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7-Amino-1,2,3,4-tetrahydroquinazoline-2,4-dithione

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The title molecule, $C_8H_7N_3S_2$, is essentially planar and exists in the thione form. The molecules associate *via* N—H···S interactions to form double chains, which pack in a zigzag fashion *via* N—H···N interactions and π–π stacking.

Comment

Recently, investigations on the photochemistry of thio-carbonyl compounds have received significant attention. Photoinduced single proton transfer reactions, leading to conversion of the thione form to the corresponding thiol, have been observed for a number of heterocycles such as 2(1H)-pyridinethione (Moran *et al.*, 2002), 4-pyrimidinethione (Lapinski *et al.*, 1998) and 2(1H)-quinolinethione (Prusi-nowska *et al.*, 1995). Photoinduced double proton transfer reactions have been observed for three molecules, namely dithiooxamide (Lapinski *et al.*, 2004), 2,6-dithiopurine (Rost-kowska *et al.*, 2003) and 2,4-dithiouracil (Khvorostov *et al.*, 2005). Recently, it was observed that triple proton transfer in trithiocyanuric acid (Rostkowska *et al.*, 2005) is possible. We report here the synthesis and structure determination of a novel thiocarbonyl derivative of quinazoline by a hydro-thermal reaction; the title compound, (I), may be another example of a photoinduced double proton transfer molecule. The crystal structure analysis shows that (I) is planar (Fig. 1), the mean deviation being 0.015 Å from the least-squares plane The S1—C7 and S2—C6 bond distances (Table 1) indicate that the molecule exists in the thione form.

(I)
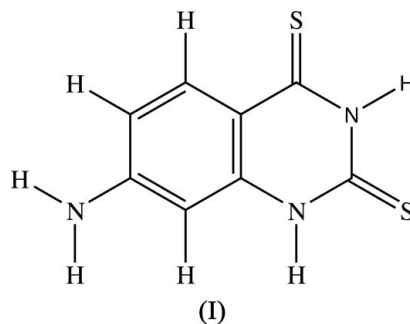
The molecules are linked *via* N—H···S hydrogen bonds (Table 2) to form double chains parallel to the *b* axis, and these are linked *via* N—H···N hydrogen bonds and π–π stacking so as to aggregate in a zigzag fashion (see Fig. 2).

Experimental

A mixed solution of ethanol (10 ml) with water (10 ml) containing *m*-phenylenediamine dihydrochloride (5 mmol) and CS_2 (10 mmol) was
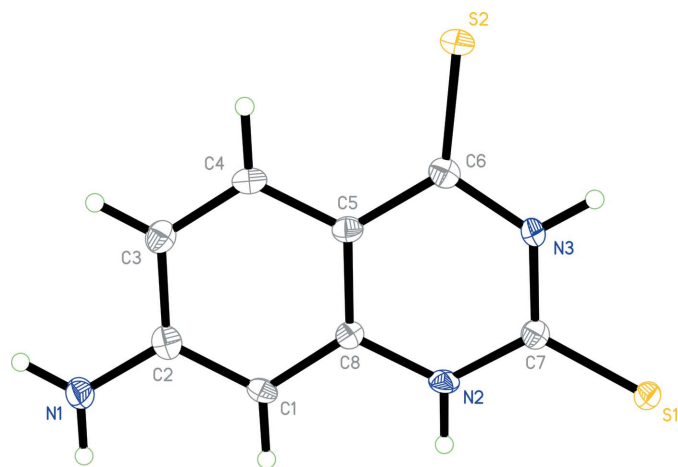
Figure 1
Molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.
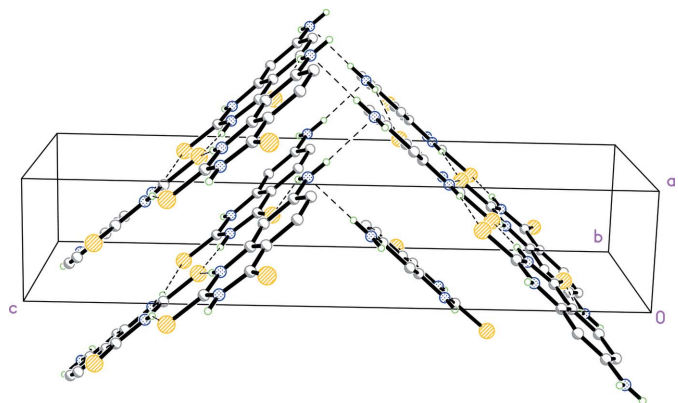

Figure 2
The unit-cell contents for (I). Only H atoms involved in hydrogen bonding are shown and these interactions are depicted by dashed lines.

placed in a 25 ml autoclave with a Teflon liner. The autoclave was heated to 310 K, maintained at that temperature for 5 h, at which time the temperature was raised to 370 K, and kept there for 5 d. The autoclave was cooled to room temperature at a rate of 0.5 K min^{-1}. Red prismatic crystals were collected. The crystals were dissolved in a mixture containing ethanol and water (1:1; 20 ml), and ammonia (2 ml, 1 mol l^{-1}) was added to the solution. The solution was again transferred to the autoclave, heated to 310 K and maintained at that temperature for 5 h. Then the temperature was raised to 370 K and maintained for 5 d. The autoclave was cooled to room temperature at a rate of 0.5 K min^{-1}. Yellow block-shaped crystals were obtained for X-ray diffraction. Elemental analysis found: C 46.71, H 3.91, N 20.98%; calculated for $C_8H_7N_3S_2$: C 45.91, H 3.37, N 20.08, S 30.64%.

Crystal data

$C_8H_7N_3S_2$	$Z = 4$
$M_r = 209.29$	$D_x = 1.636$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 4.419$ (1) Å	$\mu = 0.57$ mm^{-1}
$b = 8.639$ (2) Å	$T = 123$ (2) K
$c = 22.260$ (5) Å	Block, yellow
$V = 849.8$ (3) Å^3	$0.29 \times 0.16 \times 0.06$ mm

Data collection

Bruker SMART APEX 2000 CCD diffractometer	4846 measured reflections
φ and ω scans	1845 independent reflections
Absorption correction: multi-scan	1803 reflections with $I > 2\sigma(I)$
(*SADABS*; Sheldrick, 1996)	$R_{int} = 0.024$
$T_{min} = 0.851$, $T_{max} = 0.966$	$\theta_{max} = 27.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$+ 0.3576P]$
$wR(F^2) = 0.096$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.17$	$(\Delta/\sigma)_{max} < 0.001$
1845 reflections	$\Delta\rho_{max} = 0.60$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
H-atom parameters constrained	Absolute structure: (Flack, 1983), 719 Friedel pairs
	Flack parameter: 0.07 (12)

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.684 (3)	N3—C7	1.351 (3)
S2—C6	1.653 (3)	N3—C6	1.375 (3)
N2—C7	1.343 (3)	C5—C6	1.436 (4)
N2—C8	1.389 (3)		
C7—N2—C8	123.8 (2)	C5—C6—S2	125.8 (2)
C7—N3—C6	126.3 (2)	N2—C7—N3	116.6 (2)
N3—C6—C5	115.5 (2)	N2—C7—S1	122.1 (2)
N3—C6—S2	118.8 (2)	N3—C7—S1	121.3 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$B\cdots$S2^i	0.88	2.51	3.315 (3)	152
N1—H1$C\cdots$N1^{ii}	0.88	2.38	3.247 (3)	171
N2—H2$A\cdots$S1^{iii}	0.88	2.65	3.520 (2)	171
N3—H3$B\cdots$S1^{iv}	0.88	2.52	3.369 (2)	164

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

All H atoms were included in the riding-model approximation with C—H and N—H distances of 0.95 and 0.88 Å, respectively, and with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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