

*International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). Graphics were generated using *SHELXTL-Plus*. The positional and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are given in Table 2.\* A drawing of the molecule with the atom-labeling scheme is shown in Fig. 1, and the packing diagram is presented in Fig. 2.

**Related literature.** The chiral tricyclic amine was prepared (Whitesell *et al.*, 1988) as a chiral auxiliary for asymmetric inductions. Materials incorporating this twofold symmetric (*C*<sub>2</sub>) amine are possible candidates for nonlinear optical applications (Chemla & Zyss, 1987). The packing of this salt (as well as the full hydrochloride salt currently being prepared) is of interest as an example of species with molecular *C*<sub>2</sub> symmetry. Conformations of similar tricyclic amine compounds are discussed by Minton, Whitesell, Mountzouris, Abboud & Davis (1990), Chen,

Whitesell, Price, Abboud & Davis (1990), and Abboud, Minton, Whitesell & Davis (1990).

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\* Lists of anisotropic displacement parameters, H-atom positional parameters, bond lengths and angles involving H atoms, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55814 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0476]

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## Structure of Phenotellurazine

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**Abstract.** Dibenzob[*b,e*]tellurazine, C<sub>12</sub>H<sub>9</sub>NTe, *M*<sub>r</sub> = 294.8, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 6.023 (2), *b* = 8.007 (3), *c* = 20.877 (9) Å, *V* = 1006.8 (7) Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.945 Mg m<sup>-3</sup>, λ(Mo Kα) = 0.71073 Å, μ = 2.915 mm<sup>-1</sup>, *F*(000) = 560, *T* = 296 K, *R* = 0.042 for 1504 reflections and 130 parameters. The structure is similar to that of phenoxatellurine and is a hetero-

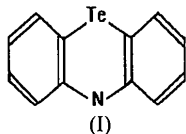
cyclic analogue of anthracene in which the two CH groups of the central ring have been replaced by Te and NH. The central ring is folded along the N–Te axis, φ = 142 (3)°, and the outer phenyl rings make an angle of χ = 151 (1)° with each other. The average bond distances are Te–C = 2.098 (8), N–C = 1.401 (9), C–C = 1.389 (3) Å with bond angles C–Te–C = 90.9 (3), C–N–C = 125.5 (6) and (average) C–C–C = 119.9 (2)°. All of these values appear to be normal. There are no abnormally short contact distances, and in particular, none that might be attributed to hydrogen bonding.

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**Experimental.** A sample of phenotellurazine (I), prepared as described by Junk & Irgolic (1989), was purified by sublimation and then recrystallized from



methylene dichloride. A pale-yellow prism of dimensions  $0.3 \times 0.3 \times 0.45$  mm was mounted on a glass fiber with epoxy cement. Preliminary examination and data collection were performed on a Siemens  $R3m/V$  X-ray diffractometer equipped with an oriented-graphite monochromator and using  $\text{Mo } K\alpha$  radiation. Unit-cell dimensions were obtained from measurements of  $2\theta$  for 25 reflections ( $2\theta_{\text{av}} = 22.4^\circ$ ) and  $\omega$  scans indicated acceptable crystal quality. Intensity data were collected with  $\theta$ - $2\theta$  scans ( $\omega$ -scan range  $0.60^\circ$  plus  $K\alpha$  separation, variable scan rate from  $2.0$  to  $29.3^\circ \text{ min}^{-1}$ ) and three standard reflections, measured every 97 reflections, showed no significant trends. Data were collected in the range  $4 < 2\theta < 50^\circ$ ,  $0 \leq h \leq 2$ ,  $0 \leq k \leq 9$ ,  $-24 \leq l \leq 24$  and  $3 \leq h \leq 7$ ,  $0 \leq k \leq 8$ ,  $0 \leq l \leq 22$ . Background corrections were made by stationary counter-stationary crystal measurements taken at the beginning and end of each scan, each for one-half of the total scan time. Lorentz, polarization and empirical absorption ( $T_{\text{max}} = 0.971$ ,  $T_{\text{min}} = 0.790$ ) corrections were applied to the data and a learnt-profile technique used to improve intensity measurements (Diamond, 1969). The intensities of 1535 reflections were measured, 31 of which were discarded because of systematic extinction. The structure was solved by direct methods and calculations were carried out with the *SHELXTL-Plus* programs (Sheldrick, 1990). Structure factors were those of Cromer & Waber (1974). Full-matrix least-squares refinement of coordinates and anisotropic temperature factors for non-H atoms was performed; the coordinates of the H atom attached to N were allowed to vary. The remaining H atoms were assigned positions  $0.96 \text{ \AA}$  from the C atoms to which they were attached. All H atoms were assigned an isotropic temperature factor  $U = 0.08 \text{ \AA}^2$ . For 130 parameters, the minimization of  $\sum w(|F_o| - |F_c|)^2$  for all 1504 data with  $w^{-1} = \sigma^2(F_o) + 0.001|F_o|^2$  gave  $R = 0.0423$ ,  $wR = 0.0482$ , and goodness of fit = 1.13 with the largest  $\Delta/\sigma = 0.003$  and  $R_{\text{int}} = 0.015$ . [For those reflections for which  $|F_o| < \sigma(F_o)$ ,  $|F_c|$  was set equal to  $\sigma(F_o)$ .] After removal of reflections for which  $I < 0$ , the largest peak on the final difference map was  $1.27 \text{ e \AA}^{-3}$  and the largest hole was  $0.63 \text{ e \AA}^{-3}$ . The present choice of enantiomer was verified in earlier calculations using  $\eta$  defined by Rogers (1981). Refined coordi-

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ )

Equivalent isotropic  $U$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	x	y	z	U
Te(1)	591 (1)	3947 (1)	8626 (1)	49 (1)
N(1)	-3855 (11)	1686 (10)	8592 (3)	48 (2)
H1N	-5171 (196)	1510 (117)	8607 (42)	80
C(1)	-310 (13)	1852 (8)	9164 (3)	37 (2)
C(2)	-2380 (12)	1098 (9)	9058 (3)	39 (2)
C(3)	-3049 (16)	-205 (9)	9455 (3)	45 (2)
C(4)	-1686 (17)	-736 (9)	9949 (3)	49 (3)
C(5)	353 (17)	6 (10)	10050 (3)	51 (3)
C(6)	1042 (13)	1193 (9)	9650 (3)	42 (2)
C(7)	-3267 (14)	2214 (8)	7975 (3)	38 (2)
C(8)	-4687 (16)	1832 (9)	7463 (3)	47 (2)
C(9)	-4156 (17)	2359 (9)	6846 (3)	49 (3)
C(10)	-2223 (17)	3238 (10)	6730 (3)	52 (3)
C(11)	-788 (16)	3581 (9)	7226 (3)	50 (2)
C(12)	-1341 (13)	3110 (9)	7849 (3)	36 (2)

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

Te(1)—C(1)	2.090 (6)	Te(1)—C(12)	2.106 (7)
N(1)—C(2)	1.400 (9)	N(1)—C(7)	1.402 (9)
N(1)—H1N	0.805 (119)	C(1)—C(2)	1.403 (10)
C(1)—C(6)	1.376 (9)	C(2)—C(3)	1.391 (10)
C(3)—C(4)	1.385 (11)	C(4)—C(5)	1.380 (14)
C(5)—C(6)	1.391 (10)	C(7)—C(8)	1.403 (11)
C(7)—C(12)	1.389 (11)	C(8)—C(9)	1.392 (10)
C(9)—C(10)	1.382 (13)	C(10)—C(11)	1.376 (12)
C(11)—C(12)	1.395 (9)		
C(1)—Te(1)—C(12)	90.9 (3)	C(2)—N(1)—C(7)	125.5 (6)
C(2)—N(1)—H1N	123 (6)	C(7)—N(1)—H1N	110 (6)
Te(1)—C(1)—C(2)	119.5 (5)	Te(1)—C(1)—C(6)	120.2 (5)
C(2)—C(1)—C(6)	120.1 (6)	N(1)—C(2)—C(1)	121.9 (6)
N(1)—C(2)—C(3)	118.8 (7)	C(1)—C(2)—C(3)	119.1 (6)
C(2)—C(3)—C(4)	120.1 (8)	C(3)—C(4)—C(5)	120.6 (7)
C(4)—C(5)—C(6)	119.5 (7)	C(1)—C(6)—C(5)	120.5 (7)
N(1)—C(7)—C(8)	118.7 (7)	N(1)—C(7)—C(12)	122.7 (6)
C(8)—C(7)—C(12)	118.6 (6)	C(7)—C(8)—C(9)	119.9 (8)
C(8)—C(9)—C(10)	120.7 (7)	C(9)—C(10)—C(11)	119.9 (7)
C(10)—C(11)—C(12)	119.8 (8)	Te(1)—C(12)—C(7)	118.7 (4)
Te(1)—C(12)—C(11)	120.0 (6)	C(7)—C(12)—C(11)	121.0 (7)

nates and equivalent isotropic temperature factors for the structure are listed in Table 1, bond distances and angles in Table 2, and Fig. 1 presents a stereoview of the structure.\*

**Related literature.** The structure of 3,7-dimethyl-10-ethylphenotellurazine has been reported by Furmanova, Sorokina, Abakarov & Sadekov (1985). It agrees in general with the present structure but differs in detail. The largest differences are in the angles of fold,  $\varphi = 133$ ,  $\chi = 138$  and  $\text{C—N—C} = 117.7 (5)^\circ$ . Molecules similar to phenotellurazine include phenoselenazine, reported most recently by

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55712 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1023]

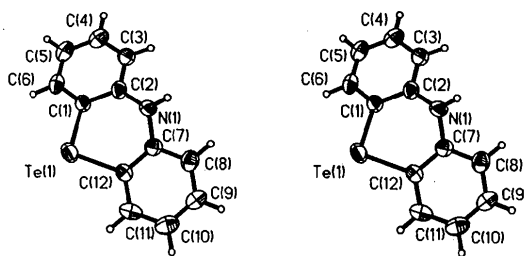


Fig. 1. Stereoview of phenotellurazine with thermal ellipsoids at 0.50 probability.

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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