

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-Benzylisochroman-1-one

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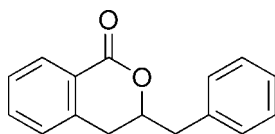
Received 22 January 2009; accepted 4 February 2009

Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.136; data-to-parameter ratio = 18.4.

In the molecule of the title compound, $\text{C}_{16}\text{H}_{14}\text{O}_2$, the aromatic rings are oriented at a dihedral angle of 78.49 (3)°. The heterocyclic ring adopts a twist conformation. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains along the c axis.

Related literature

For related structures, see: Schmalte *et al.* (1982); Schnebel *et al.* (2003). For a description of the Cambridge Structural Database, see: Allen (2002). For bond-length data, see: Allen *et al.* (1987). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{14}\text{O}_2$
 $M_r = 238.27$
Monoclinic, $P2_1/c$

$a = 12.503$ (5) Å
 $b = 8.0200$ (9) Å
 $c = 12.892$ (5) Å

$\beta = 102.43$ (2)°
 $V = 1262.4$ (7) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.08$ mm⁻¹
 $T = 294$ (2) K
 $0.32 \times 0.26 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
 $T_{\min} = 0.820$, $T_{\max} = 0.983$

7148 measured reflections
3024 independent reflections
2546 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.136$
 $S = 1.03$
3024 reflections

164 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9B}\cdots\text{O1}^i$	0.97	2.53	3.2922 (18)	135

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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: *SHELXTL* (Sheldrick, 2008).

The authors gratefully acknowledge the financial support of the Higher Education Commission, Islamabad, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2615).

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supplementary materials

Acta Cryst. (2009). E65, o498 [doi:10.1107/S1600536809004164]

3-Benzylisochroman-1-one

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Comment

The title compound was prepared in order to evaluate its potential as antibacterial and antifungal agents. The CCDC search (Allen, 2002) showed that the crystal structures of *rac-exo*-tricarboxyl-(h6-3-phenyl isochromanone) -chromium (Schnebel *et al.*, 2003) and 3,4-dihydro-8-hydroxy-3-(4-hydroxy- phenyl)-isocoumarin (Schmalle *et al.*, 1982) have been reported, which have close resemblance as far as isochromane and attached phenyl ring is considered. We report herein the synthesis and crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C1-C6) and C (C11-C16) are, of course, planar, and they are oriented at a dihedral angle of 78.49 (3)°. Ring B (O2/C5-C9) is not planar, having total puckering amplitude, Q_T , of 2.420 (3) Å and twisted conformation [$\varphi = 151.98$ (3)° and $\theta = 88.50$ (3)°] (Cremer & Pople, 1975).

In the crystal structure, intermolecular C-H...O hydrogen bonds (Table 1) link the molecules into chains along the *c* axis, in which they may be effective in the stabilization of the structure.

Experimental

As shown in Scheme 2, a mixture of homophthalic acid (1.98 g, 11.0 mmol) and 2-phenylacetyl chloride (7.08 g, 46 mmol) was heated under reflux for 6 h at 473 K. After concentration, the residue was chromatographed on silica gel column using petroleum ether (333–353 K) to give 3-benzyl-1*H*-isochromen-1-one. 2-(2-oxo-3-phenylpropyl) benzoic acid was obtained by refluxing a solution of 3-benzyl-1*H*-isochromen-1-one (4 g, 15.9 mmol) in ethanol (200 ml) and potassium hydroxide (5%, 200 ml) for 6 h. NaBH₄ (1.6 g) was added to a solution of 2-(2-oxo-3-phenylpropyl) benzoic acid (4.81 g, 17.8 mmol) in sodium hydroxide (1%, 180 ml) and the resulting solution was stirred overnight at room temperature. After being acidified with HCl, the whole mixture was extracted with dichloromethane (2 × 15 ml). Usual work-up gave crude racemic hydroxy-acid, 2-(2-hydroxy-3-phenylpropyl)benzoic acid, which was dissolved in acetic anhydride (5 ml) and heated under reflux for 2 h to get the title compound (yield; 73%, m.p. 605–606 K). The crude compound was purified by column chromatography on silica gel with petroleum ether and recrystallized in ethanol.

Refinement

H atoms were positioned geometrically, with C-H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

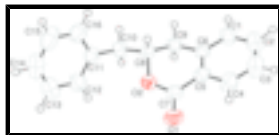


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.



Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

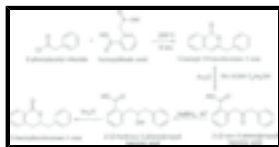


Fig. 3. The formation of the title compound.

3-Benzylisochroman-1-one

Crystal data

$C_{16}H_{14}O_2$

$M_r = 238.27$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 12.503\ (5)\ \text{\AA}$

$b = 8.0200\ (9)\ \text{\AA}$

$c = 12.892\ (5)\ \text{\AA}$

$\beta = 102.43\ (2)^\circ$

$V = 1262.4\ (7)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 504$

$D_x = 1.254\ \text{Mg m}^{-3}$

Melting point: $332(1)\ \text{K}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1316 reflections

$\theta = 5.3\text{--}25.2^\circ$

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

$T = 294\ \text{K}$

Block, colorless

$0.32 \times 0.26 \times 0.21\ \text{mm}$

Data collection

Bruker SMART CCD area-detector

3024 independent reflections

diffractometer	
Radiation source: fine-focus sealed tube	2546 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.021$
$T = 294$ K	$\theta_{\text{max}} = 28.5^\circ$
ω and φ scans	$\theta_{\text{min}} = 3.0^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -16 \rightarrow 16$
$T_{\text{min}} = 0.820$, $T_{\text{max}} = 0.983$	$k = -10 \rightarrow 10$
7148 measured reflections	$l = -10 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 0.1131P]$
$wR(F^2) = 0.136$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3024 reflections	$\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$
164 parameters	$\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xkFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.058 (7)

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.00370 (8)	0.34647 (13)	-0.09298 (6)	0.0694 (3)
O2	0.04862 (6)	0.20854 (10)	0.05531 (6)	0.0513 (2)
C1	-0.27750 (10)	0.22424 (17)	0.10333 (11)	0.0614 (3)
H1A	-0.2984	0.1604	0.1558	0.074*
C2	-0.35219 (11)	0.3310 (2)	0.04219 (14)	0.0770 (4)
H2A	-0.4230	0.3383	0.0536	0.092*
C3	-0.32221 (12)	0.4264 (2)	-0.03548 (14)	0.0793 (4)

supplementary materials

H3A	-0.3728	0.4978	-0.0766	0.095*
C4	-0.21631 (11)	0.41640 (17)	-0.05268 (11)	0.0653 (3)
H4A	-0.1961	0.4815	-0.1050	0.078*
C5	-0.14040 (9)	0.30857 (13)	0.00867 (8)	0.0481 (3)
C6	-0.17032 (8)	0.21122 (13)	0.08690 (8)	0.0461 (2)
C7	-0.02835 (9)	0.29314 (14)	-0.01389 (8)	0.0495 (3)
C8	0.02859 (8)	0.16880 (12)	0.15967 (7)	0.0439 (2)
H8A	0.0341	0.2713	0.2019	0.053*
C9	-0.08557 (9)	0.09595 (13)	0.14927 (8)	0.0484 (3)
H9A	-0.0896	-0.0113	0.1138	0.058*
H9B	-0.1002	0.0786	0.2194	0.058*
C10	0.11974 (9)	0.05047 (14)	0.21118 (9)	0.0528 (3)
H10A	0.1079	0.0185	0.2804	0.063*
H10B	0.1147	-0.0498	0.1683	0.063*
C11	0.23470 (9)	0.12051 (13)	0.22511 (9)	0.0506 (3)
C12	0.30019 (11)	0.08497 (17)	0.15378 (11)	0.0647 (3)
H12A	0.2725	0.0181	0.0952	0.078*
C13	0.40664 (12)	0.1470 (2)	0.16750 (15)	0.0809 (4)
H13A	0.4488	0.1209	0.1185	0.097*
C14	0.44906 (13)	0.2457 (2)	0.25261 (17)	0.0870 (5)
H14A	0.5200	0.2870	0.2622	0.104*
C15	0.38502 (15)	0.2832 (2)	0.32398 (15)	0.0894 (5)
H15A	0.4131	0.3509	0.3820	0.107*
C16	0.27839 (13)	0.22086 (18)	0.31051 (11)	0.0708 (4)
H16A	0.2365	0.2475	0.3597	0.085*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0738 (6)	0.0907 (7)	0.0453 (4)	-0.0159 (5)	0.0161 (4)	0.0086 (4)
O2	0.0496 (4)	0.0622 (5)	0.0443 (4)	-0.0036 (3)	0.0148 (3)	0.0004 (3)
C1	0.0497 (6)	0.0677 (7)	0.0686 (7)	-0.0063 (5)	0.0169 (5)	-0.0006 (6)
C2	0.0477 (6)	0.0826 (9)	0.0997 (11)	0.0023 (6)	0.0136 (7)	0.0030 (8)
C3	0.0588 (7)	0.0751 (9)	0.0955 (10)	0.0062 (6)	-0.0020 (7)	0.0134 (8)
C4	0.0631 (7)	0.0630 (7)	0.0640 (7)	-0.0052 (6)	0.0008 (5)	0.0107 (6)
C5	0.0504 (6)	0.0486 (5)	0.0430 (5)	-0.0087 (4)	0.0045 (4)	-0.0041 (4)
C6	0.0464 (5)	0.0478 (5)	0.0438 (5)	-0.0073 (4)	0.0086 (4)	-0.0074 (4)
C7	0.0566 (6)	0.0529 (6)	0.0387 (5)	-0.0123 (4)	0.0098 (4)	-0.0043 (4)
C8	0.0483 (5)	0.0457 (5)	0.0380 (5)	-0.0015 (4)	0.0100 (4)	-0.0036 (4)
C9	0.0506 (5)	0.0508 (5)	0.0453 (5)	-0.0044 (4)	0.0138 (4)	0.0014 (4)
C10	0.0533 (6)	0.0488 (6)	0.0564 (6)	0.0023 (4)	0.0121 (4)	0.0014 (5)
C11	0.0500 (5)	0.0460 (5)	0.0527 (6)	0.0073 (4)	0.0047 (4)	0.0031 (4)
C12	0.0598 (7)	0.0671 (7)	0.0680 (7)	0.0025 (6)	0.0152 (6)	-0.0048 (6)
C13	0.0600 (8)	0.0819 (9)	0.1042 (11)	0.0051 (7)	0.0252 (8)	0.0072 (9)
C14	0.0520 (7)	0.0795 (10)	0.1203 (14)	-0.0013 (7)	-0.0019 (8)	0.0093 (10)
C15	0.0777 (10)	0.0835 (10)	0.0908 (11)	-0.0065 (8)	-0.0181 (8)	-0.0153 (8)
C16	0.0699 (8)	0.0730 (8)	0.0633 (8)	0.0056 (6)	0.0009 (6)	-0.0116 (6)

Geometric parameters (Å, °)

C1—C2	1.382 (2)	C8—H8A	0.9800
C1—C6	1.405 (2)	C9—H9A	0.9700
C1—H1A	0.9300	C9—H9B	0.9700
C2—C3	1.375 (3)	C10—C11	1.517 (2)
C2—H2A	0.9300	C10—H10A	0.9700
C3—C4	1.391 (3)	C10—H10B	0.9700
C3—H3A	0.9300	C11—C16	1.378 (2)
C4—C5	1.3968 (19)	C11—C12	1.386 (2)
C4—H4A	0.9300	C12—C13	1.396 (2)
C5—C6	1.3889 (18)	C12—H12A	0.9300
C5—C7	1.496 (2)	C13—C14	1.365 (3)
C6—C9	1.5023 (19)	C13—H13A	0.9300
C7—O1	1.2054 (17)	C14—C15	1.377 (3)
C7—O2	1.3463 (17)	C14—H14A	0.9300
C8—O2	1.4559 (19)	C15—C16	1.399 (3)
C8—C9	1.521 (2)	C15—H15A	0.9300
C8—C10	1.5214 (19)	C16—H16A	0.9300
C2—C1—C6	120.63 (13)	C8—C9—H9A	109.5
C2—C1—H1A	119.7	C6—C9—H9B	109.5
C6—C1—H1A	119.7	C8—C9—H9B	109.5
C3—C2—C1	120.23 (14)	H9A—C9—H9B	108.1
C3—C2—H2A	119.9	C11—C10—C8	114.97 (12)
C1—C2—H2A	119.9	C11—C10—H10A	108.5
C2—C3—C4	120.14 (13)	C8—C10—H10A	108.5
C2—C3—H3A	119.9	C11—C10—H10B	108.5
C4—C3—H3A	119.9	C8—C10—H10B	108.5
C3—C4—C5	119.97 (13)	H10A—C10—H10B	107.5
C3—C4—H4A	120.0	C16—C11—C12	117.44 (14)
C5—C4—H4A	120.0	C16—C11—C10	120.88 (11)
C6—C5—C4	120.18 (13)	C12—C11—C10	121.68 (12)
C6—C5—C7	120.30 (10)	C11—C12—C13	121.74 (15)
C4—C5—C7	119.46 (12)	C11—C12—H12A	119.1
C5—C6—C1	118.84 (11)	C13—C12—H12A	119.1
C5—C6—C9	117.73 (11)	C14—C13—C12	120.23 (15)
C1—C6—C9	123.42 (11)	C14—C13—H13A	119.9
O1—C7—O2	117.57 (12)	C12—C13—H13A	119.9
O1—C7—C5	123.76 (11)	C13—C14—C15	118.86 (16)
O2—C7—C5	118.59 (11)	C13—C14—H14A	120.6
O2—C8—C9	110.33 (8)	C15—C14—H14A	120.6
O2—C8—C10	106.16 (9)	C14—C15—C16	120.99 (16)
C9—C8—C10	113.54 (11)	C14—C15—H15A	119.5
O2—C8—H8A	108.9	C16—C15—H15A	119.5
C9—C8—H8A	108.9	C11—C16—C15	120.73 (15)
C10—C8—H8A	108.9	C11—C16—H16A	119.6
C6—C9—C8	110.56 (11)	C15—C16—H16A	119.6
C6—C9—H9A	109.5	C7—O2—C8	118.82 (10)

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C9-H9B\cdots O1^i$	0.97	2.53	3.2922 (18)	135

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

Fig. 1

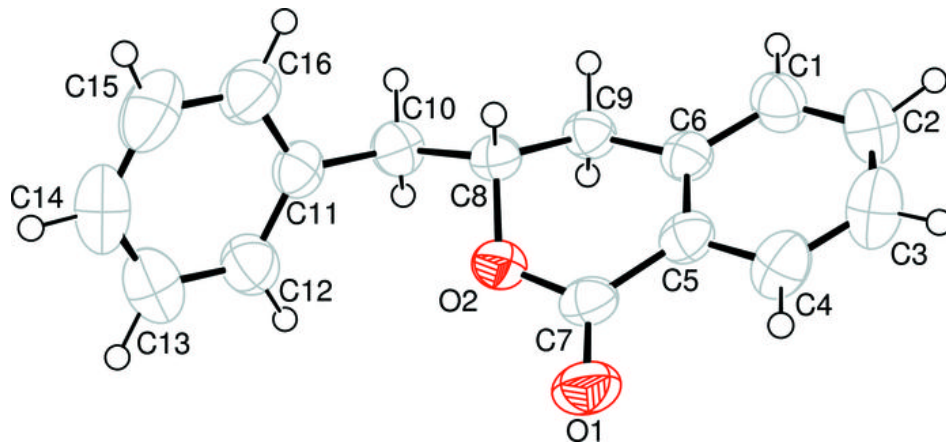


Fig. 2

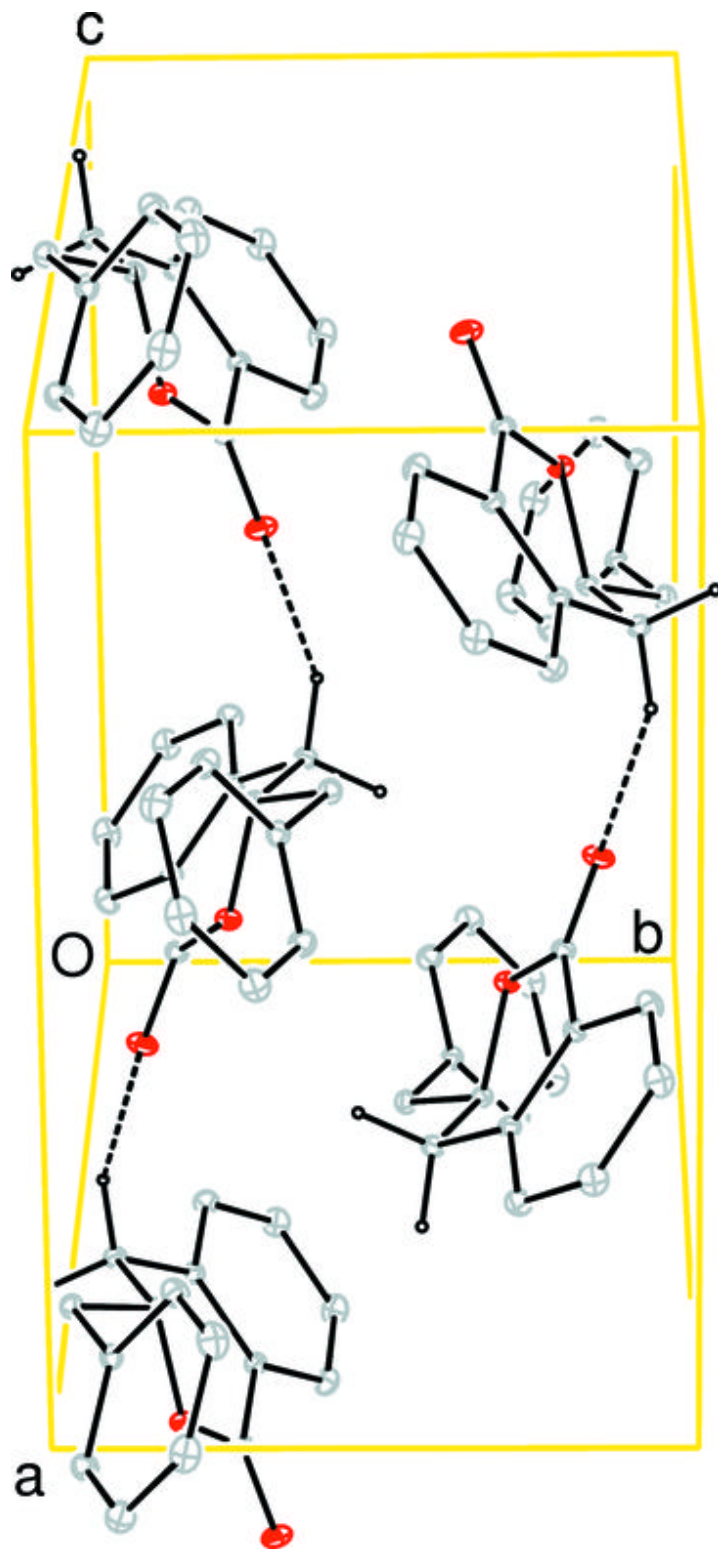


Fig. 3

