

**(*S,S*)-*N*-Cyclohexyl-3,4-dihydroxypyrrolidine**

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**Key indicators**

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

$R$  factor = 0.040

$wR$  factor = 0.097

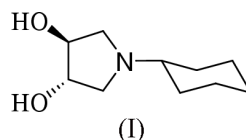
Data-to-parameter ratio = 8.9

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

The title compound,  $\text{C}_{10}\text{H}_{19}\text{NO}_2$ , has two symmetry-independent molecules in the asymmetric unit with almost identical geometry. The pyrrolidine rings have envelope conformations and the cyclohexyl rings slightly distorted chair conformations. The molecules are joined by hydrogen bonds, *via* the hydroxyl groups, in zigzag chains running along the [101] direction.

**Comment**

The title compound, (I), is one of a series of pyrrolidine-based  $\beta$  aminoalcohols derived from tartaric acid that can be used as chiral auxiliaries in the enantioselective alkylation of benzaldehyde (Rocha Gonsalves *et al.*, 2002).



The asymmetric unit contains two independent molecules (1 and 2) with similar geometry. Extensive testing with *PLATON* (Spek, 2002) did not show any missed symmetry element that might relate the two molecules.

The pyrrolidine rings are puckered, with an average torsion angle of  $25.03\text{ (9)^\circ}$  [molecule 2:  $24.70\text{ (9)^\circ}$ ]. The conformation is close to an envelope form. The least-squares plane defined by atoms C2, C3, C4 and C5 is planar within  $0.016\text{ (2) \AA}$  [molecule 2:  $0.008\text{ (2) \AA}$ ]. Atom N1 is tilted out of this plane by  $0.567\text{ (4) \AA}$  [molecule 2:  $0.561\text{ (4) \AA}$ ]. The Cremer & Pople (1975) puckering parameters, calculated using the atom sequence N1,C2,...,C5, are  $q_2 = 0.372\text{ (3) \AA}$  [molecule 2:  $0.369\text{ (3) \AA}$ ] and  $\varphi_2 = 3.9\text{ (4)^\circ}$  [molecule 2:  $358.1\text{ (4)^\circ}$ ]. The  $\varphi_2$  angle for the pure envelope form is  $0^\circ$ . There is an approximate mirror plane passing through N1 and the centre of the C3–C4 bond, the  $C_s(1)$  asymmetry parameter (Duax & Norton, 1975) being  $3.4\text{ (2)^\circ}$  [molecule 2:  $1.5\text{ (2)^\circ}$ ].

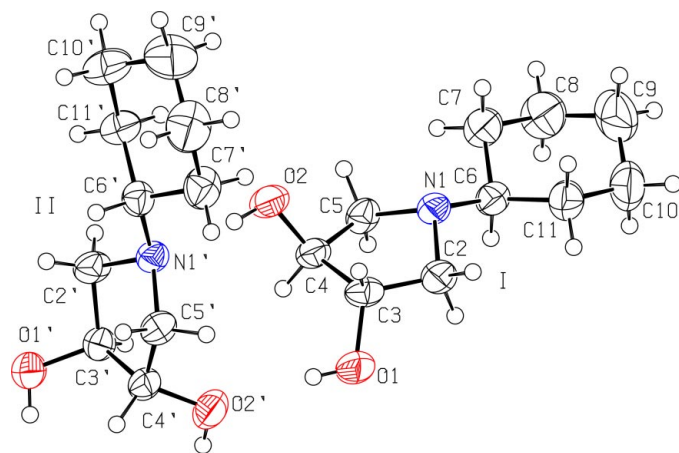
The cyclohexyl rings have a slightly distorted chair conformation, with an average torsion angle of  $55.49\text{ (15)^\circ}$  [molecule 2:  $55.90\text{ (14)^\circ}$ ] and puckering parameters, calculated using the atom sequence C6,C7,...,C11,  $Q = 0.568\text{ (4) \AA}$  [molecule 2:  $0.570\text{ (4) \AA}$ ] and  $\theta = 177.7\text{ (4)^\circ}$  [molecule 2:  $178.8\text{ (4)^\circ}$ ].

The bond distances and angles are unexceptional. The environment of atom N1 is clearly pyramidal, as shown by the average value of the C–N–C angles of  $109\text{ (3)^\circ}$  [molecule 2:  $109\text{ (3)^\circ}$ ] and the distance of N1 from the plane through atoms C2, C5 and C6 [molecule 1:  $0.498\text{ (2) \AA}$ ; molecule 2:  $0.503\text{ (2) \AA}$ ]. The pyrrolidine endocyclic C–C bond length opposite the N atom is significantly longer than the two other

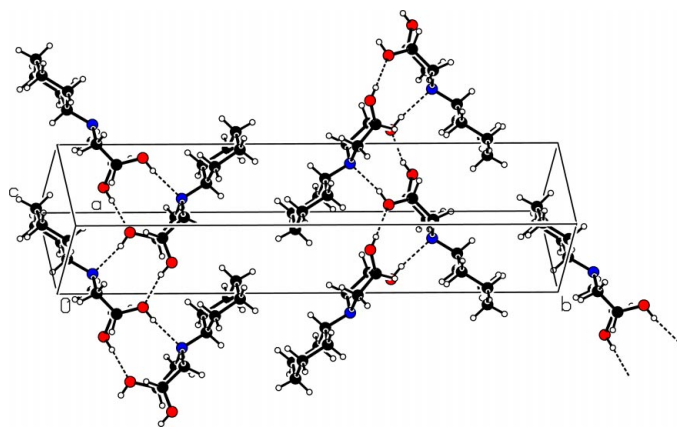
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**Figure 1**  
ORTEP (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**  
View of the unit cell, showing the molecular packing and the hydrogen-bonded zigzag chains.

C—C bonds. The exocyclic N—C bond is shorter than the two endocyclic bonds.

The molecules are linked in zigzag chains running in the [101] direction, *via* hydrogen bonds involving the hydroxyl groups and the pyrrolidine N atom. The O2 [O2'] atoms are donors to the lone pairs of the pyrrolidine N atoms. The O1 [O1'] hydroxyl groups donate their H atoms to the lone pairs of the O2 [O2'] atoms (Fig. 2). Although the hydrogen bonds involving atoms O2 and O2' as donors have similar geometry, the same is not true for those with atoms O1 and O1' as donors. In fact, the O1'—H1'...O2<sup>i</sup> bond is significantly shorter and more nearly linear than the O1—H1...O2' bond.

## Experimental

The title compound was synthesized as described by Rocha Gonsalves *et al.* (2002). (*R,R*)-Cyclohexyldioxopyrrolidine was obtained by refluxing tartaric acid with cyclohexylamine in xylene, using Dean–Stark conditions. In a second step, the (*R,R*)-cyclohexyldioxopyrrolidine was reduced to the title compound with lithium aluminium hydride. Crystallization was carried out in ethyl acetate/petroleum ether, giving white crystals.

## Crystal data

C<sub>10</sub>H<sub>19</sub>NO<sub>2</sub>  
*M<sub>r</sub>* = 185.26  
 Monoclinic, *P*2<sub>1</sub>  
*a* = 6.605 (5) Å  
*b* = 25.230 (8) Å  
*c* = 6.870 (5) Å  
 $\beta$  = 113.22 (5)°  
*V* = 1052.1 (12) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.170 Mg m<sup>-3</sup>  
 Cu *K*α radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 18.3–31.5°  
 $\mu$  = 0.64 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, white  
 0.42 × 0.40 × 0.36 mm

## Data collection

Enraf–Nonius CAD-4 diffractometer  
 Profile data from  $\omega$ -2 $\theta$  scans  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
*T*<sub>min</sub> = 0.719, *T*<sub>max</sub> = 0.791  
 6050 measured reflections  
 2126 independent reflections  
 1928 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.098  
 $\theta_{\text{max}}$  = 72.4°  
*h* = -8 → 8  
*k* = 0 → 31  
*l* = -8 → 7  
 3 standard reflections  
 frequency: 180 min  
 intensity decay: 10.2%

## Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.040  
*wR*(*F*<sup>2</sup>) = 0.097  
*S* = 1.09  
 2126 reflections  
 239 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.0651P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{Å}^{-3}$

**Table 1**

Selected geometric parameters (Å).

N1—C6	1.465 (3)	C2—C3	1.500 (4)
N1—C2	1.480 (3)	C3—C4	1.543 (3)
N1—C5	1.484 (3)	C4—C5	1.505 (3)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...O2'	0.82	2.01	2.795 (3)	160
O2—H2...N1'	0.82	2.00	2.814 (3)	170
O1'—H1'...O2 <sup>i</sup>	0.82	1.94	2.760 (3)	176
O2'—H2'...N1 <sup>i</sup>	0.82	2.07	2.885 (3)	174

Symmetry code: (i) *x* - 1, *y*, *z* - 1.

All H atoms, except those of the hydroxyl groups, were positioned at calculated idealized locations and allowed to refine as riding on their parent atoms using *SHELXL97* defaults for bond lengths and angles. Initial positions for the hydroxyl H atoms were obtained using a circular Fourier calculation, and they were refined with constrained bond lengths and angles, allowing torsional freedom. The (*S,S*) absolute configuration of the chiral centers C3 and C4, known from the synthetic route, was assumed. Examination of the crystal structure with *PLATON* (Spek, 2002) showed that the largest void in the structure has a volume of 32 (3) Å<sup>3</sup>, which is too small to be occupied by a solvent molecule.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *HELENA* (Spek, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL97*.

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## References

- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structure*, Vol. I. New York: Plenum.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Rocha Gonsalves, A. M. d'A., Serra, M. E. S., Murtinho, D., Silva, V. F., Matos Beja, A., Paixão, J. A., Ramos Silva, M. & Alte da Veiga, L. (2002). *J. Mol. Catal. A: Chem.* In the press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2002). *PLATON* and *HELENA*. University of Utrecht, The Netherlands.