Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Decarboxylative/Sonogashira-type Cross-Coupling using $PdCl_2(Cy^*Phine)_2$

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Table of Contents

1. General Considerations

Unless otherwise noted, all reagents were purchased commercially from Strem Chemicals, Sigma-Aldrich, or Alfa Aesar and used as received without further purification. All operations were carried out in an argon atmosphere using glovebox and Schlenk techniques unless otherwise specified. Anhydrous tetrahydrofuran (THF) and toluene were obtained from an argon purged solvent purification system comprised of columns of activated alumina and molecular sieves. Anhydrous *N,N'*-dimethylformamide (DMF), acetonitrile (CH_3CN), dimethyl sulfoxide (DMSO) and 1,4-dioxane were purchased from Sigma-Aldrich as sure-sealed solvents and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) on 0.25 mm E. Merck silica gel plates (60F-254) using UV light as the visualizing agent. E. Column chromatography was carried out on silica gel (200-300 mesh) by elution with appropriate solvents. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous materials, unless otherwise stated. Gas chromatography analysis was performed on an Agilent HP-7890 instrument with a flame ionization detector (FID) and an HP-5MS capillary column (30 m, 0.25 mm i.d., 0.25 μm film thicknesses) using helium as the carrier gas. Gas chromatography-mass spectrometry analysis was carried out on an Agilent HP-7890 instrument with an Agilent HP-5975 with triple-axis detector and HP-5MS capillary column using helium carrier gas. NMR spectra were from a Bruker DRX-600, instrument and calibrated using residual non-deuterated solvent ($CDCl_3$: δ_H = 7.26 ppm, δ_C = 77.16 ppm; C_6D_6: δ_H = 7.16 ppm, δ_C = 128.06 ppm) as an internal reference. Infrared (IR) spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer. High resolution mass spectra (HRMS) were recorded on an Agilent 6210 Series 1969A ESI-TOF (time of flight) mass spectrometer using ESI (electrospray ionization). $PdCl_2$(Cy*Phine)$_2$ was prepared according to previously reported method.[1]

2. General procedure for decarboxylative cross-coupling (Method 1)

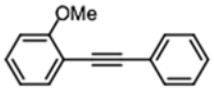
To a sealable reaction tube equipped with a magnetic stir bar was charged with $PdCl_2$(Cy*Phine)$_2$ (6.4 mg, 1 mol%), Cs_2CO_3 (391 mg, 1.2 mmol), organic chloride (0.5 mmol), alkynyl carboxylic acid (0.6 mmol) and THF (2 mL). The tube was then crimp-sealed with a cap fitted with a Teflon-lined septum and heated to 80 °C for given hours with vigorous stirring. The mixture was cooled to room temperature, diluted with EtOAc and filtered through a pad of Celite. The filtrate was

concentrated *in vacuo* affording the crude product which was purified by flash chromatography on silica gel.

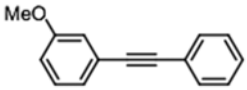
3. General procedure of tandem Sonogashira/decarboxylative cross-coupling for synthesis of symmetric di(heteroaryl)alkynes (Method 2)

To a sealable reaction tube equipped with a magnetic stir bar was charged with $PdCl_2(Cy^*Phine)_2$ (25.7 mg, 5 mol%), Cs_2CO_3 (391 mg, 1.2 mmol), heteroaryl chloride (1.0 mmol), propiolic acid (0.4 mmol) and 1,4-dioxane (2 mL). The tube was then crimp-sealed with a cap fitted with a Teflon-lined septum and heated to 120 °C for 24 h with vigorous stirring. The mixture was cooled to room temperature, diluted with EtOAc and filtered through a pad of Celite The filtrate was concentrated *in vacuo* affording the crude product which was purified by flash chromtography on silica gel.

4. Characterization data for products

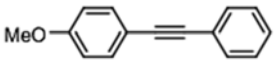


1-methoxy-2-(phenylethynyl)benzene (3a). Following method 1, 71.5 mg (0.5 mmol) of 2-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3a** as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.60 (dd, *J* = 8.0, 1.6 Hz, 2 H), 7.54 (dd, *J* = 7.5, 1.7 Hz, 1 H), 7.40–7.34 (m, 4 H), 7.01–6.90 (m, 2 H), 3.95 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 159.9, 133.6, 131.7, 129.8, 128.8, 128.4, 128.3, 128.2, 126.3, 123.5, 120.5, 112.4, 110.7, 93.5, 85.7, 55.9 ppm. The physical data were in full accordance with the literature value.[2]

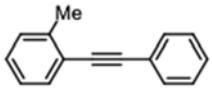


1-methoxy-3-(phenylethynyl)benzene (3b). Following method 1, 71.5 mg (0.5 mmol) of 3-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3b** as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.62–7.55 (m, 2 H), 7.40–7.37 (m, 3 H), 7.30 (dd, *J* = 8.4, 7.5 Hz, 1 H), 7.19 (d, *J* = 7.5 Hz, 1 H), 7.12 (dd, *J* = 2.7, 1.4 Hz, 1 H), 6.94 (ddd, *J* = 8.3, 2.7, 1.0 Hz, 1 H), 3.86 (s, 3 H) ppm. ^{13}C NMR (151 MHz,

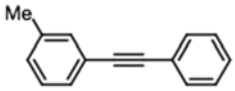
$CDCl_3$) δ = 159.3, 131.7, 129.5, 128.41, 128.37, 124.3, 124.2, 123.2, 116.3, 115.0, 89.3, 89.2, 55.3 ppm. The physical data were in full accordance with the literature value.[1]



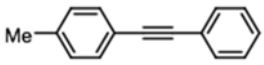
1-methoxy-4-(phenylethynyl)benzene (3c). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3c** as a yellow solid (103 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.52 (dd, *J* = 8.1, 1.5 Hz, 2 H), 7.48 (d, *J* = 8.9 Hz, 2 H), 7.40–7.27 (m, 3 H), 6.89 (d, *J* = 8.9 Hz, 2 H), 3.83 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 160.2, 133.6, 132.0, 129.8, 128.9, 128.5, 124.2, 115.9, 89.9, 88.6, 55.9 ppm. The physical data were in full accordance with the literature value.[2]



1-methyl-2-(phenylethynyl)benzene (3d). Following method 1, 63 mg (0.5 mmol) of 1-chloro-2-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3d** as a colourless oil (95 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.64–7.59 (m, 2 H), 7.57 (s, 1 H), 7.40 (dd, *J* = 9.2, 7.1 Hz, 3 H), 7.31–7.27 (m, 2 H), 7.24 (dd, *J* = 7.9, 4.2 Hz, 1 H), 2.59 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 140.2, 131.9, 131.6, 129.5, 128.40, 128.36, 128.2, 125.6, 123.6, 123.1, 93.4, 88.4, 20.8 ppm. The physical data were in full accordance with the literature value.[1]

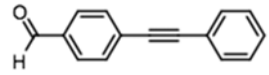


1-methyl-3-(phenylethynyl)benzene (3e). Following method 1, 63 mg (0.5 mmol) of 1-chloro-3-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3e** as a yellow oil (95 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.63–7.57 (m, 2H), 7.45–7.35 (m, 5 H), 7.30 (td, *J* = 7.6, 1.6 Hz, 1 H), 7.20 (d, *J* = 7.7 Hz, 1 H), 2.41 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 138.1, 132.2, 131.6, 129.2, 128.7, 128.5, 128.4, 128.3, 128.2, 123.4, 123.1, 89.7, 89.1, 21.3 ppm. The physical data were in full accordance with the literature value.[1]

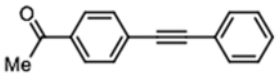


1-methyl-4-(phenylethynyl)benzene (3f). Following method 1, 63 mg (0.5 mmol) of 1-chloro-4-methylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3f** as a yellow solid (95

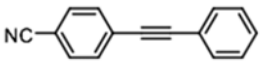
mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.58–7.53 (m, 2 H), 7.47 (dd, *J* = 8.1, 1.6 Hz, 2 H), 7.39–7.32 (m, 3 H), 7.19 (d, *J* = 7.8 Hz, 2 H), 2.40 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.4, 131.6, 131.5, 129.1, 128.3, 128.1, 123.5, 120.2, 89.6, 88.7, 21.5 ppm. The physical data were in full accordance with the literature value.[2]



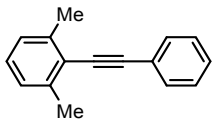
4-(phenylethynyl)benzaldehyde (3g). Following method 1, 70 mg (0.5 mmol) of 4-chlorobenzaldehyde and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3g** as a yellow solid (98 mg, 95%) using 6:94 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 10.04 (s, 1 H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 2 H), 7.61–7.50 (m, 2 H), 7.44–7.35 (m, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 192.18, 192.17, 136.2, 132.9, 132.5, 130.4, 130.3, 129.7, 129.2, 123.2, 94.2, 89.3 ppm. The physical data were in full accordance with the literature value.[1]



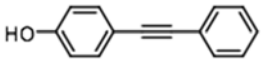
1-(4-(phenylethynyl)phenyl)ethan-1-one (3h). Following method 1, 77 mg (0.5 mmol) of 1-(4-chlorophenyl)ethan-1-one and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3h** as a yellow solid (100 mg, 91%) using 1:19 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.98–7.93 (m, 2 H), 7.62 (d, *J* = 8.3 Hz, 2 H), 7.57 (dd, *J* = 6.7, 3.0 Hz, 2 H), 7.40–7.34 (m, 3 H), 2.62 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 197.3, 136.2, 131.8, 131.7, 128.8, 128.5, 128.3, 128.2, 122.7, 92.7, 88.6, 26.6 ppm. The physical data were in full accordance with the literature value.[2]



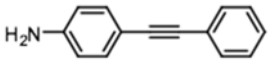
4-(phenylethynyl)benzonitrile (3i). Following method 1, 68.5 mg (0.5 mmol) of 4-chlorobenzonitrile and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3i** as a yellow solid (95 mg, 94%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.66 (d, *J* = 8.6 Hz, 2 H), 7.63 (d, *J* = 8.6 Hz, 2 H), 7.58–7.54 (m, 2 H), 7.40 (dd, *J* = 5.2, 2.0 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 132.08, 132.06, 131.8, 129.1, 128.5, 128.3, 122.2, 118.6, 111.5, 93.8, 87.7 ppm. The physical data were in full accordance with the literature value.[2]



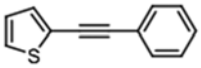
1,3-dimethyl-2-(phenylethynyl)benzene (3j). Following method 1, 70 mg (0.5 mmol) of 2-chloro-1,3-dimethylbenzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3j** as a colourless oil (100 mg, 97%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.59 (dd, *J* = 6.4, 1.9 Hz, 2 H), 7.43–7.31 (m, 3 H), 7.19–7.14 (m, 1 H), 7.11 (dd, *J* = 7.8, 2.3 Hz, 2 H), 2.57 (s, 6 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 140.3, 131.4, 128.4, 128.1, 127.8, 126.7, 123.9, 123.0, 97.9, 87.1, 21.2 ppm. The physical data were in full accordance with the literature value.[1]



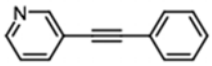
4-(phenylethynyl)phenol (3k). Following method 1, 64.3 mg (0.5 mmol) of 4-chlorophenol and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3k** as a yellow solid (80 mg, 82%) using 1:5 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54 (dd, J = 8.0, 1.6 Hz, 2 H), 7.46 (d, *J* = 8.8 Hz, 2 H), 7.39–7.32 (m, 3 H), 6.84 (d, *J* = 8.7 Hz, 2 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 155.8, 133.3, 131.5, 128.3, 128.0, 123.6, 115.6, 115.5, 89.3, 88.0 ppm. The physical data were in full accordance with the literature value.[2]



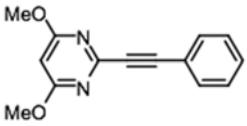
4-(phenylethynyl)aniline (3l). Following method 1, 63.8 mg (0.5 mmol) of 4-chloroaniline and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3l** as a yellow solid (81 mg, 84%) using 1:5 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (d, *J* = 6.8 Hz, 2 H), 7.42–7.30 (m, 5 H), 6.67 (d, *J* = 8.5 Hz, 2 H), 3.84 (s, 2 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 146.6, 133.0, 131.4, 128.3, 127.7, 123.9, 114.8, 112.7, 90.1, 87.3 ppm. The physical data were in full accordance with the literature value.[2]



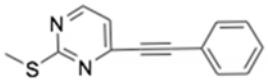
2-(phenylethynyl)thiophene (3m). Following method 1, 59.3 mg (0.5 mmol) of 2-chlorothiophene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3m** as a white oil (87 mg, 95%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.58–7.50 (m, 2 H), 7.37 (dd, *J* = 4.7, 3.0 Hz, 3 H), 7.34–7.29 (m, 2 H), 7.04 (ddd, *J* = 5.0, 3.2, 1.9 Hz, 1 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 131.9, 131.4, 128.4, 128.4, 127.3, 127.1, 123.3, 122.9, 93.0, 82.6 ppm. The physical data were in full accordance with the literature value.[1]



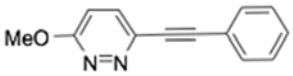
3-(phenylethynyl)pyridine (3n). Following method 1, 56.8 mg (0.5 mmol) of 3-chloropyridine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3n** as a white solid (88 mg, 98%) using 1:4 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.79 (s, 1 H), 8.57 (d, *J* = 4.0 Hz, 1 H), 7.83 (d, *J* = 7.9 Hz, 1 H), 7.61–7.52 (m, 2 H), 7.42–7.37 (m, 3 H), 7.30 (ddd, *J* = 7.9, 4.9, 0.9 Hz, 1 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 152.2, 148.5, 138.5, 128.8, 128.5, 123.1, 122.5, 92.7, 85.9 ppm. The physical data were in full accordance with the literature value.[3]



4,6-dimethoxy-2-(phenylethynyl)pyrimidine (3o). Following method 1, 87 mg (0.5 mmol) of 2-chloro-4,6-dimethoxypyrimidine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3o** as a yellow solid (118 mg, 98%) using 1:19 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.72–7.65 (m, 2 H), 7.44–7.35 (m, 3 H), 6.04 (s, 1 H), 4.01 (s, 6 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 171.1, 151.2, 132.6, 129.5, 128.4, 121.5, 90.0, 88.1, 86.5, 54.4 ppm. The physical data were in full accordance with the literature value.[1]

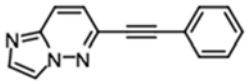


4-(methylthio)-2-(phenylethynyl)pyrimidine (3p). Following method 1, 80 mg (0.5 mmol) of 2-chloro-4-(methylthio)pyrimidine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3p** as a yellow oil (108 mg, 96%) using 1:25 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.50 (s, 1 H), 7.64–7.59 (m, 2 H), 7.45–7.36 (m, 3 H), 7.11 (d, *J* = 4.8 Hz, 1 H), 2.60 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 173.2, 157.0, 150.9, 132.4, 129.9, 128.5, 121.2, 118.5, 93.8, 86.8, 14.2 ppm. The physical data were in full accordance with the literature value.[1]

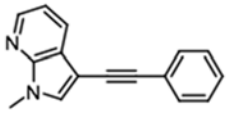


3-methoxy-6-(phenylethynyl)pyridazine (3q). Following method 1, 72.3 mg (0.5 mmol) of 3-chloro-6-methoxypyridazine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3q** as a yellow solid (100 mg, 95%) using 1:4 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.63–7.55 (m, 2 H), 7.50 (d, *J* = 9.1 Hz, 1 H), 7.36 (dd, *J* = 5.3, 2.0 Hz, 3 H),

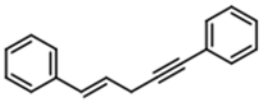
6.93 (d, *J* = 9.1 Hz, 1 H), 4.15 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 163.4, 143.7, 132.3, 132.0, 129.2, 128.4, 121.9, 116.6, 92.1, 85.7, 55.1 ppm. The physical data were in full accordance with the literature value.[4]



6-(phenylethynyl)imidazo[1,2-*b*]pyridazine (3r). Following method 1, 76.8 mg (0.5 mmol) of 6-chloroimidazo[1,2-b]pyridazine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3r** as a yellow solid (108 mg, 98%) using 2:1 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.99 (d, *J* = 9.8 Hz, 2 H), 7.83(s, 1 H), 7.63 (d, *J* = 8.1 Hz, 2 H), 7.42 (m, 3 H), 7.25 (d, *J* =9.3 Hz, 1 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 138.7, 137.8, 134.6, 132.1, 129.7, 128.5, 125.2, 121.2, 120.6, 117.0, 92.4, 84.8 ppm. The physical data were in full accordance with the literature value.[3]

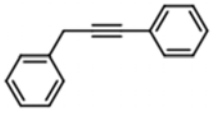


1-methyl-3-(phenylethynyl)-1*H*-pyrrolo[2,3-b]pyridine (3s). Following method 1, 83 mg (0.5 mmol) of 3-chloro-1-methyl-1*H*-pyrrolo[2,3-*b*]pyridine and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3s** as a dark yellow solid (105 mg, 90%) using 1:4 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.38 (dd, *J* = 4.7, 1.6 Hz, 1 H), 8.08 (dd, *J* = 7.8, 1.6 Hz, 1 H), 7.58–7.52 (m, 2 H), 7.43 (s, 1 H), 7.38–7.28 (m, 3 H), 7.14 (dd, *J* = 7.8, 4.7 Hz, 1 H), 3.88 (s, 3 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 147.3, 144.0, 132.3, 131.3, 128.4, 128.3, 127.8, 123.9, 121.4, 116.5, 95.7, 91.1, 82.3, 31.4 ppm. IR (film) ν_{max} 2210, 1537, 1455, 1360, 1297, 1144, 792, 771, 754, 694 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}N_2^+$ $(M+H)^+$, 233.1000, found: 233.1076.

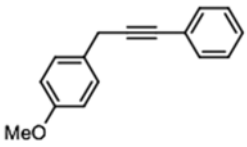


(Z)-pent-1-en-4-yne-1,5-diyldibenzene (3t). Following method 1, 76.3 mg (0.5 mmol) of (*E*)-(3-chloroprop-1-en-1-yl)benzene and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3t** as a yellow solid (103 mg, 95%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.54 (dd, *J* = 7.7, 2.0 Hz, 2 H), 7.48–7.44 (m, 2 H), 7.42–7.34 (m, 6 H), 6.79 (d, *J* = 15.7 Hz, 1 H), 6.32 (dt, *J* = 15.7, 5.7 Hz, 1 H), 3.43 (dd, *J* = 5.6, 1.9 Hz, 2 H) ppm. ^{13}C NMR (151

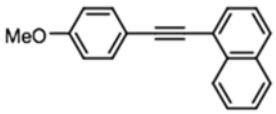
MHz, CDCl3) δ = 137.2, 131.7, 131.5, 128.6, 128.3, 127.9, 127.4, 126.3, 124.3, 123.7, 86.8, 83.0, 23.1 ppm. The physical data were in full accordance with the literature value.[5]



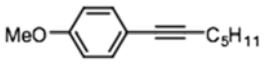
prop-1-yne-1,3-diyldibenzene (3u). Following method 1, 63.3 mg (0.5 mmol) of benzyl chloride and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded the **3u** as a colorless oil (93 mg, 97%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.55–7.51 (m, 1 H), 7.50–7.46 (m, 3 H), 7.41 (t, *J* = 7.5 Hz, 2 H), 7.38–7.29 (m, 5 H), 3.90 (s, 2 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 136.8, 131.7, 128.6, 128.3, 128.0, 127.9, 126.7, 123.8, 87.6, 82.8, 25.8 ppm. The physical data were in full accordance with the literature value.[6]



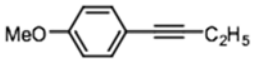
1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (3v). Following method 1, 78.3 mg (0.5 mmol) of 4-methoxybenzyl chloride and 87.6 mg (0.6 mmol) of phenylpropiolic acid afforded **3v** as a yellow oil (110 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.53–7.42 (m, 2 H), 7.36 (d, *J* = 8.7 Hz, 2 H), 7.32 (dd, *J* = 5.1, 2.0 Hz, 3 H), 6.92 (d, *J* = 8.6 Hz, 2 H), 3.83 (s, 3 H), 3.81 (s, 2 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 158.45, 131.67, 128.97, 128.83, 128.26, 127.80, 123.81, 114.02, 88.06, 82.47, 55.33, 24.93 ppm. The physical data were in full accordance with the literature value.[6]



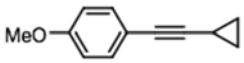
1-((4-methoxyphenyl)ethynyl)naphthalene (4b). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 117.7 mg (0.6 mmol) of 3-(naphthalen-1-yl)propiolic acid afforded **4b** as a yellow solid (127 mg, 98%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.07 (s, 1 H), 7.83 (dd, *J* = 9.0, 5.4 Hz, 3 H), 7.65–7.42 (m, 5 H), 6.93 (d, *J* = 8.6 Hz, 2 H), 3.84 (s, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 159.7, 133.2, 133.1, 132.7, 131.2, 128.5, 128.0, 127.80, 127.77, 126.5, 121.0, 115.5, 114.1, 89.9, 88.6, 55.3 ppm. The physical data were in full accordance with the literature value.[7]



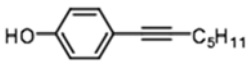
1-(hept-1-yn-1-yl)-4-methoxybenzene (4c). Following general method I, 71.5 mg (0.5 mmol) of 4-chloroanisole and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4c** as a yellow oil (100 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.34 (d, *J* = 8.8 Hz, 2 H), 6.81 (d, *J* = 8.9 Hz, 2 H), 2.39 (t, *J* = 7.1 Hz, 3 H), 1.65–1.52 (m, 2 H), 1.49–1.30 (m, 4 H), 0.93 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 159.0, 132.8, 116.3, 113.8, 88.8, 80.2, 55.2, 31.2, 28.6, 22.3, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.[8]



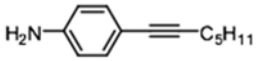
1-(but-1-yn-1-yl)-4-methoxybenzene (4d). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 58.9 mg (0.6 mmol) of pent-2-ynoic acid afforded **4d** as a yellow oil (80 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.34 (d, *J* = 8.7 Hz, 2 H), 6.81 (d, *J* = 8.7 Hz, 2 H), 3.79 (s, 3 H), 2.41 (q, *J* = 7.5 Hz, 2 H), 1.23 (t, *J* = 7.5 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 159.0, 132.8, 116.2, 113.8, 90.0, 79.6, 55.2, 14.0, 13.1 ppm. The physical data were in full accordance with the literature value.[9]



1-(cyclopropylethynyl)-4-methoxybenzene (4e). Following method 1, 71.5 mg (0.5 mmol) of 4-chloroanisole and 66.1 mg (0.6 mmol) of 3-cyclopropylpropiolic acid afforded **4e** as a yellow oil (85 mg, 99%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.32 (d, *J* = 8.8 Hz, 2 H), 6.80 (d, *J* = 8.9 Hz, 2 H), 3.79 (s, 3 H), 1.43 (tt, *J* = 8.2, 5.1 Hz, 1 H), 0.88–0.81 (m, 2 H), 0.81–0.73 (m, 2 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 158.9, 132.8, 115.9, 113.6, 91.6, 75.4, 55.1, 8.4 ppm. The physical data were in full accordance with the literature value.[10]

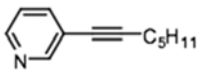


4-(hept-1-yn-1-yl)phenol (4f). Following method 1, 64.3 mg (0.5 mmol) of 4-chlorophenol and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4f** as a yellow oil (83 mg, 89%) using 1:9 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.20 (d, *J* = 8.6 Hz, 2 H), 6.67 (dd, *J* = 8.8, 7.5 Hz, 3 H), 2.29 (s, 2 H), 1.50 (q, *J* = 7.3 Hz, 2 H), 1.39–1.17 (m, 4 H), 0.84 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 155.0, 133.1, 129.5, 116.7, 115.4, 88.9, 80.2, 31.2, 28.6, 22.3, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.[11]

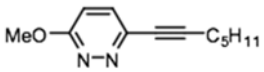


4-(hept-1-yn-1-yl)phenol (4g). Following method 1, 63.8 mg (0.5 mmol) of 4-chloroaniline and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4g** as a yellow oil (80 mg, 86%) using 1:9 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.20 (d, *J* = 8.5 Hz, 2 H), 6.57 (d, *J* = 8.5 Hz,2 H), 3.71 (s, 2 H), 2.38 (t, *J* = 7.1 Hz, 2 H), 1.65–1.52 (m, 2 H), 1.48–1.16 (m, 4 H), 0.93 (t, *J* = 7.2 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 144.9, 131.7, 113.7, 112.7, 86.8, 79.7, 30.1, 27.7, 21.2, 18.4, 13.0 ppm. The physical data were in full accordance with the literature value.[12]

2-(hept-1-yn-1-yl)thiophene (4h). Following method 1, 59.3 mg (0.5 mmol) of 2-chlorothiophene and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4h** as a yellow oil (80 mg, 89%) using petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.18–7.15 (m, 1 H), 7.12 (dd, *J* = 3.6, 1.1 Hz, 1 H), 6.94 (dd, *J* = 5.2, 3.6 Hz, 1 H), 2.43 (t, *J* = 7.2 Hz, 2 H), 1.61 (q, *J* = 7.3 Hz, 2 H), 1.50–1.20 (m, 4 H), 0.94 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 130.8, 126.7, 125.8, 124.3, 94.6, 73.7, 31.2, 28.3, 22.2, 19.7, 14.0 ppm. The physical data were in full accordance with the literature value.[8]

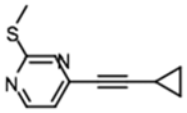


3-(hept-1-yn-1-yl)pyridine (4i). Following method 1, 56.8 mg (0.5 mmol) of 3-chloropyridine and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4i** as a yellow oil (78 mg, 90%) using 1:9 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.61 (s, 1 H), 8.50–8.40 (m, 1 H), 7.65 (d, *J* = 7.9 Hz, 1 H), 7.19 (dd, *J* = 7.9, 4.9 Hz, 2 H), 2.41 (t, *J* = 7.1 Hz, 2 H), 1.60 (q, *J* = 7.3 Hz, 2 H), 1.50–1.28 (m, 4 H), 0.91 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 152.3, 147.9, 138.4, 122.8, 121.2, 94.1, 31.1, 28.3, 22.2, 19.4, 14.0 ppm. The physical data were in full accordance with the literature value.[8]

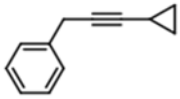


3-(hept-1-yn-1-yl)-6-methoxypyridazine (4j). Following method 1, 72.3 mg (0.5 mmol) of 3-chloro-6-methoxypyridazine and 84.2 mg (0.6 mmol) of oct-2-ynoic acid afforded **4j** as a brown oil (98 mg, 96%) using 1:9 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.32 (d, *J* = 9.1 Hz, 1 H), 6.84 (d, *J* = 9.1 Hz, 1 H), 4.08 (s, 3 H), 2.40 (t, *J* = 7.1 Hz, 2

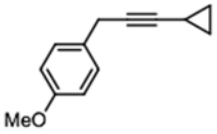
H), 1.59 (pentet, J = 7.2 Hz, 2 H), 1.45–1.33 (m, 2 H), 1.34–1.23 (m, 2 H), 0.87 (t, J = 7.2 Hz, 3 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 163.2, 144.0, 132.2, 130.7, 120.0, 116.5, 94.1, 54.9, 31.1, 27.9, 22.2, 19.3, 14.0 ppm. IR (film) ν_{max} 2955, 1593, 1542, 1463, 1411, 1324, 1289, 1267, 1012, 843, 797, 736 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{17}N_2O^+$ $(M+H)^+$, 205.1263, found: 205.1338.



4-(cyclopropylethynyl)-2-(methylthio)pyridine (4k). Following method 1, 80 mg (0.5 mmol) of 2-chloro-4-(methylthio)pyrimidine and 66.1 mg (0.6 mmol) of 3-cyclopropylpropiolic acid afforded **4k** as a yellow oil (90 mg, 95%) using 1:9 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.38 (d, J = 5.1 Hz, 1 H), 6.89 (d, J = 5.1 Hz, 1 H), 2.53 (s, 3 H), 1.53–1.39 (m, 1 H), 0.93 (ddq, J = 10.1, 4.9, 2.3 Hz, 4 H) ppm. ^{13}C NMR (151 MHz, $CDCl_3$) δ = 172.6, 156.4, 151.0, 118.0, 99.7, 74.0, 13.8, 8.9 ppm. IR (film) ν_{max} 2224, 1554, 1524, 1416, 1339, 1322, 1202, 864 cm^{-1}. HRMS (ESI) calcd for $C_{10}H_{11}N_2S^+$ $(M+H)^+$, 191.0565, found: 191.0638.

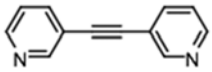


(3-cyclopropylprop-2-yn-1-yl)benzene (4l). Following method 1, 63.3 mg (0.5 mmol) of benzyl chloride and 66.1 mg (0.6 mmol) of 3-cyclopropylpropiolic acid afforded **4l** as a yellow oil (77 mg, 99%) using 3:97 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.42–7.30 (m, 4 H), 7.31–7.12 (m, 1 H), 3.61 (dd, J = 2.0, 0.8 Hz, 2 H), 1.40–1.23 (m, 1 H), 0.87–0.76 (m, 2 H), 0.76–0.66 (m, 2 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 137.5, 128.4, 127.9, 126.4, 85.6, 73.0, 25.2, 8.1 ppm. The physical data were in full accordance with the literature value.[6]

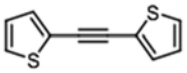


1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (4m). Following method 1, 78.3 mg (0.5 mmol) of 4-methoxybenzyl chloride and 66.1 mg (0.6 mmol) of 3-cyclopropylpropiolic acid afforded **4m** as a yellow oil (91 mg, 98%) using 1:19 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.26 (d, J = 8.8 Hz, 2 H), 6.88 (d, J = 8.7 Hz, 2 H), 3.82 (s, 3 H), 3.56–3.43 (m, 2 H), 1.35–1.21 (m, 1 H), 0.83–0.73 (m, 2 H), 0.71–0.59 (m, 2 H) ppm. ^{13}C NMR

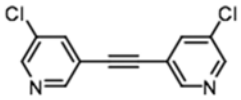
(151 MHz, CDCl3) δ = 158.2, 129.5, 128.8, 113.8, 85.3, 73.4, 55.3, 24.3, 8.0 ppm. IR (film) ν_{max} 1510, 1246, 1174, 1033, 812 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{15}O^+$ $(M+H)^+$, 187.1045, found: 187.1075.



1,2-di(pyridin-3-yl)ethyne (6a). Following method 2, 113.6 mg (1.0 mmol) of 3-chloropyridine and 28.5 mg (0.4 mmol) of propiolic acid afforded **6a** as a yellow oil (38 mg, 53%) using 2:1 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.56 (d, *J* = 7.6 Hz, 2 H), 7.32 (ddd, *J* = 8.4, 7.4, 1.7 Hz, 2 H), 6.96 (td, *J* = 7.5, 1.1 Hz, 2 H), 6.92 (d, J = 8.3 Hz, 2 H), 3.95 (s, 6 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 159.9, 133.6, 129.6, 120.4, 112.8, 110.7, 89.8, 55.9 ppm. The physical data were in full accordance with the literature value.[13]



1,2-di(thiophen-2-yl)ethyne (6b). Following method 2, 118.0 mg (1.0 mmol) of 2-chlorothiophene and 28.5 mg (0.4 mmol) of propiolic acid afforded **6b** as a yellow oil (45 mg, 58%) using 2:1 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.56 (d, *J* = 7.6 Hz, 2 H), 7.32 (ddd, J = 8.4, 7.4, 1.7 Hz, 2 H), 6.96 (td, *J* = 7.5, 1.1 Hz, 2 H), 6.92 (d, J = 8.3 Hz, 2 H), 3.95 (s, 6 H) ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 159.9, 133.6, 129.6, 120.4, 112.8, 110.7, 89.8, 55.9 ppm. The physical data were in full accordance with the literature value.[9]



1,2-bis(5-chloropyridin-3-yl)ethyne (6c). Following method 2, 148 mg (1.0 mmol) of 2,6-dichloropyridine and 28.5 mg (0.4 mmol) of propiolic acid afforded **6c** as a white solid (24 mg, 24%) using 1:19 EtOAc:petroleum ether as the column eluent. ^{1}H NMR (600 MHz, $CDCl_3$) δ = 8.65 (s, 1 H), 8.57 (s, 1 H), 7.84 – 7.83 (m, 1 H).ppm. ^{13}C NMR (151 MHz, CDCl3) δ = 149.9, 148.5, 138.5, 132.0, 120.5, 89.0ppm. IR (film) ν_{max} 1737, 1374, 1241, 1046, 737 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_7Cl_2N_2^+$ $(M+H)^+$, 248.9981, found: 248.9977.

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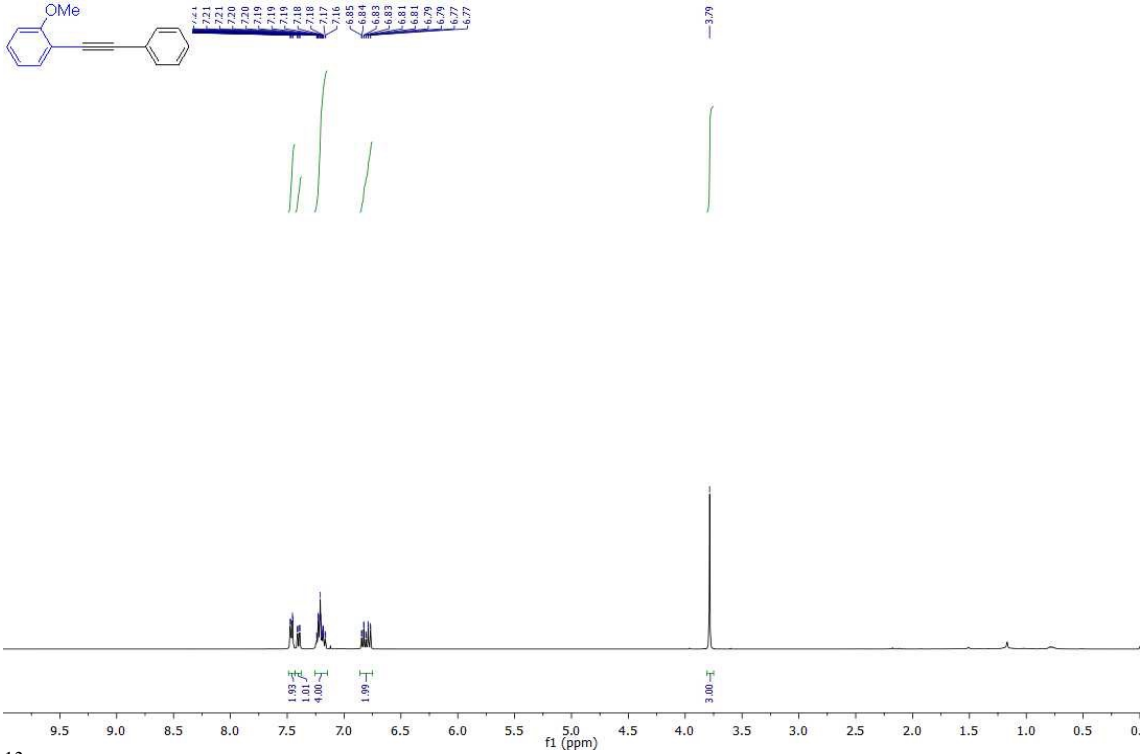
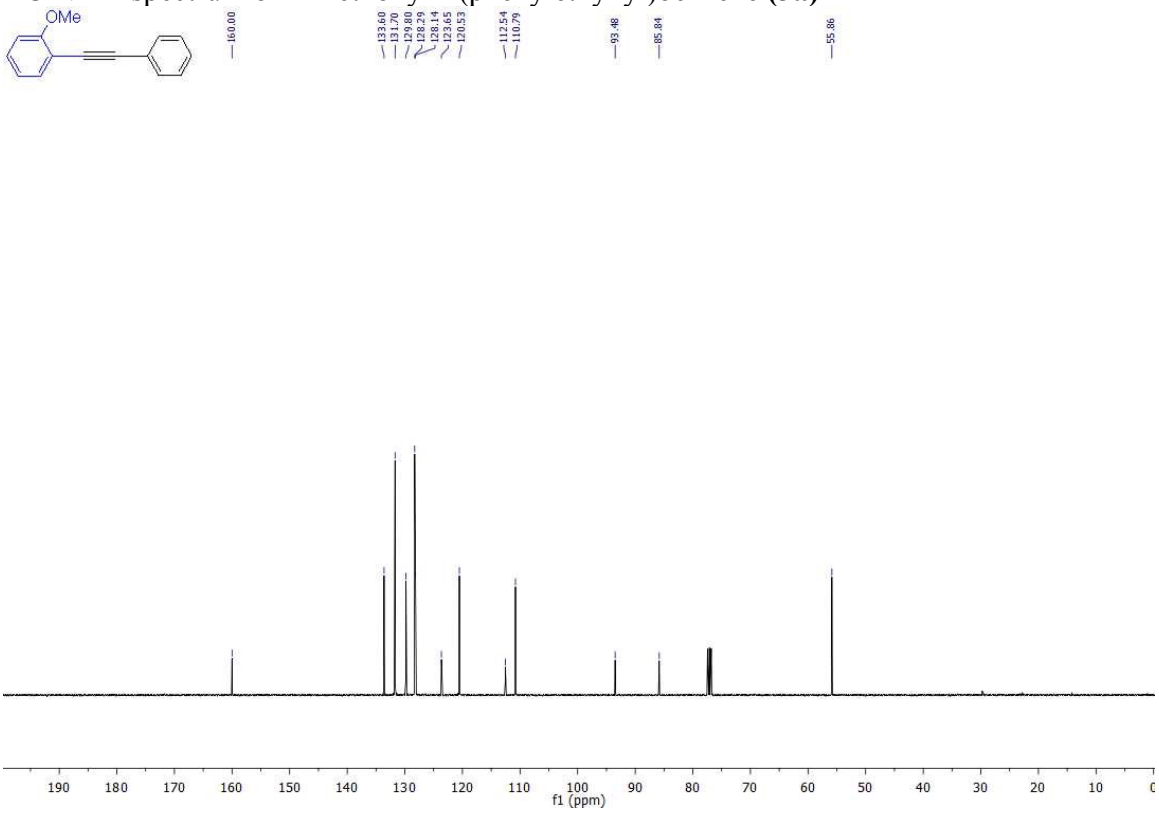
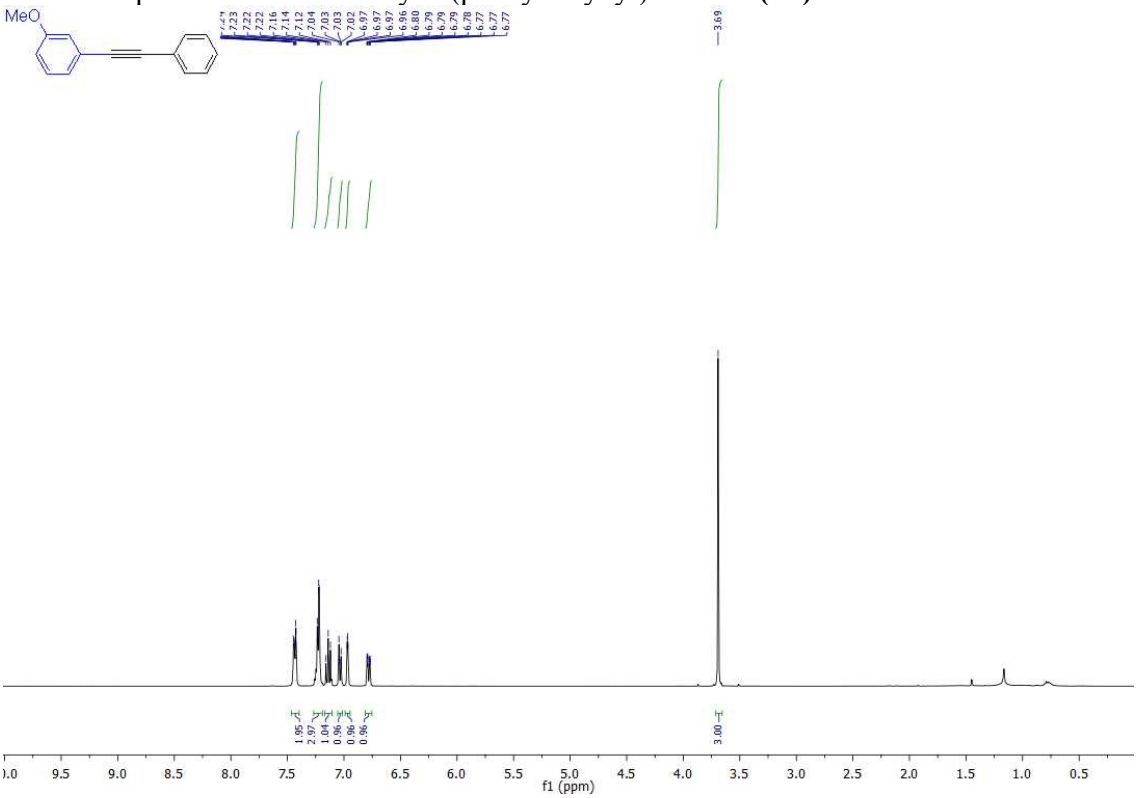
^{1}H NMR spectrum of 1-methoxy-2-(phenylethynyl)benzene **(3a)**



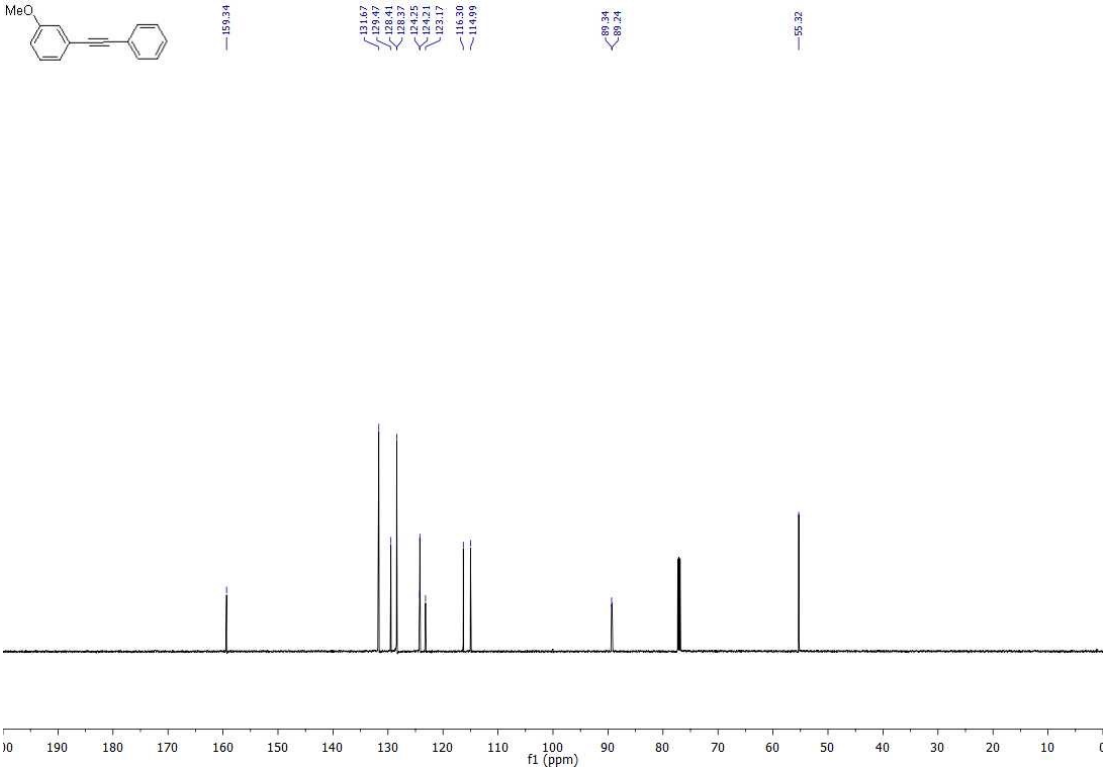
^{13}C NMR spectrum of 1-methoxy-2-(phenylethynyl)benzene **(3a)**



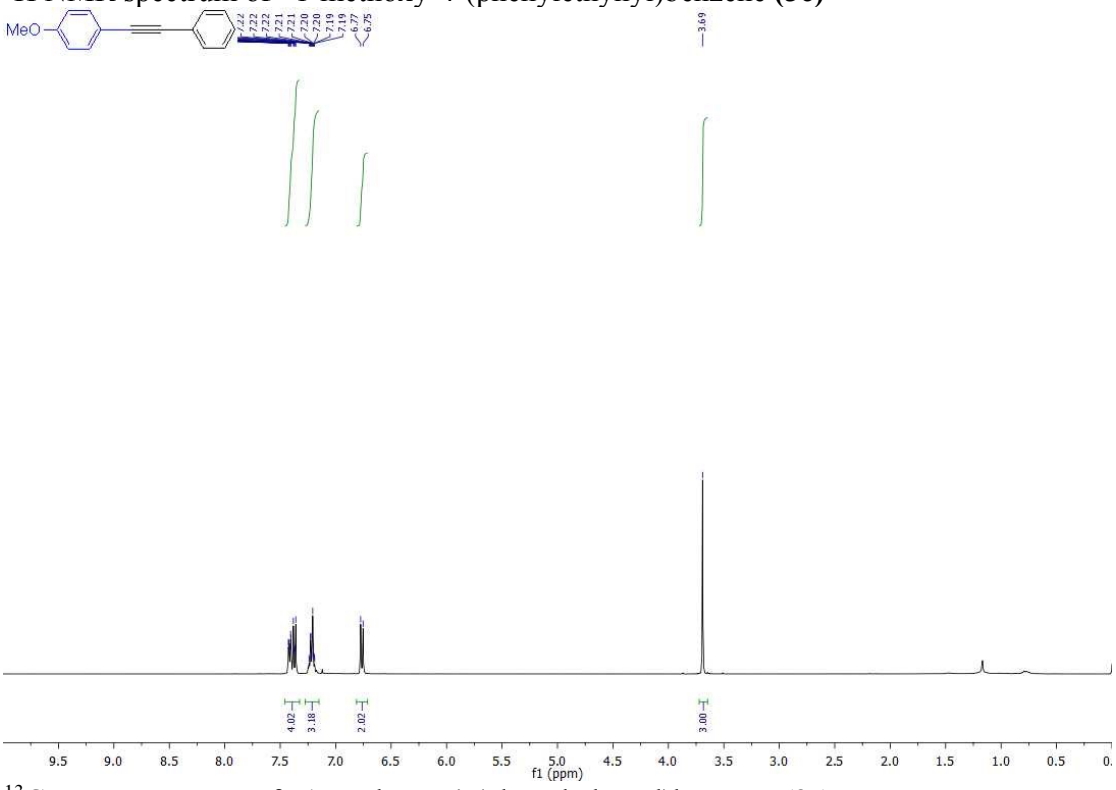
^{1}H NMR spectrum of 1-methoxy-3-(phenylethynyl)benzene **(3b)**



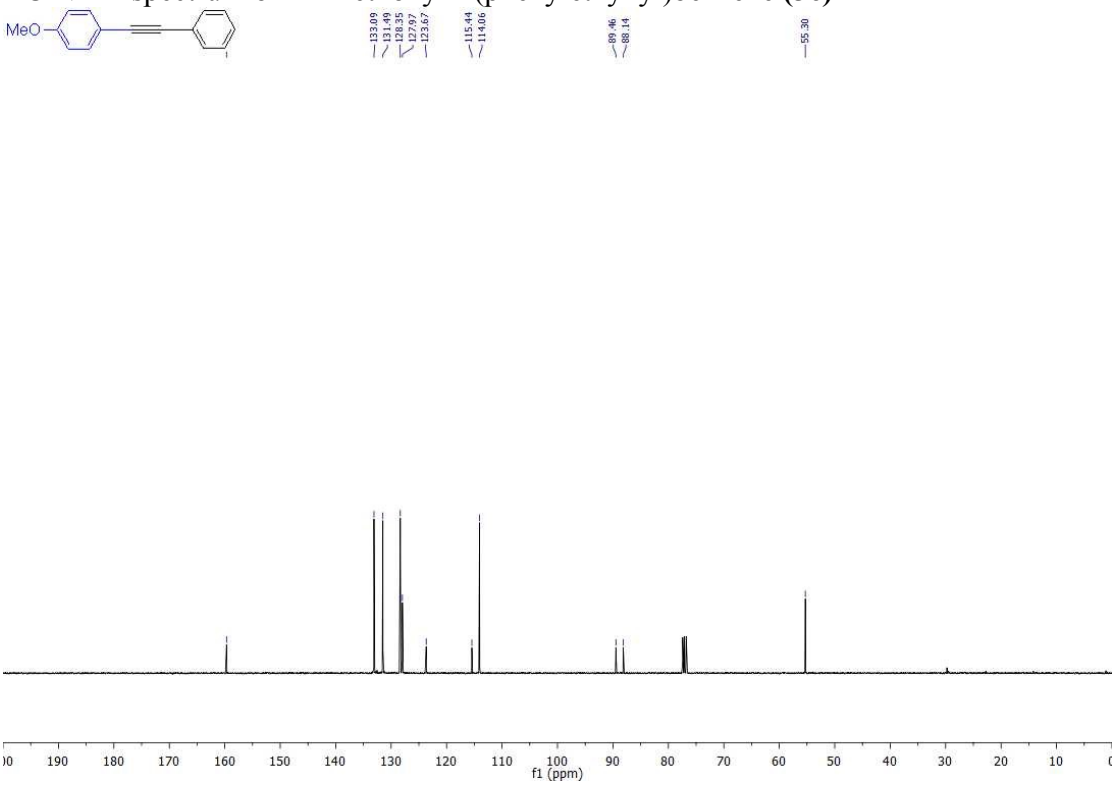
^{13}C NMR spectrum of 1-methoxy-3-(phenylethynyl)benzene **(3b)**



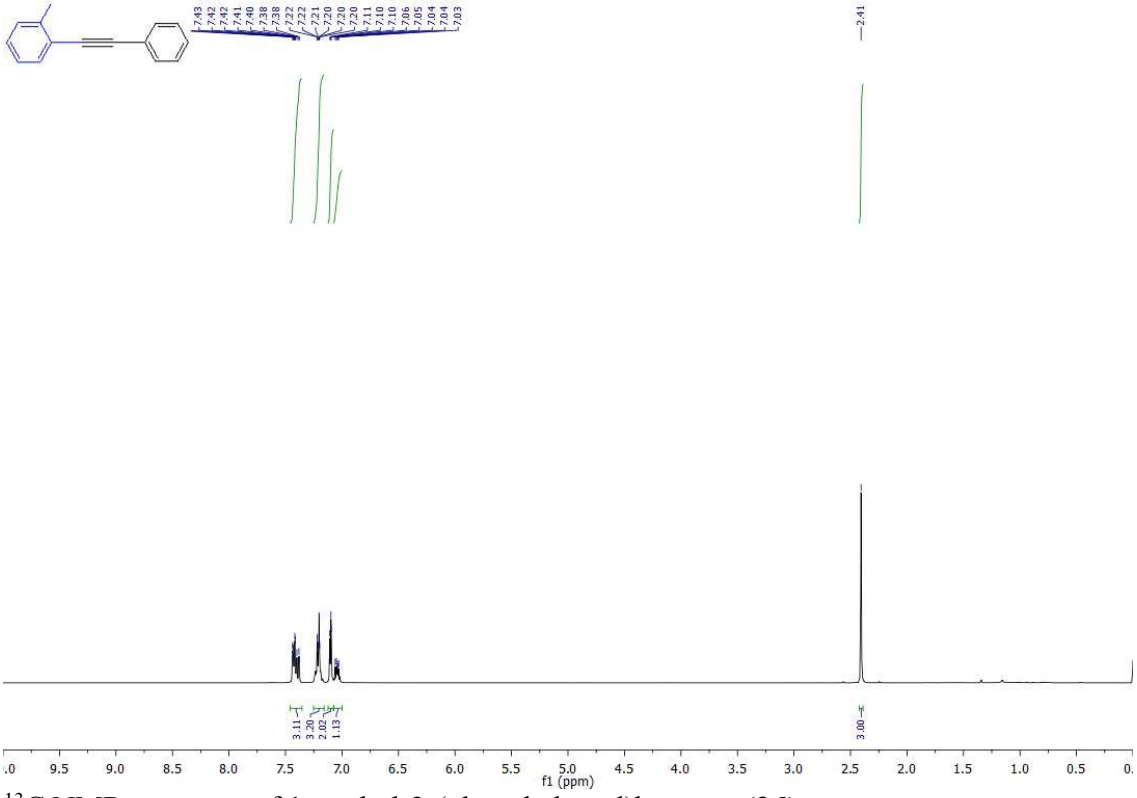
^{1}H NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3c)**



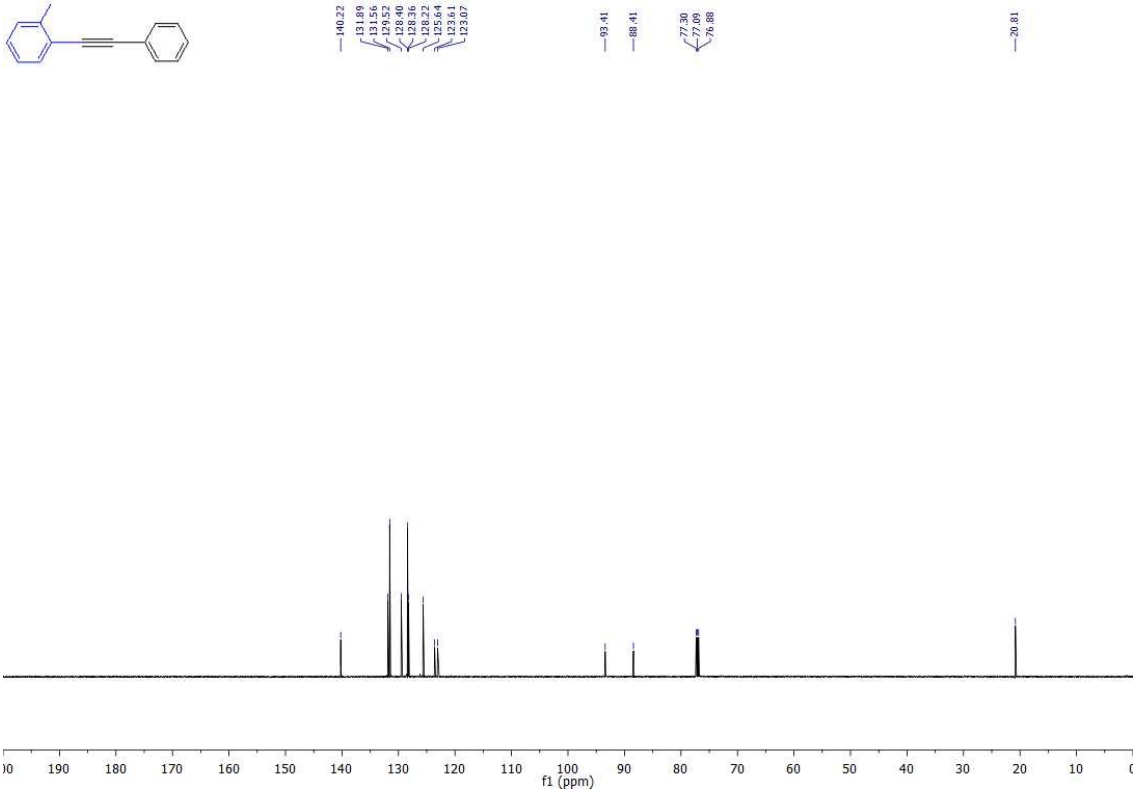
^{13}C NMR spectrum of 1-methoxy-4-(phenylethynyl)benzene **(3c)**



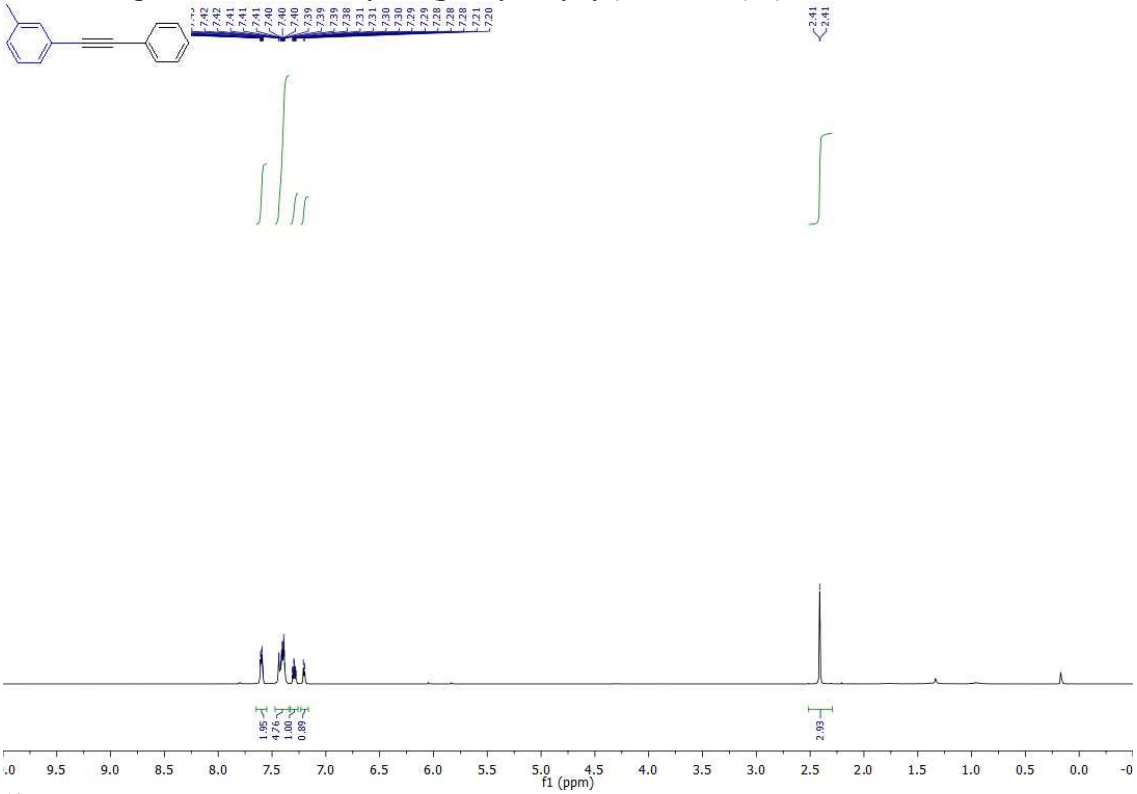
^{1}H NMR spectrum of 1-methyl-2-(phenylethynyl)benzene **(3d)**



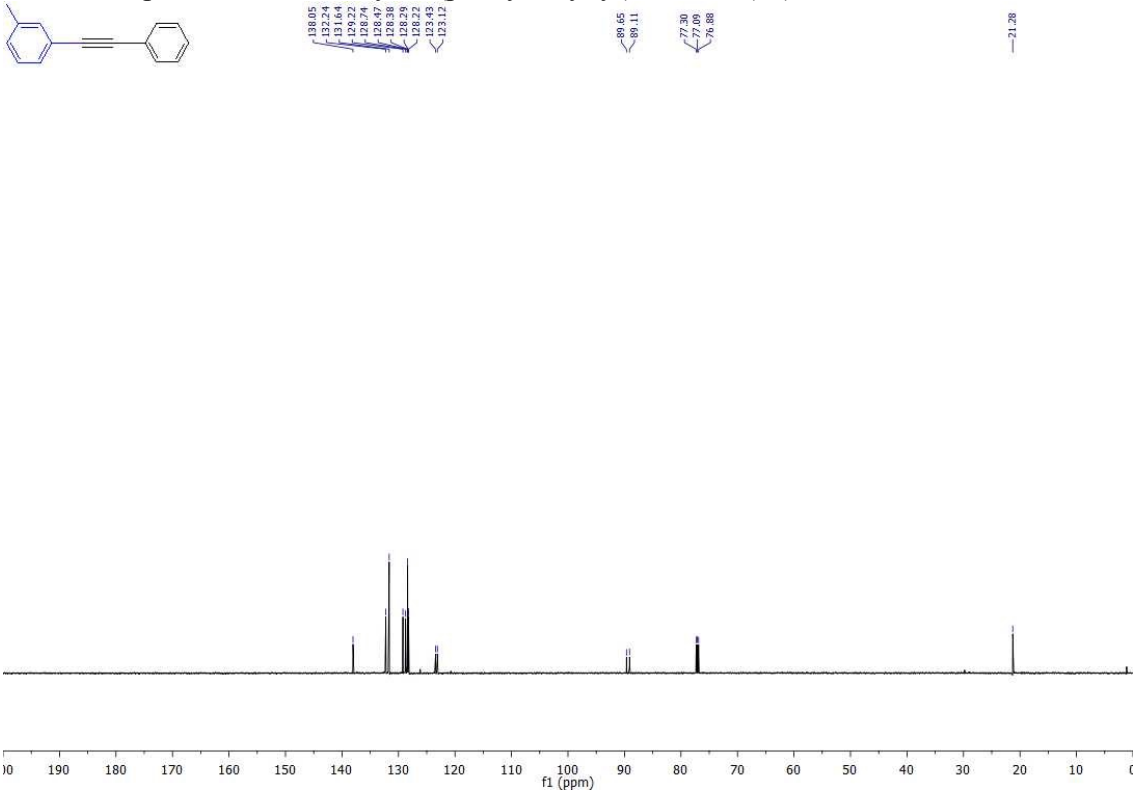
^{13}C NMR spectrum of 1-methyl-2-(phenylethynyl)benzene **(3d)**



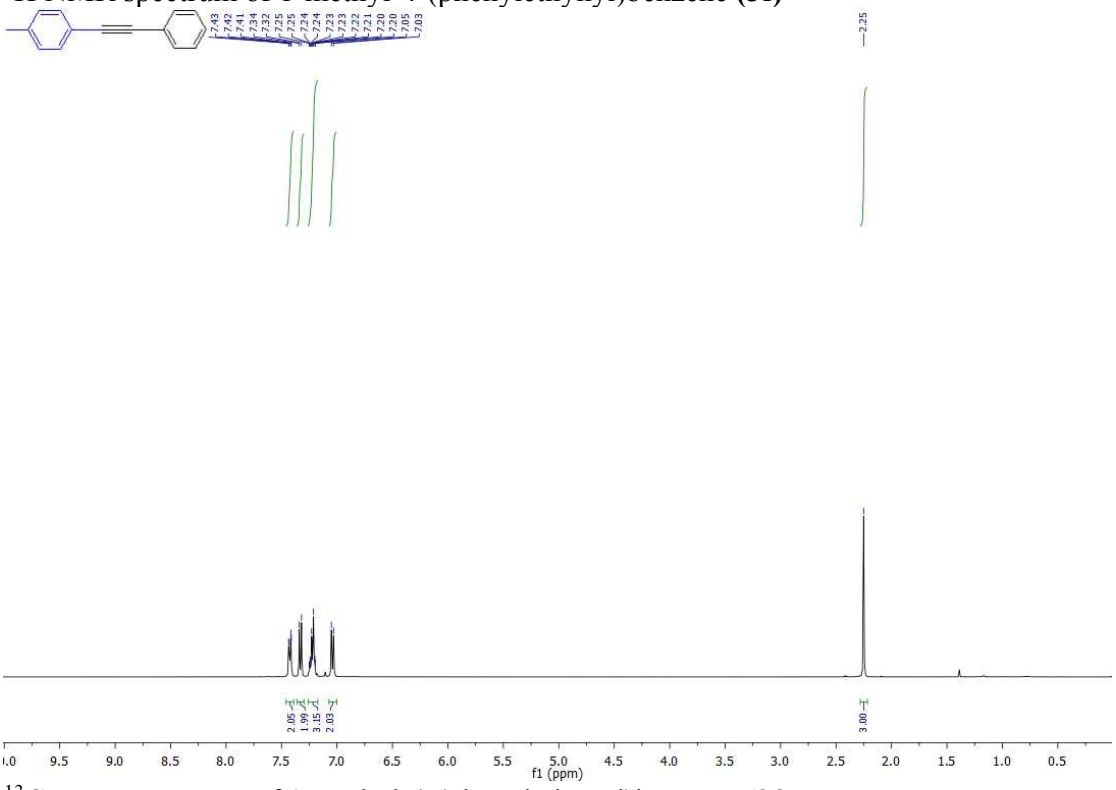
^{1}H NMR spectrum of 1-methyl-3-(phenylethynyl)benzene (**3e**)



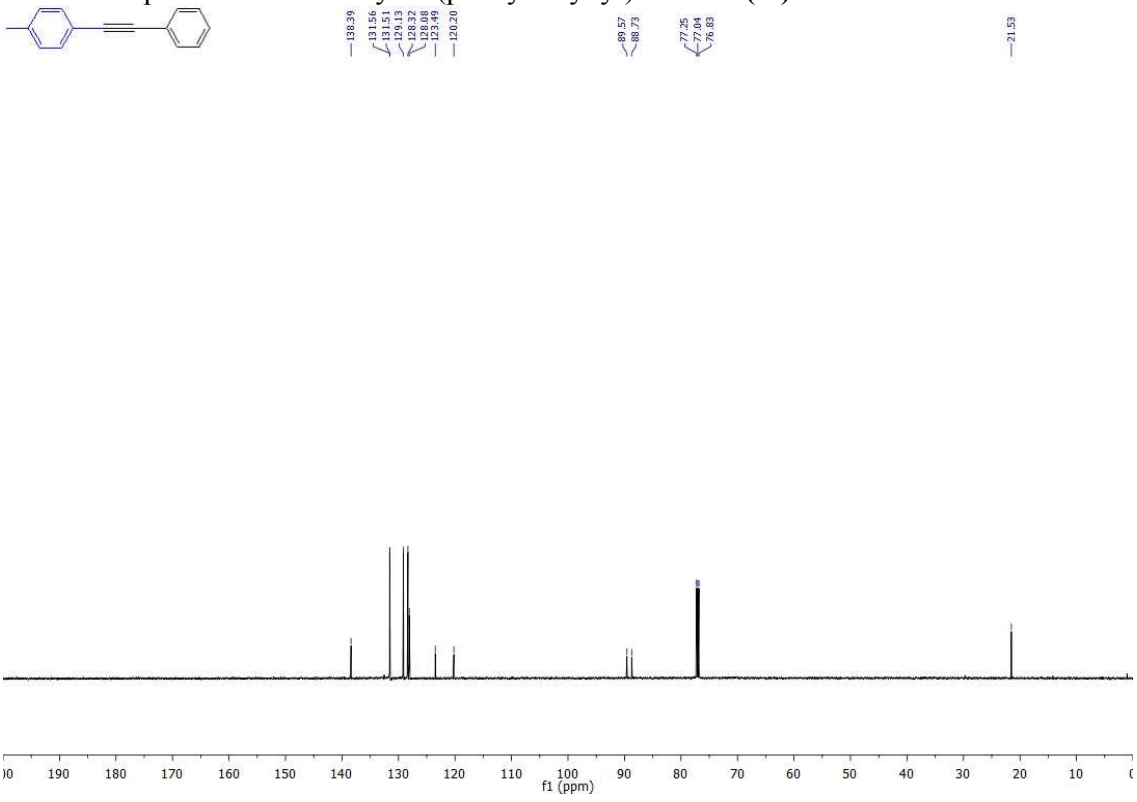
^{13}C NMR spectrum of 1-methyl-3-(phenylethynyl)benzene (**3e**)



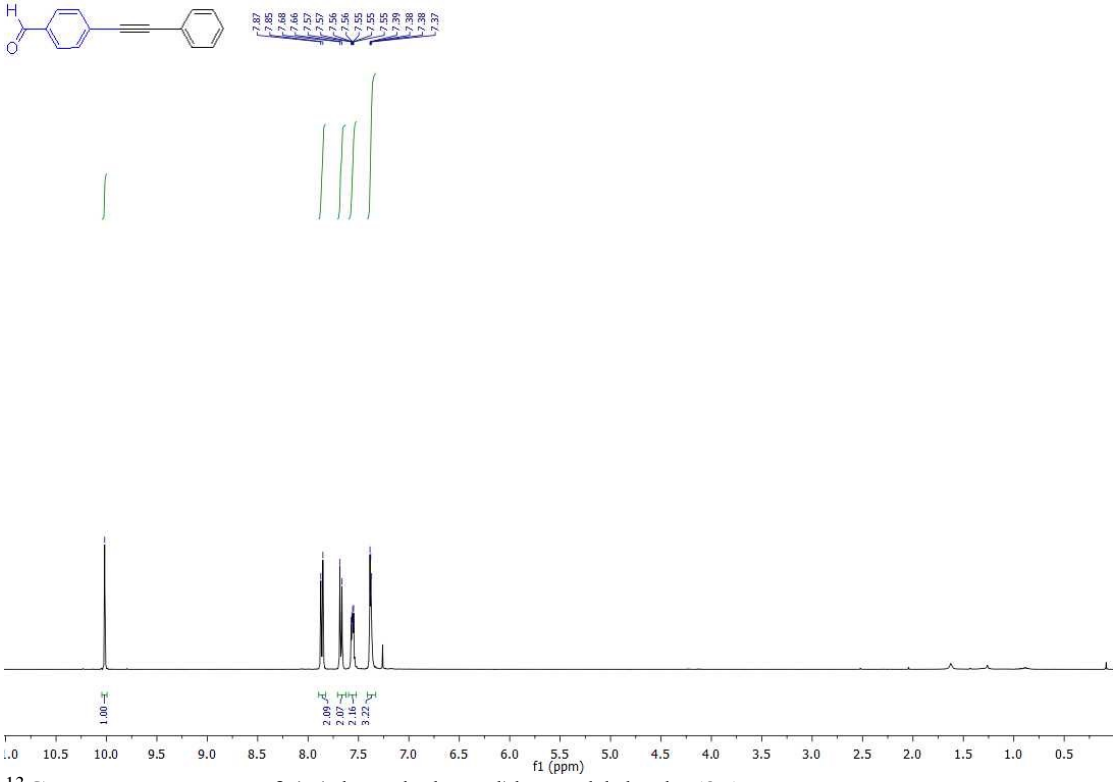
^{1}H NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(3f)**



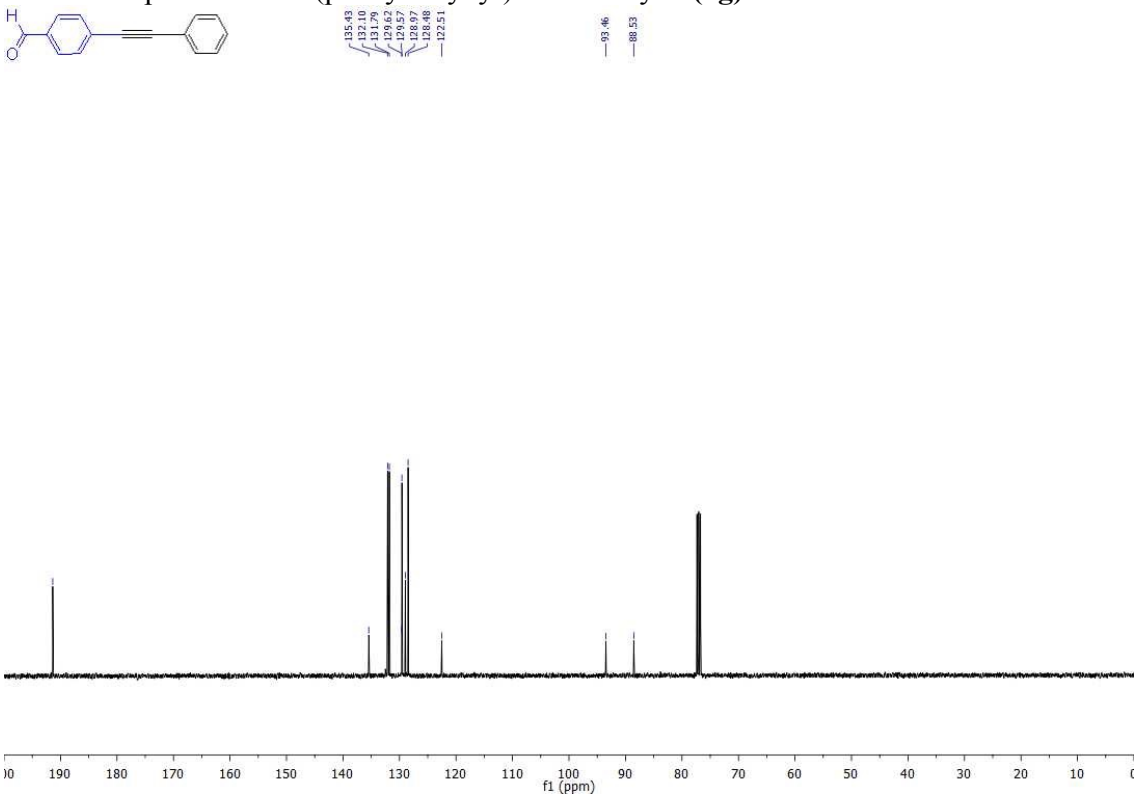
^{13}C NMR spectrum of 1-methyl-4-(phenylethynyl)benzene **(3f)**



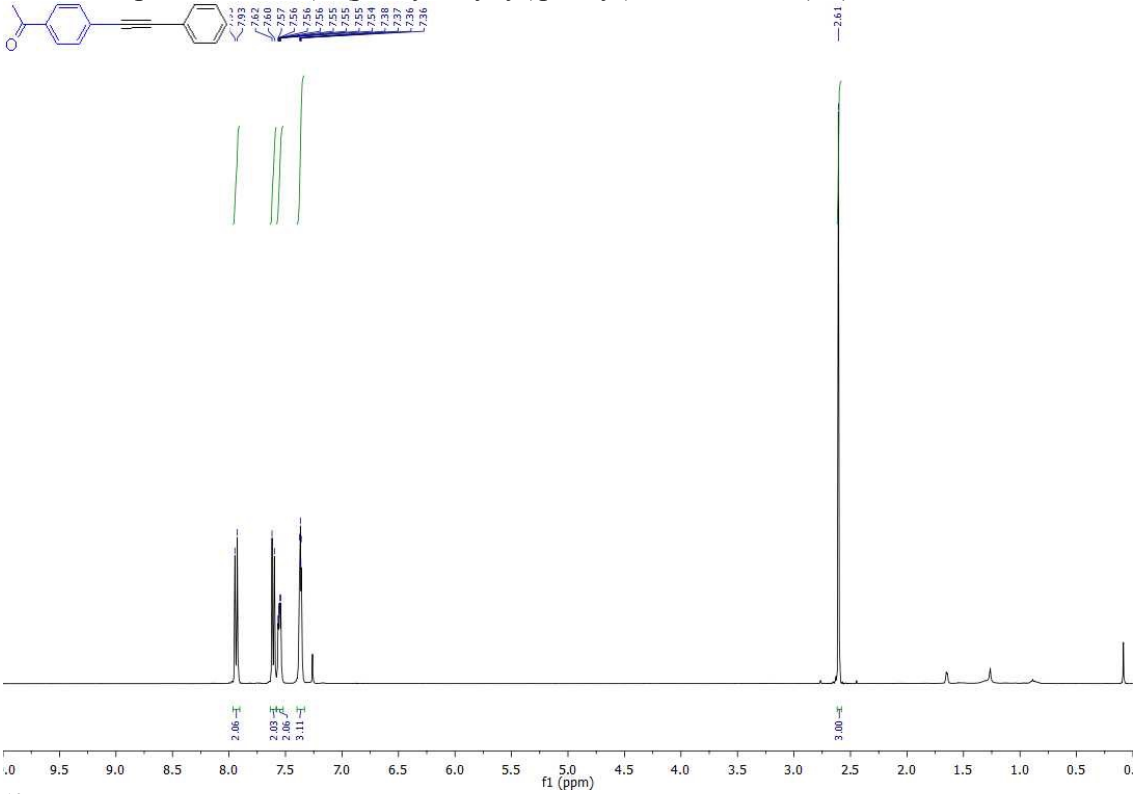
^{1}H NMR spectrum of 4-(phenylethynyl)benzaldehyde **(3g)**



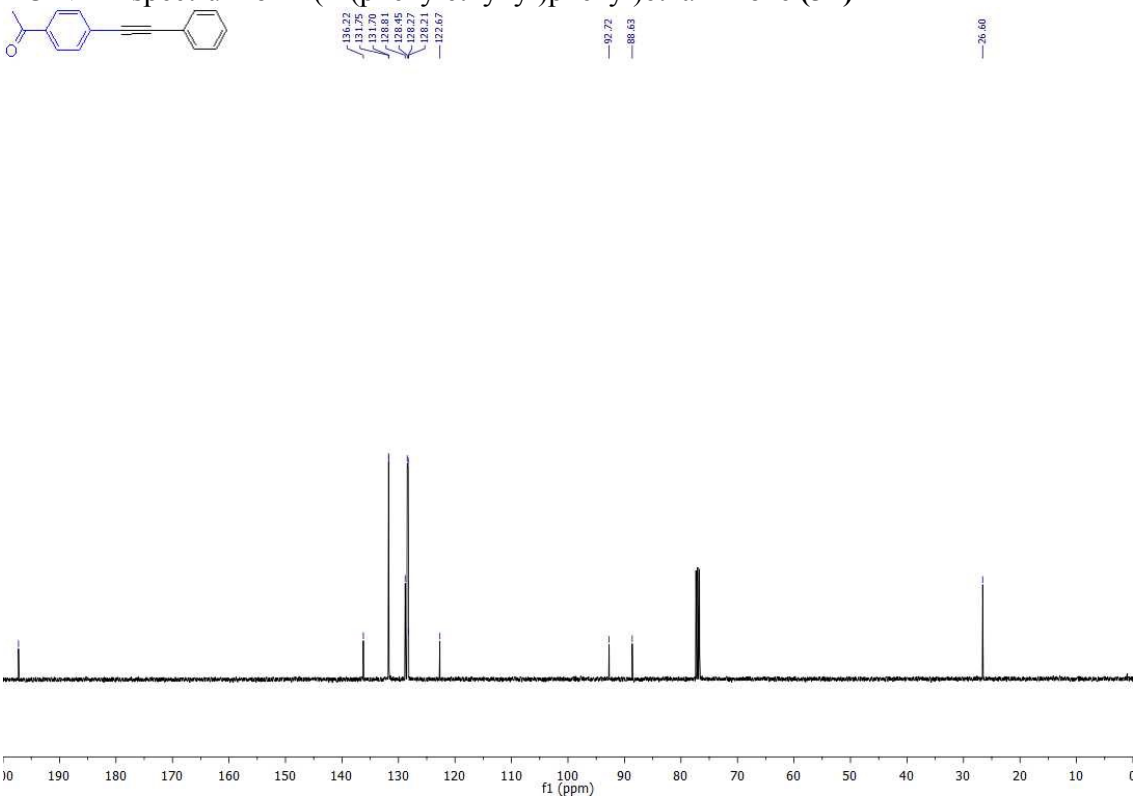
^{13}C NMR spectrum of 4-(phenylethynyl)benzaldehyde **(3g)**



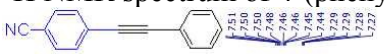
^{1}H NMR spectrum of 1-(4-(phenylethynyl)phenyl)ethan-1-one **(3h)**

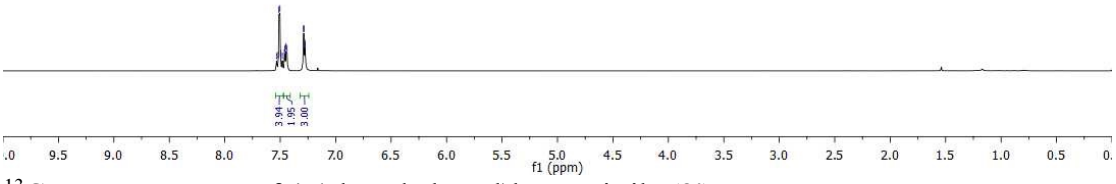


^{13}C NMR spectrum of 1-(4-(phenylethynyl)phenyl)ethan-1-one **(3h)**

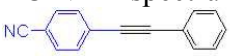


^{1}H NMR spectrum of 4-(phenylethynyl)benzonitrile **(3i)**

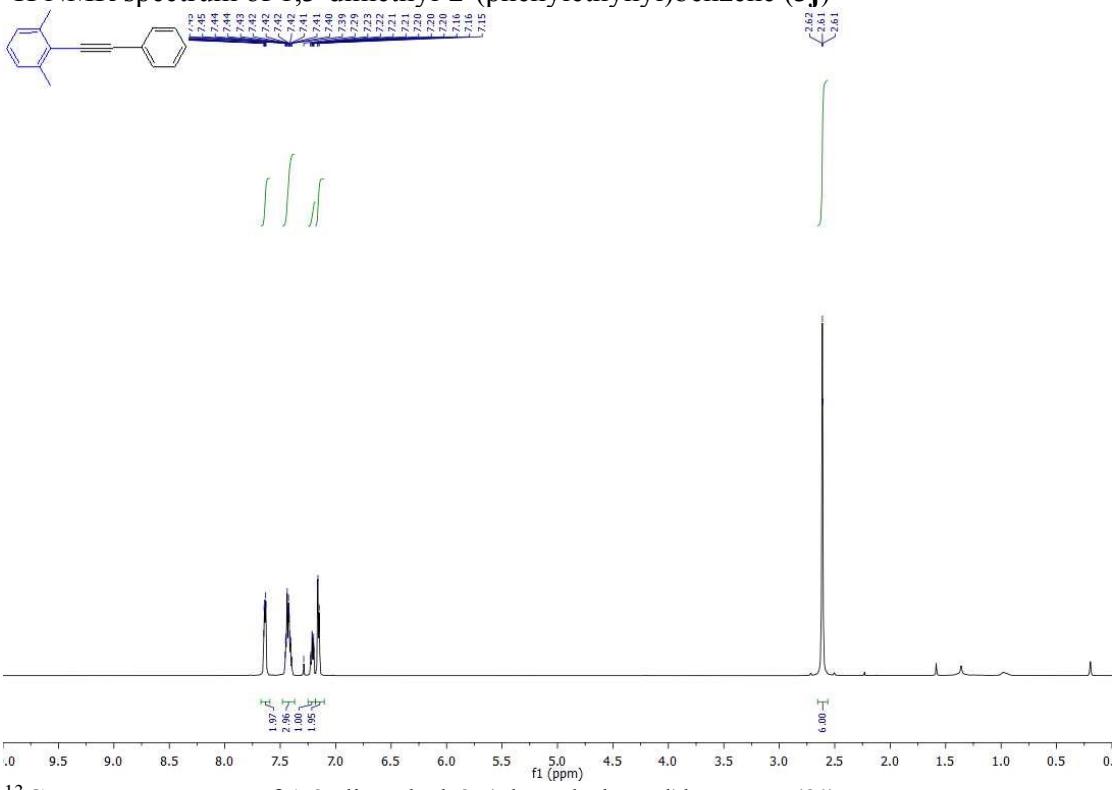




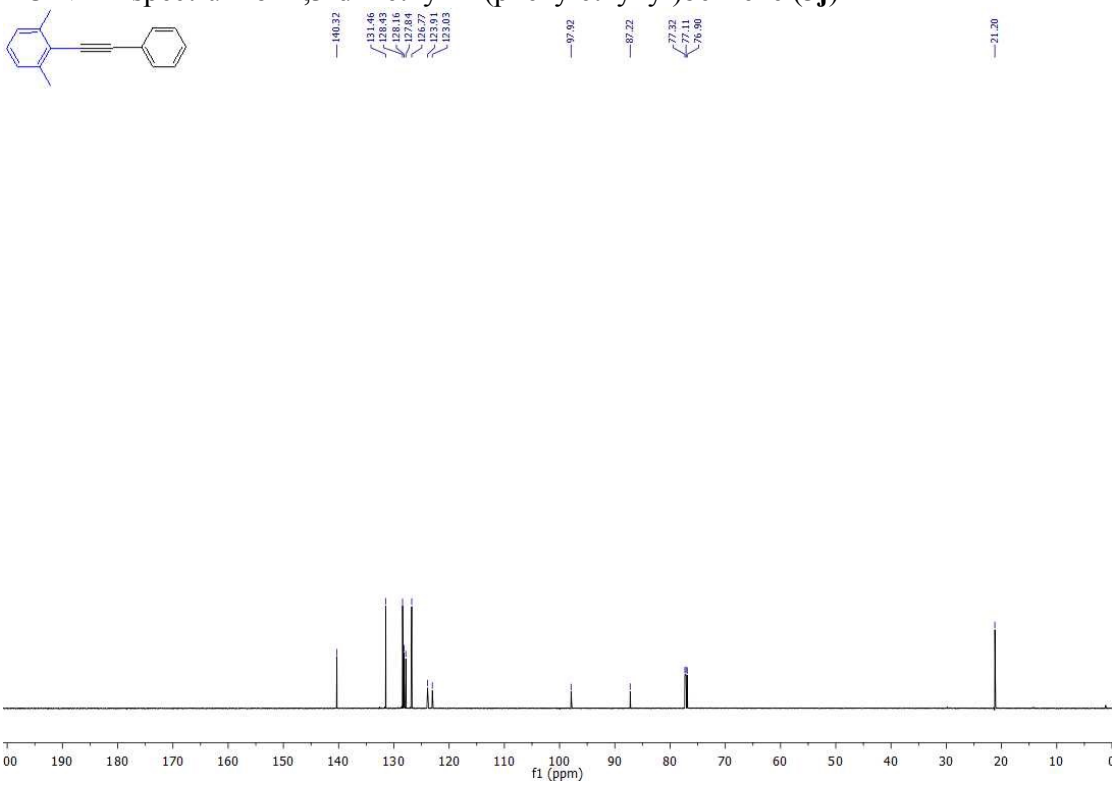
^{13}C NMR spectrum of 4-(phenylethynyl)benzonitrile **(3i)**



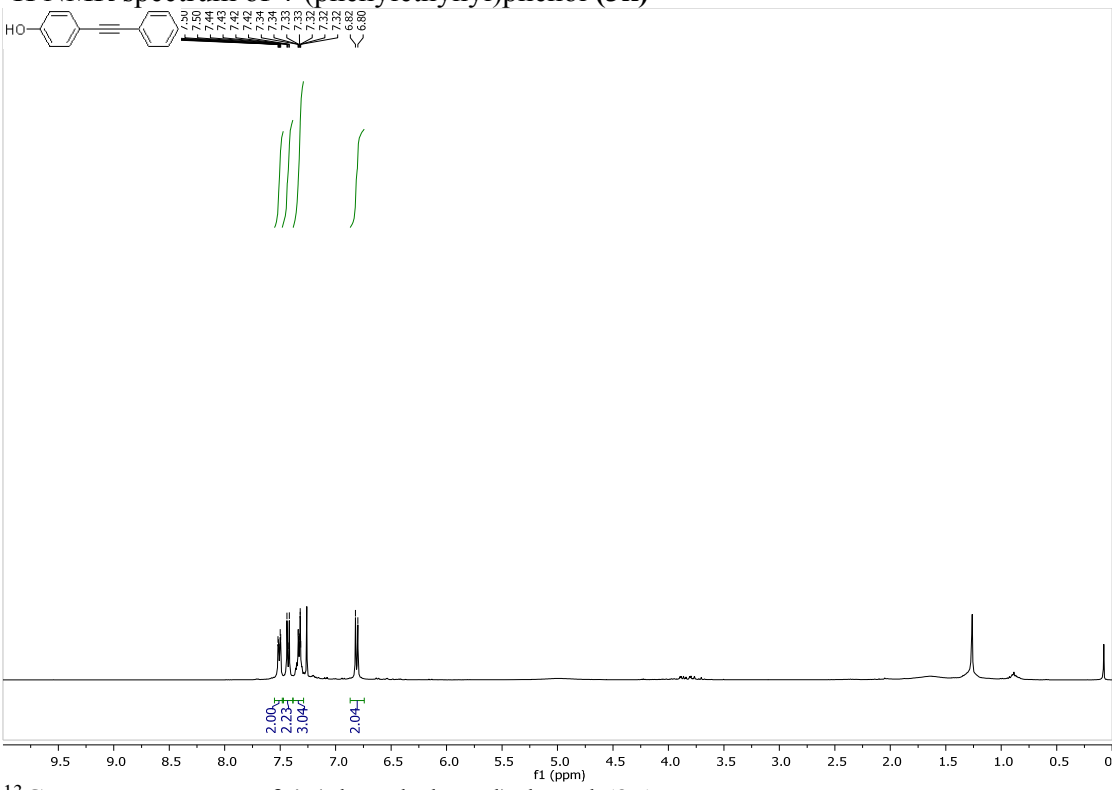
^{1}H NMR spectrum of 1,3-dimethyl-2-(phenylethynyl)benzene (**3j**)



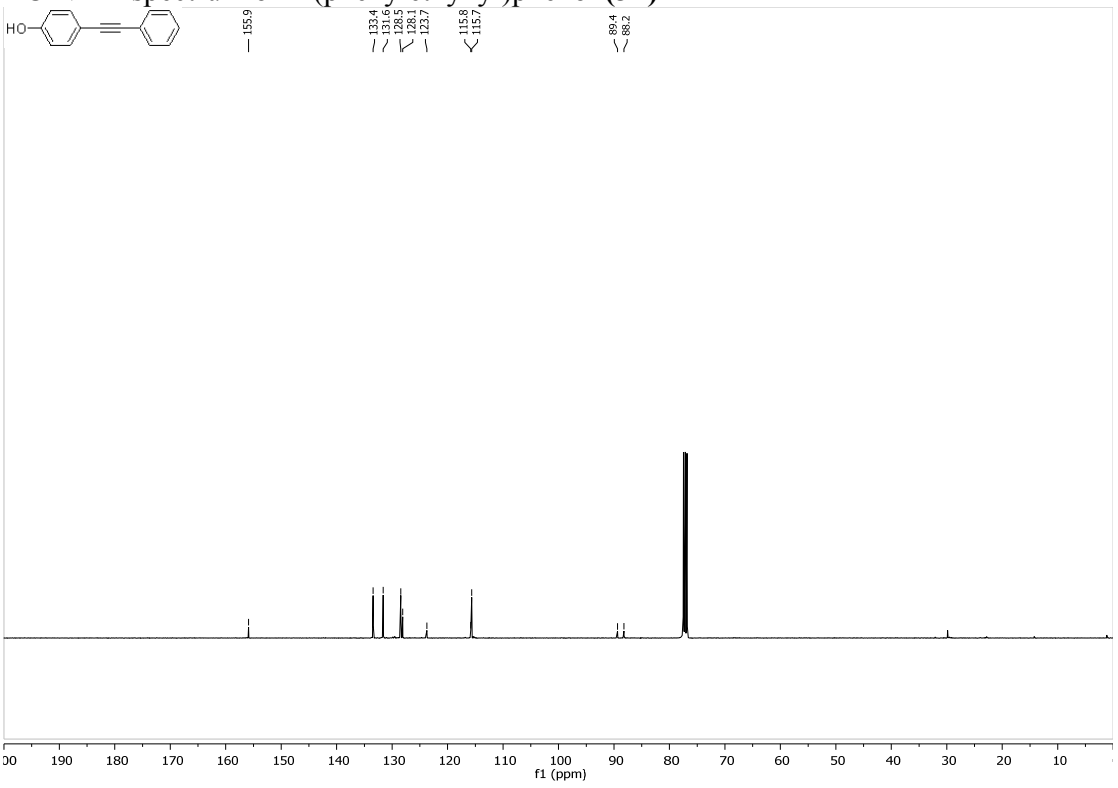
^{13}C NMR spectrum of 1,3-dimethyl-2-(phenylethynyl)benzene (**3j**)



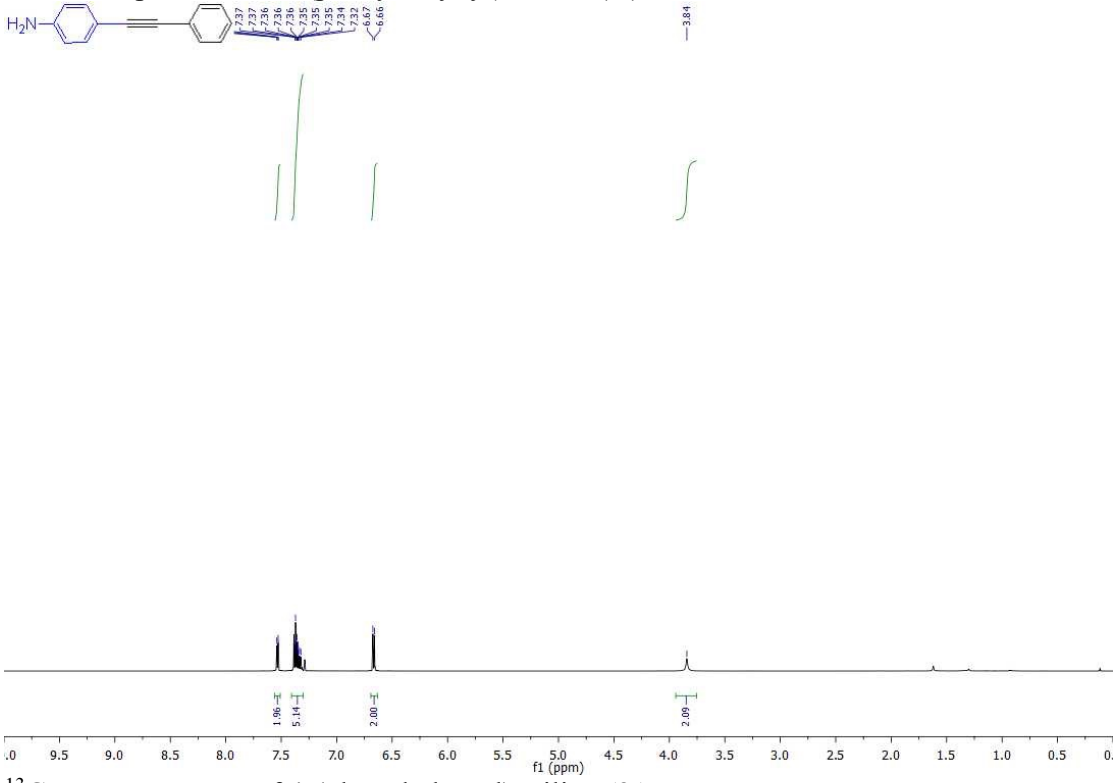
^{1}H NMR spectrum of 4-(phenylethynyl)phenol **(3k)**



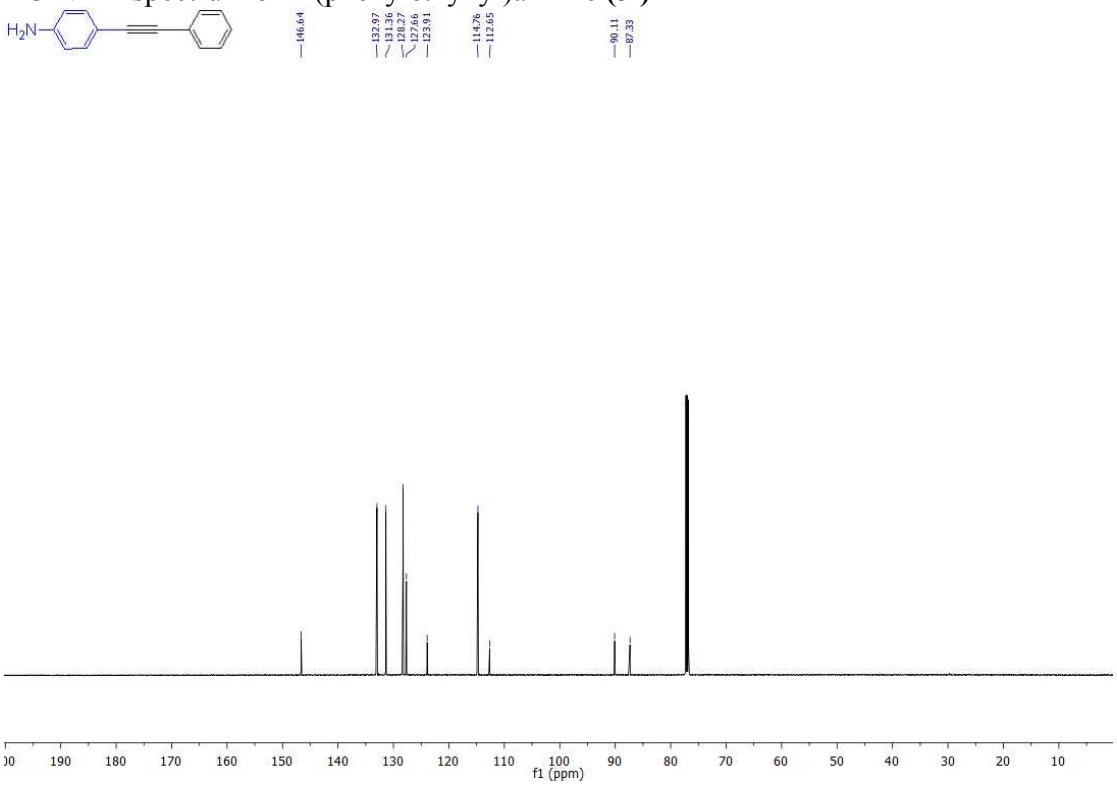
^{13}C NMR spectrum of 4-(phenylethynyl)phenol **(3k)**



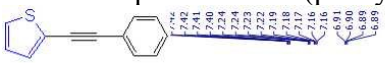
^{1}H NMR spectrum of 4-(phenylethynyl)aniline **(3l)**

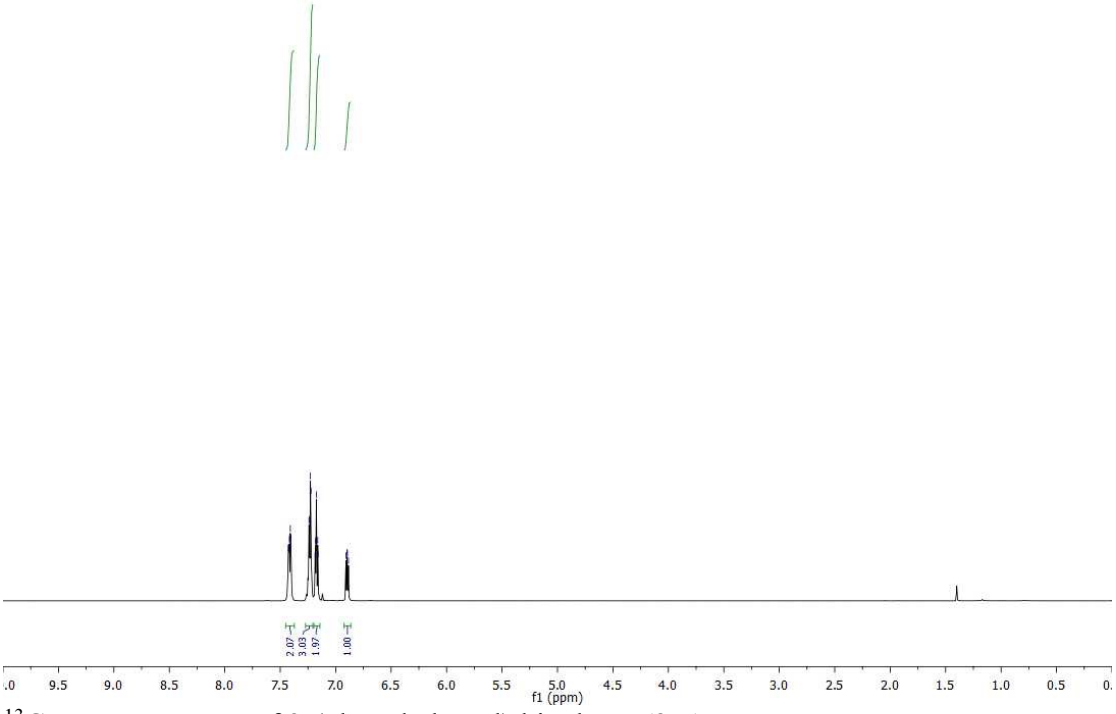


^{13}C NMR spectrum of 4-(phenylethynyl)aniline **(3l)**

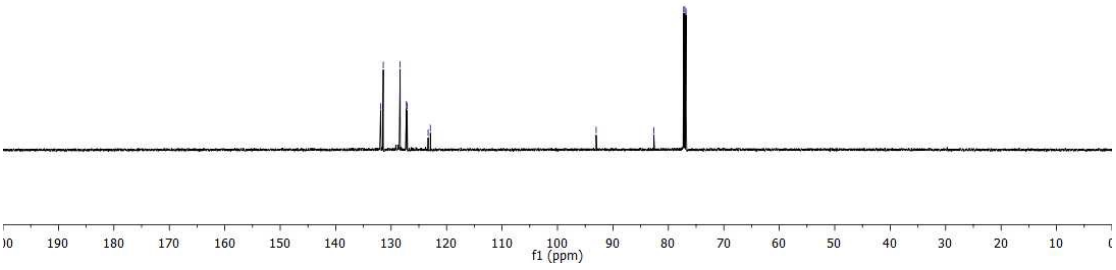


^{1}H NMR spectrum of 2-(phenylethynyl)thiophene **(3m)**

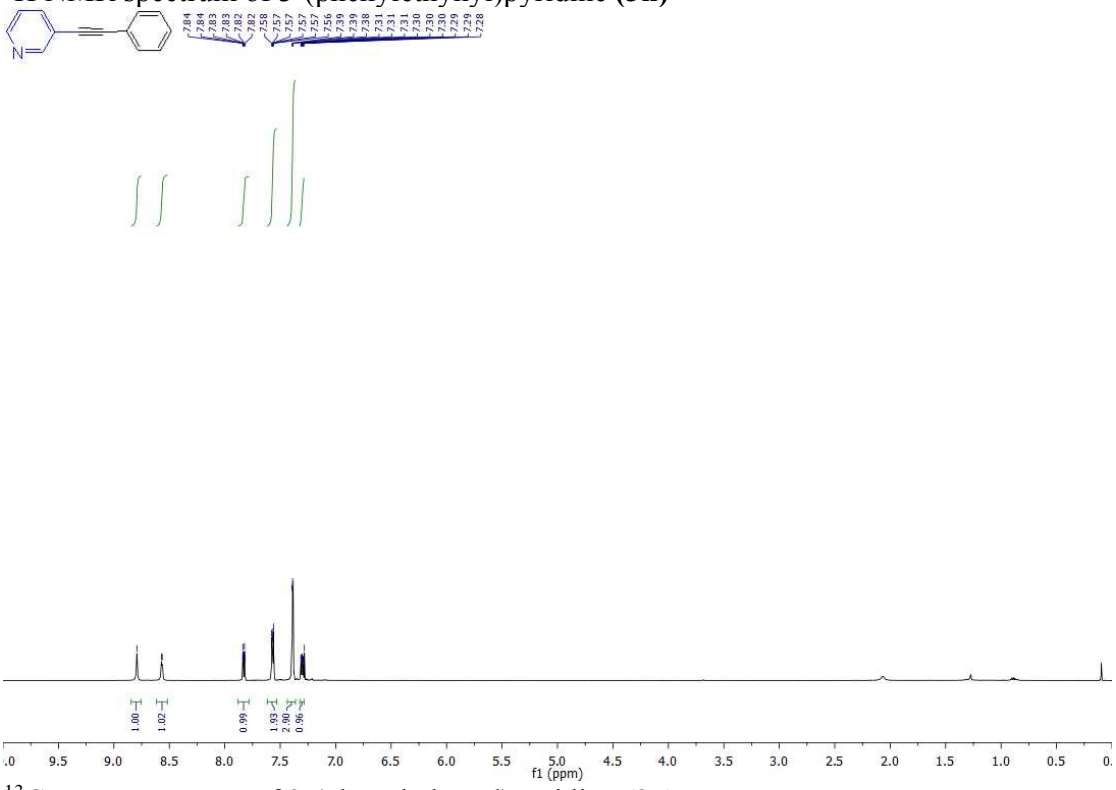




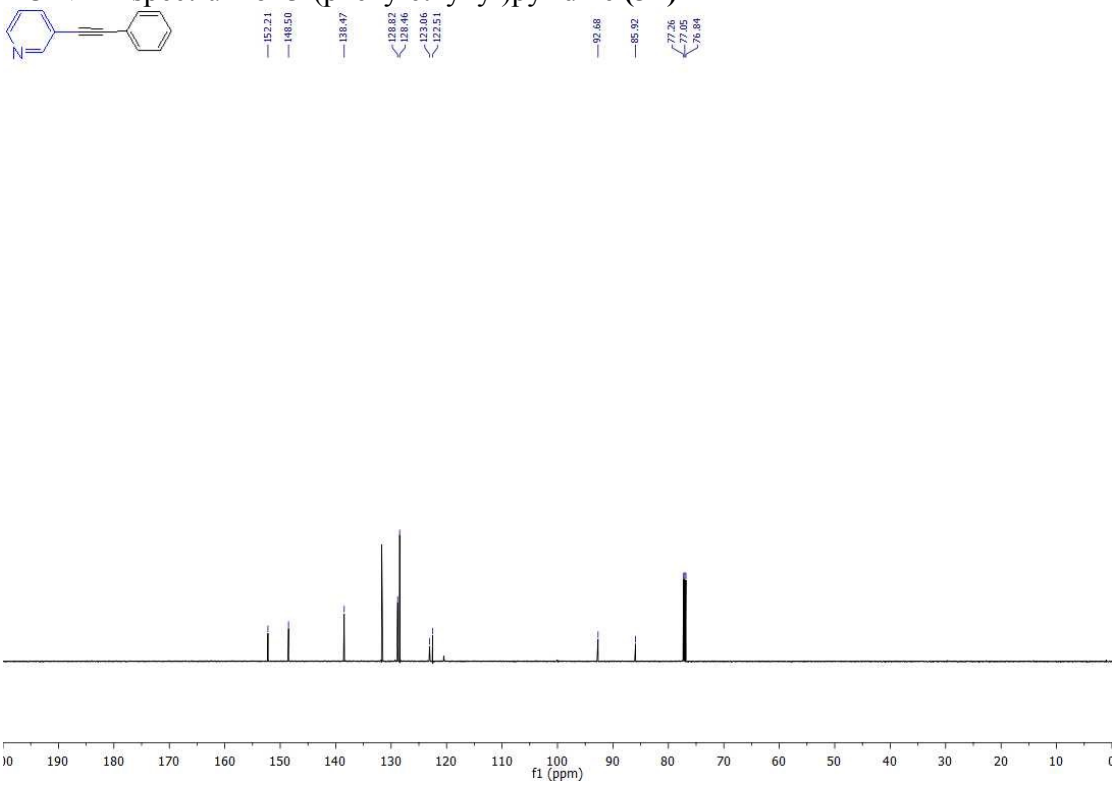
^{13}C NMR spectrum of 2-(phenylethynyl)thiophene **(3m)**



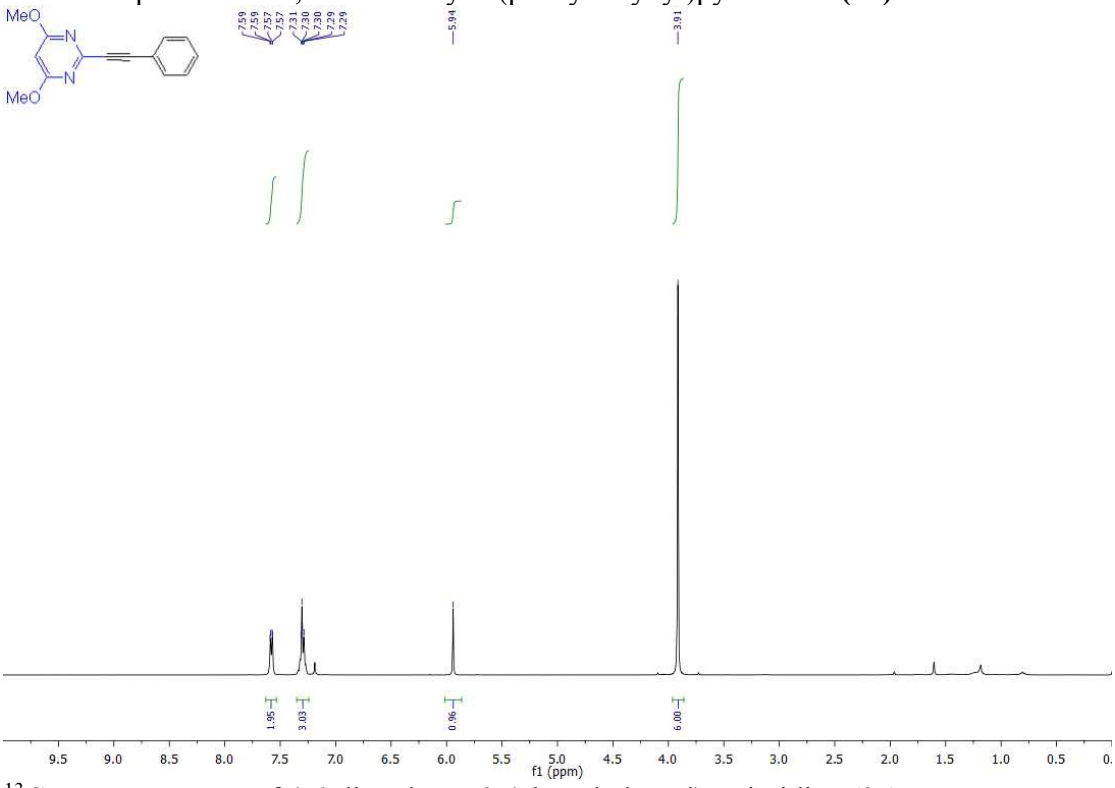
^{1}H NMR spectrum of 3-(phenylethynyl)pyridine **(3n)**



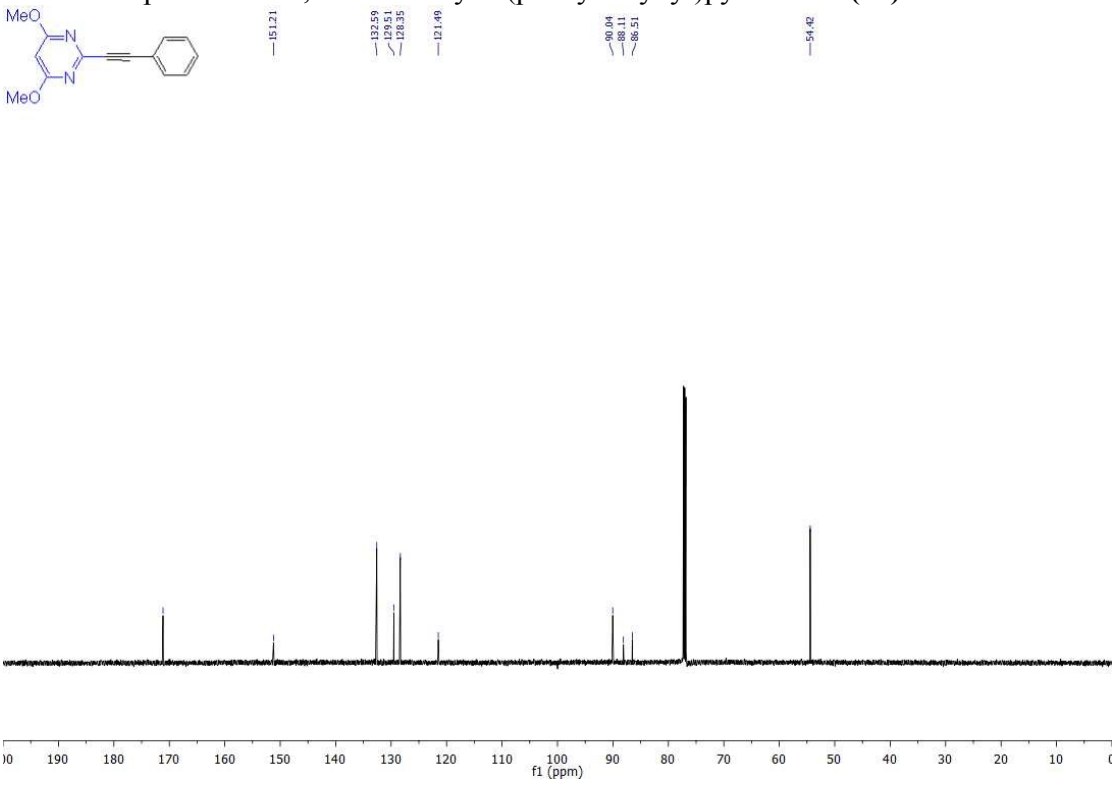
^{13}C NMR spectrum of 3-(phenylethynyl)pyridine **(3n)**



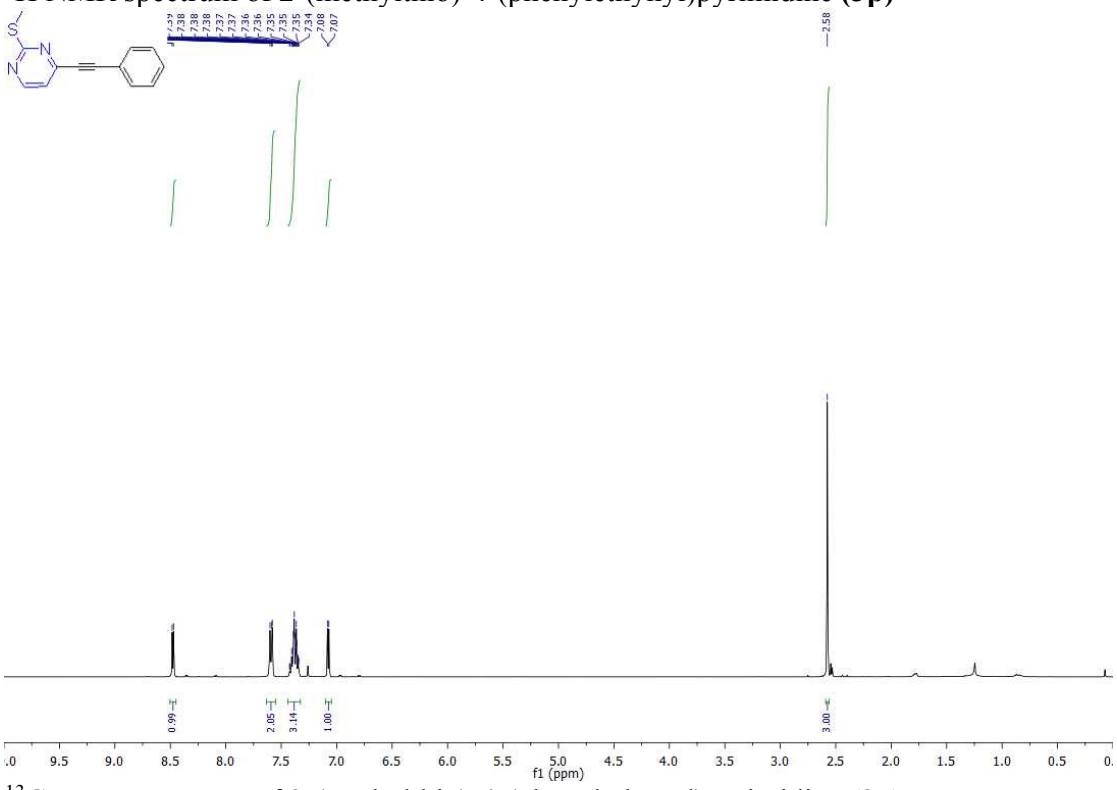
^{1}H NMR spectrum of 4,6-dimethoxy-2-(phenylethynyl)pyrimidine **(3o)**



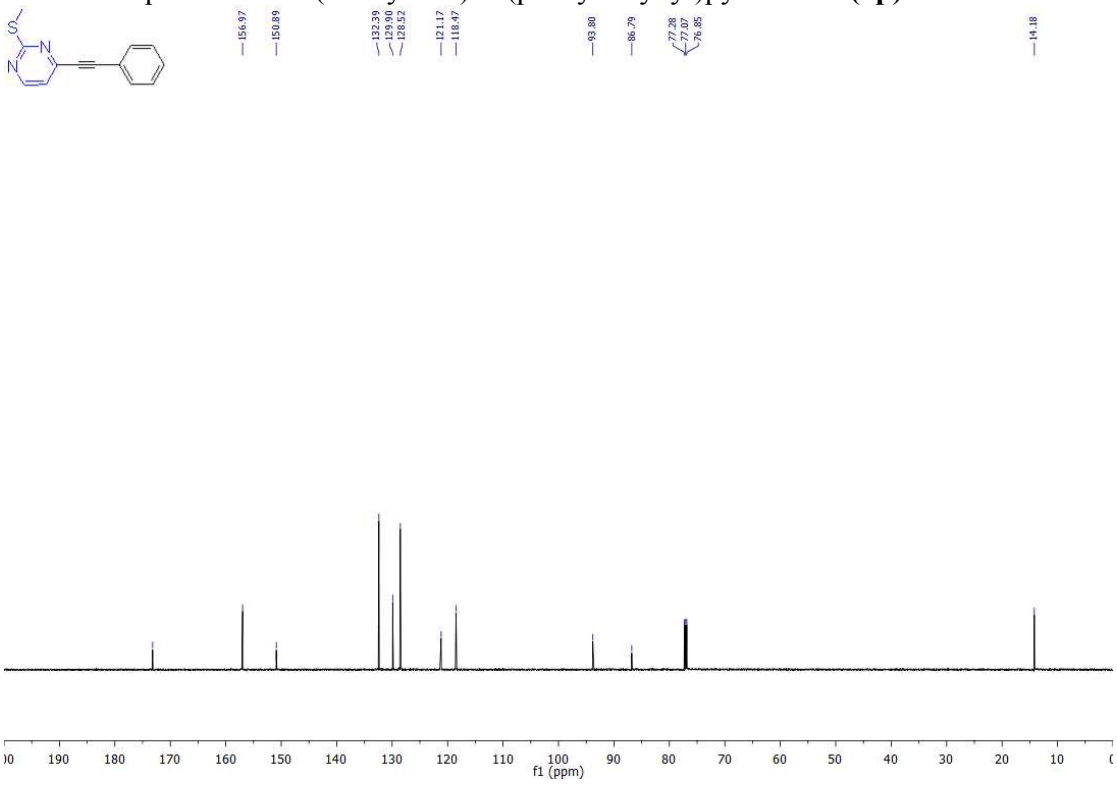
^{13}C NMR spectrum of 4,6-dimethoxy-2-(phenylethynyl)pyrimidine **(3o)**



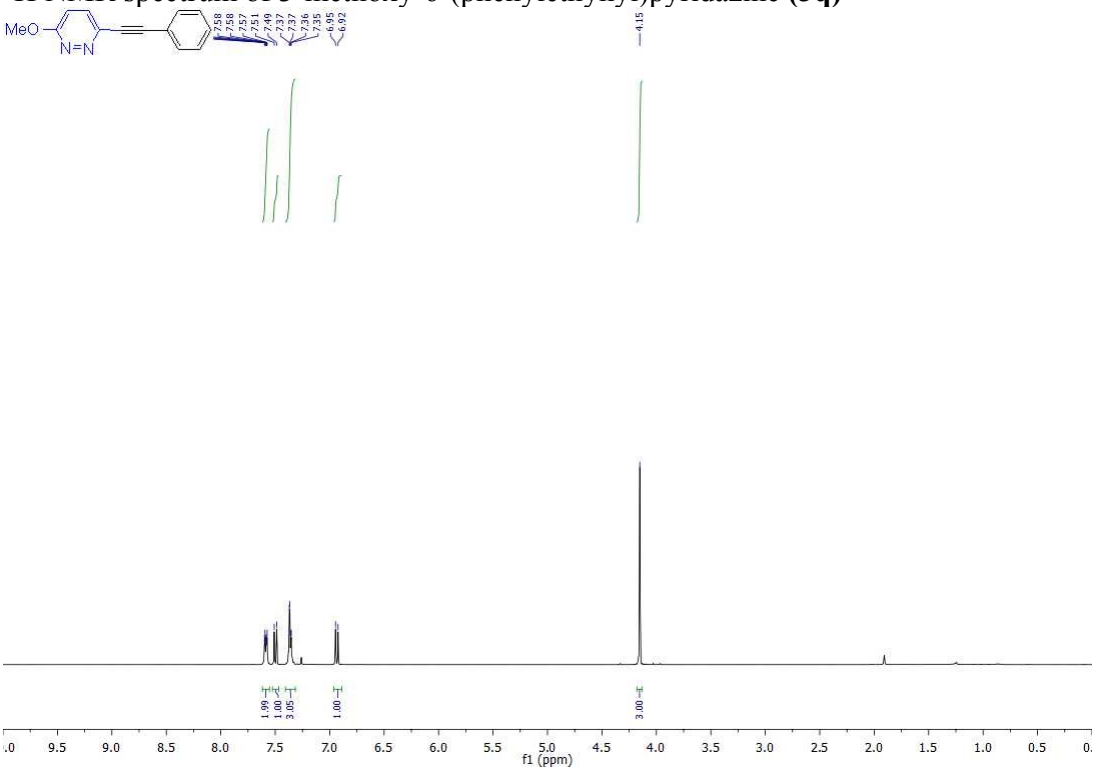
^{1}H NMR spectrum of 2-(methylthio)-4-(phenylethynyl)pyrimidine **(3p)**



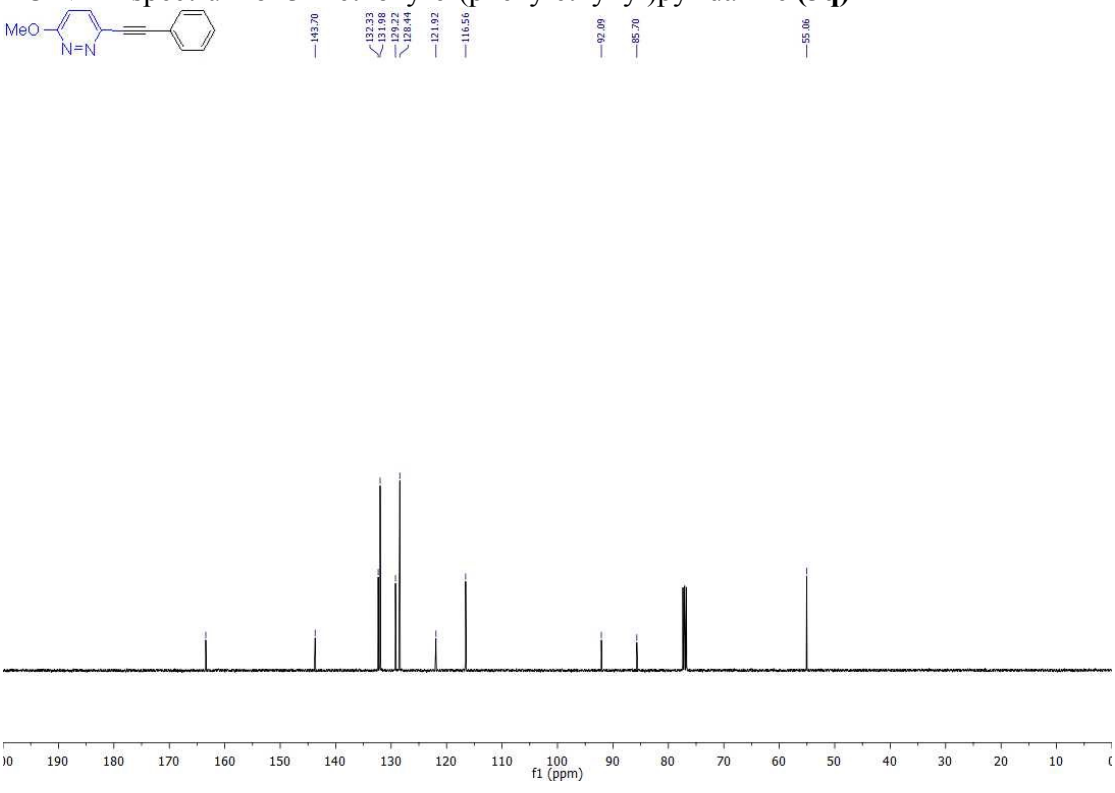
^{13}C NMR spectrum of 2-(methylthio)-4-(phenylethynyl)pyrimidine **(3p)**



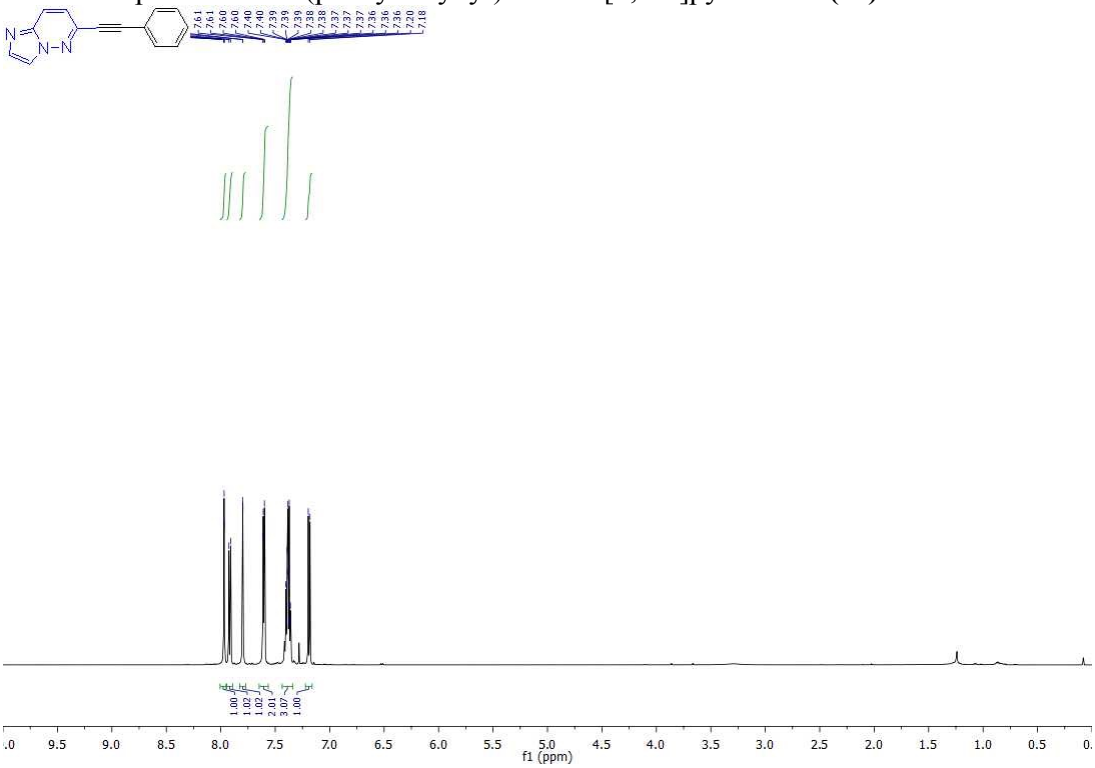
^{1}H NMR spectrum of 3-methoxy-6-(phenylethynyl)pyridazine **(3q)**



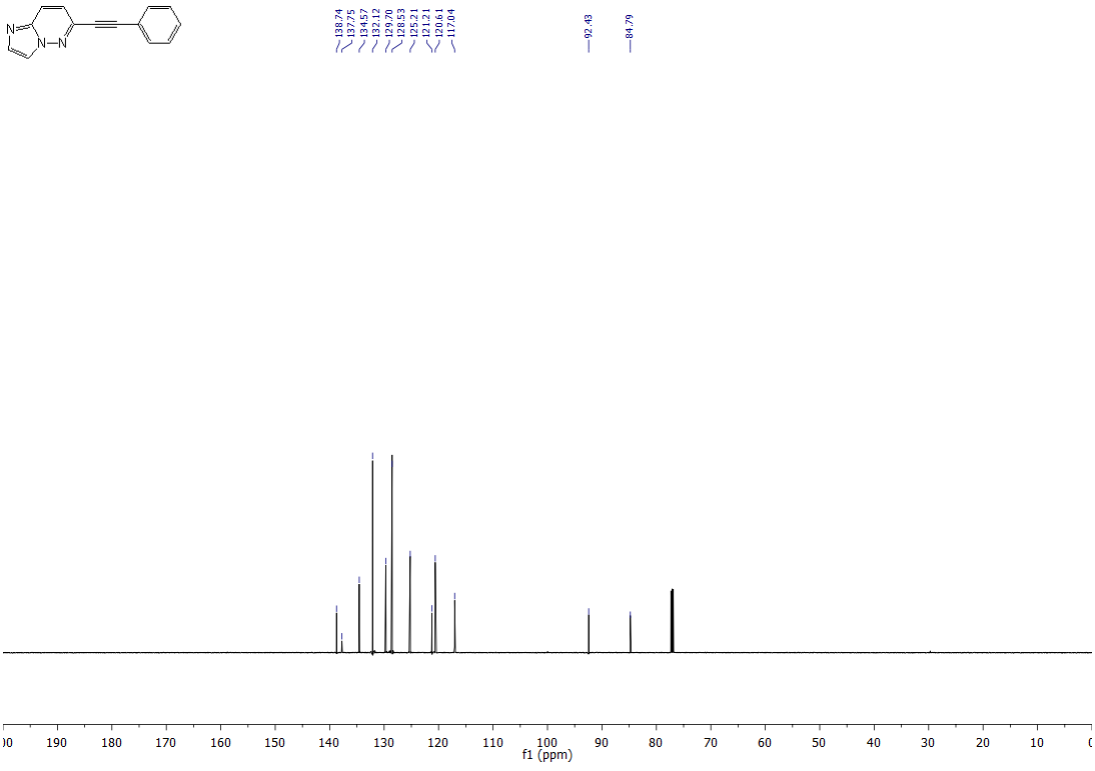
^{13}C NMR spectrum of 3-methoxy-6-(phenylethynyl)pyridazine **(3q)**



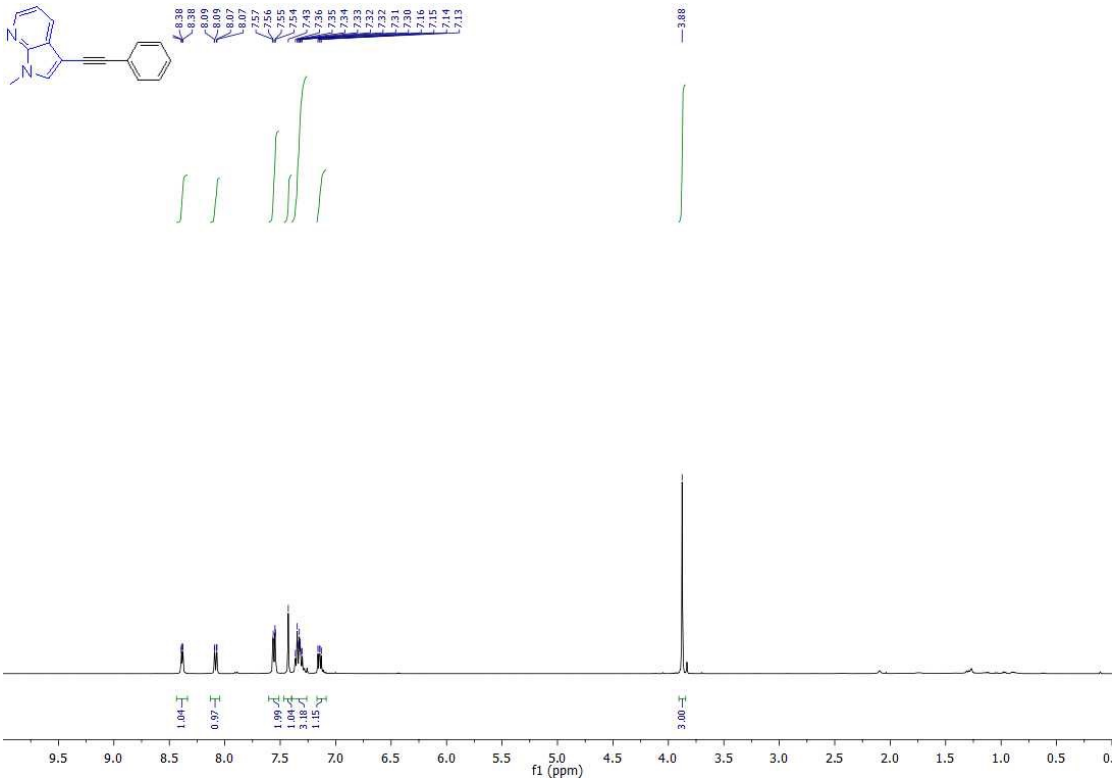
^{1}H NMR spectrum of 6-(phenylethynyl)imidazo[1,2-b]pyridazine **(3r)**



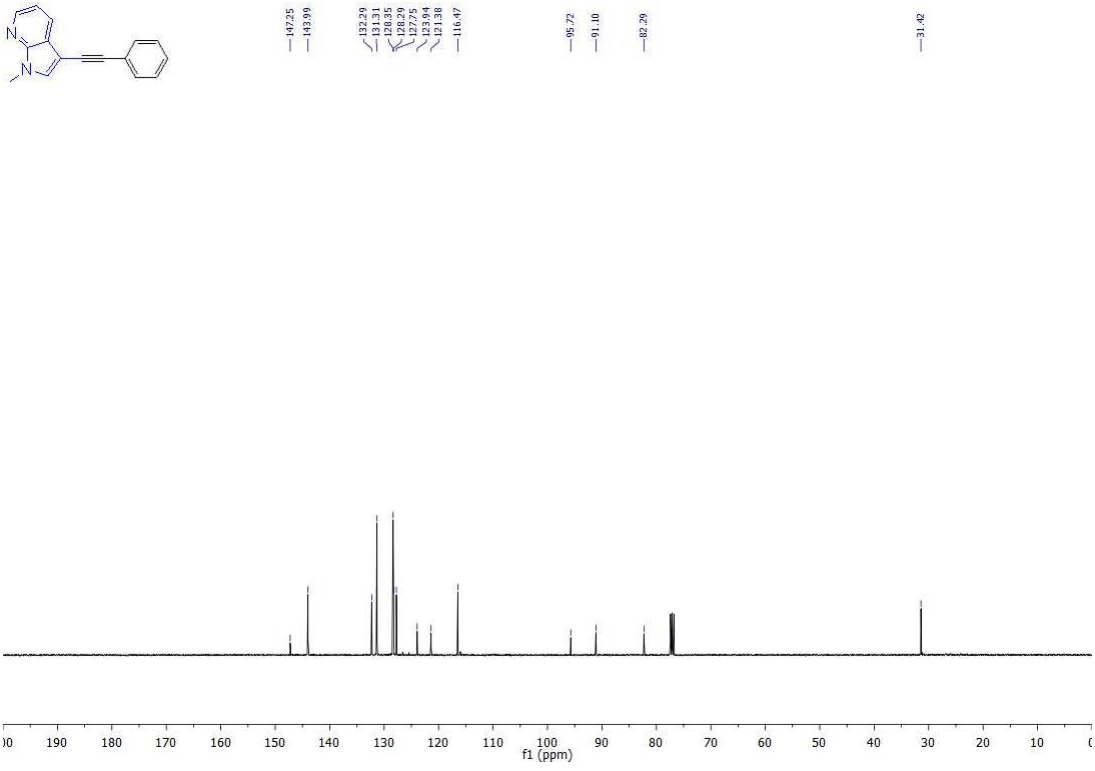
^{13}C NMR spectrum of 6-(phenylethynyl)imidazo[1,2-b]pyridazine (**3r**)



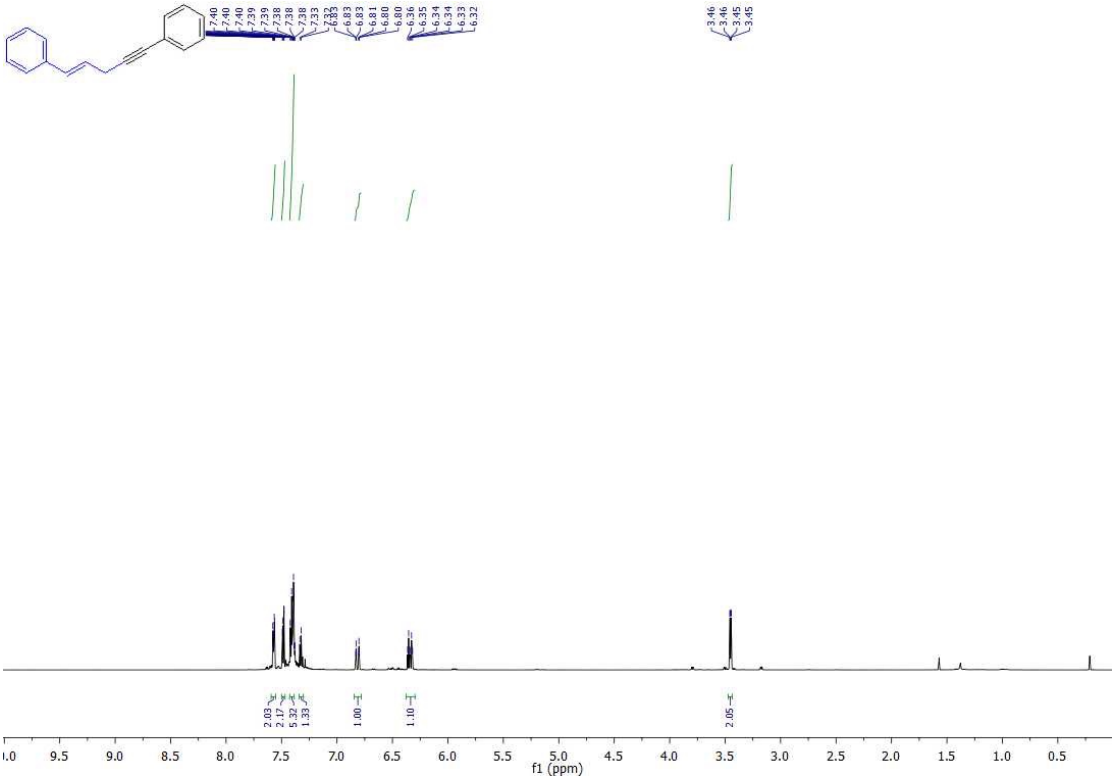
^{1}H NMR spectrum of 1-methyl-3-(phenylethynyl)-1H-pyrrolo[2,3-b]pyridine (**3s**)



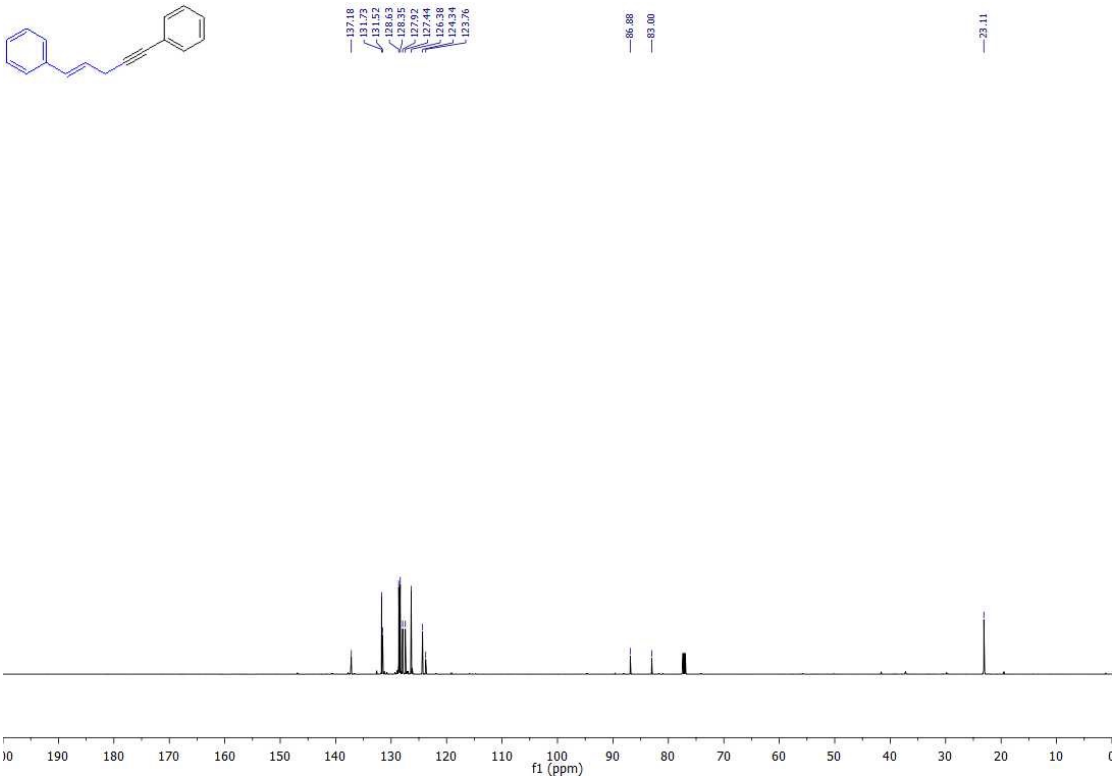
^{13}C NMR spectrum of 1-methyl-3-(phenylethynyl)-1H-pyrrolo[2,3-b]pyridine (**3s**)



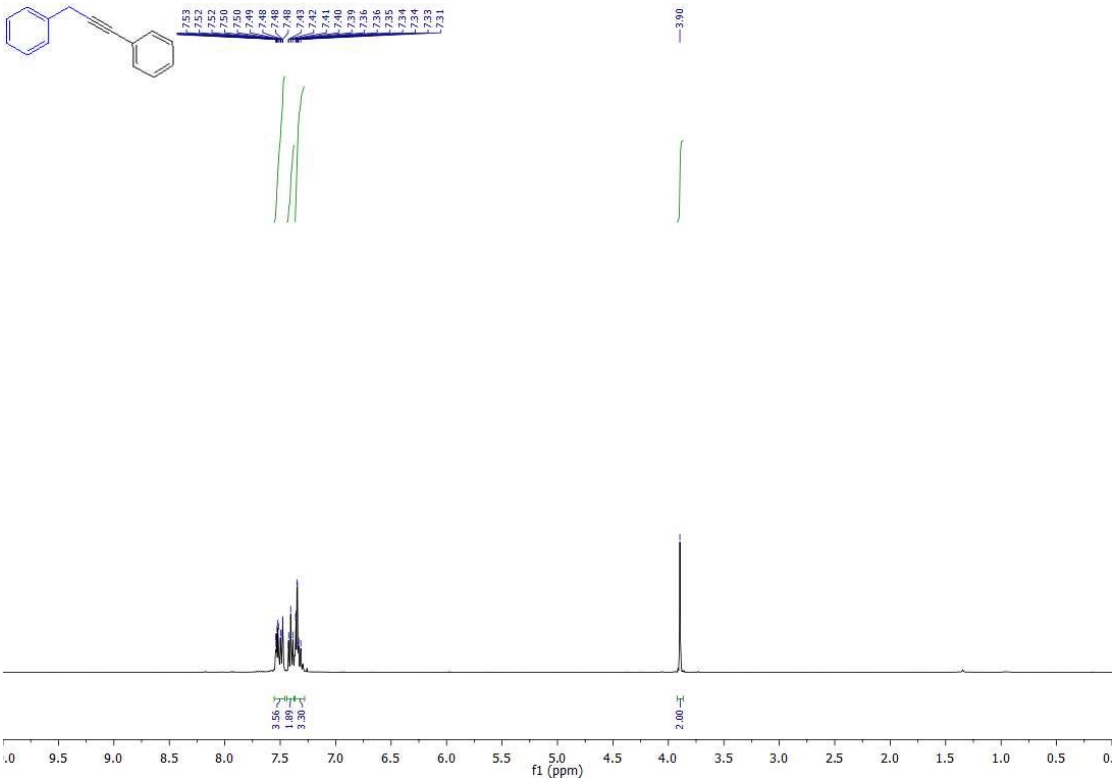
^{1}H NMR spectrum of (E)-pent-1-en-4-yne-1,5-diyldibenzene (**3t**)



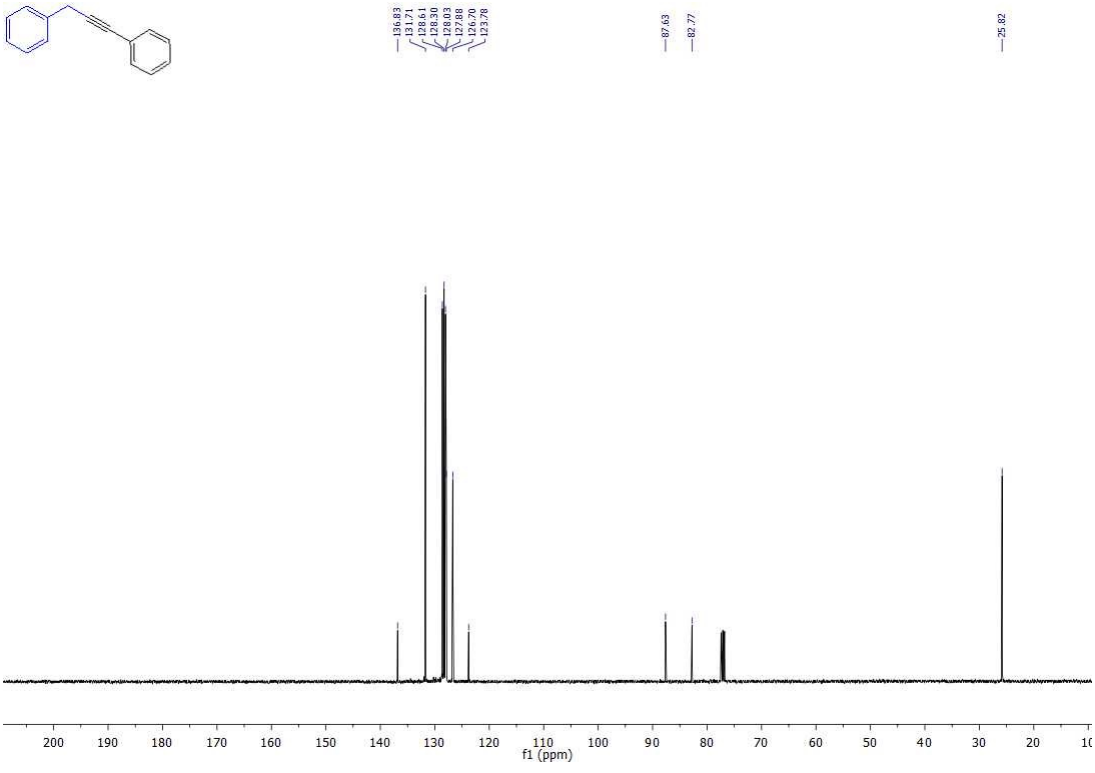
^{13}C NMR spectrum of (E)-pent-1-en-4-yne-1,5-diyldibenzene (**3t**)



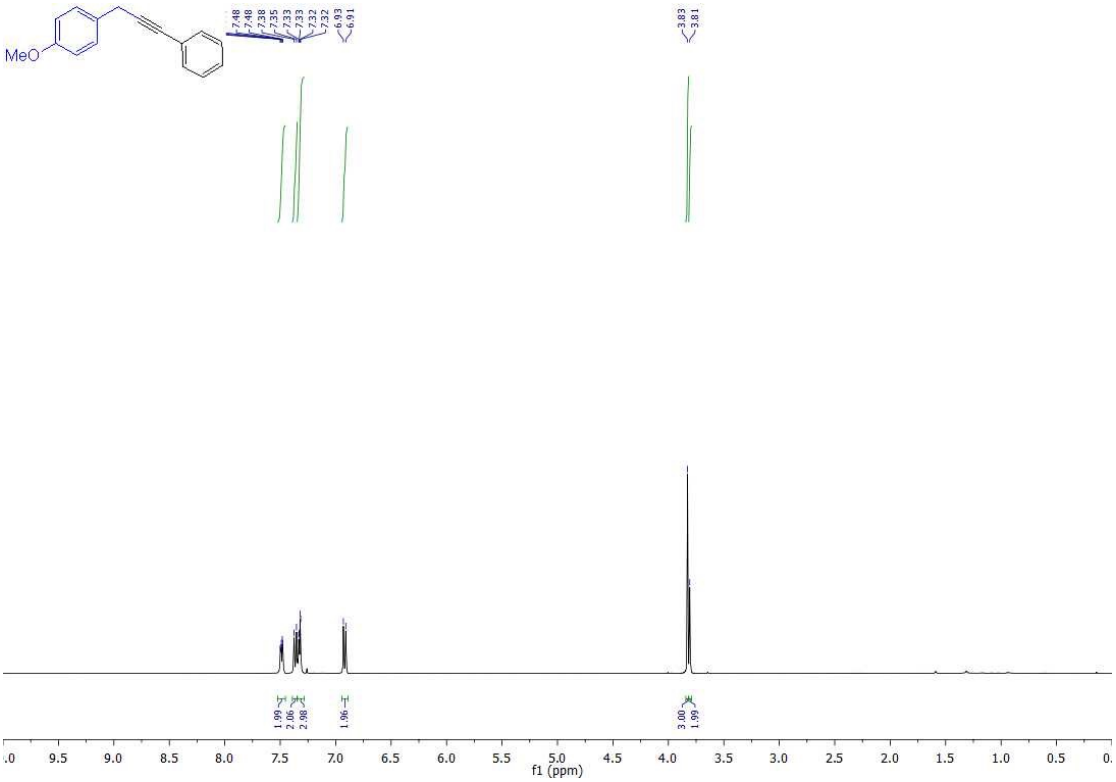
^{1}H NMR spectrum of prop-1-yne-1,3-diyldibenzene **(3u)**



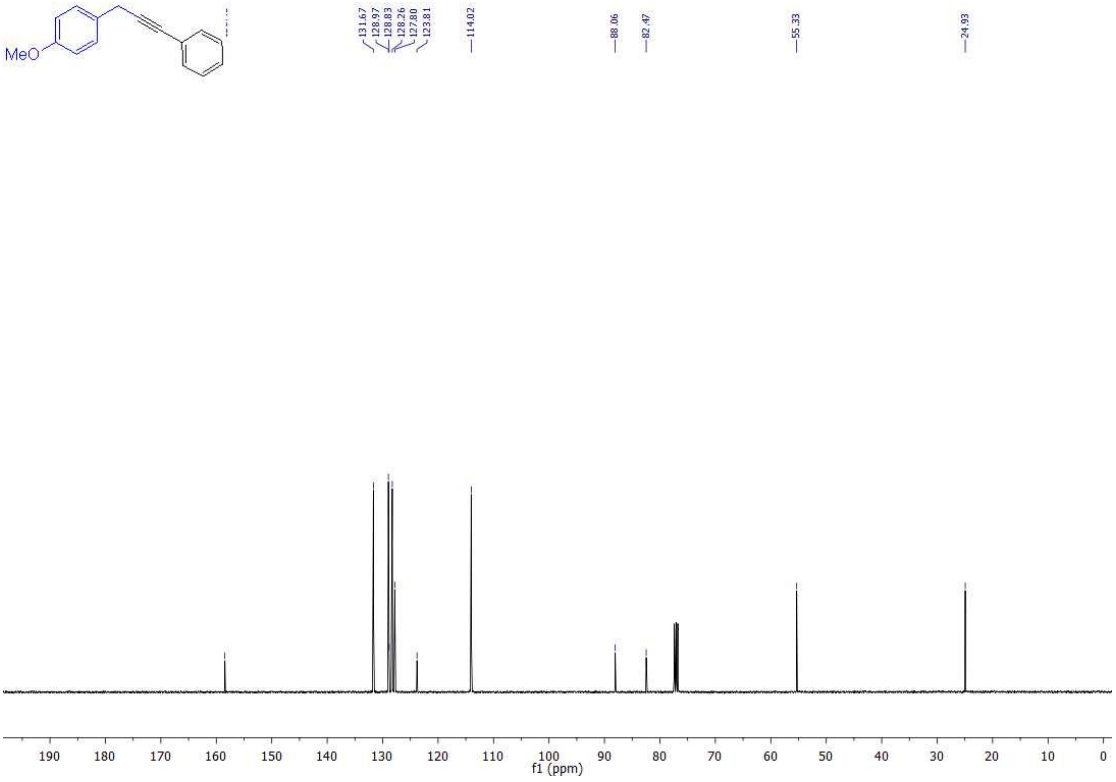
^{13}C NMR spectrum of prop-1-yne-1,3-diyldibenzene **(3u)**



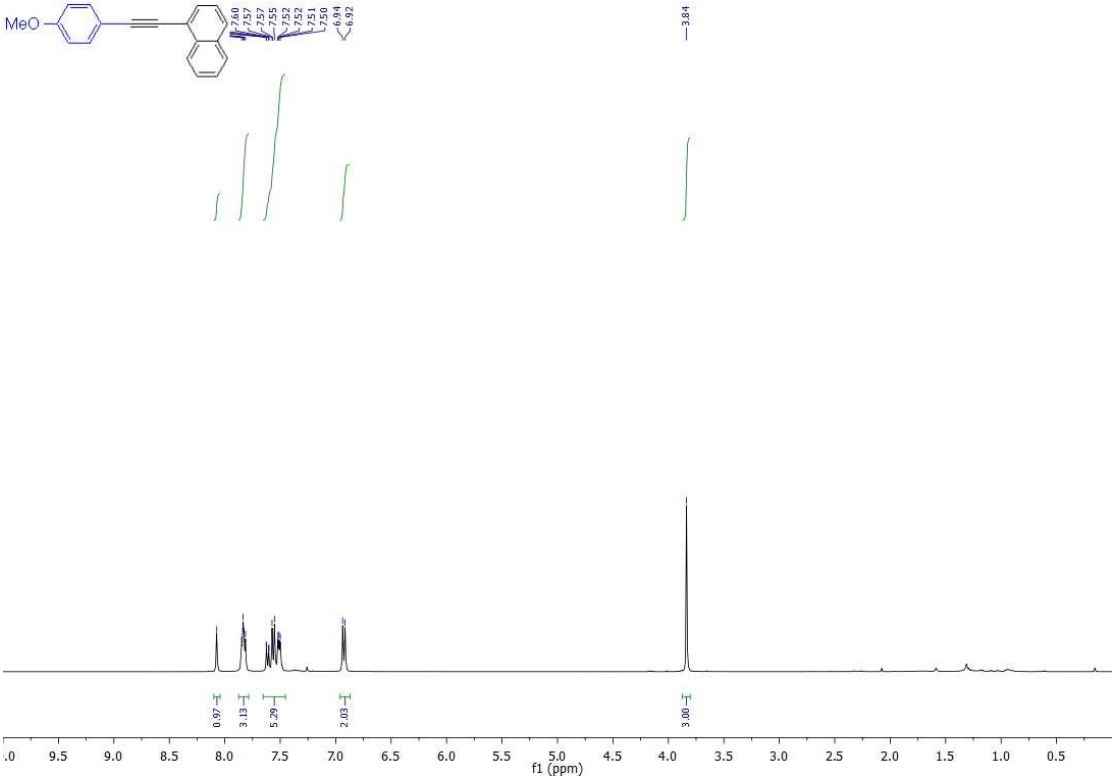
^{1}H NMR spectrum of 1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (**3v**)



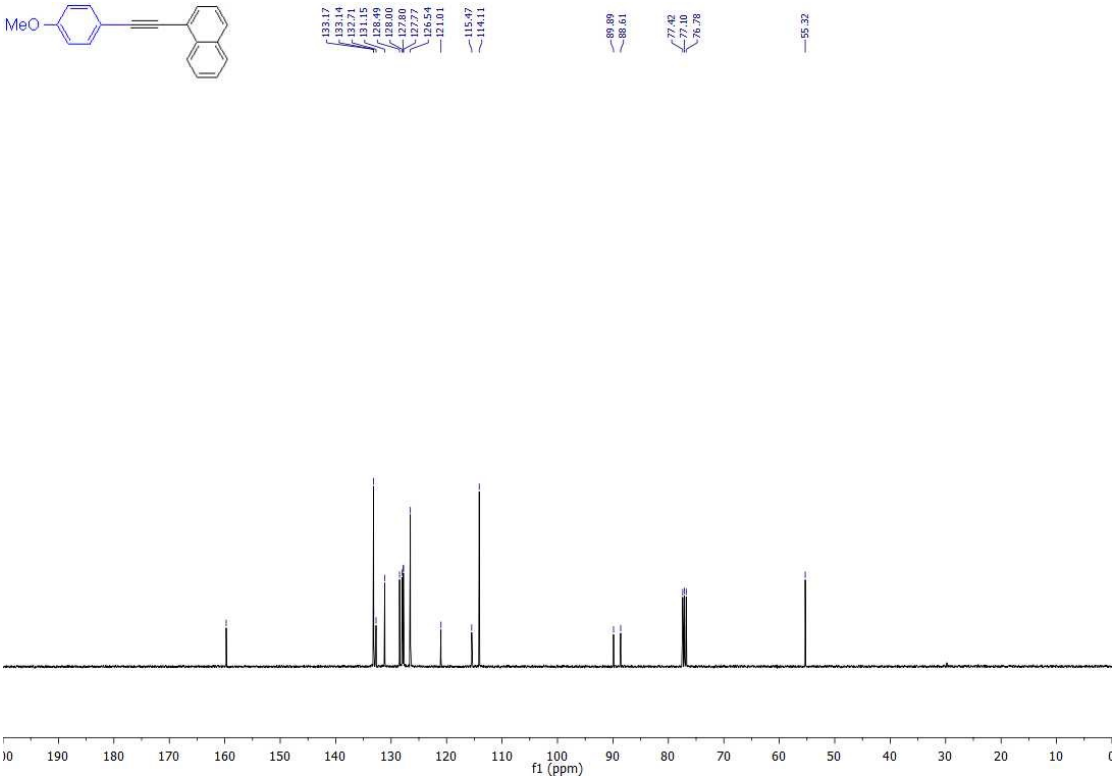
^{13}C NMR spectrum of 1-methoxy-4-(3-phenylprop-2-yn-1-yl)benzene (**3v**)



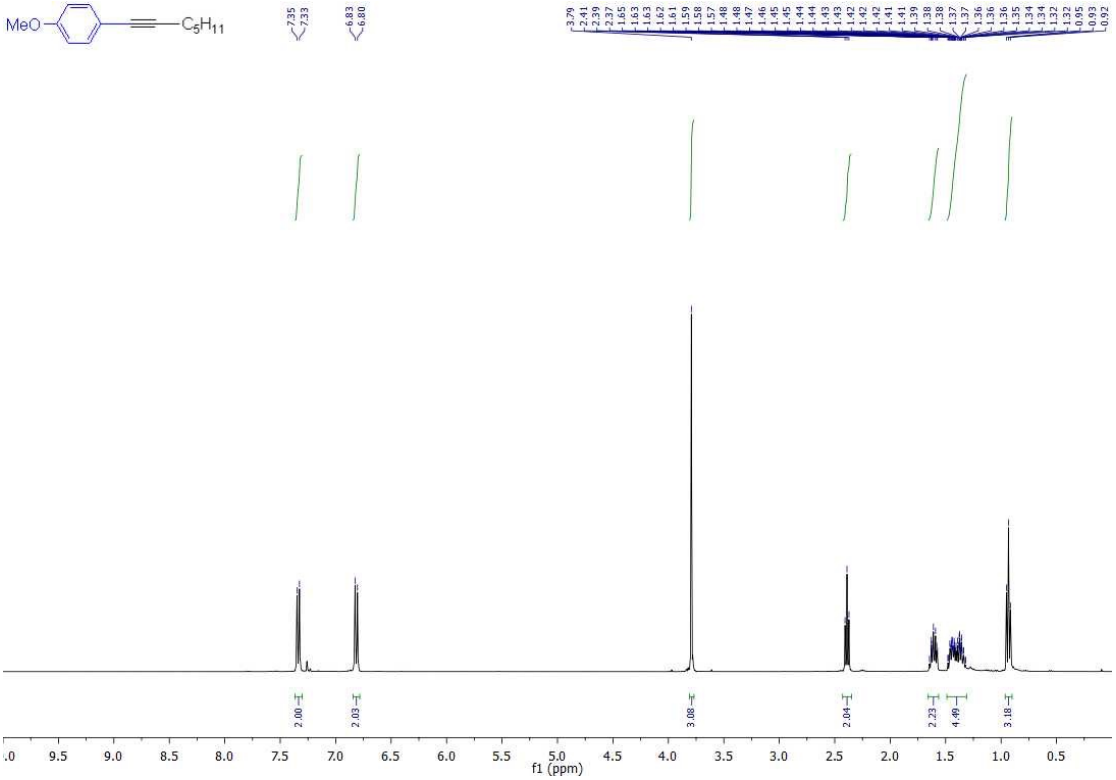
^{1}H NMR spectrum of 1-((4-methoxyphenyl)ethynyl)naphthalene (**4b**)



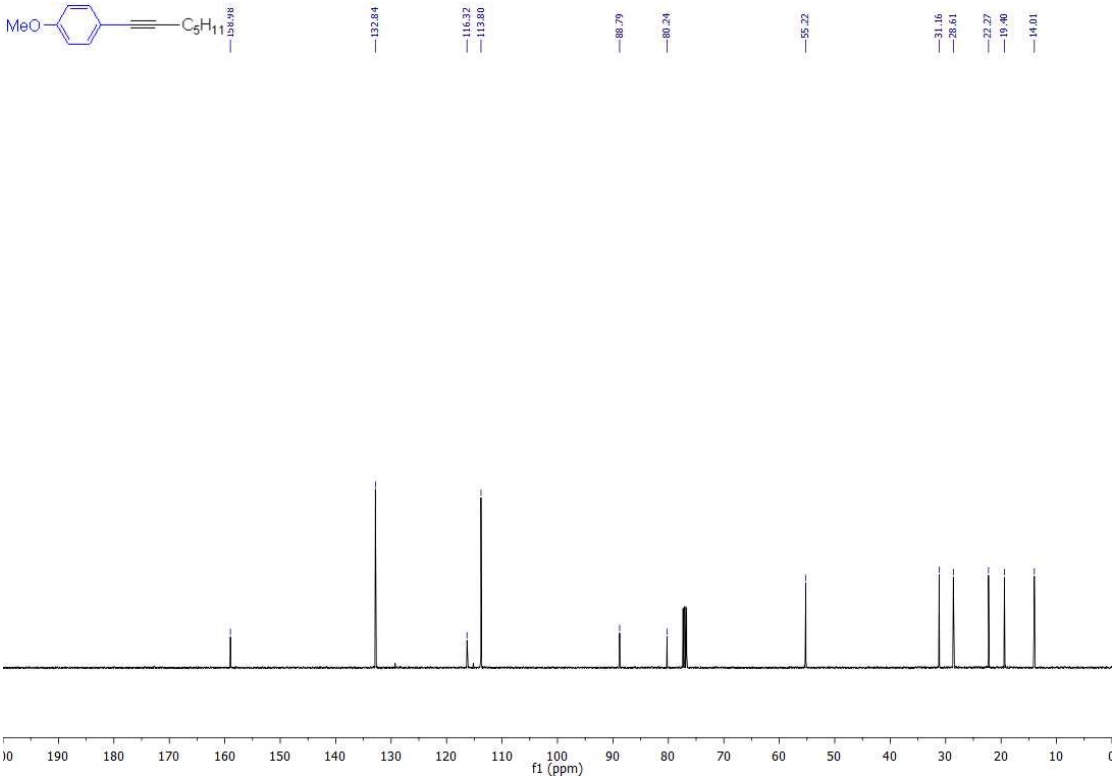
^{13}C NMR spectrum of 1-((4-methoxyphenyl)ethynyl)naphthalene (**4b**)



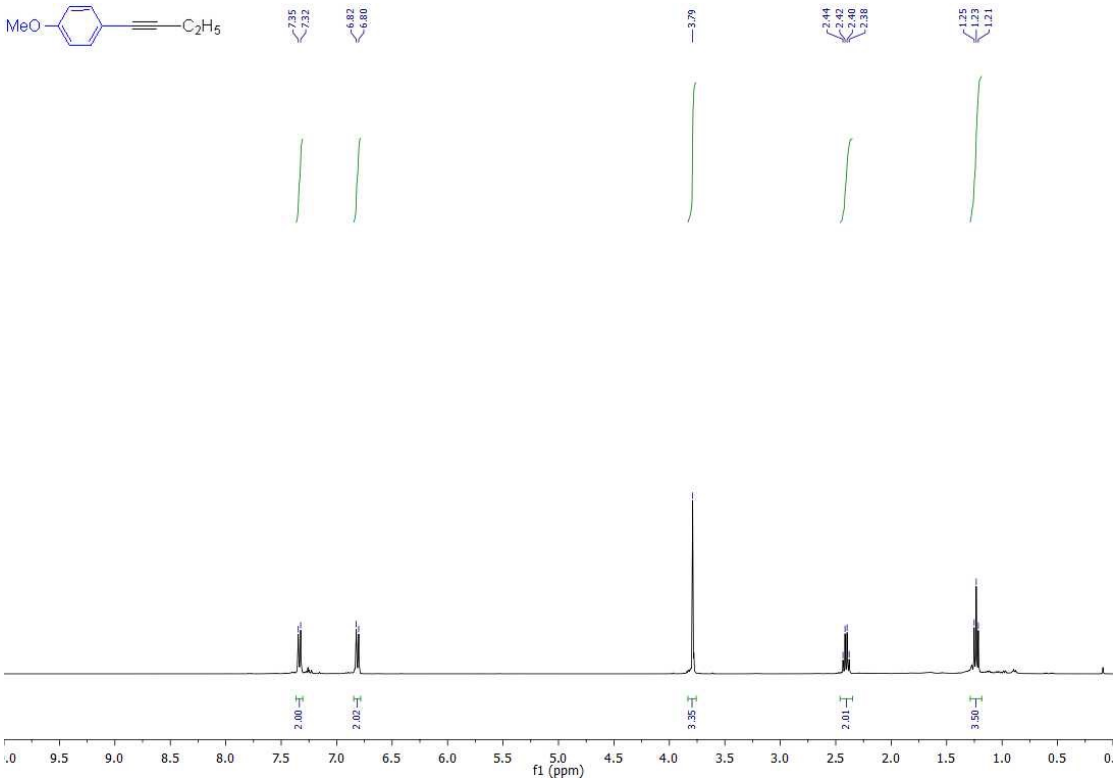
^{1}H NMR spectrum of 1-(hept-1-yn-1-yl)-4-methoxybenzene (**4c**)



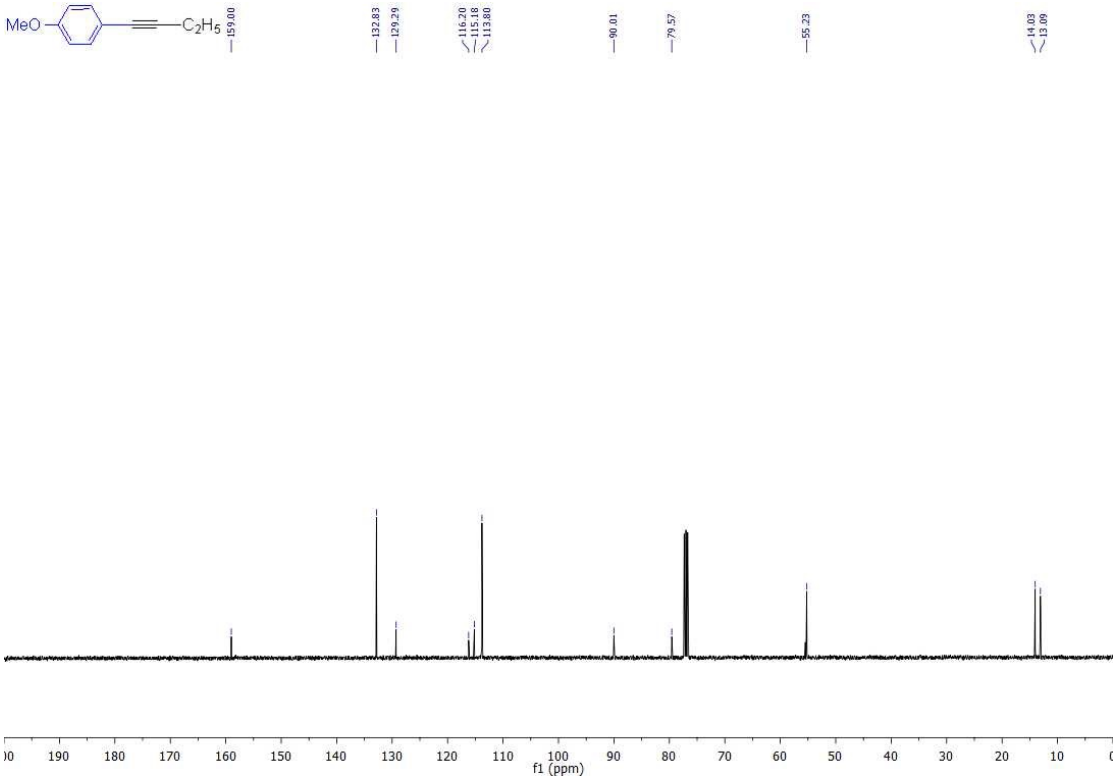
^{13}C NMR spectrum of 1-(hept-1-yn-1-yl)-4-methoxybenzene (**4c**)



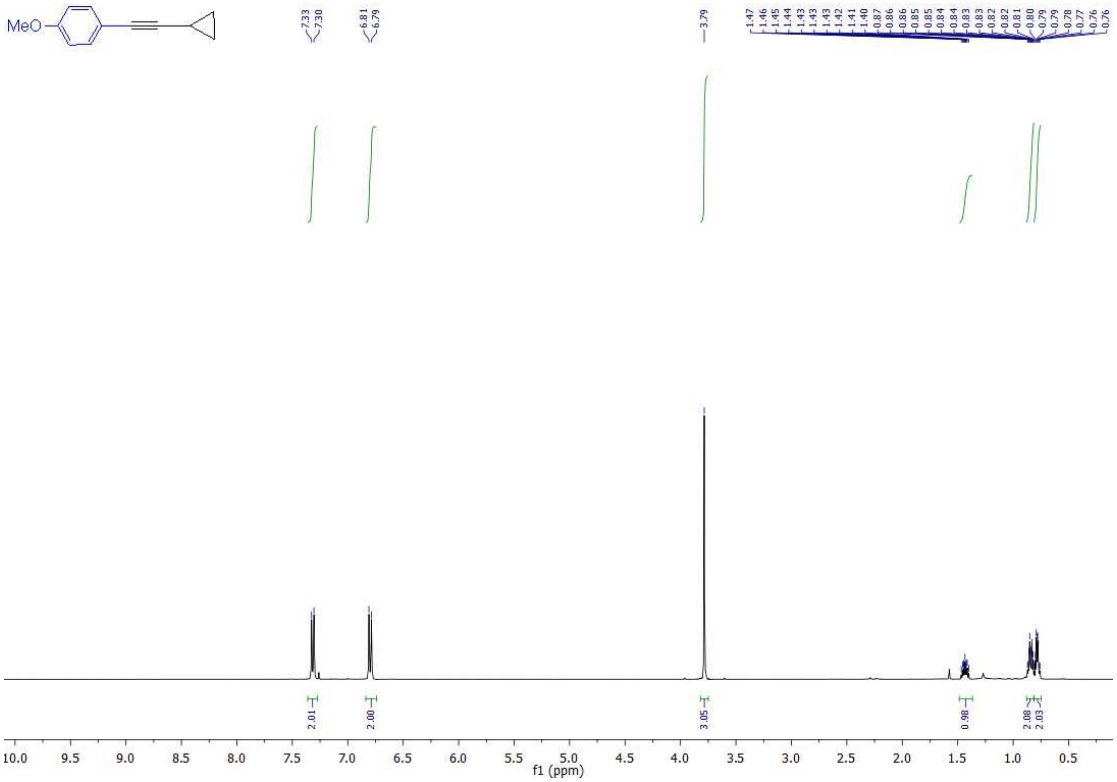
^{1}H NMR spectrum of 1-(but-1-yn-1-yl)-4-methoxybenzene (**4d**)



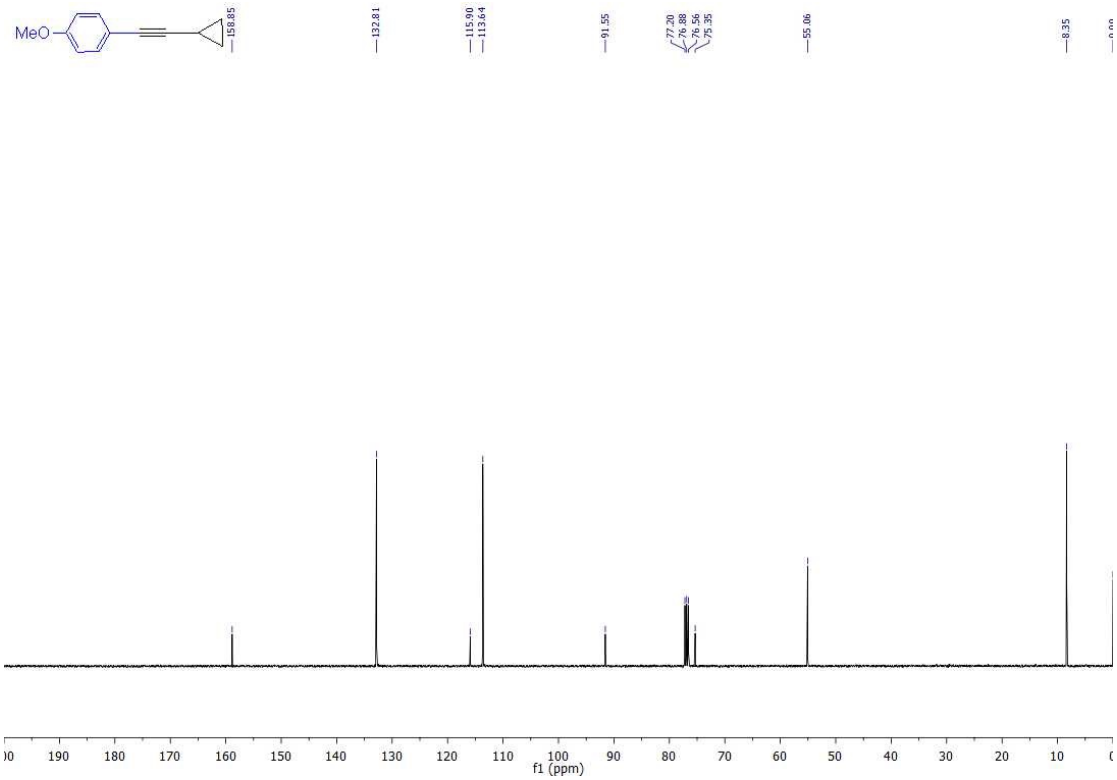
^{13}C NMR spectrum of 1-(but-1-yn-1-yl)-4-methoxybenzene (**4d**)



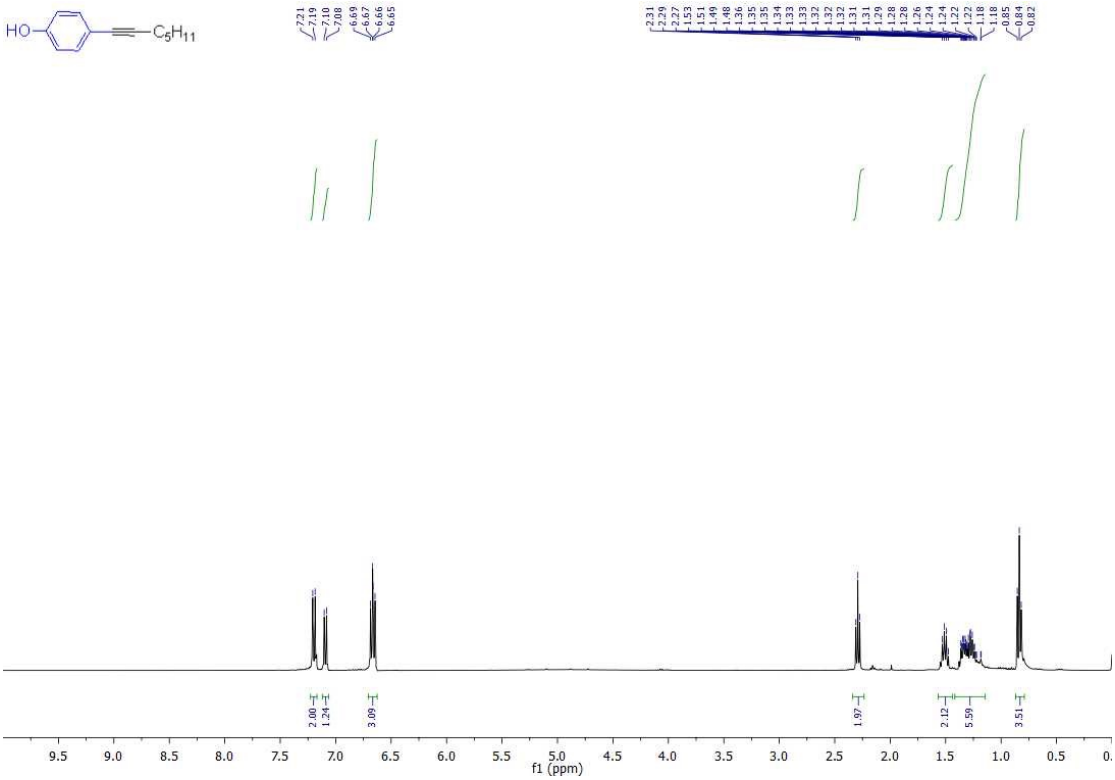
^{1}H NMR spectrum of 1-(cyclopropylethynyl)-4-methoxybenzene (**4e**)



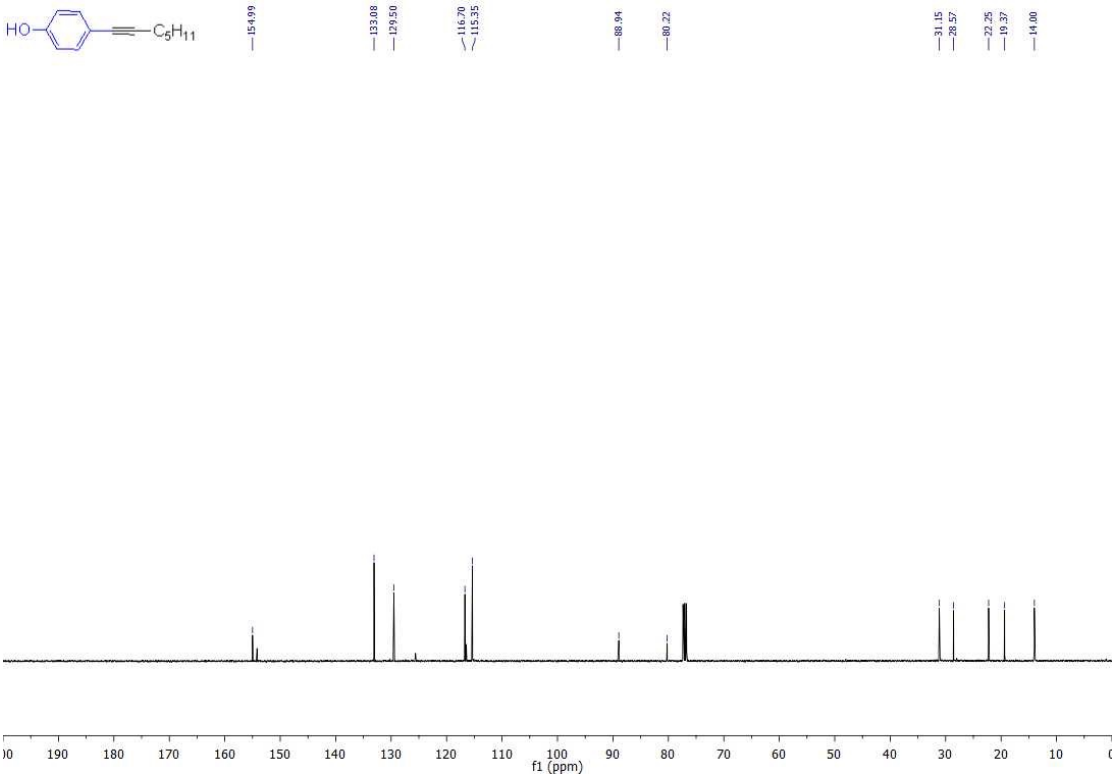
^{13}C NMR spectrum of 1-(cyclopropylethynyl)-4-methoxybenzene (**4e**)



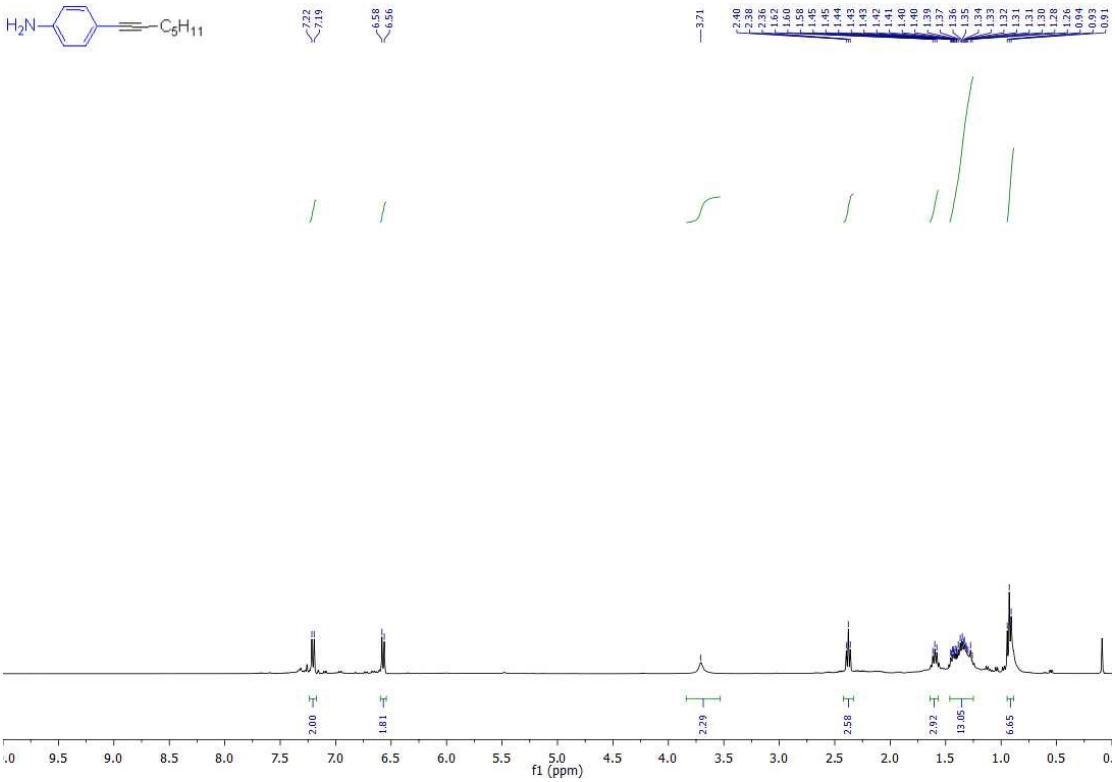
^{1}H NMR spectrum of 4-(hept-1-yn-1-yl)phenol (**4f**)



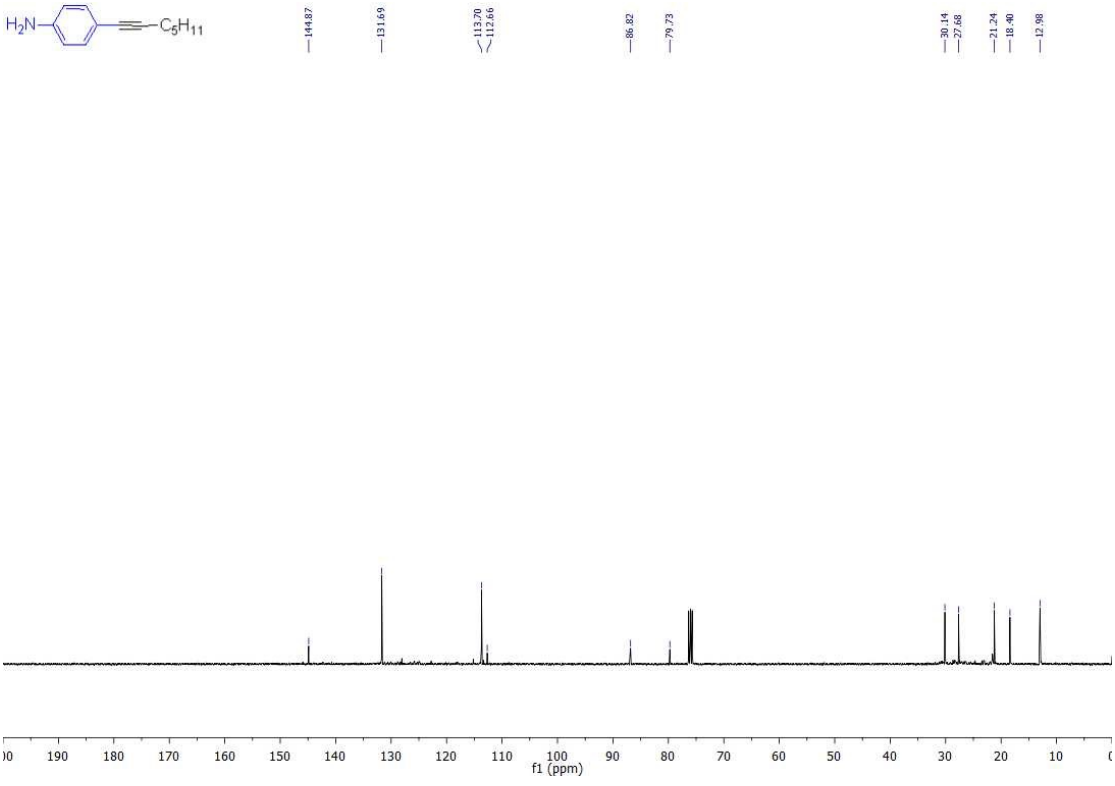
^{13}C NMR spectrum of 4-(hept-1-yn-1-yl)phenol (**4f**)



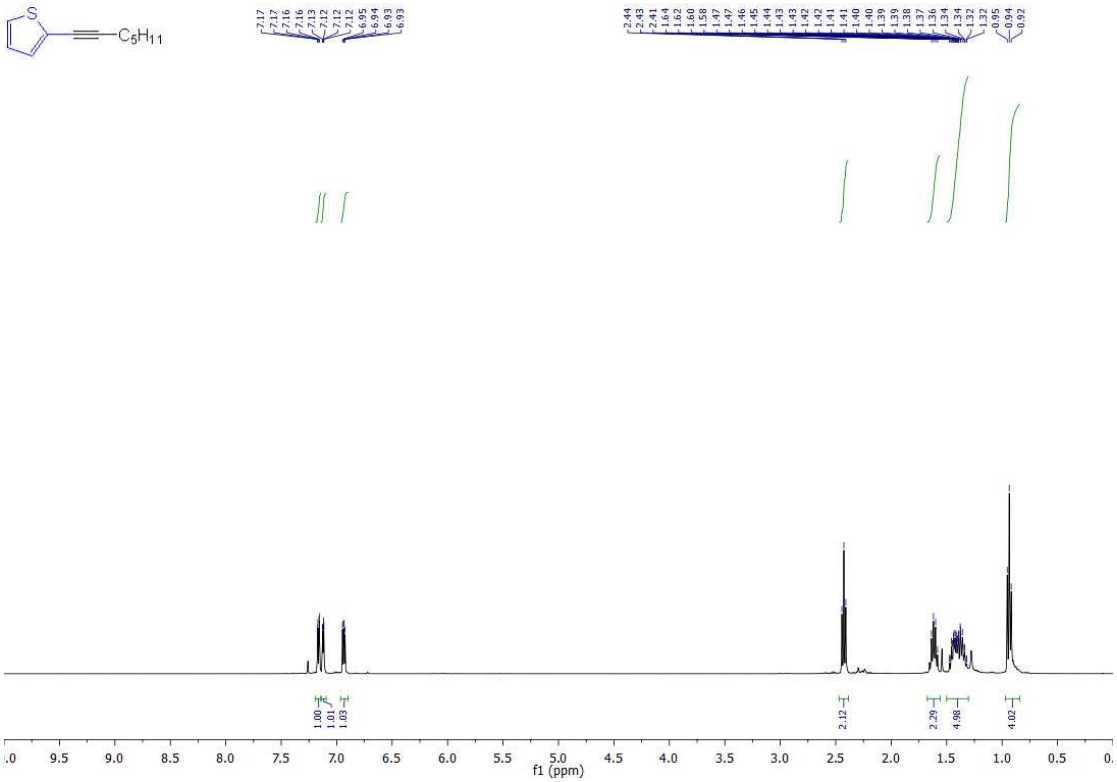
^{1}H NMR spectrum of 4-(hept-1-yn-1-yl)aniline (**4g**)



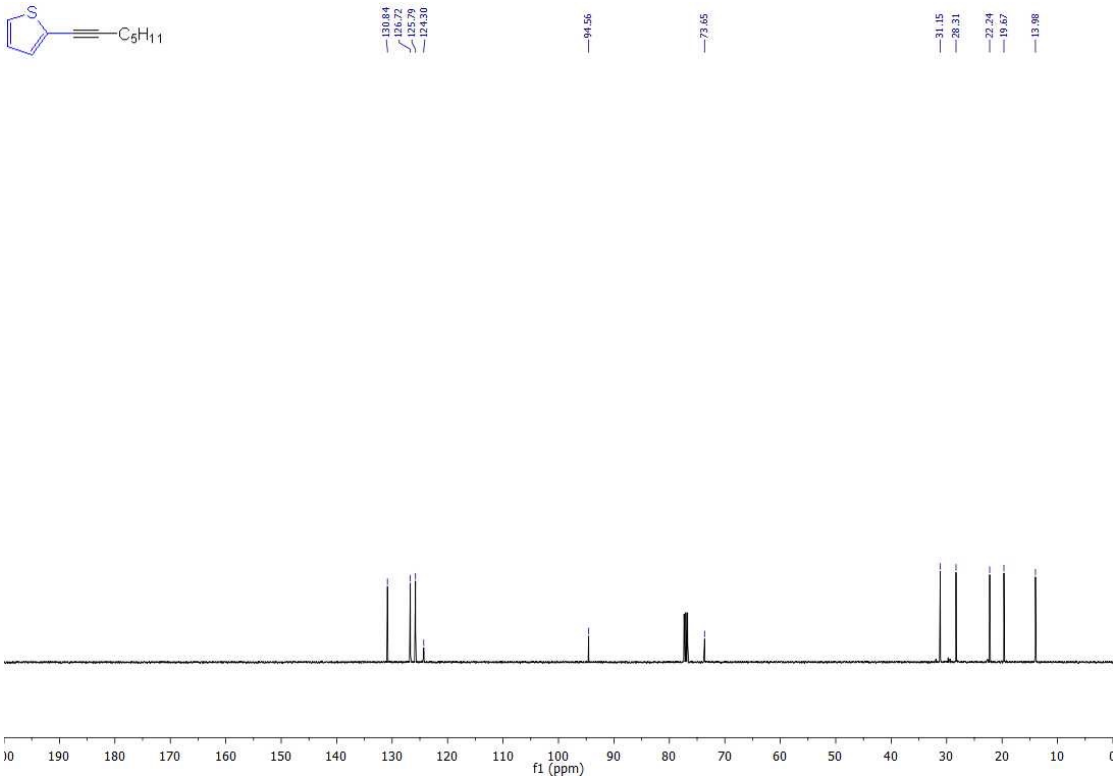
^{13}C NMR spectrum of 4-(hept-1-yn-1-yl)aniline (**4g**)



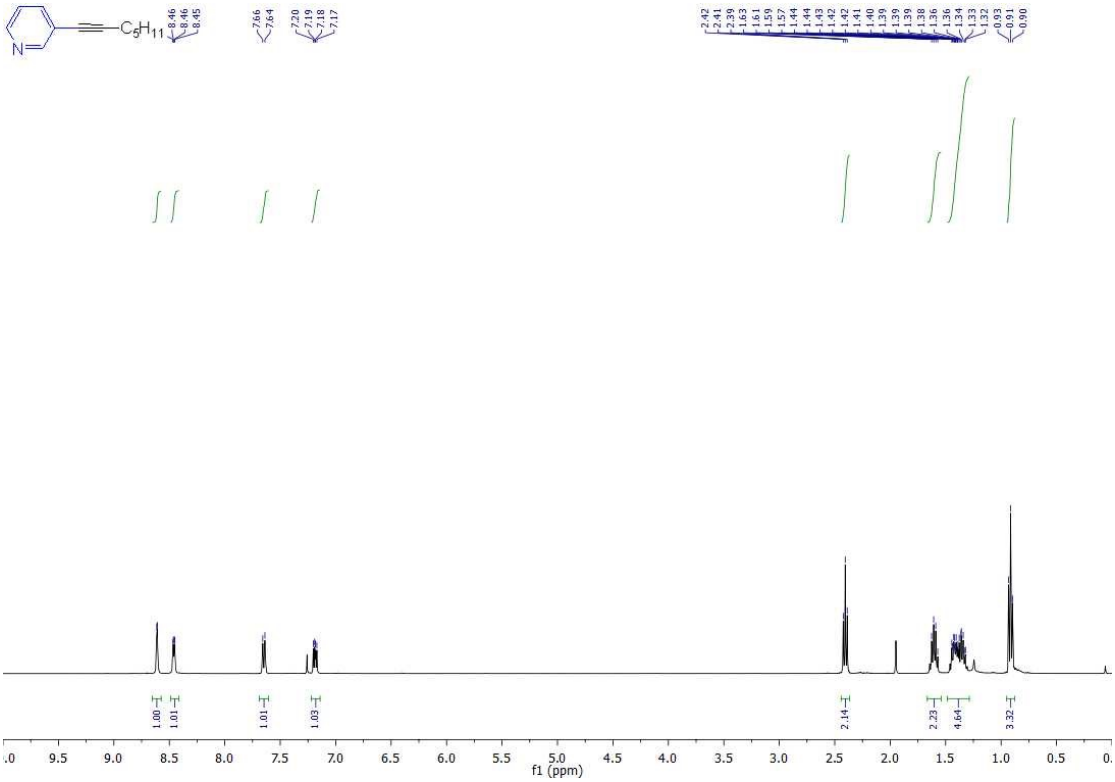
^{1}H NMR spectrum of 2-(hept-1-yn-1-yl)thiophene (**4h**)



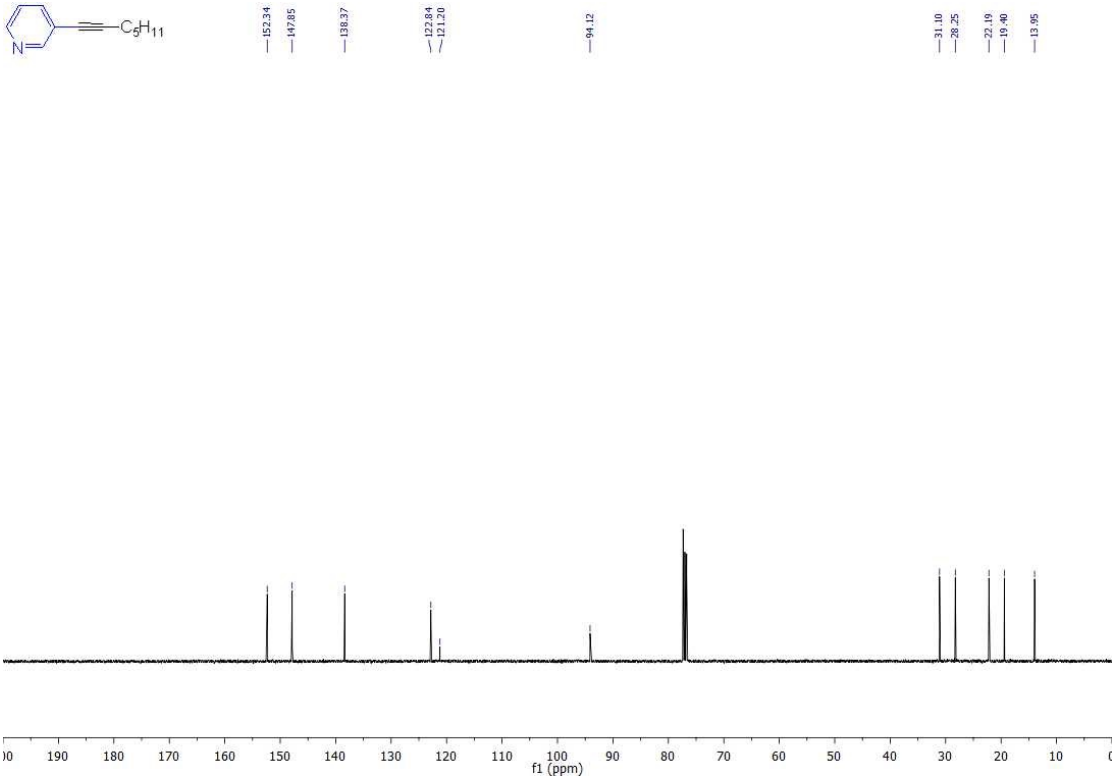
^{13}C NMR spectrum of 2-(hept-1-yn-1-yl)thiophene (**4h**)



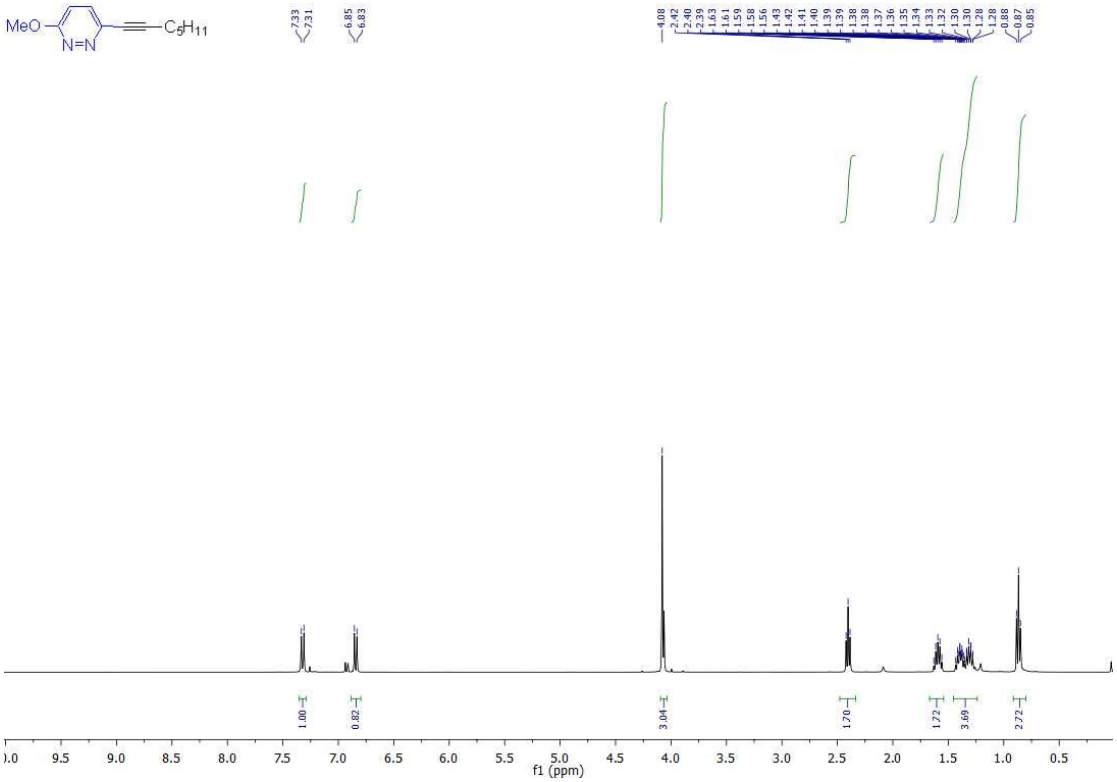
^{1}H NMR spectrum of 3-(hept-1-yn-1-yl)pyridine (**4i**)



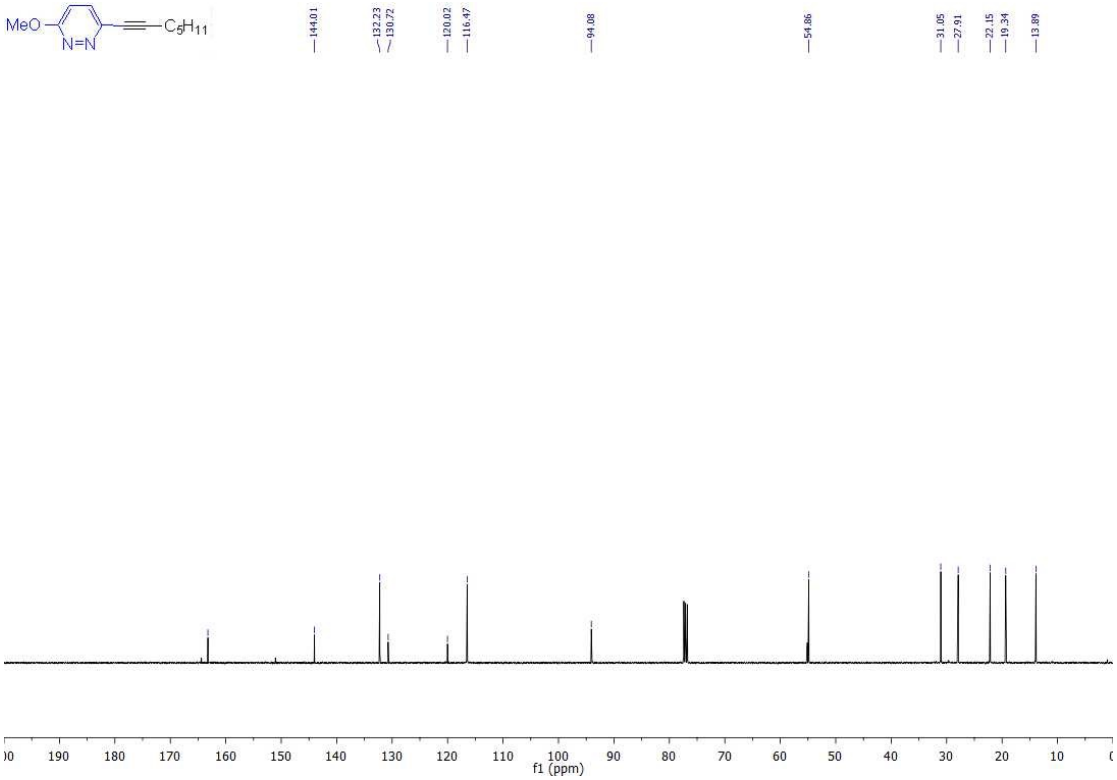
^{13}C NMR spectrum of 3-(hept-1-yn-1-yl)pyridine (**4i**)



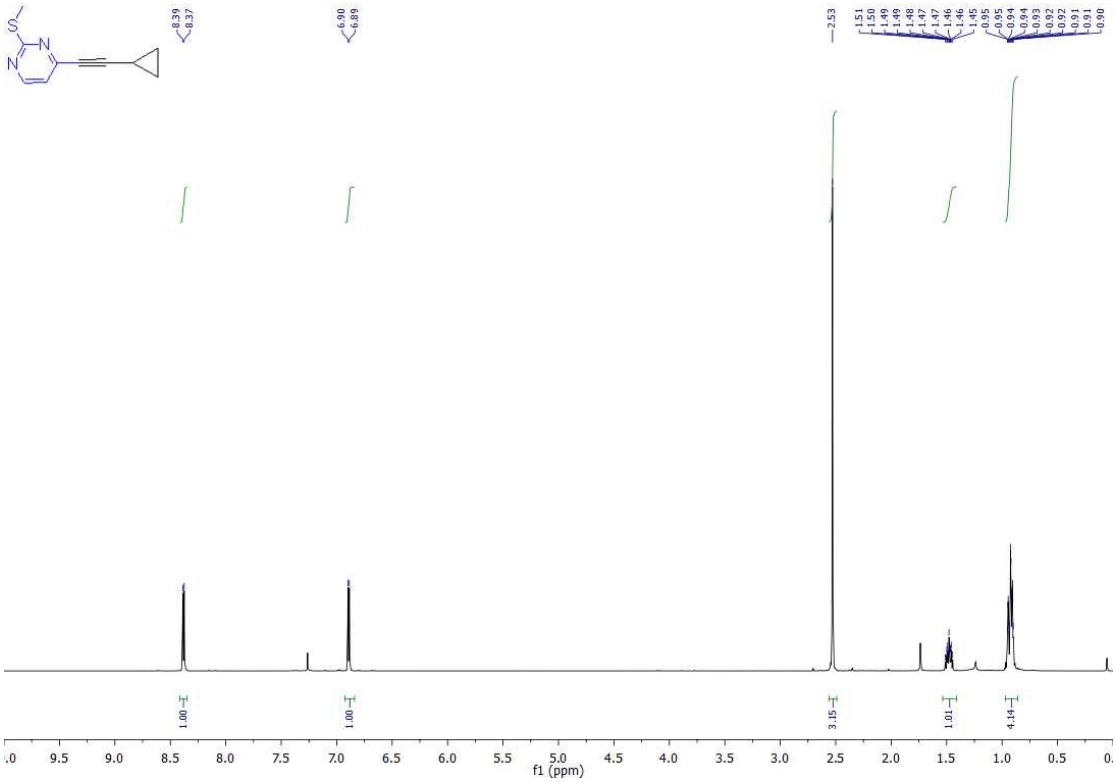
^{1}H NMR spectrum of 3-(hept-1-yn-1-yl)-6-methoxypyridazine (**4j**)



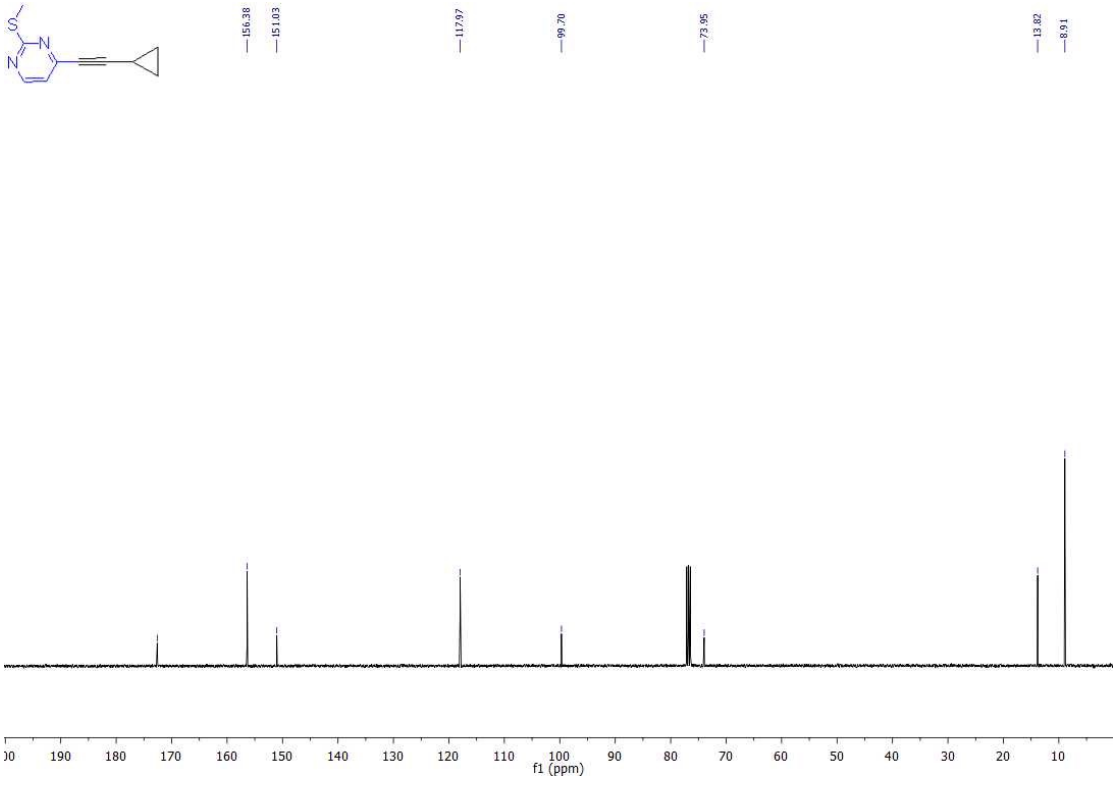
^{13}C NMR spectrum of 3-(hept-1-yn-1-yl)-6-methoxypyridazine (**4j**)



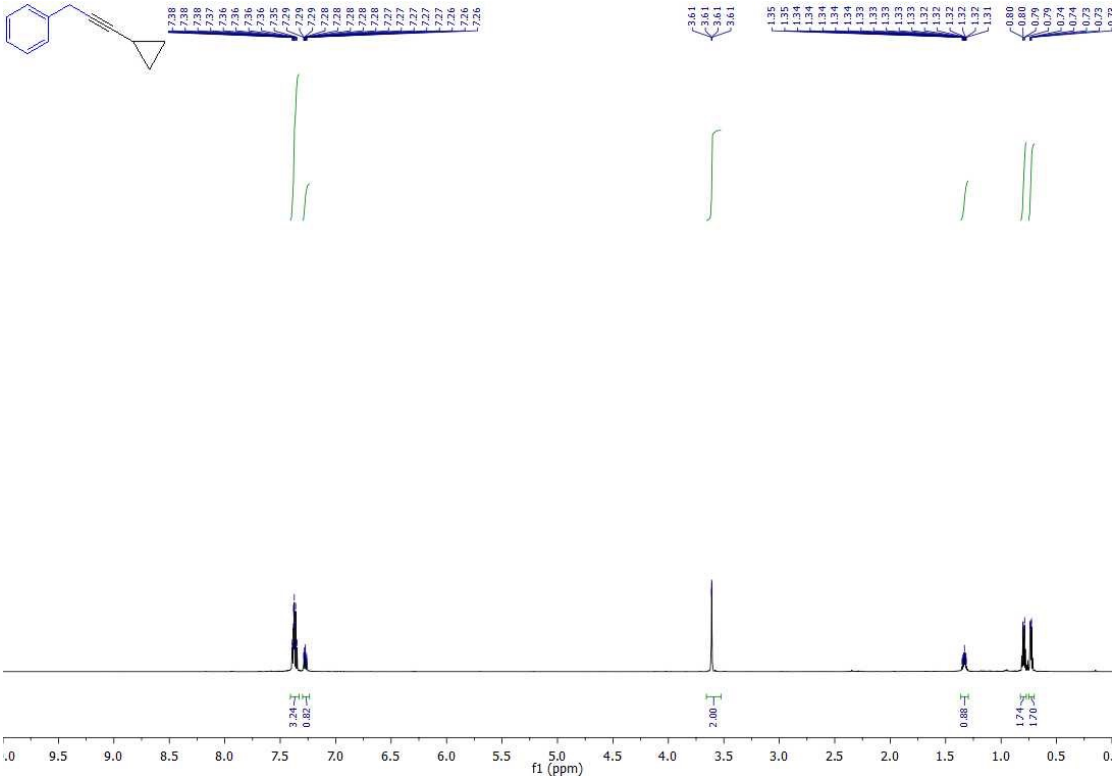
^{1}H NMR spectrum of 4-(cyclopropylethynyl)-2-(methylthio)pyrimidine (**4k**)



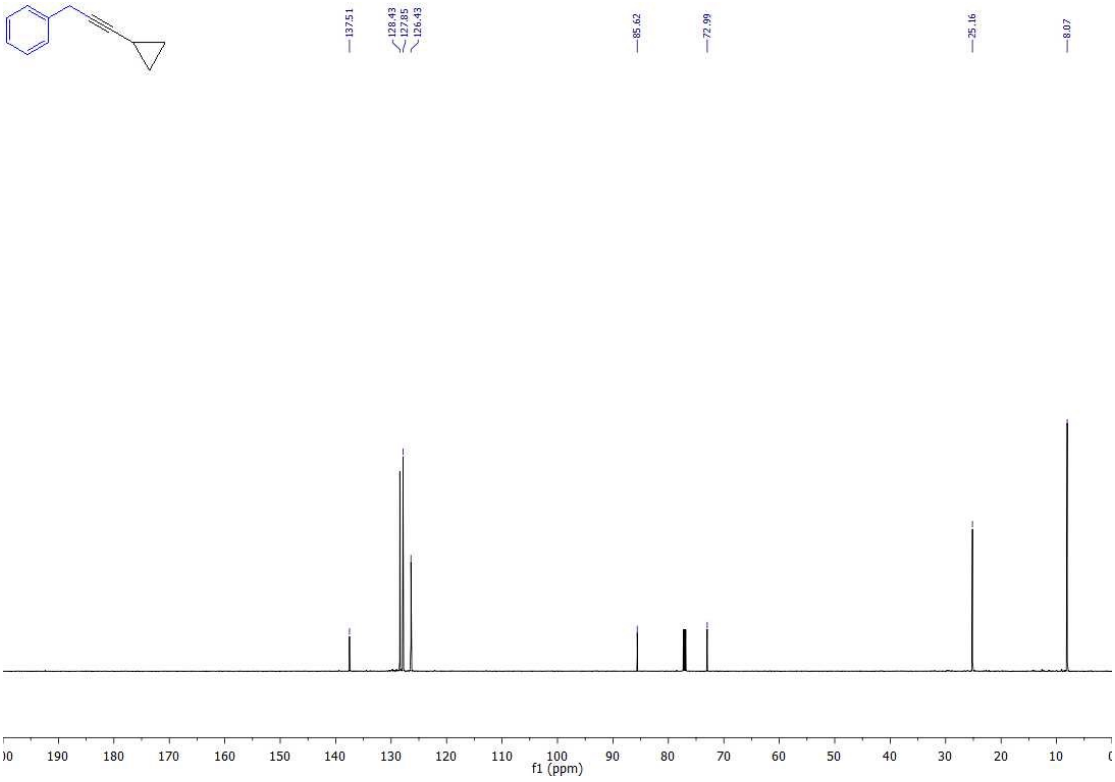
^{13}C NMR spectrum of 4-(cyclopropylethynyl)-2-(methylthio)pyrimidine (**4k**)



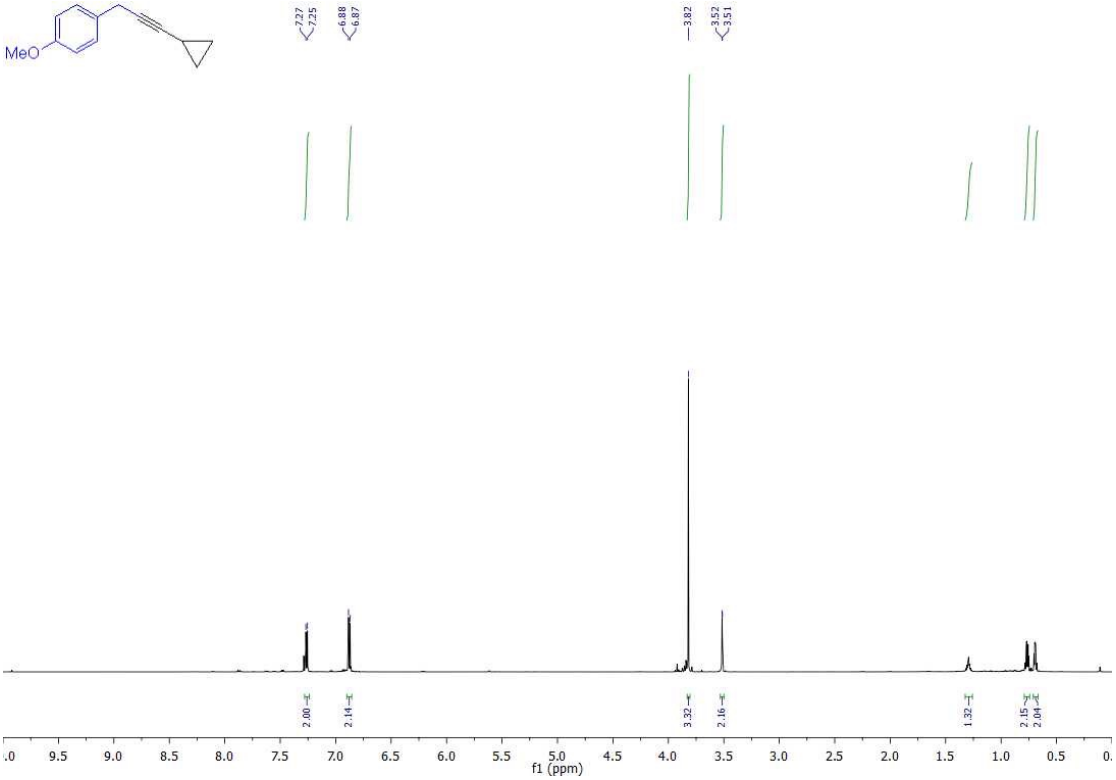
^{1}H NMR spectrum of (3-cyclopropylprop-2-yn-1-yl)benzene (**4l**)



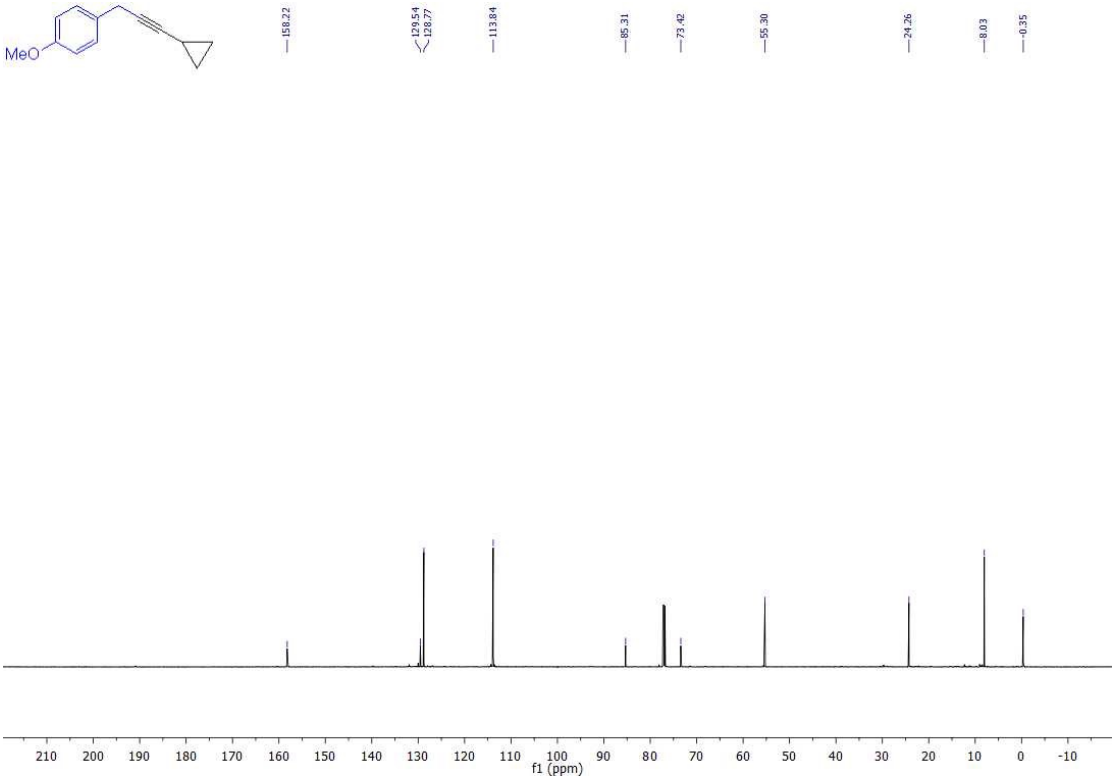
^{13}C NMR spectrum of (3-cyclopropylprop-2-yn-1-yl)benzene (**4l**)



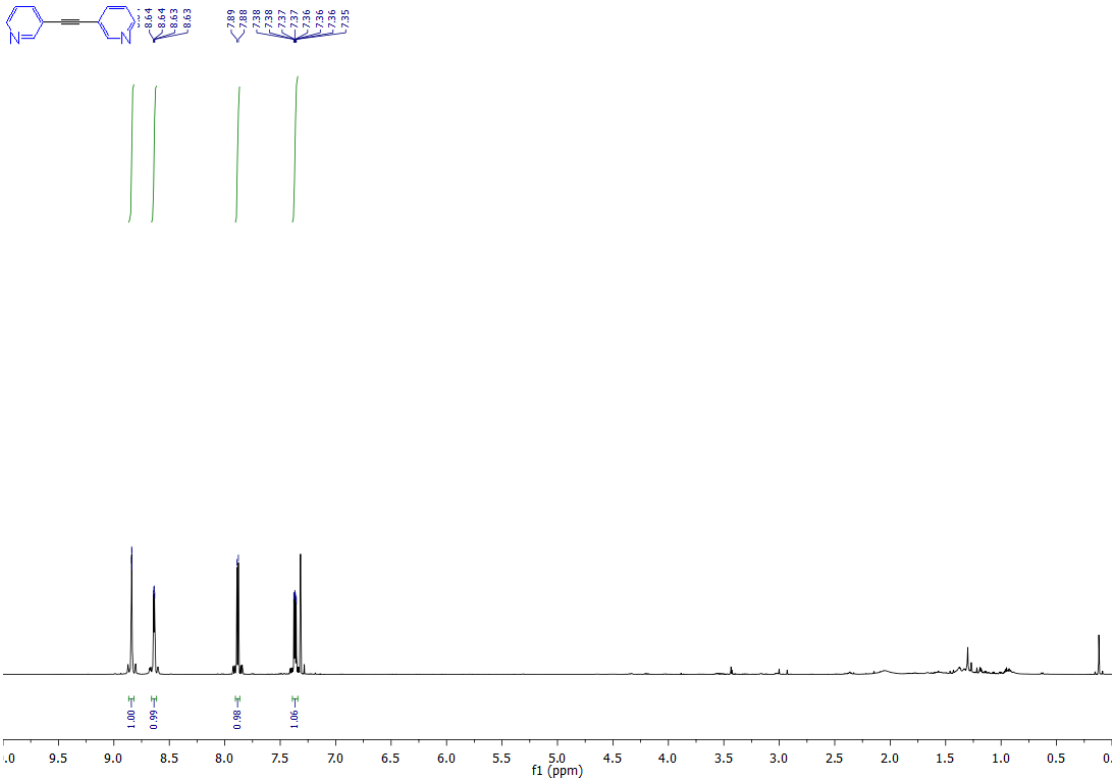
^{1}H NMR spectrum of 1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (**4m**)



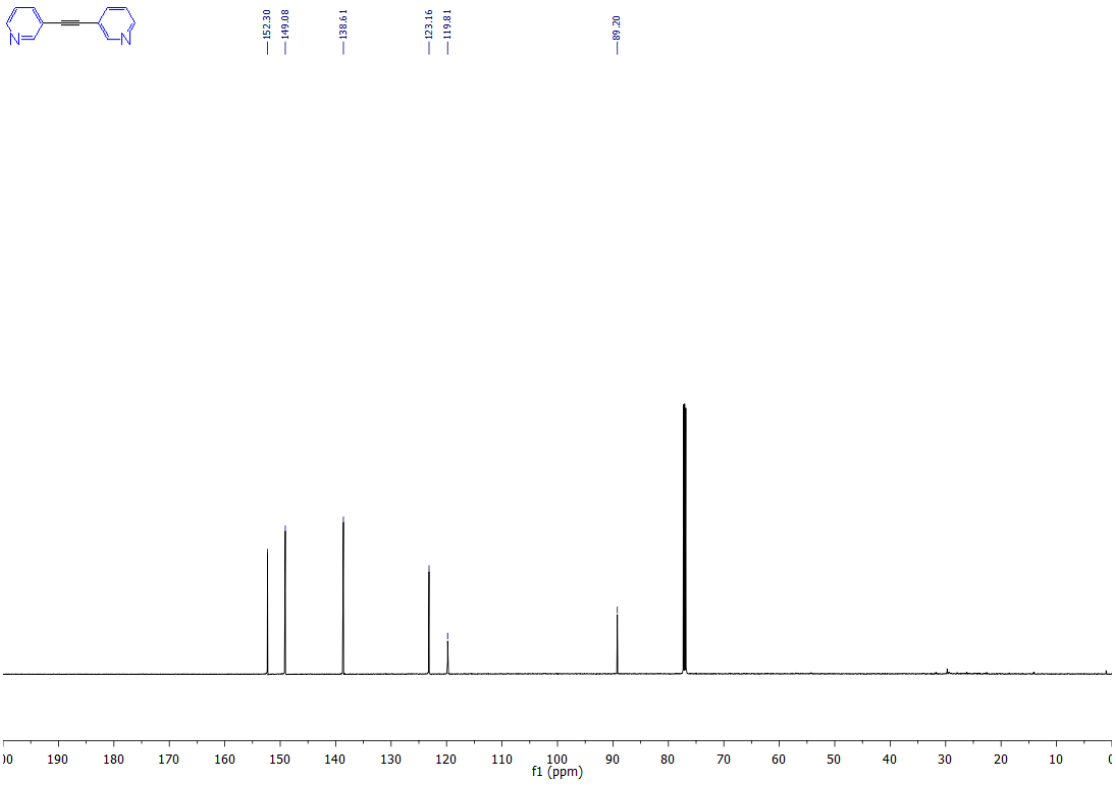
^{13}C NMR spectrum of 1-(3-cyclopropylprop-2-yn-1-yl)-4-methoxybenzene (**4m**)



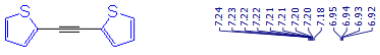
^{1}H NMR spectrum of 1,2-di(pyridin-3-yl)ethyne (**6a**)

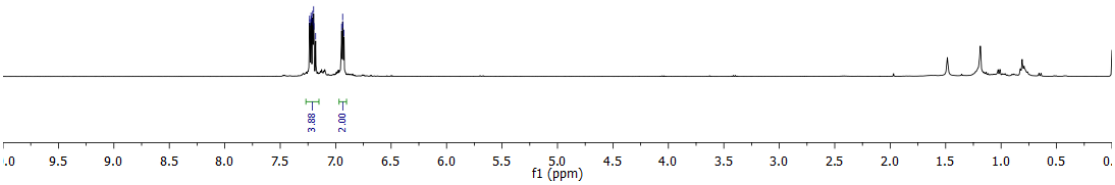


^{13}C NMR spectrum of 1,2-di(pyridin-3-yl)ethyne (**6a**)

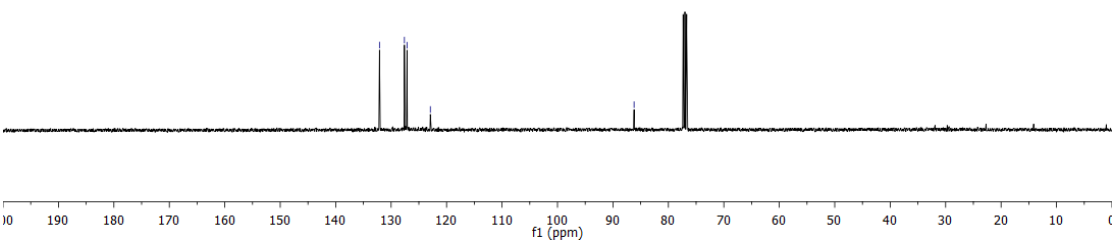


^{1}H NMR spectrum of 1,2-di(thiophen-2-yl)ethyne (**6b**)

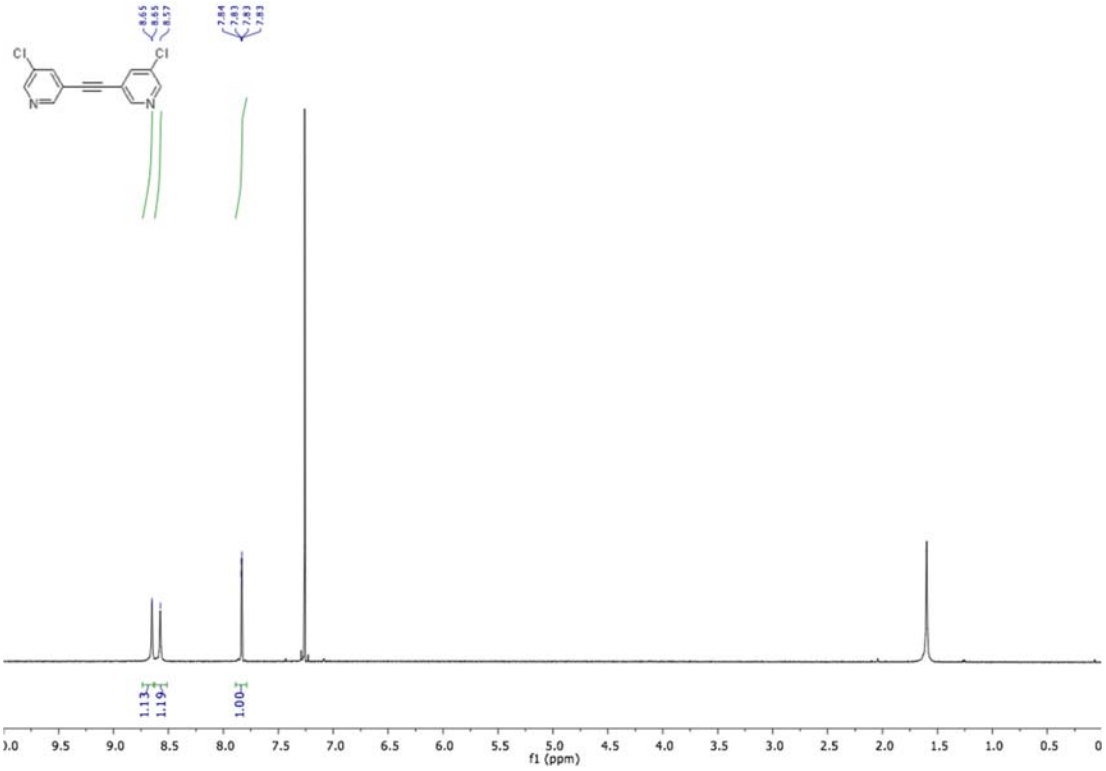




^{13}C NMR spectrum of 1,2-di(thiophen-2-yl)ethyne (**6b**)



^{1}H NMR spectrum of 1,2-bis(5-chloropyridin-3-yl)ethyne (**6c**)



^{13}C NMR spectrum of 1,2-bis(5-chloropyridin-3-yl)ethyne (**6c**)