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# 7-Amino-1*H*-3,1-benzothiazine-2,4-dithione

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**Key indicators**

Single-crystal X-ray study
*T* = 273 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.059
*wR* factor = 0.160
Data-to-parameter ratio = 17.3

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



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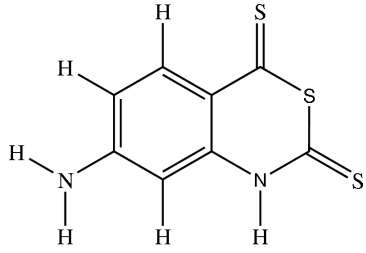
X-ray crystal structure determination shows that the title compound, $C_8H_6N_2S_3$, has a highly planar molecule, packing *via* amino–thione hydrogen bonds.

## Comment

The chemistry of thiocarbonyl (thione) analogues of such carbonyl functions as ketones, acids, esters and amides has been the subject of extensive study (Duus, 1979). Lakshmikantham *et al.* (1984) reported the crystal structure of the first thione analogue of a cyclic anhydride. We report here the synthesis and characterization of a new thione analogue of a cyclic anhydride containing nitrogen, 7-amino-1*H*-benzo[*d*]-[1,3]thiazine-2,4-dithione, (I), obtained by the hydrothermal reaction of 1,3-phenylenediamine and carbon disulfide.




(I)

The present crystal structure analysis shows that the molecule of (I) is highly planar, with a small (0.0054 Å) mean

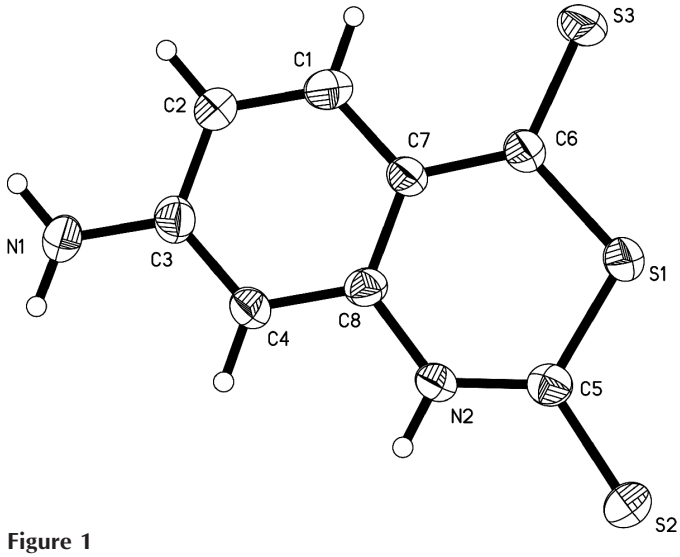



**Figure 1**
A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at 50% probability level and H atoms are shown as small spheres of arbitrary radii.

deviation from the plane including all non-H atoms. The molecule features long S1—C5 [1.727 (3) Å] and S1—C6 [1.735 (3) Å] bonds, and short S2—C5 [1.663 (3) Å] and S3—C6 [1.660 (3) Å] bonds.

The molecules of (I) pack in the solid state *via* amino–thione hydrogen bonds, with N2···S3$^{i}$ 3.407 (3) Å, N1···S2$^{ii}$ 3.530 (3) Å and N1···S2$^{iii}$ 3.610 (3) Å [symmetry codes: (i) $x$, $\frac{1}{2}-y$, $z+\frac{1}{2}$; (ii) $1+x$, $\frac{1}{2}-y$, $z+\frac{1}{2}$; (iii) $1-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$; Table 2).

## Experimental

An ethanol solution (20 ml) containing *m*-phenylenediamine dihydrochloride (5 mmol) and carbon disulfide (10 mmol) was placed in a 25 ml autoclave with a Teflon liner. The pH of the solution was adjusted to 7–8 with sodium hydroxide solution. The autoclave was heated to 373 K, maintained at that temerature for 96 h and cooled to room temperature at a rate of 0.5 K min$^{-1}$. Red block-shaped crystals of (I) were obtained for X-ray diffraction. Analysis, calculated for $C_8H_6N_2S_3$: C 42.45, H 2.67, N 12.38, S 42.50%; found: C 43.16, H 2.55, N 12.67, S 41.97%. Spectroscopic analysis: $^1$H NMR ($CD_3COCD_3$, $\delta$, p.p.m.): 6.65 (ArH), 6.68 ($NH_2$), 6.73 (ArH), 8.25 (ArH), 12.04 (NH).

*Crystal data*

$C_8H_6N_2S_3$
$M_r$ = 226.33
Monoclinic, $P2_1/c$
$a$ = 3.9117 (9) Å
$b$ = 17.232 (4) Å
$c$ = 13.686 (3) Å
$\beta$ = 97.116 (4)°
$V$ = 915.4 (3) Å$^3$
$Z$ = 4
$D_x$ = 1.642 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 4962 reflections
$\theta$ = 2.0–27.0°
$\mu$ = 0.76 mm$^{-1}$
$T$ = 273 (2) K
Block, red
0.38 × 0.22 × 0.16 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.762, $T_{max}$ = 0.889
5279 measured reflections
2042 independent reflections
1779 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028
$\theta_{max}$ = 27.5°
$h = -4 \rightarrow 5$
$k = -17 \rightarrow 22$
$l = -17 \rightarrow 15$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.160
$S$ = 1.14
2042 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0963P)^2 + 0.2434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.0001$
$\Delta\rho_{max}$ = 0.75 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—C5 | 1.727 (3) | C7—C8 | 1.409 (4) |
| S1—C6 | 1.735 (3) | C7—C6 | 1.431 (4) |
| S2—C5 | 1.663 (3) | C4—C3 | 1.388 (4) |
| S3—C6 | 1.660 (3) | C4—C8 | 1.393 (4) |
| N2—C5 | 1.338 (4) | N1—C3 | 1.359 (4) |
| N2—C8 | 1.396 (4) | C1—C2 | 1.366 (4) |
| C7—C1 | 1.405 (4) | C3—C2 | 1.409 (4) |
| | | | |
| C5—S1—C6 | 107.22 (14) | C7—C6—S1 | 119.8 (2) |
| C5—N2—C8 | 127.8 (2) | S3—C6—S1 | 113.21 (17) |
| C1—C7—C8 | 117.6 (3) | C2—C1—C7 | 122.1 (3) |
| C1—C7—C6 | 119.8 (3) | N2—C8—C4 | 116.4 (2) |
| C8—C7—C6 | 122.7 (3) | N2—C8—C7 | 123.0 (2) |
| C3—C4—C8 | 120.6 (3) | C4—C8—C7 | 120.6 (3) |
| N2—C5—S2 | 123.8 (2) | N1—C3—C4 | 121.0 (3) |
| N2—C5—S1 | 119.5 (2) | N1—C3—C2 | 119.8 (3) |
| S2—C5—S1 | 116.64 (17) | C4—C3—C2 | 119.1 (3) |
| C7—C6—S3 | 127.0 (2) | C1—C2—C3 | 120.0 (3) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$B$···S3$^{i}$ | 0.86 | 2.58 | 3.407 (3) | 163 |
| N1—H1$B$···S2$^{ii}$ | 0.86 | 2.72 | 3.530 (3) | 157 |
| N1—H1$C$···S2$^{iii}$ | 0.86 | 2.79 | 3.610 (3) | 160 |

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

All H atoms were positioned geometrically, with C—H distances of 0.93 Å and N—H distances of 0.86 Å, and were refined using a riding-atom model, with $U_{iso}$(H) = 1.2$U_{eq}$(C, N).

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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