We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1358). Services for accessing these data are described at the back of the journal.

References

Antipin, M. Yu., Struchkov, Yu. T., Pisareva, S. A., Medved, T. Ya. & Kabachnik, M. I. (1980). *J. Struct. Chem.* **21**, 644–649.
Carmalt, C. J., Cowley, A. H., Decken, A., Lawson, Y. G. & Norman, N. C. (1996). *Acta Cryst.* C**52**, 931–933.
Carroll, P. J. & Titus, D. D. (1977). *J. Chem. Soc. Dalton Trans.* pp. 824–829.
Grim, S. O. & Walton, E. D. (1980). *Inorg. Chem.* **19**, 1982–1987.
Jones, P. G. & Bembenek, E. (1996). *Acta Cryst.* C**52**, 2396–2399.
Schmidbaur, H., Reber, G., Schier, A., Wagner, F. E. & Müller, G. (1988). *Inorg. Chim. Acta*, **147**, 143–150.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1991). *XSCANS Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*a*). *XEMP. Empirical Absorption Correction Program*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*b*). *XP. Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1997). C**53**, 1854–1855

8-Hydroxy-4-methyl-9-phenylthio-7,8,9,10-tetrahydro-7,8-benzocoumarin

SENTHIL KUMAR,[a] KANDASAMY CHINNAKALI,[a] KANDASAMY SIVAKUMAR,[b] HOONG-KUN FUN[b] AND KAMARAJ SRIRAGHAVAN[c]

[a]*Department of Physics, Anna University, Chennai 600 025, India,* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [c]*Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India. E-mail: hkfun@usm.my*

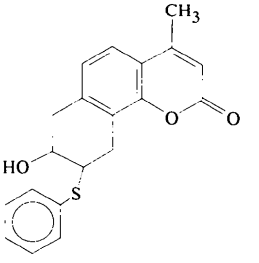
(*Received 12 May 1997; accepted 26 August 1997*)

Abstract

In the title molecule (alternative name: 8-hydroxy-4-methyl-9-phenylthio-7,8,9,10-tetrahydro-2*H*-benzo-[*f*]chromen-2-one; $C_{20}H_{18}O_3S$), the tetrahydrobenzene ring is in a half-chair conformation. The planes of the coumarin and thiophenyl rings form a dihedral angle of 126.31 (5)°. The crystal structure is stabilized by O—H···O hydrogen bonds involving carbonyl and hydroxy O atoms.

Comment

Coumarin derivatives are found in natural products and exhibit antifungal and anticoagulant properties (Parrish *et al.*, 1974; Barry & Toste, 1996). Amino and hydroxy coumarin derivatives are widely used in laser dyes (Maeda, 1984). The crystal structure determination of the title compound, (I), was undertaken as part of our structural studies on coumarin derivatives.



(I)

The coumarin ring system and tetrahydrobenzene ring have normal bond lengths and angles (Chinnakali, Sivakumar & Natarajan, 1992; Chinnakali *et al.*, 1997). The mean value of the C—C lengths in the phenyl ring is 1.376 (3) Å. The coumarin moiety is planar within ±0.029 (1) Å. Planarity of the coumarin system is usually observed (Gnanaguru *et al.*, 1985). The tetrahydrobenzene ring adopts a half-chair conformation with C13 and C14 deviating from the mean plane by −0.329 (2) and 0.436 (2) Å, respectively. The asymmetry parameter (Nardelli, 1983*a*) ΔC_2(C7—C8) is 0.018 (1). The thiophenyl ring is planar and makes a dihedral angle of 126.31 (5)° with the coumarin plane.

In the crystal, the glide-related molecules are linked by O—H···O hydrogen bonds involving the hy-

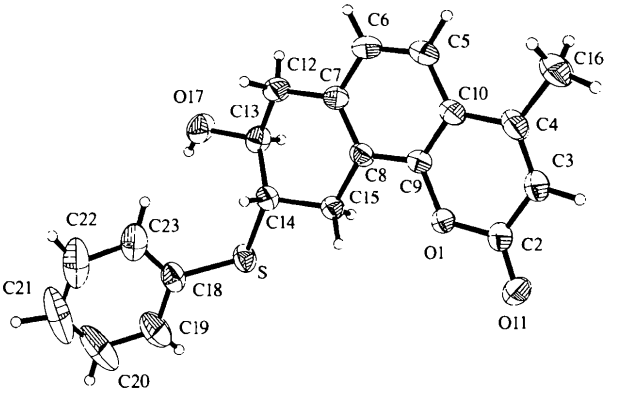


Fig. 1 The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

© 1997 International Union of Crystallography
Printed in Great Britain – all rights reserved

droxy and carbonyl O atoms [O17···O11^{i} 2.967 (2), H17O···O11^{i} 2.15 (3) Å and O17—H17O···O11^{i} 164 (2)°; symmetry code: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$].

Experimental

Ring opening of the compound 4-methyl-7,10-dihydro-8,9-epoxy-7,8-benzocoumarin with thiophenyl resulted in the title compound (Sriraghavan, 1997). Single crystals were grown by slow evaporation of the solvent from a solution of the compound in chloroform–methanol.

Crystal data

$C_{20}H_{18}O_3S$
M_r = 338.40
Monoclinic
$P2_1/c$
a = 5.3861 (6) Å
b = 11.1410 (11) Å
c = 27.411 (3) Å
β = 93.433 (8)°
V = 1641.9 (4) Å^3
Z = 4
D_x = 1.369 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 37 reflections
θ = 5.29–12.48°
μ = 0.212 mm^{-1}
T = 293 (2) K
Thick plate
0.66 × 0.38 × 0.14 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4309 measured reflections
2899 independent reflections
2236 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

θ_{max} = 25°
$h = -1 \rightarrow 6$
$k = -1 \rightarrow 13$
$l = -32 \rightarrow 32$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.118
S = 1.032
2898 reflections
289 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.194 e Å^{-3}
$\Delta\rho_{min}$ = −0.185 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C12—C7—C8—C15	3.7 (3)	C12—C13—C14—C15	−63.2 (2)
C8—C7—C12—C13	−16.7 (3)	C7—C8—C15—C14	−20.7 (3)
C7—C12—C13—C14	46.0 (2)	C13—C14—C15—C8	49.8 (2)

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Structure refinement: *SHELXL93* (Sheldrick, 1993). 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. KS thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1193). Services for accessing these data are described at the back of the journal.

References

Barry, M. T. & Toste, F. D. (1996). *J. Am. Chem. Soc.* **118**, 6305–6306.
Chinnakali, K., Fun, H.-K., Sriraghavan, K. & Ramakrishnan, V. T. (1997). *Acta Cryst.* In the press.
Chinnakali, K., Sivakumar, K. & Natarajan, S. (1992). *Acta Cryst.* C**48**, 386–387.
Gnanaguru, K., Ramasubbu, N., Venkatesan, K. & Ramamurthy, V. (1985). *J. Inorg. Chem.* **50**, 2337–2346.
Maeda, M. (1984). In *Laser Dyes: Properties of Organic Compounds for Dye Lasers*. New York: Academic Press.
Nardelli, M. (1983*a*). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1983*b*). *Comput. Chem.* **7**, 95–98.
Parrish, J. A., Fitzpatrick, T. B., Tanenbaum, L. & Pathak, M. A. (1974). *New Engl. J. Med.* **291**, 206–209.
Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sriraghavan, K. (1997). PhD thesis, University of Madras, India.

Acta Cryst. (1997). C**53**, 1855–1857

5-Amino-1,6-dimethyl-1,2-dihydroquinolin-2-one Monohydrate

KANDASAMY CHINNAKALI,[a]† IBRAHIM ABDUL RAZAK,[a] HOONG-KUN FUN,[a] KAMARAJ SRIRAGHAVAN[b] AND VAYLAKKAVOOR T. RAMAKRISHNAN[b]

[a]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India. E-mail: hkfun@usm.my*

(*Received 23 May 1997; accepted 30 July 1997*)

Abstract

The quinolone ring system of the title compound, $C_{11}H_{12}N_2O.H_2O$, is essentially planar and the water molecule links neighbouring molecules *via* hydrogen bonds.

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.

© 1997 International Union of Crystallography
Printed in Great Britain – all rights reserved