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1,6-Bis(1-phenyl-1*H*-tetrazol-5-ylsulfanyl)hexane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.110
Data-to-parameter ratio = 15.9

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

The title compound, $C_{20}H_{22}N_8S_2$, has a center of inversion at the mid-point of the central C—C bond, and all C—C σ bonds in the hexamethylene group adopt the normal *anti* staggered conformation. The two tetrazolyl rings are parallel to each other. The dihedral angle between the phenyl ring and the attached tetrazolyl ring is 41.8 (3)°.

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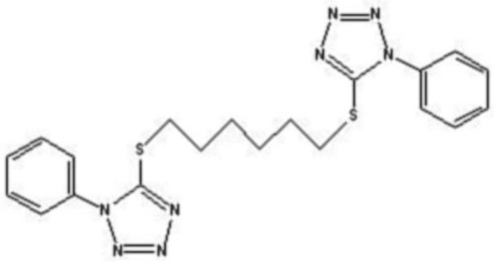
Comment

As a type of ditopic ligand, dithioethers can be used as bridging ligands in the construction of coordination polymers with soft metal ions, and a series of flexible or rigid chain-linked dithioethers containing *N*-heterocyclic moieties have been synthesized and investigated (Sharma *et al.*, 1999; Constable *et al.*, 2002; Bu *et al.*, 2003; Hong *et al.*, 2000; Zheng *et al.*, 2003). Earlier studies have shown that several tetrazole derivatives possess diverse pharmacological properties (Juby *et al.*, 1968, 1982), but only a few complexes of monosubstituted tetrazole derivatives have been characterized crystallographically (van den Heuvel *et al.*, 1983; Lyakhov *et al.*, 2003). We report here the synthesis and structure of a new tetrazole dithioether, 1,6-bis(1-phenyl-1*H*-tetrazol-5-ylsulfanyl)hexane, (I).



(I)

In (I), there is an inversion center at the mid-point of the C10—C10*A* bond, and all C—C σ bonds in the hexamethylene group adopt the normal *anti* staggered conformation to reduce steric strain (Fig. 1). The two tetrazolyl rings are parallel, the C7—S1···S1*A*—C7*A* angle being 180°. The dihedral angle between the phenyl ring and the attached tetrazolyl ring is 41.8 (3)°. The Csp^2—S bond distance [C7—S1 = 1.726 (2) Å] is significantly shorter than the Csp^3—S bond [C8—S1 = 1.811 (2) Å], as a result of *p*-π conjugation; these distances are similar to those observed in other dithioethers (Zhang *et al.*, 2003; Zheng & Liu, 2003). The other bond distances are within normal ranges (Allen *et al.*, 1987).

In the crystal structure, the molecules are linked into a two-dimensional network *via* intermolecular C—H···N hydrogen bonds, as depicted in Fig. 2; details are given in Table 1.

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Experimental

The title compound was synthesized as follows: a solution of 1,6-dibromohexane (1.22 g, 5 mmol) in ethanol (10 ml) was added dropwise to a mixture of 1-phenyl-5-thio-1,2,3,4-tetrazole (1.96 g, 11 mmol), KOH (0.615 g, 11 mmol) and ethanol (10 ml). The reaction mixture was stirred for 24 h at room temperature. The precipitate was filtered off, washed with water and recrystallized from ethanol. Yield: 70%; m.p. 432–433 K. IR (KBr, ν cm^{-1}): 2940 (*m*), 2865 (*w*), 1596 (*m*), 1499 (*vs*), 1403 (*m*), 1383 (*s*), 1280 (*m*), 1239 (*m*), 1074 (*m*), 1016 (*m*), 765 (*s*), 695 (*s*). ^{1}H NMR (CDCl$_3$, p.p.m.): δ 1.49 (*m*, 4H), 1.83 (*m*, 4H), 3.37 (*t*, 4H), 7.54 (*m*, 10H); ^{13}C NMR (CDCl$_3$, p.p.m.): 27.97, 28.93, 33.10, 123.83, 129.77, 130.08, 133.71, 154.34. Analysis calculated for $C_{20}H_{22}N_8S_2$: C 54.72, H 5.02, N 25.54%; found: C 54.65, H 5.12, N 25.45%. Crystals suitable for single-crystal X-ray analysis were obtained by recrystallization from an acetonitrile solution.

Crystal data

$C_{20}H_{22}N_8S_2$
$M_r = 438.58$
Monoclinic, $P2_1/c$
$a = 7.467$ (3) Å
$b = 16.753$ (6) Å
$c = 8.606$ (3) Å
$\beta = 101.587$ (7)°
$V = 1054.7$ (6) Å^3
$Z = 2$

$D_x = 1.381$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 594 reflections
$\theta = 2.8$–22.9°
$\mu = 0.28$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.26 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.935$, $T_{\max} = 0.967$
6037 measured reflections

2160 independent reflections
1305 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.048$
$\theta_{\max} = 26.4°$
$h = -6 \rightarrow 9$
$k = -20 \rightarrow 19$
$l = -10 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.110$
$S = 1.00$
2160 reflections
136 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.0623P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···N4$^{\rm i}$	0.97	2.61	3.576 (3)	173

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

H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) values constrained to $1.2U_{\rm eq}$ of the parent atom.

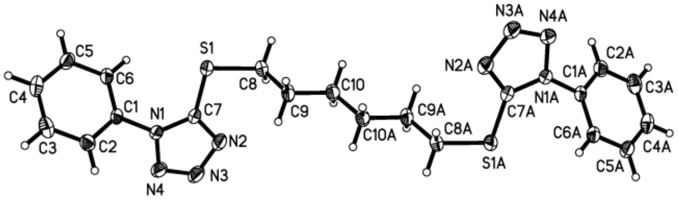

Figure 1
View of (I), with the atom labeling scheme and displacement ellipsoids drawn at the 30% probability level. Atoms labeled with the suffix *A* are related to those without the suffix *A* by ($-x$, $2 - y$, $-z$).

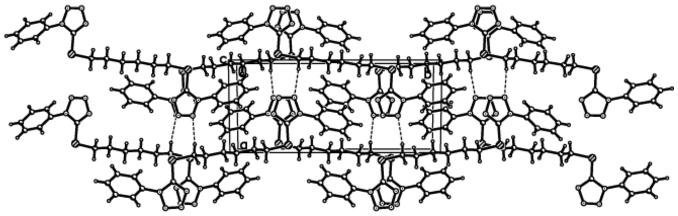

Figure 2
The two-dimensional network formed by C—H···N hydrogen bonds (dashed lines).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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