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Supporting Information

Visible-Light-Driven Graphitic Carbon Nitride-Catalyzed ATRA of Alkynes: Highly Regio- and Stereoselective Synthesis of (*E*)-*β*-Functionalized Vinylsulfones

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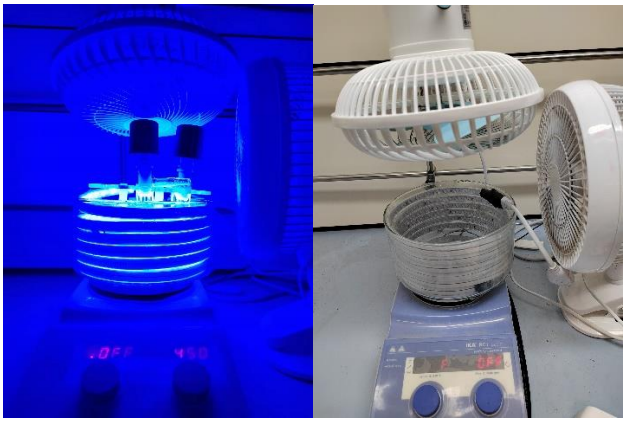
Contents

1. General information

Reagents were used as received from commercial suppliers without further purification, unless otherwise stated. NMR spectra were recorded for ^{1}H NMR (400 MHz and 600 MHz), ^{13}C NMR (100 MHz and 150 MHz) and ^{19}F NMR (376 and 565 MHz) using TMS as an internal standard and Bruker AV 400 and 600 as an instrument. The following abbreviations were used to describe peak patterns where appropriate: singlet (s), doublet (d), triplet (t), multiplet (m). High-resolution mass spectroscopy (HRMS) were obtained using Bruker Apex IV RTMS. The surface morphology and fine structures were observed by transmission electron microscope (TEM, Titan G260-300). The X-ray photoelectron spectroscopy (XPS) signals were obtained by a thermo K-Alpha spectrometer with an Al Ka X-ray source (1486.6 eV). The binding energy was calibrated using the C1s level at 284.6 eV as an internal standard. IR spectra were recorded with a FT-IR Bruker EQUINOX55 spectrometer in KBr pellets. The X-ray diffraction data was collected on a XtaLab PRO MM007HF-DW diffractometer. The surface morphology and structure were observed by field-emission scanning electron microscope (FE-SEM, Gemini SEM 300).

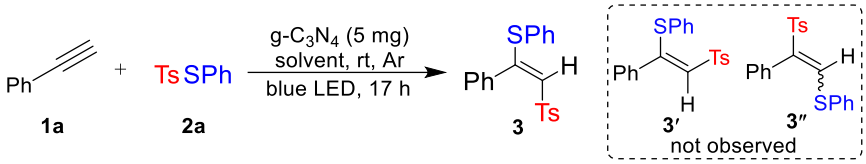
Reaction set-up for the photocatalytic reactions with blue LED



LED strips: 7 W/m *5 m was purchased from www. taobao. com.

2. Screening of reaction conditions

Table S1 Screening of different solvents[a]

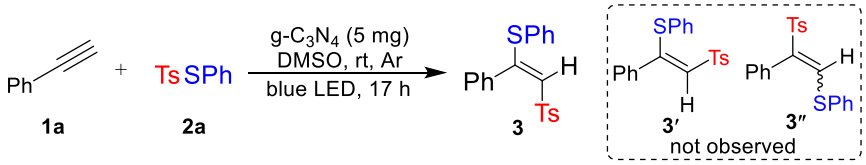


1a 2a 3 3′ 3″

Entry	Solvent	Isolated yield (%)
1	toluene	NR
2	CF_3Ph	NR
3	DME	45
4	THF	67
5	1,4-dioxane	75
6	CH_3CN	trace
7	CH_2Cl_2	73
8	DCE	47
9	EtOAc	NR
10	dimethyl carbonate	55
11	diethyl carbonate	NR
12	DMF	88
13	**DMSO**	**89**
14	acetone	86

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), g-C_3N_4 (5 mg) in solvent (1 mL) at room temperature under blue LED (7 W) irradiation for 17 h under argon. NR = not reaction.

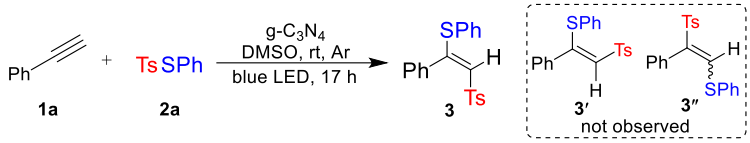
Table S2 Screening of reactant amount[a]



Entry	**1a** (equiv.)	**2a** (equiv.)	Isolated yield (%)
1	1	1	51
2	1	1.2	51
3	1	1.5	73
4	**1**	**2**	**89**
5	1	2.5	86
6	1.5	1	34

[a]Reaction conditions: **1a** (0.1 mmol), g-C_3N_4 (5 mg) in DMSO (1.0 mL) at rt under irradiation of blue LED (7 W) for 17 h under argon.

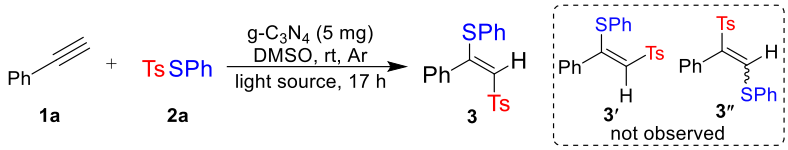
Table S3 Screening of catalyst loading[a]



Entry	g-C_3N_4	Isolated yield (%)
1	2 mg	32
2	3 mg	33
3	4 mg	79
4	**5 mg**	**89**
5	6 mg	85

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (2 equiv.) in DMSO (1.0 mL) at rt under irradiation of blue LED (7 W) for 17 h under argon.

Table S4 Screening of different light source[a]



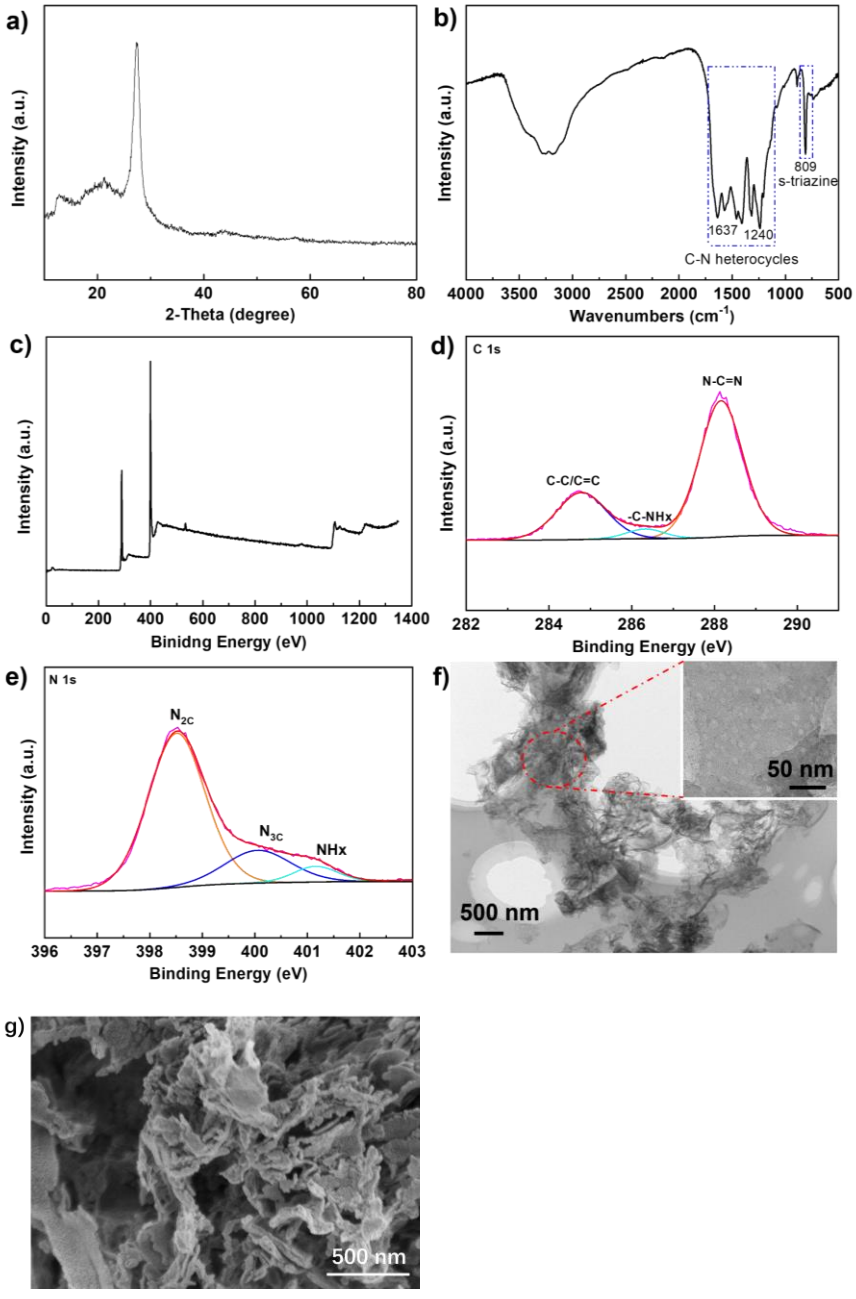
Entry	Light source	Isolated yield (%)
1	Purple LED	32
2	**Blue LED**	**89**
3	Green LED	trace
4	Red LED	trace
5	White LED	43

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (2 equiv.), g-C_3N_4 (5 mg) in DMSO (1.0 mL) at rt under irradiation of different light source (7 W) for 17 h under argon.

3. Synthesis and characterization of g-C_3N_4

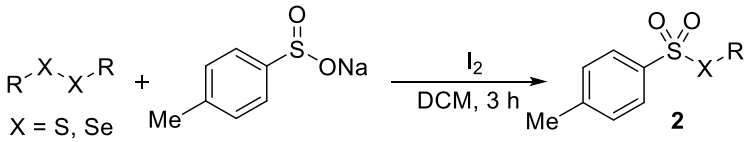
g-C_3N_4 was synthesized according to a two-step thermal polymerization. First, a certain amount of urea was added to a crucible with a cover, then heated to 550 °C (2.5 °C min^{-1}) for 4 h. Subsequently, the obtained yellow solid was further heated to 500 °C (5 °C min^{-1}) for 2 h in an open crucible to obtain the target g-C_3N_4.

Figure S1. a) XRD patterns, b) FT-IR spectra, c) XPS survey, d) C1s spectra, e) N1s spectra, f) TEM image of g-C_3N_4, g) SEM image of g-C_3N_4



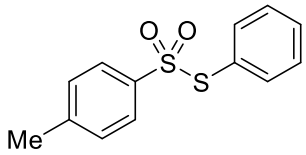
4. Synthesis of *X*-phenyl 4-methylbenzenesulfonothioate

General procedure 1



I_2 (2.0 equiv.) was added to the mixture of sodium benzenesulfinate (3.2 equiv.) and disulfide or diselenide (1.0 equiv.) in DCM. The mixture was stirred at room temperature until the disulfide or diselenide was consumed (determined by TLC, typically 3 h). The reaction was then quenched by saturated sodium thiosulfate. The aqueous layer was extracted three times with DCM. The combined organic layer was dried with anhydrous Na_2SO_4, concentrated and then subjected to flash column chromatography to obtain the desired product.[1]

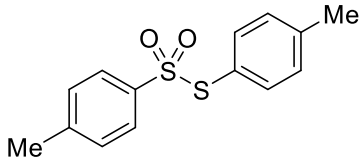
***S*-Phenyl 4-methylbenzenesulfonothioate (2a)**



According to **General procedure 1** with phenyl disulfide (1.1 g, 5 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (2.85 g, 16 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2a** as a colorless solid (1.1 g, 81% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 – 7.35 (m, 3H), 7.32 – 7.27 (m, 4H), 7.15 (d, J = 8.1 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.6, 139.7, 136.1, 131.1, 129.1, 127.5, 127.1, 21.3. The NMR spectra were in accord with that reported in literature.[2]

***S*-(*p*-Tolyl) 4-methylbenzenesulfonothioate (2b)**

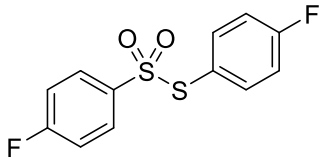


According to **General procedure 1** with *p*-tolyl disulfide (1.23 mg, 5 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (2.85 g, 16 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2b** as a colorless solid (1.04 g, 75%

yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, J = 8.3 Hz, 2H), 7.21 (t, J = 7.8 Hz, 4H), 7.12 (d, J = 8.0 Hz, 2H), 2.40 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.5, 142.0, 140.3, 136.3, 130.1, 129.3, 127.4, 124.4, 21.5, 21.4. The NMR spectra were in accord with that reported in literature.[3]

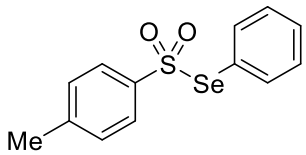
***S*-(4-Fluorophenyl) 4-fluorobenzenesulfonothioate (2c)**



1,2-Bis-(4-fluorophenyl) disulfide (254 mg, 1.0 mmol, 1.0 equiv.) was poured in a round bottom flask and dissolved in acetonitrile (40 mL) required to prepare 0.1 M solution. To this solution, a stoichiometric amount of 37% (w/w) hydrochloric acid (98.6 mg ,2.0 mmol,1.0 equiv.) and hydrogen peroxide 30% (510.0 mg, 3.0 mmol, 3 equiv.) were added. The mixture was stirred at rt for 8 h. Upon completion (monitored by TLC), the reaction was quenched with $NaHSO_3$, and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to afford the pure product **2c** as a colorless oil (246.3 mg, 86% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.56 (m, 2H), 7.38 – 7.34 (m, 2H), 7.14 – 7.09 (m, 2H), 7.08 – 7.03 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 166.1 (d, J = 110.1 Hz), 164.4 (d, J = 107.3 Hz), 138.8 (d, J = 9 Hz), 130.4 (d, J = 9.6 Hz), 123.22 (d, J = 1.1 Hz), 116.9 (d, J = 22.1 Hz), 116.2 (d, J = 22.7 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ: -102.44, -106.78. The NMR spectra were in accord with that reported in literature.[4]

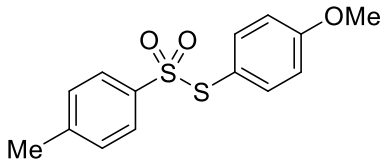
***Se*-Phenyl 4-methylbenzenesulfonoselenoate (2f)**



According to **General procedure 1** with diphenyl diselenide (1.56 g, 5 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (2.85 g, 16 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2f** as a yellow solid (1.2 g, 78% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.51 (dd, J = 8.0, 1.2 Hz, 2H), 7.47 (td, J = 7.4, 1.2 Hz, 1H), 7.39 (d, J = 8.3 Hz, 2H), 7.33 (dd, J = 10.8, 4.5 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 144.5, 142.7, 137.2, 130.8, 129.5, 129.2, 128.0, 127.0, 21.6. The NMR spectra were in accord with that reported in literature.[3]

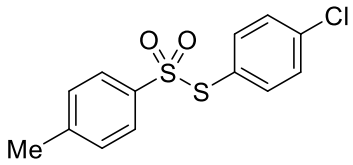
***S*-(4-Methoxyphenyl) 4-methylbenzenesulfonothioate (2g)**



According to **General procedure 1** with bis(4-methoxyphenyl) disulphide (835.17 mg, 3 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (1.71 g, 9.6 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2g** as a colorless solid (629.1 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (d, J = 8.2 Hz, 2H), 7.17 (t, J = 8.9 Hz, 4H), 6.79 (d, J = 8.7 Hz, 2H), 3.75 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.0, 144.4, 139.9, 138.0, 129.2, 127.2, 118.3, 114.7, 55.2, 21.4. The NMR spectra were in accord with that reported in literature.[5]

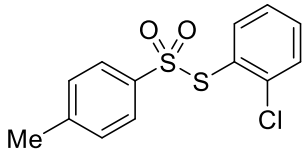
***S*-(4-Chlorophenyl) 4-methylbenzenesulfonothioate (2h)**



According to **General procedure 1** with bis(4-chlorophenyl) disulfide (1.44 g, 5 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (343.3 mg, 1.92 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2h** as a colorless solid (791.1 mg, 55% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (d, J = 8.2 Hz, 2H), 7.28 (d, J = 8.8 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 7.20 (d, J = 8.1 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.0, 140.0, 138.0, 137.6, 129.6, 129.4, 127.4, 126.4, 21.6. The NMR spectra were in accord with that reported in literature.[6]

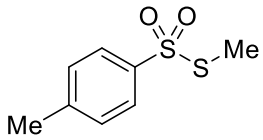
***S*-(2-Chlorophenyl) 4-methylbenzenesulfonothioate (2i)**



According to **General procedure 1** with 1,1'-disulfanediylbis(2-chlorobenzene) (172 mg, 0.6 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (343.3 mg, 1.92 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2i** as a colorless solid (122.6 mg, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, J = 7.7 Hz, 1H), 7.47 (d, J = 8.2 Hz, 2H), 7.40 (d, J = 6.7 Hz, 2H), 7.33 – 7.28 (m, 1H), 7.21 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.0, 140.5, 140.1, 139.3, 132.8, 130.2, 129.4, 127.5, 127.3, 126.9, 21.5. The NMR spectra were in accord with that reported in literature.[7]

***S*-Methyl 4-methylbenzenesulfonothioate (2j)**

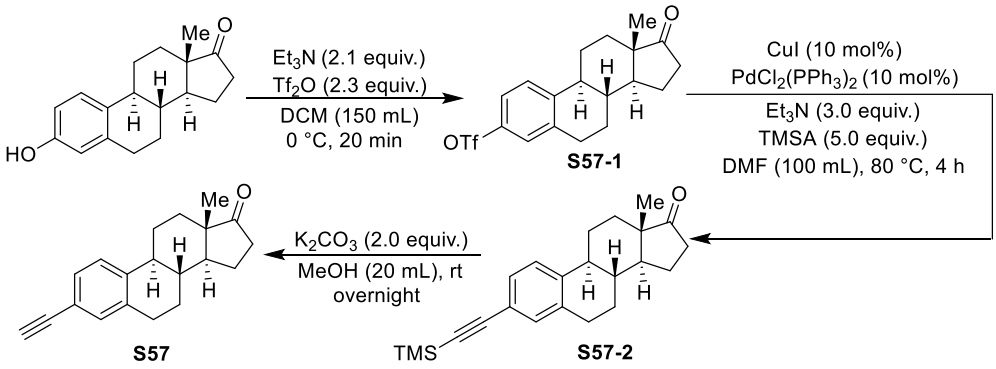


According to **General procedure 1** with dimethyl disulfide (471.0 mg, 5 mmol, 1.0 equiv.) and sodium *p*-toluenesulfinate (2.85 g, 16 mmol, 3.2 equiv.) for 3 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **2j** as a yellow solid (806.8 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 2.45 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.7, 140.7, 129.7, 126.9, 21.4, 17.8. The NMR spectra were in accord with that reported in literature.[8]

5. Synthesis of complex alkynes

(8*R*,9*S*,13*S*,14*S*)-3-Ethynyl-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (S57)



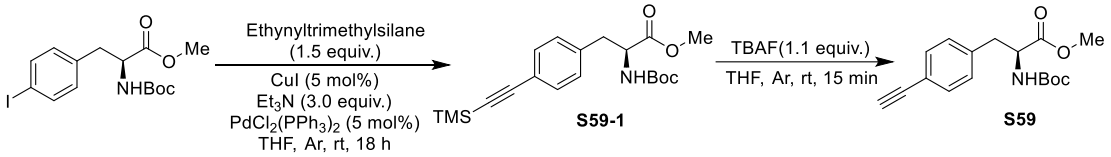
To a solution of estrone (2.0 g, 7.5 mmol, 1.0 equiv.) in dry CH_2Cl_2 (125 mL) at 0 °C, triethylamine (2.2 mL, 15.8mol, 5 equiv.) and trifluoromethanesulfonic anhydride (4.9 g, 17.3 mmol, 2.3 equiv.) were added. The reaction mixture was stirred at 0 ºC for 20 min. After the completion of reaction, the reaction was quenched with water. The combined organic layers were washed with saturated brine (2 × 30 mL), extracted with ethyl acetate (3 x 20 mL) and dried with over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product **S57-1** as a colorless solid (2.4 g, 81% yield).

Then, $Pd(PPh_3)_2Cl_2$ (259.0 mg, 0.37 mol, 10 mol%.), CuI (70.3 mg, 0.4 mmol, 10 mol%), Et_3N (2.2 mL, 11.1 mmol, 3 equiv.) and trimethylsilylacetylene (2.2 mL, 18.5 mmol, 5 equiv.) were added to the mixture of **S57-1** (1.5 g, 3.7 mmol, 1 equiv.) in DMF (74 mL) at 80 °C for 4 h. Upon completion (monitored by TLC), the reaction was quenched with water, and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product **S57-2** as a colorless solid (1.1 g, 85% yield).

To the reaction mixture of **S57-2** (1.0 g, 3.0 mmol, 1 equiv.) in MeOH (30 mL), K_2CO_3 (830.0 mg, 6.0 mmol, 2.0 equiv.) was added and the reaction was stirred at room temperature overnight. After the completion of reaction, the reaction was quenched with water and extracted with ethyl acetate (3 x 20 mL) for 2-3 times and dried with over anhydrous Na_2SO_4. The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **S57** as a white solid (675.5 mg, 81% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.2 Hz, 1H), 7.25 – 7.23 (m, 2H), 3.02 (s, 1H), 2.90 – 2.87 (m, 2H), 2.51 (dd, *J* = 19.1, 8.7 Hz, 1H), 2.41 (ddd, *J* = 10.1, 7.0, 3.6 Hz, 1H), 2.29 (td, *J* = 10.9, 3.8 Hz, 1H), 2.18 – 2.11 (m, 1H), 2.08 – 2.06 (m, 3H), 1.98 – 1.95 (m, 1H), 1.67 – 1.51 (m, 5H), 0.91 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) *δ*: 140.8, 136.6, 132.6, 129.4, 125.3, 119.4, 83.7, 76.4, 50.5, 47.9, 44.4, 37.9, 35.8, 31.5, 29.0, 26.3, 25.5, 21.5, 13.8.The NMR spectra were in accord with that reported in literature.[9]

Methyl (*S*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-ethynylphenyl)propanoate (S59)



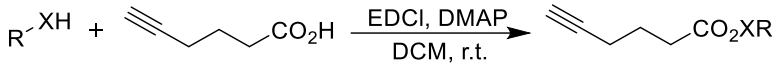
To a stirred solution of (*S*)-2-((*tert*-butoxycarbonyl)amino)-3(4-iodophenyl) propionate (1.0 g, 2.46 mmol, 1.0 equiv.), $PdCl_2(PPh_3)_2$ (5 mol%), CuI (5 mol%) and triethylamine (3 equiv.) in anhydrous tetrahydrofuran (0.1 M) was added at room temperature. The reaction mixture was bubbled with argon for 5 minutes and ethynyltrimethylsilane (1.5 equiv.) was then added under argon. The reaction mixture was stirred at room temperature for 18 h. Upon complete conversion, the crude reaction mixture was concentrated under vacuum and was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to afford the **S59-1** (822.1 mg, 89% yield).

Next, methyl (*S*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-((trimethylsilyl)ethynyl)phenyl) propanoate **S59-1** (0.50 g, 1.3 mmol, 1.0 equiv.) and TBAF (1.39 mL, 1.4 mmol, 1.1 equiv.) were dissolved in anhydrous tetrahydrofuran (12 mL) in a round bottom flask equipped with a stir bar at room temperature for 15 minutes. Upon complete conversion of the starting material, the crude reaction mixture was concentrated under vacuum and was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the desired product **S59** as a white oil (196.7 mg, 77% yield).[10]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 7.7 Hz, 2H), 4.99 (d, *J* = 7.4 Hz, 1H), 4.58 (d, *J* = 6.8 Hz, 1H), 3.71 (s, 3H), 3.12 (dt, *J* = 20.5, 10.3 Hz, 1H), 3.08 – 3.05 (m, 1H), 1.41 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) *δ*: 172.1, 155.0,

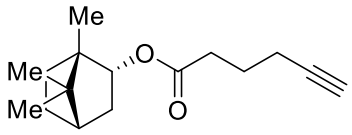
137.0, 132.2, 129.3, 120.8, 83.4, 80.0, 77.3, 54.2, 52.3, 38.3, 28.3. The NMR spectra were in accord with that reported in literature.[10]

General procedure 2



Alcohol or amine hydrochloride (1.0 equiv.), hexa-5-alkynoic acid (1.0 equiv.), EDCI (1.1 equiv.), DMAP (0.1 equiv.) and anhydrous DCM (30.0 mL) were added to a 100 mL Schlenk flask in turn under argon. The reaction mixture was stirred at room temperature for 3 h. After the reaction was completed, the reaction mixture was quenched with water and then extracted with DCM (3 × 10.0 mL). The combined organic layer was dried with Na_2SO_4 and evaporated under reduced pressure. Then, the residue was purified by column chromatography on silica gel to obtain the product.[11]

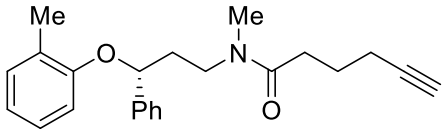
(1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl hex-5-ynoate (S60)



According to **General procedure 2**, **S60** was synthesized using L(-)-borneol (6.0 mmol). The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1) to afford the pure product as a colorless oil (1.1 g, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.89 (ddd, *J* = 9.9, 3.3, 2.2 Hz, 1H), 2.46 (t, *J* = 7.4 Hz, 2H), 2.39 – 2.30 (m, 1H), 2.27 (td, *J* = 7.0, 2.6 Hz, 2H), 1.97 (dd, *J* = 5.2, 2.6 Hz, 1H), 1.96 – 1.89 (m, 1H), 1.85 (dd, *J* = 14.5, 7.2 Hz, 2H), 1.79 – 1.68 (m, 1H), 1.66 (d, *J* = 4.5 Hz, 1H), 1.26 (dddd, *J* = 13.8, 12.3, 6.8, 3.3 Hz, 2H), 0.98 – 0.92 (m, 1H), 0.90 (s, 3H), 0.87 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 173.3, 83.3, 79.9, 69.0, 48.8, 47.8, 44.9, 36.8, 33.4, 28.1, 27.1, 23.8, 19.7, 18.8, 17.9, 13.5. The NMR spectra were in accord with that reported in literature.[11]

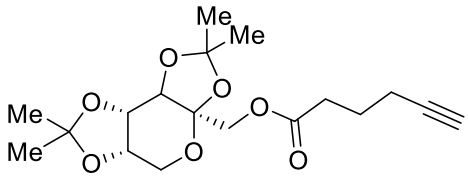
(*R*)-*N*-Methyl-*N*-(3-phenyl-3-(*o*-tolyloxy)propyl)hex-5-ynamide (S62)



According to **General procedure 2**, **S62** was synthesized using atomoxetine hydrochloride (5 mmol). The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1) to afford the pure product as a colorless oil (1.3 g, 73% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.36 – 7.28 (m, 4H), 7.27 – 7.21 (m, 1H), 7.12 (dd, *J* = 13.5, 7.3 Hz, 1H), 6.95 (t, *J* = 7.8 Hz, 1H), 6.81 – 6.74 (m, 1H), 6.57 – 6.53 (m, 1H), 5.16 (td, *J* = 8.0, 4.1 Hz, 1H), 3.65 – 3.46 (m, 2H), 2.97 (s, 1H), 2.93 (s, 1H), 2.50 – 2.37 (m, 2H), 2.37 – 2.30 (m, 4H), 2.27 (ddd, *J* = 9.2, 8.2, 2.5 Hz, 1H), 2.22 – 2.05 (m, 3H), 1.96 (dt, *J* = 8.1, 2.4 Hz, 1H), 1.86 – 1.74 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 172.2, 172.1, 155.7, 155.3, 141.6, 141.0, 130.7, 130.6, 128.8, 128.6, 127.8, 127.5, 126.8, 126.7, 126.6, 126.5, 125.6, 125.5, 120.6, 120.3, 112.6, 112.4, 83.84, 83.83, 77.5, 76.3, 68.9, 68.8, 46.4, 45.4, 37.5, 36.3, 35.8, 33.4, 31.8, 31.1, 23.8, 23.6, 17.9, 17.8, 16.52, 16.50; HRMS (ESI): *m/z* calcd for $C_{23}H_{27}NO_2Na$ $[M+Na]^+$: 372.1939, found: 372.1940.

((3a*R*,5a*S*,8a*S*)-2,2,7,7-Tetramethyltetrahydro-3a*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*] pyran-3a-yl)methyl hex-5-ynoate (S64)

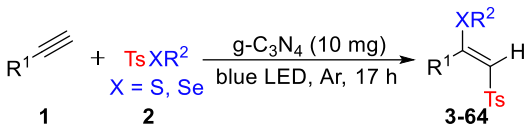


According to **General procedure 2**, **S64** was synthesized using diacetonefructose (5 mmol). The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product as a colorless oil (1.3 g, 74% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 4.58 (dd, *J* = 7.2, 2.6 Hz, 1H), 4.39 (dd, *J* = 11.7, 3.1 Hz, 1H), 4.30 – 4.28 (m, 1H), 4.24 – 4.20 (m, 1H), 4.01 (dd, *J* = 11.7, 3.2 Hz, 1H), 3.92 – 3.86 (m, 1H), 3.78 – 3.71 (m, 1H), 2.52 – 2.45 (m, 2H), 2.29 – 2.22 (m, 2H), 1.97 – 1.93 (m, 1H), 1.88 – 1.81 (m, 2H), 1.52 (d, *J* = 3.0 Hz, 3H), 1.46 (s, 3H), 1.38 (d, *J* = 5.7 Hz, 3H), 1.32 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 172.3, 109.1, 108.7, 101.5, 83.1, 70.7, 70.5, 70.0, 69.2, 65.1, 61.2, 32.6, 26.4, 25.8, 25.2, 24.0, 23.4, 17.7. The NMR spectra were in accord with that reported in literature.[11]

6. Synthesis of (*E*)-*β*-functionalized vinylsulfones

General procedure 3:



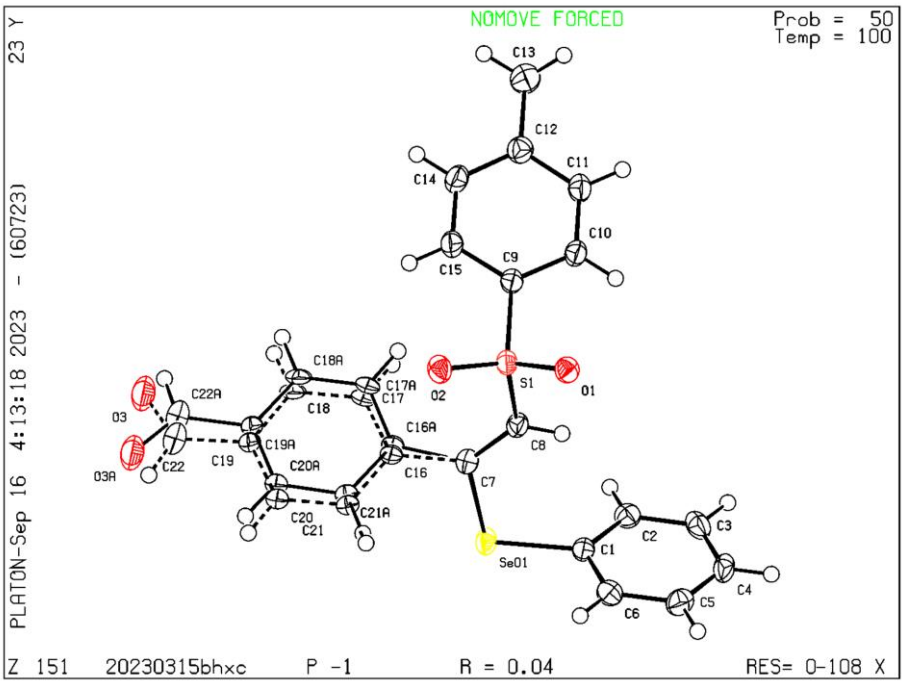
1 (0.2 mmol) and g-C_3N_4 (10 mg) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere in DMSO (2 mL) at rt for thiosulfonates (0.4 mmol) and DCM (2 mL) at -20 °C for selenosulfonates (0.4 mmol) under blue LED (7 W) irradiation for 17 h. Upon completion (monitored by TLC), the reaction mixture was filtered. The filtrate was transferred to a separate funnel using 20 mL ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to afford the pure products.

Crystal structure determination of 19

A suitable crystal of **19** was mounted with glue at the end of a glass fiber. Data collection was performed with a XtaLAB PRO MM007HF-DW diffractometer system equipped with a RA-Micro7HF-MR-DW(Cu/Mo) X-ray generator and Pilatus3R-200K-A detector (Rigaku, Japan, Cu Kα, λ = 1.54184 Å). The structure was solved by direct methods (SHELXTL-97) and refined by full-matrix least-squares (SHELXTL-97) refinements based on F^2. Anisotropic thermal parameters were applied to all non-hydrogen atoms. The hydrogen atoms were generated geometrically. Crystal data and structure refinement parameters are summarized in Table S5.

The single crystal suitable for X-ray diffraction was obtained by slow evaporation of a saturated solution of **19** (petroleum ether) in a loosely capped vial and has been deposited with the Cambridge Crystallographic Data Centre (CCDC 2267691). Copies of the data can be obtained free of charge on application to the CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

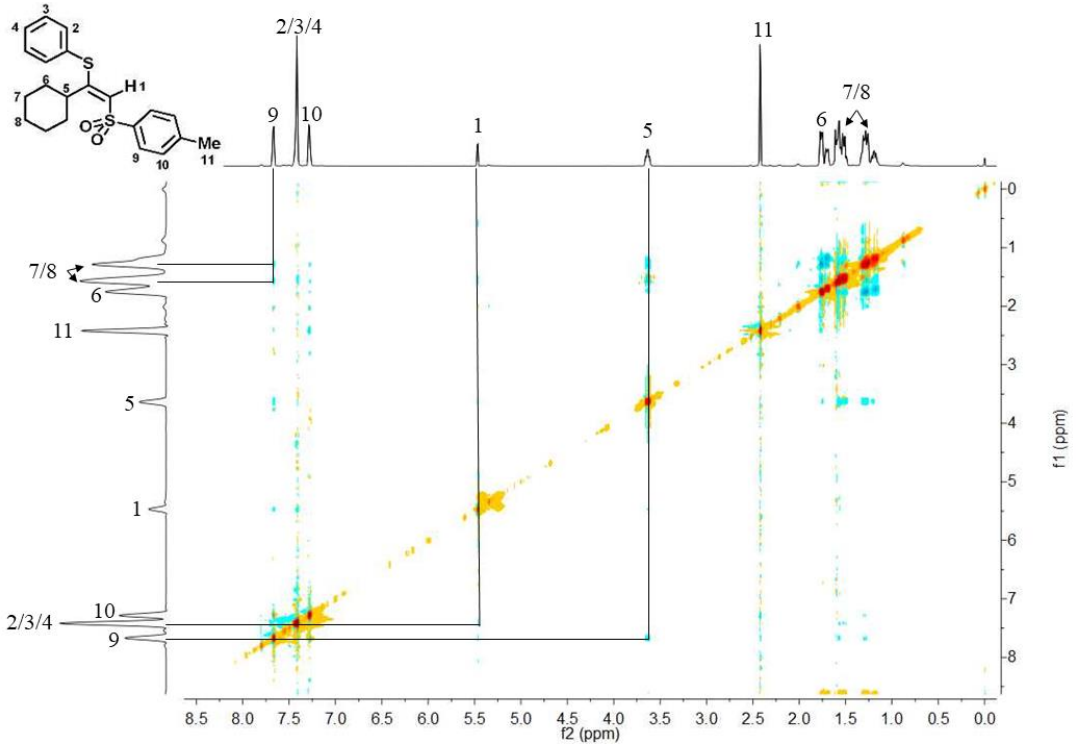
Table S5 Crystal data and structure refinement for 19



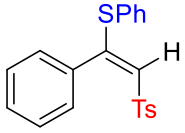
Identification code	**19**
Empirical formula	$C_{22}H_{18}O_3SSe$
Formula weight	441.38
Temperature/K	100(10)
Crystal system	triclinic
Space group	P-1
a/Å	7.2763(2)
b/Å	7.6736(3)
c/Å	18.9654(5)
α/°	83.579(2)
β/°	82.280(2)
γ/°	64.680(3)
Volume/Å^3	946.79(6)
Z	2
ρ_{calc}g/cm^3	1.548
μ/mm^{-1}	3.881
F(000)	448.0
Crystal size/mm^3	0.2 x 0.15 x 0.05

Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	4.715 to 71.046
Index ranges	-8 ≤ h ≤ 5, -9 ≤ k ≤ 9, -23 ≤ l ≤ 21
Reflections collected	7774
Independent reflections	3503 [Rint = 0.0344, Rsigma = 0.0347]
Data/restraints/parameters	3503/252/270
Goodness-of-fit on F^2	1.076
Final R indexes [I>=2σ (I)]	R1 = 0.0389, wR2 = 0.1051
Final R indexes [all data]	R1 = 0.0404, wR2 = 0.1061
Largest diff. peak/hole / e $Å^{-3}$	0.836/-1.085

NOESY spectrum of 36



(*E*)-Phenyl(1-phenyl-2-tosylvinyl)sulfane (3)

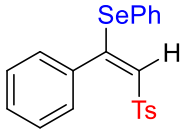


According to **General procedure 3** with phenylacetylene (20.4 mg, 0.20 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on

silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **3** as a colorless oil (32.6 mg, 89% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.51 (dd, J = 7.6, 1.8 Hz, 2H), 7.46 – 7.42 (m, 3H), 7.35 (ddd, J = 6.7, 4.0, 1.2 Hz, 1H), 7.30 – 7.27 (m, 4H), 7.25 – 7.22 (m, 2H), 7.10 (d, J = 8.0 Hz, 2H), 5.95 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 159.5, 143.4, 139.0, 135.3, 133.5, 130.4, 130.1, 129.5, 129.2, 129.0, 128.9, 127.8, 127.3, 122.6, 21.5. The NMR spectra were in accord with that reported in literature.[12]

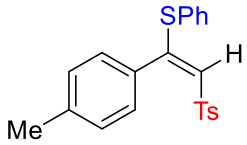
(*E*)-Phenyl(1-phenyl-2-tosylvinyl)selane (4)



According to **General procedure 3** with phenylacetylene (20.3 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.4 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **4** as a colorless oil (78.5 mg, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, J = 7.1 Hz, 2H), 7.42 (dq, J = 14.2, 7.2 Hz, 3H), 7.32 – 7.28 (m, 4H), 7.24 (s, 1H), 7.18 (d, J = 7.3 Hz, 2H), 7.10 (d, J = 7.8 Hz, 2H), 6.15 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.5, 138.8, 136.6, 134.6, 130.2, 130.1, 129.2, 128.4, 127.8, 127.4, 126.8, 125.8, 21.53. The NMR spectra were in accord with that reported in literature.[13]

(*E*)-Phenyl(1-(p-tolyl)-2-tosylvinyl)sulfane (5)

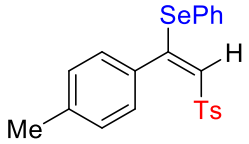


According to **General procedure 3** with 4-ethynyltoluene (24.0 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (106.7 mg, 0.4 mm ol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromato graphy on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **5** as a colorless oil (55.6 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.50 (dd, J = 7.2, 2.0 Hz, 2H), 7.43 (d, J = 6.6 Hz, 3H), 7.31 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 8.1 Hz, 2H), 7.11 (d, J = 6.4 Hz, 4H), 5.89 (s,

1H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.6, 143.4, 139.8, 139.2, 135.3, 130.7, 130.4, 130.1, 129.2, 129.0, 128.5, 127.3, 122.3, 21.5, 21.4; HRMS (ESI): *m/z* calcd for $C_{22}H_{20}O_2S_2Na$ $[M+Na]^+$: 403.0797, found: 403.0796.

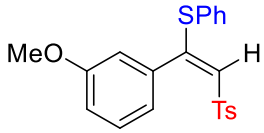
(*E*)-Phenyl(1-(*p*-tolyl)-2-tosylvinyl)selane (6)



According to **General procedure 3** with 4-ethynyltoluene (23.5 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.2 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **6** as a colorless oil (83.1 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, J = 7.6 Hz, 2H), 7.40 (dt, J = 14.2, 7.0 Hz, 3H), 7.33 (d, J = 7.9 Hz, 2H), 7.10 (dd, J = 10.7, 6.1 Hz, 6H), 6.09 (s, 1H), 2.37 (d, J = 4.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.5, 139.5, 138.9 136.5, 131.7, 130.1, 130.0, 129.6, 129.2, 128.5, 128.4, 127.4, 127.0, 125.4, 21.5, 21.4. The NMR spectra were in accord with that reported in literature.[13]

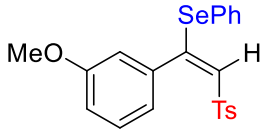
(*E*)-(1-(3-Methoxyphenyl)-2-tosylvinyl)(phenyl)sulfane (7)



According to **General procedure 3** with 3-ethynylanisole (27.9 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (103.5 mg, 0.4 mmo l, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatog raphy on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **7** as a colorless oil (72.5 mg, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (dd, J = 7.0, 2.1 Hz, 2H), 7.45 (d, J = 6.5 Hz, 3H), 7.29 (d, J = 8.2 Hz, 2H), 7.19 (t, J = 7.9 Hz, 1H), 7.09 (d, J = 8.0 Hz, 2H), 6.88 (dd, J = 8.3, 2.2 Hz, 1H), 6.83 (d, J = 7.5 Hz, 1H), 6.65 (s, 1H), 5.93 (s, 1H), 3.74 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.1, 158.9, 143.4, 139.0, 135.4, 134.6, 130.5, 130.1, 129.1, 128.9, 128.8, 127.4, 122.8, 121.5, 115.8, 113.9, 55.2, 21.4; HRMS (ESI): *m/z* calcd for $C_{22}H_{20}O_3S_2Na$ $[M+Na]^+$: 419.0752, found: 419.0748.

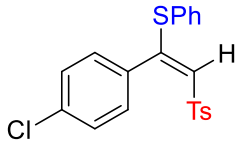
(*E*)-(1-(3-Methoxyphenyl)-2-tosylvinyl)(phenyl)selane (8)



According to **General procedure 3** with 3-ethynylanisole (25.8 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.8 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **8** as a colorless oil (79.5 mg, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.60 (d, J = 7.3 Hz, 2H), 7.42 (dt, J = 14.2, 7.1 Hz, 3H), 7.30 (d, J = 7.7 Hz, 2H), 7.17 (t, J = 7.9 Hz, 1H), 7.09 (d, J = 7.8 Hz, 2H), 6.85 (d, J = 8.2 Hz, 1H), 6.79 (d, J = 7.5 Hz, 1H), 6.60 (s, 1H), 6.14 (s, 1H), 3.73 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.8, 156.7, 143.5, 138.7, 136.6, 135.7, 130.2, 130.1, 129.1, 128.9, 127.5, 126.7, 126.0, 120.9, 115.5, 113.3, 55.1, 21.5; HRMS (ESI): m/z calcd for $C_{22}H_{20}O_3SSeNa$ $[M+Na]^+$: 467.0196, found: 467.0194.

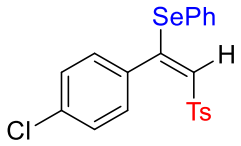
(*E*)-(1-(4-Chlorophenyl)-2-tosylvinyl)(phenyl)sulfane (9)



According to **General procedure 3** with 4-chlorophenylacetylene (28.2 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (109.0 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column c hromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **9** as a colorless oil (62.2 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, J = 10.8 Hz, 5H), 7.34 – 7.27 (m, 3H), 7.17 (dd, J = 12.4, 8.3 Hz, 5H), 5.95 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.0, 143.8, 138.9, 135.8, 135.3, 132.0, 130.6, 130.4, 130.1, 129.3, 128.1, 127.3, 123.2, 21.5; HRMS (ESI): m/z calcd for $C_{21}H_{18}O_2S_2Cl$ $[M+H]^+$: 401.0437, found: 401.0444.

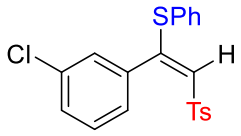
(*E*)-(1-(4-Chlorophenyl)-2-tosylvinyl)(phenyl)selane (10)



According to **General procedure 3** with 4-chlorophenylacetylene (27.0 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (125.4 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **10** as a colorless oil (83.3 mg, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ： 7.56 (d, J = 7.5 Hz, 2H), 7.47 – 7.42 (m, 1H), 7.39 (t, J = 7.3 Hz, 2H), 7.34 (d, J = 7.9 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.15 (t, J = 7.1 Hz, 4H), 6.17 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.5, 143.9, 138.6, 136.45, 135.4, 133.1, 130.2, 129.8, 129.4, 128.1, 127.4, 126.4, 21.5. The NMR spectra were in accord with that reported in literature.[13]

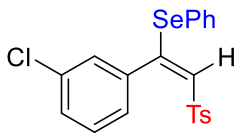
(*E*)-(1-(3-Chlorophenyl)-2-tosylvinyl)(phenyl)sulfane (11)



According to **General procedure 3** with 3-chlorophenylacetylene (29.0 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (108.1 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column c hromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **11** as a colorless oil (62.0 mg, 73% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (dd, J = 17.8, 6.5 Hz, 6H), 7.29 (d, J = 8.5 Hz, 3H), 7.21 (s, 1H), 7.14 (d, J = 8.0 Hz, 2H), 6.97 (s, 1H), 5.96 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.9, 135.4, 135.1, 133.8, 130.7, 130.2, 129.5, 129.3, 129.2, 128.6, 127.6, 127.3, 123.5, 21.5; HRMS (ESI): m/z calcd for $C_{21}H_{17}O_2S_2ClNa$ $[M+Na]^+$: 423.0256, found: 423.0257.

(*E*)-(1-(3-Chlorophenyl)-2-tosylvinyl)(phenyl)selane (12)

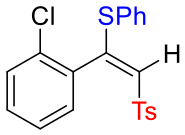


According to **General procedure 3** with 3-chlorophenylacetylene (27.2 mg, 0.20

mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.0 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **12** as a colorless oil (64.1 mg, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59 – 7.55 (m, 2H), 7.42 (dt, J = 14.3, 7.0 Hz, 3H), 7.31 (d, J = 8.2 Hz, 2H), 7.24 (dd, J = 13.5, 6.1 Hz, 2H), 7.15 (t, J = 5.2 Hz, 3H), 6.93 (s, 1H), 6.18 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.0, 136.6, 136.2, 133.8, 130.3, 130.3, 129.4, 129.2, 128.0, 127.4, 126.9, 126.9, 21.6. The NMR spectra were in accord with that reported in literature.[14]

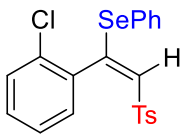
(*E*)-(1-(2-Chlorophenyl)-2-tosylvinyl)(phenyl)sulfane (13)



According to **General procedure 3** with 2-chlorophenylacetylene (29.2 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (107.9 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column c hromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **13** as a colorless oil (73.8. mg, 86% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54 (d, J = 7.1 Hz, 2H), 7.43 (d, J = 7.3 H z, 3H), 7.37 (d, J = 8.1 Hz, 3H), 7.31 (d, J = 3.2 Hz, 2H), 7.24 (s, 1H), 7.1 6 (d, J = 8.0 Hz, 2H), 5.98 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, CDC l_3) δ: 156.1, 143.8, 138.3, 135.5, 132.8, 132.3, 131.0, 130.5, 130.0, 129.5, 129. 3, 128.4, 127.5, 126.2, 123.3, 21.6; HRMS (ESI): m/z calcd for $C_{21}H_{17}O_2S_2Cl$ Na $[M+Na]^+$: 423.0256, found: 423.0257.

(*E*)-(1-(2-Chlorophenyl)-2-tosylvinyl)(phenyl)selane (14)

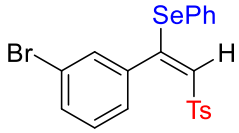


According to **General procedure 3** with 2-chlorophenylacetylene (26.9 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (125.2 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column

chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **14** as a colorless oil (61.3 mg, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, J = 7.3 Hz, 2H), 7.44 – 7.31 (m, 6H), 7.26 (s, 2H), 7.17 (d, J = 7.1 Hz, 3H), 6.25 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.1, 144.0, 138.0, 136.8, 133.5, 132.1, 130.2, 130.1, 130.0, 129.4, 127.6, 127.4, 126.8, 126.1, 21.6. The NMR spectra were in accord with that reported in literature.[14]

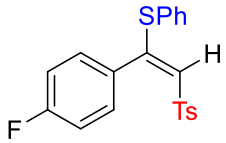
(*E*)-(1-(3-Bromophenyl)-2-tosylvinyl)(phenyl)selane (15)



According to **General procedure 3** with 3'-bromophenyl acetylene (36.8 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.9 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **15** as a colorless oil (91.6 mg, 93% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, J = 7.4 Hz, 2H), 7.47 – 7.38 (m, 4H), 7.30 (d, J = 7.8 Hz, 2H), 7.22 – 7.13 (m, 4H), 7.05 (s, 1H), 6.19 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.9, 144.0, 138.5, 136.6, 136.5, 132.0, 130.7, 130.3, 130.2, 129.4, 127.4, 127.4, 126.9, 126.2, 121.8, 21.6; HRMS (ESI): m/z calcd for $C_{21}H_{17}O_2SSeBrNa$ $[M+Na]^+$: 514.9196, found: 514.9194.

(*E*)-(1-(4-Fluorophenyl)-2-tosylvinyl)(phenyl)sulfane (16)

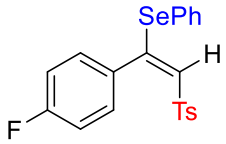


According to **General procedure 3** with 4-fluorophenylacetylene (23.3 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (108.1 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column c hromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **16** as a colorless oil (56.9 mg, 74% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (d, J = 7.5 Hz, 2H), 7.45 (t, J = 5.8 Hz, 3H), 7.30 (d, J = 8.1 Hz, 2H), 7.24 – 7.19 (m, 2H), 7.13 (d, J = 8.0 Hz, 2H), 6.97 (t, J = 8.6 Hz,

2H), 5.96 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.7, 143.7, 139.0, 135.2, 131.2 (d, J = 4.3 Hz), 130.5, 130.1, 128.2 (d, J = 205.2 Hz), 123.2, 115.0 (d, J = 4.3 Hz), 21.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -110.7; HRMS (ESI): m/z calcd for $C_{21}H_{18}O_2S_2F$ $[M+H]^+$: 385.0732, found: 385.0728.

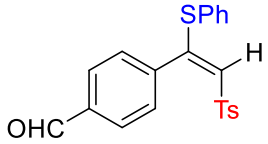
(*E*)-(1-(4-Fluorophenyl)-2-tosylvinyl)(phenyl)selane (17)



According to **General procedure 3** with 4-fluorophenylacetylene (23.1 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.6 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **17** as a colorless oil (69.9 mg, 81% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 – 7.54 (m, 2H), 7.41 (dd, J = 16.2, 7.3 Hz, 3H), 7.32 (d, J = 8.2 Hz, 2H), 7.20 – 7.13 (m, 4H), 6.96 (t, J = 8.7 Hz, 2H), 6.18 (s, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.4, 162.0, 143.8, 138.7, 136.5, 130.6 (d, J = 8.5 Hz), 130.2, 128.3 (d, J = 199.7 Hz), 126.7, 126.4, 115.0 (d, J = 21.8 Hz), 21.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -111.12. The NMR spectra were in accord with that reported in literature.[14]

(*E*)-4-(1-(Phenylthio)-2-tosylvinyl)benzaldehyde (18)

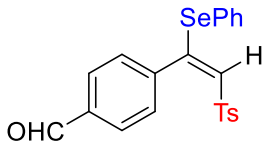


According to **General procedure 3** with 4-ethynylbenzaldehyde (27.1 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.7 mg, 0. 4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chr omatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pr oduct **18** as a colorless oil (61.5 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.04 (s, 1H), 7.82 (d, J = 8.1 Hz, 2H), 7.50 (d, J = 7.8 Hz, 2H), 7.46 (s, 2H), 7.43 (d, J = 4.8 Hz, 2H), 7.40 (s, 1H), 7. 33 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 5.97 (s, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 191.6, 157.6, 144.0, 139.8, 138.8, 136.7, 135.

4, 130.7, 130.2, 129.8, 129.5, 129.0, 128.2, 127.3, 123.3, 21.6; HRMS (ESI): m/z calcd for $C_{22}H_{18}O_3S_2Na$ $[M+Na]^+$: 417.0595, found: 417.0594.

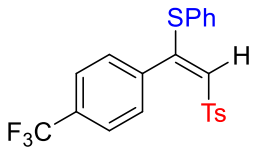
(*E*)-4-(1-(Phenylselanyl)-2-tosylvinyl)benzaldehyde (19)



According to **General procedure 3** with 4-ethynylbenzaldehyde (26.2 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (122.3 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **19** as a colorless oil (69.1 mg, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.02 (s, 1H), 7.80 (d, J = 8.1 Hz, 2H), 7.55 (d, J = 7.0 Hz, 2H), 7.44 (t, J = 7.3 Hz, 1H), 7.40 – 7.33 (m, 6H), 7.15 (d, J = 8.0 Hz, 2H), 6.20 (s, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 191.5, 144.1, 140.9, 138.5, 136.6, 136.4, 136.1, 130.3, 130.2, 129.5, 129.1, 129.0, 127.4, 126.5, 126.2, 21.6; HRMS (ESI): m/z calcd for $C_{22}H_{18}O_3SSeNa$ $[M+Na]^+$: 465.0040, found: 465.0038.

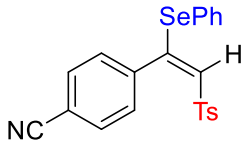
(*E*)-Phenyl(2-tosyl-1-(4-(trifluoromethyl)phenyl)vinyl)sulfane (20)



According to **General procedure 3** with 4'-trifluoromethylphenyl acetylene (17. 0 mg, 0.1 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (1 32.2 mg, 0.5 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **20** as a colorless oil (37.0 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (dd, J = 10.6, 5.0 Hz, 4H), 7.46 (t, J = 6.7 Hz, 3H), 7.33 (d, J = 8.1 Hz, 2H), 7.28 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 8.1 Hz, 2H), 5.99 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.2, 143.9, 138.7, 137.3, 135.4, 131.4 (q, J = 32.5 Hz), 130.7, 130.2, 129.5, 129.4, 128.3 (q, J = 270.6 Hz), 127.3, 124.8 (q, J = 3.7 Hz), 123.8, 21.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.86; HRMS (ESI): m/z calcd for $C_{22}H_{17}O_2S_2F_3Na$ $[M+Na]^+$: 457.0520, found: 457.0518.

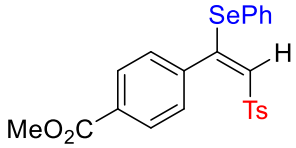
(*E*)-4-(1-(Phenylselanyl)-2-tosylvinyl)benzonitrile (21)



According to **General procedure 3** with 4-ethynylbenzonitrile (26.4 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.6 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **21** as a colorless oil (62.3 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 – 7.51 (m, 4H), 7.44 (t, J = 7.3 Hz, 1H), 7.39 – 7.33 (m, 4H), 7.29 (d, J = 8.3 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 6.21 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.9, 144.2, 139.6, 138.3, 136.5, 131.5, 130.4, 130.2, 129.5, 129.1, 127.3, 126.9, 125.9, 118.2, 112.8, 21.5. The NMR spectra were in accord with that reported in literature.[14]

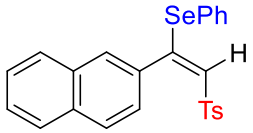
Methyl (*E*)-4-(1-(phenylselanyl)-2-tosylvinyl)benzoate (22)



According to **General procedure 3** with methyl 4-ethynylbenzoate (32.4 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (123.5 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **22** as a colorless oil (86.3 mg, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (d, J = 8.3 Hz, 2H), 7.57 – 7.54 (m, 2H), 7.43 (d, J = 7.2 Hz, 1H), 7.39 (d, J = 7.5 Hz, 2H), 7.32 (d, J = 8.2 Hz, 3H), 7.24 (s, 1H), 7.13 (d, J = 8.1 Hz, 2H), 6.18 (s, 1H), 3.93 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.4, 155.4, 144.0, 139.3, 138.6, 136.5, 130.6, 130.2, 130.2, 129.4, 129.0, 128.4, 127.4, 126.3, 126.3, 52.2, 21.5; HRMS (ESI): m/z calcd for $C_{23}H_{20}O_4SSeNa$ $[M+Na]^+$: 495.0145, found: 495.0148

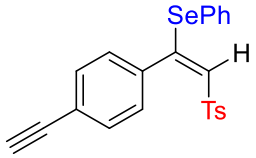
(*E*)-(1-(Naphthalen-2-yl)-2-tosylvinyl) (phenyl)selane (23)



According to **General procedure 3** with naphthylene-2-acetylene (29.3 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.0 mg, 0.40 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **23** as a colorless oil (68.0 mg, 76% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82 (d, J = 7.6 Hz, 1H), 7.73 (t, J = 7.9 Hz, 2H), 7.63 (d, J = 6.6 Hz, 2H), 7.57 – 7.49 (m, 3H), 7.46 – 7.38 (m, 3H), 7.32 (d, J = 8.4 Hz, 1H), 7.23 (d, J = 8.2 Hz, 2H), 6.94 (d, J = 8.0 Hz, 2H), 6.24 (s, 1H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.9, 143.6, 138.8, 136.6, 133.3, 132.3, 132.1, 130.2, 130.1, 129.08, 128.3, 128.0, 127.7, 127.5, 127.5, 127.0, 126.4, 125.9, 21.4. The NMR spectra were in accord with that reported in literature.[14]

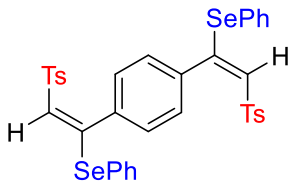
(*E*)-(1-(4-Ethynylphenyl)-2-tosylvinyl) (phenyl)selane (24)



According to **General procedure 3** with 1,4-diethynylbenzene (25.2 mg, 0.20 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (64.5 mg, 0.20 mmol, 1.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **24** as a colorless oil (53.3 mg, 52% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 – 7.54 (m, 2H), 7.43 (d, J = 7.2 Hz, 1H), 7.38 (t, J = 7.6 Hz, 4H), 7.32 (d, J = 8.2 Hz, 2H), 7.14 (dd, J = 8.2, 1.7 Hz, 4H), 6.17 (s, 1H), 3.14 (s, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.8, 143.8, 138.6, 136.5, 135.1, 131.5, 130.2, 129.3, 128.5, 127.4, 126.5, 126.3, 123.1, 83.0, 78.5, 21.5; HRMS (ESI): *m/z* calcd for $C_{23}H_{18}O_2SSeNa$ $[M+Na]^+$: 461.0090, found: 461.0092.

1,4-Bis((*E*)-1-(phenylselanyl)-2-tosylvinyl)benzene (25)

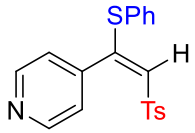


According to **General procedure 3** with 1,4-diethynylbenzene (12.6 mg, 0.1 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (120.8 mg, 0.40 mmol,

4.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **25** as a colorless oil (55.9 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61 (d, J = 6.9 Hz, 4H), 7.45 (dt, J = 14.1, 6.9 Hz, 6H), 7.32 (d, J = 8.1 Hz, 4H), 7.17 (d, J = 8.0 Hz, 4H), 7.08 (s, 4H), 6.18 (s, 2H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.1, 143.8, 138.8, 136.6, 135.6, 130.3, 129.5, 128.0, 127.6, 126.8, 126.6, 21.6; HRMS (ESI): *m/z* calcd for $C_{36}H_{30}O_4S_2Se_2Na$ $[M+Na]^+$: 772.9814, found: 772.9811

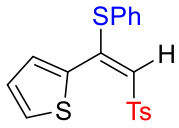
(*E*)-4-(1-(Phenylthio)-2-tosylvinyl)pyridine (26)



According to **General procedure 3** with 4-ethynylpyridine (20.6 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (264.4 mg, 1.0 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **26** as a colorless oil (30.0 mg, 41% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 – 7.42 (m, 6H), 7.33 (d, J = 8.3 Hz, 2H), 7.16 (d, J = 8.2 Hz, 5H), 5.98 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.8, 149.3, 144.1, 141.9, 138.6, 135.4, 130.8, 130.3, 129.5, 127.9, 127.3, 123.8, 21.5; HRMS (ESI): *m/z* calcd for $C_{20}H_{18}NO_2S_2$ $[M+H]^+$: 368.0779, found: 368.0776.

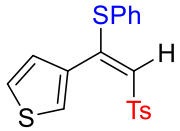
(*E*)-2-(1-(Phenylthio)-2-tosylvinyl)thiophene (27)



According to **General procedure 3** with 2-ethynylthiophene (21.6 mg, 0.2 mmol, 1.0 equiv.) and S-phenyl 4-methylbenzenesulfonothioate **2a** (264.4 mg, 1.0 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **27** as a colorless oil (58.1 mg, 78% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.49 – 7.45 (m, 3H), 7.41 (dt, J = 13.0, 8.3 Hz, 6H), 7.13 (d, J = 8.1 Hz, 2H), 7.01 (dd, J = 4.9, 3.8 Hz, 1H), 6.02 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 151.1, 143.6, 138.5, 134.9, 133.5, 132.2, 130.4, 130.1, 129.6, 129.5, 129.2, 127.3, 127.2, 124.1, 21.5; HRMS (ESI): *m/z* calcd for $C_{19}H_{16}O_2S_3Na$ $[M+Na]^+$: 395.0210, found: 395.0208.

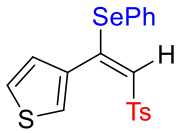
(*E*)-3-(1-(Phenylthio)-2-tosylvinyl)thiophene (28)



According to **General procedure 3** with 3-ethynylthiophene (21.6 mg, 0.2 mm ol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (264.4 mg, 1.0 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the pro duct **28** as a colorless oil (66.1 mg, 89% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.50 – 7.42 (m, 6H), 7.33 (d, J = 8.2 Hz, 2H), 7.20 (dd, J = 5.0, 3.0 Hz, 1H), 7.12 (d, J = 8.0 Hz, 2H), 7.04 – 7.01 (m, 1H), 5.98 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.5, 138.9, 135.2, 133.2, 130.4, 130.1, 129.2, 128.6, 127.9, 127.2, 125.1, 123.2, 21.5; HRMS (ESI): *m/z* calcd for $C_{19}H_{17}O_2S_3$ $[M+H]^+$: 373.0391, found: 373.0393.

(*E*)-3-(1-(Phenylselanyl)-2-tosylvinyl)thiophene (29)

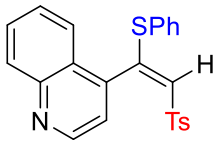


According to **General procedure 3** with 3-ethynylthiophene (22.3 mg, 0.2 mm ol, 1.0 equiv.) and Se-phenyl 4-methylbenzenesulfonoselenoate **2f** (93.8 mg, 0.3 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the pro duct **28** as a colorless oil (33.9 mg, 46% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 – 7.55 (m, 2H), 7.41 – 7.33 (m, 6H), 7.19 (dd, J = 5.0, 3.0 Hz, 1H), 7.12 (d, J = 8.0 Hz, 2H), 6.99 (dd, J = 5.0, 1.1 Hz, 1H), 6.20 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.2, 143.5, 138.6, 136.4, 134.2,

130.1, 130.0, 129.2, 128.3, 127.2, 127.0, 126.8, 126.4, 125.2, 21.5; HRMS (ESI): *m/z* calcd for $C_{19}H_{16}O_2NaS_2Se$ $[M+Na]^+$: 442.9655, found: 442.9651.

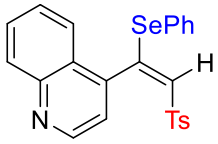
(*E*)-4-(1-(Phenylthio)-2-tosylvinyl)quinoline (30)



According to **General procedure 3** with 4-ethynylquinoline (30.6 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (264.4 mg, 1.0 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **30** as a colorless oil (45.7 mg, 55% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.85 (d, J = 4.3 Hz, 1H), 8.04 (d, J = 8.4 Hz, 1H), 7.68 – 7.62 (m, 2H), 7.52 (dd, J = 7.7, 1.5 Hz, 2H), 7.47 – 7.36 (m, 5H), 7.22 (d, J = 4.3 Hz, 1H), 7.07 (d, J = 8.2 Hz, 2H), 6.85 (d, J = 8.1 Hz, 2H), 6.25 (s, 1H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.3, 149.1, 147.9, 143.8, 139.5, 137.5, 135.6, 130.9, 130.2, 129.7, 129.4, 129.1, 127.6, 127.4, 126.9, 125.3, 125.0, 121.7, 21.3; HRMS (ESI): *m/z* calcd for $C_{24}H_{20}NO_2S_2$ $[M+H]^+$: 418.0935, found: 418.0935.

(*E*)-4-(1-(Phenylselanyl)-2-tosylvinyl)quinoline (31)

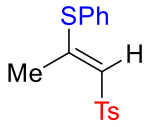


According to **General procedure 3** with 4-ethynylquinoline (30.4 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (123.2 mg, 0.4 mmol, 5.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **31** as a colorless oil (78.6 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.79 (d, J = 4.4 Hz, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.67 – 7.62 (m, 2H), 7.55 – 7.52 (m, 2H), 7.44 – 7.38 (m, 2H), 7.37 – 7.31 (m, 3H), 7.14 – 7.10 (m, 3H), 6.87 (d, J = 8.1 Hz, 2H), 6.51 (s, 1H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.0, 149.1, 147.8, 144.0, 140.7, 137.2, 136.9, 130.5, 130.1, 129.7, 129.5, 129.2, 128.6, 127.5, 126.7, 125.

5, 125.2, 124.5, 120.7, 21.4; HRMS (ESI): *m/z* calcd for $C_{24}H_{20}NO_2SSe$ $[M+H]^+$: 466.0380, found: 466.0379.

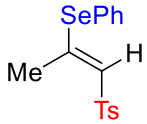
(*E*)-Phenyl(1-tosylprop-1-en-2-yl)sulfane (32)



According to **General procedure 3** with propyne (40.0 mg, 1.0 mmol, 5.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (53.0 mg, 0.2 mmol, 1.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **32** as a colorless oil (39.0 mg, 64% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.0 Hz, 2H), 7.43 (q, J = 5.9 Hz, 5H), 7.29 (d, J = 8.0 Hz, 2H), 5.62 (s, 1H), 2.42 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 158.0, 143.8, 139.7, 135.5, 130.5, 130.1, 129.7, 128.4, 126.9, 120.1, 21.6, 18.8. HRMS (ESI): *m/z* calcd for $C_{16}H_{16}O_2S_2Na$ $[M+Na]^+$: 327.0489, found: 327.0486.

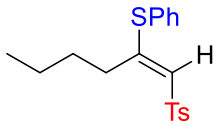
(*E*)-Phenyl(1-tosylprop-1-en-2-yl)selane (33)



According to **General procedure 3** with propyne (20.0 mg, 0.5 mmol, 5.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (31.1 mg, 0.1 mmol, 1.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **33** as a colorless oil (22.0 mg, 63% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.49 – 7.45 (m, 3H), 7.41 (dt, J = 13.0, 8.3 Hz, 6H), 7.13 (d, J = 8.1 Hz, 2H), 7.01 (dd, J = 4.9, 3.8 Hz, 1H), 6.02 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 155.6, 143.9, 139.4, 136.6, 130.1, 130.0, 129.8, 126.9, 125.9, 124.0, 21.6, 19.9. The NMR spectra were in accord with that reported in literature.[15]

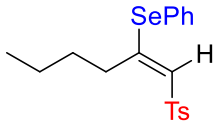
(*E*)-Phenyl(1-tosylhex-1-en-2-yl)sulfane (34)



According to **General procedure 3** with 1-hexyne (16.5 mg, 0.2 mmol, 1.0 eq uiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (106.0 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **34** as a colorless oil (61.1 mg, 88% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (d, J = 7.9 Hz, 2H), 7.41 (s, 6H), 7.28 (s, 1H), 5.53 (s, 1H), 2.78 – 2.71 (m, 3H), 2.40 (s, 3H), 1.36 (dd, J = 14.6, 7.3 Hz, 3H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.7, 139.9, 135.5, 130.3, 130.0, 129.7, 128.5, 126.8, 119.6, 32.0, 32.0, 22.6, 21.5, 13.8; HRMS (ESI): *m/z* calcd for $C_{19}H_{22}O_2S_2Na$ $[M+Na]^+$: 369.0959, found: 369.0954.

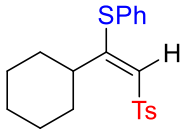
(*E*)-Phenyl(1-tosylhex-1-en-2-yl)selane (35)



According to **General procedure 3** with 1-hexyne (16.4 mg, 0.2 mmol, 1.0 eq uiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.2 mg, 0.4 mmol, 2. 0 equiv.) for 17 h, the reaction mixture was purified by column chromatograph y on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **35** a s a colorless oil (56.3 mg, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, J = 8.1 Hz, 2H), 7.52 (d, J = 7.1 Hz, 2H), 7.41 (d, J = 7.0 Hz, 1H), 7.36 (t, J = 7.3 Hz, 2H), 7.28 (d, J = 8.1 Hz, 2H), 5.84 (s, 1H), 2.86 – 2.77 (m, 2H), 2.41 (s, 3H), 1.58 – 1.50 (m, 2H), 1.35 (dd, J = 14.9, 7.4 Hz, 2H), 0.90 – 0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.5, 143.8, 139.5, 136.7, 130.0, 129.9, 129.7, 126.9, 125.9, 123.6, 33.0, 32.1, 22.5, 21.5, 13.8. The NMR spectra were in accord with that reported in literature.[13]

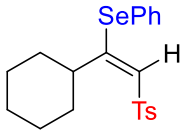
(*E*)-(1-Cyclohexyl-2-tosylvinyl)(phenyl) sulfane (36)



According to **General procedure 3** with cyclohexylacetylene (23.0 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.9 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **36** as a colorless oil (72.1 mg, 91% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, J = 7.8 Hz, 2H), 7.40 (s, 5H), 7.27 (d, J = 7.8 Hz, 2H), 5.45 (s, 1H), 3.61 (t, J = 10.9 Hz, 1H), 2.40 (s, 3H), 1.77 – 1.68 (m, 4H), 1.57 – 1.43 (m, 4H), 1.25 (d, J = 9.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.2, 143.6, 140.3, 135.8, 130.2, 130.1, 129.6, 128.5, 126.8, 119.5, 40.9, 31.8, 26.1, 25.6, 21.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{24}O_2S_2Na$ $[M+Na]^+$: 395.1115, found: 395.1117.

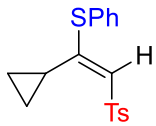
(*E*)-(1-Cyclohexyl-2-tosylvinyl)(phenyl)selane (37)



According to **General procedure 3** with cyclohexylacetylene (22.3 mg, 0.2 mm ol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.4 mg, 0. 4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chr omatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pr oduct **37** as a colorless oil (61.2 mg, 73% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (d, J = 8.0 Hz, 2H), 7.49 (d, J = 7.2 H z, 2H), 7.40 (d, J = 6.9 Hz, 1H), 7.35 (t, J = 7.2 Hz, 2H), 7.26 (s, 2H), 5.7 1 (s, 1H), 3.64 (t, J = 11.4 Hz, 1H), 2.41 (s, 3H), 1.79 – 1.65 (m, 4H), 1.42 (dt, J = 23.3, 11.5 Hz, 4H), 1.20 (d, J = 12.3 Hz, 2H); ^{13}C NMR (100 MH z, $CDCl_3$) δ: 143.7, 140.0, 137.0, 130.1, 129.8, 129.6, 126.9, 125.8, 123.3, 41. 8, 32.5, 25.9, 25.6, 21.6. The NMR spectra were in accord with that reported in literature.[16]

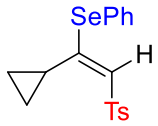
(*E*)-(1-Cyclopropyl-2-tosylvinyl)(phenyl)sulfane (38)



According to **General procedure 3** with ethynylcyclopropane (13.3 mg, 0.2 m mol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (107.3 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pro duct **38** as a colorless oil (23.1 mg, 35% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, J = 8.2 Hz, 2H), 7.43 – 7.35 (m, 5 H), 7.28 (d, J = 8.1 Hz, 2H), 5.59 (s, 1H), 2.92 – 2.85 (m, 1H), 2.42 (s, 3H), 1.04 – 1.00 (m, 2H), 0.94 (dt, J = 8.5, 3.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.2, 143.5, 140.1, 135.4, 130.2, 130.1, 129.6, 128.2, 126.7, 121.5, 21.5, 13.7, 8.4; HRMS (ESI): *m/z* calcd for $C_{18}H_{18}O_2S_2Na$ $[M+Na]^+$: 353.0646, found: 353.0641.

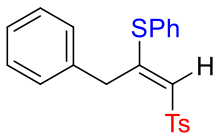
(*E*)-(1-Cyclopropyl-2-tosylvinyl)(phenyl)selane (39)



According to **General procedure 3** with ethynylcyclopropane (13.2 mg, 0.2 m mol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (123.3 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column c hromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **39** as a colorless oil (56.9 mg, yield 75%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, J = 8.2 Hz, 2H), 7.46 – 7.41 (m, 2H), 7.39 (d, J = 7.2 Hz, 1H), 7.34 (t, J = 7.2 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 5.87 (s, 1H), 2.93 – 2.86 (m, 1H), 2.42 (s, 3H), 0.96 (dd, J = 5.4, 3.1 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.1, 143.6, 139.6, 136.2, 130.1, 129.7, 129.6, 126.8, 125.8, 125.7, 21.5, 14.7, 9.2. The NMR spectra were in accord with that reported in literature.[13]

(*E*)-Phenyl(3-phenyl-1-tosylprop-1-en-2-yl)sulfane (40)

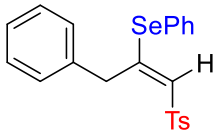


According to **General procedure 3** with 3-phenyl-1-propyne (26.7 mg, 0.2 mm ol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (106.2 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chro

matography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pro duct **40** as a colorless oil (70.4 mg, 81% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, J = 8.0 Hz, 2H), 7.44 – 7.33 (m, 6H), 7.28 (s, 1H), 7.22 (d, J = 6.9 Hz, 5H), 5.70 (s, 1H), 4.22 (s, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 160.5, 143.8, 139.4, 136.5, 135.5, 130.3, 130.0, 129.7, 129.0, 128.4, 126.9, 121.0, 37.0, 21.5; HRMS (ESI): *m/z* calcd for $C_{22}H_{20}O_2S_2Na$ $[M+Na]^+$: 403.0802, found: 403.0800.

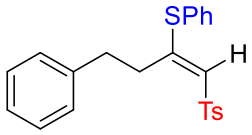
(*E*)-Phenyl(3-phenyl-1-tosylprop-1-en-2-yl)selane (41)



According to **General procedure 3** with 3-phenyl-1-propyne (23.4 mg, 0.2 mm ol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (125.3 mg, 0. 4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chr omatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pr oduct **41** as a colorless oil (72.7 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64 (d, J = 8.2 Hz, 2H), 7.47 – 7.38 (m, 3H), 7.35 (d, J = 7.5 Hz, 2H), 7.28 (dd, J = 6.9, 5.5 Hz, 5H), 7.21 – 7.17 (m, 2H), 6.00 (s, 1H), 4.31 (s, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.0, 144.0, 139.2, 136.6, 136.5, 130.0, 129.9, 129.8, 129.2, 128.5, 127.1, 127.0, 126.1, 125.0, 38.3, 21.6.; HRMS (ESI): *m/z* calcd for $C_{22}H_{20}O_2SSeNa$ $[M+Na]^+$: 451.0241, found: 451.0240.

(*E*)-Phenyl(4-phenyl-1-tosylbut-1-en-2-yl)sulfane (42)

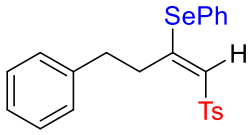


According to **General procedure 3** with 4-phenyl-1-butyne (25.8 mg, 0.2 mmo l, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.1 mg, 0.4 m mol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chroma tography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the produ ct **42** as a colorless oil (66.9 mg, 86% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (d, J = 8.3 Hz, 2H), 7.44 (s, 5H), 7.34 – 7.29 (m, 3H), 7.28 (d, J = 1.5 Hz, 3H), 7.23 (d, J = 7.1 Hz, 1H), 5.61 (s, 1H), 3.09 – 3.03 (m,

2H), 3.00 – 2.95 (m, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.5, 143.8, 140.5, 139.7, 135.5, 130.4, 130.0, 129.7, 128.5, 128.43, 128.36, 126.8, 126.3, 120.0, 36.2, 34.5, 21.5; HRMS (ESI): *m/z* calcd for $C_{23}H_{22}O_2S_2Na$ $[M+Na]^+$: 417.0959, found: 417.0961.

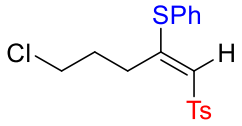
(*E*)-Phenyl(4-phenyl-1-tosylbut-1-en-2-yl)selane (43)



According to **General procedure 3** with 4-phenyl-1-butyne (12.8 mg, 0.2 mmo l, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **1ab** (62.5 mg, 0.2 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pro duct **43** as a colorless oil (36.1 mg, 83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.2 Hz, 2H), 7.58 – 7.53 (m, 2H), 7.45 (t, J = 7.3 Hz, 1H), 7.39 (t, J = 7.3 Hz, 2H), 7.30 (dd, J = 15.2, 8.0 Hz, 4H), 7.26 – 7.20 (m, 3H), 5.95 (s, 1H), 3.13 (dd, J = 10.2, 6.2 Hz, 2H), 2.95 (dd, J = 10.3, 6.2 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.6, 143.9, 140.4, 139.3, 136.7, 130.1, 129.9, 129.8, 128.5, 128.4, 126.9, 126.3, 125.8, 124.0, 36.3, 35.4, 21.5. The NMR spectra were in accord with that reported in literature.[14]

(*E*)-(5-Chloro-1-tosylpent-1-en-2-yl)(phenyl) sulfane (44)

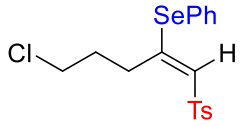


According to **General procedure 3** with 5-chloro-1-pentyne (21.9 mg, 0.2 mm ol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pro duct **44** as a colorless oil (55.0 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, J = 8.2 Hz, 2H), 7.45 – 7.41 (m, 5H), 7.30 (d, J = 8.2 Hz, 2H), 5.58 (s, 1H), 3.61 (t, J = 6.5 Hz, 2H), 2.94 – 2.88 (m, 2H), 2.42 (s, 3H), 2.18 – 2.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.0, 143.9, 139.5, 135.4, 130.5, 130.1, 129.8, 128.1, 126.8, 120.5, 44.2, 32.7, 29.8, 21.5; HRMS (ESI): *m/z*

calcd for $C_{18}H_{19}O_2S_2ClNa$ $[M+Na]^+$: 389.0413, found: 389.0415.

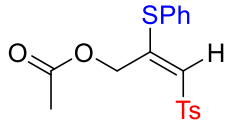
(*E*)-(5-Chloro-1-tosylpent-1-en-2-yl) (phenyl)selane (45)



According to **General procedure 3** with 5-chloro-1-pentyne (20.7 mg, 0.2 mm ol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (122.6 mg, 0. 4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chr omatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pr oduct **45** as a colorless oil (67.9 mg, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.2 Hz, 2H), 7.53 – 7.50 (m, 2H), 7.45 – 7.41 (m, 1H), 7.37 (t, J = 7.3 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 5.90 (s, 1H), 3.59 (t, J = 6.5 Hz, 2H), 2.97 (dd, J = 9.0, 6.6 Hz, 2H), 2.42 (s, 3H), 2.15 – 2.07 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.9, 144.1, 139.1, 136.6, 130.1, 130.03, 129.8, 126.9, 125.7, 124.6, 44.1, 32.9, 30.8, 21.6; HRMS (ESI): *m/z* calcd for $C_{18}H_{20}O_2SClSe$ $[M+H]^+$: 415.0038, found: 415.0040.

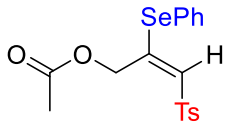
(*E*)-2-(Phenylthio)-3-tosylallylacetate (46)



According to **General procedure 3** with propargyl acetate (20.5 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.6 mg, 0.4 mm ol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromato graphy on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **46** as a colorless oil (60.0 mg, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, J = 7.8 Hz, 2H), 7.42 (s, 5H), 7.30 (d, J = 7.8 Hz, 2H), 5.52 (s, 1H), 5.39 (s, 2H), 2.42 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.0, 156.8, 144.2, 138.7, 135.5, 130.6, 130.2, 129.8, 127.3, 127.0, 121.0, 60.9, 21.6, 20.6; HRMS (ESI): *m/z* calcd for $C_{18}H_{18}O_4S_2Na$ $[M+Na]^+$: 385.0544, found: 385.0540.

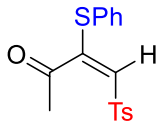
(*E*)-2-(Phenylselanyl)-3-tosylallylacetate (47)



According to **General procedure 3** with propargyl acetate (19.3 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (125.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chro matography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the pro duct **47** as a colorless oil (57.1 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.2 Hz, 2H), 7.50 (d, J = 7.0 H z, 2H), 7.43 (d, J = 7.3 Hz, 1H), 7.37 (t, J = 7.4 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 5.70 (s, 1H), 5.48 (d, J = 1.4 Hz, 2H), 2.41 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.9, 155.9, 144.3, 138.4, 136.9, 130.3, 130.3, 129.9, 127.1, 124.9, 123.8, 62.3, 21.6, 20.6; HRMS (ESI): *m/z* calcd for $C_{18}H_{18}O_4SSeNa$ $[M+Na]^+$: 432.9989, found: 432.9990.

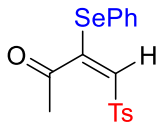
(*E*)-3-(Phenylthio)-4-tosylbut-3-en-2-one (48)



According to **General procedure 3** with 3-butyn-2-one (15.4 mg, 0.2 mmol, 1. 0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (104.2 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatogr aphy on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **4 8** as a colorless oil (43.4 mg, 58% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.3 Hz, 2H), 7.45 (dt, J = 12.7, 5.0 Hz, 5H), 7.31 (d, J = 8.1 Hz, 2H), 5.57 (s, 1H), 2.54 (s, 3H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 198.2, 157.9, 144.5, 137.6, 135.5, 130.8, 130.2, 129.9, 127.7, 126.2, 119.8, 30.9, 21.6; HRMS (ESI): *m/z* calcd for $C_{17}H_{16}O_3S_2Na$ $[M+Na]^+$: 355.0433, found: 355.0430.

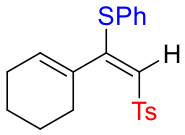
(*E*)-3-(Phenylselanyl)-4-tosylbut-3-en-2-one (49)



According to **General procedure 3** with 3-butyn-2-one (13.4 mg, 0.2 mmol, 1. 0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (126.0 mg, 0.4 mm ol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromato graphy on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **49** as a colorless oil (43.9 mg, 59% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.3 Hz, 2H), 7.56 – 7.54 (m, 2H), 7.44 (d, J = 7.4 Hz, 1H), 7.39 (d, J = 7.5 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 5.86 (s, 1H), 2.47 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 199.2, 144.7, 137.2, 136.7, 130.5, 130.2, 130.0, 127.8, 123.9, 123.3, 30.7, 21.7; HRMS (ESI): *m/z* calcd for $C_{17}H_{16}O_3SSeNa$ $[M+Na]^+$: 402.9883, found: 402.9886.

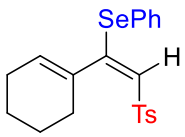
(*E*)-(1-(Cyclohex-1-en-1-yl)-2-tosylvinyl) (phenyl)sulfane (50)



According to **General procedure 3** with 1-ethynylcyclohex-1-ene (21.3 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **50** as a colorless oil (58.7 mg, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (d, J = 8.2 Hz, 2H), 7.46 – 7.38 (m, 6H), 7.28 (s, 1H), 5.79 (s, 1H), 5.53 (s, 1H), 2.42 (s, 3H), 2.08 (d, J = 1.8 Hz, 2H), 1.98 (d, J = 2.0 Hz, 2H), 1.50 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.5, 143.5, 140.0, 135.3, 132.4, 131.2, 130.0, 129.8, 129.3, 129.3, 127.4, 121.0, 28.4, 25.2, 22.1, 21.5, 21.2; HRMS (ESI): *m/z* calcd for $C_{21}H_{22}O_2S_2Na$ $[M+Na]^+$: 393.0959, found: 393.0960.

(*E*)-(1-(Cyclohex-1-en-1-yl)-2-tosylvinyl) (phenyl)selane (51)

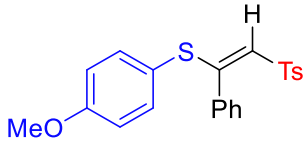


According to **General procedure 3** with 1-ethynylcyclohex-1-ene (21.3 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the

product **51** as a colorless oil (55.9 mg, 67% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.65 (d, J = 8.0 Hz, 2H), 7.52 (d, J = 7.7 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H), 7.35 (t, J = 7.4 Hz, 2H), 7.27 (d, J = 7.8 Hz, 2H), 6.05 (s, 1H), 5.47 (s, 1H), 2.41 (s, 3H), 2.05 (s, 2H), 1.93 (s, 2H), 1.45 (d, J = 2.3 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 160.4, 143.7, 139.7, 136.6, 133.4, 130.1, 129.73, 129.66, 129.4, 127.6, 126.8, 124.2, 28.4, 25.1, 22.0, 21.6, 21.1. The NMR spectra were in accord with that reported in literature.[17]

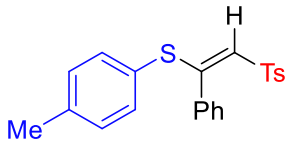
(*E*)-(4-Methoxyphenyl)(1-phenyl-2-tosylvinyl)sulfane (52)



According to **General procedure 3** with phenylacetylene (20.4 mg, 0.2 mmol, 1.0 equiv.) and *S*-(4-methoxyphenyl) 4-methylbenzenesulfonothioate **2g** (140.2 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **52** as a colorless oil (58.1 mg, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, J = 8.4 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 7.28 (t, J = 8.2 Hz, 4H), 7.20 (d, J = 7.5 Hz, 2H), 7.09 (d, J = 7.9 Hz, 2H), 6.96 (d, J = 8.4 Hz, 2H), 5.86 (s, 1H), 3.85 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.4, 160.7, 143.3, 139.3, 137.0, 133.6, 129.4, 129.1, 129.0, 127.8, 127.3, 122.2, 119.4, 115.7, 55.4, 21.5; HRMS (ESI): *m/z* calcd for $C_{22}H_{20}O_3S_2Na$ $[M+Na]^+$: 419.0752, found: 419.0751.

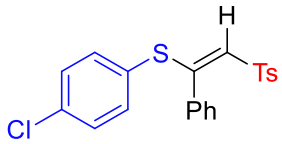
(*E*)-(1-Phenyl-2-tosylvinyl)(*p*-tolyl)sulfane (53)



According to **General procedure 3** with phenylacetylene (20.4 mg, 0.2 mmol, 1.0 equiv.) and *S*-(*p*-tolyl) 4-methylbenzenesulfonothioate **2b** (111.4 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **53** as a colorless oil (47.0 mg, 62% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.40 (d, J = 7.9 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 7.27 (dt, J = 7.5, 6.0 Hz, 6H), 7.21 (d, J = 7.5 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H), 5.90 (s, 1H), 2.40 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 160.3, 143.3, 141.0, 139.2, 135.3, 133.6, 131.0, 129.5, 129.2, 129.0, 127.8, 127.3, 125.3, 122.3, 21.5, 21.4; HRMS (ESI): *m/z* calcd for $C_{22}H_{21}O_2S_2$ $[M+H]^+$: 381.0983, found: 381.0988.

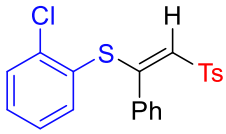
(*E*)-(4-Chlorophenyl)(1-phenyl-2-tosylvinyl)sulfane (54)



According to **General procedure 3** with phenylacetylene (20.4 mg, 0.2 mmol, 1.0 equiv.) and *S*-(4-chlorophenyl) 4-methylbenzenesulfonothioate **2h** (119.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **54** as a colorless oil (75.5 mg, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 – 7.33 (m, 5H), 7.29 (d, J = 7.3 Hz, 4H), 7.20 (d, J = 7.3 Hz, 2H), 7.10 (d, J = 8.0 Hz, 2H), 5.96 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.6, 143.6, 138.8, 136.9, 136.5, 133.2, 130.3, 129.6, 129.2, 129.0, 127.8, 127.3, 123.2, 21.5. The NMR spectra were in accord with that reported in literature.[18]

(*E*)-(2-Chlorophenyl)(1-phenyl-2-tosylvinyl)sulfane (55)

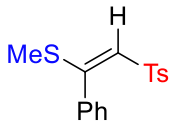


According to **General procedure 3** with phenylacetylene (20.4 mg, 0.2 mmol, 1.0 equiv.) and *S*-(2-chlorophenyl) 4-methylbenzenesulfonothioate **2i** (119.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **55** as a colorless oil (74.3 mg, 93% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61 (dd, J = 7.6, 1.5 Hz, 1H), 7.53 (dd, J = 8.0, 1.0 Hz, 1H), 7.42 (td, J = 7.8, 1.6 Hz, 1H), 7.34 (dd, J = 7.5, 1.2 Hz, 2H), 7.28 (d, J = 7.7 Hz, 5H), 7.08 (d, J = 8.1 Hz, 3H), 5.87 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.5, 143.4, 139.3, 138.9, 137.7, 133.1, 132.2, 131.0, 129.7, 129.2, 129.1,

128.1, 127.8, 127.4, 123.3, 21.5; HRMS (ESI): *m/z* calcd for $C_{21}H_{17}O_2S_2ClNa$ $[M+Na]^+$: 423.0256, found: 423.0257.

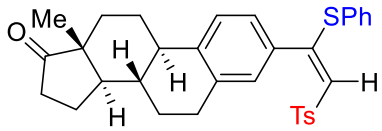
(*E*)-Methyl(1-phenyl-2-tosylvinyl)sulfane (56)



According to **General procedure 3** with phenylacetylene (20.4 mg, 0.2 mmol, 1.0 equiv.) and *S*-methyl 4-methylbenzenesulfonothioate **2j** (119.5 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **56** as a colorless oil (31.8 mg, 52% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.36 (t, J = 8.2 Hz, 3H), 7.28 (t, J = 6.8 Hz, 2H), 7.16 – 7.10 (m, 4H), 6.20 (s, 1H), 2.37 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.2, 143.5, 139.2, 134.2, 129.4, 129.2, 128.8, 127.8, 127.4, 120.8, 21.5, 16.3; HRMS (ESI): *m/z* calcd for $C_{16}H_{16}O_2S_2Na$ $[M+Na]^+$: 327.0489, found: 327.0488

(8*S*,9*R*,13*R*,14*R*)-13-Methyl-3-((*E*)-1-(phenylthio)-2-tosylvinyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (57)

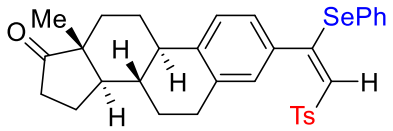


According to **General procedure 3** with (8*S*,9*R*,13*R*,14*R*)-3-ethynyl-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one **S57** (55.6 mg, 0.2 mmol, 1.0 equiv.) and S-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.4 mmol, 2.0 equiv.) for 72 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **57** as a colorless oil (62.0 mg, 57% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.53 (dd, J = 7.5, 1.6 Hz, 2H), 7.46 (d, J = 5.9 Hz, 3H), 7.33 – 7.29 (m, 2H), 7.23 (d, J = 8.0 Hz, 1H), 7.09 (dd, J = 18.4, 7.9 Hz, 3H), 6.87 (s, 1H), 5.85 (s, 1H), 2.82 (dd, J = 9.0, 6.2 Hz, 2H), 2.53 (dd, J = 18.9, 8.8 Hz, 1H), 2.45 – 2.41 (m, 1H), 2.38 (s, 3H), 2.32 (t, J = 8.8 Hz, 1H), 2.18 (td, J = 18.5, 8.5 Hz, 2H), 2.10 (dd, J = 11.9, 5.3 Hz, 1H), 2.06 – 1.97 (m, 3H), 1.64 (d, J = 11.8 Hz, 1H), 1.56 –

1.52 (m, 2H), 1.47 – 1.42 (m, 1H), 0.95 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 159.7, 143.2, 141.4, 139.3, 136.0, 135.4, 131.1, 130.5, 130.2, 129.3, 129.0, 127.4, 126.4, 124.9, 122.0, 50.5, 47.9, 44.5, 38.0, 35.8, 31.6, 29.7, 29.2, 26.4, 25.6, 21.6, 21.5, 13.9; HRMS (ESI): *m/z* calcd for $C_{33}H_{34}O_3S_2Na$ $[M+Na]^+$: 565.1847, found: 565.1849.

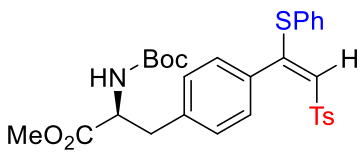
(8*S*,9*R*,13*R*,14*R*)-13-Methyl-3-((*E*)-1-(phenylselanyl)-2-tosylvinyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (58)



According to **General procedure 3** with (8*S*,9*R*,13*R*,14*R*)-3-ethynyl-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one **S57** (55.6 mg, 0.2 mmol, 1.0 equiv.) and Se-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.6 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **58** as a colorless oil (56.4 mg, 48% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.60 (d, J = 7.2 Hz, 2H), 7.42 (dt, J = 14.7, 7.2 Hz, 3H), 7.33 (d, J = 8.1 Hz, 2H), 7.20 (d, J = 8.0 Hz, 1H), 7.10 (d, J = 7.9 Hz, 2H), 7.03 (d, J = 7.8 Hz, 1H), 6.84 (s, 1H), 6.05 (s, 1H), 2.82 – 2.78 (m, 2H), 2.55 – 2.50 (m, 1H), 2.37 (s, 3H), 2.30 (s, 1H), 2.20 – 2.13 (m, 1H), 2.11 – 2.06 (m, 1H), 2.04 – 1.99 (m, 2H), 1.63 (dd, J = 12.4, 10.2 Hz, 4H), 1.56 – 1.51 (m, 3H), 1.47 – 1.41 (m, 1H), 0.95 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 157.4, 143.3, 141.2, 139.0, 136.6, 136.0, 132.1, 130.2, 130.1, 129.1, 128.7, 127.4, 127.0, 125.9, 125.2, 124.9, 50.5, 47.9, 44.5, 38.0, 35.8, 31.6, 29.2, 26.4, 25.6, 21.6, 21.6, 13.9; HRMS (ESI): *m/z* calcd for $C_{33}H_{34}O_3SSeNa$ $[M+Na]^+$: 613.1292, found: 613.1291.

Methyl(*S*,*E*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-(1-(phenylthio)-2-tosylvinyl)phenyl)propanoate (59)

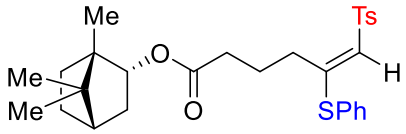


According to **General procedure 3** with methyl (*S*)-2-((*tert*-butoxycarbonyl)amino)-3-(4-ethynylphenyl)propanoate **S59** (60.8 mg, 0.2

mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.6 mg, 0.4 mmol, 2.0 equiv.) for 72 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **59** as a colorless oil (60.1 mg, 53% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.50 (d, J = 6.3 Hz, 2H), 7.47 – 7.42 (m, 3H), 7.27 (s, 2H), 7.18 – 7.13 (m, 4H), 7.07 (d, J = 7.7 Hz, 2H), 5.94 (s, 1H), 5.02 (d, J = 8.0 Hz, 1H), 4.61 (dd, J = 13.4, 6.2 Hz, 1H), 3.74 (s, 3H), 3.15 – 3.06 (m, 2H), 2.39 (s, 3H), 1.45 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 172.1, 159.4, 155.1, 143.5, 139.1, 137.8, 135.3, 132.2, 130.5, 130.1, 129.3, 129.2, 128.8, 128.7, 127.3, 122.7, 80.2, 54.3, 52.3, 38.2, 28.3, 21.5; HRMS (ESI): *m/z* calcd for $C_{30}H_{33}NO_6S_2Na$ $[M+Na]^+$: 590.1647, found: 590.1644.

(1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl(*E*)-5-(phenylthio)-6-tosylhex-5-enoate (60)

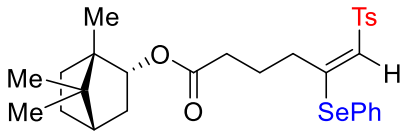


According to **General procedure 3** with methyl (1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl hex-5-ynoate **S60** (53.2 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.4 mmol, 2.0 equiv.) for 72 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **60** as a colorless oil (74.6 mg, 74% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.59 (d, J = 8.3 Hz, 2H), 7.35 (s, 5H), 7.23 – 7.19 (m, 2H), 5.49 (s, 1H), 4.84 (ddd, J = 9.9, 3.2, 2.1 Hz, 1H), 2.81 – 2.75 (m, 2H), 2.37 – 2.33 (m, 5H), 1.95 – 1.89 (m, 3H), 1.68 (d, J = 3.5 Hz, 1H), 1.61 (t, J = 4.4 Hz, 1H), 1.55 (d, J = 6.3 Hz, 1H), 1.26 (dd, J = 8.4, 6.3 Hz, 1H), 0.92 (dd, J = 13.7, 3.4 Hz, 1H), 0.84 (s, 3H), 0.81 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 173.2, 161.8, 143.8, 139.7, 135.5, 130.4, 130.1, 129.8, 128.3, 126.8, 120.2, 80.0, 48.7, 47.8, 44.9, 36.8, 33.9, 31.4, 28.0, 27.1, 25.1, 21.6, 19.7, 18.8, 13.6; HRMS (ESI): *m/z* calcd for $C_{29}H_{36}O_4S_2Na$ $[M+Na]^+$: 535.1953, found: 535.1951.

(1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl(*E*)-5-(phenylselanyl)-6-tosyl

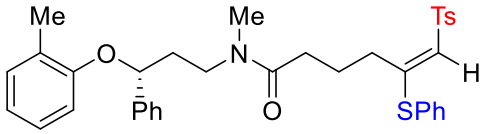
hex-5-enoate (61)



According to **General procedure 3** with methyl (1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl hex-5-ynoate **S60** (53.2 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.7 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **61** as a colorless oil (82.4 mg, 74% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.66 (d, J = 8.2 Hz, 2H), 7.51 (d, J = 7.9 Hz, 2H), 7.42 (dd, J = 10.6, 4.2 Hz, 1H), 7.36 (dd, J = 10.6, 4.5 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.86 (s, 1H), 4.90 (dd, J = 9.9, 1.9 Hz, 1H), 2.93 – 2.88 (m, 2H), 2.41 (s, 3H), 2.39 (d, J = 7.7 Hz, 2H), 1.99 – 1.93 (m, 3H), 1.77 – 1.73 (m, 1H), 1.34 – 1.27 (m, 2H), 0.99 (d, J = 3.2 Hz, 1H), 0.97 (d, J = 3.4 Hz, 1H), 0.90 (s, 3H), 0.87 (s, 3H), 0.84 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 173.1, 159.9, 143.9, 139.3, 136.7, 130.1, 130.0, 129.8, 126.9, 125.7, 124.3, 79.9, 48.7, 47.8, 44.8, 36.8, 33.8, 32.3, 28.0, 27.1, 25.3, 21.6, 19.7, 18.8, 13.5; HRMS (ESI): *m/z* calcd for $C_{29}H_{36}O_4SSeNa$ $[M+Na]^+$: 583.1397, found: 583.1392.

(*R*,*E*)-*N*-Methyl-*N*-(3-phenyl-3-(*o*-tolyloxy)propyl)-5-(phenylthio)-6-tosylhex-5-enamide (62)

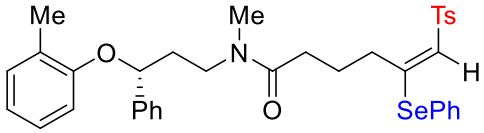


According to **General procedure 3** with methyl (*R*)-*N*-methyl-*N*-(3-phenyl-3-(*o*-tolyloxy)propyl)hex-5-ynamide **S62** (69.8 mg, 0.2 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.4 mmol, 2.0 equiv.) for 72 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **62** as a colorless oil (84.9 mg, 69% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.63 (dd, J = 8.2, 3.4 Hz, 2H), 7.44 – 7.37 (m, 5H), 7.32 (dt, J = 15.2, 7.9 Hz, 5H), 7.25 (d, J = 2.8 Hz, 2H), 7.22 (t, J = 7.3 Hz, 1H), 7.12

(dd, *J* = 12.7, 7.3 Hz, 1H), 6.96 – 6.92 (m, 1H), 6.77 (dt, *J* = 14.7, 7.3 Hz, 1H), 6.58 (t, *J* = 8.4 Hz, 1H), 5.52 (d, *J* = 13.5 Hz, 1H), 5.22 – 5.17 (m, 1H), 3.66 – 3.50 (m, 2H), 2.96 (d, *J* = 16.1 Hz, 3H), 2.89 – 2.75 (m, 2H), 2.39 (d, *J* = 3.0 Hz, 3H), 2.35 (d, *J* = 8.5 Hz, 3H), 2.28 – 2.09 (m, 3H), 2.06 – 1.92 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) *δ*: 171.96, 171.91, 162.3, 155.7, 155.4, 143.8, 143.7, 141.6, 141.0, 139.7, 139.6, 135.5, 130.7, 130.6, 130.30, 130.27, 130.01, 129.99, 129.71, 129.70, 128.8, 128.5, 128.4, 128.3, 127.7, 127.5, 126.9, 126.71, 126.69, 126.67, 126.6, 126.5, 125.7, 125.5, 120.5, 120.2, 119.8, 119.7, 112.7, 112.5, 77.5, 76.5, 46.5, 45.4, 37.6, 36.5, 35.8, 33.4, 32.8, 32.1, 31.41, 31.39, 25.4, 25.1, 21.5, 16.6, 16.5; HRMS (ESI): *m/z* calcd for $C_{36}H_{39}NO_4S_2Na$ $[M+Na]^+$: 636.2218, found: 636.2216.

(*R*,*E*)-*N*-Methyl-*N*-(3-phenyl-3-(*o*-tolyloxy)propyl)-5-(phenylselanyl)-6-tosylhex-5-enamide (63)

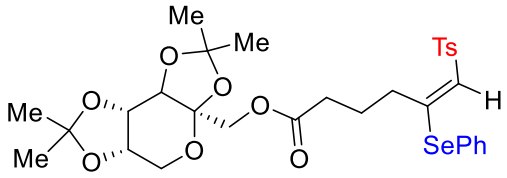


According to **General procedure 3** with methyl (*R*)-*N*-methyl-*N*-(3-phenyl-3-(*o*-tolyloxy)propyl)hex-5-ynamide **S62** (69.8 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (105.8 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **63** as a colorless oil (118.5 mg, 90% yield).

^{1}H NMR (600 MHz, $CDCl_3$) *δ*: 7.63 (dd, *J* = 8.0, 4.1 Hz, 2H), 7.49 (t, *J* = 8.6 Hz, 2H), 7.39 (dd, *J* = 6.7, 4.2 Hz, 1H), 7.36 – 7.27 (m, 7H), 7.25 – 7.21 (m, 2H), 7.11 (dd, *J* = 11.7, 7.5 Hz, 1H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.77 (dt, *J* = 14.3, 7.3 Hz, 1H), 6.58 (t, *J* = 9.1 Hz, 1H), 5.82 (d, *J* = 17.4 Hz, 1H), 5.19 (dd, *J* = 8.6, 4.9 Hz, 1H), 3.57 (dddd, *J* = 26.6, 18.1, 9.7, 4.7 Hz, 2H), 2.95 (d, *J* = 12.8 Hz, 3H), 2.92 – 2.83 (m, 2H), 2.39 (s, 3H), 2.35 (s, 3H), 2.34 (s, 3H), 2.21 – 2.16 (m, 1H), 2.02 – 1.96 (m, 1H), 1.95 – 1.89 (m, 1H), 1.76 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) *δ*: 172.0, 171.9, 160.5, 160.5, 155.7, 155.4, 143.88, 143.85, 141.6, 141.0, 139.33, 139.29, 136.66, 136.65, 131.4, 130.7, 130.6, 130.3, 130.0, 129.87, 129.85, 129.76, 129.75, 128.8, 128.5, 127.9, 127.8, 127.5, 126.9, 126.86, 126.82, 126.68, 126.66, 126.6, 125.9, 125.8, 125.7, 125.6, 123.9,

123.8, 120.5, 120.2, 112.7, 112.5, 77.5, 76.5; HRMS (ESI): *m/z* calcd for $C_{36}H_{39}NO_4SSeNa$ $[M+Na]^+$: 684.1663, found: 684.1668.

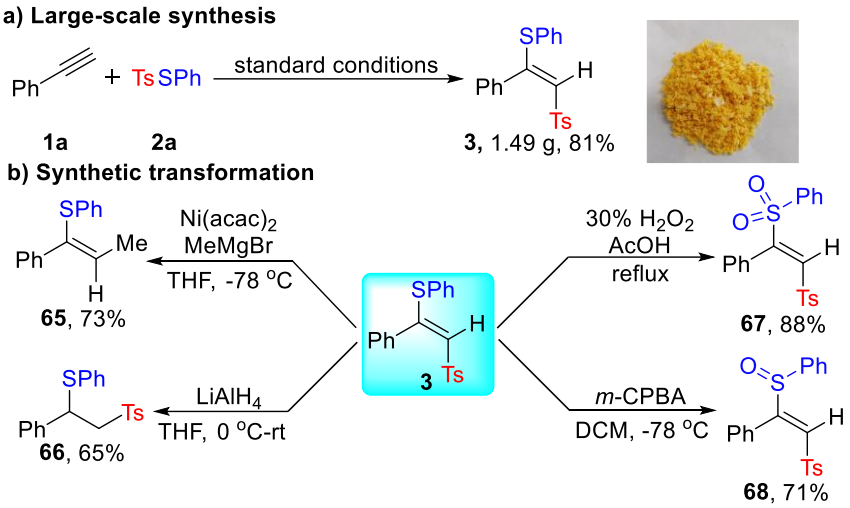
((3a*R*,5a*S*,8a*S*)-2,2,7,7-Tetramethyltetrahydro-3a*H*-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl (*E*)-5-(phenylselanyl)-6-tosylhex-5-enoate (64)



According to **General procedure 3** with methyl ((3a*R*,5a*S*,8a*S*)-2,2,7,7-tetramethyltetrahydro-3a*H*-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl hex-5-ynoate **S64** (70.8 mg, 0.2 mmol, 1.0 equiv.) and *Se*-phenyl 4-methylbenzenesulfonoselenoate **2f** (124.6 mg, 0.4 mmol, 2.0 equiv.) for 17 h, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1) to yield the product **64** as a colorless oil (38.6 mg, 30% yield).

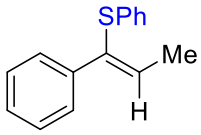
^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.65 (d, J = 8.1 Hz, 2H), 7.51 (d, J = 7.8 Hz, 2H), 7.43 (dd, J = 10.6, 4.1 Hz, 1H), 7.37 (t, J = 7.5 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.86 (s, 1H), 4.61 (dd, J = 7.9, 2.4 Hz, 1H), 4.43 (d, J = 11.7 Hz, 1H), 4.33 (d, J = 2.5 Hz, 1H), 4.25 (d, J = 8.0 Hz, 1H), 4.06 (d, J = 11.7 Hz, 1H), 3.92 (d, J = 13.0 Hz, 1H), 3.77 (d, J = 13.0 Hz, 1H), 2.92 – 2.87 (m, 2H), 2.45 (t, J = 7.5 Hz, 2H), 2.42 (s, 3H), 1.98 (dt, J = 15.2, 7.6 Hz, 2H), 1.55 (s, 3H), 1.49 (s, 3H), 1.42 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 172.1, 159.6, 144.0, 139.3, 136.7, 130.1, 130.0, 129.8, 126.9, 125.8, 124.4, 109.2, 108.8, 101.6, 70.8, 70.5, 70.1, 65.3, 61.3, 33.3, 32.2, 26.5, 26.0, 25.3, 25.0, 24.1, 21.6; HRMS (ESI): *m/z* calcd for $C_{31}H_{38}O_9SSeNa$ $[M+Na]^+$: 689.1299, found: 689.1302.

7. Synthetic application



According to **General procedure 3** with phenylacetylene **1a** (510.6 mg, 5.0 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (2.6 g, 10 mmol, 2.0 equiv.) for 17 h. After completion, the reaction mixture was centrifuged to separate g-C_3N_4 and the liquid mixture. Then, the product **3** was isolated by flash chromatography (petroleum ether/ethyl acetate = 20/1) as a yellow solid (1.49 g, 81% yield).

(*Z*)-Phenyl(1-phenylprop-1-en-1-yl)sulfane (65)

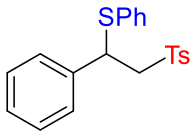


(*E*)-Phenyl(1-phenyl-2-tosylvinyl) sulfane **3** (73.3 mg, 0.2 mmol, 1.0 equiv.) and $Ni(acac)_2$ (5 mg, 0.02 mmol,10 mol%) were sparged with Ar for 5 minutes. Dry THF (6 mL 33 mM) was added and the mixture was cooled down to -78 ℃. Then, methyl magnesium bromide (0.2 mL, [3M] in Et_2O, 3 equiv.) was added slowly to the mixture, and the reaction was stirred at room temperature for 2 h. Next, the reaction mixture was quenched with NH_4Cl and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **65** as a colorless oil (45.3 mg, 73% yield).[19]

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.49 – 7.46 (m, 2H), 7.16 (dd, J = 12.1, 4.8 Hz, 2H), 7.08 (ddt, J = 17.0, 15.3, 7.7 Hz, 5H), 6.97 – 6.94 (m, 1H), 6.45 (q, J = 6.8 Hz, 1H),

2.01 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) *δ*: 140.6, 136.0, 134.6, 134.2, 128.7, 128.1, 127.9, 127.4, 127.3, 125.3, 16.9. The NMR spectra were in accord with that reported in literature.[20]

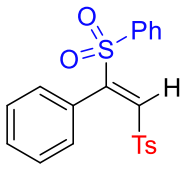
Phenyl(1-phenyl-2-tosylethyl)sulfane (66)



To a solution of (*E*)-phenyl(1-phenyl-2-tosylvinyl) sulfane **3** (73.3mg, 0.2 mmol, 1.0 equiv.) in THF (1 mL) at 0 °C was added a solution of 2.5 M Lithium aluminium hydride solution (0.1 mL, 0.3 mmol, 1.5 equiv.) dropwise. The resulting solution was stirred at rt for 2 h. Upon completion (monitored by TLC), the reaction mixture was quenched with 1 M HCl solution at 0 °C and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether) to yield the product **66** as a colorless oil (47.9 mg, 65% yield).

^{1}H NMR (600 MHz, DMSO-d6) *δ*: 7.53 (d, *J* = 7.5 Hz, 2H), 7.47 – 7.45 (m, 2H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.29 (dd, *J* = 9.4, 6.2 Hz, 2H), 7.19 – 7.12 (m, 5H), 7.05 (ddd, *J* = 7.0, 6.1, 2.1 Hz, 1H), 2.06 (d, *J* = 5.6 Hz, 1H), 1.94 (d, *J* = 5.6 Hz, 1H), 0.99 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d6) *δ*: 143.5, 141.1, 136.5, 130.3, 129.1, 129.0, 128.8, 128.34, 127.0, 126.2, 33.8, 26.6, 25.8.

(*E*)-1-Methyl-4-((2-phenyl-2-(phenylsulfonyl) vinyl)sulfonyl) benzene (67)

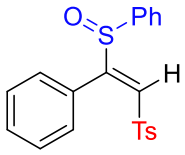


(*E*)-Phenyl(1-phenyl-2-tosylvinyl) sulfane **3** (73.2 mg, 0.2 mmol, 1.0 equiv.) in glacial acetic acid (1.0 mL) were sequentially added to an oven-dried flask equipped with a magnetic stir bar under argon atmosphere with a reflux condenser. The mixture was heated to reflux, and 30% hydrogen peroxide (5 mL) was added slowly. The solution was refluxed for 1 h. After the completion of reaction, the reaction mixture was diluted with water, washed by saturated aq. Na_2CO_3 (10 mL), and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and

concentrated in vacuo. The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to yield the product **67** as a colorless oil (70.1 mg, 88% yield).[21]

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.77 (s, 1H), 7.58 (t, J = 7.4 Hz, 1H), 7.51 – 7.48 (m, 2H), 7.45 (d, J = 8.2 Hz, 2H), 7.40 (t, J = 7.8 Hz, 2H), 7.35 (t, J = 7.5 Hz, 1H), 7.22 – 7.17 (m, 4H), 6.91 (d, J = 7.9 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.5, 145.4, 137.9, 134.4, 130.2, 130.1, 129.8, 129.1, 129.0, 128.2, 127.7, 126.9, 21.7; HRMS (ESI): *m/z* calcd for $C_{21}H_{18}O_4S_2Na$ $[M+Na]^+$: 421.0544, found: 421.0543.

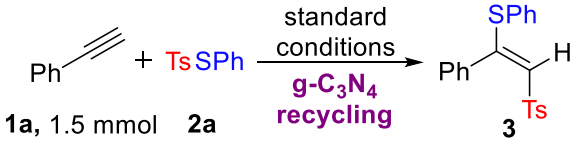
(*E*)-1-Methyl-4-((2-phenyl-2-(phenylsulfonyl) vinyl)sulfonyl)benzene (68)



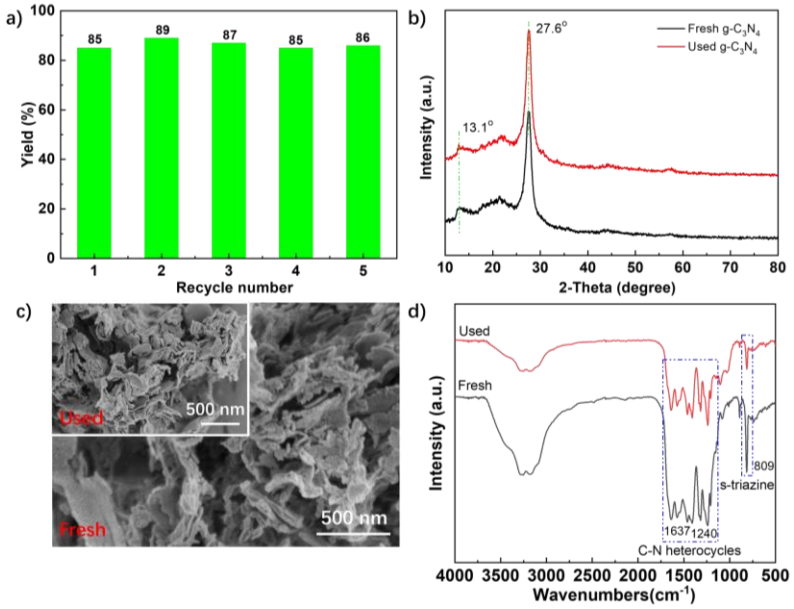
(*E*)-Phenyl(1-phenyl-2-tosylvinyl) sulfane **3** (36.7 mg, 0.1 mmol, 1.0 equiv.) in DCM (1.0 mL) were added to an oven-dried flask equipped with a magnetic stir bar under argon atmosphere. *m*-CPBA (17.3 mg, 0.1 mmol) in DCM (1 mL) was then added dropwise at -78 ℃. The resulting mixture was allowed to warm to room temperature overnight. After the completion of reaction, the reaction mixture was washed by saturated aq. Na_2CO_3 (10 mL), extracted with DCM (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **68** as a colorless oil (27.1 mg, 71% yield).[22]

^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.48 (d, J = 8.2 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 7.30 (t, J = 7.5 Hz, 1H), 7.24 (d, J = 7.6 Hz, 2H), 7.21 – 7.14 (m, 5H), 7.12 (d, J = 8.0 Hz, 2H), 6.84 (d, J = 7.8 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 160.5, 144.9, 140.1, 137.4, 132.0, 130.1, 129.7, 129.6, 129.2, 129.1, 128.1, 128.0, 127.9, 125.3, 21.6; HRMS (ESI): *m/z* calcd for $C_{21}H_{18}O_3S_2Na$ $[M+Na]^+$: 405.0595, found: 405.0592.

Evaluation of g-C_3N_4 recycling.

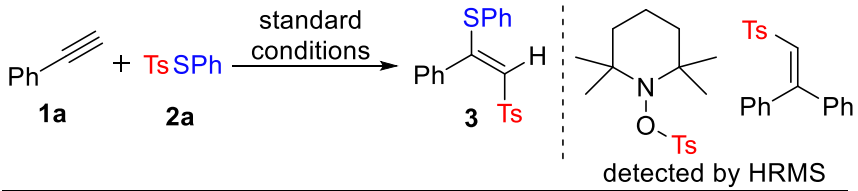


According to **General procedure 3** with phenylacetylene **1a** (153.2 mg, 1.5 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (793.0 mg, 3 mmol, 2.0 equiv.) for 17 h. After completion, the reaction mixture was centrifuged to separate g-C_3N_4 and the liquid mixture. Then, the product was isolated by flash chromatography (petroleum ether/ethyl acetate = 20/1) as a yellow solid. The catalyst g-C_3N_4 was thoroughly washed 3 times with DCM and dried in an oven at 60 °C for overnight and reused in the subsequent recycling reaction.



8. Mechanistic Studies

8.1 Active species trapping experiment



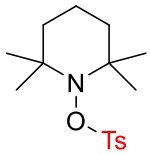
Qunencher	Quenching group	Yield (%)
TEMPO	Free radicals	0
Ethene-1,1-diyldibenzene	Free radicals	0
$HCOONH_4$	Holes	81
CCl_4	e^-	83

According to **General procedure 3** with thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.), g-C_3N_4 (5 mg) and TEMPO (31.2 mg, 0.2 mmol, 2.0 equiv.) or ethene-1,1-diyldibenzene (36.0 mg, 0.2 mmol, 2.0 equiv.) and phenylacetylene **1a** (10.2 mg, 0.1 mmol, 1.0 equiv.) for 17 h. After the completion of reaction, the reaction

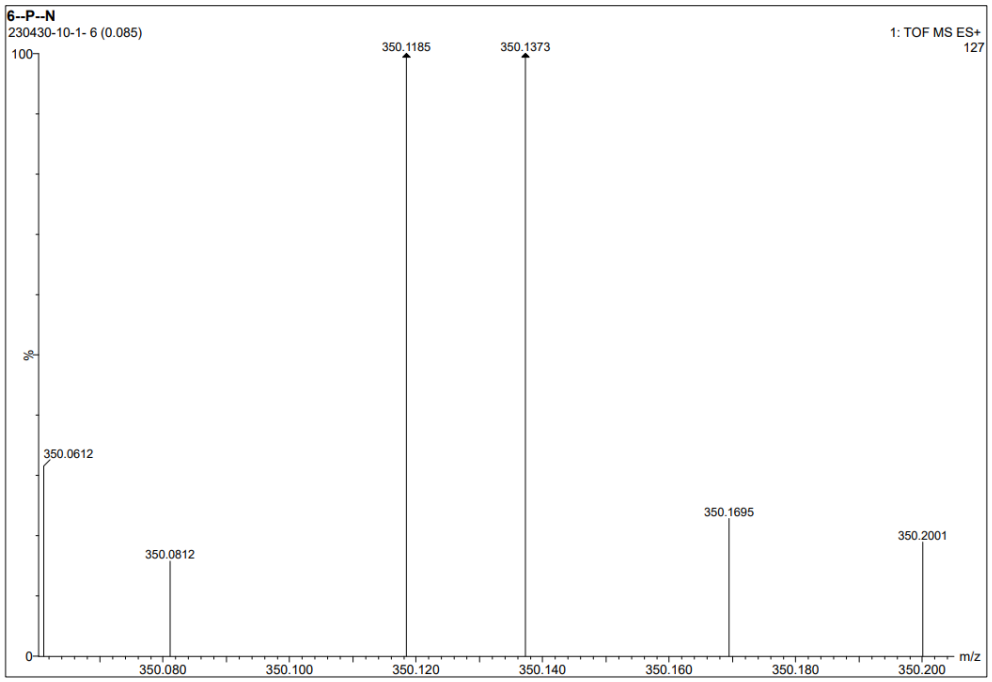
mixture was filtered and the precipitate was washed by ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The filtrate was removed by rotary evaporator under vacuum and the residue was analyzed by HRMS. **3** was not detected in the reaction mixture.

According to **General procedure 3** with thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.), g-C_3N_4 (5 mg) and $HCOONH_4$ (12.6 mg, 0.2 mmol, 2.0 equiv.) or CCl_4 (30.8 mg, 0.2 mmol, 2.0 equiv.) and phenylacetylene **1a** (10.2 mg, 0.1 mmol, 1.0 equiv.) for 17 h. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel to afford the pure product **3**.

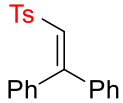
2,2,6,6-Tetramethylpiperidin-1-yl 4-methylbenzenesulfonate



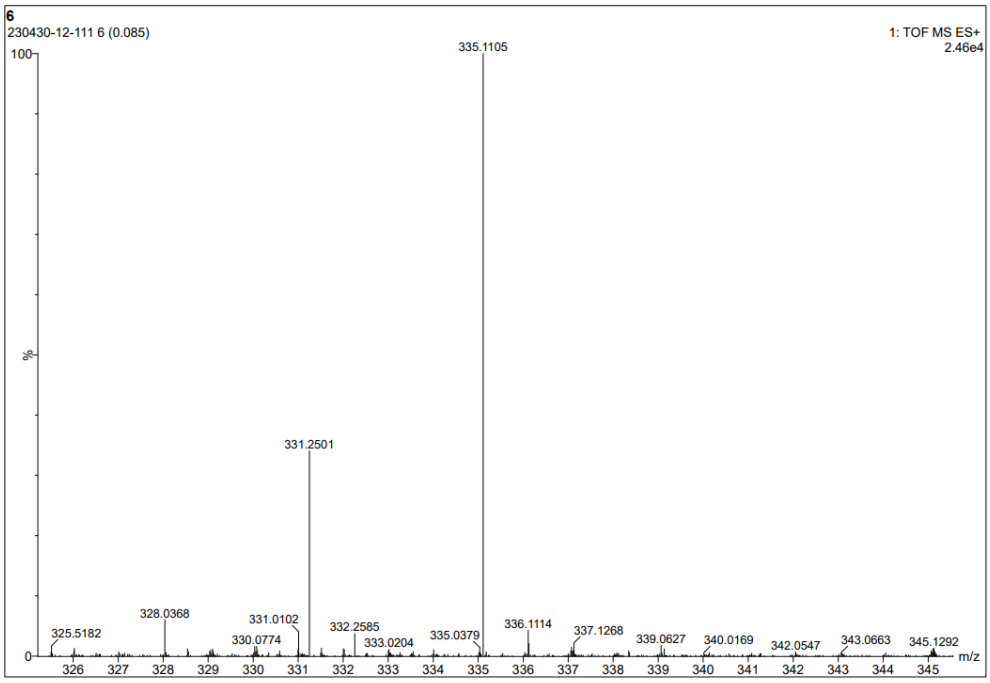
HRMS (ESI): *m/z* calcd for $C_{16}H_{25}NO_3SK$ $[M+K]^+$: 350.1192, found: 350.1185.



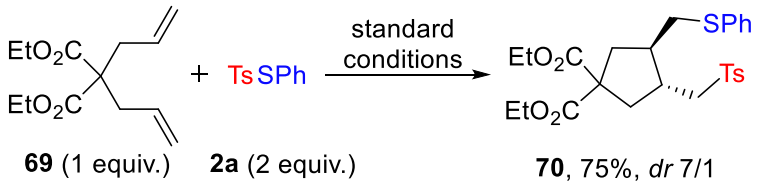
(2-Tosylethene-1,1-diyl)dibenzene



HRMS (ESI): *m/z* calcd for $C_{21}H_{19}O_2S$ $[M+H]^+$: 335.1106, found: 335.1105.

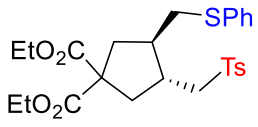


8.2 Radical clock experiment



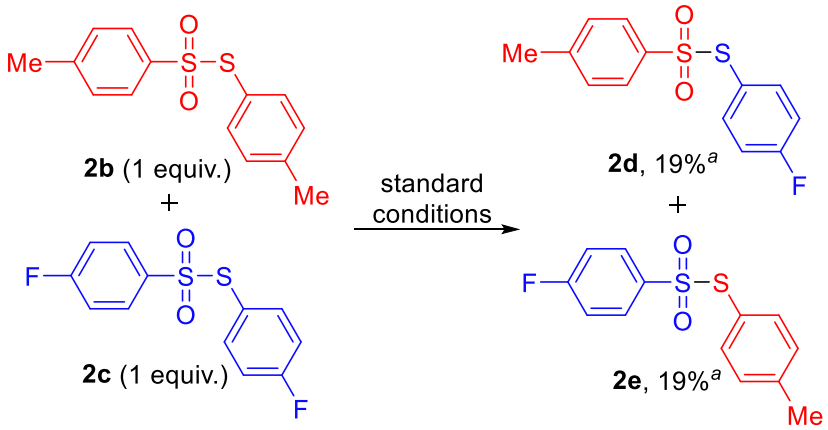
According to **General procedure 3** with thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.), g-C_3N_4 (5 mg) and diethyl diallylmalonate (24 mg, 0.1 mmol 1.0 equiv.) for 17 h. Upon completion (monitored by TLC), the reaction mixture was filtered. The filtrate was transferred to a separate funnel using 20 mL ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product as a colorless oil (37.8 mg, 75% yield).

Diethyl(3*R*,4*R*)-3-((phenylthio)methyl)-4-(tosylmethyl)cyclopentane-1,1-dicarboxylate (70)



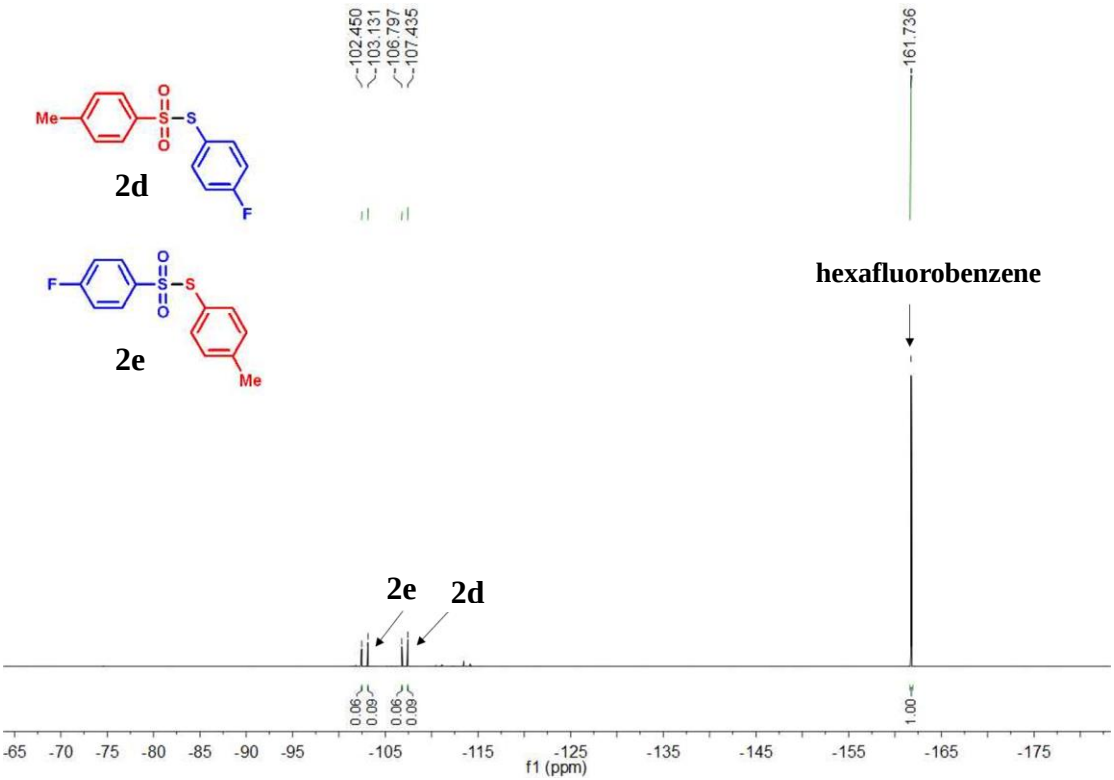
^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.78 (d, J = 7.3 Hz, 2H), 7.35 (d, J = 7.6 Hz, 2H), 7.30 – 7.23 (m, 4H), 7.20 – 7.16 (m, 1H), 4.20 – 4.12 (m, 4H), 3.23 (dd, J = 13.9, 3.9 Hz, 1H), 3.10 (dd, J = 13.7, 9.5 Hz, 1H), 2.89 (dd, J = 12.5, 6.1 Hz, 1H), 2.74 – 2.67 (m, 1H), 2.61 – 2.48 (m, 2H), 2.47 – 2.41 (m, 4H), 2.40 – 2.32 (m, 2H), 2.29 (dd, J = 13.6, 6.5 Hz, 1H), 1.27 – 1.18 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ: 172.3, 171.8, 144.7, 136.4, 135.4, 129.9, 129.7, 129.5, 128.94, 128.90, 128.0, 127.9, 126.3, 61.8, 61.60, 61.55, 60.3, 58.8, 58.3, 55.8, 44.0, 41.2, 39.6, 38.9, 38.8, 38.0, 37.9, 37.5, 36.4, 34.0, 21.6, 13.92, 13.91. The NMR spectra were in accord with that reported in literature.[23]

8.3 Cross-over experiment

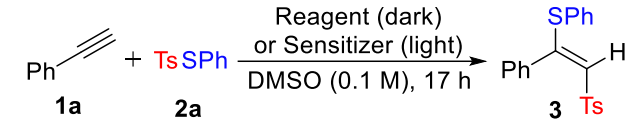


Following the **General procedure 3**, thiosulfonates **2b** (27.8 mg, 0.1 mmol, 1 equiv.), **2c** (28.6 mg, 0.1 mmol, 1 equiv.) and g-C_3N_4 (5 mg) in DMSO (1 mL) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 17 h. Upon completion (monitored by TLC), the reaction mixture was filtered. The filtrate was analyzed by ^{19}F NMR with hexafluorobenzene as internal standard. **2d** and **2e** of thiosulfonates were obtained in 19 % yield.[23]

^{19}F NMR (565 MHz, $CDCl_3$) δ: -102.45, -103.13, -106.80, -107.43, -161.74; HRMS (ESI): m/z calcd for $C_{13}H_{11}O_2S_2FNa$ $[M+Na]^+$: 305.0082, found: 305.0083.



8.4 Control experiment

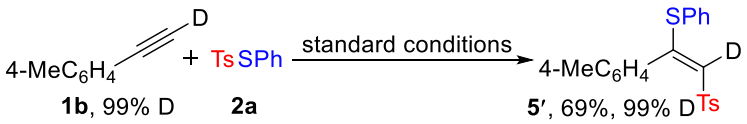


Electron transfer reagent: SmI_2, NR; Zn, NR; CAN, trace;
Triplet sensitizer: Benzil, 57%

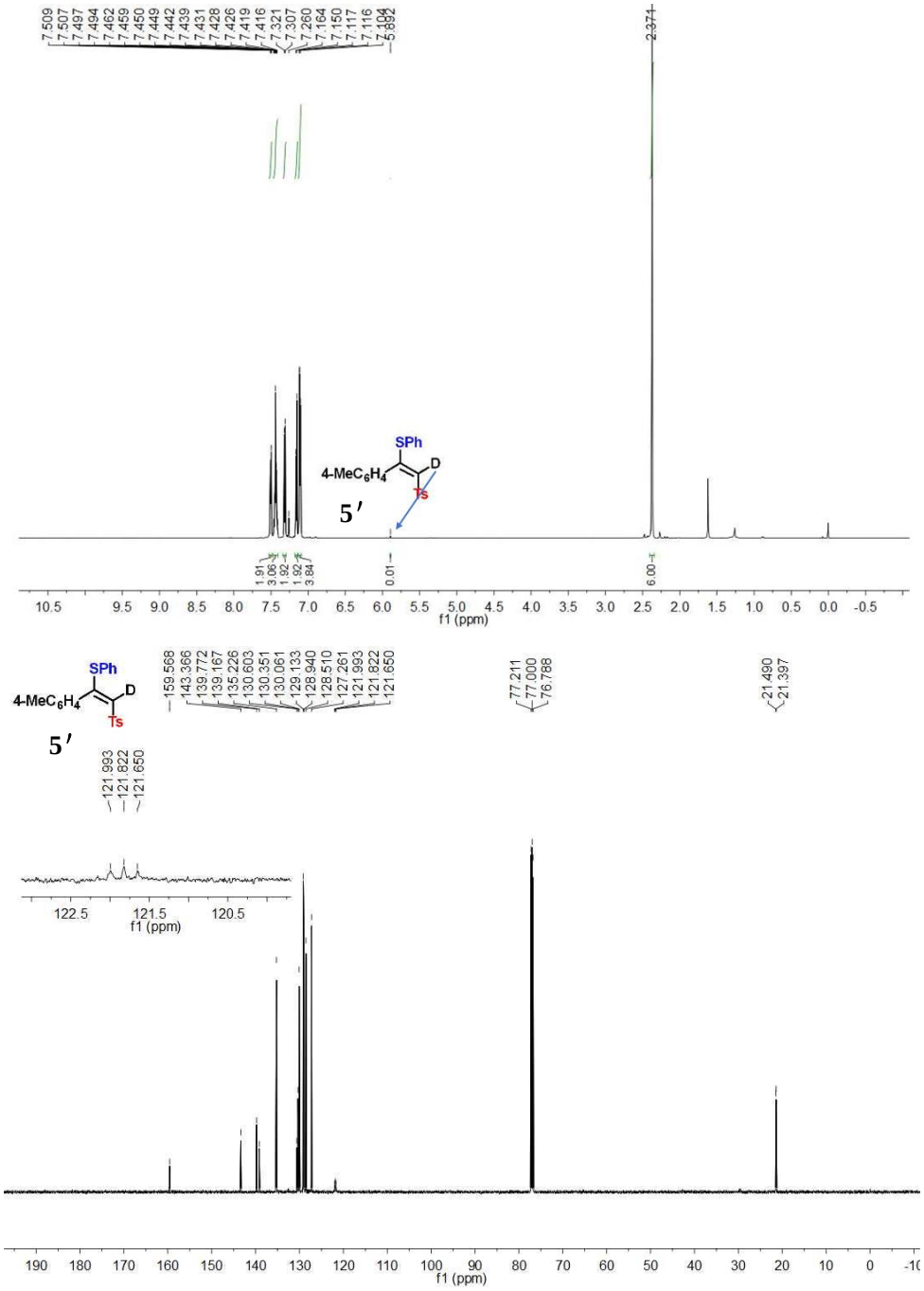
Electron transfer reagent: Phenylacetylene **1a** (10.2 mg, 0.1 mmol, 1.0 equiv.), thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.) and reagent (1 equiv.) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere in DMSO (1 mL) at rt. The reaction was stirred for 17 h under dark.

Triplet sensitizer: Phenylacetylene **1a** (10.2 mg, 0.1 mmol, 1.0 equiv.), thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.) and benzil (1 equiv.) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere in DMSO (1 mL) at rt under blue LED (7 W) irradiation for 17 h. The reaction mixture was purified by flash column chromatography on silica gel to afford the pure product.

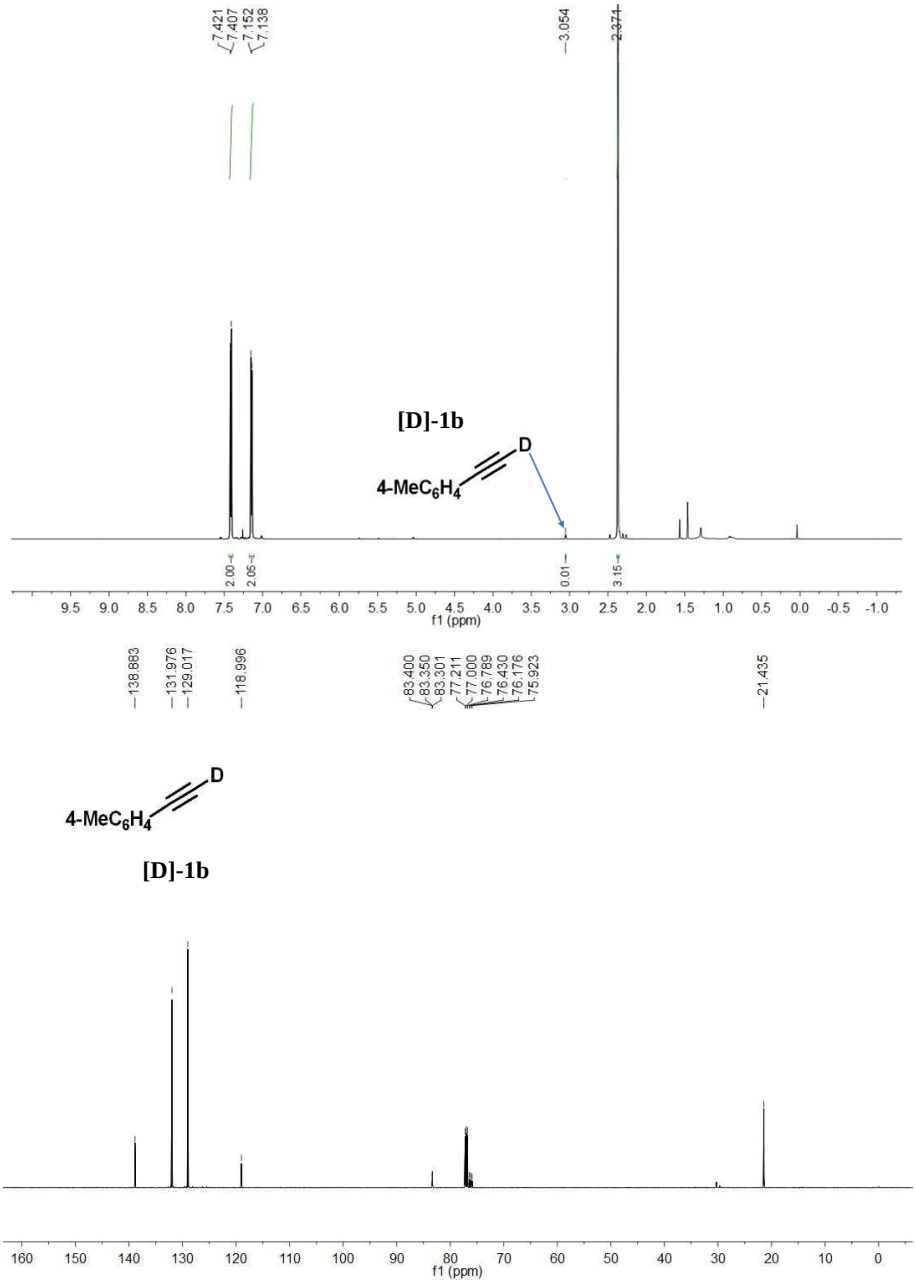
8.5 Deuterium labeling experiment

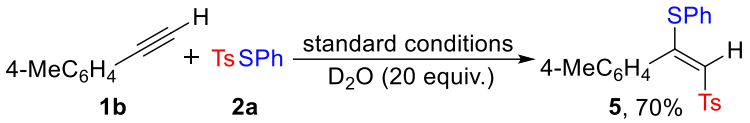


According to **General procedure 3** with thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.), g-C_3N_4 (5 mg) and deuterated alkyne **[D]-1b** (11.7 mg, 0.1 mmol 1.0 equiv.) for 17 h. Upon completion (monitored by TLC), the reaction mixture was filtered. The filtrate was transferred to a separate funnel using 20 mL ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product as a colorless oil (26.3 mg, 69% yield).

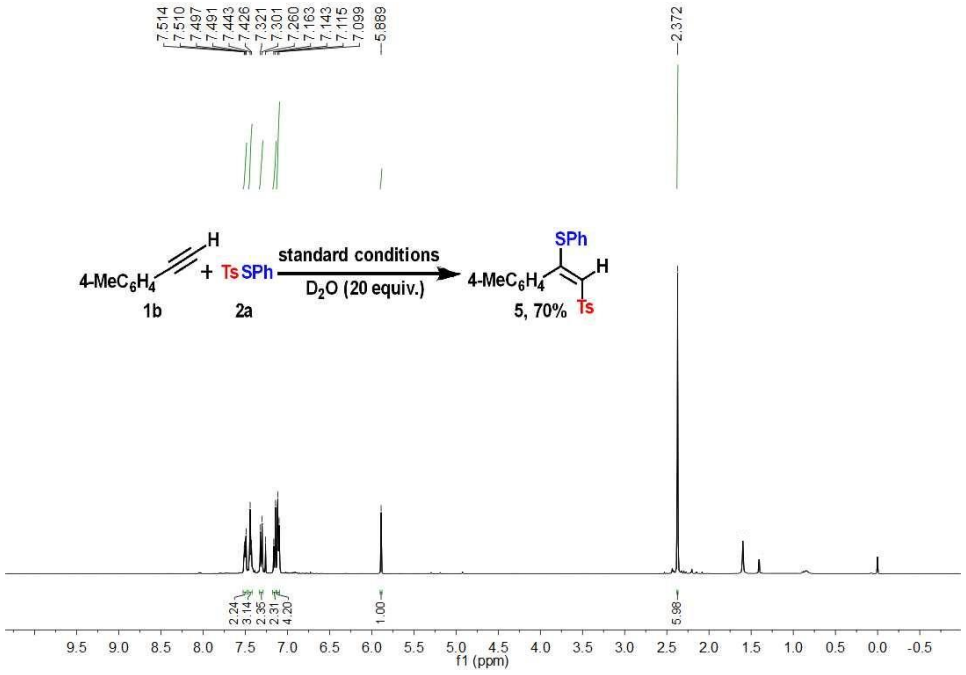


Synthesis of deuterated alkyne [D]-1b: a solution of 1-ethynyl-4-methylbenzene (1.00 mmol, 116.2 mg) in THF (5 mL) was treated with *n*-butyllithium 2.5 M in hexane (0.440 mL, 1.10 mmol) at -78 °C for 30 min and then warmed up to room temperature until total consumption of the starting material. Then, the reaction was quenched with D_2O (100%). Diethyl ether was added and the organic layer was separated, dried over $MgSO_4$ and the solvent was removed under vacuum. The relative integrated intensities of the phenyl protons to the methylidyne protons, which had been 4.0 to 1.0 in the starting phenylacetylene, were now 4.0 to 0.01, indicating 99% deuteriation at the CH group and thus a 99% yield of **[D]-1b**.





According to **General procedure 3** with thiosulfonates **2a** (52.9 mg, 0.2 mmol, 2 equiv.), g-C_3N_4 (5 mg), alkyne **1b** (11.6 mg, 0.1 mmol 1.0 equiv.) and D_2O (40.1 mg, 2 mmol, 20 equiv.) for 17 h. Upon completion (monitored by TLC), the reaction mixture was filtered. The filtrate was transferred to a separate funnel using 20 mL ethyl acetate and 30 mL water and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1) to afford the pure product as a colorless oil (26.6 mg, 70% yield).



8.6 Quantum yield measurement

Determination of the light intensity at 450 nm:

The photon flux of the LED (λ_{max} = 450 nm) was determined by ferrioxalate actinometry similar to a procedure by Yoon.[24] First, a 10 mL 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution). A 20 mL buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (20 mg) and sodium acetate (4.5 g) in H_2SO_4 (20 mL of a 0.5 M solution). Both solutions were

stored in the dark. To determine the photon flux of the LED, 2.00 mL of the ferrioxalate solution was placed in a cuvette and irradiated for 90 seconds at 450 nm. After irradiation, the phenanthroline solution (0.35 mL) was added to the cuvette, and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate with phenanthroline. The absorption of the solution was measured at 510 nm. A non-irradiated sample was also prepared identically and the absorption at 510 nm was also measured.

Conversion was calculated using equation 1.

$$\text{mol } Fe^{2+} = \frac{V \bullet \Delta A(510\text{ nm})}{1 \bullet \varepsilon} \quad (1)$$

Where V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.0 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11100 L $mol^{-1}cm^{-1}$).

The value of mol Fe^{2+} was 1.93 x 10^{-7} mol.

The photon flux can be calculated using equation 2.

$$\text{Photon flux} = \frac{\text{mol}Fe^{2+}}{\Phi \bullet t \bullet f} \quad (2)$$

Where Φ is the quantum yield for the ferrioxalate actinometer (0.85 at 450 nm), t is the irradiation time, and f is the fraction of light absorbed at λmax = 450 nm by the ferrioxalate actinometer. This value is calculated using equation 3 where A is the absorbance of the ferrioxalte solution at 450 nm. An absorption spectrum gave an A value of > 3, indicating that the fraction of absorbed light (f) is > 0.999.

$$f = 1 - 10^{-A(450\text{ nm})}$$

The photon flux was calculated to be 2.52 × 10^{-9} einsteins s^{-1}

Determination of the reaction quantum yield

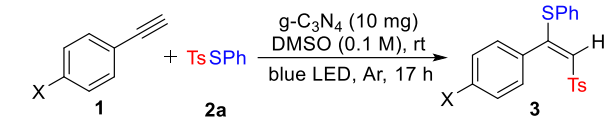
Phenylacetylene **1a** (20.4 mg, 0.20 mmol, 1.0 equiv.) and *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.40 mmol, 2.0 equiv.) and g-C_3N_4 (10 mg) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar in DMSO (2 mL) at rt. The reaction vial was sealed, evacuated and backfilled three times with argon, then irradiated at 450 nm at ambient temperature for 90 minutes using the same setup as for the photon flux determination. Then, the NMR

yield was determined (25%) using CH_2Br_2 as internal standard. The reaction quantum yield was determined using equation 4, where photon flux was determined as above described, t is the reaction time, f is the fraction of incident light absorbed by the reaction mixture. This value is calculated using equation 3 where A is the absorbance of the reaction mixture at 450 nm. An absorption spectrum gave an A (450 nm) value of > 3, indicating that the fraction of absorbed light (f) is > 0.999.

$$\Phi = \frac{\text{mol of product formed}}{\text{photo flux} \bullet t \bullet f} \tag{4}$$

The reaction quantum yield (Φ) was determined to be 3.67, which is above unity, indicating that a free radical chain mechanism might be involved in the reaction.

8.7 Hammett plot analysis



X = Me, H, F, Cl ,CF_3

Compound **1** (0.2 mmol, 1.0 equiv.), *S*-phenyl 4-methylbenzenesulfonothioate **2a** (105.8 mg, 0.40 mmol, 2.0 equiv.) and g-C_3N_4 (10 mg) and DMSO (2 mL) were added to a group of 10 mL glass vials (5 intotal) at room temperature. The vials were purged with Ar and sealed with PTFE cap. This reaction mixture was irradiated at 450 nm at ambient temperature. One of the reaction vials was quenched by Et_2O every 30 minutes. Then, the reaction mixture was filtered. The filtrate was transferred to a separate funnel using 20 mL Et_2O and 30 mL water and extracted with Et_2O (3 x 20 mL). The combined organic layers were dried with over Na_2SO_4 and concentrated in vacuo. All samples were analyzed by ^1H NMR (CH_2Br_2 as the internal standard) to determine the yields. The kinetic rate was plotted for each aryl alkynes substrate (Figure S2), the slopes of which were used as the rate constant to calculate the $lg(K_X/K_H)$ (Table S6).

Figure S2. Reaction profiles of the catalyst system

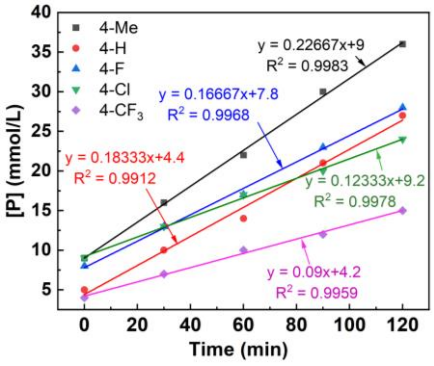
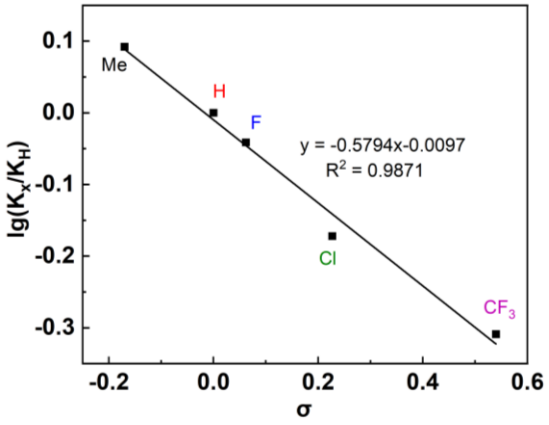


Table S6 Kinetic data of the catalyst system

Entry	σ	K_X	$lg(K_X/K_H)$
p-CH_3	-0.17	0.22667	0.092160
H	0	0.18333	0
p-F	0.062	0.16667	-0.041376
p-Cl	0.227	0.12333	-0.172165
p-CF_3	0.54	0.09	-0.308991

The $lg(K_X/K_H)$ for each aryl alkynes was then plotted against the corresponding σ value reported by the literature (Figure S3).[25] A linear correlation with slope (ρ = -0.58) was observed in the Hammett plot of our reaction system, which indicated that (1) the electron-deficient alpha-alkenyl radical was formed; (2) this was a stepwise reaction and the radical addition to the triple bond was the rate-determining step.

Figure S3. Hammett plot of *para*-substituted aryl alkynes



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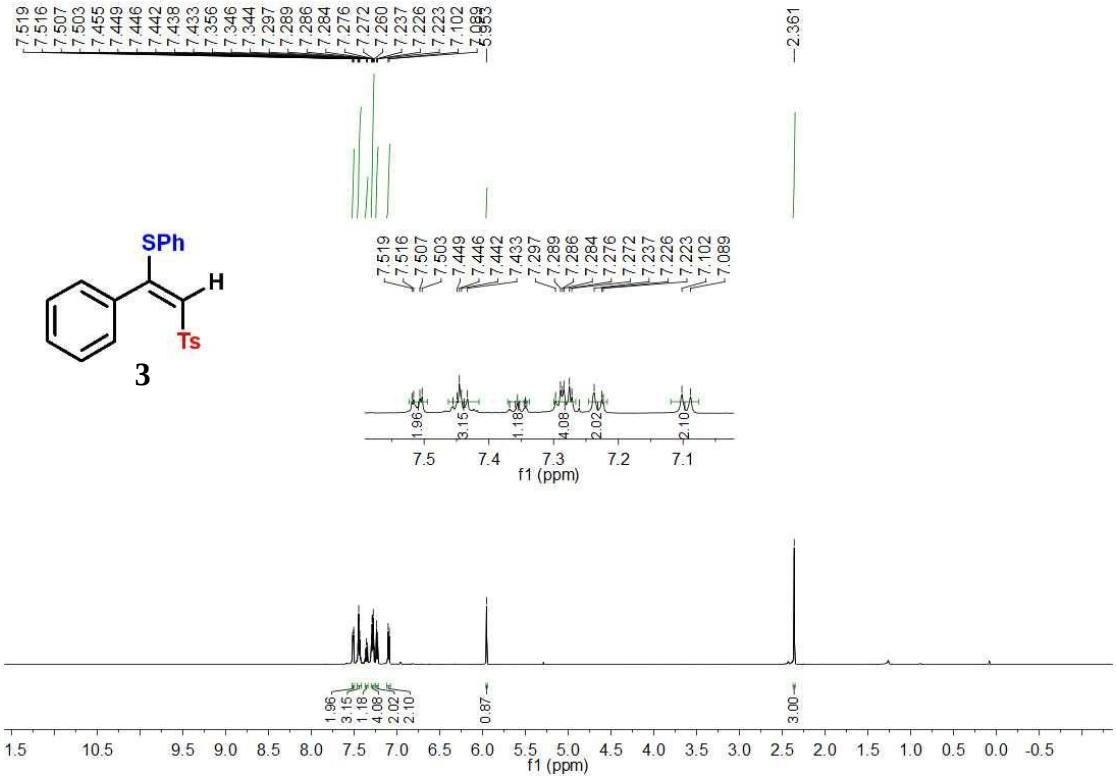
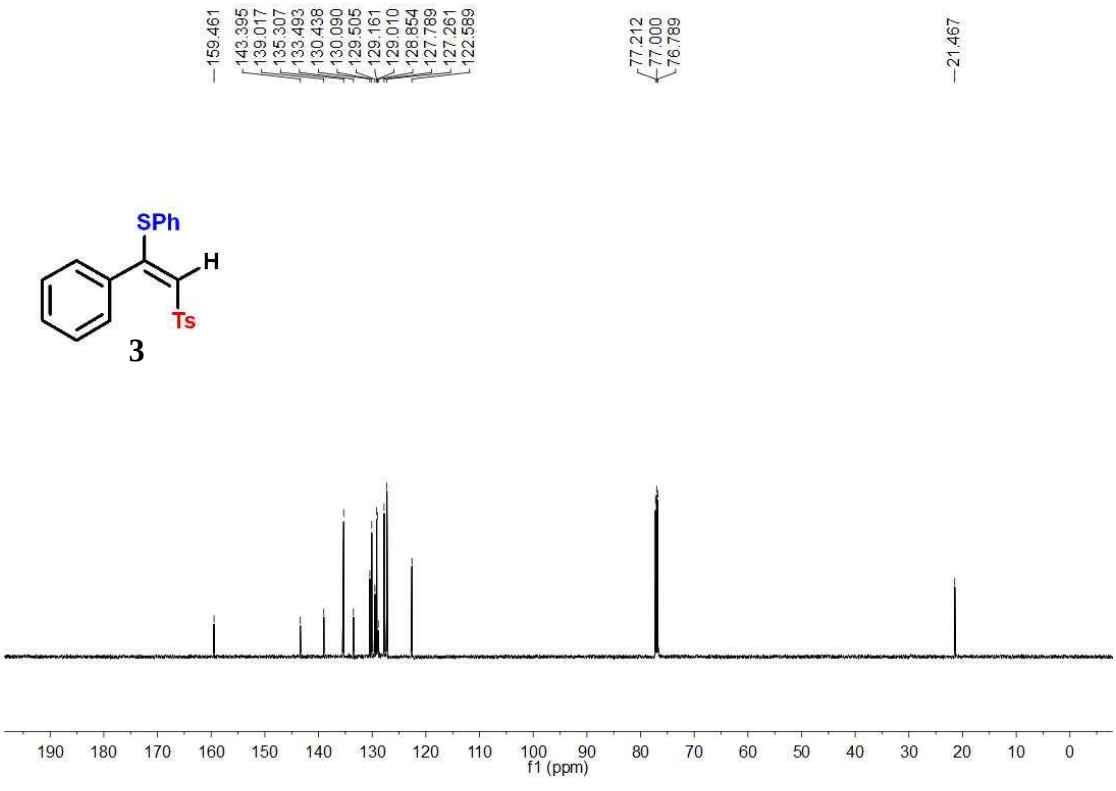
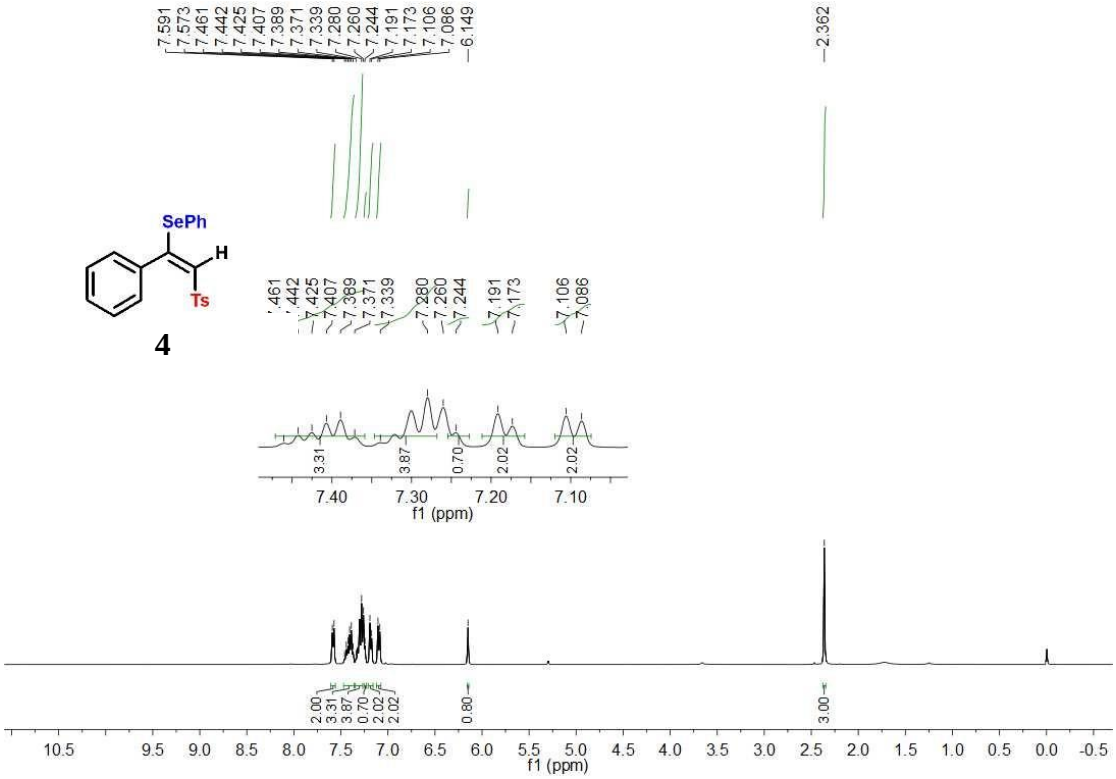
10. NMR Spectra



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7.173
7.106
7.086
6.149
2.362
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H
Ts
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3.87
0.70
2.02
2.02
7.40
7.30
7.20
7.10
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3.87
0.70
2.02
2.02
0.80
3.00
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8.0
7.5
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6.5
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5.0
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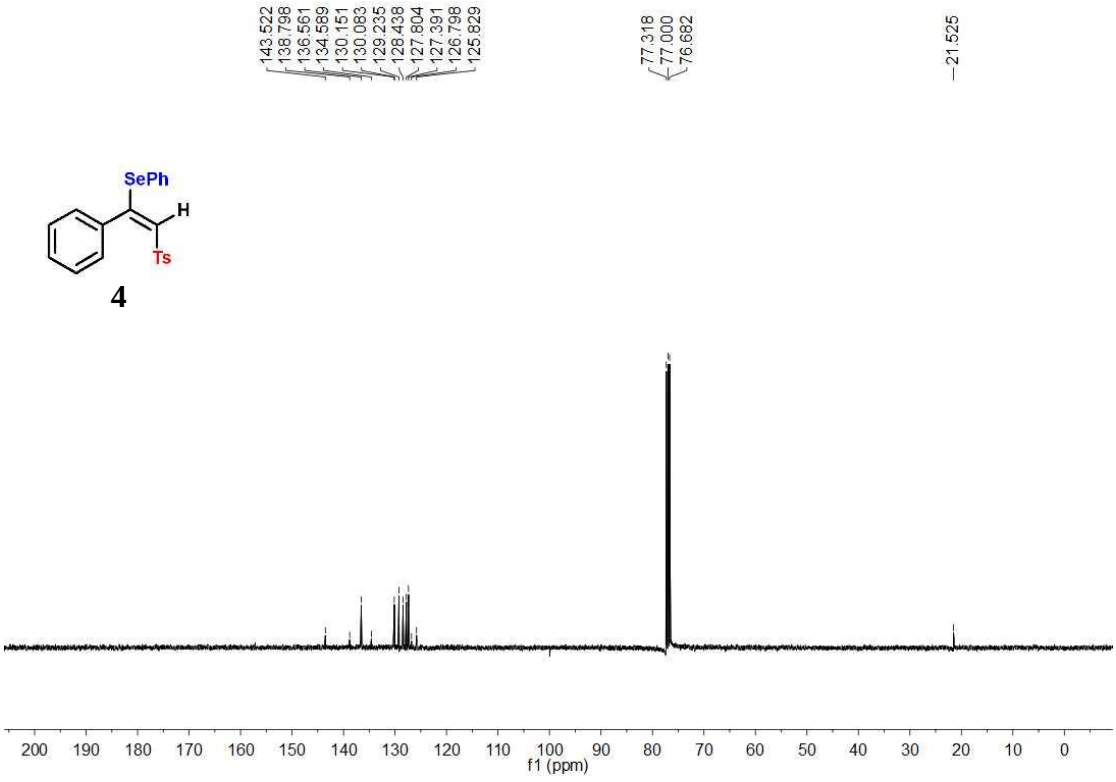
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77.000
76.682
21.525
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H
Ts
4
200
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180
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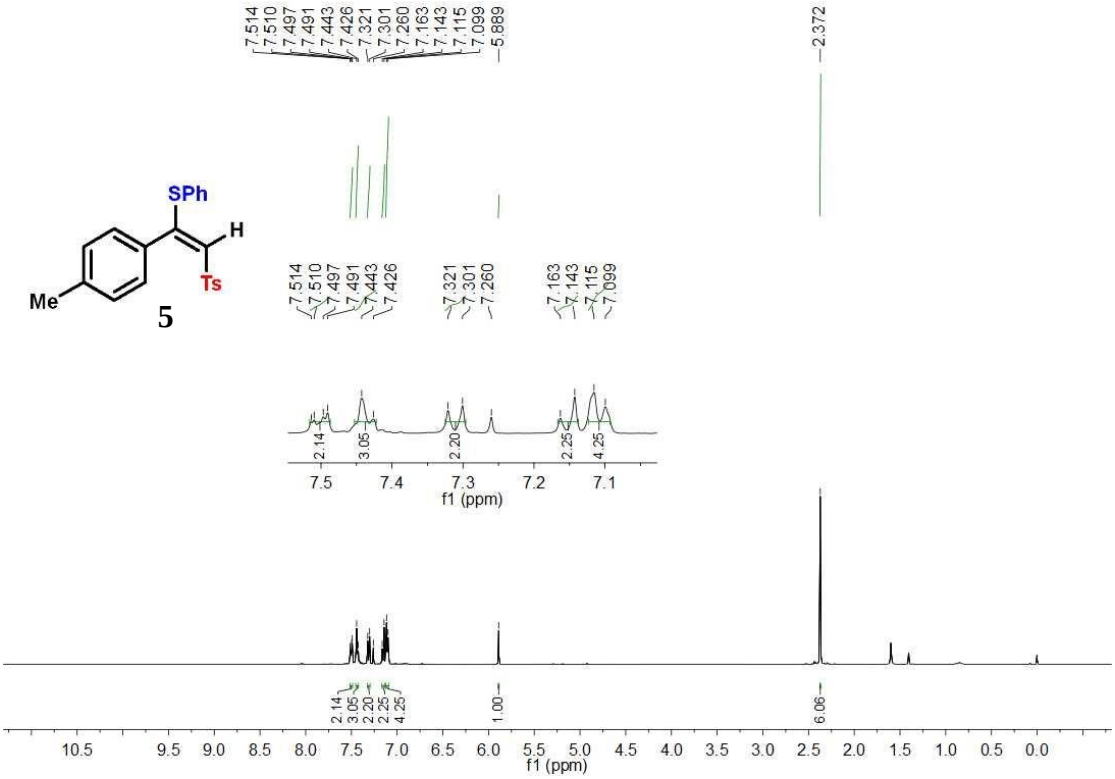
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5.889
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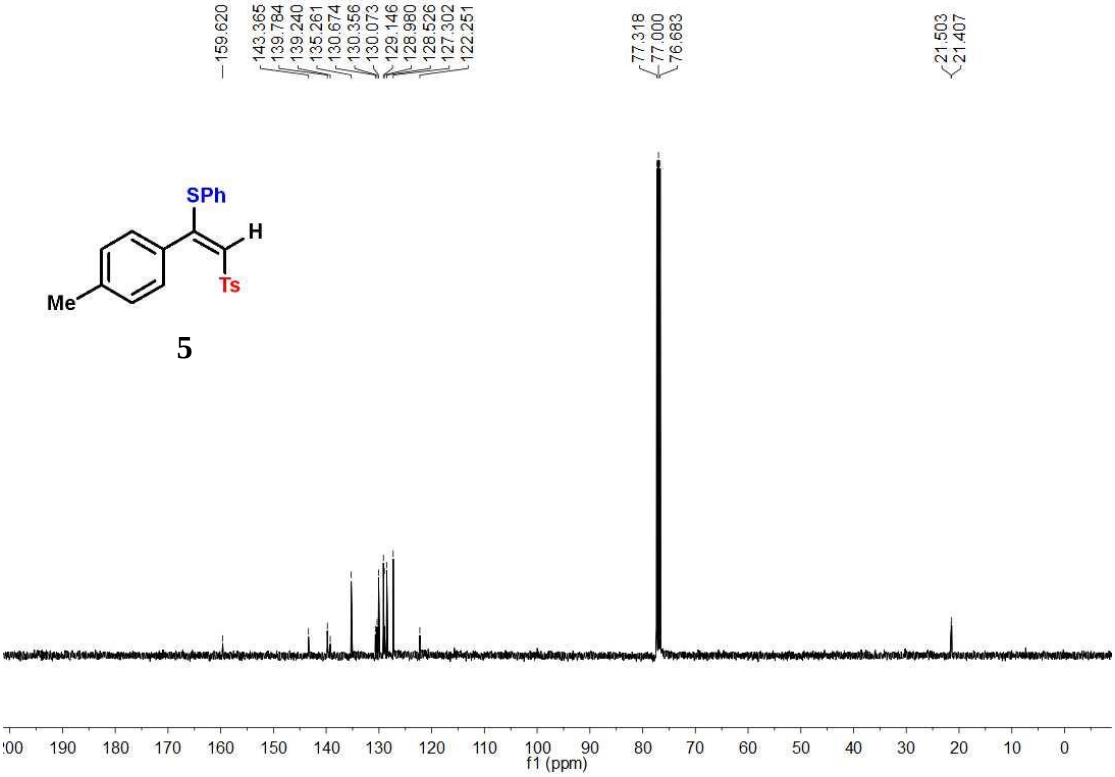
f1 (ppm)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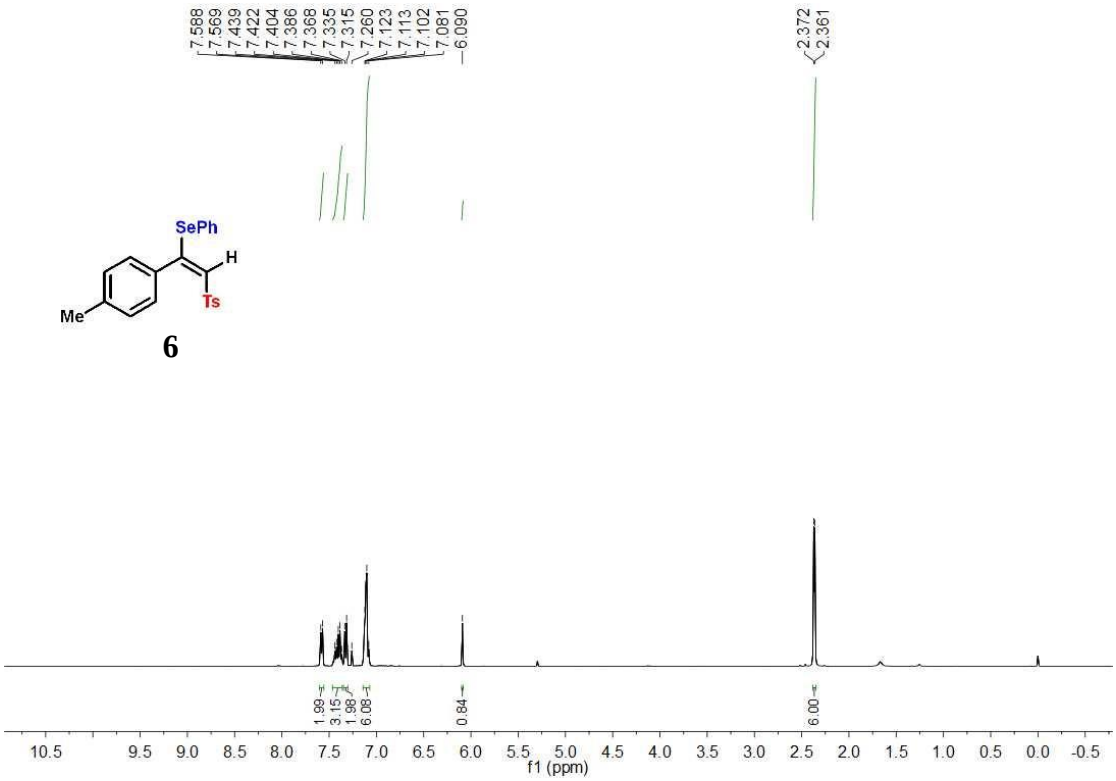
SPh
H
Ts
Me
5
f1 (ppm)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7.102
7.081
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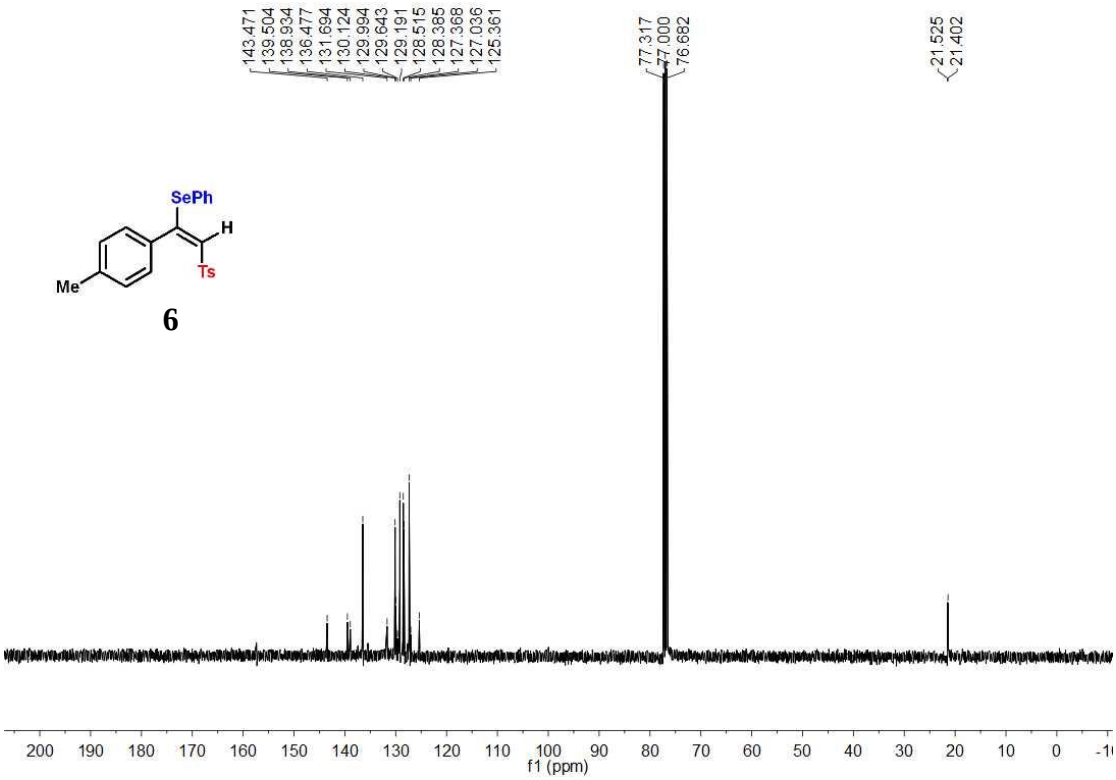
6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Ts
Me
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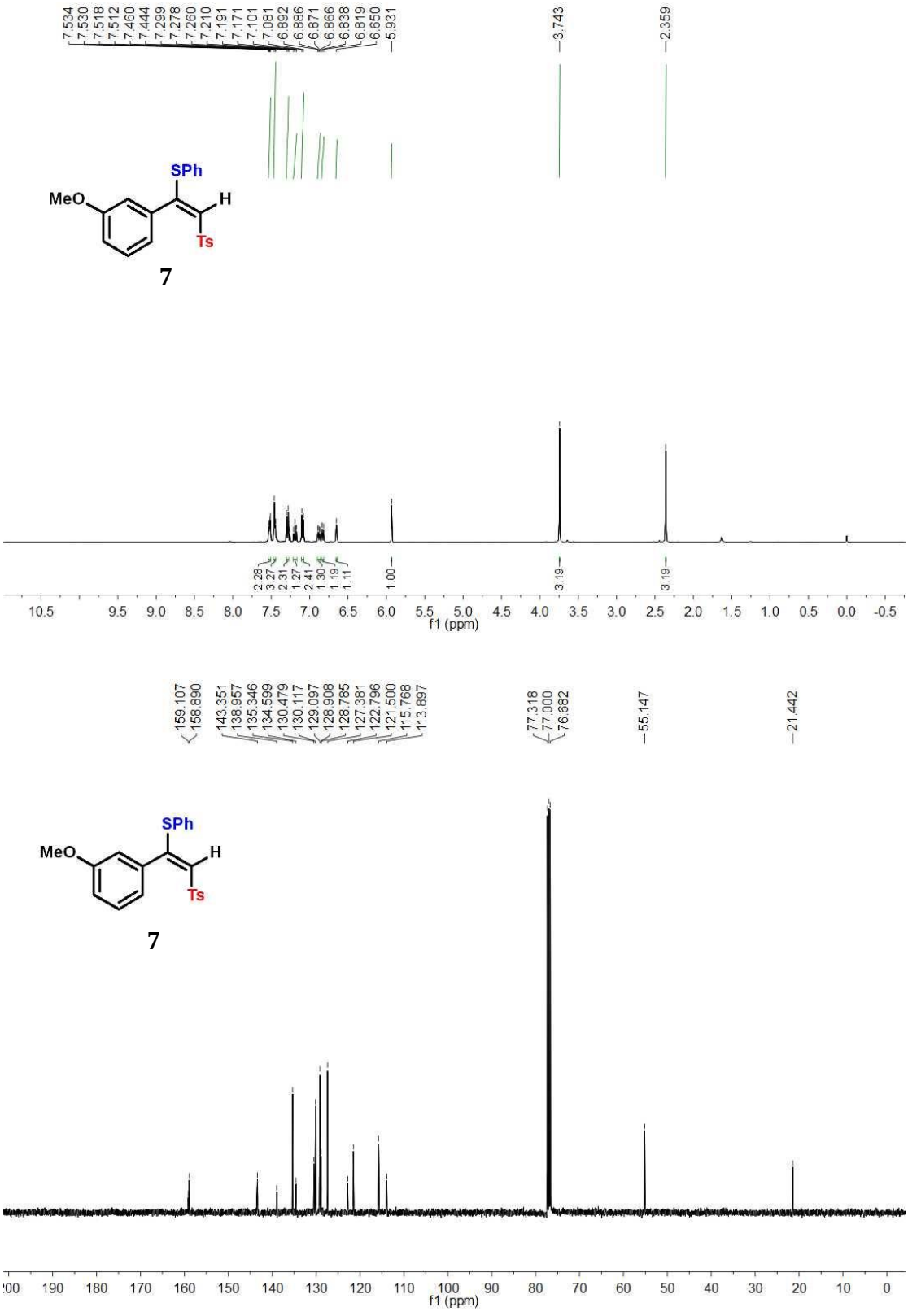
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6.819
6.650
5.931
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MeO
H
Ts
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3.27
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1.27
2.41
1.30
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1.11
1.00
3.19
3.19
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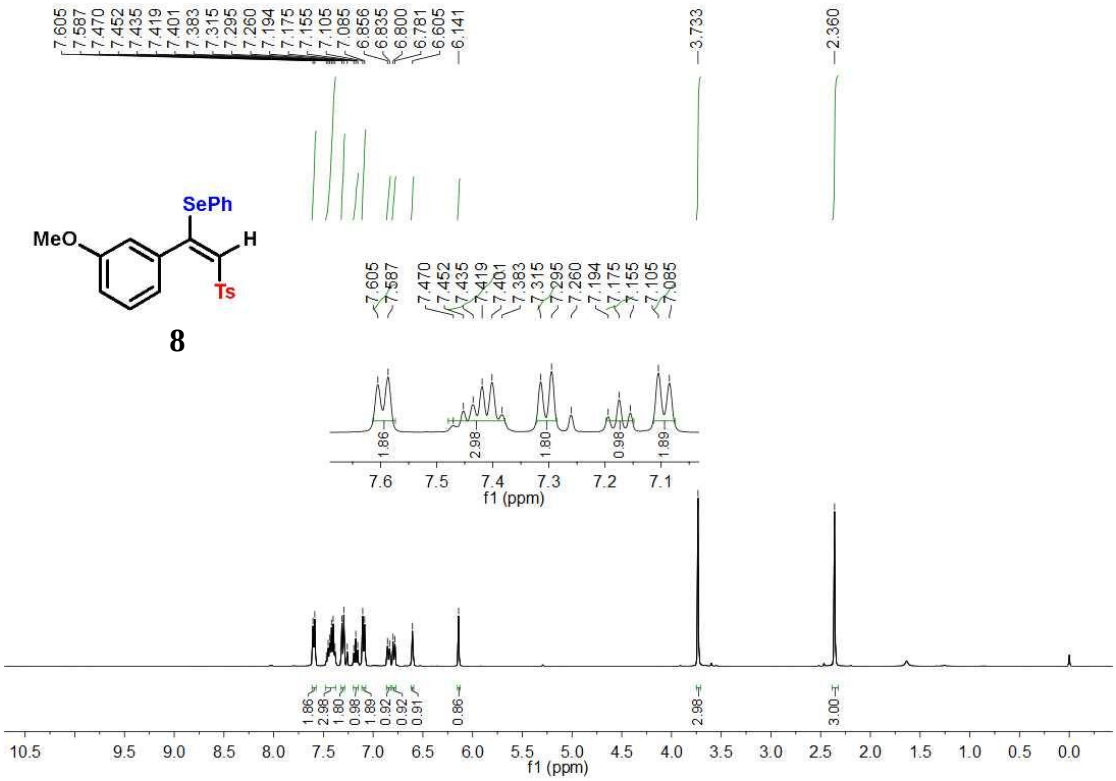
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0.92
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6.5
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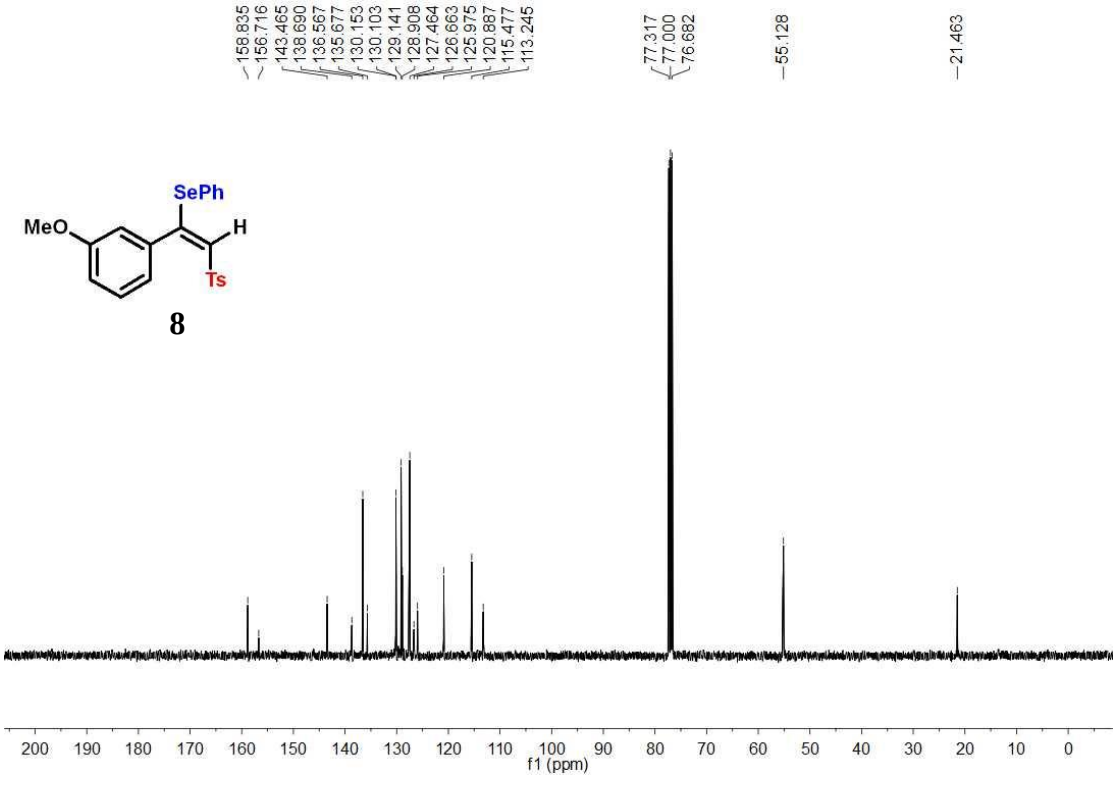
f1 (ppm)

8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SePh
MeO
H
Ts
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180
170
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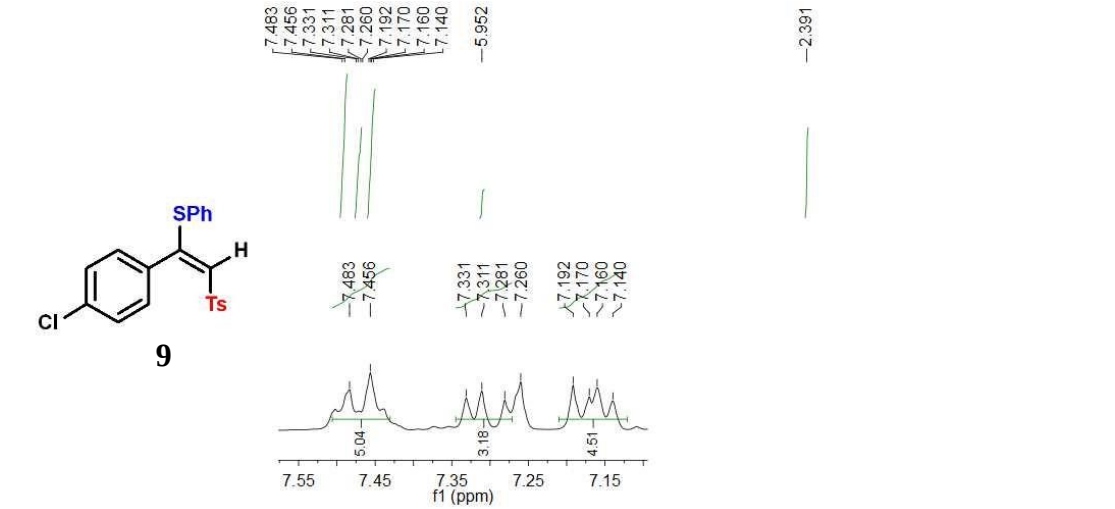
8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SPh
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Ts
Cl
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3.18
4.51
7.55
7.45
7.35
7.25
7.15
f1 (ppm)
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3.18
4.51
1.00
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8.0
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6.0
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2.5
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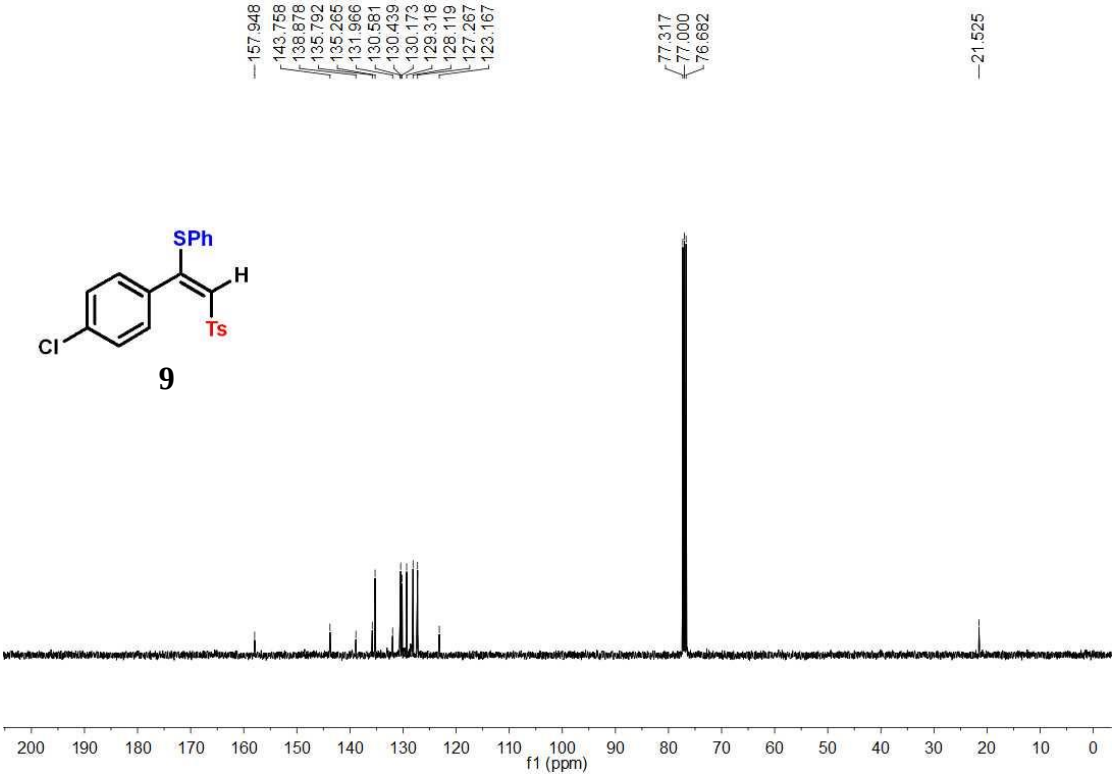
0.0
f1 (ppm)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123.167
77.317
77.000
76.682
21.525
SPh
H
Ts
Cl
9
200
190
180
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90
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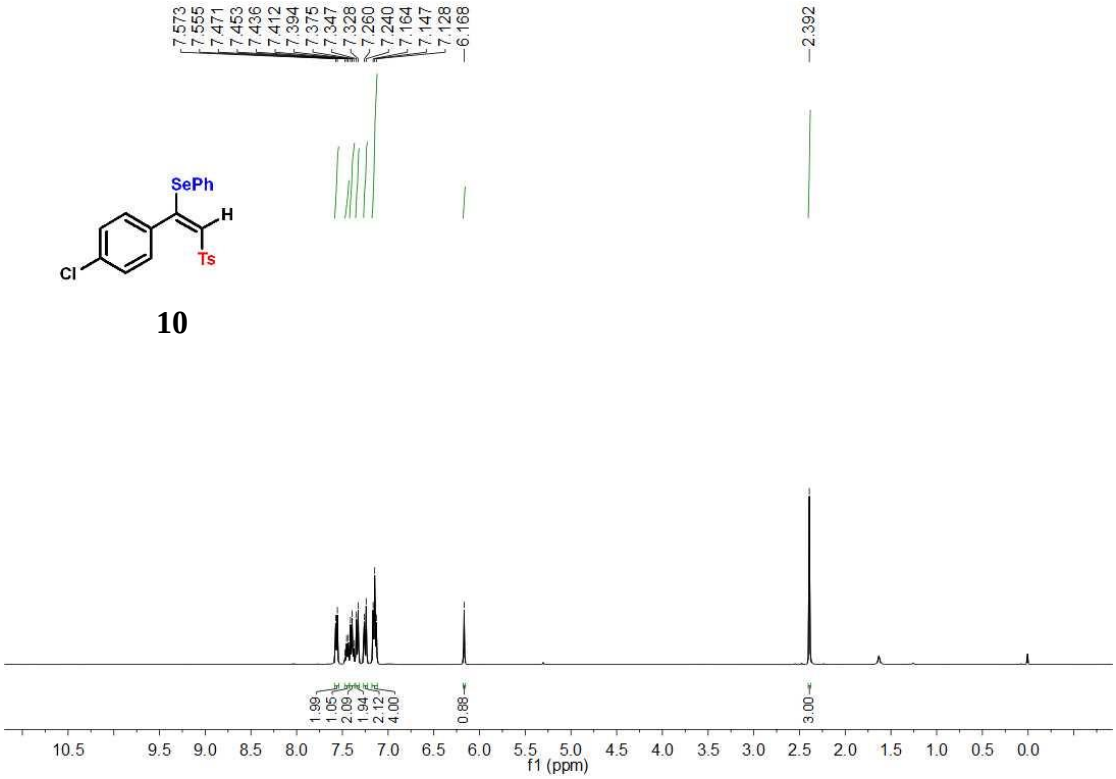
f1 (ppm)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7.164
7.147
7.128
6.168
2.392
SePh
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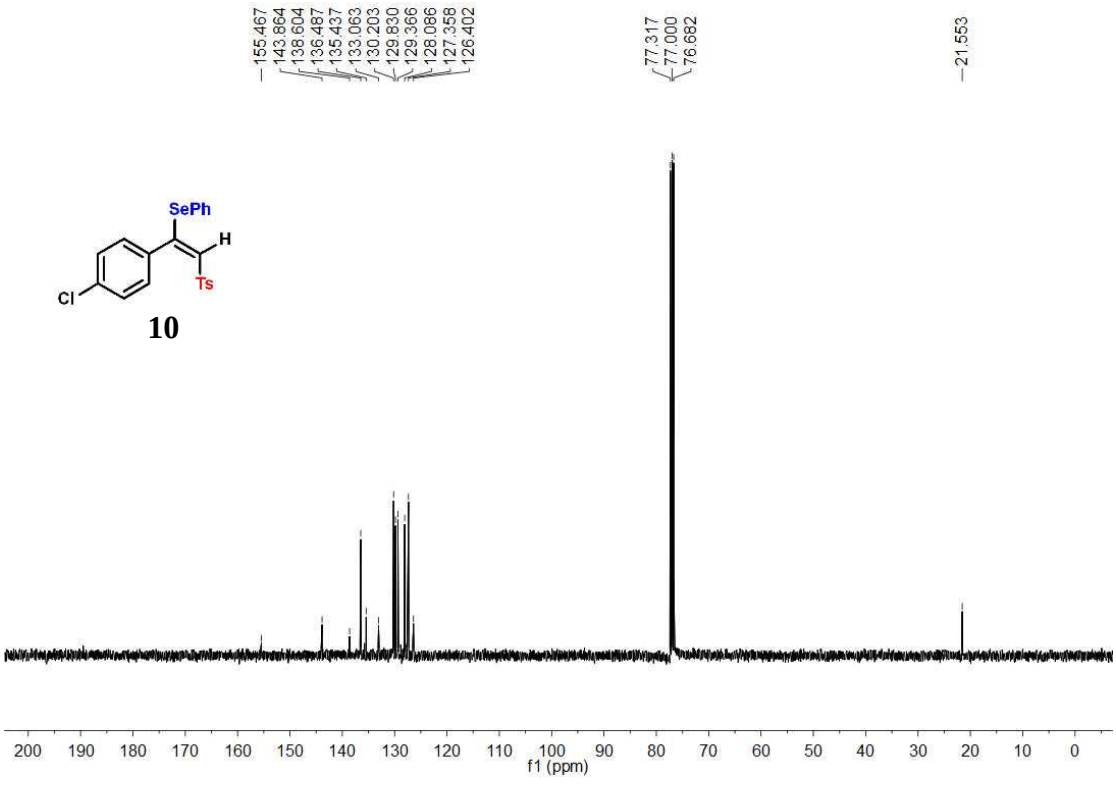
f1 (ppm)

10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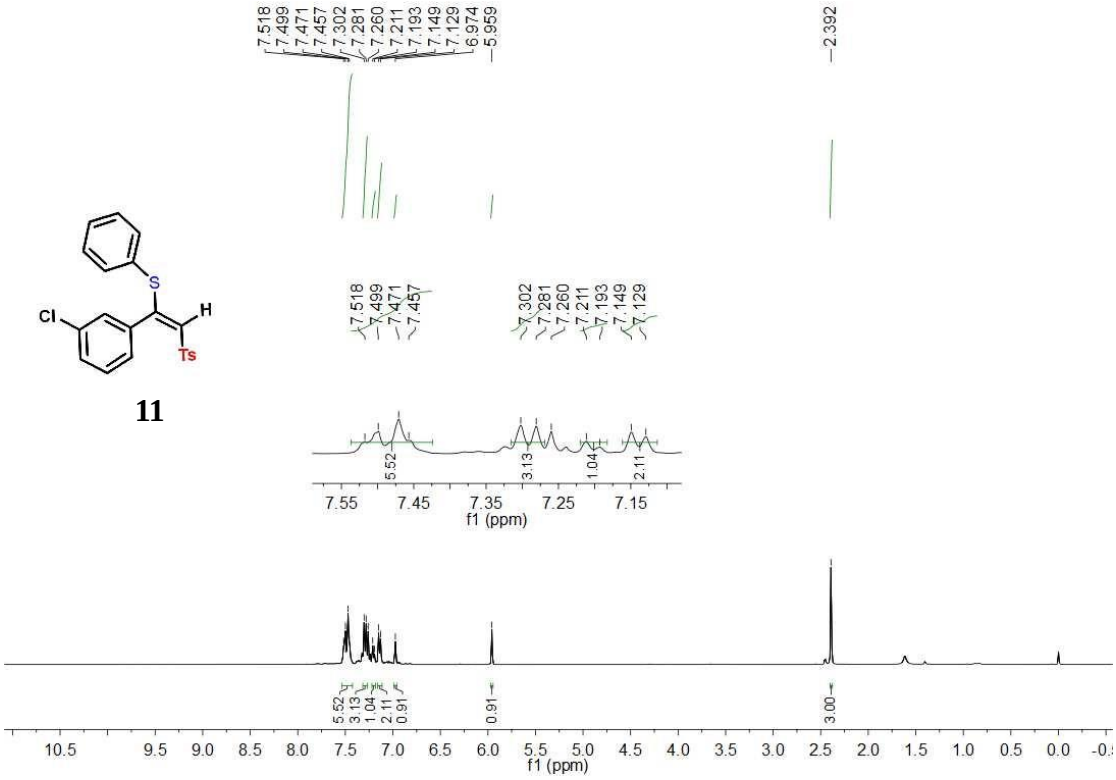
SePh
H
Ts
Cl
f1 (ppm)

10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2.392
S
Cl
H
Ts
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7.35
7.25
7.15
f1 (ppm)
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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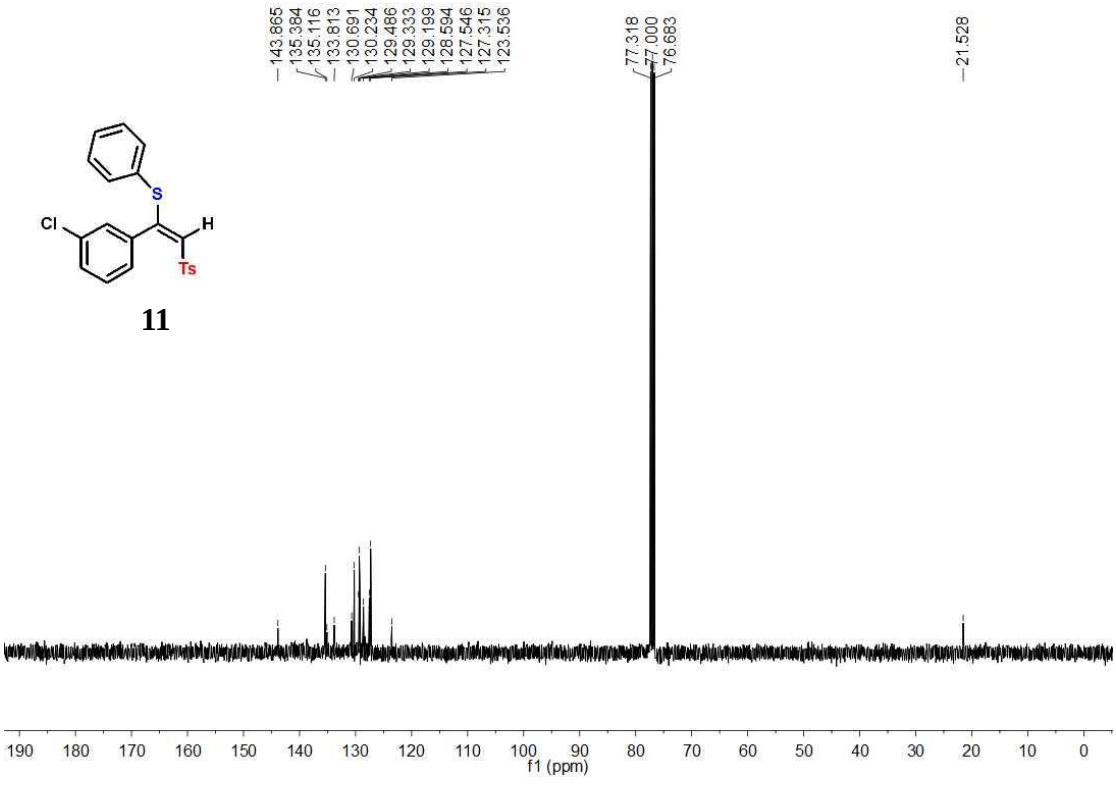
11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S
Cl
H
Ts
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180
170
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120
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40
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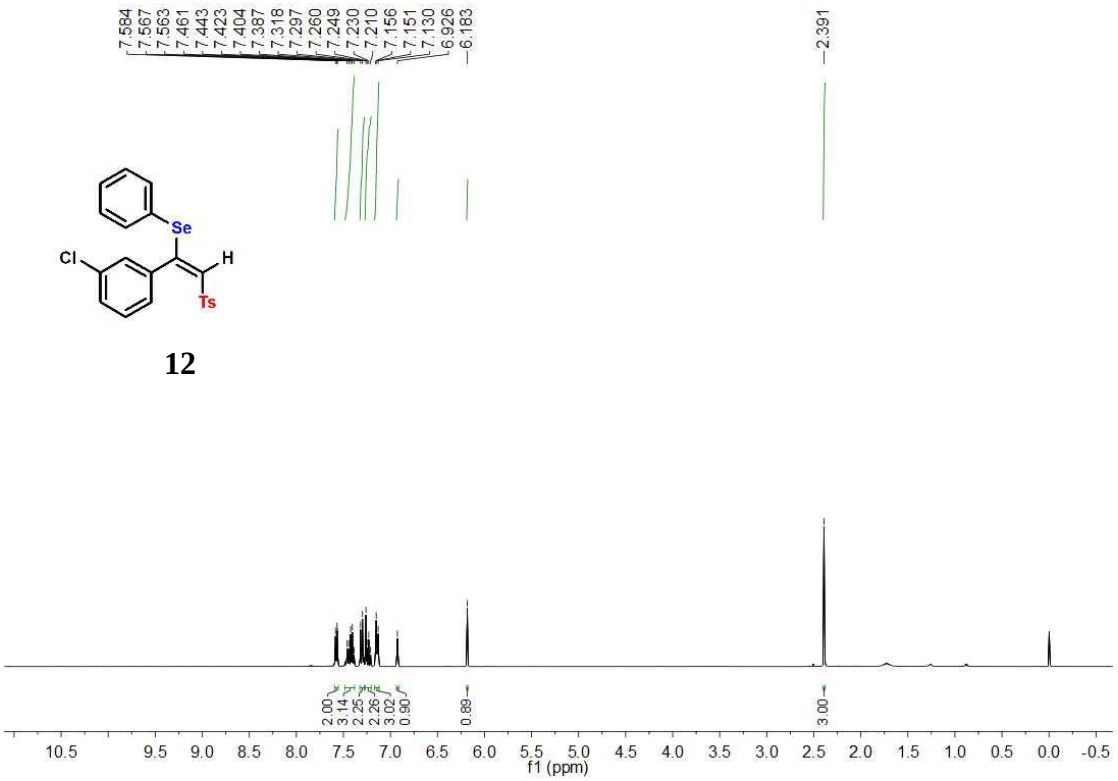
0
f1 (ppm)

11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Se
Cl
H
Ts
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9.0
8.5
8.0
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7.0
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6.0
5.5
5.0
4.5
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2.5
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1.5
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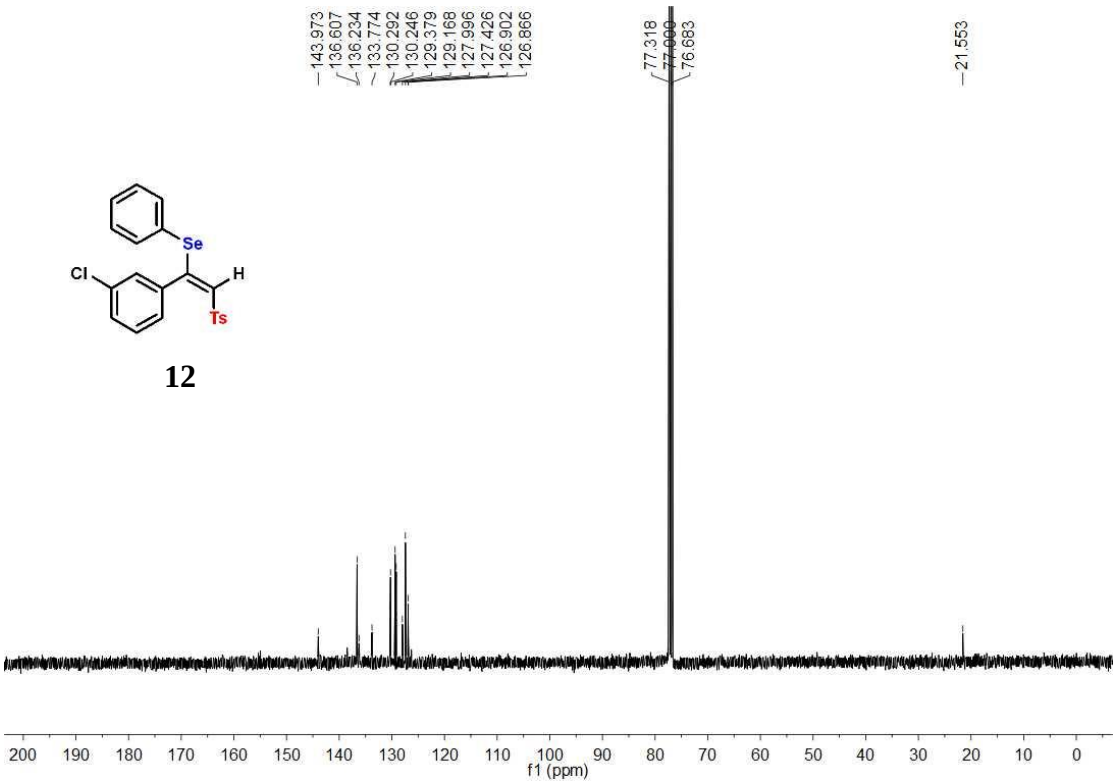
-0.5
f1 (ppm)

12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21.553
Se
Cl
H
Ts
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190
180
170
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80
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60
50
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30
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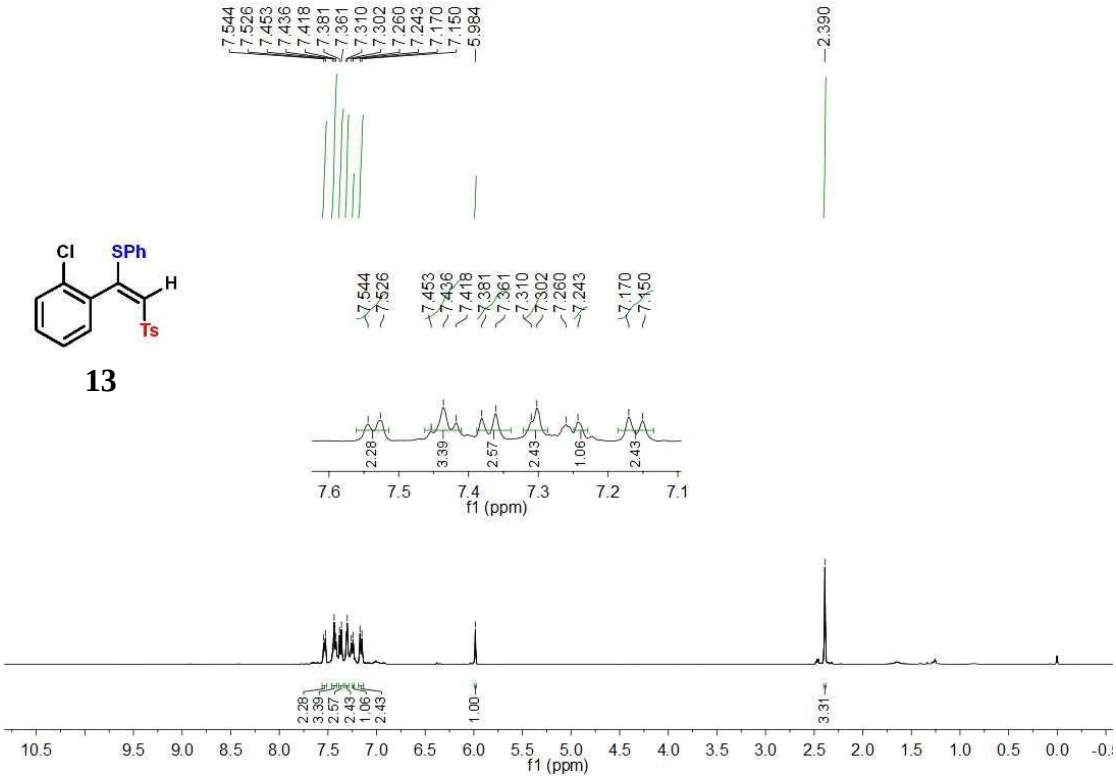
f1 (ppm)

12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5.984
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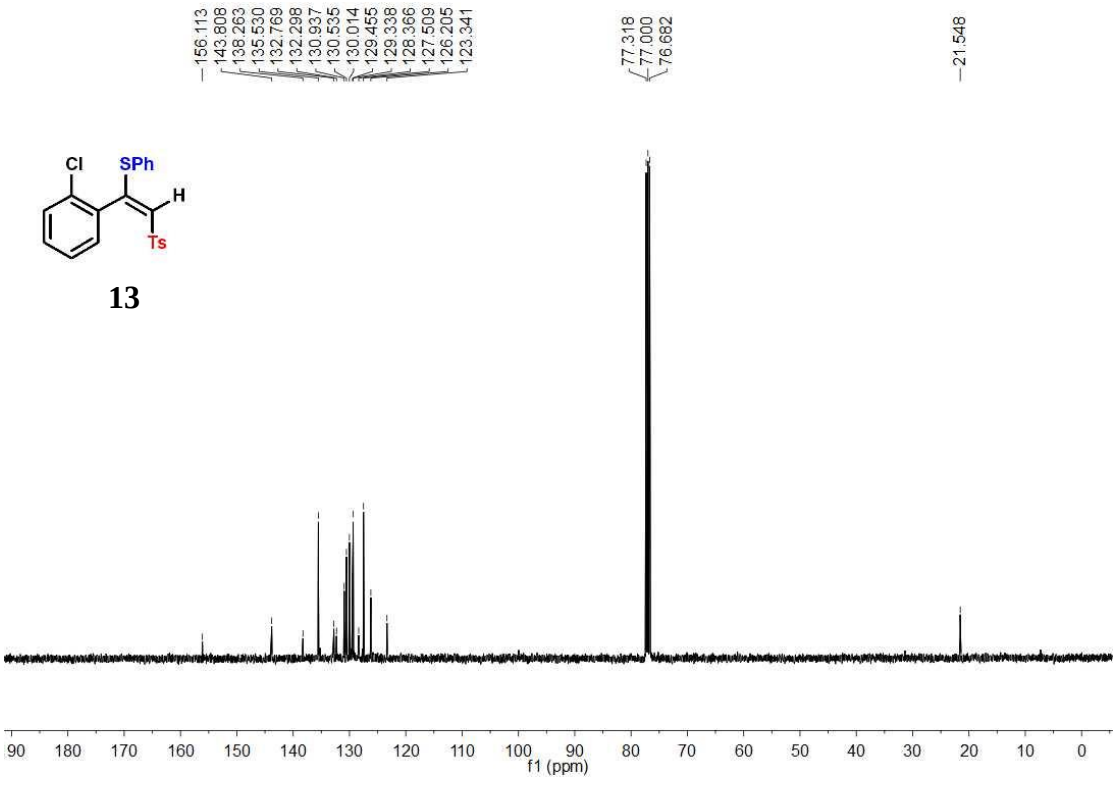
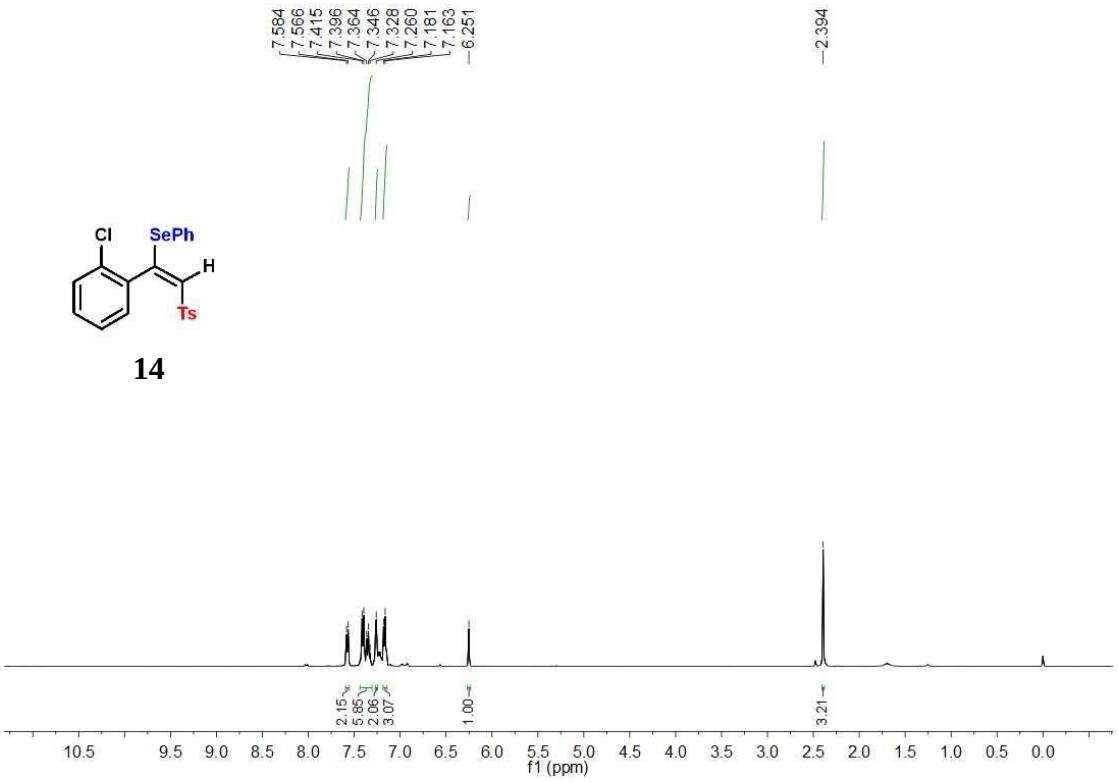
Cl
SPh
H
Ts
2.28
3.39
2.57
2.43
1.06
2.43
f1 (ppm)
1.00
3.31

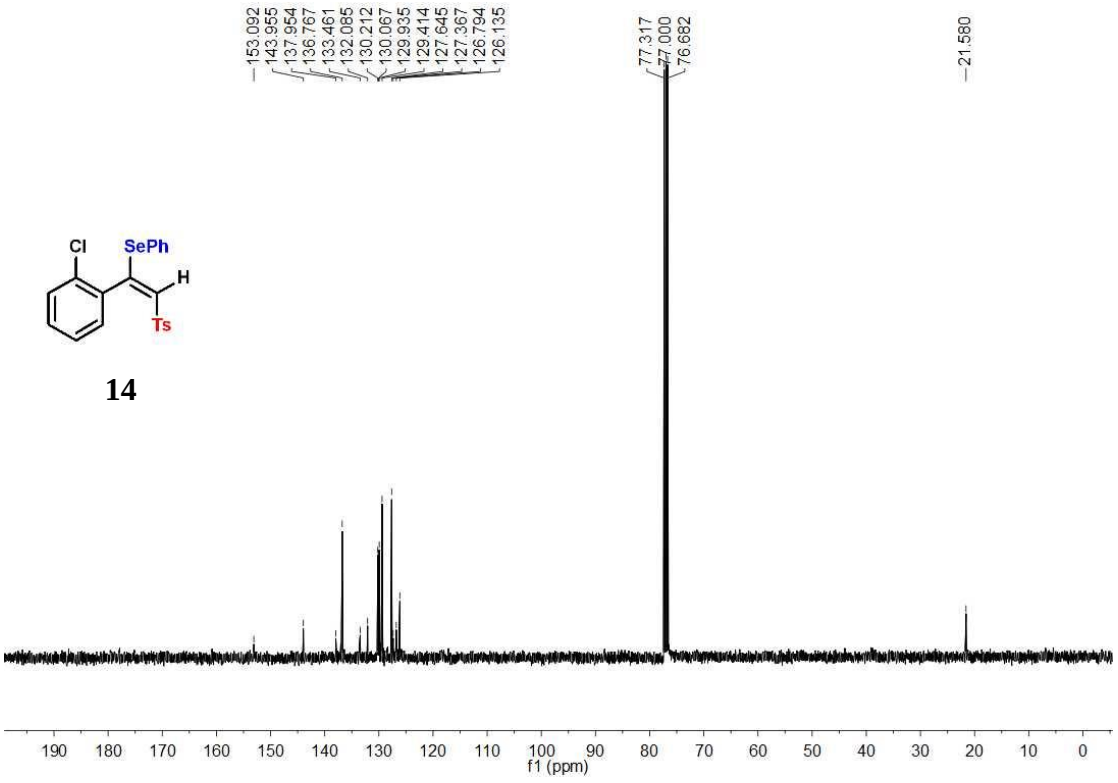
13


156.113
143.808
138.263
135.530
132.769
132.298
130.937
130.535
130.014
129.455
129.338
128.366
127.509
126.205
123.341
77.318
77.000
76.682
21.548
Cl
SPh
H
Ts
f1 (ppm)

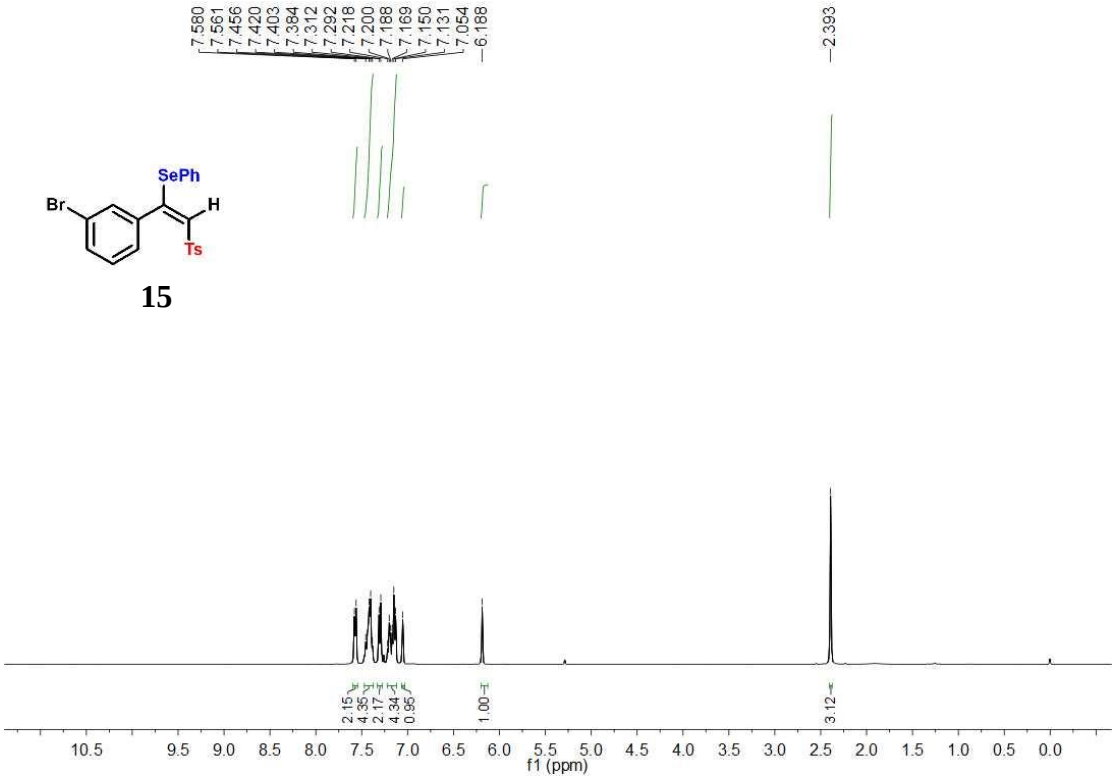
13


7.584
7.566
7.415
7.396
7.364
7.346
7.328
7.260
7.181
7.163
6.251
2.394
Cl
SePh
H
Ts
2.15
5.85
2.06
3.07
1.00
3.21
f1 (ppm)

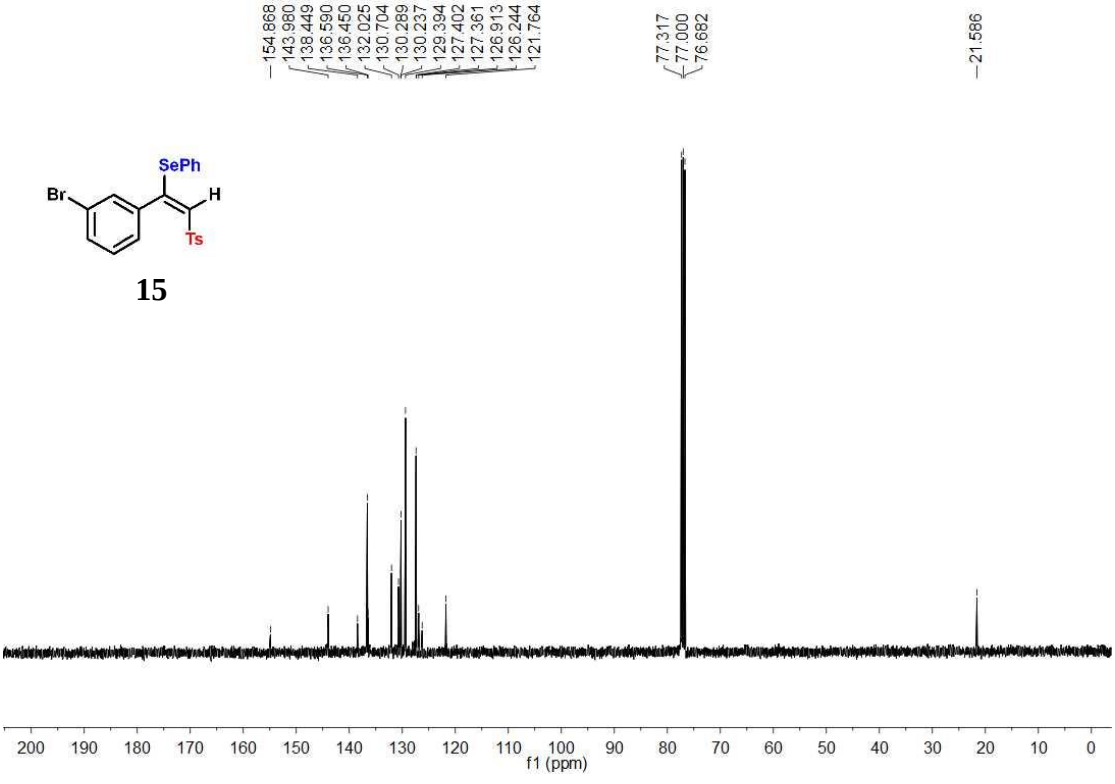
14


153.092
143.955
137.954
136.767
133.461
132.085
130.212
130.067
129.935
129.414
127.645
127.367
126.794
126.135
77.317
77.000
76.682
21.580
Cl
SePh
H
Ts
f1 (ppm)

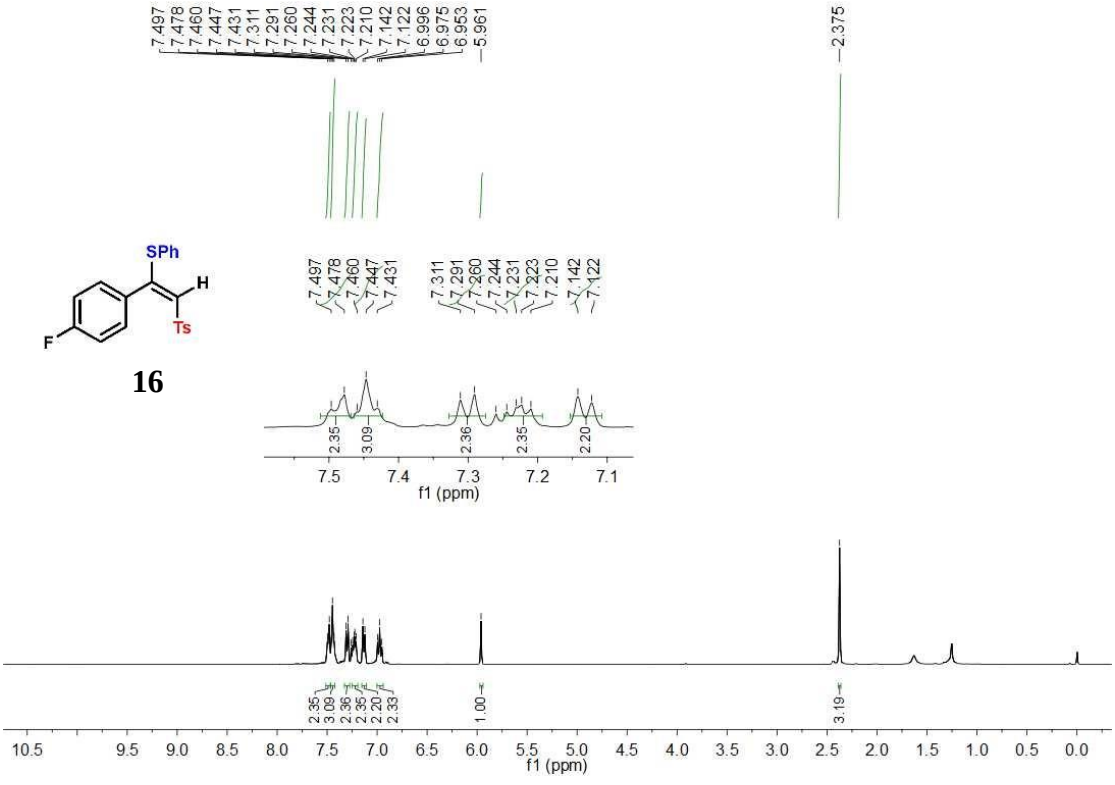
14

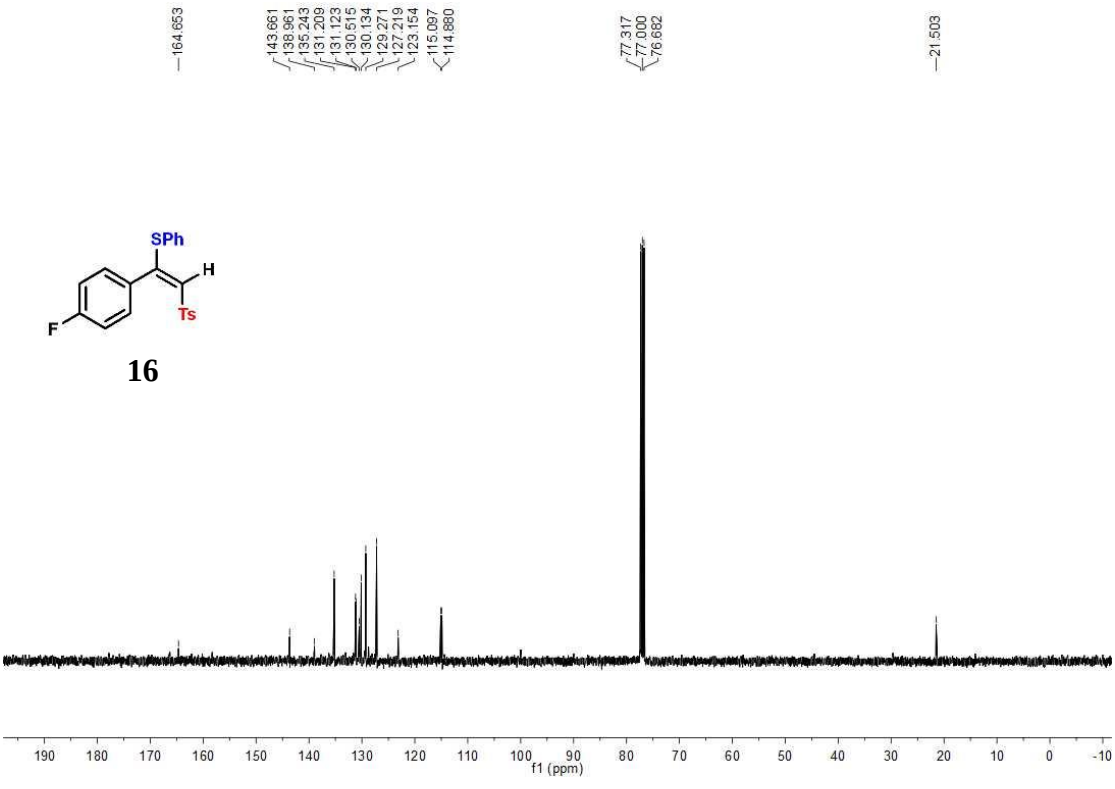

7.580
7.561
7.456
7.420
7.403
7.384
7.312
7.292
7.218
7.200
7.188
7.169
7.150
7.131
7.054
6.188
2.393
SePh
Br
H
Ts
2.15
4.35
2.17
4.34
0.95
1.00
3.12
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

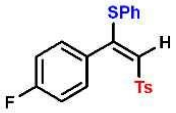
15


154.868
143.980
138.449
136.590
136.450
132.025
130.704
130.289
130.237
129.394
127.402
127.361
126.913
126.244
121.764
77.317
77.000
76.682
21.586
SePh
Br
H
Ts
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

15

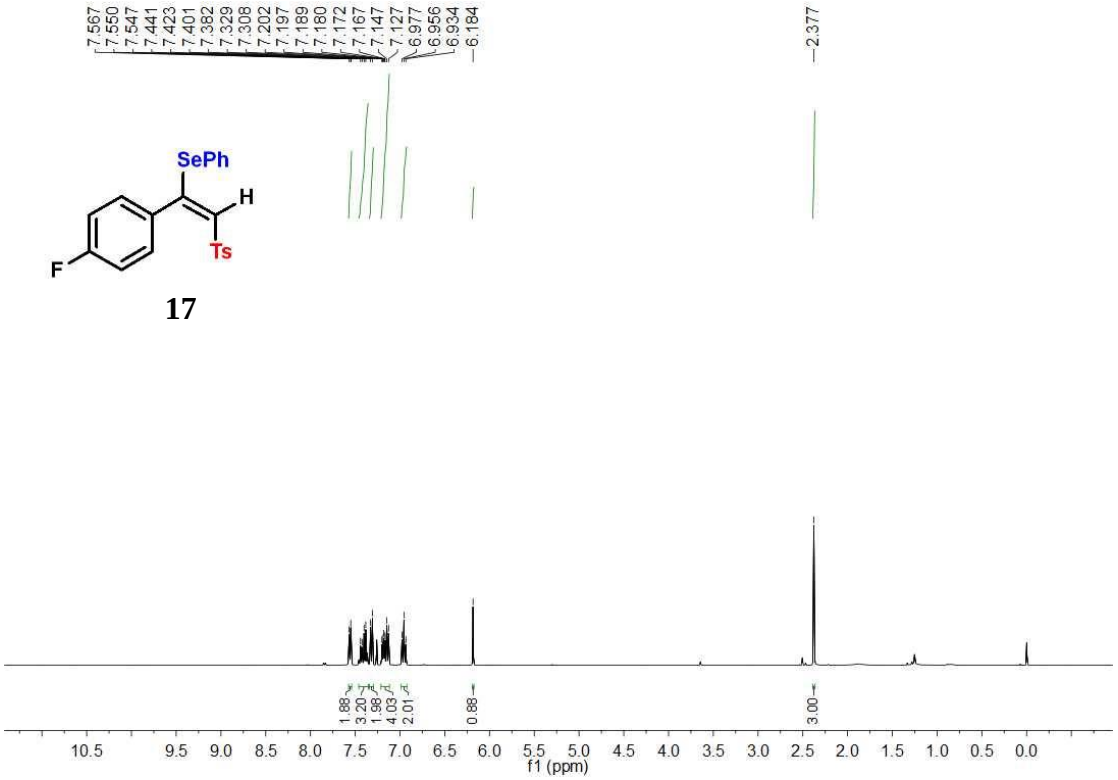

7.497
7.478
7.460
7.447
7.431
7.311
7.291
7.260
7.244
7.231
7.223
7.210
7.142
7.122
6.996
6.975
6.953
5.961
2.375
SPh
H
Ts
F
16
2.35
3.09
2.36
2.35
2.20
f1 (ppm)
1.00
3.19


164.653
143.661
138.961
135.243
131.209
131.123
130.515
130.134
129.271
127.219
123.154
115.097
114.880
77.317
77.000
76.682
21.503
SPh
H
Ts
F
16
f1 (ppm)

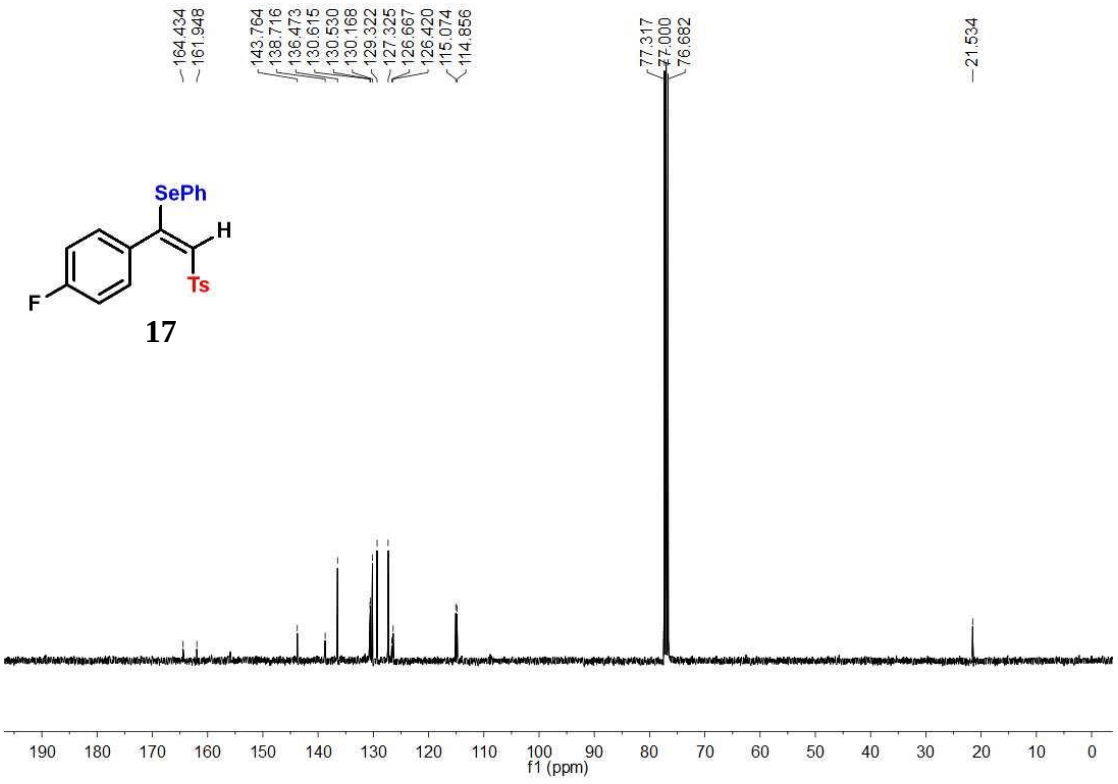

SPh
H
Ts
F

16

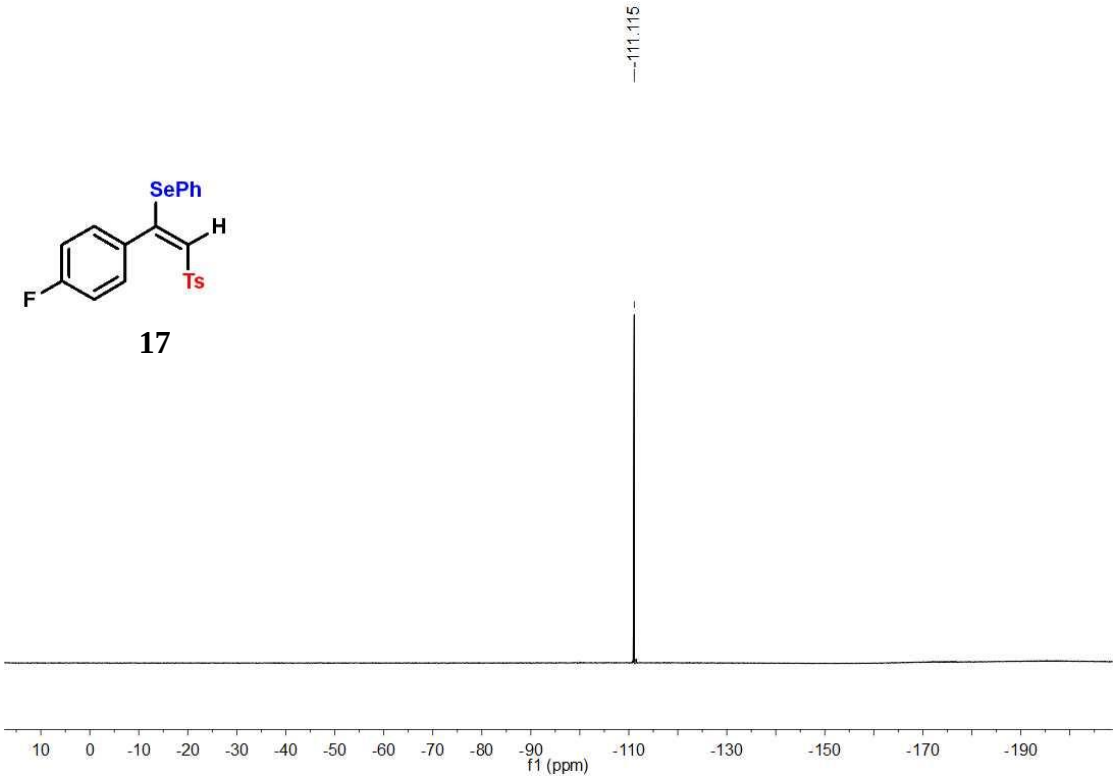
-110.730
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

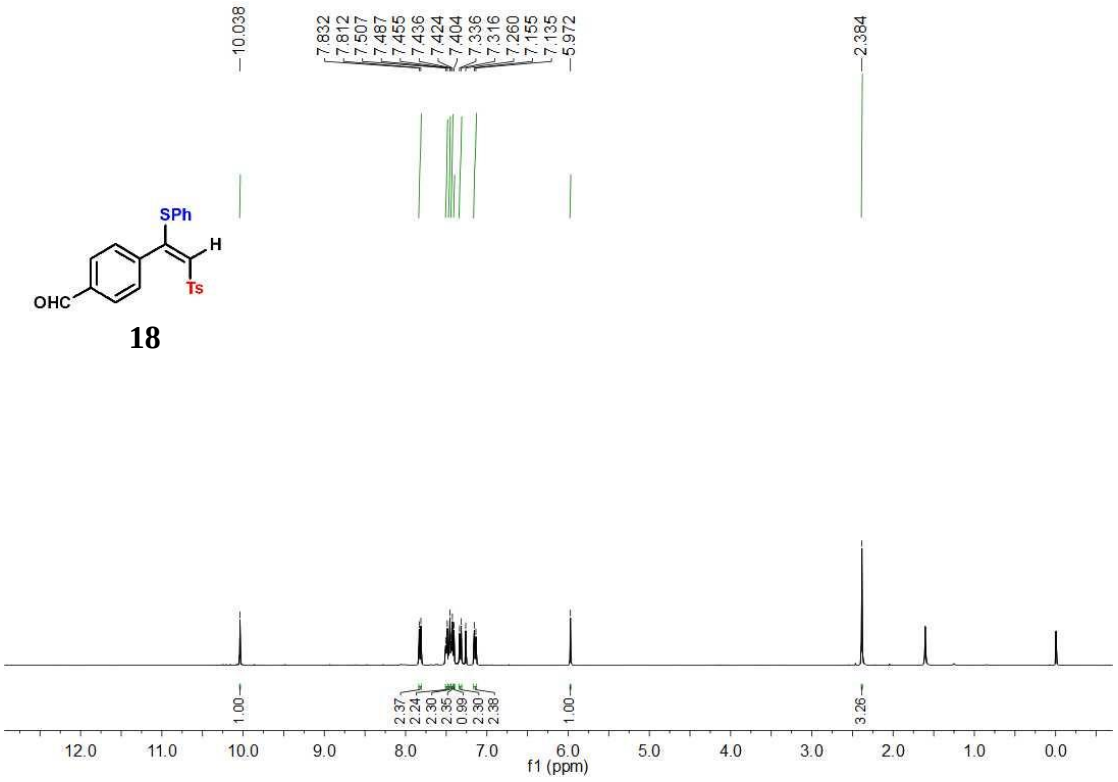

7.567
7.550
7.547
7.441
7.423
7.401
7.382
7.329
7.308
7.202
7.197
7.189
7.180
7.172
7.167
7.147
7.127
6.977
6.956
6.934
6.184
2.377
SePh
H
Ts
F
1.88
3.20
1.98
4.03
2.01
0.88
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

17

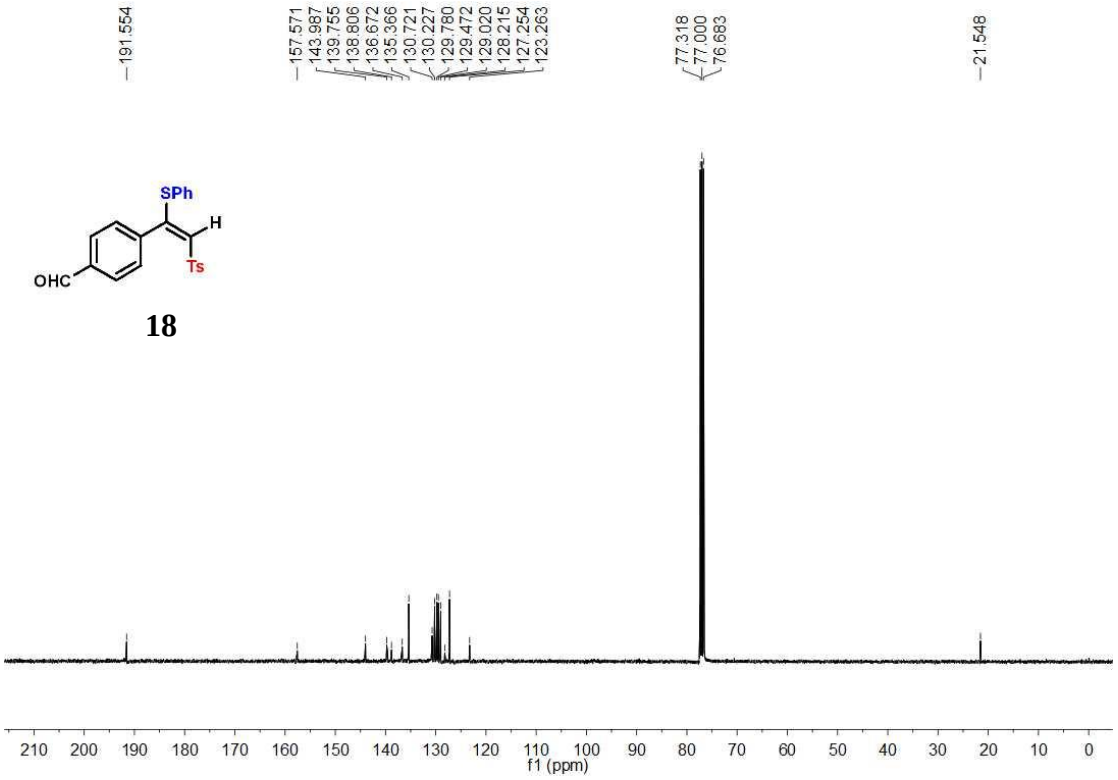

164.434
161.948
143.764
138.716
136.473
130.615
130.530
130.168
129.322
127.325
126.667
126.420
115.074
114.856
77.317
77.000
76.682
21.534
SePh
H
Ts
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

17

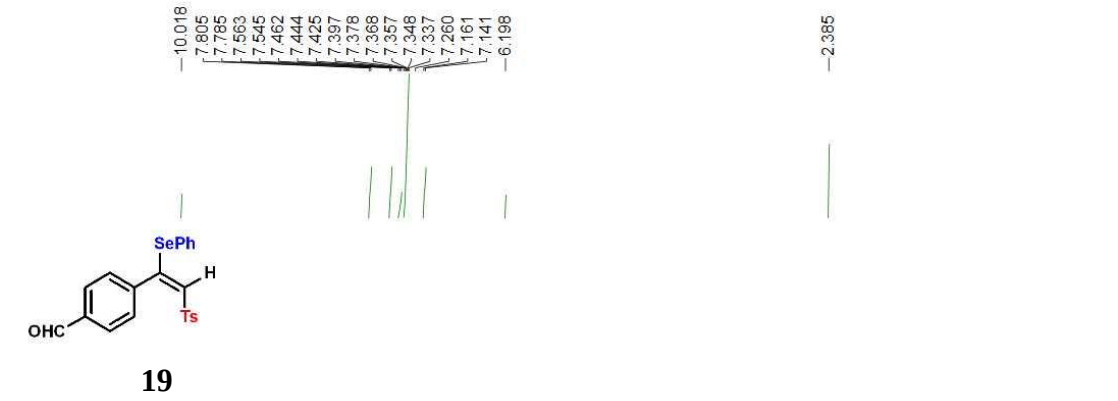

SePh
H
Ts
F
17
-111.115
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
f1 (ppm)


10.038
7.832
7.812
7.507
7.487
7.455
7.436
7.424
7.404
7.336
7.316
7.260
7.155
7.135
5.972
2.384
SPh
H
Ts
OHC
1.00
2.37
2.24
2.30
2.35
0.99
2.30
2.38
1.00
3.26
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

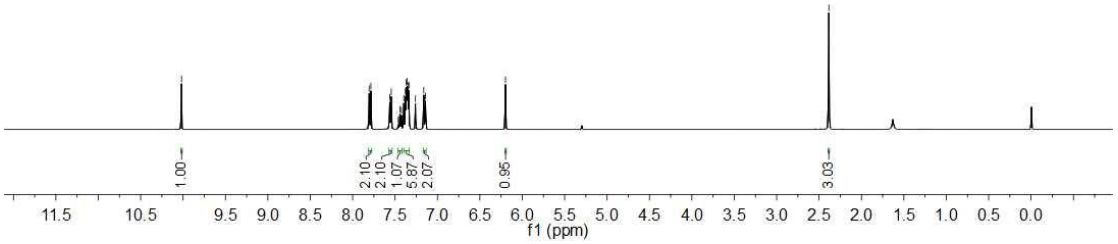
18

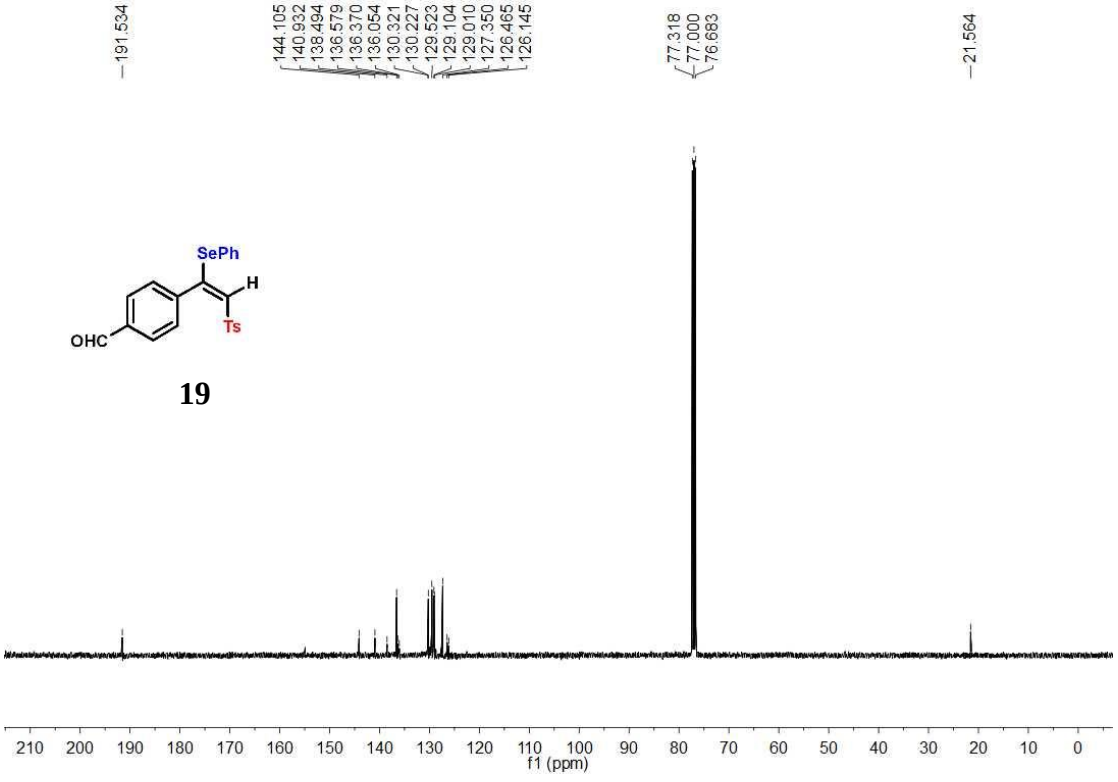

191.554
157.571
143.987
139.755
138.806
136.672
135.366
130.721
130.227
129.780
129.472
129.020
128.215
127.254
123.263
77.318
77.000
76.683
21.548
SPh
H
Ts
OHC
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

18

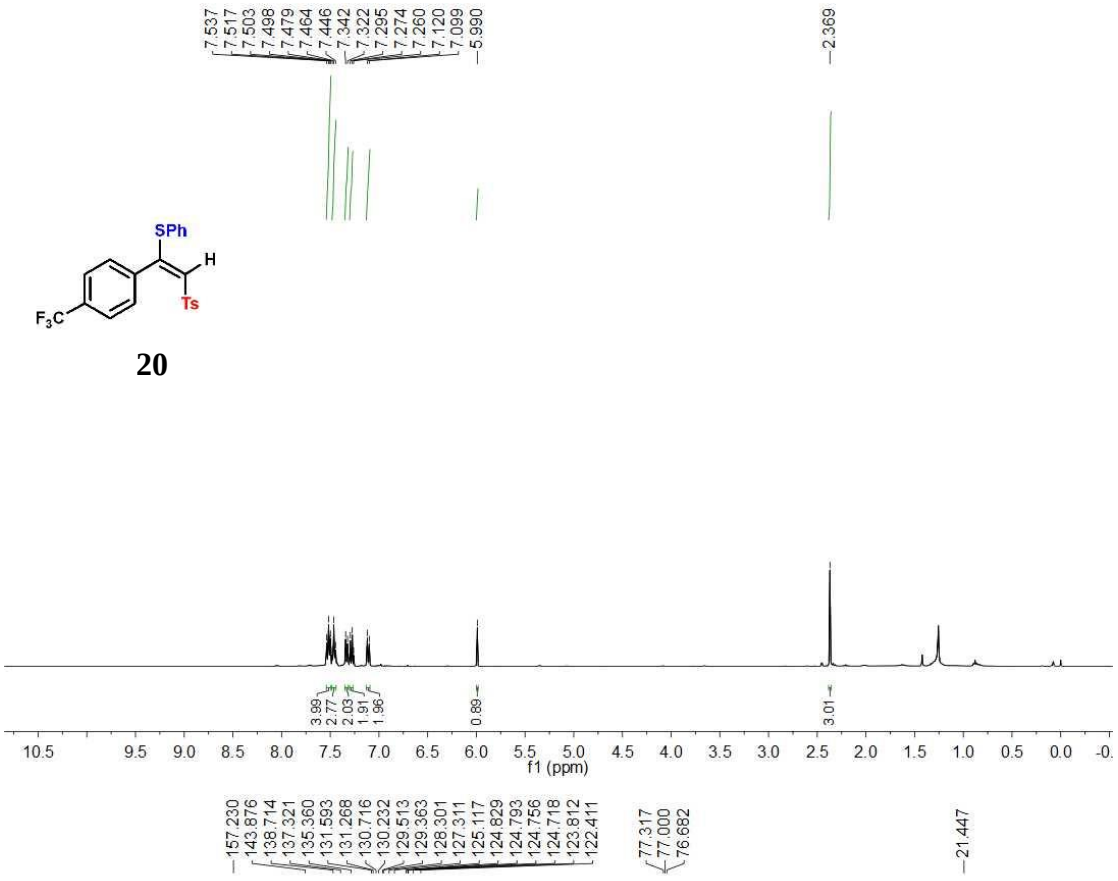

10.018
7.805
7.785
7.563
7.545
7.462
7.444
7.425
7.397
7.378
7.368
7.357
7.348
7.337
7.260
7.161
7.141
6.198
2.385
SePh
H
Ts
OHC

19

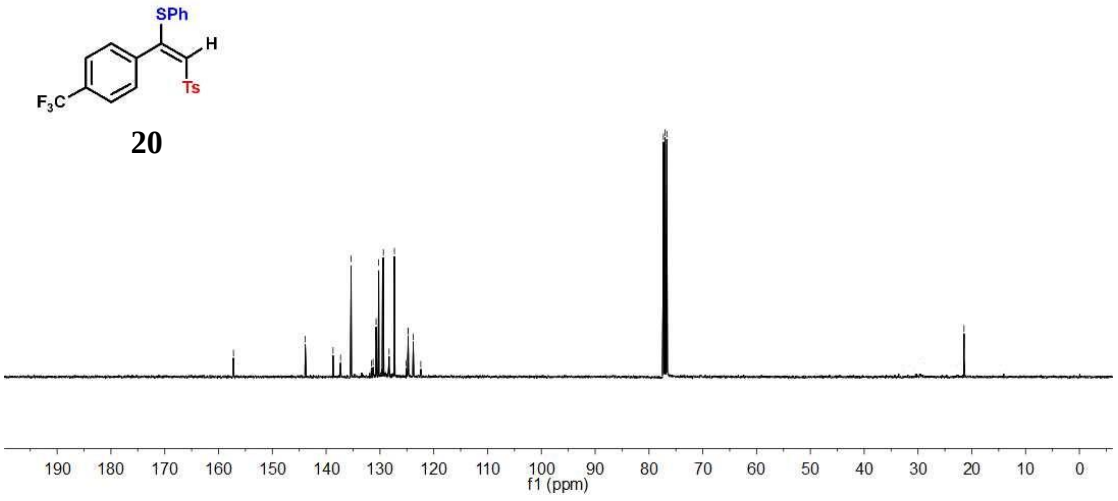

1.00
2.10
2.10
1.07
5.87
2.07
0.95
3.03
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


191.534
144.105
140.932
138.494
136.579
136.370
136.054
130.321
130.227
129.523
129.104
129.010
127.350
126.465
126.145
77.318
77.000
76.683
21.564
SePh
H
Ts
OHC
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

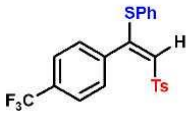
19


7.537
7.517
7.503
7.498
7.479
7.464
7.446
7.342
7.322
7.295
7.274
7.260
7.120
7.099
5.990
2.369
SPh
H
Ts
F3C
3.99
2.77
2.03
1.91
1.96
0.89
3.01
f1 (ppm)
157.230
143.876
138.714
137.321
135.360
131.593
131.268
130.716
130.232
129.513
129.363
128.301
127.311
125.117
124.829
124.793
124.756
124.718
123.812
122.411
77.317
77.000
76.682
21.447

20


SPh
H
Ts
F3C
f1 (ppm)

20


SPh
H
Ts
F3C

20

-62.860
10
0
-10
-20
-30
-40
-50
-60
-70
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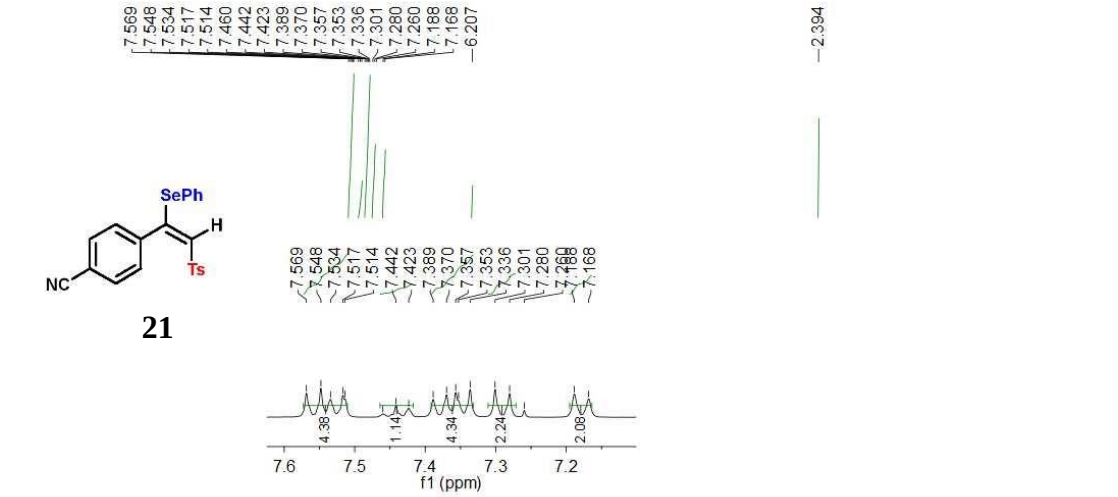
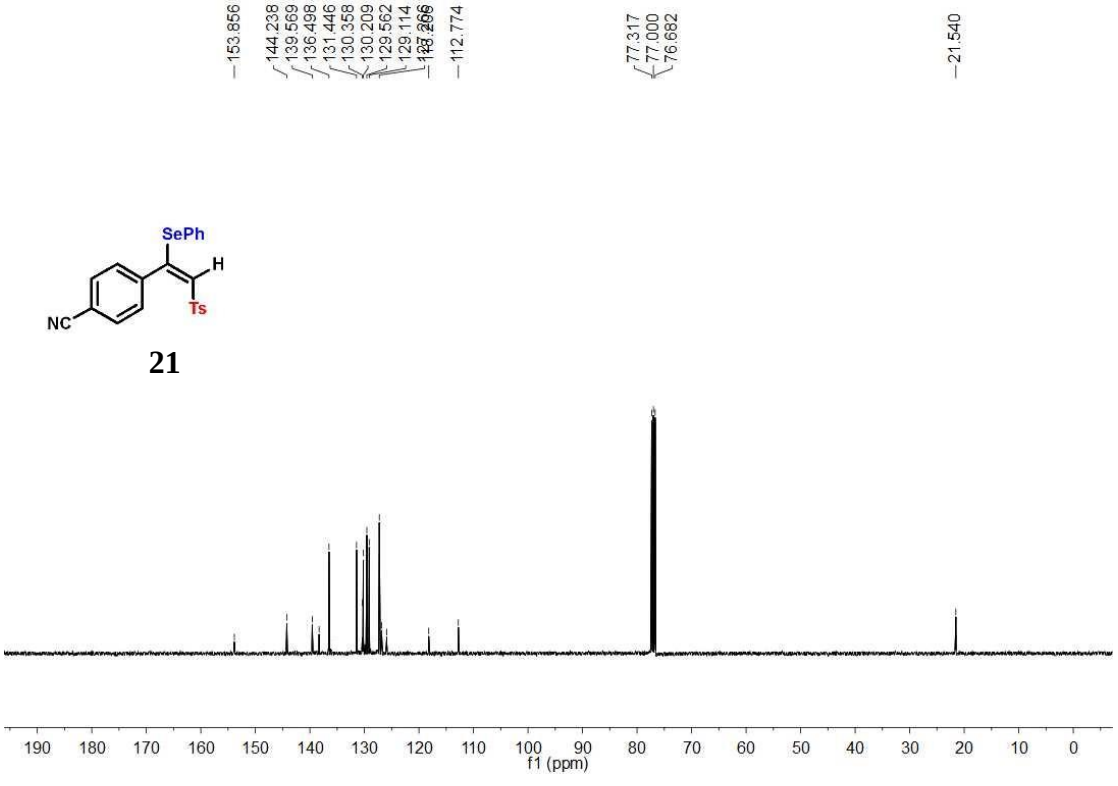
-90
-110
-130
-150
-170
-190
f1 (ppm)

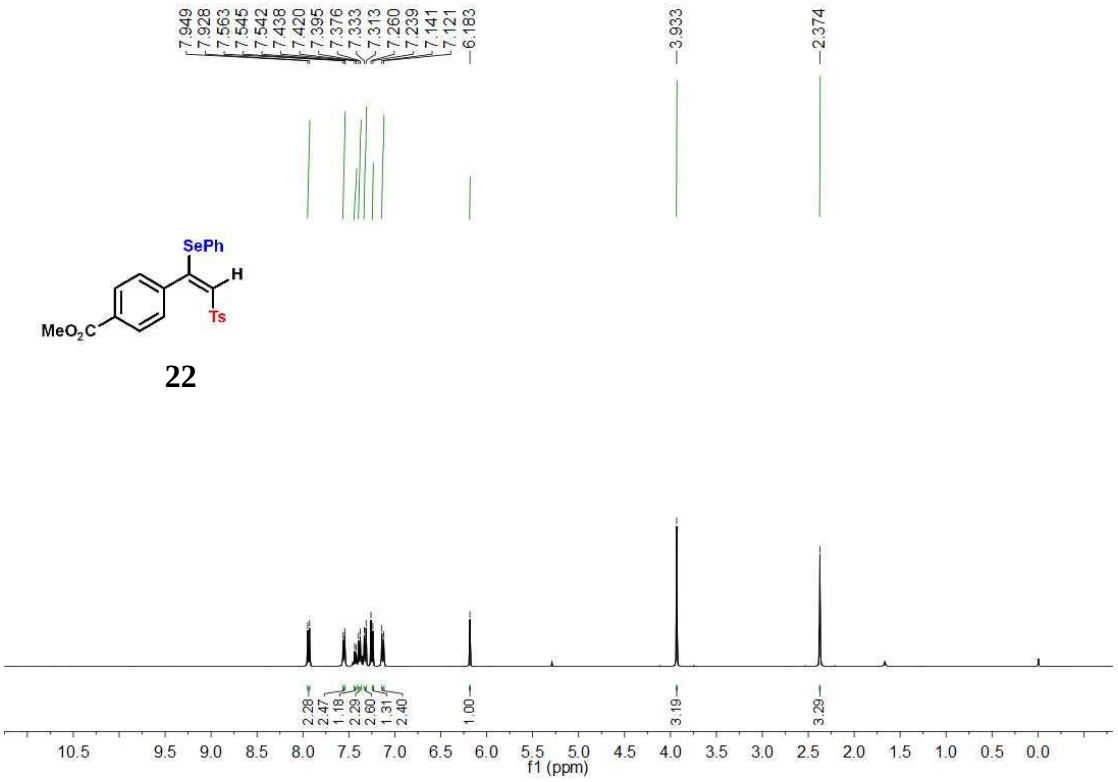


21

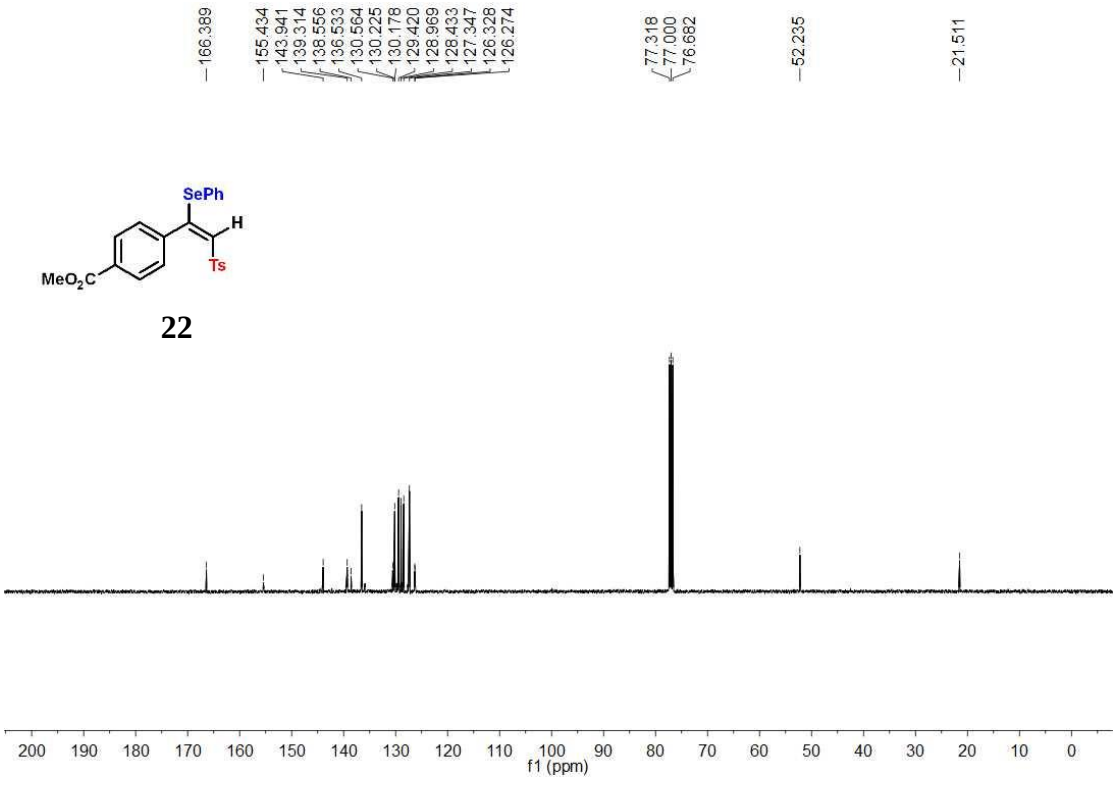


SePh
H
Ts
NC

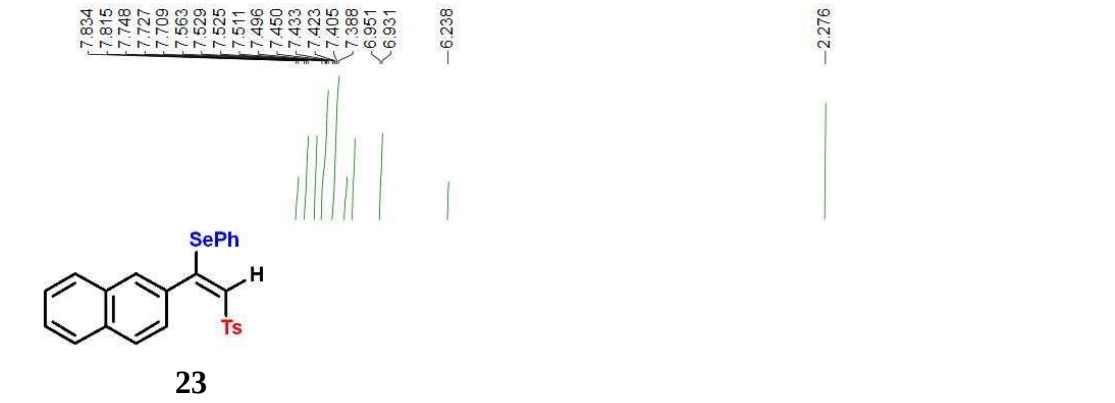
21


7.949
7.928
7.563
7.545
7.542
7.438
7.420
7.395
7.376
7.333
7.313
7.260
7.239
7.141
7.121
6.183
3.933
2.374
SePh
H
Ts
MeO2C
2.28
2.47
1.18
2.29
2.60
1.31
2.40
1.00
3.19
3.29
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

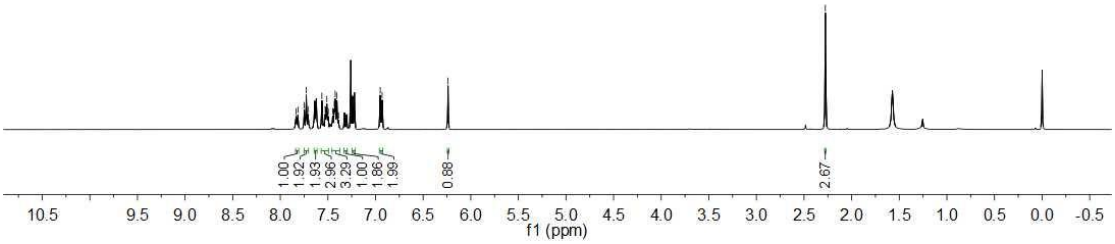
22

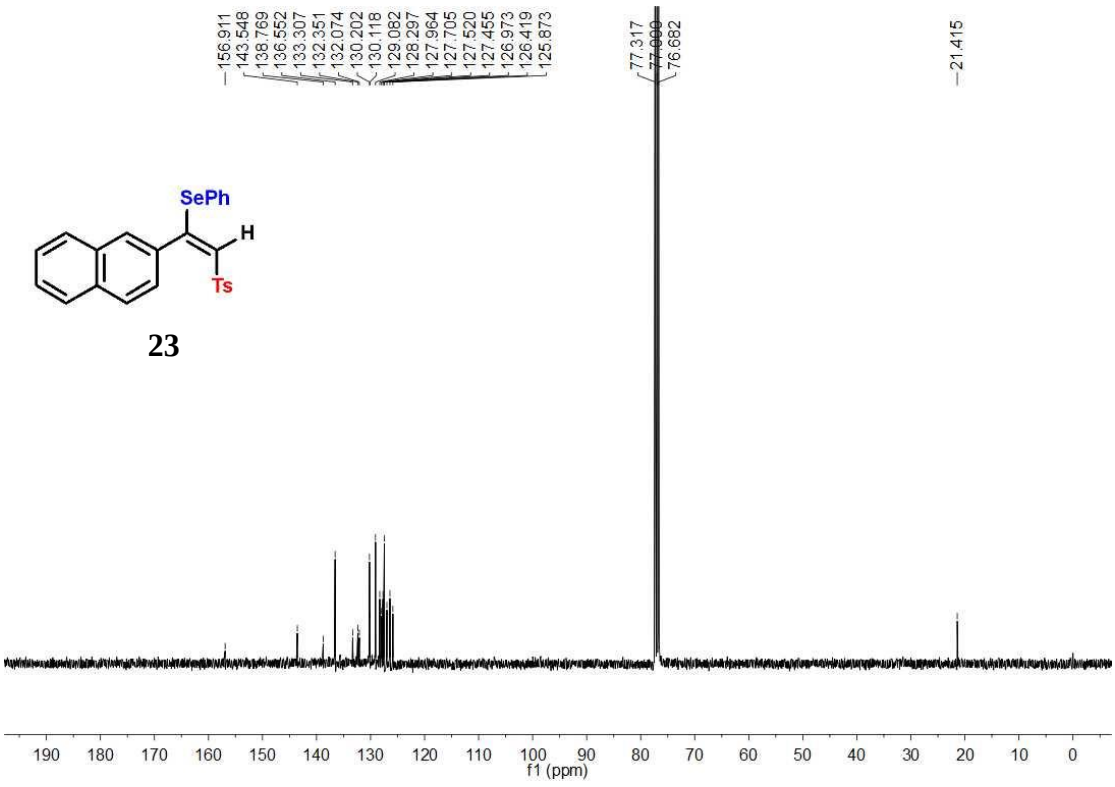

166.389
155.434
143.941
139.314
138.556
136.533
130.564
130.225
130.178
129.420
128.969
128.433
127.347
126.328
126.274
77.318
77.000
76.682
52.235
21.511
SePh
H
Ts
MeO2C
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

22

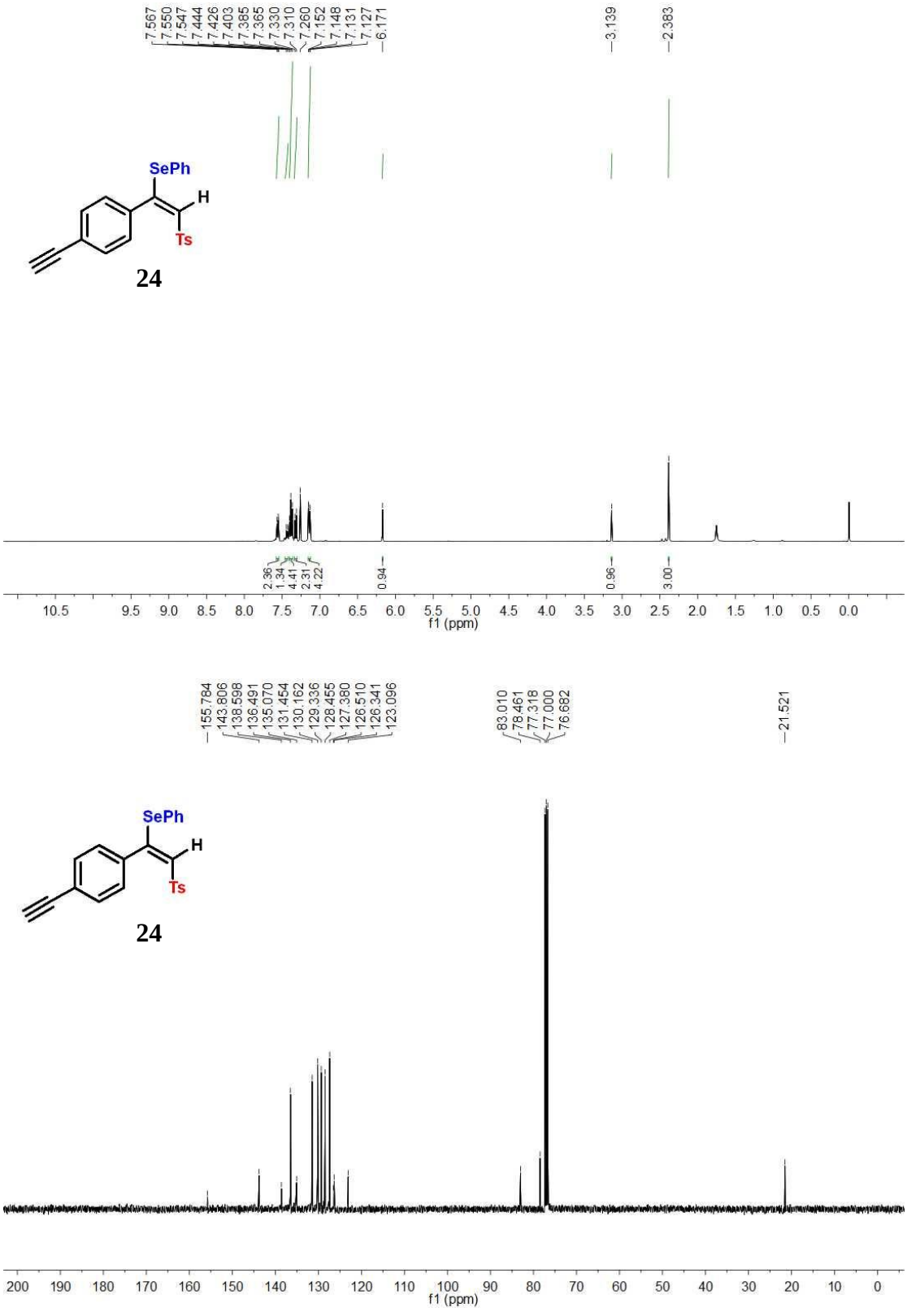

7.834
7.815
7.748
7.727
7.709
7.563
7.529
7.525
7.511
7.496
7.450
7.433
7.423
7.405
7.388
6.951
6.931
6.238
2.276
SePh
H
Ts

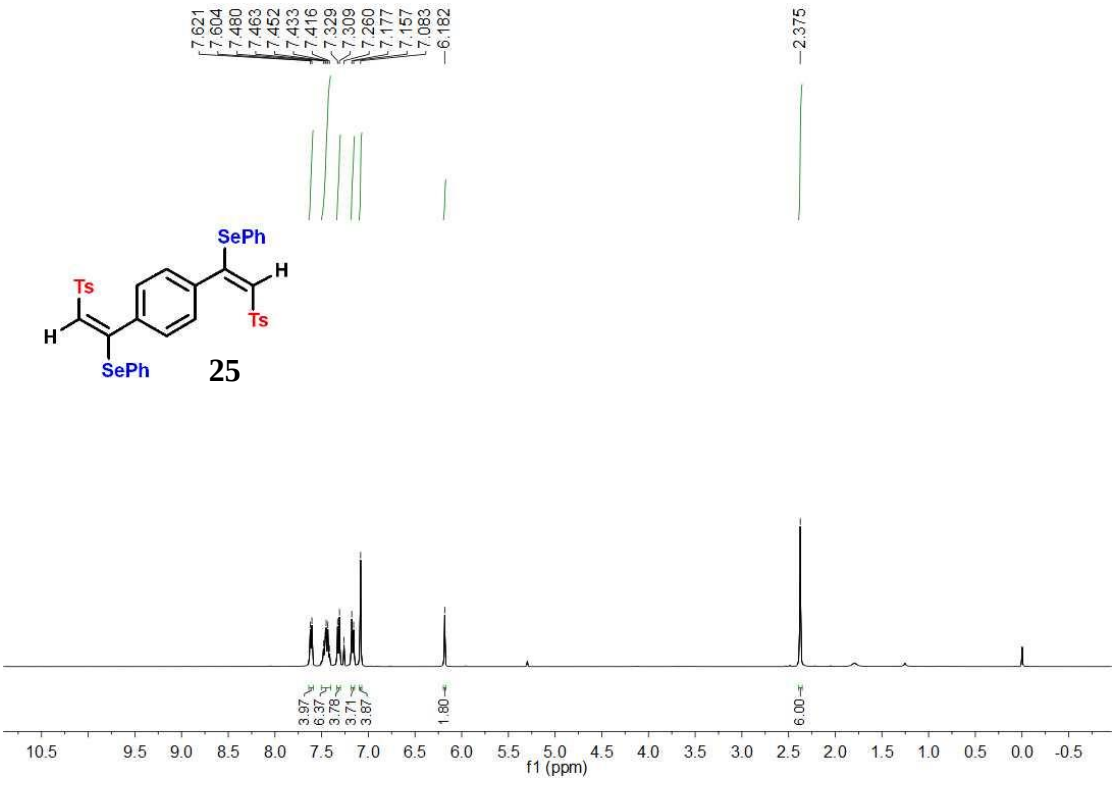
23


1.00
1.92
1.93
2.96
3.29
1.00
1.86
1.99
0.88
2.67
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


156.911
143.548
138.769
136.552
133.307
132.351
132.074
130.202
130.118
129.082
128.297
127.964
127.705
127.520
127.455
126.973
126.419
125.873
77.317
77.000
76.682
21.415
SePh
H
Ts
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

23


7.567
7.550
7.547
7.444
7.426
7.403
7.385
7.365
7.330
7.310
7.260
7.152
7.148
7.131
7.127
6.171
3.139
2.383
SePh
H
Ts
24
2.36
1.34
4.41
2.31
4.22
0.94
0.96
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
155.784
143.806
138.598
136.491
135.070
131.454
130.162
129.336
128.455
127.380
126.510
126.341
123.096
83.010
78.461
77.318
77.000
76.682
21.521
SePh
H
Ts
24
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.621
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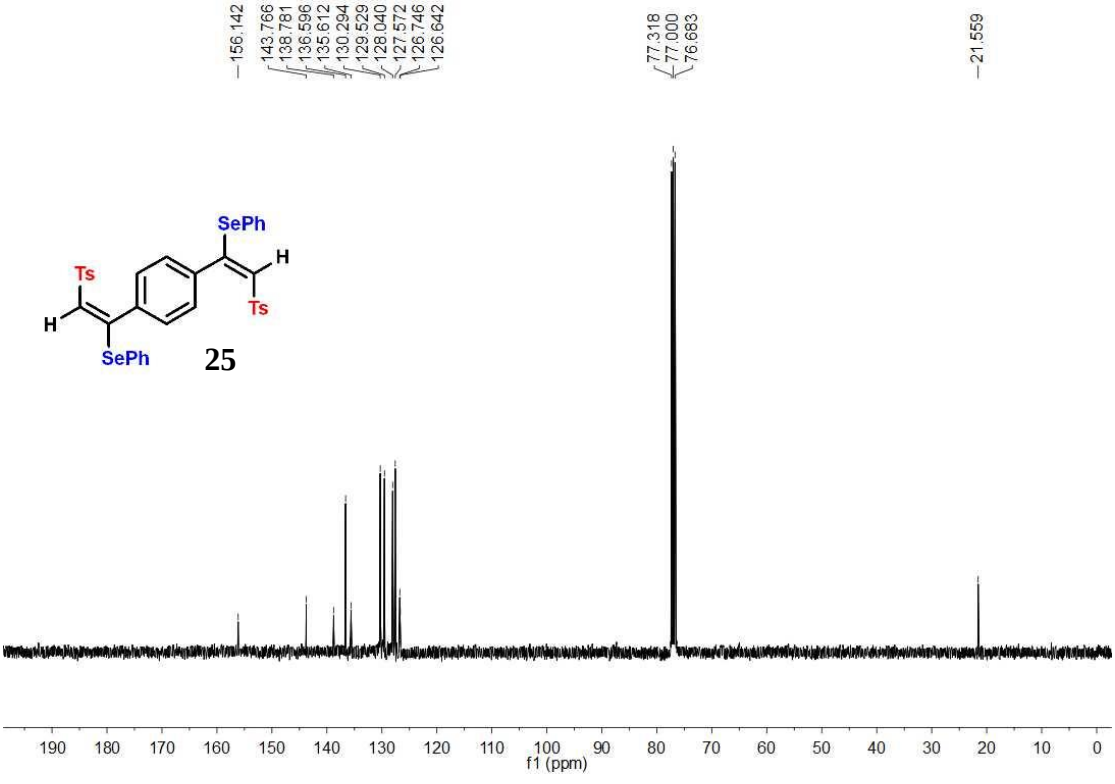
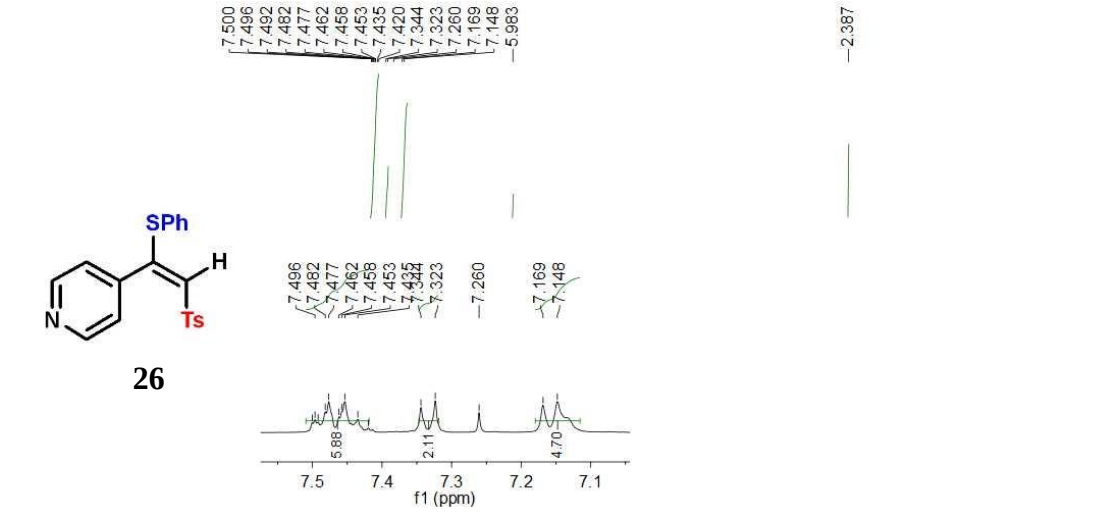
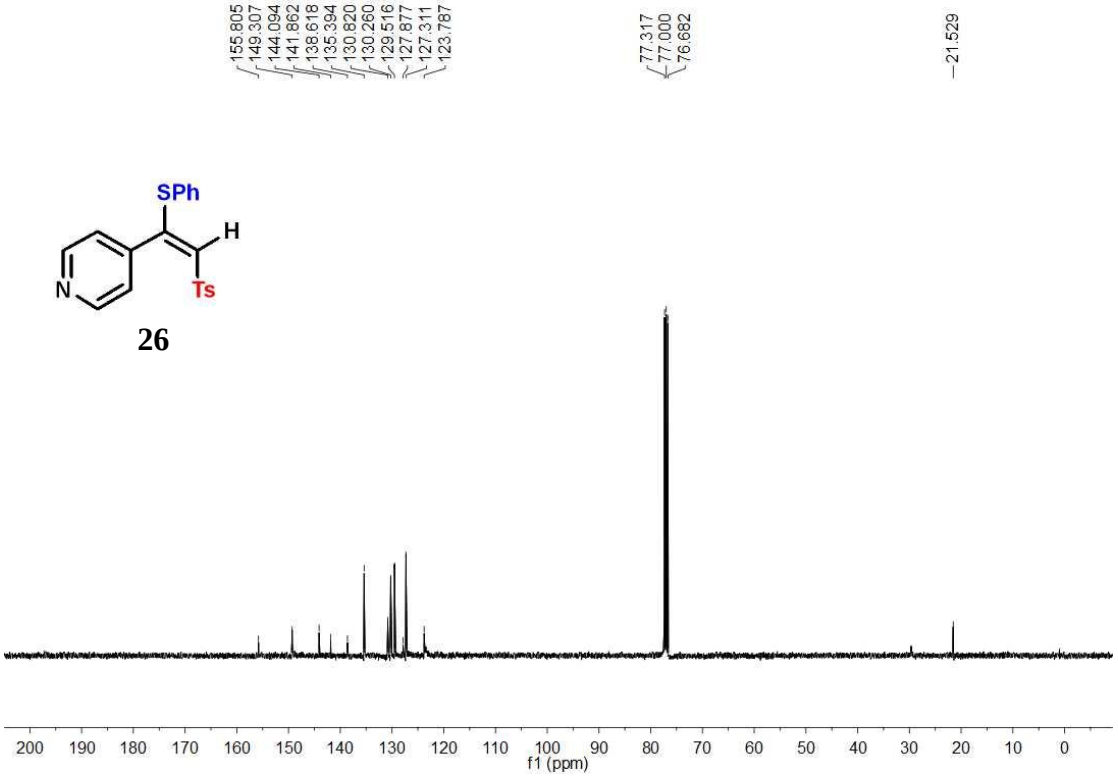
7.463
7.452
7.433
7.416
7.329
7.309
7.260
7.177
7.157
7.083
6.182
2.375
SePh
H
Ts
Ts
H
SePh
25
3.97
6.37
3.78
3.71
3.87
1.80
6.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

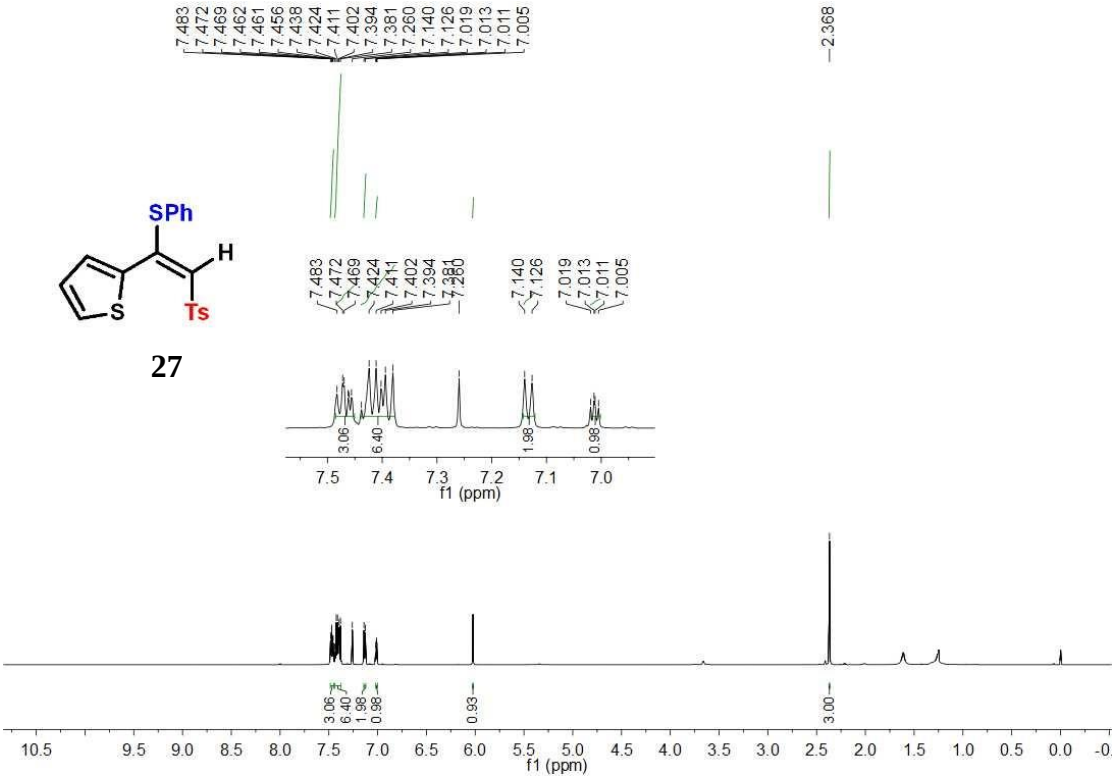

156.142
143.766
138.781
136.596
135.612
130.294
129.529
128.040
127.572
126.746
126.642
77.318
77.000
76.683
21.559
SePh
H
Ts
Ts
H
SePh
25
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.500
7.496
7.492
7.482
7.477
7.462
7.458
7.453
7.435
7.420
7.344
7.323
7.260
7.169
7.148
5.983
2.387
SPh
H
N
Ts
5.88
2.11
4.70
7.5
7.4
7.3
7.2
7.1
f1 (ppm)
1.00
3.02
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

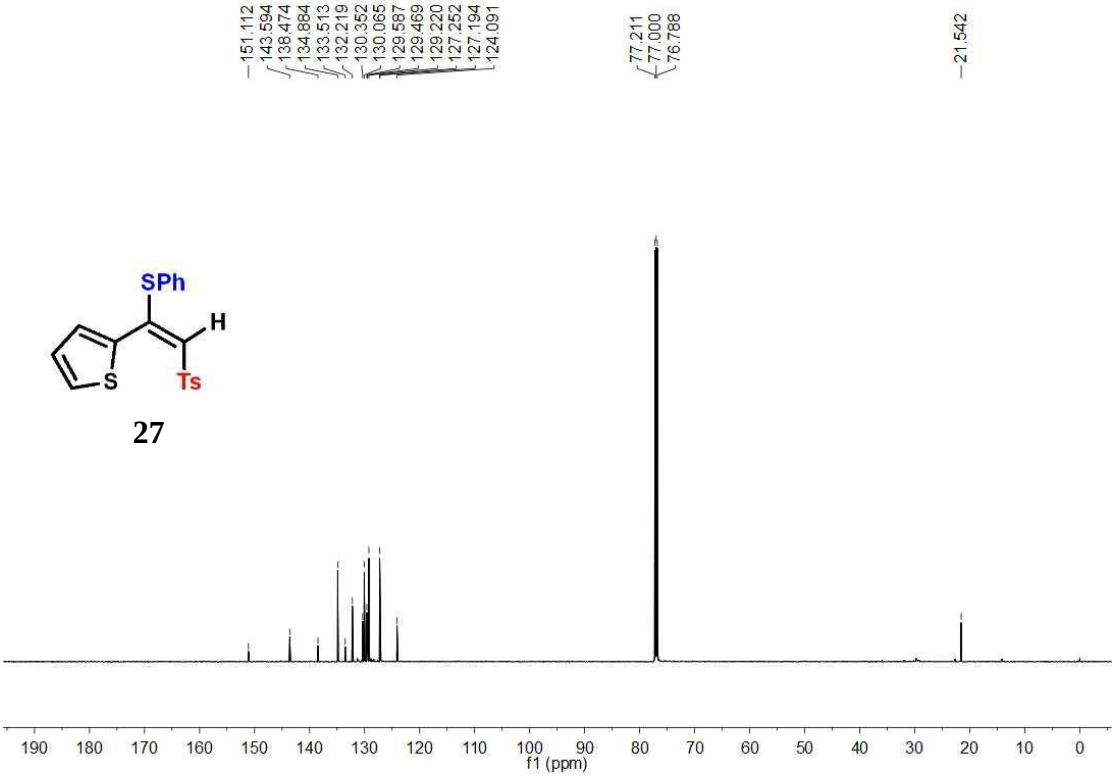
26


155.805
149.307
144.094
141.862
138.618
135.394
130.820
130.260
129.516
127.877
127.311
123.787
77.317
77.000
76.682
21.529
SPh
H
N
Ts
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

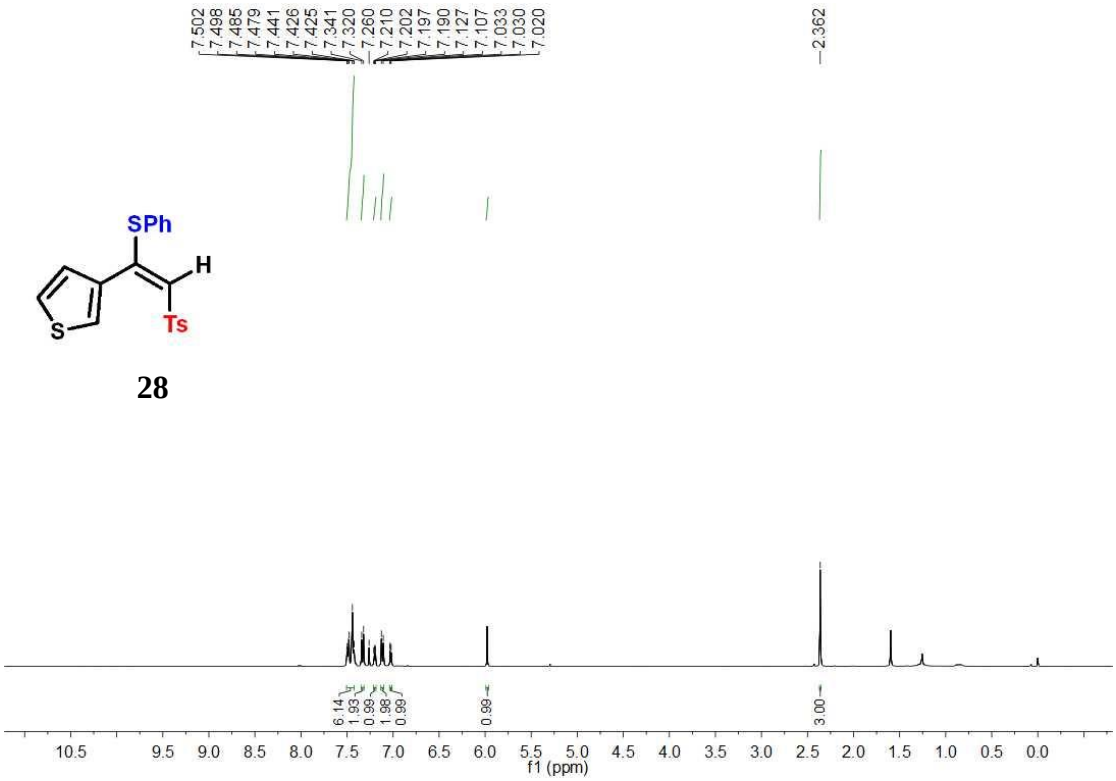
26


7.483
7.472
7.469
7.462
7.461
7.456
7.438
7.424
7.411
7.402
7.394
7.381
7.260
7.140
7.126
7.019
7.013
7.011
7.005
2.368
SPh
H
S
Ts
3.06
6.40
1.98
0.98
0.93
3.00
f1 (ppm)

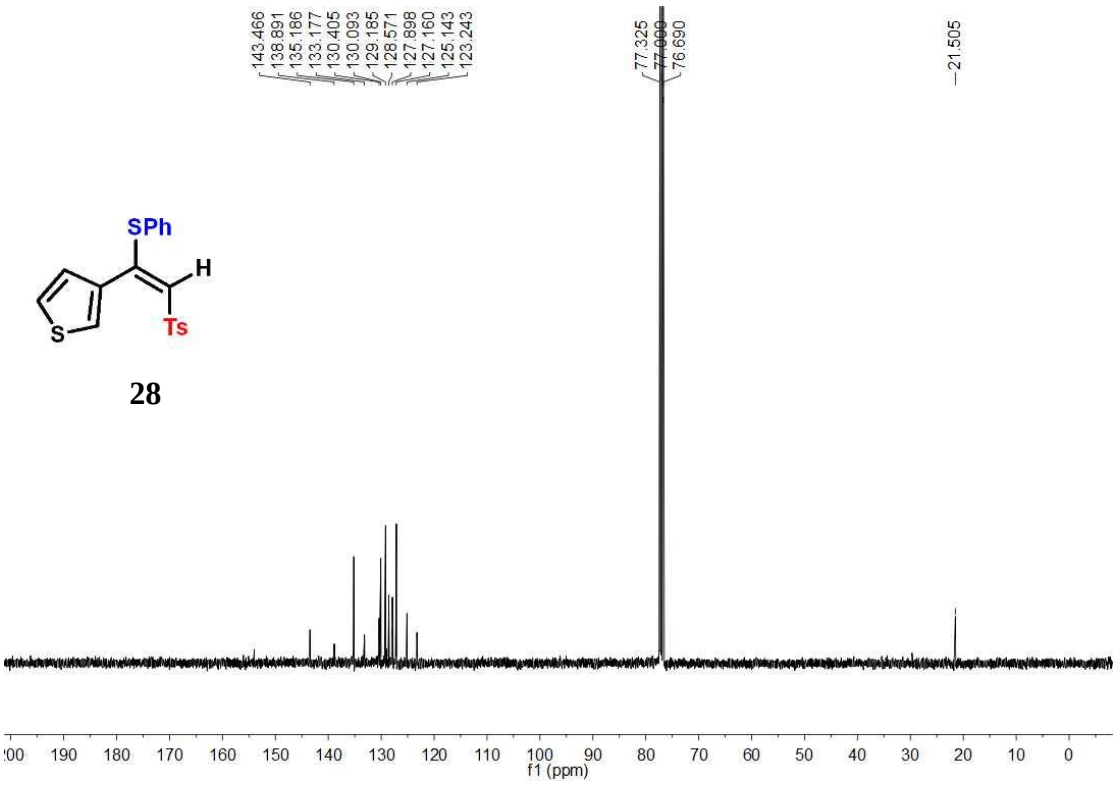
27


151.112
143.594
138.474
134.884
133.513
132.219
130.352
130.065
129.587
129.469
129.220
127.252
127.194
124.091
77.211
77.000
76.788
21.542
SPh
H
S
Ts
f1 (ppm)

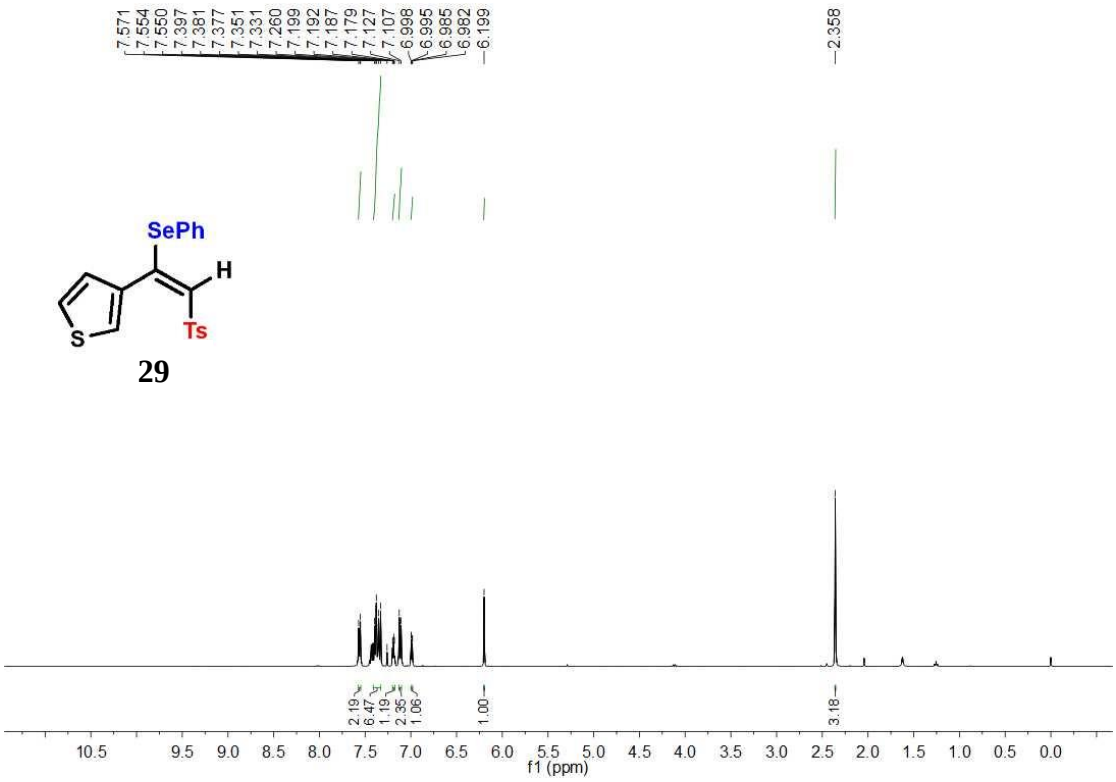
27


7.502
7.498
7.485
7.479
7.441
7.426
7.425
7.341
7.320
7.260
7.210
7.202
7.197
7.190
7.127
7.107
7.033
7.030
7.020
2.362
SPh
H
Ts
S
6.14
1.93
0.99
1.98
0.99
0.99
3.00
f1 (ppm)

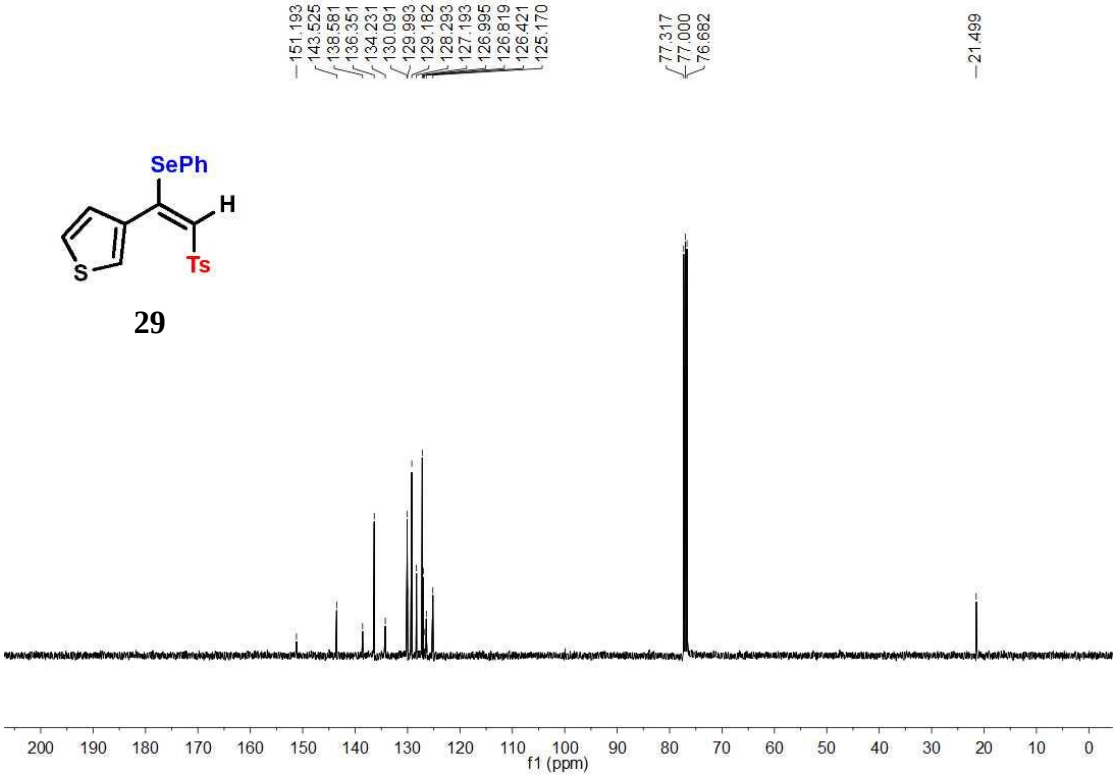
28


143.466
138.891
135.186
133.177
130.405
130.093
129.185
128.571
127.898
127.160
125.143
123.243
77.325
77.000
76.690
21.505
SPh
H
Ts
S
f1 (ppm)

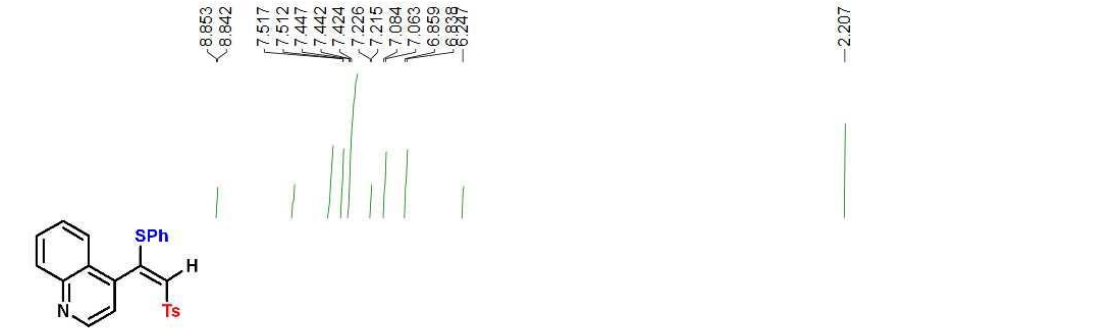
28


7.571
7.554
7.550
7.397
7.381
7.377
7.351
7.331
7.260
7.199
7.192
7.187
7.179
7.127
7.107
6.998
6.995
6.985
6.982
6.199
2.358
SePh
H
Ts
S
2.19
6.47
1.19
2.35
1.06
1.00
3.18
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

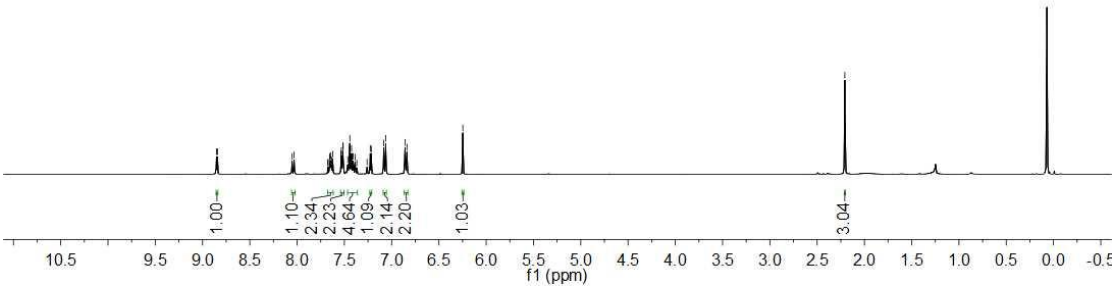
29

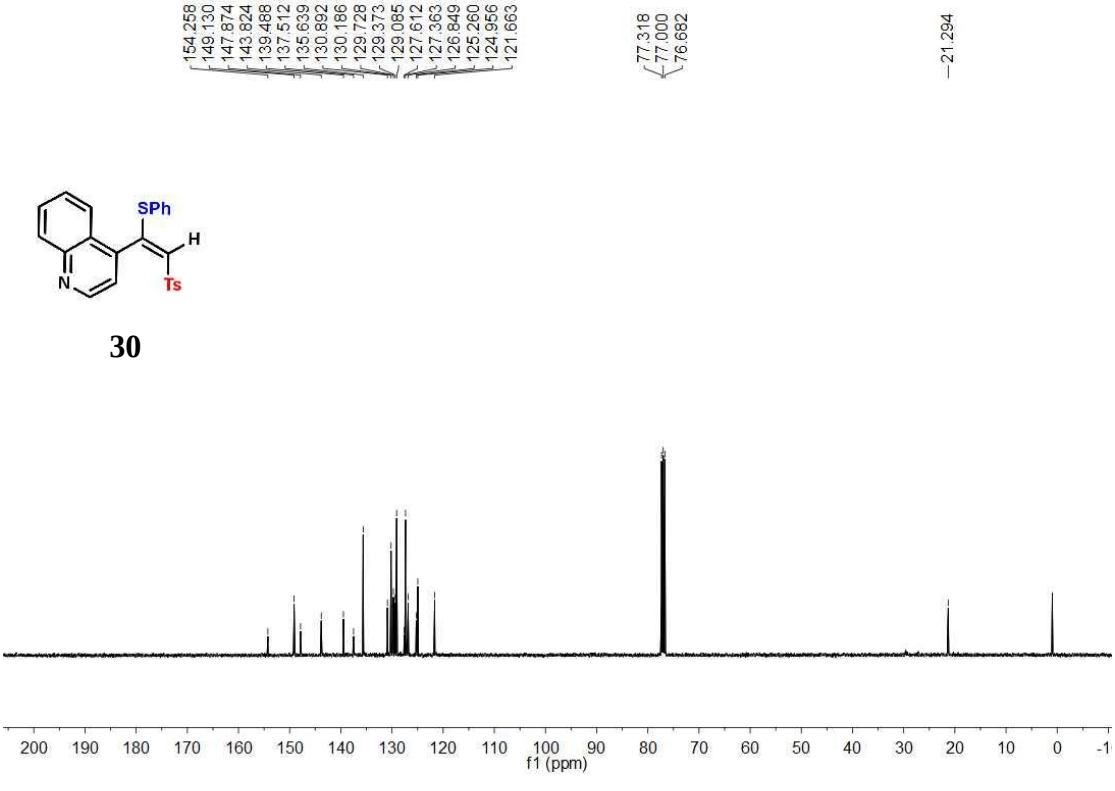

151.193
143.525
138.581
136.351
134.231
130.091
129.993
129.162
128.293
127.193
126.995
126.819
126.421
125.170
77.317
77.000
76.682
21.499
SePh
H
Ts
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

29

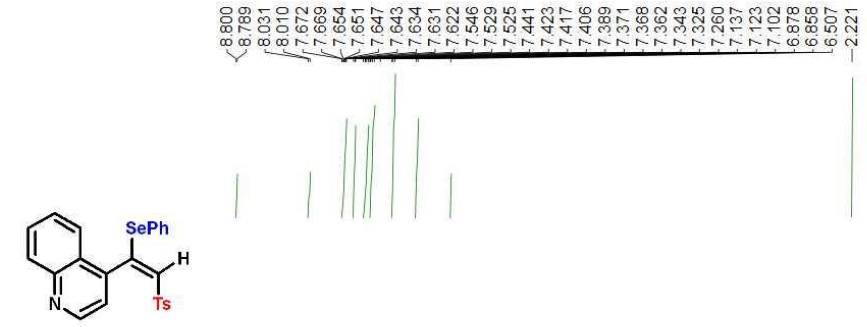

8.853
8.842
7.517
7.512
7.447
7.442
7.424
7.226
7.215
7.084
7.063
6.859
2.207
SPh
H
N
Ts

30

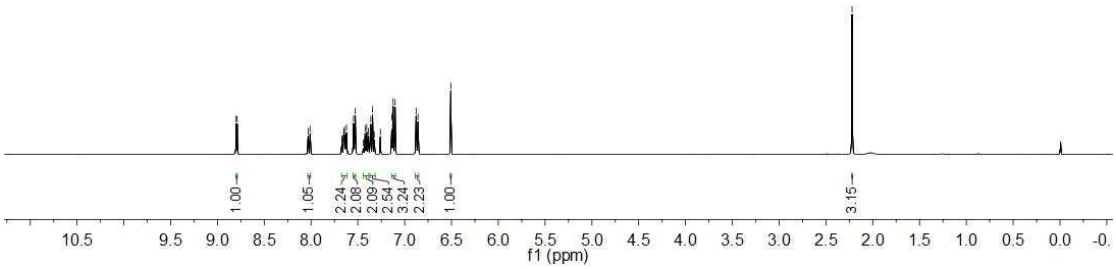

1.00
1.10
2.34
2.23
4.64
1.09
2.14
2.20
1.03
3.04
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

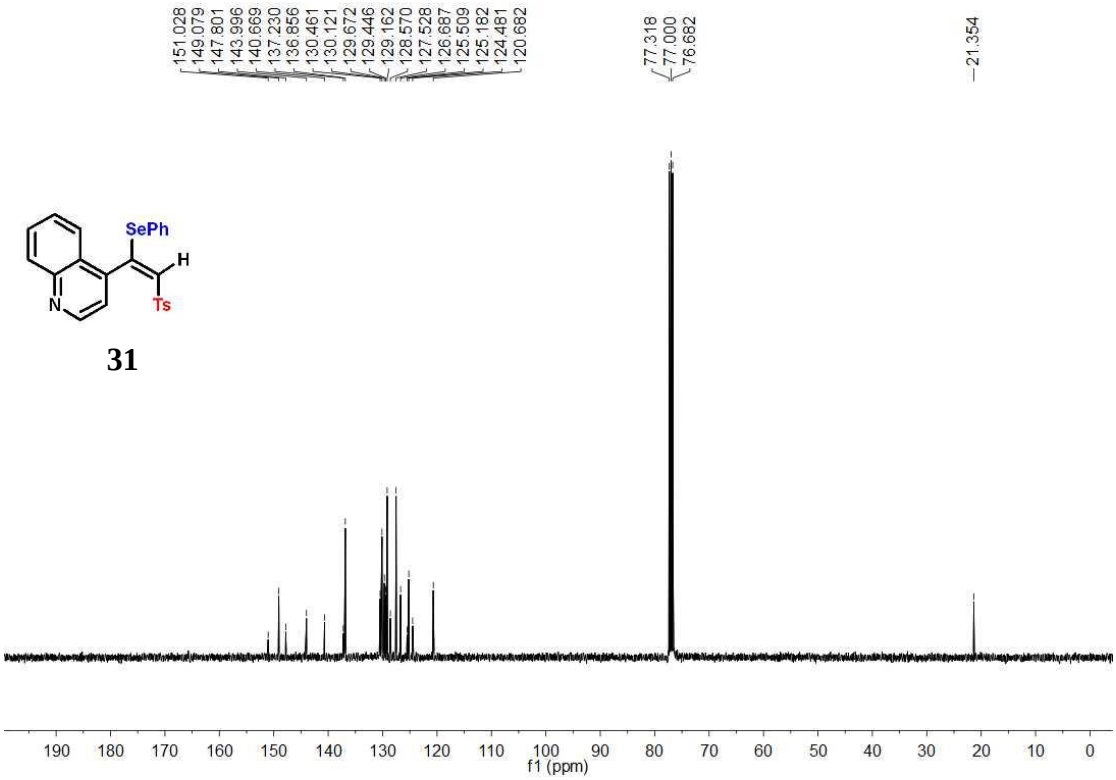

154.258
149.130
147.874
143.824
139.488
137.512
135.639
130.892
130.186
129.728
129.373
129.085
127.612
127.363
126.849
125.260
124.956
121.663
77.318
77.000
76.682
21.294
SPh
H
N
Ts
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

30

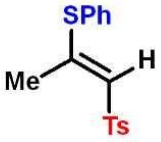

8.800
8.789
8.031
8.010
7.672
7.669
7.654
7.651
7.647
7.643
7.634
7.631
7.622
7.546
7.529
7.525
7.441
7.423
7.417
7.406
7.389
7.371
7.368
7.362
7.343
7.325
7.260
7.137
7.123
7.102
6.878
6.858
6.507
2.221
SePh
H
Ts
N

31


1.00
1.05
2.24
2.08
2.09
2.54
3.24
2.23
1.00
3.15
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

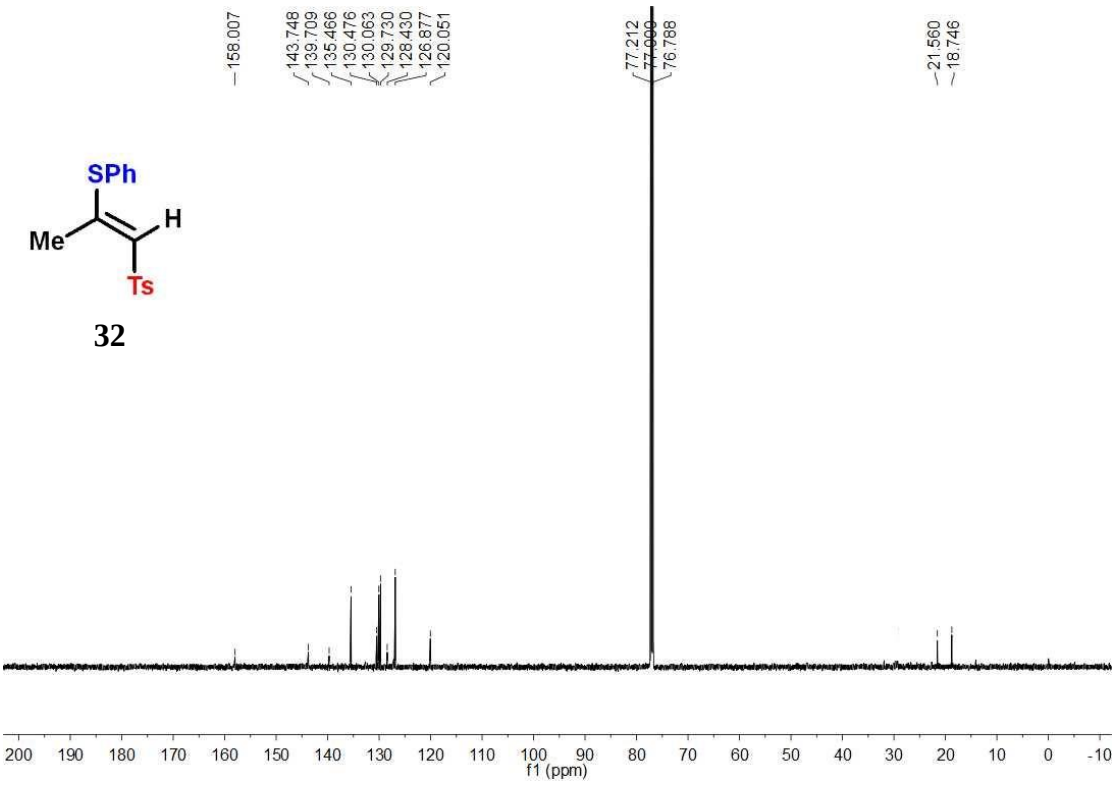

151.028
149.079
147.801
143.996
140.669
137.230
136.856
130.461
130.121
129.672
129.446
129.162
128.570
127.528
126.687
125.509
125.162
124.481
120.682
77.318
77.000
76.682
21.354
SePh
H
Ts
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

31


SPh
Me
H
Ts

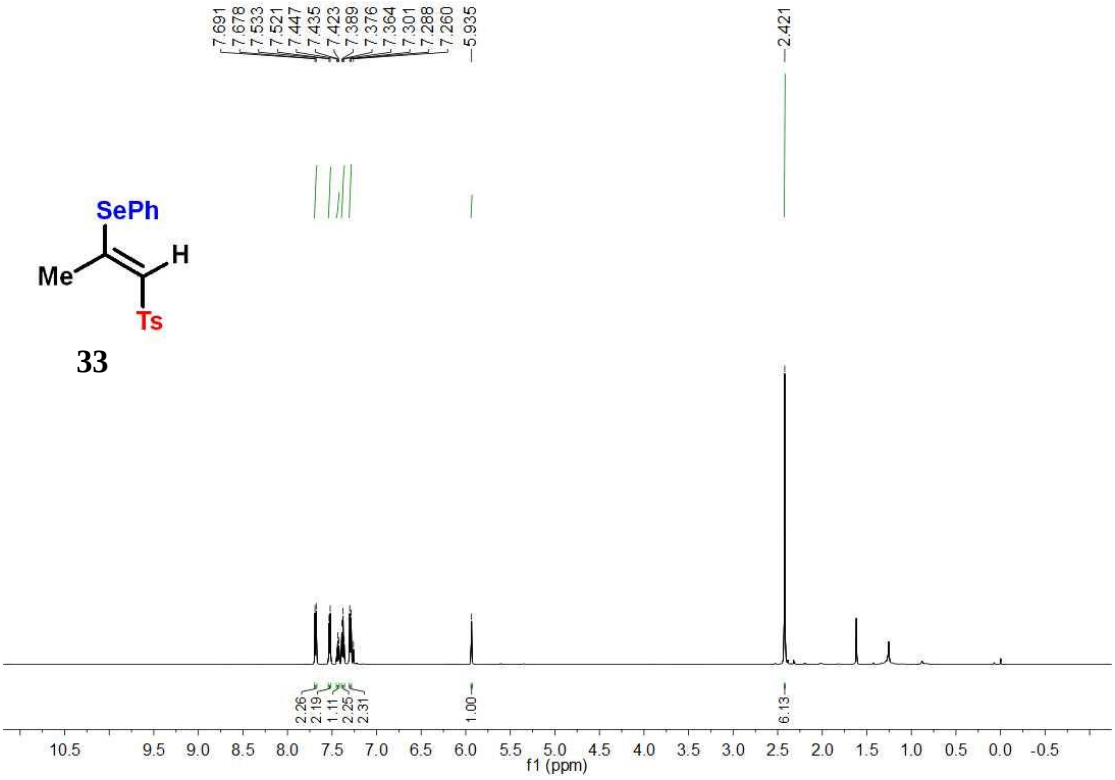
32

7.681
7.668
7.452
7.448
7.438
7.428
7.418
7.296
7.283
7.260
5.624
2.418
2.339
1.92
4.67
2.04
0.87
3.00
2.88
f1 (ppm)

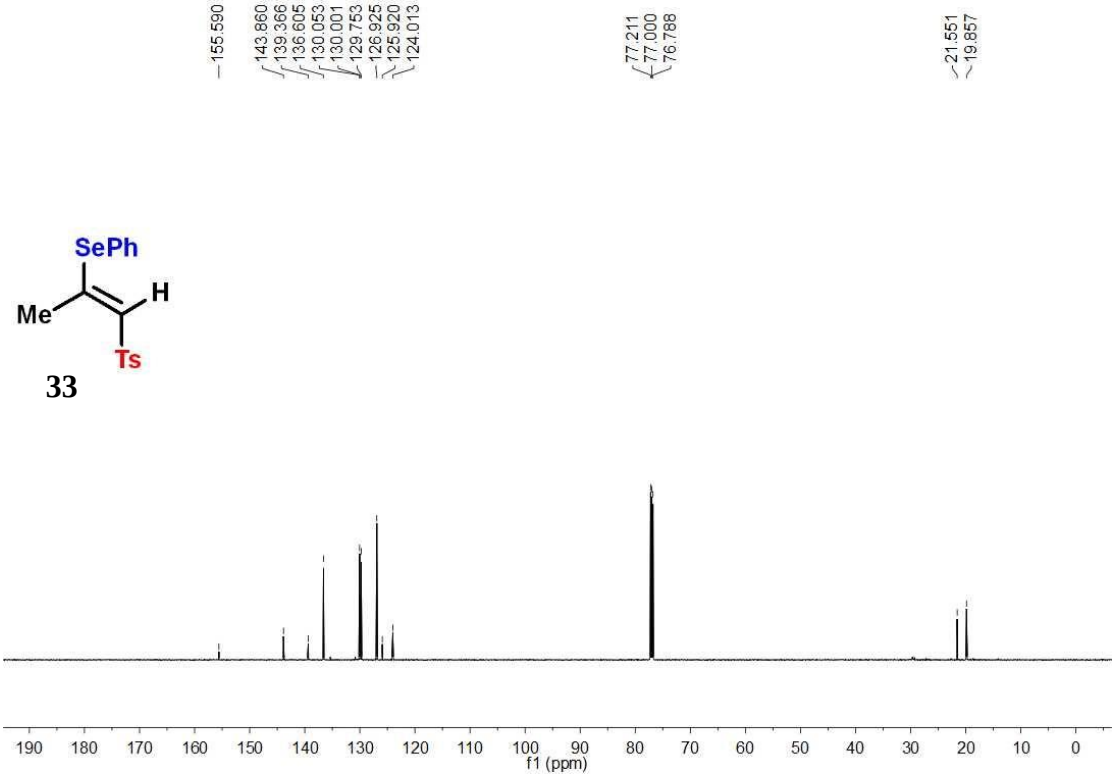

158.007
143.748
139.709
135.466
130.476
130.063
129.730
128.430
126.877
120.051
77.212
77.000
76.788
21.560
18.746
f1 (ppm)

SPh
Me
H
Ts

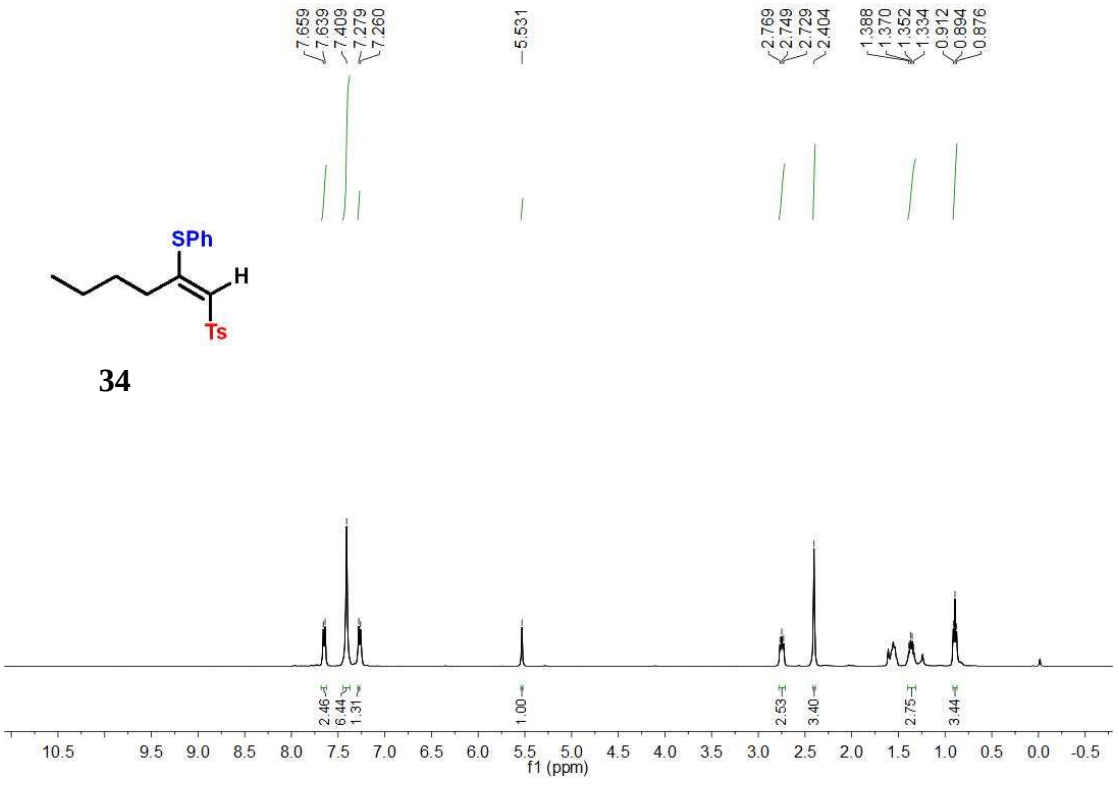
32


7.691
7.678
7.533
7.521
7.447
7.435
7.423
7.389
7.376
7.364
7.301
7.288
7.260
5.935
2.421
SePh
Me
H
Ts
2.26
2.19
1.11
2.25
2.31
1.00
6.13
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

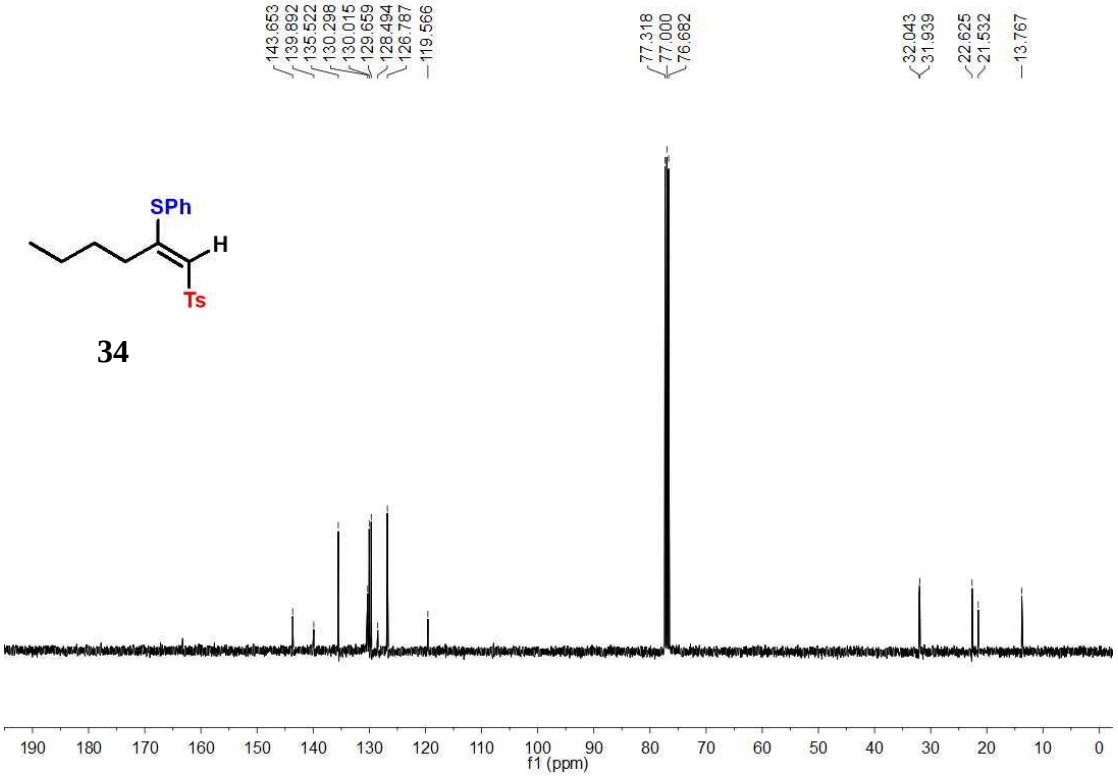
33


155.590
143.860
139.366
136.605
130.053
130.001
129.753
126.925
125.920
124.013
77.211
77.000
76.788
21.551
19.857
SePh
Me
H
Ts
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

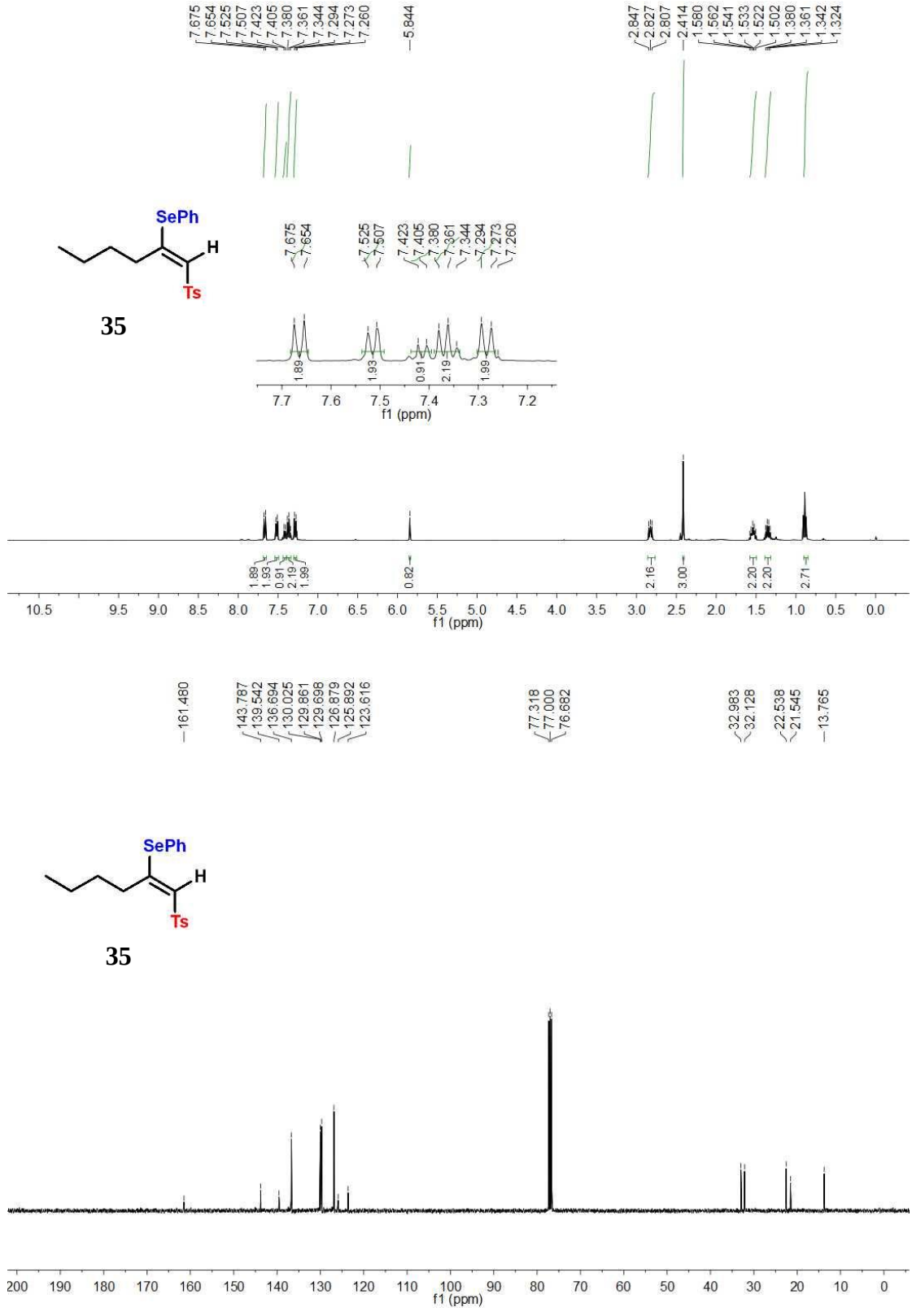
33


7.659
7.639
7.409
7.279
7.260
5.531
2.769
2.749
2.729
2.404
1.388
1.370
1.352
1.334
0.912
0.894
0.876
SPh
H
Ts
2.46
6.44
1.31
1.00
2.53
3.40
2.75
3.44
f1 (ppm)

34


143.653
139.892
135.522
130.298
130.015
129.659
128.494
126.787
119.566
77.318
77.000
76.682
32.043
31.939
22.625
21.532
13.767
SPh
H
Ts
f1 (ppm)

34


7.675
7.654
7.525
7.507
7.423
7.405
7.380
7.361
7.344
7.294
7.273
7.260
5.844
2.847
2.827
2.807
2.414
1.580
1.562
1.541
1.533
1.522
1.502
1.380
1.361
1.342
1.324
SePh
H
Ts
35
1.89
1.93
0.91
2.19
1.99
f1 (ppm)
0.82
2.16
3.00
2.20
2.20
2.71
161.480
143.787
139.542
136.694
130.025
129.861
129.698
126.879
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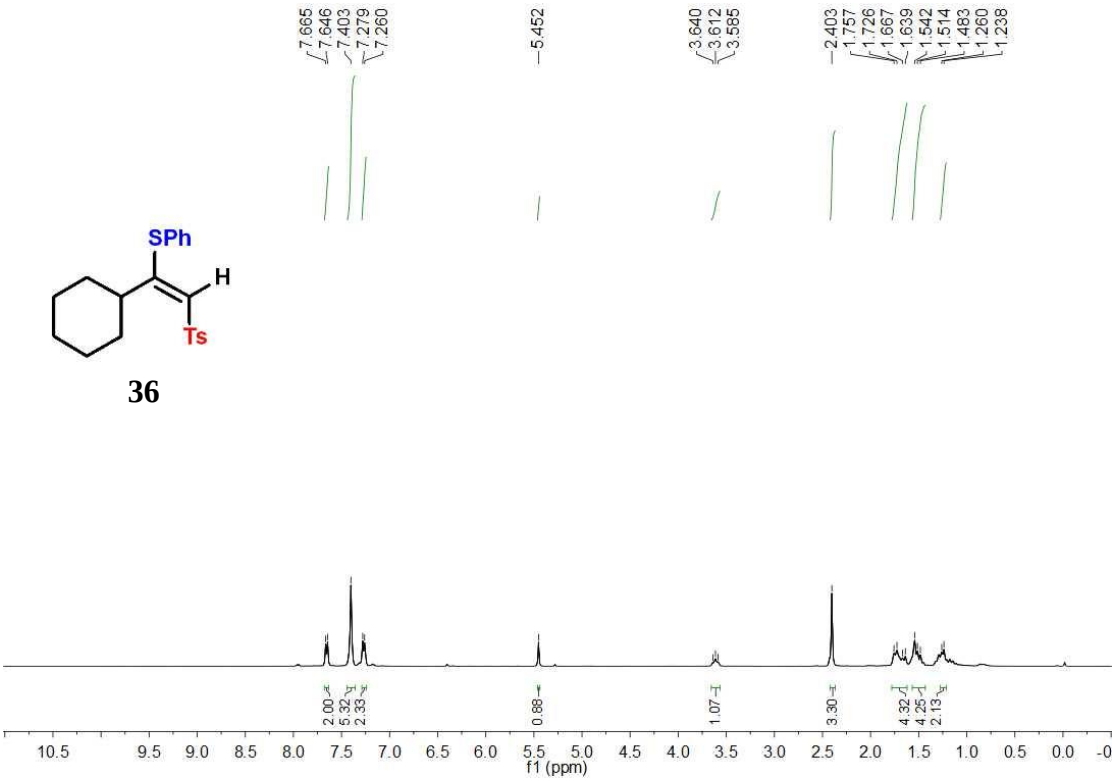
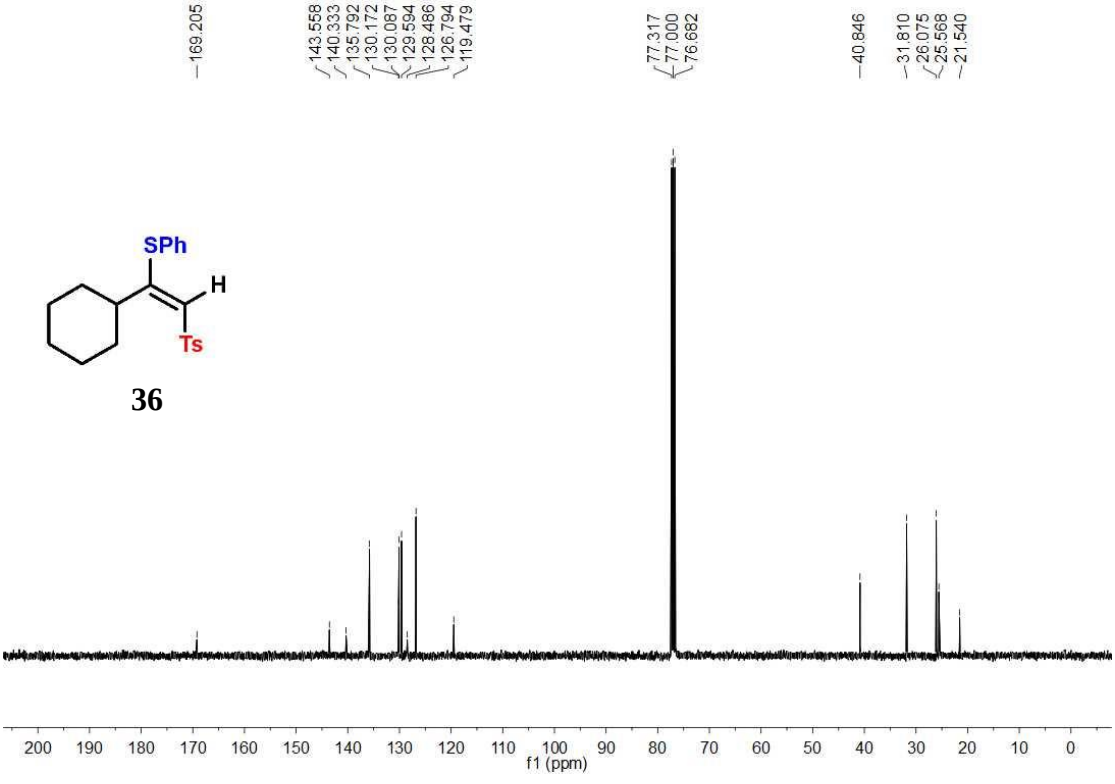
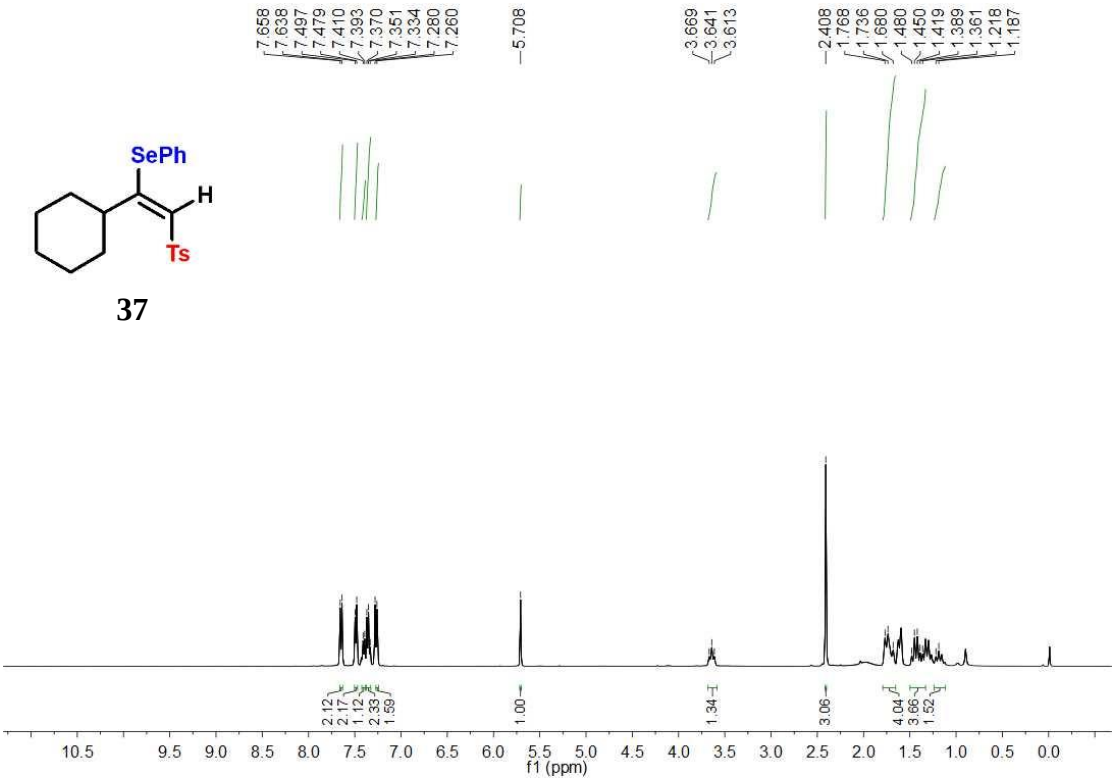
32.983
32.128
22.538
21.545
13.765


7.665
7.646
7.403
7.279
7.260
5.452
3.640
3.612
3.585
2.403
1.757
1.726
1.667
1.639
1.542
1.514
1.483
1.260
1.238
SPh
H
Ts
2.00
5.32
2.33
0.88
1.07
3.30
4.32
4.25
2.13
f1 (ppm)

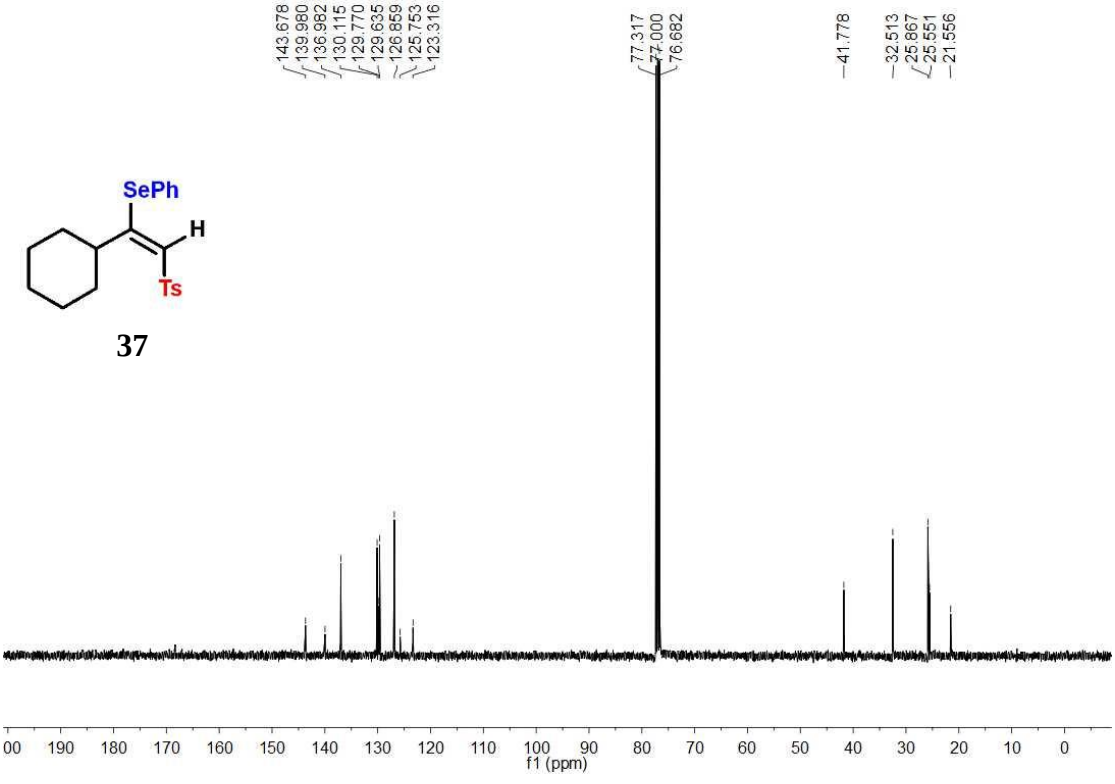
36


169.205
143.558
140.333
135.792
130.172
130.087
129.594
128.486
126.794
119.479
77.317
77.000
76.682
40.846
31.810
26.075
25.568
21.540
SPh
H
Ts
f1 (ppm)

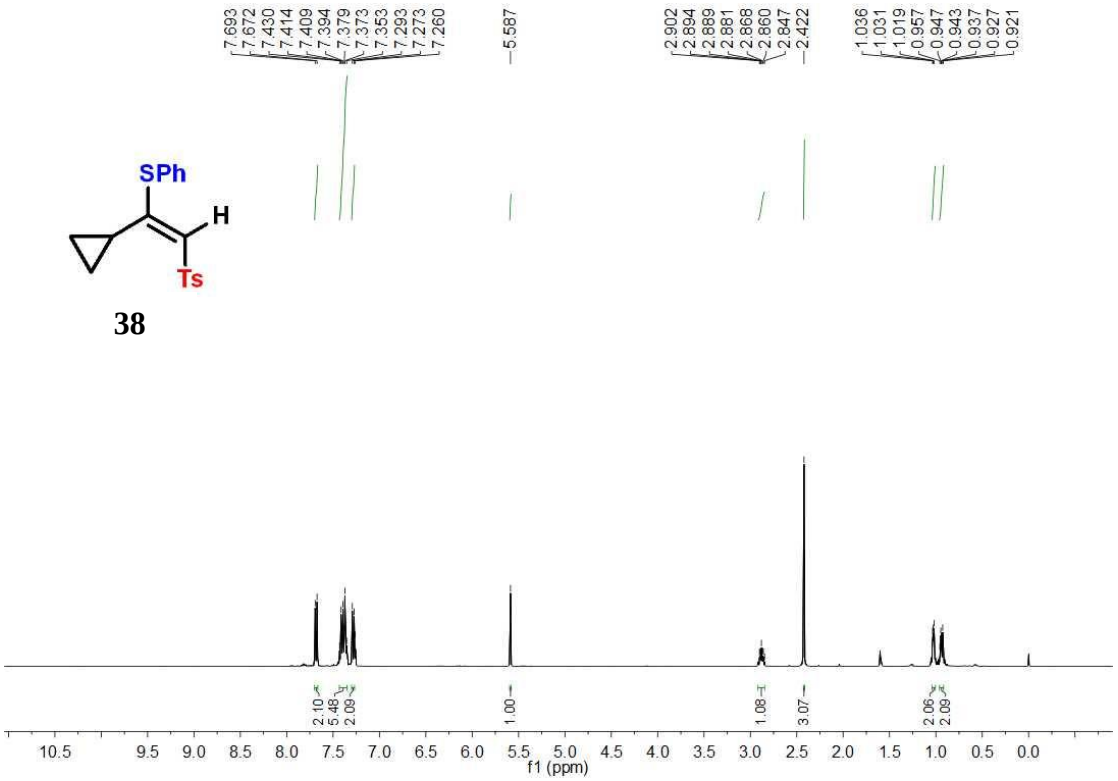
36


7.658
7.638
7.497
7.479
7.410
7.393
7.370
7.351
7.334
7.280
7.260
5.708
3.669
3.641
3.613
2.408
1.768
1.736
1.680
1.480
1.450
1.419
1.389
1.361
1.218
1.187
SePh
H
Ts
2.12
2.17
1.12
2.33
1.59
1.00
1.34
3.06
4.04
3.66
1.52
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

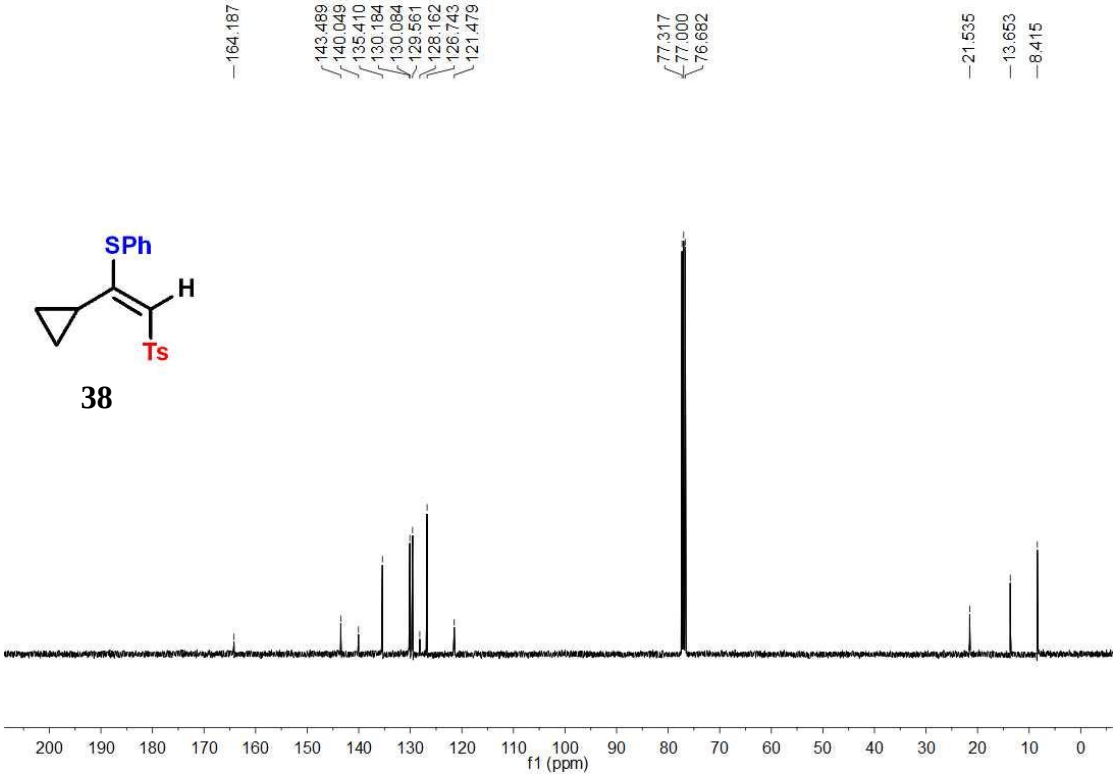
37


143.678
139.980
136.982
130.115
129.770
129.635
126.859
125.753
123.316
77.317
77.000
76.682
41.778
32.513
25.867
25.551
21.556
SePh
H
Ts
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

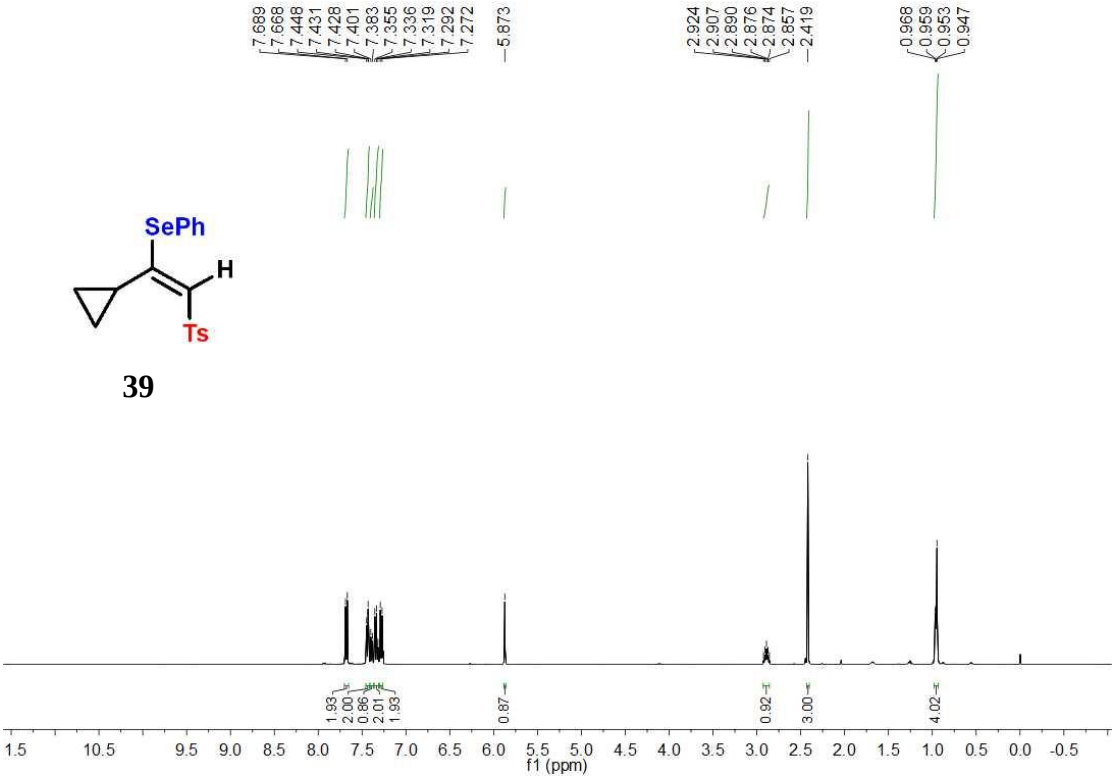
37


7.693
7.672
7.430
7.414
7.409
7.394
7.379
7.373
7.353
7.293
7.273
7.260
5.587
2.902
2.894
2.889
2.881
2.868
2.860
2.847
2.422
1.036
1.031
1.019
0.957
0.947
0.943
0.937
0.927
0.921
SPh
H
Ts
2.10
5.48
2.09
1.00
1.08
3.07
2.06
2.09
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

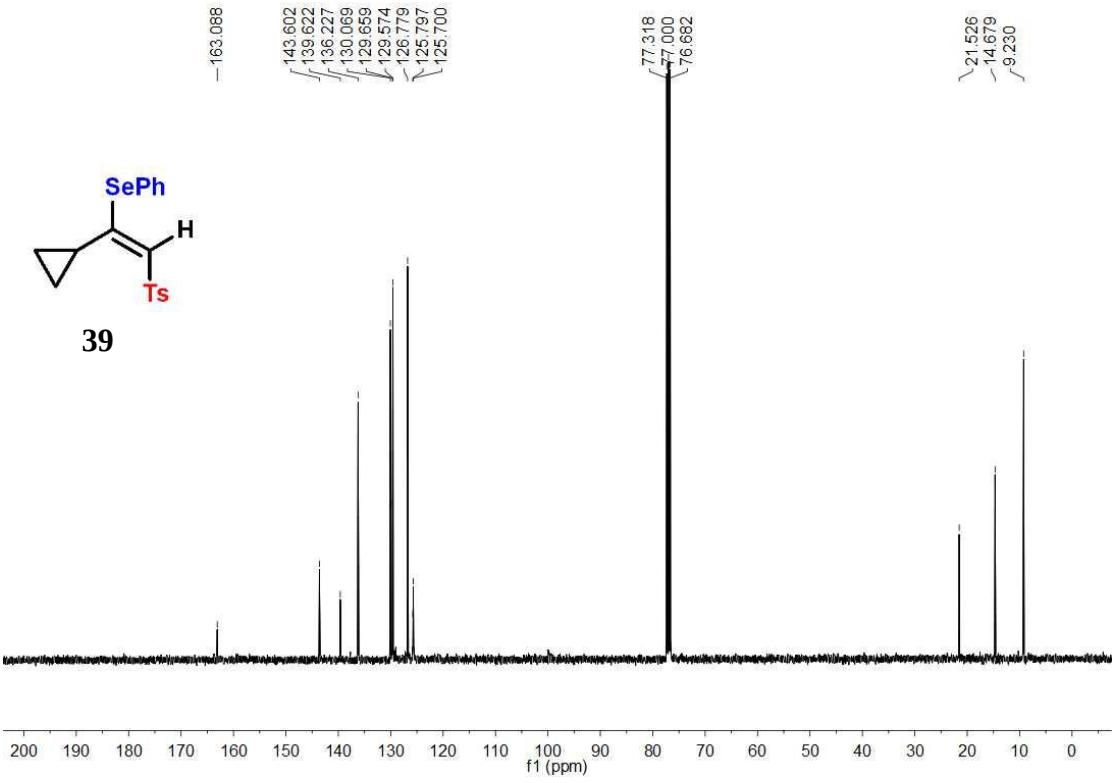
38


164.187
143.489
140.049
135.410
130.184
130.084
129.561
128.162
126.743
121.479
77.317
77.000
76.682
21.535
13.653
8.415
SPh
H
Ts
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

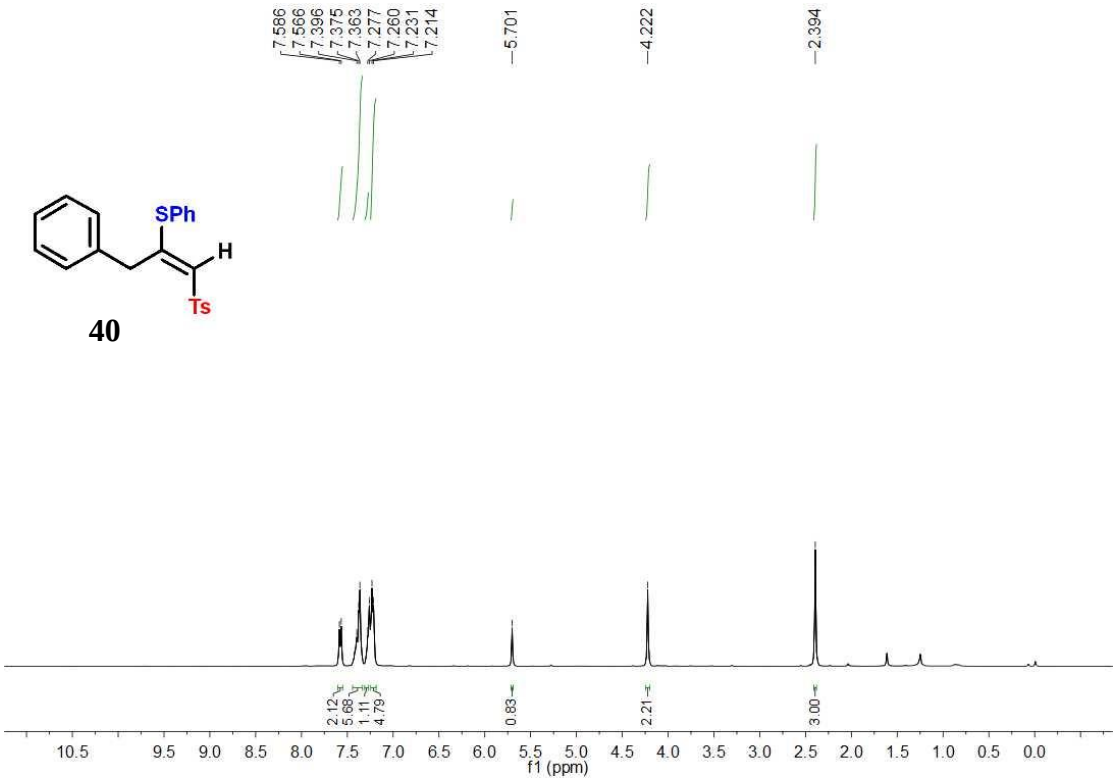
38


7.689
7.668
7.448
7.431
7.428
7.401
7.383
7.355
7.336
7.319
7.292
7.272
5.873
2.924
2.907
2.890
2.876
2.874
2.857
2.419
0.968
0.959
0.953
0.947
SePh
H
Ts
1.93
2.00
0.86
2.01
1.93
0.87
0.92
3.00
4.02
f1 (ppm)

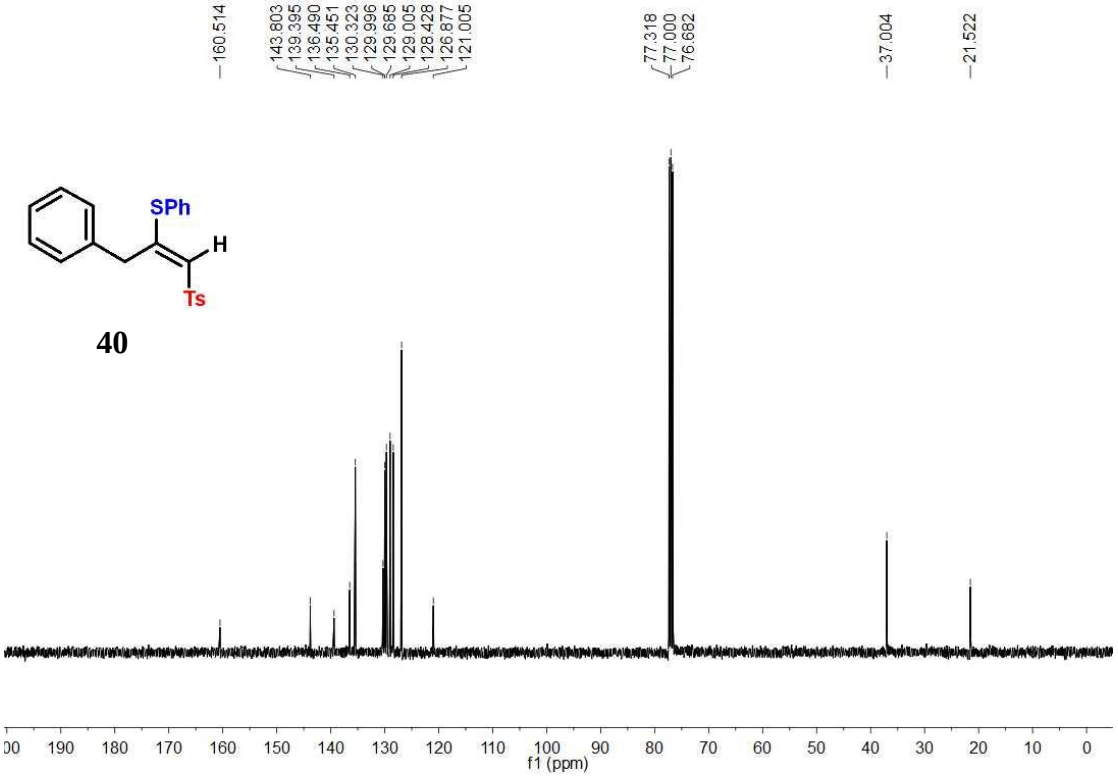
39


163.088
143.602
139.622
136.227
130.069
129.659
129.574
126.779
125.797
125.700
77.318
77.000
76.682
21.526
14.679
9.230
SePh
H
Ts
f1 (ppm)

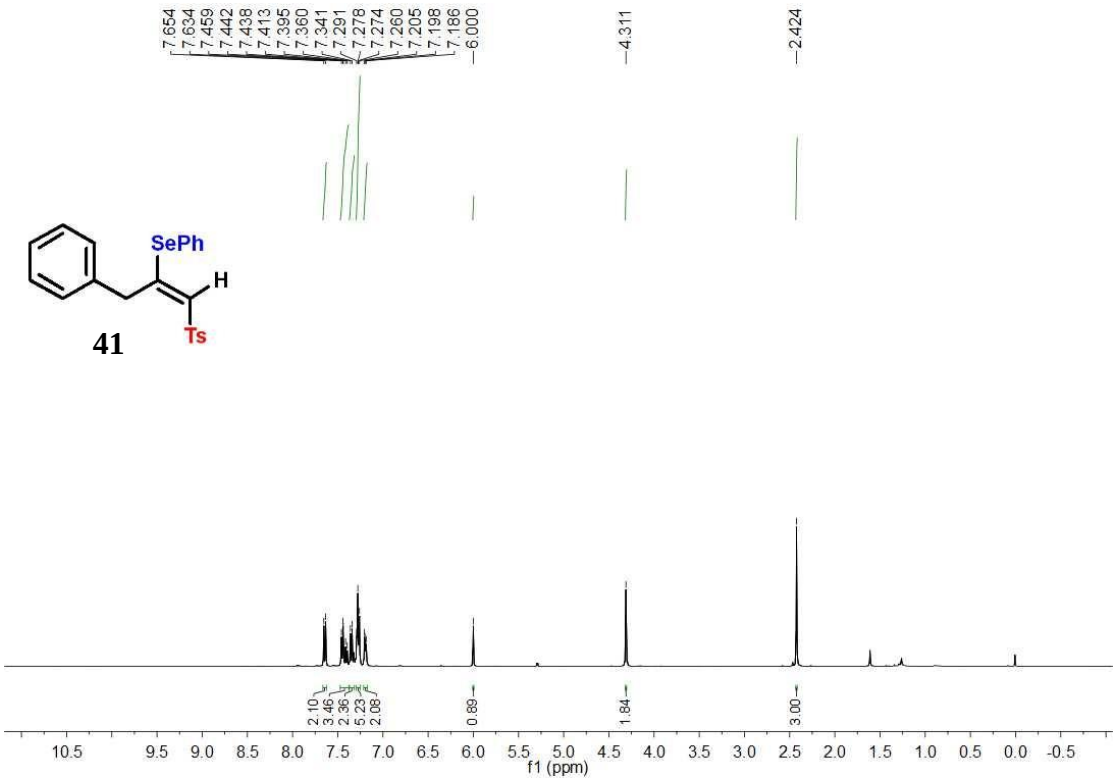
39


7.586
7.566
7.396
7.375
7.363
7.277
7.260
7.231
7.214
5.701
4.222
2.394
SPh
H
Ts
2.12
5.68
1.11
4.79
0.83
2.21
3.00
f1 (ppm)

40


160.514
143.803
139.395
136.490
135.451
130.323
129.996
129.685
129.005
128.428
126.877
121.005
77.318
77.000
76.682
37.004
21.522
SPh
H
Ts
f1 (ppm)

40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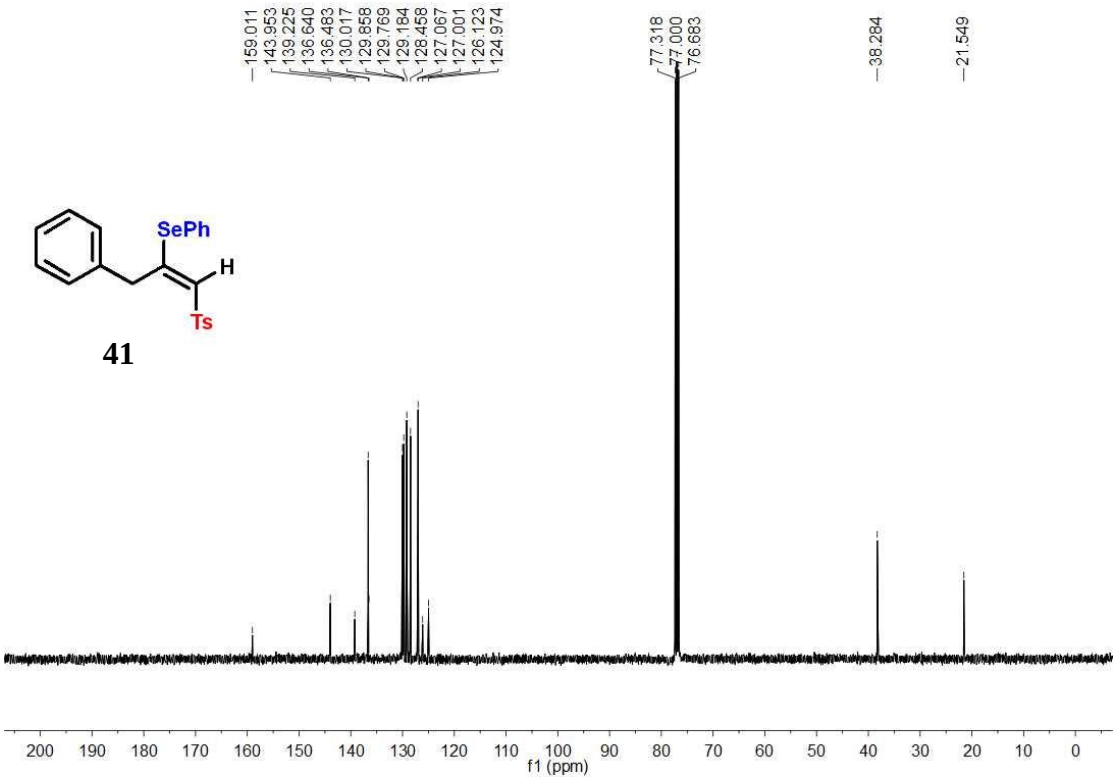
7.634
7.459
7.442
7.438
7.413
7.395
7.360
7.341
7.291
7.278
7.274
7.260
7.205
7.198
7.186
6.000
4.311
2.424
SePh
H
Ts
41
2.10
3.46
2.36
5.23
2.08
0.89
1.84
3.00
f1 (ppm)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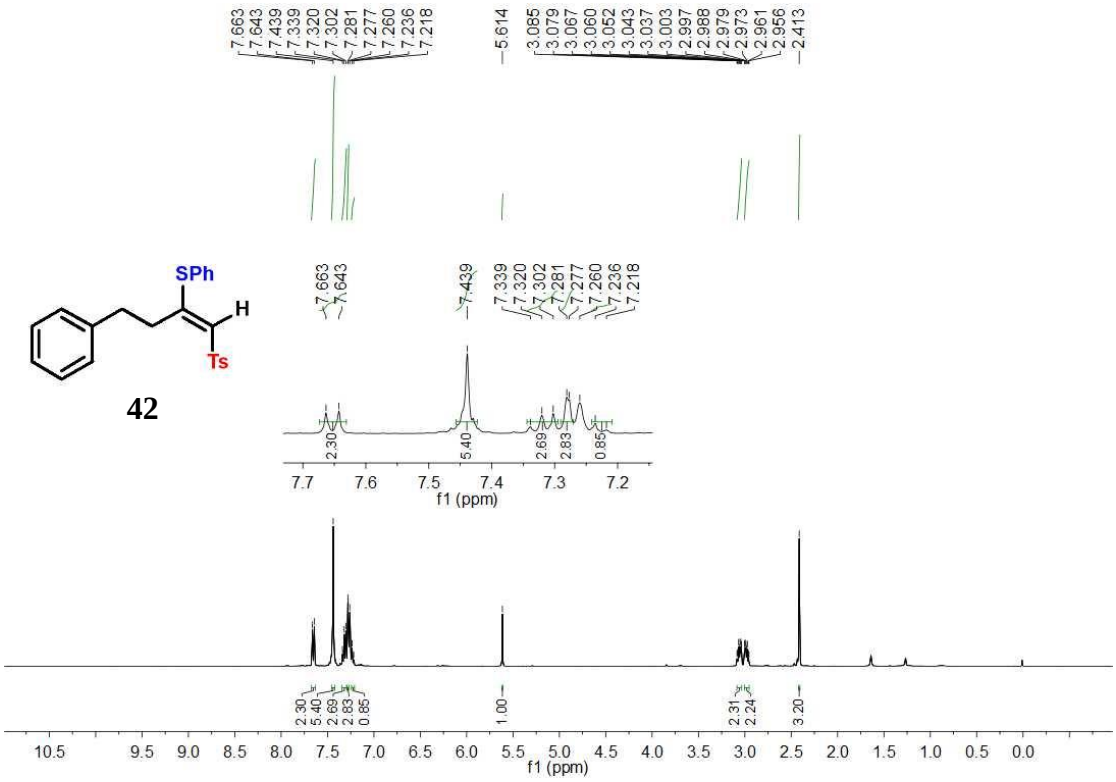
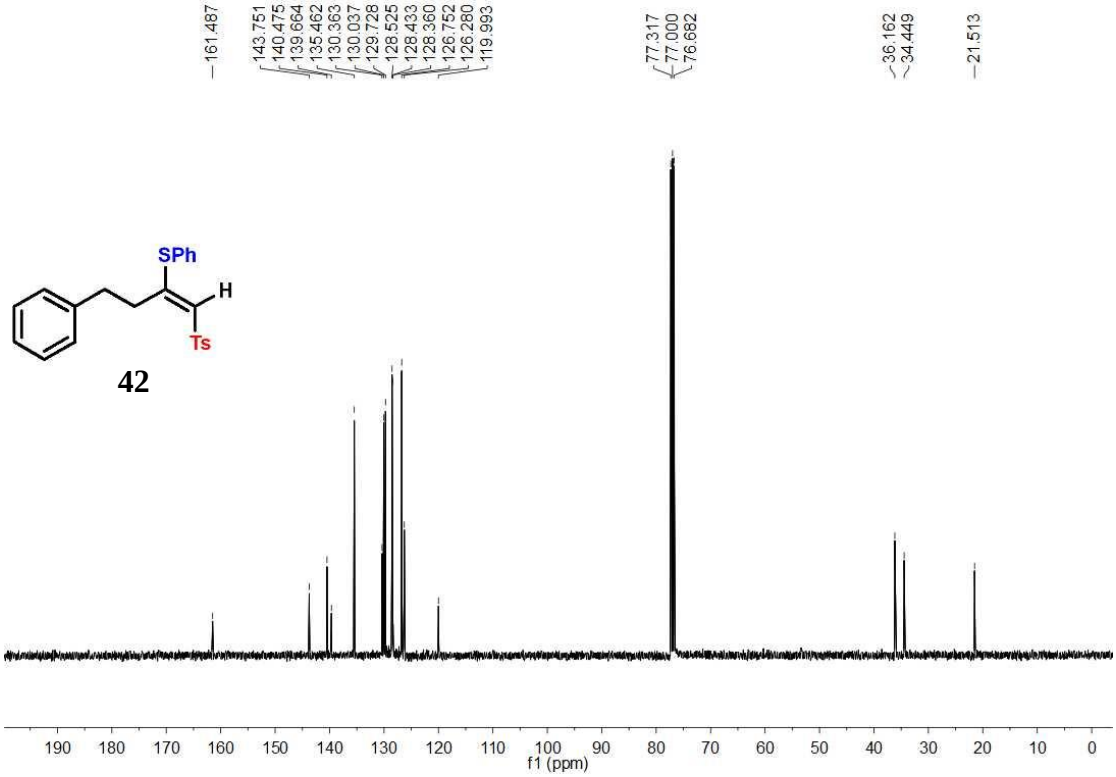
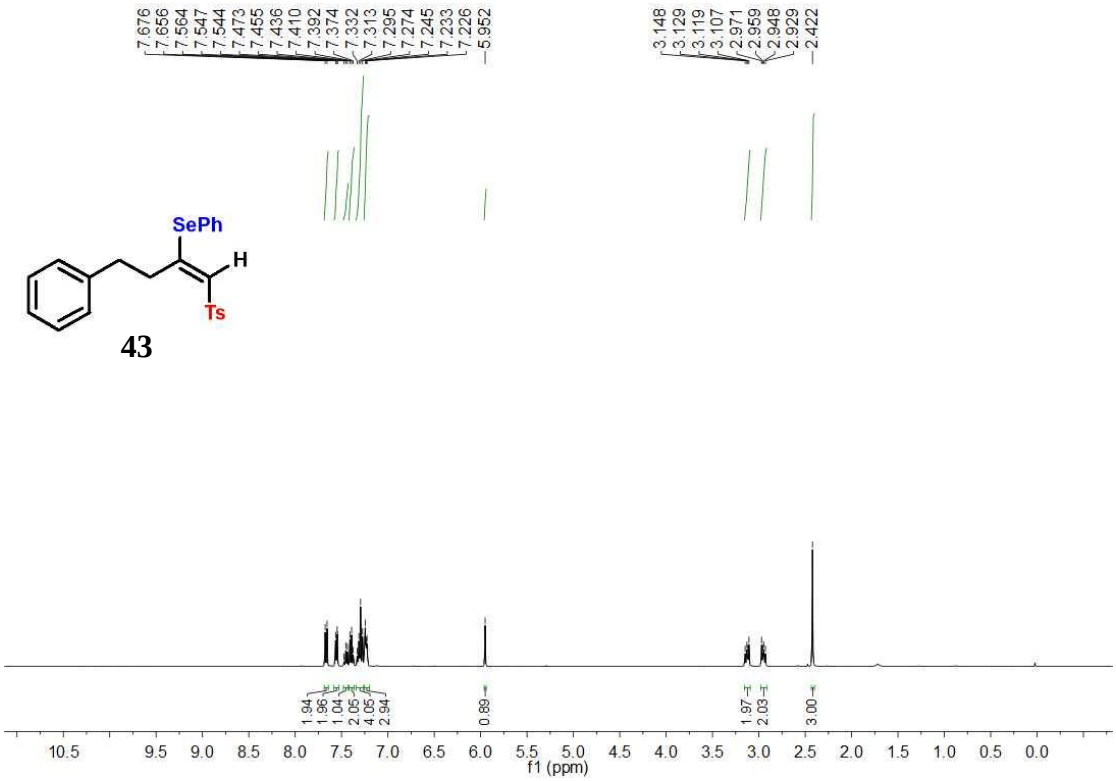
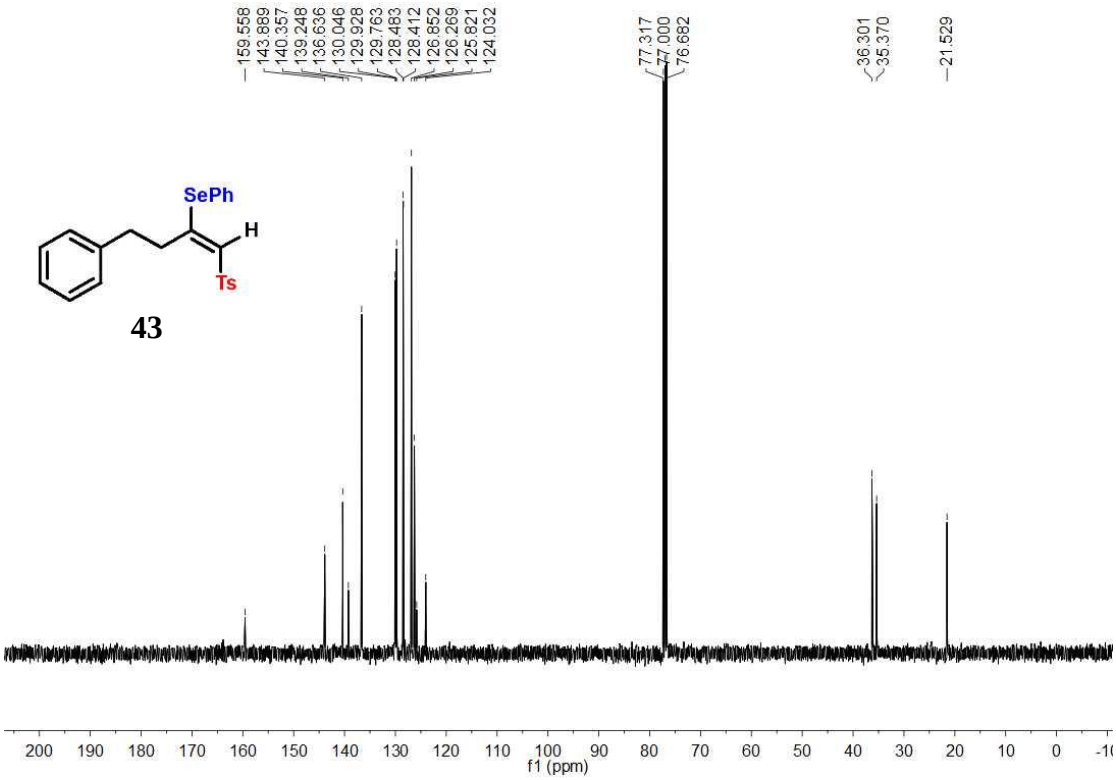
143.953
139.225
136.640
136.483
130.017
129.858
129.769
129.184
128.458
127.067
127.001
126.123
124.974
77.318
77.000
76.683
38.284
21.549
SePh
H
Ts
41
f1 (ppm)


7.663
7.643
7.439
7.339
7.320
7.302
7.281
7.277
7.260
7.236
7.218
5.614
3.085
3.079
3.067
3.060
3.052
3.043
3.037
3.003
2.997
2.988
2.979
2.973
2.961
2.956
2.413
SPh
H
Ts
42
2.30
5.40
2.69
2.83
0.85
1.00
2.31
2.24
3.20
f1 (ppm)

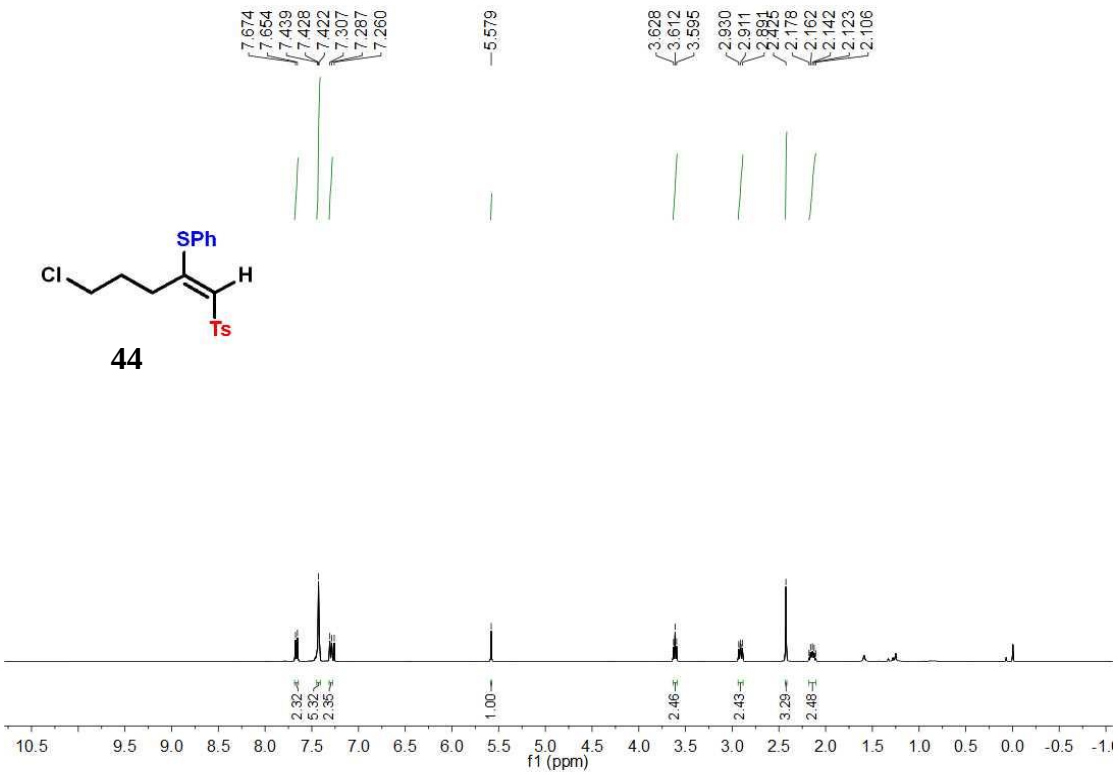

161.487
143.751
140.475
139.664
135.462
130.363
130.037
129.728
128.525
128.433
128.360
126.752
126.280
119.993
77.317
77.000
76.682
36.162
34.449
21.513
SPh
H
Ts
42
f1 (ppm)

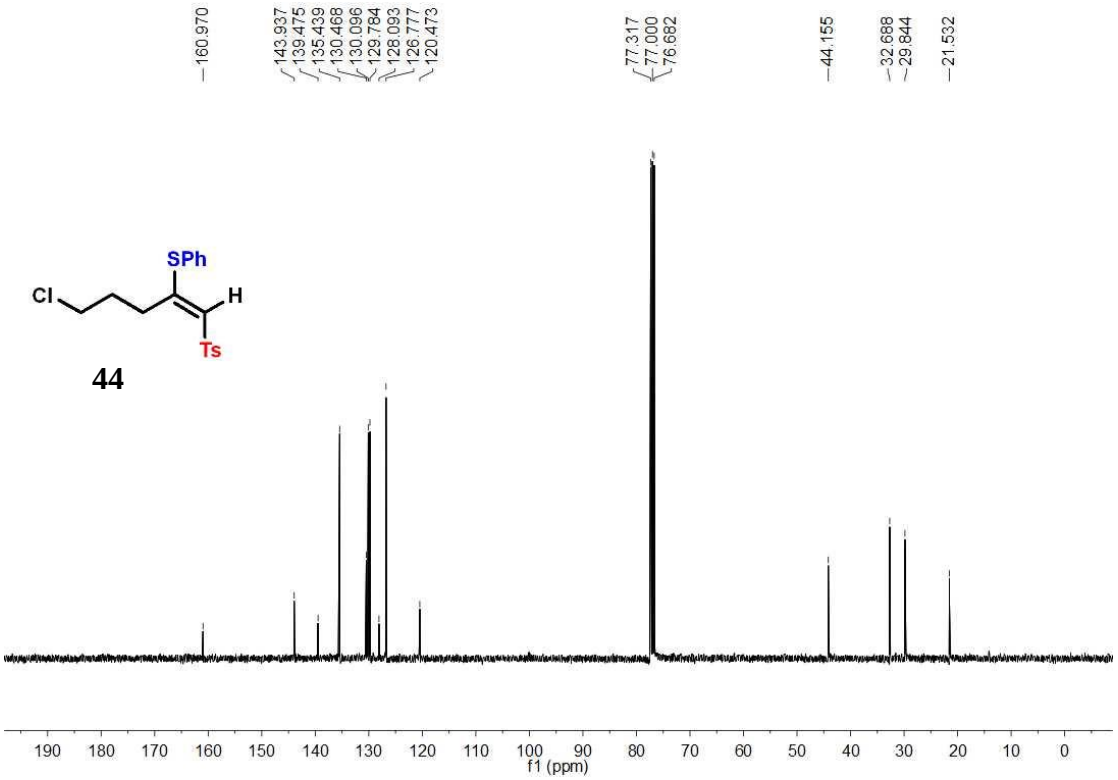

7.676
7.656
7.564
7.547
7.544
7.473
7.455
7.436
7.410
7.392
7.374
7.332
7.313
7.295
7.274
7.245
7.233
7.226
5.952
3.148
3.129
3.119
3.107
2.971
2.959
2.948
2.929
2.422
SePh
H
Ts
1.94
1.96
1.04
2.05
4.05
2.94
0.89
1.97
2.03
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

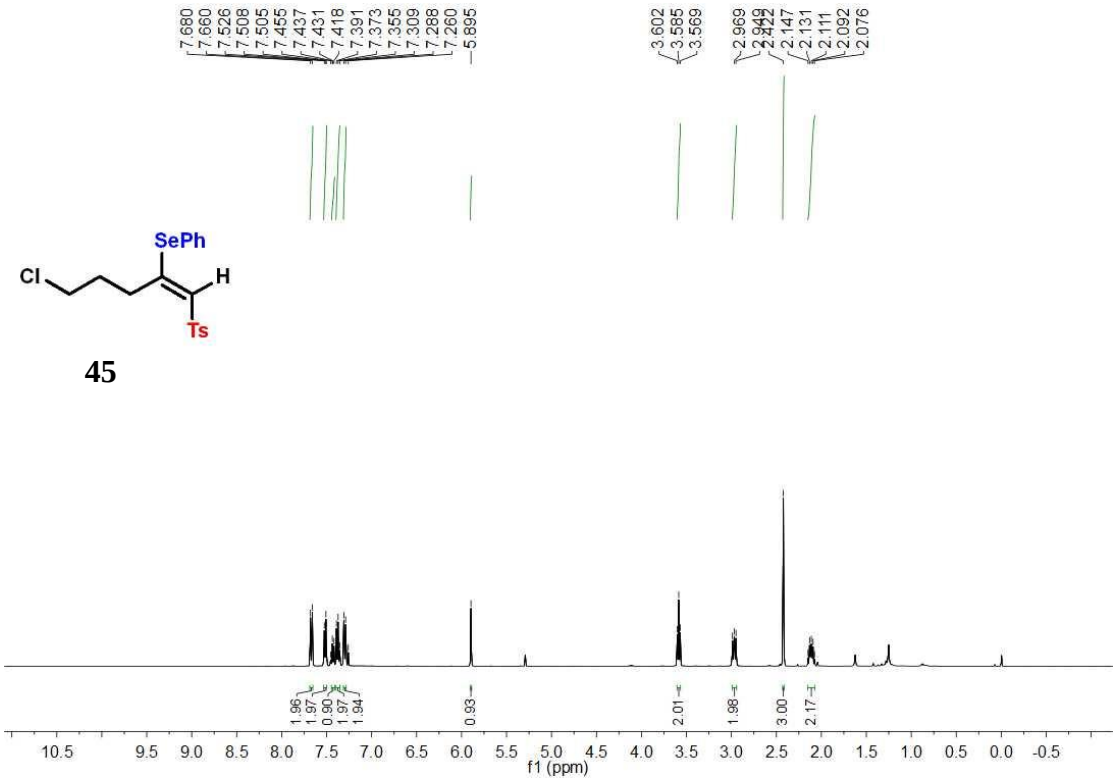
43


159.558
143.889
140.357
139.248
136.636
130.046
129.928
129.763
128.483
128.412
126.852
126.269
125.821
124.032
77.317
77.000
76.682
36.301
35.370
21.529
SePh
H
Ts
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

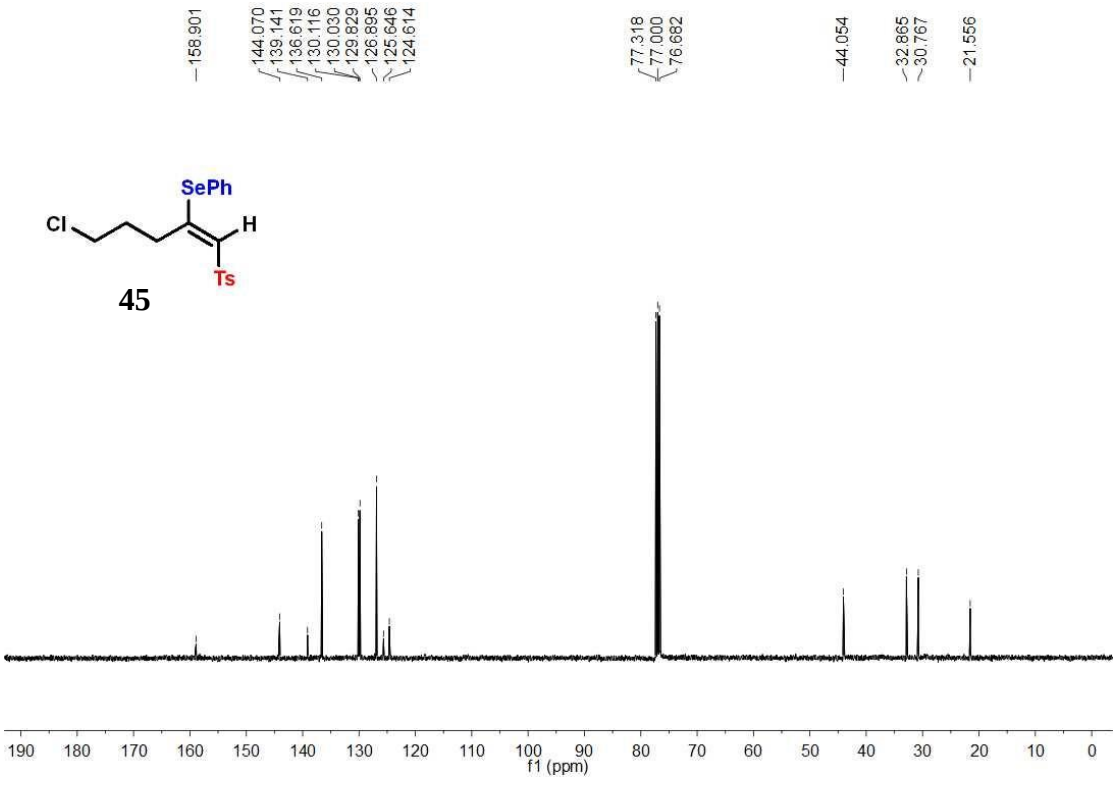
43


7.674
7.654
7.439
7.428
7.422
7.307
7.287
7.260
5.579
3.628
3.612
3.595
2.930
2.911
2.425
2.178
2.162
2.142
2.123
2.106
SPh
Cl
H
Ts
44
2.32
5.32
2.35
1.00
2.46
2.43
3.29
2.48
f1 (ppm)

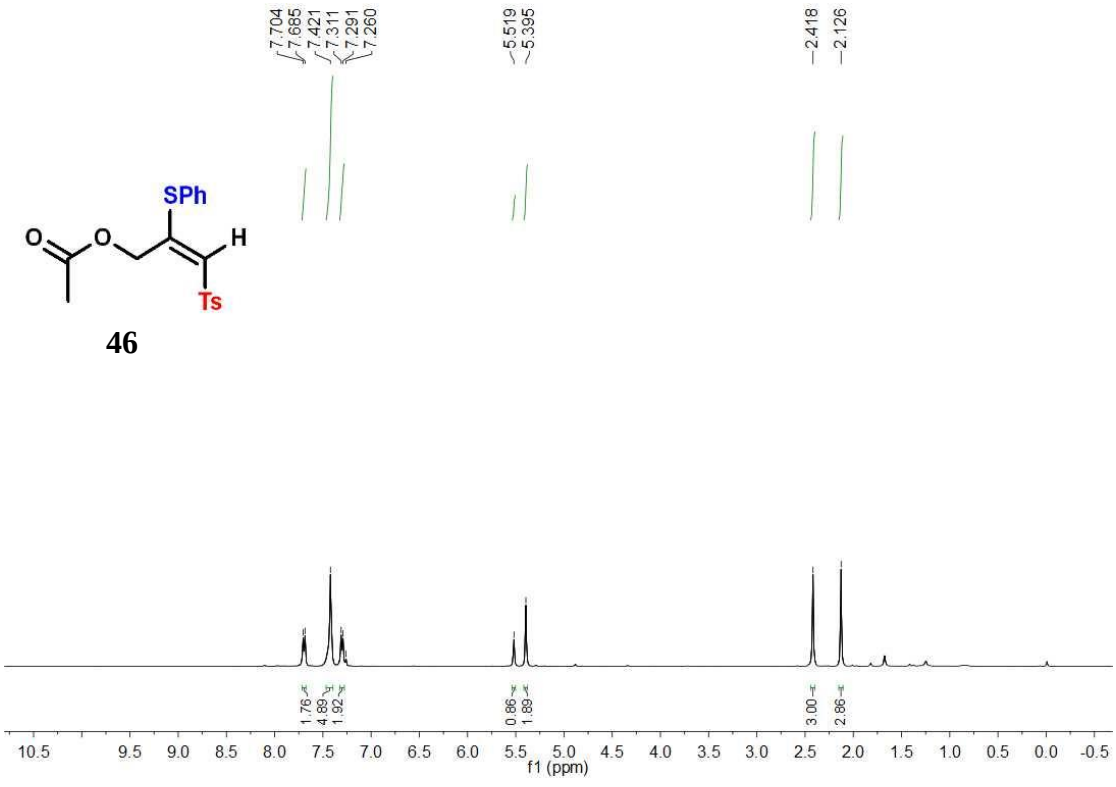

160.970
143.937
139.475
135.439
130.468
130.096
129.784
128.093
126.777
120.473
77.317
77.000
76.682
44.155
32.688
29.844
21.532
SPh
Cl
H
Ts
44
f1 (ppm)


7.680
7.660
7.526
7.508
7.505
7.455
7.437
7.431
7.418
7.391
7.373
7.355
7.309
7.288
7.260
5.895
3.602
3.585
3.569
2.969
2.147
2.131
2.111
2.092
2.076
SePh
Cl
H
Ts
1.96
1.97
0.90
1.97
1.94
0.93
2.01
1.98
3.00
2.17
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

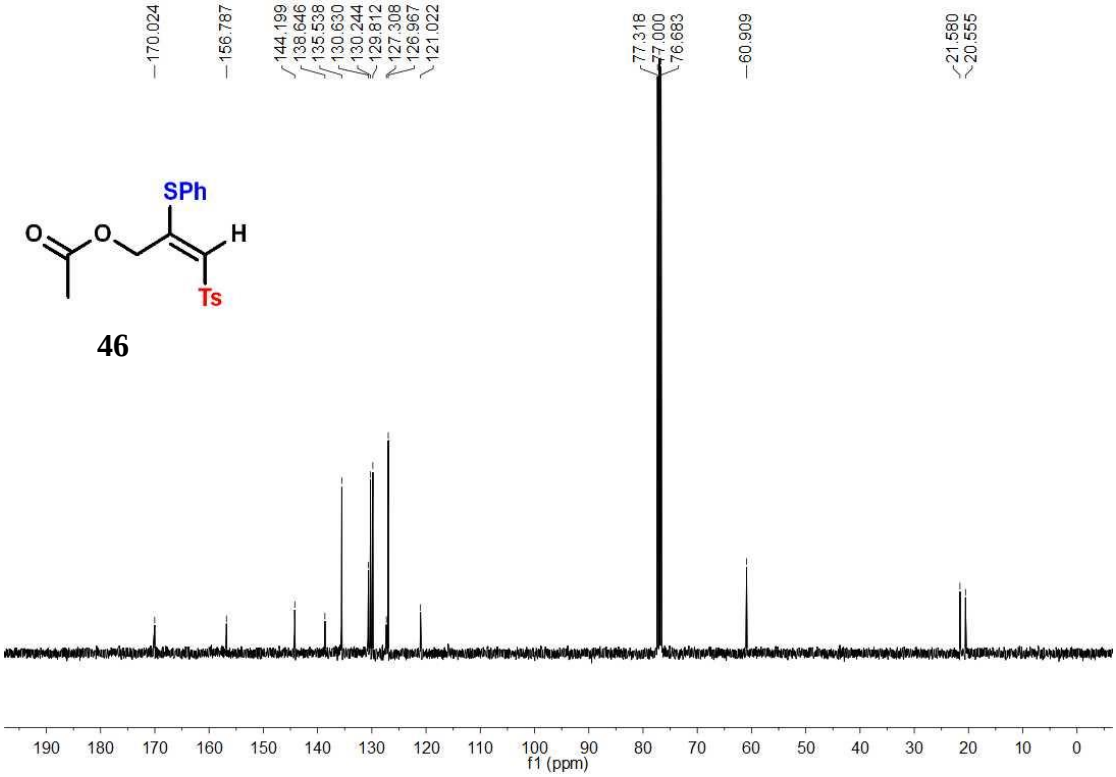
45


158.901
144.070
139.141
136.619
130.116
130.030
129.829
126.895
125.646
124.614
77.318
77.000
76.682
44.054
32.865
30.767
21.556
SePh
Cl
H
Ts
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

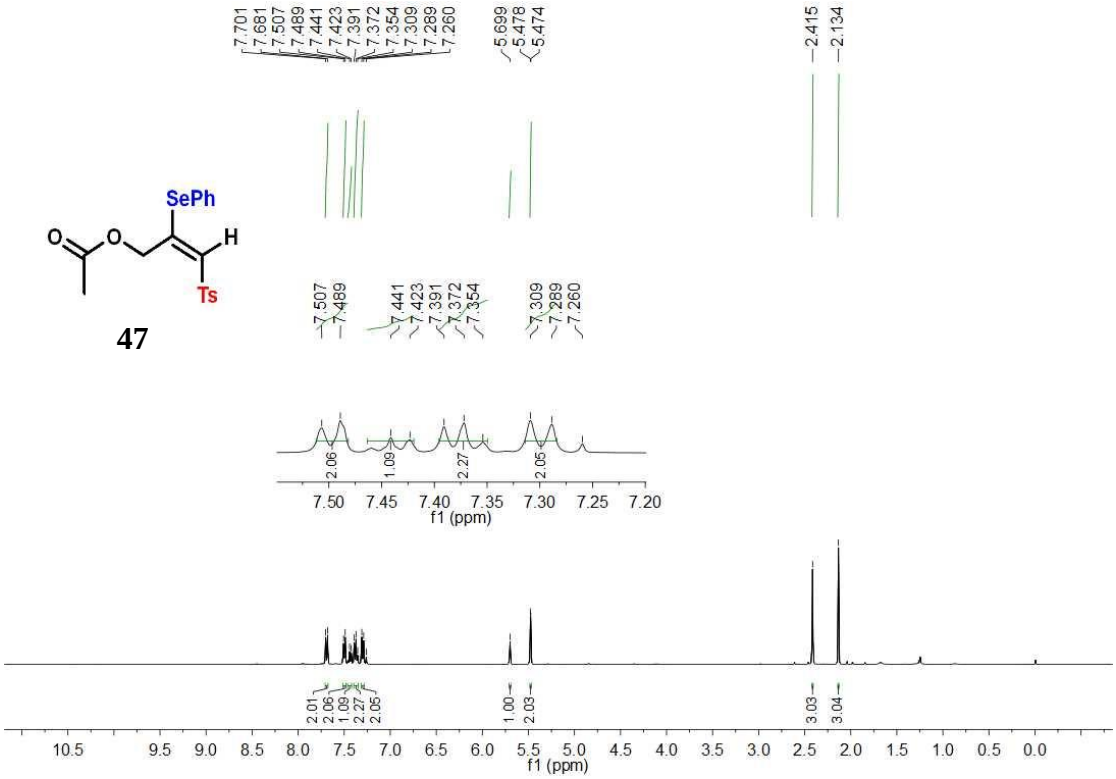
45

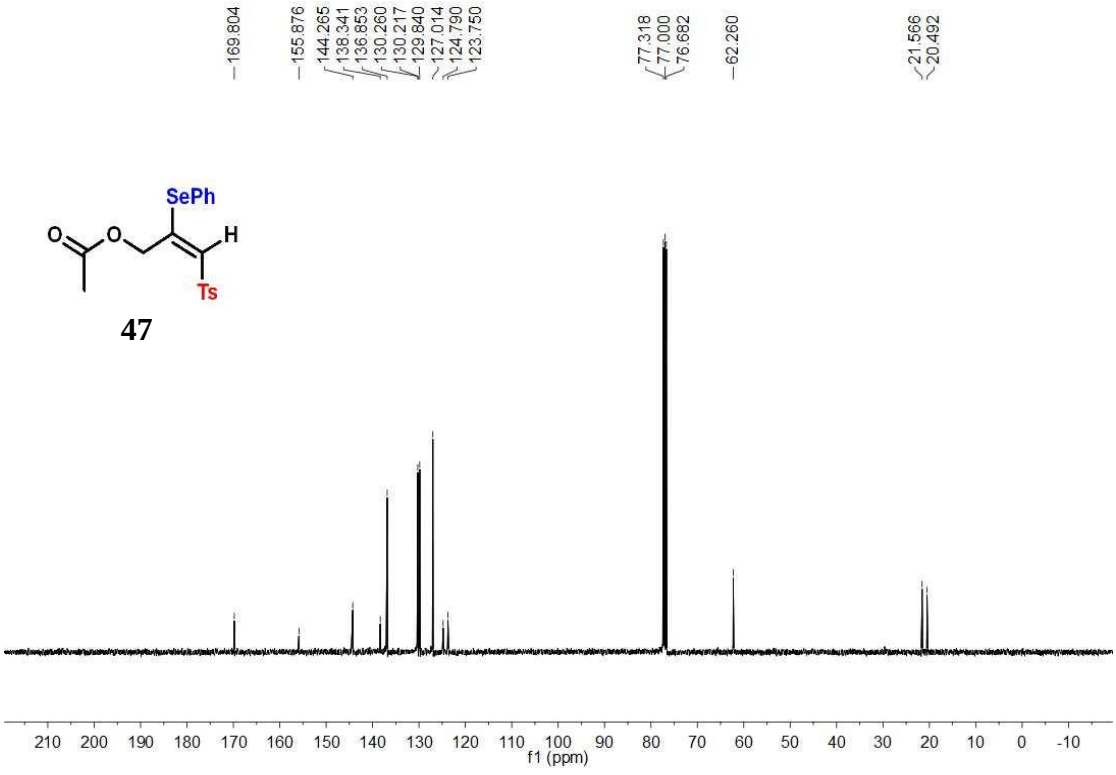

7.704
7.685
7.421
7.311
7.291
7.260
5.519
5.395
2.418
2.126
SPh
O
O
H
Ts
1.76
4.89
1.92
0.86
1.89
3.00
2.86
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

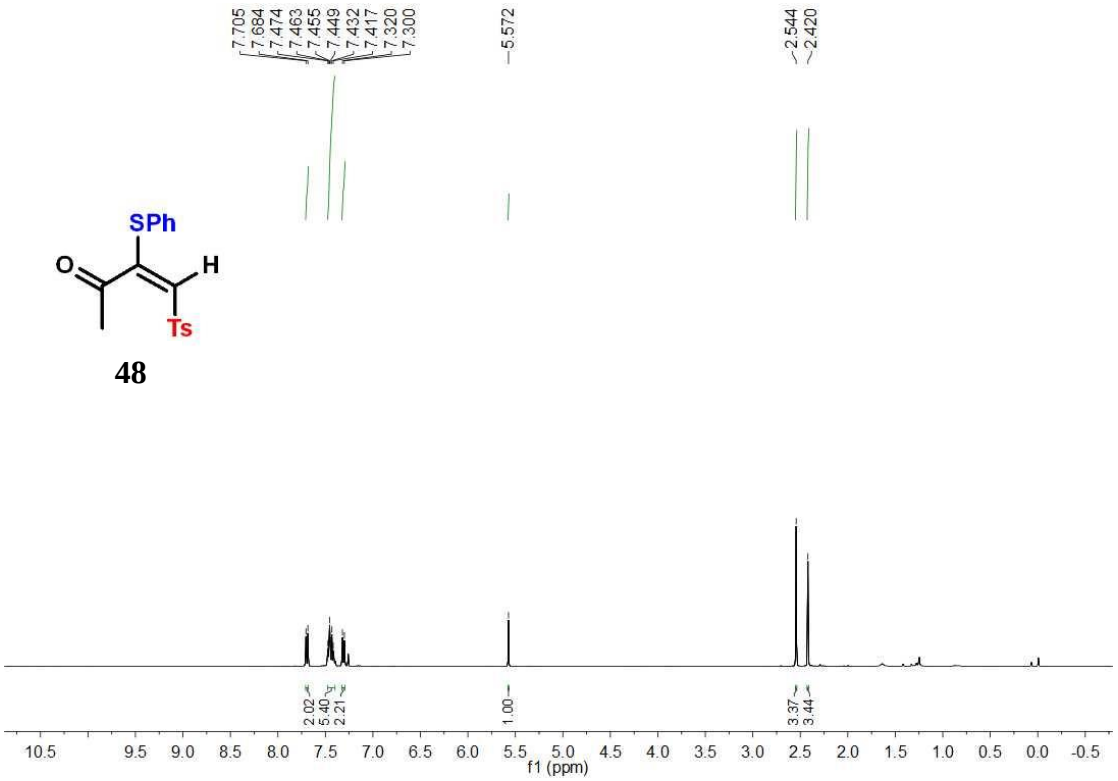
46


170.024
156.787
144.199
138.646
135.538
130.630
130.244
129.812
127.308
126.967
121.022
77.318
77.000
76.683
60.909
21.580
20.555
SPh
O
O
H
Ts
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

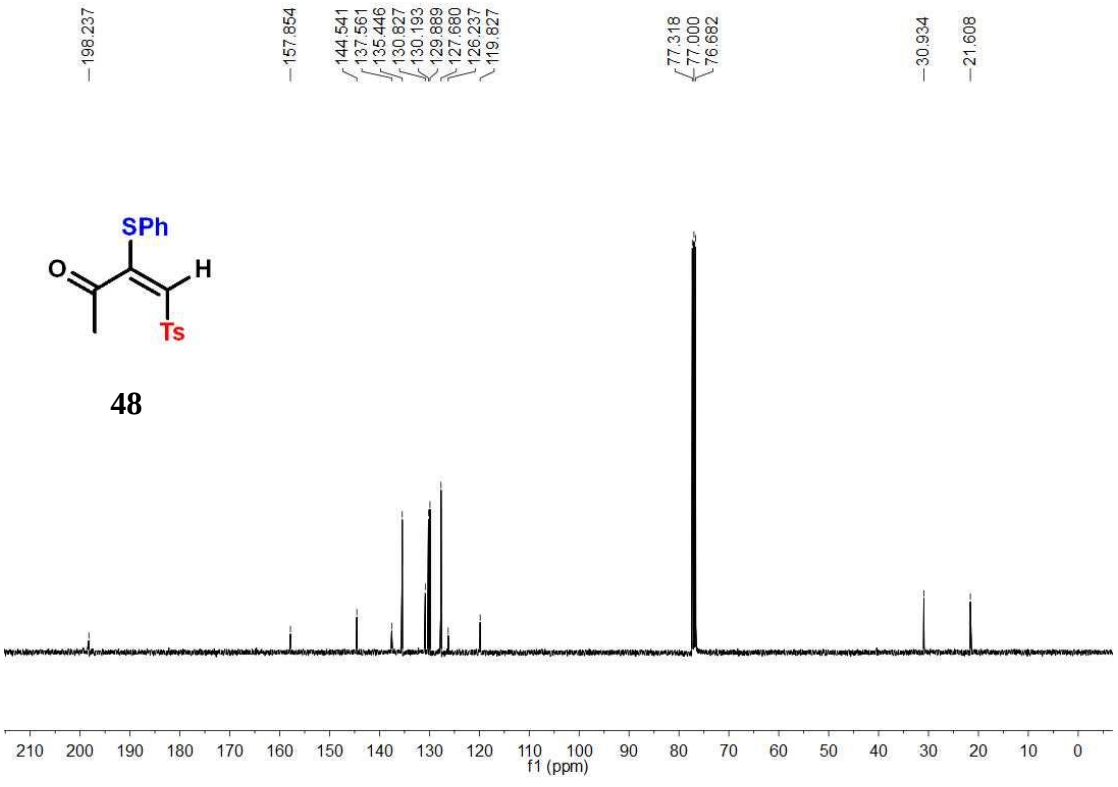
46


7.701
7.681
7.507
7.489
7.441
7.423
7.391
7.372
7.354
7.309
7.289
7.260
5.699
5.478
5.474
2.415
2.134
SePh
O
O
H
Ts
47
2.06
1.09
2.27
2.05
7.50 7.45 7.40 7.35 7.30 7.25 7.20
f1 (ppm)
2.01
2.06
1.09
2.27
2.05
1.00
2.03
3.03
3.04
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.804
155.876
144.265
138.341
136.853
130.260
130.217
129.840
127.014
124.790
123.750
77.318
77.000
76.682
62.260
21.566
20.492
SePh
O
O
H
Ts
47
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.705
7.684
7.474
7.463
7.455
7.449
7.432
7.417
7.320
7.300
5.572
2.544
2.420
SPh
O
H
Ts
2.02
5.40
2.21
1.00
3.37
3.44
f1 (ppm)

48


198.237
157.854
144.541
137.561
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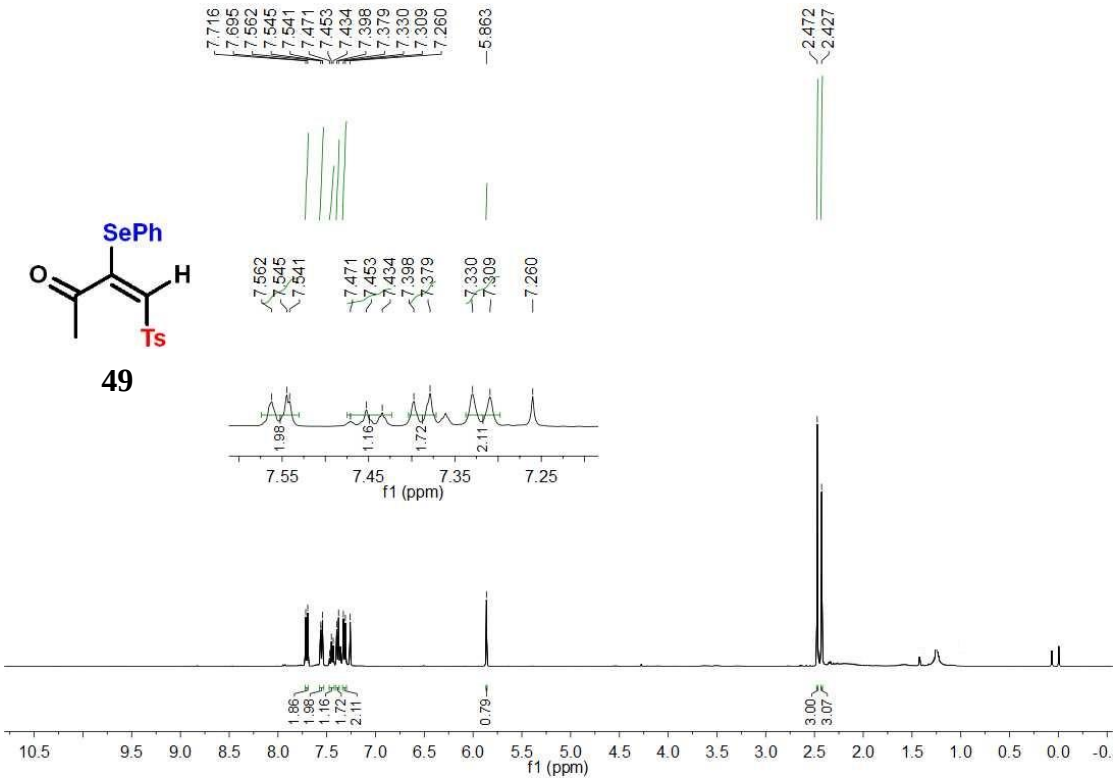
130.827
130.193
129.889
127.680
126.237
119.827
77.318
77.000
76.682
30.934
21.608
SPh
O
H
Ts
f1 (ppm)

48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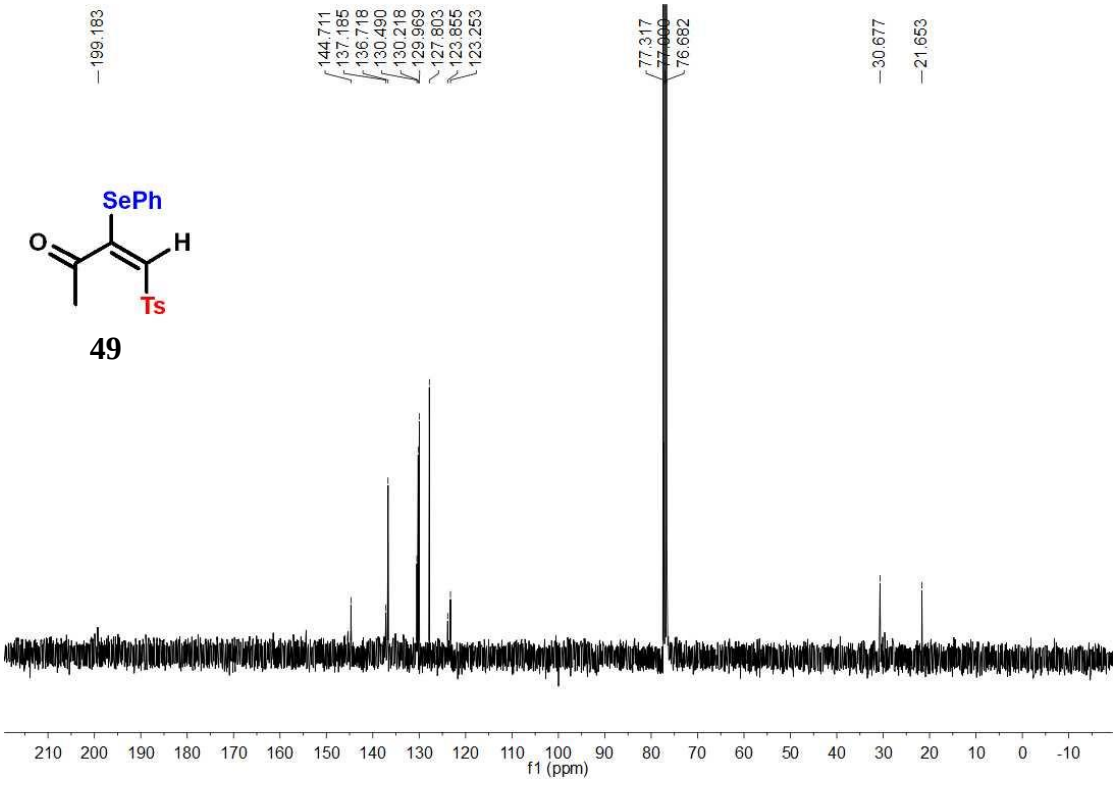
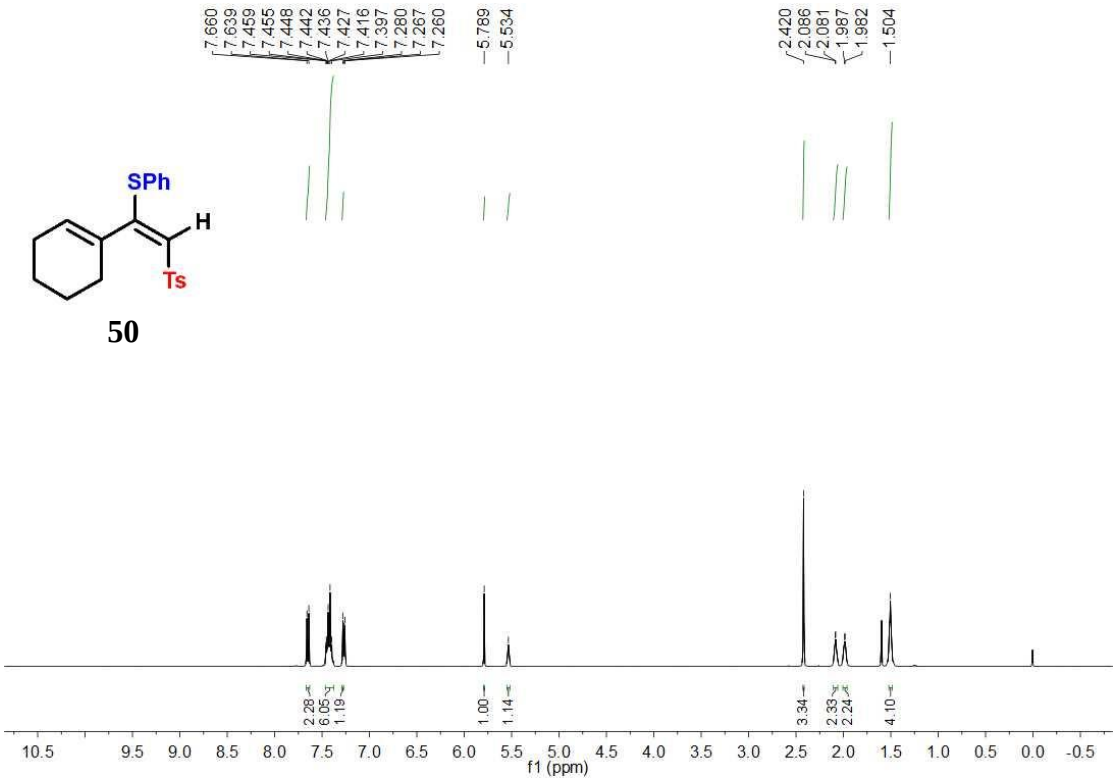
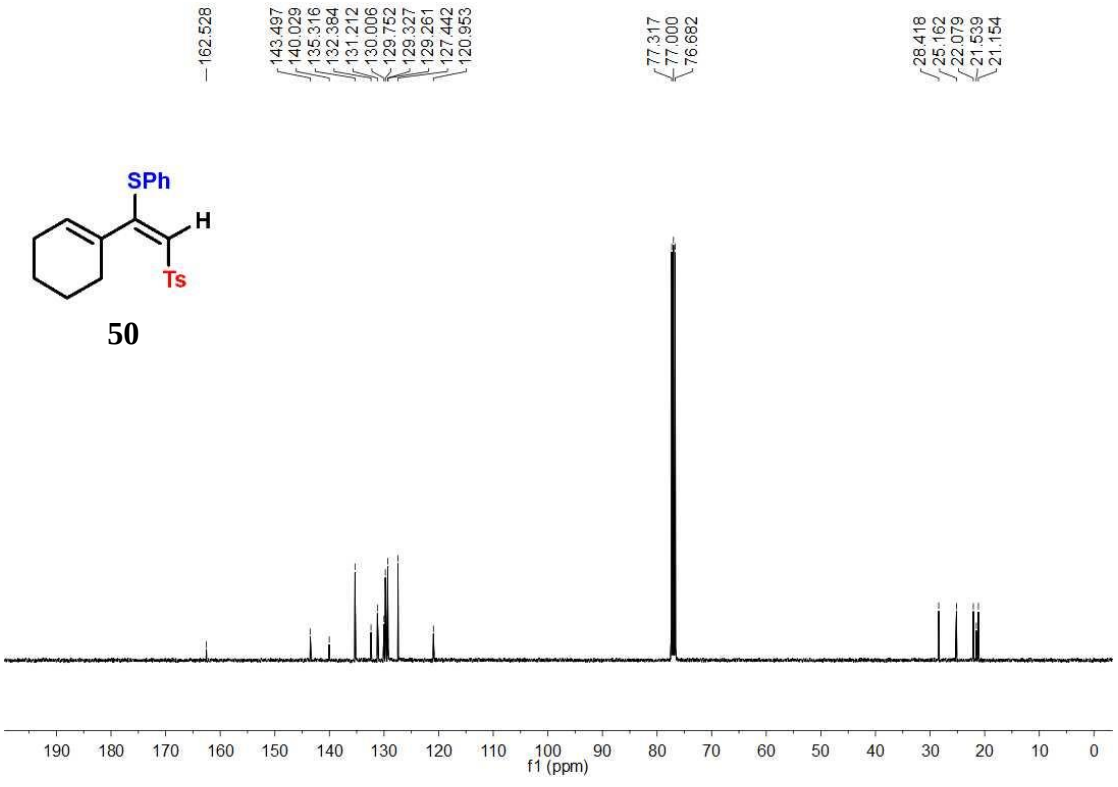
7.695
7.562
7.545
7.541
7.471
7.453
7.434
7.398
7.379
7.330
7.309
7.260
5.863
2.472
2.427
SePh
O
H
Ts
49
1.98
1.16
1.72
2.11
7.55
7.45
7.35
7.25
f1 (ppm)
1.86
1.98
1.16
1.72
2.11
0.79
3.00
3.07
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

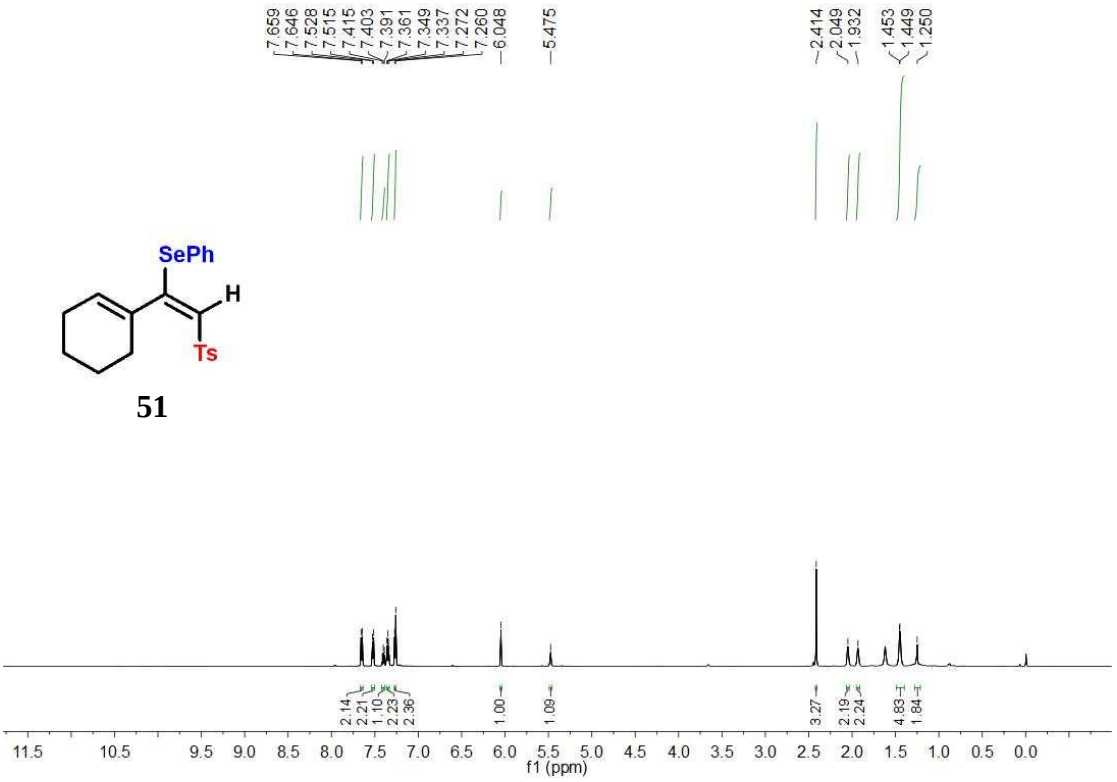

199.183
144.711
137.185
136.718
130.490
130.218
129.969
127.803
123.855
123.253
77.317
76.682
30.677
21.653
SePh
O
H
Ts
49
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.660
7.639
7.459
7.455
7.448
7.442
7.436
7.427
7.416
7.397
7.280
7.267
7.260
5.789
5.534
2.420
2.086
2.081
1.987
1.982
1.504
SPh
H
Ts
2.28
6.05
1.19
1.00
1.14
3.34
2.33
2.24
4.10
f1 (ppm)

50


162.528
143.497
140.029
135.316
132.384
131.212
130.006
129.752
129.327
129.261
127.442
120.953
77.317
77.000
76.682
28.418
25.162
22.079
21.539
21.154
SPh
H
Ts
f1 (ppm)

50


7.659
7.646
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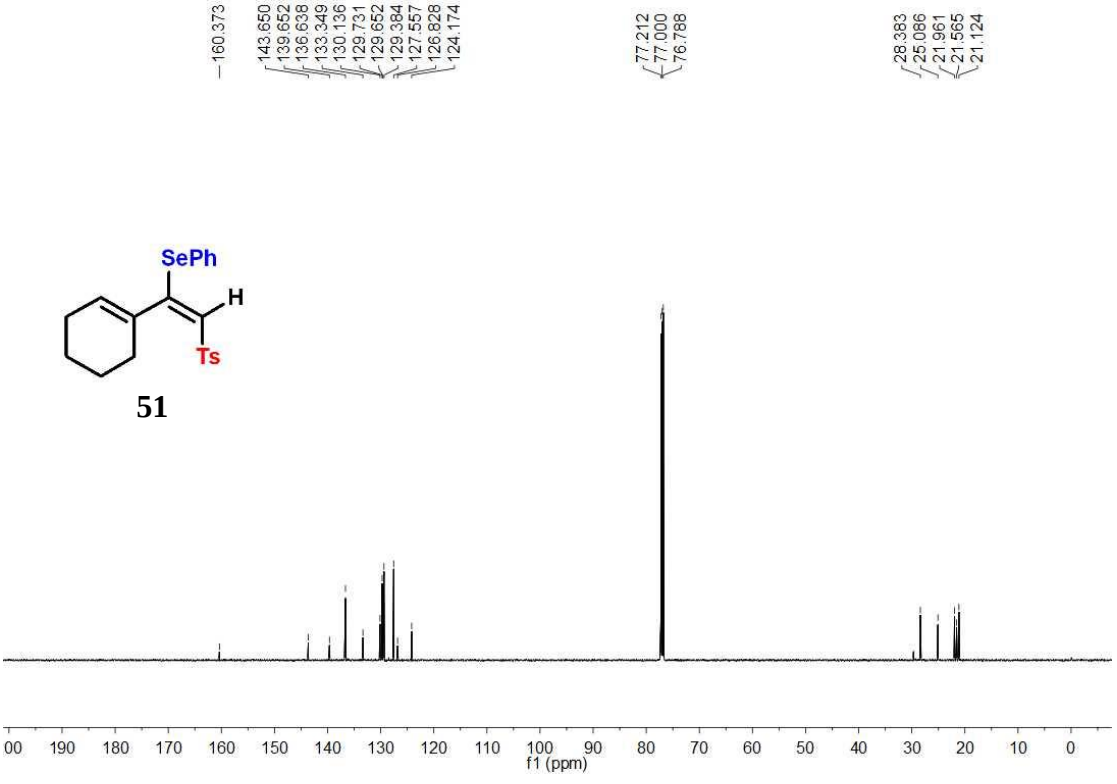
7.515
7.415
7.403
7.391
7.361
7.349
7.337
7.272
7.260
6.048
5.475
2.414
2.049
1.932
1.453
1.449
1.250
SePh
H
Ts
51
2.14
2.21
1.10
2.23
2.36
1.00
1.09
3.27
2.19
2.24
4.83
1.84
f1 (ppm)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127.557
126.828
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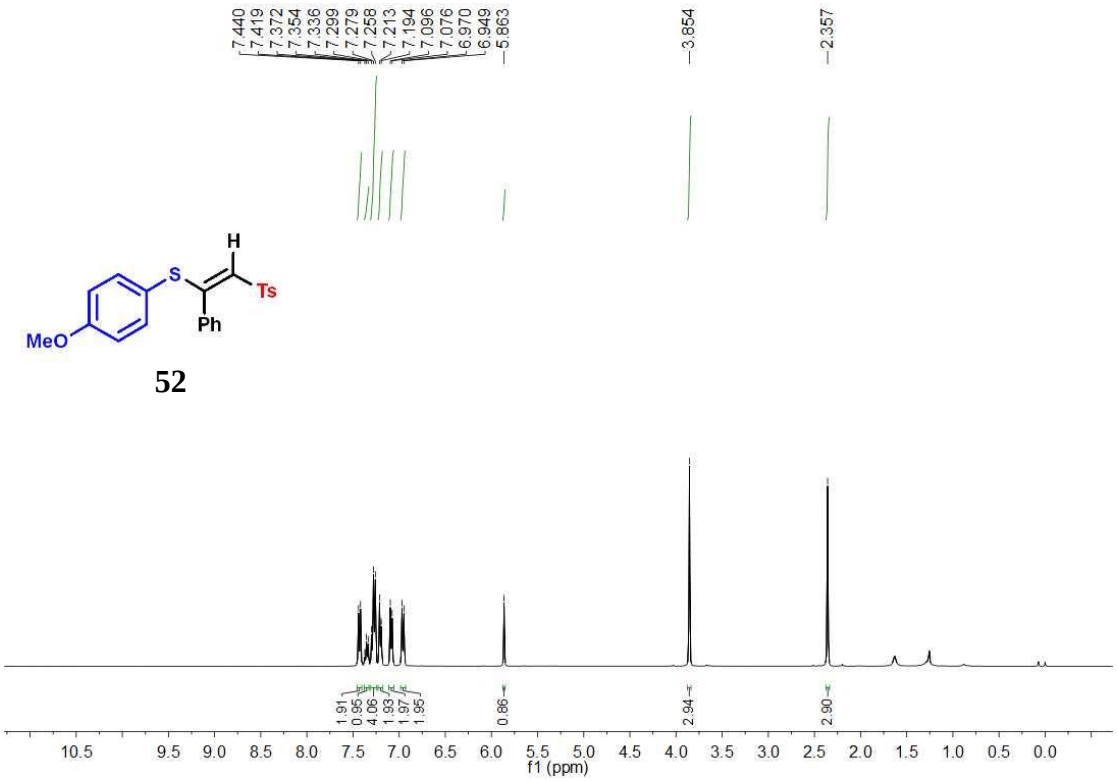
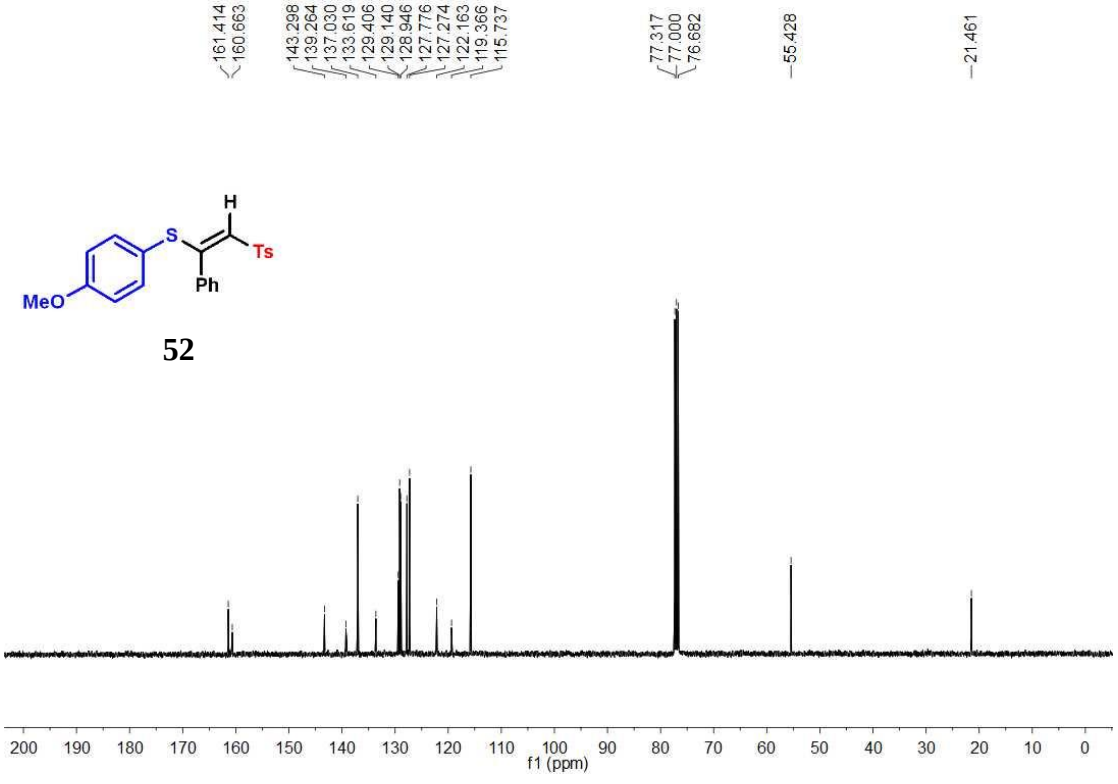
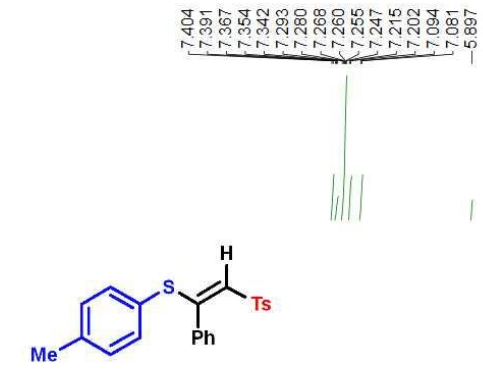
77.212
77.000
76.788
28.383
25.086
21.961
21.565
21.124
SePh
H
Ts
51
f1 (ppm)


7.440
7.419
7.372
7.354
7.336
7.299
7.279
7.258
7.213
7.194
7.096
7.076
6.970
6.949
5.863
3.854
2.357
H
S
Ts
Ph
MeO
1.91
0.95
4.06
1.93
1.97
1.95
0.86
2.94
2.90
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

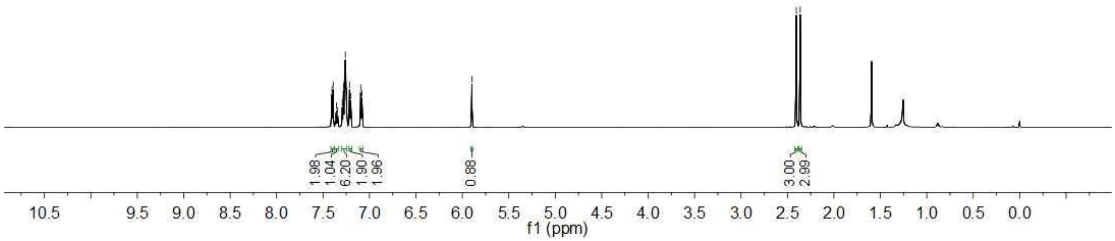
52

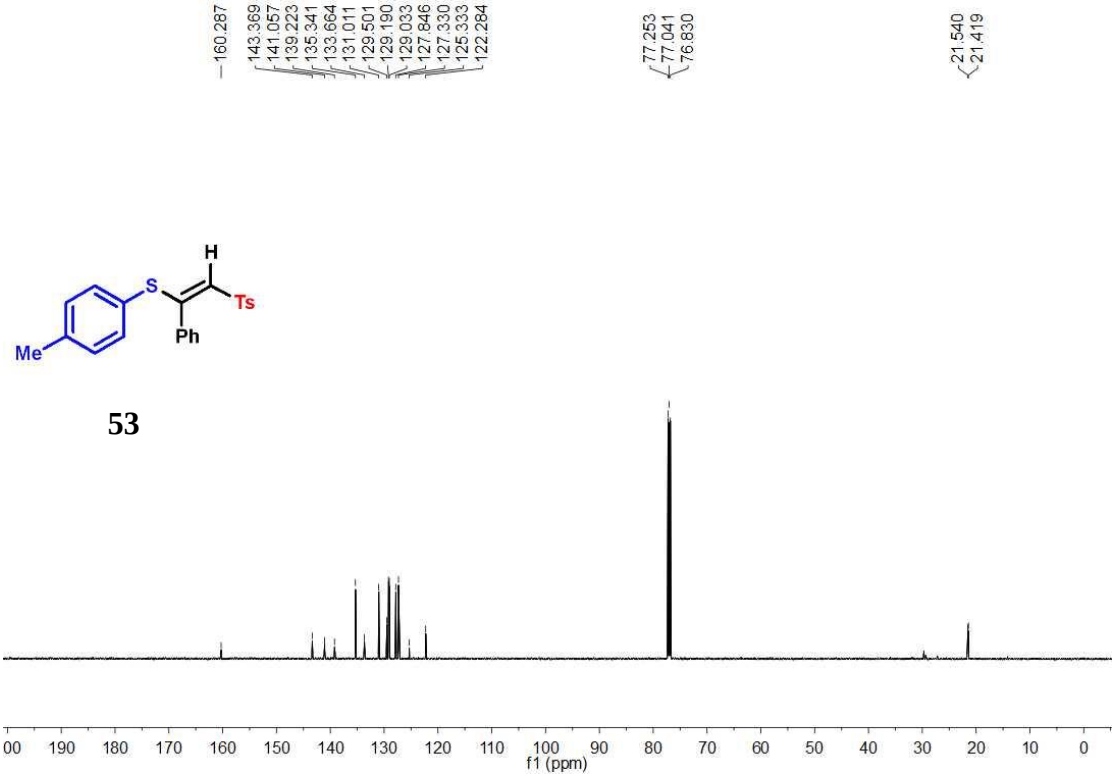

161.414
160.663
143.298
139.264
137.030
133.619
129.406
129.140
128.946
127.776
127.274
122.163
119.366
115.737
77.317
77.000
76.682
55.428
21.461
H
S
Ts
Ph
MeO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

52

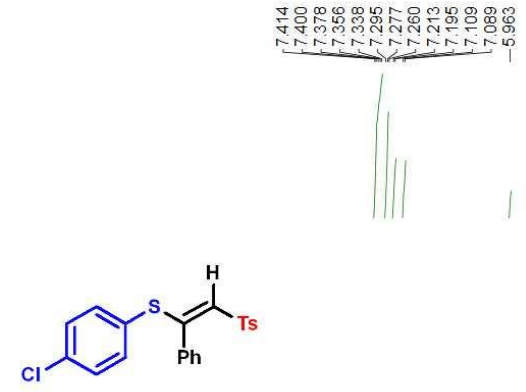

7.404
7.391
7.367
7.354
7.342
7.293
7.280
7.268
7.260
7.255
7.247
7.215
7.202
7.094
7.081
5.897
2.405
2.362
H
S
Ts
Ph
Me

53

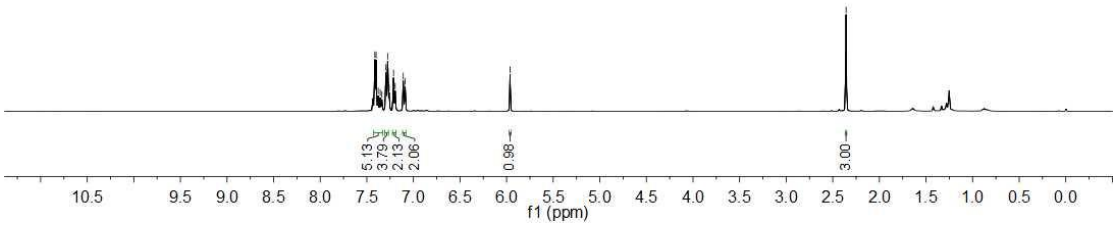

1.98
1.04
6.20
1.90
1.96
0.88
3.00
2.99
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


160.287
143.369
141.057
139.223
135.341
133.664
131.011
129.501
129.190
129.033
127.846
127.330
125.333
122.284
77.253
77.041
76.830
21.540
21.419
H
S
Ts
Ph
Me
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

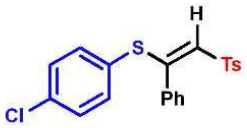
53


7.414
7.400
7.378
7.356
7.338
7.295
7.277
7.260
7.213
7.195
7.109
7.089
5.963
2.357
H
S
Ts
Ph
Cl

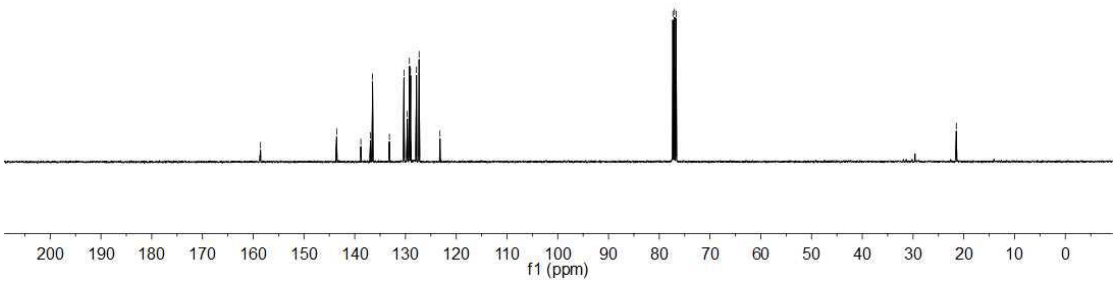
54

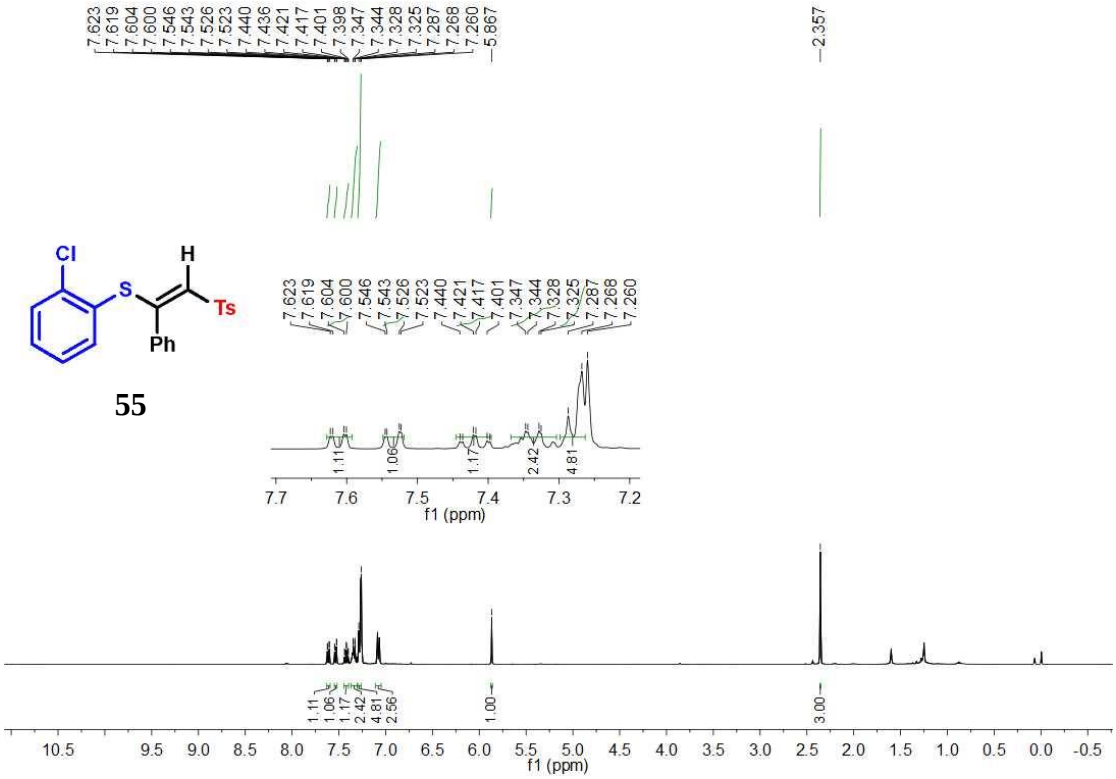

5.13
3.79
2.13
2.06
0.98
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

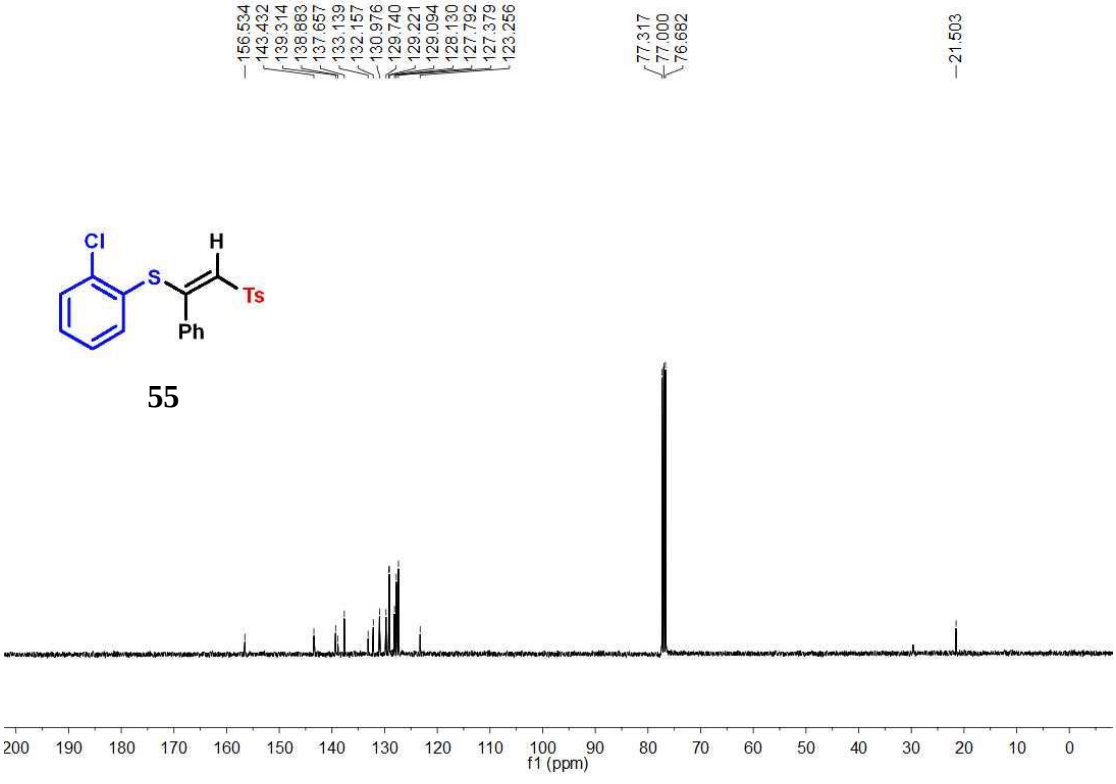

158.561
143.563
138.792
136.905
136.483
133.166
130.305
129.642
129.222
129.011
127.837
127.309
123.211
77.317
77.000
76.682
21.475

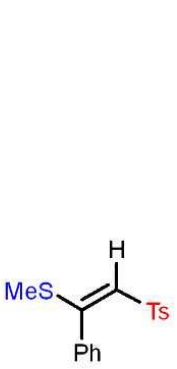

H
S
Ts
Ph
Cl

54


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

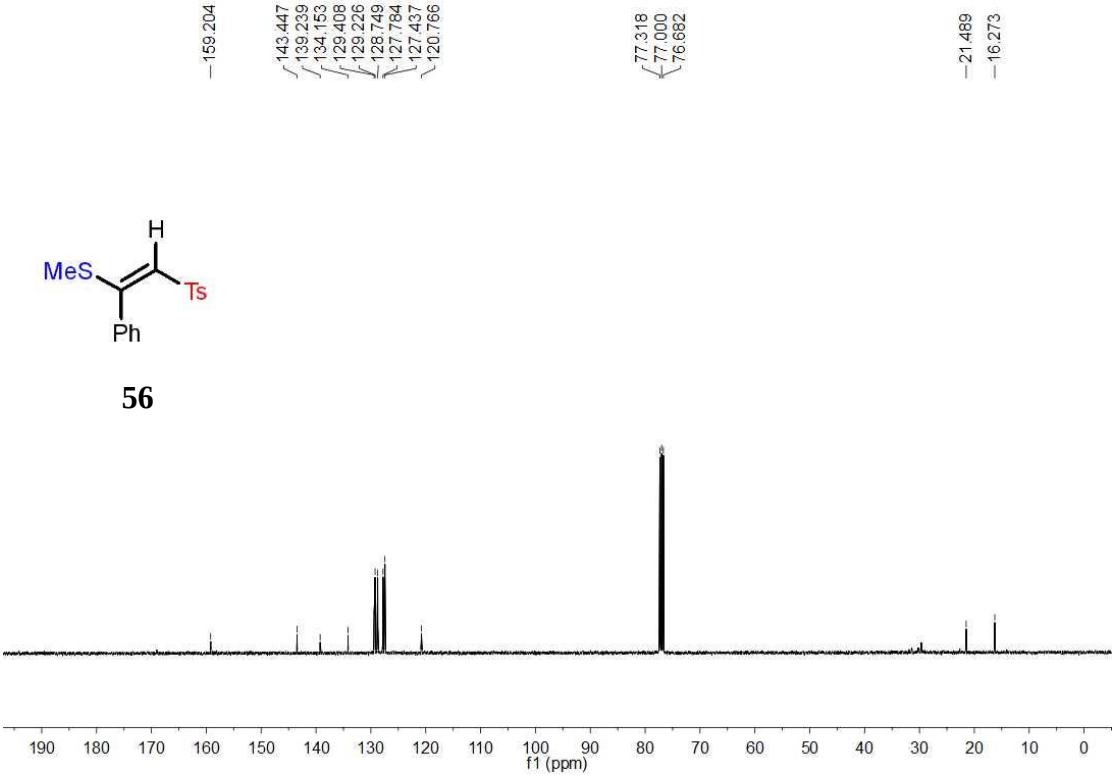

7.623
7.619
7.604
7.600
7.546
7.543
7.526
7.523
7.440
7.436
7.421
7.417
7.401
7.398
7.347
7.344
7.328
7.325
7.287
7.268
7.260
5.867
2.357
Cl
H
S
Ts
Ph
55
1.11
1.06
1.17
2.42
4.81
2.56
1.00
3.00
f1 (ppm)


156.534
143.432
139.314
138.883
137.657
133.139
132.157
130.976
129.740
129.221
129.094
128.130
127.792
127.379
123.256
77.317
77.000
76.682
21.503
Cl
H
S
Ts
Ph
55
f1 (ppm)


H
MeS
Ts
Ph

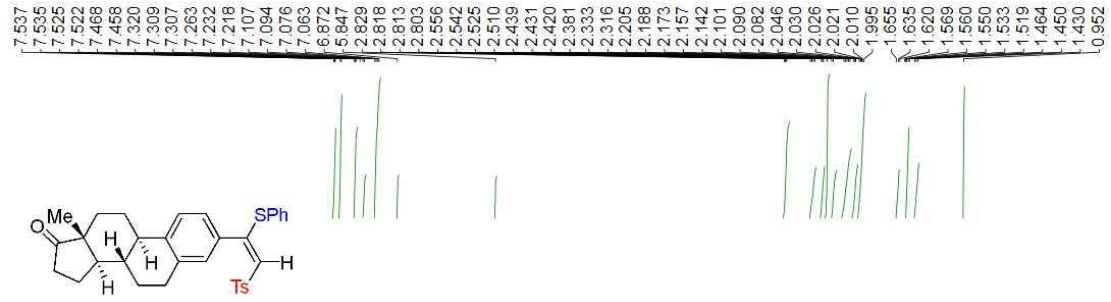
56

7.381
7.360
7.339
7.294
7.275
7.260
7.146
7.132
7.114
6.201
2.372
2.316
2.83
2.21
3.73
0.93
2.93
2.92
f1 (ppm)

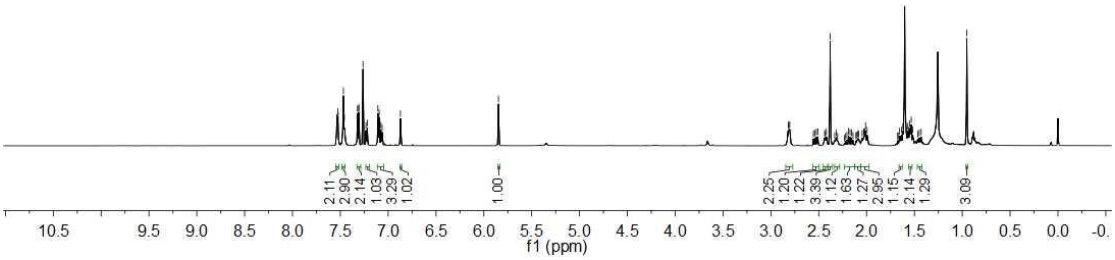

159.204
143.447
139.239
134.153
129.408
129.226
128.749
127.784
127.437
120.766
77.318
77.000
76.682
21.489
16.273
f1 (ppm)

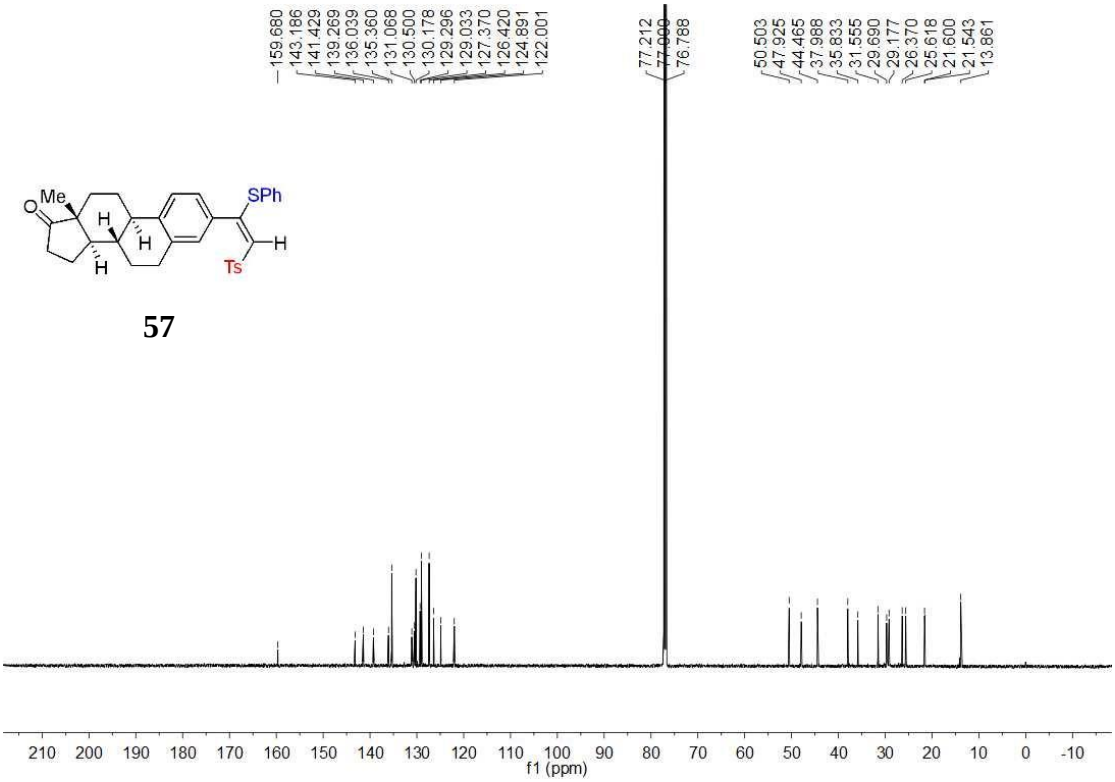
H
MeS
Ts
Ph

56

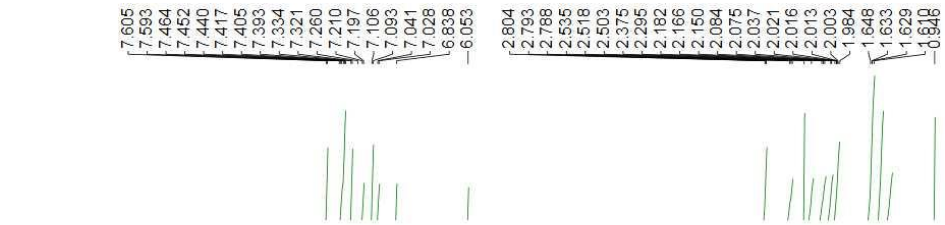


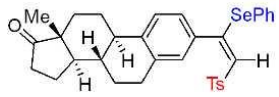
57



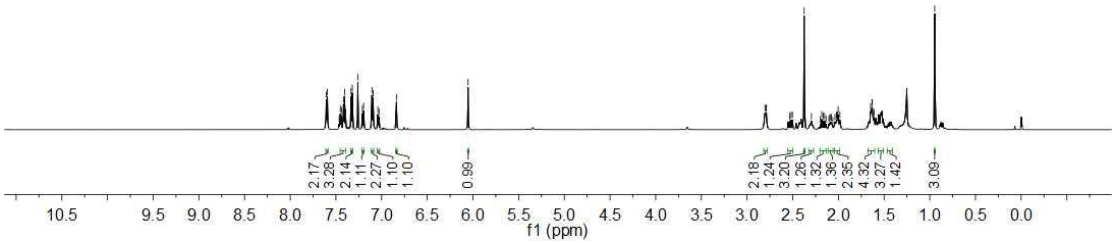


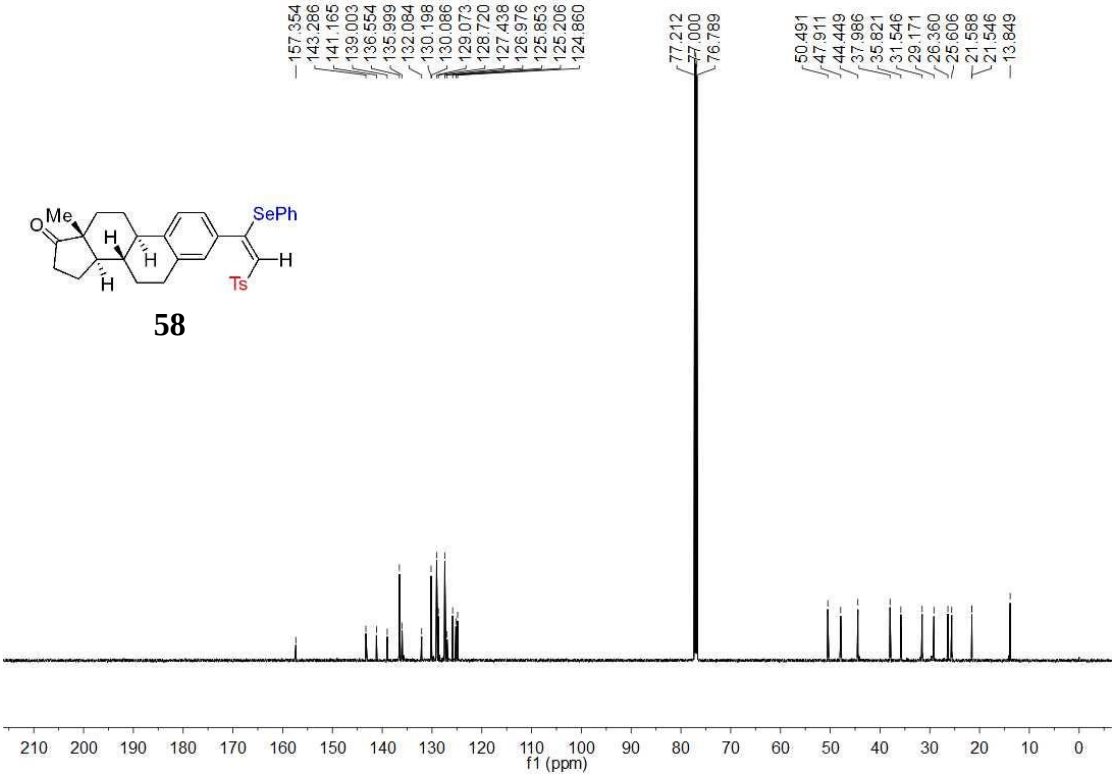
57


7.605
7.593
7.464
7.452
7.440
7.417
7.405
7.393
7.334
7.321
7.260
7.210
7.197
7.106
7.093
7.041
7.028
6.838
6.053
2.804
2.793
2.788
2.535
2.518
2.503
2.375
2.295
2.182
2.166
2.150
2.084
2.075
2.037
2.021
2.016
2.013
2.003
1.984
1.648
1.633
1.629
1.610
0.948


Me
O
H
H
H
SePh
H
Ts

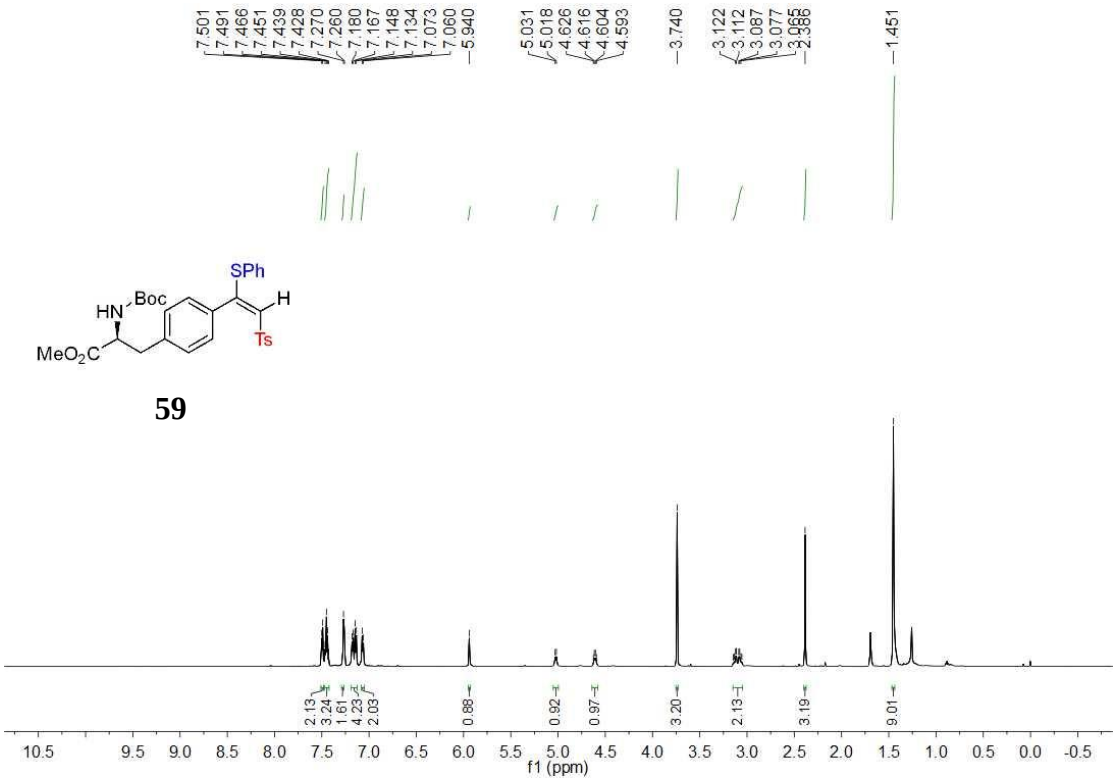
58


2.17
3.28
2.14
1.11
2.27
1.10
1.10
0.99
2.18
1.24
3.20
1.26
1.32
1.36
2.35
4.32
3.27
1.42
3.09
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

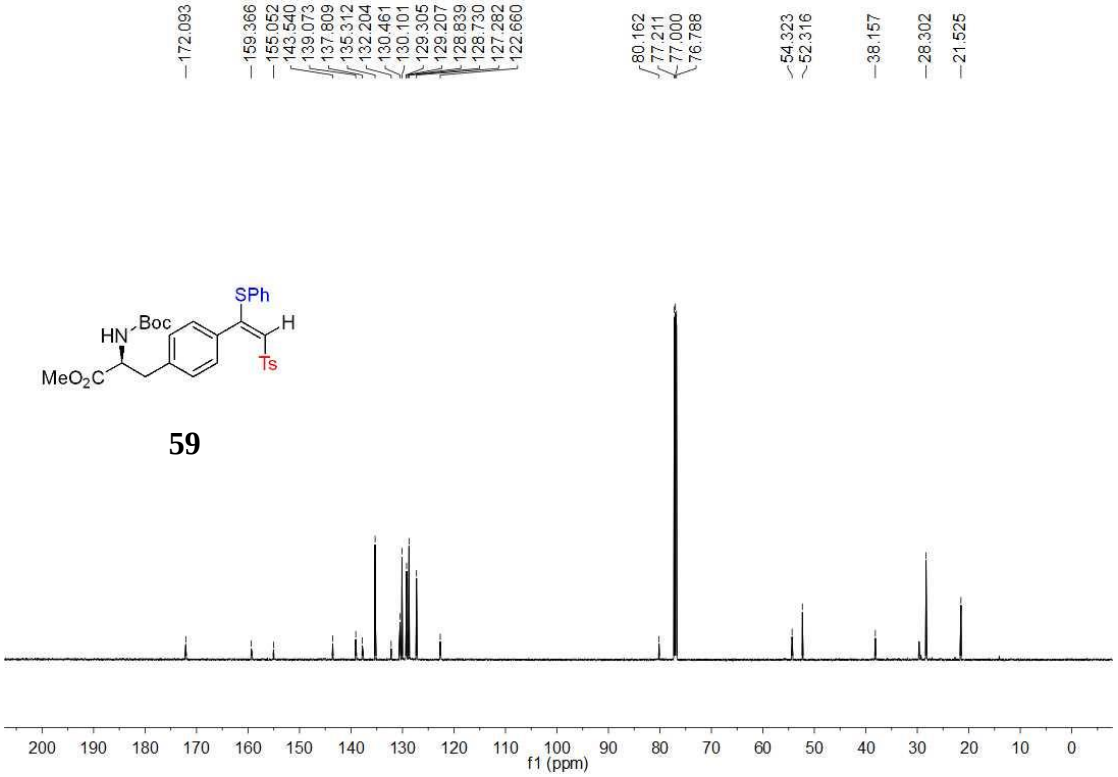

157.354
143.286
141.165
139.003
136.554
135.999
132.084
130.198
130.086
129.073
128.720
127.438
126.976
125.853
125.206
124.860
77.212
77.000
76.789
50.491
47.911
44.449
37.986
35.821
31.546
29.171
26.360
25.606
21.588
21.546
13.849
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Me
O
H
H
H
SePh
H
Ts

58


7.501
7.491
7.466
7.451
7.439
7.428
7.270
7.260
7.180
7.167
7.148
7.134
7.073
7.060
5.940
5.031
5.018
4.626
4.616
4.604
4.593
3.740
3.122
3.112
3.087
3.077
3.065
2.386
1.451
SPh
Boc
HN
H
Ts
MeO2C
2.13
3.24
1.61
4.23
2.03
0.88
0.92
0.97
3.20
2.13
3.19
9.01
f1 (ppm)

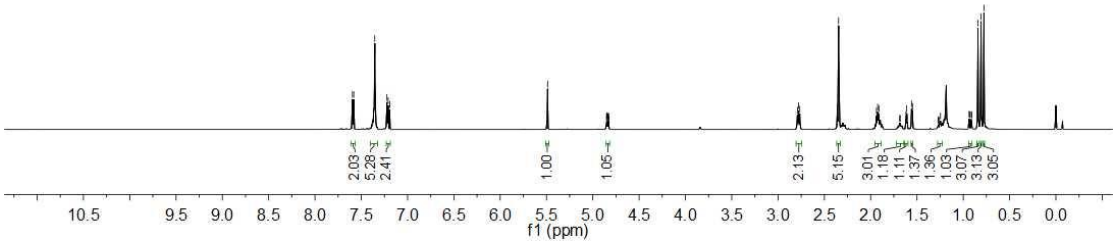
59

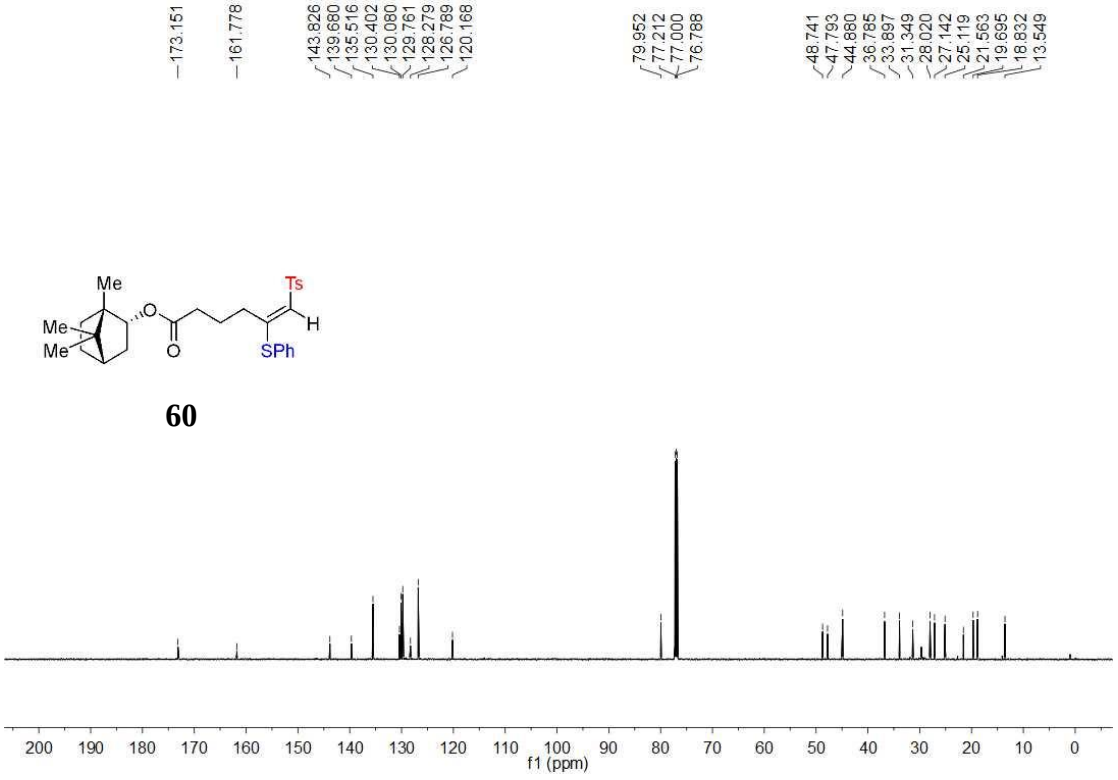

172.093
159.366
155.052
143.540
139.073
137.809
135.312
132.204
130.461
130.101
129.305
129.207
128.839
128.730
127.282
122.660
80.162
77.211
77.000
76.788
54.323
52.316
38.157
28.302
21.525
SPh
Boc
HN
H
Ts
MeO2C
f1 (ppm)

59


7.595
7.581
7.354
7.223
7.210
7.193
5.488
4.852
4.849
4.847
4.843
4.835
4.832
4.830
2.792
2.778
2.765
2.361
2.347
2.336
1.937
1.924
1.911
1.619
1.612
1.559
1.549
1.246
0.937
0.931
0.914
0.909
0.841
0.808
Me
Ts
Me
O
H
Me
O
SPh

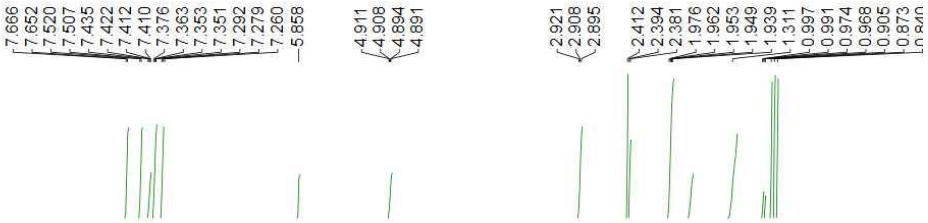
60

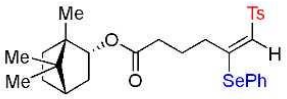

2.03
5.28
2.41
1.00
1.05
2.13
5.15
3.01
1.18
1.11
1.37
1.36
1.03
3.07
3.13
3.05
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


173.151
161.778
143.826
139.680
135.516
130.402
130.080
129.761
128.279
126.789
120.168
79.952
77.212
77.000
76.788
48.741
47.793
44.880
36.785
33.897
31.349
28.020
27.142
25.119
21.563
19.695
18.832
13.549
Me
Ts
Me
O
H
Me
O
SPh

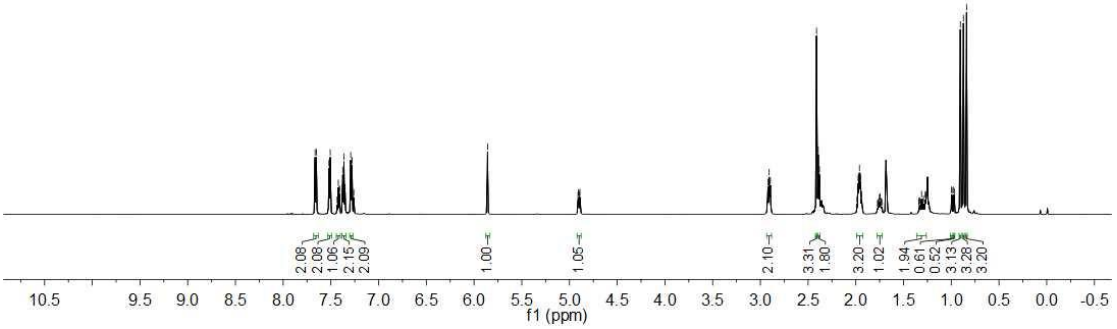
60

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

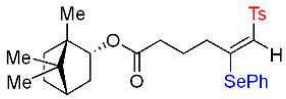

7.666
7.652
7.520
7.507
7.435
7.422
7.412
7.410
7.376
7.363
7.353
7.351
7.292
7.279
7.260
5.858
4.911
4.908
4.894
4.891
2.921
2.908
2.895
2.412
2.394
2.381
1.976
1.962
1.953
1.949
1.939
1.311
0.997
0.991
0.974
0.968
0.905
0.873


Me
Ts
Me
O
H
Me
O
SePh

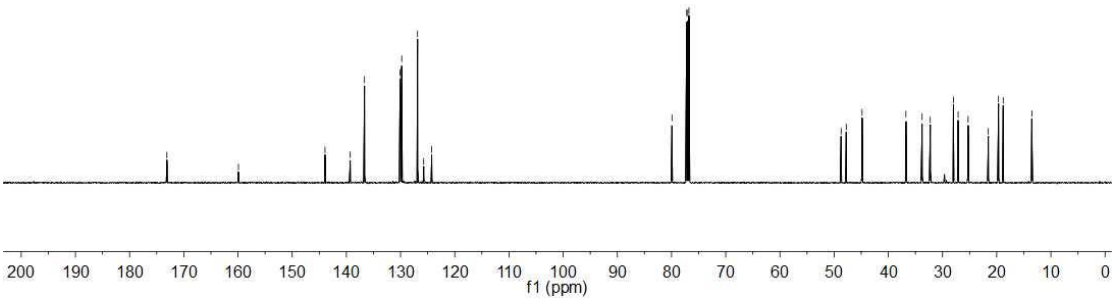
61

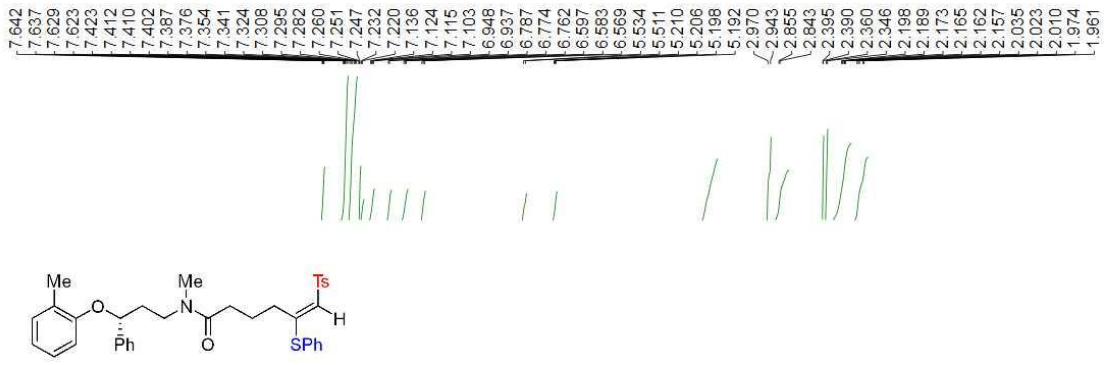

2.08
2.08
1.06
2.15
2.09
1.00
1.05
2.10
3.31
1.80
3.20
1.02
1.94
0.61
0.52
3.13
3.28
3.20
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


173.124
159.910
143.944
139.295
136.658
130.083
129.946
129.782
126.871
125.735
124.257
79.921
77.213
77.000
76.789
48.708
47.761
44.842
36.752
33.769
32.261
27.993
27.113
25.279
21.551
19.665
18.799
13.527


Me
Ts
Me
O
H
Me
O
SePh

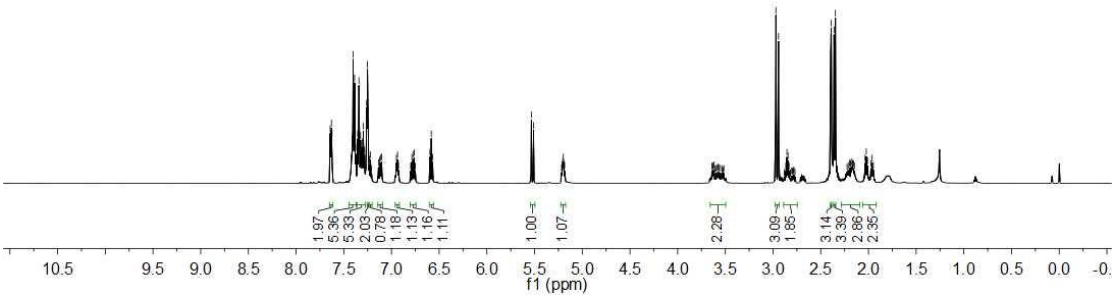
61

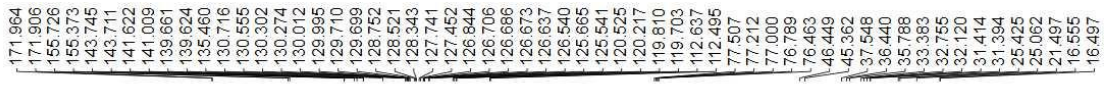

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.642
7.637
7.629
7.623
7.423
7.412
7.410
7.402
7.387
7.376
7.354
7.341
7.324
7.308
7.295
7.282
7.260
7.251
7.247
7.232
7.220
7.136
7.124
7.115
7.103
6.948
6.937
6.787
6.774
6.762
6.597
6.583
6.569
6.534
5.511
5.210
5.206
5.198
5.192
2.970
2.943
2.855
2.843
2.395
2.390
2.360
2.346
2.198
2.189
2.173
2.165
2.162
2.157
2.035
2.023
2.010
1.974
1.961

Me
Me
Ts
O
N
H
Ph
O
SPh

62


1.97
5.36
5.33
2.03
0.78
1.18
1.13
1.16
1.11
1.00
1.07
2.28
3.09
1.85
3.14
3.39
2.86
2.35
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.964
171.906
155.726
155.373
143.745
143.711
141.622
141.009
139.661
139.624
135.460
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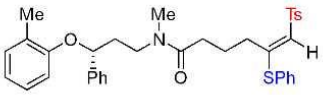
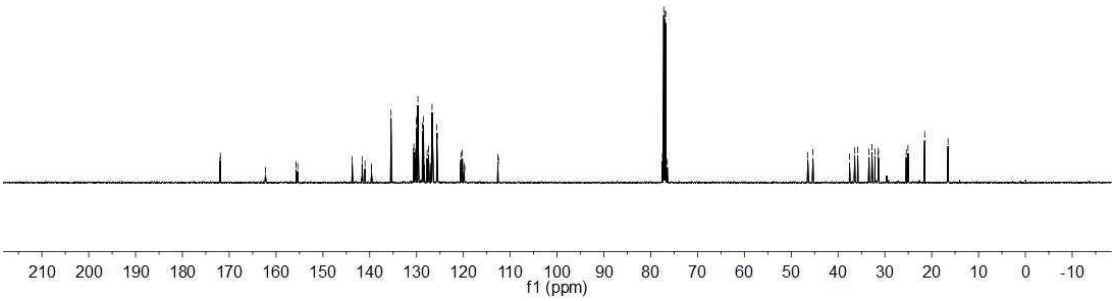
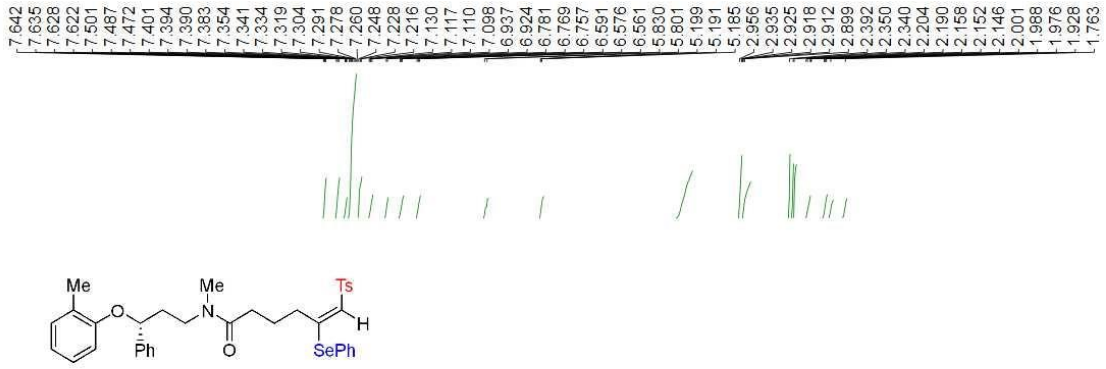
130.555
130.302
130.274
130.012
129.995
129.710
129.699
128.752
128.521
128.343
127.741
127.452
126.844
126.706
126.686
126.673
126.637
126.540
125.665
125.541
120.525
120.217
119.810
119.703
112.637
112.495
77.507
77.212
77.000
76.789
76.463
46.449
45.362
37.548
36.440
35.788
33.383
32.755
32.120
31.414
31.394
25.425
25.062
21.497
16.555
16.497

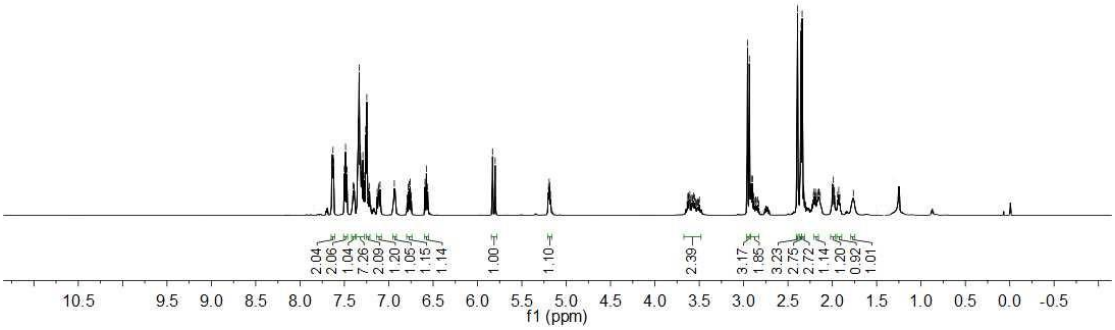

Me
Me
Ts
O
N
H
Ph
O
SPh

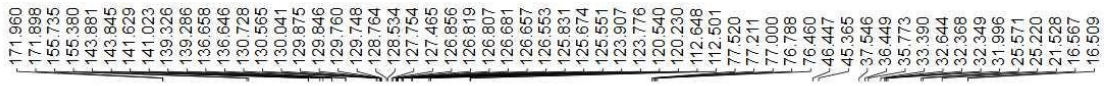
62

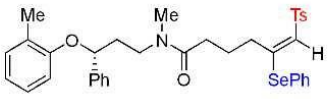

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



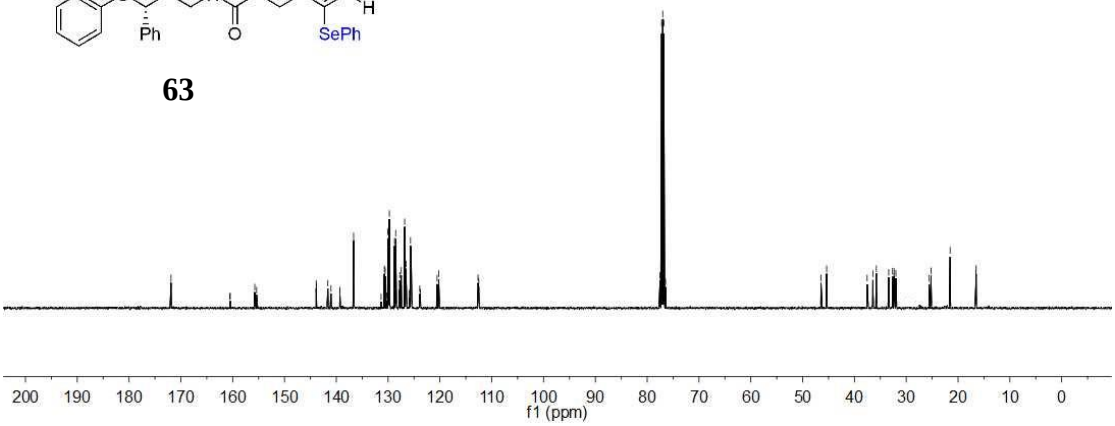
63

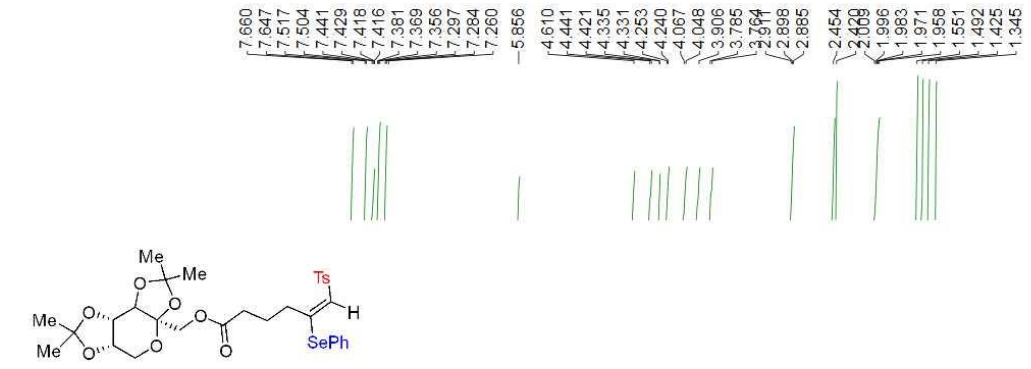




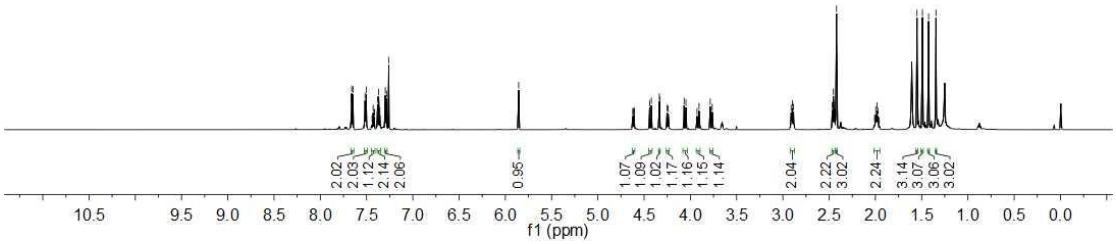


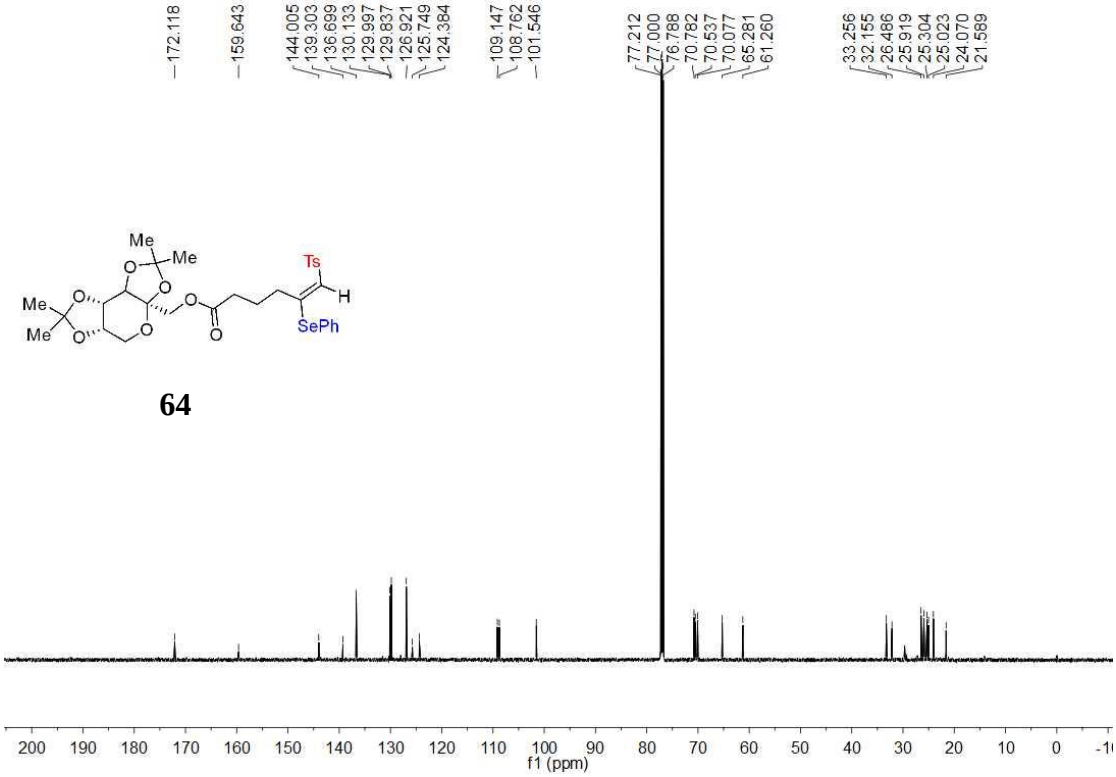
63



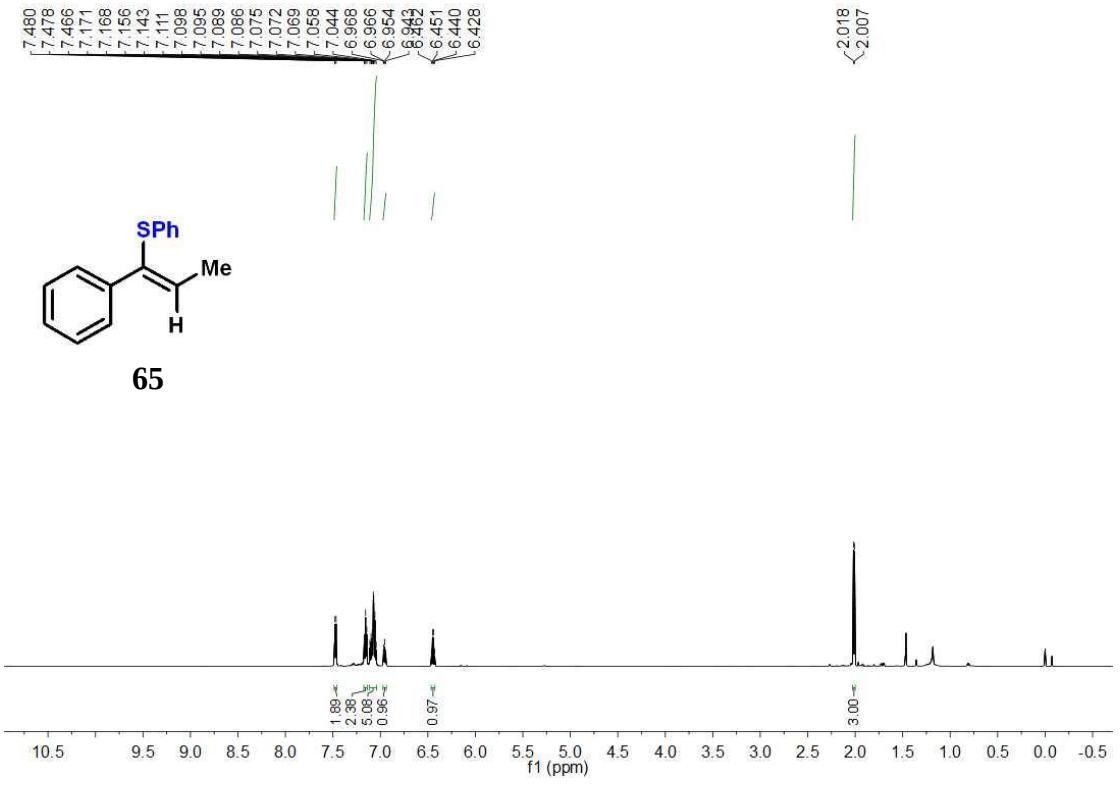

7.660
7.647
7.517
7.504
7.441
7.429
7.418
7.416
7.381
7.369
7.356
7.297
7.284
7.260
5.856
4.610
4.441
4.421
4.335
4.331
4.253
4.240
4.067
4.048
3.906
3.785
3.764
2.911
2.898
2.885
2.454
2.420
2.069
1.996
1.983
1.971
1.958
1.551
1.492
1.425
1.345
Me
Me
Me
Me
O
O
O
O
O
O
Ts
H
SePh

64

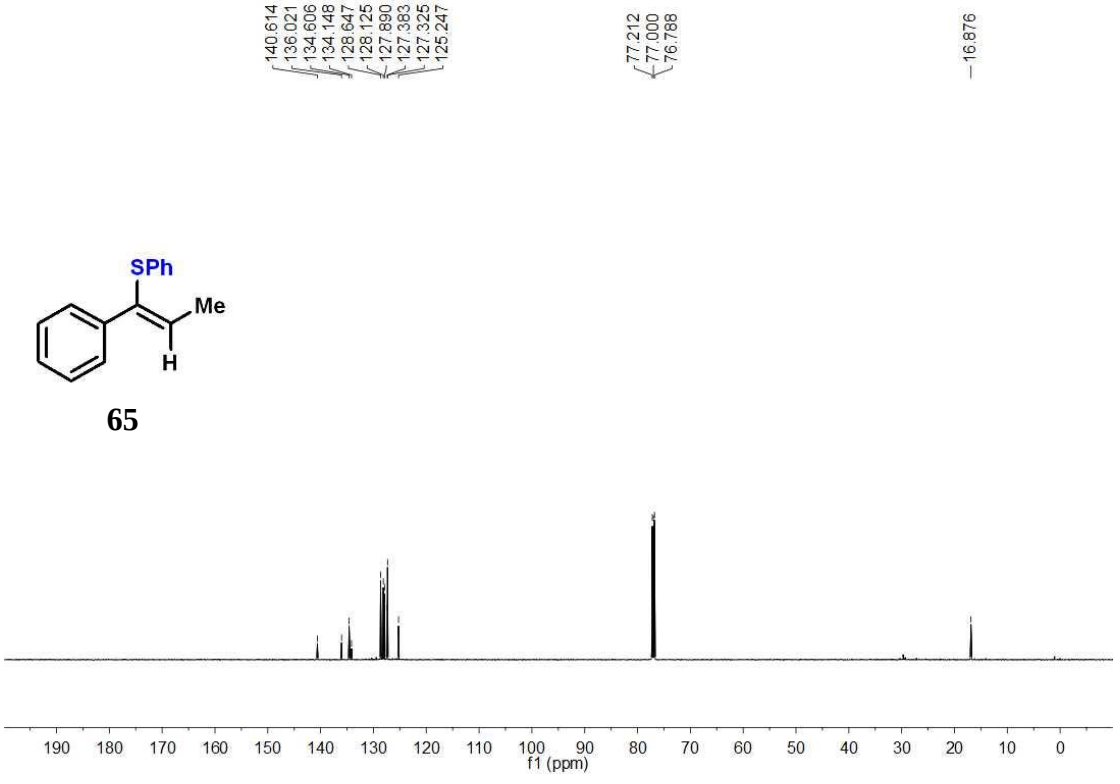

2.02
2.03
1.12
2.14
2.06
0.95
1.07
1.09
1.02
1.17
1.16
1.15
1.14
2.04
2.22
3.02
2.24
3.14
3.07
3.06
3.02
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


172.118
159.643
144.005
139.303
136.699
130.133
129.997
129.837
126.921
125.749
124.384
109.147
108.762
101.546
77.212
77.000
76.788
70.782
70.537
70.077
65.281
61.260
33.256
32.155
26.486
25.919
25.304
25.023
24.070
21.589
Me
Me
Me
Me
O
O
O
O
O
O
Ts
H
SePh
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

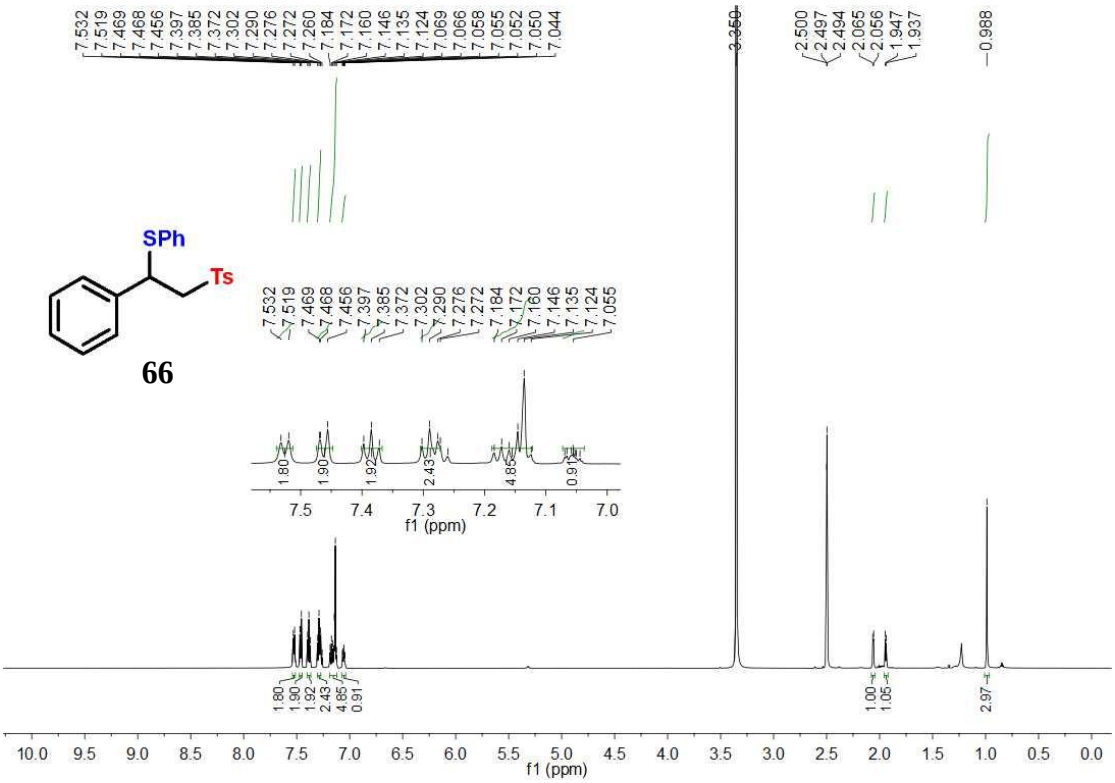
64

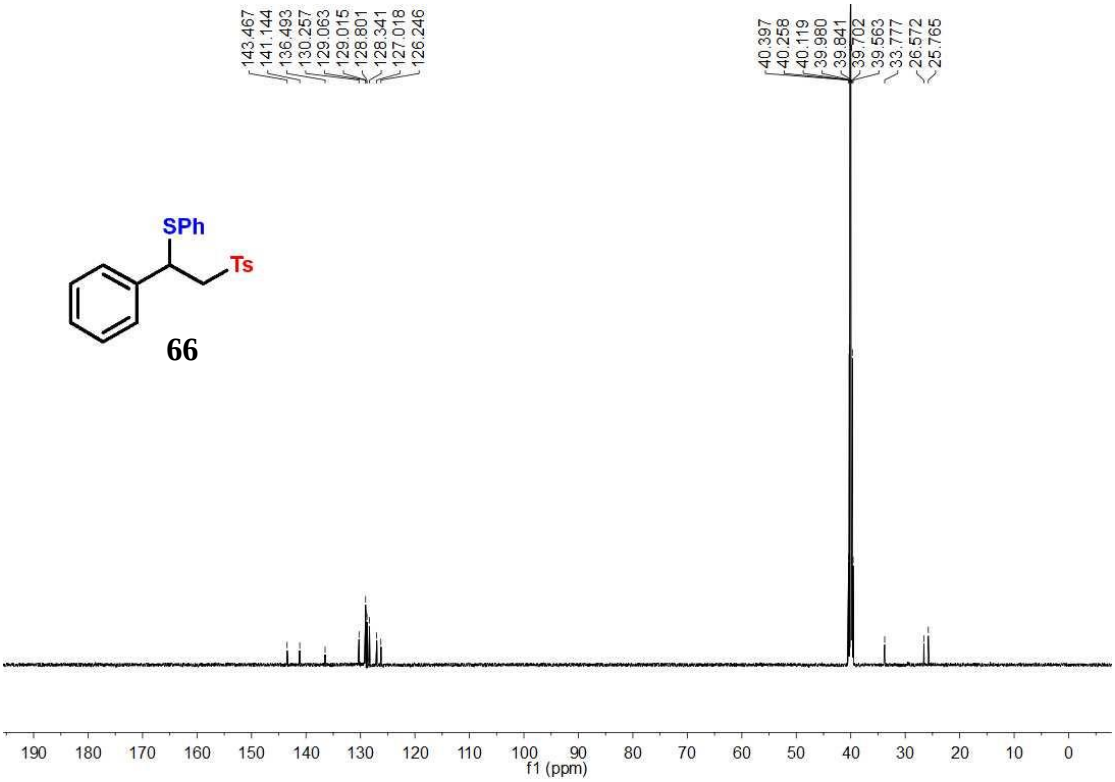

7.480
7.478
7.466
7.171
7.168
7.156
7.143
7.111
7.098
7.095
7.089
7.086
7.075
7.072
7.069
7.058
7.044
6.968
6.966
6.954
6.451
6.440
6.428
2.018
2.007
SPh
Me
H
1.89
2.38
5.08
0.96
0.97
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

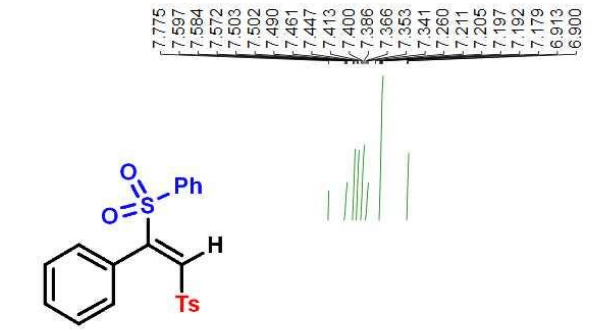
65


140.614
136.021
134.606
134.148
128.647
128.125
127.890
127.383
127.325
125.247
77.212
77.000
76.788
16.876
SPh
Me
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

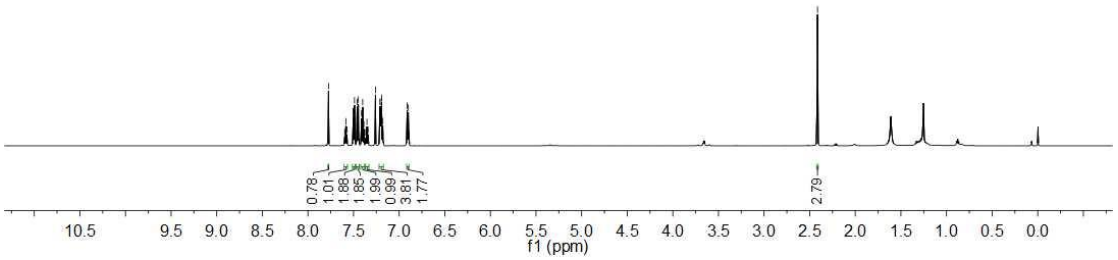
65

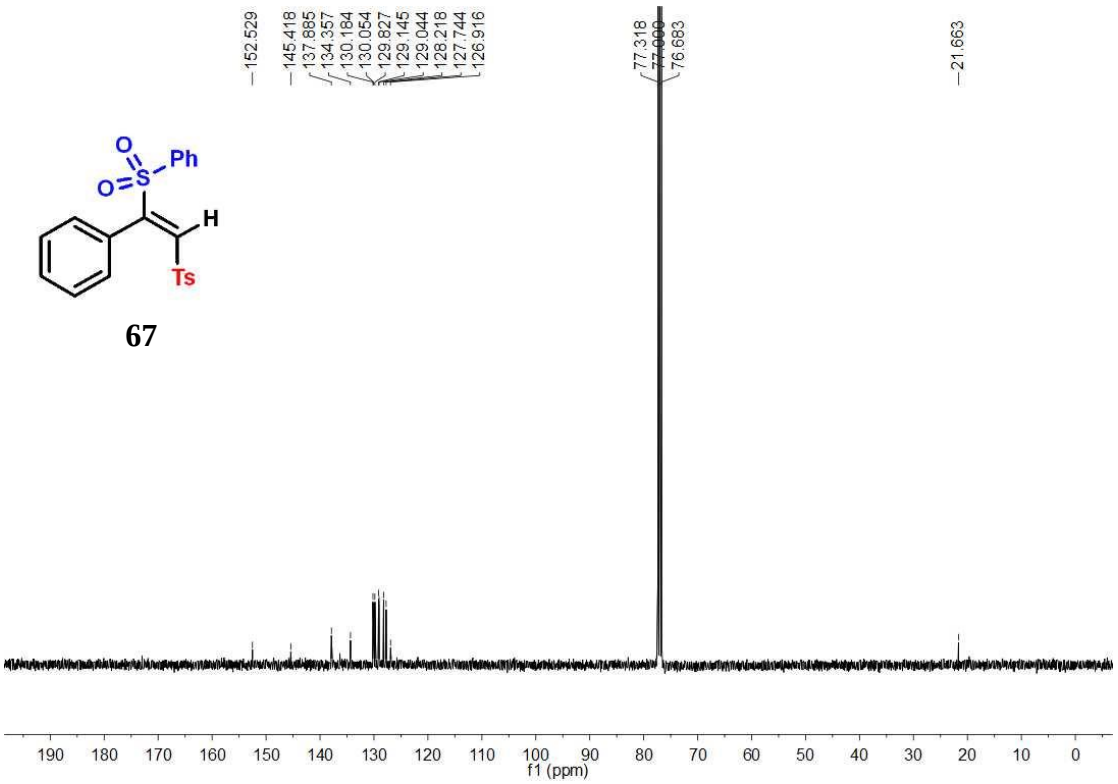

SPh
Ts
66
f1 (ppm)


SPh
Ts
66
f1 (ppm)

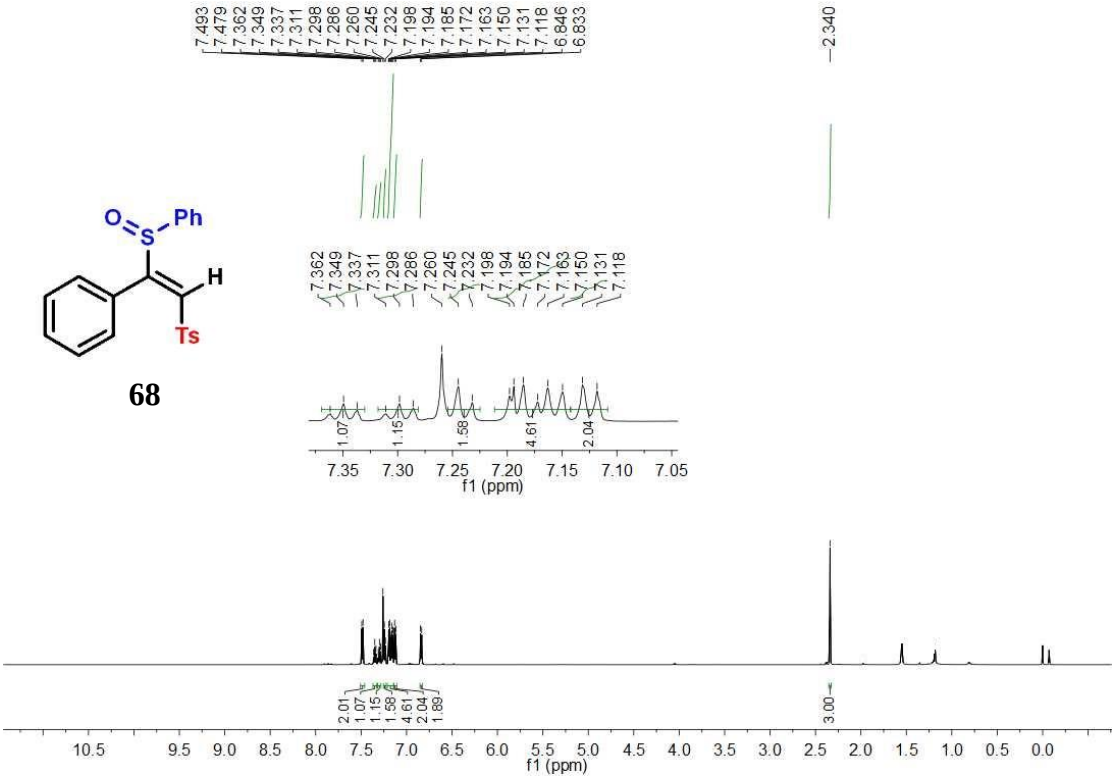

7.775
7.597
7.584
7.572
7.503
7.502
7.490
7.461
7.447
7.413
7.400
7.386
7.366
7.353
7.341
7.260
7.211
7.205
7.197
7.192
7.179
6.913
6.900
2.414
O
Ph
O=S
H
Ts

67

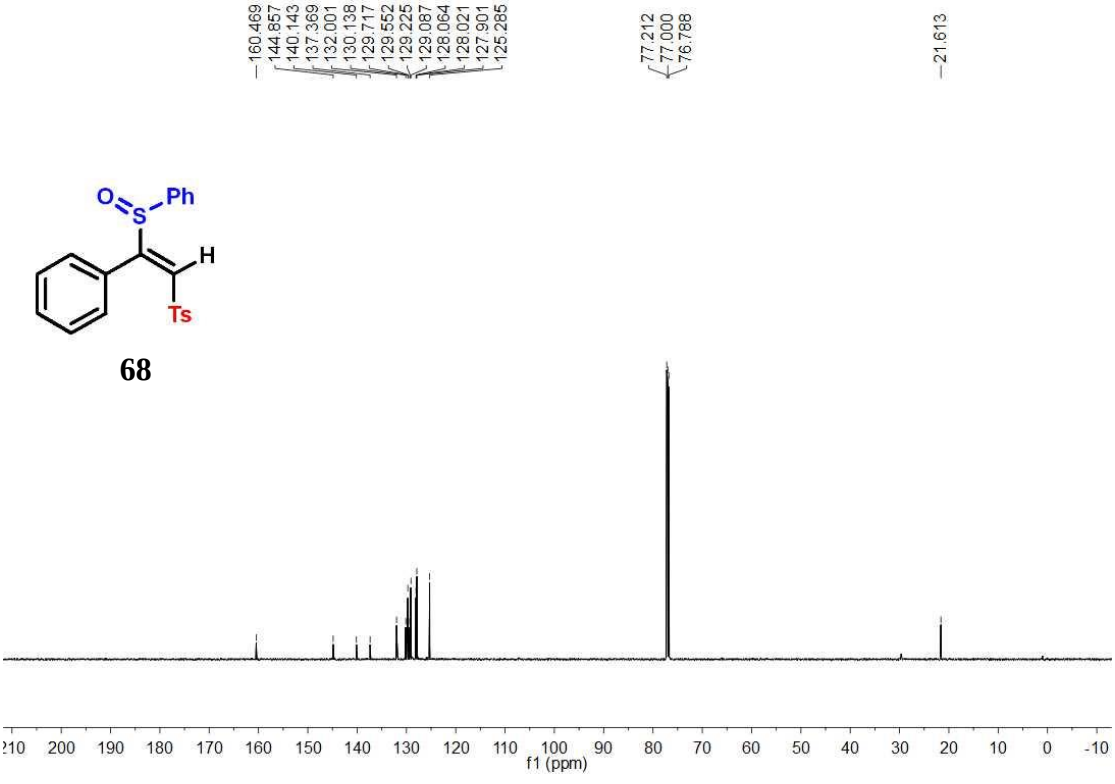

0.78
1.01
1.88
1.85
1.99
0.99
3.81
1.77
2.79
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


152.529
145.418
137.885
134.357
130.184
130.054
129.827
129.145
129.044
128.218
127.744
126.916
77.318
77.000
76.683
21.663
O
Ph
O=S
H
Ts
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

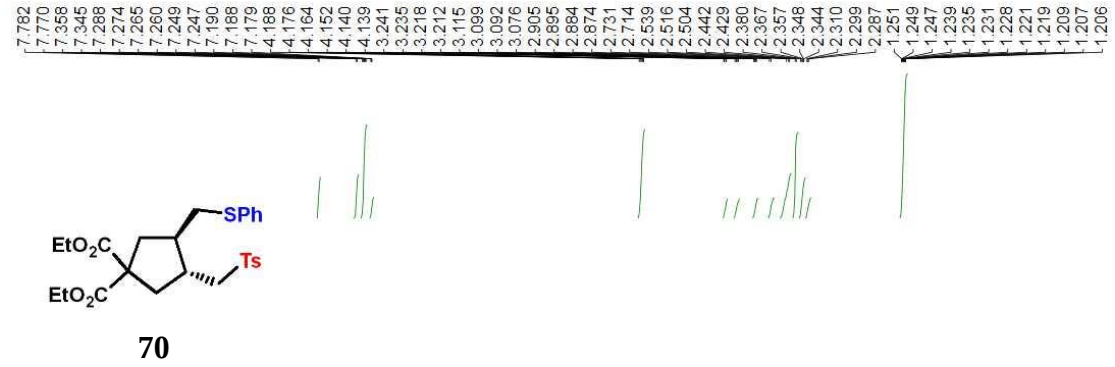
67


7.493
7.479
7.362
7.349
7.337
7.311
7.298
7.286
7.260
7.245
7.232
7.198
7.194
7.185
7.172
7.163
7.150
7.131
7.118
6.846
6.833
2.340
Ph
S
O
H
Ts
1.07
1.15
1.58
4.61
2.04
2.01
3.00
f1 (ppm)

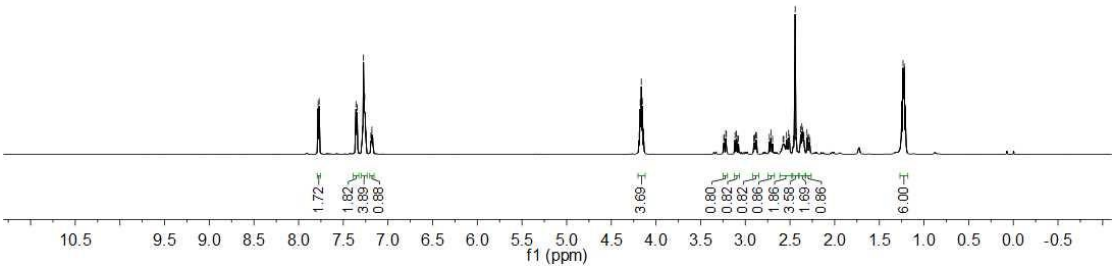
68


160.469
144.857
140.143
137.369
132.001
130.138
129.717
129.552
129.225
129.087
128.064
128.021
127.901
125.285
77.212
77.000
76.788
21.613
Ph
S
O
H
Ts
f1 (ppm)

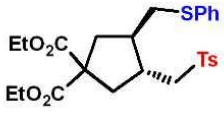
68


7.782
7.770
7.358
7.345
7.288
7.274
7.265
7.260
7.249
7.247
7.190
7.188
7.179
4.188
4.176
4.164
4.152
4.140
4.139
3.241
3.235
3.218
3.212
3.115
3.099
3.092
3.076
2.905
2.895
2.884
2.874
2.731
2.714
2.539
2.516
2.504
2.442
2.429
2.380
2.367
2.357
2.348
2.344
2.310
2.299
2.287
1.251
1.249
1.247
1.239
1.235
1.231
1.228
1.221
1.219
1.209
1.207
1.206
SPh
Ts
EtO2C
EtO2C

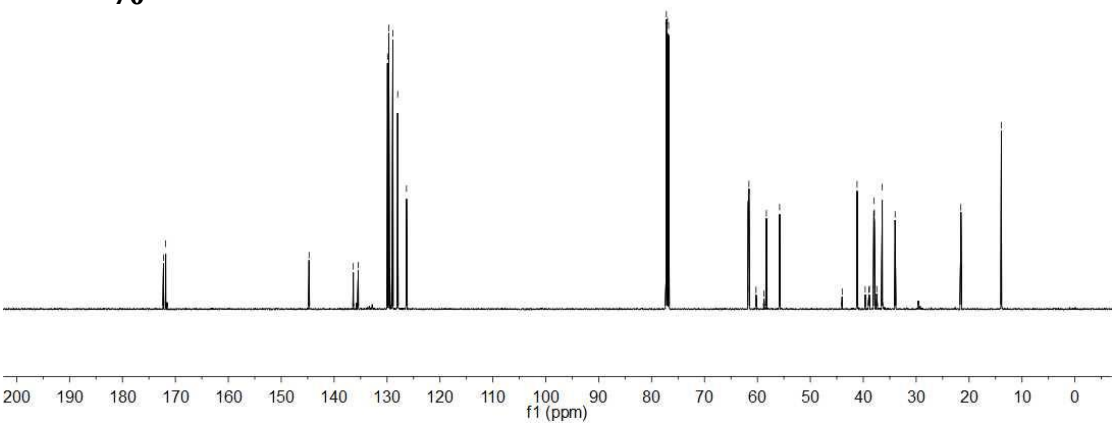
70


1.72
1.82
3.89
0.88
3.69
0.80
0.82
0.82
0.86
1.86
3.58
1.69
0.86
6.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


172.252
171.831
144.737
136.382
135.438
129.900
129.677
129.459
128.942
128.905
127.983
127.928
126.330
77.211
77.000
76.787
61.762
61.604
61.549
60.267
58.309
55.813
41.172
38.827
37.997
37.902
37.451
36.414
33.978
21.561
13.919
13.906


SPh
Ts
EtO2C
EtO2C

70


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)