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5,7-Bis(benzyloxy)-4*H*-benzopyran-4-one

Zhi-Fang Hao,[a] Qiang Xu,[b] Zhi-Feng Lu[c] and Jian-Xin Li[a]*

[a]Institute of Medicinal Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, [b]School of Life Science, Nanjing University, Nanjing 210093, People's Republic of China, and [c]School of Chemistry & Chemical Engineering, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail:
luzf_nju@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.141
Data-to-parameter ratio = 13.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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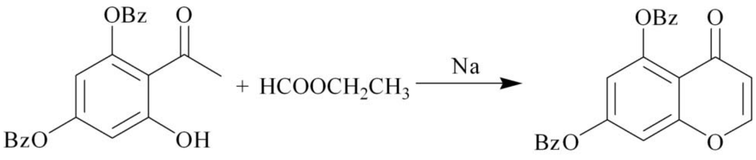
The title compound, $C_{23}H_{18}O_4$, contains two molecules in the asymmetric unit which differ from each other in the dihedral angles between the benzyl and chromone groups. There are intramolecular and intermolecular C—H···O interactions as well as π–π stacking interactions in the crystal structure.

Comment

5,7-Dihydroxychromone is a flavanoid decomposition product that has been found as a constituent in certain plant extracts (Pendse *et al.*, 1973) and is a germination and growth inhibitor (Spencer & Tjarks, 1985). In the process of preparing it, we obtained the title compound, (I), as one of the intermediates. As part of this study, we have undertaken the X-ray crystallographic analysis of (I).



(I)

The title structure (Fig. 1) contains two independent molecules, *A* and *B*, in the asymmetric unit. The geometrical parameters of the chromone group in (I) are comparable to those of related structures reported earlier (Wallet & Cody, 1995). The chromone group is essentially planar in both molecules, with maximum deviations of 0.112 (3) Å for atom O2 and 0.108 (3) Å for O2′. The benzyl group attached at atom O3 is almost coplanar with the chromone group, with a dihedral angle of 6.3 (2)° in *A* and 9.2 (2)° in *B*, while the other benzyl group at O4 is twisted away from it, with a dihedral angle of 75.1 (2)° in *A* and 63.2 (2)° in *B*.

In the crystal structure, the molecular packing is stabilized by intramolecular and intermolecular C—H···O interactions (Table 2) as well as π–π stacking interactions (Table 3).

Experimental

A mixture of 2,4-bis(benzyloxy)-6-hydroxyacetophenone (3.2 g) and sodium (0.9 g) in ethyl formate (15 ml) was stirred at 263 K for 6 h. After addition of methanol (5 ml) and ice-water (30 ml), the mixture was acidified with acetic acid. The excess of ethyl formate was removed by a current of air. The mixture was filtered and the solid was dissolved in an ethanol–acetone (1:3 *v*/*v*) mixed solvent. When the solution was acidified with concentrated hydrochloric acid (10 drops), a white solid was isolated in 70% yield (2.3 g). Single crystals of (I) were obtained by slow evaporation of a petroleum ether–$CHCl_3$ (1:1 *v*/*v*) solution of (I).

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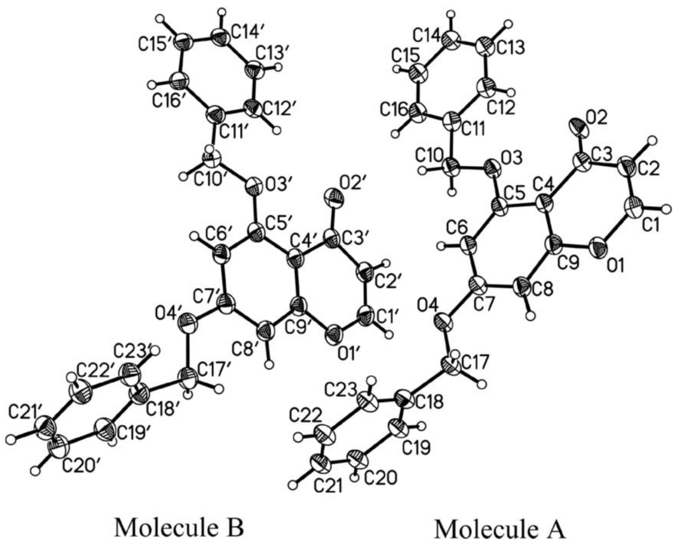

Figure 1
View of the asymmetric unit of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

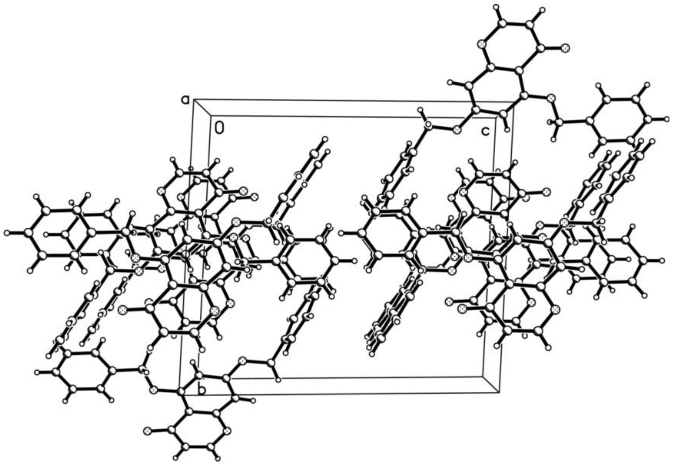

Figure 2
The crystal packing of (I), viewed down the *a* axis.

Crystal data

$C_{23}H_{18}O_4$
$M_r = 358.37$
Triclinic, $P\overline{1}$
$a = 9.251$ (1) Å
$b = 13.493$ (2) Å
$c = 14.785$ (2) Å
$\alpha = 92.881$ (2)°
$\beta = 95.480$ (2)°
$\gamma = 90.096$ (2)°
$V = 1834.8$ (4) Å^3
$Z = 4$
$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2940 reflections
$\theta = 2.6$–25.8°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART Apex CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.97$, $T_{\max} = 0.98$
9179 measured reflections
6337 independent reflections
4578 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 10$
$k = -16 \rightarrow 13$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.141$
$S = 1.00$
6337 reflections
487 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0748P)^2 + 0.255P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—O2	1.220 (3)	C3′—O2′	1.211 (3)
C5—O3	1.352 (3)	C5′—O3′	1.330 (3)
C7—O4	1.352 (3)	C7′—O4′	1.347 (3)
C5—O3—C10	119.9 (2)	C5′—O3′—C10′	119.9 (2)
C7—O4—C17	115.7 (2)	C7′—O4′—C17′	117.4 (2)
C1′—O1′—C9′	118.4 (2)		
O3—C10—C11—C12	−2.5 (3)	O3′—C10′—C11′—C12′	0.9 (3)
O4—C17—C18—C19	76.2 (3)	O4′—C17′—C18′—C19′	123.7 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2	0.93	2.55	3.462 (3)	167
C12—H12···O3	0.93	2.30	2.648 (3)	102
C12′—H12′···O2′	0.93	2.58	3.494 (3)	169
C12′—H12′···O3′	0.93	2.30	2.665 (3)	103
C1—H1···O3′$^{\rm i}$	0.93	2.58	3.508 (3)	173
C1′—H1′···O3$^{\rm ii}$	0.93	2.53	3.439 (3)	165

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

Table 3
π–π interactions (Å, °)..

CgI	CgJ	Symmetry code	$CgI\cdots CgJ$	Dihedral angle	Interplanar distance	Offset
A	B	$-x, 2-y, -z$	3.784 (2)	1.2 (2)	3.422 (3)	1.62
B	A	$-x, 2-y, -z$	3.784 (2)	1.2 (2)	3.422 (3)	1.62
B	B	$-x, 2-y, -z$	3.623 (2)	0.0 (2)	3.425 (3)	1.18
C	D	$1-x, 1-y, -z$	3.927 (2)	0.9 (2)	3.480 (3)	1.82
D	C	$1-x, 1-y, -z$	3.927 (2)	0.9 (2)	3.480 (3)	1.82
D	D	$1-x, 1-y, -z$	3.699 (2)	0.0 (2)	3.479 (3)	1.26

$Cg(A)$, $Cg(B)$, $Cg(C)$ and $Cg(D)$ denote the centroids of the aromatic rings O1/C1–C4/C9, C4–C9, O1′/C1′–C4′/C9′ and C4′–C9′, respectively. The offset is defined as the distance between $Cg(I)$ and the perpendicular projection of $Cg(J)$ on ring I.

H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances in the range 0.93–0.97 Å and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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References

Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Pendse, R., Rama, A. V. R. & Venkataraman, K. (1973). *Phytochemistry*, **12**, 2033–2034.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Spencer, G. F. & Tjarks, L. W. (1985). *J. Plant Growth Regul.* **4**, 177–180.

Wallet, J.-C. & Cody, V. (1995). *Acta Cryst.* C**51**, 1193–1195.