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Structure of (2,2′-Bipyridyl)(4-hydroxy-L-prolinato)palladium(II) Chloride Dihydrate

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Abstract. $[Pd(C_{10}H_8N_2)(C_5H_8NO_3)]Cl.2H_2O$, $M_r = 464{\cdot}2$, m.p. = 458 K, triclinic, $P1$, $a = 11{\cdot}100$ (3), $b = 13{\cdot}139$ (4), $c = 6{\cdot}887$ (4) Å, $\alpha = 105{\cdot}80$ (3), $\beta = 103{\cdot}50$ (3), $\lambda = 106{\cdot}63$ (3)°, $V = 871{\cdot}64$ Å^3, $Z = 2$, $D_x = 1{\cdot}769$, D_m(by flotation) = 1·777 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, Zr filter, $\mu = 12{\cdot}34$ cm^{-1}, $F(000) = 468$, room temperature, $R = 0{\cdot}053$, $wR = 0{\cdot}068$ for 1844 observed reflections. The two N atoms of bipyridyl and the imino N atom and carboxyl O atom of hydroxyproline give distorted square-planar coordination around palladium. Average Pd—O = 1·988 (10), Pd—N = 2·028 (11) Å. The proline rings are in the envelope conformation. The N atoms of the bipyridyl ring have bite angles of 80·9 (4) and 80·5 (4)° at palladium. The pyridine rings are twisted about the 2,2′ bond because of chelation strain. The two molecules are stacked along the c axis and linked with weak hydrogen bonds through the water molecules.

Introduction. In continuation of our work on the structural aspects of palladium(II) complexes, we report the structure of the title compound, a mixed-ligand chelate. A literature survey shows that very little work has been done on the synthesis and structural aspects of mixed-ligand palladium(II) amino-acid complexes (Nakayama, Komorita & Shimura, 1984).

Experimental. The complex was synthesized as follows:

$$\text{(i) } K_2PdCl_4 + 2,2'\text{-bipyridyl} \overset{CH_3OH}{\rightarrow} Pd(bpy)Cl_2 + 2KCl;$$

$$\text{(ii) } Pd(bpy)Cl_2 + 4\text{-hydroxyprol} + NaHCO_3 \overset{H_2O}{\rightarrow} [Pd(bpy)(hyd.prol)]^+ Cl^- + NaCl + H_2O + CO_2\uparrow.$$

Pale yellow crystals of the complex were prepared by repeated recrystallization from water. Intensity data for 1891 reflections (crystal size 0·47 × 0·30 × 1·10 mm) were collected on an indigenously fabricated computer-controlled four-circle diffractometer with Zr-filtered Mo $K\alpha$ radiation in the range $6 < 2\theta < 55°$ with ω–2θ step scan width of 1·8° with 33 steps deg^{-1} and 1·0 s step^{-1}. Data corrected for Lp and absorption effects (max. and min. values of the absorption coefficients are 0·697 and 0·570 respectively). Unit-cell parameters were obtained from a least-squares fit of the 2θ values of 25 reflections measured on the diffractometer in the range $5 < \theta < 12°$. Two standard reflections measured for every 100 reflections showed no significant change during the data collection process. The Pd atoms were located from a Patterson map. As expected, space group $P1$ with two molecules in the asymmetric unit presented some difficulties in its structure solution, because of false symmetry in the electron density maps (Stout & Jensen, 1968). However, the structure was solved by delineating chemically meaningful non-hydrogen atoms from a series of Fourier and difference Fourier maps. The non-hydrogen atoms were refined with anisotropic temperature factors. H atoms were fixed at calculated positions and two cycles of refinement with fixed isotropic temperature factors (0·05 Å^2) showed no significant changes in their parameters. The x,y,z coordinates for Pd were fixed to define the origin while the absolute configuration was established from the configuration of the amino acid. The scattering factor for palladium with corrections for dispersion effects was taken from *International Tables for X-ray*

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Crystallography (1974). *SHELX*76 (Sheldrick, 1976) and ND-500 computer used for all calculations. For 1844 unique reflections with $I \geq 2{\cdot}5\sigma(I)$, final $R = 0{\cdot}054$ and $wR = 0{\cdot}068$ with $w = k/[\sigma^2(F_o) + g(F_o)^2]$ where k and g refined to 1·000 and 0·001 respectively. $\Delta\rho$ in final difference map $-1{\cdot}5$ to $0{\cdot}9$ e Å^{-3} with the largest peak near Pd. No extinction corrections were applied. $(\Delta/\sigma)_{\max} = 0{\cdot}65$ for non-hydrogen atoms.

Discussion. The final positional parameters are given in Table 1.* Bond distances and angles are listed in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule is shown in Fig. 1 and the molecular packing in Fig. 2. The structure reveals no significant difference between the two moieties of the asymmetric unit. The central Pd atom in both the molecules is

Table 1. *Final atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^3) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
Pd(1)	19	13	41	30 (1)
N(1)	−737 (10)	1242 (9)	57 (16)	36 (1)
C(1)	−709 (14)	1620 (12)	−1810 (19)	41 (1)
C(2)	−1737 (14)	2199 (12)	−1891 (20)	43 (1)
C(3)	−2851 (13)	1424 (11)	−1394 (20)	40 (1)
C(4)	−2189 (12)	778 (11)	−171 (21)	40 (1)
C(5)	−2776 (12)	−538 (11)	−1400 (19)	36 (1)
O(1)	−1906 (9)	−1022 (7)	−1362 (15)	39 (1)
O(2)	−3993 (9)	−1072 (9)	−2288 (17)	52 (1)
O(3)	−1208 (11)	3308 (8)	−420 (17)	58 (1)
N(2)	1997 (9)	982 (8)	1421 (14)	20 (1)
C(6)	2617 (14)	2132 (11)	2187 (19)	38 (1)
C(7)	3973 (16)	2706 (13)	3129 (21)	54 (1)
C(8)	4768 (14)	2050 (13)	3330 (22)	54 (1)
C(9)	4182 (14)	876 (12)	2479 (20)	45 (1)
C(10)	2823 (13)	344 (13)	1561 (19)	38 (1)
N(3)	746 (11)	−1245 (9)	−95 (15)	30 (1)
C(11)	2082 (12)	−927 (10)	666 (16)	28 (1)
C(12)	2703 (14)	−1701 (12)	572 (20)	46 (1)
C(13)	1906 (14)	−2859 (11)	−282 (20)	44 (1)
C(14)	496 (15)	−3199 (12)	−1105 (20)	47 (1)
C(15)	−85 (12)	−2406 (11)	−1001 (18)	31 (1)
Pd(2)	−748 (1)	−1666 (1)	3761 (2)	28 (1)
N(4)	−2609 (10)	−2917 (9)	2554 (16)	34 (1)
C(16)	−3037 (13)	−3353 (11)	4174 (18)	35 (1)
C(17)	−4542 (13)	−3800 (13)	3355 (21)	42 (1)
C(18)	−4858 (14)	−2924 (12)	2490 (20)	46 (1)
C(19)	−3609 (10)	−2387 (10)	1852 (17)	24 (1)
C(20)	−3043 (13)	−1095 (12)	2781 (20)	43 (1)
O(4)	−1707 (9)	−617 (8)	3679 (15)	41 (1)
O(5)	−3751 (10)	−522 (9)	2885 (17)	53 (1)
O(6)	−5063 (9)	−4906 (8)	1720 (15)	46 (1)
N(5)	1076 (10)	−415 (9)	5134 (15)	29 (1)
C(21)	1318 (13)	745 (11)	5676 (17)	34 (1)
C(22)	2539 (14)	1558 (12)	6784 (20)	47 (1)
C(23)	3651 (14)	1216 (12)	7451 (19)	41 (1)
C(24)	3397 (14)	69 (12)	6854 (19)	43 (1)
C(25)	2147 (12)	−731 (10)	5738 (16)	28 (1)
N(6)	394 (9)	−2623 (8)	4019 (15)	26 (1)
C(26)	1738 (12)	−1989 (11)	5055 (17)	34 (1)
C(27)	2652 (13)	−2533 (11)	5368 (19)	38 (1)
C(28)	2187 (14)	−3707 (12)	4680 (19)	42 (1)
C(29)	821 (14)	−4381 (12)	3533 (20)	47 (1)
C(30)	−35 (13)	−3767 (10)	3310 (18)	35 (1)
O(*W*1)	107 (11)	2888 (10)	4333 (15)	62 (1)
O(*W*2)	−3608 (12)	−6301 (10)	755 (17)	63 (1)
O(*W*3)	8105 (11)	3822 (10)	4580 (17)	70 (1)
O(*W*4)	3982 (14)	5054 (13)	6725 (19)	93 (1)
Cl(1)	1928 (4)	4287 (3)	9238 (6)	52 (1)
Cl(2)	7076 (4)	5251 (4)	8030 (6)	50 (1)

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

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

Molecule I			
Pd(1)–O(1)	2·001 (8)	N(2)–C(6)	1·355 (15)
Pd(1)–N(1)	2·021 (14)	C(6)–C(7)	1·372 (19)
Pd(1)–N(2)	2·028 (8)	C(7)–C(8)	1·410 (27)
Pd(1)–N(3)	2·027 (13)	C(8)–C(9)	1·379 (20)
N(1)–C(1)	1·502 (20)	C(9)–C(10)	1·369 (18)
C(1)–C(2)	1·541 (25)	C(10)–N(2)	1·414 (21)
C(2)–C(3)	1·533 (21)	C(10)–C(11)	1·504 (18)
C(3)–C(4)	1·558 (23)	C(11)–C(12)	1·378 (23)
N(1)–C(4)	1·503 (17)	C(12)–C(13)	1·393 (18)
C(4)–C(5)	1·560 (18)	C(13)–C(14)	1·421 (22)
C(5)–O(1)	1·299 (19)	C(14)–C(15)	1·368 (24)
C(5)–O(2)	1·236 (14)	C(15)–N(3)	1·405 (15)
C(2)–O(3)	1·386 (15)	N(3)–C(11)	1·346 (17)
Molecule II			
Pd(2)–O(4)	1·974 (12)	N(5)–C(21)	1·394 (19)
Pd(2)–N(4)	2·042 (9)	C(21)–C(22)	1·343 (16)
Pd(2)–N(5)	2·013 (9)	C(22)–C(23)	1·449 (24)
Pd(2)–N(6)	2·036 (12)	C(23)–C(24)	1·373 (22)
N(4)–C(16)	1·497 (19)	C(24)–C(25)	1·360 (16)
C(16)–C(17)	1·508 (19)	C(25)–N(5)	1·381 (19)
C(17)–C(18)	1·520 (25)	C(25)–C(26)	1·486 (19)
C(18)–C(19)	1·584 (20)	C(26)–C(27)	1·408 (23)
C(19)–N(4)	1·524 (19)	C(27)–C(28)	1·380 (20)
C(19)–C(20)	1·516 (18)	C(28)–C(29)	1·413 (17)
C(20)–O(4)	1·342 (15)	C(29)–C(30)	1·420 (24)
C(20)–O(5)	1·235 (22)	C(30)–N(6)	1·344 (16)
C(17)–O(6)	1·428 (16)	N(6)–C(26)	1·377 (14)
Molecule I			
O(1)–Pd(1)–N(1)	83·0 (4)	N(2)–C(6)–C(7)	124·6 (15)
O(1)–Pd(1)–N(3)	95·9 (4)	C(6)–C(7)–C(8)	117·4 (14)
N(1)–Pd(1)–N(2)	100·2 (4)	C(7)–C(8)–C(9)	119·8 (13)
N(2)–Pd(1)–N(3)	80·9 (4)	C(8)–C(9)–C(10)	120·4 (16)
N(1)–C(1)–C(2)	102·5 (12)	C(9)–C(10)–N(2)	120·9 (13)
C(1)–C(2)–C(3)	103·3 (13)	C(10)–N(2)–C(6)	116·8 (10)
C(1)–C(2)–O(3)	112·5 (13)	C(9)–C(10)–C(11)	124·6 (15)
C(2)–C(3)–C(4)	105·9 (12)	N(2)–C(10)–C(11)	114·5 (10)
C(3)–C(4)–N(1)	105·2 (12)	C(10)–C(11)–C(12)	123·6 (12)
C(4)–N(1)–C(1)	105·2 (10)	C(11)–C(12)–C(13)	118·3 (13)
N(1)–C(4)–C(5)	109·2 (12)	C(12)–C(13)–C(14)	119·1 (15)
C(3)–C(4)–C(5)	113·2 (9)	C(13)–C(14)–C(15)	120·9 (12)
C(4)–C(5)–O(1)	115·4 (10)	C(14)–C(15)–N(3)	118·5 (12)
C(4)–C(5)–O(2)	121·4 (14)	C(15)–N(3)–C(11)	120·4 (13)
O(1)–C(5)–O(2)	123·3 (12)	N(3)–C(11)–C(12)	122·7 (11)
Molecule II			
O(4)–Pd(2)–N(4)	85·0 (0)	N(5)–C(21)–C(22)	122·7 (15)
O(4)–Pd(2)–N(5)	94·4 (4)	C(21)–C(22)–C(23)	118·7 (14)
N(4)–Pd(2)–N(6)	100·0 (4)	C(22)–C(23)–C(24)	117·7 (11)
N(5)–Pd(2)–N(6)	80·5 (4)	C(23)–C(24)–C(25)	122·0 (15)
N(4)–C(16)–C(17)	105·7 (11)	C(24)–C(25)–N(5)	120·7 (13)
C(16)–C(17)–C(18)	103·6 (12)	C(25)–N(5)–C(21)	118·1 (9)
C(16)–C(17)–O(6)	110·2 (13)	C(24)–C(25)–C(26)	127·2 (14)
C(17)–C(18)–C(19)	104·7 (13)	N(5)–C(25)–C(26)	112·0 (9)
C(18)–C(19)–N(4)	106·1 (11)	C(25)–C(26)–C(27)	123·2 (10)
C(19)–N(4)–C(16)	104·0 (11)	C(26)–C(27)–C(28)	119·4 (11)
N(4)–C(19)–C(20)	113·7 (9)	C(27)–C(28)–C(29)	121·4 (15)
C(18)–C(19)–C(20)	112·7 (11)	C(28)–C(29)–C(30)	115·5 (13)
C(19)–C(20)–O(4)	114·8 (14)	C(29)–C(30)–N(6)	123·9 (11)
C(19)–C(20)–O(5)	123·0 (11)	C(30)–N(6)–C(26)	119·2 (13)
O(4)–C(20)–O(5)	121·9 (12)	N(6)–C(26)–C(27)	120·5 (12)

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* 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 43730 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

four-coordinated with a distorted square-planar geometry. The two N atoms of the bipyridyl ligand and the imino nitrogen and carboxyl O atom of hydroxyproline are chelated to Pd forming a cationic complex with Cl acting as a counter ion. The bond distances Pd—N [average 2·028 (11) Å] and Pd—O [average 1·988 (10) Å] are normal and in the ranges of literature values (Patel, Ranganathan & Padmanabhan, 1986). The bipyridyl ligands have bite angles of 80·9 (4) and 80·5 (4)° while the hydroxyprolines have 83·0 (4) and 84·8 (5)°. The two pyridine rings are slightly twisted (~3·0 and 2·66°) about the C—C (2,2′) bond due to chelation strain. The five atoms defining the coordination plane (Pd, three N atoms and O atom) are coplanar within 0·04 (1) Å. The puckering parameters calculated according to Cremer & Pople (1975) for the five-membered pyrrolidine rings are given in Table 4. The φ values are near 0° (355·9 and 7·1°) indicating the proline rings to be in the envelope conformation. The apex atoms are C(1) and C(16), *i.e.* the C^δ atoms, which is rather unusual. This may be due to non-bonded interaction between H—C^δ and the H—C(6), H—C(30) atoms of the bipyridyl rings respectively. Such unusual interactions are known in bis(bipyridyl) complexes (Cordes, Dushan, Swepston, Pennington, Condren, Jensen & Walsh, 1982), where the steric

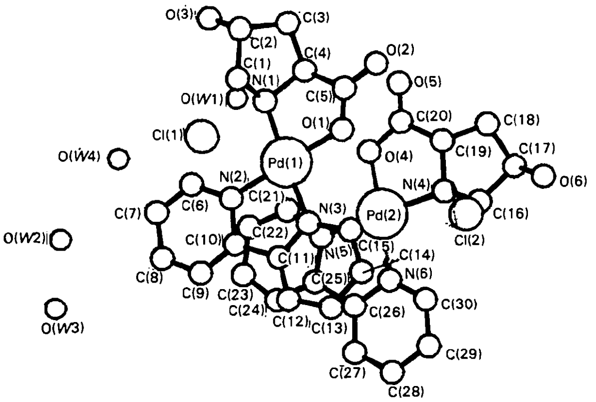

Fig. 1. General view of the molecule.

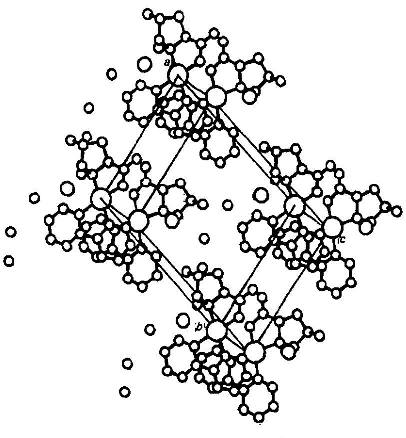

Fig. 2. Unit-cell packing diagram.

Table 3. *Best planes and deviations* (Å) *of atoms from them*

Molecule I

*P*1		*P*2		*P*3	
Pd(1)	−0·015 (1)	Pd(1)	−0·100 (1)	Pd(1)	0·006 (1)
N(1)	0·012 (2)	N(1)	0·161 (2)	N(2)	−0·004 (1)
N(2)	−0·005 (1)	C(4)	−0·160 (2)	N(3)	−0·009 (1)
N(3)	0·012 (1)	C(5)	0·052 (2)	C(10)	−0·001 (1)
O(1)	−0·005 (1)	O(1)	0·047 (1)	C(11)	0·009 (1)
C(6)*	−0·010 (2)				
C(15)*	0·046 (2)				

*P*4	
N(1)	−0·011 (2)
C(2)	0·011 (2)
C(3)	−0·017 (2)
C(4)	0·017 (2)
C(1)*	0·597 (2)

Molecule II

*P*5		*P*6		*P*7	
Pd(2)	0·032 (1)	Pd(2)	0·060 (1)	Pd (2)	−0·028 (1)
N(4)	−0·027 (1)	N(4)	−0·096 (1)	N(5)	0·037 (1)
N(5)	−0·029 (1)	C(19)	0·100 (2)	N(6)	0·026 (1)
N(6)	0·012 (1)	C(20)	−0·035 (2)	C(25)	−0·026 (2)
O(4)	0·013 (1)	O(4)	−0·028 (1)	C(26)	−0·009 (2)
C(30)*	0·097 (2)				
C(21)*	−0·057 (2)				

*P*8	
N(4)	0·018 (1)
C(17)	−0·018 (2)
C(18)	0·027 (2)
C(19)	−0·027 (2)
C(16)*	0·551 (2)

Dihedral angles (°)

Planes	
1–2	7·19 (8)
1–3	1·26 (7)
1–4	42·31 (8)
5–6	3·12 (7)
5–7	3·73 (8)
5–8	139·87 (8)

* Not included in least-squares-plane calculation.

Table 4. *Some torsional angles* (°) *and puckering parameters with e.s.d.'s in parentheses*

Molecule I

N(1)—C(4)—C(5)—O(1)	20·9 (3)
N(2)—C(10)—C(11)—N(3)	−1·1 (3)
N(1)—C(1)—C(2)—C(3)	−39·2 (4)
C(1)—C(2)—C(3)—C(4)	21·9 (4)
C(2)—C(3)—C(4)—N(1)	3·2 (4)
C(3)—C(4)—N(1)—C(1)	−27·7 (4)
C(4)—N(1)—C(1)—C(2)	41·7 (4)
q_2	0·41 (1) Å
φ_2	355·9 (2)°

[Calculated with C(1) as first atom]

Molecule II

N(4)—C(19)—C(20)—O(4)	13·4 (3)
N(5)—C(25)—C(26)—N(6)	−1·9 (3)
N(4)—C(16)—C(17)—C(18)	−40·2 (4)
C(16)—C(17)—C(18)—C(19)	26·8 (4)
C(17)—C(18)—C(19)—N(4)	−4·8 (4)
C(18)—C(19)—N(4)—C(16)	−19·1 (3)
C(19)—N(4)—C(16)—C(17)	36·8 (4)
q_2	0·38 (1) Å
φ_2	7·1 (1)°

[Calculated with C(16) as first atom]

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 $Na[(Me_3Si)(R)C_2B_4H_5]$ with $SnCl_2$ in THF (tetrahydrofuran) produces an air-sensitive THF–stannacarbaborane intermediate, $(C_4H_8O)_2Sn(Me_3Si)(R)C_2B_4H_4$ (I) ($R = Me_3Si$, 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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