2-Benzyl-1-hydroxy-5,6-dihydro-3H-naphtho[2,1-b]pyran-3-one

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The title compound, $C_{20}H_{16}O_3$, is a representative of a class of substituted α-pyrones with a distorted pyran ring.

Comment

There are several examples of the 'structure-correlation principle' whereby the structure of a compound may presage the transition state for its formation or destruction, especially when the activation barrier is low (Bürgi & Dunitz, 1983). α-Pyrones (2-pyrones) can undergo highly endothermic ring-opening reactions to the valence isomeric oxovinylketenes on flash vacuum thermolysis (Wentrup et al., 1994). Acute O—C=O angles of ca 115°, and slightly elongated O—C(=O) single bonds of 1.39 Å were found for all 4-hydroxy-2-pyrones in the Cambridge Structural Database (2003 update; Allen, 2002). This could be interpreted as reminiscent of the transition states for the ring opening to ketenes, analogous to similar observations on other cyclic carbonyl compounds (Pool & White, 2000; Plüg et al., 2000; Shumway et al., 2001; Birney et al., 2002). However, a large effect is not expected for reactions with such high activation barriers. The data indicate the effect is indeed small or negligible. Although the small O—C=O angles and long O1—C2 bonds are found in the fully unsaturated 2-pyrones, (II), they also exist in the partially and fully saturated 2-pyrones (III)–(V). Indeed, the smallest O—C=O angle [113.9 (5)°] is also found in the 3,4-dihydro-2-pyrones, (IV), thereby indicating an intrinsic structural effect in lactone-type compounds. The corresponding O—C=O angle in esters averages 122.4 (2)° (690 examples).
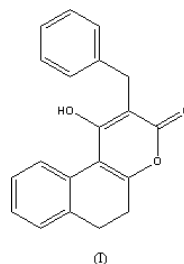
In the following analysis, the structure of the title compound, (I) (Fig. 1), is compared to a survey using well determined structures of diunsaturated, (II) [70], 5,6-dihydro-, (III) [62], 3,4-dihydro-, (IV) [12], and tetrahydro-2-pyrones, (V) [89], where [n] is the number of observations in each set.
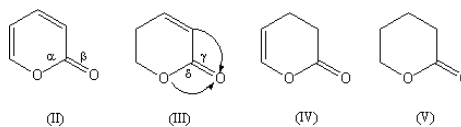
The average O—C and C=O bond distances α and β, and the angles γ and δ are for compound (I) and (II)–(IV): 1.383 (3), 1.389 (2), 1.351 (2), 1.394 (3) and 1.347 (2), and
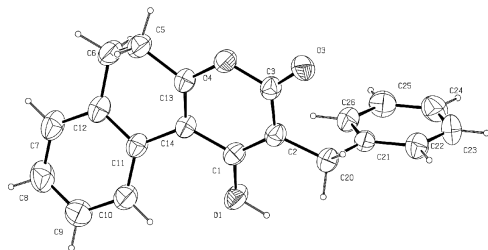
Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level.

1.222 (3), 1.213 (2), 1.208 (1), 1.208 (1) and 1.202 (1) Å, and 127.3 (2), 127.2 (2), 124.1 (2), 128.5 (4) and 124.3 (2), and 115.2 (2), 115.8 (2), 117.6 (2), 113.1 (6) and 117.6 (2)°, respectively.

Compound (I) forms a hydrogen-bonded polymer down the *a* axis, with an intermolecular hydrogen bond between atoms O1 and O3 (Table 2). There is a close intramolecular distance between atoms H1 and H20A of 1.95 Å.

Experimental

Details of the synthesis of (I) is given in Kappe & Wildpanner (1988).

Crystal data

$C_{20}H_{16}O_3$	Mo $K\alpha$ radiation
$M_r = 304.33$	Cell parameters from 25
Orthorhombic, $P2_12_12_1$	reflections
$a = 9.039$ (2) Å	$\theta = 10–12°$
$b = 8.175$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 20.304$ (2) Å	$T = 293$ (2) K
$V = 1500.3$ (5) Å^3	Hexagonal plate, colourless
$Z = 4$	$0.34 \times 0.28 \times 0.13$ mm
$D_x = 1.347$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	1162 reflections with $I > 2\sigma(I)$
ω-2θ scans	$\theta_{max} = 25.0°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 10$
	$k = 0 \rightarrow 9$
$T_{min} = 0.965$, $T_{max} = 0.993$	$l = 0 \rightarrow 24$
1528 measured reflections	3 standard reflections
1528 independent reflections	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$+ 0.2016P]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.93$	$(\Delta/\sigma)_{max} < 0.001$
1528 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.370 (4)	C3—O3	1.222 (3)
C2—C3	1.419 (4)	C3—O4	1.383 (3)
C1—C2—C3	120.0 (2)	O3—C3—C2	127.3 (2)
O3—C3—O4	115.2 (2)	O4—C3—C2	117.5 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O3^i	0.94 (4)	1.93 (4)	2.819 (3)	155 (3)

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

All H atoms were calculated except for H1 which was obtained from a difference electron-density map. The molecule itself is achiral, but the space group is non-centrosymmetric. In the absence of significant anomalous scattering, the absolute configuration is indeterminate.

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *PLATON*98 (Spek, 1988); software used to prepare material for publication: *SHELXL*97.

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