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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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STRUCTURES OF TRIS(METHYLTRIHYDROBORATO)YTTERBIUM
(III)ETHERATE AND TRIS(METHYLTRIHYDROBORATO)HOLMIUM
(III)BIS(PYRIDINE)**

R. Shinomoto, A. Zalkin, and N.M. Edelstein

April 1987

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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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Abstract. The complexes $M(\text{BH}_3\text{CH}_3)_3 \cdot \text{O}(\text{C}_2\text{H}_5)_2$ ($M = \text{Lu}, \text{Yb}, \text{Ho}$);
 $M(\text{BH}_3\text{CH}_3)_3 \cdot \text{OC}_4\text{H}_8$ ($M = \text{Lu}, \text{Yb}, \text{Ho}$); $M(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{OC}_4\text{H}_8$ ($M = \text{Ho}, \text{Yb}$); and
 $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{C}_6\text{H}_5\text{N}$ have been prepared and characterized. The crystal
structures of $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{O}(\text{C}_2\text{H}_5)_2$ and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{C}_6\text{H}_5\text{N}$ have been
determined. Crystals of $(\text{CH}_3\text{BH}_3)_3\text{Yb} \cdot \text{O}(\text{C}_2\text{H}_5)_2$ are monoclinic, $P2_1/n$, $Z=4$,
with $a = 10.745(4)$ Å, $b = 15.647(4)$ Å, $c = 9.215(3)$ Å, and $\beta = 90.78(2)^\circ$ at
23 °C; crystals of $(\text{CH}_3\text{BH}_3)_3\text{Ho} \cdot 2(\text{C}_6\text{H}_5\text{N})$ are triclinic, $P\bar{1}$, $Z=2$, with $a =$
 $9.370(4)$ Å, $b = 12.155(2)$ Å, $c = 8.840(2)$ Å, $\alpha = 80.47(2)^\circ$, $\beta = 107.41(2)^\circ$
and $\gamma = 95.15(2)^\circ$ at 23 °C. The structures were refined by least-squares to
a conventional R factor of 0.023, $[2767 \text{ data}, F^2 > 2\sigma(F^2)]$ for the Yb complex
and 0.040, $[1554 \text{ data}, F^2 > 3\sigma(F^2)]$ 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) Å, 2.41(2) Å,
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) Å, 2.495(6) Å
and 2.520(5) Å and Ho-N 2.535(4) Å 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 $Yb(BH_3CH_3)_3 \cdot O(C_2H_5)_2$.

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 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 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 $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ was obtained that melts at $59-60^{\circ}\text{C}$. Calculated for $\text{C}_7\text{H}_{28}\text{B}_3\text{OYb}$: C, 25.19; H, 8.46. Found: C, 25.06; H, 8.44.

$\text{M}(\text{BH}_3\text{CH}_3)_4 \cdot \text{OEt}_2$ (M = Lu or Ho) were prepared in a similar manner as $\text{Yb}(\text{BH}_3\text{CH}_3)_4 \cdot \text{OEt}_2$. Satisfactory analyses were obtained for these compounds. NMR data are listed in Table II.

Preparation of $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$

$\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ (0.33 g, 0.98 mmol) was dissolved in 10 mL 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 $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$ was recovered that melts at $110-2^{\circ}\text{C}$. Calculated for $\text{C}_7\text{H}_{26}\text{B}_3\text{LuO}$: C, 25.20; H, 7.85. Found: C, 25.42; H, 7.75.

$\text{M}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THF}$ (M = Yb or Ho) were prepared in a similar manner as $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$. Satisfactory analyses were obtained for these compounds. NMR data are listed in Table II.

Preparation of $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$

$\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ (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) ether. A yield of 0.17 g (29%) of $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$ that melts at $133-4^{\circ}\text{C}$ was obtained. Calculated for $\text{C}_{11}\text{H}_{34}\text{B}_3\text{HoO}_2$: C, 33.4; H, 8.7. Found: C, 33.0; H, 8.6.

$\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$ was prepared in a similar manner as the Ho compound. Satisfactory analyses were obtained. NMR data are given in Table II.

Preparation of $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{py}$.

$\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ (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 $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{py}$ was obtained. M. p. = $132-4^\circ\text{C}(\text{dec})$. Calculated for $\text{C}_{13}\text{H}_{28}\text{B}_3\text{HoN}_2$: C, 38.11; H, 6.89; N, 6.83. Found: C, 36.65; H, 6.93; N, 6.33.

Preparation of $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{py}$. $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ (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%) $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{py}$ was recovered. M. p. = $115-9^\circ$. Calculated for $\text{C}_8\text{H}_{23}\text{B}_3\text{HoN}$: 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 drybox. A modified FACS-I Picker diffractometer was used to collect θ - 2θ scanned single crystal X-ray diffraction data using graphite monochromated $\text{MoK}\alpha$ radiation ($\lambda_{\text{K}\alpha 1} = 0.70930 \text{ \AA}$). 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 was 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 tables. 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 Tables III. Listings of selected interatomic distances and angles are given in Table IV.

Discussion. In analogous fashion to the preparation of actinide methyltrihydroborates [3], the preparation of $\text{Yb}(\text{BH}_3\text{CH}_3)_3$ 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, $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$. Similarly $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ 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 ether. From its IR spectrum, the mode of ligation of $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ appears to be tridentate. A large, broad peak is found at 2100 cm^{-1} with a shoulder at $\sim 2190 \text{ cm}^{-1}$ ($\text{B-H}_{\text{bridging}}$ stretch). In addition a peak is found at 1270 cm^{-1} (bridge deformation). These peaks are characteristic of tridentate ligation according to Marks et al [7]. The IR spectra of $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ are similar to $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$ in these regions.

When the etherate is dissolved in THF, pumped dry, and sublimed, mono-THF adducts are obtained; e.g., $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$, $\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$, and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$. Similarly when THT (THT = tetrahydrothiophene) is used, the mono-THT adduct is obtained; e.g., $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THT}$. 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 $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$.

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 $\text{Y}(\text{BH}_4)_3 \cdot 3\text{THF}$ [8], and the Sc-B_{tridentate} distance of 2.330(5) Å found for $\text{Sc}(\text{BH}_4)_3 \cdot 2\text{THF}$ [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].

The Yb-O distance is 2.24(1) Å, which may be compared with the Y-O distance of 2.350(6) Å and 2.412(7) Å found for $\text{Y}(\text{BH}_4)_3 \cdot 3\text{THF}$ [8] and Sc-O distance of 2.170(2) Å and 2.163(3) Å found for $\text{Sc}(\text{BH}_4)_3 \cdot 2\text{THF}$ [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., $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$ and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$. Similarly when THT or pyridine are used, the bis-THT and bis-py adducts are obtained, respectively; e. g., $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THT}$ and $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{py}$. Thus it appears that both the mono- and bis-unidentate base adducts of $\text{Ln}(\text{BH}_3\text{CH}_3)_3$ (Ln = 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 nonequivalent BH_3CH_3 sites could be observed down to -70°C .

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

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 $\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{py}$ to be approximately trigonal bipyramidal with the tridentate methytrihydroborates occupying the three equatorial positions and the pyridines the 2 axial positions (see Fig. 2). The average Ho-B distance is $2.50 \pm 0.02 \text{ \AA}$, which may be compared to the Yb-B distance of $2.45 \pm 0.04 \text{ \AA}$ in $\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$; Since the ionic radius of Ho(III) is about $0.03 \text{ \AA}$ larger than Yb(III) and the increase in coordination number is from 10 to 11, these distances are in very good agreement [15]. The average Ho-N distance is $2.55 \pm 0.02 \text{ \AA}$ and is comparable to the Yb-N distance of $2.61(1) \text{ \AA}$ found in $(\text{C}_5\text{H}_5)_3\text{Yb}(\text{NC}_4\text{H}_4\text{N})\text{Yb}(\text{C}_5\text{H}_5)_3$ [16].

Supplementary Material. Tables of anisotropic thermal parameters, estimated hydrogen positions and thermal parameters, hydrogen distances, and the observed structure factors.

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

Compound						
$\text{Yb}(\text{BH}_3\text{CH}_3)_3 \cdot \text{OEt}_2$	2950m, 1270s, 970w,	2640w, 1190m, 890m,	2430w, 1145m, 825m,	2190m, 1085s, 770m,	2100s, 1030s, 720m	1310s, 990m,
$\text{Lu}(\text{BH}_3\text{CH}_3)_3 \cdot \text{THF}$	2950m, 1090s, 720w,	2190w(sh) 1035w, 680w	2100s, 995s,	1310s, 960m,	1265s, 920m,	1175w, 850s,
$\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{THF}$	2950m, 1170w, 850s,	2180m, 1085s, 720m,	2110s, 1035m, 665m	1345w, 1005s,	1300s, 940m,	1255s, 920m,
$\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot 2\text{py}$	2950m, 1570w, 1220s, 1035s, 750s,	2360m, 1485s, 1150m, 1015w, 720s,	2310w, 1440s, 1135w, 1000s, 700s,	2130s, 1365w, 1125w, 980w, 620s,	1630m, 1300s, 1090w, 945w, 470w,	1595s, 1255s, 1065s, 925w, 425s
$\text{Ho}(\text{BH}_3\text{CH}_3)_3 \cdot \text{py}$	2950m, 1485s, 1215s, 945m, 465w,	2120m, 1440s, 1150m, 875w, 425s	1630m, 1360w, 1085s, 750s,	1620m, 1305s, 1065s, 720w,	1600s, 1255w, 1035s, 690s,	1570w, 1230w, 1005s, 620s,

Table II. Proton Magnetic Resonance Data^{a,b}

Compound		
Lu(BH ₃ CH ₃) ₃ ·OEt ₂	0.39 (9H, quart) 1.71 (9H, quart)	0.78 (6H, mult) 3.27 (4H, mult)
Yb(BH ₃ CH ₃) ₃ ·OEt ₂	-162.83 (9H, s), +36.38 (6H, s),	-25.07 (9H, s), +92.65 (4H, s)
Ho(BH ₃ CH ₃) ₃ ·OEt ₂ ^c	-220.70 (4H, s), +84.80 (9H, s)	-95.61 (6H, s),
Lu(BH ₃ CH ₃) ₃ ·THF	0.41 (9H, quart) 1.84 (9H, quart)	0.81 (4H, mult) 3.33 (4H, mult)
Yb(BH ₃ CH ₃) ₃ ·THF	-160.4 (9H, s) 38.3 (4H, s)	-29.4 (9H, s) 82.2 (4H, s)
Ho(BH ₃ CH ₃) ₃ ·THF ^c	-178.61 (4H, s) 77.11 (9H, s)	-87.8 (4H, s)
Yb(BH ₃ CH ₃) ₃ ·2THF	-69.21 (9H, s) 8.10 (8H, s)	-7.77 (8H, s) 26.36 (9H, s)
Ho(BH ₃ CH ₃) ₃ ·2THF	-148.08 (9H, s) -13.15 (8H, s)	-35.83 (8H, s) 31.67 (9H, s)
Ho(BH ₃ CH ₃) ₃ ·2py ^d	-206.53 (9H, s), -1.77 (4H, s), +22.95 (9H, s)	-30.65 (4H, s), +3.13 (2H, s)
Ho(BH ₃ CH ₃) ₃ ·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-d₆.

b) Shifts in ppm from tetramethylsilane. Positive sign indicates downfield shift. All values are at 30°C.

c) BH₃ peak could not be found.

d) [Ho(BH₃CH₃)₃·2py] = 2.3 x 10⁻² g/mL.

Table III. Positional Parameters with Estimated Standard Deviations^a

Atom	$(\text{CH}_3\text{BH}_3)_3\text{Yb}\cdot\text{O}(\text{C}_2\text{H}_5)_2$		
	x	y	z
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)

Table III (continued)

$$\text{(CH}_3\text{BH}_3\text{)}_3\text{Ho}\cdot 2\text{(NC}_5\text{H}_5\text{)}$$

Atom	x	y	z
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.

Table IV. Selected Distances and Angles

Atoms	Dist(A)
Yb - O	2.230(10)
Yb - B(1)	2.405(16)
Yb - B(2)	2.429(15)
Yb - B(3)	2.400(16)
B(1) - C(1)	1.665(20)
B(2) - C(2)	1.612(18)
B(3) - C(3)	1.633(20)

Atoms	Dist(A)
Ho - B(1)	2.483(6)
Ho - B(2)	2.520(5)
Ho - B(3)	2.495(6)
Ho - N(1)	2.535(4)
Ho - N(2)	2.555(4)
B(1) - C(1)	1.596(7)
B(2) - C(2)	1.597(7)
B(3) - C(3)	1.593(8)

Atoms	Angle(°)	Atoms	Angle(°)	Atoms	Angle(°)
O -Yb -B(1)	102.2(5)	N(1)-Ho-N(2)	171.27(11)	Ho-N(1)-C(4)	121.5(3)
O -Yb -B(2)	103.8(5)	N(1)-Ho-B(1)	95.44(15)	Ho-N(1)-C(8)	122.1(3)
O -Yb -B(3)	104.7(5)	N(1)-Ho-B(2)	88.66(14)	Ho-N(2)-C(9)	123.0(3)
B(1)-Yb -B(2)	113.6(5)	N(1)-Ho-B(3)	88.91(15)	Ho-N(2)-C(13)	120.6(3)
B(1)-Yb -B(3)	115.1(6)	N(2)-Ho-B(1)	93.14(15)	Ho-B(1)-C(1)	177.2(4)
B(2)-Yb -B(3)	115.3(5)	N(2)-Ho-B(2)	88.73(14)	Ho-B(2)-C(2)	177.8(4)
Yb -O -C(4)	119.4(9)	N(2)-Ho-B(3)	86.06(16)	Ho-B(3)-C(3)	176.4(5)
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)				

Figure Captions

Fig 1: ORTEP [17] drawing of $(\text{CH}_3\text{BH}_3)_3\text{Yb}\cdot\text{O}(\text{C}_2\text{H}_5)_2$; thermal ellipsoids at 50% probability level.

Fig 2: ORTEP [17] drawing of $(\text{CH}_3\text{BH}_3)_3\text{Ho}\cdot 2(\text{NC}_5\text{H}_5)$; thermal ellipsoids at 50% probability level.

XBL 8410-4298

Figure 1

XBL 8410-4300

Figure 2

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