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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.041
 wR factor = 0.085
Data-to-parameter ratio = 13.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[4-phenyl-5-(1*H*-1,2,4-triazol-3-yl)-
4*H*-1,2,4-triazol-3-yl] disulfide

The title compound, $\text{C}_{20}\text{H}_{14}\text{N}_{12}\text{S}_2$, was synthesized by the reaction of 3-hydrazino-1*H*-1,2,4-triazole with phenyl isocyanate in benzene and by ring closure in an alkaline medium. Intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds are observed and these form a five-membered ring.

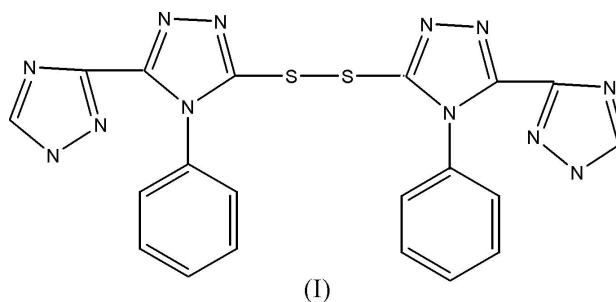
Received 3 February 2005

Accepted 28 February 2005

Online 11 March 2005

Comment

Azole derivatives, such as derivatives of pyrazole, imidazole, triazole (including benzotriazole), tetrazole, indole, *etc.*, exhibit extensive biological activities. They have become a central focus in the study of agricultural chemicals, adjustment reagents for plant growth, and so on (Haddock & Hopwood, 1982). We have reported the crystal structures of two triazole compounds (Pan & Yang, 2005; Yang & Pan, 2004). In a search for more effective antibacterial medicines, we have synthesized the title compound, (I).



The dihedral angle between the C15–C20 and N7/N8/C12/N9/C11 rings is $70.06(8)^\circ$ and that between the C5–C10 ring and the N1/N2/C2/N3/C1 plane is $68.48(8)^\circ$. The torsion angles C1–S1–S2–C11 and S2–S1–C1–N3 are $-78.49(9)$ and $-65.25(17)^\circ$, respectively. Intermolecular $\text{N11}-\text{H11}\cdots\text{N2}^i$ and $\text{N11}-\text{H11}\cdots\text{N6}^i$ hydrogen bonds form a five-membered ring [Fig. 2; symmetry code (i): $2 - x, 1 - y, -z$]. An intermolecular $\text{N5}-\text{H5}\cdots\text{N12}^{ii}$ hydrogen bond is also observed [symmetry code (ii): $2 - x, \frac{1}{2} + y, \frac{1}{2} + z$].

Experimental

3-Hydrazino-1*H*-1,2,4-triazole (0.02 mol, 2.54 g) was dissolved in benzene (50 ml) and phenyl isocyanate (0.02 mol, 2.70 g) was added. The mixture was refluxed for 8 h and the precipitate formed was collected by filtration and washed with benzene. The product was recrystallized from benzene and dried under reduced pressure to give 4-phenyl-1-(1*H*-1,2,4-triazole-3-hydrazino)thiosemicarbazide. Ring closure of this compound in an alkaline medium is a well known method for the synthesis of the title compound (Cansiz *et al.*, 2004). The compound (2.0 mmol, 0.97 g) was dissolved in dimethylform-

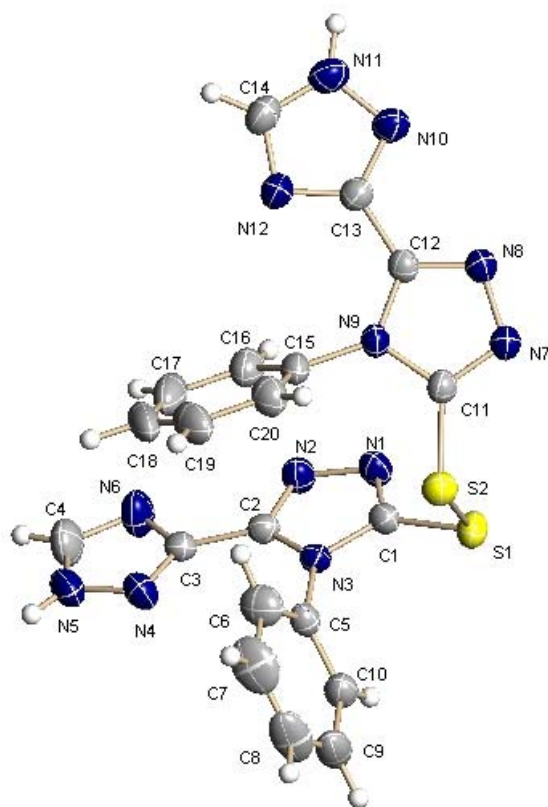


Figure 1

The structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

amide (30 ml) and kept at room temperature for 40 d, producing colourless single crystals, which were collected and washed with distilled water.

Crystal data

$C_{20}H_{14}N_{12}S_2$
 $M_r = 486.55$
 Monoclinic, $P2_1/c$
 $a = 9.8647$ (8) Å
 $b = 13.3308$ (10) Å
 $c = 16.9443$ (14) Å
 $\beta = 101.630$ (1)°
 $V = 2182.5$ (3) Å³
 $Z = 4$

$D_x = 1.481$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2532 reflections
 $\theta = 4.9$ – 48.9 °
 $\mu = 0.28$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.32 \times 0.30 \times 0.29$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.916$, $T_{\max} = 0.922$
 12720 measured reflections

4763 independent reflections
 3123 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\text{max}} = 27.0$ °
 $h = -12 \rightarrow 12$
 $k = -17 \rightarrow 12$
 $l = -15 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.085$
 $S = 0.87$
 4763 reflections
 363 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0331P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

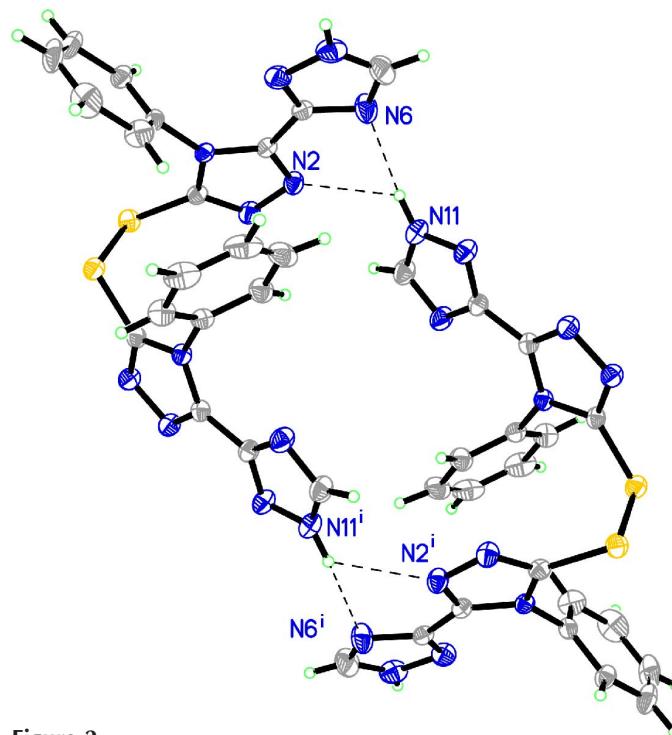


Figure 2

Diagram of (I), showing the intermolecular hydrogen bonds as dashed lines (the symmetry code is as in Table 1).

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N11-H11 \cdots N2^i$	0.90 (2)	2.49 (2)	2.999 (2)	117.0 (16)
$N11-H11 \cdots N6^i$	0.90 (2)	2.18 (2)	3.045 (3)	163.6 (19)
$N5-H5 \cdots N12^{ii}$	0.86 (2)	2.17 (3)	3.003 (3)	163 (2)

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

All H atoms were located in a difference map and their parameters were refined. The N–H distances are 0.86 (2) and 0.90 (2) Å and the C–H distances are in the range 0.82 (3)–0.97 (2) Å.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2002); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

The authors acknowledge financial support by the Zhejiang Provincial Natural Science Foundation of China (No. M203115).

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