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 $3H$-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

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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 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, $4\text{-Ph-}C_2S_2^+ . X^-$; $X = \text{Cl}$ (Hordvik & Sletten, 1966), $X = \text{Br}$ (Hordvik & Baxter, 1969), $X = \text{SCN}$ (Hordvik & Kjøge, 1969), $X = \text{Cl.H}_2O$ (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) Å^3, $Z = 8$, $D_x = 1.384$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ 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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conformation of the present compound has been unambiguously established in order to provide the first structure characterization of an S-oxide derivative of a 1,3-oxathiolan-5-one ring as well as a better understanding of its ^1H NMR spectrum. Although the heterocyclic ring is far from being planar, the double S=O bond [1.495 (3) Å] can be considered almost perpendicular to the mean plane ring. The S atom is involved in two quite different S—C distances, 1.855 (5) and 1.784 (5) Å. The anisotropic influence of the S=O bond can explain the magnetic non-equivalence of the two H atoms attached to the heterocyclic methylene C atom ($J = 17.7$ Hz).

Experimental. The title compound was prepared according to the procedure described by Pedersen (1969) by oxidization of cyclohexanespiro-2'-(1',3'-oxathiolan-5'-one) (Barton & Willis, 1972) with hydrogen peroxide at 273 K. Suitable white crystals of the main product of the above reaction were obtained after recrystallization from an n-hexane/ether/acetic acid (10:10:1) solution. ^1H NMR (CDCl₃) δ: 1.4–2.2 (10H, m), 3.6 (1H, d, $J =$ 17.7 Hz), 3.8 (1H, d, $J = 17.7$ Hz). A single crystal, whose dimensions were $0.25 \times 0.22 \times 0.22$ mm, was mounted on a Philips PW 1100 automatic diffractometer. The cell parameters were determined by least-squares analysis of 20 carefully centered reflections. Data collection was carried out at room temperature, using the ω–2θ scan technique within $2\theta \leq$ 110° ($0 \leq h \leq 26$, $0 \leq k \leq 10$, $0 \leq l \leq 7$); scan width $(0.7 + 0.15\tan\theta)°$; scan speed $0.12°$ s^{-1}. Stationary background measurements were taken before and after each scan for a time equal to half the scan time. Three standard reflections, monitored every 120 readings, showed no systematic trend. 1368 reflections were measured of which 829 were considered observed with $I \geq 3\sigma(I)$. After correction for background, the intensities were assigned a standard deviation calculated with an instability K factor of 0.03 (Corfield, Doedens & Ibers, 1967). The intensities were corrected for Lorentz–polarization effects and for absorption effects (Walker & Stuart, 1983); maximum and minimum transmission factors 0.95 and 0.81 respectively.

All the calculations were performed on a PC HP 486/25, using *SHELX*76 (Sheldrick, 1976), *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *ORTEP*II (Johnson, 1976) programs. Atomic scattering factors for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and those for H atoms from Stewart, Davidson & Simpson (1965). An anomalous-dispersion correction was applied to F_c (Sheldrick, 1976). During the refinement, the function minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma^2(F_o)$. The struc-

Table 1. *Final positional parameters* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^3$) *for non-H atoms*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S	9568 (1)	1363 (1)	737 (2)	37 (1)
O(1)	9538 (2)	2810 (3)	988 (5)	55 (2)
O(2)	8711 (1)	753 (3)	2777 (4)	39 (2)
O(3)	9063 (2)	706 (4)	5620 (5)	65 (3)
C(1)	8861 (2)	724 (4)	806 (7)	31 (3)
C(2)	8486 (2)	1628 (5)	−224 (8)	45 (4)
C(3)	7910 (3)	1088 (7)	−290 (10)	65 (5)
C(4)	7896 (3)	−310 (7)	−1044 (10)	68 (5)
C(5)	8270 (2)	−1188 (6)	78 (9)	56 (4)
C(6)	8850 (2)	−670 (5)	101 (8)	38 (3)
C(7)	9135 (2)	718 (4)	3984 (8)	42 (3)
C(8)	9671 (2)	700 (5)	3006 (7)	39 (3)

Table 2. *Bond distances* (Å) *and angles* (°)

S—O(1)	1.495 (3)	C(1)—C(6)	1.515 (7)
S—C(1)	1.855 (5)	C(2)—C(3)	1.520 (8)
S—C(8)	1.784 (5)	C(3)—C(4)	1.532 (10)
O(2)—C(1)	1.463 (6)	C(4)—C(5)	1.517 (10)
O(2)—C(7)	1.355 (6)	C(5)—C(6)	1.520 (8)
O(3)—C(7)	1.189 (7)	C(7)—C(8)	1.490 (8)
C(1)—C(2)	1.500 (7)		

O(1)—S—C(1)	107.5 (2)	C(1)—C(2)—C(3)	111.2 (5)
O(1)—S—C(8)	105.9 (2)	C(2)—C(3)—C(4)	111.8 (5)
C(1)—S—C(8)	88.5 (2)	C(3)—C(4)—C(5)	110.7 (6)
C(1)—O(2)—C(7)	115.2 (4)	C(4)—C(5)—C(6)	111.4 (5)
S—C(1)—O(2)	104.7 (3)	C(5)—C(6)—C(1)	110.5 (4)
S—C(1)—C(2)	110.0 (3)	O(2)—C(7)—O(3)	121.3 (5)
S—C(1)—C(6)	109.9 (3)	O(2)—C(7)—C(8)	112.1 (4)
O(2)—C(1)—C(2)	108.1 (4)	O(3)—C(7)—C(8)	126.7 (5)
O(2)—C(1)—C(6)	109.8 (4)	S—C(8)—C(7)	107.5 (4)
C(2)—C(1)—C(6)	113.9 (4)		

ture was solved by direct methods and was refined by full-matrix least squares (on F), by assigning anisotropic displacement parameters to non-H atoms and isotropic ones to the H atoms. All the H atoms were detected on a difference Fourier map, calculated at R = 0.07, and successfully refined. 157 parameters were refined. The reliability factors were $R = wR =$ 0.040; $S = 1.27$; $\Delta/\sigma_{max} = 0.07$; $\Delta\rho_{max} = 0.2$, $\Delta\rho_{min} =$ -0.2 e Å^{-3}. Table 1* reports final parameters while Table 2 gives selected bond distances and angles. A perspective view of the molecule is shown in Fig. 1.

Related literature. The structure of the title compound reveals that the 1',3'-oxathiolan-5'-one S-oxide is far from being planar, the total puckering amplitude (Cremer & Pople, 1975) being 0.391 (8) Å. Its conformation is an envelope with a local pseudo-mirror running along S and the midpoint of the

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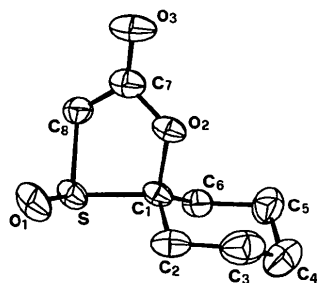
Fig. 1. Perspective view of the title molecule (*ORTEP*II; Johnson, 1976) with 30% probability ellipsoids.

O(2)—C(7) bond [$D_s(S)$ = 0.0081 (21) (Nardelli, 1983)]. Such a conformation is quite similar to that reported by Uang, Liu & Wang (1990) for an oxathiolanone bornane derivative and is also in agreement with the structure suggested for several oxathiolanones by Farines (1973) and Pihlaja, Nikkila, Neuvonen & Keskinen (1976) from NMR data. The two quite different S—C distances observed in this ring can be justified taking into account the different values between the angles S—C(8)—C(7) [107.5 (4)°] and S—C(1)—O(2) [104.7 (3)°]. Therefore, the hybridization (and hence the overlapping ability and electronegativity) of both C(8) and C(1) atoms will vary with the internal angle and there is no single rule for predicting the results. Finally, the ^{1}H NMR spectra for this compound clearly show the anisotropic influence of the S—O(1) bond on the two protons bonded to the C(8) atom, which are each located at different sides of the ring.

Obviously, the position of the S=O(1) bond, almost perpendicular to this ring, destroys their magnetic equivalence, so they then appear as an *AB* spin system (J = 17.7 Hz).

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Structure of Tris(phenylthio)phosphine Sulfide

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Abstract. $C_{18}H_{15}PS_4$, M_r = 390.5, monoclinic, $P2_1/n$, a = 10.1406 (5), b = 10.1948 (6), c = 18.870 (1) Å, β = 97.253 (2)°, V = 1935.2 (3) Å^3, Z = 4, D_x = 1.34 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å (graphite monochromator), μ = 5.49 cm^{-1}, $F(000)$ = 808, T = 297 K, R = 0.045 for 2507 observed reflections with I > 3$\sigma(I)$. Distances: P=S 1.899 (1), P—S (av.) 2.102 (2) Å; angles (av.): S=P—S 117.1 (3), S—P—S 109.9 (2)°. These are the first crystallographic data for the SP(SCR)$_3$ unit.

Experimental. SPCl$_3$ (Aldrich) was slowly added to NaSC$_6$H$_5$ (Shibao, Keder & Eckert, 1990) in thiophenol under an N$_2$ atmosphere at 295 K and allowed to react. The mixture was filtered and colorless crystals (plates) formed by slow solvent evaporation at room temperature. X-ray measurements were performed on a crystal 0.58 × 0.71 × 0.16 mm using a Huber (Crystal-Logic automated) four-circle diffractometer with $\theta/2\theta$ scan mode to a maximum 2θ of 50° at a scan speed of 6° min^{-1}. Lattice