

**Photoredox-catalyzed hydrosulfonylation reaction of
electron-deficient alkenes with substituted Hantzsch esters
and sulfur dioxide**

Xuefeng Wang, Min Yang, Wenlin Xie, Xiaona Fan,* and Jie Wu*

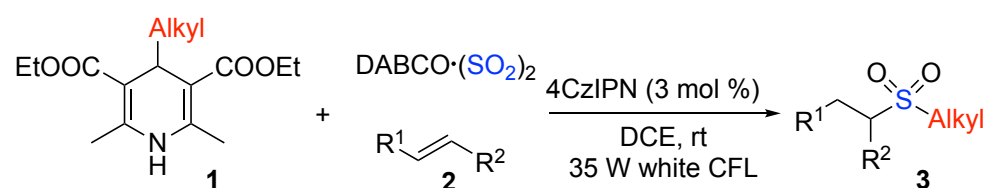
Supporting Information

1. General experimental methods (S2).
2. General experimental procedure and characterization data (S2-S10).
3. ^1H and ^{13}C NMR spectra of compounds **3-6** (S11-S30).

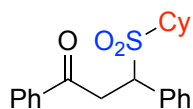
General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. 1,2-dichloroethane (DCE, extra dry, water < 50 ppm) is purchased from *Energy Chemicals* and used as received. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63µm, 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 254 nm ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 30–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million(ppm) from solvent residual peak on the δ scale. ^1H and ^{13}C NMR spectra were recorded in chloroform-*d* or DMSO-*d*₆ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a microTOF II Instrument.

General experimental procedure for the photoredox-catalyzed hydrosulfonation reaction of electron-deficient alkenes with substituted Hantzsch esters and sulfur dioxide

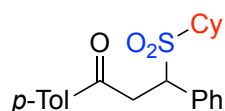


1,2-Dichloroethane (3.0 mL) was added to a sealed tube containing alkene **2** (0.2 mmol), DABCO.(SO₂)₂ (0.3 mmol, 1.5 equiv), substituted Hantzsch ester **1** (0.3 mmol, 1.5 equiv) and 4-CzIPN (3 mol %) under Ar atmosphere via a syringe. The mixture was then exposed to a 35w white CFL lamp at room temperature (25 °C) for 48 hours. After the conversion was completed as indicated by TLC, the solvent was evaporated under reduced pressure and the residue was purified directly by flash column chromatography to afford the corresponding product **3**.



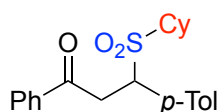
3-(Cyclohexylsulfonyl)-1,3-diphenylpropan-1-one (**3a**)¹

White solid, 56.6 mg (80% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.4 Hz, 2H), 7.57 – 7.55 (m, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.32 (m, 3H), 5.05 (dd, *J* = 9.7, 3.3 Hz, 1H), 4.05 (dd, *J* = 17.9, 3.3 Hz, 1H), 3.83 (dd, *J* = 17.9, 9.7 Hz, 1H), 2.59 (tt, *J* = 12.1, 3.3 Hz, 1H), 2.17 (d, *J* = 12.7 Hz, 1H), 2.01 (d, *J* = 12.9 Hz, 1H), 1.84 (d, *J* = 12.8 Hz, 2H), 1.65 – 1.46 (m, 3H), 1.21 – 1.06 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.31, 136.29, 133.75, 133.67, 129.59, 129.16, 129.10, 128.83, 128.26, 60.24, 58.12, 37.33, 26.55, 25.14, 24.90, 23.26.



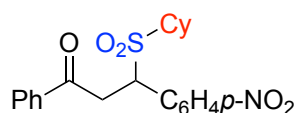
3-(Cyclohexylsulfonyl)-3-phenyl-1-(p-tolyl)propan-1-one (**3b**)¹

White solid, 54.5 mg (74% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.55 (d, *J* = 6.7 Hz, 2H), 7.40 – 7.32 (m, 3H), 7.23 (d, *J* = 8.1 Hz, 2H), 5.04 (dd, *J* = 9.7, 3.1 Hz, 1H), 4.01 (dd, *J* = 17.8, 3.2 Hz, 1H), 3.81 (dd, *J* = 17.8, 9.7 Hz, 1H), 2.59 (tt, *J* = 12.1, 3.3 Hz, 1H), 2.39 (s, 3H), 2.17 (d, *J* = 12.7 Hz, 1H), 2.01 (d, *J* = 12.7 Hz, 1H), 1.84 (d, *J* = 10.3 Hz, 2H), 1.64 – 1.50 (m, 3H), 1.21 – 1.06 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.90, 144.68, 133.86, 133.72, 129.61, 129.50, 129.13, 129.05, 128.39, 60.29, 58.12, 37.16, 26.53, 25.14, 24.90, 23.28, 21.79.



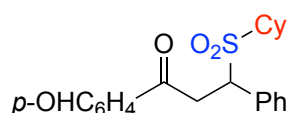
3-(Cyclohexylsulfonyl)-1-phenyl-3-(p-tolyl)propan-1-one (**3c**)¹

White solid, 56.0 mg (76% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 7.7 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.45 – 7.42 (m, 4H), 7.17 (d, *J* = 7.8 Hz, 2H), 5.01 (dd, *J* = 9.7, 3.0 Hz, 1H), 4.02 (dd, *J* = 17.9, 3.0 Hz, 1H), 3.81 (dd, *J* = 17.9, 9.7 Hz, 1H), 2.61 (t, *J* = 12.1 Hz, 1H), 2.32 (s, 3H), 2.17 (d, *J* = 12.6 Hz, 1H), 2.02 (d, *J* = 12.6 Hz, 1H), 1.84 (d, *J* = 10.5 Hz, 2H), 1.62 – 1.47 (m, 3H), 1.20 – 1.08 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.46, 139.05, 136.38, 133.73, 130.54, 129.90, 129.42, 128.84, 128.30, 59.96, 57.90, 37.27, 29.84, 26.59, 25.18, 24.90, 23.22, 21.35.



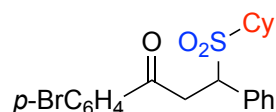
3-(Cyclohexylsulfonyl)-3-(4-nitrophenyl)-1-phenylpropan-1-one (**3d**)¹

Yellow solid, 34.2 mg (43% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 8.24 (d, *J* = 8.8 Hz, 2H), 7.94 – 7.92 (m, 2H), 7.78 (d, *J* = 8.8 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 5.13 (dd, *J* = 10.0, 3.1 Hz, 1H), 4.11 (dd, *J* = 18.2, 3.1 Hz, 1H), 3.84 (dd, *J* = 18.2, 10.0 Hz, 1H), 2.58 (tt, *J* = 12.0, 3.4 Hz, 1H), 2.14 (d, *J* = 12.9 Hz, 1H), 2.06 (d, *J* = 12.8 Hz, 1H), 1.90 – 1.87 (m, 2H), 1.69 – 1.69 (m, 1H), 1.64 – 1.51 (m, 2H), 1.23 – 1.09 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.82, 166.55, 141.08, 134.21, 130.78, 129.03, 128.29, 124.40, 124.26, 59.62, 59.07, 37.71, 26.46, 25.07, 24.91, 23.51.



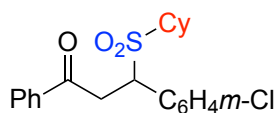
3-(Cyclohexylsulfonyl)-1-(4-hydroxyphenyl)-3-phenylpropan-1-one (**3e**)¹

White solid, 47.5 mg (64% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.43 (s, 1H), 7.87 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 7.6 Hz, 2H), 7.38 – 7.32 (m, 3H), 6.83 (d, *J* = 8.8 Hz, 2H), 4.98 (dd, *J* = 8.9, 4.4 Hz, 1H), 3.87 (dd, *J* = 17.7, 9.0 Hz, 1H), 3.80 (dd, *J* = 17.7, 4.4 Hz, 1H), 2.95 (tt, *J* = 11.8, 3.2 Hz, 1H), 2.11 (d, *J* = 12.5 Hz, 1H), 1.81 – 1.70 (m, 3H), 1.57 (d, *J* = 6.7 Hz, 1H), 1.36 – 1.28 (m, 2H), 1.19 – 1.11 (m, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 193.44, 162.46, 133.25, 130.81, 129.90, 128.54, 128.44, 127.67, 115.28, 60.15, 57.19, 36.30, 24.96, 24.72, 24.44, 24.38.



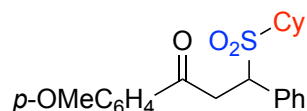
1-(4-Bromophenyl)-3-(cyclohexylsulfonyl)-3-phenylpropan-1-one (**3f**)¹

White solid, 43.3 mg (50% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 8.6 Hz, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.41 – 7.33 (m, 3H), 5.01 (dd, *J* = 9.6, 3.3 Hz, 1H), 4.02 (dd, *J* = 17.9, 3.4 Hz, 1H), 3.75 (dd, *J* = 17.9, 9.6 Hz, 1H), 2.58 (tt, *J* = 12.1, 3.4 Hz, 1H), 2.15 (d, *J* = 12.9 Hz, 1H), 2.00 (d, *J* = 12.7 Hz, 1H), 1.85 (d, *J* = 12.8 Hz, 2H), 1.65 – 1.58 (m, 2H), 1.53 – 1.47 (m, 1H), 1.21 – 1.04 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.46, 135.04, 133.55, 132.21, 129.79, 129.55, 129.25, 129.09, 60.23, 58.17, 37.31, 26.60, 25.15, 24.91, 23.24.



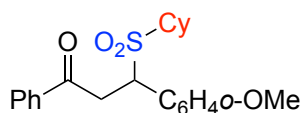
3-(3-Chlorophenyl)-3-(cyclohexylsulfonyl)-1-phenylpropan-1-one (**3g**)¹

White solid, 33.5 mg (43% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.4 Hz, 2H), 7.57 (d, *J* = 5.3 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 3H), 7.33 – 7.31 (m, 2H), 5.01 (dd, *J* = 9.7, 3.2 Hz, 1H), 4.05 (dd, *J* = 18.1, 3.2 Hz, 1H), 3.78 (dd, *J* = 18.1, 9.7 Hz, 1H), 2.62 (tt, *J* = 12.1, 3.4 Hz, 1H), 2.15 (d, *J* = 12.6 Hz, 1H), 2.05 (d, *J* = 12.1 Hz, 1H), 1.87 (d, *J* = 11.7 Hz, 2H), 1.67 – 1.58 (m, 2H), 1.55 – 1.49 (m, 1H), 1.22 – 1.10 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.02, 136.09, 135.77, 135.06, 133.93, 130.34, 129.57, 129.40, 128.90, 128.29, 127.97, 59.60, 58.43, 37.44, 29.83, 26.51, 25.10, 24.89, 23.36.



3-(Cyclohexylsulfonyl)-1-(4-methoxyphenyl)-3-phenylpropan-1-one (**3h**)¹

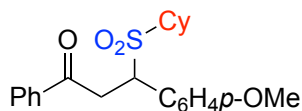
White solid, 53.9 mg (70% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.5 Hz, 2H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.38 – 7.30 (m, 3H), 6.90 (d, *J* = 8.5 Hz, 2H), 5.04 (dd, *J* = 9.6, 2.7 Hz, 1H), 3.98 (dd, *J* = 17.8, 2.8 Hz, 1H), 3.84 (s, 3H), 3.78 (dd, *J* = 17.7, 9.7 Hz, 1H), 2.59 (t, *J* = 12.0 Hz, 1H), 2.16 (d, *J* = 12.5 Hz, 1H), 2.01 (d, *J* = 12.5 Hz, 1H), 1.84 (d, *J* = 12.0 Hz, 2H), 1.66 (d, *J* = 16.7 Hz, 1H), 1.58 – 1.47 (m, 2H), 1.19 – 1.05 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 193.70, 163.98, 133.74, 130.57, 129.58, 129.37, 129.10, 129.01, 113.94, 60.32, 58.08, 55.61, 36.85, 26.49, 25.12, 24.88, 23.26.



3-(Cyclohexylsulfonyl)-3-(2-methoxyphenyl)-1-phenylpropan-1-one (**3i**)¹

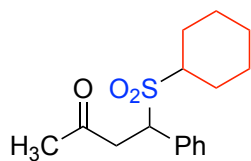
Colorless oil, 38.3 mg (50% yield). ¹H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.94 (m, 2H), 7.58 – 7.51 (m, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.31 (ddd, *J* = 8.3, 7.6, 1.7 Hz, 1H), 7.00 – 6.92 (m, 2H), 5.70 (dd, *J* = 9.4, 4.2 Hz, 1H), 4.12 (dd, *J* = 17.8, 4.2 Hz, 1H), 3.94 (s, 3H), 3.75 (dd, *J* = 17.8, 9.4 Hz, 1H), 2.62 (tt, *J* = 12.0, 3.3 Hz, 1H), 2.38 (d, *J* = 12.8 Hz, 1H), 1.97 (d, *J* = 12.9 Hz, 1H), 1.91 – 1.82 (m, 2H), 1.65 (d, *J* = 10.2 Hz, 1H), 1.55 –

1.43 (m, 2H), 1.22 – 1.09 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.60, 157.47, 133.58, 130.15, 129.63, 128.80, 128.31, 122.20, 121.49, 111.08, 58.77, 56.03, 52.30, 37.12, 26.70, 25.44, 25.32, 23.62.



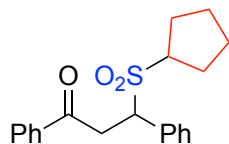
3-(Cyclohexylsulfonyl)-3-(4-methoxyphenyl)-1-phenylpropan-1-one (**3j**)¹

White solid, 53.8 mg (70% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.92 (m, 2H), 7.58 – 7.54 (m, 1H), 7.47 – 7.42 (m, 4H), 6.91 – 6.87 (m, 2H), 4.99 (dd, J = 9.8, 3.2 Hz, 1H), 4.01 (dd, J = 17.8, 3.2 Hz, 1H), 3.82 – 3.75 (m, 4H), 2.61 (tt, J = 12.1, 3.4 Hz, 1H), 2.15 (d, J = 14.2 Hz, 1H), 2.02 (d, J = 12.7 Hz, 1H), 1.85 (d, J = 12.6 Hz, 2H), 1.65 – 1.47 (m, 3H), 1.21 – 1.09 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.49, 160.09, 136.37, 133.74, 130.74, 128.84, 128.29, 125.37, 114.60, 59.63, 57.85, 55.40, 37.30, 29.84, 26.57, 25.18, 24.93, 23.22.



4-(Cyclohexylsulfonyl)-4-phenylbutan-2-one (**3k**)¹

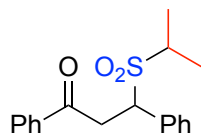
Colorless oil, 35.1 mg (60% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.45 (m, 2H), 7.39 – 7.35 (m, 3H), 4.82 (dd, J = 9.1, 3.9 Hz, 1H), 3.53 (dd, J = 17.9, 3.9 Hz, 1H), 3.17 (dd, J = 17.9, 9.1 Hz, 1H), 2.53 (tt, J = 12.1, 3.4 Hz, 1H), 2.14 (s, 3H), 2.10 (d, 1H), 1.96 (d, J = 11.0 Hz, 1H), 1.82 (d, J = 13.0 Hz, 2H), 1.61 (d, J = 11.3 Hz, 1H), 1.54 – 1.45 (m, 2H), 1.18 – 1.02 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 203.69, 133.56, 129.46, 129.18, 129.14, 59.86, 58.07, 41.70, 30.61, 26.50, 25.10, 24.85, 23.20.



3-(Cyclopentylsulfonyl)-1,3-diphenylpropan-1-one (**3l**)¹

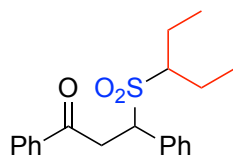
White solid, 53.4 mg (81% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.93 (m, 2H), 7.56 – 7.54 (m, 3H), 7.46 (t, J = 7.7 Hz, 2H), 7.37 – 7.33 (m, 3H), 4.98 (dd, J = 9.6, 3.3 Hz, 1H), 4.10 (dd, J = 17.9, 3.4 Hz, 1H), 3.84 (dd, J = 17.9, 9.6 Hz, 1H), 3.08 (tt, J =

8.8, 7.0 Hz, 1H), 2.17 – 1.91 (m, 3H), 1.83 – 1.70 (m, 3H), 1.61 – 1.50 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.29, 133.76, 133.71, 129.69, 129.15, 128.85, 128.76, 128.64, 128.28, 62.20, 58.62, 37.23, 28.08, 26.21, 26.16, 25.69.



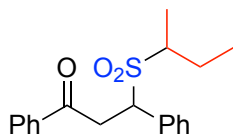
3-(Isopropylsulfonyl)-1,3-diphenylpropan-1-one (**3m**)¹

White solid, 48.6 mg (77% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, J = 7.4 Hz, 2H), 7.57 (t, J = 7.3 Hz, 3H), 7.45 (t, J = 7.7 Hz, 2H), 7.39 – 7.33 (m, 3H), 5.07 (dd, J = 9.7, 3.2 Hz, 1H), 4.07 (dd, J = 17.9, 3.2 Hz, 1H), 3.83 (dd, J = 17.9, 9.7 Hz, 1H), 2.87 (hept, J = 6.8 Hz, 1H), 1.37 (d, J = 6.7 Hz, 3H), 1.27 (d, J = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.29, 133.80, 129.64, 129.21, 129.15, 128.87, 128.78, 128.60, 128.30, 60.43, 50.25, 37.52, 16.72, 13.81.



3-(Pentan-3-ylsulfonyl)-1,3-diphenylpropan-1-one (**3n**)¹

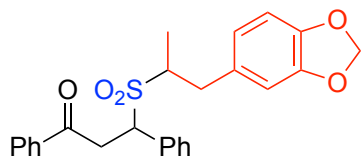
Colorless oil, 54.6 mg (80% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.92 (m, 2H), 7.56 – 7.53 (m, 3H), 7.44 (t, J = 7.7 Hz, 2H), 7.39 – 7.33 (m, 3H), 5.08 (dd, J = 9.5, 3.4 Hz, 1H), 4.06 (dd, J = 17.9, 3.4 Hz, 1H), 3.81 (dd, J = 17.9, 9.5 Hz, 1H), 2.53 – 2.48 (m, 1H), 1.96 – 1.88 (brs, 1H), 1.81 – 1.70 (brs, 3H), 0.96 (td, J = 7.5, 3.7 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.36, 136.31, 133.88, 133.70, 129.62, 129.10, 129.07, 128.81, 128.25, 61.46, 61.18, 37.47, 20.96, 18.77, 11.12, 10.78.



3-(*sec*-Butylsulfonyl)-1,3-diphenylpropan-1-one (**3o**)¹ 1:1 *dr*.

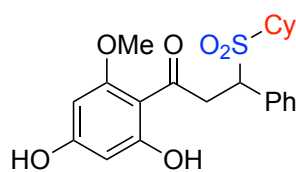
White solid, 46.0 mg (70% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.92 (m, 2H), 7.58 – 7.54 (m, 3H), 7.44 (t, J = 7.8 Hz, 2H), 7.39 – 7.33 (m, 3H), 5.09 (ddd, J = 16.8, 9.6, 3.3 Hz, 1H), 4.06 (dd, J = 17.9, 3.3 Hz, 1H), 3.83 (ddd, J = 17.9, 10.6, 9.7 Hz, 1H), 2.67 – 2.57 (brs, 1H), 2.09 – 1.94 (brs, 1H), 1.54 (ddq, J = 14.7, 9.8, 7.4 Hz, 1H),

1.34 (d, $J = 6.8$ Hz, 1.5H), 1.25 (d, $J = 7.0$ Hz, 1.5H), 0.92 (td, $J = 7.5, 1.9$ Hz, 3H). ^{13}C NMR (101 MHz, Chloroform- d) δ 195.29, 136.30, 133.74, 129.66, 129.60, 129.16, 129.11, 128.83, 128.26, 60.62, 56.30, 56.01, 37.56, 37.44, 23.46, 20.74, 13.48, 11.05, 10.87, 10.50.



3-((1-(Benzo[d][1,3]dioxol-5-yl)propan-2-yl)sulfonyl)-1,3-diphenylpropan-1-one (**3p**)

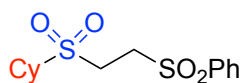
1:1 mixture of diastereoisomers. White solid, 60.8 mg (70% yield). ^1H NMR (400 MHz, Chloroform- d) δ 7.96 – 7.93 (m, 2H), 7.63 – 7.55 (m, 3H), 7.47 – 7.34 (m, 5H), 6.69 (t, $J = 7.6$ Hz, 1H), 6.52 – 6.41 (m, 1.5H), 6.39 (d, $J = 1.6$ Hz, 0.5H), 5.93 – 5.90 (m, 2H), 5.11 (ddd, $J = 13.0, 9.6, 3.2$ Hz, 1H), 4.13 – 4.06 (m, 1H), 3.85 (ddd, $J = 17.9, 9.6, 7.1$ Hz, 1H), 3.33 (dd, $J = 13.6, 3.5$ Hz, 0.5H), 3.25 (dd, $J = 13.5, 3.3$ Hz, 0.5H), 2.94 – 2.84 (brs, 1H), 2.53 (ddd, $J = 13.6, 11.3, 5.9$ Hz, 1H), 1.25 (d, $J = 6.9$ Hz, 1.5H), 1.15 (d, $J = 6.9$ Hz, 1.5H). ^{13}C NMR (101 MHz, Chloroform- d) δ 195.18, 148.02, 147.96, 146.76, 146.66, 136.30, 133.80, 133.63, 133.48, 130.54, 130.27, 129.79, 129.64, 129.40, 129.29, 128.87, 128.30, 122.60, 122.36, 109.49, 109.27, 108.57, 108.48, 101.18, 101.14, 61.30, 61.17, 56.57, 56.50, 37.78, 37.61, 36.11, 33.26, 14.33, 13.34, 10.66. HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{25}\text{O}_5\text{S}^+$: 437.1417 ($\text{M} + \text{H}^+$), found: 437.1416.



3-(Cyclohexylsulfonyl)-1-(2,4-dihydroxy-6-methoxyphenyl)-3-phenylpropan-1-one (**3q**)

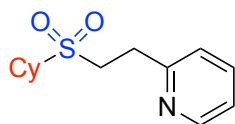
White solid, 57.5mg (69% yield). ^1H NMR (400 MHz, DMSO- d_6) δ 13.05 (s, 1H), 10.73 (s, 1H), 7.50 – 7.45 (m, 2H), 7.39 – 7.34 (m, 3H), 6.00 (s, 1H), 5.84 (s, 1H), 4.96 (dd, $J = 9.6, 3.9$ Hz, 1H), 3.92 – 3.84 (m, 4H), 3.75 (dd, $J = 17.6, 9.7$ Hz, 1H), 2.80 (tt, $J = 11.9, 3.1$ Hz, 1H), 2.13 (d, $J = 11.8$ Hz, 1H), 1.77 – 1.70 (m, 3H), 1.56 (s, 1H), 1.38 – 1.26 (m, 2H), 1.17 – 1.11 (m, 3H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 199.14, 165.65, 165.27, 162.90, 133.31, 129.74, 128.51, 104.48, 95.70, 91.59, 59.95, 57.28, 56.05, 41.69,

25.16, 24.70, 24.40, 24.16. HRMS (ESI) calcd for $C_{22}H_{27}O_6S^+$: 419.1523 ($M + H^+$), found: 419.1503.



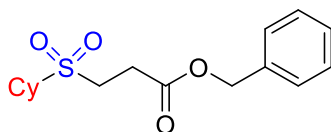
((2-(Cyclohexylsulfonyl)ethyl)sulfonyl)benzene (4)¹

White solid, 26.5 mg (42% yield). 1H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.90 (m, 2H), 7.72 (t, J = 7.5 Hz, 1H), 7.62 (t, J = 7.7 Hz, 2H), 3.55 – 3.51 (m, 2H), 3.35 – 3.31 (m, 2H), 2.90 (tt, J = 12.2, 3.4 Hz, 1H), 2.16 (d, J = 11.4 Hz, 2H), 1.95 (d, J = 13.2 Hz, 2H), 1.75 (d, J = 11.5 Hz, 1H), 1.54 (qd, J = 12.4, 3.1 Hz, 2H), 1.36 – 1.19 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.44, 134.68, 129.84, 128.17, 62.50, 48.47, 42.48, 25.23, 25.11, 25.05.



2-(2-(Cyclohexylsulfonyl)ethyl)pyridine (5)

Yellow oil, 15.0 mg (30% yield). 1H NMR (400 MHz, Chloroform-*d*) δ 8.53 (s, 1H), 7.64 (t, J = 7.2 Hz, 1H), 7.26 – 7.18 (m, 2H), 3.47 – 3.43 (m, 2H), 3.34 – 3.30 (m, 2H), 2.75 (tt, J = 12.2, 3.3 Hz, 1H), 2.18 (d, J = 12.1 Hz, 2H), 1.91 (d, J = 7.3 Hz, 2H), 1.71 (d, J = 5.8 Hz, 1H), 1.59 – 1.51 (m, 2H), 1.28 – 1.18 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.79, 149.55, 136.89, 123.64, 122.15, 61.52, 48.42, 29.65, 25.26, 25.21, 25.16. HRMS (ESI) calcd for $C_{13}H_{20}NO_2S^+$: 254.1209 ($M + H^+$), found: 254.1263.



Benzyl 3-(cyclohexylsulfonyl)propanoate (6)¹

Colorless oil, 26.6mg (43% yield). 1H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.35 (m, 5H), 5.16 (s, 2H), 3.25 (t, J = 7.6 Hz, 2H), 2.92 (t, J = 7.6 Hz, 2H), 2.84 (t, J = 12.1 Hz, 1H), 2.17 (d, J = 12.8 Hz, 2H), 1.93 (d, J = 12.2 Hz, 2H), 1.73 (d, J = 9.7 Hz, 1H), 1.58 – 1.48 (m, 2H), 1.32 – 1.21 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.64, 135.44, 128.79, 128.65, 128.49, 67.35, 61.72, 44.75, 26.52, 25.20.

Reference:

1. Liu, T.; Li, Y.; Lai, L.; Cheng, J.; Sun, J.; Wu, J. *Org. Lett.* **2018**, *20*, 3605.

