Crystal structure of 5,5′-[oxybis(ethylthio)]-bis(1-methyl-1*H*-tetrazole), $C_8H_{14}N_8OS_2$

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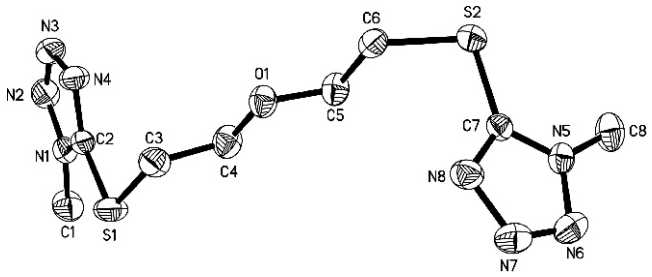

Abstract

$C_8H_{14}N_8OS_2$, monoclinic, $P12_1/c1$ (no. 14), a = 15.037(1) Å, b = 6.8075(6) Å, c = 13.834(1) Å, β = 96.693(7)°, V = 1406.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.134, T = 293 K.

Source of material

Starting materials and solvents for synthesis were purchased commercially, except for 5,5′-[oxybis(ethylthio)]bis(1-methyl-1*H*-tetrazole) (L), which was prepared according to reference [1]. A mixture of L (0.30 g, 1 mmol) and water (10 mL) was stirred and heated to 60 °C. When the mixture was cooled to room temperature, colorless crystals were obtained in 26 % yield.

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding with d(C—H) = 0.96 - 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C) for CH_2 groups and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 groups.

Discussion

Up to now, several chain-linked dithioether ligands containing *N*-heterocyclic units have been synthesized and investigated due to their diverse coordination modes and important properties of their metal complexes [2].

The asymmetric unit of the title crystal structure contains one 5,5′-[oxybis(ethylthio)]bis(1-methyl-1*H*-tetrazole) molecule. Because of the flexible nature of the linker, this molecule can adopt different conformations. Here, it adopts a *trans*-conformation with the dihedral angle between the two 5-membered rings of 82.8°. In the crystal structure, there are intermolecular hydrogen bonds between C6 and N3, C3 and N7, as well as C8 and N8 (d(C6···N3) = 3.560 Å, d(C3···N7) = 3.397 Å and d(C8···N8) = 3.362 Å, ∠C6–H6B–N3 = 142.14°, ∠C3–H3B–N7 = 132.68° and ∠C8–H8B–N8 = 131.24°), which interlink molecules to form a 3D supramolecular structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.17 × 0.19 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.85 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5979, 3218
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2234
$N(param)_{refined}$:	172
Programs:	SHELXS-97, SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	4*e*	0.7727	0.5090	0.4024	0.072
H(4B)	4*e*	0.6704	0.5142	0.4162	0.072
H(5A)	4*e*	0.7147	0.7883	0.5265	0.067
H(5B)	4*e*	0.8165	0.7515	0.5167	0.067
H(6A)	4*e*	0.8351	0.6604	0.6862	0.064
H(6B)	4*e*	0.7371	0.7354	0.6920	0.064
H(1A)	4*e*	0.4984	0.1894	0.0716	0.114
H(1B)	4*e*	0.5858	0.0708	0.1055	0.114
H(1C)	4*e*	0.5872	0.3010	0.1080	0.114
H(3A)	4*e*	0.6798	0.1903	0.4731	0.069
H(3B)	4*e*	0.7817	0.1853	0.4585	0.069
H(8A)	4*e*	1.0192	1.3974	0.5936	0.099
H(8B)	4*e*	0.9516	1.3798	0.6713	0.099
H(8C)	4*e*	0.9188	1.3435	0.5610	0.099

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}
S(2)	4*e*	0.83199(4)	1.00139(9)	0.67976(5)	0.0494(4)	0.0537(4)	0.0693(4)	0.0024(3)	0.0163(3)	–0.0148(3)
S(1)	4*e*	0.69535(4)	0.1605(1)	0.30954(5)	0.0442(4)	0.0981(6)	0.0709(4)	–0.0074(3)	0.0176(3)	–0.0348(4)
O(1)	4*e*	0.7500(1)	0.5105(2)	0.5400(1)	0.056(1)	0.053(1)	0.0493(9)	–0.0085(8)	–0.0001(7)	–0.0021(7)
N(4)	4*e*	0.5287(1)	0.1866(3)	0.3722(1)	0.047(1)	0.069(1)	0.054(1)	–0.002(1)	0.0154(9)	–0.007(1)
N(5)	4*e*	0.9895(1)	1.1204(3)	0.6234(1)	0.049(1)	0.044(1)	0.0425(9)	–0.0061(9)	–0.0006(8)	0.0012(8)
N(8)	4*e*	0.9852(1)	0.8066(3)	0.6453(1)	0.055(1)	0.047(1)	0.060(1)	0.008(1)	0.0145(9)	0.0015(9)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(6)	4*e*	1.0697(1)	1.0480(4)	0.6064(2)	0.045(1)	0.076(2)	0.063(1)	–0.004(1)	0.0089(9)	0.004(1)
N(1)	4*e*	0.5279(1)	0.1828(3)	0.2162(1)	0.056(1)	0.047(1)	0.049(1)	–0.0037(9)	0.0063(9)	–0.0068(9)
N(7)	4*e*	1.0656(1)	0.8606(3)	0.6192(2)	0.050(1)	0.072(2)	0.065(1)	0.011(1)	0.012(1)	0.001(1)
N(2)	4*e*	0.4418(1)	0.1905(3)	0.2345(2)	0.049(1)	0.065(1)	0.076(2)	0.001(1)	–0.002(1)	–0.009(1)
N(3)	4*e*	0.4437(1)	0.1934(3)	0.3276(2)	0.044(1)	0.071(1)	0.076(2)	–0.003(1)	0.013(1)	–0.008(1)
C(7)	4*e*	0.9391(1)	0.9709(3)	0.6472(1)	0.043(1)	0.041(1)	0.036(1)	0.0007(9)	0.0026(8)	–0.0048(9)
C(4)	4*e*	0.7279(2)	0.4582(4)	0.4409(2)	0.061(2)	0.065(2)	0.051(1)	–0.012(1)	–0.002(1)	–0.002(1)
C(2)	4*e*	0.5799(1)	0.1795(3)	0.3009(2)	0.043(1)	0.047(1)	0.047(1)	–0.006(1)	0.0089(9)	–0.010(1)
C(5)	4*e*	0.7671(2)	0.7134(4)	0.5519(2)	0.057(2)	0.053(2)	0.056(1)	–0.009(1)	0.000(1)	–0.000(1)
C(6)	4*e*	0.7901(2)	0.7534(4)	0.6591(2)	0.046(1)	0.060(2)	0.055(1)	–0.010(1)	0.011(1)	–0.003(1)
C(1)	4*e*	0.5519(2)	0.1863(5)	0.1168(2)	0.106(2)	0.078(2)	0.046(1)	–0.005(2)	0.015(1)	–0.005(1)
C(3)	4*e*	0.7240(2)	0.2398(4)	0.4334(2)	0.042(1)	0.067(2)	0.063(1)	0.004(1)	–0.001(1)	–0.011(1)
C(8)	4*e*	0.9679(2)	1.3279(4)	0.6113(2)	0.084(2)	0.043(1)	0.069(2)	–0.009(1)	0.000(1)	0.006(1)

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