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Key indicators

Single-crystal X-ray study
T = 273 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.049
wR factor = 0.143
Data-to-parameter ratio = 18.5

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

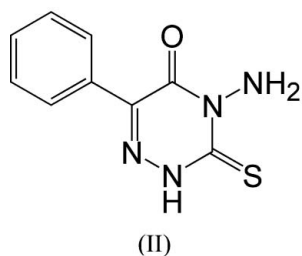
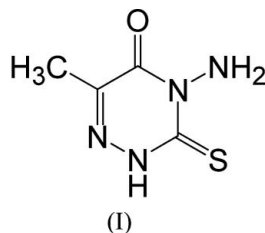
4-Amino-6-phenyl-3-thioxo-2,3-dihydro-1,2,4-triazin-5(4H)-one

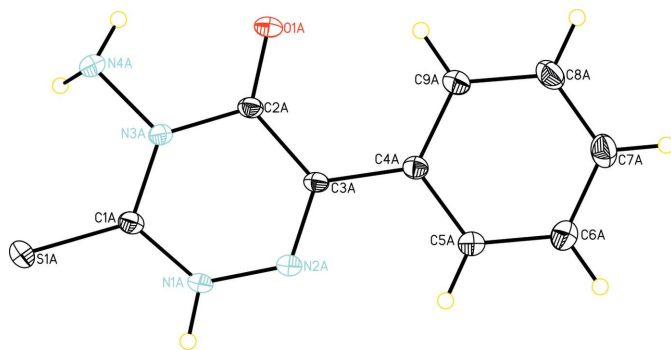
The title compound $\text{C}_9\text{H}_8\text{N}_4\text{OS}$, crystallizes with two molecules in the asymmetric unit. The crystal structure is stabilized by intermolecular hydrogen bonds between the protonated N atom of the triazine ring and the carbonyl O atom of an adjacent triazine ring, forming chains running parallel to the *a* axis.

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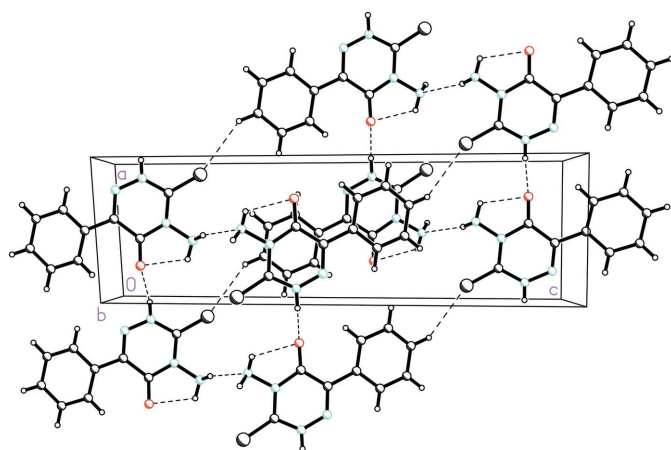
Comment

1,2,4-Triazines and many compounds derived from them are found to possess a wide variety of pharmacological and herbicidal activities (Holla *et al.*, 1999; Gruzdyev *et al.*, 1983; Abdel-Rahman, 2001; Sathiakumar & Delzell, 1997). In addition to its biological activity, there has been considerable interest in the chemistry of 4-amino-6-methyl-3-thioxo-3,4-dihydro-2H-1,2,4-triazin-5-one, (I), known in biochemical research as 6-azathiopyrimine or 6-ATT. Not only has its structure been determined (Voutsas, *et al.*, 1978; Ghassemzadeh, *et al.*, 1998), but the structures of its complexes with copper (Razak, *et al.*, 1999; Ghassemzadeh, *et al.*, 2002; Ghassemzadeh, *et al.*, 2004; Yazdanbakhsh, *et al.*, 2004), silver (Adhami, *et al.*, 1999; Ghassemzadeh, *et al.*, 2004), palladium (Neumuller, *et al.*, 1999; Ghassemzadeh, *et al.*, 1998; Ghassemzadeh, *et al.*, 2000; Ghassemzadeh, *et al.*, 2005), platinum (Ghassemzadeh, *et al.*, 2005), and lithium (Ghassemzadeh, *et al.*, 2003) have been reported. The structure of a related derivative, 4-amino-6-*tert*-butyl-3-thioxo-3,4-dihydro-2H-1,2,4-triazin-5-one has also been recently reported (Sridhar *et al.*, 2006).



**Figure 1**

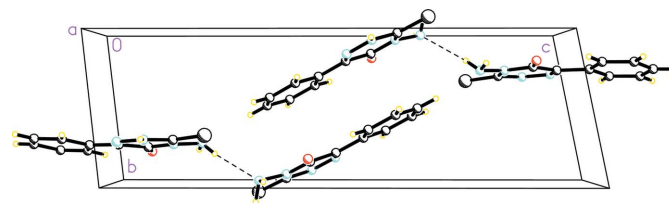
View of one of the two essentially identical molecules of (II) in the asymmetric unit, showing its atom-numbering scheme; the molecule not shown is numbered identically, except its labels bear the suffix B. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

The molecular packing for (II), viewed down the *b* axis, showing the chains formed along the *a* axis. The vertical chain in the center of this view contains only A-type molecules, while the chains at each end of the cell contain B-type molecules. Dashed lines indicated hydrogen bonds.

triazin-5-one (II), is reported here. It crystallizes in the triclinic space group $P\bar{1}$ with two molecules in the asymmetric unit. In all essential details, the molecular geometries of both molecules (Fig. 1 and Table 1) are identical and are in good agreement with related structures containing the 5-oxo-3-thioxo-1,2,4-triazine system (Ghassemzadeh *et al.*, 1998; Sridhar *et al.*, 2006). The triazine ring is planar and exists in the thione form; the C=S bond lengths are slightly longer than that expected for a pure double bond (1.61 Å; Pauling, 1960). The N—N bond distances are intermediate between those expected for single (1.45 Å) and double (1.20 Å) bonds. The C—N bonds show marked differences in their lengths, those between N2 and C3 being the shortest and those between N3 and C2 being the longest. These values are intermediate between those expected for single and double C—N bonds (1.47 and 1.27 Å, respectively).

The crystal structure is stabilized by intermolecular N—H...O hydrogen bonds (Table 2). The two molecules of the asymmetric unit display similar, but not identical, packing interactions. In each case, the protonated N atom (N1A or N1B) of the triazine ring links to the carbonyl O atom of an

**Figure 3**

The molecular packing for (II), viewed down the *a* axis, showing the hydrogen bonds (dashed lines) linking chains of A-type molecules and B-type molecules.

adjacent molecule of the same type, forming a chain running parallel to the *a* axis (Figs. 2 and 3).

Experimental

Benzoylformic acid (15g, 0.1mol) was mixed with thiocarbonylhydrazide (10.6g, 0.1 mol) in 500 ml water and the mixture was heated to boiling (Dornow *et al.*, 1964). A yellow precipitate separated and was filtered. The product was recrystallized from an acetone–toluene (1:1) mixture (yield: 91.8%; m.p. 506 K). Elemental analysis (calculated) (%): C 49.22 (49.09); H 3.53 (3.64); N 25.65 (25.45).

Crystal data

$C_9H_8N_4OS$	$V = 945.0 (3) \text{ \AA}^3$
$M_r = 220.25$	$Z = 4$
Triclinic, $P\bar{1}$	$D_x = 1.548 \text{ Mg m}^{-3}$
$a = 6.5965 (11) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.0573 (12) \text{ \AA}$	$\mu = 0.32 \text{ mm}^{-1}$
$c = 21.238 (4) \text{ \AA}$	$T = 273 (2) \text{ K}$
$\alpha = 81.061 (3)^\circ$	Plate, colourless
$\beta = 89.307 (4)^\circ$	$0.55 \times 0.27 \times 0.10 \text{ mm}$
$\gamma = 75.444 (3)^\circ$	

Data collection

Bruker Apex II CCD area-detector diffractometer	10897 measured reflections
φ and ω scans	5336 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3757 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.845$, $T_{\max} = 0.970$	$R_{\text{int}} = 0.026$
	$\theta_{\max} = 30.8^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.1411P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.143$	$(\Delta/\sigma)_{\max} = 0.002$
$S = 1.05$	$\Delta\rho_{\max} = 0.33 \text{ e \AA}^{-3}$
5336 reflections	$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$
289 parameters	
H-atom parameters constrained	

Table 1

Selected bond lengths (Å).

S1A—C1A	1.6541 (17)	N3A—C1A	1.371 (2)
S1B—C1B	1.6520 (18)	N3A—C2A	1.385 (2)
O1A—C2A	1.223 (2)	N1B—N2B	1.341 (2)
O1B—C2B	1.220 (2)	N1B—C1B	1.352 (2)
N1A—N2A	1.342 (2)	N2B—C3B	1.298 (2)
N1A—C1A	1.353 (2)	N3B—C1B	1.367 (2)
N2A—C3A	1.298 (2)	N3B—C2B	1.391 (2)

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1A—H1AA···O1A ⁱ	0.88 (2)	2.00 (2)	2.810 (2)	153 (2)
N4A—H4A2···O1A	0.81 (3)	2.22 (2)	2.643 (2)	112 (2)
N1B—H1BA···O1B ⁱ	0.82 (2)	2.02 (2)	2.806 (2)	158 (2)
N4B—H4B1···N4A ⁱⁱ	0.84 (3)	2.41 (3)	3.228 (3)	167 (2)
N4B—H4B2···O1B	0.86 (3)	2.20 (2)	2.633 (2)	111.3 (19)

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

All C-bound H atoms were initially located in a difference Fourier map, and were then placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Positional parameters for N-bound H atoms were refined and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2* (Bruker, 2006); data reduction: *APEX2* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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