Absorption correction:
ψ scan (*XSCANS*; Siemens, 1994)
$T_{\min} = 0.172$, $T_{\max} = 0.273$
7512 measured reflections
6067 independent reflections
4258 reflections with $I > 2\sigma(I)$

$h = -1 \rightarrow 15$
$k = -1 \rightarrow 18$
$l = -21 \rightarrow 21$
3 standard reflections every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.096$
$S = 0.934$
6067 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.960$ e Å^{-3}
$\Delta\rho_{\min} = -0.943$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Te—S2	2.528 (1)	Te—I4	2.950 (1)
Te—S1	2.581 (1)	Te—I2	3.100 (1)
Te—I1	2.921 (1)	S1—C1	1.735 (5)
Te—I3	2.933 (1)	S2—C1	1.734 (5)
S2—Te—S1	70.52 (4)	I3—Tc—I4	89.68 (2)
S2—Te—I1	91.09 (4)	S2—Te—I2	151.15 (3)
S1—Te—I1	93.94 (4)	S1—Te—I2	80.63 (3)
S2—Te—I3	90.55 (4)	I1—Te—I2	90.48 (2)
S1—Te—I3	89.16 (4)	I3—Te—I2	89.38 (2)
I1—Te—I3	176.84 (2)	I4—Te—I2	125.34 (2)
S2—Te—I4	83.51 (3)	C1—S1—Te	85.6 (2)
S1—Te—I4	153.99 (3)	C1—S2—Te	87.3 (2)
I1—Te—I4	87.82 (2)		

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1007). Services for accessing these data are described at the back of the journal.

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2,2-Bis(2-methoxyphenyl)-4-methyl-2*H*-benzo[*h*]chromene

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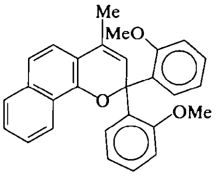
Abstract

In the title compound, $C_{28}H_{24}O_3$, the pyran ring adopts a half-chair conformation. The dihedral angle between the pyran and the naphthalene ring system is 8.50 (6)°. The phenyl rings are nearly orthogonal to each other and they form dihedral angles of 84.21 (7) and 49.89 (8)° with the pyran ring.

Comment

The title compound is a newly added member of the chromane family. Generally, chromane derivatives are very useful in the treatment of inflammation, atherosclerosis, restenosis and immune disorders such as arthritis and transplant rejection (Trivedi, 1998). Furthermore, it has been very recently reported that 3,4-disubstituted chromane derivatives are useful in the prevention and treatment of estrogen-related diseases or syndromes (Jacobson *et al.*, 1998). The structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

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Bond distances and angles in the chromene ring system agree with the reported values (Aldoshin *et al.*, 1996). The pyran ring adopts the half-chair conformation with O1 and C1 deviating from the mean plane by 0.219 (1) and −0.205 (1) Å, respectively; the asymmetry parameter C_2(O1—C1) = 4.6 (2)° (Spek, 1990). The mean plane through the pyran ring makes a dihedral angle of 8.50 (6)° with the naphthalene ring system. The phenyl groups are nearly orthogonal [dihedral angle 86.79 (8)°] to each other and they form dihedral angles of 84.21 (7) and 49.89 (8)° with the best plane through the pyran ring. In the conformation adopted around the C1—C15 bond, the phenyl ring (C15–C20) is eclipsed with respect to the C1—C2 bond; similarly in the conformation around the C1—C22 bond, the phenyl ring (C22–C27) is eclipsed with respect to the O1—C1 bond of the pyran ring. In one of the phenyl rings, the methoxy group is coplanar whereas in the other, it is slightly twisted. Along the *a* direction, the naphthalene rings of the inversion-related molecules are stacked at a perpendicular distance of 3.539 (3)° indicating significant π–π interactions.

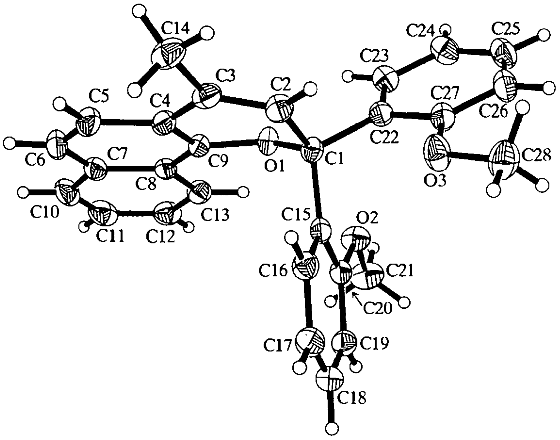

Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme for the non-H atoms.

Experimental

To *o*-anisyllithium (3 equivalents, 7.07 mmol), prepared by the direct metallation of anisole (0.768 ml, 7.07 mmol) using *n*-butyllithium and tetramethylethylenediamine, was added 4-methyl-α-naphthocoumarin (0.5 g, 2.35 mmol) at 253 K under a nitrogen atmosphere. After completion of the reaction, it was quenched by the addition of saturated sodium bicarbonate (3 ml). The organic layer was separated and chromatographed over silica gel to give the title compound in 80–90% yield (m.p. 441–443 K). Single crystals were grown by slow evaporation of the compound in a methanol–chloroform (1:1) solvent system.

Crystal data

$C_{28}H_{24}O_3$
M_r = 408.47
Triclinic
$P\overline{1}$
a = 8.5295 (4) Å
b = 11.4017 (5) Å
c = 11.5115 (5) Å
α = 85.406 (1)°
β = 72.810 (1)°
γ = 80.316 (1)°
V = 1053.71 (8) Å^3
Z = 2
D_x = 1.287 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 4669 reflections
θ = 1.81–28.30°
μ = 0.082 mm^{-1}
T = 293 (2) K
Parallelepiped
0.44 × 0.28 × 0.18 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
7209 measured reflections
4976 independent reflections

3728 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.021
$\theta_{\rm max}$ = 28.3°
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = 0 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.141
S = 1.027
4976 reflections
284 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.1508P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max}$ = 0.24 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.014 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C9	1.372 (2)	O3—C28	1.411 (2)
O1—C1	1.450 (2)	C1—C2	1.518 (2)
O2—C20	1.365 (2)	C2—C3	1.336 (2)
O2—C21	1.417 (2)	C3—C4	1.463 (2)
O3—C27	1.368 (2)	C4—C9	1.377 (2)
C2—C1—C15—C16	−5.3 (2)		
C21—O2—C20—C19	7.5 (2)		
O1—C1—C22—C23	5.4 (2)		
C28—O3—C27—C26	−1.2 (2)		

After checking their presence in the difference map, all the H atoms were placed at geometrically calculated positions and a riding model was used for their refinement; rotating group refinement was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1251). Services for accessing these data are described at the back of the journal.

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9-Hydroxy-4-methyl-8-phenylthio-7,8,9,10-tetrahydrobenzo[*h*]coumarin

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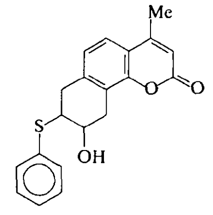
Abstract

The title molecule, $C_{20}H_{18}O_3S$, exists as an enantiomeric pair, the two halves of which are linked by mutual O—H···O hydrogen bonds involving the hydroxyl group and the carbonyl O atom. The tetrahydrobenzene ring adopts a half-chair conformation in one of the enantiomers, while in the other, it is in a conformation intermediate between sofa and half-chair. The coumarin moieties of the molecules are stacked along the *a* direction, with significant π–π interactions.

Comment

The coumarin subunit is of interest because it is found in many natural products displaying diverse biological properties. The range of compounds includes antifungal compounds, anticoagulants and compounds active against psoriasis (Parrish *et al.*, 1974). The amino and hydroxy derivatives of coumarin are widely used in dye lasers (Maeda, 1984). The crystal structure determination of the title compound, (I), one of these coumarin derivatives, was performed in order to elucidate its molecular conformation.



(I)

The molecules of (I) crystallize as an enantiomeric pair in the asymmetric unit of a non-centrosymmetric space group. This pair is linked by mutual O—H···O intermolecular hydrogen bonds involving the hydroxyl group and the carbonyl O atom, to form a dimer. The bond lengths and angles observed in the structure are comparable with those reported for other coumarin derivatives (Chinnakali *et al.*, 1998*a*,*b*; Kumar *et al.*, 1997). The coumarin rings of the two molecules are individually planar. The tetrahydrobenzene ring adopts a half-chair conformation [ΔC_2(C7*A*—C8*A*) = 3.7 (6)°] in one molecule, while in the other it is in a conformation intermediate between sofa and half-chair [ΔC_s(C8*B*) = 10.7 (5)° and ΔC_2(C7*B*—C8*B*) = 10.1 (6)°] (Duax *et al.*, 1976).

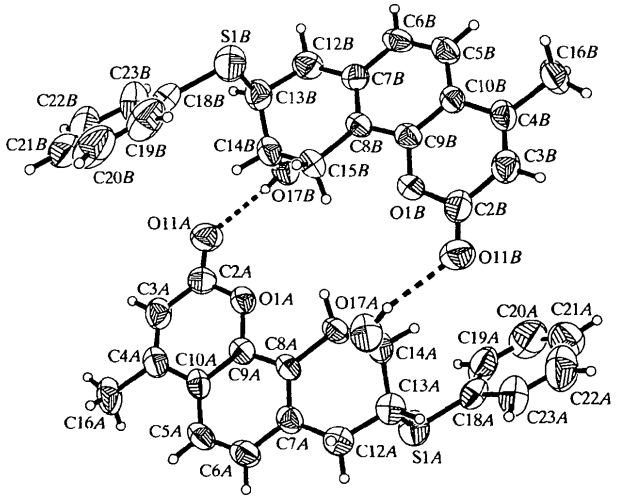


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as spheres of arbitrary radii.

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