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Key indicators

Single-crystal X-ray study
 T = 292 K
 Mean $\sigma(C-C)$ = 0.005 Å
 R factor = 0.056
 wR factor = 0.192
 Data-to-parameter ratio = 13.1

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

3-(4,6-Dimethylpyrimidin-2-yl)-2-methyl- chromen-4-one

In the crystal structure of the title compound, $C_{16}H_{14}N_2O_2S$, intermolecular C—H...O and C—H...N hydrogen bonds and π - π stacking interactions are observed.

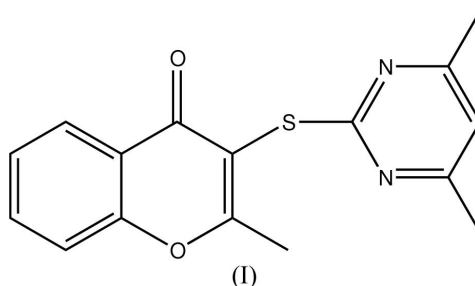
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Comment

Chromones are a very important class of active natural products that occur in aloes and other plants, and they are often used as synthetic leads for drug discovery. Recently, the structural modification of the chromone backbone by the introduction of a heterocyclic substituent at either the 2- or 3-position has attracted considerable attention. For example, some chromones bearing heterocyclic thioether substituents were reported to be anticancer agents (Kim *et al.*, 2004).



In this paper, we present the X-ray crystallographic analysis of the title compound, (I). The bond lengths and angles are unremarkable. The dihedral angle between the pyran ring plane and the pyrimidine plane is 71.1 (2)° (Fig. 1). As shown in Fig. 2, the molecules are linked by C—H...O intermolecular hydrogen bonds. Atoms C16 in adjacent molecules

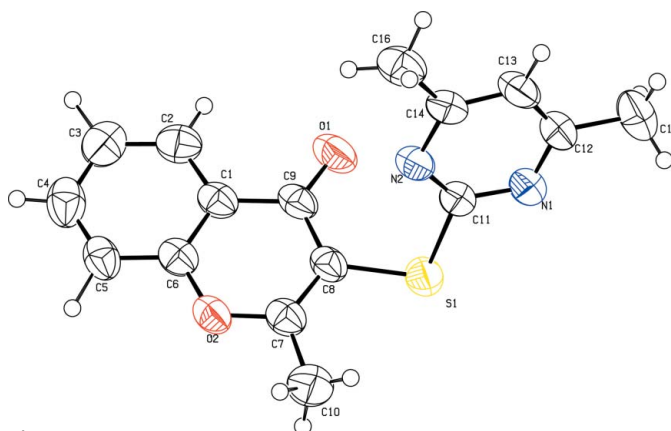


Figure 1
 View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

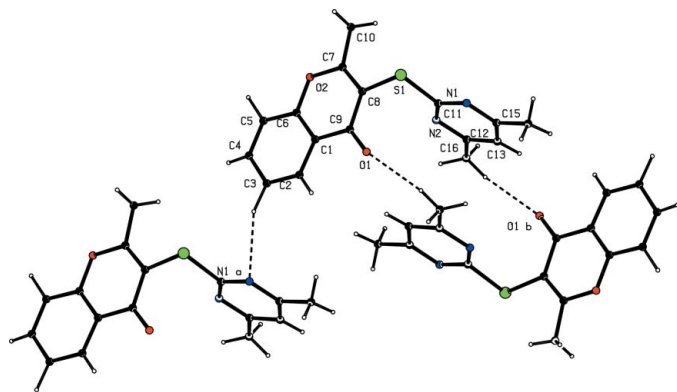


Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (a) $x, y - 1, z$; (b) $-x + 1, -y + 1, -z + 2$.

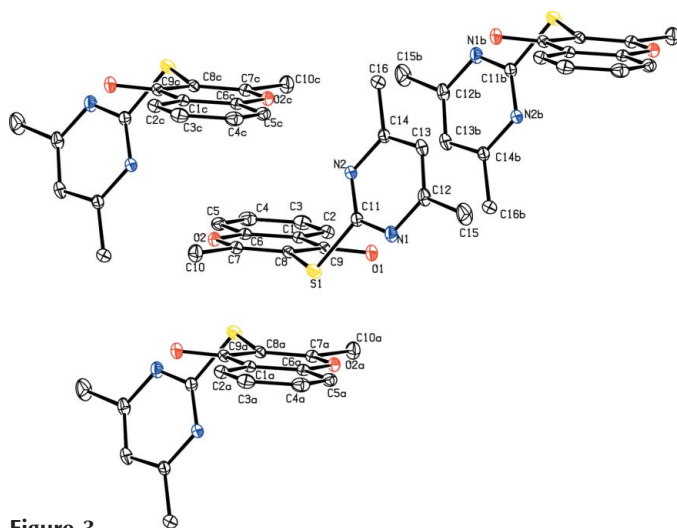


Figure 3
Intermolecular π - π stacking between benzene rings. H atoms have been omitted. [Symmetry codes: (a) $-x, -y, 1 - z$; (b) $-x + 1, -y + 1, -z + 2$; (c) $1 - x, y, 1 - z$.]

act as donors, *via* H atoms H16B, to O1 atoms, to form a dimer. As a result, an 18-membered ring is formed (Fig. 2), the topological motif of which corresponds to the first level graph-set descriptor $R_2^2(18)$ (Bernstein *et al.*, 1995). An additional weaker hydrogen bond is formed between a benzene H atom and the N atom of the pyrimidine ring (Table 1).

The dihedral angles between the benzopyran rings of adjacent stacked molecules (Fig. 3) are $0.34(2)^\circ$. The distances between adjacent ring centroids are $3.454(2)$ and $3.743(2)$ Å. These short approaches suggest the existence of π - π interactions.

Experimental

To a solution of 3-chloro-2-methylchromen-4-one (1 mmol) and 4,6-dimethylpyrimidine-2-thiol (1 mmol) in dimethylformamide (10 ml) was added *tert*-butyl potassium (1.1 mmol). The mixture was stirred for 8 h at 298 K. The resultant mixture was then purified by column chromatography on silica gel, with acetone/hexane (3:7, *v/v*) as eluant, to afford compound (I) (yield 51%, m.p. 481 K). ^1H NMR

(CDCl_3 , 400 MHz): δ 8.21 (*d*, 1H), 7.69 (*t*, 1H), 7.49 (*d*, 1H), 7.42 (*t*, 1H), 6.76 (*s*, 1H), 2.52 (*s*, 3H), 2.40 (*s*, 6H). MS (EI 70 eV) *m/z* (%): 298 (51), 283 (88), 265 (17), 240 (18), 190 (5), 14 7(32), 121 (9), 107 (29), 91 (19), 77 (7), 69 (100). Crystals suitable for single-crystal X-ray diffraction were grown from acetone at 277 K.

Crystal data

$\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$
 $M_r = 298.35$
Triclinic, $P\bar{1}$
 $a = 7.0515(9)$ Å
 $b = 10.2953(12)$ Å
 $c = 11.0039(13)$ Å
 $\alpha = 113.247(2)^\circ$
 $\beta = 91.839(2)^\circ$
 $\gamma = 92.225(2)^\circ$
 $V = 732.46(15)$ Å³

$Z = 2$
 $D_x = 1.353$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 1202 reflections
 $\theta = 2.3$ – 24.0°
 $\mu = 0.23$ mm⁻¹
 $T = 292(2)$ K
Plate, colorless
 $0.28 \times 0.24 \times 0.06$ mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
 φ and ω scans
Absorption correction: none
3684 measured reflections
2529 independent reflections

1909 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -7 \rightarrow 8$
 $k = -12 \rightarrow 9$
 $l = -12 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.192$
 $S = 1.08$
2529 reflections
193 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1186P)^2 + 0.0235P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C16}-\text{H16B}\cdots\text{O1}^{\text{i}}$	0.96	2.48	3.379 (4)	157
$\text{C3}-\text{H3}\cdots\text{N1}^{\text{ii}}$	0.93	2.67	3.570 (5)	162

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

H atoms were positioned geometrically and refined with riding constraints, with $\text{C}-\text{H} = 0.93$ or 0.96 Å. $U_{\text{iso}}(\text{H})$ values were set equal to $xU_{\text{eq}}(\text{carrier atom})$, where $x = 1.2$ for Csp^2 and $x = 1.5$ for methyl C atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; 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*.

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