

Table 2. Selected geometric parameters (Å, °)

N1—C2	1.343 (4)	C5—C6	1.493 (6)
C2—C5	1.502 (5)	N2—C11	1.411 (5)
C5—N2	1.274 (5)		
N2—C5—C6	126.5 (3)	C6—C5—C2	117.9 (3)
N2—C5—C2	115.6 (3)		
N1—C2—C5—N2	-178.7 (4)	C13—C14—O1—C17	0.3 (6)

Molecule (I) crystallized in the orthorhombic system; space groups *Cmc*₂₁, *Cmcm* or *C2cm* allowed by the systematic absences. With *Z* = 4, space group *Cmcm* requires that the molecule have either *mm* or *2/m* symmetry; this is not possible and this space group option was rejected. Of the two remaining space groups (both of which require that the molecule have crystallographic *m* symmetry), only the *Cmc*₂₁ choice afforded a solution. The molecule is achiral (has mirror symmetry) and with only C, H, N and O atoms (which have very small anomalous dispersion components) we deemed it very unlikely that a correct determination of the direction of the polar axis in the *Cmc*₂₁ space group would be possible. As a consequence of this decision, we measured one unique octant of data to a θ_{\max} of 27.5° with Mo K α radiation and obtained a reflection/parameter ratio of 9.18. As anticipated, the refinement did not allow a determination of the chiral axis direction.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* and *CELDIM* in *CAD-4-PC Software*. Data reduction: *DATRD2* in *NRCVAX96* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX96* and *SHELXL93* (Sheldrick, 1993). Molecular graphics: *NRCVAX96*, *ORTEPII* (Johnson, 1976), *PLATON* (Spek, 1996a) and *PLUTON* (Spek 1996b). Software used to prepare material for publication: *NRCVAX96*, *SHELXL93* and *WordPerfect* macro *PREPCIF* (Ferguson, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1460). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 890–892

N-(6-Amino-3,4-dihydro-3-methyl-5-nitroso-4-oxypyrimidin-2-yl)glycine–Water (1/2)

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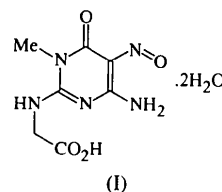
(Received 17 February 1997; accepted 10 March 1997)

Abstract

The title compound, C₇H₉N₅O₄·2H₂O, makes use of the two waters of crystallization to form a hydrogen-bonded network which utilizes all available N—H and O—H groups as hydrogen-bond donors. The shortest intermolecular O—H...O bond is 2.491 (3) Å.

Comment

The title compound, (I), is of potential biological interest since it is an adduct of the amino acid glycine and a substituted uracil nucleobase. In addition, this type of compound can coordinate with transition metal ions such as Cd^{II} and Hg^{II}, whose complexes we are currently investigating.



A view of the asymmetric unit is shown in Fig. 1 and details of the geometry are given in Table 1. The dimensions of the N2—C2—N1—C6—N6 frag-

ment indicate a delocalization of charge with C—N distances in the range 1.311 (3)–1.332 (3) Å. The nitroso bond lengths (values in brackets) differ from those found in 4-amino-5-nitroso-2-methoxy-1-methyl-6-oxo-1,6-dihydropyrimidine (Low *et al.*, 1994), C5—N5 1.316 (3) [1.352 (2) Å] and N5—O5 1.296 (2) [1.266 (2) Å], whereas the bond angle O5—N5—C5 117.6 (2) [117.8 (2)°] is almost identical. The nitroso O5 atom takes part as an acceptor in an intramolecular N—H···O hydrogen bond with the adjacent 6-amino group to form a planar six-membered ring; this conformation is maintained in six-deuterated DMSO solution, as shown by ¹H NMR, where the corresponding signals to both H atoms attached at N6 are well differentiated (chemical shift for H61 and H62 are 10.93 and 8.31 p.p.m., respectively). The mean plane of glycyI atoms C21, C22, O21 and O22 is inclined at 86.0 (1)° to the mean plane of the uracil ring and its substituents (N2, C3, O4, N5, O5, N6). The uracil moiety and atoms N2, C3, O4, N5, O5 and N6 lie in a plane, with atoms N2 and N6 showing the largest deviations from the plane at distances of −0.078 (2) and −0.074 (2) Å, respectively. The largest deviations from the mean plane of the ring atoms themselves are C2 −0.017 (2) and C6 0.022 (2) Å.

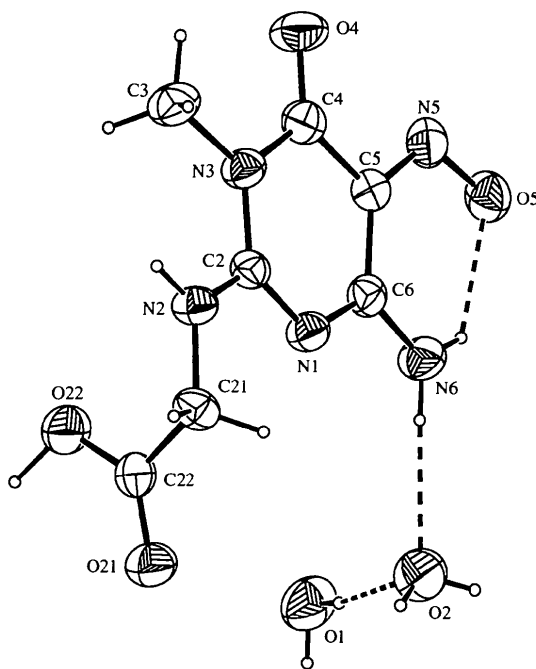


Fig. 1. The molecular structure of the title compound with the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level (ORTEP; Johnson, 1976).

Molecules of the title compound are linked by strong [2.491 (3) Å] O—H···O hydrogen bonds [O22—H22···O5^v 2.491 (3) Å; see Table 3] to form zigzag

chains which extend in the **b** direction. These zigzag chains are linked to adjacent chains to form puckered sheets which run parallel to the **c** axis via O—H···O; O—H···N and N—H···O hydrogen bonds involving three of the four H atoms of the two water molecules. The puckered sheets are linked to form a network via O—H···O hydrogen bonds involving the fourth water H atom [O1—H1A···O2ⁱ, O···O 2.779 (3) Å]. Examination of the structure with PLATON (Spek, 1995a) showed that there are no solvent-accessible voids in the crystal lattice.

Experimental

The title compound was prepared by adding 6-amino-3,4-dihydro-3-methyl-2-methoxy-5-nitroso-4-oxopyrimidine (2.022 g, 10.0 mmol) to a solution of glycine (0.751 g, 10.0 mmol) in 32 ml of 0.31 M aqueous KOH (9.92 mmol). The mixture was stirred at 343 K for 50 min. The wine-red solution was cooled and the pH was adjusted to 3.0 by dropwise addition of glacial acetic acid to neutralize and induce crystallization. After 5 h at room temperature, the orange crystalline solid was collected by filtration and washed successively with water, ethanol and diethyl ether to yield the dihydrate title compound (2.125 g, 7.27 mmol; 81% yield).

Crystal data

C₇H₉N₅O₄·2H₂O
M_r = 263.22
 Orthorhombic
Pbca
a = 9.2739 (13) Å
b = 16.682 (2) Å
c = 14.690 (2) Å
V = 2272.6 (5) Å³
Z = 8
D_x = 1.539 Mg m^{−3}
D_m not measured

Mo *K*α radiation
 λ = 0.7107 Å
 Cell parameters from 25 reflections
 θ = 8.55–14.55°
 μ = 0.134 mm^{−1}
T = 294 (2) K
 Plate
 0.41 × 0.41 × 0.03 mm
 Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 2485 measured reflections
 2485 independent reflections
 1207 reflections with $I > 2\sigma(I)$

θ_{\max} = 27.0°
 $h = 0 \rightarrow 11$
 $k = 0 \rightarrow 21$
 $l = 0 \rightarrow 18$
 3 standard reflections
 frequency: 120 min
 intensity variation: 0.4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.121$
 $S = 0.889$
 2485 reflections
 177 parameters
 H atoms riding: see below
 $w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
 $\Delta\rho_{\max} = 0.246 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.272 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C2	1.323 (3)	N3—C3	1.468 (3)
N1—C6	1.332 (3)	N3—C4	1.381 (3)
C2—N2	1.319 (3)	C4—O4	1.210 (3)
C2—N3	1.376 (3)	C4—C5	1.469 (3)
N2—C21	1.447 (3)	C5—N5	1.316 (3)
C21—C22	1.497 (3)	C5—C6	1.444 (3)
C22—O21	1.207 (3)	N5—O5	1.296 (2)
C22—O22	1.292 (3)	C6—N6	1.311 (3)
C2—N2—C21—C22	−81.3 (3)	C6—C5—N5—O5	0.0 (4)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1A...O2	0.82	1.97	2.779 (3)	169
O1—H1B...O21 ⁱ	0.82	1.99	2.784 (3)	163
N2—H2...O1 ⁱⁱ	0.86	2.09	2.862 (3)	149
O2—H2A...O4 ⁱⁱⁱ	0.82	2.39	2.901 (3)	121
O2—H2A...N5 ⁱⁱⁱ	0.82	2.46	3.228 (3)	158
O2—H2B...O1 ^{iv}	0.82	2.10	2.908 (3)	170
N6—H6A...O2	0.86	2.07	2.914 (3)	169
N6—H6B...O5	0.86	2.02	2.645 (3)	129
N6—H6B...O4 ⁱⁱⁱ	0.86	2.50	2.900 (3)	109
O22—H22...O5 ^v	1.10	1.44	2.491 (3)	155
O22—H22...N5 ^v	1.10	2.08	2.902 (3)	129

Symmetry codes: (i) $x - \frac{1}{2}, y, \frac{1}{2} - z$; (ii) $\frac{1}{2} + x, -\frac{1}{2} - y, 1 - z$; (iii) $\frac{3}{2} - x, -y, z - \frac{1}{2}$; (iv) $\frac{1}{2} + x, y, \frac{1}{2} - z$; (v) $\frac{3}{2} - x, y - \frac{1}{2}, z$.

H atoms were placed in calculated positions, except for H22, which was located in a $\Delta\rho$ map, 1.10 Å from O22, and held in that position. They were treated as riding atoms in the refinement (C—H 0.93–0.98, N—H 0.86, and O—H 0.82 and 1.10 Å).

Data collection: CAD-4-PC (Enraf–Nonius, 1992). Cell refinement: SET4 and CELDIM in CAD-4-PC. Data reduction: DATRD2 in NRCVAX94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: SOLVER in NRCVAX94. Program(s) used to refine structure: NRCVAX94 and SHELXL93 (Sheldrick, 1993). Molecular graphics: NRCVAX94, PLATON and PLUTON (Spek, 1995b). Software used to prepare material for publication: NRCVAX94, SHELXL93 and WordPerfect macro PREPCIF.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1473). Services for accessing these data are described at the back of the journal.

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trans- and cis-S—C—C—S Conformations in 5-(2,2'-Dithien-5-yl)-2'-deoxyuridine

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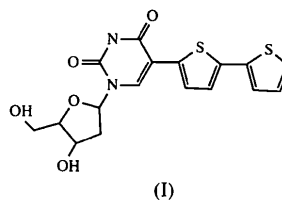
(Received 9 September 1996; accepted 12 February 1997)

Abstract

The title compound, C₁₇H₁₆N₂O₅S₂, adopts a *transoid-cisoid* disordered crystal structure of the terminal thienyl group at room temperature, with occupancies of 0.55 and 0.45. The structure was solved by direct methods and refined by least-squares calculations to a final *R* of 0.048. Molecular cohesion is stabilized by hydrogen bonding involving the two hydroxyl groups of the sugar moiety and the uridine ring.

Comment

As part of our attempts to identify antiherpes agents by rational drug-design approaches, the structures of a series of deoxyuridine analogues have been characterized (Creuven *et al.*, 1996; Olivier *et al.*, 1994, and references therein). In this contribution, we present the crystal structure of 5-(2,2'-dithien-5-yl)-2'-deoxyuridine, (I), a compound that shows moderate activity against herpes simplex virus type 1 (Luyten *et al.*, 1995).



The molecular structure with the atom-numbering scheme is given in Fig. 1. The observed geometry corresponds to standard values (Allen *et al.*, 1987) and is similar to that of previously reported structures for analogues of 5-substituted 2'-deoxyuridines (Olivier *et al.*, 1994; Creuven *et al.*, 1996).