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Key indicators

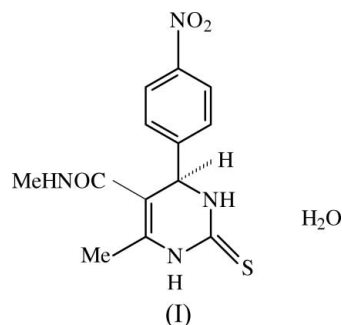
Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.043
 wR factor = 0.129
Data-to-parameter ratio = 13.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1,4-Dihydro-6-methyl-5-(*N*-methylcarbamoyl)-4-(4-nitrophenyl)pyrimidine-2(3*H*)-thione monohydrate

The title compound, $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}_3\text{S}\cdot\text{H}_2\text{O}$, is an isomer of 1,4-dihydro-6-methyl-5-(*N*-methylcarbamoyl)-4-(2-nitrophenyl)pyrimidine-2(3*H*)-thione. The nitro group is coplanar with the benzene ring to which it is attached. Intermolecular hydrogen bonds stabilize the crystal structure.

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Comment

1,4-Dihydropyrimidine (DHPM) shows a very similar pharmacological profile to classical dihydropyridine calcium channel modulators (Lu *et al.*, 2002). The structure determination of the title compound, (I), is part of an ongoing study of this series of compounds (Ravikumar & Sridhar, 2005; Sridhar & Ravikumar, 2005). The title compound is an isomer of 1,4-dihydro-6-methyl-5-(*N*-methylcarbamoyl)-4-(2'-nitrophenyl)-2-(3*H*)-pyrimidinethione, (II) (Chandra Mohan *et al.*, 2003). The two compounds crystallize in the same space group; however, (I) is a hydrate, but (II) is a hemihydrate.



The bond lengths and angles (Table 1) are in good agreement with values found in related structures. The dihydropyrimidine ring adopts a flattened boat conformation [asymmetry parameter (Nardelli, 1983) $\Delta C_s(\text{N}1) = 0.053$ (1)]. The stern atom N1 is displaced by 0.150 (2) Å and the bow atom C4 is displaced by 0.318 (2) Å from the mean plane defined by atoms C2, C3, N5 and C6. In (II), the ring conformation is reported to be a half-chair.

The benzene ring is almost perpendicular to the dihydropyrimidine ring, with a dihedral angle of 79.15 (4)°. The corresponding angles for (II) are 87.4 (1) and 78.9 (1)°, respectively, for the two molecules in the asymmetric unit. The carbamoyl side chain is in an extended conformation [C3—C31—N33—C34 = −176.77 (16)°]. The twist of the benzene ring defined by the torsion angle N5—C4—C7—C8 [161.89 (16)°] is different in (II) [−102.2 (5) and 89.9 (5)°], which might be attributed to the different position of the nitro group (Fig. 2). The nitro group is coplanar with the benzene ring to which it is attached [dihedral angle = 4.4 (2)°], whereas

in (II) it is significantly inclined to the benzene ring [dihedral angles: -35.3 (7) and -20.1 (9) $^\circ$]. N—H \cdots O and N—H \cdots S hydrogen bonds (Table 2) stabilize the crystal structure. The nitro group is not involved in any hydrogen bonding; however, in (II) it forms an N—H \cdots O hydrogen bond.

Experimental

Compound (I) was prepared by the method of Sadanandam *et al.* (1992) and was recrystallized from a methanol/water (9:1) solution.

Crystal data

$C_{13}H_{14}N_4O_3S \cdot H_2O$	$Z = 2$
$M_r = 324.36$	$D_x = 1.422 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 4.8373$ (5) \AA	Cell parameters from 5088 reflections
$b = 9.8906$ (11) \AA	$\theta = 2.3\text{--}28.0^\circ$
$c = 16.0930$ (17) \AA	$\mu = 0.24 \text{ mm}^{-1}$
$\alpha = 81.414$ (2) $^\circ$	$T = 273$ (2) K
$\beta = 84.968$ (2) $^\circ$	Block, colorless
$\gamma = 86.248$ (2) $^\circ$	$0.23 \times 0.12 \times 0.09 \text{ mm}$
$V = 757.32$ (14) \AA^3	

Data collection

Bruker SMART CCD area detector diffractometer	2437 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.019$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
7319 measured reflections	$h = -5 \rightarrow 5$
2669 independent reflections	$k = -11 \rightarrow 11$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0786P)^2 + 0.2758P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.129$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
2669 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
203 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected bond lengths (\AA).

O1—N10	1.207 (3)	N10—C10	1.477 (3)
O2—N10	1.206 (3)	C2—C3	1.336 (2)
O32—C31	1.236 (2)		

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N1—H1 \cdots S1 ⁱ	0.86	2.65	3.4586 (16)	157
N5—H5 \cdots O1W	0.86	2.22	3.022 (3)	155
N33—H33 \cdots O32 ⁱⁱ	0.86	1.98	2.8144 (19)	163
O1W—H1W \cdots O1W ⁱⁱⁱ	0.79	2.44	2.860 (5)	114
O1W—H2W \cdots O1W ^{iv}	0.80	2.43	2.901 (4)	119

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

The H atoms of the water molecule were located in a difference density map and refined using a riding model; their isotropic displacement parameters were refined. All other H atoms were

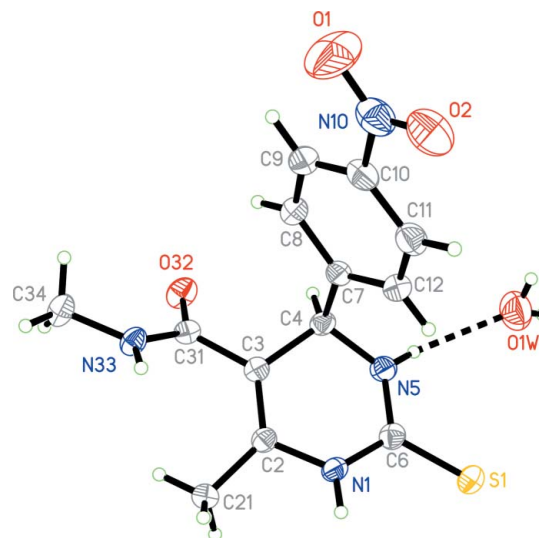


Figure 1

A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The dashed line indicates a hydrogen bond.

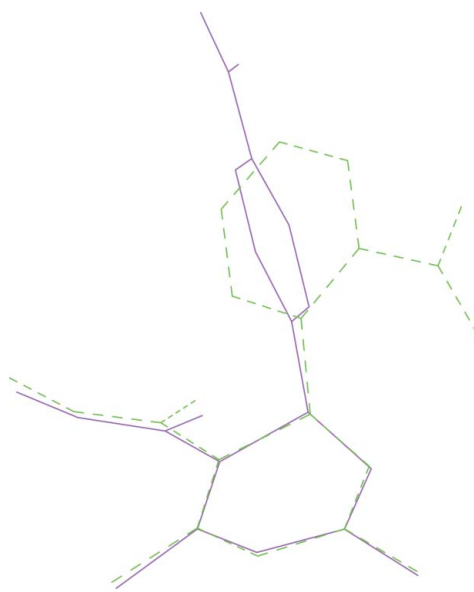


Figure 2

A least-squares fit of the pyrimidine rings of (I) (dashed lines) and one of the two molecules of (II) (full lines) (r.m.s deviation = 0.056 \AA).

included in calculated positions ($C\text{---}H = 0.93\text{--}0.98 \text{ \AA}$ and $N\text{---}H = 0.86 \text{ \AA}$) and refined using a riding model, with $U_{\text{iso}}(H)$ values set at 1.2 (N,C) or 1.5 (CH_3) times the U_{eq} values of the parent atoms. The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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