Electronic Supplementary Information (ESI)
for molecules

K$_2$S$_2$O$_8$-Promoted Aryl Thioamides Synthesis from Aryl Aldehydes Using Thiourea as the Sulfur Source

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Part I. Experimental Section

1. Table S1. Screening of Various Solvents

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NMP</td>
<td>&lt;5%</td>
</tr>
<tr>
<td>2</td>
<td>1,4-dioxane</td>
<td>trace</td>
</tr>
<tr>
<td>3</td>
<td>DCE</td>
<td>trace</td>
</tr>
<tr>
<td>4</td>
<td>toluene</td>
<td>0%</td>
</tr>
<tr>
<td>5</td>
<td>PhCl</td>
<td>0%</td>
</tr>
<tr>
<td>6</td>
<td>DMSO</td>
<td>0%</td>
</tr>
<tr>
<td>7</td>
<td>HOCH₂CH₂OH</td>
<td>0%</td>
</tr>
</tbody>
</table>

<sup>a</sup>Conditions: 1a (0.25 mmol), 2 (1.25 mmol), K₂S₂O₈ (0.5 mmol), thiourea (0.5 mmol), H₂O (0.5 mL), Py (1.25 mmol), 125°C for 24 h in solvent (1.5 mL), unless otherwise noted. <sup>b</sup>Isolated yield.

2. Three control experiments for mechanism study.

<table>
<thead>
<tr>
<th>(0.25 mmol)</th>
<th>H₂O (0.5 mL)</th>
<th>Py (5 equiv), 125 °C</th>
<th>Yield (%)&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenyl acetic acid</td>
<td>K₂S₂O₈ (2 equiv)</td>
<td>&lt;5%</td>
<td></td>
</tr>
<tr>
<td>(1.5 mL)</td>
<td>H₂O (0.5 mL)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>(0.25 mmol)</th>
<th>H₂O (0.5 mL)</th>
<th>Py (5 equiv), 125 °C</th>
<th>Yield (%)&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenyl isocyanate</td>
<td>K₂S₂O₈ (2 equiv)</td>
<td>0%</td>
<td></td>
</tr>
<tr>
<td>(1.5 mL)</td>
<td>CS(NH₂)₂ (2 equiv)</td>
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<td></td>
</tr>
</tbody>
</table>
Part II $^1$H NMR and $^{13}$C NMR

$^1$H NMR of 4-chloro-$N$, $N$-dimethylbenzothioamide 3a
$^1$H NMR of $N,N,4$-trimethylbenzothioamide $3c$
$^1$H NMR of 4-methoxy-N, N-dimethylbenzothioamide 3d
$^{13}$C NMR of 4-methoxy-$N$, $N$-dimethylbenzothioamide 3d
$^1$H NMR of 4-bromo-$N, N$-dimethylbenzothioamide 3e
\(^1\)H NMR of 4-fluoro-\(N, N\)-dimethylbenzothioamide 3f
$^{13}$C NMR of 4-fluoro-$N$, $N$-dimethylbenzothioamide 3f
$^1$H NMR of 3-chloro-$N, N$-dimethylbenzothioamide 3g
$^1$H NMR of 4-(trifluoromethyl)-$N$, $N$-dimethylbenzothioamide 3j
$^1$H NMR of 4-tert-butyl-$N,N$-dimethylbenzothioamide 3k
$^{13}$C NMR of 4-tert-butyl-$N$, $N$-dimethylbenzothioamide 3k
$^1H$ NMR of 3,5-di-tert-butyl-$N$, $N$-dimethylbenzothioamide 3l
$^{13}$C NMR of 3,5-di-tert-butyl-$N,N$-dimethylbenzothioamide 31
$^1$H NMR of 4-benzyl-$N, N$-dimethylbenzothioamide 3n
$^{13}$C NMR of 4-benzyl-$N, N$-dimethylbenzothioamide 3n
Supplementary Material (ESI) for *molecules*

$^1$H NMR of 4-hydroxy-$N$, $N$-dimethylbenzothioamide 3o

![NMR spectrum of 4-hydroxy-$N$, $N$-dimethylbenzothioamide 3o](image)
$^1$H NMR of 4-(dimethylamino)-N, N-dimethylbenzothioamide 3p
$^{13}$C NMR of 4-(dimethylamino)-$N$, $N$-dimethylbenzothioamide 3p
$^1$H NMR of $N, N$-dimethylnaphthalene-2-carbothioamide $3q$
$^{13}$C NMR of $N, N$-dimethylnaphthalene-2-carbothioamide 3q
$^1$H NMR of $N$, $N$-dimethylfuran-2-carbothioamide 3s
$^{13}$C NMR of $N$, $N$-dimethylfuran-2-carbothioamide 3s
$^1$H NMR of N, N-dimethylthiophene-2-carbothioamide 3t
$^{13}$C NMR of $N, N$-dimethylthiophene-2-carbothioamide 3t
$^1$H NMR of $N, N$-dimethylpyridine-3-carbothioamide 3u
$^{13}$C NMR of $N, N$-dimethylpyridine-3-carbothioamide 3u
$^1$H NMR of N,N-dimethyl-5-(quinolin-2-yl)thiophene-2-carbothioamide 3v
$^{13}$C NMR of $N, N$-dimethyl-5-(quinolin-2-yl)thiophene-2-carbothioamide 3v