(S)-N-(1H-Tetrazol-5-ylmethyl)pyrrolidine-2-carboxamide dihydrate

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In the title compound, $C_7H_{12}N_6O\cdot 2H_2O$, all bond lengths and angles show normal values. The pyrrolidine ring adopts a C1–C^γ-*endo* conformation. An extensive network of N—H···O and O—H···O hydrogen bonds stabilizes the crystal packing.

Comment

Many proline and proline-derived catalysts have been developed for the direct asymmetric aldol reaction (List *et al.*, 2000; Saito *et al.*, 2004; Notz *et al.*, 2004; Tang *et al.*, 2003). With the aim of searching for new chiral organic catalysts for this reaction, we have used *N*-carbobenzyloxy-(*S*)-proline and glycine tetrazole in a two-step synthesis to prepare the title compound, (I), which shows good catalytic properties in the anhydrous form.

(I)
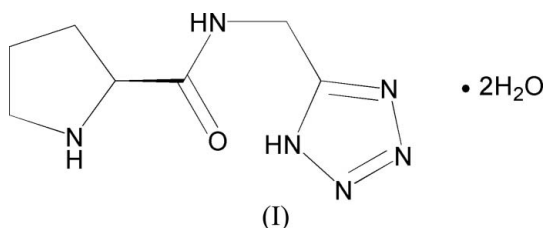
In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987). The pyrrolidine ring (atoms N1/C1–C4) adopts a C1–C^γ-*endo* conformation. The mean plane through atoms C1/C5/N2/C6 makes a dihedral angles of 69.6 (1)° with the mean plane of the tetrazole ring. The uncoordinated water molecules are involved in the formation of an extensive network of intermolecular O—H···O and N—H···O hydrogen bonds (Table 1), which stabilizes the crystal structure (Fig. 2).

Experimental

The title compound was prepared in a two-step synthesis using *N*-carbobenzyloxy-(*S*)-proline and glycine tetrazole (Zheng *et al.*, 2006). Fine rod-shaped colourless crystals for single-crystal X-ray diffraction were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_7H_{12}N_6O\cdot 2H_2O$	$Z = 4$
$M_r = 232.26$	$D_x = 1.361$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 9.2217$ (8) Å	$\mu = 0.11$ mm^{-1}
$b = 12.0008$ (11) Å	$T = 293$ (2) K
$c = 10.2391$ (10) Å	Rod, colourless
$V = 1133.14$ (18) Å^3	$0.45 \times 0.22 \times 0.22$ mm

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Data collection

Bruker SMART APEX CCD diffractometer	1533 independent reflections
ω scans	1427 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.088$
7136 measured reflections	$\theta_{max} = 28.2°$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.050$	
$wR(F^2) = 0.141$	
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
1533 reflections	where $P = (F_o^2 + 2F_c^2)/3$
173 parameters	$(\Delta/\sigma)_{max} = 0.006$
	$\Delta\rho_{max} = 0.46$ e Å^{-3}
	$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
OW1—H1W1⋯OW2^i	0.86 (3)	1.91 (3)	2.757 (3)	173 (5)
OW1—H2W1⋯N6^{ii}	0.85 (3)	2.03 (3)	2.879 (3)	174 (3)
N2—H2⋯OW1	0.86 (3)	1.98 (3)	2.835 (3)	176 (3)
OW2—H2W2⋯N5	0.85 (3)	2.06 (2)	2.878 (4)	161 (5)

Symmetry codes: (i) $x + 1, y, z$; (ii) $x + \frac{1}{2}, -y + \frac{5}{2}, -z + 1$.

Atoms H1, H2, H3, H4 and the water H atoms were located in a difference map and refined isotropically, with the distance restraints N—H = 0.86 (3) Å, C1—H4 = 0.97 (1) Å and O—H = 0.85 (3) Å. The remaining H atoms were positioned geometrically (C—H = 0.97 Å and N—H = 0.86 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}$(parent atom). In the absence of significant anomalous scattering effects, the 528 Friedel pairs were merged before the final refinement.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

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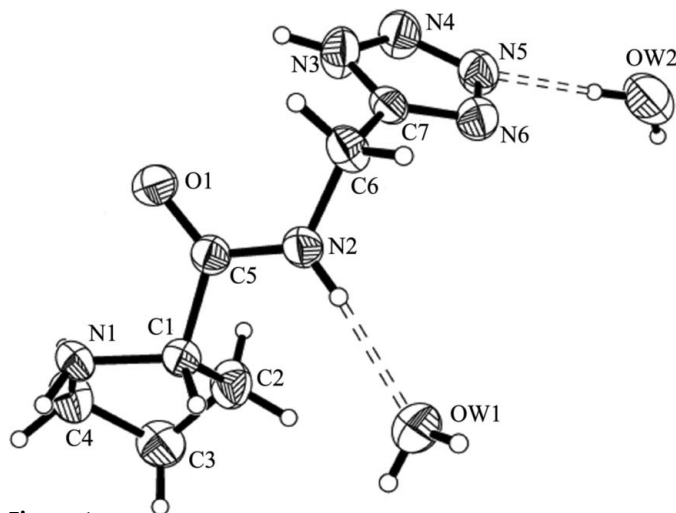Saito, S. & Yamamoto, H. (2004). *Acc. Chem. Res.* **37**, 570–579.

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Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.
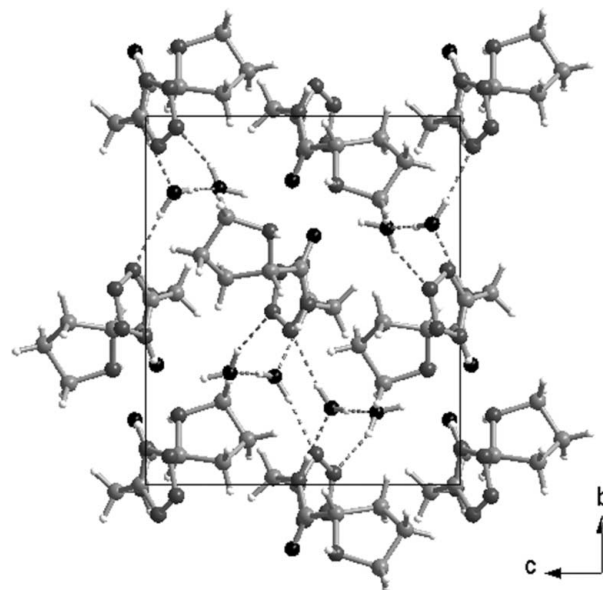

Figure 2
The packing viewed down the *a* axis. The dashed lines indicate the intermolecular hydrogen bonds.