Supporting Information

D-Glucose as Green Ligand for Selective Copper catalyzed Phenol Formation from Aryl Halides with an Easy Catalyst Removal

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General Experimental Procedures

All the reactions were carried out in 15-20 mL reaction tube under normal atmospheric air. Commercially available D-glucose and other carbohydrates are purchased from Aldrich chemicals. Copper(II) acetate monohydrate along with other copper salts and halobenzenes are purchased from Aldrich chemicals, Alfa Aesar, SRL (India) and Awra chemicals (India). Potassium hydroxide purchased from Ranbaxy (India). Dimethylsulphoxide was purchased from SRL (India) and all these reagents were used without further purification. Reaction temperatures were controlled by Varivolt temperature modulator, melting points were determined using a Guna 230 volts apparatus. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 precoated plates (0.25 mm) and visualized by UV fluorescence lamp. Silica gel (particle size 100-200 mesh) purchased from SRL (India), was used for chromatography. \(^1\)H NMR and \(^13\)C NMR spectra were recorded at 20°C on a Bruker 400 MHz instrument. Spectra were reported relative to Me\(_4\)Si (δ 0.0 ppm) or CHCl\(_3\) residual peak (δ 7.26 ppm). \(^13\)C 100 MHz NMR were reported relative to CDCl\(_3\) (δ 77.16 ppm). All the products were characterized by their NMR, GC/MS and HRMS spectra. The first-order peak patterns are indicated as s (singlet), d (doublet), dd (doublet of doublet), t (triplet), q (quadruplet). Complex non-first-order signals are indicated as m (multiplet). FTIR spectra were recorded on a Nicolet 6700 spectrometer and are reported in frequency of absorption (cm\(^{-1}\)). High resolution mass spectroscopy (HRMS) was recorded on Q-Tof Micro mass spectrometer and GCMS recorded on JEOL GCMATE II mass spectrometer.

General Procedure for Hydroxylation of Aryl Halides (0.5 mmol scale) Provided in Table 2.

\[
\begin{align*}
\begin{array}{c}
\text{Cu(OAc)}_2 \cdot \text{H}_2\text{O} (5 \text{ mol} \%) \\
\text{L1} (5 \text{ mol} \%)
\end{array}
\end{align*}
\]

Aryl halide (0.5 mmol), Cu(OAc)\(_2\)\cdot H\(_2\)O (5 mg, 0.025 mmol) and D-glucose (4.5 mg, 0.025 mmol) were taken in a 15 mL reaction tube. 2 mL of 1:1 mixture of DMSO and
H$_2$O was added to it followed by KOH pallet (112-228 mg, 2-4 mmol) at room temperature (in case of liquid aryl halides, 0.5 mmol of aryl halide was added after the addition of solvent). Then the reaction tube was fitted with a glass stopper and shielded with Teflon tape. The reaction mixture was heated at 120 °C (while heating the reaction mixture, colour of the reaction mixture is changing from blue colour to reddish brown) with occasional mechanical shaking (otherwise KOH sticks to glass wall of the reaction tube). After complete disappearance of aryl halide (the reaction was monitored by TLC), the reaction mixture was allowed to cool to room temperature. Then the reaction mixture was neutralized with few drops of aq. HCl (6 M HCl). In case of water soluble phenols, the solvent was evaporated and the crude residue was purified by column chromatography on silica gel using ethyl acetate/hexanes as eluent to afford the corresponding phenol. In case of water insoluble phenols, the purification was done after water workup.

General Procedure for Hydroxylation of Aryl Halides (10 mmol scale) Provided in Table 2.

In a 100 ml round bottom flask, Cu(OAc)$_2$ . H$_2$O (0.5 mmol, 99.5 mg) and D-glucose (0.5 mmol, 90 mg) were taken. 20 mL DMSO and 20 mL H$_2$O were added to it. Aryl halide (10 mmol) and KOH (60 mmol, 3.36 gm) were added to the reaction mixture. Then the reaction mixture was heated at 120 °C and the reaction was monitored using TLC. After the completion, the reaction was allowed to cool to room temperature. The reaction mixture was neutralized with aq. HCl (2 M-6 M HCl), then the phenol was extracted from water layer to organic layer by using dichloromethane as solvent. The further purification of phenol was done by column chromatography on silica gel using ethyl acetate/hexanes as the eluent.
Table 1. Hydroxylation of PhI using various copper sources, solvent ratios, bases and temperature.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Cu/L1 (mol %)</th>
<th>DMSO/H2O</th>
<th>Temp. (°C)</th>
<th>Yield(%) a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CuI/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>99</td>
</tr>
<tr>
<td>2</td>
<td>Cu(OAc)₂·H₂O/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>99</td>
</tr>
<tr>
<td>3</td>
<td>CuBr/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>70</td>
</tr>
<tr>
<td>4</td>
<td>CuCl/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>55</td>
</tr>
<tr>
<td>5</td>
<td>CuSO₄·H₂O/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>72</td>
</tr>
<tr>
<td>6</td>
<td>CuCl₂·2H₂O/L1 (20/40)</td>
<td>1:1</td>
<td>120</td>
<td>86</td>
</tr>
<tr>
<td>7</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>1:1</td>
<td>120</td>
<td>99/0 b</td>
</tr>
<tr>
<td>8</td>
<td>Cu₂O/L1 (5/5)</td>
<td>1:1</td>
<td>120</td>
<td>20</td>
</tr>
<tr>
<td>9</td>
<td>Cu(OAc)₂·H₂O/L1 (5/10)</td>
<td>1:1</td>
<td>120</td>
<td>99</td>
</tr>
<tr>
<td>10</td>
<td>Cu(OAc)₂·H₂O/L1 (10/10)</td>
<td>1:1</td>
<td>120</td>
<td>85</td>
</tr>
<tr>
<td>11</td>
<td>Cu(OAc)₂·H₂O/L1 (10/20)</td>
<td>1:1</td>
<td>120</td>
<td>75</td>
</tr>
<tr>
<td>12</td>
<td>Cu(OAc)₂·H₂O/L1 (20/20)</td>
<td>1:1</td>
<td>120</td>
<td>87</td>
</tr>
<tr>
<td>13</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>1:9</td>
<td>120</td>
<td>60</td>
</tr>
<tr>
<td>14</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>2:8</td>
<td>120</td>
<td>70</td>
</tr>
<tr>
<td>15</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>3:7</td>
<td>120</td>
<td>82</td>
</tr>
<tr>
<td>16</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>4:6</td>
<td>120</td>
<td>87</td>
</tr>
<tr>
<td>17</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>H₂O</td>
<td>120</td>
<td>25(48h)</td>
</tr>
<tr>
<td>18</td>
<td>Cu(OAc)₂·H₂O/L1 (20/40)</td>
<td>1:1</td>
<td>100</td>
<td>99</td>
</tr>
<tr>
<td>19</td>
<td>Cu(OAc)₂·H₂O/L1 (20/40)</td>
<td>1:1</td>
<td>80</td>
<td>50</td>
</tr>
<tr>
<td>20</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>1:1</td>
<td>100</td>
<td>55</td>
</tr>
<tr>
<td>21</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>1:1</td>
<td>120</td>
<td>92(34h)c</td>
</tr>
<tr>
<td>22</td>
<td>Cu(OAc)₂·H₂O/L1 (5/5)</td>
<td>1:1</td>
<td>120</td>
<td>40(34h)d</td>
</tr>
</tbody>
</table>

a Isolated yield. b Reaction without ligand. c CsOH is the base. d NaOH is the base
**Table 2.** Competitive reaction study of hydroxylation of aryl halide in presence of external nucleophiles.\(^a\)

\[
\text{I} + \text{NuH} \quad \text{Cu(OAc)}_2 \cdot \text{H}_2\text{O} - \text{L1 (5 mol \%)} \quad \text{DMSO/H}_2\text{O (1:1), 120 °C, 24 h}} \quad \text{KOH (8 equiv.)} \quad \text{OH} \quad + \text{other coupling product}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Nucleophile (1.5 equiv.)(^b)</th>
<th>Yield of phenol</th>
<th>Other coupling product</th>
</tr>
</thead>
</table>
| 1     | \[
\text{OH}
\]
| 90    | trace                          |                 |
| 2     | \[
\text{OH}
\]
| 94    | 0                              |                 |
| 3     | \[
\text{OH}
\]
| 95    | 0                              |                 |
| 4     | \[
\text{NH}
\]
| 98    | 0                              |                 |

\(^a\) PhI (0.5 mmol), Cu(OAc)\(_2\), H\(_2\)O (0.025 mmol), D-glucose (0.025 mmol), KOH (4 mmol) in 2 mL DMSO/H\(_2\)O at 120 °C. 0.75 mmol of nucleophiles are used unless otherwise mentioned. \(^b\) 10 Equivalents of ethanol (5 mmol) was used.
Characterization data

![Phenol](image)

**Phenol** (Table 3, entry 1):

Following the general method, iodobenzene (56 µL, 0.5 mmol) was reacted with potassium hydroxide (112 mg, 2.0 mmol) to give phenol with 99% yield as colorless liquid with characteristic odor (eluent: ethyl acetate/hexane = 15:85). R_f = 0.28 (in 10% EtOAc in hexane); IR (neat): 1070, 1229, 1369, 1477, 1597, 3339 cm^{-1}; ^1H NMR (400 MHz, CDCl_3) δ 6.38 (s, 1H), 6.82 (td, J = 8 Hz, 1.2 Hz, 2H), 6.90 (dt, J = 8 Hz, 0.8 Hz, 1H), 7.19 (t, J = 8 Hz, 2H); ^13C NMR (100 MHz, CDCl_3) δ 115.5, 121.0, 129.8, 155.3; HRMS [M^+Na] Calculated for C_6H_6ONa: 117.0316; found: 117.0320.

![4-Methoxyphenol](image)

**4-Methoxyphenol** (Table 3, entry 2)

Following the general method, 4-methoxy-1-iodobenzene (117 mg, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give phenol with 99% yield as white solid (eluent: ethyl acetate/hexane = 20:80). MP 55-57 °C. R_f = 0.4 (in 20% EtOAc/Hexane); IR 1031, 1108, 1214, 1337, 1447, 1599, 1718, 2930, 3380 cm^{-1}; ^1H NMR (400 MHz, CDCl_3) δ 3.81 (s, 3H), 5.71 (s, 1H), 6.70-6.84 (m, 4H); ^13C NMR (100 MHz, CDCl_3) δ 56.0, 115.0, 116.2, 149.6, 153.7; HRMS [M^+K] Calculated for C_7H_8O_2K: 163.0161; found: 163.0156.

![3-Methoxyphenol](image)

**3-Methoxyphenol** (Table 3, entry 3)
Following the general method, 3-methoxy-1-iodobenzene (59.5 µL, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give phenol with 99% yield as colorless liquid (eluent: ethyl acetate/hexane = 20:80). $R_f = 0.58$ (in 20% EtOAc/Hexane); IR 1025, 1099, 1202, 1335, 1440, 1575, 1695, 3000, 3407 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 3.77 (s, 3H), 6.37 (s, 1H), 6.46 (d, $J = 2$ Hz, 2H), 6.48 – 6.57 (m, 1H), 7.14 (t, $J = 8$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 55.4, 101.7, 106.6, 108.2, 130.4, 156.7, 160.8; HRMS [M$^{+}+1$] Calculated for C$_7$H$_9$O$_2$: 125.0603; found: 125.0603.

2-Methoxyphenol (Table 3, entry 4)

Following the general method, 2-methoxy-1-iodobenzene (66 µL, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give phenol with 52% yield as colorless liquid (eluent: ethyl acetate/hexane = 20:80). $R_f = 0.75$ (in 20% EtOAc/Hexane); IR 1041, 1154, 1362, 1496, 1603, 1704, 2251, 2954, 3385 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 3.89 (s, 3H), 5.78 (d, $J = 1.2$ Hz, 1H), 6.84-6.95 (m, 3H), 6.95-7.06 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 55.9, 110.8, 114.6, 120.2, 121.5, 145.7, 146.7; GCMS (EI$^+$): $m/z$ =124.

2-Methylphenol (Table 3, entry 5)

Following the general method, 2-methyl-1-iodobenzene (63.6 µL, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 84% yield as colorless liquid (eluent: ethyl acetate/hexane = 20:80). $R_f = 0.78$ (in 20% EtOAc/Hexane); IR 1072, 1123, 1273, 1369, 1462, 1639, 1726, 2068, 2959, 3457 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.21 (s, 3H), 6.72 (d, $J = 8$Hz, 1H), 6.83 (t, $J = 7.6$ Hz,
1H), 7.04 (t, J = 7.6 Hz, 1H), 7.09 (d, J = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 15.8, 115.1, 120.9, 124.1, 127.2, 131.2, 153.6; GCMS (EI⁺) : m/z = 108.

**3,5-dimethylphenol** (Table 3, entry 6)

Following the general method, 3,5-dimethyl-1-iodobenzene (73µL, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 67% yield as colorless liquid (eluent: ethyl acetate/hexane = 15:85). Rᵣ = 0.58 (in 20% EtOAc/Hexane); IR 1026, 1157, 1252, 1312, 1458, 1630, 2057, 2923, 3457 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 2.26 (s, 6H), 4.75 (s, 1H), 6.46 (s, 2H), 6.57 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 21.3, 113.1, 122.5, 139.6, 155.5; HRMS [M⁺+1] Calculated for C₈H₁₁O: 123.0810; found: 123.0809.

**Naphthalene-1-ol** (Table 3, entry 7)

Following the general method, 1-iodonaphthalene (73µL mg, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 82%, as white solid (eluent: ethyl acetate/hexane = 25:75). MP 94-96 °C. Rᵣ = 0.62 (in 20% EtOAc/Hexane); IR 1015, 1044, 1082, 1240, 1270, 1386, 3054, 3309 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 5.41 (s, 1H), 6.79 (s, 1H), 7.25 – 7.39 (m, 1H), 7.39 – 7.59 (m, 3H), 7.81 (s, 1H), 8.18 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 108.8, 120.8, 121.6, 124.4, 125.4, 126.0, 126.6, 127.8, 134.9, 151.4; HRMS [M⁺+1] Calculated for C₁₀H₉O: 145.0653; found: 145.0658.
4-Nitrophenol (Table 3, entries 8, 17 & 22)

Following the general method, 4-nitro-1-iodobenzene or 4-nitro-1-bromobenzene or 4-nitro-1-chlorobenzene (124.5 mg or 101 mg or 78.8 mg, 0.5 mmol) was reacted with potassium hydroxide (112 mg, 2.0 mmol) to give corresponding phenol with 99% yield in all cases as yellow solid (eluent: ethyl acetate/hexane = 25:75). MP 110-115 °C. \( R_f = 0.32 \) (in 20% EtOAc/Hexane); IR 1114, 1176, 1204, 1295, 1342, 1498, 1594, 1613, 1638, 3433 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 6.86 (d, \( J = 8.8 \) Hz, 2H), 8.10 (d, \( J = 9.2 \) Hz, 2H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 115.9, 126.5, 141.5, 161.9; HRMS [M\(^{+1}\)] Calculated for C\(_6\)H\(_6\)NO\(_3\): 140.0348; found: 140.0348.

3-Nitrophenol (Table 3, entry 9)

Following the general method, 3-nitro-1-iodobenzene (124.5 mg, 0.5 mmol) was reacted with potassium hydroxide (112 mg, 2.0 mmol) to give corresponding phenol with 83% yield as yellow solid (eluent: ethyl acetate/hexane = 25:75). MP 96-98 °C. \( R_f = 0.42 \) (in 20% EtOAc/Hexane); IR 1213, 1354, 1525, 1620, 3395 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.20 (dd, \( J = 8 \) Hz, 3.6Hz, 1H), 7.39 (t, \( J = 8 \) Hz, 1H), 7.70 (t, \( J = 2.4 \) Hz, 1H), 7.78 (dd, \( J = 8.4 \) Hz, 2 Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 110.6, 115.8, 122.3, 130.4, 149.1, 156.6; HRMS [M\(^{+1}\)] Calculated for C\(_6\)H\(_6\)NO\(_3\): 140.0348; found: 140.0355.

4-Hydroxyacetophenone (Table 3, entries 10 and 15)

Following the general method, 4-iodoacetophenone or 4-bromoacetophenone (123 mg, or 99.5 mg, 0.5 mmol) was reacted with potassium hydroxide (112 mg, 2.0 mmol) to give...
corresponding phenol with 83% or 69% yield, as white solid (eluent: ethyl acetate/hexane = 30:70). MP 109-111 °C; \( R_f = 0.29 \) (in 20% EtOAc/Hexane); IR 1170, 1190, 1343, 1445, 1508, 1593, 1657, 1708, 2956, 3249 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 2.51 (s, 3H), 6.88 (d, \( J = 8.8 \) Hz, 2H), 7.83 (d, \( J = 8.8 \) Hz, 2H), 8.22 (s, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 26.4, 115.7, 129.5, 131.4, 161.8, 199.0; HRMS \([M+1]^+\) Calculated for C\(_8\)H\(_9\)O\(_2\): 137.0603; found: 137.0606.

![4-Hydroxybenzophenone](image)

**4-Hydroxybenzophenone** (Table 3, entries 11 and 21)

Following the general method, 4-iodobenzophenone or 4-chlorobenzophenone (154 mg or 2.16 gm, 0.5 mmol or 10 mmol) was reacted with potassium hydroxide (168 mg or 3.36 gm, 3.0 mmol or 60 mmol) to give corresponding phenol with 93% or 30% yield as yellow solid (eluent: ethyl acetate/hexane = 25:75). MP 132-135 °C; IR 1025, 1111, 1282, 1371, 1448, 1597, 1645, 1706, 2834, 2946, 3378 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 6.95 (dd, \( J = 8.8, 0.8 \) Hz, 2H), 7.47 (t, \( J = 7.6 \) Hz, 2H), 7.57 (t, \( J = 7.6 \) Hz, 1H), 7.76 (t, \( J = 8 \) Hz, 4H) (phenolic proton merged in aromatic zone); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 115.6, 128.4, 129.4, 130.0, 132.4, 133.3, 138.1, 161.2, 197.3; HRMS \([M+1]^+\) Calculated for C\(_{13}\)H\(_{11}\)O\(_2\): 199.0759; found: 199.0768.

![3-Hydroxybenzophenone](image)

**3-Hydroxybenzophenone** (Table 3, entry 12)

Following the general method, 4-iodobenzophenone (154 mg, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 87% yield as yellow solid (eluent: ethyl acetate/hexane = 25:75). MP 113-115 °C. IR 1024, 1297, 1418, 1452, 1589, 1653, 2834, 2946, 3348 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.08 – 7.15 (m, 1H), 7.21 – 7.33 (m, 2H), 7.36 – 7.48 (m, 3H), 7.52 – 7.60 (m, 1H), 7.74 -
- 7.81 (m, 2H), (phenolic proton merged in aromatic zone); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 116.8, 120.4, 122.7, 128.4, 129.6, 130.3, 132.9, 137.3, 138.6, 156.4, 197.9; HRMS [M$^+$+1] Calculated for C$_{13}$H$_{11}$O$_2$: 199.0759; found: 199.0751.

1,2-dihydroxybenzene (Table 3, entries 13, 14, 18 and 20)

Following the general method, 2-bromophenol or 2-iodophenol or 2-iodo-1-bromobenzene or 1,2-diiodobenzene (53.3 µL or 110 mg or 64.2 µL or 65.3 µL, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 70%, 83%, 83%, 54% yield respectively, as colorless liquid (eluent: ethyl acetate/hexane = 30:70). $R_f$ = 0.44 (in 20% EtOAc/Hexane); IR 1095, 1267, 1361, 1467, 1594, 1703, 3340 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 5.29 (s, 2H), 6.78-6.85 (m, 2H), 6.85-6.92 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 115.7, 121.4, 143.6; HRMS [M+K]$^+$ Calculated for C$_6$H$_6$O$_2$K: 149.0005; found: 149.0010.

4-hydroxybenzonitrile (Table 3, entry 16)

Following the general method, 3-nitro-1-iodobenzene (91 mg, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 99%, as white solid (eluent: ethyl acetate/hexane = 30:70). MP 110-113 °C. $R_f$ = 0.17 (in 20% EtOAc/Hexane); IR 1114, 1176, 1204, 1295, 1342, 1498, 1594, 1613, 1638, 2162, 3433 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) δ 3.60 (s, 1H), 7.69 (d, $J$ = 7.6 Hz, 2H), 7.86 (d, $J$ = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 127.0, 130.1, 131.4, 131.8, 166.8; GCMS (EI$^+$) : m/z =142.
Following the general method, 1,3-diiodobenzene (165 mg, 0.5 mmol) was reacted with potassium hydroxide (224 mg, 4.0 mmol) to give corresponding phenol with 80% yield as colorless liquid (eluent: ethyl acetate/hexane = 25:75). $R_f = 0.52$ (in 20% EtOAc/Hexane); IR 1235, 1462, 1582, 1639, 3467 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.18 (s, 2H), 6.78-6.83 (m, 1H), 6.96 (t, $J = 8$ Hz, 1H), 7.20-7.29 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 115.1, 124.7, 130.1, 131.1; HRMS [M+K]$^+$ Calculated for C$_6$H$_6$O$_2$K: 149.0005; found: 149.0002
Phenol ($^1$H & $^{13}$C)
2-Methoxyphenol ($^1$H & $^{13}$C)
3,5-dimethylphenol ($^1$H & $^{13}$C)
4-Nitrophenol (\(^1\mathrm{H} & \^{13}\mathrm{C}\))

[Spectroscopic data for 4-Nitrophenol (1H & 13C)]

[Spectroscopic data for 4-Nitrophenol (1H & 13C)]
4-Hydroxyacetophenone \(^{1}H \& ^{13}C\)

**Electronic Supplementary Material (ESI) for Chemical Communications**

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1,2-dihydroxybenzene ($^1$H & $^{13}$C)
4-hydroxybenzonitrile ($^1$H & $^{13}$C)

**Bruker**

**Current Data Parameters**

**Hardware:** BNC-907

**Software:** PGS-2001

**Instrument:** DRX-400D

**Acquisition Parameters**

Date: 10/10/2010

Time: 18:02

Frequency: 400.13 MHz

**Processing Parameters**

FT: 400.13 MHz

**Chart E1**

**Chart E2**

**Chart E3**

**Spectrum:**

- **Compound:** 4-hydroxybenzonitrile
- **Dissolution:** Methanol
- **NMR Spectra:**
  - $^1$H NMR
  - $^{13}$C NMR

**Additional Information:**

- **ESI:** Electronic Supplementary Material (ESI) for Chemical Communications
- **Journal:** This journal is © The Royal Society of Chemistry 2011