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Bi-1,1′-cycloheptane-1,1′-diol

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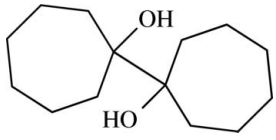
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.052; wR factor = 0.127; data-to-parameter ratio = 17.7.

The title compound, $C_{14}H_{26}O_2$, is a vicinal diol bearing two sterically demanding conformationally flexible cycloheptyl rings adjacent to the hydroxy groups. The molecule shows approximate non-crystallographic C_2 symmetry. Bond lengths and angles are normal. In the crystal structure, infinite chains are formed by alternating intra- and intermolecular hydrogen bonds between the hydroxy groups.

Related literature

For the synthesis of the title compound, see: Corey *et al.* (1976).



Experimental

Crystal data

$C_{14}H_{26}O_2$
M_r = 226.35
Orthorhombic, *Pbca*
a = 9.8750 (10) Å
b = 12.3662 (12) Å
c = 21.7093 (19) Å
V = 2651.1 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 200 (2) K
0.41 × 0.35 × 0.22 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
T_{min} = 0.980, T_{max} = 0.987
12976 measured reflections
2619 independent reflections
2208 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.127
S = 1.16
2619 reflections
148 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H801···O2^{i}	0.84	1.96	2.7889 (15)	168
O2—H802···O1	0.84	2.11	2.5856 (15)	116

Symmetry code: (i) $x-\frac{1}{2}, y, -z+\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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 Sandra Albrecht for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2371).

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
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  doi:10.1107/S1600536807058345

supplementary materials

Acta Cryst. (2007). E**63**, o4752 [**doi:10.1107/S1600536807058345**]

Bi-1,1'-cycloheptane-1,1'-diol

R. Betz and P. Klüfers

Comment

Bis-1,1'-cycloheptyl-1,1'-diol was prepared as a chelating molecule bearing the sterically demanding but conformationally flexible cycloheptyl groups. In the molecule two cycloheptyl groups are connected by a single bond. The two C atoms participating in the connection are further substituted with a hydroxy group each thus comprising a vicinal diol. Both cycloheptane rings adopt a chair-like conformation (Fig. 1). Bond lengths and angles are normal.

In the crystal packing (Fig. 2), one-dimensional chains of hydrogen-bonded molecules (Fig. 3) are the dominant structural motif.

Experimental

The title compound was prepared in adoption of a published procedure (Corey *et al.*, 1976) upon pinacolic coupling of cycloheptanone.

Crystals suitable for X-ray analysis were obtained after recrystallization from boiling diethylether/*n*-pentane.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined.

Figures

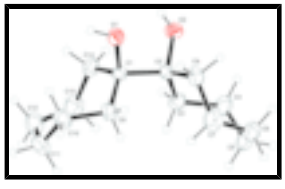

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

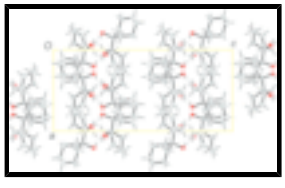

Fig. 2. The packing of (I), viewed along [0 1 0].

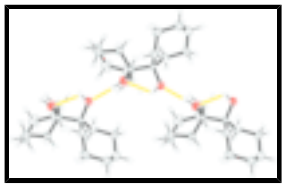

Fig. 3. An individual hydrogen-bonded chain.

Bi-1,1'-cycloheptane-1,1'-diol

Crystal data

$C_{14}H_{26}O_2$	F_{000} = 1008
M_r = 226.35	D_x = 1.134 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	θ = 3.9–26.0º
a = 9.8750 (10) Å	μ = 0.07 mm^{-1}
b = 12.3662 (12) Å	*T* = 200 (2) K
c = 21.7093 (19) Å	Block, colourless
V = 2651.1 (4) Å^3	0.41 × 0.35 × 0.22 mm
Z = 8	

Data collection

Nonius KappaCCD diffractometer	2619 independent reflections
Radiation source: fine-focus sealed tube	2208 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.051
T = 200(2) K	θ_{max} = 26.0º
ω–scans	θ_{min} = 4.3º
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	*h* = −11→12
T_{min} = 0.980, T_{max} = 0.987	*k* = −13→15
12976 measured reflections	*l* = −26→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.052	Only H-atom displacement parameters refined
$wR(F^2)$ = 0.127	$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 0.4645P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.16	$(\Delta/\sigma)_{max} < 0.001$
2619 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
148 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.05372 (10)	0.31065 (9)	0.21196 (5)	0.0340 (3)
H801	−0.1253	0.3298	0.2298	0.0460 (10)*

O2	0.20358 (10)	0.34212 (10)	0.22483 (5)	0.0351 (3)
H802	0.1460	0.3084	0.2459	0.0460 (10)*
C1	−0.00279 (14)	0.39937 (11)	0.17517 (7)	0.0241 (3)
C14	−0.01382 (16)	0.50382 (12)	0.21354 (7)	0.0315 (4)
H141	−0.0933	0.4965	0.2410	0.0460 (10)*
H142	0.0673	0.5083	0.2402	0.0460 (10)*
C13	−0.02705 (18)	0.61143 (13)	0.17922 (9)	0.0401 (4)
H131	0.0110	0.6697	0.2053	0.0460 (10)*
H132	0.0276	0.6079	0.1411	0.0460 (10)*
C12	−0.17232 (19)	0.64067 (14)	0.16229 (10)	0.0471 (5)
H121	−0.2296	0.6327	0.1994	0.0460 (10)*
H122	−0.1749	0.7177	0.1500	0.0460 (10)*
C11	−0.23341 (19)	0.57329 (15)	0.11070 (9)	0.0458 (5)
H111	−0.1862	0.5917	0.0719	0.0460 (10)*
H112	−0.3295	0.5944	0.1058	0.0460 (10)*
C10	−0.22698 (16)	0.45112 (14)	0.11944 (8)	0.0373 (4)
H101	−0.2846	0.4163	0.0879	0.0460 (10)*
H102	−0.2651	0.4329	0.1603	0.0460 (10)*
C9	−0.08306 (15)	0.40386 (13)	0.11493 (7)	0.0301 (4)
H91	−0.0897	0.3296	0.0982	0.0460 (10)*
H92	−0.0310	0.4475	0.0849	0.0460 (10)*
C2	0.14850 (14)	0.36422 (12)	0.16421 (7)	0.0254 (3)
C3	0.23549 (15)	0.45530 (13)	0.13821 (7)	0.0302 (4)
H31	0.2413	0.5133	0.1695	0.0460 (10)*
H32	0.1887	0.4858	0.1018	0.0460 (10)*
C4	0.37994 (16)	0.42418 (15)	0.11911 (8)	0.0394 (4)
H41	0.4402	0.4872	0.1255	0.0460 (10)*
H42	0.4122	0.3652	0.1462	0.0460 (10)*
C5	0.39112 (18)	0.38754 (17)	0.05225 (8)	0.0470 (5)
H51	0.3473	0.4426	0.0258	0.0460 (10)*
H52	0.4882	0.3851	0.0409	0.0460 (10)*
C6	0.32847 (18)	0.27796 (16)	0.03777 (8)	0.0466 (5)
H61	0.3829	0.2213	0.0584	0.0460 (10)*
H62	0.3350	0.2655	−0.0072	0.0460 (10)*
C7	0.18045 (18)	0.26413 (15)	0.05714 (8)	0.0422 (4)
H71	0.1445	0.1972	0.0382	0.0460 (10)*
H72	0.1273	0.3257	0.0408	0.0460 (10)*
C8	0.15943 (17)	0.25790 (12)	0.12723 (8)	0.0342 (4)
H81	0.2355	0.2157	0.1447	0.0460 (10)*
H82	0.0758	0.2159	0.1348	0.0460 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0257 (6)	0.0342 (6)	0.0421 (7)	0.0008 (4)	0.0071 (5)	0.0105 (5)
O2	0.0247 (6)	0.0522 (7)	0.0285 (6)	−0.0008 (5)	−0.0035 (5)	0.0092 (5)
C1	0.0226 (7)	0.0252 (7)	0.0245 (7)	−0.0016 (6)	0.0009 (6)	0.0027 (6)
C14	0.0293 (8)	0.0370 (9)	0.0283 (8)	0.0006 (7)	0.0029 (6)	−0.0067 (7)

C13	0.0451 (10)	0.0283 (9)	0.0470 (10)	−0.0026 (7)	0.0071 (8)	−0.0079 (7)
C12	0.0503 (11)	0.0298 (9)	0.0611 (12)	0.0112 (8)	0.0109 (10)	0.0040 (8)
C11	0.0373 (10)	0.0494 (11)	0.0507 (11)	0.0142 (8)	−0.0017 (8)	0.0132 (9)
C10	0.0271 (9)	0.0443 (10)	0.0406 (9)	0.0032 (7)	−0.0076 (7)	0.0001 (8)
C9	0.0263 (8)	0.0342 (8)	0.0297 (8)	0.0016 (6)	−0.0048 (6)	−0.0039 (6)
C2	0.0229 (7)	0.0300 (8)	0.0233 (7)	0.0013 (6)	−0.0022 (6)	0.0017 (6)
C3	0.0247 (8)	0.0352 (9)	0.0308 (8)	−0.0045 (6)	0.0011 (6)	−0.0005 (7)
C4	0.0247 (8)	0.0541 (11)	0.0395 (10)	−0.0028 (7)	0.0041 (7)	0.0007 (8)
C5	0.0313 (9)	0.0723 (13)	0.0372 (10)	0.0043 (9)	0.0090 (8)	0.0032 (9)
C6	0.0440 (10)	0.0637 (12)	0.0321 (9)	0.0191 (9)	0.0039 (8)	−0.0071 (8)
C7	0.0415 (10)	0.0457 (10)	0.0395 (10)	0.0072 (8)	−0.0042 (8)	−0.0147 (8)
C8	0.0300 (8)	0.0293 (8)	0.0432 (10)	0.0036 (6)	0.0013 (7)	−0.0005 (7)

Geometric parameters (Å, °)

O1—C1	1.4472 (17)	C9—H91	0.9900
O1—H801	0.8400	C9—H92	0.9900
O2—C2	1.4501 (17)	C2—C3	1.525 (2)
O2—H802	0.8400	C2—C8	1.544 (2)
C1—C9	1.530 (2)	C3—C4	1.534 (2)
C1—C14	1.541 (2)	C3—H31	0.9900
C1—C2	1.574 (2)	C3—H32	0.9900
C14—C13	1.531 (2)	C4—C5	1.525 (2)
C14—H141	0.9900	C4—H41	0.9900
C14—H142	0.9900	C4—H42	0.9900
C13—C12	1.524 (3)	C5—C6	1.522 (3)
C13—H131	0.9900	C5—H51	0.9900
C13—H132	0.9900	C5—H52	0.9900
C12—C11	1.521 (3)	C6—C7	1.531 (3)
C12—H121	0.9900	C6—H61	0.9900
C12—H122	0.9900	C6—H62	0.9900
C11—C10	1.524 (3)	C7—C8	1.538 (2)
C11—H111	0.9900	C7—H71	0.9900
C11—H112	0.9900	C7—H72	0.9900
C10—C9	1.540 (2)	C8—H81	0.9900
C10—H101	0.9900	C8—H82	0.9900
C10—H102	0.9900		
C1—O1—H801	109.5	H91—C9—H92	107.4
C2—O2—H802	109.5	O2—C2—C3	105.30 (11)
O1—C1—C9	108.61 (12)	O2—C2—C8	106.56 (12)
O1—C1—C14	108.21 (12)	C3—C2—C8	113.40 (12)
C9—C1—C14	113.26 (12)	O2—C2—C1	105.72 (11)
O1—C1—C2	101.77 (11)	C3—C2—C1	112.75 (12)
C9—C1—C2	111.87 (12)	C8—C2—C1	112.34 (12)
C14—C1—C2	112.35 (12)	C2—C3—C4	116.00 (13)
C13—C14—C1	118.14 (13)	C2—C3—H31	108.3
C13—C14—H141	107.8	C4—C3—H31	108.3
C1—C14—H141	107.8	C2—C3—H32	108.3
C13—C14—H142	107.8	C4—C3—H32	108.3

C1—C14—H142	107.8	H31—C3—H32	107.4
H141—C14—H142	107.1	C5—C4—C3	113.52 (14)
C12—C13—C14	113.82 (14)	C5—C4—H41	108.9
C12—C13—H131	108.8	C3—C4—H41	108.9
C14—C13—H131	108.8	C5—C4—H42	108.9
C12—C13—H132	108.8	C3—C4—H42	108.9
C14—C13—H132	108.8	H41—C4—H42	107.7
H131—C13—H132	107.7	C6—C5—C4	115.57 (15)
C11—C12—C13	114.89 (14)	C6—C5—H51	108.4
C11—C12—H121	108.5	C4—C5—H51	108.4
C13—C12—H121	108.5	C6—C5—H52	108.4
C11—C12—H122	108.5	C4—C5—H52	108.4
C13—C12—H122	108.5	H51—C5—H52	107.4
H121—C12—H122	107.5	C5—C6—C7	115.52 (15)
C12—C11—C10	115.78 (15)	C5—C6—H61	108.4
C12—C11—H111	108.3	C7—C6—H61	108.4
C10—C11—H111	108.3	C5—C6—H62	108.4
C12—C11—H112	108.3	C7—C6—H62	108.4
C10—C11—H112	108.3	H61—C6—H62	107.5
H111—C11—H112	107.4	C6—C7—C8	113.98 (14)
C11—C10—C9	114.00 (14)	C6—C7—H71	108.8
C11—C10—H101	108.8	C8—C7—H71	108.8
C9—C10—H101	108.8	C6—C7—H72	108.8
C11—C10—H102	108.8	C8—C7—H72	108.8
C9—C10—H102	108.8	H71—C7—H72	107.7
H101—C10—H102	107.6	C7—C8—C2	118.76 (13)
C1—C9—C10	115.95 (13)	C7—C8—H81	107.6
C1—C9—H91	108.3	C2—C8—H81	107.6
C10—C9—H91	108.3	C7—C8—H82	107.6
C1—C9—H92	108.3	C2—C8—H82	107.6
C10—C9—H92	108.3	H81—C8—H82	107.1
O1—C1—C14—C13	−153.65 (13)	C14—C1—C2—C3	−51.89 (16)
C9—C1—C14—C13	−33.17 (19)	O1—C1—C2—C8	62.95 (14)
C2—C1—C14—C13	94.79 (16)	C9—C1—C2—C8	−52.83 (16)
C1—C14—C13—C12	86.06 (18)	C14—C1—C2—C8	178.48 (12)
C14—C13—C12—C11	−72.2 (2)	O2—C2—C3—C4	72.10 (16)
C13—C12—C11—C10	53.4 (2)	C8—C2—C3—C4	−44.03 (18)
C12—C11—C10—C9	−69.5 (2)	C1—C2—C3—C4	−173.12 (13)
O1—C1—C9—C10	75.89 (16)	C2—C3—C4—C5	89.91 (18)
C14—C1—C9—C10	−44.36 (18)	C3—C4—C5—C6	−70.9 (2)
C2—C1—C9—C10	−172.58 (13)	C4—C5—C6—C7	53.6 (2)
C11—C10—C9—C1	89.02 (18)	C5—C6—C7—C8	−70.2 (2)
O1—C1—C2—O2	−52.88 (13)	C6—C7—C8—C2	84.19 (19)
C9—C1—C2—O2	−168.66 (11)	O2—C2—C8—C7	−148.94 (14)
C14—C1—C2—O2	62.64 (15)	C3—C2—C8—C7	−33.56 (19)
O1—C1—C2—C3	−167.42 (12)	C1—C2—C8—C7	95.73 (16)
C9—C1—C2—C3	76.80 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H801···O2^{i}	0.84	1.96	2.7889 (15)	168
O2—H802···O1	0.84	2.11	2.5856 (15)	116

Symmetry codes: (i) *x*−1/2, *y*, −*z*+1/2.

Fig. 1

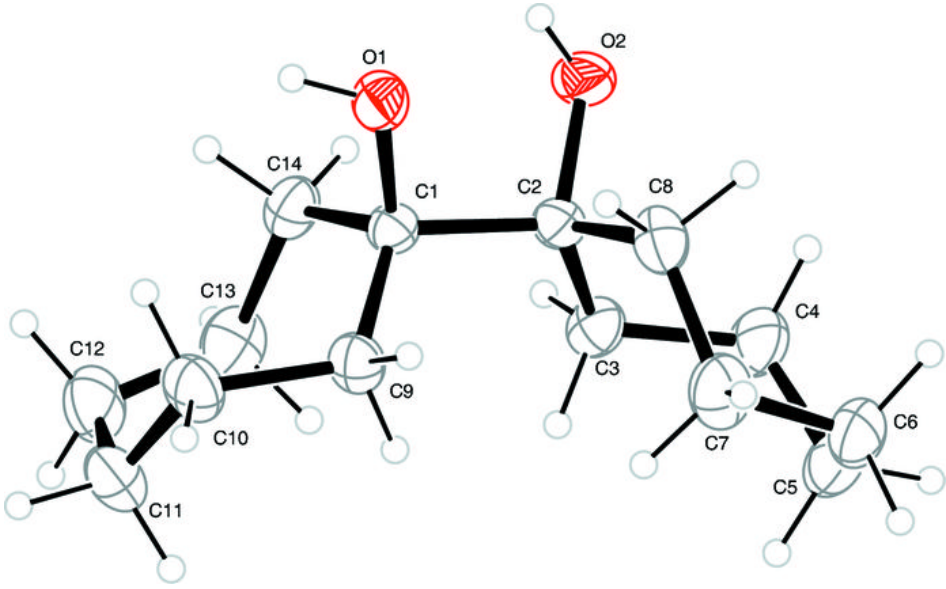

Fig. 2

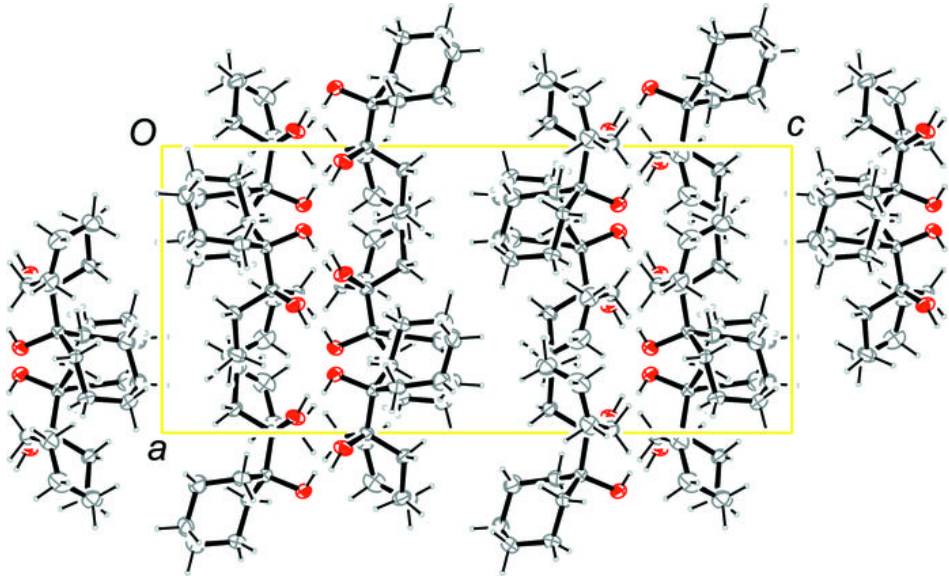

Fig. 3