5,5-Dihydroxy-1,3-dipropionyl-1,3-diazacyclohexane

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.066
wR factor = 0.135
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{10}H_{18}N_2O_4$, the diazacyclohexane ring is in a normal chair conformation. The molecule packs to form hydrogen-bonded dimers around a center of symmetry.

Comment

1,3-Disubstituted 1,3-diazacyclohexan-5-ones are key intermediates used for the introduction of a variety of substituents at the 5-position to form the analogous nitramines, which may be useful energetic materials. The diazacyclohexane ring in the title compound, (I), has a normal chair conformation. Chemically, this compound could have a twofold axis passing through C2 and C5. This did not happen in the crystal where both the orientation and the packing environment of the propionyl moieties are different from one another. The molecules form hydrogen-bonded dimers around a center of symmetry (Fig. 2). Both hydroxyl-H atoms are donors to O1 with one bond being weaker than the other (see Table 1). The second carbonyl-O atom, O3, does not participate in any hydrogen bonding.
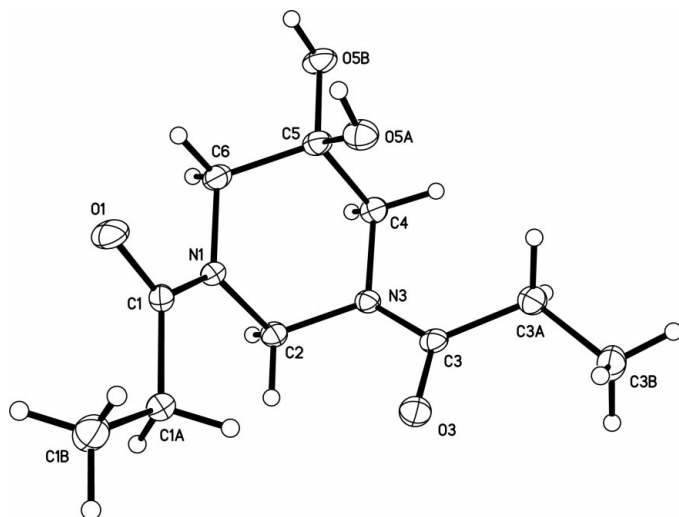

(I)
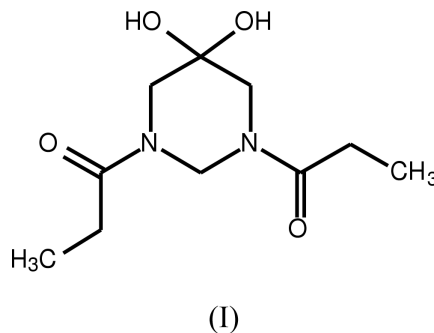
Experimental

The Dess–Martin (Dess & Martin, 1991) procedure was found to oxidize the dipropionylated 5-hydroxy-1,3-diazacyclohexane cleanly to the ketone which, on aqueous work-up, readily afforded the title compound as a geminal diol (sometimes called a 'ketone-hydrate' because it is formed by the addition of water across the ketone double bond). The crystal, however, contained no water.

Crystal data

$C_{10}H_{18}N_2O_4$	$D_x = 1.365$ Mg m^{-3}
$M_r = 230.26$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3629
$a = 24.2121$ (16) Å	reflections
$b = 5.3063$ (4) Å	$\theta = 2.7$–28.1°
$c = 19.7644$ (12) Å	$\mu = 0.11$ mm^{-1}
$\beta = 118.023$ (1)°	$T = 294$ (2) K
$V = 2241.6$ (3) Å^3	Thick plate, colorless
$Z = 8$	$0.11 \times 0.35 \times 0.40$ mm

Figure 1
View of compound (I) with 30% probability ellipsoids.

Figure 2
Hydrogen-bonded dimers, formed around a center of symmetry. Both hydroxyl-H atoms donate to the same carbonyl-O atom (O1*A*).

The coordinates and isotropic displacement parameters were refined for the two hydroxyl-H atoms. All other H atoms were included using a riding model.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001).

References

Bruker (2001). *SMART* (Version 5.624) and *SAINT* (Version 6.04) for Windows-NT, and *SHELXTL* (Version 5.10) for UNIX. Bruker AXS Inc., Madison, Wisconsin, USA.
Dess, D. B. & Martin, J. C. (1991). *J. Am. Chem. Soc.* **113**, 7277–7287.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Data collection

Bruker SMART 1000 diffractometer	2044 independent reflections
φ and ω scans	1621 reflections with $I > 2\sigma(I)$
Absorption correction: by integration (Bruker, 2001)	$R_{int} = 0.041$
$T_{min} = 0.961$, $T_{max} = 0.984$	$\theta_{max} = 25.3°$
6438 measured reflections	$h = -28 \rightarrow 29$
	$k = -6 \rightarrow 6$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 2.4823P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.135$	$(\Delta/\sigma)_{max} = 0.003$
$S = 1.24$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2044 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
155 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O5A—H5$A\cdots$O1^i	0.84 (3)	2.26 (3)	2.918 (3)	136 (3)
O5B—H5$B\cdots$O1^i	0.92 (4)	1.97 (4)	2.833 (3)	157 (3)

Symmetry code: (i) $-x, -y, -z$.