

*Doyle-Kirmse Reaction Using 3,3-Difluoroallyl Sulfide and N-Sulfonyl-1,2,3-triazole:
An Efficient Access to gem-Difluoroallylated Multifunctional Quaternary Carbon*

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Supporting Information

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General All other reactions were performed in a 10 mL test tube for 10 h. For chromatography, 300-400 mesh silica gel (Qingdao, China) was employed. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were measured in CDCl_3 and recorded on Bruker ARX 600 or 500 spectrometer. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of CDCl_3 (7.26), to the carbon resonance of CDCl_3 (77.16). Coupling constants (J) were given in Hertz (Hz). The term m, q, t, d, s referred to multiplet, quartet, triplet, doublet, singlet. Exact masses (HRMS) were recorded on a high resolution magnetic mass spectrometer using electrospray ionization (ESI) techniques. *N*-sulfonyl-1,2,3-triazole **1**^[1] and 3,3-difluoroallyl sulfides **2**^[2] were prepared according to previous reported procedures. Materials obtained from commercial suppliers were used without further purification.

Representative experimental procedure for the synthesis of gem-difluoroallyl imines

3: To a 10 mL test tube equipped with a magnetic stir bar was charged with 4-(4-chlorophenyl)-1-tosyl-1*H*-1,2,3-triazole **1a** (0.15 mmol), phenyl 2-bromo-3,3-difluoroallyl sulfide **2a** (0.15 mmol), and 4.5 mL of DCE. The solution was stirred at 120 °C under argon for 10 h. Upon completion of the reaction, the solvent was evaporated under vacuum, the crude product was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20/1) as the eluant, giving the pure product **3a** as a white solid (76.2 mg, 89% yield).

Experimental procedure for the synthesis of N-(4-bromo-2-(4-chlorophenyl)-3,3-

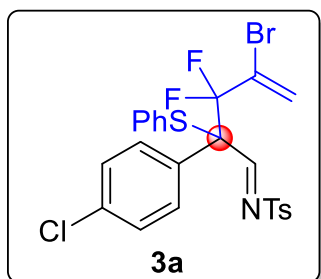
difluoro-2-(phenylthio)pent-4-en-1-yl)-4-methylbenzenesulfonamide 4: Sodium cyanoborohydride (NaBH₃CN, 2 equiv.) was added to a solution of *gem*-difluoroallyl imines (0.5 mmol) in dry THF (10 mL) at room temperature. Subsequently, the reaction mixture was stirred at room temperature for 12 h. After the completion, the reaction mixture was quenched with water and extracted with ethyl acetate. Evaporation of solvent followed purification of the crude product was purified by flash chromatography to give 143.2 mg (50%) of pure amine **4**.

Experimental procedure for the synthesis of tetrahydropyrrole with multiple

substitutions 5: To the mixture of CuI (5 mol%), TMEDA (10 mol%) and K₃PO₄ (2 equiv.), amine **4** (0.15 mmol) and Toluene (3 mL) were added in 10 mL microwave tube under at room temperature. The mixture was stirred at 75 °C until **4** was completely consumed by TLC. Upon completion of the reaction, the solvent was evaporated under vacuum, the crude product was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (20/1) as the eluant, giving the pure product **5** as a yellow oil liquid. (60.5 mg, 82% yield).

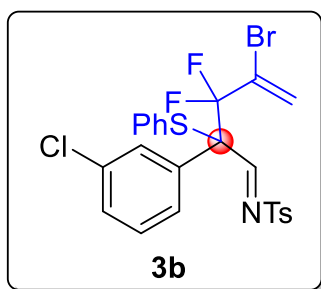
Compound characterization data:

(Z)-N-(4-bromo-2-(4-chlorophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3a):



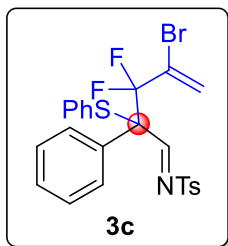
^1H NMR (500 MHz, CDCl_3) δ 8.72 (s, 1H), 7.66 – 7.63 (m, 2H), 7.55 (d, $J = 8.3$ Hz, 2H), 7.22 (d, $J = 8.8$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.07 – 7.03 (m, 3H), 6.93 (t, $J = 7.8$ Hz, 2H), 5.71 (t, $J = 2.2$ Hz, 1H), 5.60 (t, $J = 2.6$ Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 165.81 (dd, $J = 4.7$, 2.8 Hz), 144.91, 136.68, 135.42, 134.37, 132.95, 130.15, 129.79, 129.04, 128.31, 128.18, 126.70, 126.05 (dd, $J = 6.9$, 5.5 Hz) 121.13 (t, $J = 31.2$ Hz), 118.02, 115.96, 67.31 (dd, $J = 24.3$, 21.9 Hz), 21.82; ^{19}F NMR (471 MHz, CDCl_3) δ -97.76 (d, $J = 240.4$ Hz, 1F), -99.47 (d, $J = 240.3$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{19}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 569.9975, found 569.9975.

(Z)-N-(4-bromo-2-(3-chlorophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3b):



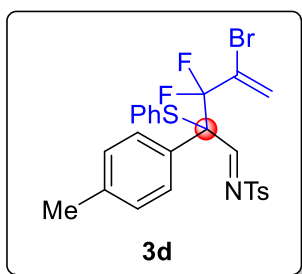
^1H NMR (500 MHz, CDCl_3) δ 8.70 (s, 1H), 7.66 (d, $J = 1.3$ Hz, 1H), 7.57 (t, $J = 9.1$ Hz, 1H), 7.25 – 7.22 (m, 1H), 7.19 (dd, $J = 9.4$, 6.8 Hz, 1H), 7.12 – 7.07 (m, 3H), 6.98 (t, $J = 7.7$ Hz, 1H), 5.73 (t, $J = 2.2$ Hz, 1H), 5.60 (t, $J = 2.6$ Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 165.94 (dd, $J = 4.4$, 2.9 Hz), 144.95, 137.03, 134.38, 133.84, 133.05, 131.63, 130.32, 129.82, 129.71, 129.42, 129.12, 128.26, 126.57, 126.14 – 125.98 (m), 121.13 (t, $J = 31.3$ Hz), 120.06, 118.01, 67.23 (dd, $J = 24.4$, 22.0 Hz), 21.82; ^{19}F NMR (471 MHz, CDCl_3) δ -97.48 (d, $J = 240.9$ Hz), -98.95 (d, $J = 240.9$ Hz); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{19}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 569.9975, found 569.9972.

(Z)-N-(4-bromo-3,3-difluoro-2-phenyl-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3c):



^1H NMR (500 MHz, CDCl_3) δ 8.72 (s, 1H), 7.67 – 7.64 (m, 2H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.25 (dd, $J = 5.3, 1.9$ Hz, 3H), 7.20 – 7.16 (m, 2H), 7.13 – 7.09 (m, 2H), 7.05 (t, $J = 7.5$ Hz, 1H), 6.93 (t, $J = 7.7$ Hz, 2H), 5.68 – 5.66 (m, 1H), 5.56 (t, $J = 2.4$ Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.46 (dd, $J = 4.3, 2.3$ Hz), 144.77, 136.76, 134.52, 131.28, 131.04, 129.95, 129.75, 129.28, 128.90, 128.28, 127.21, 126.03 – 125.86 (m), 121.48 (t, $J = 31.4$ Hz), 120.25, 118.20, 116.13, 67.58 (dd, $J = 24.4, 21.9$ Hz), 21.81; ^{19}F NMR (471 MHz, CDCl_3) δ -96.85 (d, $J = 240.7$ Hz, 1F), -98.12 (d, $J = 240.7$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{20}\text{BrF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 536.0165, found 536.0168.

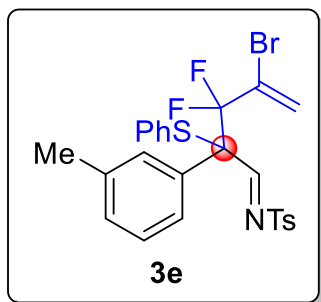
(Z)-N-(4-bromo-3,3-difluoro-2-(phenylthio)-2-(p-tolyl)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3d):



^1H NMR (500 MHz, CDCl_3) δ 8.71 (s, 1H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.52 (d, $J = 7.7$ Hz, 2H), 7.17 (d, $J = 8.4$ Hz, 2H), 7.13 – 7.10 (m, 2H), 7.04 (dd, $J = 7.8, 2.9$ Hz, 3H), 6.93 (t, $J = 7.7$ Hz, 2H), 5.70 – 5.66 (m, 1H), 5.57 (t, $J = 2.4$ Hz, 1H), 2.36 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.67 (dd, $J = 4.3, 1.9$ Hz), 144.72, 139.25, 136.75, 134.58, 131.09, 129.88, 129.73, 128.77, 128.27, 127.37, 126.06 – 125.83 (m), 121.56 (t, $J = 31.4$ Hz), 120.28, 118.23, 116.17, 67.43 (dd, $J = 24.6, 21.8$ Hz), 21.80, 21.27; ^{19}F NMR (471 MHz, CDCl_3) δ -96.68 (d, $J = 240.6$ Hz, 1F), -98.07 (d, $J = 240.6$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{22}\text{BrF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 550.0321, found 550.0323.

(Z)-N-(4-bromo-3,3-difluoro-2-(phenylthio)-2-(m-tolyl)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3e):

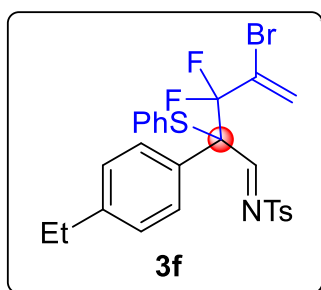
^1H NMR (500 MHz, CDCl_3) δ 8.69 (s, 1H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.42 – 7.35 (m, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.15 (d, $J = 7.2$ Hz, 2H), 7.11 (d, $J = 7.7$ Hz, 1H), 7.06 (dd, $J = 7.1, 6.4$ Hz, 2H), 6.96 (t, $J = 7.7$ Hz, 2H), 5.70 (d, $J = 1.5$ Hz, 1H), 5.59 – 5.56 (m, 1H), 2.35 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.93 (dd, $J = 3.9,$



1.9 Hz), 144.75, 137.59, 136.94, 134.59, 131.66, 131.00, 129.99 (d, $J = 5.7$ Hz), 129.72, 128.88, 128.20, 127.92, 127.37, 125.98 (t, $J = 6.1$ Hz), 121.61 (t, $J = 31.5$ Hz), 120.26, 118.21, 116.14, 67.38 (dd, $J = 24.6, 22.2$ Hz), 21.77, 21.65; ^{19}F NMR (471 MHz, CDCl_3) δ -96.05 (d, $J = 241.3$ Hz, 1F), -97.10 (d, $J = 241.3$ Hz, 1F); HRMS (ESI) calcd

for $\text{C}_{25}\text{H}_{22}\text{BrF}_2\text{NO}_2\text{S}_2$ $[(\text{M} + \text{H})^+]$: 550.0321, found 550.0324.

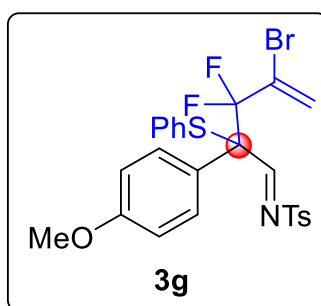
(Z)-N-(4-bromo-2-(4-ethylphenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3f):



^1H NMR (500 MHz, CDCl_3) δ 8.70 (s, 1H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.52 (d, $J = 7.8$ Hz, 2H), 7.18 (d, $J = 8.2$ Hz, 2H), 7.15 – 7.12 (m, 2H), 7.06 (t, $J = 7.3$ Hz, 3H), 6.94 (t, $J = 7.7$ Hz, 2H), 5.69 – 5.67 (m, 1H), 5.56 (t, $J = 2.3$ Hz, 1H), 2.56 (d, $J = 7.6$ Hz, 2H), 2.36 (s, 3H), 1.15 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.87, 145.49,

144.73, 136.83, 134.55, 131.06, 129.90, 129.73, 128.87, 128.26, 127.54, 125.93 (t, $J = 6.2$ Hz), 121.60 (t, $J = 31.5$ Hz), 120.28, 118.23, 116.16, 67.38 (dd, $J = 24.6, 22.0$ Hz), 28.52, 21.79, 15.30; ^{19}F NMR (471 MHz, CDCl_3) δ -96.43 (d, $J = 240.8$ Hz, 1F), -97.73 (d, $J = 240.8$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{24}\text{BrF}_2\text{NO}_2\text{OS}_2$ $[(\text{M} + \text{H})^+]$: 564.0478, found 564.0482.

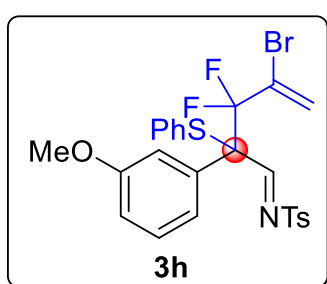
(Z)-N-(4-bromo-3,3-difluoro-2-(4-methoxyphenyl)-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3g):



^1H NMR (500 MHz, CDCl_3) δ 8.71 (s, 1H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.25 (d, $J = 7.7$ Hz, 1H), 7.17 (d, $J = 7.9$ Hz, 3H), 7.16 – 7.13 (m, 2H), 7.13 (d, $J = 1.2$ Hz, 1H), 7.07 (t, $J = 7.5$ Hz, 1H), 6.95 (t, $J = 7.7$ Hz, 2H), 6.78 (dd, $J = 8.2, 2.4$ Hz, 1H), 5.74 – 5.71 (m, 1H), 5.56 (t, $J = 2.4$ Hz, 1H), 3.60 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ

166.47 (d, $J = 3.5$ Hz), 158.92, 144.79, 136.85, 134.53, 132.43, 130.00, 129.74, 128.26, 127.15, 125.85 (t, $J = 6.1$ Hz), 123.57, 121.49 (t, $J = 31.4$ Hz), 120.20, 116.76, 115.13, 67.42 (dd, $J = 24.3, 21.9$ Hz), 55.23 (d, $J = 2.3$ Hz), 21.76 (d, $J = 1.9$ Hz); ^{19}F NMR (471 MHz, CDCl_3) δ -96.75 (d, $J = 241.0$ Hz, 1F), -97.93 (d, $J = 240.9$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{22}\text{BrF}_2\text{NO}_3\text{S}_2$ [(M + H) $^+$]: 566.0271, found 566.0275.

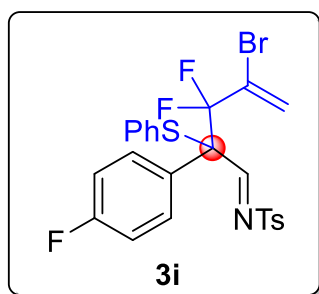
(Z)-N-(4-bromo-3,3-difluoro-2-(3-methoxyphenyl)-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3h):



^1H NMR (500 MHz, CDCl_3) δ 8.71 (s, 1H), 7.57 (dd, $J = 11.7, 4.5$ Hz, 4H), 7.18 (d, $J = 8.9$ Hz, 2H), 7.10 (dd, $J = 8.2, 1.1$ Hz, 2H), 7.04 (t, $J = 7.5$ Hz, 1H), 6.93 (t, $J = 7.7$ Hz, 2H), 6.76 (d, $J = 9.0$ Hz, 2H), 5.70 (t, $J = 2.2$ Hz, 1H), 5.58 (t, $J = 2.4$ Hz, 1H), 3.73 (s, 3H), 2.36 (s, 3H); ^{13}C

NMR (126 MHz, CDCl_3) δ 166.65, 160.01, 144.73, 136.68, 134.58, 132.64, 129.89, 129.74, 128.87, 128.28, 127.34, 125.86 (t, $J = 6.1$ Hz), 122.71, 121.60 (t, $J = 31.5$ Hz), 120.29, 118.24, 116.17, 113.33, 67.23 (dd, $J = 24.4, 21.8$ Hz), 55.36, 21.81; ^{19}F NMR (471 MHz, CDCl_3) δ -96.99 (d, $J = 240.0$ Hz, 1F), -98.63 (d, $J = 240.1$ Hz, 1F); HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{22}\text{BrF}_2\text{NO}_3\text{S}_2$ [(M + H) $^+$]: 566.0271, found 566.0272.

(Z)-N-(4-bromo-3,3-difluoro-2-(4-fluorophenyl)-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3i):

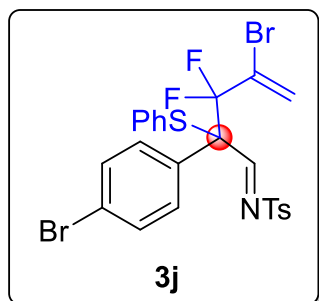


^1H NMR (600 MHz, CDCl_3) δ 8.72 (s, 1H), 7.69 (dd, $J = 8.0, 5.4$ Hz, 2H), 7.55 (d, $J = 8.2$ Hz, 2H), 7.18 (d, $J = 8.2$ Hz, 2H), 7.05 (dd, $J = 13.3, 7.4$ Hz, 3H), 6.96 – 6.90 (m, 4H), 5.69 (s, 1H), 5.58 (t, $J = 2.4$ Hz, 1H), 2.36 (s, 3H); ^{13}C

NMR (151 MHz, CDCl_3) δ 166.01 (dd, $J = 4.4, 2.5$ Hz), 163.78, 162.12, 144.90, 136.67, 134.33, 133.50 (d, $J = 8.3$ Hz), 129.78, 128.29, 126.78, 125.99 – 125.87 (m), 121.24 (t, $J = 31.3$ Hz), 119.79, 118.07, 116.35, 115.01 (d, $J = 21.5$ Hz), 67.16 (dd, $J = 24.3, 21.9$ Hz), 21.82; ^{19}F NMR (471 MHz, CDCl_3) δ -97.72 (d, $J = 240.1$ Hz, 1F), -99.34 (d, $J = 240.1$ Hz, 1F), -111.82 (s, 1F); HRMS (ESI) calcd

for $C_{24}H_{19}BrF_3NO_2S_2$ $[(M + H)^+]$: 554.0071, found 554.0072.

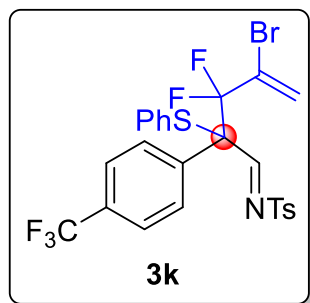
(Z)-N-(4-bromo-2-(4-bromophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3j):



1H NMR (600 MHz, $CDCl_3$) δ 8.71 (s, 1H), 7.58 (d, $J = 8.3$ Hz, 2H), 7.54 (d, $J = 8.3$ Hz, 2H), 7.37 (d, $J = 8.7$ Hz, 2H), 7.17 (d, $J = 8.2$ Hz, 2H), 7.03 (t, $J = 8.6$ Hz, 3H), 6.91 (t, $J = 7.7$ Hz, 2H), 5.69 (s, 1H), 5.58 (t, $J = 2.5$ Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.60 (dd, $J = 4.3$, 2.6 Hz), 144.90, 136.64, 133.19, 131.10, 130.13, 129.76,

129.01, 128.24, 126.55, 126.34 – 125.86 (m), 123.78, 120.98 (t, $J = 31.2$ Hz), 119.60, 117.89, 116.17, 67.36 (dd, $J = 24.2$, 21.8 Hz), 21.80; ^{19}F NMR (471 MHz, $CDCl_3$) δ -97.78 (d, $J = 240.3$ Hz, 1F), -99.48 (d, $J = 240.3$ Hz, 1F); HRMS (ESI) calcd for $C_{24}H_{19}Br_2F_2NO_2S_2$ $[(M + H)^+]$: 613.9270, found 613.9275.

(Z)-N-(4-bromo-3,3-difluoro-2-(phenylthio)-2-(4-(trifluoromethyl)phenyl)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3k):

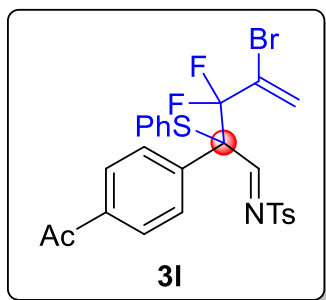


1H NMR (500 MHz, $CDCl_3$) δ 8.74 (s, 1H), 7.85 (d, $J = 8.2$ Hz, 2H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.50 (d, $J = 8.4$ Hz, 2H), 7.19 (d, $J = 8.1$ Hz, 2H), 7.06 – 7.03 (m, 3H), 6.96 – 6.91 (m, 2H), 5.67 (t, $J = 2.1$ Hz, 1H), 5.58 (t, $J = 2.6$ Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.60 (dd, $J = 4.4$, 3.1 Hz), 145.03, 136.76, 134.21, 132.08, 131.15 (q, $J =$

32.7 Hz), 130.30, 129.82, 129.13, 128.32, 126.11 (dd, $J = 7.0$, 5.5 Hz), 124.80 (dd, $J = 7.3$, 3.6 Hz), 122.76, 120.92 (t, $J = 31.2$ Hz), 120.00, 117.93, 115.87, 67.48 (dd, $J = 24.2$, 22.0 Hz), 21.79; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.78 (s, 3F), -97.81 (d, $J = 240.8$ Hz, 1F), -99.43 (d, $J = 240.8$ Hz, 1F); HRMS (ESI) calcd for $C_{25}H_{19}BrF_5NO_2S_2$ $[(M + H)^+]$: 604.0039, found 604.0042.

(Z)-N-(2-(4-acetylphenyl)-4-bromo-3,3-difluoro-2-(phenylthio)pent-4-en-1-

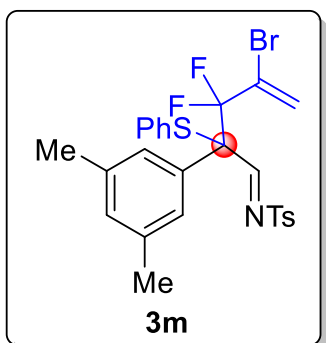
ylidene)-4-methylbenzenesulfonamide (3l):



^1H NMR (500 MHz, CDCl_3) δ 8.82 (s, 1H), 7.89 (s, 4H), 7.64 (d, $J = 8.3$ Hz, 2H), 7.27 (d, $J = 8.2$ Hz, 2H), 7.12 (t, $J = 8.5$ Hz, 3H), 7.00 (t, $J = 7.7$ Hz, 2H), 5.75 (d, $J = 2.2$ Hz, 1H), 5.64 (t, $J = 2.5$ Hz, 1H), 2.61 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 197.72, 165.64 (dd, $J = 4.4$, 3.0 Hz), 144.96, 137.22, 136.76, 134.28, 131.89, 130.21,

129.79, 129.05, 128.28, 127.67, 126.05 (dd, $J = 6.9$, 5.6 Hz), 120.99 (t, $J = 31.2$ Hz), 119.99, 117.93, 115.86, 67.57 (dd, $J = 24.2$, 22.0 Hz), 26.83, 21.80; ^{19}F NMR (471 MHz, CDCl_3) δ -97.64 (d, $J = 240.8$ Hz, 1F), -99.14 (d, $J = 240.8$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{26}\text{H}_{22}\text{BrF}_2\text{NO}_3\text{S}_2$ [(M + H) $^+$]: 578.0271, found 578.0268.

(Z)-N-(4-bromo-2-(3,5-dimethylphenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3m):

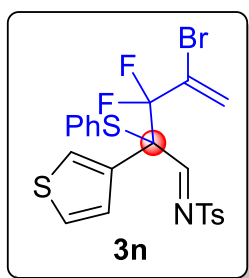


^1H NMR (500 MHz, CDCl_3) δ 8.66 (s, 1H), 7.59 (d, $J = 8.3$ Hz, 2H), 7.19 (dd, $J = 7.5$, 4.6 Hz, 4H), 7.10 (d, $J = 11.9$ Hz, 3H), 7.00 (t, $J = 7.7$ Hz, 2H), 6.87 (s, 1H), 5.74 (d, $J = 1.6$ Hz, 1H), 5.59 (s, 1H), 2.36 (s, 3H), 2.18 (s, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 167.47 (d, $J = 3.3$ Hz), 144.74, 137.48, 137.17, 134.69, 130.91, 130.01, 129.70, 128.88,

128.60, 128.16, 127.57, 126.05 (t, $J = 6.1$ Hz), 121.79 (t, $J = 31.7$ Hz), 120.28, 118.23, 116.17, 67.41 – 66.97 (m), 21.76, 21.51; ^{19}F NMR (471 MHz, CDCl_3) δ -95.17 (d, $J = 242.1$ Hz, 1F), -95.98 (d, $J = 242.1$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{26}\text{H}_{24}\text{BrF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 564.0478, found 564.0481.

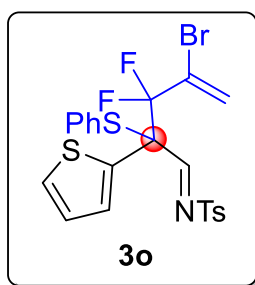
(Z)-N-(4-bromo-3,3-difluoro-2-(phenylthio)-2-(thiophen-3-yl)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3n):

^1H NMR (500 MHz, CDCl_3) δ 8.70 (s, 1H), 7.61 (d, $J = 8.3$ Hz, 2H), 7.57 (dd, $J = 3.0$, 1.2 Hz, 1H), 7.32 (d, $J = 5.1$ Hz, 1H), 7.21 (d, $J = 8.1$ Hz, 2H), 7.17 (dd, $J = 5.1$, 3.0 Hz, 1H), 7.09 (t, $J = 7.4$ Hz, 1H), 6.99 (dd, $J = 8.2$, 1.3 Hz, 2H), 6.93 (t, $J = 7.7$ Hz,



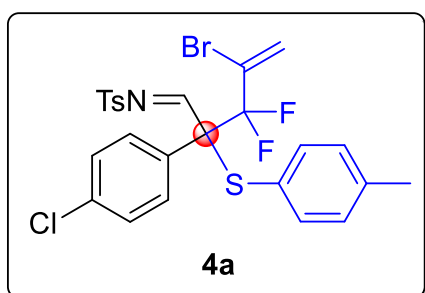
2H), 5.80 (dd, $J = 2.7, 1.4$ Hz, 1H), 5.60 – 5.57 (m, 1H), 2.37 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.74 (t, $J = 4.1$ Hz), 144.94, 137.25, 134.38, 130.20, 129.84, 129.20, 128.88, 128.37, 126.70, 125.25 (t, $J = 6.2$ Hz), 124.72, 121.52 (t, $J = 31.2$ Hz), 120.01, 117.95, 115.89, 64.40 (t, $J = 23.3$ Hz), 21.82; ^{19}F NMR (471 MHz, CDCl_3) δ -98.37 (d, $J = 241.2$ Hz, 1F), -98.98 (d, $J = 241.1$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{22}\text{H}_{18}\text{BrF}_2\text{NO}_2\text{S}_3$ [(M + H) $^+$]: 541.9729, found 541.9733.

(Z)-N-(4-bromo-3,3-difluoro-2-(phenylthio)-2-(thiophen-2-yl)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (3o)



^1H NMR (500 MHz, CDCl_3) δ 8.71 (s, 1H), 7.64 (d, $J = 8.3$ Hz, 2H), 7.39 (dd, $J = 3.4, 1.7$ Hz, 1H), 7.23 (dd, $J = 9.8, 4.6$ Hz, 3H), 7.09 (ddd, $J = 9.0, 8.3, 4.3$ Hz, 3H), 6.96 (t, $J = 7.7$ Hz, 2H), 6.88 (dd, $J = 5.1, 3.8$ Hz, 1H), 5.85 (t, $J = 2.3$ Hz, 1H), 5.60 (t, $J = 2.5$ Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.17 (t, $J = 4.1$ Hz), 144.94, 137.54, 134.43, 132.00, 130.47, 129.80, 128.96, 128.61, 128.39, 126.46, 126.27, 125.69 – 125.49 (m), 121.26 (t, $J = 31.1$ Hz), 117.87, 115.81, 65.07 – 64.58 (m), 21.85; ^{19}F NMR (471 MHz, CDCl_3) δ -98.39 (d, $J = 240.2$ Hz, 1F), -99.42 (d, $J = 240.0$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{22}\text{H}_{18}\text{BrF}_2\text{NO}_2\text{S}_3$ [(M + H) $^+$]: 541.9729, found 541.9730.

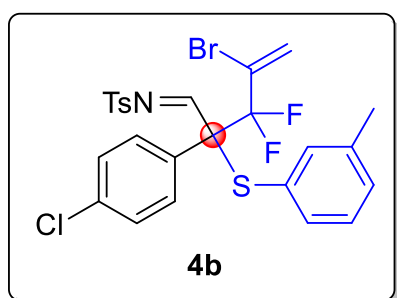
(Z)-N-(4-bromo-2-(4-chlorophenyl)-3,3-difluoro-2-(p-tolylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4a):



^1H NMR (500 MHz, CDCl_3) δ 8.69 (s, 1H), 7.62 (d, $J = 8.0$ Hz, 2H), 7.55 (d, $J = 8.3$ Hz, 2H), 7.19 (dd, $J = 14.7, 8.4$ Hz, 4H), 6.96 (s, 1H), 6.89 – 6.81 (m, 3H), 5.69 (t, $J = 2.2$ Hz, 1H), 5.58 (t, $J = 2.5$ Hz, 1H), 2.36 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 166.04 (dd, $J = 4.5, 2.7$ Hz), 144.85, 139.05, 137.65, 135.33, 133.59, 132.93, 131.10, 129.72, 128.84, 128.19,

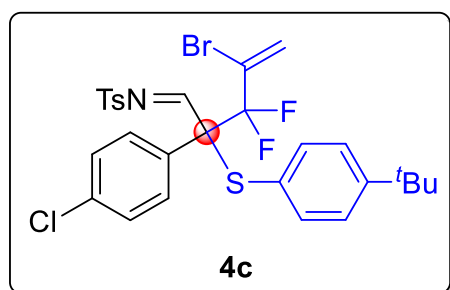
126.01 (dd, $J = 6.8, 5.5$ Hz), 121.15 (t, $J = 31.3$ Hz), 120.07, 118.02, 115.95, 67.23 (dd, $J = 24.4, 21.9$ Hz), 21.80, 21.20; ^{19}F NMR (471 MHz, CDCl_3) δ -97.69 (d, $J = 240.4$ Hz, 1F), -99.36 (d, $J = 240.4$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{25}\text{H}_{21}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 583.9932, found 583.9928.

(Z)-N-(4-bromo-2-(4-chlorophenyl)-3,3-difluoro-2-(*m*-tolylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4b):



^1H NMR (500 MHz, CDCl_3) δ 8.73 (s, 1H), 7.65 (d, $J = 8.5$ Hz, 2H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.20 (dd, $J = 13.0, 8.3$ Hz, 4H), 6.93 (d, $J = 8.1$ Hz, 2H), 6.68 (d, $J = 8.0$ Hz, 2H), 5.70 (t, $J = 2.2$ Hz, 1H), 5.58 (t, $J = 2.6$ Hz, 1H), 2.37 (s, 3H), 2.07 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 165.63 (dd, $J = 4.5, 2.7$ Hz), 144.78, 140.63, 137.13, 135.31, 134.43, 132.96, 129.80, 129.70, 129.50 (d, $J = 1.2$ Hz), 128.24, 128.11, 125.92 (dd, $J = 6.8, 5.6$ Hz), 122.94, 121.22 (t, $J = 31.3$ Hz), 120.06, 118.00, 115.93, 67.37 (dd, $J = 24.4, 21.8$ Hz), 21.83, 21.37; ^{19}F NMR (471 MHz, CDCl_3) δ -97.91 (d, $J = 240.3$ Hz, 1F), -99.54 (d, $J = 240.3$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{25}\text{H}_{21}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 583.9932, found 583.9931.

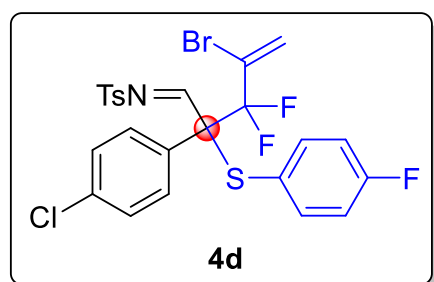
(Z)-N-(4-bromo-2-((4-*tert*-butyl)phenyl)thio)-2-(4-chlorophenyl)-3,3-difluoropent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4c):



^1H NMR (600 MHz, CDCl_3) δ 8.79 (s, 1H), 7.74 – 7.67 (m, 4H), 7.29 (dd, $J = 11.6, 8.5$ Hz, 4H), 7.11 (d, $J = 8.5$ Hz, 2H), 7.07 (d, $J = 8.5$ Hz, 2H), 5.77 (d, $J = 2.1$ Hz, 1H), 5.65 (t, $J = 2.4$ Hz, 1H), 2.43 (s, 3H), 1.20 (s, 9H); ^{13}C NMR (151 MHz, CDCl_3) δ 166.13 – 166.00 (m), 153.65, 144.93, 137.10, 135.21, 134.25, 132.85, 129.67, 128.33, 127.90, 126.20, 122.83, 121.11 (t, $J = 31.3$ Hz), 119.68, 117.97, 116.25), 67.18 (dd, $J = 24.4, 21.9$ Hz), 34.69, 31.04, 21.80; ^{19}F NMR (471 MHz, CDCl_3) δ -97.62 (d, $J = 240.6$ Hz, 1F), -99.05 (d, $J = 240.6$ Hz,

1F); HRMS (ESI): calcd for C₂₈H₂₇BrClF₂NO₂S₂ [(M + H)⁺]: 626.0401, found 626.0439.

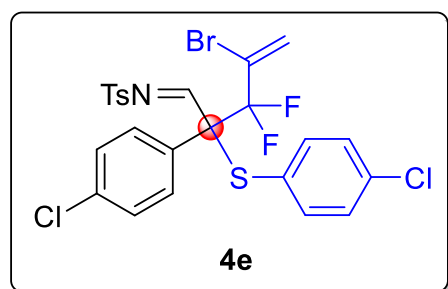
(Z)-N-(4-bromo-2-(4-chlorophenyl)-3,3-difluoro-2-((4-fluorophenyl)thio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4d):



¹H NMR (500 MHz, CDCl₃) δ 8.74 (s, 1H), 7.66 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.7 Hz, 2H), 7.19 (d, *J* = 8.2 Hz, 2H), 6.98 (dd, *J* = 8.7, 5.3 Hz, 2H), 6.51 (t, *J* = 8.6 Hz, 2H), 5.70 (s, 1H), 5.59 (t, *J* = 2.5 Hz, 1H), 2.37 (s, 3H);

¹³C NMR (126 MHz, CDCl₃) δ 165.15 (dd, *J* = 4.7, 2.8 Hz), 162.84, 145.19, 138.90 (d, *J* = 8.8 Hz), 135.52, 134.27, 132.90, 129.77, 128.25, 128.12, 126.03 (dd, *J* = 6.8, 5.6 Hz), 121.96 (d, *J* = 3.2 Hz), 121.05 (t, *J* = 31.2 Hz), 120.02, 117.97, 116.22 (d, *J* = 22.0 Hz), 67.47 (t, *J* = 22.6 Hz), 21.77; ¹⁹F NMR (471 MHz, CDCl₃) δ -97.90 (d, *J* = 240.1 Hz, 1F), -99.79 (d, *J* = 240.1 Hz, 1F), -108.73 (s, 1F); HRMS (ESI): calcd for C₂₄H₁₈BrClF₃NO₂S₂ [(M + H)⁺]: 587.9681, found 587.9684.

(Z)-N-(4-bromo-2-(4-chlorophenyl)-2-((4-chlorophenyl)thio)-3,3-difluoropent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4e):

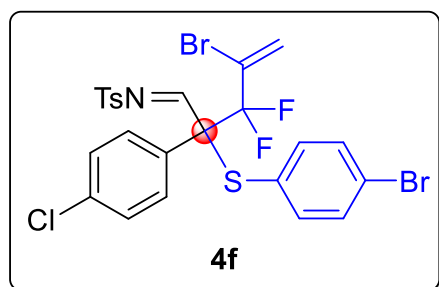


¹H NMR (500 MHz, CDCl₃) δ 8.77 (s, 1H), 7.67 (d, *J* = 7.7 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.8 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 2H), 6.74 (d, *J* = 8.6 Hz, 2H), 5.71 (t, *J* = 2.1 Hz, 1H), 5.61 (t, *J* = 2.6 Hz, 1H), 2.39

(s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 164.84 (dd, *J* = 4.4, 2.9 Hz), 145.23, 137.39, 135.60, 134.29, 132.87, 129.84, 129.04, 128.31, 128.02, 126.23 – 126.05 (m), 125.31, 120.94 (t, *J* = 31.1 Hz), 119.99, 117.93, 115.86, 67.46 (dd, *J* = 24.0, 21.8 Hz), 21.94; ¹⁹F NMR (471 MHz, CDCl₃) δ -97.89 (d, *J* = 240.0 Hz, 1F), -99.72 (d, *J* = 239.9 Hz, 1F); HRMS (ESI): calcd for C₂₄H₁₈BrCl₂F₂NO₂S₂ [(M + H)⁺]: 603.9385, found

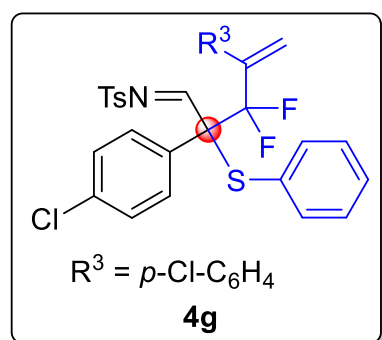
603.9384.

(Z)-N-(4-bromo-2-((4-bromophenyl)thio)-2-(4-chlorophenyl)-3,3-difluoropent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4f):



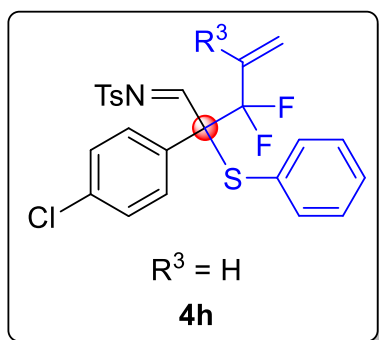
^1H NMR (500 MHz, CDCl_3) δ 8.78 (s, 1H), 7.67 (d, $J = 7.7$ Hz, 2H), 7.52 (d, $J = 8.3$ Hz, 2H), 7.25 (d, $J = 8.8$ Hz, 2H), 7.20 (d, $J = 8.0$ Hz, 2H), 6.89 (d, $J = 8.6$ Hz, 2H), 6.80 (d, $J = 8.6$ Hz, 2H), 5.71 (t, $J = 2.2$ Hz, 1H), 5.61 (t, $J = 2.6$ Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 164.83 (dd, $J = 4.5, 2.8$ Hz), 145.24, 137.53, 135.61, 134.30, 132.85, 131.97, 129.89, 128.32, 128.02, 126.24 – 126.03 (m), 125.07, 120.90 (t, $J = 31.1$ Hz), 119.97, 117.91, 115.85, 67.36 (dd, $J = 24.0, 21.8$ Hz), 22.03; ^{19}F NMR (471 MHz, CDCl_3) δ -97.89 (d, $J = 239.9$ Hz, 1F), -99.67 (d, $J = 239.9$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{18}\text{Br}_2\text{ClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 647.8880, found 647.8882.

(Z)-N-(2,4-bis(4-chlorophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4g):



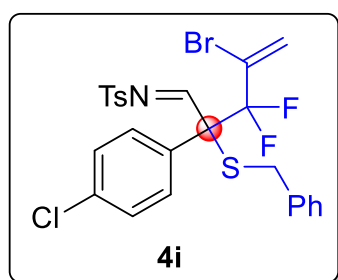
^1H NMR (500 MHz, CDCl_3) δ 8.52 (s, 1H), 7.52 (d, $J = 8.5$ Hz, 2H), 7.44 (d, $J = 8.3$ Hz, 2H), 7.21 (dd, $J = 17.2, 8.6$ Hz, 6H), 7.08 – 7.04 (m, 3H), 7.02 (d, $J = 7.2$ Hz, 2H), 6.92 (t, $J = 7.7$ Hz, 2H), 5.34 (s, 1H), 5.31 (s, 1H), 2.43 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 165.93 (d, $J = 4.0$ Hz), 144.74, 140.75 (t, $J = 23.1$ Hz), 136.27, 135.14 (d, $J = 12.7$ Hz), 134.31, 134.16, 132.95, 130.36, 130.01, 129.79, 129.73, 128.86, 128.74, 128.15, 127.90, 127.21, 124.61 (t, $J = 8.8$ Hz), 122.80, 120.74, 118.70, 67.85 (t, $J = 23.9$ Hz), 21.81; ^{19}F NMR (471 MHz, CDCl_3) δ -95.84 (d, $J = 245.2$ Hz, 1F), -97.42 (d, $J = 245.1$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{30}\text{H}_{23}\text{Cl}_2\text{F}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 602.0593, found 602.0590.

(E)-N-(2-(4-chlorophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4h):



^1H NMR (500 MHz, CDCl_3) δ 8.67 (d, $J = 1.7$ Hz, 1H), 7.63 (d, $J = 8.3$ Hz, 2H), 7.49 (d, $J = 8.5$ Hz, 2H), 7.32 (d, $J = 8.7$ Hz, 2H), 7.27 (d, $J = 9.2$ Hz, 2H), 7.22 (d, $J = 7.2$ Hz, 2H), 7.15 (t, $J = 7.5$ Hz, 1H), 7.02 (t, $J = 7.7$ Hz, 2H), 5.87 (ddd, $J = 23.8, 17.3, 11.2$ Hz, 1H), 5.45 (dd, $J = 27.1, 14.2$ Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 168.67, 145.02, 136.65, 135.45, 134.26, 132.09, 130.67, 129.90, 129.46 (t, $J = 25.4$ Hz), 128.95, 128.48, 128.21, 127.45, 123.09 (t, $J = 9.3$ Hz), 119.53, 117.53, 66.53 (t, $J = 24.9$ Hz), 21.84; ^{19}F NMR (471 MHz, CDCl_3) δ -97.45 (dd, $J = 244.4, 10.8$ Hz, 1H), -99.95 (dd, $J = 244.4, 12.7$ Hz, 1H); HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{20}\text{ClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 492.0670, found 492.0674.

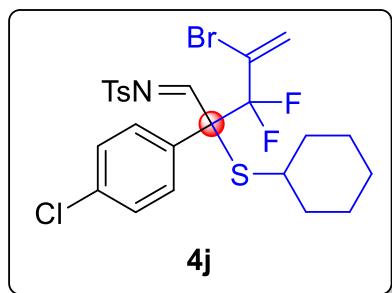
(Z)-N-(2-(benzylthio)-4-bromo-2-(4-chlorophenyl)-3,3-difluoropent-4-en-1-ylidene)-4-methylbenzenesulfonamide (4i):



^1H NMR (500 MHz, CDCl_3) δ 8.84 (s, 1H), 7.81 (d, $J = 8.3$ Hz, 2H), 7.54 (d, $J = 8.4$ Hz, 2H), 7.25 (dd, $J = 11.4, 8.5$ Hz, 4H), 7.16 – 7.13 (m, 3H), 6.89 (dd, $J = 6.4, 3.0$ Hz, 2H), 5.66 (d, $J = 2.2$ Hz, 1H), 5.61 (t, $J = 2.6$ Hz, 1H), 3.05 (dd, $J = 30.0, 11.4$ Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 162.89 – 162.78 (m), 145.34, 135.46, 134.86, 133.82, 132.76, 130.15, 129.52, 128.75, 128.29, 127.93, 127.72 (d, $J = 1.3$ Hz), 120.78 (t, $J = 31.2$ Hz), 119.67, 117.61, 115.56, 65.45 (dd, $J = 24.8, 22.4$ Hz), 34.23, 21.81; ^{19}F NMR (471 MHz, CDCl_3) δ -99.49 (d, $J = 240.3$ Hz, 1F), -101.05 (d, $J = 240.3$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{25}\text{H}_{21}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 583.9932, found 582.9932.

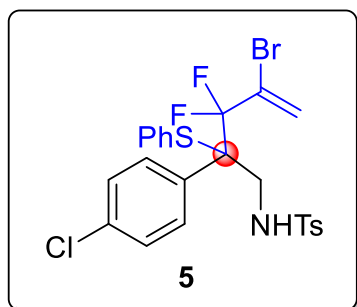
(Z)-N-(4-bromo-2-(4-chlorophenyl)-2-(cyclohexylthio)-3,3-difluoropent-4-en-1-

ylidene)-4-methylbenzenesulfonamide (4j):



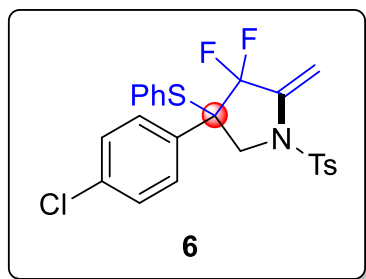
^1H NMR (500 MHz, CDCl_3) δ 8.73 (s, 1H), 7.79 (d, $J = 8.3$ Hz, 2H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.29 (d, $J = 8.1$ Hz, 2H), 7.19 (d, $J = 8.8$ Hz, 2H), 5.62 (d, $J = 2.2$ Hz, 1H), 5.56 (t, $J = 2.5$ Hz, 1H), 2.39 (s, 3H), 2.25 – 2.16 (m, 1H), 1.49 (dd, $J = 15.9, 12.4$ Hz, 2H), 1.45 – 1.38 (m, 2H), 1.25 (ddd, $J = 49.7, 10.4, 7.1$ Hz, 4H), 0.89 (dd, $J = 21.4, 10.5$ Hz, 2H); ^{13}C NMR (126 MHz, CDCl_3) δ 164.36 – 164.22 (m), 145.25, 135.27, 132.92, 130.02, 128.42, 127.92, 126.12 – 125.67 (m), 121.03 (t, $J = 31.3$ Hz), 119.74, 117.68, 115.62, 65.41 (dd, $J = 24.5, 21.9$ Hz), 43.41, 35.02, 34.49, 25.94 (d, $J = 9.9$ Hz), 25.14, 21.81; ^{19}F NMR (471 MHz, CDCl_3) δ -98.90 (d, $J = 239.4$ Hz, 1F), -100.75 (d, $J = 239.4$ Hz, 1F); HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{25}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 576.0245, found 576.0247.

N-(4-bromo-2-(4-chlorophenyl)-3,3-difluoro-2-(phenylthio)pent-4-en-1-yl)-4-methylbenzenesulfonamide (5):



^1H NMR (600 MHz, CDCl_3) δ 7.71 (d, $J = 8.2$ Hz, 2H), 7.47 (d, $J = 8.4$ Hz, 2H), 7.34 (d, $J = 8.1$ Hz, 2H), 7.29 (dd, $J = 19.0, 8.3$ Hz, 5H), 7.10 (t, $J = 7.8$ Hz, 2H), 5.77 (d, $J = 2.5$ Hz, 1H), 5.68 (d, $J = 2.7$ Hz, 1H), 5.08 (dd, $J = 7.6, 3.5$ Hz, 1H), 3.91 (dd, $J = 12.6, 7.8$ Hz, 1H), 3.36 (dd, $J = 12.6, 3.6$ Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 143.99, 137.01, 136.05, 135.18, 132.27, 131.20, 130.04, 129.03, 128.47, 127.22, 126.61 (t, $J = 6.0$ Hz), 121.48 (t, $J = 32.8$ Hz), 120.79, 119.08, 117.36, 63.72 (t, $J = 24.9$ Hz), 42.88, 21.75; ^{19}F NMR (471 MHz, CDCl_3) δ -91.79 (d, $J = 242.6$ Hz, 1H), -92.68 (d, $J = 242.6$ Hz, 1H); HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{21}\text{BrClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 571.9932, found 571.9938.

4-(4-chlorophenyl)-3,3-difluoro-2-methylene-4-(phenylthio)-1-tosylpyrrolidine (6):



^1H NMR (500 MHz, CDCl_3) δ 7.56 (d, $J = 8.2$ Hz, 2H), 7.16 (t, $J = 7.5$ Hz, 1H), 7.11 (d, $J = 8.2$ Hz, 2H), 7.02 (dd, $J = 12.8, 8.1$ Hz, 4H), 6.83 (d, $J = 7.5$ Hz, 2H), 6.79 (d, $J = 8.5$ Hz, 2H), 5.40 (dd, $J = 4.0, 2.5$ Hz, 1H), 4.94 (dd, $J = 5.1, 2.4$ Hz, 1H), 3.92 (dd, $J = 10.4, 2.5$ Hz, 1H), 3.52 (d, $J = 10.4$ Hz, 1H), 2.20 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 145.12, 138.82 (dd, $J = 29.9, 23.0$ Hz), 137.79, 134.30, 133.96, 133.53, 130.37, 129.94, 128.97, 128.93, 128.59, 128.06, 127.54, 121.42 (dd, $J = 261.1, 252.3$ Hz), 95.25 (d, $J = 4.1$ Hz), 59.70 (dd, $J = 22.5, 17.9$ Hz), 53.81 (d, $J = 2.5$ Hz), 21.74; ^{19}F NMR (471 MHz, CDCl_3) δ -91.30 (dd, $J = 238.7, 4.3$ Hz, 1H), -114.36 (d, $J = 238.7$ Hz, 1H); HRMS (ESI): calcd for $\text{C}_{24}\text{H}_{20}\text{ClF}_2\text{NO}_2\text{S}_2$ [(M + H) $^+$]: 492.0670, found 492.0675.

References

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^1H NMR, ^{13}C NMR and ^{19}F NMR spectra

