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#### Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.054  
 $wR$  factor = 0.143  
 Data-to-parameter ratio = 17.6

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

## Hydrogen-bonded sheets in 4-amino-8,8-dimethyl-2-(methylsulfanyl)-8,9-dihydro-pyrimidino[4,5-*b*]quinolin-6(7*H*)-one

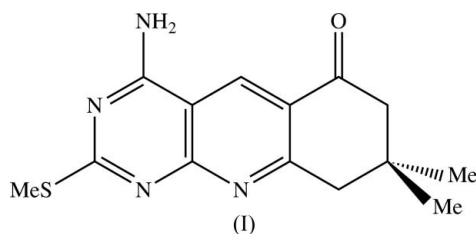
The title molecule,  $\text{C}_{14}\text{H}_{16}\text{N}_4\text{OS}$ , shows strong bond fixation within the fused heterocyclic rings. In the crystal structure, molecules are linked into sheets by a combination of  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

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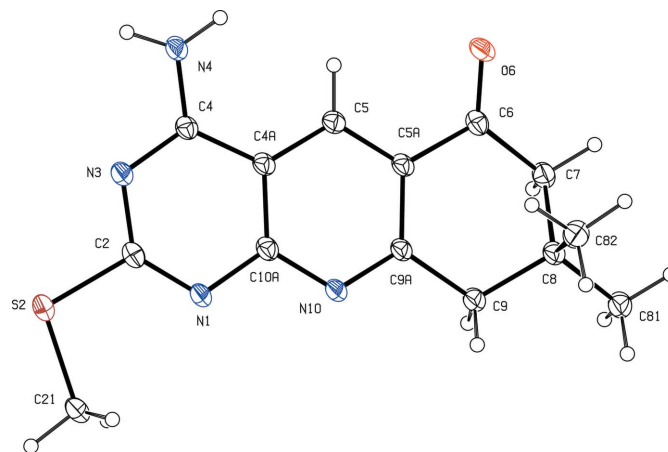
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#### Comment

We report here the structure of the title compound, (I) (Fig. 1), which was prepared by microwave irradiation of a two-component mixture of a 6-aminopyrimidine and the condensation product formed from dimedone and formaldehyde. By contrast a similar reaction between 6-aminopyrimidines, 5,5-dimethylcyclohexane-1,3-dione and a large excess of formaldehyde yielded spiranopyridopyrimidines (Quiroga *et al.*, 2006).

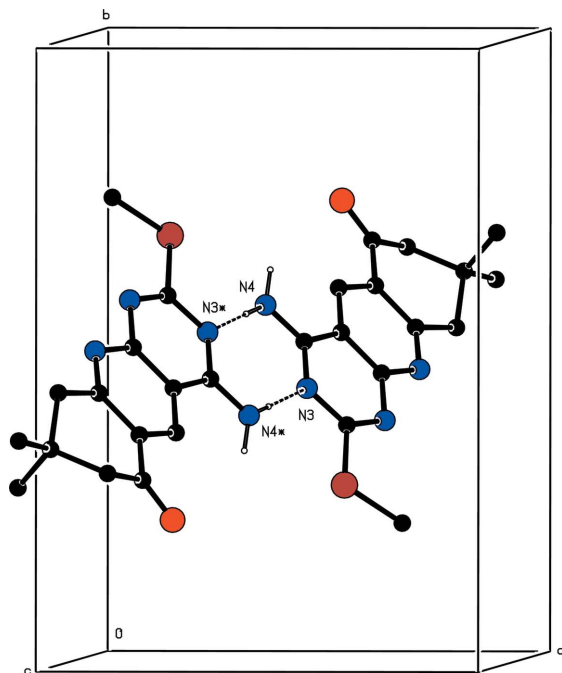


The bond distances within the fused heterocyclic system (Table 1) provide evidence for significant bond fixation of the naphthalene type. Thus, for example, the bonds  $\text{N1}-\text{C2}$ ,  $\text{N3}-\text{C4}$  and  $\text{C9A}-\text{N10}$  are all significantly shorter than the bonds  $\text{C2}-\text{N3}$ ,  $\text{N10}-\text{C10A}$  and  $\text{C10A}-\text{N1}$ , while  $\text{C5}-\text{C5A}$  is the shortest of the  $\text{C}-\text{C}$  bonds. The carbocyclic ring adopts a

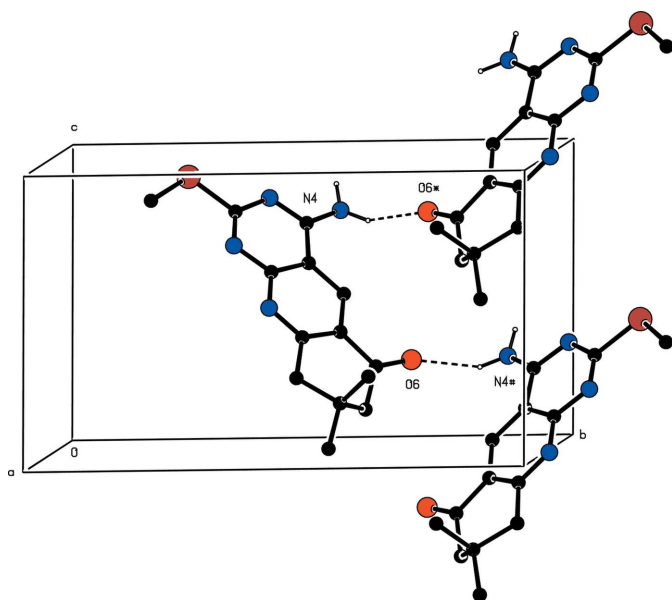


**Figure 1**

The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
Part of the crystal structure of compound (I), showing the formation of the  $R_2^2(8)$  substructure. Hydrogen bonds are shown as dashed lines and for the sake of clarity the H atoms bonded to C atoms have been omitted. The atoms marked with an asterisk (\*) are at the symmetry position  $(1 - x, 1 - y, 2 - z)$ .



**Figure 3**  
Part of the crystal structure of compound (I), showing the formation of the  $C(8)$  substructure. The hydrogen bonds are shown as dashed lines and for the sake of clarity the H atoms bonded to C atoms have been omitted. The atoms marked with an asterisk (\*) or a hash (#) are at the symmetry positions  $(x, \frac{3}{2} - y, \frac{1}{2} + z)$  and  $(x, \frac{3}{2} - y, -\frac{1}{2} + z)$ , respectively.

conformation best described as intermediate between an envelope form, with the fold across the vector  $C7 \cdots C9$ , and a half-chair form. The ring-puckering parameters (Cremer & Pople, 1975) for the atom sequence  $C5A - C6 - C7 - C8 -$

$C9 - C9A$  are  $\theta = 129.2(2)^\circ$  and  $\varphi = 342.4(4)^\circ$ ; the idealized values for the envelope and half-chair forms, respectively, are  $\theta = 126.3$  and  $129.8^\circ$ , and  $\varphi = (60k)$  and  $(60k + 30)^\circ$ , where  $k$  is zero or an integer. The two  $S - C$  distances are clearly different, and atom  $C21$  lies almost in the plane of the adjacent pyrimidine ring.

The molecules are linked into sheets by two hydrogen bonds (Table 2), and the formation of the sheets is readily analysed in terms of two simple substructures, each formed by just one hydrogen bond. In the first substructure, amino atom  $N4$  in the molecule at  $(x, y, z)$  acts as a hydrogen-bond donor, via  $H4A$ , to the pyrimidine ring atom  $N3$  in the molecule at  $(1 - x, 1 - y, 2 - z)$ , so generating by inversion an  $R_2^2(8)$  (Bernstein *et al.*, 1995) ring centred at  $(\frac{1}{2}, \frac{1}{2}, 1)$  (Fig. 2). In the second substructure, amino atom  $N4$  at  $(x, y, z)$  acts as a hydrogen-bond donor, via  $H4B$ , to atom  $O6$  in the molecule at  $(x, \frac{3}{2} - y, \frac{1}{2} + z)$ , so forming a simple  $C(8)$  chain running parallel to the  $[001]$  direction and generated by the  $c$ -glide plane at  $y = 0.75$  (Fig. 3). The combination of these two substructures generates a sheet parallel to  $(100)$  (Fig. 4), but there are no direction-specific interactions between adjacent sheets; in particular  $C - H \cdots \pi$  hydrogen bonds and  $\pi - \pi$  stacking interactions are absent.

## Experimental

A mixture of 4,6-diamino-2-methylsulfanylpyrimidine (1.0 mmol), 2,2-methylenebis(3-hydroxy-5,5-dimethylcyclohex-2-en-1-one) (1.0 mmol) and triethylamine (0.5 mmol) was placed in an open Pyrex-glass vessel and irradiated in a domestic microwave oven for 80 s at 600 W. The resulting solid product was collected by filtration, washed with cold ethanol, dried and then recrystallized from ethanol to provide crystals of compound (I) suitable for single-crystal X-ray diffraction; yield 60%, m.p. 580 K.

### Crystal data

$C_{14}H_{16}N_4OS$   
 $M_r = 288.37$   
 Monoclinic,  $P2_1/c$   
 $a = 10.7138(11) \text{ \AA}$   
 $b = 15.1368(15) \text{ \AA}$   
 $c = 8.9112(6) \text{ \AA}$   
 $\beta = 101.208(6)^\circ$   
 $V = 1417.6(2) \text{ \AA}^3$

$Z = 4$   
 $D_x = 1.351 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.23 \text{ mm}^{-1}$   
 $T = 120(2) \text{ K}$   
 Block, colourless  
 $0.40 \times 0.24 \times 0.18 \text{ mm}$

### Data collection

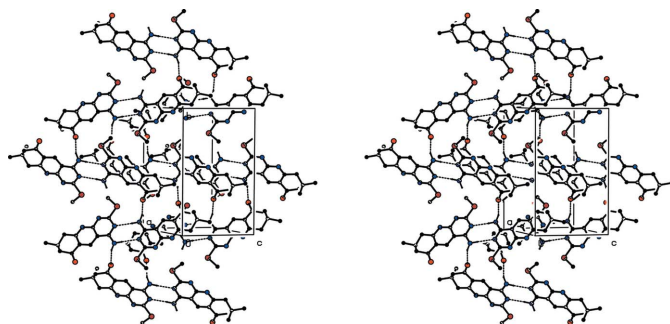
Bruker-Nonius KappaCCD  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.926, T_{\max} = 0.960$

20540 measured reflections  
 3245 independent reflections  
 2115 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.072$   
 $\theta_{\max} = 27.5^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.143$   
 $S = 1.02$   
 3245 reflections  
 184 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.6375P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.35 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$



**Figure 4**

A stereoscopic view of part of the crystal structure of compound (I), showing the formation of a sheet parallel to (100). The hydrogen bonds are shown as dashed lines and for the sake of clarity the H atoms bonded to C atoms have been omitted.

**Table 1**

Selected geometric parameters (Å, °).

N1—C2	1.314 (3)	C9A—N10	1.328 (3)
C2—N3	1.364 (3)	N10—C10A	1.356 (3)
N3—C4	1.338 (3)	C10A—N1	1.371 (3)
C4—C4A	1.445 (3)	C4A—C10A	1.408 (3)
C4A—C5	1.401 (3)	C2—S2	1.753 (2)
C5—C5A	1.384 (3)	S2—C21	1.794 (3)
C5A—C9A	1.413 (3)	C4—N4	1.332 (3)
C2—S2—C21	101.78 (12)		
N1—C2—S2—C21	2.1 (2)	N3—C2—S2—C21	−177.21 (17)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4A $\cdots$ N3 <sup>i</sup>	0.90	2.17	3.067 (3)	173
N4—H4B $\cdots$ O6 <sup>ii</sup>	0.90	2.15	2.946 (3)	147

Symmetry codes: (i)  $-x + 1, -y + 1, -z + 2$ ; (ii)  $x, -y + \frac{3}{2}, z + \frac{1}{2}$ .

All H atoms were located in difference maps and then treated as riding atoms with distances C—H = 0.95 Å (aromatic), 0.98 Å (CH<sub>3</sub>) or 0.99 Å (CH<sub>2</sub>) and N—H = 0.90 Å, and with  $U_{iso}(H) = kU_{eq}(C,N)$ , where  $k = 1.5$  for methyl H atoms and 1.2 for all other H atoms.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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