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, $\beta = 97.253$ (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), $\mu = 5.49$ 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(SC*R*)$_3$ 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

parameters were determined from 35 reflections in the range $6.0 < 2\theta < 23.1°$. Analytical absorption correction; maximum/minimum transmission factors were 0.887/0.711 (Busing & Levy, 1957). Maximum $\sin\theta/\lambda$ of 0.595 Å^{-1}; h, k, l range, 0–12, 0–12, −22–22. Three standard reflections were measured after every 97 reflections and showed no significant variation in intensity $[(I_{max} − I_{min})/I_{av} = 0.027]$. 3823 reflections were measured, 3606 were unique ($R_{int} = 2.3\%$) and 1099 were unobserved with $I < 3\sigma(I)$. The direct methods program SHELXS86 (Sheldrick, 1985) was used in solving the structure; P and S atoms located; C atoms were located by full-matrix least-squares refinement and difference Fourier syntheses; P, S and C positions and anisotropic thermal parameters were refined; $\Sigma w||F_o| − |F_c||^2$ minimized where $w = 1/[\sigma(F_o)]^2$. H-atom positions were calculated (C—H bond length 1.0 Å) and included as fixed contributors with isotropic thermal parameters fixed to 5.0 Å^2. 208 parameters refined; $R = 0.045$, $wR = 0.062$, $S = 1.93$; $(\Delta/\sigma)_{max}$ 0.012; $\Delta\rho_{max}/_{min} = 0.46/ −0.44$ e Å^{-3}. Scattering factors were taken from International Tables for X-ray Crystallography (1974, Vol. IV). The UCLA Crystallographic Program Package (Strouse, 1985) was used throughout.
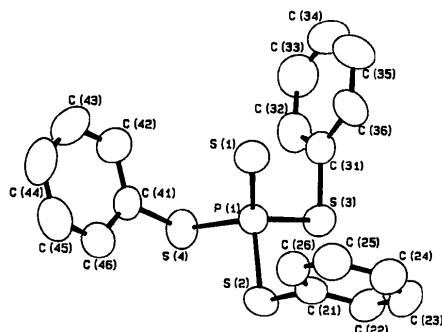

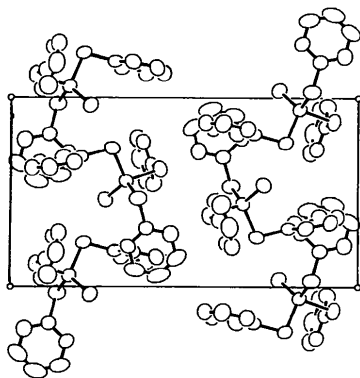Fig. 1. ORTEPII (Johnson, 1976) drawing (50% probability ellipsoids) of SP(SC$_6$H$_5$)$_3$.

Fig. 2. Packing diagram (a axis towards viewer, c axis horizontal).

Table 1. Atomic coordinates and equivalent isotropic temperature factors (Å^2 × 10^4) with e.s.d.'s in parentheses

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
P(1)	0.12174 (9)	0.44007 (9)	0.33194 (5)	536 (6)
S(1)	0.2208 (1)	0.5388 (1)	0.27163 (5)	666 (7)
S(2)	0.0345 (1)	0.26403 (9)	0.29126 (5)	669 (7)
S(3)	−0.04433 (9)	0.5319 (1)	0.36620 (6)	684 (7)
S(4)	0.2246 (1)	0.3751 (1)	0.42937 (5)	697 (7)
C(21)	−0.0232 (4)	0.3086 (3)	0.2008 (2)	582 (23)
C(22)	−0.1558 (4)	0.3373 (4)	0.1818 (2)	725 (28)
C(23)	−0.2003 (4)	0.3667 (5)	0.1109 (2)	835 (32)
C(24)	−0.1140 (5)	0.3701 (4)	0.0606 (2)	773 (30)
C(25)	0.0181 (5)	0.3413 (4)	0.0801 (2)	763 (30)
C(26)	0.0654 (4)	0.3102 (4)	0.1502 (2)	696 (27)
C(31)	0.0214 (3)	0.6900 (3)	0.3895 (2)	578 (23)
C(32)	0.0639 (4)	0.7198 (5)	0.4601 (2)	769 (31)
C(33)	0.1077 (5)	0.8459 (6)	0.4778 (3)	1008 (42)
C(34)	0.1053 (5)	0.9394 (5)	0.4268 (4)	1017 (43)
C(35)	0.0649 (5)	0.9100 (4)	0.3570 (3)	906 (38)
C(36)	0.0216 (4)	0.7849 (4)	0.3375 (2)	693 (27)
C(41)	0.3860 (3)	0.3513 (4)	0.4038 (2)	572 (22)
C(42)	0.4749 (4)	0.4550 (4)	0.4108 (2)	760 (29)
C(43)	0.6019 (5)	0.4352 (6)	0.3936 (3)	948 (38)
C(44)	0.6401 (4)	0.3169 (6)	0.3702 (3)	922 (37)
C(45)	0.5504 (5)	0.2140 (5)	0.3631 (2)	851 (34)
C(46)	0.4229 (4)	0.2307 (4)	0.3801 (2)	681 (27)

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

P(1)—S(1)	1.899 (1)	S(2)—C(21)	1.793 (4)
P(1)—S(3)	2.099 (1)	S(3)—C(31)	1.778 (4)
P(1)—S(2)	2.103 (1)	S(4)—C(41)	1.780 (4)
P(1)—S(4)	2.103 (1)		
S(1)—P(1)—S(2)	117.66 (6)	S(3)—P(1)—S(4)	101.50 (6)
S(1)—P(1)—S(3)	117.02 (6)	C(21)—S(2)—P(1)	101.7 (1)
S(1)—P(1)—S(4)	116.70 (6)	C(31)—S(3)—P(1)	101.0 (1)
S(2)—P(1)—S(3)	100.16 (6)	C(41)—S(4)—P(1)	100.2 (1)
S(2)—P(1)—S(4)	100.89 (6)		

The program PLOTMD (Luo, Ammon & Gilliland, 1989) was used to modify the labels of the ORTEP drawing (Johnson, 1976) displayed in Fig. 1. Positional parameters and isotropic temperature factors are listed in Table 1; bond lengths and angles are listed in Table 2.* In Fig. 2, the crystal packing is displayed.

Related literature. The structure of (Me$_3$SnS)$_3$PS (Shihada & Weller, 1988), possessing a strain-free SP(SR)$_3$ unit, has been reported. The terminal P—S bond length in that compound [1.948 (2) Å] is substantially longer than the one found here [1.899 (1) Å].

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Structure of 7-Acetyl-2,5,9-trinitro-2,5,7,9-tetraazabicyclo[4.3.0]nonan-8-one

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Abstract. $C_7H_9N_7O_8$, $M_r = 319.2$, monoclinic, $P2_1/n$, $a = 10.615$ (1), $b = 9.115$ (2), $c = 12.948$ (2) Å, $\beta = 108.57$ (1)°, $V = 1187.6$ (3) Å^3, $Z = 4$, $D_x = 1.785$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.38$ mm^{-1}, $F(000) = 656$, $T = 295$ K, final $R = 0.039$, $wR = 0.044$ for 1327 independent observed reflections. The five-membered ring has a normal envelope conformation while the six-membered ring has adopted a twisted boat conformation. Of the three nitroamine groups one is planar and the other two are pyramidal with N—N to C—N—C angles of 0.5, 43.4 and 44.8° respectively.

Experimental. A clear colorless prism 0.15 × 0.20 × 0.26 mm data crystal was provided by Dr Clifford L. Coon of Livermore National Laboratory, California. An automated Siemens *R*3*m*/*V* diffractometer with incident beam monochromator was used for data collection. 25 centered reflections within $20.0 \le 2\theta \le 77.0°$ were used for determining lattice parameters. $[\sin(\theta)/\lambda]_{max} = 0.54$ Å^{-1}; range of hkl: $-11 \le h \le 10$, $-9 \le k \le 2$, $0 \le l \le 13$. Standards 200, 020 and 002, monitored every 97 reflections, showed random variation of 2.5% over data collection: $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]°$, ω-scan rate a function of count rate (7.0° min^{-1} minimum, 15.0° min^{-1} maximum), 1808 reflections measured, 1545 unique, $R_{int} = 1.2\%$, 1327 observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects.

The structure solution, by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL*80 (Sheldrick, 1980). $\Sigma w(|F_o| - |F_c|)^2$ was minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.000225$. Secondary-extinction parameter $p = 0.004$ (1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 236 parameters were refined: atomic coordinates for all atoms, anisotropic thermal parameters for all non-H atoms and isotropic thermal parameters for H atoms. $(\Delta/\sigma)_{max} = 0.12$, ratio of observations to parameters = 5.6:1, $R = 0.039$, $wR = 0.044$, $S = 1.39$ ($R = 0.046$ for all data). Final difference Fourier excursions were 0.18 and -0.20 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).† Atom numbering for Tables 1 (atom coordinates) and 2 (bond distances and angles) follows that shown in Fig. 1.
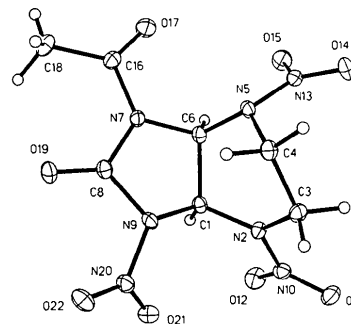

Fig. 1. A thermal ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level.