Alkoxy sulfanyl Radical Species: Acquisition and Transformation towards Sulfonate Esters through Electrochemistry

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Supporting Information

1. General method ......................................................................................................S2
2. Optimization of the reaction conditions .................................................................S2
3. General Procedures ...............................................................................................S3
4. Hydrogen-deuterium exchange ..............................................................................S4
5. Mechanistic Studies ...............................................................................................S5
6. Cyclic Voltammetry Studies ..................................................................................S7
7. Characterization data ..............................................................................................S8
8. $^1$H and $^{13}$C NMR spectra ..................................................................................S30
9. Crystallographic details of 3as and 5m ................................................................S95
1. General method

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 μm, 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 δ scale. ¹H, and ¹³C NMR spectra were recorded in CDCl₃ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values were quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument. The electrochemical reactions were performed on IKa ElectraSyn 2.0. Cyclic voltammograms were recorded on a CHI 760E potentiostat.

2. Optimization of the reaction conditions

Table S1. Initial studies for the electrochemical reaction of 2-methyl-1,1-diphenylprop-2-en-1-ol 4a, inorganic sulfite, and Methanol 2a

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<th>Entry</th>
<th>“SO₂” (equiv)</th>
<th>Solvent</th>
<th>Electrolyte</th>
<th>I₉ (mA)</th>
<th>Yield (%)[b]</th>
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<td>NaHSO₃ (4.0)</td>
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**3. General Procedures**

**General Procedure I:** Experimental procedures for the reaction of alkenes 1, alcohols 2, and NaHSO₃

\[
\begin{align*}
\text{Ar} & \quad \text{R}^1 \quad \text{R}^2 \\
1 & + \quad \text{ROH} + \quad \text{NaHSO}_3 \\
\text{Ar} & \quad \text{R}^1 \quad \text{R}^2 \\
2 & \quad \text{MeCN, 12h, rt} \\
3 & \quad \text{Bu}_4\text{NBF}_4, \text{4 mA} \\
\end{align*}
\]

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, alkene 1 (0.5 mmol), NaHSO₃ (2.0 mmol, 208.0 mg), Bu₄NBF₄ (0.5 mmol, 164.6 mg), alcohol 2 (2.0 mL) and MeCN (8.0 mL) were added under N₂ atmosphere. [Liquid compounds were added after the addition of alcohol and MeCN]. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 4 mA for 12 hours. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 4/1) to give the corresponding product 3.

![Figure S1. Reaction setup. (a) ElectraSyn 2.0 vial (10 mL). (b) ElectraSyn 2.0 cap equipped with Pt (down) and graphite (up). (c) ElectraSyn 2.0.](image)
General Procedure II: Experimental procedures for the reaction of 2-methyl-1,1-diphenylprop-2-en-1-ol 4a, Methanol 2a and Na$_2$S$_2$O$_5$.

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, tertiary alcohol 4 (0.5 mmol), Na$_2$S$_2$O$_5$ (1.0 mmol, 190.0 mg), “Bu$_4$NClO$_4$ (0.5 mmol, 171.0 mg), methanol 2a (2.0 mL) and DCM (8.0 mL) were added under N$_2$ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 3 mA for 12 hours. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 4/1) to give the corresponding product 5.

4. Hydrogen-deuterium exchange of compound 3aa

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, 3aa (61.3 mg, 0.2 mmol), “Bu$_4$NBF$_4$ (0.25 mmol, 82.3 mg), MeOD (1.0 mL) and MeCN (4.0 mL) were added under N$_2$ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 4 mA. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 4/1) to give the corresponding product 3aa-d.
5. Mechanistic Studies

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, N-methacryloyl-N-methylbenzamide 6 (0.5 mmol, 101.6 mg), NaHSO₃ (2.0 mmol, 208.0 mg), "Bu₄NBF₄ (0.5 mmol, 164.6 mg), MeOH (2.0 mL) and MeCN (8.0 mL) were added under N₂ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 4 mA for 12 hours. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 3/1) to give the corresponding product 7 (25.6 mg, 18% yield).
To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, N-methacryloyl-N-methylbenzamide 8 (0.5 mmol, 72.1 mg), NaHSO₃ (2.0 mmol, 208.0 mg), nBu₄NBF₄ (0.5 mmol, 164.6 mg), MeOH (2.0 mL) and MeCN (8.0 mL) were added under N₂ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 4 mA for 12 hours. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 3/1) to give the corresponding products 3bj (32.1 mg, 24% yield) and 9 (20.5 mg, 15% yield).

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, 4-(Trifluoromethyl)styrene 1bj (0.5 mmol, 101.6 mg), NaHSO₃ (2.0 mmol, 208.0 mg), nBu₄NBF₄ (0.5 mmol, 164.6 mg), MeOH (2.0 mL) and MeCN (8.0 mL) were added under N₂ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and cathode (platinum plate) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 4 mA for 12 hours. After electrolysis, the solvent was evaporated and the residue was purified directly by flash column chromatography (n-hexane/EtOAc (v/v): 4/1) to give the corresponding product 3bj (55.1 mg, 37% yield) and 9 (18.6 mg, 14% yield).

To an ElectraSyn vial (10 mL) equipped with a magnetic stir bar, N-butyl-N-(1-
phenylvinyl)acetamide 1bk (0.5 mmol, 101.6 mg), NaHSO₃ (2.0 mmol, 208.0 mg), 
³Bu₄NBF₄ (0.5 mmol, 164.6 mg), MeOH (2.0 mL) and MeCN (8.0 mL) were added 
under N₂ atmosphere. The ElectraSyn vial cap equipped with anode (graphite) and 
cathode (platinum plate) were inserted into the mixture. The reaction mixture was 
electrolyzed under a constant current of 4 mA for 12 hours. After electrolysis, the 
solvent was evaporated and the residue was purified directly by flash column 
chromatography (n-hexane/EtOAc (v/v): 4/1) to give the corresponding product 10 
(130.7 mg, 84% yield).

6. Cyclic Voltammetry Studies

The cyclic voltammograms were recorded at rt using a glassy carbon disk 
working electrode (diameter, 1.0 mm), a Pt wire auxiliary electrode and a SCE 
reference electrode. The scan rate is 100 mV/s.

![Cyclic voltammograms of compound 1a, NaHSO₃ and SO₂ in MeCN/MeOH (v/v: 4/1) with 0.05 M ³Bu₄NBF₄. Black, blank; Red, compound 1a (10.0 mM), Eₚ/₂ = 1.80 V; Blue, NaHSO₃ (2.5 mM), Eₚ/₂ = 1.23 V; Green, SO₂.](image-url)
**Figure S3.** Cyclic voltammograms of compound 4a, Na$_2$S$_2$O$_5$ in CH$_2$Cl$_2$/MeOH (v/v: 4/1) with 0.05 M $^n$Bu$_4$NClO$_4$; Black, blank; Red, Na$_2$S$_2$O$_5$ (1.0 mM), $E_{p/2}$ = 1.15 V; Blue, compound 4a (10.0 mM).

**Figure S4.** Cyclic voltammograms of compound 6 in MeCN/MeOH (v/v: 4/1) with 0.05 M $^n$Bu$_4$NBF$_4$. Black, blank; Red, compound 6.

7. Characterization data

![Structural formula](image)

**3aa:** Methyl 2-([1,1'-biphenyl]-4-yl)-2-methoxyethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.67-7.55 (m, 14H), 7.51-7.33 (m, 5H), 4.78 (dd, $J$ = 9.3, 3.0 Hz, 1H), 3.91 (s, 3H), 3.65 (dd, $J$ = 14.9, 9.3 Hz, 1H), 3.36 (dd, $J$ = 14.9, 3.1 Hz, 1H), 3.32 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 141.8, 140.4, 137.5, 128.9, 127.8, 127.7, 127.1, 127.0, 78.1, 57.2, 57.1, 56.2; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{18}$O$_4$NaS$^+$ 329.0818, found 329.0827.

![Structural formula](image)

**3ab:** Methyl 2-methoxy-2-(o-tolyl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.42-7.36 (m, 1H), 7.28-7.22 (m, 2H), 7.21-7.15 (m, 1H), 4.99 (dd, $J$ = 9.2, 2.6 Hz, 1H), 3.91 (s, 3H), 3.54 (dd, $J$ = 15.0, 9.2 Hz, 1H), 3.28 (s, 3H), 3.23 (dd, $J$ = 15.0, 2.6 Hz, 1H), 2.38 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$
136.6, 135.5, 131.0, 128.4, 126.7, 125.7, 75.1, 56.9, 56.2, 18.9; HRMS (ESI) m/z [M + Na]^+ calcd for C_{11}H_{16}O_{4}NaS^+ 267.0662, found 267.0673.

3ac: Methyl 2-methoxy-2-(p-tolyl)ethane-1-sulfonate

^1^H NMR (400 MHz, CDCl_{3}) δ 7.25-7.16 (m, 4H), 4.69 (dd, J = 9.2, 3.2 Hz, 1H), 3.88 (s, 3H), 3.60 (dd, J = 14.8, 9.2 Hz, 1H), 3.28(dd, J = 14.8, 3.2 Hz, 1H), 3.26(s, 3H), 2.36 (s, 3H); ^1^C NMR (100 MHz, CDCl_{3}) δ 138.7, 135.6, 129.7, 126.6, 78.2, 57.3, 56.9, 56.1, 21.2; HRMS (ESI) m/z [M + Na]^+ calcd for C_{11}H_{16}O_{4}NaS^+ 267.0662, found 267.0673.

3ad: Methyl 2-methoxy-2-(4-methoxyphenyl)ethane-1-sulfonate

^1^H NMR (400 MHz, CDCl_{3}) δ 7.26 (d, J = 8.7 Hz, 1H), 6.92 (d, J = 8.6 Hz, 2H), 4.67 (dd, J = 9.1, 3.3 Hz, 1H), 3.88 (s, 3H), 3.82 (s, 3H), 3.61 (dd, J = 14.8, 9.1 Hz, 1H), 3.28 (dd, J = 14.8, 3.4 Hz, 1H), 3.25(s, 3H); ^1^C NMR (100 MHz, CDCl_{3}) δ 160.0, 130.5, 127.9, 114.3, 77.9, 57.2, 56.7, 56.1, 55.4; HRMS (ESI) m/z [M + Na]^+ calcd for C_{11}H_{16}O_{4}NaS^+ 283.0611, found 283.0619.

3ae: Methyl 2-(4-(tert-butyl)phenyl)-2-methoxyethane-1-sulfonate

^1^H NMR (400 MHz, CDCl_{3}) δ 7.44-7.37 (m, 2H), 7.29-7.23 (m, 2H), 4.71 (dd, J = 9.4, 2.9 Hz, 1H), 3.88 (s, 3H), 3.60 (dd, J = 14.9, 9.4 Hz, 1H), 3.29 (dd, J = 14.9, 2.9 Hz, 1H), 3.28 (s, 1H), 1.32 (s, 9H); ^1^C NMR (100 MHz, CDCl_{3}) δ 151.9, 135.5, 126.3, 125.9, 78.1, 57.2, 57.0, 56.1, 34.7, 31.3; HRMS (ESI) m/z [M + Na]^+ calcd for C_{14}H_{22}O_{4}NaS^+ 309.1131, found 309.1140.
3af: Methyl 2-(4-fluorophenyl)-2-methoxyethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.37-7.30 (m, 2H), 7.13-7.04 (m, 2H), 4.71 (dd, $J = 9.0$, 3.4 Hz, 1H), 3.88 (s, 3H), 3.59 (dd, $J = 14.8$, 9.0 Hz, 1H), 3.27(dd, $J = 14.8$, 3.4 Hz, 1H), 3.27(s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 162.9 (d, $J = 247.6$ Hz), 134.4 (d, $J = 3.2$ Hz), 128.3 (d, $J = 8.3$ Hz), 116.0 (d, $J = 21.7$ Hz), 77.8, 57.1, 57.0, 56.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{10}$H$_{13}$O$_4$FNaS$^+$ 271.0411, found 271.0422.

3ag: Methyl 2-(4-chlorophenyl)-2-methoxyethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.41-7.34 (m, 2H), 7.31-7.27 (m, 2H), 4.70 (dd, $J = 8.9$, 3.4 Hz, 1H), 3.89 (s, 3H), 3.58 (dd, $J = 14.8$, 9.0 Hz, 1H), 3.28(s, 3H), 3.26(dd, $J = 14.8$, 3.4 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 137.2, 134.7, 129.3, 128.0, 77.8, 57.1, 57.0, 56.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{10}$H$_{13}$O$_4$ClNaS$^+$ 287.0115, found 287.0127.

3ah: Methyl 2-(4-bromophenyl)-2-methoxyethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.57-7.50 (m, 2H), 7.26-7.20 (m, 2H), 4.69 (dd, $J = 9.0$, 3.4 Hz, 1H), 3.89 (s, 3H), 3.58 (dd, $J = 14.8$, 9.0 Hz, 1H); 3.28(s, 3H), 3.26(dd, $J = 14.8$, 3.4 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 137.7, 132.2, 128.3, 122.8, 77.9, 57.1, 57.0, 56.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{10}$H$_{13}$O$_4$BrNaS$^+$ 330.9610, found 330.9615.

3ai: Methyl 2-(4-(chloromethyl)phenyl)-2-methoxyethane-1-sulfonate
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.43 (d, $J = 8.1$ Hz, 2H), 7.35 (d, $J = 8.1$ Hz, 2H), 4.74 (dd, $J = 9.1$, 3.2 Hz, 1H), 4.59 (s, 2H), 3.89 (s, 3H), 3.59 (dd, $J = 14.8$, 9.1 Hz, 1H), 3.29(s, 1H), 3.28(dd, $J = 14.8$, 3.2 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 138.9, 138.2, 129.3, 127.0, 78.1, 57.1, 56.1, 45.7; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{11}$H$_{15}$O$_4$NaCl$^+$ 301.0272, found 3301.0277.

3aj: Methyl 2-mesityl-2-methoxyethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 6.84 (s, 2H), 5.23 (dd, $J = 8.9$, 3.0 Hz, 1H), 3.89 (s, 3H), 3.84 (dd, $J = 15.0$, 8.9 Hz, 1H), 3.28 (dd, $J = 15.0$, 3.0 Hz, 1H), 3.23 (s, 3H), 2.39 (s, 6H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 137.9, 136.8, 130.8, 130.4 (m), 74.6, 56.3, 56.1, 54.6, 20.8, 20.3 (m); HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{13}$H$_{20}$O$_4$NaS$^+$ 295.0975, found 295.0985.

3ak: Methyl 2-methoxy-2-(3,4,5-trimethoxyphenyl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 6.58 (s, 2H), 4.67 (dd, $J = 9.5$, 2.7 Hz, 1H), 3.92 (s, 3H), 3.88 (s, 6H), 3.85 (s, 3H), 3.60 (dd, $J = 14.9$, 9.5 Hz, 1H), 3.32 (s, 2H), 3.31 (dd, $J = 14.9$, 2.7 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 153.6, 137.9, 134.2, 103.0, 78.4, 60.7, 57.1, 57.0, 56.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{13}$H$_{20}$O$_7$NaS$^+$ 343.0822, found 343.0832.

3al: Methyl 2-methoxy-2-(naphthalen-2-yl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.96-7.73 (m, 4H), 7.60-7.47 (m, 2H), 7.44 (dd, $J = 8.5$, 1.7 Hz, 1H), 4.89 (dd, $J = 9.1$, 3.1 Hz, 1H), 3.88 (s, 3H), 3.70 (dd, $J = 14.9$, 9.2 Hz, 1H), 3.37 (dd, $J = 14.9$, 3.2 Hz, 1H), 3.31 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ
135.8, 133.5, 133.2, 129.1, 128.0, 127.8, 126.6, 126.2, 123.5, 78.5, 57.0, 56.1; HRMS (ESI) m/z [M + Na]^+ calcd for C_{14}H_{16}O_4NaS^+ 303.0662, found 303.0673.

![Structure of 3am](image)

**3am**: Methyl 2-methoxy-2-(naphthalen-1-yl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.13 (d, $J$ = 8.3 Hz, 1H), 7.90 (d, $J$ = 8.1 Hz, 1H), 7.84 (d, $J$ = 8.2 Hz, 1H), 7.67-7.43 (m, 4H), 5.49 (dd, $J$ = 9.3, 2.0 Hz, 1H), 3.93 (s, 3H), 3.69 (dd, $J$ = 15.1, 9.3 Hz, 1H), 3.45 (dd, $J$ = 15.1, 2.3 Hz, 1H), 3.39 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 134.1, 133.8, 130.3, 129.2, 126.9, 126.1, 125.5, 124.2, 122.4, 76.3, 57.4, 56.4, 56.3; HRMS (ESI) m/z [M + Na]^+ calcd for C$_{14}$H$_{16}$O$_4$NaS$^+$ 303.0662, found 303.0673.

![Structure of 3an](image)

**3an**: Methyl 2-methoxy-2-(thiophen-2-yl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 (dd, $J$ = 5.0, 0.8 Hz, 1H), 7.07 (dd, $J$ = 3.5, 0.7 Hz, 1H), 7.01 (dd, $J$ = 5.0, 3.5 Hz, 1H), 5.00 (dd, $J$ = 9.0, 3.5 Hz, 1H), 3.89 (s, 3H), 3.71 (dd, $J$ = 14.8, 9.0 Hz, 1H), 3.42 (dd, $J$ = 14.8, 3.5 Hz, 1H), 3.33 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 141.9, 126.9, 126.5, 126.3, 74.1, 57.2, 56.9, 56.3; HRMS (ESI) m/z [M + Na]^+ calcd for C$_8$H$_{12}$O$_4$NaS$_2$$^+$ 259.0069, found 259.0079.

![Structure of 3ao](image)

**3ao**: Methyl 2-methoxy-2-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.45-7.35 (m, 4H), 7.35-7.29 (m, 1H), 3.73 (s, 3H), 3.59 (d, $J$ = 14.7 Hz, 1H), 3.43 (d, $J$ = 14.7 Hz, 1H), 3.11 (s, 3H), 1.90 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 142.0, 128.6, 128.1, 126.2, 76.9, 61.5, 55.8, 50.4, 21.0; HRMS (ESI) m/z [M + Na]^+ calcd for C$_{11}$H$_{16}$O$_4$NaS$^+$ 267.0662, found 267.0672.
3ap: Methyl 2-((1,1'-biphenyl)-4-yl)-3,3,3-trifluoro-2-methoxypropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.70-7.56 (m, 6H), 7.49-7.40 (m, 2H), 7.39-7.32 (m, 1H), 4.06 (d, $J$ = 15.4 Hz, 1H), 3.90 (d, $J$ = 15.4 Hz, 1H), 3.70 (s, 3H), 3.65-3.59 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 142.2, 140.0, 131.9, 128.9, 127.8, 127.68, 127.67, 127.2, 127.1, 124.3 (q, $J$ = 291.4 Hz), 80.0 (q, $J$ = 27.4 Hz), 56.1, 54.1 (d, $J$ = 1.8 Hz), 52.4; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{17}$O$_4$F$_3$NaS$^+$ 397.0692, found 397.0696.

3aq: Methyl 2-cyclobutyl-2-methoxy-2-phenylethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.44-7.37 (m, 2H), 7.36-7.29 (m, 3H), 3.78(s, 3H), 3.73 (dd, $J$ = 29.1, 14.9 Hz, 2H), 3.37-3.20 (m, 4H), 2.02-1.88 (m, 2H), 1.88-1.68 (m, 3H), 1.56-1.41 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 139.0, 128.1, 127.7, 127.2, 80.9, 55.4, 53.9, 51.9, 42.5, 24.2, 23.7, 17.3; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{14}$H$_{20}$O$_4$NaS$^+$ 307.0975, found 307.0983.

3ar: Methyl 2-cyclohexyl-2-methoxy-2-phenylethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39-7.21 (m, 5H), 4.05 (d, $J$ = 15.1 Hz, 1H), 3.83(d, $J$ = 15.1 Hz, 1H), 3.82(s, 3H), 3.34 (s, 3H), 2.28-2.18 (m, 1H), 2.08-1.94 (m, 1H), 1.79-1.61 (m, 3H), 1.60-1.49 (m, 1H), 1.38-1.08 (m, 2H), 0.92-0.77 (m, 1H), 0.76-0.62 (m, 1H), 0.54-0.36 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 138.2, 127.6, 127.4, 127.1, 82.5, 55.4, 51.9, 51.0, 44.7, 28.6, 26.5, 26.4, 26.09, 26.08; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{22}$O$_4$NaS$^+$ 335.1288, found 335.1293.
**3as**: Methyl 2-methoxy-2,2-diphenylethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.37-7.29 (m, 8H), 7.28-7.22 (m, 2H), 4.26 (s, 2H), 3.51 (s, 3H), 3.21 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 142.9, 128.3, 127.6, 126.7, 80.4, 55.5, 55.4, 51.5; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{18}$O$_4$NaS$^+$ 329.0818, found 329.0824.

**3at**: Methyl 2-(4-fluorophenyl)-2-methoxy-2-phenylethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.37-7.23 (m, 7H), 7.03-6.95 (m, 2H), 4.26 (d, $J = 14.8$ Hz, 1H), 4.20 (d, $J = 14.8$ Hz, 1H), 3.57 (s, 3H), 3.19 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 161.9 (d, $J = 247.0$ Hz), 142.5, 138.8 (d, $J = 3.3$ Hz), 128.5 (d, $J = 8.3$ Hz), 128.4, 127.9, 126.6, 115.1 (d, $J = 21.5$ Hz), 80.1, 55.4, 55.2, 51.4; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{17}$O$_4$FNaS$^+$ 347.0724, found 347.0732.

**3au**: Methyl 2-methoxy-2-phenyl-2-(pyridin-3-yl)ethane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.54-8.30 (m, 1H), 7.71-7.54 (m, 2H), 7.40-7.35 (m, 2H), 7.34-7.27 (m, 2H), 7.25-7.19 (m, 1H), 7.15-7.10 (m, 1H), 4.83 (d, $J = 14.9$ Hz, 1H), 4.38 (d, $J = 14.9$ Hz, 1H), 3.62 (s, 3H), 3.32 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 161.9, 148.1, 141.8, 136.9, 128.3, 127.6, 126.1, 122.3, 121.0, 81.8, 55.6, 53.5, 51.8; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{15}$H$_{17}$NO$_4$NaS$^+$ 330.0770, found 330.0776.
3av: Methyl 1-methoxy-1-phenylpropane-2-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.44-7.37 (m, 2H), 7.36-7.28 (m, 3H), 4.94 (d, $J = 2.5$ Hz, 1H), 3.91 (s, 3H), 3.42-3.22 (m, 4H), 1.35 (d, $J = 7.1$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 137.8, 128.8, 128.3, 126.6, 80.1, 63.3, 57.6, 55.9, 8.2; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{11}$H$_{16}$O$_4$NaS$^+$ 267.0662, found 267.0672.

3aw: Methyl 1-methoxy-1,1-diphenylpropane-2-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51-7.45 (m, 2H), 7.45-7.31 (m, 8H), 4.53 (q, $J = 7.0$ Hz, 1H), 3.71 (s, 3H), 2.91 (s, 3H), 1.50 (d, $J = 7.0$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 138.6, 137.7, 129.5, 129.3, 128.2, 128.0, 127.8, 127.4, 84.7, 63.5, 54.8, 52.0, 13.2; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{20}$O$_4$NaS$^+$ 343.0975, found 343.0981.

3ax: Methyl 1-(methoxydiphenylmethyl)cyclopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.62-7.55 (m, 4H), 7.41-7.31 (m, 6H), 3.19 (s, 3H), 2.82 (s, 3H), 1.89-1.78 (m, 2H), 1.68-1.56 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 137.9, 130.3, 128.0, 127.3, 81.6, 54.4, 51.1, 47.0, 12.3; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{18}$H$_{20}$O$_4$NaS$^+$ 355.0975, found 355.0979.

3ay: Methyl 1-((2-(benzyloxy)phenyl)(methoxy)methyl)cyclopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51-7.44 (m, 2H), 7.43-7.36 (m, 2H), 7.35-7.26 (m, 3H), 7.07-6.84 (m, 2H), 5.59 (s, 1H), 5.17-5.03 (m, 2H), 3.76 (s, 3H), 3.31 (s, 3H),
1.40-1.24 (m, 2H), 1.09-1.00 (m, 1H), 0.31 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 156.5, 136.6, 129.5, 128.6, 128.1, 127.5, 126.8, 124.5, 120.9, 111.8, 75.1, 70.2, 57.6, 56.5, 41.2, 11.5, 7.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{19}$H$_{22}$O$_5$NaS$^+$ 385.1080, found 385.1088.

3az: Methyl-2-methoxy-2-phenylcyclohexane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.49-7.43 (m, 2H), 7.41-7.34 (m, 2H), 7.33-7.27 (m, 1H), 3.59-3.55 (m, 1H), 3.18 (s, 3H), 2.90 (s, 3H), 2.84-2.73 (m, 1H), 2.47-2.30 (m, 2H), 2.11-1.98 (m, 1H), 1.85-1.70 (m, 2H), 1.69-1.54 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 141.4, 128.1, 127.9, 127.6, 77.2, 66.9, 54.5, 48.7, 24.6, 24.1, 20.6, 20.4; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{14}$H$_{20}$O$_4$NaS$^+$ 307.0975, found 307.0985.

3ba: Methyl-1-methoxy-2,3-dihydro-1$H$-indene-2-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.43-7.39 (m, 1H), 7.35-7.28 (m, 2H), 7.28-7.23 (m, 1H), 5.32 (d, $J$ = 4.0 Hz, 1H), 4.04-3.95 (m, 1H), 3.92 (s, 3H), 3.59 (s, 3H), 3.56-3.34 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.8, 139.3, 129.5, 127.7, 125.3, 124.8, 85.6, 64.4, 57.8, 55.5, 32.7; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{11}$H$_{14}$O$_4$NaS$^+$ 265.0505, found 265.0511.

3bb: Methyl 1-methoxy-1,2,3,4-tetrahydronaphthalene-2-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.41-7.10 (m, 4H), 4.84 (d, $J$ = 5.3 Hz, 0.67H), 4.66 (d, $J$ = 2.2 Hz, 0.33H), 3.94 (s, 1H), 3.89 (s, 2H), 3.76-3.69 (m, 0.67H), 3.52-3.45 (m, 0.33H), 3.42 (s, 2H), 3.40 (s, 1H), 3.17-3.07 (m, 0.33H), 2.96-2.81 (m, 1.67H), 2.63-2.51 (m, 0.33H), 2.49-2.38 (m, 0.67H), 2.34-2.23 (m, 0.33H), 2.22-2.09 (m, 0.67H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 137.0, 135.7, 133.7, 132.9, 130.0, 129.5, 129.2, 128.7,
128.4, 128.4, 126.5, 125.7, 76.2, 75.4, 62.3, 60.2, 56.6, 56.5, 56.0, 55.7, 27.7, 26.7, 22.5, 18.7; HRMS (ESI) m/z [M + Na]+ calcd for C_{12}H_{16}O_4NaS^+ 279.0662, found 279.0667.

3bc: Methyl 4-(1-methoxy-2-(methoxysulfonyl)-1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)ethyl)benzoate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.96 (d, $J = 8.8$ Hz, 2H), 7.45 (d, $J = 7.8$ Hz, 2H), 7.23 (s, 1H), 6.94 (s, 1H), 4.36 (d, $J = 14.5$ Hz, 1H), 4.09 (d, $J = 14.5$ Hz, 1H), 3.88 (s, 3H), 3.52 (s, 3H), 3.20 (s, 3H), 1.84 (s, 3H), 1.74-1.64 (m, 4H), 1.36-1.31 (m, 6H), 1.24 (s, 3H), 1.23 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.8, 147.7, 145.1, 142.0, 135.6, 134.2, 131.2, 128.9, 128.8, 127.3, 124.3, 81.2, 56.5, 55.1, 52.1, 51.1, 35.2, 35.0, 34.1, 33.9, 32.1, 32.0, 31.7, 31.6, 20.4; HRMS (ESI) m/z [M + Na]+ calcd for C$_{27}$H$_{36}$O$_6$NaS$^+$ 511.2125, found 511.2129.

3bd: Methyl 1-methoxy-1-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)propane-2-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.31 (d, $J = 8.0$ Hz, 1H), 7.11 (d, $J = 8.0$ Hz, 1H), 7.06 (s, 1H), 4.67 (dd, $J = 9.4$, 2.1 Hz, 1H), 3.91 (s, 3H), 3.59 (dd, $J = 14.9$, 9.5 Hz, 1H), 3.34-3.19 (m, 4H), 2.93 (dd, $J = 8.7$, 3.9 Hz, 2H), 2.57-2.47 (m, 1H), 2.46-2.39 (m, 1H), 2.35-2.26 (m, 1H), 2.21-2.12 (m, 1H), 2.12-2.02 (m, 2H), 2.00-1.94 (m, 1H), 1.69-1.42 (m, 6H), 0.92 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 220.9, 140.4, 137.3, 137.3, 135.9, 127.1, 127.0, 126.0, 123.9, 123.8, 57.2, 57.2, 57.0, 56.2, 50.5, 48.0, 44.4,
38.0, 35.9, 31.6, 29.4, 29.4, 26.4, 25.7, 21.6, 13.9; HRMS (ESI) m/z [M + Na]⁺ calcd for C₂₂H₃₄O₄NaS⁺ 429.1706, found 429.1711.

3be: Methyl 4-([1,1'-biphenyl]-4-yl)-2-methoxy-2-methylbut-3-yn-1-sulfonate

¹H NMR (400 MHz, CDCl₃) δ 7.62-7.50 (m, 6H), 7.49-7.42 (m, 2H), 7.40-7.33 (m, 1H), 3.95 (s, 3H), 3.71-3.55 (m, 2H), 3.50 (s, 3H), 1.81 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 141.7, 140.2, 132.3, 128.9, 127.8, 127.10, 127.08, 120.7, 87.4, 87.2, 71.1, 58.6, 56.2, 52.0, 25.7; HRMS (ESI) m/z [M + Na]⁺ calcd for C₁₉H₂₀O₄NaS⁺ 367.0975, found 367.0987.

3bf: Butyl 2-([1,1'-biphenyl]-4-yl)-2-butoxyethane-1-sulfonate

¹H NMR (400 MHz, CDCl₃) δ 7.63-7.55 (m, 4H), 7.48-7.39 (m, 4H), 7.38-7.32 (m, 1H), 4.87 (dd, J = 8.7, 3.5 Hz, 1H), 4.27-4.12 (m, 2H), 3.62 (dd, J = 14.8, 8.8 Hz, 1H), 3.40 (t, J = 6.6 Hz, 2H), 3.32 (dd, J = 14.8, 3.6 Hz, 1H), 1.72-1.62 (m, 2H), 1.61-1.52 (m, 2H), 1.44-1.33 (m, 4H), 0.96-0.86 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 141.6, 140.5, 138.6, 128.9, 127.63, 127.59, 127.1, 127.0, 76.6, 70.3, 69.3, 57.6, 31.8, 31.2, 19.3, 18.7, 13.9, 13.5; HRMS (ESI) m/z [M + Na]⁺ calcd for C₂₂H₃₄O₄NaS⁺ 413.1757, found 413.1766.

3bg: Isopropyl 2-([1,1'-biphenyl]-4-yl)-2-isopropoxyethane-1-sulfonate

¹H NMR (400 MHz, CDCl₃) δ 7.63-7.53 (m, 4H), 7.49-7.40 (m, 4H), 7.39-7.31 (m, 1H), 5.02 (dd, J = 8.8, 3.5 Hz, 1H), 4.98-4.90 (m, 1H), 3.67-3.50 (m, 2H), 3.28 (dd, J = 14.6, 3.5 Hz, 1H), 1.40 (d, J = 6.3 Hz, 3H), 1.37 (d, J = 6.2 Hz, 3H), 1.24 (d, J = 6.0 Hz, 3H), 1.11 (d, J = 6.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 141.4, 140.5, 139.5,
128.8, 127.5, 127.1, 127.0, 76.9, 73.9, 70.1, 59.0, 23.3, 23.2, 23.1, 21.0; HRMS (ESI) m/z [M + Na]+ calcd for C_{20}H_{26}O_{4}NaS^{+} 385.1444, found 385.1448.

3bh: Bethyl-d₅ 2-((1,1'-biphenyl)-4-yl)-2-(methoxy-d₃)ethane-1-sulfonate-1,1-d₂
$^1$H NMR (400 MHz, CDCl₃) δ 7.68-7.52 (m, 4H), 7.51-7.33 (m, 5H), 4.77 (s, 1H); $^{13}$C NMR (100 MHz, CDCl₃) δ 141.8, 140.4, 137.5, 128.9, 127.7, 127.6, 127.1, 127.0, 77.9, 56.9 (m), 56.3 (m), 55.5 (m); HRMS (ESI) m/z [M + Na]+ calcd for C_{16}H_{10}D₈O₄NaS⁺ 337.1320, found 337.1326.

3bi: Methyl 2-cyclopropyl-2-methoxy-2-phenylethane-1-sulfonate
$^1$H NMR (400 MHz, CDCl₃) δ 7.44-7.27 (m, 5H), 3.86 (d, J = 14.7 Hz, 1H), 3.79 (s, 3H), 3.72 (d, J = 14.7 Hz, 1H), 3.31 (s, 3H), 1.61-1.48 (m, 1H), 0.67-0.50 (m, 2H), 0.49-0.39 (m, 1H), 0.37-0.27 (m, 1H); $^{13}$C NMR (100 MHz, CDCl₃) δ 139.5, 128.1, 127.9, 127.0, 79.2, 55.9, 55.5, 51.6, 19.4, 2.7, 2.0; HRMS (ESI) m/z [M + Na]+ calcd for C_{13}H_{18}O₄NaS⁺ 293.0818, found 293.0828.

3bj: Methyl 2-methoxy-2-(4-(trifluoromethyl)phenyl)ethane-1-sulfonate
$^1$H NMR (400 MHz, CDCl₃) δ 7.67 (d, J = 8.1 Hz, 2H), 7.49 (d, J = 8.1 Hz, 2H), 4.80 (q, J = 9.0, 3.3 Hz, 1H), 3.90 (s, 3H), 3.60 (q, J = 14.8, 9.0 Hz, 1H), 3.30 (s, 3H), 3.25 (q, J = 14.8, 3.3 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl₃) δ 142.8, 131.1 (q, J = 32.7 Hz), 127.0, 126.1 (q, J = 3.7 Hz), 123.9 (q, J = 272.1 Hz), 78.0, 57.4, 56.9, 56.1; HRMS (ESI) m/z [M + Na]+ calcd for C_{11}H_{13}O₄F₃NaS⁺ 321.0379, found 321.0385.
5a: Methyl 2-methyl-3-oxo-2,3-diphenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35-7.42 (m, 8H), 7.21-7.25 (m, 2H), 4.03 (d, $J = 14.8$ Hz, 1H), 3.77 (d, $J = 14.8$ Hz, 1H), 3.64 (s, 3H), 2.02 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 200.7, 139.4, 135.7, 132.2, 129.4, 129.4, 128.3, 128.2, 126.6, 59.2, 55.5, 52.9, 21.5; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{18}$NaO$_4$S$^+$ 341.0818, found 341.0832.

5b: Methyl 2-methyl-3-oxo-2,3-di-p-tolylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.33 (d, $J = 7.5$ Hz, 2H), 7.27 (d, $J = 7.4$ Hz, 2H), 7.20 (d, $J = 7.2$ Hz, 2H), 7.03 (d, $J = 7.5$ Hz, 2H), 3.99 (d, $J = 14.4$ Hz, 1H), 3.74 (d, $J = 14.7$ Hz, 1H), 3.64 (s, 3H), 2.35 (s, 3H), 2.29 (s, 3H), 2.00 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 200.3, 142.9, 137.9, 136.6, 132.8, 130.0, 129.8, 128.9, 126.5, 59.3, 55.5, 52.6, 21.6, 21.5, 21.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{19}$H$_{22}$NaO$_4$S$^+$ 369.1131, found 369.1141.

5c: Methyl 2,3-bis(4-methoxyphenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.47 (d, $J = 8.9$ Hz, 2H), 7.29 (d, $J = 8.8$ Hz, 2H), 6.93 (d, $J = 8.8$ Hz, 2H), 6.73 (d, $J = 8.9$ Hz, 2H), 3.93 (d, $J = 14.9$ Hz, 1H), 3.81 (s, 3H), 3.75-3.78 (m, 4H), 3.65 (s, 3H), 2.02 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 199.1,
162.6, 159.3, 132.2, 131.6, 127.9, 127.6, 114.6, 113.4, 59.4, 55.5, 55.4, 55.3, 52.2, 21.9; HRMS (ESI) m/z [M + Na]$^+$ calced for C$_{19}$H$_{22}$NaO$_6$S$^+$ 401.1029, found 401.1035.

5d: Methyl 2,3-bis(3-fluorophenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.39-7.45 (m, 1H), 7.21-7.27 (m, 1H), 7.07-7.18 (m, 6H), 3.99 (d, $J = 14.8$ Hz, 1H), 3.72–3.75 (m, 4H), 2.02 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 198.9 (d, $^4J_{C-F} = 3.0$ Hz), 163.3 (d, $^1J_{C-F} = 247.0$ Hz), 162.2 (d, $^1J_{C-F} = 247.0$ Hz), 141.6 (d, $^3J_{C-F} = 7.0$ Hz), 137.3 (d, $^3J_{C-F} = 6.0$ Hz), 131.1 (d, $^3J_{C-F} = 8.0$ Hz), 130.0 (d, $^2J_{C-F} = 7.0$ Hz), 125.0 (d, $^4J_{C-F} = 3.0$ Hz), 122.4 (d, $^4J_{C-F} = 2.0$ Hz), 119.4 (d, $^2J_{C-F} = 22.0$ Hz), 116.3 (d, $^2J_{C-F} = 23.0$ Hz), 115.6 (d, $^2J_{C-F} = 21.0$ Hz), 113.9 (d, $^2J_{C-F} = 22.0$ Hz), 58.8, 55.6, 52.8 (d, $^4J_{C-F} = 1.0$ Hz), 21.3; HRMS (ESI) m/z [M + Na]$^+$ calced for C$_{17}$H$_{16}$F$_2$NaO$_4$S$^+$ 377.0630, found 377.0637.

5e: Methyl 2,3-bis(4-fluorophenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.43-7.47 (m, 2H), 7.36-7.39 (m, 2H), 7.12 (t, $J = 8.5$ Hz, 2H), 6.94 (t, $J = 8.5$ Hz, 2H), 3.94 (d, $J = 14.8$ Hz, 1H), 3.77 (d, $J = 14.8$ Hz, 1H), 3.69 (s, 3H), 2.03 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 198.8, 164.9 (d, $^1J_{C-F} = 254.0$ Hz), 162.5 (d, $^1J_{C-F} = 247.0$ Hz), 135.0 (d, $^4J_{C-F} = 4.0$ Hz), 132.3 (d, $^3J_{C-F} = 9.0$ Hz), 131.3 (d, $^4J_{C-F} = 3.0$ Hz), 128.5 (d, $^3J_{C-F} = 8.0$ Hz), 116.4 (d, $^2J_{C-F} = 21.0$ Hz), 115.4 (d, $^2J_{C-F} = 21.0$ Hz), 59.1, 55.5, 52.4, 21.8; HRMS (ESI) m/z [M + Na]$^+$ calced for C$_{17}$H$_{16}$F$_2$NaO$_4$S$^+$ 377.0630, found 377.0635.
5f: Methyl 2,3-bis(3-chlorophenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.47 (t, $J = 1.7$ Hz, 1H), 7.36-7.41 (m, 4H), 7.27-7.30 (m, 1H), 7.19 (t, $J = 7.8$ Hz, 1H), 7.13-7.15 (m, 1H), 4.00 (d, $J = 14.8$ Hz, 1H), 3.70-3.73 (m, 4H), 2.00 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 198.9, 141.0, 136.9, 135.6, 134.7, 132.4, 130.8, 129.6, 129.4, 128.8, 127.2, 126.8, 124.9, 58.8, 55.6, 52.8, 21.3; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{16}$Cl$_2$NaO$_4$S$^+$ 409.0039, found 409.0045.

5g: Methyl 2,3-bis(4-chlorophenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40 (d, $J = 8.6$ Hz, 2H), 7.35 (d, $J = 8.7$ Hz, 2H), 7.32 (d, $J = 8.7$ Hz, 2H), 7.24 (d, $J = 8.6$ Hz, 2H), 3.94 (d, $J = 14.8$ Hz, 1H), 3.90 (d, $J = 14.8$ Hz, 1H), 3.70 (s, 3H), 2.01 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 199.0, 138.9, 137.6, 134.5, 133.3, 131.0, 129.7, 128.7, 128.1, 58.9, 55.5, 52.6, 21.5; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{16}$Cl$_2$NaO$_4$S$^+$ 409.0039, found 409.0049.

5h: Methyl 2,3-bis(4-bromophenyl)-2-methyl-3-oxopropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.56 (d, $J = 8.2$ Hz, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.24-7.28 (m, 4H), 3.93 (d, $J = 14.8$ Hz, 1H), 3.70-3.74 (m, 4H), 2.00 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 199.1, 138.1, 133.7, 132.7, 131.7, 131.0, 128.4, 127.6, 122.8, 58.9, 55.5, 52.6, 21.4; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{16}$Br$_2$NaO$_4$S$^+$ 496.9028, found 496.9032.
5i: Methyl 3-oxo-2,3-diphenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.96-7.98 (m, 2H), 7.49-7.53 (m, 1H), 7.39-7.42 (m, 2H), 7.30-7.32 (m, 4H), 7.24-7.28 (m, 1H), 5.23 (dd, $J = 4.2, 8.4$ Hz, 1H), 4.36 (dd, $J = 8.4, 14.7$ Hz, 1H), 3.74 (s, 3H), 3.41 (dd, $J = 4.2, 14.6$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 196.0, 136.2, 135.4, 133.7, 129.5, 128.9, 128.8, 128.3, 128.1, 56.1, 52.0, 48.5; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{16}$NaO$_4$S$^+$ 327.0662, found 327.0668.

5j: Methyl 2-methyl-3-oxo-2-phenylbutane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39-7.42 (m, 2H), 7.32-7.35 (m, 1H), 7.28-7.30 (m, 2H), 3.91 (d, $J = 14.9$ Hz, 1H), 3.61-3.66 (m, 4H), 1.98 (s, 3H), 1.94 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 207.1, 138.5, 129.2, 128.2, 126.5, 57.3, 55.5, 53.6, 25.1, 19.4; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{12}$H$_{16}$NaO$_4$S$^+$ 279.0662, found 279.0667.

5k: Methyl 2-((1,1'-biphenyl)-4-yl)-2-methyl-3-oxobutane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.63 (d, $J = 8.4$ Hz, 2H), 7.59 (d, $J = 7.3$ Hz, 2H), 7.45 (t, $J = 7.3$ Hz, 2H), 7.34-7.38 (m, 3H), 3.96 (d, $J = 14.8$ Hz, 1H), 3.63-3.67 (m, 4H), 2.03 (s, 3H), 1.97 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 207.1, 141.0, 140.0, 137.5, 128.9, 127.7, 127.0, 126.9, 57.3, 55.5, 53.4, 25.2, 19.5; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{18}$H$_{20}$NaO$_4$S$^+$ 355.0975, found 355.0981.

5l: Methyl 2-methyl-3-oxo-2-phenylhexane-1-sulfonate

5m: Methyl 2-methyl-3-oxo-2-phenylhexane-1-sulfonate
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38-7.42 (m, 2H), 7.31-7.35 (m, 1H), 7.26-7.28 (m, 2H), 3.95 (d, \(J = 14.9\) Hz, 1H), 3.60-3.64 (m, 4H), 2.28-2.36 (m, 1H), 2.10-2.18 (m, 1H), 1.93 (s, 3H), 1.42-1.61 (m, 2H), 0.76 (t, \(J = 7.4\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 209.1, 138.6, 129.1, 128.1, 126.5, 57.5, 55.5, 53.3, 38.9, 19.2, 17.5, 13.5; HRMS (ESI) m/z [M + Na]\(^+\) calcd for C\(_{14}\)H\(_{20}\)NaO\(_4\)S\(^+\) 307.0975, found 307.0980.

\(5m\): Methyl 2,4-dimethyl-3-oxo-2-phenylpentane-1-sulfonate

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38-7.42 (m, 2H), 7.32-7.35 (m, 1H), 7.27-7.30 (m, 2H), 3.88 (d, \(J = 15.0\) Hz, 1H), 3.68 (d, \(J = 15.0\) Hz, 1H), 3.59 (s, 3H), 2.62-2.72 (m, 1H), 1.98 (s, 3H), 1.06 (d, \(J = 6.8\) Hz, 3H), 0.78 (d, \(J = 6.6\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 213.6, 137.6, 128.9, 128.2, 127.0, 57.4, 55.4, 53.9, 35.4, 21.2, 20.9, 18.6; HRMS (ESI) m/z [M + Na]\(^+\) calcd for C\(_{14}\)H\(_{20}\)NaO\(_4\)S\(^+\) 307.0975, found 307.0980.

\(5n\): Methyl 4,4,4-trifluoro-2-methyl-3-oxo-2-phenylbutane-1-sulfonate

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38-7.46 (m, 3H), 7.24-7.26 (m, 2H), 4.08 (d, \(J = 14.8\) Hz, 1H), 3.75 (s, 3H), 3.56 (d, \(J = 14.8\) Hz, 1H), 3.09 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 189.8 (q, \(^2J_{C-F} = 33.02\) Hz), 135.3, 129.6, 129.0, 126.1, 115.9 (q, \(^1J_{C-F} = 292.0\) Hz), 57.5, 55.7, 51.5, 17.9 (q, \(^4J_{C-F} = 2.0\) Hz); HRMS (ESI) m/z [M + Na]\(^+\) calcd for C\(_{12}\)H\(_{13}\)F\(_3\)O\(_4\)S\(^+\) 333.0379, found 333.0384.

\(5o\): Methyl 3-cyclopentyl-2-methyl-3-oxo-2-phenylpropane-1-sulfonate

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38-7.42 (m, 2H), 7.25-7.34 (m, 3H), 3.87 (d, \(J = 15.0\) Hz, 1H), 3.66 (d, \(J = 15.0\) Hz, 1H), 3.57 (s, 3H), 2.66-2.74 (m, 1H), 1.94 (s, 3H),
1.77-1.83 (m, 1H), 1.58-1.72 (m, 4H), 1.46-1.54 (m, 1H), 1.34-1.41 (m, 1H), 1.24-1.29 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 213.7, 138.2, 129.0, 128.0, 127.0, 57.4, 55.4, 53.8, 46.4, 33.1, 33.0, 26.6, 26.3, 18.7; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{16}$H$_{22}$NaO$_4$S$^+$ 333.1131, found 333.1137.

5p: Methyl 3-cyclohexyl-2-methyl-3-oxo-2-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38-7.41 (m, 2H), 7.27-7.34 (m, 3H), 3.86 (d, $J = 15.0$ Hz, 1H), 3.66 (d, $J = 15.0$ Hz, 1H), 3.59 (s, 2H), 3.59 (s, 3H), 2.34-2.41 (m, 1H), 1.97 (s, 3H), 1.71-1.72 (m, 2H), 1.53-1.58 (m, 2H), 1.34-1.43 (m, 2H), 1.12-1.17 (m, 2H), 1.04-1.07 (m, 1H), 0.89-0.97 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 212.1, 137.5, 128.9, 128.1, 127.0, 57.4, 55.4, 53.8, 46.0, 30.9, 30.8, 25.6, 25.5, 25.4, 18.6; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{24}$NaO$_4$S$^+$ 347.1288, found 347.1285.

5qa: Methyl 3-cyclopropyl-2-methyl-3-oxo-2-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.43 (m, 2H), 7.30-7.36 (m, 3H), 3.85 (d, $J = 15.0$ Hz, 1H), 3.67 (d, $J = 15.0$ Hz, 1H), 3.55 (s, 2H), 2.00 (s, 3H), 1.63-1.68 (m, 1H), 0.97-1.08 (m, 2H), 0.84-0.91 (m, 1H), 0.67-0.73 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 209.8, 138.9, 129.0, 128.0, 127.1, 57.4, 55.4, 53.7, 19.7, 17.6, 12.9, 11.6; HRMS (ESI) m/z [M + H]$^+$ calcd for C$_{14}$H$_{19}$O$_4$S$^+$ 283.0999, found 283.1000.

5qb: Methyl 2-cyclopropyl-2-methyl-3-oxo-3-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.64-7.66 (m, 2H), 7.46-7.49 (m, 1H), 7.39-7.43 (m, 2H), 3.94 (d, $J = 14.1$ Hz, 1H), 3.88 (s, 3H), 3.53 (d, $J = 14.1$ Hz, 1H), 1.21-1.25 (m,
5ra: Methyl 3-cyclobutyl-2-methyl-3-oxo-2-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36-7.40 (m, 2H), 7.28-7.33 (m, 1H), 7.21-7.23 (m, 2H), 3.93 (d, $J$ = 14.9 Hz, 1H), 3.58-3.61 (m, 4H), 3.18-3.26 (m, 1H), 2.14-2.27 (m, 2H), 1.99-2.07 (m, 1H), 1.86 (s, 3H), 1.72-1.82 (m, 2H), 1.55-1.64 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 210.0, 138.4, 129.0, 128.0, 126.7, 57.4, 55.5, 51.1, 44.0, 23.6, 23.5, 18.6, 17.0; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{15}$H$_{20}$NaO$_4$S$^+$ 319.0975, found 319.0982.

5rb: Methyl 2-cyclobutyl-2-methyl-3-oxo-3-phenylpropane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.50-7.52 (m, 2H), 7.45-7.47 (m, 1H), 7.38-7.41 (m, 2H), 4.12 (d, $J$ = 14.1 Hz, 1H), 3.86 (s, 3H), 3.14 (d, $J$ = 14.1 Hz, 1H), 2.68-2.74 (m, 1H), 1.81-1.92 (m, 5H), 1.70-1.74 (m, 1H), 1.55 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 206.7, 139.5, 130.6, 128.0, 127.0, 55.6, 55.4, 51.1, 44.0, 23.6, 23.5, 18.5, 18.1; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{15}$H$_{20}$NaO$_4$S$^+$ 319.0975, found 319.0982.

5s: Methyl 2-benzyl-2-methyl-3-oxo-4-phenylbutane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.23-7.35 (m, 6H), 7.06-7.09 (m, 2H), 7.00-7.01 (m, 2H), 3.97 (d, $J$ = 14.2 Hz, 1H), 3.84 (d, $J$ = 18.0 Hz, 1H), 3.78 (s, 3H), 3.22 (d, $J$ = 18.0 Hz, 1H), 3.11 (d, $J$ = 14.2 Hz, 1H), 2.95 (d, $J$ = 13.0 Hz, 1H), 2.67 (d, $J$ = 13.0 Hz, 1H), 2.09 (s, 3H), 1.80-1.88 (m, 2H), 1.68 (d, $J$ = 13.0 Hz, 1H), 1.56 (s, 3H), 1.42-1.45 (m, 2H), 1.26 (s, 3H), 1.17-1.25 (m, 2H), 1.09-1.16 (m, 2H), 0.90-0.99 (m, 2H), 0.88-0.94 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 206.4, 138.8, 130.8, 128.0, 127.4, 58.4, 55.6, 48.7, 18.8, 18.2, 3.2, 2.4; HRMS (ESI) m/z [M + H]$^+$ calcd for C$_{14}$H$_{15}$O$_4$S$^+$ 283.0999, found 283.0998.
Hz, 1H), 1.64 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 210.0, 134.6, 133.5, 130.5
129.8, 128.5, 128.2, 127.4, 126.7, 57.7, 55.7, 49.8, 46.6, 46.3, 19.5; HRMS (ESI) m/z
[M + Na]$^+$ calcd for C$_{19}$H$_{22}$NaO$_4$S$^+$ 369.1131, found 369.1136.

5t: Methyl 2-methyl-3-oxo-2-pentyloctane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 3.85 (s, 3H), 3.73 (d, $J = 14.4$ Hz, 1H), 3.20 (d, $J =
14.4$ Hz, 1H), 2.45-2.58 (m, 2H), 1.48-1.72 (m, 8H), 1.22-1.35 (m, 9H), 0.85-0.91 (m,
6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 212.0, 55.5, 55.4, 49.0, 38.8, 37.4, 31.8, 31.1,
23.4, 22.9, 22.3, 22.1, 20.6, 13.7; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{15}$H$_{30}$NaO$_4$S$^+$ 329.1757, found 329.1766.

5u: Methyl (9-methyl-10-oxo-9,10-dihydrophenanthren-9-yl)methanesulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.21 (d, $J = 7.8$ Hz, 1H), 8.08-8.11 (m, 2H), 7.70 (t, $J$
= 7.2 Hz, 1H), 7.50-7.52 (m, 1H), 7.41-7.47 (m, 3H), 4.49 (d, $J = 14.6$ Hz, 1H), 3.91
(d, $J = 14.6$ Hz, 1H), 3.59 (s, 3H), 1.51 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 198.5,
138.5, 136.9, 135.1, 129.2, 129.1, 128.5, 128.3, 128.1, 127.9, 127.5, 124.2, 123.3,
57.9, 55.6, 48.8, 30.0; HRMS (ESI) m/z [M + Na]$^+$ calcd for C$_{17}$H$_{16}$O$_4$S$^+$ 339.0662,
found 339.0664.

5v: Methyl 2-(benzo[def]thiazol-2-yl)-5-oxo-5-phenylpentane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.99 (d, $J = 8.2$ Hz, 1H), 7.86-7.88 (m, 3H), 7.47-7.56
(m, 2H), 7.38-7.43 (m, 3H), 4.05 (dd, $J = 7.7$, 14.4 Hz, 1H), 3.92-3.99 (m, 1H), 3.77
(s, 3H), 3.58 (dd, $J = 4.9$, 14.4, Hz, 1H), 3.03 (t, $J = 7.3$ Hz, 2H), 2.37-2.54 (m, 2H);
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 198.4, 170.7, 153.0, 136.5, 134.8, 133.3, 128.6, 128.0, 126.4, 125.4, 123.0, 121.8, 131.9, 55.9, 53.6, 39.1, 35.1, 29.7; HRMS (ESI) m/z [M + Na]$^+$ calcld for C$_{19}$H$_{19}$NNaO$_4$S$_2$ 412.0648, found 412.0654.

5w: Methyl 2-(benzo[d]oxazol-2-yl)-5-oxo-5-phenylpentane-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.86 (d, $J = 7.2$ Hz, 2H), 7.66-7.70 (m, 1H), 7.47-7.55 (m, 2H), 7.40 (t, $J = 7.8$ Hz, 2H), 7.31-7.35 (m, 2H), 3.97 (dd, $J = 8.2$, 14.5 Hz, 1H), 3.81-3.88 (m, 1H), 3.79 (s, 3H), 3.57 (dd, $J = 4.8$, 14.6 Hz, 1H), 3.00-3.10 (m, 2H), 2.34-2.50 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 198.0, 165.3, 150.6, 140.7, 136.3, 133.2, 128.5, 127.8, 125.1, 124.5, 119.8, 110.6, 55.9, 51.9, 35.0, 27.5; HRMS (ESI) m/z [M + Na]$^+$ calcld for C$_{19}$H$_{19}$NNaO$_5$S$^+$ 396.0876, found 396.0885.

7: Methyl (2,4-dimethyl-1,3-dioxo-1,2,3,4-tetrahydroisoquinolin-4-yl)methanesulfonate

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.31 (dd, $J = 7.9$, 1.1 Hz, 1H), 7.73-7.65 (m, 1H), 7.54-7.49 (m, 1H), 7.46 (d, $J = 7.9$ Hz, 1H), 4.35 (d, $J = 14.6$ Hz, 1H), 3.80 (d, $J = 14.6$ Hz, 1H), 3.58 (s, 3H), 3.43 (s, 3H), 1.67 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 174.1, 163.7, 139.2, 133.8, 129.4, 128.4, 125.7, 124.7, 58.6, 55.7, 45.3, 31.0, 27.5; HRMS (ESI) m/z [M + Na]$^+$ calcld for C$_{13}$H$_{15}$NO$_5$NaS$^+$ 320.0563, found 320.0574.

8: Methyl 5-methoxy-2-phenylpent-2-ene-1-sulfonate
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.42-7.38 (m, 2H), 7.37-7.32 (m, 2H), 7.31-7.25 (m, 1H), 6.18 (t, $J = 7.3$ Hz, 1H), 4.37 (s, 2H), 3.65 (s, 3H), 3.54 (t, $J = 6.3$ Hz, 2H), 3.36 (s, 3H), 2.63 (dd, $J = 13.3$, 6.5 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 140.7, 135.2, 128.9, 128.6, 127.7, 126.5, 71.4, 58.8, 56.2, 51.5, 30.1; HRMS (ESI) m/z [M + Na]$^+$ calec for C$_{13}$H$_{18}$O$_4$NaS 293.0818, found 293.0828.

9: Methyl (E)-2-(4-(trifluoromethyl)phenyl)ethene-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.71 (d, $J = 8.3$ Hz, 2H), 7.68-7.61 (m, 3H), 6.84 (d, $J = 15.6$ Hz, 1H), 3.89 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 143.44, 135.2, 133.0 (q, $J = 32.8$ Hz), 128.8, 126.2 (q, $J = 3.7$ Hz), 123.6 (q, $J = 273.6$ Hz), 123.0, 56.4; HRMS (EI) m/z [M]$^+$ calec for C$_{10}$H$_9$F$_3$O$_3$S$^+$ 266.0219, found 266.0219.

10: Methyl 2-(N-butylacetamido)-2-phenylethene-1-sulfonate

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.56-7.39 (m, 5H), 6.29 (s, 1H), 3.79 (s, 3H), 3.33 (t, $J = 7.6$ Hz, 2H), 2.25 (s, 3H), 1.53-1.41 (m, 2H), 1.26-1.17 (m, 2H), 0.86 (t, $J = 7.3$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.0, 154.0, 132.0, 131.6, 129.5, 128.7, 120.5, 55.9, 47.0, 30.3, 23.1, 19.97, 13.7; HRMS (ESI) m/z [M + Na]$^+$ calec for C$_{15}$H$_{21}$NNaO$_4$S$^+$ 334.1083, found 334.1090.

8. $^1$H and $^{13}$C NMR spectra

$^1$H NMR spectra of compound 3aa (400MHz, CDCl$_3$)
$^{13}$C NMR spectra of compound 3aa (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound **3ab** (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound **3ab** (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ac (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ac (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ad (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ad (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ae (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ae (100MHz, CDCl$_3$)
$^1\text{H}$ NMR spectra of compound 3af (400MHz, CDCl$_3$)

$^{13}\text{C}$ NMR spectra of compound 3af (100MHz, CDCl$_3$)

\[
\text{MeO} \quad \text{SO} \quad \text{OMe} \\
\text{F} \quad \text{MeO} \quad \text{SO} \quad \text{OMe}
\]
$^1$H NMR spectra of compound 3ag (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ag (100MHz, CDCl$_3$)

\[
\begin{align*}
\text{Cl} & \quad \text{MeO} \\
\text{SO} & \quad \text{OMe}
\end{align*}
\]
$^1$H NMR spectra of compound 3ah (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ah (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ai (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ai (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3aj (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3aj (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ak (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ak (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3al (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3al (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3am (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3am (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3an (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3an (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ao (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ao (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ap (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ap (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3aq (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3aq (100MHz, CDCl$_3$)

The diagrams show the NMR spectra of compound 3aq in CDCl$_3$ solvent. The chemical shifts are indicated on the right side of the spectra.
$^1$H NMR spectra of compound 3ar (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ar (100MHz, CDCl$_3$)
\( ^1 \text{H NMR spectra of compound \textbf{3as} (400MHz, CDCl}_3 \text{)} \)

\[
\text{[Diagram of NMR spectrum with peaks]} 
\]

\( ^{13} \text{C NMR spectra of compound \textbf{3as} (100MHz, CDCl}_3 \text{)} \)

\[
\text{[Diagram of NMR spectrum with peaks]} 
\]
$^1$H NMR spectra of compound 3at (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3at (100MHz, CDCl$_3$)
\( ^1H \) NMR spectra of compound 3au (400MHz, CDCl\(_3\))

\( ^{13}C \) NMR spectra of compound 3au (100MHz, CDCl\(_3\))
$^1$H NMR spectra of compound 3av (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3av (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3aw (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3aw (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ax (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ax (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ay (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ay (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3az (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3az (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3ba (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3ba (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3bb (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bb (100MHz, CDCl$_3$)
$^1\text{H NMR spectra of compound } \textbf{3bc} (400MHz, CDCl}_3$

$^{13}\text{C NMR spectra of compound } \textbf{3bc} (100MHz, CDCl}_3$
$^1$H NMR spectra of compound 3bd (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bd (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3be (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3be (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3bf (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bf (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3bg (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bg (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3bh (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bh (100MHz, CDCl$_3$)
$^{1}H$ NMR spectra of compound 3bi (400MHz, CDCl$_3$)

$^{13}C$ NMR spectra of compound 3bi (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 3bj (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 3bj (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5a (400 MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5a (100 MHz, CDCl$_3$)
\(^1\)H NMR spectra of compound \(5b\) (400MHz, CDCl\(_3\))

\[^{13}\text{C}\) NMR spectrum of compound \(5b\) (100 MHz, CDCl\(_3\))
$^1$H NMR spectra of compound 5c (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5c (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5d (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5d (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5e (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5e (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5f (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5f (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound $5g$ (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound $5g$ (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5h (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5h (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5i (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5i (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5j (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5j (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5k (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5k (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 51 (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 51 (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5m (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5m (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5n (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5n (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5o (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5o (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5p (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5p (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5qa (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5qa (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5qb (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5qb (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5ra (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5ra (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5rb (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5rb (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5s (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5s (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 5t (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5t (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound $5u$ (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound $5u$ (100 MHz, CDCl$_3$)
\(^1\)H NMR spectra of compound 5v (400MHz, CDCl\(_3\))

\(^{13}\)C NMR spectrum of compound 5v (100 MHz, CDCl\(_3\))
$^1$H NMR spectra of compound 5w (400MHz, CDCl$_3$)

$^{13}$C NMR spectrum of compound 5w (100 MHz, CDCl$_3$)
$^1$H NMR spectra of compound 7 (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 7 (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 8 (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 8 (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 9 (400MHz, CDCl$_3$)

$^{13}$C NMR spectra of compound 9 (100MHz, CDCl$_3$)
$^1$H NMR spectra of compound 10 (400MHz, CDCl$_3$)

\[
\begin{array}{c}
\text{N} \\
\text{O} \\
\text{OMe}
\end{array}
\]

$^{13}$C NMR spectra of compound 10 (100MHz, CDCl$_3$)

\[
\begin{array}{c}
\text{N} \\
\text{O} \\
\text{OMe}
\end{array}
\]
9. Crystallographic details

9.1 Crystallographic details of 3as

Table S2 Crystal data and structure refinement for 3as.

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<th>Parameter</th>
<th>Value</th>
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<td>Empirical formula</td>
<td>C_{16}H_{18}O_{4}S</td>
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<tr>
<td>Formula weight</td>
<td>306.36</td>
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<tr>
<td>Temperature/K</td>
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<tr>
<td>Crystal system</td>
<td>monoclinic</td>
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<tr>
<td>Space group</td>
<td>P2_1/n</td>
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<td>a/Å</td>
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<td>b/Å</td>
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<td>c/Å</td>
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<td>F(000)</td>
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<tr>
<td>Crystal size/mm³</td>
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</tr>
<tr>
<td>Radiation</td>
<td>MoKα (λ = 0.71073)</td>
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<tr>
<td>2θ range for data collection/°</td>
<td>4.398 to 56.632</td>
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9.2 Crystallographic details of 5m

Table S2 Crystal data and structure refinement for 5m.

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<th>Value</th>
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<td>Formula weight</td>
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<tr>
<td>Temperature/K</td>
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<tr>
<td>Crystal system</td>
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<tr>
<td>Space group</td>
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<td>a/Å</td>
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<tr>
<td>b/Å</td>
<td>8.3647(11)</td>
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<td>Volume/Å³</td>
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<td>ρ calc/g/cm³</td>
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<tr>
<td>μ/mm⁻¹</td>
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F(000) 608.0
Crystal size/mm$^3$ 0.260× 0.210 × 0.180
Radiation MoKα ($\lambda = 0.71073$)
2Θ range for data collection/° 5.332 to 49.994
Index ranges -13 ≤ h ≤ 9, -9 ≤ k ≤ 9, -18 ≤ l ≤ 18
Reflections collected 7236
Independent reflections 2589 [R$_{int} = 0.0208$, R$_{sigma} = 0.0238$]
Data/restraints/parameters 2589/0/176
Goodness-of-fit on F$^2$ 0.984
Final R indexes [I>=2σ (I)] R$_1 = 0.079$, wR$_2 = 0.1025$
Final R indexes [all data] R$_1 = 0.0523$, wR$_2 = 0.1099$
Largest diff. peak/hole / e Å$^{-3}$ 0.18/-0.28