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Dimethyl 1-(7-acetoxy-4-methyl-2-oxo-2*H*-chromen-8-ylmethyl)-1*H*-pyrazole-3,4-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.148
Data-to-parameter ratio = 17.0

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

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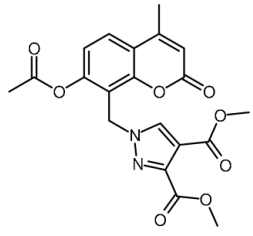
In the title compound, $C_{20}H_{18}N_2O_8$, the coumarin moiety is oriented approximately perpendicular to the plane of the pyrazole ring. Weak intermolecular C—H···O interactions link the molecules into a complex network that can be represented by $C(X)$ chains (X is 8, 10, 11 and 13) and $R_2^2(26)$ and $R_2^2(10)$ rings.

Comment

Coumarins (2*H*-1-benzopyrans) possess a variety of biological activities, such as antibacterial, antifungal, antimicrobial, anticancer, anti-ulcer and antifeedant (Thamotharan *et al.*, 2004, and references therein). Pyrazoles and their derivatives have been reported to show analgesic and anti-inflammatory activities (Thamotharan *et al.*, 2003, and references therein). The title compound, (I), exhibits good antimicrobial activity in preliminary screening. As part of our ongoing studies of coumarin derivatives, the crystal structure analysis of (I) has been carried out and the results are presented here.



(I)

A view of the molecule of (I), with the atomic numbering scheme, is shown in Fig. 1. The bond lengths and angles in the coumarin moiety in (I) are comparable with those of a related structure (Thamotharan *et al.*, 2004). The bond lengths and angles in the pyrazole moiety are comparable to those found in related compounds (particularly, N2—N1—C5 > N1—N2—C3 and N2—C3—C4 > N1—C5—C4; Table 1; Thamotharan *et al.*, 2003). The dihedral angle between the planes of the coumarin moiety and the pyrazole ring is 71.44 (5)°. The planes of the 3- and 4-carboxylate groups are oriented at angles of 60.59 (14) and 8.89 (14)°, respectively, with respect to the plane of the pyrazole moiety. The dihedral angle between the planes of the coumarin moiety and its attached carboxylate group is 69.33 (13)°.

In the crystal structure, atom C11 is involved in an intermolecular C—H···O interaction with carbonyl atom O17 of an adjacent molecule (Table 2). This interaction links the molecules into a chain that runs parallel to the *a* axis and has a graph-set motif of $C(11)$ (Bernstein *et al.*, 1995). Atom C12 has an intermolecular C—H···O interaction with another

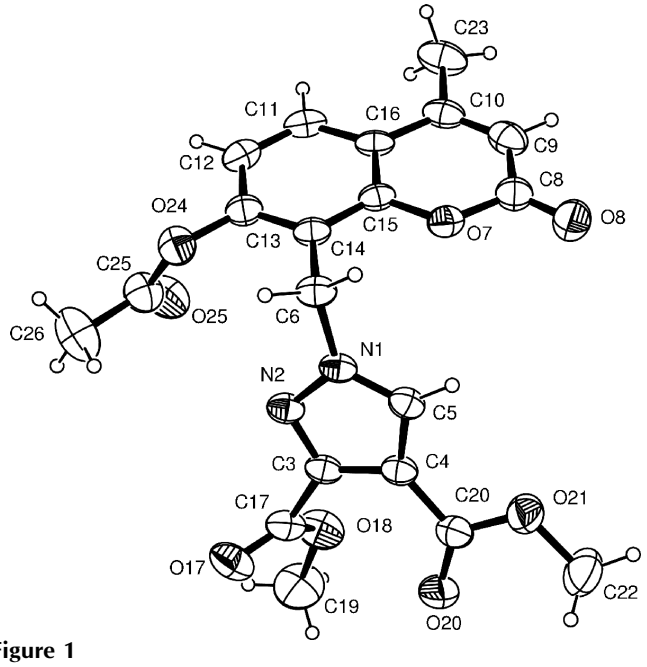


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

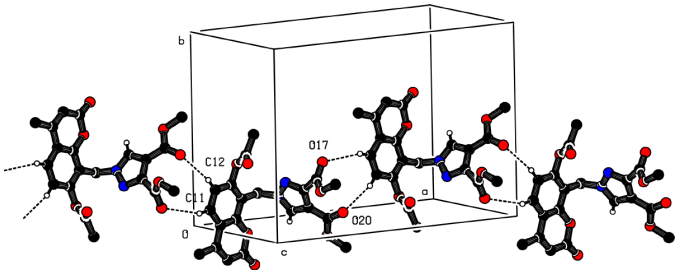


Figure 2
Part of the crystal structure of (I), showing the formation of a chain of $R_2^2(10)$ rings.

carbonyl atom O20 of an adjacent molecule. This interaction also links the molecules into a chain which runs parallel to the *a* axis and has a graph-set motif of *C*(10). These two interactions combine to link the molecules into dimers that have a graph-set motif of $R_2^2(10)$ (Fig. 2). Atom C19 (*via* H193) acts as a donor for a weak intermolecular C—H···O interaction with atom O21 and this interaction links the molecules into a *C*(8) chain which runs parallel to the *b* axis. Atom C22 acts as donor, through both H222 and H223, in weak intermolecular C—H···O interactions with atoms O21 and O8, respectively, in different neighbouring molecules. The former interaction produces loops that have a graph-set motif of $R_2^2(26)$, while the latter interaction generates a chain which runs parallel to the *b* axis and has a graph-set motif of *C*(13). Details of all the hydrogen bonds are given in Table 2.

Experimental

The title compound, (I), was prepared by heating 3-[(7-acetoxy-4-methylcoumarin-8-yl)methyl]sydnone in dry xylene at 403 K for about 3 h, cooling the reaction mixture and treating with petroleum ether to yield a yellow solid, which was crystallized from absolute ethanol (m.p. 458–459 K).

Crystal data

$C_{20}H_{18}N_2O_8$	D_x = 1.365 Mg m^{-3}
M_r = 414.36	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3600 reflections
a = 23.1233 (17) Å	θ = 2.5–26.0°
b = 12.7597 (9) Å	μ = 0.11 mm^{-1}
c = 15.2076 (11) Å	T = 273 (2) K
β = 116.032 (1)°	Prism, colourless
V = 4031.7 (5) Å^3	0.25 × 0.18 × 0.10 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	3177 reflections with $I > 2\sigma(I)$
	R_{int} = 0.021
ω scans	θ_{max} = 28.0°
Absorption correction: none	$h = -30 \rightarrow 30$
12054 measured reflections	$k = -15 \rightarrow 16$
4672 independent reflections	$l = -17 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 0.4379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	
$wR(F^2)$ = 0.148	
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
4672 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
275 parameters	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected bond angles (°).

C5—N1—N2	112.81 (12)	N2—C3—C4	112.18 (13)
C3—N2—N1	103.86 (13)	N1—C5—C4	107.25 (14)

Table 2
C—H···O interactions (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O17^{i}	0.93	2.56	3.367 (2)	145
C12—H12···O20^{i}	0.93	2.49	3.349 (2)	154
C19—H193···O21^{ii}	0.96	2.57	3.436 (3)	150
C22—H222···O8^{iii}	0.96	2.47	3.421 (3)	172
C22—H223···O25^{iv}	0.96	2.55	3.264 (3)	131

Symmetry codes: (i) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iii) $\frac{1}{2}-x, \frac{1}{2}-y, 2-z$; (iv) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All the methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(C). All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms, with 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: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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