Studies on cyclization reactions of
3-amino-2,4-dihydroxybutanoic acid derivatives

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Copies of $^1$H and $^{13}$C NMR spectra of compounds (4-20) ..................................................S2

Crystallographic data of compounds (15a, 17a, 20a) ..............................................................S22

Copies of $^1$H and $^{13}$C NMR spectra
Compound 4a/4b

Compound 4a/4b, 600 MHz, CDCl₃

Compound 4a/4b, 62.9 MHz, CDCl₃

S2
Compound 5a/5b

Compound 5a/5b, 250 MHz, CDCl₃

Compound 5a/5b, 62.9 MHz, CDCl₃
Compound 6a

Compound 6a, 360 MHz, CDCl₃

Compound 6a, 90.6 MHz, CDCl₃
Compound 6b

Compound 6b, 250 MHz, CDCl₃

Compound 6b, 62.9 MHz, CDCl₃
Compound 8a

Compound 8a, 250 MHz, CDCl$_3$

![NMR spectrum of Compound 8a, 250 MHz, CDCl$_3$](image)

Compound 8a, 62.9 MHz, CDCl$_3$

![NMR spectrum of Compound 8a, 62.9 MHz, CDCl$_3$](image)
Compound 9a/9b

Compound 9a/9b, 300 MHz, CDCl$_3$

\[
\begin{align*}
\text{TsO} & \quad \text{NH} \\
& \quad \text{O} \\
\text{Boc} & \quad \text{OMe} \\
& \quad \text{OTBS}
\end{align*}
\]

Compound 9a/9b, 90.6 MHz, CDCl$_3$

\[
\begin{align*}
\text{TsO} & \quad \text{NH} \\
& \quad \text{O} \\
\text{Boc} & \quad \text{OMe} \\
& \quad \text{OTBS}
\end{align*}
\]
Compound 10a/10b, 250 MHz, CDCl$_3$

Compound 10a/10b, 62.9 MHz, CDCl$_3$
Compound 11a/11b

Compound 11a/11b, 360 MHz, CDCl₃

Compound 11a/11b, 62.9 MHz, CDCl₃
Compound 12a/12b

Compound 12a/12b, 360 MHz, CDCl₃

Compound 12a/12b, 90.6 MHz, CDCl₃
Compound 13a/13b

Compound 13a/13b, 300 MHz, CDCl₃

Compound 13a/13b, 90.6 MHz, CDCl₃
Compound 14a/14b

Compound 14a/14b, 360 MHz, CDCl$_3$

Compound 14a/14b, 90.6 MHz, CDCl$_3$
Compound 15a

Compound 15a, 360 MHz, CDCl₃

Compound 15a, 90.6 MHz, CDCl₃

S14
Compound 15b
Compound **16a/16b**

**Compound 16a/16b, 250 MHz, CDCl₃**

![NMR spectrum of compound 16a/16b, 250 MHz, CDCl₃](image)

**Compound 16a/16b, 75.5 MHz, CDCl₃**

![NMR spectrum of compound 16a/16b, 75.5 MHz, CDCl₃](image)
Compound 17a

Compound 17a, 360 MHz, CDCl₃

\[
\begin{align*}
\text{HO-} & \quad \text{NPhthO} \\
& \quad \text{O} \quad \text{OMe} \\
& \quad \text{OTBS}
\end{align*}
\]

Compound 17a, 90.6 MHz, CDCl₃

\[
\begin{align*}
\text{HO-} & \quad \text{NPhthO} \\
& \quad \text{O} \quad \text{OMe} \\
& \quad \text{OTBS}
\end{align*}
\]
Compound 17b

Compound 17b, 360 MHz, CDCl$_3$

Compound 17b, 90.6 MHz, CDCl$_3$
Compound 19a

Compound 19a, 360 MHz, CDCl$_3$

\[
\text{MsO} \quad \text{NPhthO} \quad \text{OMe} \\
\text{OH}
\]

Compound 19a, 90.6 MHz, CDCl$_3$

\[
\text{MsO} \quad \text{NPhthO} \quad \text{OMe} \\
\text{OH}
\]
Compound 20a

Compound 20a, 360 MHz, DMSO-$d_6$

Compound 20a, 90.6 MHz, DMSO-$d_6$
Crystallographic Data

X-ray diffraction data for compounds 15a, 17a and 20a were collected by using a VENTURE PHOTON100 CMOS Bruker diffractometer with Micro-focus LuS source MoKα radiation. Crystals were mounted on a CryoLoop (Hampton Research) with Paratone-N (Hampton Research) as cryoprotectant and then flash-frozen in a nitrogen gas stream at 100 K. The temperature of the crystals was maintained at the selected value (100 K) by means of a N-Helix device cooling within an accuracy of ±1 K. The data were corrected for Lorentz polarization and absorption effects. The structures were solved by direct methods using SHELXS-97\(^1\) and refined against \(F^2\) by full-matrix least-squares techniques using SHELXL-2016\(^2\) with anisotropic displacement parameters for all non-hydrogen atoms. Hydrogen atoms were located on a difference Fourier map and introduced into the calculations as a riding model with isotropic thermal parameters. All calculations were performed by using the Crystal Structure crystallographic software package WINGX.\(^3\)

ORTEP drawings are shown in Figures S1, S2 and S3. The crystal data collection and refinement parameters are given in Table S1. CCDC 1520833, 1520834 and 1520835 contain the supplementary crystallographic data for this study. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via [http://www.ccdc.cam.ac.uk/Community/Requestastructure](http://www.ccdc.cam.ac.uk/Community/Requestastructure).

![ORTEP drawing of compound 15a](image)

**Fig. S1.** An ORTEP drawing of compound 15a. Thermal ellipsoids are shown at the 30% level.

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Fig. S2. An ORTEP drawing of compound 17a. Thermal ellipsoids are shown at the 30% level.

Fig. S3. An ORTEP drawing of compound 20a. Thermal ellipsoids are shown at the 30% level.
Table S1. Crystallographic data and structure refinement details.

<table>
<thead>
<tr>
<th>Compound</th>
<th>15a</th>
<th>17a</th>
<th>20a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C_{10}H_{13}N_{7}O_{7}</td>
<td>C_{19}H_{27}N_{6}O_{6}Si</td>
<td>C_{12}H_{9}N_{5}O_{4}</td>
</tr>
<tr>
<td>M,</td>
<td>259.21</td>
<td>393.50</td>
<td>247.20</td>
</tr>
<tr>
<td>Crystal size, mm</td>
<td>0.09 × 0.05 × 0.01</td>
<td>0.12 × 0.11 × 0.08</td>
<td>0.13 × 0.07 × 0.01</td>
</tr>
<tr>
<td>Crystal system</td>
<td>monoclinic</td>
<td>monoclinic</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 2₁</td>
<td>P 2₁</td>
<td>P 2₁</td>
</tr>
<tr>
<td>a, Å</td>
<td>6.0454(6)</td>
<td>8.1774(4)</td>
<td>8.2084(14)</td>
</tr>
<tr>
<td>b, Å</td>
<td>14.0347(14)</td>
<td>8.2158(3)</td>
<td>5.2477(8)</td>
</tr>
<tr>
<td>c, Å</td>
<td>7.1416(8)</td>
<td>15.0345(7)</td>
<td>24.351(4)</td>
</tr>
<tr>
<td>α, °</td>
<td>90</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>β, °</td>
<td>101.033(3)</td>
<td>98.231(2)</td>
<td>90.768(5)</td>
</tr>
<tr>
<td>γ, °</td>
<td>90</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Cell volume, Å³</td>
<td>594.73(11)</td>
<td>999.67(8)</td>
<td>1048.8(3)</td>
</tr>
<tr>
<td>Z ; Z’</td>
<td>2 ; 1</td>
<td>2 ; 1</td>
<td>4 ; 2</td>
</tr>
<tr>
<td>T, K</td>
<td>100(1)</td>
<td>100(1)</td>
<td>100(1)</td>
</tr>
<tr>
<td>Radiation type ; wavelength Å</td>
<td>MoKα ; 0.71073</td>
<td>MoKα ; 0.71073</td>
<td>MoKα ; 0.71073</td>
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<tr>
<td>F₀₀₀</td>
<td>272</td>
<td>420</td>
<td>512</td>
</tr>
<tr>
<td>μ, mm⁻¹</td>
<td>0.124</td>
<td>0.152</td>
<td>0.124</td>
</tr>
<tr>
<td>θ range, °</td>
<td>2.903 - 30.409</td>
<td>2.517 - 30.554</td>
<td>2.481 - 26.435</td>
</tr>
<tr>
<td>Reflection collected</td>
<td>20 042</td>
<td>42 103</td>
<td>20 958</td>
</tr>
<tr>
<td>Reflections unique</td>
<td>3 515</td>
<td>6 114</td>
<td>4 289</td>
</tr>
<tr>
<td>R int</td>
<td>0.0943</td>
<td>0.0235</td>
<td>0.0587</td>
</tr>
<tr>
<td>GOF</td>
<td>1.062</td>
<td>1.055</td>
<td>1.159</td>
</tr>
<tr>
<td>Refl. obs. (I &gt; 2σ(I))</td>
<td>2 242</td>
<td>5 766</td>
<td>3 895</td>
</tr>
<tr>
<td>Parameters</td>
<td>166</td>
<td>252</td>
<td>202</td>
</tr>
<tr>
<td>Flack parameter</td>
<td>0.1(4)</td>
<td>0.03(2)</td>
<td>0.9(6)</td>
</tr>
<tr>
<td>wR² (all data)</td>
<td>0.3029</td>
<td>0.0635</td>
<td>0.3717</td>
</tr>
<tr>
<td>R value (I &gt; 2σ(I))</td>
<td>0.1101</td>
<td>0.0254</td>
<td>0.1518</td>
</tr>
<tr>
<td>Largest diff. peak and hole (e⁻Å³)</td>
<td>1.060 ; −0.377</td>
<td>0.260 ; −0.226</td>
<td>1.245 ; −0.734</td>
</tr>
</tbody>
</table>