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3-[(7-Acetoxy-4-methylcoumarin-8-yl)-methyl]sydnone

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.052
wR factor = 0.151
Data-to-parameter ratio = 19.2

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

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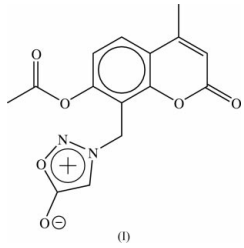
In the title compound, $C_{15}H_{12}N_2O_6$, the sydnone ring is oriented nearly perpendicular to the plane of the coumarin moiety. Weak intermolecular C—H···O interactions link the molecules into a complex network that can be described by $C(X)$ chains (X is 6, 8 and 9) and $R_2^2(20)$ rings.

Comment

Sydnones are relatively non-toxic, but potent, porphyrinogenic and anti-inflammatory compounds (Thamotharan, Parthasarathi, Sanyal *et al.*, 2003; Thamotharan, Parthasarathi, Hunnur *et al.*, 2003; and references therein). Coumarins (2*H*-1-benzopyrans) possess a variety of biological activities such as antibacterial (Ahluwalia *et al.*, 1989), antifungal (Bhakuni & Chaturvedi, 1984), antimicrobial (Ahluwalia *et al.*, 1987), anticancer (Gschwendt *et al.*, 1984), anti-ulcer (Kyogoku *et al.*, 1979) and antifeedant (Simmonds *et al.*, 1990). It was also found that coumarins display a very strong anti-invasive activity *in vitro* against human mammary carcinoma cells (Parmar *et al.*, 1994). In view of their importance, the crystal structure of the title compound, (I), was determined.



(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 sydnone moiety in (I) are comparable with those of related compounds (Thamotharan, Parthasarathi, Sanyal *et al.*, 2003; Thamotharan, Parthasarathi, Hunnur *et al.*, 2003). No unusual bond lengths or angles were observed in the coumarin moiety (Vijayalakshmi *et al.*, 2000). The dihedral angle between the planes of the sydnone ring and the coumarin moiety is 79.94 (4)°.

In the crystal structure, carbonyl atom O5 of the sydnone moiety accepts two different intermolecular C—H···O interactions, one from the C6—H61 group in one neighbouring molecule and the other from the C6—H62 group in a different neighbouring molecule (Table 1). These two interactions link the molecules into two different continuous chains and each has a graph-set motif of $C(6)$ (Bernstein *et al.*, 1995) running parallel to the *b* and *c* axes, respectively. Another carbonyl atom, O8, accepts two different intermolecular C—H···O interactions, from the C4—H4 and C12—H12 groups in different adjacent molecules. These two interactions also

produce two different chains. The former interaction has a graph-set motif of $C(9)$, while the latter has a $C(8)$ motif, both running parallel to the c axis. Atom C9 is involved in an intermolecular C—H···O interaction with atom O18 of a neighbouring centrosymmetrically related molecule. This interaction produces loops that have a graph-set motif of $R_2^2(20)$. Finally, the disordered atom H194 on C19 has an intermolecular C—H···O interaction with atom O1 of the sydnone moiety of another neighbouring centrosymmetrically related molecule. This interaction also links the molecules into dimers and generates a graph-set motif of $R_2^2(20)$.

Experimental

The preparation of the title compound will be described in a future publication. Recrystallization from absolute ethanol gave colourless crystals which were suitable for crystallographic analysis (m.p. 453–455 K).

Crystal data

$C_{15}H_{12}N_2O_6$	D_x = 1.515 Mg m^{-3}
M_r = 316.27	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4215 reflections
a = 11.5067 (3) Å	θ = 2.0–30.0°
b = 13.1916 (3) Å	μ = 0.12 mm^{-1}
c = 9.1773 (2) Å	T = 160 (2) K
β = 95.6210 (9)°	Plate, colourless
V = 1386.34 (6) Å^3	0.30 × 0.30 × 0.05 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.074
φ and ω scans with κ offsets	θ_{max} = 30.1°
Absorption correction: none	$h = -16 \rightarrow 16$
37980 measured reflections	$k = -18 \rightarrow 18$
4057 independent reflections	$l = -12 \rightarrow 12$
2403 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.151	$(\Delta/\sigma)_{max} < 0.001$
S = 1.02	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
4056 reflections	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
211 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O8^{i}	0.95	2.31	3.237 (2)	166
C6—H61···O5^{ii}	0.99	2.51	3.451 (2)	159
C6—H62···O5^{iii}	0.99	2.58	3.3715 (19)	137
C9—H9···O18^{iv}	0.95	2.48	3.319 (2)	148
C12—H12···O8^{v}	0.95	2.58	3.2878 (18)	132
C19—H194···O1^{vi}	0.98	2.53	3.367 (2)	143

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

The H atoms of the ester methyl group are disordered over two orientations and two sets of idealized positions rotated from each other by 60° were used. The major conformation is present in 52 (2)% of the molecules. All methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.95–0.99 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C). Reflection 011 was partially obscured by the beam stop and was omitted.

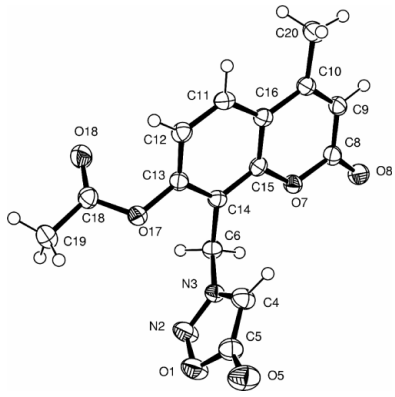


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The minor conformation of disordered methyl H atoms is not shown.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); 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 (Version 1.07; Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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