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X-ray Crystal and Molecular Structure of Tris[hydridotris(pyrazol-1-yl)borato]ytterbium(III), $\mathrm{Yb(HBPz_3)_3}$

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The crystal and molecular structure of tris[hydridotris(pyrazol-1-yl)borato]ytterbium(III) has been determined by single-crystal X-ray diffraction techniques. The crystals of $\mathrm{Yb(HBPz_3)_3}$ are orthorhombic, space group $P2_12_12_1$, with unit cell constants a = 13.729 (2) Å, b = 21.465 (3) Å, c = 11.461 (1) Å, and Z = 4. The structure was solved by the heavy-atom method, and full-matrix least-squares refinement with 2662 unique reflections ($I_o \geq 2\sigma(I_o)$) led to final discrepancy indexes of R = 0.038 and R_w = 0.048. The ytterbium is eight-coordinate with bicapped-trigonal-prismatic coordination geometry. Two tridentate ligands each span one height and cap one rectangular face of the prism. The bidentate ligand spans the remaining prism height, and the uncoordinated pyrazolyl group is directed away from the ytterbium and the "uncapped" rectangular face of the coordination polyhedron. The molecule possesses approximate C_s symmetry. The ytterbium, the three boron atoms, and the three pyrazolyl groups are essentially coplanar. The Yb–N bond lengths range from 2.401 (8) to 2.601 (8) Å, the longest bond lengths being associated with the capping nitrogen atoms.

Introduction

The coordination complexes of the lanthanide metals continue to provide a fascinating and fertile field of research to structural chemists. The lanthanide(III) ions characteristically have high and variable coordination numbers and consequently exhibit variable coordination geometries as well.[1-4] It is then not surprising that the details of the structural intricacies, in most instances, are only revealed by single-crystal X-ray structural analysis.

Although the volume of structural data on lanthanide complexes is large and rapidly expanding, a review of the literature shows a dearth of information on complexes containing three potentially tridentate ligands. The solid-state structures of tris(terpyridyl),[5] tris(dipicolinate),[6] and tris(oxydiacetate)[7] complexes of the lanthanides have been determined. In each case the ligands are of the "linear" type (the central metal atom and the three ligating atoms being essentially coplanar), and the lanthanide(III) ion is nine-coordinate with tricapped-trigonal-prismatic, TCTP, geometry, the preferred coordination polyhedron for nine-coordination.[8]

The present work was undertaken to establish unequivocally the solid-state structure of a tris[hydridotris(pyrazol-1-yl)borato]lanthanide(III) complex, $\mathrm{Ln(HBPz_3)_3}$,[9] where the $\mathrm{HBPz_3^-}$ ligand is potentially tridentate but now in a "tripod"-like fashion. As will be seen, the ytterbium(III) ion in $\mathrm{Yb(HBPz_3)_3}$ is not nine- but eight-coordinate and exhibits the rather less common coordination geometry for eight-coordination, the bicapped trigonal prism, BCTP, confirming the unpredictable nature of the coordination modes of the lanthanide ions.

Experimental Section

The title compound was prepared by a previously reported procedure.[9] Crystals suitable for X-ray diffraction were obtained by slow crystallization from $\mathrm{CH_2Cl_2}$/hexane. A crystal was sealed in a thin-walled glass capillary and mounted on a Picker FACS-1 automated diffractometer. A summary of the crystal data and important features of the data collection are given in Table I.

Table I. Summary of Crystal Data and Intensity Data Collection

compd	$\mathrm{Yb(HBPz_3)_3}$
formula	$\mathrm{YbN_{18}C_{27}B_3H_{30}}$
cell parameters[a]	
a	13.729 (2) Å
b	21.465 (3) Å
c	11.461 (1) Å
Z	4
space group	$P2_12_12_1$ (D_2^4, No. 19)
cryst vol	1.1×10^{-2} mm^3
cryst shape	hexagonal rod
faces and dist (in mm) from arbitrary origin within cryst	100, 0.26; $\bar{1}00$, 0.26; 010, 0.06; $0\bar{1}0$, 0.06; 011, 0.09; $0\bar{1}\bar{1}$, 0.09; $01\bar{1}$, 0.09; $0\bar{1}1$, 0.09
temp	20 °C
radiation	Cu Kα (Ni filtered)
μ	55.62 cm^{-1}
range in abs cor factors	0.282–0.550
receiving aperture	3 mm × 3 mm, 30 cm from cryst
takeoff angle	3.1°
scan speed	2° min^{-1} ($4° \leq 2\theta \leq 105°$), 1° min^{-1} ($105° < 2\theta \leq 122°$)
scan range	0.95° below Kα_1 to 0.95° above Kα_2
bkgd counting time	20 s at each end of the scan for $4° \leq 2\theta \leq 105°$; 40 s for $105° < 2\theta \leq 122°$
2θ limits	4.0–122.0°
p factor[10]	0.04
unique data collected	2959
unique data used ($I_o \geq 2\sigma(I_o)$)	2662
final no. of parameters varied	313
error in observn of unit wt[b]	1.50
R[c]	0.038
R_w[c]	0.048

[a] Unit cell parameters were obtained by a least-squares analysis of the setting angles of 12 reflections with 2θ between 50 and 75°. [b] Error in observation of unit weight $[\Sigma w(|F_o| - |F_c|)^2/(N_o - N_v)]^{1/2}$, where N_o is the number of observations and N_v is the number of variables. [c] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma wF_o^2]^{1/2}$.

Data were corrected for absorption and for Lorentz and polarization factors. The structure was solved by a combination of Patterson and difference-Fourier syntheses and full-matrix least-squares refinement.[11] Atomic scattering factors were taken from Cromer and Waber's

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(11) Besides locally written programs, the following were used in solution and refinement of the structure: FORDAP, the Fourier summation program by A. Zalkin; AGNOST, the absorption and extinction program from Northwestern University; SFLS-5, structure factor and least-squares refinement by C. J. Prewitt; ORFFE, calculation of bond lengths and angles by W. Busing and H. A. Levy; ORTEP, thermal ellipsoid plotting program by C. K. Johnson.

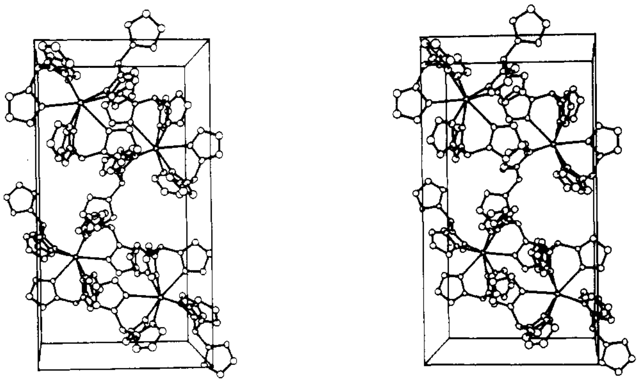

Figure 1. Stereoview (ORTEP) of the molecular packing of $Yb(HBPz_3)_3$. The *a* axis projects out of the page, the *b* axis runs from top to bottom, and the *c* axis runs horizontal to the right. 20% thermal ellipsoids are used in all figures.

tabulations[12] for all atoms except hydrogen for which the values of Stewart et al.[13] were used. Anomalous dispersion terms[14] for ytterbium were included in F_c. The hydrogen atoms were included as fixed contributions in calculated positions with C–H and B–H distances of 0.95 Å and with thermal parameters 1 Å^2 greater than those of the atoms to which they are attached. The ytterbium, nitrogen, and boron atoms and one carbon atom, C(95), were refined with anisotropic thermal parameters. The remaining carbon atoms were refined with isotropic thermal parameters. C(95) was refined anisotropically to resolve the ambiguity in the positions of C(95) and N(92) in the uncoordinated pyrazolyl group. Refinements were carried out with both possible dispositions of the atoms. With the arrangements finally chosen as being correct, the model refined to values of $R = 0.039$ and $R_w = 0.048$ and gave thermal parameters for the two atoms roughly comparable (as viewed from ORTEP plots) to those of atoms N(91), B(3), and N(81). The alternative arrangement refined to $R = 0.040$ and $R_w = 0.050$, and the thermal parameters were "unreasonable";[15] N(92), refined as a carbon atom, and C(95), refined as a nitrogen, had thermal parameters that were noticeably smaller and larger, respectively, than that of N(91).

The final model with 313 parameters varied refined to $R = 0.038$ and $R_w = 0.048$. Refinement of the other enantiomer converged to a model with $R = 0.046$ and $R_w = 0.059$, indicating the original choice of enantiomer was correct. The final positional and thermal parameters for the non-hydrogen atoms are given in Table II.

Results and Discussion

The crystal structure consists of discrete molecular units. A molecular packing diagram is shown in Figure 1. A stereoview of the molecule is shown in Figure 2. Figure 3 describes the atom numbering scheme. The first digit of the number assigned to a carbon or nitrogen denotes the pyrazolyl ring (1–9) of which it is a member while the second digit denotes its position within the ring.

The most important result of the X-ray analysis is that it establishes unambiguously that the ytterbium ion in the molecule is eight-coordinate. The eight nitrogen atoms are provided by two hydridotris(pyrazol-1-yl)borate ligands coordinating in a tridentate fashion while the third acts as a bidentate chelating ligand. The molecular point symmetry is close to C_s. A noncrystallographic mirror plane bisecting the molecule contains the ytterbium atom, the three boron atoms, and three pyrazolyl rings (N(31)...C(35), N(61)...C(65), N(91)...C(95)). Figure 2 is a view along this plane while Figure 3 shows a view perpendicular to it.

Table II. Final Positional Parameters for Non-Hydrogen Atoms of $Yb(HBPz_3)_3$

atom	x^a	y	z	B, Å^2
Yb	0.00604 (3)	0.68860 (2)	0.24151 (3)	
N(12)	−0.1062 (6)	0.7673 (4)	0.3092 (8)	
N(11)	−0.0853 (5)	0.8291 (4)	0.3073 (7)	
N(22)	0.1236 (6)	0.7688 (4)	0.2801 (7)	
N(21)	0.0998 (5)	0.8305 (4)	0.2852 (7)	
N(32)	−0.0092 (7)	0.7753 (3)	0.0839 (5)	
N(31)	−0.0103 (7)	0.8365 (3)	0.1108 (5)	
N(42)	−0.0986 (6)	0.6072 (4)	0.3307 (8)	
N(41)	−0.0697 (7)	0.5707 (4)	0.4223 (9)	
N(52)	0.1254 (6)	0.6086 (4)	0.3051 (8)	
N(51)	0.1121 (6)	0.5739 (4)	0.4044 (8)	
N(62)	0.0260 (6)	0.6982 (3)	0.4615 (6)	
N(61)	0.0251 (7)	0.6478 (4)	0.5348 (6)	
N(72)	−0.1148 (6)	0.6595 (4)	0.1003 (7)	
N(71)	−0.1002 (6)	0.6134 (4)	0.0177 (8)	
N(82)	0.1024 (5)	0.6618 (4)	0.0717 (7)	
N(81)	0.0817 (6)	0.6137 (4)	−0.0013 (8)	
N(91)	−0.0132 (7)	0.5177 (3)	−0.0605 (6)	
N(92)	−0.0201 (7)	0.5254 (3)	−0.1779 (6)	
B(1)	0.0017 (8)	0.8558 (4)	0.2388 (7)	
B(2)	0.0249 (11)	0.5821 (6)	0.4831 (11)	
B(3)	−0.0065 (13)	0.5723 (4)	0.0248 (8)	
C(95)	−0.0161 (9)	0.4569 (4)	−0.0328 (8)	
C(13)	−0.1884 (7)	0.7620 (5)	0.3714 (9)	4.2 (2)
C(14)	−0.2192 (8)	0.8191 (5)	0.4135 (9)	5.0 (2)
C(15)	−0.1531 (8)	0.8590 (5)	0.3704 (9)	4.2 (2)
C(23)	0.2137 (8)	0.7661 (5)	0.3200 (10)	4.8 (2)
C(24)	0.2481 (8)	0.8252 (5)	0.3530 (9)	5.0 (2)
C(25)	0.1743 (8)	0.8644 (5)	0.3289 (9)	4.4 (2)
C(33)	−0.0242 (7)	0.7717 (4)	−0.0324 (8)	4.3 (2)
C(34)	−0.0341 (8)	0.8317 (5)	−0.0769 (9)	5.2 (2)
C(35)	−0.0250 (8)	0.8706 (4)	0.0147 (8)	4.5 (2)
C(43)	−0.1900 (9)	0.5873 (6)	0.3038 (11)	5.6 (3)
C(44)	−0.2182 (10)	0.5414 (6)	0.3796 (12)	6.6 (3)
C(45)	−0.1418 (9)	0.5315 (6)	0.4511 (11)	5.8 (3)
C(53)	0.2104 (8)	0.5907 (5)	0.2559 (10)	5.2 (2)
C(54)	0.2515 (9)	0.5440 (6)	0.3224 (12)	6.3 (3)
C(55)	0.1877 (10)	0.5343 (6)	0.4135 (12)	6.2 (3)
C(63)	0.0268 (8)	0.7479 (4)	0.5287 (8)	4.3 (2)
C(64)	0.0286 (9)	0.7299 (5)	0.6463 (10)	5.7 (2)
C(65)	0.0259 (8)	0.6670 (5)	0.6459 (9)	5.3 (2)
C(73)	−0.2018 (7)	0.6820 (5)	0.0761 (8)	4.2 (2)
C(74)	−0.2446 (8)	0.6545 (5)	−0.0188 (10)	5.5 (2)
C(75)	−0.1768 (8)	0.6118 (5)	−0.0539 (10)	5.2 (2)
C(83)	0.1830 (7)	0.6883 (5)	0.0322 (8)	4.2 (2)
C(84)	0.2159 (8)	0.6564 (5)	−0.0687 (9)	4.4 (2)
C(85)	0.1494 (8)	0.6108 (5)	−0.0863 (9)	4.4 (2)
C(93)	−0.0246 (7)	0.4688 (5)	−0.2218 (8)	4.8 (2)
C(94)	−0.0243 (8)	0.4246 (5)	−0.1353 (9)	5.0 (2)

[a] Estimated standard deviations in the least significant figure(s) are given in parentheses in this and all subsequent tables.

Examination of the figures reveals that the coordination polyhedron formed by the eight nitrogen atoms about the ytterbium is a bicapped trigonal prism, BCTP, the considerably less common polytopal form for eight-coordination. The more commonly formed geometries for discrete eight-coordinate complexes are[16] D_{2d} dodecahedron, DOD, and the D_{4d} square antiprism, SAP. There are several sets of parameters that have been used D_{2d} justify the assignment of D_{4d} shape of a particular complex to one of these reference polyhedra or to measure deviations therefrom. Probably the most elegant and useful methods are those of Porai-Koshits and Aslanov[17] and Muetterties and Guggenberger.[18] The values of the shape parameters, δ and ϕ, for $Yb(HBPz_3)_3$ are listed in Table III along with the values for the idealized polyhedra. Figure 4 shows a view of the coordination polyhedron of the complex.

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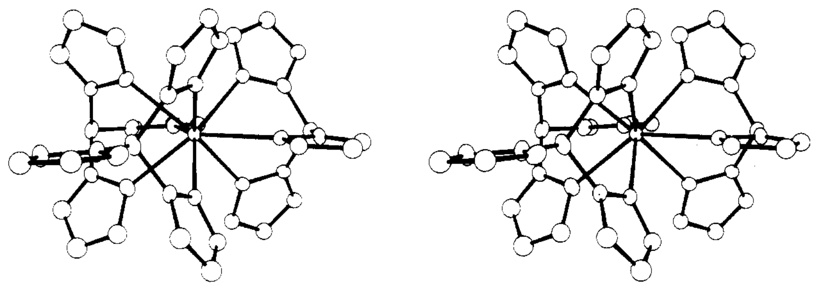

Figure 2. Stereoview (ORTEP) of the $Yb(HBPz_3)_3$ molecule. The view is along the noncrystallographic mirror plane containing Yb, B(1), B(2), and B(3).

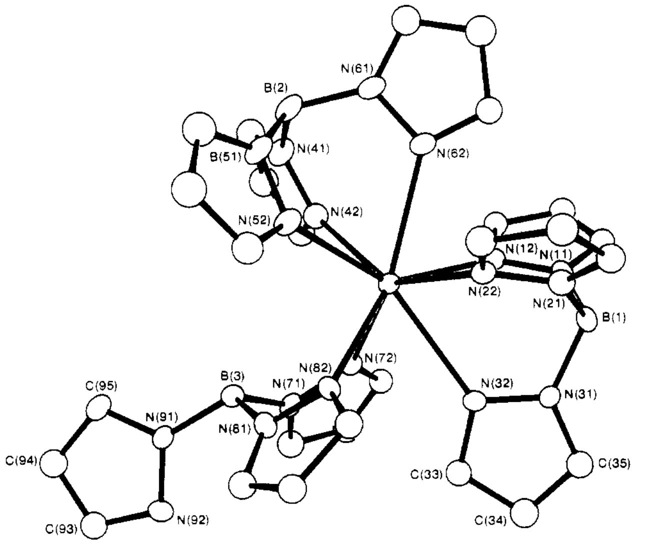

Figure 3. View (ORTEP) of $Yb(HBPz_3)_3$, perpendicular to the noncrystallographic mirror plane, showing the atom numbering scheme.

Table III. Shape Parameters (Deg) for $Yb(HBPz_3)_3$[a]

		DOD	SAP	BCTP	$Yb(HBPz_3)_3$
δ^b	N(12)-N(22), h_2	29.5	0.0	21.8	34.2 (3)
	N(52)-N(72)	29.5	0.0	0.0	0.1 (5)
	N(12)-N(72), t_2	29.5	52.4	48.2	40.0 (4)
	N(22)-N(82), t_2	29.5	52.4	48.2	40.8 (4)
	N(12)-N(42), t_2	29.5	52.4	48.2	43.1 (3)
	N(22)-N(52), t_2	29.5	52.4	48.2	41.6 (3)
ϕ^c	N(12)-N(32)-N(82)-N(52)	0.0	24.5	14.1	11.0
	N(22)-N(62)-N(42)-N(72)	0.0	24.5	14.1	11.9
	N(12)-N(62)-N(52)-N(82)	0.0	24.5	14.1	13.1
	N(22)-N(32)-N(72)-N(42)	0.0	24.5	14.1	13.6

[a] Parameters for idealized polyhedra are taken from ref 22. [b] δ parameters are the dihedral angles (deg) between faces meeting at the edge specified. Edge labeling scheme is that from ref 21. [c] The φ parameter is a measure of the degree of planarity of the BAAB trapezoids derived from the DOD.

Reference to Table III and Figure 4 shows that the coordination polyhedron is indeed a very good example of the bicapped trigonal prism. The rectangular face of the prism (N(42)-N(52)-N(72)-N(82)) is effectively planar, and the triangular top and bottom faces of the prism (N(12)-N(42)-N(72) and N(22)-N(52)-N(82)) are inclined by only 3° (see supplementary material for least-squares planes).

The ligands are disposed such that each tridentate ligand bridges the height of the prism and caps a rectangular face. The bidentate ligand spans the remaining prism height. The configuration at B(3), the boron atom of the bidentate ligand, is such that the hydrogen atom is directed toward the ytterbium (for an assumed B-H bond length of 1.20 Å, the hydrogen is 3.23 Å away from the metal atom). The uncoordinated pyrazolyl group is pointed away from the metal and thus has no opportunity to coordinate to the uncapped rectangular face to form a nine-coordinate tricapped-trigonal-prismatic complex. The normalized edge lengths for the coordination polyhedron of $Yb(HBPz)_3$ and the idealized edge lengths of the reference polyhedra are given in Table IV. The values based on both the hard-sphere model (HSM) of Muetterties and Guggenberger[18] and the most favored polyhedron (MFP) of Drew[4] are given. In the latter study the prism is no longer constrained to rigorous trigonal symmetry. The agreement between the observed and the HSM dimensions is not good. The height of the prism, as represented typically by N(42)-N(52) (h_1), 3.09 Å, although greater than N(42)-N(72) (t_1), 2.88 Å, is much shorter than N(42)-N(12) (t_2), 3.45 Å. Drew[4] has noted that, if the BCTP is produced from the D_{3h} TCTP by removal of a capping ligand to form the unique rectangular face, contraction of that uncapped face and an opening out of the remaining four ligand atoms of the coordination polyhedron would be expected. This effect is observed in this structure in the relative lengthening of the unique height of the prism, h_2 (the edge common to the two

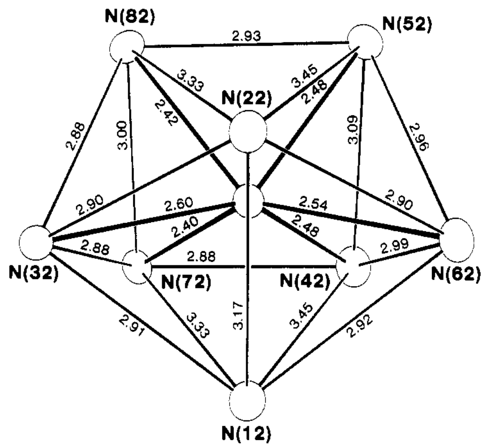

Figure 4. View (ORTEP) of the coordination polyhedron of $Yb(HBPz_3)_3$ showing its bicapped-trigonal-prismatic shape. Bond lengths and edge lengths are in Å.

Table IV. Normalized Edge Lengths (Å) of Polyhedra with Bicapped-Trigonal-Prismatic Geometry

edge		BCTP HSM[a]	BCTP MFP[b]	$Yb(HBPz_3)$[c]
N(72)-N(82), N(42)-N(52)	h_1	1.491	1.25	1.22, 1.25
N(12)-N(22)	h_2	1.491	1.50	1.28
N(12)-N(72), N(22)-N(82)	t_2	1.155	1.36	1.35, 1.35
N(12)-N(42), N(22)-N(52)	t_2	1.155	1.36	1.40, 1.40
N(42)-N(72), N(52)-N(82)	t_1	1.155	1.13	1.17, 1.19
N(22)-N(32), N(12)-N(32)	p_2	1.155	1.18	1.17, 1.18
N(12)-N(62), N(22)-N(62)	p_2	1.155	1.18	1.18, 1.17
N(42)-N(62), N(52)-N(62)	p_1	1.155	1.21	1.21, 1.20
N(72)-N(32), N(82)-N(32)	p_1	1.155	1.21	1.17, 1.17

[a] Hard-sphere model.[18] [b] Most favored polyhedron.[4] [c] Calculated with average Yb-N bond length, 2.47 Å.

Table V. Selected Bond Distances and Angles in $Yb(HBPz_3)_3$[a]

(i) Bond Distances (Å)

Yb-N(12)	2.414 (9)	Yb-N(42)	2.482 (8)
Yb-N(22)	2.401 (8)	Yb-N(52)	2.484 (8)
Yb-N(72)	2.401 (8)	Yb-N(32)	2.601 (6)
Yb-N(82)	2.423 (7)	Yb-N(62)	2.544 (7)
B-N	1.530 (15)	N-C	1.332 (12)
N-N	1.365 (11)	C-C	1.362 (16)

(ii) Angles at the Yb Atom (Deg)

N(12)-Yb-N(42)	89.5 (3)	N(22)-Yb-N(52)	89.9 (3)
N(12)-Yb-N(72)	87.5 (3)	N(22)-Yb-N(82)	87.2 (3)
N(42)-Yb-N(72)	72.2 (3)	N(52)-Yb-N(82)	73.2 (3)
N(12)-Yb-N(22)	82.4 (2)	N(42)-Yb-N(52)	76.9 (3)
N(32)-Yb-N(62)	129.7 (2)	N(72)-Yb-N(82)	76.0 (3)
N(32)-Yb-N(12)	70.8 (3)	N(62)-Yb-N(12)	72.2 (3)
N(32)-Yb-N(22)	70.7 (3)	N(62)-Yb-N(22)	71.8 (3)
N(32)-Yb-N(72)	70.3 (3)	N(62)-Yb-N(42)	73.2 (3)
N(32)-Yb-N(82)	69.9 (3)	N(62)-Yb-N(52)	72.2 (3)

[a] The average B-N, N-N, C-N, and C-C distances are given.

capped faces of the prism, N(12)–N(22)) such that $h_2 > h_1$, and a relative shortening of the t_1 edges, resulting in $t_2 > t_1$. This dramatic difference in edge lengths at the triangular face of the prism also provides an explanation for the unexpectedly large value, 34.2°, for the δ angle at the h_2 edge. The value calculated for the idealized shape, where the prism was constrained to be trigonally symmetric, is 21.8°. However, the lengthening of t_2 relative to t_1 acts to increase this angle to its observed value. The idealized polyhedral shape, despite these adjustments, would still possess C_{2v} symmetry, and this is the approximate symmetry of the coordination polyhedron of $Yb(HBPz_3)_3$.

The ytterbium nitrogen bond lengths and the interbond angles at the ytterbium are shown in Table V. The Yb–N bond lengths range from 2.401 (8) to 2.601 (6) Å. This is in agreement with previously reported values,[6,19] the range being from 2.37 (3) Å in orthorhombic $[Yb(dpa)_3]^{3-}$[6a] (dpa) = dipicolinic acid) to 2.61 (1) Å in (μ-pyr)$[(\eta\text{-}C_5H_5)_3Yb]_2$ (pyr = pyrazine).[19c] The coordination of pyrazolyl groups within each tridentate ligand is not symmetrical. The capping pyrazolyl groups have longer Yb–N bond lengths than those coordinating at the apices of the prism. Additionally, the ligand coordinating through N(12), N(22), and N(32) has a greater range of Yb–N bond lengths than the ligand with coordinating nitrogens N(45), N(52), and N(62). The difference between the longest and the shortest bond distances are 0.20 and 0.06 Å, respectively. Such variation is also observed in the bite distances. The difference between the longest and shortest bite distances for N(12), N(22), and N(32) is 0.27 Å whereas for N(2), N(52), and N(62) it is only 0.11 Å (see Figure 4 and supplementary material). These distinct differences between the two tridentate ligands are almost certainly due in part to the fact that the unique height of the prism is longer than the other two heights. The bite distance of the bidentate ligand, N(72)–N(82), 3.00 (1) Å, lies in the range of bite distances exhibited by the tridentate ligands. Although the significant variation in these distances detracts somewhat from the meaning of an average value, on the whole it appears that the bite distances are somewhat larger than those found in transition-metal hydridotris(pyrazol-1-yl)borate complexes (average values: ($Yb(HBPz_3)_3$, 3.00 Å; $Co(HBPz_3)_3$,[20] 2.89 Å).

The bond lengths and bond angles in the hydridotris(pyrazol-1-yl)borate ligands (Table V and supplementary material) are consistent with those found in structural studies of other complexes containing hydridotris(pyrazol-1-yl)borate ligands.[20,21] Each of the nine pyrazolyl rings is planar; the deviation of any atom from the calculated least-square plane is less than 0.015 Å (supplementary material). The angle between the plane B(3)–N(71)–N(81) and the plane N(71)–N(81)–N(72)–N(82) in the bidentate ligand is 126.3°. Corresponding dihedral angles in the other two ligands between the plane B(1)–N(11)–N(21) and the plane N(11)–N(21)–N(12)–N(22) and between the plane B(2)–N(41)–N(51) and the plane N(41)–N(51)–N(42)–N(52) are 129.4 and 130.5°, respectively. Thus, the boat conformation of the bidentate ligand is only very slightly "steeper" than would be expected from the geometries of the tridentate ligands. Similarly the dihedral angles between the pyrazolyl rings within each ligand range from 112.6 to 128.6°. The angle between the two coordinated pyrazolyl groups of the bidentate ligand is 121.8°.

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Supplementary Material Available: Tables of structure factor amplitudes, anisotropic thermal parameters, hydrogen atom parameters, and least-squares planes and an exhaustive listing of interatomic distances and angles (16 pages). Ordering information is given on any current masthead page.

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