

## Supporting Information

# Photocatalytic Synthesis of Alkynylsulfones and Alkenylsulfones Using Sulfonylhydrazides and Alkynes

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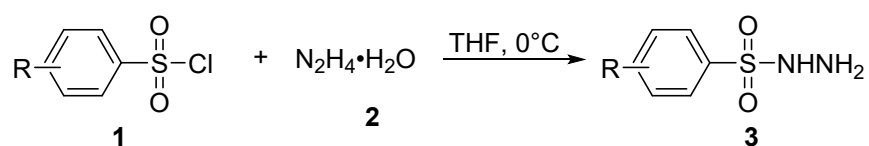
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## General information

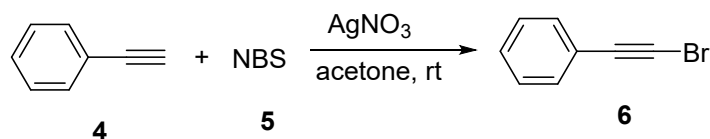
Bromoalkyne were synthesized according to literature procedure. A part of Arylsulfonylhydrazides were synthesized according to literature procedure. Other commercially available reagents and solvents were purchased and used without further purification. All catalytic experiments were performed under an atmosphere of argon by using Glove Box.  $^1\text{H}$  NMR (400 MHz) and  $^{13}\text{C}$  NMR (100 MHz) spectra were recorded on a Bruker NMR spectrometer in  $\text{CDCl}_3$  using TMS as an internal reference with chemical shift values reported in ppm. Abbreviations used in the NMR follow-up experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. High-resolution mass spectra (HRMS) were obtained by fast atom bombardment (FAB) using a double focusing magnetic sector mass spectrometer and electron impact ( $\text{EI}^+$ ) ionization technique.

### General procedure for synthesis of substrates

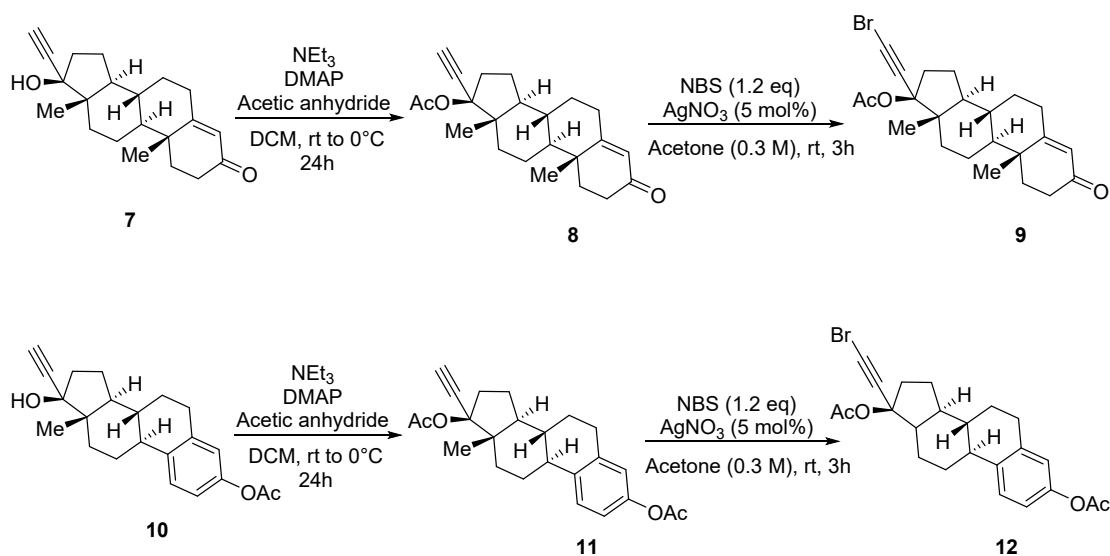


Preparation of sulfonyl hydrazides. A water (30mL) solution of hydrazine hydrate (0.5 g, 10 mmol), was cooled in ice-water bath to  $-5^\circ\text{C}$ . The temperature was then maintained below  $8^\circ\text{C}$ , while the THF(10 mL) solution of sulfonyl chloride (0.3 g, 1 mmol) was slowly added with stirring. After the addition was completed the reaction mixture was stirred

at room temperature for thirty minutes and tetrahydrofuran was evaporated at reduced pressure. The white solid product was separated by filtration, washed with water (3×15 mL) and dried on air overnight to give pure product (90% yield).<sup>[1]</sup>



Synthesis of aryl halide:  $\text{AgNO}_3$  (0.17 g, 1 mmol) was added to a solution of phenylacetylene (2.58 mL, 20 mmol) in acetone (50 mL). Then NBS (4.27 g, 24 mmol) was added in portion. The mixture was stirred for 3 h at room temperature, and then concentrated in vacuo. The residue was dissolved in petroleum ether and filtered through a short column of silica gel. Solvent was removed in vacuo to afford a pale-yellow oil of bromoalkyne (99% yield).<sup>[2]</sup>

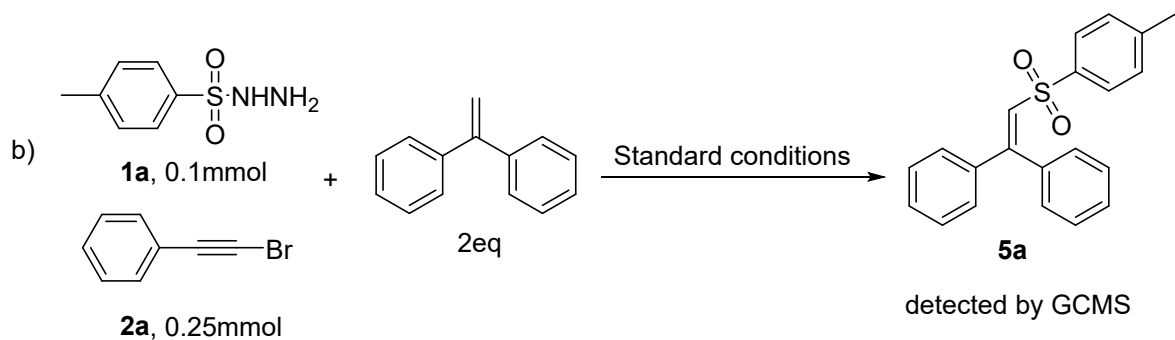
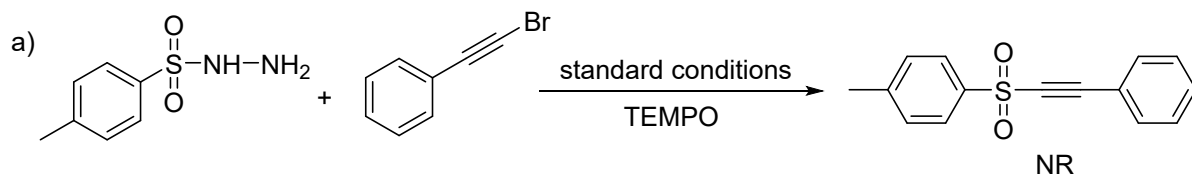
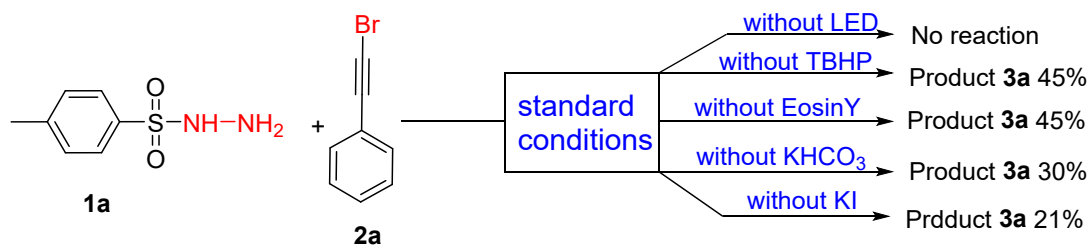


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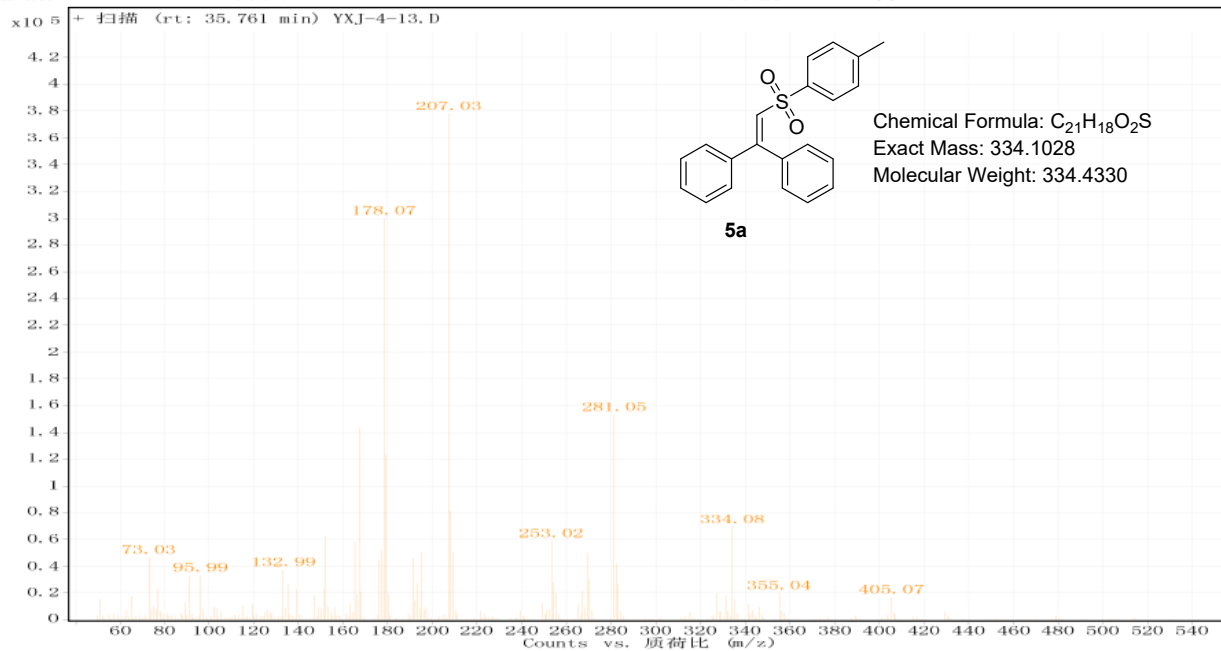
Synthesis of bromo ethisterone: DMAP (1.4 mmol),  $\text{NEt}_3$  (4 mmol) and acetic anhydride (4 mmol) were added to a solution of ethisterone (1.4 mmol) in  $\text{CH}_2\text{Cl}_2$  (0.1 M) at 0 °C. The reaction mixture was stirred for 24 h at room temperature and then concentrated. The residue was purified by chromatography (Hexane: Ethyl acetate= 80: 20) to give the ethisterone acetate in 98% yield.<sup>[3]</sup>

To a solution of ethisterone acetate (1 equiv) in acetone (0.3 M) was added NBS (1.2 equiv) and  $\text{AgNO}_3$  (5 mol%) at room temperature and then the mixture was stirred for 3 h. After removing excess acetone, the concentrated residue was quenched with  $\text{NH}_4\text{Cl}$  solution. Organic layer was extracted with diethyl ether (20 ml x 2), dried over anhydrous  $\text{Na}_2\text{SO}_4$ , concentrated under reduced pressure, and chromatographed to give 62% yield (Hexane: Ethyl acetate= 85: 15).

## Control experiments

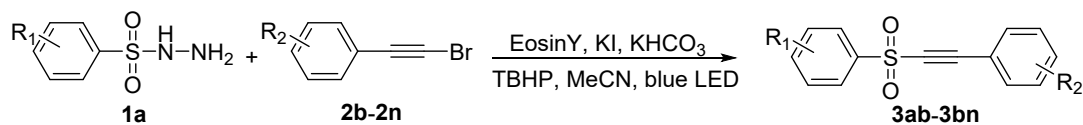


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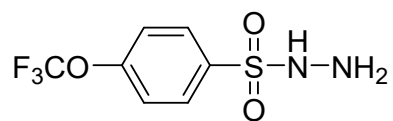
## General experimental methods



Under Ar or  $\text{N}_2$  atmosphere, to an oven-dried Schlenk tube was equipped with magnetic stir bar and charged with arylsulfonyl hydrazides (0.1 mmol, 1.0 equiv), bromoalkyne (0.25 mmol, 2.5 equiv), Eosin Y (0.002 mmol, 0.02 equiv), KI (0.1 mmol, 1.0 equiv),  $\text{KHCO}_3$  (0.1 mmol, 1.0 equiv), TBHP (0.1 mmol, 1.0 equiv), and MeCN (1 mL). The mixture was then irradiated by 30 W blue LED (460 nm-470 nm) at room temperature until the starting material disappeared from the TLC. The reaction mixture was concentrated in vacuum and the residue was purified by chromatography on silica gel, eluting with the mixture of ethyl acetate/petroleum ether to give alkynyl sulfones products.

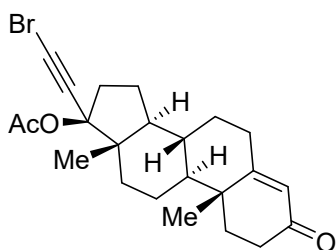
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$^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra data of products or materials



**4-(trifluoromethoxy)benzenesulfonylhydrazide(3)**

$^1\text{H}$  NMR (400 MHz, Methanol- $d_4$ )  $\delta$  8.01 (d,  $J = 8.9$  Hz, 2H), 7.49 (d,  $J = 8.2$  Hz, 2H), 4.85 (s, 2H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  146.7, 139.1, 134.8, 129.1, 128.8, 128.7, 128.0, 127.4.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -57.69. HRMS-EI $^+$  ( $m/z$ )  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_7\text{H}_7\text{N}_2\text{O}_3\text{F}_3\text{S}$  257.0202, found 257.0201.

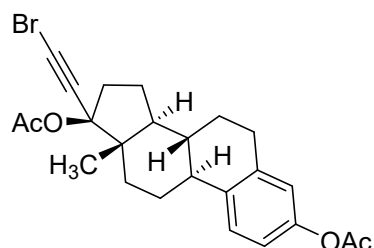


**(8R,9S,10R,13S,14S,17S)-17-(bromoethynyl)-10,13-dimethyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-17-yl acetate.(9)<sup>[3]</sup>**

$^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  5.74 (s, 1H), 2.72 (td,  $J = 9.5, 4.8$  Hz, 1H), 2.48 – 2.26 (m, 5H), 2.03 – 1.97 (m, 1H), 1.81 – 1.66 (m, 6H), 1.57 (td,  $J = 11.0, 3.4$  Hz, 1H), 1.52 – 1.31 (m, 4H), 1.20 (s, 4H), 1.14 – 0.97 (m, 3H), 0.90 (s, 3H).

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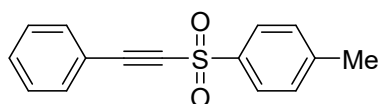
$^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  199.5, 170.9, 169.4, 124.0, 85.1, 53.2, 48.4, 47.9, 46.7, 38.6, 37.1, 36.0, 35.7, 34.0, 33.0, 32.7, 31.5, 23.6, 21.4, 20.7, 17.4, 13.5.



**(8R,9S,13S,14S,17S)-17-(bromoethynyl)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthrene-3,17-diyl diacetate.**(12)<sup>[3]</sup>

$^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.31 – 7.26 (m, 1H), 6.85 (dd,  $J = 8.4, 2.6$  Hz, 1H), 6.79 (d,  $J = 2.5$  Hz, 1H), 2.87 (dd,  $J = 8.4, 4.1$  Hz, 2H), 2.76 (m,  $J = 15.1, 9.7, 5.9$  Hz, 1H), 2.37 (dt,  $J = 12.8, 3.6$  Hz, 1H), 2.29 (s, 3H), 2.05 (d,  $J = 1.4$  Hz, 3H), 2.02 – 1.79 (m, 4H), 1.72 – 1.62 (m, 1H), 1.56 – 1.34 (m, 4H), 0.89 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  169.9, 169.5, 148.4, 138.2, 137.8, 126.5, 121.5, 118.6, 85.3, 48.3, 48.0, 46.6, 43.6, 38.8, 37.1, 33.3, 29.5, 27.1, 26.2, 23.4, 21.4, 21.2, 13.5.



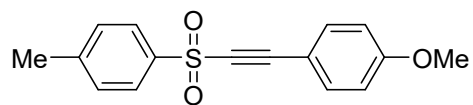
**1-methyl-4-((phenylethynyl)sulfonyl)benzene (3aa)**<sup>[4]</sup>

21.8 mg, 85% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.99 – 7.94 (m, 2H), 7.54 – 7.50 (m, 2H), 7.50 – 7.44 (m, 1H), 7.38 (dt,



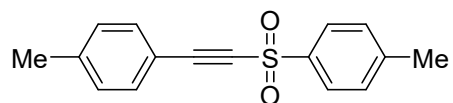
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$J = 8.6, 7.2$  Hz, 4H), 2.47 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  145.4, 139.0, 132.7, 131.5, 130.0, 128.7, 127.5, 118.0, 93.0, 85.6, 21.7.



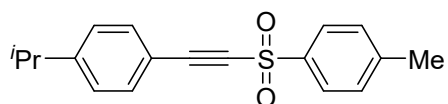
**1-methoxy-4-(tosylethynyl)benzene(3ab)<sup>[4]</sup>**

25.8 mg, 90% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.98 – 7.92 (m, 2H), 7.49 – 7.44 (m, 2H), 7.40 – 7.36 (m, 2H), 6.90 – 6.84 (m, 2H), 3.83 (s, 3H), 2.46 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  145.3, 139.1, 138.6, 133.2, 132.4, 123.0, 129.9, 128.6, 127.5, 117.8, 93.4, 85.3, 21.7, 21.1.



**1-methyl-4-((p-tolyethynyl)sulfonyl)benzene (3ac)<sup>[4]</sup>**

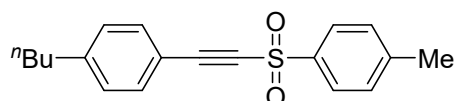
23.0 mg, 82% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (d,  $J = 8.1$  Hz, 2H), 7.39 (t,  $J = 8.3$  Hz, 4H), 7.16 (d,  $J = 7.9$  Hz, 2H), 2.46 (s, 3H), 2.37 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  145.2, 142.3, 139.1, 132.7, 130.0, 129.5, 127.5, 114.9, 93.7, 85.2, 21.8, 21.7.



**1-isopropyl-4-(tosylethynyl)benzene (3ad)<sup>[4]</sup>**

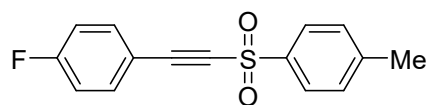
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23.9 mg, 77% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (d,  $J = 8.4$  Hz, 2H), 7.43 (s, 2H), 7.38 (d,  $J = 7.9$  Hz, 2H), 7.22 (d,  $J = 8.2$  Hz, 2H), 2.91 (p,  $J = 6.9$  Hz, 1H), 2.46 (s, 3H), 1.22 (d,  $J = 6.9$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  153.1, 145.2, 139.2, 132.9, 130.0, 127.4, 126.9, 115.2, 93.8, 85.1, 34.3, 23.6, 21.7.



### 1-butyl-4-(tosylethynyl)benzene (3ae)<sup>[5]</sup>

27.5 mg, 88% yield; colorless liquid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.99 – 7.93 (m, 2H), 7.46 – 7.35 (m, 4H), 7.20 – 7.14 (m, 2H), 2.65 – 2.58 (m, 2H), 2.46 (s, 3H), 1.61 – 1.52 (m, 2H), 1.31 (tt,  $J = 14.1, 7.1$  Hz, 2H), 0.91 (t,  $J = 7.3$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  147.3, 145.3, 139.1, 132.7, 130.0, 128.8, 127.4, 115.0, 93.8, 85.2, 35.8, 33.1, 22.3, 21.8, 13.9.



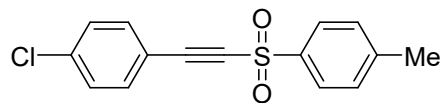
### 1-fluoro-4-(tosylethynyl)benzene (3af)<sup>[4]</sup>

20.6 mg, 71% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.95 (d,  $J = 8.4$  Hz, 2H), 7.53 (dd,  $J = 8.9, 5.2$  Hz, 2H), 7.41 (s, 2H), 7.07 (t,  $J = 8.7$  Hz, 2H), 2.47 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  164.4 (d,  $^1J_{\text{C-F}} = 253$  Hz), 145.5, 138.8, 135.1, 135.1 (d,  $^3J_{\text{C-F}} = 1.0$

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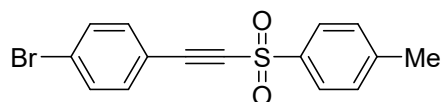
H<sub>z</sub>), 127.5, 116.3 (d, <sup>2</sup>J<sub>C-F</sub> = 23.2 Hz), 114.2, 114.2, 91.8, 85.6, 85.6, 21.8.

<sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -104.62.



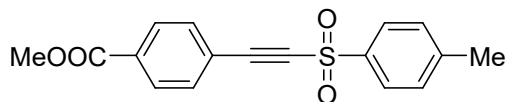
**1-chloro-4-(tosylethynyl)benzene (3ag)**<sup>[4]</sup>

25.6 mg, 88% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 7.38 (dd, *J* = 18.7, 8.3 Hz, 4H), 2.48 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 145.6, 138.7, 138.0, 133.9, 130.1, 129.2, 127.6, 116.5, 91.5, 86.5, 21.8.



**1-butyl-4-(tosylethynyl)benzene (3ah)**<sup>[4]</sup>

30.2 mg, 90% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.43 – 7.35 (m, 4H), 2.48 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 145.6, 138.7, 134.0, 132.1, 130.1, 127.6, 126.5, 117.0, 91.6, 86.6, 21.8.

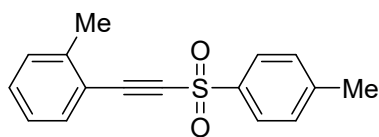


**1-methyl 4-(tosylethynyl)benzoate (3ai)**<sup>[4]</sup>

9.5 mg, 30% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.06 – 7.94 (m, 4H), 7.61 – 7.57 (m, 2H), 7.44 – 7.38 (m, 2H), 3.93 (s,

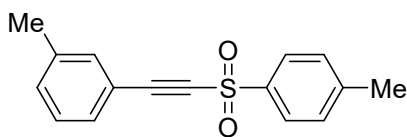
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3H), 2.48 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.8, 145.7, 138.6, 132.7, 132.4, 130.1, 129.7, 127.7, 122.4, 91.2, 87.6, 52.5, 21.8.



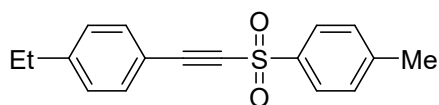
**1-methyl-2-(tosylethynyl)benzene (3aj)**<sup>[6]</sup>

23.5 mg, 87% yield, yellow oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.99 – 7.94 (m, 2H), 7.46 (dd,  $J$  = 7.8, 1.4 Hz, 1H), 7.41 – 7.32 (m, 3H), 7.24–7.14 (m, 2H), 2.47 (s, 3H), 2.38 (s, 3H).  $^{13}\text{C}$  NMR (101MHz, Chloroform-*d*)  $\delta$  145.3, 142.4, 139.3, 133.0, 131.5, 130.0, 129.9, 127.4, 125.9, 117.9, 92.6, 89.3, 21.7, 20.4.



**1-methyl-3-(tosylethynyl)benzene (3ak)**<sup>[4]</sup>

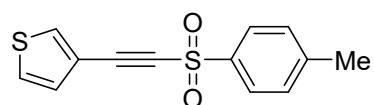
21.8 mg, 80% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.96 (d,  $J$  = 8.4 Hz, 2H), 7.39 (d,  $J$  = 8.1 Hz, 2H), 7.32 (dd,  $J$  = 8.8, 1.9 Hz, 2H), 7.30 – 7.22 (m, 2H), 2.47 (s, 3H), 2.33 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.1, 145.1, 139.3, 134.7, 129.9, 127.4, 114.4, 109.7, 94.1, 84.9, 55.5, 21.7.



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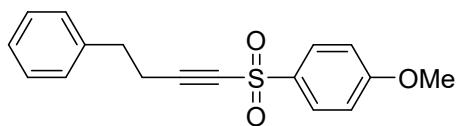
### 1-ethyl-4-(tosylethynyl)benzene (3al)<sup>[7]</sup>

25.6 mg, 90% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.41 (dd, *J* = 20.1, 8.2 Hz, 4H), 7.19 (d, *J* = 8.2 Hz, 2H), 2.66 (q, *J* = 7.6 Hz, 2H), 2.46 (s, 3H), 1.21 (t, *J* = 7.6 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 148.5, 145.2, 139.2, 132.8, 130.0, 128.3, 127.5, 115.1, 93.7, 85.2, 29.0, 21.7, 15.1.



### 3-(tosylethynyl)thiophene (3am)<sup>[4]</sup>

19.7 mg, 71% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.97 – 7.93 (m, 2H), 7.74 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 2H), 7.32 (dd, *J* = 5.1, 3.0 Hz, 1H), 7.17 (dd, *J* = 5.1, 1.2 Hz, 1H), 2.47 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 144.3, 137.9, 133.5, 129.0, 128.7, 126.5, 125.5, 116.2, 87.6, 84.5, 20.7.



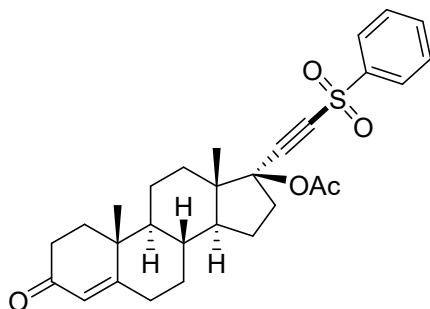
### 1-methoxy-4-((4-phenylbut-1-yn-1-yl)sulfonyl)benzene (3an)

12.6 mg, 42% yield; colorless oil, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.85 (m, 2H), 7.27 – 7.21 (m, 3H), 7.13 – 7.09 (m, 2H), 7.04 – 6.98 (m, 2H), 3.90 (s, 3H), 2.84 (t, *J* = 7.4 Hz, 2H), 2.64 (td, *J* = 7.4, 0.6 Hz, 2H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 164.0, 139.0, 129.6,

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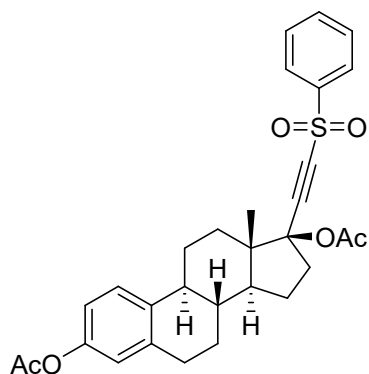
128.6, 128.3, 126.8, 114.4, 95.5, 55.8, 55.7, 33.2, 21.2. HRMS-EI<sup>+</sup> (m/z)

[M+Na]<sup>+</sup> calcd for 323.0712, found 323.0712.



**(10aR,11bR)-10a,11b-dimethyl-3-oxo-9-((phenylsulfonyl)ethynyl)-2,3,5,6,6a,7,7a,8,9,10,10a,11,11a,11b-tetradecahydro-1H-cyclopenta[b]phenanthren-9-yl acetate.(3ao)**

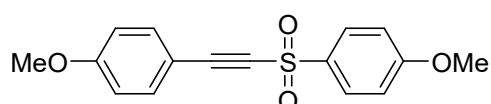
<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 7.6 Hz, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 2H), 5.75 (s, 1H), 2.69-2.40 (m, 1H), 2.45 – 2.26 (m, 5H), 2.01 (s, 4H), 1.83 – 1.67 (m, 5H), 1.55 (dd, *J* = 11.4, 3.6 Hz, 2H), 1.44 – 1.32 (m, 3H), 1.17 (s, 3H), 0.99 (dd, *J* = 12.4, 4.4 Hz, 2H), 0.89 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 199.5, 170.5, 169.1, 142.1, 134.3, 129.5, 127.3, 124.2, 95.3, 83.6, 83.3, 53.1, 49.2, 48.8, 38.6, 36.8, 35.9, 35.8, 34.1, 33.1, 32.7, 31.5, 23.7, 21.1, 20.6, 17.5, 13.6. TOFMS-ESI<sup>+</sup> (m/z) [M+H]<sup>+</sup>calcd for C<sub>29</sub>H<sub>34</sub>O<sub>5</sub>S 495.21997, found 495.21903. m.p.: 198 °C-200 °C.



**(13S)-13-methyl-17-((phenylsulfonyl)ethynyl)-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthrene-3,17-diyl diacetate.(3ap)**

$^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.03 (dd,  $J = 7.3, 1.7$  Hz, 2H), 7.69 – 7.53 (m, 3H), 7.28 – 7.21 (m, 1H), 6.89 – 6.77 (m, 2H), 2.85 (dd,  $J = 8.9, 4.3$  Hz, 2H), 2.29 (s, 3H), 2.23 – 2.07 (m, 2H), 2.02 (s, 4H), 1.86 – 1.75 (m, 3H), 1.61 (m,  $J = 13.0, 4.1$  Hz, 1H), 1.42 (m,  $J = 19.5, 8.3, 6.1$  Hz, 5H), 0.87 (s, 3H).

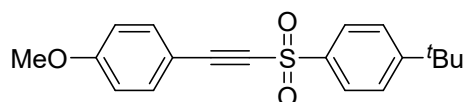
$^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  169.9, 169.0, 148.5, 142.1, 138.0, 137.3, 134.1, 129.3, 127.2, 126.4, 121.6, 118.8, 95.5, 83.3, 49.2, 48.7, 43.4, 38.7, 36.8, 33.2, 29.4, 27.0, 26.0, 23.3, 21.2, 21.0, 13.4. TOFMS-ESI<sup>+</sup> ( $m/z$ )  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{30}\text{H}_{32}\text{O}_6\text{S}$  521.19924, found 521.19885. m.p.: 170 °C-175 °C



**1-methoxy-4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzene (3bb)<sup>[8]</sup>**

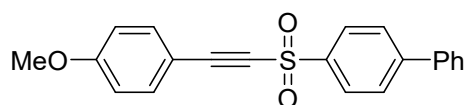
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25.7 mg, 80% yield; yellow oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.99 (d,  $J = 8.9$  Hz, 2H), 7.45 (d,  $J = 8.9$  Hz, 2H), 7.04 (d,  $J = 9.0$  Hz, 2H), 6.86 (d,  $J = 8.9$  Hz, 2H), 3.89 (s, 3H), 3.82 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  164.1, 162.1, 134.6, 133.8, 129.7, 114.5, 114.4, 109.8, 93.6, 85.1, 55.8, 55.5.



**1-(tert-butyl)-4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzene (3b c)**<sup>[6]</sup>

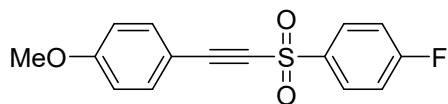
32.2 mg, 98% yield; yellow oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.98 (d,  $J = 8.7$  Hz, 2H), 7.62 – 7.57 (m, 2H), 7.50 – 7.45 (m, 2H), 6.90 – 6.84 (m, 2H), 3.82 (s, 3H), 1.36 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.1, 158.0, 139.1, 134.7, 127.2, 126.4, 114.4, 109.7, 94.1, 84.9, 55.5, 35.4, 31.1.



**4-((phenylethynyl)sulfonyl)-1,1'-biphenyl (3bd)**<sup>[9]</sup>

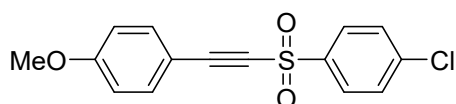
26.7 mg, 85% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.13 (d,  $J = 8.9$  Hz, 2H), 7.53 – 7.38 (m, 5H), 6.88 (d,  $J = 8.9$  Hz, 2H), 3.83 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.4, 153.1, 153.1, 140.3, 134.8, 129.7, 121.0, 114.5, 109.2, 95.5, 84.3, 55.5.





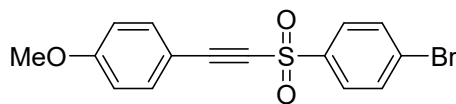
**1-fluoro-4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzene (3be)**<sup>[10]</sup>

21.8 mg, 75% yield; yellow oil, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.14 – 8.06 (m, 2H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.31 – 7.22 (m, 2H), 6.93 – 6.85 (m, 2H), 3.83 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 166.0 (d, <sup>1</sup>*J*<sub>C-F</sub> = 257.6 Hz) 162.3, 138.2, 138.2, 134.8, 130.4, 130.3, 116.7 (d, <sup>2</sup>*J*<sub>C-F</sub> = 23.2 Hz), 114.49, 114.49, 109.3, 94.9, 84.5, 55.5. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -102.68.



**1-chloro-4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzene (3bf)**<sup>[10]</sup>

29.5 mg, 96% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.02 – 7.99 (m, 2H), 7.59 – 7.54 (m, 2H), 7.50 – 7.45 (m, 2H), 6.91 – 6.85 (m, 2H), 3.83 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 162.4, 140.7, 140.6, 134.8, 129.7, 128.8, 114.5, 109.2, 95.3, 84.4, 55.5.

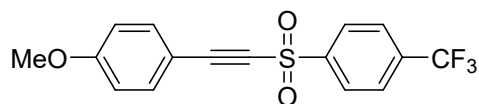


**1-bromo-4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzene (3bg)**<sup>[10]</sup>

29.9 mg, 85% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.97 – 7.90 (m, 2H), 7.76 – 7.70 (m, 2H), 7.51 – 7.45 (m, 2H), 6.92 –

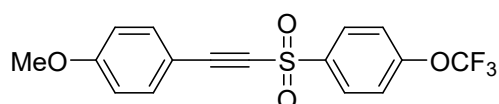
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6.85 (m, 2H), 3.83 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.4, 141.1, 134.8, 132.7, 132.5, 129.4, 129.0, 128.9, 114.5, 109.2, 95.4, 84.3, 55.5.



**1-methoxy-4-(((4-(trifluoromethyl)phenyl)sulfonyl)ethynyl)benzene (3bh)<sup>[10]</sup>**

27.2 mg, 80% yield; yellow solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.21 (d,  $J = 8.1$  Hz, 2H), 7.86 (d,  $J = 8.3$  Hz, 2H), 7.53 – 7.46 (m, 2H), 6.92 – 6.86 (m, 2H), 3.84 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.5, 145.5, 135.7, 135.4, 134.9, 127.9, 126.6, 126.5, 126.5, 126.5, 124.5, 121.8, 114.6, 109.0, 96.3, 84.0, 55.5.

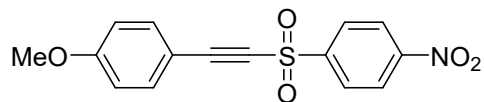


**1-((phenylethynyl)sulfonyl)-4-(trifluoromethoxy)benzene (3bi)**

32.1 mg, 90% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.16 – 8.10 (m, 2H), 7.53 – 7.46 (m, 2H), 7.41 (d,  $J = 9.0$  Hz, 2H), 6.93 – 6.85 (m, 2H), 3.84 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.4, 153.1 (q,  $^2J_{\text{C-F}} = 4.0$  Hz), 140.3, 134.8, 129.7, 121.3 (q,  $^1J_{\text{C-F}} = 46.5$  Hz), 118.9, 114.5, 109.2, 95.5, 84.3, 55.5.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)

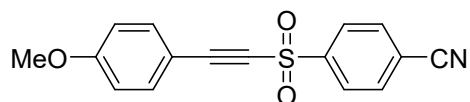
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$\delta$  -57.64, -57.65. HRMS-EI<sup>+</sup> (m/z) [M+H]<sup>+</sup> calcd for C<sub>16</sub>H<sub>11</sub>O<sub>4</sub>F<sub>3</sub>S 357.0403 found 357.0403. m.p.: 90 °C~ 93 °C.



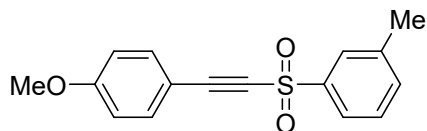
**1-methoxy-4-(((4-nitrophenyl)sulfonyl)ethynyl)benzene (3bj)**

17.5 mg, 50% yield; yellow solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.42 (s, 2H), 8.28 (s, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.9 Hz, 2H), 3.84 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  162.7, 150.8, 147.5, 135.0, 128.7, 124.6, 114.6, 108.7, 97.3, 83.7, 55.5. TOFMS-ESI<sup>+</sup> (m/z) [M+H]<sup>+</sup>calcd for C<sub>16</sub>H<sub>11</sub>NO<sub>3</sub> 298.0532, found 298.0521. m.p.: 135 °C~ 138 °C.



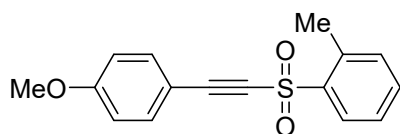
**4-(((4-methoxyphenyl)ethynyl)sulfonyl)benzonitrile (3bk)**

17.8 mg, 60% yield; white solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.23 – 8.17 (m, 2H), 7.92 – 7.87 (m, 2H), 7.52 – 7.47 (m, 2H), 6.93 – 6.87 (m, 2H), 3.84 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  162.7, 146.0, 135.0, 133.2, 128.0, 117.6, 117.1, 114.6, 108.8, 97.0, 83.8, 55.5. TOFMS-ESI<sup>+</sup> (m/z) [M+H]<sup>+</sup>calcd for C<sub>16</sub>H<sub>11</sub>NO<sub>3</sub> 318.0431, found 318.0431. m.p.: 150 °C~ 153 °C.



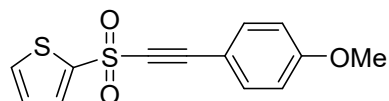
**1-(((4-methoxyphenyl)ethynyl)sulfonyl)-3-methylbenzene (3bl)**

27.2 mg, 95% yield; yellow oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 (d,  $J = 1.4$  Hz, 2H), 7.51 – 7.44 (m, 4H), 6.90 – 6.84 (m, 2H), 3.83 (s, 3H), 2.47 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.2, 141.9, 139.7, 134.8, 134.7, 129.2, 127.6, 124.5, 114.4, 109.6, 94.4, 84.7, 55.5, 21.4. HRMS-EI $^+$  ( $m/z$ ) [ $\text{M}+\text{H}$ ] $^+$  calcd for  $\text{C}_{16}\text{H}_{14}\text{O}_3\text{S}$  287.0736, found 287.0734.



**1-(((4-methoxyphenyl)ethynyl)sulfonyl)-2-methylbenzene (3bm)<sup>[8]</sup>**

25.5 mg, 89% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.12 (d,  $J = 9.4$  Hz, 1H), 7.58 – 7.34 (m, 5H), 6.88 (d,  $J = 8.9$  Hz, 2H), 3.83 (s, 3H), 2.82 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.2, 140.0, 138.3, 134.8, 133.9, 132.7, 128.6, 126.5, 114.5, 109.5, 93.1, 84.4, 55.5, 20.1.

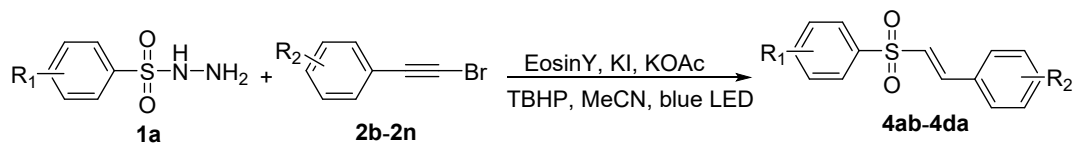


**2-(((4-methoxyphenyl)ethynyl)sulfonyl)thiophene (3bn)**

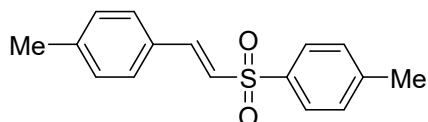
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20.9 mg, 75% yield; yellow oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.85 (dd,  $J = 3.9, 1.4$  Hz, 1H), 7.75 (dd,  $J = 5.0, 1.4$  Hz, 1H), 7.53 – 7.47 (m, 2H), 7.16 (dd,  $J = 5.0, 3.8$  Hz, 1H), 6.92 – 6.86 (m, 2H), 3.83 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  162.3, 143.4, 134.8, 134.5, 134.0, 127.9, 114.5, 109.4, 94.5, 85.0, 55.5. HRMS-EI $^+$  (m/z)  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{13}\text{H}_{10}\text{O}_3\text{S}_2$  279.0144, found 279.0141.

## General procedure for the synthesis of (*E*)-Vinyl Sulfones

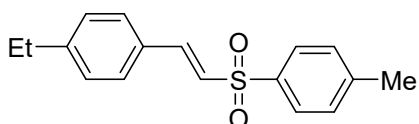


### (*E*)-1-methyl-4-((4-methylstyryl)sulfonyl)benzene (4ac)<sup>[11]</sup>



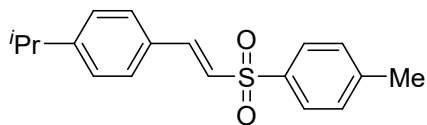
12.3 mg, 42 yield; White solid, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86 – 7.79 (m, 2H), 7.62 (d, *J* = 15.4 Hz, 1H), 7.35 (dd, *J* = 12.5, 8.1 Hz, 4H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.79 (d, *J* = 15.3 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 144.3, 142.0, 141.7, 138.0, 129.9, 129.8, 128.5, 127.7, 126.4, 21.6, 21.5.

### (*E*)-1-methyl-4-((4-methylstyryl)sulfonyl)benzene (4ad)<sup>[11]</sup>



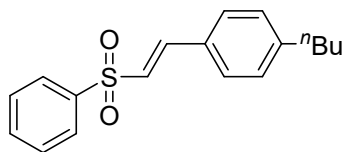
17.2mg, 60% yield; colorless oil, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86 – 7.79 (m, 2H), 7.62 (d, *J* = 15.4 Hz, 1H), 7.35 (dd, *J* = 12.5, 8.1 Hz, 4H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.79 (d, *J* = 15.3 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 144.3, 142.0, 141.7, 138.0, 129.9, 129.8, 128.5, 127.7, 126.4, 21.6, 21.5.

**(E)-1-isopropyl-4-(2-tosylvinyl)benzene (4ae)**<sup>[11]</sup>



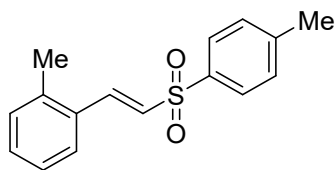
13.5 mg, 43% yield; colorless oil, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.84 – 7.79 (m, 2H), 7.64 (d, *J* = 15.4 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.36 – 7.31 (m, 2H), 7.26 – 7.22 (m, 2H), 6.79 (d, *J* = 15.4 Hz, 1H), 2.94 – 2.88 (m, 1H), 2.43 (s, 3H), 1.25 (s, 3H), 1.23 (s, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 152.6, 144.2, 142.1, 138.0, 130.1, 129.9, 128.7, 127.7, 127.2, 126.5, 34.1, 23.7, 21.6.

**(E)-1-butyl-4-(2-(phenylsulfonyl)vinyl)benzene35 (4af)**<sup>[12]</sup>



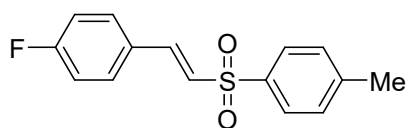
16.5 mg, 55% yield; colourless oil, <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 2H), 7.63 (d, *J* = 15.4 Hz, 1H), 7.41 – 7.31 (m, 4H), 7.21 – 7.17 (m, 2H), 6.79 (d, *J* = 15.4 Hz, 1H), 2.64 – 2.59 (m, 2H), 2.43 (s, 3H), 1.58 (d, *J* = 1.8 Hz, 2H), 1.37 – 1.30 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 146.7, 144.2, 142.1, 138.0, 129.9, 129.2, 128.6, 127.7, 126.5, 35.6, 33.3, 22.3, 21.6, 13.9.

**(E)-1-methyl-2-(2-tosylvinyl)benzene (4aj)**<sup>[13]</sup>



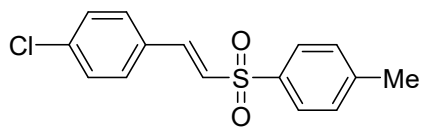
13.6 mg, 46% yield; colorless oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.94 (d,  $J = 15.3$  Hz, 1H), 7.83 (d,  $J = 8.0$  Hz, 2H), 7.43 (d,  $J = 7.8$  Hz, 1H), 7.35 (d,  $J = 8.0$  Hz, 2H), 7.30 – 7.25 (m, 1H), 7.19 (dd,  $J = 16.3, 8.0$  Hz, 2H), 6.77 (d,  $J = 15.3$  Hz, 1H), 2.45 (d,  $J = 5.3$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.4, 139.6, 138.2, 137.7, 131.4, 131.1, 130.9, 130.0, 128.5, 127.7, 126.9, 126.5, 21.7, 19.8.

**(*E*)-1-fluoro-4-(2-tosylvinyl)benzene (4ah)**<sup>[11]</sup>



12.4 mg, 40% yield; white soild,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86 – 7.80 (m, 2H), 7.62 (d,  $J = 15.4$  Hz, 1H), 7.52 – 7.43 (m, 2H), 7.38 – 7.32 (m, 2H), 7.12 – 7.05 (m, 2H), 6.78 (d,  $J = 15.4$  Hz, 1H), 2.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  164.32 ( $^1J_{\text{C-F}} = 254.5$  Hz), 144.5, 140.6, 137.7, 130.5 (d,  $^3J_{\text{C-F}} = 9.1$  Hz), 130.0, 127.7, 116.3 ( $^2J_{\text{C-F}} = 22.2$  Hz), 21.6.  $^{19}\text{F}$  NMR (376 MHz, Chloroform-*d*)  $\delta$  -107.94.

**(*E*)-1-chloro-4-(2-tosylvinyl)benzene (4ai)**<sup>[11]</sup>

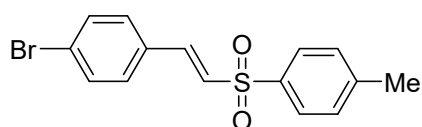




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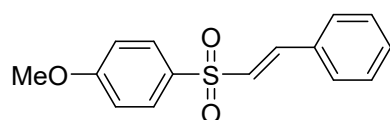
14.1 mg, 48% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.85 – 7.80 (m, 2H), 7.60 (d,  $J = 15.4$  Hz, 1H), 7.43 – 7.33 (m, 6H), 6.83 (d,  $J = 15.4$  Hz, 1H), 2.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.6, 140.4, 137.5, 137.2, 131.0, 130.0, 129.7, 129.4, 128.3, 127.8, 21.6.

**(*E*)-1-bromo-4-(2-tosylvinyl)benzene (4ag)**<sup>[11]</sup>



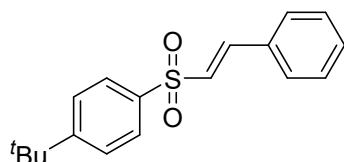
16.9 mg, 50% yield; white solid  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.82 (d,  $J = 8.3$  Hz, 2H), 7.59 (d,  $J = 15.4$  Hz, 1H), 7.52 (d,  $J = 8.5$  Hz, 2H), 7.38 – 7.30 (m, 4H), 6.84 (d,  $J = 15.5$  Hz, 1H), 2.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.6, 140.5, 137.5, 132.4, 131.4, 130.0, 129.9, 128.4, 127.8, 125.5, 21.6.

**(*E*)-1-methoxy-4-(styrylsulfonyl)benzene (4ba)**<sup>[11]</sup>



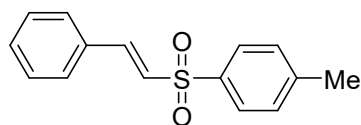
12.4 mg, 40% yield; white solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.00 (d,  $J = 9.0$  Hz, 2H), 7.53 – 7.44 (m, 3H), 7.40 – 7.30 (m, 3H), 7.05 (d,  $J = 9.0$  Hz, 2H), 3.90 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  163.6, 141.4, 131.0, 130.1, 129.9, 129.1, 128.5, 128.0, 114.6, 55.7.

**(*E*)-1-(tert-butyl)-4-(styrylsulfonyl)benzene (4ca)**<sup>[14]</sup>



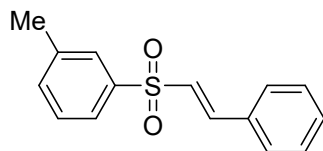
12.0 mg, 46% yield; colorless oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.90 – 7.84 (m, 2H), 7.67 (d,  $J = 15.4$  Hz, 1H), 7.58 – 7.46 (m, 5H), 7.40 (d,  $J = 1.0$  Hz, 2H), 6.86 (d,  $J = 15.4$  Hz, 1H), 1.34 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  157.4, 142.0, 137.7, 132.5, 131.1, 129.1, 128.5, 127.7, 127.6, 126.4, 35.3, 31.1.

**(*E*)-1-methyl-4-(styrylsulfonyl)benzene (4da)**<sup>[11]</sup>



14.2 mg, 53% yield; White solid,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.83 (d,  $J = 8.1$  Hz, 2H), 7.66 (d,  $J = 15.4$  Hz, 1H), 7.48 (dd,  $J = 7.7$ , 2.1 Hz, 2H), 7.37 (dt,  $J = 19.4$ , 6.7 Hz, 5H), 6.85 (d,  $J = 15.3$  Hz, 1H), 2.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  144.4, 142.0, 137.8, 132.5, 131.1, 130.0, 129.1, 128.5, 127.7, 127.7, 21.6.

**(*E*)-1-methyl-3-(styrylsulfonyl)benzene (4ea)**<sup>[11]</sup>



11.1 mg, 50% yield; colorless oil,  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.78 – 7.65 (m, 3H), 7.52 – 7.47 (m, 2H), 7.44 – 7.38 (m, 5H), 6.86 (d,  $J = 15.4$  Hz, 1H), 2.44 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$

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127.5, 124.8, 21.3.

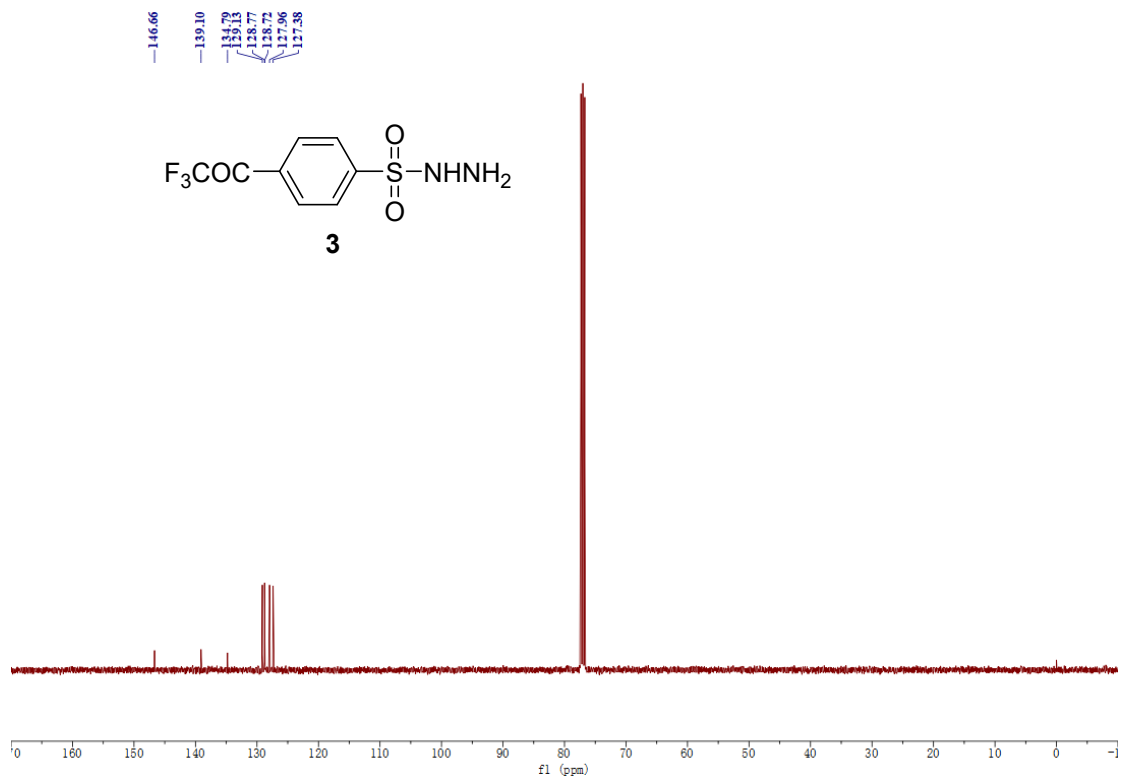
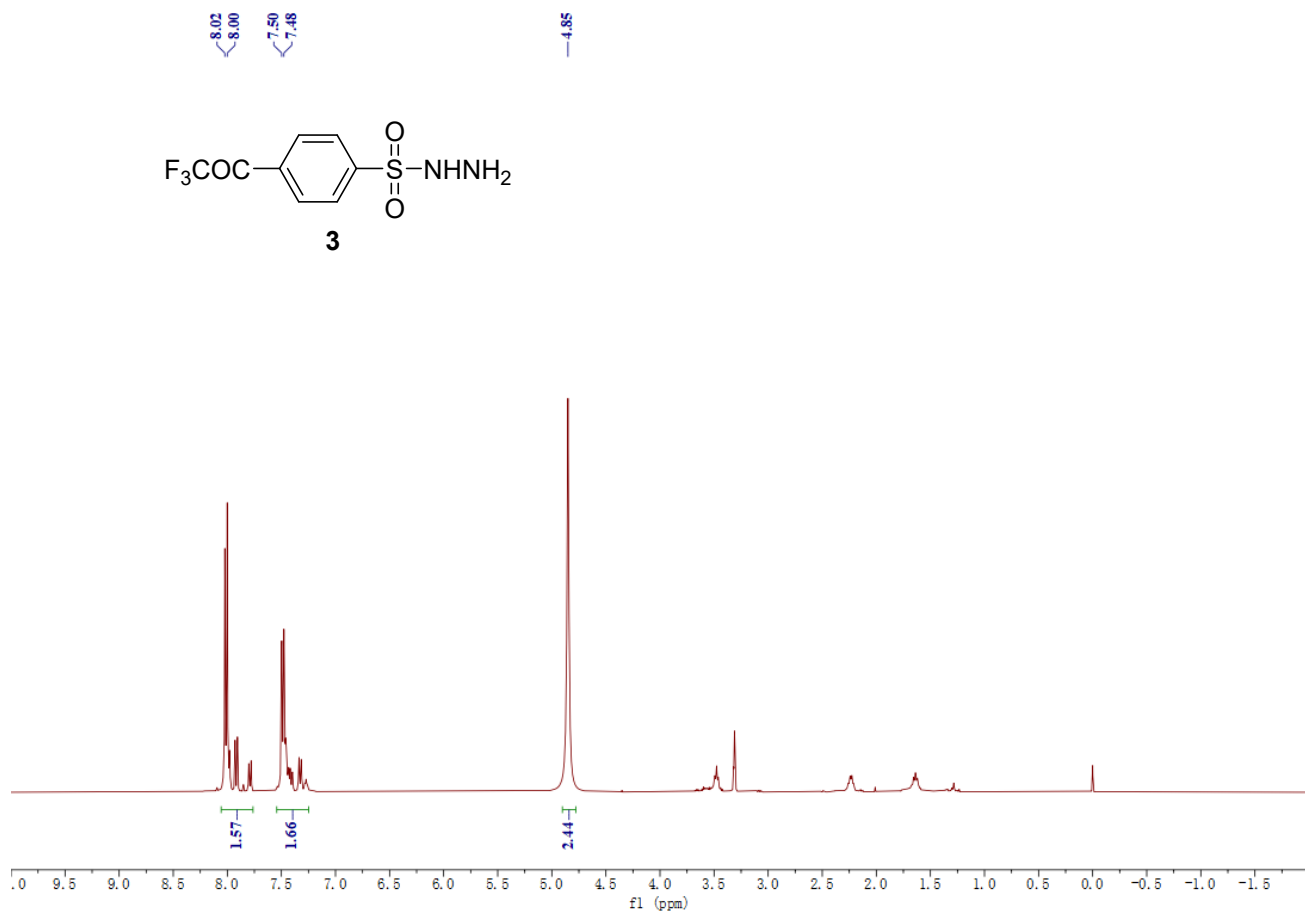
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## Reference

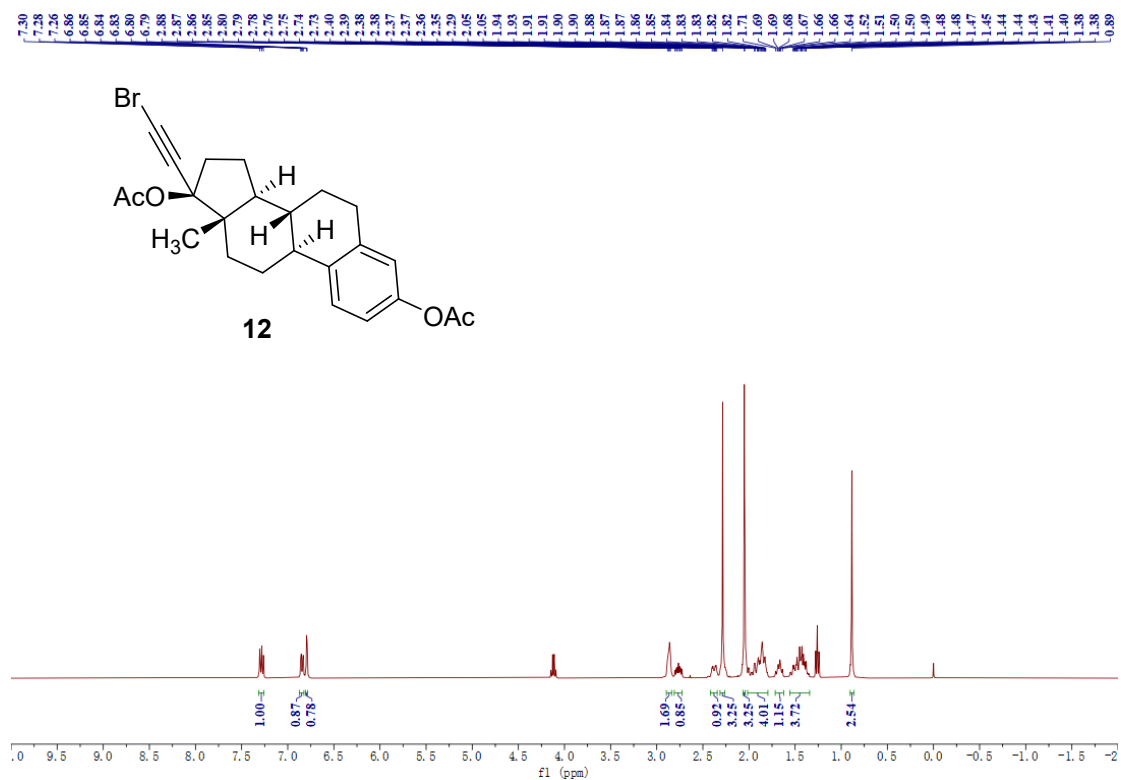
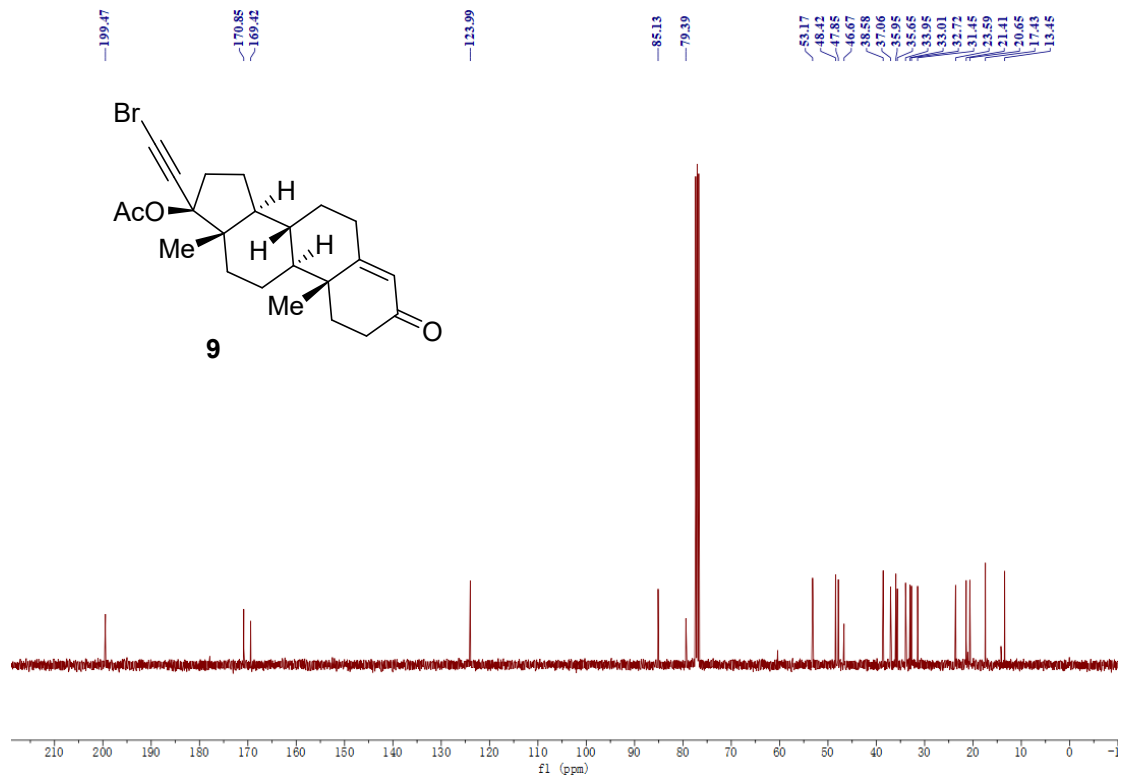
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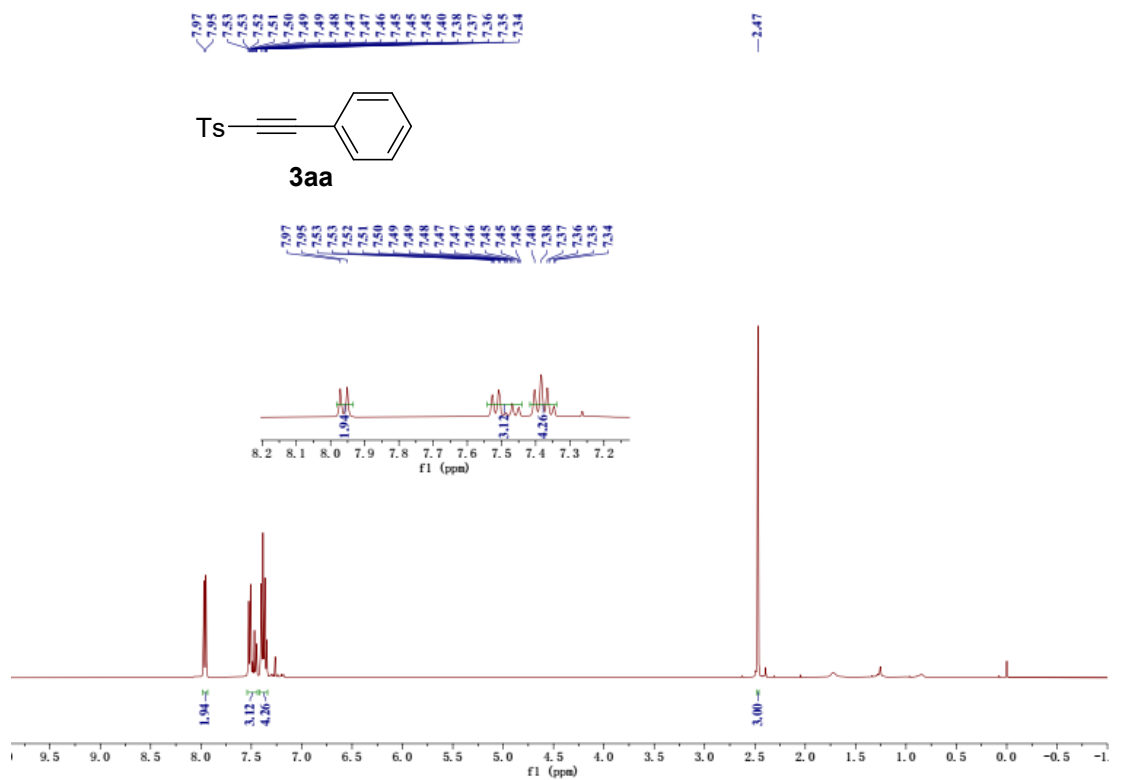
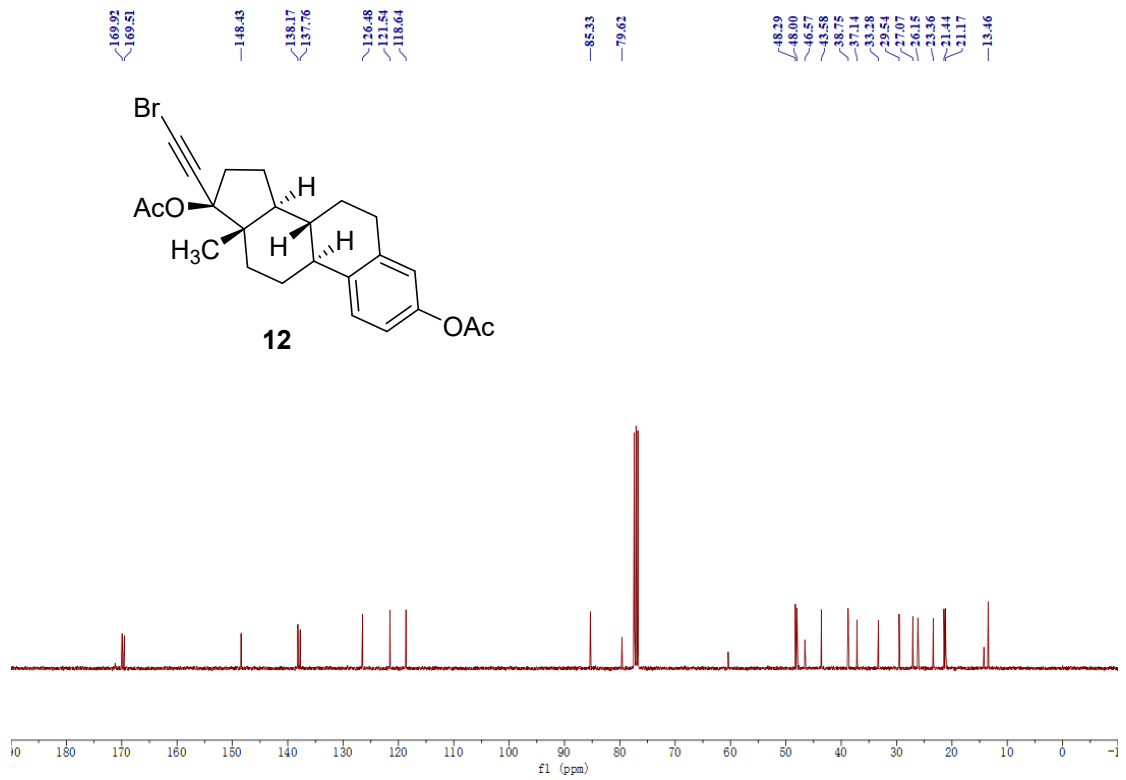
# $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra

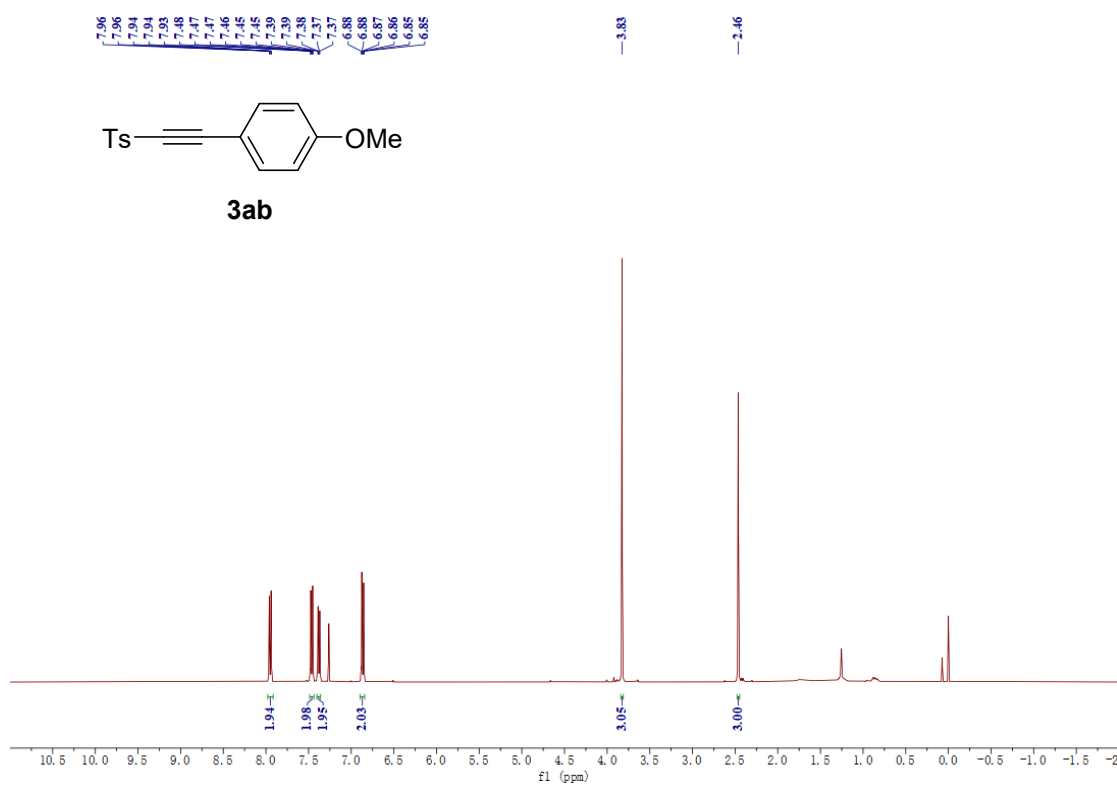
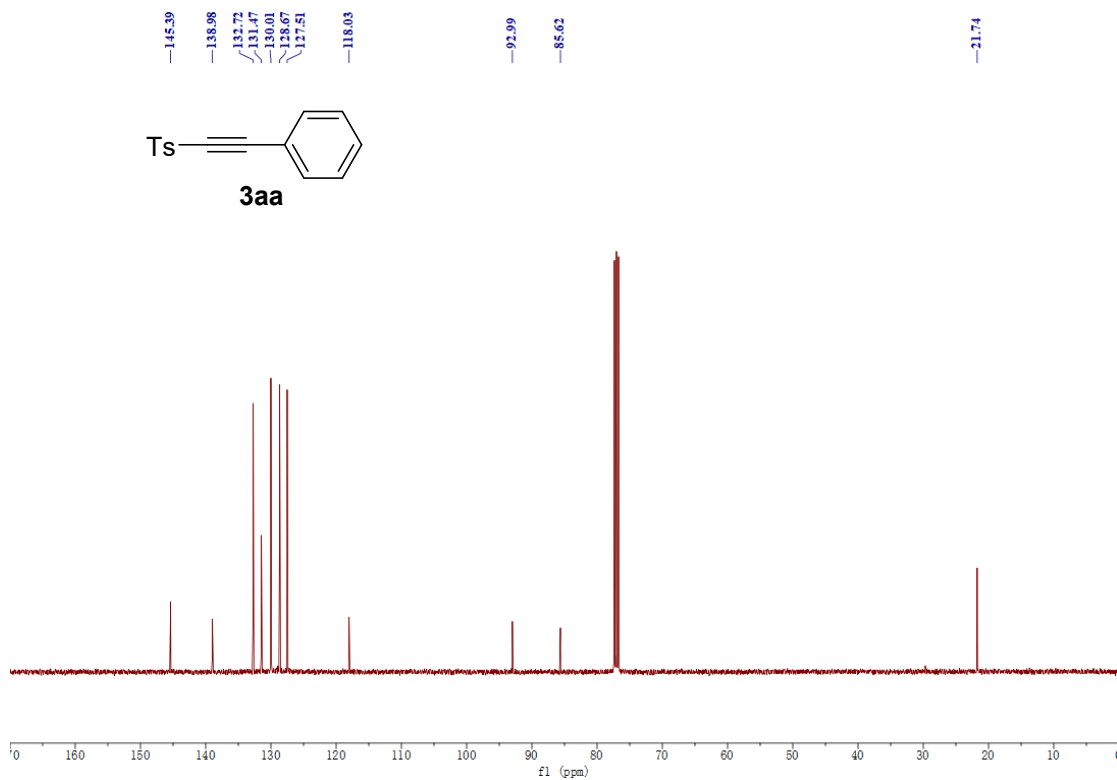


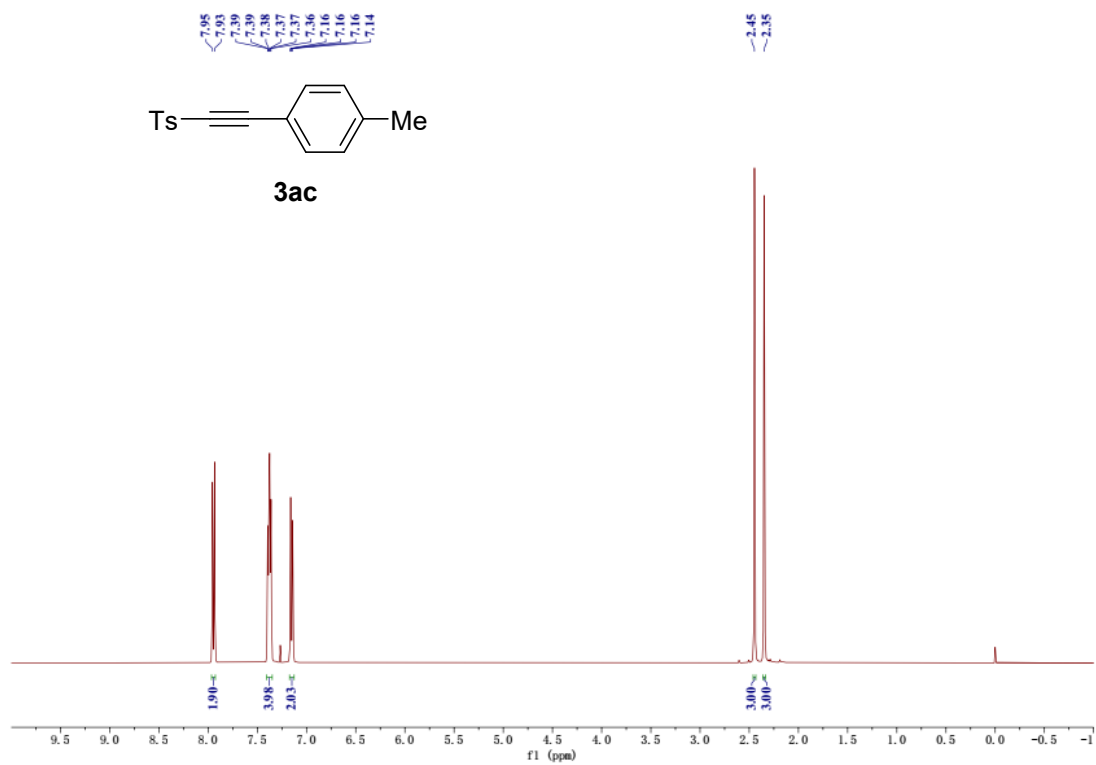
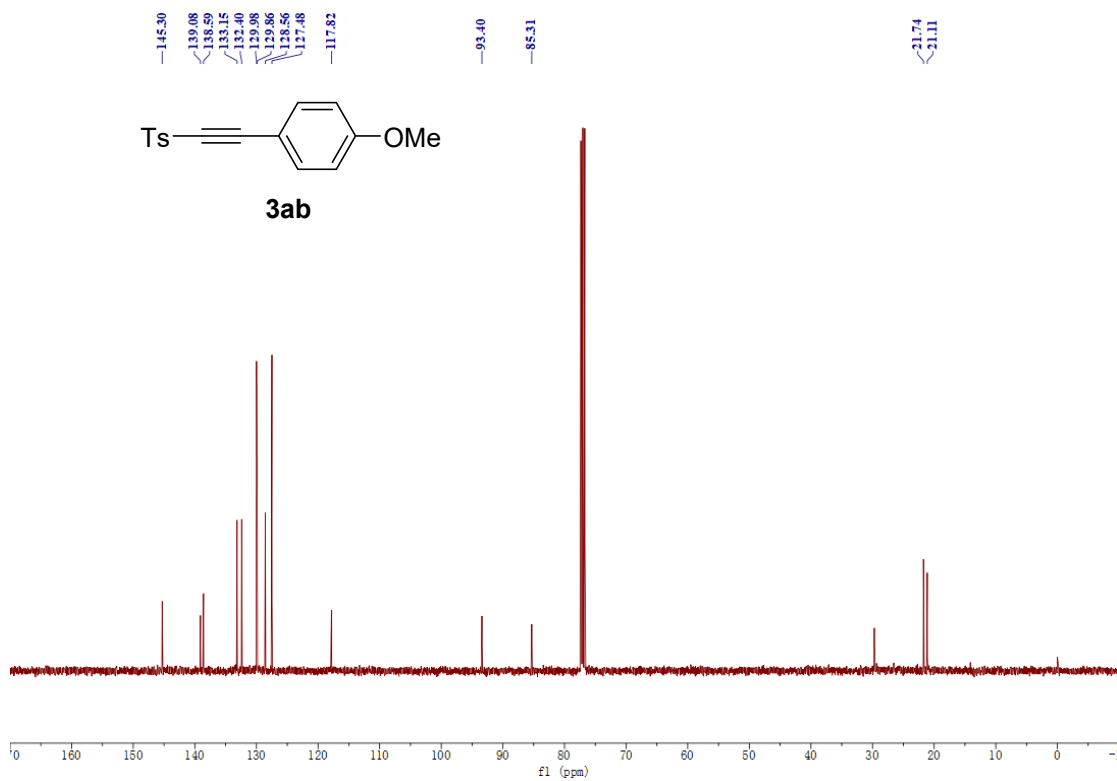


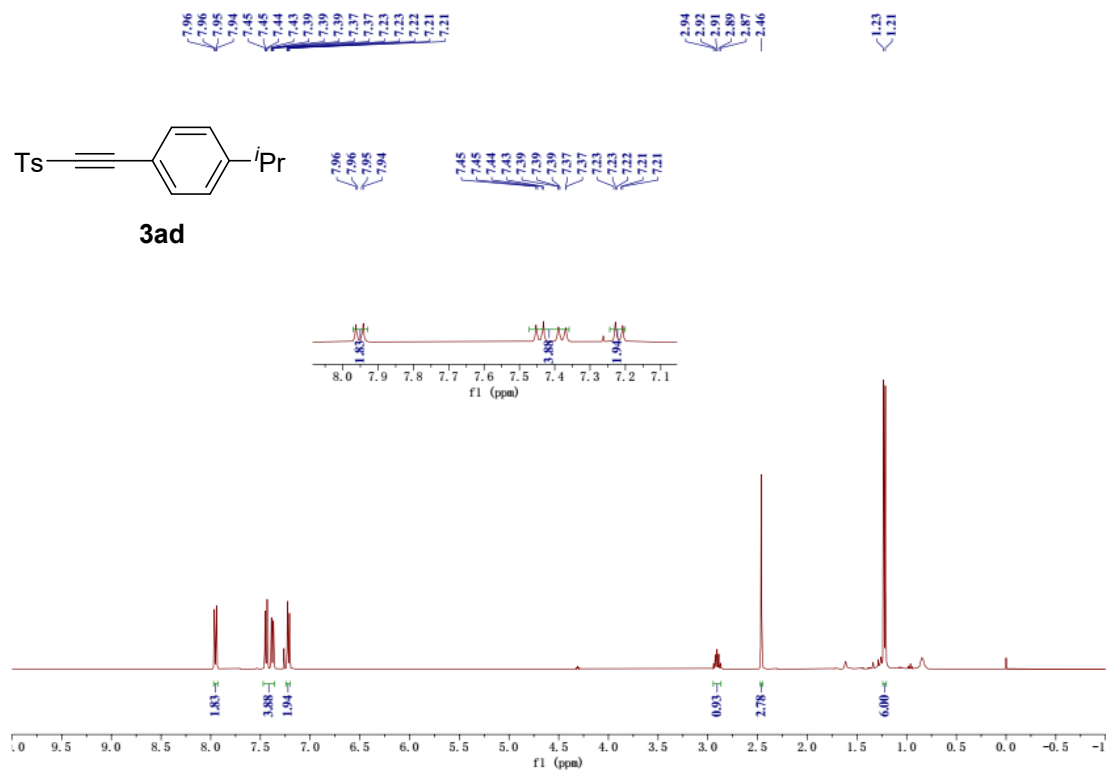
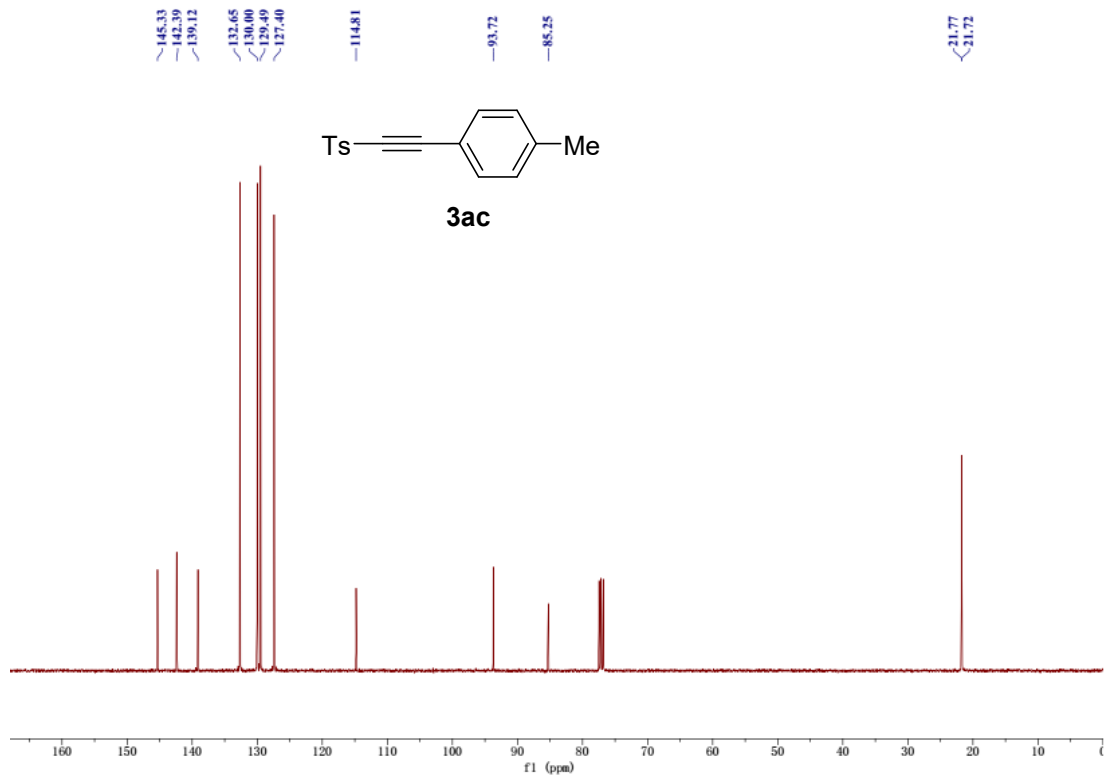


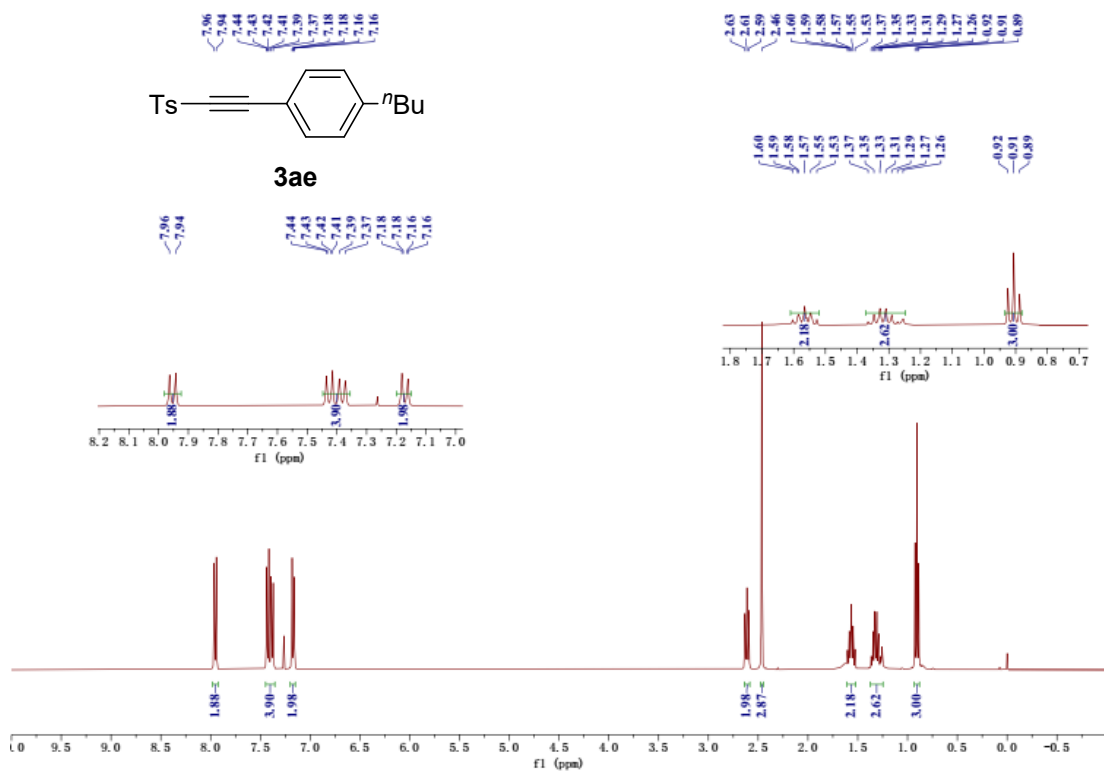
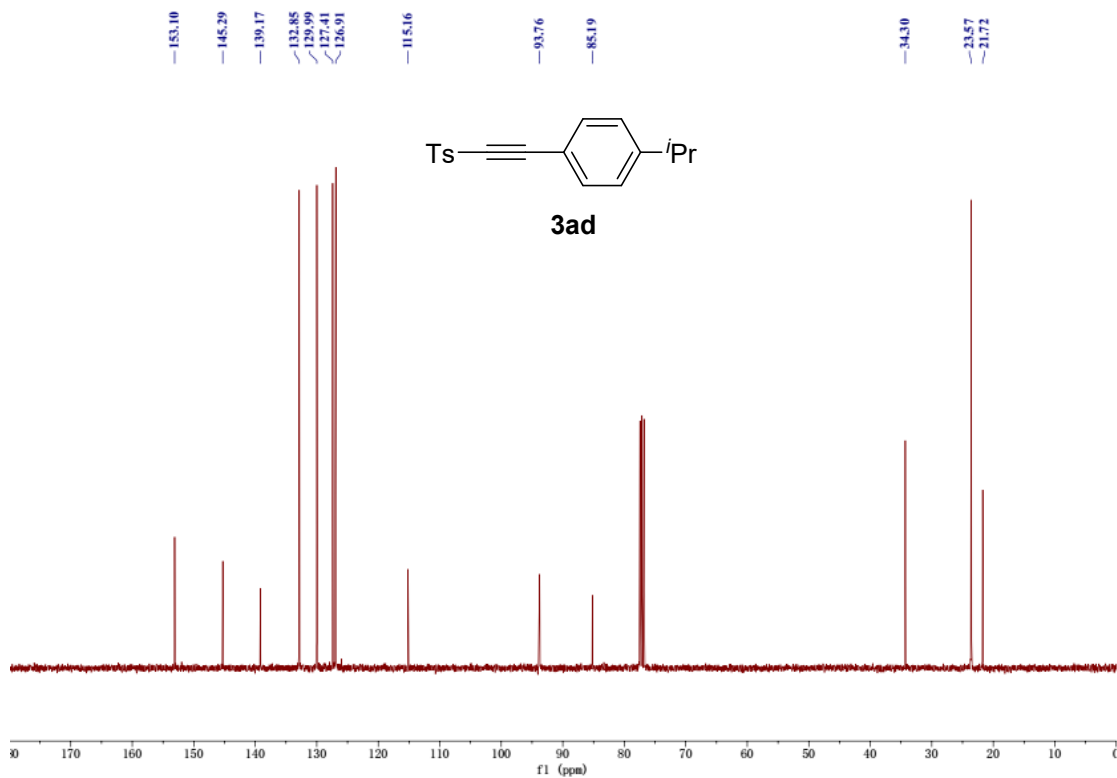


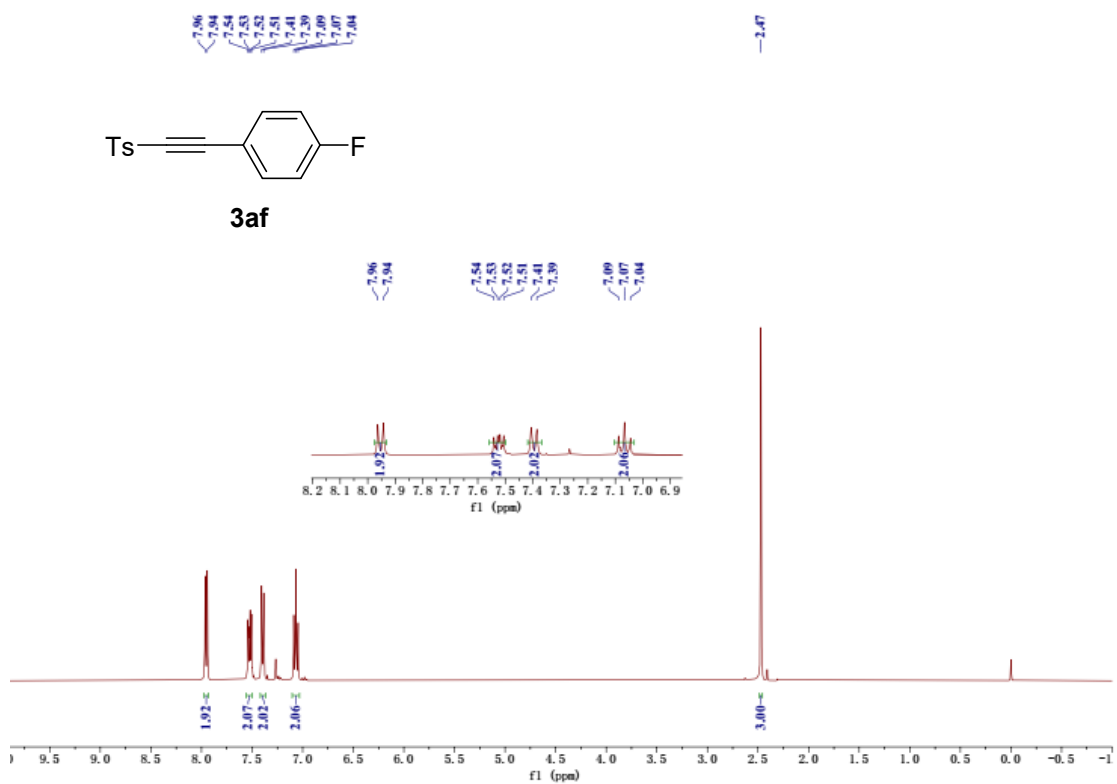
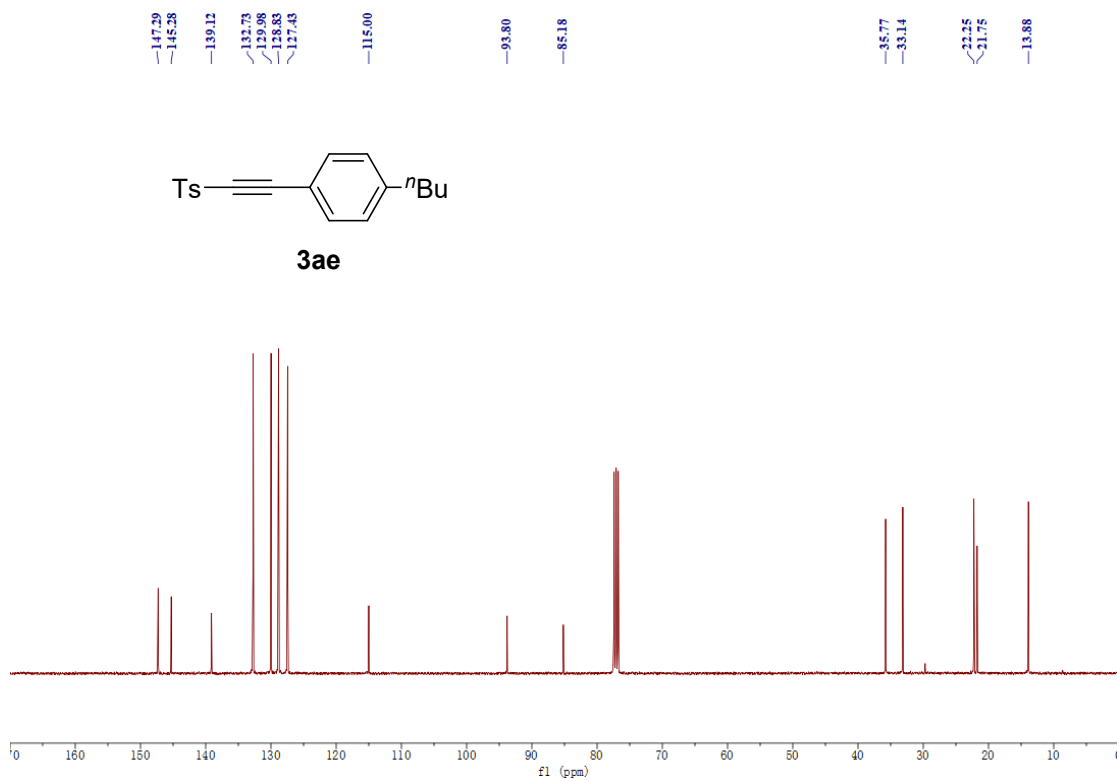


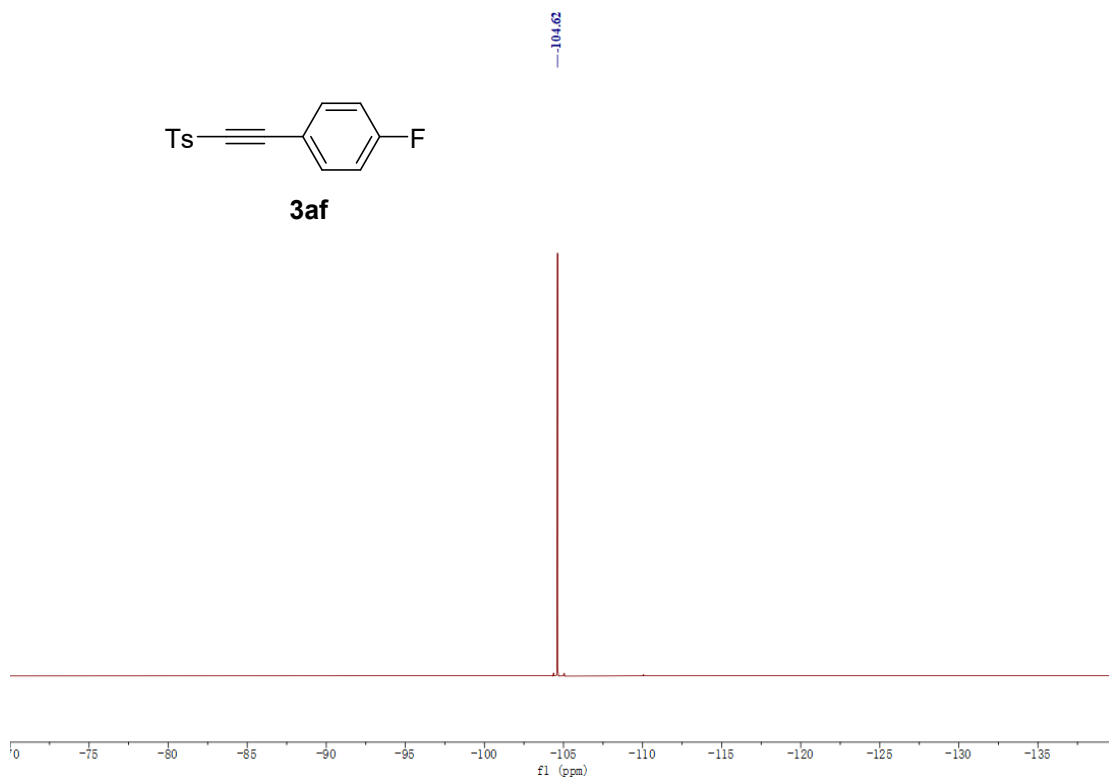
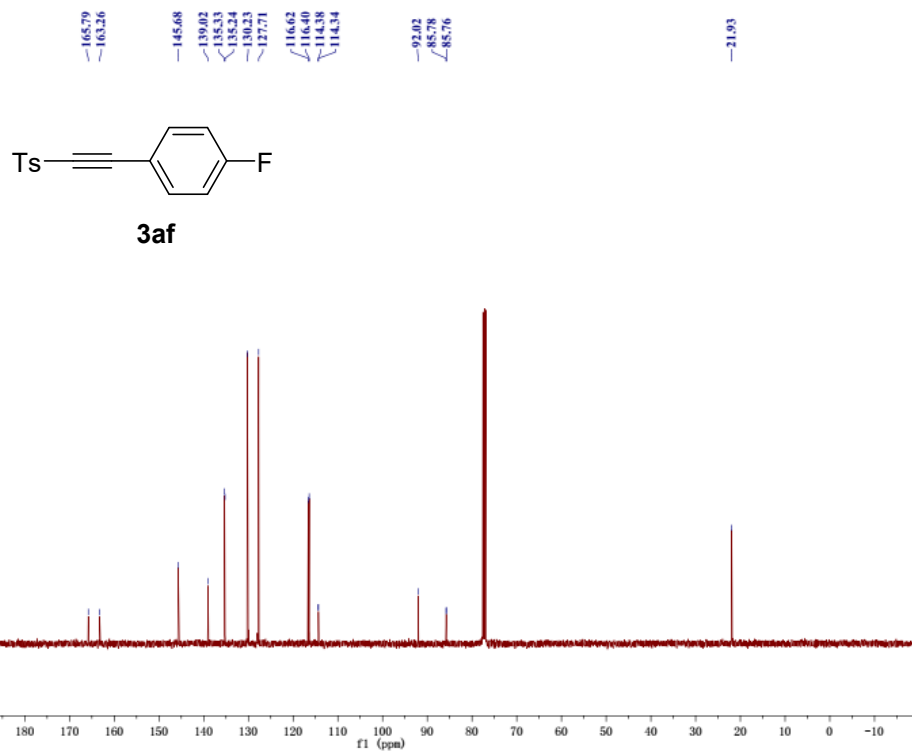






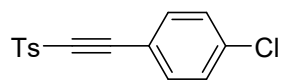




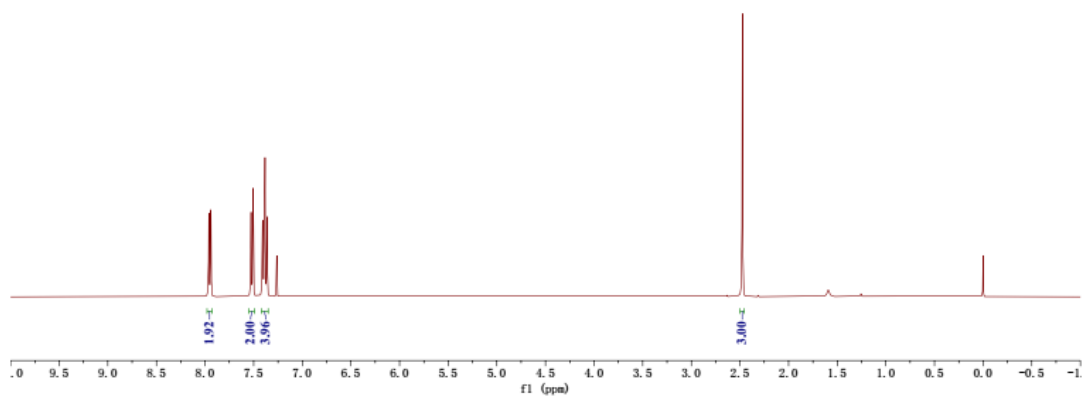


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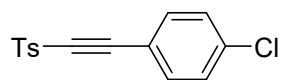
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