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1,2-Dicyclopentylethane-1,2-diol

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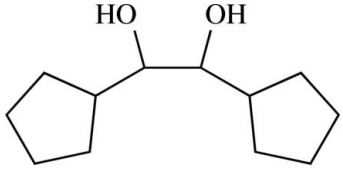
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.060; wR factor = 0.185; data-to-parameter ratio = 20.0.

The title compound, $C_{12}H_{22}O_2$, was accidentally prepared as a sterically demanding vicinal diol in the attempted preparation of dicyclopentylglycolic acid. The cyclopentyl rings adopt envelope conformations. Intermolecular hydrogen bonds are present in the structure.

Related literature

The title compound was accidentally prepared in the attempted preparation of dicyclopentylglycolic acid by analogy with a literature procedure (Gauerke & Marvel, 1928).



Experimental

Crystal data

$C_{12}H_{22}O_2$
M_r = 198.30
Monoclinic, $C2/c$
a = 22.5500 (7) Å
b = 5.6290 (1) Å
c = 17.9416 (6) Å
β = 91.255 (2)°
V = 2276.85 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 200 (2) K
0.27 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
4812 measured reflections
2604 independent reflections
1881 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.185
S = 1.06
2604 reflections
130 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.84	2.00	2.8218 (16)	168
O2—H2···O1^{ii}	0.84	1.96	2.7656 (16)	162

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

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; 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 Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2290).

References

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Gauerke, C. G. & Marvel, C. S. (1928). *J. Am. Chem. Soc.* **50**, 1178–1182.
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supplementary materials

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1,2-Dicyclopentylethane-1,2-diol

R. Betz, S. Herdlicka and P. Klüfers

Comment

The title compound, $C_{12}H_{22}O_2$, was accidentally prepared as sterically demanding vicinal diol on the attempted preparation of dicyclopentylglycolic acid. The cyclopentyl rings adopt envelope conformations. Intermolecular hydrogen bonds are present in the structure. It was obtained as high-boiling by-product from the reaction between cyclopentyl magnesium bromide and diethyloxalate followed by aqueous workup.

The molecular structure is shown in Fig. 1. Intermolecular hydrogen bonds between the hydroxyl-groups are present in the structure (Fig. 2).

Experimental

The title compound was accidentially prepared on the attempted preparation of dicyclopentylglycolic acid in analogy to a literature procedure (Gauerke & Marvel, 1928) by reaction of cyclopentyl magnesium bromide with diethyloxalate followed by aqueous workup. It was obtained as high-boiling fraction crystallizing upon storage at room temperature.

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 to U_{iso}(H) = 0.085 (2).

Figures

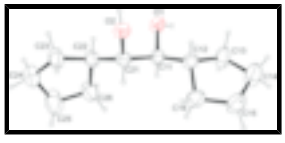

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

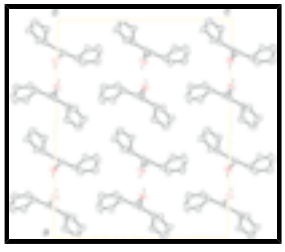

Fig. 2. The packing of (I), viewed along [$\bar{1}$ 0 0]. H atoms omitted for clarity except for the O bonded H atoms.

1,2-Dicyclopentylethane-1,2-diol

Crystal data

$C_{12}H_{22}O_2$	F_{000} = 880
M_r = 198.30	D_x = 1.157 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 12652 reflections
a = 22.5500 (7) Å	θ = 3.1–27.5º
b = 5.62900 (10) Å	μ = 0.08 mm^{-1}
c = 17.9416 (6) Å	*T* = 200 (2) K
β = 91.255 (2)º	Rod, colourless
V = 2276.85 (11) Å^3	0.27 × 0.08 × 0.06 mm
Z = 8	

Data collection

Nonius KappaCCD diffractometer	1881 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.030
Monochromator: MONTEL, graded multilayered X-ray optics	θ_{max} = 27.5º
T = 200(2) K	θ_{min} = 3.6º
φ/ω–scan	*h* = −28→28
Absorption correction: none	*k* = −7→6
4812 measured reflections	*l* = −23→23
2604 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
$R[F^2 > 2\sigma(F^2)]$ = 0.060	Only H-atom displacement parameters refined
$wR(F^2)$ = 0.185	$w = 1/[\sigma^2(F_o^2) + (0.0927P)^2 + 1.7562P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
2604 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
130 parameters	$\Delta\rho_{min}$ = −0.46 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. 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.19267 (5)	0.9503 (2)	−0.00195 (8)	0.0370 (4)
H1	0.1863	1.0961	−0.0082	0.085 (2)*
O2	0.18742 (5)	0.4504 (2)	−0.01071 (7)	0.0346 (3)
H2	0.2216	0.5098	−0.0109	0.085 (2)*
C11	0.13749 (7)	0.8286 (3)	0.00517 (10)	0.0316 (4)
H11	0.1127	0.9189	0.0410	0.085 (2)*
C12	0.10422 (8)	0.8168 (3)	−0.06917 (11)	0.0356 (4)
H12	0.1275	0.7180	−0.1044	0.085 (2)*
C13	0.09340 (10)	1.0604 (4)	−0.10395 (14)	0.0561 (6)
H131	0.0823	1.1771	−0.0654	0.085 (2)*
H132	0.1295	1.1177	−0.1288	0.085 (2)*
C14	0.04305 (11)	1.0267 (4)	−0.16023 (15)	0.0632 (7)
H141	0.0587	0.9963	−0.2105	0.085 (2)*
H142	0.0176	1.1699	−0.1625	0.085 (2)*
C15	0.00808 (10)	0.8134 (5)	−0.13313 (15)	0.0594 (6)
H151	−0.0325	0.8621	−0.1197	0.085 (2)*
H152	0.0051	0.6910	−0.1727	0.085 (2)*
C16	0.04134 (9)	0.7162 (4)	−0.06535 (13)	0.0481 (5)
H161	0.0421	0.5404	−0.0666	0.085 (2)*
H162	0.0222	0.7678	−0.0189	0.085 (2)*
C21	0.15098 (8)	0.5835 (3)	0.03865 (10)	0.0335 (4)
H21	0.1123	0.4974	0.0417	0.085 (2)*
C22	0.17734 (9)	0.5939 (3)	0.11726 (11)	0.0402 (5)
H22	0.2130	0.6995	0.1175	0.085 (2)*
C23	0.19545 (12)	0.3478 (4)	0.14780 (12)	0.0555 (6)
H231	0.1688	0.2227	0.1275	0.085 (2)*
H232	0.2368	0.3095	0.1346	0.085 (2)*
C24	0.18967 (11)	0.3682 (4)	0.23172 (12)	0.0559 (6)
H241	0.1821	0.2111	0.2544	0.085 (2)*
H242	0.2259	0.4374	0.2550	0.085 (2)*
C25	0.13821 (17)	0.5284 (7)	0.24015 (15)	0.0936 (12)
H251	0.1015	0.4333	0.2445	0.085 (2)*
H252	0.1434	0.6239	0.2862	0.085 (2)*

C26	0.13290 (11)	0.6878 (4)	0.17485 (12)	0.0518 (6)
H261	0.1428	0.8532	0.1892	0.085 (2)*
H262	0.0920	0.6845	0.1538	0.085 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0323 (6)	0.0247 (6)	0.0538 (8)	−0.0004 (5)	−0.0027 (5)	0.0035 (5)
O2	0.0353 (6)	0.0234 (6)	0.0452 (8)	0.0019 (5)	0.0049 (5)	−0.0019 (5)
C11	0.0305 (8)	0.0252 (8)	0.0391 (10)	0.0024 (6)	0.0036 (7)	−0.0001 (7)
C12	0.0335 (9)	0.0310 (9)	0.0422 (10)	0.0019 (7)	0.0000 (7)	0.0010 (7)
C13	0.0566 (13)	0.0442 (12)	0.0665 (15)	−0.0093 (10)	−0.0210 (11)	0.0201 (11)
C14	0.0619 (14)	0.0542 (13)	0.0721 (17)	−0.0039 (11)	−0.0278 (12)	0.0120 (12)
C15	0.0421 (11)	0.0650 (15)	0.0704 (16)	−0.0022 (10)	−0.0122 (10)	0.0034 (12)
C16	0.0387 (10)	0.0393 (10)	0.0661 (14)	−0.0053 (8)	−0.0039 (9)	0.0039 (9)
C21	0.0333 (9)	0.0262 (8)	0.0413 (10)	0.0024 (7)	0.0069 (7)	0.0026 (7)
C22	0.0463 (10)	0.0350 (9)	0.0394 (11)	0.0041 (8)	0.0039 (8)	0.0050 (8)
C23	0.0762 (15)	0.0459 (12)	0.0447 (12)	0.0224 (11)	0.0076 (10)	0.0126 (9)
C24	0.0754 (15)	0.0497 (12)	0.0421 (12)	0.0032 (11)	−0.0056 (10)	0.0060 (10)
C25	0.124 (3)	0.109 (3)	0.0483 (15)	0.059 (2)	0.0312 (16)	0.0249 (16)
C26	0.0691 (14)	0.0417 (11)	0.0451 (12)	0.0119 (10)	0.0118 (10)	0.0035 (9)

Geometric parameters (Å, °)

O1—C11	1.428 (2)	C16—H161	0.9900
O1—H1	0.8400	C16—H162	0.9900
O2—C21	1.433 (2)	C21—C22	1.520 (3)
O2—H2	0.8400	C21—H21	1.0000
C11—C12	1.517 (2)	C22—C23	1.541 (3)
C11—C21	1.533 (2)	C22—C26	1.548 (3)
C11—H11	1.0000	C22—H22	1.0000
C12—C13	1.524 (3)	C23—C24	1.518 (3)
C12—C16	1.530 (3)	C23—H231	0.9900
C12—H12	1.0000	C23—H232	0.9900
C13—C14	1.515 (3)	C24—C25	1.480 (4)
C13—H131	0.9900	C24—H241	0.9900
C13—H132	0.9900	C24—H242	0.9900
C14—C15	1.522 (3)	C25—C26	1.479 (3)
C14—H141	0.9900	C25—H251	0.9900
C14—H142	0.9900	C25—H252	0.9900
C15—C16	1.517 (3)	C26—H261	0.9900
C15—H151	0.9900	C26—H262	0.9900
C15—H152	0.9900		
C11—O1—H1	109.5	H161—C16—H162	108.7
C21—O2—H2	109.5	O2—C21—C22	112.06 (14)
O1—C11—C12	110.94 (14)	O2—C21—C11	109.88 (13)
O1—C11—C21	107.51 (13)	C22—C21—C11	113.56 (15)
C12—C11—C21	113.26 (14)	O2—C21—H21	107.0

O1—C11—H11	108.3	C22—C21—H21	107.0
C12—C11—H11	108.3	C11—C21—H21	107.0
C21—C11—H11	108.3	C21—C22—C23	113.01 (17)
C11—C12—C13	113.06 (15)	C21—C22—C26	112.74 (16)
C11—C12—C16	114.70 (16)	C23—C22—C26	103.89 (16)
C13—C12—C16	102.19 (15)	C21—C22—H22	109.0
C11—C12—H12	108.9	C23—C22—H22	109.0
C13—C12—H12	108.9	C26—C22—H22	109.0
C16—C12—H12	108.9	C24—C23—C22	104.89 (17)
C14—C13—C12	105.73 (17)	C24—C23—H231	110.8
C14—C13—H131	110.6	C22—C23—H231	110.8
C12—C13—H131	110.6	C24—C23—H232	110.8
C14—C13—H132	110.6	C22—C23—H232	110.8
C12—C13—H132	110.6	H231—C23—H232	108.8
H131—C13—H132	108.7	C25—C24—C23	103.41 (19)
C13—C14—C15	105.77 (19)	C25—C24—H241	111.1
C13—C14—H141	110.6	C23—C24—H241	111.1
C15—C14—H141	110.6	C25—C24—H242	111.1
C13—C14—H142	110.6	C23—C24—H242	111.1
C15—C14—H142	110.6	H241—C24—H242	109.0
H141—C14—H142	108.7	C26—C25—C24	109.8 (2)
C16—C15—C14	106.86 (18)	C26—C25—H251	109.7
C16—C15—H151	110.3	C24—C25—H251	109.7
C14—C15—H151	110.3	C26—C25—H252	109.7
C16—C15—H152	110.3	C24—C25—H252	109.7
C14—C15—H152	110.3	H251—C25—H252	108.2
H151—C15—H152	108.6	C25—C26—C22	106.20 (18)
C15—C16—C12	105.86 (17)	C25—C26—H261	110.5
C15—C16—H161	110.6	C22—C26—H261	110.5
C12—C16—H161	110.6	C25—C26—H262	110.5
C15—C16—H162	110.6	C22—C26—H262	110.5
C12—C16—H162	110.6	H261—C26—H262	108.7
O1—C11—C12—C13	−56.3 (2)	O1—C11—C21—C22	64.27 (18)
C21—C11—C12—C13	−177.36 (16)	C12—C11—C21—C22	−172.80 (14)
O1—C11—C12—C16	−172.99 (14)	O2—C21—C22—C23	−50.0 (2)
C21—C11—C12—C16	65.99 (19)	C11—C21—C22—C23	−175.26 (16)
C11—C12—C13—C14	−160.39 (19)	O2—C21—C22—C26	−167.46 (15)
C16—C12—C13—C14	−36.6 (2)	C11—C21—C22—C26	67.3 (2)
C12—C13—C14—C15	25.7 (3)	C21—C22—C23—C24	−151.59 (18)
C13—C14—C15—C16	−4.3 (3)	C26—C22—C23—C24	−29.1 (2)
C14—C15—C16—C12	−18.6 (3)	C22—C23—C24—C25	34.7 (3)
C11—C12—C16—C15	156.42 (17)	C23—C24—C25—C26	−27.6 (4)
C13—C12—C16—C15	33.7 (2)	C24—C25—C26—C22	9.4 (4)
O1—C11—C21—O2	−62.12 (17)	C21—C22—C26—C25	135.1 (2)
C12—C11—C21—O2	60.81 (18)	C23—C22—C26—C25	12.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O1—H1···O2^{i}	0.84	2.00	2.8218 (16)	168
O2—H2···O1^{ii}	0.84	1.96	2.7656 (16)	162

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

Fig. 1

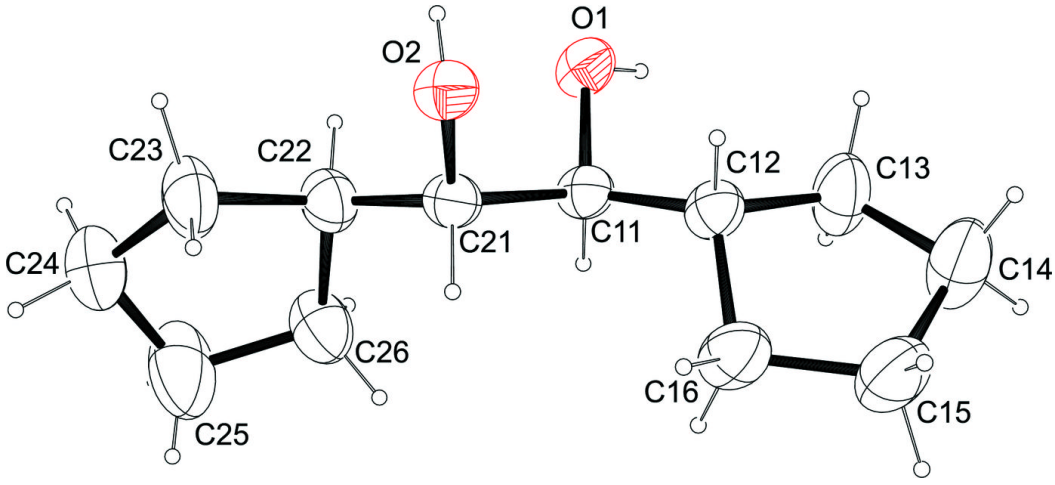

Fig. 2