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cis-(Piperidine-2,6-diyl)dimethanol

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Key indicators

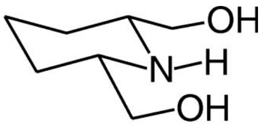
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.083
Data-to-parameter ratio = 11.9

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

Molecules of the title compound, $C_7H_{15}NO_2$, form O—H···O hydrogen-bonded centrosymmetric dimers. These dimers further aggregate *via* O—H···N hydrogen bonds.

Comment

The title compound, (I), was prepared and investigated by X-ray diffraction in order to explore the geometric parameters of 2,6-*cis*-substituted piperidines. Such heterocycles have recently attracted attention as a promising new type of auxiliary for powerful and selective oxovanadium-based oxidation catalysts (Hartung *et al.*, 2007).


(I)

Atoms N1 and C4 in compound (I) are displaced in opposite directions [−0.695 (1) and 0.625 (2) Å, respectively] from the mean plane through atoms C2/C3/C5/C6, giving rise to the $^1C_4$ conformation of the piperidine ring (Fig. 1). The side-chain substituents are located in relative *cis* configurations, as expected from ^{1}H NMR analysis of the starting material used to prepare (I). The heteroatoms in both hydroxymethyl side chains adopt synclinal orientations (Table 1), similar to the preferred *gauche* arrangement of diacceptor-substituted ethane entities in the uncondensed phase (Kirby, 1983).

In the crystal structure, molecules of (I) form O—H···O hydrogen-bonded centrosymmetric dimers. These dimers aggretate *via* O—H···N hydrogen bonds (Fig. 2).

Experimental

A solution of *cis*-dimethylpiperidine-2,6-dicarboxylate (Henderson *et al.*, 1996) (8.65 g, 43.0 mmol) in tetrahydrofuran (THF) (100 ml) was added under an inert gas atmosphere (N_2) at 298 K to a solution of $LiAlH_4$ (3.26 g, 86.0 mmol) in THF (50 ml). The reaction mixture was heated under reflux for 3 h. It was subsequently cooled (273 K) and diluted with H_2O (50 ml) to afford a slurry, which was concentrated under reduced pressure. The remaining oil was treated with pyridine (100 ml). Solids which precipitated at this point were removed by filtration. The filtrate was concentrated under reduced pressure and the remaining residue was crystallized from THF (4.58 g, 73%; m.p. 401 K). Colourless crystals of (I) suitable for X-ray diffraction were grown by allowing pentane to diffuse slowly into a saturated solution of (I) in THF at 298 K.

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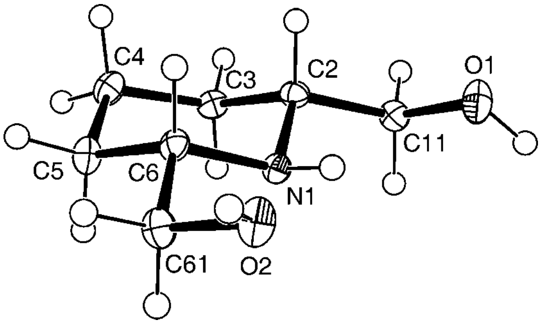

Figure 1
The molecular structure of (I). Displacement ellipsoids are plotted at the 50% probability level.

Crystal data

$C_7H_{15}NO_2$
$M_r = 145.20$
Monoclinic, $P2_1/c$
$a = 9.555$ (3) Å
$b = 8.5120$ (19) Å
$c = 9.906$ (2) Å
$\beta = 94.95$ (2)°

$V = 802.7$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 173$ (2) K
0.25 × 0.10 × 0.10 mm

Data collection

Oxford Diffraction KM-4 Xcalibur2 diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{\min} = 0.945$, $T_{\max} = 1.000$

5200 measured reflections
1809 independent reflections
1307 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.083$
$S = 1.07$
1809 reflections

152 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected torsion angles (°).

N1—C6—C61—O2	58.55 (11)	N1—C2—C11—O1	−62.04 (10)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H9···O2$^{\rm i}$	0.837 (17)	1.906 (18)	2.7432 (13)	177.7 (16)
O2—H8···N1$^{\rm ii}$	0.878 (16)	1.881 (17)	2.7575 (13)	175.8 (14)

Symmetry codes: (i) $-x, -y, -z+2$; (ii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

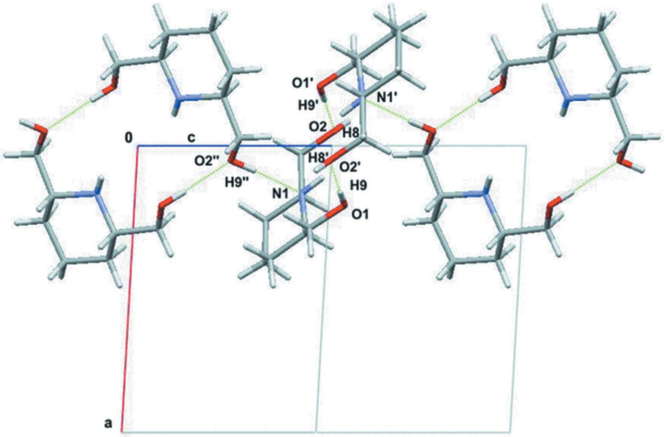

Figure 2
A partial packing diagram of (I), viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines. N atoms are depicted in blue, O in red, C in grey and H in white. Symmetry operators for primed and double-primed atoms are $(-x, -y, 2-z)$ and $(-x, -\frac{1}{2}+y, \frac{3}{2}-z)$, respectively.

All H atoms were located in a difference Fourier map and refined freely.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

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