The *X*-Ray Crystal and Molecular Structure of the Nucleoside β-Uridine

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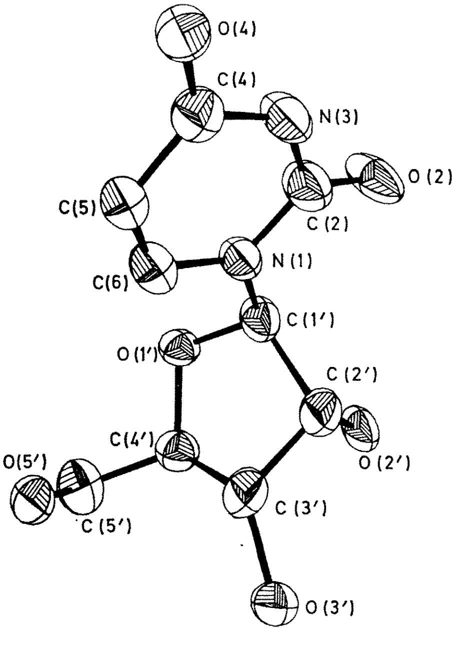
Summary The molecular conformation of β-uridine is within the range of the molecular conformation for several heavy-atom uridine structures previously reported.

RECENT determinations of the structures of halogenated derivatives of the nucleoside β-uridine have shown wide variation in molecular conformation even within crystals containing more than one independent molecule. This wide variation suggests that there is no one preferential conformation, but rather a broad permissible range. We present evidence that this conformational range may be too broad for the native compound, β-uridine.

Crystal data: the crystals belong to the space group $P2_1$ with two molecules per asymmetric unit, $a = 4{\cdot}974$, $b = 14{\cdot}631$, $c = 13{\cdot}947$ Å, and $\beta = 95{\cdot}76°$. Intensity data were collected on a Picker automatic diffractometer with $0° \leqslant 2\theta \leqslant 130°$ using Cu-K_α radiation. This produced 1786 independent reflections. The structure was then solved from phases of 365 normalized structure factors ($E \geqslant 1{\cdot}25$) which were obtained from the Syd Hall tangent formula program. An E synthesis based on the above phases clearly showed the correct structure which refined by means of full-matrix least-squares to an $R = 0{\cdot}076$ for all data greater than $\sin\theta = 0{\cdot}2$. The major problem in refinement is that the data suffer from secondary extinction at low $\sin\theta$ which produces an incorrect scale factor; better data are being retaken.

The conformation of both molecules in the asymmetric unit is essentially the same and can be summarized as follows: no atom in either molecule which forms part of the pyrimidine ring, nor any atom which is directly bonded to it, is removed from a least-squares plane through the ring by more than 0·07 Å. The glycosidic torsion angles χ, as defined by Sundaralingam,[1] are 16·8 and 23·8° for molecules 1 and 2, respectively. The conformation for both molecules is thus *anti* with $-90° \leqslant \chi \leqslant 90°$. The ribose conformation is C(3′) *endo*, and is displaced 0·42 and 0·59 Å from the three-atom plane [C(4′)–O(1′)–C(1′)] in molecules 1 and 2. In both molecules, the orientation of the C(5′)–O(5′) bond with respect to both the C(4′)–C(3′) bond and the C(4′)–O(1′) bond is *gauche*. This *gauche–gauche* conformation is the most common one for uridine structures (see Figure).



FIGURE

The conformation of three derivative structures is summarized as follows: the glycosidic torsion angles for 5-chlorouridine,[2] 5-bromouridine,[3] and 5-iodouridine[4] (two molecules per asymmetric unit) are 51, 51, (12, 56°), respectively. The C(5′)–O(5′) bond is *gauche–gauche* with respect to the C(4′)–C(3′) and the C(4′)–O(1′) bonds for the chloro-, bromo-, and the first molecule of the iodo-derivative, but *gauche–trans* for the second. The C(2′) or C(3′) carbon is oriented C(2′) *endo*, C(3′) *endo*, [C(2′), C(3′)] *endo* for the above molecules. The deviation of these atoms from the previously discussed three-atom plane is 0·44, 0·49, (0·42, 0·42 Å). The largest deviation from a least-squares plane through the pyrimidine ring of any light-atom bonded to the ring is C(1′) 0·21, C(1′) 0·23, [O(2) 0·12, O(2) 0·13 Å] for the given molecules. We suggest that the variation in conformation of the reported derivative structures is the result of packing forces associated with bulky heavy atoms; while intermolecular packing forces in the native compound do not disrupt the individual conformations.

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