Supporting Information

Copper-Iodide Nanoparticle (CuI NPs): An Efficient Catalyst for the Synthesis of Alkynyl Esters

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General Experimental

General Method. ^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (100 MHz) spectra were recorded in $CDCl_3$ Chemical shifts for protons and carbons are reported in ppm from tetramethylsilane and are referenced to the carbon resonance of the solvent. Data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet), coupling constants in Hertz and integration. High-resolution mass spectra were recorded on electrospray mass spectrometer. Mass spectral data was recorded on Agilent G6530AA (LC-HRMS-Q-TOF) model of High-resolution liquid chromatography mass spectrometer with Quadrupole Time-of-flight at USIC (University Science Instrument Centre), University of Delhi, Delhi, India.TLC analysis was performed on commercially prepared 60 F254 silica gel plates and visualized by either UV irradiation or by staining with I2. X-ray diffraction (XRD) patterns were recorded on Rigaku Rotaflex spectrometer at 2θ range of 10–90° with Cu Kα radiation. The elemental composition and electronic structure analysis were obtained from X-ray photoelectron spectra (XPS) of PHI Versa Probe II instrument equipped with a monochromatic Al Kα source at the Department of Chemistry, Indiana University, Bloomington, Indiana 47405, United States.Field emission scanning Electron microscopy (FESEM) measurement was performed on a Zeiss Gemini SEM 500 thermal field emission type with acceleration voltage 0.02 – 30kV at USIC (University Science Instrument Centre), University of Delhi, Delhi, India.The Raman spectra of the samples were collected on a Renishaw Laser Raman spectrometer (Model: InviaII) using a 514 nm
laser source.All purchased chemicals were used as received. All melting points are uncorrected.

General Procedure for the Synthesis of 3-alkynoates.

Alkyne (1),α-Diazoester (2) and CuI werecommercially available from Sigma Aldrich Chemical Co. and TCI Chemicals.

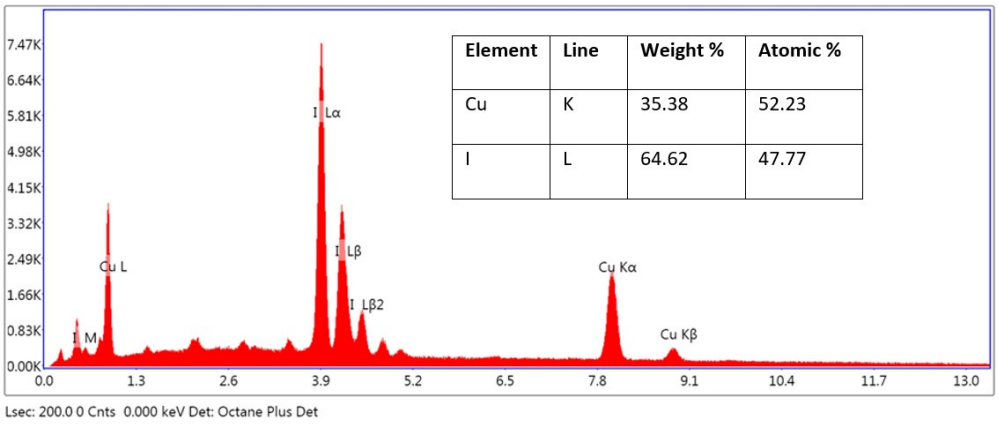
Procedure for the synthesis of 3-Alkynoates (3a-3r): A mixture of alkyne **1** (0.5 mmol, 1.0 equiv), α-diazoester**2** (0.5 mmol, 1.0 equiv), and CuI nanoparticles (3.0 mol %) were stirred at 40^{o}C for 20-30 minutes. After the complete consumption of reactant, isolation of catalyst was carried out by centrifugation *via* addition of ethanol in the reaction mixture. Ethanol was used for washing recovered nano catalyst three to four times and finally catalyst

was dried in oven. The organic layer was concentrated using rotary evaporator to afford reaction mixture. The crude material obtained was purified by column chromatography on silica gel (100−200 mesh) (hexane:ethyl acetate; 95/5).

Procedure for the synthesis of 3-Alkynoates (6a, 6b): A mixture of alkyne **1** (0.5 mmol, 1 equiv), α-diazoester**2** (1.0 mmol, 2.0 equiv), and CuInanoparticles (6.0 mol %) were stirred at 40°C for 45-50 minutes. After the complete consumption of reactant, isolation of catalyst was carried out by centrifugation *via* addition of ethanol in the reaction mixture. Ethanol was used for washing recovered nano catalyst three to four times and finally catalyst was dried in oven. The organic layer was concentrated using rotary evaporator to afford reaction mixture. The crude material obtained was purified by column chromatography on silica gel (100−200 mesh) (hexane:ethyl acetate; 90/10).

EDAX Image and analysis

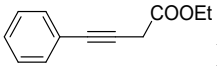
EDAX shows the composition analysis of the synthesized CuI nanoparticles.[1,2] The Energy-dispersive X-ray spectroscopy analysis in Figure SI confirms the presence of the desired elements only that is, copper and iodine. It shows that the CuI nanoparticles synthesized are of high purity. The atomic ratio (%) has slight difference which may be attributed to the presence of surface defects in CuI.



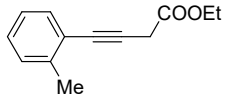
Element	Line	Weight %	Atomic %
Cu	K	35.38	52.23
I	L	64.62	47.77

Figure SI Percentage composition of synthesized CuI nanoparticles from EDX

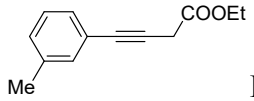
Spectral data for the compounds 3a-r and 6a-b



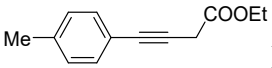
Ethyl 4-phenylbut-3-ynoate(3a). The product was obtained as a pale white oil (70.0 mg, 75%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.32(m, 2H), 7.19–7.16(m, 3H + residual $CDCl_3$), 4.10 (q, J = 7.1Hz, 2H), 3.38 (s, 2H), 1.17 (t, J = 6.4Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.0, 131.5, 128.0, 127.9, 122.8, 91.7, 83.2, 61.4, 26.4, 13.9;HRMS (ESI) $[M]^+$ Calcd for $[C_{12}H_{12}O_2]$ 188.0837, found 188.0837.



Ethyl 4-(*o*-tolyl)but-3-ynoate(3b).The product was obtained as apalewhite oil (70.7 mg, 70%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32(d, J = 7.8Hz, 1H), 7.12–7.10 (m, 2H), 7.05–7.01(m, 1H), 4.15 (q, J = 7.3Hz, 2H), 3.46(s, 2H), 2.36 (s, 3H), 1.23 (t, J = 6.8Hz, 3H);^{13}C{^{1}H} NMR(100 MHz, $CDCl_3$) δ 168.4, 140.3, 131.9, 129.3, 128.1, 125.4, 122.7, 85.0, 82.4, 61.6, 26.8, 20.6, 14.1;HRMS (ESI) $[M]^+$ Calcd for $[C_{13}H_{14}O_2]$ 202.0994, found 202.0993.

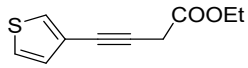


Ethyl 4-(*m*-tolyl)but-3-ynoate(3c). The product was obtained as apalewhite oil (72.7 mg, 72%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18–7.15(m, 2H), 7.11–7.07(m, 1H), 7.03–7.01(m, 1H), 4.13 (q, J = 7.3Hz, 2H), 3.40(s, 2H), 2.22 (s, 3H), 1.21 (t, J = 7.3Hz, 3H);^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.6, 138.1, 132.6, 129.3, 129.0, 128.3, 123.0, 83.8, 81.0, 61.9, 27.0, 21.4, 14.3;HRMS (ESI) $[M]^+$ Calcd for $[C_{13}H_{14}O_2]$ 202.0994, found 202.0993.

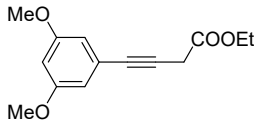


Ethyl 4-(*p*-tolyl)but-3-ynoate(3d). The product was obtained as ayellow oil (73.7 mg, 73%);^{1}H NMR (400 MHz, $CDCl_3$) δ7.21 (d, J = 8.2Hz, 2H), 6.96–6.94 (m, 2H), 4.07 (q, J = 7.3Hz, 2H), 3.35 (s, 2H), 2.18 (s, 3H),1.15 (t, J = 6.8Hz, 3H); ^{13}C{^{1}H}

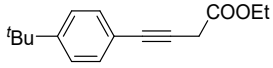
NMR (100 MHz, $CDCl_3$) δ 168.0, 137.8, 131.3, 128.6, 119.7, 83.2, 80.3, 61.2, 26.3, 21.0, 13.8;HRMS (ESI) $[M]^+$ Calcd for $[C_{13}H_{14}O_2]$ 202.0994, found 202.0993.



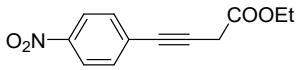
Ethyl 4-(thiophen-3-yl)but-3-ynoate (3e). The product was obtained as abrown oil (77.6 mg, 80%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43(dd, J = 2.9Hz, 1H), 7.16–7.14(m, 1H), 7.03–7.02(m, 1H), 4.14 (q, J = 7.1Hz, 2H), 3.39 (s, 2H), 1.20 (t, J = 11.7Hz, 3H);^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ168.2, 130.0, 128.8, 125.2, 122.0, 81.0, 78.7, 61.7, 26.8, 14.2;HRMS (ESI) $[M]^+$ Calcd for $[C_{10}H_{10}O_2S]$ 194.0402, found 194.0402.



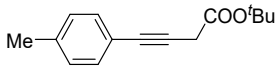
Ethyl 4-(3,5-dimethoxyphenyl)but-3-ynoate(3f). The product was obtained as abrown oil (80.6 mg, 65%);^{1}H NMR (400 MHz, $CDCl_3$) δ 6.51(d, J = 2.2Hz, 2H), 6.34–6.32(m, 1H), 4.12 (q, J = 6.8Hz, 2H), 3.66(s, 6H), 3.40 (s, 2H), 1.20 (t, J = 7.8Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.0, 160.3, 124.1, 109.3, 101.5, 83.2, 80.7, 61.5, 55.1, 26.5, 13.9;HRMS (ESI) $[M+H]^+$ Calcd for $[C_{14}H_{16}O_4]$ 248.1049, found 248.1050.



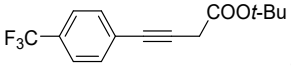
Ethyl 4-(4-(*tert*-butyl)phenyl)but-3-ynoate (3g). The product was obtained as a pale yellow oil (89.0 mg, 73%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.30(m, 2H), 7.26–7.24(m, 2H), 4.15 (q, J = 7.3Hz, 2H), 3.42(s, 2H), 1.25–1.22(m, 12H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.3, 151.3, 131.4, 125.2, 120.0, 83.5, 80.4, 61.6, 34.7, 31.1, 26.8, 14.1;HRMS (ESI) $[M]^+$ Calcd for $[C_{16}H_{20}O_2]$ 244.1463, found 244.1464.



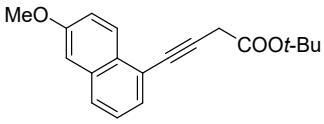
Ethyl 4-(4-nitrophenyl)but-3-ynoate (3h). The product was obtained as a brown oil (64.0 mg, 55%);^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19–8.16 (m, 2H), 7.55–7.53 (m, 2H), 4.11 (q, J = 1.8 Hz, 2H), 3.31 (s, 2H), 1.17 (t, J = 3.6 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ 163.8, 146.8, 138.5, 128.2, 124.2, 97.0, 92.2, 60.9, 26.1, 14.0;HRMS (ESI) $[M]^+$ Calcd for $[C_{12}H_{11}NO_4]$ 233.0688, found 233.0688.



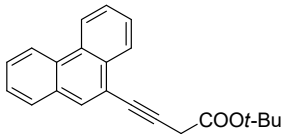
***tert*-Butyl 4-(*p*-tolyl)but-3-ynoate(3i).** The product was obtained as ayellow oil (88.5 mg, 77%);^{1}H NMR (400 MHz, $CDCl_3$) δ7.25 (d, J = 8.2Hz, 2H), 7.00 (d, J = 8.2Hz, 2H), 3.32 (s, 2H), 2.23 (s, 3H),1.40 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 167.4, 137.9, 131.4, 128.8, 119.9, 83.3, 81.7, 80.9, 27.8, 27.7, 21.3;HRMS (ESI) $[M]^+$ Calcd for $[C_{15}H_{18}O_2]$ 230.1307, found 230.1306.



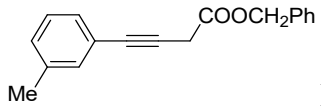
***tert*-Butyl 4-(4-(trifluoromethyl)phenyl)but-3-ynoate(3j).**The product was obtained as abrown oil (85.2 mg, 60%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53–7.48(m, 4H), 3.41(s, 2H), 1.45(s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 166.9, 131.9, 127.4, 126.9, 125.0 (q,$J_{C\text{-}F}$ = 3.82 Hz), 122.5, 84.6, 82.1, 27.9, 27.8;HRMS (ESI) $[M]^+$ Calcd for $[C_{15}H_{15}F_3O_2]$ 284.1024, found 284.1023.



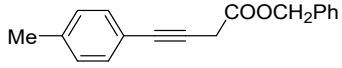
***tert*-Butyl 4-(6-methoxynaphthalen-1-yl)but-3-ynoate (3k).** The product was obtained as ayellow oil (103.6 mg, 70%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88(s, 1H), 7.66–7.62(m, 2H), 7.46–7.44(m, 1H), 7.14–7.11(m, 1H), 7.08–7.07(m, 1H), 3.89(s, 3H), 3.46 (s, 2H), 1.50 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 167.5, 158.1,134.0, 131.3, 129.2, 129.1, 128.3, 127.3, 126.6, 125.4, 119.2, 118.0, 105.6, 83.7, 81.3, 55.3, 28.0, 27.9;HRMS (ESI) $[M]^+$ Calcd for $[C_{19}H_{20}O_3]$ 296.1412, found 296.1411.



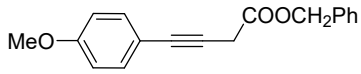
***tert*-Butyl 4-(phenanthren-9-yl)but-3-ynoate(3l).** The product was obtained as apale white oil (107.4 mg, 68%);^{1}H NMR (400 MHz, $CDCl_3$) δ8.67–8.60(m, 2H), 8.57–8.55 (m, 1H), 8.00 (s, 1H), 7.84–7.82(m, 1H), 7.70–7.67 (m, 2H), 7.65–7.55(m, 2H), 3.64 (s, 2H), 1.58 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 167.3, 131.6, 131.3, 131.0, 130.0, 129.9, 128.3, 127.2, 127.0, 126.9, 126.8, 122.6, 122.5, 119.5, 86.4, 82.0, 81.6, 28.2, 27.9;HRMS (ESI) $[M]^+$ Calcd for $[C_{22}H_{20}O_2]$ 316.1463, found 316.1464**.**



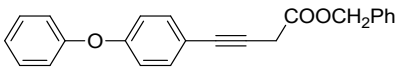
Benzyl 4-(*m*-tolyl)but-3-ynoate(3m). The product was obtained as apalewhite oil (92.4 mg, 70%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.27(m, 5H), 7.22(d, J = 8.1Hz, 2H), 7.13(t, J = 7.6Hz, 1H), 7.06(d, J = 7.7Hz, 1H), 5.15(s, 2H), 3.48(s, 2H), 2.25(s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.0, 137.7, 135.3, 132.2, 129.0, 128.6, 128.4, 128.2, 128.1, 128.0, 122.6, 122.2, 91.5, 80.5, 67.1, 26.6, 21.0;HRMS (ESI) $[M]^+$ Calcd for $[C_{18}H_{16}O_2]$ 264.1150, found 264.1149.



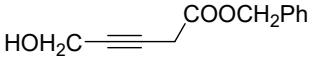
Benzyl 4-(*p*-tolyl)but-3-ynoate(3n). The product was obtained as a pale white oil (99.0 mg, 75%);^{1}H NMR (400 MHz, $CDCl_3$)δ 7.35–7.26(m, 7H), 7.03 (d, J = 7.8 Hz, 2H), 5.14 (s, 2H), 3.47 (s, 2H),2.26 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)δ168.3, 138.4, 135.7, 131.8, 129.8, 129.2, 128. 8, 128.5, 128.4, 127.7, 120.1, 84.0, 80.6, 67.3, 26. 9, 21.6;HRMS (ESI) $[M]^+$ Calcd for $[C_{18}H_{16}O_2]$ 264.1150, found 264.1151.



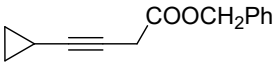
Benzyl 4-(4-methoxyphenyl)but-3-ynoate(3o). The product was obtained as ayellow oil (109.2 mg, 78%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.31(m, 7H), 6.80 (dd, J = 6.8, 2.0Hz, 2H), 5.19 (s, 2H), 3.77 (s, 3H),3.52 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ168.4, 159.3, 135.3, 133.0, 128.4, 128.2, 128.1, 114.9, 113.7, 83.3, 79.4, 67.0, 55.0, 26.6;HRMS (ESI) $[M]^+$ Calcd for $[C_{18}H_{16}O_3]$ 280.1099, found 280.1098.



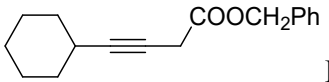
Benzyl 4-(4-phenoxyphenyl)but-3-ynoate (3p). The product was obtained as abrown oil (128.2 mg, 75%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.32(m, 9H), 7.14–7.10(m, 1H), 7.02–7.00(m, 2H), 6.91–6.89(m, 2H), 5.19(s, 2H), 3.53 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.1, 157.4, 156.4, 135.4, 133.3, 129.8, 128.6, 128.5, 128.4, 128.2, 123.7, 119.3, 118.2, 83.1, 80.2, 67.2, 26.7;HRMS (ESI) $[M]^+$ Calcd for $[C_{23}H_{18}O_3]$ 342.1256, found 342.1256.



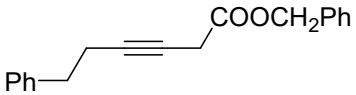
Benzyl 5-hydroxypent-3-ynoate (3q). The product was obtained as apalewhite oil (52.0 mg, 51%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.24(m, 5H), 5.08–5.06(m, 3H), 4.16–4.13(m, 2H), 3.26(s, 2H); $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 165.8, 135.1, 128.6, 128.5, 128.3, 128.2, 96.7, 89.9, 59.1, 51.0, 26.0;HRMS (ESI) $[M]^+$ Calcd for $[C_{12}H_{12}O_3]$ 204.0786, found 204.0786.



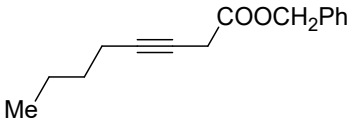
Benzyl 4-cyclopropylbut-3-ynoate(3r). The product was obtained as ayellow oil (54.06 mg, 53%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28–7.26(m, 5H), 5.07(s, 2H), 3.17(s, 2H), 1.19–1.12(m, 1H), 0.66–0.61(m, 2H), 0.59–0.57(m, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 168.0, 131.9, 131.6, 127.3, 123.4, 121.8, 78.7, 61.7, 26.7, 14.1, 0.3;HRMS (ESI) $[M]^+$ Calcd for $[C_{14}H_{14}O_2]$ 214.0994, found 214.0994.



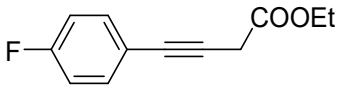
Benzyl 4-cyclohexylbut-3-ynoate (3s). The product was obtained as a white oil(82.05 mg, 64%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.23 (m, 5H), 5.08 (s, 2H), 3.23 (s, 2H), 2.32-2.28 (m, 1H), 1.72-1.65 (m, 2H), 1.64-1.57 (m, 2H), 1.45-1.30 (m, 2H), 1.24-1.04 (m, 4H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 168.8, 135.5, 128.5, 128.4, 128.3, 128.1, 127.9, 88.7, 88.1, 71.2, 66.9, 36.5, 32.6, 29.0, 26.1, 25.8, 24.7; HRMS (ESI) [M+H]Calcd for $[C_{17}H_{21}O_2]$ 257.1542, found 257.1536.



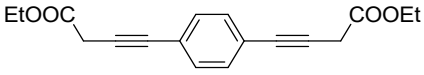
Benzyl 6-phenylhex-3-ynoate (3t). The product was obtained as a pale yellow oil (86.18 mg, 62%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.27 (m, 5H), 7.25-7.21 (m, 2H), 7.17-7.12 (m, 3H), 5.11 (s, 2H), 3.24 (s, 2H), 2.79-2.69 (m, 2H), 2.48-2.38 (m, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 168.7, 140.6, 135.4, 128.5, 128.4, 128.3, 128.1, 128.0, 126.2, 126.1, 88.5, 83.2, 67.1, 35.0, 26.1, 21.0; HRMS (ESI) [M+H]Calcd for $[C_{19}H_{19}O_2]$ 279.1307, found 279.1304



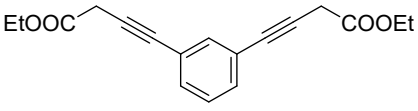
benzyl oct-3-ynoate (3u). The product was obtained as a pale yellow oil(67.85 mg, 59%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 5H), 5.09 (s, 2H), 3.22 (s, 2H), 2.14-2.04 (m, 2H), 1.45-1.22 (m, 4H), 0.86-0.79 (m, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.9, 135.5, 128.5, 128.3, 128.2, 128.0, 88.0, 84.0, 67.0, 30.7, 26.1, 21.9, 18.4, 13.6; HRMS (ESI) [M+H]Calcd for [$C_{15}H_{19}O_2$] 231.1385, found 231.1384.



ethyl 4-(4-fluorophenyl)but-3-ynoate (3v). The product was obtained as a yellow oil(72.10 mg, 70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.28 (m, 2H), 6.98-6.89 (m, 2H), 4.16 (q, J = 7.1 Hz, 2H), 3.41 (s, 2H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.2, 162.4 (d, $J_{C\text{-}F}$ = 252.2 Hz), 133.6 (d, $J_{C\text{-}F}$ = 7.7 Hz), 129.1 (d, $J_{C\text{-}F}$ = 7.7 Hz), 119.0, 115.8 (d, $J_{C\text{-}F}$ = 22.22 Hz), 115.6 (d, $J_{C\text{-}F}$ = 22.22 Hz), 94.9 (d, $J_{C\text{-}F}$ = 569.1 Hz), 81.6 (d, $J_{C\text{-}F}$ = 149.9 Hz), 61.7, 26.6, 14.2; HRMS (ESI) [M+H]Calcd for [$C_{12}H_{12}FO_2$] 207.0821, found 207.0811.



Diethyl 4,4'-(1,4-phenylene)bis(but-3-ynoate) (6a). The product was obtained as abrown oil (92.3 mg, 62%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.36(m, 1H), 7.33(s, 2H), 7.24–7.19(m, 1H), 4.19 (q, J = 6.8Hz, 4H),3.47(s, 4H), 1.29–1.25 (m, 6H); ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 168.0, 131.4, 122.7, 92.1, 82.9, 61.6, 26.6, 14.0; HRMS (ESI) [M]$^{+}$ Calcd for [$C_{18}H_{18}O_4$] 298.1205, found 298.1206.

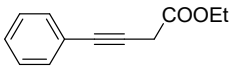


Diethyl 4,4'-(1,3-phenylene)bis(but-3-ynoate)(6b). The product was obtained as abrown oil (87.9 mg, 59%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56–7.55(m, 1H), 7.40(dd, J = 7.8, 1.8 Hz, 3H), 4.21(q, J = 7.3Hz, 4H), 3.48(s, 4H), 1.29 (t, J = 7.3Hz, 6H); ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 168.0, 135.2, 132.0, 131.7, 128.2, 123.2, 122.2, 97.9, 92.2, 61.7, 26.6, 14.1;HRMS (ESI) [M]$^{+}$ Calcd for [$C_{18}H_{18}O_4$] 298.1205, found 298.1204.

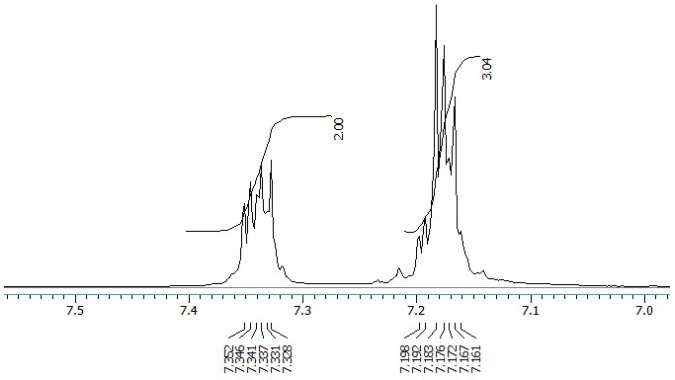
COPIES OF ^{1}H NMR, ^{13}C{^{1}H} NMR, HRMS

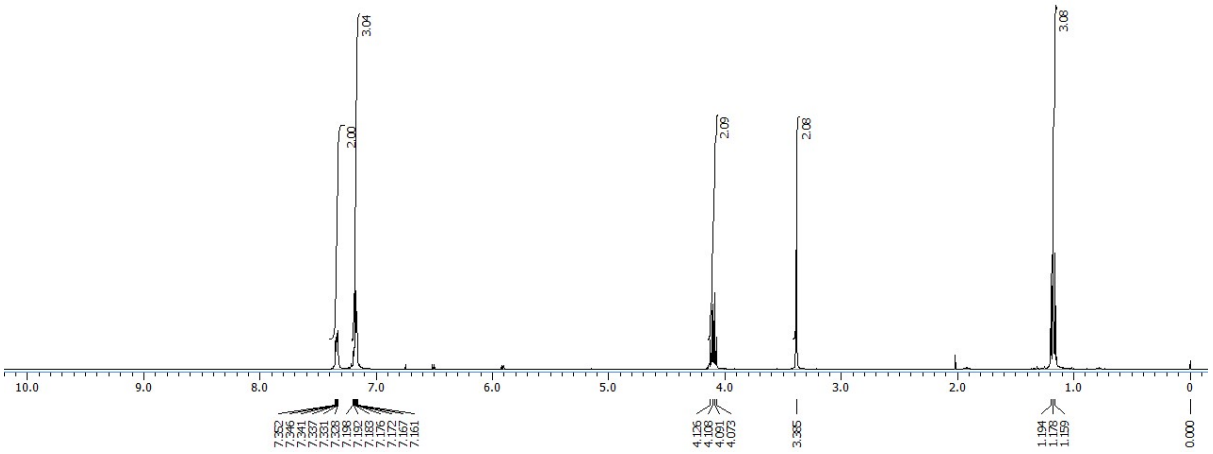
^{1}H NMR

(400 MHz, $CDCl_3$)



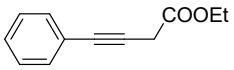
Ethyl 4-phenylbut-3-ynoate(3a)



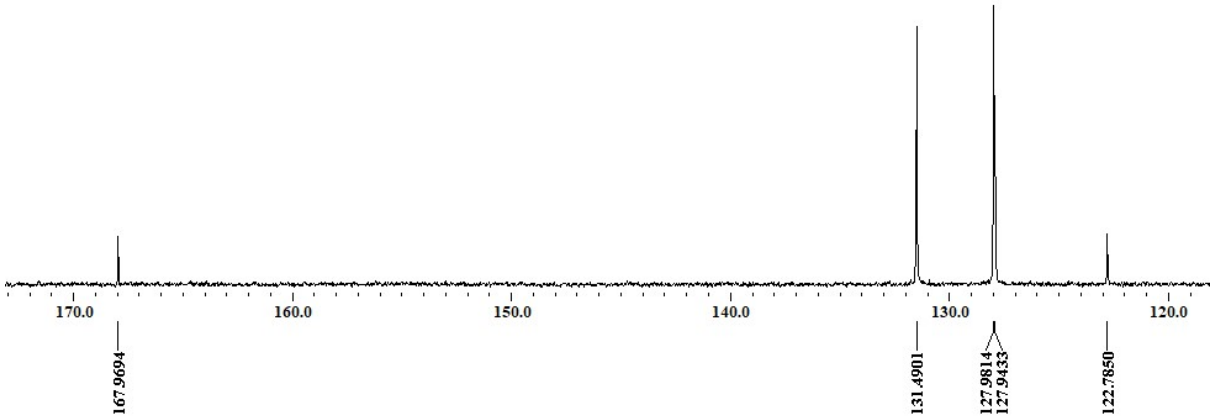


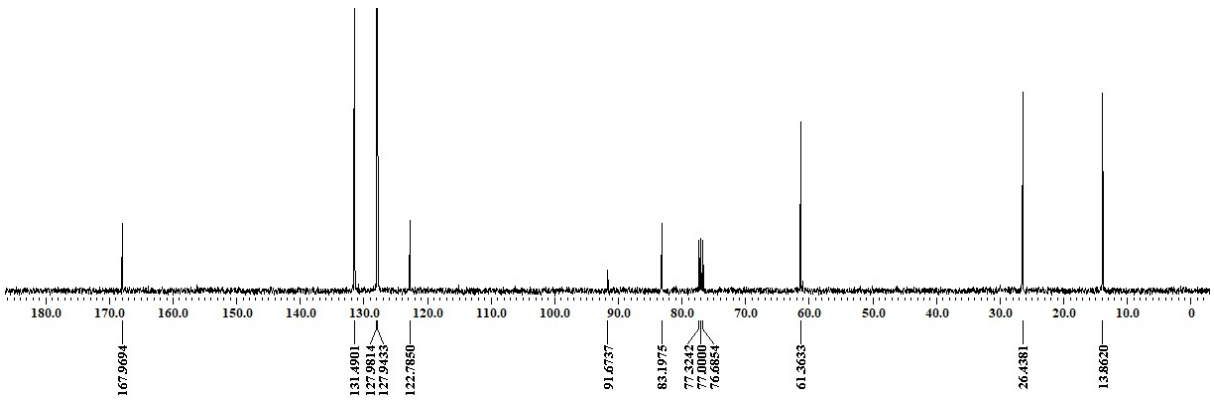
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

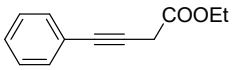


Ethyl 4-phenylbut-3-ynoate(3b)

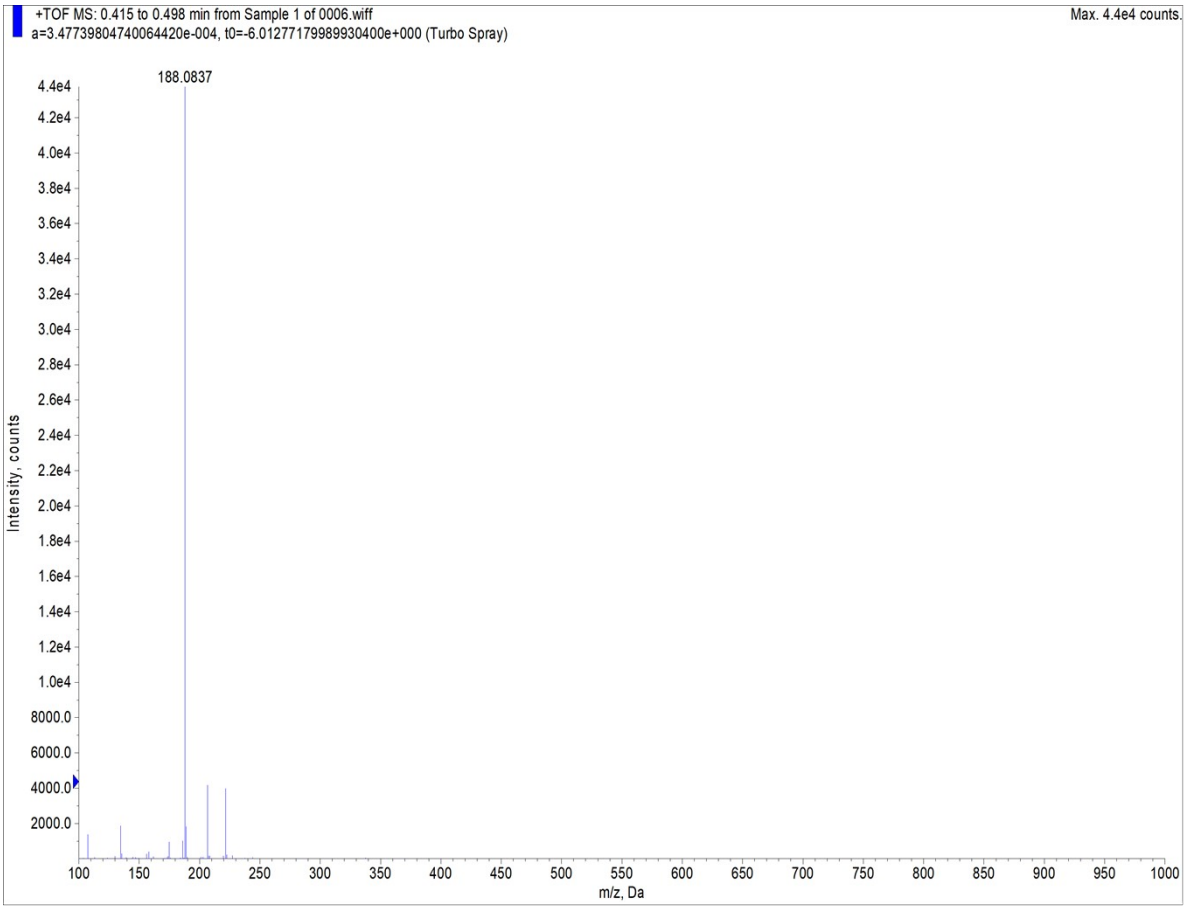




HRMS

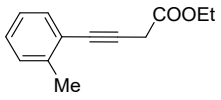


Ethyl 4-phenylbut-3-ynoate(3a)

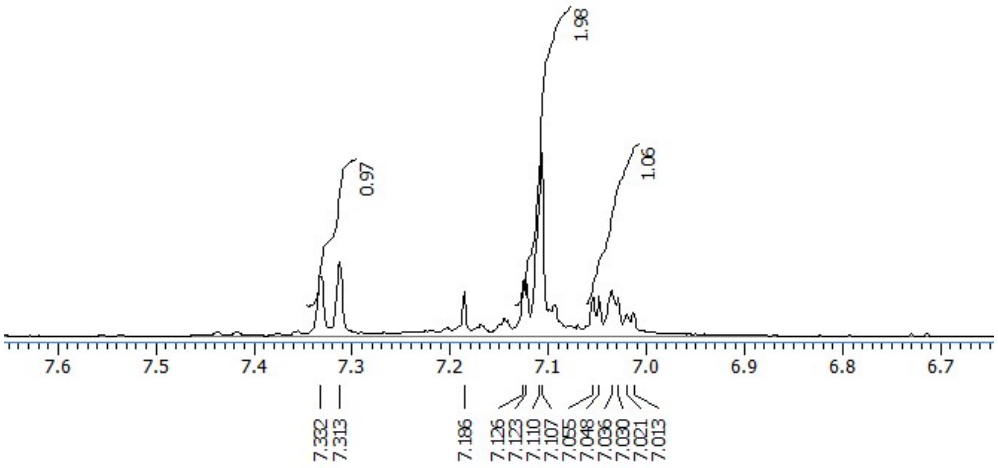


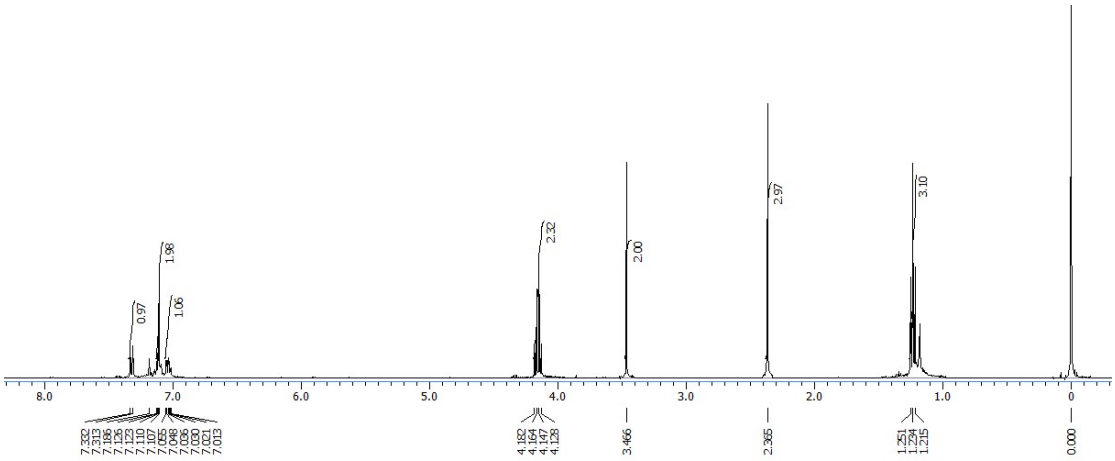
^{1}H NMR

(400 MHz, $CDCl_3$)



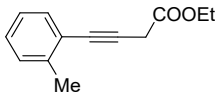
Ethyl 4-(*o*-tolyl)but-3-ynoate(3b)



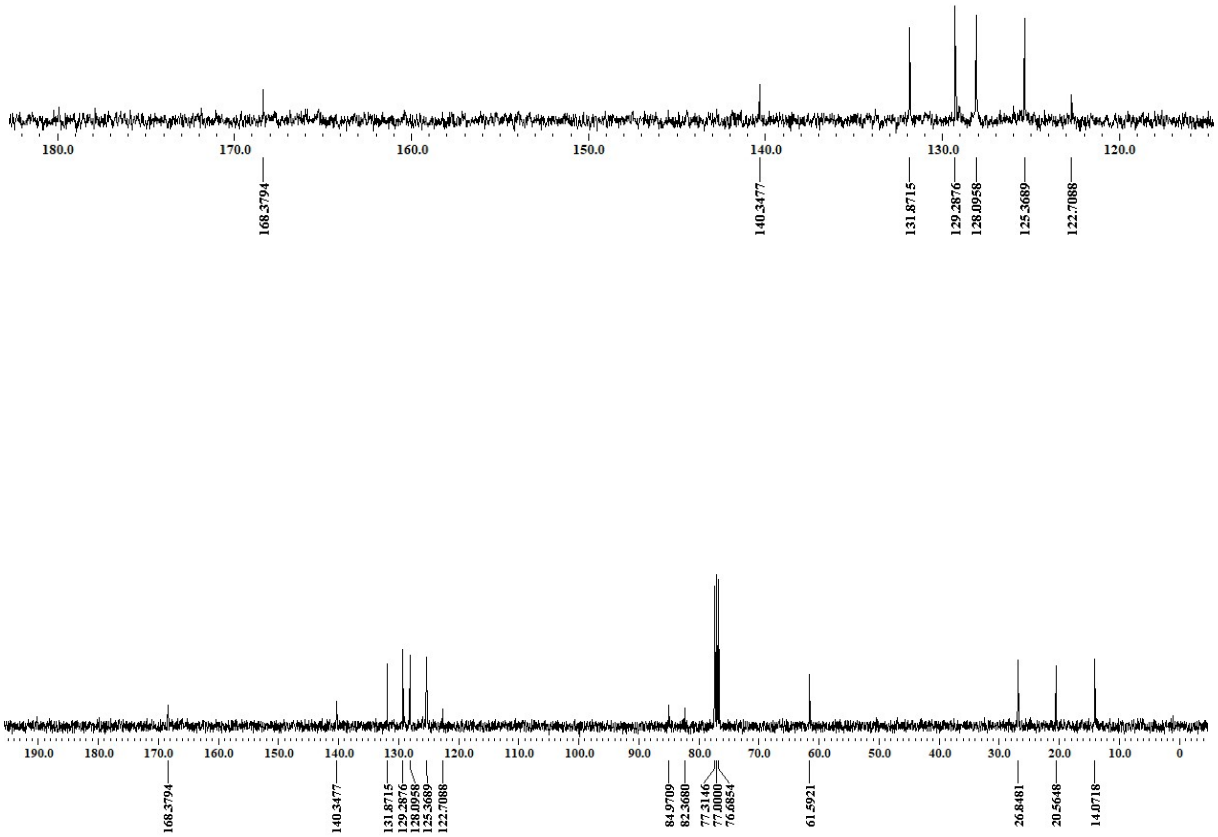


$^{13}C\{^{1}H\}$ NMR

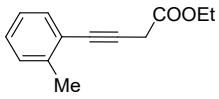
(100 MHz, $CDCl_3$)



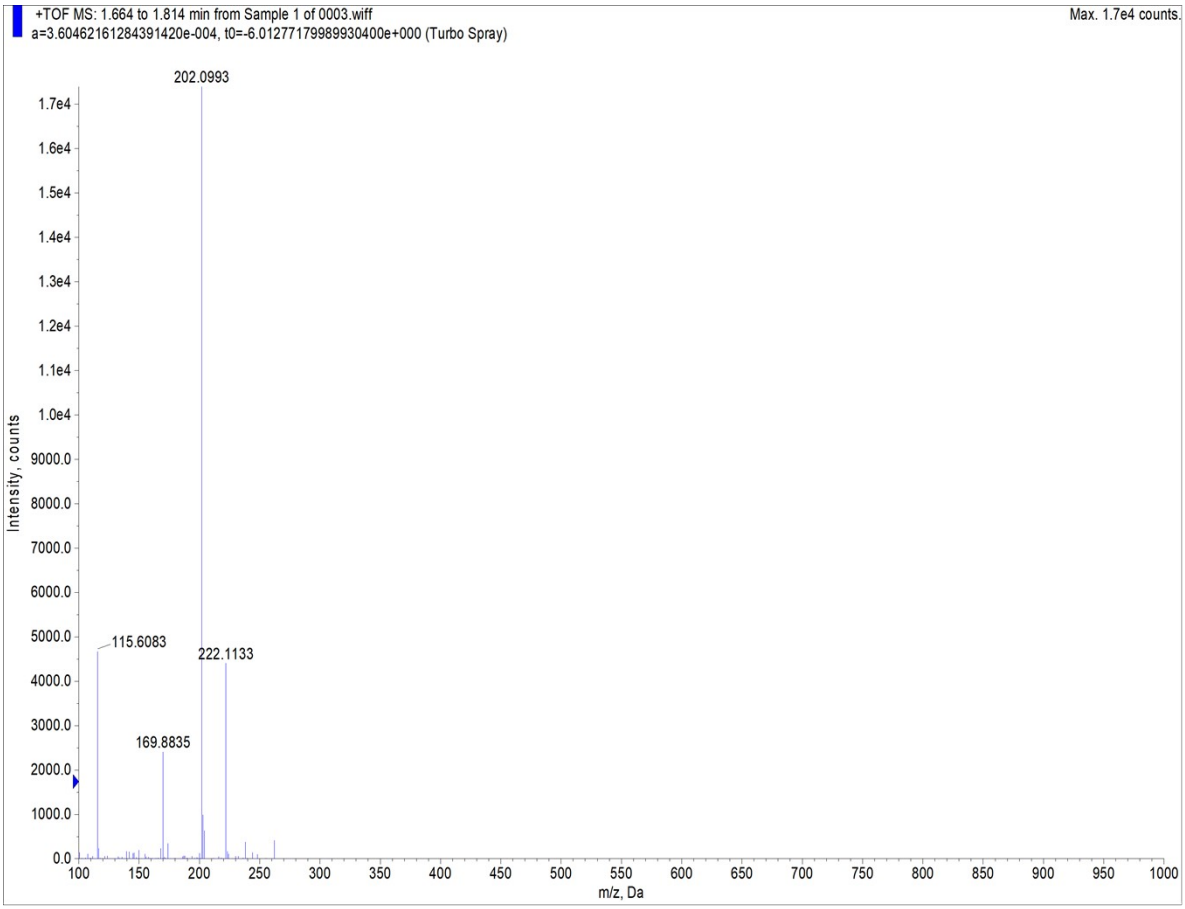
Ethyl 4-(*o*-tolyl)but-3-ynoate(3b)



HRMS

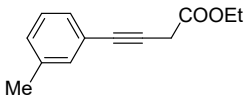


Ethyl 4-(*o*-tolyl)but-3-ynoate (3b)

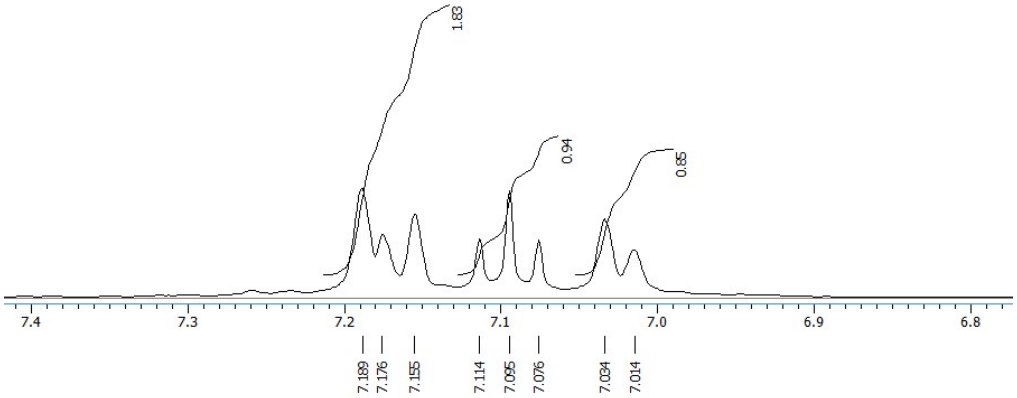


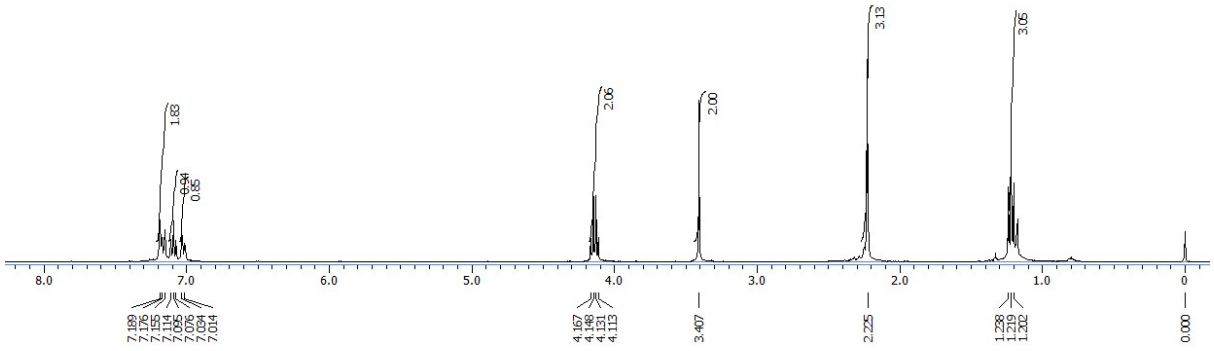
^{1}H NMR

(400 MHz, $CDCl_3$)



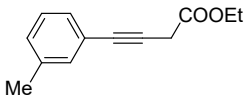
Ethyl 4-(*m*-tolyl)but-3-ynoate (3c)



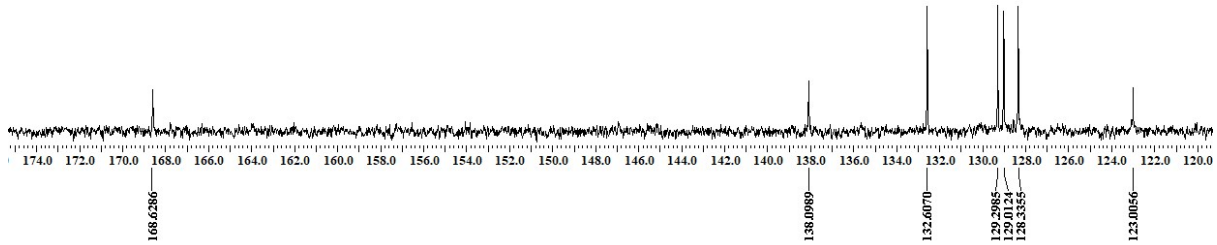


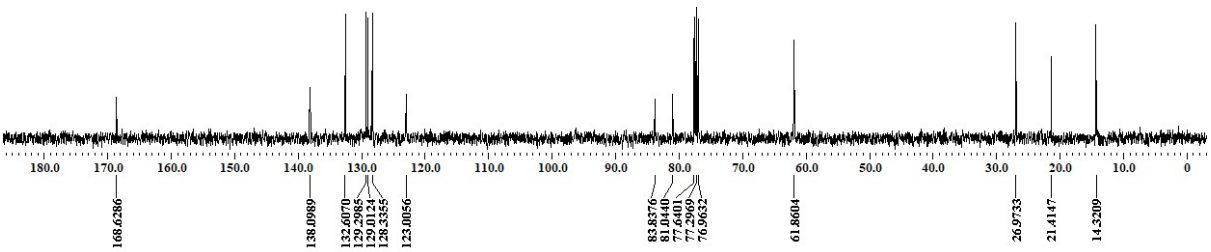
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

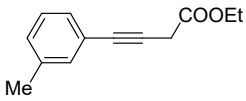


Ethyl 4-(*m*-tolyl)but-3-ynoate (3c)





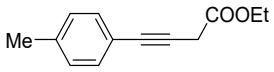
HRMS



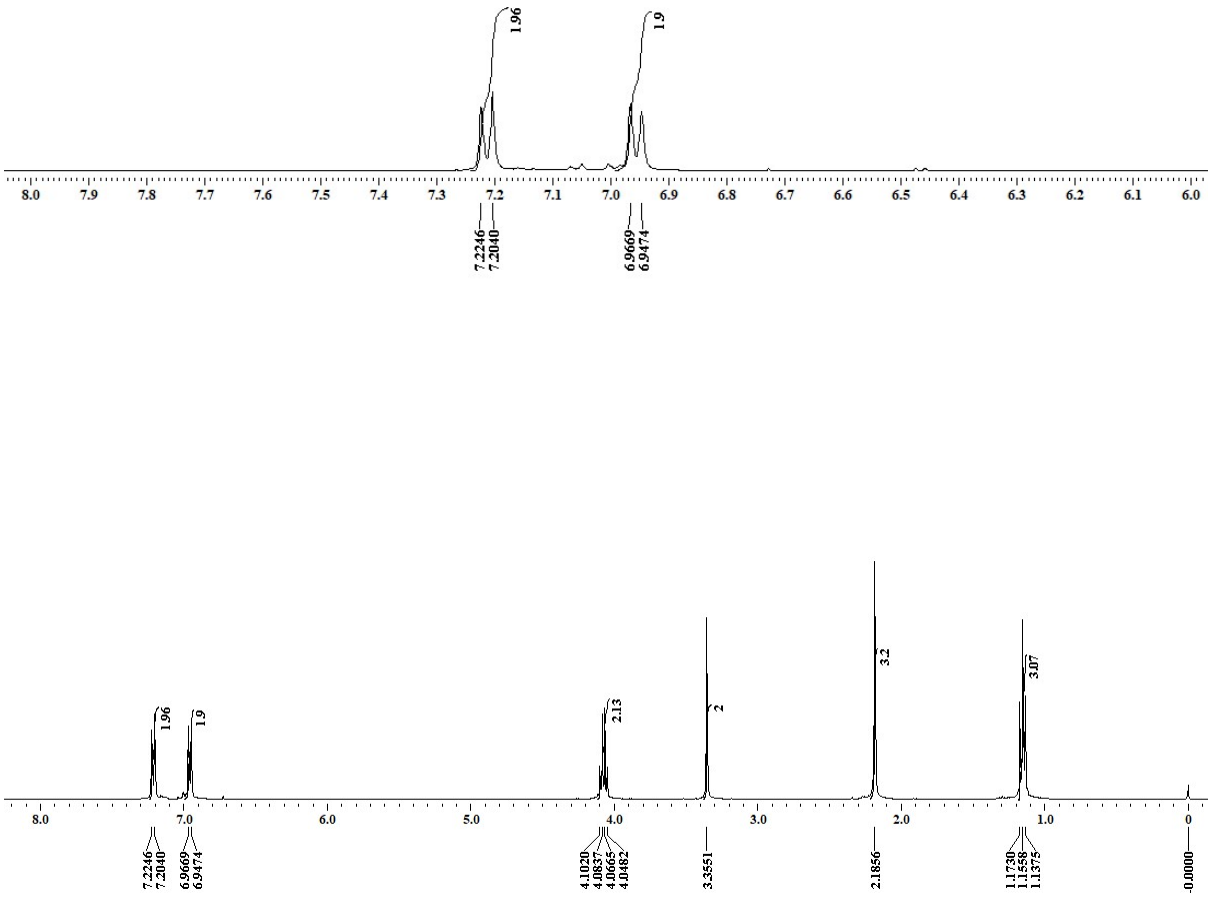
Ethyl 4-(*m*-tolyl)but-3-ynoate (3c)

^{1}H NMR

(400 MHz, $CDCl_3$)

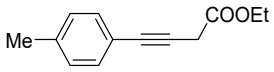


Ethyl 4-(*p*-tolyl)but-3-ynoate (3d)

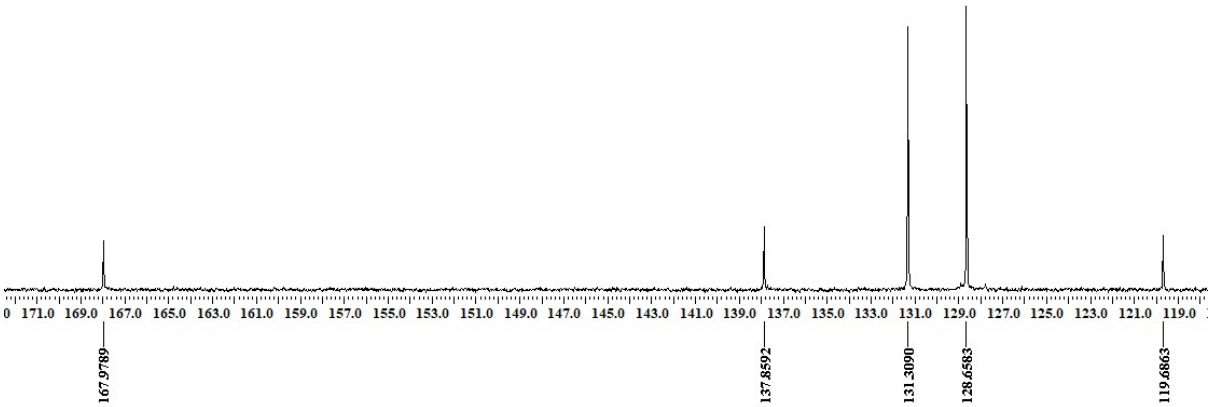


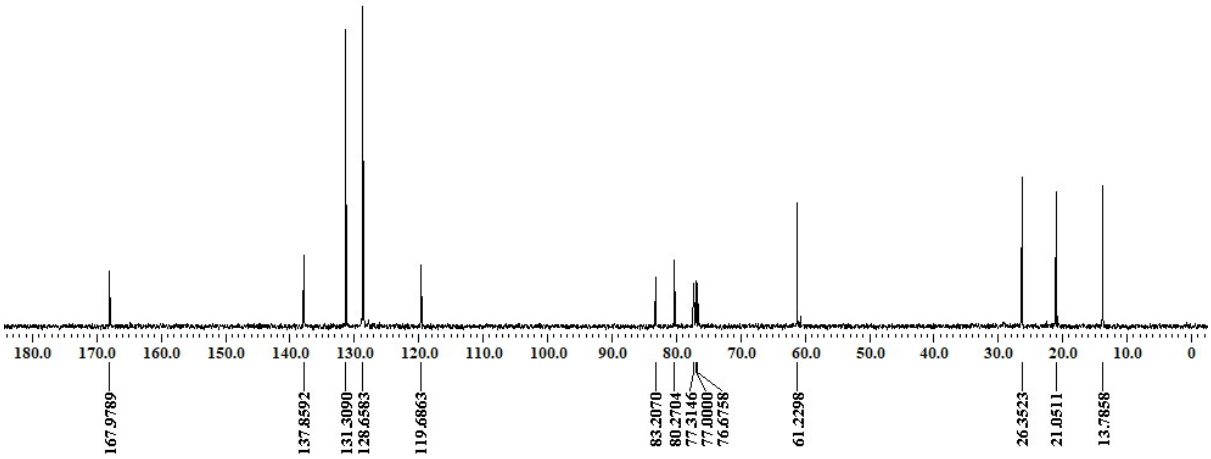
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

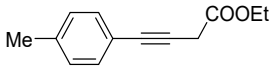


Ethyl 4-(*p*-tolyl)but-3-ynoate (3d)

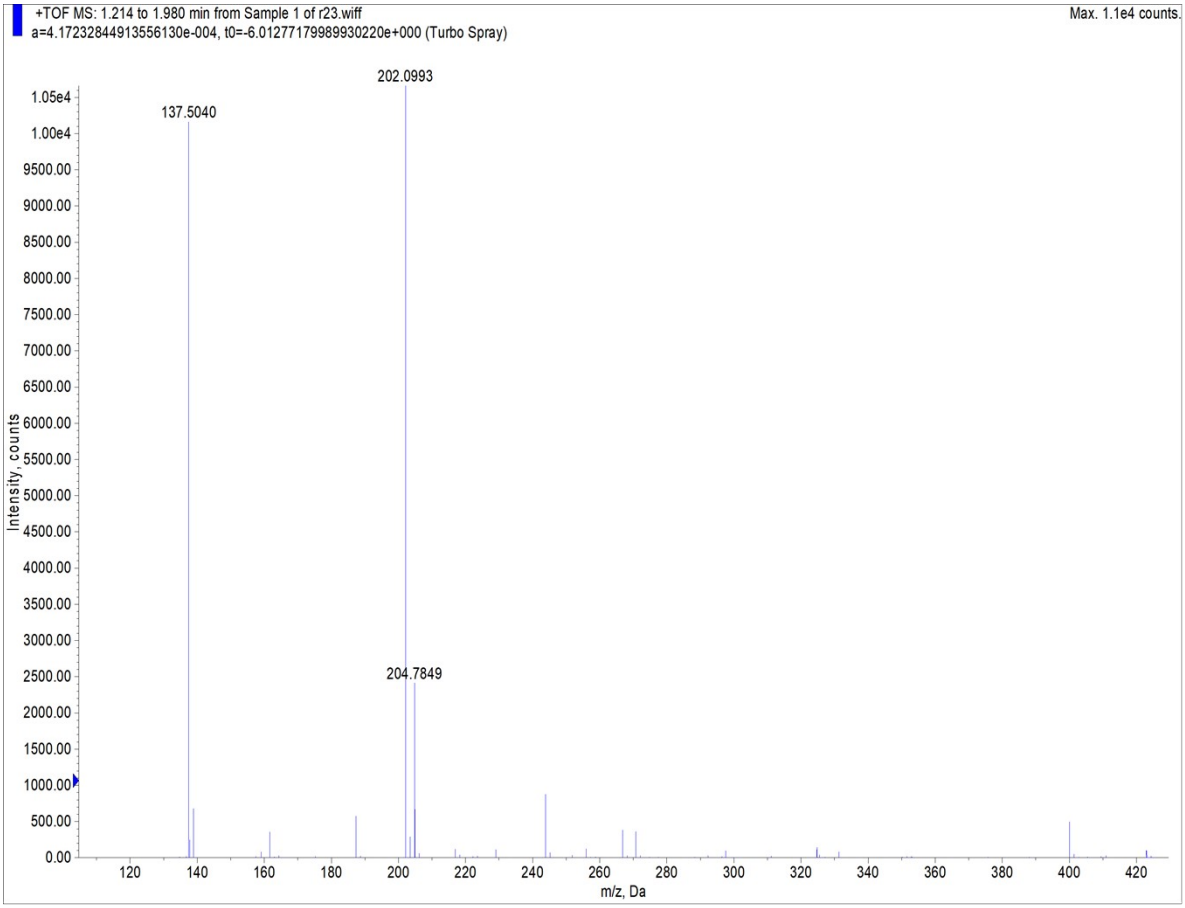




HRMS

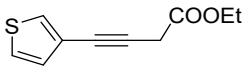


Ethyl 4-(*p*-tolyl)but-3-ynoate (3d)

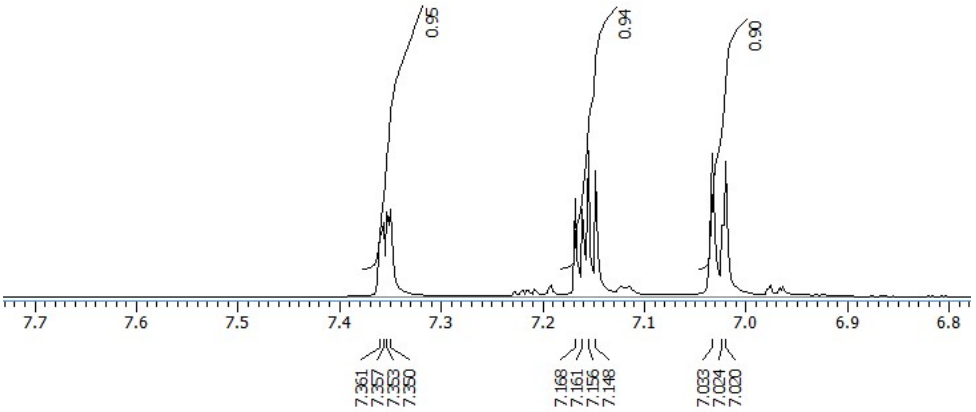


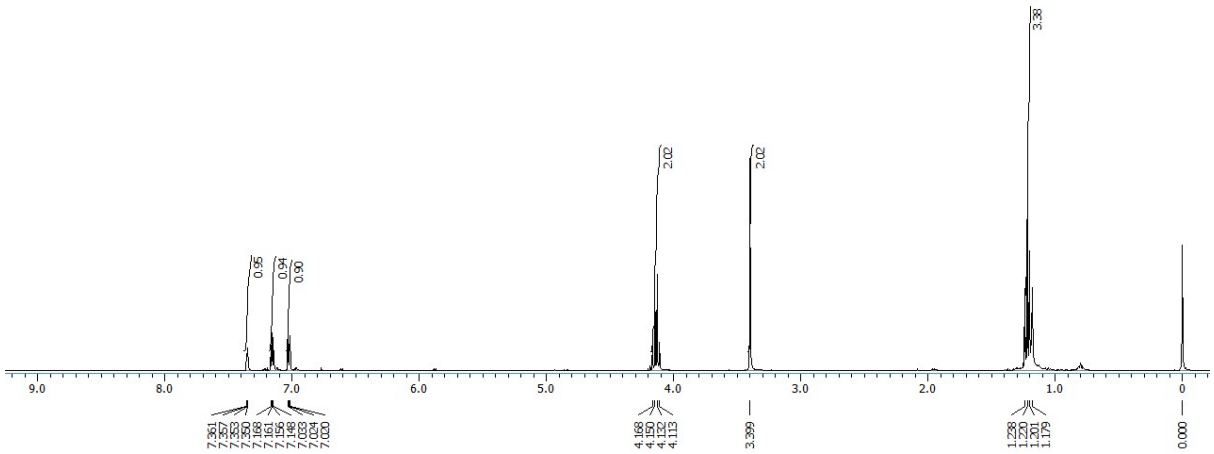
^{1}H NMR

(400 MHz, $CDCl_3$)



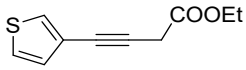
Ethyl 4-(thiophen-3-yl)but-3-ynoate (3e)



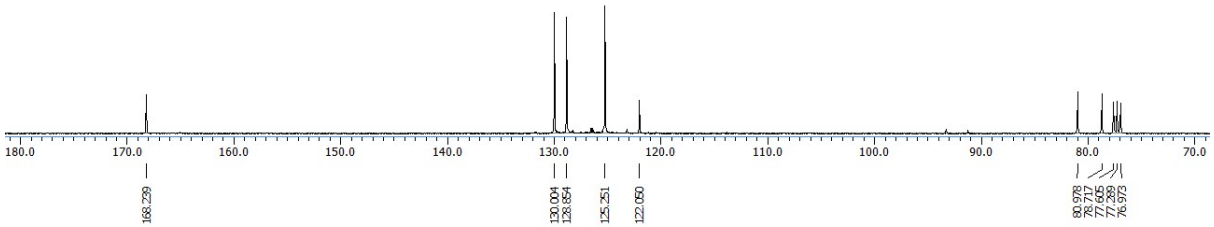


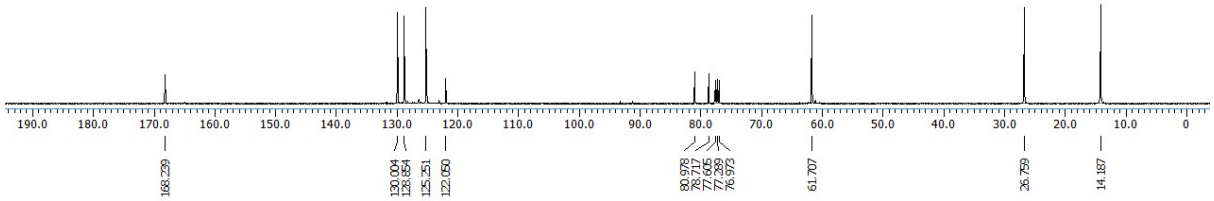
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

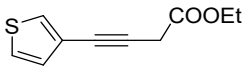


Ethyl4-(thiophen-3-yl)but-3-ynoate (3e)

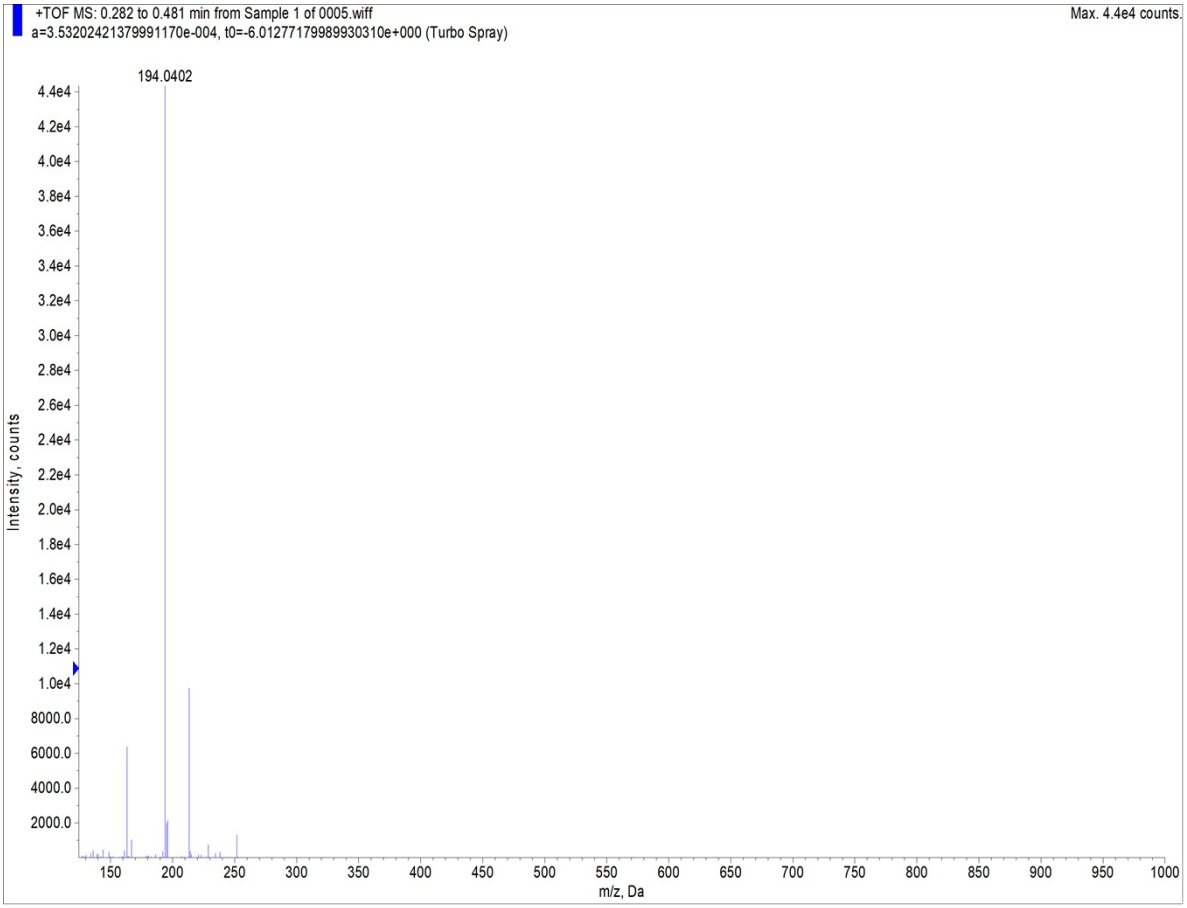




HRMS

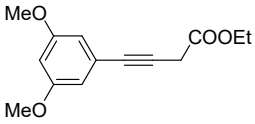


Ethyl 4-(thiophen-3-yl)but-3-ynoate (3e)

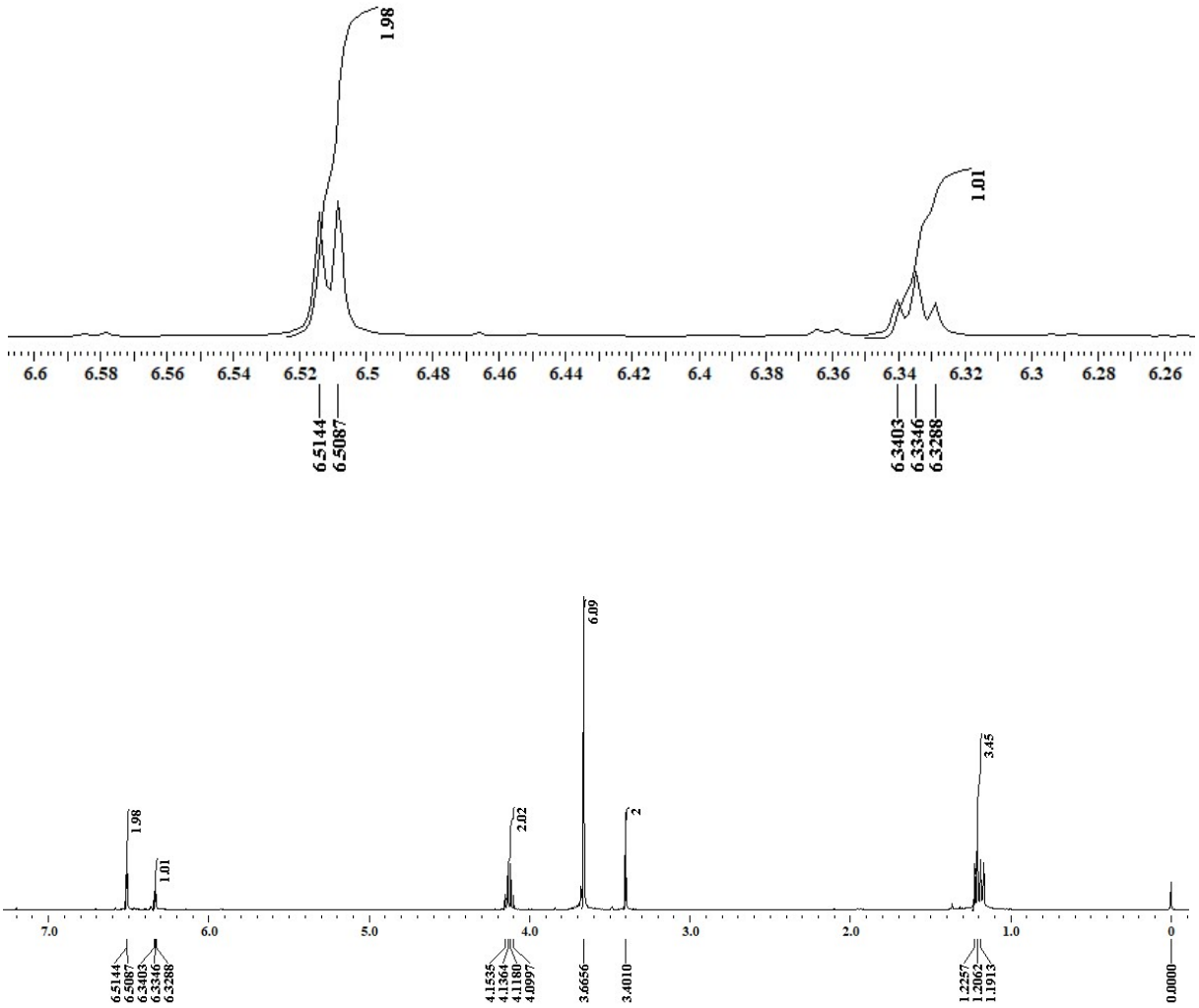


^{1}H NMR

(400 MHz, $CDCl_3$)

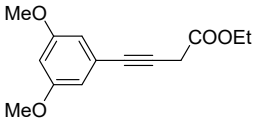


Ethyl 4-(3,5-dimethoxyphenyl)but-3-ynoate(3f)

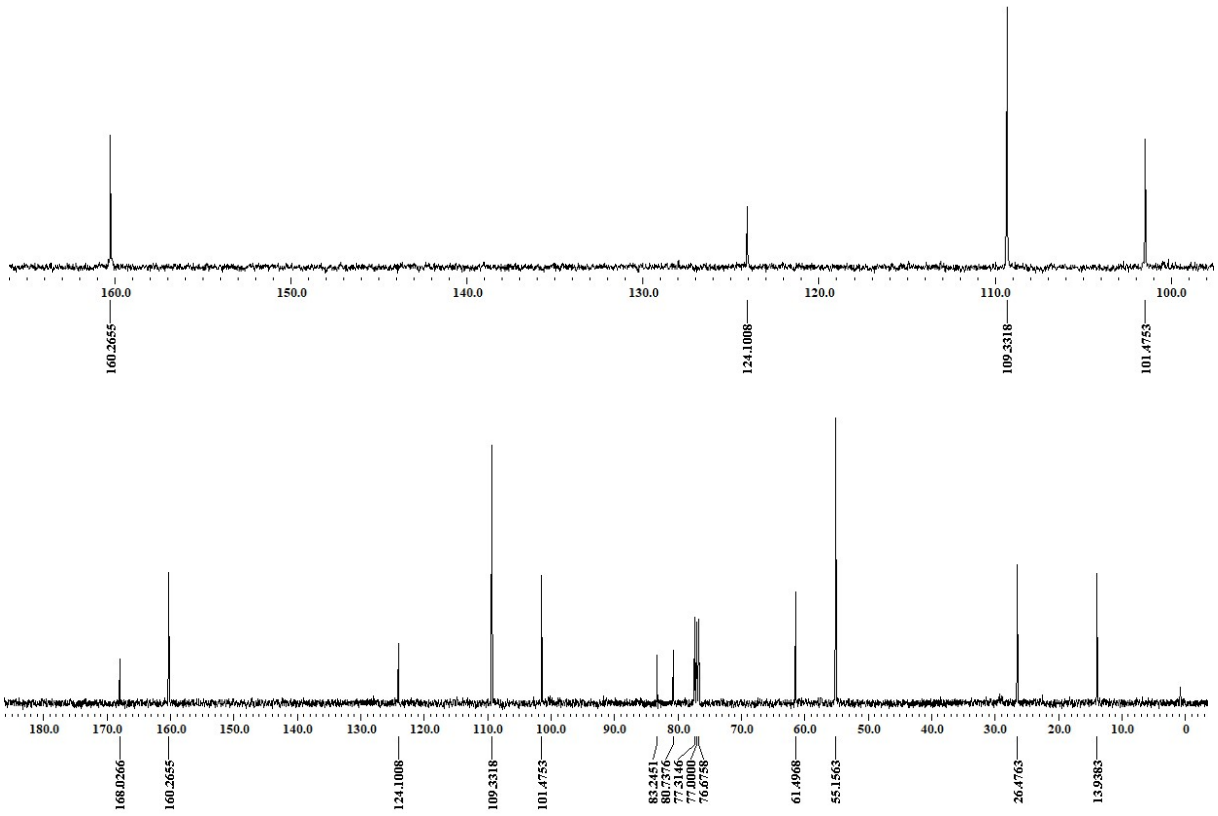


$^{13}C\{^{1}H\}$ NMR

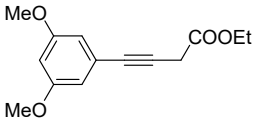
(100 MHz, $CDCl_3$)



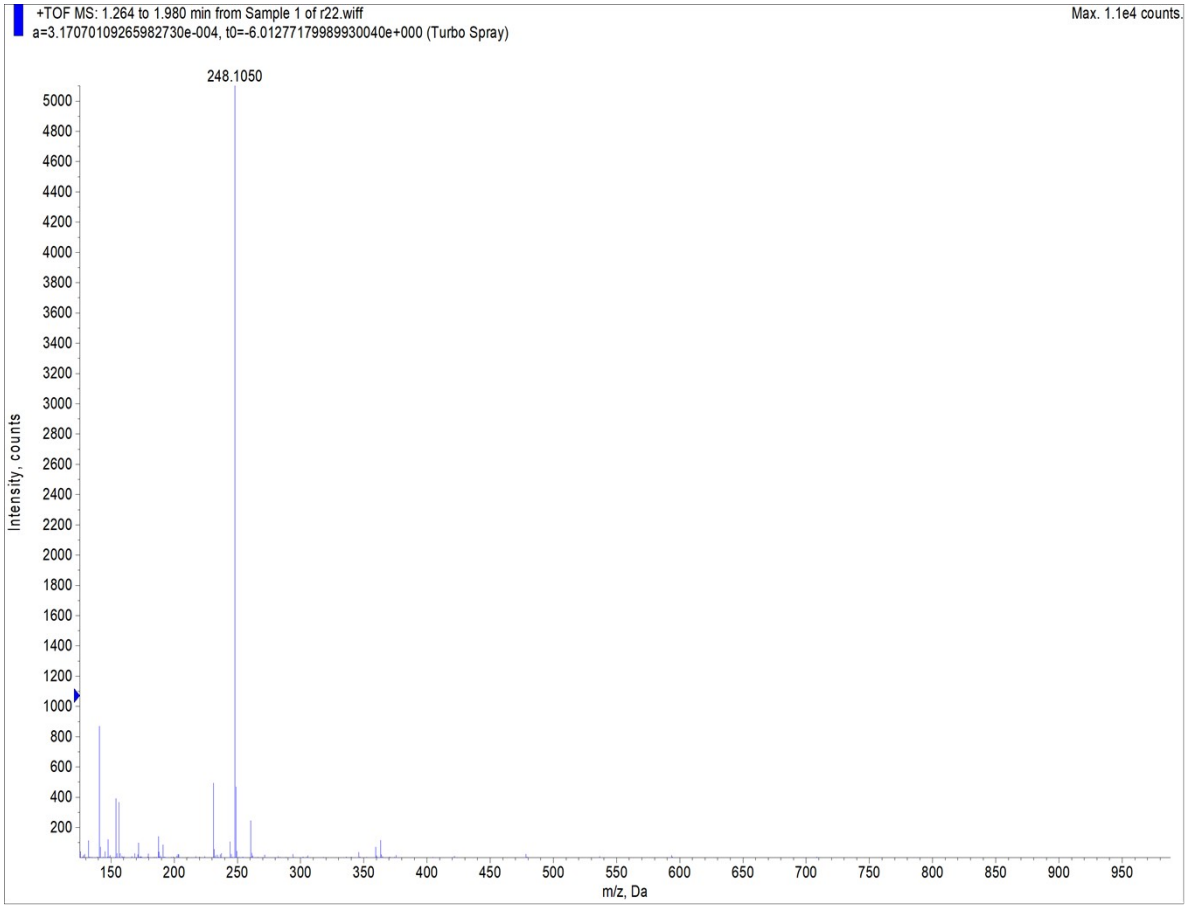
Ethyl 4-(3,5-dimethoxyphenyl)but-3-ynoate (3f)



HRMS

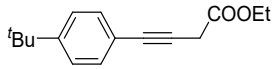


Ethyl 4-(3,5-dimethoxyphenyl)but-3-ynoate(3f)

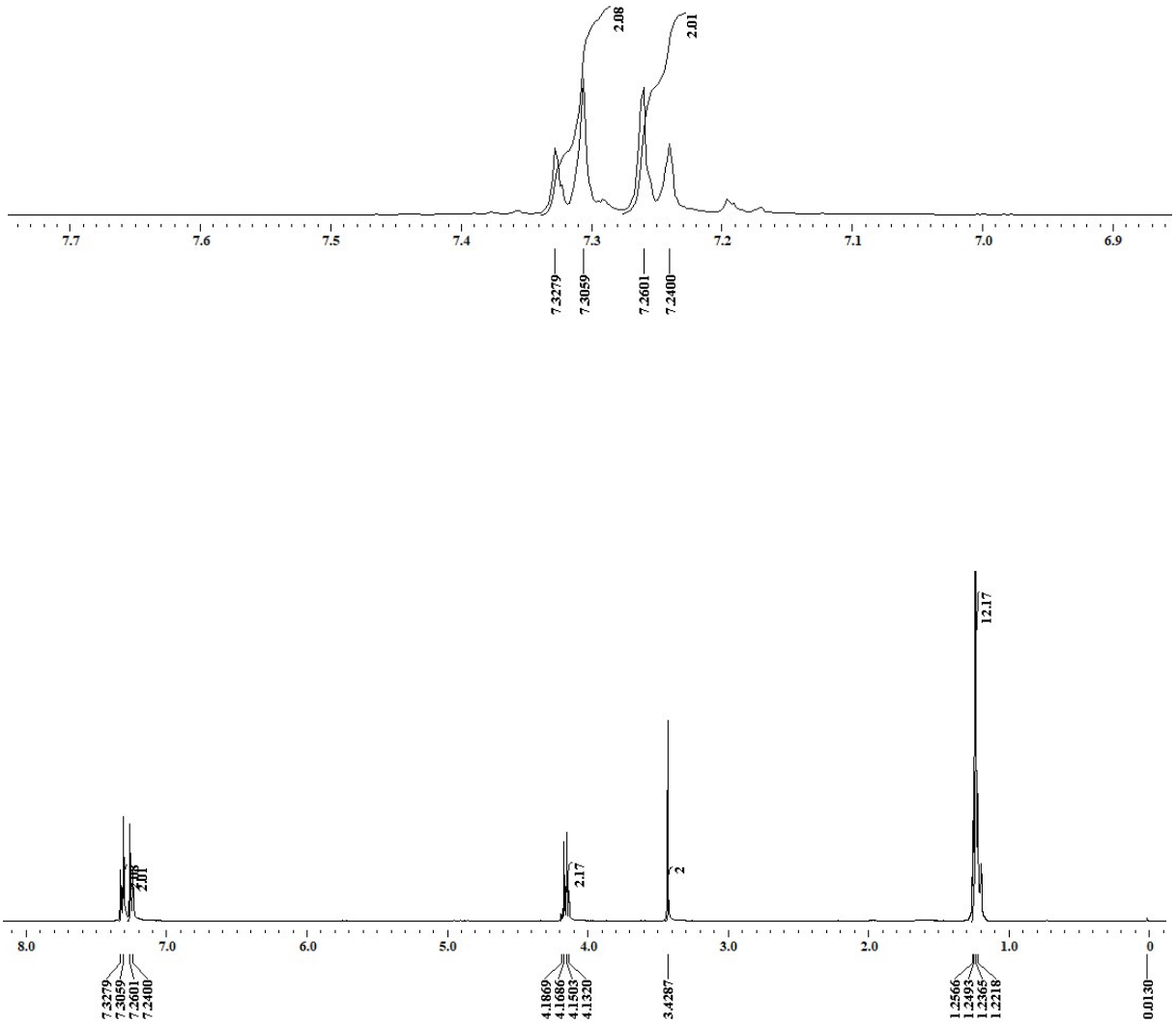


^{1}H NMR

(400 MHz, $CDCl_3$)

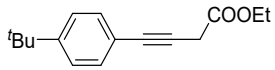


Ethyl 4-(4-(*tert*-butyl)phenyl)but-3-ynoate (3g)

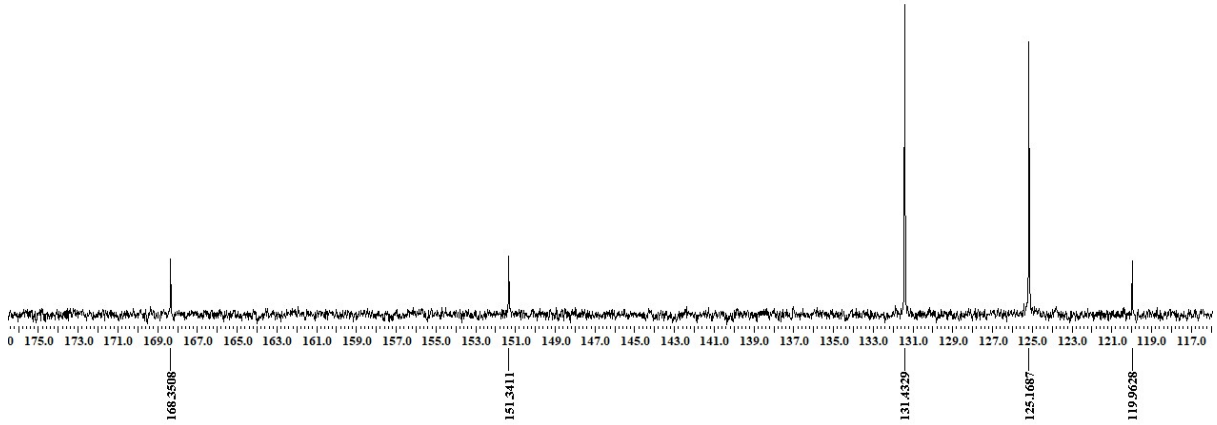


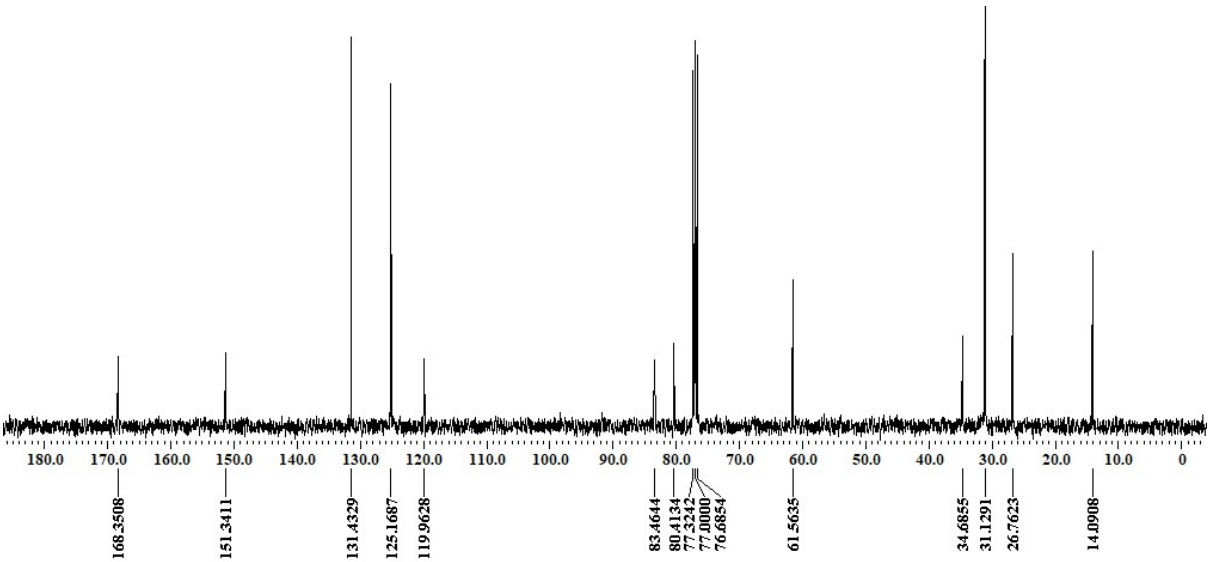
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

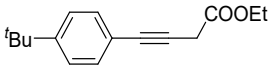


Ethyl 4-(4-(*tert*-butyl)phenyl)but-3-ynoate (3g)

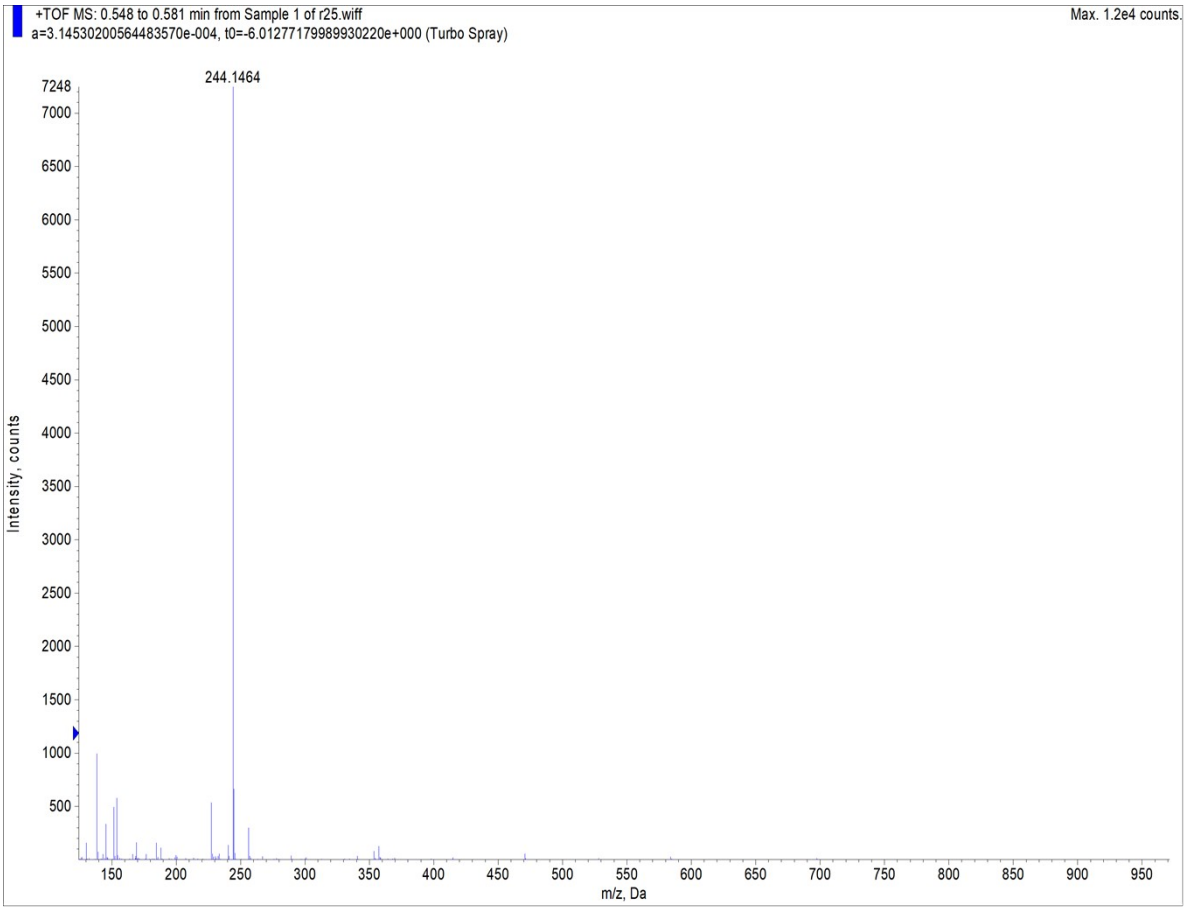




HRMS

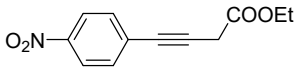


Ethyl 4-(4-(*tert*-butyl)phenyl)but-3-ynoate(3g)

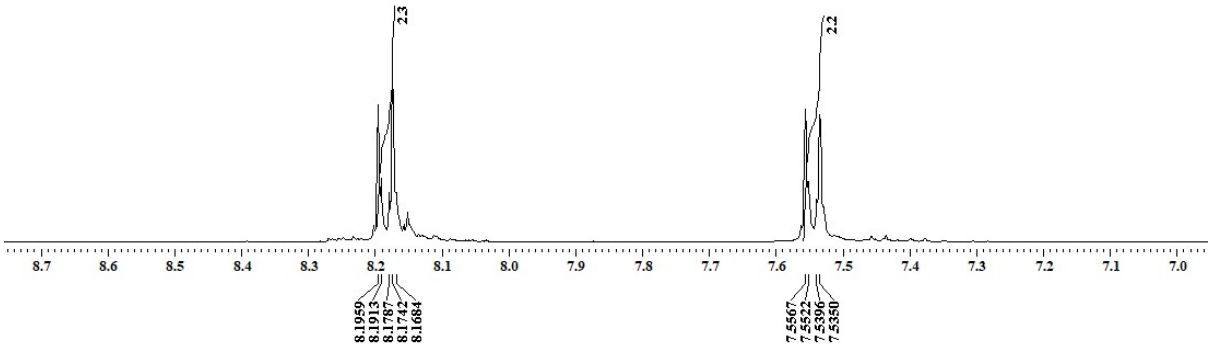


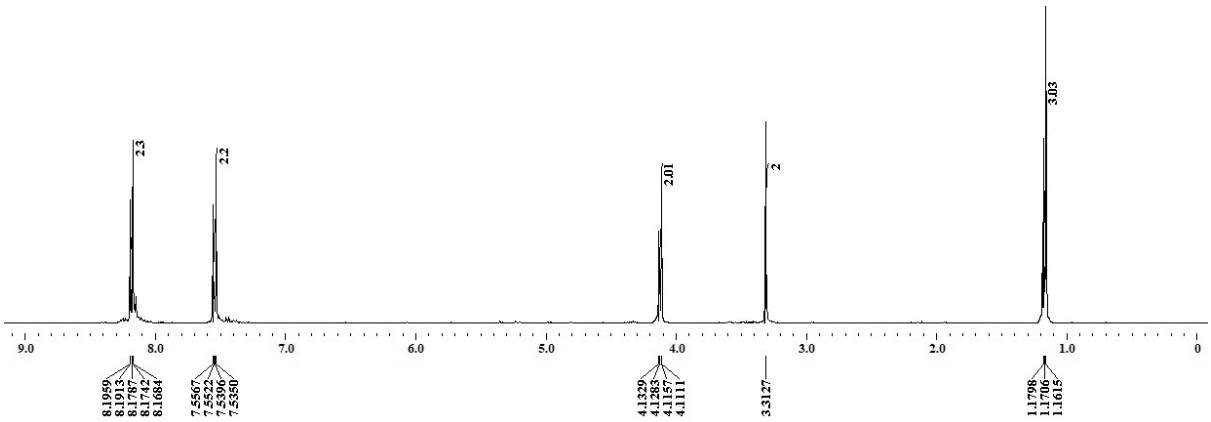
^{1}H NMR

(400 MHz, $CDCl_3$)



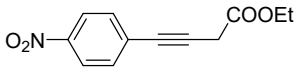
Ethyl 4-(4-nitrophenyl)but-3-ynoate (3h)



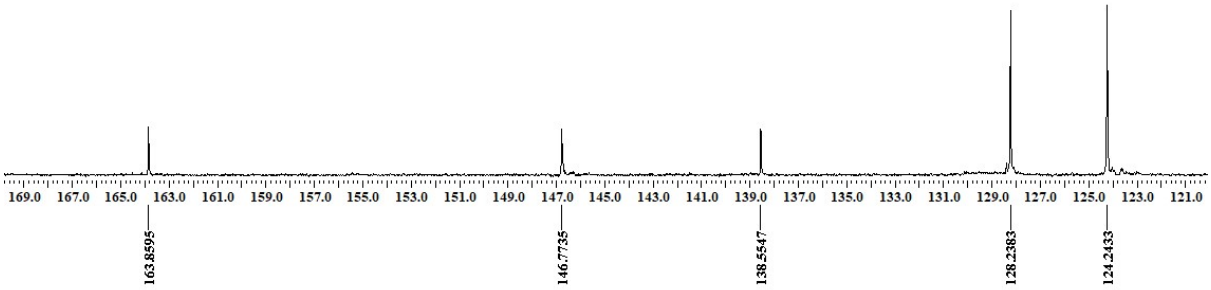


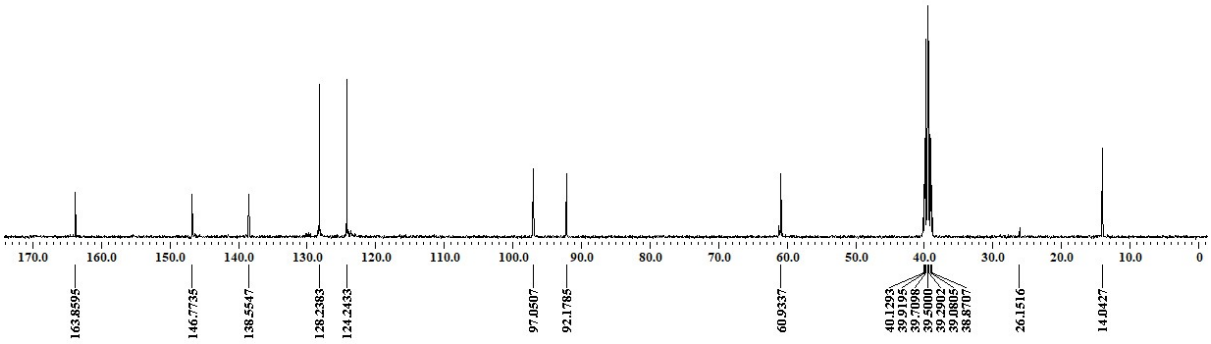
$^{13}C\{^{1}H\}$ NMR

(100 MHz, DMSO-d_6)

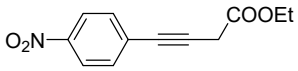


Ethyl 4-(4-nitrophenyl)but-3-ynoate (3h)

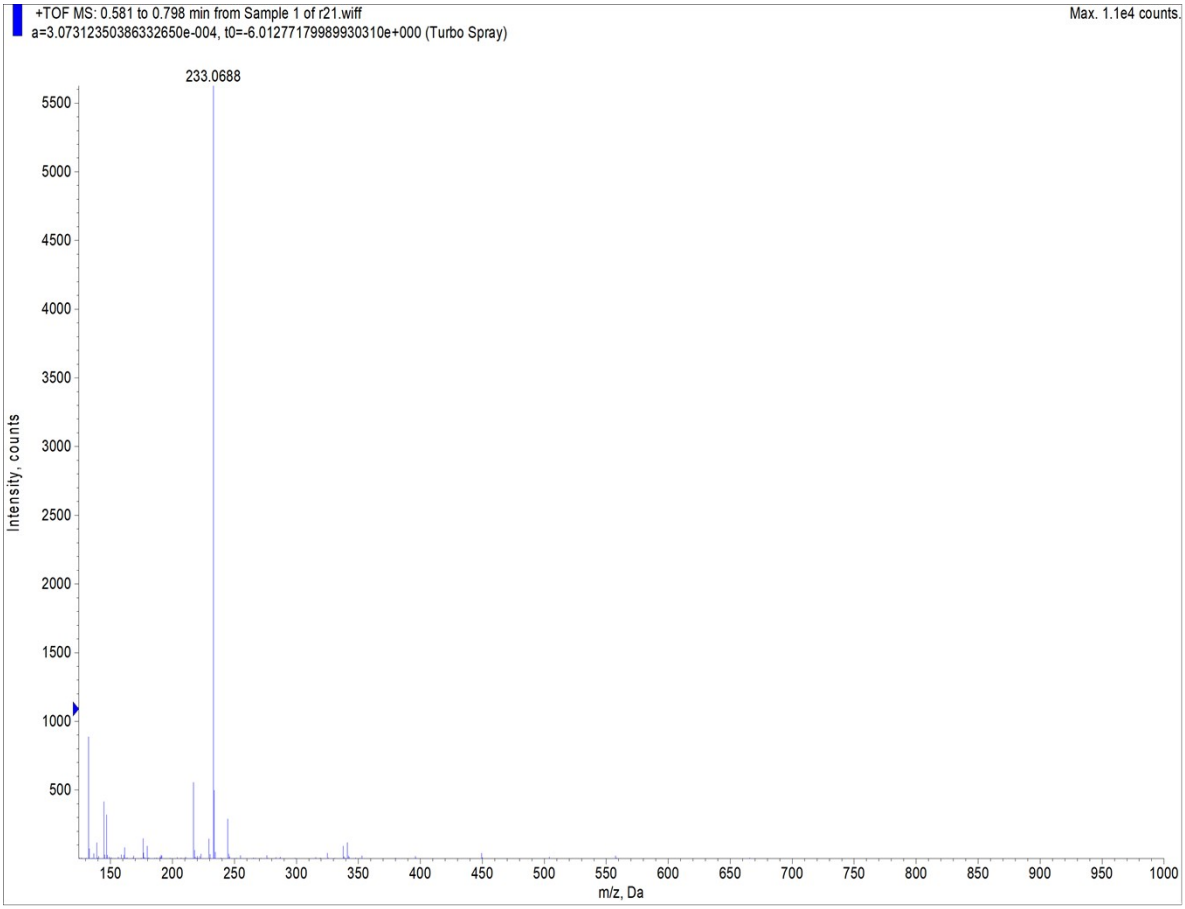




HRMS

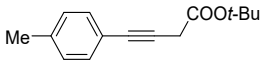


Ethyl 4-(4-nitrophenyl)but-3-ynoate (3h)

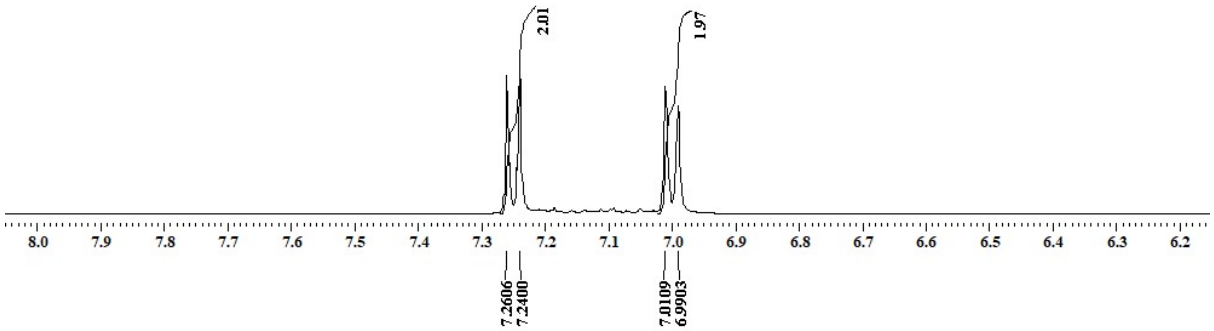


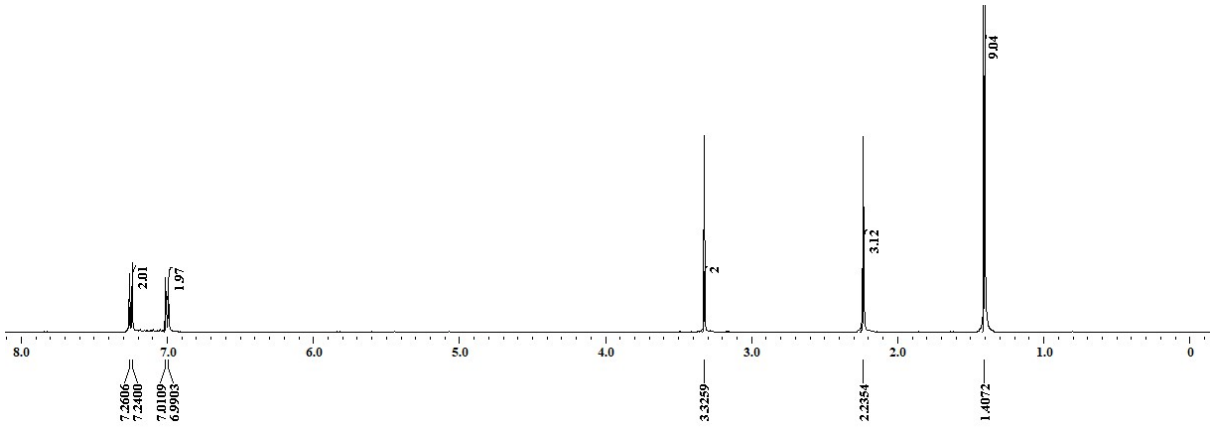
^{1}H NMR

(400 MHz, $CDCl_3$)



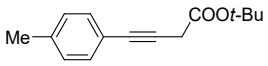
***tert*-Butyl 4-(*p*-tolyl)but-3-ynoate(3i)**



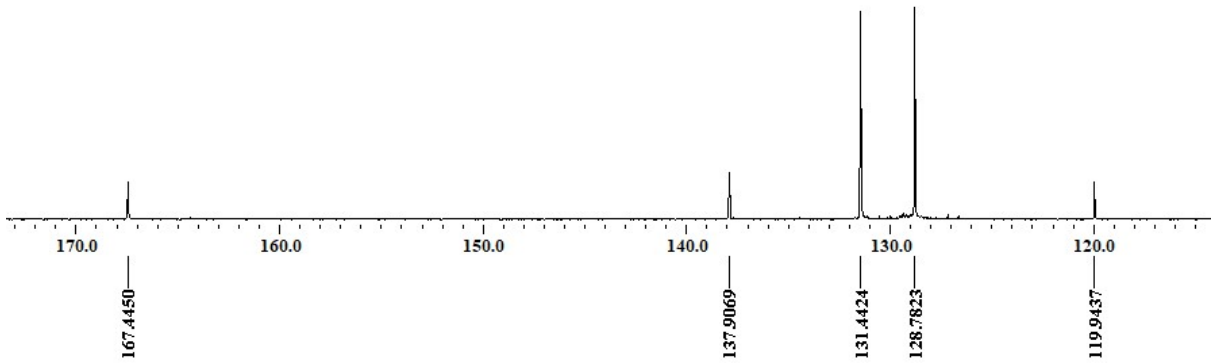


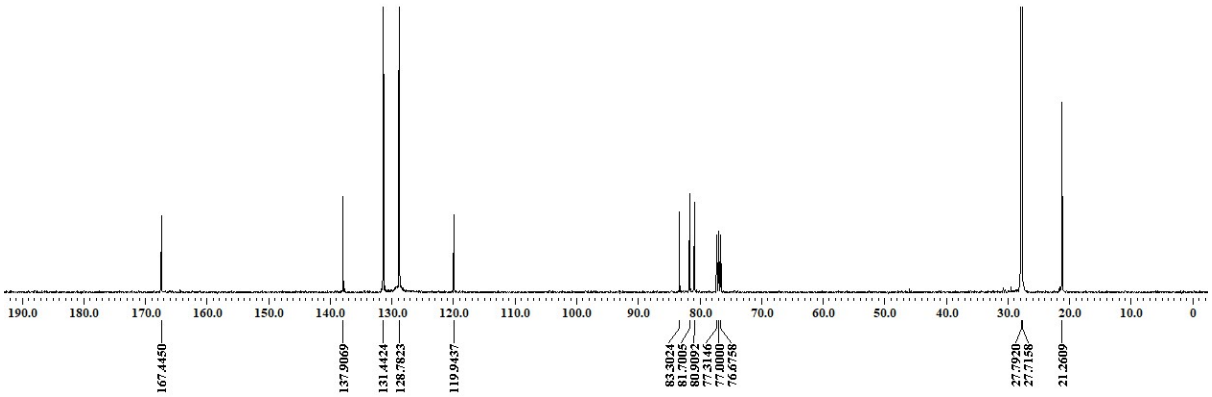
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

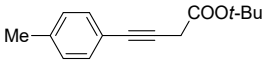


***tert*-Butyl 4-(*p*-tolyl)but-3-ynoate (3i)**

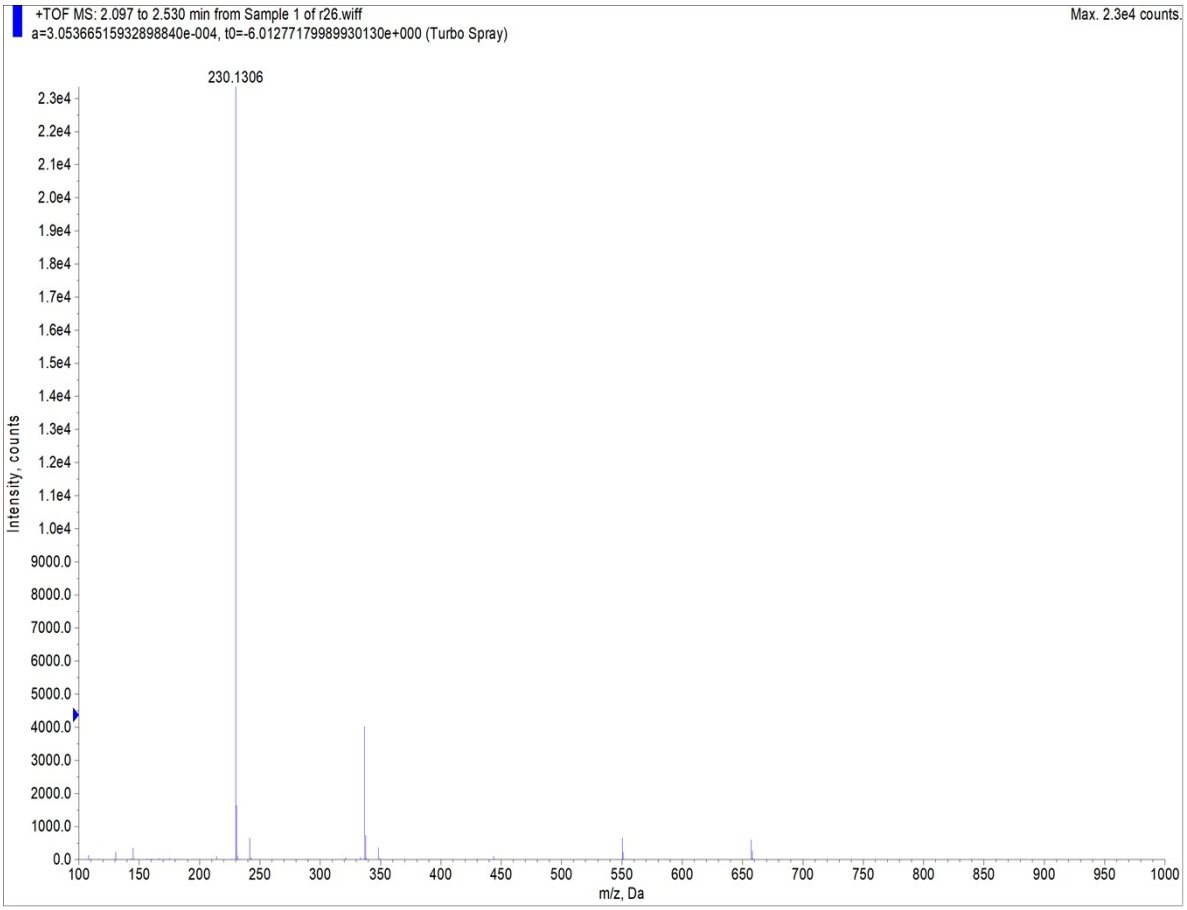




HRMS

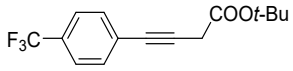


tert-Butyl 4-(*p*-tolyl)but-3-ynoate (3i)

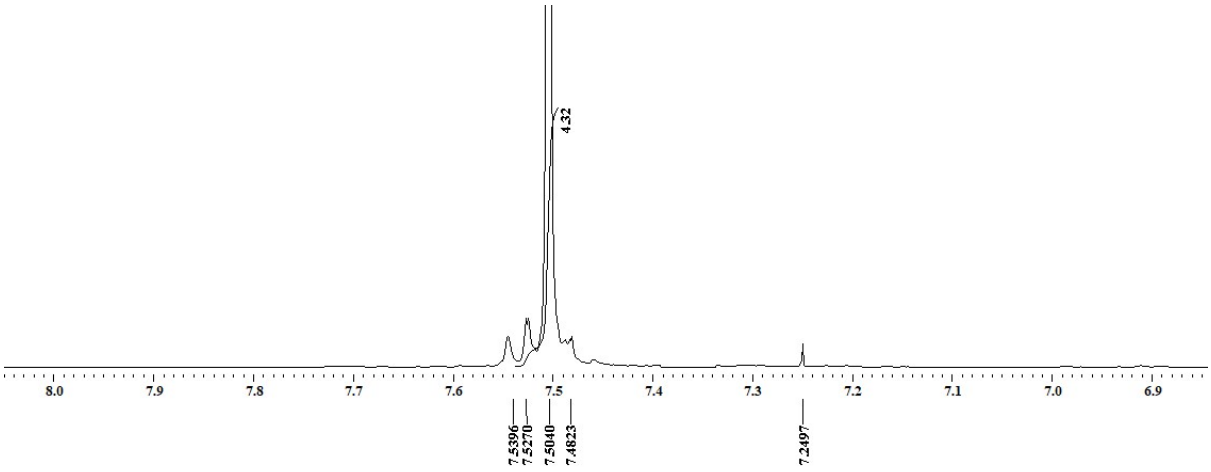


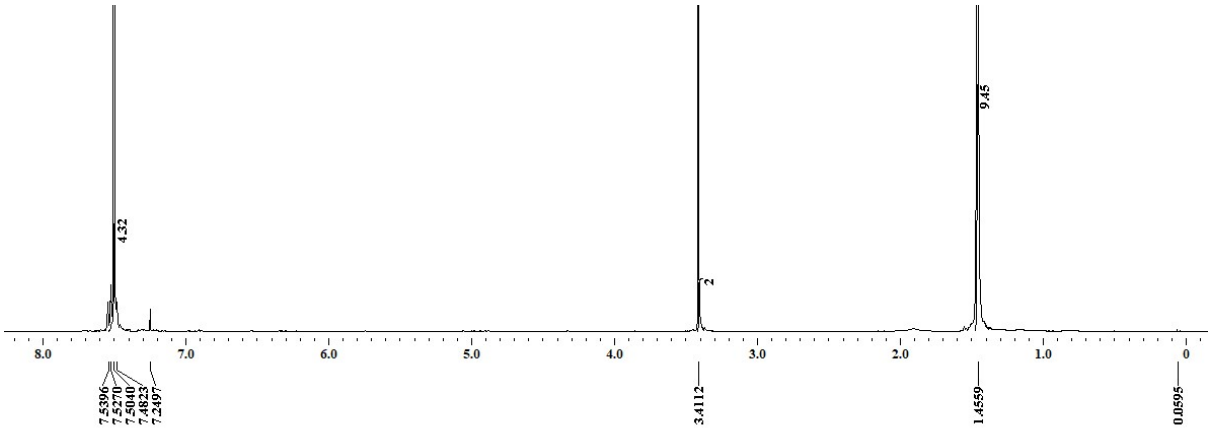
^{1}H NMR

(400 MHz, $CDCl_3$)



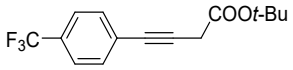
***tert*-Butyl 4-(4-(trifluoromethyl)phenyl)but-3-ynoate (3j)**



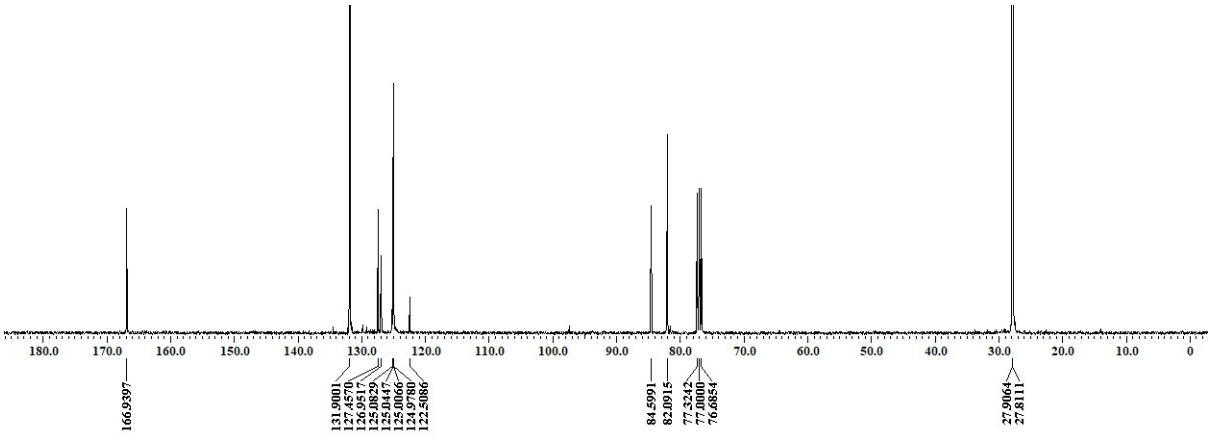


$^{13}C\{^{1}H\}$ NMR

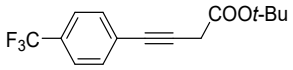
(100 MHz, $CDCl_3$)



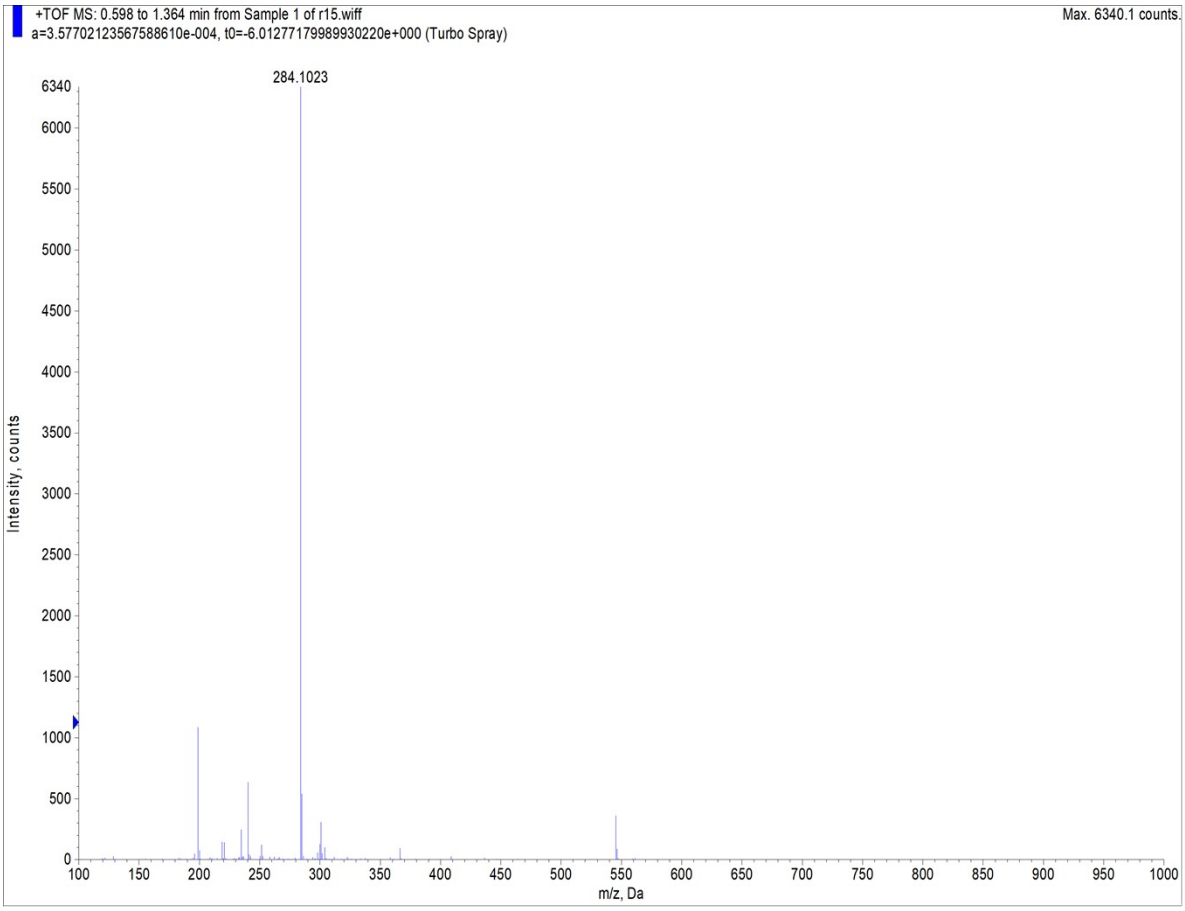
tert-Butyl 4-(4-(trifluoromethyl)phenyl)but-3-ynoate (3j)



HRMS

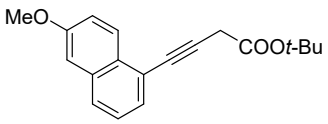


tert-Butyl 4-(4-(trifluoromethyl)phenyl)but-3-ynoate (3j)

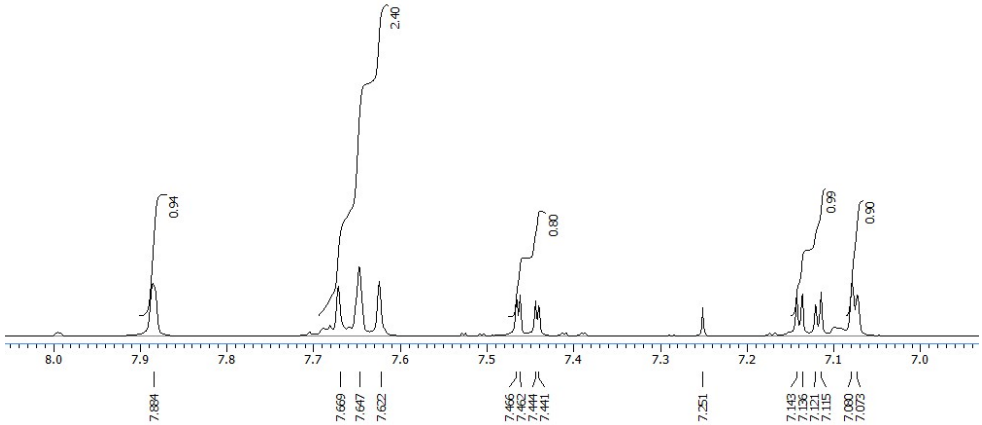


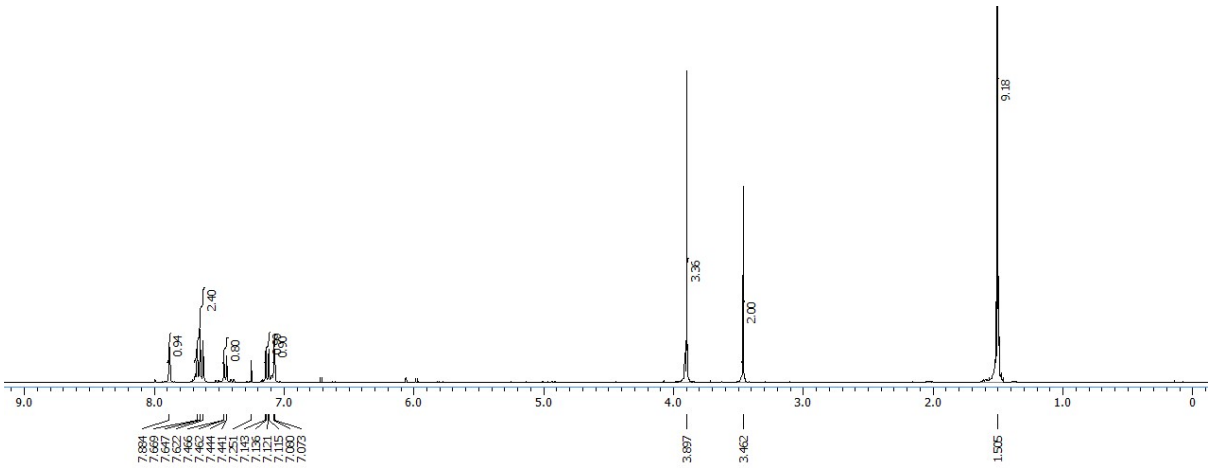
^{1}H NMR

(400 MHz, $CDCl_3$)



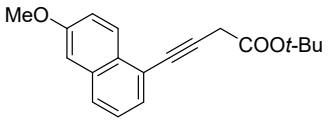
***tert*-Butyl 4-(6-methoxynaphthalen-1-yl)but-3-ynoate (3k)**



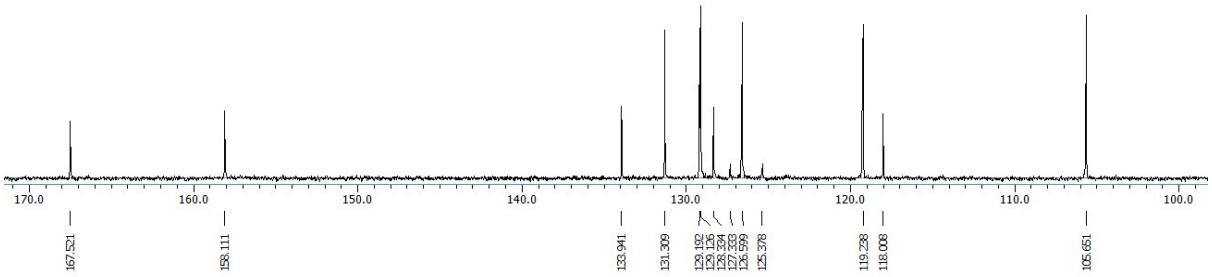


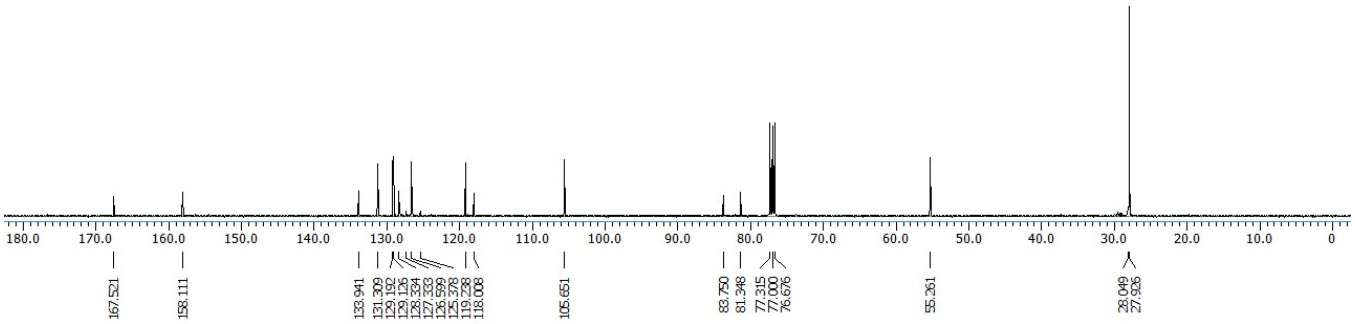
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

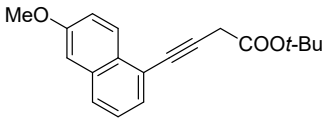


***tert*-Butyl 4-(6-methoxynaphthalen-1-yl)but-3-ynoate (3k)**

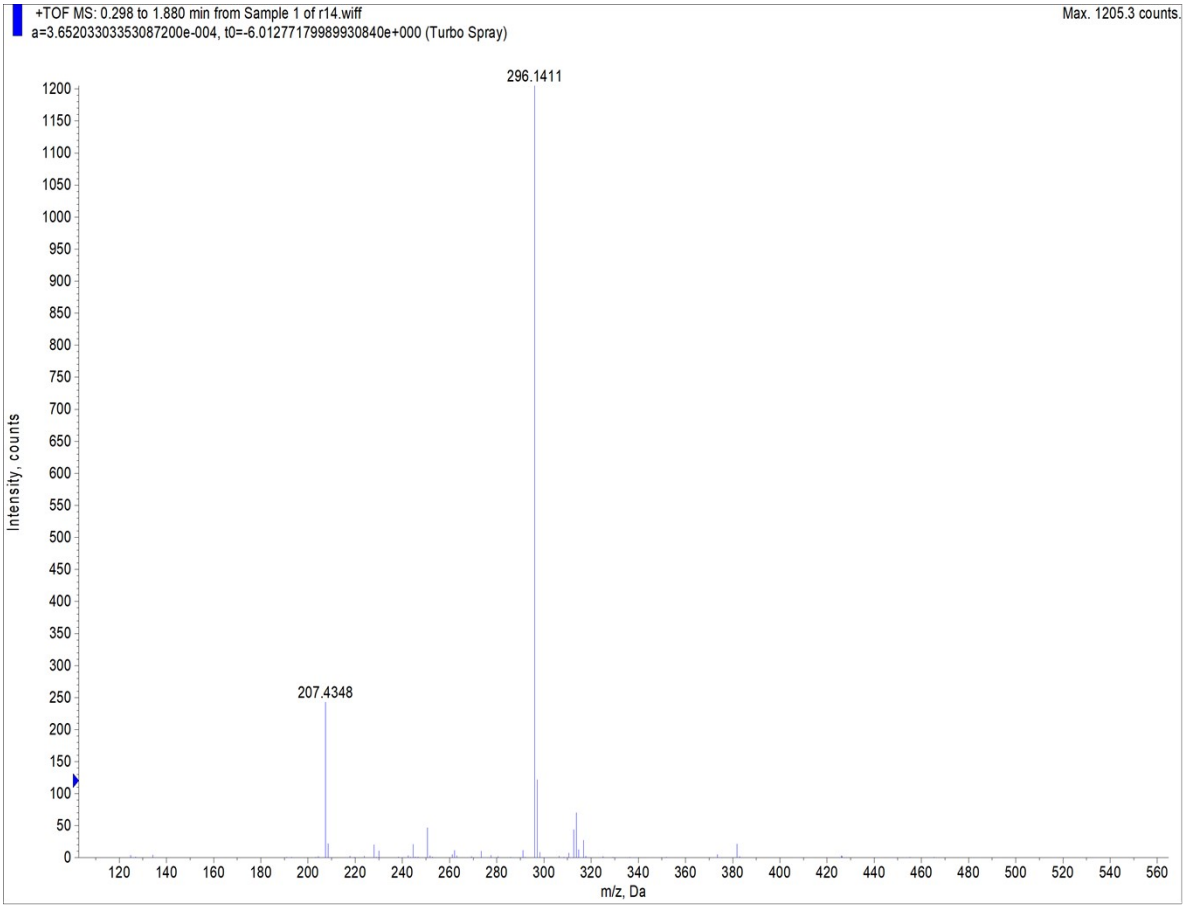




HRMS

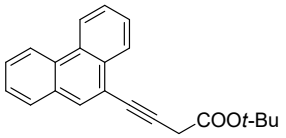


tert-Butyl 4-(6-methoxynaphthalen-1-yl)but-3-ynoate(3k)

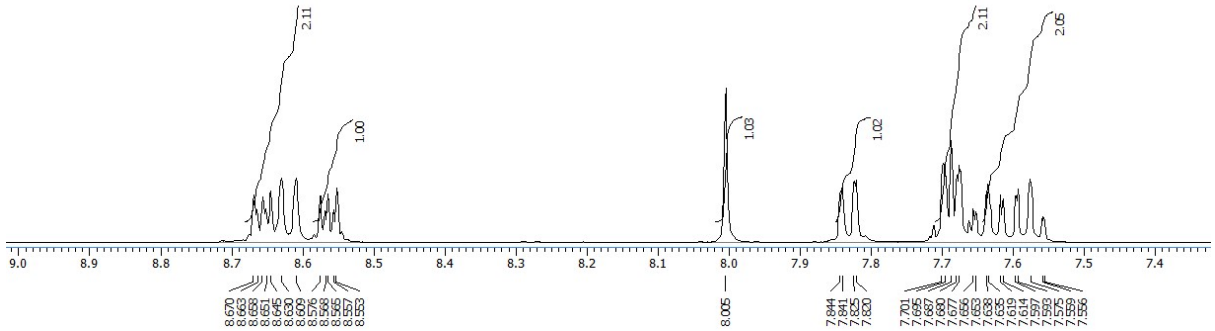


^{1}H NMR

(400 MHz, $CDCl_3$)



***tert*-Butyl 4-(phenanthren-9-yl)but-3-ynoate (3l)**



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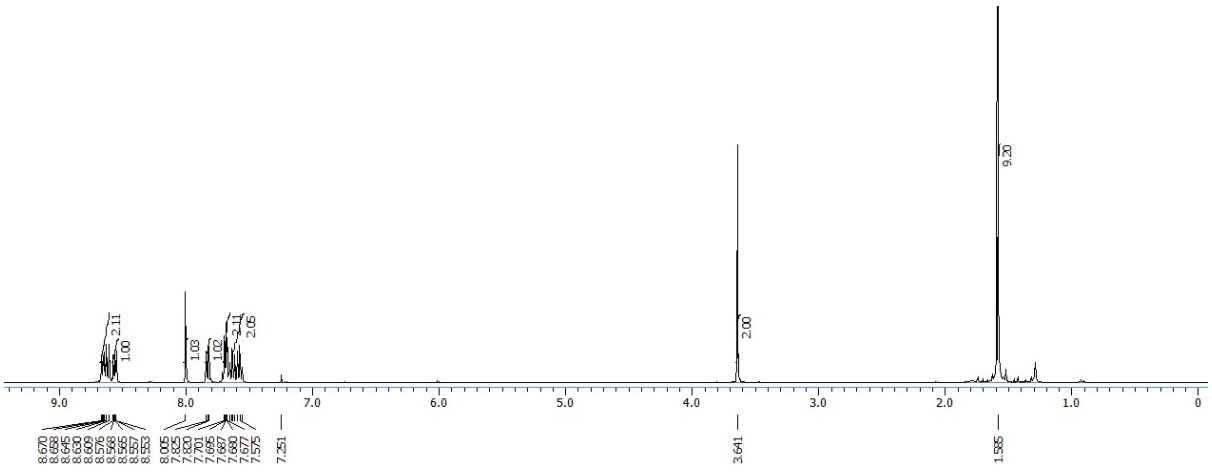
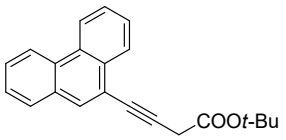
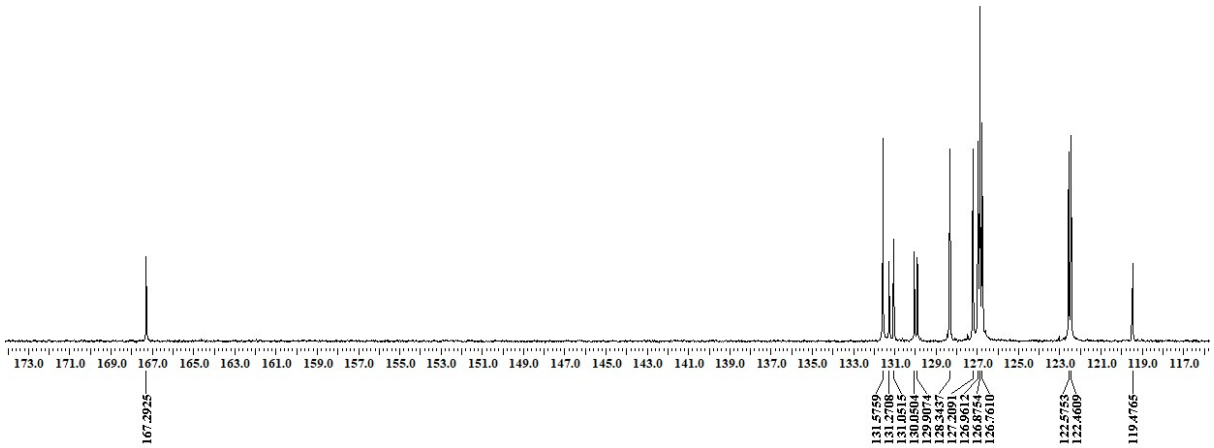


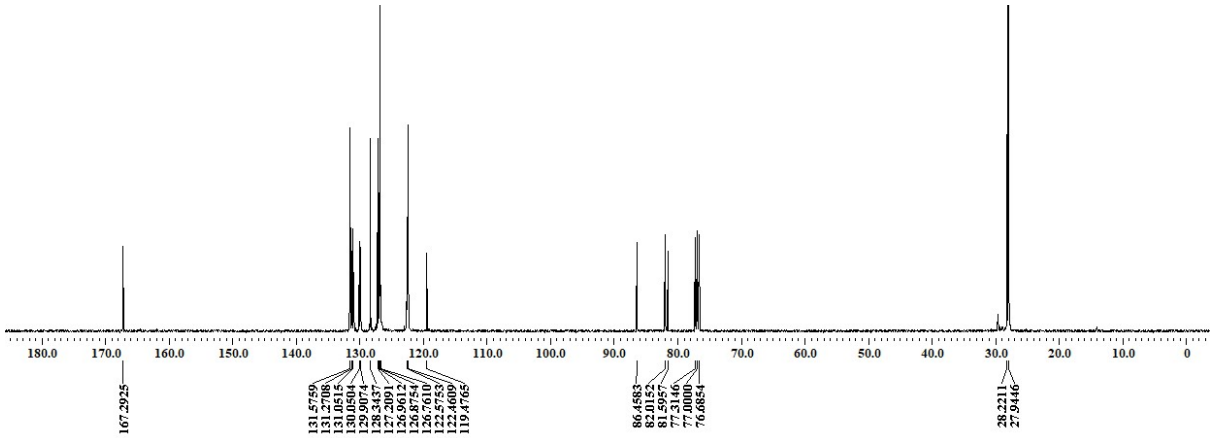
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

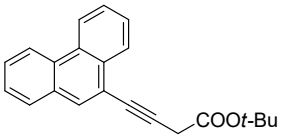


***tert*-Butyl 4-(phenanthren-9-yl)but-3-ynoate (3l)**

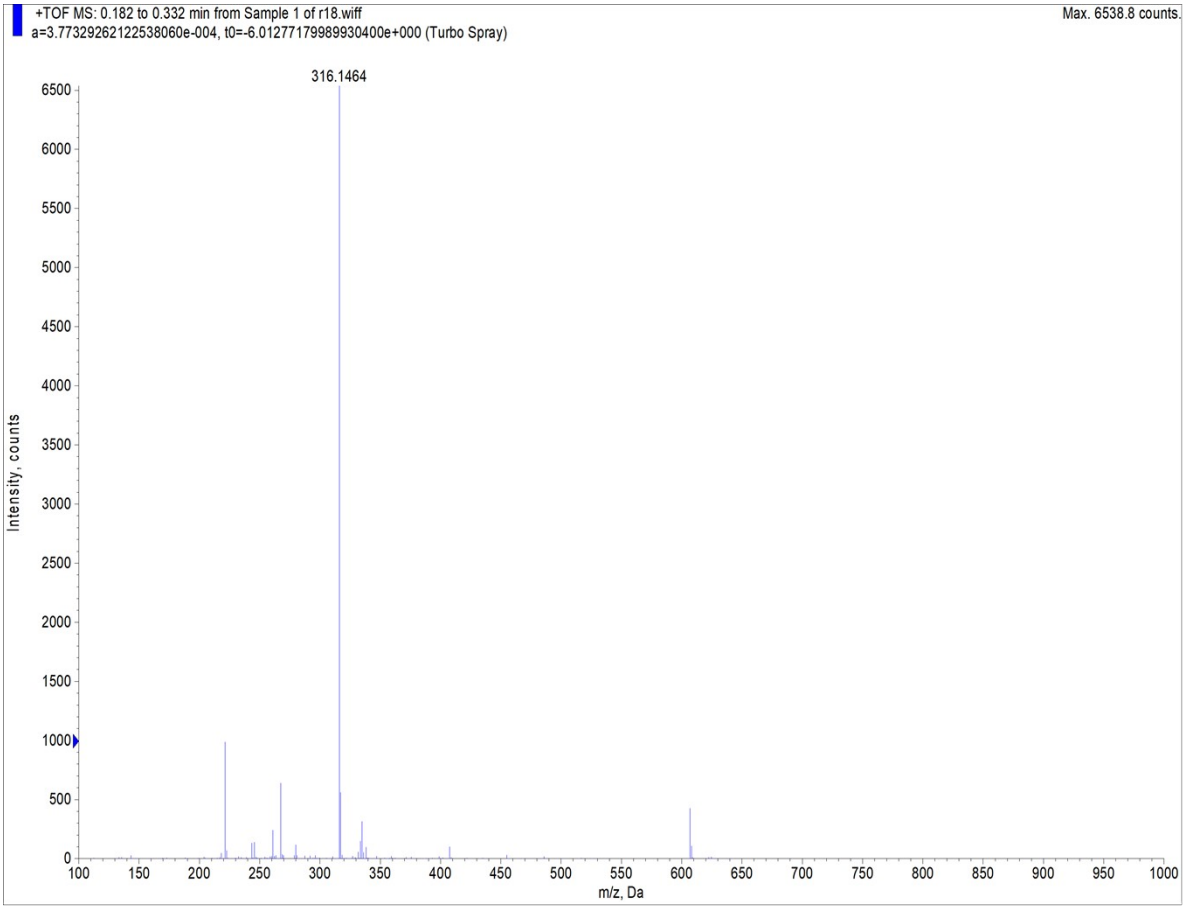




HRMS

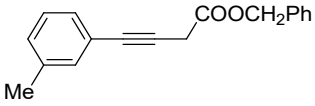


tert-Butyl 4-(phenanthren-9-yl)but-3-ynoate (3l)

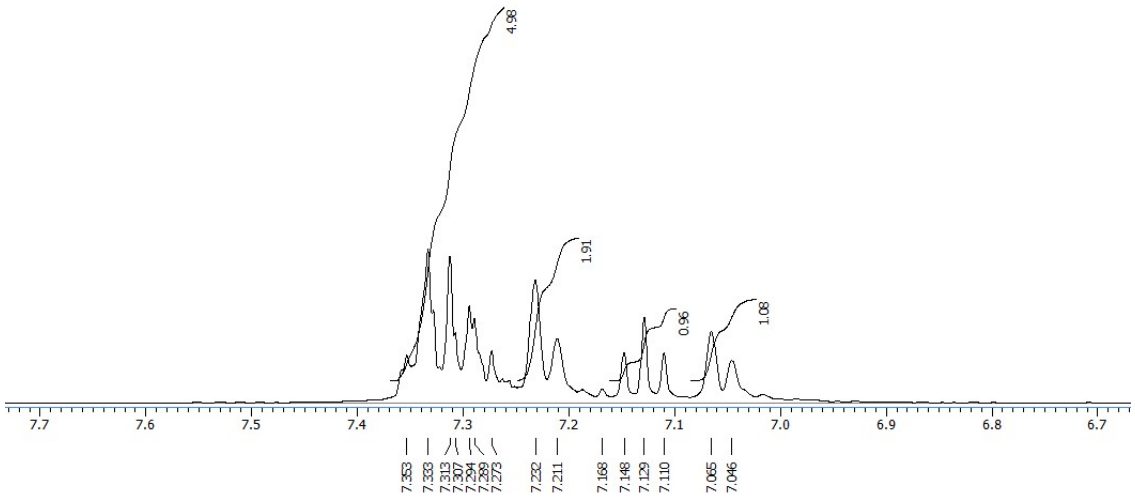


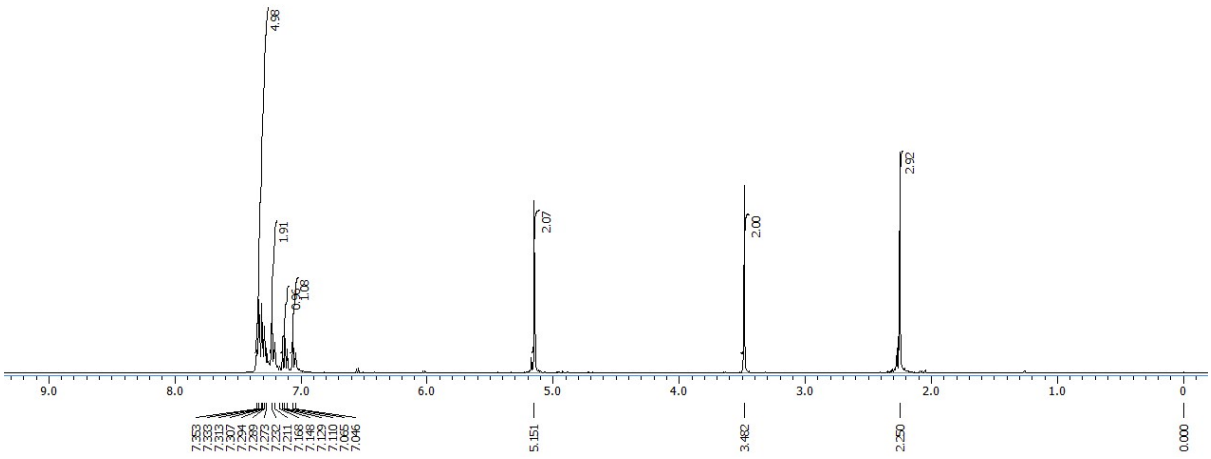
^{1}H NMR

(400 MHz, $CDCl_3$)



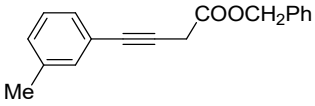
Benzyl 4-(*m*-tolyl)but-3-ynoate(3m)



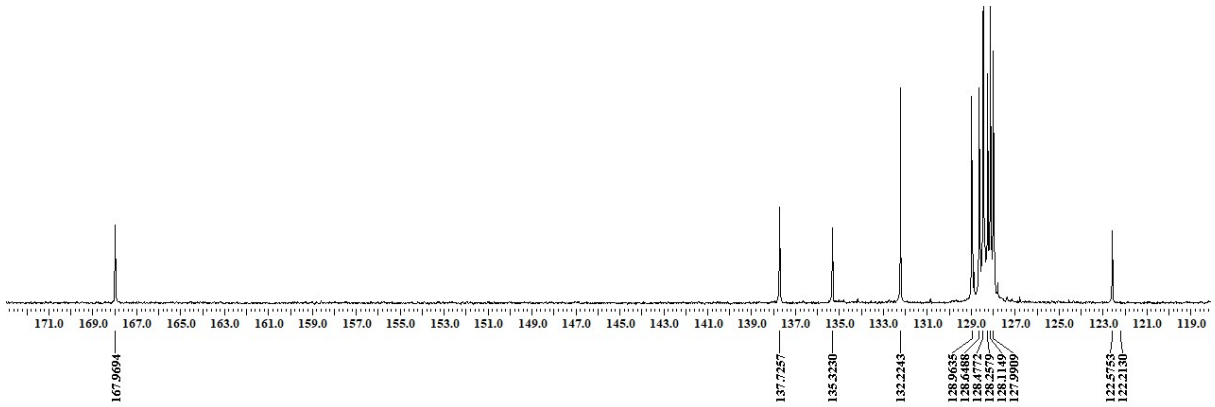


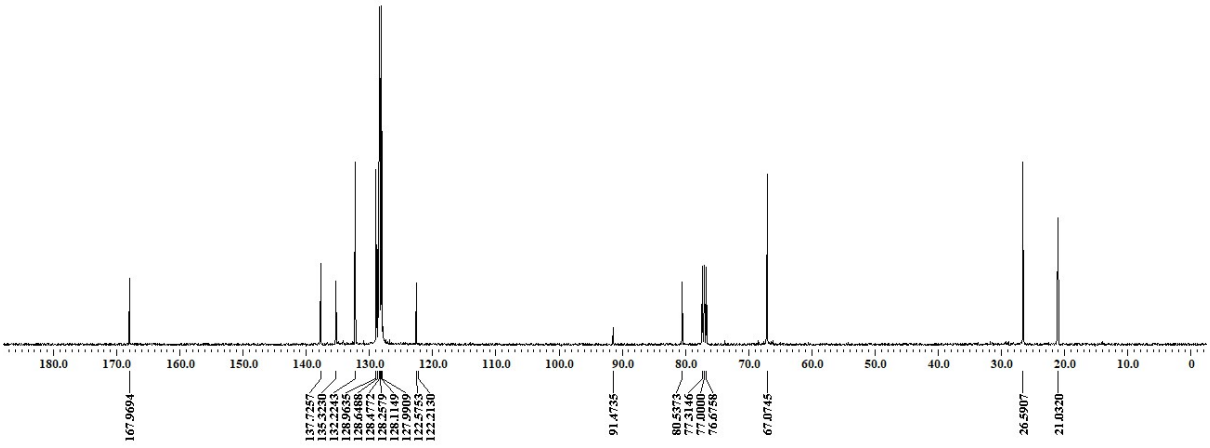
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

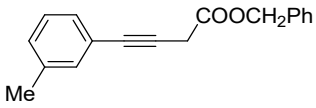


Benzyl 4-(*m*-tolyl)but-3-ynoate (3m)

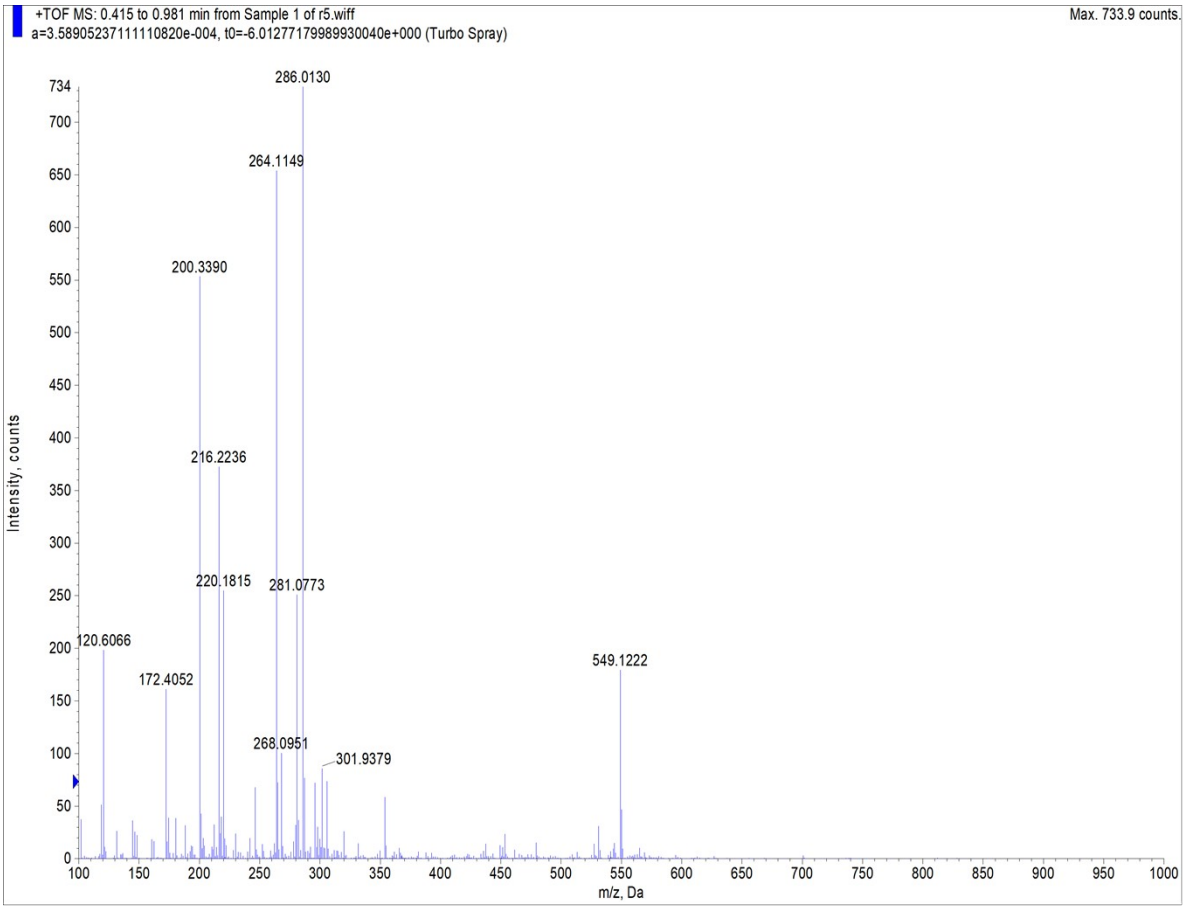




HRMS

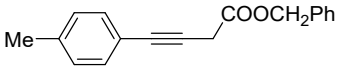


Benzyl 4-(*m*-tolyl)but-3-ynoate (3m)

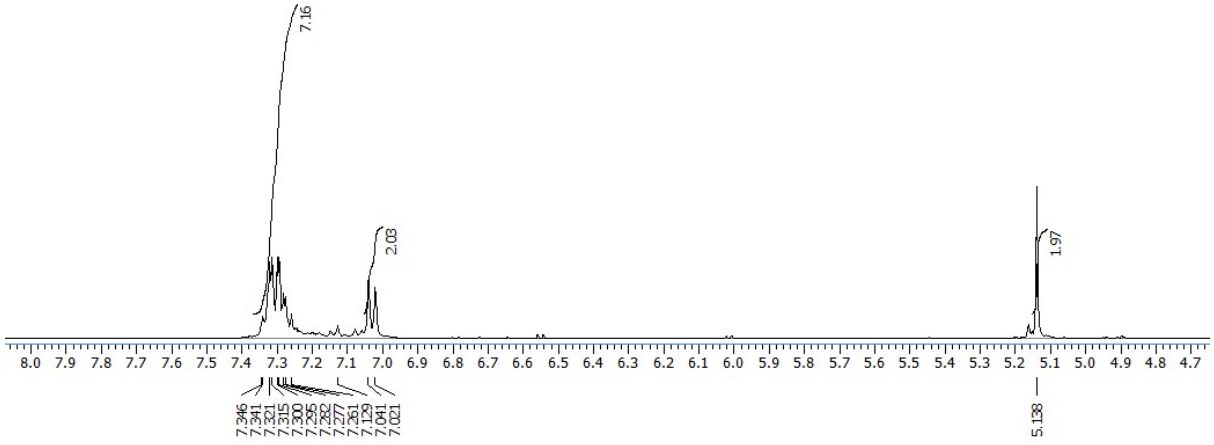


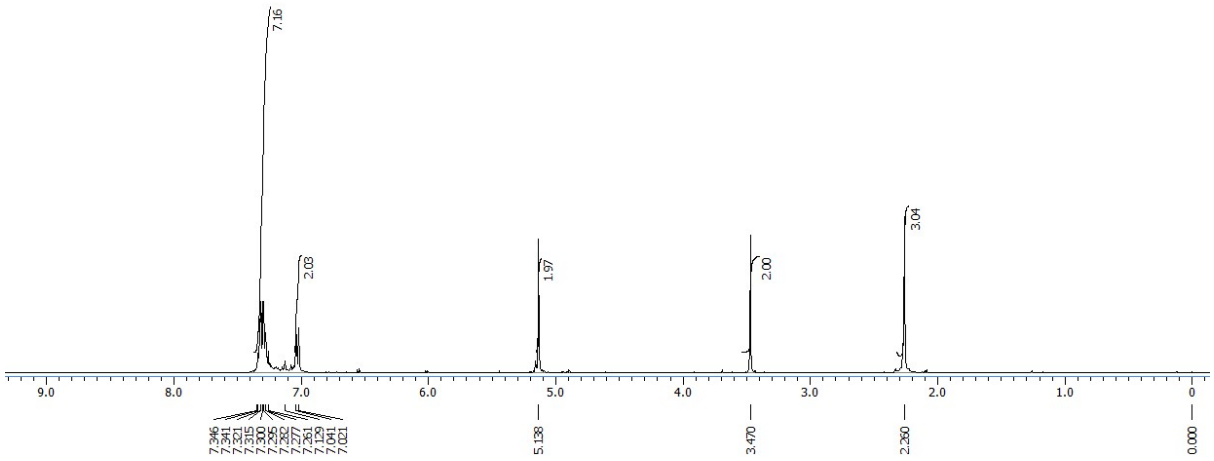
^{1}H NMR

(400 MHz, $CDCl_3$)



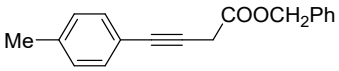
Benzyl 4-(*p*-tolyl)but-3-ynoate(3n)



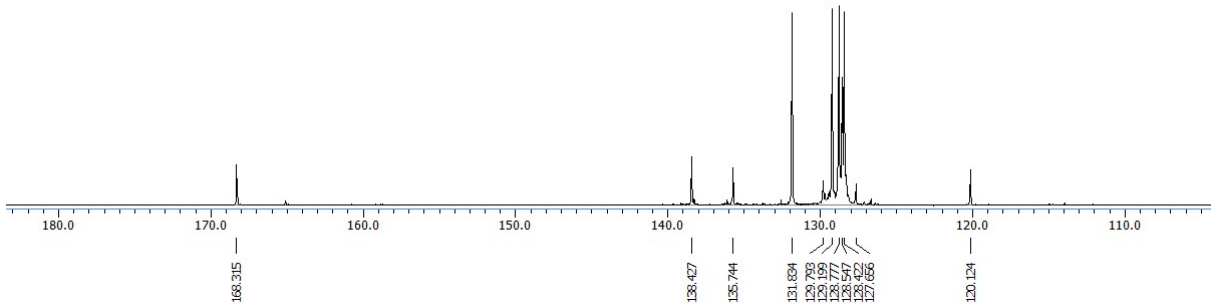


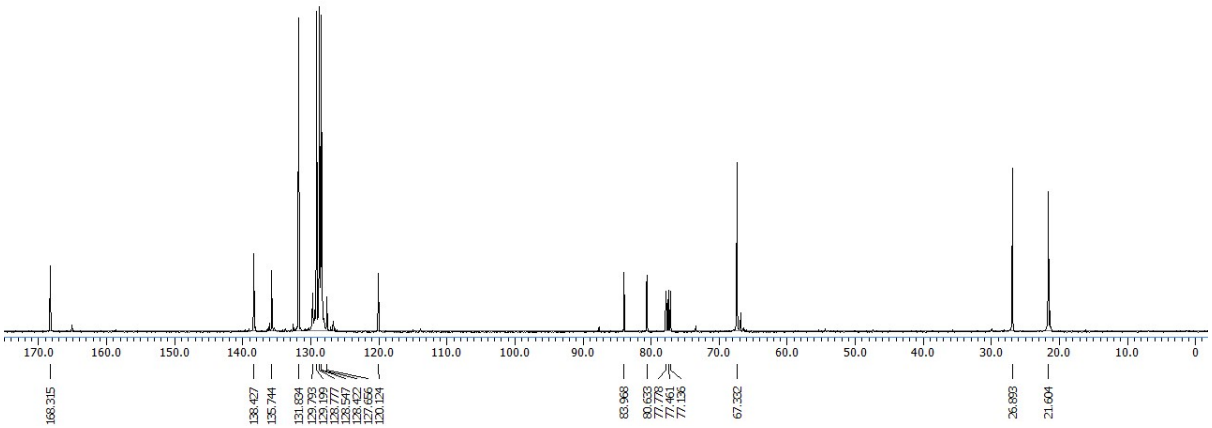
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

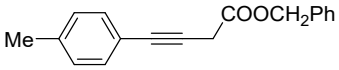


Benzyl 4-(*p*-tolyl)but-3-ynoate (3n)

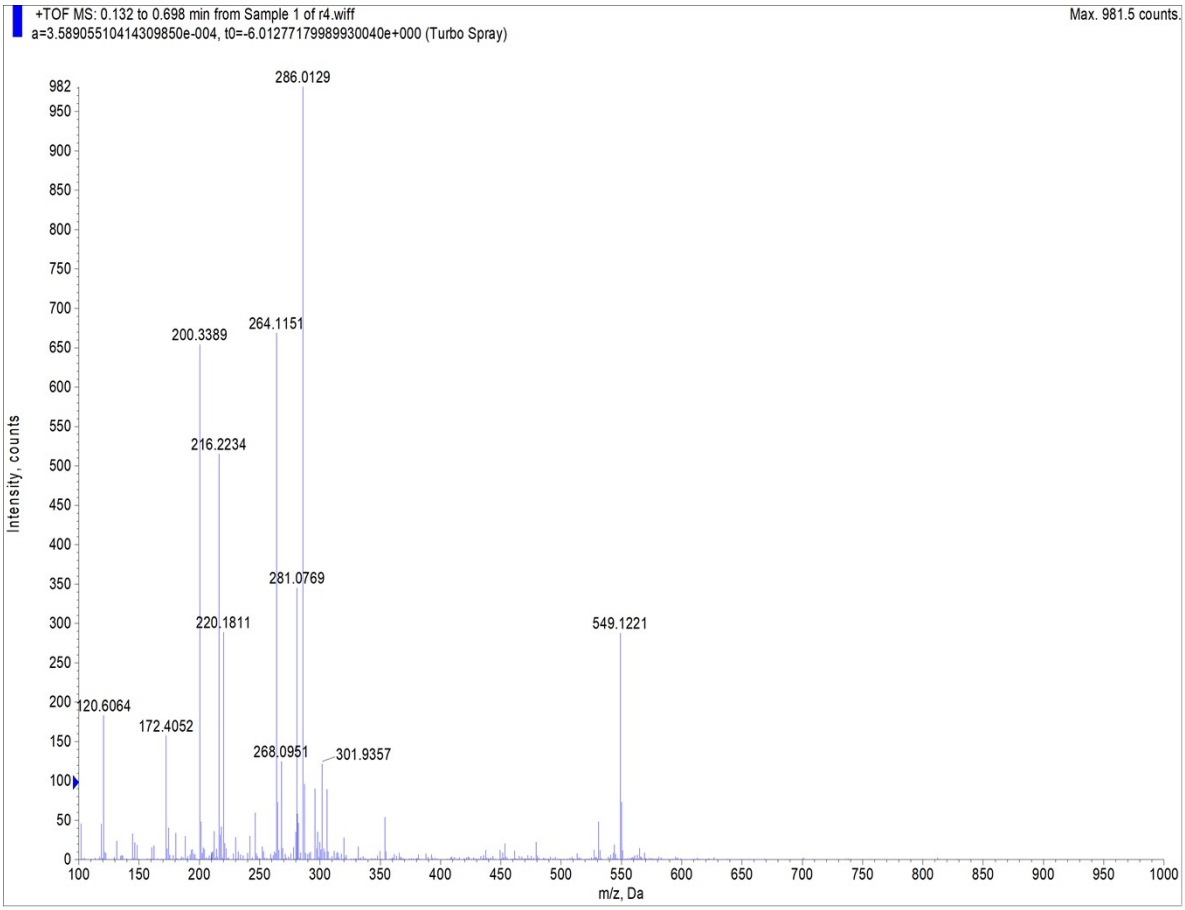




HRMS

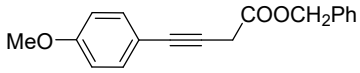


Benzyl 4-(*p*-tolyl)but-3-ynoate (3n)

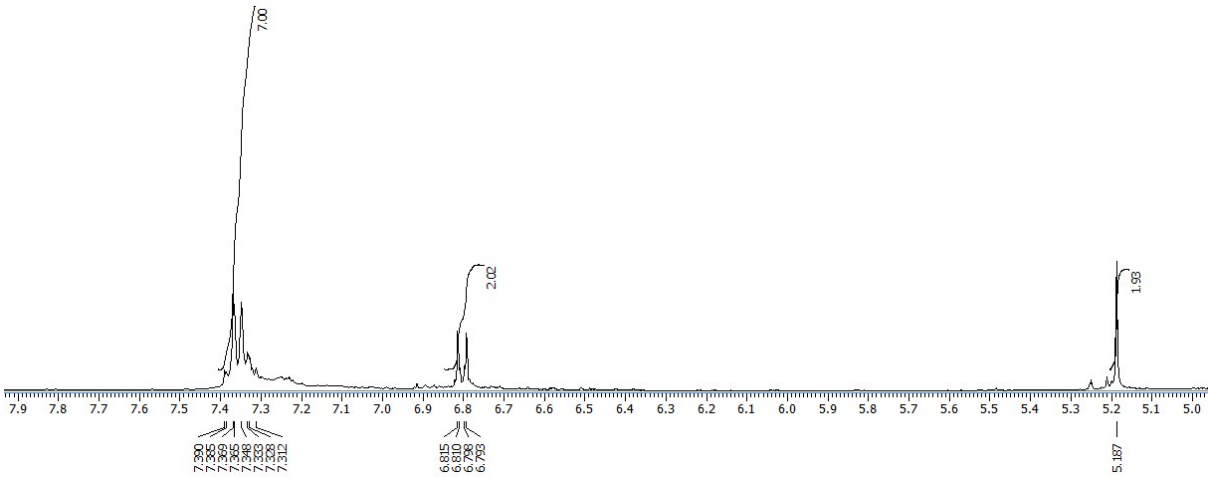


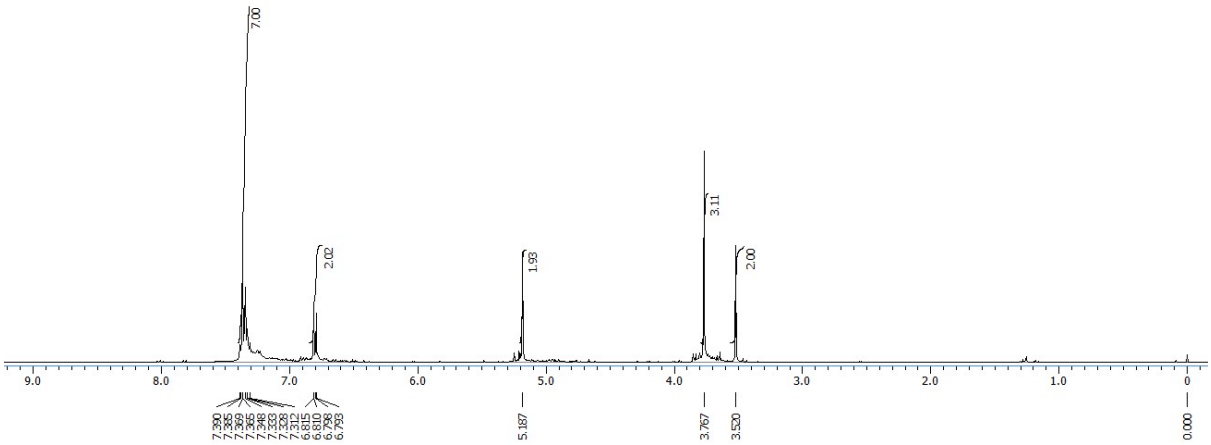
^{1}H NMR

(400 MHz, $CDCl_3$)



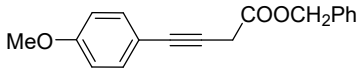
Benzyl 4-(4-methoxyphenyl)but-3-ynoate (3o)



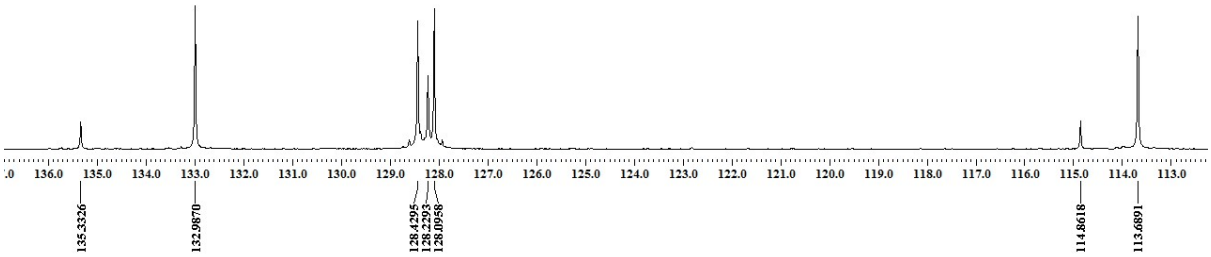


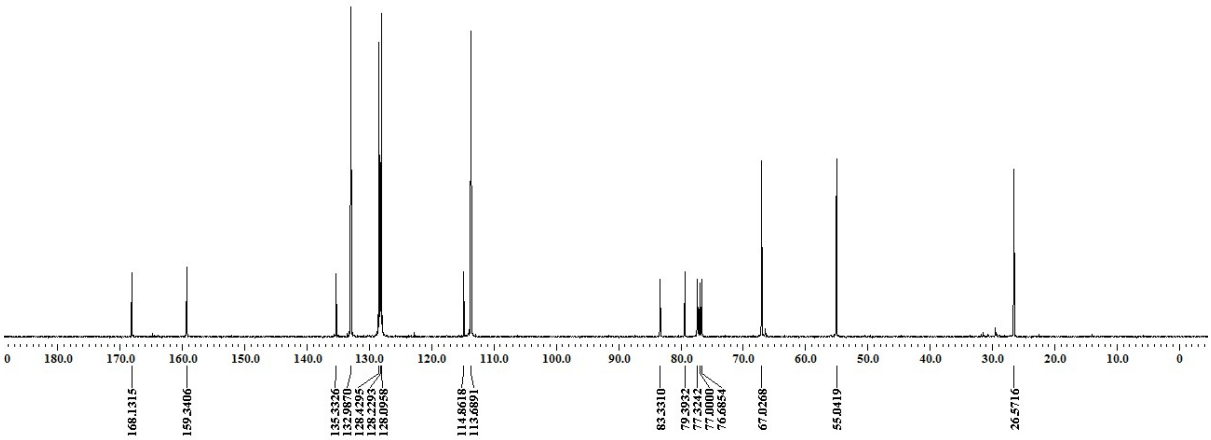
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

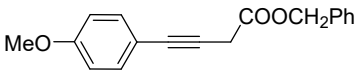


Benzyl 4-(4-methoxyphenyl)but-3-ynoate(3o)

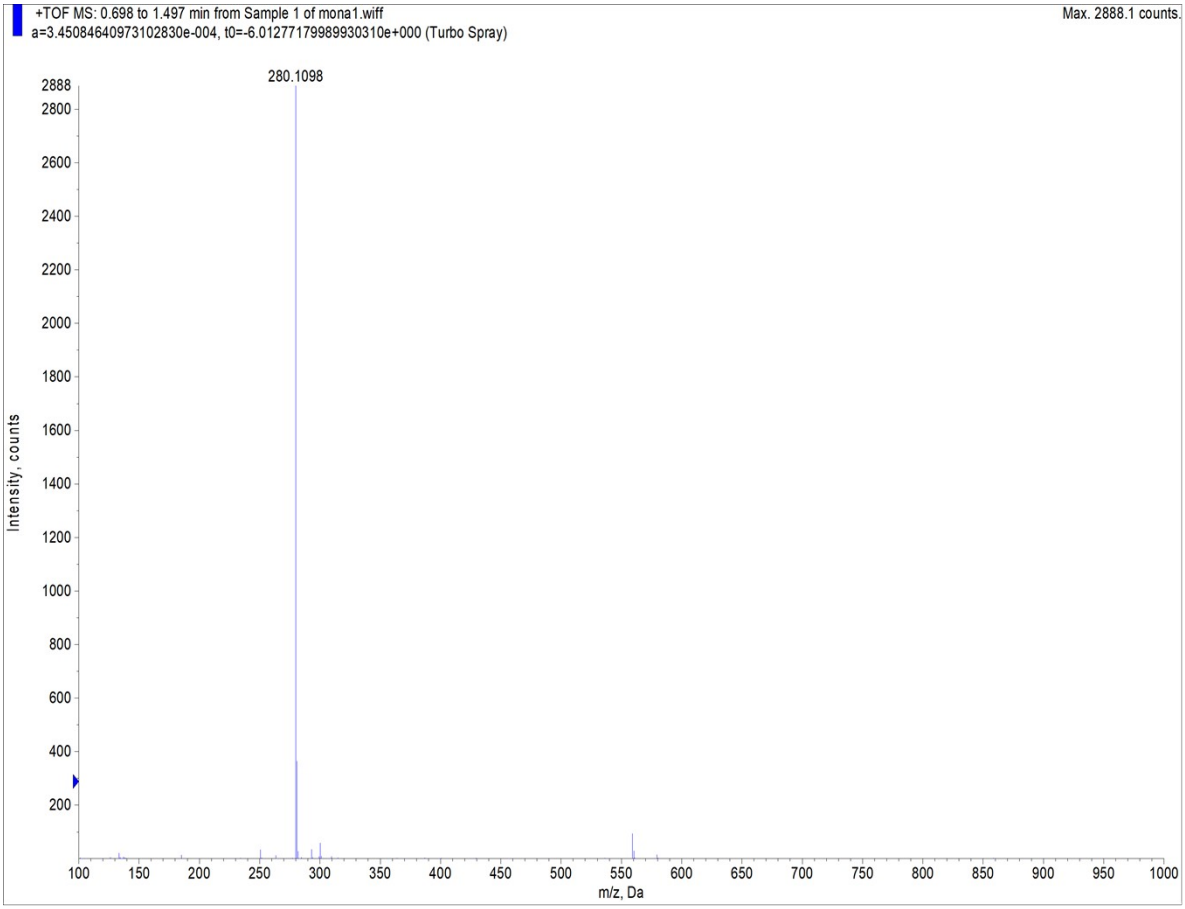




HRMS

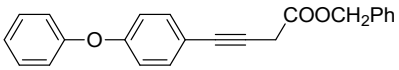


Benzyl 4-(4-methoxyphenyl)but-3-ynoate (3o)

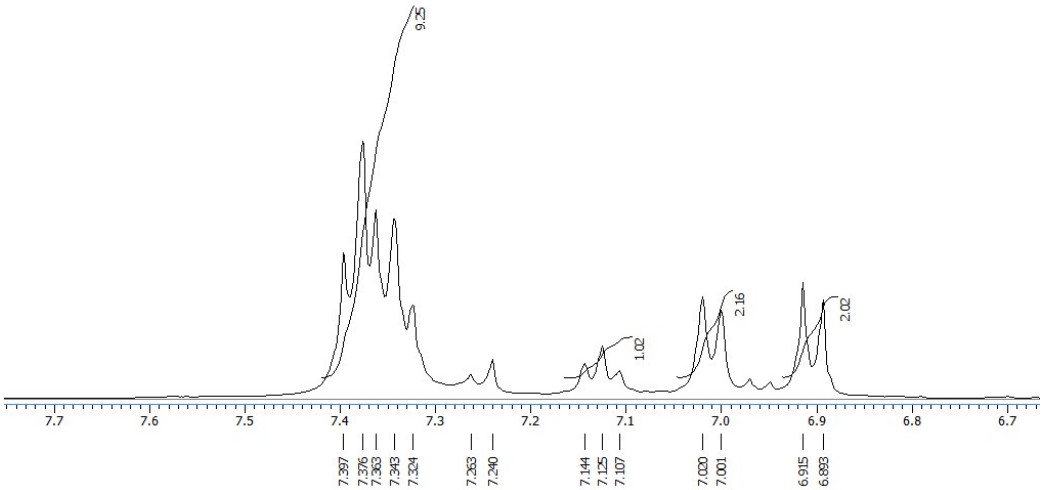


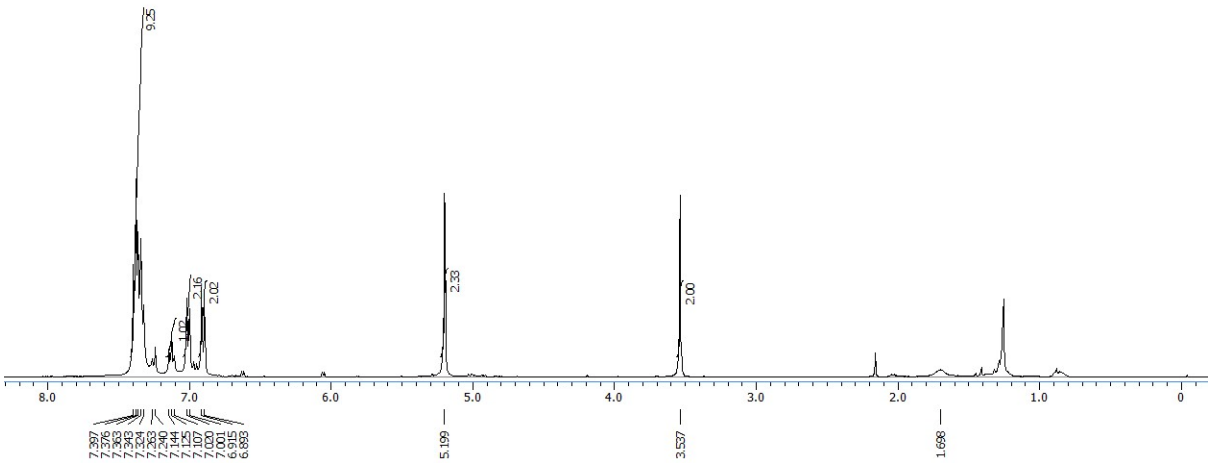
^{1}H NMR

(400 MHz, $CDCl_3$)



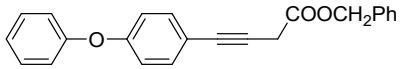
Benzyl 4-(4-phenoxyphenyl)but-3-ynoate (3p)



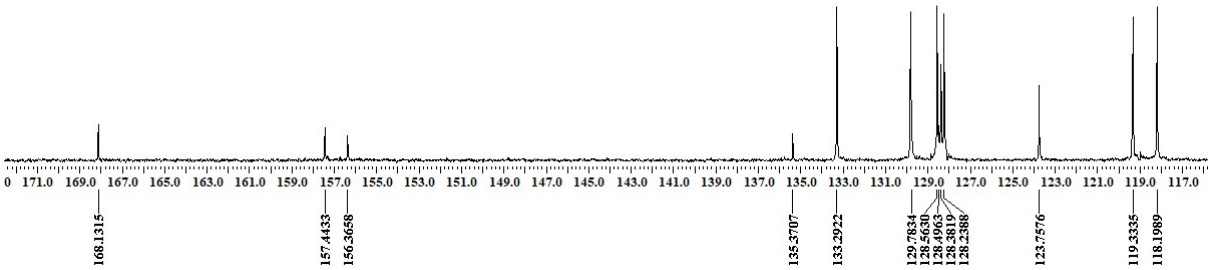


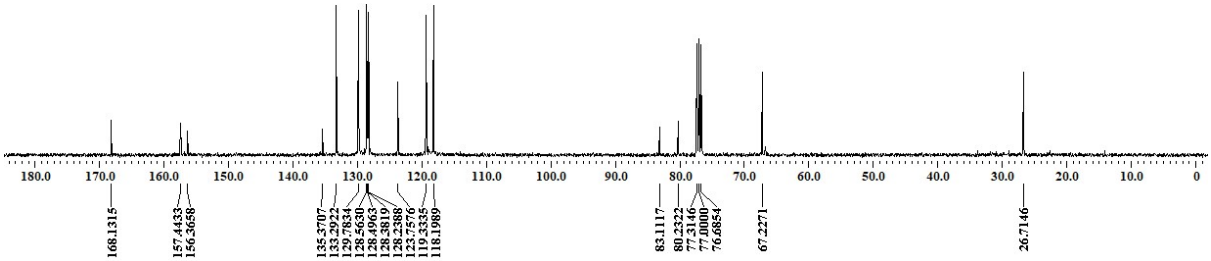
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

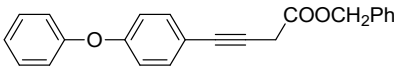


Benzyl 4-(4-phenoxyphenyl)but-3-ynoate (3p)

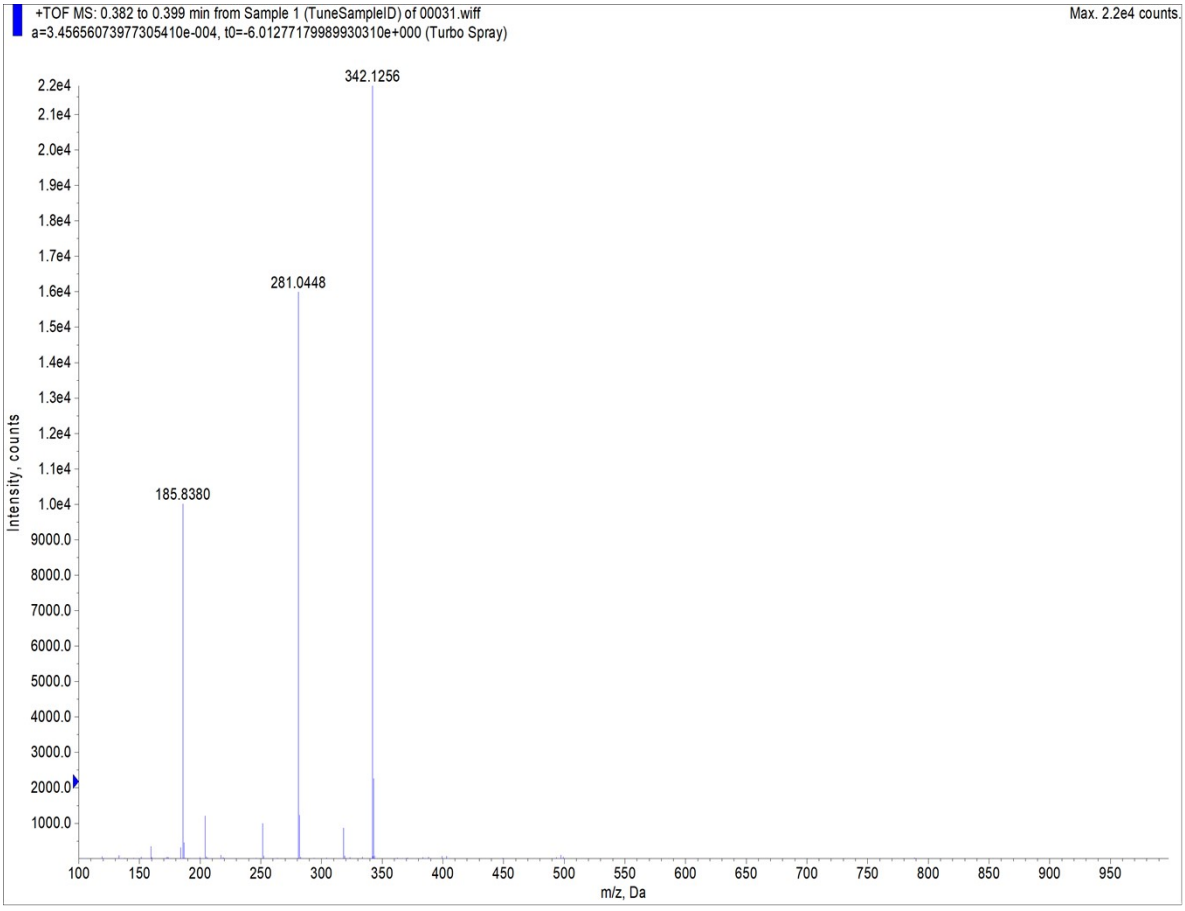




HRMS

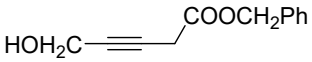


Benzyl 4-(4-phenoxyphenyl)but-3-ynoate (3p)

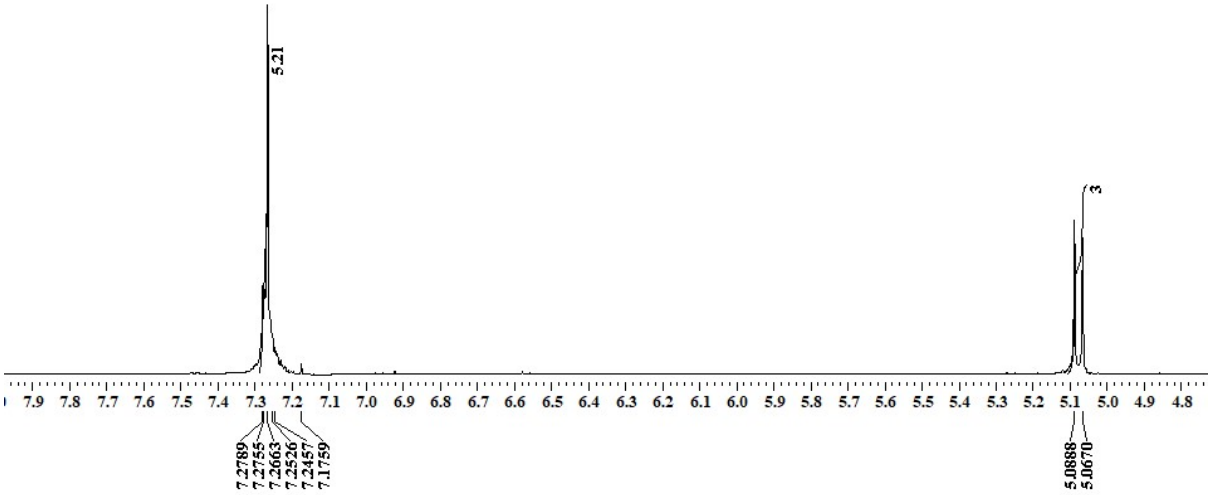


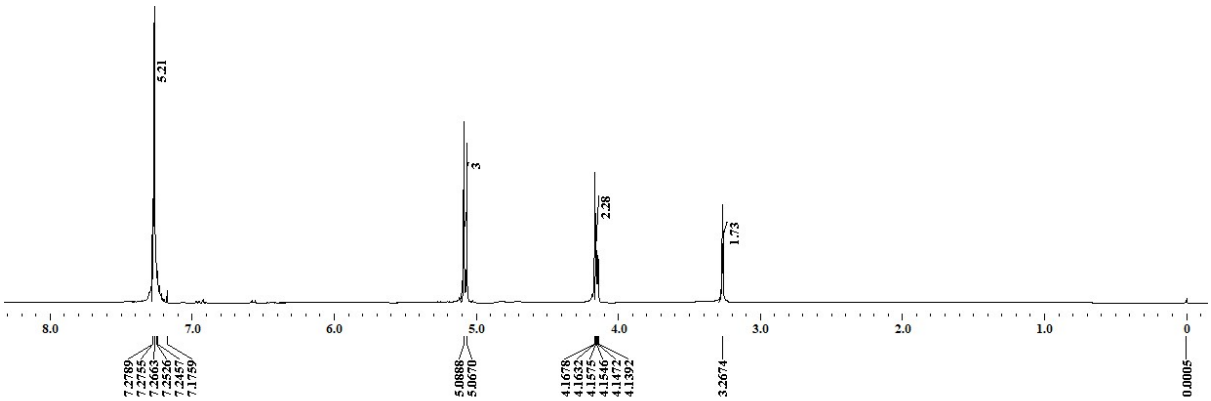
^{1}H NMR

(400 MHz, $CDCl_3$)



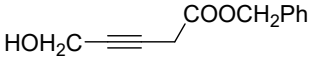
Benzyl 5-hydroxypent-3-ynoate (3q)



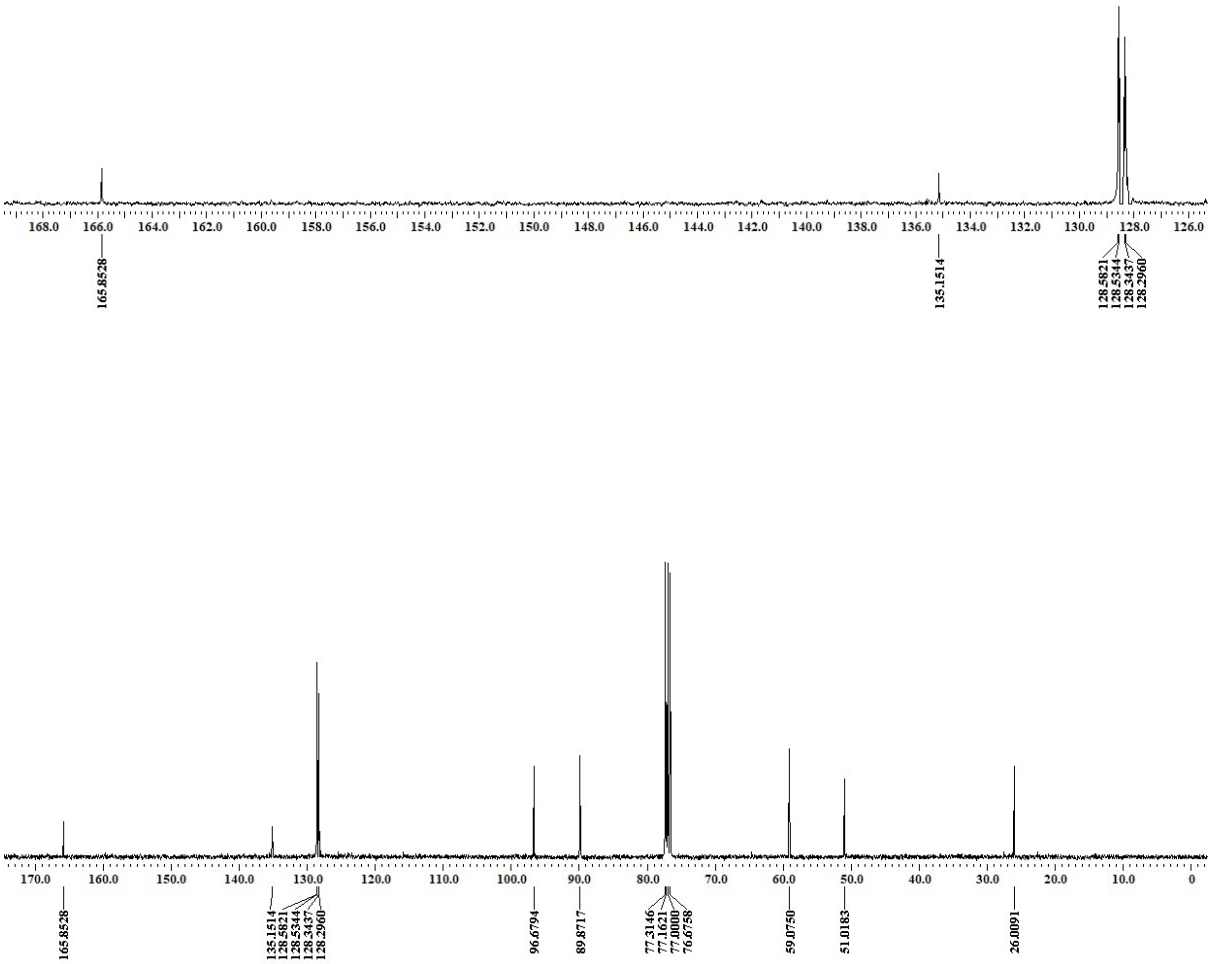


$^{13}C\{^{1}H\}$ NMR

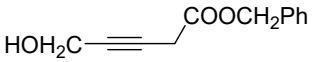
(100 MHz, $CDCl_3$)



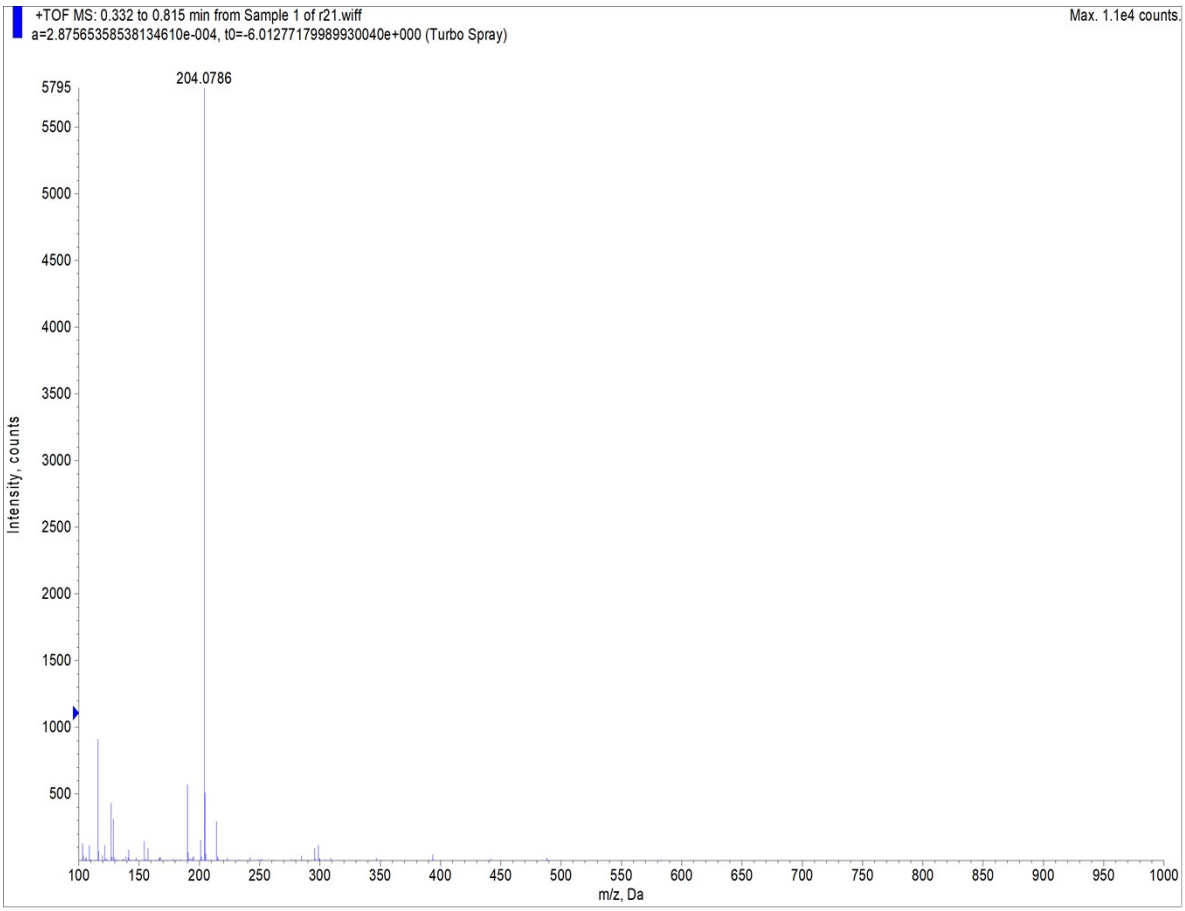
Benzyl 5-hydroxypent-3-ynoate (3q)



HRMS

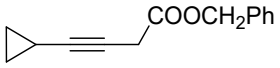


Benzyl 5-hydroxypent-3-ynoate (3q)

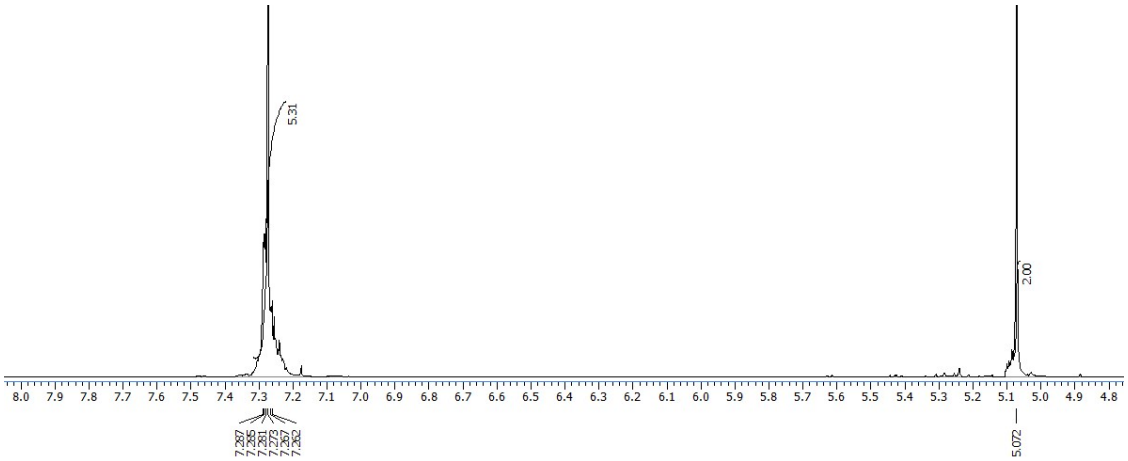


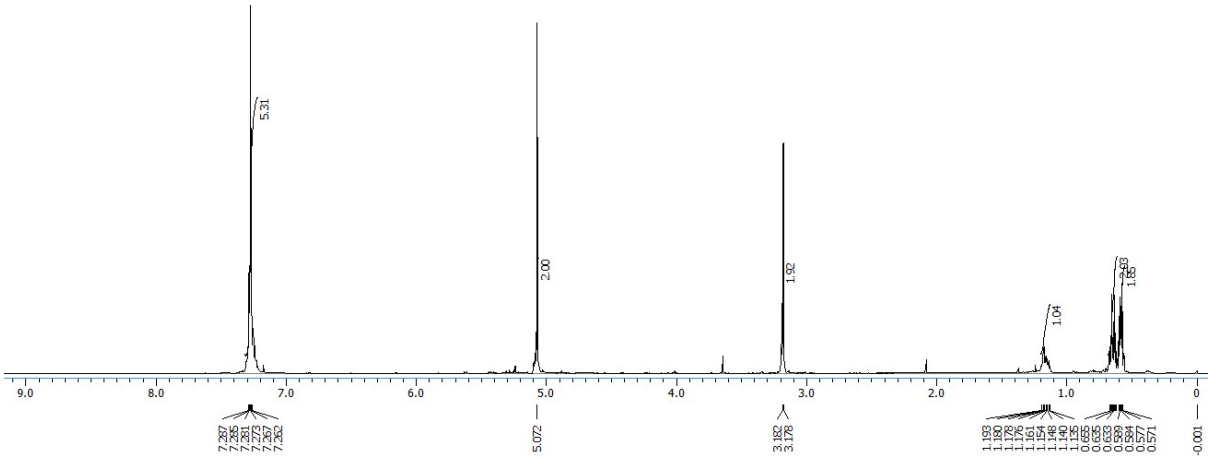
^{1}H NMR

(400 MHz, $CDCl_3$)



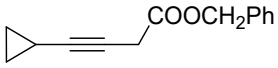
Benzyl 4-cyclopropylbut-3-ynoate(3r)



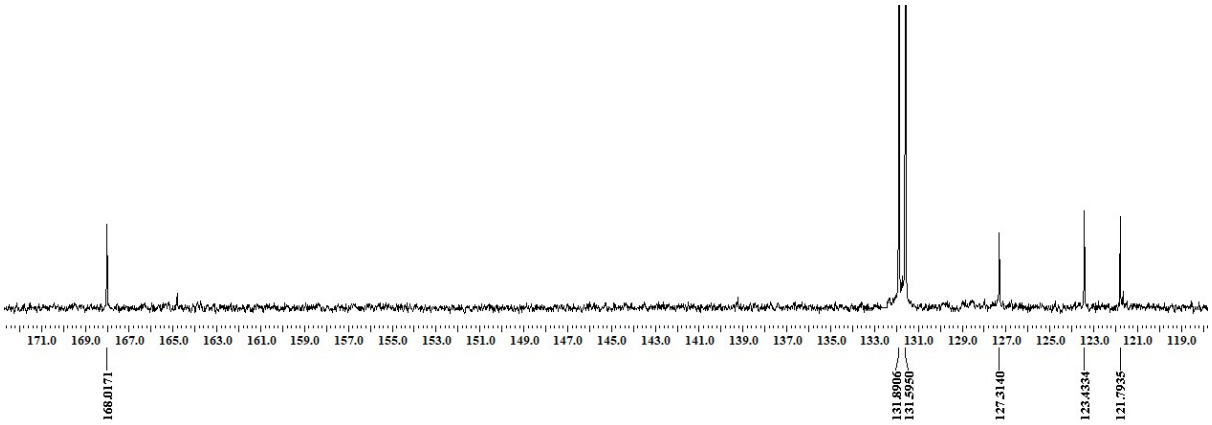


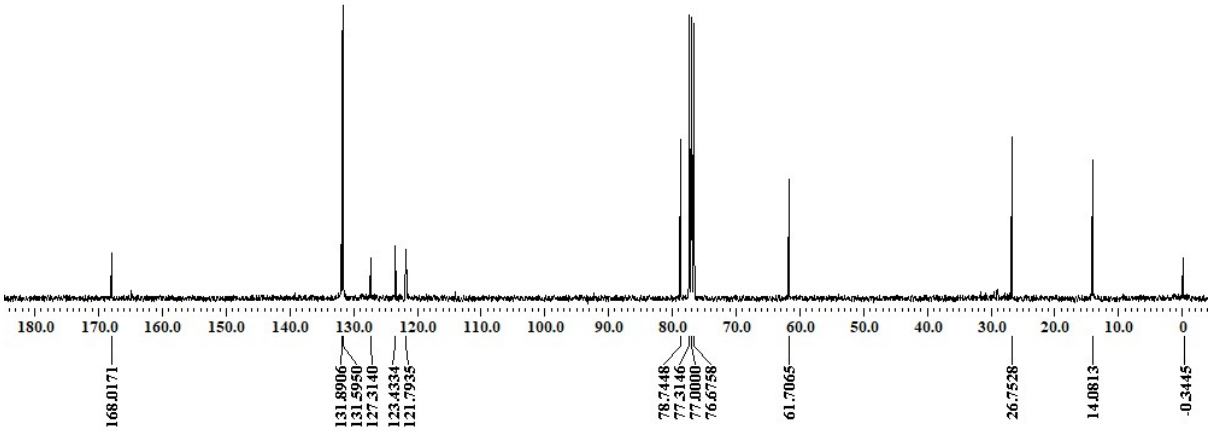
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

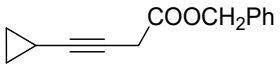


Benzyl 4-cyclopropylbut-3-ynoate(3r)

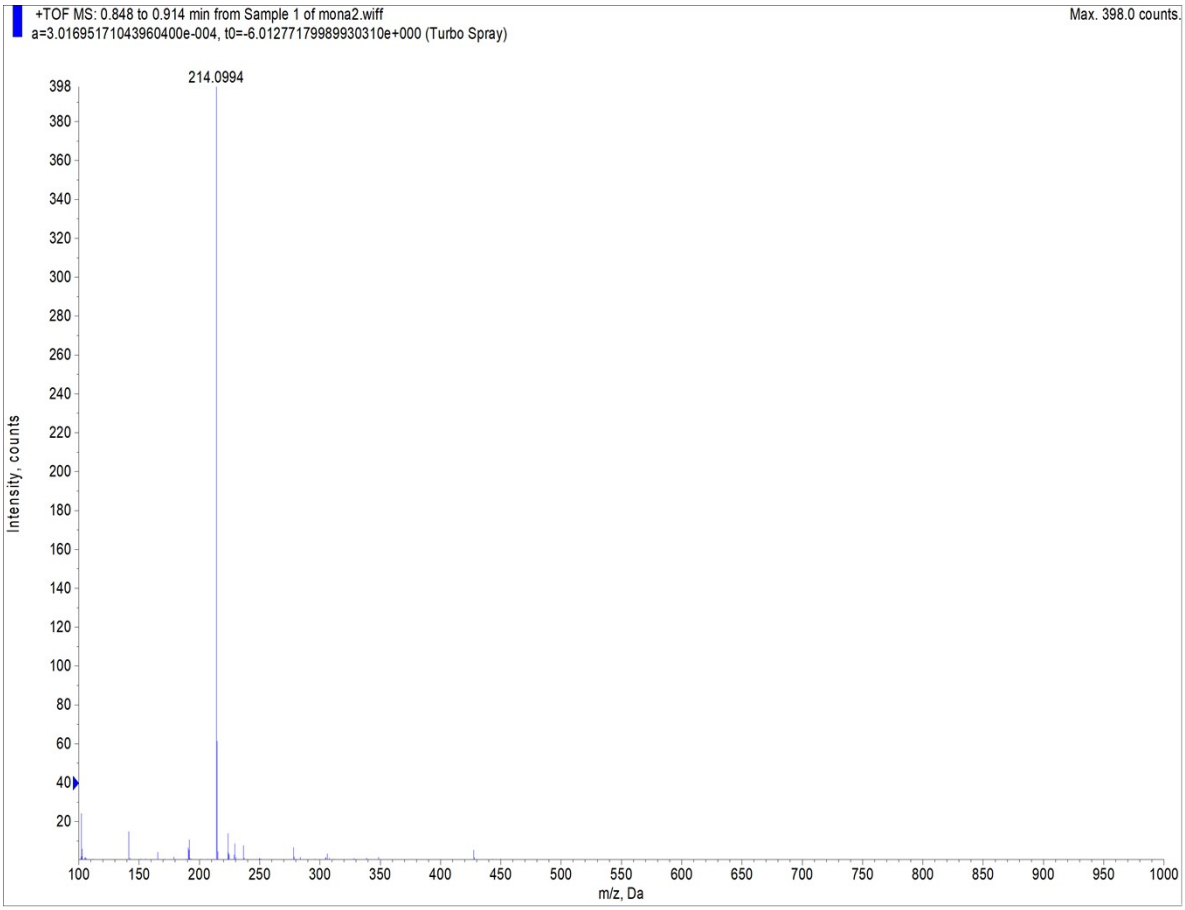




HRMS

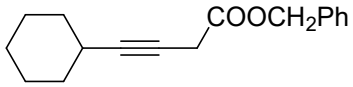


Benzyl 4-cyclopropylbut-3-ynoate(3r)

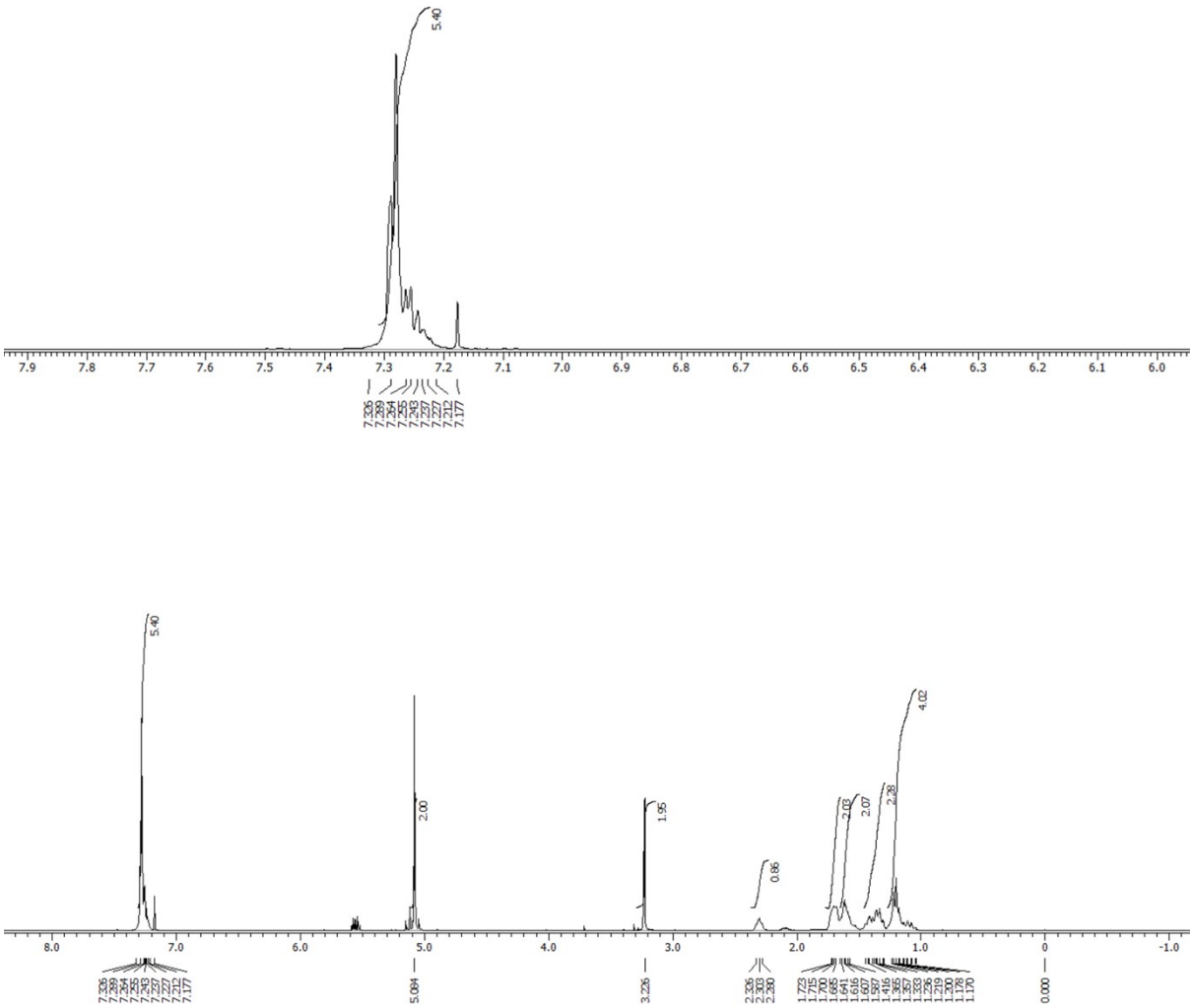


^{1}H NMR

(400 MHz, $CDCl_3$)

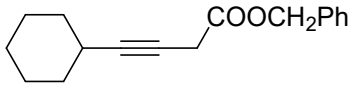


benzyl 4-cyclohexylbut-3-ynoate (3s)

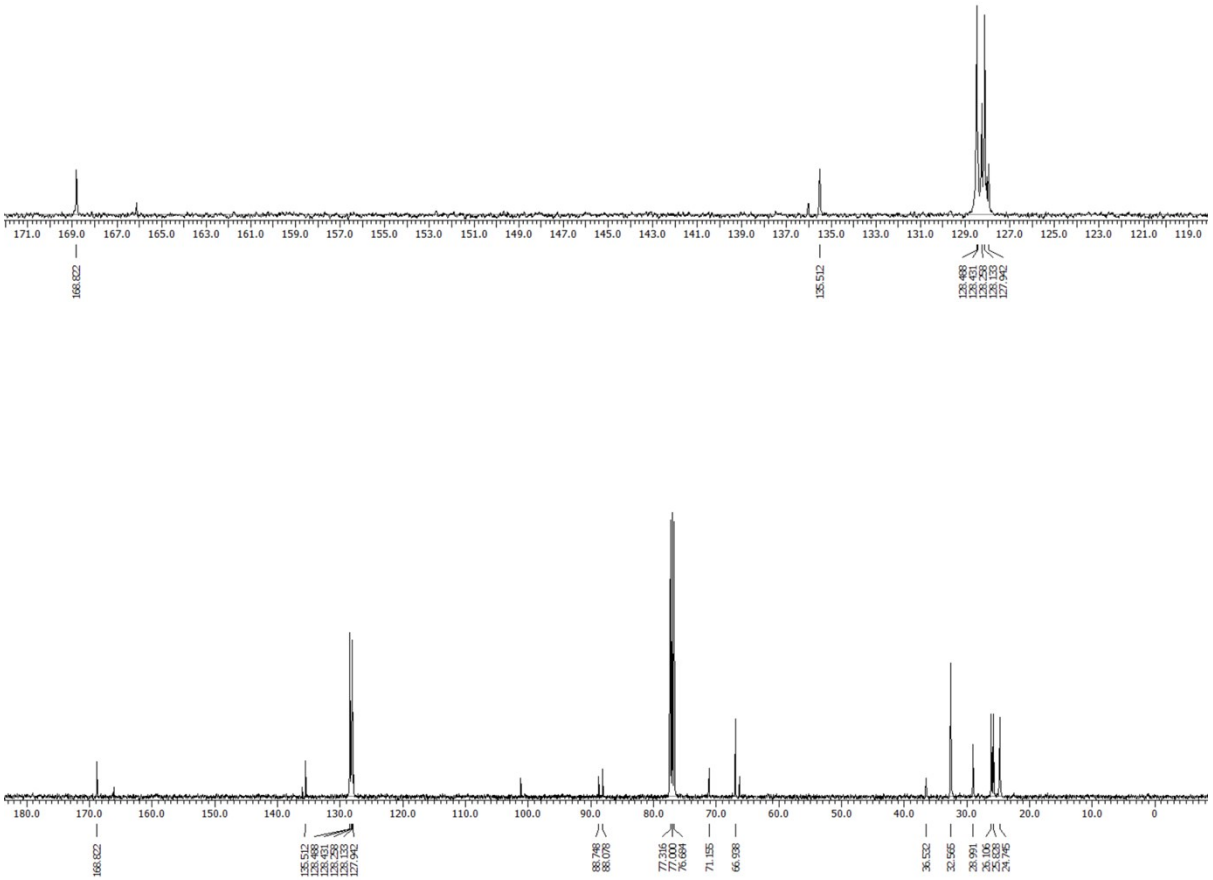


$^{13}C\{^{1}H\}$ NMR

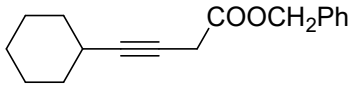
(400 MHz, $CDCl_3$)



benzyl 4-cyclohexylbut-3-ynoate (3s)



HRMS



benzyl 4-cyclohexylbut-3-ynoate (3s)

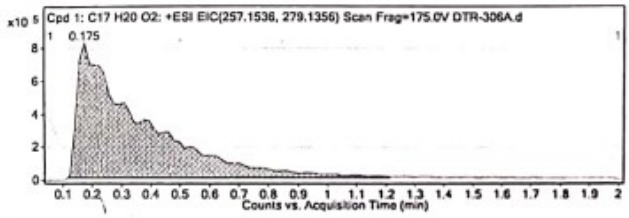
Qualitative Compound Report

Data File	DTR-306A.d	Sample Name	DTR-306A
Sample Type	Sample	Position	P1-A3
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	21-01-2023 12:58:23
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

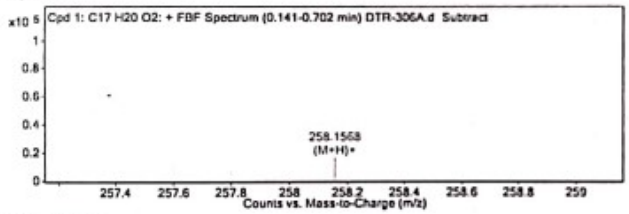
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C17 H20 O2	0.175	256.1463	93435	C17 H20 O2	256.1463	-0.14	C17 H20 O2	C17 H20 O2

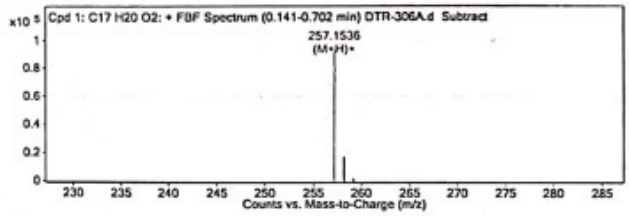
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C17 H20 O2	257.1536	0.175	Find By Formula	256.1463



MS Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

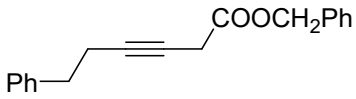
m/z	z	Abund	Formula	Ion
257.1536	1	93435.15	C17H21O2	(M+H)+
258.1568	1	16450.53	C17H21O2	(M+H)+
259.1597	1	2027.35	C17H21O2	(M+H)+

— End Of Report —

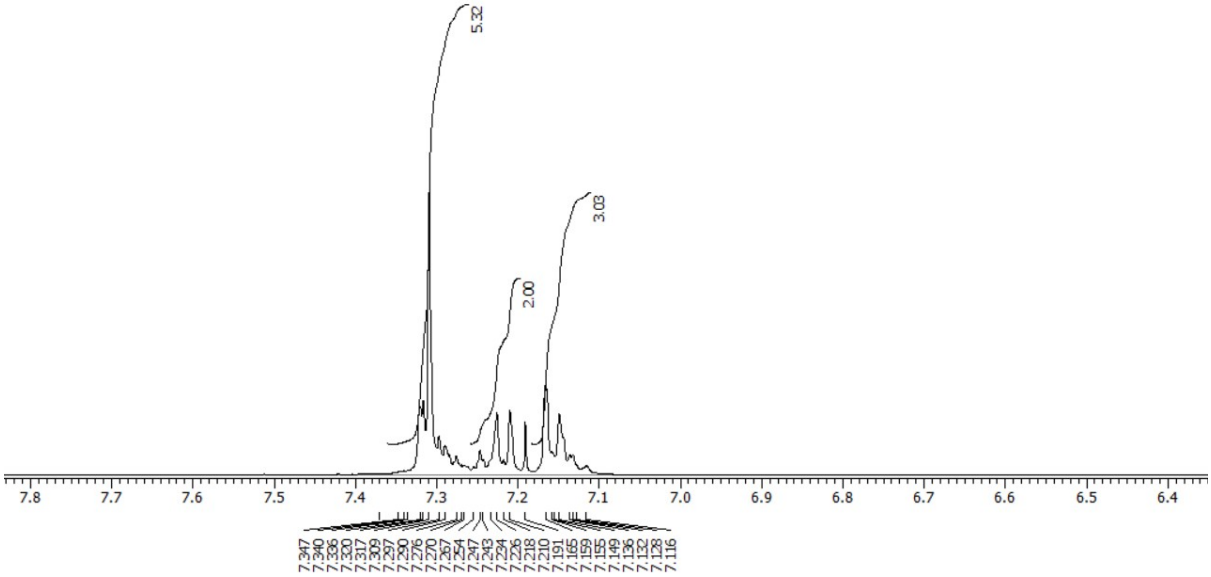
Agilent Technologies Page 1 of 1 Printed at: 13:38 on:21-01-2023

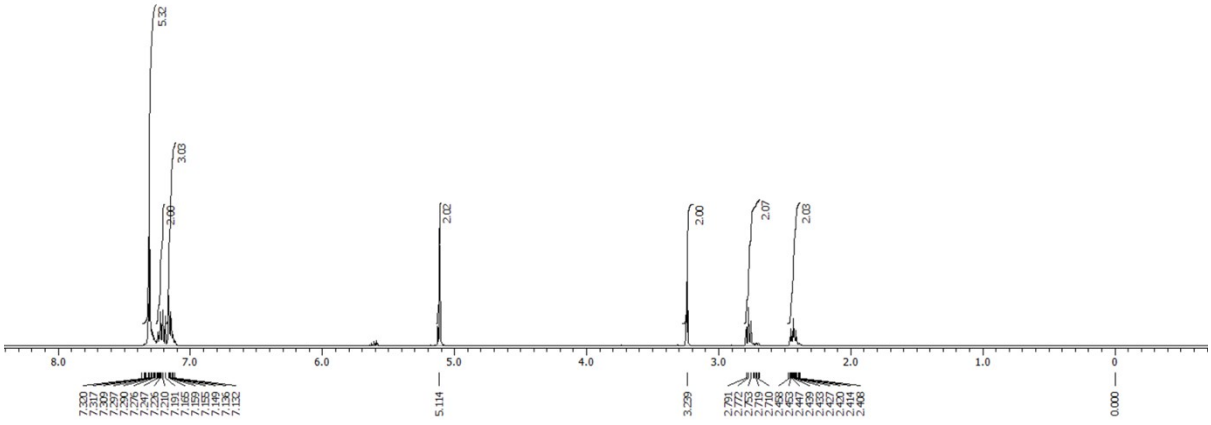
^{1}H NMR

(400 MHz, $CDCl_3$)



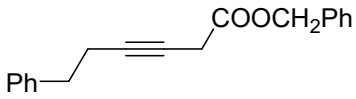
benzyl 6-phenylhex-3-ynoate (3t)



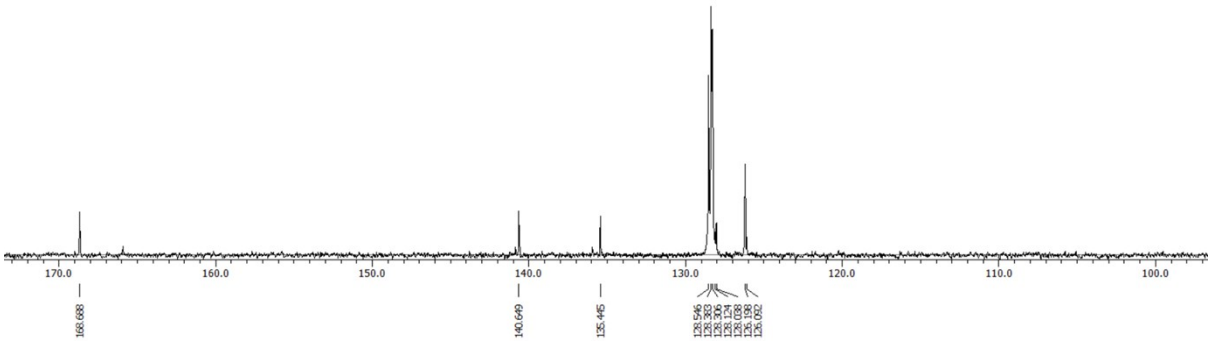


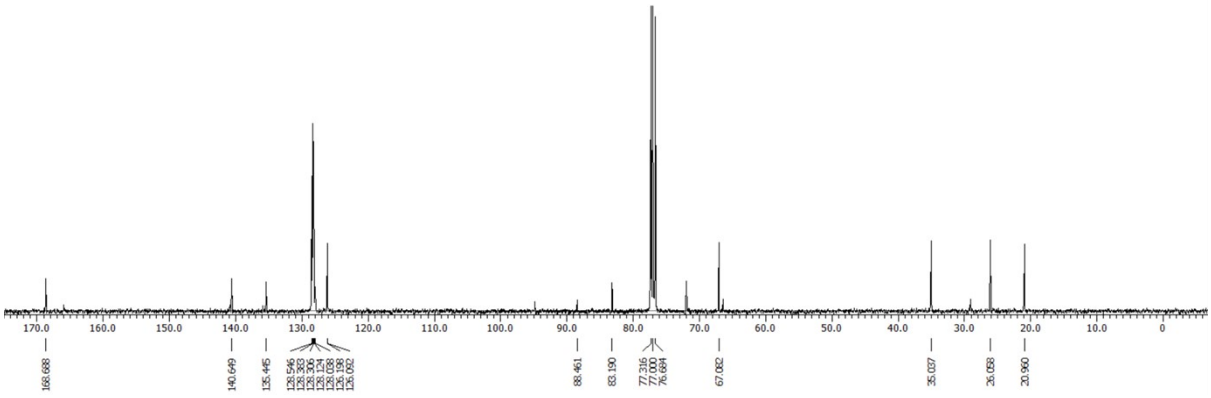
$^{13}C\{^{1}H\}$ NMR

(101 MHz, $CDCl_3$)

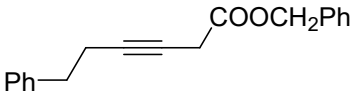


benzyl 6-phenylhex-3-ynoate (3t)





HRMS



benzyl 6-phenylhex-3-ynoate (3t)

Qualitative Compound Report

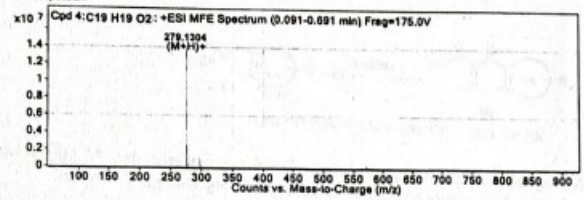
Data File	DTR-312	Sample Name	DTR-312
Sample Type	Sample	Position	P1-B8
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	28-01-2023 13:20:15
IRM Calibration Status	[redacted]	DA Method	Default.m
Comment			
Sample Group	Info.	3	
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

Compound Table

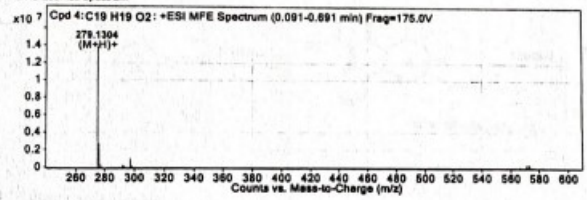
Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 4: C19 H18 O2	0.164	278.1307	C19 H18 O2	C19 H18 O2	-2.32	C19 H18 O2

Compound Label	m/z	RT	Algorithm	Mass
Cpd 4: C19 H18 O2	279.1304	0.164	Find by Molecular Feature	278.1307

MFE MS Spectrum



MFE MS Zoomed Spectrum

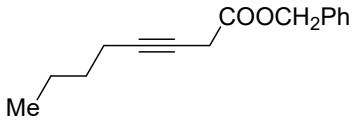


MS Spectrum Peak List

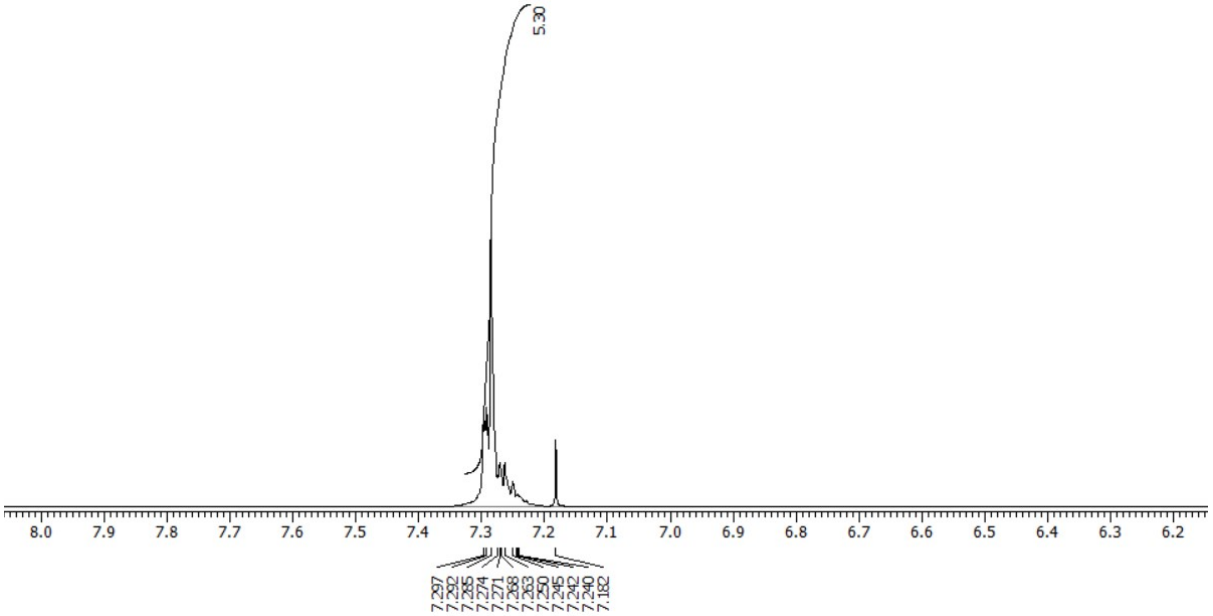
m/z	z	Abund	Formula	Ion
275.0709	1	13911574	C19 H19 O2	(M+H)+
276.0745	1	2694706.82	C19 H19 O2	(M+H)+
277.0774	1	345589.17	C19 H19 O2	(M+H)+
279.1304	1	34210.15	C19 H19 O2	(M+H)+

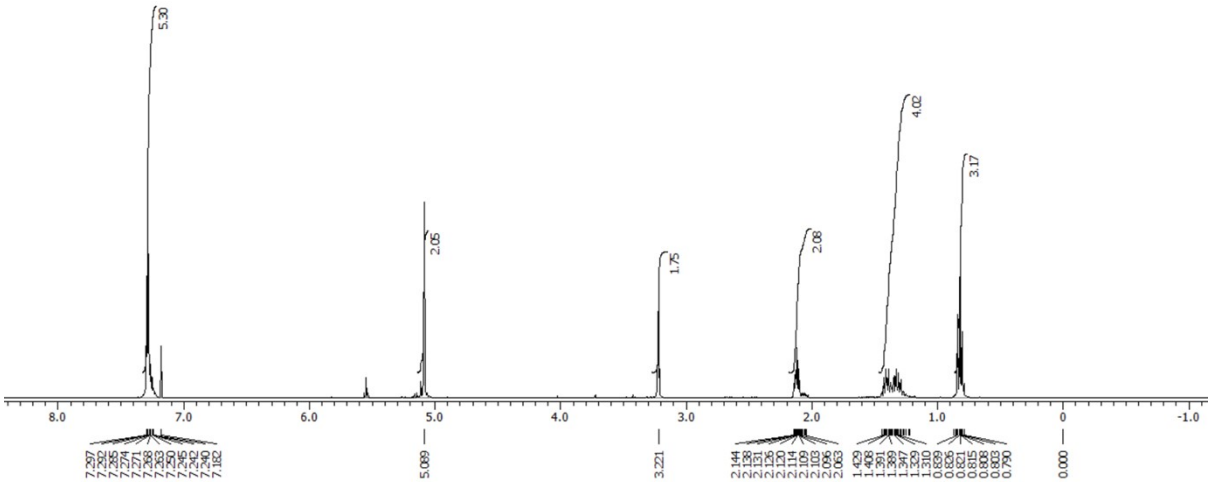
^{1}H NMR

(400 MHz, $CDCl_3$)



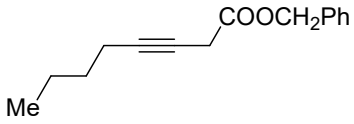
benzyl oct-3-ynoate (3u)



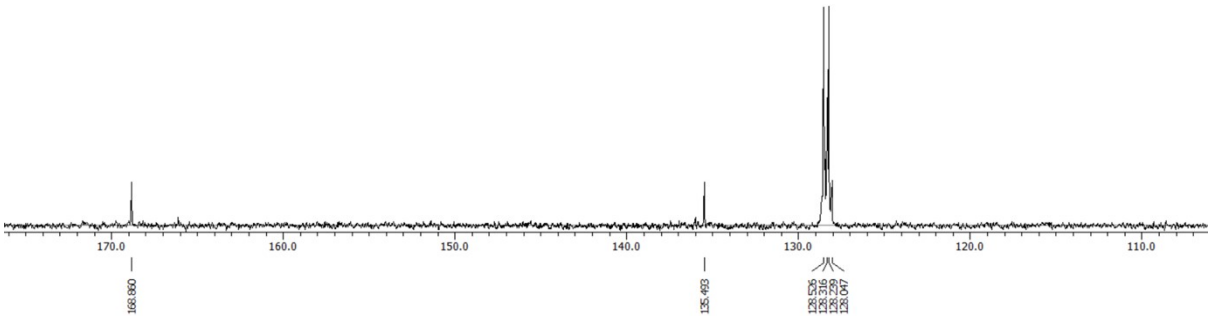


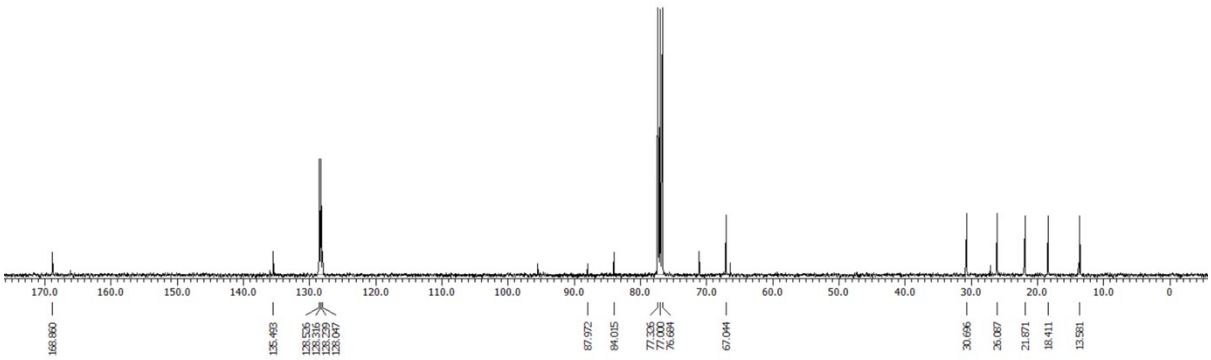
$^{13}C\{^{1}H\}$ NMR

(101 MHz, $CDCl_3$)

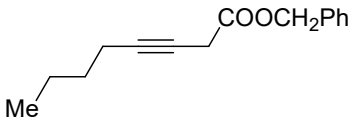


benzyl oct-3-ynoate (3u)





HRMS



benzyl oct-3-ynoate (3u)

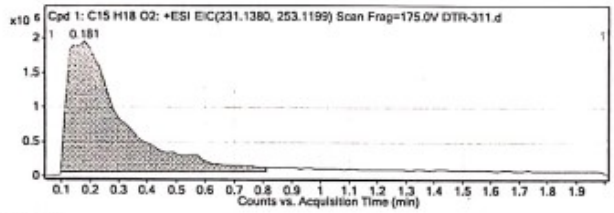
Qualitative Compound Report

Data File	DTR-311.d	Sample Name	DTR-311
Sample Type	Sample	Position	P1-A2
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	23-01-2023 12:48:14
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

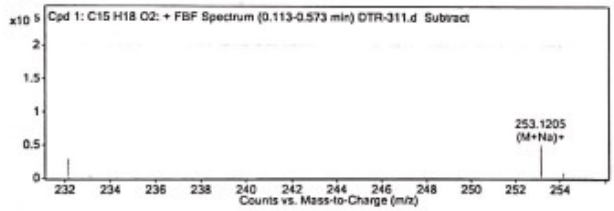
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C15 H18 O2	0.181	230.1311	193943	C15 H18 O2	230.1307	1.96	C15 H18 O2	C15 H18 O2

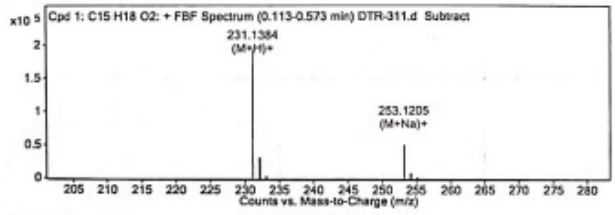
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C15 H18 O2	231.1384	0.181	Find By Formula	230.1311



MS Spectrum



MS Zoomed Spectrum



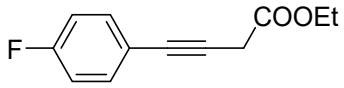
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
231.1384	1	193942.7	C15H19O2	(M+H)+
232.1416	1	29411.83	C15H19O2	(M+H)+
233.1443	1	3334.01	C15H19O2	(M+H)+
234.1433	1	325.84	C15H19O2	(M+H)+
253.1205	1	51234.72	C15H18NaO2	(M+Na)+
254.1238	1	8175.76	C15H18NaO2	(M+Na)+
255.1271	1	1119.34	C15H18NaO2	(M+Na)+
256.1227	1	73.4	C15H18NaO2	(M+Na)+

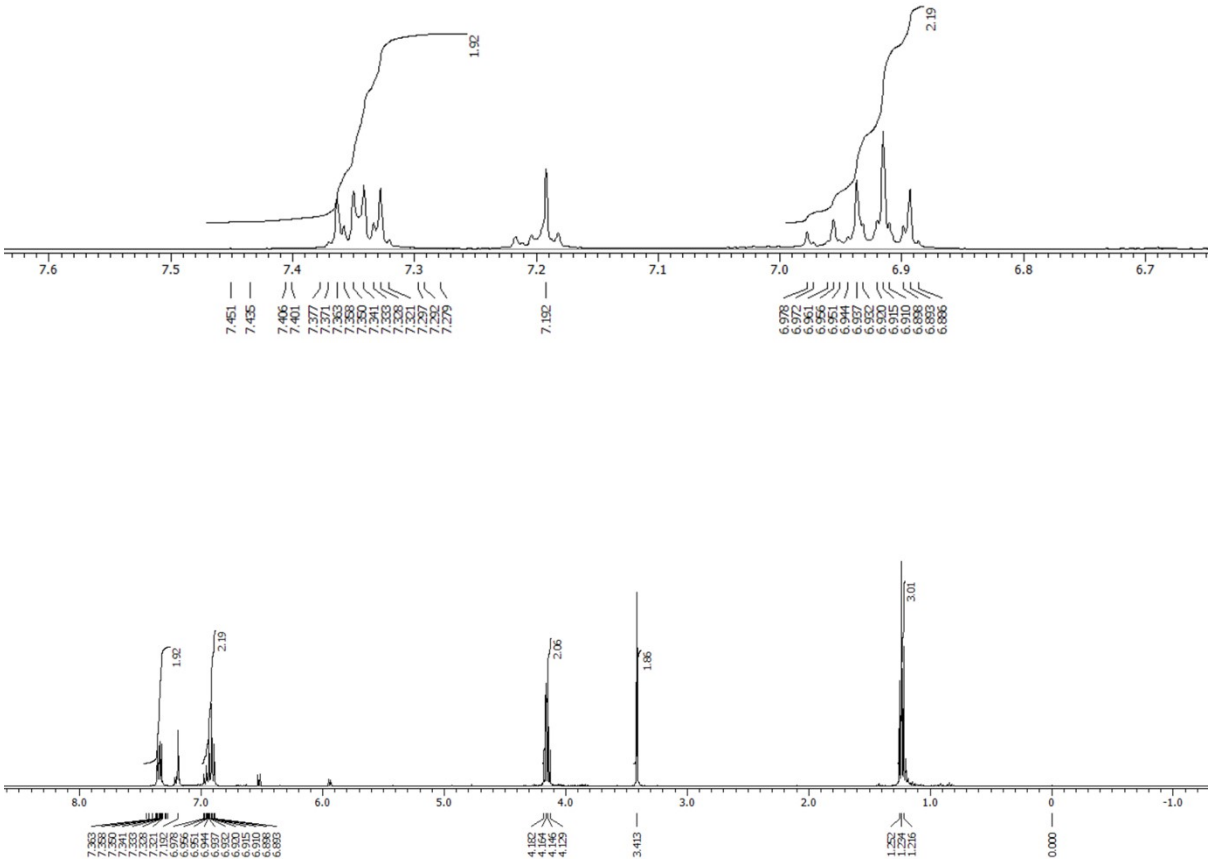
--- End Of Report ---

^{1}H NMR

(400 MHz, $CDCl_3$)

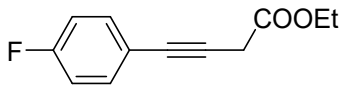


ethyl 4-(4-fluorophenyl)but-3-ynoate (3v)

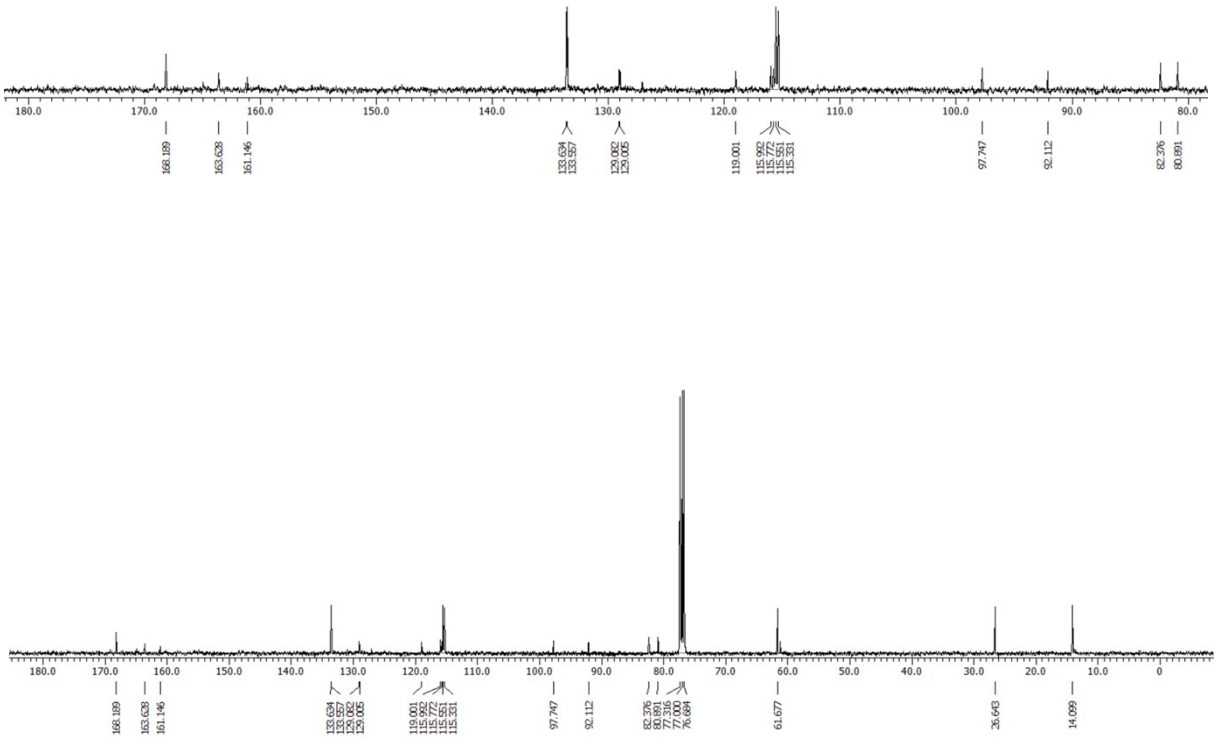


$^{13}C\{^{1}H\}$ NMR

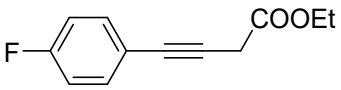
(101 MHz, $CDCl_3$)



ethyl 4-(4-fluorophenyl)but-3-ynoate (3v)



HRMS



ethyl 4-(4-fluorophenyl)but-3-ynoate (3v)

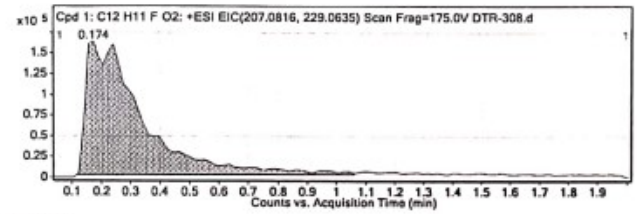
Qualitative Compound Report

Data File	DTR-308.d	Sample Name	DTR-308
Sample Type	Sample	Position	P1-A4
Instrument Name	Instrument 1	User Name	
Acq Method	MS Scan.m	Acquired Time	21-01-2023 12:59:07
IRM Calibration Status	Success	DA Method	Default.m
Comment			
Sample Group		Info.	3
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)		

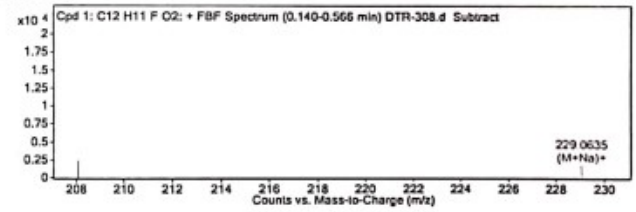
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C12 H11 F O2	0.174	206.0739	18000	C12 H11 F O2	206.0743	-1.97	C12 H11 F O2	C12 H11 F O2

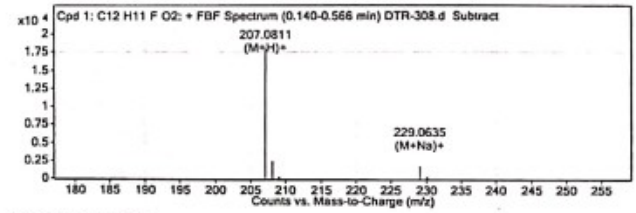
Compound Label	m/z	RT	Algorithm	Mass
Cpd 1: C12 H11 F O2	207.0811	0.174	Find By Formula	206.0739



MS Spectrum



MS Zoomed Spectrum



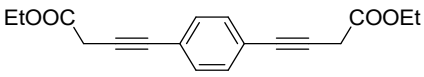
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
207.0811	1	17999.52	C12H12FO2	(M+H)+
208.0849	1	2395.68	C12H12FO2	(M+H)+
209.0792	1	88.1	C12H12FO2	(M+H)+
229.0635	1	1757.62	C12H11FNaO2	(M+Na)+
230.0669	1	213.49	C12H11FNaO2	(M+Na)+
231.0732	1	43.88	C12H11FNaO2	(M+Na)+

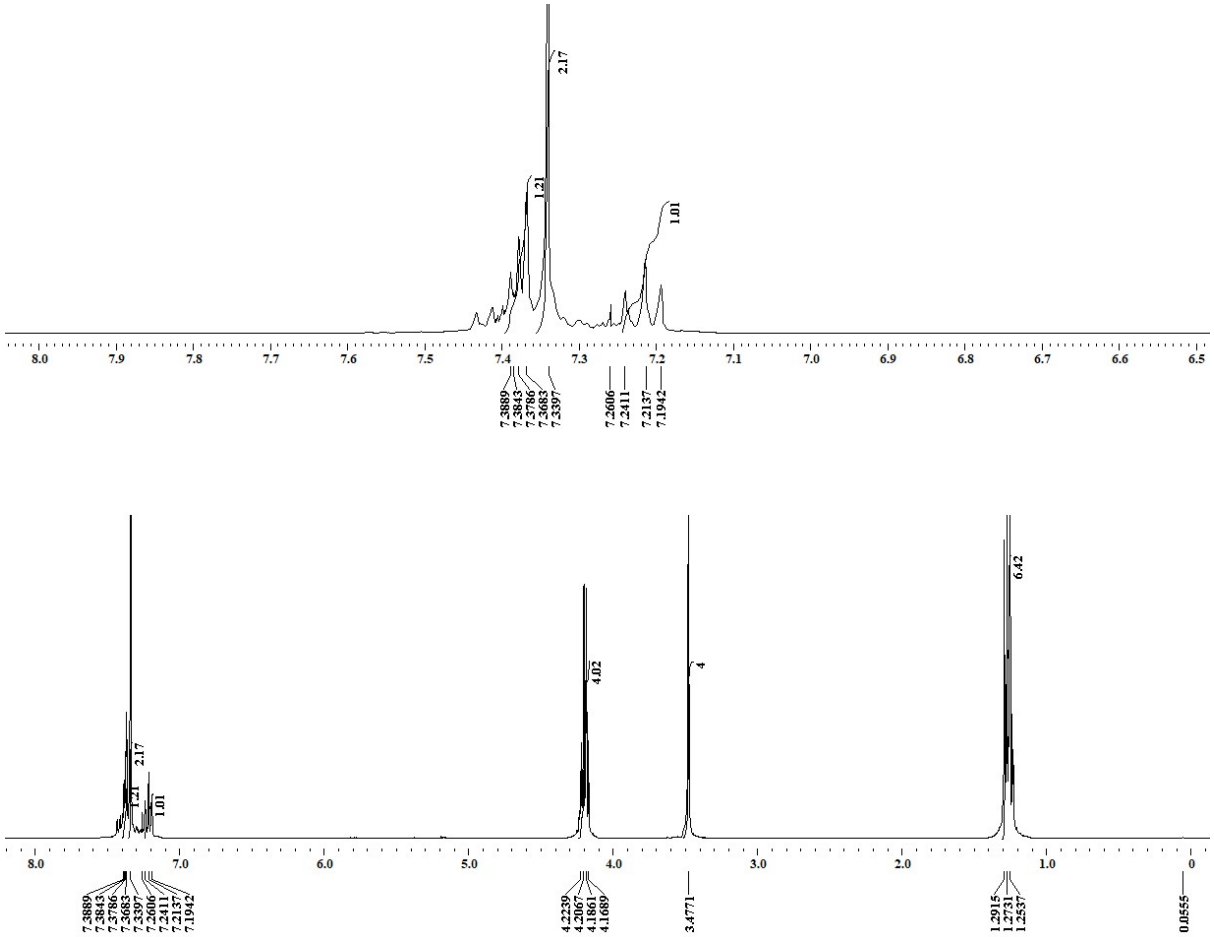
--- End Of Report ---

^{1}H NMR

(400 MHz, $CDCl_3$)

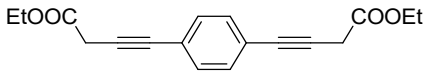


Diethyl 4,4'-(1,4-phenylene)bis(but-3-ynoate) (6a)

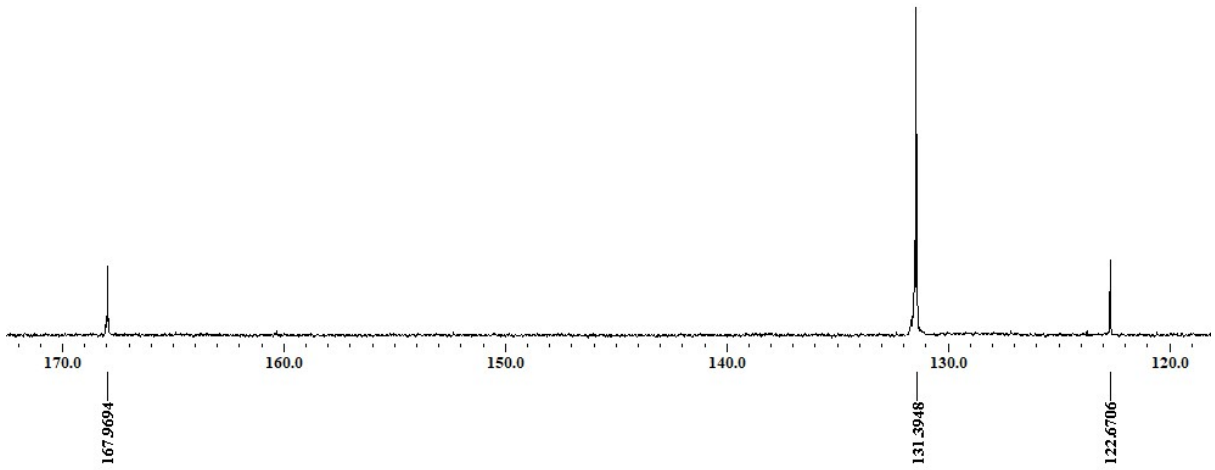


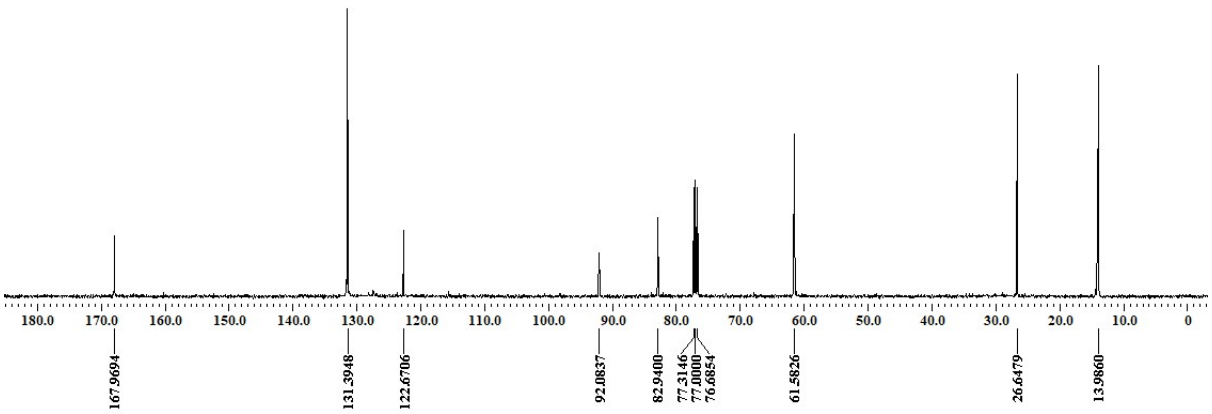
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

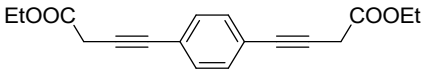


Diethyl 4,4'-(1,4-phenylene)bis(but-3-ynoate) (6a)

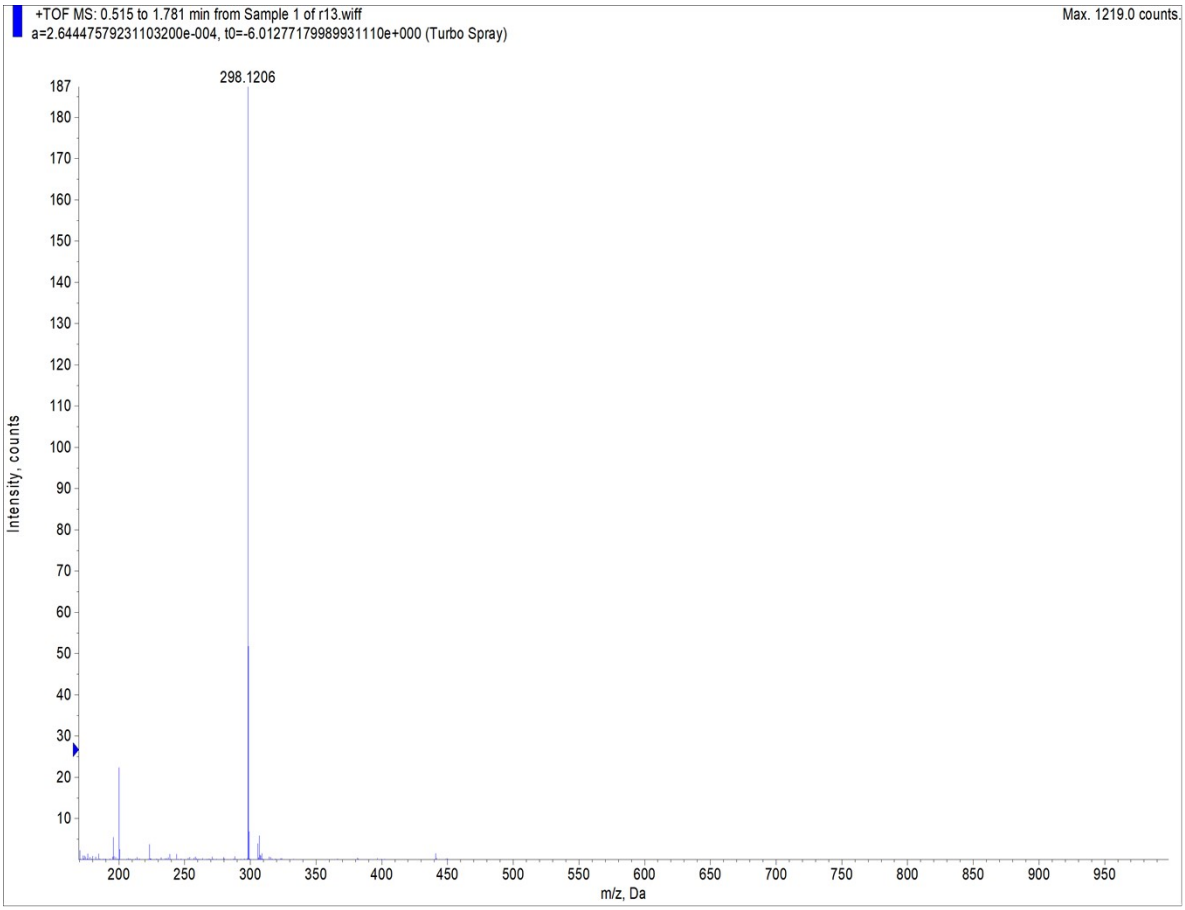




HRMS

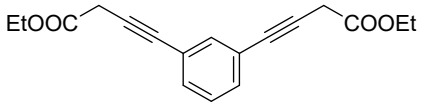


Diethyl 4,4'-(1,4-phenylene)bis(but-3-ynoate)(6a)

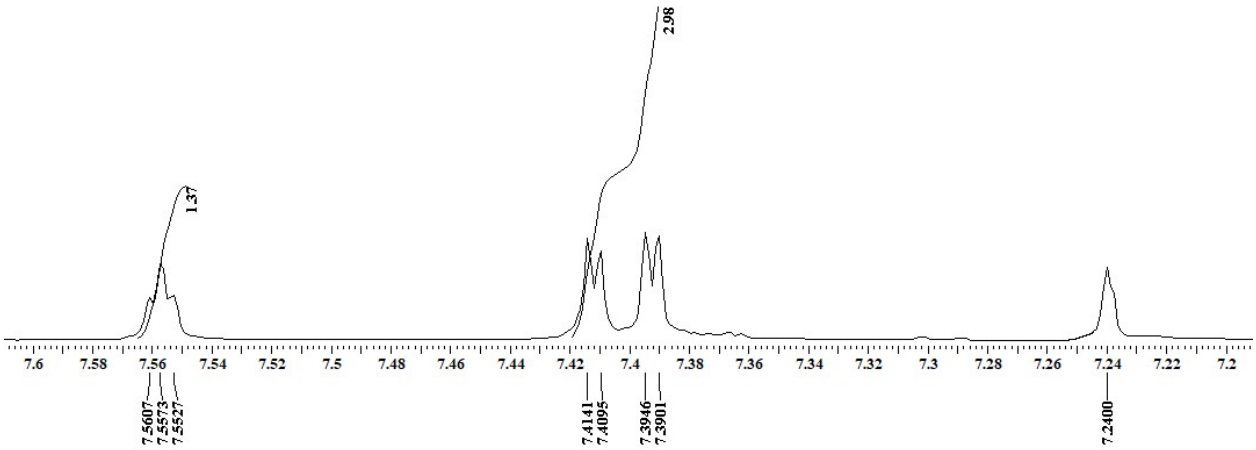


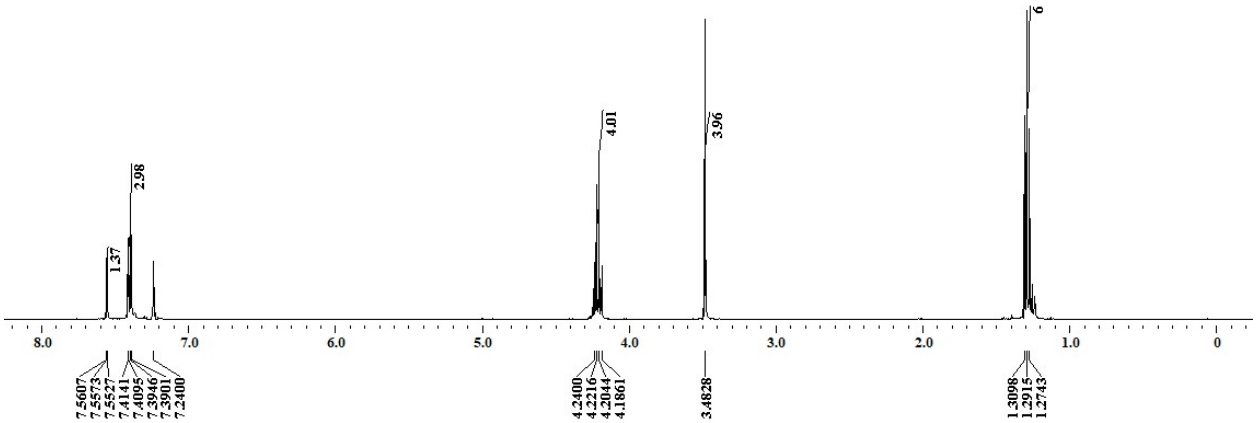
^{1}H NMR

(400 MHz, $CDCl_3$)



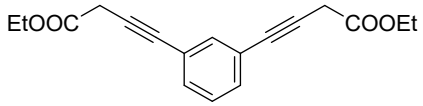
Diethyl 4,4'-(1,3-phenylene)bis(but-3-ynoate)(6b)



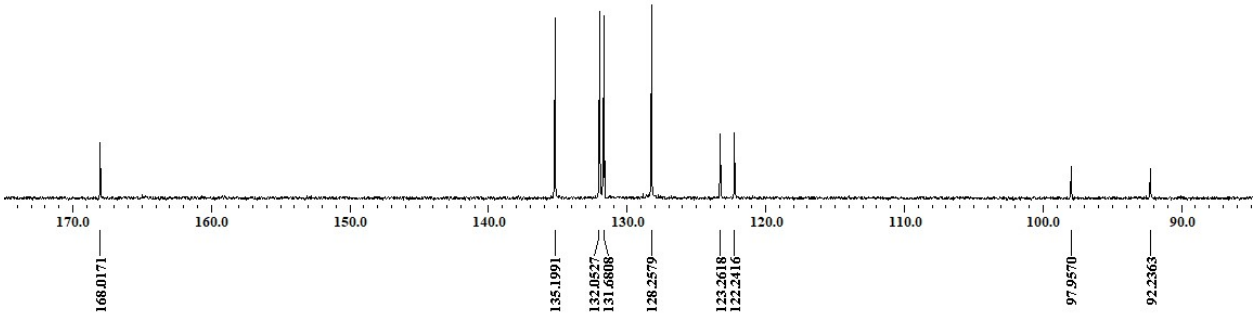


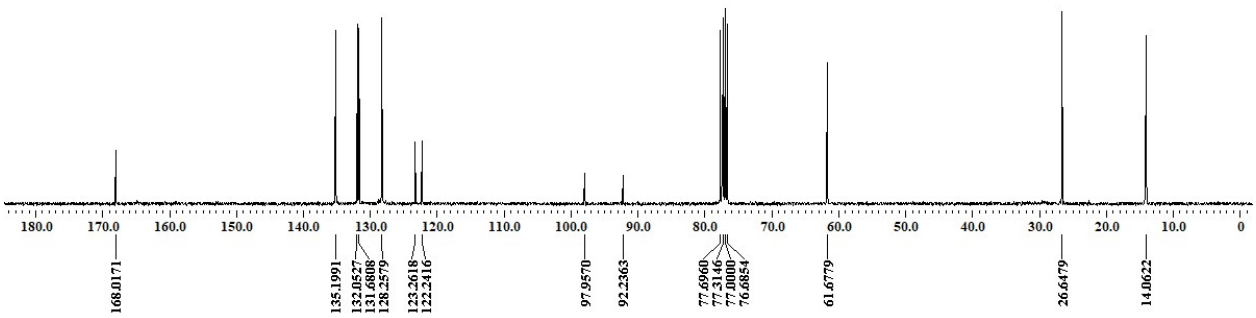
$^{13}C\{^{1}H\}$ NMR

(100 MHz, $CDCl_3$)

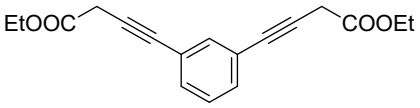


Diethyl 4,4'-(1,3-phenylene)bis(but-3-ynoate)(6b)

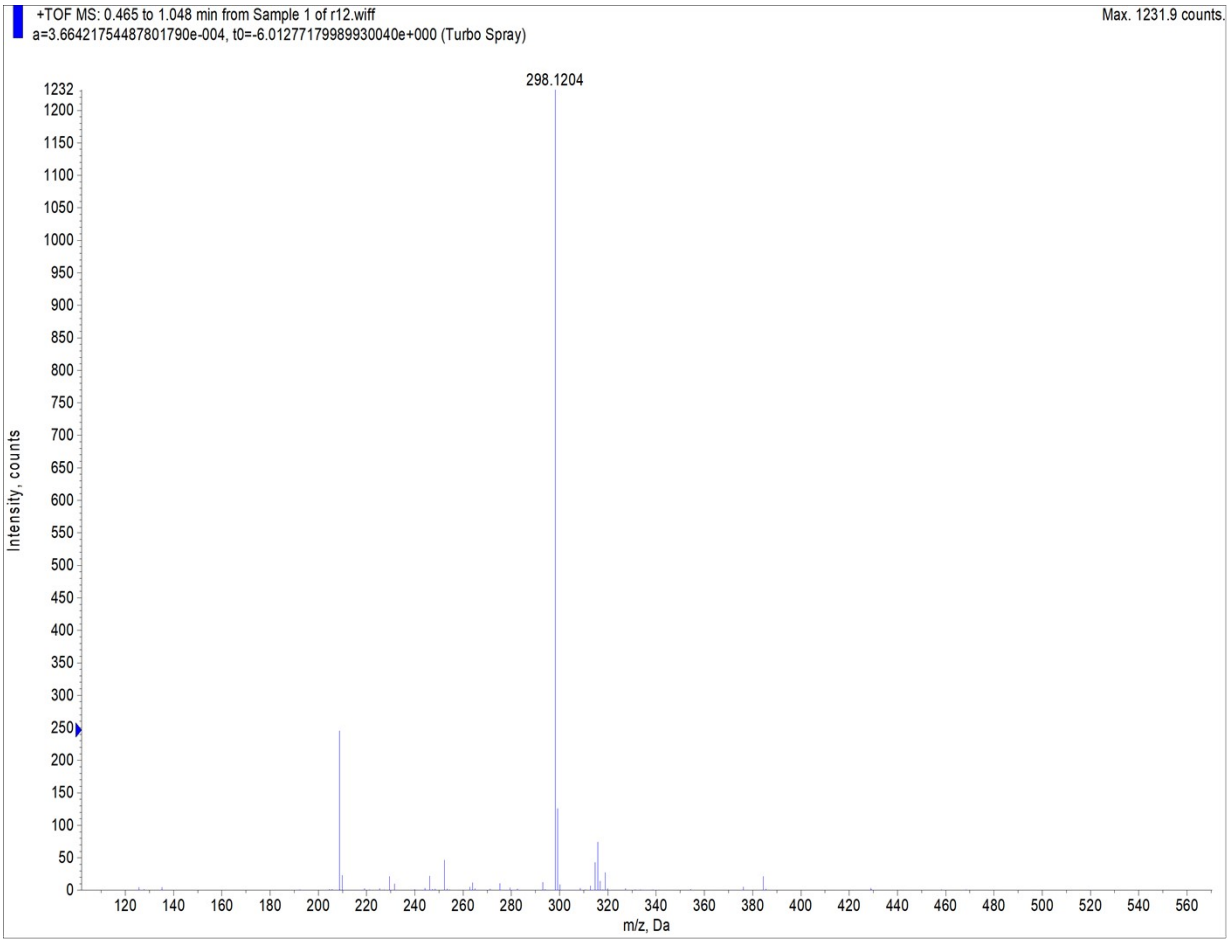




HRMS



Diethyl 4,4'-(1,3-phenylene)bis(but-3-ynoate)(6b)



Reference:

1. K. M. Archana, S. Rajalakshmi, P. S. Kumar, V. G. Krishnaswamy, R. Rajagopal, D.T. Kumar, C. G. P Doss, *Envir. Res*., 2021, **200**, 111759.
2. K. M. Archana, D. Yogalakshmi, R. Rajagopal, *SN Appl. Sciences*., 2019, **1**, 1–14.