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7-Methoxy-2,3-dihydro-1*H*-cyclopenta[*b*]-chromen-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.143
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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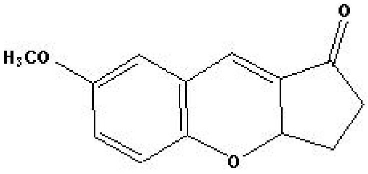
In the crystal structure of the title compound, $C_{13}H_{12}O_3$, the pyran ring adopts a half-chair conformation. Weak intermolecular C—H⋯O hydrogen bonds link the molecules into a three-dimensional network. In addition, C—H⋯π interactions are found.

Comment

Chromenes (2*H*-1-benzopyrane 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). Recently, we have reported two crystal structures of chromene derivatives (Huo *et al.*, 2004*a*,*b*). In this paper, we report the crystal structure of a new chromene compound, *viz*. the title compound, (I).



(I)

Selected geometric parameters are listed in Table 1. An ellipsoid plot of the molecule is shown in Fig. 1. The molecule contains three fused rings. The pyran ring adopts a half-chair conformation. The dihedral angle between the O2/C3/C2 and O2/C12/C7/C6 planes is 35.6 (1)°. The main geometric feature of note is the angle C6—C2—C1 of 130.22 (19)°, probably caused by the ring fusion. The molecule contains a chiral atom,

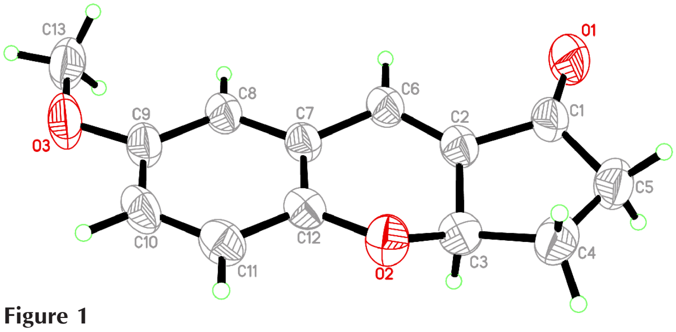


Figure 1
A view of the molecular structure of (I), showing the atom-numbering scheme, with displacement ellipsoids at the 50% probability level.

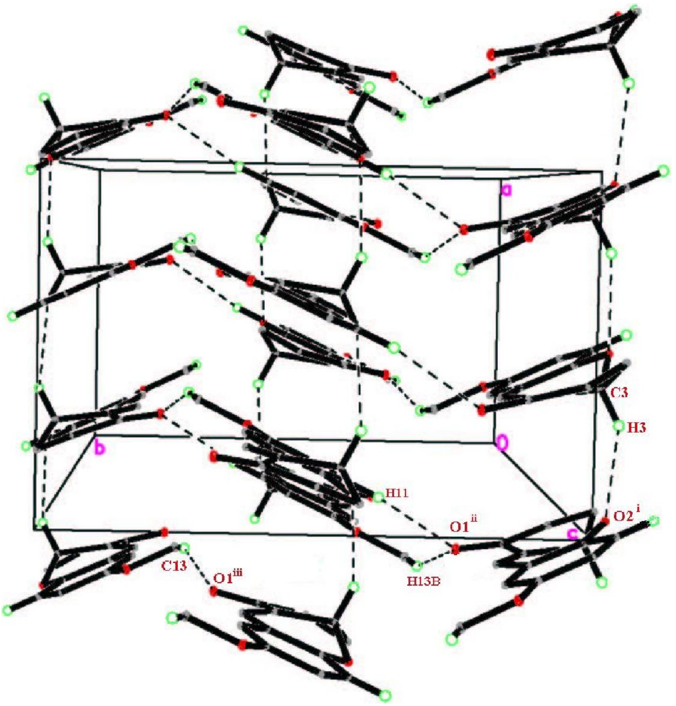


Figure 2
The crystal packing in (I), viewed approximately down the c axis. Hydrogen bonds are shown as dashed lines [symmetry code: (i) $x - \frac{1}{2}, y, \frac{3}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $x, \frac{1}{2} - y, \frac{1}{2} + z$]. Only selected atoms are labelled. Some of the H atoms have been omitted for clarity.

C3, but the crystal structure belongs to a centrosymmetric space group and is, therefore, a racemic mixture.

Glide-related molecules are linked *via* C3—H3···O2^{i} hydrogen bonds, forming one-dimensional chains along the a axis [symmetry code: (i) $x - \frac{1}{2}, y, \frac{3}{2} - z$]. This hydrogen bond has the pyran O atom as acceptor and the chiral C atom acting as donor, and generates chains with graph-set motif $C(3)$ (Bernstein *et al.*, 1995). The other two C—H···O interactions (Table 2) play an important role in linking the chains into a three-dimensional network (Fig. 2 and Table 2). In addition, there is one intermolecular C—H···π contact (Table 2), identified by *PLATON* (Spek, 2003), which involves the π system of the C7–C12 ring.

Experimental

Compound (I) was synthesized *via* a Baylis–Hillman reaction. At room temperature, a clear solution of 2-hydroxy-5-methoxybenzaldehyde (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 10 d to complete the reaction. The mixture was diluted with water (10 ml) and extracted with ethyl acetate. The organic layer was concentrated under reduced pressure. Chromatography of the crude product on silica gel, using ethyl acetate and petroleum ether (b.p. 333–363 K) (1:4) as eluant, gave the product (I) in 15% yield. The compound (30 mg) was dissolved in $CHCl_3$ (1 ml). The solution was allowed to evaporate slowly at room temperature over several days to yield colourless crystals suitable for X-ray crystallography.

Crystal data

$C_{13}H_{12}O_3$
$M_r = 216.23$
Orthorhombic, *Pbca*
$a = 8.0201$ (9) Å
$b = 12.0980$ (13) Å
$c = 22.173$ (3) Å
$V = 2151.3$ (4) Å^3
$Z = 8$
$D_x = 1.335$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 13031 reflections
$\theta = 3.1–26.9°$
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.972$, $T_{max} = 0.981$
8308 measured reflections

1898 independent reflections
1547 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 14$
$l = -26 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.143$
$S = 1.11$
1898 reflections
146 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.428P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.214 (2)	C2—C6	1.326 (3)
O2—C3	1.427 (2)	C3—C2	1.500 (3)
O2—C12	1.379 (2)	C3—C4	1.519 (3)
O3—C9	1.370 (2)	C4—C5	1.527 (3)
O3—C13	1.414 (3)	C5—C1	1.508 (3)
C1—C2	1.472 (3)	C6—C7	1.454 (2)
O1—C1—C2	125.9 (2)	C1—C5—C4	105.78 (17)
O1—C1—C5	126.6 (2)	C2—C6—C7	118.53 (19)
O2—C3—C2	112.01 (15)	C3—C4—C5	104.98 (16)
O2—C3—C4	112.31 (16)	C6—C2—C1	130.22 (19)
O2—C12—C11	117.81 (17)	C6—C2—C3	120.62 (17)
O2—C12—C7	121.95 (15)	C8—C7—C6	123.12 (18)
O3—C9—C10	115.54 (16)	C12—O2—C3	114.53 (14)
O3—C9—C8	124.93 (19)	C12—C7—C6	117.58 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.98	2.56	3.404 (2)	145
C11—H11···O1^{ii}	0.93	2.48	3.384 (3)	165
C13—H13B···O1^{iii}	0.96	2.49	3.382 (3)	155
C4—H4B···Cg^{iv}	0.98	2.81	3.700 (3)	153

Symmetry codes: (i) $x - \frac{1}{2}, y, \frac{3}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $x - \frac{1}{2}, y, \frac{1}{2} - z$. Cg is the centroid of the C7–C12 ring.

H atoms were placed in calculated positions (0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) values set to $1.5U_{eq}$(parent atom) for the Csp^3-bound H atoms and $1.2U_{eq}$(parent atom) for Csp^2-bound H atoms.

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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