THE SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF THE CYCLOPENTADIENYLALUMINUM ISOPROPOXIDE COMPOUNDS $[(C_5H_5)_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ AND $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$

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Abstract

The cyclopentadienylaluminum alkoxide compounds $[(C_5H_5)_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**1**) and $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**2**) have been prepared by reacting $(C_5H_5)_2Mg$ and $(C_5Me_4H)_2Mg$, respectively, with $Cl_2AlO\text{-}i\text{-}Pr$. Whereas replacement of both chloride ligands on aluminum by (C_5H_5) was achieved to form **1**, only one chloride ligand could be substituted by the bulkier (C_5Me_4H) to produce the mono(tetramethylcyclopentadienyl)aluminum compound **2**.

Introduction

The considerable interest in the fluxional nature and bonding of cyclopentadienylaluminum compounds notwithstanding,[1] little structural data on cyclopentadienylaluminum compounds has been available until very recently.[2] In particular, examples of dicyclopentadienylaluminum compounds are relatively scarce.[3] Prior to our synthesis of the compounds described in this paper, only two molecular structures of dicyclopentadienylaluminum compounds had been reported, our recently reported structure of Cp_2AlMe ($Cp = C_5H_5$)[3a] and the structure of the cationic metallocene $[Cp^*{}_2Al][Cp^*AlCl_3]$ recently reported by Schnöckel and coworkers.[3b] The unusual dihapto coordination geometry of the cyclopentadienyl rings that was found for Cp_2AlMe in the solid state prompted us to explore further the synthesis of dicyclopentadienylaluminum derivatives in order to determine the prevalence of η^2-Cp coordination in these compounds and to investigate the role of the formally empty *p* orbital on the metal in stabilizing this mode of ring coordination. With respect to the latter issue, we were interested in attaching a heteroatom to the dicylopentadienylaluminum fragment since it would have the potential, however small,[4] to achieve π-overlap with the vacant *p* orbital and thereby reduce its interaction with the cyclopentadienyl rings. In our initial efforts to explore this question we prepared and crystallographically characterized $[Cp_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2$, which was selectively crystallized from other products formed upon reacting Cp_2Mg with *in situ* generated dichloraluminum isopropoxide. While we were preparing to publish this work, Kunicki *et. al* reported the crystal structure of this compound,[5] which they prepared by an analogous method involving the reaction of NaCp with Cl_2Al-O-*i*-Pr. The crystallographic data that was reported for the dicyclopentadienylaluminum isopropoxide dimer is essentially identical to our own.[6] Therefore we will limit our discussion of this compound to our alternate synthesis and additional findings and observations. In trying to make the analogous bis(tetramethylcyclopentadienyl)aluminum isoproxide complex by replacing Cp_2Mg with $(C_5Me_4H)_2Mg$ in the reaction, we instead obtained $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**2**), in which only one chloride on the aluminum was replaced by a cyclopentadienyl ring. This alkoxide bridged dimer was also crystallographically characterized, and the structural data is described herein.

Results and Discussion

Dicyclopentadienylaluminum isopropoxide, **1**, was prepared according to equation (1) by adding magnesocene to a toluene solution of Cl_2AlO-*i*-Pr, which was generated *in situ* by comproportionating two equivalents of trichloroaluminum with one equivalent of triisopropoxyaluminum.

$$2AlCl_3 + AlO\text{-}i\text{-}Pr_3 \xrightarrow{\text{toluene}} 2\ Cl_2AlO\text{-}i\text{-}Pr \xrightarrow[\text{- }MgCl_2]{Cp_2Mg} [Cp_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2 \qquad (1)$$

The ^1H NMR spectrum of the crude reaction mixture displayed at least three other reaction products besides **1**. Based on the chemical shifts of the different species, we suspect one other oligomer, possibly a trimer, of $Cp_2Al(O\text{-}i\text{-}Pr)$ and oligomers of $CpAl(O\text{-}i\text{-}Pr)_2$ to be present. Compound **1** was selectively recrystallized in 26% overall yield from a toluene solution of the mixture cooled at -78 °C. As mentioned, the X-ray structure of this compound has already been reported. Nevertheless, an ORTEP drawing of the molecule is shown in Figure **1** for purposes of comparison with the molecular structure of $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**2**) (*vide infra*), which is shown in Figure 2. The dimensions of the Al_2O_2 core, its planarity, the Al–O distances and the O–Al–O and Al–O–Al bond angles for **1** are consistent with that of other dimeric aluminum alkoxide compounds.[7] The overall geometry of another bis(cyclopentadienyl) group 13 element alkoxide, $[Cp_2Ga(\mu\text{-}OEt)]_2$,[8] is very similar that of compound **1**. While the bond angles of the Ga_2O_2 core of this molecule are comparable to that of compound **1**, the Ga–O bond lengths are slightly longer than the Al–O bond lengths in **1**. A similar difference in Ga–O *vs.* Al–O bond lengths is exhibited in monomeric aluminum and gallium alkoxide compounds reported by Power and coworkers.[4a] Another similarity between the molecular structures of **1** and $[Cp_2Ga(\mu\text{-}OEt)]_2$ is the η^1-

coordination of the cyclopentadienyl rings to the metal. The presence of discrete single and double bonds in the cyclopentadienyl rings can be inferred from their C–C bond distances. Despite the η^1-Cp coordination geometry exhibited in the solid state structure of **1**, a single resonance for the rings is observed in the ^{1}H and ^{13}C solution NMR spectra of the compound due to a fluxional process which averages the environments of all of the carbons and protons of the rings. The $\delta(^{27}Al)$ for the compound of 103 ppm (relative to an $Al(OH)_3$ external standard) is considerably downfield of the ^{27}Al NMR shift for the the tricoordinate aluminum in Cp_2AlMe and more comparable to the ^{27}Al NMR shifts of the tetracoordinate aluminums in $Cp_3Al(L)$ (L = C=N*t*-Bu, N=CCMe$_3$).[2a] This downfield shifting of ^{27}Al NMR δ values for the tetracoordinate cyclopentadienylaluminum compounds relative to tricoordinate aluminum runs counter to the trend typically observed for organoaluminum and aluminum alkoxide compounds.[9] We attribute this anomaly in the chemical shifts to the aromatic character of the cyclopentadienyl ring attached to the aluminum since this has be shown to cause considerable anisotropic upfield shifting of the ^{27}Al NMR signals in other cyclopentadienylaluminum compounds.[3b,10]

In order to determine whether bulkier cyclopentadienyl ligands could promote monomerization of the dicyclopentadienylaluminum isopropoxide species, $(C_5Me_4H)_2Mg$ was used in place of magnesocene in the reaction with *in situ* generated $Cl_2Al(O\text{-}i\text{-}Pr)$. As shown in equation (2), the steric bulk of the tetramethylcyclopentadienyl ligand interferes instead with the attachment of two rings to the dimeric aluminum framework, forming $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**2**) exclusively and leaving one untransferred cyclopentadienyl ring on the magnesium.

$$Cl_2AlO\text{-}i\text{-}Pr \xrightarrow[-\ 1/2\ MgCl_2]{(C_5Me_4H)_2Mg} [(C_5Me_4H)ClAl(\mu\text{-}O\text{-}i\text{-}Pr)]_2 + \frac{1}{x}\,[(C_5Me_4H)MgCl]_x \qquad (2)$$

Use of half of an equivalent of the bis(tetramethylcyclopentadienyl)magnesium reagent per aluminum also affords compound **2**, along with $MgCl_2$, which can be removed by filtration.

The dimensions of the Al_2O_2 core of compound **2** in the solid state are similar to those of compound **1**. An ORTEP drawing of the molecule is shown in Figure 2. Fractional atomic coordinates are listed in Table II. Selected bond lengths and bond angles are listed Table III. The two tetramethylcyclopentadienyl rings in the dimer avoid eachother by adopting a *trans* arrangement about the Al_2O_2 plane. One can see from the molecular structure that the tetramethylcyclopentadienyl rings effectively block the remaining chloride ligands from further substitution. The ^{27}Al NMR signal for **2**, at δ 85, occurs at a higher field than that of compound **1**. This may be due to greater electron donation from the tetramethylcyclopentadienyl ring to the aluminum.

Table I. Crystallographic Data for $[(C_5Me_4H)AlCl(\mu\text{-}O\mathit{i}Pr)]_2$ (2)

formula	$C_{24}H_{40}Al_2Cl_2O_2$	*T*, K	296
fw	485.4	μ, cm^{-1}	1.54
lattice type	monoclinic	radiation	Mo Kα (0.71073 Å)
space group	$P2_1/n$	indpt rflns	1575 (R_{int} = 0.96%)
Z	2	indpt obsd rflns	1203 ($F > 5\sigma(F)$)
a, Å	9.275(2)	$R(F_o)$, %	5.93
b, Å	12.922(3)	$Rw(F_o)$, %	6.36
c Å	11.494(3)	GOF	1.49
β, °	97.99(2)	Data/Parameter	8.8:1
V, Å^3	1364.1(6)	$\Delta\rho$, eÅ^{-3}	0.24
ρ(calc), g cm^{-3}	1.599	$\Delta\rho$, eÅ^{-3}	-0.21

Table II. Atomic coordinates (x10^4) and equivalent isotropic displacement coefficients (Å^2 x 10^3) for non-hydrogen atoms in 2

	x	y	z	U(eq)*
Al(1)	35(1)	755.8(8)	4155(1)	40(1)
Cl(1)	2124(1)	1320(1)	3902(1)	74(1)
O(1)	-38(2)	647(2)	5727(2)	40(1)
C(1)	-1600(4)	1449(3)	3170(3)	43(1)
C(2)	-2924(4)	937(3)	3462(3)	41(1)
C(3)	-3369(4)	238(3)	2610(3)	44(1)
C(4)	-2465(4)	337(3)	1686(3)	48(2)
C(5)	-1455(4)	1078(3)	1980(3)	48(1)
C(6)	-3632(4)	1162(4)	4536(4)	55(2)
C(7)	-4624(5)	-501(4)	2584(5)	73(2)
C(8)	-2684(5)	-290(4)	569(4)	80(2)

	x	y	z	U(eq)*
C(9)	-405(5)	1532(4)	1216(4)	71(2)
C(10)	195(5)	1449(3)	6650(4)	53(2)
C(11)	-36(5)	2503(3)	6088(4)	76(2)
C(12)	-814(6)	1248(4)	7541(4)	76(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table III. Selected Bond Lengths and Angles for $[(C_5Me_4H)AlCl(\mu\text{-}O\textit{i}Pr)]_2$ (2)

Bond Lengths (Å)		Angles (Å)	
Al(1)–O(1)	1.823(3)	O(1)–Al(1)–O(1a)	81.3(1)
Al(1)–O(1a)	1.817(3)	Al(1)–O(1)–Al(1a)	98.7(1)
Al(1)—Cl(1)	2.127(2)	Al(1)–O(1)–C(10)	129.5(2)
Al(1)–C(1)	1.977(4)	O(1)–Al(1)–Cl(1)	108.8(1)
Al(1)–Al(1a)	2.762(2)	O(1a)–Al(1)–C1(1)	111.1(1)
Cl(1)–C(2)	1.480(5)	Cl(1)–Al(1)–C(1)	114.0(1)
C(1)–C(5)	1.473(6)	Al(1)–C(1)–C(5)	102.8(3)
C(2)–C(3)	1.346(5)	O(1)–Al(1)–C(1)	118.1(1)
C(3)–C(4)	1.447(6)	O(1a)–Al(1)–C(1)	119.2(1)
C(4)–C(5)	1.349(6)	O(1)–Al(1)–O(1a)	81.3(1)

Conclusion

The solid state structural information on the compounds $[Cp_2Al(\mu\text{-}O\text{-}\textit{i}\text{-}Pr)]_2$ and $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}\textit{i}\text{-}Pr)]_2$, combined with the solution NMR data on the compounds, contributes to our understanding of the nature of the bonding in cyclopentadienylaluminum compounds and and to our understanding of their fluxional behavior. Due to the strength of the alkoxide bridge, we were unable to force the dimeric aluminum alkoxide apart to include two bulky tetramethylcyclopentadienyl rings, obtaining instead the mono(tetramethylcyclopentadienyl)aluminum alkoxide dimer **2**. The employment of a bulkier alkoxide substituent which is sterically incapable of forming a bridge will be more conducive to our goal of preparing a monomeric dicyclopentadienylaluminum alkoxide compound that is directly comparable to Cp_2AlMe. We have successfully prepared $Cp_2Al(BHT)$ (BHT = 2,6-di-t-butyl-4-

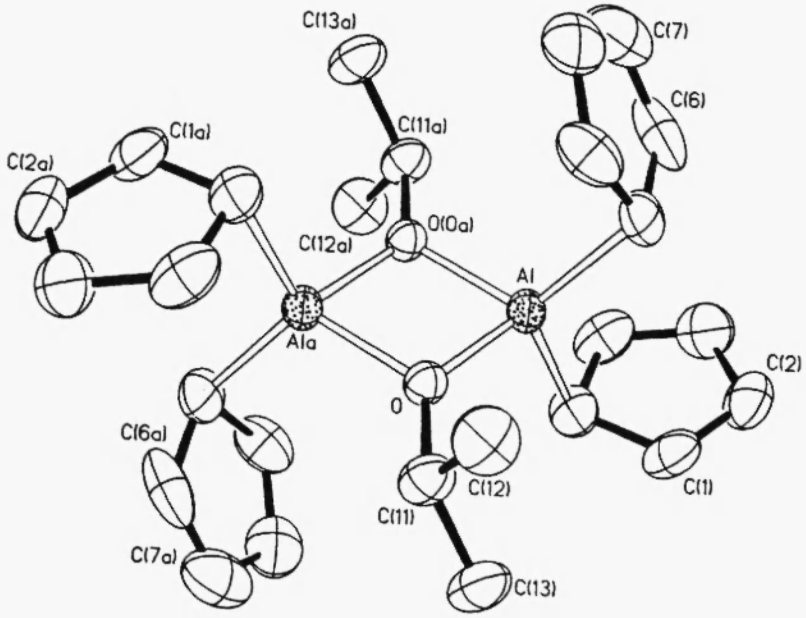

Figure 1. ORTEP drawing of the molecular structure of $[(C_5H_5)_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**1**). Thermal ellipsoids are shown at the 35% probability level. Distances (Å) and angles (°) of Al_2O_2 core: Al–O (1.825(2)), Al–O_a (1.813(2)), O–A–O_a (80.7(1)), Al–O–Al_a (99.3(1)).

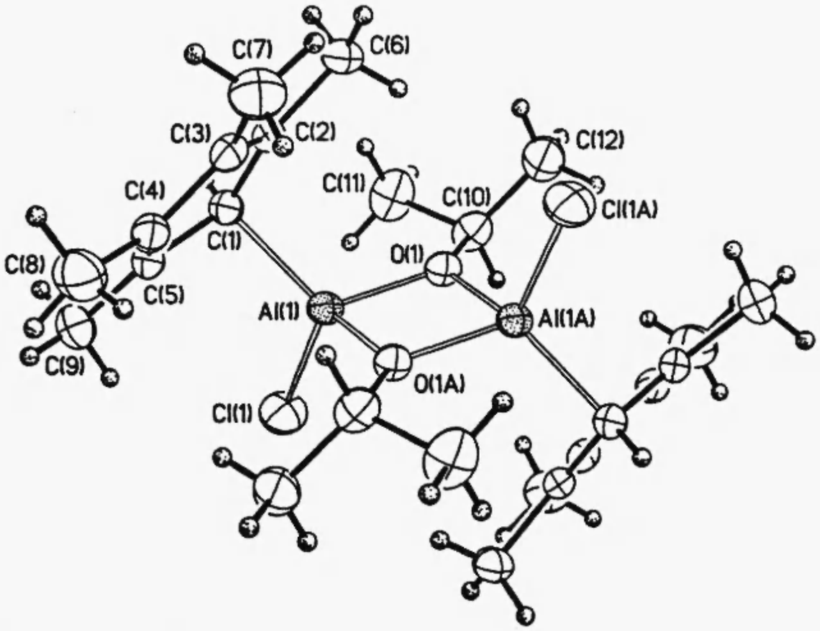

Figure 2. ORTEP drawing of the molecular structure of $[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (**2**). Thermal ellipsoids are shown at the 35% probability level.

methylphenoxide), the monomeric nature of which was confirmed by ebulliometric molecular weight analysis. We are in the process of growing X-ray quality crystals of the compound and anticipate reporting the details of the synthesis and structural characterization of this compound in a future publication.

Experimental Section

General Considerations. All manipulations were performed using a combination of glovebox, high-vacuum, and Schlenk techniques. Toluene and petroleum ether were distilled under nitrogen over sodium benzophenone ketyl and stored in line-pots over sodium benzophenone ketyl, from which they were vacuum transferred. The NMR solvents benzene-d_6 and toluene-d_8 were dried over activated 4 Å molecular sieves. Argon was purified by passage over oxy tower BASF catalyst (Kontes) and 4 Å molecular sieves. Aluminum isopropoxide was used as received from Aldrich. Aluminum chloride (Aldrich) was sublimed prior to use. Magnesocene[11] and octamethylmagnesocene[12] were prepared as described in the literature.

NMR spectra were recorded on an IBM NR-300 (300.13 MHz ^{1}H; 75.46 MHz, ^{13}C; 78.21 MHz, ^{27}Al) and an IBM NR-200 (200.13 MHz, ^{1}H; 50.32 MHz, ^{13}C; 52.15 MHz, ^{27}Al). All chemical shifts are reported in ppm and referenced to solvent (^{13}C, ^{1}H) or $Al(OH)_3$ (^{27}Al, external reference, δ 0 ppm). Elemental analyses were determined by Desert Analytics and the University of Idaho analytical facilities.

$[(C_5H_5)_2Al(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (1). A mixture of aluminum chloride (1.24 g, 9.34 mmol) and aluminum isopropoxide (0.950 g, 4.67 mmol) were brought up in 100 mL of toluene and stirred overnight at 50°C, producing a clear, peach-colored solution. After the solution was allowed to cool to room temperature, solid magnesocene (2.16 g, 14.0 mmol) was added through a side arm attachment. The reaction immediately turned a chalky white but became clear after being heated at 95°C for 48 hrs. The toluene solution was filtered to remove the $MgCl_2$, concentrated to 20 mL and cooled to -78°C to precipitate the product as a white powder (mp 104.8-107.0 °C, uncorrected; yield 0.36 g, 26%). ^{1}H NMR (C_6D_6): δ 6.11 (s, C_5H_5), 3.67 (h, ^3J = 6.3 Hz, $(CH_3)_2CHO$), 0.88 (d, ^3J = 6.3 Hz, $(CH_3)_2CHO$) . ^{13}C NMR (C_6D_6): δ 113.7 (C_5H_5), 70.2 ($(CH_3)_2CHO$), 24.6 ($(CH_3)_2CHO$). ^{27}Al NMR (C_7D_8): δ 103. IR (KBr, Nujol): $\upsilon[\mathrm{cm}^{-1}]$ = 1792 (w), 1605 (w), 1292 (w), 1208(w), 1171 (sh), 1121 (sh), 1108 (s), 971 (s), 931 (s), 828 (s), 839 (s), 753 (s), 667 (s), 509 (sh), 468 (s). Anal. Calcd for $C_{13}H_{17}AlO$: C, 72.20; H, 7.92. Found: C, 72.1; H, 7.8.

$[(C_5Me_4H)AlCl(\mu\text{-}O\text{-}i\text{-}Pr)]_2$ (2). A mixture of aluminum chloride (0.90 g, 6.8 mmol) and aluminum isopropoxide (0.69 g, 3.4 mmol) were brought up in 50 mL of toluene and stirred overnight at 50°C. The solution was cooled to room temperature, and solid magnesocene (1.4g, 5.1 mmol) was added . The reaction immediately turned a chalky white and became red as it was stirred at 45°C for 12 hrs . The solution was filtered to remove the $MgCl_2$, and the toluene was removed under reduced pressure and replaced with 20 mL petroleum ether, from which the product was precipitated at -78°C (mp 166.9 °C, uncorrected; yield 1.3 g, 52%). ^1H NMR (C_6D_6): δ 3.86 (h, ^3J = 6.3 Hz $(CH_3)_2C\mathit{H}O$), 3.37 (br, $C_5Me_4\mathit{H}$), 2.23 (s, $C_5(C\mathit{H}_3)_4H$), 1.89 (s, $C_5(C\mathit{H}_3)_4H$), 1.36 (d, ^3J = 6.3 Hz, $(C\mathit{H}_3)_2CHO$), 1.21 (d, ^3J = 6.3 Hz, $(C\mathit{H}_3)_2CHO$). ^{13}C NMR (C_6D_6): δ 133.5, 129.3 (C_5Me_4H), 71.9 ($(CH_3)_2\mathit{C}HO$), 66.0 (C_5Me_4H), 24.3, 24.4 ($(\mathit{C}H_3)_2CHO$), 14.4, 11.3 ($C_5(\mathit{C}H_3)_4H$). ^{27}Al NMR ($CDCl_3$): δ 85. IR (KBr, Nujol): υ[cm^{-1}] = 2731 (w), 2609 (w), 2362 (w), 1940 (w), 1798 (w), 1605 (w), 1230 (sh), 1172 (sh), 1145 (sh), 1110 (s), 938 (s), 834 (s), 722 (s), 689 (s), 658 (s), 551 (s), 519 (s), 464(s). Anal. Calcd for $C_{12}H_{20}AlClO$: C, 59.38; H, 8.30. Found: C, 59.30; H, 8.04.

Crystal Structure Determination of $[(C_5Me_4H)AlCl(\mu\text{-}O\mathit{i}Pr)]_2$ (2). Crystals of of **2** were grown by cooling a saturated toluene solution of the compound at -60°C. A single, colorless crystal with dimensions 0.15 x 0.30 x 0.40 mm was mounted in a thin-walled glass capillary under a nitrogen atmosphere. Crystal data collection and refinement parameters are given in Table I. The unit-cell parameters were obtained by the least-squares refinement of the angular settings of 24 reflections ($20° < 2\theta < 24°$).

The systematic absences in the diffraction data are uniquely consistent for $P2_1/n$. The structure was solved using direct methods, completed by subsequent difference Fourier syntheses and refined by full-matrix least squares procedures. Absorption corrections were ignored (μ = 1.54 cm^{-1}). The molecule is centered on an inversion point. All non-hydrogen atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were treated as idealized contributions.

All software and sources of the scattering factors are contained in either the SHELXTL (5.1) or the SHELXTL PLUS (4.2) program libraries (G. Sheldrick, Siemens XRD, Madison, WI).

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