

Fig. 1. ORTEP (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 50% probability level.

Coordinates\* and equivalent isotropic thermal parameters are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.

**Related literature.** Structure of 2-ethynyl-1,3-dimethoxybenzene: Evans, Horn, Fronczek, Gan-

\* Tables of H-atom positional parameters, least-squares planes, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52329 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

dour & Watkins (1990). Structure of 1,2-bis(9-anthryl)acetylene: Becker, Skelton & White (1985). Structure of bis(*m*-chlorophenyl)acetylene: Espiritu & White (1977). Structure of diphenylacetylene: Mavridis & Moustakali-Mavridis (1977). Structure of *p*-butyl-*p*'-methoxydiphenylacetylene: Cotrait (1977).

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## Structure of 1-Chloro-3,3,5,5-tetraphenyl-1,3,5,2,4,6-selenadiphosphatriazine

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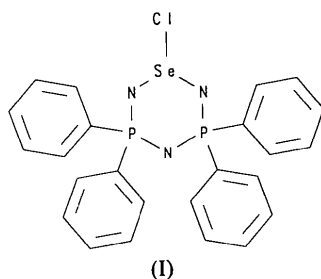
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**Abstract.** C<sub>24</sub>H<sub>20</sub>ClN<sub>3</sub>P<sub>2</sub>Se, *M<sub>r</sub>* = 526.8, triclinic, *P* $\bar{1}$ , *a* = 9.759 (1), *b* = 11.046 (4), *c* = 13.332 (2) Å,  $\alpha$  = 65.50 (2),  $\beta$  = 64.89 (1),  $\gamma$  = 74.96 (2)°, *V* = 1178.1 (4) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.49 g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha)$  = 0.71073 Å,  $\mu$  = 18.4 cm<sup>-1</sup>, *F*(000) = 532, *T* = 293 K, *R* = 0.036 for 2048 reflections with *F<sub>o</sub>*<sup>2</sup> > 3σ(*F<sub>o</sub>*<sup>2</sup>). In the SeNPNP ring the P<sub>2</sub>N<sub>3</sub> linkage is planar within 0.053 (2) Å and the Se atom is 0.499 (1) Å out of the

plane on the same side as the Cl atom. The bond distances of the Se—N—P—N sequence average 1.730 (9), 1.637 (5) and 1.584 (4) Å and the ring angles at Se and P are 111.5 and 117.8 (2)°, respectively.

**Experimental.** Title compound (I) prepared by the reaction of [ClPh<sub>2</sub>PNPPh<sub>2</sub>Cl]<sup>+</sup>.Cl<sup>-</sup> with Me<sub>3</sub>-



SiNSeNSiMe<sub>3</sub> (Bestari, Cordes, Oakley & Young, 1990). Data crystal obtained by slow cooling of an acetonitrile solution. Pale yellow crystal approximately 0.37 × 0.40 × 0.50 mm embedded in epoxy in a capillary tube because of air sensitivity. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using  $\omega$ - $2\theta$  scans of 8° min<sup>-1</sup> in  $\theta$ . Unit cell determined from least-squares analysis of angle data for 25 reflections with 18 < 2 $\theta$  < 20°. Absorption correction based on  $\psi$  scans varied from 0.94 to 1.00. Data collected to (sin $\theta$ )/ $\lambda$  of 0.55 Å<sup>-1</sup>, -10 ≤  $h$  ≤ 10, 0 ≤  $k$  ≤ 12, -14 ≤  $l$  ≤ 14. The intensities of three standard reflections (144, 233, 326) diminished 10.6% during the 12.5 h of data collection; a non-linear correction was made which ranged from 1.00 to 1.16. 3460 reflections measured, 3259 unique ( $R_{\text{int}} = 0.020$ ), 1206 reflections with  $I < 3\sigma(I)$  where  $\sigma^2(I) = \sigma_{\text{cs}}^2(I) + (0.05I)^2$ ;  $\sigma_{\text{cs}}(I)$  is standard deviation of  $I$  based on counting statistics. Solved by direct methods using MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized  $\sum w(F_o - F_c)^2$ . Five intense low-angle reflections (011, 101, 012, 120 and 112) were given zero weight in the least squares because of evidence of extinction, and an extinction parameter included in the refinement refined to 1.22 × 10<sup>-6</sup>. H atoms were

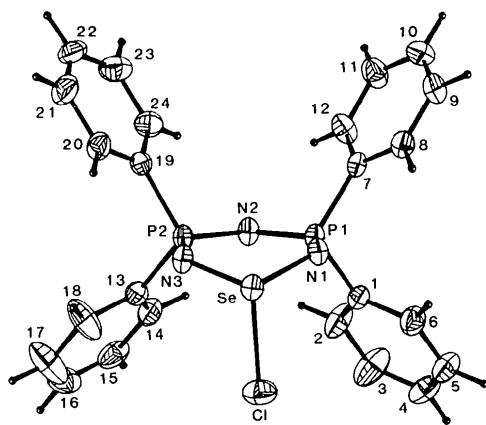


Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{\text{eq}} = (4/3) \sum_i \sum_j b_{ij} a_i \cdot a_j$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Se	0.99131 (6)	0.30493 (5)	0.05037 (4)	3.20 (1)
Cl	1.1197 (2)	0.2456 (2)	0.1800 (1)	5.70 (5)
P(1)	0.7823 (2)	0.1064 (1)	0.2164 (1)	3.01 (4)
P(2)	0.6984 (2)	0.3713 (1)	0.2139 (1)	2.97 (4)
N(1)	0.9221 (4)	0.1551 (4)	0.0903 (3)	3.3 (1)
N(2)	0.6799 (4)	0.2160 (4)	0.2720 (3)	3.4 (1)
N(3)	0.8472 (4)	0.4219 (4)	0.0935 (3)	3.4 (1)
C(1)	0.8601 (5)	-0.0212 (5)	0.3220 (4)	3.3 (1)
C(2)	0.8058 (7)	-0.0276 (6)	0.4364 (5)	5.8 (2)
C(3)	0.8678 (8)	-0.1252 (7)	0.5163 (5)	7.5 (3)
C(4)	0.9830 (7)	-0.2151 (6)	0.4810 (5)	5.5 (2)
C(5)	1.0370 (7)	-0.2111 (6)	0.3686 (5)	5.7 (2)
C(6)	0.9758 (7)	-0.1133 (6)	0.2880 (5)	4.8 (2)
C(7)	0.6685 (5)	0.0248 (5)	0.1928 (4)	3.1 (1)
C(8)	0.7277 (6)	-0.0302 (5)	0.1043 (5)	4.4 (2)
C(9)	0.6349 (7)	-0.0935 (6)	0.0902 (5)	5.4 (2)
C(10)	0.4856 (6)	-0.0995 (6)	0.1608 (5)	5.6 (2)
C(11)	0.4254 (6)	-0.0430 (6)	0.2465 (5)	5.2 (2)
C(12)	0.5171 (6)	0.0167 (5)	0.2636 (5)	4.7 (2)
C(13)	0.7034 (5)	0.4265 (5)	0.3212 (4)	3.3 (1)
C(14)	0.6562 (6)	0.3550 (5)	0.4388 (4)	4.5 (2)
C(15)	0.6621 (7)	0.4015 (6)	0.5180 (5)	5.9 (2)
C(16)	0.7187 (7)	0.5181 (6)	0.4795 (5)	6.9 (2)
C(17)	0.7650 (8)	0.5922 (7)	0.3629 (6)	8.8 (2)
C(18)	0.7576 (8)	0.5471 (6)	0.2839 (5)	7.1 (2)
C(19)	0.5369 (5)	0.4656 (5)	0.1752 (4)	3.2 (1)
C(20)	0.5523 (6)	0.5849 (5)	0.0802 (5)	4.5 (2)
C(21)	0.4260 (7)	0.6589 (6)	0.0543 (5)	5.9 (2)
C(22)	0.2852 (7)	0.6155 (6)	0.1243 (5)	6.3 (2)
C(23)	0.2689 (6)	0.4946 (7)	0.2170 (5)	5.9 (2)
C(24)	0.3939 (6)	0.4209 (6)	0.2428 (4)	4.5 (2)

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

Se—Cl	2.336 (2)	P(1)—C(7)	1.787 (6)
Se—N(1)	1.739 (4)	P(2)—N(2)	1.587 (4)
Se—N(3)	1.722 (4)	P(2)—N(3)	1.642 (4)
P(1)—N(1)	1.632 (4)	P(2)—C(13)	1.794 (6)
P(1)—N(2)	1.582 (4)	P(2)—C(19)	1.796 (6)
P(1)—C(1)	1.803 (6)		
Cl—Se—N(1)	101.5 (2)	N(2)—P(2)—N(3)	117.8 (2)
Cl—Se—N(3)	103.1 (2)	N(2)—P(2)—C(13)	108.7 (3)
N(1)—Se—N(3)	111.5 (2)	N(2)—P(2)—C(19)	111.1 (3)
N(1)—P(1)—N(2)	117.8 (2)	N(3)—P(2)—C(13)	107.3 (3)
N(1)—P(1)—C(1)	108.1 (3)	N(3)—P(2)—C(19)	105.2 (3)
N(1)—P(1)—C(7)	105.5 (3)	C(13)—P(2)—C(19)	106.1 (3)
N(2)—P(1)—C(1)	108.4 (3)	Se—N(1)—P(1)	117.9 (3)
N(2)—P(1)—C(7)	110.3 (3)	P(1)—N(2)—P(2)	125.5 (3)
C(1)—P(1)—C(7)	106.2 (3)	Se—N(3)—P(2)	119.1 (3)

constrained to idealized positions (C—H = 0.95 Å) with fixed isotropic  $B$  values of 1.2 times the  $B_{\text{eq}}$  of the attached C atoms. All non-H atoms were refined anisotropically for a total of 281 parameters.  $R = 0.036$ ,  $wR = 0.040$ ,  $S = 1.0$ , where  $I > 3\sigma(I)$  and non-Poisson  $w^{-1} = [\sigma^2(I) + (0.05I)^2]/4F^2$ . Final  $(\Delta/\sigma)_{\text{max}} < 0.2$ ,  $\Delta\rho_{\text{max}} = 0.28$  (7) and  $\Delta\rho_{\text{min}} = -0.26$  (7) e Å<sup>-3</sup> on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf-Nonius (1982)

*SDP*. \* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

**Related literature.** The structural parameters of this molecule can be compared with those found in the six-membered Se-containing rings of  $(\text{PhC})_2\text{N}_3(\text{SeCl})$  (Oakley, Reed, Cordes, Craig & Graham, 1987) and  $[(\text{Ph}_2\text{P})(\text{PhC})\text{N}_3\text{Se}]_2$  (Bestari, Cordes, Oakley & Young, 1990) as well as those of the sulfur analog  $(\text{Ph}_2\text{P})_2\text{N}_3\text{SCl}$  (Burford, Chivers, Hojo, Laidlaw, Richardson & Trsic, 1985).

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\* Tables of distances and angles in the phenyl groups, anisotropic temperature factors, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52342 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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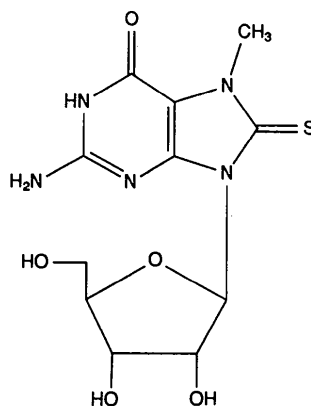
## Structure of 7-Methyl-8-thioxo-7,8-dihydroguanosine Monohydrate

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**Abstract.** 2-Amino-7-methyl-9-( $\beta$ -D-ribofuranosyl)-8(9*H*)-thioxopurin-6(1*H*)-one (1) monohydrate,  $\text{C}_{11}\text{H}_{15}\text{N}_5\text{O}_5\text{S}\cdot\text{H}_2\text{O}$ ,  $M_r = 347.34$ , orthorhombic,  $P2_12_12_1$ ,  $a = 6.9207$  (2),  $b = 10.5170$  (9),  $c = 20.315$  (2) Å,  $V = 1478.6$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.560$  g cm<sup>-3</sup>,  $\text{Cu K}\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 22.866$  cm<sup>-1</sup>,  $F(000) = 728$ ,  $T = 295$  K,  $R = 0.0286$  for 2874 reflections ( $F \geq 4\sigma_F$ ). The sugar conformation and puckering parameters are  ${}^2T_1$  (C2'-*endo*),  $P = 156.3^\circ$  and  $\tau_m = 38.7^\circ$ . The side chain is *gauche-gauche*. The glycosidic torsion angle is  $63.6$  (2) $^\circ$  corresponding to the *syn* conformation which is stabilized by an O5'—H $\cdots$ N3 intramolecular hydrogen bond [ $d(\text{H}\cdots\text{N}3) = 2.13$  (3) Å;  $\angle\text{O}5'—\text{H}\cdots\text{N}3 = 153$  (3) $^\circ$ ]. The purine ring is nearly planar [r.m.s. deviation: 0.011 (2) Å]; the dihedral angle between the pyrimidine and imidazole rings is  $0.24$  (7) $^\circ$ . All possible hydrogen donors participate in hydrogen bonding as do all possible hydrogen acceptors including the 8-thioxo group [ $d(\text{H}10\text{B}\cdots\text{S}13) = 2.79$  (2) Å;  $\angle\text{N}10—\text{H}10\text{B}\cdots\text{S}13 = 111$  (2) $^\circ$ ].

**Experimental.** The title compound (1)·H<sub>2</sub>O was synthesized by the procedure of Henry, Kini, Larson, Robins, Alaghamandan & Smee (1989). Colorless, transparent prismatic crystals grew from an ethyl acetate/methanol/acetone/water solution (18:1:1:1) following chromatography. Table 1 summarizes data collection and refinement.



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