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Methyl [8-(3,5-dimethylpyrazol-1-ylmethyl)-4-methyl-2-oxo-2*H*-chromen-7-yloxy]acetate 0.19-hydrate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
H-atom completeness 99%
Disorder in main residue
R factor = 0.059
wR factor = 0.161
Data-to-parameter ratio = 14.1

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

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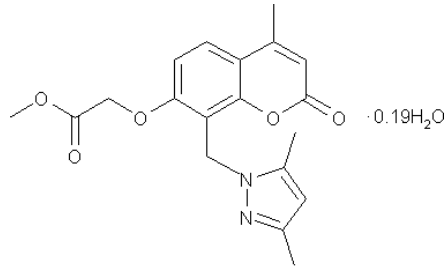
In the title compound, $C_{19}H_{20}N_2O_5\cdot0.19H_2O$, the coumarin moiety is oriented almost perpendicular to the plane of the pyrazole ring. The crystal structure is stabilized by weak intermolecular C—H$\cdots\pi$ interactions.

Comment

Coumarins (2*H*-1-benzopyrans) possess a variety of biological activities such as antibacterial, antifungal, antimicrobial, anticancer, anti-ulcer and antifeedant (Thamotharan, Parthasarathi, Mallur *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), shows good antimicrobial activity in preliminary screening. As part of our ongoing studies of coumarin derivatives, the crystal structure analysis of the title compound, (I), has been carried out.



(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 related structures (Thamotharan, Parthasarathi, Mallur *et al.*, 2004; Thamotharan, Parthasarathi, Shinge *et al.*, 2004). The bond lengths and angles in the pyrazole moiety are comparable to those found in related structures [particularly, N2—N1—C5 > N1—N2—C3 and N2—C3—C4 > N1—C5—C4 (Table 1); Thamotharan *et al.*, 2003; Thamotharan, Parthasarathi, Shinge *et al.*, 2004). The coumarin moiety is oriented at an angle of 83.24 (6)° with respect to the plane of the pyrazole ring. The corresponding angle was found to be 71.44 (5)° in a related structure (Thamotharan, Parthasarathi, Shinge *et al.*, 2004). The dihedral angle between the planes of the acetic acid methyl ester group and the coumarin moiety is 6.64 (11)°. Carbonyl atom O19 of the acetic acid methyl ester group is disordered over two sites, with the major conformation existing in 51.2 (6)% of the molecules.

In the crystalline state, atom C18 (*via* H182) is involved in a weak intermolecular C—H$\cdots\pi$ interaction with the centroid (*Cg*1) of the ester group attached to the six-membered ring of

a centrosymmetrically related molecule (Table 2). An intermolecular short contact is observed between one of the disordered carbonyl atoms, O19*A*, and O1*W* (2.75 Å).

Experimental

The title compound, (I), was prepared by refluxing [8-(3,5-dimethylpyrazol-1-yl)methyl]-4-methyl-2-oxo-2*H*-chromen-7-yloxy]acetic acid hydrazide (0.01 mol) with methanol (5 ml) and acetyl acetone (0.01 mol) on a water bath for about 8 h. The title compound was recrystallized from absolute ethanol (m.p. 423–426 K).

Crystal data

$C_{19}H_{20}N_2O_5\cdot0.19H_2O$	$Z = 2$
$M_r = 359.79$	$D_x = 1.327$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.8151$ (9) Å	Cell parameters from 1506 reflections
$b = 10.6692$ (11) Å	$\theta = 2.5$–$25.0°$
$c = 10.9050$ (11) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 96.281$ (2)°	$T = 273$ (2) K
$\beta = 108.032$ (2)°	Prism, colourless
$\gamma = 108.383$ (2)°	0.22 × 0.14 × 0.08 mm
$V = 900.79$ (16) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2476 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.013$
ω scans	$\theta_{max} = 28.0°$
Absorption correction: none	$h = -11 \rightarrow 10$
5544 measured reflections	$k = -13 \rightarrow 14$
3630 independent reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 0.1112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.059$	
$wR(F^2) = 0.161$	
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3630 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
258 parameters	$\Delta\rho_{min} = -0.12$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected bond angles (°).

C5—N1—N2	112.48 (16)	N2—C3—C4	110.70 (18)
C3—N2—N1	104.63 (16)	N1—C5—C4	105.55 (18)

Table 2
C—H···π interaction (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H182···*Cg*1^{i}	0.97	2.78	3.564 (3)	139

Symmetry code: (i) $1-x, 2-y, -z$. *Cg*1 is the centroid of ???????.

Carbonyl atom O19 is disordered over two sites and two positions were assigned for carbonyl atom O19. Refinement of the site-occupation factor for this atom led to a value of 0.512 (6) for the major conformation. Similarity restraints were applied to the disordered atoms, so as to maintain similar geometry about the chemically equivalent atoms. 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 $U_{iso}(H) = 1.2U_{eq}(C)$. At this stage, the maximum difference density of 0.63 e Å^{-3} indicated the presence of a possible additional atom. This peak was found near H241, at a distance of 2.26 Å. Attempts to refine this peak as a water oxygen (O1*W*) with full occupancy yielded a high U_{iso} value and hence it was refined with partial occupancy. The occupancy of O1*W* was found to be 0.19 (1) in the subsequent refinement and this value was fixed during the final cycle of refinement. The H atoms of the water molecule could not be located.

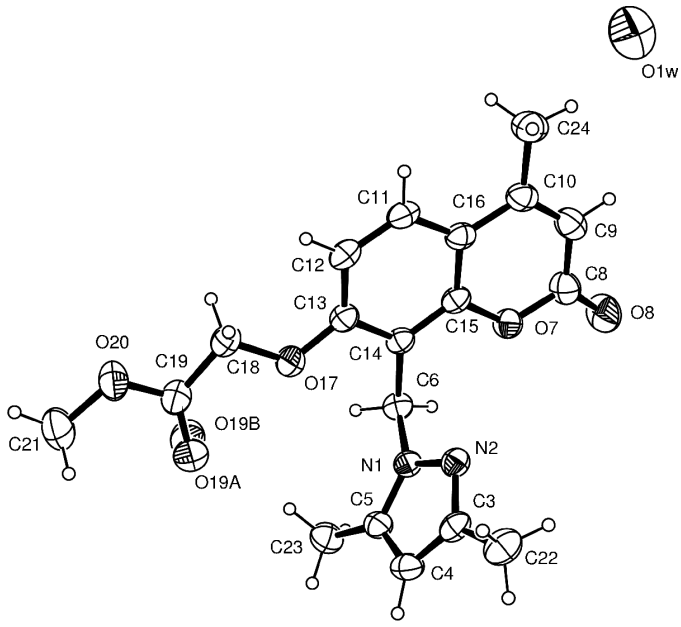


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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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