The non-methyl and methyl-H atoms were given refined isotropic displacement factors of 0.083 (5) and 0.110 (7) Å^2, respectively. 336 equivalent reflections within the range h −9 to −6, k 0 to 9, l −31 to −1 were measured.

Data collection: Rigaku/AFC software (Rigaku, 1974). Cell refinement: Rigaku/AFC software. Data reduction: Rigaku/AFC software. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

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(R)-N-(1-Hydroxybut-2-yl)pyrrolidine-2,5-dione

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Abstract

The asymmetric unit of the title compound, $C_8H_{13}NO_3$, presents two independent molecules that give two intermolecular hydrogen bonds; the hydroxyl group of the first molecule is both acceptor and donor; that of the second one links to a carbonyl-O atom. This packing

accounts for the small differences between the two independent molecules.

Comment

The title compound, (I), has been prepared in order to extend our previous study (Biju Kumar et al., 1997) on the synthesis of novel ring systems and to obtain more information on the well known biological activity of succinimide derivatives (Khazhakyan & Khachaturyan, 1987).
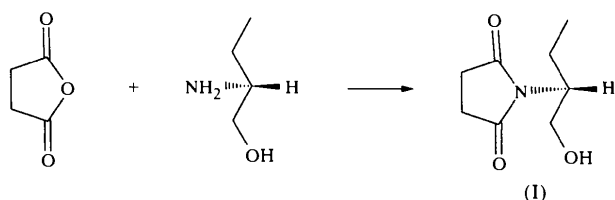
The asymmetric unit of this compound contains two independent molecules. We gave both the same numbering, changing only the suffix (A and B). Fig. 1 shows molecule A. The molecular chirality was assigned on the grounds of the reactants' chirality (see Scheme). Selected bond lengths and angles are reported in Table 1. The differences between equivalent geometric parameters of the two independent molecules are mostly less than three s.u.'s. Relevant exceptions are observed for the parameters involving ethyl groups for which a remarkable apparent bond shortening is found [C3B—C4B 1.382 (10) versus C3A—C4A 1.499 (6) Å in molecules B and A, respectively], due to the very large anisotropic displacement parameters affecting C3B and C4B. This probably means a dynamical disorder of the ethyl group of molecule B, but no attempt to split the two atoms was carried out. Larger differences are found for torsion angles, the most relevant ones being reported in Table 1. These differences are most probably due to packing forces; in fact, the environments of
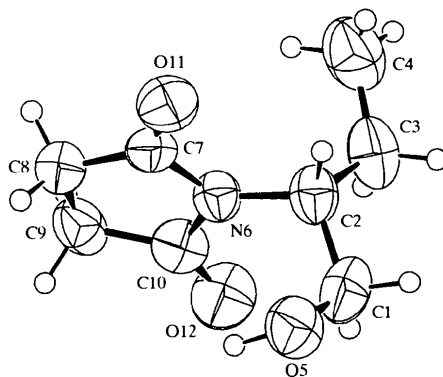

Fig. 1. ORTEPII (Johnson, 1976) plot of molecule A with 50% probability displacement ellipsoids. (The numbering scheme for molecule B is the same as for A.)

the two molecules are different, mainly because O12*B* is engaged in a hydrogen bond [O5*A*···O12*B* 2.736 (4), H5*A*···O12*B* 1.78 (5) Å, O5*A*—H5*A*···O12*B* 178 (4)°], while O12*A* interacts only weakly with the other molecules. It is also noticeable that O5*A* is a donor in the cited hydrogen bond, and it is also an acceptor in the second hydrogen bond [O5*B*···O5*A*ⁱ 2.799 (4), H5*B*···O5*A*ⁱ 1.91 (6) Å, O5*B*—H5*B*···O5*A*ⁱ 162 (5)°; where (i) is $x - \frac{1}{2}, \frac{1}{2} - y, -z$]. For *N*-(2-hydroxyethyl)-succinimide (Pitkanen *et al.*, 1988) and for *N*-(cyano-methyl)succinimide (Tu *et al.*, 1991), the angle between the *N*-bonded group and the pyrrolidine ring is about 90 and 75°, respectively; for (I), the torsion angle C1—C2—N6—C7 is near −60° (Table 1).

Experimental

The title compound was prepared by heating (*R*)-2-amino-1-butanol (1 mmol) with succinic anhydride (1 mmol) at 413 K for 30 min. The reaction mixture was allowed to cool, diluted with water and extracted with diethyl ether. The organic layer was washed with sodium bicarbonate and brine and dried over anhydrous MgSO₄. Evaporation of the solvent provided the compound. Crystallization was carried out in a mixture of hexane and Et₂O. Other solvent systems such as EtOAc–hexane and CH₂Cl₂ did not yield crystals at all. ^{1}H NMR data: 0.90 (3H, *t*, –CH₃), 1.80 (3H, *m*, –CH₂—CH—N), 2.75 (4H, *s*, ring protons), 3.80, 4.20 p.p.m. (1H, 1H, *m*, *m*, –CH₂—O). ^{13}C NMR data: 10.65 (–CH₃), 20.85 (–CH₂), 28.05 (methylene-C atoms on the ring), 56.15 (N—CH), 62.37 (CH₂—O), 178.66 p.p.m. (carbonyl-C atoms on the ring).

Crystal data

C₈H₁₃NO₃	Mo *K*α radiation
$M_r = 171.2$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 50
$P2_12_12_1$	reflections
$a = 9.619\,(1)$ Å	$\theta = 3.60–12.56°$
$b = 10.325\,(2)$ Å	$\mu = 0.093$ mm^{-1}
$c = 18.910\,(3)$ Å	$T = 293\,(2)$ K
$V = 1878.1\,(5)$ Å^3	Prism
$Z = 8$	$0.50 \times 0.30 \times 0.24$ mm
$D_x = 1.211$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Siemens *P*4 diffractometer	$\theta_{max} = 25°$
$2\theta/\omega$ scans	$h = 0 \rightarrow 11$
Absorption correction: none	$k = 0 \rightarrow 12$
3706 measured reflections	$l = -22 \rightarrow 22$
3306 independent reflections	3 standard reflections
1915 reflections with	every 97 reflections
$I > 2\sigma(I)$	intensity decay: none
$R_{int} = 0.015$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.008$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$\Delta\rho_{max} = 0.137$ e Å^{-3}
$wR(F^2) = 0.116$	$\Delta\rho_{min} = -0.139$ e Å^{-3}
$S = 0.970$	
3306 reflections	
268 parameters	
H atoms treated by a mixture of independent and constrained refinement	
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0223 (19)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1*A*—O5*A*	1.415 (5)	C1*B*—O5*B*	1.405 (5)
C2*A*—N6*A*	1.468 (4)	C2*B*—N6*B*	1.477 (4)
N6*A*—C10*A*	1.375 (4)	N6*B*—C10*B*	1.364 (4)
N6*A*—C7*A*	1.383 (4)	N6*B*—C7*B*	1.397 (4)
C10*A*—O12*A*	1.213 (4)	C10*B*—O12*B*	1.223 (3)

C1*A*—C2*A*—N6*A*—C10*A*	115.9 (4)
C3*A*—C2*A*—N6*A*—C10*A*	−118.2 (3)
C1*A*—C2*A*—N6*A*—C7*A*	−61.7 (4)
C3*A*—C2*A*—N6*A*—C7*A*	64.2 (4)
C10*A*—N6*A*—C7*A*—C8*A*	4.3 (3)
C7*A*—N6*A*—C10*A*—O12*A*	178.6 (3)
C7*A*—N6*A*—C10*A*—C9*A*	−0.6 (3)
C8*A*—C9*A*—C10*A*—N6*A*	−3.3 (3)
C1*B*—C2*B*—N6*B*—C10*B*	119.0 (4)
C3*B*—C2*B*—N6*B*—C10*B*	−114.2 (4)
C1*B*—C2*B*—N6*B*—C7*B*	−62.6 (4)
C3*B*—C2*B*—N6*B*—C7*B*	64.2 (5)
C10*B*—N6*B*—C7*B*—C8*B*	−2.0 (4)
C7*B*—N6*B*—C10*B*—O12*B*	−174.1 (3)
C7*B*—N6*B*—C10*B*—C9*B*	7.2 (4)
C8*B*—C9*B*—C10*B*—O12*B*	172.2 (3)
C8*B*—C9*B*—C10*B*—N6*B*	−9.1 (4)

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93, *PARST* (Nardelli, 1983) and *PARSTCIF* (Nardelli, 1991) .

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