

Fig. 1. Perspective view of (I) drawn by *PLUTO* (Motherwell & Clegg, 1978).

The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* A perspective view of the molecule with atom labelling is presented in Fig. 1.

The other crystal data are as follows. For (Ia): monoclinic, $C2$, $a = 61.384$ (11), $b = 10.913$ (2), $c = 9.972$ (2) Å, $\beta = 93.94$ (2)°, $V = 6664$ (2) Å³, $Z = 4$; for (II): orthorhombic, $P2_12_12_1$, $a = 11.179$ (6), $b = 53.567$ (7), $c = 9.646$ (1) Å, $V = 5776$ (3) Å³, $Z = 4$.

Related literature. Concanamycin A has also been produced from *Streptomyces diastatochromogenes* S45 by Kinashi, Someno & Sakaguchi (1980). An antifungal antibiotic A661-I, isolated by Shoji,

Wakisaka, Mayama & Watanabe (1974), is a mixture (1:1) of concanamycins A and B. The crystal structure of concanamycin A diacetate was analyzed because suitable crystals of concanamycin A had not been obtained (Westley, Liu, Sello, Evans, Troupe, Blount, Chiu, Todaro & Miller, 1984).

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* Lists of H-atom coordinates, anisotropic temperature factors for the non-H atoms, bond lengths, bond angles and structure factors for (I) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54957 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0564]

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The Structure of a Trisubstituted 3H-1,2-Dithiole

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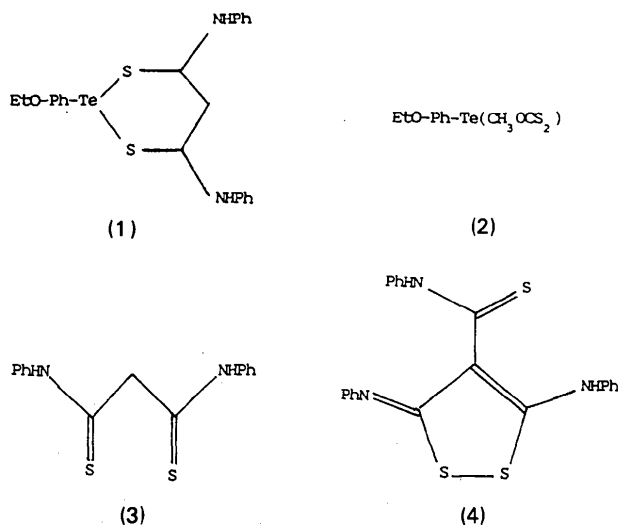
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Abstract. *N*-Phenyl-5-phenylamino-3-phenylimino-3H-1,2-dithiole-4-carbothioamide, $C_{22}H_{17}N_3S_3$, $M_r = 419.59$, monoclinic, $P2_1/n$, $a = 12.8198$ (9), $b = 6.9843$ (8), $c = 23.0717$ (13) Å, $\beta = 102.981$ (5)°, $V = 2013.0$ (3) Å³, $Z = 4$, $D_x = 1.390$ Mg m⁻³, Mo $K\alpha$, λ

$= 0.71073$ Å, $\mu = 36.67$ mm⁻¹, $T = 291$ K, $R = 0.060$, $wR = 0.060$ for 2323 unique reflections with $I > 2\sigma(I)$. The phenylthiocarbamido part of the 1,2-dithiole ring is significantly conjugated and the conjugated system includes the N(1) and N(2) atoms of

two of the ring substituents. The 1,2-dithiole ring is almost planar with a χ^2 value of 18. C(1) is at the greatest distance from this plane, being 0.005 (5) Å below it. All non-H atoms, apart from the phenyl C atoms, are close to the dithiole ring plane. The S(1)—S(2) distance of 2.044 (2) Å may indicate slight π -bond character [Hordvik (1967). PhD thesis. Univ. of Bergen, Norway].

Experimental. An attempt to prepare EtOPhTeSC(NHPh)CH₂C(NHPh)S, (1), from EtOPhTeSC(S)OCH₃, (2), and PhNHC(S)CH₂C(S)NHPh, (3), gave the title compound, C₂₂H₁₇N₃S₃, (4), containing a 3,4,5-trisubstituted 3H-dithiole ring.



0.123 g EtOPhTeSP(S)(OCH₃)₂ was mixed with 0.100 g PhNHC(S)CH₂C(S)NHPh in methanol at room temperature. The mixture was allowed to stand overnight and orange prisms or plates crystallized out. Crystal size 0.16 × 0.50 × 0.61 mm. An Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Mo K α radiation was used for the determination of cell parameters and for recording intensity data. The unit cell was determined from 25 reflections with $10 < \theta < 13.1^\circ$. The variation of 3 standard reflections measured every 2 h was used for scaling intensity data. ω -scan mode was used, max. $2\theta = 60^\circ$. 2323 unique reflections of a total of 6280 had $I > 2\sigma(I)$ and were treated as observed. Range of hkl was h 0 to 20, k 0 to 12 and l -34 to 34. Corrections were carried out for Lorentz-polarization and absorption (transmission factors 0.9441–0.9817). The structure of the compound was solved by direct methods. Refinements were carried out by the full-matrix least-squares technique, the sum minimized being $\sum w(\Delta F)^2$. Anisotropic temperature factors were applied to all non-H atoms. The H atoms were placed geometrically at a fixed C—H distance of 0.95 Å and refined isotropically using the

Table 1. Fractional atomic coordinates and their e.s.d.'s

	x	y	z	B _{eq} * (Å ²)
S(1)	0.5563 (1)	0.6990 (3)	0.04961 (5)	4.21 (3)
S(2)	0.4158 (1)	0.6853 (3)	0.07748 (5)	4.00 (3)
S(3)	0.5886 (1)	0.6596 (3)	0.29164 (5)	4.66 (3)
N(1)	0.4094 (3)	0.6708 (7)	0.1900 (2)	3.56 (9)
N(2)	0.7428 (3)	0.7050 (6)	0.1263 (1)	3.42 (8)
N(3)	0.7529 (3)	0.6982 (6)	0.2409 (1)	3.03 (8)
C(1)	0.4764 (3)	0.6842 (7)	0.1534 (2)	2.82 (9)
C(2)	0.5878 (3)	0.6896 (7)	0.1709 (2)	2.49 (8)
C(3)	0.6418 (3)	0.6976 (8)	0.1217 (2)	2.81 (9)
C(4)	0.2963 (3)	0.6632 (8)	0.1754 (2)	3.3 (1)
C(5)	0.2462 (4)	0.519 (1)	0.1983 (2)	5.3 (1)
C(6)	0.1355 (4)	0.510 (1)	0.1856 (3)	7.3 (2)
C(7)	0.0762 (4)	0.649 (1)	0.1530 (2)	6.7 (2)
C(8)	0.1256 (4)	0.793 (1)	0.1305 (2)	6.1 (2)
C(9)	0.2357 (4)	0.8028 (9)	0.1421 (2)	4.5 (1)
C(10)	0.7849 (3)	0.7147 (8)	0.0736 (2)	3.6 (1)
C(11)	0.8030 (5)	0.553 (1)	0.0448 (2)	6.4 (2)
C(12)	0.8476 (5)	0.566 (1)	-0.0055 (3)	7.4 (2)
C(13)	0.8719 (4)	0.740 (1)	-0.0247 (2)	7.3 (2)
C(14)	0.8556 (5)	0.899 (1)	0.0042 (2)	7.3 (2)
C(15)	0.8117 (5)	0.888 (1)	0.0542 (2)	5.9 (2)
C(16)	0.6473 (3)	0.6859 (7)	0.2333 (2)	2.69 (9)
C(17)	0.8396 (3)	0.6935 (7)	0.2916 (2)	2.79 (9)
C(18)	0.9395 (3)	0.6762 (8)	0.2800 (2)	3.7 (1)
C(19)	1.0302 (4)	0.6762 (9)	0.3253 (2)	4.5 (1)
C(20)	1.0223 (4)	0.6857 (9)	0.3834 (2)	4.8 (1)
C(21)	0.9237 (4)	0.7003 (9)	0.3949 (2)	5.0 (1)
C(22)	0.8318 (4)	0.7068 (8)	0.3503 (2)	4.0 (1)

$$* B_{eq} = 4/3[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + abc\cos\gamma B(1,2) + accos\beta B(1,3) + bccos\alpha B(2,3)].$$

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

S(1)—S(2)	2.044 (2)	N(2)—C(10)	1.436 (6)
S(1)—C(3)	1.773 (4)	C(16)—S(3)	1.692 (4)
S(2)—C(1)	1.746 (4)	C(16)—N(3)	1.327 (5)
C(1)—C(2)	1.393 (5)	N(3)—C(17)	1.420 (4)
C(2)—C(3)	1.455 (6)		
C(1)—N(1)	1.335 (6)	Phenyl rings (means)	
C(2)—C(16)	1.467 (5)	C(4)→C(9)	1.365
C(3)—N(2)	1.275 (5)	C(10)→C(15)	1.367
N(1)—C(4)	1.412 (5)	C(17)→C(22)	1.370
S(1)—S(2)—C(1)	95.1 (2)	C(2)—C(16)—S(3)	123.6 (3)
S(2)—S(1)—C(3)	96.2 (2)	C(2)—C(16)—N(3)	114.9 (4)
S(2)—C(1)—C(2)	119.1 (3)	S(3)—C(16)—N(3)	121.4 (3)
C(1)—C(2)—C(3)	114.2 (3)	C(16)—N(3)—C(17)	133.9 (4)
S(1)—C(3)—C(2)	115.3 (3)	C(1)—N(1)—C(4)	128.5 (3)
S(1)—C(3)—N(2)	118.6 (3)	C(3)—N(2)—C(10)	119.9 (3)
C(2)—C(3)—N(2)	126.0 (3)		
C(2)—C(1)—N(1)	125.4 (3)	Phenyl rings (means)	
S(2)—C(1)—N(1)	115.4 (3)	C(4)→C(9)	120.0
C(1)—C(2)—C(16)	123.9 (4)	C(10)→C(15)	120.0
C(3)—C(2)—C(16)	122.0 (3)	C(17)→C(22)	120.0

program HYDRO. In the last cycle, 304 parameters were refined as the temperature factors of the H atoms were kept constant. $R = 0.060$, $wR = 0.060$, $w^{-1} = \sigma^2(F) = \sigma^2(I)/4LpI$, $\sigma^2(I) = \sigma^2(I)_{\text{count}} + (0.03I)^2$, $S = 1.698$. Refinements were stopped when max. Δ/σ was equal to 0.01. From a difference Fourier map, max. ρ was 0.389 and min. $\rho = -0.464 \text{ e \AA}^{-3}$. Atomic scattering factors were taken from the CAD-4 program. All computer programs used belonged to the Enraf-Nonius (1987) *Structure Determination Package*.

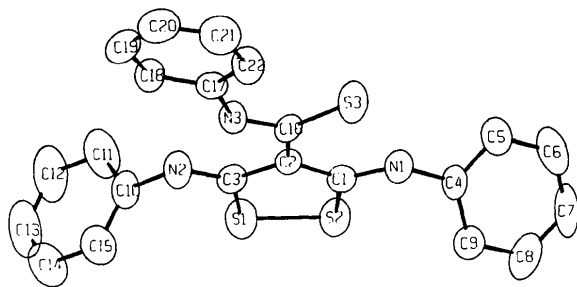


Fig. 1. ORTEP drawing of $C_{22}H_{17}N_3S_3$.

The bond lengths and angles in the molecules are calculated from the coordinates in Table 1* and listed in Table 2, together with the calculated standard deviations. An ORTEP (Johnson, 1965) drawing of the molecule is shown in Fig. 1.

Related literature. The 3*H*-1,2-dithiole ring in the present molecule can be compared with the corresponding ring in $C_{13}H_{15}NO_3S_3$ (Schaumann, Bäuch, Sieveking & Adiwidjaja, 1982). The S—S and the

* 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 54979 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0516]

S—C bond lengths are very similar in the two structures, although two C—C bonds are significantly different. This is probably related to the greater delocalization outside the ring in the present structure. These two neutral structures can also be compared with the cationic rings in 4-phenyl-1,2-dithiolium halides, 4-Ph- $C_2S_2^+X^-$; $X = Cl$ (Hordvik & Sletten, 1966), $X = Br$ (Hordvik & Baxter, 1969), $X = SCN$ (Hordvik & Kjøge, 1969), $X = Cl.H_2O$ (Grundtvig & Hordvik, 1971). The bond distances inside these rings are all shorter than the corresponding bond distances in the neutral rings, while the angles are very similar. All four rings of (4) are planar.

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Structure of Cyclohexanespiro-2'-(1',3'-oxathiolan-5'-one 3'-oxide)

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Abstract. $C_8H_{12}O_3S$, $M_r = 188.25$, orthorhombic, $Pcab$, $a = 24.547(6)$, $b = 10.242(3)$, $c = 7.185(3)$ Å,

$V = 1806(1)$ Å³, $Z = 8$, $D_x = 1.384$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 28.719$ cm⁻¹, $F(000) = 800$, room temperature, $R = 0.040$ for 829 observed reflections having $I \geq 3\sigma(I)$. The solid-state

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