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NMR spectra of symmetrical 1,2-diarylalkynes
NMR spectra of products

# **General information**

All photoinitiated reactions were carried out under inert atmosphere (argon or dinitrogen) at 25 °C. Thin layer chromatography was performed using pre-coated plates obtained from Merck (TLC silica gel 60 F254). TLC plates were visualized with 254 nm ultraviolet light (UV). Silica gel chromatography purifications were performed by flash chromatography using EM Science silica gel 60 (230-400 mesh).

NMR spectra were obtained on a Bruker Avance III HD (400 MHz <sup>1</sup>H, 101 MHz <sup>13</sup>C, 376 MHz <sup>19</sup>F). The chemical shifts are frequency referenced relative to the residual undeuterated solvent peaks.<sup>1</sup> Coupling constants *J* are given in Hertz as positive values regardless of their real individual signs. The multiplicity of the signals is indicated as "s", "d", "t" or "m" for singlet, doublet, triplet or multiplet, respectively. The abbreviation "br" is given for broadened signals.

High Resolution Mass Spectrometry spectra were carried out using AB Sciex TripleTOF 5600+ equipped with a TurboV and PhotoSpray ion sources. FT-IR spectra were obtained in a Bruker "Alpha-T" FTIR (KBr).

Chemicals and solvents were obtained from commercial sources and used without further purification.

### Light sources and photoreactors

LED strip lights with  $\lambda_{max}$ = 400 nm (violet, "Arlight RT-B60-10mm 12V UV400", 14.4 W/m), 470nm (blue, "Arlight RT 2-5000 24V Blue 2x2", 19.2 W/mm), 525 nm (green, "Arlight RT 2-5000 12V Green 2x", 14.4 W/m), 590 nm (orange, "Arlight RT 2-5000 24V Yellow 2x", 14.4 W/m), 625 nm (red, "Arlight RT 2-5000 12V Red 2x", 14.4 W/m) were used. A piece of LED strip was mounted onto internal surface of aluminum cup (internal diameter 90 mm, height 79 mm) with ventilation holes (diameter 8 mm) at the bottom. All led strips were powered by regulated DC power supply (violet: 12V 1.21A; blue: 24V 0.735A; green: 12V 1.25A; orange: 24V 0.635A; red: 12V 1.31A).



Figure S1. Photoreactors with different wavelength

The aluminum cup with LED light strip were placed in the PVC tube (internal diameter 150 mm, height 200 mm) with ventilation holes at the bottom (diameter 10 mm). Duct fan was placed on the top of PVC tube (air flow 280  $m^3/h$ ) to provide sufficient cooling of reaction mixture.



1. Side view of photoreactor setup



4. Upper view of photoreactor setup



2. Photoreactor setup without cooling fan



3. Upper view of photoreactor setup



5. Reaction mixture before and after reaction

Figure S2. Reaction setup

# **Optimization of reaction conditions**

Entry	Solvent (0.125M)	TsI, eq.	Time, h	Yield, %
1	CH <sub>2</sub> Cl <sub>2</sub>	1.2	4	67
2	PhMe	1.2	4	83
3	CH <sub>3</sub> CN	1.2	4	84
4	THF	1.2	4	48
5	EtOAc	1.2	4	82
6	n-heptane	1.2	4	80
7	n-hexane	1.2	4	80
8	CH <sub>3</sub> CN	1.6	4	99
9	CH <sub>3</sub> CN	1.6	2	90
10	CH <sub>3</sub> CN	1.6	1	90
11	CH <sub>3</sub> CN	1.6	0.5	90
12	CH <sub>3</sub> CN	1	12	79
13	CH <sub>3</sub> CN	2	1	99
14	$CH_2Cl_2$	2	1	93
15	(CH <sub>3</sub> ) <sub>2</sub> CO	2	1	85
16	MTBE	2	1	85
17	THF	2	1	95

**Table S1. Optimization of reaction conditions** 

 Table S2. Optimization of light wavelength

Entry	Solvent (0.125M)	TsI, eq.	Time, h	Yield, %
1	CH <sub>3</sub> CN, 500W halogen lamp	2	1	91
2	CH <sub>3</sub> CN, 400 nm (violet)	2	1	99
2	$CH_3CN$ , 470 nm (blue)	2	1	95
3	CH <sub>3</sub> CN, 525 nm (green)	2	1	92
4	CH <sub>3</sub> CN, 590 nm (orange)	2	1	85
5	CH <sub>3</sub> CN, 625 nm (red)	2	1	77
6	CH <sub>3</sub> CN, 25-28 °C, in the dark	2	1	trace

# Synthesis of starting materials

Starting materials were synthesized according to Symmetrical 1,2-diaryl alkynes **1** were synthesized according to the modified literature procedure below.<sup>2</sup>

Arylalkyl acetylenes **4** (except **4a-d**) were synthesized by Sonogashira coupling of aliphatic alkynes (1.05 eq.) and aryl iodides (1.0 eq.) in  $Et_3N$  using Pd(PPh\_3)<sub>2</sub>Cl<sub>2</sub> (2 mol%) and CuI (1 mol%) as catalysts.<sup>3</sup> Acetylenes **4a-d** were obtained by alkylation of lithium

phenylacetylide with dimethylsulfate, iodoethane, 1-iodobutane, 1-iodobexane in refluxing THF (82%, 90%, 92 and 98% yield, respectively).<sup>4</sup>



Scheme S1. Synthesis of starting materials

Sulfonyl iodides were synthesized from sodium arylsulfinates and iodine according to the literature procedure.<sup>5</sup>

### General procedure for synthesis of ArSO<sub>2</sub>I (GP-1)

A solution of  $I_2$  (2.53 g, 10 mmol) in EtOH (20 ml) was added dropwise to a solution of sodium arylsulfinate (10.56 mmol) in water (40 ml) at 5 °C. A mixture was stirred at 5 °C during 0.5h. Then it was filtered and the residue was washed with cold water. The residue was dried under high vacuum in the dark (covered with aluminum foil) at room temperature for 12h to give product as a yellow or orange powder with 72-98% yield. It was found, that product was pure and it was used without further purification. The product was stored in the freezer (-25 °C) in the glass-stoppered round-bottomed flask. The flask was brought to room temperature in the dark (covered with aluminum foil) prior to stopper removal. It was found, that products were stable under this conditions up to several month.

4-methylbenzene-1-sulfonyl iodide (2)

°,-I S,-I

Following **GP-1**, 4-methylbenzene-1-sulfonyl iodide (13.5 g, 90%) was obtained on 53.3 mmol scale as a yellow powder. The NMR data are in agreement with previously reported.<sup>6</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.75 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 2H), 2.47 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 147.7, 146.4, 129.8, 125.6, 22.0.

4-fluorobenzene-1-sulfonyl iodide (8a)



Following **GP-1**, 4-fluorobenzene-1-sulfonyl iodide (2.8 g, 98%) was obtained as an orange powder.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.18-7.63 (m, 2H), 7.26 (t, *J* = 8.4 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.02 (d, J = 259.5 Hz), 145.80 (d, J = 2.9 Hz), 128.65 (d, J = 10.0 Hz), 116.61 (d, J = 23.2 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -100.1(s, 1F, Ar-F).

4-chlorobenzene-1-sulfonyl iodide (8b)



Following **GP-1**, 4-chlorobenzene-1-sulfonyl iodide (2.9 g, 95%) was obtained as an orange powder.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.86-7.74 (m, 2H), 7.59-7.48 (m, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.0, 141.7, 129.6, 127.0.

4-bromobenzene-1-sulfonyl iodide (8c)



Following **GP-1**, 4-bromobenzene-1-sulfonyl iodide (2.5 g, 72%) was obtained as a yellow powder.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.71 (s, 4H).

# <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.4, 132.6, 130.4, 126.9.

#### General procedure for synthesis of symmetrical 1,2-diarylalkynes (GP-2)

A 100 ml Schlenk flask equipped with a magnetic stirring bar was charged with  $Pd(OAc)_2$  (56 mg, 2.5 mol%, 0.25 mmol), PPh<sub>3</sub> (131 mg, 5 mol%, 0.5 mmol) and CuI (190 mg, 10 mol%, 1 mmol). The Schlenk flask was evacuated and back-filled with argon followed by addition of CH<sub>3</sub>CN (10 ml), Et<sub>3</sub>N (3.04 g, 3 eq., 30 mmol) and aryl iodide (10 mmol, 1 eq.) if aryl iodide was liquid. Solid aryl iodides were transferred to the reaction flask after CuI. Next, lumps of CaC<sub>2</sub> (1.92 g, 3 eq., 30 mmol) in one portion. The reaction flask was transferred to a preheated oil bath (40°C) and left connected to the Schlenk line to avoid pressure buildup. After 12h the reaction mixture was cooled, filtered through silica gel pad (height 4 cm, diameter 3.5 cm), the solids remaining on the surface of silica gel pad were washed with CH<sub>2</sub>Cl<sub>2</sub> until no product isolated by flash chromatography on a silica gel (eluent: petroleum ether or petroleum ether-CH<sub>2</sub>Cl<sub>2</sub> mixture).

1,2-bis(p-tolyl)ethyne (1b)



Following **GP-2**, 1,2-bis(p-tolyl)ethyne (1.0 g, 97%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>7</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.49-7.36 (m, 4H), 7.20-7.09 (m, 4H), 2.37 (s, 6H, -CH<sub>3</sub>). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 138.3, 131.6, 129.2, 120.5, 89.0, 21.6.

1,2-bis(m-tolyl)ethyne (1c)

Following **GP-2**, 1,2-bis(m-tolyl)ethyne (1.01 g, 98%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>8</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41-7.31 (m, 4H), 7.25 (t, *J* = 7.6 Hz, 2H), 7.20-7.11 (m, 2H), 2.37 (s, 6H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 138.1, 132.3, 129.2, 128.8, 128.4, 123.3, 89.4, 21.4.



Following **GP-2**, 1,2-bis(4-fluorophenyl)ethyne (1.06 g, 94%) was obtained as a white powder. The NMR data are in agreement with previously reported. <sup>9</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.83-7.59 (m, 4H), 7.35-7.16 (m, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 162.69 (d, J = 249.7 Hz), 133.58 (d, J = 8.6 Hz), 119.35 (d, J = 3.8 Hz), 115.83 (d, J = 22.0 Hz), 88.1.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -110.6 (s, 2F, Ar-F).

1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1e)



Following **GP-2**, 1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1.48 g, 94%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>10</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.75-7.56 (m, 8H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 132.1, 130.65 (q, *J* = 32.7 Hz), 126.5, 125.54 (q, *J* = 3.8 Hz), 123.99 (q, *J* = 272.2 Hz), 90.3.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.9 (s, 6F, -CF<sub>3</sub>).

1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1f)



Following **GP-2**, 1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1.55 mg, 95%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>11</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87-7.77 (m, 2H), 7.76-7.67 (m, 2H), 7.66-7.57 (m, 2H), 7.50 (t, *J* = 7.8 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 134.9, 131.27 (q, *J* = 32.7 Hz), 129.1, 128.66 (q, *J* = 3.8 Hz), 125.42 (q, *J* = 3.7 Hz), 123.81 (q, *J* = 272.5 Hz) 123.7, 89.4.

1,2-bis(4-chlorophenyl)ethyne (1g)



Following **GP-2**, 1,2-bis(4-chlorophenyl)ethyne (1.24 g, 95%) was obtained as a brown powder. The NMR data are in agreement with previously reported.<sup>9</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.48-7.41 (m, 4H), 7.37-7.29 (m, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 134.7, 132.9, 128.9, 121.6, 89.3.

1,2-bis(4-nitrophenyl)ethyne (1h)



Following **GP-2**, 1,2-bis(4-nitrophenyl)ethyne (0.85 g, 63%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>12</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.51-8.02 (m, 4H), 7.84-7.51 (m, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 147.8, 132.8, 129.0, 123.9, 92.1.

1,2-bis(3-nitrophenyl)ethyne (1i)



Following **GP-2**, 1,2-bis(3-nitrophenyl)ethyne (1.2 g, 90%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>13</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.40 (t, *J* = 1.9 Hz, 2H), 8.24 (ddd, *J* = 8.3, 2.3, 1.1 Hz, 2H), 7.86 (dt, *J* = 7.8, 1.3 Hz, 2H), 7.58 (t, *J* = 8.0 Hz, 2H).<sup>13</sup>

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.3, 137.5, 129.7, 126.7, 124.2, 123.8, 89.3.

diethyl 4,4'-(ethyne-1,2-diyl)dibenzoate (1j)



Following **GP-2**, diethyl 4,4'-(ethyne-1,2-diyl)dibenzoate (1.32 g, 82%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>14</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.25-7.89 (m, 4H), 7.84-7.45 (m, 4H), 4.39 (q, *J* = 7.1 Hz, 4H, -CH<sub>2</sub>-), 1.40 (t, *J* = 7.1 Hz, 6H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.1, 131.7, 130.4, 129.7, 127.4, 91.5, 61.3, 14.5.

1,2-bis(4-methoxyphenyl)ethyne (1k)



Following **GP-2**, 1,2-bis(4-methoxyphenyl)ethyne (1.15 g, 96%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>12</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.54-7.37 (m, 4H), 6.95-6.81 (m, 4H), 3.82 (s, 6H, -OCH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 159.5, 133.0, 115.8, 114.1, 88.1, 55.4.

1,2-di(thiophen-2-yl)ethyne (11)



Following **GP-2**, (0.85 g, 89%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>2</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31 (dd, J = 5.2, 1.2 Hz, 1H), 7.29 (dd, J = 3.7, 1.2 Hz, 1H), 7.02 (dd, J = 5.2, 3.6 Hz, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 132.2, 127.8, 127.3, 123.0, 86.3.



Following **GP-2**, 1,2-di(thiophen-3-yl)ethyne (0.83 g, 87%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>9</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.51 (dd, J = 3.0, 1.2 Hz, 2H), 7.33-7.27 (m, 2H), 7.19 (dd, J = 5.0, 1.2 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 129.9, 128.6, 125.5, 122.3, 84.1.

1,2-di(naphthalen-2-yl)ethyne (**1n**)



Following **GP-2**, 1,2-di(naphthalen-2-yl)ethyne (0.9 g, 65%) was obtained as a white powder. The NMR data are in agreement with previously reported.<sup>15</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.11 (s, 2H), 7.91-7.78 (m, 6H), 7.64 (dd, *J* = 8.5, 1.7 Hz, 2H), 7.59-7.45 (m, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 133.2, 133.0, 131.7, 128.6, 128.2, 128.0, 127.9, 126.8, 126.7, 120.8, 90.3.

# Synthesis and further functionalization of β-iodovinyl sulfones

# General procedure 2 (GP-3): iodosulfonylation of disubstituted acetylenes

A screw capped 8 ml vial equipped with a magnetic stirring bar was charged with disubstituted alkyne (0.5 mmol),  $CH_3CN$  (4 ml) followed by sulfonyl iodide (1 mmol). The argon flow was bubbled through the reaction mixture for 1 minute. The reaction mixture was stirred under the violet light irradiation (wavelength 400 nm) during 1h. The temperature of the reaction mixtures in the vials was measured during and after the completion of the reaction, and it did not exceed 27 °C. The solvent was then evaporated in vacuum to provide a crude product. The pure product was obtained after the column chromatography purification ( $CH_2Cl_2$ -Petroleum ether mixture as the eluent) as a white, yellow, pink oil or solid.

Procedure for 15-mmol scale synthesis of 3a:



1. Reaction mixture before reaction



4. Upper view of reaction setup



2. The flask in the photoreactor



3. Side view of reaction setup



5. Reaction mixture after reaction

# Figure S3. Reaction setup for 15-mmol scale synthesis

A 250 ml Schlenk flask equipped with a magnetic stir bar was charged with 1,2diphenylethyne (15 mmol, 2.64 g) and TsI (30 mmol, 8.46 g). The flask was evacuated and filled with argon, and the cycle was repeated twice, followed by addition of dry CH<sub>3</sub>CN (120 ml). The solution was degassed using 3 freeze-pump-thaw cycles and then placed into photoreactor and stirred for 1.5h under violet light irradiation. The temperature of the reaction mixtures in the vials was measured during and after the completion of the reaction, and it did not exceed 27 °C.The reaction mixture was diluted with water (240 ml) filtered off. The residue was washed with the mixture of CH<sub>3</sub>CN (100 ml) and H<sub>2</sub>O (200 ml). After drying under low pressure the pure product was obtained as a white solid with 96% (6.6 g) yield.

### Procedure for 10-mmol scale synthesis of 5h:

A 250 ml Schlenk flask equipped with a magnetic stir bar was charged with 1-(oct-1ynyl)-4-(trifluoromethyl)benzene (10 mmol, 2.53 g) and TsI (20 mmol, 5.64 g). The flask was evacuated and filled with argon, and the cycle was repeated twice, followed by addition of dry CH<sub>3</sub>CN (80 ml). The solution was degassed using 3 freeze-pump-thaw cycles and then placed into photoreactor and stirred for 1.5h under violet light irradiation. The reaction mixture was evaporated. The product was obtained with using column chromatography purification of the crude residue (CH<sub>2</sub>Cl<sub>2</sub>-Petroleum ether mixture as the eluent). The pure product was isolated as a white solid with 80% (4.3 g) yield.

#### General procedure 3 (GP-4): Suzuki-Miyaura cross-coupling of β-iodovinylsulfones

The chosen  $\beta$ -iodo vinylsulfone (1 eq., 1 mmol), arylboronic acid (1.1 eq., 1.1 mmol), SPhos (2 mol%, 0.02 mmol, 8.21 mg), Pd(OAc)<sub>2</sub> (1 mol%, 0.01 mmol, 2.25 mg), n-Bu<sub>4</sub>NBr (10 mol%, 0.1 mmol, 32.2 mg), K<sub>2</sub>CO<sub>3</sub> (2 eq., 2 mmol, 276 mg) were mixed with toluene (1 ml) and water (1 ml). Then the mixture was heated to reflux during 12 h. After that reaction mixture was quenched with water (10 ml) and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3x10 ml). The solvent was dried using Na<sub>2</sub>SO<sub>4</sub> then this filtered and removed under vacuum. Next the crude product was purified by the silica gel column chromatography with using the mixture CH<sub>2</sub>Cl<sub>2</sub> and petroleum ether as the eluent to afford the desired product.

#### General procedure 4 (GP-5): Sonogashira cross-coupling of β-iodovinylsulfone

The 1-ethynyl-4-methoxybenzene (1.2eq., 1.2 mmol, 159 mg), (Z)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene (1 eq., 1 mmol, 536.4 mg), PdCl<sub>2</sub>(Ph<sub>3</sub>P)<sub>2</sub> (3 mol%, 0.03 mmol, 21 mg), CuI (3 mol%, 0.03 mmol, 6 mg) and Et<sub>3</sub>N (3 ml) were mixed. Then the mixture was heated to 50 °C during 12 h. The solvent was removed under vacuum. Next the crude product was purified by the silica gel column chromatography with using the mixture CH<sub>2</sub>Cl<sub>2</sub>/ petroleum ether = 1/1 as the eluent to afford the desired product.

#### **Characterization of Products**

(*E*)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (**3***a*)



Following **GP-3**, (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (227 mg, 99%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 196-198 °C. The NMR data are in agreement with previously reported.<sup>16</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.43-7.30 (m, 8H, Ph-H), 7.26 (d, J = 8.2 Hz, 2H, Ts-H), 7.21-7.14 (m, 2H, Ph-H), 7.10 (d, J = 8.2 Hz, 2H, Ts-H), 2.37 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.3, 144.4, 142.7, 139.5, 136.9, 130.4, 129.3, 129.1, 128.7, 128.5, 128.0, 127.5, 118.2, 21.7.

IR (v/cm<sup>-1</sup>): 3062 (W), 3048 (W), 2977 (VW), 2920 (VW), 1622 (M), 1597 (M), 1588 (W), 1492 (W), 1447 (M), 1400 (W), 1384 (W), 1326 (VS), 1304 (M), 1278 (W), 1235 (VW), 1174 (M), 1161 (M), 1148 (VS), 1115 (W), 1085 (M), 1021 (W), 1001 (W), 946 (W), 907 (W), 860 (VW), 811 (M), 799 (W), 781 (S), 750 (W), 710 (M), 699 (S), 696 (S), 669 (W), 656 (W), 629 (M), 582 (S), 559 (VS), 530 (M), 495 (W), 481 (W), 448 (VW).



Following **GP-3**, (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (226 mg, 93%) was obtained as a white powder, m.p. (CHCl<sub>3</sub>) 174-175 °C. The NMR data are in agreement with previously reported.<sup>17</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.38-7.28 (m, 4H), 7.26-7.17 (m, 4H), 7.17-7.12 (m, 2H), 7.12-7.06 (m, 2H), 2.44 (s, 3H, -CH<sub>3</sub>), 2.43 (s, 3H, -CH<sub>3</sub>), 2. 42 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.9, 144.2, 140.0, 139.3, 139.2, 137.1, 136.7, 130.3, 129.2, 129.1, 128.7, 128.6, 127.5, 118.9, 21.8, 21.6.

IR (v/cm<sup>-1</sup>): 3041 (W), 3026 (W), 2949 (W), 2918 (W), 2867 (W), 1911 (VW), 1793 (VW), 1617 (M), 1596 (M), 1509 (M), 1494 (W), 1449 (W), 1404 (W), 1382 (W), 1313 (S), 1300 (S), 1290 (M), 1208 (W), 1188 (M), 1178 (M), 1146 (VS), 1122 (W), 1084 (S), 1039 (W), 1021 (M), 949 (W), 927 (M), 823 (S), 809 (S), 786 (W), 757 (W), 727 (W), 705 (M), 686 (M), 665 (W), 652 (S), 613 (VW), 589 (S), 566 (VS), 535 (S), 518 (W), 505 (M), 468 (M), 418 (VW).

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3c)



Following **GP-3**, (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (232 mg, 95%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 172-173 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.19 (m, 6H), 7.17-7.08 (m, 4H), 7.08-7.05 (s, 1H), 7.04-6.99. (m, 2H), 2.41 (s, 3H, -CH<sub>3</sub>), 2.39 (s, 3H, -CH<sub>3</sub>), 2. 37 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.5, 144.1, 142.5, 139.4, 138.2, 137.6, 137.3, 130.9, 130.1, 129.9, 129.2, 128.7, 128.3, 128.1, 127.9, 127.5, 124.8, 118.1, 21.7, 21.6, 21.5.

IR (v/cm<sup>-1</sup>): 3061 (VW), 3037 (W), 3025 (W), 2976 (W), 2950 (W), 2916 (W), 2853 (W), 2732 (VW), 1903 (VW), 1788 (VW), 1621 (W), 1595 (M), 1583 (W), 1492 (W), 1480 (W), 1455 (W), 1414 (W), 1374 (W), 1313 (S), 1301 (S), 1288 (M), 1266 (W), 1234 (W), 1168 (W), 1143 (VS), 1115 (W), 1085 (S), 1039 (W), 1018 (W), 996 (W), 966 (W), 893 (W), 828 (W), 802 (S), 750 (VW), 702 (S), 690 (S), 669 (M), 654 (W), 636 (W), 629 (W), 593 (M), 565 (S), 549 (W), 527 (W), 522 (W), 503 (W), 494 (M), 480 (W), 459 (VW), 435 (W), 415 (VW).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{22}IO_2S^+)$  [M+H]<sup>+</sup> 489.0380; found: 489.0377.



Following **GP-3**, (238 mg, 96%) (E)-4,4'-(1-iodo-2-tosylethene-1,2diyl)bis(fluorobenzene) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 196-197 °C (lit. data<sup>18</sup>: m.p. 188-190 °C). The NMR data are in agreement with previously reported<sup>18</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.37 (dd, J = 8.7, 5.2 Hz, 2H), 7.29-7.26 (m, 2H), 7.18-7.11 (m, 4H), 7.09-7.01 (m, 4H), 2.39 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 163.27 (d, J = 250.1 Hz), 162.99 (d, J = 250.2 Hz), 149.0, 144.8, 138.5 (d, J = 3.5 Hz), 135.4 (d, J = 3.6 Hz), 136.7, 132.5 (d, J = 8.6 Hz), 129.7 (d, J = 8.5 Hz), 132.5, 132.4, 129.7, 129.6, 129.5, 128.6, 117.5, 115.8 (d, J = 21.9 Hz), 115.2 (d, J = 22.1 Hz), 21.8.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -110.8 (s, 1F, Ar-F), -111.1 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3103 (VW), 3065 (W), 3045 (VW), 2921 (VW), 1633 (W), 1624 (W), 1601 (M), 1508 (VS), 1407 (VW), 1384 (VW), 1324 (S), 1304 (M), 1290 (W), 1241 (S), 1233 (S), 1178 (W), 1158 (S), 1148 (VS), 1095 (W), 1085 (M), 1017 (W), 958 (W), 925 (W), 860 (W), 847 (S), 836 (M), 807 (M), 770 (M), 744 (W), 712 (W), 709 (M), 694 (M), 672 (W), 641 (W), 630 (W), 589 (S), 569 (S), 532 (M), 515 (W), 509 (W), 490 (M), 472 (W), 462 (W).

(E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (**3e**)



Following **GP-3**, (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (295 mg, 99%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  242-243 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.64 (t, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.1 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.28-7.22 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 1H), 2.39 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.6, 145.7, 145.3, 142.4, 136.1, 131.61 (q, J = 32.8 Hz, 1C, <u>C</u>-CF<sub>3</sub>), 131.10 (q, J = 32.8 Hz, 1C, <u>C</u>-CF<sub>3</sub>), 130.8, 129.7, 128.6, 127.7, 125.65 (q, J = 3.7 Hz, 2C, -<u>C</u>-C-CF<sub>3</sub>), 125.15 (q, J = 3.7 Hz, 2C, -<u>C</u>-C-CF<sub>3</sub>), 123.87 (q, J = 272.5 Hz, 1C, -CF<sub>3</sub>), 123.84 (q, J = 272.4 Hz, 1C, -CF<sub>3</sub>), 115.6, 21.8.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.74 (s, 3F, -CF<sub>3</sub>), -62.75 (s, 3F, -CF<sub>3</sub>).

IR (v/cm<sup>-1</sup>): 1629 (M), 1614 (M), 1409 (M), 1325 (VS), 1304 (M), 1191 (M), 1176 (S), 1153 (S), 1123 (S), 1109 (M), 1085 (M), 1067 (S), 1018 (M), 936 (W), 856 (M), 848 (M), 813 (M), 757 (W), 710 (W), 700 (W), 682 (W), 638 (W), 628 (M), 606 (W), 598 (M), 562 (M), 530 (M), 503 (W), 467 (W).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{16}F_6IO_2S^+)$  [M+H]<sup>+</sup> 596.9814; found: 596.9814.

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (**3**f)



Following **GP-3**, (295 mg, 99%) (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  159-161 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.70-7.63 (m, 2H), 7.63-7.54 (m, 3H), 7.54-7.47 (m, 2H), 7.29 (s, 1H), 7.24-7.19 (m, 2H), 7.16-7.11 (m, 2H), 2.38 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.8, 145.5, 142.9, 139.8, 136.1, 133.7, 131.1 (q, J = 32.8 Hz), 130.6 (q, J = 32.9 Hz), 129.7, 129.3, 128.8, 128.6, 127.4 (q, J = 3.7 Hz), 126.5 (q, J = 281.7 Hz, -CF<sub>3</sub>), 126.3 (q, J = 3.7 Hz), 126.1 (q, J = 3.7 Hz), 124.1 (q, J = 3.8 Hz), 115.9, 21.7.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.7 (s, 3F, CF<sub>3</sub>), -62.8 (s, 3F, CF<sub>3</sub>).

IR (v/cm<sup>-1</sup>): 2958 (VW), 2925 (W), 2853 (VW), 1638 (M), 1384 (M), 1331 (W), 1223 (VW), 1149 (W), 1126 (W), 1081 (VW), 969 (VW), 810 (W), 702 (W), 684 (W), 662 (W), 559 (W), 531 (W).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{16}F_6IO_2S^+)$  [M+H]<sup>+</sup> 596.9814; found: 596.9811.

(E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (**3g**)



Following **GP-3**, (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (214 mg, 81%) was obtained as a pale-pink powder, m.p. ( $CH_2Cl_2$ ) 199-200 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41-7.36 (m, 4H), 7.36-7.28 (m, 4H), 7.19 (d, *J* = 8.2 Hz, 2H), 7.16-7.11 (m, 2H), 2.44 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.0, 144.9, 140.8, 137.6, 136.5, 135.7, 135.3, 131.8, 129.6, 128.9, 128.8, 128.6, 128.3, 116.9, 21.8.

IR (v/cm<sup>-1</sup>): 3080 (VW), 3052 (VW), 2921 (VW), 1911 (VW), 1612 (W), 1594 (W), 1489 (M), 1398 (M), 1378 (W), 1314 (VS), 1304 (M), 1291 (M), 1266 (W), 1183 (W), 1145 (S),

1085 (S), 1015 (M), 956 (W), 931 (M), 835 (M), 829 (M), 810 (S), 727 (M), 712 (M), 687 (M), 672 (W), 656 (W), 641 (W), 569 (VS), 531 (M), 484 (W), 418 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{16}Cl_2IO_2S^+)$  [M+H]<sup>+</sup> 528.9287; found: 528.9287.

(E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h)



Following **GP-3** (due to low solubility of **1h** under experimental conditions, TsI was added in two portions (1 eq., 0.5 mmol). The first portion of TsI was added initially, the second one - after 1h of irradiation in photoreactor. The total irradiation time of reaction mixture was 2 hours), (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (198 mg, 72%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 238-239 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.34-8.26 (m, 2H), 8.26-8.19 (m, 2H), 7.61-7.52 (m, 2H), 7.38-7.31 (m, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 2.41 (s, 1H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.2, 148.4, 148.3, 148.0, 146.0, 144.7, 135.5, 131.6, 130.0, 128.7, 128.2, 123.9, 123.6, 114.6, 21.9.

IR (v/cm<sup>-1</sup>): 3114 (W), 2925 (VW), 2854 (VW), 1635 (M), 1595 (M), 1515 (VS), 1490 (W), 1379 (W), 1349 (VS), 1320 (S), 1286 (M), 1184 (W), 1150 (S), 1084 (M), 1014 (W), 939 (W), 868 (M), 820 (W), 815 (M), 764 (W), 702 (M), 571 (S), 523 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{16}IN_2O_6S^+)$  [M+H]<sup>+</sup> 550.9768; found: 550.9770.

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3i)



Following **GP-3** (due to low solubility of **1i** under experimental conditions, TsI was added in two portions (1 eq., 0.5 mmol). The first portion of TsI was added initially, the second one - after 1h of irradiation in photoreactor. The total irradiation time of reaction mixture was 2 hours), (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (256 mg, 93%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 279-280 °C.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 8.50-8.03 (m, 4H), 7.97-7.64 (m, 4H), 7.40-7.15 (m, 4H), 2.32 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 147.5, 147.1, 147.0, 144.9, 143.7, 139.6, 135.7, 133.4, 130.3, 129.8, 129.7, 128.0, 124.3, 123.3, 121.6, 117.3, 21.0.

IR (v/cm<sup>-1</sup>): 3107 (W), 3069 (W), 2924 (VW), 2865 (VW), 1626 (W), 1614 (W), 1594 (W), 1533 (VS), 1523 (S), 1491 (W), 1476 (W), 1431 (W), 1404 (W), 1350 (VS), 1330 (M), 1312 (S), 1304 (M), 1193 (W), 1188 (W), 1183 (W), 1144 (S), 1120 (W), 1082 (M), 1022 (VW), 1016 (VW), 981 (W), 908 (W), 899 (VW), 860 (W), 820 (M), 810 (M), 751 (W), 731 (W), 699 (M), 690 (S), 676 (M), 658 (W), 635 (W), 588 (M), 562 (S), 530 (W), 475 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{16}IN_2O_6S^+)$  [M+H]<sup>+</sup> 550.9768; found: 550.9768.

(E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j)



Following **GP-3** (due to low solubility of **1j** under experimental conditions, TsI was added in two portions (1 eq., 0.5 mmol). The first portion of TsI was added initially, the second one - after 1h of irradiation in photoreactor. The total irradiation time of reaction mixture was 2 hours), (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (293 mg, 97%) was obtained as a pale-pink powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 194-195 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.09 (d, *J* = 8.2 Hz, 2H), 8.05 (d, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 8.1 Hz, 2H), 7.31-7.21 (m, 4H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.42 (qd, *J* = 7.2, 2.9 Hz, 4H, -<u>CH<sub>2</sub></u>-CH<sub>3</sub>), 2.40 (s, 3H, -CH<sub>3</sub>), 1.43 (t, *J* = 7.1 Hz, 6H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.1, 165.9, 149.1, 146.7, 145.1, 143.3, 136.2, 131.4, 131.0, 130.5, 129.7, 129.7, 129.4, 128.8, 127.3, 116.5, 61.5, 61.3, 21.8, 14.5, 14.5.

IR (v/cm<sup>-1</sup>): 3062 (W), 2977 (W), 2934 (W), 2905 (VW), 1717 (VS), 1629 (W), 1596 (M), 1465 (W), 1447 (W), 1406 (M), 1384 (W), 1369 (M), 1328 (S), 1311 (M), 1274 (VS), 1180 (M), 1152 (S), 1103 (S), 1085 (M), 1019 (M), 938 (W), 868 (W), 812 (M), 781 (M), 706 (S), 687 (M), 641 (W), 634 (W), 608 (VW), 567 (S), 536 (M), 513 (W).

ESI-HRMS (m/z): calc. for  $(C_{27}H_{26}IO_6S^+)$  [M+H]<sup>+</sup> 605.0489; found: 605.0482.

(E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (3k)



Following **GP-3**, (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (234 mg, 90%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 187-189 °C (lit. data<sup>18</sup>: m.p. 176-178 °C). The NMR data are in agreement with previously reported<sup>18</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35-7.29 (m, 2H), 7.29-7.25 (m, 2H), 7.16-7.04 (m, 4H), 6.90-7.81 (m, 4H), 3.85 (s, 3H, -OCH<sub>3</sub>), 3.83 (s, 3H, -OCH<sub>3</sub>), 2.36 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 160.2, 160.2, 148.5, 144.1, 137.3, 135.0, 132.1, 131.9, 129.5, 129.3, 128.5, 119.4, 113.8, 113.3, 55.4, 21.7.

IR (v/cm<sup>-1</sup>): 2927 (VW), 2836 (VW), 1624 (M), 1607 (M), 1578 (W), 1513 (W), 1465 (W), 1441 (W), 1326 (W), 1294 (W), 1249 (W), 1178 (W), 1149 (W), 1112 (W), 1085 (W), 1026 (W), 922 (VW), 847 (W), 823 (W), 810 (W), 754 (W), 697 (W), 643 (W), 596 (W), 573 (M), 544 (W), 521 (W).

(E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3l)



Following **GP-3**, (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (189 mg, 80%) was obtained as a yellow powder, m.p. ( $CH_2Cl_2$ ) 159-161 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.50 (dd, J = 5.1, 1.2 Hz, 1H), 7.45-7.37 (m, 4H), 7.17-7.10 (m, 2H), 7.02-6.97 (m, 2H), 6.95 (dd, J = 3.6, 1.3 Hz, 1H), 2.38 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.3, 144.5, 143.7, 139.7, 136.7, 131.1, 130.0, 129.4, 129.0, 129.0, 128.5, 126.9, 126.8, 111.7, 21.8.

IR (v/cm<sup>-1</sup>): 3109 (M), 3070 (W), 2959 (VW), 2924 (W), 2853 (VW), 1805 (W), 1733 (W), 1592 (M), 1517 (W), 1493 (M), 1452 (W), 1420 (M), 1410 (M), 1375 (W), 1352 (M), 1345 (W), 1309 (S), 1301 (S), 1290 (S), 1236 (S), 1211 (W), 1186 (M), 1151 (VS), 1127 (S), 1083 (S), 1041 (M), 1018 (M), 892 (M), 860 (M), 840 (M), 811 (S), 750 (M), 728 (S), 704 (VS), 667 (S), 637 (M), 599 (M), 568 (S), 548 (VS), 525 (S), 484 (M), 449 (W).

ESI-HRMS (m/z): calc. for  $(C_{17}H_{14}IO_2S_3^+)$  [M+H]<sup>+</sup> 472.9195; found: 472.9195.

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (**3m**)



Following **GP-3**, (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (191 mg, 81%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 216-217 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.50 (dd, J = 3.0, 1.3 Hz, 1H), 7.35-7.29 (m, 2H), 7.29-7.22 (m, 3H), 7.15-7.08 (m, 2H), 7.02 (dd, J = 5.0, 1.3 Hz, 1H), 6.91 (dd, J = 4.6, 1.6 Hz, 1H), 2.36 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 13C NMR (101 MHz, CDCl3) δ 145.8, 144.3, 141.4, 138.6, 137.2, 129.4, 128.9, 128.2, 128.1, 127.6, 125.9, 125.6, 125.1, 111.8, 21.8.

IR (v/cm<sup>-1</sup>): 3109 (M), 3090 (M), 3075 (W), 1662 (W), 1618 (W), 1596 (M), 1490 (W), 1406 (W), 1354 (VW), 1320 (VS), 1303 (M), 1230 (W), 1181 (W), 1153 (VS), 1145 (S), 1117 (W), 1087 (M), 1019 (W), 985 (W), 953 (W), 877 (W), 865 (M), 850 (W), 830 (M), 812 (S), 794 (W), 778 (W), 722 (W), 704 (M), 690 (M), 668 (S), 636 (M), 632 (M), 625 (M), 606 (W), 577 (M), 546 (M), 524 (M), 467 (W), 452 (W).

ESI-HRMS (m/z): calc. for  $(C_{17}H_{14}IO_2S_3^+)$  [M+H]<sup>+</sup> 472.9195; found: 472.9194.

(E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n)



Following **GP-3** (concentration of alkyne was decreased twice and *n*-BuOAc used as a solvent instead of CH<sub>3</sub>CN due to low solubility of **10** under experimental conditions), (E)-2,2'- (1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (277 mg, 99%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 215-217 °C with decomposition.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.95-7.77 (m, 8H), 7.62-7.50 (m, 4H), 7.49-7.41 (m, 1H), 7.39-7.32 (m, 1H), 7.23 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 2.30 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.9, 144.4, 139.8, 137.1, 136.8, 133.5, 133.4, 133.0, 132.5, 130.3, 129.3, 128.7, 128.6, 128.6, 128.3, 128.0, 127.8, 127.6, 127.2, 126.9, 126.8, 126.6, 125.3, 118.5, 21.7.

IR (v/cm<sup>-1</sup>): 3050 (W), 2921 (W), 2853 (W), 1614 (M), 1596 (M), 1503 (W), 1493 (W), 1432 (W), 1402 (W), 1382 (W), 1359 (W), 1324 (S), 1313 (S), 1302 (M), 1271 (W), 1244 (W), 1182 (W), 1146 (VS), 1117 (M), 1085 (S), 1019 (W), 979 (M), 927 (M), 899 (W), 863 (M), 825 (M), 806 (S), 772 (W), 758 (M), 750 (S), 710 (M), 677 (S), 649 (M), 626 (M), 576 (S), 559 (S), 548 (W), 542 (M), 530 (M), 509 (M), 477 (M), 457 (W), 422 (VW).

ESI-HRMS (m/z): calc. for  $(C_{29}H_{22}IO_2S^+)$  [M+H]<sup>+</sup> 561.0380; found: 561.0381.



Following **GP-3**, (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (181 mg, 91%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 128-130 °C. The NMR data are in agreement with previously reported<sup>16</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43-7.36 (m, 2H), 7.26-7.20 (m, 3H), 7.20-7.14 (m, 2H), 7.14-7.06 (m, 2H), 2.51 (s, 3H, -CH<sub>3</sub>), 2.39 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.3, 144.1, 143.1, 137.5, 129.6, 128.8, 127.9, 127.8, 127.7, 115.9, 27.2, 21.7.

IR (v/cm<sup>-1</sup>): 3060 (W), 3051 (W), 3030 (W), 2926 (W), 2854 (VW), 1960 (VW), 1926 (VW), 1895 (VW), 1813 (VW), 1768 (VW), 1627 (M), 1594 (M), 1576 (W), 1493 (W), 1442 (M), 1399 (W), 1374 (W), 1317 (S), 1305 (S), 1291 (M), 1212 (M), 1183 (M), 1154 (VS), 1120 (M), 1080 (S), 1031 (W), 1020 (M), 1004 (M), 992 (M), 977 (W), 921 (W), 848 (S), 818 (S), 801 (M), 764 (VS), 712 (S), 695 (S), 645 (S), 634 (M), 584 (W), 557 (VS), 531 (S), 499 (M), 473 (M), 412 (W).

ESI-HRMS (m/z): calc. for  $(C_{16}H_{16}IO_2S^+)$  [M+H]<sup>+</sup> 398.9910; found: 398.9913.

(E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (5b)



Following **GP-3**, (E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (187 mg, 91%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 128-130 °C (lit. data<sup>19</sup>: m.p. 114-115 °C). The NMR data are in agreement with previously reported<sup>19</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.31-7.23 (m, 2H), 7.22-7.11 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.05-6.98 (m, 2H), 2.95 (q, *J* = 7.4 Hz, 2H, -<u>CH<sub>2</sub></u>-CH<sub>3</sub>), 2.36 (s, 3H, Ar-CH<sub>3</sub>), 1.32 (t, *J* = 7.4 Hz, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.9, 143.9, 142.9, 137.9, 130.5, 129.4, 128.6, 127.9, 127.8, 127.7, 115.0, 77.2, 33.6, 21.7, 12.9.

IR (v/cm<sup>-1</sup>): 3055 (W), 2993 (W), 2972 (W), 2934 (W), 2871 (W), 1622 (M), 1589 (M), 1489 (W), 1463 (W), 1441 (M), 1397 (W), 1374 (W), 1320 (S), 1314 (S), 1304 (S), 1291 (M), 1271 (W), 1206 (W), 1183 (W), 1153 (VS), 1124 (M), 1081 (S), 1038 (M), 1019 (M), 937 (W),

912 (W), 857 (W), 817 (M), 800 (W), 752 (S), 714 (S), 702 (M), 693 (S), 655 (W), 639 (M), 625 (M), 554 (S), 530 (S), 495 (W), 472 (W), 429 (W).

ESI-HRMS (m/z): calc. for  $(C_{17}H_{18}IO_2S^+)$  [M+H]<sup>+</sup> 413.0067; found: 413.0070.

(E)-1-(1-iodo-1-phenylhex-1-en-2-ylsulfonyl)-4-methylbenzene (5c)

Following **GP-3**, (E)-1-(1-iodo-1-phenylhex-1-en-2-ylsulfonyl)-4-methylbenzene (200 mg, 91%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 95-97 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.29-7.25 (m, 2H), 7.19-7.16 (m, 2H), 7.10 (d, *J* = 8.1 Hz, 2H), 7.02 (d, *J* = 6.6 Hz, 2H), 2.97-2.90 (m, 2H), 2.39 (s, 3H), 1.79 (p, *J* = 7.7 Hz, 2H), 1.53 (q, *J* = 7.4 Hz, 2H), 1.03 (t, *J* = 7.3 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.1, 143.7, 142.9, 137.9, 129.3, 128.5, 127.9, 127.6, 115.1, 39.6, 30.4, 22.8, 21.6, 13.8.

IR (v/cm<sup>-1</sup>): 3062 (VW), 2957 (M), 2924 (W), 2864 (W), 1654 (M), 1613 (M), 1596 (M), 1487 (W), 1440 (M), 1375 (W), 1316 (VS), 1292 (M), 1226 (W), 1182 (W), 1148 (VS), 1125 (M), 1084 (S), 1038 (W), 858 (W), 811 (S), 765 (M), 743 (W), 713 (S), 699 (S), 641 (M), 626 (W), 603 (W), 554 (VS), 531 (S), 501 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{22}IO_2S^+)$  [M+H]<sup>+</sup> 441.0380; found: 441.0377.

(E)-1-(1-iodo-1-phenyloct-1-en-2-ylsulfonyl)-4-methylbenzene (5d)

Following **GP-3**, (E)-1-(1-iodo-1-phenyloct-1-en-2-ylsulfonyl)-4-methylbenzene (210 mg, 90%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 90-91 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.29-7.25 (m, 2H), 7.21-7.14 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 2H), 7.02 (dd, *J* = 8.0, 1.6 Hz, 2H), 2.95-2.89 (m, 2H), 2.39 (s, 3H), 1.83-1.74 (m, 2H), 1.53-1.45 (m, 2H), 1.42-1.36 (m, 4H), 0.98-0.93 (m, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 149.2, 143.8, 142.9, 137.9, 129.3, 128.5, 127.9, 127.7, 127.6, 115.1, 39.9, 31.5, 29.3, 28.3, 22.7, 21.6, 14.2.

IR (v/cm<sup>-1</sup>): 3062 (VW), 3051 (VW), 2955 (W), 2921 (W), 2870 (W), 2850 (W), 1649 (W), 1616 (M), 1597 (W), 1494 (VW), 1468 (W), 1440 (W), 1378 (W), 1321 (M), 1305 (W), 1256 (VW), 1209 (VW), 1183 (VW), 1153 (M), 1130 (W), 1085 (M), 1020 (W), 895 (VW), 844

(W), 811 (W), 800 (W), 762 (M), 750 (W), 717 (M), 695 (M), 659 (W), 641 (W), 626 (W), 607
(W), 555 (M), 530 (M), 496 (W), 435 (VW).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{26}IO_2S^+)$  [M+H]<sup>+</sup> 469.0693; found: 469.0696.

(E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5e)

Following **GP-3**, (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (216 mg, 86%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 133-135 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.28 (d, *J* = 8.3 Hz, 2H), 7.15-7.10 (m, 4H), 6.96-6.91 (m, 2H), 2.89-2.82 (m, 2H), 2.39 (s, 3H), 1.72 (q, *J* = 8.1 Hz, 2H), 1.48-1.40 (m, 2H), 1.38-1.31 (m, 4H), 0.94-0.89 (m, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.0, 144.2, 141.3, 137.8, 134.6, 129.4, 129.3, 127.9, 127.7, 113.0, 39.8, 31.4, 29.3, 28.3, 22.7, 21.7, 14.2.

IR (v/cm<sup>-1</sup>): 3064 (W), 3053 (W), 2954 (M), 2926 (M), 2909 (M), 2871 (M), 2848 (M), 1912 (W), 1888 (VW), 1797 (VW), 1641 (W), 1618 (M), 1597 (M), 1586 (M), 1486 (M), 1460 (M), 1398 (M), 1379 (W), 1322 (S), 1302 (M), 1256 (W), 1210 (W), 1183 (M), 1152 (VS), 1132 (S), 1082 (S), 1035 (M), 1015 (M), 896 (VW), 853 (W), 835 (M), 821 (S), 810 (S), 787 (M), 757 (W), 720 (S), 687 (M), 648 (M), 636 (M), 621 (W), 571 (S), 545 (VS), 528 (S), 503 (M), 472 (W), 449 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{25}CIIO_2S^+)$  [M+H]<sup>+</sup> 503.0303; found: 503.0304.

(E)-ethyl 4-(1-iodo-2-tosyloct-1-enyl)benzoate (5f)

$$EtO_2C$$

Following **GP-3**, (E)-ethyl 4-(1-iodo-2-tosyloct-1-enyl)benzoate (267 mg, 99%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 139-140 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89-7.81 (m, 2H), 7.31 (d, J = 8.3 Hz, 2H), 7.10 (t, J = 8.2 Hz, 4H), 4.39 (q, J = 7.1 Hz, 2H), 2.88-2.79 (m, 2H), 2.38 (s, 3H), 1.77-1.68 (m, 2H), 1.46-1.30 (m, 9H), 0.95-0.88 (m, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.9, 149.6, 147.2, 144.4, 137.6, 130.3, 129.5, 128.9, 127.8, 127.7, 113.2, 61.2, 39.8, 31.4, 29.3, 28.2, 22.6, 21.7, 14.4, 14.2.

IR (v/cm<sup>-1</sup>): 3062 (VW), 3044 (VW), 2954 (M), 2931 (M), 2912 (M), 2873 (W), 2859 (M), 2848 (M), 1796 (VW), 1714 (VS), 1674 (W), 1621 (M), 1599 (M), 1568 (W), 1492 (W), 1464 (M), 1448 (W), 1405 (M), 1369 (M), 1322 (S), 1312 (M), 1283 (S), 1271 (VS), 1211 (W),

1181 (S), 1152 (VS), 1131 (M), 1108 (S), 1102 (S), 1082 (S), 1019 (M), 964 (W), 855 (W), 835 (M), 812 (S), 800 (W), 770 (S), 755 (W), 719 (S), 704 (S), 678 (M), 648 (W), 638 (M), 620 (W), 566 (S), 544 (S), 534 (S), 500 (W), 482 (W), 427 (VW).

ESI-HRMS (m/z): calc. for  $(C_{24}H_{30}IO_4S^+)$  [M+H]<sup>+</sup> 541,0904; found: 541,0900.

 $(E) \hbox{-} 1 \hbox{-} fluoro \hbox{-} 4 \hbox{-} (1 \hbox{-} iodo \hbox{-} 2 \hbox{-} tosyloct \hbox{-} 1 \hbox{-} enyl) benzene (\mathbf{5g})$ 



Following **GP-3**, (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (240 mg, 99%) was obtained as a white powder, m.p. (CHCl<sub>3</sub>) 130-132  $^{\circ}$ C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31-7.21 (m, 2H), 7.16-7.07 (m, 2H), 7.03-6.94 (m, 2H), 6.88-6.77 (m, 2H), 2.97-2.78 (m, 2H, -<u>CH<sub>2</sub></u>-C=C), 2.37 (s, 3H, Ar-CH<sub>3</sub>), 1.83-1.68 (m, 2H, -CH<sub>2</sub>-), 1.53-1.25 (m, 6H, -CH<sub>2</sub>-), 1.00-0.85 (m, 3H, -CH<sub>2</sub>-<u>CH3</u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 162.5 (d, J = 250.0 Hz), 150.1, 144.1, 139.0 (d, J = 3.6 Hz), 138.0, 130.1 (d, J = 8.6 Hz), 129.4, 127.6, 114.8 (d, J = 22.0 Hz), 113.6, 40.0, 31.5, 29.4, 28.3, 22.7, 21.7, 14.2.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -111.8 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3069 (W), 3054 (W), 2953 (M), 2909 (M), 2848 (M), 1919 (VW), 1903 (VW), 1875 (VW), 1805 (VW), 1649 (M), 1619 (M), 1595 (S), 1503 (S), 1463 (M), 1403 (W), 1378 (W), 1321 (S), 1305 (M), 1226 (S), 1183 (W), 1151 (VS), 1132 (S), 1085 (S), 1032 (W), 1015 (W), 963 (VW), 897 (VW), 858 (W), 835 (S), 794 (M), 780 (M), 719 (M), 708 (M), 700 (M), 637 (M), 590 (M), 547 (S), 530 (S), 513 (M), 484 (W), 464 (W), 441 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{25}FIO_2S^+)$  [M+H]<sup>+</sup> 487.0598; found: 487.0596.

(E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene (5h)

$$F_3C$$

Following **GP-3**, (E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4methylbenzene (246 mg, 92%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 141-142 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.39 (d, J = 8.1 Hz, 2H), 7.24 (d, J = 8.3 Hz, 2H), 7.15-7.03 (m, 4H), 2.97-2.83 (m, 2H, -<u>CH<sub>2</sub></u>-C=C), 2.37 (s, 3H, -CH<sub>3</sub>), 1.87-1.67 (m, 2H, -CH<sub>2</sub>-), 1.54-1.27 (m, 6H, -CH<sub>2</sub>-), 1.04-0.82 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.7, 146.3, 144.3, 137.6, 130.41 (q, J = 32.7 Hz), 129.6, 128.3, 127.7, 124.72 (q, J = 3.7 Hz), 123.83 (q, J = 272.4 Hz), 111.7, 39.8, 31.5, 29.4, 28.3, 22.7, 21.7, 14.2.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.9 (s, 3F, -CF<sub>3</sub>).

IR (v/cm<sup>-1</sup>): 3058 (W), 2957 (M), 2926 (M), 2910 (M), 2873 (W), 2847 (M), 1934 (VW), 1912 (VW), 1799 (VW), 1622 (M), 1605 (M), 1597 (M), 1576 (VW), 1510 (VW), 1493 (W), 1462 (M), 1446 (W), 1407 (M), 1379 (W), 1322 (VS), 1303 (M), 1256 (VW), 1211 (W), 1190 (W), 1152 (VS), 1127 (VS), 1109 (S), 1082 (S), 1068 (S), 1035 (M), 1019 (M), 898 (VW), 848 (M), 832 (S), 810 (S), 767 (M), 743 (W), 714 (S), 702 (M), 672 (W), 659 (VW), 650 (W), 639 (M), 618 (W), 599 (M), 560 (M), 543 (S), 528 (S), 472 (W), 430 (VW).

ESI-HRMS (m/z): calc. for  $(C_{22}H_{25}F_3IO_2S^+)$  [M+H]<sup>+</sup> 537.0567; found: 537.0565.

(E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (5i)



Following **GP-3**, (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (202 mg, 81%) was obtained as a pale-pink powder, m.p. ( $CH_2Cl_2$ ) 105-106 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.26-7.19 (m, 2H), 7.10-7.01 (m, 3H), 6.70 (ddd, J = 8.4, 2.6, 1.0 Hz, 1H), 6.65 (dt, J = 7.6, 1.3 Hz, 1H), 6.38-6.26 (m, 1H), 3.67 (s, 3H, -OCH<sub>3</sub>), 3.02-2.81 (m, 2H, -CH<sub>2</sub>-), 2.35 (s, 3H,Ar -CH<sub>3</sub>), 1.91-1.66 (m, 2H, -CH<sub>2</sub>-), 1.57-1.28 (m, 6H, -CH<sub>2</sub>-), 1.02-0.84 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 158.6, 149.6, 143.8, 143.7, 138.0, 129.3, 128.7, 127.8, 120.7, 115.1, 114.3, 112.6, 55.1, 39.8, 31.5, 29.4, 28.4, 22.7, 21.6, 14.2.

IR (v/cm<sup>-1</sup>): 3062 (W), 3042 (W), 2952 (M), 2911 (M), 2849 (M), 1909 (VW), 1833 (VW), 1794 (VW), 1749 (VW), 1640 (W), 1619 (M), 1591 (S), 1488 (M), 1468 (M), 1461 (S), 1445 (M), 1422 (M), 1321 (VS), 1302 (M), 1276 (S), 1261 (S), 1252 (S), 1202 (M), 1186 (M), 1180 (M), 1168 (M), 1151 (VS), 1131 (S), 1115 (M), 1085 (S), 1036 (S), 1022 (M), 994 (W), 964 (W), 942 (M), 873 (M), 811 (S), 800 (M), 785 (M), 766 (S), 750 (M), 717 (S), 702 (M), 692 (S), 644 (M), 619 (M), 586 (M), 561 (W), 539 (S), 484 (W), 468 (W).

ESI-HRMS (m/z): calc. for  $(C_{22}H_{28}IO_3S^+)$  [M+H]<sup>+</sup> 499.0798; found: 499.0800.



Following **GP-3**, (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (236 mg, 92%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 137-138 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (d, *J* = 8.1, 1.6 Hz, 1H), 7.60-7.50 (m, 2H), 7.45 (t, *J* = 7.9 Hz, 1H), 7.27 (d, *J* = 7.7 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 2.99-2.78 (m, 2H, -<u>CH<sub>2</sub></u>-C=C), 2.34 (s, 3H, Ar-CH<sub>3</sub>), 1.89-1.63 (m, 2H, -CH<sub>2</sub>-), 1.53-1.27 (m, 6H, -CH<sub>2</sub>-), 1.01-0.84 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 151.6, 147.5, 144.9, 144.3, 137.5, 134.1, 129.8, 129.0, 127.6, 123.3, 122.6, 110.1, 39.7, 31.4, 29.4, 28.3, 22.7, 21.6, 14.2.

IR (v/cm<sup>-1</sup>): 3096 (W), 3079 (W), 2952 (M), 2923 (M), 2867 (W), 2853 (M), 1916 (VW), 1621 (M), 1604 (M), 1594 (M), 1532 (VS), 1491 (W), 1465 (W), 1456 (W), 1428 (W), 1402 (W), 1379 (W), 1352 (S), 1309 (S), 1301 (S), 1293 (S), 1217 (W), 1183 (W), 1145 (VS), 1130 (S), 1081 (S), 1043 (W), 1016 (W), 933 (W), 904 (W), 880 (VW), 833 (W), 809 (S), 738 (M), 717 (S), 683 (M), 654 (W), 636 (W), 619 (W), 570 (M), 546 (M), 529 (M), 479 (W), 452 (VW).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{25}INO_4S^+)$  [M+H]<sup>+</sup> 514.0543; found: 514.0541.

(E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (5k)



Following **GP-3**, (E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (216 mg, 88%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 116-117 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.25-7.20 (m, 2H), 7.18-7.07 (m, 4H), 7.05-6.98 (m, 1H), 6.71-6.65 (m, 1H), 3.01-2.81 (m, 2H, -CH<sub>2</sub>-), 2.39 (s, 3H, Ar-CH<sub>3</sub>), 1.84-1.71 (m, 2H, -CH<sub>2</sub>-), 1.56-1.28 (m, 6H, -CH<sub>2</sub>-), 1.04-0.80 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.7, 144.4, 144.2, 137.8, 133.6, 129.5, 129.1, 128.7, 127.7, 127.6, 126.4, 111.9, 39.8, 31.5, 29.4, 28.4, 22.7, 21.7, 14.2.

IR (v/cm<sup>-1</sup>): 3063 (M), 3040 (W), 2955 (S), 2926 (S), 2868 (M), 2854 (M), 1940 (VW), 1912 (W), 1872 (VW), 1798 (W), 1644 (W), 1610 (M), 1595 (M), 1586 (M), 1562 (M), 1495 (W), 1465 (M), 1460 (M), 1430 (W), 1417 (W), 1405 (M), 1379 (VW), 1309 (S), 1300 (VS),

1288 (S), 1213 (W), 1183 (M), 1146 (VS), 1129 (S), 1082 (VS), 1039 (M), 1015 (M), 989 (W), 964 (W), 915 (M), 901 (M), 876 (M), 810 (S), 785 (S), 757 (W), 727 (S), 709 (S), 692 (S), 661 (M), 638 (M), 620 (W), 566 (S), 550 (M), 528 (S), 503 (M), 476 (W), 438 (W).

ESI-HRMS (m/z): calc. for  $(C_{21}H_{25}CIIO_2S^+)$  [M+H]<sup>+</sup> 503.0303; found: 503.0305.

(E)-3-(1-iodo-2-tosyloct-1-enyl)thiophene (5l)



Following **GP-3**, (E)-3-(1-iodo-2-tosyloct-1-enyl)thiophene (213 mg, 90%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 103-104 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36-7.24 (m, 4H), 7.12 (d, J = 8.1 Hz, 2H), 7.02 (m, 1H), 6.54 (dd, J = 5.1, 1.2 Hz, 1H), 3.08-2.90 (m, 2H, -CH<sub>2</sub>-), 2.40 (s, 3H, Ar-CH<sub>3</sub>), 1.95-1.74 (m, 2H, -CH<sub>2</sub>-), 1.58-1.35 (m, 6H, -CH<sub>2</sub>-), 1.06-0.86 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.8, 143.6, 141.8, 138.1, 129.3, 128.1, 127.3, 126.1, 124.7, 108.6, 39.7, 31.6, 29.4, 28.4, 22.7, 21.7, 14.2.

IR (v/cm<sup>-1</sup>): 3102 (M), 3085 (W), 2955 (S), 2927 (S), 2912 (S), 2870 (M), 2851 (S), 1915 (VW), 1800 (VW), 1648 (W), 1619 (S), 1597 (M), 1511 (W), 1493 (W), 1463 (M), 1447 (M), 1401 (M), 1378 (W), 1354 (VW), 1321 (VS), 1304 (S), 1292 (M), 1254 (W), 1221 (M), 1188 (M), 1150 (VS), 1128 (S), 1083 (S), 1044 (M), 1034 (M), 1019 (M), 994 (W), 949 (M), 867 (W), 837 (S), 813 (S), 791 (S), 762 (M), 746 (S), 737 (S), 723 (S), 710 (S), 699 (S), 656 (S), 632 (M), 613 (S), 540 (S), 524 (S), 480 (W), 437 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{24}IO_2S_2^+)$  [M+H]<sup>+</sup> 475.0257; found: 475.0260.

(E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m)



Following **GP-3**, (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (211 mg, 89%) was obtained as an yellow oil.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32-7.21 (m, 3H), 7.16-7.00 (m, 3H), 6.78 (t, *J* = 4.3 Hz, 1H), 2.98-2.82 (m, 2H, -<u>CH<sub>2</sub></u>-C=C), 2.34 (s, 3H, Ar-CH<sub>3</sub>), 1.85-1.66 (m, 2H, -CH<sub>2</sub>-), 1.52-1.24 (m, 6H, -CH<sub>2</sub>-), 0.98-0.78 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 152.5, 144.6, 143.8, 137.6, 130.1, 129.3, 128.8, 127.6, 126.4, 104.8, 40.3, 31.5, 29.4, 28.3, 22.7, 21.7, 14.2.

IR (v/cm<sup>-1</sup>): 3105 (W), 2959 (W), 2926 (W), 2854 (W), 1636 (M), 1596 (W), 1384 (W), 1309 (W), 1289 (W), 1148 (M), 1082 (W), 858 (VW), 813 (W), 722 (M), 693 (W), 654 (W), 581 (W), 556 (M), 540 (M), 523 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{24}IO_2S_2^+)$  [M+H]<sup>+</sup> 475.0257; found: 475.0256.

(E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n)



Following **GP-3**, (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (207 mg, 79%) was obtained as a yellow powder, m.p. ( $CH_2Cl_2$ ) 111-113 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.81-7.74 (m, 1H), 7.67-7.59 (m, 1H), 7.45-7.34 (m, 3H), 7.30 (d, J = 8.3 Hz, 2H), 6.91 (d, J = 8.1 Hz, 2H), 3.09-2.93 (m, 2H, -<u>CH<sub>2</sub></u>-C=C-), 2.31 (s, 3H, Ar-CH<sub>3</sub>), 1.94-1.77 (m, 2H, -CH<sub>2</sub>-), 1.60-1.34 (m, 6H, -CH<sub>2</sub>-), 1.06-0.92 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 153.4, 144.3, 143.9, 141.2, 138.5, 137.2, 129.2, 128.0, 126.5, 125.6, 125.0, 124.8, 122.0, 104.5, 39.9, 31.5, 29.4, 28.4, 22.7, 21.6, 14.2.

IR (v/cm<sup>-1</sup>): 3065 (W), 2953 (M), 2929 (M), 2907 (M), 2869 (M), 2846 (M), 1908 (VW), 1892 (VW), 1616 (M), 1597 (M), 1506 (W), 1491 (W), 1457 (M), 1429 (M), 1402 (W), 1377 (W), 1323 (VS), 1303 (S), 1249 (W), 1186 (M), 1150 (VS), 1116 (M), 1084 (S), 1036 (M), 1018 (M), 992 (M), 960 (W), 930 (W), 870 (M), 810 (S), 799 (M), 765 (M), 751 (S), 725 (S), 712 (S), 699 (S), 677 (M), 646 (M), 632 (W), 596 (M), 568 (S), 547 (S), 529 (S), 511 (M), 493 (M), 477 (W), 427 (M).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{26}IO_2S_2^+)$  [M+H]<sup>+</sup> 525.0413; found: 525.0417.

(E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a)

Following **GP-3**, (E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (142 mg, 84%) was obtained as an yellow oil.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.79-7.71 (m, 2H), 7.39-7.31 (m, 2H), 3.16 (q, *J* = 1.6 Hz, 3H, -C=C-CH<sub>3</sub>), 2.44 (s, 3H, Ar-CH<sub>3</sub>), 2.26 (q, *J* = 1.6 Hz, 3H, -C=C-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.7, 140.6, 137.8, 130.1, 127.5, 117.9, 33.0, 27.9, 21.8.

IR (v/cm<sup>-1</sup>): 3050 (W), 2922 (W), 1911 (W), 1646 (M), 1610 (S), 1598 (S), 1494 (M), 1435 (M), 1400 (M), 1377 (M), 1314 (S), 1304 (VS), 1290 (S), 1160 (VS), 1129 (S), 1080 (S),

1041 (M), 1018 (M), 908 (M), 810 (S), 729 (W), 709 (S), 687 (VS), 636 (S), 573 (M), 557 (S), 531 (S), 493 (M), 466 (M), 425 (W).

ESI-HRMS (m/z): calc. for  $(C_{11}H_{14}IO_2S^+)$  [M+H]<sup>+</sup> 336.9754; found: 336.9752.

(Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a')

$$H_3C$$
  $Ts$   $H_3C$   $H_$ 

Following **GP-3**, (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (19 mg, 12%) was obtained as an yellow oil.

<sup>1</sup>H NMR (400 MHz, CDCl3) δ 7.91-7.81 (m, 2H), 7.38-7.29 (m, 2H), 2.74 (q, *J* = 1.2 Hz, 3H, -C=C-CH<sub>3</sub>), 2.44 (s, 3H, Ar-CH<sub>3</sub>), 2.18 (q, *J* = 1.2 Hz, 3H, -C=C-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.6, 141.9, 136.9, 129.7, 128.4, 102.9, 35.7, 21.8, 17.0.

IR (v/cm<sup>-1</sup>): 3064 (W), 2962 (W), 2925 (W), 2853 (VW), 1918 (VW), 1721 (VW), 1651 (VW), 1593 (M), 1492 (W), 1449 (M), 1399 (M), 1379 (M), 1312 (VS), 1303 (VS), 1185 (M), 1163 (S), 1126 (S), 1082 (S), 1018 (M), 930 (M), 809 (S), 729 (S), 656 (VS), 578 (VS), 560 (VS), 493 (M), 457 (M).

ESI-HRMS (m/z): calc. for  $(C_{11}H_{14}IO_2S^+)$  [M+H]<sup>+</sup> 336.9754; found: 336.9757

(E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b)



Following **GP-3**, (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (149 mg, 82%) was obtained as a white powder, m.p. (n-Hexane) 80-82 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.21 (q, *J* = 7.2 Hz, 2H, -<u>CH<sub>2</sub></u>-CH<sub>3</sub>), 2.66 (q, *J* = 7.4 Hz, 2H, -<u>CH<sub>2</sub></u>-CH<sub>3</sub>), 2.43 (s, 3H, Ar-CH<sub>3</sub>), 1.10 (t, *J* = 7.4 Hz, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub>), 1.04 (t, *J* = 7.2 Hz, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub>)</u>.</u>

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.2, 144.6, 138.6, 130.0, 129.1, 127.4, 37.6, 34.2, 21.8, 14.9, 12.8.

IR (v/cm<sup>-1</sup>): 2979 (M), 2929 (M), 2874 (W), 2857 (W), 1923 (W), 1811 (VW), 1654 (M), 1599 (S), 1490 (M), 1456 (M), 1437 (M), 1374 (M), 1316 (S), 1290 (S), 1257 (M), 1212 (M), 1182 (M), 1159 (VS), 1135 (S), 1077 (S), 1044 (M), 1014 (M), 1000 (M), 970 (W), 928 (M), 908 (M), 817 (S), 805 (M), 797 (M), 770 (M), 707 (S), 692 (VS), 644 (S), 613 (M), 540 (S), 523 (S), 486 (M), 457 (W), 417 (W).

ESI-HRMS (m/z): calc. for  $(C_{13}H_{18}IO_2S^+)$  [M+H]<sup>+</sup> 365.0067; found: 365.0067.

(E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (7c)

Following **GP-3**, (E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (158 mg, 81%) was obtained as pale yellow oil. The NMR data are in agreement with previously reported<sup>20</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.79-7.71 (m, 2H), 7.38-7.30 (m, 2H), 3.20-3.07 (m, 2H, -CH<sub>2</sub>-C=C-), 2.65-2.53 (m, 2H, -CH<sub>2</sub>-C=C-), 2.44 (s, 3H, Ar-CH<sub>3</sub>), 1.67-1.42 (m, 4H, -CH<sub>2</sub>-), 0.97-0.86 (m, 6H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.8, 144.5, 138.8, 130.0, 128.0, 127.5, 45.2, 42.6, 24.0, 22.0, 21.8, 14.2, 13.1.

IR (v/cm<sup>-1</sup>): 3062 (W), 3030 (W), 2962 (VS), 2931 (S), 2872 (S), 1917 (VW), 1596 (S), 1494 (M), 1463 (S), 1402 (M), 1379 (M), 1316 (VS), 1303 (S), 1237 (M), 1181 (M), 1154 (VS), 1135 (VS), 1118 (S), 1083 (S), 1039 (W), 1017 (M), 986 (M), 971 (M), 919 (W), 894 (W), 813 (S), 780 (W), 712 (S), 687 (S), 629 (M), 585 (M), 573 (M), 547 (S), 528 (S), 508 (W), 475 (W).

ESI-HRMS (m/z): calc. for  $(C_{15}H_{22}IO_2S^+)$  [M+H]<sup>+</sup> 393.0380; found: 393.0385.

(*E*)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (7*d*)



Following **GP-3**, (E)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (203 mg, 89%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 100-102 °C. The NMR data are in agreement with previously reported<sup>16</sup>.

<sup>1</sup>H NMR (400 MHz,CDCl<sub>3</sub>) δ 7.45-7.22 (m, 6H), 7.22-6.98 (m, 4H), 4.46 (q, *J* = 7.1 Hz, 2H, -<u>CH<sub>2</sub></u>-CH<sub>3</sub>), 2.41 (s, 3H, Ar-CH<sub>3</sub>), 1.46 (t, *J* = 7.1 Hz, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 163.8, 146.7, 145.0, 139.8, 137.4, 129.7, 129.5, 128.4, 127.9, 127.5, 114.2, 63.4, 21.8, 14.1.

IR (v/cm<sup>-1</sup>): 3096 (VW), 3072 (VW), 3044 (VW), 2986 (W), 2925 (W), 2904 (W), 2868 (W), 1982 (VW), 1957 (VW), 1929 (VW), 1883 (VW), 1810 (VW), 1718 (VS), 1677 (W), 1604 (M), 1591 (M), 1486 (M), 1473 (W), 1442 (M), 1397 (W), 1365 (W), 1326 (S), 1300 (M), 1289 (M), 1250 (S), 1201 (S), 1155 (S), 1115 (W), 1085 (S), 1036 (S), 1006 (M), 917 (W), 889 (W),

860 (W), 838 (VW), 818 (M), 801 (W), 787 (M), 771 (W), 737 (M), 704 (M), 691 (S), 654 (W), 627 (W), 613 (W), 586 (S), 547 (S), 540 (S), 510 (M), 479 (W), 434 (VW).

ESI-HRMS (m/z): calc. for  $(C_{18}H_{18}IO_4S^+)$  [M+H]<sup>+</sup> 456.9965; found: 456.9964.

(E)-3-iodo-1,3-diphenyl-2-tosylprop-2-en-1-one (7e)



Following **GP-3**, (E)-3-iodo-1,3-diphenyl-2-tosylprop-2-en-1-one (210 mg, 86%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 140-141 °C. The NMR data are in agreement with previously reported<sup>21</sup>.

<sup>1</sup>H NMR (400 MHz,CDCl<sub>3</sub>) δ 8.30-8.18 (m, 2H), 7.76-7.65 (m, 1H), 7.65-7.54 (m, 2H), 7.39-7.23 (m, 5H), 7.23-7.15 (m, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 2.39 (s, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 190.8, 149.5, 145.0, 140.2, 137.5, 134.8, 134.0, 130.4, 129.7, 129.5, 129.3, 128.6, 128.1, 127.6, 113.9, 21.8.

IR (v/cm<sup>-1</sup>): 3051 (W), 2923 (VW), 1674 (S), 1610 (M), 1596 (S), 1581 (M), 1488 (W), 1444 (W), 1401 (W), 1326 (S), 1308 (M), 1291 (M), 1246 (S), 1198 (M), 1188 (W), 1173 (M), 1153 (VS), 1085 (S), 1054 (M), 1024 (M), 1017 (W), 1000 (W), 898 (M), 813 (M), 797 (W), 759 (S), 720 (W), 698 (S), 684 (S), 627 (W), 608 (W), 596 (W), 571 (M), 545 (S), 515 (M), 480 (W), 427 (VW).

(E)-2-iodo-1-phenyl-3-tosylhex-2-en-1-one (7f)



Following **GP-3**, (E)-2-iodo-1-phenyl-3-tosylhex-2-en-1-one (143 mg, 63%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 124-125 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.01-7.93 (m, 2H), 7.86-7.77 (m, 2H), 7.63-7.55 (m, 1H), 7.55-7.47 (m, 2H), 7.40-7.29 (m, 2H), 2.70-2.46 (m, 2H, -CH<sub>2</sub>-), 2.44 (s, 3H, Ar-CH<sub>3</sub>), 1.94-1.35 (m, 2H, -CH<sub>2</sub>-), 0.99 (t, J = 7.3 Hz, 3H, -CH<sub>2</sub>-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 190.8, 147.5, 145.4, 135.5, 133.9, 133.0, 130.2, 129.8, 128.9, 128.8, 113.4, 38.5, 21.8, 21.4, 14.3.

IR (v/cm<sup>-1</sup>): 1674 (M), 1634 (M), 1447 (VW), 1319 (W), 1230 (W), 1155 (W), 1085 (VW), 1007 (VW), 814 (W), 722 (W), 679 (W), 654 (W), 543 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{20}IO_3S^+)$  [M+H]<sup>+</sup> 455.0172, found: 455.0170.

(E)-3-iodo-3-phenyl-2-tosylprop-2-en-1-ol (7g)



Following **GP-3**, (E)-3-iodo-3-phenyl-2-tosylprop-2-en-1-ol (149 mg, 72%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 117-118 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.30-7.24 (m, 2H), 7.23-7.11 (m, 3H), 7.11-7.04 (m, 2H), 7.00-6.94 (m, 2H), 4.93 (s, 2H, -<u>CH<sub>2</sub></u>-OH), 2.36 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 147.6, 144.4, 142.0, 137.2, 129.5, 129.1, 127.8, 127.6, 119.4, 21.7.

IR (v/cm<sup>-1</sup>): 3047 (W), 2970 (VW), 2927 (VW), 2885 (W), 1889 (VW), 1611 (M), 1591 (M), 1489 (W), 1464 (W), 1440 (M), 1401 (M), 1380 (W), 1316 (S), 1303 (M), 1236 (W), 1183 (W), 1148 (S), 1095 (M), 1072 (M), 1036 (S), 1014 (M), 960 (M), 839 (M), 810 (M), 760 (S), 694 (S), 679 (M), 638 (M), 608 (W), 553 (M), 543 (S), 516 (M), 495 (M), 453 (W).

ESI-HRMS (m/z): calc. for  $(C_{16}H_{16}IO_3S^+)$  [M+H]<sup>+</sup> 414.9859; found: 414.9856.

(E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (7h)



Following **GP-3**, (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (204 mg, 89%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 156-158 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.11-8.01 (m, 2H), 7.42-7.32 (m, 2H), 7.22-7.12 (m, 4H), 4.88 (s, 2H, -<u>CH</u><sub>2</sub>-OH), 2.45 (brs, -OH), 2.41 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.8, 148.2, 147.7, 145.5, 136.8, 129.9, 128.4, 127.9, 123.1, 115.1, 68.8, 21.8.

IR (v/cm<sup>-1</sup>): 3105 (VW), 3064 (W), 3025 (VW), 2929 (W), 2852 (VW), 1934 (VW), 1660 (VW), 1611 (M), 1599 (M), 1592 (M), 1586 (M), 1511 (VS), 1489 (M), 1446 (W), 1404 (W), 1378 (M), 1351 (VS), 1297 (S), 1288 (S), 1224 (M), 1185 (M), 1130 (VS), 1084 (M), 1073 (S), 1019 (S), 1009 (S), 965 (W), 875 (W), 860 (M), 828 (M), 821 (S), 800 (W), 755 (M), 719 (S), 698 (M), 676 (M), 666 (M), 636 (W), 616 (W), 574 (M), 545 (S), 509 (M), 477 (W), 462 (W), 435 (W).

ESI-HRMS (m/z): calc. for  $(C_{16}H_{13}INO_4S^+)$  [M]<sup>+</sup> 441.9604, found: 441.9607.



Following **GP-3**, (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (203 mg, 78%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 161-163 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.66-7.51 (m, 2H), 7.24-6.71 (m, 11H), 6.32 (d, *J* = 12.0 Hz, 1H), 4.69 (d, *J* = 12.0 Hz, 1H), 3.86 (s, 3H, -OCH<sub>3</sub>), 2.33 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 159.5, 149.6, 144.1, 141.9, 138.0, 132.1, 129.1, 129.0, 127.7, 127.6, 126.8, 119.2, 114.2, 83.2, 55.5, 21.7.

IR (v/cm<sup>-1</sup>): 3083 (VW), 3062 (W), 3033 (W), 2961 (W), 2916 (W), 2837 (W), 1946 (VW), 1886 (W), 1654 (W), 1612 (S), 1595 (M), 1581 (S), 1509 (VS), 1487 (M), 1462 (M), 1442 (M), 1412 (M), 1400 (M), 1383 (W), 1318 (M), 1304 (S), 1293 (M), 1280 (S), 1251 (VS), 1188 (M), 1173 (S), 1132 (S), 1079 (VS), 1047 (S), 1020 (S), 1001 (M), 956 (W), 918 (W), 876 (W), 840 (M), 823 (S), 812 (M), 799 (W), 775 (S), 765 (M), 721 (S), 708 (M), 693 (S), 682 (S), 638 (M), 624 (M), 593 (S), 557 (S), 537 (M), 530 (VS), 514 (M), 488 (M), 467 (M), 462 (M), 424 (VW).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{20}IO_3S^+)$  [M]<sup>+</sup> 503.0172; found: 503.0173.

(E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (7j)



Following **GP-3**, (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (200 mg, 85%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 148-150 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.21-7.08 (m, 4H), 7.08-6.83 (m, 3H), 6.63 (s, 1H), 5.20-4.98 (m, 1H), 3.90 (d, *J* = 10.5 Hz, 1H), 2.34 (s, 3H), 2.25 (m, 1H, -<u>CH</u><sub>2</sub>-CH-OH), 2.15-1.97 (m, 1H, -<u>CH</u>-CH<sub>3</sub>), 1.84 (m, 1H, -<u>CH</u><sub>2</sub>-CH-OH), 1.16-1.03 (m, 6H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.1, 143.9, 141.9, 138.3, 129.2, 128.8, 127.6, 127.3, 116.1, 81.7, 45.2, 25.1, 23.9, 21.9, 21.7.

IR (v/cm<sup>-1</sup>): 3061 (W), 3052 (W), 2949 (M), 2923 (M), 2899 (W), 2865 (M), 1956 (VW), 1895 (VW), 1810 (VW), 1653 (VW), 1603 (M), 1595 (M), 1585 (M), 1490 (M), 1465 (W), 1443

(M), 1405 (M), 1379 (W), 1365 (W), 1346 (W), 1321 (S), 1304 (M), 1296 (S), 1287 (S), 1250
(W), 1213 (W), 1195 (W), 1185 (M), 1145 (VS), 1111 (M), 1090 (S), 1056 (M), 1032 (M), 1017
(M), 981 (M), 956 (W), 923 (W), 853 (W), 835 (M), 814 (S), 795 (W), 779 (VW), 767 (M), 708
(S), 699 (VS), 666 (M), 654 (S), 628 (W), 593 (S), 561 (M), 535 (S), 515 (M), 484 (M), 412
(VW).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{22}IO_2S^+)$  [M]<sup>+</sup> 453.0380, found: 453.0381.

(E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k)



Following **GP-3**, (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (mg, 89%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  183-185 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.27-8.14 (m, 2H), 7.40-7.32 (m, 2H), 7.32-7.23 (m, 4H), 7.21-7.15 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.10-7.03 (m, 2H), 3.93 (s, 3H, -OCH<sub>3</sub>), 2.38 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 189.3, 165.0, 149.7, 144.9, 140.4, 137.6, 132.9, 129.6, 129.5, 128.6, 128.0, 127.6, 127.1, 114.7, 113.5, 55.8, 21.8.

IR (v/cm<sup>-1</sup>): 3045 (W), 3017 (VW), 2974 (W), 2922 (W), 2846 (W), 2583 (VW), 1924 (VW), 1891 (VW), 1808 (VW), 1767 (VW), 1662 (S), 1611 (S), 1591 (VS), 1511 (S), 1488 (M), 1464 (M), 1446 (M), 1422 (M), 1401 (W), 1319 (S), 1309 (S), 1296 (M), 1271 (S), 1258 (VS), 1200 (M), 1185 (M), 1165 (VS), 1148 (VS), 1118 (M), 1084 (S), 1053 (M), 1017 (S), 979 (W), 946 (W), 905 (M), 835 (S), 816 (M), 797 (W), 778 (S), 744 (M), 713 (M), 692 (S), 648 (W), 629 (W), 619 (S), 564 (S), 540 (S), 524 (S), 508 (W), 495 (W), 479 (M), 421 (VW).

ESI-HRMS (m/z): calc. for  $(C_{23}H_{20}IO_4S^+)$  [M+H]<sup>+</sup> 519.0121; found: 519.0125.

(E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (7l)



Following **GP-3**, (E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (222 mg, 90%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 141-143 °C.
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.07 (dd, *J* = 3.9, 1.2 Hz, 1H), 7.85 (dd, *J* = 4.9, 1.2 Hz, 1H), 7.37-7.32 (m, 2H), 7.20-7.14 (m, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 2.38 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 182.9, 149.6, 145.0, 141.2, 140.2, 137.4, 136.9, 136.5, 129.7, 129.5, 129.0, 128.6, 128.0, 127.5, 114.6, 21.8.

IR (v/cm<sup>-1</sup>): 3095 (VW), 3057 (VW), 2921 (VW), 1647 (S), 1607 (M), 1588 (M), 1511 (W), 1486 (W), 1442 (W), 1407 (S), 1353 (M), 1319 (S), 1304 (M), 1294 (M), 1263 (S), 1201 (W), 1185 (W), 1149 (VS), 1083 (S), 1061 (M), 1021 (M), 999 (W), 887 (W), 859 (W), 813 (M), 775 (M), 731 (M), 704 (S), 694 (S), 666 (M), 610 (W), 573 (M), 563 (S), 543 (S), 534 (S), 505 (M), 478 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{16}IO_3S_2^+)$  [M+H]<sup>+</sup> 494.9580; found: 494.9575.

(E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9a)



Following **GP-3**, (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (230 mg, 99%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 202-203 °C (lit. data<sup>18</sup>: m.p. 205-206 °C). The NMR data are in agreement with previously reported<sup>18</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.49-7.28 (m, 10H), 7.23-7.13 (m, 2H), 7.03-6.88 (m, 2H).

 $^{13}$ C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.54 (d, J = 256.3 Hz), 148.9, 142.4, 139.2, 135.90 (d, J = 3.1 Hz), 131.37 (d, J = 9.6 Hz), 130.4, 129.5, 129.3, 128.6, 128.1, 127.5, 118.5, 115.98 (d, J = 22.7 Hz).

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -103.6 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3103 (VW), 3055 (W), 1621 (W), 1591 (S), 1492 (S), 1445 (M), 1405 (W), 1326 (S), 1299 (W), 1291 (M), 1277 (VW), 1235 (M), 1176 (M), 1163 (M), 1148 (VS), 1097 (W), 1084 (S), 1029 (W), 1013 (W), 1000 (VW), 951 (W), 908 (W), 845 (M), 834 (M), 818 (W), 782 (M), 752 (VW), 717 (W), 697 (S), 669 (W), 655 (W), 631 (W), 605 (W), 582 (S), 558 (S), 529 (S), 510 (W), 501 (W), 495 (W), 445 (VW).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{15}FIO_2S^+)$  [M+H]<sup>+</sup> 464.9816; found: 464.9811.



Following **GP-3**, (E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (250 mg, 99%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 106-107 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.43-7.30 (m, 2H), 7.21-7.09 (m, 2H), 7.08-6.90 (m, 3H), 6.78 (s, 1H), 3.15-2.74 (m, 2H, -CH<sub>2</sub>-), 1.77 (p, *J* = 7.7 Hz, 2H, -CH<sub>2</sub>-), 1.60-1.31 (m, 6H, -CH<sub>2</sub>-), 1.03-0.79 (m, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.48 (d, J = 256.8 Hz), 150.4, 144.1, 136.77 (d, J = 3.3 Hz), 133.7, 130.42 (d, J = 9.7 Hz), 129.2, 128.9, 127.7, 126.4, 116.20 (d, J = 22.7 Hz), 112.4, 39.8, 31.5, 29.4, 28.4, 22.7, 14.2.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -103.7 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3103 (VW) 3067 (W), 2957 (M), 2927 (M), 2913 (M), 2871 (M), 2854 (M), 1901 (VW) 1770 (VW) 1609 (W), 1599 (M), 1591 (S), 1560 (M), 1495 (S), 1472 (W), 1466 (M), 1461 (M), 1406 (M), 1307 (S), 1289 (VS), 1266 (W), 1235 (S), 1213 (W), 1191 (W), 1158 (S), 1145 (VS), 1129 (S), 1097 (M), 1091 (M), 1081 (VS), 1053 (M), 1037 (W), 1012 (W), 913 (W), 900 (M), 874 (W), 833 (S), 816 (M), 787 (S), 742 (W), 728 (S), 714 (S), 698 (M), 692 (S), 661 (W), 638 (W), 566 (S), 550 (M), 530 (S), 505 (M), 485 (W), 442 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{22}ClFIO_2S^+)$  [M+H]<sup>+</sup> 507.0052; found: 507.0052.

(E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9c)



Following **GP-3**, (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (140 mg, 76%) was obtained as a white off oil.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.94-7.83 (m, 2H), 7.30-7.14 (m, 3H), 3.22 (q, *J* = 7.3 Hz, 2H, -CH<sub>2</sub>-), 2.65 (q, *J* = 7.4 Hz, 2H, -CH<sub>2</sub>-), 1.11 (t, *J* = 7.4 Hz, 3H, -CH<sub>3</sub>), 1.06 (t, *J* = 7.2 Hz, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.66 (d, J = 256.5 Hz), 144.7, 137.57 (d, J = 3.2 Hz), 130.23 (d, J = 9.5 Hz), 129.7, 116.78 (d, J = 22.7 Hz), 37.7, 34.3, 15.0, 12.8. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -103.5 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3104 (W), 3075 (W), 2974 (S), 2936 (M), 2876 (M), 1591 (VS), 1494 (S), 1459 (M), 1456 (M), 1404 (M), 1372 (M), 1359 (M), 1324 (S), 1291 (VS), 1261 (M), 1238 (S), 1185 (S), 1160 (VS), 1152 (VS), 1132 (VS), 1097 (M), 1083 (S), 1077 (S), 1012 (M), 1000 (M), 926 (S), 909 (M), 839 (S), 820 (M), 714 (S), 696 (S), 645 (W), 573 (M), 553 (S), 545 (S), 526 (S), 493 (M).

ESI-HRMS (m/z): calc. for  $(C_{12}H_{15}FIO_2S^+)$  [M+H]<sup>+</sup> 368.9816; found: 368.9818.

(*E*)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9d)



Following **GP-3**, (E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (224 mg, 90%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  138-140 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (d, *J* = 3.8 Hz, 1H), 7.84 (d, *J* = 4.9 Hz, 1H), 7.44 (dd, *J* = 8.7, 5.0 Hz, 2H), 7.35-7.20 (m, 5H), 7.12 (d, *J* = 6.4 Hz, 2H), 6.96 (t, *J* = 8.5 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 182.7, 165.87 (d, J = 257.3 Hz), 149.3, 141.1, 140.0, 137.2, 136.5, 136.30 (d, J = 3.0 Hz), 131.40 (d, J = 9.9 Hz), 129.9, 129.1, 128.2, 127.5, 116.21 (d, J = 22.8 Hz), 115.2.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -102.6 (s, 1F, Ar-F).

IR (u/cm<sup>-1</sup>): 3110 (M), 3103 (W), 3076 (W), 3052 (W), 3030 (W), 1889 (W), 1774 (VW), 1646 (VS), 1601 (S), 1586 (VS), 1510 (S), 1493 (VS), 1485 (S), 1440 (M), 1409 (VS), 1354 (S), 1323 (VS), 1294 (S), 1265 (S), 1235 (VS), 1203 (M), 1192 (W), 1165 (M), 1162 (M), 1143 (VS), 1082 (S), 1062 (S), 1029 (W), 1019 (S), 1001 (M), 921 (W), 892 (M), 861 (M), 856 (M), 834 (S), 812 (M), 777 (S), 752 (VS), 738 (S), 707 (S), 695 (S), 666 (S), 647 (W), 628 (W), 624 (W), 608 (M), 573 (S), 563 (S), 543 (VS), 533 (S), 508 (M), 487 (S), 467 (M), 446 (M), 422 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{13}FIO_3S_2^+)$  [M+H]<sup>+</sup> 498.9329; found: 498.9328.



Following **GP-3**, (E)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (237 mg, 99%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 195-196 °C (lit. data<sup>18</sup>: m.p. 197-199 °C). The NMR data are in agreement with previously reported<sup>18</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36-7.14 (m, 12H), 7.13-7.05 (m, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.8, 142.4, 140.1, 139.0, 138.4, 131.7, 130.4, 130.0, 129.6, 129.3, 129.0, 128.7, 128.1, 127.5, 118.9.

IR (v/cm<sup>-1</sup>): 3061 (VW) 3049 (W), 2958 (VW) 2927 (VW) 1619 (W), 1588 (W), 1577 (M), 1493 (W), 1474 (M), 1447 (M), 1394 (W), 1327 (VS), 1311 (W), 1294 (W), 1282 (M), 1235 (W), 1175 (M), 1161 (M), 1150 (S), 1092 (S), 1085 (M), 1017 (M), 1001 (W), 951 (W), 906 (W), 823 (M), 784 (S), 757 (S), 702 (S), 697 (S), 662 (W), 656 (W), 612 (M), 584 (S), 574 (M), 549 (S), 470 (W), 443 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{15}CIIO_2S^+)$  [M+H]<sup>+</sup> 480.9520; found: 480.9518.

(E)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9f)



Following **GP-3**, (E)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (251 mg, 96%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  134-136 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32-7.20 (m, 4H), 7.18-7.07 (m, 2H), 7.01-6.88 (m, 1H), 6.80-6.67 (m, 1H), 3.00-2.81 (m, 2H, -CH<sub>2</sub>-), 1.76 (p, *J* = 7.6 Hz, 2H, -CH<sub>2</sub>-), 1.53-1.30 (m, 6H, -CH<sub>2</sub>-), 1.00-0.81 (m, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.4, 144.0, 140.1, 139.2, 133.8, 129.2, 129.2, 129.0, 128.9, 127.8, 126.4, 112.5, 39.7, 31.5, 29.3, 28.4, 22.7, 14.2.

IR (v/cm<sup>-1</sup>): 3067 (W), 2957 (M), 2926 (S), 2871 (M), 2855 (M), 1908 (VW), 1645 (VW), 1609 (W), 1581 (M), 1561 (M), 1475 (M), 1467 (M), 1460 (M), 1430 (W), 1418 (W), 1407 (W), 1394 (M), 1304 (VS), 1294 (S), 1276 (S), 1175 (W), 1146 (VS), 1129 (S), 1089 (VS),

1081 (VS), 1053 (W), 1039 (W), 1011 (M), 989 (W), 914 (W), 900 (M), 875 (W), 822 (S), 787 (M), 762 (S), 722 (M), 709 (S), 692 (M), 686 (S), 661 (W), 631 (W), 624 (W), 590 (M), 561 (S), 523 (M), 500 (W), 464 (M), 444 (W), 439 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{22}Cl_2IO_2S^+)$  [M+H]<sup>+</sup> 522.9757; found: 522.9753.

(E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9g)



Following **GP-3**, (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (154 mg, 80%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  84-85 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.85-7.78 (m, 2H), 7.56-7.49 (m, 2H), 3.21 (q, *J* = 7.2 Hz, 2H, -CH<sub>2</sub>-), 2.66 (q, *J* = 7.4 Hz, 2H, -CH<sub>2</sub>-), 1.12 (t, *J* = 7.4 Hz, 3H, -CH<sub>3</sub>), 1.07 (t, *J* = 7.2 Hz, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.5, 140.3, 140.0, 130.1, 129.8, 128.8, 37.7, 34.3, 15.0, 12.9.

IR (v/cm<sup>-1</sup>): 3093 (W), 2977 (M), 2932 (M), 2871 (M), 1912 (W), 1647 (VW), 1602 (S), 1581 (M), 1573 (M), 1476 (S), 1457 (S), 1434 (M), 1393 (S), 1369 (M), 1359 (M), 1303 (VS), 1280 (S), 1258 (M), 1187 (M), 1176 (M), 1155 (VS), 1130 (S), 1103 (M), 1090 (VS), 1077 (S), 1048 (M), 1012 (S), 1002 (M), 928 (S), 912 (M), 835 (S), 824 (S), 800 (M), 768 (M), 758 (VS), 708 (S), 676 (S), 646 (M), 634 (M), 624 (M), 596 (W), 583 (W), 572 (M), 561 (M), 533 (S), 510 (M), 488 (W), 469 (S), 406 (W).

ESI-HRMS (m/z): calc. for  $(C_{12}H_{15}CIIO_2S^+)$  [M+H]<sup>+</sup> 384.9520; found: 384.9520.

(E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9h)



Following **GP-3**, (E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (216 mg, 84%) was obtained as a white powder, m.p.  $(CH_2Cl_2)$  137-138 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.03 (d, *J* = 3.8 Hz, 1H), 7.83 (d, *J* = 4.9 Hz, 1H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.32-7.18 (m, 6H), 7.10 (d, *J* = 7.2 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 182.6, 149.1, 141.1, 140.7, 140.0, 138.8, 137.3, 136.6, 130.0, 129.9, 129.2, 129.1, 128.2, 127.5, 115.6.

IR (v/cm<sup>-1</sup>): 3101 (VW), 3088 (W), 3080 (W), 1649 (S), 1608 (M), 1588 (M), 1578 (M), 1510 (M), 1486 (W), 1473 (M), 1444 (M), 1406 (S), 1394 (M), 1350 (S), 1323 (VS), 1277 (M), 1266 (S), 1260 (S), 1243 (M), 1226 (M), 1202 (M), 1174 (M), 1151 (VS), 1112 (W), 1092 (S), 1085 (S), 1057 (S), 1019 (M), 1009 (S), 999 (M), 966 (W), 949 (W), 918 (W), 901 (W), 887 (M), 854 (M), 830 (S), 782 (S), 760 (S), 747 (M), 731 (S), 703 (M), 695 (S), 682 (M), 661 (M), 642 (W), 626 (W), 619 (M), 569 (S), 561 (S), 540 (S), 491 (W), 465 (M), 437 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{13}CIIO_3S_2^+)$  [M+H]<sup>+</sup> 514.9034; found: 514.9031.

(E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9i)



Following **GP-3**, (E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (259 mg, 99%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 210-211 °C (lit. data<sup>18</sup>: m.p. 186-188 °C). The NMR data are in agreement with previously reported<sup>18</sup>.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.50-7.30 (m, 10H), 7.25-7.17 (m, 4H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 148.8, 142.4, 139.0, 139.0, 132.0, 130.4, 130.0, 129.6, 129.4, 128.7, 128.7, 128.1, 127.5, 118.9.

IR (v/cm<sup>-1</sup>): 3059 (W), 3049 (W), 1901 (VW), 1619 (M), 1587 (W), 1573 (S), 1492 (W), 1468 (M), 1447 (M), 1389 (M), 1327 (VS), 1310 (M), 1294 (W), 1281 (M), 1233 (W), 1175 (M), 1161 (M), 1148 (VS), 1082 (S), 1068 (M), 1030 (W), 1027 (W), 1013 (S), 1001 (W), 951 (M), 907 (W), 862 (VW), 839 (VW), 818 (S), 783 (S), 744 (VS), 701 (S), 696 (S), 658 (M), 620 (VW), 611 (M), 583 (VS), 567 (S), 545 (S), 482 (VW), 409 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{15}BrIO_2S^+)$  [M+H]<sup>+</sup> 524.9015; found: 524.9016.

(E)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (9j)



Following **GP-3**, (E)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (266 mg, 94%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 144-145 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.49-7.41 (m, 2H), 7.23-7.10 (m, 4H), 6.96 (dt, J = 6.7, 1.8 Hz, 1H), 6.79-6.73 (m, 1H), 3.04-2.78 (m, 2H, -CH<sub>2</sub>-), 1.77 (p, J = 7.6 Hz, 2H, -CH<sub>2</sub>-), 1.54-1.30 (m, 6H, -CH<sub>2</sub>-), 0.98-0.89 (m, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.4, 143.9, 139.8, 133.8, 132.2, 129.2, 129.0, 128.9, 128.6, 127.8, 126.4, 112.6, 39.7, 31.5, 29.4, 28.4, 22.7, 14.2.

IR (v/cm<sup>-1</sup>): 3066 (W), 2955 (M), 2925 (M), 2869 (W), 2853 (M), 1609 (W), 1585 (W), 1572 (M), 1467 (M), 1460 (M), 1389 (M), 1304 (VS), 1293 (M), 1274 (M), 1177 (W), 1146 (VS), 1128 (M), 1091 (W), 1080 (VS), 1066 (M), 1008 (M), 900 (W), 818 (M), 787 (M), 760 (M), 752 (S), 721 (W), 705 (M), 692 (M), 680 (M), 581 (M), 561 (M), 528 (W), 517 (M), 499 (W), 438 (VW), 411 (W).

ESI-HRMS (m/z): calc. for  $(C_{20}H_{22}BrClIO_2S^+)$  [M+H]<sup>+</sup> 566.9252; found: 566.9248.

(E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9k)



Following **GP-3**, (E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (169 mg, 79%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 96-97 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.76-7.66 (m, 4H), 3.20 (q, *J* = 7.2 Hz, 2H, -CH<sub>2</sub>-), 2.65 (q, *J* = 7.4 Hz, 2H, -CH<sub>2</sub>-), 1.12 (t, *J* = 7.4 Hz, 3H, -CH<sub>3</sub>), 1.08 (t, *J* = 7.2 Hz, 3H, -CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.5, 140.6, 132.8, 130.1, 128.9, 128.8, 37.7, 34.3, 15.0, 12.9.

IR (v/cm<sup>-1</sup>): 3090 (W), 2974 (M), 2967 (M), 2929 (M), 2869 (M), 1911 (W), 1644 (VW), 1602 (S), 1573 (S), 1472 (M), 1455 (S), 1434 (M), 1388 (S), 1369 (M), 1349 (W), 1302 (VS), 1278 (S), 1258 (M), 1178 (M), 1154 (VS), 1129 (VS), 1113 (M), 1107 (M), 1076 (VS), 1049 (M), 1009 (S), 953 (W), 929 (S), 913 (M), 833 (M), 820 (S), 801 (W), 770 (W), 747 (VS), 704 (S), 669 (S), 644 (M), 623 (W), 588 (W), 583 (W), 561 (W), 548 (S), 533 (S), 524 (M), 505 (M), 452 (W), 417 (W).

ESI-HRMS (m/z): calc. for  $(C_{12}H_{15}BrIO_2S^+)$  [M+H]<sup>+</sup> 428.9015; found: 428.9008.



Following **GP-3**, (E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (238 mg, 85%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 154-155 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01 (d, *J* = 3.8 Hz, 1H), 7.82 (d, *J* = 4.8 Hz, 1H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.33-7.16 (m, 6H), 7.08 (d, *J* = 7.2 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 182.6, 149.1, 141.1, 139.9, 139.3, 137.3, 136.6, 132.2, 129.9, 129.9, 129.3, 129.1, 128.2, 127.5, 115.6.

IR (v/cm<sup>-1</sup>): 3088 (W), 3059 (W), 3017 (VW) 1646 (VS), 1606 (M), 1586 (M), 1571 (S), 1511 (M), 1486 (M), 1469 (M), 1442 (M), 1407 (S), 1389 (M), 1353 (S), 1323 (S), 1295 (W), 1263 (S), 1233 (M), 1200 (M), 1178 (W), 1150 (VS), 1112 (W), 1082 (S), 1065 (S), 1019 (M), 1008 (S), 918 (VW) 887 (W), 859 (M), 822 (M), 780 (S), 744 (VS), 729 (S), 693 (S), 679 (M), 659 (M), 642 (W), 619 (W), 573 (M), 563 (S), 538 (VS), 480 (W), 416 (W).

ESI-HRMS (m/z): calc. for  $(C_{19}H_{13}BrIO_3S_2^+)$  [M+H]<sup>+</sup> 558.8529; found: 558.8525.

(Z)-2-(1,2-diphenyl-2-tosylvinyl)naphthalene (10a)



Following **GP-4**, (Z)-2-(1,2-diphenyl-2-tosylvinyl)naphthalene (276 mg, 60%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 232-234 °C.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.76-7.62 (m, 2H), 7.62-7.49 (m, 2H), 7.44-7.33 (m, 4H), 7.33-7.18 (m, 9H), 7.18-7.04 (m, 4H), 2.34 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 152.3, 143.6, 142.2, 139.2, 138.4, 137.4, 133.5, 132.2, 132.0, 131.7, 129.2, 128.5, 128.1, 127.9, 127.8, 127.7, 127.5, 127.3, 127.2, 126.6, 126.3, 126.2, 21.0.

IR (v/cm<sup>-1</sup>): 3060 (W), 3020 (W), 1596 (M), 1581 (W), 1493 (W), 1444 (M), 1310 (S), 1302 (S), 1290 (M), 1196 (W), 1142 (VS), 1085 (M), 1032 (W), 946 (W), 902 (W), 888 (W), 872 (W), 829 (M), 810 (M), 786 (M), 755 (M), 734 (W), 709 (S), 697 (S), 664 (VS), 644 (W), 613 (W), 593 (S), 583 (S), 571 (M), 538 (S), 506 (M), 477 (M), 473 (W), 440 (VW).

1-(1,2-diphenyl-2-tosylvinyl)naphthalene (10b)



Following **GP-4**, 1-(1,2-diphenyl-2-tosylvinyl)naphthalene (363 mg, 79%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 195-198 °C.

<sup>1</sup>H NMR (400 MHz, DMSO- $d_6$ )  $\delta$  8.08 (d, J = 8.5 Hz, 1H), 7.77-7.69 (m, 1H), 7.62 (d, J = 8.3 Hz, 1H), 7.59-7.51 (m, 1H), 7.51-7.35 (m, 6H), 7.35-7.21 (m, 6H), 7.21-7.07 (m, 2H), 7.02-6.88 (m, 3H), 2.33 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 150.4, 143.7, 143.7, 138.4, 138.1, 137.2, 133.3, 132.8, 130.8, 130.0, 129.2, 128.9, 128.2, 128.0, 127.9, 127.8, 127.8, 127.5, 127.2, 127.0, 126.5, 125.9, 125.9, 125.0, 21.0.

IR (v/cm<sup>-1</sup>): 3057 (M), 3042 (M), 2999 (W), 2956 (W), 2920 (W), 2855 (VW) 1964 (W), 1898 (W), 1817 (W), 1613 (M), 1595 (M), 1575 (W), 1505 (M), 1493 (M), 1445 (S), 1400 (W), 1390 (M), 1310 (M), 1300 (S), 1287 (VS), 1263 (M), 1248 (M), 1231 (M), 1208 (W), 1201 (W), 1188 (W), 1182 (W), 1152 (S), 1139 (VS), 1085 (S), 1047 (M), 1034 (M), 1017 (M), 1000 (M), 985 (W), 972 (W), 961 (W), 954 (W), 906 (W), 867 (W), 844 (W), 814 (S), 800 (S), 785 (S), 772 (VS), 757 (M), 754 (M), 736 (M), 720 (W), 713 (S), 705 (S), 696 (S), 684 (S), 664 (S), 654 (M), 634 (M), 625 (M), 594 (S), 576 (M), 566 (VS), 544 (M), 522 (M), 512 (S), 508 (S), 492 (M), 480 (M), 417 (W).

ESI-HRMS (m/z): calc. for  $(C_{31}H_{25}O_2S^+)$  [M+H]<sup>+</sup> 461.1570; found: 461.1569.

(E)-(1-(4-tert-butylphenyl)-2-tosylethene-1,2-diyl)dibenzene (10c)



Following **GP-4**, (E)-(1-(4-tert-butylphenyl)-2-tosylethene-1,2-diyl)dibenzene (317 mg, 68%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 196-197 °C.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.42-7.11 (m, 14H), 7.11-7.00 (m, 2H), 6.91-6.78 (m, 2H), 2.33 (s, 3H, Ar-CH<sub>3</sub>), 1.09 (s, 9H, t-Bu).

<sup>13</sup>C NMR (101 MHz, DMSO) δ 152.1, 150.0, 143.4, 141.4, 139.5, 137.7, 137.6, 133.6, 129.2, 128.6, 128.4, 128.1, 127.6, 127.5, 124.5, 34.1, 30.8, 21.0.

IR (v/cm<sup>-1</sup>): 3080 (W), 3062 (W), 3029 (W), 2966 (M), 2929 (W), 2904 (W), 2868 (W), 1954 (VW) 1906 (VW) 1651 (W), 1595 (M), 1510 (W), 1491 (M), 1461 (W), 1444 (M), 1402 (M), 1380 (W), 1364 (W), 1313 (S), 1302 (S), 1291 (S), 1268 (M), 1224 (W), 1200 (W), 1181 (W), 1148 (VS), 1118 (M), 1085 (S), 1074 (M), 1023 (M), 974 (W), 922 (W), 858 (W), 848 (M), 837 (S), 819 (M), 811 (S), 757 (M), 752 (M), 742 (W), 708 (S), 696 (VS), 667 (M), 653 (S), 603 (S), 580 (S), 572 (S), 550 (M), 541 (S), 515 (M), 503 (M), 460 (W).

ESI-HRMS (m/z): calc. for  $(C_{31}H_{31}O_2S^+)$  [M+H]<sup>+</sup> 467.2039; found: 467.2041.

(E)-(1-(4-methoxyphenyl)-2-tosylethene-1,2-diyl)dibenzene (10d)



Following **GP-4**, (E)-(1-(4-methoxyphenyl)-2-tosylethene-1,2-diyl)dibenzene (282 mg, 64%) was obtained as a white powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 180-181-  $^{\circ}$ C.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.34-7.26 (m, 5H), 7.26-7.18 (m, 7H), 7.18-7.11 (m, 2H), 6.90-6.79 (m, 2H), 6.66-6.54 (m, 2H), 3.58 (s, 3H, -OCH<sub>3</sub>), 2.33 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 158.5, 152.1, 143.4, 140.8, 139.6, 137.7, 133.9, 132.9, 132.4, 130.6, 129.2, 128.6, 128.1, 127.6, 127.5, 113.1, 54.9, 21.0.

IR (v/cm<sup>-1</sup>): 3065 (W), 3053 (W), 3042 (W), 2964 (M), 2934 (W), 2911 (W), 2839 (W), 2549 (VW) 2034 (VW) 1972 (VW) 1951 (W), 1924 (VW) 1887 (W), 1808 (VW) 1653 (W), 1605 (S), 1596 (S), 1581 (M), 1563 (M), 1508 (S), 1491 (S), 1463 (M), 1443 (S), 1414 (M), 1403 (W), 1301 (VS), 1290 (VS), 1268 (S), 1251 (VS), 1226 (M), 1188 (M), 1174 (S), 1155 (S), 1143 (VS), 1121 (S), 1117 (S), 1086 (S), 1079 (S), 1027 (S), 1015 (M), 1001 (M), 991 (W), 977 (M), 955 (W), 923 (W), 913 (M), 859 (M), 841 (S), 818 (S), 813 (S), 785 (S), 758 (S), 747 (S), 705 (S), 699 (S), 689 (S), 683 (S), 650 (S), 601 (S), 592 (S), 566 (M), 549 (VS), 533 (M), 512 (S), 493 (M), 483 (M), 443 (VW).

ESI-HRMS (m/z): calc. for  $(C_{28}H_{25}O_3S^+)$  [M+H]<sup>+</sup> 441.1519; found: 441.1520.

(2-tosylethene-1,1,2-triyl)tribenzene (10e)



Following **GP-4**, (2-tosylethene-1,1,2-triyl)tribenzene (287 mg, 70%) was obtained as a white powder, m.p. ( $CH_2Cl_2$ ) 195-198 °C.

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.38-7.26 (m, 5H), 7.26-7.11 (m, 9H), 7.11-6.91 (m, 5H), 2.33 (s, 3H, Ar-CH<sub>3</sub>).

<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) δ 152.3, 143.5, 141.9, 140.8, 139.3, 137.4, 133.4, 132.2, 129.2, 128.5, 128.3, 128.1, 127.8, 127.6, 127.5, 21.0.

IR (u/cm<sup>-1</sup>): 3100 (W), 3083 (W), 3055 (M), 3029 (M), 2921 (W), 2856 (VW) 2734 (VW) 1952 (VW) 1893 (VW) 1810 (VW) 1607 (M), 1596 (S), 1576 (M), 1490 (S), 1444 (S), 1398 (W), 1379 (W), 1321 (VS), 1300 (S), 1293 (S), 1262 (M), 1228 (M), 1212 (W), 1185 (M), 1178 (M), 1160 (VS), 1148 (VS), 1119 (M), 1106 (M), 1085 (S), 1033 (M), 1027 (M), 1017 (M), 1001 (M), 979 (M), 921 (W), 906 (W), 853 (M), 830 (S), 814 (S), 797 (W), 757 (S), 744 (S), 708 (VS), 704 (VS), 699 (VS), 693 (VS), 678 (S), 658 (S), 621 (W), 600 (VS), 571 (M), 559 (VS), 526 (M), 506 (S), 486 (W), 472 (M), 442 (W).

ESI-HRMS (m/z): calc. for  $(C_{27}H_{23}O_2S^+)$  [M+H]<sup>+</sup> 411.1413; found: 411.1412.

(Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1-ynyl)benzene (10f)



Following **GP-5**, (Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1ynyl)benzene (416 mg, 77%) was obtained as a pale yellow powder, m.p. (CH<sub>2</sub>Cl<sub>2</sub>) 99-101 °C.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.52-7.40 (m, 2H), 7.38-7.19 (m, 6H), 7.15-7.00 (m, 2H), 6.93-6.74 (m, 2H), 3.81 (s, 3H, Ar-OCH<sub>3</sub>), 3.10-2.76 (m, 2H, -CH<sub>2</sub>-), 2.52-2.19 (m, 3H, Ar-CH<sub>3</sub>), 1.99-1.71 (m, 2H, -CH<sub>2</sub>-), 1.68-1.44 (m, 2H, -CH<sub>2</sub>-), 1.44-1.16 (m, 4H, -CH<sub>2</sub>-), 1.08-0.75 (m, 3H, -CH<sub>2</sub>-<u>CH<sub>3</sub></u>).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 160.8, 150.3, 143.9, 140.3, 140.3, 138.0, 133.5, 130.2 (q, J = 32.5 Hz, 1C, <u>C</u>-CF<sub>3</sub>), 129.6, 129.3, 127.7, 124.7 (q, J = 3.7 Hz, 2C, <u>C</u>-C-CF<sub>3</sub>), 124.1 (q, J = 272.2 Hz, 1C, -CF<sub>3</sub>), 114.3, 113.9, 102.7, 86.9, 55.5, 32.7, 31.7, 29.6, 22.7, 21.6, 14.2.

<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -62.7 (s, 1F, Ar-F).

IR (v/cm<sup>-1</sup>): 3067 (VW), 2957 (M), 2929 (M), 2873 (W), 2856 (M), 2190 (M), 1605 (S), 1578 (M), 1567 (M), 1509 (S), 1463 (W), 1407 (W), 1322 (VS), 1303 (M), 1291 (M), 1252 (S), 1155 (S), 1125 (S), 1107 (S), 1082 (M), 1067 (S), 1033 (M), 1019 (M), 899 (VW), 856 (M), 830 (M), 811 (M), 767 (W), 714 (S), 674 (M), 652 (M), 611 (M), 571 (M), 545 (M), 529 (M).

ESI-HRMS (m/z): calc. for  $(C_{31}H_{32}F_3O_3S^+)$  [M+H]<sup>+</sup> 541.2019; found: 541.2026.

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NMR spectra of symmetrical 1,2-diarylalkynes



Figure S4. <sup>1</sup>H NMR (600 MHz, Chloroform-d) of 1,2-di(p-tolyl)ethyne (1b).



Figure S5. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-di(p-tolyl)ethyne (1b).



Figure S6. <sup>13</sup>C DEPTQ-135 NMR 1,2-di(p-tolyl)ethyne (1b).



Figure S7. 1H NMR (600 MHz, Chloroform-d) of 1,2-di(m-tolyl)ethyne (1c).



Figure S8. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-di(m-tolyl)ethyne (1c).



Figure S9. <sup>13</sup>C DEPTQ-135 NMR 1,2-di(m-tolyl)ethyne (1c).



Figure S10. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(4-fluorophenyl)ethyne (1d).



Figure S11. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(4-fluorophenyl)ethyne (1d).



Figure S12. <sup>19</sup>F{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(4-fluorophenyl)ethyne (1d).



Figure S13. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1e).



Figure S14. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1e).



Figure S15. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of 1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1e).



Figure S16. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1f).



Figure S17. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1f).



Figure S18. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of 1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1f).



Figure S19. <sup>13</sup>C DEPTQ-135 NMR 1,2-bis(3-(trifluoromethyl)phenyl)ethyne (1f)



Figure S20. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(4-chlorophenyl)ethyne (1g).



Figure S21. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(4-chlorophenyl)ethyne (1g).



Figure S22. <sup>13</sup>C DEPTQ-135 NMR 1,2-bis(4-chlorophenyl)ethyne (1g).



Figure S23. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(4-nitrophenyl)ethyne (1h).



Figure S24. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(4-nitrophenyl)ethyne (1h).



Figure S25. <sup>13</sup>C DEPTQ-135 NMR 1,2-bis(4-nitrophenyl)ethyne (1h).


Figure S26. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(3-nitrophenyl)ethyne (1i).



Figure S27. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-bis(3-nitrophenyl)ethyne (1i).



Figure S28. <sup>13</sup>C DEPTQ-135 NMR 1,2-bis(3-nitrophenyl)ethyne (1i).



Figure S29. 1H NMR (600 MHz, Chloroform-d) of diethyl 4,4'-(ethyne-1,2-diyl)dibenzoate (1j).



Figure S30. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of diethyl 4,4'-(ethyne-1,2-diyl)dibenzoate (1j).



Figure S31. <sup>13</sup>C DEPTQ-135 NMR diethyl 4,4'-(ethyne-1,2-diyl)dibenzoate (1j).



Figure S32. 1H NMR (600 MHz, Chloroform-d) of 1,2-bis(4-methoxyphenyl)ethyne (1k).



Figure S33. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of diethyl 1,2-bis(4-methoxyphenyl)ethyne (1k).



Figure S34. <sup>13</sup>C DEPTQ-135 NMR diethyl 1,2-bis(4-methoxyphenyl)ethyne (1k).



Figure S35. 1H NMR (600 MHz, Chloroform-d) of 1,2-di(thiophen-2-yl)ethyne (11).



Figure S36. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-di(thiophen-2-yl)ethyne (11).



Figure S37. <sup>13</sup>C DEPTQ-135 NMR 1,2-di(thiophen-2-yl)ethyne (11).



Figure S38. 1H NMR (600 MHz, Chloroform-d) of 1,2-di(thiophen-3-yl)ethyne (1m).



Figure S39. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-di(thiophen-3-yl)ethyne (1m).



Figure S40. <sup>13</sup>C DEPTQ-135 NMR 1,2-di(thiophen-3-yl)ethyne (1m).



Figure S41. 1H NMR (600 MHz, Chloroform-d) of 1,2-di(naphthalen-2-yl)ethyne (1n).



Figure S42. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of 1,2-di(naphthalen-2-yl)ethyne (1n).

## NMR spectra of products



Figure S43. 1H NMR (600 MHz, Chloroform-d) of (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (3a).



Figure S44. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (3a).



Figure S45. <sup>13</sup>C DEPTQ-135 NMR (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (3a).



Figure S46. 1H-13C HSQC (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (3a).



Figure S47. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).



Figure S48. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).



Figure S49. <sup>13</sup>C DEPTQ-135 NMR (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).



Figure S50. 1H-13C HSQC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).



Figure S51. 1H-13C HMBC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).



Figure S52. 1H NMR (600 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3c).



Figure S53. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3c).



Figure S54. 13C DEPTQ-135 NMR (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3c).



Figure S55. 1H-13C HSQC (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3c).



Figure S56. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(fluorobenzene)(3d).



Figure S57. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(fluorobenzene)(3d).



< -110.79 < -111.07





Figure S59. <sup>13</sup>C DEPTQ-135 NMR (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(fluorobenzene)(3d).



Figure S60. 1H-13C HSQC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(fluorobenzene)(3d).



Figure S61. 1H-13C HMBC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(fluorobenzene)(3d).


Figure S62. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3e).



Figure S63. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3e).



Figure S64. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3e).



Figure S65. <sup>13</sup>C DEPTQ-135 NMR (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3e).



Figure S66. 1H NMR (600 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f).



Figure S67. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f).



Figure S68. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f).



Figure S69. <sup>13</sup>C DEPTQ-135 NMR (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f).



Figure S70. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (3g).



Figure S71. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (3g).



Figure S72. <sup>13</sup>C DEPTQ-135 NMR (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (3g).



Figure S73. 1H-13C HSQC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(chlorobenzene) (3g).





Figure S74. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S75. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S76. 13C DEPTQ-135 NMR (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S77. 1H-1H COSY (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S78. 1H-13C HSQC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S79. 1H-13C HMBC (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3h).



Figure S80. 1H NMR (600 MHz, DMSO-d6) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3i).



Figure S81. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(nitrobenzene) (3i).



Figure S82. 1H NMR (600 MHz, Chloroform-d) of (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S83. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S84. <sup>13</sup>C DEPTQ-135 NMR (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S85. 1H-1H COSY (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S86. 1H-13C HSQC (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S87. 1H-13C HMBC (E)-diethyl 4,4'-(1-iodo-2-tosylethene-1,2-diyl)dibenzoate (3j).



Figure S88. 1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (3k).



Figure S89. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (3k).



Figure S90. 1H NMR (600 MHz, Chloroform-d) of (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3l).



Figure S91. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3l).



Figure S92. 1H NMR (600 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3m).



Figure S93. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3m).



Figure S94. <sup>13</sup>C DEPTQ-135 NMR (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3m).



Figure S95. 1H NMR (600 MHz, Chloroform-d) of (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n).



Figure S96. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n).



Figure S97. <sup>13</sup>C DEPTQ-135 NMR (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n).


Figure S98. 1H-13C HSQC (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n).



Figure S99. 1H-13C HMBC (E)-2,2'-(1-iodo-2-tosylethene-1,2-diyl)dinaphthalene (3n).



Figure S100. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (5a).



Figure S101. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (5a).



Figure S102. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (5a).



Figure S103. 1H-13C HSQC (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (5a).



Figure S104. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (5b).



Figure S105. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (5b).



Figure S106. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylhex-1-en-2-ylsulfonyl)-4-methylbenzene (5c).



Figure S107. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylhex-1-en-2-ylsulfonyl)-4-methylbenzene (5c).



Figure S108. 1H-1H COSY (E)-1-(1-iodo-1-phenylhex-1-en-2-ylsulfonyl)-4-methylbenzene (5c).



Figure S109. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenyloct-1-en-2-ylsulfonyl)-4-methylbenzene (5d).



Figure S110. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenyloct-1-en-2-ylsulfonyl)-4-methylbenzene (5d).



Figure S111. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(1-iodo-1-phenyloct-1-en-2-ylsulfonyl)-4-methylbenzene (5d).



Figure S112. 1H NMR (600 MHz, Chloroform-d) of (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5e).





Figure S114. 1H-1H COSY (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5e).



Figure S115. 1H NMR (600 MHz, Chloroform-d) of (E)-ethyl-4-(1-iodo-2-tosyloct-1-enyl)benzoate (5f).





Figure S117. 1H NMR (600 MHz, Chloroform-d) of (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).





Figure S119. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).



Figure S120. <sup>13</sup>C DEPTQ-135 NMR (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).



Figure S121. 1H-1H COSY (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).



Figure S122. 1H-13C HSQC (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).



Figure S123. 1H-13C HMBC (E)-1-fluoro-4-(1-iodo-2-tosyloct-1-enyl)benzene (5g).



Figure S124. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene (5h).



Figure S125. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene



Figure S126. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene (5h).



Figure S127. 1H-13C HSQC (E)-1-(1-iodo-1-(4-(trifluoromethyl)phenyl)oct-1-en-2-ylsulfonyl)-4-methylbenzene (5h).



Figure S128. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (5i).





Figure S130. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (5i).



Figure S131. 1H-13C HSQC (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (5i).



Figure S132. 1H-13C HMBC (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-methoxybenzene (5i).



Figure S133. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (5j).


Figure S134. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (5j).



Figure S135. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (5j).



Figure S136. 1H NMR (600 MHz, Chloroform-d) of (E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (5k).





Figure S138. 1H NMR (600 MHz, Chloroform-d) of (E)-3-(1-iodo-2-tosyloct-1-enyl)thiophene (5l).



Figure S139. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-(1-iodo-2-tosyloct-1-enyl)thiophene (5l).



Figure S140. 1H-1H COSY (E)-3-(1-iodo-2-tosyloct-1-enyl)thiophene (5l).



Figure S141. 1H NMR (600 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S142. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S143. <sup>13</sup>C DEPTQ-135 NMR (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S144. 1H-1H COSY (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S145. 1H-13C HSQC (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S146. 1H-13C HMBC (E)-2-(1-iodo-2-tosyloct-1-enyl)thiophene (5m).



Figure S147. 1H NMR (600 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S148. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S149. 13C DEPTQ-135 NMR (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S150. 1H-1H COSY (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S151. 1H-13C HSQC (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S152. 1H-13C HMBC (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (5n).



Figure S153. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a).





Figure S155. 1H-1H NOESY (E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a).



Figure S156. 1H-13C HSQC (E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a).



Figure S157. 1H-13C HMBC (E)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a).



Figure S158. 1H NMR (600 MHz, Chloroform-d) of (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a').





Figure S160. 1H-1H COSY (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a').



Figure S161. NOESY (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a').



Figure S162. 1H-13C HSQC (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a').



Figure S163. 1H-13C HMBC (Z)-1-(3-iodobut-2-en-2-ylsulfonyl)-4-methylbenzene (7a').



Figure S164. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S165. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S166. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S167. 1H-1H COSY (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S168. 1H-1H NOESY (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S169. 1H-13C HSQC (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).


Figure S170. 1H-13C HMBC (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (7b).



Figure S171. 1H NMR (600 MHz, Chloroform-d) of (E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (7c).



Figure S172. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (7c).



Figure S173. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (7c).



Figure S174. 1H-1H COSY (E)-1-(5-iodooct-4-en-4-ylsulfonyl)-4-methylbenzene (7c).



Figure S175. 1H NMR (600 MHz, Chloroform-d) of (E)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (7d).



Figure S176. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (7d).



Figure S177. <sup>13</sup>C DEPTQ-135 NMR (E)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (7d).



Figure S178. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-1,3-diphenyl-2-tosylprop-2-en-1-one (7e).



Figure S179. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-1,3-diphenyl-2-tosylprop-2-en-1-one (7e).



Figure S180. 1H-13C HSQC (E)-3-iodo-1,3-diphenyl-2-tosylprop-2-en-1-one (7e).



Figure S181. 1H NMR (600 MHz, Chloroform-d) of (E)-2-iodo-1-phenyl-3-tosylhex-2-en-1-one (7f).



Figure S182. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-2-iodo-1-phenyl-3-tosylhex-2-en-1-one (7f).



Figure S183. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-2-tosylprop-2-en-1-ol (7g).



Figure S184. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-2-tosylprop-2-en-1-ol (7g).



Figure S185. <sup>13</sup>C DEPTQ-135 NMR (E)-3-iodo-3-phenyl-2-tosylprop-2-en-1-ol (7g).



Figure S186. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (7h).



Figure S187. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (7h).



Figure S188. 1H-13C HSQC (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (7h).



Figure S189. 1H-13C HMBC (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (7h).



Figure S190. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S191. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S192. <sup>13</sup>C DEPTQ-135 NMR (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S193. 1H-1H COSY (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S194. 1H-13C HSQC (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S195. 1H-13C HMBC (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (7i).



Figure S196. 1H NMR (600 MHz, Chloroform-d) of (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (7j).



Figure S197. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (7j).



Figure S198. 1H-1H COSY (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (7j).



Figure S199. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S200. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S201. <sup>13</sup>C DEPTQ-135 NMR (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S202. 1H-1H COSY (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S203. 1H-13C HSQC (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S204. 1H-13C HMBC (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (7k).



Figure S205. 1H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (7l).


Figure S206. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (7l).



Figure S207. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9a).



Figure S208. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9a).



Figure S209. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9a).



Figure S210. <sup>13</sup>C DEPTQ-135 NMR (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9a).



Figure S211. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9b).



Figure S212. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9b).



Figure S213. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9b).



Figure S214. <sup>13</sup>C DEPTQ-135 NMR (E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9b).





Figure S215. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9c).



Figure S216. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9c).





Figure S217. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9c).



Figure S218. <sup>13</sup>C DEPTQ-135 NMR (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9c).



Figure S219. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9d).



Figure S220. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9d).



Figure S221. <sup>19</sup>F NMR (188 MHz, Chloroform-*d*) of (E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9d).



Figure S222. <sup>13</sup>C DEPTQ-135 NMR (E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9d).



Figure S223. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9e).



Figure S224. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9e).



Figure S225. <sup>13</sup>C DEPTQ-135 NMR (E)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9e).

-10

210 200 190 180 170 160 150 140 130 120 110 100 90 f1 (ppm)



Figure S226. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9f).



Figure S227. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9f).



Figure S228. <sup>13</sup>C DEPTQ-135 NMR (E)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (9f).





Figure S229. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9g).



Figure S230. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9g).



Figure S231. <sup>13</sup>C DEPTQ-135 NMR (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9g).



Figure S232. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9h).



Figure S233. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9h).



Figure S234. <sup>13</sup>C DEPTQ-135 NMR (E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9h).



Figure S235. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9i).

f1 (ppm) -3

-2

-1

10.37<u>4</u> 4.00 ₹



Figure S236. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9i).



Figure S237. <sup>13</sup>C DEPTQ-135 NMR (E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (9i).



Figure S238. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (9j).



Figure S239. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (9j).





Figure S240. <sup>13</sup>C DEPTQ-135 NMR (E)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (9j).



Figure S241. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9k).


Figure S242. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9k).



Figure S243. <sup>13</sup>C DEPTQ-135 NMR (E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (9k).



Figure S244. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9l).



Figure S245. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9l).



Figure S246. <sup>13</sup>C DEPTQ-135 NMR (E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (9l).



Figure S247. <sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>) of (Z)-2-(1,2-diphenyl-2-tosylvinyl)naphthalene (10a).



Figure S248. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>) of (Z)-2-(1,2-diphenyl-2-tosylvinyl)naphthalene (10a).



Figure S249. <sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>) of 1-(1,2-diphenyl-2-tosylvinyl)naphthalene (10b).



Figure S250. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>) of 1-(1,2-diphenyl-2-tosylvinyl)naphthalene (10b).



Figure S251. <sup>13</sup>C DEPTQ-135 NMR 1-(1,2-diphenyl-2-tosylvinyl)naphthalene (10b).



Figure S252. <sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>) of (E)-(1-(4-tert-butylphenyl)-2-tosylethene-1,2-diyl)dibenzene (10c).



Figure S253. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>) of (E)-(1-(4-tert-butylphenyl)-2-tosylethene-1,2-diyl)dibenzene (10c).



Figure S254. <sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>) of (E)-(1-(4-methoxyphenyl)-2-tosylethene-1,2-diyl)dibenzene (10d).



Figure S255. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>) of (E)-(1-(4-methoxyphenyl)-2-tosylethene-1,2-diyl)dibenzene (10d).



Figure S256. <sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>) of (2-tosylethene-1,1,2-triyl)tribenzene (10e).



Figure S257. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>) of (2-tosylethene-1,1,2-triyl)tribenzene (10e).



Figure S258. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of (Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1-ynyl)benzene (10f).



Figure S259. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of (Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1-ynyl)benzene (10f).



Figure S260. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) of (Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1-ynyl)benzene (10f).



Figure S261. <sup>13</sup>C DEPTQ-135 NMR (Z)-1-methoxy-4-(4-tosyl-3-(4-(trifluoromethyl)phenyl)dec-3-en-1-ynyl)benzene (10f).



Figure S262. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of 4-fluorobenzene-1-sulfonyl iodide (8a).



Figure S263. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of 4-fluorobenzene-1-sulfonyl iodide (8a).



Figure S264. <sup>19</sup>F NMR (188 MHz, CDCl<sub>3</sub>) of 4-fluorobenzene-1-sulfonyl iodide (8a).



Figure S265. <sup>13</sup>C DEPTQ-135 NMR 4-fluorobenzene-1-sulfonyl iodide (8a).



Figure S266. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of 4-chlorobenzene-1-sulfonyl iodide (8b).



Figure S267. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of 4-chlorobenzene-1-sulfonyl iodide (8b).



Figure S268. <sup>13</sup>C DEPTQ-135 NMR 4-chlorobenzene-1-sulfonyl iodide (8b).



Figure S269. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) of 4-bromobenzene-1-sulfonyl iodide (8c).



Figure S270. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>) of 4-bromobenzene-1-sulfonyl iodide (8c).



Figure S271. <sup>13</sup>C DEPTQ-135 NMR 4-bromobenzene-1-sulfonyl iodide (8c).