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Preparation of some Lewis Base Adducts of Tris(methyltrihydroborato)Ho and Yb and Crystal Structures of Tris(methyltrihydroborato)ytterbium(III)etherate and Tris(methyltrihydroborato)holmium(III)bis(pyridine)*

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Summary. The complexes $M(BH_3CH_3)_3 \cdot O(C_2H_5)_2$ (M = Lu, Yb, Ho); $M(BH_3CH_3)_3 \cdot OC_4H_8$ (M = Lu, Yb, Ho); $M(BH_3CH_3)_3 \cdot 2OC_4H_8$ (M = Ho, Yb); and $Ho(BH_3CH_3)_3 \cdot 2C_6H_5N$ have been prepared and characterized. The crystal structures of $Yb(BH_3CH_3)_3 \cdot O(C_2H_5)_2$ and $Ho(BH_3CH_3)_3 \cdot 2C_6H_5N$ have been determined. Crystals of $(CH_3BH_3)_3Yb \cdot O(C_2H_5)_2$ are monoclinic, $P2_1/n$, $Z = 4$, with $a = 10.745(4)$, $b = 15.647(4)$, $c = 9.215(3)$ Å, and $\beta = 90.78(2)°$ at 23 °C; crystals of $(CH_3BH_3)_3Ho \cdot 2(C_5H_5N)$ are triclinic, $P1$, $Z = 2$, with $a = 9.370(4)$, $b = 12.155(2)$, $c = 8.840(2)$ Å, $\alpha = 80.47(2)°$, $\beta = 107.41(2)°$ and $\gamma = 95.15(2)°$ at 23 °C. The structures were refined by least-squares to a conventional R factor of 0.023, [2767 data, $F^2 > 2\sigma(F^2)$] for the Yb complex, and 0.040 [1554 data, $F^2 > 3\sigma(F^2)$] for the Ho complex. Both complexes are monomeric. The ytterbium atom is tetrahedrally coordinated to three boron atoms and an ethyl oxygen atom with distances: Yb–B 2.40(2), 2.41(2), 2.43(2) Å, and Yb–O 2.23(1) Å. The holmium atom is at the center of a trigonal bipyramid that has three boron atoms on the equator and two nitrogen atoms at the apices with distances: Ho–B 2.483(6), 2.495(6) and 2.520(5) Å and Ho–N 2.535(4) and 2.555(4) Å.

Introduction

The actinide(IV)tetrahydroborates $M(BH_4)_4$ (M = Th, Pa, U) are polymeric solids [1, 2]. In order to obtain monomeric compounds for spectroscopic and magnetic measurements, the $BH_3CH_3^-$ ligand has been utilized in place of BH_4^-. The $M(BH_3CH_3)_4$ compounds (M = Th, U, Np) have been characterized along with some Lewis base adducts of $Th(BH_3CH_3)_4$ and $U(BH_3CH_3)_4$ [3–6]. A limited number of Lewis base adducts of lanthanide, Sc, or Y(III) tetrahydroborates are known [7–9]. We report in this paper the synthesis of some new Lewis base adducts of lanthanide(III) methyltrihydroborate compounds and the structural characterization of $Ho(BH_3CH_3)_3 \cdot 2py$ (py = pyridine) and $Yb(BH_3CH_3) \cdot O(C_2H_5)_2$.

Experimental

All preparations were carried out under an atmosphere of argon or under high vacuum. Diethyl ether was distilled from sodium benzophenone ketyl under argon. Toluene was dried over Na and distilled under argon. Pyridine was dried over NaOH, and then distilled from BaO. Benzene-d_6 and toluene-d_8 were degassed and dried over Na. Melting points (m.p.) were determined in sealed argon-filled capillaries and are uncorrected. $LiBH_3CH_3$ was prepared according to the procedure of Wartik and Schlesinger [10]. $YbCl_3$ and $HoCl_3$ were prepared according to the procedure of Taylor and Carter [11].

Infrared spectra were recorded on a Perkin-Elmer IR-238 spectrometer and calibrated with polystyrene film; Nujol and Fluorolube mulls were prepared in an argon-filled dry box with dry, degassed Nujol and Fluorolube. ^1H NMR spectra were recorded on a JEOL FX 90Q 90 MHz FT spectrometer and referenced to tetramethylsilane. Samples were dissolved in benzene-d_6 and toluene-d_8 inside an argon-filled dry box.

Preparation of $Yb(BH_3CH_3)_3 \cdot OEt_2$

To 0.81 g (22.6 mmol) $LiBH_3CH_3$, 2.11 g (7.5 mmol) $YbCl_3$ and 40 ml diethyl ether were added, and the reaction mixture was stirred for 2 days. The solvent was pumped off and the residue was sublimed at 50 °C *in vacuo* onto a cold finger. A yield of 1.01 g (40%) of white, crystalline $Yb(BH_3CH_3)_3 \cdot OEt_2$ was obtained that melted at 59–60 °C. *Anal.* Calc. for $C_7H_{28}B_3OYb$: C, 25.19; H, 8.46. Found: C, 25.06; H, 8.44%.

$M(BH_3CH_3)_4 \cdot OEt_2$ (M = Lu or Ho) were prepared in a similar manner as $Yb(BH_3CH_3)_4 \cdot OEt_2$. Satisfactory analyses were obtained for these compounds. IR data are listed in Table I and NMR data in Table II.

Preparation of $Lu(BH_3CH_3)_3 \cdot THF$

$Lu(BH_3CH_3)_3 \cdot OEt_2$ (0.33 g, 0.98 mmol) was dissolved in 10 ml tetrahydrofuran (THF) and the solution allowed to equilibrate overnight. The THF was pumped off and the white residue was sublimed at 100 °C *in vacuo* for several hours. A yield of 0.27 g (68%) of white $Lu(BH_3CH_3)_3 \cdot THF$ was recovered that melted at 110–112 °C. *Anal.* Calc. for $C_7H_{26}B_3LuO$: C, 25.20; H, 7.85. Found: C, 25.42; H, 7.75%.

$M(BH_3CH_3)_4 \cdot THF$ (M = Yb or Ho) were prepared in a similar manner as $Lu(BH_3CH_3) \cdot THF$. Satisfactory

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TABLE I. Infrared Spectroscopic Data (in cm^{-1})

Compound										
$Yb(BH_3CH_3)_3 \cdot OEt_2$	2950m,	2640w,	2430w,	2190m,	2100s,	1310s,	1270s,	1190m,	1145m,	1085s,
	1030s,	990m,	970w,	890m,	825m,	770m,	720m			
$Lu(BH_3CH_3)_3 \cdot THF$	2950m,	2190w(sh),	2100s,	1310s,	1265s,	1175w,	1090s,	1035w,	995s,	960m,
	920m,	850s,	720w,	680w						
$Ho(BH_3CH_3)_3 \cdot 2THF$	2950m,	2180m,	2110s,	1345w,	1300s,	1255s,	1170w,	1085s,	1035m,	1005s,
	940m,	920m,	850s,	720m,	665m					
$Ho(BH_3CH_3)_3 \cdot 2py$	2950m,	2360m,	2310w,	2130s,	1630m,	1595s,	1570w,	1485s,	1440s,	1365w,
	1300s,	1255s,	1220s,	1150m,	1135w,	1125w,	1090w,	1065s,	1035s,	1015w,
	1000s,	980w,	945w,	925w,	750s,	720s,	700s,	620s,	470w,	425s
$Ho(BH_3CH_3)_3 \cdot py$	2950m,	2120m,	1630m,	1620m,	1600s,	1570w,	1485s,	1440s,	1360w,	1305s,
	1255w,	1230w,	1215s,	1150m,	1085s,	1065s,	1035s,	1005s,	945m,	875w,
	750s,	720w,	690s,	620s,	465w,	425s				

TABLE II. Proton Magnetic Resonance Data[a, b]

Compound		
$Lu(BH_3CH_3)_3 \cdot OEt_2$	0.39 (9H, quart) 1.71 (9H, quart)	0.78 (6H, mult) 3.27 (4H, mult)
$Yb(BH_3CH_3)_3 \cdot OEt_2$	−162.83 (9H, s) +36.38 (6H, s)	−25.07 (9H, s) +92.65 (4H, s)
$Ho(BH_3CH_3)_3 \cdot OEt_2$[c]	−220.70 (4H, s) +84.80 (9H, s)	−95.61 (6H, s)
$Lu(BH_3CH_3)_3 \cdot THF$	0.41 (9H, quart) 1.84 (9H, quart)	0.81 (4H, mult) 3.33 (4H, mult)
$Yb(BH_3CH_3)_3 \cdot THF$	−160.4 (9H, s) 38.3 (4H, s)	−29.4 (9H, s) 82.2 (4H, s)
$Ho(BH_3CH_3)_3 \cdot THF$[c]	−178.61 (4H, s) 77.11 (9H, s)	−87.8 (4H, s)
$Yb(BH_3CH_3)_3 \cdot 2THF$	−69.21 (9H, s) 8.10 (8H, s)	−7.77 (8H, s) 26.36 (9H, s)
$Ho(BH_3CH_3)_3 \cdot 2THF$	−148.08 (9H, s) −13.15 (8H, s)	−35.83 (8H, s) 31.67 (9H, s)
$Ho(BH_3CH_3)_3 \cdot 2py$[d]	−206.53 (9H, s) −1.77 (4H, s) +22.95 (9H, s)	−30.65 (4H, s) +3.13 (2H, s)
$Ho(BH_3CH_3)_3 \cdot py$	−50.61 (9H, s) −5.66 (2H, s) +32.15 (9H, s)	−8.93 (2H, s) −1.91 (1H, s)

[a] All samples in benzene-d_6. [b] Shifts in ppm from tetramethylsilane. Positive sign indicates downfield shift. All values are at 30 °C. [c] BH_3 peak could not be found. [d] $[Ho(BH_3CH_3)_3 \cdot 2py] = 2.3 \times 10^{-2}$ g/ml.

analyses were obtained for these compounds. IR data are listed in Table I and NMR data in Table II.

Preparation of $Ho(BH_3CH_3)_3 \cdot 2THF$

$Ho(BH_3CH_3)_3 \cdot OEt_2$ (0.48 g, 1.5 mmol) was dissolved in 10 ml THF and pumped to dryness. The product was redissolved in 10 ml toluene, filtered, reduced to 2 ml volume, and cooled to −20 °C. Pale orange crystals were obtained, which were isolated and washed with cold (−78 °C) diethyl ether. A yield of 0.17 g (29%) of $Ho(BH_3CH_3)_3 \cdot 2THF$ that melted at 133–134 °C was obtained. *Anal.* Calc. for $C_{11}H_{34}B_3HoO_2$: C, 33.4; H, 8.7. Found: C, 33.0; H, 8.6%.

$Yb(BH_3CH_3)_3 \cdot 2THF$ was prepared in a similar manner as the Ho compound. Satisfactory analyses were obtained. IR data are given in Table I and NMR data in Table II.

Preparation of $Ho(BH_3CH_3)_3 \cdot 2py$

$Ho(BH_3CH_3)_3 \cdot OEt_2$ (0.37 g, 1.1 mmol) was dissolved in 5 ml toluene, to which 0.19 ml (2.4 mmol) pyridine was added with stirring. The solution was filtered, reduced to 3 ml volume and cooled to −20 °C. Pale orange crystals were obtained and washed with cold (−78 °C) ether. A yield of 0.21 g (43%) of $Ho(BH_3CH_3)_3 \cdot 2py$ was obtained. m.p. = 132–134 °C(dec). *Anal.* Calc. for $C_{13}H_{28}B_3HoN_2$: C, 38.11; H, 6.89; N, 6.83. Found: C, 36.65; H, 6.93; N, 6.33%.

Preparation of $Ho(BH_3CH_3)_3 \cdot py$

$Ho(BH_3CH_3)_3 \cdot OEt_2$ (0.19 g, 0.58 mmol) was dissolved in 5 ml toluene. Pyridine (0.05 ml, 0.6 mmol) was added with stirring. The solution was pumped to dryness and the residue was sublimed *in vacuo* at 125 °C for several hours. A yield of 0.06 g (~30%) $Ho(BH_3CH_3)_3 \cdot py$ was recovered. m.p. = 115–119 °C. *Anal.* Calc. for $C_8H_{23}B_3HoN$: C, 29.06; H, 7.01; N, 4.24. Found: C, 29.31; H, 7.19; N, 3.95%.

Infrared and ^1H NMR data are listed in Tables I and II.

X-ray Diffraction

The air-sensitive crystals were sealed inside quartz capillaries in an argon-filled dry box. A modified FACS-I Picker diffractometer was used to collect θ–2θ scanned single crystal X-ray diffraction data using graphite monochromated Mo Kα radiation ($\lambda_{K\alpha 1}$ = 0.70930 Å). For the Yb complex a half sphere of data was collected to a 2θ angle of 55°; for the Ho complex a full sphere of data was collected to a 2θ angle of 50°. The data were corrected for absorption effects and equivalent reflections were averaged. Experimental details of the data collection and the results of the least-squares refinements are given in the Supplementary Material. The structures were solved by Patterson and Fourier methods. The positional and anisotropic thermal parameters of the non-hydrogen atoms were refined by full-matrix least-squares. The hydrogen atoms in the holmium complex were indicated in the difference maps, and were included in the least-squares procedures with isotropic thermal parameters and distance-restrained positional parameters [12]. In the ytterbium complex the hydrogen atoms could not be identified, and were not included in the least-squares. Neutral atomic scattering factors were used [13] with the corrections for anomalous scattering applied.

Atomic parameters are listed in Table III. Listings of selected interatomic distances and angles are given in Table IV.

TABLE III. Positional Parameters with Estimated Standard Deviations[a]

Atom	x	y	z
$(CH_3BH_3)_3Yb \cdot O(C_2H_5)_2$			
Yb	0.24397(4)	0.07764(3)	0.12527(6)
O	0.2308(9)	−0.0592(6)	0.1912(11)
B(1)	0.0455(15)	0.1331(10)	0.1974(18)
B(2)	0.4178(14)	0.1320(12)	0.2692(18)
B(3)	0.2764(15)	0.0736(10)	−0.1317(18)
C(1)	−0.0923(13)	0.1741(13)	0.2400(21)
C(2)	0.5340(12)	0.1680(12)	0.3633(18)
C(3)	0.2958(18)	0.0731(12)	−0.3072(15)
C(4)	0.3489(20)	−0.1158(17)	0.1891(28)
C(5)	0.3514(24)	−0.1705(16)	0.062(3)
C(6)	0.1227(26)	−0.1047(16)	0.224(3)
C(7)	0.089(3)	−0.1032(17)	0.3774(28)
$(CH_3BH_3)_3Ho \cdot 2(NC_5H_5)$			
Ho	0.23807(2)	0.22726(2)	0.17785(2)
N(1)	0.4825(4)	0.30193(29)	0.1244(5)
N(2)	0.0154(4)	0.1452(3)	0.2762(5)
B(1)	0.0761(6)	0.3035(5)	−0.0907(7)
B(2)	0.3278(6)	0.0324(4)	0.2170(7)
B(3)	0.2834(6)	0.3539(5)	0.3842(7)
C(1)	−0.0301(6)	0.3572(6)	−0.2596(7)
C(2)	0.3811(6)	−0.0926(4)	0.2469(8)
C(3)	0.3147(8)	0.4280(6)	0.5243(8)
C(4)	0.6011(6)	0.3299(5)	0.2423(7)
C(5)	0.7340(6)	0.3722(5)	0.2194(10)
C(6)	0.7478(7)	0.3889(5)	0.0666(11)
C(7)	0.6301(8)	0.3604(6)	−0.0554(9)
C(8)	0.4997(6)	0.3166(5)	−0.0223(7)
C(9)	0.0037(6)	0.1549(4)	0.4212(6)
C(10)	−0.1193(7)	0.1136(5)	0.4730(7)
C(11)	−0.2354(6)	0.0606(5)	0.3714(8)
C(12)	−0.2251(6)	0.0492(5)	0.2232(8)
C(13)	−0.0990(6)	0.0918(5)	0.1815(6)

[a] Estimated standard deviations in this and subsequent tables are indicated in parentheses.

Discussion

In an analogous fashion to the preparation of actinide methyltrihydroborates [3], the preparation of $Yb(BH_3CH_3)_3$ was attempted by mixing $YbCl_3$ with $LiBH_3CH_3$ in chlorobenzene, but no volatile product was obtained. On the other hand, when $YbCl_3$ is mixed with $LiBH_3CH_3$ in diethyl ether, a volatile white product is recovered, $Yb(BH_3CH_3)_3 \cdot OEt_2$. Similarly $Lu(BH_3CH_3)_3 \cdot OEt_2$ and $Ho(BH_3CH_3)_3 \cdot OEt_2$ are obtained when $LiBH_3CH_3$ is reacted with $LuCl_3$ and $HoCl_3$, respectively, in diethyl ether. No volatile product is obtained when $SmCl_3$ is reacted with $LiBH_3CH_3$ in diethyl ether. From its IR spectrum, the mode of ligation of $Yb(BH_3CH_3)_3 \cdot OEt_2$ appears to be tridentate. A large, broad peak is found at 2100 cm^{-1} with a shoulder at ~2190 cm^{-1}

TABLE IV. Selected Bond Distances and Angles

$(CH_3BH_3)_3Yb\cdot O(C_2H_5)_2$		$(CH_3BH_3)_3Ho\cdot 2(NC_5H_5)$	
Distances (Å)			
Yb–O	2.230(10)	Ho–B(1)	2.483(6)
YB–B(1)	2.405(16)	Ho–B(2)	2.520(5)
Yb–B(2)	2.429(15)	Ho–B(3)	2.495(6)
Yb–B(3)	2.400(16)	Ho–N(1)	2.535(4)
B(1)–C(1)	1.665(20)	Ho–N(2)	2.555(4)
B(2)–C(2)	1.612(18)	B(1)–C(1)	1.596(7)
B(3)–C(3)	1.633(20)	B(2)–C(2)	1.597(7)
		B(3)–C(3)	1.593(8)
Angles (°)			
O–Yb–B(1)	102.2(5)	N(1)–Ho–N(2)	171.27(11)
O–Yb–B(2)	103.8(5)	N(1)–Ho–B(1)	95.44(15)
O–Yb–B(3)	104.7(5)	N(1)–Ho–B(2)	88.66(14)
B(1)–Yb–B(2)	113.6(5)	N(1)–Ho–B(3)	88.91(15)
B(1)–Yb–B(3)	115.1(6)	N(2)–Ho–B(1)	93.14(15)
B(2)–Yb–B(3)	115.3(5)	N(2)–Ho–B(2)	88.73(14)
Yb–O–C(4)	119.4(9)	N(2)–Ho–B(3)	86.06(16)
Yb–O–C(6)	127.2(11)	B(1)–Ho–B(2)	118.09(19)
Yb–B(1)–C(1)	177.3(12)	B(1)–Ho–B(3)	114.51(19)
Yb–B(2)–C(2)	179.4(10)	B(2)–Ho–B(3)	127.34(19)
Yb–B(3)–C(3)	178.4(12)	Ho–N(1)–C(4)	121 5(3)
		Ho–N(1)–C(8)	122.1(3)
		Ho–N(2)–C(9)	123.0(3)
		Ho–N(2)–C(13)	120.6(3)
		Ho–B(1)–C(1)	177.2(4)
		Ho–B(2)–C(2)	177.8(4)
		Ho–B(3)–C(3)	176.4(5)

(B–H(bridging) stretch). In addition, a peak is found at 1270 cm^{-1} (bridge deformation). These peaks are characteristic of tridentate ligation according to Marks and Kolb [7]. The IR spectra of $Lu(BH_3CH_3)_3\cdot OEt_2$ and $Ho(BH_3CH_3)_3\cdot OEt_2$ are similar to $Yb(BH_3CH_3)_3\cdot OEt_2$ in these regions.

When the etherate is dissolved in THF, pumped dry and sublimed, mono-THF adducts are obtained; e.g., $Yb(BH_3CH_3)_3\cdot$THF, $Lu(BH_3CH_3)_3\cdot$THF and $Ho(BH_3CH_3)_3\cdot$THF. Similarly when THT (THT = tetrahydrothiophene) is used, the mono-THT adduct is obtained; e.g., $Ho(BH_3CH_3)_3\cdot$THT. The IR spectra of these adducts are similar to the lanthanide methyltrihydroborate etherates in the B–H stretching regions, indicative of tridentate ligation of the methyltrihydroborate group. To confirm our analysis of the IR spectra of these mono-adducts, an X-ray crystal structure determination was made of $Yb(BH_3CH_3)_3\cdot OEt_2$.

The crystal structure confirmed the tridentate coordination of the methyltrihydroborate groups. The metal is surrounded by three tridentate methyltrihydroborates and one ether group in an approximately tetrahedral arrangement, with the methyltrihydroborates bent slightly towards the ether group (see Fig. 1). All Yb–B–C angles are close to 180°, indicative of tridentate ligation. The average Yb–B distance is 2.45 ± 0.04 Å. This may be compared to the pseudo-lanthanide Y–B(tridentate) distance of 2.58(1) Å found for $Y(BH_4)_3\cdot$3THF [8], and the Sc–B(tridentate) distance of 2.330(5) Å found for $Sc(BH_4)_3\cdot$2THF [9]. These values compare favorably since the ionic radius of Y(III) is about 0.04 Å longer and Sc(III) about 0.11 Å shorter than that of Yb(III) [8].

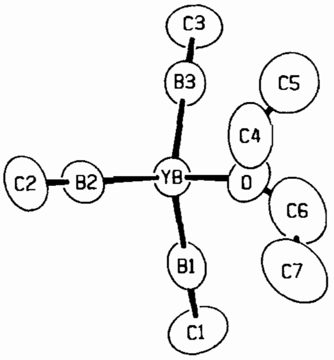

Fig. 1. ORTEP [16] drawing of $(CH_3BH_3)_3Yb\cdot O(C_2H_5)_2$; thermal ellipsoids at 50% probability level.

The Yb–O distance is 2.24(1) Å, which may be compared with the Y–O distance of 2.350(6) and 2.412(7) Å found for $Y(BH_4)_3\cdot$3THF [8] and the Sc–O distance of 2.170(2) and 2.163(3) Å found for $Sc(BH_4)_3\cdot$2THF [9], again consistent with the changes in ionic radii of the metal.

When the etherates are dissolved in THF, pumped dry, and then recrystallized from toluene, the bis-adducts are obtained; e.g., $Yb(BH_3CH_3)_3\cdot$2THF and $Ho(BH_3CH_3)_3\cdot$2THF. Similarly when THT or pyridine are used, the bis-THT and bis-py adducts are obtained, respectively; e.g., $Ho(BH_3CH_3)_3\cdot$2THT and $Ho(BH_3CH_3)_3\cdot$2py. Thus it appears that both the mono- and bis-unidentate base adducts of $Ln(BH_3CH_3)_3$ (Ln = lanthanide = Ho, Yb, Lu) are stable, with the former being volatile. In all cases, the bis-adduct decomposes to the mono-adduct when sublimed at ~100 °C *in vacuo*. The geometry of the bis-adducts appears to be trigonal bipyramidal with the base occupying the axial positions, judging by their proton NMR spectra since no non-equivalent BH_3CH_3 sites could be observed down to –70 °C.

The ^1H NMR of $Ho(BH_3CH_3)_3\cdot$2py is concentration dependent, suggesting the following equilibrium is taking place in solution:

$$Ho(BH_3CH_3)_3\cdot 2py \rightleftharpoons Ho(BH_3CH_3)_3\cdot py + py$$

The exchange of free and coordinated pyridine is rapid on the ^1H NMR time scale since only one set of averaged signals can be observed down to –70 °C.

The X-ray crystal structure analysis shows the geometry of $Ho(BH_3CH_3)_3\cdot$2py to be approximately trigonal bipyramidal with the tridentate methyltrihydroborates occupying the three equatorial posi-

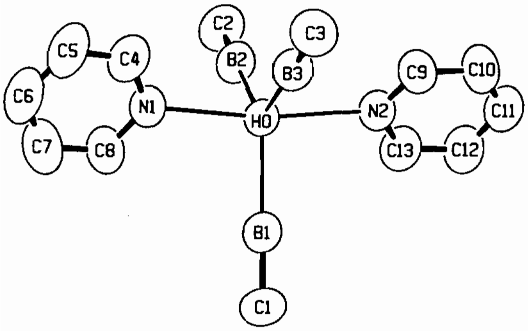

Fig. 2. ORTEP [16] drawing of $(CH_3BH_3)_3Ho{\cdot}2(NC_5H_5)$; thermal ellipsoids at 50% probability level.

tions and the pyridines the 2 axial positions (see Fig. 2). The average Ho–B distance is 2.50 ± 0.02 Å, which may be compared to the Yb–B distance of 2.45 ± 0.04 Å in $Yb(BH_3CH_3)_3{\cdot}OEt_2$; since the ionic radius of Ho(III) is about 0.03 Å larger than Yb(III) and the increase in coordination number is from 10 to 11, these distances are in very good agreement [14]. The average Ho–N distance is 2.55 ± 0.02 Å and is comparable to the Yb–N distance of 2.61(1) Å found in $(C_5H_5)_3Yb(NC_4H_4N)Yb(C_5H_5)_3$ [15].

Supplementary Material

Tables of anisotropic thermal parameters, estimated hydrogen positions and thermal parameters, hydrogen distances and the observed structure factors are available from the authors.

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