Supporting Information

Iodine Catalyzed Oxidation of Alcohols and Aldehydes to Carboxylic Acids in Water: A Metal-Free Route to Synthesis of Furandicarboxylic Acid and Terephthalic Acid

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Contents
1. General information .........................................................................................................S3
2. Effect of catalysts on the C-H oxidation (Table S1). .......................................................S4
3. Effect of amount of catalyst loading for the oxidation reaction (Table -S2) ..............S5
4. Effect of oxidant used for the reaction (Table-S3)....................................................S5
5. Effect of amount of oxidant used for oxidation reaction (Table-S4).........................S6
6. Optimization of reaction conditions for oxidation of aldehyde (Table-S5).............S6
7. Effect of base, solvent and temperature used for the reaction (Table- S5)............S7
8. General procedure for oxidation of alcohols and aldehydes.......................................S8
9. Gram scale/ one pot synthesis of FDCA and Terephthalic acid...............................S8-S9
10. References ..................................................................................................................S10
11. Identification of products, \(^{1}H\) and \(^{13}C\{^{1}H\}\) NMR data...............................S11-S17
12. \(^{1}H\) and \(^{13}C\{^{1}H\}\) NMR spectra of prepared compounds...............................S18-S58
General Information

Unless otherwise stated, all reactions were performed under open atmosphere. $^1$H and $^{13}$C NMR spectra were recorded on a Bruker Spectrospin DPX-300 NMR spectrometer at 300 and 75.47 MHz, respectively. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts are related to residual solvent peaks [CDCl$_3$: 7.26 ($^1$H), 77.16 ($^{13}$C); DMSO-d$_6$: 2.50 ($^1$H), 39.52 ($^{13}$C)]. All alcohols and aldehydes were purchased from commercial sources and hydroxymethylfurfural (HMF) was prepared according to literature procedure.$^{[1]}$ Deionised water, TBHP (70% in water), iodine and NaOH were used as received.
**Experimental Section:**

**Table S1:** Effect of catalysts on the C-H oxidation.

![Reaction Scheme](image)

<table>
<thead>
<tr>
<th>Entry No.</th>
<th>Catalyst</th>
<th>% of yield (isolated)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>I₂</td>
<td>90</td>
</tr>
<tr>
<td>3</td>
<td>KI</td>
<td>48</td>
</tr>
<tr>
<td>4</td>
<td>NaI</td>
<td>46</td>
</tr>
<tr>
<td>5</td>
<td>TBAI</td>
<td>15</td>
</tr>
<tr>
<td>6</td>
<td>TBAB</td>
<td>0</td>
</tr>
</tbody>
</table>
Table S2: Effect of amount of catalyst loading for the oxidation reaction.

\[
\text{Table S2: Effect of amount of catalyst loading for the oxidation reaction.}
\]

\[
\begin{array}{ccc}
\text{Entry No.} & \text{Amount of I}_2 (\text{X mol\%}) & \% \text{ of yield (isolated)} \\
1 & - & - \\
2 & 2 & 23 \\
3 & 4 & 35 \\
4 & 5 & 46 \\
5 & 6 & 58 \\
6 & 8 & 63 \\
7 & 10 & 90 \\
8 & 12 & 90 \\
9 & 15 & 90 \\
10 & 20 & 90 \\
\end{array}
\]

Table S3: Effect of oxidant used for the reaction.

\[
\text{Table S3: Effect of oxidant used for the reaction.}
\]

\[
\begin{array}{ccc}
\text{Entry No.} & \text{Oxidant} & \% \text{ of yield (isolated)} \\
1 & - & - \\
2 & \text{aq. TBHP} & 90 \\
3 & \text{O}_2 & 0 \\
4 & \text{H}_2\text{O}_2 & 5 \\
5 & \text{DTBP} & 0 \\
6 & \text{K}_2\text{S}_2\text{O}_8 & 0 \\
7 & \text{NMO} & 0 \\
\end{array}
\]
Table S4: Effect of amount of oxidant used for oxidation reaction

![Chemical reaction diagram](attachment:image.png)

<table>
<thead>
<tr>
<th>Entry No.</th>
<th>aq. TBHP (x equiv.)</th>
<th>% of yield (isolated)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>15</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>32</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>47</td>
</tr>
<tr>
<td>5&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4</td>
<td>90</td>
</tr>
<tr>
<td>6</td>
<td>5</td>
<td>90</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>90</td>
</tr>
</tbody>
</table>

<sup>a</sup> Since the TBHP used was 70% in water, it is equivalent to 2.8 equivalent of pure TBHP.

Table S5: Optimization of reaction conditions for oxidation of aldehyde

![Chemical reaction diagram](attachment:image.png)

<table>
<thead>
<tr>
<th>Entry No.</th>
<th>Iodine (X mol%)</th>
<th>aq. TBHP (Y equiv.)</th>
<th>NaOH (Z mol%)</th>
<th>Reaction temperature/°C</th>
<th>Yield (isolated)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>70</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>1</td>
<td>10</td>
<td>70</td>
<td>58</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>1.5</td>
<td>10</td>
<td>70</td>
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<td>2</td>
<td>10</td>
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<td>7</td>
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<td>4</td>
<td>20</td>
<td>70</td>
<td>95</td>
</tr>
</tbody>
</table>
**Table S6**: Effect of base, solvent and temperature used for the reaction

<table>
<thead>
<tr>
<th>Entry No.</th>
<th>Base (20mol%)</th>
<th>Solvent</th>
<th>Temperature °C</th>
<th>% of yield (isolated)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NaOH</td>
<td>H₂O</td>
<td>RT</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>NaOH</td>
<td>H₂O</td>
<td>50</td>
<td>42</td>
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<tr>
<td>3</td>
<td>NaOH</td>
<td>H₂O</td>
<td>60</td>
<td>65</td>
</tr>
<tr>
<td>4*</td>
<td>NaOH</td>
<td>H₂O</td>
<td>70</td>
<td>90</td>
</tr>
<tr>
<td>5</td>
<td>KOH</td>
<td>H₂O</td>
<td>70</td>
<td>81</td>
</tr>
<tr>
<td>6</td>
<td>Na₂CO₃</td>
<td>H₂O</td>
<td>70</td>
<td>37</td>
</tr>
<tr>
<td>7</td>
<td>K₂CO₃</td>
<td>H₂O</td>
<td>70</td>
<td>31</td>
</tr>
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<td>8</td>
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</tr>
<tr>
<td>9</td>
<td>NaOAc</td>
<td>H₂O</td>
<td>70</td>
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<td>H₂O</td>
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<td>26</td>
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<tr>
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<td>NaOH</td>
<td>DCM</td>
<td>70</td>
<td>0</td>
</tr>
<tr>
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<td>NaOH</td>
<td>1,4 dioxane</td>
<td>70</td>
<td>0</td>
</tr>
<tr>
<td>14</td>
<td>NaOH</td>
<td>CH₃CN</td>
<td>70</td>
<td>31</td>
</tr>
<tr>
<td>15</td>
<td>NaOH</td>
<td>tert-Butanol</td>
<td>70</td>
<td>38</td>
</tr>
</tbody>
</table>

* Reaction was also carried out with 1 equiv. of NaOH at 100 °C with yield of 90%.

**General procedure for oxidation of alcohols**
A 15 ml screw capped vial was charged with a magnetic bead, 5 mmol of alcohol, 0.5 mmol (10 mol%) of I₂ and 20 mmol (4 equiv.) of aq. TBHP (70% in H₂O). Afterwards, 1 mmol (20 mol%) of NaOH with additional 2 mL of deionized water (pH = 10) were added to the reaction mixture and it was heated at 70 °C for 10-16 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. Na₂S₂O₃ solution. The organic layer was dried over anhydrous Na₂SO₄ and after evaporation of the solvent analytically pure carboxylic acids were obtained.[²,³]

**General procedure for oxidation of aldehydes**

A 15 ml screw capped vial was charged with a magnetic bead, 5 mmol of aldehyde, 0.5 mmol (10 mol%) of I₂ and 20 mmol (4 equiv.) of aq. TBHP. Afterwards, 1 mmol (20 mol%) of NaOH with additional 2 mL of deionized water (pH =10) were added to the reaction mixture and it was heated at 70 °C for 10-16 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. Na₂S₂O₃ solution. The organic layer was dried over anhydrous Na₂SO₄ and after evaporation of the solvent analytically pure carboxylic acids were obtained.[²,³]

**Gram scale synthesis of FDCA from HMF**

A 1000 ml of round bottom flask was charged with a magnetic bead, 79.36 mmol (10 gm) of HMF, 15.87 mmol (20 mol%) of I₂ and 634 mmol (8 equiv.) of aq. TBHP. Afterwards, 39.70 mmol (50 mol%) of NaOH with additional 100 mL of H₂O were added to the reaction mixture and it was heated at 70 °C for 36 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. Na₂S₂O₃ solution. The organic layer was dried over anhydrous Na₂SO₄ and after evaporation of the solvent analytically pure FDCA was obtained in 53% yield (6.55 gm).[²,⁴]

**One pot synthesis of FDCA from D-fructose**
To a 150 mL flask equipped with stirrer bars, D-fructose (4.5 g, 25 mmol), isopropyl alcohol (50 mL), and hydrochloric acid (12.5m aqueous solution, 0.1 mL) were added. The reaction flask was heated on an oil bath to 120 °C with stirring. The reaction was stopped after 4 h and the mixture was filtrated to remove insoluble humin by-product. Solvent in the reaction mixture was then evaporated and dried to give the crude HMF product (3.15 gm). Afterwards, I₂ (5 mmol), aq. TBHP (200 mmol, 8 equiv.), NaOH (12.5 mmol) and H₂O (50 mL) were added and the reaction mixture was stirred at 70 °C for 36 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. Na₂S₂O₃ solution. The organic layer was dried over anhydrous Na₂SO₄ and after evaporation of the solvent FDCA was obtained in 41% yield (1.61 gm).

**Gram scale synthesis of Terephthalic acid**

A 100 ml of round bottom flask was charged with a magnetic bead, 14.48 mmol (2 gm) of 1, 4-benzenedimethanol, 2.89 mmol (20 mol%) of I₂ and 115.84 mmol (8 equiv.) of aq. TBHP. Afterwards, 7.24 mmol (50 mol%) of NaOH with additional 10 mL of H₂O were added to the reaction mixture and it was heated at 70 °C for 36 h. Afterwards, the reaction mixture was neutralized by aq. HCl and extracted with EtOAc and aq. Na₂S₂O₃ solution. The organic layer was dried over anhydrous Na₂SO₄ and after evaporation of the solvent analytically pure terephthalic was obtained.[3]

**References:**
Identification of products:
Compound 1

![Structure of Compound 1](image1)

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.62 (br, 1H), 8.05-8.08 (d, J= 9Hz, 2H), 6.93-6.96 (d, J= 9Hz, 2H), 3.88 (s, 3H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 171.51, 164.06, 132.36, 121.61, 113.76, 55.48

Compound 2

![Structure of Compound 2](image2)

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.13 (br, 1H), 8.16 (m, J= 9Hz, 2H), 7.55 (t, 1H), 7.50 (t, 2H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 172.33, 133.82, 130.24, 129.35, 128.50.

Compound 3

![Structure of Compound 3](image3)

$^1$H-NMR (DMSO-d$_6$, 300 MHz, ppm): 13.62 (br, 1H), 8.30-8.32 (d, J= 6Hz, 2H), 8.16-8.19 (d, 2H); $^{13}$C-NMR (DMSO-d$_6$, 75 MHz, ppm): 166.16, 150.36, 136.81, 131.04, 123.79.

Compound 4
\( \text{HO} \text{COOH} \)

\(^1\text{H-NMR}\) (DMSO-\(d_6\), 300 MHz, ppm): 12.43 (br, 1H), 10.24 (s, 1H), 7.82-7.85 (d, J= 9Hz, 2H), 6.84-6.87 (d, J= 9Hz, 2H); \(^{13}\text{C-NMR}\) (DMSO-\(d_6\), 75 MHz, ppm): 167.65, 162.06, 132.00, 121.82, 115.57.

**Compound 5**

\( \text{Me} \text{COOH} \)

\(^1\text{H-NMR}\) (CDCl\(_3\), 300 MHz, ppm): 11.57 (br, 1H), 7.91 (d, J= 9Hz, 2H), 7.17-7.20 (d, J= 9Hz, 2H), 2.34 (s, 3H); \(^{13}\text{C-NMR}\) (CDCl\(_3\), 75 MHz, ppm): 172.55, 144.67, 130.28, 129.22, 126.62, 21.77.

**Compound 6**

\( \text{Cl} \text{COOH} \)

\(^1\text{H-NMR}\) (DMSO-\(d_6\), 300 MHz, ppm): 13.20 (br, 1H), 7.85-7.88 (d, J= 9Hz, 2H), 7.68-7.72 (d, J= 12Hz, 2H); \(^{13}\text{C-NMR}\) (DMSO-\(d_6\), 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

**Compound 7**

\( \text{Br} \text{COOH} \)
$^1$H-NMR (DMSO-$d_6$, 300 MHz, ppm): 13.19 (br, 1H), 7.84-7.87 (d, J= 9Hz, 2H), 7.67-7.71 (d, J= 12Hz, 2H); $^{13}$C-NMR (DMSO-$d_6$, 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

**Compound 8**

![Compound 8](image)

$^1$H-NMR (CDCl$_3$, 300 MHz): 8.13-8.16 (m, 2H), 7.15 (m, 2H); $^{13}$C-NMR (CDCl$_3$, 75 MHz): 171.5, 166.7, 133.2, 125.8 (d, J = 2.6 Hz), 116.1; $^{19}$F-NMR (CDCl$_3$): -104.1 (s, 1F).

**Compound 9**

![Compound 9](image)

$^1$H-NMR (DMSO-$d_6$, 300 MHz, ppm): 13.46 (br, 1H), 8.12-8.15 (d, J= 9Hz, 2H), 7.86-7.89 (d, J= Hz, 2H); $^{13}$C-NMR (DMSO-$d_6$, 75 MHz, ppm): 167.05, 132.14, 131.74, 130.48, 127.32.

**Compound 10**

![Compound 10](image)

$^1$H-NMR (DMSO-$d_6$, 300 MHz, ppm): 13.42 (br, 1H), 8.11-8.14 (d, J= 9Hz, 2H), 7.83-7.86 (d, J= 9Hz, 2H); $^{13}$C-NMR (DMSO-$d_6$, 75 MHz, ppm): 167.15, 137.24, 132.14, 131.74, 118.48, 115.32.
Compound 11

\[
\begin{align*}
\text{H-NMR (CDCl}_3\text{, 300 MHz, ppm): } & 12.76 \text{ (br, 1H), 7.80 (s, 1H), 7.17-7.25 (m, 3H), 2.27 (s, 3H); } \\
\text{\footnotesize{13C-NMR (CDCl}_3\text{, 75 MHz, ppm): 172.96, 138.31, 134.65, 130.75, 129.33, 127.44, 21.25.}}
\end{align*}
\]

Compound 12

\[
\begin{align*}
\text{H-NMR (DMSO-d}_6\text{, 300 MHz, ppm): } & 13.87 \text{ (br, 1H), 7.73-8.30 (m, 4H); } \\
\text{\footnotesize{13C-NMR (DMSO-d}_6\text{, 75 MHz, ppm): 166.16, 150.36, 136.81, 131.04, 123.79.}}
\end{align*}
\]

Compound 13

\[
\begin{align*}
\text{H-NMR (DMSO-d}_6\text{, 300 MHz, ppm): } & 13.06 \text{ (br, 1H), 6.90 (s, 2H), 1.98-2.34 (m, 9H); } \\
\text{\footnotesize{13C-NMR (CDCl}_3\text{, 75 MHz, ppm): 171.28, 138.43, 134.33, 128.70, 21.45, 19.50.}}
\end{align*}
\]

Compound 14
$^1$H-NMR (DMSO-d$_6$, 300 MHz, ppm): 13.82 (br, 1H), 7.64(t, 1H), 7.17-7.25 (m, 2H); $^{13}$C-NMR (DMSO-d$_6$, 75 MHz, ppm): 162.56, 161.40, 158.06, 133.54, 112.76.

**Compound 15**

![F.png](attachment:F.png)

$^1$H-NMR (DMSO-d$_6$, 300 MHz, ppm): 13.92 (br, 1H), 7.63(t, 1H), 7.17-7.23 (m, 2H); $^{13}$C-NMR (DMSO-d$_6$, 75 MHz, ppm): 162.66, 161.30, 158.06, 133.44, 112.86.

**Compound 16**

![O.png](attachment:O.png)

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.95 (br, 1H), 7.57 (s, 1H), 7.27 (s, 1H), 6.48 (s, 1H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 163.87, 147.49, 143.80, 120.23, 112.30.

**Compound 17**

![S.png](attachment:S.png)

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 12.16 (br, 1H), 7.82 (d, 1H), 7.56 (d, 1H), 7.06 (s, 1H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 168.12, 135.11, 134.13, 132.89, 128.12.

**Compound 18**

![N.png](attachment:N.png)
$^1$H-NMR (DMSO-$d_6$, 300 MHz, ppm): 13.68 (br, 1H), 8.76-8.68 (d, J= 9Hz, 2H), 7.80-7.82 (d, 2H); $^{13}$C-NMR (DMSO-$d_6$, 75 MHz, ppm): 166.60, 151.30, 138.54, 123.20.

**Compound 19**

\[ \text{HO-OC-CH}_2-\text{COOH} \]

$^1$H-NMR (DMSO–$d_6$, 300 MHz, ppm): 8.04 (s, 4H), 13.30 (br, s, 2H); $^{13}$C-NMR (DMSO–$d_6$, 75 MHz, ppm): 129.91, 134.90, 167.12.

**Compound 20**

\[ \text{HO-OC-}\text{C}_{\text{O}}\text{H}_2\text{C}-\text{COOH} \]

$^1$H-NMR (DMSO–$d_6$, 300 MHz, ppm): 7.30 (s, 2H), $^{13}$C-NMR (DMSO–$d_6$, 75 MHz, ppm): 159.40, 147.53, 118.77.

**Compound 21**

\[ \text{HO-CH}_2\text{COOH} \]

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 7.38-7.27 (m, 5H), 3.66 (s, 2H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 178.01, 133.20, 129.30, 128.6, 127.32, 41.05.

**Compound 22**

\[ \text{CH}_3\text{CH}_2\text{COOH} \]
$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.70 (s, 1H), 2.35 (t, J = 7.4 Hz, 2H), 1.68 (m, 2H), 1.00 (t, J = 7.5 Hz, 3H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 180.00, 36.00, 18.72, 13.35.

**Compound 23**

\[
\text{\begin{center}
\includegraphics[width=0.2\textwidth]{compound_23}
\end{center}}
\]

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.73 (s, 1H), 2.35 (t, J = 7.4 Hz, 2H), 1.68 (m, 2H), 1.00 (m, J = 7.5 Hz, 2H), 0.98 (t, 3H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 180.05, 26.00, 23.78, 18.75, 13.15

**Compound 24**

\[
\text{\begin{center}
\includegraphics[width=0.2\textwidth]{compound_24}
\end{center}}
\]

$^1$H-NMR (CDCl$_3$, 300 MHz, ppm): 11.80 (br, s, 1H), 2.34 (t, J = 7.7 Hz, 2H), 1.61 (m, 2H), 1.29 (m, 8H), 0.88 (m, 3H); $^{13}$C-NMR (CDCl$_3$, 75 MHz, ppm): 176.87, 34.3, 31.9, 29.2, 29.1, 25.9, 22.30, 13.45.

**Compound 25**

\[
\text{\begin{center}
\includegraphics[width=0.2\textwidth]{compound_25}
\end{center}}
\]

$^1$H-NMR (DMSO-d$_6$, 300 MHz, ppm): 11.30 (b, 2H), 2.43 (m, 4H); $^{13}$C-NMR (DMSO-d$_6$, 75 MHz, ppm): 174.09, 29.18

$^1$H- NMR (in CDCl$_3$) spectra of compound 1
$^{13}$C{'H'}- NMR (in CDCl$_3$) spectra of compound 1
$^1$H- NMR (in DMSO-d$_6$) spectra of compound 3
$^{13}$C{H$^1$} NMR (in DMSO-$d_6$) spectra of compound 3
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 4
$^{13}$C{H$^1$} NMR (in DMSO-d$_6$) spectra of compound 4
\(^1\)H- NMR (in CDCl\(_3\)) spectra of compound 5
$^{13}$C\{H\}-NMR (in CDCl$_3$) spectra of compound 5
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 6
$^1$H- NMR (in DMSO-d$_6$) spectra of compound 7
$^{13}$C\{H\}$^1$- NMR (in DMSO-d$_6$) spectra of compound 7
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 9

$^{13}$C{H$^1$}- NMR (in DMSO-$d_6$) spectra of compound 9
$^{19}$F- NMR (in DMSO-$d_6$) spectra of compound 9
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 9
$^{13}$C-$^1$H$^1$- NMR (in DMSO-$d_6$) spectra of compound 10
$^1$H- NMR (in CDCl$_3$) spectra of compound 11
$^{13}$C{H$^1$} NMR (in CDCl$_3$) spectra of compound 11

$^1$H- NMR (in DMSO-d$_6$) spectra of compound 12
$^{1}$H- NMR (in DMSO-d$_6$) spectra of compound 13
$^{13}$C-H-NMR (in DMSO-d$_6$) spectra of compound 13
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 15
$^{13}$C{H$^1$} NMR (in DMSO-d$_6$) spectra of compound 15
$^{19}$F- NMR (in DMSO-$d_6$) spectra of compound 15
$^1$H- NMR (in CDCl$_3$) spectra of compound 16
$^{13}\text{C}\{\text{H}^1\}$-NMR (in CDCl$_3$) spectra of compound 16
$^1$H- NMR (in CDCl$_3$) spectra of compound 17
$^{13}$C {$^1$H$^1$}-NMR (in CDCl$_3$) spectra of compound 17
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 18
$^{13}$C-$^1$H- NMR (in DMSO-d$_6$) spectra of compound 18
$^1$H-NMR (in DMSO-d$_6$) spectra of compound 19
$^1$H- NMR (in DMSO-\textit{d}_6) spectra of compound 20
$^{13}$C{H$^1$} - NMR (in DMSO-d$_6$) spectra of compound 20
$^1$H- NMR (in DMSO-$d_6$) spectra of compound 21
$^{13}$C{H$^1$}- NMR (in DMSO-d$_6$) spectra of compound 21
$^{13}$C{$^{1}$H} - NMR (in CDCl3) spectra of compound 24
\(^1\)H- NMR (in DMSO-d\(_6\)) spectra of compound 25
$^{13}$C\textsuperscript{H$^1$} - NMR (in CDCl$_3$) spectra of compound 25
$^1$H- NMR (in CDCl$_3$) spectra of HMF
$^{13}$C\{H\} - NMR (in CDCl$_3$) spectra of HMF