

## **Selective Synthesis of $\alpha$ , $\beta$ -alkynyl ketones via nickel catalysed carbonylative Sonogashira reaction using oxalic acid as a sustainable C1 source**

Shaifali,<sup>[a],[b]</sup> Shankar Ram,<sup>[a],[b]</sup> Vandna Thakur,<sup>[a],[b]</sup> Pralay Das\*<sup>[a],[b]</sup>

<sup>[a]</sup>Natural Product Chemistry & Process Development Division, CSIR-Institute of Himalayan Bioresource Technology, Palampur 176061, HP, India

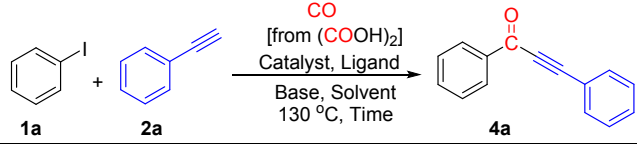
<sup>[b]</sup>Academy of Scientific & Innovative Research, New Delhi 110025, India

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## A. Optimization Studies

**Table S1:** Screening of reaction condition



S.No.	Catalyst (mol%)	Ligand (mol%)	Base (Equiv.)	Solvent	Yield (%) <sup>b</sup>
1	NiCl <sub>2</sub> .6H <sub>2</sub> O(15)	Dppb (15)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	50
2	NiCl <sub>2</sub> .6H <sub>2</sub> O(10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	60
3	NiCl <sub>2</sub> (5)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	40
4	NiBr <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	55
5	NiI <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	63
6	Ni(OTf) <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	55
7	Ni(OAc) <sub>2</sub> .4H <sub>2</sub> O (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	nd
8	---	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	nr
9	NiCl <sub>2</sub> (10)	---	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	25
10	NiCl <sub>2</sub> (10)	PPh <sub>3</sub> (10)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	Traces
11	NiCl <sub>2</sub> (10)	Dppp (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	Traces
12	NiCl <sub>2</sub> (10)	Xantphos (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	10
13	NiCl <sub>2</sub> (10)	Dppf (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	nd
<b>14</b>	NiCl <sub>2</sub> (10)	<b>Dppb (5)</b>	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	<b>70</b>
15	NiCl <sub>2</sub> (10)	Dppb (5)	Na <sub>2</sub> CO <sub>3</sub> (2)	DMF	63
16	NiCl <sub>2</sub> (10)	Dppb (5)	NaHCO <sub>3</sub> (2)	DMF	60
17	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>3</sub> PO <sub>4</sub> (2)	DMF	45
18	NiCl <sub>2</sub> (10)	Dppb (5)	NEt <sub>3</sub> (2)	DMF	20
19	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (1)	DMF	45
20	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (3)	DMF	62
21	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMA	55
22	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	Toluene	Traces
23	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2) PEG-Dioxane (1:1)		Traces
24 <sup>c</sup>	NiCl <sub>2</sub> (10)	Dppb(5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	nr
25 <sup>d</sup>	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	32
26 <sup>e</sup>	NiCl <sub>2</sub> (10)	Dppb (5)	K <sub>2</sub> CO <sub>3</sub> (2)	DMF	60

[a] Reaction condition: Iodobenzene (1 equiv.), Phenyl acetylene (2 equiv.), catalyst (0.10 equiv.) ligand (0.05 equiv.), base (2 equiv.), Oxalic acid (6 equiv.), DMF (0.5mL in outer vial, 2mL in inner vial), at 130 °C for 7h; dppe = 1,2-bis(diphenylphosphino)ethane, dppp = 1,3-bis(diphenylphosphino)propane, dppb = 1,4-bis(diphenylphosphino)butane, dppf = 1,1'-bis(diphenylphosphino)ferrocene, Xantphos= 4,5-bis(diphenylphosphino)-9,9 dimethylxant-hene; [b] Isolated yields; [c] at 100 °C [d] at 120 °C; [e] Reaction time = 12h.

We initiated optimization studies using iodobenzene **1a** and phenylacetylene **2a** employing oxalic acid as an *ex situ* CO source and K<sub>2</sub>CO<sub>3</sub> as base in presence of NiCl<sub>2</sub>.6H<sub>2</sub>O along with dppb. To our delight, we got desired carbonylative product **4a**

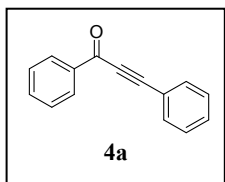
in 50% (Table S1, entry 1). Furthermore, decreasing the ligand concentration to half (1/2) i.e., 5 mol % improved the yield of the reaction to 60% (Table S1, entry 2). We got 40% of carbonylative product **4a** on decreasing the catalyst loading to 5 mol% (Table S1, entry 3). However, to enhance the yield of the product, we investigated amount of oxalic acid, different bases, suitable solvent, reaction time and temperature of given reaction. Intriguingly, different nickel catalysts such as NiBr<sub>2</sub>, NiI<sub>2</sub> and Ni(OTf)<sub>2</sub> gave **4a** in 55, 63 and 55% yields respectively and in case of Ni(OAc)<sub>4</sub>.4H<sub>2</sub>O the desired product was not detected (Table S1, entries 4-7). While, the reaction was not feasible in absence of catalyst (Table S1, entry 8). Also, to ascertain the role of ligand, we carried out the reaction in absence of ligand and carbonylated product **4a** was formed in 25% along with **5a** in 45% yield (Table S1, entry 9). It was found that 0.05 equiv. of ligand has contributed for formation of product in selective manner and increases the yield of the product. To evaluate the effect of ligand, we had investigated different ligands (Table S1, entries 10-14). Monodentate ligand i.e. triphenylphosphine (PPh<sub>3</sub>) did not give appreciable amount of product whereas, bidentate ligand dppb was found to be most suitable for carbonylative Sonogashira coupling reaction. Different bases such as Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, NEt<sub>3</sub> and K<sub>3</sub>PO<sub>4</sub> were also scrutinized and among them, K<sub>2</sub>CO<sub>3</sub> was the most efficient base for the given reaction (Table S1, entries 15-18). We observed the profound effect of solvent as the reaction is favoured by polar solvents than that of non-polar solvents (Table S1, Entries 21-23). We also monitored the reaction at lower temperature i.e. 100 °C and 120 °C (Table S1, entries 24 and 25). We got 32% of desired product at 120 °C, while no product was detected at 100 °C as decomposition of oxalic acid is feasible at >120 °C (Table S1, entries 24 and 25). On increasing the reaction time from 7 to 12h, We observed the reduction of yield of **4a** from 70 to 60% (Table S1, entry 26).

## A. General method

High quality reagents were purchased from Sigma Aldrich, Tech Chem Solutions (TCI, India), Loba Chemie, alpha aesar and Sd Fine-chem Ltd. Thin layer chromatography was performed using pre-coated silica gel plates 60F<sub>254</sub> (Merck) in UV light detector. ESI-MS spectra were determined using a Waters micro mass Q-TOF Ultima Spectrometer. Melting points were recorded using LAB INDIA MR-VIS<sup>+</sup>. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded using a Bruker Avance 600 spectrometer operating at 600 MHz (<sup>1</sup>H) and 150 MHz (<sup>13</sup>C) and Bruker Avance 300 spectrometer operating at 300 MHz (<sup>1</sup>H) and 75 MHz (<sup>13</sup>C). Spectra were recorded at 25 °C in CDCl<sub>3</sub> [residual CHCl<sub>3</sub> (δ<sub>H</sub> 7.26 ppm) or CDCl<sub>3</sub> (δ<sub>C</sub> 77.00 ppm)]. Chemical shifts were recorded in δ (ppm) relative to the TMS and NMR solvent signal. Coupling constants (J) are given in Hz and multiplicities of signals are reported as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad singlet.

## B. Experimental procedure for nickel catalyzed carbonylative Sonogashira coupling of aryl iodides using oxalic acid as an *ex situ* CO surrogates:

### 1, 3-Diphenylprop-2-yn-1-one <sup>1</sup>



In a 2 mL vial iodobenzene (0.49 mmol, 100 mg), phenyl acetylene (0.98 mmol, 100 mg), K<sub>2</sub>CO<sub>3</sub> (0.98 mmol), nickel chloride (0.049, 6.3 mg), dppb (0.024 mmol, 10.4 mg) and DMF (1.5 mL) was added and this reaction vial was placed in a 5 mL reaction vessel which contained oxalic acid (2.941 mmol, 264 mg), DMF (0.5 mL). The 5 mL reaction vessel tighten with the solid top cap sealed with Teflon tape and heated at 130 °C for required time. The reaction progress was monitored by TLC and after completion of the reaction, the inner vial was removed. Further, water was added in the reaction mixture contents of inner vial and extracted with ethyl acetate. The combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude mixture was further purified by silica gel

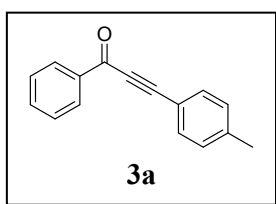
column chromatography using hexane as eluent, afforded **4a** as pale yellow oily liquid (70 mg, 70%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.40-7.54 (m, 5H), 7.61-7.70 (m, 3H), 8.23 (d,  $J = 7.38$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$ (ppm) 86.90, 93.13, 120.15, 128.64, 128.70, 129.59, 130.81, 133.09, 134.14, 178.04.

ESI-MS (M+H) $^+$  calculated for  $\text{C}_{15}\text{H}_{11}\text{O}^+$  is 207.0804 and observed 207.0821.

### 1-Phenyl-3-(p-tolyl)prop-2-yn-1-one<sup>1</sup>



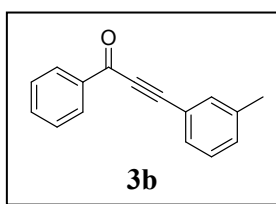
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-4-methylbenzene (0.98 mmol, 113 mg), gave after purification with silica gel column chromatography in hexane **3a** as yellow oil (70 mg, 65%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 2.33 (s, 3H), 7.15 (d,  $J = 7.8$  Hz, 2H), 7.43 (t,  $J = 7.5$  Hz, 2H), 7.50-7.55 (m, 3H), 8.14 (d,  $J = 7.2$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$ (ppm) 21.73, 86.75, 93.80, 117.00, 128.56, 129.46, 129.52, 133.10, 133.98, 136.95, 141.54, 178.05.

ESI-MS(M+H) $^+$ calculated for  $\text{C}_{16}\text{H}_{13}\text{O}^+$  is 221.0961 observed 221.0960.

### 1-Phenyl-3-(m-tolyl)prop-2-yn-1-one<sup>2</sup>



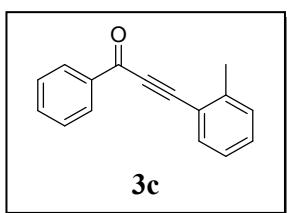
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) 1-ethynyl-3-methylbenzene (0.98 mmol, 113 mg), gave after purification with silica gel column chromatography in hexane **3b** as yellow oil (59 mg, 55%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 2.31 (s, 3H), 7.23-7.24 (m, 2H), 7.46 (m, 4H), 7.54 (m, 1H), 8.15 (d,  $J = 8.0$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 21.19, 86.67, 93.52, 119.93, 128.61, 129.58, 130.25, 131.76, 133.57, 134.08, 138.54, 178.08.

ESI-MS(M+H)<sup>+</sup>calculated for **C<sub>16</sub>H<sub>13</sub>O<sup>+</sup>** is 221.0961 observed 221.0960.

**1-Phenyl-3-(o-tolyl)prop-2-yn-1-one<sup>2</sup>**



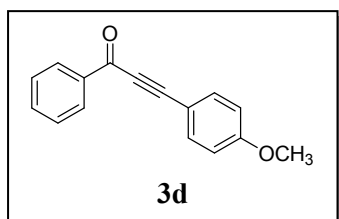
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-2-methylbenzene (0.98 mmol, 113 mg), gave after purification with silica gel column chromatography in hexane **3c** as yellow oil (61 mg, 57%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 2.64 (s, 3H), 7.28-7.35 (m, 2H), 7.40-7.45 (m, 1H), 7.55 (t,  $J = 7.3$  Hz, 2H), 7.68 (t,  $J = 8.3$  Hz, 2H), 8.28 (d,  $J = 2.4$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 20.8, 90.76, 92.19, 120.01, 125.95, 128.64, 129.55, 130.82, 133.09, 134.04, 137.05, 142.37, 178.07.

ESI-MS( $\text{M}+\text{H}$ ) $^+$  calculated for  $\text{C}_{16}\text{H}_{13}\text{O}^+$  is 221.0961 observed 221.0965 [ $\text{M}-\text{H}+\text{HCOOH}$ ] $^-$  =266.06455.

### 3-(4-Methoxyphenyl)-1-phenylprop-2-yn-1-one<sup>1</sup>



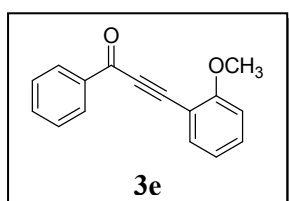
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-4-methoxybenzene (0.98 mmol, 135 mg), gave after purification with silica gel column chromatography in hexane **3d** as yellow solid (69 mg, 60%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 3.85 (s, 3H), 6.93 (d,  $J = 8.5$  Hz, 2H), 7.52 (t,  $J = 7.0$  Hz, 2H), 7.59-7.65 (m, 3H), 8.22 (d,  $J = 7.9$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 55.44, 86.90, 94.30, 111.94, 114.45, 128.56, 133.88, 135.14, 137.10, 161.76, 178.03.

ESI-MS( $\text{M}+\text{H}$ ) $^+$  calculated for is  $\text{C}_{16}\text{H}_{13}\text{O}_2^+$  237.0910 observed is 237.0912.

### 3-(2-Methoxyphenyl)-1-phenylprop-2-yn-1-one



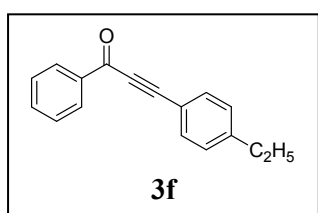
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-2-methoxybenzene (0.98 mmol, 129 mg), gave after purification with silica gel column chromatography in hexane **3e** as yellow oil (64 mg, 55%).

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 4.00 (s, 3H), 6.98 (d,  $J = 8.3$  Hz, 1H), 7.01 (t,  $J = 7.56$  Hz, 1H), 7.46-7.49 (m, 1H), 7.54 (t,  $J = 7.8$  Hz, 2H), 7.64 (t,  $J = 7.5$  Hz, 2H), 8.33-8.35 (m, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 90.52, 91.25, 109.50, 110.87, 120.70, 128.50, 129.76, 132.59, 133.84, 135.00, 137.20, 161.89, 178.10.

ESI-MS(M+H)<sup>+</sup> calculated for is  $\text{C}_{16}\text{H}_{13}\text{O}_2^+$  237.0910 observed is 237.0904.

### 3-(4-Ethylphenyl)-1-phenylprop-2-yn-1-one<sup>1</sup>



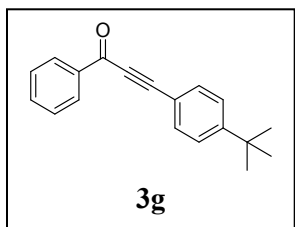
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethyl-4-ethynylbenzene (0.98 mmol, 127 mg), gave after purification with silica gel column chromatography in hexane **3f** as yellow solid (80 mg, 70%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 1.28 (t,  $J = 7.6$  Hz, 3H), 2.72 (q,  $J = 15.15$  Hz, 7.6 Hz, 2H), 7.27 (d,  $J = 7.9$  Hz, 2H), 7.53 (t,  $J = 7.5$  Hz, 2H), 7.63 (d,  $J = 7.8$  Hz, 2H), 8.24 (d,  $J = 7.6$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 15.18, 29.05, 86.76, 93.88, 117.23, 128.32, 128.60, 129.56, 133.25, 134.01, 137.00, 144.78, 178.11.

ESI-MS (M+H)<sup>+</sup> calculated for  $\text{C}_{17}\text{H}_{15}\text{O}^+$  is 235.1117 observed 235.1129.

### 3-(4-(Tert-butyl)phenyl)-1-phenylprop-2-yn-1-one<sup>1</sup>



Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-(tert-butyl)-4-ethynylbenzene (0.98 mmol, 154mg), gave after purification with silica gel column chromatography in hexane **3g** as yellow oil (71 mg, 55%).

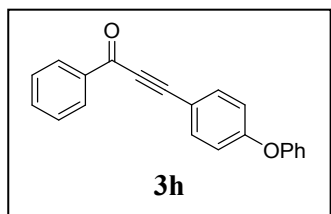
$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 1.36 (s, 9H), 7.46 (d,  $J = 8.1$  Hz, 2H), 7.54 (t,  $J = 7.4$  Hz, 2H), 7.63-7.67 (m, 3H), 8.25 (d,  $J = 7.2$  Hz, 2H).



$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 31.06, 35.11, 86.75, 93.82, 117.05, 125.78, 128.60, 129.57, 133.00, 134.01, 137.03, 154.60, 178.11.

ESI-MS(M+H)<sup>+</sup> calculated for  $\text{C}_{19}\text{H}_{19}\text{O}^+$  is 263.1430 observed 263.1418.

### 3-(4-Phenoxyphenyl)-1-phenylprop-2-yn-1-one



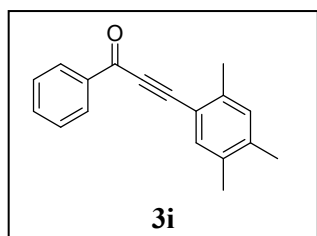
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-4-phenoxybenzene (0.98 mmol, 194 mg) gave after purification with silica gel column chromatography in hexane **3h** as yellow oil (60 mg, 45%).

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.02-7.04 (m, 2H), 7.09-7.11 (m, 2H), 7.23 (t,  $J = 7.44$  Hz, 1H), 7.41-7.11 (m, 2H), 7.54 (t,  $J = 7.56$  Hz, 2H), 7.64-7.69 (m, 3H), 8.23 (d,  $J = 7.2$  Hz, 2H)

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.96, 93.46, 114.03, 118.04, 120.10, 124.63, 128.63, 129.55, 130.10, 134.04, 135.16, 137.00, 155.51, 160.18, 178.02.

ESI-MS(M+H)<sup>+</sup> calculated for  $\text{C}_{21}\text{H}_{15}\text{O}_2^+$  is 299.1060 observed 299.1060

### 1-Phenyl-3-(2, 4, 5-trimethylphenyl)prop-2-yn-1-one



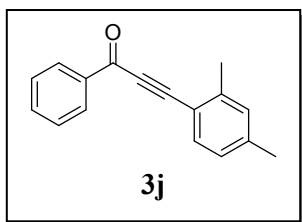
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-2,4,5-trimethylbenzene (0.98 mmol, 141 mg), gave after purification with silica gel column chromatography in hexane **3i** as yellow solid (73 mg, 60%).

$^1\text{H}$  (300 MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 2.25 (s, 3H), 2.27 (s, 3H), 2.52 (s, 3H), 7.06 (s, 1H), 7.43 (s, 1H), 7.51 (t,  $J = 7.3$  Hz, 2H), 7.62 (t,  $J = 7.3$  Hz, 1H), 8.325 (d,  $J = 7.3$  Hz, 2H)

$^{13}\text{C}$  (75 MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 19.02, 19.94, 20.25, 90.44, 93.22, 117.094, 128.57, 129.50, 131.30, 133.85, 134.33, 137.21, 139.68, 140.35, 178.11.

ESI-MS(M+H)<sup>+</sup> calculated for  $\text{C}_{18}\text{H}_{17}\text{O}^+$  is 249.1274 observed 249.1296

### 3-(2, 4-Dimethylphenyl)-1-phenylprop-2-yn-1-one



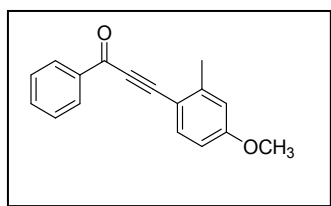
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-2,4-dimethylbenzene (0.98 mmol, 127 mg), gave after purification with silica gel column chromatography in hexane **3j** as yellow solid (69 mg, 60%)

<sup>1</sup>H (300 MHz, CDCl<sub>3</sub>), δ (ppm) 2.36 (s, 3H), 2.56 (s, 3H), 7.20 (s, 2H), 7.49-7.56 (m, 3H), 7.63-7.65 (m, 1H), 8.25 (d, *J* = 7.3 Hz, 2H)

<sup>13</sup>C (75 MHz, CDCl<sub>3</sub>), δ (ppm) 20.36, 20.68, 90.50, 92.58, 119.67, 128.61, 129.53, 129.78, 131.78, 131.96, 134.02, 135.54, 137.12, 139.15, 178.07.

ESI-MS(M+H)<sup>+</sup> calculated for C<sub>17</sub>H<sub>15</sub>O<sup>+</sup> is 235.1117 and observed 235.1111.

### 3-(4-Methoxy-2-methylphenyl)-1-phenylprop-2-yn-1-one



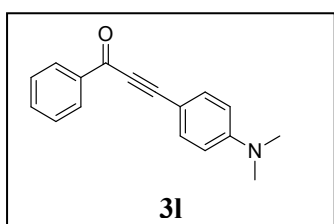
Prepared as described for **3a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 1-ethynyl-4-methoxy-2-methylbenzene (0.98 mmol, 143 mg), gave after purification with silica gel column chromatography in hexane **3m** as yellow solid (76 mg, 62%).

<sup>1</sup>H (300 MHz, CDCl<sub>3</sub>), δ (ppm) 2.60 (s, 3H), 3.86 (s, 3H), 6.785 (d, *J* = 8.6 Hz, 1H), 6.82 (s, 1H), 7.53 (t, *J* = 7.56 Hz, 2H), 7.62 (d, *J* = 7.8 Hz, 2H) 8.245 (d, *J* = 7.8 Hz, 2H).

<sup>13</sup>C (75 MHz, CDCl<sub>3</sub>), δ (ppm) 21.18, 55.37, 90.17, 93.40, 111.86, 112.05, 115.53, 128.57, 129.44, 133.79, 135.70, 137.27, 144.59, 161.70, 178.06.

ESI-MS(M+H)<sup>+</sup> calculated for C<sub>17</sub>H<sub>15</sub>O<sub>2</sub><sup>+</sup> is 251.1067 observed 251.1055.

### 3-(4-(Dimethylamino)phenyl)-1-phenylprop-2-yn-1-one<sup>1</sup>



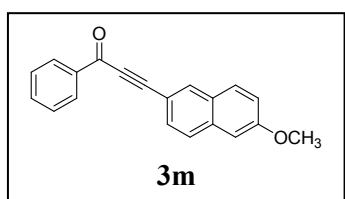
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 4-ethynyl-N,N-dimethylaniline (0.98 mmol, 142 mg), gave after purification with silica gel column chromatography in hexane **3l** as yellow solid (69 mg, 57%)

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 3.04 (s, 6H), 6.66 (d,  $J = 8.8$  Hz, 2H), 7.50 (t,  $J = 7.66$  Hz, 2H), 7.56-7.60 (m, 3H), 8.22 (d,  $J = 7.4$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$ (ppm) 40.01, 87.90, 97.59, 105.69, 110.61, 111.60, 128.46, 129.39, 130.52, 133.50, 135.50, 151.80, 177.97.

ESI-MS(M+H) $^+$  calculated for  $\text{C}_{17}\text{H}_{16}\text{NO}^+$  is 250.1220 observed 250.1220.

### 3-(6-Methoxynaphthalen-2-yl)-1-phenylprop-2-yn-1-one



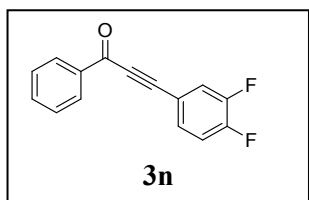
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 2-ethynyl-6-methoxynaphthalene (0.98 mmol, 178 mg), gave after purification with silica gel column chromatography in hexane **3m** as yellow solid (100 mg, 75%).

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 3.97 (s, 3H), 7.15 (d,  $J = 2.3$  Hz), 7.22-7.24 (m, 1H), 7.56 (t,  $J = 7.7$  Hz, 2H), 7.65-7.68 (m, 2H), 7.78 (t,  $J = 9.0$  Hz, 2H), 8.20 (s, 1H), 8.29 (d,  $J = 7.0$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 55.44, 87.10, 94.41, 105.97, 114.78, 120.01, 127.28, 128.24, 128.62, 129.22, 129.84, 134.00, 134.26, 135.61, 137.10, 159.43, 178.02.

ESI-MS(M+H) $^+$  calculated for  $\text{C}_{17}\text{H}_{15}\text{O}_2^+$  is 287.1067 observed 287.1052.

### 3-(3,4-Difluorophenyl)-1-phenylprop-2-yn-1-one



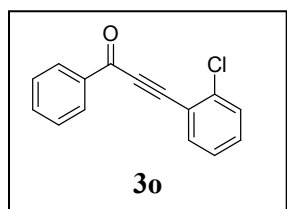
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 4-ethynyl-1,2-difluorobenzene (0.98 mmol, 105 mg), gave after purification with silica gel column chromatography in hexane **3n** as white solid (53 mg, 45%).

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.23-7.28 (m, 1H), 7.47-7.48 (m, 1H), 7.49-7.56 (m, 3H), 7.66-7.69 (m, 1H), 8.21 (d,  $J = 7.26$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.92, 90.15, 116.94-117.02 (splitting occurred), 118.07-118.19 (d,  $J = 18.6$  Hz), 121.95-122.08 (d,  $J = 18.4$  Hz), 128.74, 129.58, 130.08-130.14 (splitting occurred), 134.39, 136.62, 149.29-151.06 (q,  $J = 15.0$  Hz, 251 Hz), 151.30-153.08 (q,  $J=14.8$  Hz, 253 Hz), 177.64.

ESI-MS(M+H)<sup>+</sup> calculated for  $\text{C}_{15}\text{H}_9\text{F}_2\text{O}^+$  is 243.630 observed 243.0616.

### 3-(2-Chlorophenyl)-1-phenylprop-2-yn-1-one



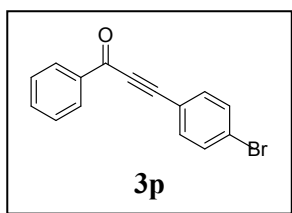
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 4-ethynyl-2-chlorobenzene (.98 mmol, 133 mg), gave after purification with silica gel column chromatography in hexane **3o** as yellow solid (82 mg, 70%)

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.32-7.35 (m, 1H), 7.42-7.44 (m, 1H), 7.50-7.56 (m, 3H), 7.64-7.67 (m, 1H), 7.72-7.74 (m, 1H), 8.31-8.33 (m, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 89.05, 91.05, 120.47, 126.88, 128.70, 129.69, 129.81, 131.82, 134.28, 135.09, 136.80, 137.53, 177.84.

ESI-MS (M+H)<sup>+</sup> calculated for  $\text{C}_{15}\text{H}_{10}\text{ClO}^+$  is 241.0415 observed 241.0415.

### 17. 3-(2-Bromophenyl)-1-phenylprop-2-yn-1-one<sup>2</sup>



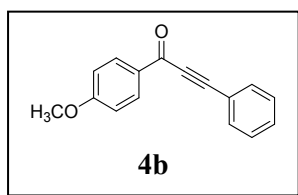
Prepared as described for **4a** starting from 4-iodobenzene (0.49 mmol, 100 mg) and 4-ethynyl-4-bromobenzene (0.98 mmol, 175 mg), gave after purification with silica gel column chromatography in hexane **3p** as yellow solid (42 mg, 30%)

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.54-7.61 (m, 6H), 7.67 (t,  $J = 7.55$  Hz, 1H), 8.21 (d,  $J = 7.31$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 87.69, 91.65, 119.07, 125.62, 128.69, 129.70, 132.13, 134.29, 134.35, 136.74, 177.84.

ESI-MS (M+H)<sup>+</sup> calculated for  $\text{C}_{15}\text{H}_{10}\text{BrO}^+$  is 284.9910 observed 284.9910

### 1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-one<sup>1</sup>



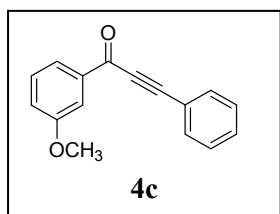
Prepared as described for **4a** starting from 4-iodoanisole (0.42 mmol, 100 mg) and phenyl acetylene (0.85 mmol, 87 mg) gave after purification with silica gel column chromatography in hexane **4b** as pale yellow solid (75 mg, 65%).

<sup>1</sup>H (600MHz, CDCl<sub>3</sub>), δ (ppm) 3.90 (s, 3H), 6.99 (d, *J* = 8.5 Hz, 2H), 7.41-7.47 (m, 3H), 7.66-7.68 (m, 3H), 8.19 (d, *J* = 8.5 Hz, 2H).

<sup>13</sup>C (150MHz, CDCl<sub>3</sub>), δ (ppm) 55.59, 86.95, 92.29, 113.90, 120.41, 128.65, 130.38, 130.56, 131.98, 132.95, 164.51, 176.66.

ESI-MS(M+H)<sup>+</sup> calculated for C<sub>16</sub>H<sub>13</sub>O<sub>2</sub><sup>+</sup> is 237.0910 and observed is 237.913.

### 1-(3-Methoxyphenyl)-3-phenylprop-2-yn-1-one<sup>1</sup>



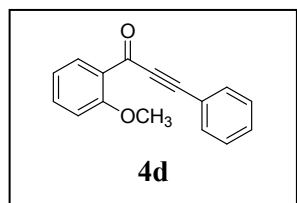
Prepared as described for **4a** starting from 3-iodoanisole (0.427 mmol, 100 mg) and phenyl acetylene (0.85 mmol, 87 mg) gave after purification with silica gel column chromatography in hexane **4c** as yellow oil (66 mg, 57%).

<sup>1</sup>H (600MHz, CDCl<sub>3</sub>), δ (ppm) 3.96 (s, 3H), 7.00-7.10 (m, 2H), 7.37-7.45 (m, 3H), 7.54 (t, *J* = 7.8 Hz, 1H), 7.62-7.64 (m, 2H), 8.09 (d, *J* = 7.7 Hz, 1H).

<sup>13</sup>C (150MHz, CDCl<sub>3</sub>), δ (ppm) 55.90, 89.18, 91.54, 112.19, 120.29, 120.69, 126.76, 128.55, 130.39, 132.39, 132.58, 132.92, 134.95, 159.80, 176.70.

ESI-MS (M+H)<sup>+</sup> calculated C<sub>16</sub>H<sub>13</sub>O<sub>2</sub><sup>+</sup> is 237.0910 and observed is 237.9130.

### 1-(2-Methoxyphenyl)-3-phenylprop-2-yn-1-one<sup>1</sup>



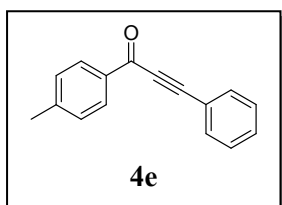
Prepared as described for **4a** starting from 2-iodoanisole (0.427 mmol, 100 mg) and phenyl acetylene (0.85 mmol, 87 mg) gave after purification with silica gel column chromatography in hexane **4d** as yellow oil (64 mg, 55%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 3.89 (s, 3H), 7.19 (d,  $J = 6$  Hz, 1H), 7.41-7.49 (m, 4H), 7.67-7.70 (m, 3H), 7.86 (d,  $J = 7.6$  Hz, 1H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 55.50, 87.01, 93.10, 112.85, 120.14, 121.08, 122.87, 129.65, 129.90, 130.80, 133.08, 138.30, 159.84, 177.77.

ESI-MS ( $\text{M}+\text{H}$ ) $^+$  calculated  $\text{C}_{16}\text{H}_{13}\text{O}_2^+$  is 237.0910 and observed is 237.913.

### 3-Phenyl-1-(p-tolyl)prop-2-yn-1-one<sup>2</sup>



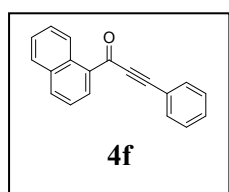
Prepared as described for **4a** starting from 4-iodotoluene (0.458 mmol, 100 mg) and phenyl acetylene (0.92 mmol, 93 mg) gave after purification with silica gel column chromatography in hexane **4e** as pale yellow solid (70 mg, 65%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 2.47 (s, 3H), 7.33 (d,  $J = 7.83$  Hz, 2H), 7.41-7.50 (m, 3H), 7.70 (d,  $J = 6.91$  Hz, 2H), 8.13 (d,  $J = 8.01$  Hz, 2H).

$^{13}\text{C}$  (75MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 21.86, 86.99, 92.61, 120.28, 128.67, 129.36, 129.72, 130.69, 133.69, 134.64, 145.25, 177.73.

ESI-MS ( $\text{M}+\text{H}$ ) $^+$  calculated for  $\text{C}_{16}\text{H}_{13}\text{O}^+$  is 221.0961 observed 221.0966.

### 1-(Naphthalen-1-yl)-3-phenylprop-2-yn-1-one<sup>2</sup>



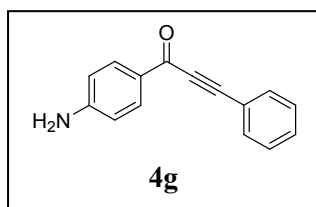
Prepared as described for **4a** starting from 1-iodonaphthalene (0.393 mmol, 100 mg) and phenyl acetylene (0.78 mmol, 80 mg) gave after purification with silica gel column chromatography in hexane **4f** as yellow oil (89 mg, 71%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.40-7.49 (m, 3H), 7.56-7.72 (m, 5H), 7.92 (d,  $J = 8.1$  Hz, 1H), 8.10 (d,  $J = 8.1$  Hz, 1H), 8.65 (d,  $J = 7.2$  Hz, 1H), 9.25 (d,  $J = 8.8$  Hz, 1H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$ (ppm) 88.51, 91.72, 120.38, 124.49, 126.02, 126.79, 128.60, 128.68, 128.97, 130.62, 130.76, 132.97, 133.90, 134.52, 135.11, 179.74.

ESI-MS (M+H) $^+$  calculated for  $\text{C}_{19}\text{H}_{13}\text{O}^+$  is 257.0966 observed 257.0961.

#### 1-(4-Aminophenyl)-3-phenylprop-2-yn-1-one



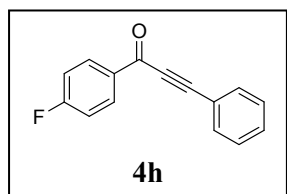
Prepared as described for **4a** starting from 4-iodoaniline (0.45 mmol, 100 mg) and phenyl acetylene (0.91 mmol, 93 mg) gave after purification with silica gel column chromatography in hexane **4g** as yellow solid (76 mg, 70%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 4.36 (s, 2H), 6.67 (d,  $J = 8.58$  Hz, 2H), 7.36-7.45 (m, 3H), 7.64 (d,  $J = 7.62$  Hz, 2H), 8.05 (d,  $J = 8.61$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 87.15, 91.54, 113.73, 120.65, 127.82, 128.60, 130.34, 132.36, 132.85, 152.39, 176.29.

ESI-MS(M+H) $^+$  calculated for  $\text{C}_{15}\text{H}_{12}\text{NO}^+$  is 207.0923, observed 222.0913.

#### 1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-one<sup>1</sup>



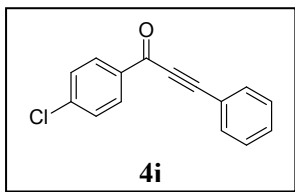
Prepared as described for **4a** starting from 1-fluoro 4-iodobenzene (0.45 mmol, 100 mg) and phenyl acetylene (0.90 mmol, 92 mg) gave after purification with silica gel column chromatography in hexane **4h** as yellow solid (67 mg, 55%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.21 (t,  $J = 8.53$  Hz, 2H), 7.42-7.51 (m, 3H), 7.69 (d,  $J = 6.9$  Hz, 2H), 8.24-8.29 (m, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.60, 93.35, 115.72-116.013 (Splitting found d,  $J = 22$  Hz), 119.00, 128.00, 130.89, 132.29, 133.05, 133.42, 164.77-168.168 (d,  $J = 254$  Hz splitting found), 176.36.

ESI-MS(M+H) $^+$  calculated for  $\text{C}_{15}\text{H}_{10}\text{FO}^+$  is 225.0710 observed 225.0721.

#### 1-(4-Chlorophenyl)-3-phenylprop-2-yn-1-one<sup>1</sup>



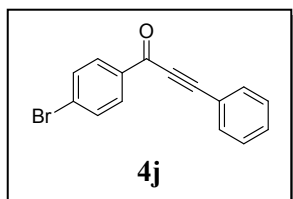
Prepared as described for **4a** starting from 1-chloro-4-iodobenzene (0.42 mmol, 100 mg) and phenyl acetylene (0.85 mmol, 87 mg) gave after purification with silica gel column chromatography in hexane **4i** as yellow solid (67 mg, 57%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.43-7.50 (m, 5H), 7.70 (d,  $J = 7.62$  Hz, 2H), 8.17 (d,  $J = 8.1$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.58, 93.65, 119.89, 128.75, 129.02, 130.88, 130.99, 133.31, 140.73, 176.69.

ESI-MS (M+H) $^+$  calculated for  $\text{C}_{15}\text{H}_{10}\text{ClO}^+$  is 207.0415 observed 241.0420.

#### 1-(4-Bromophenyl)-3-phenylprop-2-yn-1-one



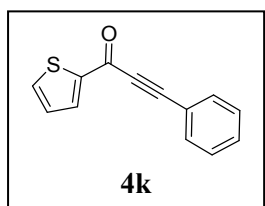
Prepared as described for **4a** starting from 1-bromo-4-iodobenzene (0.35 mmol, 100 mg) and phenyl acetylene (0.71 mmol, 72 mg) gave after purification with silica gel column chromatography in hexane **4j** as yellow solid (57 mg, 41%).

$^1\text{H}$  (300MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.12 (t,  $J = 8.6$  Hz, 2H), 7.52 (t,  $J = 7.7$  Hz, 2H), 7.62-7.65 (m, 1H), 7.68-7.70 (m, 2H), 8.20-8.21 (m, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 87.69, 91.65, 119.07, 125.62, 128.69, 19.70, 132.13, 134.29, 134.35, 136.74, 177.94.

ESI-MS (M+H) $^+$  calculated for  $\text{C}_{15}\text{H}_{10}\text{BrO}^+$  is 284.9910 observed 284.9910.

#### 3-Phenyl-1-(thiophen-2-yl)prop-2-yn-1-one





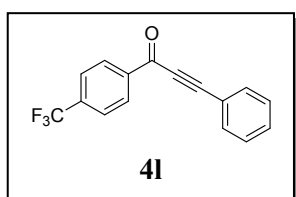
Prepared as described for **4a** starting from 2-iodothiophene (0.476 mmol, 100 mg) and phenyl acetylene (0.95 mmol, 97 mg) gave after purification with silica gel column chromatography in hexane **4k** as brown solid (46 mg, 42%).

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.18-7.19 (m, 1H), 7.42 (t,  $J = 7.5$  Hz, 2H), 7.47-7.49 (m, 1H), 7.65-7.67 (m, 2H), 7.72 – 7.35 (m, 1H), 8.00-8.01 (m, 1H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.50, 91.77, 119.96, 128.36, 128.71, 130.87, 133.06, 135.10, 135.27, 144.96, 168.82.

ESI-MS (M+H) $^+$ calculated for  $\text{C}_{13}\text{H}_9\text{OS}^+$  is 213.0369 observed 213.0372.

### 3-Phenyl-1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one<sup>2</sup>



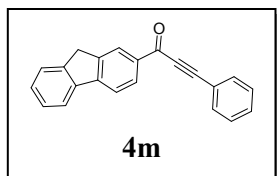
Prepared as described for **4a** starting from 1-iodo-4-(trifluoromethyl)benzene (0.36 mmol, 100 mg) and phenyl acetylene (0.73 mmol, 75 mg) gave after purification with silica gel column chromatography in hexane **4l** as yellow solid (51 mg, 38%).

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 7.43-7.46 (m, 2H), 7.50-7.53 (m, 1H), 7.69-7.70 (m, 2H), 7.79 (d,  $J = 8.2$  Hz, 2H), 8.33 (d,  $J = 8.1$  Hz, 2H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 86.59, 94.50, 119.69, 122.65, 124.46, 125.69-125.77 (q, splitting occurred), 128.81, 129.82, 131.21, 133.21, 134.90-135.50 (q, splitting occurred), 139.40, 176.74.

ESI-MS (M+H) $^+$ calculated for  $\text{C}_{16}\text{H}_{10}\text{F}_3\text{O}^+$  is 275.0687 observed 275.0678.

### 1-(9H-fluoren-2-yl)-3-phenylprop-2-yn-1-one



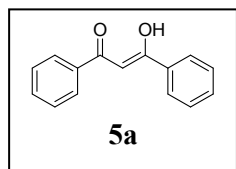
Prepared as described for **4a** starting from 2-iodo-9H-fluorene (0.34 mmol, 100 mg) and phenylacetylene (0.68 mmol, 69.8 mg) gave after purification with silica gel column chromatography in eluent Hexane : Ethylacetate (95:5) **4m** as yellow solid (50 mg, 50%).

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 4.00 (s, 2H), 7.40-7.45 (m, 4H), 7.48-7.50 (m, 1H), 7.61 (d,  $J = 7.26$  Hz, 1H), 7.71-7.73 (m, 2H), 7.89 (t,  $J = 8.3$  Hz, 2H), 8.29-8.30 (m, 1H), 8.38 (s, 1H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 36.90, 87.27, 92.73, 119.78, 120.37, 121.15, 125.34, 126.05, 127.18, 128.37, 128.70, 129.41, 130.69, 133.04, 135.59, 140.39, 143.36, 144.83, 147.64, 177.85.

ESI-MS (M+H) $^+$ calculated for  $\text{C}_{16}\text{H}_{10}\text{F}_3\text{O}^+$  is 275.0687 observed 275.0678.

### (Z)-3-hydroxy-1,3-diphenylprop-2-en-1-one<sup>3</sup>



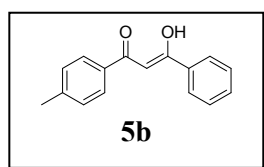
Prepared as described for **4a** starting from Iodobenzene (0.34 mmol, 100 mg) and phenylacetylene (0.98 mmol, 100 mg) gave after purification with silica gel column chromatography in hexane as eluent **5a** as colourless liquid (45 mg, 45%). The prepared compound **5a** was found to be exist as enol form of 1,3 diketones. (NMR matched with Ref. 3)

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 6.89 (s, 1H), 7.51-7.53 (m, 4H), 7.57-7.59 (m, 2H), 8.01 (d,  $J = 7.38$  Hz, 4H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 93.17, 127.18, 128.70, 132.46, 135.58, 185.79.

ESI-MS (M+H) $^+$ calculated for  $\text{C}_{15}\text{H}_{13}\text{O}_2^+$  is 225.0910 observed 225.0920.

### (Z)-3-hydroxy-3-phenyl-1-(p-tolyl)prop-2-en-1-one



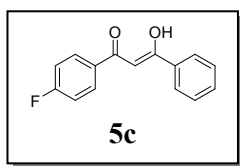
Prepared as described for **4a** starting from 4- Iodotoluene (0.34 mmol, 100 mg) and phenylacetylene (0.91 mmol, 93.57 mg) gave after purification with silica gel column chromatography in eluent Hexane **5b** as yellow solid (55 mg, 55%). The prepared compound **5b** was found to be exist as enol form of 1,3 diketones.

$^1\text{H}$  (600MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 2.46 (s, 3H), 6.86 (s, 1H), 7.31 (d,  $J = 7.98$  Hz, 2H), 7.50-7.52 (m, 2H), 7.56-7.59 (m, 1H), 7.92 (d,  $J = 8.16$  Hz, 2H), 8.01 (d,  $J = 7.3$  Hz, 2H), 16.9 (s, 1H).

$^{13}\text{C}$  (150MHz,  $\text{CDCl}_3$ ),  $\delta$  (ppm) 21.66, 92.85, 127.11, 127.26, 128.66, 129.42, 132.31, 132.88, 135.64, 143.29, 185.18, 186.08.

ESI-MS (M+H) $^+$ calculated for  $\text{C}_{16}\text{H}_{15}\text{O}_2^+$  is 239.1067 observed 239.1058 and (M+Na) $^+$  261.0785

**(Z)-1-(4-fluorophenyl)-3-hydroxy-3-phenylprop-2-en-1-one**



Prepared as described for **4a** starting from 4- fluoroiodobenzene (0.45 mmol, 100 mg) and phenylacetylene (0.90 mmol, 93 mg) in gave after purification with silica gel column chromatography in eluent Hexane **5c** as yellow solid (54 mg, 50%). The prepared compound **5c** was found to be exist as enol form of 1,3 diketones.

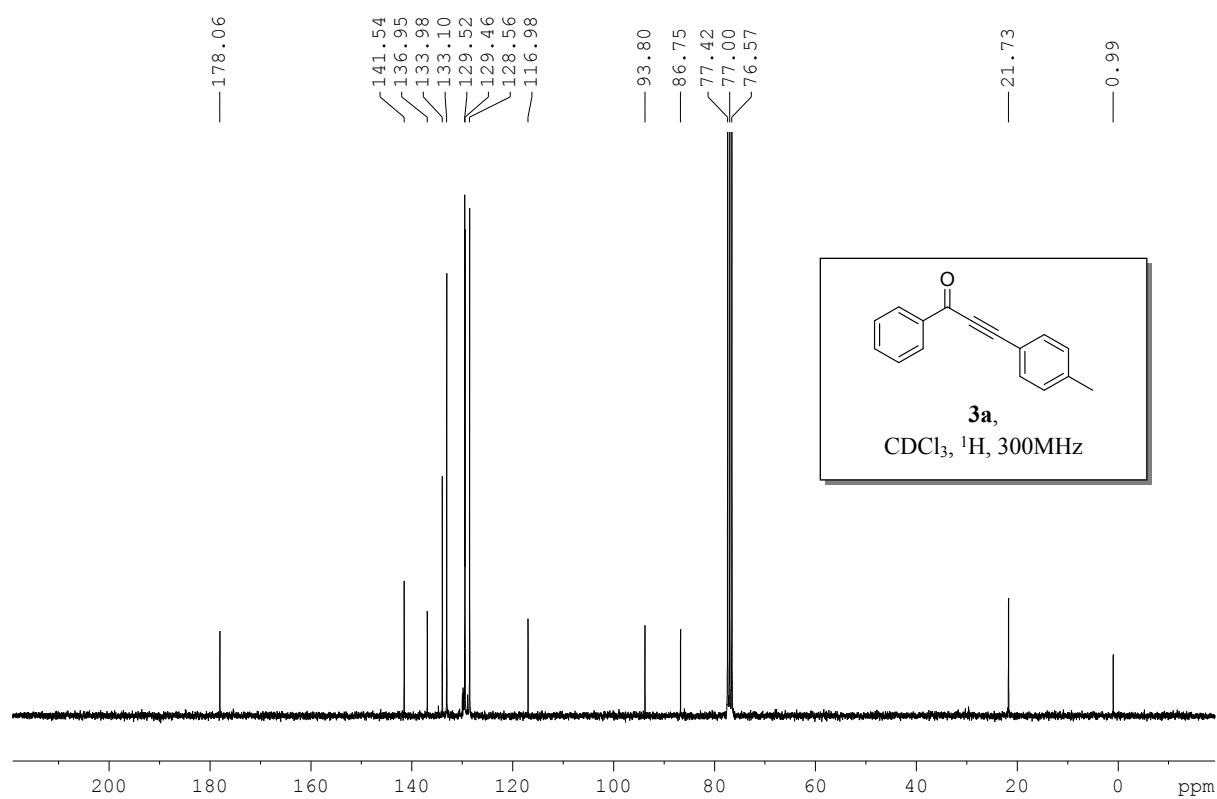
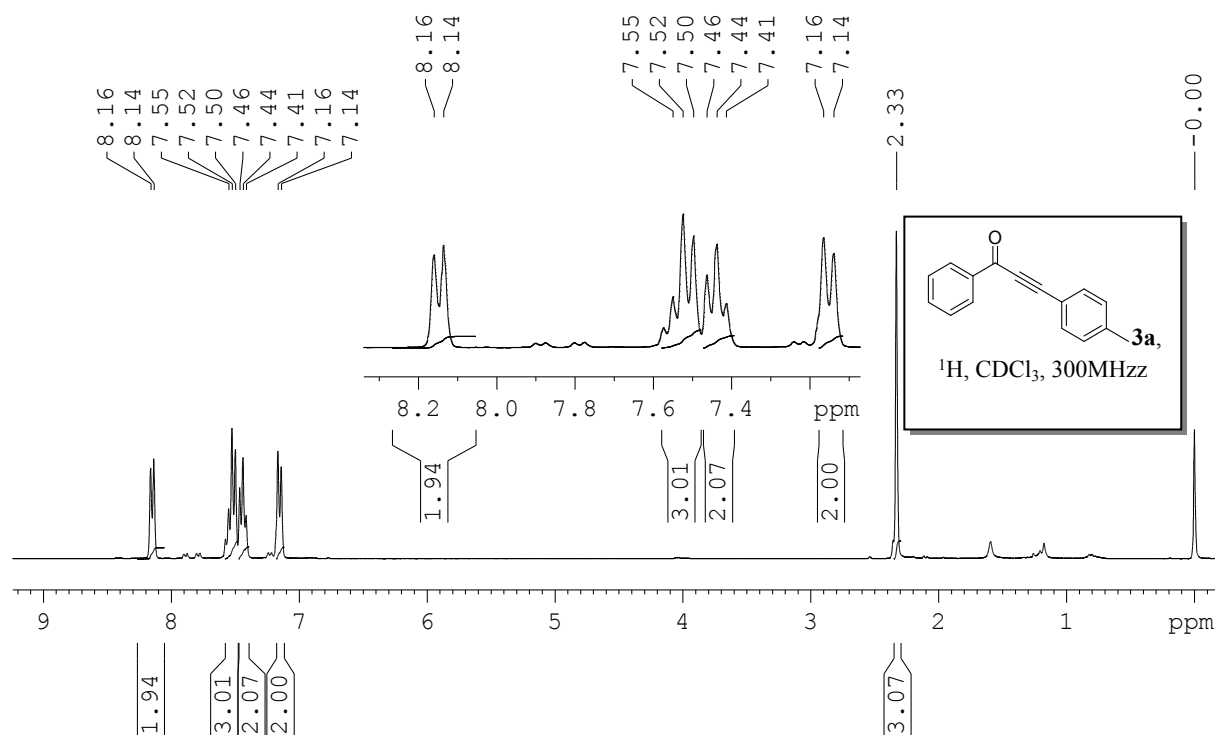
**<sup>1</sup>H** (600MHz, CDCl<sub>3</sub>),  $\delta$  (ppm) 6.80 (s, 1H), 6.86 (s, 1H), 7.17 (t,  $J = 8.5$  Hz, 2H), 7.49 (t,  $J = 7.7$  Hz, 2H), 7.56 (t,  $J = 7.3$  Hz, 1H), 7.97-8.02 (m, 4H), 16.8 (s, 1H).

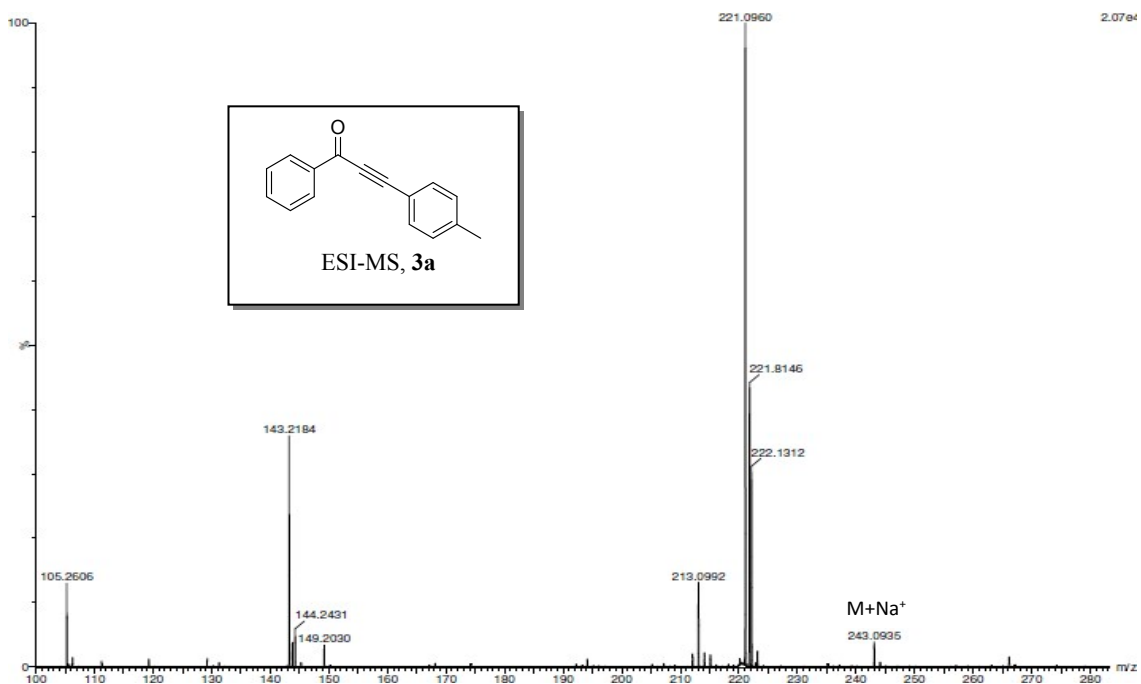
**<sup>13</sup>C** (150MHz, CDCl<sub>3</sub>),  $\delta$  (ppm) 92.85, 115.78-115.92 (d,  $J = 23.4$  Hz), 127.14, 128.73, 129.66-129.68 (d,  $J = 8.8$  Hz), 131.92-132.94 (d,  $J = 3.2$  Hz), 132.53, 135.30, 162.28-164.59 (d,  $J = 253.81$  Hz), 185.94.

ESI-MS (M+H)<sup>+</sup>calculated for C<sub>16</sub>H<sub>15</sub>O<sub>2</sub><sup>+</sup> is 243.0816 observed 239.0822 and (M+Na)<sup>+</sup> 265.0412

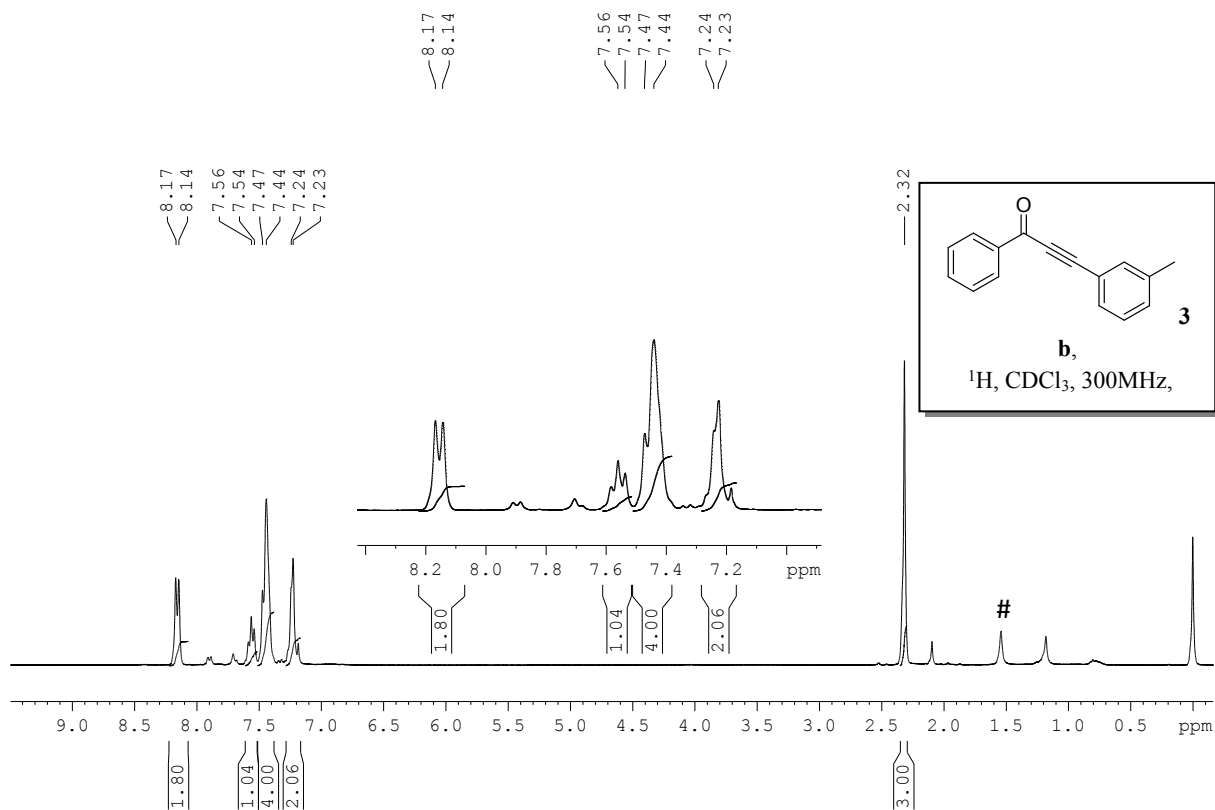
# A. $^1\text{H}$ and $^{13}\text{C}$ spectra of alkynes

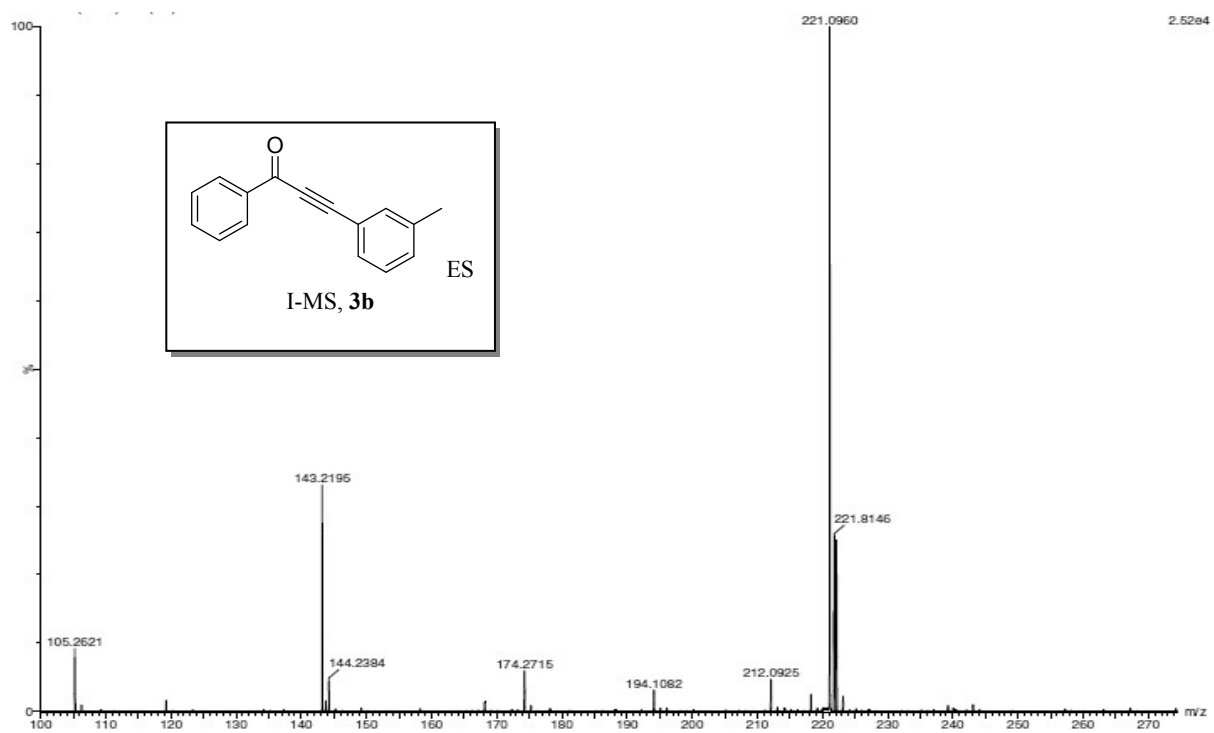
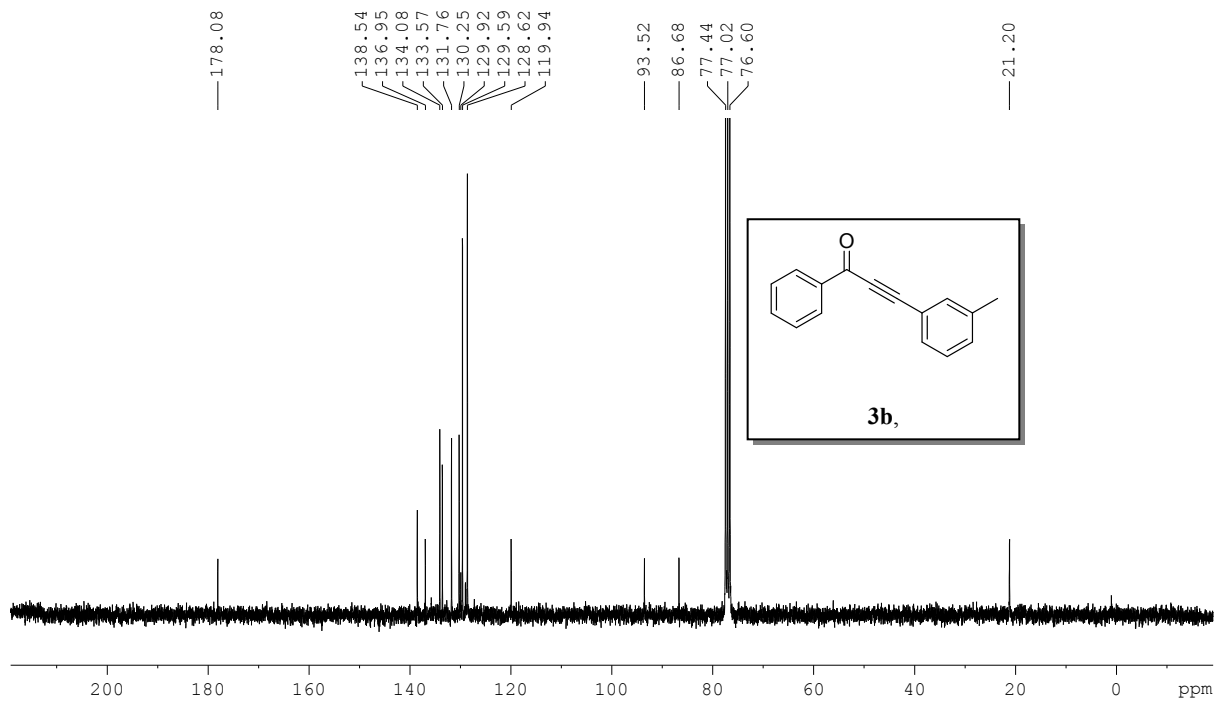
## 1-Phenyl-3-(p-tolyl)prop-2-yn-1-one



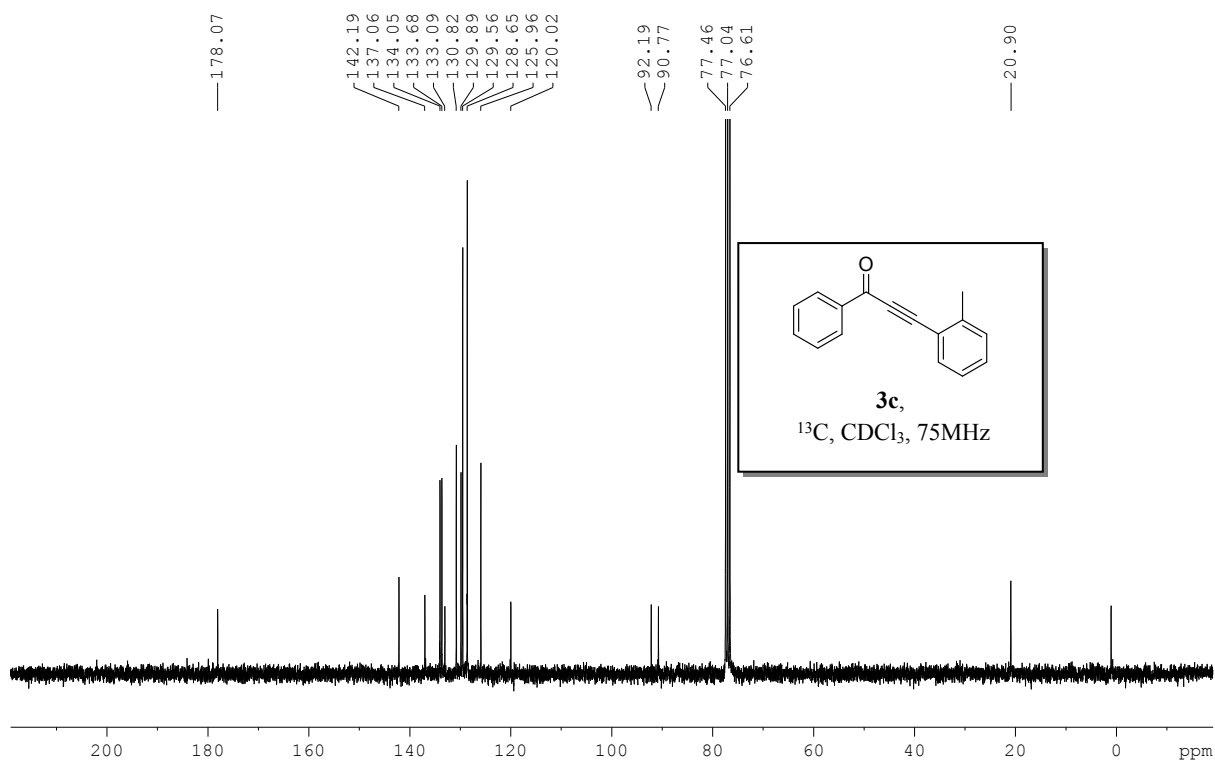
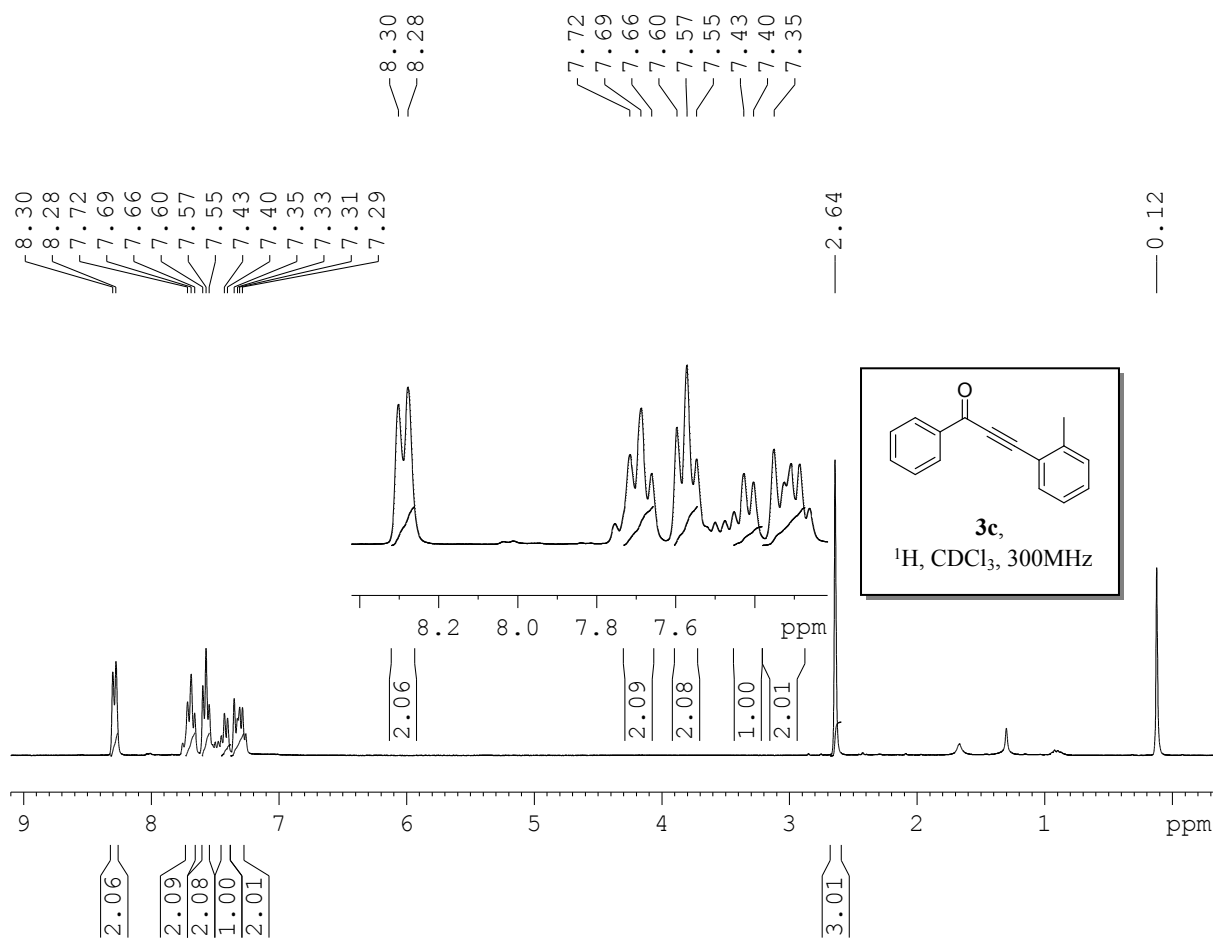


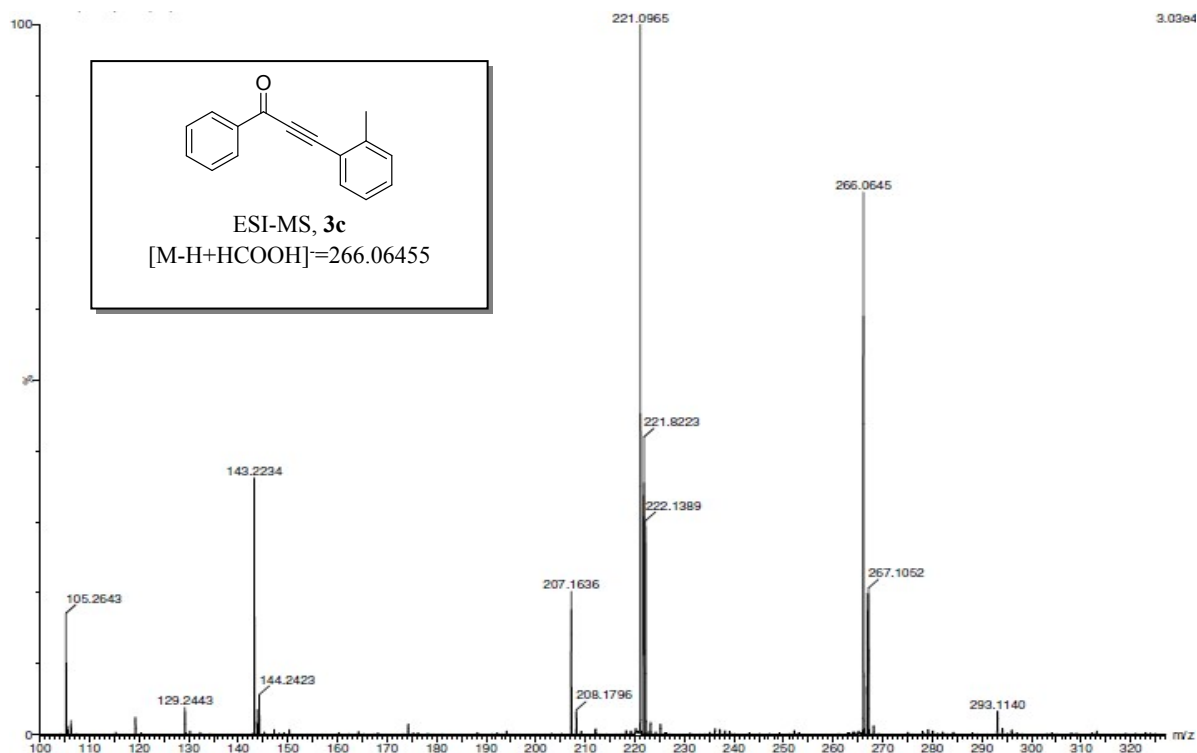
**1-Phenyl-3-(m-tolyl)prop-2-yn-1-one**



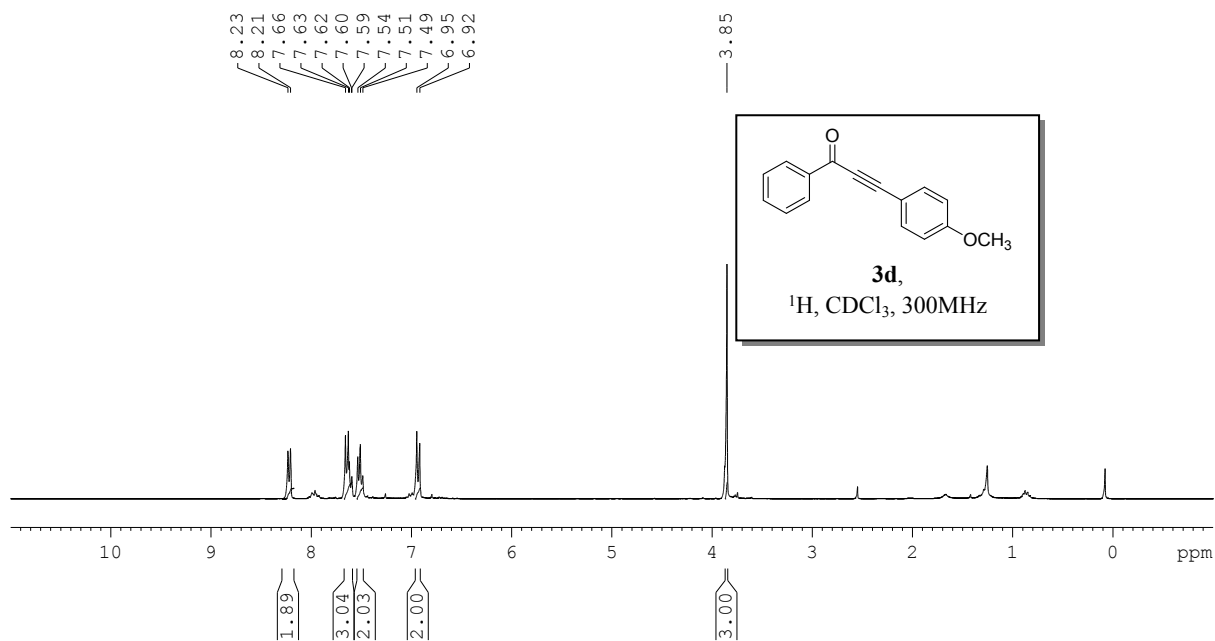


# 1-Phenyl-3-(o-tolyl)prop-2-yn-1-one

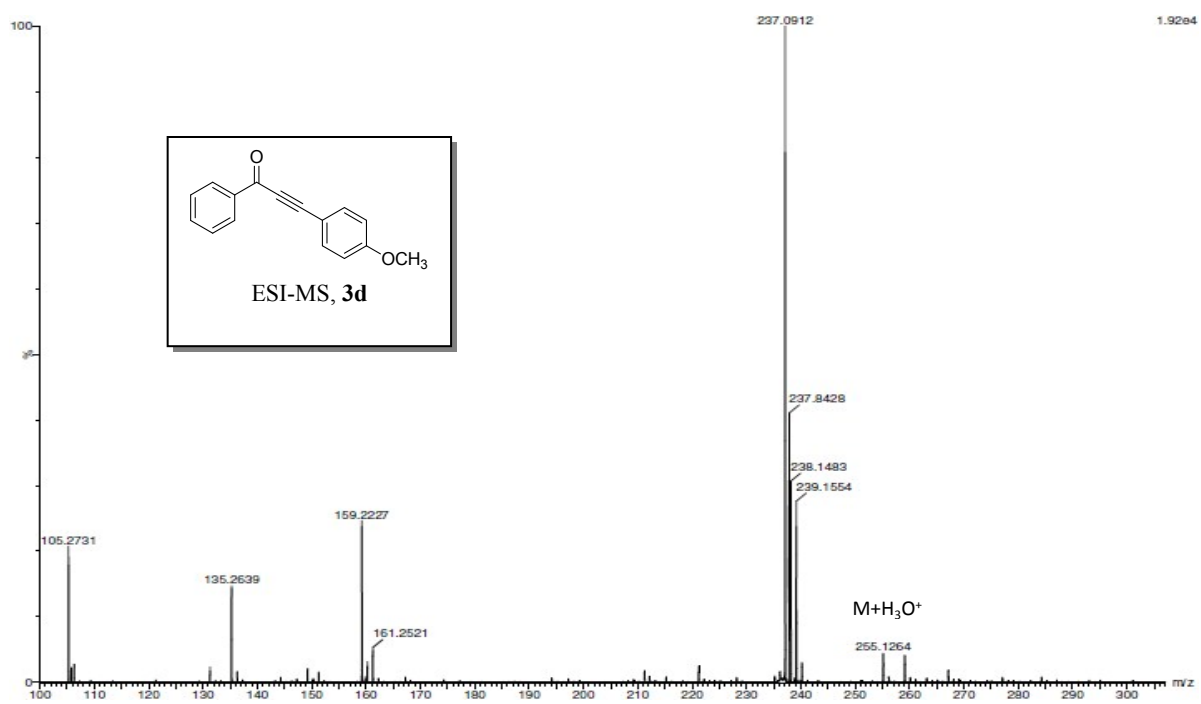
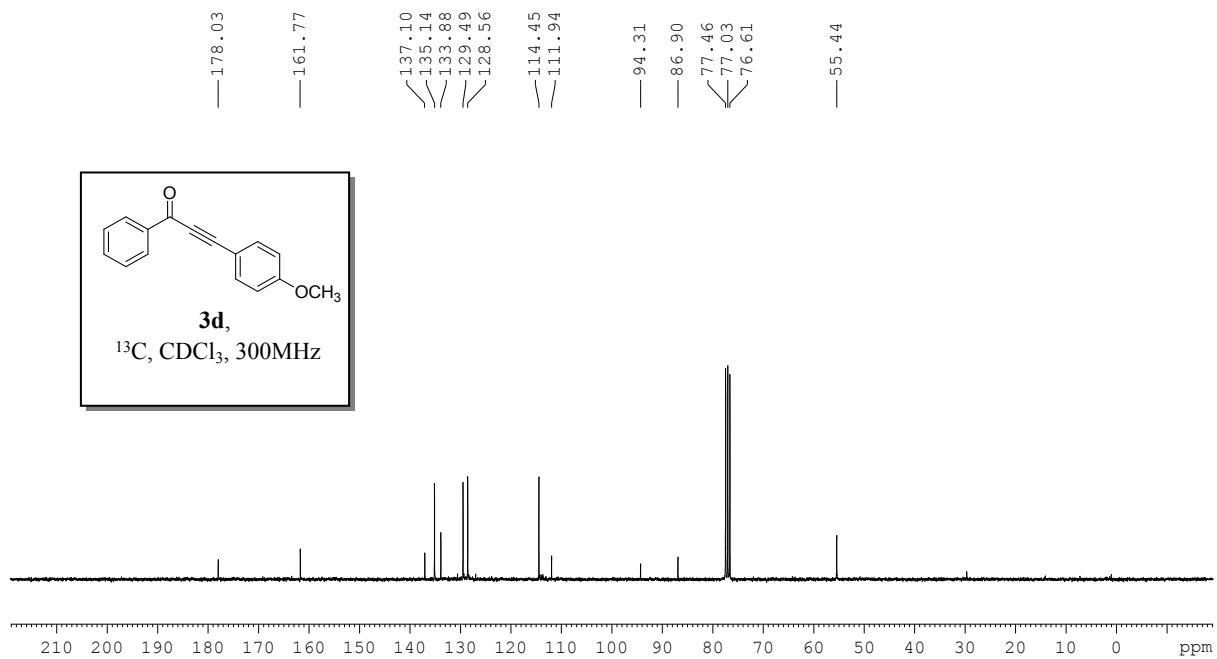




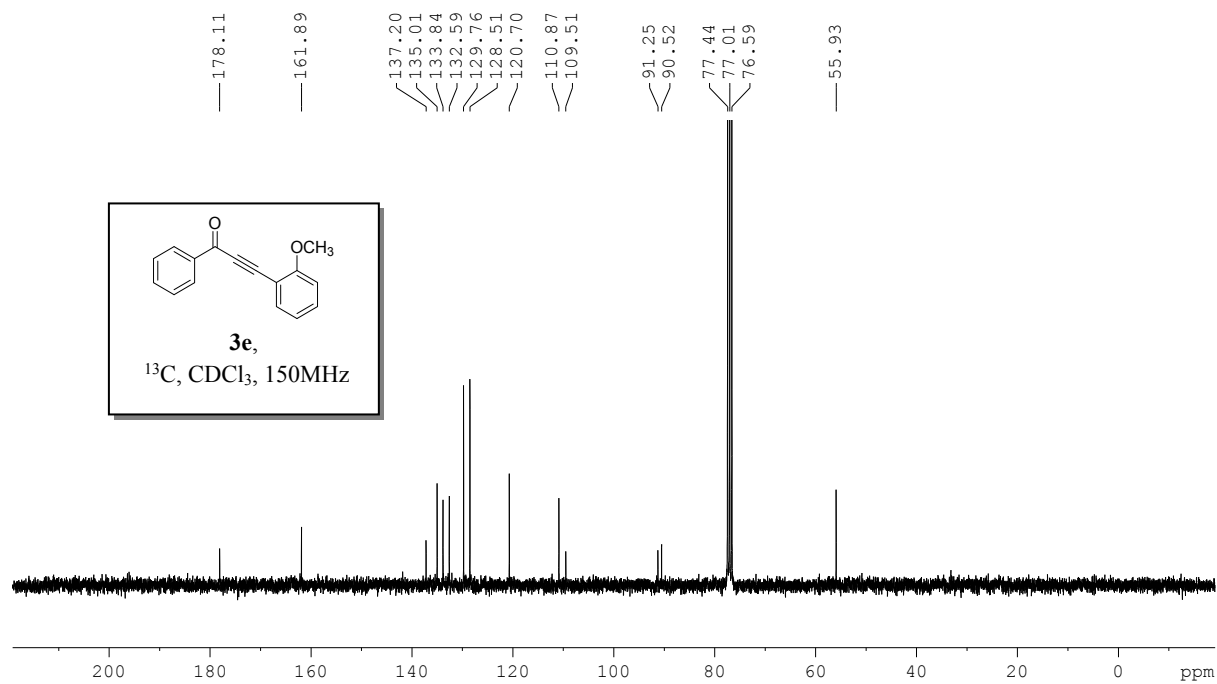
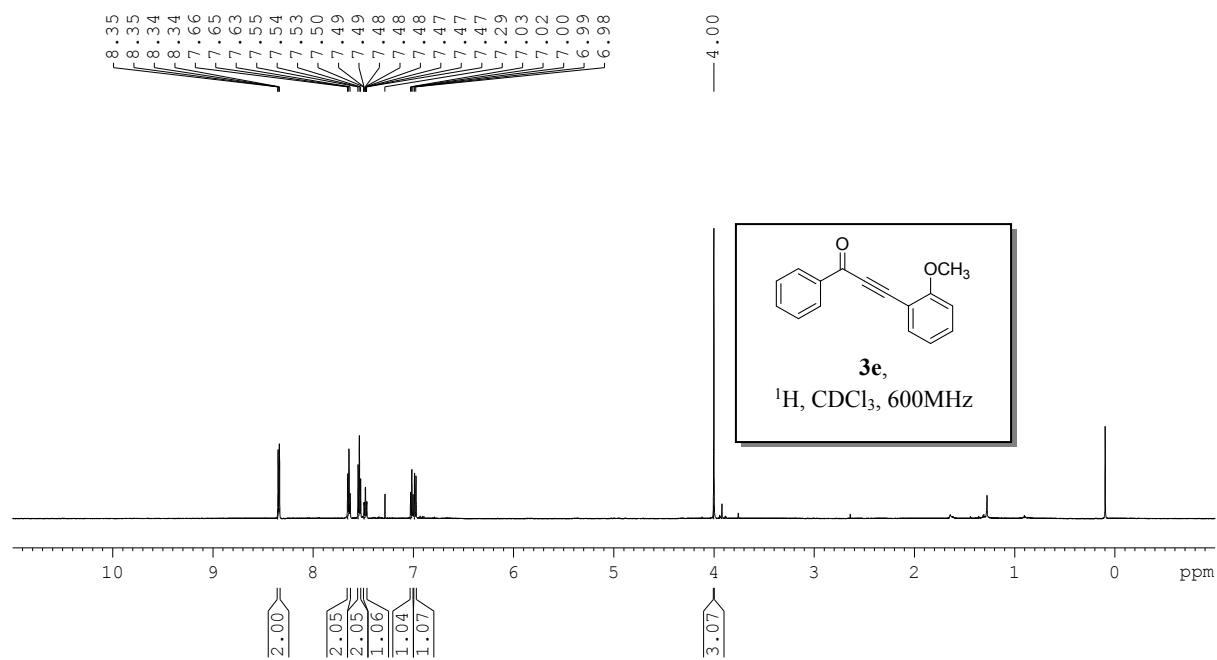
### 3-(4-Methoxyphenyl)-1-phenylprop-2-yn-1-one

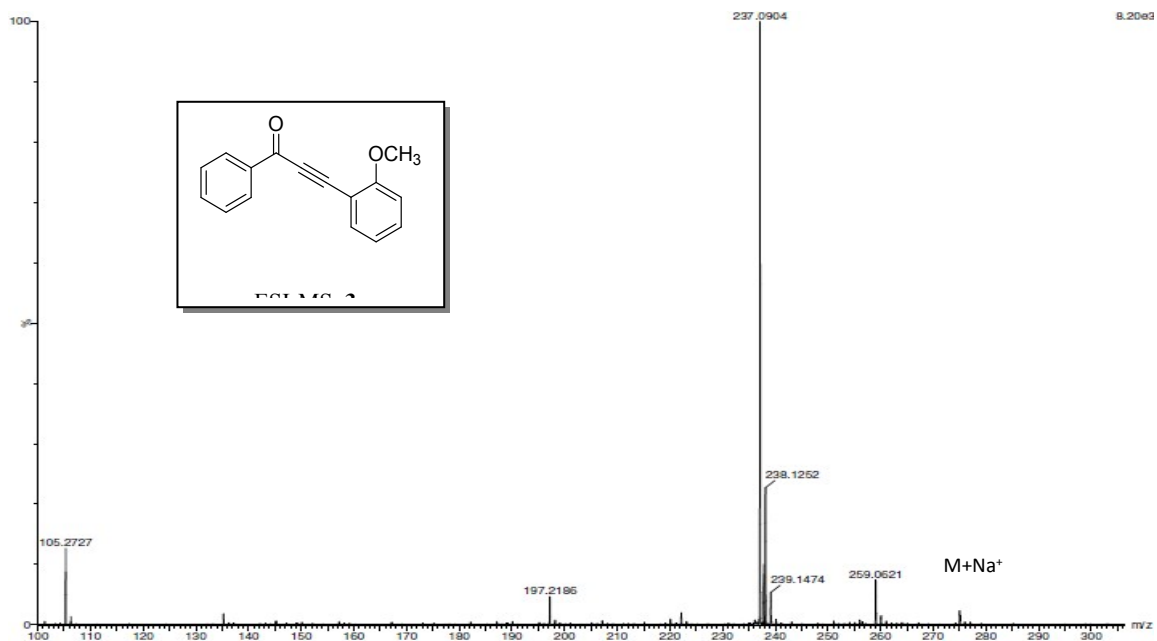




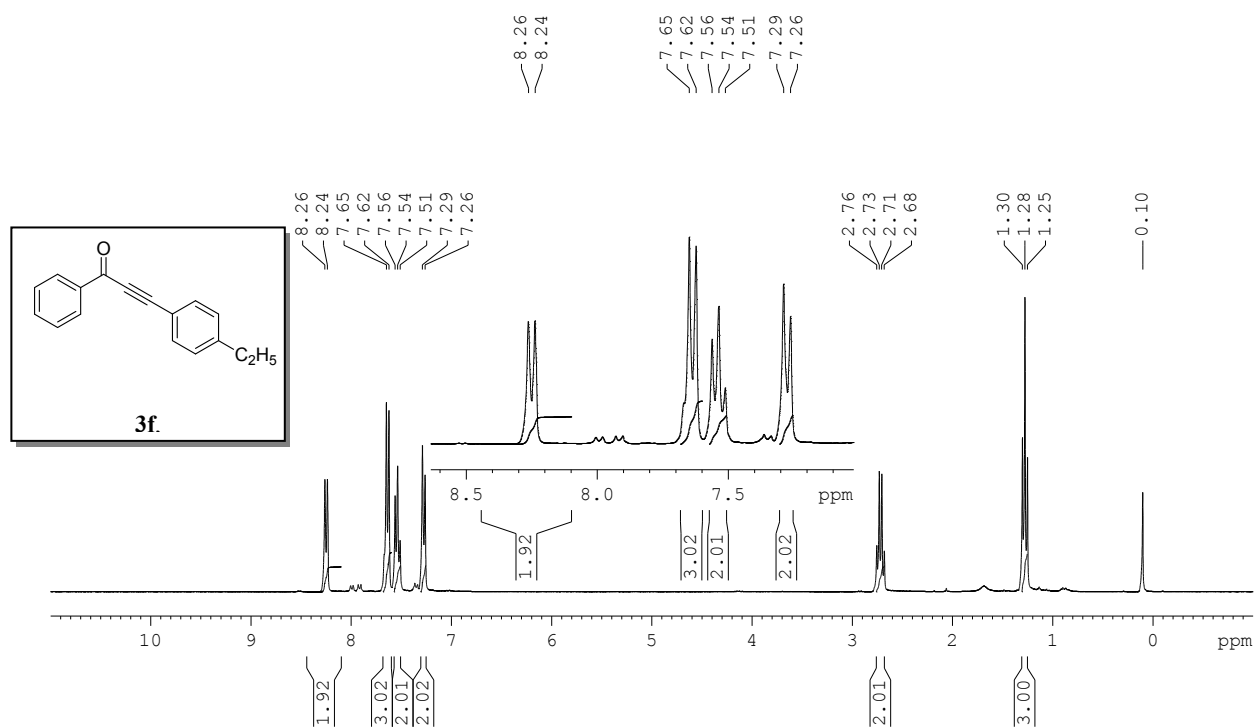


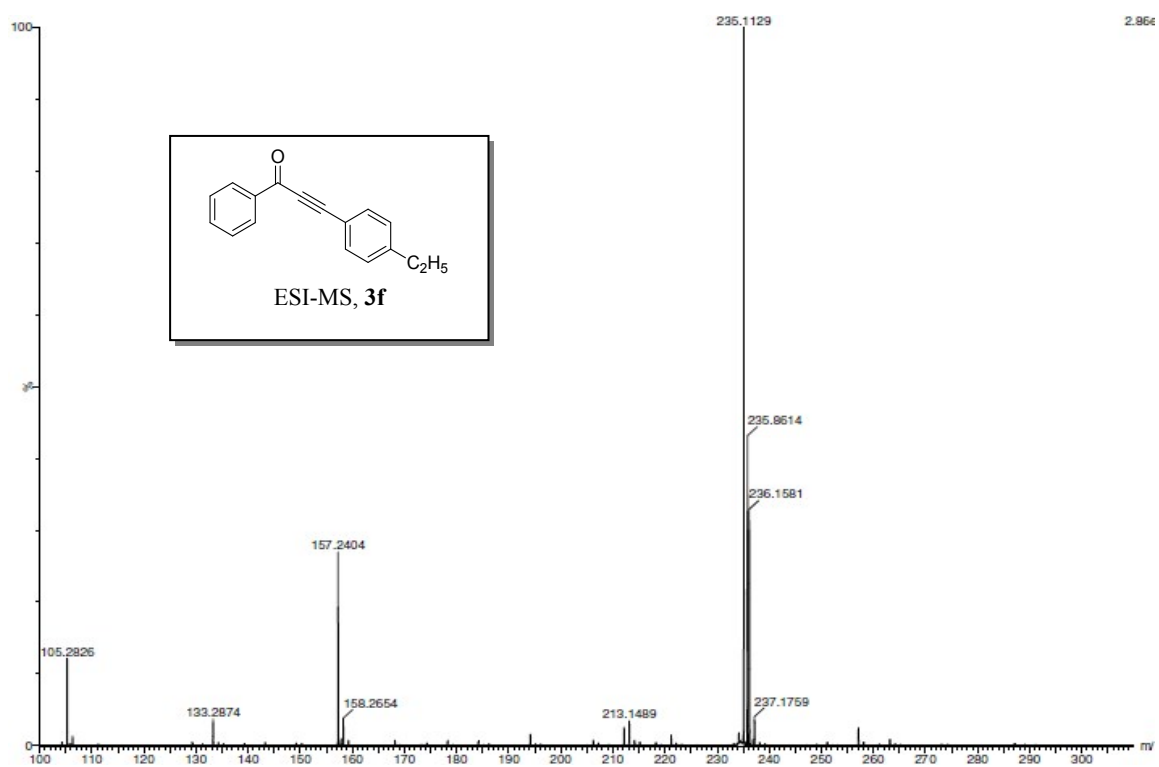
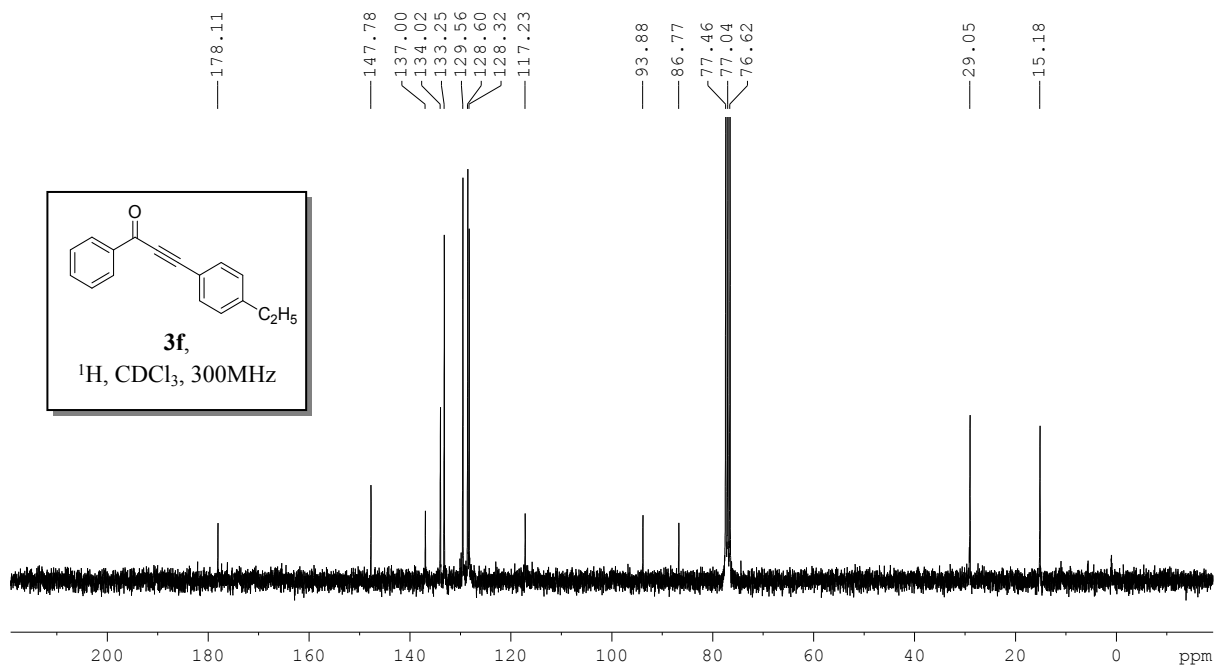
### 3-(2-Methoxyphenyl)-1-phenylprop-2-yn-1-one



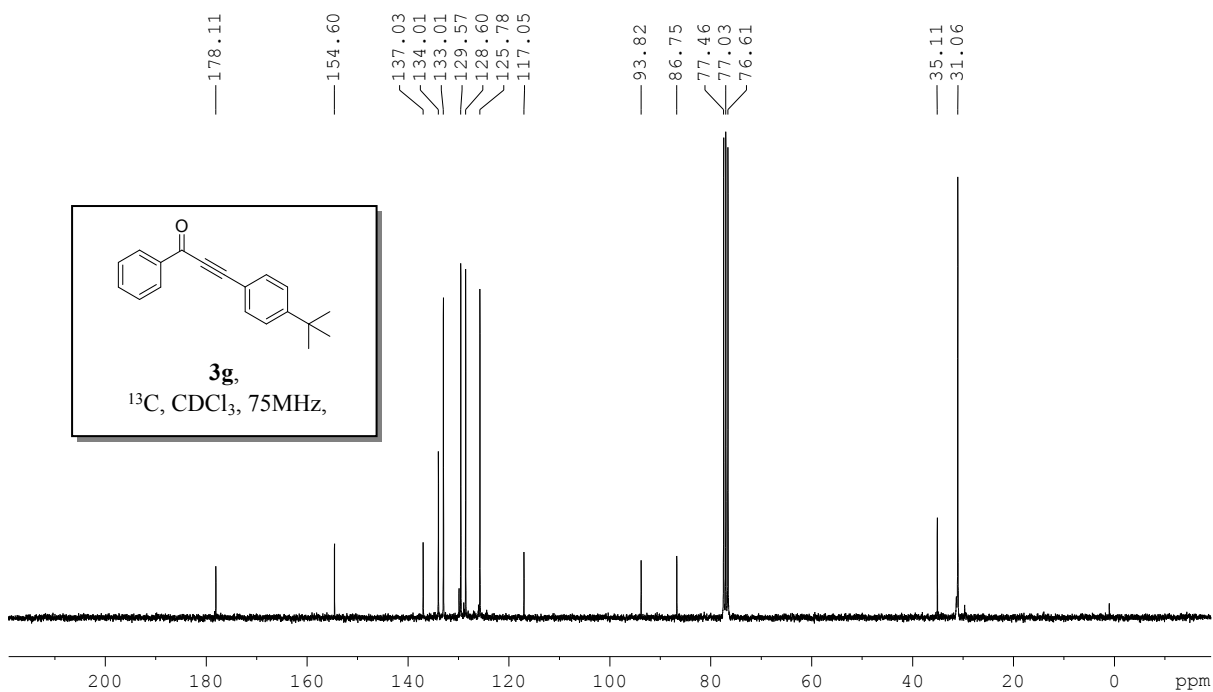
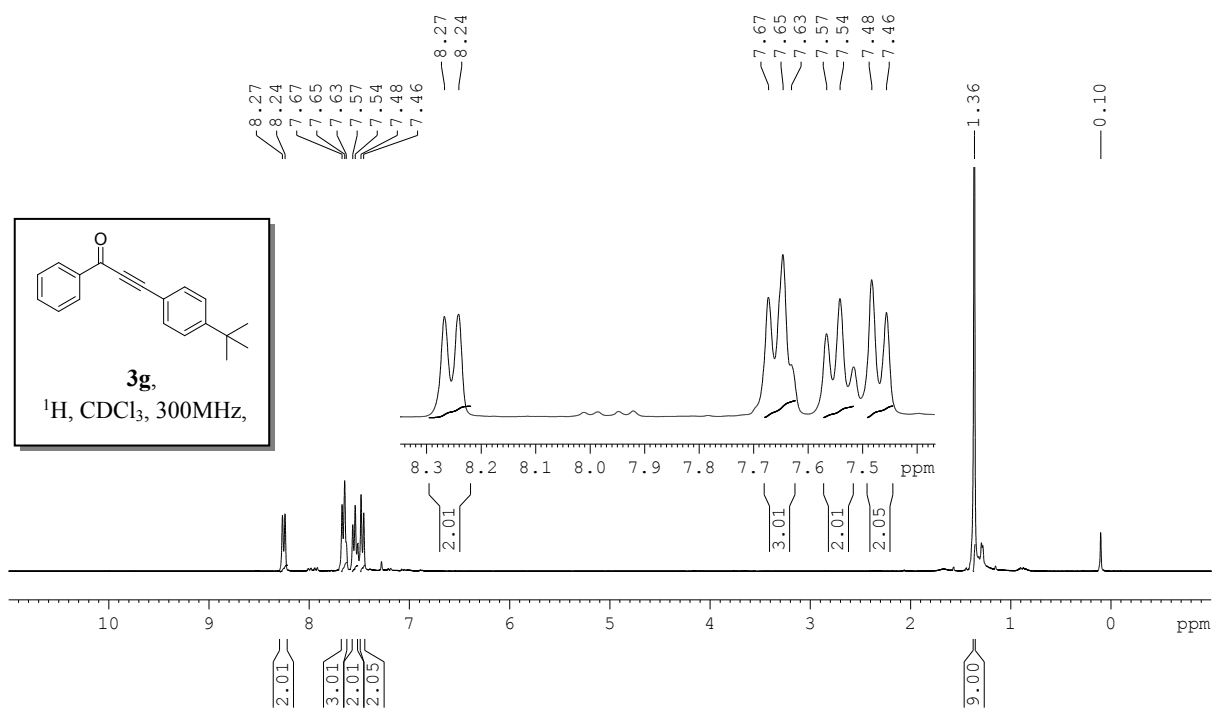


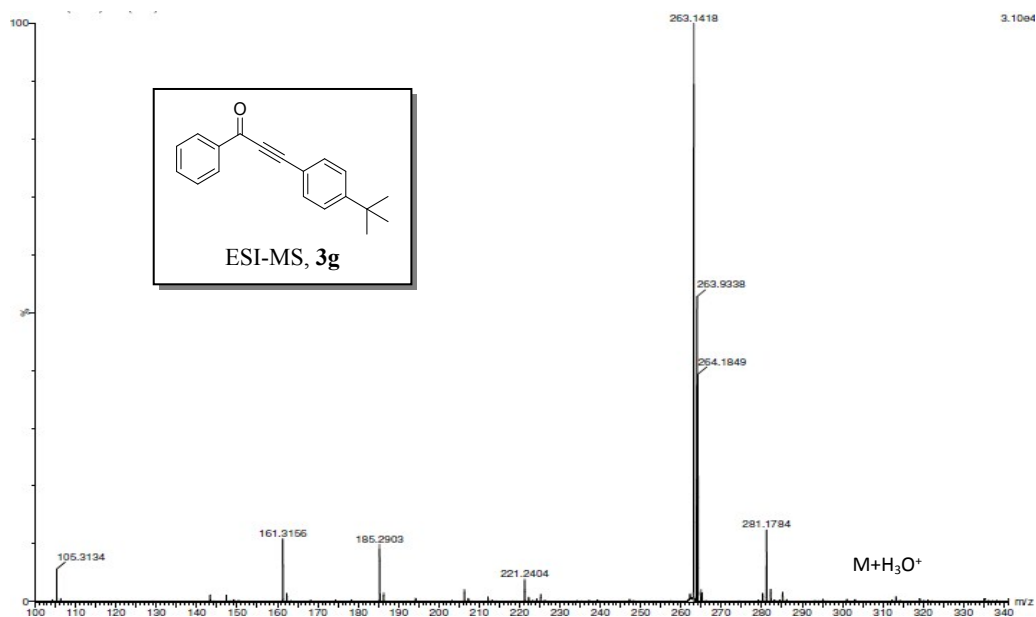
### 3-(4-Ethylphenyl)-1-phenylprop-2-yn-1-one



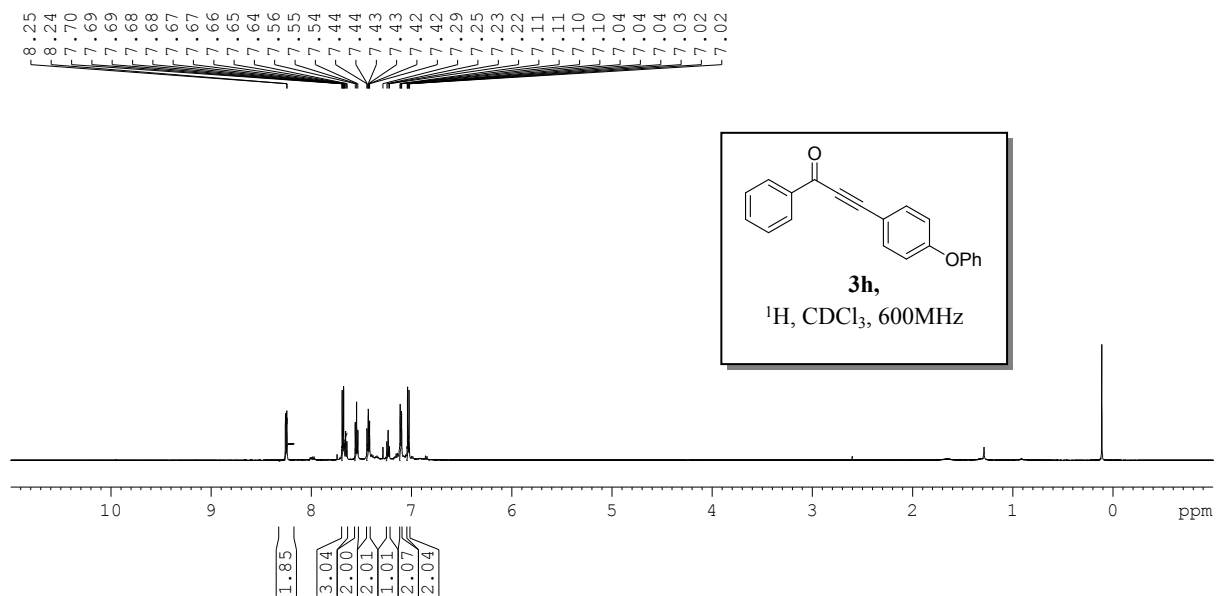


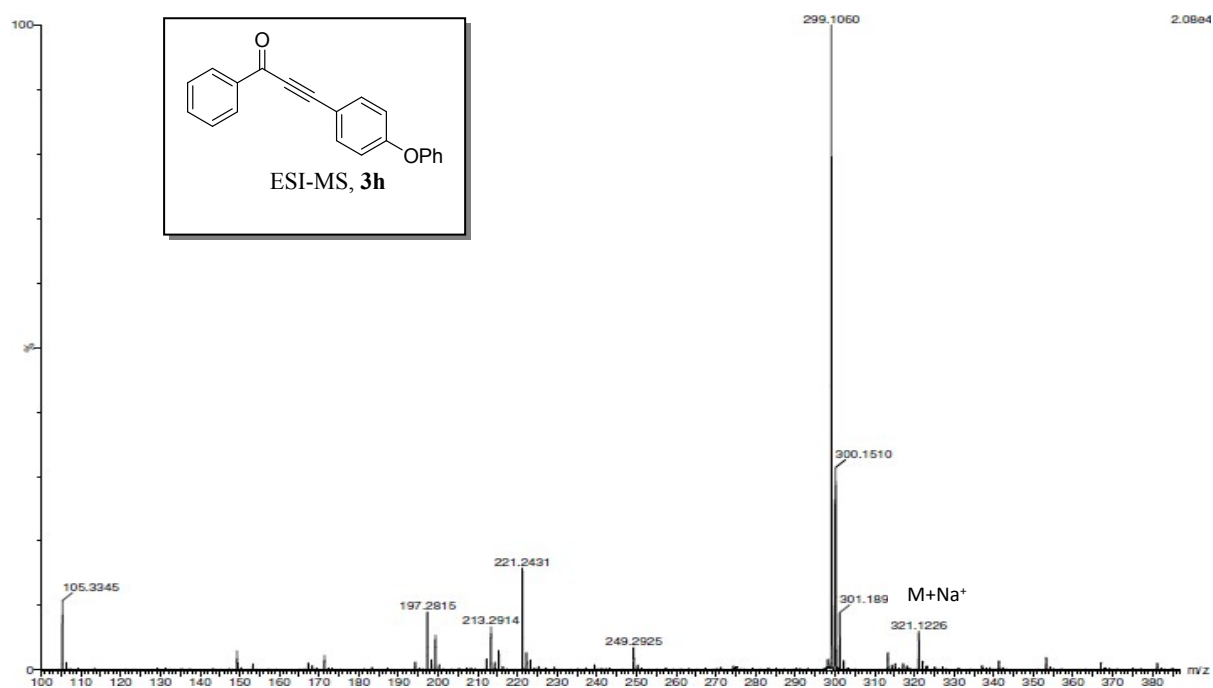
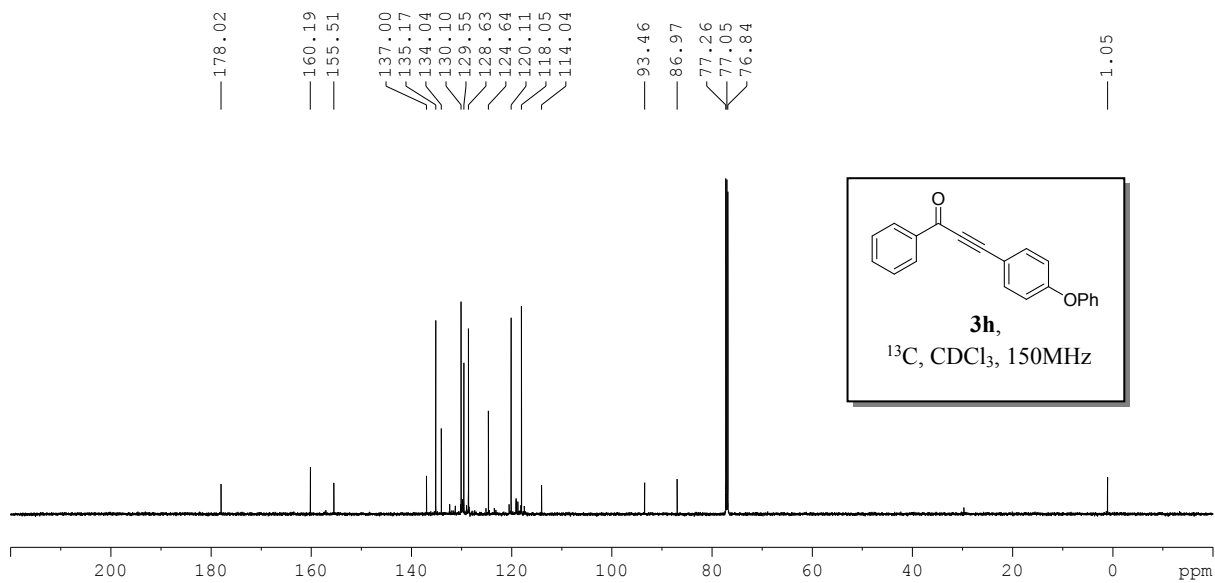
### 3-(4-(Tert-butyl)phenyl)-1-phenylprop-2-yn-1-one



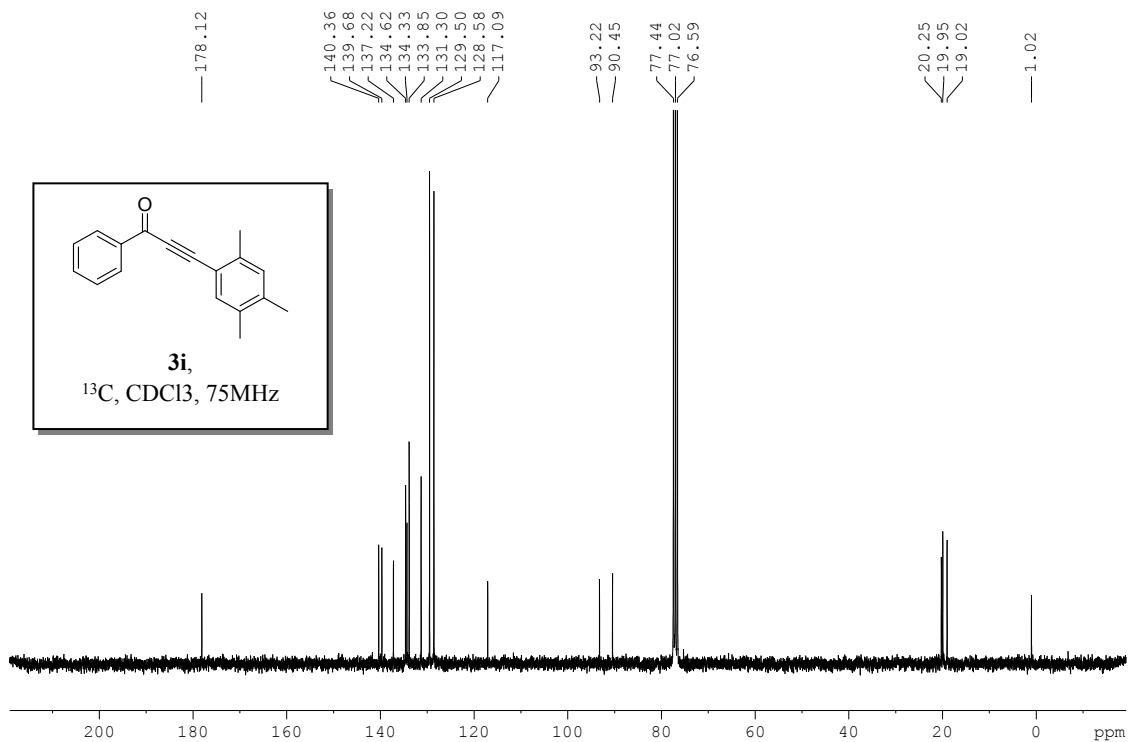
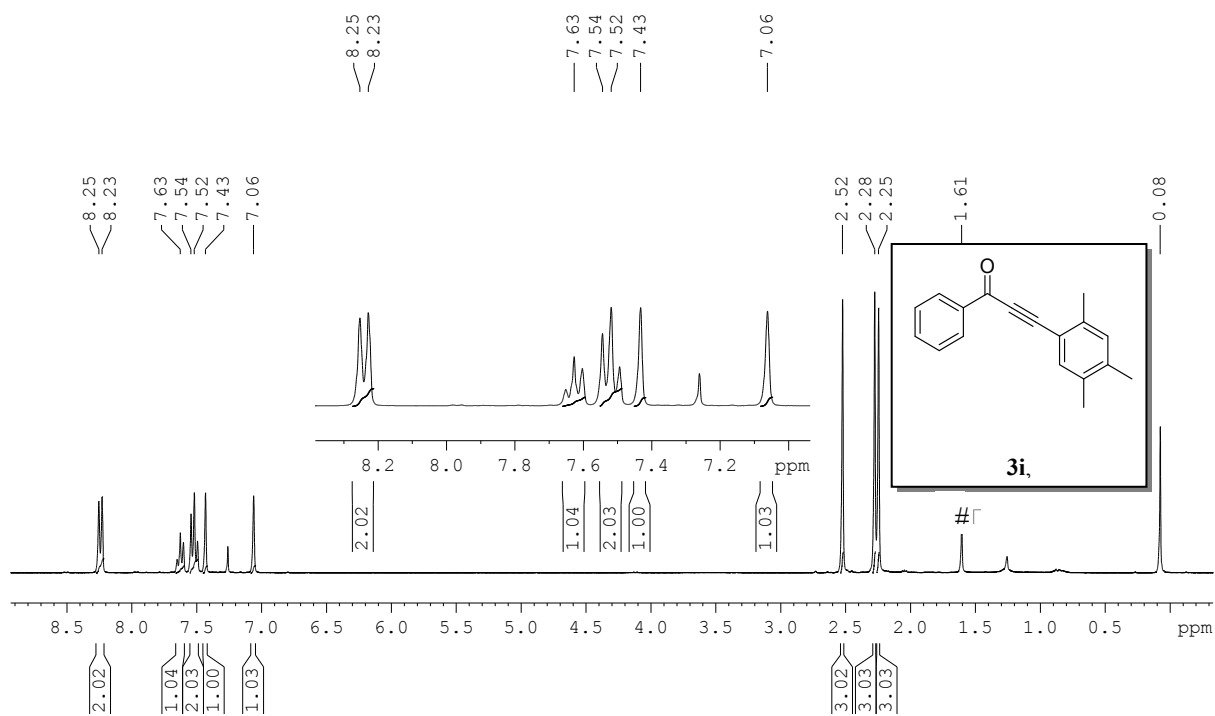


### 3-(4-Phenoxyphenyl)-1-phenylprop-2-yn-1-one

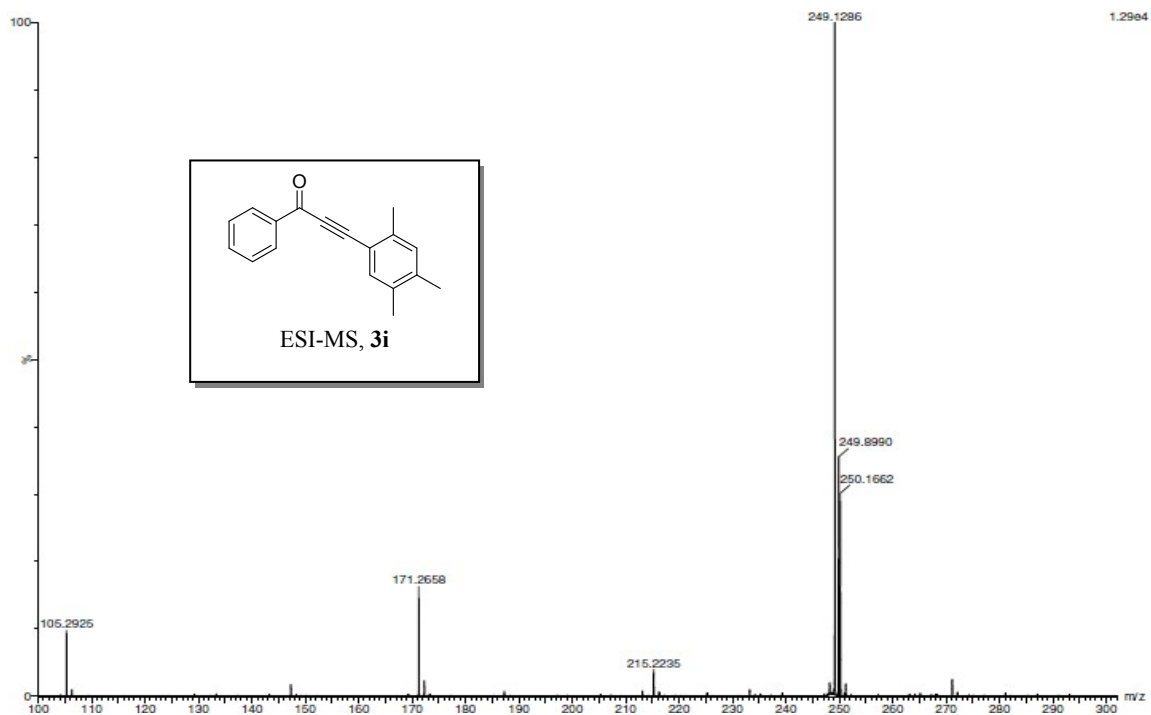




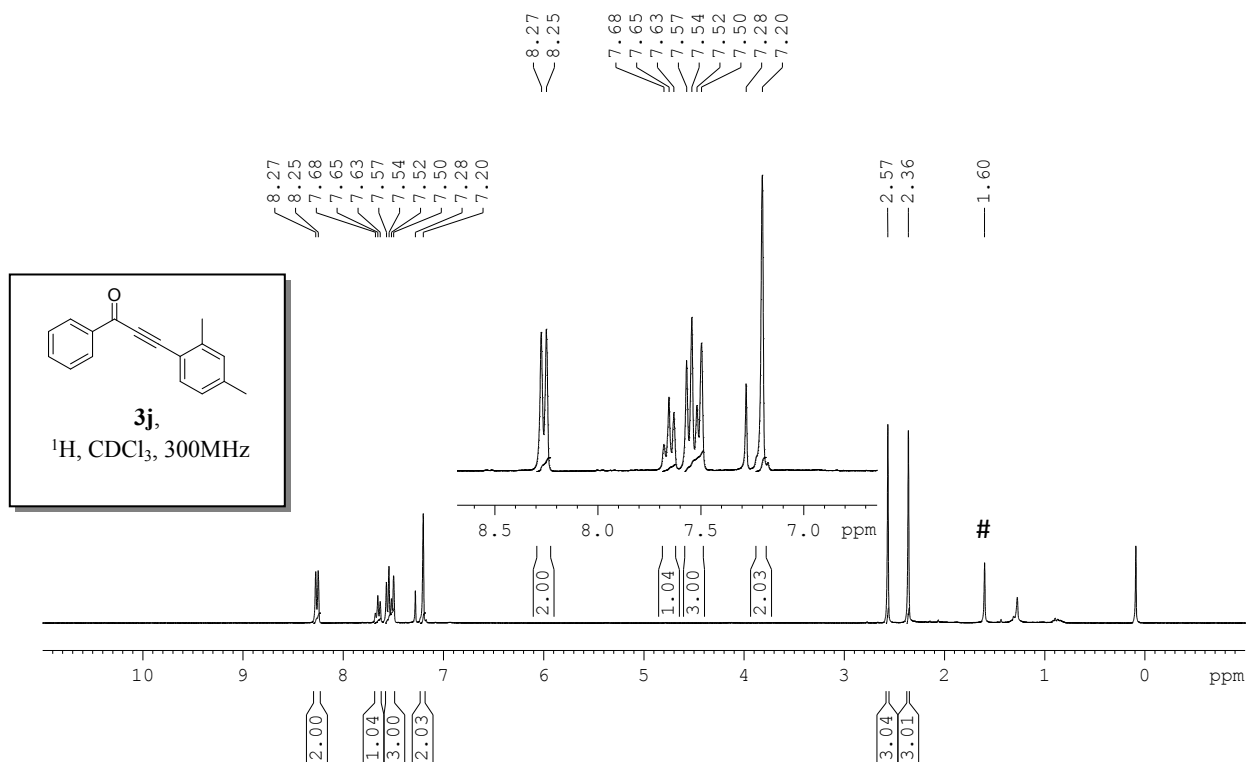
# 1-Phenyl-3-(2,4,5-trimethylphenyl)prop-2-yn-1-one

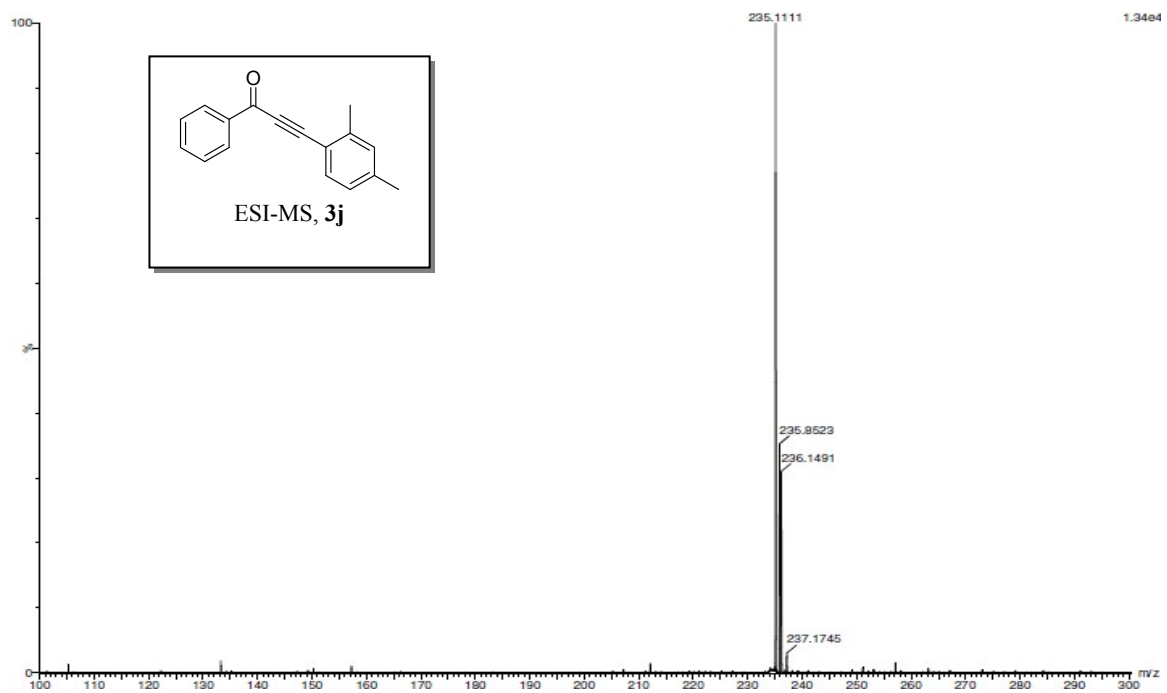
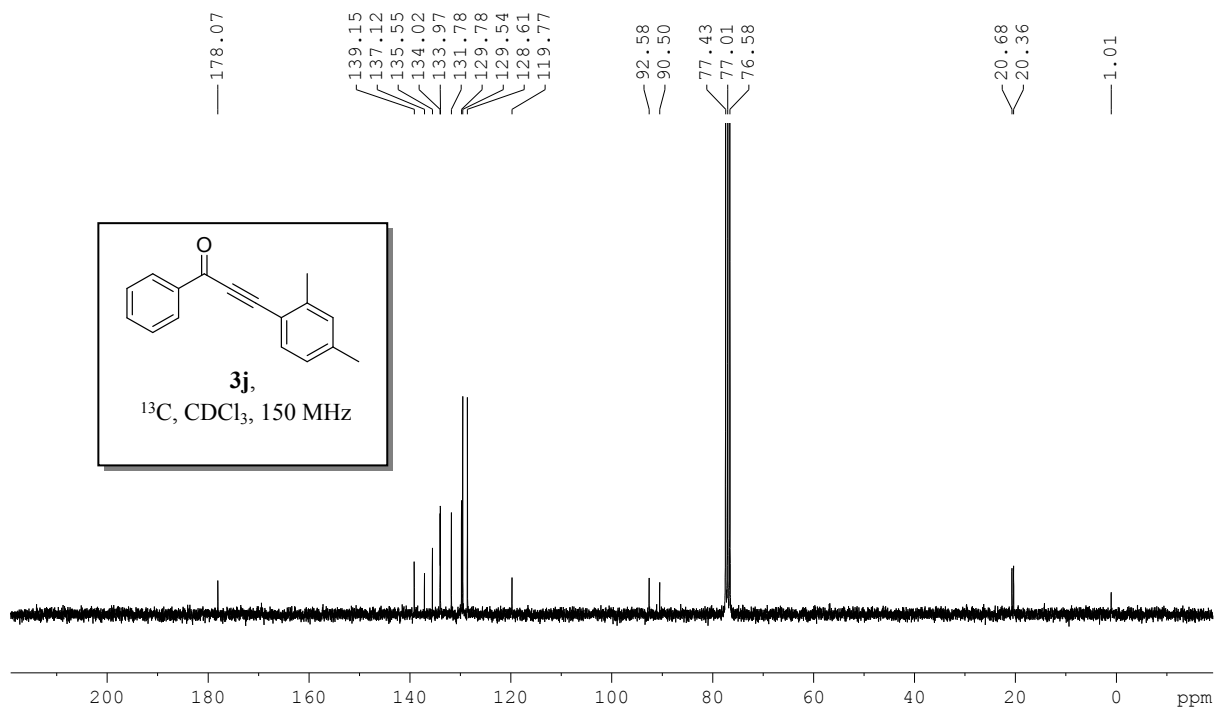




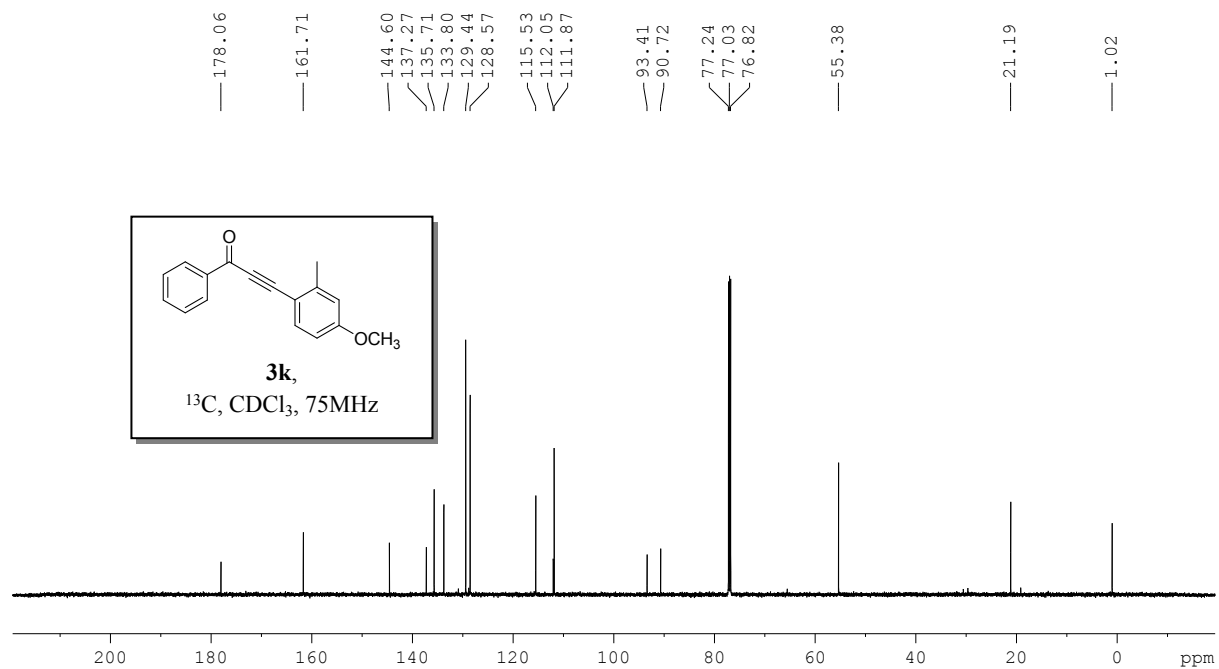
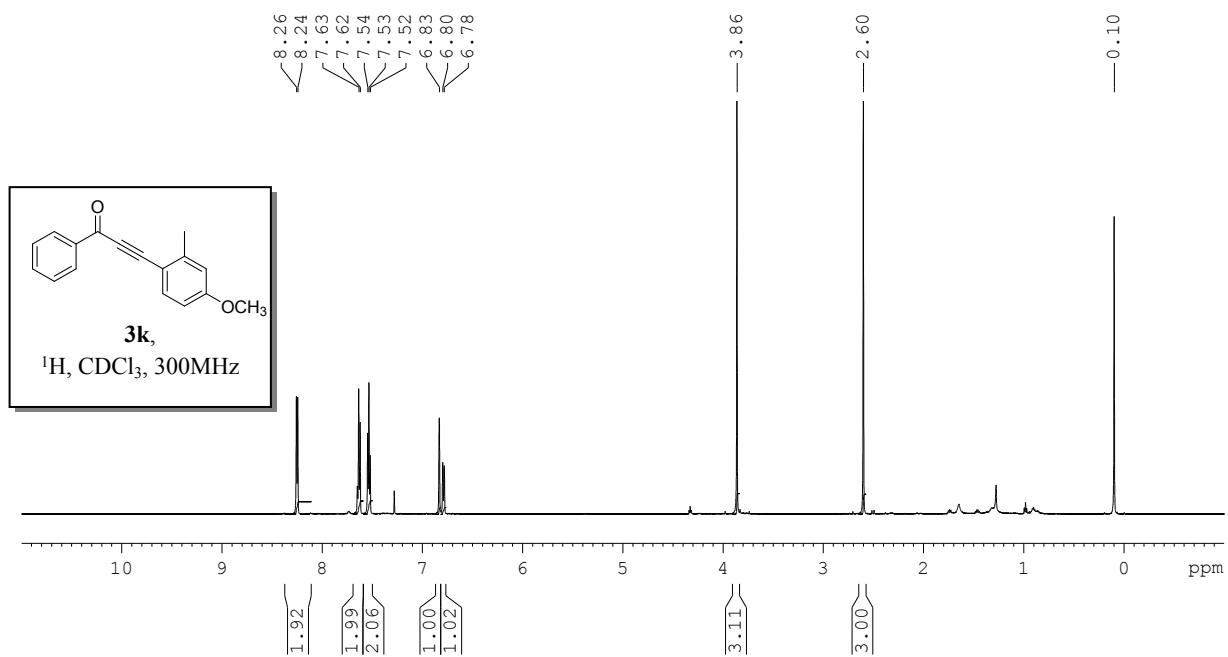


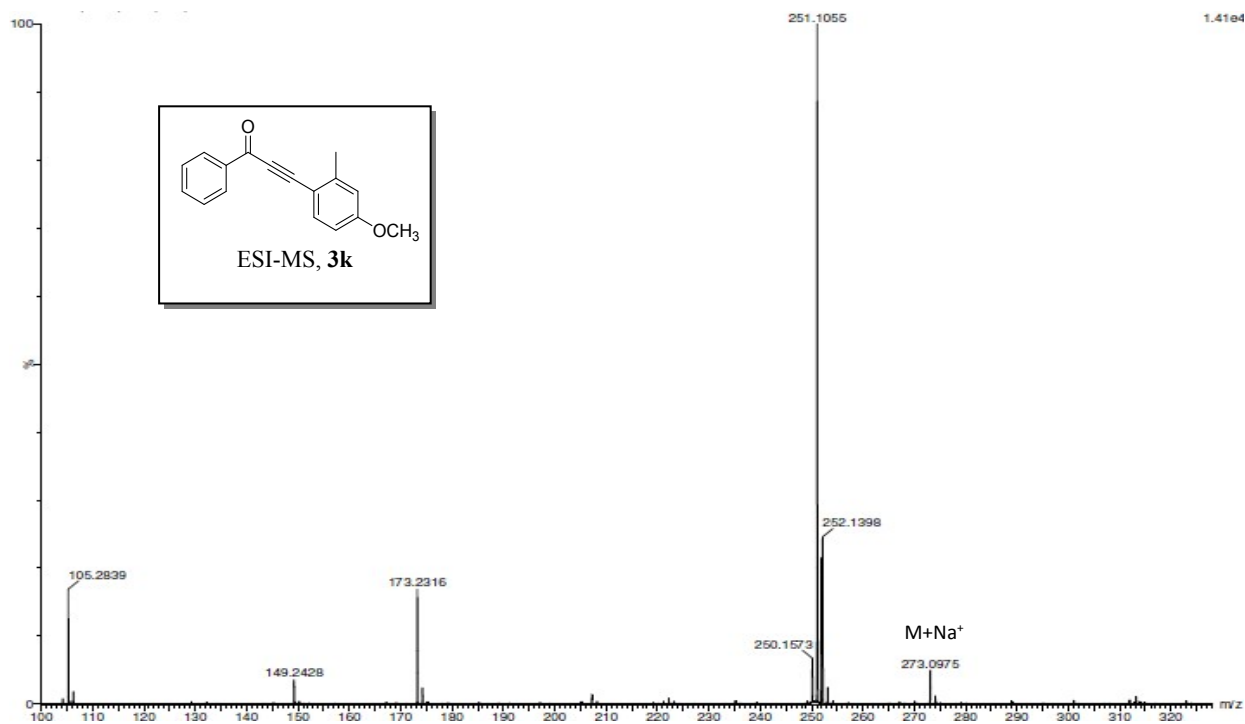
### 3-(2, 4-Dimethylphenyl)-1-phenylprop-2-yn-1-one



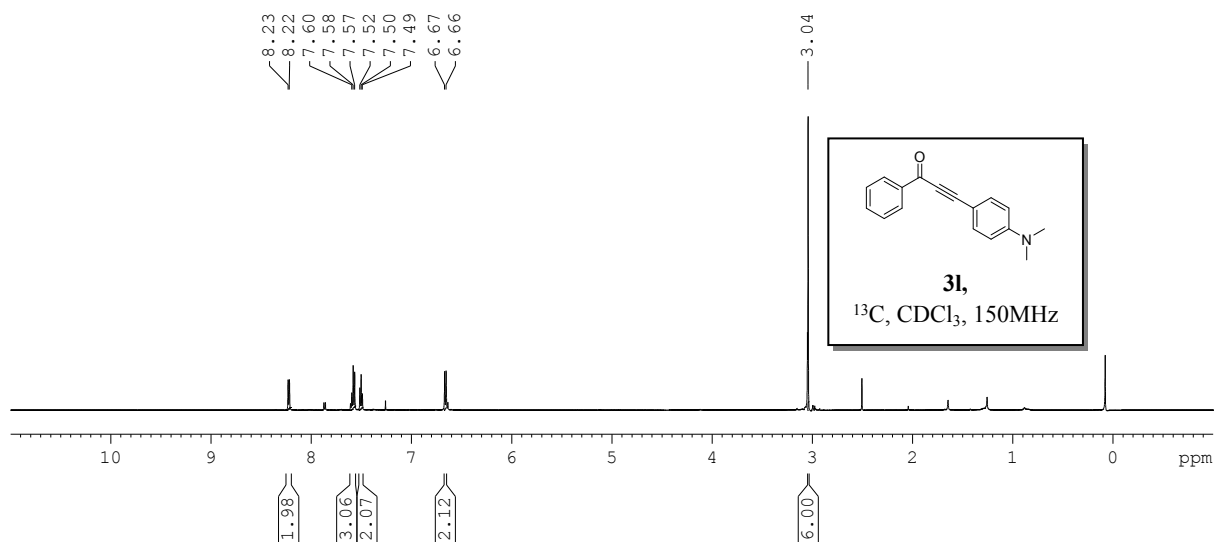


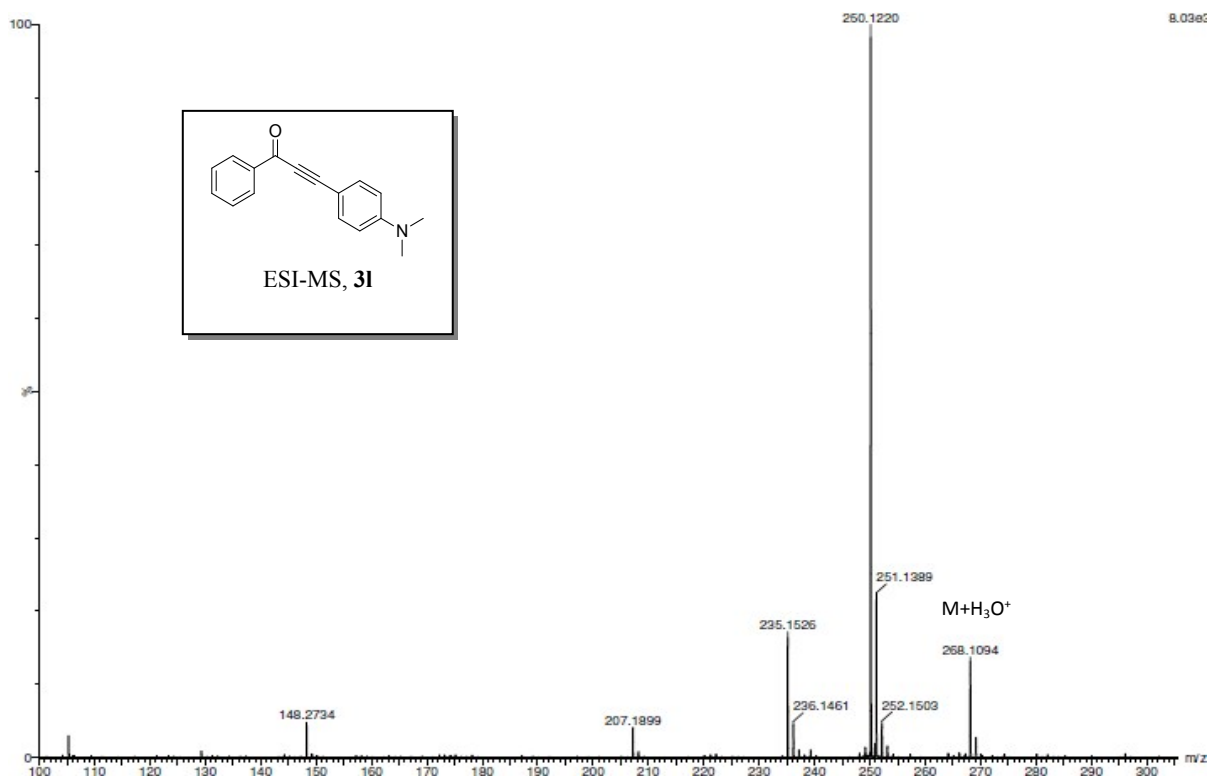
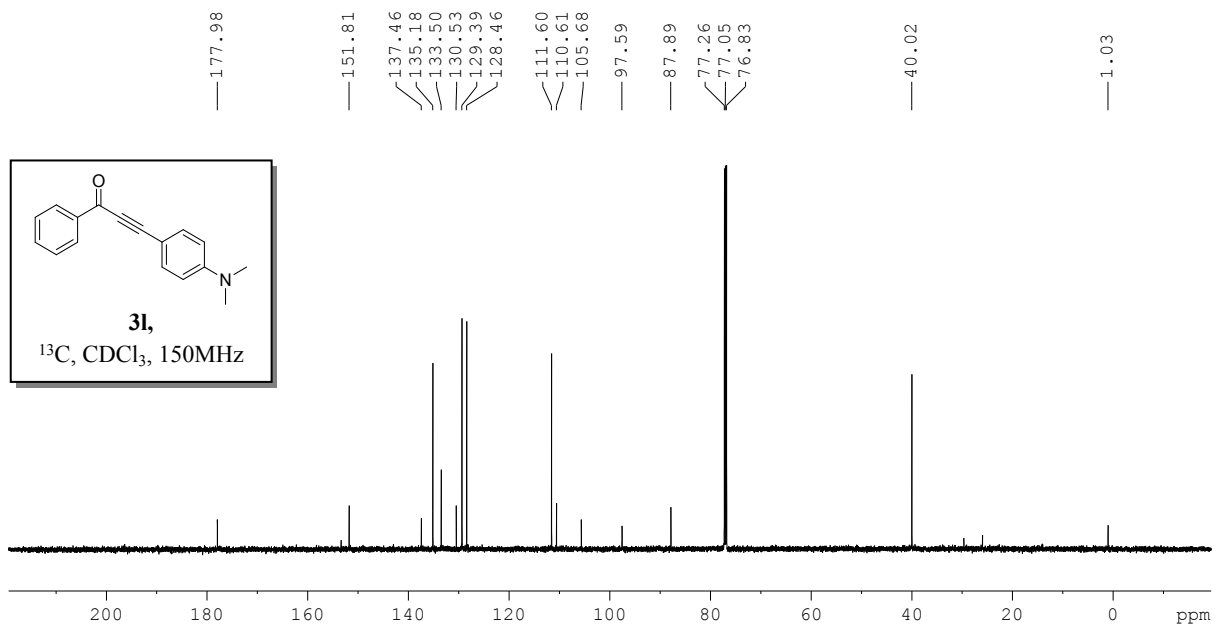
### 3-(4-methoxy-2-methylphenyl)-1-phenylprop-2-yn-1-one



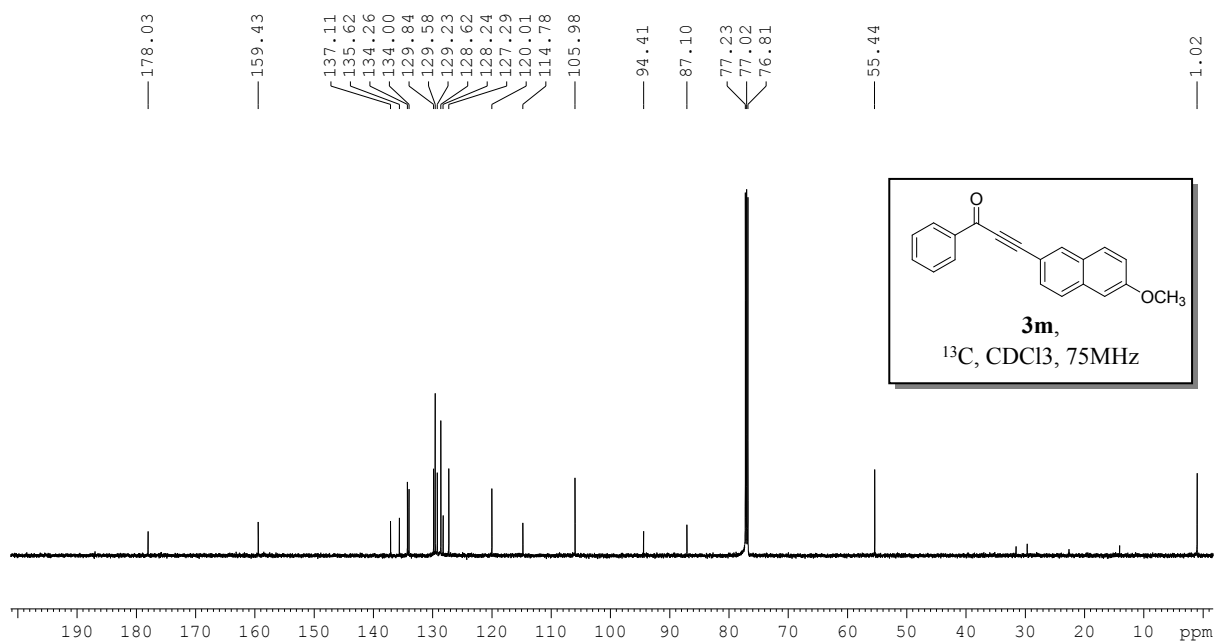
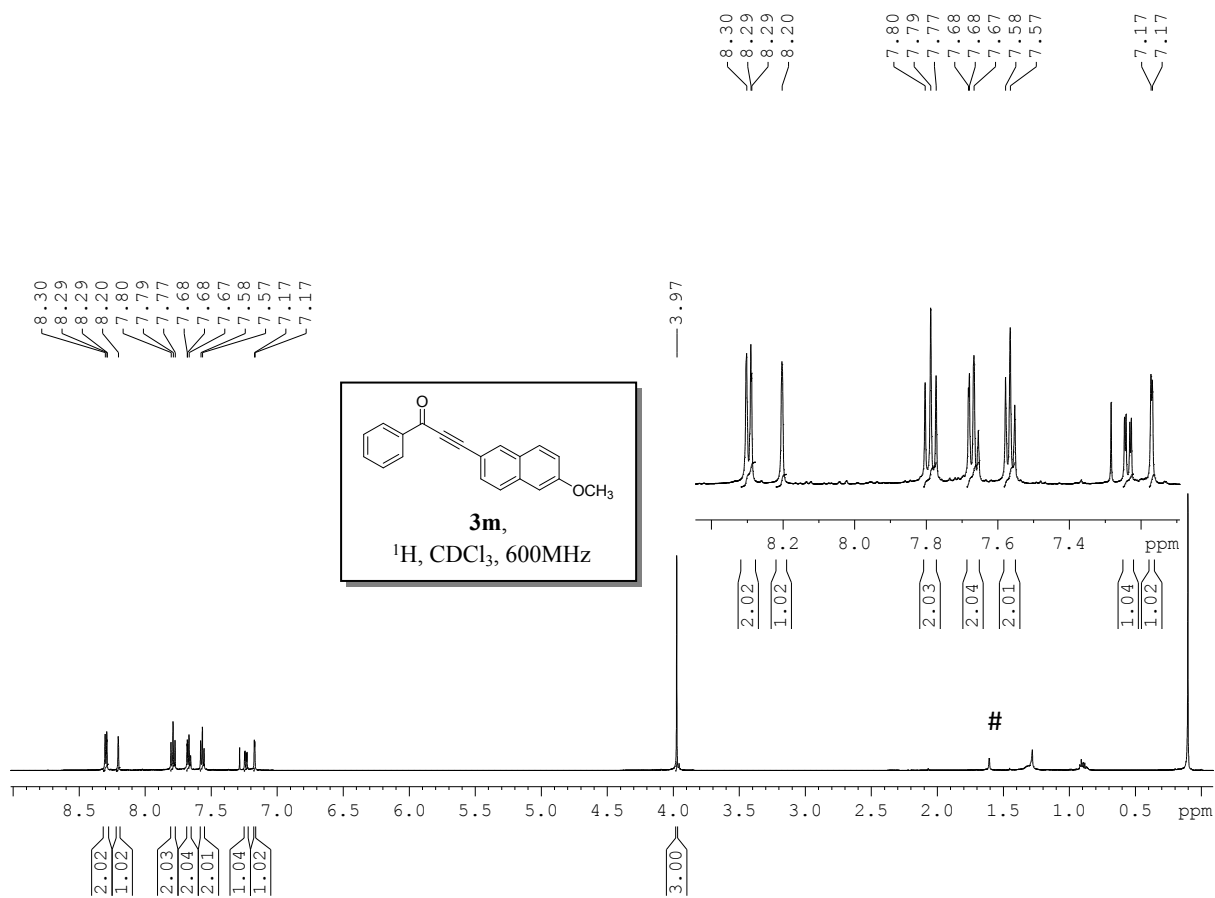


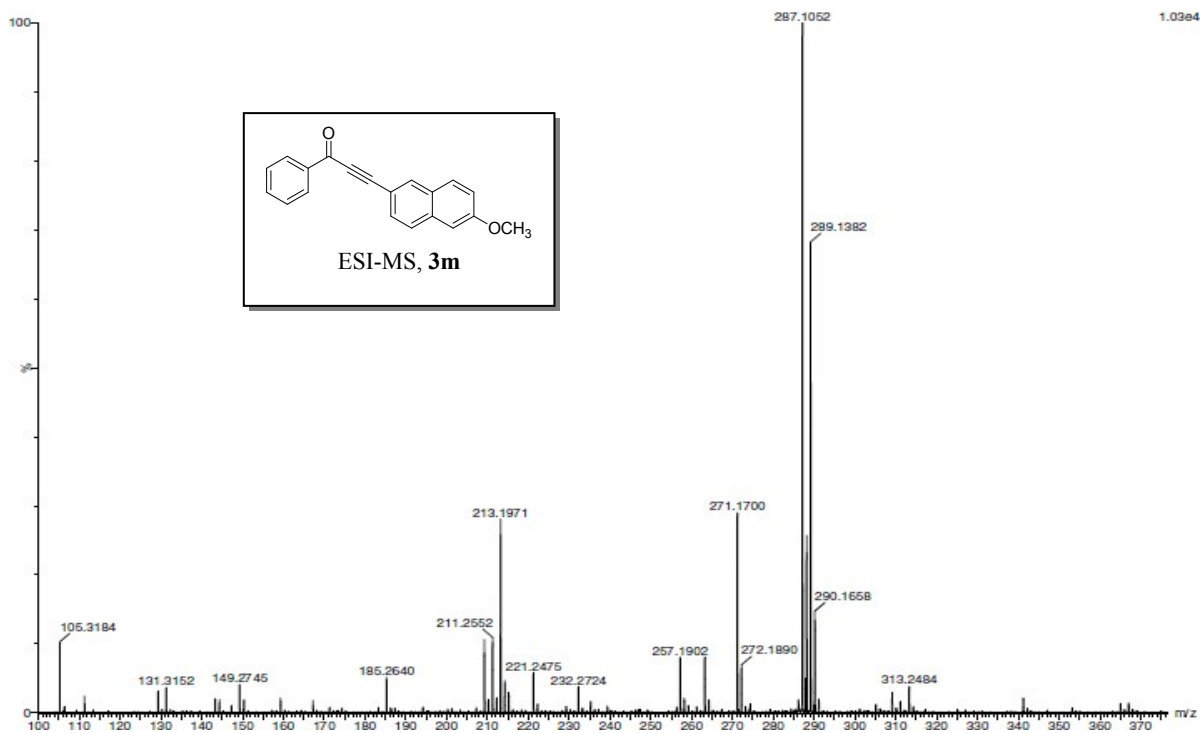
### 3-(4-(Dimethylamino)phenyl)-1-phenylprop-2-yn-1-one



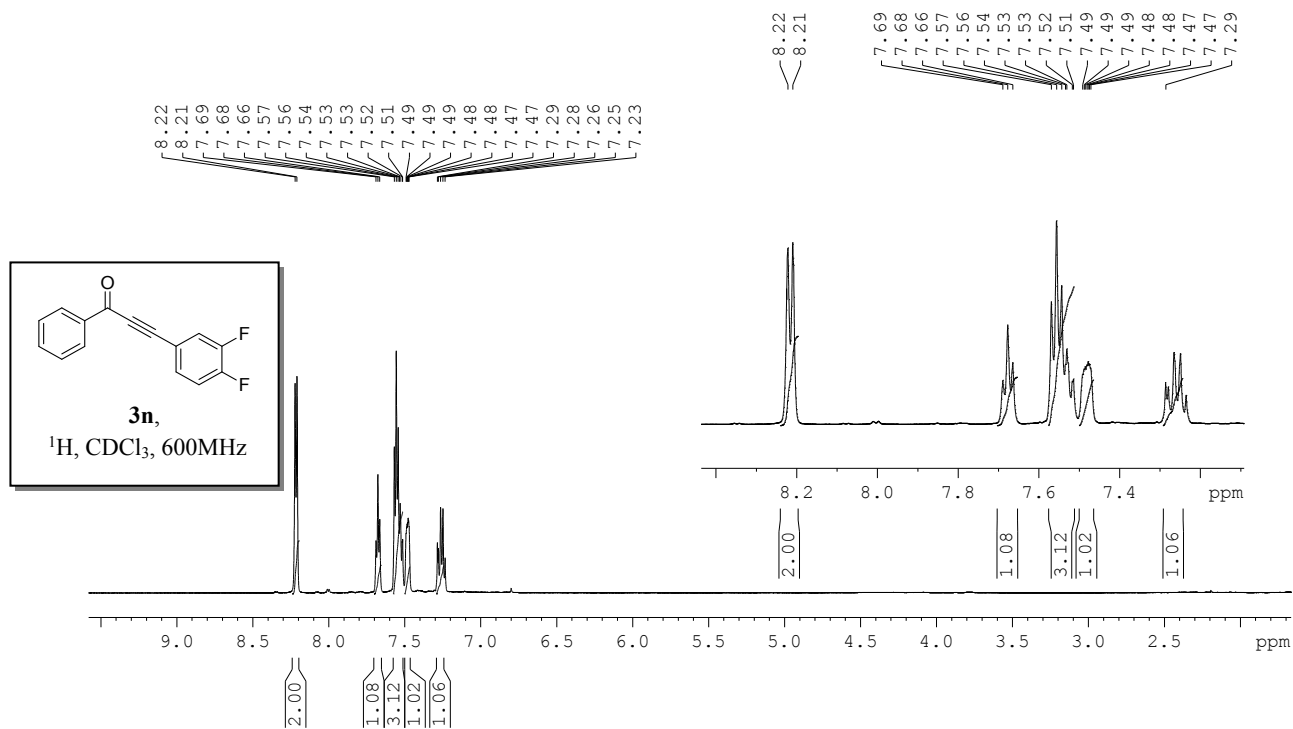


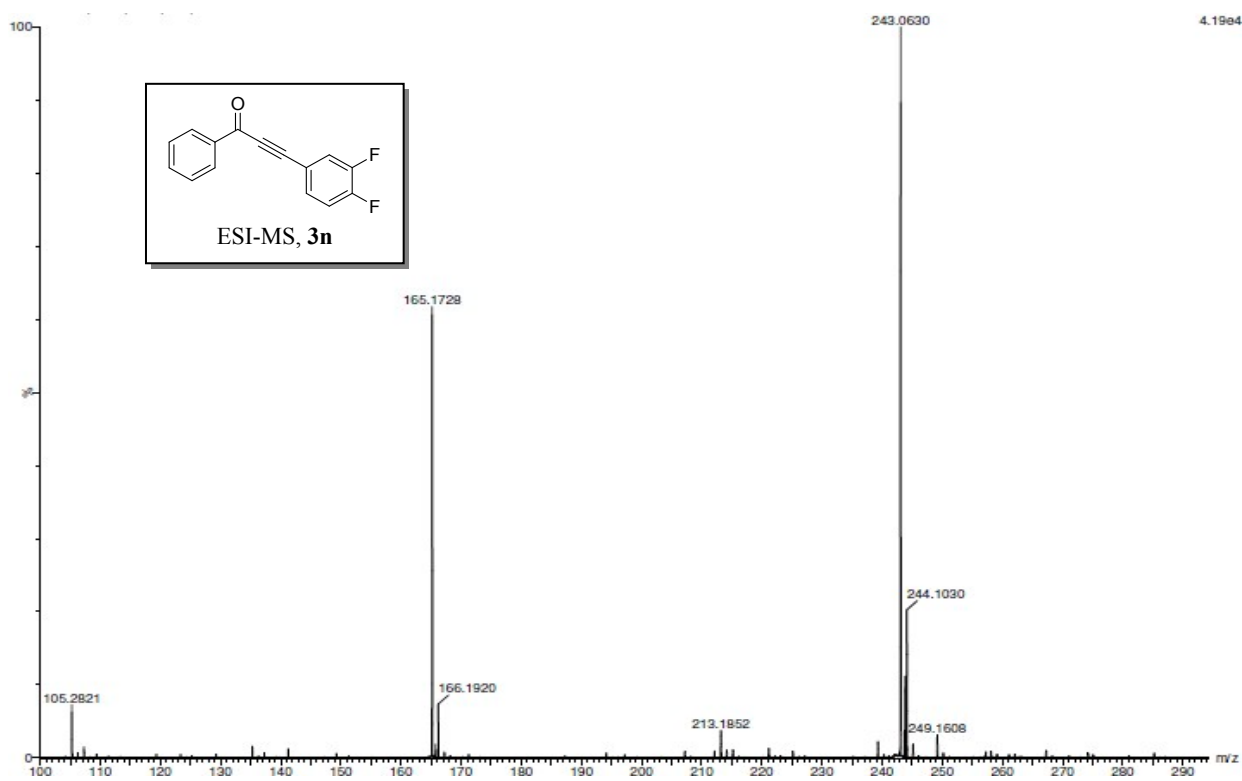
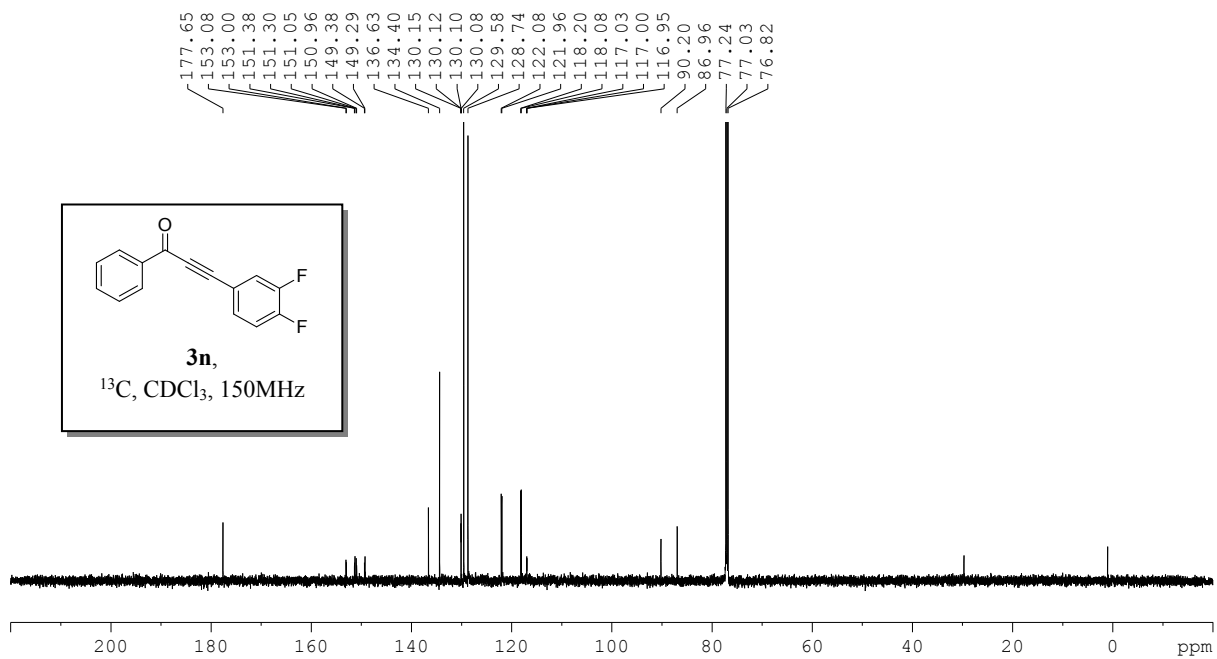
### 3-(6-Methoxynaphthalen-2-yl)-1-phenylprop-2-yn-1-one





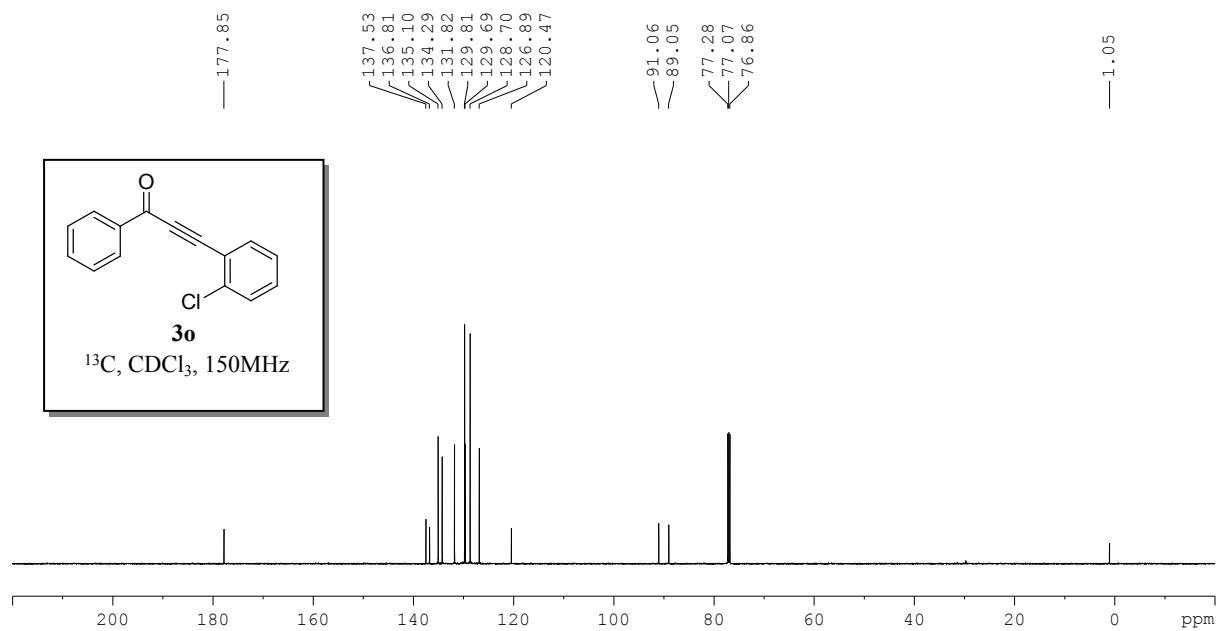
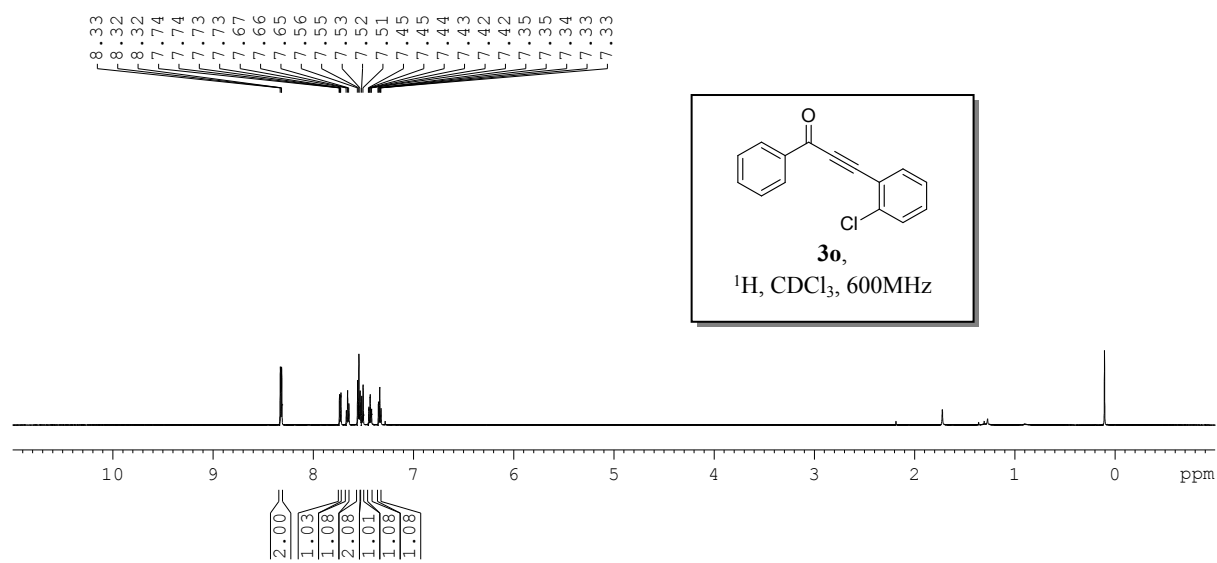
### 3-(3, 4-Difluorophenyl)-1-phenylprop-2-yn-1-one

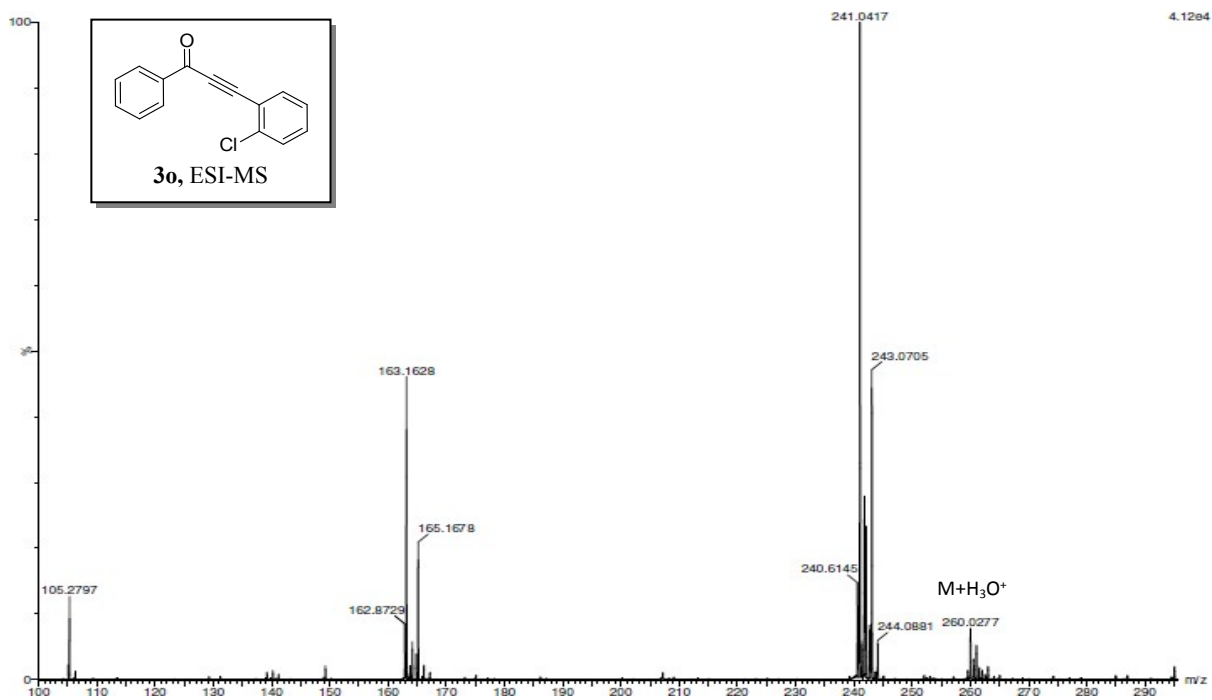




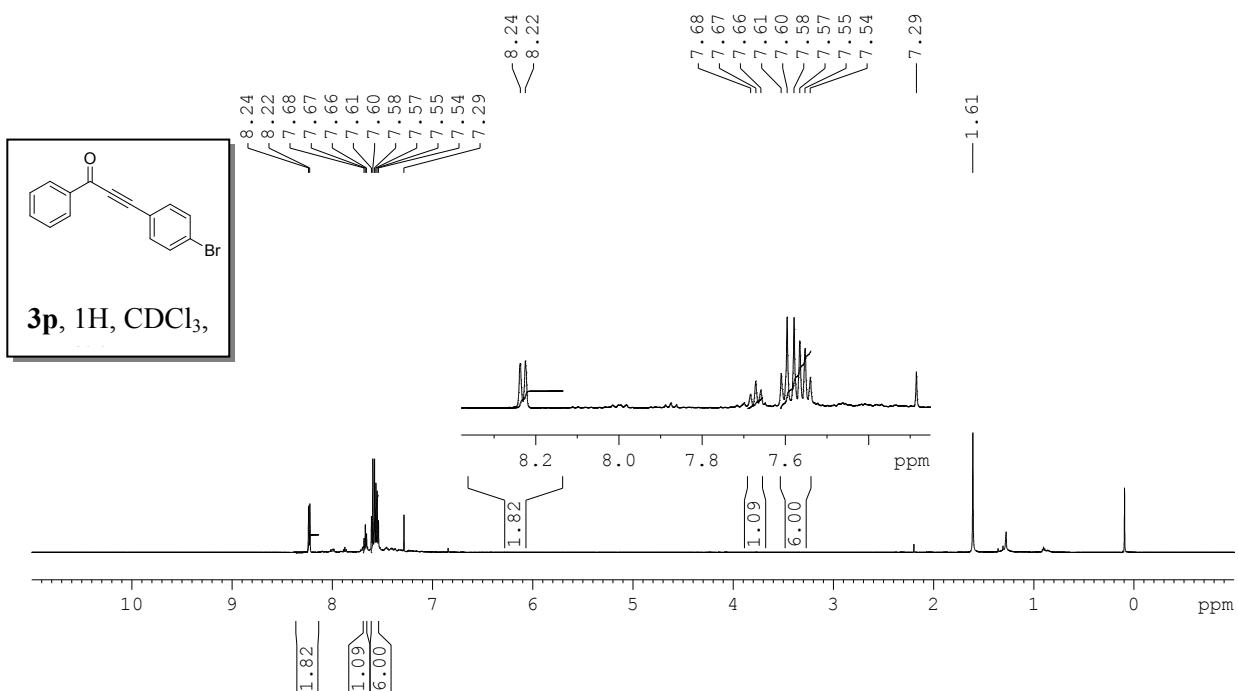


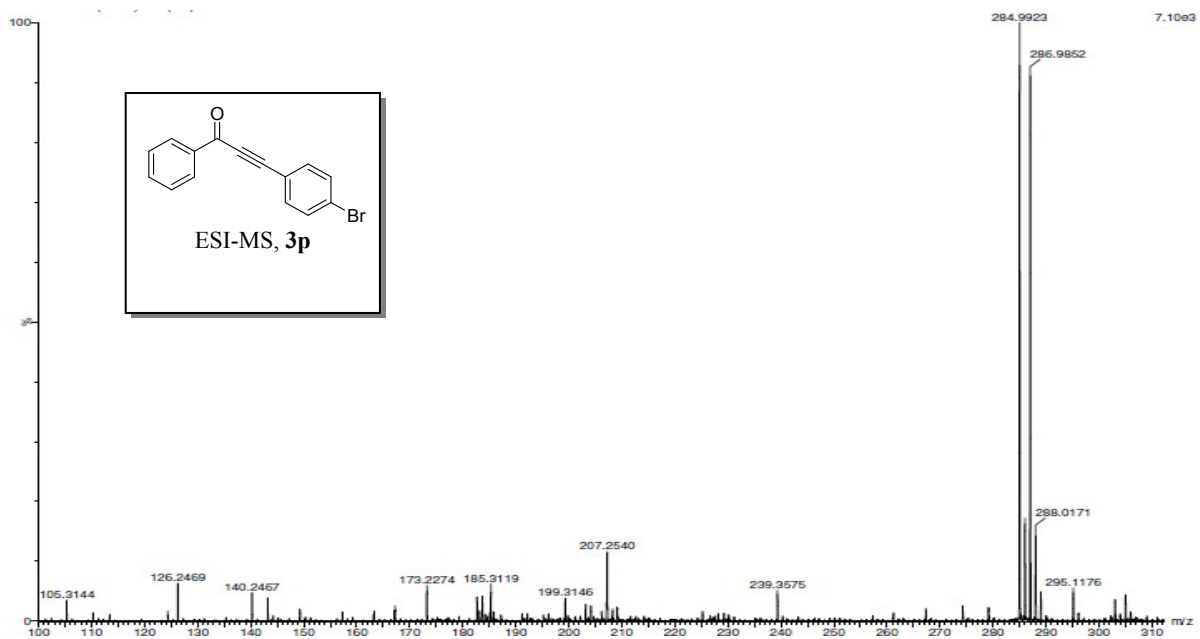
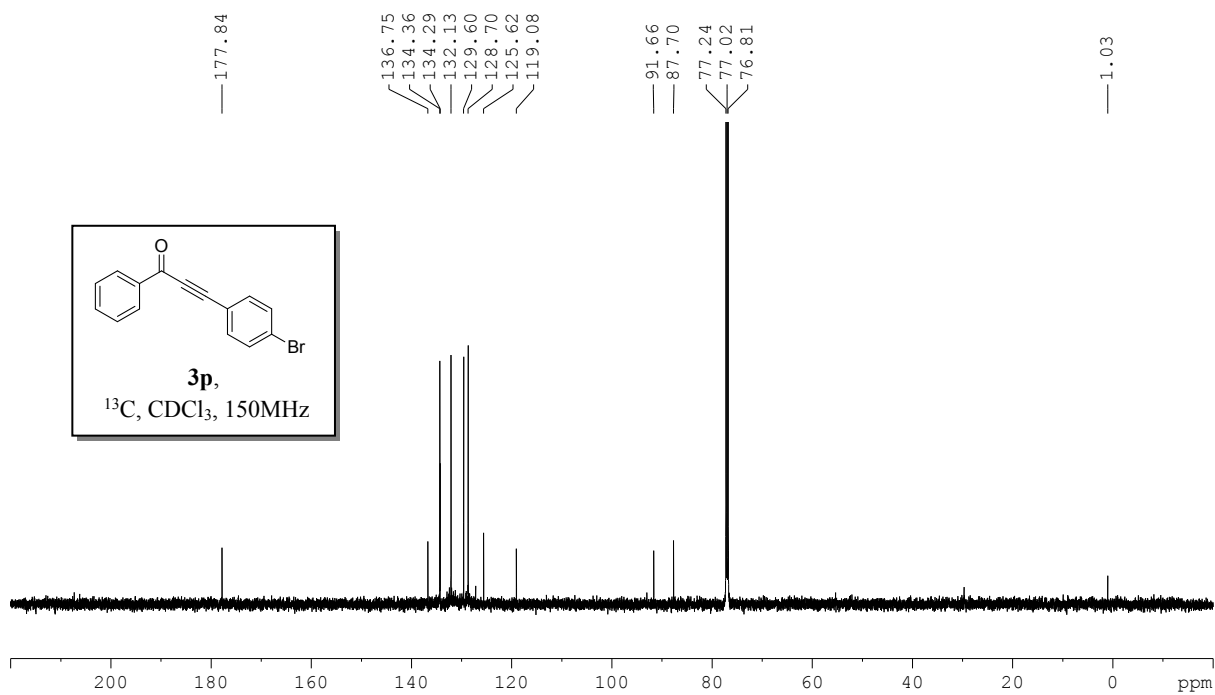
### 3-(2-Chlorophenyl)-1-phenylprop-2-yn-1-one



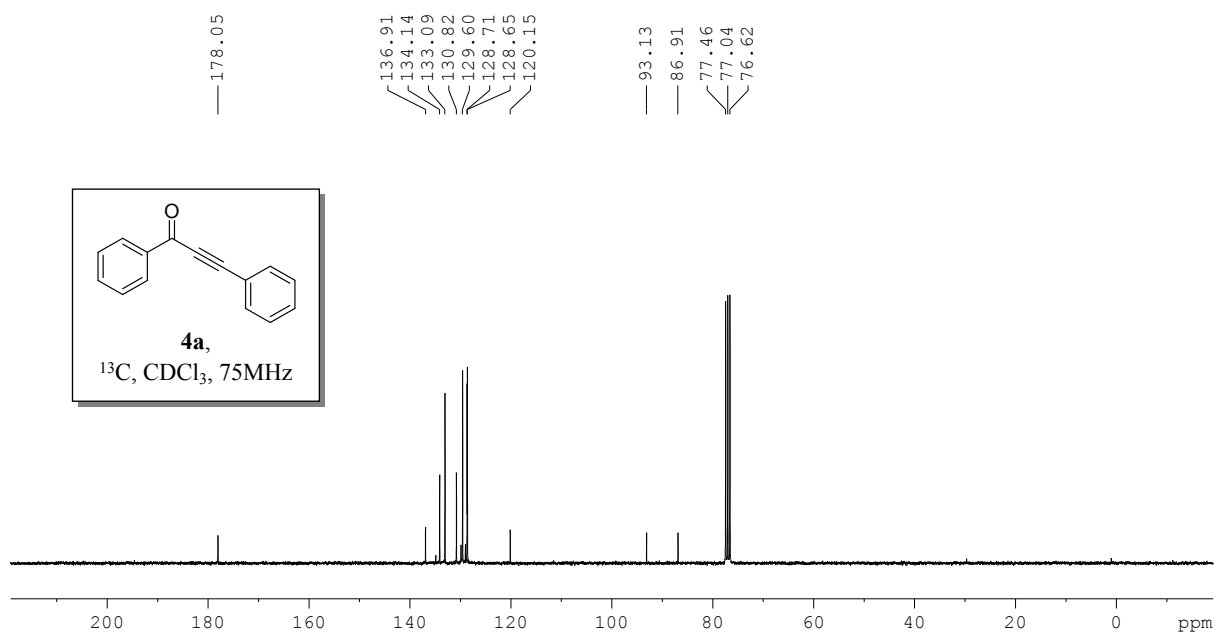
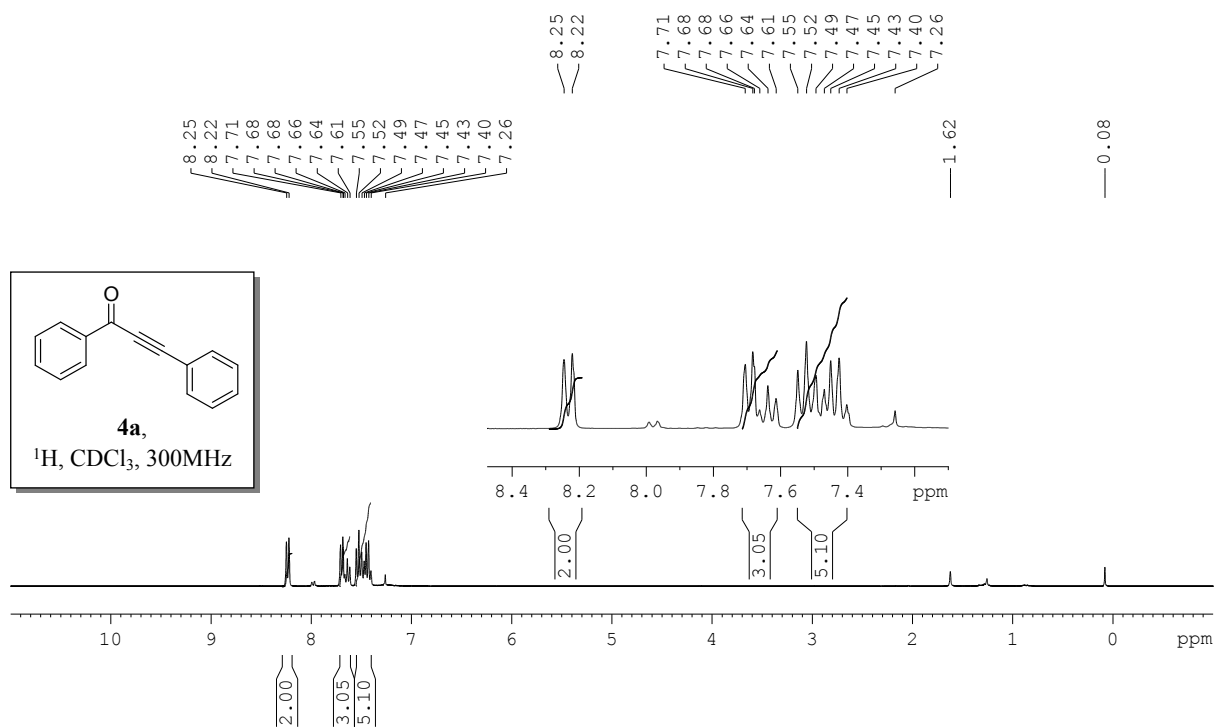


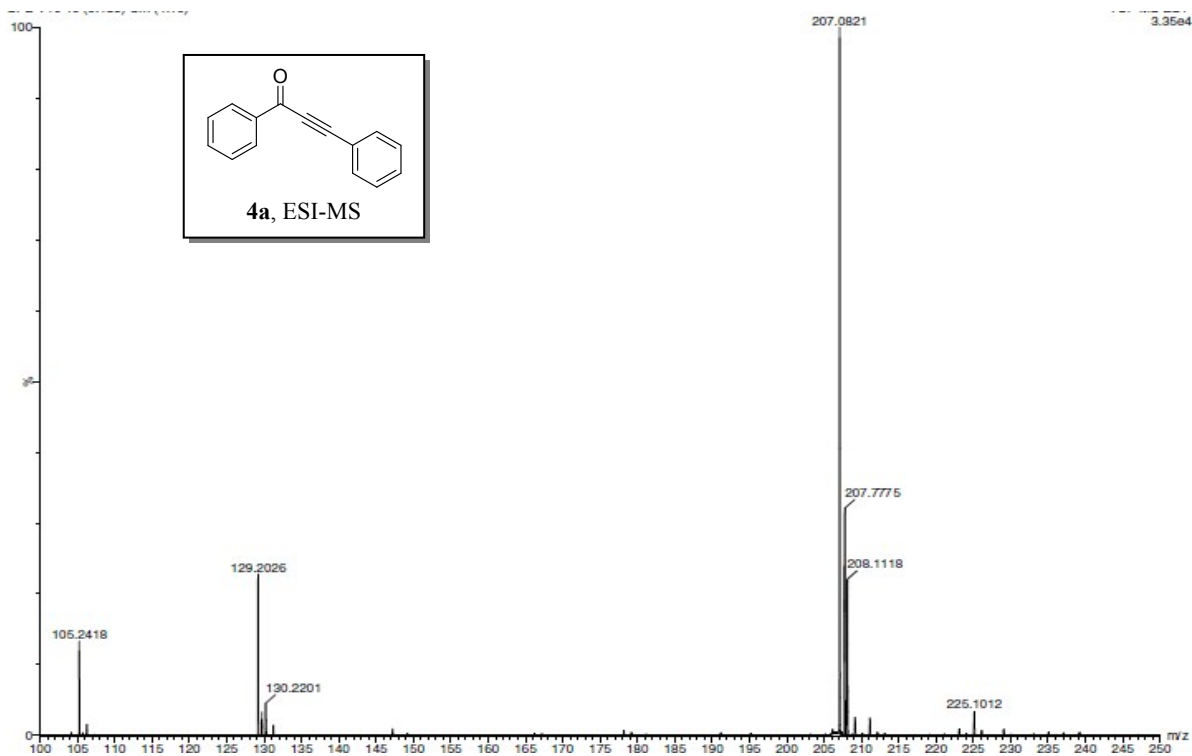
### 3-(2-Bromophenyl)-1-phenylprop-2-yn-1-one



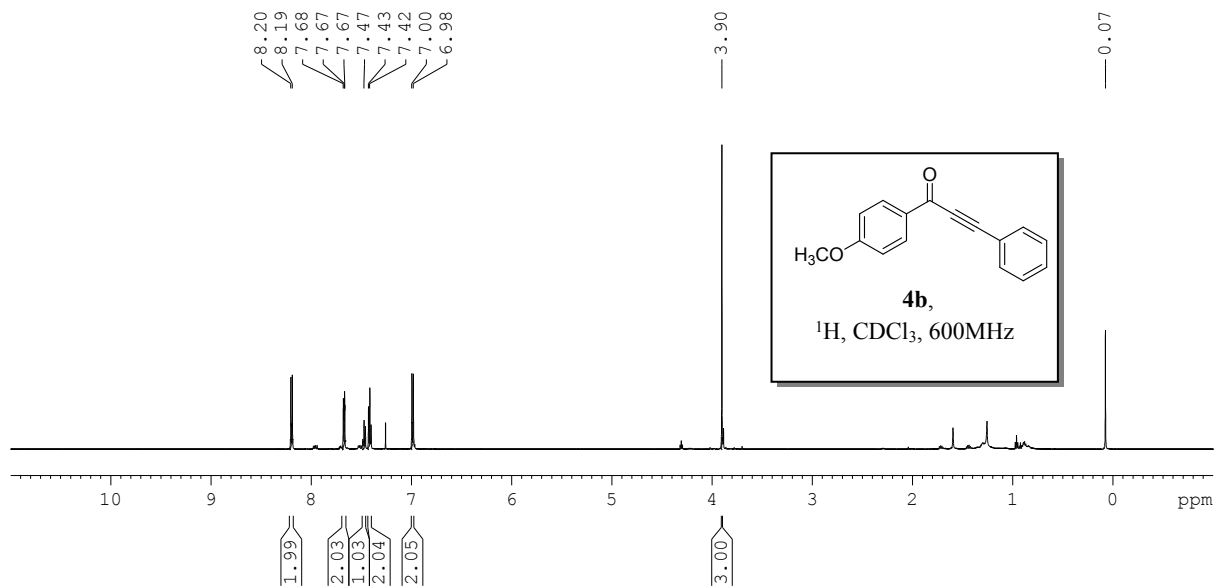


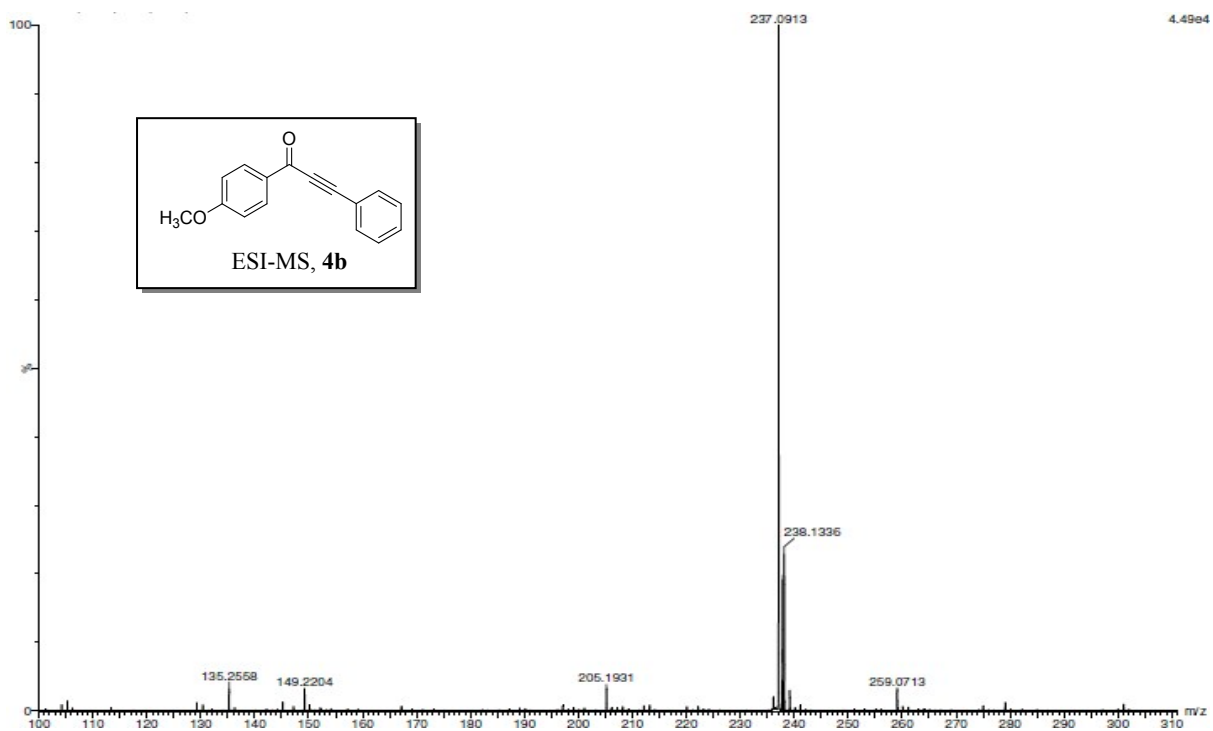
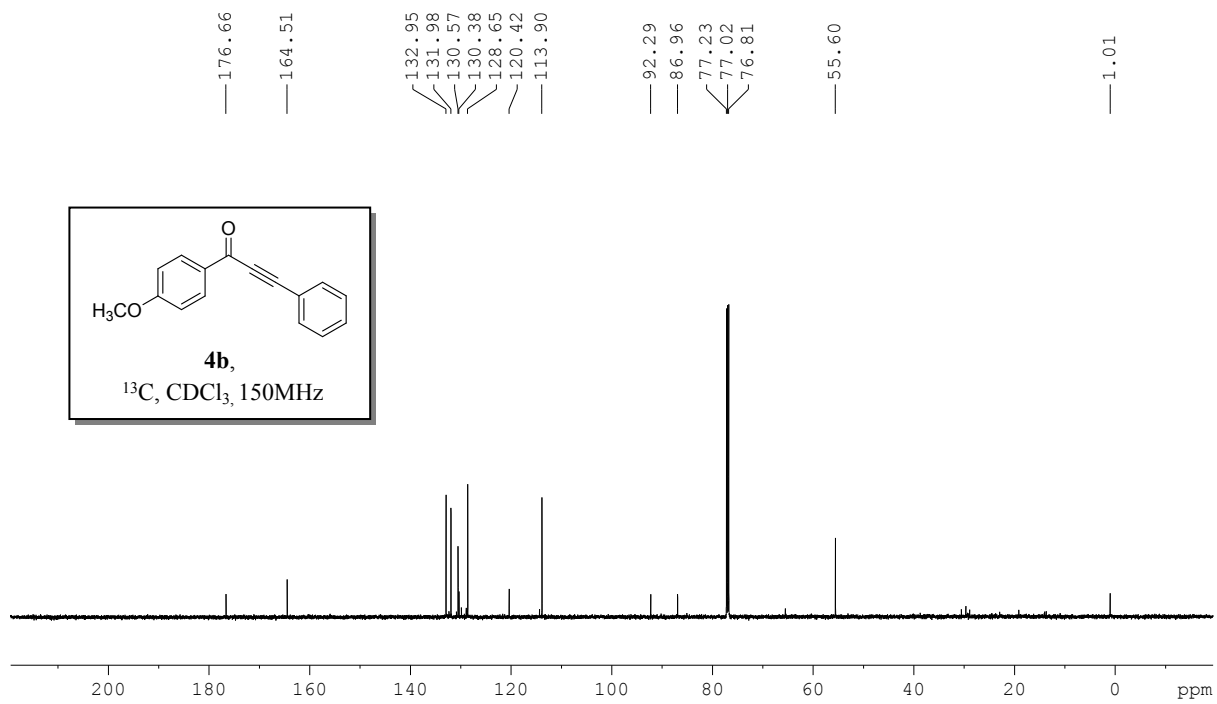
# 1, 3-Diphenylprop-2-yn-1-one



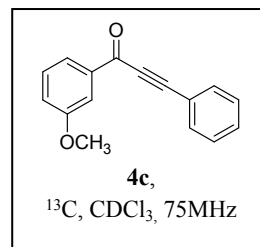
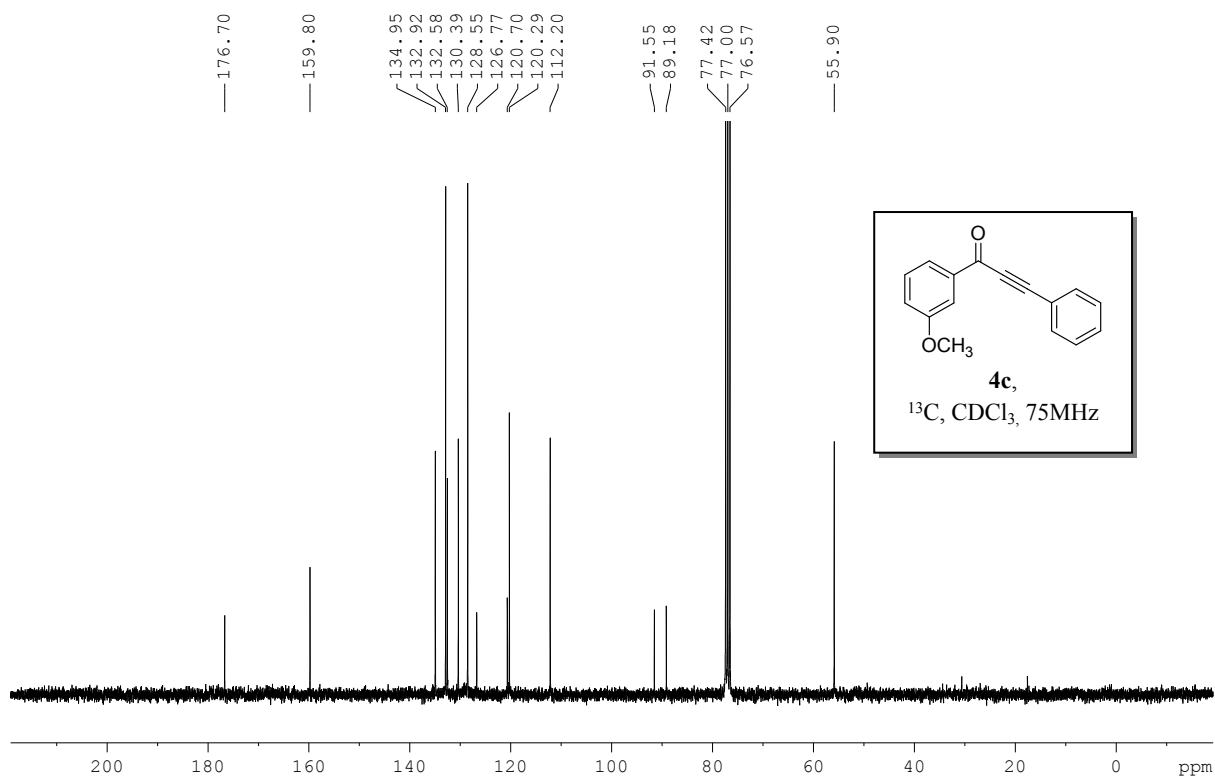
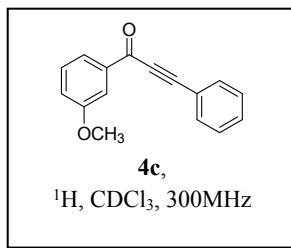
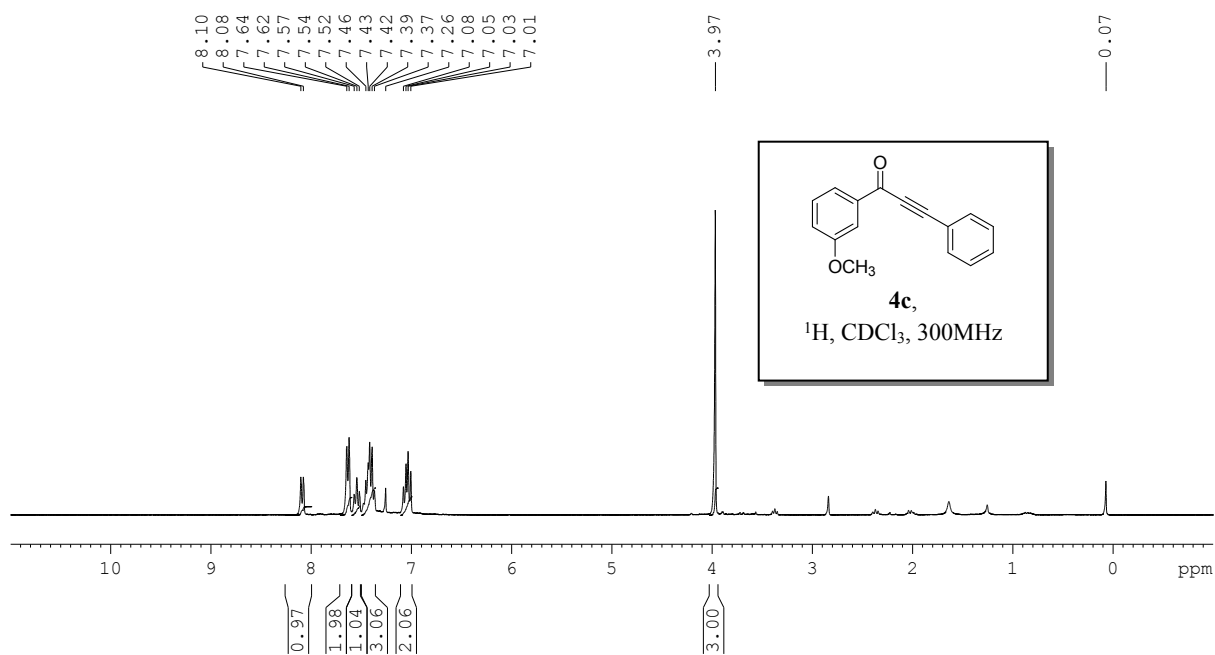


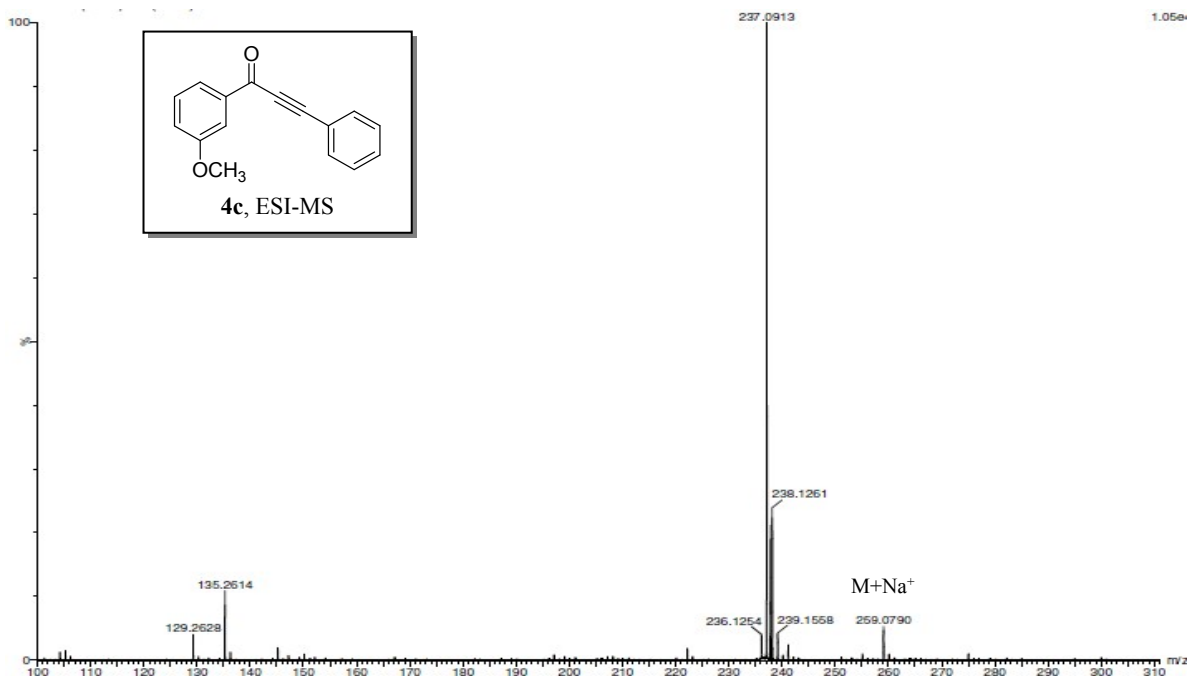
**1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-one**



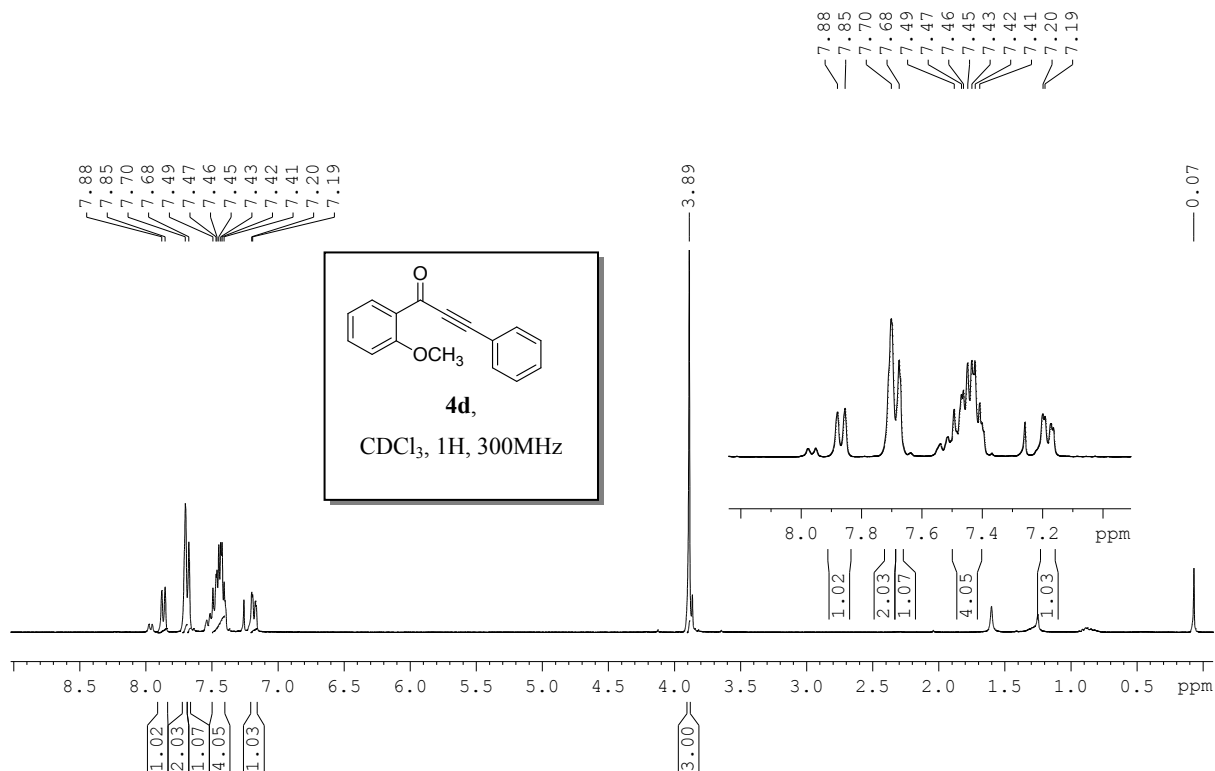


# 1-(3-Methoxyphenyl)-3-phenylprop-2-yn-1-one

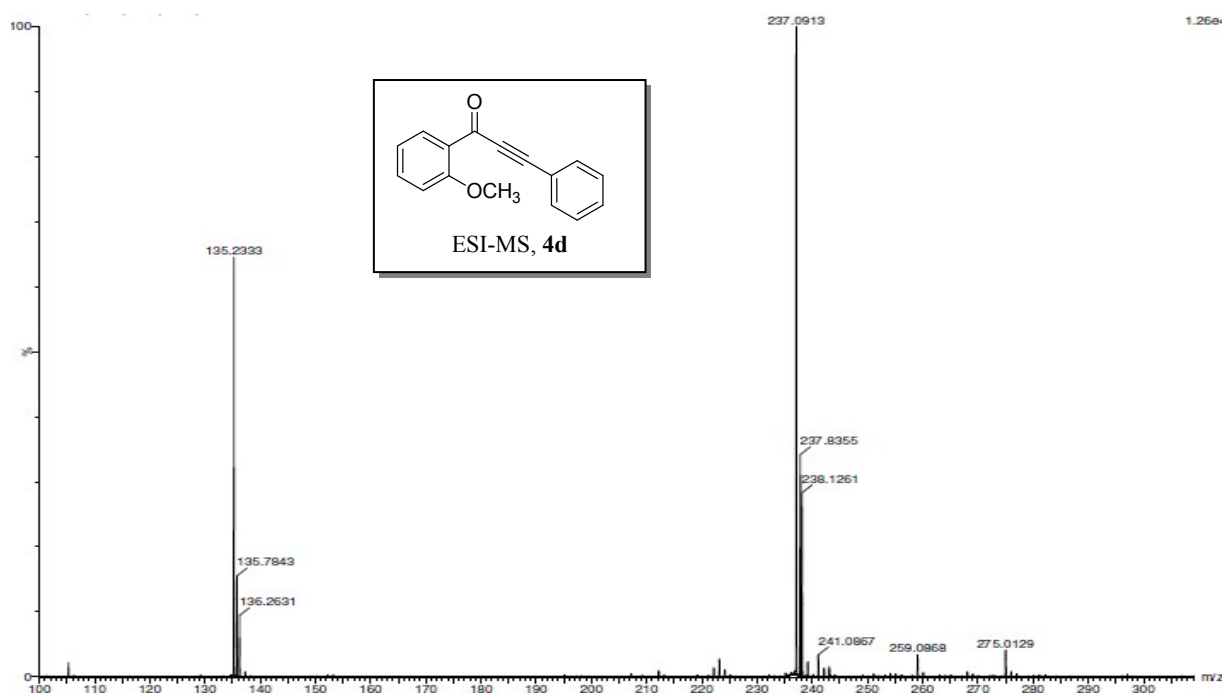
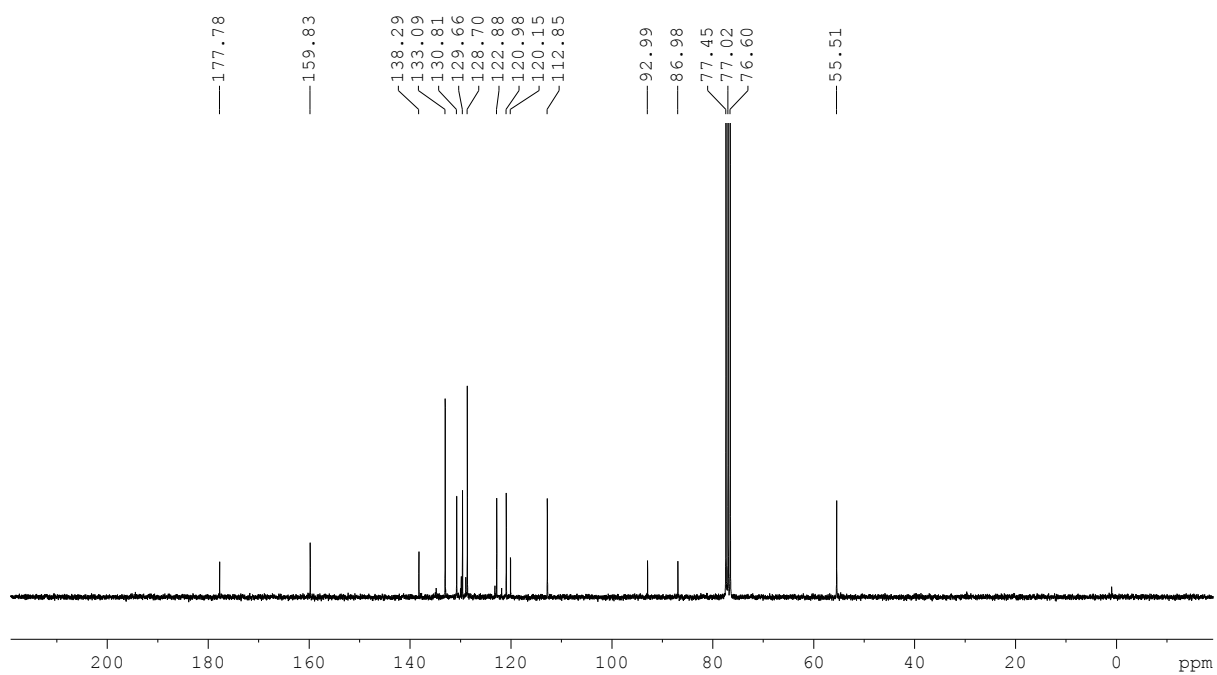




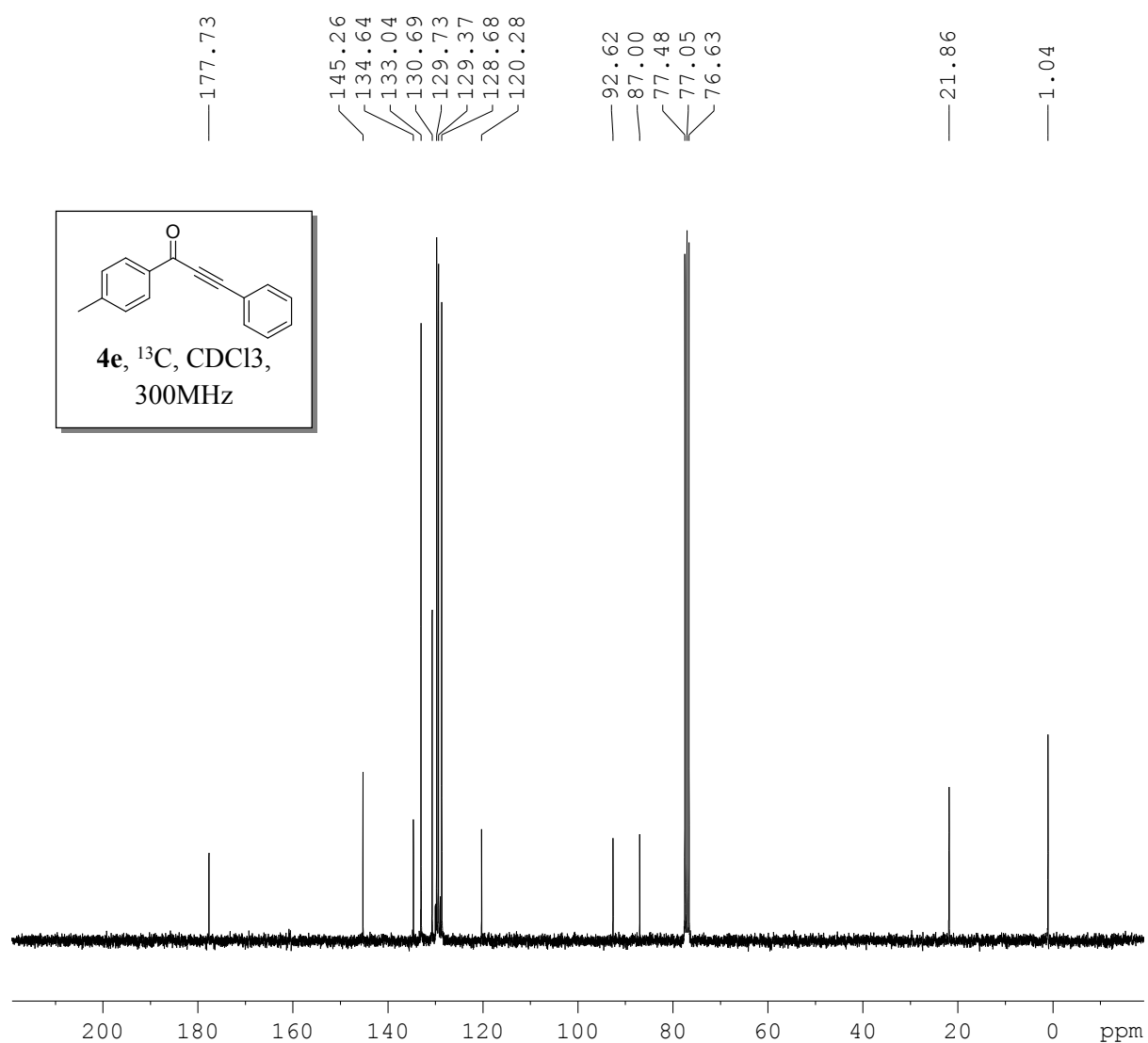
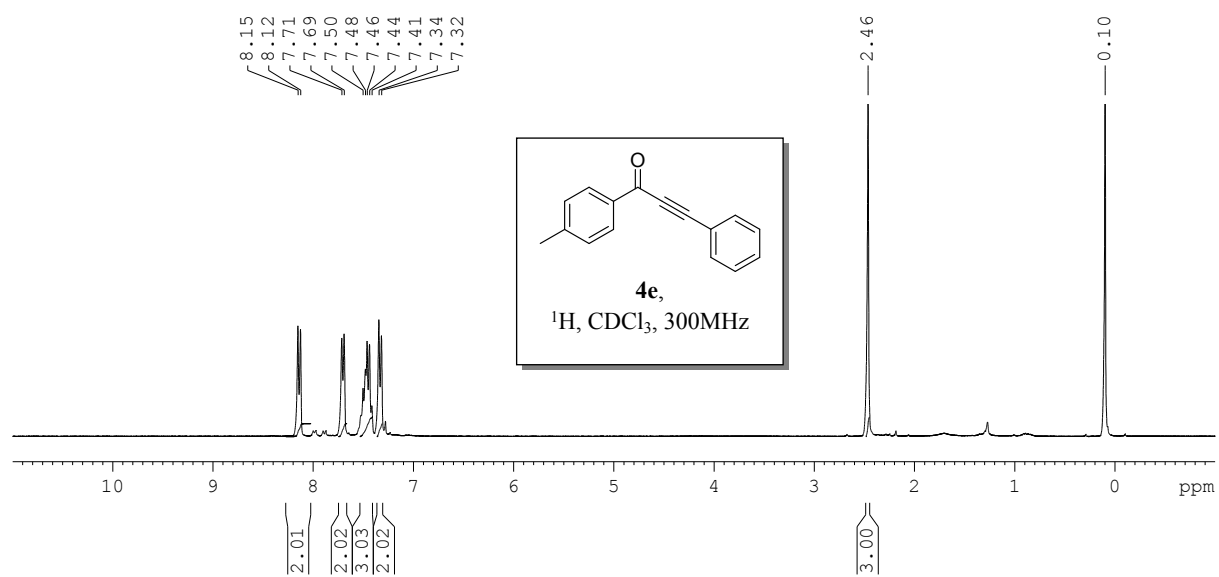
**1-(2-Methoxyphenyl)-3-phenylprop-2-yn-1-one**

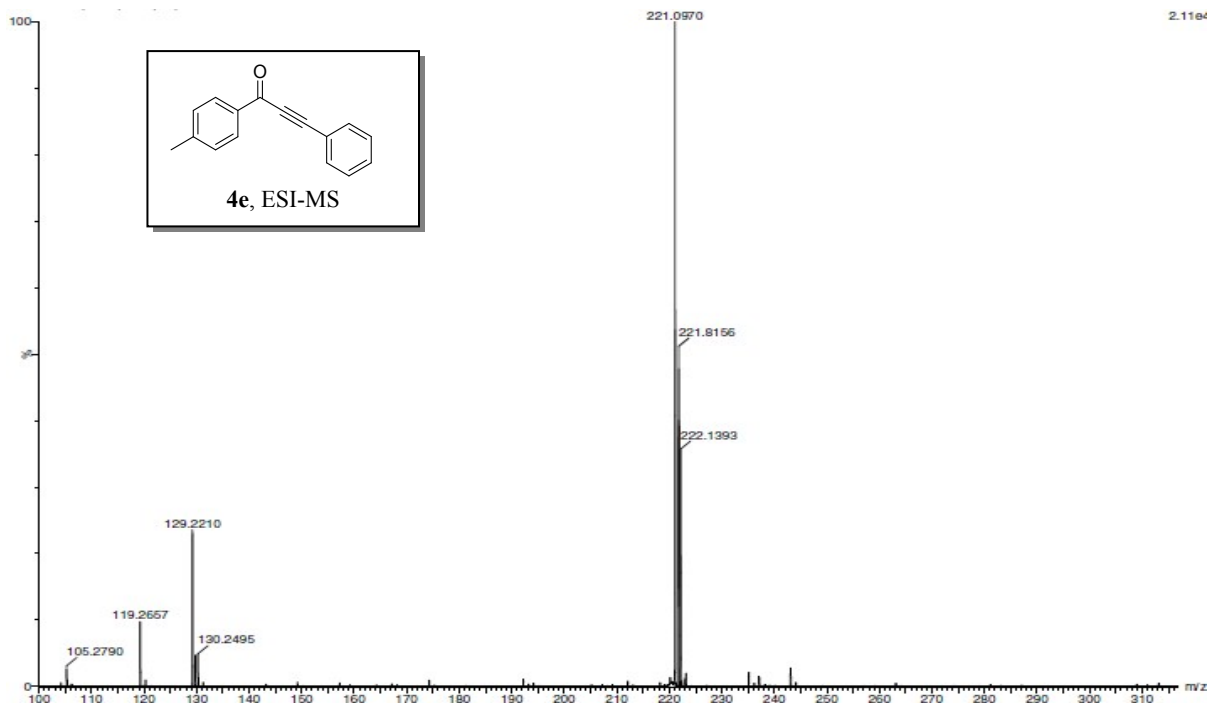




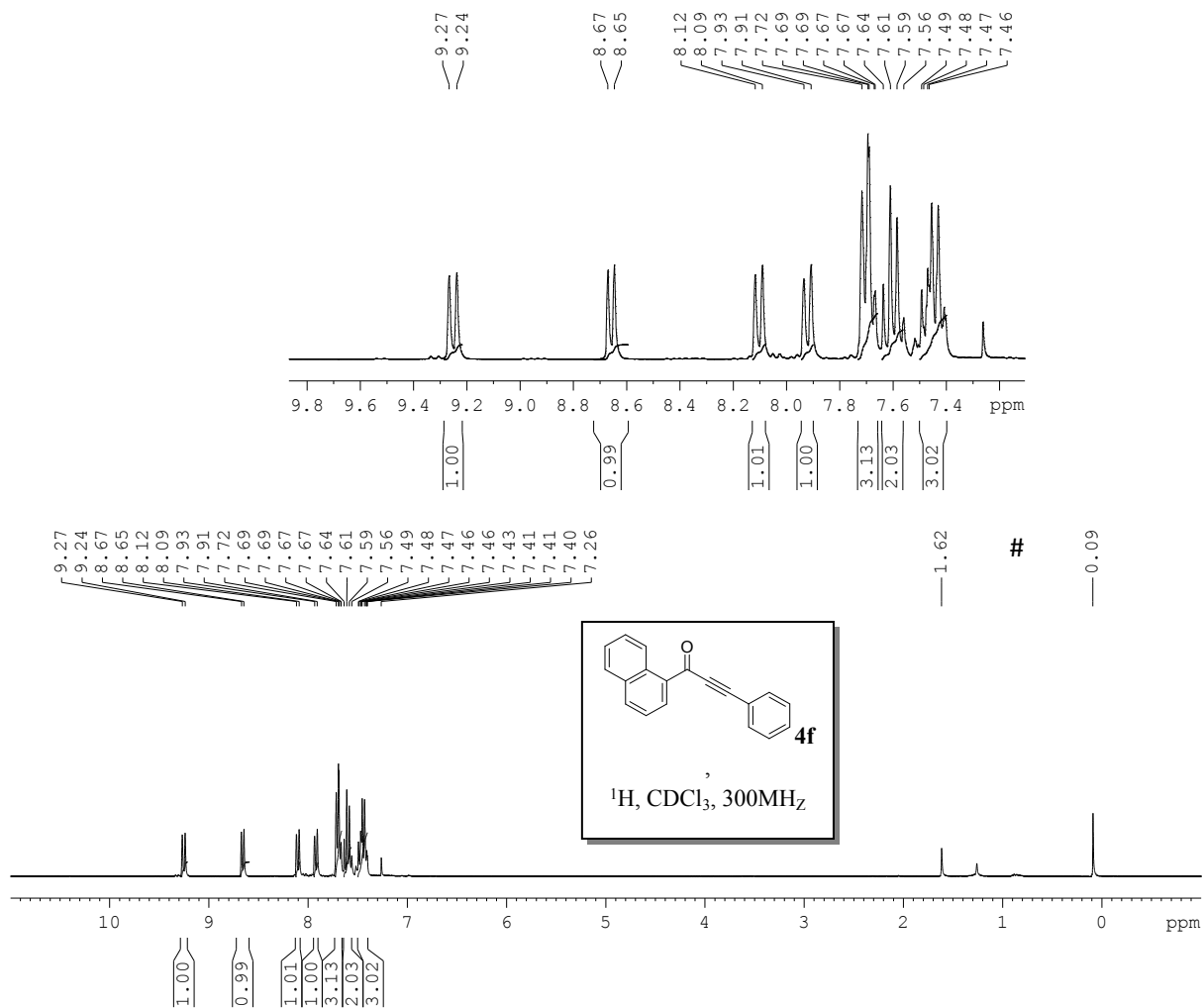


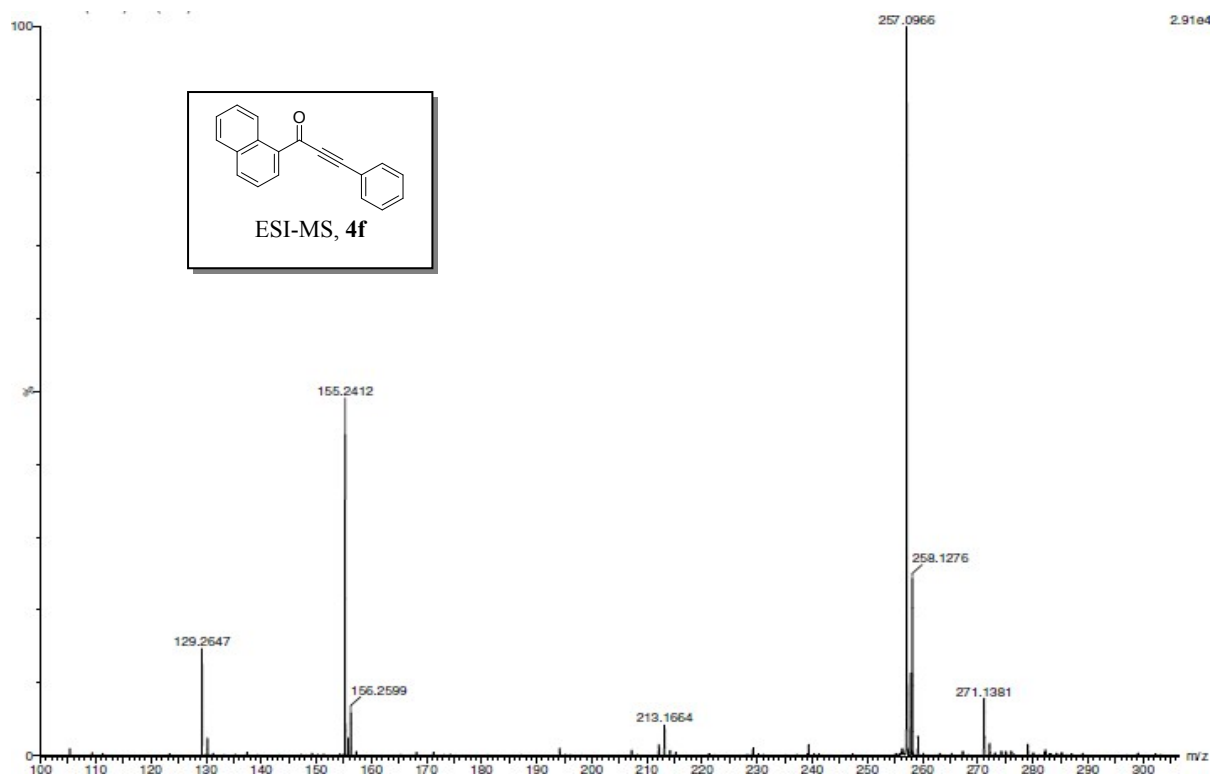
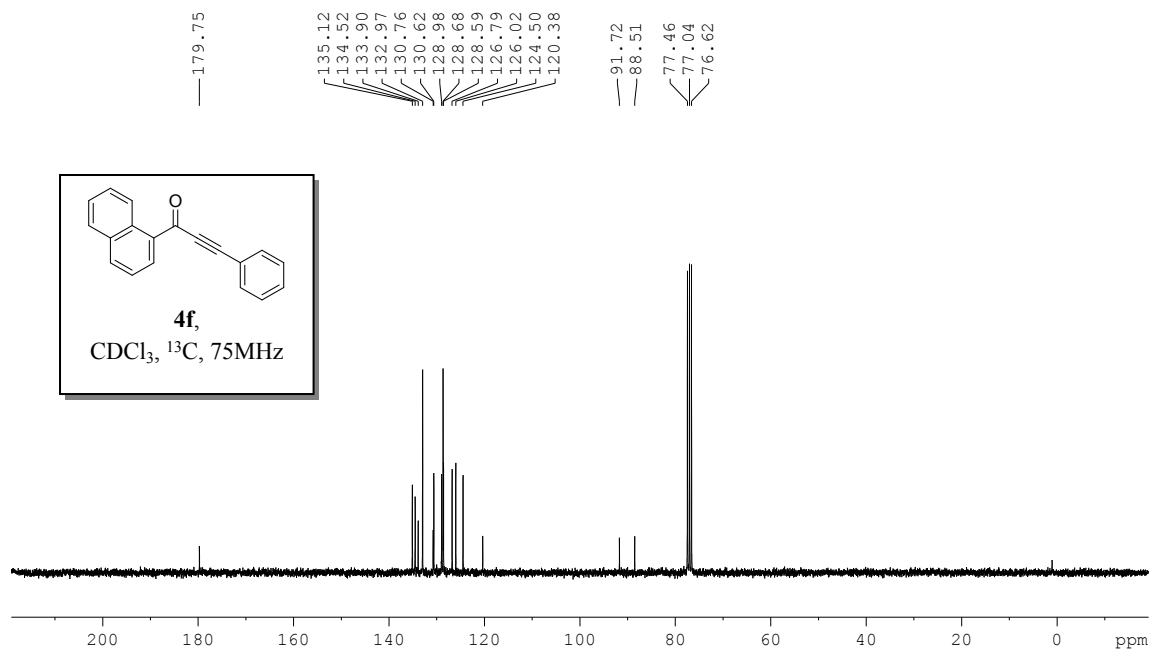
### 3-Phenyl-1-(p-tolyl)prop-2-yn-1-one



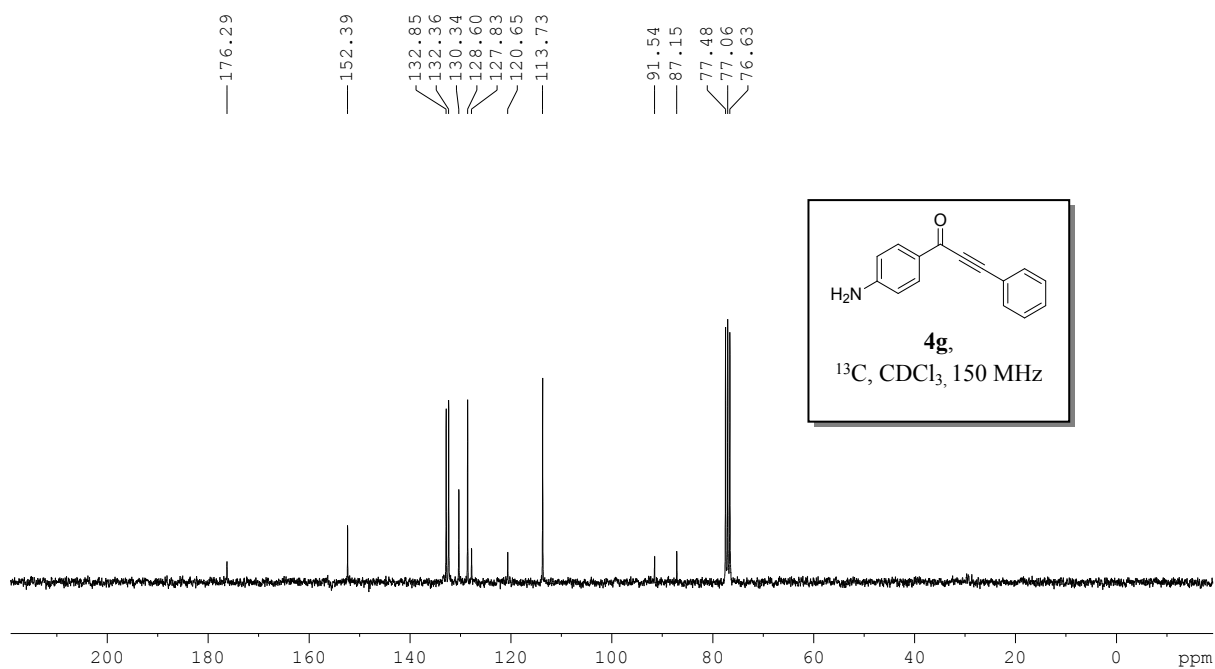
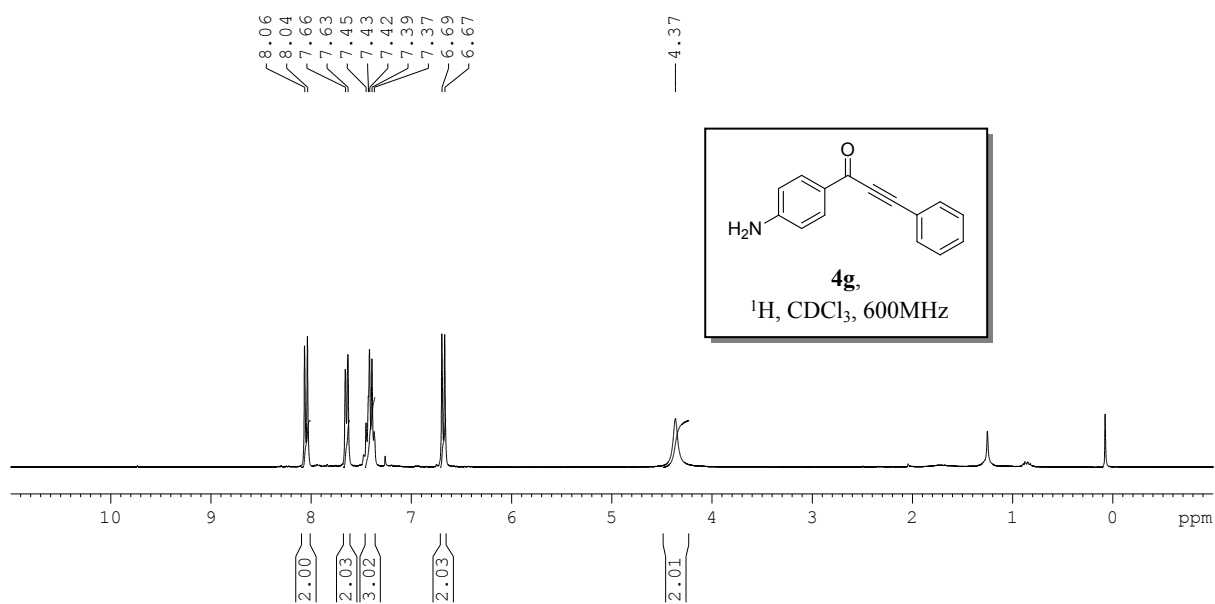


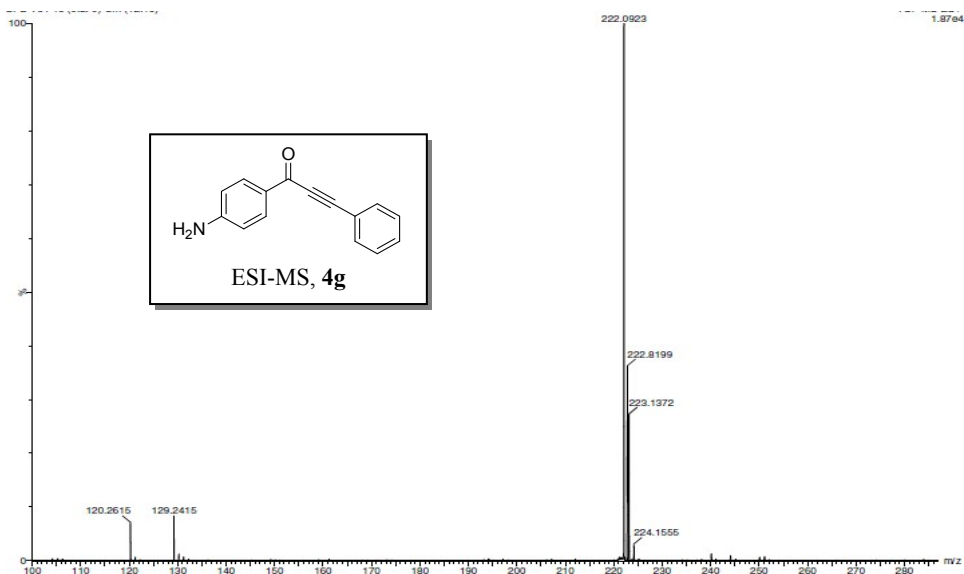
**1-(Naphthalen-1-yl)-3-phenylprop-2-yn-1-one**



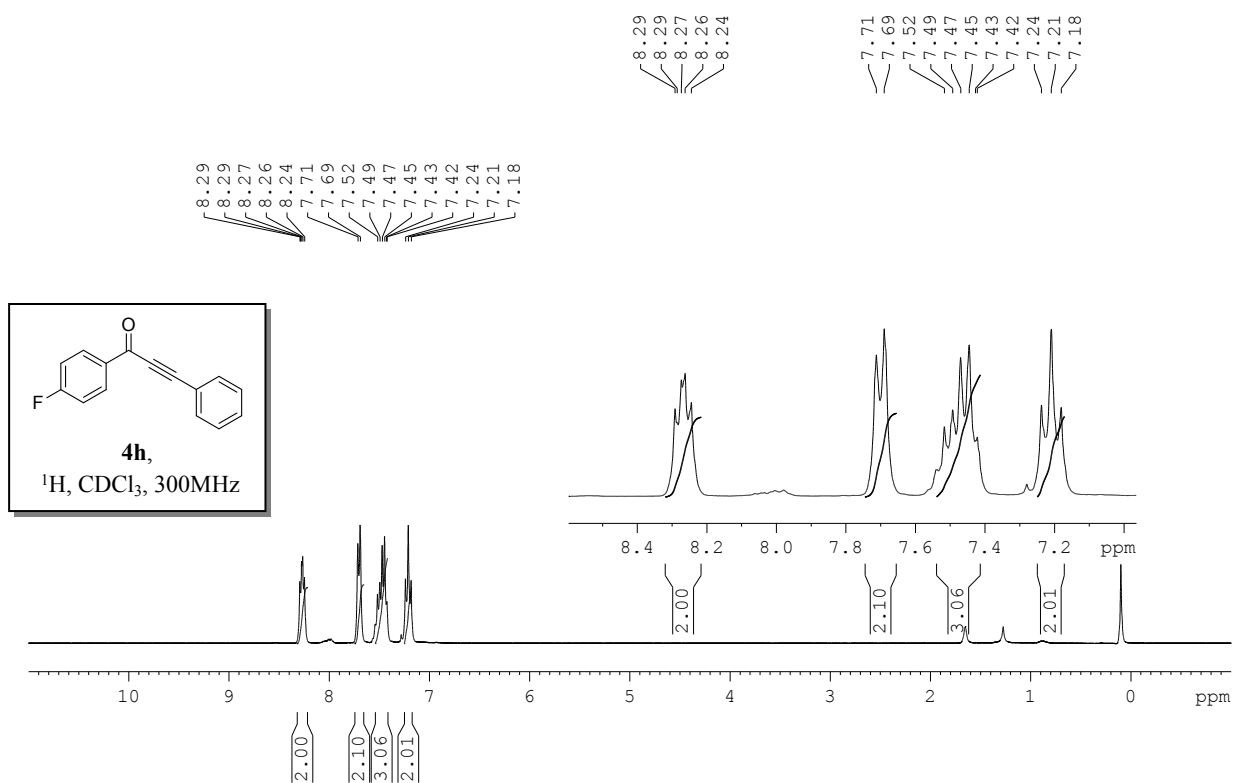


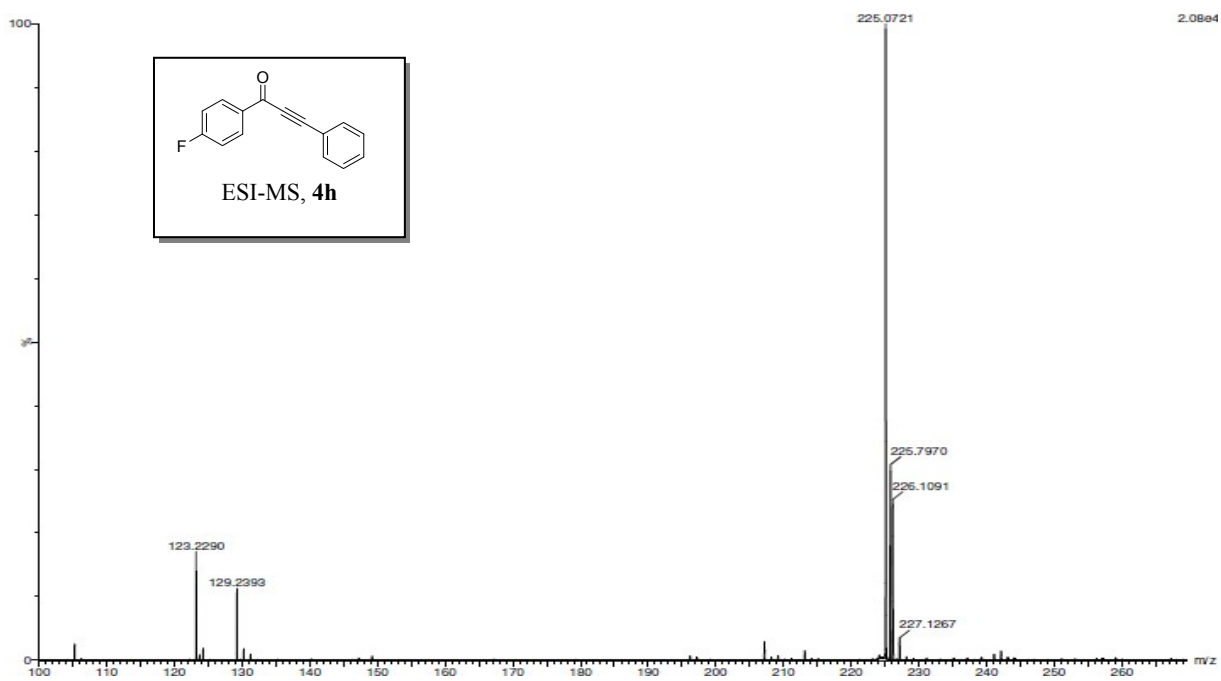
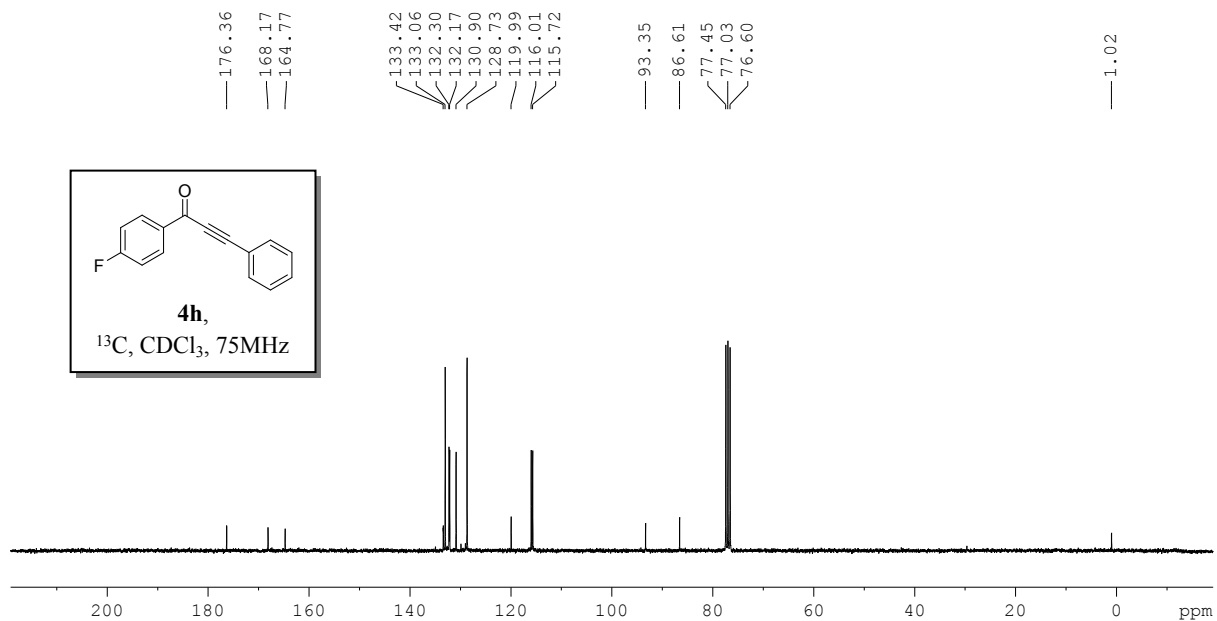
# 1-(4-Aminophenyl)-3-phenylprop-2-yn-1-one





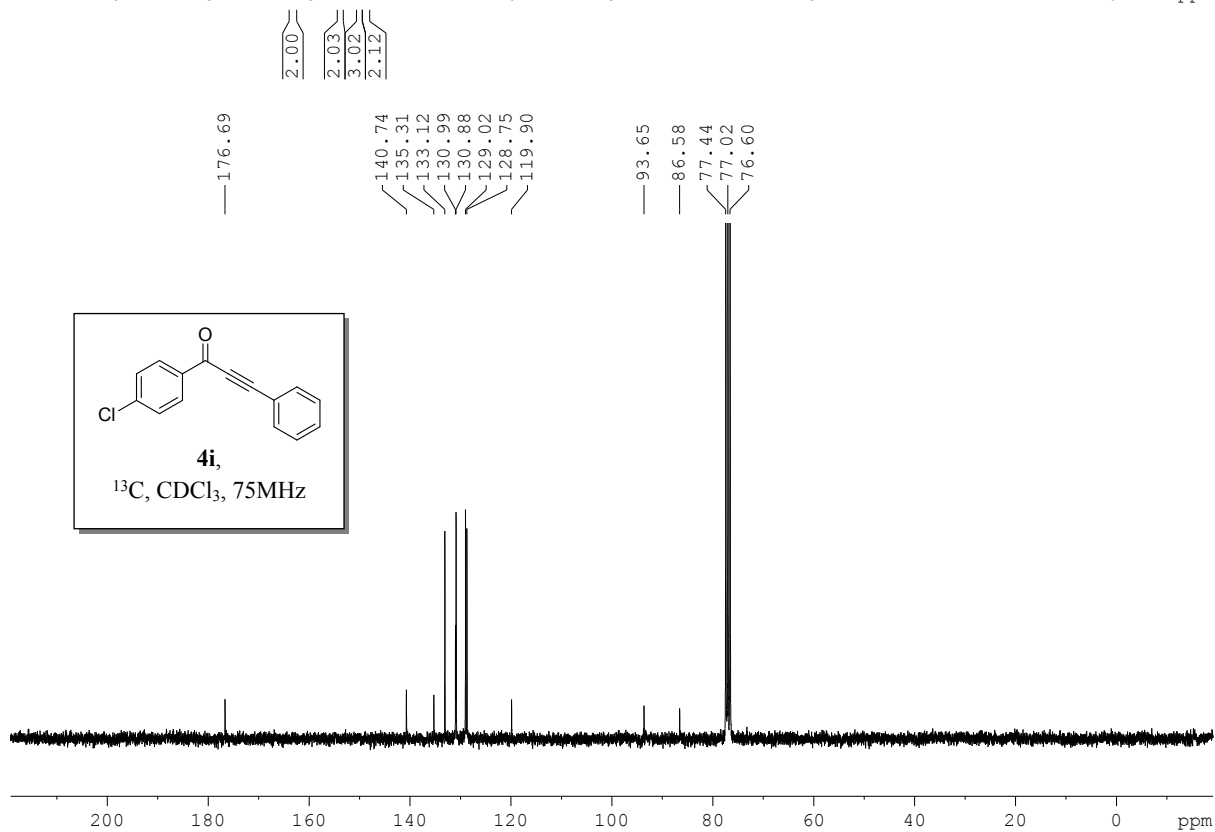
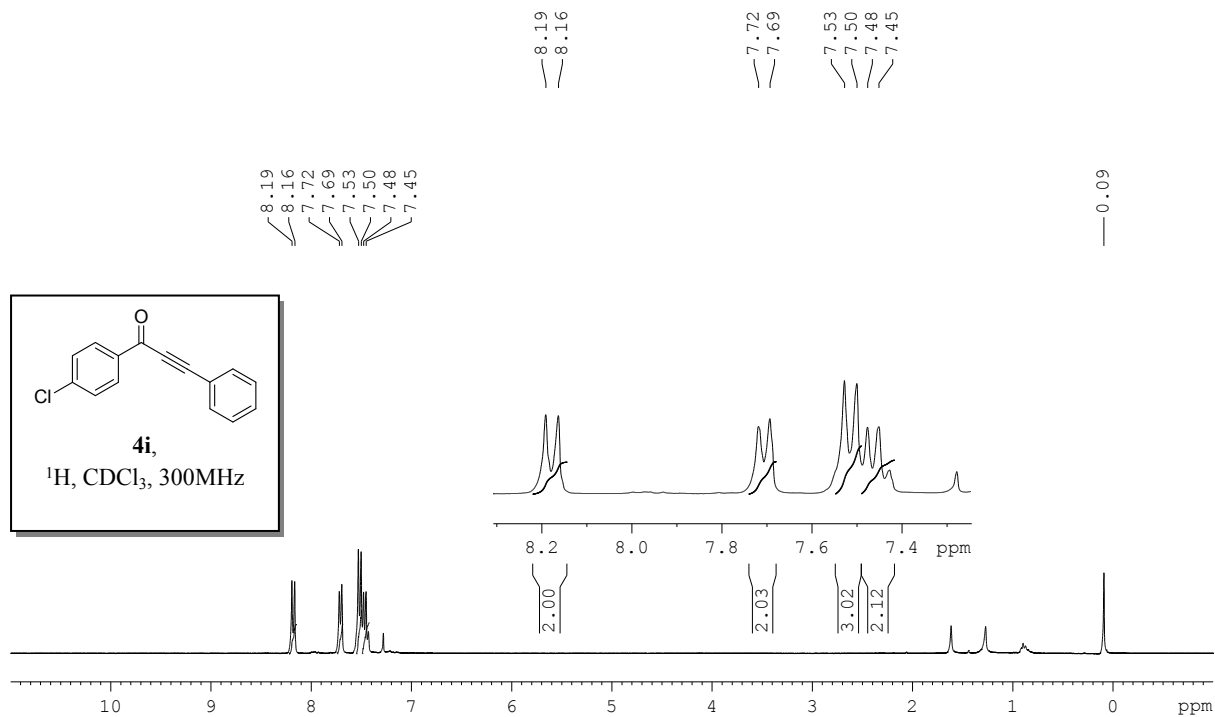
### 1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-one

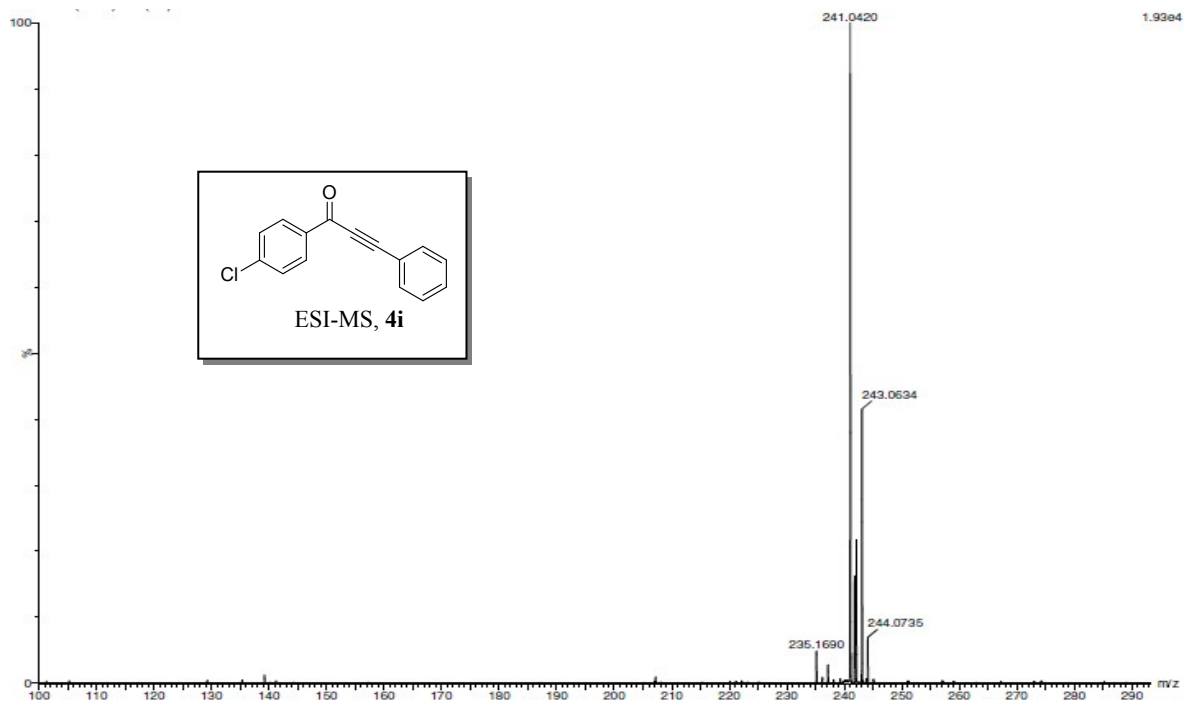




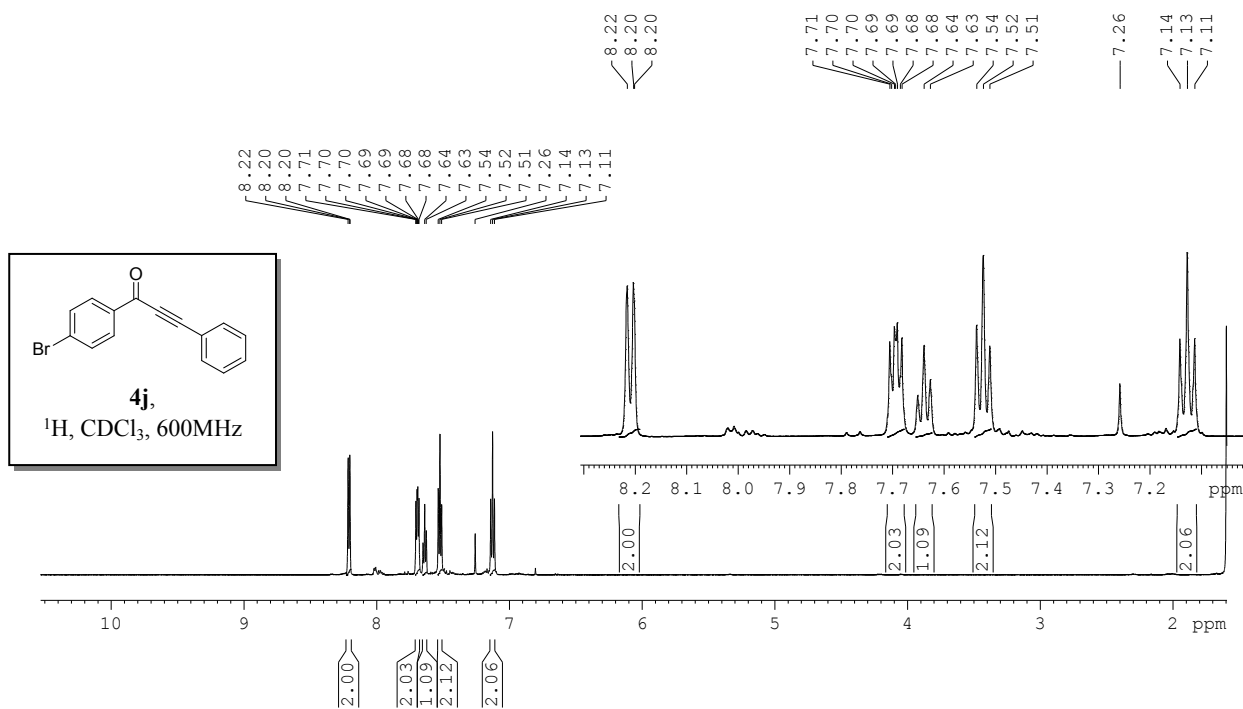


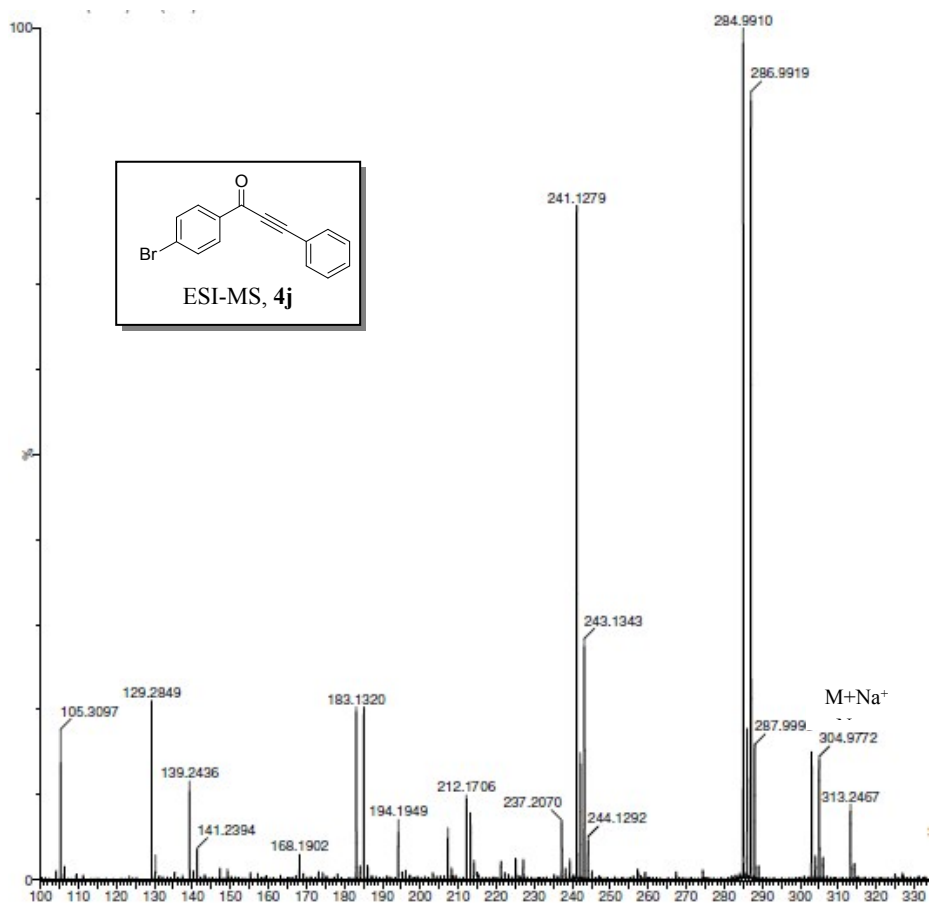
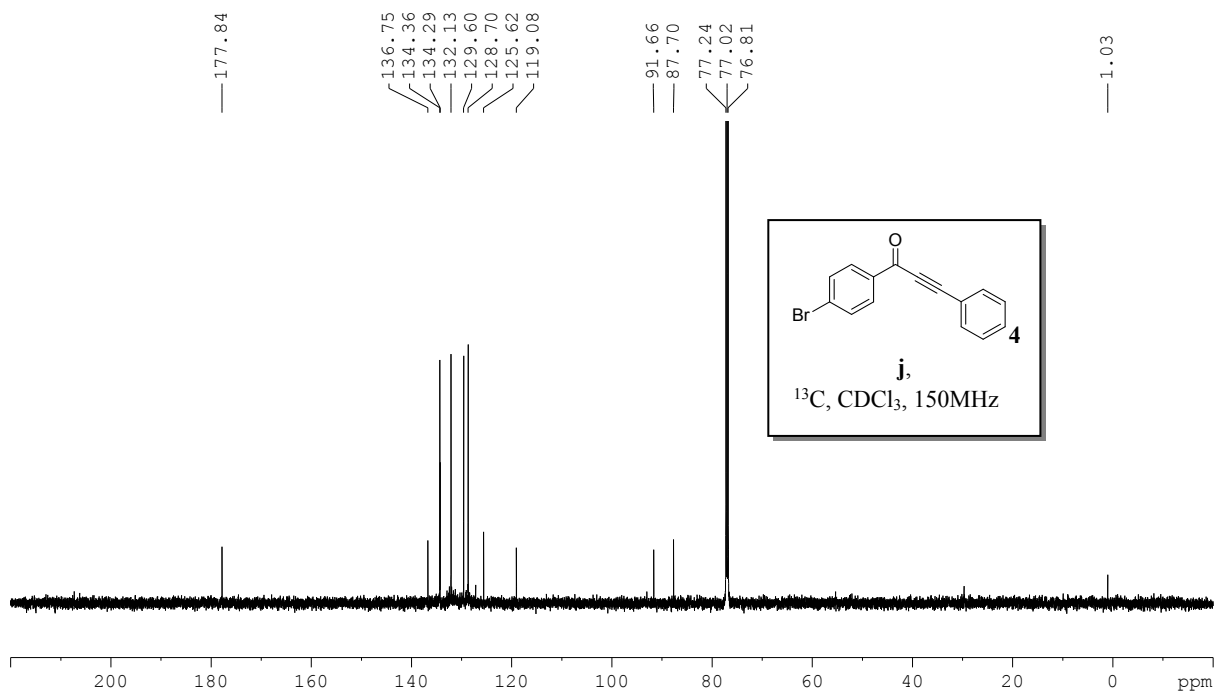
# 1-(4-Chlorophenyl)-3-phenylprop-2-yn-1-one



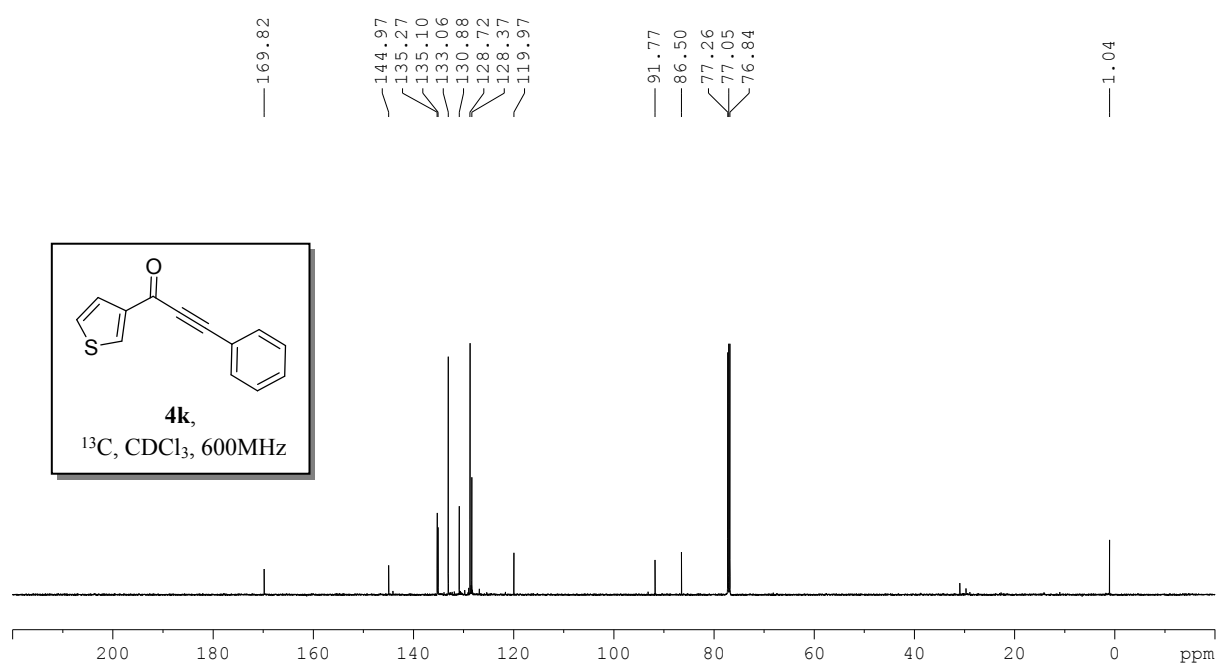
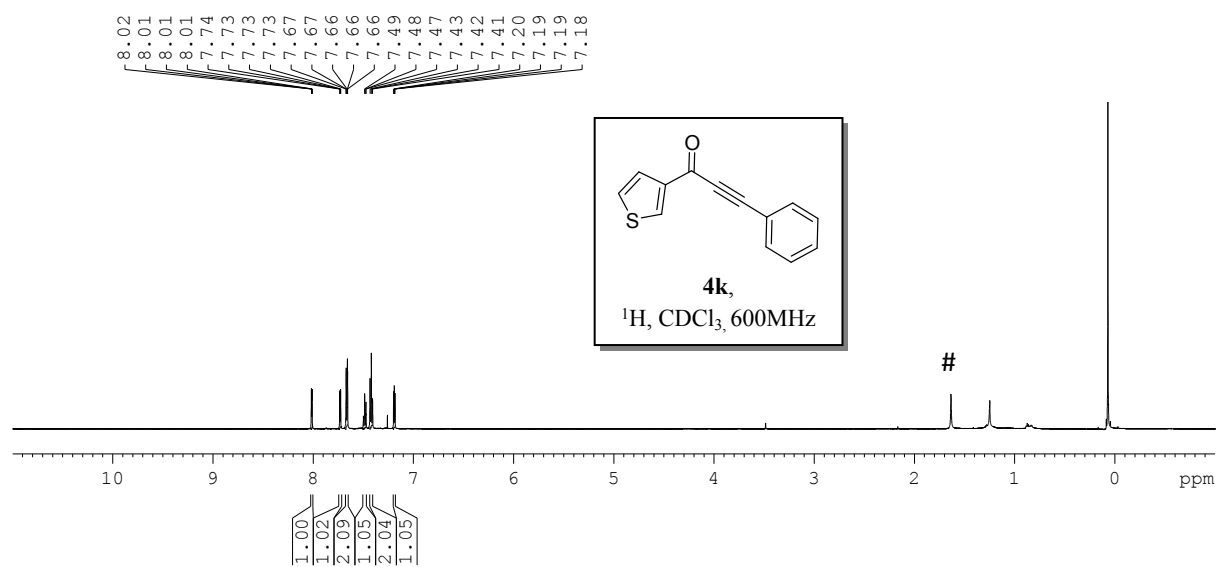


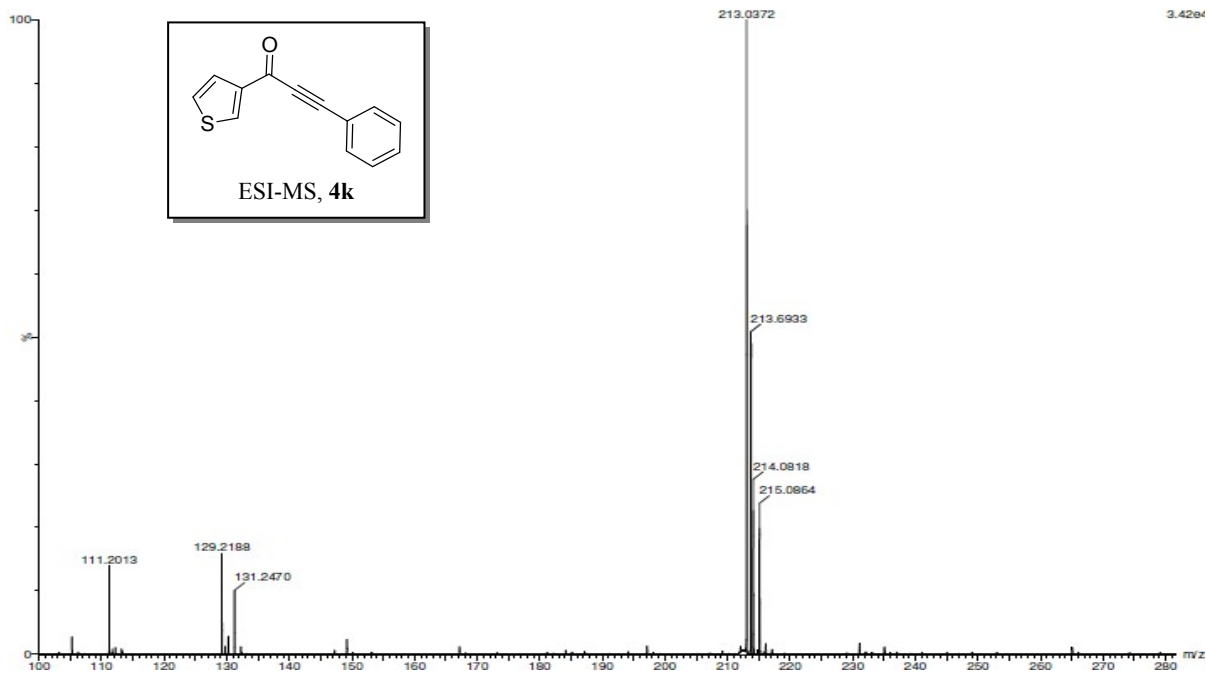
### 1-(4-Bromophenyl)-3-phenylprop-2-yn-1-one



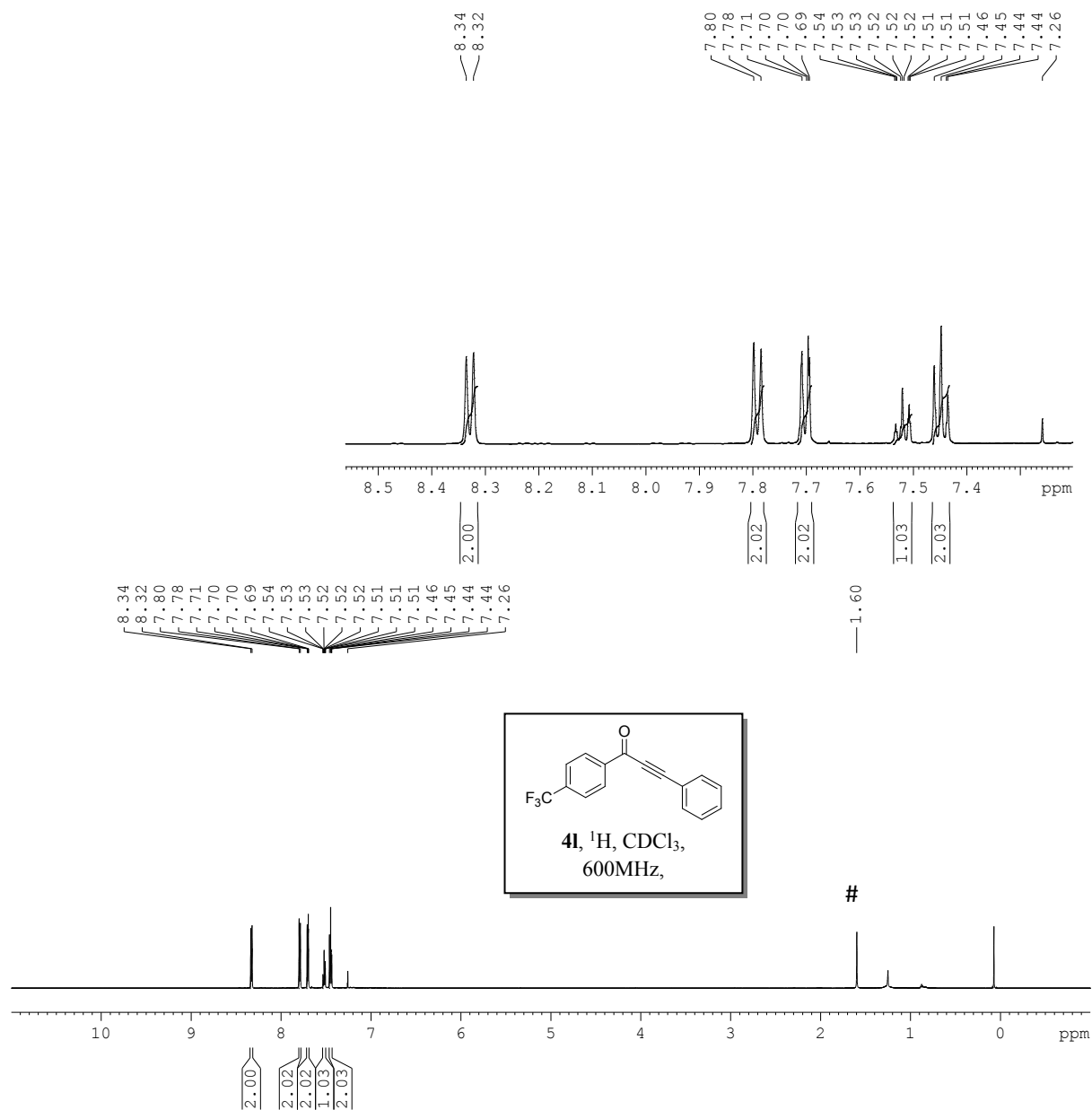


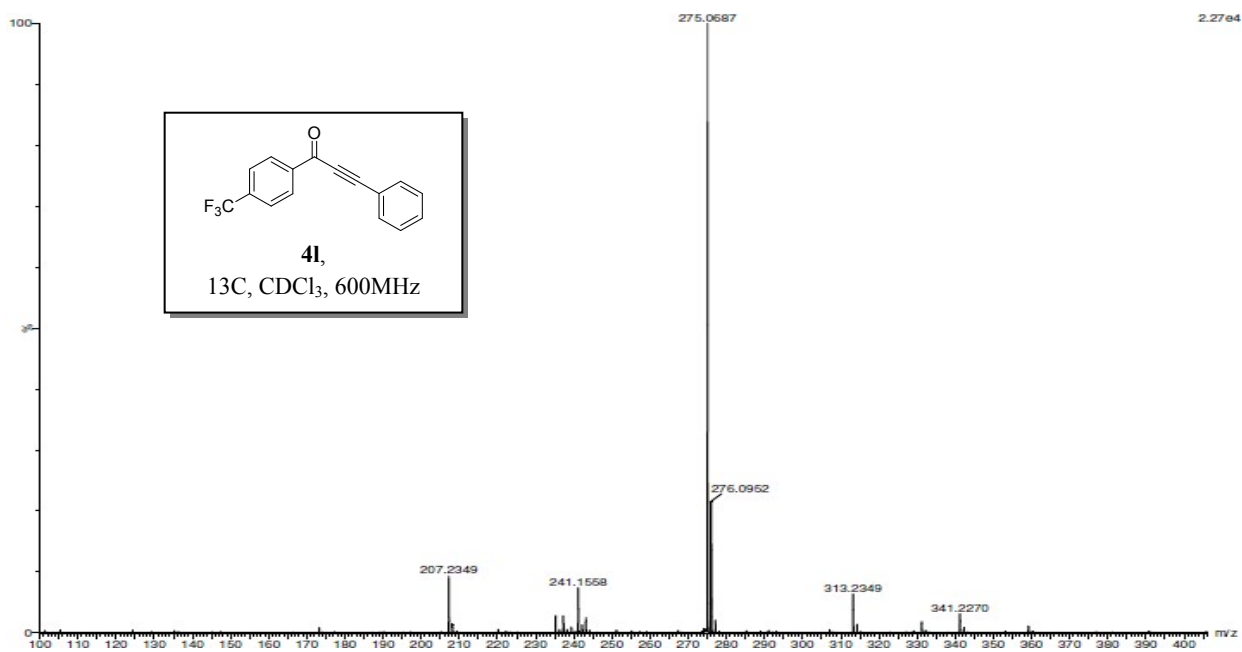
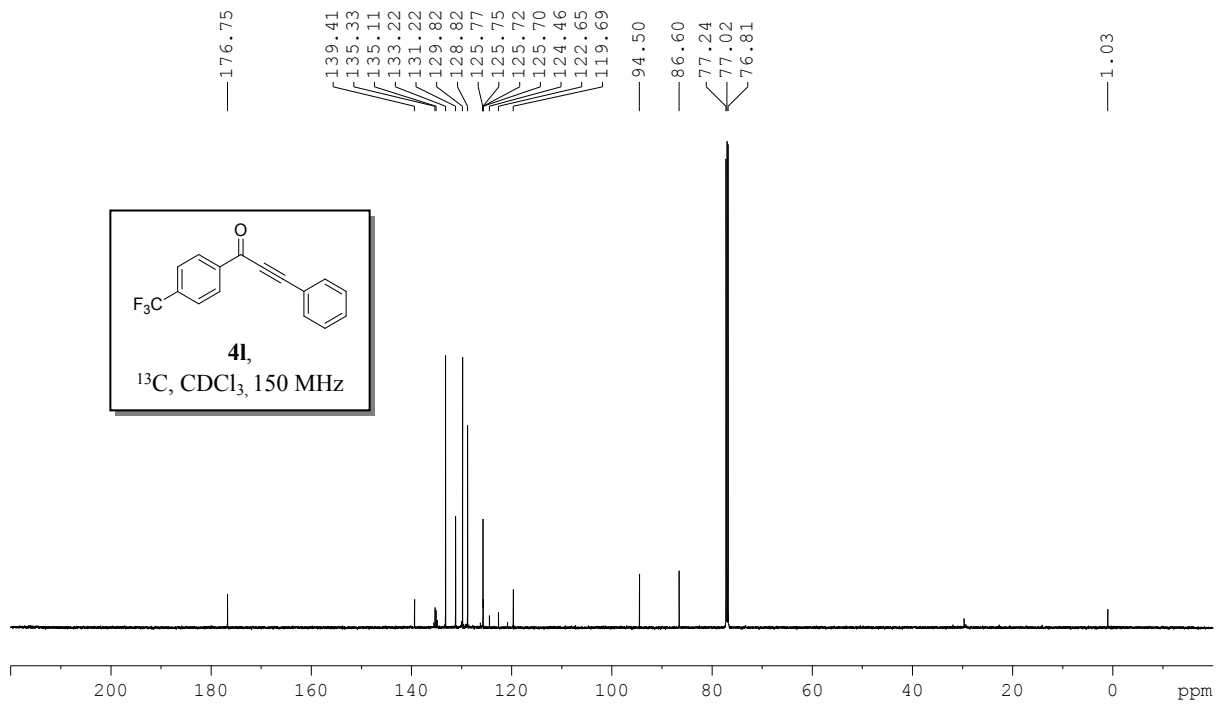
### 3-Phenyl-1-(thiophen-2-yl)prop-2-yn-1-one



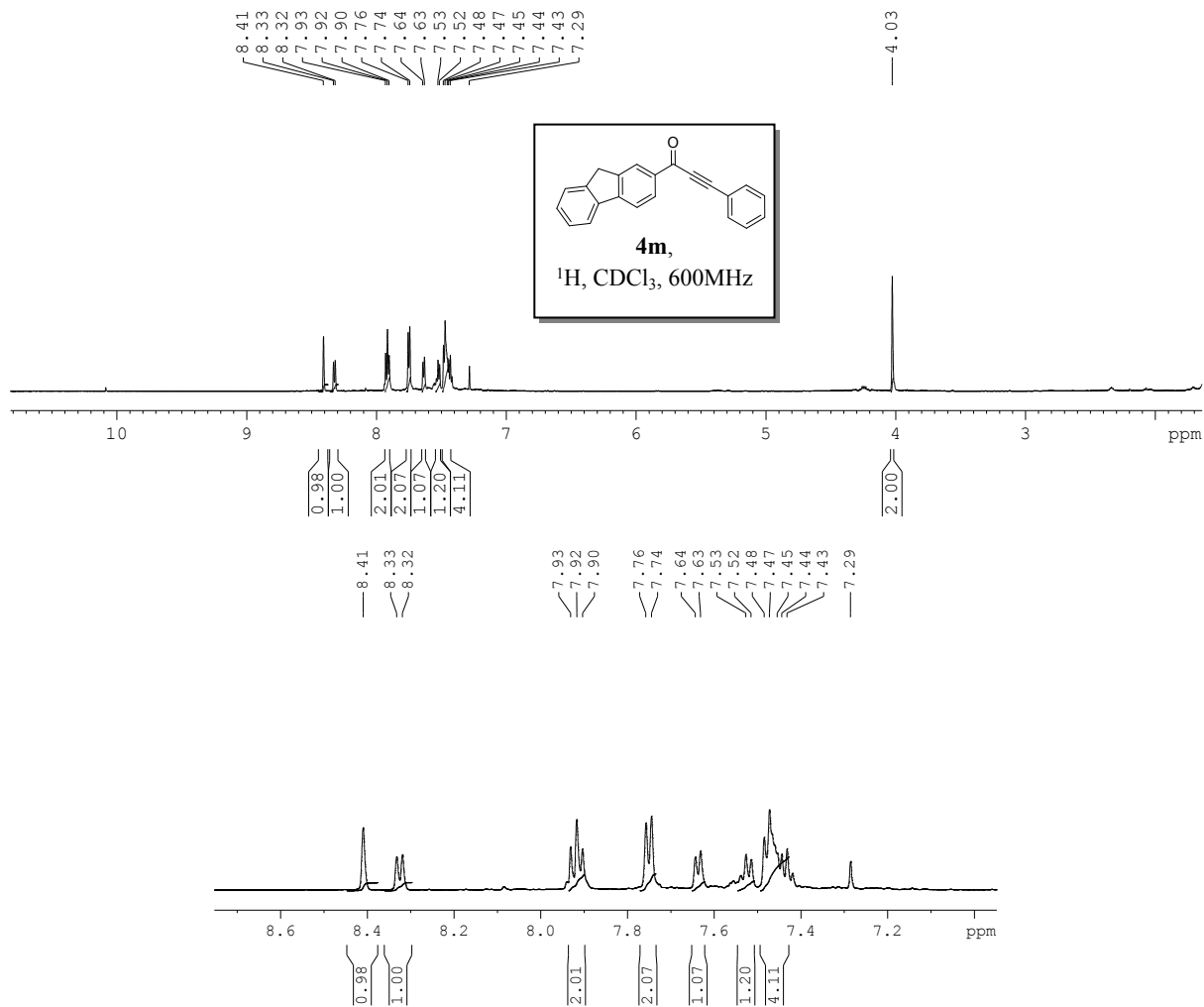


**3-Phenyl-1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one**

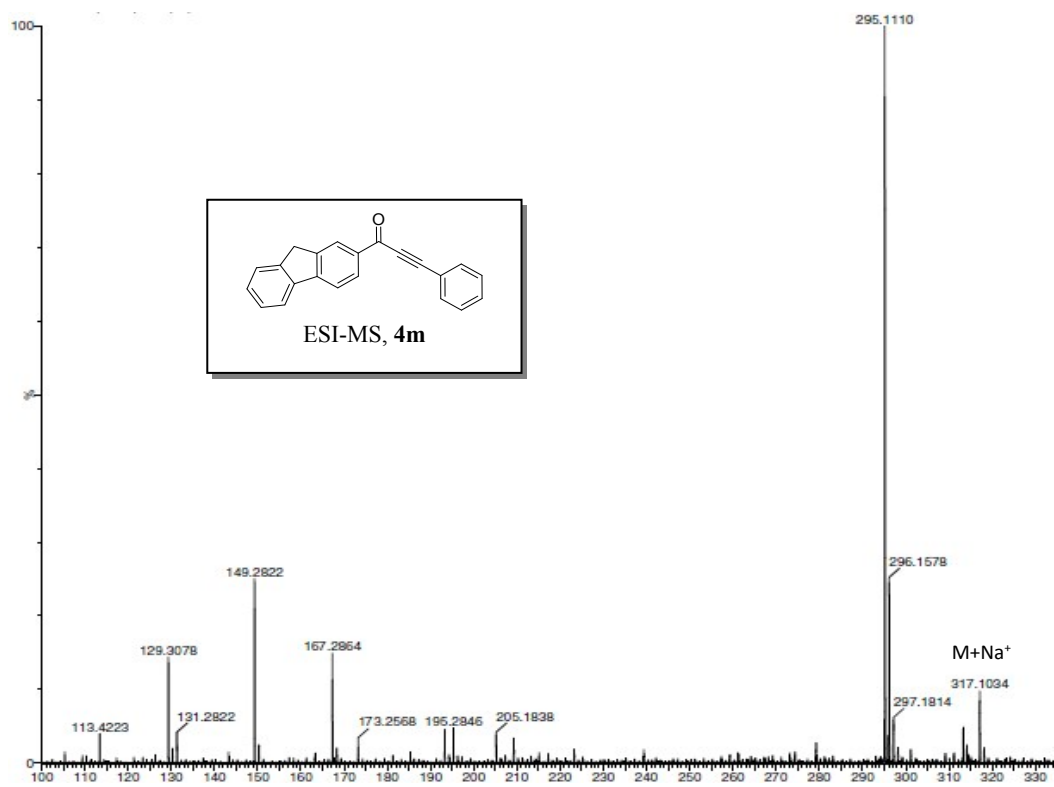
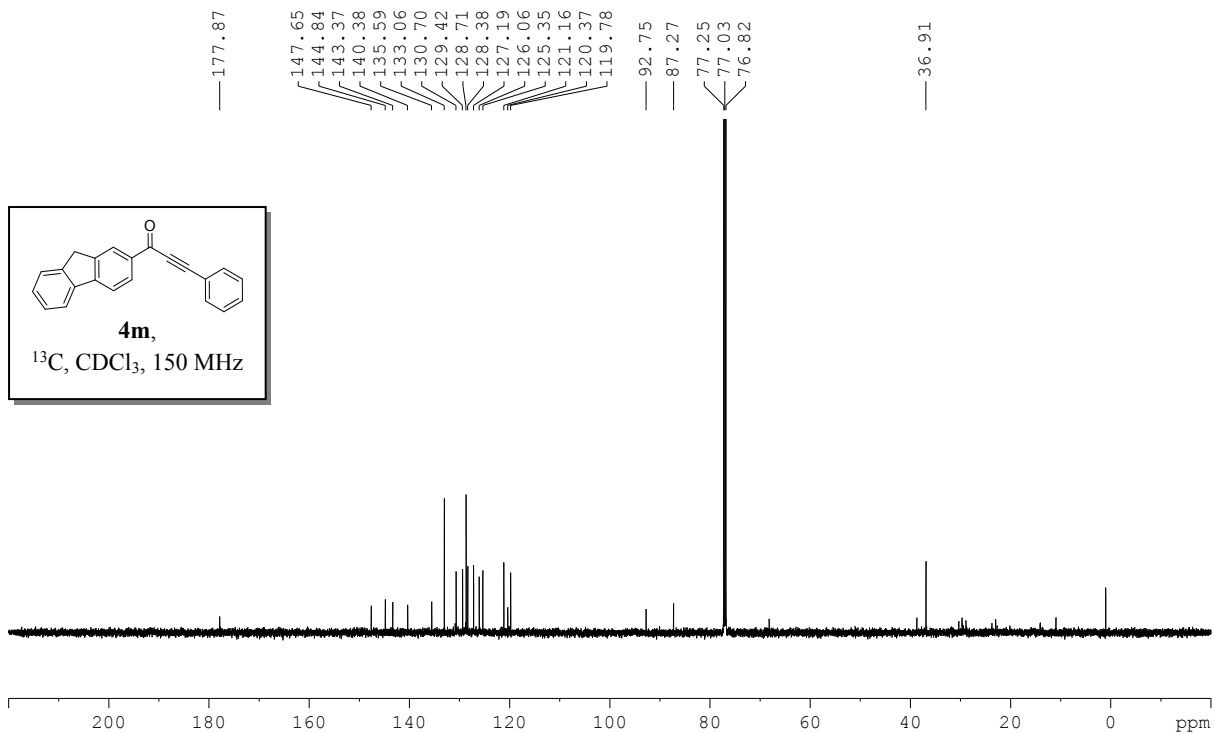




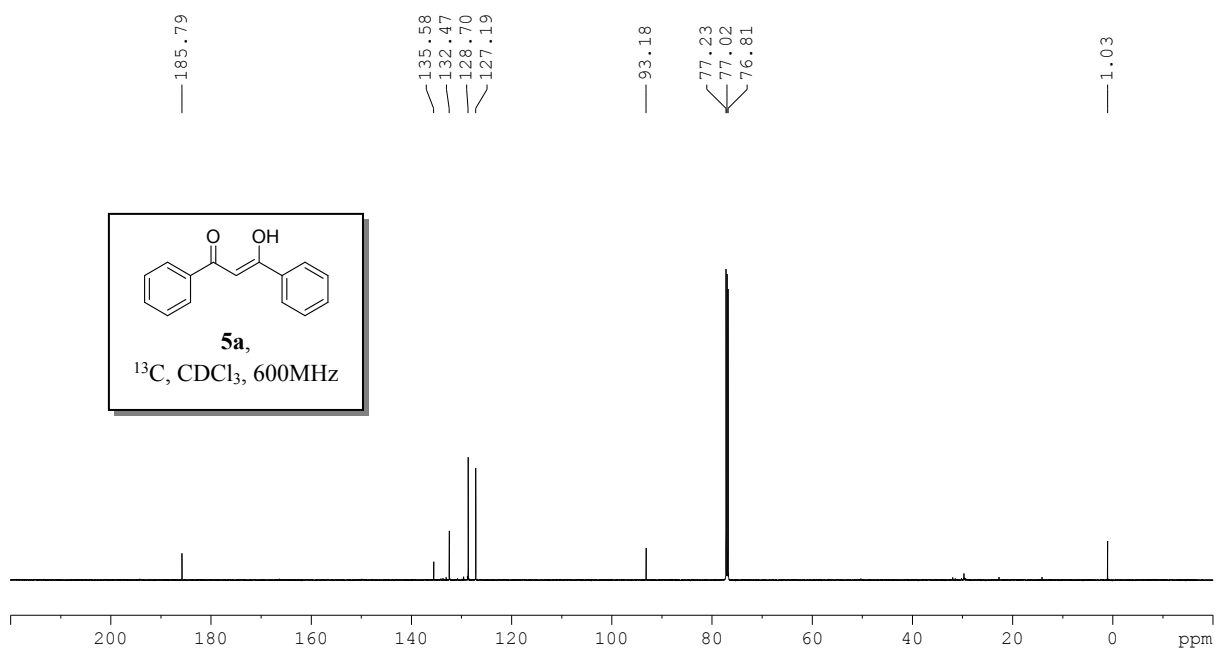
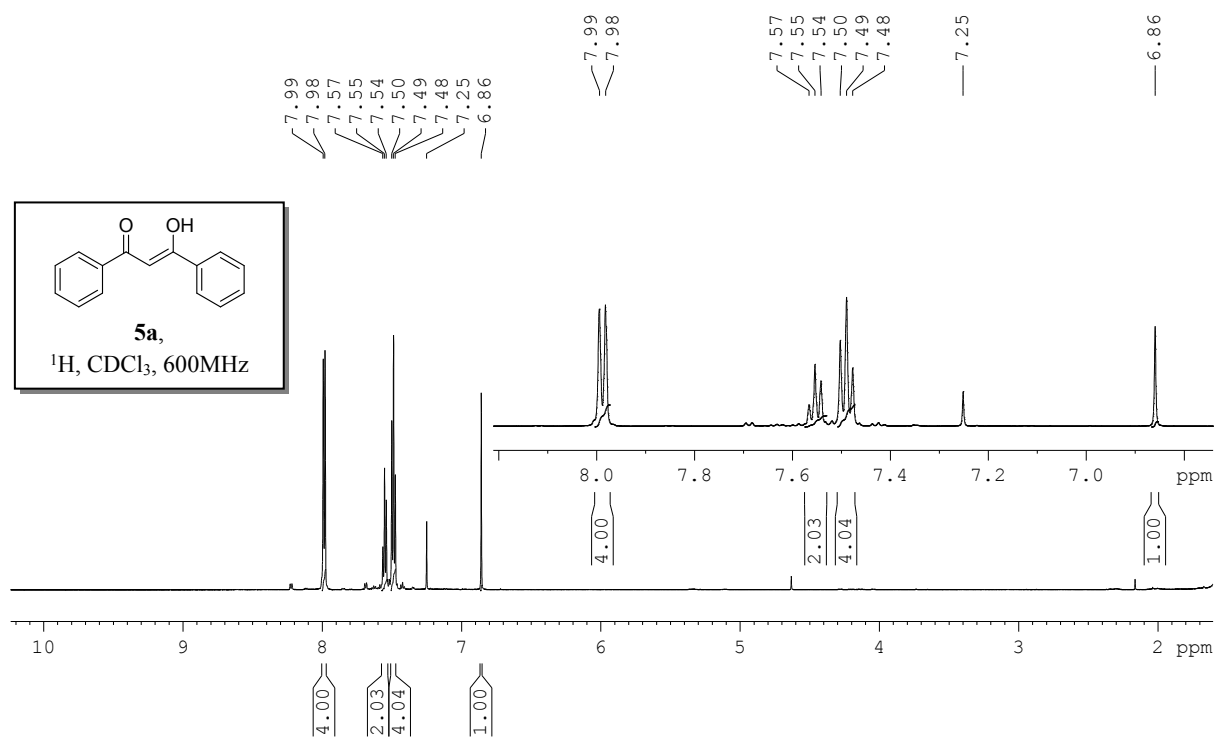
# 1-(9H-fluoren-2-yl)-3-phenylprop-2-yn-1-one

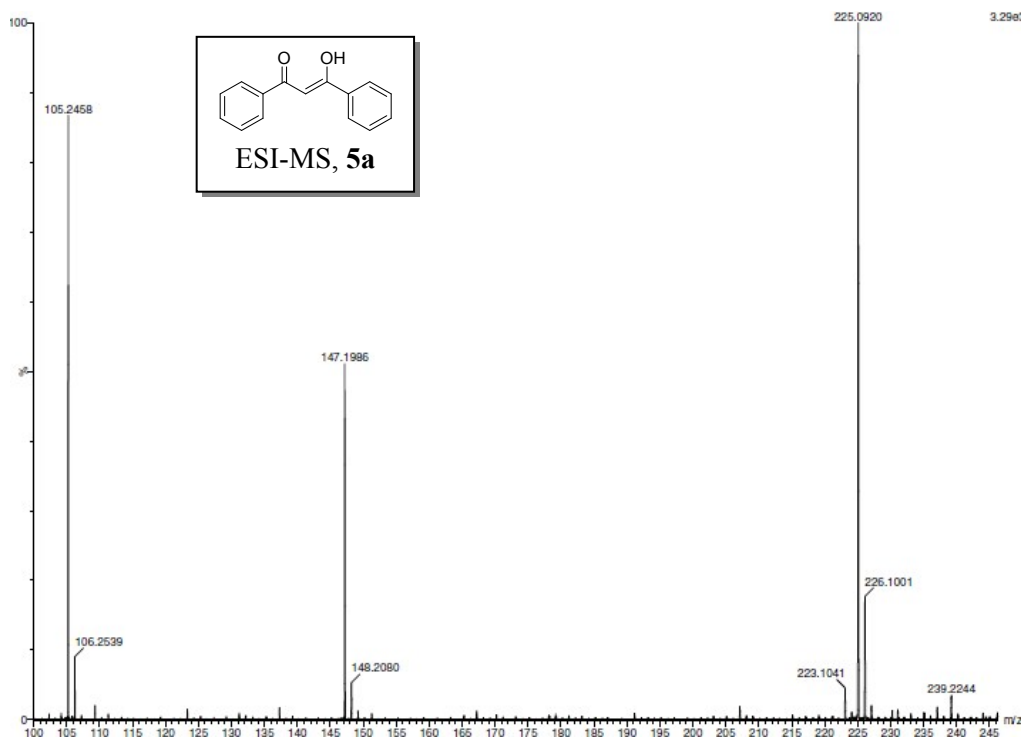




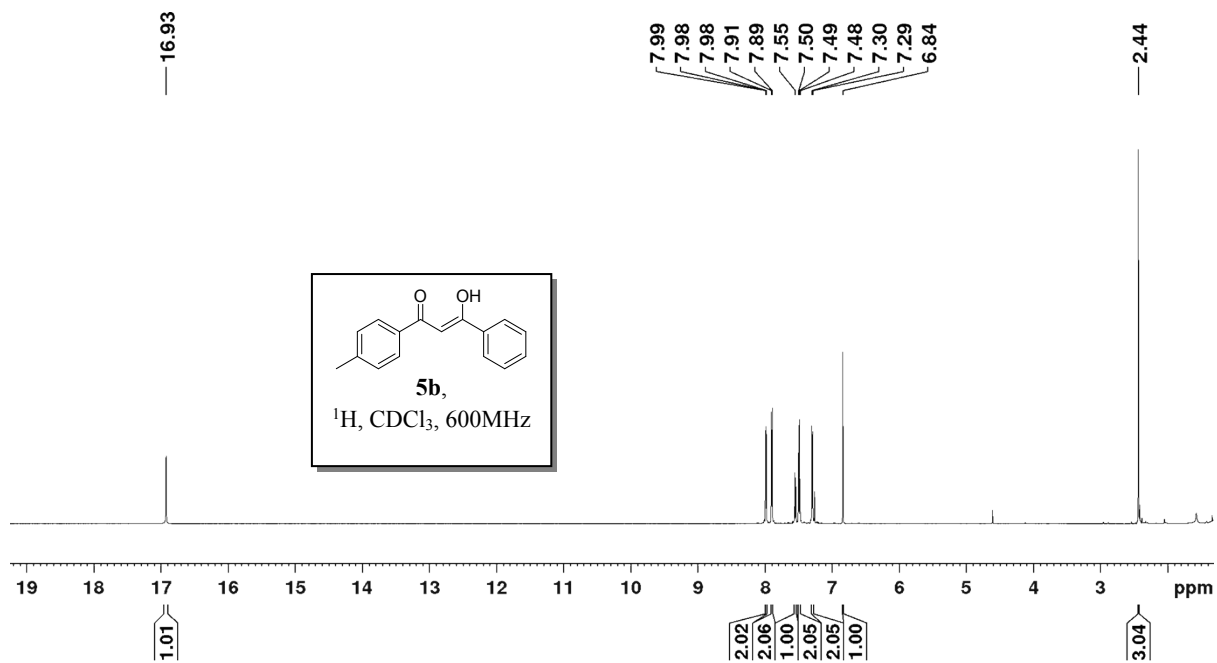


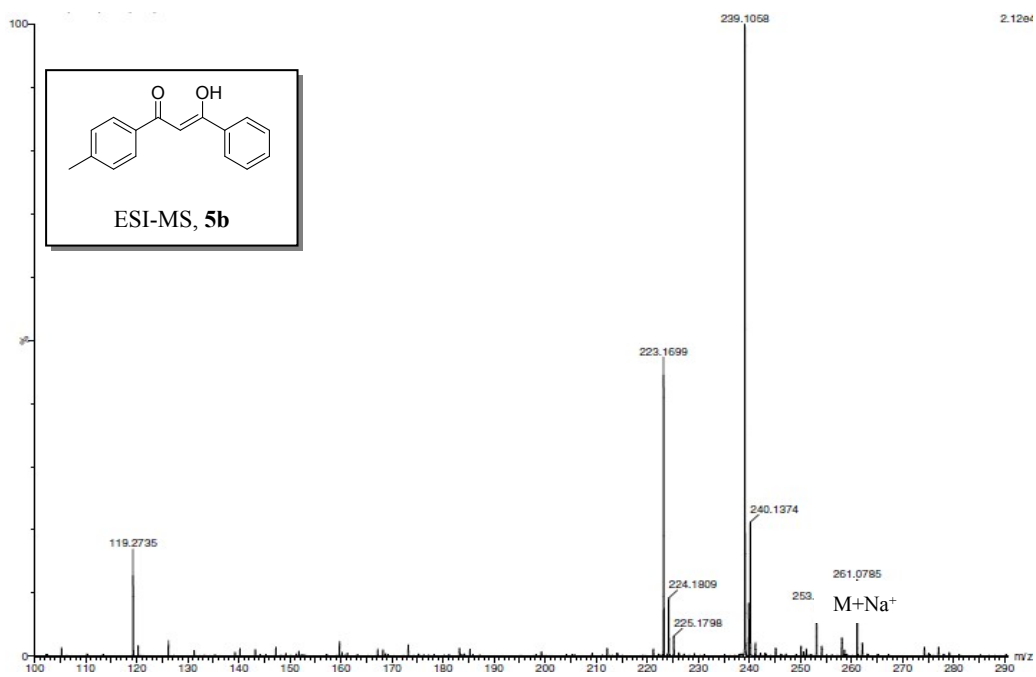
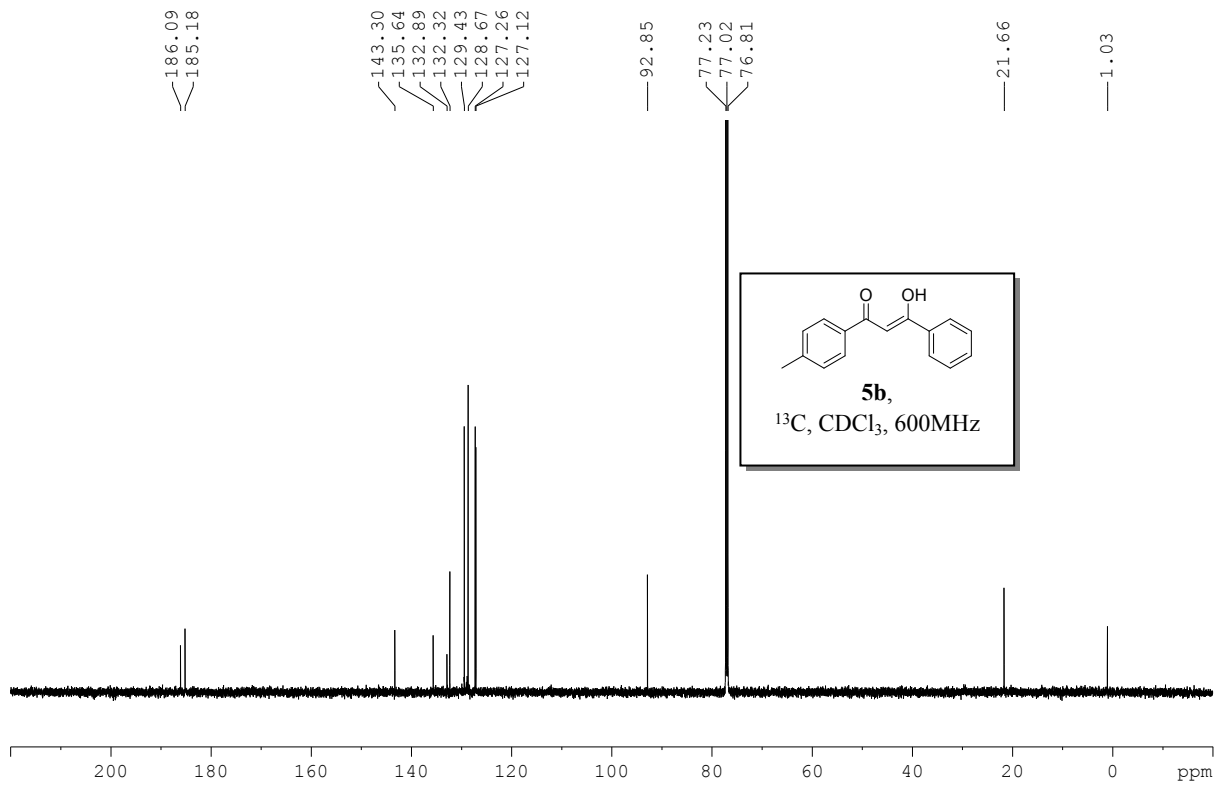
**(Z)-3-hydroxy-1,3-diphenylprop-2-en-1-one**



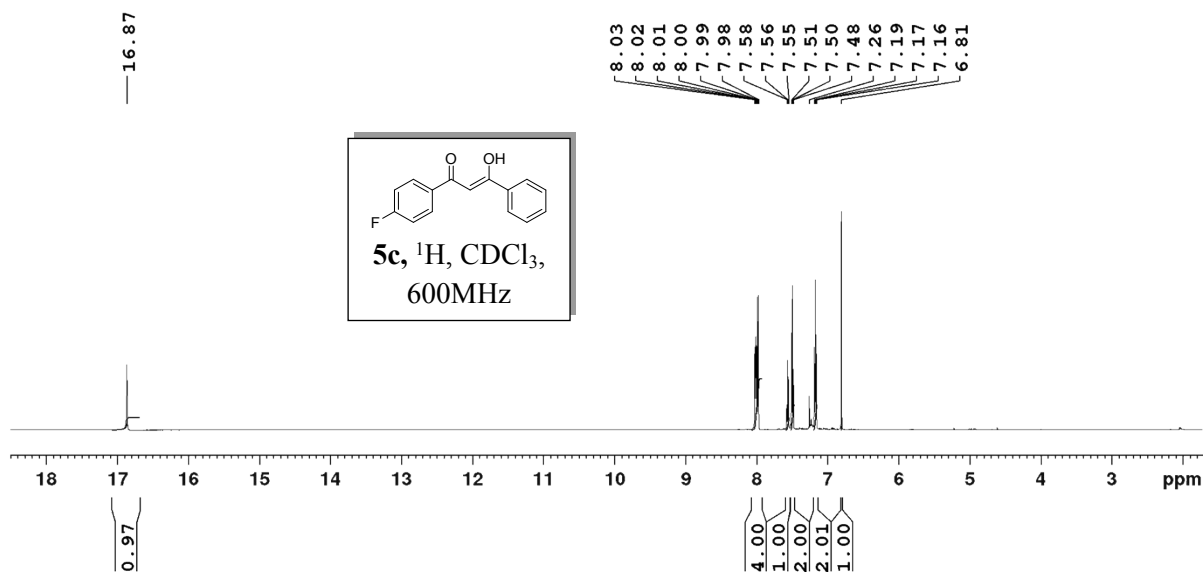
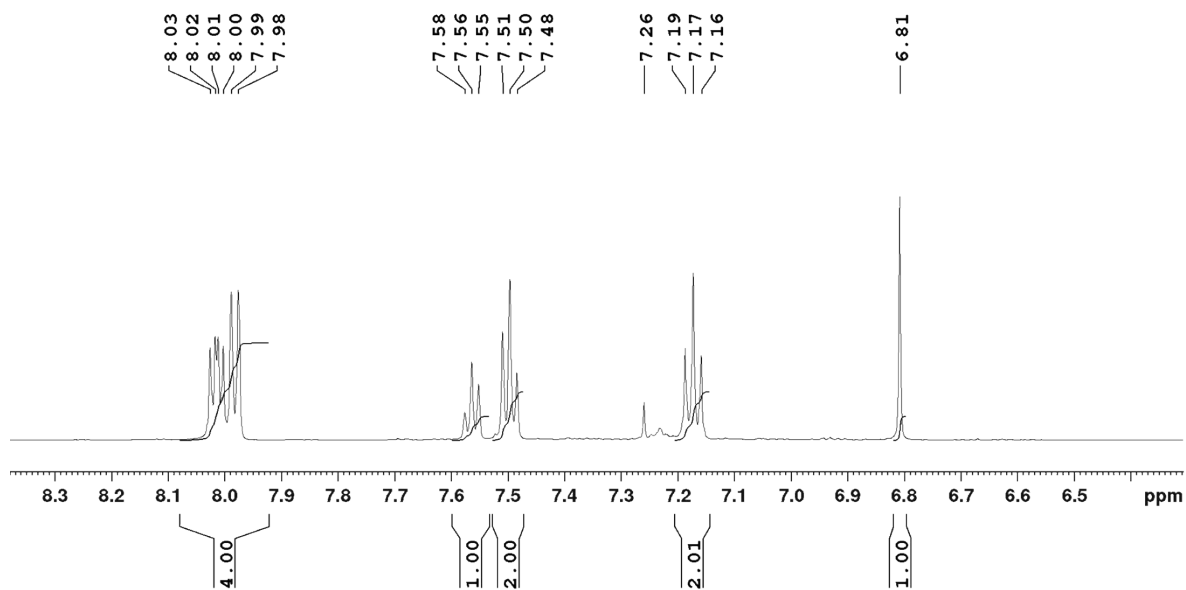


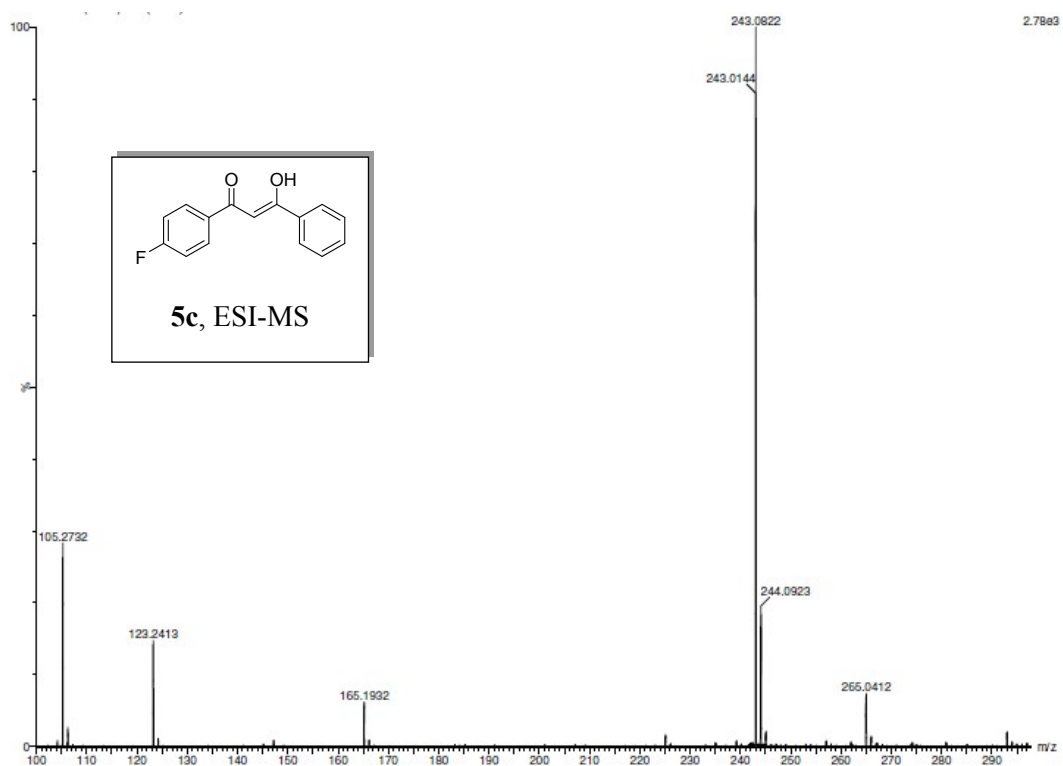
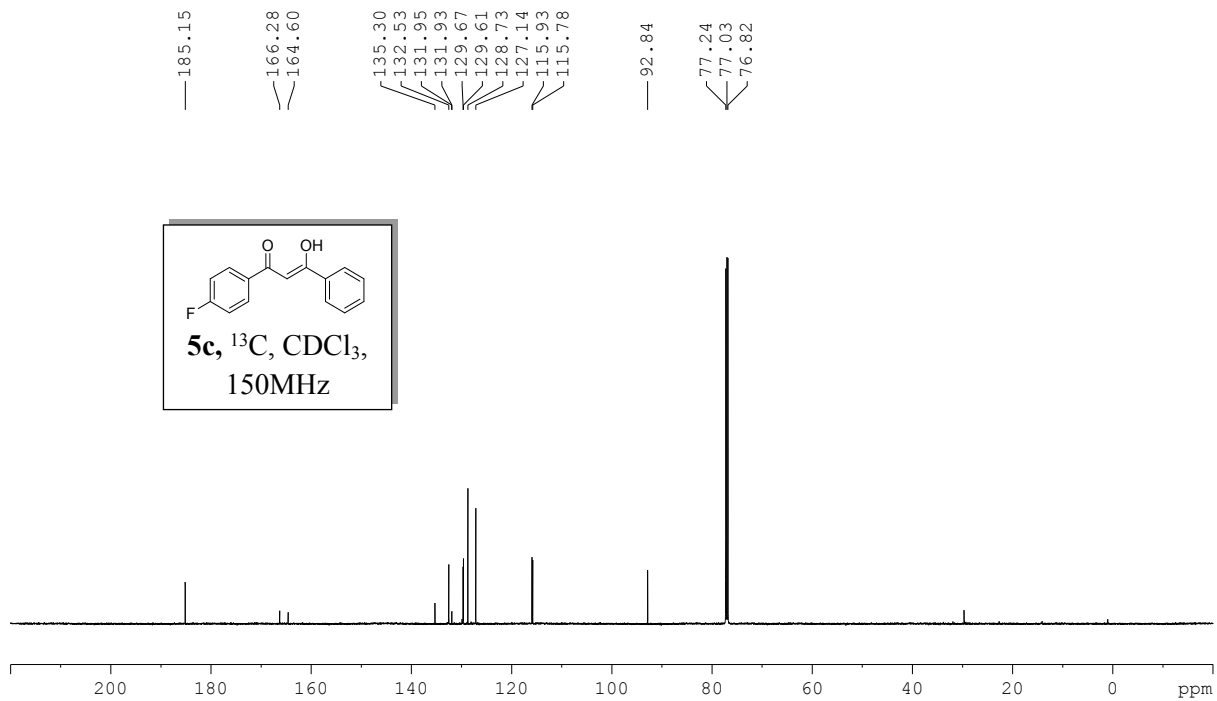
**(Z)-3-hydroxy-3-phenyl-1-(p-tolyl)prop-2-en-1-one**





**(Z)-1-(4-fluorophenyl)-3-hydroxy-3-phenylprop-2-en-1-one**





# = Water in  $\text{CDCl}_3$

## D. References

1. Sun, G., Lei, M.; Hu L.; A facile and efficient method for the synthesis of alkynone by carbonylative Sonogashira coupling using  $\text{CHCl}_3$  as the CO source, *RSC Adv.*, **2016**, *6*, 28442-28446.
2. Peng, J.-B.; Wu, F.-P.; Li, C.-L.; Qi, X.; Wu, X.-F.; A Convenient and Efficient Palladium-Catalyzed Carbonylative Sonogashira Transformation with Formic Acid as the CO Source *Eur. J. Org. Chem.* **2017**, 1434–1437.
3. Schranck, J.; Tlili A.; Alsabeh P. G.; Neumann, H.; Stradiotto M.; Beller M.; Palladium-Catalysed Carbonylative  $\alpha$ -Arylation of Acetone and Acetophenones to 1, 3-Diketones *Chem. Eur. J.*, **2013**, *19*, 12624 – 12628.