Supplementary Material

PhIO-Mediated oxidative dethioacetalization/dethioketalization under water-free conditions

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1. Mechanistic Studies

To a solution of 2,2-diphenyl-1,3-dithiolane (1 mmol) in DCM (4 mL) was added $^{18}$O-labeled PhIO (1.2 mmol, $^{16}$O:$^{18}$O = 72:28). The mixture was stirred at room temperature until TLC revealed a complete consumption of the substrate. The solvent was removed by reduced pressure to obtain the crude products which were further purified by flash column chromatography to afford the parent aldehydes. HRMS analysis of the PhIO and aldehyde product showed that both of them had $^{18}$O-labeled ingredients. The spectra diagrams are shown below.

Figure 1. HRMS spectrum of PhI$^{16}/^{18}$O.
Figure 2. HRMS spectrum of product 3aa.
2. Data of parent aldehydes and ketones

**Benzaldehyde (2a)** Following the general procedure, 2a was purified by silica gel chromatography (EtOAc/PE = 0/100). Yield: 153 mg, 90%, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 10.02 (d, $J = 1.7$ Hz, 1H), 7.88 (dt, $J = 8.2$, $1.4$ Hz, 2H), 7.63 (td, $J = 7.3$, $1.6$ Hz, 1H), 7.53 (td, $J = 7.7$, $1.8$ Hz, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 192.3, 136.5, 134.4, 129.7, 129.0. HRMS (ESI) calcd for C$_7$H$_4$O$^+$ [M + H$^+$] 107.0491, found 107.0496.

**Isopropylbenzaldehyde (2b)** Following the general procedure, 2b was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 270 mg, 91%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.97 (s, 1H), 7.89 – 7.73 (m, 2H), 7.43 – 7.34 (m, 2H), 2.99 (p, $J = 6.9$ Hz, 1H), 1.28 (dd, $J = 7.0$, $0.8$ Hz, 6H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 192.1, 156.3, 134.5, 130.0, 127.2, 34.5, 23.7. HRMS (ESI) calcd for C$_{10}$H$_{13}$O$^+$ [M + H$^+$] 149.0961, found 149.0966.

**Methoxybenzaldehyde (2c)** Following the general procedure, 2c was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 245 mg, 90%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.88 (s, 1H), 7.92 - 7.73 (m, 2H), 7.00 (dt, $J = 8.8$, $1.0$ Hz, 2H), 3.88 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 190.9, 164.6, 132.0, 130.0, 114.3, 55.6. HRMS (ESI) calcd for C$_9$H$_{13}$O$_2$ [M + H$^+$] 137.0599, found 137.0592.

**Methylthiobenzaldehyde (2e)** Following the general procedure, 2e was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 274 mg, 90%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.91 (s, 1H), 7.76 (d, $J = 8.1$ Hz, 2H), 7.31 (d, $J = 8.6$ Hz, 2H), 2.52 (d, $J = 0.9$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 191.3, 147.9, 132.9, 130.0, 125.2, 14.7. HRMS (ESI) calcd for C$_9$H$_{13}$NO$^+$ [M + H$^+$] 153.0369, found 153.0364.

**Benzyl oxy-3-methoxybenzaldehyde (2f)** Following the general procedure, 2f was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 441 mg, 91%, a yellow solid, mp. 76-78 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.83 (s, 1H), 7.44 (dd, $J = 8.2$, $1.8$ Hz, 3H), 7.41 - 7.35 (m, 3H), 7.35 - 7.29 (m, 1H), 6.99 (d, $J = 8.2$ Hz, 1H), 5.24 (s, 2H), 3.94 (d, $J = 1.1$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 191.0, 153.6, 150.1, 136.0, 130.3, 128.8, 128.2, 127.2, 126.6, 112.4, 109.3, 70.9, 56.1. HRMS (ESI) calcd for C$_{15}$H$_{17}$O$_2$ [M + H$^+$] 243.1016, found 243.1011.

**Formylphenyl acetate (2g)** Following the general procedure, 2g was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 302 mg, 92%, a white solid, mp. 96-98 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.99 (s, 1H), 7.97 – 7.86 (m, 2H), 7.28 (d, $J = 8.4$ Hz, 2H), 2.34 (d, $J = 0.6$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 190.9, 168.7, 155.4, 134.0, 131.2, 122.4, 21.2. HRMS (ESI) calcd for C$_9$H$_{12}$O$_3$ [M + H$^+$] 165.0546, found 165.0541.

**Chlorobenzaldehyde (2h)** Following the general procedure, 2h was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 247 mg, 88%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.97 (s, 1H), 7.84 (td, $J = 1.9$, $1.1$ Hz, 1H), 7.76 (dq, $J = 7.5$, $1.1$ Hz, 1H), 7.59 (ddt, $J = 8.1$, $2.2$, $1.0$ Hz, 1H), 7.48 (t, $J = 7.8$ Hz, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 191.0, 137.8, 135.5, 134.4, 130.4, 129.3, 128.0. HRMS (ESI) calcd for C$_7$H$_5$ClO$^+$ [M + H$^+$] 141.0102, found 141.0107.

**Bromobenzaldehyde (2i)** Following the general procedure, 2i was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 315 mg, 85%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 10.36 (dd, $J = 5.4$, $2.6$ Hz, 1H), 7.91 (ddt, $J = 4.6$, 2.9, $1.5$ Hz, 1H), 7.64 (ddd, $J = 5.9$, 4.1, 2.3 Hz, 1H), 7.44 (tdd, $J = 5.1$, 3.8, 2.3 Hz, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 191.9, 135.4, 133.9, 133.5, 129.9, 127.9, 127.1. HRMS (ESI) calcd for C$_7$H$_5$BrO$^+$ [M + H$^+$] 184.9597, found 184.9592.

**Methyl 2-formylenzoate (2j)** Following the general procedure, 2j was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 263 mg, 80%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 10.60 (d, $J = 4.5$ Hz, 1H), 8.01 – 7.85 (m, 2H), 7.70 – 7.56 (m, 2H), 4.01 – 3.92 (m, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 192.1, 166.7, 137.0, 133.0, 132.4, 132.0, 130.4, 128.4, 52.8. HRMS (ESI) calcd for C$_9$H$_9$O$_3$ [M + H$^+$] 165.0546, found 165.0541.
(E)-2-Methyl-3-phenylacrylaldehyde (2k) Following the general procedure, 2k was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 269 mg, 92%, colorless oil. 1H NMR (400 MHz, CDCl3) δ 9.59 (s, 1H), 7.57 – 7.51 (m, 2H), 7.49 – 7.43 (m, 2H), 7.43 – 7.37 (m, 1H), 7.27 (d, J = 1.6 Hz, 1H), 2.08 (d, J = 1.4 Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 195.7, 149.9, 138.4, 135.1, 130.1, 129.6, 128.7, 11.0. HRMS (ESI) calcd for C10H11O+ [M + H+] 147.0804, found 147.0804.

1-Naphthaldehyde (2l) Following the general procedure, 2l was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 281 mg, 90%, a white solid, mp. 104-105 °C. 1H NMR (400 MHz, CDCl3) δ 10.14 (s, 1H), 8.30 (d, J = 1.5 Hz, 1H), 8.05 – 7.83 (m, 4H), 7.63 (ddd, J = 8.2, 6.9, 1.4 Hz, 1H), 7.57 (ddd, J = 8.2, 6.9, 1.4 Hz, 1H). 13C NMR (101 MHz, CDCl3) δ 192.3, 136.5, 134.6, 134.1, 132.6, 129.6, 129.2, 129.1, 128.1, 127.1, 122.7. HRMS (ESI) calcd for C11H9O+ [M + H+] 157.0648, found 157.0643.

Furan-2-carbaldehyde (2m) Following the general procedure, 2m was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 163 mg, 90%, yellow oil. 1H NMR (400 MHz, CDCl3) δ 9.64 (dd, J = 2.2, 1.2 Hz, 1H), 7.67 (dq, J = 1.8, 0.9 Hz, 1H), 7.24 (dq, J = 3.6, 0.9 Hz, 1H), 6.59 (ddt, J = 3.7, 1.9, 1.1 Hz, 1H). 13C NMR (101 MHz, CDCl3) δ 177.9, 153.0, 148.1, 121.1, 112.6. HRMS (ESI) calcd for C3H5O+ [M + H+] 97.0284, found 97.0289.

Heptanal (2n) Following the general procedure, 2n was purified by silica gel chromatography (EtOAc/PE = 0/100). Yield: 217 mg, 95%, colorless oil. 1H NMR (400 MHz, CDCl3) δ 9.75 (t, J = 1.9 Hz, 1H), 2.41 (td, J = 7.4, 1.9 Hz, 2H), 1.62 (p, J = 7.4 Hz, 2H), 1.37 – 1.21 (m, 6H), 0.94 – 0.80 (m, 3H). 13C NMR (101 MHz, CDCl3) δ 203.0, 43.9, 31.5, 28.8, 22.5, 22.0, 14.0. HRMS (ESI) calcd for C7H15O+ [M + H+] 115.1117, found 115.1117.

4-Hydroxy-3-methoxybenzaldehyde (2o) Following the general procedure, 2o was purified by silica gel chromatography (EtOAc/PE = 15/85). Yield: 213 mg, 70%, a white solid, mp. 80-82 °C. 1H NMR (400 MHz, CDCl3) δ 9.81 (d, J = 0.9 Hz, 1H), 7.48 – 7.35 (m, 2H), 7.03 (dd, J = 8.6, 1.3 Hz, 1H), 6.67 – 6.24 (m, 1H), 3.94 (t, J = 1.6 Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 191.0, 151.8, 147.2, 129.8, 127.6, 114.5, 108.8, 56.1. HRMS (ESI) calcd for C8H8O3+ [M + H+] 153.0546, found 153.0551.

4-Hydroxybenzaldehyde (2p) Following the general procedure, 2p was purified by silica gel chromatography (EtOAc/PE = 15/85). Yield: 208 mg, 85%, a pale-yellow solid, mp. 110-113 °C. 1H NMR (400 MHz, DMSO-d6) δ 10.60 (s, 1H), 9.79 (d, J = 2.7 Hz, 1H), 8.14 – 7.62 (m, 2H), 7.32 – 6.80 (m, 2H). 13C NMR (101 MHz, DMSO-d6) δ 191.4, 163.8, 132.6, 128.9, 116.3. HRMS (ESI) calcd for C7H7O2+ [M + H+] 123.0441, found 123.0446.

Tert-butyl (4-formylphenyl)carbonate (2q) Following the general procedure, 2q was purified by silica gel chromatography (EtOAc/PE = 5/95). Yield: 422 mg, 95%, colorless oil. 1H NMR (400 MHz, CDCl3) δ 9.98 (s, 1H), 7.96 – 7.84 (m, 2H), 7.41 – 7.29 (m, 2H), 1.56 (s, 9H). 13C NMR (101 MHz, CDCl3) δ 190.9, 155.7, 150.9, 133.8, 131.2, 121.9, 84.4, 27.7. HRMS (ESI) calcd for C12H15O5+ [M + H+] 223.0965, found 223.0969.

4-((Trimethylsilyloxy)benzaldehyde (2r) Following the general procedure, 2r was purified by preparative high-performance liquid chromatography (DCM/Hexane = 10/90, 10 ml/min). Yield: 369 mg, 95%, colorless oil. 1H NMR (400 MHz, CDCl3) δ 9.89 (s, 1H), 7.88 – 7.72 (m, 2H), 7.03 – 6.86 (m, 2H), 0.31 (s, 9H). 13C NMR (101 MHz, CDCl3) δ 190.7, 160.8, 131.7, 130.2, 120.1, 0.0. HRMS (ESI) calcd for C10H15O2Si+ [M + H+] 195.0836, found 195.0833.

4-Methylbenzaldehyde (2s) Following the general procedure, 2s was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 221 mg, 92%, colorless oil. 1H NMR (400 MHz, CDCl3) δ 9.92 (s, 1H), 7.74 (d, J = 8.2 Hz, 2H), 7.29 (d, J = 8.1 Hz, 2H), 2.39 (s, 3H). 13C NMR (101 MHz, CDCl3) δ 191.9, 145.5, 134.2, 129.8, 129.7, 129.1, 21.8. HRMS (ESI) calcd for C8H8O+ [M + H+] 121.0648, found 121.0643.

Benzenophene (2aa) Following the general procedure, 2aa was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 335 mg, 92%, a white solid, mp. 46-48 °C. 1H NMR (400 MHz, CDCl3) δ 7.87 – 7.75 (m, 4H), 7.63 – 7.55 (m, 2H), 7.53 – 7.43 (m, 4H). 13C NMR (101 MHz, CDCl3) δ 196.8, 137.6, 132.5, 130.1, 128.3. HRMS (ESI) calcd for C13H10O+ [M + H+] 183.0804, found 183.0809.

N-(2-Acetylphenyl)acetamide (2ab) Following the general procedure, 2ab was purified by silica gel chromatography (EtOAc/PE = 10/90). Yield: 314 mg, 88%, a white solid, mp. 74-76 °C. 1H NMR (400 MHz,
Following the general procedure, 2ac was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 247 mg, 80%, colorless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 7.93 - 7.83\) (m, 2H), 7.47 - 7.37 (m, 2H), 2.61 - 2.54 (m, 3H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 202.9, 169.5, 141.0, 135.2, 131.6, 122.3, 121.7, 120.7, 28.7, 25.6\). HRMS (ESI) calcd for C\(_{10}H_{12}NO\(^{2+}\) [M + H\(^{+}\)] 178.0863, found 178.0868.

1-(4-Chlorophenyl)ethan-1-one (2af) Following the general procedure, 2af was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 209 mg, 78%, colorless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 7.70\) (dd, \(J = 7.7, 1.4\) Hz, 1H), 7.38 (td, \(J = 7.5, 1.5\) Hz, 1H), 7.31 - 7.21 (m, 2H), 2.58 (s, 3H), 2.53 (s, 3H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 196.6, 137.5, 132.8\) (q, \(J_{CF} = 38.3\) Hz), 129.5 (q, \(J_{CF} = 3.8\) Hz), 129.3, 125.1 (q, \(J_{CF} = 3.9\) Hz), 123.7 (q, \(J_{CF} = 270.9\) Hz), 119.8, 26.6. HRMS (ESI) calcd for C\(_{9}H_{8}F_{2}O^{+}\) [M + H\(^{+}\)] 181.0653, found 181.0652.

4-Methylpentan-2-one (2ah) Following the general procedure, 2ah was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 186 mg, 90%, colorless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 2.24\) (d, \(J = 7.0\) Hz, 2H), 2.06 (s, 4H), 0.86 (d, \(J = 6.7\) Hz, 6H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 208.9, 52.7, 30.3, 24.6, 22.5\). HRMS (ESI) calcd for C\(_{6}H_{13}O^{+}\) [M + H\(^{+}\)] 101.0961, found 101.0966.

9H-fluoren-9-one (2ag) Following the general procedure, 2ag was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 348 mg, 95%, a yellow solid, mp. 81-82 °C. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 7.62\) (d, \(J = 7.4\) Hz, 2H), 7.51 - 7.41 (m, 4H), 7.29 - 7.22 (m, 2H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 193.9, 144.4, 134.7, 134.2, 129.1, 124.3, 120.3\). HRMS (ESI) calcd for C\(_{13}H_{9}O^{+}\) [M + H\(^{+}\)] 181.0648, found 181.0653.

1-(Thiophen-2-y1)ethan-1-one (2ai) Following the general procedure, 2ai was purified by silica gel chromatography (EtOAc/PE = 2/98). Yield: 207 mg, 82%, colorless oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 7.69\) (d, \(J = 3.7\) Hz, 1H), 7.63 (d, \(J = 5.0\) Hz, 1H), 7.12 (t, \(J = 4.3\) Hz, 1H), 2.56 (d, \(J = 0.7\) Hz, 3H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 190.8, 144.6, 133.8, 132.5, 128.2, 27.0\). HRMS (ESI) calcd for C\(_{7}H_{8}S^{+}\) [M + H\(^{+}\)] 127.0212, found 127.0217.

3,4-Dihydronaphthalen-1(2H)-one (2aj) Following the general procedure, 2aj was purified by silica gel chromatography (EtOAc/PE = 1/99). Yield: 263 mg, 90%, pale-yellow oil. \(^{1}H\) NMR (400 MHz, CDCl\(_3\)) \(\delta 8.02\) (d, \(J = 7.9\) Hz, 1H), 7.45 (t, \(J = 7.5\) Hz, 1H), 7.29 (t, \(J = 7.6\) Hz, 1H), 7.24 (d, \(J = 7.7\) Hz, 1H), 2.95 (t, \(J = 6.1\) Hz, 2H), 2.64 (t, \(J = 6.5\) Hz, 2H), 2.13 (p, \(J = 6.5\) Hz, 2H). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta 198.4, 144.5, 133.4, 132.6, 128.8, 127.2, 126.6, 39.2, 29.7, 23.3\). HRMS (ESI) calcd for C\(_{16}H_{12}O^{+}\) [M + H\(^{+}\)] 271.1693, found 271.1698.
3. \(^1\)H-NMR and \(^{13}\)C-NMR Spectra

\(^1\)H-NMR Spectrum of 2-Phenyl-1,3-dithiolane (1a)
$^{13}$C-NMR Spectrum of 2-Phenyl-1,3-dithiolane (1a)
\[1\text{H-NMR Spectrum of 2-}(4\text{-Isopropylphenyl})\text{-}1,3\text{-dithiolane (1b)}\]
$^{13}$C-NMR Spectrum of 2-(4-Isopropylphenyl)-1,3-dithiolane (1b)
\( ^1H \text{-NMR Spectrum of 2-(4-Methoxyphenyl)-1,3-dithiolane (1c)} \)
$^{13}$C-NMR Spectrum of 2-(4-Methoxyphenyl)-1,3-dithiolane (1c)
$^1$H-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)-N, N-dimethylaniline (1d)
$^{13}$C-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)-$N$, $N$-dimethylaniline (1d)
$^1$H-NMR Spectrum of 2-(4-(Methylthio)phenyl)-1,3-dithiolane (1e)
$^{13}$C-NMR Spectrum of 2-(4-(Methylthio)phenyl)-1,3-dithiolane (1e)
$^1$H-NMR Spectrum of 2-(4-(Benzyloxy)-3-methoxyphenyl)-1,3-dithiolane (1f)
$^{13}$C-NMR Spectrum of 2-(4-(Benzyloxy)-3-methoxyphenyl)-1,3-dithiolane (1f)
$^1$H-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)phenyl acetate (1g)
$^{13}$C-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)phenyl acetate (1g)
$^1$H-NMR Spectrum of 2-(3-Chlorophenyl)-1,3-dithiolane (1h)
$^{13}$C-NMR Spectrum of 2-(3-Chlorophenyl)-1,3-dithiolane (1h)
^1H-NMR Spectrum of 2-(2-Bromophenyl)-1,3-dithiolane (1i)
$^{13}$C-NMR Spectrum of 2-(2-Bromophenyl)-1,3-dithiolane (1i)
H-NMR Spectrum of Methyl 2-(1,3-dithiolan-2-yl)benzoate (1j)
$^{13}$C-NMR Spectrum of Methyl 2-(1,3-dithiolan-2-yl)benzoate (1j)
$^1$H-NMR Spectrum of (E)-2-(1-Phenylprop-1-en-2-yl)-1,3-dithiolane (1k)
13C-NMR Spectrum of (E)-2-(1-Phenylprop-1-en-2-yl)-1,3-dithiolane (1k)
$^1$H-NMR Spectrum of 2-(Naphthalen-1-yl)-1,3-dithiolane (1l)
$^{13}$C-NMR Spectrum of 2-(Naphthalen-1-yl)-1,3-dithiolane (1l)

![13C-NMR Spectrum](image)

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$^1$H-NMR Spectrum of 2-(1,3-Dithiolan-2-yl)furan (1m)
$^{13}$C-NMR Spectrum of 2-(1,3-Dithiolan-2-yl)furan (1m)
$^1$H-NMR Spectrum of 2-Hexyl-1,3-dithiolane (1n)
\(^{13}\)C-NMR Spectrum of 2-Hexyl-1,3-dithiolane (1n)
$^1$H-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)-2-methoxyphenol (1o)
$^{13}$C-NMR Spectrum of 4-(1,3-Dithiolan-2-yl)-2-methoxyphenol (1o)
1H-NMR Spectrum of 4-(1,3-Dithian-2-yl)phenol (1p)
$^{13}$C-NMR Spectrum of 4-(1,3-Dithian-2-yl)phenol (1p)
$^{1}$H-NMR Spectrum of 4-(1,3-Dithian-2-yl)phenyl tert-butyl carbonate (1q)
$^{13}$C-NMR Spectrum of 4-(1,3-Dithian-2-yl)phenyl tert-butyl carbonate (1q)
$^1$H-NMR Spectrum of 2-(4-Methoxyphenyl)-1,3-dithiane

\[ \text{1c'} \]
$^{13}$C-NMR Spectrum of 2-(4-Methoxyphenyl)-1,3-dithiane

1c'
$^1$H-NMR Spectrum of 4-(1,3-Dithian-2-yl)-N, N-dimethylaniline (1d')
$^{13}$C-NMR Spectrum of 4-(1,3-Dithian-2-yl)-$N$, $N$-dimethylaniline (1d')
$^1$H-NMR Spectrum of (4-(1,3-dithian-2-yl)phenoxy)trimethylsilane (1r)
\[^{13}\text{C-NMR Spectrum of (4-(1,3-dithian-2-yl)phenoxy)trimethylsilane (1r)}\]
\(^1\text{H-NMR Spectrum of (p-Tolylmethylene)bis(p-tolylsulfane) (1s)}\)
$^{13}$C-NMR Spectrum of (p-Tolylmethylene)bis(p-tolylsulfane) (1s)
$^1$H-NMR Spectrum of 2,2-Diphenyl-1,3-dithiolane (1aa)
$^{13}$C-NMR Spectrum of 2,2-Diphenyl-1,3-dithiolane (1aa)
$^1$H-NMR Spectrum of N-(2-(2-Methyl-1,3-dithiolan-2-yl)phenyl)acetamide (1ab)
$^{13}$C-NMR Spectrum of N-(2-(2-Methyl-1,3-dithiolan-2-yl)phenyl)acetamide (1ab)
$^1$H-NMR Spectrum of 2-(4-Chlorophenyl)-2-methyl-1,3-dithiolane (1ac)
$^{13}$C-NMR Spectrum of 2-(4-Chlorophenyl)-2-methyl-1,3-dithiolane (1ac)
$^1$H-NMR Spectrum of 2-Methyl-2-(o-tolyl)-1,3-dithiolane (1ad)
$^{13}$C-NMR Spectrum of 2-Methyl-2-(o-tolyl)-1,3-dithiolane (1ad)
$^1$H-NMR Spectrum of 2-Methyl-2-(3-(trifluoromethyl)phenyl)-1,3-dithiolane (1ae)
$^{13}$C-NMR Spectrum of 2-Methyl-2-(3-(trifluoromethyl)phenyl)-1,3-dithiolane (1ae)
$^1$H-NMR Spectrum of 2-Isobutyl-2-methyl-1,3-dithiolane (1af)
$^{13}$C-NMR Spectrum of 2-Isobutyl-2-methyl-1,3-dithiolane (1af)
$^1$H-NMR Spectrum of Spiro[fluorene-9,2'-[1,3]dithiolane] (1ag)
\(^{13}\text{C}-\text{NMR Spectrum of Spiro[fluorene-9,2'-[1,3]dithiolane]}\) (1ag)
$^1$H-NMR Spectrum of 2-Methyl-2-(thiophen-2-yl)-1,3-dithiolane (1ah)
$^{13}$C-NMR Spectrum of 2-Methyl-2-(thiophen-2-yl)-1,3-dithiolane (1ah)
$^1$H-NMR Spectrum of 3,4-Dihydro-2H-spiro[naphthalene-1,2'-[1,3]dithiane] (1ai)
13C-NMR Spectrum of 3,4-Dihydro-2H-spiro[naphthalene-1,2'-[1,3]dithiane] (1ai)
'$^1$H-NMR Spectrum of (8R,9S,13S,14S)-13-Methyl-6,7,8,9,11,12,13,14,15,16 decahydrospiro[cyclopenta[a]phenanthrene-17,2'-[1,3]dithiolan]-3-ol (1aj)
$^{13}$C-NMR Spectrum of ($8R$,9$S$,13$S$,14$S$)-13-Methyl-6,7,8,9,11,12,13,14,15,16 decahydrospiro[cyclopenta-$a$phenanthrene-17,2'-$[1,3]$dithiolan]-3-ol (1aj)
$^1$H-NMR Spectrum of Benzaldehyde (2a)
$^{13}$C-NMR Spectrum of Benzaldehyde (2a)
$^1$H-NMR Spectrum of 4-Isopropylbenzaldehyde (2b)
$^{13}$C-NMR Spectrum of 4-Isopropylbenzaldehyde (2b)
$^1$H-NMR Spectrum of 4-Methoxybenzaldehyde (2c)
$^{13}$C-NMR Spectrum of 4-Methoxybenzaldehyde (2c)
$^1$H-NMR Spectrum of 4-(Dimethylamino)benzaldehyde (2d)

2d

$^1$H-NMR spectrum showing chemical shifts at various peaks.
$^{13}$C-NMR Spectrum of 4-(Dimethylamino)benzaldehyde (2d)
$^1$H-NMR Spectrum of 4-(Methylthio)benzaldehyde (2e)
$^{13}$C-NMR Spectrum of 4-(Methylthio)benzaldehyde (2e)
$^1$H-NMR Spectrum of 4-(Benzyloxy)-3-methoxybenzaldehyde (2f)

![NMR Spectrum Image]

2f

Chemical shifts and peaks are indicated with numerical values on the spectrum. The spectrum shows characteristic signals for the protons in the molecule, including aromatic protons and hydroxy groups.
\textbf{\textsuperscript{13}C-NMR Spectrum of 4-(Benzyloxy)-3-methoxybenzaldehyde (2f)}
$^1$H-NMR Spectrum of 4-Formylphenyl acetate (2g)
\( ^{13}\text{C-NMR Spectrum of 4-Formylphenyl acetate (2g)} \)
$^1$H-NMR Spectrum of 3-Chlorobenzaldehyde (2h)
$^{13}$C-NMR Spectrum of 3-Chlorobenzaldehyde (2h)
$^1$H-NMR Spectrum of 2-Bromobenzaldehyde (2i)
$^{13}$C-NMR Spectrum of 2-Bromobenzaldehyde (2i)
$^1$H-NMR Spectrum of Methyl 2-formylbenzoate (2j)
$^{13}$C-NMR Spectrum of Methyl 2-formylbenzoate (2j)
\[^{1}\text{H}-\text{NMR Spectrum of (E)-2-Methyl-3-phenylacrylaldehyde (2k)}\]
$^{13}$C-NMR Spectrum of (E)-2-Methyl-3-phenylacrylaldehyde (2k)
$^1$H-NMR Spectrum of 1-Naphthaldehyde (2I)
$^{13}$C-NMR Spectrum of 1-Naphthaldehyde (2I)
$^1$H-NMR Spectrum of Furan-2-carbaldehyde (2m)
$^{13}$C-NMR Spectrum of Furan-2-carbaldehyde (2m)
$^1$H-NMR Spectrum of Heptanal (2n)
$^{13}$C-NMR Spectrum of Heptanal (2n)
$^1$H-NMR Spectrum of 4-Hydroxy-3-methoxybenzaldehyde (2o)
$^{13}$C-NMR Spectrum of 4-Hydroxy-3-methoxybenzaldehyde (2o)
$^1$H-NMR Spectrum of 4-Hydroxybenzaldehyde (2p)
$^{13}$C-NMR Spectrum of 4-Hydroxybenzaldehyde (2p)
$^1$H-NMR Spectrum of Tert-butyl (4-formylphenyl) carbonate (2q)
$^{13}$C-NMR Spectrum of Tert-butyl (4-formylphenyl) carbonate (2q)
$^1$H-NMR Spectrum of 4-((Trimethylsilyl)oxy)benzaldehyde (2r)
$^{13}$C-NMR Spectrum of 4-((Trimethylsilyl)oxy)benzaldehyde (2r)
$^1$H-NMR Spectrum of 4-Methylbenzaldehyde (2s)
$^{13}$C-NMR Spectrum of 4-Methylbenzaldehyde (2s)
$^1$H-NMR Spectrum of Benzophenone (2aa)
13C-NMR Spectrum of Benzophenone (2aa)
$^1$H-NMR Spectrum of $N$-(2-Acetylphenyl)acetamide (2ab)

![NMR Spectrum of N-(2-Acetylphenyl)acetamide (2ab)]
$^{13}$C-NMR Spectrum of $N$-(2-Acetylphenyl)acetamide (2ab)
$^1$H-NMR Spectrum of 1-(4-Chlorophenyl)ethan-1-one (2ac)
$^{13}$C-NMR Spectrum of 1-(4-Chlorophenyl)ethan-1-one (2ac)
$^1$H-NMR Spectrum of 1-(o-Tolyl)ethan-1-one (2ad)
$^{13}$C-NMR Spectrum of 1-(o-Tolyl)ethan-1-one (2ad)
1H-NMR Spectrum of 1-(3-(Trifluoromethyl)phenyl)ethan-1-one (2ae)
$^{13}$C-NMR Spectrum of 1-(3-(Trifluoromethyl)phenyl)ethan-1-one (2ae)
$^1$H-NMR Spectrum of 4-Methylpentan-2-one (2af)
$^{13}$C-NMR Spectrum of 4-Methylpentan-2-one (2af)
1H-NMR Spectrum of 9H-fluoren-9-one (2ag)
13C-NMR Spectrum of 9H-fluoren-9-one (2ag)
$^1$H-NMR Spectrum of 1-(Thiophen-2-yl)ethan-1-one (2ah)
\textbf{13C-NMR Spectrum of 1-(Thiophen-2-yl)ethan-1-one (2ah)}
$^1$H-NMR Spectrum of 3,4-Dihydronaphthalen-1(2H)-one (2ai)
$^{13}$C-NMR Spectrum of 3,4-Dihydronaphthalen-1(2H)-one (2ai)
$^1$H-NMR Spectrum of (8R,9S,13S,14S)-3-Hydroxy-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (2aj)
\(^{13}\text{C-NMR Spectrum of (8R,9S,13S,14S)-3-Hydroxy-13-methyl-6,7,8,9,11,12,13,14,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (2aj)}}
^{1}H-NMR Spectrum of 1,2-Di-\textit{p}-tolyldisulfane (3s)
$^{13}$C-NMR Spectrum of 1,2-Di-$p$-tolyl disulfane (3s)