

*Electronic supplementary information (ESI)*

## Supporting Information

### Photoredox-catalyzed intermolecular azidosulfonylation of alkenes with DABCO<sup>·</sup>(SO<sub>2</sub>)<sub>2</sub>, trimethylsilyl azide and thianthrenium salts

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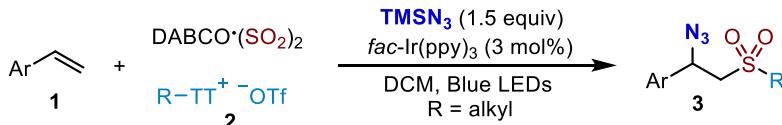
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## 1. General Information

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 $\mu$ m, standard grade). Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the  $\delta$  scale.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded in  $\text{CDCl}_3$  on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

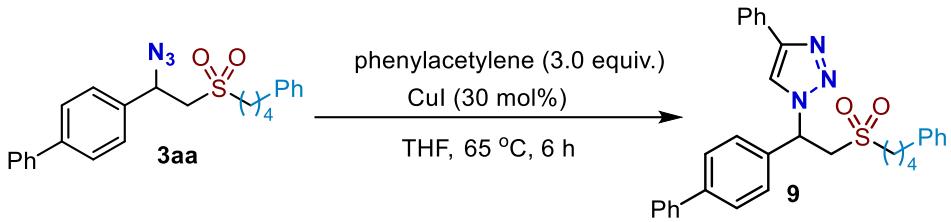
Alkyl thianthrenium salts were prepared according to literature procedures.<sup>1</sup>

## 2. General Experimental Procedure and Characterization Data.

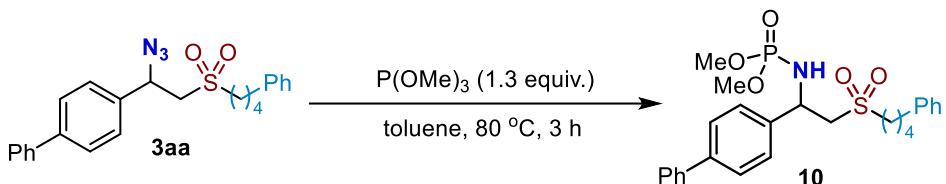


$\text{TMSN}_3$  (0.3 mmol) was added to a mixture of alkenes **1** (0.2 mmol), thianthrenium salts **2** (0.4 mmol),  $\text{DABCO}\cdot(\text{SO}_2)_2$  (0.16 mmol) and  $\text{fac-Ir}(\text{ppy})_3$  (3 mol%) in  $\text{DCM}$  (3 mL) under  $\text{N}_2$  atmosphere. The mixture was placed around a 30 W blue LEDs and stirred under blue light irradiation for 12 hours at 20 °C. After completion of reaction as monitored by TLC analysis, the solvent was evaporated and the residue was purified directly by flash column chromatography on silica gel (petroleum ether /ethyl acetate = 4:1-1:1) to give the corresponding products **3**.

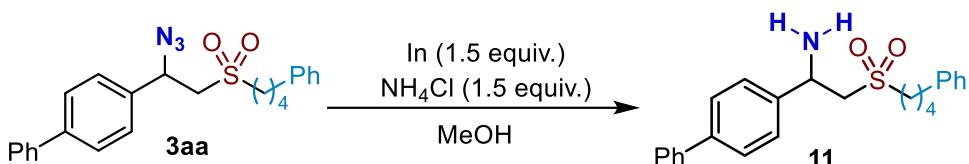
General experimental procedure for further transformation of  $\beta$ -azido sulfones.<sup>2-3</sup>



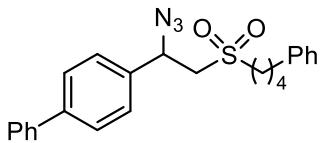
In a flame-dried flask, 4-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-1,1'-biphenyl **3aa** (0.3 mmol) and CuI (30 mol%) were added. Under the protection of N<sub>2</sub>, phenylacetylene (3.0 equiv.) and THF (3.0 mL) were injected respectively into the tube via syringes. The reaction mixture was stirred at 65 °C for 6 h. After completion, the reaction mixture was concentrated in vacuo, the crude residue was subjected to flash column chromatography on silica gel (petroleum ether/ethyl acetate/DCM = 6:1:6 – 1:1:6) to yield the desired product **9** in 90% yield.



In a flame-dried flask, 4-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-1,1'-biphenyl **3aa** (0.3 mmol) were added. Under the protection of N<sub>2</sub>, P(OMe)<sub>3</sub> (1.3 equiv.) and toluene (1.8 mL) were injected respectively into the tube via syringes. The reaction mixture was stirred at 80 °C for 3 h. After completion, the reaction mixture was concentrated in vacuo, the crude residue was subjected to flash column chromatography on silica gel (petroleum ether/ethyl acetate/DCM = 1:1:2 - 1:6:2) to yield the desired product **10** in 96% yield.

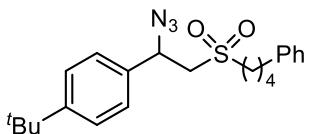


In a flame-dried flask, 4-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-1,1'-biphenyl **3aa** (0.1 mmol) were added, followed by NH<sub>4</sub>Cl (0.15 mmol), indium powder (0.15 mmol). Under the protection of N<sub>2</sub>, MeOH (2 mL) were injected into the tube via syringes. The reaction mixture was stirred at 65 °C for 6 h. After completion, the reaction mixture was concentrated in vacuo, the crude residue was subjected to flash column chromatography on silica gel (petroleum ether /ethyl acetate = 1:1 – 1:2) to yield the desired product **11** in 64% yield.



4-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-1,1'-biphenyl (**3aa**, 62.1 mg, 74%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.62 (d, *J* = 8.1 Hz, 2H), 7.56 (d, *J* = 7.3 Hz, 2H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.39 – 7.33 (m, 3H), 7.27 (t, *J* = 7.3 Hz, 2H), 7.20 – 7.13 (m, 3H), 5.15 (dd, *J* = 9.8, 3.4 Hz, 1H), 3.42 (dd, *J* = 14.9, 9.9 Hz, 1H), 3.14 – 3.02 (m, 3H), 2.63 (t, *J* = 7.4 Hz, 2H), 1.91 – 1.82 (m, 2H), 1.78 – 1.69 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 142.5, 141.2, 140.0, 135.5, 60.5, 58.2, 54.7, 35.3, 30.2, 21.6. **HRMS (ESI)** calcd for C<sub>24</sub>H<sub>25</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 442.1560 (M + Na<sup>+</sup>), found: 442.1564.



1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-(*tert*-butyl)benzene (**3ba**, 63.1 mg, 79%), yellow oil

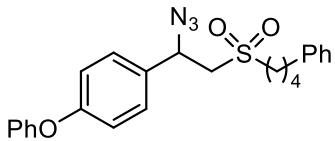
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.44 (d, *J* = 8.1 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.23 – 7.15 (m, 3H), 5.09 (dd, *J* = 9.9, 3.1 Hz, 1H), 3.39 (dd, *J* = 14.9, 10.0 Hz, 1H), 3.12 – 2.95 (m, 3H), 2.66 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.82 (m, 2H), 1.80 – 1.69 (m, 2H), 1.32 (s, 9H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 152.7, 141.2, 133.6, 128.5, 128.4, 126.6, 126.4, 126.1, 60.4, 58.2, 54.6, 35.3, 34.8, 31.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>22</sub>H<sub>29</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 422.1873 (M + Na<sup>+</sup>), found: 422.1883.



1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-methoxybenzene (**3ca**, 38.8 mg, 52%), yellow oil

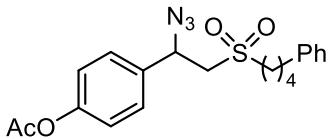
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.30 (t, *J* = 7.4 Hz, 2H), 7.27 – 7.20 (m, 3H), 7.17 (d, *J* = 7.4 Hz, 2H), 6.94 (d, *J* = 8.6 Hz, 2H), 5.07 (dd, *J* = 9.8, 3.6 Hz, 1H), 3.82 (s, 3H), 3.39 (dd, *J* = 14.9, 9.8 Hz, 1H), 3.14 – 2.89 (m, 3H), 2.65 (t, *J* = 7.5 Hz, 2H), 1.92 – 1.82 (m, 2H), 1.80 – 1.70 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 160.3, 141.3, 128.5, 128.4, 128.4, 128.3, 126.1, 114.7,

60.3, 58.2, 55.4, 54.6, 35.3, 30.2, 21.5. **HRMS (ESI)** calcd for C<sub>19</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>3</sub>S<sup>+</sup>: 396.1352 (M + Na<sup>+</sup>), found: 396.1362.



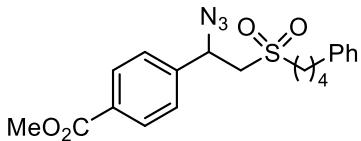
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-phenoxybenzene (**3da**, 55.7 mg, 64%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.37 (t, J = 7.8 Hz, 2H), 7.32 – 7.26 (m, 4H), 7.23 – 7.13 (m, 4H), 7.06 – 7.00 (m, 4H), 5.10 (dd, J = 9.9, 3.2 Hz, 1H), 3.39 (dd, J = 14.9, 10.0 Hz, 1H), 3.15 – 3.03 (m, 3H), 2.67 (t, J = 7.4 Hz, 2H), 1.93 – 1.83 (m, 2H), 1.81 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 158.5, 156.2, 141.2, 130.9, 130.0, 128.5, 128.5, 128.4, 126.1, 124.1, 119.6, 119.0, 60.2, 58.2, 54.7, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>24</sub>H<sub>25</sub>N<sub>3</sub>NaO<sub>3</sub>S<sup>+</sup>: 458.1509 (M + Na<sup>+</sup>), found: 458.1512.



4-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)phenyl acetate (**3ea**, 43.4 mg, 54%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.35 (d, J = 8.5 Hz, 2H), 7.30 (t, J = 7.4 Hz, 2H), 7.23 – 7.14 (m, 5H), 5.13 (dd, J = 10.1, 3.1 Hz, 1H), 3.37 (dd, J = 14.9, 10.2 Hz, 1H), 3.16 – 2.97 (m, 3H), 2.67 (t, J = 7.4 Hz, 2H), 2.32 (s, 3H), 1.92 – 1.83 (m, 2H), 1.82 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 169.3, 151.3, 141.2, 134.3, 128.5, 128.4, 128.0, 126.1, 122.7, 60.1, 58.2, 54.8, 35.3, 30.1, 21.5, 21.2. **HRMS (ESI)** calcd for C<sub>20</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>4</sub>S<sup>+</sup>: 424.1301 (M + Na<sup>+</sup>), found: 424.1310.



Methyl 4-(1-azido-2-((4-phenylbutyl)sulfonyl)ethyl)benzoate (**3fa**, 63.4 mg, 79%), yellow oil

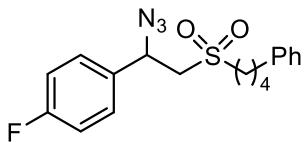
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 8.10 (d, J = 8.1 Hz, 2H), 7.42 (d, J = 8.1 Hz, 2H), 7.29 (t, J = 7.4 Hz, 2H), 7.24 – 7.14 (m, 3H), 5.19 (dd, J = 10.0, 2.8 Hz, 1H), 3.93 (s, 3H), 3.39 (dd, J = 14.9,

10.1 Hz, 1H), 3.20 – 3.01 (m, 3H), 2.67 (t,  $J$  = 7.4 Hz, 2H), 1.94 – 1.84 (m, 2H), 1.82 – 1.74 (m, 2H).  **$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 166.3, 141.5, 141.2, 131.2, 130.7, 128.5, 128.4, 127.0, 126.2, 60.3, 58.0, 54.8, 52.4, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for  $\text{C}_{20}\text{H}_{23}\text{N}_3\text{NaO}_4\text{S}^+$ : 424.1301 ( $\text{M} + \text{Na}^+$ ), found: 424.1310.



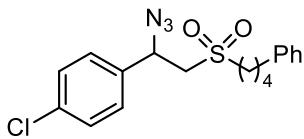
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-(trifluoromethyl)benzene (**3ga**, 53.5 mg, 65%), yellow oil

**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 7.70 (d,  $J$  = 8.0 Hz, 2H), 7.48 (d,  $J$  = 8.0 Hz, 2H), 7.30 (t,  $J$  = 7.4 Hz, 2H), 7.23 – 7.16 (m, 3H), 5.21 (dd,  $J$  = 10.1, 2.8 Hz, 1H), 3.38 (dd,  $J$  = 14.9, 10.2 Hz, 1H), 3.17 – 3.00 (m, 3H), 2.67 (t,  $J$  = 7.4 Hz, 2H), 1.94 – 1.85 (m, 2H), 1.83 – 1.74 (m, 2H).  **$^{19}\text{F}$  NMR** (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) -62.75 (s).  **$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 141.0 (d,  ${}^1J_{\text{CF}} = 39.9$  Hz), 131.6 (q,  ${}^2J_{\text{CF}} = 32.8$  Hz), 128.5, 128.4, 127.3, 126.5 (q,  ${}^3J_{\text{CF}} = 3.6$  Hz), 126.2, 125.0, 122.3, 60.2, 58.0, 54.9, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for  $\text{C}_{19}\text{H}_{20}\text{F}_3\text{N}_3\text{NaO}_2\text{S}^+$ : 434.1121 ( $\text{M} + \text{Na}^+$ ), found: 434.1128.



1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-fluorobenzene (**3ha**, 48.4 mg, 67%), yellow oil

**$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 7.35 – 7.27 (m, 4H), 7.23 – 7.16 (m, 3H), 7.12 (t,  $J$  = 8.5 Hz, 2H), 5.12 (dd,  $J$  = 10.0, 3.2 Hz, 1H), 3.38 (dd,  $J$  = 14.9, 10.1 Hz, 1H), 3.13 – 3.00 (m, 3H), 2.67 (t,  $J$  = 7.4 Hz, 2H), 1.93 – 1.84 (m, 2H), 1.82 – 1.72 (m, 2H).  **$^{19}\text{F}$  NMR** (376 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) -111.27 – -111.17 (m).  **$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 163.1 (d,  ${}^1J_{\text{CF}} = 249.3$  Hz), 141.2, 132.6 (d,  ${}^3J_{\text{CF}} = 3.3$  Hz), 128.8, 128.8, 128.5, 128.4, 116.6 (d,  ${}^2J_{\text{CF}} = 21.8$  Hz), 60.0, 58.2, 54.7, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for  $\text{C}_{18}\text{H}_{20}\text{FN}_3\text{NaO}_2\text{S}^+$ : 384.1152 ( $\text{M} + \text{Na}^+$ ), found: 384.1159.



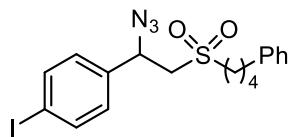
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-chlorobenzene (**3ia**, 56.7 mg, 75%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.40 (d, *J* = 8.4 Hz, 2H), 7.32 – 7.26 (m, 4H), 7.23 – 7.16 (m, 3H), 5.11 (dd, *J* = 10.0, 3.2 Hz, 1H), 3.36 (dd, *J* = 14.9, 10.0 Hz, 1H), 3.14 – 2.99 (m, 3H), 2.66 (t, *J* = 7.4 Hz, 2H), 1.92 – 1.83 (m, 2H), 1.81 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 164.3, 161.8, 141.2, 132.6, 132.6, 128.8, 128.8, 128.5, 128.4, 126.2, 116.7, 116.4, 60.0, 58.2, 54.7, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>18</sub>H<sub>20</sub>ClN<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 400.0857 (M + Na<sup>+</sup>), found: 400.0865.



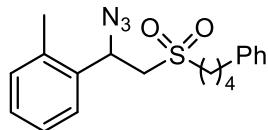
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-bromobenzene (**3ja**, 63.4 mg, 75%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.56 (d, *J* = 8.4 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 2H), 7.23 – 7.16 (m, 5H), 5.09 (dd, *J* = 10.0, 3.3 Hz, 1H), 3.35 (dd, *J* = 14.9, 10.0 Hz, 1H), 3.12 – 2.99 (m, 3H), 2.66 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.83 (m, 2H), 1.82 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 135.8, 132.7, 128.5, 128.4, 126.2, 123.6, 60.1, 58.0, 54.8, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>18</sub>H<sub>20</sub>BrN<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 444.0352 (M + Na<sup>+</sup>), found: 444.0357.



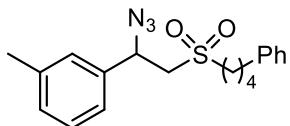
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-iodobenzene (**3ka**, 69.5 mg, 74%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.77 (d, *J* = 8.3 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 2H), 7.23 – 7.15 (m, 3H), 7.08 (d, *J* = 8.3 Hz, 2H), 5.07 (dd, *J* = 10.0, 3.3 Hz, 1H), 3.35 (dd, *J* = 14.9, 10.0 Hz, 1H), 3.12 – 2.98 (m, 3H), 2.67 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.83 (m, 2H), 1.82 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 138.6, 136.4, 128.7, 128.5, 128.4, 126.2, 95.3, 60.2, 58.0, 54.8, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>18</sub>H<sub>20</sub>IN<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 492.0213 (M + Na<sup>+</sup>), found: 492.0220.



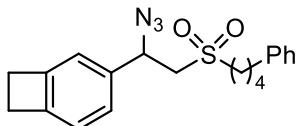
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-2-methylbenzene (**3la**, 58.6 mg, 82%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.35 – 7.26 (m, 5H), 7.25 – 7.16 (m, 4H), 5.39 (dd, *J* = 9.8, 2.9 Hz, 1H), 3.37 (dd, *J* = 15.0, 9.9 Hz, 1H), 3.13 – 2.94 (m, 3H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.41 (s, 3H), 1.94 – 1.83 (m, 2H), 1.81 – 1.71 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 135.5, 134.8, 131.5, 129.2, 128.5, 128.4, 127.1, 126.4, 126.1, 57.5, 57.2, 54.6, 35.3, 30.1, 21.6, 19.3. **HRMS (ESI)** calcd for C<sub>19</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 380.1403 (M + Na<sup>+</sup>), found: 380.1412.



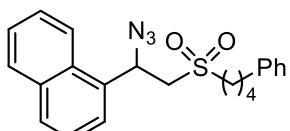
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-3-methylbenzene (**3ma**, 51.5 mg, 72%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.34 – 7.27 (m, 3H), 7.23 – 7.16 (m, 4H), 7.12 (d, *J* = 7.8 Hz, 2H), 5.07 (dd, *J* = 10.0, 3.1 Hz, 1H), 3.39 (dd, *J* = 15.0, 10.0 Hz, 1H), 3.11 – 3.02 (m, 3H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.38 (s, 3H), 1.93 – 1.82 (m, 2H), 1.81 – 1.70 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.3, 139.4, 136.5, 130.3, 129.4, 128.5, 128.4, 127.6, 126.1, 123.9, 60.7, 58.2, 54.6, 35.3, 30.2, 21.5, 21.5. **HRMS (ESI)** calcd for C<sub>19</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 380.1403 (M + Na<sup>+</sup>), found: 380.1414.



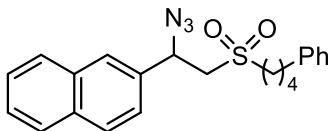
3-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)bicyclo[4.2.0]octa-1(6),2,4-triene (**3na**, 56.9 mg, 77%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.21 (t, *J* = 7.4 Hz, 2H), 7.15 – 7.04 (m, 4H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.94 (s, 1H), 4.98 (dd, *J* = 9.9, 3.4 Hz, 1H), 3.31 (dd, *J* = 15.0, 10.0 Hz, 1H), 3.10 (s, 4H), 2.98 (dd, *J* = 9.9, 4.9 Hz, 3H), 2.57 (t, *J* = 7.5 Hz, 2H), 1.86 – 1.73 (m, 2H), 1.69 – 1.65 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 146.3, 146.0, 140.2, 134.1, 127.4, 127.3, 125.1, 124.8, 122.4, 120.1, 60.3, 57.3, 53.5, 34.2, 29.1, 28.5, 28.5, 20.5. **HRMS (ESI)** calcd for C<sub>20</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 392.1403 (M + Na<sup>+</sup>), found: 392.1412.



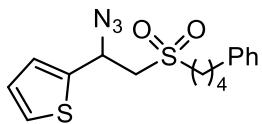
1-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)naphthalene (**3oa**, 60.6 mg, 77%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 8.10 (d, *J* = 8.4 Hz, 1H), 7.94 – 7.86 (m, 2H), 7.63 – 7.48 (m, 4H), 7.28 (t, *J* = 7.3 Hz, 2H), 7.22 – 7.14 (m, 3H), 5.92 (dd, *J* = 9.9, 2.4 Hz, 1H), 3.48 (dd, *J* = 15.1, 10.0 Hz, 1H), 3.23 (dd, *J* = 15.1, 2.6 Hz, 1H), 3.18 – 3.05 (m, 2H), 2.64 (t, *J* = 7.5 Hz, 2H), 1.93 – 1.84 (m, 2H), 1.79 – 1.71 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 134.2, 132.3, 130.0, 129.8, 129.4, 128.5, 128.4, 127.5, 126.5, 126.1, 125.4, 124.9, 122.4, 58.1, 57.8, 54.7, 35.3, 30.1, 21.6. **HRMS (ESI)** calcd for C<sub>22</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 416.1403 (M + Na<sup>+</sup>), found: 416.1411.



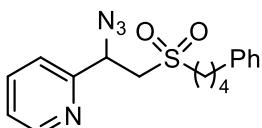
2-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)naphthalene (**3pa**, 63.7 mg, 81%), yellow solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.93 – 7.81 (m, 4H), 7.57 – 7.52 (m, 2H), 7.42 – 7.37 (m, 1H), 7.29 (t, *J* = 7.4 Hz, 2H), 7.22 – 7.13 (m, 3H), 5.29 (dd, *J* = 9.9, 3.4 Hz, 1H), 3.48 (dd, *J* = 15.0, 9.9 Hz, 1H), 3.16 (dd, *J* = 15.0, 3.4 Hz, 1H), 3.12 – 3.04 (m, 2H), 2.63 (t, *J* = 7.5 Hz, 2H), 1.93 – 1.84 (m, 2H), 1.76 – 1.72 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 133.9, 133.5, 133.2, 129.8, 128.5, 128.4, 128.2, 127.9, 127.1, 127.1, 126.7, 126.1, 123.6, 61.0, 58.2, 54.7, 35.3, 30.1, 21.6. **HRMS (ESI)** calcd for C<sub>22</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 416.1403 (M + Na<sup>+</sup>), found: 416.1412.



2-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)thiophene (**3qa**, 37.0 mg, 53%), yellow oil

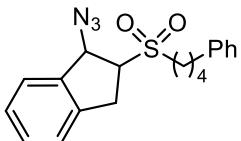
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.38 (d, *J* = 4.9 Hz, 1H), 7.29 (t, *J* = 7.4 Hz, 2H), 7.23 – 7.15 (m, 3H), 7.11 (d, *J* = 3.1 Hz, 1H), 7.05 – 7.01 (m, 1H), 5.41 (dd, *J* = 9.7, 3.7 Hz, 1H), 3.45 (dd, *J* = 14.9, 9.8 Hz, 1H), 3.21 (dd, *J* = 14.9, 3.7 Hz, 1H), 3.11 – 3.02 (m, 2H), 2.66 (t, *J* = 7.4 Hz, 2H), 1.96 – 1.82 (m, 2H), 1.82 – 1.70 (m, 2H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 139.0, 128.5, 128.4, 127.3, 127.1, 127.0, 126.2, 58.4, 56.3, 54.7, 35.3, 30.1, 21.5. **HRMS (ESI)** calcd for C<sub>16</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>2</sub>S<sub>2</sub><sup>+</sup>: 372.0811 (M + Na<sup>+</sup>), found: 372.0819.



2-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)pyridine (**3ra**, 23.4 mg, 34%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 8.63 (d, *J* = 4.2 Hz, 1H), 7.81 – 7.76 (m, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.35 – 7.27 (m, 3H), 7.22 – 7.16 (m, 3H), 5.09 (dd, *J* = 7.6, 5.5 Hz, 1H), 3.61 – 3.55 (m, 2H), 3.09 – 3.03 (m, 2H), 2.66 (t, *J* = 7.5 Hz, 2H), 1.95 – 1.87 (m, 2H), 1.80 – 1.72 (m, 2H).

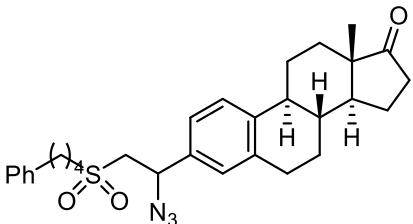
**<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 155.4, 150.2, 141.2, 137.6, 128.5, 128.4, 126.1, 124.1, 122.5, 60.5, 56.1, 54.7, 35.3, 30.2, 21.5. **HRMS (ESI)** calcd for C<sub>17</sub>H<sub>20</sub>N<sub>4</sub>NaO<sub>2</sub>S<sup>+</sup>: 367.1199 (M + Na<sup>+</sup>), found: 367.1211.



1-Azido-2-((4-phenylbutyl)sulfonyl)-2,3-dihydro-1H-indene (**3sa**, 44.1 mg, 62%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.40 (d, *J* = 7.7 Hz, 1H), 7.38 – 7.32 (m, 2H), 7.32 – 7.26 (m, 3H), 7.19 (t, *J* = 9.3 Hz, 3H), 5.34 (d, *J* = 6.6 Hz, 1H), 3.78 (dd, *J* = 15.6, 8.3 Hz, 1H), 3.51 – 3.31 (m, 2H), 3.12 – 3.02 (m, 2H), 2.69 (t, *J* = 7.5 Hz, 2H), 2.01 – 1.90 (m, 2H), 1.87 – 1.76 (m, 2H).

**<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 141.2, 138.7, 137.9, 129.8, 128.5, 128.4, 128.1, 126.2, 125.1, 124.5, 66.8, 65.7, 52.5, 35.3, 31.1, 30.2, 21.2. **HRMS (ESI)** calcd for C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 378.1247 (M + Na<sup>+</sup>), found: 378.1252.

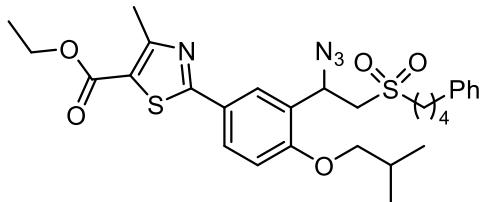


(8*R*,9*S*,13*S*,14*S*)-3-(1-Azido-2-((4-phenylbutyl)sulfonyl)ethyl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[a]phenanthren-17-one (**3ta**, 48.9 mg, 47%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.36 – 7.27 (m, 3H), 7.23 – 7.16 (m, 3H), 7.10 (d, *J* = 8.1 Hz, 1H), 7.05 (s, 1H), 5.05 (dd, *J* = 10.0, 3.3 Hz, 1H), 3.39 (dd, *J* = 14.9, 10.1 Hz, 1H), 3.11 – 3.03 (m, 3H), 2.96 – 2.91 (m, 2H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.58 – 2.47 (m, 1H), 2.45 – 2.38 (m, 1H), 2.35 – 2.26 (m, 1H), 2.21 – 2.06 (m, 2H), 2.05 – 1.94 (m, 2H), 1.92 – 1.84 (m, 2H), 1.81 – 1.71 (m, 2H), 1.69 – 1.59 (m, 2H), 1.56 – 1.42 (m, 4H), 0.91 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** 220.7,

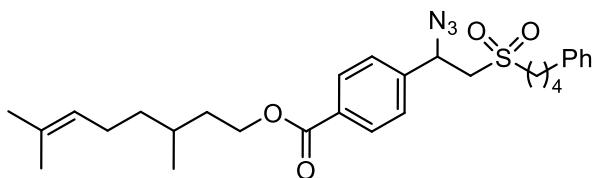
141.3, 141.2, 137.8, 134.0, 128.5, 128.4, 127.5, 127.4, 126.5, 126.5, 126.1, 124.2, 124.1, 60.5, 60.4, 58.2, 54.6, 50.5, 47.9, 44.4, 37.9, 35.8, 35.3, 31.5, 30.2, 29.4, 26.3, 25.7, 21.6, 21.5, 13.8.

**HRMS (ESI)** calcd for C<sub>30</sub>H<sub>37</sub>N<sub>3</sub>NaO<sub>3</sub>S<sup>+</sup>: 542.2448 (M + Na<sup>+</sup>), found: 542.2453.



Ethyl 2-(3-(1-azido-2-((4-phenylbutyl)sulfonyl)ethyl)-4-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (**3ua**, 85.1 mg, 77%), white solid

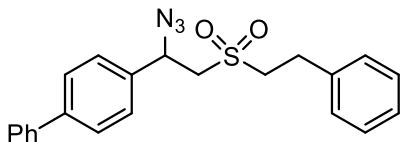
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.99 (s, 1H), 7.95 – 7.90 (m, 1H), 7.34 – 7.25 (m, 2H), 7.20 (t, J = 7.8 Hz, 3H), 6.98 (d, J = 8.6 Hz, 1H), 5.56 (d, J = 10.1 Hz, 1H), 4.36 (q, J = 7.1 Hz, 1H), 3.92 – 3.80 (m, 2H), 3.44 (dd, J = 14.9, 10.5 Hz, 1H), 3.23 – 3.11 (m, 3H), 2.78 (s, 3H), 2.68 (t, J = 7.4 Hz, 2H), 2.23 – 2.13 (m, 1H), 1.97 – 1.88 (m, 2H), 1.85 – 1.76 (m, 2H), 1.39 (t, J = 7.1 Hz, 3H), 1.13 – 1.05 (m, 6H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 168.8, 162.3, 161.1, 158.0, 141.3, 129.1, 128.5, 128.4, 126.2, 126.1, 126.1, 125.7, 121.4, 112.2, 75.2, 61.3, 56.6, 56.1, 54.8, 35.4, 30.2, 28.3, 21.6, 19.3, 19.3, 17.6, 14.4. **HRMS (ESI)** calcd for C<sub>29</sub>H<sub>36</sub>N<sub>4</sub>NaO<sub>5</sub>S<sub>2</sub><sup>+</sup>: 607.2019 (M + Na<sup>+</sup>), found: 607.2025.



3,7-Dimethyloct-6-en-1-yl 4-(1-azido-2-((4-phenylbutyl)sulfonyl)ethyl)benzoate (**3va**, 83.1 mg, 79%), yellow oil

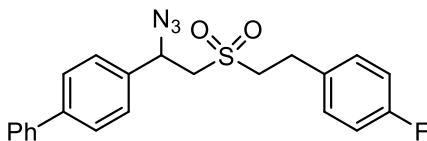
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 8.10 (d, J = 8.0 Hz, 2H), 7.42 (d, J = 8.1 Hz, 2H), 7.29 (t, J = 7.4 Hz, 2H), 7.24 – 7.15 (m, 3H), 5.19 (dd, J = 10.0, 2.5 Hz, 1H), 5.10 (t, J = 6.7 Hz, 1H), 4.43 – 4.32 (m, 2H), 3.39 (dd, J = 14.9, 10.2 Hz, 1H), 3.15 – 3.03 (m, 3H), 2.67 (t, J = 7.4 Hz, 2H), 2.09 – 1.96 (m, 2H), 1.94 – 1.85 (m, 2H), 1.84 – 1.74 (m, 3H), 1.71 – 1.55 (m, 8H), 1.45 – 1.36 (m, 1H), 1.30-1.19 (m, 1H), 0.97 (d, J = 6.3 Hz, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 165.8, 141.3, 141.2, 131.6, 131.5, 130.7, 128.5, 128.4, 126.9, 126.2, 124.5, 63.9, 60.3, 58.0, 54.8, 37.0,

35.5, 35.3, 30.1, 29.5, 25.8, 25.4, 21.5, 19.5, 17.7. **HRMS (ESI)** calcd for C<sub>29</sub>H<sub>39</sub>N<sub>3</sub>NaO<sub>4</sub>S<sup>+</sup>: 548.2553 (M + Na<sup>+</sup>), found: 548.2559.



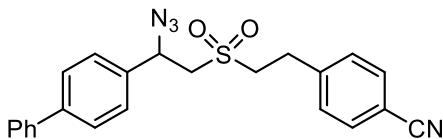
4-(1-Azido-2-(phenethylsulfonyl)ethyl)-1,1'-biphenyl (**3ab**, 50.9 mg, 65%), yellow oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.64 (d, J = 8.0 Hz, 2H), 7.58 (d, J = 7.7 Hz, 2H), 7.46 (t, J = 7.5 Hz, 2H), 7.40 – 7.31 (m, 5H), 7.28 (d, J = 7.0 Hz, 1H), 7.22 (d, J = 7.3 Hz, 2H), 5.17 (dd, J = 9.9, 3.2 Hz, 1H), 3.47 – 3.30 (m, 3H), 3.19 – 3.07 (m, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 168.8, 162.3, 161.1, 158.0, 141.3, 129.1, 128.5, 128.4, 126.2, 126.1, 126.1, 125.7, 121.4, 112.2, 75.2, 61.3, 56.6, 56.1, 54.8, 35.4, 30.2, 28.3, 21.6, 19.3, 19.3, 17.6, 14.4. **HRMS (ESI)** calcd for C<sub>22</sub>H<sub>21</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 414.1247 (M + Na<sup>+</sup>), found: 414.1254.



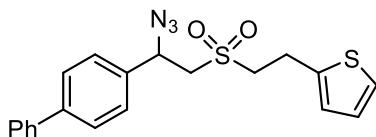
4-(1-Azido-2-((4-fluorophenethyl)sulfonyl)ethyl)-1,1'-biphenyl (**3ac**, 59.8 mg, 73%), white oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.65 (d, J = 8.2 Hz, 2H), 7.58 (d, J = 7.3 Hz, 2H), 7.47 (t, J = 7.5 Hz, 2H), 7.41 – 7.36 (m, 3H), 7.21 – 7.15 (m, 2H), 7.01 (t, J = 8.6 Hz, 2H), 5.18 (dd, J = 9.9, 3.5 Hz, 1H), 3.41 – 3.28 (m, 3H), 3.17 – 3.09 (m, 3H). **<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)**: δ (ppm) -115.38 – -115.29 (m). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 161.9 (d, <sup>1</sup>J<sub>CF</sub> = 245.8 Hz), 142.6, 139.9, 135.3, 133.1 (d, <sup>4</sup>J<sub>CF</sub> = 3.5 Hz), 130.1 (d, <sup>3</sup>J<sub>CF</sub> = 8.0 Hz), 129.0, 128.2, 127.9, 127.4, 127.1, 115.9 (d, <sup>2</sup>J<sub>CF</sub> = 21.6 Hz), 60.5, 58.7, 56.2, 27.4. **HRMS (ESI)** calcd for C<sub>22</sub>H<sub>20</sub>FN<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 432.1152 (M + Na<sup>+</sup>), found: 432.1159.



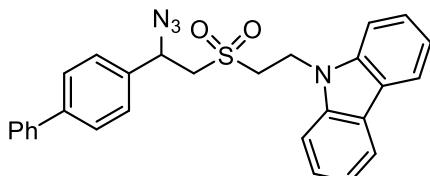
4-(2-((2-([1,1'-Biphenyl]-4-yl)-2-azidoethyl)sulfonyl)ethyl)benzonitrile (**3ad**, 56.6 mg, 68%), white oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.67 (d, *J* = 8.2 Hz, 2H), 7.64 – 7.56 (m, 4H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.42 – 7.37 (m, 3H), 7.34 (d, *J* = 8.1 Hz, 2H), 5.19 (dd, *J* = 10.0, 3.5 Hz, 1H), 3.46 – 3.33 (m, 3H), 3.26 – 3.14 (m, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 143.0, 142.7, 139.8, 135.1, 132.7, 129.4, 129.0, 128.2, 128.0, 127.4, 127.1, 118.5, 111.2, 60.6, 58.9, 55.4, 28.0. **HRMS (ESI)** calcd for C<sub>23</sub>H<sub>20</sub>N<sub>4</sub>NaO<sub>2</sub>S<sup>+</sup>: 439.1199 (M + Na<sup>+</sup>), found: 439.1208.



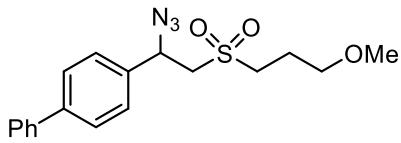
2-(2-((2-([1,1'-Biphenyl]-4-yl)-2-azidoethyl)sulfonyl)ethyl)thiophene (**3ae**, 48.5 mg, 61%), yellow solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.64 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 7.4 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.40 – 7.36 (m, 3H), 7.22 – 7.18 (m, 1H), 6.97 – 6.93 (m, 1H), 6.90 (d, *J* = 3.1 Hz, 1H), 5.18 (dd, *J* = 10.0, 3.4 Hz, 1H), 3.52 – 3.44 (m, 1H), 3.42 – 3.33 (m, 4H), 3.11 (dd, *J* = 15.0, 3.5 Hz, 1H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 143.0, 142.7, 139.8, 135.1, 132.7, 129.4, 129.0, 128.2, 128.0, 127.4, 127.1, 118.5, 111.2, 60.6, 58.9, 55.4, 28.0. **HRMS (ESI)** calcd for C<sub>20</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>2</sub>S<sub>2</sub><sup>+</sup>: 420.0811 (M + Na<sup>+</sup>), found: 420.0820.



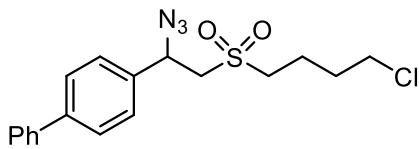
9-((2-((2-((1,1'-Biphenyl)-4-yl)-2-azidoethyl)sulfonyl)ethyl)-9*H*-carbazole (**3af**, 61.5 mg, 64%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 8.11 (d, *J* = 7.8 Hz, 2H), 7.52 – 7.41 (m, 10H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.32 – 7.26 (m, 2H), 6.91 (d, *J* = 8.2 Hz, 2H), 4.99 – 4.89 (m, 2H), 4.82 – 4.74 (m, 1H), 3.94 – 3.80 (m, 1H), 3.53 – 3.40 (m, 1H), 2.96 (dd, *J* = 15.1, 10.4 Hz, 1H), 2.81 (dd, *J* = 15.0, 2.9 Hz, 1H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 142.4, 140.0, 139.7, 134.7, 128.9, 127.0, 128.86, 127.1, 127.1, 126.3, 123.3, 120.7, 120.0, 108.8, 60.1, 59.2, 52.9, 36.8. **HRMS (ESI)** calcd for C<sub>28</sub>H<sub>24</sub>N<sub>4</sub>NaO<sub>2</sub>S<sup>+</sup>: 503.1512 (M + Na<sup>+</sup>), found: 503.1521.



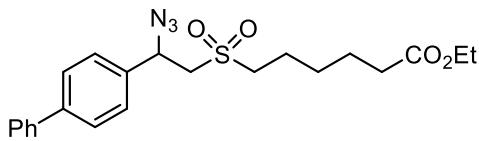
4-(1-Azido-2-((3-methoxypropyl)sulfonyl)ethyl)-1,1'-biphenyl (**3ag**, 59.6 mg, 83%), yellow solid

**$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 7.66 (d,  $J = 8.2$  Hz, 2H), 7.59 (d,  $J = 7.3$  Hz, 2H), 7.49 – 7.36 (m, 5H), 5.20 (dd,  $J = 10.1, 3.2$  Hz, 1H), 3.50 (dd,  $J = 15.0, 8.9$  Hz, 3H), 3.34 (s, 3H), 3.27 – 3.13 (m, 3H), 2.19 – 2.08 (m, 2H).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 142.4, 140.0, 135.6, 129.0, 128.1, 127.9, 127.3, 127.2, 70.2, 60.4, 58.7, 58.5, 52.1, 22.3. **HRMS (ESI)** calcd for  $\text{C}_{18}\text{H}_{21}\text{N}_3\text{NaO}_3\text{S}^+$ : 382.1196 ( $\text{M} + \text{Na}^+$ ), found: 382.1201.



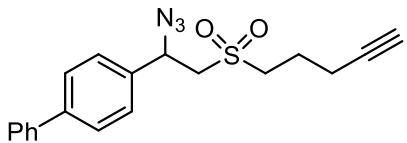
4-(1-Azido-2-((4-chlorobutyl)sulfonyl)ethyl)-1,1'-biphenyl (**3ah**, 57.4 mg, 76%), white solid

**$^1\text{H NMR}$  (400 MHz,  $\text{DMSO-d}_6$ ):**  $\delta$  (ppm) 7.77 – 7.68 (m, 4H), 7.59 (d,  $J = 8.2$  Hz, 2H), 7.48 (t,  $J = 7.5$  Hz, 2H), 7.39 (t,  $J = 7.3$  Hz, 1H), 5.29 (dd,  $J = 9.7, 3.7$  Hz, 1H), 3.89 (dd,  $J = 14.7, 9.7$  Hz, 1H), 3.70 – 3.58 (m, 3H), 3.22 (d,  $J = 6.3$  Hz, 2H), 1.89 – 1.80 (m, 4H).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{DMSO-d}_6$ ):**  $\delta$  (ppm) 141.1, 139.9, 136.9, 129.5, 128.5, 128.2, 127.6, 127.2, 59.3, 56.5, 53.0, 45.1, 31.1, 19.2. **HRMS (ESI)** calcd for  $\text{C}_{18}\text{H}_{20}\text{ClN}_3\text{NaO}_2\text{S}^+$ : 400.0857 ( $\text{M} + \text{Na}^+$ ), found: 400.0862.



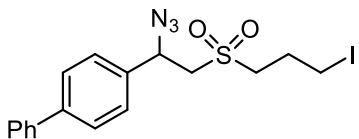
Ethyl 6-((2-([1,1'-biphenyl]-4-yl)-2-azidoethyl)sulfonyl)hexanoate (**3ai**, 34.4 mg, 40%), white oil

**$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 7.66 (d,  $J = 8.2$  Hz, 2H), 7.61 – 7.57 (m, 2H), 7.49 – 7.41 (m, 4H), 7.38 (t,  $J = 7.3$  Hz, 1H), 5.19 (dd,  $J = 10.0, 3.3$  Hz, 1H), 4.16 – 4.10 (m, 2H), 3.47 (dd,  $J = 14.9, 10.1$  Hz, 1H), 3.18 – 3.07 (m, 3H), 2.33 (t,  $J = 7.3$  Hz, 2H), 1.94 – 1.84 (m, 2H), 1.69 (dd,  $J = 15.4, 7.4$  Hz, 2H), 1.53 – 1.45 (m, 2H), 1.26 (t,  $J = 7.1$  Hz, 3H).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):**  $\delta$  (ppm) 173.3, 142.5, 140.0, 135.5, 128.9, 128.2, 127.9, 127.3, 127.1, 60.5, 60.4, 58.3, 54.6, 33.8, 27.9, 24.3, 21.6, 14.3. **HRMS (ESI)** calcd for  $\text{C}_{22}\text{H}_{27}\text{N}_3\text{NaO}_4\text{S}^+$ : 452.1614 ( $\text{M} + \text{Na}^+$ ), found: 452.1624.



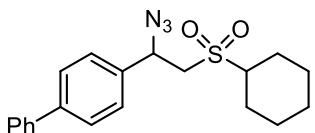
4-(1-Azido-2-(pent-4-yn-1-ylsulfonyl)ethyl)-1,1'-biphenyl (**3aj**, 41.7 mg, 59%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.66 (d, *J* = 8.2 Hz, 2H), 7.61 – 7.57 (m, 2H), 7.49 – 7.41 (m, 4H), 7.38 (t, *J* = 7.3 Hz, 1H), 5.20 (dd, *J* = 10.1, 3.3 Hz, 1H), 3.49 (dd, *J* = 15.0, 10.1 Hz, 1H), 3.31 – 3.24 (m, 2H), 3.16 (dd, *J* = 15.0, 3.3 Hz, 1H), 2.44 – 2.38 (m, 2H), 2.14 – 2.02 (m, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 142.5, 140.0, 135.5, 129.0, 128.2, 127.9, 127.3, 127.1, 81.8, 70.4, 60.5, 58.6, 53.6, 20.9, 17.5. **HRMS (ESI)** calcd for C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 376.1090 (M + Na<sup>+</sup>), found: 376.1099.



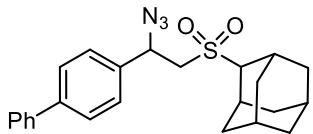
4-(1-Azido-2-((3-iodopropyl)sulfonyl)ethyl)-1,1'-biphenyl (**3ak**, 68.3 mg, 75%), white solid

**<sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)**: δ (ppm) 7.76 – 7.68 (m, 4H), 7.60 (d, *J* = 8.2 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.3 Hz, 1H), 5.28 (dd, *J* = 9.6, 3.2 Hz, 1H), 4.00 (dd, *J* = 14.8, 9.9 Hz, 1H), 3.65 (dd, *J* = 14.8, 3.7 Hz, 1H), 3.36 (t, *J* = 6.9 Hz, 2H), 3.28 (t, *J* = 7.6 Hz, 2H), 2.30 – 2.20 (m, 2H). **<sup>13</sup>C NMR (100 MHz, DMSO-d<sub>6</sub>)**: δ (ppm) 147.1, 141.2, 139.8, 137.3, 131.0, 129.4, 128.5, 128.3, 128.2, 127.7, 127.3, 125.6, 121.5, 120.1, 58.2, 55.4, 52.2, 18.4, 15.8. **HRMS (ESI)** calcd for C<sub>17</sub>H<sub>18</sub>IN<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 478.0057 (M + Na<sup>+</sup>), found: 478.0064.



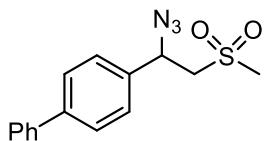
4-(1-Azido-2-(cyclohexylsulfonyl)ethyl)-1,1'-biphenyl (**3al**, 52.5 mg, 71%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.65 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 7.3 Hz, 2H), 7.48 – 7.42 (m, 4H), 7.38 (t, *J* = 7.3 Hz, 1H), 5.20 (dd, *J* = 9.7, 3.6 Hz, 1H), 3.50 (dd, *J* = 14.7, 9.7 Hz, 1H), 3.08 (dd, *J* = 14.7, 3.6 Hz, 1H), 3.02 – 2.92 (m, 1H), 2.18 (t, *J* = 12.9 Hz, 2H), 1.98 – 1.89 (m, 2H), 1.75 – 1.59 (m, 2H), 1.56 – 1.50 (m, 1H), 1.34 – 1.24 (m, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 142.3, 140.1, 135.9, 129.0, 128.1, 127.8, 127.4, 127.1, 62.2, 60.2, 55.0, 25.7, 25.1, 23.9. **HRMS (ESI)** calcd for C<sub>20</sub>H<sub>23</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 392.1403 (M + Na<sup>+</sup>), found: 392.1408.



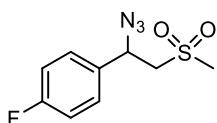
(*1R,3S,5r,7r*)-2-((2-([1,1'-biphenyl]-4-yl)-2-azidoethyl)sulfonyl)adamantane (**3am**, 50.6 mg, 60%), white solid

**$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 7.65 (d,  $J = 8.2$  Hz, 2H), 7.60 – 7.56 (m, 2H), 7.49 – 7.42 (m, 4H), 7.38 (t,  $J = 7.3$  Hz, 1H), 5.19 (dd,  $J = 9.0, 4.0$  Hz, 1H), 3.54 (dd,  $J = 14.8, 9.1$  Hz, 1H), 3.23 – 3.11 (m, 2H), 2.63 – 2.36 (m, 4H), 2.04 – 1.81 (m, 5H), 1.77 – 1.63 (m, 5H).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 142.4, 140.1, 136.0, 128.9, 128.1, 127.8, 127.4, 127.1, 67.3, 60.4, 57.2, 39.0, 38.9, 37.0, 31.8, 31.5, 28.4, 27.6, 27.4, 26.7. **HRMS (ESI)** calcd for  $\text{C}_{24}\text{H}_{27}\text{N}_3\text{NaO}_2\text{S}^+$ : 444.1716 ( $\text{M} + \text{Na}^+$ ), found: 444.1721.



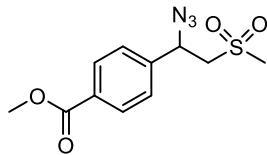
4-(1-Azido-2-(methylsulfonyl)ethyl)-1,1'-biphenyl (**3an**, 42.4 mg, 70%), yellow solid

**$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 7.67 (d,  $J = 8.2$  Hz, 2H), 7.59 (d,  $J = 7.5$  Hz, 2H), 7.49 – 7.37 (m, 5H), 5.20 (dd,  $J = 10.2, 3.3$  Hz, 1H), 3.50 (dd,  $J = 14.9, 10.3$  Hz, 1H), 3.20 (dd,  $J = 14.9, 2.8$  Hz, 1H), 3.04 (s, 3H).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 142.5, 140.0, 135.3, 129.0, 128.2, 127.9, 127.4, 127.1, 60.7, 60.3, 43.1. **HRMS (ESI)** calcd for  $\text{C}_{15}\text{H}_{15}\text{N}_3\text{NaO}_2\text{S}^+$ : 324.0777 ( $\text{M} + \text{Na}^+$ ), found: 324.0781.



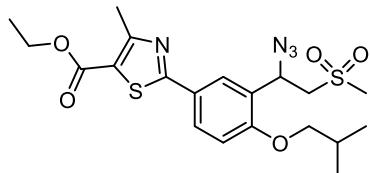
1-(1-Azido-2-(methylsulfonyl)ethyl)-4-fluorobenzene (**3hn**, 25.9 mg, 50%), white solid <sup>4</sup>

**$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 7.39 – 7.33 (m, 2H), 7.18 – 7.12 (m, 2H), 5.15 (dd,  $J = 10.2, 3.4$  Hz, 1H), 3.44 (dd,  $J = 14.9, 10.2$  Hz, 1H), 3.17 – 3.11 (m, 1H), 3.02 (s, 3H).  **$^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) -111.19 – -111.10 (m).  **$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )**:  $\delta$  (ppm) 163.1 (d,  $^1J_{\text{CF}} = 249.4$  Hz), 132.4 (d,  $^4J_{\text{CF}} = 3.3$  Hz), 128.8 (d,  $^3J_{\text{CF}} = 8.4$  Hz), 116.6 (d,  $^2J_{\text{CF}} = 21.8$  Hz), 60.2, 60.2, 43.1.



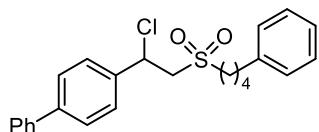
Methyl 4-(1-azido-2-(methylsulfonyl)ethyl)benzoate (**3fn**, 30.2 mg, 53%), white oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 8.12 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 5.22 (dd, *J* = 10.2, 3.2 Hz, 1H), 3.94 (s, 3H), 3.47 (dd, *J* = 14.9, 10.3 Hz, 1H), 3.17 (dd, *J* = 15.0, 2.5 Hz, 1H), 3.05 (s, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 166.2, 141.3, 131.3, 130.8, 127.0, 60.5, 59.9, 52.4, 43.2. **HRMS (ESI)** calcd for C<sub>11</sub>H<sub>13</sub>N<sub>3</sub>NaO<sub>4</sub>S<sup>+</sup>: 306.0519 (M + Na<sup>+</sup>), found: 306.0532.



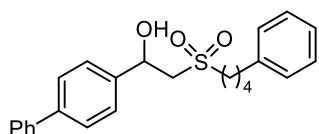
Ethyl 2-(3-(1-azido-2-(methylsulfonyl)ethyl)-4-isobutoxyphenyl)-4-methylthiazole-5-carboxylate (**3un**, 33.5 mg, 36%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 8.01 (d, *J* = 2.1 Hz, 1H), 7.95 – 7.91 (m, 1H), 6.99 (d, *J* = 8.7 Hz, 1H), 5.58 (dd, *J* = 10.5, 2.5 Hz, 1H), 4.39 – 4.33 (m, 2H), 3.91 – 3.83 (m, 2H), 3.48 (dd, *J* = 14.9, 10.6 Hz, 1H), 3.29 – 3.23 (m, 1H), 3.10 (s, 3H), 2.78 (s, 3H), 2.25 – 2.15 (m, 1H), 1.42 – 1.37 (m, 3H), 1.12 – 1.08 (m, 6H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 168.8, 162.3, 161.1, 158.0, 129.1, 126.2, 126.1, 125.6, 121.4, 112.2, 75.2, 61.3, 58.7, 56.1, 43.2, 28.3, 19.3, 19.3, 17.6, 14.4. **HRMS (ESI)** calcd for C<sub>20</sub>H<sub>26</sub>N<sub>4</sub>NaO<sub>5</sub>S<sub>2</sub><sup>+</sup>: 489.1237 (M + Na<sup>+</sup>), found: 489.1245.



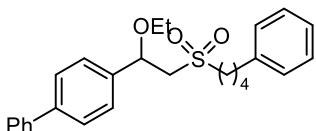
4-(2-Benzylsulfonyl)-1-chloroethyl)-1,1'-biphenyl (**4**, 30.6 mg, 37%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):** δ (ppm) 7.64 – 7.59 (m, 2H), 7.58 – 7.54 (m, 2H), 7.51 – 7.42 (m, 4H), 7.40 – 7.36 (m, 1H), 7.29 – 7.24 (m, 2H), 7.22 – 7.16 (m, 1H), 7.11 (d, *J* = 7.1 Hz, 2H), 5.43 (t, *J* = 6.8 Hz, 1H), 3.81 (dd, *J* = 15.1, 7.4 Hz, 1H), 3.63 (dd, *J* = 15.1, 6.3 Hz, 1H), 2.88 – 2.73 (m, 2H), 2.64 – 2.52 (m, 2H), 1.89 – 1.59 (m, 4H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):** δ (ppm) 142.6, 141.1, 139.9, 137.6, 129.0, 128.5, 128.4, 127.9, 127.9, 127.6, 127.1, 126.1, 61.3, 55.3, 54.3, 35.2, 30.1, 21.6. **HRMS (ESI)** calcd for C<sub>24</sub>H<sub>25</sub>ClNaO<sub>2</sub>S<sup>+</sup>: 435.1156 (M + Na<sup>+</sup>), found: 435.1164.



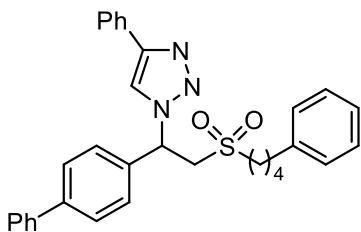
1-([1,1'-Biphenyl]-4-yl)-2-(benzylsulfonyl)ethan-1-ol (**5**, 42.1 mg, 53%), white solid

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.62 – 7.55 (m, 4H), 7.47 – 7.41 (m, 4H), 7.38 – 7.34 (m, 1H), 7.31 – 7.26 (m, 2H), 7.22 – 7.15 (m, 3H), 5.38 (d, *J* = 10.2 Hz, 1H), 3.42 (dd, *J* = 14.6, 10.2 Hz, 1H), 3.20 – 3.08 (m, 4H), 2.66 (t, *J* = 7.5 Hz, 2H), 1.96 – 1.87 (m, 2H), 1.82 – 1.73 (m, 2H).  
**<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 141.6, 141.2, 140.4, 140.0, 128.9, 128.5, 128.4, 127.7, 127.6, 127.1, 126.1, 68.8, 60.4, 54.6, 35.3, 30.2, 21.5. **HRMS (ESI)** calcd for C<sub>24</sub>H<sub>26</sub>NaO<sub>3</sub>S<sup>+</sup>: 417.1495 (M + Na<sup>+</sup>), found: 417.1509.



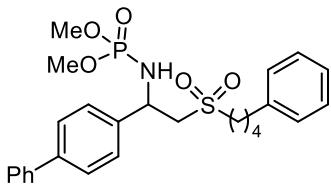
4-(1-Ethoxy-2-((4-phenylbutyl)sulfonyl)ethyl)-1,1'-biphenyl (**6**, 46.2 mg, 55%), white oil

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**: δ (ppm) 7.63 – 7.57 (m, 4H), 7.48 – 7.43 (m, 2H), 7.40 – 7.34 (m, 3H), 7.33 – 7.27 (m, 2H), 7.23 – 7.17 (m, 3H), 4.90 (dd, *J* = 10.4, 2.2 Hz, 1H), 3.53 – 3.38 (m, 3H), 3.29 – 3.14 (m, 2H), 3.00 (dd, *J* = 15.1, 1.2 Hz, 1H), 2.69 (t, *J* = 7.6 Hz, 2H), 1.99 – 1.90 (m, 2H), 1.83 – 1.74 (m, 2H), 1.20 (t, *J* = 7.0 Hz, 3H). **<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**: δ (ppm) 141.6, 141.4, 140.4, 138.1, 128.9, 128.5, 128.4, 127.7, 127.6, 127.1, 126.9, 126.1, 76.7, 64.6, 60.1, 55.1, 35.4, 30.4, 21.7, 15.2. **HRMS (ESI)** calcd for C<sub>26</sub>H<sub>30</sub>NaO<sub>3</sub>S<sup>+</sup>: 445.1808 (M + Na<sup>+</sup>), found: 445.1816.



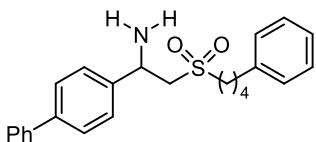
1-(1-([1,1'-Biphenyl]-4-yl)-2-((4-phenylbutyl)sulfonyl)ethyl)-4-phenyl-1*H*-1,2λ<sup>2</sup>,3λ<sup>2</sup>-triazole (**9**, 140.9 mg, 90%), white solid

**<sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)**: δ (ppm) 8.93 (s, 1H), 7.85 (d, *J* = 7.3 Hz, 2H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 7.9 Hz, 4H), 7.49 – 7.44 (m, 4H), 7.41 – 7.33 (m, 2H), 7.25 (t, *J* = 7.3 Hz, 2H), 7.19 – 7.11 (m, 3H), 6.46 (dd, *J* = 9.5, 4.4 Hz, 1H), 4.67 (dd, *J* = 14.8, 9.6 Hz, 1H), 4.25 (dd, *J* = 14.8, 4.4 Hz, 1H), 3.07 – 2.99 (m, 2H), 2.56 – 2.52 (m, 2H), 1.73 – 1.64 (m, 2H), 1.62 – 1.54 (m, 2H). **<sup>13</sup>C NMR (100 MHz, DMSO-d<sub>6</sub>)**: δ (ppm) 147.1, 142.0, 141.2, 139.8, 137.3, 130.9, 129.5, 128.7, 128.6, 128.3, 128.3, 127.7, 127.2, 126.3, 125.6, 121.5, 58.5, 55.5, 53.1, 34.9, 30.0, 21.1. **HRMS (ESI)** calcd for C<sub>32</sub>H<sub>31</sub>N<sub>3</sub>NaO<sub>2</sub>S<sup>+</sup>: 544.2029 (M + Na<sup>+</sup>), found: 544.2036.



Dimethyl (1-([1,1'-biphenyl]-4-yl)-2-((4-phenylbutyl)sulfonyl)ethyl)phosphoramide (**10**, 144.5 mg, 96%), white solid

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.63 – 7.53 (m, 4H), 7.47 – 7.41 (m, 4H), 7.36 (t,  $J$  = 7.3 Hz, 1H), 7.23 (d,  $J$  = 7.5 Hz, 2H), 7.17 (t,  $J$  = 7.2 Hz, 1H), 7.07 (d,  $J$  = 7.0 Hz, 2H), 4.92 – 4.81 (m, 1H), 4.47 (t,  $J$  = 9.8 Hz, 1H), 3.72 (d,  $J$  = 11.2 Hz, 3H), 3.57 – 3.48 (m, 4H), 3.38 (dd,  $J$  = 14.7, 5.0 Hz, 1H), 2.64 – 2.49 (m, 4H), 1.76 – 1.69 (m, 2H), 1.60 – 1.51 (m, 2H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 141.3, 141.1, 140.0, 139.3, 128.9, 128.5, 128.3, 127.7, 127.7, 127.0, 126.9, 126.1, 59.3, 54.1, 53.6, 53.3, 51.3, 35.2, 30.0, 21.4. **HRMS (ESI)** calcd for  $\text{C}_{26}\text{H}_{32}\text{NPNaO}_5\text{S}^+$ : 524.1631 ( $\text{M} + \text{Na}^+$ ), found: 524.1636.



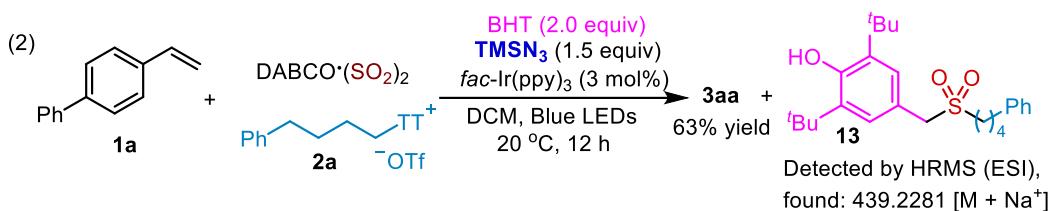
1-([1,1'-Biphenyl]-4-yl)-2-((4-phenylbutyl)sulfonyl)ethan-1-amine (**11**, 25.2 mg, 64%), white solid

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.60 – 7.55 (m, 4H), 7.46 – 7.42 (m, 4H), 7.36 (t,  $J$  = 7.3 Hz, 1H), 7.29 – 7.25 (m, 2H), 7.21 – 7.13 (m, 3H), 4.72 (dd,  $J$  = 9.5, 2.4 Hz, 1H), 3.32 (dd,  $J$  = 14.0, 9.7 Hz, 1H), 3.19 (dd,  $J$  = 14.0, 2.9 Hz, 1H), 3.01 (dd,  $J$  = 9.2, 6.6 Hz, 2H), 2.63 (t,  $J$  = 7.5 Hz, 2H), 2.10 – 2.01 (m, 2H), 1.91 – 1.82 (m, 2H), 1.77 – 1.69 (m, 2H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 141.2, 141.2, 140.4, 128.9, 128.5, 128.4, 127.7, 127.6, 127.1, 126.8, 126.1, 54.4, 35.3, 31.5, 30.2, 30.1, 21.7. **HRMS (ESI)** calcd for  $\text{C}_{24}\text{H}_{27}\text{NNaO}_2\text{S}^+$ : 416.1655 ( $\text{M} + \text{Na}^+$ ), found: 416.1665.

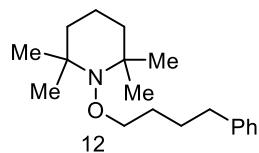
### 3. Radical trapping experiments



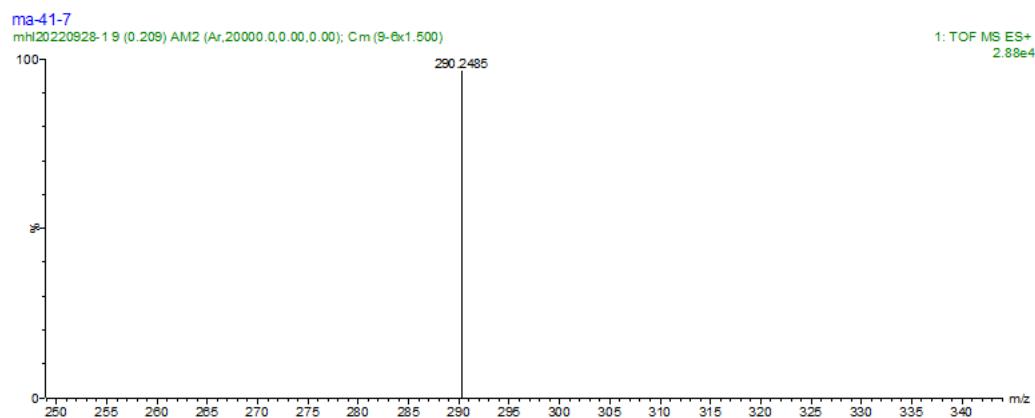
TMSN<sub>3</sub> (0.3 mmol) was added to a mixture of alkenes **1a** (0.2 mmol), thianthrenium salts **2a** (0.4 mmol), DABCO·(SO<sub>2</sub>)<sub>2</sub> (0.16 mmol), *fac*-Ir(ppy)<sub>3</sub> (3 mol%) and TEMPO (0.4 mmol) in DCM (3 mL) under N<sub>2</sub> atmosphere. The mixture was placed around a 30 W blue LEDs and stirred under blue light irradiation for 12 hours at 20 °C. As a result, significant inhibition of the reactivity was observed, and the corresponding trapping product **12** was detected by HRMS.

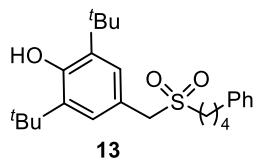


TMSN<sub>3</sub> (0.3 mmol) was added to a mixture of alkenes **1a** (0.2 mmol), thianthrenium salts **2a** (0.4 mmol), DABCO·(SO<sub>2</sub>)<sub>2</sub> (0.16 mmol), *fac*-Ir(ppy)<sub>3</sub> (3 mol%) and BHT (0.4 mmol) in DCM (3 mL) under N<sub>2</sub> atmosphere. The mixture was placed around a 30 W blue LEDs and stirred under blue light irradiation for 12 hours at 20 °C. As a result, the isolated yield of **3aa** was decreased to 63% and another radical trapping adduct **13** was discovered by HRMS.

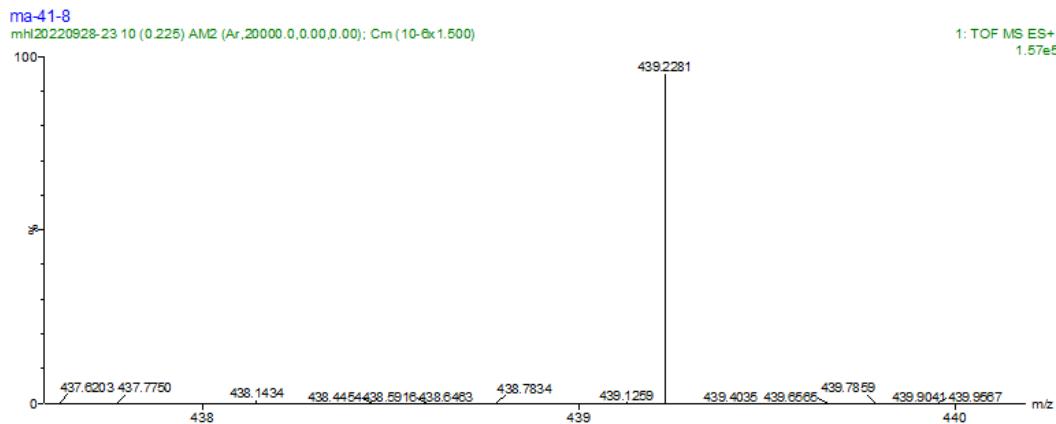


**HRMS (ESI)** calcd for C<sub>19</sub>H<sub>32</sub>NO<sup>+</sup>: 290.2478 (M + H<sup>+</sup>), found: 290.2485.





**HRMS (ESI)** calcd for C<sub>25</sub>H<sub>36</sub>NaO<sub>3</sub>S<sup>+</sup>: 439.2277 (M + Na<sup>+</sup>), found: 439.2281.



#### 4. Reference

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5.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compounds 3, 4, 5, 6, 9, 10, 11.

