

## References

- Constable, E. C. & Holmes, J. M. (1987). *Inorg. Chim. Acta*, **126**, 187–193.
- Dutta, R. L. & Hossain, Md. M. (1985). *J. Sci. Ind. Res.* **44**, 635–674.
- Fun, H.-K., Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Gong, X.-Y. & Guo, Y.-M. (1997). *Acta Cryst.* **C53**, 1454–1455.
- Fun, H.-K., Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Guo, Y.-M. & Gong, X.-Y. (1997). *Acta Cryst.* **C53**, 1452–1454.
- Lu, Z.-L., Bian, Z.-W., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Chinnakali, K., Razak, I. A. & Fun, H.-K. (1997). *Acta Cryst.* **C53**, 1614–1615.
- Lu, Z.-L., Duan, C.-Y., Tian, Y.-P., You, X.-Z., Fun, H.-K. & Sivakumar, K. (1996). *Acta Cryst.* **C52**, 1507–1509.
- Ma, Y.-X., Lu, Z.-L., Song, Q.-B. & Wu, X.-L. (1994). *J. Coord. Chem.* **32**, 353–359.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). *SADABS. Siemens Area Detector Absorption Correction Software*. University of Göttingen, Germany.
- Siemens (1996a). *SMART. Software Reference Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1996b). *SAINTE. Software Reference Manual*. Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1996c). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1999). **C55**, 91–92

#### 4-Methyl-7,7a,13a,14-tetrahydrobenzo[e]-pyrano[2',3':5,6]naphtho[2,3-b][1,4]dioxin-2-one

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(Received 13 May 1998; accepted 31 July 1998)

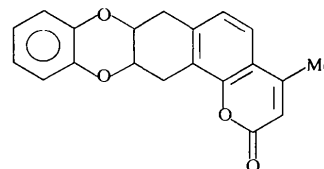
## Abstract

In the title molecule, C<sub>20</sub>H<sub>16</sub>O<sub>4</sub>, the coumarin moiety is planar and both the tetrahydrobenzene and the dioxin rings adopt a half-chair conformation. The mean planes through the tetrahydrobenzene and dioxin rings form a dihedral angle of 72.8 (1)°.

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## Comment

The majority of DNA monointercalating antitumour drugs have a common general structure, comprising a tri- or tetracyclic chromophore to which are attached one or two flexible side chains bearing cationic charges (Palmer *et al.*, 1988). Recently it has been reported that a series of substituted dibenzo[1,4]dioxins show remarkable activity against wild-type P388 leukaemia *in vitro* and *in vivo* (Lee *et al.*, 1992). Because of their antitumour activity and ecotoxicity, different substituted dibenzo[1,4]dioxins have been synthesized and the crystal structure determination of one of them, (I), is reported here.



(I)

Bond lengths and bond angles in the coumarin moiety and C—O distances in the dioxin ring are comparable with reported values (Kumar *et al.*, 1997; Chinnakali *et al.*, 1998; Rissanen *et al.*, 1987). The tetrahydrobenzene ring adopts a half-chair conformation with asymmetry parameter  $\Delta C_2(C8—C7) = 0.045$  (2) (Nardelli, 1983). The dioxin ring also adopts a half-chair conformation with C13 and C14 deviating from the O17—C18—C23—O24 plane by  $-0.436$  (4) and  $0.318$  (4) Å, re-

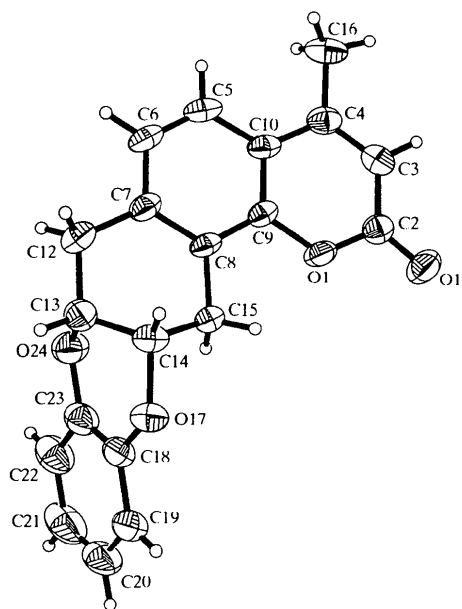


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

spectively [ $\Delta C_2(C13-C14) = 0.020$  (1)]. The coumarin moiety is planar with a maximum deviation at C3 of  $-0.051$  (4) Å. The tetrahydrobenzene ring forms dihedral angles of  $6.98$  (8) and  $72.8$  (1)° with the coumarin and dioxin rings, respectively. The dihedral angle between the phenyl and dioxin rings is  $2.3$  (1)°. The carbonyl-O atom is involved in a weak C—H···O intermolecular hydrogen bond with the methyl-C atom. The crystal structure is stabilized by this weak hydrogen bond and van der Waals interactions.

## Experimental

The title compound was synthesized by ring opening of 4-methyl-7,10-dihydro-7,8-benzocoumarin-8,9-oxide with pyrocatechol followed by cyclization using Mitsunobu reagent (triphenylphosphine–diethylazodicarboxylate) (Sriraghavan & Ramakrishnan, 1998). Single crystals were obtained from ethanol by slow evaporation.

### Crystal data

C<sub>20</sub>H<sub>16</sub>O<sub>4</sub>  
 $M_r = 320.33$   
 Orthorhombic  
*Fdd2*  
 $a = 57.344$  (2) Å  
 $b = 9.7802$  (4) Å  
 $c = 10.6263$  (5) Å  
 $V = 5959.6$  (4) Å<sup>3</sup>  
 $Z = 16$   
 $D_x = 1.428$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 3676 reflections  
 $\theta = 1.42-27.50$ °  
 $\mu = 0.099$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Thin plate  
 $0.56 \times 0.36 \times 0.04$  mm  
 Colourless

### Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 9438 measured reflections  
 3416 independent reflections

2158 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.066$   
 $\theta_{max} = 27.50$ °  
 $h = 0 \rightarrow 73$   
 $k = 0 \rightarrow 12$   
 $l = -13 \rightarrow 13$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.065$   
 $wR(F^2) = 0.155$   
 $S = 1.042$   
 3416 reflections  
 218 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.206$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.238$  e Å<sup>-3</sup>  
 Extinction correction: none  
 Scattering factors from *International Tables for Crystallography* (Vol. C)  
 Absolute structure: Flack (1983)  
 Flack parameter =  $0.92$  (180)

Table 1. Selected geometric parameters (Å)

C3—C4	1.348 (5)	C13—O24	1.448 (4)
C5—C6	1.355 (5)	C13—C14	1.491 (5)
C7—C12	1.502 (5)	C14—O17	1.459 (4)
C8—C15	1.512 (4)	C14—C15	1.514 (5)
C12—C13	1.509 (5)	O17—C18	1.371 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16B···O11 <sup>i</sup>	0.960 (9)	2.468 (8)	3.405 (5)	165.1 (7)

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

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different  $\varphi$  angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in  $\omega$ . The crystal-to-detector distance was 4 cm and the detector swing angle was  $-35$ °. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of the data collection and analysing the duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares techniques. Although all H atoms were located from a difference Fourier map, as the ratio of reflections to parameters was low they were geometrically fixed and allowed to ride on the atoms to which they were attached.

Data collection: SMART (Siemens, 1996a). Cell refinement: SAINT (Siemens, 1996b). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Sheldrick, 1996). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

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: HA1226). Services for accessing these data are described at the back of the journal.

## References

- Chinnakali, K., Fun, H.-K., Sriraghavan, K. & Ramakrishnan, V. T. (1998). *Acta Cryst.* **C54**, 367–368, 542–544.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 878–881.  
 Kumar, S., Chinnakali, K., Sivakumar, K., Fun, H.-K. & Sriraghavan, K. (1997). *Acta Cryst.* **C53**, 1854–1855.  
 Lee, H. H., Palmer, B. D., Boyd, M., Baguley, B. C. & Denny, W. A. (1992). *J. Med. Chem.* **35**, 258–266.  
 Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.  
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.  
 Palmer, B. D., Rewcastle, G. W., Atwell, G. J., Baguley, B. C. & Denny, W. A. (1988). *J. Med. Chem.* **31**, 707–712.  
 Rissanen, K., Valkonen, J. & Tarhanen, J. (1987). *Acta Cryst.* **C43**, 488–490.  
 Sheldrick, G. M. (1996). *SHELXTL Reference Manual*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1996a). *SMART Software Reference Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1996b). *SAINTE Software Reference Manual*. Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sriraghavan, K. & Ramakrishnan, V. T. (1998). To be published.