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2,2′-Bis(1-phenyl-1*H*-tetrazol-5-ylsulfanyl) ether

Wei Wang,[a,b]* Bing Zhao[b] and Wen-Qin Zhang[a]

[a]Department of Chemistry, Tianjin University, Tianjin 300072, People's Republic of China, and [b]School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: wangweitfj@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.119
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.

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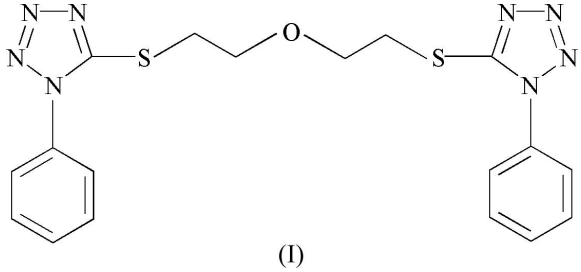
In the title compound, $C_{18}H_{18}N_8OS_2$, the two 1-phenyl-1*H*-tetrazole-5-thiol end-groups lie on the same side of the ether chain and are oriented in the same direction. The two phenyl groups attached to the tetrazole rings are almost perpendicular to each other, the dihedral angle being 87.7 (3)°.

Comment

Dithioethers are often used as bridging ligands in the construction of coordination polymers with soft metal ions. Flexible or rigid chain-linked dithioethers containing *N*-heterocyclic groups have been synthesized and investigated (Constable *et al.*, 2002; Hong *et al.*, 2000). Earlier studies have reported that several tetrazole derivatives possess diverse pharmacological properties (July *et al.*, 1968, 1982). We have reported some tetrazole derivatives using flexible and rigid chains as linkers; these include 1,2-diethyl (Wang, Liu, Zheng & Zhang, 2004), 1,4-dibutyl (Wang, Liu & Zhang, 2005) and 1,2-phenylene (Luo *et al.*, 2005). Now, using a flexible 2,2′-ether as linker, we have synthesized a new tetrazole derivative, *viz*. 2,2′-bis(1-phenyl-1*H*-tetrazole-5-ylsulfanyl) ether, (I). We present its crystal structure here.



(I)

In the structure of (I), the two 1-phenyl-1*H*-tetrazole-5-thiol end-groups are located on the same side of the ether chain and are oriented in the same direction. The two phenyl rings are approximately perpendicular to each other, the dihedral angle being 87.7 (3)°. The dihedral angle between the two 1*H*-tetrazole rings is 63.0 (3)°, and those between the phenyl and attached tetrazole rings are 62.7 (3) and 43.6 (3)°. The C10—O1—C9—C8 and C9—O1—C10—C11 torsion angles are 172.2 (3) and 176.8 (3)°, respectively, indicating that the ether chain linking the 1-phenyl-1*H*-tetrazole-5-thiol groups is planar.

As is usual for substituted 1*H*-tetrazoles, atom C7 has a distorted trigonal geometry, with the N4—C7—N1 [108.7 (3)°] and N4—C7—S1 [128.1 (2)°] angles deviating significantly from the ideal sp^2-hybridized value. As a result of π–π conjugation, the Csp^2—S bonds [S1—C7 = 1.743 (3) Å and S2—C12 = 1.739 (4) Å] are significantly shorter than the

Csp^3—S bonds [C8—S1 = 1.818 (3) Å and C11—S2 = 1.812 (3) Å]. These values compare with the values of 1.726 (2), 1.720 (8), 1.800 (3) and 1.811 (2) Å reported in the literature (Wang, Liu & Zhang, 2004; Wang, Zhao *et al.*, 2005).

Experimental

A solution of 1-(2-bromoethoxy)-2-bromoethane (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 70%; m.p. 417–418 K). IR (KBr, ν cm^{-1}): 3055, 2898, 1594, 1500, 1462, 1385, 1282, 1103, 763, 695; ^{1}H NMR ($CDCl_3$): δ 3.57 (*t*, 4H), 3.88 (4H, *t*), 7.50–7.56 (10H, *m*). Analysis calculated for $C_{22}H_{18}N_8OS_2$: C 50.70, H 4.23, N 26.29%; found: C 50.82, H 4.13, N 26.37%. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in acetonitrile.

Crystal data

$C_{18}H_{18}N_8OS_2$
M_r = 426.52
Orthorhombic, *Pbca*
a = 7.2873 (15) Å
b = 18.657 (4) Å
c = 30.047 (6) Å
V = 4085.2 (15) Å^3
Z = 8
D_x = 1.387 Mg m^{-3}
Mo Kα radiation
Cell parameters from 900 reflections
θ = 2.7–22.2°
μ = 0.29 mm^{-1}
T = 293 (2) K
Block, colourless
0.38 × 0.20 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.933, T_{max} = 0.955
21954 measured reflections
4175 independent reflections
2572 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 26.4°
$h = -8 \rightarrow 9$
$k = -21 \rightarrow 23$
$l = -27 \rightarrow 37$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.119
S = 1.04
4175 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 3.8597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

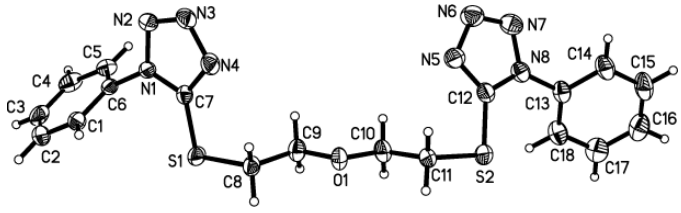


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

All H atoms were positioned geometrically and refined as riding, with Csp^2—H = 0.93 Å and Csp^3—H = 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(parent).

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