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1,3-Dimethyl-5-(thiazol-2-yldiazenyl)pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.187
Data-to-parameter ratio = 19.2

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

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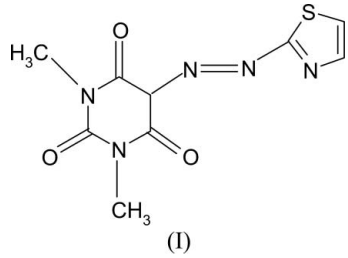
In the molecule of the title compound, $C_9H_9N_5O_3S$, the thiazole ring is oriented with respect to the barbituric acid ring at a dihedral angle of 10.53 (7)°. In the crystal structure, intermolecular N—H···N hydrogen bonds link the molecules into centrosymmetric dimers; these dimers are arranged in sheets parallel to the *ac* plane.

Comment

The thiazole ring has been identified as a central structural element of a number of biologically active natural products (Zabriskie *et al.*, 1988) and of pharmacologically active compounds (Metzger, 1984). The bioactivity of thiazoles is mainly due to their structural similarities with the imidazolyl units in proteins (Kornis, 1984) as well as their biological, structural, electronic and spectroscopic properties (Comba, 1993). Their existence may modify the bioactive and pharmaceutical characteristics of the adducts (Chohan *et al.*, 2002). Azo derivatives are used extensively in analytical chemistry and in the dyestuff industry as metallochromic and acid–base indicators (Rau, 1990). They are also used in the fields of nonlinear optics and optical data storage (Taniike *et al.*, 1996). Azo dyes have wide applicability as optical materials and so their structures have also attracted considerable attention (Biswas & Umapathy, 2000). Barbituric acid derivatives have been widely used in medical applications as sedative, hypnotic and local anaesthetic drugs (Chen *et al.*, 1990). They have also been studied as antitumour (Gulliya, 1999), anticancer (Gulliya, 1997) and anti-osteoporosis agents (Sakai & Satoh, 1999), and used as nonlinear optical materials (Ikeda *et al.*, 1989), disperse dyes with strong fluorescent properties (Tada, 1985) and agrochemical products (Lee & Carter, 1989), such as insecticides or fungicides. This study was undertaken in order to ascertain the crystal structure of the title compound, (I).



(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

An examination of the deviations from the least-squares planes through individual rings shows that the thiazole and

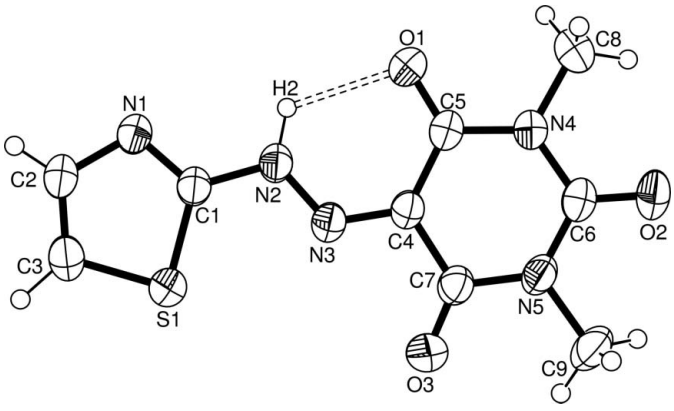


Figure 1
The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond is shown as dashed lines.

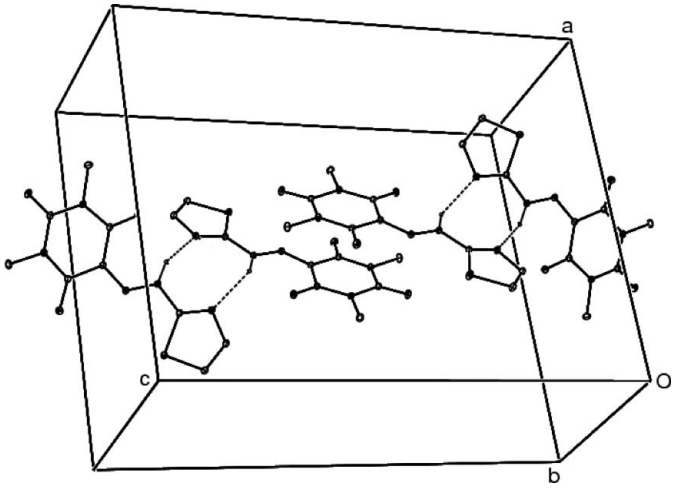


Figure 2
Packing diagram of (I). Intermolecular N—H···N hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

barbituric acid rings *A* (S1/N1/C1–C3) and *B* (N4/N5/C4–C7), respectively, are both planar. The dihedral angle between the two rings is 10.53 (7)°. The intramolecular N—H···O hydrogen bond (Table 1) results in the formation of a third planar ring *C* (H2/O1/N2/N3/C4/C5), with a maximum displacement from the mean plane of 0.1347 (5) Å for H2; this ring makes dihedral angles of 5.53 (8) and 5.07 (6)° with rings *A* and *B*, respectively.

As can be seen from the packing diagram (Fig. 2), intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into dimers, which are stacked along the *a* axis, forming a layer parallel to the *ac* plane. Dipole-dipole and van der Waals interactions are effective in the molecular packing.

Experimental

2-Aminothiazole (0.20 g, 2.0 mmol) was dissolved in a hot glacial acetic acid–propionic acid mixture (2:1, 8 ml). The solution was rapidly cooled in an ice-salt bath and then added dropwise with stirring to a cold solution of nitrosylsulfuric acid (3 ml) over a period of 30 min. The mixture was stirred for an additional 2 h at 273 K. The resulting diazonium salt was also cooled in an ice-salt bath and then added dropwise with stirring to 1,3-dimethylbarbituric acid (0.31 g, 2.0 mmol) in an aqueous solution (10 ml) of KHO (2 mmol). The solution was stirred at 273 K for 2 h and the pH of the reaction mixture was maintained at 5–6 by the simultaneous addition of a saturated sodium carbonate solution (30 ml). The mixture was stirred for a further 1 d. The resulting solid was filtered off, washed with cold water and crystallized from ethanol (yield 0.45 g, 84%; m.p 494–495 K).

Crystal data

$C_9H_9N_5O_3S$
M_r = 267.27
Monoclinic, $C2/c$
a = 15.3047 (5) Å
b = 7.4568 (2) Å
c = 19.5403 (5) Å
β = 92.152 (2)°
V = 2228.45 (11) Å^3
Z = 8
D_x = 1.593 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 294 (2) K
Block, orange
0.30 × 0.22 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.915, T_{max} = 0.954
31797 measured reflections
3405 independent reflections
2691 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 30.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.187
S = 1.17
3405 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.093P)^2 + 0.8065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.95 (3)	2.11 (3)	2.688 (2)	118 (2)
N2—H2···N1^{i}	0.95 (3)	2.23 (3)	3.057 (2)	145 (2)

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

Methyl H atoms were positioned geometrically, with C—H = 0.96 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C). The remaining H atoms were located in a difference synthesis and refined isotropically [N—H = 0.95 (3) Å, U_{iso}(H) = 0.063 (8) Å^2 and Csp^2—H = 0.90 (3) and 1.00 (4) Å, U_{iso}(H) = 0.062 (8)–0.095 (12) Å^2].

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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