Supplementary Information

Tin (ii) Chloride ($SnCl_2$) Mediated Reduction of α, β-alkynyl Carbonyl Compounds

U. Ralepelle*, E. Ndubuisi Agbo, K. Lekgau, H. Chauke, I. Cukrowski, W. Nxumalo*

Experiment

The reactions were conducted in oven-dried (150 ˚C) glassware, magnetically stirred, and monitored using analytical thin layer chromatography (TLC). The visualization of spots was done using ultraviolet fluorescence (UV) light (254 nm). All the commercially available reagents and solvents were purchased from Merk and Sigma Aldrich and were used without further purification, unless stated otherwise. Tetrahydrofuran (THF) was freshly distilled over sodium/benzophenone under nitrogen gas prior to use. Flash column chromatography was carried out on silica gel 60 (230-400 mesh). All reagents were measured at room temperature. The structural properties of compounds were recorded and confirmed by: Nuclear Magnetic Nuclear Magnetic Resonance (NMR) (Bruker Ascend 400 MHz Topspin 3.2) and High-Resolution Mass Spectrometry (HRMS) (Sciex X500R QTOF). All chemical shifts are expressed in part per million (ppm) abbreviated as δ, with respect to an internal standard tetramethyl silane of the ^{1}H and ^{13}C NMR spectra, $CDCl_3$ (^{1}H NMR = 7.25 ppm and ^{13}C NMR = 77.0 ppm). The ^{1}H NMR spectra were reported as follows: δ (position of proton, multiplicity, coupling constant *J*, and number of protons). The multiplicities are expressed by s = singlet, d = doublets, t = triplets, dd = doublet of doublets, m = multiplets and brs = broad singlet.

4.1. General procedure for the Sonagashira cross coupling reaction.

To an oven-dried sealed reaction tube with magnetic stirrer bar, was added 6-nitroquinoxalin-2-yl benzenesulfonate, $PdCl_2(PPh_3)_2$ (5 mol %), CuI (10 mol %), Et_3N (2 eq) and 1-hexyn-3-ol (1.2 eq) in dry THF (10 mL) under nitrogen atmosphere. The reaction mixture was allowed to stir at 80˚C for 16 h. Upon completion, monitored by a thin layer chromatography, saturated ammonium chloride solution (10 mL) was added to the reaction mixture to quench the reaction and the resulting mixture was extracted with EtOAc. The organic layers were combined, dried over anhydrous Na_2SO_4. The crude mixture was purified by flash column chromatography on silica gel eluting with mixture of EtOAc: n-hexane to afford the desired 1-(6-nitroquinoxalin-

2-yl) hex-1-yn-3-ol. Similar reaction procedure was adopted in the synthesis of other *N*-heterocyclic alkynylated derivatives reported in this investigation [24].

4.1.1. 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-ol (5)

Brown solid (435.2 mg, 95%), EtOAc: hexane (2.3, R_f=0.23), mp. 119.1-121.0 °C; δ_H (400 MHz, $CDCl_3$) 8.96 (1H, s), 8.95 (1H, d, *J* = 2.4 Hz), 8.52 (1H, dd, *J* = 9.2 and 2.4 Hz), 8.17 (1H, d, *J* = 9.2 Hz), 4.74 (1H, t, *J* = 6.7 Hz), 2.79 (1H, brs), 1.85-1.90 (2H, m), 1.56-1.62 (2H, m), 0.99 (3H, t, *J* = 7.4 Hz); δ ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 149.0,147.9, 144.4, 141.8, 139.8, 130.8, 125.6, 124.2, 97.7, 81.9, 62.6, 39.3, 18.4, 13.7; HRMS (ESI) $[M + H]^+$: m/z 272.1042; Calculated mass for $C_{14}H_{14}N_3O_3$ is 272.1037.

4.1.2. Synthesis of 1-(6-chloroquinoxalin-2-yl) hex-1-yn-3-ol

Brown solid (430 mg, 90%), EtOAc: hexane (2.3, R_f=0.23), mp. 104.1- 108.3 °C; δ_H (400 MHz, $CDCl_3$) δ 8.83 (1H, s), 8.05 (1H, d, *J* = 2.4 Hz), 7.70 (1H, dd, *J* = 9.2 and 2.4 Hz), 7.97 (1H, d, *J* = 9.2 Hz), 4.70 (1H, t, *J* = 6.7 Hz), 2.79 (1H, brs), 1.84-1.90 (2H, m), 1.55-1.61 (2H, m), 0.98 (3H, t, *J* = 7.4 Hz); δ ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 147.9, 141.2, 140.5, 139.0, 136.5, 131.9, 130.3, 128.1, 95.4, 80.5, 62.6, 39.4, 18.5, 13.7; HRMS (ESI) $[M + H]^+$: m/z 261.0770; Calculated mass for $C_{14}H_{13}ClN_2O$ is 261.0716.

4.1.3. Synthesis of 1-(pyrazin-2-yl) hex-1-yn-3-ol

brown oil (318 mg, 90%), EtOAc: n-hexane (2:3, R_f=0.35). δ_H (400 MHz, $CDCl_3$) δ 8.55-8.49 (2H, m,), 8.67 (1H, s), 4.57 (1H, t, *J* = 9.0 Hz), 2.10 (1H, brs), 1.79-1.85 (2H, m), 1.52-1.58 (2H, m), 0.97 (3H, t, *J* = 7.3). δ ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 13.7, 18.4, 39.4, 62.4, 81.3, 94.5, 139.7, 144.4, 145.9, 147.6. HRMS (ESI) $[M + H]^+$: m/z 177.1028; Calculated mass for $C_{10}H_{12}N_2O$ is 177.0950.

4.1.4. Synthesis of 1-(pyrimidine-2-yl) hex-1-yn-3-ol

dark brown oil (252 mg, 98%), EtOAc: n-hexane (2:3, R_f=0.28). δ_H (400 MHz, $CDCl_3$) δ 8.62 (2H, d, *J* = 4.97 Hz), 7.30 (1H, t, J = 5.0 Hz), 4.64 (1H, t, *J* = 6.7 Hz), 2.16 (1H, brs), 1.79-1.85 (2H, m), 1.51-1.60 (2H, m), 0.95 (3H, t, *J* = 7.4 Hz). δ ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 157.2(x2), 152.5, 119.9, 90.1, 82.9, 62.2, 39.2, 18.4, 13.7. HRMS (ESI) $[M + H]^+$: m/z 177.1026; Calculated mass for $C_{10}H_{13}N_2O$ is 177.0950.

4.1.5. Synthesis of 1-(pyridine-2-yl) hex-1-yn-3-ol

yellow oil (252 mg, 98%), EtOAc: n-hexane (4:1, R_f=0.47). δ_H (400 MHz, $CDCl_3$) δ 8.48 (1H, d, *J* = 5.3 Hz), 7.56-7.60 (1H, m), 7.34 (1H, d, *J* = 7.9 Hz), 7.14-7.18 (1H, m), 4.67 (1H, t, *J* =

6.9 Hz), 2.18 (1H, brs), 1.80-1.86 (2H, m), 1.54-1.60 (2H, m), 0.97 (3H, t, *J* = 7.4 Hz); δ ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 149.8, 142.9, 136.6, 127.41, 123.2, 91.7, 83.7, 62.4, 39.8, 18.7, 13.9; HRMS (ESI) $[M + H]^+$: m/z 176.1067; Calculated mass for $C_{11}H_{14}NO$ is 176.0997.

4.2. General procedure for Oxidation reaction

To a solution of 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-ol in dichloromethane (30 mL) was added 5eq. of PCC at 0 ˚C, and then warmed slowly to room temperature. The mixture was stirred for 2 hours at room temperature. The reaction mixture was filtered off through a short silica to allow the product to separate from unreacted PCC and the filtrate were concentrated under vacuum pressure. The resulting crude was further diluted with DCM, washed with brine, and extracted with DCM. The crude was purified by flash column chromatography to obtain 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-one [24]. Similar reaction method was adopted in the oxidation of *N*-heterocyclic alkynylated derivatives reported in this investigation.

4,2,1 Synthesis of 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-one (1)

Brown solid (158mg, 80%); methanol: DCM (2:3, R_f=0.57), mp = 132.9 -137.2˚C (Lit 135.4-138.0 ˚C); δ_H (400 MHz, $CDCl_3$) δ 9.09 (1H, s) 9.02 (1H, d, *J* = 2.4 Hz) ,8.59 (1H, dd, *J* = 9.3 and 2.4 Hz), 8.26 (1H, d, *J* = 9.2 Hz) 2.76 (2H, t, *J* = 7.3 Hz), 1.80 (2H, sext, *J* = 7.4 Hz), 1.01 (3H, t, *J* = 7.4 Hz); δ^{13}C NMR (100 MHz, $CDCl_3$, ppm)186.7, 148.9, 148.6, 144.3, 140.5, 140.0, 131.3, 125.7, 124.5, 90.1, 83.7, 47.4, 17.3, 13.5; HRMS (ESI) $[M + H]^+$:m/z 270.0883; Calculated mass for $C_{14}H_{12}N_3O_3$ is 270.0880.

4.2.2 Synthesis of 1-(6-chloroquinoxalin-2-yl) hex-1-yn-3-one (7)

Brown solid (340 mg, 76%); methanol: DCM (2:3, R_f=0.66),mp = 120.6-128.0˚C ; δ_H (400 MHz, $CDCl_3$) δ 8.97 (1H, s) 8.12 (1H, d, *J* = 2.4 Hz),8.05 (1H, d, *J* = 9.2Hz), 7.78 (1H, dd, *J* = 9.2 Hz and 2.4 Hz), 2.74 (2H, t, *J* = 7.6 Hz), 1.80 (2H, sext), 1.00 (3H, t, *J* = 7.17 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 187.2, 147.9, 141.8, 140.7, 137.8, 137.1, 132.4, 130.7, 128.3, 88.9, 84.0, 47.4, 17.3, 13.5. HRMS (ESI) $[M + H]^+$:m/z 259.0625; Calculated mass for $C_{14}H_{12}ClN_2O$ is 259.0593.

4.2.3. Synthesis of 1-(pyrazin-2-yl) hex-1-yn-3-one (11)

Brown oil (269 mg, 86%); methanol: DCM(4:1, R_f=0.24) , δ_H (400 MHz, $CDCl_3$) δ 8.80 (1H, s, H-3), 8.62 (2H, d, *J* = 13.9 Hz), 2.70 (2H, t, *J* = 7.1 Hz), 1.78 (2H, sext), 099 (3H, t, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 186.2, 147.8, 143.9, 143.7, 136.9, 87.8, 83.3, 46.4, 16.4, 13.1. HRMS (ESI) $[M + H]^+$:m/z 175.0866; Calculated mass for $C_{10}H_{11}N_2O$ is 175.0793.

4.2.4. Synthesis of 1-(pyrimidine-2-yl) hex-1-yn-3-one (9)

Brown oil (120 mg, 77%); methanol: DCM(1:4, Rf=0.22), δ_H (400 MHz, $CDCl_3$) δ 8.79, (2H, d, J = 5.1 Hz), 7.35 (1H, t, J = 4.9 Hz), 2.70 (2H, t, *J* = 7.4 Hz), 1.77 (2H, sext), 0.97 (3H, t, J = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 187.2, 157.5, 157.5, 151.5, 121.1, 82.7, 78.0, 47.4, 17.2, 13.4. HRMS (ESI) $[M + H]^+$: m/z 175.0866; Calculated mass for $C_{10}H_{11}N_2O$ is 175.0793.

4.2.5. **Synthesis of 1-(pyridine-2-yl) hex-1-yn-3-one (14)**

The Jones reagent was used for the oxidation reaction [26] using 1-(pyridin-2-yl) hex-1-yn-3-ol (0.200 g) in acetone (40 mL) and added a mixture of chromium trioxide (1.5 eq., 0.170 g), concentrated sulfuric acid (0.073 mL) and water (4 mL) dropwise over a period of 15 min at 0˚C. After stirring at room temperature for 16 hours, the reaction was further diluted with water (15 mL). The product was isolated by extracting with diethyl ether and drying over $NaHSO_4$. The solvent was removed through evaporator vacuum and the crude was purified over flash chromatography in silica gel eluting with a mixture of ethyl acetate: n-hexane (4:1, R_f=0.33) to afford a pure product of 1-(pyrimidin-2-yl) hex-1-yn-3-one as a light brown oil (0.146 g, 73%). δ_H (400 MHz, $CDCl_3$) 8.66 (1H, d, *J* = 4.7 Hz), 7.73 (1H, m), 7.58 (1H. d, *J* = 7.9 Hz), 7.36 (1H, m), 2.68 (2H, t, *J* = 7.3 Hz), 1.76 (2H, sext), 0.96 (3H, t, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 187.9, 150.5, 140.8, 136.6, 128.6, 124.7, 87.6, 85.8, 47.5, 16.6, 13.7. HRMS (ESI) $[M + H]^+$: m/z 178.1216; Calculated mass for $C_{11}H_{15}NO$ is 178.1154.

4,3. General procedure for the reduction reaction using $SnCl_2$

To a suspension of α, β - alkynyl carbonyl compound in EtOAc (10 mL) was added $SnCl_2$ (2 and 5 eq.) and the reaction mixture was stirred for 3hours at 60˚C. After cooling to room temperature, $NaHCO_3$ solution (10 mL) was added, and the reaction mixture stirred for 10 minutes. The suspension was partitioned, and the aqueous layer washed with EtOAc. The combined organic layers were washed with brine, removed under vacuum pressure, and purified by column chromatography to afford the desired reduced product [27]. This method was adopted in the synthesis of the reduced compounds below.

4.3.1. Synthesis of 1-(6- nitroquinoxalin-2-yl) hexan-3-one (6)

Brown solid (21 mg, 47%); EtOAc: n-hexane (4:1, R_f=0.50), mp = 122.8-160.0˚C; δ_H (400 MHz, $CDCl_3$) δ 8.98 (1H, d, *J* = 2.4 Hz), 8.93 (1H, s),8.49 (1H, dd, *J* = 8.8 Hz and 2.4Hz), 8.09 (1H, d, *J* = 8.8 Hz), 3.35 (2H, t, *J* = 6.7 Hz), 3.12 (2H, t, *J* = 6.4 Hz), 2.51 (2H, t, *J* = 7.5 Hz), 1.64 (2H, sext), 0.92 (3H, t, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 207.2(x4),

159.5, 148.6, 130.4, 125.7, 123.3, 44.7, 39.42, 31.9, 17.3, 13.7. HRMS (ESI) $[M + H]^+$: m/z 274.1193; Calculated mass for $C_{14}H_{16}N_3O_3$ 274.1113.

4.3.2. Synthesis of 1-(6- aminoquinoxalin-2-yl) hexan-3-one (3)

Yellow solid (21 mg, 47%); EtOAc: n-hexane (2:3, R_f=0.23), mp = 96.8-98.0˚C (Lit 97.0-97.6 °C) [25] [28]; δ_H (400 MHz, $CDCl_3$, ppm) δ 8.60 (1H, s); 7.75 (1H, d, *J* = 8.8 Hz), 7.15 (1H, d, *J* = 2.4 Hz), 7.14 (1H, dd, *J* = 8.8 Hz and 2.4 Hz),4.11 (2H, brs), 3.20 (2H, t, *J* = 7.1 Hz), 2.99 (2H, t, *J* = 7.1 Hz), 2.46 (2H, t, *J* = 7.3 Hz), 2.46 (2H, t, *J* = 7.3 Hz), 1.57-1.66 (2H, m), 0.90 (3H, t, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 209.9, 151.7, 147.4, 145.4, 138.8, 135.2, 129.6, 122.0, 107.9, 44.9, 40.7, 31.9, 17.3, 13.8; HRMS (ESI) $[M + H]^+$: m/z 244.1440; Calculated mass for $C_{14}H_{18}N_3O$ is 244.1372

4.3.3. Synthesis of 1-(6-chloroquinoxalin-2-yl)hexan-3-one (8)

Brown oil (37 mg, 73%); EtOAc: n-hexane (2:3, R_f=0.64), δ_H (400 MHz, $CDCl_3$) δ 8.78 (1H, s,H-3); 8.05 (1H, d, *J* = 2.4 Hz), 7.91 (1H, d, *J* = 8.8 Hz), 7.66 (1H, dd, *J* = 8.8 Hz and 2.4 Hz), 3.29 (2H, t, *J* = 6.9 Hz), 3.07 (2H, t, *J* = 7.0Hz), 2.49 (2H, t, *J* = 7.5 Hz), 1.62 (2H, sext), 0.91 (3H, t, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 209.7, 156.2, 147.0, 141.5, 140.6, 134.7, 130.9, 130.0, 127.6, 44.9, 39.9, 31.9, 17.3, 13.8. HRMS (ESI) $[M + H]^+$: m/z 263.0933; Calculated mass for $C_{14}H_{16}ClN_2O$ is 263.0906.

4.3.4. Synthesis of 1-(pyrazin-2-yl) hexane-3-one (13)

Yellow oil in 95% yield; EtOAc: n-hexane (2:3, R_f=0.28). δ_H (400 MHz, $CDCl_3$) δ 8.53 (1H, s), 8.40-8.46 (2H, m), 3.09 (2H, d, *J* = 9.18Hz), 2.94 (2H, t, *J* = 7.1 Hz), 2.43 (2H, t, *J* = 7.4 Hz), 1.58 (2H, sext), 0.88 (3H, t, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 209.5, 156.3, 144.9, 143.6, 142.0, 44.7, 40.5, 31.9, 17.2, 13.7. HRMS (ESI) $[M + H]^+$: m/z 179.1177; Calculated mass for $C_{10}H_{15}N_2O$ is 179.1106.

4.4.5. Synthesis of (E)-1-(pyrazin-2-yl) hex-1-en-3-one (12)

Light yellow oil in 38% yield; EtOAc: n-hexane (2:3, R_f=0.21). δ_H (400 MHz, $CDCl_3$) δ 8.69 (1H, s), 8.54-8.61 (2H, m), 7.55 (1H, d, *J* = 15.9 Hz), 7.30 (1H, d, *J* = 15,9), 2.67 (2H, t, *J* = 7.3 Hz), 1.72 (2H, sext), 0.97 (3H, t, *J* = 7.4, Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 200.12, 148.9, 145.3, 144.4, 139.3, 136.6, 131.3, 43.8, 17.5, 13.8. HRMS (ESI) $[M + H]^+$: m/z 177.1021; Calculated mass for $C_{10}H_{13}N_2O$ is 177.0983.

4.3.6. Synthesis of 1-(pyrimidine-2-yl) hexane-3-one (10)

Light brown oil in 80% yield; EtOAc: n-hexane (2:3, R_f=0.26). δ_H (400 MHz, $CDCl_3$) δ 8.64 (2H, d, *J* = 4.93 Hz), 7.13 (1H, m, *J* = 4.8 Hz), 3.28 (2H, t, *J* = 7.5 Hz), 2.98 (2H, t, *J* = 7.5

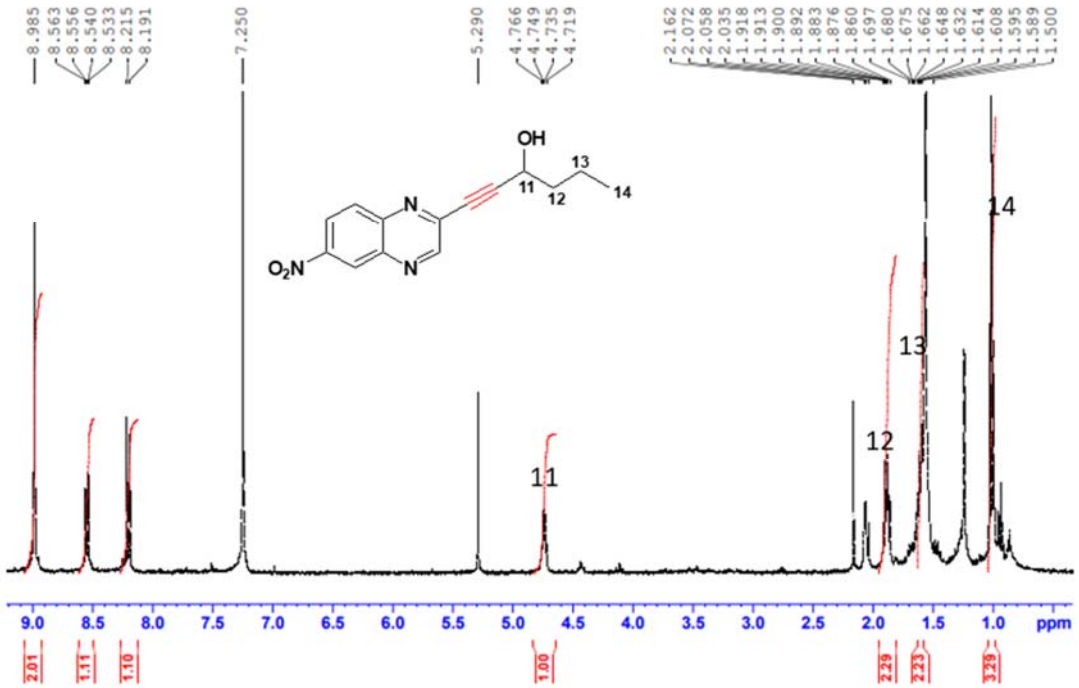
Hz), 2.47 (2H, t, *J* = 7.4 Hz), 1.61 (2H, sext), 0.90 (3H, t, *J* = 7.4, Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) 209.9, 169.8, 156.9, 138.4, 118.6, 44.8, 39.8, 22.7, 17.3, 13.8. HRMS (ESI) $[M + H]^+$: m/z 179.1177; Calculated mass for $C_{10}H_{15}N_2O$ is 179.1106.

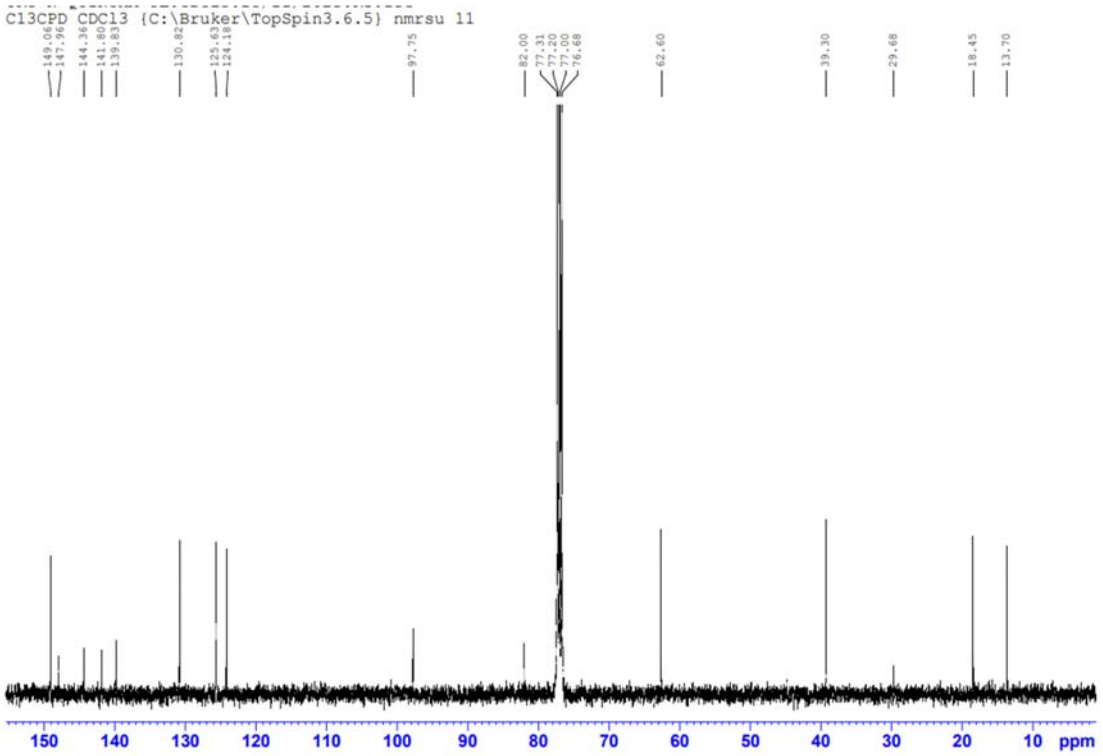
4.3.7. Synthesis of 1-(pyridine-2-yl) hexane-3-one (15)

Dark yellow oil (26 mg, 60%), EtOAc: n-hexane (4:1, Rf=0.51). δ_H (400 MHz, $CDCl_3$, ppm) δ 8.53 (1H, d, *J* = 4.2 Hz), 7.78 (1H, m, *J* = 7.2 Hz), 7.4 (1H, d, *J* = 7.7 Hz), 7.28-7.25 (1H, m, *J* = 5.9 Hz), 3.16(2H, t, *J* = 6.8 Hz), 3.04 (2H, t, *J* = 6.7 Hz), 2.40 (2H, t, *J* = 7.9 Hz), 1.57 (2H, sext), 0.87 (3H, t, *J* = 7.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 210.3, 160.3, 148.4, 137.1, 122.1, 121.4, 44.8, 41.6, 31.3, 17.2, 13.7. HRMS (ESI) $[M + H]^+$: m/z 174.0904; Calculated mass for $C_{11}H_{16}NO$ is 174.0841.

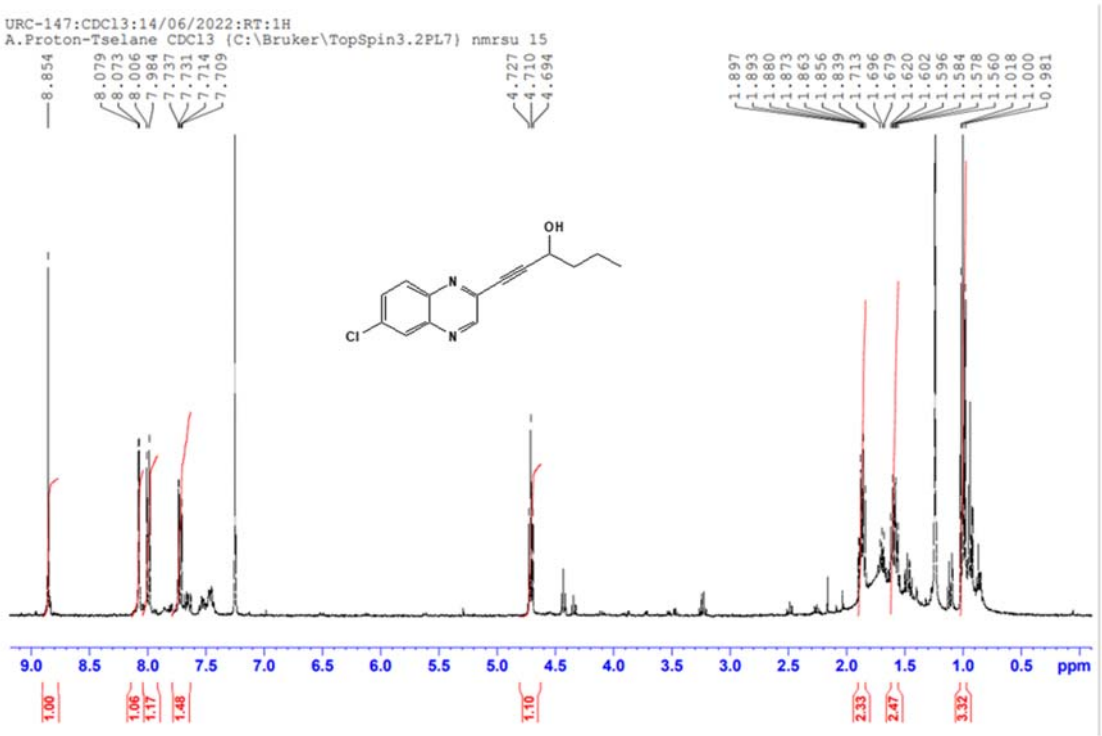
5.1. Sonogashira coupling.

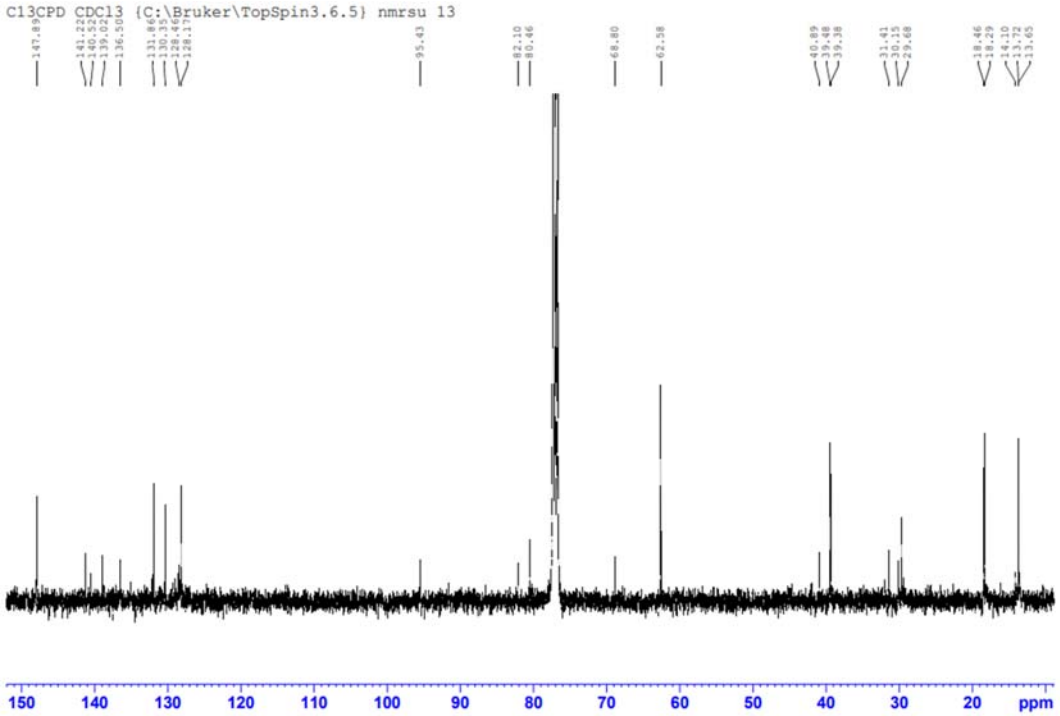
5.1.1. 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-ol (5)





5.1.2. 1-(6-chloroquinoxalin-2-yl) hex-1-yn-3-ol

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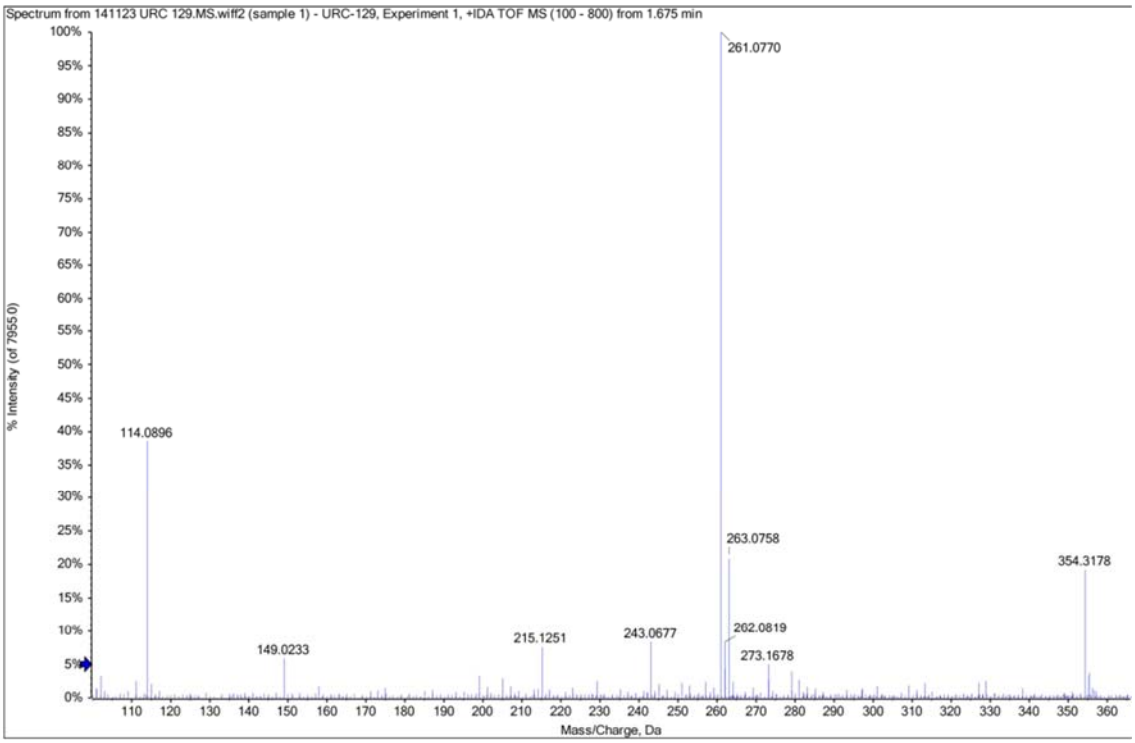
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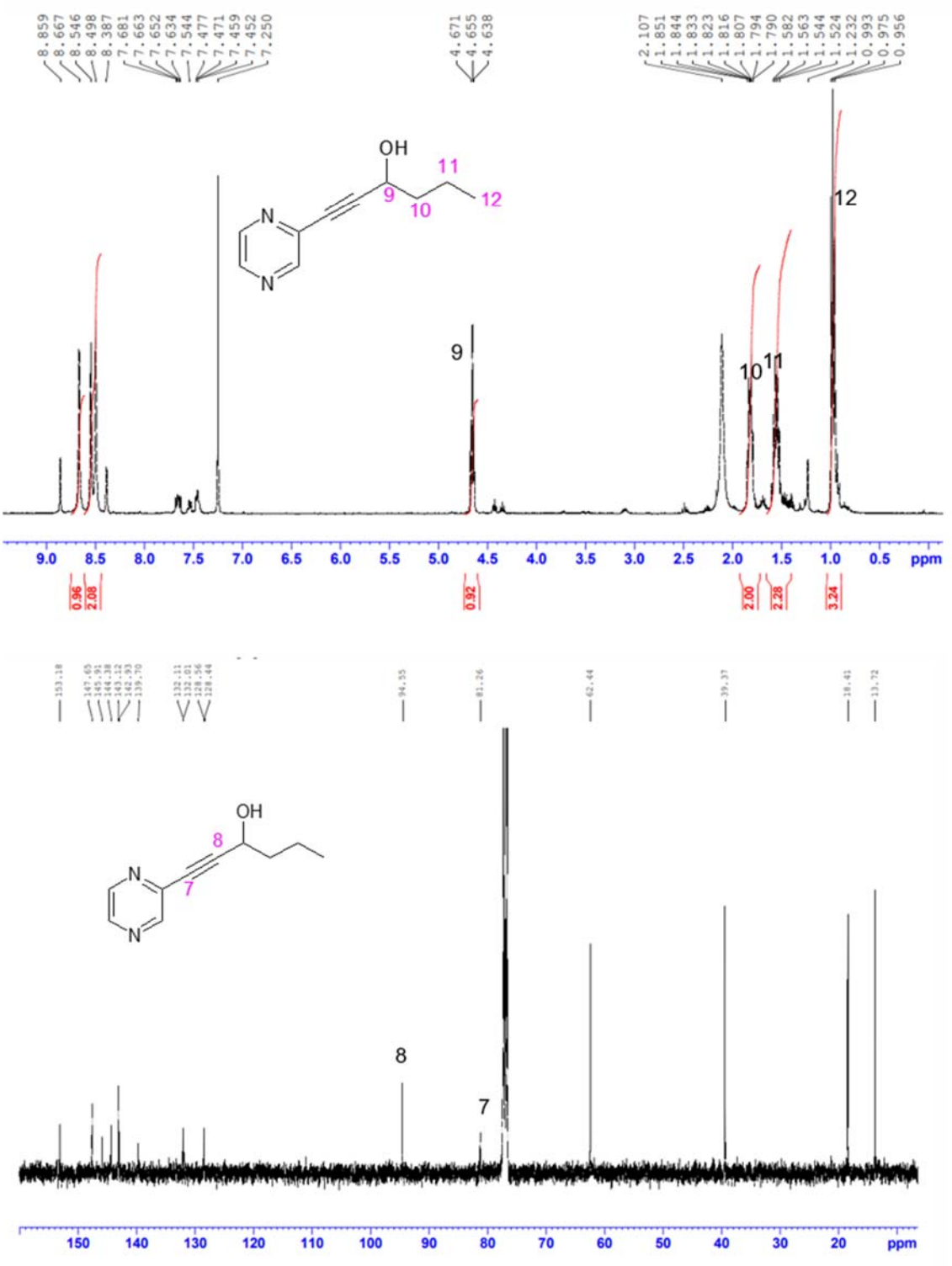
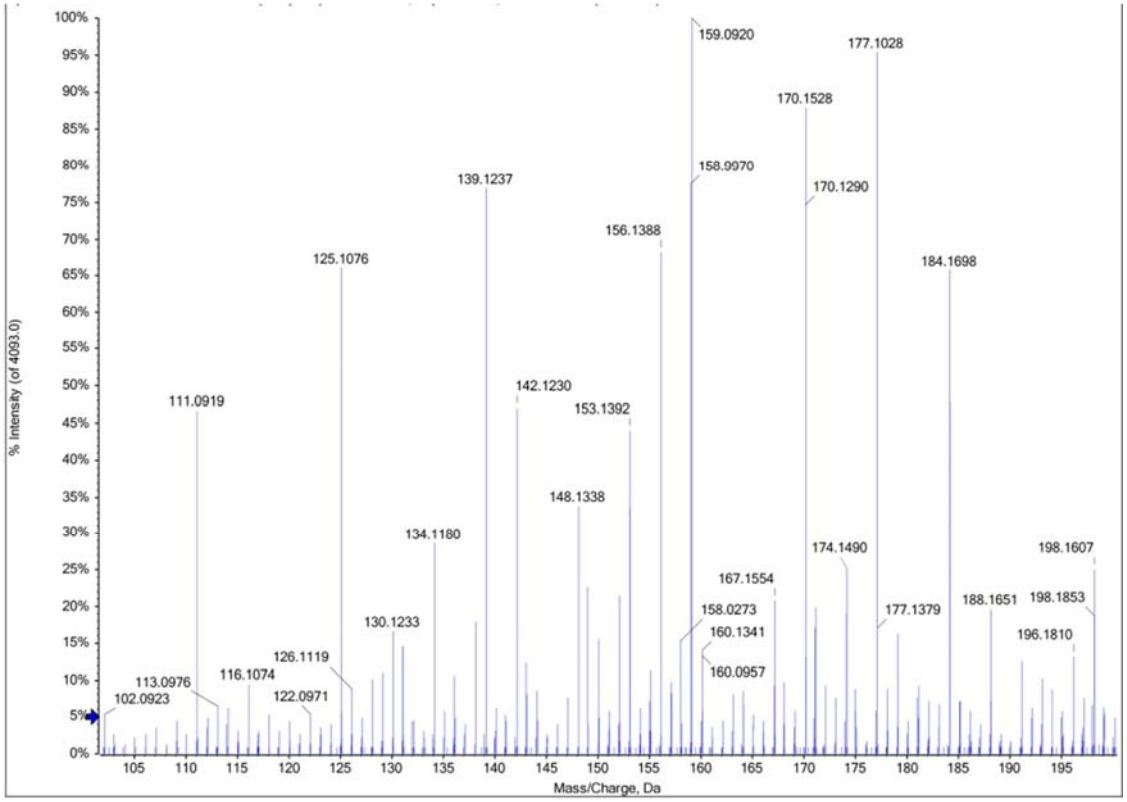
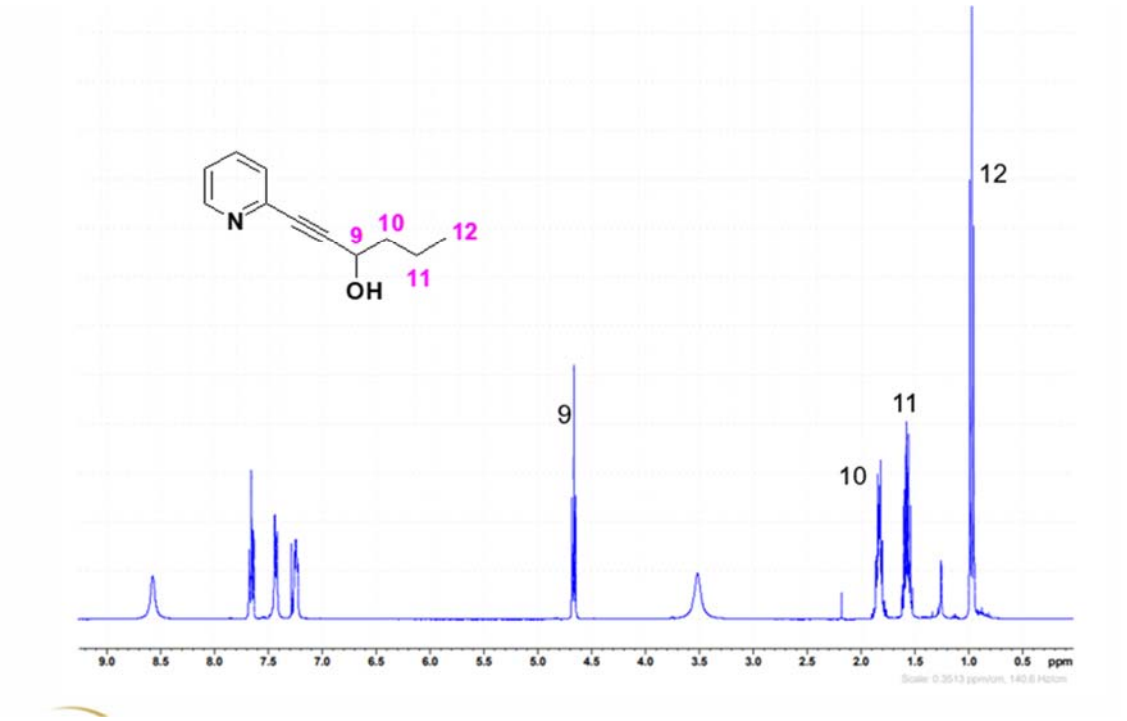
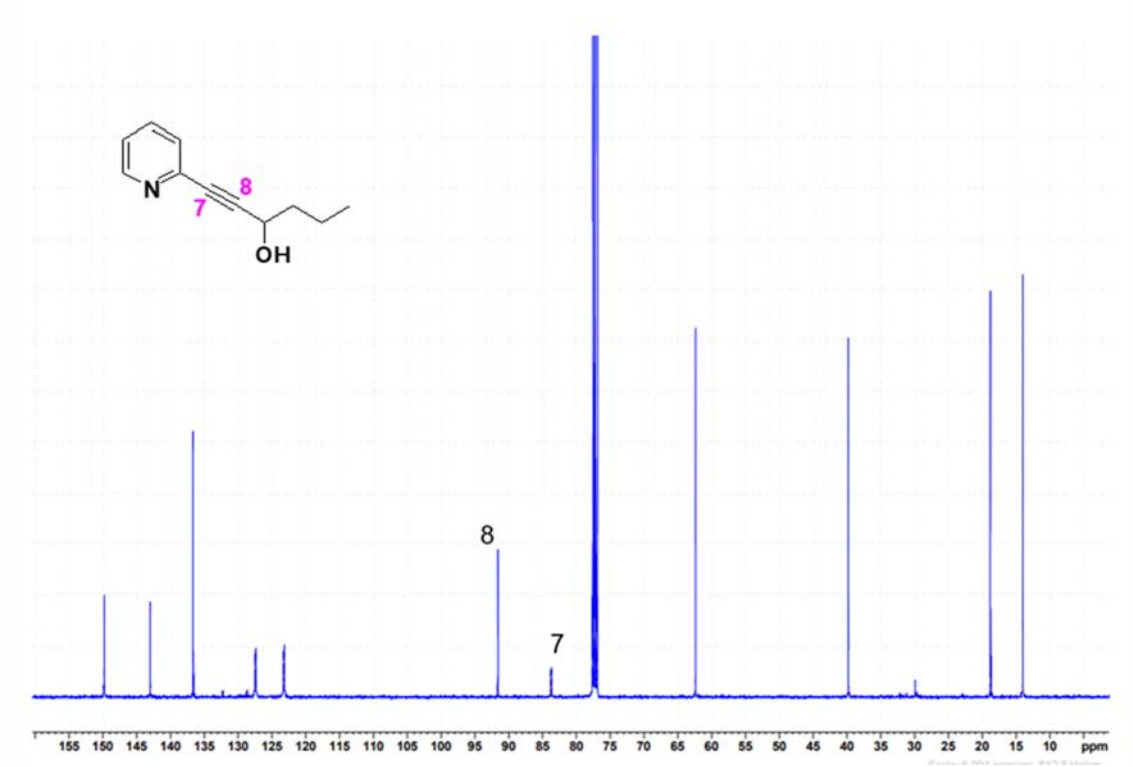
5.1.3. 1-(pyrazin-2-yl) hex-1-yn-3-ol

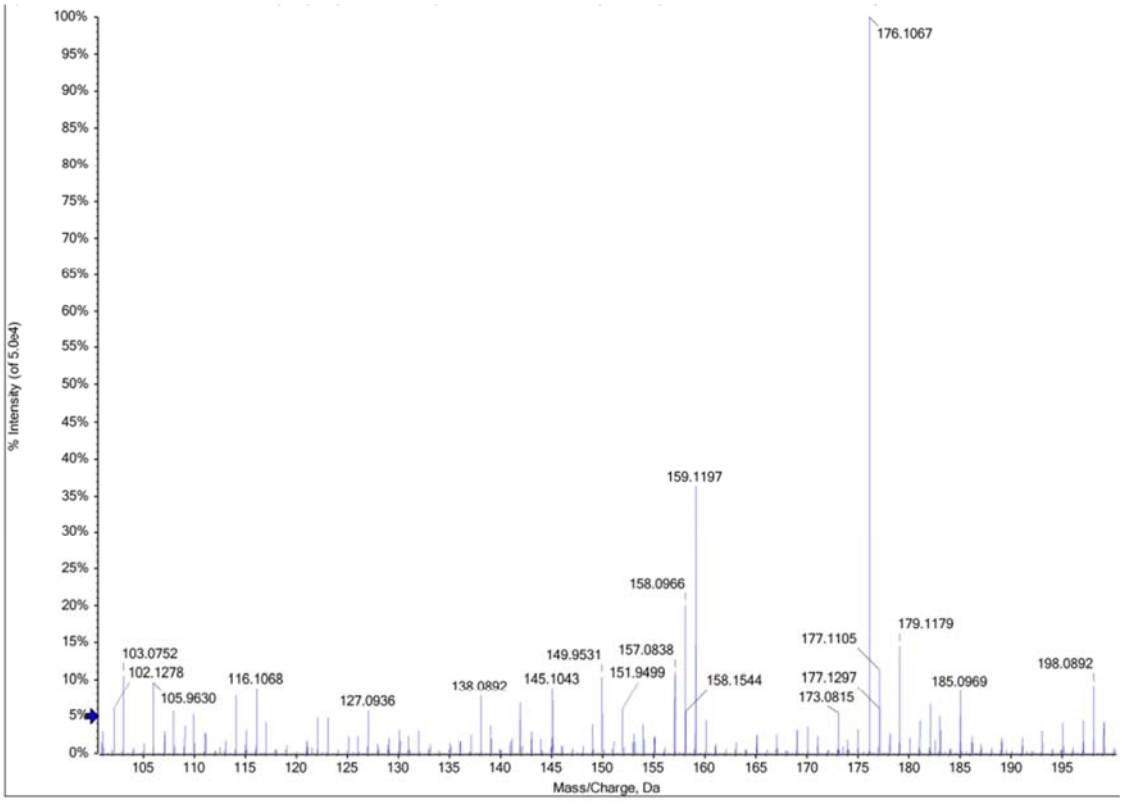




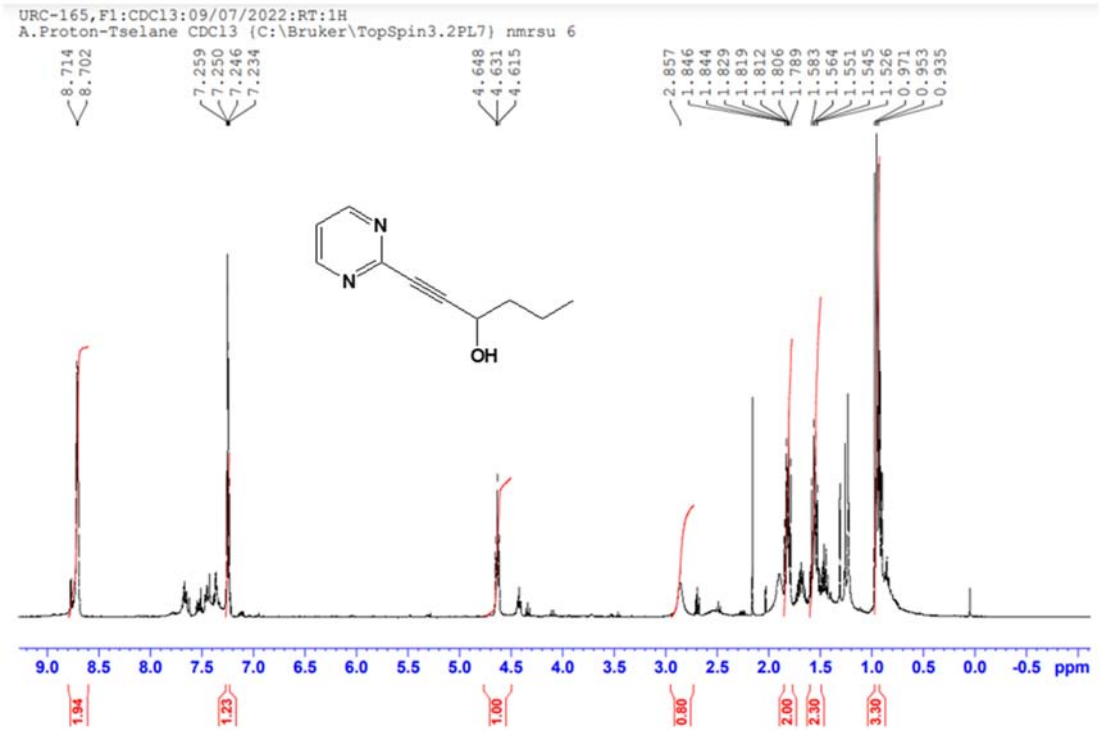
5.1.4. 1-(pyridine-2-yl) hex-1-yn-3-ol

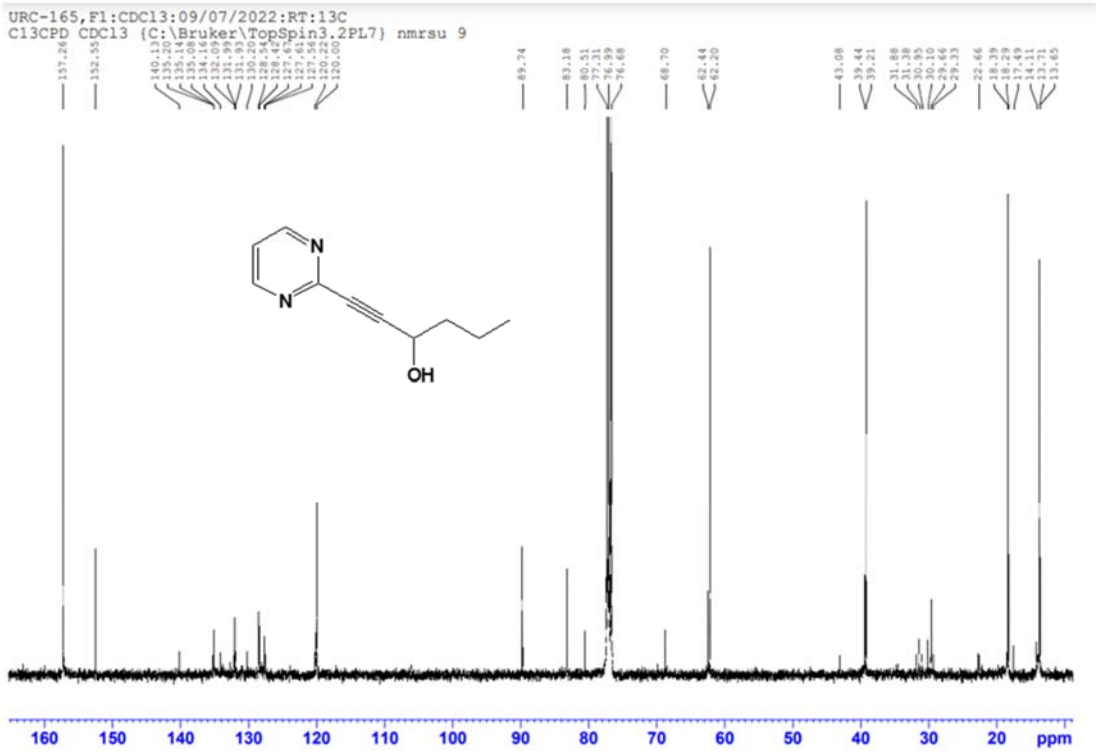


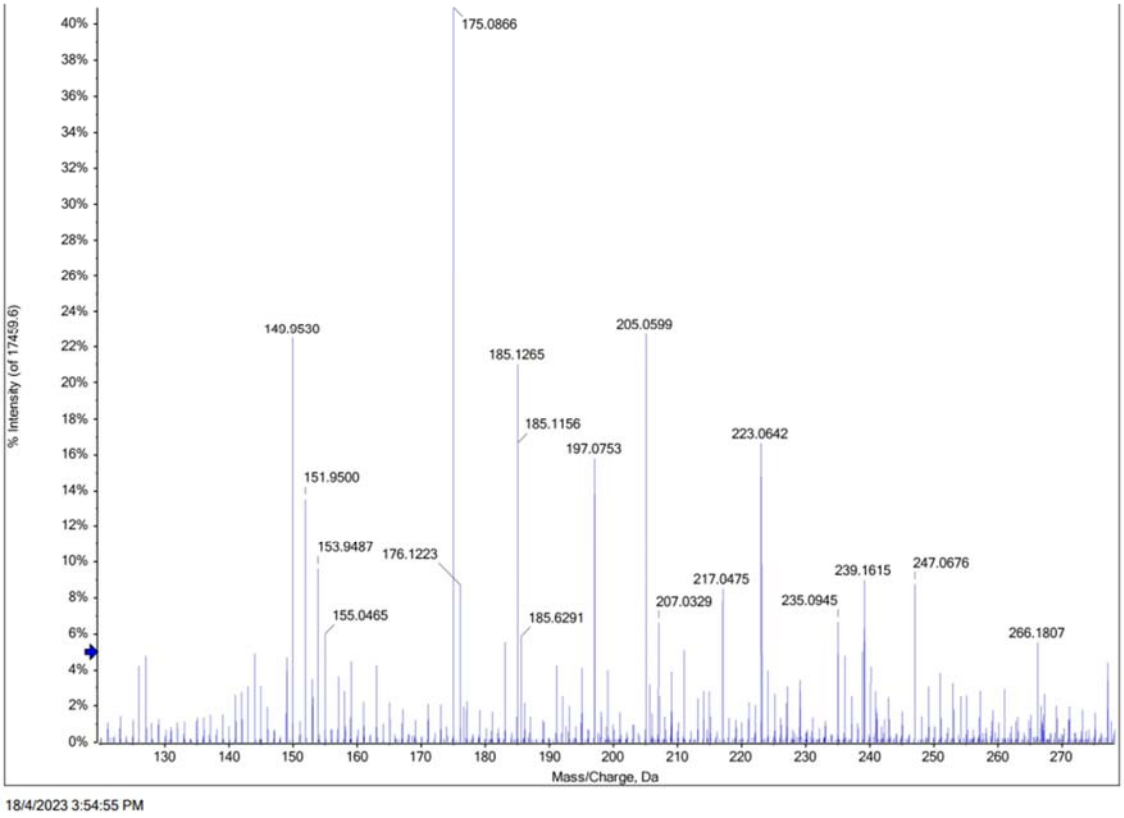

N
8
7
OH
8
7
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 ppm


% Intensity (of 5.0e4)
Mass/Charge, Da
176.1067
159.1197
158.0966
179.1179
177.1105
103.0752
102.1278
105.9630
116.1068
127.0936
138.0892
145.1043
149.9531
157.0838
151.9499
158.1544
177.1297
173.0815
185.0969
198.0892

5.1.5. 1-(pyrimidine-2-yl) hex-1-yn-3-ol

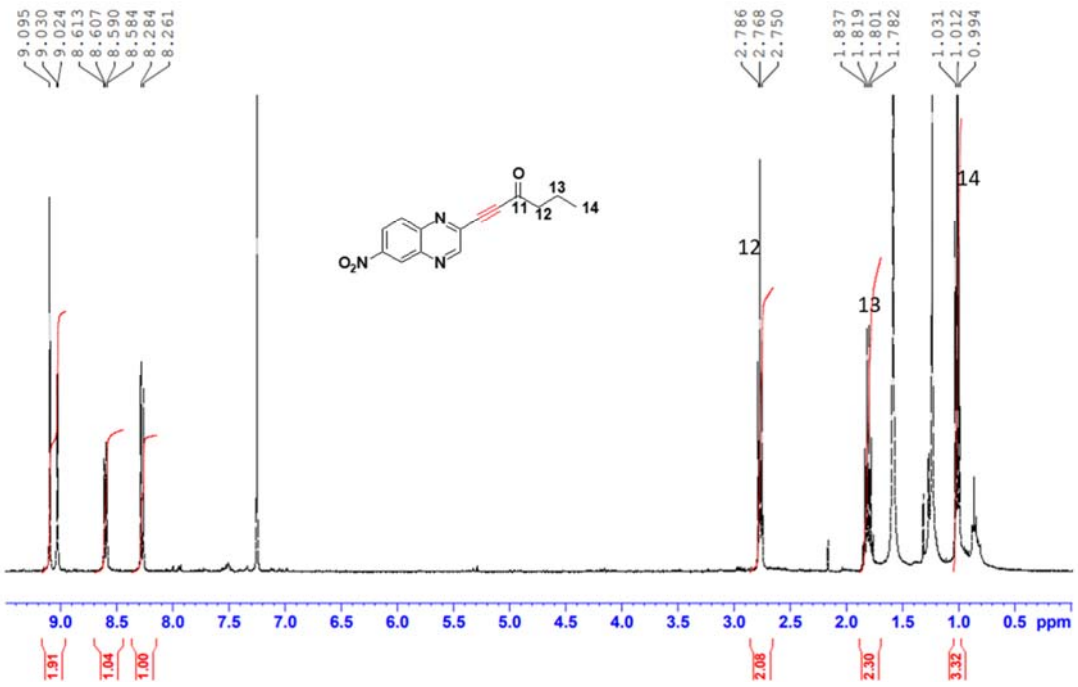


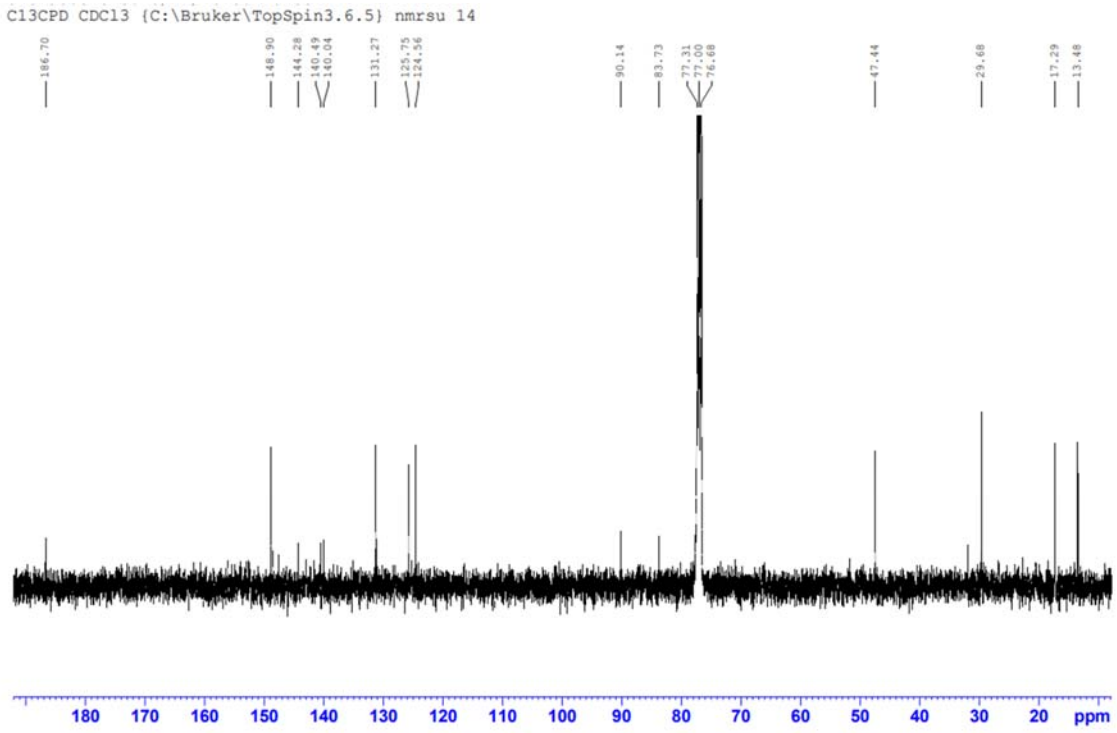




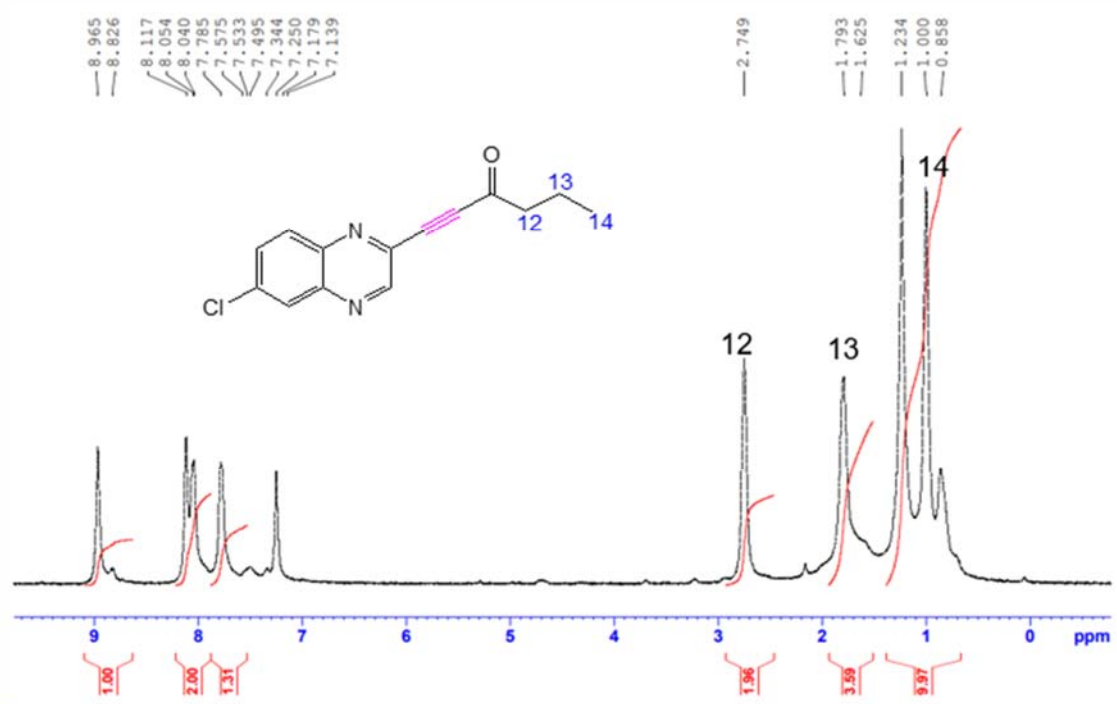
5.2.oxidation

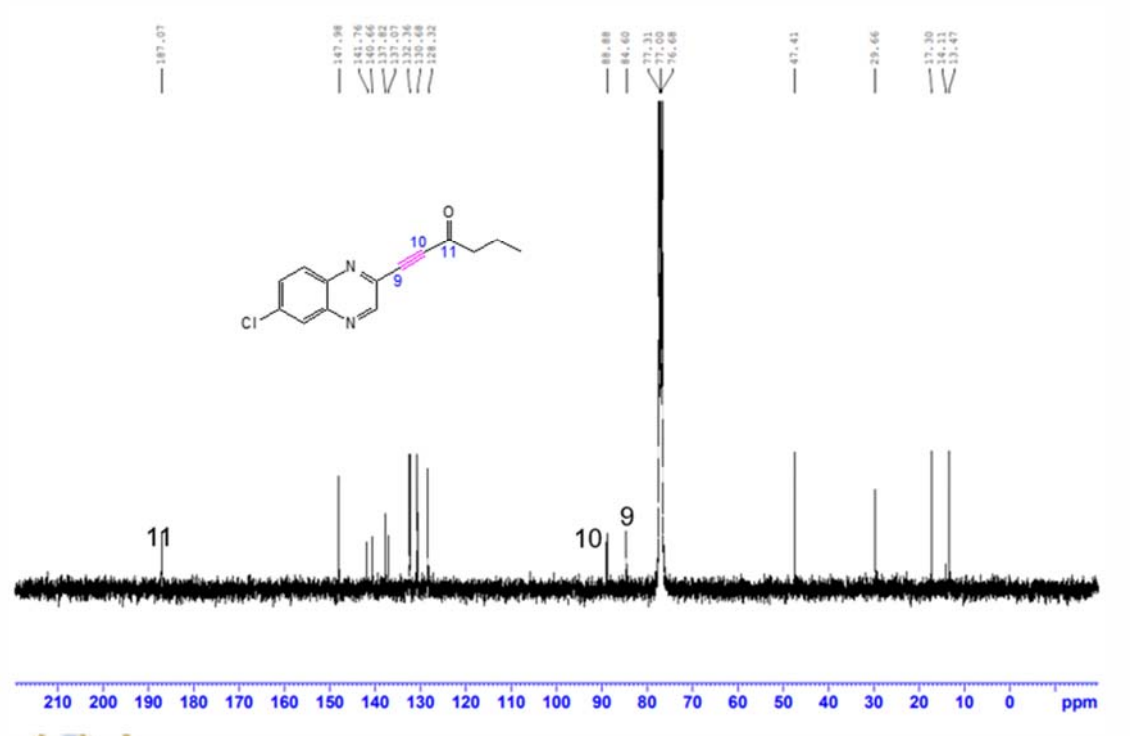
5.2.1. 1-(6-nitroquinoxalin-2-yl) hex-1-yn-3-one (1)

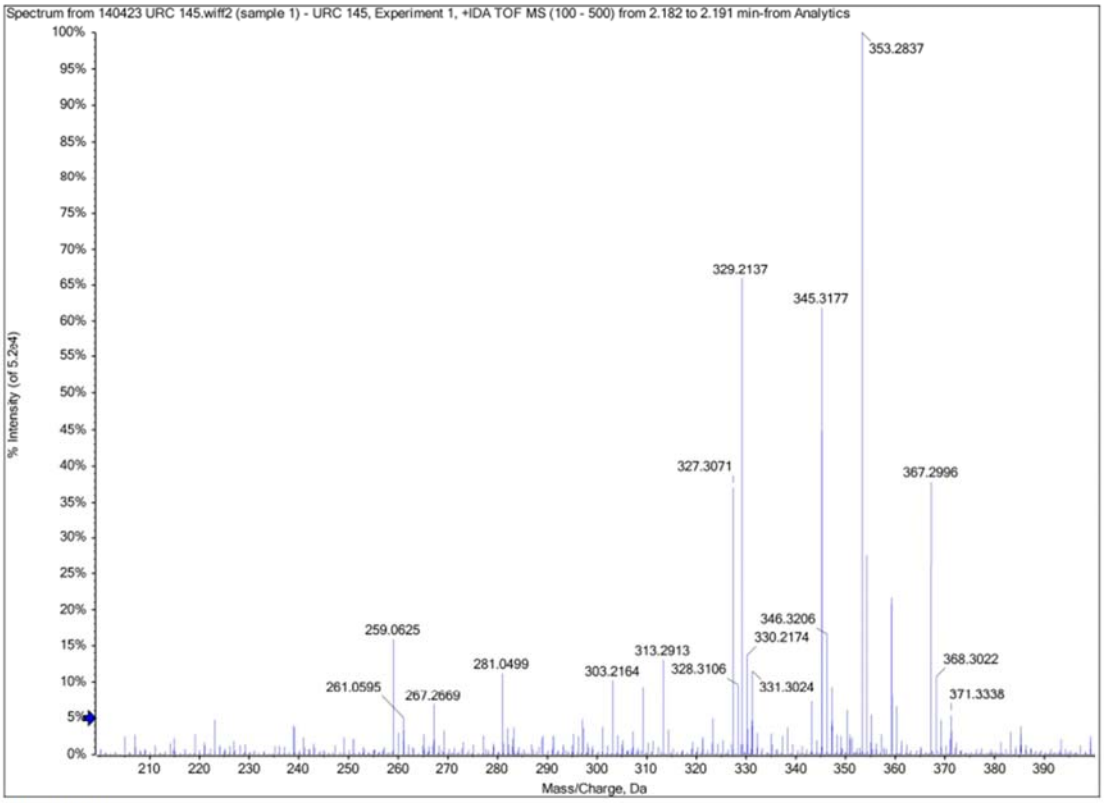




5.2.2. 1-(6-chloroquinoxalin-2-yl) hex-1-yn-3-one (7)

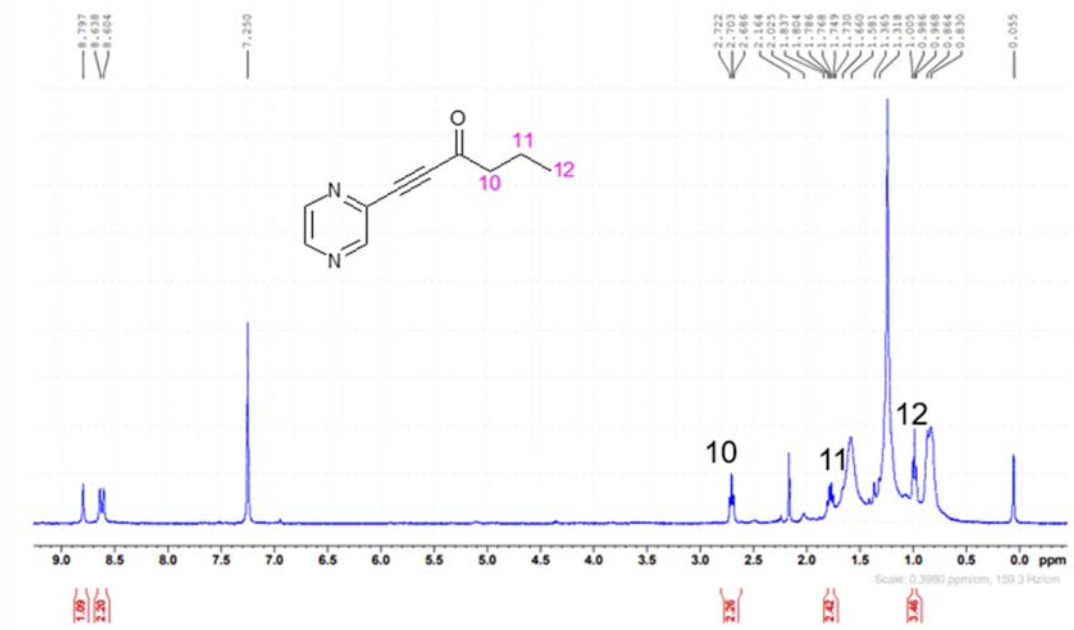


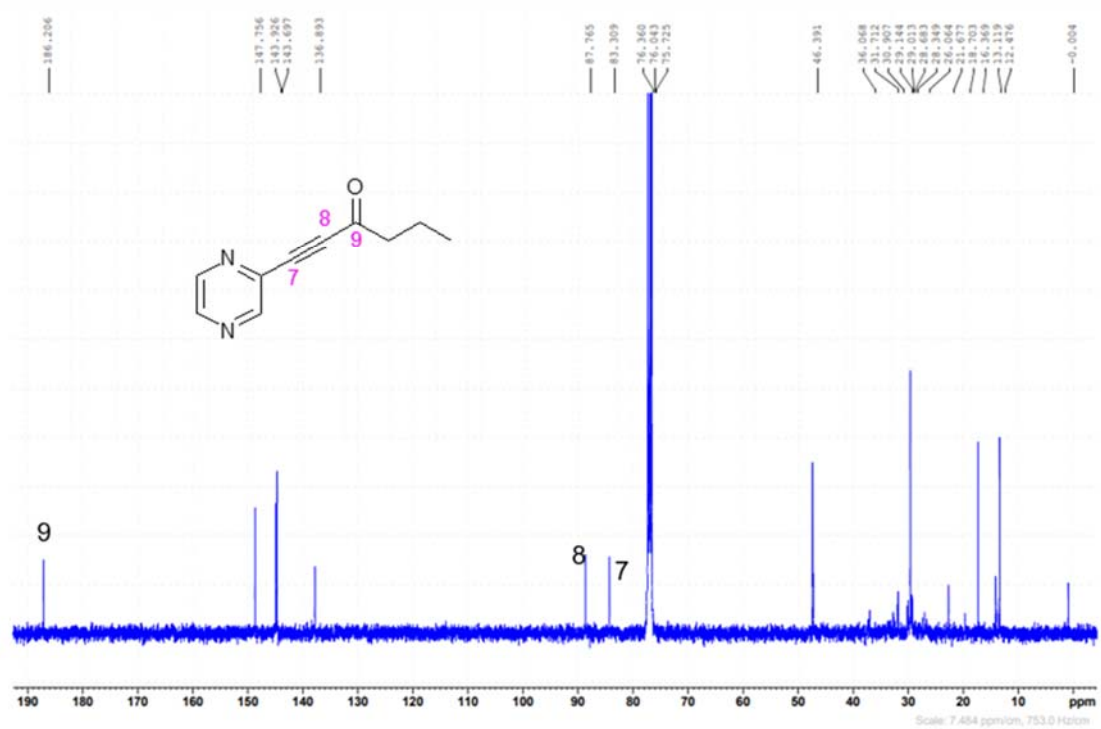


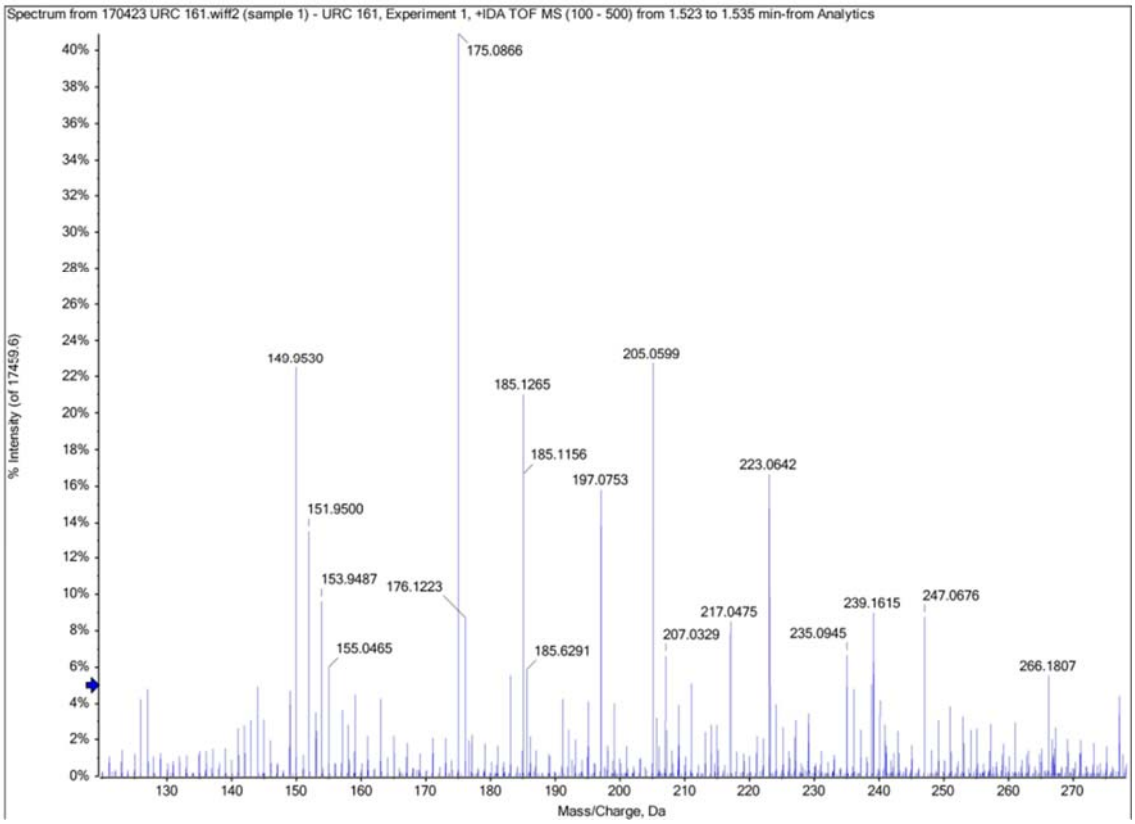


18/4/2023 3:52:16 PM

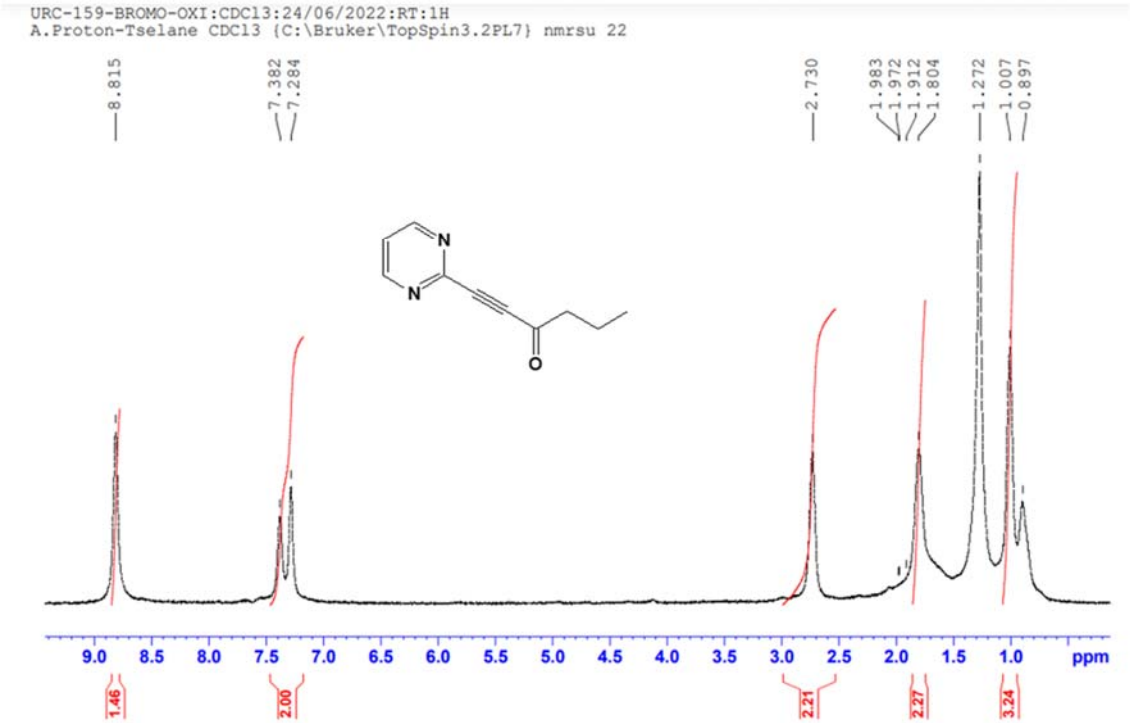
5.2.3. 1-(pyrazin-2-yl) hex-1-yn-3-one (11)

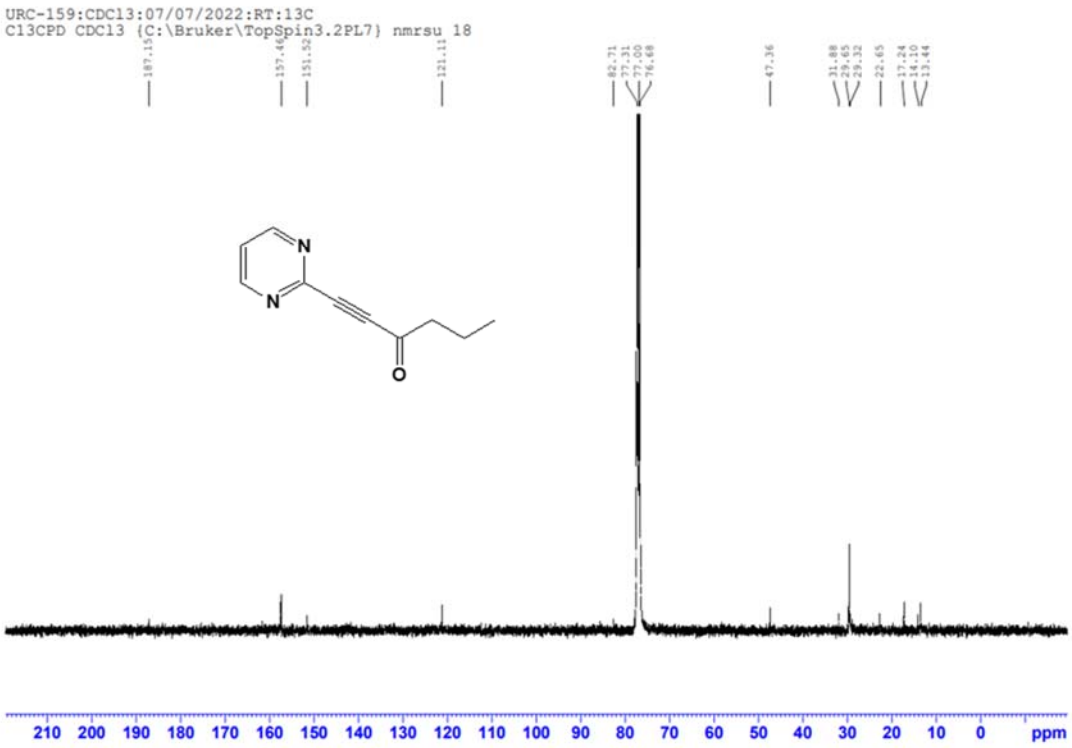


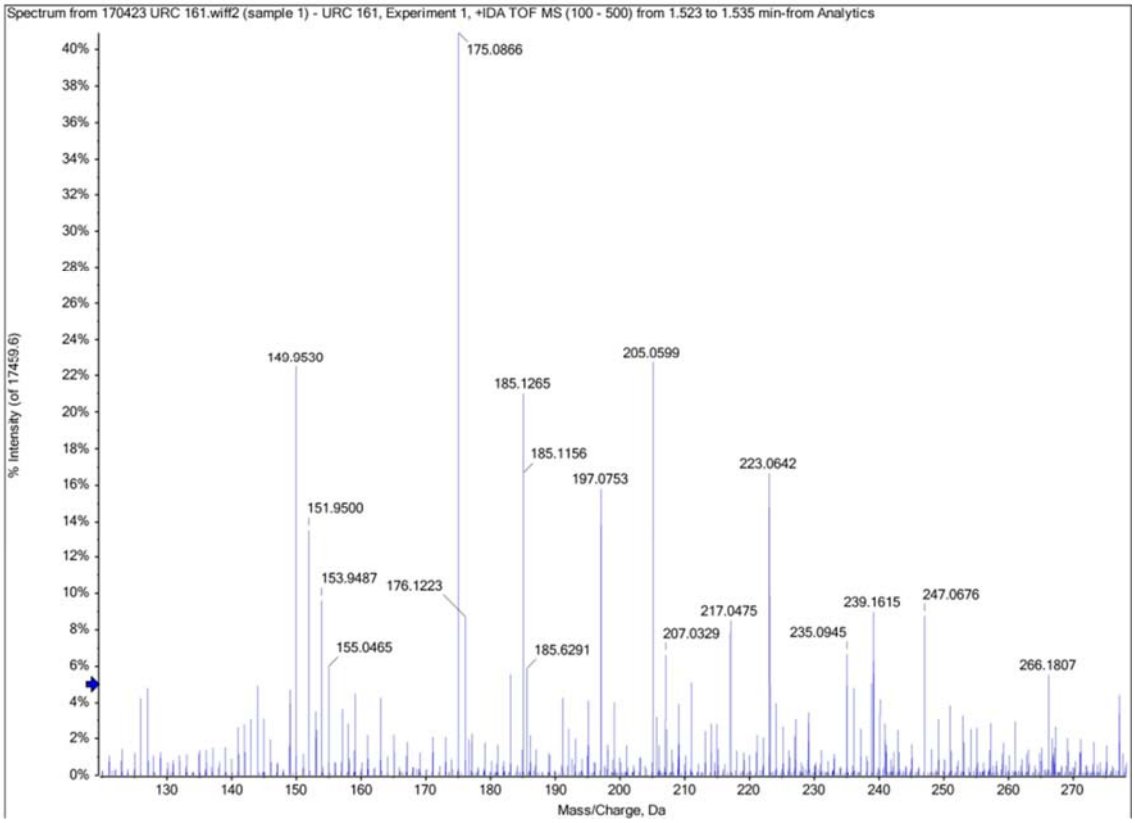




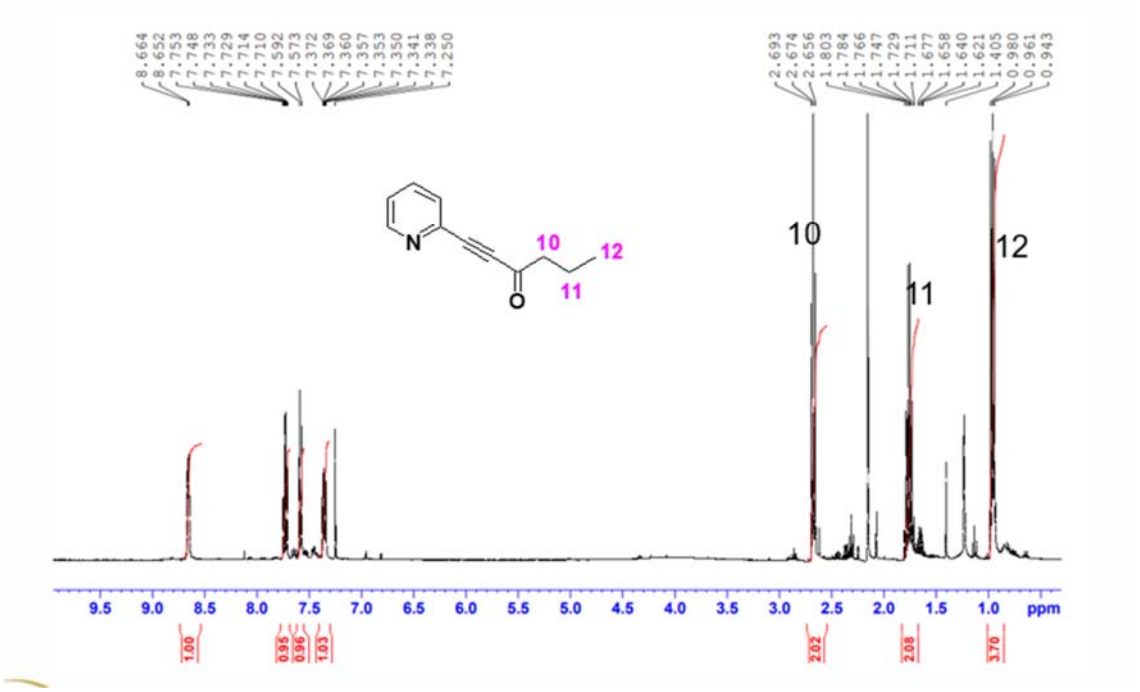
5.2.4. 1-(pyrimidine-2-yl) hex-1-yn-3-one (9)

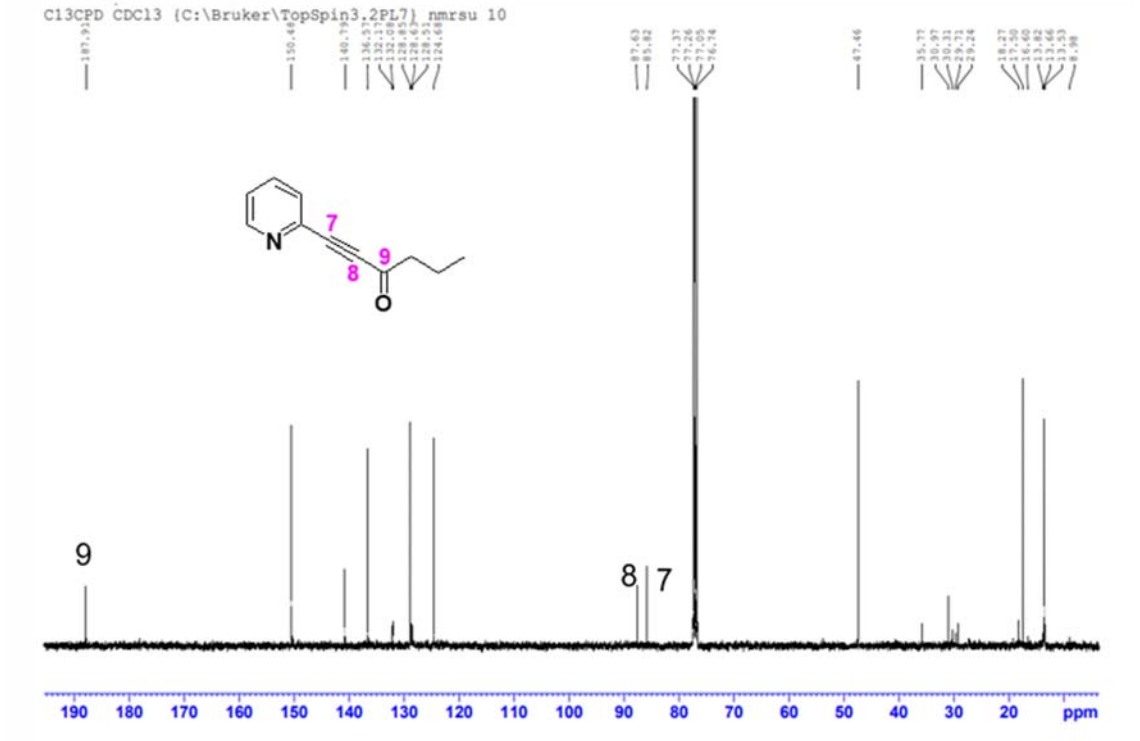


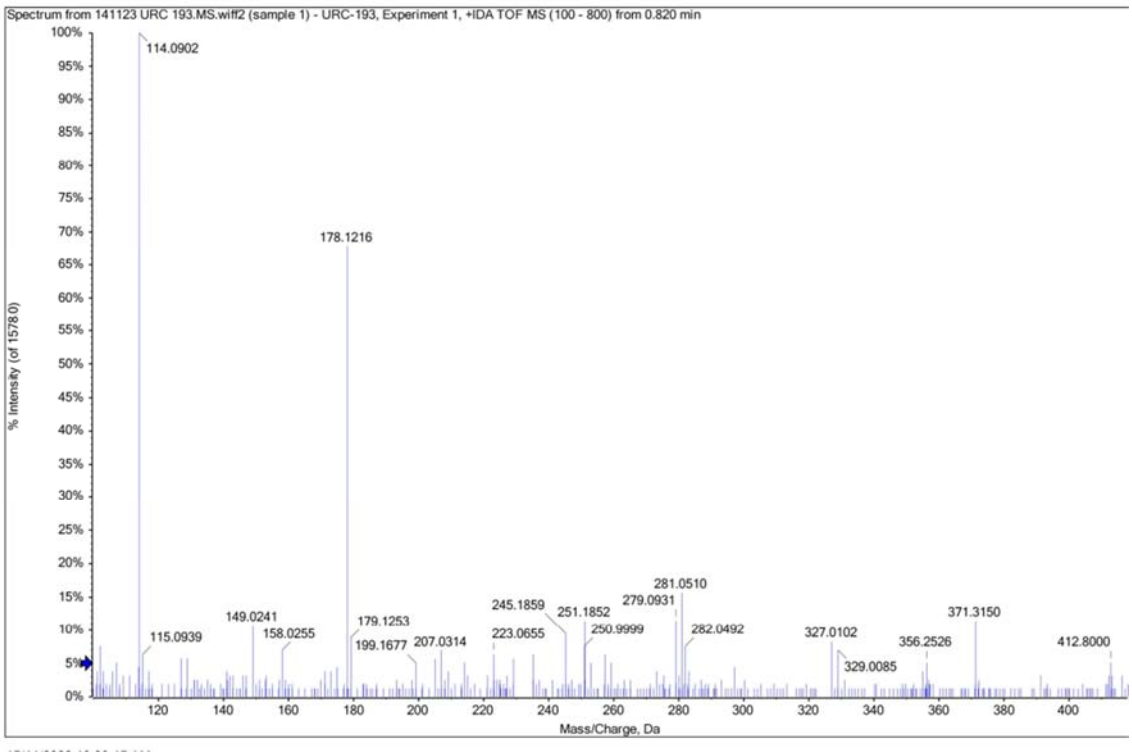

URC-159:CDCl3:07/07/2022:RT:13C
C13CPD CDCl3 {C:\Bruker\TopSpin3.2PL7} nmrsu 18
187.15
157.44
151.52
121.11
82.71
77.31
77.00
76.68
47.36
31.88
29.65
29.32
22.65
17.24
14.10
13.44
N
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Spectrum from 170423 URC 161.wiff2 (sample 1) - URC 161, Experiment 1, +IDA TOF MS (100 - 500) from 1.523 to 1.535 min-from Analytics
% Intensity (of 17459.6)
Mass/Charge, Da
175.0866
140.9530
205.0599
185.1265
185.1156
197.0753
223.0642
151.9500
153.9487
176.1223
217.0475
239.1615
247.0676
207.0329
235.0945
155.0465
185.6291
266.1807

5.2.5. 1-(pyridine-2-yl) hex-1-yn-3-one (9)

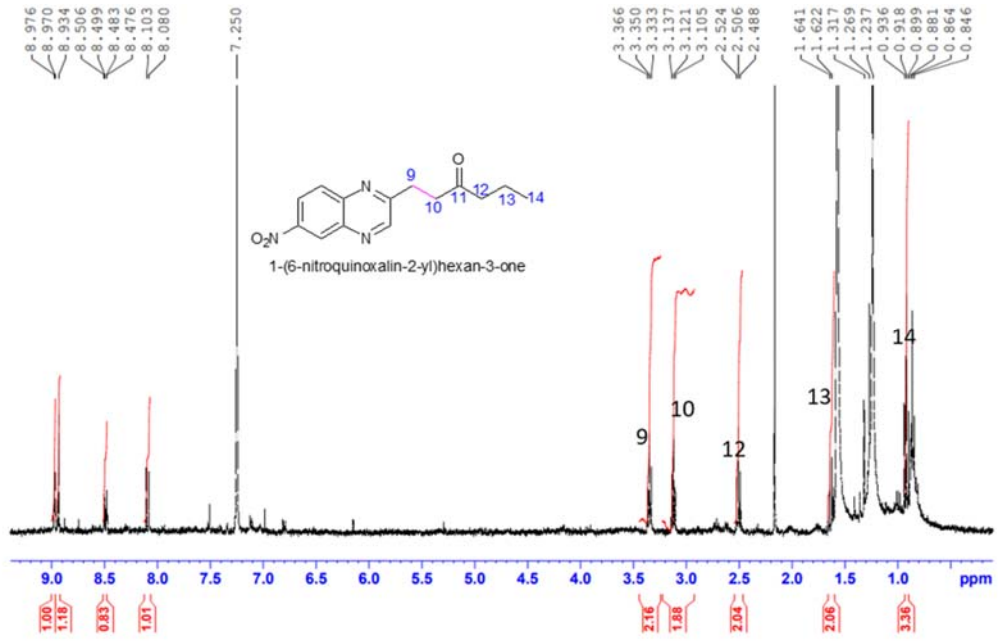


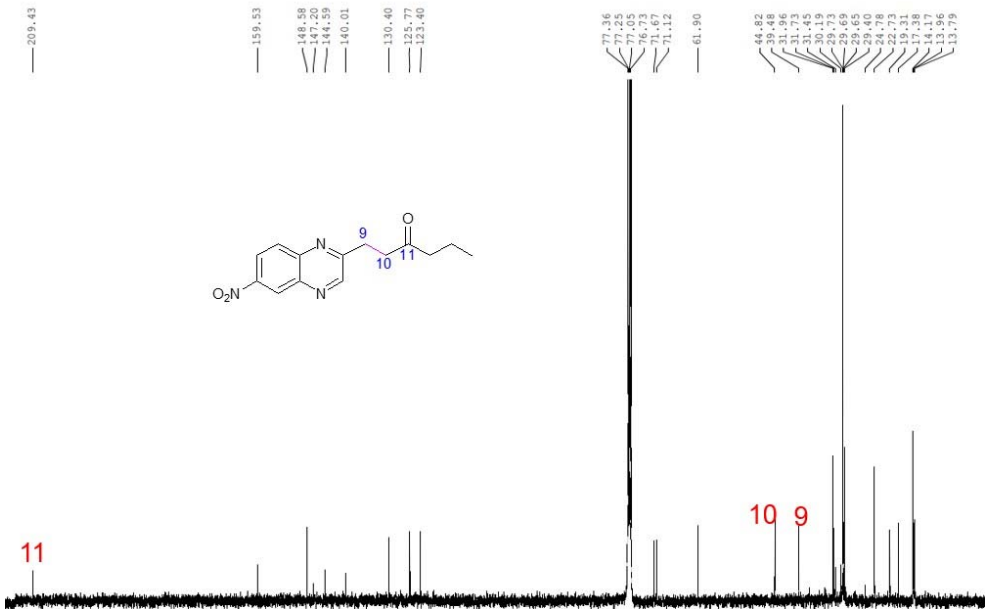


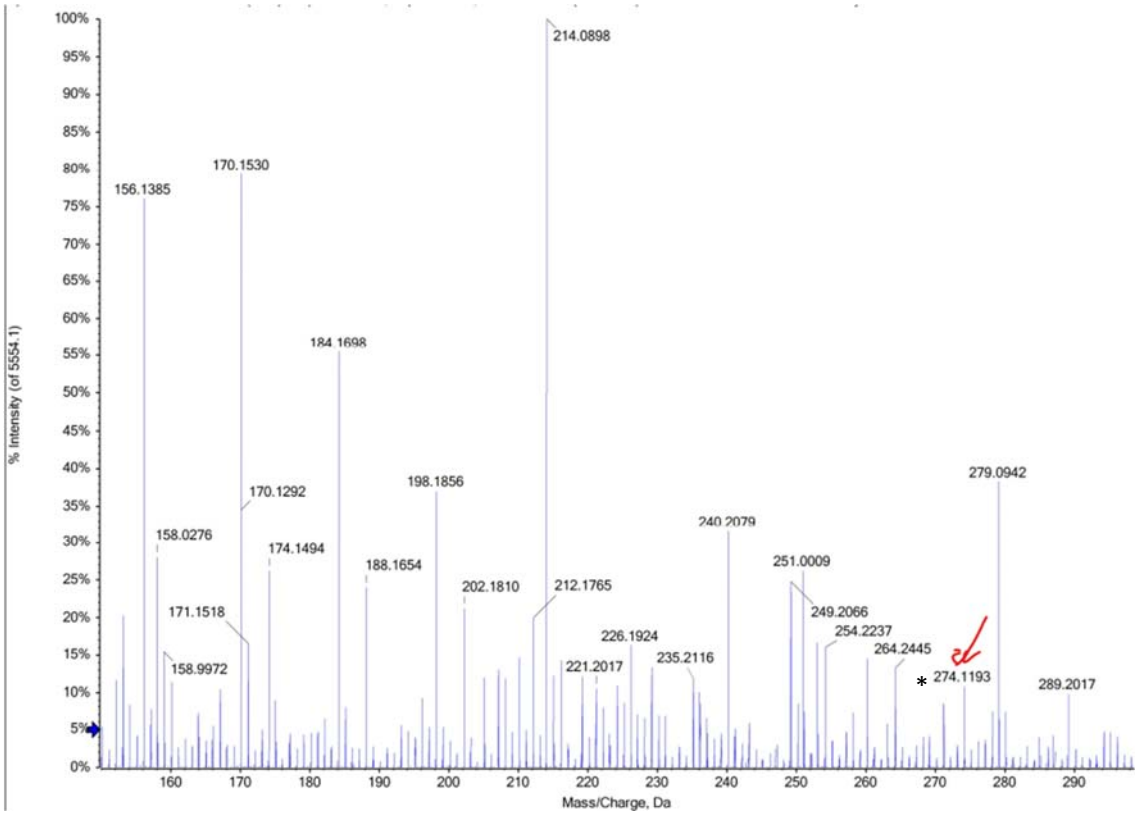


5.3. Reduction

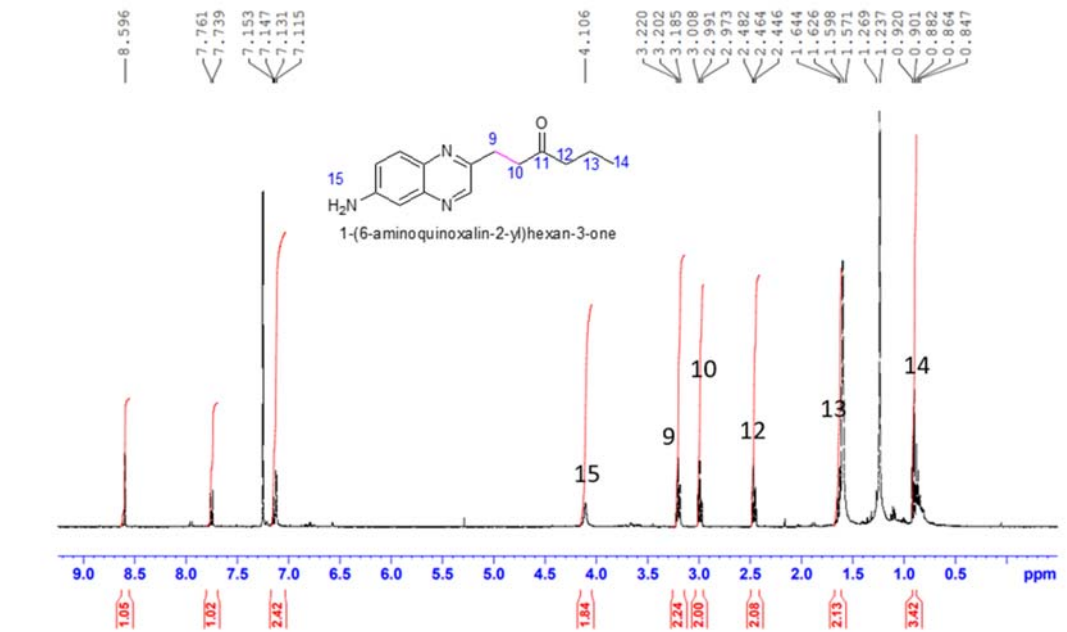
5.3.11-(6- nitroquinoxalin-2-yl) hexan-3-one (6)

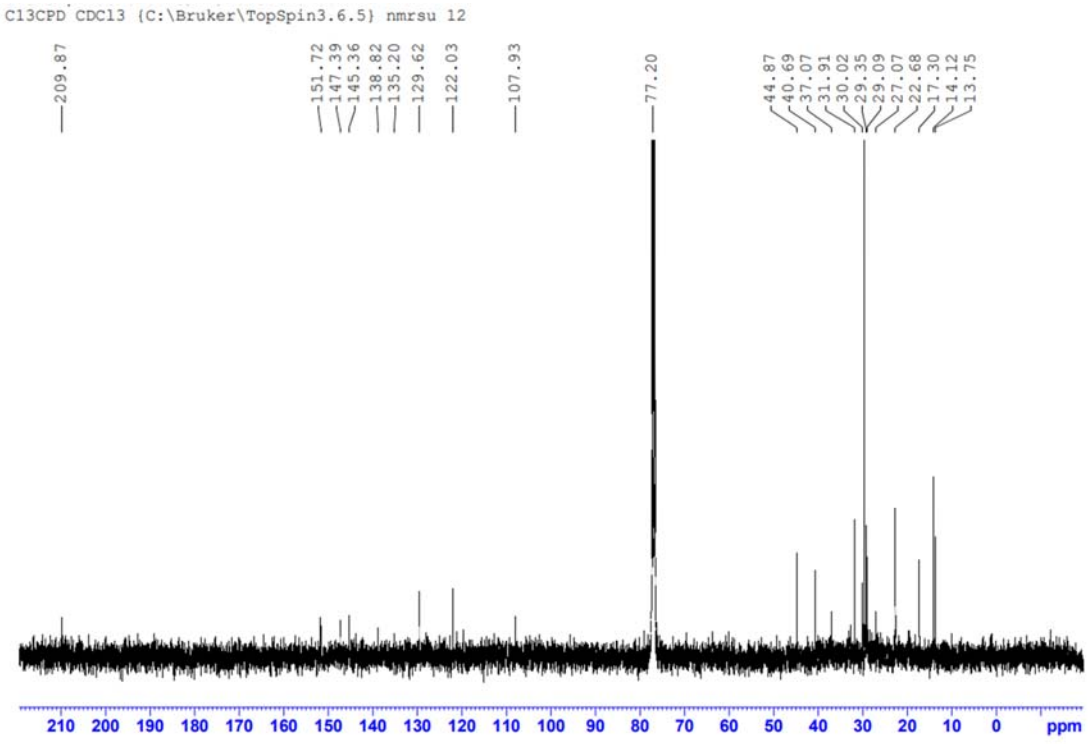


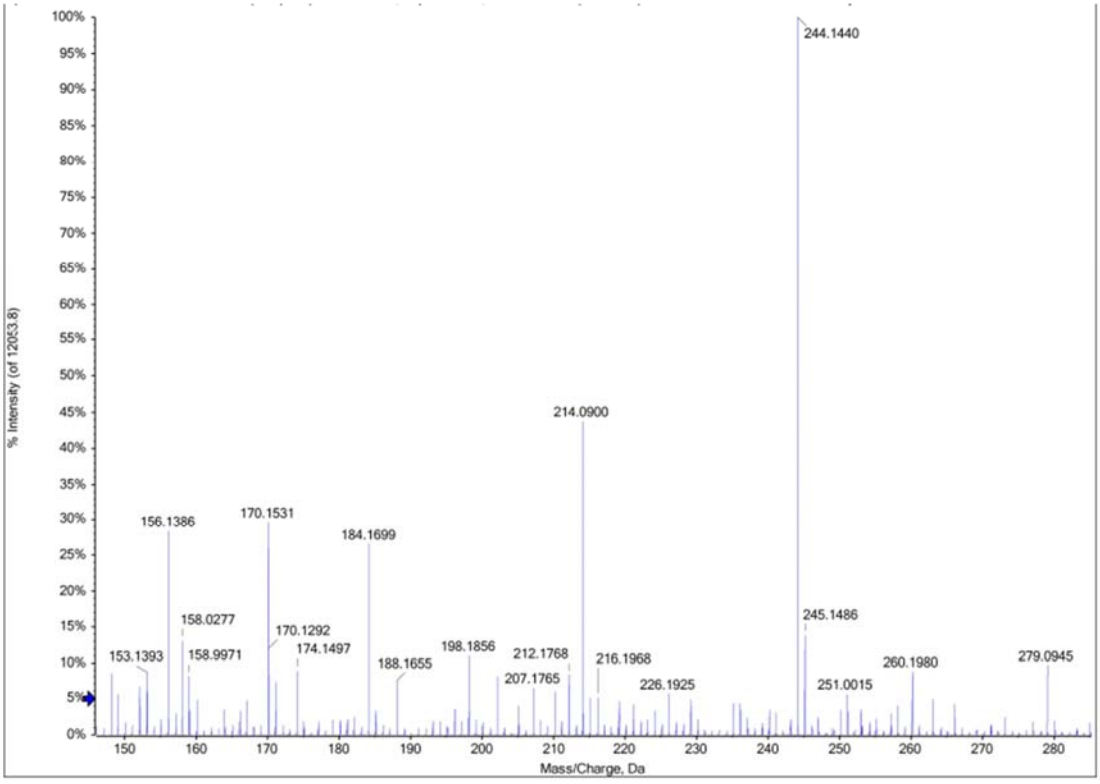

209.43
159.53
148.58
147.20
144.59
140.01
130.40
125.77
123.40
77.36
77.25
77.05
76.73
71.67
71.12
61.90
44.82
39.48
31.96
31.73
31.45
30.13
29.73
29.69
29.65
29.40
24.78
22.73
19.31
17.38
14.17
13.96
13.79
O
N
9
10
11
O2N
N
11
10
9


% Intensity (of 5554.1)
100%
95%
90%
85%
80%
75%
70%
65%
60%
55%
50%
45%
40%
35%
30%
25%
20%
15%
10%
5%
0%
214.0898
170.1530
156.1385
184.1698
170.1292
198.1856
158.0276
174.1494
188.1654
202.1810
212.1765
240.2079
279.0942
251.0009
171.1518
249.2066
254.2237
226.1924
264.2445
158.9972
221.2017
235.2116
274.1193
289.2017
160
170
180
190
200
210
220
230
240
250
260
270
280
290
Mass/Charge, Da

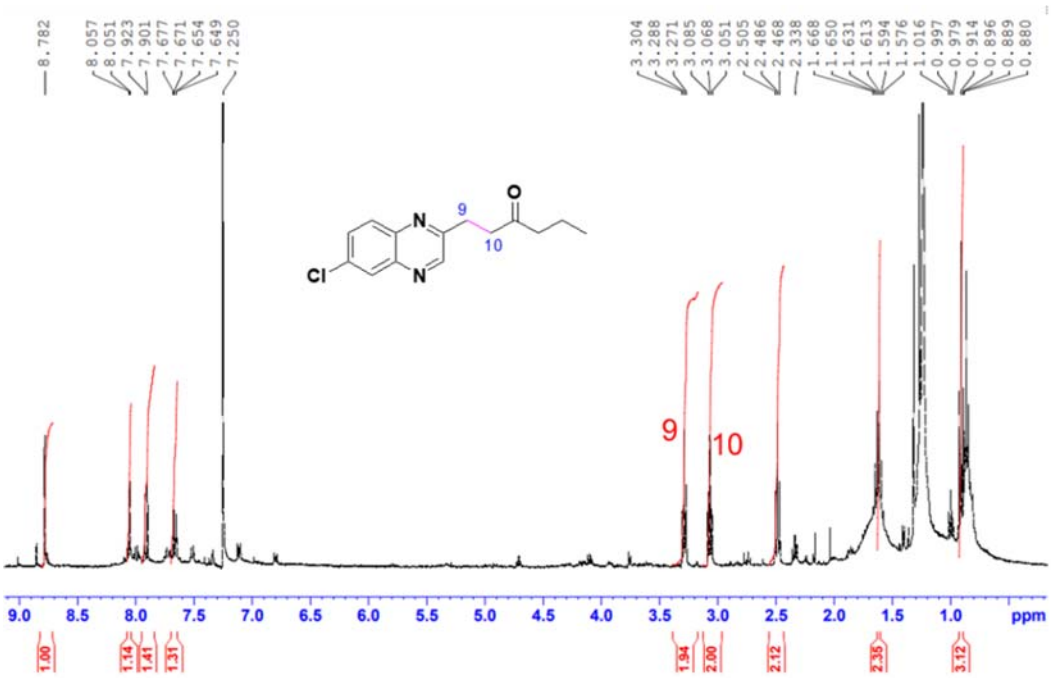
5.3.2. 1-(6- aminoquinoxalin-2-yl) hexan-3-one (3)

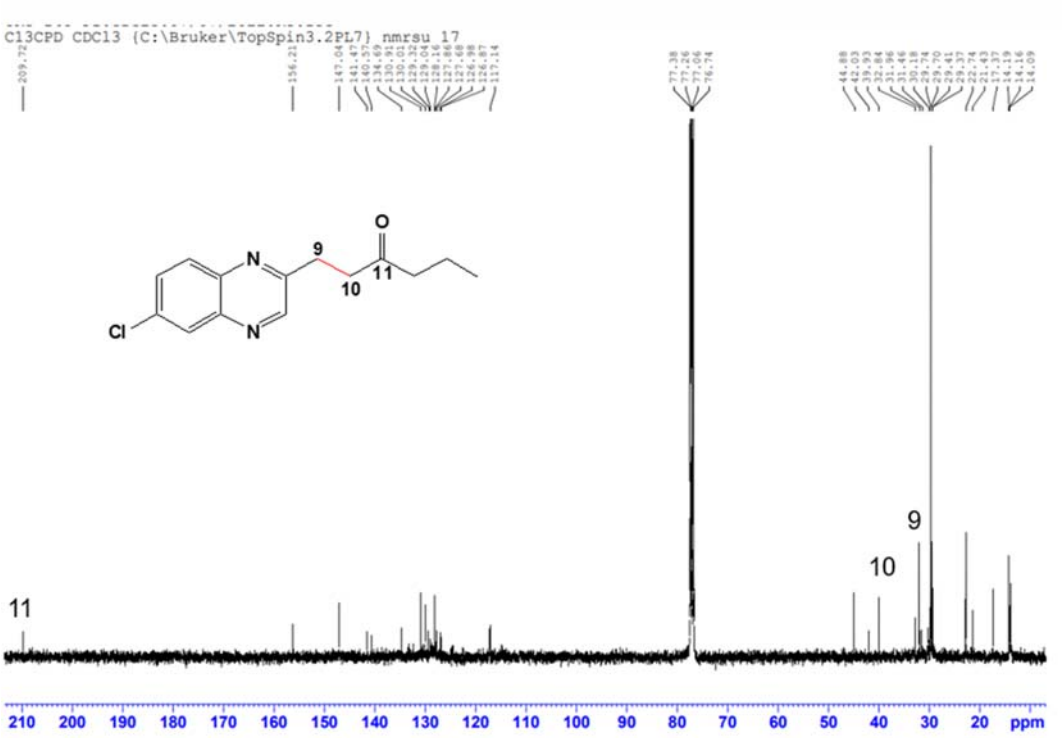


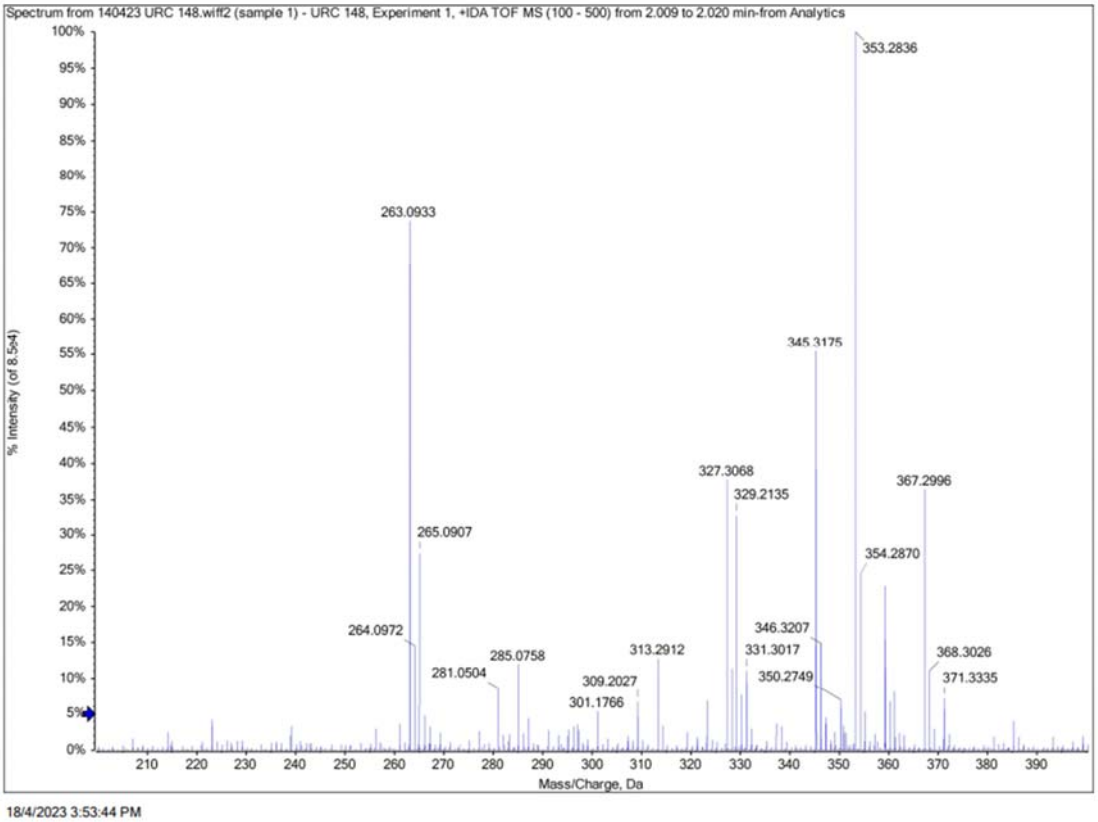




5.3.3. 1-(6-chloroquinoxalin-2-yl)hexan-3-one (8)

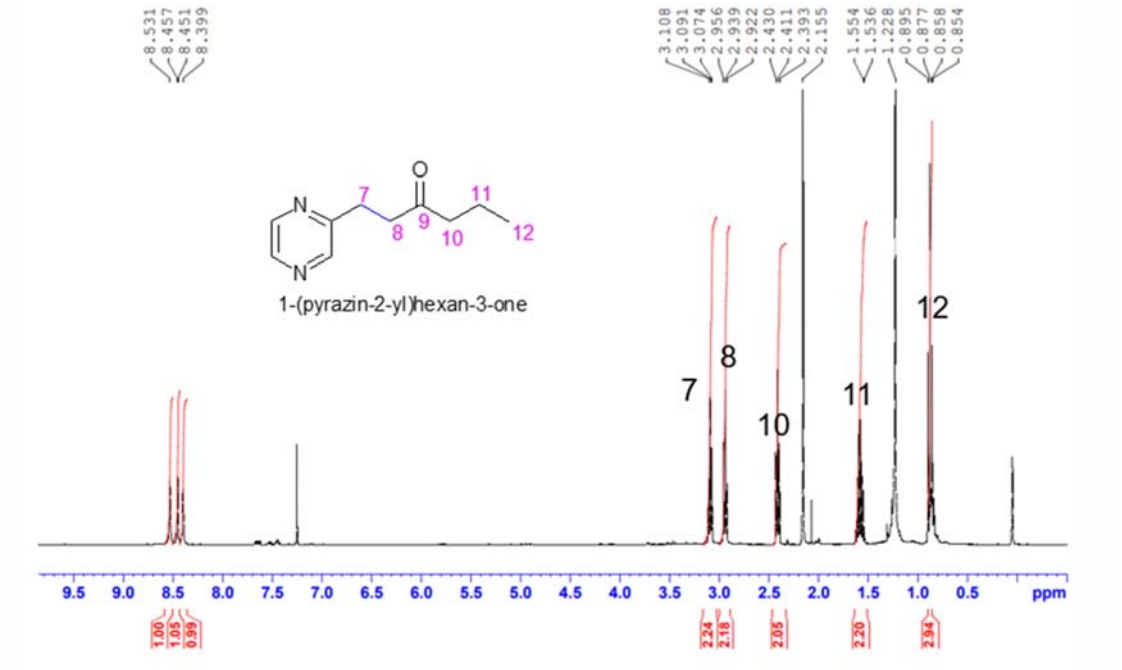


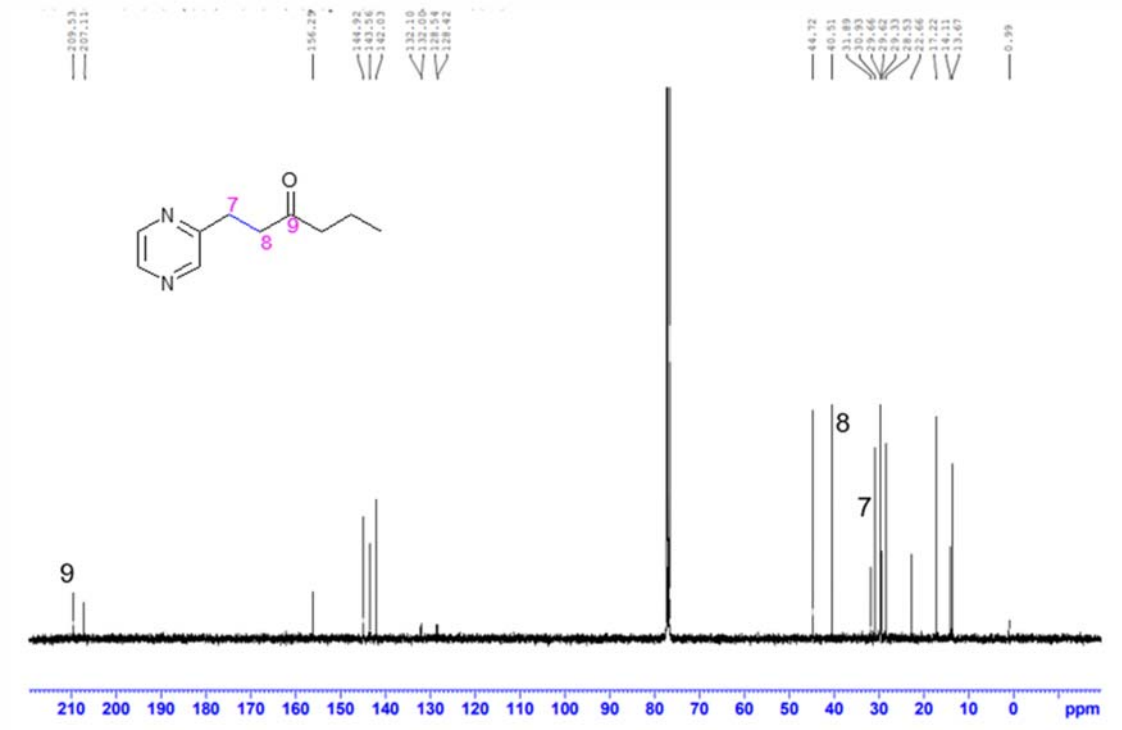


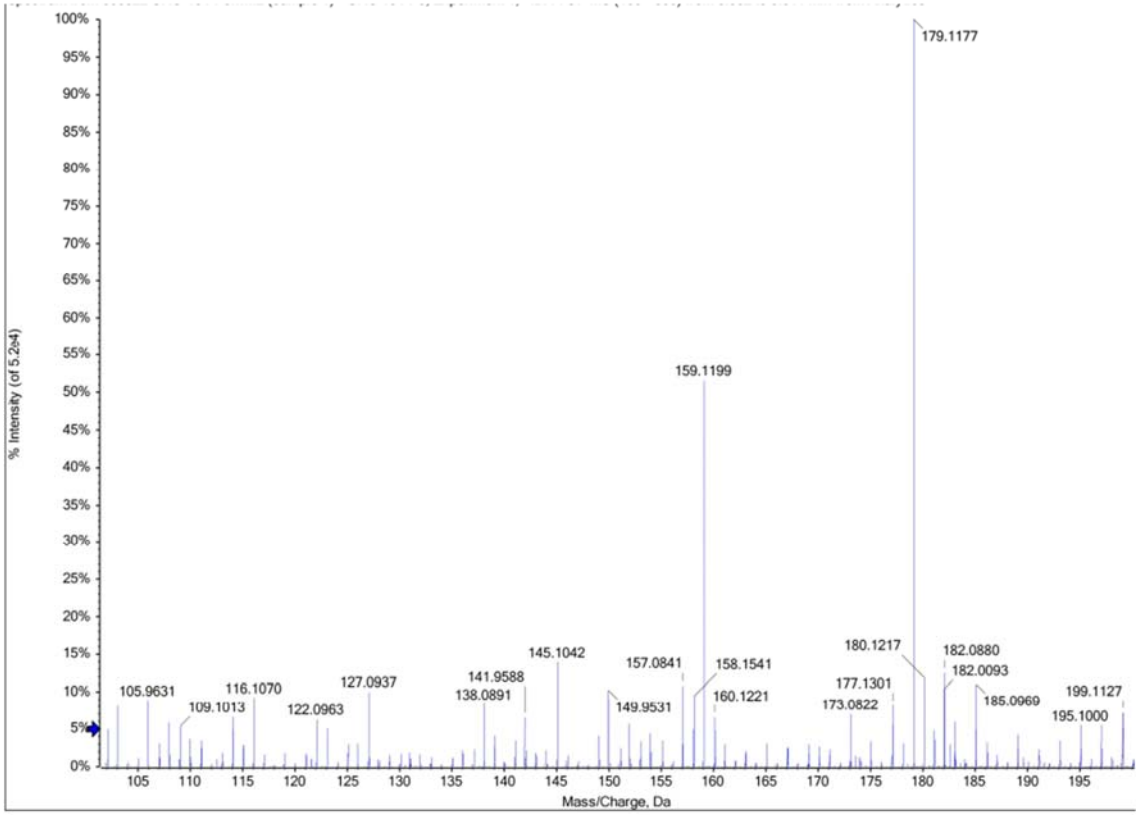


18/4/2023 3:53:44 PM

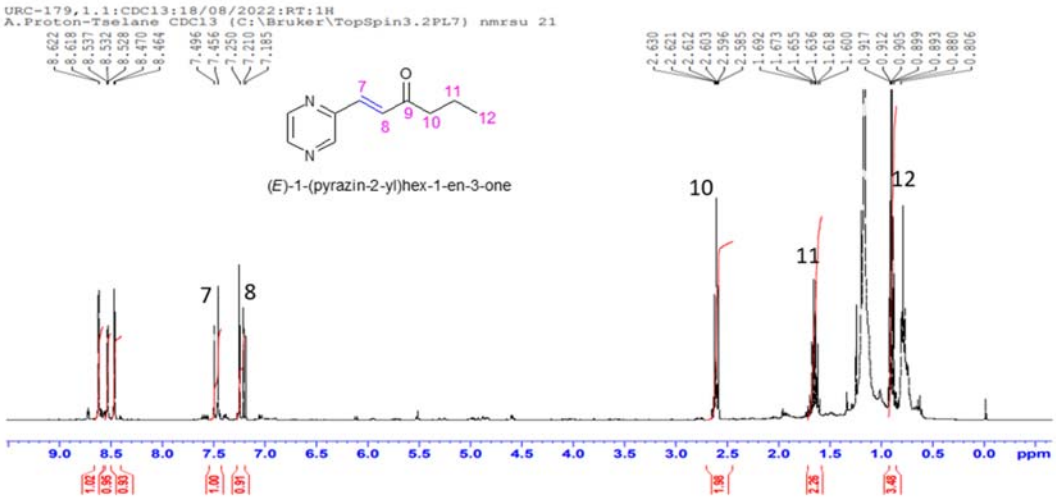
5.3.4. 1-(pyrazin-2-yl) hexane-3-one (13)

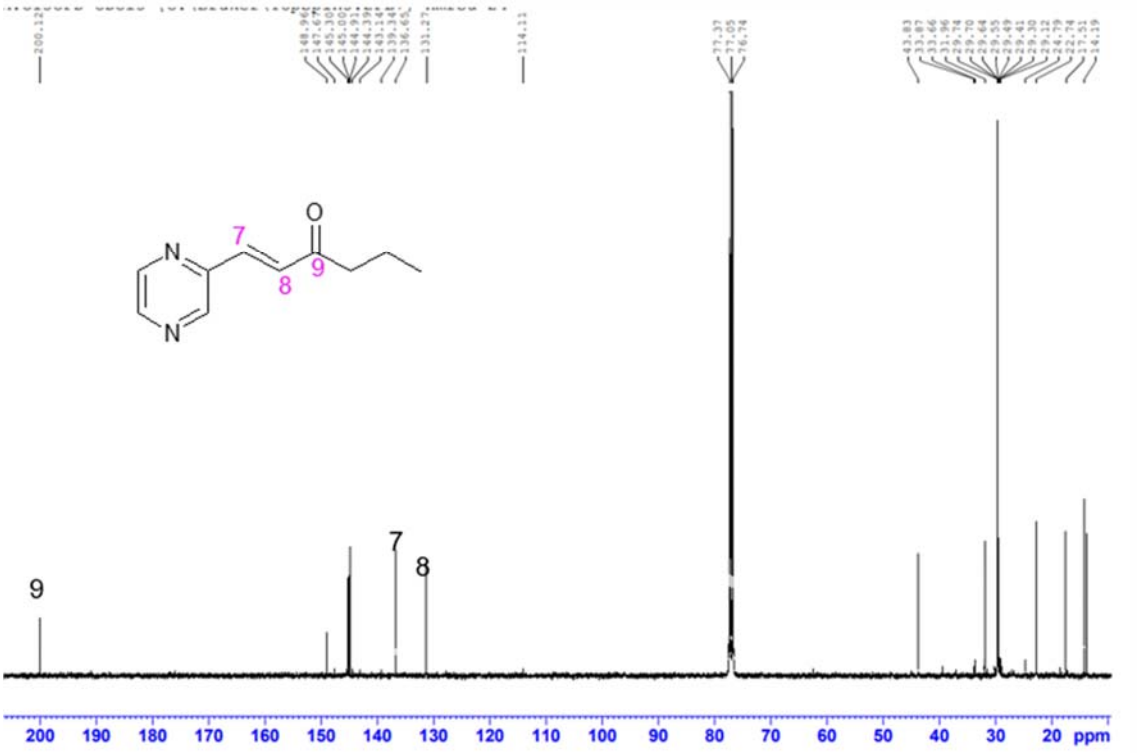


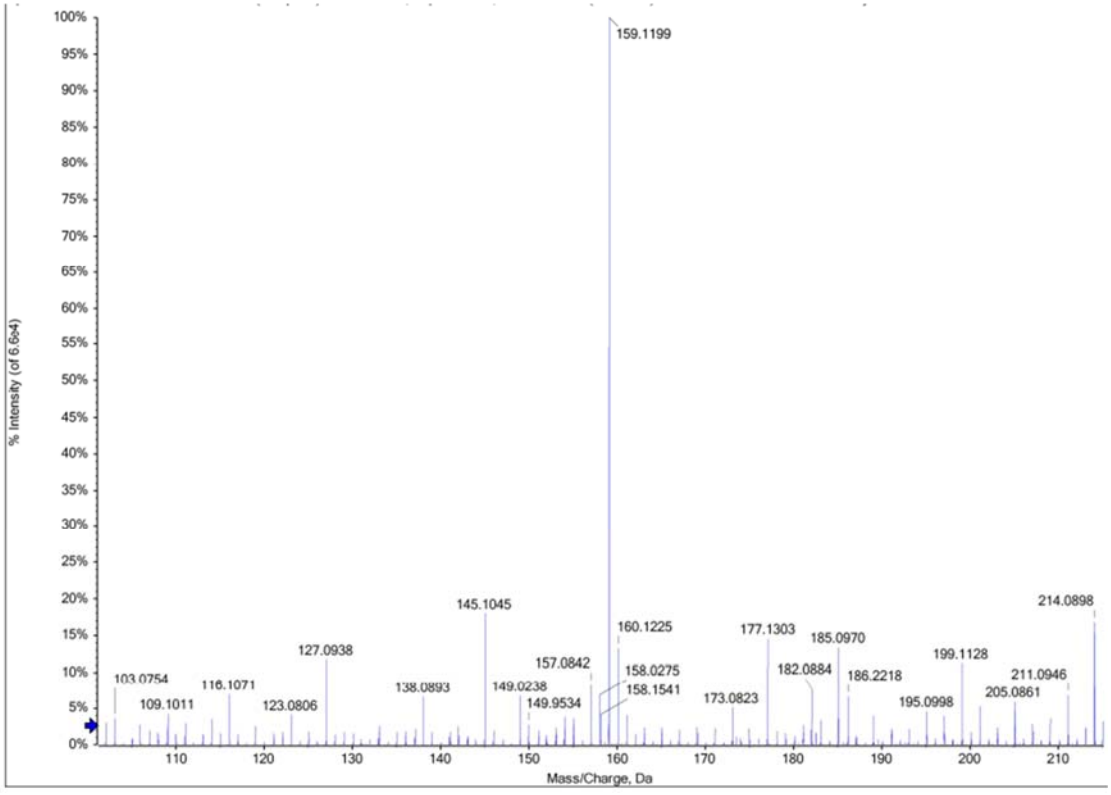




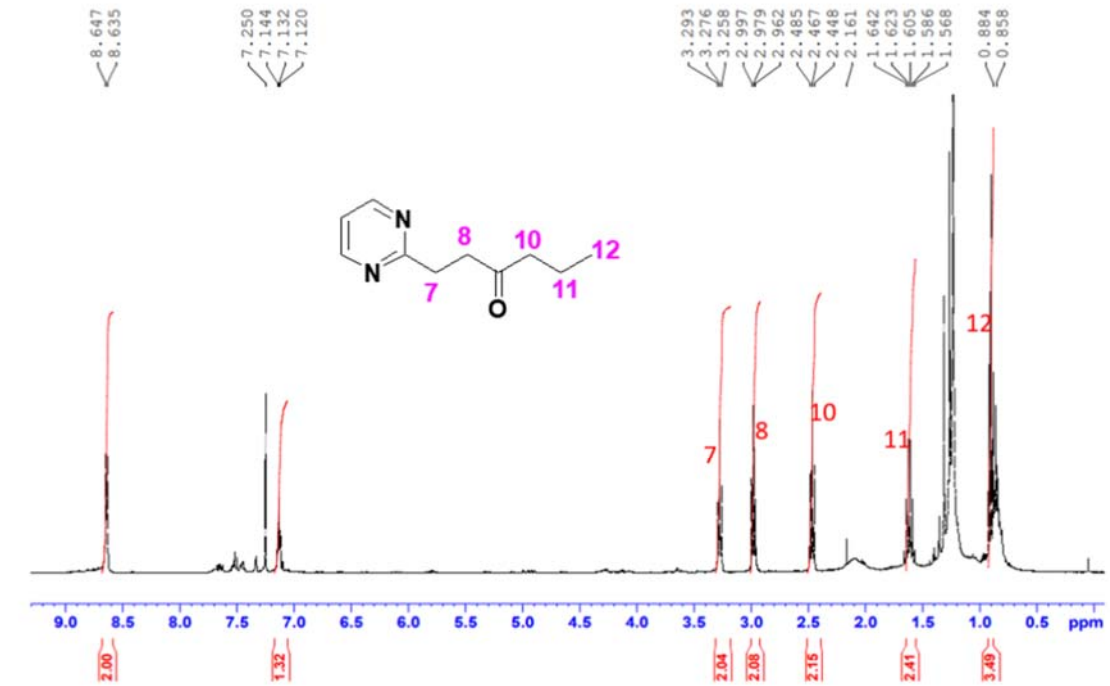
5.3.4. (E)-1-(pyrazin-2-yl) hex-1-en-3-one (12)

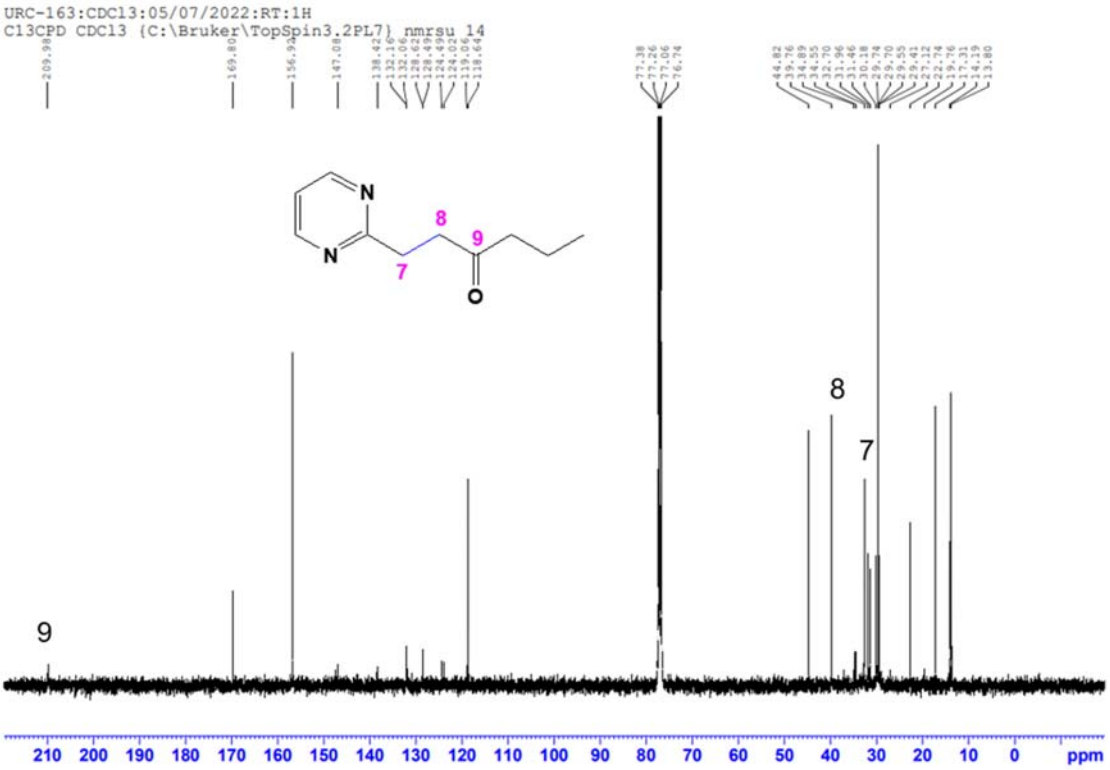


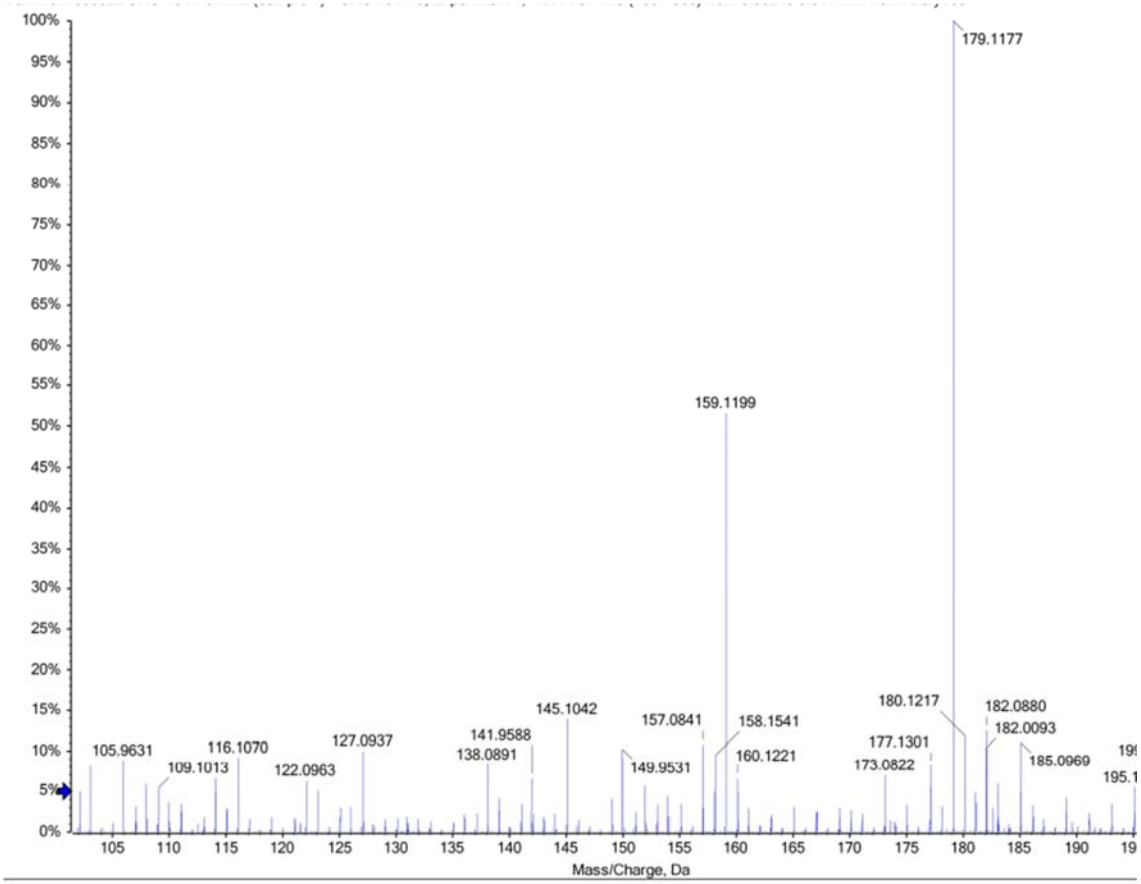

O
7
8
9
N
N
9
7
8
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


% Intensity (of 6.6e4)
Mass/Charge, Da
159.1199
145.1045
160.1225
177.1303
214.0898
185.0970
127.0938
157.0842
158.0275
182.0884
199.1128
103.0754
116.1071
138.0893
149.0238
158.1541
186.2218
211.0946
109.1011
123.0806
149.9534
173.0823
195.0998
205.0861

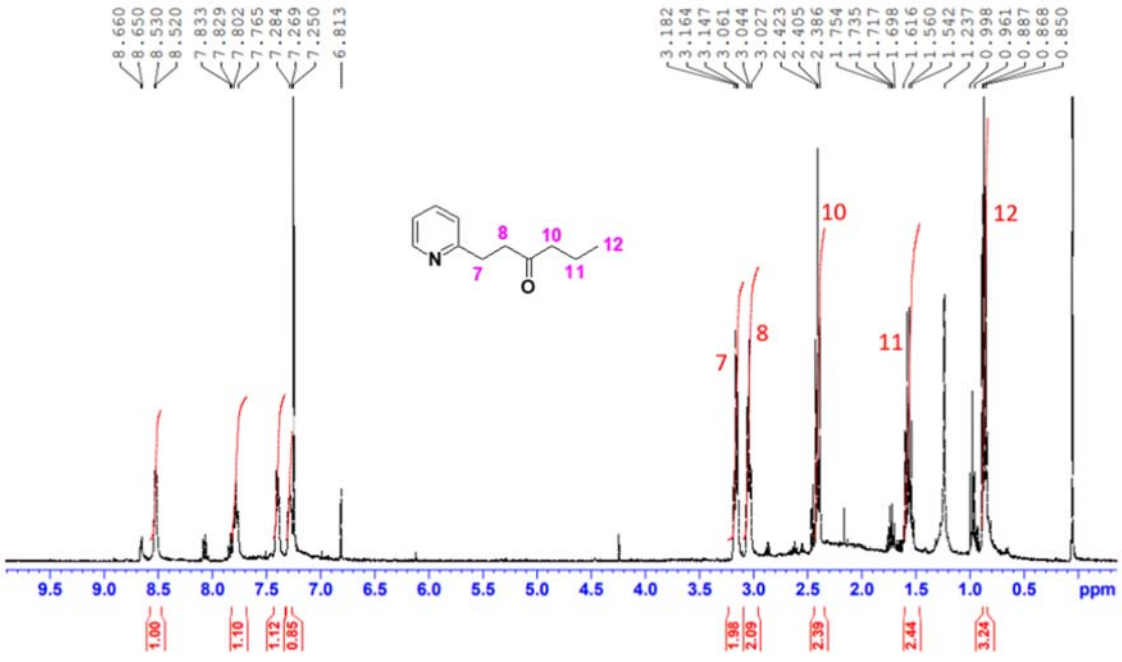
5.3.5. 1-(pyrimidine-2-yl) hexane-3-one (10)

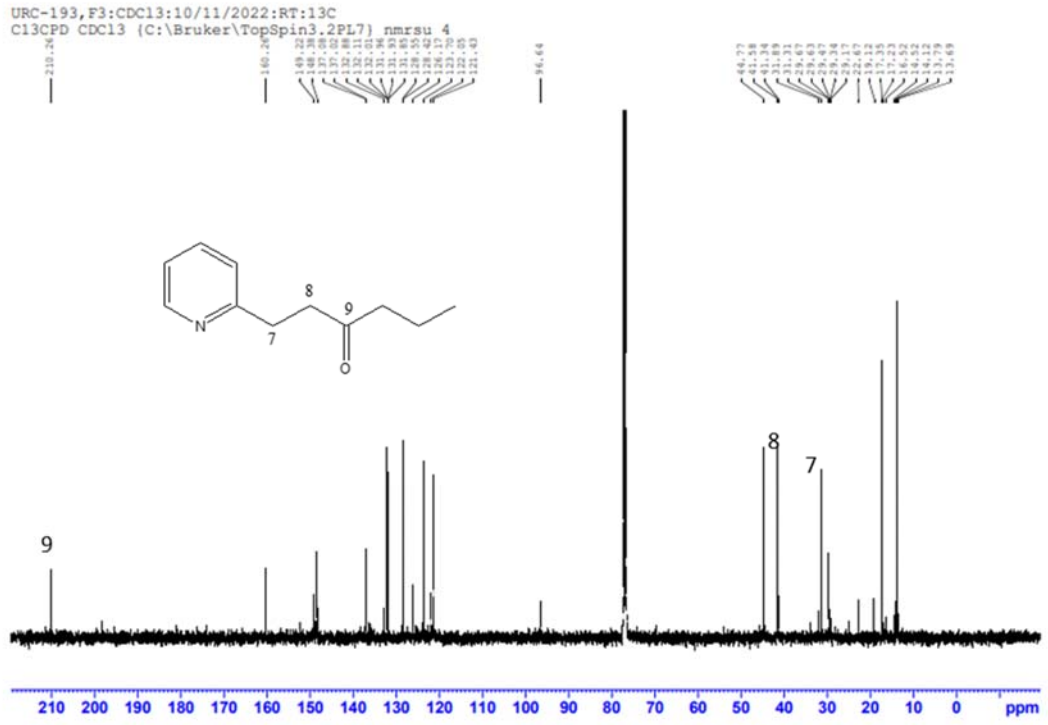


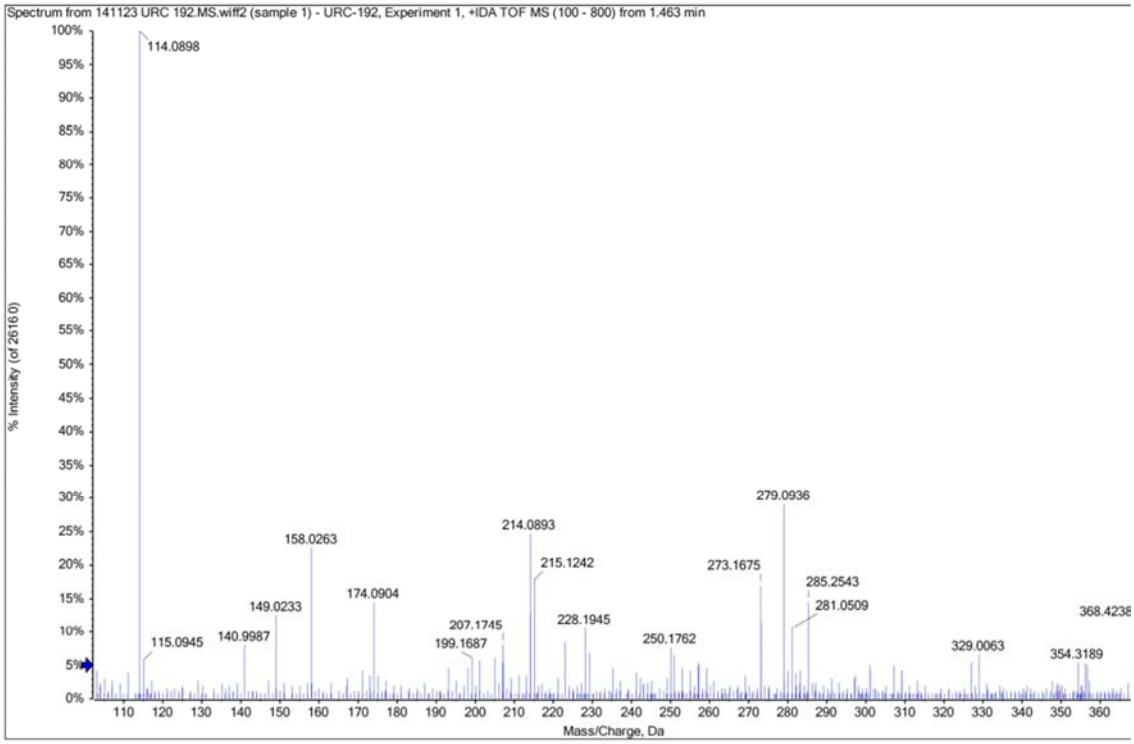


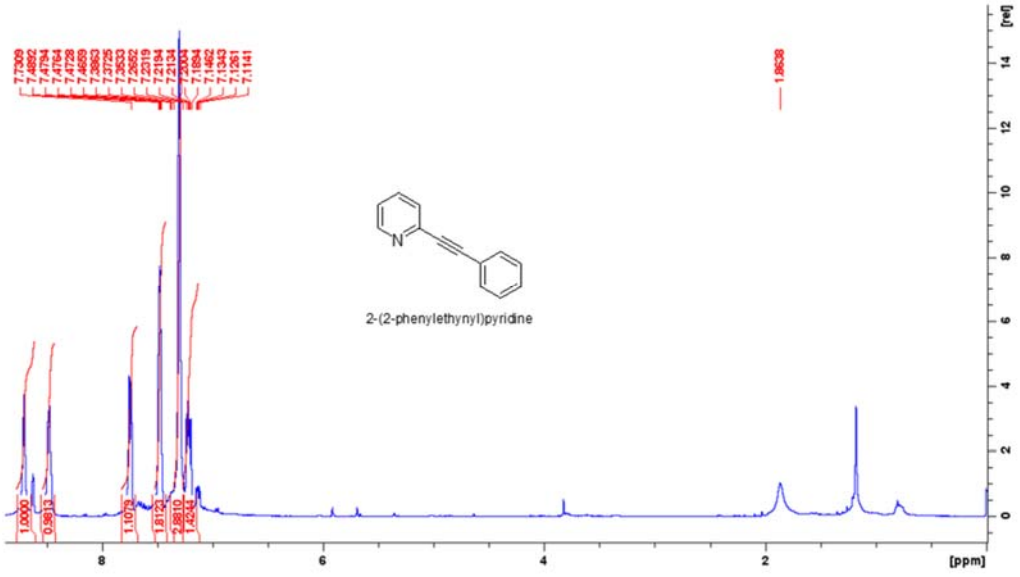


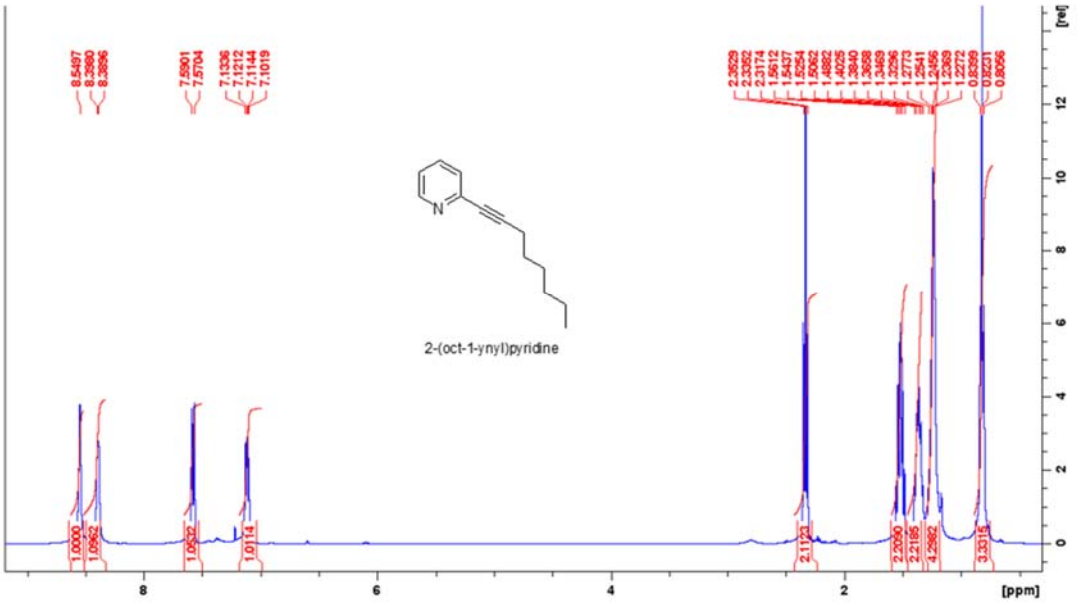
5.3.6. Synthesis of 1-(pyridine-2-yl) hexane-3-one (15)

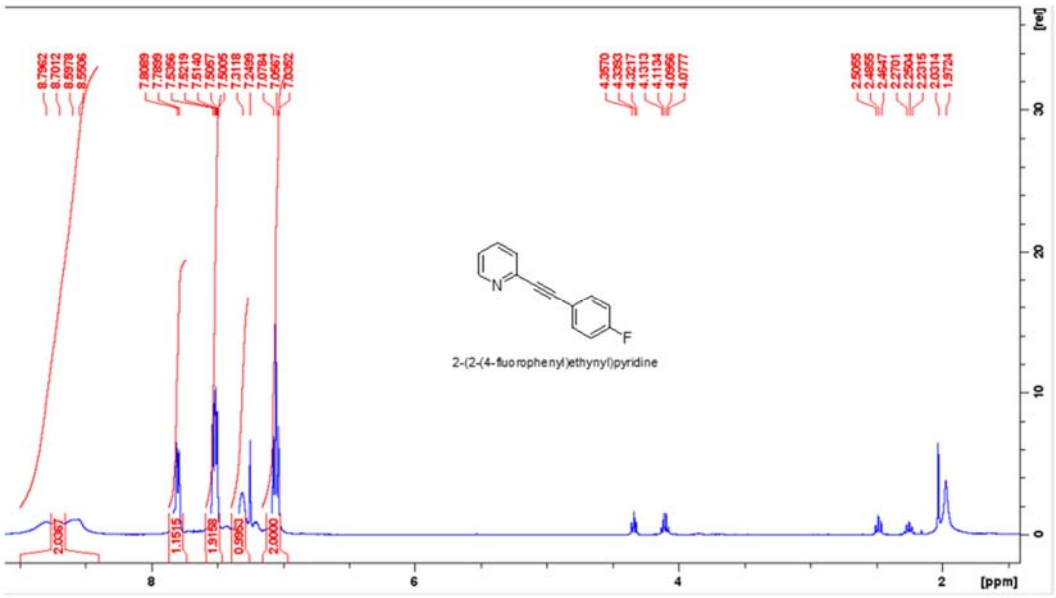


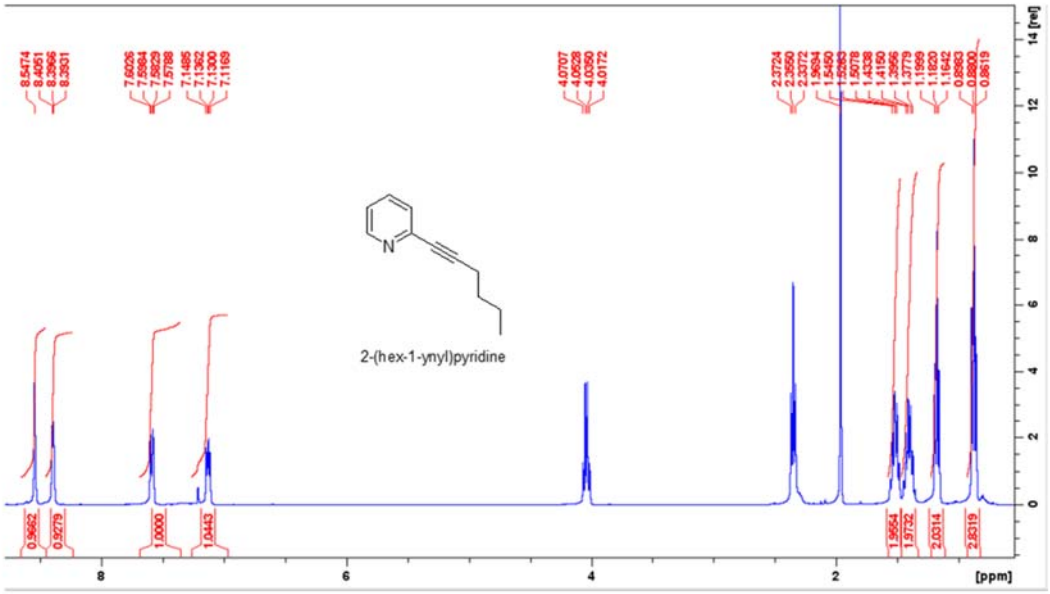

URC-193,F3:CDCl3:10/11/2022:RT:13C
C13CPD CDCl3 (C:\Bruker\TopSpin3.2PL7) nmrsu 4
N
8
9
7
O
9
8
7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Spectrum from 141123 URC 192.MS.wiff2 (sample 1) - URC-192, Experiment 1, +IDA TOF MS (100 - 800) from 1.463 min
% Intensity (of 2616.0)
Mass/Charge, Da
114.0898
115.0945
140.9987
149.0233
158.0263
174.0904
199.1687
207.1745
214.0893
215.1242
228.1945
250.1762
273.1675
279.0936
281.0509
285.2543
329.0063
354.3189
368.4238


2-(2-phenylethynyl)pyridine
[ppm]


2-(oct-1-ynyl)pyridine
[ppm]


8.7962
8.7012
8.5978
8.5506
7.8089
7.7889
7.5366
7.5219
7.5140
7.5057
7.5005
7.3118
7.2499
7.0784
7.0667
7.0362
4.3570
4.3383
4.3217
4.1313
4.1134
4.0956
4.0777
2.5065
2.4865
2.4647
2.2701
2.2504
2.2315
2.0314
1.9724
2.0057
1.1515
1.9158
0.9953
2.0000
N
F
2-(2-(4-fluorophenyl)ethynyl)pyridine
8
6
4
2
[ppm]
[rel]


8.5474
8.4051
8.3966
8.3931
7.6026
7.5984
7.5829
7.5788
7.1485
7.1362
7.1300
7.1169
4.0707
4.0628
4.0360
4.0172
2.3724
2.3550
2.3372
1.9634
1.5460
1.5243
1.5078
1.4788
1.4169
1.3966
1.3779
1.1999
1.1820
1.1642
0.8983
0.8800
0.8619
0.9662
0.9279
1.0000
1.0443
1.9564
1.9722
2.0014
2.8319
N
2-(hex-1-ynyl)pyridine
8
6
4
2
[ppm]
[rel]