Crystal structure of 1,6-bis(1-phenyl-1*H*-tetrazol-5-ylthio)hexane, $C_6H_{12}(SCN_4C_6H_5)_2$

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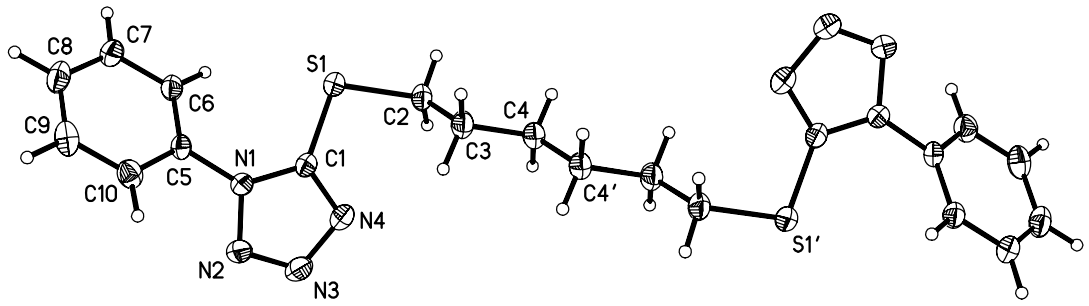

Abstract

$C_{20}H_{22}N_8S_2$, monoclinic, $P12_1/c1$ (no. 14), a = 7.481(4) Å, b = 16.759(9) Å, c = 8.621(5) Å, β = 101.555(8)°, V = 1059.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.096, T = 298 K.

Source of material

Sodium hydroxide (2 g, 0.05 mol) was added slowly to 5-mercapto-1-phenyltetrazole (8.3 g, 0.05 mol) in 20 ml of dry DMSO. The reaction mixture was stirred at 60 °C for 1 h. Then 1.4 ml (0.025 mol) of 1,6-dichlorohexane was added in portions to the solution resulting in the formation of gray suspension. The suspension was stirred for 4 h, cooled to room temperature and filtered. The solvent was removed under reduced pressure. The product was recrystallized in DMSO. Then the white crystal was obtained by evaporating the solution at the room temperature (m.p. 127-129 °C).

Elemental analysis: found – C, 54.28 %; H, 4.54 %; N, 25.43 %; calc. for $C_{20}H_{22}N_8S_2$ – C, 54.77 %; H, 5.06 %; N, 25.55 %. ^{1}H NMR data are available in the CIF.

Discussion

Tetrazole (CN_4H_2) and its derivatives have been paid much attention due to their practical applications. 5-Substituted 1*H*-tetrazoles and 1,5-disubstituted tetrazoles are often used as metabolism-resistant isosteric replacements for carboxylic acids and as *cis* amide bond surrogates, respectively, in SAR-driven analogue synthesis in medicinal chemistry [1-3]. Besides, tetrazoles also receive application in agriculture, as plant growth regulators, herbicides and fungicides [4]. In spite of these extensive investigations, there are few studies on the crystal structures of bis(5-mercapto-1-phenyltetrazoles). Here we describe the crystal structure of a new derivative [1,6-bis(1-phenyl-1*H*-tetrazol-5-ylthio)hexane].

The molecule of the compound is centrosymmetric. The two inversion related tetrazole rings are parallel to each other, while the benzene and tetrazole planes form a dihedral angle of 41.08°. The C1—N4 and C1—N1 distances of 1.318(2) Å and 1.349(2) Å, respectively, agree well with the corresponding distances in 1,3-bis(1-methyl-1*H*-tetrazol-5-ylthio)propane (1.329(2) Å and 1.334(2) Å, [5]), while the C1—S1 distance of 1.732(2) Å is slightly shorter than the corresponding distance of 1.817(2) Å in [5]. Intermolecular C2–H···N2′ interactions (2.662 Å, 174.3°) are medium hydrogen bonds which link the molecules into a corrugated 2D network parallel to $(10\bar{2})$.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.46 × 0.52 × 0.58 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.77 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5240, 1862
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1328
$N(param)_{refined}$:	180
Programs:	SHELXS-97 [6], SHELXL-97 [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.582(3)	0.819(1)	1.181(3)	0.072(7)
H(2)	4*e*	0.382(3)	0.839(1)	1.190(3)	0.074(7)
H(3)	4*e*	0.554(3)	0.871(1)	0.932(2)	0.067(7)
H(4)	4*e*	0.345(3)	0.885(1)	0.913(2)	0.061(6)
H(5)	4*e*	0.618(3)	0.968(1)	1.130(2)	0.066(7)
H(6)	4*e*	0.406(3)	0.980(1)	1.124(3)	0.077(7)
H(7)	4*e*	0.291(3)	0.566(1)	1.094(2)	0.049(6)
H(8)	4*e*	0.352(3)	0.436(1)	1.037(2)	0.070(7)
H(9)	4*e*	0.204(2)	0.375(1)	0.801(2)	0.058(6)
H(10)	4*e*	–0.004(3)	0.449(1)	0.624(3)	0.070(7)
H(11)	4*e*	–0.051(2)	0.585(1)	0.675(2)	0.040(5)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.0750(2)	0.66769(9)	0.9183(2)	0.0374(8)	0.0325(9)	0.049(1)	0.0004(7)	0.0022(7)	0.0041(8)
N(2)	4*e*	–0.1008(2)	0.6949(1)	0.8841(2)	0.0395(9)	0.042(1)	0.088(1)	0.0018(8)	–0.0003(9)	0.011(1)
N(3)	4*e*	–0.0961(2)	0.7671(1)	0.9333(3)	0.048(1)	0.043(1)	0.113(2)	0.0087(8)	0.012(1)	0.012(1)
N(4)	4*e*	0.0775(2)	0.7904(1)	0.9992(2)	0.052(1)	0.034(1)	0.091(2)	0.0056(8)	0.010(1)	–0.001(1)
S(1)	4*e*	0.41579(7)	0.72188(3)	1.04944(7)	0.0443(3)	0.0318(3)	0.0845(5)	0.0003(2)	–0.0035(3)	–0.0047(3)
C(1)	4*e*	0.1811(2)	0.7278(1)	0.9886(2)	0.045(1)	0.029(1)	0.049(1)	0.0004(8)	0.0043(9)	0.0022(9)
C(2)	4*e*	0.4652(3)	0.8233(1)	1.1185(3)	0.053(1)	0.033(1)	0.064(2)	–0.006(1)	–0.005(1)	–0.005(1)
C(3)	4*e*	0.4651(3)	0.8846(1)	0.9909(3)	0.051(1)	0.036(1)	0.057(1)	–0.005(1)	0.011(1)	–0.007(1)
C(4)	4*e*	0.5017(3)	0.9677(1)	1.0609(3)	0.046(1)	0.037(1)	0.056(1)	–0.006(1)	0.008(1)	–0.006(1)
C(5)	4*e*	0.1158(2)	0.5869(1)	0.8845(2)	0.0402(9)	0.029(1)	0.046(1)	–0.0062(8)	0.0092(9)	0.0015(9)
C(6)	4*e*	0.2346(3)	0.5432(1)	0.9945(3)	0.046(1)	0.033(1)	0.047(1)	–0.0043(9)	0.004(1)	0.000(1)
C(7)	4*e*	0.2679(3)	0.4647(1)	0.9616(3)	0.052(1)	0.034(1)	0.062(2)	0.000(1)	0.010(1)	0.004(1)
C(8)	4*e*	0.1812(3)	0.4303(1)	0.8227(3)	0.066(1)	0.034(1)	0.068(2)	–0.006(1)	0.026(1)	–0.005(1)
C(9)	4*e*	0.0610(3)	0.4742(1)	0.7146(3)	0.069(1)	0.049(2)	0.049(1)	–0.019(1)	0.017(1)	–0.009(1)
C(10)	4*e*	0.0289(3)	0.5537(1)	0.7433(2)	0.052(1)	0.046(1)	0.044(1)	–0.008(1)	0.004(1)	0.005(1)

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