Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1205). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 367–368

8-Acetyl-4-methyl-9-phenylthio-7,8,9,10-tetrahydro-7,8-benzocoumarin†

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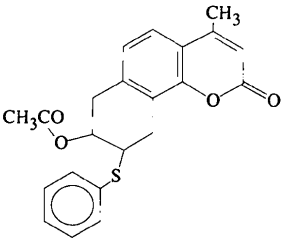
(*Received 27 May 1997; accepted 23 October 1997*)

Abstract

The coumarin and phenyl rings of the title molecule, $C_{22}H_{20}O_4S$, are individually planar. The tetrahydrobenzene ring adopts a half-chair conformation. The crystal structure is stabilized by C—H···O hydrogen bonds involving the carbonyl O atoms.

Comment

The coumarin sub-unit is of interest because it is found in many natural products displaying diverse biological activities. The range of compounds includes antifungal compounds, anticoagulants, and compounds active against psoraris and carcinogens (Parrish *et al.*, 1974; Barry & Toste, 1996). The amino- and hydroxycoumarin derivatives are widely used in dye lasers (Maeda, 1984). The crystal structure determination of the title compound, (I), was undertaken as part of our structural studies on coumarin derivatives.



(I)

Bond lengths and valence angles in the benzocoumarin ring system are comparable with those observed in related derivatives (Chinnakali *et al.*, 1998; Kumar *et al.*, 1997). The coumarin moiety is planar with a maximum deviation of 0.030 (2) Å for C6. The tetrahydrobenzene ring adopts a half-chair conformation with asymmetry parameter ΔC_2(C7–C8) = 0.013 (1) (Nardelli, 1983*a*). The thiophenyl and acetyl groups are planar and make dihedral angles of 62.08 (5) and 95.84 (7)°, respectively, with the best plane through atoms C7, C8, C12 and C15 of the tetrahydrobenzene ring. The carbonyl oxygen is involved in C—H···O hydrogen bonds, the geometries of which are given in Table 2.

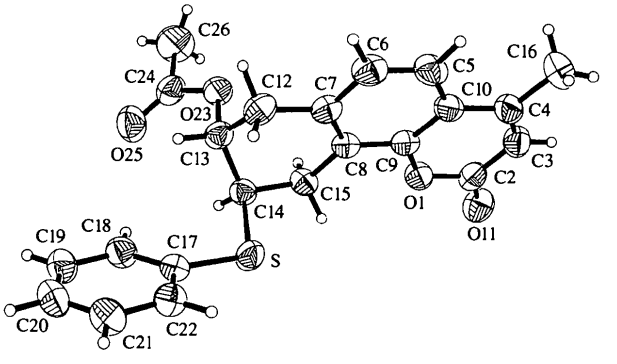


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Ring opening of the compound 4-methyl-7,10-dihydro-7,8-benzocoumarin-8,9-oxide with thiophenyl furnished two regioisomeric hydroxycoumarins, which on acetylation gave the corresponding acetylated compounds (Sriraghavan, 1998). Single crystals were grown by slow evaporation of the compound from a chloroform–methanol solution.

† Alternative name: 4-methyl-9-phenylthio-7,8,9,10-tetrahydrobenzo-[*h*]coumarin-8-yl acetate.

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Crystal data

$C_{22}H_{20}O_4S$
M_r = 380.44
Monoclinic
$P2_1/c$
a = 9.3028 (10) Å
b = 8.5506 (6) Å
c = 23.960 (2) Å
β = 97.636 (8)°
V = 1889.0 (3) Å^3
Z = 4
D_x = 1.338 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 27 reflections
θ = 5.15–12.53°
μ = 0.196 mm^{-1}
T = 293 (2) K
Rectangular block
0.84 × 0.68 × 0.54 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5750 measured reflections
4303 independent reflections
3450 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 27.49°
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 11$
$l = -31 \rightarrow 31$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.116
S = 1.048
4303 reflections
324 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.183P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.007
$\Delta\rho_{max}$ = 0.217 e Å^{-3}
$\Delta\rho_{min}$ = −0.238 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C12—C7—C8—C15	4.0 (2)	C12—C13—C14—C15	−57.7 (2)
C8—C7—C12—C13	−19.1 (2)	C7—C8—C15—C14	−15.7 (2)
C7—C12—C13—C14	46.0 (2)	C13—C14—C15—C8	41.5 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O11^{i}	1.01 (2)	2.58 (2)	3.470 (2)	147 (2)
C19—H19···O11^{ii}	0.98 (2)	2.45 (2)	3.378 (2)	159 (2)

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

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations *PARST* (Nardelli, 1983*b*).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Post Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1196). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 368–370

Diethyl 1-(3,4-Dichlorophenyl)-5-oxo-3-(2-thienyl)-2,2-pyrrolidinedicarboxylate

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(*Received 24 April 1997; accepted 13 November 1997*)

Abstract

In the title molecule, $C_{20}H_{19}Cl_2NO_5S$, the pyrrolidine ring is in an envelope conformation. The dichlorophenyl and thiophene rings are planar. Of the two ethoxycarbonyl side chains, one is nearly planar but the other is distorted from planarity. The structure is stabilized by weak C—H···O hydrogen bonds and van der Waals interactions.

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