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Solid-State Structures of [HB(3-Bu^t-5-Mepz)$_3$]SnCl and [HB(3-Bu^{t}pz)$_3$]CdCl (pz = Pyrazolyl Ring). Stereochemical Influence of a Central Atom Lone Pair

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The complexes [HB(3-Bu^t-5-Mepz)$_3$]SnCl and [HB(3-Bu^{t}pz)$_3$]CdCl(pz = pyrazolyl ring) have been prepared and their solid-state structures determined crystallographically. The two structures allow a determination of the impact of the lone pair on ligand geometry and on the metal–donor atom distances for two metals with equivalent ionic radii. The geometry about tin in [HB(3-Bu^t-5-Mepz)$_3$]SnCl is best described as a distorted trigonal bipyramid with the chloride ligand in an axial site and the tin lone pair occupying an equatorial position. The axial bond lengths are long, especially the Sn–Cl bond length at 2.601(1) Å. The structure of [HB(3-Bu^{t}pz)$_3$]CdCl is a distorted tetrahedron with nearly 3-fold rotational symmetry. The Cd–Cl bond length is 2.355(2) Å, much shorter than the Sn–Cl distance in [HB(3-Bu^t-5-Mepz)$_3$]SnCl. The lone pair on the central tin atom that is not present in the cadmium complex is stereochemically active and has the effect of increasing bond lengths, especially for the axial ligands. Crystal data: [HB(3-Bu^t-5-Mepz)$_3$]SnCl·$^1/_2$C$_6$H$_6$, monoclinic, $P2_1/n$, a = 9.656(3) Å, b = 17.844(4) Å, c = 18.123(5) Å, β = 99.73(2)°, V = 3077.7(14) Å^3, Z = 4, $R(F)$ = 4.19%; [HB(3-Bu^{t}pz)$_3$]CdCl, orthorhombic, *Pnma*, a = 16.137(5) Å, b = 15.985(4) Å, c = 9.885(3) Å, V = 2549.9(13) Å^3, Z = 4, $R(F)$ = 3.90%.

Introduction

We have recently been interested in the preparation and characterization of complexes of the early transition metals,[1] the f-block elements,[2] and the post transition metals[3] using the versatile poly(pyrazolyl)borate ligand system.[4] Although these metals represent a diverse series of elements, of overriding importance in all of this chemistry are the size of the metal atom and the steric influences of these bulky ligands. With the post transition metals, another important factor can be the presence of a lone pair of electrons on the metal.

VSEPR theory, in which both lone pairs and bond pairs of electrons occupy vertices of regular polyhedra, is generally used to predict the stereochemistry of post transition metal complexes.[5] We felt that, by using bulky poly(pyrazolyl)borate ligands, it would be possible to prepare complexes of tin(II) and/or lead(II) with a stereochemically inactive lone pair. To date, this approach has not proven successful in the chemistry of tin(II),[3d] but the complex [HB(3,5-Me$_2$pz)$_3$]$_2$Pb (pz = pyrazolyl ring) has a trigonally distorted octahedral structure in which the lone pair is clearly stereochemically inactive.[3e] The analogous tin(II) complex is five-coordinate with a stereochemically active lone pair.[6]

Another possible route to a complex of tin(II) with a stereochemically inactive lone pair is the preparation of four-coordinate complexes using one tris(pyrazolyl)borate ligand containing bulky *tert*-butyl substituents at the 3-position of the pyrazolyl rings. It has been shown that these ligands force tetrahedral geometry on complexes of a number of metals.[7] Reported here are the preparation and solid-state structure of [HB(3-Bu^t-5-Mepz)$_3$]SnCl. For comparative purposes, we have also prepared [HB(3-Bu^{t}pz)$_3$]CdCl and determined its solid-state structure. The ionic radius of cadmium(II) is equivalent to that of tin(II),[8] but the lone pair on the central atom is not present in the cadmium complex. The two structures allow a determi-

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Table I. Crystallographic Data for the Structural Analyses

	$[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$	$[HB(3\text{-}Bu^tpz)_3]CdCl$
formula	$C_{27}H_{43}N_6BClSn$	$C_{21}H_{34}N_6BCdCl$
fw	617	529
cryst system	monoclinic	orthorhombic
space group	$P2_1/n$	*Pnma*
a, Å	9.656(3)	16.137(5)
b, Å	17.844(4)	15.985(4)
c, Å	18.123(5)	9.885(3)
β, deg	99.73(2)	
V, Å^3	3077.7(14)	2549.9(13)
Z	4	4
T, °C	23	26
λ, Å	0.710 73	0.71073
ρ_{calc}, g cm^{-3}	1.335	1.379
abs coeff, cm^{-1}	9.44	9.80
$R(F)$, %	4.19	3.90
$R_w(F)$, %	5.18	4.30

nation of the impact of the lone pair on ligand geometry and on the metal-donor atom distances.

Experimental Section

General Procedures. Operations were carried out under a nitrogen atmosphere by using either standard Schlenk techniques or Vacuum Atmospheres dryboxes. All solvents were dried, degassed, and distilled prior to use. The ^1H NMR spectra were recorded on a Bruker AM-300 or AM-400 spectrometer using a 5-mm broad-band probe. Proton chemical shifts are reported in ppm downfield from TMS using the solvent $CDCl_3$ ($\delta_H = 7.24$ ppm) as an internal standard. The ^{113}Cd NMR spectra were recorded on a Bruker AM-500 spectrometer using a 5-mm broad-band probe, and the shifts are reported in ppm versus 0.1 M $Cd(ClO_4)_2$. The ^{119}Sn NMR spectra were recorded on a Bruker AM-400, and the shifts are reported in ppm with reference to $SnMe_4$. All NMR spectra were recorded in $CDCl_3$. Mass spectra were obtained on a VG 70SQ mass spectrometer. The clusters assigned to specific ions show the appropriate isotopic patterns as calculated for the atoms present. Elemental analyses were performed by Robertson Laboratories, Inc., and by the Microanalytical Laboratories of the University of Alberta Chemistry Department. $K[HB(3\text{-}Bu^tpz)_3]$,[7a] $Tl[HB(3\text{-}Bu^tpz)_3]$,[7a] and $K[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]$[9] were prepared by the published methods.

[Hydrotris(3-*tert*-butyl-5-methyl-1-pyrazolyl)borato]chlorotin(II), $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl$. To a stirred suspension of $SnCl_2$ (0.195 g, 1.03 mmol) in CH_2Cl_2 (20 mL) was slowly added $K[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]$ (0.470 g, 1.02 mmol) in CH_2Cl_2 (10 mL). The mixture was stirred for 6 h, and the CH_2Cl_2 was removed under vacuum. The product was extracted with toluene (10 mL), and the extract was filtered. A colorless crystalline solid was obtained by cooling the toluene solution to −30 °C overnight (0.405 g, 0.706 mmol, 69%). Single crystals were grown by allowing a benzene solution of the material to slowly evaporate (0.281 g, 0.456 mmol, 44%).

^1H NMR: δ 5.99 (s, 1, pz 4-H), 2.43 (s, 3, CH_3), 1.43 (s, 9, $(CH_3)_3C$). ^{119}Sn NMR: δ −577.9. Mass spectrum: m/z 542 (M^+ − Cl). Anal. Calcd for $C_{27}H_{43}N_6BSnCl$($[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$, confirmed by ^1H NMR): C, 52.59; H, 7.03; N, 13.63. Found: C, 52.78; H, 7.24; N, 13.56.

[Hydrotris(3-*tert*-butyl-1-pyrazolyl)borato]chlorocadmium(II), $[HB(3\text{-}Bu^tpz)_3]CdCl$. Method A. $CdCl_2$ (0.25 g, 1.4 mmol) was suspended in THF (35 mL). $K[HB(3\text{-}Bu^tpz)_3]$ (6.8 mL of 0.2 M in THF) was added via syringe with rapid stirring. This solution was stirred for 4 h, and the THF was removed under vacuum. The product was extracted with benzene (30 mL), and the extract was filtered. The benzene was removed under vacuum to reveal a white solid (0.49 g, 0.93 mmol, 66%).

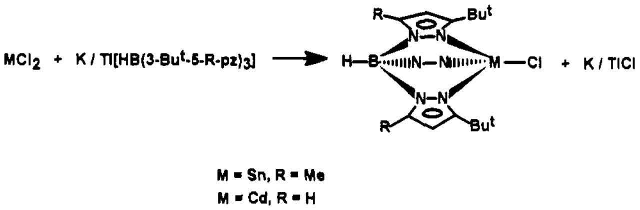
Method B. A solution of $Tl[HB(3\text{-}Bu^tpz)_3]$ in THF (25 mL) was added dropwise to a stirred suspension of $CdCl_2$ (0.25 g, 1.4 mmol) in THF (10 mL). The resulting mixture was stirred an additional 4 h, and the THF was removed under vacuum. The product was extracted with benzene (25 mL), and the extract was filtered. The benzene was removed under vacuum to leave a white solid (0.45 g, 0.85 mmol, 61%); dec pt 210–214 °C.

^1H NMR: δ 7.56, 6.05 (d, d; 1, 1; J = 2.2, 2.3; pz 4,5-H), 1.38 (s, 9, $(CH_3)_3C$). ^{113}Cd NMR: δ 330.1. Mass spectrum: m/z 529 (M^+ − H), 407 (M^+ − Bu^tpz). Anal. Calcd for $C_{21}H_{34}N_6BCdCl$: C, 47.65; H, 6.49; N, 15.88. Found: C, 47.81; H, 6.59; N, 15.89.

Crystallographic Analyses. Crystal, data collection, and refinement parameters are collected in Table I. Colorless crystals, grown from benzene for $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$ and from toluene/hexane for $[HB(3\text{-}Bu^tpz)_3]CdCl$, were mounted on glass fibers with epoxy cement. The unit-cell parameters were obtained from the least-squares fit of 25 reflections (20° < 2θ < 25°). For the Cd complex, the orthorhombic space groups *Pnma* and $Pn2_1a$ were indicated by the systematic absences in the data. The former was chosen to accommodate the molecular mirror plane which was aligned perpendicular to the b axis. Three standard reflections monitored every 197 reflections showed insignificant variations. Empirical corrections for absorption were applied (216 Ψ-scan reflections, pseudoellipsoid model). The metal atom was located from Patterson syntheses. All hydrogen atoms were included as idealized isotropic contributions (d(CH) = 0.960 Å, U = 1.2U of attached atom). All non-hydrogen atoms were refined with anisotropic thermal parameters. All computer programs and the sources of the scattering factors are contained in the SHELXTL-PLUS program library (4.2) (G. Sheldrick, Siemens Corp., Madison, WI). Tables II and III contain the atomic coordinates and isotropic thermal parameters for $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$ and $[HB(3\text{-}Bu^tpz)_3]CdCl$, respectively.

Results and Discussion

The new complexes were prepared by the reaction of either the thallium or the potassium salt of the ligand with the respective MCl_2 derivative.



The cadmium complex is air stable in the solid phase, and in solution, the tin complex slowly decomposes in air. They are soluble in aromatic or halocarbon solvents but only sparingly soluble in hydrocarbons.

$[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$. Crystals suitable for an X-ray investigation were obtained from benzene. The ligand having the 5-methyl substituent was used because suitable crystals of $[HB(3\text{-}Bu^tpz)_3]SnCl$ could not be obtained.

Figure 1 shows an ORTEP drawing of the molecule, and selected bond distances and angles are shown in Table IV. The large displacement of the chloride ligand from the potential 3-fold symmetry axis of the hydrotris(pyrazolyl)borate ligand clearly demonstrates that the lone pair on tin is stereochemically active. The structure is best described as a severely distorted trigonal bipyramid with N(5) and the chloride ligand in the axial sites and N(1), N(3), and the tin lone pair occupying the equatorial sites. The axial Sn–N(5) bond length of 2.549(3) Å is considerably longer than the two equatorial Sn–N distances (average = 2.281 Å). The axial N(5)–Sn–Cl bond angle is 154.3(1)°, and the equatorial N(1)–Sn–N(3) bond angle is 91.2(1)°. Overall, the structure is very similar to that determined previously for $[HB(3,5\text{-}Me_2pz)_3]SnCl$;[6] the effects of the bulky *tert*-butyl groups are not pronounced.

Compression of the axial and equatorial bond angles and shorter equatorial bond distances are general features of four-coordinate, pseudo trigonal bipyramidal tin(II) structures.[10] For example, in

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Table II. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$) for $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$

	x	*y*	*z*	*U*(eq)[a]
Sn	1912.8(3)	1857.7(2)	1886.2(2)	40.8(1)
Cl	−588(1)	1350(1)	1368(1)	65(1)
N(1)	1229(4)	1858(2)	3036(2)	39(1)
N(2)	985(3)	2551(2)	3335(2)	33(1)
N(3)	976(4)	3015(2)	1628(2)	39(1)
N(4)	775(4)	3454(2)	2231(2)	34(1)
N(5)	3652(3)	2787(2)	2598(2)	34(1)
N(6)	3098(3)	3268(2)	3061(2)	31(1)
B	1502(5)	3301(3)	3040(3)	32(2)
C(1)	−28(6)	3087(3)	4399(3)	59(2)
C(2)	367(5)	2453(3)	3935(3)	40(2)
C(3)	202(5)	1700(3)	4030(3)	48(2)
C(4)	755(5)	1344(3)	3462(3)	43(2)
C(5)	833(6)	498(3)	3377(3)	58(2)
C(6)	1367(9)	148(4)	4136(4)	95(3)
C(7)	−639(7)	196(3)	3084(5)	91(3)
C(8)	1818(7)	260(3)	2846(4)	84(3)
C(11)	−416(6)	4656(3)	2475(3)	53(2)
C(12)	11(5)	4064(3)	1975(3)	41(2)
C(13)	−263(5)	4019(3)	1211(3)	48(2)
C(14)	345(5)	3369(3)	1002(3)	45(2)
C(15)	327(6)	3107(4)	204(3)	61(2)
C(16)	1365(6)	2483(4)	137(3)	85(3)
C(17)	696(9)	3789(5)	−255(3)	100(4)
C(18)	−1149(6)	2842(4)	−126(4)	80(3)
C(21)	3899(6)	4253(3)	4033(3)	54(2)
C(22)	4140(4)	3689(3)	3455(3)	35(1)
C(23)	5377(5)	3481(3)	3235(3)	39(2)
C(24)	5044(4)	2918(2)	2696(2)	31(1)
C(25)	6008(4)	2503(3)	2266(3)	39(2)
C(26)	7474(6)	2839(4)	2431(4)	83(3)
C(27)	6084(7)	1686(3)	2478(4)	71(3)
C(28)	5494(6)	2571(4)	1425(3)	69(2)
C(1BZ)	3586(10)	124(8)	−83(9)	124(5)
C(2BZ)	4505(19)	402(5)	502(6)	109(5)
C(3BZ)	5837(17)	284(6)	597(5)	111(5)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table III. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$) for $[HB(3\text{-}Bu^tpz)_3]CdCl$

	x	*y*	*z*	*U*(eq)[a]
Cd	3536(1)	7500	847(1)	40(1)
Cl(1)	4940(1)	7500	199(2)	81(1)
B(1)	1643(5)	7500	1637(8)	42(3)
N(1)	2879(2)	6522(3)	2080(3)	38(1)
N(2)	2048(2)	6713(3)	2231(4)	41(1)
N(3)	2460(3)	7500	−575(5)	43(2)
N(4)	1716(3)	7500	81(6)	41(2)
C(1)	1702(3)	6128(4)	3016(5)	54(2)
C(2)	2281(3)	5552(4)	3383(5)	57(2)
C(3)	3012(3)	5821(3)	2782(4)	41(2)
C(4)	3869(3)	5429(4)	2875(5)	52(2)
C(5)	4138(4)	5110(5)	1502(7)	108(4)
C(6)	3850(5)	4717(5)	3908(8)	94(3)
C(7)	4490(3)	6074(4)	3386(6)	64(2)
C(8)	1101(5)	7500	−826(8)	54(3)
C(9)	1438(5)	7500	−2097(9)	65(3)
C(10)	2296(5)	7500	−1917(7)	50(3)
C(11)	2976(6)	7500	−2955(7)	66(3)
C(12)	3519(4)	6718(5)	−2786(6)	88(3)
C(13)	2579(7)	7500	−4377(8)	105(5)
Hb(1)	984(39)	7500	1987(62)	54(20)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

$[HB(3,5\text{-}Me_2pz)_3]SnCl$ and $[B(pz)_4]_2Sn$, respectively, the axial Sn–N bond distances are 2.491(2) and 2.48 Å (average value), the average equatorial bond distances are 2.21 and 2.26 Å, the equatorial N–Sn–N bond angles are 76.56(9) and 83(1)°, and the axial N–Sn–Cl(N) bond angles are 156.91(7) and 150.1(1)°.[6,3d]

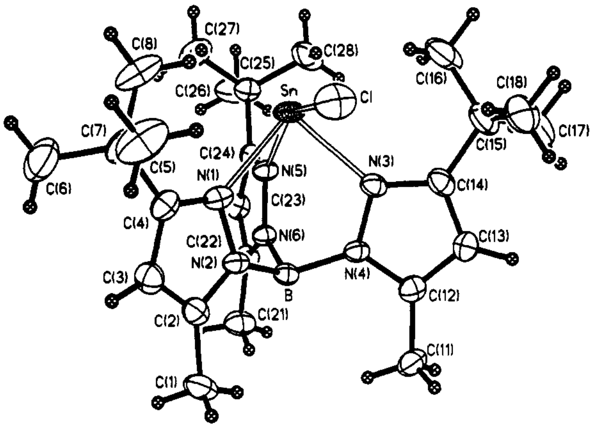

Figure 1. ORTEP drawing of $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl$.

Table IV. Selected Bond Distances and Angles

$[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl\cdot{}^1/_2C_6H_6$		$[HB(3\text{-}Bu^tpz)_3]CdCl$	
	Bond Distances (Å)		
Sn–Cl	2.601(1)	Cd–Cl	2.355(2)
Sn–N(1)	2.290(4)	Cd–N(1)	2.248(4)
Sn–N(3)	2.272(4)	Cd–N(3)	2.233(5)
Sn–N(5)	2.549(3)		
N(1)–N(2)	1.386(5)	N(1)–N(2)	1.384(5)
N(3)–N(4)	1.385(5)	N(3)–N(4)	1.365(8)
N(5)–N(6)	1.371(5)		
N(2)–B	1.555(6)	N(2)–B	1.534(6)
N(4)–B	1.540(6)	N(4)–B	1.543(10)
N(6)–B	1.536(6)		
	Bond Angles (deg)		
Cl–Sn–N(1)	86.3(1)	Cl–Cd–N(1)	127.0(1)
Cl–Sn–N(3)	85.9(1)	Cl–Cd–N(3)	125.2(1)
Cl–Sn–N(5)	154.3(1)		
N(1)–Sn–N(3)	91.2(1)	N(1)–Cd–N(1a)	88.1(2)
N(1)–Sn–N(5)	78.8(1)	N(1)–Cd–N(3)	88.6(1)
N(3)–Sn–N(5)	73.7(1)		
Sn–N(1)–N(2)	116.8(3)	Cd–N(1)–N(2)	111.2(3)
Sn–N(3)–N(4)	117.1(3)	Cd–N(3)–N(4)	112.6(4)
Sn–N(5)–N(6)	115.2(2)		
B–N(2)–N(1)	123.2(4)	B–N(2)–N(1)	123.6(4)
B–N(4)–N(3)	123.3(3)	B–N(4)–N(3)	122.8(5)
B–N(6)–N(5)	120.2(3)		
N(2)–B–N(4)	111.0(3)	N(2)–B–N(2a)	110.1(5)
N(2)–B–N(6)	109.7(3)	N(2)–B–N(4)	110.4(4)
N(4)–B–N(6)	108.9(4)		

The Sn–Cl bond length is long, at 2.601 Å, and very similar to that found in $[HB(3,5\text{-}Me_2pz)_3]SnCl$ (2.629(1) Å). For comparison, the Sn–Cl bond length in $[H_2B(pz)_2]SnCl$ is 2.485(3) Å[3d] and in [1,4-dioxane]$SnCl_2$ it is 2.474(2) Å.[10b]

It has been shown earlier with other poly(pyrazolyl)borate complexes of tin(II) that the coordination environment of the tin can be correlated with ^{119}Sn chemical shift values.[11] For a number of [poly(pyrazolyl)borato]chlorotin(II) complexes with N_3Cl donor atom sets, the solution chemical shift range is −528 to −569 ppm. The chemical shift for $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl$ is −577.9 ppm. For a structure in which the lone pair was stereochemically inactive, a significantly different chemical shift would be expected, as we have observed with analogous lead complexes using ^{207}Pb NMR.[3e] Thus, the solution structure of $[HB(3\text{-}Bu^t\text{-}5\text{-}Mepz)_3]SnCl$ is likely the same as that observed in the solid phase.

$[HB(3\text{-}Bu^tpz)_3]CdCl$. Figure 2 shows an ORTEP drawing of the analogous cadmium complex, and selected bond distances and angles are shown in Table IV. The molecule has a crystallographically imposed mirror plane and is close to having 3-fold rotational symmetry with the Cd–N(1) and Cd–N(3) distances differing by only 0.015 Å and the N(1)–Cd–N(1a) and N(1)–Cd–N(3) angles differing by only 0.5°. As expected for

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(11) Reger, D. L. *Synlett* **1992**, 469.

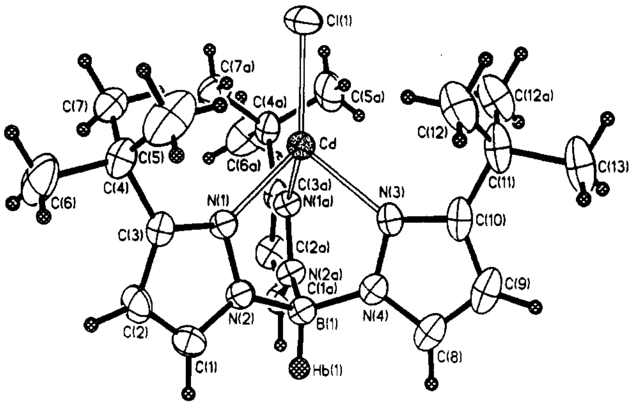

Figure 2. ORTEP drawing of [HB(3-Bu^{t}pz)$_3$]CdCl.

this molecule, which lacks the lone pair present in the tin analog, the chlorine atom is located on this nearly 3-fold axis (the B···Cd–Cl angle is 178.6(2)°).

In comparing the two structures, we do not expect the 5-methyl groups present in the tin compound to have an important influence on metal–ligand bond distances. In the closest set of matched structures available in the literature having these two ligands, [HB(3-Bu^{t}pz)$_3$]CoNCS[7a] and [HB(3-Bu^t-5-Mepz)$_3$]CoO$_2$,[7e] the Co–N bond distances differ by only 0.020 Å, with the latter having the shorter distances.

Clearly, the stereochemically active lone pair present on the tin atom causes significant differences in the geometric arrangement of the ligands. The differences in metal–ligand bond lengths are also large even though the ionic radii of the two metals are the same.[8] The average Sn–N distance is 2.37 Å and the average Cd–N distance is 2.24 Å, a difference of 0.13 Å. The Sn–Cl bond is 0.246 Å longer than the Cd–Cl bond. Thus, the influence of the lone pair on tin and the subsequent changes in geometry cause a substantial increase in the metal–ligand bond lengths when compared to those of the cadmium analog. This bond lengthening is most pronounced with the axial ligands.

It has been suggested previously that metal–ligand distances are shorter in lead(II) structures where the lone pair is stereochemically active.[12] We have previously observed that the average Pb–N distances in [HB(pz)$_3$]$_2$Pb, a six-coordinate compound with a stereochemically active lone pair, and [HB(3,5-Me$_2$pz)$_3$]$_2$Pb, a six-coordinate compound with a stereochemically inactive lone pair, are essentially the same, 2.607(14) and 2.610(5) Å, respectively.[3e] The bond lengths in these two lead complexes are considerably longer than the average Pb–N distance of 2.458(17) Å in [B(pz)$_4$]$_2$Pb,[3e] a compound with a four-coordinate structure similar to that of [HB(3-Bu^t-5-Mepz)$_3$]SnCl. It appears that the main factor influencing bond distances in these lead structures is coordination number.

The major conclusion from the two four-coordinate structures reported here is that the effect of the stereochemically active lone pair in [HB(3-Bu^t-5-Mepz)$_3$]SnCl compared with [HB(3-Bu^{t}pz)$_3$]CdCl is to *increase* bond lengths, especially for the axial ligands. This increase in bond lengths appears not to arise from intramolecular steric effects but is to be associated with the influence of the lone pair that changes the overall geometry of the tin(II) complex. A second result is that it has not been possible, to date, to prepare a tin(II) poly(pyrazolyl)borate compound that has a stereochemically inactive lone pair.

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Supplementary Material Available: Tables of complete data collection information, bond distances, bond angles, anisotropic thermal parameters, and positional parameters of H atoms (11 pages). Ordering information is given on any current masthead page.

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[12] Hancock, R. D.; Shaikjee, M. S.; Dobson, S. M., Boeyens, J. C. A. *Inorg. Chim. Acta* **1988**, *154*, 229.