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# Structure of 5,7,12,14-Tetrathiapentacene*

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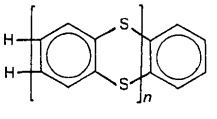
**Abstract.** $C_{18}H_{10}S_4$, $M_r = 354{\cdot}0$, monoclinic, $C2/c$, $a = 21{\cdot}039\ (2)$, $b = 10{\cdot}0272\ (7)$, $c = 7{\cdot}4082\ (8)$ Å, $\beta = 96{\cdot}770\ (7)°$, $V = 1552{\cdot}0\ (2)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}517$, $D_m = 1{\cdot}51$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 5{\cdot}84$ cm$^{-1}$, $F(000) = 728$, $T = 293$ K, $R = 0{\cdot}035$ for 1598 observed reflections. The centrosymmetric polycyclic molecule has a general chair-shaped conformation folded along the S···S lines by 131·5 (2)°. The terminal phenylene rings and the central tetrasubstituted benzene ring are approximately planar. The heterocycles have boat conformations, with the two S atoms bent out of the plane of the remaining four atoms by 0·651 (1) and 0·665 (1) Å respectively.

**Introduction.** A recent intense development in the field of conducting polymeric complexes based on poly-1,4-phenylenesulfide aroused interest in the structure of polyphenylenesulfides with two bridging S atoms between benzene rings (Sergeev, Nedel'kin & Arnautov, 1985). The structure of thianthrene (I, $n = 1$), the first representative of a ladder-like polythianthrene homologous series, was reported earlier (Rowe & Post, 1958). In this paper we discuss the results of an X-ray study of the model oligomer, *viz* tetrathiapentacene (II, $n = 2$), isolated directly from the products of homopolycondensation of diphenyl sulfide (Sergeev, Nedel'kin, Sizoi, Nekrasov & Ciryapkin, 1984; Sergeev, Shitikov & Nedel'kin, 1977).




**Experimental.** Density measured by flotation in $C_2H_2Br_4/CCl_4$. Pale-yellow single crystal 0·3 × 0·2 × 0·2 mm used for measurement of unit-cell parameters (12 reflections with $18 \le 2\theta \le 20°$) and intensities of 1842 reflections ($-28 \le h \le 28$, $0 \le k \le 15$, $0 \le l \le 13$) on a four-circle automatic Hilger & Watts diffractometer (Mo $K\alpha$, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{max} = 60°$). No significant variation in intensities of two standard reflections measured after every 100 reflections. Absorption ignored. Structure solved by direct methods (*MULTAN*; Germain, Main & Woolfson, 1971), revealing all non-H atoms and refined by full-matrix least squares with anisotropic thermal parameters for non-H atoms using 1598 independent reflections, excluding 244 reflections with $I \le 2\sigma(I)$ and minimizing $\sum w(|F_o| - |F_c|)^2$; $w = [\sigma^2(F_o) + 0{\cdot}03F_o^2]^{-1}$, 119 refined parameters. Scattering factors and anomalous-scattering corrections from *International Tables for X-ray Crystallography* (1974). H atoms located by a difference synthesis and refined isotropically. Final $R = 0{\cdot}035$, $wR = 0{\cdot}033$, $S = 2{\cdot}98$, max. shift/$\sigma = 0{\cdot}5$, final electron-density fluctuations ±0·4 e Å$^{-3}$. All calculations carried out with an Eclipse S(200) computer using *INEXTL* programs (Gerr, Yanovsky & Struchkov, 1983).

Table 1. *Atomic coordinates* ($\times 10^5$ *for* S *atoms*, $\times 10^4$ *for* C *atoms*) *and equivalent isotropic thermal parameters of non-*H *atoms*

$B_{eq} = \frac{1}{3}\sum_i\sum_j B_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j)$.

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| S(1) | 2655 (3) | 30941 (6) | −385 (9) | 4·0 (1) |
| S(2) | 13875 (3) | 9937 (6) | −5592 (8) | 3·8 (1) |
| C(1) | 1017 (1) | 3202 (2) | 1274 (3) | 3·3 (1) |
| C(2) | 1134 (1) | 4247 (2) | 2506 (4) | 4·1 (1) |
| C(3) | 1737 (1) | 4415 (2) | 3433 (3) | 4·4 (1) |
| C(4) | 2223 (1) | 3534 (3) | 3204 (3) | 4·3 (1) |
| C(5) | 2105 (1) | 2458 (2) | 2037 (3) | 3·8 (1) |
| C(6) | 1505 (1) | 2302 (2) | 1052 (3) | 3·2 (1) |
| C(7) | 614 (1) | 448 (2) | −196 (3) | 2·9 (1) |
| C(8) | 125 (1) | 1359 (2) | 11 (3) | 3·0 (1) |
| C(9) | 484 (1) | −908 (2) | −204 (3) | 3·1 (1) |

**Discussion.** Atomic coordinates are given in Table 1, bond lengths, bond angles and the general conformation of the molecule are given in Fig. 1.†

* 1,4-Benzodithiino[2,3-*b*]thianthrene.

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42713 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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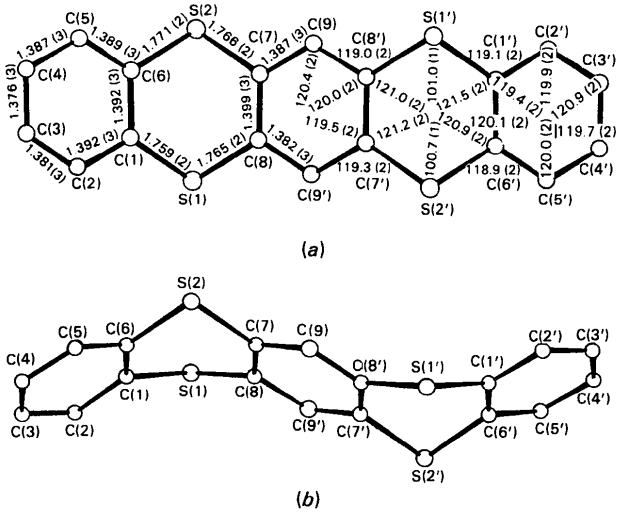


Fig. 1. The structure of molecule (II). (*a*) Bond lengths (Å) and angles (°). (*b*) General conformation of the molecule.

The pentacyclic centrosymmetric molecule (II) has a general chair-shaped conformation, previously suggested on the basis of spectral data (Ziegler & Rode, 1976). There are three approximately planar moieties in (II): *A* and *A'*, *i.e.* terminal phenylene rings with bonded S atoms [atomic deviations from the *A* and *A'* mean planes not exceeding 0·053 (2) Å], and *B*, *i.e.* a central tetrasubstituted benzene ring with bonded S atoms [atomic deviations from the mean plane not exceeding 0·036 (2) Å]. The dihedral angle between the *A* and *B* planes is 131·5 (2)°. The S(1) and S(2) atoms are displaced by 0·129 (1) and 0·101 (1) Å out of C(1)···C(6), and 0·050 (1) and 0·085 (1) Å out of C(7)···C(8) benzene planes, respectively. The heterocycles have boat conformations, the S(1) and S(2) atoms [and also S(1') and S(2')] bending out of the plane of the remaining four atoms of the ring by 0·651 (1) and 0·665 (1) Å respectively. The heterocycles in (I) and 2,7-dimethylthianthrene (III) (Weakley, 1982) have the same conformation. The folding of the heterocycles along the S···S line in (II) is 128·4 (1)° and coincides with that found in (I) (128·1°) and (III) (130·2°).

Some asymmetry in the distribution of the S–C distances in (II) is unclear, the observed values of these distances [1·759–1·771 (2) Å] agreeing with values found in (I) [1·766–1·778 (10) Å], (III) [1·760–1·774 (7) Å] and with the standard value of the ordinary S–C($sp^2$) bond length of 1·77 Å (Argay, Kálmán, Nahlovski & Ribár, 1975).

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# Structure of *N*-Acetyl-L-prolyl-L-phenylalanyl-L-leucine Monohydrate


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**Abstract.** $C_{22}H_{31}N_3O_5.H_2O$, $M_r = 435·5$. The peptide, (Leu$^9$)-angiotensinogen(7–9), crystallizes as a water solvate in the monoclinic space group $P2_1$ with $a = 6·572$ (2), $b = 21·823$ (6), $c = 8·512$ (3) Å, $\beta = 100·49$ (3)°, $V = 1200·4$ Å$^3$, $Z = 2$, $D_x = 1·205$ g cm$^{-3}$, Cu $K\bar{\alpha}$, $\lambda = 1·5418$ Å, $\mu = 7·5$ cm$^{-1}$, $F(000) = 468$, $T = 293$ K, $R = 0·057$ for 565 observed reflections with $I > 2\sigma(I)$ and $2\theta < 120°$. The proline ring has a type *A* conformation. The phenylalanine and leucine side chains are in the most commonly occurring conformations. The basic conformation of the peptide is a type I $\beta$-turn with Pro-Phe at the corner and an


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