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7a,8,9,10-Tetrahydrobenzo[*f*]cyclopenta[*b*]-chromen-10-one

Fangjun Huo, Caixia Yin and Pin Yang*

Institute of Molecular Science, Chemical Biology and Molecular Engineering, Laboratory of Education Ministry, Shanxi University, Taiyuan, Shanxi 030006, People's Republic of China

Correspondence e-mail: yangpin@sxu.edu.cn

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Key indicators

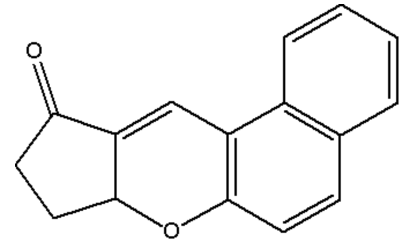
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.098
Data-to-parameter ratio = 10.3

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

In the title compound, $C_{16}H_{12}O_2$, the pyran ring adopts a half-chair conformation. Intermolecular C—H$\cdots$O hydrogen bonds, in the form of $R_2^2(8)$ rings, link the molecules into dimers. In addition, C—H$\cdots\pi$ interactions are found.

Comment

Chromenes (2*H*-1-benzopyran derivatives) are frequently found in naturally occurring heterocycles, many of which exhibit biological activity (Bowers *et al.*, 1976) and have been widely employed as important intermediates in the synthesis of many natural products and medicinal agents (Cassidy *et al.*, 1992; Wang & Finn, 2000). Thus, various synthetic methods for the formation of these compounds have been reported (Chang & Grubbs, 1998; Kaye & Nocanda,2000; Lee *et al.*, 2003; Parker & Mindt, 2001). In this paper, a new chromene compound, *viz.* 7a,8,9,10-tetrahydrobenzo[*f*]cyclopenta[*b*]-chromen-10-one, (I), is reported.



(I)

There are four fused rings in (I). The pyran ring adopts a half-chair conformation. The O2/C3/C2 and O2/C16/C7/C6 planes are inclined at an angle of 43.35 (1)°. The main geometric discrepancy is in the angle C1—C2—C6 of

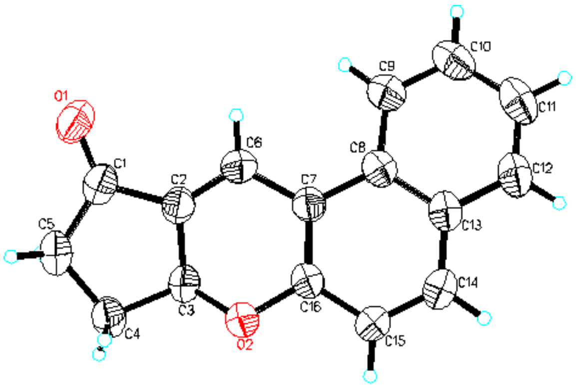


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme.

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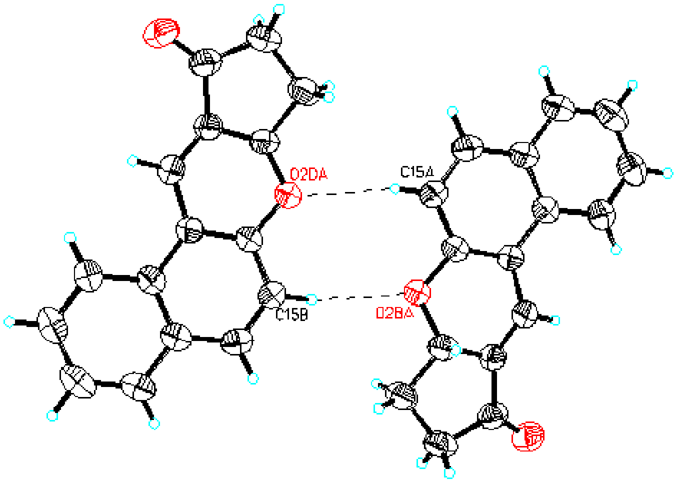

Figure 2
The dimer formed by C—H···O hydrogen bonds, which are indicated by dashed lines.

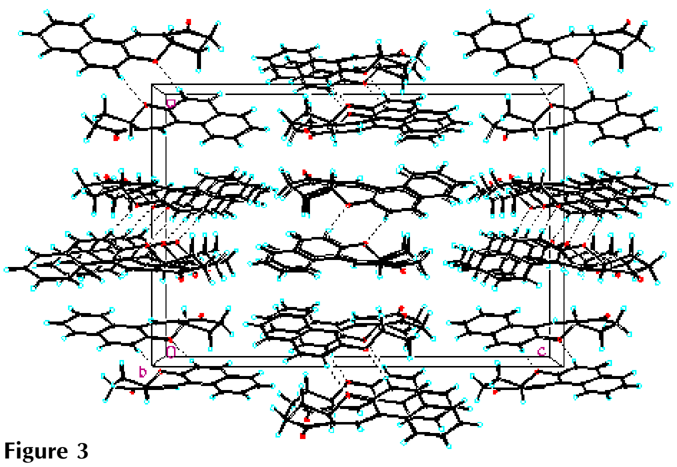

Figure 3
The crystal packing in (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

130.48 (17)°, probably a consequence of the steric restraints imposed by the four fused rings. The molecule contains a chiral atom, C3, but the crystal structure belongs to a centrosymmetric space group and is, therefore, a racemic mixture. The pairs of enantiomers related by the inversion centre are linked *via* C—H···O hydrogen bonds (Fig. 2). Specifically, the C15—H15···O2^{ii} [symmetry code: (ii) $1 - x$, $-y$, $1 - z$] hydrogen bond has the pyran O atoms as acceptors and aromatic CH atoms acting as donors. The weak interactions generate rings with graph-set motif $R_2^2(8)$ (Bernstein *et al.*, 1995). In addition, there are two intermolecular C—H···π contacts (Table 2), identified by *PLATON* (Spek, 2003), which involve the π system of the C8–C13 ring (with centroid *Cg*). Hydrogen-bonding information is given in Table 2 and a crystal packing diagram is shown in Fig. 3.

Experimental

Compound (I) was synthesized *via* a Baylis–Hillman reaction. At room temperature, a clear solution of 2-hydroxy-1-naphthaldehyde (1 mmol), cyclopent-2-enone (2 mmol) and imidazole (1 mmol) in tetrahydrofuran (1.5 ml) was mixed with deionized water (1.5 ml). The mixture was stirred at ambient temperature for 48 h to bring the reaction to completion. The mixture was diluted with water (10 ml) and extracted with ethyl acetate. The organic layer was concentrated under reduced pressure. After the usual work-up, chromatography of the crude product on silica gel, using ethyl acetate and petroleum (1:4) as eluant, gave the pure product (I) in 61% yield. The compound (60 mg) was dissolved in $CHCl_3$ (2 ml). The solution was allowed to evaporate slowly at room temperature over several days to yield yellow crystals suitable for X-ray crystallography.

Crystal data

$C_{16}H_{12}O_2$
$M_r = 236.26$
Orthorhombic, *Pbca*
$a = 15.5021$ (19) Å
$b = 6.6902$ (8) Å
$c = 22.676$ (3) Å
$V = 2351.7$ (5) Å^3
$Z = 8$
$D_x = 1.335$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 2391 reflections
$\theta = 2.2$–22.2°
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.30 × 0.30 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.974$, $T_{max} = 0.991$
9238 measured reflections

1684 independent reflections
1377 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 23.3$°
$h = -17 \rightarrow 15$
$k = -6 \rightarrow 7$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 1.04$
1684 reflections
163 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.3759P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.220 (2)	C2—C6	1.336 (2)
O2—C16	1.368 (2)	C2—C3	1.487 (2)
O2—C3	1.4388 (19)	C3—C4	1.521 (2)
C1—C2	1.466 (2)	C4—C5	1.528 (3)
C1—C5	1.507 (3)	C6—C7	1.449 (2)
C16—O2—C3	114.26 (13)	O2—C3—C4	110.99 (14)
O1—C1—C2	126.30 (17)	C2—C3—C4	105.04 (14)
O1—C1—C5	126.32 (16)	C3—C4—C5	104.57 (14)
C2—C1—C5	107.37 (16)	C1—C5—C4	105.93 (14)
C6—C2—C1	130.48 (17)	C2—C6—C7	118.57 (16)
C6—C2—C3	119.94 (15)	O2—C16—C7	122.18 (15)
C1—C2—C3	109.38 (15)	O2—C16—C15	115.66 (15)
O2—C3—C2	110.84 (14)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1^{i}	0.93	2.59	3.508 (2)	172
C15—H15···O2^{ii}	0.93	2.48	3.388 (2)	165
C4—H4*A*···*Cg*^{iii}	0.97	3.03	3.923 (2)	154
C10—H10···*Cg*^{iv}	0.93	2.95	3.676 (2)	136

Symmetry codes: (i) $x, \frac{3}{2} - y, \frac{1}{2} + z$; (ii) $1 - x, -y, 1 - z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $1 + x, -\frac{1}{2} - y, z - \frac{1}{2}$. *Cg* is the centroid of the C8–C13 ring.

H atoms were placed in calculated positions and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) values set to $1.5U_{\rm eq}$(parent atom) for the Csp^3-bound H atoms and $1.2U_{\rm eq}$(parent atom) for Csp^2-bound H atoms. The C—H distances were fixed in the range 0.93–0.97 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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