

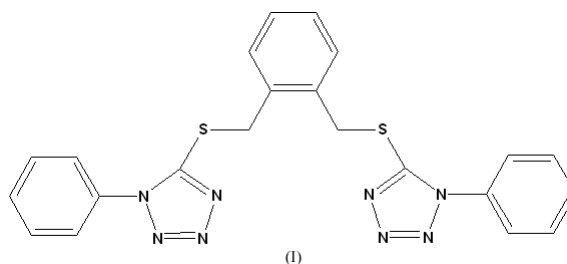
1,1'-Diphenyl-5,5'-[*o*-phenylenebis(methylene-thio)]di-1*H*-tetrazoleXiao-Lan Luo,^a Wei Wang,^b
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Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.051
 wR factor = 0.138
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.In the crystal packing of the title compound, $\text{C}_{22}\text{H}_{18}\text{N}_8\text{S}_2$, the two terminal (1-phenyltetrazol-5-yl)sulfanyl groups adopt a *trans* configuration with respect to the central benzene ring and the dihedral angle between the two tetrazole planes is $54.2(2)^\circ$.

Comment

Early studies reported that several tetrazole derivatives possess diverse pharmacological properties (Juby *et al.*, 1968, 1982) and the crystal structures of complexes of several monosubstituted tetrazole derivatives were studied (Heuvel *et al.*, 1983; Lyakhov *et al.*, 2003). However, there are few studies on tetrazole derivatives containing two or more tetrazole groups. We have reported some tetrazole derivatives using flexible chains as linkers, such as 1,2-diethyl, 1,6-dihexyl, and 1,4-dibutyl (Wang *et al.*, 2004*a,b*, 2005). We report here a new tetrazole derivative using rigid 1,2-phenylene as linker, namely 1,1'-diphenyl-5,5'-[*o*-phenylenebis(methylenethio)]di-1*H*-tetrazole, (I).The two terminal (1-phenyl-1,2,3,4-tetrazol-5-yl)sulfanyl groups adopt a *trans* configuration with respect to the central benzene ring (Fig. 1) and the dihedral angle between the two tetrazole planes is $54.2(2)^\circ$. The improper torsion angle of the two C—S bonds ($\text{S1}-\text{C8}\cdots\text{C15}-\text{S2}$) is $134.2(3)^\circ$, forcing the two 1-phenyl-1,2,3,4-tetrazole rings to extend in opposite directions to minimize the steric hindrance. The dihedral angles between the two tetrazole rings and the central benzene ring are $62.7(1)$ and $116.8(2)^\circ$. The dihedral angle between the two benzene rings (attached to tetrazole rings) is $40.2(2)^\circ$.The p - π conjugation of atom S1 with the tetrazole ring affects the bond distance C7—S1 [$1.730(3)$ Å] which is shorter than C8—S1 [$1.833(3)$ Å]. This effect is also observed for S2 and for other tetrazole-thio derivatives.An isomer of (I) is reported in the following paper (Wang *et al.*, 2005).

Experimental

A solution of 1,2-dibromomethylbenzene (1.32 g, 5 mmol) in tetrahydrofuran (10 ml) was added dropwise to a mixture of 5-mercapto-

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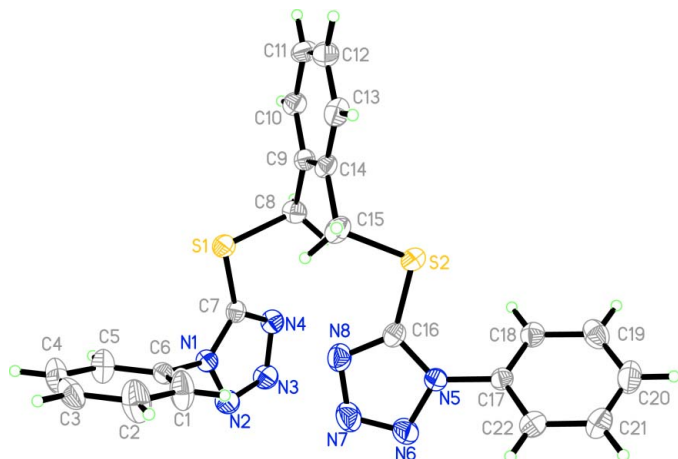


Figure 1
View of the title compound, showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

1-phenyl-1,2,3,4-tetrazole (1.96 g, 11 mmol), KOH (0.62 g, 11 mmol) and ethanol (20 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 78%, m.p. 433–434 K). IR (KBr, ν , cm^{-1}): 3064 (w), 2361 (m), 1595 (m), 1500 (s), 1456 (w), 1391 (s), 1275 (m), 1240 (s), 1092 (m), 1074 (m), 1017 (m), 919 (w), 764 (s), 692 (s), 555 (m); ^1H NMR (CDCl_3): δ 4.75 (s, 4H), 7.24–7.54 (m, 14H); ^{13}C NMR (CDCl_3): δ 34.94, 123.79, 129.11, 129.78, 130.16, 133.93, 153.55. Analysis calculated for $\text{C}_{22}\text{H}_{18}\text{N}_8\text{S}_2$: C 57.64, H 3.93, N 24.45%; found: C 57.79, H 3.90, N 24.38%. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in a mixture of acetonitrile and chloroform in the volume ratio 1:1.

Crystal data

$\text{C}_{22}\text{H}_{18}\text{N}_8\text{S}_2$
 $M_r = 458.56$
Orthorhombic, *Pbca*
 $a = 12.485$ (4) Å
 $b = 13.666$ (4) Å
 $c = 25.801$ (8) Å
 $V = 4402$ (2) Å³
 $Z = 8$
 $D_x = 1.384$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 908 reflections
 $\theta = 2.4$ – 21.8°
 $\mu = 0.27$ mm⁻¹
 $T = 273$ (2) K
Block, colourless
 $0.24 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.820$, $T_{\text{max}} = 0.960$
21475 measured reflections

3881 independent reflections
2519 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -14 \rightarrow 13$
 $k = -16 \rightarrow 15$
 $l = -30 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.138$
 $S = 1.05$
3881 reflections
277 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 2.6547P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.005$
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

All H atoms were positioned geometrically and refined in a riding model (C–H = 0.93–0.97 Å), with U_{iso} values set at 1.2 (CH and CH₂) or 1.5 (CH₃) times U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1997); 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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