enantiomers; from O(2) in one enantiomer to H(1) in the other enantiomer with hydrogen-bond lengths and angle [O(2)···H(1)] = 2·15 (3), [O(2)···O(1)] = 2·919 (4) Å and [O(2)···H(1)···O(1)] = 178·8 (3)°. These hydrogen bonds are probably a major factor in restricting the free rotation of the hydroxyethyl group.

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1-(2,2′-Bipyridine)-2-methyl-3-(trimethylsilyl)-2,3-dicarba-1-plumba-*closo*-heptaborane(6)

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Abstract. $C_{16}H_{24}B_4N_2PbSi$, $M_r = 522{\cdot}9$, triclinic, $P\bar{1}$, $a = 7{\cdot}007$ (3), $b = 9{\cdot}318$ (6), $c = 16{\cdot}462$ (13) Å, $\alpha = 103{\cdot}84$ (6), $\beta = 96{\cdot}40$ (5), $\gamma = 95{\cdot}21$ (4)°, $V = 1029{\cdot}4$ (11) Å^3, $Z = 2$, $D_x = 1{\cdot}69$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 83·12 cm^{-1}, $F(000) = 500$, $T = 295$ K. Final $R = 0{\cdot}035$ for 2781 observed reflections. The structure shows that the title compound (V) consists of a distorted pentagonal bipyramid with the apical Pb atom displaced significantly from the centroidal position toward the basal borons above the C_2B_3 face. The Pb-bound Lewis base, 2,2′-bipyridine, is not exactly opposite the C—C$_{\text{(cage)}}$ bond, rather it is tilted significantly toward the two basal borons above the C_2B_3 face with B—Pb—N orientation angles of about 81–141°. Bond distances in the cage structure are Pb—C 2·835 (8) and 2·770 (8), Pb—B 2·566 (10), 2·466 (8) and 2·601 (9), and Pb—N 2·615 (7) and 2·663 (7) Å. The crystal packing in the unit cell reveals that the intermolecular dipole–dipole type of interactions within the dimeric plumbacarborane precursor (II) have been broken during the formation of its donor–acceptor complex (V) with the Lewis base, 2,2′-bipyridine.

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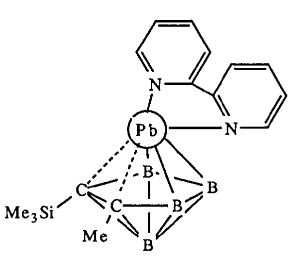
Introduction. The first structural evidence for the insertion of an unsubstituted lead into a carborane cage in the production of *closo*-1-Pb-2,3-$(SiMe_3)_2$-2,3-$C_2B_4H_4$ (I) was furnished very recently (Hosmane, Siriwardane, Zhu, Zhang & Maguire, 1989). Since then a number of C-substituted *closo*-plumbacarborane derivatives such as 1-Pb-2-(Me)-3-$(SiMe_3)$-2,3-$C_2B_4H_4$ (II) and 1-Pb-2-(H)-3-$(SiMe_3)$-2,3-$C_2B_4H_4$ (III) have been synthesized and their crystal structures have been determined (Hosmane, Lu, Zhu, Siriwardane, Shet & Maguire, 1990). In the crystal structures, the molecular dimers of (I) with solvated benzene molecules are packed, with a crystallographic inversion center, as symmetrical layers on top of each other in its extended network, while each molecular dimer of (II) consists of two crystallographically independent molecules that are packed as two parallel networks of layers of molecular dimers with a crystallographic center of inversion between them. In this respect, the *closo*-plumbacarboranes differ significantly from the *closo*-germacarboranes and *closo*-stannacarboranes (Hosmane & Maguire, 1988, 1990). However, all of them behave as Lewis acids in the formation of their donor–acceptor complexes with the Lewis base 2,2′-bipyridine (Hosmane & Maguire, 1988, 1990). Recently, the crystal structure of 1-Pb$(C_{10}H_8N_2)$-2,3-$(SiMe_3)_2$-2,3-$C_2B_4H_4$ (IV) has been determined to show that the intermolecular dipole–dipole type of interactions within the symmetric plumbacarborane dimers of the precursor (I) were broken during the formation of (IV) (Hosmane, Lu, Zhu, Siriwardane, Shet & Maguire, 1990). In order to explore further the unusual packing behavior, the extent of the slippage of the apical Pb within the cage, as well as the strange tilting of the metal-bound Lewis base as found in the structure of (IV), and also to find a structural pattern in the new system, we have conducted a structural investigation of the title compound (V) in which one of the cage C atoms carries a methyl group (Fig. 1), and report herein the results.

Experimental. The title compound 1-Pb$(C_{10}H_8N_2)$-2-(Me)-3-$(SiMe_3)$-2,3-$C_2B_4H_4$ (V) was prepared as a pale orange solid in a reaction involving *closo*-1-Pb-2-(Me)-3-$(SiMe_3)$-2,3-$C_2B_4H_4$ (II) and 2,2′-bipyridine in a molar ratio of 1:1 in benzene at room temperature over a period of three days (Hosmane, Lu, Zhu, Siriwardane, Shet & Maguire, 1990). This solid was sublimed *in vacuo* at 413 K and then crystallized from acetonitrile to collect (V) in high purity and in 61% yield.



B = BH

Fig. 1. Graphic representation of the title compound (V).

Crystals of (V) are pale yellow plates, unit-cell parameters by least-squares fit of 15 reflections in the range $10 \leq 2\theta \leq 25°$, crystal dimensions $0{\cdot}10 \times 0{\cdot}10 \times 0{\cdot}30$ mm, space group $P\bar{1}$; automatic Nicolet *R3m/V* diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode (scan rate 3·0–15·0° min^{-1}, depending upon the intensity), 3962 measured reflections, 3638 independent reflections in the range $3 \leq 2\theta \leq 50°$, $R_{\text{int}} = 0{\cdot}014$, *hkl* range h $0 \rightarrow 8$, k $-11 \rightarrow 11$, l $-19 \rightarrow 19$; 2781 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity (~4%) during the data collection; Lorentz–polarization correction, no extinction corrections. Absorption correction based on ψ scans, the maximum and minimum transmission factors were 1·000 and 0·456, respectively. Structure solved by direct-methods program used in Nicolet software package *SHELXTL-Plus* (Sheldrick, 1988), refinement by full-matrix least squares using *SHELXTL-Plus*, anisotropic; boron-cage H atoms were located in difference Fourier maps, H atoms were included with fixed isotropic displacement parameters (0·08 Å^2); the positions of the cage H atoms were fixed and the calculated positions of the methyl and bipyridine-ring H atoms were constrained tetrahedrally; $w = 1/(\sigma^2 F_o + 0{\cdot}00054 F_o^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}035$, $wR = 0{\cdot}040$, $S = 1{\cdot}15$ for 217 parameters, $(\Delta/\sigma)_{\max} = 0{\cdot}001$, $\Delta\rho_{(\max,\min)} = 0{\cdot}88$, $-1{\cdot}19$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, B, N, Si and Pb were those stored in *SHELXTL-Plus*.

Discussion. The final atomic parameters of the non-H atoms are given in Table 1.* Selected bond lengths, bond angles and torsion angles with their standard deviations are given in Table 2. The identification of the atoms and the configuration of the molecule are shown in the thermal ellipsoid (*SHELXTL-Plus*; Sheldrick, 1988) drawing of Fig. 2. Fig. 3 represents the unit-cell packing arrangement. The structure

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* Lists of structure factors, anisotropic temperature factors, selected bond angles and torsion angles, mean deviations of the least-squares planes and their dihedral angles between the C_2B_3 face and 2,2′-bipyridine rings, and H parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52748 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
Pb	0·5328 (1)	0·8878 (1)	0·7471 (1)	0·049 (1)
Si	0·2038 (4)	0·6696 (3)	0·9051 (2)	0·060 (1)
N(1)	0·6511 (9)	0·8380 (6)	0·5989 (4)	0·046 (2)
N(2)	0·6207 (10)	1·1172 (7)	0·6822 (4)	0·053 (3)
C(1)	0·2295 (11)	0·7530 (8)	0·8147 (4)	0·045 (3)
C(2)	0·1827 (11)	0·9029 (8)	0·8101 (5)	0·048 (3)
B(3)	0·1763 (13)	0·9288 (10)	0·7207 (6)	0·052 (3)
B(4)	0·2264 (13)	0·7669 (10)	0·6585 (5)	0·051 (3)
B(5)	0·2707 (13)	0·6612 (10)	0·7270 (5)	0·048 (3)
B(6)	0·0668 (14)	0·7585 (10)	0·7318 (5)	0·050 (3)
C(3)	0·1086 (14)	1·0032 (10)	0·8821 (6)	0·069 (4)
C(4)	−0·0467 (14)	0·6542 (12)	0·9275 (6)	0·085 (5)
C(5)	0·3608 (19)	0·7803 (15)	1·0006 (7)	0·113 (6)
C(6)	0·2855 (19)	0·4828 (12)	0·8819 (8)	0·105 (6)
C(7)	0·6549 (12)	0·6982 (9)	0·5564 (6)	0·059 (3)
C(8)	0·6999 (12)	0·6571 (10)	0·4761 (6)	0·063 (3)
C(9)	0·7479 (11)	0·7685 (10)	0·4378 (6)	0·062 (4)
C(10)	0·7484 (10)	0·9133 (10)	0·4801 (5)	0·052 (3)
C(11)	0·7016 (9)	0·9450 (7)	0·5610 (4)	0·039 (2)
C(12)	0·7035 (10)	1·1013 (8)	0·6125 (5)	0·042 (3)
C(13)	0·7880 (11)	1·2216 (9)	0·5881 (6)	0·056 (3)
C(14)	0·7878 (14)	1·3603 (10)	0·6368 (7)	0·070 (4)
C(15)	0·6981 (14)	1·3827 (10)	0·7109 (7)	0·076 (4)
C(16)	0·6243 (13)	1·2563 (10)	0·7315 (6)	0·069 (4)

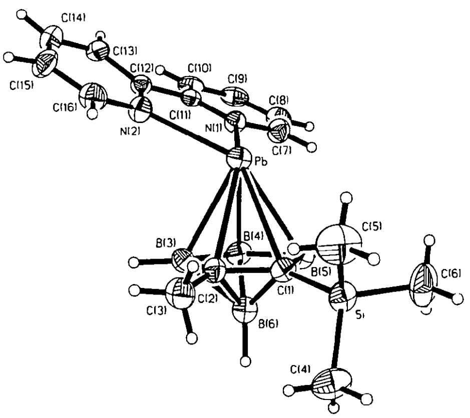

Fig. 2. Thermal ellipsoid (*SHELXTL-Plus*) drawing of the molecule. The thermal ellipsoids are scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

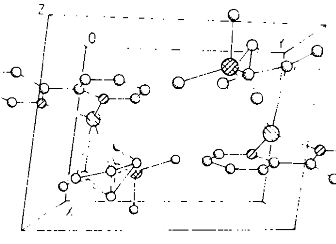

Fig. 3. The molecular packing in the unit cell. H atoms are removed for clarity.

Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

Pb—N(1)	2·615 (7)	Pb—N(2)	2·663 (7)
Pb—C(1)	2·835 (8)	Pb—C(2)	2·770 (8)
Pb—B(3)	2·566 (10)	Pb—B(4)	2·466 (8)
Pb—B(5)	2·601 (9)	Si—C(1)	1·856 (9)
Si—C(4)	1·833 (11)	Si—C(5)	1·837 (11)
Si—C(6)	1·848 (12)	N(1)—C(7)	1·326 (9)
N(1)—C(11)	1·338 (10)	N(2)—C(12)	1·327 (10)
N(2)—C(16)	1·351 (10)	C(1)—C(2)	1·481 (11)
C(1)—B(5)	1·564 (11)	C(1)—B(6)	1·691 (12)
C(2)—B(3)	1·544 (14)	C(2)—B(6)	1·694 (10)
C(2)—C(3)	1·498 (12)	B(3)—B(4)	1·698 (13)
B(3)—B(6)	1·757 (14)	B(4)—B(5)	1·687 (14)
B(4)—B(6)	1·745 (14)	B(5)—B(6)	1·759 (14)
C(7)—C(8)	1·367 (13)	C(8)—C(9)	1·373 (14)
C(9)—C(10)	1·362 (12)	C(10)—C(11)	1·377 (11)
C(11)—C(12)	1·498 (9)	C(12)—C(13)	1·384 (12)
C(13)—C(14)	1·348 (11)	C(14)—C(15)	1·415 (16)
C(15)—C(16)	1·375 (14)		

N(1)—Pb—N(2)	60·9 (2)	N(1)—Pb—C(1)	134·3 (2)
N(2)—Pb—C(1)	141·6 (2)	N(1)—Pb—C(2)	137·1 (2)
N(2)—Pb—C(2)	111·5 (2)	C(1)—Pb—C(2)	30·6 (2)
N(1)—Pb—B(3)	105·4 (3)	N(2)—Pb—B(3)	87·8 (3)
C(1)—Pb—B(3)	55·6 (3)	C(2)—Pb—B(3)	33·4 (3)
N(1)—Pb—B(4)	80·8 (3)	N(2)—Pb—B(4)	102·2 (3)
C(1)—Pb—B(4)	58·3 (3)	C(2)—Pb—B(4)	58·7 (3)
B(3)—Pb—B(4)	39·4 (3)	N(1)—Pb—B(5)	101·8 (2)
N(2)—Pb—B(5)	141·0 (2)	C(1)—Pb—B(5)	33·1 (2)
C(2)—Pb—B(5)	55·6 (2)	B(3)—Pb—B(5)	61·9 (3)
B(4)—Pb—B(5)	38·8 (3)	Pb—N(1)—C(7)	118·7 (6)
Pb—N(1)—C(11)	124·0 (4)	Pb—N(2)—C(12)	121·8 (5)
Pb—N(2)—C(16)	118·3 (6)	Pb—C(1)—C(2)	72·3 (4)
Pb—C(1)—B(5)	65·2 (4)	Pb—C(1)—B(6)	91·4 (5)
Pb—C(2)—C(1)	77·1 (4)	Pb—C(2)—B(3)	66·0 (5)
Pb—C(2)—B(6)	93·6 (5)	Pb—C(2)—C(3)	135·5 (5)
Pb—B(3)—C(2)	80·6 (5)	Pb—B(3)—B(4)	67·1 (4)
Pb—B(3)—B(6)	99·3 (5)	Pb—B(4)—B(3)	73·5 (4)
Pb—B(4)—B(5)	74·9 (4)	Pb—B(4)—B(6)	103·5 (5)
Pb—B(5)—C(1)	81·7 (4)	Pb—B(5)—B(4)	66·3 (4)
Pb—B(5)—B(6)	98·0 (5)		

N(2)—Pb—N(1)—C(7)	−176·3 (1)	N(2)—Pb—N(1)—C(11)	1·0 (1)
N(1)—Pb—N(2)—C(12)	−8·4 (1)	N(1)—Pb—N(2)—C(16)	−172·3 (1)
N(1)—Pb—B(4)—B(6)	177·4 (1)	N(2)—Pb—B(4)—B(6)	−125·2 (1)
Pb—N(1)—C(7)—C(8)	175·0 (1)	Pb—N(1)—C(11)—C(10)	−174·8 (1)
Pb—N(1)—C(11)—C(12)	5·2 (1)	Pb—N(2)—C(12)—C(11)	14·3 (1)
Pb—N(2)—C(12)—C(13)	−165·6 (1)	C(16)—N(2)—C(12)—C(11)	178·2 (1)
Pb—N(2)—C(16)—C(15)	168·7 (1)	C(9)—C(10)—C(11)—C(12)	178·5 (1)
N(1)—C(11)—C(12)—N(2)	−12·5 (1)	N(1)—C(11)—C(12)—C(13)	167·3 (1)
C(10)—C(11)—C(12)—N(2)	167·5 (1)	C(10)—C(11)—C(12)—C(13)	−12·7 (1)
C(16)—N(2)—C(12)—C(13)	−1·6 (1)	C(12)—N(2)—C(16)—C(15)	4·2 (1)

shows a significant slippage of the apical Pb from the centroidal position above the C_2B_3 face in a distorted pentagonal-bipyramidal geometry, and the Lewis base, 2,2′-bipyridine, is not situated exactly opposite the C—C$_{(cage)}$ bond, rather it is tilted significantly toward two of the basal borons above the C_2B_3 face with B—Pb—N orientation angles of about 81–141°. The distortion of the cage primarily involves the Pb—C distances [2·835 (8) and 2·770 (8) Å], which are substantially longer than the Pb—B distances [2·566 (10), 2·466 (8) and 2·601 (9) Å]. Although one of the Pb—N distances [2·663 (7) Å] is slightly longer than the other [2·615 (7) Å], a significant difference in Pb—N bond lengths [2·674 (9) *vs* 2·581 (9) Å] has been found in the structure of the similar complex (IV) and is presumably responsible in this complex for the tilting of the coordinated 2,2′-bipyridine toward two basal borons above the C_2B_3 face. Since the two Pb—N distances are almost equal in the title compound (V), there is no apparent reason for the same kind of tilting of the Pb-bound Lewis base as the one observed for (IV). However, one of the interesting features of the structure is the overall non-planarity of the coordinated 2,2′-bipyridine molecule itself. The dihedral angle between the two C_5N rings within the 2,2′-bipyridine ligand in the title compound is 12·8°, while in the stannacarborane–2,2′-bipyridine complexes, 1-Sn($C_{10}H_8N_2$)-2,3-($SiMe_3$)$_2$-2,3-$C_2B_4H_4$ and 1-Sn($C_{10}H_8N_2$)-2-(Me)-3-($SiMe_3$)-2,3-$C_2B_4H_4$, this is 5·6 and 8·8°, respectively (Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986; Siriwardane, Hosmane & Chu, 1987). The dihedral angles between the planes formed by the C_2B_3 face of the plumbacarborane cage and the C_5N rings of the bipyridine ligand containing N(1) and N(2) donor atoms, are 24·2 and 36·0°, respectively. It is interesting to point out that the dihedral angle of 30·2° between the C_2B_3 face and the calculated overall plane of the 2,2′-bipyridine molecule is significantly larger than that of 1-Pb($C_{10}H_8N_2$)-2,3-($SiMe_3$)$_2$-2,3-$C_2B_4H_4$ (IV) (27·5°) and of similar stannacarborane complexes 1-Sn($C_{10}H_8N_2$)-2,3-($SiMe_3$)$_2$-2,3-$C_2B_4H_4$ (26·8°) and 1-Sn($C_{10}H_8N_2$)-2-(Me)-3-($SiMe_3$)-2,3-$C_2B_4H_4$ (18·4°) (Hosmane, Lu, Zhu, Siriwardane, Shet & Maguire, 1990; Hosmane, de Meester, Maldar, Potts, Chu & Herber, 1986; Siriwardane, Hosmane & Chu, 1987). The crystal packing diagram (Fig. 3) shows that the intermolecular dipole–dipole type of interactions within the dimeric precursor (II) were broken during the formation of the title compound (V).

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Structure of Potassium Hydrogen Cyclopropane-1,1-dicarboxylate Hemihydrate

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Abstract. K[$C_5H_5O_4$].1/2H_2O, $M_r = 177{\cdot}2$, monoclinic, $P2_1/a$, $a = 9{\cdot}041$ (1), $b = 15{\cdot}634$ (2), $c = 9{\cdot}874$ (2) Å, $\beta = 93{\cdot}85$ (1)°, $V = 1392{\cdot}5$ (4) Å^3, $Z = 8$, $D_x = 1{\cdot}69$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 65$ cm^{-1}, $F(000) = 720$, $T = 294$ K, $R = 0{\cdot}061$ for 1873 observed independent reflections. The asymmetric unit contains two independent molecules each with an intramolecular hydrogen bond. The potassium ions are both eight coordinated by carboxylate and water oxygens (6 + 2 and 7 + 1).

Introduction. In a previous spectroscopic study, Fabregue, Cassanas, Delarbre, Maury & Bardet (1988) concluded that, in aqueous solution, the cyclopropane-1,1-dicarboxylic acid salt belongs to the point group C_2 and was, in addition, intramolecu-

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