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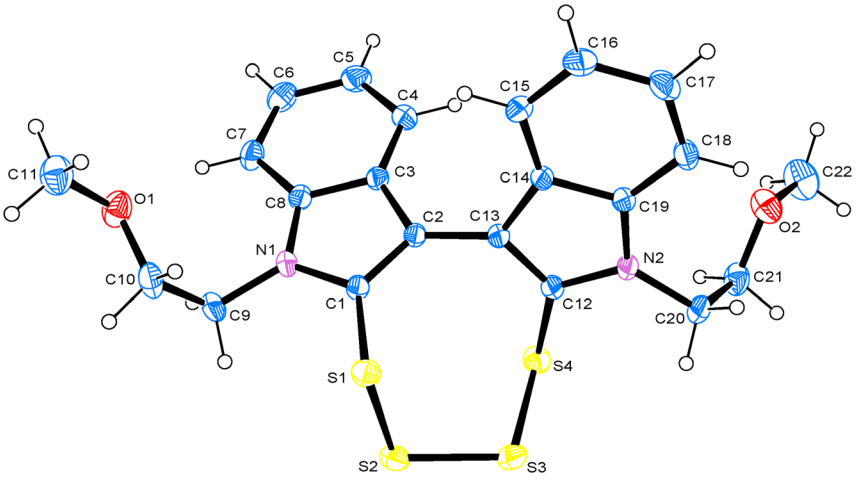
The crystal structure of 5,10-bis(2-methoxyethyl)-5,10-dihydro-[1,2,3,4]tetrathiocino[5,6-*b*:8, 7-*b*′] diindole, $C_{22}H_{22}N_2O_2S_4$



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Abstract

$C_{22}H_{22}N_2O_2S_4$, triclinic, $P\bar{1}$ (no. 2), a = 8.4865(14) Å, b = 11.756(2) Å, c = 12.863(2) Å, α = 69.693(2)°, β = 70.812(2)°, γ = 72.170(2)°, V = 1109.6(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0280, $wR_{ref}(F^2)$ = 0.0753, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.24 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.45 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5747, 3909, 0.012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3598
$N(param)_{refined}$:	274
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

All chemicals were purchased from commercial sources and used as received without further purification. A mixture of 17.5 g of 1-(2-methoxyethyl)-1*H*-indole, 9.6 g of sulfur and 60 ml of *N*,*N*-dimethylformamide was stirred under nitrogen in an oil-bath maintained at 150 °C for 6 h, and then allowed to cool slowly overnight in the oil-bath. The yellow crystals which appeared were removed and thoroughly washed with carbon disulfide. The yield of pure 5,10-bis(2-methoxyethyl)-5,10-dihydro-[1,2,3,4]tetrathiocino[5,6-*b*:8, 7-*b*′]diindole, was 11.1 g (47%). Subsequently, dissolve 1 g of the target compound in 30 ml of dichloromethane, heat reflux until the solid is completely dissolved, filtered. Finally, the title crystal was precipitated by controlling solvent volatilization.

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.30505 (5)	0.43595 (4)	0.05875 (3)	0.03909 (13)
S2	0.08586 (6)	0.36363 (4)	0.13404 (4)	0.04479 (14)
S3	−0.09841 (6)	0.49793 (4)	0.19691 (4)	0.04544 (14)
S4	−0.04806 (5)	0.49521 (4)	0.34560 (4)	0.04032 (13)
N1	0.52786 (16)	0.28226 (12)	0.19098 (11)	0.0328 (3)
N2	0.02421 (16)	0.72373 (12)	0.29725 (11)	0.0320 (3)
O1	0.88268 (16)	0.14416 (13)	0.08488 (10)	0.0517 (3)
O2	−0.14690 (17)	0.86798 (12)	0.46899 (11)	0.0512 (3)
C1	0.40182 (19)	0.39113 (14)	0.17194 (13)	0.0299 (3)
C2	0.37975 (18)	0.45613 (13)	0.24871 (13)	0.0282 (3)
C3	0.49745 (18)	0.38488 (13)	0.31816 (13)	0.0289 (3)
C4	0.5275 (2)	0.40060 (16)	0.41285 (14)	0.0381 (4)
H4	0.468197	0.470160	0.439187	0.046*
C5	0.6463 (2)	0.31108 (18)	0.46597 (16)	0.0465 (4)
H5	0.667655	0.320343	0.528705	0.056*
C6	0.7357 (2)	0.20591 (18)	0.42634 (16)	0.0494 (5)
H6	0.816184	0.147208	0.463225	0.059*
C7	0.7079 (2)	0.18704 (16)	0.33464 (16)	0.0434 (4)
H7	0.767099	0.116699	0.309526	0.052*
C8	0.58685 (19)	0.27800 (14)	0.28061 (13)	0.0317 (3)
C9	0.5777 (2)	0.18468 (15)	0.13328 (16)	0.0423 (4)
H9A	0.484888	0.190437	0.102136	0.051*
H9B	0.592905	0.104685	0.189665	0.051*
C10	0.7385 (2)	0.18906 (17)	0.03817 (15)	0.0479 (5)
H10A	0.745251	0.138493	−0.009454	0.057*
H10B	0.736857	0.273842	−0.009285	0.057*
C11	1.0371 (3)	0.1533 (2)	−0.00127 (18)	0.0589 (5)
H11A	1.050788	0.104364	−0.051121	0.088*
H11B	1.131502	0.123162	0.033640	0.088*
H11C	1.033589	0.238548	−0.044717	0.088*
C12	0.08661 (19)	0.59964 (14)	0.29602 (13)	0.0302 (3)
C13	0.26297 (19)	0.57474 (13)	0.25999 (12)	0.0283 (3)
C14	0.31247 (19)	0.68843 (13)	0.23922 (12)	0.0280 (3)
C15	0.4707(2)	0.72321 (16)	0.19720 (14)	0.0368 (4)
H15	0.571531	0.665444	0.180230	0.044*
C16	0.4739 (2)	0.84446 (17)	0.18159 (16)	0.0465 (4)
H16	0.577949	0.868785	0.153388	0.056*
C17	0.3219 (2)	0.93216 (17)	0.20771 (17)	0.0467 (4)
H17	0.328003	1.013224	0.197145	0.056*
C18	0.1652 (2)	0.90148 (15)	0.24827 (15)	0.0393 (4)
H18	0.065411	0.960070	0.265174	0.047*
C19	0.16130 (19)	0.77873 (14)	0.26325 (13)	0.0291 (3)
C20	−0.1553 (2)	0.78774 (16)	0.32461(15)	0.0398 (4)
H20A	−0.223086	0.746628	0.307457	0.048*
H20B	−0.167379	0.872246	0.275507	0.048*
C21	−0.2260 (2)	0.79165 (17)	0.44748 (16)	0.0469 (4)
H21A	−0.348532	0.824304	0.462347	0.056*
H21B	−0.204899	0.708319	0.497967	0.056*
C22	−0.2125 (3)	0.8773 (2)	0.58304 (18)	0.0694 (6)
H22A	−0.330950	0.918527	0.594549	0.104*
H22B	−0.149746	0.924073	0.596502	0.104*
H22C	−0.201041	0.795560	0.635280	0.104*

displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq} (parent C-atom).

Comment

As a special class of sulfur-rich compounds, polysulfide heterocyclic compounds show important biologically active functions and have great potential in the treatment of major diseases such as cancer and HIV [5, 6]. However, it is difficult to characterize the structure of compounds. So, the synthesis and crystal structure of the title compound are of great significance to studying the application.

Single-crystal structure analysis revealed that the title compound crystallized in the triclinic space group $P\bar{1}$. The Ortep diagram is presented in Figure 1. The bond lengths of S–S in the title molecule is 2.0335(7)–2.0817(7) Å, it is similar to reported in the literature [7–10]. The bond lengths of C1–S1 and C12–S4 in the title molecule are 1.7494(16) and 1.7483(15) Å, respectively. They are similar to reported in the literature [11–14].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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