Supporting information

Visible light-induced cyclization of electron-enriched phenyl benzyl sulfides: synthesis of tetrahydrofurans and tetrahydropyrans

Wei Li,\textsuperscript{a,b} Chao Yang,\textsuperscript{a} Guo-Lin Gao\textsuperscript{a} and Wujiong Xia\textsuperscript{*a}

\textsuperscript{a} State Key Lab of Urban Water Resource and Environment, Shenzhen Graduate School, Harbin Institute of Technology, Harbin, 150080, China

\textsuperscript{b} Key Laboratory for Food Science and Engineering, Harbin University of Commerce, Harbin, 150076, China

E-mail: xiawj@hit.edu.cn (W. X.)

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1. General information

All reagents were purchased from commercial sources unless otherwise noted. Solvent was freshly distilled prior to use unless otherwise noted. Reactions were monitored by thin-layer chromatography (TLC) and visualized by UV-light (254 nm) or by treatment with a solution of 10 g phosphomolybdic acid and 100 mL EtOH followed by heating. $^1$H NMR (400/600 MHz) and $^{13}$C NMR (100/150 MHz) spectra were obtained on Bruker AV-400/600 instrument. Chemical shifts for $^1$NMR spectra were reported in δ ppm referenced to an internal SiMe₄ standard. Chemical shifts for $^{13}$CNMR spectra were reported in parts per million relative to the center line signal of the CDCl₃ triplet at 77.0 ppm. GC-MS spectra were recorded on Agilent 5973 mass spectrometer. HR-ESI-MS spectra were recorded on Agilent LC-QTOF mass spectrometer using electrospray ionization.

2. Measurement of oxidation potential

Substrate 1a of oxidation potential was measured in MeCN with 0.1 M NH₄PF₆ and saturated calomel electrode (SCE) as the reference electrode at room temperature. The result indicates the first oxidation potential of 1a is 0.72V.

3. Proposed reaction mechanism in oxidative quenching pathway
4. Preparation and characterization of phenyl benzyl sulfides

(1) Synthetic procedure of 1a-1l

(a) NaBH4 (1.5equiv), CH3OH, 0 °C; (b) PBr3 (1.0 equiv), CH2Cl2, 0 °C; (c) K2CO3 (1.5equiv), PhSH (1.1equiv); (d) LiBu" (1.2 equiv), TMEDA (1.2 equiv), THF, 0 °C

(3-bromopropoxy)(tert-butyl)dimethylsilane (1.1equiv) or
(4-chlorobutoxy)(tert-butyl)(dimethylsilane(1.1equiv)

Synthesis procedure for 1a
To solution of n-butyllithium (1.6 M in hexane, 3.8 mL, 6.0mmol) in THF (10 mL) was added N,N,N',N'-tetramethylenediamine (TMEDA, 0.70 g, 6.0mmol) at 30 °C. After stirring for 0.5 h, dropwise introduction of (4-methoxybenzyl)(phenyl)sulfane (1.150 g, 5.0 mmol) in THF (5 mL) and kept stirring for 2 h. Then (3-bromopropoxy)(tert-butyl)dimethylsilane (1.25 g, 5.0 mmol) was added. the reaction mixture was allowed to warm to 0°C and stirred for 5 h. The resultant solution was quenched by NH4Cl solution (20 mL) and extracted with ether (3 × 30 mL). The combined organic layers were washed with aqueous brine (60 mL), dried over MgSO4 and filtered. The solvent was removed in vacuo to afford the crude product, following purified by flash chromatography on silica gel to give 1a (72% yield).

tert-butyl(4-(4-methoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1a)
Colorless liquid (72%). 1H NMR (400 MHz, CDCl3) δ 7.29 – 7.11 (m, 7H), 6.79 (d, J = 7.8 Hz, 2H), 4.11 (t, J = 6.2 Hz, 1H), 3.77 (s, 3H), 3.55 (t, J = 6.2 Hz, 2H), 2.12 – 1.81 (m, 2H), 1.58 – 1.37 (m, 2H), 0.86 (s, 9H), -0.00 (s, 6H). 13C NMR (150 MHz, CDCl3) δ 158.51, 135.12, 133.89, 132.39, 128.82, 128.59, 126.90, 113.65, 62.60, 55.19, 52.67, 32.61, 30.68, 25.93, 18.28, -5.35. GC–MS (EI, QMS, m/z) 402 (<1%), 345, 293, 235, 161 (100%), 73.

tert-butyl(4-(2-methoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1b)
Colorless liquid (61%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.09 (m, 7H), 6.95 –
6.74 (m, 2H), 4.76 (t, $J$ = 7.4 Hz, 1H), 3.90 – 3.67 (m, 3H), 3.56 (t, $J$ = 6.1 Hz, 2H),
2.13 – 1.90 (m, 2H), 1.63 – 1.48 (m, 2H), 0.86 (s, 9H), -0.00 (s, 6H). $^{13}$C NMR (150
MHz, CDCl$_3$) δ 156.73, 135.64, 131.79, 130.14, 128.44, 127.92, 127.85, 126.50,
126.50, 120.64, 110.48, 62.64, 55.45, 44.59, 31.77, 30.60, 25.92, 18.29, -5.36. GC–MS (EI,
QMS, m/z) 402 (<1%), 345, 293, 235, 161 (100%), 73.

**tert-butyldimethyl(4-(phenylthio)-4-p-tolylbutoxy)silane (1c)**
Colorless liquid (78%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.29 – 7.02 (m, 9H), 4.12 (t, $J$
= 7.4 Hz, 1H), 3.55 (t, $J$ = 6.2 Hz, 2H), 2.30 (s, 3H), 2.09 – 1.88 (m, 2H), 1.57 – 1.45
(m, 2H), 0.86 (s, 9H), 0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 138.82, 136.62,
135.26, 132.18, 129.01, 128.59, 126.82, 62.60, 52.92, 32.70, 32.59, 30.67,
25.96, 25.93, 21.07, 18.28, -5.36. GC–MS (EI, QMS, m/z) 386 (<1 %), 277, 219, 145
(100%), 73.

**tert-butyl(4-(2,5-dimethoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1e)**
Colorless liquid (66%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.30 – 7.16 (m, 9H), 4.15 (dd,
$J$ = 9.2, 5.9 Hz, 1H), 3.56 (t, $J$ = 6.3 Hz, 2H), 2.12 – 1.91 (m, 2H), 1.59 – 1.48 (m,
2H), 1.30 (s, 9H), 0.87 (s, 9H), 0.00 (d, $J$ = 4.3 Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$)
δ 149.89, 138.61, 135.38, 132.15, 128.59, 127.37, 126.80, 125.23, 62.65, 52.81,
34.42, 32.54, 31.33, 30.68, 25.94, 18.29, -5.35. GC–MS (EI, QMS, m/z) 432 (<1 %), 319,
261, 187, 131, 73, 57 (100%).
tert-butyl(4-(3,4-dimethoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1f)
Colorless liquid (51%). $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.26 – 7.16 (m, 5H), 6.73 (s, 3H), 4.09 (t, $J = 7.1$ Hz, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.56 (t, $J = 5.3$ Hz, 2H), 2.07 – 1.85 (m, 2H), 1.58 – 1.43 (m, 2H), 0.85 (s, 9H), 0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 148.71, 147.91, 134.94, 134.39, 132.64, 128.58, 127.02, 119.99, 110.64, 110.58, 62.59, 55.78, 55.73, 53.23, 32.59, 30.71, 25.91, 18.27, -5.36. GC–MS (EI, QMS, m/z) 432 (<1%), 323, 265, 191(100%), 160, 73.

tert-butyldimethyl(4-(phenylthio)-4-(3,4,5-trimethoxyphenyl)butoxy)silane (1g)
Colorless liquid (46%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.22 (d, $J = 22.0$ Hz, 5H), 6.39 (s, 2H), 4.03 (t, $J = 7.6$ Hz, 1H), 3.80 (s, 3H), 3.76 (s, 6H), 3.57 (t, $J = 5.8$ Hz, 2H), 2.09 – 1.88 (m, 2H), 1.56 – 1.51 (m, 2H), 0.85 (s, 9H), -0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 152.95, 137.59, 136.84, 134.77, 132.92, 128.66, 127.24, 104.62, 62.64, 60.84, 56.01, 53.96, 32.57, 30.74, 25.93, -0.02, -5.33. GC–MS (EI, QMS, m/z) 462 (<1%), 353, 295, 221 (100%), 190, 73.

(4-(benzo[d][1,3]dioxol-5-yl)-4-(phenylthio)butoxy)(tert-butyl)dimethylsilane (1h)
Colorless liquid (61%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.29 – 7.16 (m, 5H), 6.80 (s, 1H), 6.71 – 6.58 (m, 2H), 5.91 (s, 2H), 4.06 (dd, $J = 14.9$, 8.0 Hz, 1H), 3.55 (t, $J = 6.1$ Hz, 2H), 2.07 – 1.83 (m, 2H), 1.59 – 1.44 (m, 2H), 0.86 (s, 9H), -0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 147.70, 146.49, 135.85, 135.00, 132.34, 128.63, 126.98, 121.28, 107.82, 107.76, 100.93, 62.56, 53.22, 32.75, 30.64, 25.92, 18.28, -5.35. GC–MS (EI, QMS, m/z) 416 (<1%), 307, 249, 175 (100%), 117.
tert-butyl(4-(5-chloro-2-methoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1i)
Colorless liquid (36%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.26 – 7.10 (m, 7H), 6.72 (d, $J$ = 8.7 Hz, 1H), 4.69 (dd, $J$ = 8.8, 6.5 Hz, 1H), 3.73 (s, 3H), 3.56 (t, $J$ = 6.3 Hz, 2H), 2.02 – 1.86 (m, 2H), 1.61 – 1.51 (m, 2H), 0.86 (s, 9H), 0.01 (d, $J$ = 2.4 Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 155.34, 134.91, 132.29, 132.16, 128.53, 127.95, 127.54, 126.88, 125.66, 111.71, 62.51, 55.76, 44.38, 31.63, 30.54, 25.93, 18.30, -5.35. GC–MS (EI, QMS, m/z) 436 (<1%), 327, 269, 197, 195 (100%), 160, 73.

tert-butyl(4-(5-tert-butyl-2-methoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1j)
Colorless liquid (61%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.28 – 7.15 (m, 7H), 6.75 (d, $J$ = 8.5 Hz, 1H), 4.70 (t, $J$ = 7.6 Hz, 1H), 3.77 (s, 3H), 3.57 (t, $J$ = 6.3 Hz, 2H), 2.07 – 1.92 (m, 2H), 1.62 – 1.51 (m, 2H), 1.23 (s, 9H), 0.86 (s, 9H), 0.00 (d, $J$ = 3.1 Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 154.48, 142.90, 135.24, 132.63, 129.17, 128.40, 126.72, 125.66, 124.27, 109.98, 62.73, 55.58, 45.27, 34.06, 31.66, 31.43, 30.69, 25.94, 18.30, -5.34. GC–MS (EI, QMS, m/z) 458 (<1%), 341, 291, 197, 161, 73, 57 (100%).

tert-butyl(4-(4-methoxy-3,5-dimethylphenyl)-4-(phenylthio)butoxy)dimethylsilane (1k)
Colorless liquid (41%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.29 (d, $J$ = 7.2 Hz, 2H), 7.25 – 7.17 (m, 3H), 6.87 (s, 2H), 4.04 (dd, $J$ = 9.4, 5.7 Hz, 1H), 3.69 (s, 3H), 3.55 (t, $J$ = 6.3 Hz, 2H), 2.23 (s, 6H), 2.03 – 1.90 (m, 2H), 1.54 – 1.43 (m, 2H), 0.86 (s, 9H), -0.00 (d, $J$ = 4.8 Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 155.90, 136.70, 135.33, 132.24, 130.57, 128.60, 128.07, 126.88, 62.64, 59.63, 52.69, 32.55, 30.70, 25.92, 18.28, 16.11, -5.35. GC–MS (EI, QMS, m/z) 430 (<1%), 321, 263, 189 (100%), 174, 73.
tert-butyl(4-(3,5-di-tert-butyl-2-methoxyphenyl)-4-(phenylthio)butoxy)dimethylsilane (1l)
Colorless liquid (69%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.42 – 7.15 (m, 7H), 4.64 (t, J = 5.5 Hz, 1H), 3.79 (s, 3H), 3.56 (s, 2H), 2.16 – 1.89 (m, 2H), 1.59 – 1.44 (m, 2H), 1.39 (d, J = 22.3 Hz, 9H), 1.29 (s, 9H), 0.86 (s, 9H), -0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 155.10, 145.81, 141.40, 135.48, 134.35, 131.89, 128.66, 126.71, 123.43, 122.89, 62.48, 62.45, 45.15, 35.30, 34.56, 33.33, 31.47, 31.13, 30.71, 25.92, 18.27, -5.34, -5.35. GC−MS (EI, QMS, m/z) 514(<1%), 347, 273, 217, 161, 73, 57.

4-(4-methoxyphenyl)-4-(phenylthio)butan-1-ol (1m)
Preparation from 1a by deprotection of TBS. Colorless liquid (89%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.26 – 7.11 (m, 6H), 6.80 (d, J = 8.3 Hz, 2H), 4.12 (dd, J = 8.9, 6.0 Hz, 1H), 3.78 (s, 3H), 3.60 (t, J = 6.4 Hz, 2H), 2.07 – 1.92 (m, 2H), 1.63 – 1.51 (m, 2H), 1.26 (s, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 158.56, 134.96, 133.78, 132.36, 128.77, 128.62, 126.98, 113.70, 62.45, 55.19, 52.69, 32.59, 30.69. HRMS (ESI) exact mass calculated for [M+Na$^+$] (C$_{17}$H$_{20}$NaO$_2$S) requires m/z 311.1082, found m/z 311.1078.

(2) Synthetic procedure of 1n-1p

(a) HCl (1:1), CH$_3$OH; (b) PCC (1.5 equiv), CH$_2$Cl$_2$; (c) CH$_3$MgBr or PhMgBr (1.2 equiv), THF
5-(4-methoxyphenyl)-5-(phenylthio)pentan-2-ol (1n)
Colorless liquid (70%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.30 – 7.16 (m, 7H), 6.83 (d, $J$ = 8.0 Hz, 2H), 4.15 (t, $J$ = 7.5 Hz, 1H), 3.81 (s, 3H), 3.80 – 3.75 (m, 1H), 2.17 – 1.91 (m, 2H), 1.65 – 1.37 (m, 2H), 1.20 (s, 1H), 1.20 – 1.12 (m, 3H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 158.50, 158.47, 134.97, 133.91, 133.85, 133.77, 133.18, 132.29, 132.27, 128.73, 128.58, 126.92, 126.91, 113.66, 113.65, 70.68, 67.73, 67.61, 55.14, 53.25, 52.85, 52.83, 41.48, 37.03, 37.00, 32.54, 32.42, 31.05, 29.21, 29.11, 23.51, 23.44. HRMS (ESI) exact mass calculated for [M+Na$^+$] (C$_{18}$H$_{22}$NaO$_2$S) requires m/z 325.1238, found m/z 325.1230.

4-(4-methoxyphenyl)-1-phenyl-4-(phenylthio)butan-1-ol (1o)
Colorless liquid (52%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.38 – 7.05 (m, 12H), 6.84 – 6.72 (m, 2H), 4.63 (dt, $J$ = 12.5, 6.1 Hz, 1H), 4.09 (dt, $J$ = 16.2, 8.0 Hz, 1H), 3.77 (s, 3H), 2.07 – 1.75 (m, 4H), 1.25 (s, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 158.61, 144.39, 144.28, 134.97, 133.92, 133.79, 132.49, 128.83, 128.82, 128.65, 128.63, 128.53, 127.69, 127.66, 127.02, 125.90, 125.81, 113.74, 113.73, 74.31, 74.21, 55.24, 52.86, 36.90, 36.73, 32.43, 32.40. HRMS (ESI) exact mass calculated for [M+Na$^+$] (C$_{23}$H$_{24}$NaO$_2$S) requires m/z 387.1395, found m/z 387.1380.

5-(4-methoxyphenyl)-2-methyl-5-(phenylthio)pentan-2-ol (1p)
Colorless liquid (60%). $^1$H NMR (600 MHz, CDCl$_3$) δ 7.25 – 7.16 (m, 5H), 7.14 (d, $J$ = 8.6 Hz, 2H), 6.79 (d, $J$ = 8.6 Hz, 2H), 4.07 (dd, $J$ = 8.8, 6.1 Hz, 1H), 3.78 (s, 3H), 2.10 – 1.91 (m, 2H), 1.62 (s, 1H), 1.58 – 1.36 (m, 2H), 1.16 (d, $J$ = 1.9 Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) δ 158.52, 134.99, 133.94, 132.34, 128.76, 128.61, 126.95, 113.69, 70.75, 55.18, 53.29, 41.52, 31.08, 29.25, 29.16. HRMS (ESI) exact mass calculated for [M+Na$^+$] (C$_{19}$H$_{24}$NaO$_2$S) requires m/z 339.1395, found m/z 339.1399.
tert-butyl(5-(4-methoxyphenyl)-5-(phenylthio)pentyloxy)dimethylsilane (1q)
Colorless liquid (74%). $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.26 – 7.13 (m, 6H), 6.78 (d, $J$ = 7.6 Hz, 2H), 4.16 – 4.04 (m, 1H), 3.77 (s, 3H), 3.53 (t, $J$ = 6.0 Hz, 2H), 1.95 (m, 1H), 1.90 – 1.70 (m, 1H), 1.49 (m, 2H), 1.44 – 0.93 (m, 3H), 0.85 (s, 9H), 0.00 (s, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 158.49, 135.24, 134.03, 132.25, 128.78, 128.58, 126.84, 113.64, 77.21, 77.00, 76.79, 62.85, 55.18, 52.87, 36.09, 32.40, 25.93, 23.80, 18.30, -5.32. GC−MS (EI, QMS, m/z) 416 (<1%), 307, 249, 175 (100%), 147, 121, 73.

(3) Synthetic procedure of 1q and 1r

(a) LiBu$^+$ (3.0equiv), THF, 3-bromopropanoic acid (1.0equiv) or 4-bromobutanoic acid (1.0equiv), 0 °C.

4-(4-methoxyphenyl)-4-(phenylthio)butanoic acid (1r)
White solid (63%). mp: 110 - 112°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.27 – 7.18 (m, 5H), 7.15 (d, $J$ = 8.4 Hz, 2H), 6.81 (d, $J$ = 8.3 Hz, 2H), 4.23 – 4.10 (m, 1H), 3.78 (s, 3H), 2.39 (t, $J$ = 7.8 Hz, 2H), 2.34 – 2.12 (m, 2H), 1.25 (s, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 178.09, 158.82, 134.49, 132.78, 132.49, 128.83, 128.73, 127.21, 113.89, 55.23, 51.81, 31.64, 30.97. HRMS (ESI) exact mass calculated for [M+Na$^+$] (C$_{17}$H$_{18}$NaO$_3$S) requires m/z 325.0874, found m/z 325.0870.

4-(4-methoxyphenyl)-4-(phenylthio)butanoic acid (1s)
White solid (36%). mp: 101 - 103 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.27 – 7.16 (m,
5H), 7.14 (d, J = 8.7 Hz, 2H), 6.79 (d, J = 8.6 Hz, 2H), 4.09 (dd, J = 8.7, 6.3 Hz, 1H), 3.77 (s, 3H), 2.31 (t, J = 7.2 Hz, 2H), 2.22 – 1.85 (m, 2H), 1.76 – 1.53 (m, 2H), 1.26 (s, 1H). 13C NMR (150 MHz, CDCl3) δ 178.92, 158.62, 134.80, 133.48, 132.45, 128.76, 128.65, 127.06, 113.76, 55.20, 52.59, 35.44, 33.50, 22.72. HRMS (ESI) exact mass calculated for [M+Na+] (C18H20NaO3S) requires m/z339.1031, found m/z339.1030.

N-(4-(4-methoxyphenyl)-4-(phenylthio)butyl)-4-methylbenzenesulfonamide (1t)
Pale yellow solid (65%). mp: 118 - 119 °C. 1H NMR (600 MHz, CDCl3) δ 7.62 (d, J = 8.2 Hz, 2H), 7.22 – 7.18 (m, 3H), 7.17 – 7.10 (m, 4H), 7.01 (d, J = 8.6 Hz, 2H), 6.70 (d, J = 8.6 Hz, 2H), 4.16 (t, J = 6.2 Hz, 1H), 3.94 (dd, J = 8.8, 6.2 Hz, 1H), 3.71 (s, 3H), 2.90 – 2.74 (m, 2H), 2.35 (s, 3H), 1.94 – 1.70 (m, 2H), 1.47 – 1.26 (m, 2H). 13C NMR (150 MHz, CDCl3) δ 158.66, 143.42, 136.77, 134.65, 133.33, 132.45, 129.70, 128.69, 127.16, 127.04, 113.78, 55.22, 52.28, 42.76, 33.16, 27.56, 21.51. HRMS (ESI) exact mass calculated for [M+Na+](C24H27NNaO3S2) requires m/z464.1330, found m/z 464.1293.

5. General procedure for photocatalytic cycloetherification of phenyl benzy sulfides
To a 10 mL round bottom flask equipped with a magnetic stir bar were added phenyl benzy sulfides 1 (0.1 mmol), Trifluoroacetic acid (0.15 mmol), Ru(bpy)3Cl2 (0.005 mmol) and dry CH3NO2 (1 mL). The mixture was irradiated with blue LED (6 W) at room temperature for 4 hours. Then the mixture was filtrated on a short plug of silica gel using ethyl acetate. The filtrate was concentrated and the residue was purified by flash column chromatography to give the final product 2.

6. Characterization of photocatalytic cycloetherification products 2

Colorless liquid, 1H NMR (400 MHz, CDCl3) δ 7.25 (d, J = 3.6 Hz, 2H), 6.87 (d, J = 8.6 Hz, 2H), 4.83 (t, J = 7.2 Hz, 1H), 4.08 (dd, J = 14.7, 7.2 Hz, 1H), 3.91 (dd, J = 14.3, 7.9 Hz, 1H), 3.80 (s, 3H), 2.36 – 2.20 (m, 1H), 2.11 – 1.94 (m, 2H), 1.85 – 1.71 (m, 1H). 13C NMR (150 MHz, CDCl3) δ 158.76, 135.30, 126.94, 133.75, 80.42, 68.47, 55.25, 34.45, 26.05. GC–MS (EI, QMS, m/z) 178 (46%), 177, 147, 136, 135 (100%). HRMS (ESI) exact mass calculated for [M+H+] (C11H15O2) requires m/z 179.1072,
2-(2-methoxyphenyl)tetrahydrofuran (2b) ²
Colorless liquid, 1H NMR (600 MHz, CDCl₃) δ 7.42 (d, J = 7.5 Hz, 1H), 7.22 (t, J = 7.7 Hz, 1H), 6.95 (t, J = 7.4 Hz, 1H), 6.85 (d, J = 8.1 Hz, 1H), 5.17 (t, J = 6.9 Hz, 1H), 4.10 (dd, J = 14.1, 7.1 Hz, 1H), 3.92 (dd, J = 14.2, 7.4 Hz, 1H), 2.45 – 2.25 (m, 1H), 2.02 – 1.86 (m, 2H), 1.68 - 1.73 (m, 1H). 13C NMR (150 MHz, CDCl₃) δ 156.09, 132.19, 127.73, 125.55, 109.99, 75.86, 68.50, 55.22, 33.05, 25.87. GC−MS (EI, QMS, m/z) 178 (83%), 177, 147, 136, 135 (100%), 119. HRMS (ESI) exact mass calculated for [M+Na⁺] (C₁₁H₁₄NaO₂) requires m/z 201.0891, found m/z 201.0881.

2-p-tolyltetrahydrofuran (2c) ²
Colorless liquid, 1H NMR (400 MHz, CDCl₃) δ 7.26 (d, J = 7.4 Hz, 2H), 7.17 (d, J = 7.3 Hz, 2H), 4.89 (t, J = 7.0 Hz, 1H), 4.12 (q, J = 7.1 Hz, 1H), 3.95 (q, J = 7.2 Hz, 1H), 2.37 (s, 3H), 2.31 (m, 1H), 2.12 – 1.96 (m, 2H), 1.90 – 1.73 (m, 1H). 13C NMR (151 MHz, CDCl₃) δ 140.34, 136.68, 128.93, 125.58, 80.55, 68.54, 34.55, 26.00, 21.07. GC−MS (EI, QMS, m/z) 162 (46%), 161, 147, 119 (100%), 91.

2-(4-tert-butylphenyl)tetrahydrofuran (2d) ²
Colorless liquid, 1H NMR (400 MHz, CDCl₃) δ 7.35 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 7.7 Hz, 2H), 4.86 (t, J = 7.2 Hz, 1H), 4.08 (dd, J = 14.8, 7.1 Hz, 1H), 3.92 (dd, J = 14.2, 7.8 Hz, 1H), 2.40 – 2.20 (m, 1H), 2.11 – 1.93 (m, 2H), 1.93 – 1.75 (m, 1H), 1.31 (s, 9H). 13C NMR (150 MHz, CDCl₃) δ 150.01, 140.24, 125.43, 125.17, 80.49, 68.55, 34.45, 34.28, 31.35, 26.09. GC−MS (EI, QMS, m/z) 204 (8%), 203,189, 161, 147 (100%), 105, 91.

2-(2,5-dimethoxyphenyl)tetrahydrofuran (2e)
Colorless liquid, 1H NMR (400 MHz, CDCl₃) δ 7.03 (d, J = 2.9 Hz, 1H), 6.78 – 6.71 (m, 2H), 5.13 (t, J = 7.0 Hz, 1H), 4.10 (dd, J = 14.5, 6.9 Hz, 1H), 3.92 (dd, J = 14.8, 7.2 Hz, 1H), 3.78 (s, 6H), 2.42 – 2.34 (m, 1H), 2.00 – 1.91 (m, 2H), 1.74 – 1.66 (m, 1H). 13C NMR (150 MHz, CDCl₃) δ 153.64, 150.32, 133.56, 112.00, 111.74, 111.11,
75.85, 68.55, 55.85, 55.75, 33.17, 25.84. GC−MS (EI, QMS, m/z) 208 (90%), 193, 177(100%), 166, 165, 151, 135. HRMS (ESI) exact mass calculated for [M+H⁺] (C₁₂H₁₇O₃) requires m/z 209.1178, found m/z 209.1162.

2-(3,4-dimethoxyphenyl)tetrahydrofuran (2f)
Colorless liquid, ¹H NMR (600 MHz, CDCl₃) δ 7.04 – 6.70 (m, 3H), 4.82 (t, J = 7.2 Hz, 1H), 4.09 (q, J = 7.2 Hz, 1H), 3.95 – 3.91 (m, 1H), 3.90 (s, 3H), 3.87 (s, 3H), 3.11 – 2.31 (m, 1H), 2.12 – 1.91 (m, 2H), 1.83-1.77 (m, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 148.86, 148.08, 135.75, 117.85, 110.84, 108.83, 80.53, 68.47, 55.88, 55.78, 34.43, 25.98. GC−MS (EI, QMS, m/z) 208 (76%), 193, 177(100%), 166, 165, 151, 135. HRMS (ESI) exact mass calculated for [M+H⁺] (C₁₂H₁₇O₃) requires m/z 209.1178, found m/z 209.1170.

2-(3,4,5-trimethoxyphenyl)tetrahydrofuran (2g)
Colorless liquid, ¹H NMR (400 MHz, CDCl₃) δ 6.57 (s, 2H), 4.82 (t, J = 7.2 Hz, 1H), 4.10 (dd, J = 14.7, 7.2 Hz, 1H), 3.93 (dd, J = 14.6, 7.6 Hz, 1H), 3.87 (s, 6H), 3.83 (s, 3H), 2.27 – 2.35 (m, 1H), 2.22 – 1.91 (m, 2H), 1.85 – 1.76 (m, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 153.13, 139.06, 136.82, 102.36, 80.68, 68.60, 60.78, 56.01, 34.51, 25.93. GC−MS (EI, QMS, m/z) 238 (100%), 237, 223, 207, 195, 181. HRMS (ESI) exact mass calculated for [M+Na⁺] (C₁₃H₉O₄) requires m/z 261.1103, found m/z 261.1094.

5-(tetrahydrofuran-2-yl)benzo[d][1,3]dioxole (2h)
Colorless liquid, ¹H NMR (600 MHz, CDCl₃) δ 7.01 – 6.70 (m, 3H), 4.79 (t, J = 7.1 Hz, 1H), 4.07 (dd, J = 14.5, 7.2 Hz, 1H), 3.90 (dd, J = 14.6, 7.3 Hz, 1H), 2.31 – 2.20 (m, 1H), 2.07 – 1.90 (m, 2H), 1.79 – 1.73 (m, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 147.63, 146.60, 137.36, 118.97, 107.95, 106.28, 100.87, 80.58, 68.56, 34.64, 25.99. GC−MS (EI, QMS, m/z) 192 (84%), 191, 162, 150, 149 (100%), 135. HRMS (ESI) exact mass calculated for [M+H⁺] (C₁₁H₁₂NaO₃) requires m/z 215.0684, found m/z 215.0679.
2-(5-chloro-2-methoxyphenyl)tetrahydrofuran (2i)
Colorless liquid, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39 (d, $J = 2.7$ Hz, 1H), 7.15 (dd, $J = 8.7$, 2.7 Hz, 1H), 6.75 (d, $J = 8.7$ Hz, 1H), 5.10 (t, $J = 7.0$ Hz, 1H), 4.10 (dd, $J = 14.2$, 7.0 Hz, 1H), 3.91 (dd, $J = 14.9$, 7.3 Hz, 1H), 3.80 (s, 2H), 2.42 – 2.34 (m, 1H), 2.07 – 1.86 (m, 2H), 1.69 – 1.61 (m, 1H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 154.60, 134.26, 127.24, 125.65, 125.59, 111.19, 75.45, 68.62, 55.54, 33.07, 25.80. GC–MS (EI, QMS, m/z) 212 (33%), 177 (100%), 169, 155, 135.

2-(5-tert-butyl-2-methoxyphenyl)tetrahydrofuran (2j)
Colorless liquid, $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.45 (s, 1H), 7.22 (dd, $J = 8.5$, 2.3 Hz, 1H), 6.78 (d, $J = 8.5$ Hz, 1H), 5.14 (t, $J = 7.0$ Hz, 1H), 4.12 (dd, $J = 14.4$, 6.9 Hz, 1H), 3.92 (dd, $J = 14.8$, 7.3 Hz, 1H), 3.80 (s, 3H), 2.39 – 2.34 (m, 1H), 2.05 – 1.88 (m, 2H), 1.75 – 1.69 (m, 1H), 1.31 (s, 9H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 153.94, 142.97, 131.23, 124.15, 122.64, 109.53, 76.23, 68.44, 55.28, 34.17, 33.03, 31.55, 25.85. GC–MS (EI, QMS, m/z) 234 (26%), 219 (100%), 203, 191, 177. HRMS (ESI) exact mass calculated for [M+H$^+$] (C$_{15}$H$_{23}$O$_2$) requires m/z 235.1698 found m/z 235.1685.

2-(4-methoxy-3, 5-dimethylphenyl)tetrahydrofuran (2k)
Colorless liquid, $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 6.98 (s, 2H), 4.76 (t, $J = 7.3$ Hz, 1H), 4.08 (dd, $J = 14.5$, 7.4 Hz, 1H), 3.90 (dd, $J = 14.2$, 8.0 Hz, 1H), 3.70 (s, 3H), 2.28 (s, 6H), 2.27 – 2.22 (m, 1H), 2.07 – 1.92 (m, 2H), 1.82 – 1.76 (m, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 156.02, 138.28, 130.59, 126.14, 80.47, 68.51, 59.63, 34.30, 26.09, 16.11. GC–MS (EI, QMS, m/z) 206 (78%), 205, 191 (100%), 175, 163,149. HRMS (ESI) exact mass calculated for [M+H$^+$] (C$_{13}$H$_{18}$NaO$_2$) requires m/z 235.1204 found m/z 229.1213.

2-(3,5-di-tert-butyl-2-methoxyphenyl)tetrahydrofuran (2l)
White solid, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32 (s, 1H), 7.26 (s, 2H), 5.18 (t, $J = 7.3$ Hz, 1H), 4.12 (dd, $J = 14.3$, 7.1 Hz, 1H), 3.90 (dd, $J = 14.8$, 7.5 Hz, 1H), 3.79 (s, 3H),
2.45 – 2.27 (m, 1H), 2.15 – 1.93 (m, 2H), 1.89 – 1.80 (m, 1H), 1.39 (s, 9H), 1.30 (s, 9H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 155.17, 145.65, 141.46, 135.43, 123.47, 121.95, 75.68, 68.45, 62.54, 35.26, 34.60, 34.00, 31.52, 31.19, 26.34. GC–MS (EI, QMS, m/z) 290 (30%), 275 (100%), 247, 233, 57. HRMS (ESI) exact mass calculated for [M+H$^+$] (C$_{19}$H$_{31}$O$_2$) requires m/z 291.2324, found m/z 291.2313.

2-(4-methoxyphenyl)-5-methyltetrahydrofuran (2n)$^3$
Colorless liquid, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.28 (d, $J = 8.2$ Hz, 2H), 6.87 (d, $J = 8.3$ Hz, 2H), 4.90 (dt, $J = 66.2$, 7.2 Hz, 1H), 4.40 – 4.07 (m, 1H), 3.80 (s, 3H), 2.32 – 2.01 (m, 2H), 1.93 – 1.58 (m, 2H), 1.33 (dd, $J = 19.2$, 6.1 Hz, 3H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 158.79, 158.72, 135.87, 135.42, 127.16, 126.91, 113.66, 113.64, 80.77, 79.96, 75.76, 75.69, 55.26, 35.55, 34.48, 34.33, 33.14, 21.58, 21.38. GC–MS (EI, QMS, m/z) 192 (35%), 191, 197(100%), 161, 136, 135 (100%).

2-(4-methoxyphenyl)-5-phenyltetrahydrofuran (2o)
Colorless liquid, $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.48 – 7.22 (m, 7H), 6.96 – 6.85 (m, 2H), 5.23 (dt, $J = 14.1$, 6.8 Hz, 1H), 5.02 (dt, $J = 14.1$, 6.8 Hz, 1H), 3.81 (d, $J = 2.1$ Hz, 3H), 2.51 – 2.34 (m, 2H), 2.02 – 1.93 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 158.93, 158.87, 143.76, 143.06, 135.56, 134.91, 128.32, 128.29, 127.32, 127.22, 127.12, 126.91, 125.96, 125.55, 113.76, 113.74, 81.11, 81.08, 81.07, 81.03, 55.28, 35.59, 35.50, 34.47, 34.24. GC–MS (EI, QMS, m/z) 254 (13%), 146, 135, 118, 117 (100%). HRMS (ESI) exact mass calculated for [M+H$^+$] (C$_{17}$H$_{19}$O$_2$) requires m/z 255.1385, found m/z 255.1396.

5-(4-methoxyphenyl)-2,2-dimethyltetrahydrofuran (2p)
Colorless liquid, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.27 (d, $J = 8.5$ Hz, 2H), 6.86 (d, $J = 8.2$ Hz, 2H), 4.92 (t, $J = 6.5$ Hz, 1H), 3.79 (s, 3H), 2.44 – 2.22 (m, 1H), 2.22 – 1.80 (m, 3H), 1.35 (d, $J = 13.6$ Hz, 6H). $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 158.75, 135.52, 127.07, 113.61, 81.03, 80.16, 55.24, 39.08, 35.56, 29.10, 28.37. GC–MS (EI, QMS, m/z) 206 (57%), 205, 137 (100%), 136, 135, 108. HRMS (ESI) exact mass calculated
for [M+Na+](C13H18NaO2) requires m/z 229.1204, found m/z 229.1205.

2-(4-methoxyphenyl)tetrahydro-2H-pyran (2q)

Colorless liquid, 1H NMR (400 MHz, CDCl3) δ 7.27 (d, J = 8.6 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 4.27 (d, J = 10.4 Hz, 1H), 4.12 (d, J = 13.5 Hz, 1H), 3.79 (s, 3H), 3.60 (t, J = 11.4 Hz, 1H), 2.00 – 1.90 (m, 1H), 1.94 – 1.78 (m, 1H), 1.72 – 1.54 (m, 4H). 13C NMR (150 MHz, CDCl3) δ 158.80, 135.57, 127.12, 113.62, 79.76, 69.02, 55.24, 33.82, 25.88, 24.00. GC–MS (EI, QMS, m/z) 192 (56%), 191, 161, 136, 135 (100%).

5-(4-methoxyphenyl)dihydrofuran-2(3H)-one (2r)

Colorless liquid, 1H NMR (400 MHz, CDCl3) δ 7.27 (d, J = 8.5 Hz, 2H), 6.92 (d, J = 8.5 Hz, 2H), 5.51 – 5.41 (m, 1H), 3.82 (s, 3H), 2.71 – 2.54 (m, 3H), 2.31 – 2.12 (m, 1H). 13C NMR (150 MHz, CDCl3) δ 176.93, 159.71, 131.10, 126.92, 114.06, 81.32, 30.87, 29.19. GC–MS (EI, QMS, m/z) 192 (59%), 148, 137(100%), 135, 77. HRMS (ESI) exact mass calculated for [M+H+] (C11H23O3) requires m/z 193.0865, found m/z 193.0855.

6-(4-methoxyphenyl)tetrahydro-2H-pyran-2-one (2s)

Colorless liquid, 1H NMR (600 MHz, CDCl3) δ 7.27 (d, J = 9.1 Hz, 2H), 6.90 (d, J = 7.7 Hz, 2H), 5.30 (d, J = 10.7 Hz, 1H), 3.81 (s, 3H), 2.68 – 2.73 (m, 1H), 2.63 – 2.52 (m, 1H), 2.22 – 2.07 (m, 1H), 2.02 – 1.94 (m, 2H), 1.84 – 1.91 (m, 1H). 13C NMR (150 MHz, CDCl3) δ 171.55, 159.53, 131.76, 127.18, 113.90, 113.85, 81.51, 55.29, 30.32, 29.41, 18.61. GC–MS (EI, QMS, m/z) 206 (38%), 147, 137, 135, 134 (100%).

2-(4-methoxyphenyl)-1-tosylpyrrolidine (2t)

Pale yellow solid, mp: 119 - 120 °C. 1H NMR (600 MHz, CDCl3) δ 7.66 (d, J = 8.1 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.5 Hz, 2H), 6.83 (d, J = 8.6 Hz, 2H), 4.72 (dd, J = 7.8, 4.0 Hz, 1H), 3.79 (s, 3H), 3.61 – 3.58 (m, 1H), 3.51 – 3.30 (m, 1H), 2.42 (s, 3H), 1.98 – 1.92 (m, 1H), 1.90 – 1.74 (m, 2H), 1.69 – 1.62 (m, 1H). 13C NMR
(150 MHz, CDCl₃) δ 158.57, 143.16, 135.08, 129.49, 127.43, 127.26, 113.62, 62.76, 55.23, 49.26, 35.73, 23.92, 21.48. GC–MS (EI, QMS, m/z) 331 (5%), 176, 175 (100%). 174, 147, 121, 91. HRMS (ESI) exact mass calculated for [M+H⁺] (C₁₈H₂₂NO₃S) requires m/z 332.1320, found m/z 332.1314.

Reference
7. $^1$H and $^{13}$C NMR spectrum of phenyl benzyl sulfides 1

1a
1c
8. $^1$H and $^{13}$C NMR spectrum of products 2

2a
2b

Chemical shifts and coupling constants for compound 2b.

- Chemical shifts (ppm): 1.06, 1.97, 1.08, 2.07, 1.00, 0.95, 0.99, 0.96, 0.93

Additional peaks and structures are also shown in the spectrum.
2e

Chemical structure with NMR spectra.