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Structure of 2-N,N-Dimethylamino-5-hexadecyl-1,3-dithiolium-4-thiolate

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Abstract. $C_{21}H_{39}NS_3$, $M_r = 401\cdot7$, triclinic, $P\overline{1}$, $a = 5\cdot7017$ (6), $b = 9\cdot404$ (1), $c = 22\cdot104$ (4) Å, $\alpha = 91\cdot45$ (1), $\beta = 93\cdot17$ (1), $\gamma = 104\cdot17$ (1)°, $V = 1146\cdot5$ (3) Å³, Z = 2, $D_x = 1\cdot1636$ (3) g cm⁻³, λ (Mo $K\alpha$) = 0.71073 Å, $\mu = 3\cdot15$ cm⁻¹, F(000) = 220, T = 293 K, final R = 0.065 for 1183 unique observed reflections. The molecule is almost planar and the bond angles of the long aliphatic chain indicate no strain. The packing is dominated by electrostatic interactions between the heterocyclic part of the molecules, no close contacts or hydrogen bonds are observed.

Experimental. The title compound was prepared according to Jørgensen, Lerstrup & Bechgaard (1991). 2-Bromooctadecanoic acid was reacted with sodium N,N-dimethyldithiocarbamate to obtain 1carboxyheptadecyl-N,N-dimethyldithiocarbamate ester. The ester was then treated with acetic acid anhydride, triethylamine and carbon disulfide to obtain the title compound. X-ray intensity data were collected on an Enraf-Nonius CAD-4 diffractometer using ω -2 θ scan, and corrected for Lorentz, polarization and absorption effects. The structure was solved using direct methods for locating the non-H atoms. H atoms of the CH₂ groups were fixed at geometrically calculated positions and the CH₃ groups were considered as rigid bodies, all C-H distances were fixed at 1.08 Å. Least-squares

refinement based on F, minimizing the sum of $w(\Delta F)^2$, was performed with anisotropic S, C, and N atoms and isotropic H atoms with the constraints given above. The H atoms were refined with one common isotropic termperature factor for all CH₂ H atoms and the H atoms of each of the three CH_3 groups had one common isotropic temperature factor. Scattering factors and anomalous-dispersion correction parameters were taken from International Tables for X-ray Crystallography (1974, Vol. IV). Further experimental deatails are summarized in Table 1. Atomic coordinates and equivalent isotropic temperature factors of the non-H atoms are listed in Table 2, bond distances and angles are given in Table 3. The molecular geometry together with the atomic labelling used in the present work is shown in Fig. 1. and the packing within the unit cell is depicted in Fig. 2.* Programs used were SHELX76 (Sheldrick, 1976) for structure solution and refinement. DISTAN (Lundgren, 1985) for calculation of bond lengths and angles, ORTEPII (Johnson, 1976) and PLUTO (Motherwell & Clegg, 1978) for illustrations and the CONVERT utility programs (Hjorth, 1990) to facilitate interfacing of the different programs.

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^{*} Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53761 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Crystal data and experimental conditions

Crystal shape Crystal size (mm)	Prismatic 0.06 × 0.03 × 0.01	tropic	thermal para atoms wit	meters (Å ² h e.s.d.'s in	× 10 ³) of th parentheses	e non-H	
Determination of unit cell		$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$			$a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$.		
Number of reflections used	17		x	ν	z	U_{eq}	
θ range (°)	4.47-11.31	C(1)	0.527(2)	-0.123(1)	0.3736 (5)	0.039(7)	
		C(2)	0.331(2)	-0.169(1)	0.4080 (5)	0.040 (7)	
Intensity data collection		C(3)	0.435 (2)	0.115(1)	0.4191(5)	0.035 (6)	
Maximum $(\sin\theta)/\lambda$ (Å ⁻¹)	0.80	C(4)	0.643(2)	-0.215(1)	0.3365 (5)	0.048 (7)	
Range of h, k and l	0 to 9; -14 to 14; -35 to 35	C(5)	0.807 (2)	-0.137(1)	0.2901 (5)	0.052 (8)	
Standard reflections	114, 021, 104	C(6)	0.917(2)	-0.238(1)	0.2530 (5)	0.063 (8)	
Intensity variation	Negligible (less than 4%)	C(7)	0.091 (2)	-0.158(1)	0.2086 (5)	0.058 (8)	
No. of collected reflections	12575	C(8)	0.194 (2)	-0.257(1)	0.1689 (5)	0.061 (8)	
No. of unique reflections	9770	C(9)	0.373 (2)	-0.175(1)	0.1251 (5)	0.053 (7)	
R _{int}	0.014	C(10)	0.475 (2)	-0.276(1)	0.0851 (5)	0.059 (8)	
No. of observed reflections	1183	C(11)	0.659 (2)	-0.195(1)	0.0423 (5)	0.056 (8)	
Criterion for significance	$l > 3\sigma(l)$	C(12)	0.756 (2)	-0·297 (1)	0.0014 (5)	0.062 (8)	
		C(13)	0.945 (2)	-0.214 (1)	-0.0402 (5)	0.057 (8)	
Absorption correction		C(14)	0.044 (2)	-0·317 (1)	<i>−</i> 0·0799 (6)	0.061 (8)	
Linear absorption coefficient (cm ⁻¹)	3.15	C(15)	0.229 (2)	-0·236 (1)	-0.1221 (5)	0.062 (8)	
Transmission factor range	0.98-0.99	C(16)	0.329 (2)	-0.340(1)	-0.1613 (5)	0.066 (8)	
		C(17)	0.522 (3)	-0.263 (1)	-0.2030 (5)	0.073 (9)	
Structure refinement		C(18)	0.619 (3)	-0.365(1)	-0·2427 (6)	0.08(1)	
No. of refined parameters	239	C(19)	0.821 (3)	-0.293 (2)	-0.2827 (7)	0.09(1)	
Weighting scheme	$w = (\sigma_F^2 + 0.0004F^2)^{-1}$	C(20)	0.620 (2)	0.377(1)	0.4141 (6)	0.058 (8)	
R	0.065	C(21)	0.261 (2)	0.287(1)	0.4741 (6)	0.057 (8)	
wR	0.061	S(1)	0.2304 (6)	-0·0310 (3)	0.4449 (1)	0.041 (2)	
S	1.67	S(2)	0.6402 (7)	0.0700 (3)	0.3735 (2)	0.048 (2)	
Max. Δ/σ	0.023	S(3)	0.1781 (7)	-0.3468 (3)	0.4191 (2)	0.055 (2)	
Min. and max. $\Delta \rho$ (e Å ⁻³)	-0.38, 0.40	N	0.437 (2)	0.2517 (9)	0.4333 (4)	0.045 (6)	



Fig. 1. Perspective view and atomic numbering scheme of the title compound with thermal ellipsoids scaled to 50% probability. H atoms are included as spheres of arbitrary radius.



Fig. 2. Packing of the unit cell with the a axis horizontal and the caxis vertical.

Table 3. Bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Table 2. Fractional coordinates and equivalent iso-

C(1)—C(2)	1.38 (2)	C(9)—C(10)	1.52 (2)
C(1) - C(4)	1.48 (2)	C(10) - C(11)	1.53 (2)
C(1) - S(2)	1.77 (1)	C(11) - C(12)	1.52 (2)
C(2) - S(3)	1.72 (1)	C(12) - C(13)	1.53 (2)
C(2) - S(1)	1.74 (1)	C(13)—C(14)	1.52 (2)
C(3)—N	1.31 (1)	C(14)—C(15)	1.52 (2)
C(3) - S(1)	1.70 (1)	C(15)—C(16)	1.52 (2)
C(3)—S(2)	1.71 (1)	C(16)—C(17)	1.53 (2)
C(4)C(5)	1.51 (2)	C(17)—C(18)	1.51 (2)
C(5)-C(6)	1.52 (2)	C(18)—C(19)	1.53 (2)
C(6)—C(7)	1.52 (2)	NC(20)	1.46 (2)
C(7)—C(8)	1.51 (2)	N—C(21)	1.48 (2)
C(8)—C(9)	1.53 (2)		
C(2) - S(1) - C(3)	97.6 (5)	C(7)—C(8)—C(9)	113.6 (9)
C(1) - S(2) - C(3)	97.3 (5)	C(8)-C(9)-C(10)) 113-1 (9)
S(2) - C(1) - C(2)	114.4 (8)	C(9) - C(10) - C(10)	1) 113.6 (9)
S(2) - C(1) - C(4)	118.4 (8)	C(10)—C(11)—C	(12) 113.4 (9)
C(2) - C(1) - C(4)	127.1 (9)	C(11)—C(12)—C	(13) 112.9 (9)
S(1) - C(2) - S(3)	117.0 (6)	C(12)—C(13)—C	(14) 112.3 (9)
S(1) - C(2) - C(1)	116-1 (8)	C(13)—C(14)—C	(15) 113-1 (9)
S(3) - C(2) - C(1)	126.9 (8)	C(14)—C(15)—C	(16) 112.8 (9)
S(1) - C(3) - S(2)	114.6 (6)	C(15)—C(16)—C	(17) 115 (1)
S(1) - C(3) - N	123.0 (8)	C(16)—C(17)—C	(18) 115 (1)
S(2) - C(3) - N	122-4 (8)	C(17)C(18)C	(19) 116 (1)
C(1) - C(4) - C(5)	115.7 (9)	C(3) - N - C(20)	123 (1)
C(4) - C(5) - C(6)	113.0 (9)	C(3) - N - C(21)	121.0 (9)
C(5)-C(6)-C(7)	112.9 (9)	C(20)—N—C(21)	115-6 (9)
C(6) - C(7) - C(8)	114.0 (9)		

Related literature. The short C(3)-N bond of 1 31 (1) Å may be considered a double bond, which implies a positive charge at the N atom. This is in agreement with other studies of molecules with nitrogen bonded to the C atom in the 2-position of the dithiole ring, e.g. N-(4-diethylamino-5-methyl-1,3-dithiolo-2-ylidene)-p-methoxythiobenzamide (Dibo.

Stavaux, Lozac'h & Hordvik, 1985) and bis(dithiolotetrahydrodibenzodiazocine) (McKinnon, Secco & Duncan, 1987) with C-N distances of 1.29 and 1.42 Å, respectively. The corresponding negative charge is expected to be located at the S(3) atom as the length of the C(2)—S(3) bond [1.72(1) Å]indicates a single bond. The geometry of the dithiole ring is in agreement with that commonly observed, the C—S distances range from 1.70(1)-1.77(1) Å, the C(1)—C(2) double bond is 1.38 (2) Å, and the ring is planar within 0.02 Å. The aliphatic hexadecane substituent with bond angles ranging from 112.9 (9)-116.0 (11)° indicates no strain, in contrast to the dodecanoic acid substituent of 12-(tetrathiafulvenyl)-11-dodecanoic acid (Rindorf, Thorup, Lerstrup & Bechgaard, 1988). The C atoms of the alkyl chain and the ring are almost coplanar with a dihedral angle between the two least-squares planes of 10°. No close contacts or hydrogen bonds are observed, and the packing of the molecules (cf. Fig. 2) is then believed to be dominated by electrostatic interactions between the positive N atom and the negative S atom on the heterocyclic fragment of neighbouring molecules. This work is part of a research project on new materials with potential use

for Langmuir-Blodgett films exhibiting high conductivity or non-linear optical properties (Lerstrup, Jørgensen & Christensen, 1990).

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Structure of an 8a-Trifluoromethyl-8aH-cycloheptatrieno[b]pyrrole

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6-Methoxy-3-oxo-2-phenyl-8a-trifluoro-Abstract. methyl-8aH-cyclohepta-3a,5,7-trieno[b]pyrrole (3), $C_{17}H_{12}F_{3}NO_{2}, M_{r} = 319.29, \text{ triclinic}, P\bar{1},$ a =7.985 (1), b = 9.934 (2), c = 10.550 (2) Å, $\alpha =$ 65.61 (1), $\beta = 82.66$ (1), $\gamma = 72.46$ (1)°, V = 726.7 Å³, Z = 2, $D_x = 1.459$ g cm⁻³, λ (Mo K α) = 0.71069 Å, $\mu = 1.2 \text{ cm}^{-1}$, F(000) = 328, T = 238 (2) K, $R = 1.2 \text{ cm}^{-1}$ 0.057 for 2283 unique reflexions and 211 refined parameters. The seven-membered ring of the cycloheptatrieno[b]pyrrole system is noticeably folded, mostly as a result of the saturated C atom, 8a, which also bears the trifluoromethyl group. This ring fold is realized by twists around the C-C single bonds, whereas the double bonds are only twisted by, at most, $-5.7 (3)^\circ$. The pyrrole ring is essentially planar and forms a dihedral angle of $25.0 (3)^\circ$ with the phenyl substituent. The C—C and C—N bond lengths of the entire fused ring system indicate largely localized double bonds. The trifluoromethyl group shows no sign of appreciable disorder.

Experimental. A suitable single crystal of (3), $0.25 \times 0.30 \times 0.45$ mm, was grown from trichloromethane/ hexane. Syntex $P2_1$ diffractometer, graphitemonochromated Mo $K\alpha$ radiation. Cell parameters by least-squares procedures on 15 selected reflexions

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