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5,5-Dihydroxyethylbarbituric Acid

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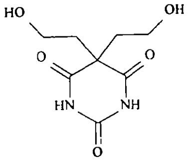
Abstract

The title compound, $C_8H_{12}N_2O_5$, does not show conformational symmetry in the crystal. It is stabilized by intermolecular hydrogen bonds producing a three-dimensional network of hydrogen bonds. The two molecules in the asymmetric unit differ in the conformation of the six-membered ring and the way in which each participates in the hydrogen-bonding network.

Comment

The title molecule, (I), was prepared as a preliminary step in the synthesis of a new achiral nucleotide analogue (Barbier *et al.*, 1993). The crystal structure was studied as part of a program to examine the packing characteristics of barbiturates containing 5,5-polar substituents which can be involved in hydrogen bonding.



(I)

The asymmetric unit of the title structure contains two non-equivalent molecules. The two molecules are pseudo-related by $x_A = x_B + \frac{1}{2}$, $y_A = \frac{1}{2} - y_B$, $z_A = z_B$, which together with the crystal symmetry, suggests the pseudo-translation vector $[\frac{1}{2},0,\frac{1}{2}]$. This is a non-crystallographic translation symmetry because 295 reflections with $h + l = 2n + 1$ have $I > 10\sigma(I)$ [802 have $I > 3\sigma(I)$].

The main differences between the two molecules are in the conformation of the six-membered ring and the hydrogen bonds formed by the hydroxyl groups. The six-membered ring of molecule *A* has a twist-boat form with the N1—C1—C4—C3, N2—C3—C4—C1 and C1—N1—C2—N2 torsion angles equal to −7.2 (3), −1.8 (3) and −7.3 (3)°, respectively, while that of molecule *B* has a boat form with corresponding torsion angles of 3.2 (3), 5.8 (3) and 11.4 (3)°. Only one hydroxyl group of molecule *A* is involved in a hydrogen bond (O5*A*···O4*A*), while both hydroxyl groups of molecule *B* are involved in hydrogen bonds with O atoms of molecule *A*. These hydrogen bonds produce a two-dimensional network in the *ab* plane.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) for 5,5-disubstituted barbiturates yielded 68 compounds for which crystal structures have been determined. All compounds, including the title

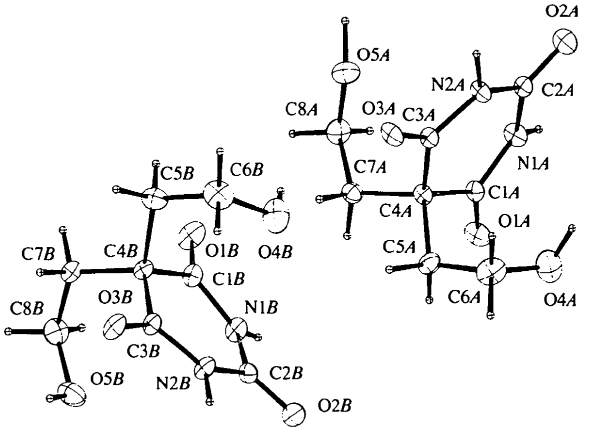


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

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compound, were found to have the triketo tautomeric form in the crystal structure. The hydrogen bonds involving the N—H groups of the barbiturate ring produce an infinite polar chain along the $[\bar{1}01]$ axis. These chains are linked by the hydrogen bonds involving the hydroxyl groups to produce a three-dimensional network of hydrogen bonds.

Experimental

Prismatic colourless crystals with well developed features were obtained by the sitting-drop vapour-phase diffusion method of a CH_2Cl_2–acetonitrile (68:32) solution (concentration 1 mg ml^{-1}) against a CH_2Cl_2–acetonitrile (58:42) solution (Van der Sluis *et al.*, 1989).

Crystal data

$C_8H_{12}N_2O_5$
$M_r = 216.20$
Monoclinic
$P2_1/c$
$a = 12.117\,(2)$ Å
$b = 12.324\,(2)$ Å
$c = 13.508\,(3)$ Å
$\beta = 108.83\,(3)°$
$V = 1909.2\,(6)$ Å^3
$Z = 8$
$D_x = 1.504$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12$–$21°$
$\mu = 0.126$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
5515 measured reflections
5464 independent reflections
2675 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.038$
$\theta_{\rm max} = 29.97°$
$h = -17 \rightarrow 16$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.290$
$S = 0.984$
5464 reflections
296 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1817P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.004$

$\Delta\rho_{\rm max} = 0.533$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.553$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.002 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1*A*—C2*A*	1.372 (2)	N1*B*—C2*B*	1.368 (2)
N1*A*—C1*A*	1.374 (3)	N1*B*—C1*B*	1.377 (3)
N2*A*—C2*A*	1.374 (2)	N2*B*—C3*B*	1.361 (2)
N2*A*—C3*A*	1.375 (2)	N2*B*—C2*B*	1.375 (2)
C1*A*—C4*A*	1.527 (2)	C1*B*—C4*B*	1.520 (2)
C3*A*—C4*A*	1.519 (2)	C3*B*—C4*B*	1.516 (2)
C2*A*—N1*A*—C1*A*	126.83 (15)	C2*B*—N1*B*—C1*B*	125.73 (15)
C2*A*—N2*A*—C3*A*	126.0 (2)	C3*B*—N2*B*—C2*B*	126.6 (2)
O1*A*—C1*A*—N1*A*	120.6 (2)	O1*B*—C1*B*—N1*B*	120.5 (2)
O1*A*—C1*A*—C4*A*	122.0 (2)	O1*B*—C1*B*—C4*B*	121.4 (2)
N1*A*—C1*A*—C4*A*	117.20 (15)	N1*B*—C1*B*—C4*B*	117.92 (15)
O2*A*—C2*A*—N1*A*	122.7 (2)	O2*B*—C2*B*—N1*B*	122.7 (2)
O2*A*—C2*A*—N2*A*	121.2 (2)	O2*B*—C2*B*—N2*B*	121.2 (2)
N1*A*—C2*A*—N2*A*	116.1 (2)	N1*B*—C2*B*—N2*B*	116.1 (2)
O3*A*—C3*A*—N2*A*	119.8 (2)	O3*B*—C3*B*—N2*B*	120.3 (2)
O3*A*—C3*A*—C4*A*	121.8 (2)	O3*B*—C3*B*—C4*B*	121.4 (2)
N2*A*—C3*A*—C4*A*	118.35 (14)	N2*B*—C3*B*—C4*B*	118.15 (14)
C3*A*—C4*A*—C1*A*	114.3 (2)	C3*B*—C4*B*—C1*B*	114.2 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—HN1*A*···O2*B*$^{\rm i}$	0.86	2.20	3.044 (3)	165
N1*B*—HN1*B*···O2*A*$^{\rm ii}$	0.86	2.07	2.918 (3)	168
N2*A*—HN2*A*···O3*A*$^{\rm iii}$	0.91 (4)	2.02 (4)	2.894 (2)	161 (3)
N2*B*—HN2*B*···O3*B*$^{\rm iv}$	0.85 (4)	2.00 (4)	2.835 (2)	165 (4)
O4*B*—HO4*B*···O3*A*	0.82	2.49	2.923 (3)	114
O5*A*—HO5*A*···O4*A*$^{\rm i}$	1.06 (4)	1.91 (3)	2.832 (3)	143 (2)
O5*B*—HO5*B*···O5*A*$^{\rm v}$	0.82	2.05	2.849 (4)	163

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

The positions of 16 H atoms were calculated, while the remaining eight (HO4*A*, HO5*A*, HN2*A*, HN2*B*, H7*A*, H7*A*′, H6*B* and H6*B*′) were obtained from a difference synthesis. An overall isotropic displacement parameter was used for all H atoms. The calculated H atoms were refined using a riding model, while the remainder were refined freely.

Data collection: *CAD-4/PC* (Kretschmar, 1996). Cell refinement: *CAD-4/PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1024). Services for accessing these data are described at the back of the journal.

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