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3-Butyl-5-hydroxy-4-iodohexahydropyrrolo[1,2-*c*][1,3]oxazin-1-one

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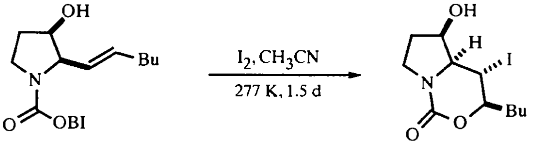
Abstract

The title compound, $C_{11}H_{18}INO_3$, crystallizes with two independent molecules in the asymmetric unit. The conformation of the *n*-butyl chain is different in each molecule. Two types of infinite O—H···O hydrogen-bonded chains are formed, with distances O···O 2.791 (3) and O···O 2.699 (3) Å.

Comment

N-heterocycles are attractive targets for synthesis, often having potent biological activity. Iodocyclocarbamation strategies, first explored by Pauls & Fraser-Reid (1980), have been shown to be particularly useful for formation of [1,3]oxazin-2-one ring systems (Parker & O'Fee, 1983). As part of our on-going interest in the chemistry of substituted pyrrolidines (Batey *et al.*, 1999), we have investigated the iodocyclocarbamation of pyrrolidine, (1). The reaction proceeded to give only one regioisomer and diastereomer of the iodocyclocarbamation product (2). The crystal structure was determined to establish the relative stereochemistry of (2).



(1) (2)

The asymmetric unit of the structure of (2) contains two independent molecules in which the *n*-butyl chains have different conformations. The *n*-butyl chain in molecule *A* has an extended conformation which can be described by the torsion angle of −175.8 (3)° for C9*A*—C10*A*—C11*A*—C12*A*, whereas in molecule *B*, the equivalent torsion angle C9*B*—C10*B*—C11*B*—C12*B* is 68.8 (4)° and this gives rise to a coiled conformation in the *n*-butyl group. In addition, the orientation of the hydroxyl groups in each of the molecules adopts a different conformation, as can be observed in the torsion angles H1*A*—O1*A*—C6*A*—C5*A* of 177 (3) for molecule *A* and H1*B*—O1*B*—C6*B*—C5*B* of 123 (3)° for molecule *B*. The differences in the conformations of the hydroxyl groups appear to facilitate the most efficient geometry for the hydrogen-bonding mode between molecules (see Fig. 2). Molecule *A* and molecule *B* both form independent, infinite, O···H—O hydrogen-bonded chains along 2_1 screw axes in the *y* direction; details of hydrogen-bond geometry are given in Table 2. The closest intermolecular I···H distance is 3.05 Å for I1*A*···H4*A*—C4*A*(x, $\frac{1}{2}-y$, $2+z$).

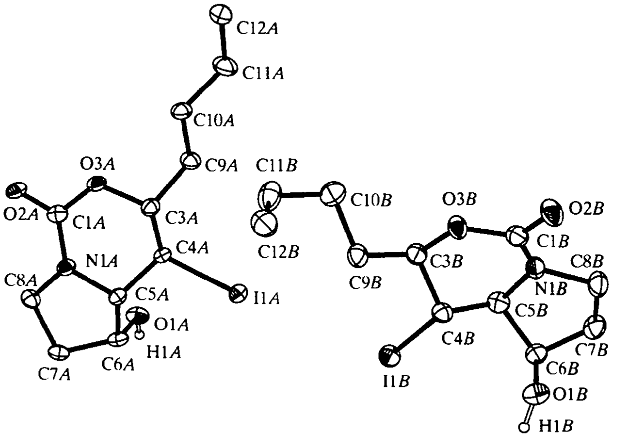


Fig. 1. View of both independent molecules showing the crystallographic labelling scheme. Ellipsoids are shown at the 30% probability level.

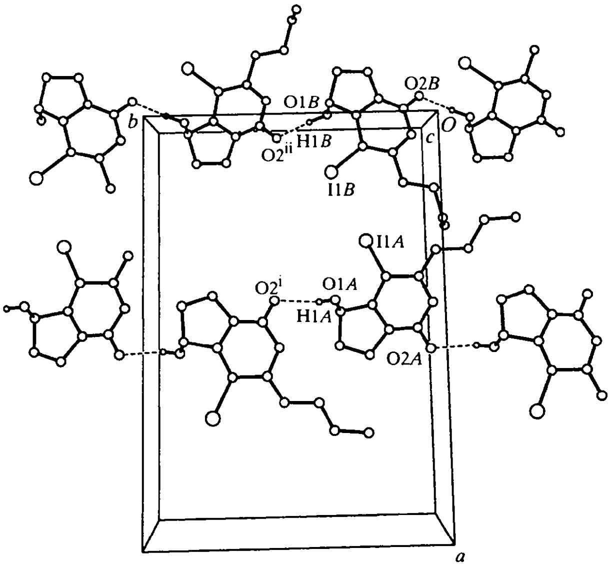


Fig. 2. Hydrogen-bonding mode viewed approximately perpendicular to the *c* axis. [Symmetry codes: (i) $1-x$, $\frac{1}{2}+y$, $-\frac{1}{2}-z$; (ii) $-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$.]

In molecule *A*, the atoms N1*A*, C1*A*, O2*A*, O3*A*, C3*A* and C5*A* form a least-squares best plane with a maximum deviation from the plane of 0.037 (2) for N1*A* and 0.699 (3) Å for C4*A*. However, in molecule *B* only the atoms C1*B*, O2*B*, O3*B*, C3*B* and C5*B* form a plane [with a maximum deviation of −0.038 (2) Å for C1*B*] and N1*B* and C4*B* are −0.168 (3) and 0.740 (4) Å from the plane, respectively.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed that there were only two other molecular structures containing the oxazinone ring system. The bond lengths and angles in the oxazinone functional group in the title compound are comparable to those in 3,4,5,6-tetrahydro-5-iodo-4,4-dimethyl-2*H*-1,3-oxazine-2-one and 3,4,5,6-tetrahydro-5-iodo-6-(*n*-pentyl)-2*H*-1,3-oxazin-2-one (Mague *et al.*, 1997).

Experimental

To a solution of pyrrolidine, (1) (0.101 g, 0.333 mmol), in CH_3CN (2 ml) at 277 K was added I_2 (91.3 mg, 0.358 mmol). The solution was stirred at 277 K for 1.5 d, extracted with Et_2O (3 × 25 ml) from H_2O (10 ml), dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was purified by flash column chromatography (SiO_2, 30–70% EtOAc in hexanes) to afford (2) as a clear colourless solid (68.1 mg, 61%). Single crystals of the compound were obtained by slow evaporation from EtOAc/hexanes (1:3).

Crystal data

$C_{11}H_{18}INO_3$
$M_r = 339.16$
Monoclinic
$P2_1/c$
$a = 20.0869 (2)$ Å
$b = 13.7875 (5)$ Å
$c = 9.4187 (6)$ Å
$\beta = 102.672 (2)°$
$V = 2544.95 (19)$ Å^3
$Z = 8$
$D_x = 1.770$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 17165 reflections
$\theta = 4.08–25.04°$
$\mu = 2.510$ mm^{-1}
$T = 100.0 (1)$ K
Block cut from needle
0.30 × 0.20 × 0.15 mm
Pale yellow

Data collection

Nonius Kappa CCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.520$, $T_{max} = 0.705$

17165 measured reflections
4470 independent reflections
3762 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 25.04°$
$h = -23 \rightarrow 23$
$k = -16 \rightarrow 0$
$l = 0 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.062$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.848$ e Å^{-3}
$\Delta\rho_{min} = -0.658$ e Å^{-3}

$S = 1.030$
4470 reflections
300 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: *SHELXTL/PC* (Sheldrick, 1997)
Extinction coefficient: 0.00045 (11)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

I1*A*—C4*A*	2.153 (3)	I1*B*—C4*B*	2.155 (3)
N1*A*—C1*A*	1.344 (4)	N1*B*—C1*B*	1.335 (4)
N1*A*—C8*A*	1.479 (4)	N1*B*—C5*B*	1.469 (4)
N1*A*—C5*A*	1.479 (4)	N1*B*—C8*B*	1.477 (4)
C1*A*—O3*A*	1.343 (4)	C1*B*—O3*B*	1.353 (4)
O1*A*—C6*A*	1.421 (3)	O1*B*—C6*B*	1.418 (4)
C1*A*—N1*A*—C5*A*	125.0 (3)	O3*A*—C1*A*—N1*A*	118.9 (3)
O2*A*—C1*A*—O3*A*	117.8 (3)		
C9*A*—C10*A*—C11*A*—C12*A*	−175.8 (3)		
C9*B*—C10*B*—C11*B*—C12*B*	68.8 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O2*A*^{i}	0.77 (4)	2.02 (4)	2.791 (3)	170 (4)
O1*B*—H1*B*···O2*B*^{ii}	0.94 (5)	1.77 (5)	2.699 (3)	170 (4)

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

In the refinement, all H atoms were included in calculated positions and treated as riding atoms, except for the hydroxyl-H atoms which were refined with isotropic displacement parameters.

Data collection: *Kappa CCD Server Software* (Nonius, 1997). Cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997). Data reduction: *DENZO-SMN*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1542). Services for accessing these data are described at the back of the journal.

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