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Methyl *N*-[4-(4,6-dimethylpyrimidin-2-ylamino)-thiocarbonyl]carbamate

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Key indicators

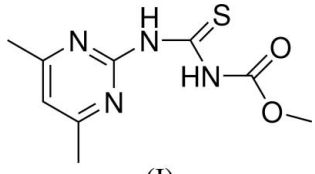
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.116
Data-to-parameter ratio = 9.5

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

The title compound, $C_9H_{11}N_4O_2S$, consisting of a pyrimidine ring and a thiourea carboxylic acid methyl ester, possesses mirror symmetry and an intramolecular N—H···N hydrogen bond. In the crystal structure, N—H···S hydrogen bonds link symmetry-related molecules to form dimers, which in turn are linked by a C—H···O hydogen bond to form a two-dimensional planar structure.

Comment

Thioureas are highly active bactericides and have been intensively studied for many years (Wang & Bo, 1998). They are used frequently as they can efficiently prevent many crop diseases with little harm to the crops and low toxicity to mammals (Tang *et al.*, 1988). Many groups have made important contributions in this field (Madan & Taneja, 1991; Bowser, 2005; Goodyear, 2003; Harris *et al.*, 2002; Sun *et al.*, 2003). The structure of the title compound, (I), was determined in order to extend the study of its structure–activity relationships.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. The

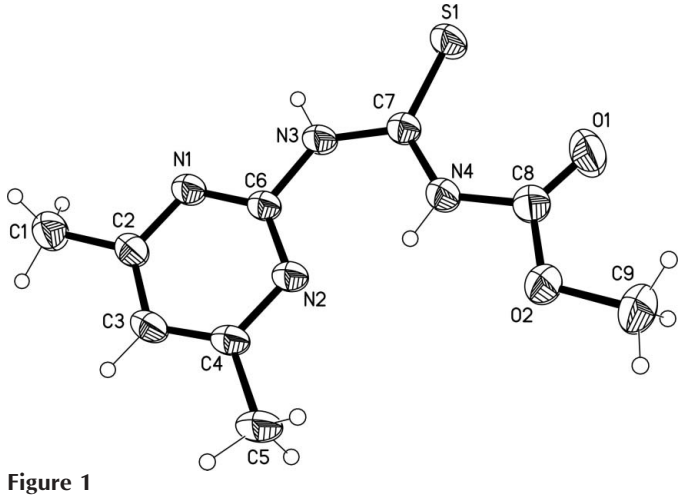


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and with displacement parameters at the 50% probability level.

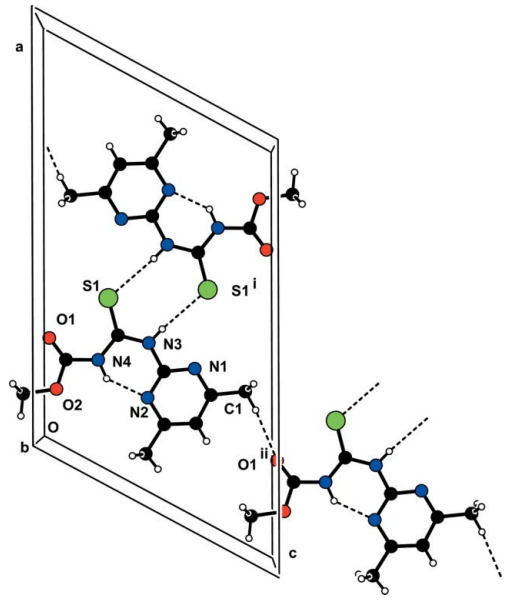


Figure 2
The crystal packing of compound (I), showing the formation of the hydrogen-bonded dimer (dashed lines). Symmetry codes are as given in Table 2.

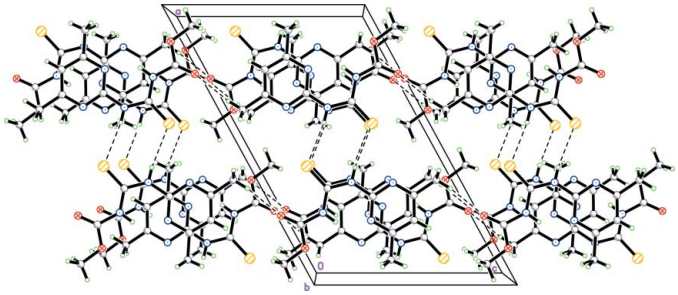


Figure 3
The crystal packing of compound (I), showing the formation of the hydrogen-bonded (dashed lines) two-dimensional layer-like structure.

molecule possesses crystallographic mirror symmetry and there is an intramolecular hydrogen bond between pyrimidine atom N2 and the thiourea NH group, N4. Details of the hydrogen bonding are given in Table 2. The bond lengths and angles of the pyrimidine moiety are similar to those observed previously for an unsubstituted pyrimidine ring (Ishida & Kashino, 1999). The bond lengths and angles in the thiourea group are also similar to those in analogous compounds (Lin *et al.*, 2004; Zhang *et al.*, 2003).

There is an intermolecular N—H···S hydrogen bond linking symmetry-related molecules to form a dimer, as shown in Fig. 2. These dimers are in turn linked by an intermolecular C—H···O hydrogen bond to form an interlaced two-dimensional planar structure, as shown in Fig. 3.

Experimental

Compound (I) was prepared according to the following method. Methyl chloroformate (0.567 g, 006 mol) was added dropwise to a solution of potassium thiocyanate (0.582 g, 0.006 mol) in ethyl acetate (10 ml). The reaction mixture was refluxed for 2 h. KCl was filtered off and 2-amino-4,6-dimethylpyrimidine (0.615 g, 0.005 mol) was added to the filtrate. The reaction mixture was heated under reflux for a further 4 h. The reaction mixture was then allowed to cool slowly to room temperature, followed by suction filtration. In order to remove the remainder of the product, it was necessary to rinse the reaction mixture thoroughly with distilled water. A vacuum drier was then used to dry the product. Finally, a light-yellow product was obtained (yield 82%). Single crystals of compound (I) suitable for X-ray analysis were obtained by slow evaporation of a solution in dimethylformamide over 5 d at room temperature. Elemental analysis for $C_9H_{11}N_4O_2S$, found: C 45.03, H 5.01, N 23.35%; calculated: C 45.00, H 5.00, N 23.30%.

Crystal data

$C_9H_{12}N_4O_2S$
$M_r = 240.29$
Monoclinic, $C2/m$
$a = 17.537$ (5) Å
$b = 6.759$ (2) Å
$c = 11.148$ (3) Å
$\beta = 118.557$ (4)°
$V = 1160.5$ (6) Å^3
$Z = 4$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1090 reflections
$\theta = 2.6$–24.0°
$\mu = 0.27$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.27 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
$T_{min} = 0.962$, $T_{max} = 0.979$
2964 measured reflections
1117 independent reflections
873 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.1°$
$h = -20 \rightarrow 20$
$k = -8 \rightarrow 8$
$l = -11 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.116$
$S = 1.06$
1117 reflections
118 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0829P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.656 (3)	N4—C8	1.383 (3)
N1—C6	1.333 (3)	O1—C8	1.176 (3)
N1—C2	1.336 (3)	O2—C8	1.329 (4)
N2—C6	1.327 (3)	O2—C9	1.443 (4)
N2—C4	1.357 (3)	C1—C2	1.490 (4)
N3—C7	1.370 (3)	C2—C3	1.378 (4)
N3—C6	1.392 (3)	C3—C4	1.375 (4)
N4—C7	1.359 (3)	C4—C5	1.492 (4)
C6—N1—C2	116.0 (2)	C3—C4—C5	124.0 (3)
C6—N2—C4	116.4 (2)	N2—C6—N1	127.2 (2)
C7—N3—C6	132.0 (2)	N2—C6—N3	120.3 (2)
C7—N4—C8	128.4 (2)	N1—C6—N3	112.4 (2)
C8—O2—C9	115.8 (3)	N4—C7—N3	114.6 (2)
N1—C2—C3	121.0 (2)	N4—C7—S1	126.81 (19)
N1—C2—C1	115.7 (3)	N3—C7—S1	118.6 (2)
C3—C2—C1	123.2 (3)	O1—C8—O2	124.9 (3)
C4—C3—C2	119.4 (2)	O1—C8—N4	127.7 (3)
N2—C4—C3	119.8 (2)	O2—C8—N4	107.4 (2)
N2—C4—C5	116.2 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···S1^{i}	0.84 (4)	2.59 (4)	3.426 (2)	174 (3)
N4—H4···N2	0.88 (4)	1.95 (4)	2.683 (3)	140 (3)
C1—H1B···O1^{ii}	0.91 (3)	2.37 (4)	3.284 (5)	174 (3)

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

The H atoms were located in difference Fourier maps and their atomic coordinates were refined, with U_{iso}(H) fixed at 0.08 Å^2 [N—H distances in the range 0.84 (4)–0.88 (4) Å and C—H distances in the range 0.84 (2)–0.96 (3) Å].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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