interaction of the opposing hydrogens of the bipyridyl ligand distort the ligands into a bowed or twisted conformation. The equation of the chelation plane (Table 4) reveals that while the bipyridyl chelate atoms [Pd(1), N(2), C(10), C(11), N(3) and Pd(2), N(6), C(26), C(25), N(5)] are coplanar within 0·04 Å, the pyrrolidine chelating atoms [Pd(1), N(1), C(4), C(5), O(1) and Pd(2), N(4), C(19), C(20), O(4)] show relatively large deviations from planarity. Since amino-acid ligands are known to form nearly planar chelate rings (Freeman, 1967) the large deviations observed in the present case confirm the importance of the suggested non-bonded interactions involving the H atoms. The intraligand twist angles τ [based on the parameters defined by Cordes *et al.* (1982)] were found to be 2·51 (6) and 3·14 (8)° and these are nearly equal to the dihedral angles between the individual pyridine rings, 2·56 (7) and 2·96 (7)° of the respective bipyridyl ligands. This arises because the bipyridyl ring is in the twisted conformation with atoms C(6) and C(15) and atoms C(30) and C(21) on the opposite sides of the coordination planes for the two molecules (Table 3). The proline rings (*P*4 and *P*8) are inclined at angles 42·3 (3) and 139·9 (3)° to the respective planes of metal coordination (*P*1 and *P*5). The Pd(1)—Pd(2) distance is 3·89 (1) Å. The two molecules are stacked above one another along the *c* axis and linked with weak hydrogen bonds through the water molecules (Table 5).

Table 5. *Hydrogen-bond distances* (Å); *e.s.d.'s* ~0·02 Å

		Symmetry code
N(1)···O(*W*1)	2·88	*x*, *y*, *z*
O(3)···O(*W*2)	3·10	*x* − 1, *y*, *z*
O(3)···Cl(1)	3·44	*x*, *y*, *z* − 1
O(6)···O(*W*2)	2·82	*x* − 1, *y* − 1, *z*
O(*W*1)···O(*W*3)	2·85	*x* − 1, *y*, *z*
O(*W*1)···Cl(1)	3·19	*x*, *y*, *z*
O(*W*2)···Cl(2)	3·18	*x* + 1, *y* + 1, *z*
O(*W*3)···Cl(2)	3·25	*x* + 1, *y* + 1, *z* + 1
O(*W*4)···O(6)	3·34	*x* + 1, *y* + 1, *z* + 1
O(*W*4)···Cl(1)	3·27	*x*, *y*, *z*
O(*W*4)···Cl(2)	3·25	*x* + 1, *y* + 1, *z* + 1
N(4)···Cl(2)	3·25	*x*, *y*, *z*

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1-(2,2′-Bipyridyl)-2-methyl-3-(trimethylsilyl)-2,3-dicarba-1-stanna-*closo*-heptaborane(6)

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Abstract. $C_{16}H_{24}B_4N_2SiSn$, $M_r = 434{\cdot}39$, monoclinic, $P2_1/n$, $a = 10{\cdot}666\,(4)$, $b = 13{\cdot}556\,(4)$, $c = 14{\cdot}879\,(5)$ Å, $\beta = 105{\cdot}04\,(3)°$, $V = 2077{\cdot}63\,(19)$ Å^3, $Z = 4$, $D_x = 1{\cdot}39$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo}\,K\alpha) = 11{\cdot}80$ cm^{-1}, $F(000) = 872$, $T = 295$ K. Final $R = 0{\cdot}039$ for 2211 observed reflections. The molecule has a distorted pentagonal bipyramidal geometry with the Sn atom occupying an apical position and bonding exclusively to three B atoms of the carbaborane cage and two N atoms of the bipyridine rings on the opposite side. Bond distances in the cage structure are Sn—C 2·818 (6), Sn—B 2·515 (7) and 2·359 (7) and Sn—N 2·475 (4) Å; the B—Sn—N bond angles are 85·4 (2) to 126·2 (2)°. The two donor–acceptor bonds between Sn(cage) and N atoms of the 2,2′-bipyridine confirm the Lewis acidity of the stannacarbaboranes.

Introduction. Recently, it has been demonstrated that the reaction of $\text{Na}[(\text{Me}_3\text{Si})(R)\text{C}_2\text{B}_4\text{H}_5]$ with SnCl_2 in THF (tetrahydrofuran) produces an air-sensitive THF–stannacarbaborane intermediate, $(\text{C}_4\text{H}_8\text{O})_2\text{Sn}(\text{Me}_3\text{Si})(R)\text{C}_2\text{B}_4\text{H}_4$ (I) ($R = \text{Me}_3\text{Si}$, Me or H), which decomposes to give THF and the corresponding stannacarbaborane as a sublimable white solid (Hosmane, Sirmokadam & Herber, 1984; Hosmane, de Meester,

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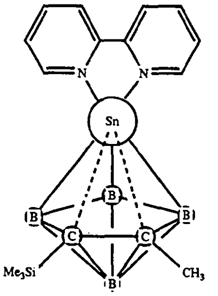
Maldar, Potts, Chu & Herber, 1986). The structures of these compounds show a pentagonal bipyramidal geometry with the Sn atom occupying an apical position (Cowley, Galow, Hosmane, Jutzi & Norman, 1984; Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986). These crystal structures provide the first unambiguous proof of how Sn^{II} is incorporated into small carbaborane clusters. Despite the presence of a lone pair of electrons on the Sn atom, the stannacarbaboranes do not react with BH_3.THF or BF_3 but react quantitatively with 2,2′-bipyridine in benzene to form the electron acceptor–donor complexes of the type $(C_{10}H_8N_2)Sn(Me_3Si)(R)C_2B_4H_4$ (II). The ^{119m}Sn Mössbauer-effect spectra of these complexes exhibit quadrupole-split doublets and clearly indicate that tin is formally in the +2 oxidation state. The structure of $(C_{10}H_8N_2)Sn(Me_3Si)_2C_2B_4H_4$ (III) was determined by single-crystal X-ray diffraction (Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986). Apparently, complexation is accompanied by a substantial elongation of the Sn—C bonds and a particular orientation of the bipyridyl group opposite the cage C atoms. It may be that this is simply due to steric effects imposed by the bulky trimethylsilyl groups bonded to each cage C atom or it could be the result of orbital effects. In order to obtain more information about the relative importance of these two effects, we have conducted a structural investigation of the title compound (IV) in which one of the cage C atoms carries a methyl group, and report herein the results.



(IV)

Experimental. The title compound (IV) was isolated in 81% yield from the crude yellow products of the reaction involving the stannacarbaborane precursor and 2,2′-bipyridine in benzene (Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986). Large well-formed rectangular orange crystals of (IV) were grown by sublimation onto a glass surface; a single crystal (0·35 × 0·25 × 0·15 mm) was coated in an epoxy resin; unit-cell parameters by least-squares fit of 15 reflections in the range $10 < 2\theta < 25°$; space group $P2_1/n$ from systematic absences ($0k0$, k odd, $h0l$, $h+l$ odd); automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode; 3000 measured reflections, 2718 independent reflections in the range $3 < 2\theta < 45°$, $R_{int} = 0{\cdot}012$; hkl range h 11→−11, k 0→14, l 0→−16; 2211 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; 3 standard reflections remeasured after every 100 reflections did not show any significant change in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic; H atoms located in difference Fourier maps, except for silyl-methyl H positions which were calculated; H atoms included as a 'fixed atom contribution' with isotropic temperature factors; $w = 1/(\sigma^2F + 0{\cdot}002856F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}039$, $wR = 0{\cdot}044$; $(\Delta/\sigma)_{max} = 0{\cdot}21$, $\Delta\rho_{(max, min)} = 0{\cdot}60, -0{\cdot}32$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N, B, Si, Sn and the real and imaginary parts of the dispersion correction for Sn and Si were those stored in *SHELX*76.

Discussion. The final atomic parameters of the non-H atoms are given in Table 1.* The identification of the atoms and the configuration of (IV) are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Compound (IV) has a distorted pentagonal bipyramidal geometry with the Sn atom occupying an apical position and is essentially η^5 bonded to both carbaborane cage and bipyridine rings. The bond lengths and selected bond angles with their standard deviations are given in Table 2. The distortion of the cage geometry involves the Sn—C distances [2·817 (5) and 2·819 (6) Å] which are comparable with those determined for $(C_{10}H_8N_2)$-$Sn(Me_3Si)_2C_2B_4H_4$ (III) in which both C(cage) atoms are bonded to bulky trimethylsilyl groups (Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986). In both (III) and (IV), the Sn atom is still involved in five short bonds, two Sn—N bond lengths, 2·474 (4) and 2·476 (4) Å, and three Sn—B distances that remain essentially unchanged when compared with those of stannacarbaborane precursors (Cowley, Galow, Hosmane, Jutzi & Norman, 1984; Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986). One of the more striking features of these donor–acceptor complexes is the 85·4 (2) to 126·2° B—Sn—N bond angle. This could result from electron repulsion between the lone pair on the Sn^{II} and the Sn—N bonded pairs or could be the result of orbital effects in that the Lewis base, 2,2′-bipyridine, is opposite the elongated Sn—C(cage)

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* Lists of structure factors, anisotropic temperature factors, selected torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43755 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

bonds. Evidently, in (IV), both Sn—C bonds are elongated as in (III) even when one of the cage C atoms carries a less bulky methyl group. Therefore, it could be concluded that the steric effects in the geometry of these complexes are minimal. Furthermore, a statement could be made that as a consequence of the formation of Sn—N bonds the Sn—C distances in both (III) and (IV) undergo a substantial elongation. However, no definitive explanation for the particular orientation of the bipyridyl group in these electron acceptor–donor complexes is apparent at this time.

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Table 1. *Fractional coordinates with equivalent isotropic thermal parameters for the non-hydrogen atoms (e.s.d.'s in parentheses)*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$*(Å^2)
Sn	0·05967 (3)	−0·11923 (2)	−0·07632 (2)	0·0519 (1)
Si	0·0720 (2)	−0·2678 (2)	0·1875 (1)	0·0743 (5)
C(1)	0·0908 (5)	−0·2596 (4)	0·0674 (3)	0·0474 (11)
C(2)	0·0134 (5)	−0·3095 (4)	−0·0155 (4)	0·0520 (12)
B(3)	0·0723 (7)	−0·3044 (5)	−0·0986 (5)	0·0609 (16)
B(4)	0·2175 (6)	−0·2445 (5)	−0·0596 (5)	0·0616 (16)
B(5)	0·2155 (6)	−0·2120 (5)	0·0486 (5)	0·0574 (15)
B(6)	0·1782 (6)	−0·3352 (5)	0·0103 (5)	0·0593 (16)
C(7)	−0·0985 (7)	−0·3742 (4)	−0·0100 (5)	0·0696 (16)
C(8)	0·1511 (15)	−0·1623 (13)	0·2559 (7)	0·2614 (73)
C(9)	−0·0919 (7)	−0·2569 (7)	0·1953 (5)	0·1107 (25)
C(10)	0·1334 (17)	−0·3897 (11)	0·2380 (9)	0·3438 (89)
N(11)	0·1064 (4)	−0·0859 (3)	−0·2279 (3)	0·0544 (11)
C(12)	0·0335 (6)	−0·1298 (4)	−0·3045 (4)	0·0663 (16)
C(13)	0·0424 (7)	−0·1066 (5)	−0·3907 (4)	0·0699 (17)
C(14)	0·1319 (6)	−0·0390 (5)	−0·4015 (4)	0·0710 (16)
C(15)	0·2065 (5)	0·0060 (4)	−0·3233 (4)	0·0602 (14)
C(16)	0·1930 (5)	−0·0197 (4)	−0·2373 (3)	0·0493 (12)
N(17)	0·2346 (4)	0·0017 (3)	−0·0737 (3)	0·0567 (11)
C(18)	0·2974 (6)	0·0431 (4)	0·0069 (4)	0·0698 (15)
C(19)	0·4000 (7)	0·1061 (4)	0·0137 (5)	0·0743 (18)
C(20)	0·4407 (7)	0·1262 (4)	−0·0610 (6)	0·0760 (19)
C(21)	0·3752 (6)	0·0866 (4)	−0·1472 (4)	0·0636 (15)
C(22)	0·2711 (5)	0·0246 (4)	−0·1506 (4)	0·0522 (12)

* Standard deviations of $U_{\rm eq}$ calculated according to Schomaker & Marsh (1983).

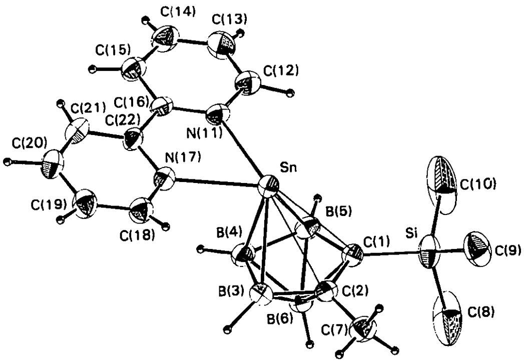

Fig. 1. *ORTEP* drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

Table 2. *Bond lengths* (Å) *and selected bond angles* (°)

Sn—C(1)	2·817 (5)	B(3)—B(4)	1·711 (10)
Sn—C(2)	2·819 (6)	B(3)—B(6)	1·768 (10)
Sn—B(3)	2·541 (7)	B(4)—B(5)	1·675 (10)
Sn—B(4)	2·359 (7)	B(4)—B(6)	1·731 (10)
Sn—B(5)	2·489 (7)	B(5)—B(6)	1·776 (10)
Sn—N(11)	2·474 (4)	N(11)—C(12)	1·341 (7)
Sn—N(17)	2·476 (4)	N(11)—C(16)	1·321 (7)
Si—C(1)	1·853 (6)	C(12)—C(13)	1·348 (9)
Si—C(8)	1·830 (15)	C(13)—C(14)	1·363 (9)
Si—C(9)	1·862 (16)	C(13)—C(15)	1·371 (8)
Si—C(10)	1·788 (8)	C(15)—C(16)	1·369 (8)
C(1)—C(2)	1·460 (8)	C(16)—C(22)	1·470 (8)
C(1)—B(5)	1·567 (9)	C(18)—N(17)	1·336 (7)
C(1)—B(6)	1·747 (9)	C(19)—C(18)	1·371 (9)
C(2)—B(3)	1·527 (9)	C(20)—C(19)	1·322 (10)
C(2)—B(6)	1·734 (9)	C(21)—C(20)	1·399 (9)
C(2)—C(7)	1·500 (9)	C(22)—C(21)	1·383 (8)
		C(22)—N(17)	1·337 (7)

C(1)—Sn—C(2)	30·0 (2)	C(1)—C(2)—B(3)	113·7 (5)
C(1)—Sn—B(3)	55·4 (2)	C(1)—C(2)—B(6)	65·7 (4)
C(1)—Sn—B(4)	59·5 (2)	C(2)—B(3)—B(4)	105·9 (5)
C(1)—Sn—B(5)	33·6 (2)	C(2)—B(3)—B(6)	63·0 (4)
C(1)—Sn—N(11)	143·4 (1)	B(4)—B(3)—B(6)	59·6 (4)
C(1)—Sn—N(17)	119·7 (1)	B(3)—B(4)—B(5)	102·4 (5)
C(2)—Sn—B(3)	32·6 (2)	B(3)—B(4)—B(6)	61·8 (4)
C(2)—Sn—B(4)	59·1 (2)	B(5)—B(4)—B(6)	62·9 (4)
C(2)—Sn—B(5)	55·7 (2)	C(1)—B(5)—B(4)	106·6 (5)
C(2)—Sn—N(11)	123·6 (2)	C(1)—B(5)—B(6)	62·6 (4)
C(2)—Sn—N(17)	142·6 (2)	B(4)—B(5)—B(6)	60·1 (4)
B(3)—Sn—B(4)	40·7 (2)	C(1)—B(6)—C(2)	49·6 (3)
B(3)—Sn—B(5)	63·3 (2)	C(1)—B(6)—B(3)	90·7 (4)
B(3)—Sn—N(11)	91·8 (2)	C(1)—B(6)—B(4)	96·8 (4)
B(3)—Sn—N(17)	126·2 (2)	C(1)—B(6)—B(5)	52·8 (3)
B(4)—Sn—B(5)	40·3 (2)	C(2)—B(6)—B(3)	51·7 (4)
B(4)—Sn—N(11)	85·4 (2)	C(2)—B(6)—B(4)	96·5 (4)
B(4)—Sn—N(17)	87·8 (2)	C(2)—B(6)—B(5)	90·7 (4)
B(5)—Sn—N(11)	121·3 (2)	B(3)—B(6)—B(4)	58·6 (4)
B(5)—Sn—N(17)	88·2 (2)	B(3)—B(6)—B(5)	96·2 (5)
N(11)—Sn—N(17)	64·2 (1)	B(4)—B(6)—B(5)	57·0 (4)
C(2)—C(1)—B(5)	111·1 (5)		
C(2)—C(1)—B(6)	64·8 (4)		
B(5)—C(1)—B(6)	64·5 (4)		

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