# Palladium(II)-catalyzed tandem cyclization of 2-ethynylaniline tethered cinnamyl acetate for the synthesis of indenoindoles

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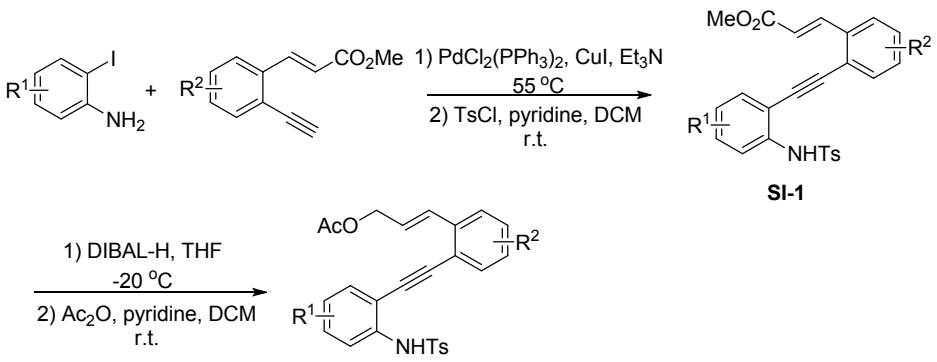
## Contents

**1.** General information

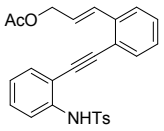
All solvents were dried and distilled using standard procedures. Unless otherwise noted, reagents were obtained from commercial sources and used without further purification. $^{1}H$ NMR and $^{13}C$ NMR were recorded in deuterated chloroform ($CDCl_3$). Coupling constants are recorded in hertz, and chemical shifts are recorded as δ values in ppm. The following abbreviations are used to describe multiplicities: s = singlet, d = doublet, dd = double doublet, t = triplet, m = multiplet. High-resolution mass spectra were carried out on a mass spectrometer with a TOF analyzer (ESI). Infrared spectra were recorded on a FT-IR spectrometer. Melting points were determined by using a local hot-stage melting point apparatus and are uncorrected. For column chromatography, silica gel of 200-300 mesh size was used.

**2.** General procedure for the synthesis of substrates **1**




In a 50 mL single neck flask，the corresponding 2-iodoaniline (5.0 mmol, 1.0 equiv.) and substituted *o*-alkynyl Methyl cinnamate (5.0 mmol, 1.0 equiv) were dissolved in $Et_3N$ (25 mL). Then $PdCl_2(PPh_3)_2$ (0.05 mmol, 0.01 equiv), CuI (0.05 mmol, 0.01 equiv) were added to the mixture and the resulting solution was stirred at 55 °C. After completion of the reaction, the mixture was filtered by a short silica column, the solvent was evaporated under reduce pressure. The residue was dissolved in DCM (20 mL), then pyridine (7.5 mmol, 1.5 equiv) and TsCl (6.0 mmol, 1.2 equiv) were added sequentially. The reaction was stirred at room temperature overnight. After completion of the reaction, the mixture was filtered by a short silica column, the solvent was evaporated under reduced pressure to obtain the crude product **SI-1**.

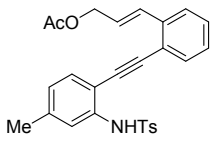
The crude product **SI-1** was dissolved in anhydrous THF (20 mL), the mixture was cooled to -20 $^{o}$C, then DIBAL-H (11.25 mL, 1 M in hexane) was added to the reaction mixture, followed by stirring for additional half an hour at the same temperature. After completion of the reaction, the mixture was quenched by water (20 mL). The mixture was filtered by celite, washed by ethyl acetate to remove the aluminium salt. The organic layer was dried over anhydrous $Na_2SO_4$, and then evaporated under reduced pressure. The residue was dissolved in DCM (10 mL), then pyridine (3.75 mmol, 1.5 equiv) and $Ac_2O$ (3 mmol, 1.2 equiv) was added sequentially. The reaction was stirred at 0 $^{o}$C. After completion of the reaction, the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography (petroleum ether : ethyl acetate = 5:1) to obtain the compound **1**.




**1a**

**(*E*)-3-(2-((2-(4-Methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1a)**

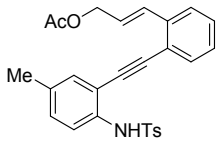
Yellow oil; (1.04 g; 46% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.67-7.57 (m, 4H), 7.45-7.25 (m, 6H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.09-7.05 (m, 2H), 6.39 (dt, *J* = 16.0 Hz, *J* = 6.0 Hz, 1H), 4.81 (dd, *J* = 6.0 Hz, *J* = 0.8 Hz, 2H), 2.31 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 144.1, 137.9, 137.6, 136.2, 132.6, 132.1, 131.4, 129.9, 129.7, 129.3, 127.9, 127.3, 126.3, 125.6, 124.7, 121.0, 120.4, 114.7, 94.4, 88.6, 65.0, 21.6, 21.0; **IR (neat, cm$^{-1}$):** 3250, 2339, 1730, 1336, 1226, 1157, 1089, 753, 660; **HRMS** calculated for $C_{26}H_{23}NO_4SNa$ $(M+Na)^+$: 468.1240; Found: 468.1242.




**1b**

**(*E*)-3-(2-((4-Methyl-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1b)**

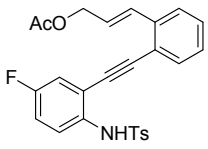
Yellow oil; (1.01 g; 44% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.65 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.44-7.41 (m, 2H), 7.37-7.26 (m, 3H), 7.18-7.13 (m, 3H), 7.06 (d, *J* = 16.0 Hz, 1H), 6.89 (dd, *J* = 8.0 Hz, *J* = 0.8 Hz, 1H), 6.39 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.35, (s, 3H), 2.32 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.6, 143.7, 140.2, 137.4, 137.1, 135.9, 132.2, 131.5, 131.2, 129.3, 128.8, 127.5, 127.0, 125.8, 125.4, 125.2, 120.9, 120.8, 111.5, 93.4, 88.5, 64.7, 21.5, 21.2, 20.7; **IR (neat, cm$^{-1}$):** 3270, 2922, 1732, 1504, 1335, 1226, 1158, 1089, 961, 812, 660; **HRMS** calculated for $C_{27}H_{25}NO_4SNa$ $(M+Na)^+$: 482.1396; Found: 482.1381.




**1c**

**(*E*)-3-(2-((5-Methyl-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1c)**

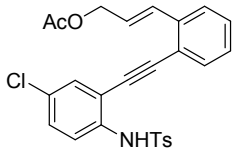
Yellow oil; (896 mg; 39% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.56-7.49 (m, 2H), 7.42 (dd, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 7.35-7.24 (m, 2H), 7.20-7.16 (m, 2H), 7.10-7.03 (m, 4H), 6.38 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.28, (s, 3H), 2.26 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 143.8, 137.7, 136.2, 134.9, 134.7, 132.5, 132.4, 131.3, 130.6, 129.5, 129.2, 127.8, 127.2, 126.0, 125.5, 121.3, 121.0, 115.1, 93.7, 88.9, 64.9, 21.4, 20.9, 20.6; **IR (neat, cm$^{-1}$):** 3168, 1709, 1493, 1337, 1256, 1158, 1091, 811, 753, 665; **HRMS** calculated for $C_{27}H_{25}NO_4SNa$ $(M+Na)^+$: 482.1396; Found: 482.1390.




**1d**

**(*E*)-3-(2-((5-Fluoro-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1d)**

Yellow solid; m.p.: 69-70 $^{o}$C; (973 mg; 42% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.61-7.55 (m, 4H), 7.42-7.35 (m, 2H), 7.30-7.26 (m, 1H), 7.14-7.00 (m, 6H), 6.37 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.0 Hz, *J* = 1.2 Hz, 2H), 2.30, (s, 3H), 2.09 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 159.6 (d, *J* = 244.4 Hz), 144.1, 138.0, 135.9, 133.6 (d, *J* = 3.1 Hz), 132.7, 131.3, 129.7, 129.6, 127.9, 127.3, 126.4, 125.6, 123.9, (d, *J* = 8.4 Hz), 120.5, 118.4 (d, *J* = 23.7 Hz), 117.5 (d, *J* = 9.2 Hz), 117.0 (d, *J* = 22.2 Hz), 94.8, 87.7 (d, *J* = 3.1 Hz), 64.9, 21.5, 21.0; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -116.5; **IR (neat, cm$^{-1}$):** 3213, 1724, 1493, 1337, 1159, 903, 744, 664; **HRMS** calculated for $C_{26}H_{22}NO_4FSNa$ $(M+Na)^+$: 486.1145; Found: 486.1142.

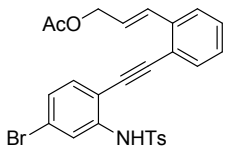



**1e**

**(*E*)-3-(2-((5-Chloro-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate**

Yellow solid; m.p.: 72-73 $^{o}$C; (959 mg; 40% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.59-7.54 (m, 2H), 7.44-7.36 (m, 3H), 7.31-7.24 (m, 2H), 7.21 (s, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 16.0 Hz, 1H ), 6.38 (dt, *J* = 16.0 Hz, *J* = 6.0 Hz, 1H), 4.81 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.32, (s, 3H), 2.10 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 144.3, 138.1, 136.2, 135.9, 132.7, 131.6, 131.2, 130.1, 129.9, 129.8, 129.7, 127.9, 127.3, 126.6, 125.7, 122.0, 120.5, 116.5, 95.4, 87.3, 64.9, 21.6, 21.0; **IR (neat, cm$^{-1}$):**

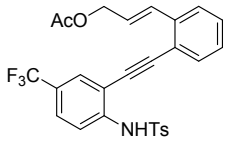
3208, 2924, 1723, 1487, 1338, 1238, 1164, 1089, 744, 664; **HRMS** calculated for $C_{26}H_{22}NO_4ClSNa$ $(M+Na)^+$: 502.0850; Found: 502.0844.




**1f**

**(*E*)-3-(2-((4-Bromo-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1f)**

Yellow solid; m.p.: 81-82 $^oC$; (1.02 g; 39% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.79 (m, 1H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.45-7.35 (m, 2H), 7.30-7.17 (m, 6H), 7.04 (d, *J* = 16.0 Hz, 1H ), 6.39 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.33 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.4, 144.0, 138.1, 137.5, 135.4, 132.6, 132.2, 130.9, 129.4, 129.1, 127.5, 127.3, 127.0, 126.0, 125.2, 123.2, 122.6, 120.2, 112.9, 95.0, 87.2, 64.5, 21.1, 20.6; **IR (neat, $cm^{-1}$):** 3193, 2925, 1728, 1488, 1329, 1234, 1163, 1090, 928, 759, 759, 665; **HRMS** calculated for $C_{26}H_{22}NO_4BrSNa$ $(M+Na)^+$: 546.0345; Found: 546.0350.

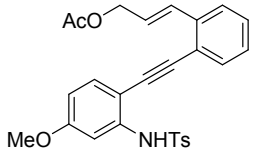



**1g**

**(*E*)-3-(2-((2-(4-Methylphenylsulfonamido)-5-(trifluoromethyl)phenyl)ethynyl)phenyl)allyl acetate (1g)**

White solid; m.p.: 101-102 $^oC$; (1.03 g; 40% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.74-7.67 (m, 4H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.52-7.48 (m, 3H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.33-7.21 (m, 3H), 7.08 (d, *J* = 15.6 Hz, 1H ), 6.41 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.83 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.36 (s, 3H), 2.09 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 144.7, 140.5, 138.2, 135.9, 132.9, 131.1, 130.0, 129.9, 129.3 (q, *J* = 3.8 Hz), 128.0, 127.4, 126.8, 126.6 (q, *J* = 3.8 Hz), 126.4 (q, *J* = 33.0 Hz), 125.7, 123.6 (q, *J* = 270.4 Hz), 120.3,

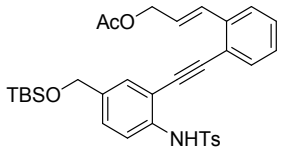
118.8, 114.1, 96.0, 87.0, 64.9, 21.6, 21.0; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -62.4; **IR (neat, cm$^{-1}$):** 3301, 2922, 1727, 1503, 1335, 1247, 1109, 892, 667; **HRMS** calculated for $C_{27}H_{22}NO_4F_3SNa$ $(M+Na)^+$: 536.1114; Found: 536.1117.




**1h**

**(*E*)-3-(2-((4-Methoxy-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1h)**

Yellow oil; (1.07 g; 45% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.43 (dd, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 7.36-7.26 (m, 4H), 7.19-7.05 (m, 3H), 7.08 (d, *J* = 16.0 Hz, 1H ), 6.62 (dd, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H), 6.39 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 3.81 (s, 3H), 2.33 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 171.0, 160.9, 144.2, 139.1, 137.7, 136.2, 133.1, 132.5, 131.6, 129.8, 129.0, 128.0, 127.4, 126.1, 125.6, 121.4, 111.2, 106.5, 105.4, 93.3, 88.8, 65.1, 55.7, 21.6, 21.1; **IR (neat, cm$^{-1}$):** 3206, 2926, 1729, 1506, 1334, 1287, 1196, 1158, 960, 891, 757, 664; **HRMS** calculated for $C_{27}H_{25}NO_5SNa$ $(M+Na)^+$: 498.1345; Found: 498.1335.

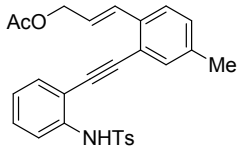



**1i**

**(*E*)-3-(2-((5-(((tert-Butyldimethylsilyl)oxy)methyl)-2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1i)**

Yellow oil; (1.26 g; 43% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.29-7.25 (m, 2H), 7.21-7.16 (m, 2H), 7.11 (s, 1H), 7.05-

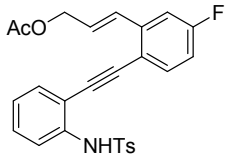
6.96 (m, 3H), 6.30 (td, $J$ = 16.0 Hz, $J$ = 6.4 Hz, 1H), 4.72 (d, $J$ = 6.0 Hz, 2H), 4.57 (s, 2H), 2.22 (s, 3H), 1.99 (s, 3H), 0.84 (s, 9H), 0.00 (s, 6H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 144.0, 138.2, 137.9, 136.3, 136.2, 132.7, 131.6, 129.7, 129.6, 129.3, 127.9, 127.7, 127.4, 126.2, 125.6, 121.1, 120.7, 114.8, 94.0, 88.8, 65.0, 64.1, 26.0, 21.6, 21.0, 18.5, -5.1; **IR (neat, cm$^{-1}$):** 3323, 2938, 2856, 2255, 1733, 1233, 1159, 1085, 838, 727, 663; **HRMS** calculated for $C_{33}H_{39}NO_5SSiNa$ $(M+Na)^+$: 612.2210; Found: 612.2215.




**1j**

**(*E*)-3-(4-Methyl-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1j)**

Yellow solid; m.p.: 85-86 °C; (965 mg; 42% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.66 (d, $J$ = 8.4 Hz, 2H), 7.60 (d, $J$ = 8.0 Hz, 1H), 7.47 (d, $J$ = 8.0 Hz, 1H), 7.39 (dd, $J$ = 8.0 Hz, $J$ = 1.2 Hz, 1H), 7.31-7.25 (m, 3H), 7.17-7.01 (m, 5H), 6.35 (dt, $J$ = 15.6 Hz, $J$ = 6.4 Hz, 1H), 4.79 (dd, $J$ = 6.4 Hz, $J$ = 1.2 Hz, 2H), 2.36 (s, 3H), 2.31 (s, 3H), 2.07 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 144.0, 137.9, 137.5, 136.2, 135.1, 132.9, 132.1, 131.4, 130.4, 129.8, 129.7, 127.3, 125.5, 125.1, 124.6, 120.8, 120.3, 114.7, 94.6, 88.1, 65.1, 21.5, 21.0; **IR (neat, cm$^{-1}$):** 3213, 1724, 1493, 1336, 1272, 1163, 813, 750, 665; **HRMS** calculated for $C_{27}H_{25}NO_4SNa$ $(M+Na)^+$: 482.1396; Found: 482.1393.

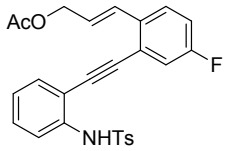



**1k**

**(*E*)-3-(5-Fluoro-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1k)**

Yellow solid; m.p.: 90-91 °C; (950 mg; 41% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.66 (d, $J$ = 8.0 Hz, 2H), 7.59 (d, $J$

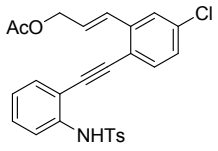
= 8.0 Hz, 1H), 7.45-7.38 (m, 2H), 7.32-7.22 (m, 3H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.10-6.97 (m, 3H), 6.38 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.0 Hz, 2H), 2.33 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.6, 162.8 (d, *J* = 249.7 Hz), 143.9, 140.1 (d, *J* = 7.6 Hz), 137.3, 136.0, 134.3 (d, *J* = 8.5 Hz), 131.9, 130.1 (d, *J* = 2.3 Hz), 129.7, 129.4, 127.3, 127.1, 124.4, 120.1, 116.9 (d, *J* = 3.0 Hz), 115.2 (d, *J* = 23.0 Hz), 114.2, 112.1 (d, *J* = 23.0 Hz), 93.1, 88.0, 64.4, 21.3, 20.7; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -109.2; **IR (neat, cm$^{-1}$):** 3327, 2922, 1734, 1494, 1337, 1224, 1158, 1089, 908, 755, 661; **HRMS** calculated for $C_{26}H_{22}NO_4FSNa$ $(M+Na)^+$: 486.1145; Found: 486.1139.




**1l**

**(*E*)-3-(4-Fluoro-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1l)**

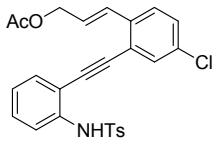
Yellow solid; m.p.: 86-87 $^oC$; (1.02 g; 44% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.55-7.52 (m, 1H), 7.41 (dd, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 7.34-7.30 (m, 1H), 7.21 (s, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.12-6.99 (m, 4H), 6.32 (dt, *J* = 16.4 Hz, *J* = 6.4 Hz, 1H), 4.80 (dd, *J* = 6.0 Hz, 2H), 2.33 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.9, 161.8 (d, *J* = 247.4 Hz), 144.2, 137.7, 136.3, 134.3 (d, *J* = 3.9 Hz), 132.3, 130.5, 130.2, 129.7, 127.4 (d, *J* = 8.4 Hz), 127.3, 126.1 (d, *J* = 1.5 Hz), 124.8, 122.6 (d, *J* = 10.0 Hz), 120.9, 118.8 (d, *J* = 22.9 Hz), 116.9 (d, *J* = 21.4 Hz), 114.4, 92.9 (d, *J* = 3.0 Hz), 89.5, 64.9, 21.6, 21.0; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -113.5; **IR (neat, cm$^{-1}$):** 3238, 1721, 1493, 1336, 1222, 1159, 1090, 908, 813, 751, 664; **HRMS** calculated for $C_{26}H_{22}NO_4FSNa$ $(M+Na)^+$: 486.1145; Found: 486.1136.




**1m**

**(*E*)-3-(5-Chloro-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1m)**

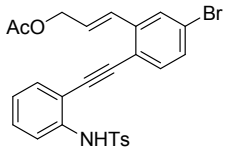
Yellow solid; m.p.: 91-92 $^{o}$C; (887 mg; 37% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.59-7.54 (m, 2H), 7.40-7.23 (m, 5H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.08 (td, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 7.40 (d, *J* = 16.0 Hz, 1H), 6.40 (dt, *J* = 16.4 Hz, *J* = 6.4 Hz, 1H), 4.81 (d, *J* = 6.0 Hz, 2H), 2.32 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 144.1, 139.5, 137.6, 136.2, 135.4, 133.7, 132.2, 130.1, 129.7, 128.0, 127.7, 127.3, 125.7, 124.7, 120.5, 119.4, 114.5, 93.2, 89.5, 64.6, 21.6, 21.0; **IR (neat, cm$^{-1}$):** 3227, 1724, 1492, 1336, 1239, 1160, 1085, 906, 813, 730, 663; **HRMS** calculated for $C_{26}H_{22}NO_4ClSNa$ $(M+Na)^+$: 502.0850; Found: 502.0846.




**1n**

**(*E*)-3-(4-Chloro-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1n)**

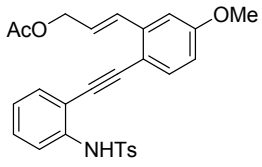
Yellow solid; m.p.: 87-88 $^{o}$C; (839 mg; 35% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.65 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.48-7.29 (m, 4H), 7.21 (s, 1H), 7.17-7.08 (m, 3H), 7.00 (d, *J* = 16.0 Hz, 1H), 6.36 (dt, *J* = 16.0 Hz, *J* = 6.0 Hz, 1H), 4.80 (dd, *J* = 6.0 Hz, *J* = 1.2 Hz, 2H), 2.33 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 144.1, 137.6, 136.4, 136.3, 133.4, 132.3, 132.1, 130.3, 130.2, 129.7, 129.5, 127.3, 126.8, 124.9, 122.4, 121.1, 114.5, 92.6, 89.7, 64.8, 21.5, 21.0; **IR (neat, cm$^{-1}$):** 3214, 1724, 1491, 1338, 1239, 1161, 919, 751, 664; **HRMS** calculated for $C_{26}H_{22}NO_4ClSNa$ $(M+Na)^+$: 502.0850; Found: 502.0846.




**1o**

**(*E*)-3-(5-Bromo-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1o)**

Yellow solid; m.p.: 93-94 $^{o}$C; (944 mg; 36% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.71 (d, *J* = 2.0 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.41-7.39 (m, 2H), 7.33-7.26 (m, 2H), 7.21 (s, 1H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 16.0 Hz, 1H), 6.39 (dt, *J* = 16.0 Hz, *J* = 6.0 Hz, 1H), 4.80 (dd, *J* = 6.0 Hz, *J* = 1.2 Hz, 2H), 2.32 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 144.2, 139.7, 137.6, 136.2, 133.8, 132.2, 130.9, 130.1, 130.0, 129.7, 128.7, 127.8, 127.3, 124.8, 123.7, 120.6, 119.9, 114.4, 93.3, 89.7, 64.7, 21.6, 21.0; **IR (neat, cm$^{-1}$):** 3254, 2924, 1733, 1225, 1159, 1089, 910, 812, 661; **HRMS** calculated for $C_{26}H_{22}NO_4BrSNa$ $(M+Na)^+$: 546.0345; Found: 546.0349.

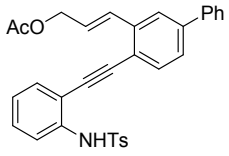



**1p**

**(*E*)-3-(5-Methoxy-2-((2-(4-methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (1p)**

Yellow oil; (974 mg; 41% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.39-7.36 (m, 2H), 7.28-7.24 (m, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.07-7.02 (m, 3H), 6.84 (dd, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 6.38 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.81 (dd, *J* = 6.0 Hz, *J* = 1.2 Hz, 2H), 3.85 (s, 3H), 2.31 (s, 3H), 2.08 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 160.3, 144.0, 139.4, 137.3, 136.1, 134.0, 131.9, 131.3, 129.6, 129.4, 127.3, 126.3, 124.6, 120.2, 115.0, 114.3, 113.4, 110.4, 94.5, 87.2, 64.8, 55.5,

21.5, 20.9; **IR (neat, cm$^{-1}$):** 3318, 2940, 2205, 1732, 1598, 1496, 1226, 1159, 1088, 908, 811, 755, 661; **HRMS** calculated for $C_{27}H_{25}NO_5SNa$ $(M+Na)^+$: 498.1345; Found: 498.1341.

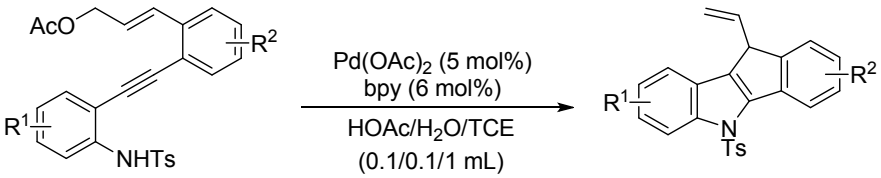



**1q**

**(*E*)-3-(4-((2-(4-Methylphenylsulfonamido)phenyl)ethynyl)-[1,1'-biphenyl]-3-yl)allyl acetate (1q)**

Yellow solid; m.p.: 100-101 $^oC$; (991 mg; 38% yield was obtained based on the amount of *o*-iodoaniline); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 7.79 (s, 1H), 7.69-7.60 (m, 5H), 7.52-7.37 (m, 6H), 7.32-7.24 (m, 2H), 7.16-7.06 (m, 4H), 6.48 (dt, *J* = 16.0 Hz, *J* = 6.4 Hz, 1H), 4.84 (dd, *J* = 6.4 Hz, *J* = 1.2 Hz, 2H), 2.32 (s, 3H), 2.09 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.8, 144.0, 142.1, 140.0, 138.2, 137.5, 136.1, 133.0, 132.1, 131.4, 129.8, 129.6, 129.0, 128.0, 127.3, 127.1, 126.6, 126.5, 124.6, 124.2, 120.3, 119.8, 114.7, 97.3, 89.1, 64.9, 21.5, 21.0; **IR (neat, cm$^{-1}$):** 3327, 2254, 1731, 1337, 1226, 1159, 1089, 907, 758, 728, 661; **HRMS** calculated for $C_{32}H_{27}NO_4SNa$ $(M+Na)^+$: 544.1553; Found: 544.1541.

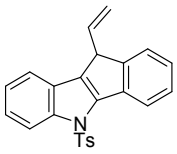
**3.** General procedure for the cyclization of substrates **1** to prepare compounds **2**




**1** **2**

To a dried Schlenk tube were added $Pd(OAc)_2$ (1.2 mg, 0.005 mmol), bpy (0.9 mg, 0.006 mmol), and TCE (1.0 mL). The mixture was stirred at room temperature for 1 min, then substrate **1** (0.1 mmol, 1.0 equiv.), HOAc (0.1 mL) and $H_2O$ (0.1 mL) were added sequentially. The mixture was stirred at 80 $^oC$ until consumption of the substrate (monitored by TLC). The solvent was removed under reduced pressure and

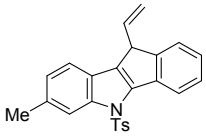
the residue was purified by flash column chromatography (petroleum ether : ethyl acetate = 8:1) to give product **2**.




**2a**

**5-Tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2a)**

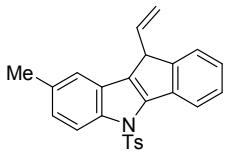
White solid; m.p.: 154-155 °C; (27.7 mg; 72% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.46 (d, *J* = 7.6 Hz, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.50-7.42 (m, 3H), 7.33-7.23 (m, 3H), 7.10 (d, *J* = 8.0 Hz, 2H), 5.69-5.60 (m, 1H), 5.49 (d, *J* = 16.4 Hz, 1H), 5.28 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.33 (d, *J* = 8.0 Hz, 1H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.3, 144.9, 142.3, 141.1, 136.0, 135.3, 134.1, 132.4, 129.9, 128.0, 126.9, 126.8, 126.2, 125.0, 124.6, 124.2, 121.9, 119.3, 117.9, 115.8, 46.9, 21.6; **IR (neat, cm$^{-1}$):** 3050, 1596, 1369, 1172, 1089, 1022, 985, 922, 753, 741, 661; **HRMS** calculated for $C_{24}H_{20}NO_2S$ $(M+H)^+$: 386.1209; Found: 386.1207.




**2b**

**7-Methyl-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2b)**

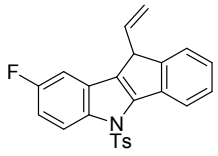
White solid; m.p.: 166-167 °C; (32.7 mg; 82% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.42 (d, *J* = 7.6 Hz, 1H), 8.07 (s, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.43-7.40 (m, 2H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.29-7.25 (m, 1H), 7.11-7.07 (m, 3H), 5.68-5.59 (m, 1H), 5.46 (d, *J* = 16.0 Hz, 1H), 5.26 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.30 (d, *J* = 8.4 Hz, 1H), 2.50 (s, 3H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.0, 144.8, 141.7, 141.5, 136.1, 135.4, 134.9, 134.2, 132.5, 129.9, 127.9, 126.7, 125.9, 125.6, 125.0, 124.6, 121.6, 118.8, 117.8, 115.9, 46.8, 22.3, 21.6; **IR (neat, cm$^{-1}$):** 3043, 2920, 1595, 1367, 1171, 1135, 982, 810, 757, 663; **HRMS** calculated for $C_{25}H_{22}NO_2S$ $(M+H)^+$: 400.1366; Found: 400.1359.




**2c**

**8-Methyl-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2c)**

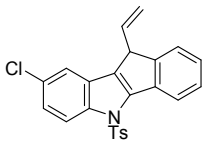
White solid; m.p.: 172-173 °C; (33.1 mg; 83% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.44 (d, *J* = 7.6 Hz, 1H), 8.12 (d, *J* = 8.8 Hz, 1H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.44-7.42 (m, 2H), 7.30-7.25 (m, 2H), 7.13-7.07 (m, 3H), 5.67-5.59 (m, 1H), 5.48 (dd, *J* = 16.8 Hz, *J* = 1.2 Hz, 1H), 5.28 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.29 (d, *J* = 8.4 Hz, 1H), 2.40 (s, 3H), 2.25 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.2, 144.7, 142.4, 139.3, 136.1, 135.2, 134.2, 133.9, 132.3, 129.8, 128.0, 127.1, 126.8, 126.1, 126.0, 125.0, 121.7, 119.2, 117.9, 115.5, 46.8, 21.6, 21.4; **IR (neat, cm$^{-1}$):** 2953, 2917, 1597, 1367, 1190, 1170, 1091, 979, 805, 737, 661; **HRMS** calculated for $C_{25}H_{22}NO_2S$ $(M+H)^+$: 400.1366; Found: 400.1359.




**2d**

**8-Fluoro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2d)**

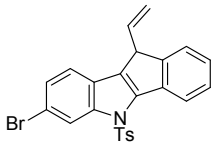
Yellow solid; m.p.: 147-148 °C; (31.8 mg; 79% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.46 (d, *J* = 7.2 Hz, 1H), 8.20 (dd, *J* = 9.2 Hz, *J* = 4.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.46-7.42 (m, 2H), 7.34-7.30 (m, 1H), 7.13-7.10 (m, 3H), 7.01 (td, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H), 5.65-5.57 (m, 1H), 5.47 (dd, *J* = 16.8 Hz, *J* = 1.2 Hz, 1H), 5.29 (dd, *J* = 10.0 Hz, *J* = 1.6 Hz, 1H), 4.30 (d, *J* = 8.4 Hz, 1H), 2.28 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 160.2 (d, *J* = 239.8 Hz), 150.3, 145.1, 144.1, 137.3, 135.6, 134.9, 133.7, 131.9 (d, *J* = 4.5 Hz), 129.9, 128.1, 127.9 (d, *J* = 10.0 Hz), 126.7, 126.6, 125.1, 122.1, 118.2, 116.9 (d, *J* = 9.2 Hz), 112.2 (d, *J* = 25.2 Hz), 104.9 (d, *J* = 23.7 Hz), 46.8, 21.6; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -118.5; **IR (neat, cm$^{-1}$):** 2915, 1609, 1369, 1169, 1021, 964, 862, 804, 662; **HRMS** calculated for $C_{24}H_{19}NO_2FS$ $(M+H)^+$: 404.1115; Found: 404.1110.




**2e**

**8-Chloro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2e)**

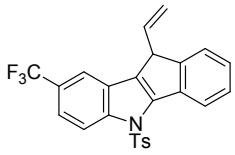
Yellow solid; m.p.: 183-184 $^{o}$C; (33.1 mg; 79% yield); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 8.45 (dd, *J* = 7.6 Hz, *J* = 2.4 Hz, 1H), 8.17 (d, *J* = 9.2 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.46-7.42 (m, 3H), 7.34-7.30 (m, 1H), 7.26-7.24 (m, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 5.65-5.56 (m, 1H), 5.48 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.29 (dd, *J* = 9.6 Hz, *J* = 2.0 Hz, 1H), 4.30 (d, *J* = 8.0 Hz, 1H), 2.28 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.3, 145.2, 143.7, 139.3, 135.5, 135.0, 133.6, 131.3, 130.1, 130.0, 128.1, 128.0, 126.7, 125.1, 124.6, 122.1, 118.8, 118.3, 116.8, 46.8, 21.6; **IR (neat, cm$^{-1}$):** 3062, 2914, 1597, 1369, 1171, 977, 805, 749, 661; **HRMS** calculated for $C_{24}H_{19}NO_2ClS$ $(M+H)^+$: 420.0820; Found: 420.0813.




**2f**

**7-Bromo-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2f)**

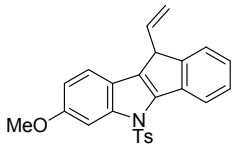
Yellow solid; m.p.: 151-152 $^{o}$C; (32.0 mg; 69% yield); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 8.44-8.42 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.45-7.41 (m, 2H), 7.38-7.29 (m, 3H), 7.14 (d, *J* = 8.4 Hz, 2H), 5.66-5.57 (m, 1H), 5.46 (dd, *J* = 16.8 Hz, *J* = 0.8 Hz, 1H), 5.27 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.31 (d, *J* = 8.0 Hz, 1H), 2.29 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.2, 145.2, 142.8, 141.5, 135.7, 135.0, 133.6, 131.7, 130.1, 128.1, 127.5, 126.8, 126.5, 125.6, 125.1, 121.9, 120.2, 118.7, 118.1, 46.8, 21.7; **IR (neat, cm$^{-1}$):** 2920, 2851, 1595, 1370, 1170, 980, 921, 807, 746, 688; **HRMS** calculated for $C_{24}H_{19}NO_2BrS$ $(M+H)^+$: 464.0314; Found: 464.0313.




**2g**

**5-Tosyl-8-(trifluoromethyl)-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2g)**

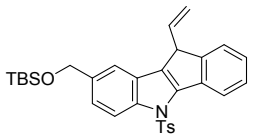
White solid; m.p.: 136-137 °C; (30.8 mg; 68% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.48 (d, *J* = 7.6 Hz, 1H), 8.36 (d, *J* = 8.8 Hz, 1H), 7.74-7.68 (m, 3H), 7.54 (dd, *J* = 8.8 Hz, *J* = 1.2 Hz, 1H), 7.48-7.44 (m, 2H), 7.36-7.32 (m, 1H), 7.14 (d, *J* = 8.4 Hz, 2H), 5.68-5.59 (m, 1H), 5.52 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.32 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.36 (d, *J* = 8.0 Hz, 1H), 2.29 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.4, 145.4, 144.0, 142.3, 135.4, 135.0, 133.4, 131.6, 130.1, 128.2, 126.9, 126.8, 126.5 (q, *J* = 32.2 Hz), 126.5, 125.3, 124.6 (q, *J* = 270.4 Hz), 122.2, 121.2 (q, *J* = 3.8 Hz), 118.5, 116.5 (q, *J* = 3.8 Hz), 115.9, 46.8, 21.7; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -61.3; **IR (neat, cm$^{-1}$):** 2917, 1596, 1314, 1267, 1174, 1125, 1092, 978, 860, 664; **HRMS** calculated for $C_{25}H_{19}NO_2F_3S$ $(M+H)^+$: 454.1083; Found: 454.1083.




**2h**

**7-Methoxy-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2h)**

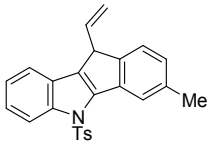
Yellow solid; m.p.: 131-132 °C; (33.2 mg; 80% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.38 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 2.4 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.42-7.34 (m, 3H), 7.27-7.23 (m, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 6.88 (dd, *J* = 8.4 Hz, *J* = 2.0 Hz, 1H), 5.68-5.59 (m, 1H), 5.45 (dt, *J* = 17.2 Hz, *J* = 0.8 Hz, 1H), 5.25 (dd, *J* = 10.0 Hz, *J* = 1.6 Hz, 1H), 4.28 (d, *J* = 8.4 Hz, 1H), 3.91 (s, 3H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 158.0, 149.7, 144.9, 142.2, 141.1, 136.1, 135.2, 134.4, 132.6, 129.9, 127.9, 126.7, 125.6, 124.9, 121.2, 120.9, 119.7, 117.8, 112.9, 100.6, 56.1, 46.8, 21.6; **IR (neat, cm$^{-1}$):** 2917, 1619, 1490, 1371, 1274, 1170, 1130, 985, 743, 662; **HRMS** calculated for $C_{25}H_{22}NO_3S$ $(M+H)^+$: 416.1315; Found: 416.1313.




**2i**

**8-(((tert-Butyldimethylsilyl)oxy)methyl)-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2i)**

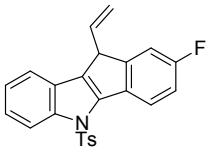
White solid; m.p.: 72-73 $^{o}$C; (38.1 mg; 72% yield); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 8.44 (d, *J* = 8.0 Hz, 1H), 8.19 (d, *J* = 8.8 Hz, 1H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.47-7.41 (m, 3H), 7.31-7.23 (m, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 5.67-5.58 (m, 1H), 5.49 (dd, *J* = 16.8 Hz, *J* = 1.2 Hz, 1H), 5.27 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.80 (s, 2H), 4.32 (d, *J* = 8.4 Hz, 1H), 2.26 (s, 3H), 0.95 (s, 9H), 0.10 (d, *J* = 1.2 Hz, 6H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 150.2, 144.8, 142.5, 140.2, 137.6, 136.0, 135.2, 134.1, 132.6, 129.8, 128.0, 126.9, 126.8, 126.1, 125.0, 122.9, 121.8, 117.9, 116.6, 115.5, 64.9, 46.9, 26.1, 21.6, 18.5, -5.0, -5.1; **IR (neat, cm$^{-1}$):** 2933, 2889, 2854, 1598, 1454, 1369, 1254, 1173 1083, 838 763, 664; **HRMS** calculated for $C_{31}H_{36}NO_3SSi$ $(M+H)^+$: 530.2180; Found: 530.2179.




**2j**

**3-Methyl-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2j)**

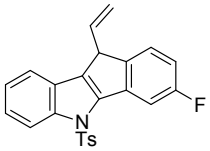
White solid; m.p.: 126-127 $^{o}$C; (31.9 mg; 80% yield); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 8.28 (s, 1H), 8.24 (d, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.33-7.21 (m, 3H), 7.12-7.08 (m, 3H), 5.66-5.58 (m, 1H), 5.46 (dd, *J* = 17.2 Hz, *J* = 1.2 Hz, 1H), 5.25 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.29 (d, *J* = 8.4 Hz, 1H), 2.49 (s, 3H), 2.26 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 147.4, 144.8, 142.3, 141.0, 137.7, 136.2, 135.2, 134.2, 132.8, 129.8, 127.0, 126.8, 124.7, 124.5, 124.1, 122.6, 119.2, 117.6, 115.8, 46.5, 22.0, 21.6; **IR (neat, cm$^{-1}$):** 2918, 1633, 1370, 1176, 985, 809, 761, 662; **HRMS** calculated for $C_{25}H_{21}NO_2SNa$ $(M+Na)^+$: 422.1185; Found: 422.1185.




**2k**

**2-Fluoro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2k)**

White solid; m.p.: 128-129 °C; (28.6 mg; 71% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.40 (dd, *J* = 8.8 Hz, *J* = 5.6 Hz, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.33-7.22 (m, 2H), 7.17-7.10 (m, 4H), 5.67-5.59 (m, 1H), 5.48 (dt, *J* = 16.8 Hz, *J* = 0.8 Hz, 1H), 5.30 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.31 (d, *J* = 8.4 Hz, 1H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 162.0 (d, *J* = 245.2 Hz), 152.7 (d, *J* = 7.6 Hz), 145.0, 141.5, 140.8, 135.4, 135.1, 132.1 (d, *J* = 3.1 Hz), 130.1 (d, *J* = 2.3 Hz), 129.9, 126.8, 126.7, 124.6, 124.3, 122.7 (d, *J* = 8.5 Hz), 119.1, 118.4, 115.8, 114.6 (d, *J* = 22.2 Hz), 113.0 (d, *J* = 23.8 Hz), 47.0 (d, *J* = 2.3 Hz), 21.6; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -115.8; **IR (neat, cm$^{-1}$):** 2918, 1592, 1374, 1218, 1189, 1122, 982, 923, 819, 757, 671; **HRMS** calculated for $C_{24}H_{19}NFO_2S$ $(M+H)^+$: 404.1115; Found: 404.1109.

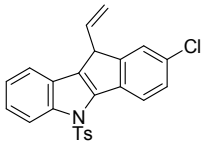



**2l**

**3-Fluoro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2l)**

White solid; m.p.: 172-173 °C; (29.0 mg; 72% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.25 (d, *J* = 8.4 Hz, 1H), 8.19 (dd, *J* = 10.4 Hz, *J* = 2.4 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.36-7.24 (m, 3H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.98 (dt, *J* = 11.2 Hz, *J* = 2.0 Hz, 1H), 5.65-5.56 (m, 1H), 5.48 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.28 (dd, *J* = 10.0 Hz, *J* = 1.6 Hz, 1H), 4.30 (d, *J* = 8.0 Hz, 1H), 2.28 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 163.1 (d, *J* = 241.4 Hz), 145.5 (d, *J* = 3.1 Hz), 145.1, 141.2, 141.0, 135.7 (d, *J* = 10.8 Hz), 135.7, 135.1, 134.2, 129.9, 126.8, 126.6, 125.7 (d, *J* = 9.2 Hz), 125.1, 124.3, 119.4, 118.1, 115.8, 112.6 (d, *J* = 23.0 Hz), 109.5 (d, *J* = 26.8 Hz), 46.4, 21.6; **$^{19}$F NMR (376 MHz, $CDCl_3$):** δ -114.1; **IR (neat, cm$^{-1}$):** 2915,

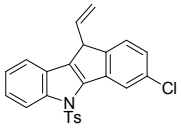
1594, 1457, 1366, 1188, 1170, 987, 911, 805, 758, 740, 672; **HRMS** calculated for $C_{24}H_{19}NFO_2S$ $(M+H)^+$: 404.1115; Found: 404.1110.




**2m**

**2-Chloro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2m)**

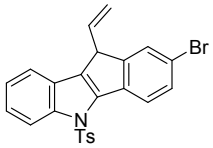
White solid; m.p.: 143-144 °C; (30.2 mg; 72% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.36 (dd, *J* = 7.6 Hz, *J* = 2.0 Hz, 1H), 8.24 (dd, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.41-7.23 (m, 4H), 7.10 (d, *J* = 8.4 Hz, 2H), 5.66-5.57 (m, 1H), 5.49 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.30 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.31 (d, *J* = 8.0 Hz, 1H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 151.9, 145.0, 141.4, 141.0, 135.1, 135.0, 132.6, 132.2, 129.9, 128.1, 126.7, 125.5, 125.0, 124.3, 122.6, 119.3, 118.6, 115.8, 46.8, 21.6; **IR (neat, $cm^{-1}$):** 3083, 2921, 1596, 1367, 1171, 1140, 982, 746, 666; **HRMS** calculated for $C_{24}H_{19}NClO_2S$ $(M+H)^+$: 420.0820; Found: 420.0814.




**2n**

**3-Chloro-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2n)**

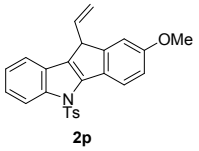
White solid; m.p.: 144-145 °C; (29.4 mg; 70% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.45 (d, *J* = 2.0 Hz, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.35-7.31 (m, 2H), 7.27-7.23 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 5.64-5.56 (m, 1H), 5.48 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.28 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.30 (d, *J* = 8.0 Hz, 1H), 2.28 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 148.4, 145.1, 141.1, 141.0, 135.6, 135.3, 135.0, 134.1, 133.8, 130.0, 126.8, 126.6, 126.0, 125.8, 125.2, 124.3, 122.0, 119.5, 118.3, 115.8, 46.5, 21.6; **IR (neat, $cm^{-1}$):** 2948, 1597, 1442, 1366, 1187, 1169, 985, 877, 761, 666; **HRMS** calculated for $C_{24}H_{19}NClO_2S$ $(M+H)^+$: 420.0820; Found: 420.0818.




**2o**

**2-Bromo-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2o)**

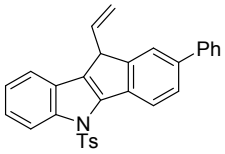
Yellow solid; m.p.: 153-154 °C; (30.1 mg; 65% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.31 (d, *J* = 9.2 Hz, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.56-7.54 (m, 2H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 5.65-5.57 (m, 1H), 5.48 (dd, *J* = 16.8 Hz, *J* = 1.6 Hz, 1H), 5.30 (dt, *J* = 9.6 Hz, *J* = 0.8 Hz, 1H), 4.31 (d, *J* = 8.0 Hz, 1H), 2.27 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 152.2, 145.1, 141.4, 141.1, 135.1, 135.0, 133.0, 132.6, 131.0, 130.0, 128.4, 126.7, 126.6, 125.0, 124.3, 123.0, 120.3, 119.3, 118.6, 115.8, 46.9, 21.6; **IR (neat, cm$^{-1}$):** 2920, 1596, 1441, 1368, 1171, 1144, 982, 762, 664; **HRMS** calculated for $C_{24}H_{19}NBrO_2S$ $(M+H)^+$: 464.0314; Found: 464.0314.




**2p**

**2-Methoxy-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2p)**

Yellow solid; m.p.: 149-150 °C; (30.7 mg; 74% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.35 (d, *J* = 8.8 Hz, 1H), 8.22 (d, *J* = 7.6 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.44-7.42 (m, 1H), 7.28-7.20 (m, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 7.02 (t, *J* = 2.4 Hz, 1H), 6.95 (dd, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H), 5.69-5.60 (m, 1H), 5.47 (dt, *J* = 16.8 Hz, *J* = 0.8 Hz, 1H), 5.27 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.29 (d, *J* = 7.6 Hz, 1H), 3.87 (s, 3H), 2.26 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 158.8, 152.5, 144.8, 142.3, 140.6, 136.2, 135.3, 130.5, 129.8, 127.1, 127.0, 126.8, 124.1, 124.0, 122.5, 118.8, 117.9, 115.7, 112.8, 111.9, 55.7, 47.0, 21.6; **IR (neat, cm$^{-1}$):** 2958, 1597, 1364, 1291, 1238, 1174, 1133, 1023, 919, 821, 742, 719, 666; **HRMS** calculated for $C_{25}H_{22}NO_3S$ $(M+H)^+$: 416.1315; Found: 416.1308.

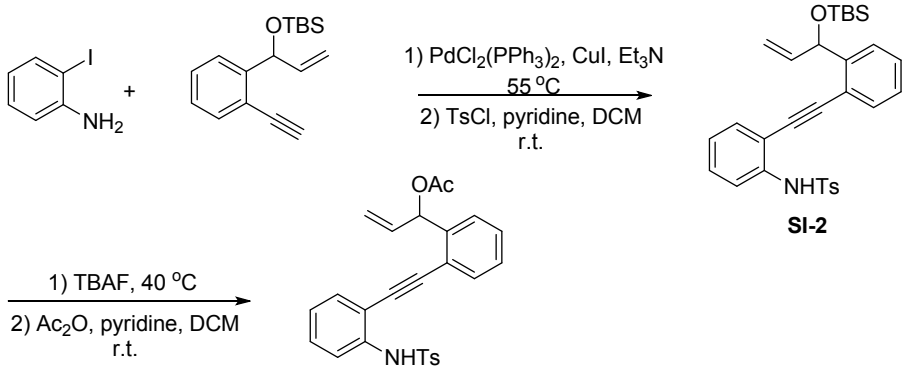



**2q**

**2-Phenyl-5-tosyl-10-vinyl-5,10-dihydroindeno[1,2-*b*]indole (2q)**

Brown solid; m.p.: 156-157 °C; (28.1 mg; 61% yield); **$^1$H NMR (400 MHz, $CDCl_3$):** δ 8.51 (d, *J* = 8.8 Hz, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 7.71-7.66 (m, 6H), 7.51-5.45 (m, 3H), 7.38-7.25 (m, 3H), 7.12 (d, *J* = 8.4 Hz, 2H), 5.75-5.65 (m, 1H), 5.53 (dd, *J* = 16.8 Hz, *J* = 1.2 Hz, 1H), 5.30 (dd, *J* = 9.6 Hz, *J* = 1.6 Hz, 1H), 4.40 (d, *J* = 7.6 Hz, 1H), 2.28 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 151.0, 144.9, 142.1, 141.1, 141.0, 139.2, 136.0, 135.3, 133.2, 132.6, 130.0, 129.0, 127.4, 127.2, 127.0, 126.8, 124.7, 124.2, 123.8, 122.0, 119.3, 118.1, 115.7, 47.0, 21.6; **IR (neat, cm$^{-1}$):** 2919, 1596, 1370, 1174, 1095, 973, 810, 755, 697, 671; **HRMS** calculated for $C_{30}H_{24}NO_2S$ $(M+H)^+$: 462.1522; Found: 462.1515.

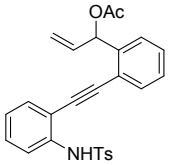
**4.** Procedure for the synthesis of substrate **3a**




In a 50 mL single neck flask, *o*-iodoaniline (4.16 mmol, 1.0 equiv.) and alkyne (4.16 mmol, 1.0 equiv) were dissolved in $Et_3N$ (25 mL). Then $PdCl_2(PPh_3)_2$ (0.04 mmol, 0.01 equiv), CuI (0.04 mmol, 0.01 equiv) were added to the mixture and the resulting solution was stirred at 55 °C. After completion of the reaction, the mixture was filtered by a short silica column, the solvent was evaporated under reduced pressure. The residue was dissolved in DCM (20 mL), then pyridine (6.24 mmol, 1.5 equiv) and TsCl (5.0 mmol, 1.2 equiv) were added sequentially. The reaction was stirred at room temperature overnight. After completion of the reaction, the mixture

was filtered by a short silica column, the solvent was evaporated under reduced pressure to obtain the crude product **SI-2** (3.24 mmol, 1.68 g).

The crude product **SI-2** was dissolved in anhydrous THF (20 mL), the mixture was heated to 60 $^{o}$C, then TBAF (3.88 mL, 1 M in THF) was added to the reaction mixture, followed by stirring for additional half an hour at the same temperature. After completion of the reaction, the solvent was evaporated under reduced pressure. The residue was dissolved in DCM (10 mL), then pyridine (4.08 mmol) and $Ac_2O$ (3.26 mmol) were added sequentially. The reaction was stirred at 0 $^{o}$C. After completion of the reaction, the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography (petroleum ether : ethyl acetate = 4:1) to obtain the compound **3a**.

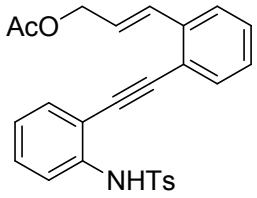


**3a**

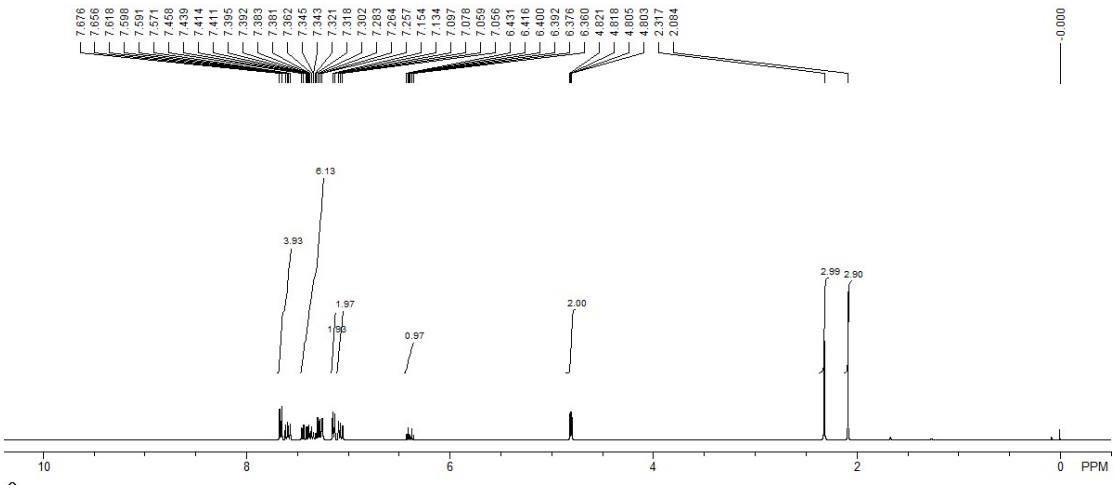
**1-(2-((2-(4-Methylphenylsulfonamido)phenyl)ethynyl)phenyl)allyl acetate (3a)**

Yellow oil; (833 mg; 45% yield was obtained based on the amount of *o*-iodoaniline); **$^{1}$H NMR (400 MHz, $CDCl_3$):** δ 7.72-7.70 (m, 3H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.48-7.25 (m, 6H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.05 (td, *J* = 7.6 Hz, *J* = 0.8 Hz, 1H), 6.71 (d, *J* = 5.2 Hz, 1H), 6.14-6.06 (m, 1H), 5.33-5.28 (m, 2H), 2.32 (s, 3H), 2.13 (s, 3H); **$^{13}$C NMR (100 MHz, $CDCl_3$):** δ 170.3, 143.9, 140.2, 138.0, 136.6, 135.2, 132.5, 132.4, 129.8, 129.6, 129.3, 128.2, 127.5, 127.3, 124.4, 121.6, 120.1, 117.6, 114.3, 93.4, 88.9, 74.2, 21.5, 21.2; **IR (neat, cm$^{-1}$):** 3251, 2922, 1730, 1338, 1229, 1158, 1089, 909, 755, 660; **HRMS** calculated for $C_{26}H_{23}NO_4SNa$ $(M+Na)^+$: 468.1240; Found: 468.1238.

**5.** Copies of $^{1}$H NMR, $^{13}$C NMR and $^{19}$F NMR spectra


AcO
NHTs

**1a**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4.803
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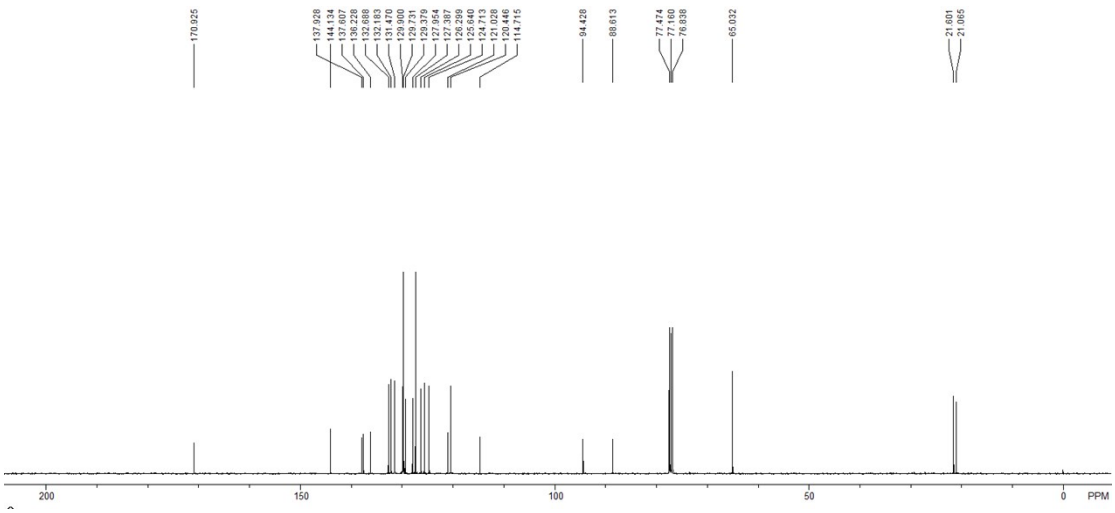
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126.299
125.640
124.713
121.028
120.446
114.715
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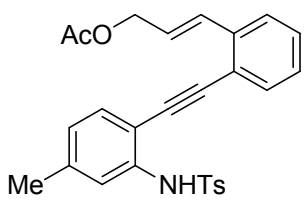
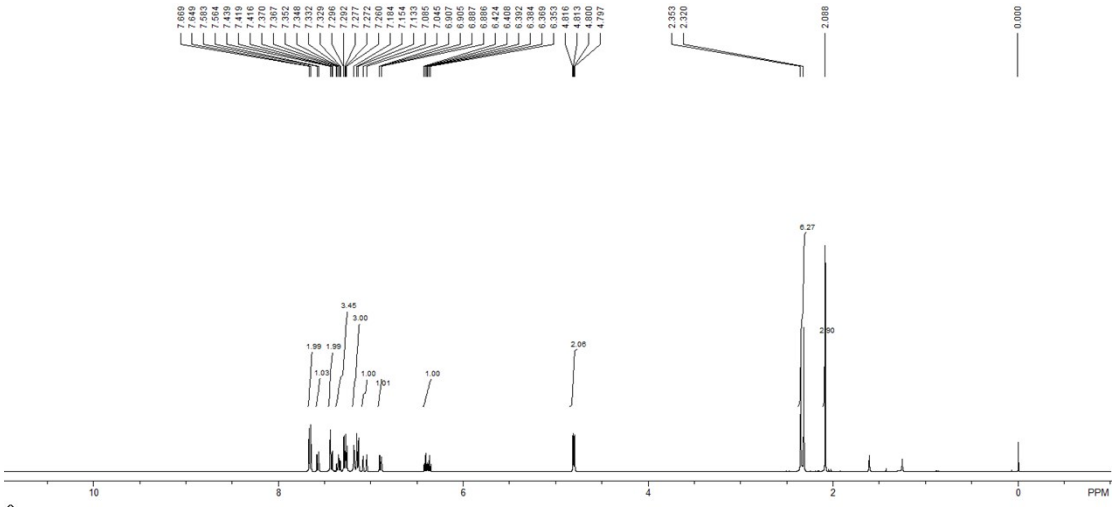

AcO
Me
NHTs

**1b**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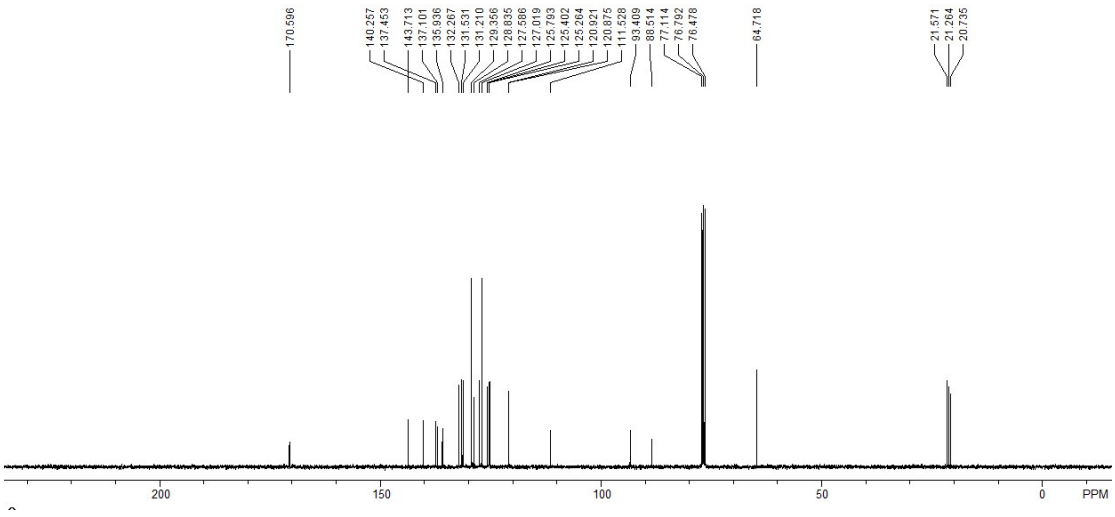
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120.921
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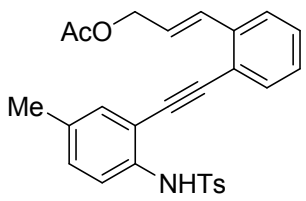
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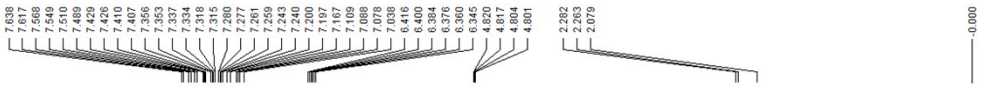
NHTs

**1c**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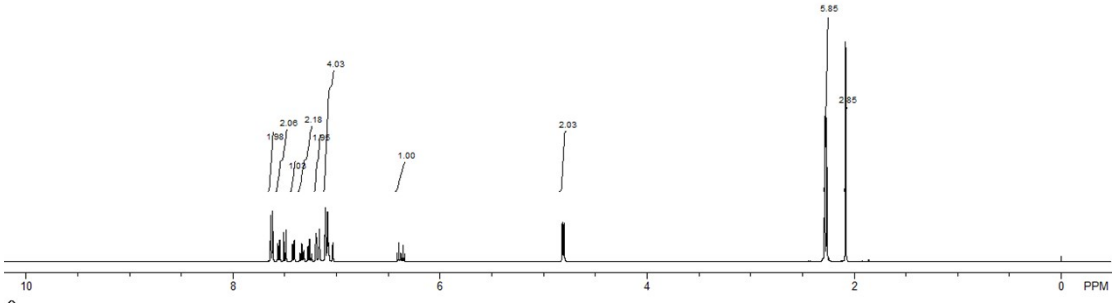
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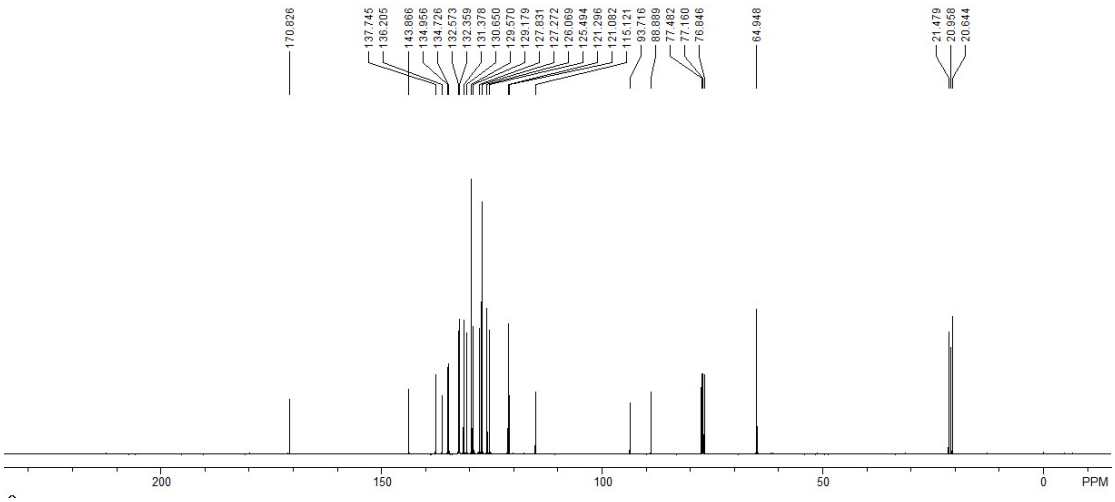
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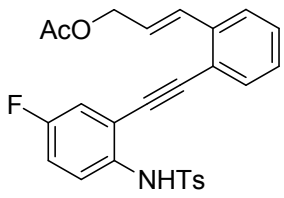
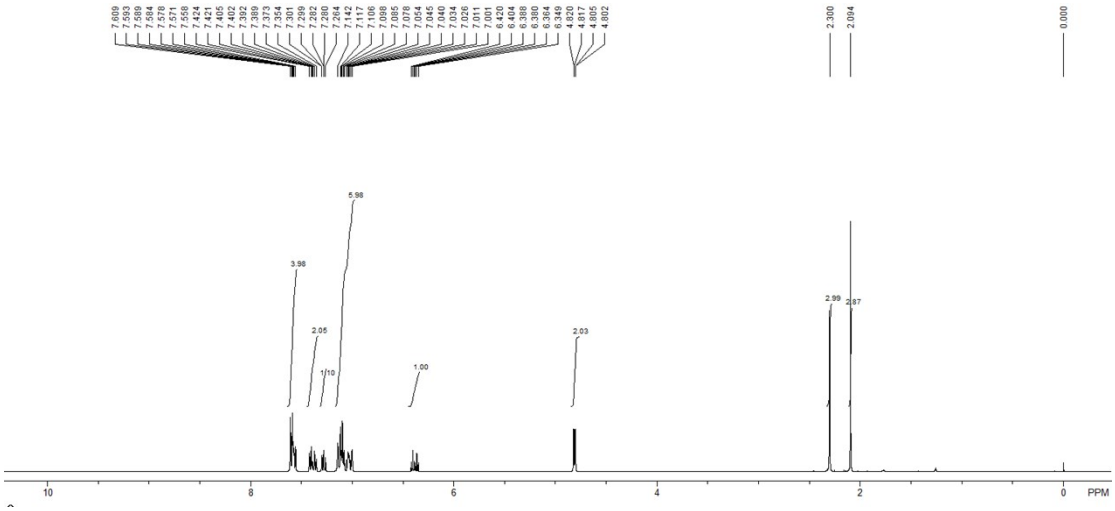

AcO
F
NHTs

**1d**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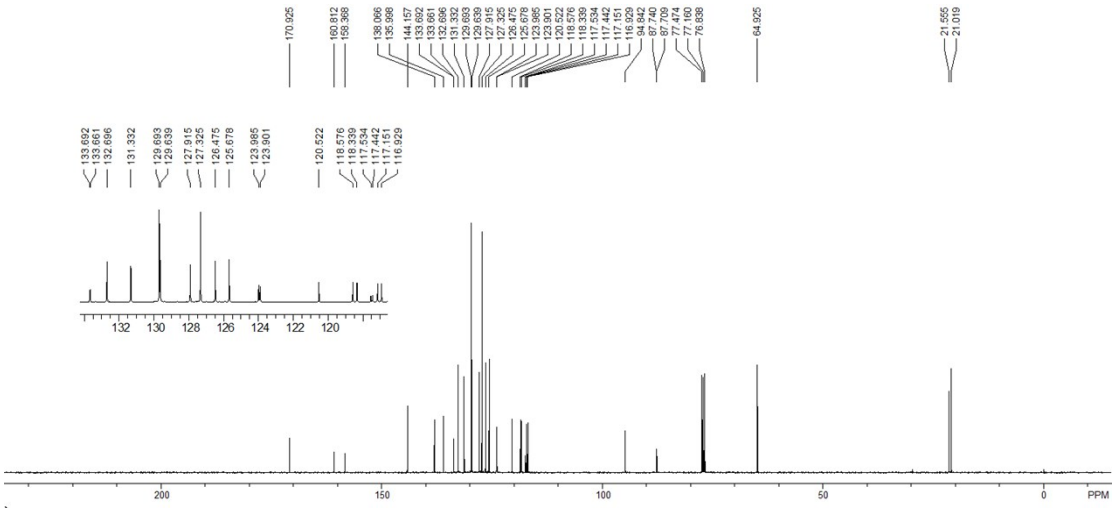
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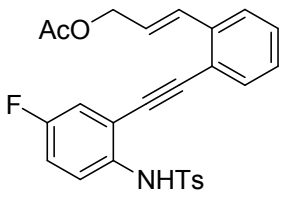
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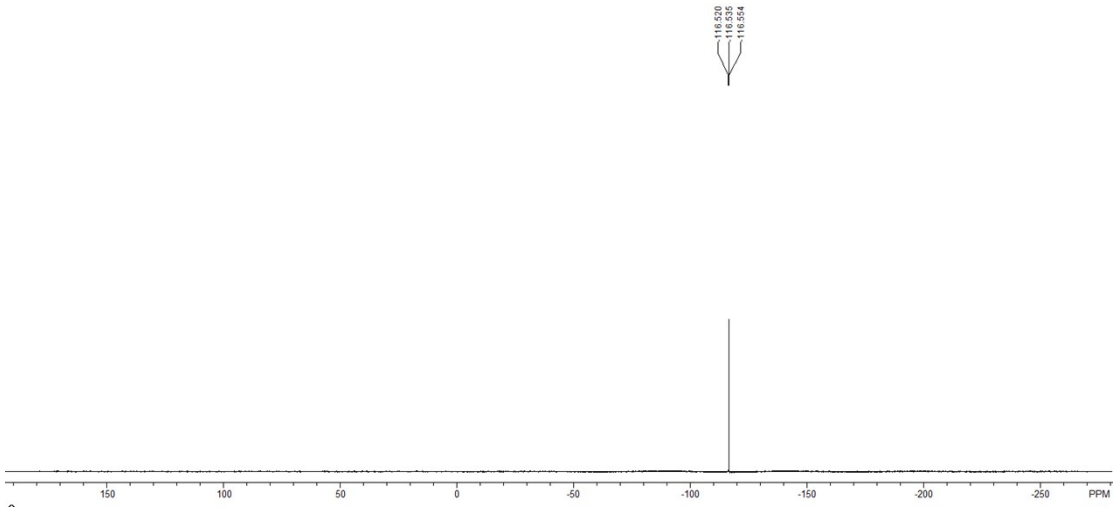
NHTs

**1d**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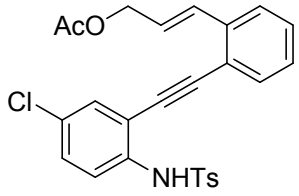
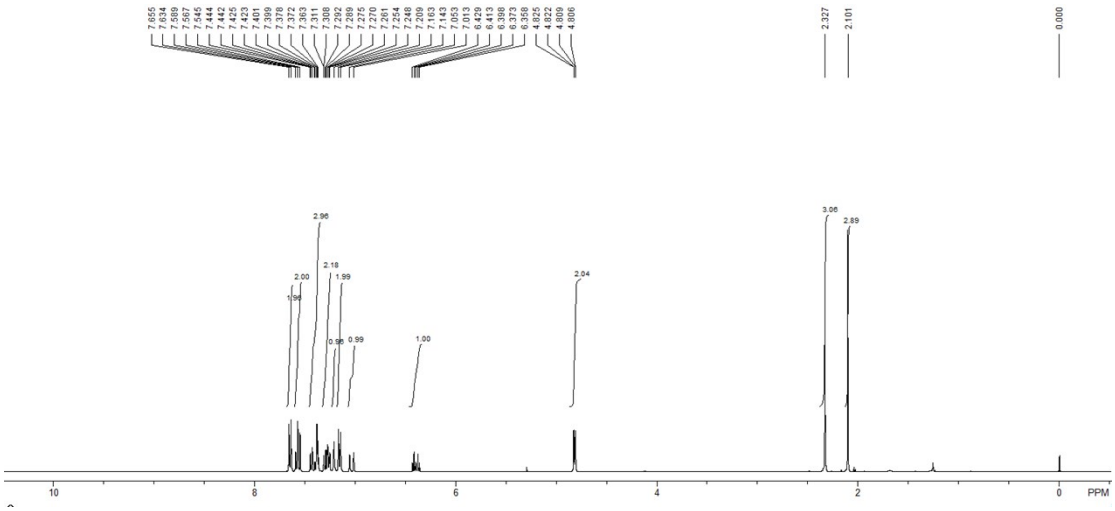

AcO
Cl
NHTs

**1e**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7.254
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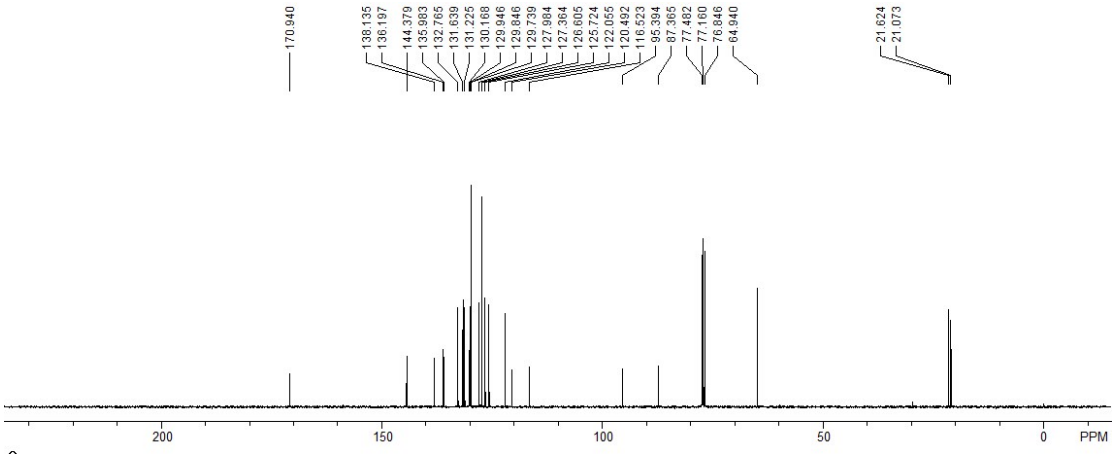
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127.364
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125.724
122.055
120.492
116.523
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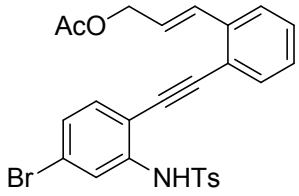
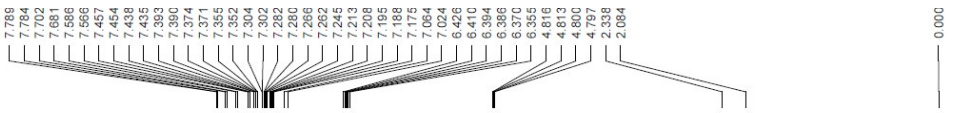

AcO
Br
NHTs

**1f**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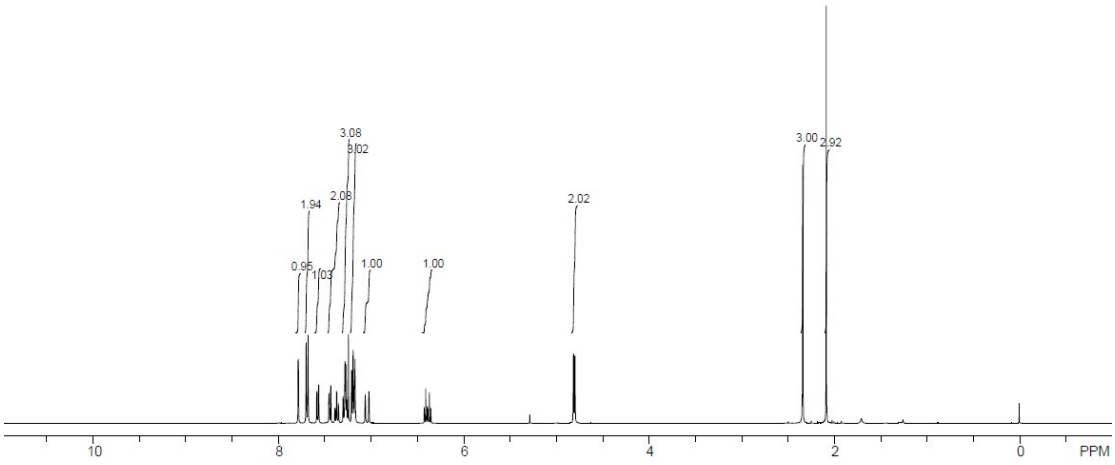
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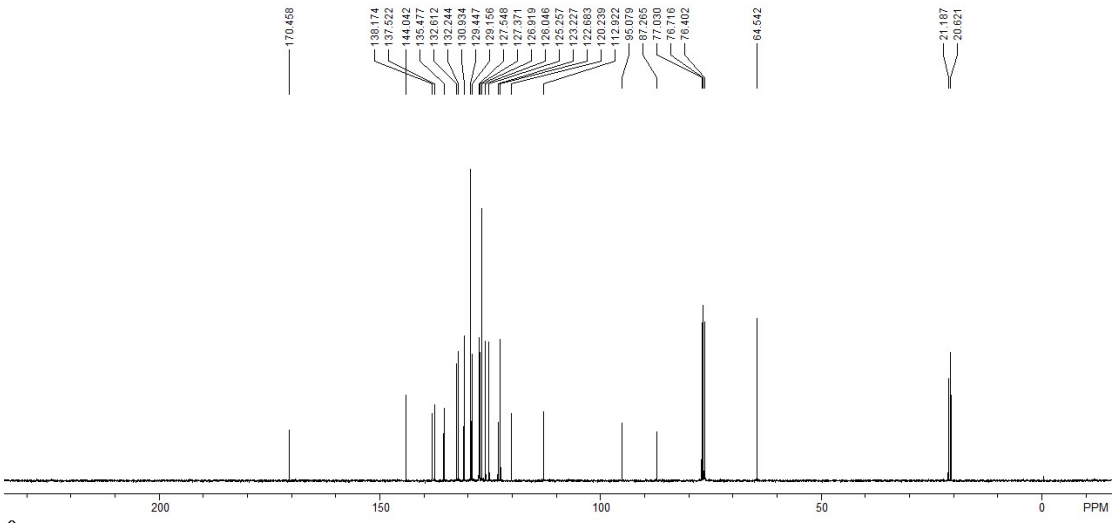
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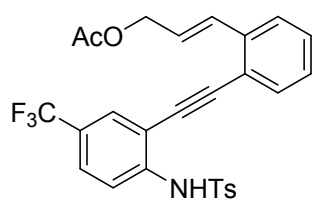
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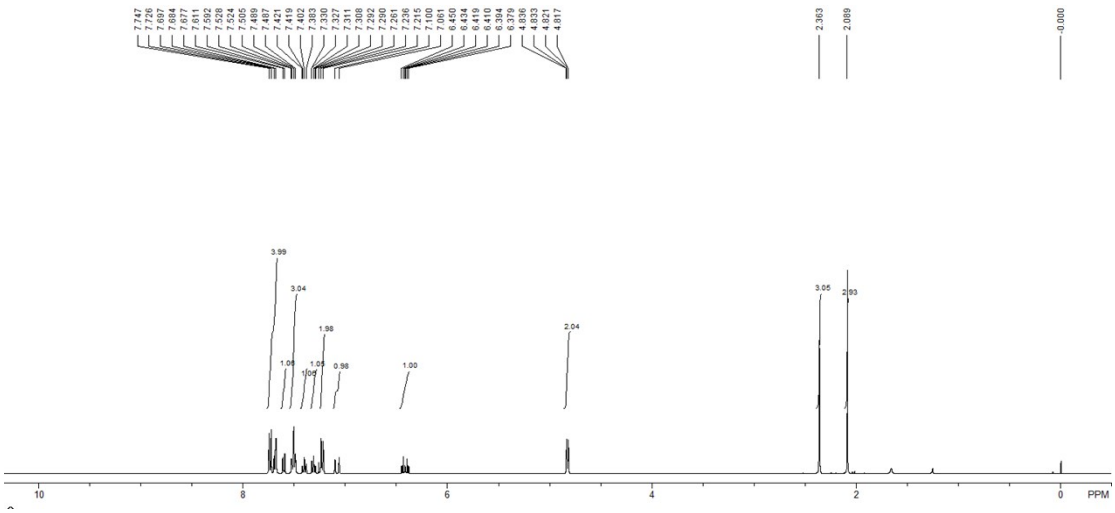
F3C
NHTs

**1g**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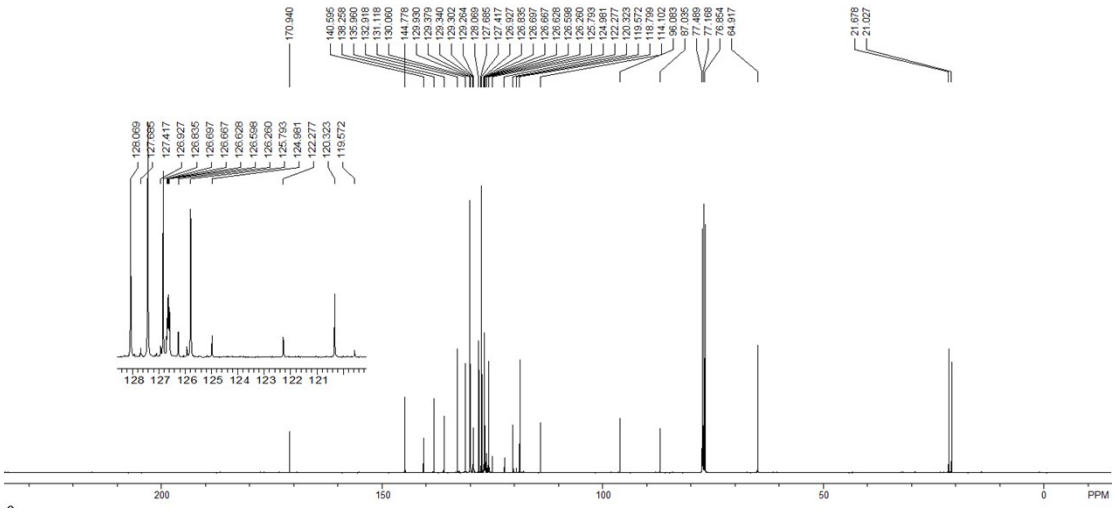
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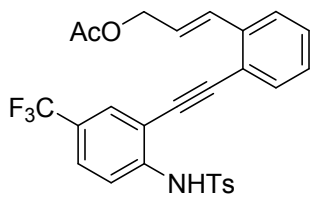
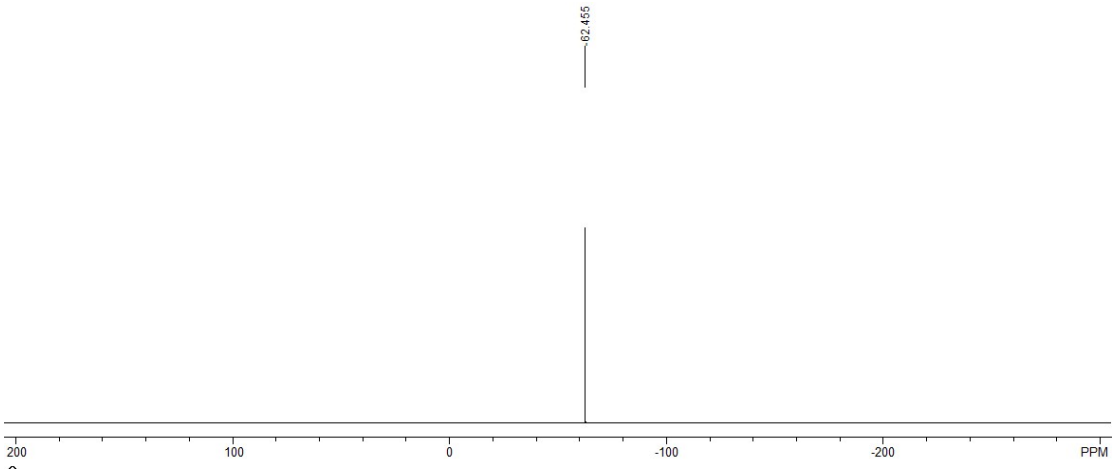
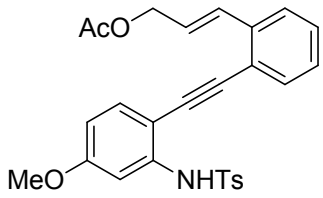
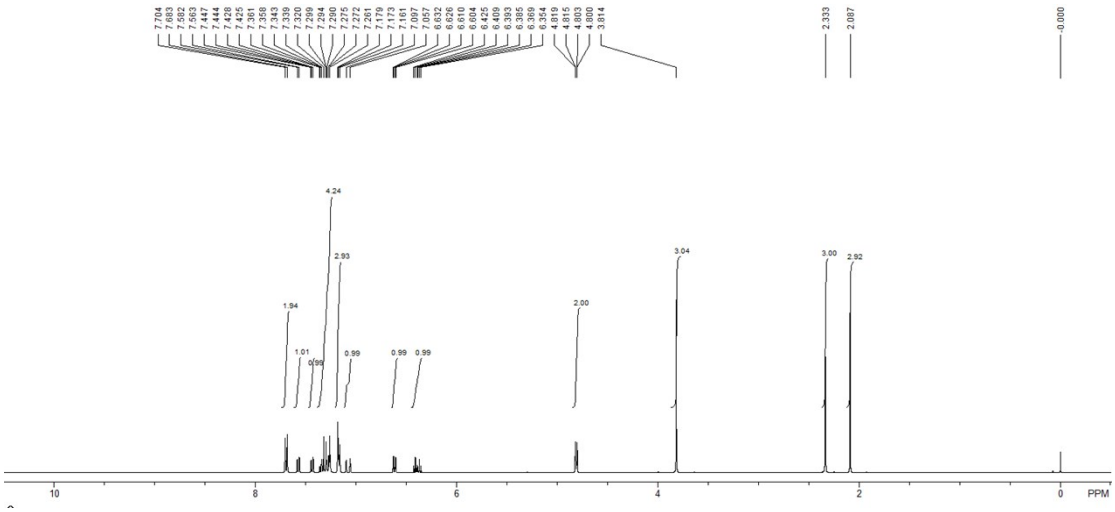



**1g**




AcO
MeO
NHTs

**1h**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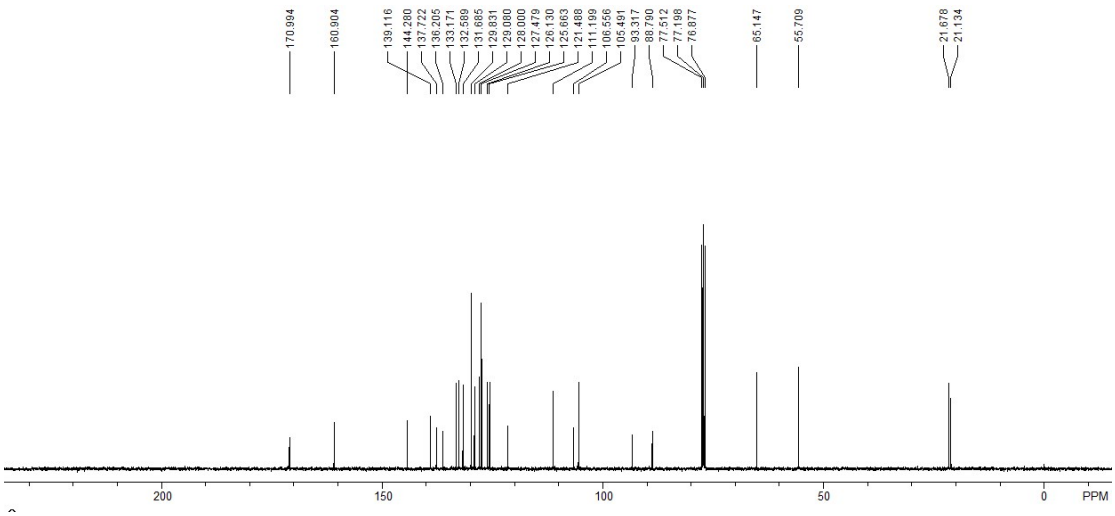
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111.199
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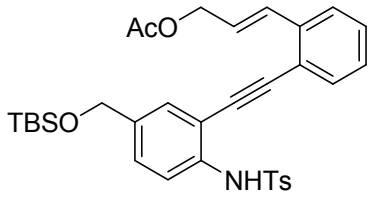
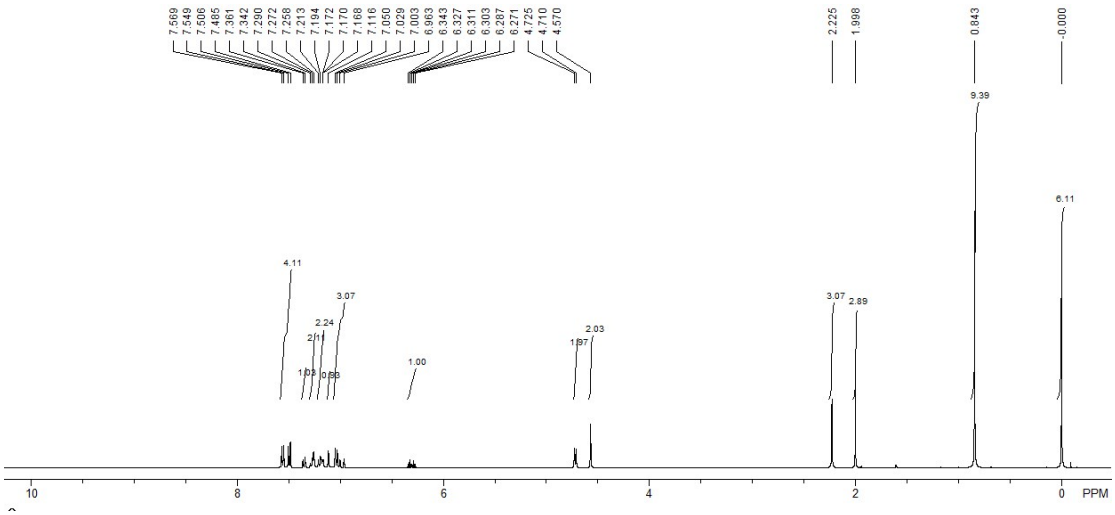

AcO
TBSO
NHTs

**1i**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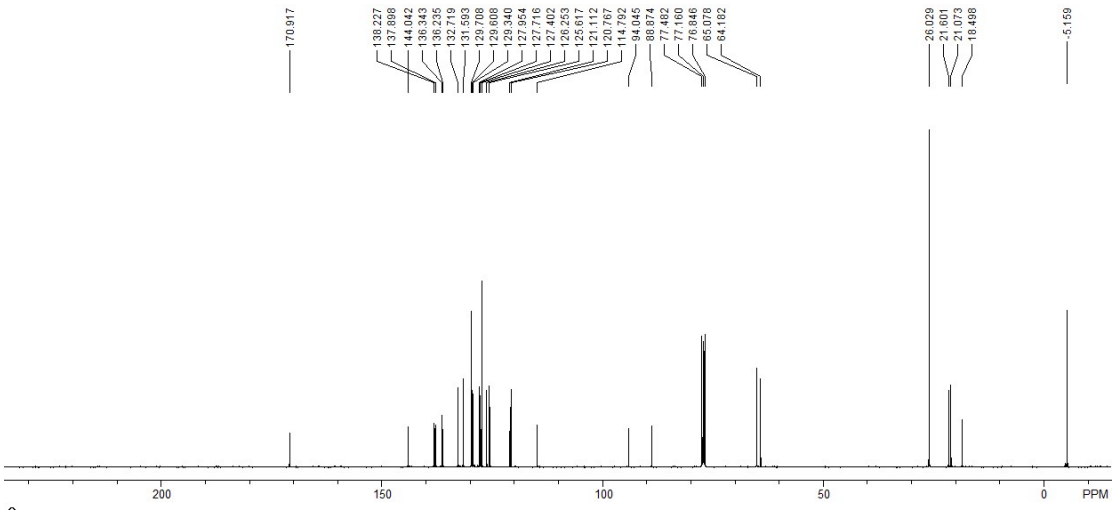
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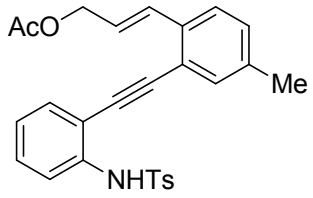
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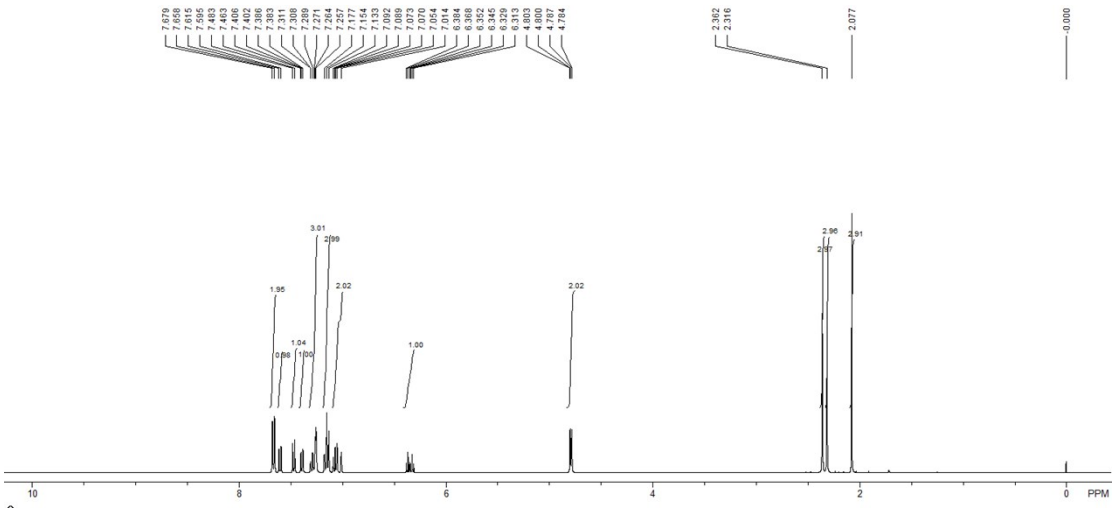
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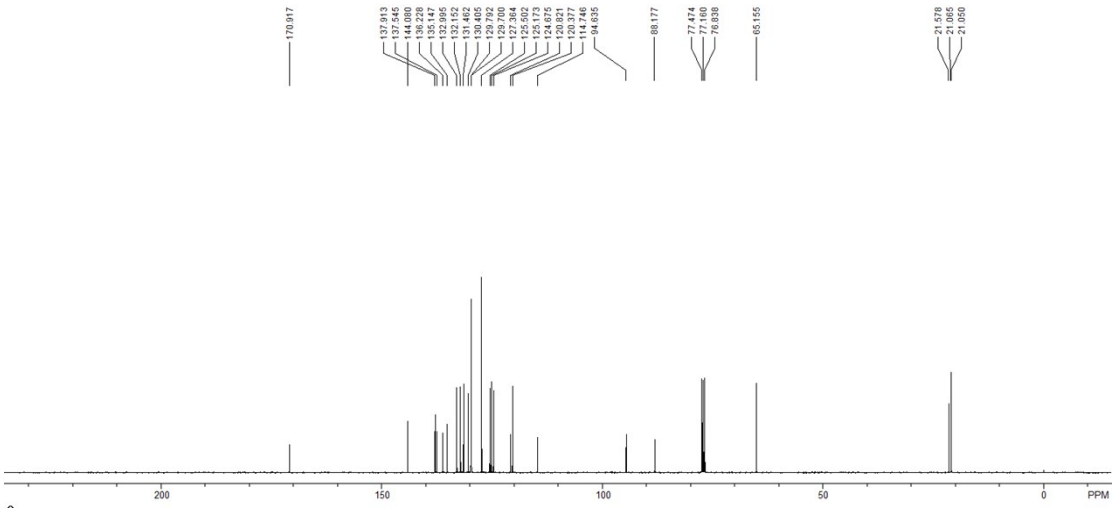
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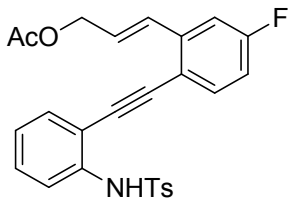
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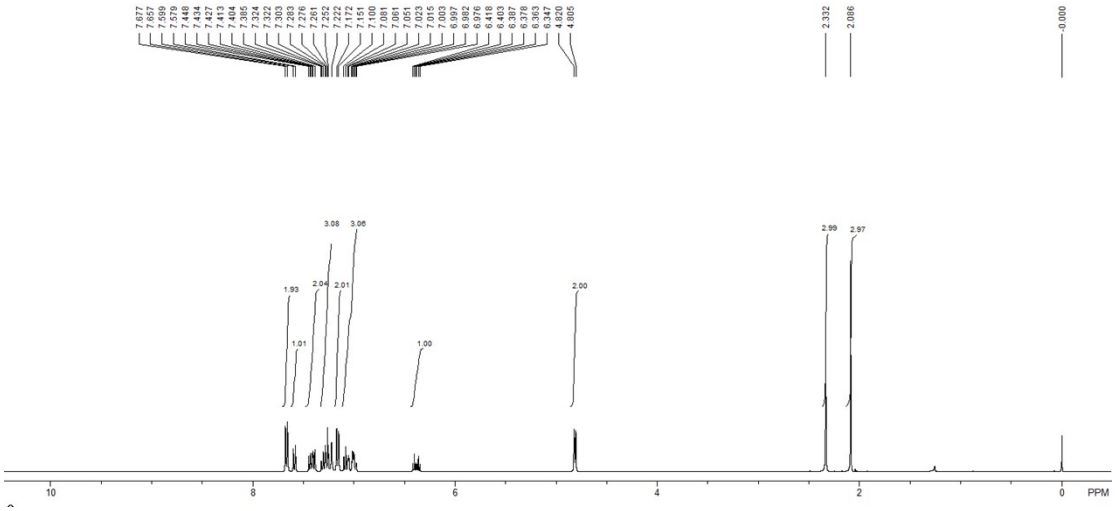
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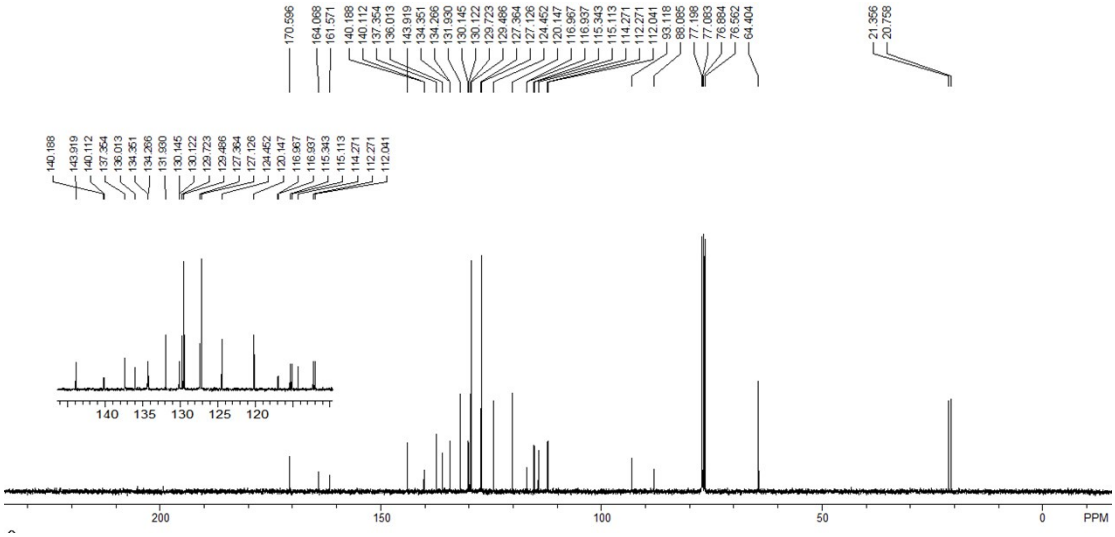
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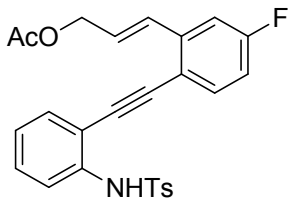


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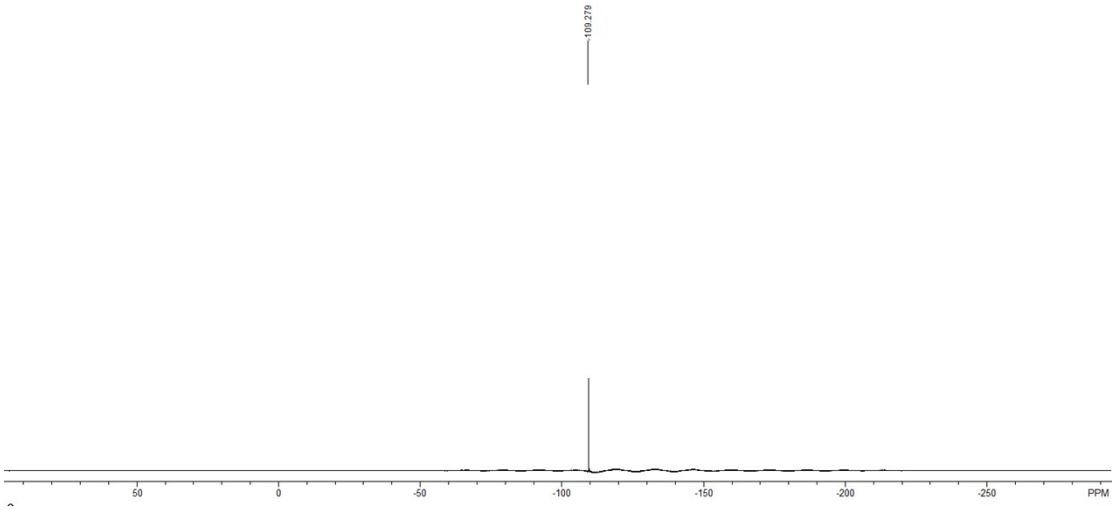

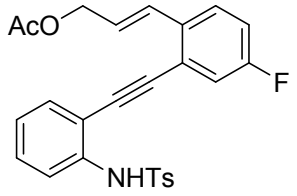
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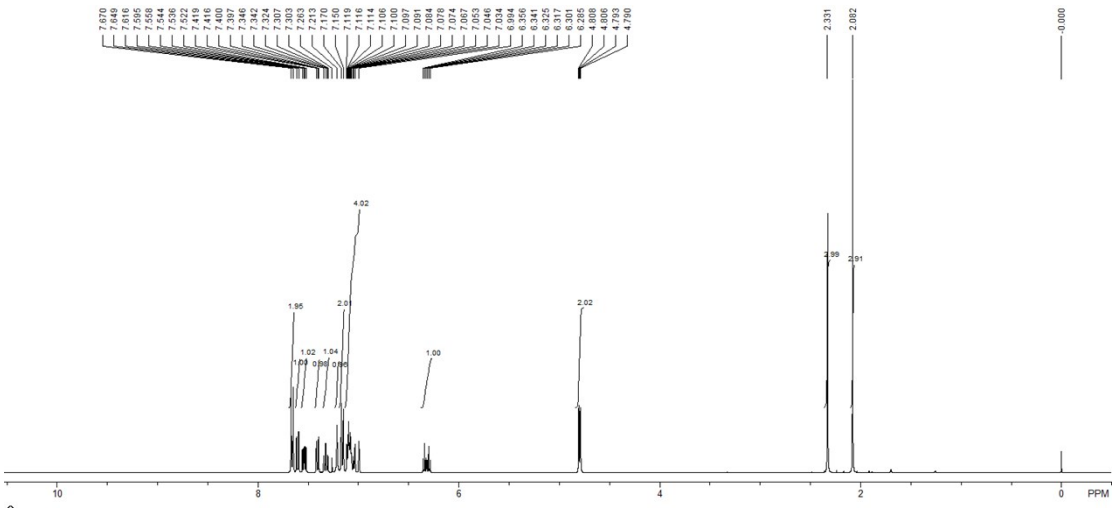
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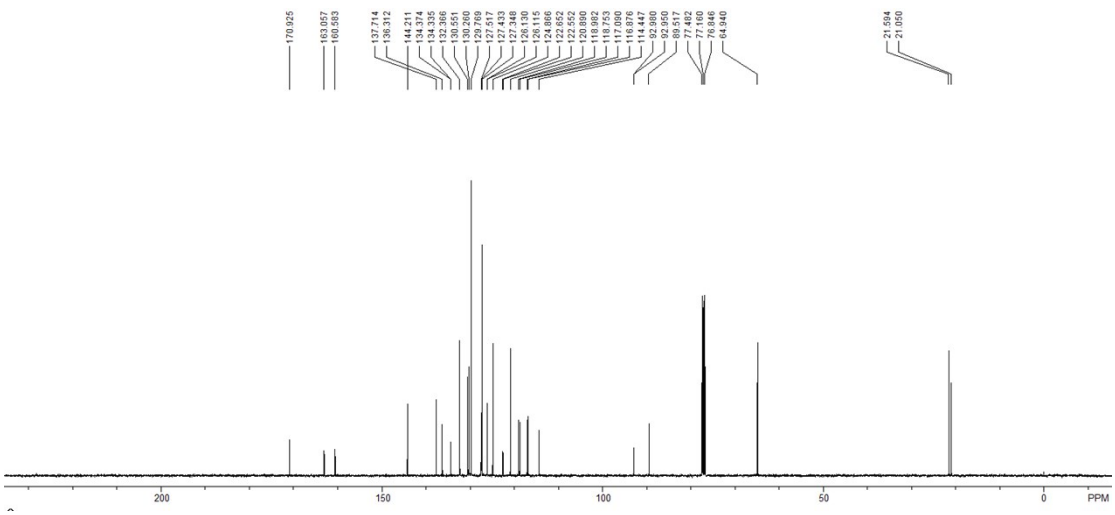
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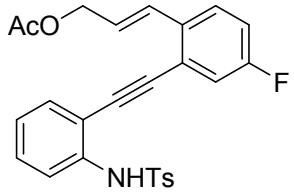
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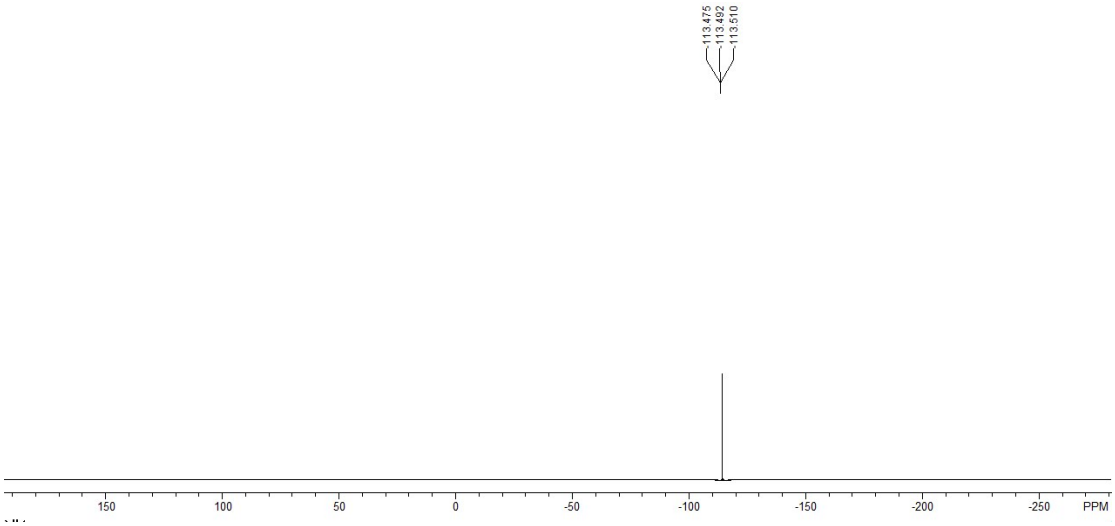
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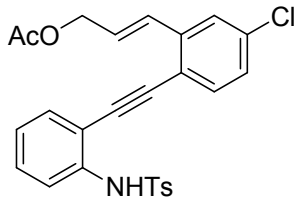
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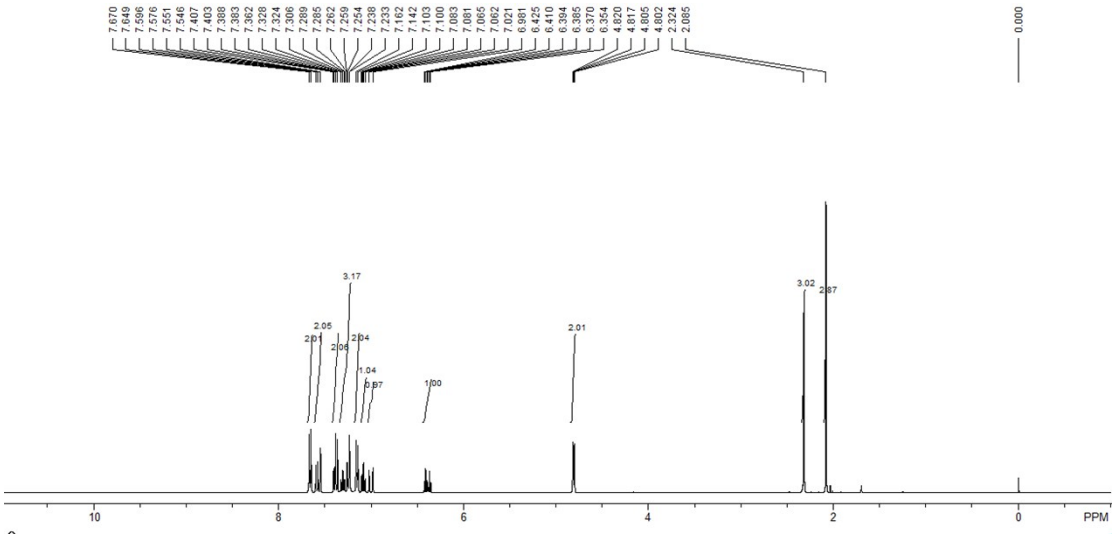
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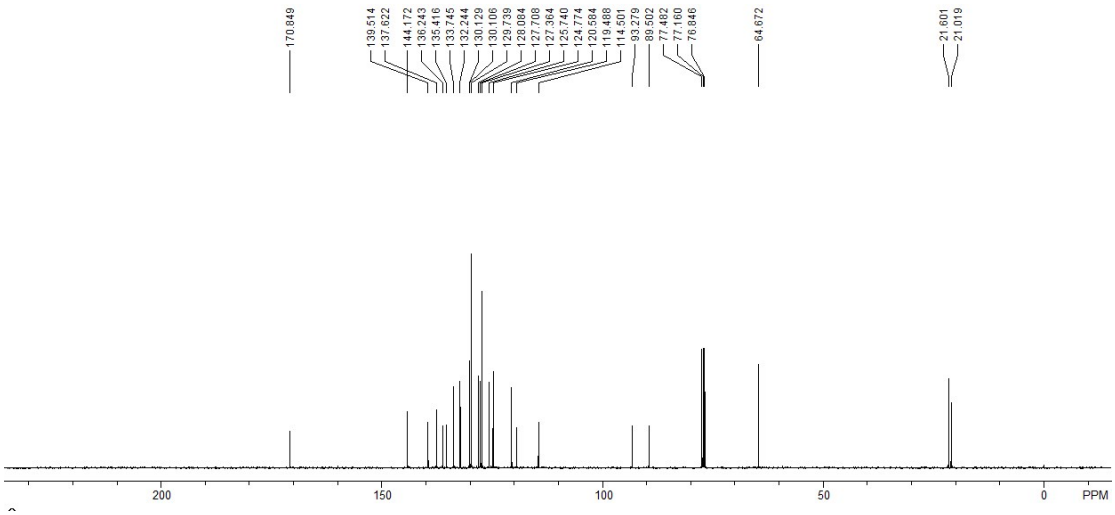
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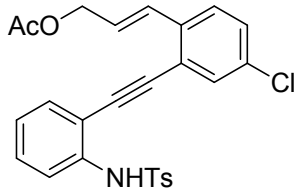
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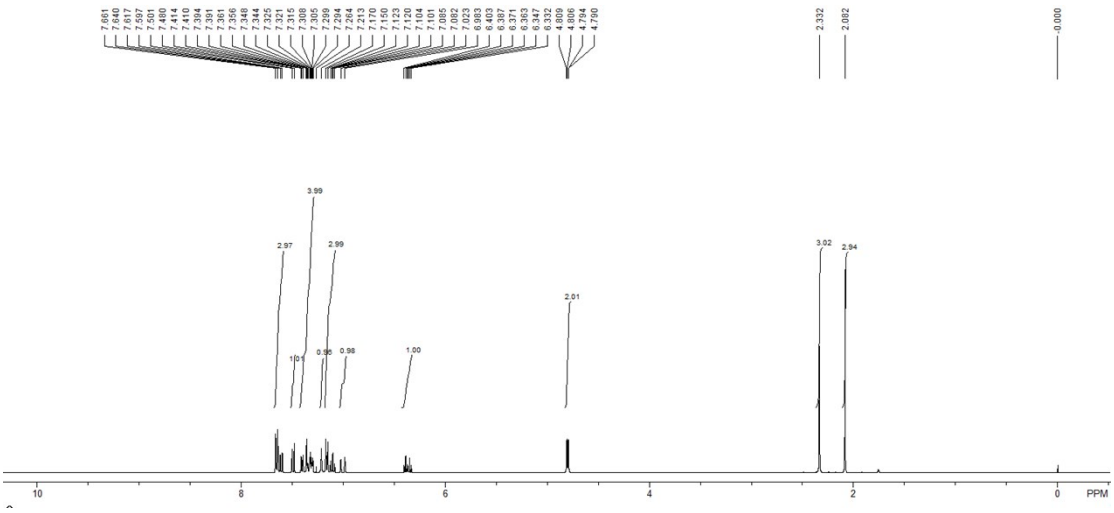
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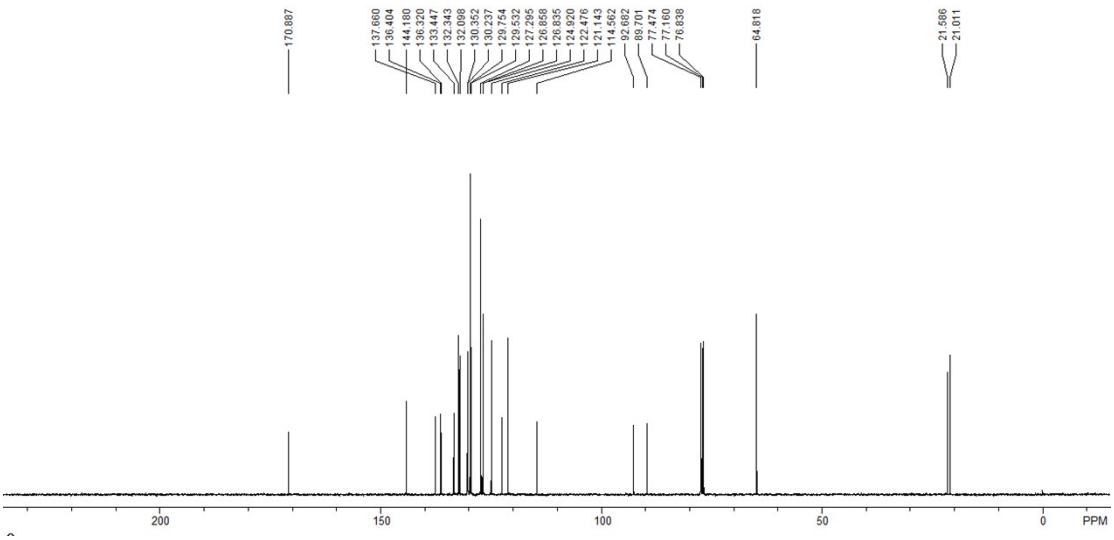
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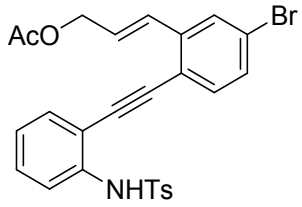
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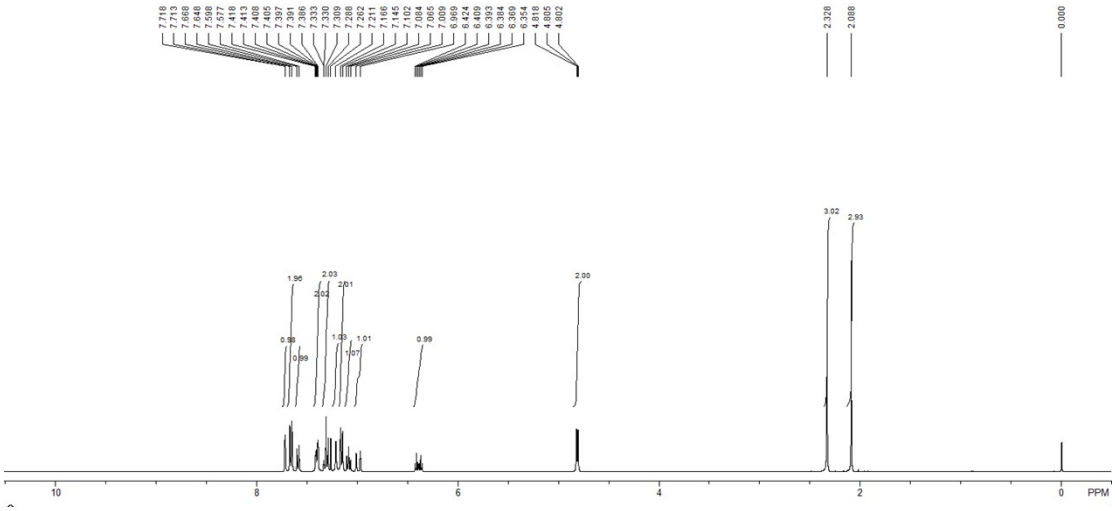
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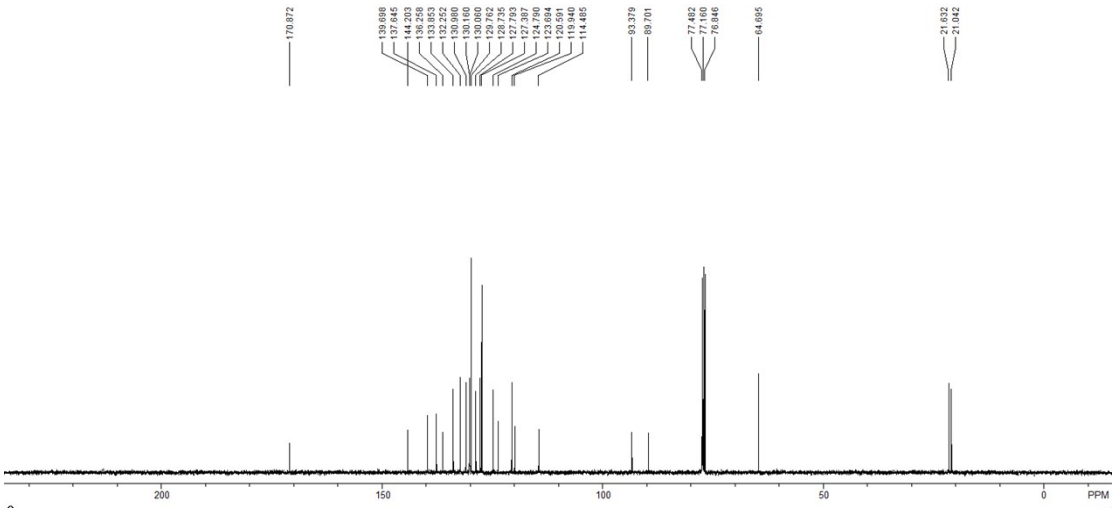
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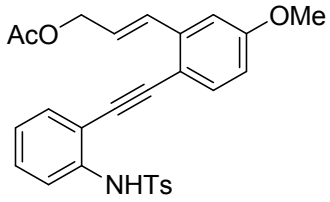
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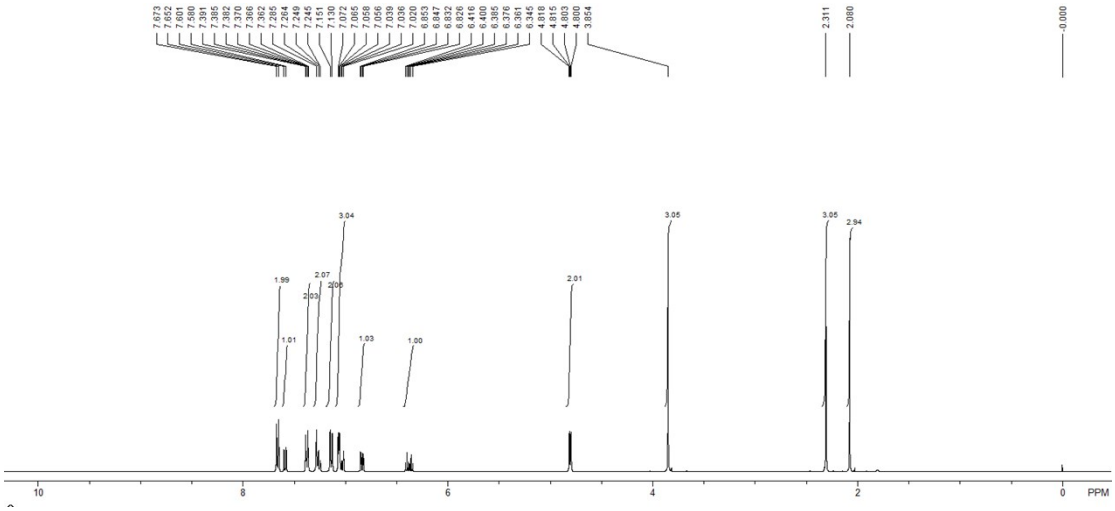
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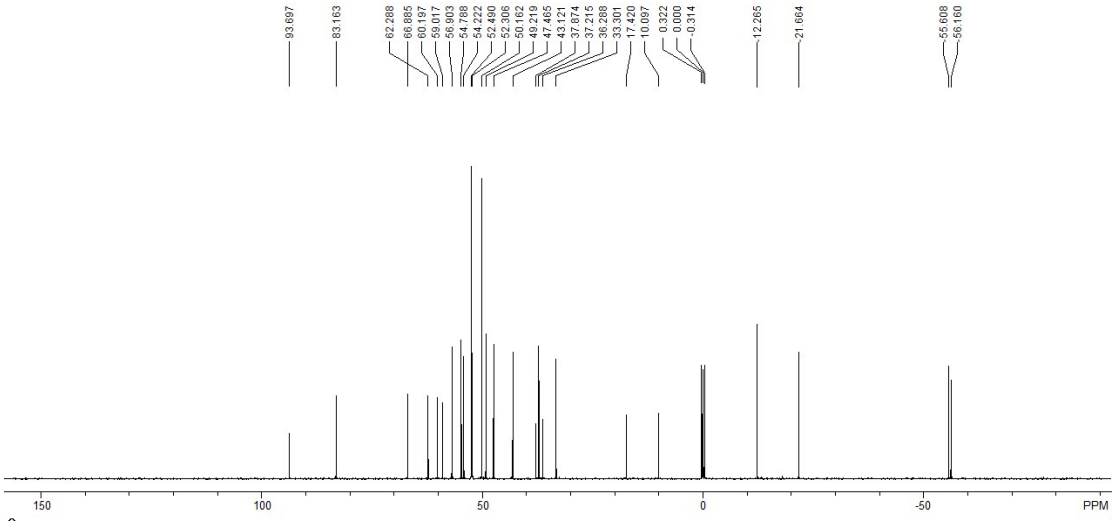
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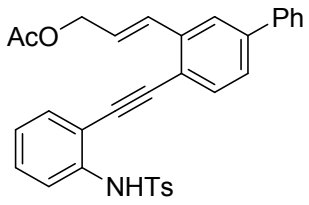
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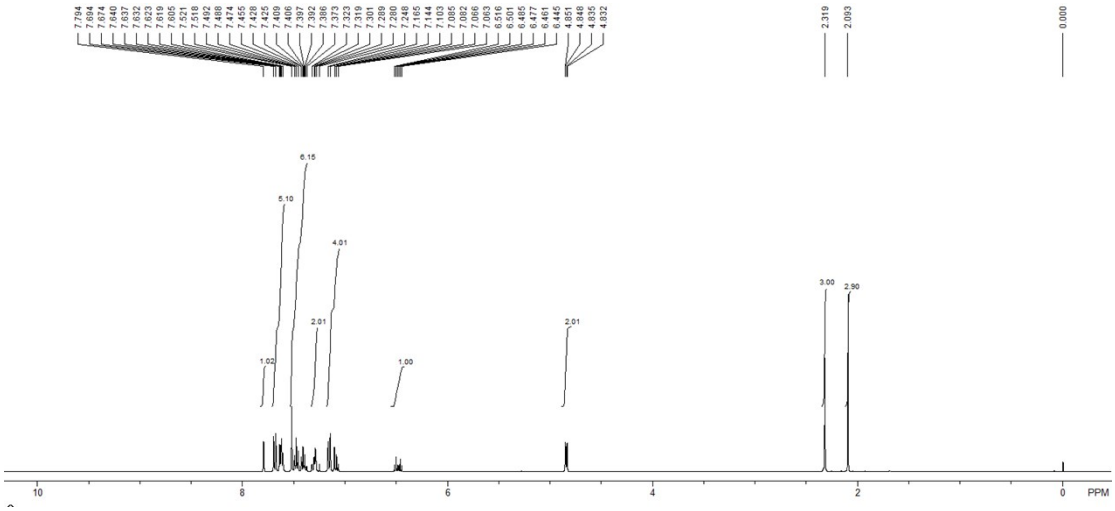
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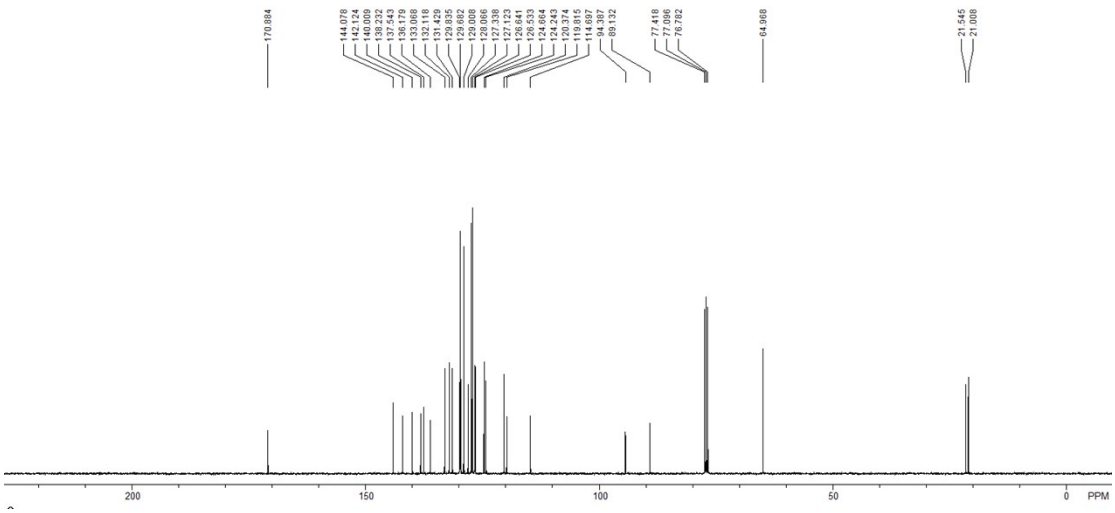
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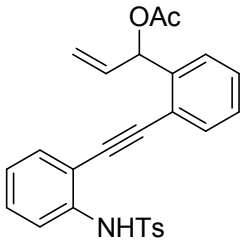
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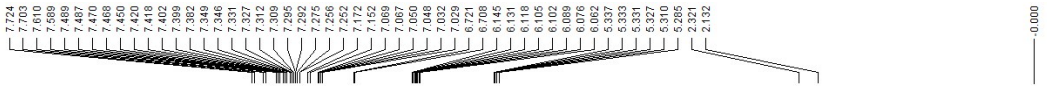
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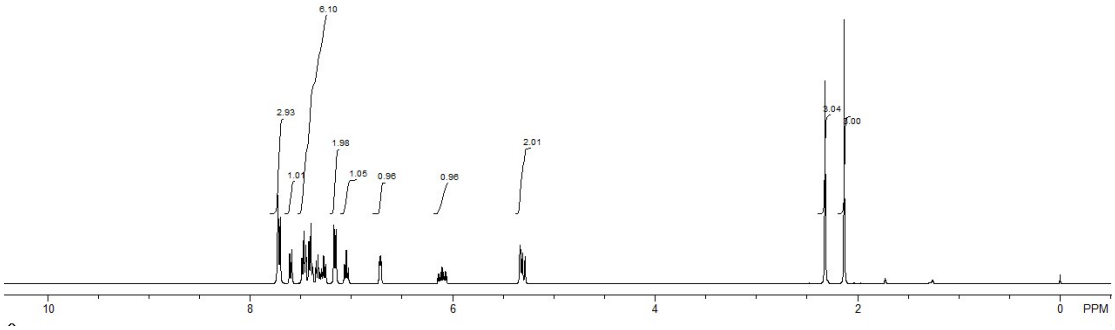
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2.01
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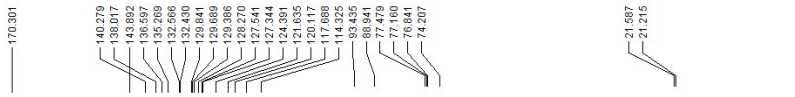
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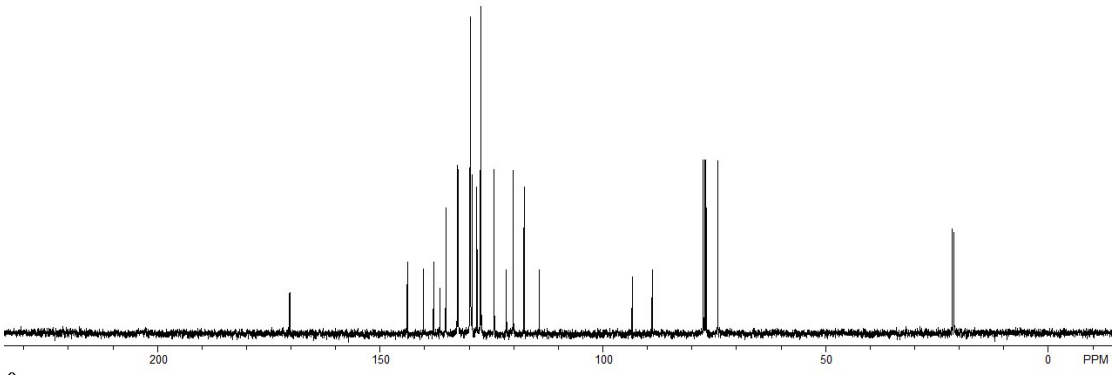
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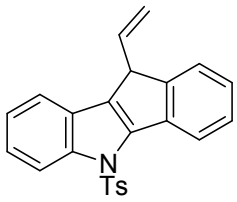
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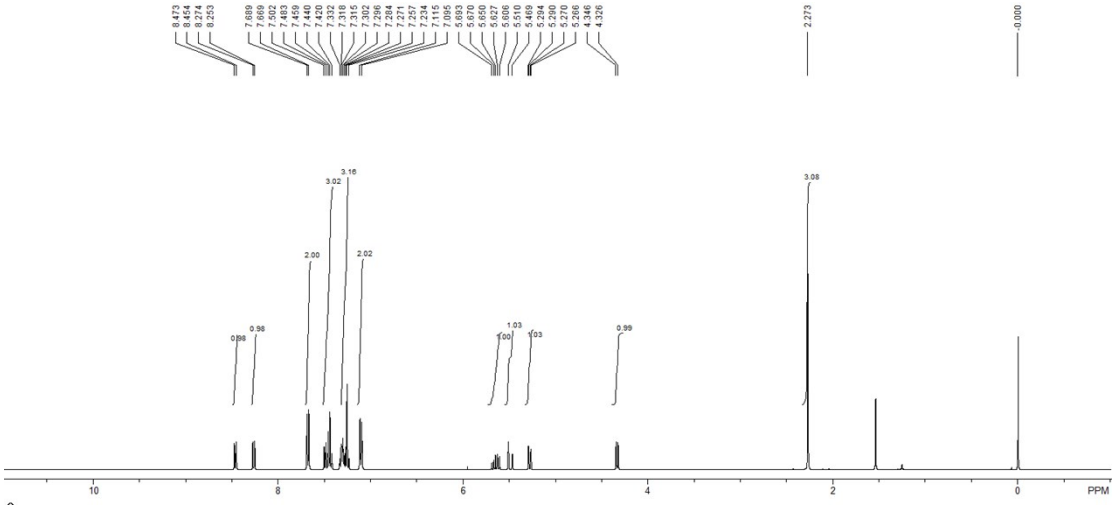
**2a**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7.155
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5.650
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5.294
5.290
5.270
5.266
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4.326
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2.00
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1.03
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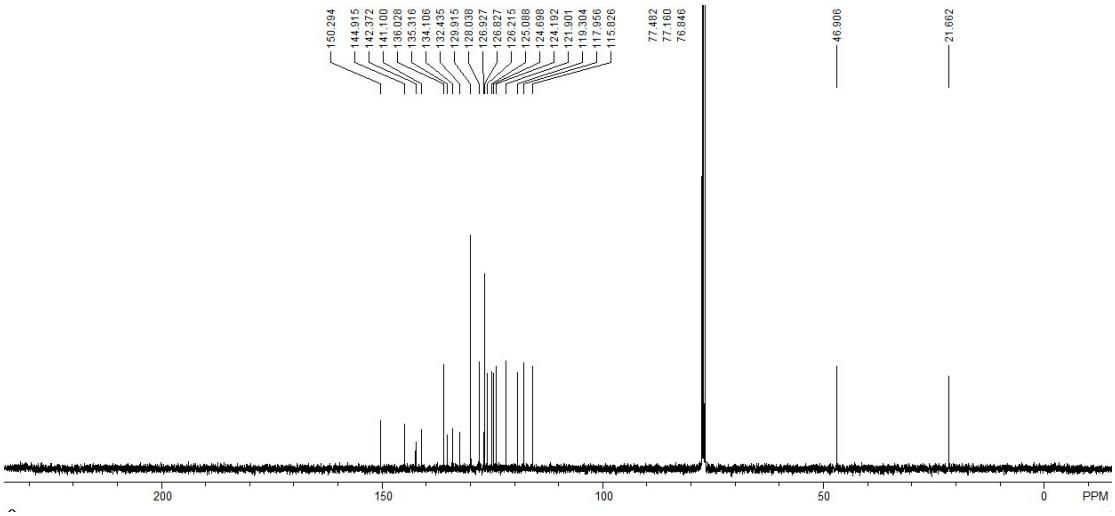
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126.827
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124.698
124.192
121.901
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77.160
76.846
46.906
21.662
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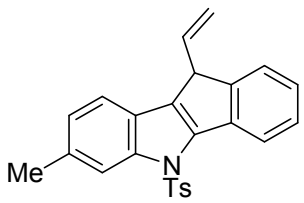
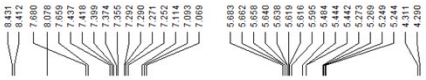

Me
N
Ts

**2b**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7.355
7.292
7.290
7.271
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5.658
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5.638
5.619
5.616
5.595
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5.244
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4.290
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2.271
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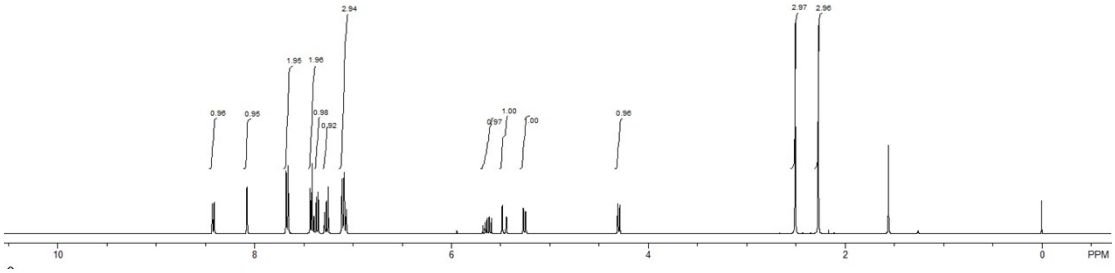
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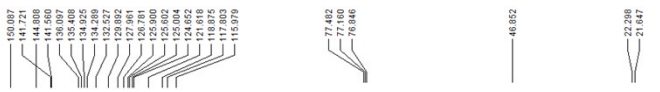
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126.600
125.602
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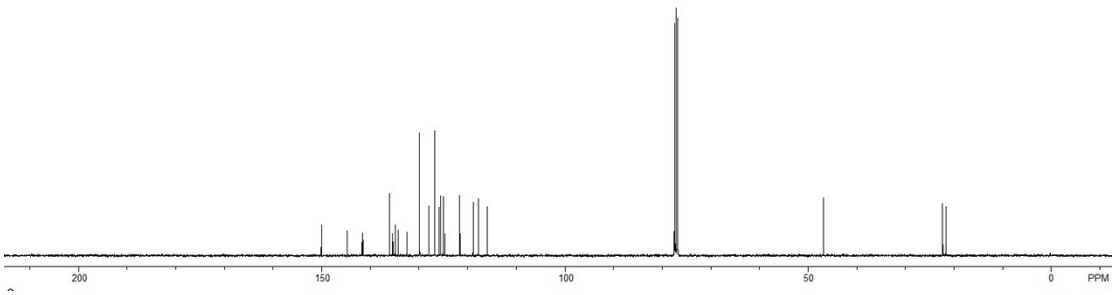
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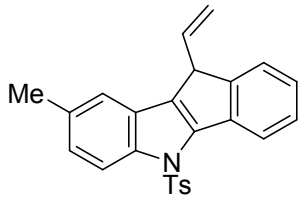
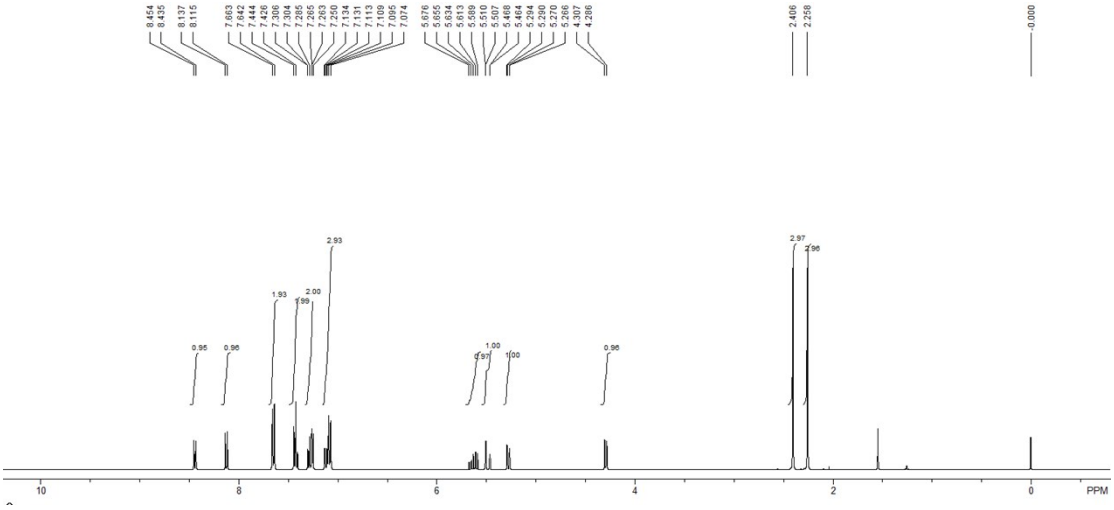

Me
N
Ts

**2c**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5.507
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5.270
5.266
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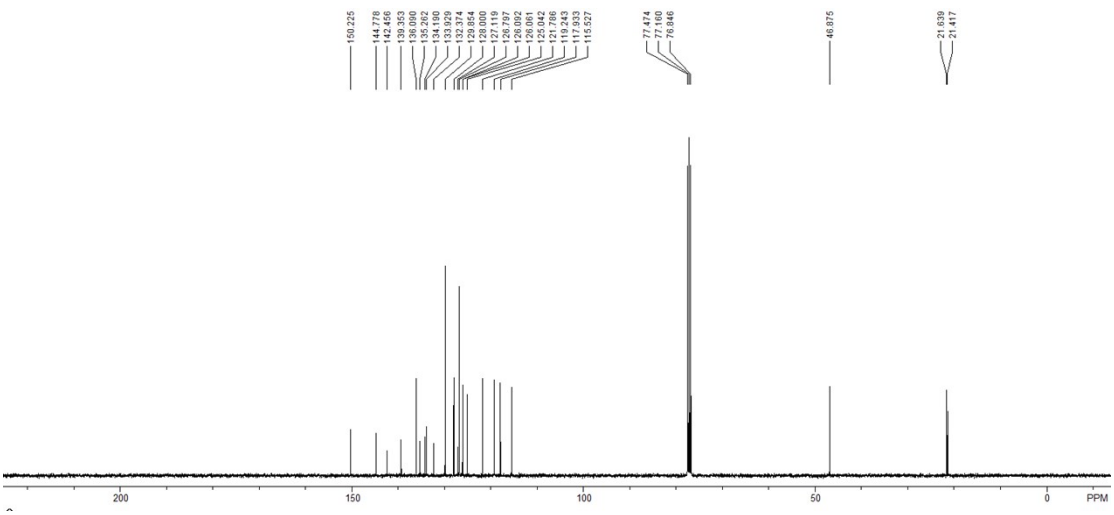
PPM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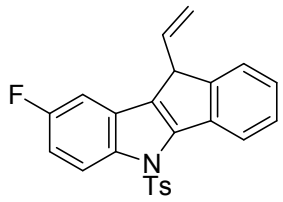
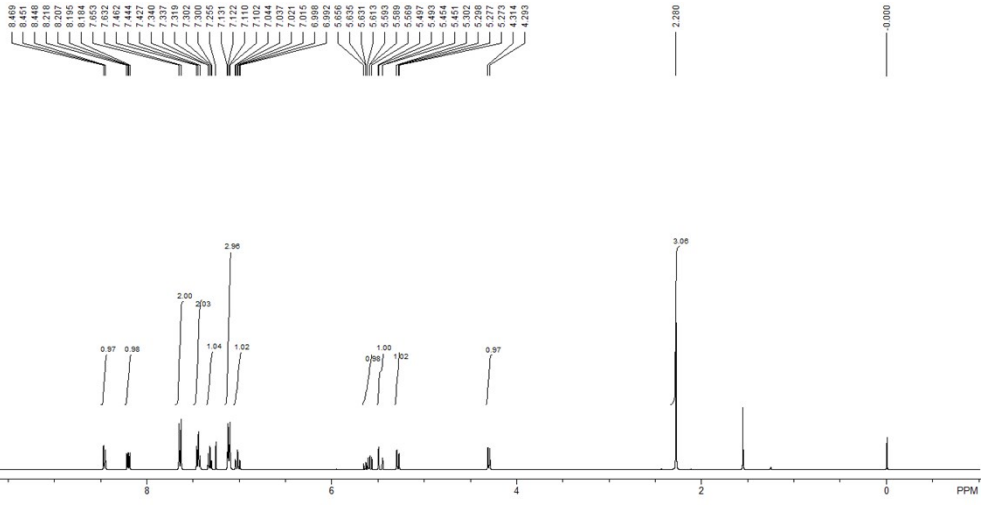

F
N
Ts

**2d**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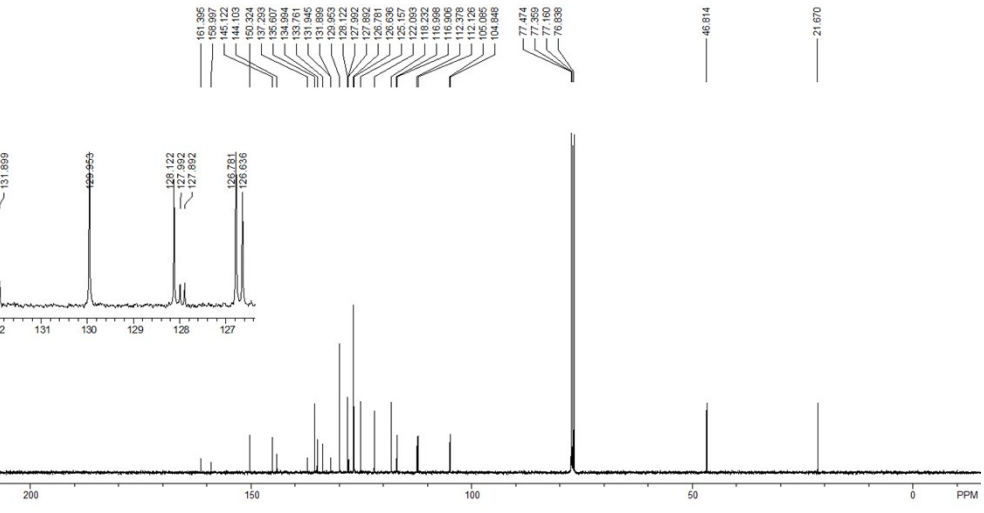
PPM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127.992
127.892
126.781
126.636
125.157
122.093
118.232
116.998
116.906
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112.139
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76.838
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21.670
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129.953
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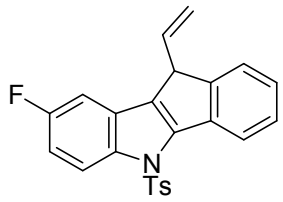
PPM

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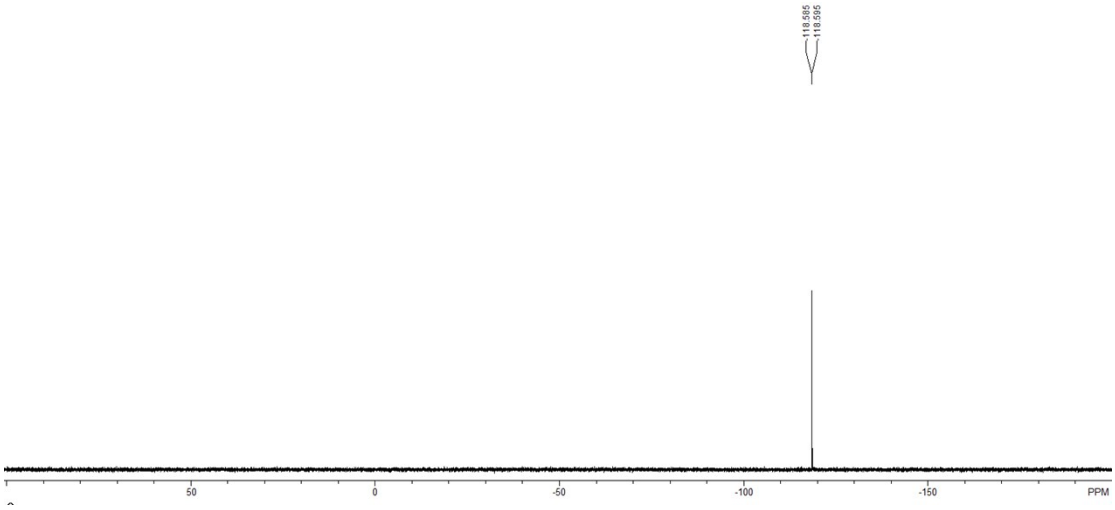
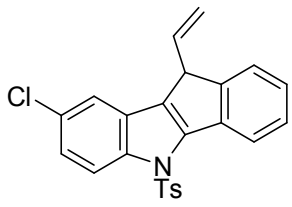
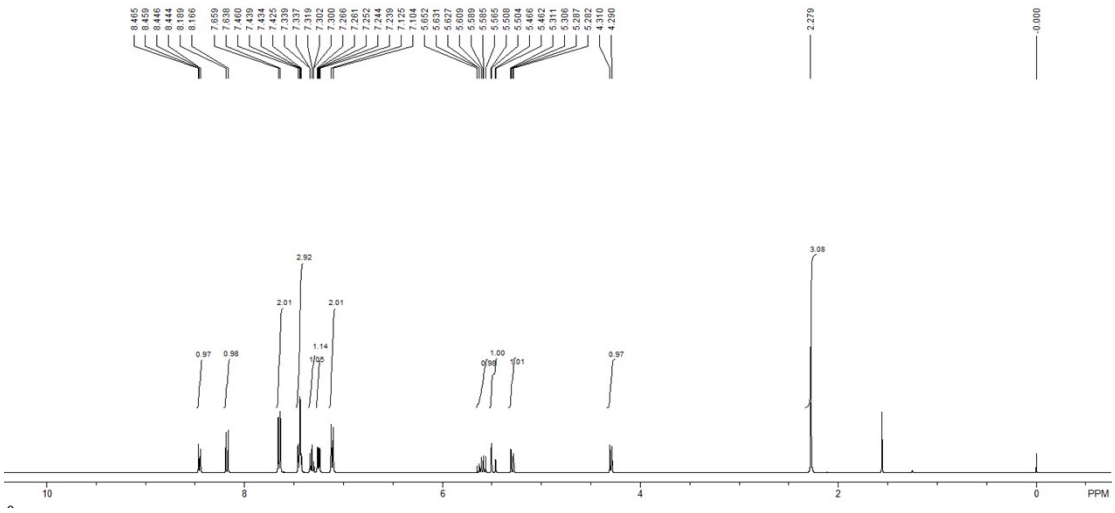



Cl
N
Ts

**2e**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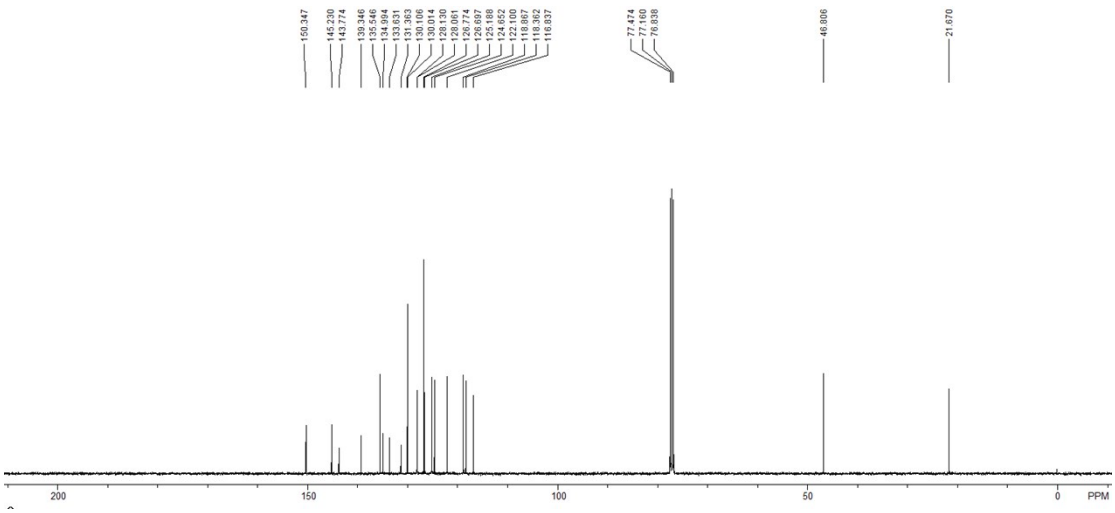
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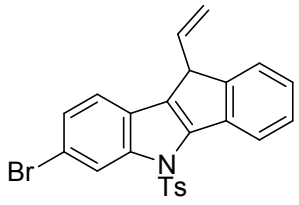
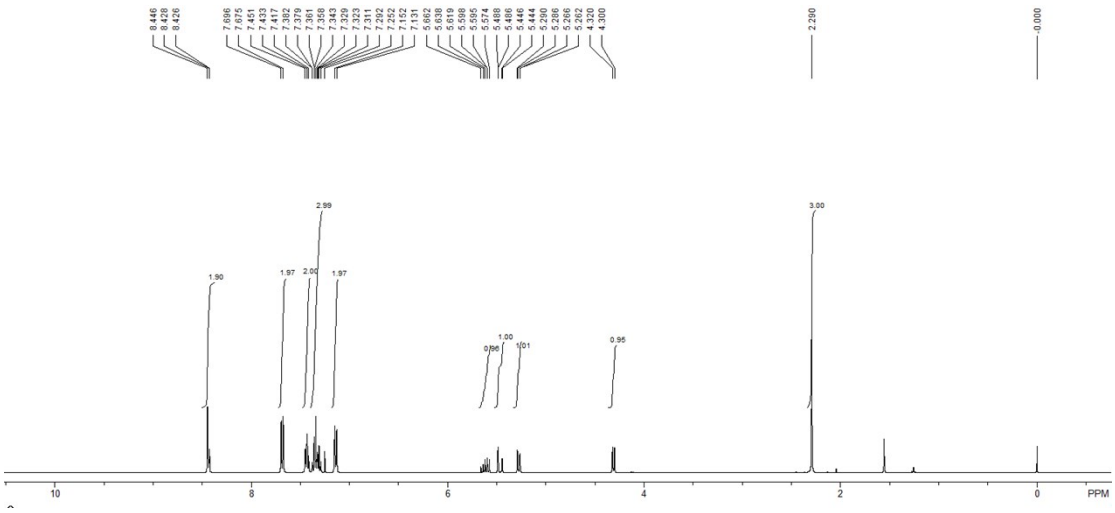

Br
N
Ts

**2f**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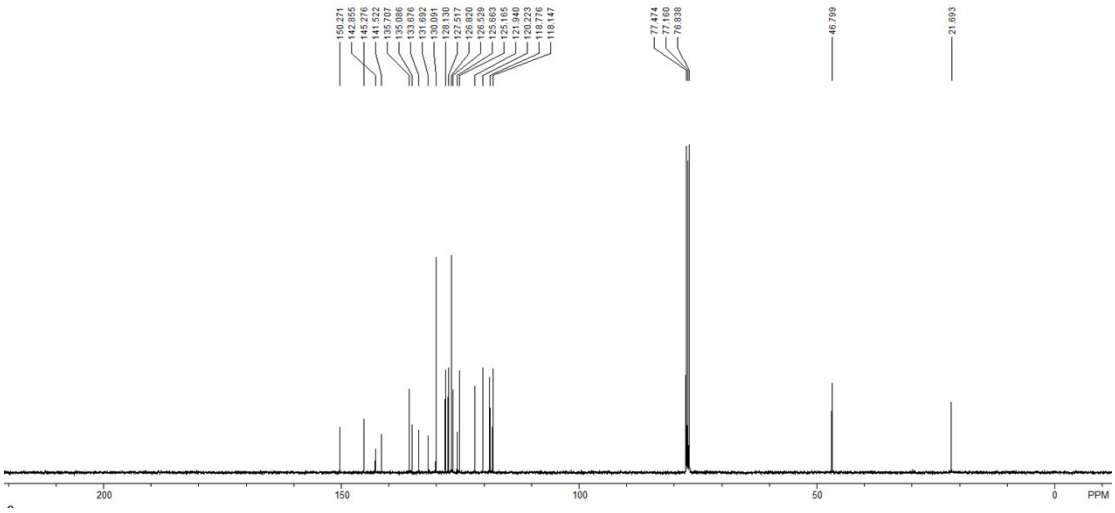
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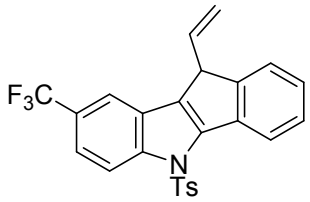
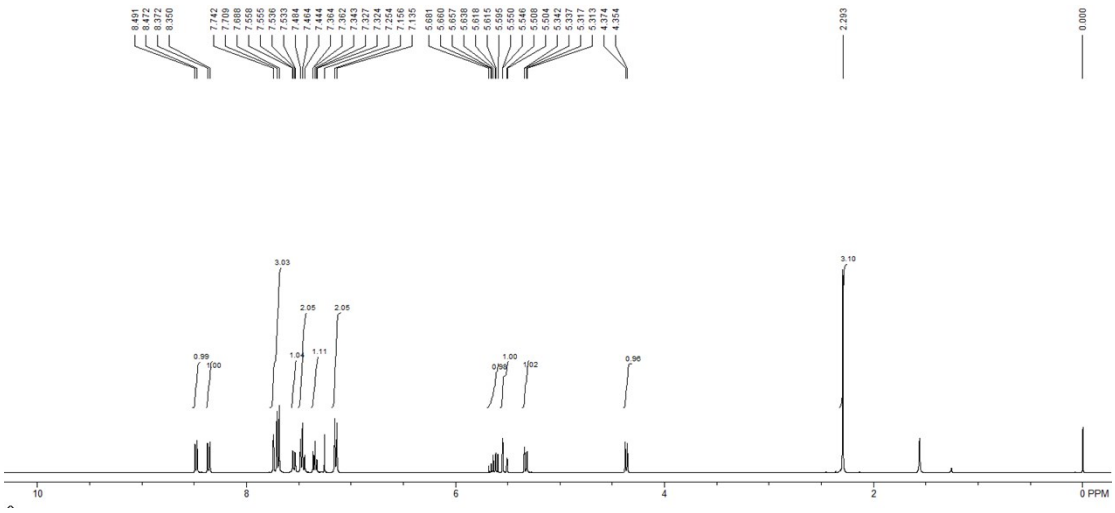

F3C
N
Ts

**2g**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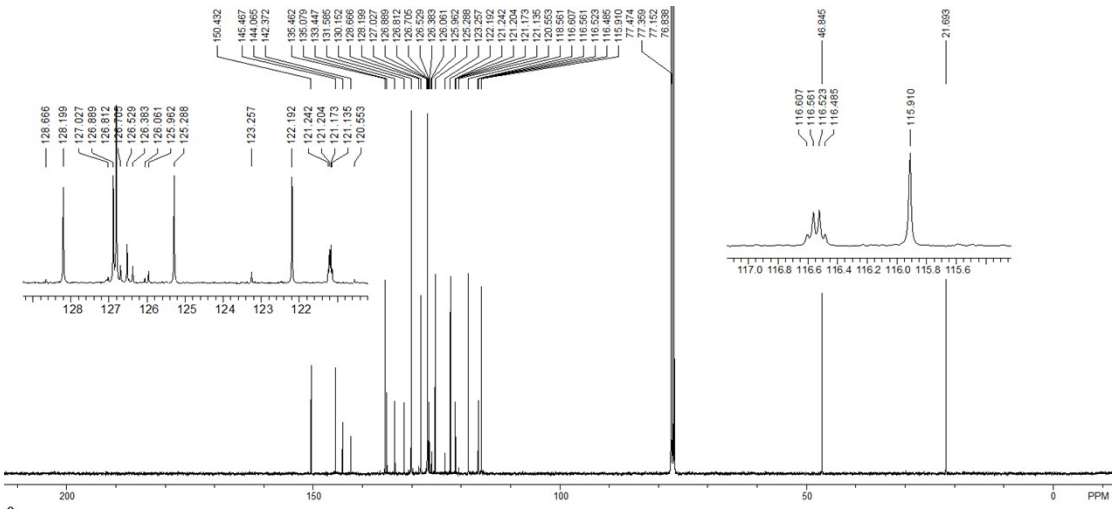
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116.607
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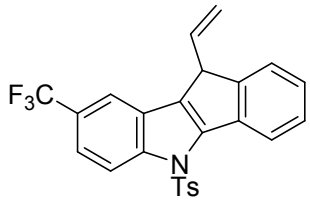


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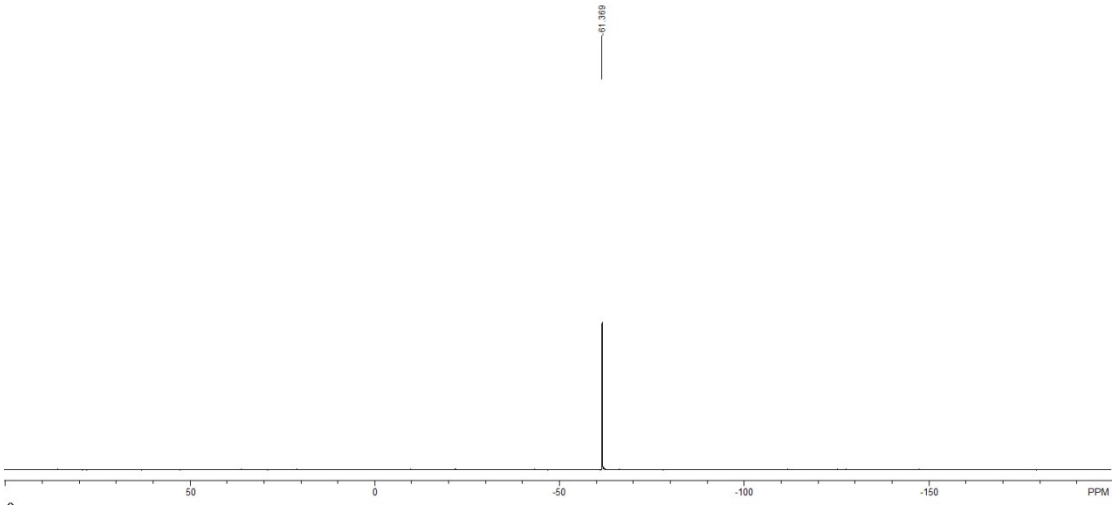
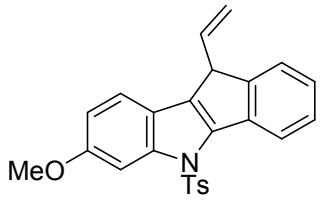
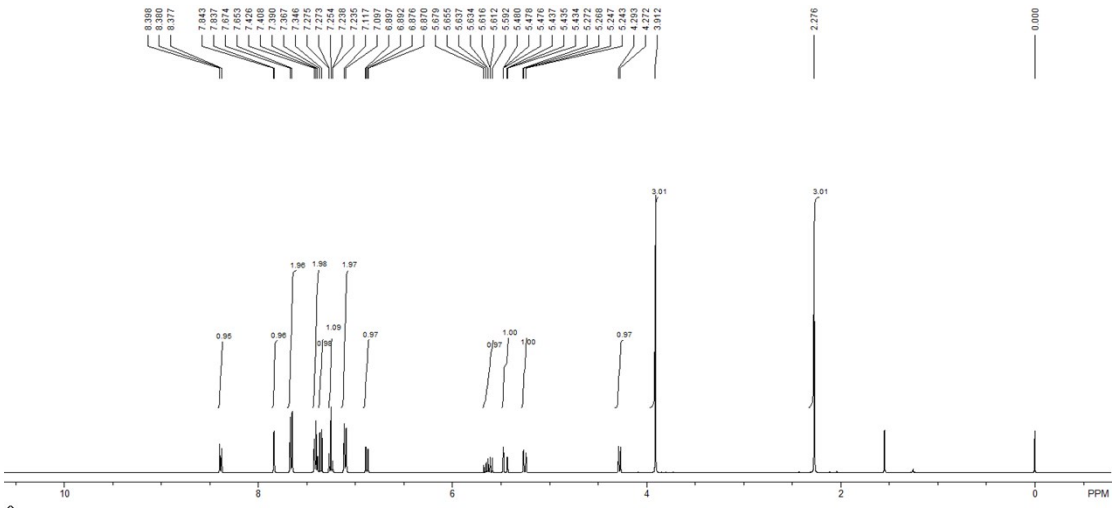



MeO
N
Ts

**2h**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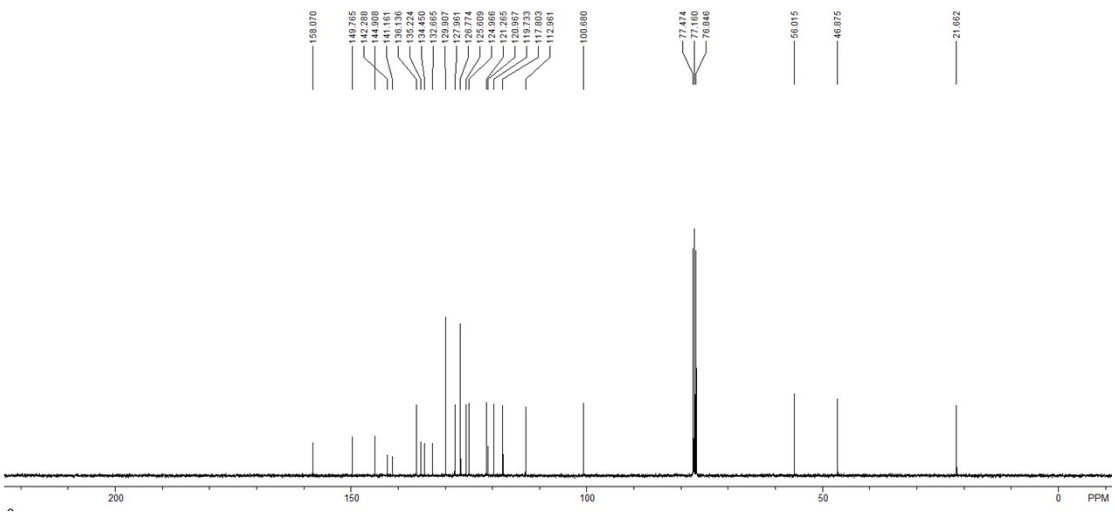
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129.907
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124.966
121.265
120.967
119.733
117.803
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77.160
76.846
56.015
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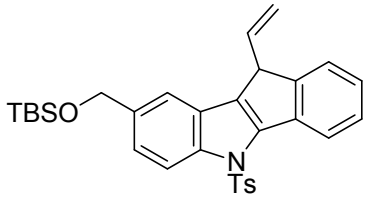
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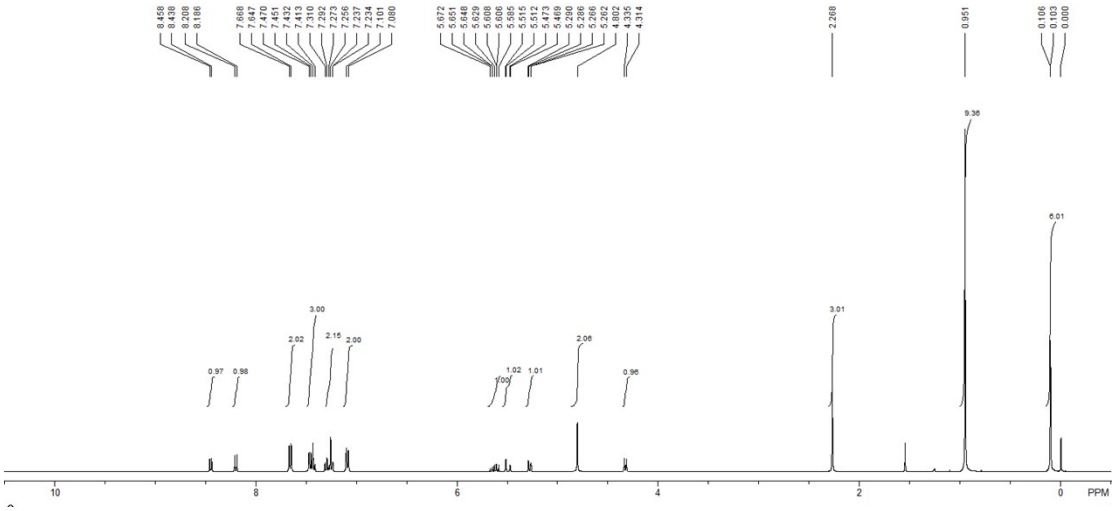
**2i**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7.237
7.234
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5.648
5.629
5.608
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5.585
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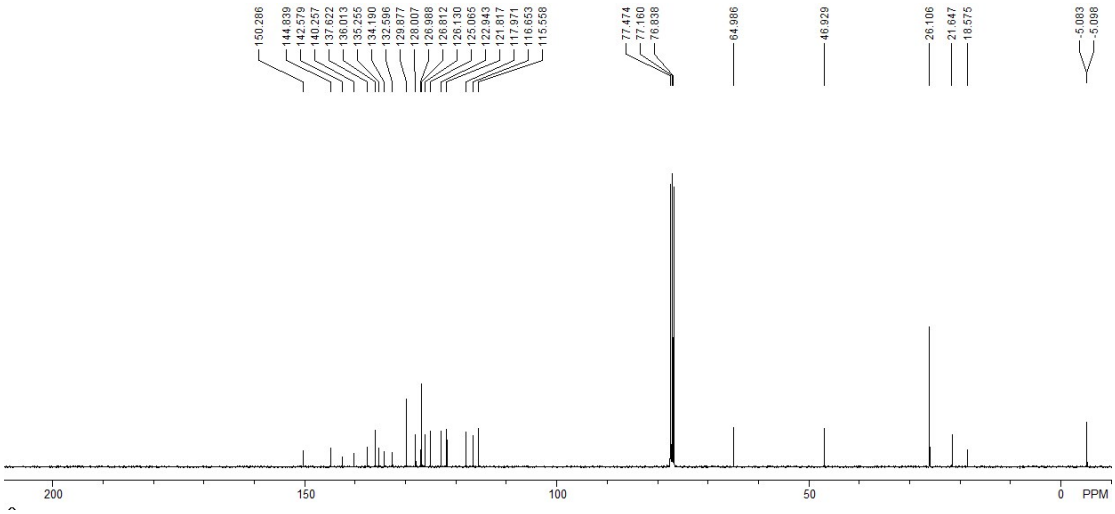
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77.160
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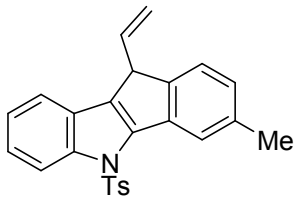
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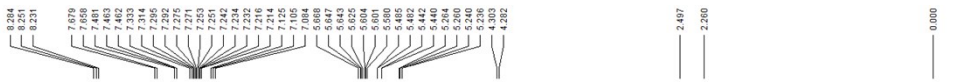
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7.292
7.275
7.271
7.253
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7.234
7.232
7.216
7.214
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7.105
7.084
5.668
5.647
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5.604
5.601
5.580
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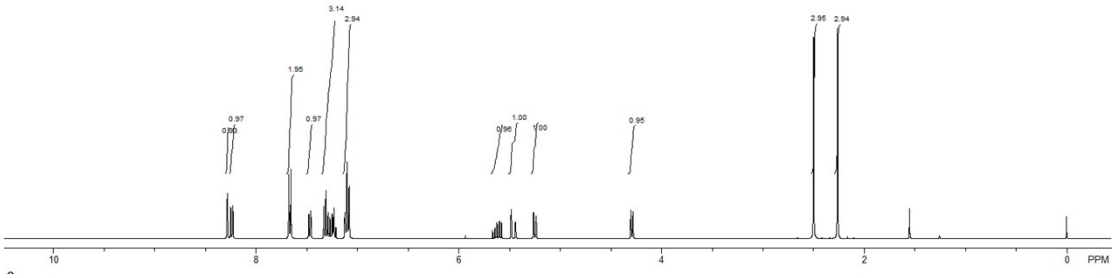
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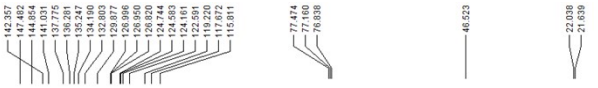
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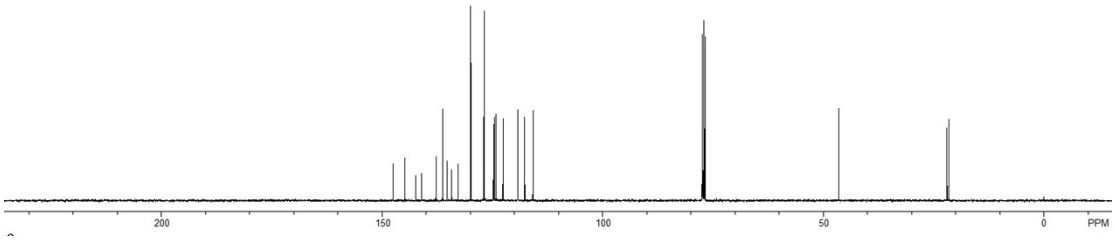
22.038
21.639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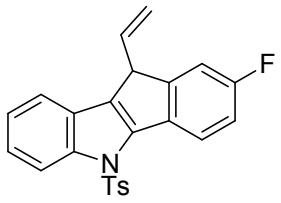
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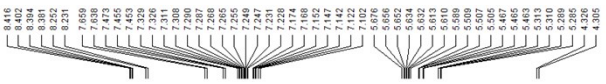
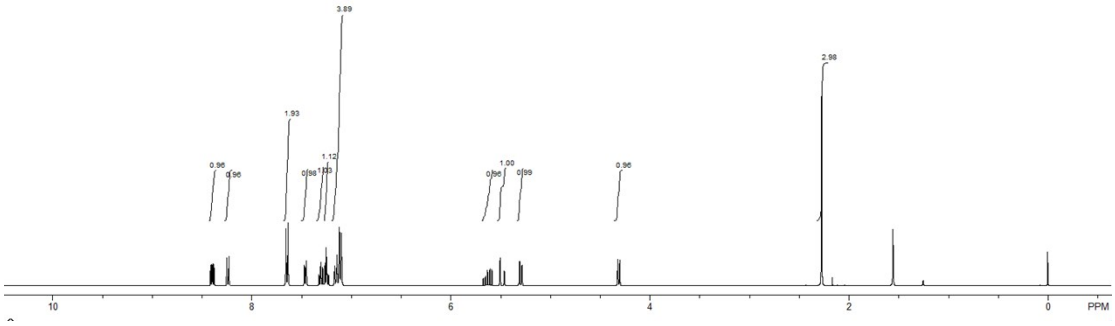
Ts

**2k**

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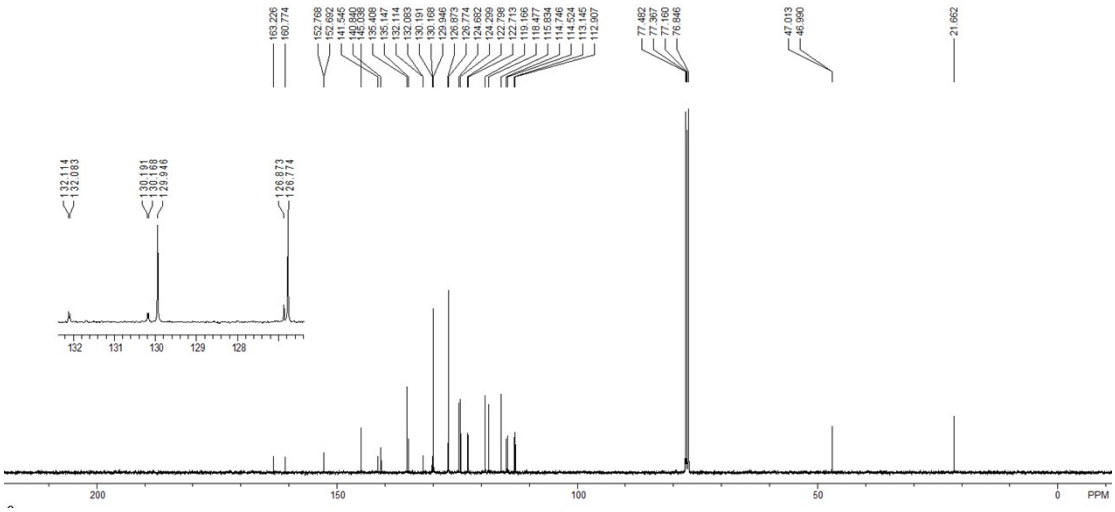
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130.168
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126.873
126.774
124.299
122.798
122.713
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118.477
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114.524
113.145
112.907
77.482
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77.160
76.846
47.013
46.980
21.662
132.114
132.083
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100
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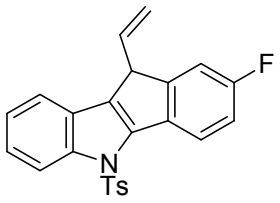
PPM

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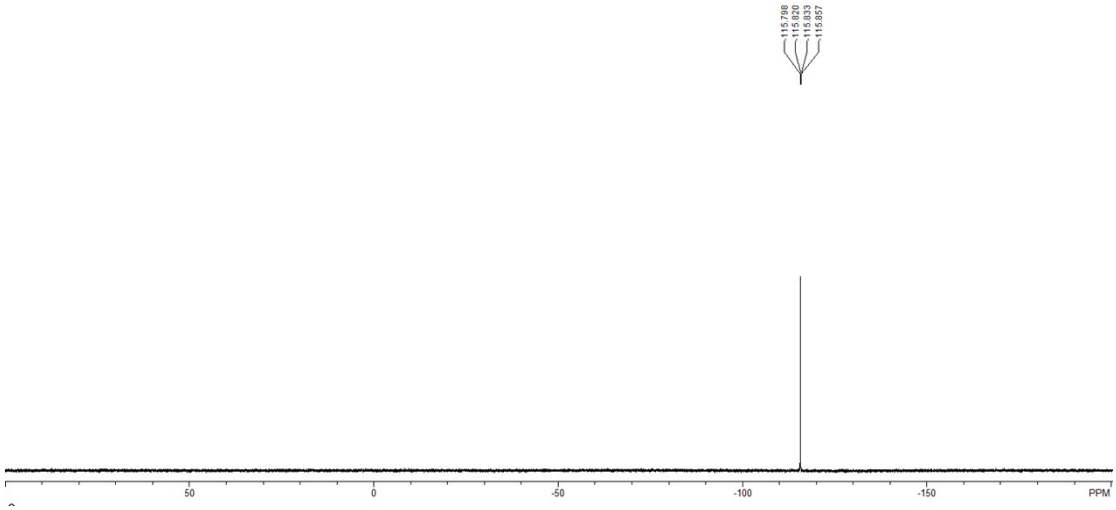
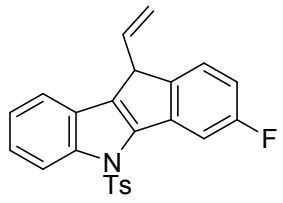
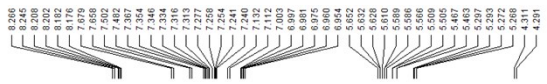
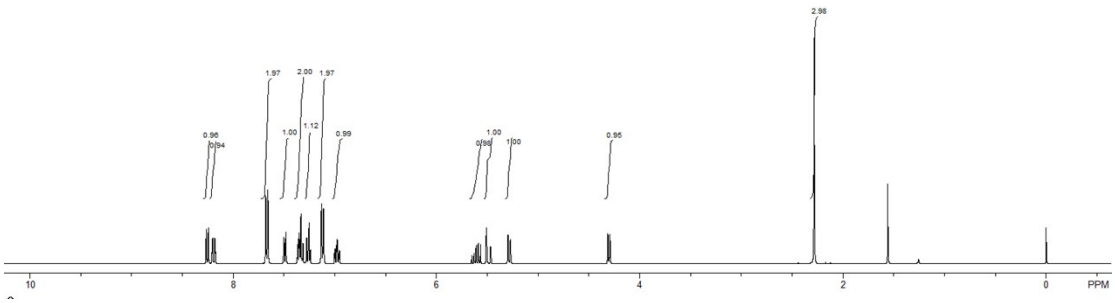



F
N
Ts

**2l**

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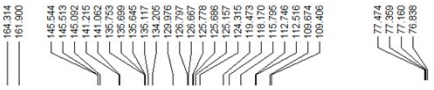


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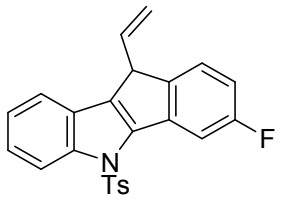


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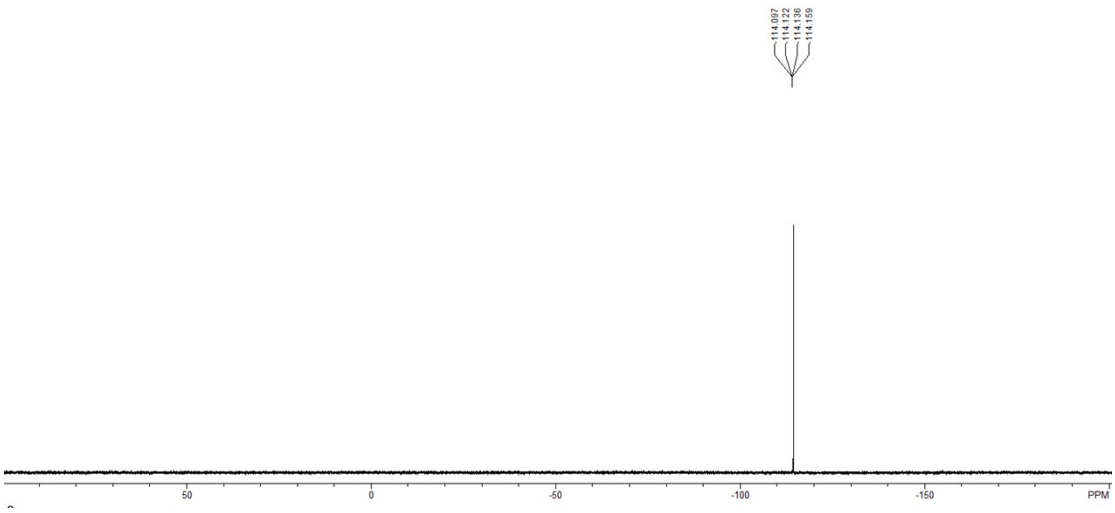

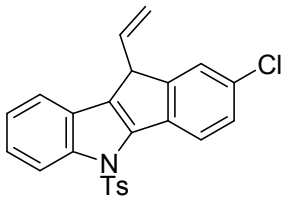


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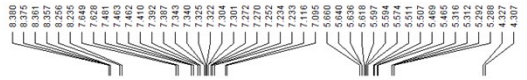

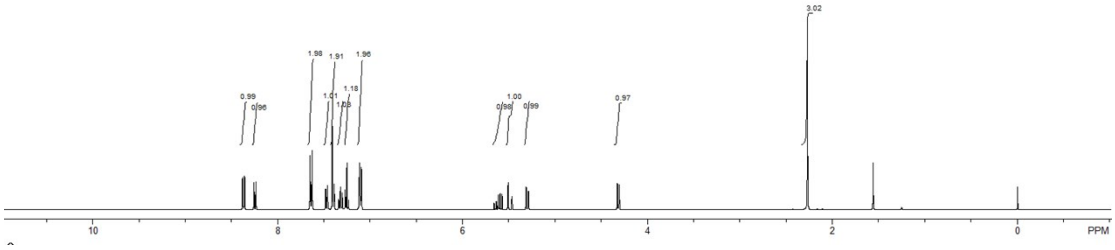

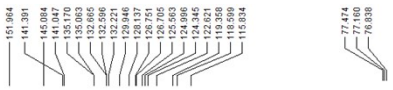

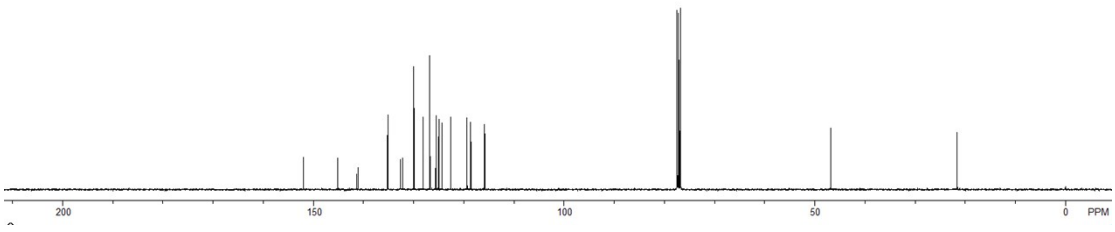

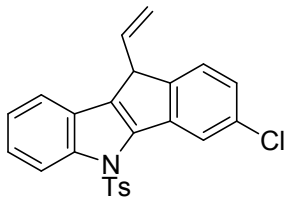
Cl
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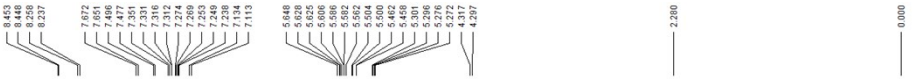
Ts

**2n**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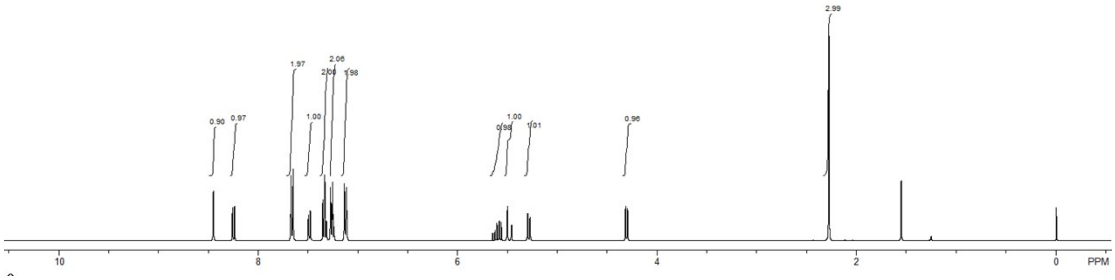
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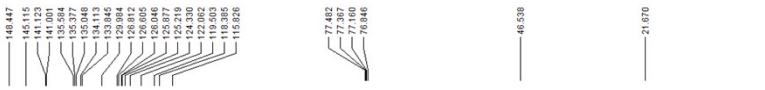
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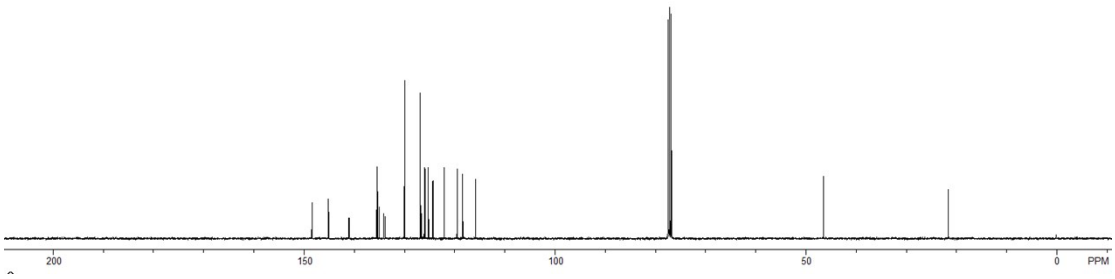
21.670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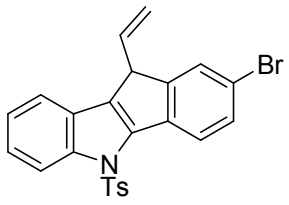
Br
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Ts

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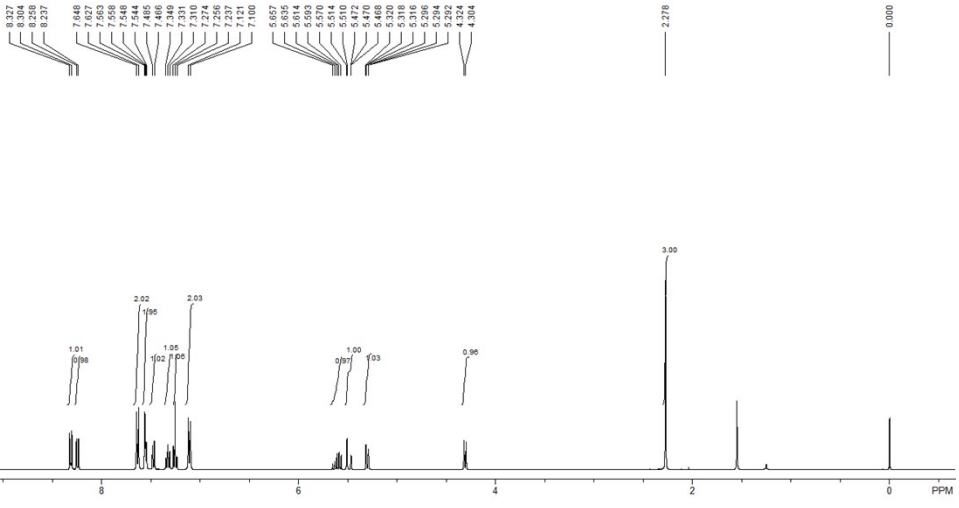
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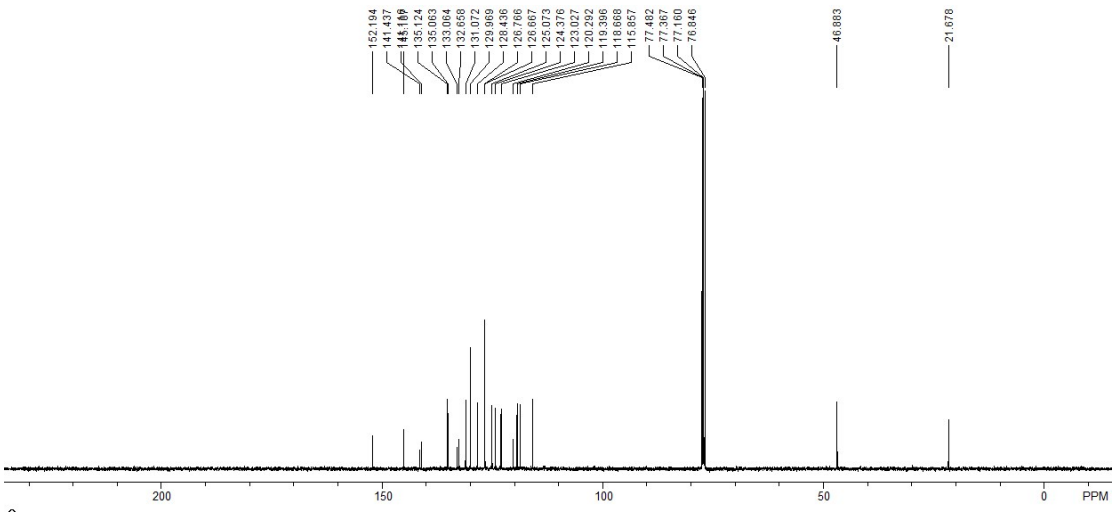
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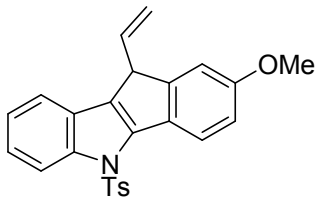
OMe
N
Ts

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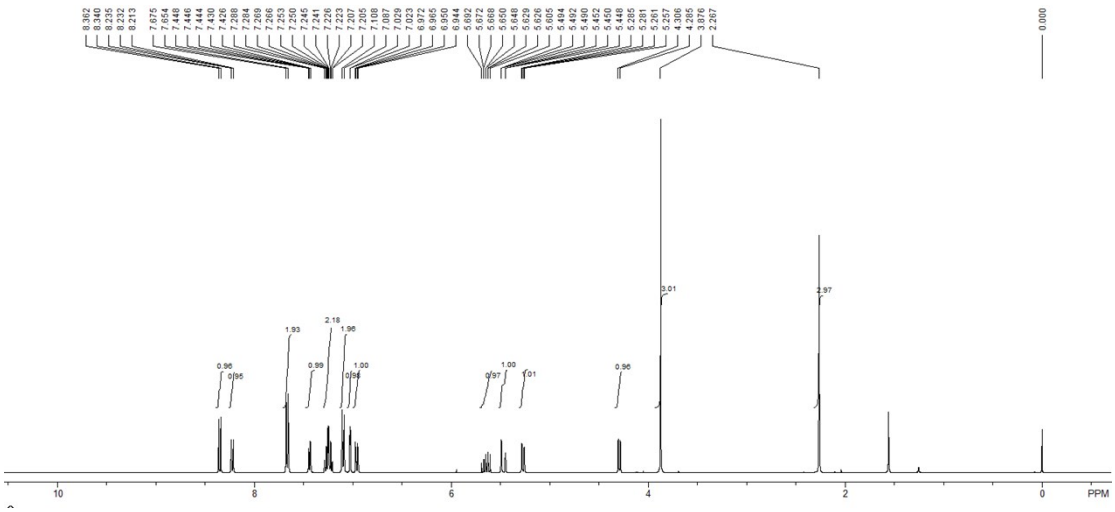
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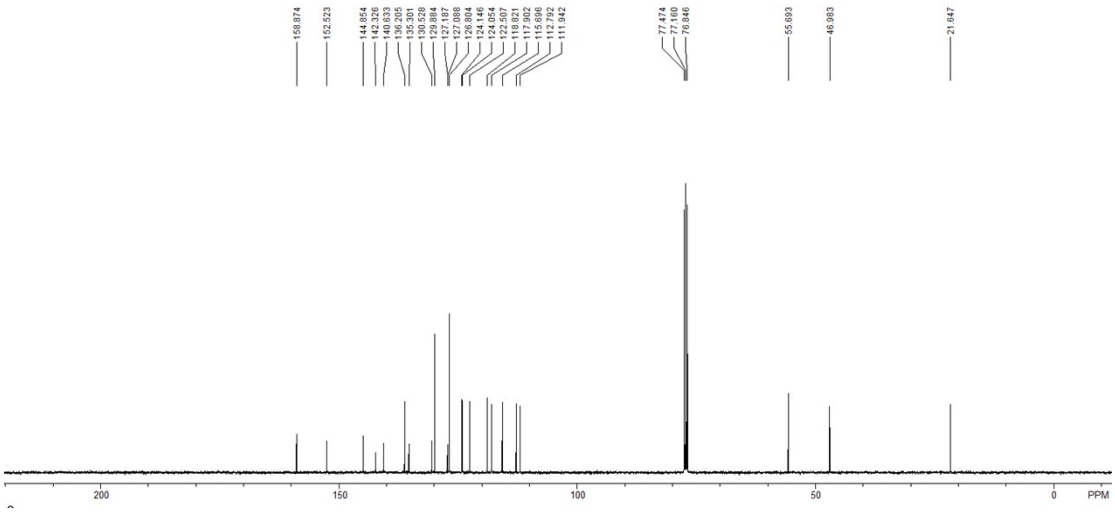
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117.902
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55.693
46.963
21.647
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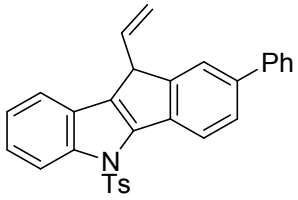
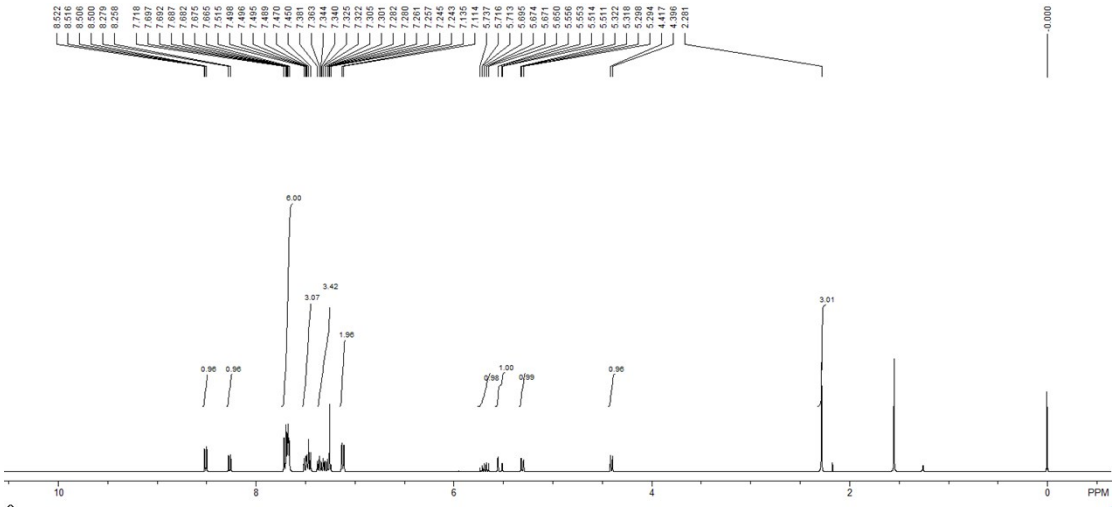
50
0
PPM


Ph
N
Ts

**2q**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7.381
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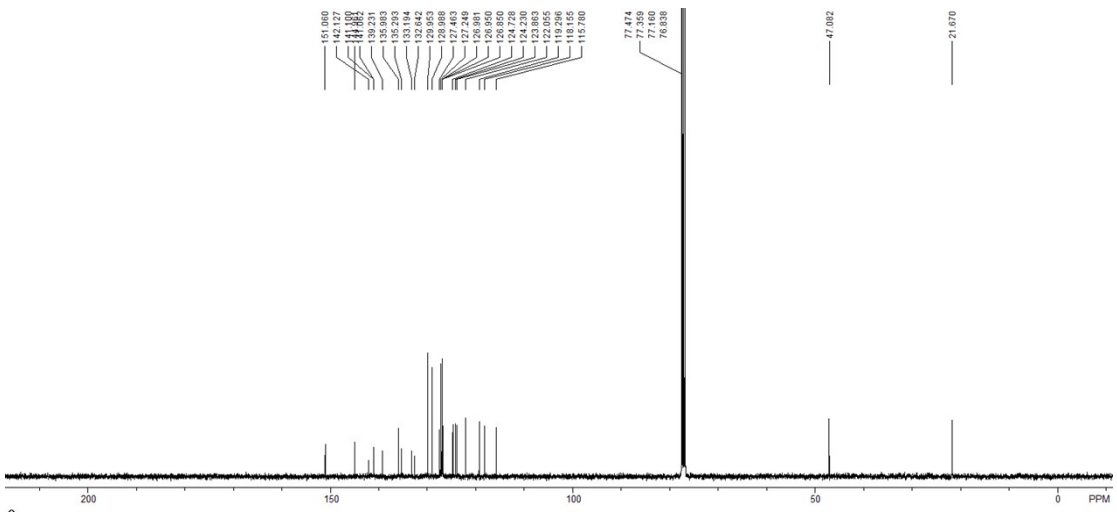
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