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rac-trans-Cycloheptane-1,2-diol

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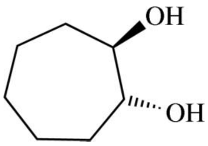
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.113; data-to-parameter ratio = 16.5.

In the title compound, $C_7H_{14}O_2$, a pair of vicinal *trans*-connected hydroxy groups is attached to a cycloheptane ring. The non-crystallographic molecular symmetry is close to C_2. The O—C—C—O torsion angle of approximately 66° resembles those in *trans*-configured cyclohexane derivatives (including the pyranoses) and is smaller than those in *trans*-configured cyclopentane derivatives (including the furanoses). Chains of O—H⋯O hydrogen bonds form a cooperatively bonded hydrogen-bond system. All other intermolecular contacts are of the van der Waals type. In terms of functional-group torsion, the title compound is thus a potential bidentate ligand with the ability to form a C_2-symmetrical chelate ring with a metal atom.

Related literature

For the synthesis of the title compound, see: Becker *et al.* (2001). For torsion angles of *cis*- and *trans*-configured cyclohexane-1,2-diols, see: Sillanpää *et al.* (1984). For chelation abilities of selected *cis*- and *trans*-configured cyclic vicinal diols with silicon as the central atom, see: Kästele *et al.* (2005).



Experimental

Crystal data

$C_7H_{14}O_2$	V = 1469.94 (8) Å^3
M_r = 130.18	Z = 8
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
a = 10.1290 (3) Å	μ = 0.08 mm^{-1}
b = 7.2290 (2) Å	T = 200 (2) K
c = 20.0750 (7) Å	0.24 × 0.15 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1682 independent reflections
Absorption correction: none	1118 reflections with $I > 2\sigma(I)$
3095 measured reflections	$R_{\rm int}$ = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.113	$\Delta\rho_{\rm max}$ = 0.13 e Å^{-3}
S = 1.05	$\Delta\rho_{\rm min}$ = −0.18 e Å^{-3}
1685 reflections	
102 parameters	

Table 1
Selected torsion angles (°).

O1—C1—C2—O2	−66.09 (12)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H81⋯O2$^{\rm i}$	0.90 (2)	1.85 (2)	2.7481 (13)	174.7 (15)
O2—H82⋯O1$^{\rm ii}$	0.90 (2)	1.84 (2)	2.7208 (14)	164.0 (15)

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

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2328).

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supplementary materials

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rac-*trans*-Cycloheptane-1,2-diol

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Comment

The vicinal diol derived from cycloheptane was prepared as a chelating molecule bearing a conformationally flexible seven-membered hydrocarbon ring. The cycloheptane ring adopts a chair-like conformation. Accordingly, the molecular symmetry is close to C_2 symmetry (Fig. 1).

In the crystal structure, hydrophobic contacts close to planes with z = 1/4 and 3/4 divide the structure in sheets (Fig. 2). Fig. 3 shows an individual sheet (the one with $1/4 < z < 3/4$) in a projection along [001]. The characteristic hydrogen bonds are highlighted. In the hydrogen-bond scheme, each hydroxy function acts both as a single hydrogen-bond donor and acceptor. The individual hydrogen bonds are embedded in cooperative chains of alternating polarity along [010] (green and yellow bonds in Fig. 3).

Experimental

The title compound was prepared by standard procedures upon acidic aqueous dihydroxylation of cycloheptene with hydrogen peroxide and formic acid (Becker *et al.*, 2001).

Refinement

All H atoms were located in difference maps and refined with individual isotropic temperature parameters. The C-bonded H atoms were refined as riding on their parent atoms. The O-bonded H atoms were refined with free positional parameters.

Figures

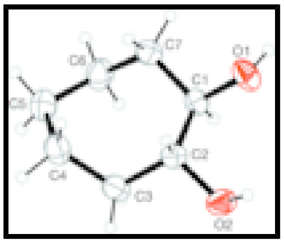

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at the 50% probability level) for non-H atoms.

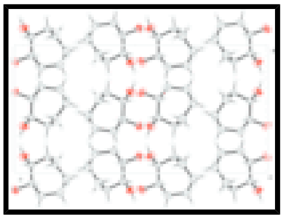

Fig. 2. The packing of the molecules of (I) in the crystal, viewed along [010].

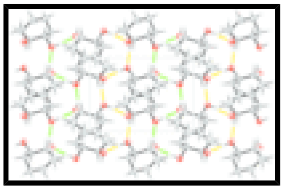

Fig. 3. The packing of the molecules with $1/4 < z < 3/4$, viewed along [001]. Chains of co-operative hydrogen bonds along [010] of alternating polarity are drawn using yellow and green bars.

rac-*trans*-Cycloheptane-1,2-diol

Crystal data

$C_7H_{14}O_2$	$F_{000} = 576$
$M_r = 130.18$	$D_x = 1.176$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1964 reflections
$a = 10.1290$ (3) Å	$\theta = 3.1$–27.5°
$b = 7.2290$ (2) Å	$\mu = 0.08$ mm^{-1}
$c = 20.0750$ (7) Å	$T = 200$ (2) K
$V = 1469.94$ (8) Å^3	Block, colourless
$Z = 8$	0.24 × 0.15 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1118 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.028$
Monochromator: MONTEL, graded multilayered X-ray optics	$\theta_{max} = 27.5°$
$T = 200(2)$ K	$\theta_{min} = 3.6°$
φ and ω scans	$h = -13 \rightarrow 13$
Absorption correction: none	$k = -9 \rightarrow 9$
3095 measured reflections	$l = -26 \rightarrow 25$
1682 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.1228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1685 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
102 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

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. The refU data value refers to the C-bonded H atoms. The value All H-atom parameters refined applies to the two O-bonded H atoms.

Five reflections were regarded as damaged and were excluded from the refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.13320 (10)	0.85835 (13)	0.03853 (5)	0.0407 (3)
H81	−0.2133 (17)	0.819 (2)	0.0249 (8)	0.063 (5)*
O2	0.12953 (8)	0.78401 (14)	0.00365 (5)	0.0396 (3)
H82	0.1148 (16)	0.898 (3)	−0.0124 (10)	0.070 (6)*
C1	−0.06343 (12)	0.70624 (17)	0.06767 (6)	0.0296 (3)
H1	−0.0705	0.5979	0.0369	0.032 (3)*
C2	0.08021 (12)	0.76549 (18)	0.07061 (6)	0.0306 (3)
H2	0.0849	0.8892	0.0928	0.026 (3)*
C3	0.17267 (13)	0.63419 (18)	0.10705 (7)	0.0369 (4)
H31	0.2649	0.6714	0.0978	0.040 (4)*
H32	0.1601	0.5078	0.0891	0.040 (4)*
C4	0.15183 (14)	0.6295 (2)	0.18236 (8)	0.0431 (4)
H41	0.2372	0.5996	0.2038	0.051 (4)*
H42	0.1261	0.7549	0.1972	0.055 (5)*
C5	0.04863 (14)	0.4926 (2)	0.20740 (7)	0.0450 (4)
H51	0.0199	0.5317	0.2524	0.060 (5)*
H52	0.0907	0.3696	0.2118	0.053 (4)*
C6	−0.07353 (14)	0.4732 (2)	0.16324 (7)	0.0420 (4)
H61	−0.1446	0.4148	0.1898	0.057 (5)*
H62	−0.0521	0.3883	0.1261	0.049 (4)*
C7	−0.12639 (12)	0.65375 (19)	0.13391 (7)	0.0354 (3)
H71	−0.2229	0.6420	0.1276	0.048 (4)*
H72	−0.1110	0.7549	0.1662	0.044 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0333 (5)	0.0345 (6)	0.0544 (7)	−0.0006 (4)	−0.0094 (5)	0.0118 (4)
O2	0.0396 (6)	0.0378 (6)	0.0414 (6)	0.0049 (4)	0.0113 (4)	0.0086 (5)
C1	0.0299 (7)	0.0270 (7)	0.0320 (7)	0.0012 (5)	−0.0037 (5)	0.0006 (5)
C2	0.0301 (7)	0.0287 (7)	0.0331 (7)	0.0001 (5)	0.0031 (5)	−0.0001 (5)
C3	0.0290 (7)	0.0381 (8)	0.0436 (9)	0.0024 (6)	0.0005 (6)	0.0029 (6)

C4	0.0393 (8)	0.0484 (9)	0.0416 (9)	0.0003 (6)	−0.0098 (6)	0.0034 (7)
C5	0.0476 (8)	0.0488 (9)	0.0387 (9)	0.0023 (7)	−0.0023 (7)	0.0090 (7)
C6	0.0445 (8)	0.0429 (8)	0.0385 (8)	−0.0073 (6)	0.0001 (6)	0.0094 (6)
C7	0.0278 (7)	0.0429 (8)	0.0356 (8)	−0.0016 (6)	0.0008 (6)	−0.0004 (6)

Geometric parameters (Å, °)

O1—C1	1.4320 (15)	C4—C5	1.524 (2)
O1—H81	0.903 (18)	C4—H41	0.9900
O2—C2	1.4403 (15)	C4—H42	0.9900
O2—H82	0.90 (2)	C5—C6	1.529 (2)
C1—C2	1.5179 (18)	C5—H51	0.9900
C1—C7	1.5228 (18)	C5—H52	0.9900
C1—H1	1.0000	C6—C7	1.5288 (19)
C2—C3	1.5209 (18)	C6—H61	0.9900
C2—H2	1.0000	C6—H62	0.9900
C3—C4	1.527 (2)	C7—H71	0.9900
C3—H31	0.9900	C7—H72	0.9900
C3—H32	0.9900		

C1—O1—H81	108.9 (10)	C3—C4—H41	108.3
C2—O2—H82	111.2 (12)	C5—C4—H42	108.3
O1—C1—C2	105.79 (10)	C3—C4—H42	108.3
O1—C1—C7	109.96 (10)	H41—C4—H42	107.4
C2—C1—C7	115.96 (11)	C4—C5—C6	115.05 (12)
O1—C1—H1	108.3	C4—C5—H51	108.5
C2—C1—H1	108.3	C6—C5—H51	108.5
C7—C1—H1	108.3	C4—C5—H52	108.5
O2—C2—C1	108.81 (10)	C6—C5—H52	108.5
O2—C2—C3	107.05 (10)	H51—C5—H52	107.5
C1—C2—C3	115.65 (11)	C5—C6—C7	115.36 (12)
O2—C2—H2	108.4	C5—C6—H61	108.4
C1—C2—H2	108.4	C7—C6—H61	108.4
C3—C2—H2	108.4	C5—C6—H62	108.4
C2—C3—C4	113.89 (11)	C7—C6—H62	108.4
C2—C3—H31	108.8	H61—C6—H62	107.5
C4—C3—H31	108.8	C1—C7—C6	113.73 (11)
C2—C3—H32	108.8	C1—C7—H71	108.8
C4—C3—H32	108.8	C6—C7—H71	108.8
H31—C3—H32	107.7	C1—C7—H72	108.8
C5—C4—C3	115.84 (12)	C6—C7—H72	108.8
C5—C4—H41	108.3	H71—C7—H72	107.7

O1—C1—C2—O2	−66.09 (12)	C2—C3—C4—C5	87.55 (15)
C7—C1—C2—O2	171.75 (10)	C3—C4—C5—C6	−37.73 (18)
O1—C1—C2—C3	173.44 (10)	C4—C5—C6—C7	−40.52 (19)
C7—C1—C2—C3	51.27 (16)	O1—C1—C7—C6	170.39 (11)
O2—C2—C3—C4	168.11 (10)	C2—C1—C7—C6	−69.68 (15)
C1—C2—C3—C4	−70.47 (15)	C5—C6—C7—C1	88.08 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H81···O2^{i}	0.90 (2)	1.85 (2)	2.7481 (13)	174.7 (15)
O2—H82···O1^{ii}	0.90 (2)	1.84 (2)	2.7208 (14)	164.0 (15)

Symmetry codes: (i) $x-1/2$, $-y+3/2$, $-z$; (ii) $-x$, $-y+2$, $-z$.

Fig. 1

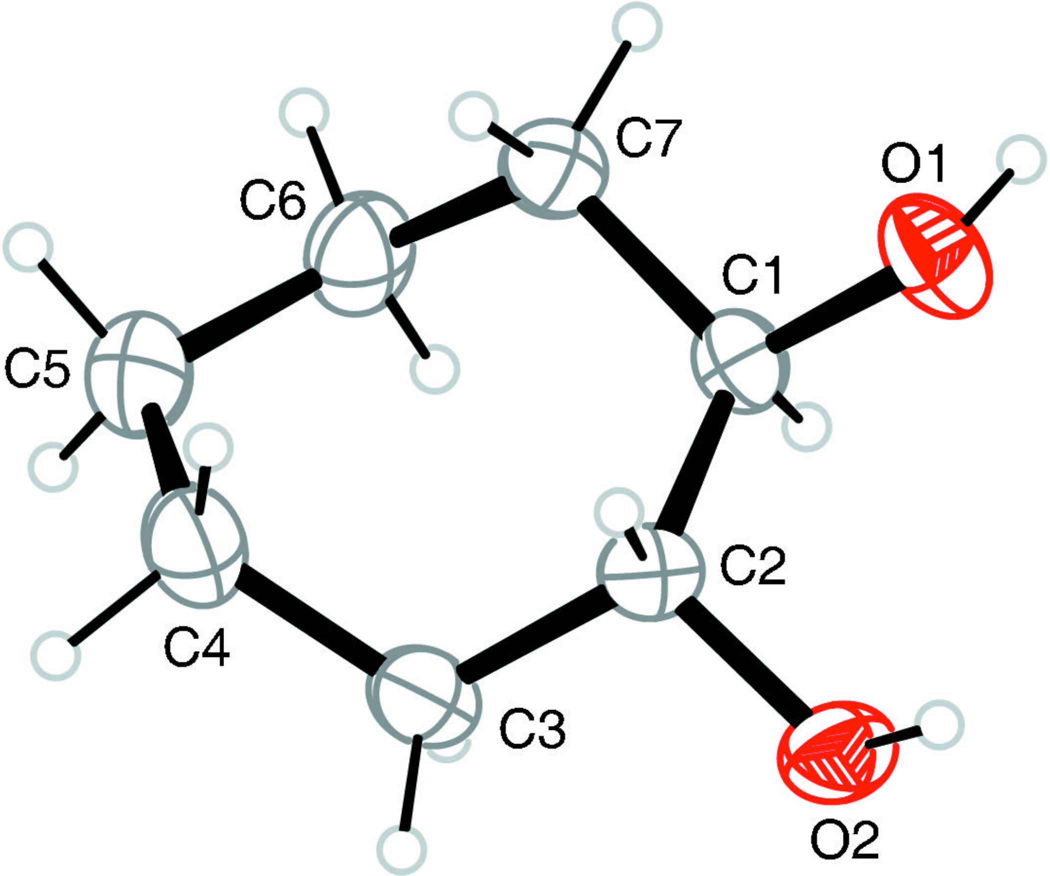

Fig. 2

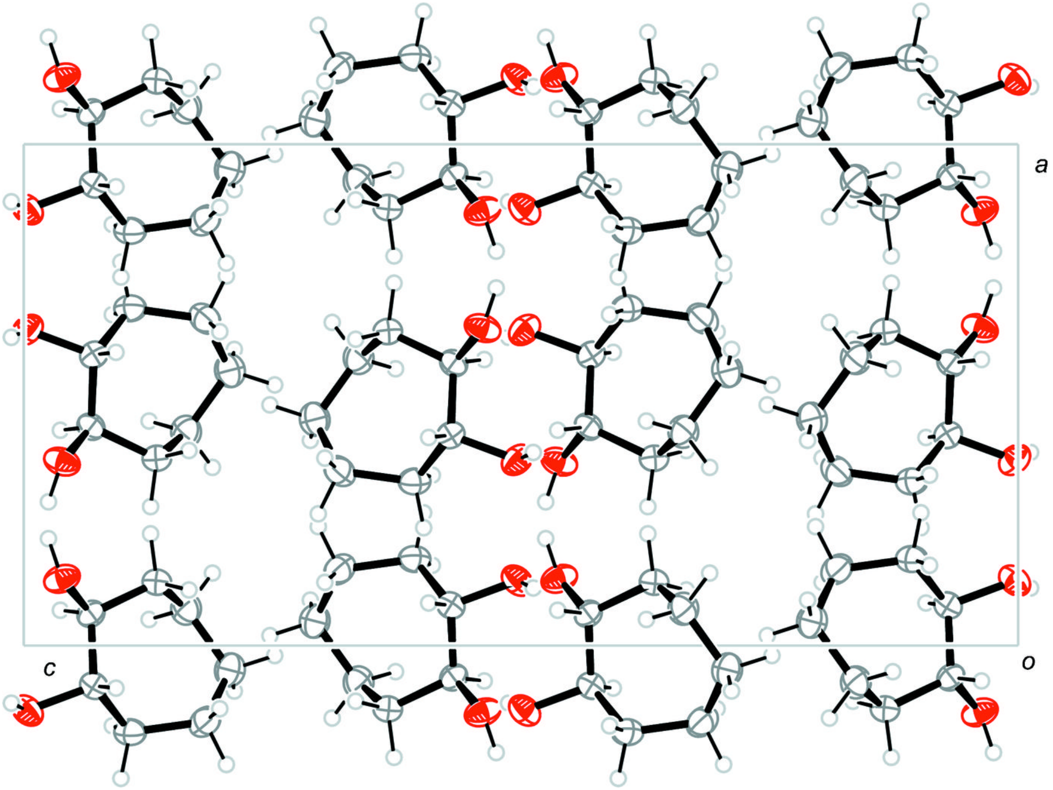

Fig. 3