

Fig. 1. Thermal-motion ellipsoid plot of $[\text{Zn}(\text{amp})(\text{NCS})_2]$. The surfaces have been drawn at the 50% probability level for non-hydrogen atoms and artificially small for H atoms.

unusual for Zn. In this respect it is of interest that the equatorial NCS ligand coordinates to Zn almost linearly [$\text{Zn}-\text{N}(40)-\text{C}(41) = 174.6(7)^\circ$] and the axial one with an angle of $149.8(7)^\circ$. Another remarkable feature of this compound is the third pyrazole group of the ligand amtp which does not coordinate to the Zn. In the compounds so far described with this type of poly(pyrazolyl)amine ligand, all pyrazole groups participate in the coordination of the central metal ion (Hulsbergen, Driessen, Reedijk & Verschoor, 1984; van Driel, Driessen & Reedijk, 1985; Kleywegt, Wiesmeijer, van Driel, Driessen, Reedijk & Noordik, 1985; Paap, 1987). In other cases, when all the pyrazole groups of a particular poly(pyrazolyl)amine ligand seem not to fit, one of the pyrazole rings splits off

and the remaining groups coordinate (Driessen, de Graaff & Wiesmeijer, 1987; Spek, Duisenberg, Driessen & Wiesmeijer, 1988).

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Structure of Bis[1,3-bis(5-methyl-4-imidazolyl)-2-thiopropane-*N,N,S*]nickel(II) Hexafluorosilicate Pentahydrate

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Abstract. $[\text{Ni}(\text{C}_{10}\text{H}_{14}\text{N}_4\text{S}_2)_2]\text{SiF}_6 \cdot 5\text{H}_2\text{O}$, $M_r = 735.46$, monoclinic, $P2_1/c$, $a = 14.047(1)$, $b = 10.611(1)$, c

$= 20.782(2) \text{ \AA}$, $\beta = 103.65(1)^\circ$, $V = 3010.1(5) \text{ \AA}^3$, $Z = 4$, $D_x = 1.623 \text{ g cm}^{-3}$, $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ \AA}$, $\mu = 33.4 \text{ cm}^{-1}$, $F(000) = 1528$, $T = 295 \text{ K}$, $R = 0.059$ for 2922 observed reflections with $I > 2.5\sigma(I)$. The Ni^{II}

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Table 1. Final coordinates and equivalent isotropic thermal parameters and their e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Ni	0.31182 (7)	0.26250 (9)	0.12963 (5)	0.0404 (4)
S(8)	0.48958 (12)	0.25899 (15)	0.18931 (8)	0.0479 (6)
S(9)	0.34337 (13)	0.39377 (15)	0.03719 (8)	0.0450 (6)
N(11)	0.3612 (5)	-0.0640 (5)	0.0279 (3)	0.057 (2)
N(13)	0.3521 (4)	0.1029 (5)	0.0881 (3)	0.042 (2)
N(21)	0.2601 (5)	0.0305 (5)	0.2831 (3)	0.057 (3)
N(23)	0.2956 (4)	0.1635 (4)	0.2121 (3)	0.045 (2)
N(31)	0.0141 (5)	0.2912 (6)	0.0366 (3)	0.075 (3)
N(33)	0.1712 (4)	0.2669 (5)	0.0735 (3)	0.047 (2)
N(41)	0.2547 (4)	0.5942 (5)	0.2238 (3)	0.051 (2)
N(43)	0.2917 (4)	0.4364 (4)	0.1663 (3)	0.043 (2)
C(10)	0.5215 (5)	0.1080 (7)	0.1616 (4)	0.066 (3)
C(12)	0.3039 (5)	0.0302 (6)	0.0407 (3)	0.052 (3)
C(14)	0.4445 (5)	0.0513 (6)	0.1064 (3)	0.046 (3)
C(15)	0.4519 (6)	-0.0514 (6)	0.0694 (3)	0.049 (3)
C(16)	0.5344 (6)	-0.1391 (7)	0.0674 (4)	0.073 (3)
C(20)	0.4669 (5)	0.2248 (7)	0.2695 (3)	0.057 (3)
C(22)	0.2271 (5)	0.0884 (6)	0.2249 (4)	0.054 (3)
C(24)	0.3755 (5)	0.1493 (6)	0.2660 (3)	0.043 (3)
C(25)	0.3546 (5)	0.0676 (6)	0.3103 (3)	0.047 (3)
C(26)	0.4114 (5)	0.0208 (7)	0.3754 (3)	0.058 (3)
C(30)	0.2344 (5)	0.3433 (7)	-0.0225 (3)	0.054 (3)
C(32)	0.0850 (5)	0.2533 (7)	0.0878 (4)	0.062 (3)
C(34)	0.1519 (5)	0.3172 (6)	0.0098 (3)	0.049 (3)
C(35)	0.0537 (6)	0.3334 (7)	-0.0136 (4)	0.065 (3)
C(36)	-0.0057 (6)	0.3821 (9)	-0.0776 (4)	0.095 (4)
C(40)	0.3057 (6)	0.5451 (6)	0.0613 (3)	0.057 (3)
C(42)	0.2689 (5)	0.4702 (6)	0.2226 (3)	0.050 (3)
C(44)	0.2885 (4)	0.5458 (5)	0.1308 (3)	0.037 (2)
C(45)	0.2676 (5)	0.6453 (6)	0.1656 (3)	0.046 (3)
C(46)	0.2566 (6)	0.7820 (6)	0.1506 (4)	0.072 (3)
Si(1)	0.79455 (13)	0.23808 (18)	0.11040 (9)	0.0512 (7)
F(2)	0.6789 (3)	0.2553 (4)	0.06848 (19)	0.069 (2)
F(3)	0.7662 (3)	0.2697 (4)	0.18231 (19)	0.078 (2)
F(4)	0.8164 (4)	0.3888 (4)	0.1031 (2)	0.094 (2)
F(5)	0.9114 (3)	0.2129 (5)	0.1508 (2)	0.102 (2)
F(6)	0.8252 (3)	0.2066 (5)	0.0392 (2)	0.097 (2)
F(7)	0.7696 (4)	0.0864 (4)	0.1195 (3)	0.106 (2)
O(1)	0.1881 (14)	0.3447 (10)	0.3469 (5)	0.31 (1)
O(2)	0.0513 (12)	0.4893 (15)	0.4632 (12)	0.43 (2)
O(3)	0.0033 (17)	0.4564 (20)	0.3605 (11)	0.45 (2)
O(4)	0.0281 (11)	0.9479 (18)	0.2696 (8)	0.37 (1)
O(5)	0.0207 (10)	0.2130 (20)	0.2706 (6)	0.37 (1)

ion is octahedrally coordinated by four imidazole N atoms and two thioether S atoms of the ligand 1,3-bis(5-methyl-4-imidazolyl)-2-thiopropane. The Ni—N distances are 2.040 (6), 2.044 (6), 2.041 (5) and 2.068 (6) Å. The Ni—S distances are 2.513 (2) and 2.496 (2) Å. The non-coordinating hexafluorosilicate anion is hydrogen bonded to the nitrogen H atoms of the imidazole moieties of the ligands and to the five independent water molecules of crystallization, so forming a two-dimensional network perpendicular to a.

Introduction. Coordination compounds with imidazole- and thioether-containing ligands are important as model systems for type I copper proteins. Recently a new method of synthesizing this kind of ligand has been developed (Bouwman & Driessen, 1988). Among the first ligands of this new series used in the preparation of coordination compounds is the ligand 1,3-bis(5-methyl-4-imidazolyl)-2-thiopropane (sbi). The synthesis and characterization of several transition-metal compounds with this ligand will be described elsewhere (Bouwman, Driessen & Reedijk, 1988). The nickel tetrafluoroborate compound yielded a single crystal suitable for

Table 2. Bond distances (Å) and bond angles (°) for the non-H atoms

Ni—S(8)	2.513 (2)	N(31)—C(35)	1.368 (10)
Ni—S(9)	2.496 (2)	N(33)—C(32)	1.321 (9)
Ni—N(13)	2.040 (6)	N(33)—C(34)	1.393 (9)
Ni—N(23)	2.068 (6)	N(41)—C(42)	1.332 (8)
Ni—N(33)	2.044 (6)	N(41)—C(45)	1.376 (9)
Ni—N(43)	2.041 (5)	N(43)—C(42)	1.333 (9)
S(8)—C(10)	1.795 (8)	N(43)—C(44)	1.370 (7)
S(8)—C(20)	1.806 (7)	C(10)—C(14)	1.504 (10)
S(9)—C(30)	1.809 (7)	C(14)—C(15)	1.352 (9)
S(9)—C(40)	1.799 (7)	C(15)—C(16)	1.494 (11)
N(11)—C(12)	1.349 (9)	C(20)—C(24)	1.500 (10)
N(11)—C(15)	1.366 (10)	C(24)—C(25)	1.346 (9)
N(13)—C(12)	1.307 (9)	C(25)—C(26)	1.485 (9)
N(13)—C(14)	1.377 (9)	C(30)—C(34)	1.496 (10)
N(21)—C(22)	1.338 (10)	C(34)—C(35)	1.361 (11)
N(21)—C(25)	1.372 (10)	C(35)—C(36)	1.486 (12)
N(23)—C(22)	1.324 (9)	C(40)—C(44)	1.520 (9)
N(23)—C(24)	1.394 (9)	C(44)—C(45)	1.351 (9)
N(31)—C(32)	1.336 (10)	C(45)—C(46)	1.484 (9)
Si(1)—F(2)	1.662 (4)	Si(1)—F(5)	1.679 (5)
Si(1)—F(3)	1.669 (4)	Si(1)—F(6)	1.669 (5)
Si(1)—F(4)	1.642 (5)	Si(1)—F(7)	1.667 (5)
S(8)—Ni—S(9)	92.98 (7)	S(9)—Ni—N(43)	81.3 (2)
S(8)—Ni—N(13)	81.6 (2)	N(13)—Ni—N(23)	91.4 (2)
S(8)—Ni—N(23)	82.8 (2)	N(13)—Ni—N(33)	96.0 (2)
S(8)—Ni—N(33)	174.9 (2)	N(13)—Ni—N(43)	170.0 (2)
S(8)—Ni—N(43)	92.2 (2)	N(23)—Ni—N(33)	101.8 (2)
S(9)—Ni—N(13)	91.2 (2)	N(23)—Ni—N(43)	95.6 (2)
S(9)—Ni—N(23)	174.2 (2)	N(33)—Ni—N(43)	89.5 (2)
S(9)—Ni—N(33)	82.6 (2)		
C(10)—S(8)—C(20)	103.2 (4)	N(23)—C(24)—C(25)	110.8 (6)
C(30)—S(9)—C(40)	101.4 (3)	C(20)—C(24)—C(25)	129.3 (6)
C(12)—N(11)—C(15)	108.3 (6)	N(21)—C(25)—C(24)	104.4 (6)
C(12)—N(13)—C(14)	105.1 (5)	C(24)—C(25)—C(26)	133.0 (7)
C(22)—N(21)—C(25)	109.6 (6)	N(21)—C(25)—C(26)	122.6 (6)
C(22)—N(23)—C(24)	105.0 (6)	S(9)—C(30)—C(34)	111.5 (4)
C(32)—N(31)—C(35)	110.2 (7)	N(31)—C(32)—N(33)	109.8 (7)
C(32)—N(33)—C(34)	105.8 (6)	N(33)—C(34)—C(30)	120.0 (6)
C(42)—N(41)—C(45)	108.8 (6)	N(33)—C(34)—C(35)	110.0 (6)
C(42)—N(43)—C(44)	105.1 (5)	C(30)—C(34)—C(35)	130.1 (6)
S(8)—C(10)—C(14)	114.4 (5)	N(31)—C(35)—C(34)	104.3 (7)
N(11)—C(12)—N(13)	111.0 (6)	N(31)—C(35)—C(36)	123.6 (8)
N(13)—C(14)—C(10)	120.9 (6)	C(34)—C(35)—C(36)	132.2 (7)
N(13)—C(14)—C(15)	110.9 (6)	S(9)—C(40)—C(44)	113.1 (4)
C(10)—C(14)—C(15)	128.2 (7)	N(41)—C(42)—N(43)	110.5 (5)
N(11)—C(15)—C(14)	104.6 (6)	N(43)—C(44)—C(40)	121.0 (5)
N(11)—C(15)—C(16)	122.4 (6)	N(43)—C(44)—C(45)	110.9 (5)
C(14)—C(15)—C(16)	133.0 (7)	C(40)—C(44)—C(45)	128.0 (5)
S(8)—C(20)—C(24)	113.4 (4)	N(41)—C(45)—C(44)	104.7 (5)
N(21)—C(22)—N(23)	110.2 (7)	N(41)—C(45)—C(46)	122.8 (6)
N(23)—C(24)—C(20)	119.9 (5)	C(44)—C(45)—C(46)	132.6 (6)
F(2)—Si(1)—F(3)	92.1 (2)	F(3)—Si(1)—F(7)	89.5 (3)
F(2)—Si(1)—F(4)	91.5 (3)	F(4)—Si(1)—F(5)	91.1 (3)
F(2)—Si(1)—F(5)	176.9 (2)	F(4)—Si(1)—F(6)	91.1 (3)
F(2)—Si(1)—F(6)	89.0 (2)	F(4)—Si(1)—F(7)	177.9 (4)
F(2)—Si(1)—F(7)	87.7 (3)	F(5)—Si(1)—F(6)	89.2 (2)
F(3)—Si(1)—F(4)	88.6 (2)	F(5)—Si(1)—F(7)	89.8 (3)
F(3)—Si(1)—F(5)	89.8 (2)	F(6)—Si(1)—F(7)	90.7 (3)
F(3)—Si(1)—F(6)	178.9 (2)		

X-ray study. Structure determination of this crystal showed the presence of an SiF₆ anion, while the bulk clearly showed the tetrafluoroborate vibrations in the IR spectrum. Apparently, BF₄ destruction of the glass yielded the Ni(sbi)₂(SiF₆) crystal. A similar experience was reported by Gagné, McCool & Marsh (1980) and Driessen, Hulsbergen, Vermin & Reedijk (1982). Subsequently a larger batch for spectroscopic investigation of the title compound was obtained by using SiF₆ anions.

Experimental. Data were collected (de Boer & Duisenberg, 1984) for a violet crystal (0.20 × 0.23 ×

0.28 mm), mounted on top of a glass fibre, with an Enraf-Nonius CAD-4F diffractometer using Ni-filtered $\text{Cu K}\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the SET4 setting angles of 12 reflections with $20 < 2\theta < 32^\circ$. Intensity data for 4668 reflections (h 0:15, k 0:11, l -23:22; $\theta < 60^\circ$) were collected in the $\omega/2\theta$ scan mode with $\Delta\omega = 0.40 + 0.15 \tan\theta^\circ$. Three reference reflections (200, 020, 004) measured every hour of X-ray exposure time indicated no decay over 86 h of X-ray exposure. The intensity data were corrected for Lp but not for absorption in view of a less than 10% variation of the intensity data for a $360^\circ \psi$ scan of the close-to-axial 202 reflection. Standard deviations in the intensities based on counting statistics were increased according to an analysis of the excess variance of the reference reflections: $\sigma^2(I) = \sigma_{\text{cs}}^2(I) + (0.02I)^2$ following McCandlish, Stout & Andrews (1975), resulting in 2922 reflections with $I > 2.5\sigma(I)$ and $\sum\sigma/\sum I = 0.025$. The space group was derived from the observed systematic absences. The structure was solved by direct methods (SHELXS86; Sheldrick, 1986) and refined on F with SHELX76 (Sheldrick, 1976). Five water molecules of crystallization were identified in a difference map. H atoms (excluding those of the water molecules) were introduced on calculated positions [$d(\text{C}, \text{N}-\text{H}) = 0.98 \text{ \AA}$] and refined with fixed geometry with respect to their carrier atoms. Refinement with weights based on $w = 1/\sigma^2(F)$ converged at $R = 0.059$ [$wR = 0.058$; $S = 2.07$; 402 parameters; $(\Delta/\sigma)_{\text{ave}} = 0.05$, $(\Delta/\sigma)_{\text{max}} = 0.7$ for O(2) and O(3)]. A final difference Fourier map did not show residual peaks outside $\pm 0.62 \text{ e \AA}^{-3}$. The maxima are in the region of the water molecules and probably indicate some positional disorder of the water molecules (in addition to relatively high thermal motion). Scattering factors of Cromer & Mann (1968) and anomalous-dispersion terms from Cromer & Liberman (1970) were used. Final parameters are given

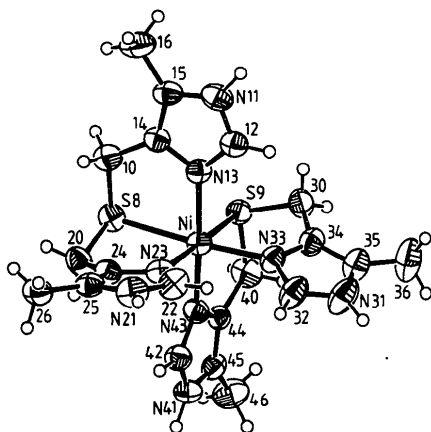


Fig. 1. Thermal-motion ellipsoid plot of the $[\text{Ni}(\text{sbi})_2]^{2+}$ cation. The surfaces have been drawn at the 40% probability level for non-hydrogen atoms and artificially small for H atoms.

Table 3. Geometry for hydrogen bonds originating from the cation

$D-H \cdots A$	$D \cdots A$ (Å)	$H \cdots A$ (Å)	$\angle D-H \cdots A$ ($^\circ$)
N(11)-H(11) \cdots F(2 ⁱ)	2.814 (7)	1.869 (7)	160.9 (8)
N(11)-H(11) \cdots F(6 ⁱ)	3.060 (8)	2.327 (8)	131.0 (7)
N(11)-H(11) \cdots F(7 ⁱ)	3.187 (9)	2.468 (9)	129.9 (5)
N(21)-H(21) \cdots O(4 ⁱⁱ)	3.32 (2)	2.56 (2)	134.3 (7)
N(21)-H(21) \cdots F(3 ⁱⁱⁱ)	2.905 (7)	2.196 (7)	128.0 (6)
N(21)-H(21) \cdots F(4 ⁱⁱⁱ)	3.195 (8)	2.386 (7)	139.5 (6)
N(31)-H(31) \cdots F(6 ^{iv})	2.814 (8)	1.890 (8)	156.0 (7)
N(41)-H(41) \cdots F(3 ^v)	2.763 (7)	1.874 (7)	149.2 (6)
N(41)-H(41) \cdots F(7 ^v)	3.355 (9)	2.507 (9)	144.6 (5)

Symmetry code: (i) $1-x, -y, -z$; (ii) $x, y-1, z$; (iii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $x-1, y, z$; (v) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

in Table 1.* Calculations were carried out on a MicroVAX II. The program PLATON (Spek, 1982) was used for the calculation of the geometrical data and the thermal-motion ellipsoid plot.

Discussion. The monoclinic unit cell of the title compound contains four $\text{Ni}(\text{sbi})_2$ cations, four SiF_6 anions and 20 water molecules of crystallization. Data on the geometry have been collected in Table 2. A thermal-motion ellipsoid plot of the cation along with the adopted numbering scheme is presented in Fig. 1.

The Ni^{II} ion is octahedrally surrounded by four azole N atoms and two thioether S atoms of two sbi ligands. The cation has an approximate non-crystallographic twofold axis along the bisector of the S(8)-Ni-S(9) bond angle. Distances from the central metal ion to the donor atoms lie in the broad range of 2.040 (6)-2.513 (2) Å. The coordination geometry of the Ni ion deviates from an ideal octahedron; the angles vary from 81.3 (2) $^\circ$ for S(9)-Ni-N(43) to 101.8 (2) $^\circ$ for N(23)-Ni-N(33). Surprisingly, the two thioether S atoms are *cis*-coordinated. The geometry of the Ni ion is comparable with the geometry in $[\text{Ni}(1,7\text{-diazabicyclo[2.2.1]heptane})_2]^{2+}$ (Hart, Boeyens & Hancock, 1983) with an N_4S_2 chromophore and the sulfurs *cis*-coordinated. Another structure with *cis*-coordinated S atoms is $[\text{Ni}(\text{dsbd})]^{2+}$, but here the *cis*-coordination is imposed by the hexadentate dsbd [= 1,12-bis(3,5-dimethyl-1-pyrazolyl)-2,11-diaza-5,8-dithiadodecane] ligand (Paap, 1987). In this compound, the Ni-N distances are somewhat longer, and the Ni-S distances slightly shorter, thus forming a more regular octahedron.

The sbi molecule is not only coordinated to the Ni^{II} ion but is also involved in mostly bifurcated and trifurcated hydrogen bonds (Table 3), through the H atoms on the non-coordinating azole nitrogens, to the fluorines of the hexafluorosilicate anion, with N \cdots F contacts of 2.763 (7)-3.355 (9) Å. The SiF_6 anion is

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and geometrical data concerning H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51046 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

also hydrogen bonded to the five water molecules with F...O contacts in the range 2.60 (1)–3.07 (2) Å. The water molecules of crystallization are both hydrogen bonded among themselves and to N and F atoms of the cation and anion respectively. A more detailed analysis of the hydrogen-bond scheme is hampered by the fact that H atoms for the water molecules could not be identified owing to their high thermal motion. In addition to the observed dynamic disorder of the water molecules there are also indications for some static disorder. The presence of relatively strong hydrogen bridges was also inferred from the infrared spectrum of this compound, where strong bands in the 2500–3200 cm⁻¹ region occurred.

The bond angles around the thioether S atoms are close to 100°, which indicates that one of the lone electron pairs on each of these sulfurs is directed towards the Ni. The relatively long Ni–S(8) and Ni–S(9) contacts of 2.513 (2) and 2.496 (2) Å respectively suggest weak bonds, but are rather normal for Ni–S(thioether) distances (Setzer, Ogle, Wilson & Glass, 1983; Hill & Hope, 1974). The imidazole rings are planar within 0.008 Å.

The geometry of the SiF₆ anion is the same as reported for [Cu(H₂O)₆]₂SiF₆ (Ray, Zalkin & Templeton, 1973).

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1-(2,2':6',2''-Terpyridine)-2-methyl-3-trimethylsilyl-2,3-dicarba-1-stanna-closo-heptaborane(6)–2,2':6',2''-Terpyridine

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Abstract. C₂₁H₂₇B₄N₃SiSn.C₁₅H₁₁N₃, *M_r* = 744.82, triclinic, *P*1̄, *a* = 8.304 (1), *b* = 13.161 (2), *c* = 17.493 (3) Å, *α* = 105.74 (1), *β* = 91.96 (1), *γ* = 92.84 (1)°, *V* = 1835.5 (5) Å³, *Z* = 2, *D_x* = 1.35 g cm⁻³, *λ*(Mo *Kα*) = 0.71069 Å, *μ* = 7.61 cm⁻¹, *F*(000) = 760, *T* = 295 K. Final *R* = 0.040 for 5550 observed reflections. The Lewis base 2,2':6',2''-terpyridine forms a donor–acceptor complex (II) with closo-1-Sn-2-(Me)-3-(SiMe₃)-2,3-C₂B₄H₄ (I). This complex (II) has a distorted pentagonal bipyramidal geometry

with the Sn atom occupying an apical position. Specifically, the complex shows extreme displacement of the tin from the centroidal position above the C₂B₃ face of the carborane cage. The Sn–C(cage) bond distances are about 0.43 Å longer than the Sn–B(unique) distance which is definitely shorter than the other two Sn–B distances by about 0.168 Å. The slippage is such that the Sn^{II} atom could be considered as η¹-bonded to the unique B of the carborane cage. The apical Sn is also bonded strongly to two consecutive N atoms and weakly to the third N atom of the terpyridine rings on the opposite side of the C₂B₃ ring.

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