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trans-5,5′-[(2-Butene-1,4-diyl)dithio]bis(1-phenyl-1,2,3,4-tetrazole)

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Key indicators

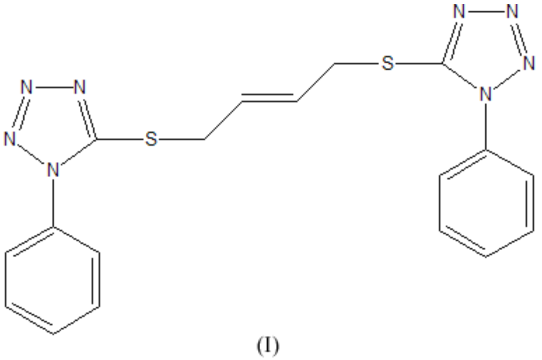
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.097
Data-to-parameter ratio = 15.8

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

The title compound, $C_{18}H_{16}N_8S_2$, crystallizes in the monoclinic space group $P2_1/c$. The two 1-phenyl-1*H*-tetrazole-5-thiol moieties are located on the same side of the butene-1,4-diyl plane but point in opposite directions. Intermolecular N···S interactions link the molecules to form one-dimensional polymeric chains along the *a* axis.

Comment

To date, a large number of flexible or rigid chain-linked dithioether ligands containing *N*-heterocyclic moieties have been synthesized and investigated, due to their diverse coordination capabilities and the important properties of their metal complexes (Zheng *et al.*, 2003; Bu *et al.*, 2002; Hong *et al.*, 2000). Earlier studies reported that several tetrazole derivatives possess diverse pharmacological properties (Juby *et al.*, 1968, 1982). However, crystallographic studies of only a few complexes of monosubstituted tetrazole derivatives have been reported (van den Heuvel *et al.*, 1983; Lyakhov *et al.*, 2003). Here, we report the synthesis and structure of the title novel tetrazole thioether, (I). Further work on the reaction of this compound with transition metal ions is still in progress.


(I)

In (I), the two 1-phenyl-1*H*-tetrazole-5-thiol moieties are located on the same side of the butene-1,4-diyl chain, but they point in opposite directions to reduce the steric repulsion between them (Fig. 1). The two tetrazole rings are close to antiparallel, with a dihedral angle of 7.4 (1)° between them, while that between the benzene rings is 38.6 (1)°. The two 1-phenyl-1*H*-tetrazole groups are in different conformations; the dihedral angle between the tetrazole and benzene rings within one group is 46.3 (1)° and that in the other is 51.8 (1)°.

The C═C bond distance of 1.297 (3) Å within the butene-1,4-diyl group is shorter than the value of 1.34 Å for a normal C═C bond (Lide, 1992–1993) and equal to the value of 1.299 (5) Å in *trans*-*S*,*S*′-(but-2-ene-1,4-diyl)bis(L-cysteine) (Shi *et al.*, 2002). The C8—C9—C10—C11 torsion angle of

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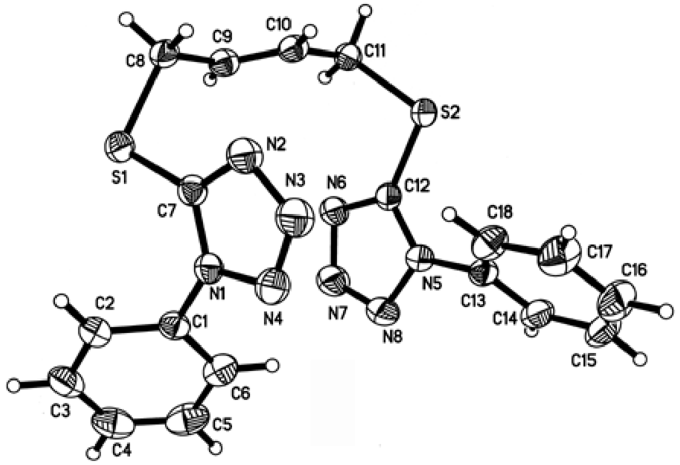

Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

−179.6 (2)° indicates that the butenediyl group is planar. Because of π–π conjugation, the Csp^2—S bonds (C7—S1 and C12—S2) are significantly shorter than the Csp^3—S bonds (C8—S1 and C11—S2), similar to the effect observed in 2,2′-[1,4-phenylenebis(methylenethio)]dithiazole (Zhang *et al.*, 2003).

As shown in Fig. 2, intermolecular N···S interactions [N2···S2($1 - x$, $-y$, $-z$) 3.304 (2) and N6···S1($2 - x$, $-y$, $-z$) 3.282 (2) Å] link the molecules of (I) to form one-dimensional polymeric chains along the *a* axis. In addition, the crystal structure is stabilized by C—H···S and C—H···N interactions (Table 1).

Experimental

The title compound was synthesized as follows. A solution of *trans*-1,4-dibromo-2-butene (1.07 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 67%, m.p. 401–402 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3073 (*w*), 3024 (*w*), 2979 (*w*), 2935 (*w*), 1596 (*m*), 1499 (*s*), 1406 (*w*), 1381 (*s*), 1316 (*m*), 1282 (*m*), 1236 (*s*), 1094 (*m*), 992 (*s*), 763 (*s*), 693 (*s*); ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 3.96 (*m*, 4H), 5.99 (*m*, 2H), 7.53 (*m*, 5H); ^{13}C NMR (CDCl$_3$, δ, p.p.m.): 34.42, 123.93, 129.03, 129.80, 130.19. Analysis calculated for $C_{18}H_{16}N_8S_2$: C 52.87, H 3.92, N 27.42%; found: C 52.90, H 4.05, N 27.50%. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in acetonitrile.

Crystal data

$C_{18}H_{16}N_8S_2$
M_r = 408.51
Monoclinic, $P2_1/c$
a = 9.727 (3) Å
b = 12.770 (4) Å
c = 15.947 (6) Å
β = 100.332 (6)°
V = 1948.7 (11) Å^3
Z = 4
D_x = 1.392 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 893 reflections
θ = 3.1–23.4°
μ = 0.30 mm^{-1}
T = 293 (2) K
Prism, colourless
0.24 × 0.20 × 0.16 mm

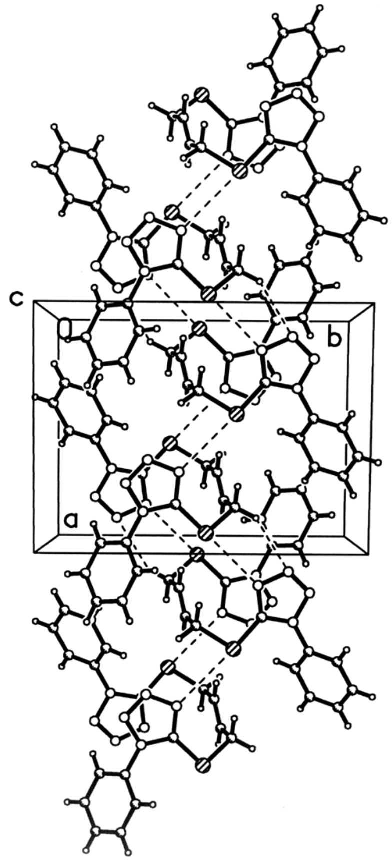

Figure 2
A packing diagram for (I), viewed down the *c* axis. The dashed lines show N···S short contacts between neighbouring molecules.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.928, $T_{\max}$ = 0.954
11 016 measured reflections
3987 independent reflections
2561 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.038
$\theta_{\max}$ = 26.4°
$h = -12 \rightarrow 9$
$k = -12 \rightarrow 15$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.097
S = 1.01
3987 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.1948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.22 e Å^{-3}
$\Delta\rho_{\min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···S1	0.93	2.84	3.226 (3)	106
C8—H8A···N7$^{\rm i}$	0.97	2.61	3.471 (3)	148
C11—H11B···N6	0.97	2.58	3.030 (3)	108

Symmetry code: (i) $2 - x, -y, -z$.

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) = 1.2$U_{\rm eq}$(parent).

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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