Synthesis of 1,2-disubstituted acetylenes via copper-catalyzed Suzuki coupling reaction of organoboronic acids with 1,1-dibromo-1-alkenes

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Supporting Information

Content

General Information All the solvents and commercially available reagents were purchased from commercial sources and used directly. ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ at room temperature on a Varian INOVA-400 spectrometer (400 MHz ^{1}H) or Bruker spectrometer (400 MHz ^{1}H). The chemical-shifts scale is based on TMS. Products were purified by flash column chromatography on 200-300 mesh silica gel.

Typical procedure for the preparation of 1,1-dibromo-1-alkenes

Typical procedure for the preparation of 1,1-dibromo-1-alkenes: To a solution of 4-methoxybenzaldehyde (2.3 g, 16.5 mmol), carbon tetrabromide (7.1 g, 21.4 mmol) in dichloromethane (100 mL) at 0 °C was added slowly a solution of PPh_3 (10.8 g, 41.2 mmol) in dichloromethane (40 mL). The reaction mixture was stirred until complete conversion of aldehyde was realized and the mixture was concentrated. The resulting crude product was purified by flash chromatography over SiO_2 with petroleum ether to afford the title compound **1a** as a white solid (4.3 g, 90%).[1]

Characterization data of 1,1-dibromo-1-alkenes

1-(2,2-dibromovinyl)-4-methoxybenzene (1a)[1]

White solid, mp 37-38 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, J = 8.8 Hz, 2H), 7.40 (s, 1H), 6.88 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H).

(2,2-dibromovinyl)benzene (1l)[1]

Pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.51 (m, 2H), 7.47 (s, 1H), 7.36-7.34 (m, 3H).

2-(2,2-dibromovinyl)naphthalene (1m)[1]

White solid, mp 94-95 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (s, 1H), 7.86-7.82 (m, 3H), 7.65-7.63 (m, 2H), 7.52-7.49 (m, 2H).

1-(2,2-dibromovinyl)-2,4-dimethoxybenzene (1n)

White solid, mp 45-46 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (dd, J = 0.4, 8.8 Hz, 1H), 6.49 (dd, J = 2.4, 8.4 Hz, 1H), 7.53 (s, 1H), 6.40 (d, J = 2.4 Hz, 1H), 3.81 (s, 3H), 3.79 (s, 3H).

1-(2,2-dibromovinyl)-4-methoxybenzene (1o)[2]

Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (s, 1H), 7.43 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 2.34 (s, 3H).

1-chloro-4-(2,2-dibromovinyl)benzene (1p)[1]

Light brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, J = 8.4 Hz, 2H), 7.43 (s, 1H). 7.34 (d, J = 8.8 Hz, 2H).

1-(2,2-dibromovinyl)-4-nitrobenzene (1q)[1]

Yellow solid, mp 93-94 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, J = 8.8 Hz, 2H), 7.71 (d, J = 8.4 Hz, 2H), 7.26 (s, 1H).

2,2-dibromovinyl-2-pyridine (1r)[1]

Light green liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (d, J = 4.4 Hz, 1H), 7.76-7.72 (m, 2H), 7.65 (s, 1H), 7.26-7.23 (m, 1H).

2,2-dibromovinyl-2-furan (1s)[1]

Pale brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, J = 1.6 Hz, 1H), 7.40 (s, 1H), 6.95 (d, J = 4.0 Hz, 1H), 6.47-6.45 (m, 1H).

1,1-dibromo-4-phenylbutene (1t)[1]

Light yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.29 (m, 2H), 7.24-7.19 (m, 3H), 6.40 (t, J = 7.2 Hz, 1H), 2.74 (t, J = 7.7 Hz, 2H), 2.45-2.41 (m, 2H).

Typical procedure for the products

General procedure for the one-pot preparation of 1,2-disubstituted acetylenes from 1,1-dibromo-1-alkenes: Under a nitrogen atmosphere, an over-dried Schlenk tube with a magnetic stirring bar was charged with 1,1-dibromo-1-alkenes (0.50 mmol), organoboron compound (1.0 mmol), CuI (0.05 mmol), 8-hydroxyquinoline (0.10 mmol), K_3PO_4 (2.0 mmol), and ethanol (2.0 mL). After the mixture was heated at 100 ^{o}C for 12 h, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on a silica gel (petroleum ether as eluting agent) to give the product.

Characterization data of the products

(4-methoxyphenyl)phenylacetylene (3a)[3]

White solid; yield: 92%; mp 51-53 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.45 (m, 4H), 7.36-7.30 (m, 3H), 6.88 (dd, J = 8.7, 2.1 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.53, 133.01, 131.40, 128.28, 127.91, 123.50, 115.29, 113.94, 89.30, 88.01, 55.28; IR (KBr): 3053, 2216, 1246, 1028 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{15}H_{12}O$ 208.0888; found 208.0887.

di(4-methoxylphenyl)acetylene (3b)[4]

White solid; yield: 96%; mp 145-147 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 8.8 Hz, 4H), 6.87 (d, J = 8.8 Hz, 4H), 3.82 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.36, 131.83, 114.61, 112.89, 86.89, 54.25; IR (KBr): 2840, 2254, 1244, 1026 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{16}H_{14}O_2$ 238; found 238.

1-tert-butyl-4-((4-methoxyphenyl)ethynyl)benzene (3c)[4]

White solid; yield: 95%; mp 122-124 ^{o}C; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.47-7.43 (m, 4H), 7.35 (d, J = 8.4 Hz, 2H), 6.87 (d, J = 8.4 Hz, 2H), 3.82 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.39, 151.11, 132.96, 131.11, 125.28, 120.46, 115.54, 113.89, 88.60, 88.11, 55.25, 34.73, 31.15; IR (KBr): 2930, 2310, 2254, 1160 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{19}H_{20}O$ 264; found 264.

4-methyl-4′-methoxyldiphenylacetylene (3d)[4]

White solid; yield: 94%; mp 126-128 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 9.2 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 3.80 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.44, 137.97, 132.93, 131.29, 129.04, 120.46, 115.55, 113.93, 88.62, 88.16, 55.24, 21.43; IR (KBr): 2920, 2211, 1246, 1028 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{16}H_{14}O$ 222.1123; found 222.1120.

1-methoxy-4-(3'-tolylethynyl)benzene (3e)[5]

White solid; yield: 92%; mp 43-45 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, J = 9.0 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.22 (t, J = 7.4 Hz, 1H), 7.12 (d, J = 7.9 Hz, 1H), 6.87 (d, J = 8.8 Hz, 2H), 3.82 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.53, 137.93, 133.01, 132.01, 128.82, 128.49, 128.18, 123.36, 115.48, 113.96, 88.98, 88.20, 55.28, 21.21; IR (KBr): 2946, 2231, 1203, 1021 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{16}H_{14}O$ 222.1120; found 222.1121.

1-methoxy-4-(2′-tolylethynyl)benzene (3f)[6]

White solid; yield: 90%; mp 75-77 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.46 (m, 3H), 7.23-7.15 (m, 3H), 6.87 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 2.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.53, 139.92, 132.90, 131.62, 129.38, 127.94, 125.52, 123.30, 115.65, 113.97, 93.30, 86.98, 55.27, 20.73; IR (KBr): 2931, 2224, 1594, 1245, 1029 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{16}H_{14}O$ 222.1124; found 222.1123.

(*p*-fluorophenyl)(*p*-anisyl)acetylene (3g)[7]

White solid; yield: 86%; mp 83-85 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.45 (m, 4H), 7.36 (d, J = 8.4 Hz, 2H), 6.88 (d, J = 8.4 Hz, 2H,) 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.48, 161.00, 159.60, 133.20 (d, J=8.3 Hz), 119.63, 115.55 (d, J = 22.1 Hz), 115.09, 113.97, 88.94, 86.93, 55.27; IR (KBr): 2923, 2217, 1287, 1029 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{15}H_{11}FO$ 226.0870; found 226.0872.

1-(4-methoxyphenyl)-2-[4-(trifluoromethyl)-phenyl]ethyne (3h)[8]

White solid; yield: 83%; mp 120-122 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.54 (m, 4H), 7.49 (d, J = 8.4 Hz, 2H,), 6.89 (d, J = 8.4 Hz, 2H,) 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.97, 133.24, 131.56, 129.50 (q, J = 33 Hz, C), 125.30 (d, J = 3 Hz, CH), 125.21(q, J = 272 Hz, CF_3), 114.53, 114.06, 91.87, 86.81, 55.31; IR (KBr): 2946, 2230, 1195, 1012 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{16}H_{11}F_3O$ 276.2560; found 276.2562.

(*p*-chlorophenyl)(*p*-anisyl)acetylene (3i)[9]

White solid; yield: 88%; mp 115-116 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.42 (m, 4H), 7.31 (d, J = 7.5 Hz, 2H), 6.88 (d, J = 8.2 Hz, 2H) 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.70, 132.81, 132.03, 131.59, 127.60, 121.03, 113.91, 112.99, 89.28, 85.93, 54.28; IR (KBr): 2937, 2217, 1176, 1012 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{15}H_{11}ClO$ 242.0498; found 242.0496.

1-bromo-4-((4-methoxyphenyl)ethynyl)benzene (3j)[4]

White solid; yield: 90%; mp 235-237 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47-7.45 (m, 4H), 7.03 (t, J = 8.6 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H) 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.76, 133.04, 132.82, 131.53, 122.55, 122.03, 114.95, 114.02, 90.52, 87.02, 55.28; IR (KBr): 2925, 2217, 1164, 1036 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{15}H_{11}BrO$ 286.0072; found 286.0073.

1-(2-(4-methoxyphenyl)ethynyl)naphthalene (3k)[6]

Yellow solid; yield: 91%; mp 60-62 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (d, J = 8.2 Hz, 1H), 7.85 (d, J = 8.2 Hz, 1H), 7.81 (d, J = 8.3 Hz, 1H), 7.73 (dd, J = 7.2, 1.1 Hz, 1H), 7.61-7.55 (m, 3H), 7.52

(dt, J = 7.6, 1.3 Hz, 1H), 7.40 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 8.9 Hz, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.65, 133.15, 133.07, 130.01, 128.36, 128.24, 126.62, 126.32, 126.22, 125.26, 121.16, 115.41, 114.01, 94.31, 86.16, 55.27; IR (KBr): 2942, 2225, 1235, 1042 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{19}H_{14}O$ 258; found 258.

diphenylacetylene (3l)[3]

White solid; yield: 90%; mp 59-61 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.52 (m, 4H), 7.35-7.33 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 131.56, 128.31, 128.22, 123.18, 89.31; IR (KBr): 2913, 2219, 1213, 1026 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{14}H_{10}$ 178; found 178.

2-(phenylethynyl)naphthalene (3m)[4]

White solid; yield: 88%; mp 105-106 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.82-7.79 (m, 3H), 7.59-7.57 (m, 3H), 7.50-7.47 (m, 2H), 7.37-7.35 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.96, 132.75, 131.63, 131.40, 128.36, 128.34, 128.26, 127.98, 127.75, 127.74, 126.64, 126.52, 123.22, 120.51, 89.76, 89.70; IR (KBr): 2923, 2218, 1493, 1023, 965 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{18}H_{12}$ 228.0939; found 228.0935.

(2,4-dimethoxyphenyl)phenylacetylene (3n)[4]

Yellow oil; yield: 93%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, J = 8.8 Hz, 2H), 7.42 (d, J = 8.4 Hz, 1H), 7.32-7.30 (m, 3H), 6.48-6.45 (m, 2H), 3.88 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.13, 161.04, 134.26, 131.43, 128.14, 127.73, 123.75, 104.85, 104.73, 98.34, 91.97, 85.69, 55.78, 55.38; IR (neat): 2926, 2227, 1493, 749 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{16}H_{14}O_2$ 238; found 238.

phenyl-*p*-tolyacetylene (3o)[3]

Colorless solid; yield: 91%; mp 69-71 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.51 (m, 2H), 7.43 (d, J = 8.0 Hz, 2H), 7.33-7.31 (m, 3H), 7.14(d, J = 8.0 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): δ 138.35, 131.50, 131.44, 129.08, 128.28, 128.04, 123.38, 120.08, 89.50, 88.67, 21.49; IR (KBr): 2923, 2216, 1493, 1023, 965 cm^{-1}; LRMS (EI): $[M]^+$ calcd for $C_{15}H_{12}$ 192; found 192.

(4-chlorophenyl)phenylacetylene (3p)[4]

White solid; yield: 86%; mp 80-82 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.51 (m, 2H), 7.46 (d, J = 8.4 Hz, 2H), 7.36-7.34 (m, 3H), 7.32(d, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.20, 132.77, 131.56, 128.66, 128.46, 128.37, 122.85, 121.71, 90.26, 88.20; IR (KBr): 3048, 2228, 1486, 1024, 973 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{14}H_9Cl$ 212.6705; found 212.6707.

1-nitro-4-(phenylethynyl)benzene (3q)[10]

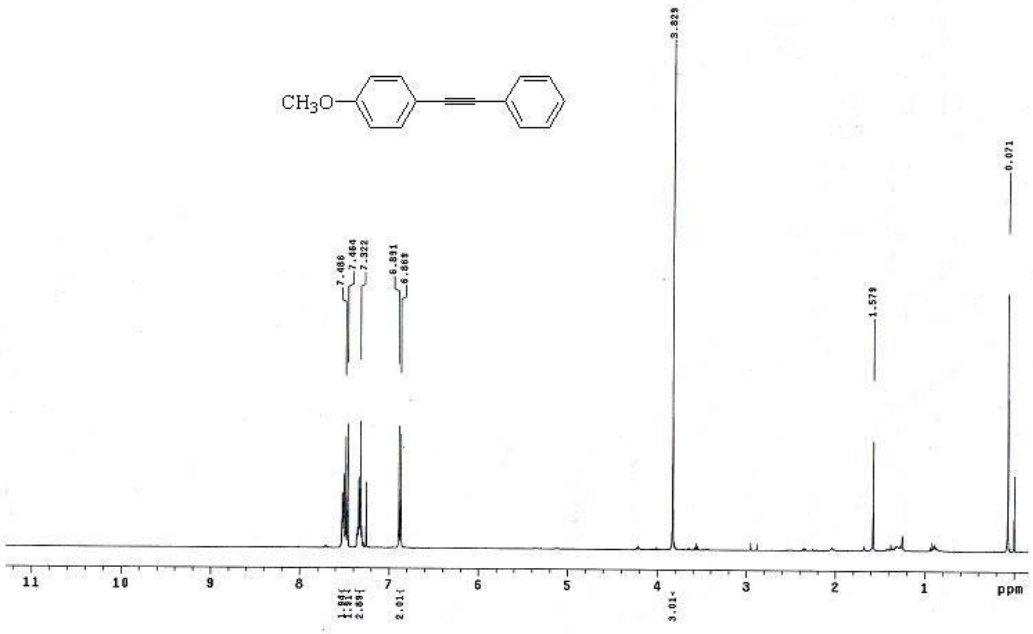
Colorless solid; yield: 75%; mp 121-123 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 8.8 Hz, 2 H), 7.67 (d, J = 9.2 Hz, 2 H), 7.57-7.55 (m, 2 H), 7.40-7.38 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.91, 132.24, 131.82, 130.23, 129.26, 128.52, 123.62, 122.04, 94.67, 87.52; IR (KBr): 2916, 2215, 1357, 1035, 931 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{14}H_9NO_2$ 223.2379; found 223.2380.

2-(2-phenylethynyl)pyridine (3r)[11]

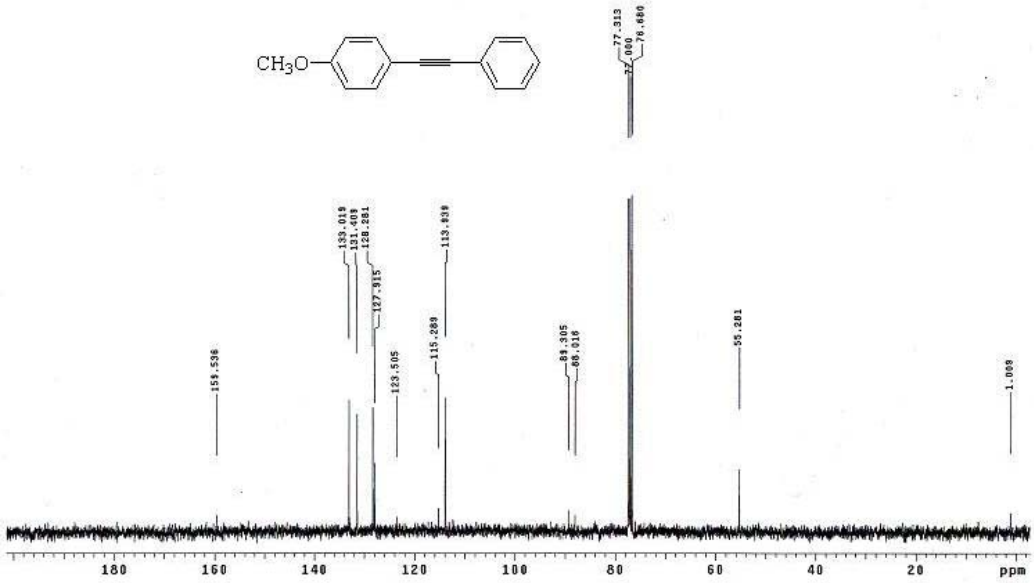
Oil; yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.78 (s, 1 H), 7.70-7.66 (m, 1 H), 7.62-7.54 (m, 2 H), 7.53 (d, J = 8.0 Hz, 1H), 7.38-7.35 (m, 3 H), 7.27-7.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 150.01, 143.37, 136.14, 131.98, 128.93, 128.34, 127.12, 122.72, 122.16, 89.15, 88.52; IR (neat): 3055, 2223, 1357, 1035, 931 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{13}H_9N$ 179.0731; found 179.0734.

2-(2-phenylethynyl)furan (3s)[12]

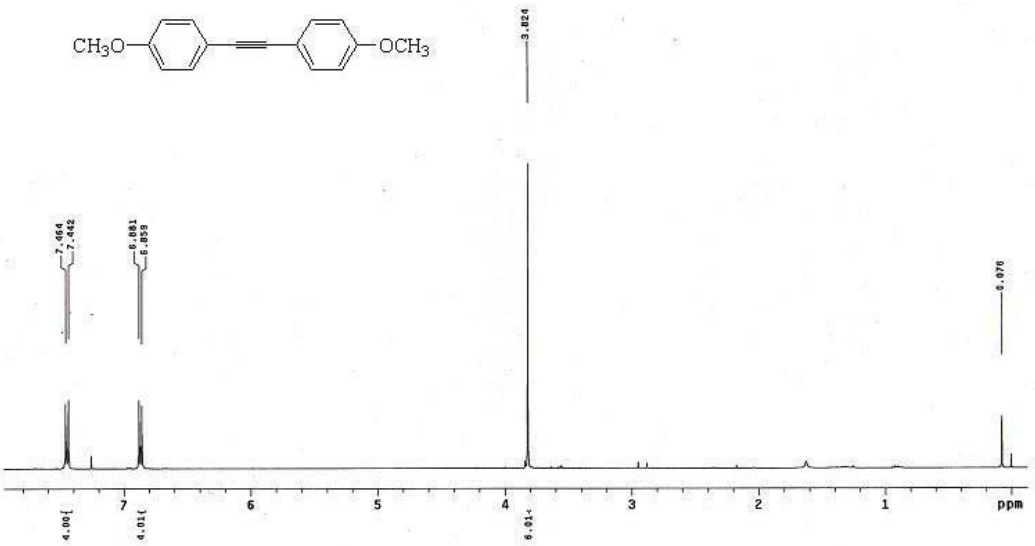
Colorless oil; yield: 91%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (dd, J = 6.1, 3.1 Hz, 2H), 7.43 (s, 1H), 7.34 (t, J = 3.7 Hz, 3H), 6.66 (d, J = 3.2 Hz, 1H), 6.44 (dd, J = 3.2, 1.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 143.62, 137.06, 131.38, 128.67, 128.36, 122.20, 115.22, 111.05, 93.20, 79.32; IR (neat): 3060, 2208, 1291, 1034, 929 cm^{-1}; HRMS (EI): $[M]^+$ calcd for $C_{12}H_8O$ 168.0575; found 168.0578.



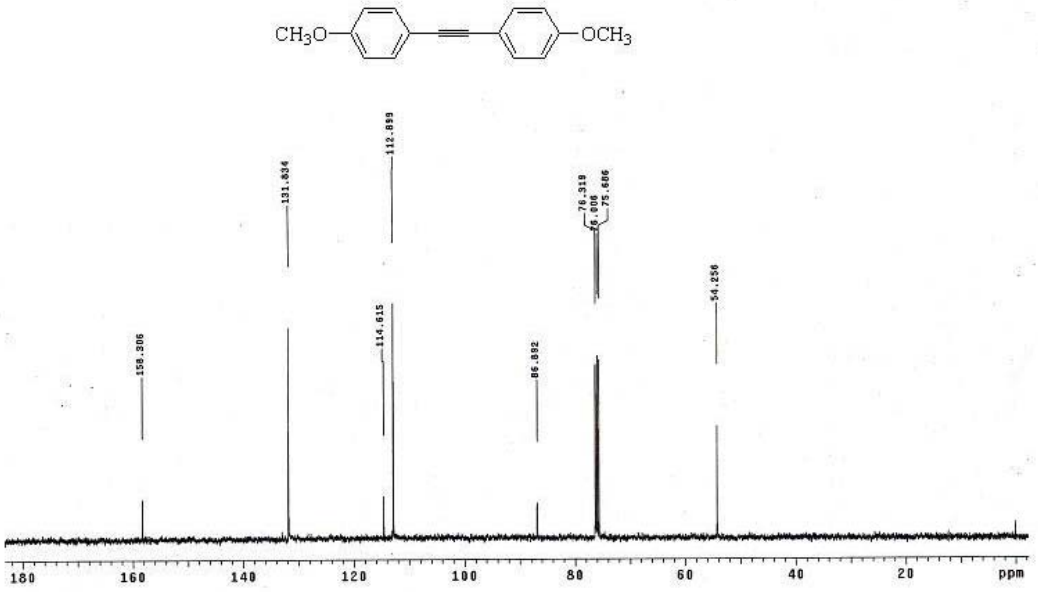
^{1}H NMR spectrum of the product 3a



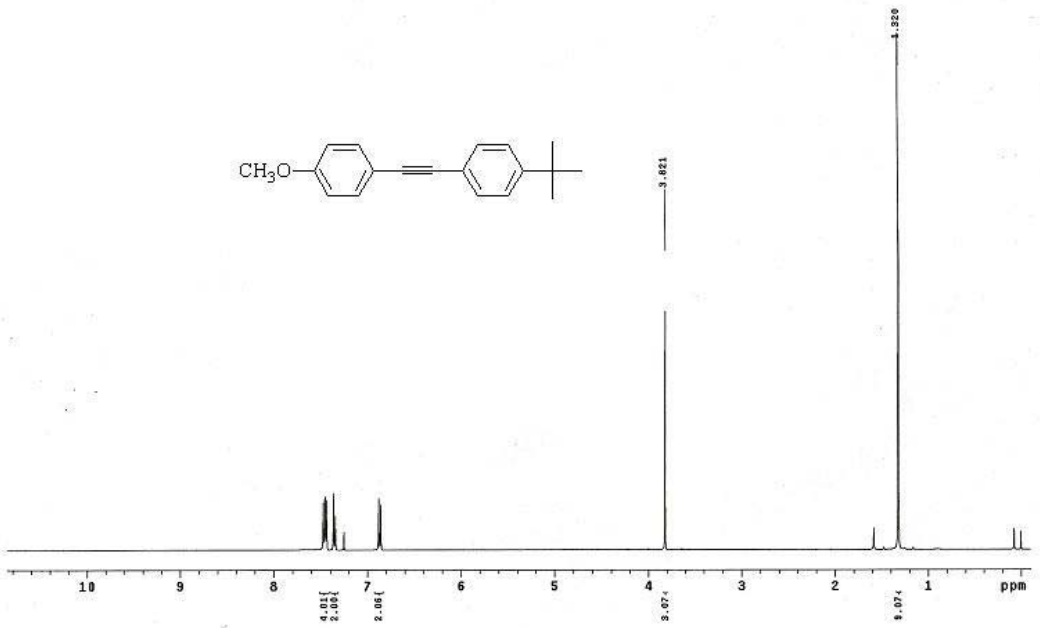
^{13}C NMR spectrum of the product 3a



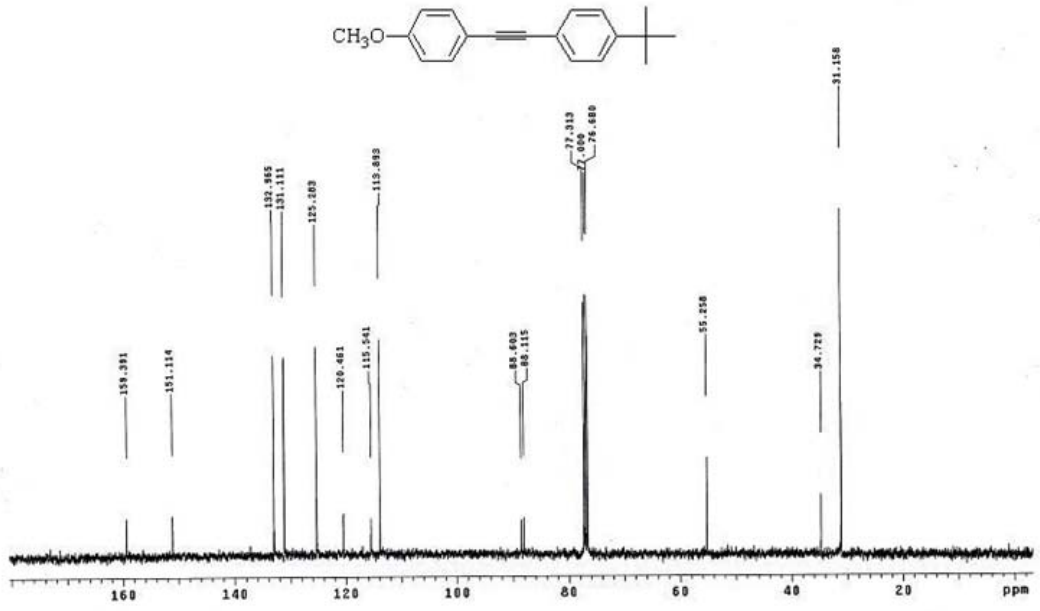
^{1}H NMR spectrum of the product 3b



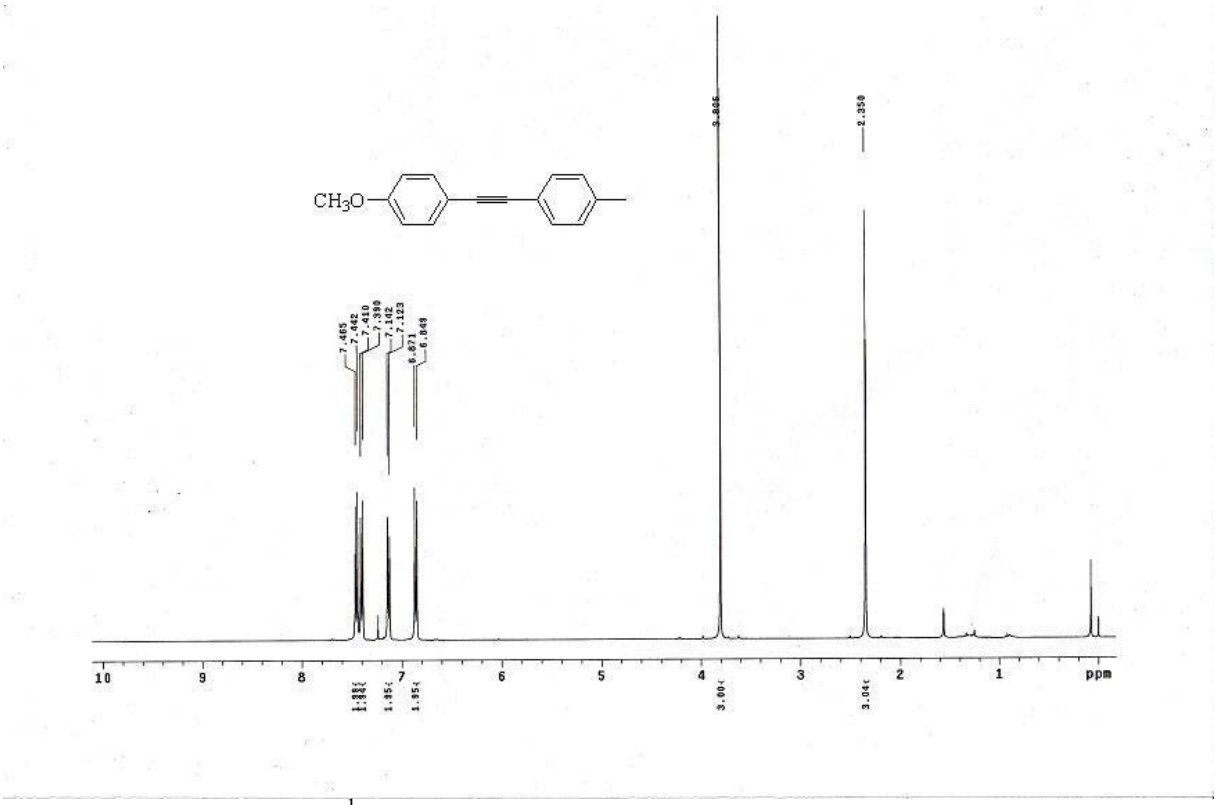
^{13}C NMR spectrum of the product 3b



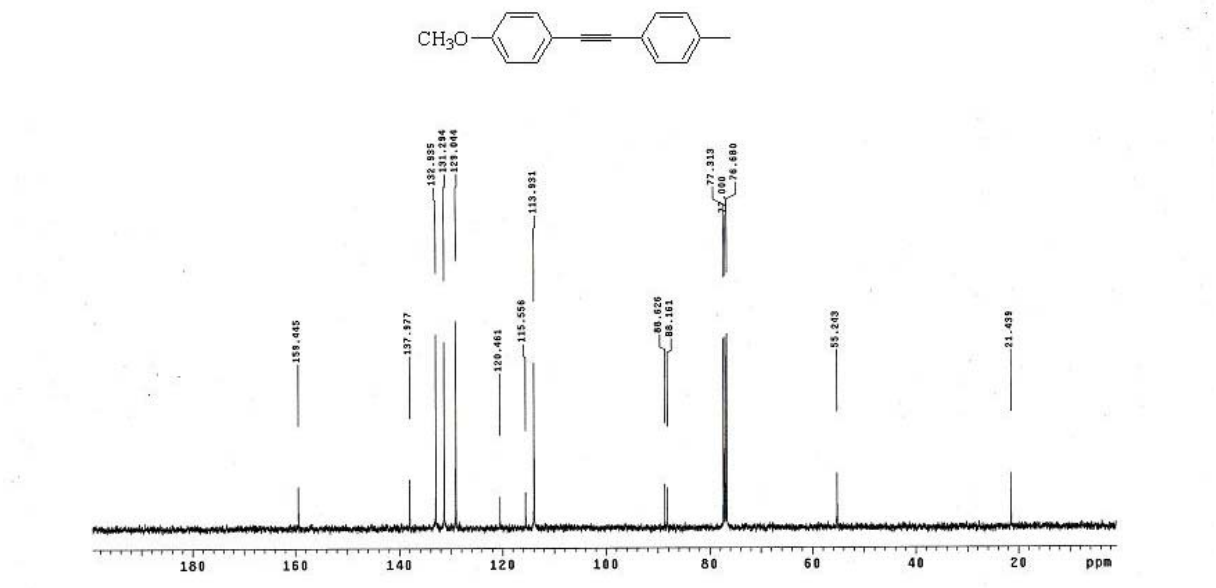
^{1}H NMR spectrum of the product 3c



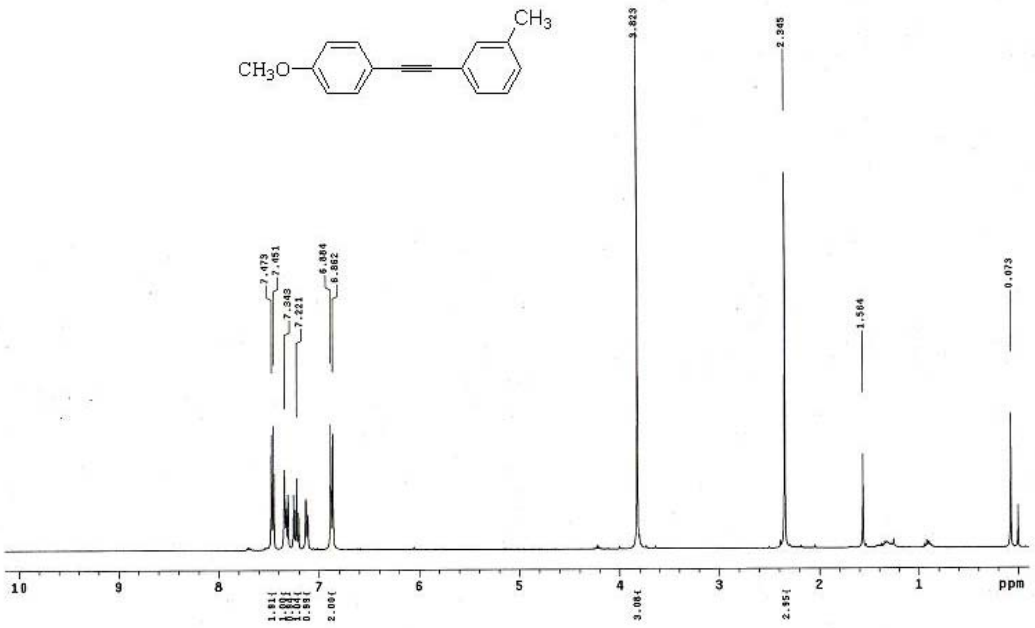
^{13}C NMR spectrum of the product 3c



^{1}H NMR spectrum of the product 3d



^{13}C NMR spectrum of the product 3d



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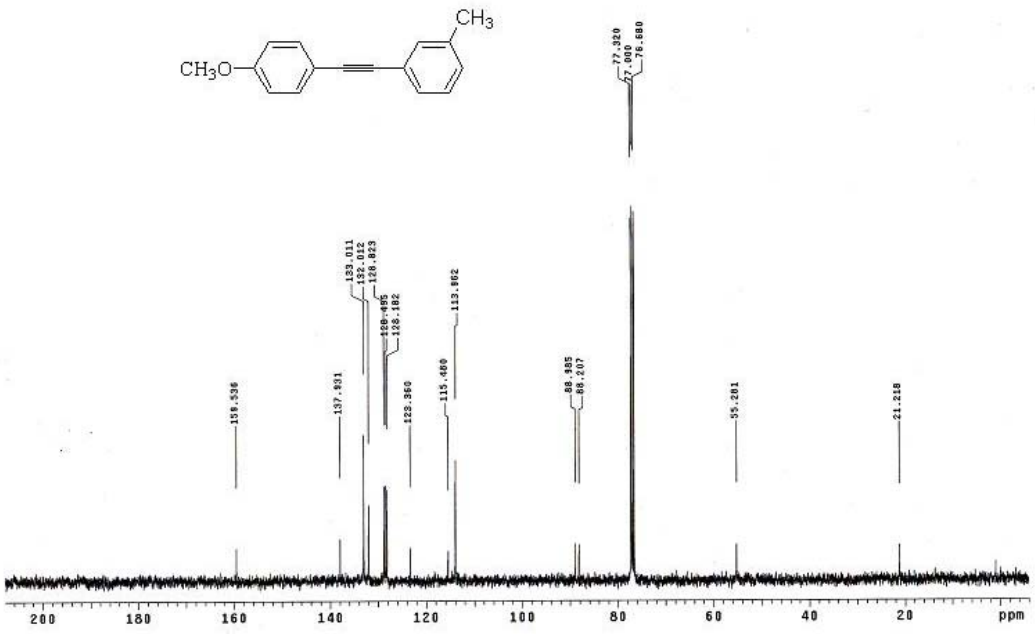
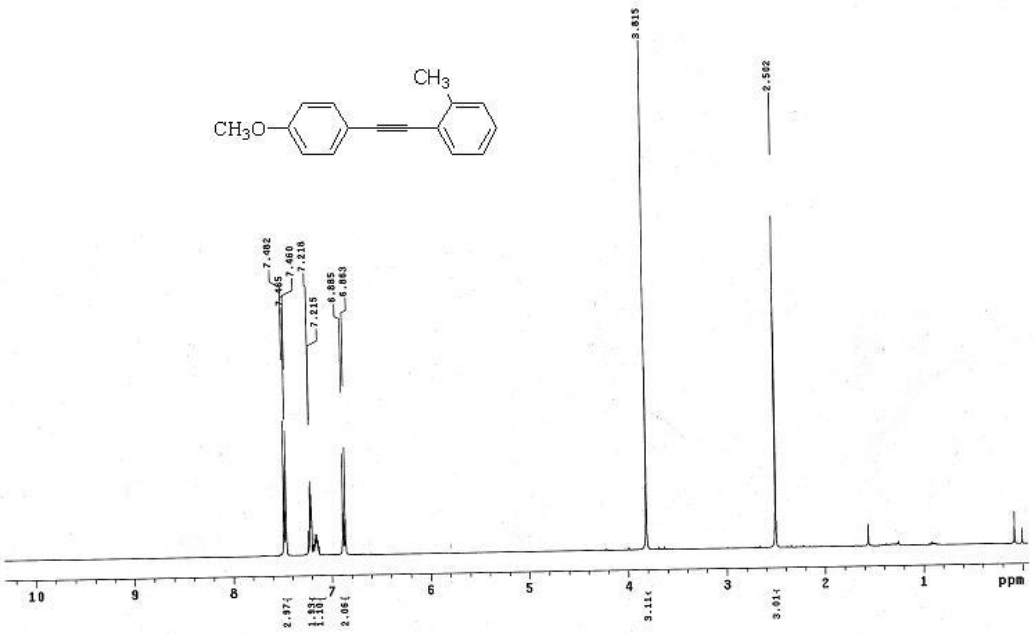
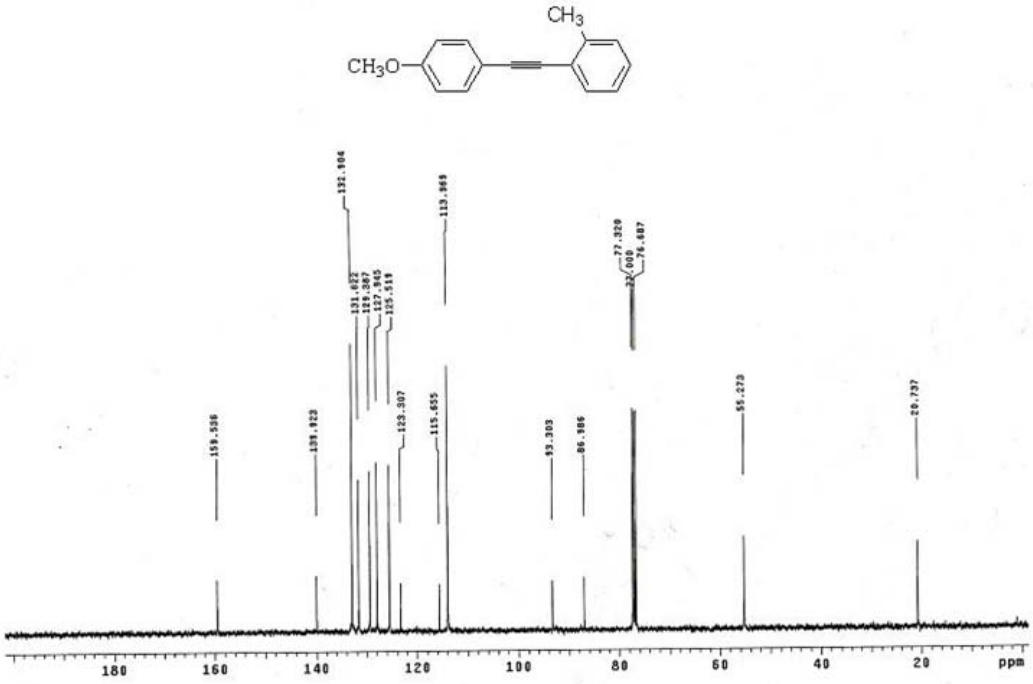


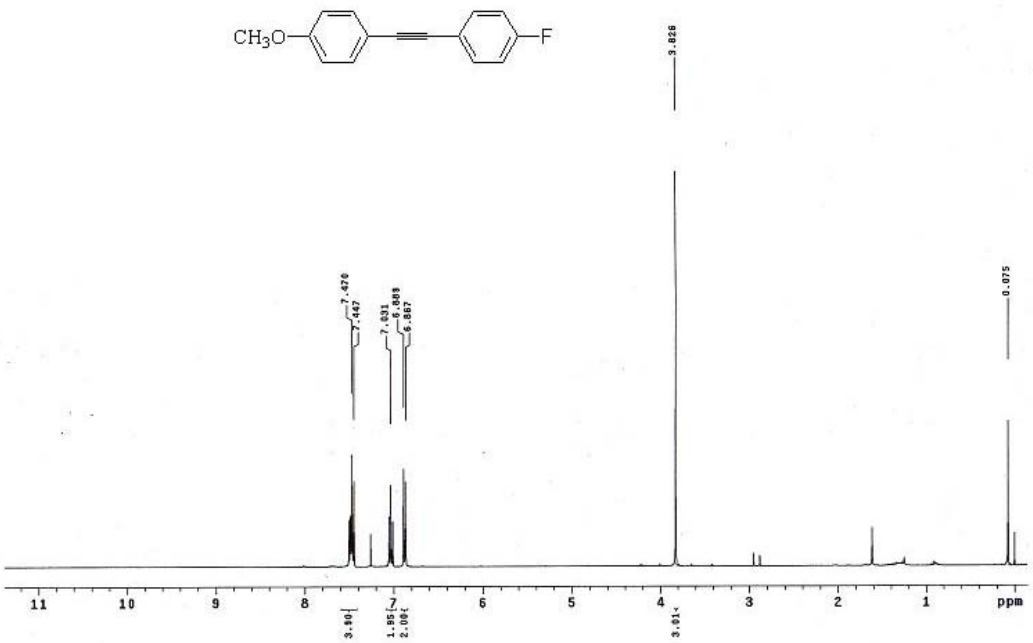
^{13}C NMR spectrum of the product 3e



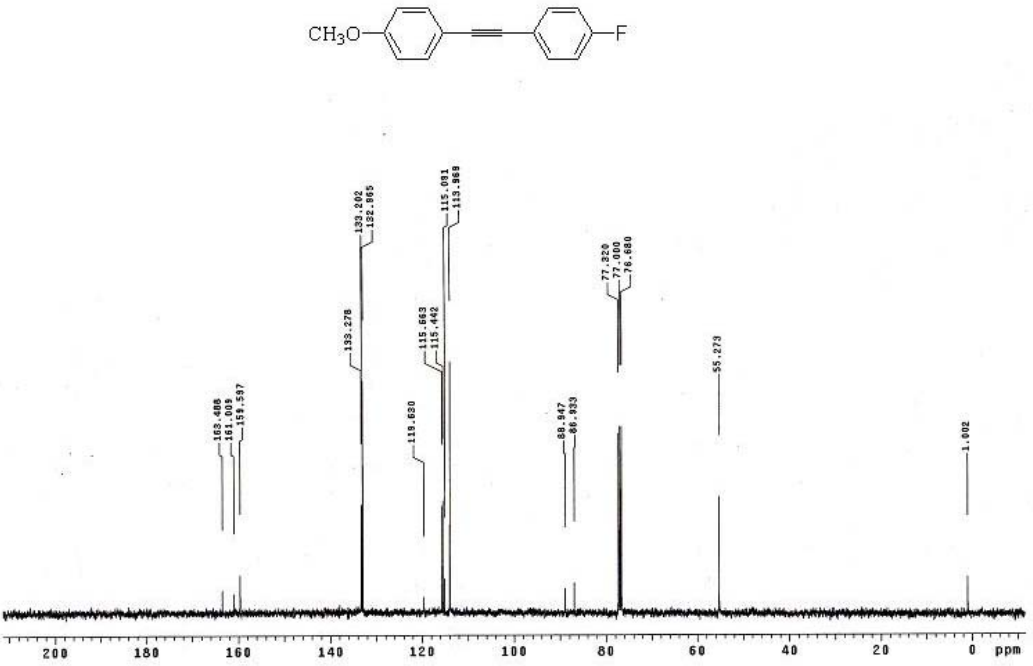
^{1}H NMR spectrum of the product 3f



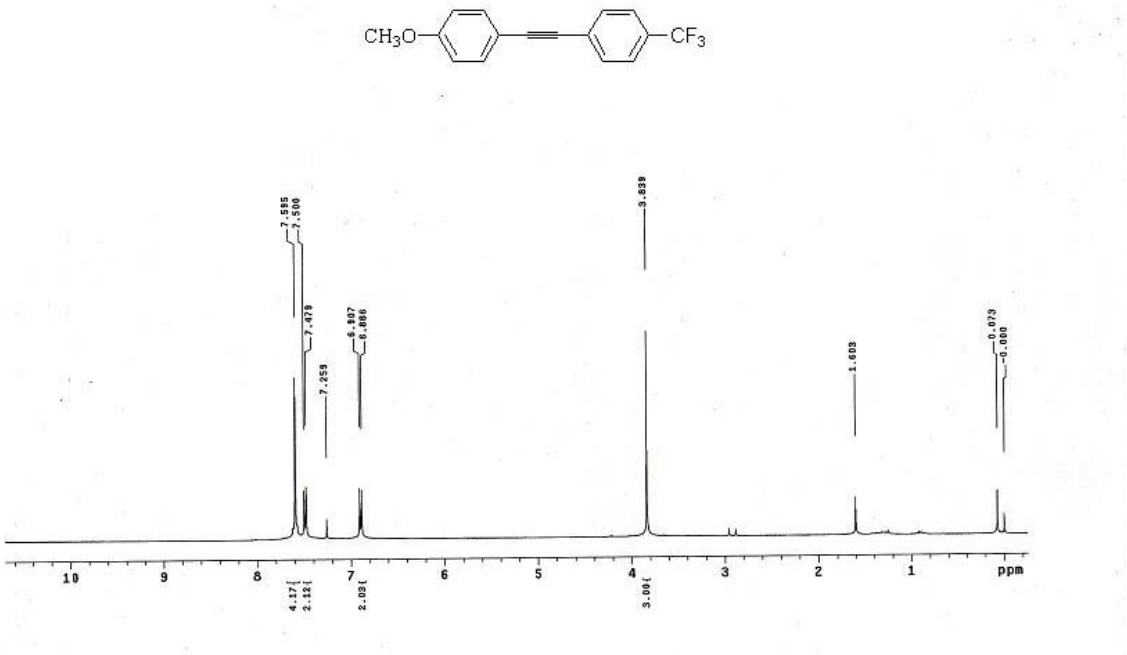
^{13}C NMR spectrum of the product 3f



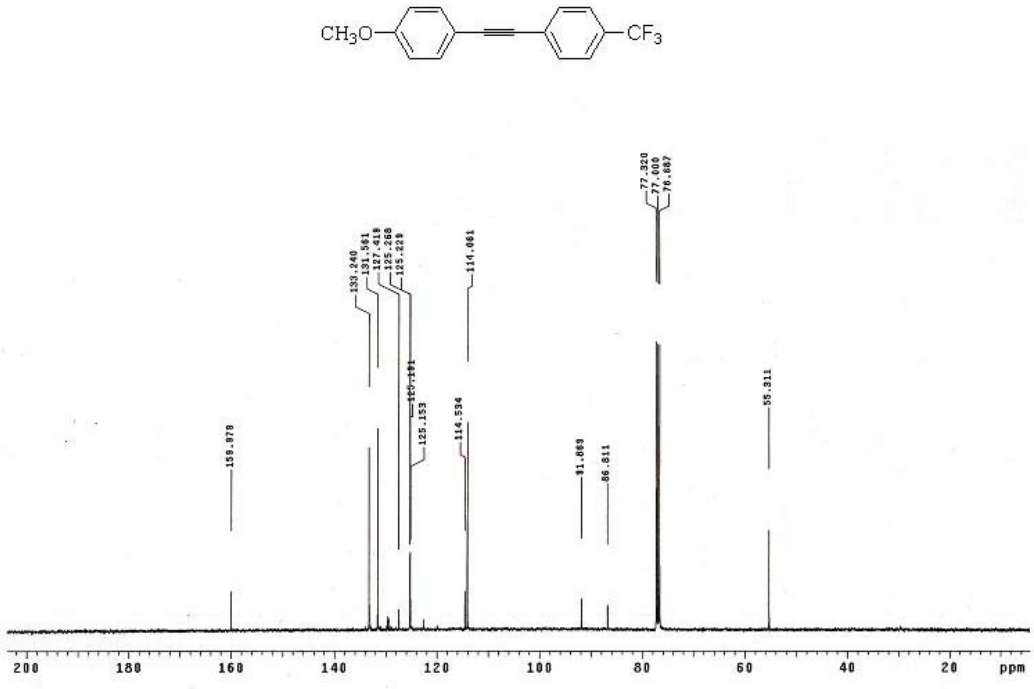
^{1}H NMR spectrum of the product 3g



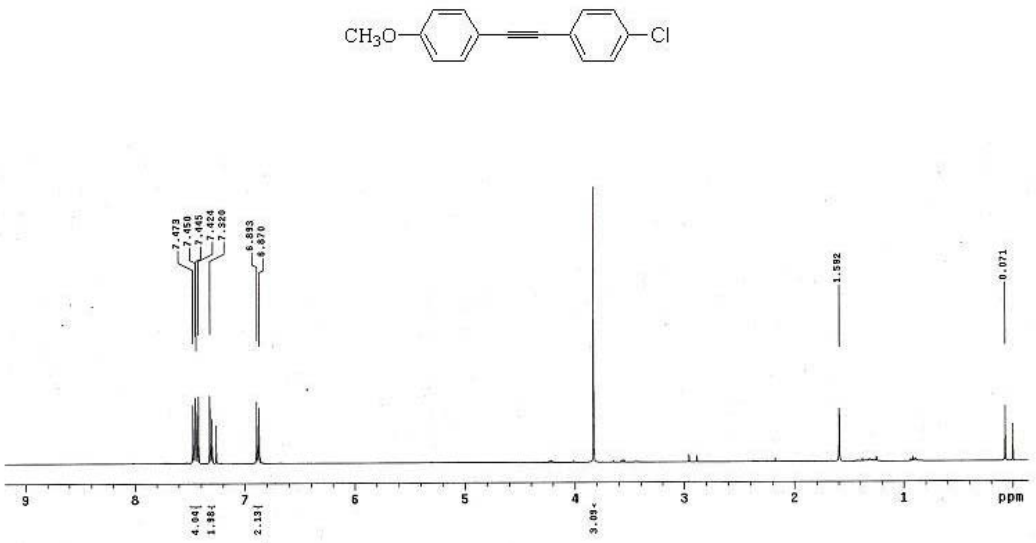
^{13}C NMR spectrum of the product 3g



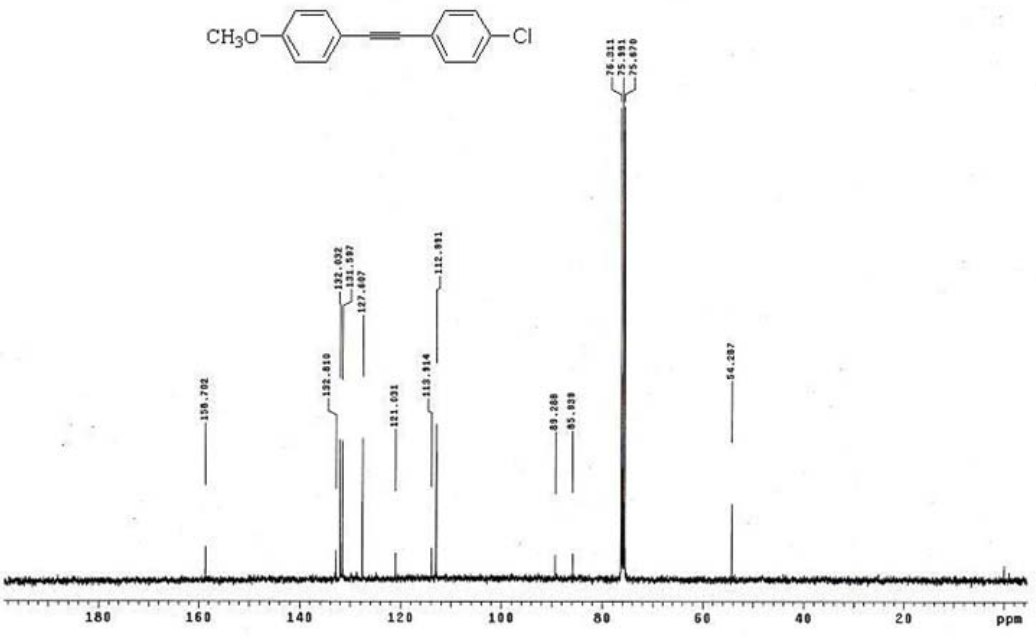
^{1}H NMR spectrum of the product 3h



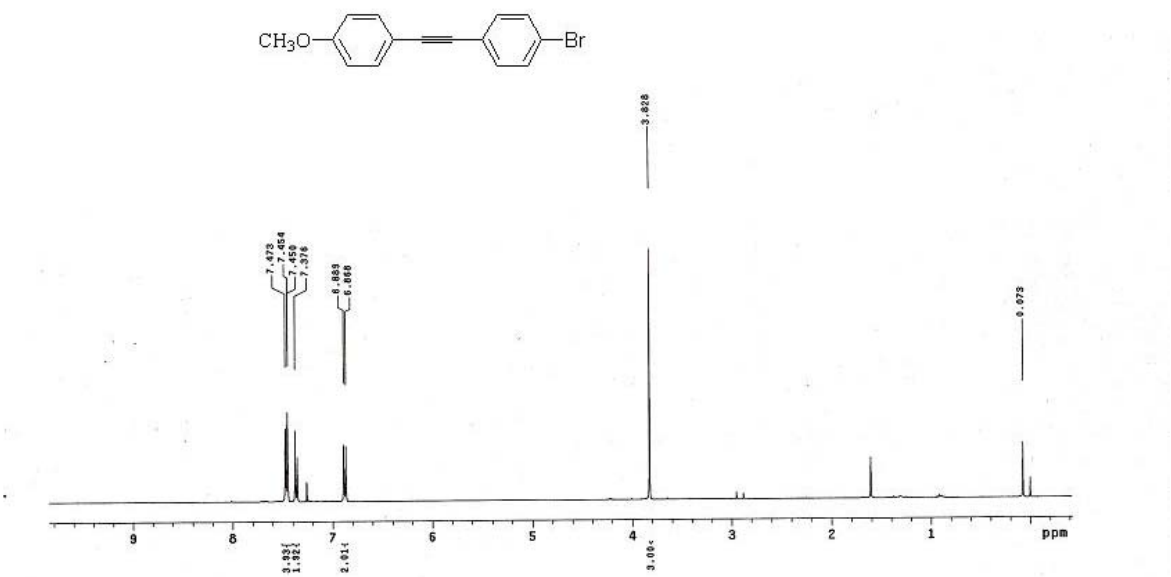
^{13}C NMR spectrum of the product 3h



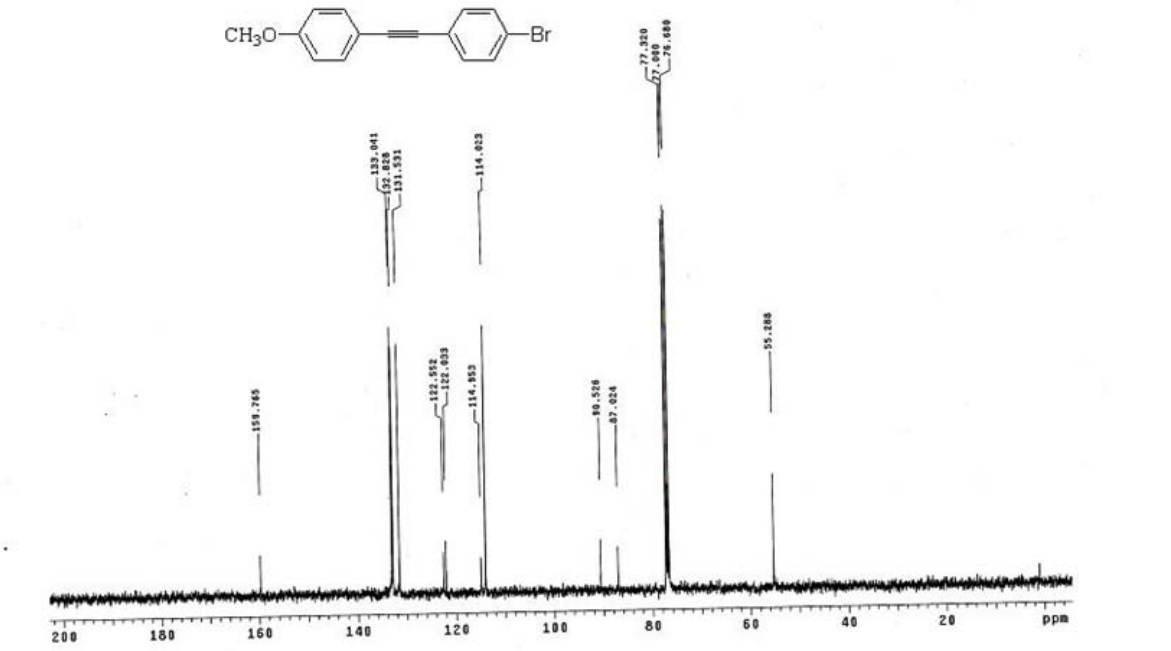
^{1}H NMR spectrum of the product 3i



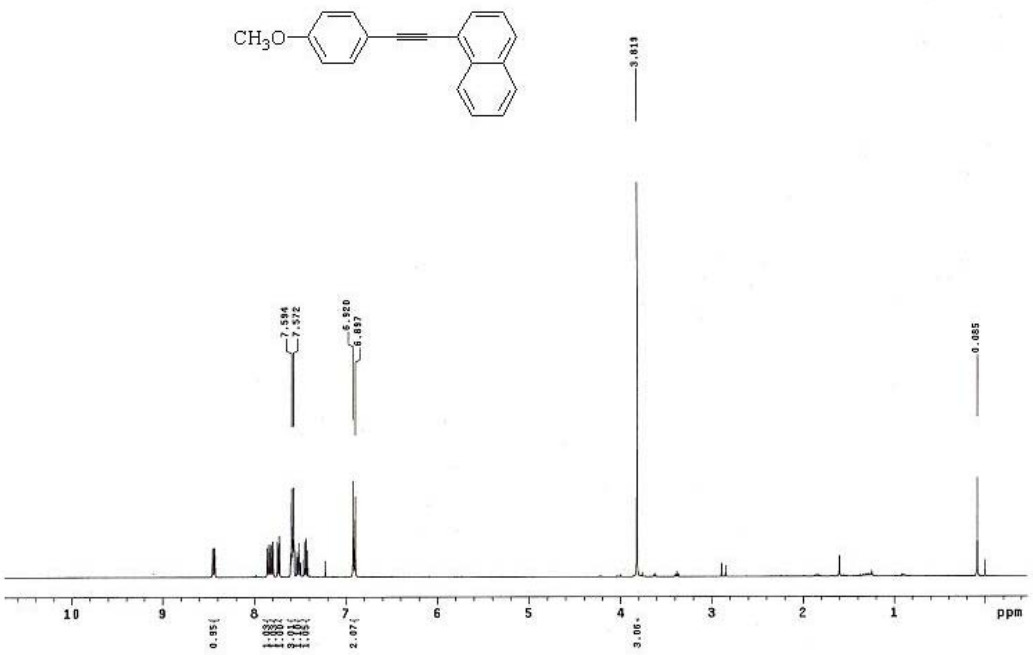
^{13}C NMR spectrum of the product 3i



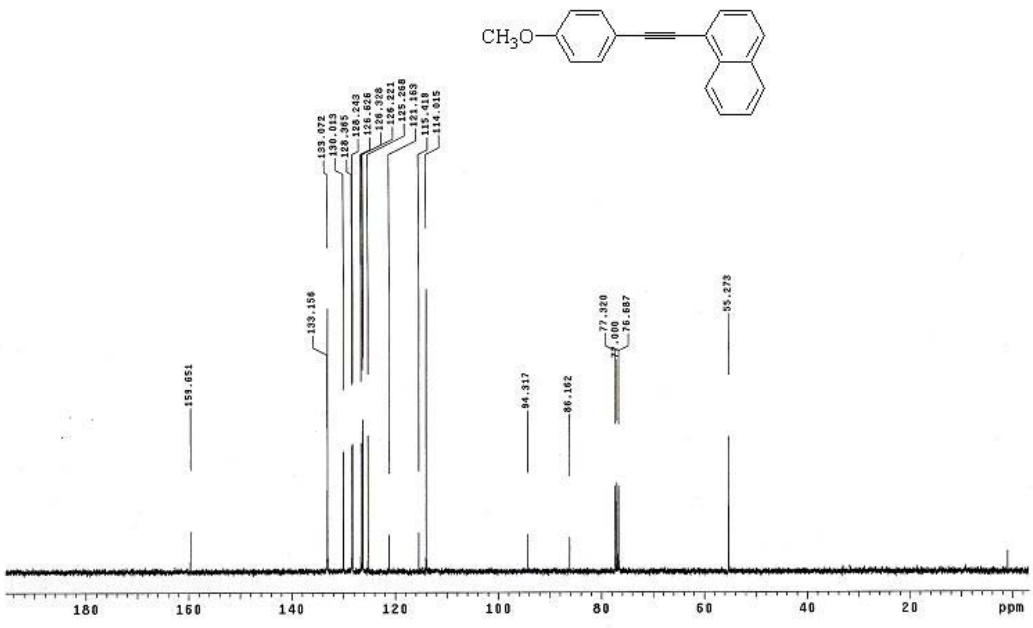
^{1}H NMR spectrum of the product 3j



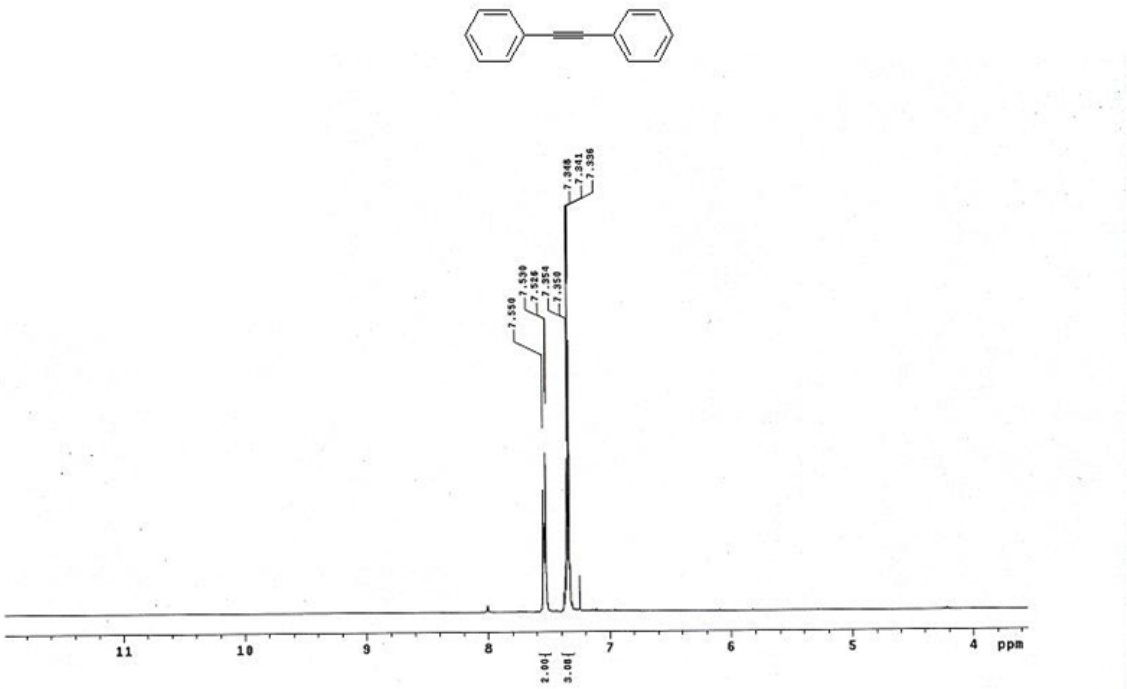
^{13}C NMR spectrum of the product 3j



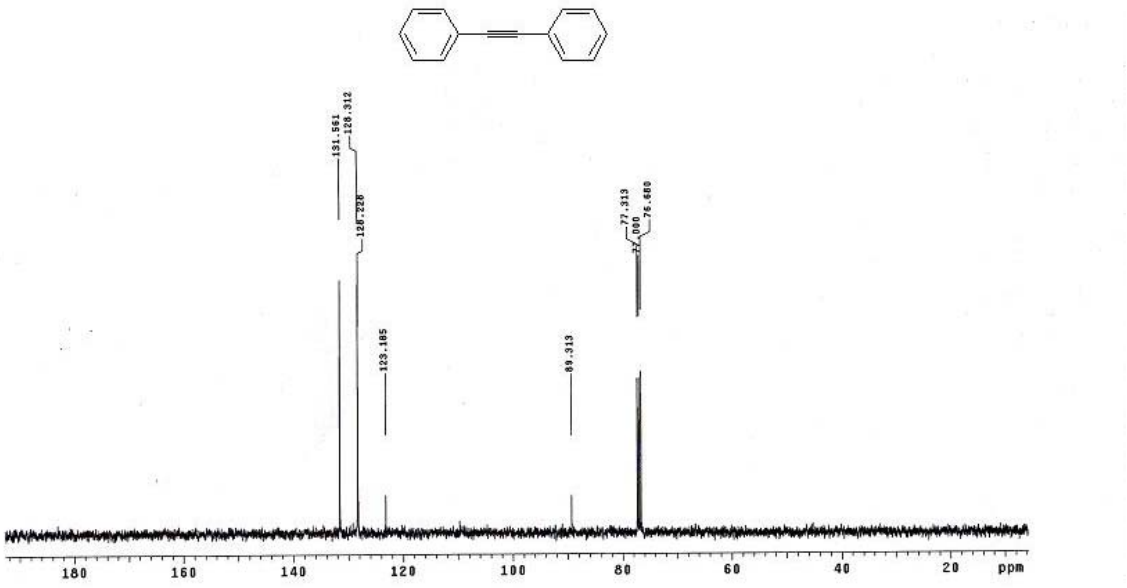
^{1}H NMR spectrum of the product 3k



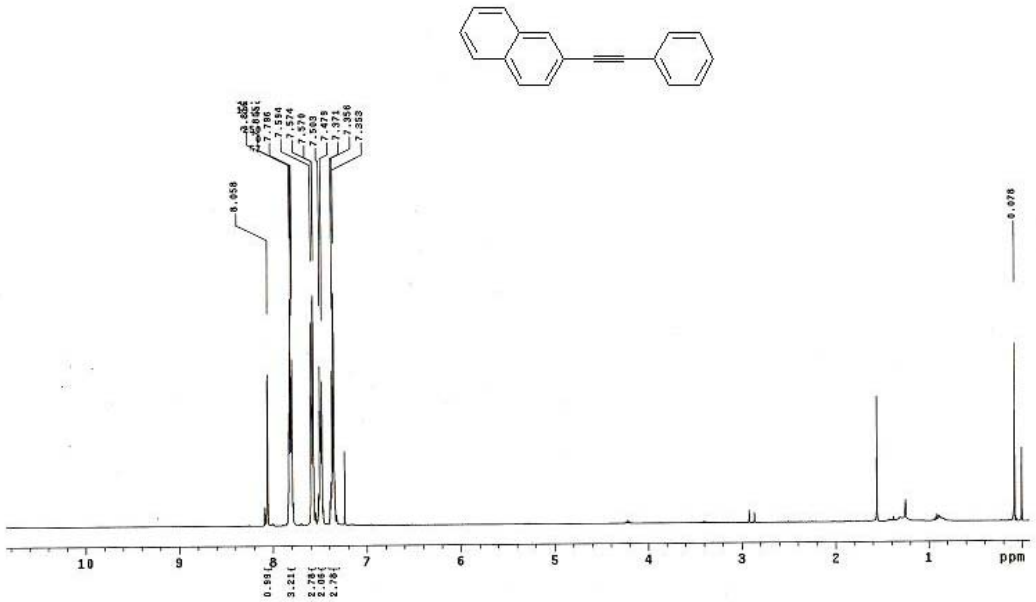
^{13}C NMR spectrum of the product 3k



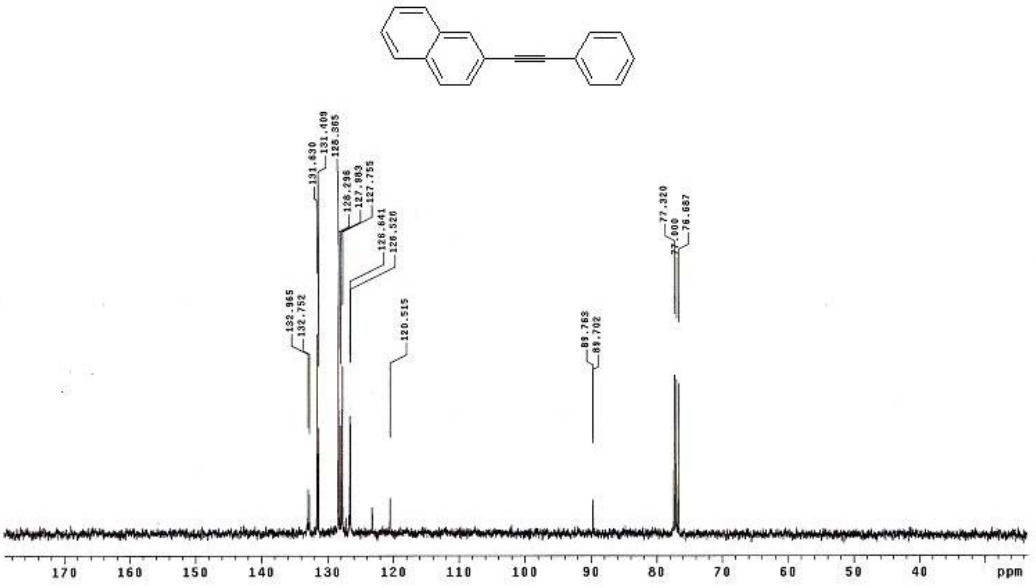
^{1}H NMR spectrum of the product 3l



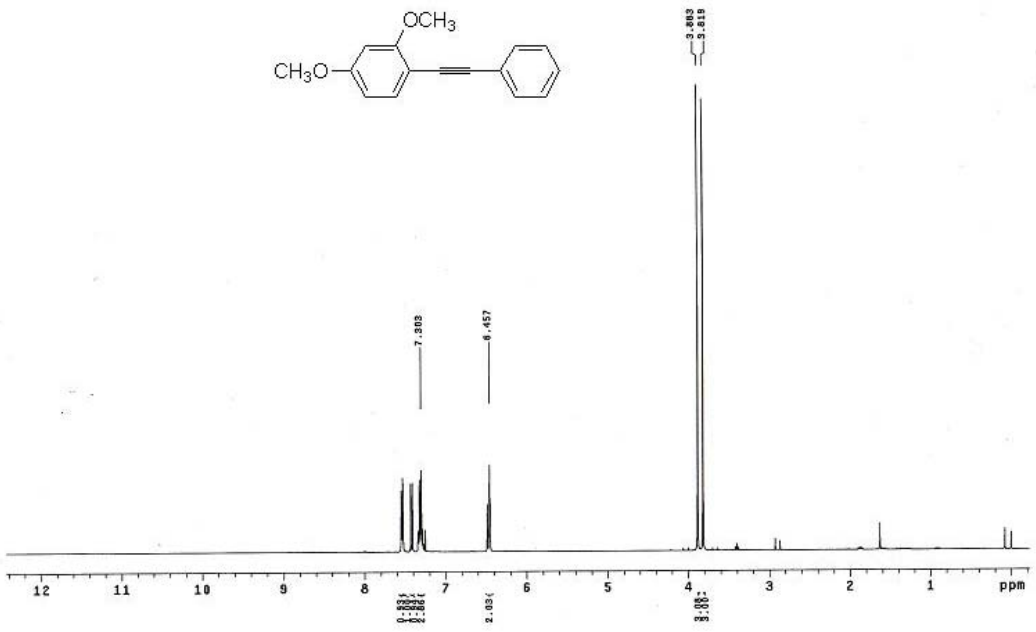
^{13}C NMR spectrum of the product 3l



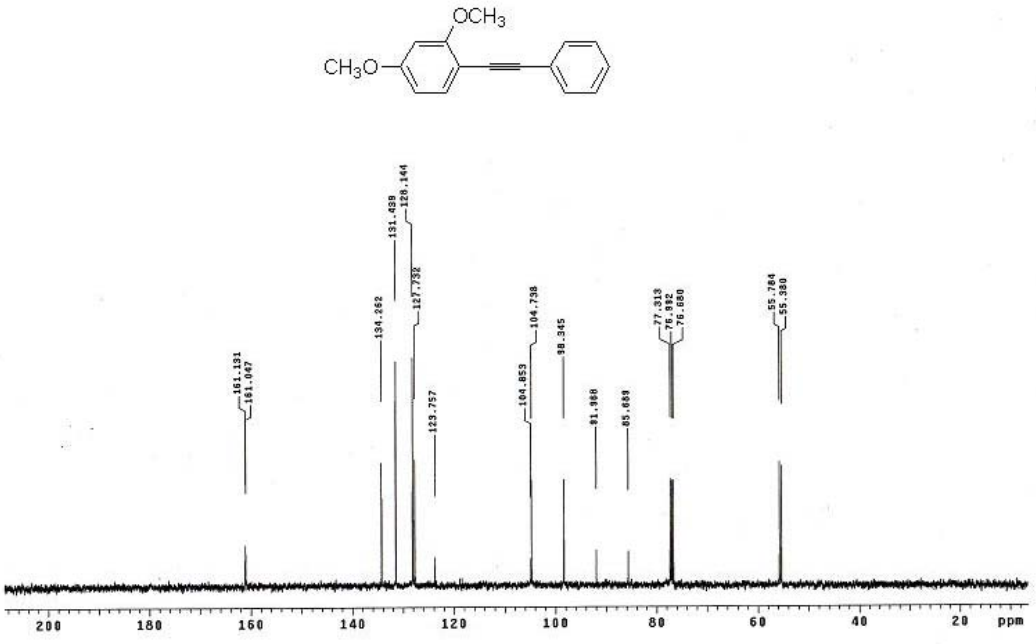
^{1}H NMR spectrum of the product 3m



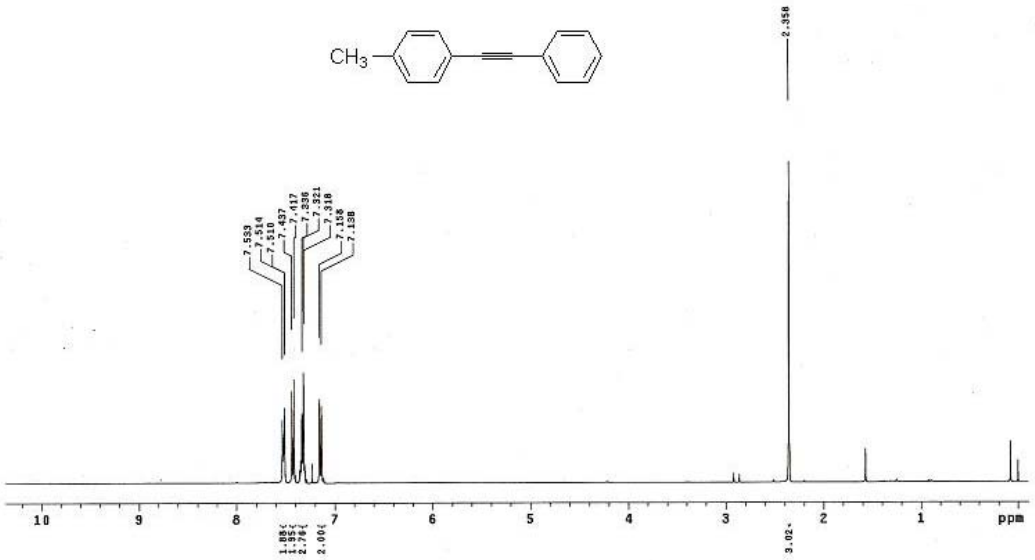
^{13}C NMR spectrum of the product 3m



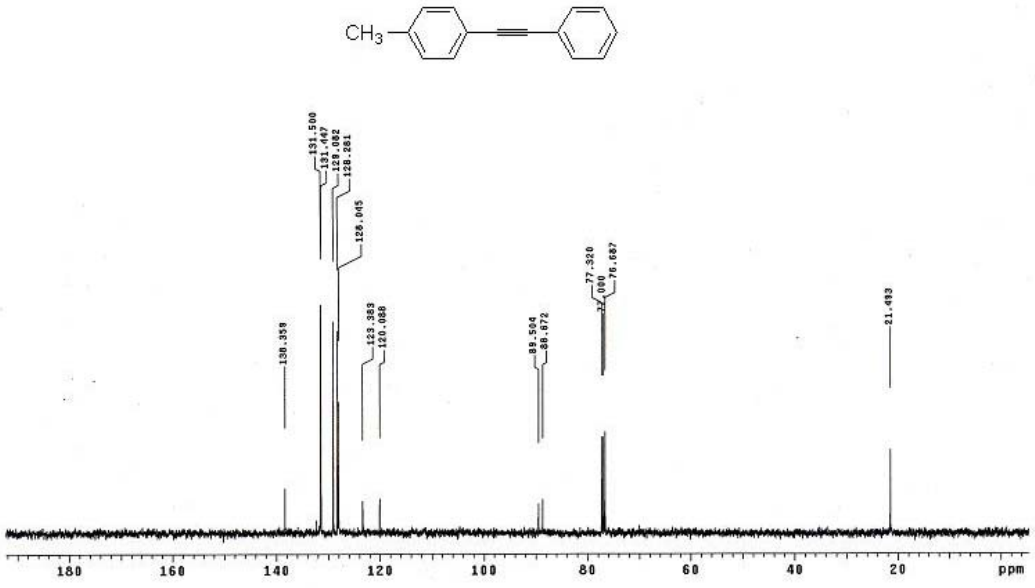
^{1}H NMR spectrum of the product 3n



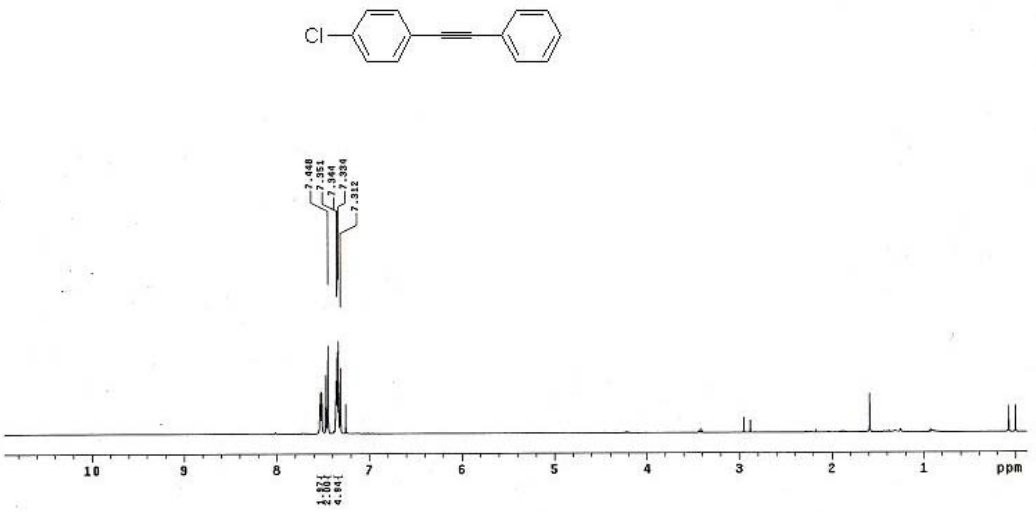
^{13}C NMR spectrum of the product 3n



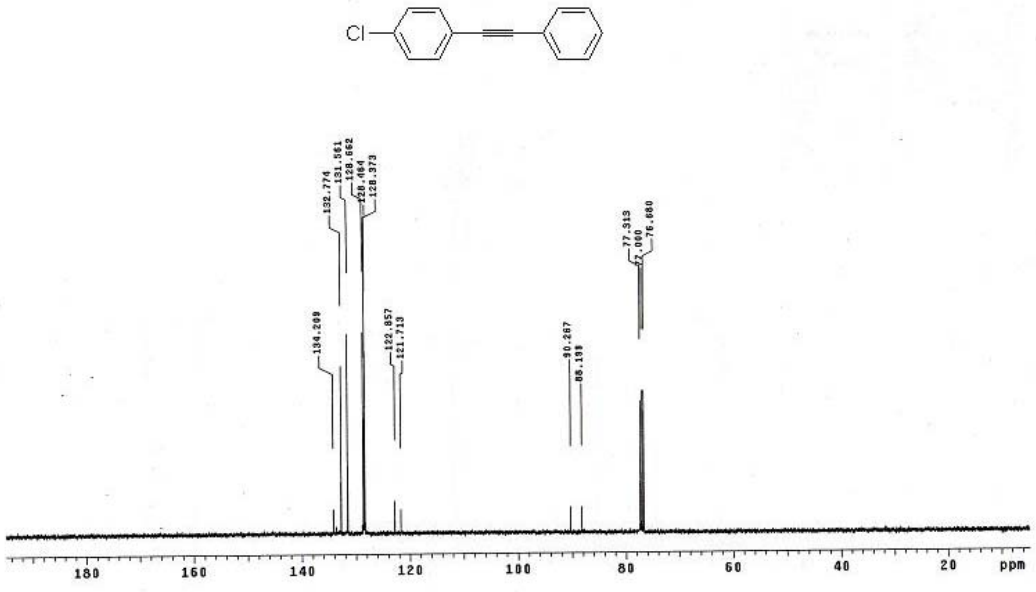
^{1}H NMR spectrum of the product 3o



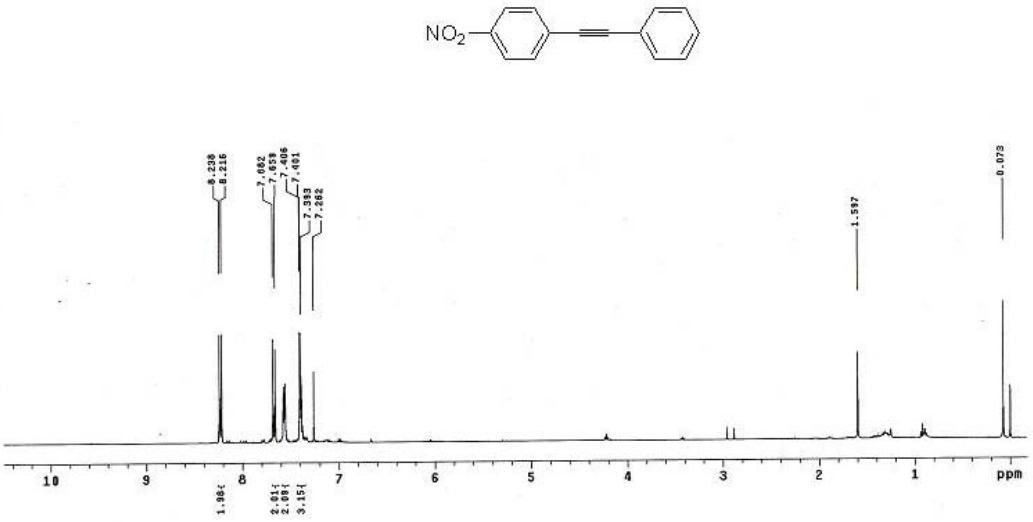
^{13}C NMR spectrum of the product 3o



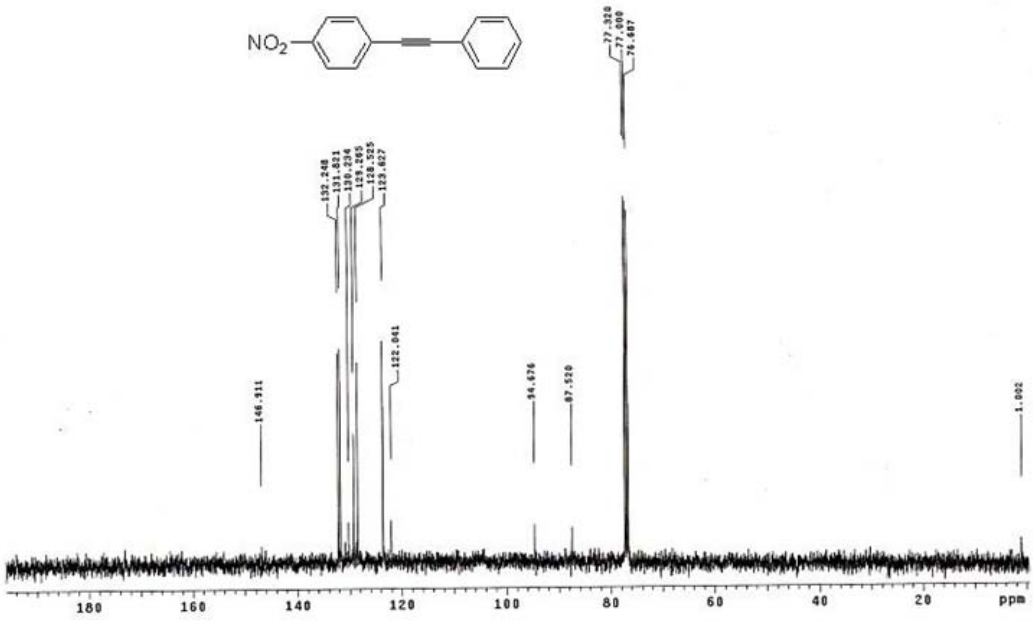
^{1}H NMR spectrum of the product 3p



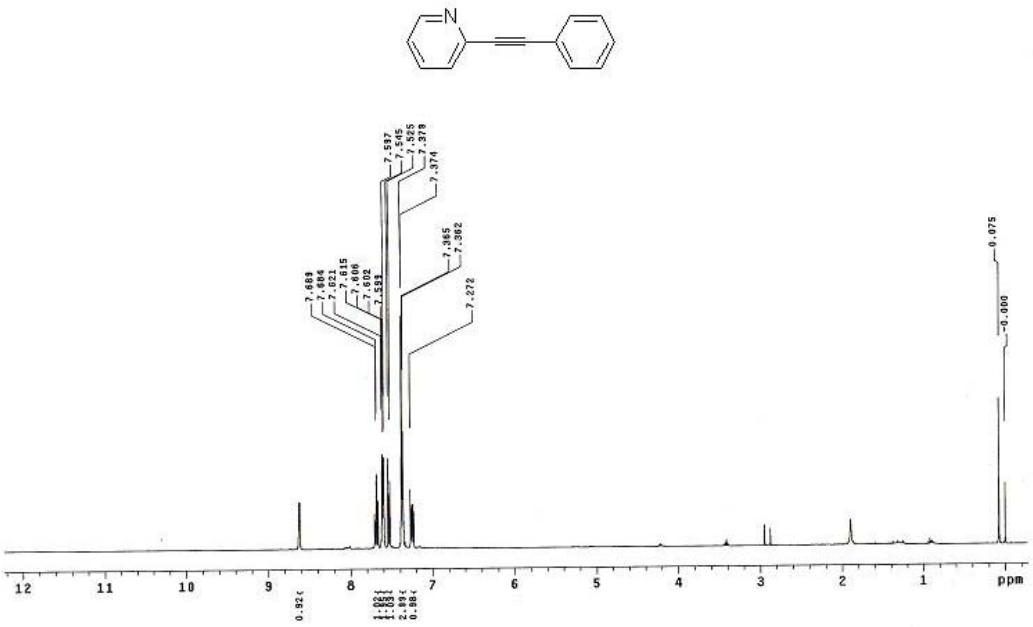
^{13}C NMR spectrum of the product 3p



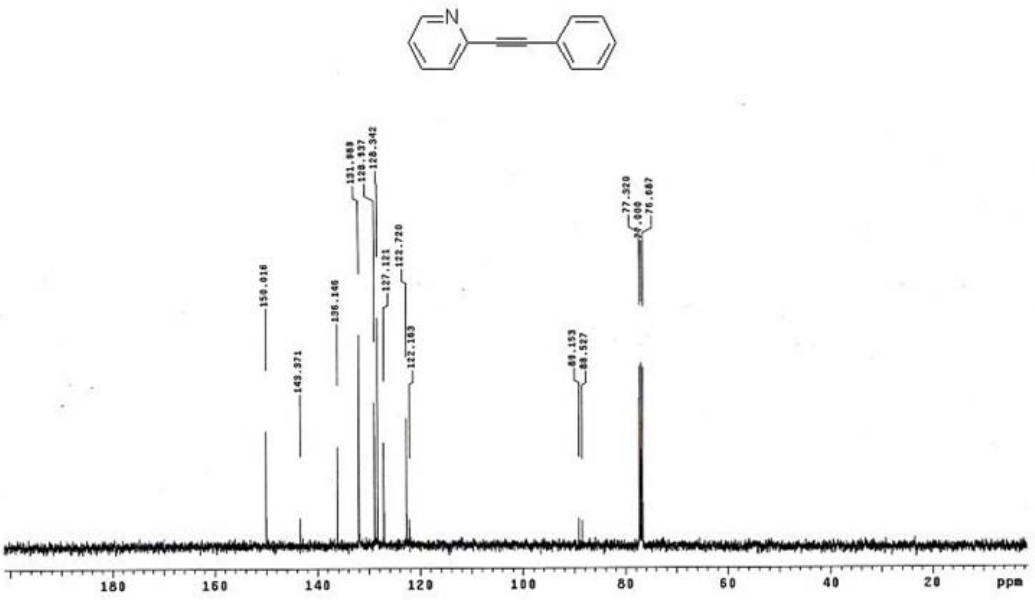
^{1}H NMR spectrum of the product 3q



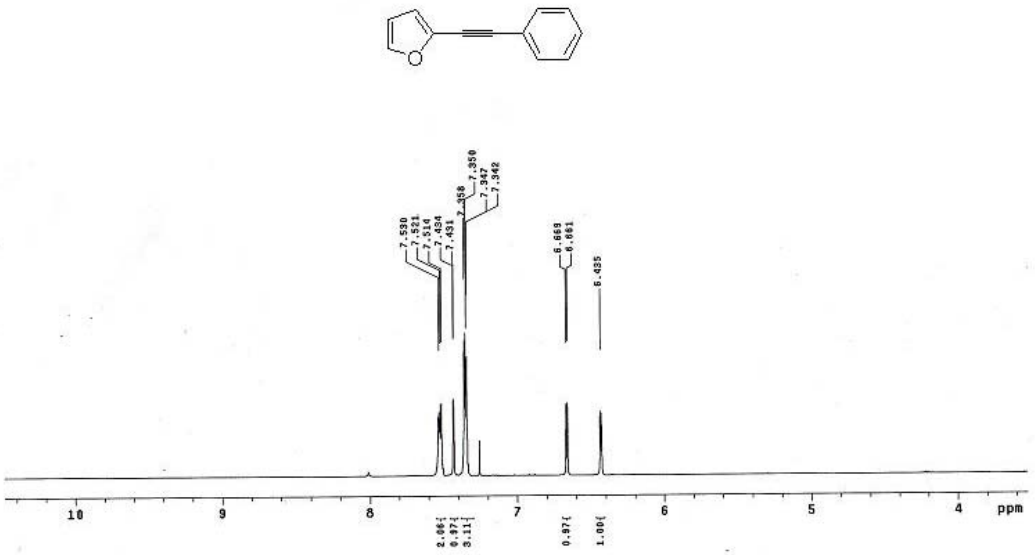
^{13}C NMR spectrum of the product 3q



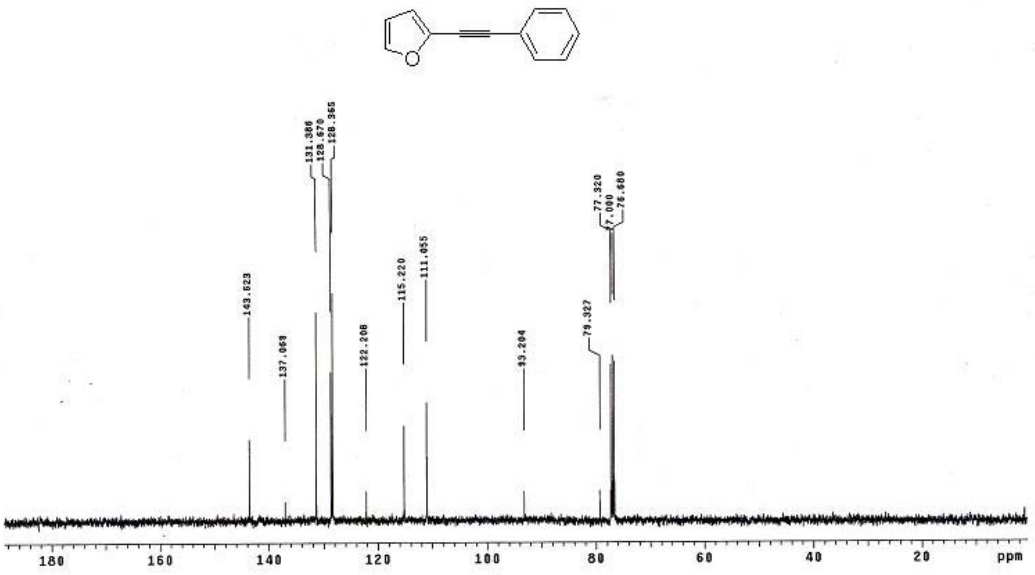
^{1}H NMR spectrum of the product 3r



^{13}C NMR spectrum of the product 3r



^{1}H NMR spectrum of the product 3s



^{13}C NMR spectrum of the product 3s

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