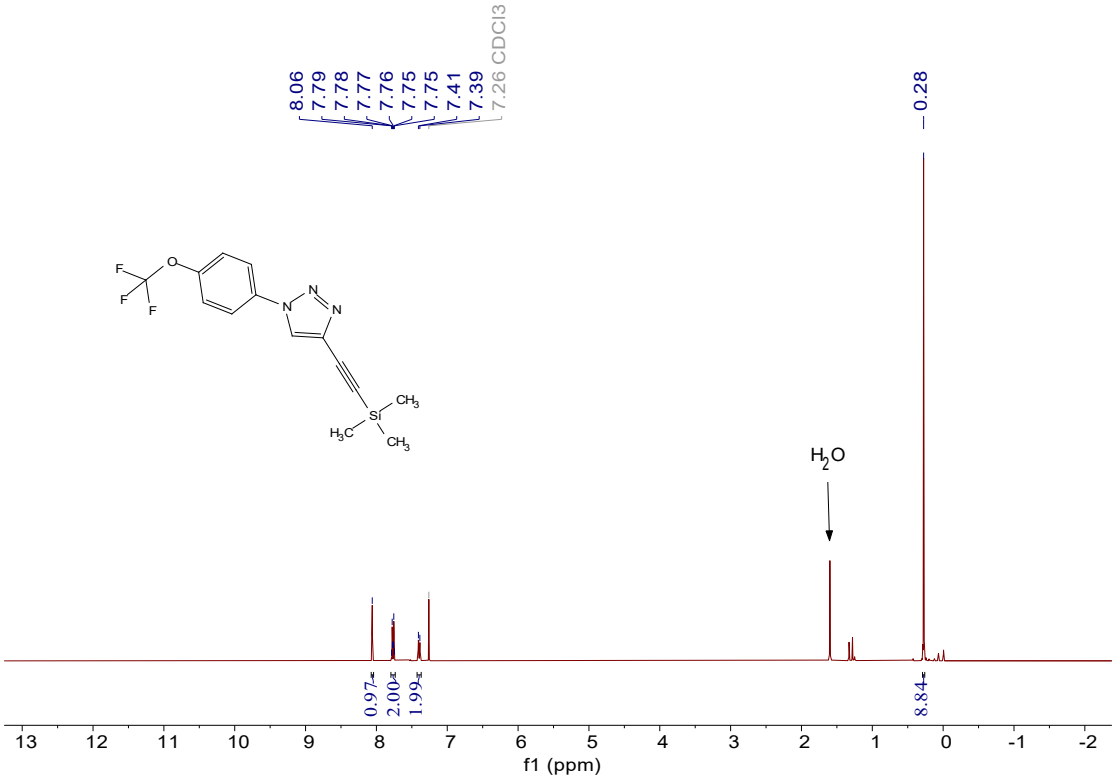


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

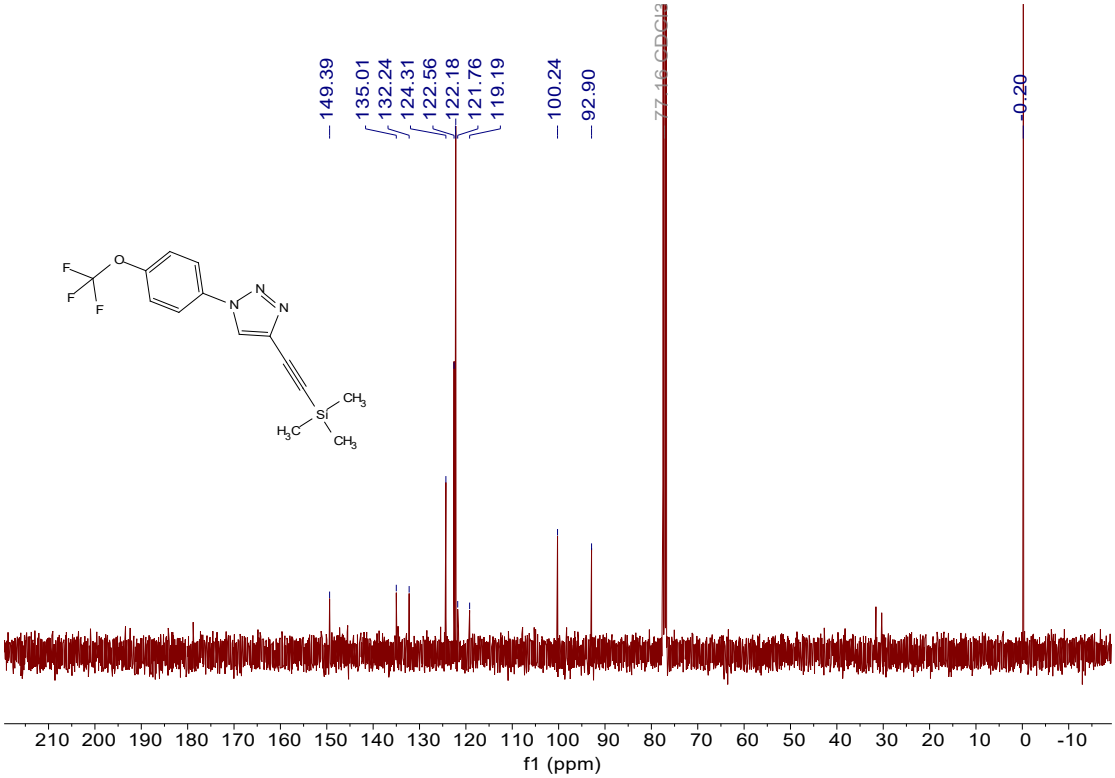


^{19}F NMR (377 MHz, Chloroform-*d*)

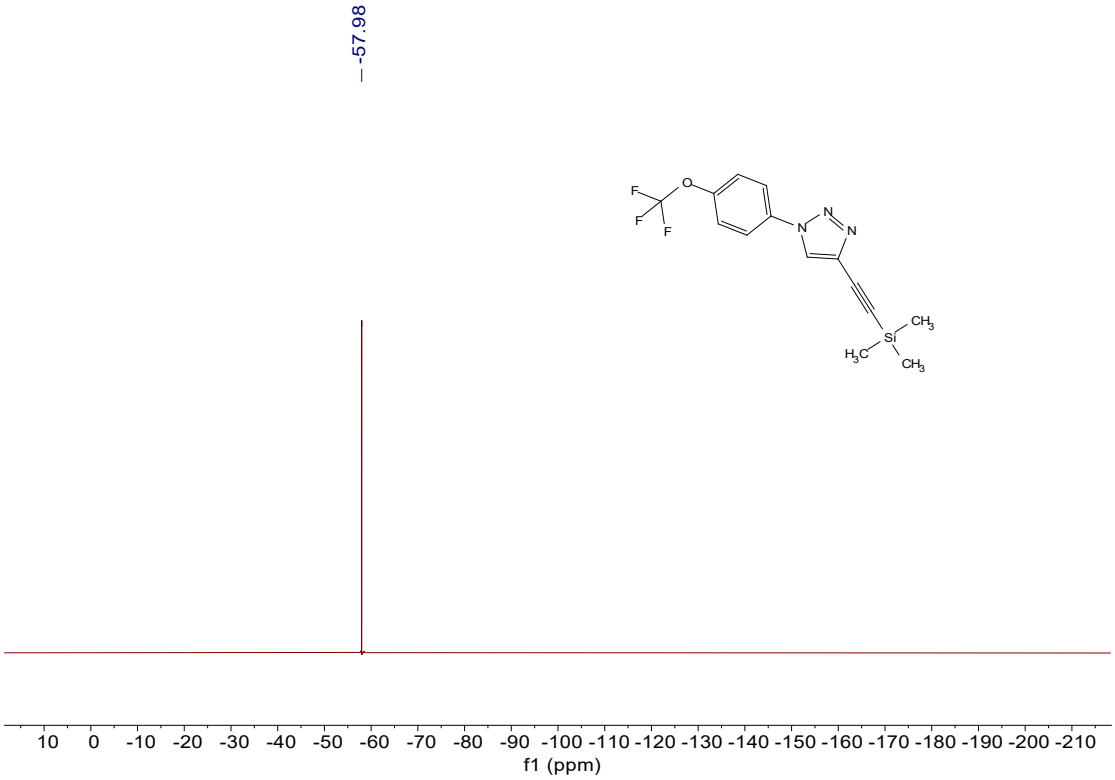
1-(4-(trifluoromethoxy)phenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3e)



^{1}H NMR (400 MHz, Chloroform-*d*)

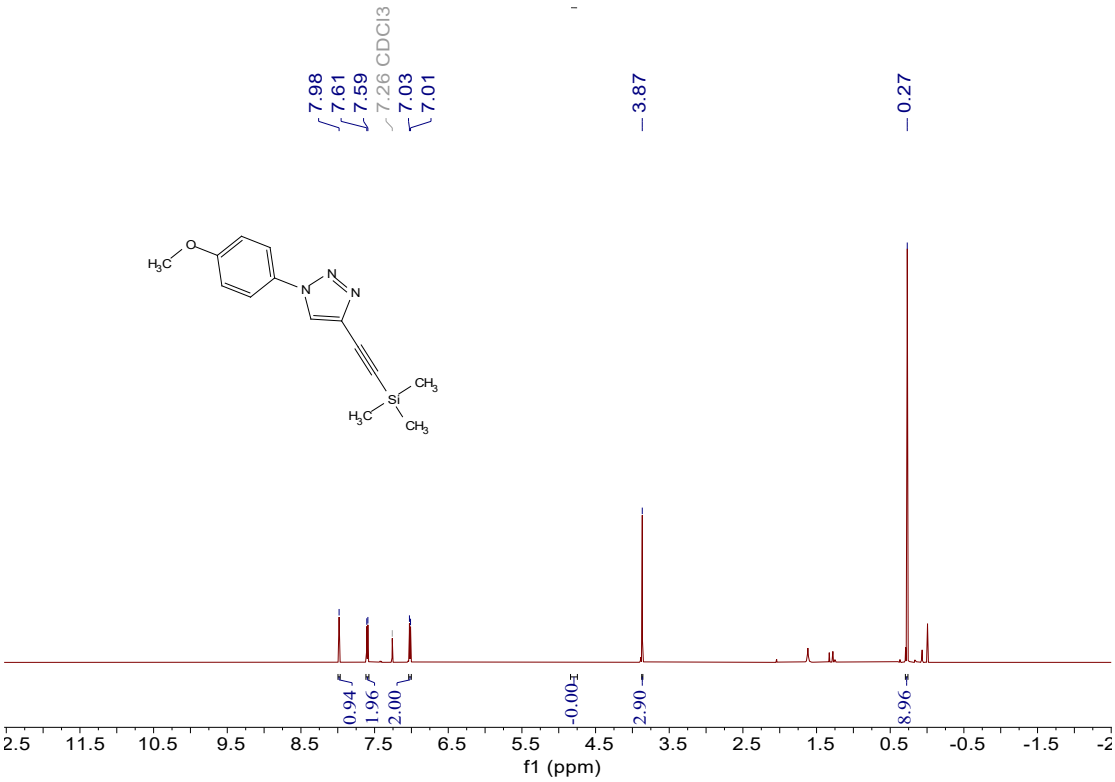


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

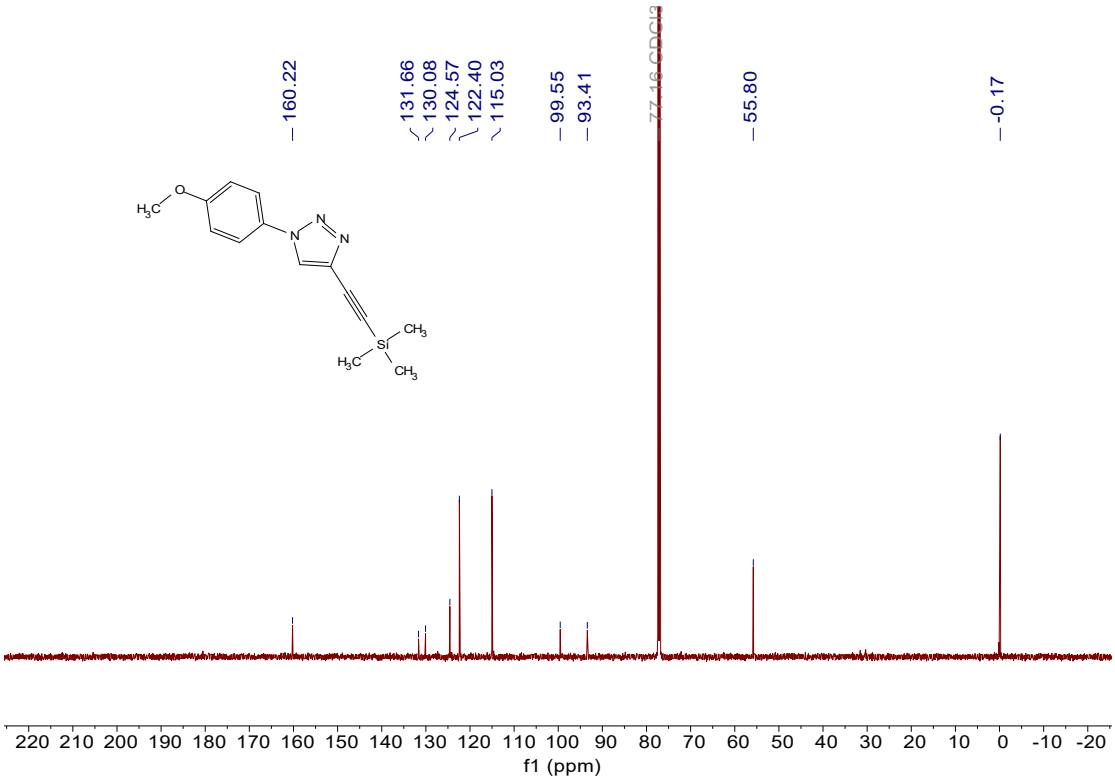


^{19}F NMR (377 MHz, Chloroform-*d*)

1-(4-methoxyphenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3f)

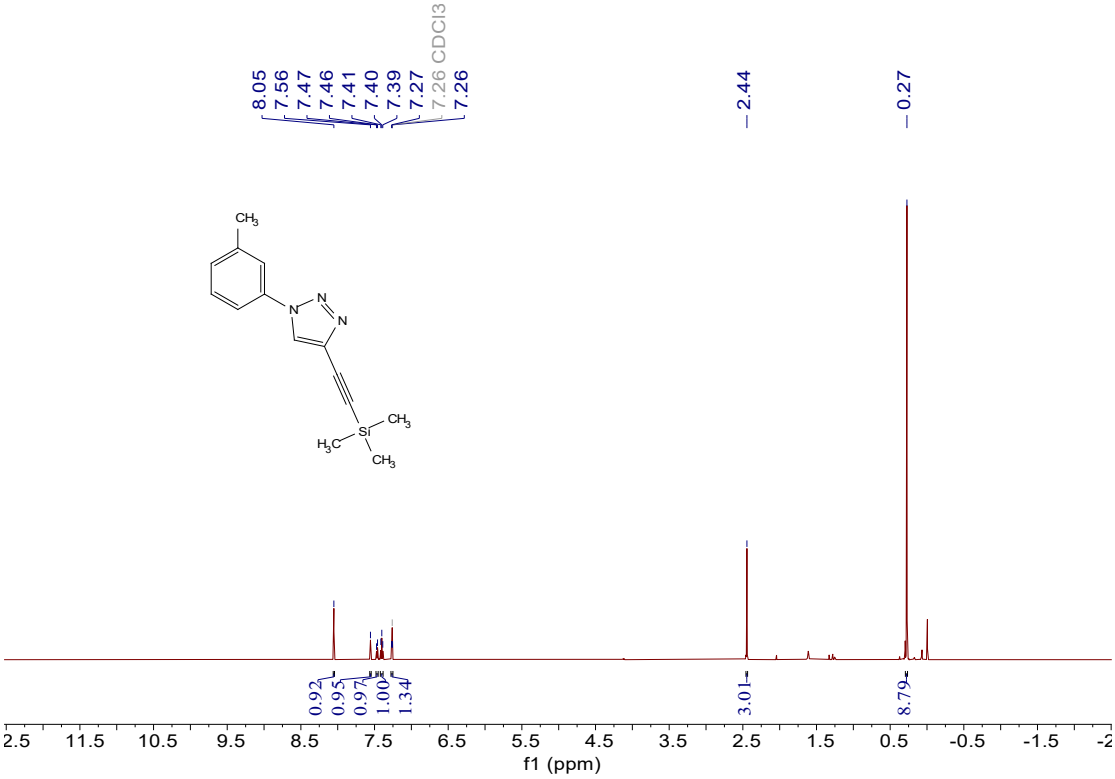


^{1}H NMR (600 MHz, Chloroform-*d*)

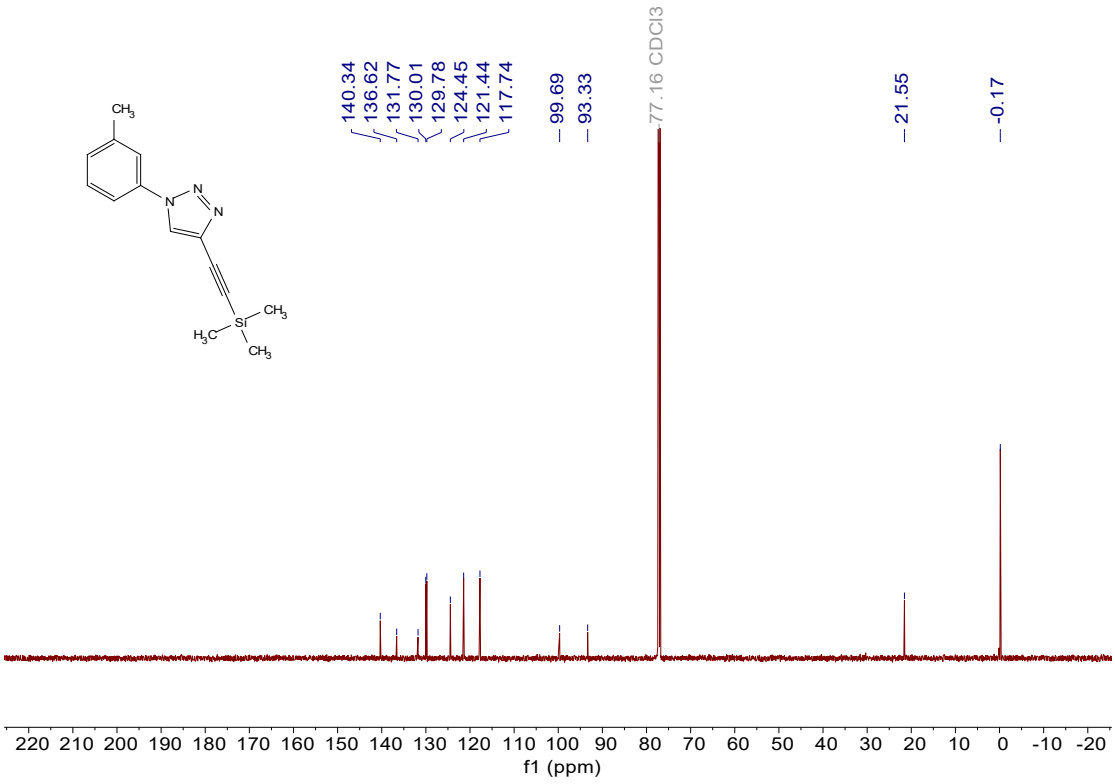


^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*)

1-(m-tolyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3g)

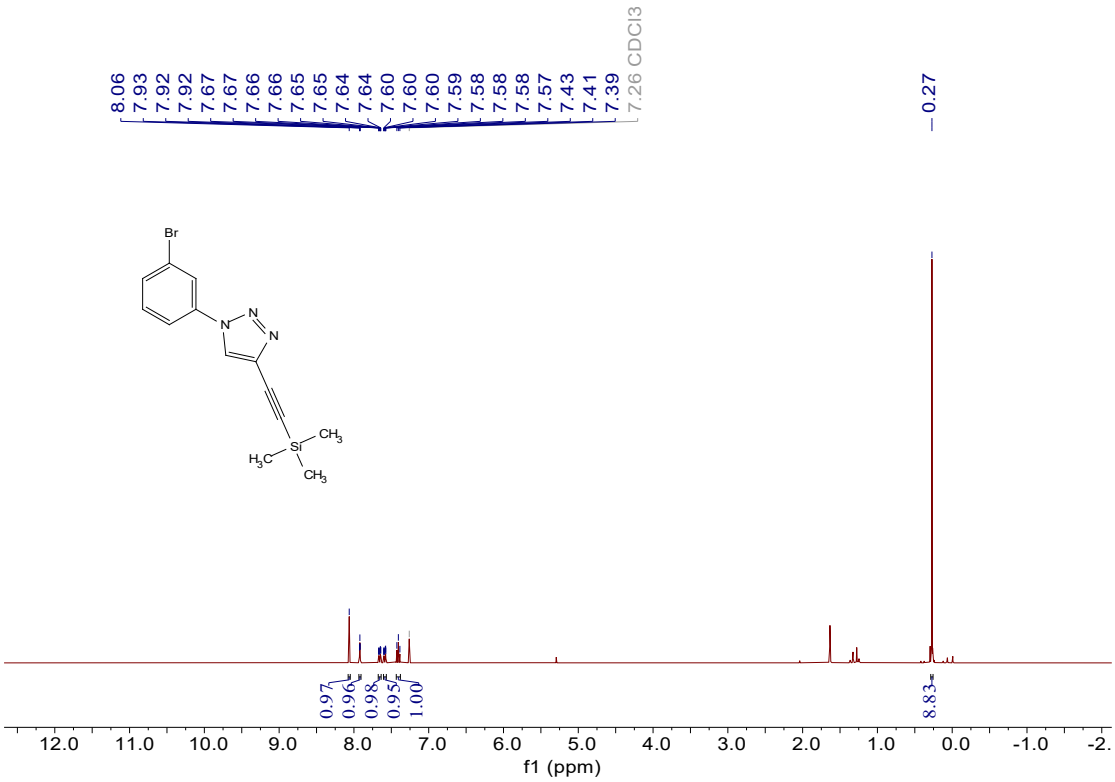


^{1}H NMR (600 MHz, Chloroform-*d*)

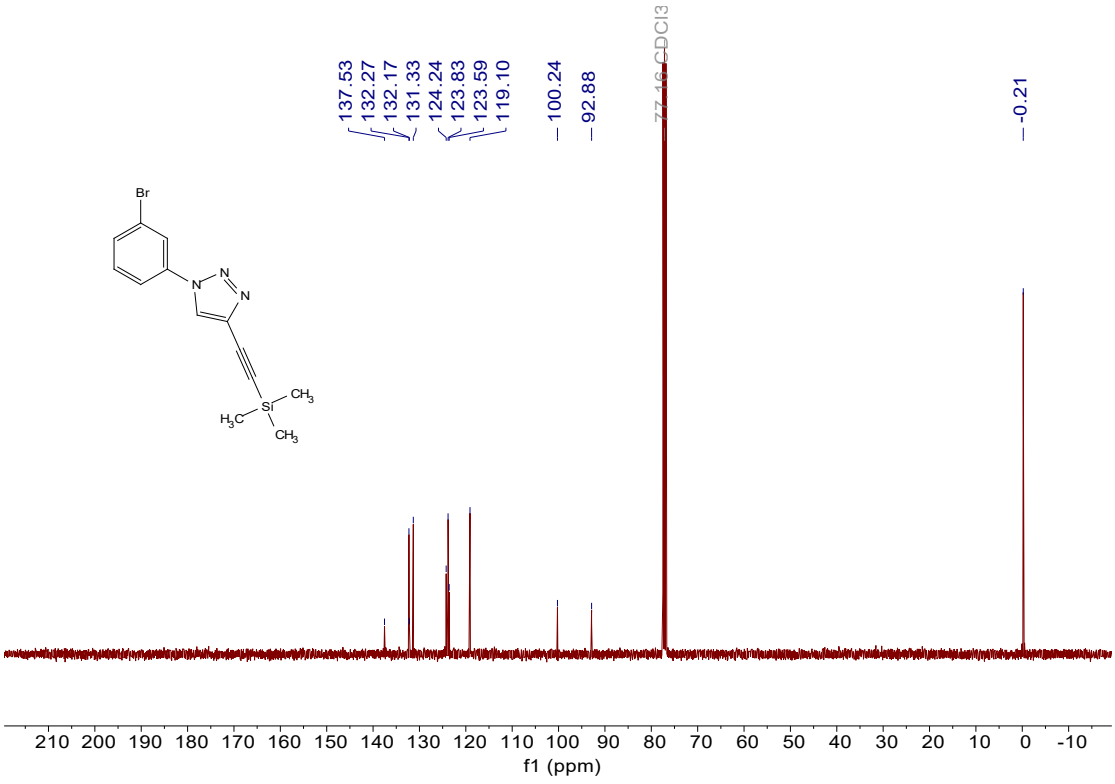


^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*)

1-(3-bromophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3h)

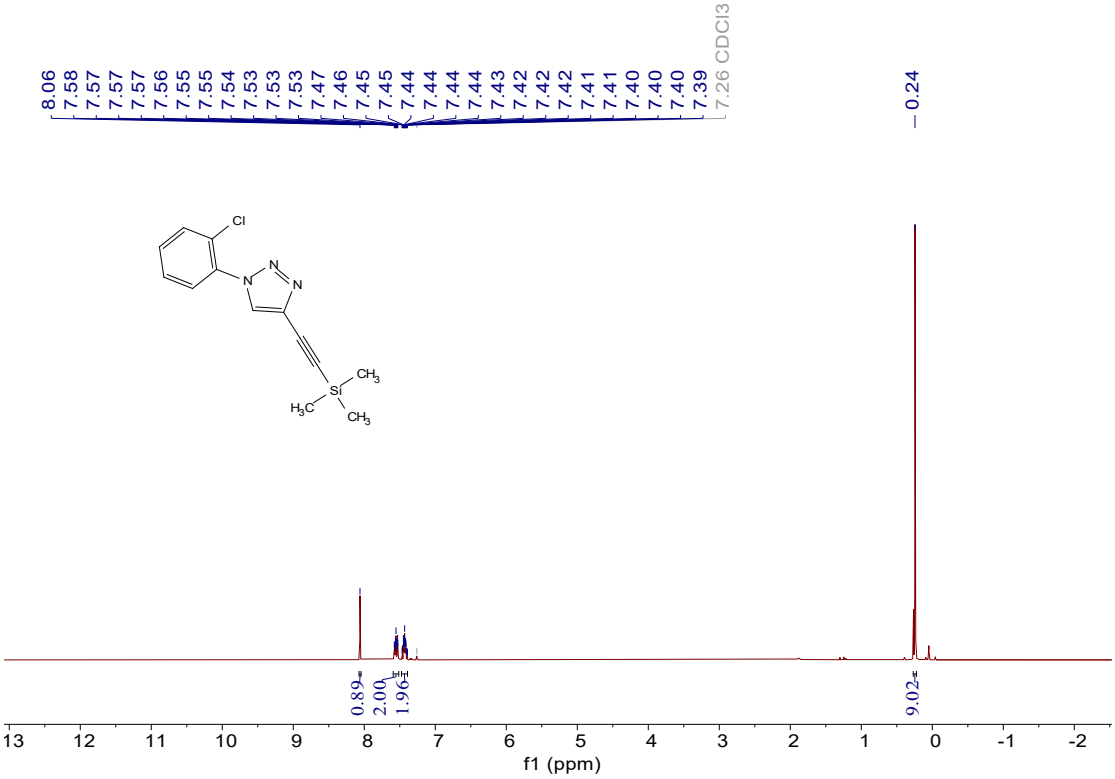


^{1}H NMR (400 MHz, Chloroform-*d*)

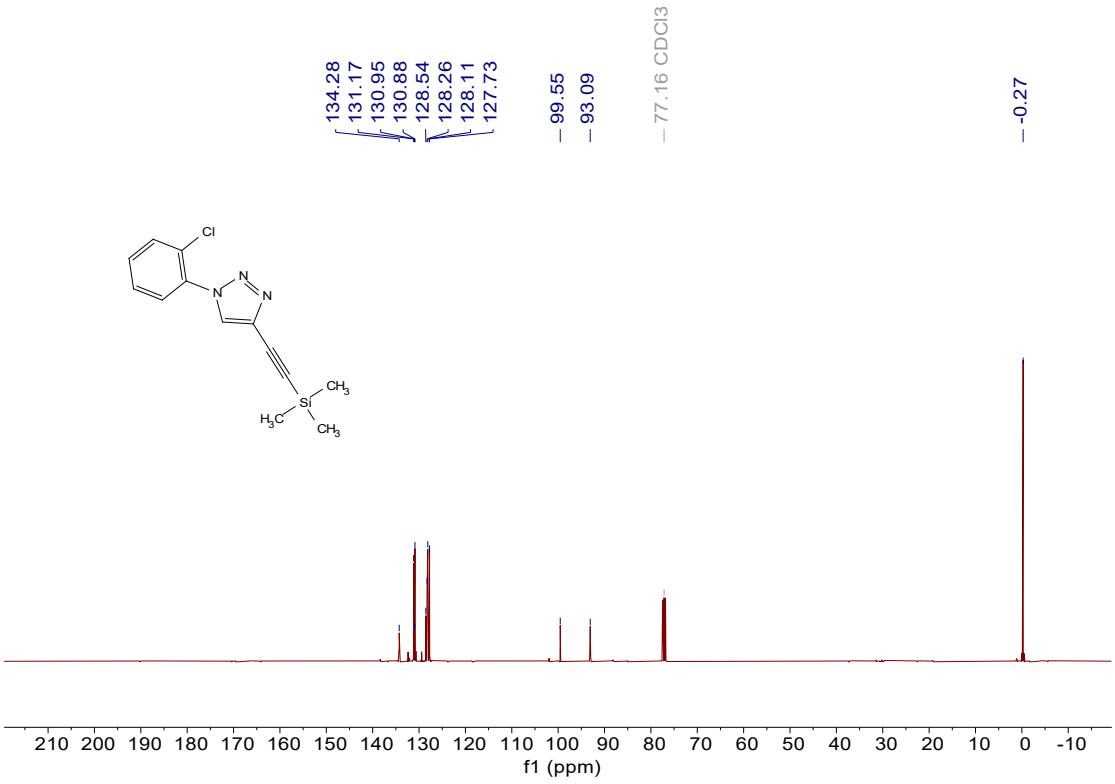


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-(2-chlorophenyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3i)

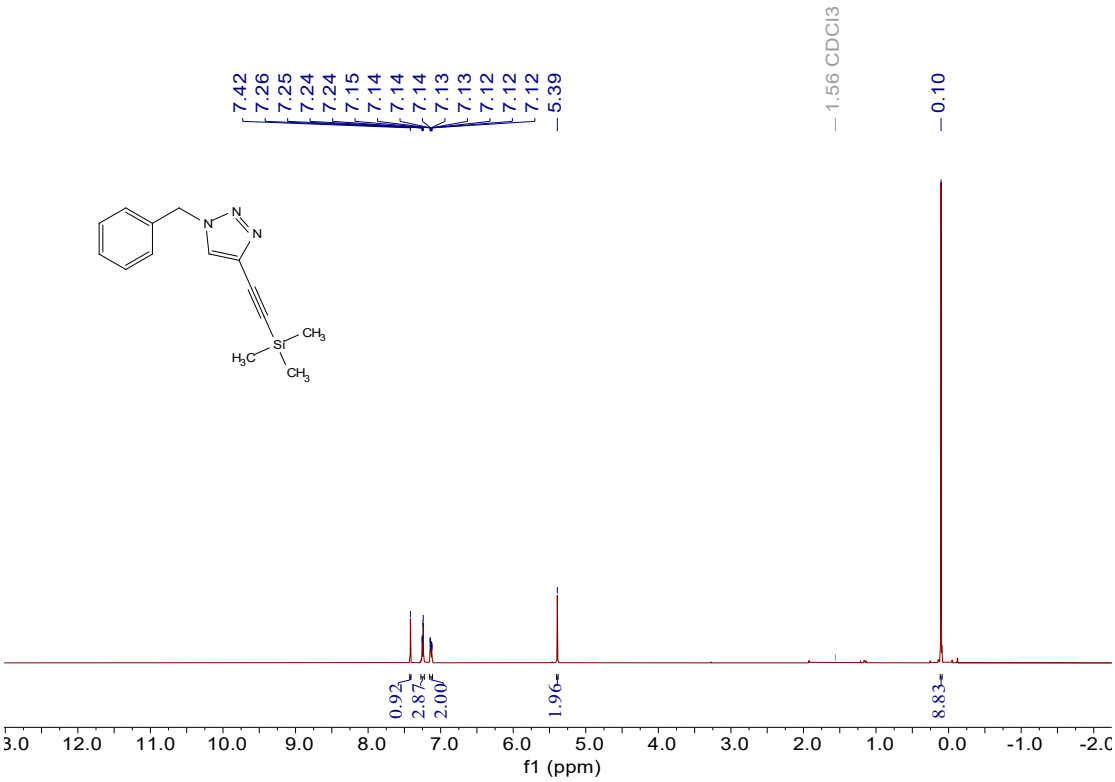


^{1}H NMR (400 MHz, Chloroform-*d*)

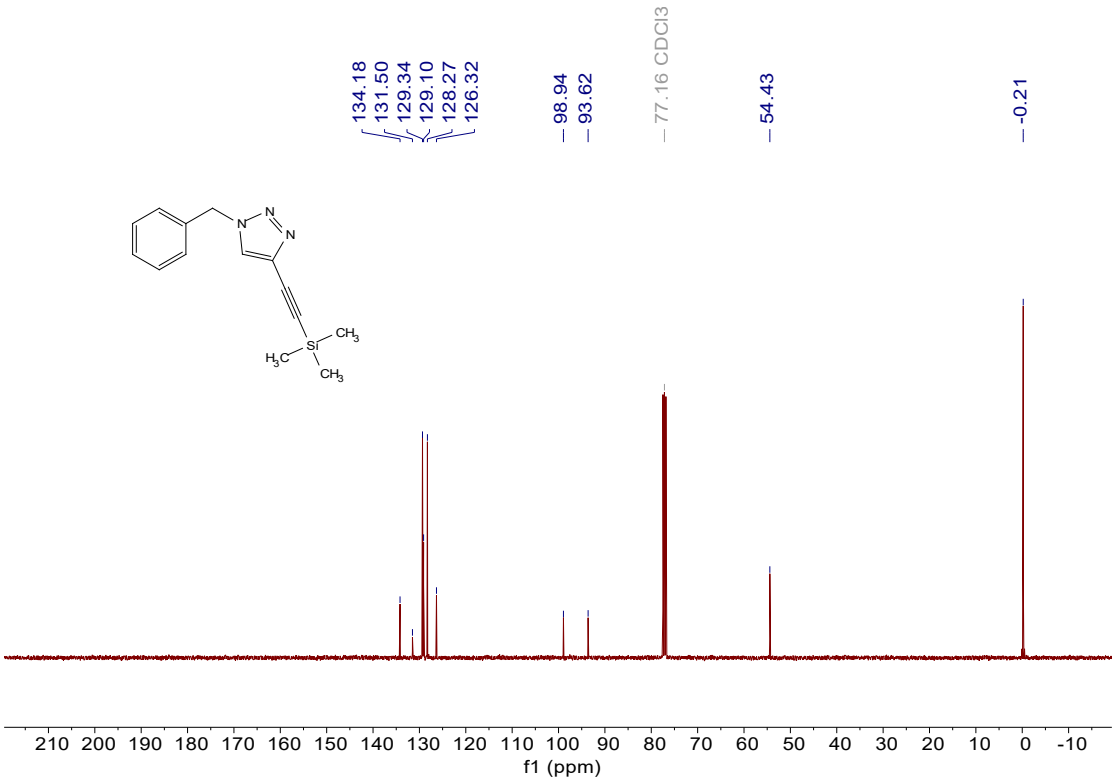


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-benzyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3j)

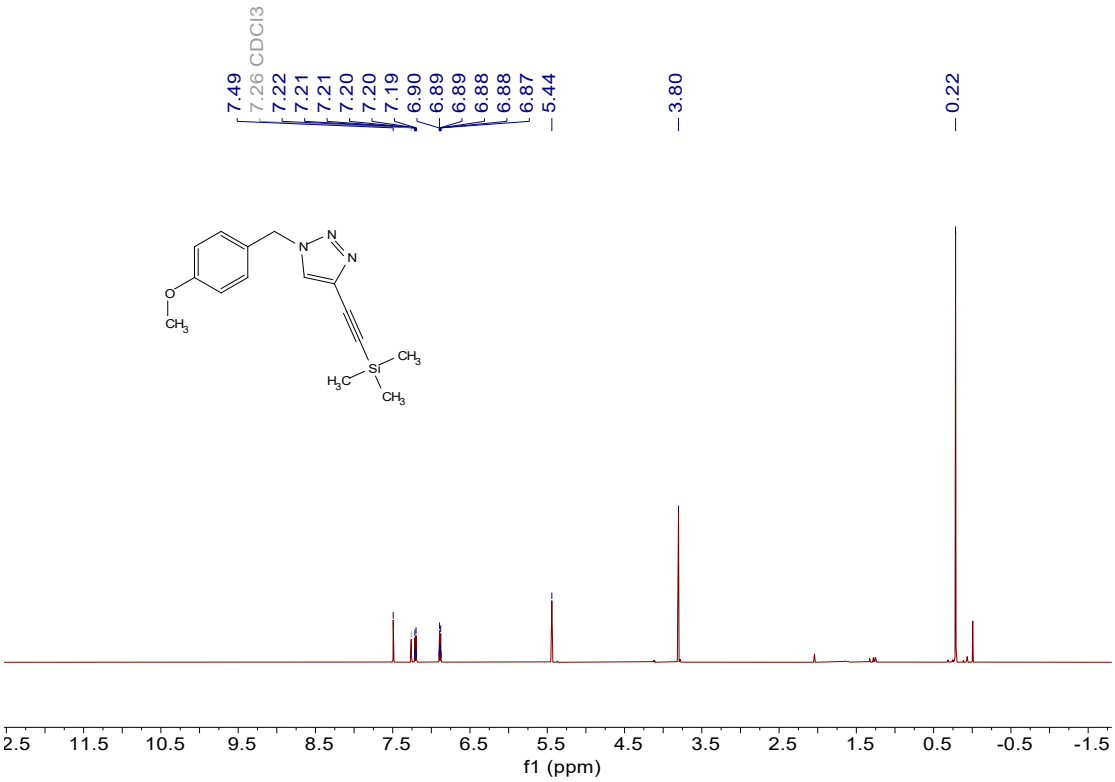


^{1}H NMR (400 MHz, Chloroform-*d*)

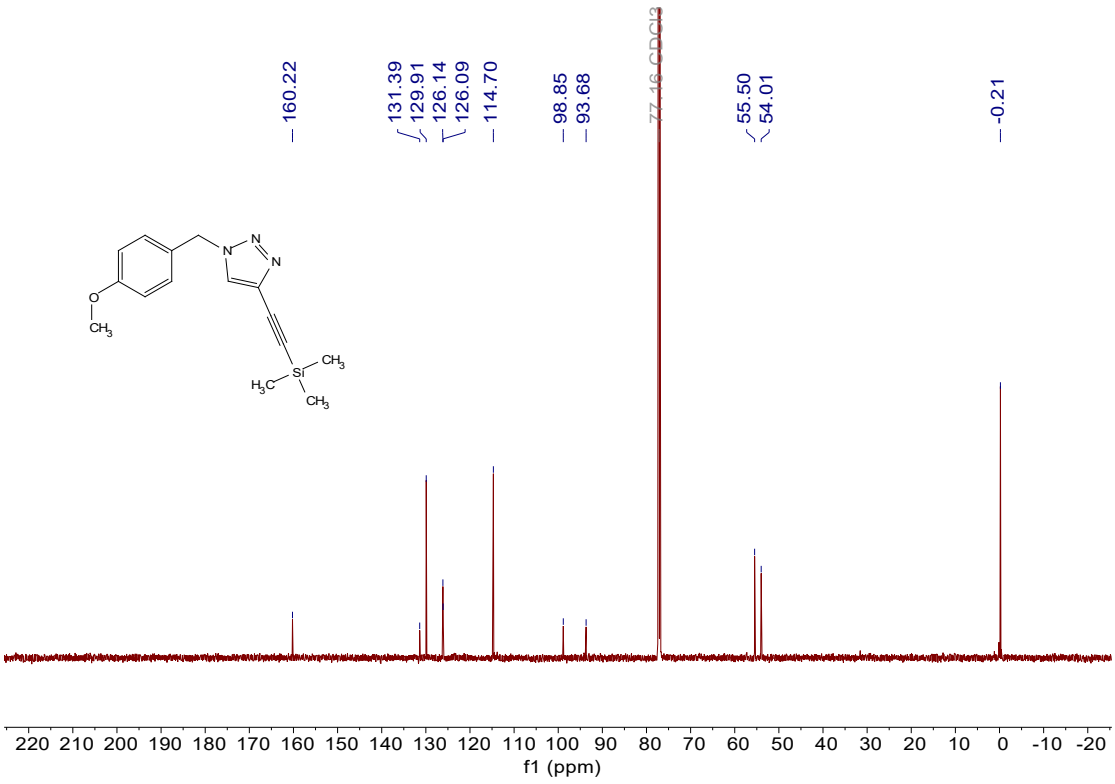


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-(4-methoxybenzyl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3k)

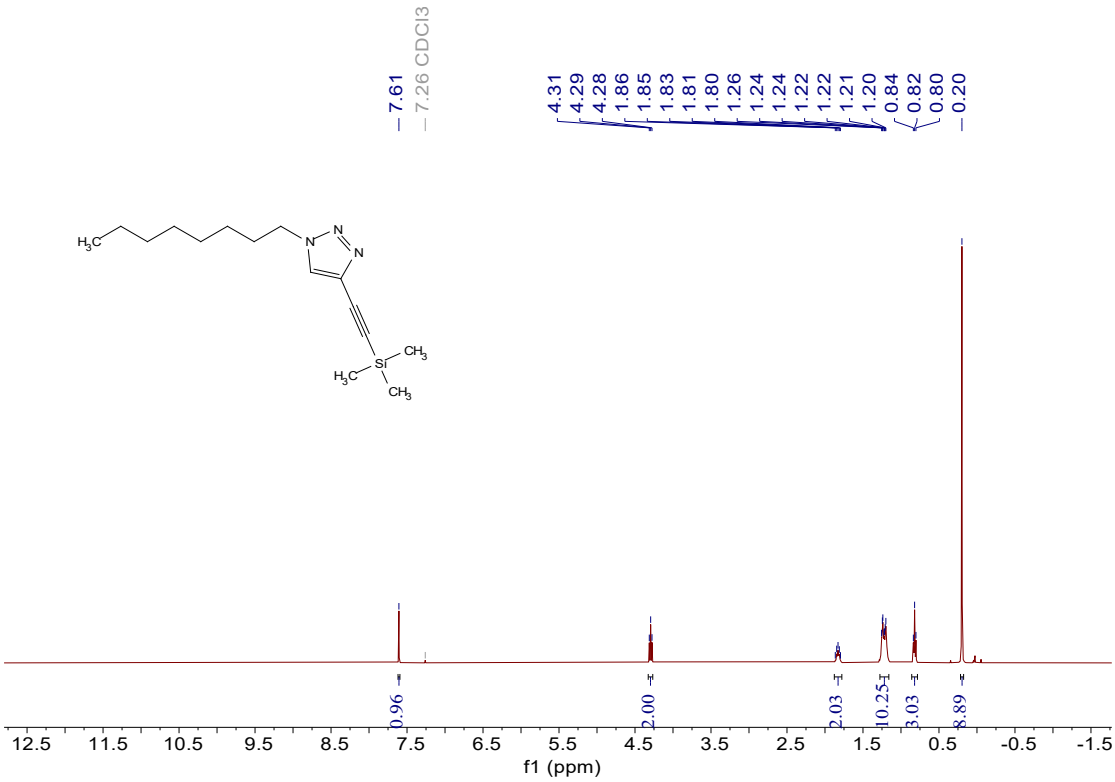


^{1}H NMR (600 MHz, Chloroform-*d*)

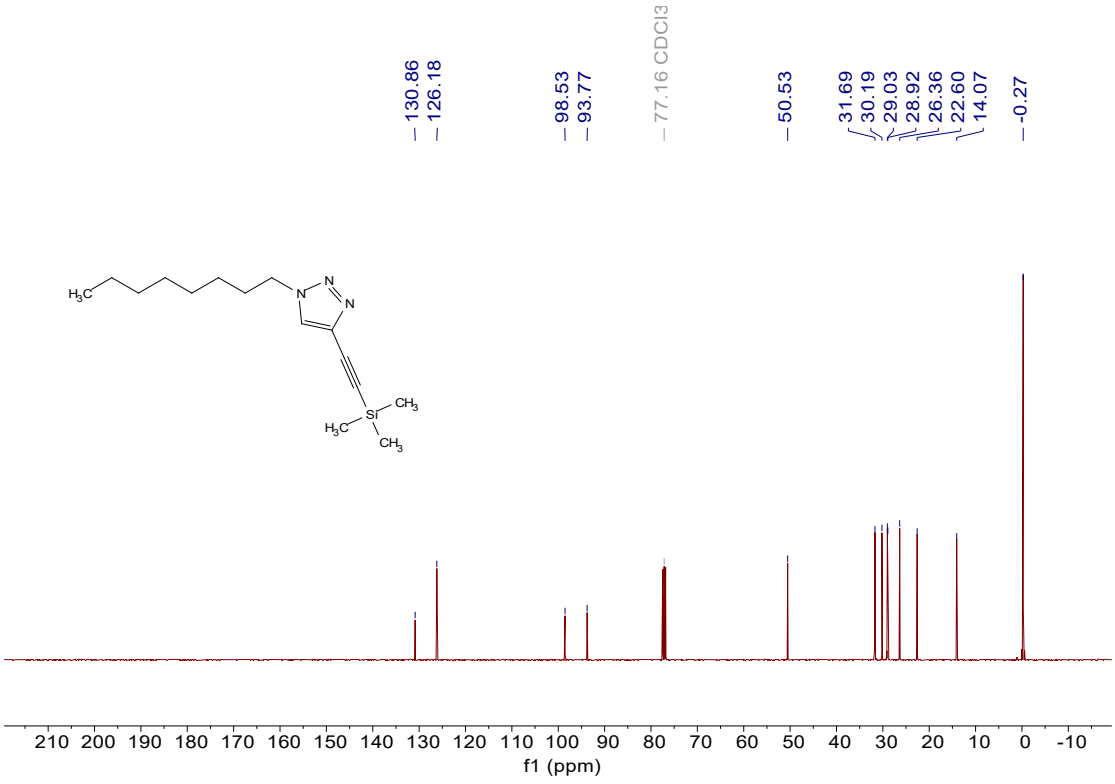


^{13}C{^{1}H} NMR (151 MHz, Chloroform-*d*)

1-octyl-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3l)

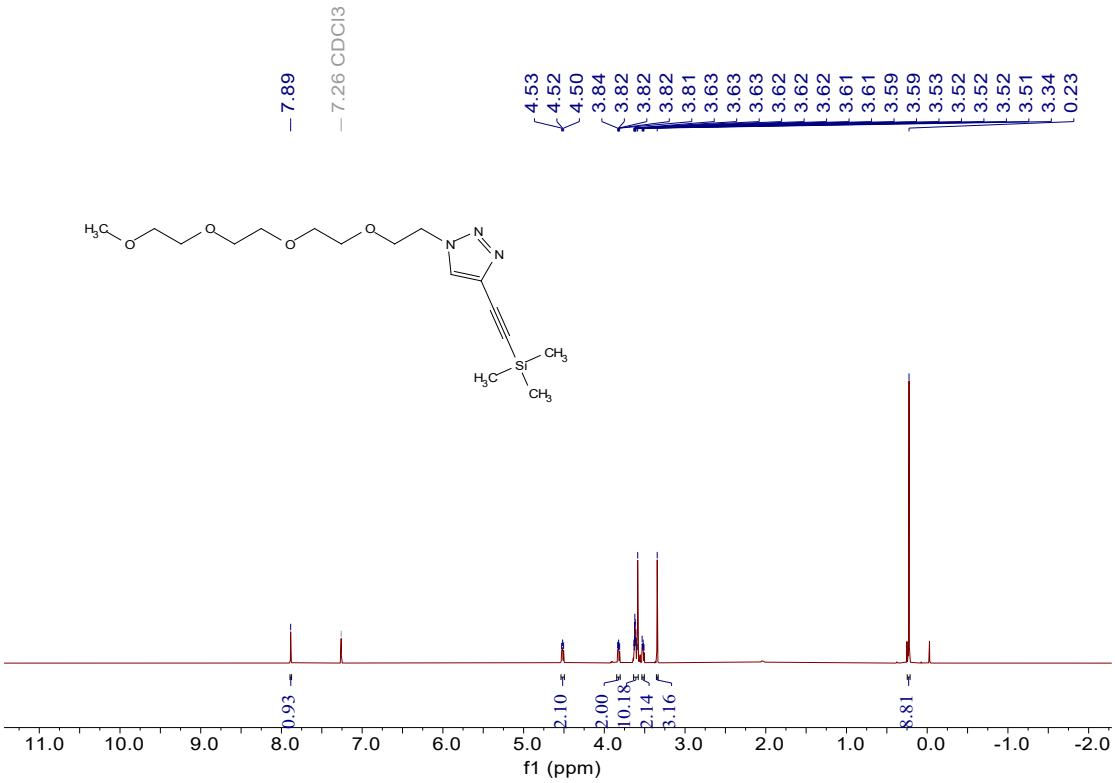


^{1}H NMR (400 MHz, Chloroform-*d*)

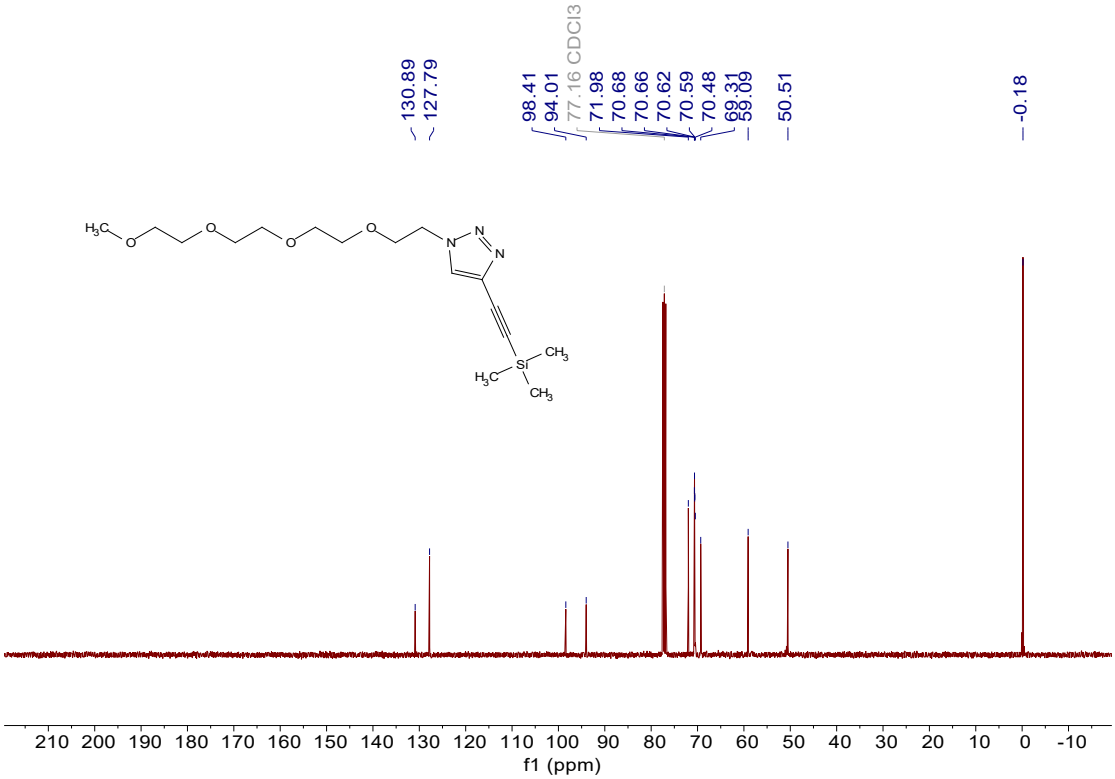


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-(2,5,8,11-tetraoxatridecan-13-yl)-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (3m)

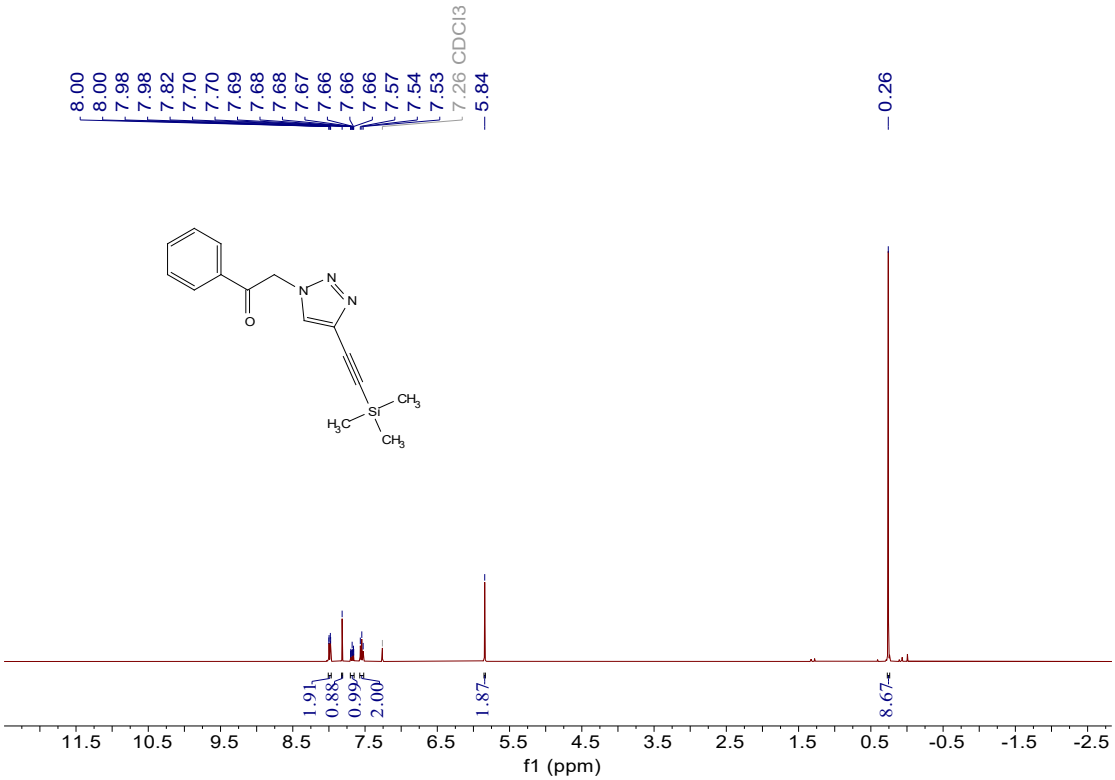


^{1}H NMR (400 MHz, Chloroform-*d*)

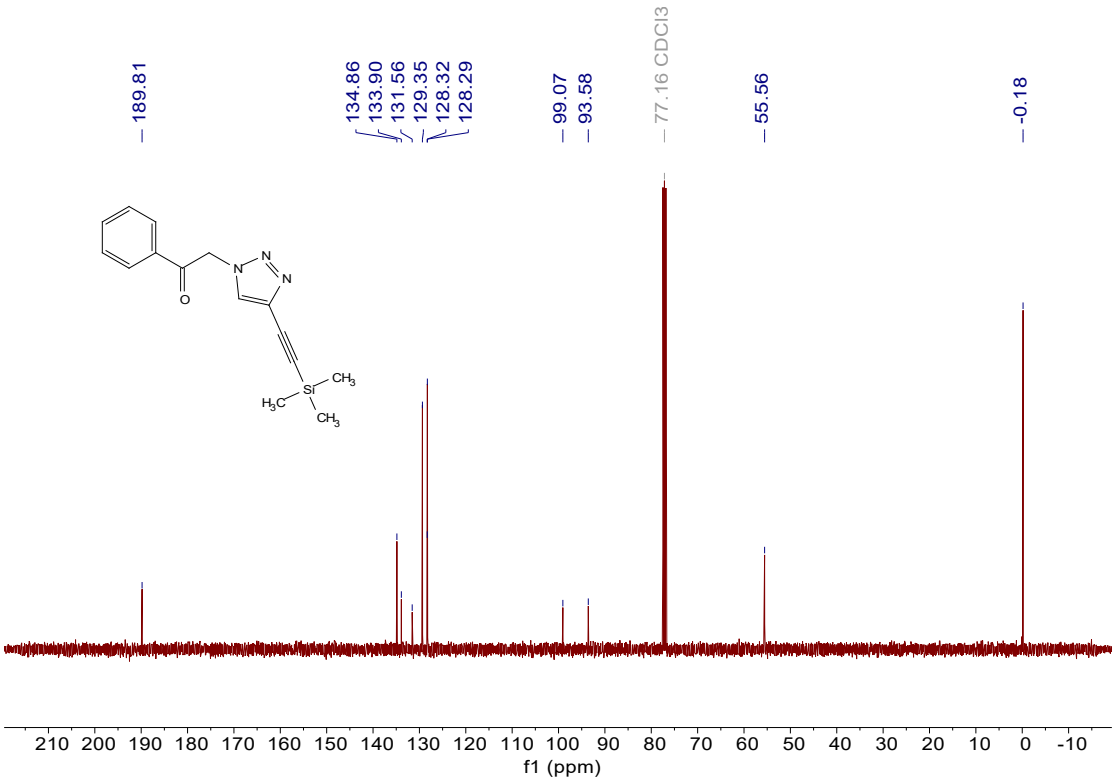


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

1-phenyl-2-(4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazol-1-yl)ethan-1-one (3n)

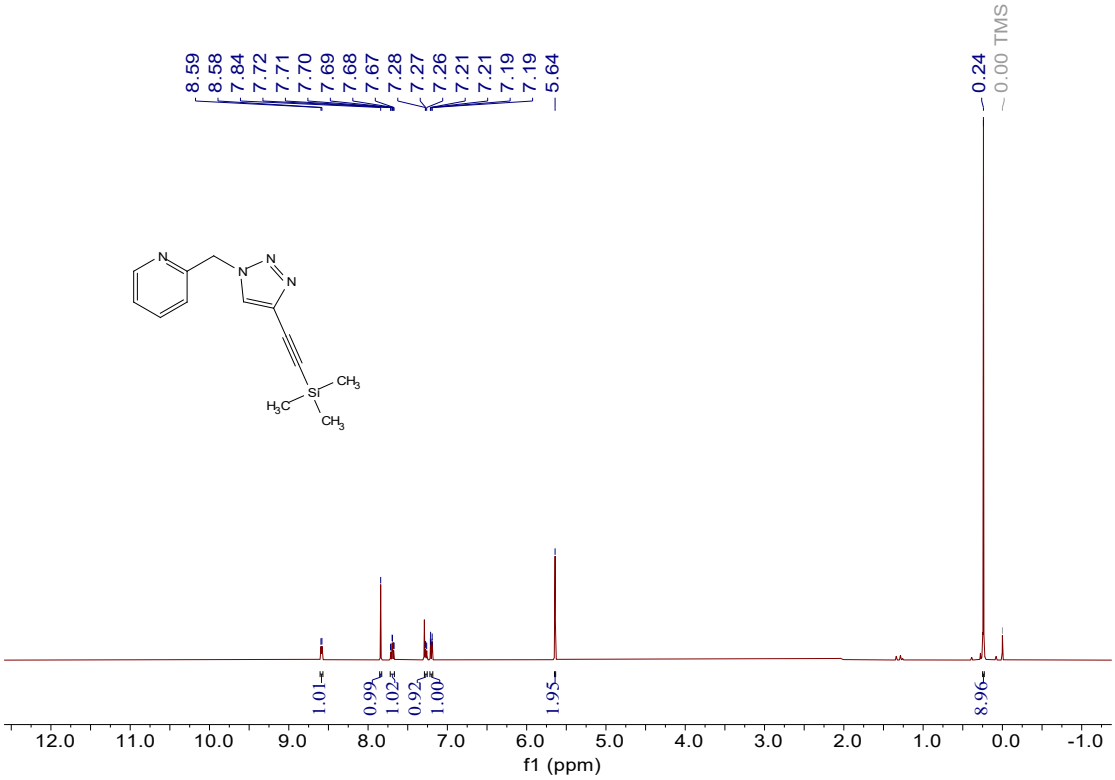


^{1}H NMR (400 MHz, Chloroform-*d*)

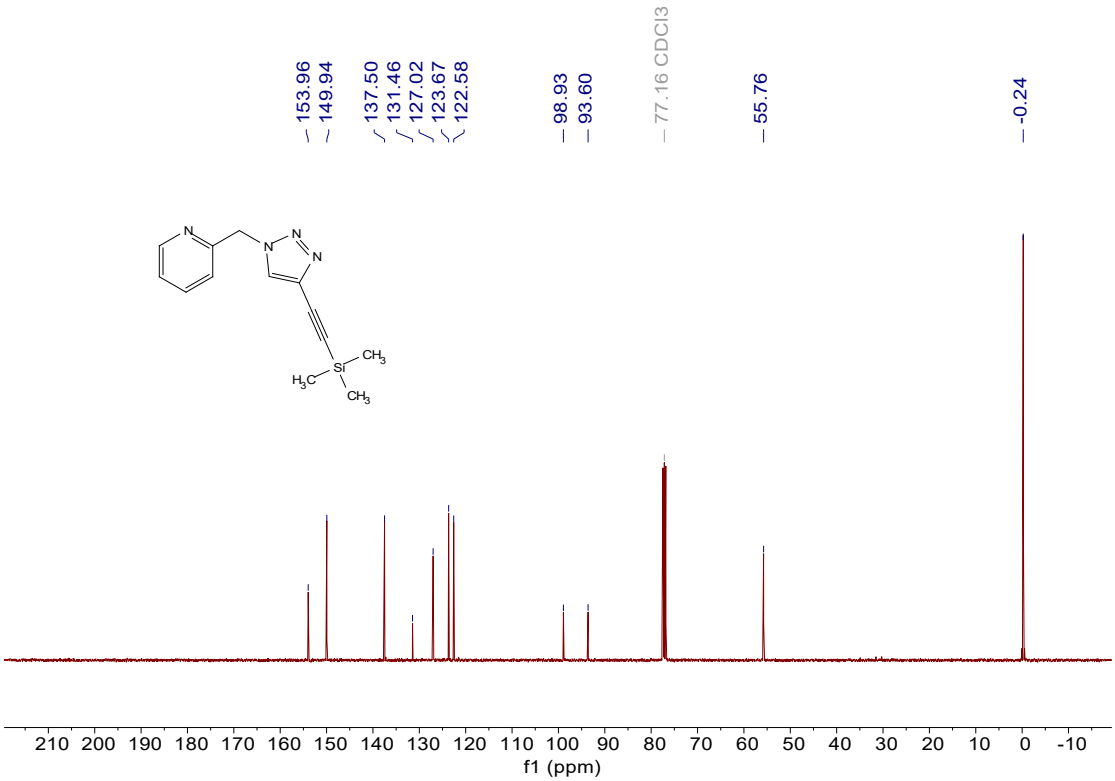


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

2-((4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazol-1-yl)methyl)pyridine (3o)



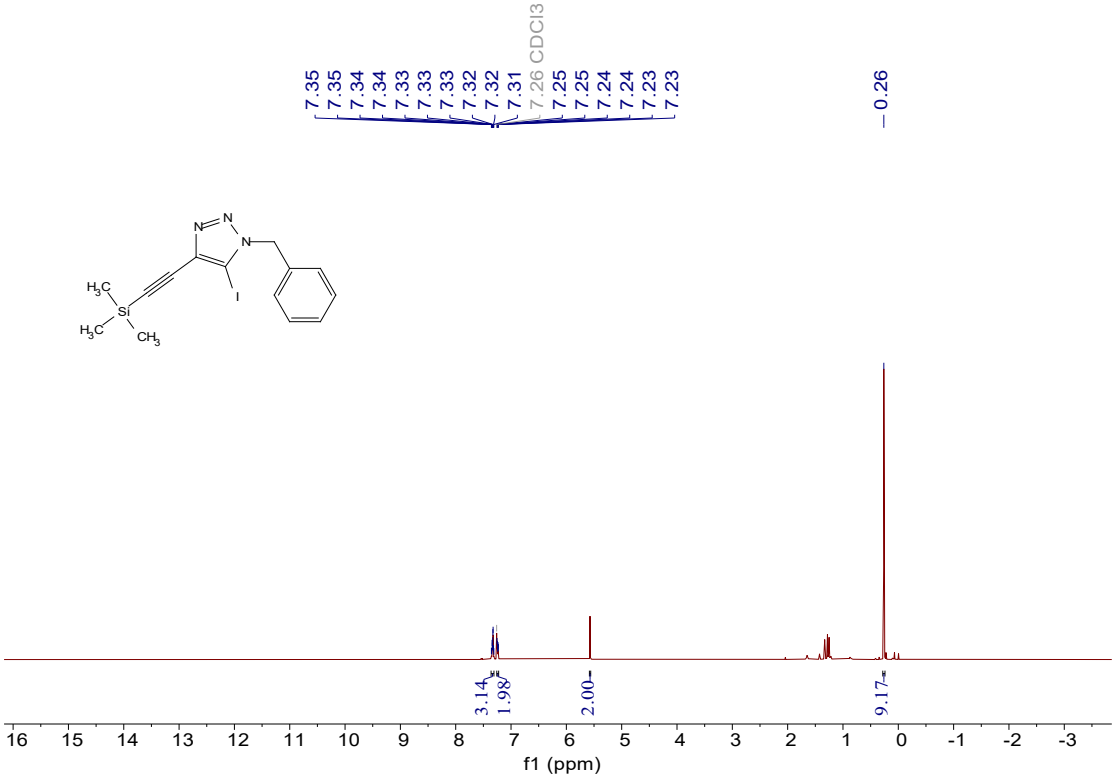
^{1}H NMR (400 MHz, Chloroform-*d*)



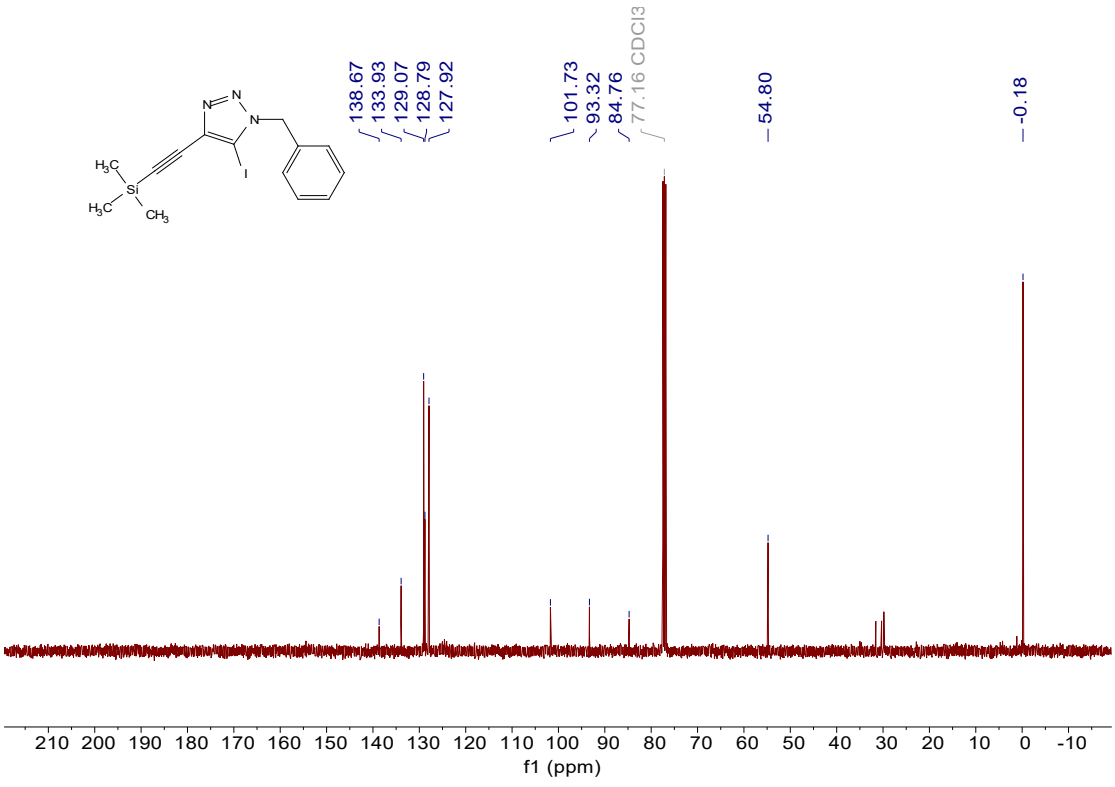
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.2 Copies of NMR spectra of compounds 4

1-benzyl-5-iodo-4-((trimethylsilyl)ethynyl)-1*H*-1,2,3-triazole (4)



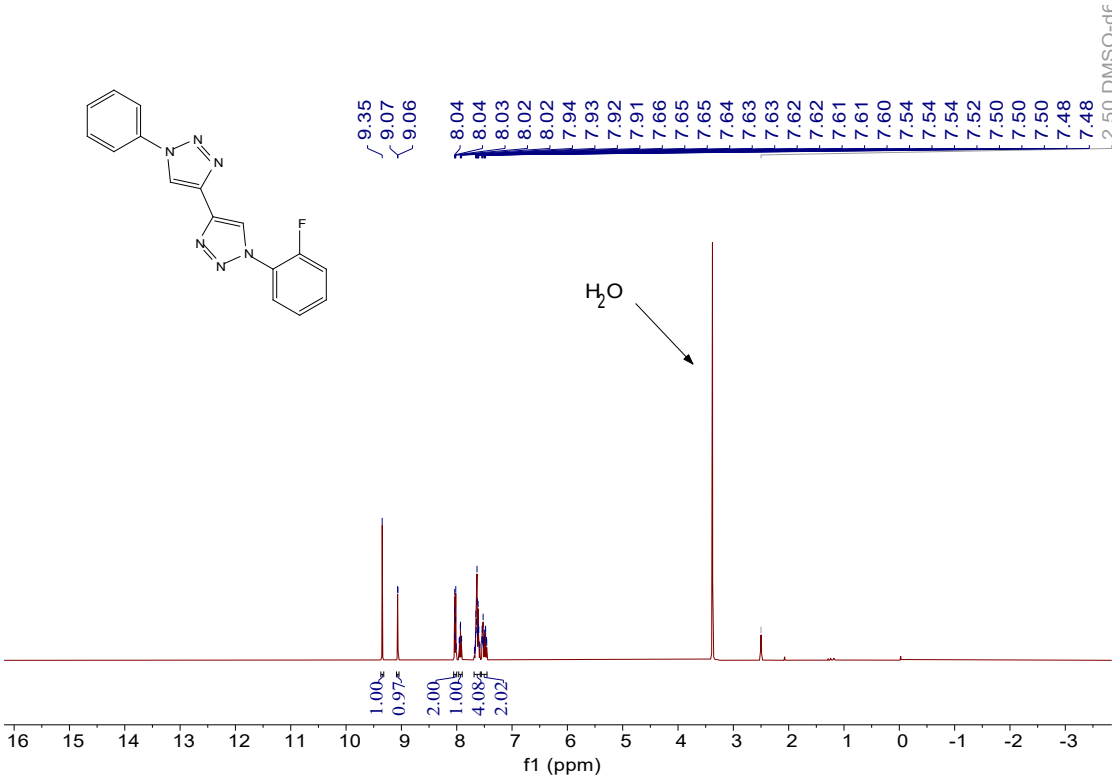
^{1}H NMR (400 MHz, Chloroform-*d*)



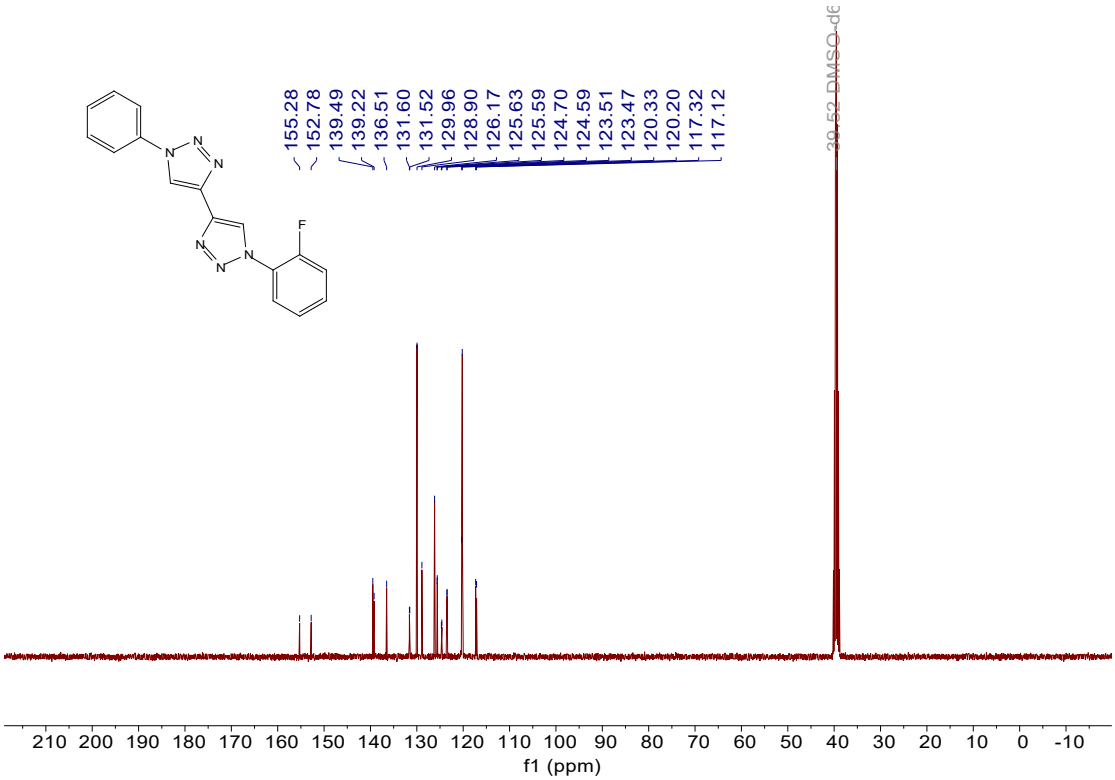
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.3 Copies of NMR spectra of compounds 5

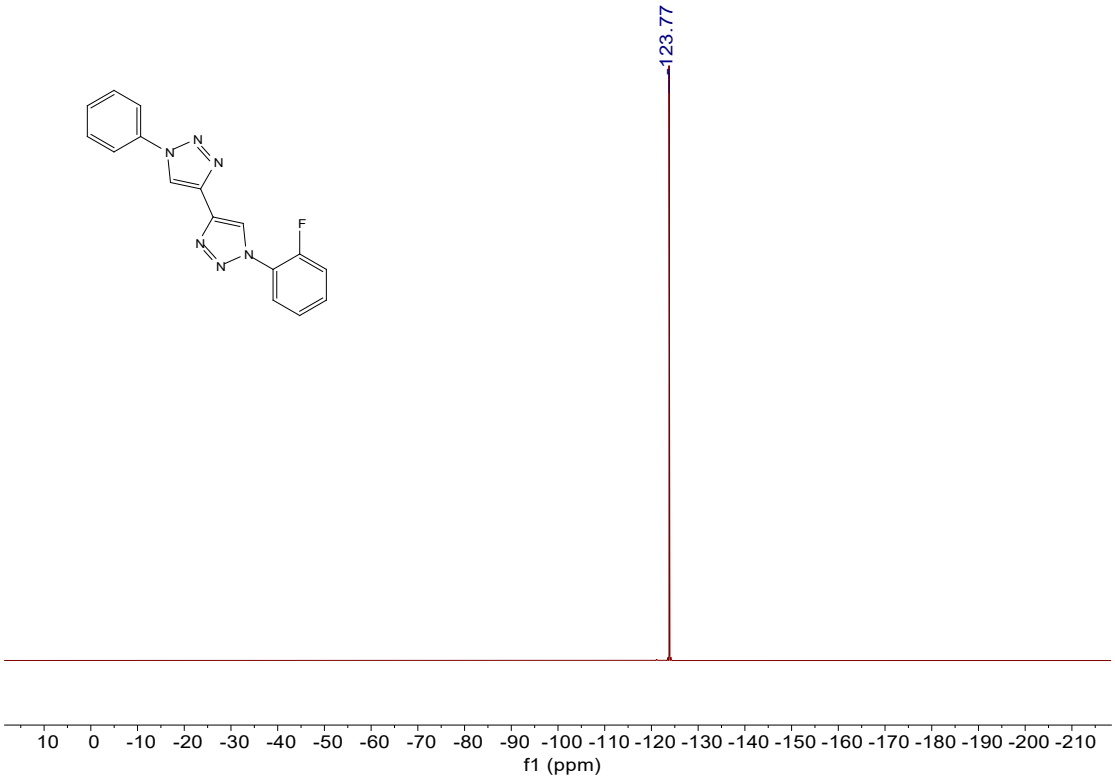
1-(2-fluorophenyl)-1'-phenyl-1*H*,1'*H*-4,4'-bi(1,2,3-triazole) (5)



^{1}H NMR (400 MHz, DMSO-d_6)



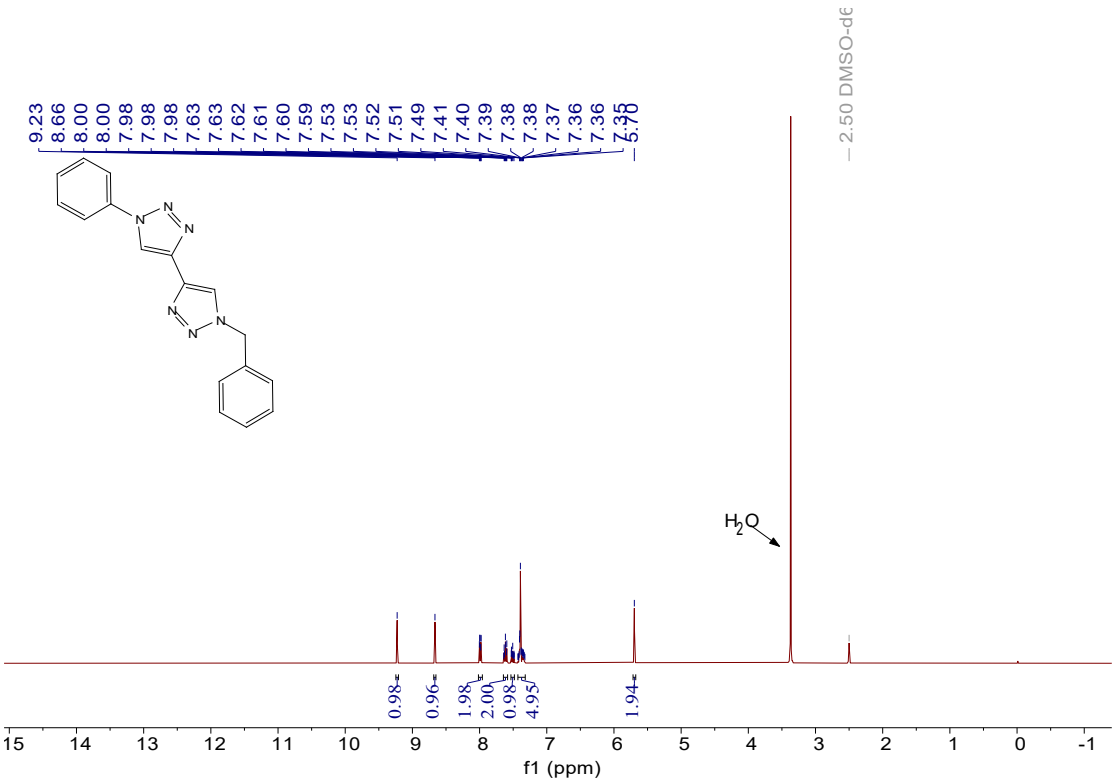
^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6)



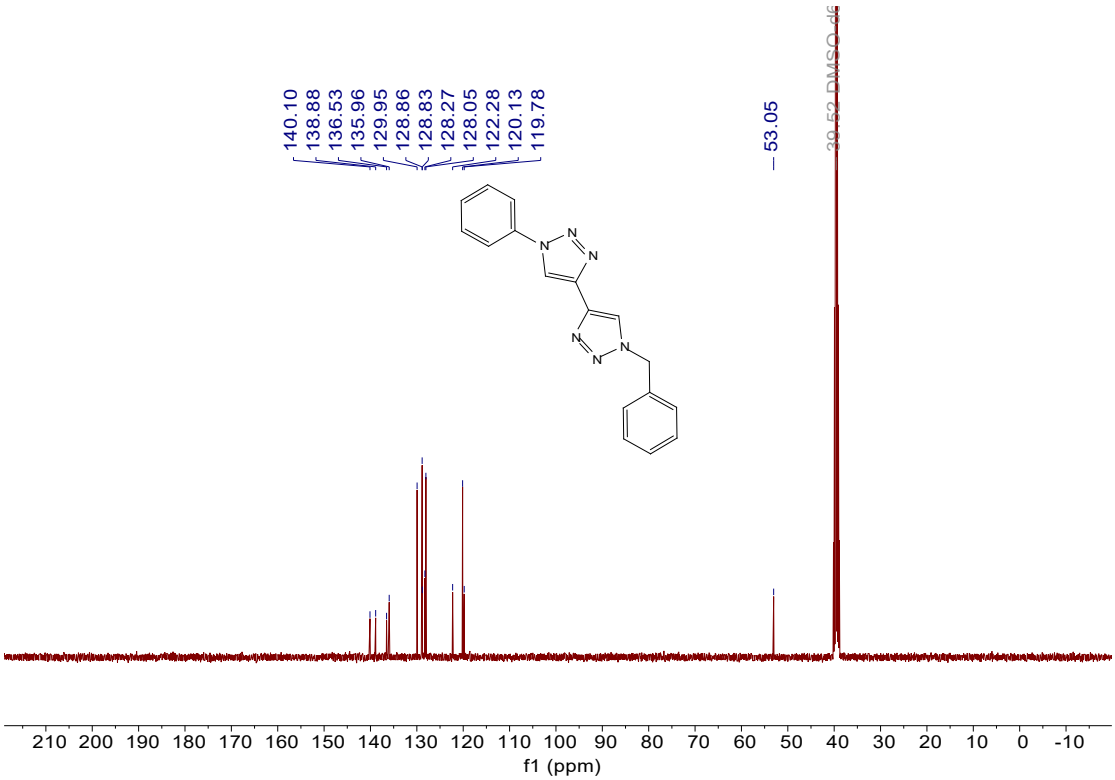
^{19}F NMR (377 MHz, DMSO-d_6)

5.4 Copies of NMR spectra of compounds 6

1-benzyl-1'-phenyl-1*H*,1'*H*-4,4'-bi(1,2,3-triazole) (6)



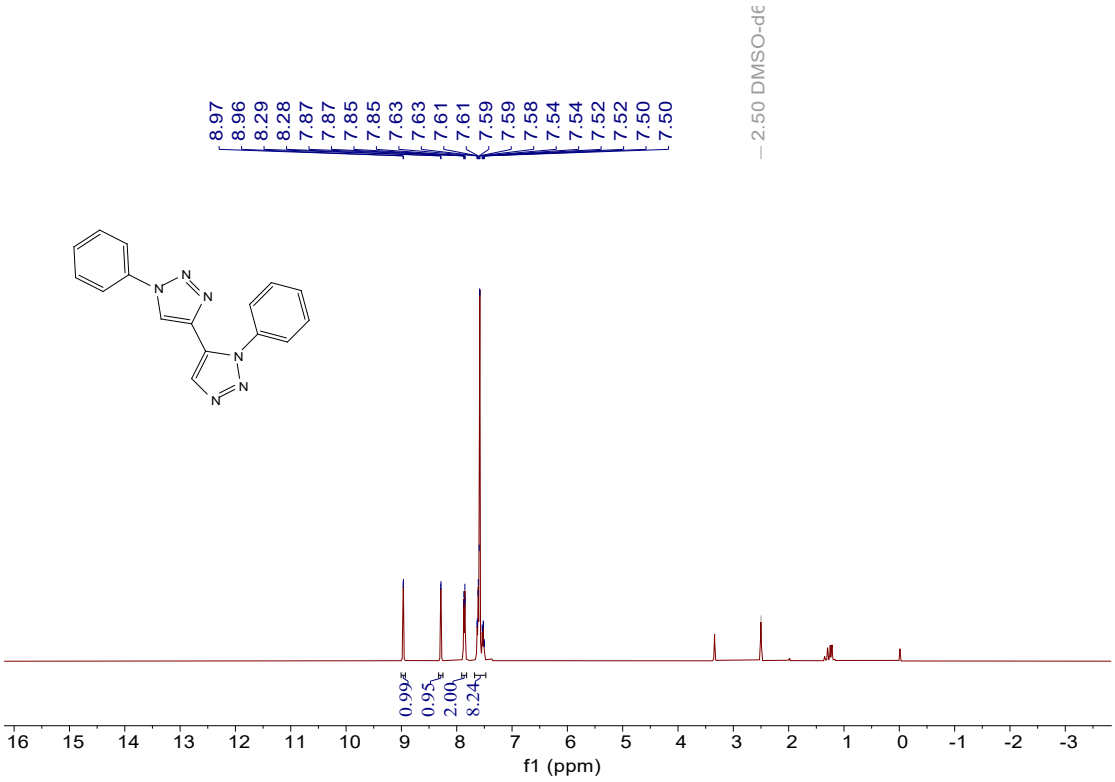
^{1}H NMR (400 MHz, DMSO-d_6)



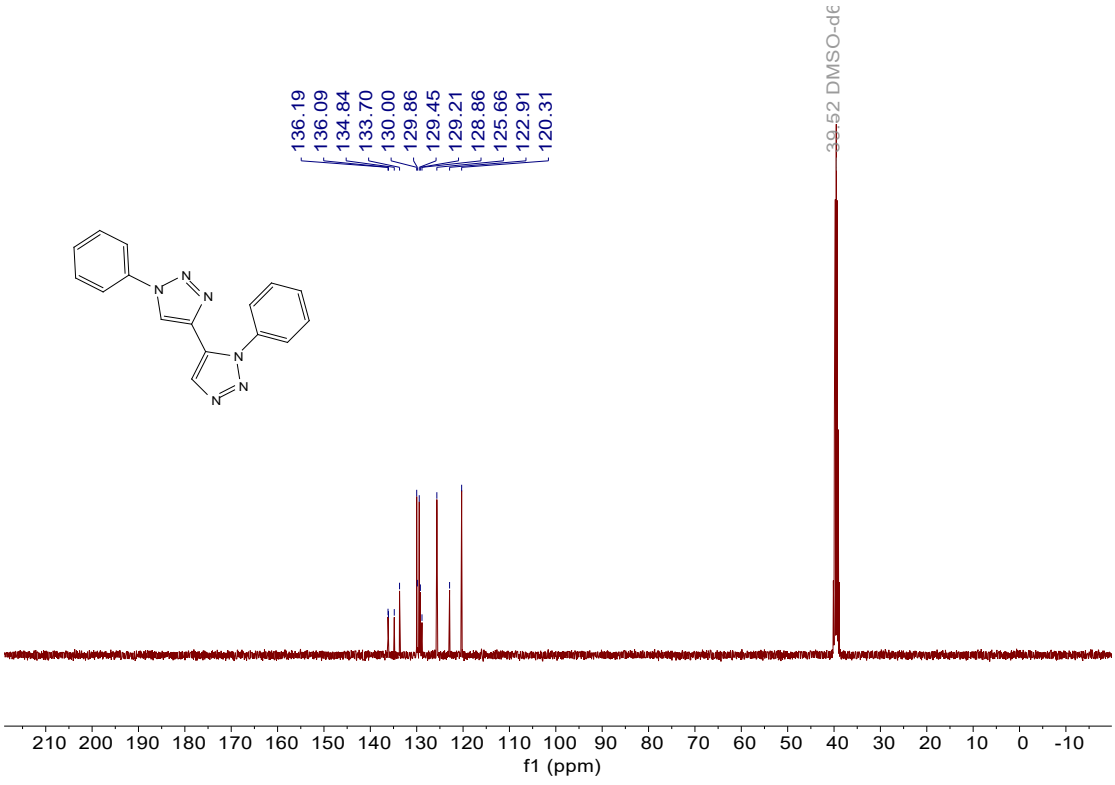
^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6)

5.5 Copies of NMR spectra of compounds 7

1,3'-diphenyl-1*H*,3'*H*-4,4'-bi(1,2,3-triazole) (7)



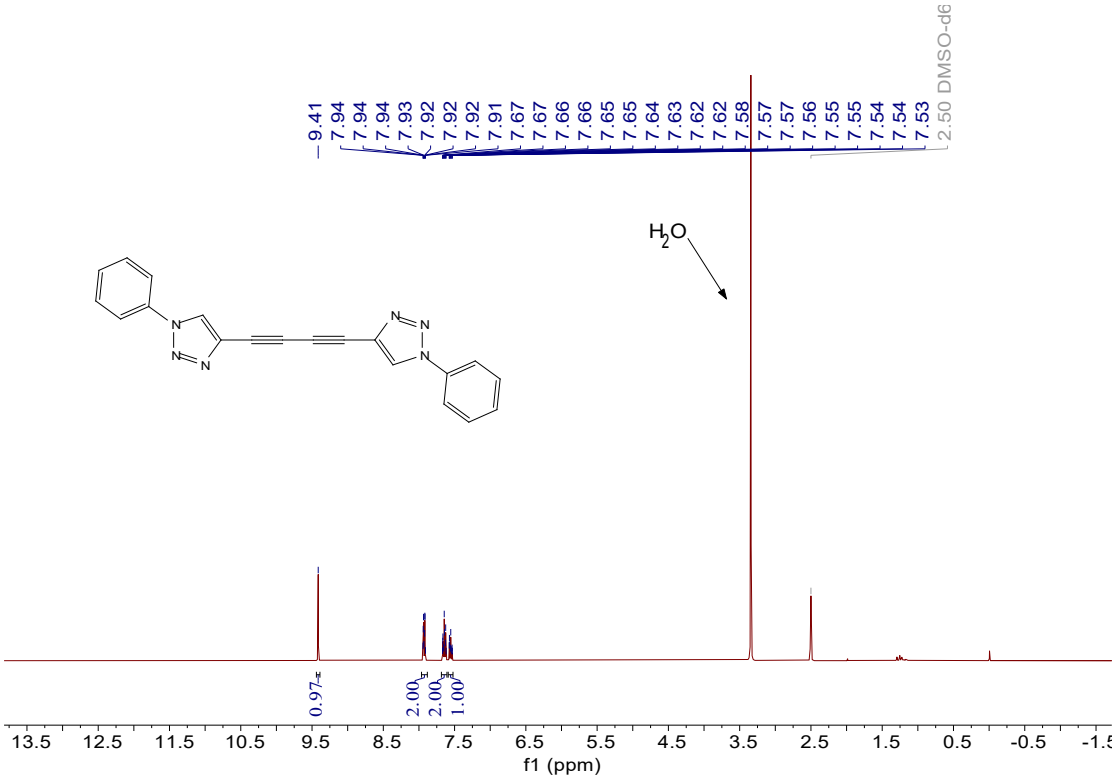
^{1}H NMR (400 MHz, DMSO-d_6)



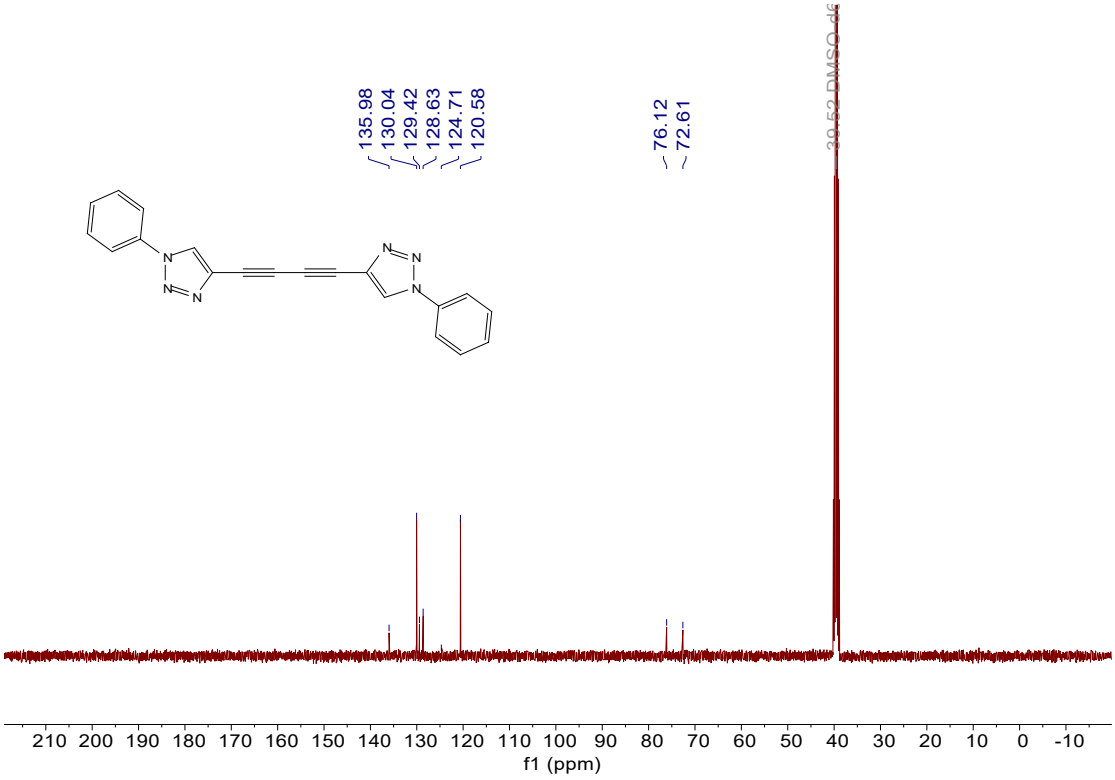
^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6)

5.6 Copies of NMR spectra of compounds 8

1,4-bis(1-phenyl-1*H*-1,2,3-triazol-4-yl)buta-1,3-diyne (8)



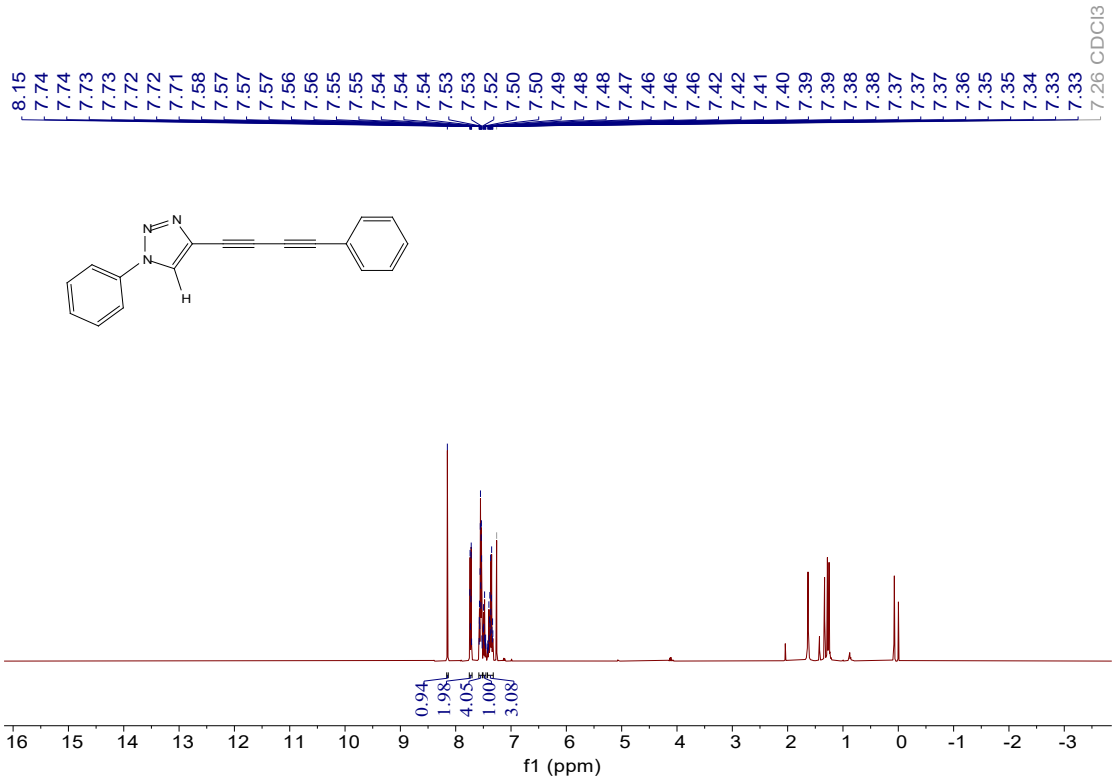
^{1}H NMR (400 MHz, DMSO-d_6)



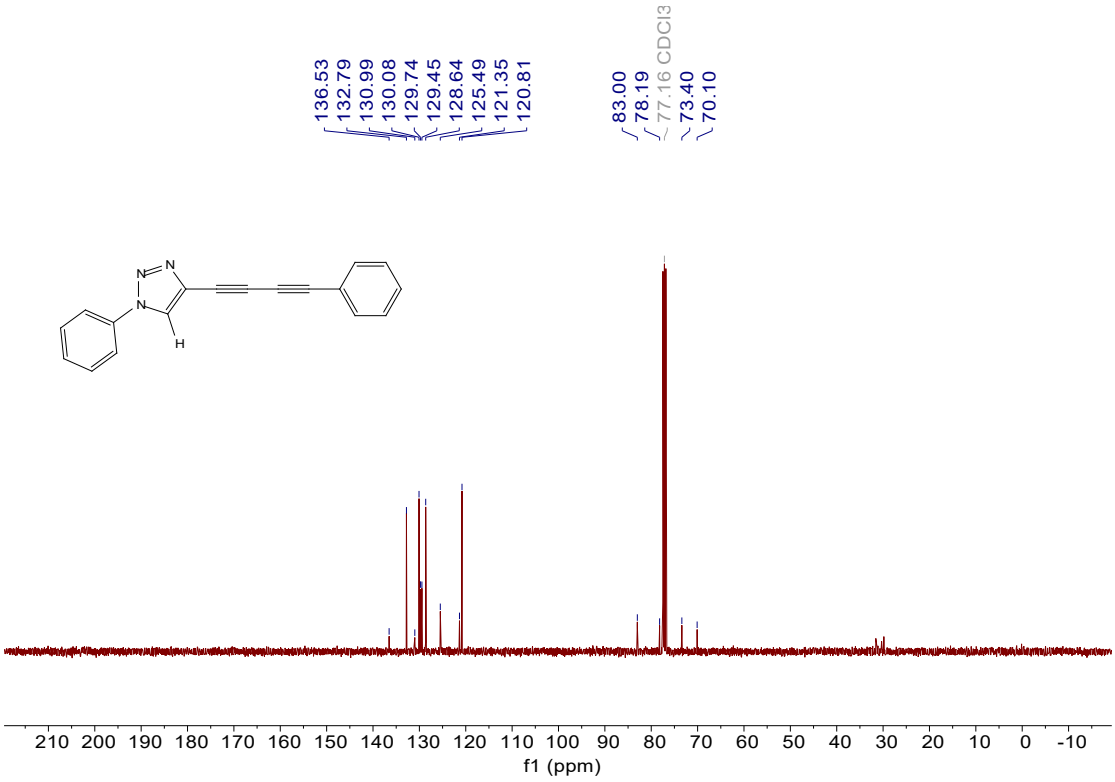
^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6)

5.7 Copies of NMR spectra of compounds 9

1-phenyl-4-(phenylbuta-1,3-diyn-1-yl)-1*H*-1,2,3-triazole (9)



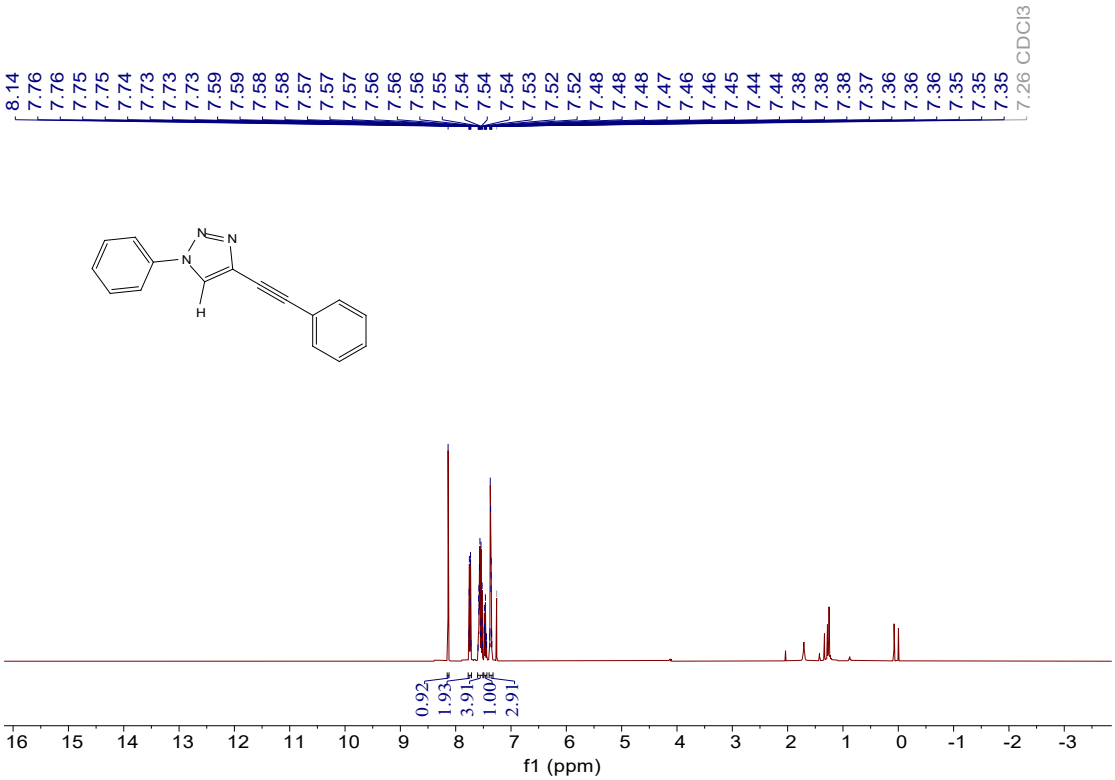
^{1}H NMR (400 MHz, Chloroform-*d*)



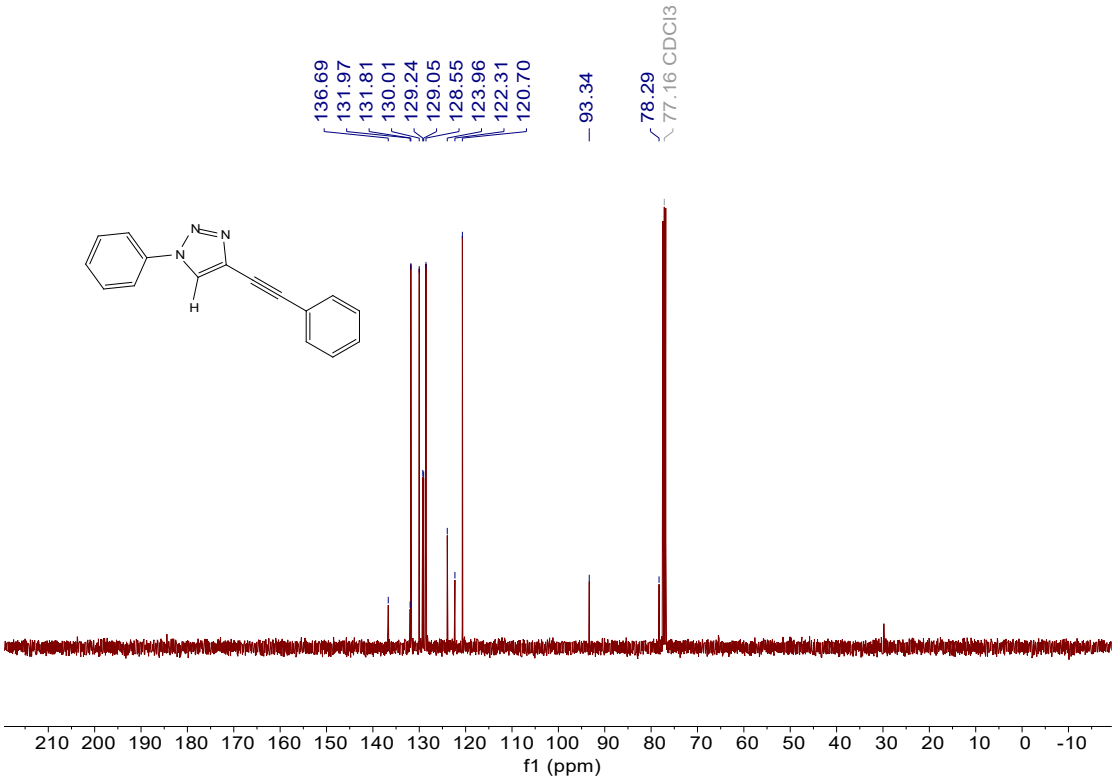
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.8 Copies of NMR spectra of compounds 10

1-phenyl-4-(phenylethynyl)-1*H*-1,2,3-triazole (10)



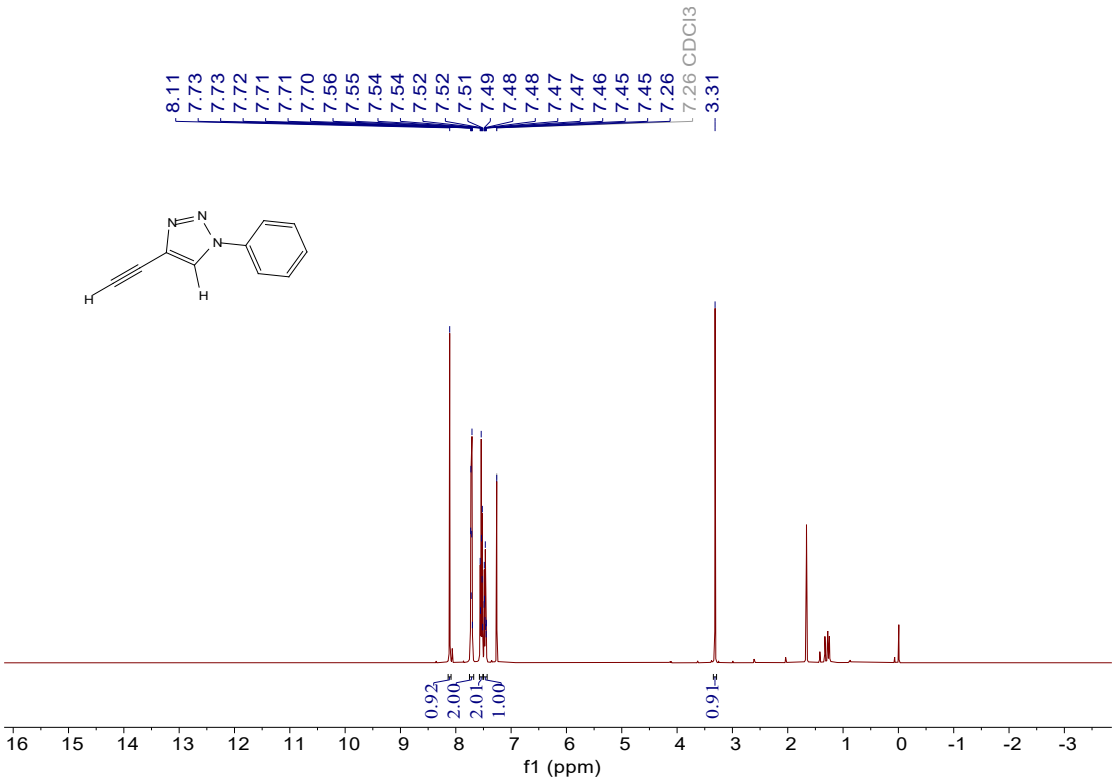
^{1}H NMR (400 MHz, Chloroform-*d*)



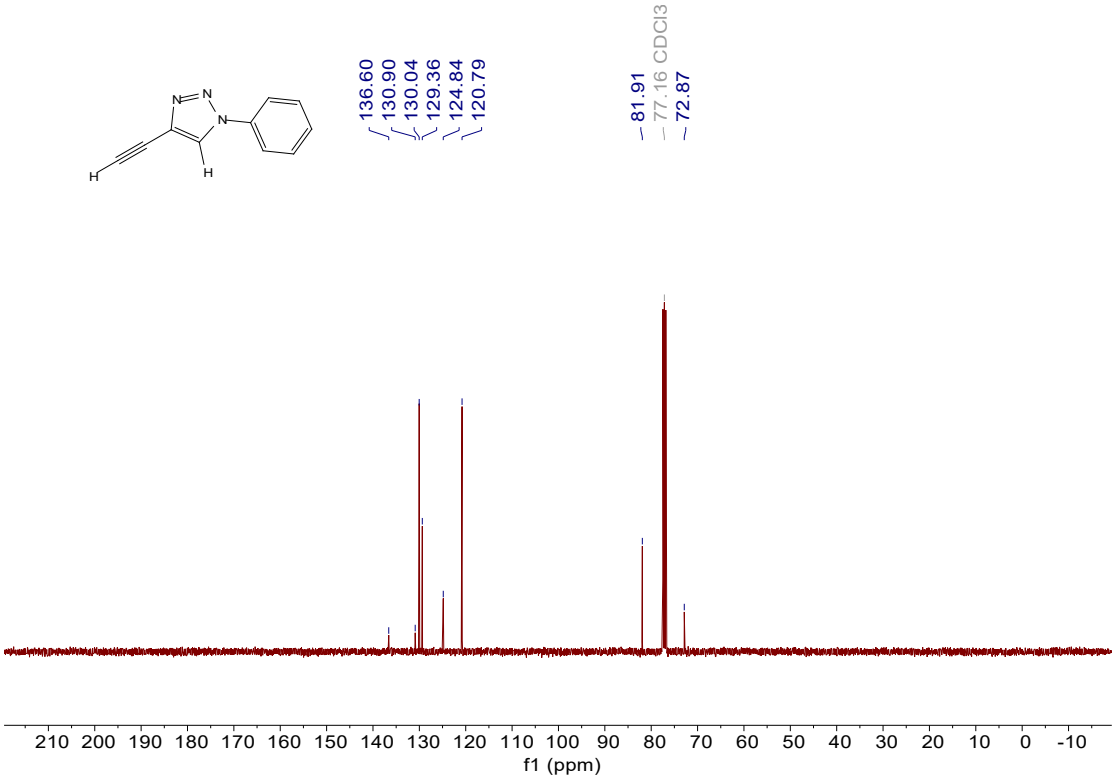
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.9 Copies of NMR spectra of compounds 11

4-ethynyl-1-phenyl-1*H*-1,2,3-triazole (11)



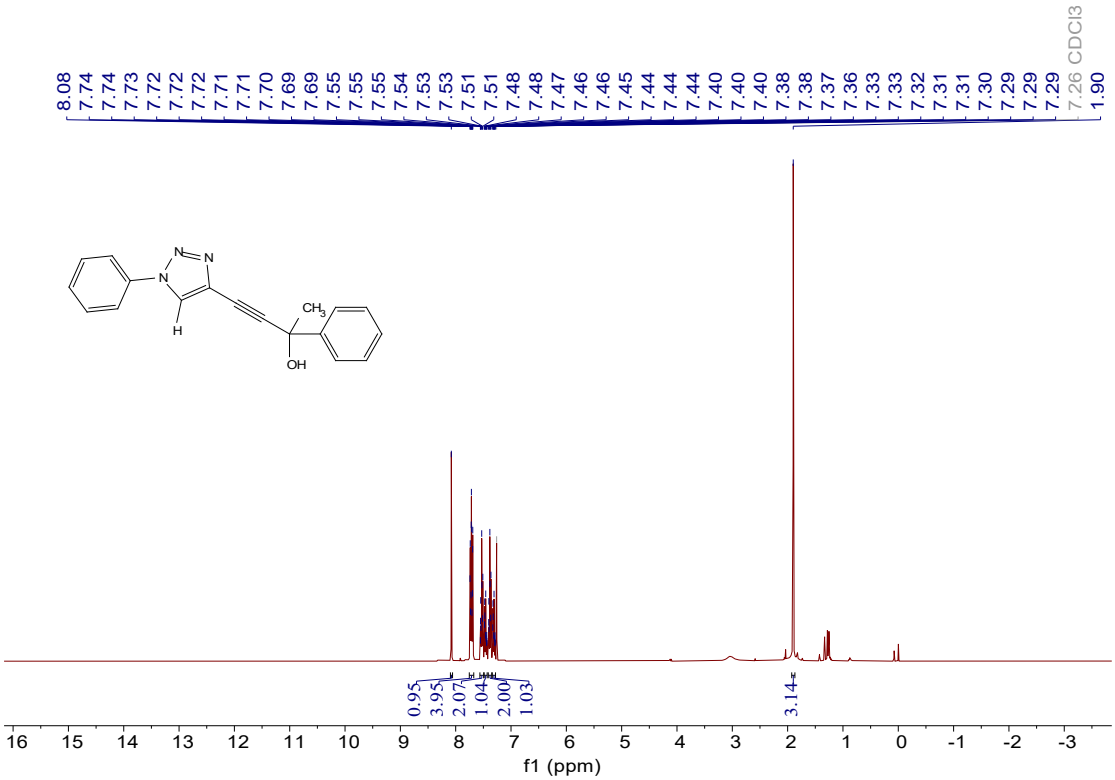
^{1}H NMR (400 MHz, Chloroform-*d*)



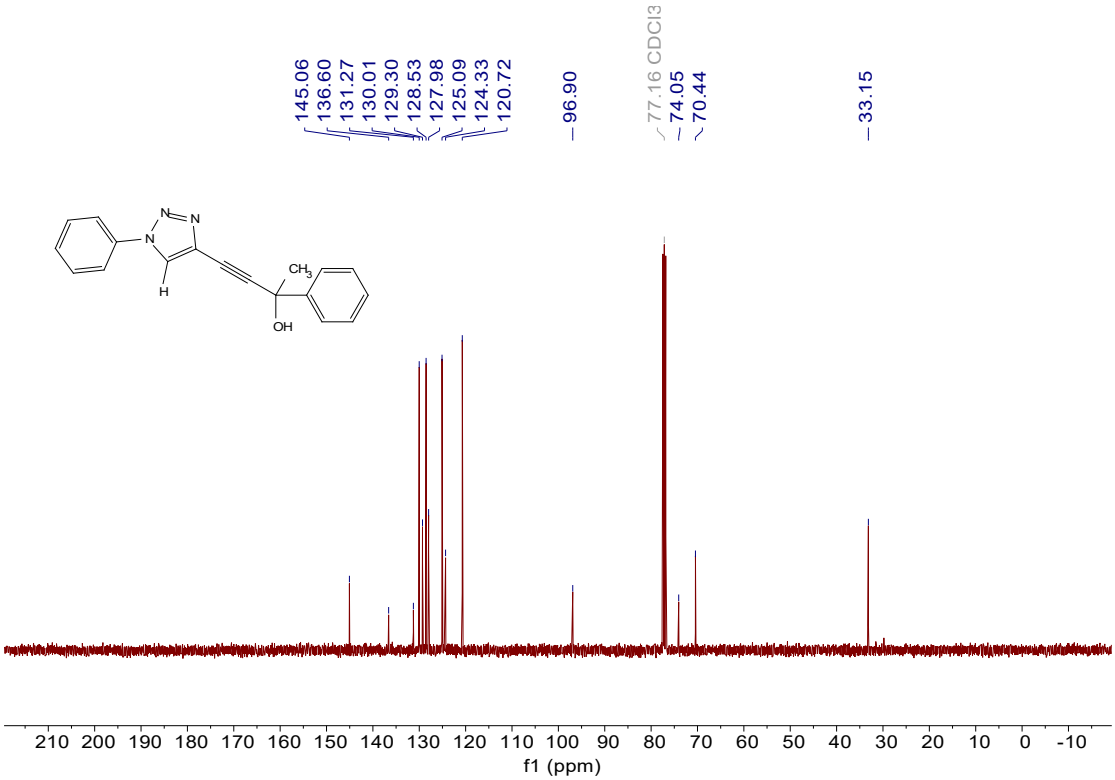
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.10 Copies of NMR spectra of compounds 12

2-phenyl-4-(1-phenyl-1*H*-1,2,3-triazol-4-yl)but-3-yn-2-ol (12a)

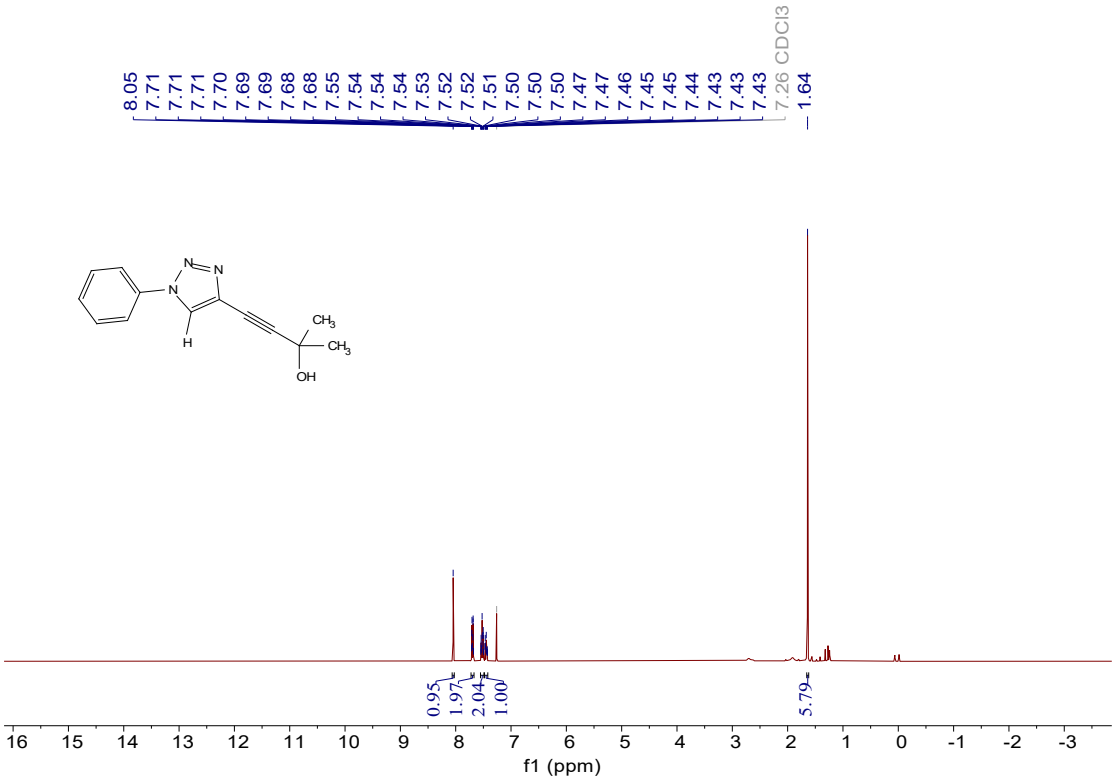


^{1}H NMR (400 MHz, Chloroform-*d*)

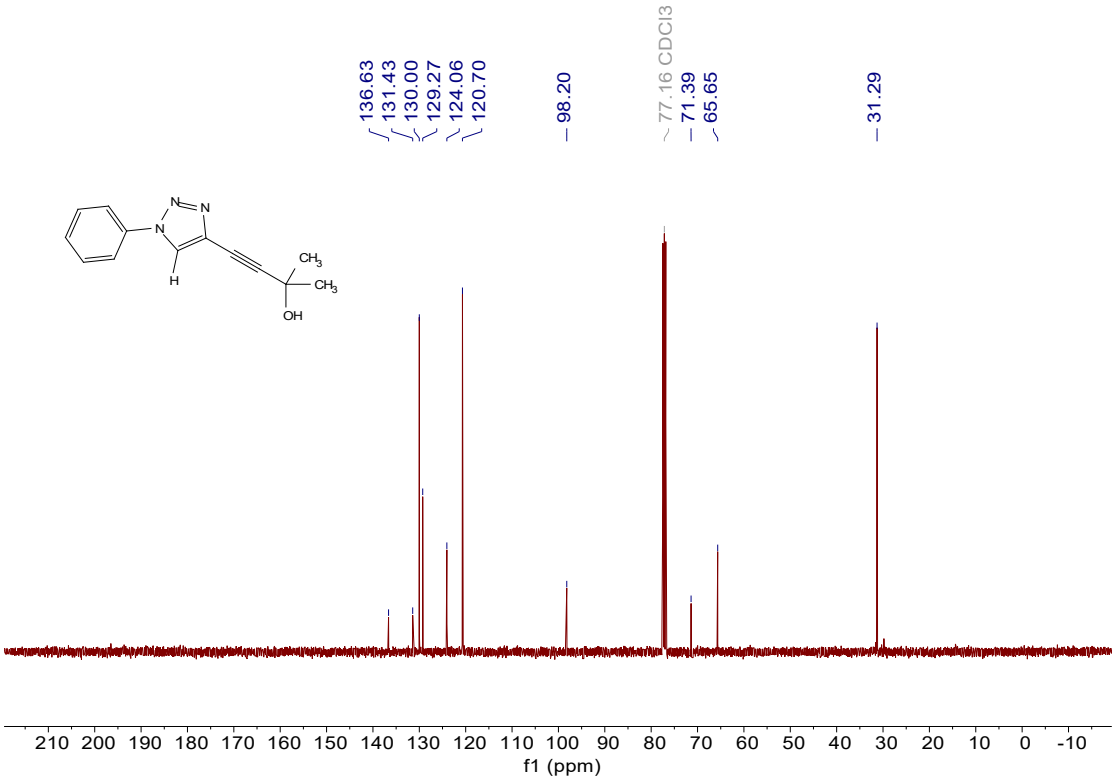


^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

2-methyl-4-(1-phenyl-1*H*-1,2,3-triazol-4-yl)but-3-yn-2-ol (12b)



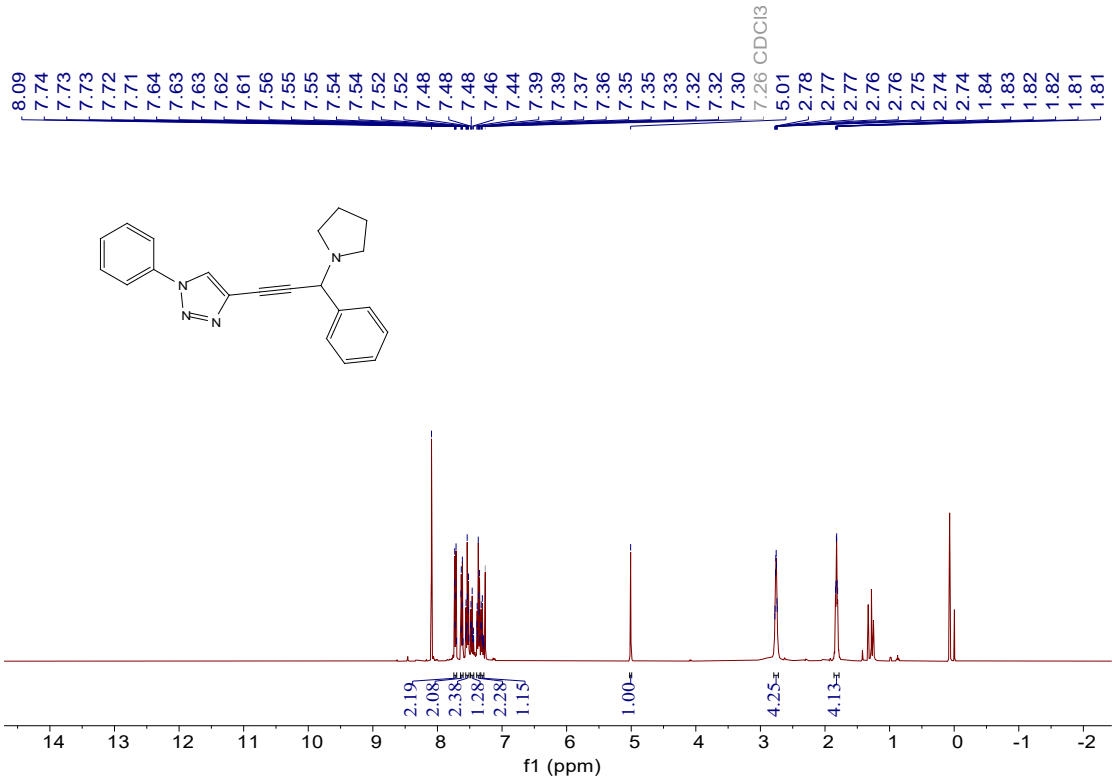
^{1}H NMR (400 MHz, Chloroform-*d*)



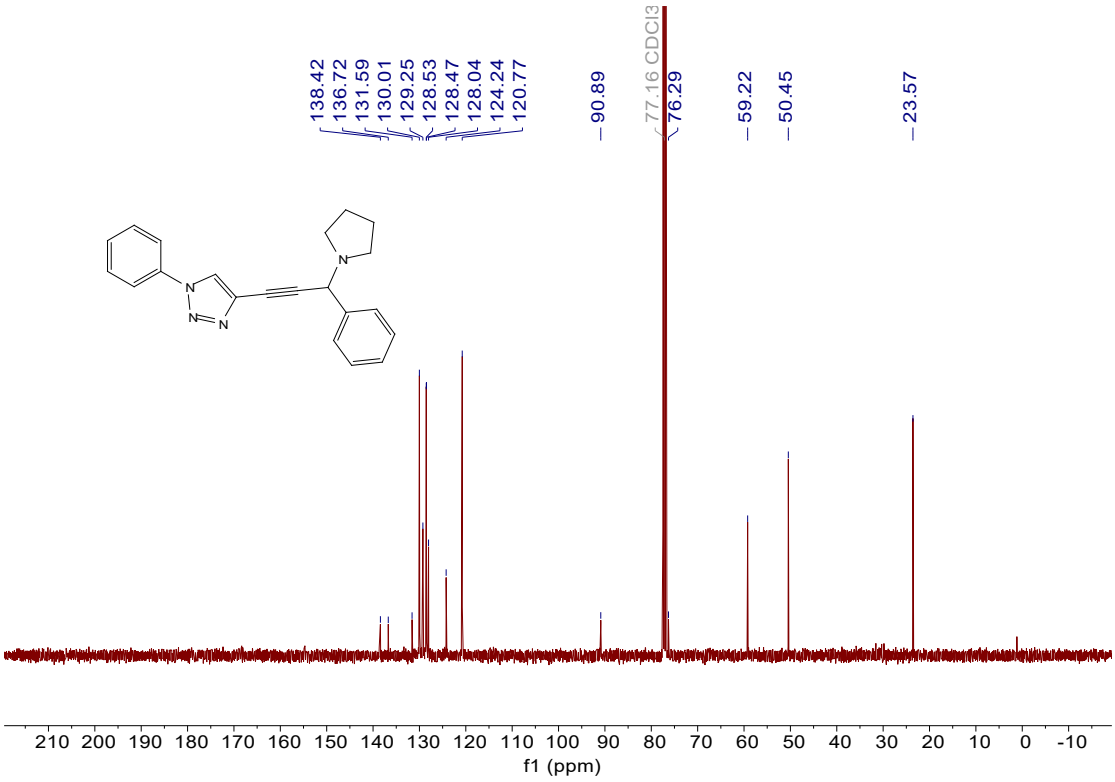
^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

5.11 Copies of NMR spectra of compounds 13

1-phenyl-4-(3-phenyl-3-(pyrrolidin-1-yl)prop-1-yn-1-yl)-1*H*-1,2,3-triazole (13)



^{1}H NMR (400 MHz, Chloroform-*d*)



^{13}C{^{1}H} NMR (101 MHz, Chloroform-*d*)

6. Reference

1. (*a*) V. Fiandanese, D. Bottalico, G. Marchese, A. Punzi and F. Capuzzolo, *Tetrahedron*, 2009, **65**, 10573–10580; (*b*) B. C. Doak, M. J. Scanlon and J. S. Simpson, *Org. Lett.*, 2011, **13**, 537–539.