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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 = 0.038
 wR factor = 0.109
Data-to-parameter ratio = 15.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1,4-Bis(1-phenyl-1*H*-tetrazol-5-ylsulfanyl)butane

In the title compound, $\text{C}_{18}\text{H}_{18}\text{N}_8\text{S}_2$, the two 1-phenyl-1*H*-tetrazole-5-thiol moieties are located on the same side of the butane-1,4-diyl chain but point in opposite directions.

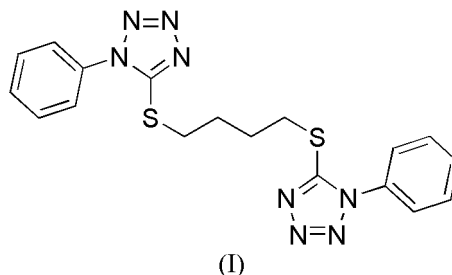
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Comment

As a type of ditopic ligand, dithioethers can be used as bridging ligands in the construction of coordination polymers with soft metal ions. 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 reported that several tetrazole derivatives possess diverse pharmacological properties (Juby *et al.*, 1968, 1982), but only a few metal complexes of monosubstituted tetrazole derivatives are known (van den Heuvel *et al.*, 1983; Lyakhov *et al.*, 2003).



We report here the synthesis and structure of a new tetrazole dithioether, namely the title compound, (I).

In the molecular structure of (I), the two 1-phenyl-1*H*-tetrazole-5-thiol moieties are located on the same side of the butane-1,4-diyl chain, but they point in opposite directions to reduce the steric repulsion between them (Fig. 1); the $\text{C}7-\text{N}1-\text{C}6-\text{C}1$ and $\text{C}12-\text{N}8-\text{C}13-\text{C}18$ torsion angles are $52.7(3)$ and $-50.2(3)^\circ$, respectively. The two tetrazole rings are close to antiparallel, with a dihedral angle of $3.8(1)^\circ$ between them, while that between the phenyl rings is $36.0(1)^\circ$.

The $\text{C}8-\text{C}9-\text{C}10-\text{C}11$ torsion angle of $-178.72(17)^\circ$ indicates that the butanediyl group is planar. The $\text{C}sp^2-\text{S}$ bond distances [$\text{C}7-\text{S}1=1.7340(19)$ Å and $\text{C}12-\text{S}2=1.738(2)$ Å] are significantly shorter than the $\text{C}sp^3-\text{S}$ distances [$\text{C}8-\text{S}1=1.813(2)$ Å and $\text{C}11-\text{S}2=1.8042(19)$ Å], as a result of $\pi-\pi$ conjugation; these distances are similar to those observed in other dithioethers (Zhang *et al.*, 2003; Zheng & Liu, 2003; Wang, Liu, Zheng & Zhang, 2004; Wang, Liu & Zhang, 2004). The other bond distances are within normal ranges (Allen *et al.*, 1987).

Experimental

A solution of 1,4-dibromobutane (1.08 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 then stirred for 24 h at room temperature. The precipitate was filtered off, washed with water and recrystallized from ethanol (yield 74%; m.p. 415–416 K). IR (KBr, ν cm⁻¹): 3068 (w), 1596 (m), 1450 (s), 1461 (w), 1388 (s), 1274 (m), 1238 (m), 1093 (m), 1072 (m), 1014 (m), 975 (w), 843 (w), 763 (s), 688 (s), 551 (m); ¹H NMR (CDCl₃, p.p.m.): 1.99–2.03 (m, 4H, CH₂-), 3.40–3.43 (m, 4H, S-CH₂-), 7.24–7.55 (m, 4H, Ph-). Analysis calculated for C₁₈H₁₈N₈S₂: C 52.68, H 4.39, N 27.32%; found: C 52.66, H 4.35, N 27.36%. Crystals suitable for single-crystal X-ray analysis were obtained by recrystallization from an acetonitrile solution.

Crystal data

C ₁₈ H ₁₈ N ₈ S ₂	$D_x = 1.414$ Mg m ⁻³
$M_r = 410.52$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2155 reflections
$a = 9.4630$ (17) Å	$\theta = 2.6$ – 26.2°
$b = 12.879$ (2) Å	$\mu = 0.30$ mm ⁻¹
$c = 16.440$ (3) Å	$T = 293$ (2) K
$\beta = 105.802$ (3)°	Prism, colorless
$V = 1927.9$ (6) Å ³	$0.42 \times 0.38 \times 0.22$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3955 independent reflections
φ and ω scans	2953 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.882$, $T_{\text{max}} = 0.936$	$\theta_{\text{max}} = 26.4^\circ$
10 993 measured reflections	$h = -11 \rightarrow 11$
	$k = -16 \rightarrow 14$
	$l = -20 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.5664P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.109$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.35$ e Å ⁻³
3955 reflections	$\Delta\rho_{\text{min}} = -0.32$ e Å ⁻³
253 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

S1–C7	1.7340 (19)	S2–C12	1.738 (2)
S1–C8	1.813 (2)	S2–C11	1.8042 (19)
C7–S1–C8	99.94 (9)	C7–N1–C6	129.70 (16)
C12–S2–C11	100.51 (9)	C12–N8–C13	130.65 (16)
N1–N2–N3–N4	−0.3 (2)	N5–N6–N7–N8	−0.3 (2)
N2–N3–N4–C7	0.3 (2)	C8–C9–C10–C11	−178.72 (17)
C12–N5–N6–N7	−0.2 (2)		

All H atoms were positioned geometrically and refined as riding (C–H = 0.93–0.97 Å). For CH and CH₂ groups, $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{carrier atom})$ and for the methyl groups they were set equal to $1.5U_{\text{eq}}(\text{carrier atom})$.

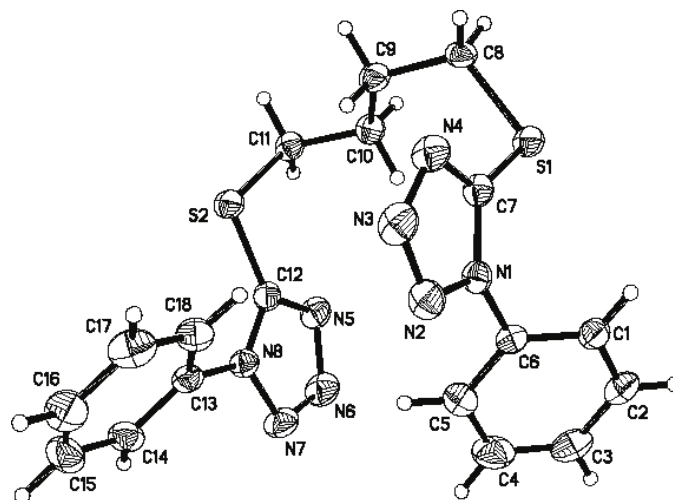


Figure 1

View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

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