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6-Amino-1,3-dimethyl-5-(4-methylthiazol-2-yldiazenyl)uracil

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.073
wR factor = 0.219
Data-to-parameter ratio = 20.8

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

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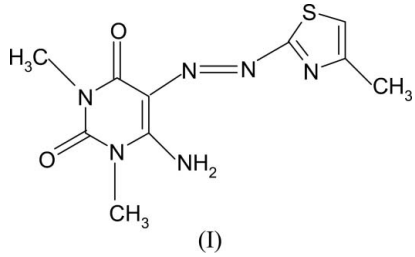
In the molecule of the title compound, $C_{10}H_{12}N_6O_2S$, the thiazole ring is oriented with respect to the uracil ring at a dihedral angle of 8.08 (9)°. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds, forming infinite chains running along the [001] direction.

Comment

6-Aminouracils are key intermediates in the synthesis of purines (Shaw, 1996), which constitute the basis of a number of drugs, such as caffeine, penciclovir, theobromine and theophylline. Methylthiouracil and propylthiouracil are thyroid inhibitors, and fluorouracil and its masked compounds are anticancer agents. Uracil units were also detected in the antibiotic tuicamycin (Danishefsky & Barbachyn, 1985). Furthermore, many mono- and bicyclic uracils are used to protect plants, mostly as herbicides (Brown, 1984). The thiazole ring has been identified as a central structural element of a number of biologically active natural products (Zabriskie *et al.*, 1988) and pharmacologically active compounds (Metzger, 1984); heterocycles containing this ring exhibit a wide spectrum of biological activities, including antiviral and antifungal activities. Azo dyes have wide applicability as optical materials and so their structures have also attracted considerable attention (Biswas & Umapathy, 2000). We report here the crystal structure of the title uracil derivative, (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 uracil rings *A* (S1/N1/C1–C3) and *B* (N4/N5/C4–C7) are both planar. The dihedral angle between the two rings is 8.08 (9)°. An intramolecular N—H···N hydrogen bond (Table 1) results in the formation of a third planar ring *C* (H62/N2/N3/N6/C4/C5), which makes dihedral angles of 7.78 (9) and 1.41 (7)° with rings *A* and *B*, respectively.

As can be seen from the packing diagram (Fig. 2), intermolecular N—H···O hydrogen bonds (Table 1) link the molecules, forming infinite chains along the [001] direction.

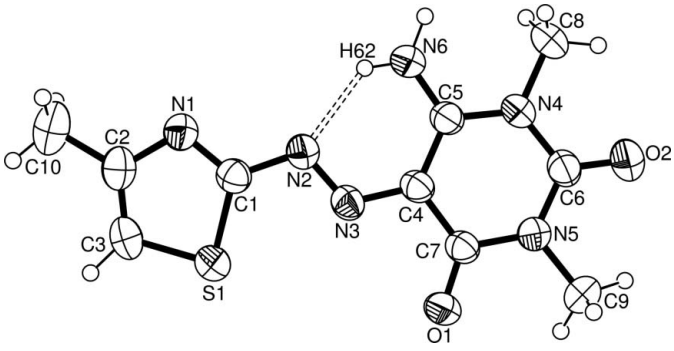


Figure 1
The molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N—H···N hydrogen bond is shown as a double dashed line.

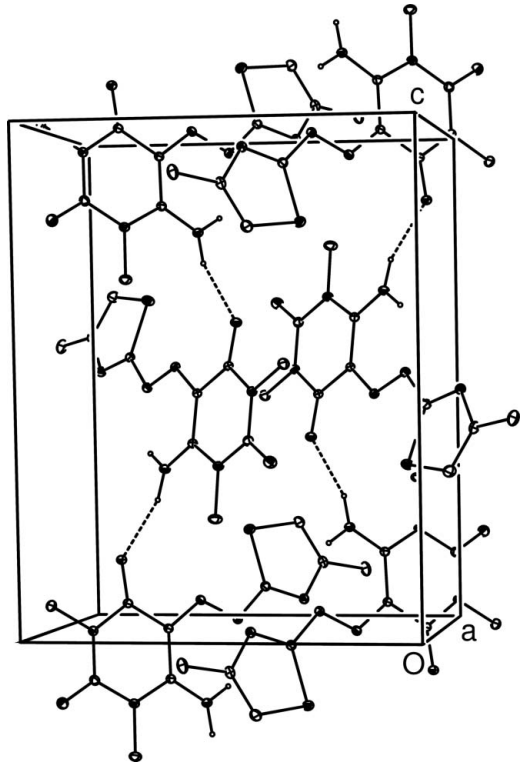


Figure 2
A packing diagram of (I). Intermolecular N—H···O hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Dipole–dipole and van der Waals interactions are effective in the molecular packing.

Experimental

2-Amino-4-methylthiazole (202 mg, 2 mmol) was dissolved in a hot glacial acetic acid–propionic acid mixture (2:1 *v*/*v*, 6 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 cooled in an ice–salt bath and then added dropwise with stirring to 6-amino-1,3-dimethyluracil (310 mg, 2 mmol) in an aqueous solution of KOH (2 mmol, in 8 ml water). The solution was stirred at 273 K for 2 h and the pH of the reaction mixture was maintained at 4–6 by the 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 490 mg, 88%; m.p. 541–543 K).

Crystal data

$C_{10}H_{12}N_6O_2S$
M_r = 280.32
Monoclinic, $P2_1/c$
a = 9.3604 (3) Å
b = 10.3052 (3) Å
c = 13.2638 (5) Å
β = 93.391 (2)°
V = 1277.20 (7) Å^3
Z = 4
D_x = 1.458 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
Block, orange
0.35 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
ω scans
Absorption correction: none
37304 measured reflections
3897 independent reflections
2108 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 30.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.219
S = 1.07
3897 reflections
187 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0966P)^2 + 0.0287P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H61···O1^{i}	0.91 (4)	1.96 (4)	2.834 (3)	160 (3)
N6—H62···N2	0.84 (4)	1.97 (4)	2.634 (4)	135 (3)

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

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 = 0.83 (4) and 0.91 (4) Å, C—H = 0.98 (4)Å and U_{iso}(H) = 0.065 (10)–0.082 (11) 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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