

1,1'-(Piperazine-1,4-diyl)dipropan-2-ol

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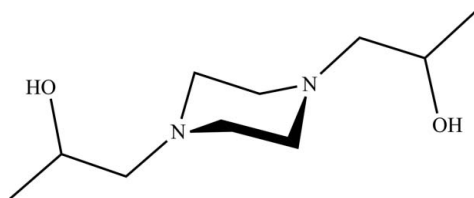
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.052; wR factor = 0.119; data-to-parameter ratio = 13.1.

The asymmetric unit of the crystal contains one-fourth of the title compound, $\text{C}_{10}\text{H}_{22}\text{N}_2\text{O}_2$, with the centre of the piperazine ring located at a site of $2/m$ symmetry. The piperazine ring adopts a chair conformation. The methine and methylene C atoms of the 2-hydroxypropyl groups show symmetry-imposed disorder over two equally occupied and mutually exclusive sets of positions. Only intramolecular $\text{O}-\text{H}\cdots\text{N}$ contacts are observed.

Related literature

For the biological properties of piperazine compounds, see: Foroumadi *et al.* (2007); Upadhayaya *et al.* (2004); Chen *et al.* (2006); Cunico *et al.* (2009); Smits *et al.* (2008); Penjišević *et al.* (2007); Becker *et al.* (2006). For hydrogen-bond graph-set motifs, see: Bernstein *et al.* (1995). For ring puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{22}\text{N}_2\text{O}_2$
 $M_r = 202.30$
Monoclinic, $C2/m$
 $a = 13.838$ (10) Å
 $b = 7.791$ (5) Å
 $c = 5.543$ (4) Å
 $\beta = 97.26$ (3)°

$V = 592.8$ (7) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 273$ K
0.40 × 0.25 × 0.10 mm

Data collection

Rigaku R-Axis RAPID
diffractometer
2493 measured reflections

604 independent reflections
441 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.139$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.119$
 $S = 1.05$
604 reflections
46 parameters

14 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.34$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	2.22	2.696 (3)	117

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2011).

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supplementary materials

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1,1'-(Piperazine-1,4-diyl)dipropan-2-ol

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Comment

Piperazine-based research has attracted considerable attention in recent years. A broad range of compounds displaying antibacterial (Foroumadi *et al.*, 2007), antifungal (Upadhayaya *et al.*, 2004), anticancer (Chen *et al.*, 2006), antiparasitic (Cunico *et al.*, 2009), antihistamin (Smits *et al.*, 2008), psycholytic (Penjišević *et al.*, 2007), and antidepressive activities (Becker *et al.*, 2006) have been found to contain this versatile core. In view of these important properties, we have undertaken the X-ray diffraction study of the title compound.

The structure of the title compound is shown in Fig. 1. The structure contains one central piperazine ring (N1/C4/C4ⁱ/N1ⁱ/C4ⁱⁱ/C4ⁱⁱⁱ) with two propanol moieties substituted at the two N atoms of the piperazine ring. The centre of the ring located at a site of $2/m$ symmetry. The N, O and methyl C atoms are located on the mirror plane, while atoms C2 and C3 show symmetry-imposed disorder.

The interatomic distances and angles in the compound show no anomalies. The piperazine ring adopts a chair conformation, as is evident from the puckering parameters (Cremer & Pople, 1975): $Q_T = 1.0333$ (10) Å, $q_2 = 0.8812$ (9) Å, $q_3 = 0.5396$ (6) Å, $\theta = 58.52$ (2)° and $\varphi_2 = 30.00$ (5)° for the atom sequence N1/C4/C4ⁱ/N1ⁱ/C4ⁱⁱ/C4ⁱⁱⁱ. Atoms N1 and N1ⁱ are on opposite sides of the C4/C4ⁱ/C4ⁱⁱ/C4ⁱⁱⁱ plane and are both displaced from it by 0.2424 (30) Å.

The molecular structure of the title compound contains two intramolecular O—H...N contacts, which form a five-membered ring with graph-set descriptor $S(5)$ (Bernstein *et al.*, 1995). No intermolecular hydrogen bonds are observed in the crystal structure. Van der Waals forces stabilize the packing.

Experimental

Piperazine (1.50 g, 17.40 mmol) was dissolved in 50 ml argon saturated methanol. Methanol solution of 2.88 g (50.00 mmol) propylene oxide was added to the piperazine solution at room temperature. The solution was left under magnetic stirrer for 24 h. The solution volume was reduced by rotary evaporator and the oily product was left for crystallization.

Refinement

H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.82, 0.93, 0.97 and 0.96 Å for OH, CH, CH₂ and CH₃ groups, respectively. The isotropic displacement parameters of the H atoms were constrained at 1.2 U_{eq} of their parent atom (1.5 U_{eq} for methyl and OH groups). Atoms C2 and C3 showed symmetry-imposed disorder and were refined anisotropically using ADP restraints (SIMU and DELU) and half occupancy.

Figures

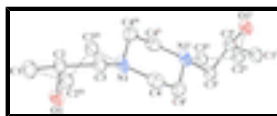


Fig. 1. ORTEP-3 drawing of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering scheme. For the sake of clarity, H atoms have been omitted. One of the disorder components is drawn with dashed bonds. [Symmetry codes: (i) $1 - x, y, 1 - z$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $x, 1 - y, z$.]

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Crystal data

$C_{10}H_{22}N_2O_2$

$M_r = 202.30$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$a = 13.838 (10) \text{ \AA}$

$b = 7.791 (5) \text{ \AA}$

$c = 5.543 (4) \text{ \AA}$

$\beta = 97.26 (3)^\circ$

$V = 592.8 (7) \text{ \AA}^3$

$Z = 2$

$F(000) = 224$

$D_x = 1.133 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3092 reflections

$\theta = 3.0\text{--}30.0^\circ$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 273 \text{ K}$

Prism, colourless

$0.40 \times 0.25 \times 0.10 \text{ mm}$

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: $10.00 \text{ pixels mm}^{-1}$

ω scans

2493 measured reflections

604 independent reflections

441 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.139$

$\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 3.0^\circ$

$h = -16 \rightarrow 16$

$k = -9 \rightarrow 9$

$l = -5 \rightarrow 6$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.119$

$S = 1.05$

604 reflections

46 parameters

14 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.2898P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.78313 (12)	0.5000	0.4674 (3)	0.0706 (7)	
H1	0.7297	0.5197	0.3907	0.106*	0.50
N1	0.60084 (15)	0.5000	0.5912 (4)	0.0715 (7)	
C1	0.86326 (18)	0.5000	0.8733 (5)	0.0754 (9)	
H1A	0.9169	0.4390	0.8197	0.113*	0.50
H1B	0.8573	0.4690	1.0384	0.113*	0.50
H1C	0.8746	0.6213	0.8640	0.113*	0.50
C2	0.7712 (2)	0.4545 (5)	0.7137 (5)	0.0603 (12)	0.50
H2	0.7597	0.3307	0.7233	0.072*	0.50
C3	0.6841 (2)	0.5495 (5)	0.7803 (6)	0.0609 (11)	0.50
H3A	0.6952	0.6724	0.7782	0.073*	0.50
H3B	0.6705	0.5166	0.9413	0.073*	0.50
C4	0.54111 (14)	0.6506 (3)	0.6021 (3)	0.0751 (7)	
H4A	0.5806	0.7526	0.5917	0.090*	
H4B	0.5150	0.6533	0.7565	0.090*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0609 (11)	0.0975 (15)	0.0541 (11)	0.000	0.0103 (8)	0.000
N1	0.0463 (11)	0.118 (2)	0.0498 (12)	0.000	0.0031 (9)	0.000
C1	0.0527 (15)	0.103 (2)	0.0677 (17)	0.000	-0.0030 (13)	0.000
C2	0.0519 (15)	0.075 (3)	0.0533 (16)	0.0024 (15)	0.0027 (13)	-0.0010 (15)
C3	0.0483 (14)	0.084 (3)	0.0490 (14)	0.0021 (14)	0.0016 (12)	-0.0047 (14)
C4	0.0807 (13)	0.0883 (15)	0.0563 (11)	-0.0195 (11)	0.0084 (9)	-0.0038 (11)

Geometric parameters (\AA , $^\circ$)

O1—C2 ⁱ	1.440 (4)	C1—H1B	0.9600
O1—C2	1.440 (4)	C1—H1C	0.9600
O1—H1	0.8200	C2—C3	1.499 (4)
N1—C4 ⁱ	1.441 (3)	C2—H2	0.9800

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N1—C4	1.441 (3)	C3—H3A	0.9700
N1—C3	1.507 (3)	C3—H3B	0.9700
N1—C3 ⁱ	1.507 (3)	C4—C4 ⁱⁱ	1.500 (4)
C1—C2	1.499 (4)	C4—H4A	0.9700
C1—C2 ⁱ	1.499 (4)	C4—H4B	0.9700
C1—H1A	0.9600		
C2 ⁱ —O1—H1	103.9	O1—C2—C3	107.7 (2)
C2—O1—H1	109.5	C1—C2—C3	112.8 (3)
C4 ⁱ —N1—C4	109.0 (2)	O1—C2—H2	109.4
C4 ⁱ —N1—C3	124.8 (2)	C1—C2—H2	109.4
C4—N1—C3	98.90 (17)	C3—C2—H2	109.4
C4 ⁱ —N1—C3 ⁱ	98.90 (17)	C2—C3—N1	105.7 (2)
C4—N1—C3 ⁱ	124.8 (2)	C2—C3—H3A	110.6
C2—C1—H1A	109.5	N1—C3—H3A	110.6
C2 ⁱ —C1—H1A	124.6	C2—C3—H3B	110.6
C2—C1—H1B	109.5	N1—C3—H3B	110.6
C2 ⁱ —C1—H1B	116.9	H3A—C3—H3B	108.7
H1A—C1—H1B	109.5	N1—C4—C4 ⁱⁱ	110.64 (15)
C2—C1—H1C	109.5	N1—C4—H4A	109.5
C2 ⁱ —C1—H1C	82.4	C4 ⁱⁱ —C4—H4A	109.5
H1A—C1—H1C	109.5	N1—C4—H4B	109.5
H1B—C1—H1C	109.5	C4 ⁱⁱ —C4—H4B	109.5
O1—C2—C1	108.1 (2)	H4A—C4—H4B	108.1
C2 ⁱ —O1—C2—C1	-70.1 (2)	C4 ⁱ —N1—C3—C2	85.4 (3)
C2 ⁱ —O1—C2—C3	52.0 (2)	C4—N1—C3—C2	-153.8 (2)
C2 ⁱ —C1—C2—O1	69.7 (2)	C3 ⁱ —N1—C3—C2	52.8 (3)
C2 ⁱ —C1—C2—C3	-49.2 (3)	C4 ⁱ —N1—C4—C4 ⁱⁱ	-58.1 (3)
O1—C2—C3—N1	56.3 (3)	C3—N1—C4—C4 ⁱⁱ	170.2 (2)
C1—C2—C3—N1	175.4 (2)	C3 ⁱ —N1—C4—C4 ⁱⁱ	-174.2 (2)

Symmetry codes: (i) $x, -y+1, z$; (ii) $-x+1, y, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N1	0.82	2.22	2.696 (3)	117

Fig. 1

