Synthesis of (E)-β-iodo vinylsulfones via iodine-promoted
iodosulfonylation of alkynes with sodium sulfinates in an aqueous
medium at room temperature

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A. General method

Melting points were measured with a melting point instrument and were uncorrected. $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker Avance (400 and 100 MHz, respectively) instrument internally referenced to tetramethylsilane (TMS) or chloroform signals. GC-MS was obtained using electron ionization (EI). High-resolution mass spectra were obtained with a LCMS-IT-TOF mass spectrometer. Single-crystal X-ray analysis was obtained using Bruker APEX2 Smart CCD. TLC was performed by using commercially prepared 100–400 mesh silica gel plates (GF254) and visualization was effected at 254 nm. All reagents and solvents were purchased from commercial sources (Adamas-beta, TCI, Alfa Aesar and Ark) and used without further purification.

B. General procedure for the synthesis of products

\[
R^1\equiv \equiv R^2 + R^3\text{-SO}_2\text{Na} \xrightarrow{I_2, H_2O, rt} \quad 1 \quad 2
\]

A mixture of sodium sulfinates (0.60 mmol), alkyne (0.30 mmol), and iodine (0.45 mmol) in water (2.0 mL) was placed in a test tube (25 mL) equipped with a magnetic stirring bar. The reaction mixture was stirred at room temperature for 2h. After the reaction was completed, the mixture was quenched by the addition of satd aq Na$_2$S$_2$O$_3$ (5 mL). Further stirring was followed by extraction with ethyl acetate (2 × 15 mL). The organic layer was dried with anhydrous MgSO$_4$, concentrated in vacuo and purified by flash silica gel chromatography using petroleum ether/ethyl acetate 20:1 to give the desired products.

C. Control experiments for the study of mechanism

\[
\text{Ph}\equiv \equiv + \quad \begin{array}{c} \text{Ph} \\ 1a \end{array} \quad \xrightarrow{\text{Standard Conditions}} \quad \begin{array}{c} \text{Ph} \\ 2a \end{array} \quad \xrightarrow{\text{BHT}} \quad \begin{array}{c} \text{Ph} \\ 3aa, 86\% \end{array}
\]

\[
\text{Ph}\equiv \equiv + \quad \begin{array}{c} \text{Ph} \\ 1a \end{array} \quad \xrightarrow{\text{Standard Conditions}} \quad \begin{array}{c} \text{Ph} \\ 2a \end{array} \quad \xrightarrow{\text{TEMPO}} \quad \begin{array}{c} \text{Ph} \\ 3aa, \text{not detected} \end{array}
\]
A mixture of 2a (0.60 mmol), 1a (0.30 mmol), iodine (0.45 mmol) and BHT (0.30 mmol) in water (2.0 mL) was placed in a test tube (25 mL) equipped with a magnetic stirring bar. The reaction mixture was stirred at room temperature for 2h. After the reaction was completed, the mixture was quenched by the addition of satd aq Na$_2$S$_2$O$_3$ (5 mL). Further stirring was followed by extraction with ethyl acetate ($2 \times 15$ mL). The organic layer was dried with anhydrous MgSO$_4$, concentrated in vacuo and purified by flash silica gel chromatography using petroleum ether/ethyl acetate 20:1 to give 3aa in 86% yield.

A mixture of 2a (0.60 mmol), 1a (0.30 mmol), iodine (0.45 mmol) and TEMPO (0.30 mmol) in water (2.0 mL) was placed in a test tube (25 mL) equipped with a magnetic stirring bar. The reaction mixture was stirred at room temperature for 2h. After the reaction was completed, the mixture was quenched by the addition of satd aq Na$_2$S$_2$O$_3$ (5 mL). Further stirring was followed by extraction with ethyl acetate ($2 \times 15$ mL). The organic layer was dried with anhydrous MgSO$_4$, concentrated in vacuo and the crude product was detected by GC-MS.
D. Single-crystal X-ray analysis of 3aa

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![Diagram of molecular structure](image-url)
E. Analytical data for 3aa-3la, 4, 5 and 6.

![Chemical structure image]

**(E)-1-((2-iodo-2-phenylvinyl)sulfonyl)-4-methylbenzene (3aa)**.\(^1\) white solid (99.1 mg, 86%); mp 80–81 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45 (d, \(J = 8.3\) Hz, 2H), 7.36 (s, 1H), 7.32 – 7.25 (m, 3H), 7.23 (dt, \(J = 3.7, 2.1\) Hz, 2H), 7.18 (d, \(J = 8.6\) Hz, 2H), 2.39 (s, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 144.5, 141.2, 139.6, 137.2, 129.7, 129.6, 127.8, 127.8, 127.6, 114.1, 21.5.

![Chemical structure image]

**(E)-1-ethyl-4-(1-iodo-2-tosylvinyl)benzene (3ab)**. white solid (107.6 mg, 87%); mp 91–92 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45 (d, \(J = 8.3\) Hz, 2H), 7.34 (s, 2H), 7.18 – 7.12 (m, 4H), 7.09 (d, \(J = 8.5\) Hz, 2H), 2.63 (q, \(J = 7.6\) Hz, 2H), 2.36 (s, 3H), 1.24 (t, \(J = 7.6\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 146.1, 144.2, 140.6, 137.0, 136.7, 129.3, 127.7, 127.6, 127.1, 114.6, 28.5, 21.4, 15.1; ESI-HRMS calcld for C\(_{17}\)H\(_{17}\)IO\(_2\)S (M + H\(^+\)) 413.0067; found 413.0059.

![Chemical structure image]

**(E)-1-butyl-4-(1-iodo-2-tosylvinyl)benzene (3ac)**. Yellow liquid (116.2 mg, 88%); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45 (d, \(J = 10.4\) Hz, 2H), 7.34 (s, 1H), 7.15 (d, \(J = 8.3\) Hz, 4H), 7.07 (d, \(J = 8.1\) Hz, 2H), 2.60(t, \(J = 7.7\) Hz, 2H), 2.37 (s, 3H), 1.64 – 1.56 (m, 2H), 1.43 – 1.33 (m, 2H), 0.96 (t, \(J = 7.3\) Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 145.0, 144.3, 140.8, 137.2, 136.7, 129.4, 127.7, 127.7, 127.6, 114.8, 35.4, 33.3, 22.2, 21.5, 13.9; ESI-HRMS calcld for C\(_{19}\)H\(_{23}\)IO\(_2\)S (M + H\(^+\)) 441.0380; found 441.0385.
(E)-1-fluoro-4-(1-iodo-2-tosylvinyl)benzene (3ad).<sup>1</sup> White solid (90.5 mg, 75%); mp 91–92 °C; <sup>1</sup>H NMR (400 MHz, CDCl₃) δ 7.49 (d, J = 8.2 Hz, 2H), 7.35 (s, 1H), 7.28 – 7.20 (m, 4H), 6.98 (t, J = 8.6 Hz, 2H), 2.40 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl₃) δ 163.1 (d, J = 251.4 Hz), 144.7, 141.6, 137.1, 135.6 (d, J = 3.5 Hz), 129.9 (d, J = 8.7 Hz), 129.7, 127.7, 115.0 (d, J = 22.1 Hz), 112.5, 21.6.

(E)-1-chloro-4-(1-iodo-2-tosylvinyl)benzene (3ae).<sup>1</sup> White solid (96.7 mg, 77%); mp 146–147 °C; <sup>1</sup>H NMR (400 MHz, CDCl₃) δ 7.50 (d, J = 8.3 Hz, 2H), 7.34 (s, 1H), 7.29 – 7.22 (m, 4H), 7.21 – 7.16 (m, 2H), 2.42 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl₃) δ 144.8, 141.7, 138.0, 137.1, 135.8, 129.7, 129.0, 128.1, 127.8, 112.0, 21.6.

(E)-1-bromo-4-(1-iodo-2-tosylvinyl)benzene (3af).<sup>2</sup> White solid (109.8 mg, 79%); mp 156–157 °C; <sup>1</sup>H NMR (400 MHz, CDCl₃) δ 7.52 – 7.47 (m, 2H), 7.45 – 7.39 (m, 2H), 7.34 (s, 1H), 7.23 (d, J = 7.9 Hz, 2H), 7.14 – 7.09 (m, 2H), 2.42 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl₃) δ 144.8, 141.7, 138.5, 137.0, 131.1, 129.7, 129.2, 127.8, 124.1, 111.9, 21.6.
(E)-1-fluoro-2-(1-iodo-2-tosylviny]benzene (3ag). white solid (94.1 mg, 78%); mp 120–121 °C; 1H NMR (400 MHz, CDCl3) δ 7.53 (d, J = 8.4 Hz, 2H), 7.40 (s, 1H), 7.36 – 7.30 (m, 1H), 7.25 – 7.20 (m, 3H), 7.16 – 7.12 (m, 1H), 6.99 – 6.94 (m, 1H), 2.41 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 157.1 (d, J = 250.8 Hz), 144.8, 142.8, 136.6, 131.5 (d, J = 8.2 Hz), 129.7, 129.1 (d, J = 1.7 Hz), 127.8, 127.4 (d, J = 15.3 Hz), 123.7 (d, J = 3.6 Hz), 115.6 (d, J = 20.7 Hz), 104.4, 21.5; ESI-HRMS calcd for C15H12FIO2S (M + Na)+ 424.9479; found 424.9470.

(E)-3-(1-iodo-2-tosylviny]phenol (3ah). white solid (104.5 mg, 87%); mp 132–133 °C; 1H NMR (400 MHz, CDCl3) δ 7.50 (d, J = 8.3 Hz, 2H), 7.34 (s, 2H), 7.20 (d, J = 8.4 Hz, 2H), 7.11 (t, J = 7.9 Hz, 1H), 6.80 – 6.71 (m, 2H), 6.70 – 6.65 (m, 1H), 6.11 (s, 1H), 2.38 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 155.1, 144.8, 140.8, 140.5, 136.7, 129.7, 129.2, 127.8, 119.7, 117.1, 114.5, 113.9, 21.6; ESI-HRMS calcd for C15H13IO3S (M + Na)+ 422.9522; found 422.9516.

(E)-1-((2-iodo-2-(4-methoxyphenyl)vinyl)sulfonyl)-4-methylbenzene (3ai).2 Yellow liquid (113.1 mg, 91%); 1H NMR (400 MHz, CDCl3) δ 7.50 (d, J = 8.3 Hz, 2H), 7.34 (s, 2H), 7.20 (d, J = 8.4 Hz, 2H), 7.11 (t, J = 7.9 Hz, 1H), 6.80 – 6.71 (m, 2H), 6.70 – 6.65 (m, 1H), 6.11 (s, 1H), 2.38 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 160.7, 144.4, 140.1, 137.3, 131.7, 129.8, 129.5, 127.7, 114.8, 113.1, 55.3, 21.5;

(E)-1-ethoxy-4-(1-iodo-2-tosylviny]benzene (3aj): Yellow liquid (119.5 mg, 93%); 1H NMR (400 MHz, CDCl3) δ 7.51 – 7.45 (m, 2H), 7.28 (s, 1H), 7.25 – 7.21 (m, 2H), 7.18 (dd, J = 8.4, 0.5 Hz, 2H), 6.79 – 6.73 (m, 2H), 4.02 (q, J = 7.0 Hz, 2H), 2.37 (s, 3H), 1.41 (t, J = 7.0 Hz, 3H); 13C
NMR (100 MHz, CDCl₃) δ 160.0, 144.3, 140.0, 137.2, 131.4, 129.8, 129.4, 127.6, 115.0, 113.4, 63.4, 21.4, 14.5; ESI-HRMS calcd for C₁₇H₁₇IO₃S (M + Na)⁺ 450.9835; found 450.9828.

(E)-2-(1-iodo-2-tosylvinyl)thiophene (3ak). Yellow solid (106.5 mg, 91%); mp 95–96 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, J = 8.3 Hz, 2H), 7.53 (dd, J = 3.7, 1.2 Hz, 1H), 7.49 (dd, J = 5.1, 1.2 Hz, 1H), 7.31 (s, 1H), 7.24 – 7.22 (m, 1H), 7.21 (d, J = 0.7 Hz, 1H), 7.00 (dd, J = 5.1, 3.7 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.6, 141.0, 140.8, 136.9, 131.3, 130.0, 129.6, 127.6, 127.3, 103.4, 21.5.

(E)-3-(1-iodo-2-tosylvinyl)pyridine (3al). Yellow solid (94.8 mg, 82%); mp 140–141 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.56 (dd, J = 4.9, 1.5 Hz, 1H), 8.46 (d, J = 2.2 Hz, 1H), 7.66 – 7.62 (m, 1H), 7.52 (d, J = 8.4 Hz, 2H), 7.44 (s, 1H), 7.31 – 7.26 (m, 3H), 2.42 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 150.1, 147.3, 145.1, 142.8, 136.9, 136.1, 135.4, 130.0, 127.8, 122.7, 108.7, 21.6.

(E)-1-((2-cyclopropyl-2-iodovinyl)sulfonyl)-4-methylbenzene (3am). White solid (88.8 mg, 85%); mp 121–122 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.83 – 7.75 (m, 2H), 7.35 – 7.33 (m, 2H), 7.03 (d, J = 2.3 Hz, 1H), 2.46 – 2.39 (m, 4H), 0.94 – 0.80 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 144.5, 138.2, 137.7, 133.4, 129.9, 127.2, 21.6, 17.2, 12.0; ESI-HRMS calcd for C₁₂H₁₃IO₂S (M + Na)⁺ 370.9573; found 370.9577.

S8
(E)-1-((2-iodohex-1-en-1-yl)sulfonyl)-4-methylbenzene (3an). Yellow liquid (85.2 mg, 82%); ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, J = 8.3 Hz, 2H), 7.33 (dd, J = 8.6, 0.6 Hz, 2H), 6.97 (s, 1H), 3.04 – 2.97 (m, 2H), 2.43 (s, 3H), 1.55 – 1.44 (m, 2H), 1.41 – 1.31 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.7, 138.8, 138.0, 130.0, 127.4, 125.4, 39.7, 31.9, 21.6, 21.6, 13.82.

(E)-1-(((1-iodo-1-phenylprop-1-en-2-yl)sulfonyl)-4-methylbenzene (3ao). White solid (76.5 mg, 64%); mp 129–130 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, J = 8.3 Hz, 2H), 7.25 – 7.20 (m, 3H), 7.16 (d, J = 7.9 Hz, 2H), 7.13 – 7.07 (m, 2H), 2.51 (s, 3H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.1, 143.8, 142.9, 137.2, 129.4, 128.6, 127.7, 127.6, 127.5, 115.7, 27.0, 21.5.

(E)-ethyl 3-iodo-3-phenyl-2-tosylacrylate (3ap): Yellow liquid (69.8 mg, 51%); ¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, J = 8.1 Hz, 2H), 7.29 (m, 1H), 7.24 (m, 2H), 7.13 (d, J = 8.4 Hz, 2H), 7.08 (m, 2H), 4.44 (q, J = 7.2 Hz, 2H), 2.39 (s, 3H), 1.44 (t, J = 7.1 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 163.6, 146.4, 144.8, 139.5, 137.0, 129.5, 129.3, 128.2, 127.7, 127.3, 114.0, 63.2, 21.6, 13.9; ESI-HRMS calcd for C₁₉H₁₇IO₄S (M + Na)⁺ 478.9784; found 478.9775.

S9
(E)-(1-iodo-2-(phenylsulfonyl)vinyl)benzene (3ba).² white solid (95.5 mg, 86%); mp 66–67 °C; H NMR (400 MHz, CDCl₃) δ 7.58 – 7.49 (m, 3H), 7.39 (s, 1H), 7.39 – 7.33 (m, 2H), 7.31 – 7.23 (m, 3H), 7.22 – 7.19 (m, 2H); C NMR (100 MHz, CDCl₃) δ 140.8, 139.9, 139.3, 133.3, 129.6, 128.8, 127.7, 127.5, 127.4, 114.6.

(E)-1-((2-iodo-2-phenylvinyl)sulfonyl)-2-methylbenzene (3ca). white solid (100.3 mg, 87%); mp 72–73 °C; H NMR (400 MHz, CDCl₃) δ 7.46 – 7.41 (m, 2H), 7.37 – 7.33 (m, 1H), 7.25 – 7.12 (m, 2H), 7.06 – 7.00 (m, 1H), 2.60 (s, 3H); C NMR (100 MHz, CDCl₃) δ 140.9, 139.1, 138.1, 137.4, 133.2, 132.0, 129.6, 129.2, 127.7, 127.4, 126.0, 114.2, 20.3; ESI-HRMS calcd for C₁₅H₁₁O₂S (M + H)⁺ 384.9754; found 384.9751.

(E)-1-((2-iodo-2-phenylvinyl)sulfonyl)-4-methoxybenzene (3da). white solid (102.1 mg, 85%); mp 111–112 °C; H NMR (400 MHz, CDCl₃) δ 7.47 (d, J = 11.9 Hz, 2H), 7.37 (s, 1H), 7.32 – 7.20 (m, 5H), 6.85 – 6.79 (m, 2H), 3.81 (s, 3H); C NMR (100 MHz, CDCl₃) δ 163.4, 141.4, 139.5, 131.4, 129.8, 129.5, 127.7, 127.5, 114.1, 113.5, 55.5.

(E)-1-fluoro-3-((2-iodo-2-phenylvinyl)sulfonyl)benzene (3ea): white solid (96.7 mg, 83%); mp 92–93 °C; H NMR (400 MHz, CDCl₃) δ 7.39 (s, 1H), 7.38 – 7.37 (m, 2H), 7.35 – 7.29 (m, 2H), 7.28 – 7.27 (m, 1H), 7.26 – 7.21 (m, 1H), 7.19 (m, 3H); C NMR (100 MHz, CDCl₃) δ 162.0 (d, J = 252.3 Hz), 142.0 (d, J = 6.6 Hz), 140.5, 139.2, 130.7 (d, J = 7.6 Hz), 129.9, 127.9, 127.4, 123.5 (d, J = 3.3 Hz), 120.6 (d, J = 21.2 Hz), 115.5, 115.1 (d, J = 24.5 Hz); ESI-HRMS calcd for C₁₄H₁₀FIO₂S (M + Na)⁺ 410.9322; found 410.9328.
(E)-1-chloro-4-((2-iodo-2-phenylvinyl)sulfonyl)benzene (3fa): white solid (99.5 mg, 82%); mp 102–103 °C; 1H NMR (400 MHz, CDCl3) δ 7.45 (d, J = 8.6 Hz, 2H), 7.39 (s, 1H), 7.34 – 7.23 (m, 5H), 7.21 – 7.14 (m, 2H); 13C NMR (100 MHz, CDCl3) δ 140.7, 139.9, 139.2, 138.3, 129.7, 129.0, 129.0, 127.8, 127.4, 115.1.

(E)-1-bromo-3-((2-iodo-2-phenylvinyl)sulfonyl)benzene (3ga): white solid (114.5 mg, 85%); mp 59–60 °C; 1H NMR (400 MHz, CDCl3) δ 7.65 – 7.61 (m, 1H), 7.55 (t, J = 1.7 Hz, 1H), 7.52 – 7.49 (m, 1H), 7.40 (s, 1H), 7.36 – 7.31 (m, 1H), 7.31 – 7.24 (m, 3H), 7.19 – 7.15 (m, 2H); 13C NMR (100 MHz, CDCl3) δ 141.8, 140.7, 139.1, 136.3, 130.7, 130.3, 130.0, 127.9, 127.4, 126.2, 122.7, 115.6; ESI-HRMS calcd for C14H10BrIO2S (M + Na)+ 470.8522; found 470.8511.

(E)-1-chloro-2-((2-iodo-2-phenylvinyl)sulfonyl)benzene (3ha): white solid (102.0 mg, 84%); mp 100–101 °C; 1H NMR (400 MHz, CDCl3) δ 7.57 (s, 1H), 7.48 – 7.44 (m, 1H), 7.42 – 7.37 (m, 2H), 7.22 – 7.16 (m, 1H), 7.15 – 7.07 (m, 5H); 13C NMR (100 MHz, CDCl3) δ 140.3, 139.2, 137.9, 134.2, 132.3, 131.3, 130.7, 129.7, 127.7, 127.3, 126.8, 114.8; ESI-HRMS calcd for C14H10ClIO2S (M + Na)+ 426.9027; found 426.9021.

(E)-1-chloro-3-((2-iodo-2-phenylvinyl)sulfonyl)benzene (3ia): white solid (98.3 mg, 81%); mp 63–64 °C; 1H NMR (400 MHz, CDCl3) δ 7.49 – 7.43 (m, 2H), 7.41 – 7.40 (m, 2H), 7.35 – 7.25 (m,
4H), 7.20 – 7.15 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 141.6, 140.7, 139.1, 134.9, 133.4, 130.1, 130.0, 127.9, 127.9, 127.3, 125.7, 115.6; ESI-HRMS calcd for C$_{14}$H$_{10}$ClIO$_2$S (M + Na)$^+$ 426.9027; found 426.9030.

(E)-(1-iodo-2-(methylsulfonyl)vinyl)benzene (3ga)$^7$ white solid (75.8 mg, 82%); mp 81–82 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.47 – 7.43 (m, 2H), 7.41 – 7.36 (m, 3H), 7.30 (s, 1H), 2.65 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 140.1, 139.3, 130.2, 128.2, 127.7, 114.8, 42.9.

(E)-(2-(ethylsulfonyl)-1-iodovinyl)benzene (3ka); white solid (82.2 mg, 85%); mp 76–77 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.48 – 7.41 (m, 2H), 7.41 – 7.34 (m, 3H), 7.20 (s, 1H), 2.71 (q, $J = 7.4$ Hz, 2H), 1.26 (t, $J = 7.4$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.3, 137.9, 130.1, 128.0, 127.6, 115.4, 49.0, 6.6; ESI-HRMS calcd for C$_{10}$H$_{11}$IO$_2$S (M + H)$^+$ 322.9597; found 322.9593.

(E)-(2-(cyclopropylsulfonyl)-1-iodovinyl)benzene (3la). white solid (84.2 mg, 84%); mp 73–74 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.46 – 7.43 (m, 2H), 7.39 – 7.33 (m, 3H), 7.30 (s, 1H), 2.17 – 2.10 (m, 1H), 1.13 – 1.06 (m, 2H), 0.93 – 0.85 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.8, 139.3, 129.9, 127.9, 127.7, 113.9, 31.6, 5.2; ESI-HRMS calcd for C$_{11}$H$_{11}$IO$_2$S (M + H)$^+$ 334.9597; found 334.9599.

(E)-(4-(phenylsulfonyl)but-3-en-1-yne-1,3-diyl)dibenzene (4).$^1$ white solid (155.9 mg, 87%); mp 82–83 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.56 (d, $J = 8.3$ Hz, 2H), 7.52 – 7.46 (m, 2H), 7.44 – 7.30 (m, 8H), 7.20 (d, $J = 8.4$ Hz, 2H), 6.96 (s, 1H), 2.39 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ

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144.1, 137.9, 136.7, 135.3, 134.1, 131.8, 129.5, 129.4, 128.9, 128.3, 127.8, 127.6, 121.4, 97.2, 88.3, 21.5.

(2-(phenylsulfonyl)ethene-1,1-diyldibenzene (5).) Yellow solid (37.1 mg, 82%); mp 98–99 °C; \( ^1 \)H NMR (400 MHz, CDCl₃) \( \delta \) 7.48 (d, \( J = 8.4 \) Hz, 2H), 7.40 – 7.33 (m, 2H), 7.32 – 7.28 (m, 4H), 7.23 – 7.18 (m, 2H), 7.18 – 7.07 (m, 4H), 7.01 (s, 1H), 2.38 (s, 3H); \( ^{13} \)C NMR (100 MHz, CDCl₃) \( \delta \) 154.5, 143.6, 139.0, 138.4, 135.4, 130.1, 129.6, 129.2, 128.8, 128.7, 128.4, 128.0, 127.6, 127.5, 21.4.

1-methyl-4-((phenylethynyl)sulfonyl)benzene (6). White solid (108.9 mg, 85%); mp 81–82 °C; \( ^1 \)H NMR (400 MHz, CDCl₃) \( \delta \) 7.96 (d, \( J = 8.4 \) Hz, 2H), 7.54 – 7.49 (m, 2H), 7.49 – 7.44 (m, 1H), 7.41 – 7.34 (m, 4H), 2.47 (s, 3H); \( ^{13} \)C NMR (100 MHz, CDCl₃) \( \delta \) 145.3, 138.9, 132.6, 131.4, 129.9, 128.6, 127.4, 117.9, 92.9, 85.5, 21.7.

References

F. NMR Spectra

$^1$H-NMR and $^{13}$C-NMR of 3aa
$^1$H-NMR and $^{13}$C-NMR of 3ab
$^1$H-NMR and $^{13}$C-NMR of 3ac
$^1$H-NMR and $^{13}$C-NMR of 3ad
$^1$H-NMR and $^{13}$C-NMR of 3ae
$^1$H-NMR and $^{13}$C-NMR of 3af
$^1$H-NMR and $^{13}$C-NMR of 3ag
$^1$H-NMR and $^{13}$C-NMR of 3ah
$^1$H-NMR and $^{13}$C-NMR of 3ai
$^1$H-NMR and $^{13}$C-NMR of 3aj
$^1$H-NMR and $^{13}$C-NMR of 3ak
$^1$H-NMR and $^{13}$C-NMR of 3al
$^1$H-NMR and $^{13}$C-NMR of 3am
$^1$H-NMR and $^{13}$C-NMR of 3an
$^1$H-NMR and $^{13}$C-NMR of 3ao
$^{1}$H-NMR and $^{13}$C-NMR of 3ap
$^1$H-NMR and $^{13}$C-NMR of 3ba
$^1$H-NMR and $^{13}$C-NMR of 3ca
$^1$H-NMR and $^{13}$C-NMR of 3da
$^1\text{H-NMR and } ^{13}\text{C-NMR of 3ea}$
$^1$H-NMR and $^{13}$C-NMR of 3fa
$^1$H-NMR and $^{13}$C-NMR of 3ga
$^1$H-NMR and $^{13}$C-NMR of 3ha
$^1$H-NMR and $^{13}$C-NMR of 3ia
$^1$H-NMR and $^{13}$C-NMR of 3ja
$^1$H-NMR and $^{13}$C-NMR of 3ka

![H-NMR Spectrum](image1)

![C-NMR Spectrum](image2)

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$^1$H-NMR and $^{13}$C-NMR of 3la
$^1$H-NMR and $^{13}$C-NMR of 4
$^{1}H$-NMR and $^{13}C$-NMR of 5
$^1$H-NMR and $^{13}$C-NMR of 6