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3-(4,6-Dimethylpyrimidin-2-ylsulfanyl)-2,2-dimethylchroman-4-one

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Key indicators

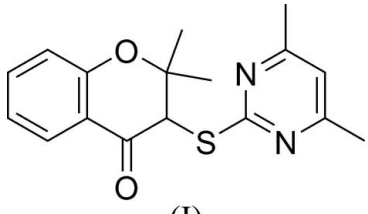
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.181
Data-to-parameter ratio = 15.7

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

In the title compound, $C_{17}H_{18}N_2O_2S$, the six-membered heterocyclic ring of the chromanone unit adopts a half-chair conformation. The crystal structure is stabilized by intermolecular C—H$\cdots\pi$ and π–π interactions.

Comment

4-Chromanone derivatives are well known to exhibit a wide spectrum of biological activities, including antiviral, anticancer and antibiotic properties (Cho *et al.*, 1996). In addition, pyrimidine derivatives also have good biological activities (Wang *et al.*, 1995). These findings prompted us to synthesize a new series of chromanone derivatives by incorporating pyrimidine at the 3-position, in the hope of finding better bioactive molecules. We present here the X-ray crystallographic analysis of the title compound, (I), which was designed and synthesized in our laboratory.



(I)

The six-membered heterocyclic ring of the chromanone unit of (I) adopts a half-chair conformation (Fig. 1). The pyrimidine ring and the chromanone unit are twisted; the benzene ring makes a dihedral angle of 82.02 (2)° with the pyrimidine ring.

The crystal packing of (I) is consolidated by intermolecular C—H$\cdots\pi$ interactions (Table 1; *Cg*1 and *Cg*2 are the centroids of the benzene and pyrimidine rings, respectively) and a π–π stacking interaction between adjacent pyrimidine rings related by an inversion centre. The centroid-to-centroid and interplanar distances are 3.462 (2) and 3.282 (2) Å, respectively (Fig. 2).

Experimental

A mixture of 4,6-dimethylpyrimidine-2-thiol (2.0 mmol), acetone (20 ml) and anhydrous potassium carbonate (2.1 mmol) was refluxed for 1 h and then 3-bromo-2,2-dimethylchroman-4-one (2.0 mmol) was added dropwise. After refluxing the mixture for 3 h, the resultant precipitate was filtered off and washed with acetone. The residue was purified by column chromatography on silica gel with hexane–diethyl ether (10:1 *v*/*v*) as eluent to afford (I) (yield 76%, m.p. 437 K). ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.90 (*d*, 1H, C14-H), 7.51 (*t*, 1H, C13-H),

7.02 (*m*, 2H, C11-H, C12-H), 6.72 (*s*, 1H, C4-H), 5.38 (*s*, 1H, C7-H), 2.41 (*s*, 6H, C16-H, C17-H), 1.66 (*s*, 3H, C5-H), 1.50 (*s*, 3H, C3-H). Crystals of (I) suitable for X-ray diffraction were grown from a methanol solution at 292 K.

Crystal data

$C_{17}H_{18}N_2O_2S$
M_r = 314.39
Triclinic, $P\overline{1}$
a = 8.3523 (12) Å
b = 9.1835 (13) Å
c = 11.4241 (16) Å
α = 92.954 (2)°
β = 99.926 (3)°
γ = 105.329 (2)°
V = 828.1 (2) Å^3
Z = 2
D_x = 1.261 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.04 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
4725 measured reflections
3191 independent reflections
1955 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.181
S = 0.97
3191 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1018P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···N1	0.98	2.28	2.905 (3)	121
C16—H16B···S1	0.96	2.80	3.216 (4)	107
C1—H1B···$Cg1^{i}$	0.96	2.97	3.614 (4)	125
C14—H14···$Cg2^{ii}$	0.93	2.89	3.702 (4)	146

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $x, y-1, z$.

H atoms were initially located in a difference Fourier map. Methyl H atoms were then constrained to an ideal geometry, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. Other H atoms were placed in geometrically idealized positions, with C—H = 0.93–0.98 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

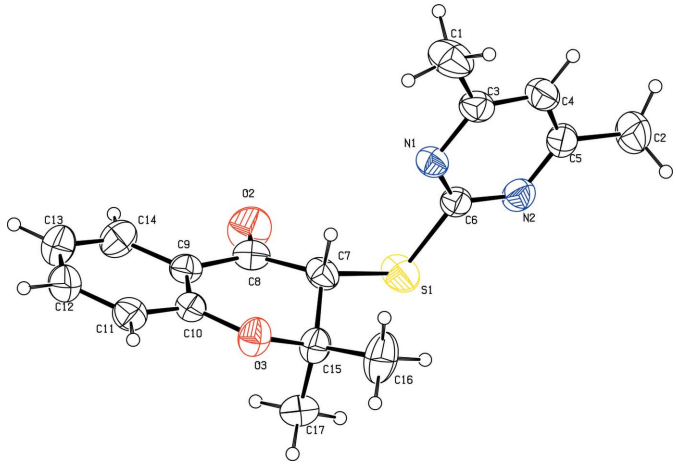


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

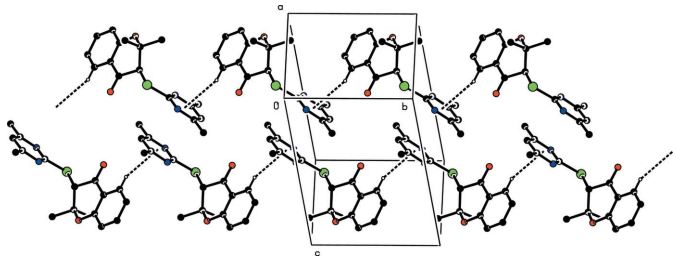


Figure 2
A partial packing diagram of (I), showing intermolecular C—H···π (dashed lines) and π–π stacking interactions.

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