Crystal and Molecular Structures of 2'-Deoxy-2'-(S)-phenylsulfinyluridine Hemihydrate

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Oxidation of 2'-phenylsulfide prepared from 3',5'-di-O-acetyl-2',2'-cyclouridine gave two diastereoisomers, the minor compound of which is 2'-deoxy-2'-(R)-phenylsulfinyluridine. This paper presents the results of an X-ray analysis of the major one.

Colorless needles were grown by the slow evaporation of an aqueous solution. Intensity data were measured with the ω-2θ scan mode on a diffractometer using graphite monochromated Cu Kα radiation. The size of the crystal used was 0.1×0.2×0.5 mm, but no correction was carried out regarding absorption. A hydrate water is located on the two-fold rotation axis and refined with

![Chemical structure](image)

**Table 1 Crystal and experimental data**

<table>
<thead>
<tr>
<th>Formula: C₁₅H₁₂N₄O₄S·1/2H₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula weight: 361.3</td>
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<tr>
<td>Crystal system: orthorhombic</td>
</tr>
<tr>
<td>Space group: P2₁2₁2</td>
</tr>
<tr>
<td>a = 14.40(2) Å</td>
</tr>
<tr>
<td>b = 13.08(1) Å</td>
</tr>
<tr>
<td>c = 8.35(1) Å</td>
</tr>
<tr>
<td>V = 1573.4(2) Å³</td>
</tr>
<tr>
<td>D, = 1.52 g/cm³</td>
</tr>
<tr>
<td>R = 0.051</td>
</tr>
<tr>
<td>No. of reflections used: 1534</td>
</tr>
<tr>
<td>Measurement: Rigaku AFC-5R</td>
</tr>
<tr>
<td>Program system: DIRECT-SEARCH system</td>
</tr>
<tr>
<td>Structure determination: MULTAN78</td>
</tr>
<tr>
<td>Refinement: block-diagonal least-squares</td>
</tr>
</tbody>
</table>

**Table 2 Final coordinates (×10⁴) of non-H atoms**

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<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Beq Å²</th>
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<td>3067(3)</td>
<td>3727(5)</td>
<td>1.47(10)</td>
</tr>
<tr>
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<td>3267(4)</td>
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<tr>
<td>O(1')</td>
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<td>5936(4)</td>
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<tr>
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<td>532(5)</td>
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<td>2707(6)</td>
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<td>4914(7)</td>
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Beq = (4/3)ΣΣBᵢαᵢβᵢαᵢ
isotropic temperature factors. The positions of the hydrogen atoms were determined from a difference Fourier synthesis and refined isotropically. The crystal and experimental data are given in Table 1.

The molecular structure is shown in Fig. 2, together with an atomic numbering scheme; the bond distances and angles are listed in Table 3. The relative configuration of sulfoxide is $S$, differing from $R$ in the minor compound. The puckering of the sugar ring is C2'-endo-C3'-exo. The C(2') and C(3') atoms are displaced from the plane containing the remaining atoms (O(1'), C(1'), and C(4')) by 0.29(1) and $-0.23(1)$ Å, respectively. The glycosidic torsional angle of O(1')-C(1')-N(1)-C(6) is 81.0(6)$^\circ$ in the anti region, while the minor compound have a syn glycosidic conformation. The torsional angles (O(5')-C(5')-C(4')-O(1') and O(5')-C(5')-C(4')-C(3')) are $-71.0(6)$ and $50.2(6)^\circ$, indicating a gauche-gauche conformation about the C(4')-C(5') bond.

Reference


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