N4	0,3971 (3)	0,2354 (4)	0,1038 (2)	2,19 (5)
C5	0,5086 (3)	0,2750 (5)	0,1563 (2)	2,44 (6)
O3	0,1683 (2)	0,1925 (4)	0,1316 (2)	3,02 (5)
O5	0,6253 (2)	0,2968 (5)	0,1294 (2)	3,67 (6)
C4	0,3950 (4)	0,2373 (7)	0,0056 (2)	3,38 (7)

Tableau 2. *Paramètres géométriques* (Å, °)

N1—N2	1,418 (3)	N4—C5	1,374 (4)
N1—C5	1,355 (4)	N4—C4	1,457 (4)
N2—C3	1,348 (4)	C5—O5	1,229 (4)
C3—N4	1,373 (4)	N1—H1	0,98 (4)
C3—O3	1,230 (4)	N2—H2	1,04 (5)
N2—N1—C5	107,2 (3)	N1—C5—N4	107,2 (3)
N1—N2—C3	107,3 (2)	N1—C5—O5	126,5 (3)
N2—C3—N4	107,3 (2)	N4—C5—O5	126,2 (3)
N2—C3—O3	127,0 (3)	N2—N1—H1	120 (3)
N4—C3—O3	125,7 (3)	C5—N1—H1	121 (2)
C3—N4—C5	109,4 (3)	N1—N2—H2	117 (3)
C3—N4—C4	125,0 (3)	C3—N2—H2	127 (3)
C5—N4—C4	125,2 (3)		

Tableau 3. *Distances interatomiques* (Å)

$O3\cdots H1^{i}$	1,84 (4)	$O3\cdots N1^{i}$	2,810 (4)
$O5\cdots H2^{ii}$	1,80 (5)	$O5\cdots N2^{ii}$	2,786 (4)

Codes de symétrie: (i) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (ii) $\frac{1}{2}+x, y, \frac{1}{2}-z$.

Tableau 4. *Déviation* (Å) *par rapport au plan moyen de* C3, N4 *et* C5

	C3	N4	C5	N1	N2
Méthylurazole*	0,0	0,0	0,0	−0,060 (3)	0,110 (3)
Urazole†	0,0	0,0	0,0	−0,032 (3)	0,107 (3)

Equations cartésiennes des plans moyens:

$$* \; 0{,}1726x - 0{,}9818y + 0{,}0797z + 0{,}6998 = 0.$$

$$\dagger \; 0{,}9048x + 0{,}3781y - 0{,}1958z - 3{,}6833 = 0.$$

Les paramètres isotropiques de H1 et H2 dont les positions ont été affinées sont fixés à 4. Pour les autres, les valeurs ont été fixées 1,3 fois plus grand que celle de l'atome C4 auquel ils sont attachés.

La résolution de la structure, l'affinement et l'analyse des données cristallographiques ont été faites avec le programme *SDP* (Frenz, 1978) du CAD-4. La structure a été affinée par la méthode des moindres carrés minimisant la fonction $\Sigma w(|F_o|-|F_c|)^2$. La figure a été dessinée par *ORTEP*II (Johnson, 1976).

Ce travail est financé par le Ministère Français de la Coopération que nous remercions. Nous remercions aussi Mr Marcel Pierrot, Directeur de Recherche au Laboratoire de Bio-Inorganique Structurale de l'Université d'Aix-Marseille III, pour les facilités d'accès au diffractomètre et aux équipements informatiques.

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances et angles des atomes d'hydrogène, ont été déposées au dépôt d'archives de l'UICr (Référence: PA1153). On peut en obtenir des copies en s'adressant à: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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7-Hydroxy-4′-methoxyflavanone

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Abstract

The γ-pyrone ring of the title molecule [2,3-dihydro-7-hydroxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one, $C_{16}H_{14}O_4$] adopts the C_2-sofa conformation; the C2 and C3 atoms show disorder. The 2-phenyl substituent is twisted out of the benzo-γ-pyrone ring. The C3—C2—C20—C21 torsion angle is 109.5 (4)° and the C3′—C2′—C20—C21 torsion angle is 16.7 (7)°.

Comment

Many flavonoid compounds have recognized therapeutic properties. The medicinal activity of flavonoids depends upon the structure and relative orientation of the various rings in the molecule (Cody, 1988). The most studied of these structures are the flavones. In contrast, only few single-crystal X-ray studies of flavanones have been reported so far (Mariezcurrena, 1978; Tomlin & Cantrell, 1990; Kendi, Özbey, Ertan, Fun & Yip, 1995). Flavanones have recognized spasmolytic, expectorant,

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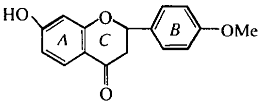
anti-ulcer, liver-protecting, anti-inflammatory and antimicrobial properties (Gabor, 1986). To investigate the spasmolytic activity, a series of flavanone ether derivatives, including the title compound, have been synthesized. The determination of the structure of the title compound, (I), was undertaken to study the conformation of the molecule and to contribute to the growing structural data becoming available for the characterization of structure–activity relationships.



(I)

The γ-pyrone ring (ring *C*) exhibits a distorted C_2-sofa conformation. Atoms C2 and C3 are disordered, with occupancy factors of 0.506 (5) for the sites C2 and C3 and 0.494 (5) for the sites C2′ and C3′. The distances of the disordered atoms from the C9–C10–C4–O1 plane for C2, C3, C2′ and C3′ are 0.473 (6), −0.332 (6), −0.327 (7) and 0.421 (8) Å, respectively. The C2—C3′ bond does not exist. The chirality of the molecule is the same whether the partial atoms C2 and C3 are present or the alternative sites are occupied.

The phenyl ring (ring *B*) is planar; the maximum deviation from the plane is 0.014 Å for C22. The angle between this plane and that of the γ-pyrone ring is 65.2 (1)°. This angle is 70.8° in 5,7,4′-trimethoxyflavanone (Mariezcurrena, 1978), 103.0 (1)° in 7-hydroxyflavanone and 121.1 (2)° in (4-oxo-2-phenyl-2,3-dihydrobenzopyran-7-yl)oxyacetic acid ethyl ester (Kendi, Özbey, Ertan, Fun & Yip, 1995). The methoxy group is slightly twisted with a C24—C23—O23—C11 torsion angle of −2.2 (5)°. The bond distances agree well with the normally accepted values. The C2—C3 bond in ring *C* is single [1.528 (7) Å] and C2′—C3′ is 1.498 (7) Å. This bond is 1.511 (5) Å in 3-chloroflavanone (Tomlin & Cantrell, 1990) and 1.502 (5) Å in 7-hydroxyflavanone.

The structure is stabilized by intermolecular hydrogen bonds between O7 and O4 [O7—H7*A* 0.850 (2), H7*A*···O4 1.842 (2) Å, O7—H7*A*···O4 176.44 (19)°].

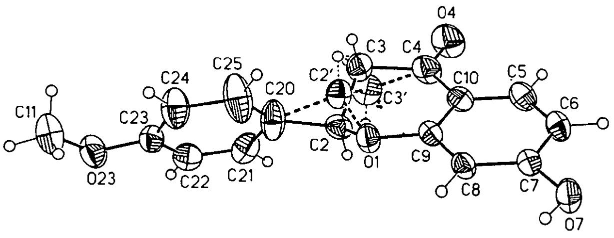


Fig. 1. A view of the molecule showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level.

Experimental

To a solution of 2,4-dihydroxy-4-methoxychalcone (4 g, 0.015 mol) in EtOH (20 ml) an NaOH solution (60 ml, 1.5%) of sodium acetate (20 g) was added. The mixture was heated for 2 h at 373 K and then left overnight at room temperature. Water (80 ml) was added and the mixture was acidified with acetic acid (20%). A precipitate started to separate at pH 7; the final pH was regulated to 5–6. After 2 h in a refrigerator, the precipitate was filtered, washed and dried. The crude product was crystallized from acetone–methanol (yield 51%, m.p. 456–458 K).

Crystal data

$C_{16}H_{14}O_4$
$M_r = 270.27$
Monoclinic
$P2_1/c$
$a = 12.841$ (7) Å
$b = 8.113$ (4) Å
$c = 14.219$ (7) Å
$\beta = 115.89$ (4)°
$V = 1332.6$ (12) Å^3
$Z = 4$
$D_x = 1.347$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 7.5$–15.0°
$\mu = 0.097$ mm^{-1}
$T = 293$ (2) K
Prismatic
0.90 × 0.42 × 0.24 mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3131 measured reflections
3001 independent reflections
1668 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0341$

$\theta_{\rm max} = 27.57°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 10$
$l = -18 \rightarrow 16$
2 standard reflections monitored every 100 reflections intensity decay: 2.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0598$
$wR(F^2) = 0.1614$
$S = 1.276$
2999 reflections
212 parameters
H atoms were generated using *HADD* in *XP* (*SHELXTL/PC*; Sheldrick, 1990*a*) and refined using a riding model

$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.165$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.200$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	*y*	*z*	$U_{\rm eq}$
O1	0.24088 (12)	0.1445 (2)	0.83759 (9)	0.0619 (4)
C2†	0.2368 (4)	0.2876 (6)	0.7811 (3)	0.0592 (12)
C3†	0.2196 (5)	0.4298 (7)	0.8433 (4)	0.070 (2)
C4	0.3286 (2)	0.4398 (3)	0.9527 (2)	0.0788 (7)
C9	0.3182 (2)	0.1379 (2)	0.94006 (13)	0.0515 (5)
C20	0.1266 (3)	0.2834 (3)	0.6799 (2)	0.0907 (9)
C2′‡	0.1889 (5)	0.3072 (5)	0.8037 (4)	0.0614 (13)
C3′‡	0.2755 (5)	0.4446 (8)	0.8374 (5)	0.076 (2)
O4	0.3627 (2)	0.5699 (2)	1.00003 (15)	0.1039 (6)
C5	0.4414 (2)	0.2591 (3)	1.1043 (2)	0.0652 (6)

C6	0.4736 (2)	0.1077 (3)	1.14838 (14)	0.0639 (6)
C7	0.4273 (2)	−0.0328 (3)	1.08669 (13)	0.0546 (5)
O7	0.46299 (13)	−0.1800 (2)	1.13346 (11)	0.0705 (4)
C8	0.3493 (2)	−0.0178 (2)	0.98327 (13)	0.0527 (5)
C10	0.3638 (2)	0.2798 (2)	0.99995 (15)	0.0572 (5)
C11	−0.2110 (3)	0.1647 (4)	0.3367 (2)	0.1029 (10)
C21	0.1525 (2)	0.3363 (3)	0.6008 (2)	0.0841 (8)
C22	0.0748 (2)	0.3225 (4)	0.4986 (2)	0.0795 (7)
C23	−0.0312 (2)	0.2513 (3)	0.4725 (2)	0.0666 (6)
O23	−0.1017 (2)	0.2397 (2)	0.36774 (12)	0.0870 (5)
C24	−0.0602 (2)	0.2005 (4)	0.5492 (2)	0.0926 (9)
C25	0.0184 (3)	0.2177 (4)	0.6522 (2)	0.1135 (11)

† Site occupancy = 0.506 (5).

‡ Site occupancy = 0.494 (5).

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

O1—C9	1.360 (2)	C20—C25	1.375 (4)
O1—C2	1.400 (5)	C5—C6	1.358 (3)
C2—C20	1.515 (5)	C5—C10	1.390 (3)
C2′—C20	1.595 (5)	C6—C7	1.403 (3)
C2—C3	1.528 (7)	C7—O7	1.346 (3)
C2′—C3′	1.498 (8)	C7—C8	1.376 (2)
C3—C4	1.576 (5)	C11—O23	1.412 (3)
C3′—C4	1.476 (7)	C21—C22	1.361 (3)
C4—O4	1.225 (3)	C22—C23	1.372 (3)
C4—C10	1.440 (3)	C23—C24	1.361 (4)
C9—C8	1.385 (3)	C23—O23	1.367 (3)
C9—C10	1.399 (3)	C24—C25	1.376 (3)
C20—C21	1.374 (4)		
C9—O1—C2	118.3 (2)	O1—C2′—C20	101.1 (3)
C9—O1—C2′	113.79 (3)	O1—C2′—C3′	113.5 (4)
O1—C2—C20	108.2 (3)	C4—C3′—C2′	104.7 (4)
O1—C2—C3	105.8 (4)	C6—C5—C10	122.2 (2)
C20—C2—C3	104.8 (4)	C5—C6—C7	119.1 (2)
C2—C3—C4	108.6 (4)	O7—C7—C8	122.6 (2)
O4—C4—C10	123.8 (2)	O7—C7—C6	116.9 (2)
O4—C4—C3	122.4 (3)	C8—C7—C6	120.6 (2)
C10—C4—C3	112.1 (3)	C7—C8—C9	119.3 (2)
C3′—C4—C10	116.1 (3)	C5—C10—C9	117.7 (2)
C3′—C4—O4	117.9 (3)	C5—C10—C4	122.6 (2)
O1—C9—C8	116.5 (2)	C9—C10—C4	119.7 (2)
O1—C9—C10	122.4 (2)	C22—C21—C20	121.5 (3)
C8—C9—C10	121.2 (2)	C21—C22—C23	120.1 (3)
C21—C20—C25	117.5 (2)	C24—C23—O23	124.7 (2)
C21—C20—C2	108.1 (3)	C24—C23—C22	119.8 (2)
C2′—C20—C21	132.9 (3)	O23—C23—C22	115.5 (2)
C2′—C20—C25	108.7 (3)	C23—O23—C11	117.7 (2)
C25—C20—C2	133.5 (3)	C23—C24—C25	119.4 (3)
C20—C2′—C3′	112.8 (4)	C20—C25—C24	121.7 (3)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: AB1220). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cholesteryl 4-(8-Octenyloxy)benzoate

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Abstract

The crystal structure of cholest-5-en-3-yl 4-(8-octenyloxy)benzoate, $C_{42}H_{64}O_3$, was determined by single-crystal X-ray diffraction. The benzoate moiety is twisted 64.6 (2)° from the least-squares plane through the tetracyclic core. Elongated molecules pack in an antiparallel fashion. Fully overlapping molecules are related by 2_1 screw symmetry.

Comment

The title structure, (I), is the second in a series of crystallographic studies of cholesteryl *n*-alkenebenzoate compounds (Socci, Farmer, Chabinyc, Fratini, Bunning & Adams, 1995). When these compounds are attached to cyclosiloxane backbones, a class of intermediate molecular weight liquid crystals is produced (Bunning, 1992; Bunning, Klei, Samulski, Crane & Adams, 1993). Ordered transparent films of these materials have potential use as matrices for optical devices. An understanding of the solid-state structure of cholesteryl esters may provide an insight into the types of packing exhibited by these molecules in the liquid-crystalline state. Relationships between the crystal structures and the packing

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