

5-Benzyl-8-(*E*)-benzylidene-6,7,7a,10a-tetrahydro-5*H*-cis-cyclopenta[5,6]-pyrano[3,3-*c*]quinolin-6-one

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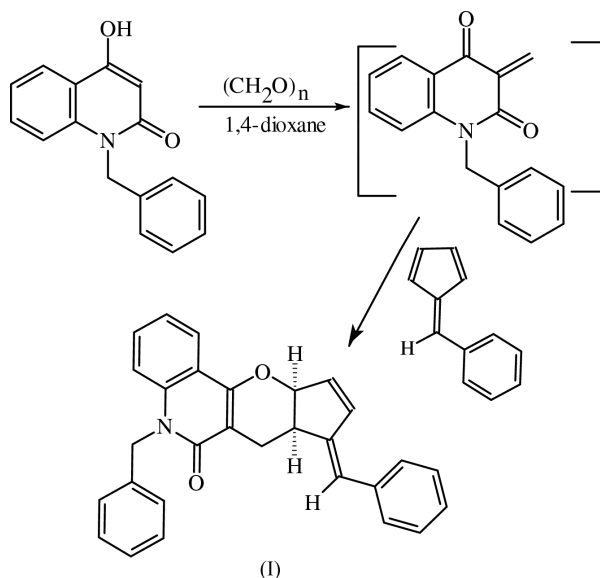
Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.059
 wR factor = 0.132
Data-to-parameter ratio = 14.7

In the title compound, $\text{C}_{29}\text{H}_{23}\text{NO}_2$, the quinolinone moiety is planar and the pyran ring is in a half-chair form. The cyclopentene fused with the pyran ring adopts an envelope conformation. There are $\text{C}-\text{H}\cdots\text{O}$, $\text{C}-\text{H}\cdots\pi$ and $\pi\cdots\pi$ intermolecular interactions.

Comment

Pyranoquinolinones constitute a large group of naturally occurring biologically active compounds, such as flindersine, veprisine, paraensidimerin and vepridimerine (Grundon, 1988; Chen *et al.*, 1994). The quinolinones are a new class of antibiotics with therapeutic properties that are potentially attributable to their wide spectrum of antimicrobial activity (Fitten, 1992; McCarter *et al.*, 1992). Substituent effects play a vital role in the lasing action of quinolinones and they can be exploited to control laser efficiency, tunability and photostability (Hammond *et al.*, 1975). In view of the above facts, the title compound, (I), was synthesized and the crystal structure determination was carried out.



The molecular structure of (I) is shown in Fig. 1. The quinolinone moiety (N1/O2/C2–C10) is planar and this planarity is associated with partial double-bond character of the $\text{O1}-\text{C6}=\text{C7}-\text{C8}(-\text{N1})=\text{O2}$ moiety. The bond lengths and bond angles (Table 1) confirm this resonance form (Chinnakali *et al.*, 1991; Kido & Nakagawa, 1982).

A study of the torsion angles (Table 1), the asymmetry parameters and a least-squares plane calculation shows that the pyrane ring adopts a half-chair conformation ($Q_T = 0.388$; Nardelli, 1995). Atoms C13, O1, C6 and C7 constitute the

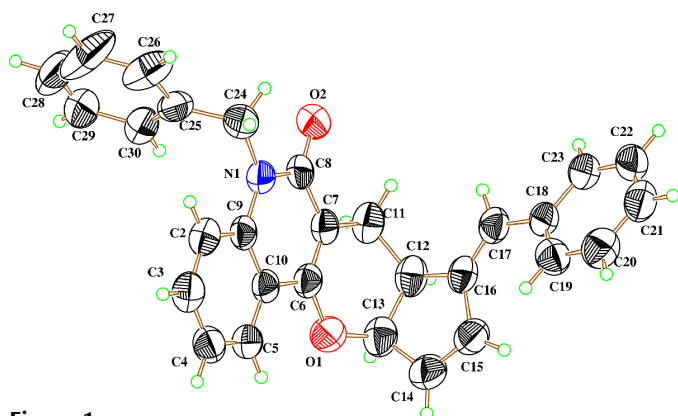


Figure 1
Molecular structure of (I), showing 50% probability displacement ellipsoids.

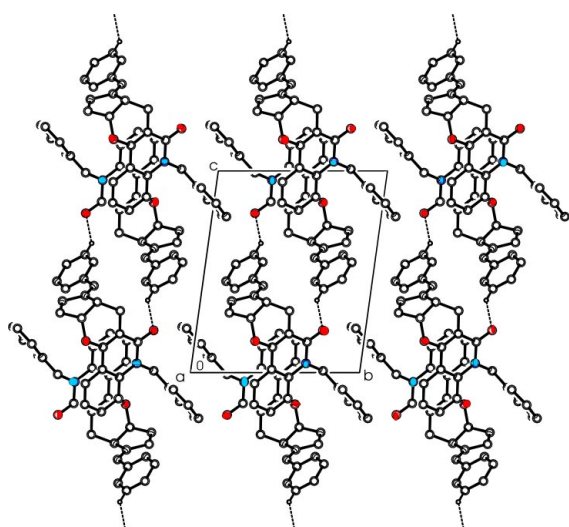


Figure 2
Crystal structure of (I), viewed down the *a* axis. The dashed lines represent the C—H...O interactions.

basal plane of the pyridone ring, and atoms C12 and C13 deviate by 0.0275 (2) and 0.315 (2) Å, respectively. The basal plane of the pyran ring makes an angle of 9.67 (9)° with the plane of the pyridone ring. The cyclopentene ring adopts an envelope conformation, with atom C12 deviating from the C13–C16 plane. The benzylidene phenyl ring is *cis* to the cyclopentene ring. The *N*-benzyl group is approximately perpendicular to the pyridone ring [84.8 (1)°].

The packing of molecules, viewed down the *a* axis, is shown in Fig. 2. The molecules form dimers *via* C—H...O interactions (Table 2), and extend in the *c* direction. The C—H... π and π ... π interactions (Desiraju, 1989) also contribute to the crystal packing, in addition to van der Waals forces. In Table 2, *Cg*1, *Cg*2 and *Cg*3 are the centroids of the benzene rings C2–C5/C10/C9, C18–C23 and C25–C30, respectively. The centroid-to-centroid distances of the π ... π interactions are 4.869 (2) Å for *Cg*4...*Cg*4ⁱⁱⁱ [symmetry code (iii) $-x, 1-y, 2-z$], 3.949 (2) Å for *Cg*4...*Cg*1ⁱⁱⁱ, 4.401 (2) Å for *Cg*1...*Cg*1ⁱⁱⁱ and 4.570 (2) Å for *Cg*4...*Cg*1^v [symmetry code: (v) $1-x, 1-y, 2-z$], where *Cg*4 is the centroid of the N1/C8/C7/C6/C10/C9 ring.

Experimental

A solution of 4-hydroxy-1-benzylquinolin-2-one (0.251 g, 1 mmol), paraformaldehyde (2.384 g, 8 mmol) and 6-phenylfulvene (0.308 g, 2 mmol) in dry 1,4-dioxane (4 ml) were refluxed for 4 h followed by column chromatography to afford a separable mixture as a pale yellow solid of the pyranoquinolone derivative (Nair *et al.*, 2001), which was then recrystallized from ethanol by slow evaporation.

Crystal data

$C_{29}H_{23}NO_2$	$Z = 2$
$M_r = 417.48$	$D_x = 1.280 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.422$ (2) Å	Cell parameters from 4237 reflections
$b = 10.585$ (3) Å	$\theta = 2.0$ – 26.9°
$c = 12.866$ (3) Å	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 79.879$ (4)°	$T = 273$ (2) K
$\beta = 76.281$ (4)°	Block, yellow
$\gamma = 79.042$ (4)°	$0.32 \times 0.20 \times 0.12 \text{ mm}$
$V = 1083.6$ (5) Å ³	

Data collection

Siemens SMART CCD area detector diffractometer	3097 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.019$
Absorption correction: none	$\theta_{\text{max}} = 26.9^\circ$
10 877 measured reflections	$h = -10 \rightarrow 10$
4237 independent reflections	$k = -12 \rightarrow 12$
	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.1865P]$
$R[F^2 > 2\sigma(F^2)] = 0.059$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.12$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
4237 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
289 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

N1—C9	1.393 (2)	C6—C7	1.348 (3)
N1—C8	1.396 (2)	O2—C8	1.235 (2)
O1—C6	1.350 (2)	C7—C8	1.445 (3)
O1—C13	1.460 (2)		
C13—O1—C6—C7	14.2 (3)	C15—C16—C12—C13	18.92 (19)
O1—C6—C7—C8	178.99 (16)	O1—C13—C12—C11	−32.5 (3)
O1—C6—C7—C11	0.4 (3)	C14—C13—C12—C16	−20.3 (2)
C6—C7—C11—C12	−29.8 (2)	C12—C16—C15—C14	−10.3 (2)
C6—O1—C13—C12	3.1 (3)	C16—C15—C14—C13	−3.5 (3)
C7—C11—C12—C13	44.0 (2)	C12—C13—C14—C15	15.4 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C24—H24A...O2	0.97	2.25	2.706 (2)	108
C22—H22...O2 ⁱ	0.93	2.47	3.275 (3)	145
C29—H29...O2 ⁱⁱ	0.93	2.68	3.388 (3)	134
C24—H24B... <i>Cg</i> 1 ⁱⁱⁱ	0.97	3.03	3.642	123
C27—H27... <i>Cg</i> 2 ^{iv}	0.93	3.09	3.906	147
C19—H19... <i>Cg</i> 3 ⁱⁱⁱ	0.93	3.29	4.033	138

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

All H atoms were included at calculated positions and refined using a riding model, with C—H bond lengths of 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The U_{eq} values of C27 and

C28 are particularly high, suggesting rotational motion of the phenyl group around the C24–C25 bond. Attempts to find alternative positions or possible disorder for these atoms were unsuccessful.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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