

The authors wish to thank Mr M. Vermeire, Institut de Physique, Université de Liège, Belgium, for technical assistance in diffractometer measurements.

References

MAIN, P., GERMAIN, G. & WOOLFSON, M. M. (1984). *MULTAN*11/84. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

NASTOPOULOS, V. & WEILER, J. (1988). *Acta Cryst.* **C44**, 500–503.

ROBERTS, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for torsion angle, mean plane and libration correction calculations. Univ. of Cambridge, England.

SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

WEILER, J. (1985). *Bull. Soc. Chim. Belg.* **94**, 1101–1102.

Acta Cryst. (1991). **C47**, 1548–1550

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

BY MICHAEL HJORTH AND NIELS THORUP

Chemistry Department B, The Technical University of Denmark, DTH 301, DK-2800 Lyngby, Denmark

AND MIKKEL JØRGENSEN AND KLAUS BECHGAARD

Centre for Interdisciplinary Studies of Molecular Interactions, University of Copenhagen, Blegdamsvej 21, DK-2100 Copenhagen Ø, Denmark

(Received 17 October 1990; accepted 15 November 1990)

Abstract. C₂₁H₃₉NS₃, $M_r = 401.7$, triclinic, $P\bar{1}$, $a = 5.7017$ (6), $b = 9.404$ (1), $c = 22.104$ (4) Å, $\alpha = 91.45$ (1), $\beta = 93.17$ (1), $\gamma = 104.17$ (1)°, $V = 1146.5$ (3) Å³, $Z = 2$, $D_x = 1.1636$ (3) g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.15$ 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.

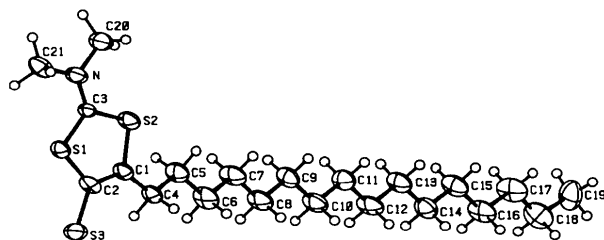
Experimental. The title compound was prepared according to Jørgensen, Lerstrup & Bechgaard (1991). 2-Bromooctadecanoic acid was reacted with sodium *N,N*-dimethyldithiocarbamate to obtain 1-carboxyheptadecyl-*N,N*-dimethyldithiocarbamate ester. The ester was then treated with acetic acid anhydride, triethylamine and carbon disulfide to obtain the title compound. X-ray intensity data were collected on an Enraf–Nonius CAD-4 diffractometer using ω - 2θ 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 temperature factor for all CH₂ H atoms and the H atoms of each of the three CH₃ 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 details 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.

* 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	Prismatic
Crystal size (mm)	0.06 × 0.03 × 0.01
Determination of unit cell	
Number of reflections used	17
θ range (°)	4.47–11.31
Intensity data collection	
Maximum (sin θ)/ λ (Å ⁻¹)	0.80
Range of h, k and l	0 to 9; -14 to 14; -35 to 35
Standard reflections	114, 021, 104
Intensity variation	Negligible (less than 4%)
No. of collected reflections	12575
No. of unique reflections	9770
R_{int}	0.014
No. of observed reflections	1183
Criterion for significance	$I > 3\sigma(I)$
Absorption correction	
Linear absorption coefficient (cm ⁻¹)	3.15
Transmission factor range	0.98–0.99
Structure refinement	
No. of refined parameters	239
Weighting scheme	$w = (\sigma_f^2 + 0.0004F^2)^{-1}$
R	0.065
wR	0.061
S	1.67
Max. Δ/σ	0.023
Min. and max. $\Delta\rho$ (e Å ⁻³)	-0.38, 0.40



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).

References

- DIBO, A., STAVAU, M., LOZAC'H, N. & HORDVIK, A. (1985). *Acta Chem. Scand. Ser. B*, **39**, 103–112.
- HJORTH, M. (1990). *CONVERT*. Utility programs for interfacing *SHELX76*, *UPALS* and *ORTEPII* programs. Technical Univ. of Denmark.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- JØRGENSEN, M., LERSTRUP, K. & BECHGAARD, K. (1991). *J. Org. Chem.* Accepted.
- LERSTRUP, K., JØRGENSEN, M. & CHRISTENSEN, J. (1990). Proc. Int. Conf. on Synth. Met., Tübingen. 2–7 September, 1990. *Synth. Met.* In the press.
- LUNDGREN, J.-O. (1985). *Crystallographic Computer Programs*. Report UUIC-B13-4-06D. Univ. of Uppsala, Sweden.
- MCKINNON, D. M., SECCO, A. S. & DUNCAN, K. A. (1987). *Can. J. Chem.* **65**, 1247–1253.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- RINDORF, G., THORUP, N., LERSTRUP, K. & BECHGAARD, K. (1989). *Synth. Met.* **30**, 391–394.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1991). **C47**, 1550–1552

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

BY GERHARD MÜLLER AND JOACHIM LACHMANN

Fakultät für Chemie, Universität Konstanz, Postfach 5560, D-7750 Konstanz 1, Germany

AND KLAUS BURGER AND KLAUS GEITH

Organisch-Chemisches Institut der Technischen Universität München, Lichtenbergstr. 4, D-8046 Garching, Germany

(Received 13 October 1990; accepted 2 January 1991)

Abstract. 6-Methoxy-3-oxo-2-phenyl-8a-trifluoromethyl-8a*H*-cyclohepta-3a,5,7-trieno[*b*]pyrrole (3), C₁₇H₁₂F₃NO₂, *M_r* = 319.29, triclinic, *P* $\bar{1}$, *a* = 7.985 (1), *b* = 9.934 (2), *c* = 10.550 (2) Å, α = 65.61 (1), β = 82.66 (1), γ = 72.46 (1)°, *V* = 726.7 Å³, *Z* = 2, *D_x* = 1.459 g cm⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71069 Å, μ = 1.2 cm⁻¹, *F*(000) = 328, *T* = 238 (2) K, *R* = 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)°. The pyrrole ring is essentially planar and forms a dihedral angle of 25.0 (3)° 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 × 0.30 × 0.45 mm, was grown from trichloromethane/hexane. Syntex *P*2₁ diffractometer, graphite-monochromated Mo *K*α radiation. Cell parameters by least-squares procedures on 15 selected reflexions