THE FORMATION AND MOLECULAR STRUCTURES OF $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ AND $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$

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Summary

A reaction between YCl_3 and excess sodium cyclopentadienide in THF solution followed by crystallization from this solvent affords $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ in 68% yield. The corresponding lanthanum complex $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$ can be obtained from $LaCl_3$ and C_5H_5Na in 76% yield via a similar procedure. The products have been characterized by their proton NMR and mass spectra, and by single crystal X-ray diffraction investigations. $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ [$(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$] crystallizes in the monoclinic space group $P2_1/n$ [$P2_1/n$] with unit cell parameters $a = 8.170(3)$ [8.371(4)], $b = 24.594(5)$ [24.636(5)], $c = 8.260(3)$ [8.454(3)] Å, $\beta = 101.32(3)$ [101.84(3)]°, $D_c = 1.45$ [1.58] g cm^{-3} for $Z = 4$ [4]. Least-squares refinement has led to a final R value of 0.041 [0.088] based on 1992 [2193] independent observed reflections. The THF molecule is coordinated to the yttrium [lanthanum] atom at a Y—O(La—O) bond length of 2.451(4) [2.57(1)] Å. The Y—C(Cp)[La—C(Cp)] bond distances average 2.71(3) [2.82(4)] Å. The title compounds are isostructural with $(\eta^5\text{-}C_5H_5)_3Gd \cdot OC_4H_8$.

Introduction

Concurrent with the development of organotransition metal chemistry, there has also been a rapid growth in organolanthanide chemistry in recent years [1,2]. Strictly speaking, the lanthanides include the 14 elements that follow lanthanum in the periodic table, although the Group IIIB metals

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yttrium and lanthanum which contain no 4f electrons are often included in discussions of lanthanide chemistry [3]. During the course of our recent joint studies on the formation and molecular structures of organic derivatives of the Group IVB metals (Ti, Zr, Hf) [4—10], it was of interest to examine comparative properties of the Group IIIB metals (Sc, Y, La). A scarcity of structural data currently exists in this area, including structural investigations only for $(C_5H_5)_3Sc$ [11], $[(\eta^5-C_5H_5)_2ScCl]_2$ [12], $(\eta^5-C_5H_5)_2YMe_2AlMe_2$ [13], $[(\eta^5-C_5H_5)_2YMe]_2$ [14], $[(\eta^5-C_5H_4SiMe_3)_2YCl]_2$ [15], and $[(\eta^5-C_5H_4SiMe_3)_2YMe]_2$ [15]. To our knowledge, no structural information on organic derivatives of lanthanum has been reported up to the present time.

Results and discussion

$(C_5H_5)_3Sc$, $(C_5H_5)_3Y$ and $(C_5H_5)_3La$ were first prepared by Birmingham and Wilkinson in 1955 [16]. Although the syntheses were carried out in THF solution, the solvent-free products were obtained by vacuum sublimation. Molecular weight measurements in THF solution indicated that both $(C_5H_5)_3Sc$ and $(C_5H_5)_3Y$ were monomeric. Magnetic susceptibility and chemical reactivity data suggested that the metal-to-ring bonds in these complexes were essentially ionic in nature.

We have measured the mass spectra of $(C_5H_5)_3Y$ and $(C_5H_5)_3La$, and the results are summarized in Table 1. Both compounds, as expected, exhibit molecular ions at m/e 284 and 334, respectively, although the most intense organometallic ions are the $(C_5H_5)_2M^+$ species in each case. Major peaks for $C_5H_5M^+$, $C_5H_6^+$, and $C_5H_5^+$ fragments are also observed. Furthermore, the proton NMR spectra of $(C_5H_5)_3Y$ and $(C_5H_5)_3La$ in THF solution exhibit singlet resonances for the equivalent cyclopentadienyl ring hydrogens at δ 5.88 and 5.97 ppm, respectively.

Structural studies on $(C_5H_5)_3Sc$ were reported by Atwood and Smith in 1973, utilizing single crystals of the compound which were grown by sublimation [11]. The structure consisted of $(\eta^5-C_5H_5)_2Sc$ units which were bridged together by the remaining cyclopentadienyl groups in a polymeric manner. Our current attempts to obtain single crystals of $(C_5H_5)_3Y$ and $(C_5H_5)_3La$ by sublimation techniques were not successful. However, crystals suitable for X-ray diffraction studies could be grown by slow cooling of $(C_5H_5)_3Y$ or $(C_5H_5)_3$La from THF solution. The products were obtained in the form of THF adducts,

TABLE 1

MASS SPECTRA OF $(C_5H_5)_3Y$ AND $(C_5H_5)_3La$ [a]

$(C_5H_5)_3Y$			$(C_5H_5)_3La$		
m/e	Rel. intensity	Assignment	m/e	Rel. intensity	Assignment
284	16.1	$(C_5H_5)_3Y^+$	334	13.3	$(C_5H_5)_3La^+$
219	93.5	$(C_5H_5)_2Y^+$	269	86.7	$(C_5H_5)_2La^+$
154	6.5	$C_5H_5Y^+$	204	30.0	$C_5H_5La^+$
66	100	$C_5H_6^+$	66	100	$C_5H_6^+$
65	74.2	$C_5H_5^+$	65	83.3	$C_5H_5^+$

[a] Recorded at 70 eV.
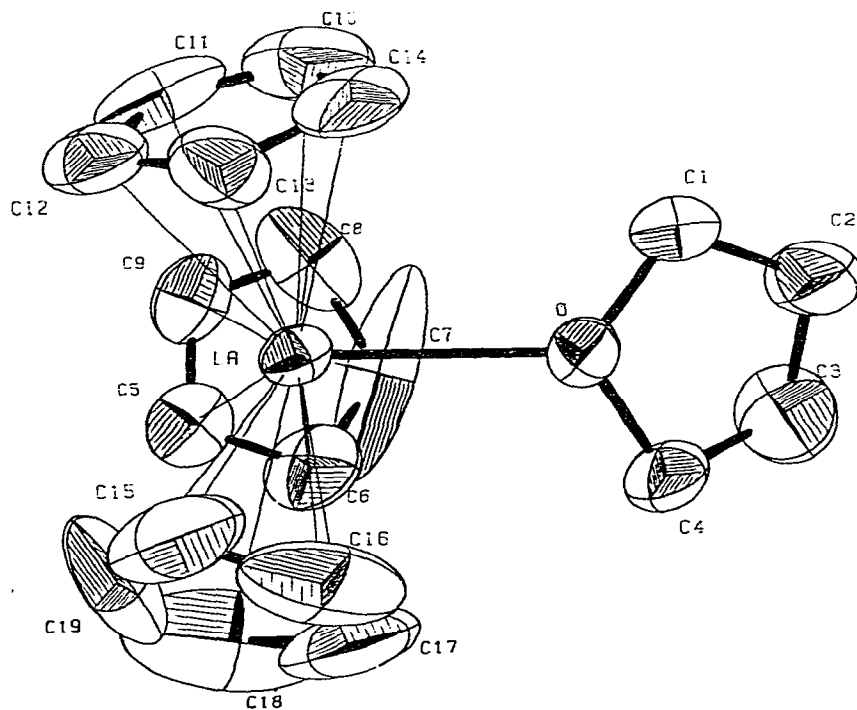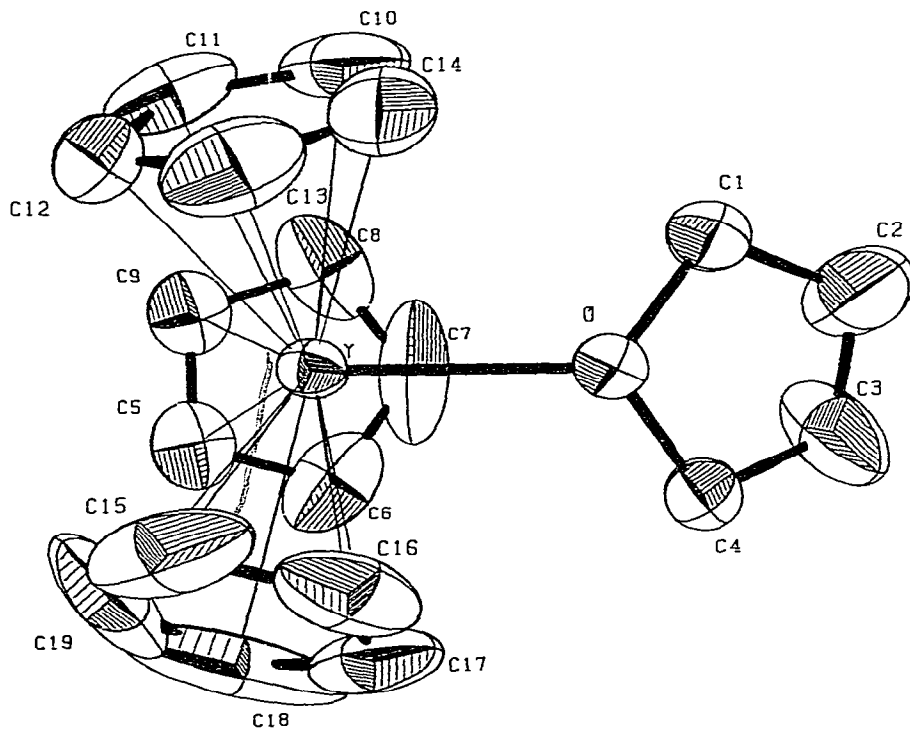
Fig. 1. Molecular structure and atom numbering scheme for (A) $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ and (B) $(\eta^5\text{-}C_5H_5)_3\text{-}La \cdot OC_4H_8$.

TABLE 2

COMPARISON OF BOND LENGTHS (Å) AND ANGLES (°) FOR $(\eta^5\text{-}C_5H_5)_3M \cdot OC_4H_8$.

Atoms	Bond length	
	M = Y	M = La
M—O	2.451(4)	2.57(1)
M—C(5)	2.693(6)	2.82(2)
M—C(6)	2.703(7)	2.76(3)
M—C(7)	2.722(7)	2.84(2)
M—C(8)	2.718(7)	2.84(2)
M—C(9)	2.689(7)	2.87(2)
M—C(10)	2.693(7)	2.82(2)
M—C(11)	2.691(6)	2.81(2)
M—C(12)	2.715(6)	2.83(2)
M—C(13)	2.766(7)	2.85(2)
M—C(14)	2.732(7)	2.87(2)
M—C(15)	2.692(8)	2.80(2)
M—C(16)	2.722(6)	2.88(2)
M—C(17)	2.715(7)	2.86(2)
M—C(18)	2.711(9)	2.79(3)
M—C(19)	2.65(1)	2.72(3)
M—Cent 1	2.438	2.575
M—Cent 2	2.453	2.575
M—Cent 3	2.454	2.576

Atoms	Angle	
	M = Y	M = La
M—O—C(1)	125.0(3)	125.6(9)
M—O—C(4)	123.7(3)	122(1)
Cent 1—M—O	96.4	96.2
Cent 2—M—O	100.8	102.2
Cent 3—M—O	100.7	100
Cent 1—M—Cent 2	119.2	120
Cent 1—M—Cent 3	117.5	117
Cent 2—M—Cent 3	115.4	116

$(C_5H_5)_3Y \cdot OC_4H_8$ and $(C_5H_5)_3La \cdot OC_4H_8$. The ability of the lanthanide series complexes $(C_5H_5)_3Ln$ (Ln = Eu, Tb, Ho, Yb) to form analogous THF adducts has been known for some time [17,18], and a crystallographic investigation of $(C_5H_5)_3Gd \cdot OC_4H_8$ has recently been completed [19].

The molecular structure and atom numbering schemes are shown in Figure 1, and a comparison of bond lengths and angles is presented in Table 2. The Y—O bond distance, 2.451(4) Å, is significantly larger than the 2.37 Å average in $Y(BH_4)_3(OC_4H_8)_3$ [20]. However, both the Y—O and La—O lengths compare favorably with the Gd—O distance of 2.494(7) Å after an appropriate correction for the 3+ ionic radii [21].

The Y—C(η^5) bond distances range from 2.65(1) to 2.77(1) Å and average 2.71(3) Å. This is longer than the values reported for $[(\eta^5\text{-}C_5H_4SiMe_3)_2YMe]_2$, 2.68 Å [15], for $[(\eta^5\text{-}C_5H_4SiMe_3)_2YCl]_2$, 2.65(1) Å [15], and for $(\eta^5\text{-}C_5H_5)_2$-YMe_2AlMe_2, 2.62(4) Å [13]. Since one would expect the Y—C(η^5) lengths for the SiMe$_3$-substituted cyclopentadienyl groups to be longer than for the $C_5H_5^-$

ligand itself, one must conclude that a severe steric problem results from the crowding of three η^5-cyclopentadienyl groups and one tetrahydrofuran ligand around the yttrium atom. This view is reinforced by a comparison with the structure of $(\eta^5\text{-}C_5H_5)_2LuBu^t \cdot OC_4H_8$ [22]. Although Lu^{3+} is only ca. 0.04 Å smaller in radius than Y^{3+}, the $Lu—C(\eta^5)$ average is 2.64 Å, and, more significantly, the $Lu—O$ length is 2.30(2) Å.

Within the isostructural series $(\eta^5\text{-}C_5H_5)_3M \cdot OC_4H_8$, M = Y, Gd [19], or La, the M-centroid distances (Table 2) agree to within 0.01 after radii corrections: $Y—centroid = 2.45$ Å, $Gd—centroid = 2.48$ Å, and $La—centroid = 2.58$ Å. Similarly, close equivalence is also noted for the angles which involve the centroids.

Experimental section

All reactions were carried out in an argon atmosphere. The argon was dried with H_2SO_4 and P_2O_5, and trace amounts of oxygen were removed using BTS catalyst. Tetrahydrofuran (THF) was predried with potassium hydroxide, then with sodium, and finally distilled under argon from sodium/benzophenone. Proton NMR spectra were recorded on a Varian A-60 spectrometer and are referenced to external TMS. Mass spectra were recorded on a Perkin-Elmer-Hitachi RMU 6L mass spectrometer. YCl_3 and $LaCl_3$ were obtained from Research Organic/Inorganic Chemicals Corp.

Preparation of $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$

C_5H_5Na (0.152 mol) was prepared in the usual manner from cyclopentadiene and sodium sand in 150 ml of THF at room temperature. The solution was cooled to 0°C, and 7.5 g (0.030 mol) of $LaCl_3$ were added. The resulting mixture was refluxed for 3 h, cooled to room temperature and allowed to stir overnight. The reaction mixture was filtered, giving a purple-red solution which on removal of the solvent produced a pink powder. This was placed in a Schlenk-type sublimer and the product sublimed at $260°C/10^{-3}$ Torr. The white sublimate was only slightly soluble in THF at room temperature, but very soluble on warming in this solvent. White crystals suitable for X-ray crystallographic studies were grown by allowing the warm THF solution to slowly cool to room temperature. The solution was then cooled to 0°C and finally to —20°C, at which point white platelets crystallized. The yield of $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$ was 76%.

Preparation of $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$

$(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ was obtained in a similar manner from C_5H_5Na, YCl_3 and THF. The product was sublimed at $160—180°C/10^{-3}$ Torr. White crystals suitable for X-ray analysis were grown from a warm THF solution. The yield was 68%.

X-ray data collection, structure determination, and refinement for $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$

Single crystals of the air-sensitive compound were sealed under N_2 in thin-walled glass capillaries. Final lattice parameters as determined from a least-squares refinement of $((\sin \theta)/\lambda)^2$ values for 15 reflections ($\theta > 20°$) accurately

TABLE 3

CRYSTAL DATA AND SUMMARY OF INTENSITY DATA COLLECTION AND STRUCTURE REFINEMENT

Compound	$(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$	$(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$	$(\eta^5\text{-}C_5H_5)_3Gd \cdot OC_4H_8$ [a]
Mol.wt.	356.30	405.40	
Space group	$P2_1/n$	$P2_1/n$	$P2_1/n$
Cell constants			
a (Å)	8.170(3)	8.371(4)	8.220(4)
b (Å)	24.594(5)	24.636(5)	24.650(9)
c (Å)	8.260(3)	8.454(3)	8.317(4)
β (deg)	101.32(3)	101.84(3)	101.39(3)
Cell vol. (Å^3)	1627.4	1706.4	1652.0
Molecules/unit cell	4	4	
ρ(calc) (g cm^{-3})	1.45	1.58	
μ(calc) (cm^{-1})	36.89	25.41	
Radiation	$MoK\alpha$	$MoK\alpha$	
Max crystal dimensions (mm)	0.50 X 0.43 X 0.75	0.20 X 0.45 X 0.55	
Scan Width (deg)	$0.80 + 0.20 \tan \theta$	$0.80 + 0.20 \tan \theta$	
Standard reflections	600 004	600 004	
Variation of standards	±2%	±2%	
Reflections measured	3074	3069	
2θ range (σ)	1—50	1—50	
Reflections, $I > 3\sigma(I)$	1992	2193	
No. of Parameters varied	190	190	
Largest parameter shift/esd	0.05	0.30	
GOF	1.58	1.83	
R	0.041	0.088	
R_w	0.044	0.087	

[a] Ref. 19.

centered on the diffractometer are given in Table 3. The space group was uniquely determined as $P2_1/n$ from the systematic absences in $0k0$ for $k = 2n + 1$ and $k0l$ for $h + l = 2n + 1$.

Data were collected on an Enraf-Nonius CAD-4 diffractometer by the θ—2θ scan technique. The method has been previously described [23]. A summary of data collection parameters is given in Table 3. The intensities were corrected for Lorentz, polarization, and for absorption effects [24].

Calculations were carried out with the SHELX system of computer programs [25]. Neutral atom scattering factors for Y, O and C were taken from Cromer

TABLE 4(a)

FINAL FRACTIONAL COORDINATES FOR $(\eta^5 C_5 H_5)_3 Y \cdot OC_4 H_8$

Atom	x/a	y/b	z/c
Y	0.92409(6)	0.14386(2)	0.05575(6)
O	1.1101 (5)	0.0830 (2)	0.2425 (4)
C(1)	1.0911 (8)	0.0678 (3)	0.4090 (7)
C(2)	1.256 (1)	0.0495 (5)	0.4960 (9)
C(3)	1.3693 (9)	0.0631 (5)	0.405 (1)
C(4)	1.2826 (8)	0.0729 (3)	0.2318 (9)
C(5)	1.0266 (9)	0.2476 (3)	0.0432 (9)
C(6)	1.1667 (9)	0.2200 (3)	0.121 (1)
C(7)	1.141 (1)	0.2045 (3)	0.275 (1)
C(8)	0.985 (1)	0.2228 (3)	0.2903 (9)
C(9)	0.8146 (9)	0.2486 (3)	0.1468 (9)
C(10)	0.7056 (9)	0.1212 (5)	0.2518 (9)
C(11)	0.6264 (9)	0.1616 (3)	0.139 (1)
C(12)	0.5859 (8)	0.1368 (4)	−0.0136 (9)
C(13)	0.6330 (9)	0.0834 (4)	0.005 (1)
C(14)	0.7042 (9)	0.0740 (4)	0.157 (1)
C(15)	0.851 (1)	0.1030 (7)	−0.252 (1)
C(16)	0.977 (1)	0.0715 (3)	−0.1779 (9)
C(17)	1.1129 (9)	0.0030 (6)	−0.1519 (9)
C(18)	1.072 (3)	0.1503 (5)	−0.210 (2)
C(19)	0.911 (3)	0.1526 (7)	−0.267 (1)
H(1) [C(1)]	1.055	0.104	0.471
H(2) [C(1)]	0.994	0.038	0.405
H(3) [C(2)]	1.251	0.003	0.504
H(4) [C(2)]	1.284	0.064	0.621
H(5) [C(3)]	1.431	0.101	0.457
H(6) [C(3)]	1.468	0.033	0.413
H(7) [C(4)]	1.292	0.036	0.156
H(8) [C(4)]	1.334	0.106	0.174
H(9) [C(5)]	1.008	0.266	−0.081
H(10) [C(6)]	1.279	0.211	0.067
H(11) [C(7)]	1.230	0.182	0.368
H(12) [C(9)]	0.789	0.267	0.118
H(14) [C(10)]	0.756	0.125	0.384
H(15) [C(11)]	0.602	0.204	0.167
H(16) [C(12)]	0.526	0.156	−0.128
H(17) [C(13)]	0.611	0.053	−0.097
H(18) [C(14)]	0.758	0.034	0.202
H(19) [C(15)]	0.719	0.088	−0.293
H(20) [C(16)]	0.969	0.028	−0.144
H(21) [C(17)]	1.240	0.089	−0.091
H(22) [C(18)]	1.157	0.184	−0.213
H(23) [C(19)]	0.830	0.186	−0.319

TABLE 4(b)

FINAL FRACTIONAL COORDINATES FOR $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$

Atom	x/a	y/b	z/c
La	0.9203(1)	0.14467(3)	0.0550(1)
O	1.113 (1)	0.0816 (5)	0.249 (1)
C(1)	1.069 (2)	0.0669 (8)	0.415 (2)
C(2)	1.255 (3)	0.047 (1)	0.501 (2)
C(3)	1.367 (2)	0.065 (1)	0.408 (4)
C(4)	1.285 (2)	0.0732 (9)	0.235 (3)
C(5)	1.030 (3)	0.2530 (7)	0.052 (3)
C(6)	1.162 (3)	0.223 (1)	0.111 (3)
C(7)	1.148 (5)	0.213 (1)	0.256 (7)
C(8)	0.986 (4)	0.2279 (9)	0.292 (3)
C(9)	0.925 (3)	0.2551 (8)	0.162 (3)
C(10)	0.685 (3)	0.127 (2)	0.245 (3)
C(11)	0.614 (3)	0.163 (1)	0.134 (4)
C(12)	0.576 (2)	0.135 (1)	−0.021 (2)
C(13)	0.627 (2)	0.0827 (8)	0.001 (2)
C(14)	0.686 (2)	0.076 (1)	0.160 (4)
C(15)	0.856 (3)	0.098 (2)	−0.253 (3)
C(16)	0.975 (5)	0.071 (1)	−0.193 (3)
C(17)	1.108 (3)	0.103 (2)	−0.164 (3)
C(18)	1.069 (8)	0.147 (1)	−0.211 (7)
C(19)	0.921 (8)	0.152 (2)	−0.266 (3)
H(1) [C(1)]	1.011	0.037	0.411
H(2) [C(1)]	1.063	0.099	0.471
H(3) [C(2)]	1.254	0.006	0.507
H(4) [C(2)]	1.282	0.062	0.612
H(5) [C(3)]	1.416	0.099	0.453
H(6) [C(3)]	1.454	0.036	0.414
H(7) [C(4)]	1.296	0.040	0.168
H(8) [C(4)]	1.329	0.105	0.188
H(9) [C(5)]	1.008	0.271	−0.055
H(10) [C(6)]	1.249	0.210	0.054
H(11) [C(7)]	1.238	0.196	0.338
H(12) [C(8)]	0.938	0.219	0.388
H(13) [C(10)]	0.728	0.133	0.362
H(15) [C(11)]	0.591	0.201	0.153
H(16) [C(12)]	0.521	0.152	−0.125
H(17) [C(13)]	0.611	0.054	−0.084
H(19) [C(15)]	0.740	0.085	−0.285
H(20) [C(16)]	0.974	0.031	−0.167
H(21) [C(17)]	1.221	0.091	−0.111
H(22) [C(18)]	1.148	0.177	−0.208
H(23) [C(19)]	0.859	0.185	−0.309

and Waber [26], and the scattering for yttrium was corrected for the real and imaginary components of anomalous dispersion using the table of Cromer and Liberman [27]. Scattering factors for H were from ref. 28.

Since $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ is isostructural with its Gd analogue, the coordinates of the atoms in the latter formed the basis for the structural model of the former. Least-squares refinement with isotropic thermal parameters led to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.092$. The hydrogen atoms of the cyclopentadienyl rings and the THF ring were placed at calculated positions 1.08 Å from the bonded carbon atom and were not refined. Refinement of the nonhydrogen atoms with anisotropic temperature factors and of the positional parameters

of the hydrogen atoms led to final values of $R \approx 0.041$ and $R_w = 0.044$. A final difference Fourier showed no feature greater than $0.3\ e^-/\text{Å}^3$. The weighting scheme was based on unit weights; no systematic variation of $w(|F_0| - |F_c|)$ vs. $|F_0|$ or $(\sin\theta)/\lambda$ was noted. The final values of the positional parameters are given in Table 4 *.

X-ray data collection, structure determination, and refinement for $(\eta^5\text{-}C_5H_5)_3La \cdot OC_4H_8$

Data collection procedures were identical to those of the yttrium analogue. The atomic positions from $(\eta^5\text{-}C_5H_5)_3Y \cdot OC_4H_8$ were used as the starting point for the least-squares refinement. The R factor based on isotropic thermal parameters with no hydrogen atoms was 0.117. Hydrogen atoms were treated in the same manner as above. The final features of the refinement are listed in Table 3 and the positional and thermal parameters are given in Table 4. The higher R factors on the La compound compared to the Y derivative appear to be due to two problems. First, the crystals of the former were of poorer quality than of the latter. (Even though many were tried and the best one selected.) Second, the larger La ion seems to permit slightly higher libration of the cyclopenta-dienyl groups. This is shown clearly in Fig. 1.

A second method of refinement was employed in an effort to improve the agreement indices. The cyclopentadienyl rings were treated as rigid groups with anisotropic thermal parameters assigned to the individual carbon atoms. The agreement factors based on this model were $R = 0.091$ and $R_w = 0.092$. Since these were less acceptable than those of the above mentioned unconstrained refinement, the parameters reported refer to the latter.

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