

Fig. 1. Stereoview of phenotellurazine with thermal ellipsoids at 0.50 probability.

Villares, Jiménez-Garay, Conde & Márquez (1976), who found  $\varphi = 143$ ,  $\chi = 150$  and  $C-N-C = 122 (1)^\circ$ , and phenothiazine, the structure of which is controversial (McDowell, 1976; van de Waal & Feil, 1977). The crystal structures of 9,10-dichalcogenanthracenes have been reviewed (Meyers, Irgolic, Zingaro, Junk, Chakravorty, Dereu, French & Pappalardo, 1988) and a redetermination of the structure of phenoxathiin has been performed recently (Fitzgerald, Gallucci & Gerkin, 1991). In the structures that contain O atoms, there is a regular decrease in the angles  $\varphi$  and  $\chi$  as one goes from Te to Se to S, and a regular increase in the angle  $C-O-C$ , regularities which do not seem to be present in the analogous structures containing NH. In all of the NH structures the H atom is equatorial

to the ring, in agreement with theoretical calculations of Malrieu & Pullman (1964).

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## 1,1-Dimethyl-1-silacyclohexan-4-one Tosylhydrazone

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**Abstract.**  $C_{14}H_{22}N_2O_2SSi$ ,  $M_r = 310.5$ , triclinic,  $P\bar{1}$ ,  $a = 11.323 (2)$ ,  $b = 12.193 (2)$ ,  $c = 6.601 (5) \text{ \AA}$ ,  $\alpha = 81.00 (3)$ ,  $\beta = 104.04 (3)$ ,  $\gamma = 103.052 (12)^\circ$ ,  $V = 856 (1) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 1.204 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 2.5 \text{ cm}^{-1}$ ,  $F(000) = 332$ ,  $T = 298 \text{ K}$ ,  $R = 0.045$  for 2547 observed reflections. The silacyclohexylidene ring assumes the unusual twist-boat conformation. Calculations show the chair conformation to be more stable for both this silacyclic ring system as well as its corresponding carbocyclic analog. The packing motif, involving centrosymmetrically paired  $N-H \cdots O$  hydrogen bonds, aromati-

tic stacking and van der Waals contacts, apparently favors the less stable conformation of this ring.

**Experimental.** Crystals were grown by slow cooling of methanol solution. A colorless brick,  $0.2 \times 0.3 \times 0.5 \text{ mm}$ , was used for data collection on an Enraf-Nonius CAD-4 diffractometer with  $\text{Mo } K\alpha$  radiation and  $\omega-2\theta$  scans. 24 reflections with  $12 \leq \theta \leq 15^\circ$  were used to refine cell constants. No systematic absences were observed. No absorption correction was applied to the data collected to  $2\theta_{\text{max}} = 53^\circ$ ;  $-14 \leq h \leq 14$ ,  $-15 \leq k \leq 15$ ,  $0 \leq l \leq 8$ . Three standard reflections measured after every 3600 s of X-ray exposure showed no deterioration. Of 3561 unique reflections (no redundant data), 2547 were observed

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Table 1. *Positional and equivalent isotropic thermal parameters* ( $\text{\AA}^2$ )
$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\text{eq}}$
S	0.33362 (6)	0.48434 (5)	0.7222 (1)	3.91 (1)
Si	0.72962 (8)	0.93540 (7)	0.3468 (1)	5.64 (2)
O(1)	0.2683 (2)	0.4422 (2)	0.5269 (3)	5.03 (4)
O(2)	0.3842 (2)	0.4079 (1)	0.8925 (3)	4.87 (4)
N(1)	0.4262 (2)	0.6665 (2)	0.5206 (3)	4.33 (5)
N(2)	0.4544 (2)	0.5796 (2)	0.6811 (3)	4.15 (5)
C(2)	0.5755 (3)	0.8669 (3)	0.1934 (5)	5.70 (8)
C(3)	0.4797 (3)	0.8425 (3)	0.3260 (5)	5.61 (7)
C(4)	0.5088 (2)	0.7543 (2)	0.5114 (4)	4.19 (6)
C(5)	0.6365 (3)	0.7724 (3)	0.6559 (4)	5.16 (7)
C(6)	0.7145 (3)	0.8883 (3)	0.6248 (5)	6.42 (9)
C(7)	0.2347 (2)	0.5506 (2)	0.8086 (4)	3.72 (5)
C(8)	0.2636 (2)	0.5773 (2)	1.0149 (4)	4.49 (6)
C(9)	0.1857 (3)	0.6281 (2)	1.0787 (4)	4.87 (7)
C(10)	0.0772 (2)	0.6527 (2)	0.9457 (5)	4.75 (6)
C(11)	0.0517 (2)	0.6260 (2)	0.7390 (5)	5.01 (7)
C(12)	0.1286 (2)	0.5763 (2)	0.6712 (4)	4.34 (6)
C(13)	-0.0084 (3)	0.7080 (3)	1.0201 (6)	6.82 (9)
C(14)	0.7501 (5)	1.0908 (4)	0.3069 (8)	10.4 (1)
C(15)	0.8567 (4)	0.8835 (5)	0.2855 (9)	12.2 (2)

Table 2. *Bond distances* ( $\text{\AA}$ ), *bond angles* ( $^\circ$ ) and *selected torsion angles* ( $^\circ$ )

S—O(1)	1.431 (2)	C(3)—C(4)	1.522 (4)
S—O(2)	1.435 (2)	C(4)—C(5)	1.517 (3)
S—N(2)	1.630 (2)	C(5)—C(6)	1.500 (4)
S—C(7)	1.759 (3)	C(7)—C(8)	1.394 (4)
Si—C(2)	1.876 (3)	C(7)—C(12)	1.385 (3)
Si—C(6)	1.872 (4)	C(8)—C(9)	1.360 (5)
Si—C(14)	1.841 (4)	C(9)—C(10)	1.390 (4)
Si—C(15)	1.846 (6)	C(10)—C(11)	1.398 (4)
N(1)—N(2)	1.409 (3)	C(10)—C(13)	1.505 (5)
N(1)—C(4)	1.257 (3)	C(11)—C(12)	1.357 (5)
C(2)—C(3)	1.505 (5)		
O(1)—S—O(2)	119.3 (1)	N(1)—C(4)—C(3)	115.6 (2)
O(1)—S—N(2)	108.5 (1)	N(1)—C(4)—C(5)	125.1 (2)
O(1)—S—C(7)	107.9 (1)	C(3)—C(4)—C(5)	119.0 (2)
O(2)—S—N(2)	103.8 (1)	C(4)—C(5)—C(6)	114.9 (2)
O(2)—S—C(7)	108.7 (1)	Si—C(6)—C(5)	114.1 (2)
N(2)—S—C(7)	108.3 (1)	S—C(7)—C(8)	119.9 (2)
C(2)—Si—C(6)	102.4 (1)	S—C(7)—C(12)	119.9 (2)
C(2)—Si—C(14)	111.2 (2)	C(8)—C(7)—C(12)	120.2 (3)
C(2)—Si—C(15)	110.6 (2)	C(7)—C(8)—C(9)	118.9 (2)
C(6)—Si—C(14)	107.8 (2)	C(8)—C(9)—C(10)	122.1 (3)
C(6)—Si—C(15)	111.5 (2)	C(9)—C(10)—C(11)	117.5 (3)
C(14)—Si—C(15)	112.9 (3)	C(9)—C(10)—C(13)	121.2 (3)
N(2)—N(1)—C(4)	116.9 (2)	C(11)—C(10)—C(13)	121.3 (2)
S—N(2)—N(1)	113.9 (1)	C(10)—C(11)—C(12)	121.5 (2)
Si—C(2)—C(3)	110.1 (2)	C(7)—C(12)—C(11)	119.7 (3)
C(2)—C(3)—C(4)	111.5 (3)		
Si—C(2)—C(3)—C(4)	-65.6 (3)	C(2)—C(3)—C(4)—N(1)	-121.4 (3)
C(2)—C(3)—C(4)—C(5)	52.8 (3)	C(3)—C(4)—N(1)—N(2)	175.2 (2)
C(3)—C(4)—C(5)—C(6)	11.4 (4)	C(4)—N(1)—N(2)—S	164.9 (2)
C(4)—C(5)—C(6)—Si	-52.4 (3)	N(1)—N(2)—S—C(7)	-63.3 (2)
C(5)—C(6)—Si—C(2)	33.1 (3)	N(2)—S—C(7)—C(8)	-76.8 (2)
C(6)—Si—C(2)—C(3)	23.3 (3)		

$[I \geq 2\sigma(I)]$  with 1014 unobserved. The structure was solved by direct methods with *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement, on  $|F|$ , minimized the function  $w(|F_o| - |F_c|)^2$ , with  $w = 4F_o^2[\sigma^2(F_o^2) + (0.04F_o^2)^2]^{-1}$ . H(N2) was located on a difference map and refined with an isotropic temperature factor; all other H atoms were placed at calculated positions with fixed temperature factors, updated after each cycle of refinement but not refined; non-H atoms were refined with anisotropic thermal parameters.

186 parameters were refined in the final cycle giving  $R = 0.045$ ,  $wR = 0.067$ ,  $S = 2.38$ ;  $(\Delta/\sigma)_{\text{max}}$  in the final cycle was 6%, with an average value of 3%. The final difference map maxima were within  $\pm 0.3 \text{ e \AA}^{-3}$ , except for a peak of  $0.5 \text{ e \AA}^{-3}$  near the Si atom. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed with *SDP* (Enraf-Nonius, 1982).

Final positional parameters are given in Table 1.\* Bond distances, bond angles and selected torsion angles are listed in Table 2. Fig. 1 (Johnson, 1976) is a perspective view of the molecule, giving the numbering scheme. From Fig. 1 and the conformational angles in Table 2, it can be seen that the silacyclic ring assumes a twist-boat conformation in this crystal form, although *MMX* calculations (*PCMODEL*, 1989) show the chair conformation to be of lower energy for this ring system (*ca*  $8.4 \text{ kJ mol}^{-1}$ ). Fig. 2 (Motherwell, 1976) is a perspective view of the packing interactions down the *c* axis. Centrosymmetrically paired hydrogen bonds linking N(2) and O(2) [ $\text{N}(2)\cdots\text{O}(2)$   $2.966(4) \text{ \AA}$ ] form dimers, which then interact *via* stacking of the aromatic rings

\* 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 55772 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0202]

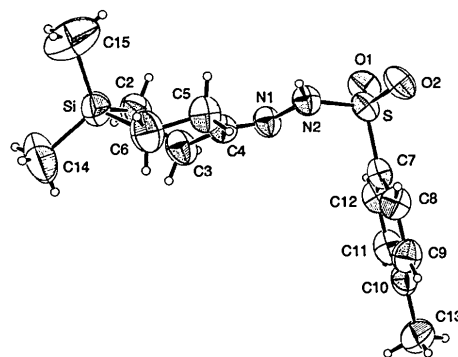


Fig. 1. Perspective view of the molecule with numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

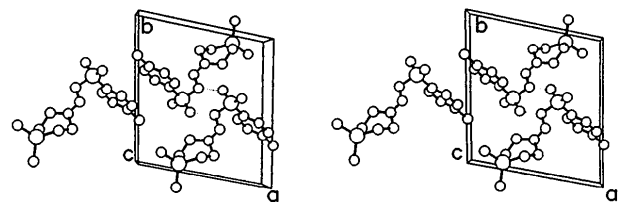


Fig. 2. Perspective view of the packing interactions down the *c* axis.

and van der Waals interactions. These packing interactions apparently favor the less stable twist-boat conformation of the silacyclohexylidene ring.

**Related literature.** The title compound was prepared in 81% yield (m.p. 421–422 K) from the reaction of 1,1-dimethyl-1-silacyclohexan-4-one (Soderquist & Negron, 1989) with tosylhydrazine in acidic ethanol. Complete spectroscopic and analytical data were obtained for the tosylhydrazone which were in complete agreement with the structural data reported herein.

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## Structure of *N,N'*-Dimethyl-4,4'-bipyridylium Dichloride Dihydrate

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**Abstract.**  $C_{12}H_{14}N_2^{2+} \cdot 2Cl^- \cdot 2H_2O$ ,  $M_r = 293$ , triclinic,  $P\bar{1}$ ,  $a = 9.696$  (3),  $b = 11.322$  (4),  $c = 7.076$  (3) Å,  $\alpha = 100.68$  (4),  $\beta = 93.40$  (3),  $\gamma = 107.04$  (4)°,  $V = 724$  (6) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.344$  Mg m<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.5418$  Å,  $\mu = 4.4$  mm<sup>-1</sup>,  $F(000) = 308$ , room temperature,  $R = 0.052$  for 2201 independent reflections. *N,N'*-dimethyl-4,4'-bipyridylium dichloride (or paraquat, viologen) like mitoguazone (an anticancer drug), affects the polyamine uptake systems, a potential target in cancer chemotherapy. The description of the three-dimensional structure of this ion should allow a better assessment of the structure–activity relationship of these bioactive molecules.

**Experimental.** Crystals of the title compound were obtained in ethanol from commercial methyl viologen dichloride hydrate (Aldrich-Chemie). Pale yellow single crystal approximately  $0.15 \times 0.30 \times$

$0.2$  mm. The unit-cell dimensions (from 25 reflections,  $13.8 < 2\theta < 26.5^\circ$ ) and reflection intensities were measured with a Philips PW1100 diffractometer, graphite-monochromated radiation, scan type 'flying step scan',  $\omega$ - $2\theta$  scan, scan range  $1.8^\circ \theta$ , scan speed  $0.02^\circ \theta s^{-1}$ ,  $\theta$  limits:  $1$ – $68^\circ$ .  $-11 \leq h \leq 11$ ,  $-13 \leq k \leq 13$ ,  $0 \leq l \leq 8$ . 2866 reflections measured, 2633 unique ( $R_{int} = 0.063$ ), three standard reflections,  $\bar{1}5\bar{1}$ ,  $\bar{3}20$  and  $1\bar{5}1$ , measured every hour (decomposition less than 3%), absorption correction by *DIFABS* (Walker & Stuart, 1983),  $T_{min} = 0.743$ ,  $T_{max} = 1.818$ , average = 1.048. Structure solved using direct methods and successive Fourier maps [*SHELXS86* and *CRYSTALS* from Sheldrick (1986) and Watkin, Carruthers & Betteridge (1985)]. H atoms found from difference series, complex atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101). All non-H atoms were given anisotropic thermal parameters,