C(21)—N(21)—S(31)	117.3 (2)		C(82)—N(82)—S(72)	118.6 (3)
C(21)—N(21)—P(11)	123.3 (2)		C(82)—N(82)—P(12)	122.0 (3)
S(31)—N(21)—P(11)	118.4 (2)		S(72)—N(82)—P(12)	118.6 (2)
C(81)—N(81)—S(71)	118.5 (3)		C(162)—C(112)—C(122)	119.1 (4)
C(81)—N(81)—P(11)	122.0 (3)		C(162)—C(112)—P(12)	123.7 (3)
S(71)—N(81)—P(11)	118.6 (2)		C(122)—C(112)—P(12)	117.2 (3)
C(121)—C(111)—C(161)	119.0 (4)		C(132)—C(122)—C(112)	120.0 (5)
C(121)—C(111)—P(11)	123.5 (3)		C(142)—C(132)—C(122)	120.9 (5)
C(161)—C(111)—P(11)	117.4 (3)		C(132)—C(142)—C(152)	119.4 (5)
C(111)—C(121)—C(131)	120.7 (4)		C(142)—C(152)—C(162)	120.9 (5)
C(141)—C(131)—C(121)	119.2 (5)		C(112)—C(162)—C(152)	119.7 (4)
C(151)—C(141)—C(131)	121.0 (5)		C(222)—C(22)—C(212)	126.8 (9)
C(141)—C(151)—C(161)	120.3 (5)		C(222)—C(22)—C(232)	91.7 (9)
C(151)—C(161)—C(111)	119.8 (5)		C(212)—C(22)—C(232)	107.0 (6)
C(211)—C(21)—C(231)	109.6 (5)		C(222)—C(22)—N(22)	107.6 (8)
C(211)—C(21)—C(221)	108.8 (5)		C(212)—C(22)—N(22)	110.3 (4)
C(231)--C(21)—C(221)	108.9 (6)		C(232)—C(22)—N(22)	111.5 (5)
C(211)—C(21)—N(21)	110.0 (3)		C(222)—C(22)—C(222')	27.2 (7)
C(231)—C(21)—N(21)	110.8 (3)		C(212)—C(22)—C(222')	104.8 (7)
C(221)—C(21)—N(21)	108.6 (4)		C(232)—C(22)—C(222')	115.9 (7)
C(831)—C(81)—C(811)	110.9 (5)		N(22)—C(22)—C(222')	107.0 (5)
C(831)—C(81)—C(821)	110.2 (5)		C(822)—C(82)—C(832)	109.8 (6)
C(811)—C(81)—C(821)	108.4 (5)		C(822)—C(82)—C(812)	108.6 (6)
C(831)—C(81)—N(81)	107.7 (4)		C(832)—C(82)—C(812)	110.7 (6)
C(811)—C(81)—N(81)	109.5 (4)		C(822)—C(82)—N(82)	109.6 (6)
C(821)—C(81)—N(81)	110.1 (4)		C(832)—C(82)—N(82)	107.8 (5)
O(12)—P(12)—N(82)	111.4 (2)		C(812)—C(82)—N(82)	110.4 (4)
O(12)—P(12)—N(22)	113.4 (2)			

For both compounds, data collection: *MolEN* (Fair, 1990); cell refinement: *MolEN*; data reduction: *Xtal*3.2 (Hall, Flack & Stewart, 1992); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *PLATON*92 (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1044). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Acetoxy-6-(1,1-dimethyl-2-propenyl)-2H-furo[2,3-h][1]benzopyran-2-one

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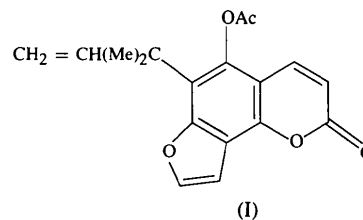Bishan D. Gupta

Regional Research Laboratory, Jammu Tawi, India

Abstract

The title ester {6-(1,1-dimethyl-2-propenyl)-2-oxo-2H-furo[2,3-h][1]benzopyran-5-yl acetate, $C_{18}H_{16}O_5$}, is a furanocoumarin. The pyrone, phenyl and furan rings are planar. Molecules are held together by C—H···O hydrogen bonds.

Comment

Many coumarin derivatives are of biological importance (Michel & Durant, 1976; Schmalle, Jarchow, Hausen & Schulz, 1982). The crystal structure of the title compound has been determined as part of our programme on the crystal structure analysis of these derivatives. The title compound, (I), was prepared by the acetylation of 5-hydroxy-6-(1,1-dimethyl-2-propenyl)-2H-furo[2,3-h][1]-benzopyran-2-one, a furanocoumarin isolated from the roots of *Heracleum thomsoni*, a herb growing wild in the Ladakh region of Jammu and Kashmir State, India (Banerjee, Gupta & Atal, 1980).

(I)

An *ORTEP*II (Johnson, 1976) drawing of the molecule with the atomic numbering scheme is shown in Fig. 1. The furocoumarin atoms are almost coplanar as in other furocoumarin derivatives (Bideau, Bravic & Desvergne, 1979; Bravic & Bideau, 1978; Dall'Acqua, Benetollo & Bombieri, 1981). The deviations from the mean plane of the three rings range from −0.058 (3) to 0.040 (2) Å, indicating a reasonably planar system. Variations in the bond lengths and angles in the phenyl ring, which are due to the fusion of the pyrone and

furan rings, have been reported for various coumarin derivatives (Shimizu, Kashino & Haisa, 1975). Two bonds in the heterocyclic rings, C3—C4 and C8—C9, have distinct double-bond character with lengths of 1.332 (3) and 1.324 (4) Å, respectively. The mean value of the four C—O bonds in the two heterocyclic rings is 1.379 (2) Å and is in agreement with the normal C_{sp^2}—O distance. The lengths of C2—C3 and C4—C10 of 1.442 (3) and 1.443 (3) Å, respectively, are consistent with lengths of the corresponding bonds in furanocoumarin [1.436 (4) and 1.437 (3) Å; Wolska, Borowiak & Gawron, 1990]. The angles around C2 agree with those of furanocoumarin [116.3 (3), 116.7 (3) and 127.0 (3)°]. The angles C12—C13—O1 and C4—C10—C5 at the junction of the phenyl and pyrone rings are smaller than and greater than 120°, respectively, and cause the approach of O1 to C12 and the displacement of C4 away from C5.
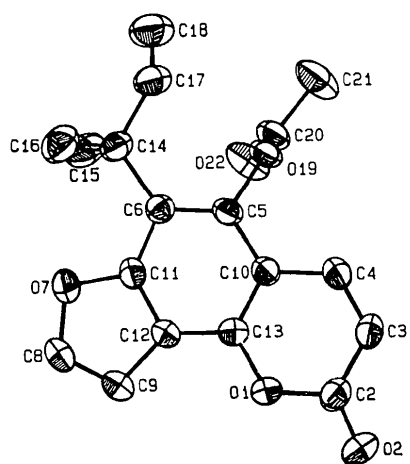

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecule with displacement ellipsoids at the 50% probability level.

The intra- and intermolecular C—H⋯O hydrogen bonds which contribute to the stabilization of the molecular and crystal structures are summarized in Table 2.

Experimental

Dried and ground roots of *Heracleum thomsoni* were extracted successively with hexane and C_6H_6. Chromatography of the hexane extract yielded 5-hydroxy-6-(1,1-dimethyl-2-propenyl)-2*H*-furo[2,3-*h*][1]benzopyran-2-one, which on treatment with Ac₂O–py gave the title compound. Recrystallization was from MeOH; m.p. 401–402 K. The density D_m was measured by flotation in CCl₄ and hexane.

Crystal data

$C_{18}H_{16}O_5$	Mo $K\alpha$ radiation
$M_r = 312.3$	$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 9.238 (1)$ Å
$b = 10.959 (1)$ Å
$c = 15.168 (1)$ Å
$\beta = 90.64 (1)°$
$V = 1535.5$ Å³
$Z = 4$
$D_x = 1.351$ Mg m⁻³
$D_m = 1.349$ Mg m⁻³

Cell parameters from 25 reflections
$\theta = 7.2$–$12.7°$
$\mu = 0.092$ mm⁻¹
$T = 293$ K
Rectangular
$0.35 \times 0.32 \times 0.12$ mm
White transparent

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2674 measured reflections
2437 independent reflections
1950 observed reflections
$[F > 4\sigma(F)]$

$R_{int} = 0.011$
$\theta_{max} = 25°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = -18 \rightarrow 18$
2 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R(F^2 > 2\sigma(F^2)) = 0.042$
$wR(F^2) = 0.110$
$S = 0.899$
1950 reflections
272 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 0.73P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.693$ (for H213)
$\Delta\rho_{max} = 0.19$ e Å⁻³
$\Delta\rho_{min} = -0.15$ e Å⁻³
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.2003 (2)	0.3563 (1)	0.5641 (1)	0.0426 (5)
C2	0.1115 (3)	0.3516 (2)	0.6371 (2)	0.0476 (8)
O2	0.0931 (2)	0.2533 (2)	0.6695 (1)	0.0720 (7)
C3	0.0513 (3)	0.4663 (2)	0.6654 (2)	0.0522 (9)
C4	0.0796 (3)	0.5710 (2)	0.6245 (2)	0.0459 (8)
C5	0.2098 (2)	0.6821 (2)	0.5025 (1)	0.0360 (6)
C6	0.2973 (2)	0.6851 (2)	0.4286 (1)	0.0359 (7)
O7	0.4519 (2)	0.5467 (1)	0.3428 (1)	0.0439 (5)
C8	0.4825 (3)	0.4228 (2)	0.3470 (2)	0.0447 (8)
C9	0.4101 (2)	0.3672 (2)	0.4102 (2)	0.0404 (8)
C10	0.1740 (2)	0.5745 (2)	0.5494 (1)	0.0355 (6)
C11	0.3543 (2)	0.5691 (2)	0.4086 (1)	0.0349 (7)
C12	0.3257 (2)	0.4603 (2)	0.4519 (1)	0.0335 (7)
C13	0.2312 (2)	0.4637 (2)	0.5223 (1)	0.0350 (6)
C14	0.3347 (2)	0.7968 (2)	0.3719 (2)	0.0421 (7)
C15	0.4947 (3)	0.8281 (3)	0.3846 (2)	0.0580 (10)
C16	0.3039 (3)	0.7653 (3)	0.2742 (2)	0.0571 (10)
C17	0.2383 (3)	0.9059 (2)	0.3895 (2)	0.0511 (9)
C18	0.2801 (4)	1.0175 (3)	0.4051 (2)	0.0715 (12)
O19	0.1454 (2)	0.7881 (1)	0.5360 (1)	0.0440 (5)
C20	0.2288 (3)	0.8594 (2)	0.5904 (2)	0.0492 (7)
C21	0.1445 (4)	0.9628 (3)	0.6263 (3)	0.0772 (15)
O22	0.3530 (2)	0.8388 (2)	0.6058 (1)	0.0698 (7)

Table 2. *Selected geometric parameters* (Å, °)

O1—C2	1.386 (3)	C6—C11	1.410 (3)
O1—C13	1.368 (2)	O7—C8	1.388 (2)
C5—C10	1.418 (3)	O7—C11	1.375 (2)
O1—C2—C3	116.0 (2)	C4—C10—C5	124.4 (2)
O1—C2—O2	116.7 (2)	C6—C11—C12	126.8 (2)
O2—C2—C3	127.2 (3)	C11—C12—C13	117.8 (2)
C6—C5—C10	124.4 (2)	O1—C13—C12	118.0 (2)
C5—C6—C11	112.0 (2)		

D—H···A	H···A	D···A	D—H···A
C16—H163···O7	2.31 (3)	2.943 (3)	122 (2)
C9—H9···O22[i]	2.60 (2)	3.155 (3)	118 (2)
C21—H211···O2[ii]	2.79 (5)	3.286 (4)	114 (3)

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

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. Isotropic refinement of the structure by least-squares methods was followed by anisotropic refinement of all the non-H atoms. All H atoms were located from a difference Fourier map and their positions and isotropic displacement parameters were refined (except U_{iso} of H213). All calculations were performed on a PC/AT computer.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Tricyclo[8.2.1.0²,⁹]trideca-5,11-dien-3-ol

FRANK M. BOHNEN, WILHELM F. MAIER AND CARL KRÜGER*

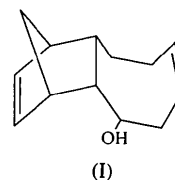*Max-Planck-Institut für Kohlenforschung, Kaiser-Wilhelm-Platz 1, D-45470 Mülheim an der Ruhr, Germany*

Abstract

The cylooctenyl fragment of the title compound, $C_{13}H_{18}O$, adopts a twist-boat conformation. The torsion angle at the ring-fusion bond is 7.0 (2)° and that at the cyclooctene double bond is 0.0 (2)°. The cyclooctenyl C=C bond length is 1.315 (3) Å and the norbornenyl C=C bond length is 1.323 (2) Å. In accord with the space-group symmetry, four molecules are held together by hydrogen bonding, with an intermolecular O···O distance of 2.736 (1) Å.

Comment

The bond lengths and angles C7—C12, C9—C10, C1—C12—C11 and C6—C7—C8 (listed in Table 3) of tricyclo[8.2.1.0²,⁹]trideca-5,11-dien-3-ol, (I), are similar to those of tricyclo[8.2.1.0²,⁹]trideca-5,11-dien-13-one, (II) (Garcia, Fronczek & McLaughlin 1991), endo-13-*syn*-methyltricyclo[8.2.1.0²,⁹]trideca-5,11-dien-13-*anti*-ol, (III) (Garcia, Morales, Fronczek & McLaughlin, 1994), and endo-13-*syn*-methyltricyclo[8.2.1.0²,⁹]trideca-5,11-dien-13-*anti*-yl acetate, (IV) (Garcia, Morales, Fronczek & McLaughlin 1994).

(I)

There is no evidence that the hydroxy group has any influence on the conformation of the cyclooctenyl ring. The atom positions of the comparable known crystal structures were taken from the Cambridge Structural Database (1995) and transferred into *SYBIL*6.1 (Tripos