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(*E*)-7-{[4-(Dimethylamino)cinnamoyl]oxy}-4-methylcoumarin

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The title compound {systematic name: 4-methyl-2-oxo-2*H*-1-benzopyran-7-yl 3-[4-(dimethylamino)phenyl]prop-2-enoate}, $C_{19}H_{13}ClO_4$, has an *E* configuration and the dihedral angle between the coumarin unit and the benzene ring is 81.70 (6)°. The molecules are linked by two C—H···O hydrogen bonds into a $C(4)C(6)[R_2^1(6)]$ chain of rings. Adjacent chains are linked by C—H···O hydrogen bonds rings into stepped sheets. Adjacent sheets are linked by weak C—H···O hydrogen bonds into a three-dimensional network.

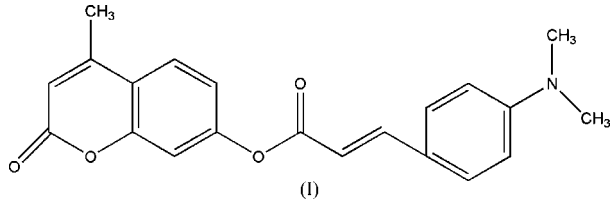
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.147
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Coumarin derivatives exhibit a wide variety of pharmacological activities including anti-HIV activity (Xie, *et al.*, 2001), antibacterial activity (Tanitame *et al.*, 2004) and inhibition of acetylcholinesterase (AChE) (Brühlmann *et al.*, 2001). We have reported the crystal structure of one coumarin derivative with 7-cinnamoyloxy (Yang *et al.*, 2006). As part of our study of the crystal structures of coumarin derivatives with 7-cinnamoyloxy, we report here the crystal structure of a new coumarin derivative, (I).



(I)

In (I) (Fig. 1), the molecules have an *E* configuration, with the coumarin unit and the benzene ring located on opposite sides of the C=C bond. The dihedral angle between the coumarin unit and the benzene ring of the 7-cinnamoyloxy is 81.70 (6)°. The coumarin unit is almost planar, the dihedral angle between the pyrone and the benzene rings being 1.2 (2)°. The geometric parameters for (I) are normal.

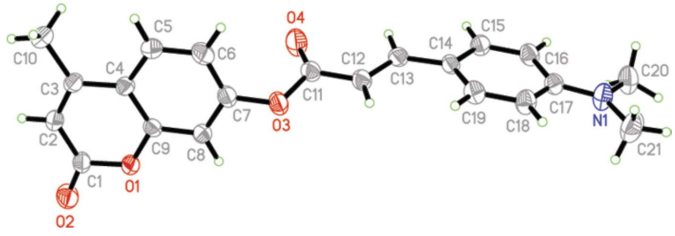


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

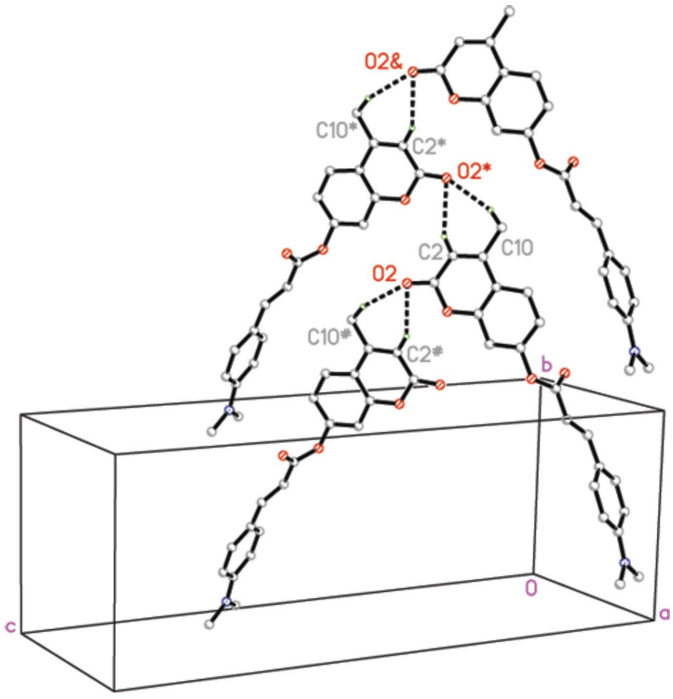


Figure 2
Part of the crystal structure of (I), showing the formation of a $C(4)$ $C(6)R_2^1(6)$ chain of rings along the [010] direction. For the sake of clarity, H atoms not involved in the motif shown have been omitted [symmetry codes: (*) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (#) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (&) $x, 1+y, z$]. Dashed lines indicate hydrogen bonds.

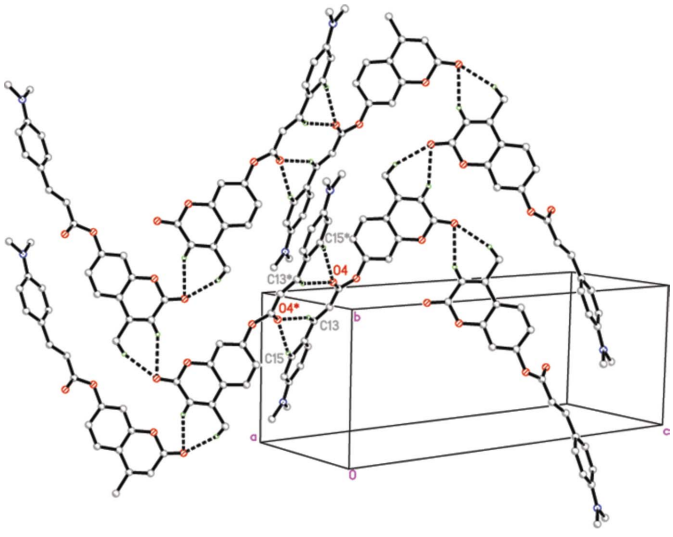


Figure 3
Part of the crystal structure of (I), showing the formation of a sheet. For the sake of clarity, H atoms not involved in the motif shown have been omitted [symmetry codes: (*) $1-x, 2-y, -z$]. Dashed lines indicate hydrogen bonds.

In the crystal structure of (I), the molecules are linked by C—H···O hydrogen bonds into a $C(4)C(6)R_2^1(6)$ chain of rings (Bernstein *et al.*, 1995) along the [010] direction. Atoms O2 act as a bifurcated acceptor, and atoms C2 and C10 in the molecule at (x, y, z) both act as hydrogen-bond donors to atom

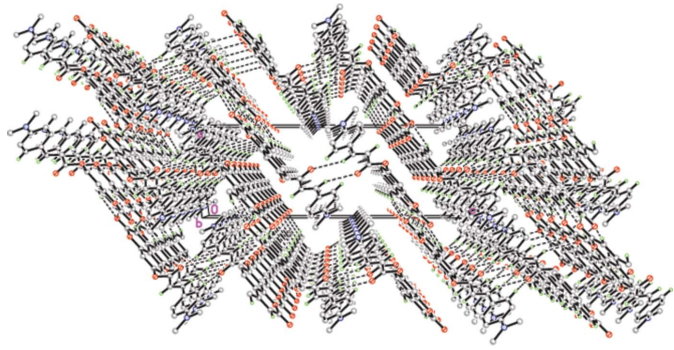


Figure 4
The packing of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

O2 in the molecule at $(-x, \frac{1}{2}+y, \frac{1}{2}-z)$ (Fig.2). Adjacent chains of molecules of (I) are linked by four C—H···O hydrogen bonds into a stepped sheet, so generating an $R_2^1(6)[R_2^2(10)R_2^2(14)]$ motif (García-Báez *et al.*, 2002) (Fig.3). Adjacent sheets are linked by one weak intermolecular C—H···O interaction [C12—H12···O1^{ii}, symmetry code: (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$], resulting in the formation of a three-dimensional network (Fig. 4 and Table 1).

Experimental

To a solution containing 4-methyl-7-hydroxycoumarin (1.76 g, 10 mmol) and anhydrous pyridine (10 ml), a solution of 4-dimethylaminocinnamyl chloride (2.09 g, 10 mmol) and anhydrous acetone (10 ml) was slowly added at 278–283 K, with stirring for 30 min. The reaction mixture was stirred continuously for 24 h at room temperature (298–300 K), and then poured into ice–water (200 ml). The resulting solid was filtered off, washed with water and dried at room temperature. Yellow crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the crude product from ethanol (m.p. 471–473 K).

Crystal data

$C_{21}H_{19}NO_4$
$M_r = 349.37$
Monoclinic, $P2_1/c$
$a = 8.174$ (3) Å
$b = 9.071$ (3) Å
$c = 24.175$ (9) Å
$\beta = 97.670$ (6)°
$V = 1776.5$ (11) Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.53 × 0.42 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.954$, $T_{max} = 0.969$
9015 measured reflections
3135 independent reflections
1480 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.147$
$S = 1.01$
3135 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0116 (15)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2^{i}	0.93	2.56	3.423 (4)	154
C10—H10A···O2^{i}	0.96	2.51	3.426 (4)	160
C12—H12···O1^{ii}	0.93	2.76	3.619 (3)	154
C13—H13···O4^{iii}	0.93	2.52	3.371 (3)	152
C15—H15···O4^{iii}	0.93	2.59	3.402 (4)	145

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(methyl C), and C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens,1996); cell refinement: *SAINT* (Siemens,1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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