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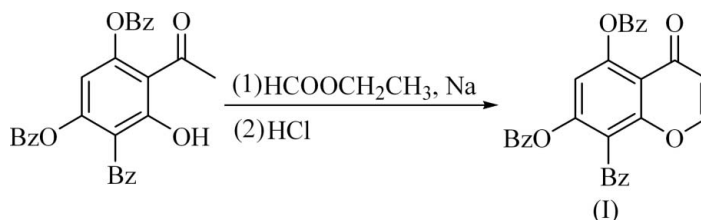
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.062
 wR factor = 0.153
Data-to-parameter ratio = 14.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

8-Benzyl-5,7-bis(benzyloxy)-4H-benzopyran-4-one

In the title compound, $\text{C}_{30}\text{H}_{24}\text{O}_4$, the chromone system is essentially planar. There are intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions in the crystal structure.Received 16 May 2006
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Comment

5,7-Dihydroxychromone, which is a germination and growth inhibitor (Spencer & Tjarks, 1985), is a flavanoid decomposition product that has been found as a constituent in certain plant extracts (Pendse *et al.*, 1973). In the process of its preparation, we obtained the intermediate (I). As part of this study, we have undertaken the X-ray crystallographic analysis of (I) in order to elucidate the conformation and configuration of this intermediate (Fig. 1 and Table 1).The geometrical parameters of the chromone system in (I) are comparable to those of the related structures reported earlier (*e.g.* Hao *et al.*, 2006). The chromone system is essentially planar. The benzyl group attached at atom O3 is almost coplanar with the chromone system, with a dihedral angle of $7.4(3)^\circ$, while the other benzyl groups, at atoms O4 and C8, are twisted away from it, with dihedral angles of $79.7(3)$ and $71.8(3)^\circ$, respectively.In the crystal structure, the molecular packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions (Table 2) involving the aromatic rings.

Experimental

A mixture of 5-benzyl-2,4-bis(benzyloxy)-6-hydroxyacetophenone (3.9 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 ethanol. After the solution was acidified with concentrated hydrochloric acid, (I) was isolated by column chromatography of the residue after evaporation of the solvent on silica gel, using CH_2Cl_2 as eluent. Single crystals of (I) were obtained by slow evaporation of a petroleum ether- CHCl_3 (1:1 v/v) solution (yield 65%).

Crystal data

$C_{30}H_{24}O_4$
 $M_r = 448.49$
 Monoclinic, $P2_1/c$
 $a = 9.723$ (2) Å
 $b = 9.746$ (2) Å
 $c = 24.698$ (5) Å
 $\beta = 95.15$ (3)°
 $V = 2330.9$ (8) Å³

$Z = 4$
 $D_x = 1.278$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.35 \times 0.32 \times 0.21$ mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (XCAD4; Harms & Wocadlo,
 1995)
 $T_{\min} = 0.959$, $T_{\max} = 0.983$
 4828 measured reflections

4549 independent reflections
 2228 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.078$
 $\theta_{\text{max}} = 26.0^\circ$
 3 standard reflections
 every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.153$
 $S = 1.00$
 4549 reflections
 308 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.32P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³
 Extinction correction: SHELXTL
 Extinction coefficient: 0.0250 (15)

Table 1

Selected geometric parameters (Å, °).

O2–C3	1.229 (4)	O4–C7	1.358 (3)
O3–C5	1.355 (3)		
C1–O1–C9	118.3 (3)	C7–O4–C17	118.7 (2)
C5–O3–C10	119.8 (2)		
O3–C10–C11–C12	−0.4 (4)	C8–C24–C25–C26	−104.8 (3)
O4–C17–C18–C19	90.5 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C19–H19 ⁱ ⋯O2 ⁱ	0.93	2.63	3.453 (3)	148
C20–H20 ⁱⁱ ⋯O2 ⁱⁱ	0.93	2.61	3.250 (3)	127
C2–H2 ⁱⁱⁱ ⋯CgA ⁱⁱⁱ	0.93	2.99	3.545 (4)	120
C17–H17A ^{iv} ⋯CgB ^{iv}	0.97	2.95	3.430 (3)	112

Symmetry codes: (i) $-x + 1, -y, -z$; (ii) $x + 1, y - 1, z$; (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x + 2, -y, -z$. CgA and CgB denote the centroids of aromatic rings C25–C30 and C18–C23, respectively.

H atoms were positioned geometrically and refined as riding on their parent C atoms, with C–H distances constrained to 0.93

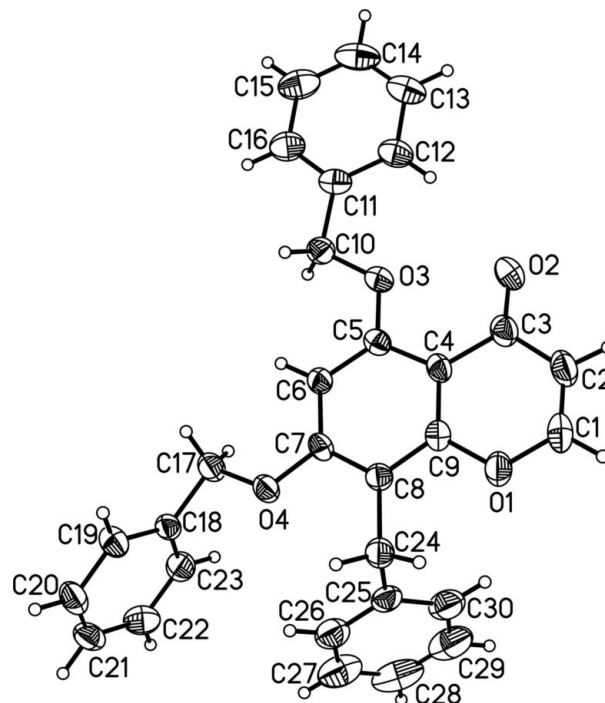


Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

(aromatic CH) or 0.97 Å (methylene CH₂), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier C})$.

Data collection: CAD-4 Software (Enraf–Nonius, 1989); cell refinement: CAD-4 Software; data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXTL (Sheldrick, 1997); 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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