Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A new triclinic polymorph of 4,5,6-trimethyl-1-phenyl-3,4-dihydropyrimidine-2(1*H*)-thione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.120
Data-to-parameter ratio = 18.1

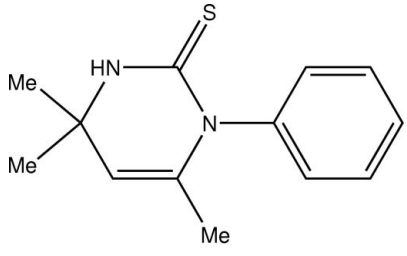
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 March 2007
Accepted 4 April 2007

The title crystal structure, $C_{13}H_{16}N_2S$, is a triclinic polymorph of the previously reported orthorhombic structure [Yamin, Kasim & Hamzah (2005). *Acta Cryst.* E**61**, o55–o57]. In both structures, the phenyl group is almost perpendicular to the pyrimidine-2-thione ring. In the triclinic structure, inversion-related molecules are linked to form a dimer by N—H···S hydrogen bonds, whereas in the orthorhombic polymorph two independent molecules are linked by N—H···S hydrogen bonds to form a dimer.

Comment

The participation of the acetone solvent in the reaction of cinnamoyl isothiocyanate with aniline led to the formation of 4,5,6-trimethyl-1-phenyl-3,4-dihydropyrimidine-2(1*H*)-thione, which crystallized in the orthorhombic space group *Pbca* with two independent molecules in the asymmetric unit (Yamin *et al.*, 2005). Under similar reaction conditions, a similar product, (I), was obtained when 2-chloropropionylchloride was reacted with ammonium thiocyanate and aniline. However, the compound crystallizes in the triclinic space group $P\overline{1}$ with only one molecule in the asymmetric unit.



(I)

The bond lengths and angles in the triclinic polymorph of (I) are comparable with those in the orthorhombic polymorph,

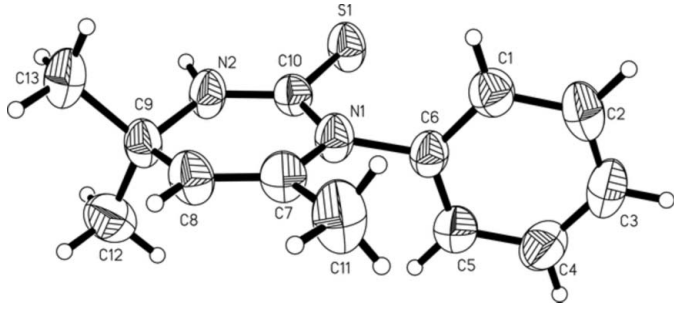


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

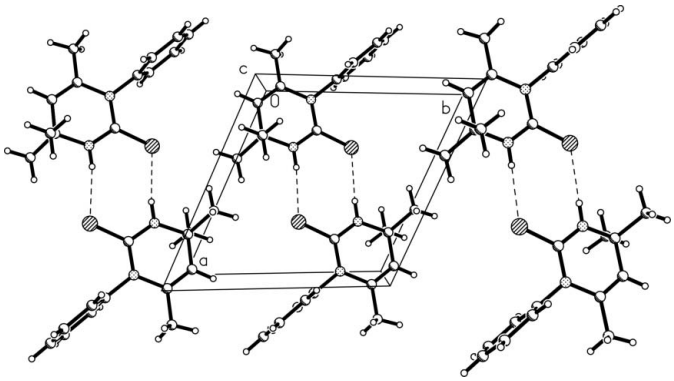


Figure 2
A packing diagram for (I), viewed down the *c* axis. The dashed lines denote N—H···S hydrogen bonds.

and are in normal ranges (Allen *et al.*, 1987). The pyrimidine-2-thione ring (N1/N2/C7–C10/S1) is essentially planar, with a maximum deviation of 0.093 (2) Å for atom C9. The dihedral angle between the phenyl and pyrimidine-2-thione rings is 86.13 (8)°, comparable with the values of 89.79 (12) and 84.42 (15)° in the orthorhombic polymorph.

In the present structure, inversion-related molecules are linked to form a dimer by N—H···S hydrogen bonds (Table 1). In the orthorhombic polymorph, the two independent molecules are linked by N—H···S hydrogen bonds to form a dimer.

Experimental

A solution of aniline (1.86 g, 0.02 mol) in acetone (20 ml) was added with stirring to a mixture of 2-chloropropionylchloride (2.54 g, 0.02 mol) and ammonium thiocyanate (1.52 g, 0.02 mol) in acetone (30 ml). The mixture was refluxed for 1 h. The resulting solution was filtered and left to stand at room temperature for 5 d. Colourless block-shaped crytals formed on slow evaporation of the solvent (yield 1.323 g, 29%; m.p: 450.2–460.9 K).

Crystal data

$C_{13}H_{16}N_2S$
M_r = 232.34
Triclinic, $P\overline{1}$
a = 8.486 (2) Å
b = 8.649 (2) Å
c = 10.336 (3) Å
α = 108.598 (4)°
β = 103.053 (4)°
γ = 107.937 (4)°
V = 638.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
0.48 × 0.44 × 0.40 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.898, T_{max} = 0.914
6568 measured reflections
2620 independent reflections
2186 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.120
S = 1.20
2620 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···S1^{i}	0.86	2.71	3.465 (2)	148

Symmetry code: (i) $-x+1, -y+1, -z+2$.

H atoms were positioned geometrically, with C—H = 0.93 or 0.96 Å and N—H = 0.86 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) for the methyl group or 1.2U_{eq}(parent) for CH and NH groups.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government and Universiti Kebangsaan Malaysia for research facilities and grant No. UKM-ST-01-FRGS003-2006.

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