Iodine catalyzed cross-dehydrogenative C–S coupling by C(sp²)–H bond activation: Direct access to aryl sulfides from aryl thiols

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**General Information:**

Unless otherwise noted, chemicals were purchased from commercial suppliers at the highest purity grade available and were used without further purification. Dithioacetal derivative 2g was synthesized according to literature procedure. Thin layer chromatography was performed on Merck pre-coated 0.25 mm silica gel plates (60F–254) using UV light as visualizing agent and/or iodine as developing agent. All the final products were purified by column chromatography using silica gel (100–200 mesh).

Melting points were recorded on Opti Melt Automated Melting Point System and are uncorrected. \(^1\)H (500 MHz) and \(^{13}\)C (125 MHz) NMR spectra were recorded on a Bruker spectrometer. Chemical shifts (\(\delta\)) were reported as parts per million (ppm) in \(\delta\) scale downfield from TMS. Spectra were referenced internally to the residual proton resonance in CDCl\(_3\) (\(\delta\) 7.26 ppm), DMSO-\(d_6\) (\(\delta\) 2.50 ppm, the middle peak) or with tetramethylsilane (TMS, \(\delta\) 0.00 ppm) as the internal standard. \(^{13}\)C NMR spectra were referenced to CDCl\(_3\) (\(\delta\) 77.0 ppm, the middle peak) and DMSO (\(\delta\) 39.5 ppm, the middle peak). Coupling constants were expressed in Hz. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, dt = doublet of triplet, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were obtained on a Brüker micrOTOF™-Q II mass spectrometer (ESI-MS).

**General procedure for the synthesis of aryl sulfides:**

To a mixture of aryl thiol 1 (0.75/0.5 mmol) and electron-rich species 2 (0.5 mmol) was added DMSO (1.5 mmol, 0.1 mL) followed by iodine (10 mol\%, 0.05 mmol, 0.013 g). Then the reaction mixture was heated on a pre-heated oil bath at 80 °C for 3-5 h under solvent-free conditions. The reaction was monitored by TLC. After completion of the reaction, the reaction mixture was diluted with CH\(_2\)Cl\(_2\)/ethyl acetate, and quenched with saturated sodium thiosulphate solution and extracted twice with CH\(_2\)Cl\(_2\)/ethyl acetate (2 X 15 mL). The organic layer was washed with water and dried over anhyd. sodium sulphate. The solvent was evaporated in vacuo, and the residue was subjected to column chromatography using ethyl acetate in hexanes as eluent to afford pure aryl sulfide derivative 3.
Table-S1: Iodine catalyzed cross-dehydrogenative coupling of aryl thiols 1 and electron-rich species 2.

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<tr>
<th>S. No.</th>
<th>Aryl thiol</th>
<th>Electron-rich arene</th>
<th>Product</th>
<th>Time</th>
<th>Yield (%)</th>
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| 1      | Me\textsuperscript{-}SH (1a) | 2\textsubscript{a} | \begin{tikzpicture}
    \node at (0,0) {\chemfig{S-O-H}};
    \node at (1.5,0) {\chemfig{S-Me-O-H}};
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| 2      | Cl\textsuperscript{-}SH (1b) | 2\textsubscript{a} | \begin{tikzpicture}
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    \node at (1.5,0) {\chemfig{S-Cl-O-H}};
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    \node at (1.5,0) {\chemfig{S-Br-O-H}};
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| 6      | Me\textsuperscript{-}SH (1a) | 2\textsubscript{b} | \begin{tikzpicture}
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    \node at (1.5,0) {\chemfig{S-Me-O-O-H}};
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| 7      | Cl\textsuperscript{-}SH (1b) | 2\textsubscript{b} | \begin{tikzpicture}
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    \node at (1.5,0) {\chemfig{S-Cl-O-O-H}};
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| 8      | Br\textsuperscript{-}SH (1c) | 2\textsubscript{b} | \begin{tikzpicture}
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Characterization Data:

1-(p-Tolylthio)naphthalen-2-ol (3aa):

Yield: 0.130 g (99%) as yellow solid; Mp: 78–79 °C (Lit:\textsuperscript{2} 77–79 °C); \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}): \(\delta\) 8.31 (d, \(J = 8.0\) Hz, 1H), 7.93 (d, \(J = 9.0\) Hz, 1H), 7.85 (d, \(J = 8.0\) Hz, 1H), 7.54 (ddd, \(J = 1.0, 7.0, 8.0\) Hz, 1H), 7.43–7.38 (m, 2H), 7.30 (s, 1H), 7.02 (s, 4H), 2.28 (s, 3H) ppm; \(^13\)C NMR (125 MHz, CDCl\textsubscript{3}): \(\delta\) 156.8 (C), 135.8 (C), 135.4 (C), 132.6 (CH), 131.7 (C), 129.9 (CH), 129.4 (C), 128.5 (CH), 127.8 (CH), 126.6 (CH), 124.7 (CH), 123.7 (CH), 116.8 (CH), 108.7 (C), 20.8 (CH\textsubscript{3}) ppm; HRMS (ESI\textsuperscript{+}): \(m/z\) calcd for C\textsubscript{17}H\textsubscript{14}SONa [M + Na\textsuperscript{+}]: 289.0658, found 289.0657.

1-(4-Chlorophenylthio)naphthalen-2-ol (3ab):

Yield: 0.139 g (98%) as brown solid; Mp: 84–85 °C (Lit:\textsuperscript{2} 85–87 °C); \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}): \(\delta\) 8.20 (d, \(J = 8.5\) Hz, 1H), 7.93 (d, \(J = 9.0\) Hz, 1H), 7.84 (d, \(J = 8.0\) Hz, 1H), 7.55–7.51 (m, 1H), 7.42–7.38 (m, 1H), 7.37 (d, \(J = 9.0\) Hz, 1H), 7.15 (s, 1H), 7.14 (d, \(J = 9.0\) Hz, 2H), 6.97 (d, \(J = 8.5\) Hz, 2H) ppm; \(^13\)C NMR (125 MHz, CDCl\textsubscript{3}): \(\delta\) 157.0 (C), 135.1 (C), 133.9 (C), 133.1 (CH), 131.8 (C), 129.5 (C), 129.2 (CH), 128.6 (CH), 128.1 (CH), 127.6 (CH), 124.4 (CH), 124.0 (CH), 116.9 (CH), 107.6 (C) ppm; HRMS (ESI\textsuperscript{+}): \(m/z\) calcd for C\textsubscript{16}H\textsubscript{11}ClSONa [M + Na\textsuperscript{+}]: 309.0111, found 309.0111.

1-(4-Bromophenylthio)naphthalen-2-ol (3ac):

Yield: 0.150 g (91%) as brown solid; Mp: 104–105 °C (Lit:\textsuperscript{2} 103–105 °C); \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}): \(\delta\) 8.05 (d, \(J = 8.5\) Hz, 1H), 7.80 (d, \(J = 9.0\) Hz, 1H), 7.71 (d, \(J = 7.5\) Hz, 1H), 7.39 (t, \(J = 7.5\) Hz, 1H), 7.27 (t, \(J = 7.5\) Hz, 1H), 7.23 (d, \(J = 9.0\) Hz, 1H), 7.16 (d, \(J = 8.5\) Hz, 2H), 7.00 (s, 1H), 6.77 (d, \(J = 8.5\) Hz, 2H) ppm; \(^13\)C NMR (125 MHz, CDCl\textsubscript{3}): \(\delta\) 157.0 (C), 135.1 (C), 134.6 (C), 133.1 (CH), 132.1 (CH), 129.5 (C), 128.6 (CH), 128.1 (CH), 127.8 (CH), 124.4 (CH), 124.0 (CH), 119.6 (C), 116.9 (CH), 107.4 (C) ppm; HRMS (ESI\textsuperscript{+}): \(m/z\) calcd for C\textsubscript{16}H\textsubscript{11}BrSONa [M + Na\textsuperscript{+}]: 352.9606, found 352.9596.
1-(Phenylthio)naphthalen-2-ol (3ad):

Yield: 0.119 g (95%) as white solid; Mp: 66–67 °C (Lit:\textsuperscript{2} 65–67 °C); \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): δ 8.32 (d, J = 8.0 Hz, 1H), 7.96 (d, J = 9.0 Hz, 1H), 7.87 (d, J = 8.5 Hz, 1H), 7.58–7.53 (m, 1H), 7.46–7.41 (m, 2H), 7.29 (br s, 1H), 7.25–7.20 (m, 2H), 7.16 (d, J = 6.5 Hz, 1H), 7.11 (d, J = 7.5 Hz, 2H) ppm; \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}): δ 156.9 (C), 135.4 (C), 135.3 (C), 132.7 (CH), 129.4 (C), 129.1 (CH), 128.5 (CH), 127.9 (CH), 126.3 (CH), 125.8 (CH), 124.6 (CH), 123.8 (CH), 116.8 (CH), 108.0 (C) ppm; HRMS (ESI\textsuperscript{+}): m/z calcd for C\textsubscript{16}H\textsubscript{12}SONa [M + Na]\textsuperscript{+}: 275.0501, found 275.0493.

1-(Naphthalen-2-ylthio)naphthalen-2-ol (3ae):

Yield: 0.128 g (85%) as white solid; Mp: 94–95 °C; \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): δ 8.31 (d, J = 8.5 Hz, 1H), 7.96 (d, J = 9.0 Hz, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.76–7.73 (m, 1H), 7.68 (d, J = 9.0 Hz, 1H), 7.60–7.57 (m, 1H), 7.51 (ddd, J = 1.0, 7.0, 8.0 Hz, 1H), 7.47 (d, J = 1.5 Hz, 1H), 7.44–7.40 (m, 4H), 7.28 (s, 1H), 7.22 (dd, J = 2.0, 9.0 Hz, 1H) ppm; \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}): δ 157.1 (C), 135.4 (C), 133.7 (C), 132.9 (CH), 132.7 (C), 131.7 (C), 129.5 (C), 128.9 (CH), 128.6 (CH), 128.0 (CH), 127.7 (CH), 127.0 (CH), 126.6 (CH), 125.6 (CH), 124.7 (CH), 124.6 (CH), 124.5 (CH), 123.9 (CH), 116.9 (CH), 108.0 (C) ppm; HRMS (ESI\textsuperscript{+}): m/z calcd for C\textsubscript{20}H\textsubscript{14}SONa [M + Na]\textsuperscript{+}: 325.0658, found 325.0656.

4-(p-Tolylthio)benzene-1,3-diol (3ba):

Yield: 0.105 g (91%) as brown solid; Mp: 77–78 °C; \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): δ 7.40 (d, J = 8.5 Hz, 1H), 7.06 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 8.0 Hz, 2H), 6.71 (br s, 1H), 6.60 (d, J = 2.5 Hz, 1H), 6.48 (dd, J = 2.5, 8.5 Hz, 1H), 6.26 (br s, 1H), 2.29 (s, 3H) ppm; \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}): δ 159.1 (C), 158.2 (C), 138.0 (CH), 135.9 (C), 132.9 (C), 129.9 (CH), 126.6 (CH), 109.2 (CH), 108.0 (C), 102.4 (CH), 20.8 (CH\textsubscript{3}) ppm; HRMS (ESI–): m/z calcd for C\textsubscript{13}H\textsubscript{11}SO\textsubscript{2} [M – H]\textsuperscript{−}: 231.0474, found 231.0482.
4-(4-Chlorophenylthio)benzene-1,3-diol (3bb):

Yield: 0.115 g (92%) as white solid; Mp: 99–100 °C; \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) 7.36 (d, \(J = 8.0\) Hz, 1H), 7.18 (d, \(J = 9.0\) Hz, 2H), 6.96 (d, \(J = 8.5\) Hz, 2H), 6.61 (br s, 1H), 6.59 (d, \(J = 2.5\) Hz, 1H), 6.49 (dd, \(J = 2.5, 8.5\) Hz, 1H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \(\delta\) 159.4 (C), 158.2 (C), 138.2 (CH), 135.2 (C), 131.8 (C), 129.2 (CH), 127.4 (CH), 109.5 (CH), 106.8 (C), 102.6 (CH) ppm; HRMS (ESI–): \(m/z\) calcd for C\(_{12}\)H\(_8\)ClSO\(_2\) [M – H]–: 250.9928, found 250.9942.

4-(4-Bromophenylthio)benzene-1,3-diol (3bc):

Yield: 0.128 g (86%) as brown solid; Mp: 116–117 °C; \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) 7.36 (d, \(J = 8.5\) Hz, 1H), 7.32 (d, \(J = 8.5\) Hz, 2H), 6.89 (d, \(J = 8.5\) Hz, 2H), 6.59 (br s, 1H), 6.58 (d, \(J = 2.5\) Hz, 1H), 6.48 (dd, \(J = 2.5, 8.5\) Hz, 1H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \(\delta\) 159.4 (C), 158.2 (C), 138.2 (CH), 135.9 (C), 132.0 (CH), 127.7 (CH), 119.6 (C), 109.5 (CH), 106.6 (C), 102.6 (CH) ppm; HRMS (ESI–): \(m/z\) calcd for C\(_{12}\)H\(_8\)BrSO\(_2\) [M – H]–: 294.9423, found 294.9433.

4-(phenylthio)benzene-1,3-diol (3bd):

Yield: 0.086 g (89%) as brown solid; Mp: 111–112 °C; \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) 7.23 (d, \(J = 8.5\) Hz, 1H), 7.07 (t, \(J = 7.5\)Hz, 2H), 6.95–6.70 (m, 1H), 6.91 (dd, \(J = 1.0, 8.5\) Hz, 2H), 6.47 (d, \(J = 2.5\) Hz, 1H), 6.34 (dd, \(J = 2.5, 8.5\) Hz, 1H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \(\delta\) 159.2 (C), 158.1 (C), 138.2 (CH), 136.5 (C), 129.0 (CH), 126.1 (CH), 125.8 (CH), 109.4 (CH), 107.1 (C), 102.4 (CH) ppm; HRMS (ESI–): \(m/z\) calcd for C\(_{12}\)H\(_9\)SO\(_2\) [M – H]–: 217.0318, found 217.0324.

4-(Naphthalen-2-ylthio)benzene-1,3-diol (3be):

Yield: 0.110 g (82%) as white solid; Mp: 95–96 °C; \(^1\)H NMR (500 MHz, DMSO-\(_d_6\)): \(\delta\) 9.82 (brs, 2H), 7.82–7.75 (m, 2H), 7.68 (d, \(J = 8.0\) Hz, 1H), 7.48 (s, 1H), 7.44–7.36 (m, 2H), 7.30 (d, \(J = 8.5\) Hz, 1H), 7.22 (d, \(J = 8.5\) Hz, 1H), 6.58 (s, 1H), 6.41 (dd, \(J = 1.5, 8.0\) Hz, 1H) ppm; \(^{13}\)C NMR S-9
(125 MHz, DMSO-$d_6$): δ 160.4 (C), 159.8 (C), 137.9 (CH), 136.8 (C), 133.4 (C), 130.9 (C), 128.3 (CH), 127.7 (CH), 126.7 (CH), 125.2 (CH), 125.0 (CH), 123.3 (CH), 108.2 (CH), 105.7 (C), 103.4 (CH) ppm; HRMS (ESI–): m/z calcd for C$_{16}$H$_{11}$SO$_2$ [M – H]$^-$: 267.0474, found 267.0482.

$p$-Tolyl(2,4,6-trimethoxyphenyl)sulfane (3ca):

Yield: 0.132 g (92%) as white solid; Mp: 111–112 °C (Lit.$^3$ 112–113 °C); $^1$H NMR (500 MHz, CDCl$_3$): δ 6.96 (d, $J$ = 2.0 Hz, 4H), 6.21 (s, 2H), 3.85 (s, 3H), 3.79 (s, 6H), 2.24 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): δ 162.6 (C), 162.3 (C), 134.9 (C), 133.9 (C), 129.1 (CH), 125.8 (CH), 99.1 (C), 91.0 (CH), 56.1 (CH$_3$), 55.2 (CH$_2$), 20.7 (CH$_3$) ppm; HRMS (ESI+): m/z calcd for C$_{16}$H$_{18}$SO$_3$Na [M + Na]$^+$: 313.0869, found 313.0869.

(4-Chlorophenyl)(2,4,6-trimethoxyphenyl)sulfane (3cb):

Yield: 0.146 g (95%) as white solid; Mp: 92–93 °C (Lit.$^3$ 93–94 °C); $^1$H NMR (500 MHz, CDCl$_3$): δ 7.09 (d, $J$ = 8.5 Hz, 2H), 6.93 (d, $J$ = 8.5 Hz, 2H), 6.19 (s, 2H), 3.84 (s, 3H), 3.79 (s, 6H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): δ 163.0 (C), 162.3 (C), 137.3 (C), 129.9 (C), 128.4 (CH), 126.8 (CH), 98.1 (C), 91.1 (CH), 56.1 (CH$_3$), 55.3 (CH$_3$) ppm; HRMS (ESI+): m/z calcd for C$_{15}$H$_{15}$ClSO$_3$Na [M + Na]$^+$: 333.0323, found 333.0317.

(4-Bromophenyl)(2,4,6-trimethoxyphenyl)sulfane (3cc):

Yield: 0.158 g (90%) as white solid; Mp: 113–114 °C (Lit.$^3$ 114–115 °C); $^1$H NMR (500 MHz, CDCl$_3$): δ 7.24 (d, $J$ = 8.5 Hz, 2H), 6.87 (d, $J$ = 8.5 Hz, 2H), 6.20 (s, 2H), 3.85 (s, 3H), 3.78 (s, 6H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): δ 163.0 (C), 162.3 (C), 138.1 (C), 131.3 (CH), 127.1 (CH), 117.7 (C), 97.9 (C), 91.1 (CH), 56.2 (CH$_3$), 55.3 (CH$_3$) ppm; HRMS (ESI+): m/z calcd for C$_{15}$H$_{15}$BrSO$_3$Na [M + Na]$^+$: 376.9817, found 376.9814.
Phenyl(2,4,6-trimethoxyphenyl)sulfane (3cd):

Yield: 0.125 g (91%) as white solid; Mp: 92–93 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.16–7.13 (m, 2H), 7.02 (d, $J$ = 7.5 Hz, 3H), 6.21 (s, 2H), 3.84 (s, 3H), 3.78 (s, 6H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 162.8 (C), 162.3 (C), 138.6 (C), 128.3 (CH), 125.4 (CH), 124.2 (CH), 98.4 (C), 91.0 (CH), 56.1 (CH$_3$), 55.2 (CH$_3$) ppm; HRMS (ESI+): $m/z$ calcd for C$_{15}$H$_{16}$SO$_3$Na [M + Na]$^+$: 299.0712, found 299.0712.

Naphthalen-2-yl(2,4,6-trimethoxyphenyl)sulfane (3ce):

Yield: 0.152 g (94%) as white solid; Mp: 87–88 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.72 (d, $J$ = 8.0 Hz, 1H), 7.65 (d, $J$ = 8.5 Hz, 1H), 7.62 (d, $J$ = 8.0 Hz, 1H), 7.42–7.37 (m, 2H), 7.34 (ddd, $J$ = 1.5, 7.0, 8.0 Hz, 1H), 6.26 (s, 2H), 3.88 (s, 3H), 3.80 (s, 6H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 162.9 (C), 162.5 (C), 136.3 (C), 133.7 (C), 131.1 (C), 127.8 (CH), 127.5 (CH), 126.7 (CH), 126.0 (CH), 124.7 (CH), 124.6 (CH), 122.8 (CH), 98.5 (C), 91.2 (CH), 56.2 (CH$_3$), 55.3 (CH$_3$) ppm; HRMS (ESI+): $m/z$ calcd for C$_{19}$H$_{18}$SO$_3$Na [M + Na]$^+$: 349.0869, found 349.0878.

2-(p-Tolylthio)benzene-1,3,5-triol (3da):

Yield: 0.115 g (93%) as brown solid; Mp: 125–126 °C; $^1$H NMR (500 MHz, DMSO-$d_6$): $\delta$ 9.50 (br s, 1H), 9.30 (br s, 2H), 7.01 (d, $J$ = 8.0 Hz, 2H), 6.87 (d, $J$ = 8.5 Hz, 2H), 5.97 (s, 2H), 2.20 (s, 3H) ppm; $^{13}$C NMR (125 MHz, DMSO-$d_6$): $\delta$ 160.8 (C), 160.4 (C), 135.8 (C), 133.4 (C), 129.2 (CH), 125.4 (CH), 94.7 (CH), 93.3 (C), 20.5 (CH$_3$) ppm; HRMS (ESI–): $m/z$ calcd for C$_{13}$H$_{11}$SO $[M - H]^-$: 247.0423, found 247.0441.
2-(4-Chlorophenylthio)benzene-1,3,5-triol (3db):

Yield: 0.112 g (84%) as brown solid; Mp: 110–111 °C; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 9.53 (br s, 2H), 7.24 (d, <i>J</i> = 8.5 Hz, 2H), 6.93 (d, <i>J</i> = 8.5 Hz, 2H), 5.97 (s, 2H) ppm; ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 161.1 (C), 160.9 (C), 138.9 (C), 129.0 (C), 128.8 (CH), 126.9 (CH), 95.1 (CH), 92.8 (C) ppm; HRMS (ESI–): <i>m/z</i> calcd for C₁₂H₈ClSO₃ [M – H]⁻: 266.9877, found 266.9888.

2-(4-Bromophenylthio)benzene-1,3,5-triol (3dc):

Yield: 0.122 g (78%) as brown solid; Mp: 127–128 °C; ¹H NMR (500 MHz, DMSO-<i>d</i>₆): δ 9.56 (br s, 1H), 9.49 (br s, 2H), 7.36 (d, <i>J</i> = 8.5 Hz, 2H), 6.86 (d, <i>J</i> = 8.5 Hz, 2H), 5.95 (s, 2H) ppm; ¹³C NMR (125 MHz, DMSO-<i>d</i>₆): δ 161.0 (C), 160.8 (C), 139.5 (C), 131.5 (CH), 127.1 (CH), 116.8 (C), 95.0 (CH), 92.5 (C) ppm; HRMS (ESI–): <i>m/z</i> calcd for C₁₂H₈BrSO₃ [M – H]⁻: 310.9372, found 376.9378.

N,N-Dimethyl-4-(p-tolylthio)benzenamine (3ea):

Yield: 0.112 g (90%) as yellow solid; Mp: 51–52 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.43 (d, <i>J</i> = 9.0 Hz, 2H), 7.13 (d, <i>J</i> = 8.5 Hz, 2H), 7.08 (d, <i>J</i> = 8.0 Hz, 2H), 6.74 (d, <i>J</i> = 9.0 Hz, 2H), 3.01 (s, 6H), 2.33 (s, 3H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 150.3 (C), 136.2 (C), 135.4 (C), 135.0 (C), 129.5 (CH), 127.7 (CH), 118.5 (C), 112.9 (CH), 40.2 (CH₃), 20.8 (CH₃) ppm; HRMS (ESI+): <i>m/z</i> calcd for C₁₅H₁₇NSNa [M + Na]⁺: 266.0974, found 266.0972.

4-(4-Chlorophenylthio)-N,N-dimethylbenzenamine (3eb):

Yield: 0.110 g (84%) as yellow solid; Mp: 120–121 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.39 (d, <i>J</i> = 8.5 Hz, 2H), 7.17 (d, <i>J</i> = 8.5 Hz, 2H), 7.03 (d, <i>J</i> = 9.0 Hz, 2H), 6.72 (d, <i>J</i> = 8.5 Hz, 2H), 3.00 (s, 6H) ppm; ¹³C
NMR (125 MHz, CDCl₃): δ 150.7 (C), 139.0 (C), 136.2 (CH), 130.6 (C), 128.7 (CH), 128.0 (CH), 116.7 (C), 113.0 (CH), 40.2 (CH₃) ppm; HRMS (ESI+): m/z calcd for C₁₄H₁₅ClNS [M + H]⁺: 264.0608, found 264.0604.

4-(4-Bromophenylthio)-N,N-dimethylbenzenamine (3ec):

Yield: 0.117 g (76%) as yellow solid; Mp: 127–128 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.40 (d, J = 9.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 6.98 (d, J = 8.5 Hz, 2H), 6.72 (d, J = 9.0 Hz, 2H), 3.01 (s, 6H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 150.7 (C), 139.8 (C), 136.2 (CH), 131.6 (C), 128.1 (CH), 118.4 (CH), 116.4 (C), 112.9 (CH), 40.2 (CH₃) ppm; HRMS (ESI+): m/z calcd for C₁₄H₁₅BrNS [M + H]⁺: 308.0103, found 308.0100.

N,N-Dimethyl-4-(phenylthio)benzenamine (3ed):

Yield: 0.082 g (72%) as brown solid; Mp: 68–69 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.29–7.24 (m, 2H), 7.08–7.03 (m, 2H), 6.96–6.90 (m, 1H), 6.54 (d, J = 8.5 Hz, 2H), 2.80 (s, 6H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 150.5 (C), 140.2 (C), 136.0 (CH), 128.6 (CH), 126.7 (CH), 124.8 (CH), 117.2 (C), 112.8 (CH), 40.1 (CH₃) ppm; HRMS (ESI+): m/z calcd for C₁₄H₁₆NS [M + H]⁺: 230.0998, found 230.0996.

4-Hydroxy-3-(p-tolylthio)-2H-chromen-2-one (3fa):

Yield: 0.135 g (96%) as white solid; Mp: 160–161 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 11.00 (br s, 1H), 7.94 (d, J = 7.5 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.41–7.30 (m, 2H), 7.15 (d, J = 8.0 Hz, 2H), 7.08 (d, J = 7.5 Hz, 2H), 2.20 (s, 3H) ppm; ¹³C NMR (125 MHz, DMSO-d₆): δ 168.1 (C), 160.9 (C), 153.0 (C), 135.2 (C), 133.5 (CH), 132.3 (C), 129.7 (CH), 127.1 (CH), 124.3 (CH), 124.2 (CH), 116.4 (CH), 115.7 (C), 95.3 (C), 20.5 (CH₃) ppm; HRMS (ESI+): m/z calcd for C₁₆H₁₂SO₃Na [M + Na]⁺: 307.0399, found 307.406.
3-(4-Chlorophenylthio)-4-hydroxy-2H-chromen-2-one (3fb):

Yield: 0.151 g (100%) as white solid; Mp: 187–188 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 7.94 (dd, J = 0.5, 8.0 Hz, 1H), 7.68 (t, J = 8.0 Hz, 1H), 7.41–7.35 (m, 2H), 7.31 (d, J = 9.0 Hz, 2H), 7.21 (d, J = 9.0 Hz, 2H) ppm; ¹³C NMR (125 MHz, DMSO-d₆): δ 168.8 (C), 161.1 (C), 153.2 (C), 135.3 (C), 133.9 (CH), 130.3 (C), 129.1 (CH), 128.2 (CH), 124.5 (CH), 124.4 (CH), 116.6 (CH), 115.9 (C), 94.2 (C) ppm; HRMS (ESI+): m/z calcd for C₁₅H₁₀ClSO₃Na [M + Na]⁺: 326.9853, found 326.9873.

3-(4-Bromophenylthio)-4-hydroxy-2H-chromen-2-one (3fc):

Yield: 0.156 g (90%) as light yellow solid; Mp: 197–198 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 7.93 (dd, J = 1.5, 8.0 Hz, 1H), 7.66 (ddd, J = 1.5, 7.0, 8.5 Hz, 1H), 7.42 (d, J = 8.5 Hz, 2H), 7.39–7.33 (m, 2H), 7.15 (d, J = 9.0 Hz, 2H) ppm; ¹³C NMR (125 MHz, DMSO-d₆): δ 168.8 (C), 161.0 (C), 153.2 (C), 135.9 (C), 133.8 (CH), 131.9 (CH), 128.5 (CH), 124.5 (CH), 124.4 (CH), 118.5 (C), 116.6 (CH), 115.8 (C), 94.2 (C) ppm; HRMS (ESI+): m/z calcd for C₁₅H₉BrSO₃Na [M + Na]⁺: 370.9348, found 370.9361.

4-Hydroxy-3-(phenylthio)-2H-chromen-2-one (3fd):

Yield: 0.135 g (100%) as white solid; Mp: 197–198 °C; ¹H NMR (500 MHz, DMSO-d₆): δ 7.96 (dd, J = 1.0, 8.0 Hz, 1H), 7.74–7.68 (m, 1H), 7.45–7.39 (m, 2H), 7.31–7.25 (m, 2H), 7.19–7.13 (m, 2H) ppm; ¹³C NMR (125 MHz, DMSO-d₆): δ 168.4 (C), 160.9 (C), 153.0 (C), 135.9 (C), 133.7 (CH), 129.1 (CH), 126.3 (CH), 125.6 (CH), 124.4 (CH), 124.3 (CH), 116.5 (CH), 115.6 (C), 94.5 (C) ppm; HRMS (ESI+): m/z calcd for C₁₅H₁₀SO₃Na [M + Na]⁺: 293.0243, found 293.0231.
4-Hydroxy-3-(naphthalen-2-ylthio)-2H-chromen-2-one (3fe):

Yield: 0.140 g (88%) as light yellow solid; Mp: 160–161 °C; $^1$H NMR (500 MHz, DMSO-d$_6$): $\delta$ 7.99 (dd, $J = 1.0$, 7.5 Hz, 1H), 7.84–7.81 (m, 2H), 7.79 (d, $J = 8.0$ Hz, 1H), 7.73 (d, $J = 1.5$ Hz, 1H), 7.68 (ddd, $J = 1.5$, 7.0, 8.5 Hz, 1H), 7.45–7.37 (m, 5H) ppm; $^{13}$C NMR (125 MHz, DMSO-d$_6$): $\delta$ 168.6 (C), 161.1 (C), 153.2 (C), 133.7 (CH), 133.7 (C), 133.5 (C), 131.2 (C), 128.6 (CH), 127.7 (CH), 127.0 (CH), 126.7 (CH), 125.6 (CH), 125.0 (CH), 124.5 (CH), 124.3 (CH), 124.0 (CH), 116.6 (CH), 115.9 (C), 94.5 (C) ppm; HRMS (ESI+): m/z calcd for C$_{19}$H$_{12}$SO$_3$Na [M + Na]$^+$: 343.0399, found 343.0392.

1-(4-Chlorophenyl)-3,3-bis(methylthio)-2-(p-tolylthio)prop-2-en-1-one (3ga):

Yield: 0.190 g (100%) as yellow solid; Mp: 81–82 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.66 (d, $J = 8.5$ Hz, 2H), 7.32 (d, $J = 8.5$ Hz, 2H), 7.14 (d, $J = 7.5$ Hz, 2H), 6.93 (d, $J = 7.5$ Hz, 2H), 2.47 (s, 3H), 2.22 (s, 3H), 2.15 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 189.6 (C), 139.4 (C), 139.2 (C), 138.8 (C), 134.6 (C), 134.1 (CH), 133.6 (C), 130.4 (CH), 129.5 (CH), 128.6 (CH), 126.6 (C), 21.0 (CH$_3$), 18.3 (CH$_3$), 16.3 (CH$_3$) ppm; HRMS (ESI+): m/z calcd for C$_{18}$H$_{17}$ClS$_3$ONa [M + Na]$^+$: 403.0022, found 403.0029.

1-(4-Chlorophenyl)-2-(4-chlorophenylthio)-3,3-bis(methylthio)prop-2-en-1-one (3gb):

Yield: 0.200 g (100%) as yellow solid; Mp: 67–68 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.67 (d, $J = 8.5$ Hz, 2H), 7.35 (d, $J = 8.5$ Hz, 2H), 7.19 (d, $J = 8.5$ Hz, 2H), 7.11 (d, $J = 8.5$ Hz, 2H), 2.47 (s, 3H), 2.18 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 189.4 (C), 139.7 (C), 137.8 (C), 136.4 (C), 134.6 (C), 134.4 (CH), 134.3 (C), 130.4 (CH), 129.5 (C), 129.0 (CH), 128.7 (CH), 18.4 (CH$_3$), 16.4 (CH$_3$) ppm; HRMS (ESI+): m/z calcd for C$_{17}$H$_{14}$Cl$_3$S$_3$ONa [M + Na]$^+$: 422.9476, found 422.9476.
2-(4-Bromophenylthio)-1-(4-chlorophenyl)-3,3-bis(methylthio)prop-2-en-1-one (3gc):

Yield: 0.210 g (94%) as yellow solid; Mp: 74–75 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.61 (d, $J$ = 8.5 Hz, 2H), 7.29 (d, $J$ = 8.5 Hz, 2H), 7.21 (d, $J$ = 8.5 Hz, 2H), 7.06 (d, $J$ = 8.5 Hz, 2H), 2.41 (s, 3H), 2.12 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 189.4 (C), 139.7 (C), 137.8 (C), 136.0 (C), 134.5 (CH), 134.4 (C), 132.0 (CH), 130.5 (CH), 130.3 (C), 128.8 (CH), 122.8 (C), 18.5 (CH$_3$), 16.5 (CH$_3$) ppm; HRMS (ESI$^+$): m/z calcd for C$_{17}$H$_{14}$BrClS$_2$ONa [M + Na]$^+$: 466.8971, found 466.8974.

1-(4-Chlorophenyl)-3,3-bis(methylthio)-2-(phenylthio)prop-2-en-1-one (3gd):

Yield: 0.183 g (100%) as yellow viscous liquid; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.58 (d, $J$ = 8.5 Hz, 2H), 7.24 (d, $J$ = 8.5 Hz, 2H), 7.19 (d, $J$ = 7.0 Hz, 2H), 7.10–7.02 (m, 3H), 2.40 (s, 3H), 2.10 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 189.5 (C), 139.4 (C), 137.8 (C), 135.9 (C), 134.5 (C), 133.5 (CH), 130.6 (C), 130.4 (CH), 128.7 (CH), 128.6 (CH), 128.4 (CH), 18.4 (CH$_3$), 16.4 (CH$_3$) ppm; HRMS (ESI$^+$): m/z calcd for C$_{17}$H$_{15}$ClS$_3$ONa [M + Na]$^+$: 388.9866, found 388.9869.

1-(4-Chlorophenyl)-3,3-bis(methylthio)-2-(naphthalen-2-ylthio)prop-2-en-1-one (3ge):

Yield: 0.204 g (98%) as yellow solid; Mp: 100–101 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.77 (s, 1H), 7.72 (d, $J$ = 5.0 Hz, 1H), 7.65 (dd, $J$ = 8.5, 15.0 Hz, 4H), 7.46–7.40 (m, 2H), 7.37 (dd, $J$ = 1.0, 8.0 Hz, 1H), 7.30 (d, $J$ = 8.5 2H), 2.51 (s, 3H), 2.22 (s, 3H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 189.6 (C), 139.4 (C), 137.3 (C), 137.0 (C), 134.5 (C), 133.2 (C), 132.8 (CH), 132.6 (C), 130.4 (CH), 129.9 (CH), 128.6 (CH), 128.4 (CH), 128.2 (C), 127.5 (CH), 126.6 (CH), 126.4 (CH), 18.5 (CH$_3$), 16.4 (CH$_3$) ppm; HRMS (ESI$^+$): m/z calcd for C$_{21}$H$_{17}$ClS$_3$ONa [M + Na]$^+$: 439.0022, found 439.0022.

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References


3dc