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Reactions of Diethylaminodimethylaluminum Dimer with the Tripodal Ligands $CH_3C(CH_2NH_2)_2(CH_2OH)$ and $CH_3C(CH_2NH_2)(CH_2OH)(CH_2SPh)$: Crystal Structure of $[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2$

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Summary

Reactions of diethylaminodimethylaluminum dimer, $[Me_2Al(\mu\text{-}NEt_2)]_2$, with the tripodal ligands 2,2-bis(aminomethyl)-1-propanol, $CH_3C(CH_2NH_2)_2(CH_2OH)$, and 2-aminomethyl-2-(thiophenylmethyl)-1-propanol, $CH_3C(CH_2NH_2)(CH_2OH)(CH_2SPh)$, have yielded a tetranuclear organoaluminum compound $\{[AlMe_2]_3[CH_3C(CH_2NH)_2(CH_2O)]_2Al\}$ (**1**) and a trinuclear compound $(AlMe_2)_2AlMe[CH_3C(CH_2NH)(CH_2O)(CH_2SPh)]$ (**2**), respectively. Both compounds were characterized by ^1H and ^{13}C NMR data, IR, and mass and elemental analysis. Compound **1** was also characterized by single-crystal X-ray diffraction analysis. Compound **1** crystallized in a tetragonal space group $P4_12_12$ (No. 92), with cell parameters $a = 10.238(2)$ Å, $c = 24.470(2)$ Å, $V = 2565(1)$ Å^3, $Z = 4$, and $R = 0.060$. It appears that the central aluminum atom is surrounded by two tripodal ligands in an octahedral geometry, whereas the other three aluminum atoms act to bridge the two ligands. Compound **2** has a pentacoordinated aluminum center surrounded by two tripodal ligands and one methyl group. The sulfur atoms on the tripodal ligand were not coordinated to the central aluminum atom.

Introduction

Organoaluminum compounds have received much attention due to their importance in synthetic applications.[1−4] In order to better understand the role of such compounds in reactions, aluminum complexes with various coordination modes have been prepared. Among these complexes, coordination of polydentates toward an aluminum center is an interesting topic which provides much information about the properties of organoaluminum compounds. Two classes of polydentates commonly seen in this type are macrocyclic crown ethers and tripodal ligands.[5−14] A number of coordination compounds of aluminum obtained from oxygen-, nitrogen-, and sulfur-containing crown ethers have been the subject of numerous studies.[15−19] Another class of compounds containing Al−N bonds is the azatranes.[20] It has been shown that AlR_3 forms stable complexes with crown ethers, which can serve as precursors for liquid clathrates. Other donors such as nitrogen and sulfur ligands are also studied. But the reactivities of tripodal ligands toward aluminum are studied much less.[21] In a previous finding, we reported on the synthesis and structural characterization of a novel, highly symmetrical aluminum complex with the tripodal ligand $CH_3C(CH_2NCH_2)_3$.[22] To extend this study, reactions of $[Me_2Al(\mu\text{-}NEt_2)]_2$ with mixed donor tripodal ligands, 2,2-bis(aminomethyl)-1-propanol and 2-aminomethyl-2-(thiophenylmethyl)-1-propanol, to yield tetranuclear and trinuclear complexes are reported herein.

Experimental Section

Apparatus and Materials. All manipulations were carried out in a N_2-flushed glovebag, drybox, or vacuum system. Solvents were degassed prior to use. All ^1H, ^{13}C, and ^{27}Al NMR spectra were measured on a Varian VXR-300 spectrometer. Chemical shifts are referenced relative to either TMS (^1H) or benzene-d_6 (^1H, δ 7.15; $^{13}C\{^1H\}$, δ 128.00), wile ^{27}Al NMR spectra were referenced relative to $Al(H_2O)_6{}^{3+}$. Mass spectral data were obtained on a VG-7025 GC/MS/MS spectrometer. IR spectra were recorded as Nujol mulls between KBr disks on a Bio-Rad FTS-40 FT-IR spectrometer.

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Synthesis of $\{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2\}$ (1). To a solution of $[Me_2Al(\mu\text{-}NEt_2)]_2$[23] (0.46 g, 3.88 mmol) in dichloromethane (30 mL) was added the ligand 2,2-bis(aminomethyl)-1-propanol (1.00 g, 3.88 mmol) in dichloromethane (20 mL) at 0 °C. A white precipitate was obtained with evolution of gas. The precipitate was collected after centrifugation and washed with dichloromethane. Sublimation at 150 °C produced colorless crystals of compound **1** in 72% yield. Mp: 302 °C. ^1H NMR (C_6D_6): δ −0.46 (s, 18, $Al(C\mathit{H}_3)_2$), 0.01 (s, 4H, $C\mathit{H}_3C(CH_2NH)_2(CH_2O)$), 0.54 (br, 4H, $CH_3C(CH_2N\mathit{H})_2(CH_2O)$), 2.11 (br, 4H, $CH_3C(C\mathit{H}_2NH)_2(CH_2O)$), 2.61 (ddd, 4H, $CH_3C(C\mathit{H}_2NH)_2(CH_2O)$), 3.19 (s, 4H, $CH_3C(CH_2NH)_2(C\mathit{H}_2O)$), ^{13}C NMR (C_6D_6): δ −9.73 ($Al(\mathit{C}H_3)_2$), 22.49 ($\mathit{C}H_3C(CH_2NH)_2(CH_2O)$), 37.38 ($CH_3\mathit{C}(CH_2NH)_2(CH_2O)$), 52.63 ($CH_3C(C(\mathit{C}H_2NH)_2(CH_2O)$), 71.34 ($CH_3C(CH_2NH)_2(\mathit{C}H_2O)$). ^{27}Al NMR (C_6D_6): δ 152 (br), 11. Analysis by mass spectroscopy gave the fragments expressed as *m/z* (EI, 70 eV; relative intensity (%) and assignment in parentheses): 412 (100, $[M - Me - H]^+$), 57 (77, $[AlMe_2]^+$, 115 (42, $[CH_3C(CH_2NH)_2(CH_2O)]^+$), 116 (41, $[HCH_3C(CH_2NH)_2(CH_2OH)]^+$), 311 (38, $[M - (AlMe_2)_2 + 3H]^+$), 43 (27, $[HAlMe]^+$), 398 (22, [M − 2Me]), 258 (11, $\{HAlCH_3C(CH_2NH)_2(CH_2O)\}^+$), 384 (10, $[M - 3Me + H]^+$), 172 (8, $[AlMe_2\{CH_3C(CH_2NH)_2(CH_2O)\}]^+$). IR (Nujol cm^{-1}): 3305 (w), 3290 (w), 2987 (s), 2960 (s), 2910 (s), 2840 (s), 1477 (s), 1380 (s), 1311 (s), 1205 (s), 1197 (m), 1160 (m), 1087 (m), 1072 (w), 1055 (br), 687 (m). Anal. Found: C, 43.7; H, 9.6; N, 12.9. Calcd: C, 44.9; H, 9.4; N, 13.1.

Synthesis of $(AlMe_2)_2AlMe[CH_3C(CH_2NH)(CH_2O)(CH_2SPh)\}_2$ (2). To a solution of $[Me_2Al(\mu\text{-}NEt_2)]_2$ (0.41 g, 1.94 mmol) in dichloromethane (20 mL) was added the ligand 2-aminomethyl-2-(thiophenylmethyl)-1-propanol (0.50 g, 1.94 mmol) in dichloromethane (20 mL) at 0 °C under nitrogen atmosphere. A white precipitate was obtained with evolution of gas. The precipitate was collected after centrifugation and was washed with dichloromethane. The crude product was recrystallized with benzene. Yield: 75%. Mp: 142 °C (dec). ^1H NMR (C_6D_6): δ −0.64, −0.645, −0.66 (s, 15H, $Al(C\mathit{H}_3)_2$), 0.59, 0.63 (s, 2 sets, 3H, $C\mathit{H}_3C(CH_2NH)(CH_2O)(CH_2SPh)$), 1.26 (s, br, 2H, $CH_3C(CH_2N\mathit{H})(CH_2O)(CH_2SPh)$), 2.20, 2.39 (m, 4H, $CH_3C(CH_2NH)(CH_2O)(C\mathit{H}_2SPh)$), 2.67, 3.24 (m, 4H, $CH_3C(C\mathit{H}_2NH)(CH_2O)(CH_2SPh)$), 3.54 (s, 4H, $CH_3C(CH_2NH)(C\mathit{H}_2O)(CH_2SPh)$), 6.90, 7.01, 7.32 (m, 10H, Ph). ^{13}C NMR (C_6D_6): δ −7.93 ($Al(C\mathit{H}_3)_2$), 20.64, 20.85 ($\mathit{C}H_3C(CH_2NH)(CH_2O)(CH_2SPh)$), 30.15 ($CH_3\mathit{C}(CH_2NH)(CH_2O)(CH_2SPh)$), 38.23, 38.36 ($CH_3C(CH_2NH)(CH_2O)(\mathit{C}H_2SPh)$), 48.14, 48.29 ($CH_3C(\mathit{C}H_2NH)(CH_2O)(CH_2SPh)$), 71.75 ($CH_3C(CH_2NH)(\mathit{C}H_2O)(CH_2SPh)$), 126.34, 126.39, 127.15, 127.27, 129.10, 129.31 (C_6H_5). ^{27}Al NMR (C_6D_6): δ 152, 58. Analysis by mass spectroscopy gave the fragments expressed as *m/z* (EI, 70 eV; relative intensity (%) and assignment in parentheses): 252 (100, $[MeAl(CH_3C(CH_2NH_2)(CH_2O)(CH_2SPh))]^+$), 159 (49, $[M - Me]^+$), 110 (28, $[PhSH]^+$), 128 (26, $[Al(CH_3C(CH_2NH_2)(CH_2O)(CH_2))]^+$), 57 (26, $[AlMe_2]^+$), 157 (17, $[AlMe_2(CH_3C(CH_2NH)(CH_2O)(CH_2))]^+$), 69 (14, $[MeAlNH_2]^+$), 43 (12, $[HAlMe]^+$), 280 (9, $[MeAl(CH_3C(CH_2NH)(CH_2O)(CH_2SPh)AlH]^+$). IR (Nujol, cm^{-1}): 3305 (w), 33 218 (w) 2913 (s), 2852 (s), 2721 (s), 1586 (m), 1464 (s), 1378 (s), 1269 (s), 1096 (m), 1044 (s), 967 (m), 811 (m), 793 (m), 681 (m), 473 (m). Anal. Found: C, 57.3; H, 8.2; N, 5.4; S, 11.7. Calcd: C, 56.5; H, 7.8; N, 4.9; S, 11.5.

Crystallographic Measurements. A suitable single crystal of the title compound was sealed in a glass capillary for X-ray structure analysis. Preliminary examination and intensity data collections were carried out with a Rigaku AFC6S diffractometer using graphite-monochromatized Cu Kα (λ = 1.541 78 Å) radiation. Intensity data were collected using the $\theta - 2\theta$ scan mode and corrected for absorption and decay. The structure was solved by SIR92[24] and refined with full matrix least squares on *F*. In the final cycles, all nonhydrogen atoms were refined anisotropically and all hydrogen atoms were fixed at idealized positions. All calculations were carried out with an SGI R4000 computer using the teXsan program package.[25] A summary of the data collection and structure solution is given in Table 1.

Table 1. Crystal and Intensity Collection Data for the Compound $\{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2\}$ (**1**)

formula	$C_{16}H_{40}N_4O_2Al_4$
fw	428.44
diffractometer	Rigaku AFC6S
cryst syst	tetragonal
space group	$P4_12_12$ (No. 92)
a, (Å)	10.238(2)
c, (Å)	24.470(2)
V, (Å^3)	2565(1)
Z	4
D_{calc} (g/cm^3)	1.109
λ(Cu Kα) (Å)	1.541 78
F(000)	928
μ (cm^{-1})	18.17
2θ range (deg)	3−120
cryst dimens (mm)	0.21 × 0.30 × 0.64
no. of measd rflns	2769
no. of unique rflns	1976
no. of obs. rflns	1614
no. of variables	119
R_F, R_W	0.055, 0.064
data collection temp (°C)	23
weighting scheme	$1/[\sigma^2(F_o)^2]$
T (min, max)	(0.96, 1.00)
GOF	1.73
computation program	teXsan package

Results and Discussion

Synthesis and Characterization. Reaction of $[Me_2Al(\mu\text{-}NEt_2)]_2$ with the tripodal 2,2-bis(aminomethyl)-1-propanol in an equimolar ratio yielded a tetranuclear organoaluminum compound, $\{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2\}$, as shown in eq 1. The compound was fully characterized by ^1H, ^{13}C,

$$2[Me_2Al(\mu\text{-}NEt_2)]_2 + 2CH_3C(CH_2NH_2)_2(CH_2OH) \rightarrow \{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2)]_2\} + 4HNEt_2 + 2MeH \quad (1)$$

and ^{27}Al NMR spectral data, IR, and mass spectral data. The ^1H NMR spectrum showed a singlet at δ 3.19 ppm corresponding to the hydroxymethyl hydrogens and two peaks (a doublet of a doublet of doublets and a multiplet) at δ 2.61 and 2.11 ppm corresponding to the methyl hydrogens of the aminomethyl group. In the IR spectrum, the peaks at 3305 and 3290 cm^{-1} are assigned for the stretching frequencies of N−H.

However, reaction of $[Me_2Al(\mu\text{-}NEt_2)]_2$ with the tripodal 2-aminomethyl-2-(thiophenylmethyl)-1-propanol in equimolar ratio yielded $(AlMe_2)_2AlMe[CH_3C(CH_2NH)(CH_2O)(CH_2SPh)]_2$, as shown in eq 2. The ^1H NMR spectrum showed a singlet for

$$[Me_2Al(\mu\text{-}NEt_2)]_2 + 2CH_3C(CH_2NH_2)_2(CH_2OH) \rightarrow 2(AlMe_2)_2AlMe[CH_3C(CH_2NH)(CH_2O)(CH_2SPh)]_2 + 6HNEt_2 + 2MeH \quad (2)$$

the methyl hydrogen of the aminomethyl group at δ = 3.54 ppm and the methyl hydrogen of the thiophenyl group as multiplets at δ 2.02 and 2.39 ppm. The ^{27}Al NMR spectrum indicates that the compound has two different coordination environments for the aluminum atoms. The signals at δ = 58 (br) and δ = 158 (br) ppm have been assigned for five- and four-coordinate, respectively, for the aluminum centers. The mass spectral data showed the fragment ion peak *m/e* at 559 ($[M - Me]^+$). These assignments are in good agreement with the elemental analysis values. In the IR spectrum, the peaks at 3305 and 3218 cm^{-1} and 681 cm^{-1} are assigned for the stretching frequencies of N−H and N−Al. A proposed structure for compound **2** is given in Figure 1.

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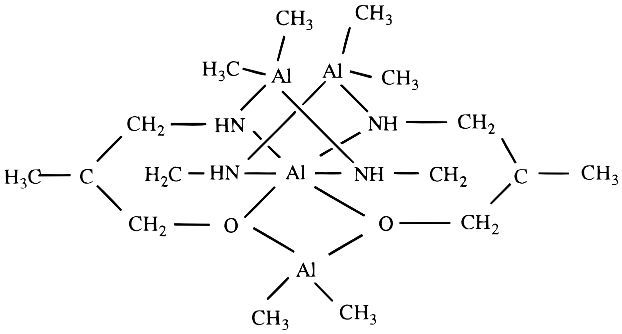

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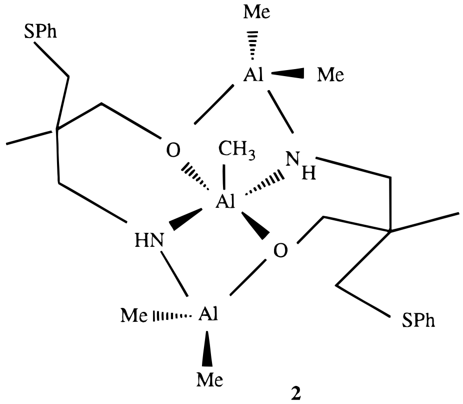

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Figure 1. A schematic sketch of compounds **1** and **2**. We assume that the sulfur-containing ligand is softer, and hence, a tetranuclear compound could not be obtained with the Al−S bonding.

The tripodal ligand $CH_3C(CH_2NH_2)_2(CH_2OH)$ contains two nitrogen atoms, one oxygen atom and three acidic hydrogen atoms. When treated with the aluminum dimer, the nitrogen and oxygen atoms of the ligands are coordinated to the aluminum atoms with the elimination of methane and amine to give compound **1**. Similarly, the ligand $CH_3C(CH_2NH_2)(CH_2OH)(CH_2SPh)$ contains a sulfur atom in addition to oxygen and nitrogen atoms. This ligand has yielded a trinuclear compound **2**.

Description of Structure. Compound **1** contains a crystallographic two-fold axis connecting the Al1 and Al3 atoms as shown in Figure 2. Selected bond distances and bond angles are given in Table 2. The structure consists of two types of aluminum centers. Al1 is in the octahedral environment and is chelated by two deprotonated 2,2-bis(aminomethyl)-1-propanol ligands. The other three aluminum atoms are in a tetrahedral environment and are coordinated by two methyl ligands and two nitrogen or oxygen atoms of the tripodal ligand. Attached to the aluminum atom are three coplanar four-membered rings, $Al_2N_2AlN_1$, $Al_2{}^*N_2{}^*Al_1N_1{}^*$, and $Al_1O_1Al_3O_1{}^*$. The Al−N and Al−O bond distances associated with the octahedral aluminum center are equivalent in length, and the associated bond angles are close to 90° and 180°, giving rise to a nearly perfect octahedral geometry. The Al−N and Al−O bond distances associated with the central aluminum are slightly longer than those of the peripheral aluminum atoms, and the difference may arise from the difference in the coordination number of the aluminum atoms.

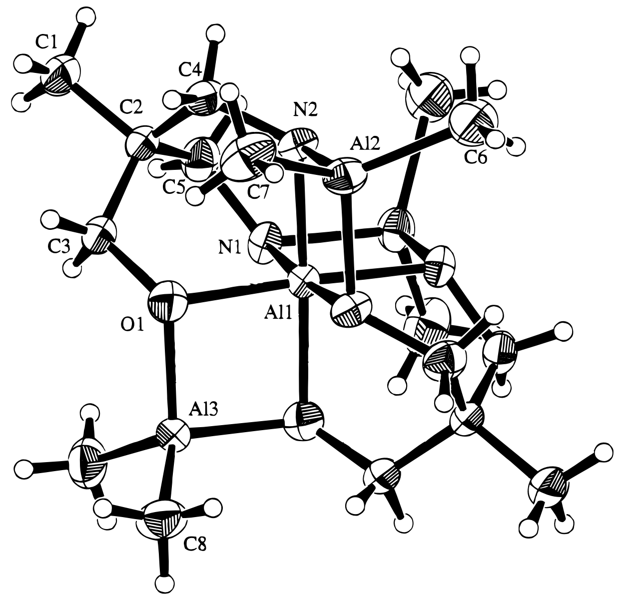

Figure 2. An ORTEP view of the compound $\{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2\}$ (**1**). Thermal ellipsoids are drawn at the 50% probability level.

Table 2. Selected Bond Lengths (Å) and Bond Angles (deg) for the Compound $\{[AlMe_2]_3Al[CH_3C(CH_2NH)_2(CH_2O)]_2\}$ (**1**)

Al(1)−O(1)	2.019(4)	Al(3)−O(1)	1.936(4)
Al(1)−N(1)	2.021(4)	Al(3)−C(8)	1.973(6)
Al(1)−N(2)	2.014(4)	O(1)−C(3)	1.504(6)
Al(2)−N(1)	1.932(4)	N(1)−C(5)	1.492(6)
Al(2)−N(2)	1.944(4)	N(2)−C(4)	1.483(6)
Al(2)−C(6)	1.970(7)	C(1)−C(2)	1.533(7)
Al(2)−C(7)	1.972(6)		

O(1)−Al(1)−O(1)	85.4(2)	N(2)−Al(2)−C(7)	112.7(2)
O(1)−Al(1)−N(1)	92.1(2)	C(6)−Al(2)−C(7)	118.1(3)
O(1)−Al(1)−N(2)	91.2(1)	O(1)−Al(3)−O(1)*	90.0(2)
O(1)−Al(1)−N(2)*	175.6(2)	O(1)−Al(3)−C(8)	111.3(2)
N(1)−Al(1)−N(1)*	175.0(3)	Al(1)−O(1)−Al(3)	92.3(1)
N(1)−Al(1)−N(2)	90.8(2)	Al(1)−O(1)−C(3)	113.6(3)
N(2)−Al(1)−N(2)*	92.3(2)	Al(3)−O(1)−C(3)	120.7(3)
N(1)−Al(2)−N(2)	90.2(2)	Al(1)−N(1)−Al(2)	92.1(2)
N(1)−Al(2)−C(6)	111.7(2)	Al(1)−N(1)−C(5)	120.0(3)
N(1)−Al(2)−C(7)	110.4(3)	Al(1)−N(2)−Al(2)	91.9(2)
N(2)−Al(2)−C(6)	110.4(3)	Al(1)−N(2)−C(4)	115.4(2)

Acknowledgment. We thank the National Science Council of the Republic of China for financial support.

Supporting Information Available: Tables of crystal data, complete lists of bond distances and bond angles, final fractional coordinates, and thermal parameters (12 pages). Ordering information is given on any current masthead page.

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