connected to four different Na^+ cations via isocarbonyl linkages. Na^+ is in a tetragonally distorted octahedral environment, with four pyridine ligands arranged in a propeller fashion in the equatorial plane and two oxygen atoms (one from an axial carbonyl C1O1 of a $[Fe_2(CO)_8]^{2-}$ unit, the other from an equatorial carbonyl C2O2 of a different $[Fe_2(CO)_8]^{2-}$ unit) in the axial positions.

Discussion

Structure. In general, coordination of the oxygen atoms of metal carbonyls to 2+ and 3+ cations causes a decrease in the M–C bond distance and an increase in the C–O bond distance.[2,3e,j,8,9] In the title compound, however, the structure of the dianion does not seem to be perturbed by the Na–isocarbonyl interactions. For example, all the equatorial Fe–C and C–O bond distances are the same within experimental error, irrespective of whether they participate in isocarbonyl linkages. The geometry of $[Fe_2(CO)_8]^{2-}$ is only slightly distorted from ideal D_{3d} symmetry, thereby implying a weak ion-pairing effect. The structural parameters of $[Fe_2(CO)_8]^{2-}$ in the title compound are very similar to those of previously determined $[Fe_2(CO)_8]^{2-}$ structures.[5] The Fe–Fe bond distance of 2.815 (1) Å in the title compound is not significantly longer than the values in $[PPN]_2[Fe_2(CO)_8]$ (2.787 (2) Å)[5a] and in $[PPh_4]_2[Fe_2(CO)_8]$ (2.792 (1) Å).[5b]

The coordination of pyridines in a "propeller assemblage" around Na^+ in the title compound resembles that observed in $(C_5H_5N)_4Yb(BH_4)_2$[10] and in $[(C_5H_5N)_4Mg][Mo(CO)_3Cp]_2$.[9] The arrangement of five pyridine ligands in an equatorial plane of pentagonal-bipyramidal geometry is also observed for Yb^{2+} in $[Yb(C_5H_5N)_5(CH_3CN)_2][Hg\{Fe(CO)_4\}_2]$.[11] The Na–N distances in the title compound have an average of 2.464 Å. The Na–N distances of 2.463 (5)[12d] and 2.471 (7) Å[12a] for six-coordinate Na^+ cations are comparable to the value reported here. The Na–N distances for four- and five-coordinate Na^+ cations are 2.391 (5)[12b] and 2.384 (6) Å,[12c] respectively.

Two Na–O distances of 2.587 (3) and 2.585 (3) Å in the title compound are quite long compared to other Na–O distances observed in the interactions between Na^+ and isocarbonyls, which range from 2.318 to 2.555 Å.[4a,13] The Na–O1–C1 and the Na–O2–C2 angles are 148.4 (3) and 177.6 (3)°, respectively. Na–O–C angles involving terminal carbonyls in the other reported structures range from 115.1 to 162.4°.[13] On the basis of the large range of Na–O–C angles observed, we believe that the M–O–C angles are not critical for effective cation–isocarbonyl interactions and that steric effects in the solid state control the angularity of these interactions.

Infrared and NMR Spectra. The infrared spectrum of $\{[(C_5H_5N)_4Na]_2[Fe_2(CO)_8]\}_\infty$ in Nujol mull shows three infrared absorptions at 1927 (m), 1855 (m, sh), and 1830 (s) cm^{-1}. On the other hand, only two major carbonyl absorptions are observed for $[PPh_4]_2[Fe_2(CO)_8]$ both in Nujol mull (1909 (m), 1847 (s, br) cm^{-1}) and in CH_3CN solution (1914 (m), 1865 (s) cm^{-1}). The absorption at 1830 cm^{-1} in the infrared spectrum of the title compound is believed to be due to the interactions between the Na^+ cations and the oxygen atoms of the carbonyl groups in $[Fe_2(CO)_8]^{2-}$.

The infrared spectrum of $\{[(C_5H_5N)_4Na]_2[Fe_2(CO)_8]\}_\infty$ in THF also suggests the presence of ion pairing (three major absorptions are at 1922 (m), 1875 (s), and 1829 (m) cm^{-1}). However, the ^{13}C NMR chemical shift of the title compound in THF-d_8, a singlet at δ 227.84 ppm for the carbonyls, fails to distinguish between unbound carbonyl groups and Na^+-bound carbonyl groups at room temperature. The singlet remains at −80 °C. In CH_3CN and in pyridine, two major infrared absorptions at 1914 (m) and 1863 (s) cm^{-1} are at the same positions as the absorptions from $[PPh_4]_2[Fe_2(CO)_8]$, despite a noticeable shoulder at 1840 (w) cm^{-1}. These results indicate that the ion pairing in CH_3CN and in pyridine solutions is significantly reduced, if not totally absent. Other IR studies of $Na_2[Fe_2(CO)_8]$ reveal three carbonyl bands at 1910 (m), 1860 (s) and 1835 (w) cm^{-1} in DMF[14] and at 1914 (sh), 1868 (s) and 1821 (m) cm^{-1} in THF.[15] These results suggest that contact ion pairing between Na^+ and $[Fe_2(CO)_8]^{2-}$ is also operative in DMF and THF.

Acknowledgment. This work was supported by the National Science Foundation through Grant CHE91-04035. NMR spectra were obtained at The Ohio State University Campus Chemical Instrument Center funded in part by NSF Grant 79-10019 and NIH Grant 1 S10 PR0140518-01A.

Supplementary Material Available: Listings of positional parameters, calculated hydrogen atom positional parameters, anisotropic thermal parameters, bond distances, and bond angles and a unit cell packing diagram (9 pages); a listing of structure factor amplitudes (24 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry,
National Sun Yat-Sen University, Kaohsiung, Taiwan, ROC

Syntheses and Molecular Structures of an Unusual Cyclic Tetrameric Organometallic Complex, $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$, Containing Tricoordinate Magnesiums, and a Linear Dimer, $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$, Containing Tetracoordinate Magnesiums

Tzong-Yih Her, Chung-Cheng Chang,* and Ling-Kang Liu†

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Introduction

The reaction of trimethylaluminum with dialkylmagnesium is perhaps one of the most celebrated reactions in the aluminum magnesium mixed-metal chemistry. Investigations mostly concentrated on dialuminum magnesium complexes, e.g., $Mg[Al(OCH_3)_2(CH_3)_2]_2$ and $Mg[Al(CH_3)_4]_2$.[1,2] Monoaluminum magnesium complexes were synthesized in different laboratories;[1–6] however, the first example of an X-ray crystal structure of $[Me_2Si(N(t\text{-}Bu))_2(AlMe_2)(MgI)]_2$ was reported recently.[5] Herein, we report the syntheses of $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$ (**1**) and $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$ (**2**) from the reactions of trimethylaluminum ($Al(CH_3)_3$) with bis(diethylamino)magnesium ($Mg[N(C_2H_5)_2]_2$) and bis(diisopropylamino)magnesium ($Mg[N(i\text{-}C_3H_7)_2]_2$), respectively.[7] Surprisingly, four molecules of $(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)$ (**2a**) associate weakly as a tetramer (**2**) in the solid state through the interactions of tricoordinate magnesiums with carbons or/and

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† Department of Chemistry, Academia Sinica, Taipei, Taiwan, ROC.

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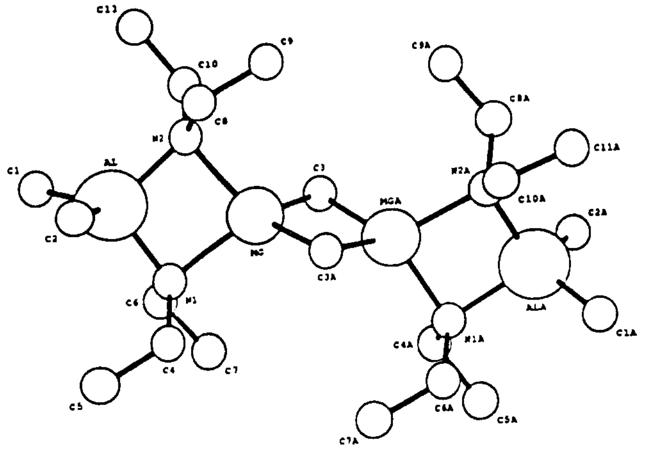

Figure 1. Molecular structure of $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$.

hydrogens, yielding the first example of the Mg$\cdots$CH$_3$–Mg interactions such as in $(LiR)_n$;[8] on the other hand, two molecules of $(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)$ (**1a**) associate strongly as a dimer (**1**) through the interactions of four-coordinate magnesiums with carbons.

Experimental Section

All operations were carried out in a N_2-flushed glovebag, drybox, or vacuum system. All solvents were distilled prior to use. Infrared spectra (Nujol mull) were recorded on a Perkin-Elmer IR spectrophotometer, mass spectra on VG 70-250S GC/MS spectrophotometer, and NMR spectra on a Varian VXR-300MHz FT-NMR spectrometer; TMS was used as internal standard. Elemental analyses were carried out at Galbraith Laboratory.

Syntheses. **$\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$ (1).** A toluene solution of 17 mL of 2 M $Al(CH_3)_3$ was added dropwise to a stirred solution of 5.63 g (0.0335 mol) of $Mg[N(C_2H_5)_2]_2$ in 100 mL of diethyl ether at room temperature under nitrogen. After 12 h a pale yellow solution was obtained, which, after removal of diethyl ether and toluene under vacuum, gave a pale yellow viscous fluid. Sublimation at 100 °C produced colorless crystals, $[Me_2Al(\mu\text{-}NEt_2)]_2$, mp 41–43 °C; yield, 20%. Continuous sublimation at 120 °C produced more colorless crystals, $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$ (**1**), mp$_{dec}$ > 79 °C; yield, 60%. Anal. Calc: C, 55.13; N, 11.69; H, 11.78. Found: C, 53.06; N, 11.15; H, 11.49.

$\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$ (2). A toluene solution of 17 mL of 2 M $Al(CH_3)_3$ was added dropwise to a stirred solution of 7.5 g (0.0335 mol) of $Mg[N(i\text{-}C_3H_7)_2]_2$ in 100 mL of diethyl ether at room temperature under nitrogen. After 12 h a pale yellow solution was obtained, which, after removal of diethyl ether and toluene under vacuum, gave a pale yellow solid. This crude product was crystallized from hexane. $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$ (**2**) was obtained in quantitative yield, and then on sublimation at 60 °C, it produced colorless crystals. Mp$_{dec}$ > 55 °C. Anal. Calc: C, 60.71; N, 9.44; H, 12.57. Found: C, 57.43; N, 9.28; H, 11.65. On sublimation, we obtained a small quantity of $\{Me_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]\}_2$ (<5%), mp 125–128 °C.

Structure Determination. Crystals for X-ray measurements were sealed in glass capillaries. Preliminary examination and intensity data collection were carried out with an Enraf-Nonius CAD-4 automatic diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Intensity data were collected by using the θ–2θ scan mode for $2\theta \leq 45°$ and corrected for absorption and decay. Both structures were solved by MULTAN and refined in full-matrix least-squares analyses on F with $w = 1.0/[\sigma^2(F_o) + 0.0001F_o^2]$. In the final cycles all non-hydrogen atoms were refined anisotropically and all hydrogen atoms were fixed at positions (d(C–H) = 1.00 Å) as calculated at the anisotropic convergence stage. Scattering factors for the neutral atoms and anomalous scattering coefficients for non-hydrogen atoms were taken from ref 9. All calculations were carried out with a Micro VAX 3600 computer using the NRC VAX program package.[10] A summary of data collection and structure solution is given in Table I, selected bond distances and

Table I. Crystallographic Data and Refinement Results

	1	2
formula	$C_{22}H_{58}N_4Al_2Mg_2$	$C_{60}H_{148}Al_4Mg_4N_8$
cryst sys	monoclinic	monoclinic
space group	$C2/c$	$C2/c$
a, Å	25.830 (6)	26.922 (3)
b, Å	10.141 (5)	10.775 (3)
c, Å	15.079 (3)	27.281 (6)
β, deg	123.95 (2)	94.02 (1)
cell vol, Å^3	3207 (2)	7894 (3)
mol wt	481.36	1187.04
Z	4	8
F(000)	1071.67	2655.29
D_{calc}, g/cm^3	0.997	0.999
μ, mm^{-1}	0.14	0.12
λ, Å	0.709 30	0.709 30
2θ(max)	44.8	45.0
cryst dimens, mm	0.45 × 0.31 × 0.38	0.75 × 0.50 × 0.47
diffractometer	Nonius CAD-4	Nonius CAD-4
mode	$\theta/2\theta$	$\theta/2\theta$
no. of measd reflns	1728	5302
no. of unique reflns	1672	5172
no. of observns		
$[I > 2.5\sigma(I)]$	1116	
$[I > 20\sigma(I)]$		3139
	Last Least-Squares Cycles	
no. of atoms	44	112
no. of params	136	344
weight	$1.0/[\sigma^2(F_o) + 0.0001F_o^2]$	$1.0/[\sigma^2(F_o) + 0.0001F_o^2]$
R	0.049	0.072
R_w	0.062	0.084
highest peak in diff map, e/Å^3	0.250	0.430

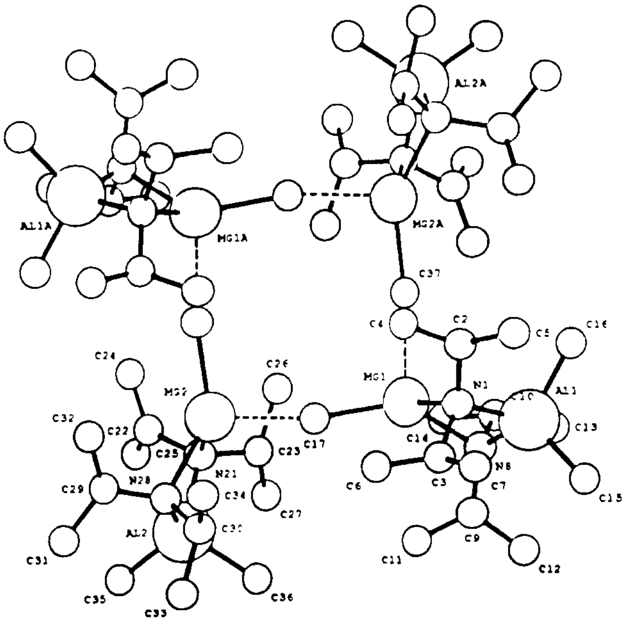

Figure 2. Molecular structure of $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$.

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Table II. Selected Bond Distances (Å) and Angles (deg)

$\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$			
Al–N(1)	1.953 (4)	N(1)–C(4)	1.481 (6)
Al–N(2)	1.948 (4)	N(1)–C(6)	1.474 (6)
Al–C(1)	1.964 (6)	N(2)–C(8)	1.486 (6)
Al–C(2)	1.959 (6)	N(2)–C(10)	1.490 (6)
Mg–N(1)	2.105 (4)	C(4)–C(5)	1.525 (8)
Mg–N(2)	2.101 (4)	C(6)–C(7)	1.518 (8)
Mg–C(3)	2.296 (5)	C(8)–C(9)	1.516 (9)
Mg–C(3a)	2.244 (5)	C(10)–C(11)	1.523 (8)
Al···Mg	2.862 (2)	Mg···Mg(a)	2.735 (3)
N(1)–Al–N(2)	94.3 (2)	Mg–N(1)–C(4)	117.0 (3)
N(1)–Al–(C1)	109.8 (2)	Mg–N(1)–C(6)	109.5 (3)
N(1)–Al–C(2)	112.6 (2)	C(4)–N(1)–C(6)	110.8 (4)
N(2)–Al–C(1)	115.1 (2)	Al–N(2)–Mg	89.9 (2)
N(2)–Al–C(2)	109.3 (2)	Al–N(2)–C(8)	114.9 (3)
C(1)–Al–C(2)	114.2 (3)	Al–N(2)–C(10)	115.2 (3)
N(1)–Mg–N(2)	85.7 (2)	Mg–N(2)–C(8)	113.1 (3)
N(1)–Mg–C(3)	121.5 (2)	Mg–N(2)–C(10)	110.9 (3)
N(1)–Mg–C(3a)	111.8 (2)	C(8)–N(2)–C(10)	111.3 (4)
N(2)–Mg–C(3)	114.5 (2)	Mg–C(3)–Mg(a)	74.1 (1)
N(2)–Mg–C(3a)	117.5 (2)	N(1)–C(4)–C(5)	115.4 (4)
C(3)–Mg–C(3a)	105.6 (2)	N(1)–C(6)–C(7)	115.6 (4)
Al–N(1)–Mg	89.6 (2)	N(2)–C(8)–C(9)	114.4 (4)
Al–N(1)–C(4)	114.6 (3)	N(2)–C(10)–C(11)	115.5 (5)
Al–N(1)–C(6)	113.8 (3)		
$\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$			
Al(1)–N(1)	1.957 (6)	N(8)–C(10)	1.48 (1)
Al(1)–N(8)	1.960 (6)	C(2)–C(4)	1.51 (1)
Al(1)–C(15)	1.968 (8)	C(2)–C(5)	1.54 (1)
Al(1)–C(16)	1.983 (8)	C(3)–C(6)	1.51 (1)
Mg(1)–N(1)	2.142 (6)	C(3)–C(7)	1.52 (1)
Mg(1)–N(8)	2.150 (5)	C(10)–C(13)	1.52 (1)
Mg(1)–C(17)	2.151 (6)	C(10)–C(14)	1.51 (2)
N(1)–C(2)	1.493 (9)	Al(1)···Mg(1)	2.832 (3)
N(1)–C(3)	1.494 (9)	Mg(2)···C(17)	2.493 (6)
N(8)–C(9)	1.48 (1)		
N(1)–Al(1)–N(8)	95.2 (2)	Mg(1)–N(8)–C(9)	116.3 (5)
N(1)–Al(1)–C(15)	114.1 (3)	Al(1)–N(8)–C(10)	114.5 (5)
N(1)–Al(1)–C(16)	109.8 (3)	Mg(1)–N(8)–C(9)	116.3 (5)
N(8)–Al(1)–C(15)	116.0 (3)	Mg(1)–N(8)–C(10)	106.4 (4)
N(8)–Al(1)–C(16)	109.5 (3)	C(9)–N(8)–C(10)	111.4 (7)
C(15)–Al(1)–C(16)	111.2 (3)	N(1)–C(2)–C(4)	116.0 (6)
N(1)–Mg(1)–N(8)	84.7 (2)	N(1)–C(2)–C(5)	117.7 (6)
N(1)–Mg(1)–C(17)	133.4 (3)	C(4)–C(2)–C(5)	108.6 (7)
N(8)–Mg(1)–C(17)	126.5 (3)	N(1)–C(3)–C(6)	116.2 (6)
Al(1)–N(1)–Mg(1)	87.3 (2)	N(1)–C(3)–C(7)	116.7 (7)
Al(1)–N(1)–C(2)	114.4 (4)	C(6)–C(3)–C(7)	111.1 (7)
Al(1)–N(1)–C(3)	112.3 (4)	Mg(1)–C(17)–Mg(2)	167.9 (3)
Mg(1)–N(1)–C(2)	112.6 (4)	N(8)–C(9)–C(11)	120.2 (7)
Mg(1)–N(1)–C(3)	109.5 (4)	N(8)–C(9)–C(12)	121.3 (8)
C(2)–N(1)–C(3)	117.0 (5)	C(11)–C(9)–C(12)	117.9 (8)
Al(1)–N(8)–Mg(1)	87.0 (2)	N(8)–C(10)–C(13)	118.5 (7)
Al(1)–N(8)–C(9)	118.6 (6)	N(8)–C(10)–C(14)	114.8 (7)
Al(1)–N(8)–C(10)	114.5 (5)	C(13)–C(10)–C(14)	107.7 (8)

angles are detailed in Table II, and drawings of the molecules are presented in Figures 1 and 2.

Results and Discussion

The reaction of trimethylaluminum and bis(diethylamino)magnesium in a 1:1 molar ratio gives two colorless crystals: one is $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]\}_2$, and the other is $\{(CH_3)_2Al[\mu\text{-}N(C_2H_5)_2]_2Mg(CH_3)\}_2$ (**1**). The reaction of trimethylaluminum and bis(diisopropylamino)magnesium in a 1:1 molar ratio yields a small amount of white solid, $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]\}_2$, and a colorless crystal of $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$ (**2**). The structures of compounds **1** and **2** determined by X-ray are shown in Figures 1 and 2, which are consistent with the data from ^1H, ^{13}C, and ^{27}Al NMR and mass spectra. The molecular weight of compound **2** was determined in cyclohexane to be 600 ± 30 by the freezing point depression.

Description of the Structures. Compound **1a** with crystallographic C_2 symmetry exists as a dimer (**1**), shown in Figure 1. The selected bond angles and bond lengths are shown in Table II. Al, N(1), Mg, and N(2) form a coplanar heterocyclic four-membered ring. The distances Al–N(1), Al–N(2), Mg–N(1), and Mg–N(2) are 1.953 (4), 1.948 (4), 2.105 (4), and 2.101 (4) Å, respectively. The atoms Mg, C(3), Mg(a), and C(3a) form an asymmetrical four-membered ring with distances of 2.296 (5), 2.244 (5), 2.244 (5), and 2.296 (5) Å, for Mg–C(3), Mg–C(3a), Mg(a)–C(3), and Mg(a)–C(3a), respectively. The contacts for Mg–C(3) and Mg–C(3a) falling into the covalent bond region are reasonably considered to be σ bonds.[11] The two magnesium atoms are linked by the methyl group, which is supported by the NMR-DEPT experiment. The contact of 2.735 (3) Å for Mg···Mg, which is less than the sum of van der Waals radius of two Mg atoms, is also expected to have an interaction between two magnesiums.

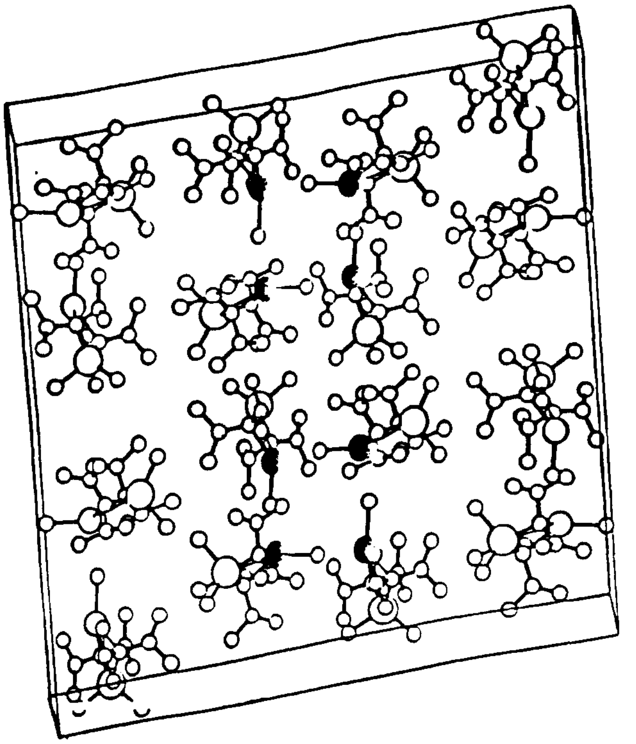

Figure 3. Packing arrangement of $\{(CH_3)_2Al[\mu\text{-}N(i\text{-}C_3H_7)_2]_2Mg(CH_3)\}_4$.

Compound **2a** with crystallographic C_2 symmetry associates weakly as a tetramer (**2**), shown in Figure 2. The packing arrangement is shown in Figure 3. The selected bond angles and bond lengths are also shown in Table II. The Mg···C contact of 2.151 (6) Å for Mg(1)–C(17) is a normal σ bond; however, the Mg···C contact of 2.493 (6) Å for Mg(2)···C(17) is quite longer than a σ bond, even longer than the contacts of Mg···C in $MgCp_2$ and $Mg(indenyl)_2$.[11] This indicates a weak interaction between Mg(2) and C(17). Also, the NMR-DEPT technique supports that it is the methyl group connected to the two magnesiums. The methyl group attached to Mg(1) is directed toward Mg(2), the methyl group attached to Mg(2) is directed toward Mg(3), and so forth. According to these linkages, the four molecules of compound **2a** are connected to form a tetramer (**2**) in cyclization. The distance between the two magnesiums is 4.665 Å. This distance is much longer than the sum of van der Waals radius of two Mg atoms. So, there is no interaction between the Mg atoms. Al(1), N(1), Mg(1), and N(8) form a noncoplanar heterocyclic four-membered ring with distances of 1.957 (6), 2.142 (6), 1.960 (6), and 2.150 (5) Å, for Al(1)–N(1), Mg(1)–N(1), Al(1)–N(8), and Mg(1)–N(8), respectively. The geometrical structures of aluminum and magnesium are distorted tetrahedron and triangle, respectively.

Spectral Data. The mass spectral data of compound **1** show the 10 highest intensity fragmental ion peaks at *m/e* 225, 96, 114,

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(11) Lindsell, W. E. In *Comprehensive Organometallic Chemistry*; Stone, F. G. A., Wilkinson, G., Abel, E. W., Eds.; Pergamon, Oxford, U.K., 1982; Vol. 1, Chapter 4, p 202 (see also references cited therein).

256, 241, 167, 58, 481 (molecular ion), 184, and 296; the ion peaks at *m/e* 481 and 241 are assigned to the molecular ion and monomer ion, respectively. The 10 highest intensity fragmental ion peaks of compound **2** appear at *m/e* 281, 124, 86, 142, 102, 57, 209, 223, 263, and 196, and the monomer ion peak appears at 296. The peaks are cut off at *m/e* 578, which is assigned to the ions eliminated from the dimer molecules. The NMR spectral data are accorded with the X-ray data (Figure 2). For compound **1**, the chemical shifts in the ^{1}H NMR spectra (in C_6D_6) of two multiplet sets at $\delta = 2.849$ (8 H) and 2.709 (8 H) ppm are due to the methylene protons of the ethyl groups under two different environments (16 H in total). The chemical shifts at $\delta = 0.897$ (t, 24 H), −0.425 (s, 12 H), and −0.935 (s, 6 H) are assigned to the methyl protons of the ethyl groups, the methyl protons attached to the aluminums, and the methyl bridged-protons between the two magnesium atoms, respectively. The chemical shifts in the ^{13}C NMR spectra (in C_6D_6) at $\delta = 39.931$, 13.276, −9.890 (broad), and −15.527 are assigned to the methylene carbons, the methyl carbons in ethyl groups, the methyl carbons attached to the aluminum atoms, and the methyl carbons attached to the magnesium atoms, respectively. For compound **2**, the chemical shifts in the ^{1}H NMR spectra (in C_6D_6) at $\delta = 3.155$ (sep, 16 H), 1.123 (d, 96 H), −0.322 (s, 24 H), and −0.736 (s, 12 H) are assigned to the methine protons, the methyl protons in the isopropyl groups, the methyl protons attached to the aluminum atoms, and the methyl protons attached to the magnesium atoms, respectively. The chemical shifts in the ^{13}C NMR spectra (C_6D_6) at $\delta = 46.591$, 25.470, 25.427, −3.435 (broad), and −11.090 are reasonably assigned to the methine carbons ($\delta = 46.591$), the two different environments of the methyl carbons in the isopropyl groups ($\delta = 25.470$, 25.427), the methyl carbons attached to the aluminum atoms ($\delta = -3.435$), and the methyl carbons attached to the magnesium atoms ($\delta = -11.090$). Infrared spectra show the following absorptions: (for compound **1**) 2980–2820 (vs), 1460 (s), 1380 (s), 1350 (m), 1318 (w), 1290 (m), 1258 (w), 1208 (s), 1190 (s), 1125 (s), 1078 (w), 1045 (m), 1005 (m), 920 (sh), 900 (m), 855 (m), 795 (m), 705–660 (w), 600–480 (w), 440 (w), 400 (w); (for compound **2**) 3010–2800 (vs), 1460 (s), 1380 (s), 1200 (s), 1170 (s), 1140 (s), 1120 (s), 1075 (w), 972 (m), 915 (m), 800 (m), 765 (w), 710–650 (m), 610 (w), 495 (w), 440 (w), 400 (w).

Both compounds are not thermally stable. They decompose to $\{(CH_3)_2Al(\mu\text{-}NR_2)\}_2$ ($R = C_2H_5$, $i\text{-}C_3H_7$) on sublimation. The products have been characterized and reported. All the spectral data are accorded with the published data.[12,13]

Acknowledgment. We thank the National Science Council for financial support.

Supplementary Material Available: Tables of crystal data, distances and angles, final fractional coordinates, torsional angles, and thermal parameters for both compounds, bonding parameters around Al and Mg for the dimer, and distances to the least-squares planes for the tetramer (20 pages); listings of observed and calculated structure factors (23 pages). Ordering information is given on any current masthead page.

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(12) Cocco, L.; Eyman, D. P. *J. Organomet. Chem.* **1979**, *179*, 1.

(13) Her, T. Y.; Chang, C. C.; Liu, L. K.; Chang, H. C.; Chen, J. H.; Tsai, J. O.; Lai, Y. Y. *Polyhedron*, submitted for publication.

Additions and Corrections

1991, Volume 30

Michael J. Chetcuti,* Phillip E. Fanwick, and John C. Gordon: Reactions of a Cobalt–Molybdenum and Related Cobalt–Tungsten Tetrahedral Clusters with Phenylacetylene: Formation of Tetrametallic Alkyne Clusters or Cluster Fragmentation?

Pages 4712 and 4713. In section 4, the species $(OC)_2Co\{\mu\text{-}\eta^3,\eta^3\text{-}C(H)C(Ph)C(H)C(Ph)C(H)C(Ph)\}W(CO)(\eta^5\text{-}C_5H_4Me)$ (Co–W) (**8**) is wrongly described as a purple complex. This species and the chromatographic band from which it is obtained are both *yellow*. Its color, yield, and spectroscopic data are correctly described in the Experimental Section.—Michael J. Chetcuti

1992, Volume 31

R. F. Gallup, C. Y. Fong,* and Susan M. Kauzlarich*: Bonding Properties of $Ca_{14}GaAs_{11}$: A Compound Containing Discrete $GaAs_4$ Tetrahedra and a Hypervalent As_3 Polyatomic Unit.

Page 118. Figure 5 shows a schematic of the MO's for a hypervalent three-center, four-electron bond. Although the figure caption is correct, the +/− symmetry of the orbitals involved is incorrectly shown in parts a and b. Figure 5a should show an antibonding a_{2u} MO, and Figure 5c should show a bonding a_{2u} MO.—Susan M. Kauzlarich