$(\Delta/\sigma)_{\max} = 0.095$
$\Delta\rho_{\max} = 0.408$ e Å^{-3}
$\Delta\rho_{\min} = -0.400$ e Å^{-3}

Absolute structure: assigned according to Shubina *et al.* (1990)

Table 1. *Selected geometric parameters (Å, °)*

C2—C3	1.537 (3)	C2′—C3′	1.502 (3)
C9—C10	1.584 (2)	C9′—C10′	1.574 (2)
C9—C15	1.573 (3)	C9′—C15′	1.580 (3)
C18—C17—C16	123.9 (2)	C18′—C17′—C16′	124.7 (2)
C18—C17—O17	117.6 (2)	C18′—C17′—O17′	119.1 (2)
C16—C17—O17	118.5 (2)	C16′—C17′—O17′	116.0 (2)
C24—O20—C20	119.1 (1)	C24′—O20′—C20′	116.3 (1)
C8—C9—C15—C16	56.0 (2)		
C9—C15—C16—C17	−99.8 (2)		
C18—C17—O17—C22	73.4 (2)		
C16—C17—O17—C22	−109.5 (2)		
C21—C20—O20—C24	128.3 (2)		
C19—C20—O20—C24	−54.3 (3)		
C8′—C9′—C15′—C16′	59.8 (2)		
C9′—C15′—C16′—C17′	−95.9 (2)		
C18′—C17′—O17′—C22′	61.2 (3)		
C16′—C17′—O17′—C22′	−122.7 (2)		
C21′—C20′—O20′—C24′	116.0 (2)		
C19′—C20′—O20′—C24′	−67.4 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O17	0.96	2.84	3.343 (2)	114
C13—H13*A*···O17	0.96	2.46	3.124 (3)	126
C15—H15*B*···O17	0.96	2.49	2.891 (2)	105
C15—H15*B*···O24′	0.96	2.77	3.318 (2)	117
C19—H19···O24	1.15 (2)	2.48 (2)	2.939 (3)	102 (1)
C8′—H8*B*···O17′	0.96	2.65	3.193 (3)	116
C13′—H13*E*···O17′	0.96	2.37	3.049 (3)	128
C15′—H15*C*···O17′	0.96	2.48	2.858 (2)	103
C18′—H18′···O24	1.07 (2)	2.34 (2)	3.380 (2)	163 (1)
C18′—H18′···O22′	1.07 (2)	2.81 (2)	2.952 (2)	86.7 (9)
C19′—H19′···O22	0.95 (2)	2.47 (2)	3.345 (3)	153 (1)
C19′—H19′···O24′	0.95 (2)	3.00 (2)	3.029 (3)	83 (1)
C18—H18···O24′^{i}	0.90 (2)	2.52 (2)	3.353 (2)	155 (1)
C19—H19···O22′^{i}	1.15 (2)	2.23 (2)	3.320 (3)	158 (2)

Symmetry code: (i) $x - 1, y, z$.

The H atoms were located on the basis of geometrical considerations and difference Fourier map suggestions (for the methyl groups). Atoms H18, H19, H18′ and H19′ were included in the refinement with isotropic displacement parameters. All other H atoms were included as fixed atoms, with isotropic displacement parameters set equal to $1.2U_{eq}$ ($1.5U_{eq}$ for methyl groups) of the parent atoms. The absolute configuration was assigned in accordance with that established previously by Shubina *et al.* (1990).

Data collection: *P*2$_1$ *Diffractometer Program* (Syntex, 1975). Cell refinement: *P*2$_1$ *Diffractometer Program*. Data reduction: *XDISK* (*SHELXTL/PC*; Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* in *SHELXL*93.

The authors are grateful to Dr A. I. Yanovsky for his help in this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1024). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 268–270

2-Benzyl-2-hydroxycyclohexanone

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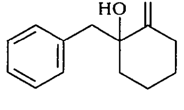
(*Received 30 September 1998; accepted 23 October 1998*)

Abstract

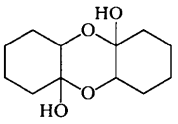
In the solid state, the title compound ($C_{13}H_{16}O_2$) exists as dimers related by a centre of inversion and linked by two equivalent hydrogen bonds O1···H2($-x$, $2 - y$, $1 - z$) [2.021 (3) Å]. The carbonyl- and hydroxyl-group O atoms attached to the cyclohexyl ring are almost eclipsed [O2—C2—C1—O1 18.5 (3)°].

Comment

Treatment of 2-hydroxycyclohexanone with sodium hydride and benzyl bromide in tetrahydrofuran afforded the highly crystalline title compound, (1), rather than the desired 2-benzyloxycyclohexanone. Similar C-alkylations of acyloins had been reported previously (van de Sande & Kopecky, 1969). It was of interest to investigate the crystal structure of (1) because 2-hydroxycyclohexanone and related compounds, although monomeric in solution, tend to form tricyclic dimers, (2), in the solid state (Jauch *et al.*, 1991). More highly substituted 2-hydroxycyclohexanone derivatives with phenyl groups in the 2- and 6-positions have, however, been found to be monomeric in the solid state (Choi *et al.*, 1996).



(1)



(2)

The crystal structure consists of discrete molecules of (1) which form dimeric units related by a crystallographic centre of inversion and linked by two equivalent intermolecular hydrogen bonds $O1\cdots H2(-x, 2-y, 1-z)$ [2.021 (3) Å]. The observed geometrical parameters generally fall within normal ranges.

The six-membered ring adopts a chair conformation which is slightly flattened at the carbonyl-C atom, C1, with the benzyl and hydroxyl groups at C2 in axial and equatorial positions, respectively. In this arrangement, the adjacent carbonyl- and hydroxyl-group O atoms are almost eclipsed [O2—C2—C1—O1 18.5 (3)°]. Although a sterically hindered benzyl group would normally be expected to be in an equatorial position to reduce 1,3-*syn* axial interactions, the alternative conformation of (1) is likely to be more destabilized by steric interactions between the benzylic methylene group and the carbonyl group.

cis-2-Hydroxy-2,6-diphenylcyclohexanone favours the alternative conformation where both phenyl substituents lie in equatorial positions and the hydroxyl group is axial (Choi *et al.*, 1996), but in *cis*-2,6-dihydroxy-2,6-diphenylcyclohexanone the situation is reversed with the phenyl groups axial and the hydroxyl groups equatorial (Choi *et al.*, 1995).

For the title compound, the formation of dimers stabilized by cyclic intermolecular hydrogen bonding probably favours the observed molecular conformation as noted in other systems containing both hydroxyl and carbonyl functional groups (Braga *et al.*, 1998).

Experimental

2-Hydroxycyclohexanone (5.0 g, 43.8 mmol) was added to a cooled (273 K) suspension of sodium hydride (1.85 g, 60% dispersion in oil, 46 mmol) in dry tetrahydrofuran (30 ml). After 15 min, a solution of benzyl bromide (7.5 g, 43.8 mmol) was added and the reaction mixture stirred at room temperature for 3 h. After quenching with water (50 ml), the resulting

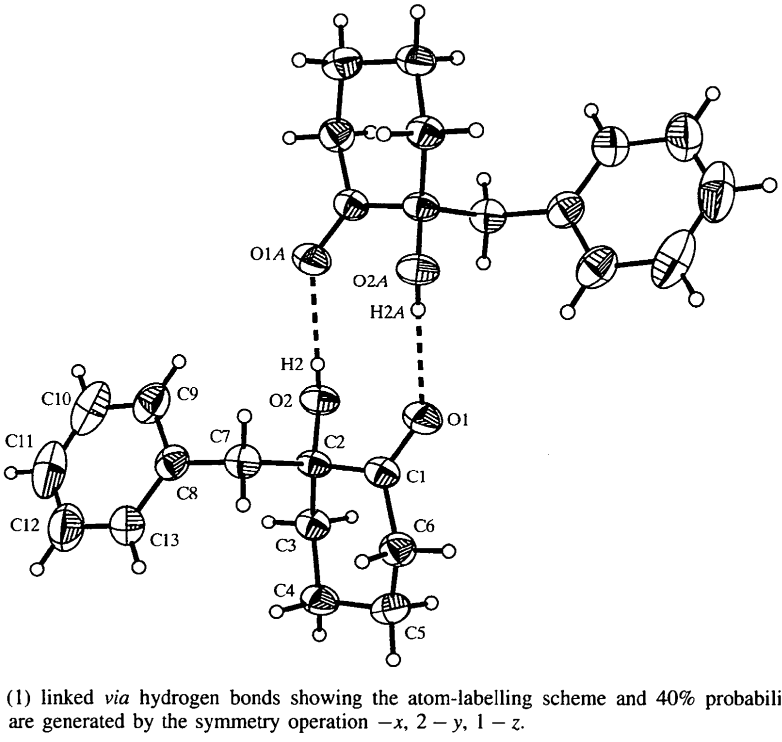


Fig. 1. A dimeric unit of (1) linked *via* hydrogen bonds showing the atom-labelling scheme and 40% probability displacement ellipsoids. Atoms with the suffix *A* are generated by the symmetry operation $-x, 2-y, 1-z$.

reaction mixture was extracted with dichloromethane (2 × 20 ml). The combined organic layers were dried over anhydrous magnesium sulfate and the solvent removed under reduced pressure. Recrystallization of the crude oily product from diethyl ether afforded 2-benzyl-2-hydroxycyclohexanone as colourless crystals (6.6 g, 32.4 mmol, 74%) [m.p. 349 K; ^{1}H NMR (200 MHz, $CDCl_3$, p.p.m.): δ = 1.55–1.96 (*m*, 4H), 2.09–2.28 (*m*, 2H), 2.46–2.80 (*m*, 2H), 2.96 (*d*, J = 14.5 Hz, 1H), 3.14 (*d*, *J* = 14.5 Hz, 1H), 3.83 (*br, s*, 1H), 7.16–7.32 (*m*, 5H); ^{13}C NMR (50 Mz, $CDCl_3$, p.p.m.): δ = 23.13, 28.49, 39.10, 40.89, 43.78, 79.78, 127.47, 127.74, 130.59, 135.86, 213.10].

Crystal data

$C_{13}H_{16}O_2$
$M_r = 204.26$
Orthorhombic
Pbca
$a = 10.857\ (3)$ Å
$b = 11.2892\ (9)$ Å
$c = 18.5162\ (9)$ Å
$V = 2269.5\ (6)$ Å^3
$Z = 8$
$D_x = 1.196$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 28 reflections
$\theta = 4.90$–$12.51°$
$\mu = 0.079$ mm^{-1}
$T = 293\ (2)$ K
Rectangular block
0.9 × 0.6 × 0.4 mm
Colourless

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
2575 measured reflections
1977 independent reflections
1297 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$

$\theta_{\rm max} = 25°$
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 22$
3 standard reflections every 97 reflections intensity decay: 10.75%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.135$
$S = 1.015$
1977 reflections
140 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.1676P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.007$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.221 (3)	C1—C2	1.528 (3)
C1—C6	1.496 (3)	O2—C2	1.420 (2)
O1—C1—C6	122.1 (2)	C3—C2—C1	109.35 (18)
O1—C1—C2	120.7 (2)	O2—C2—C7	110.54 (17)
C6—C1—C2	117.26 (19)	C3—C2—C7	113.91 (18)
O2—C2—C3	105.59 (16)	C1—C2—C7	106.93 (17)
O2—C2—C1	110.55 (17)	C1—C6—C5	112.5 (2)

The phenyl-H atoms were constrained to idealized positions (C—H 0.93 Å) as were the cyclohexyl-H atoms (C—H 0.97 Å) and hydroxyl-H atom (O—H 0.82 Å). The isotropic displacement parameters of all sets of H atoms were defined as $1.2U_{\rm iso}$ of the corresponding carrier atom.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to solve structure: *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

The use of the EPSRC Chemical Database Service at CLRC Daresbury Laboratory (Fletcher *et al.*, 1996; Allen & Kennard, 1993) is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1297). Services for accessing these data are described at the back of the journal.

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