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1,2-Cyclopentadienyl Diimine−Group 13 Complexes

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Introduction

The burgeoning academic interest and financial significance of transition metal based olefin polymerization catalysts has recently spawned exploration of related Lewis acidic main group compounds. In this vein, Jordan and co-workers have recently reported the characterization of aluminum cationic complexes incorporating aminidinate[1,2] and aminotroponiminate ligands.[3] In addition, Smith et al.[4] have reported related diketiminato complexes. In our own efforts, we have previously reported the convenient synthesis of 1,2-cyclopentadienyl diimine anions, another bidentate, monoanionic ligand that chelates to early transition metal centers via two nitrogen atoms.[5] In this paper, we expand our efforts to group 13 species, developing synthetic routes to complexes of 1,2-cyclopentadienyl diimines. These systems are characterized, and the implications with respect to applications in catalysis are considered.

Experimental Section

General Data. All preparations were done under an atmosphere of dry, O_2-free N_2 employing both Schlenk line techniques and Innovative Technologies and Vacuum Atmospheres inert atmosphere gloveboxes. Solvents were purified employing a Grubb's type column system manufactured by Innovative Technology. All organic reagents were purified by conventional methods. ^{1}H and ^{13}C{^{1}H} NMR spectra were recorded on Bruker Avance-300 and -500 spectrometers operating at 300 and 500 MHz, respectively. Trace amounts of protonated solvents were used as references, and chemical shifts are reported relative to $SiMe_4$. Guelph Chemical Laboratories Ltd. Guelph, Ontario, performed combustion analyses. The compound $H[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]$ was prepared via literature methods.[5] $AlMeCl_2$, $AlMe_2Cl$, and $AlMe_3$ were purchased from the Aldrich Chemical Co.

Synthesis of $[1,2\text{-}C_5H_3(C(Ph)NH)_2]AlCl_2$, 1. To a solution of $H[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]$ (0.092 g, 0.34 mmol) in benzene (5 mL) was added 1.0 M $MeAlCl_2$ (0.34 mL, 0.34 mmol). The solution was allowed to stir for 12 h, and the solution was filtered. The remaining solvent was removed under vacuum to yield an orange solid. Recrystallization from benzene afforded orange crystals. Yield: 79%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.04 (2H, m), 6.97 (4H, m), 6.88 (4H, m), 6.76 (2H, d, $|J_{H-H}| = 4.0$ Hz), 6.26 (1H, t, $|J_{H-H}| = 4.0$ Hz), 6.12 (2H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 175.57, 143.64, 142.32, 141.21, 130.61, 129.52, 121.19, 119.49. Anal. Calcd for $C_{19}H_{15}N_2AlCl_2$: C, 61.64; H, 4.36. Found: C, 61.48; H, 4.21.

Synthesis of $[1,2\text{-}C_5H_3(C(Ph)NH)_2]GaCl_2$, 2. To a solution of $H[1,2\text{-}C_5H_3(C(Ph)NH)_2]$ (0.087 g, 0.32 mmol) in benzene (5 mL) was added $GaCl_3$ (0.056 g, 0.32 mmol) and NEt_3 (1.00 mmol). The solution was allowed to stir for 12 h, and the solution was filtered. The remaining solvent was removed under vacuum to yield an orange powder. Recrystallization from benzene afforded orange crystals. Yield: 73%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.01 (4H, d, $|J_{H-H}| = 6.9$ Hz), 6.86 (6H, m), 6.68 (2H, d, $|J_{H-H}| = 3.9$ Hz), 6.18 (1H, t, $|J_{H-H}| = 3.9$ Hz), 6.12 (2H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 175.1, 144.5, 141.5, 130.98, 129.7, 125.8, 120.9, 120.4. Anal. Calcd for $C_{19}H_{15}N_2GaCl_2$: C, 55.26; H, 3.91. Found: C, 55.03; H, 3.78.

Synthesis of $[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]AlR_2$, R = Me (3), Et (4). These complexes were prepared in a similar manner employing the appropriate Al reagent, and thus only a single representative preparation is described. To a solution of $H[1,2\text{-}C_5H_3(C(Ph)NH)_2]$ (0.07 g, 0.26 mmol) in benzene (5 mL) was added 2.0 M Me_3Al (0.13 mL, 0.26 mmol). The solution was allowed to stir for 12 h, and the solution was filtered. The remaining solvent was removed under vacuum to yield a yellow powder. Recrystallization from benzene afforded dark orange crystals of **3**. Yield: 83%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.19 (4H, d, $|J_{H-H}| = 7.4$ Hz), 7.09 (4H, t, $|J_{H-H}| = 7.4$ Hz), 6.97 (2H, t, $|J_{H-H}| = 7.4$ Hz), 6.77 (2H, d, $|J_{H-H}| = 3.8$ Hz), 6.41 (1H, t, $|J_{H-H}| = 3.8$ Hz), 6.15 (2H, s), −0.32 (6H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.16, 143.5, 139.5, 130.44, 129.6, 121.6, 118.6, 109.9, 7.32. Anal. Calcd for $C_{21}H_{21}N_2Al$: C, 76.57; H, 6.73. Found: C, 76.38; H, 6.55. (**4**) Yield: 69%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.24 (4H, d, $|J_{H-H}| = 6.9$ Hz), 7.08 (4H, m), 6.97 (2H, m), 6.71 (2H, d, $|J_{H-H}| = 3.8$ Hz), 6.34 (1H, t, $|J_{H-H}| = 3.8$ Hz), 6.17 (2H, s), 1.30 (4H, q, $|J_{H-H}| = 8.1$ Hz), 0.24 (6H, t, $|J_{H-H}| = 8.1$ Hz). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 177.2, 144.09, 140.3, 131.08, 130.07, 129.04, 121.98, 119.21, 25.82, 10.76.

Synthesis of $[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]MR_2$, M = Al, R = CH_2Ph (5), Ph (6), M = Ga, R = Me (7), Et (8), CH_2Ph (9), Ph (10). These complexes were prepared in a similar manner employing the appropriate Grignard reagent metal halide precursor, and thus only a single representative preparation is described. To a solution of **1** (0.053 g, 0.14 mmol) in benzene (5 mL) was added 1.0 M $PhCH_2MgCl$ (0.30 mL, 0.29 mmol). The solution was allowed to stir for 12 h, and the solution was filtered. The remaining solvent was removed under vacuum to yield an orange powder. **(5)** Yield: 75%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.26 (6H, m), 7.23 (4H, m), 7.16 (4H, m), 7.06 (6H, m), 6.89 (2H, d, $|J_{H-H}| = 3.86$ Hz), 6.53 (1H, t, $|J_{H-H}| = 3.8$ Hz), 6.15 (2H, s), 2.02 (4H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.28. 146.97, 143.00, 140.20, 130.92, 129.79, 128.97, 128.38, 127.27, 122.08, 121.54, 119.21, 19.68. (**6**) Yield: 82%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.99 (4H, m), 7.40 (4H, m), 7.33 (4H, m), 7.13 (2H, m), 7.04 (2H, m), 6.91 (4H, m), 6.79 (2H, d, $|J_{H-H}| = 3.9$ Hz), 6.42 (2H, s), 6.36 (1H, t, $|J_{H-H}| = 3.9$ Hz). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.73, 142.93, 141.08, 139.79, 138.31, 129.38, 128.52, 128.29, 121.36, 119.19. (**7**) Yield: 64%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.22 (4H, m), 7.09 (2H, m), 6.97 (4H, m), 6.73 (2H, d, $|J_{H-H}| = 3.82$ Hz), 6.43 (1H, t, $|J_{H-H}| = 3.82$ Hz), 6.16 (2H, s), −0.02 (6H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.15, 144.06, 138.37, 130.22, 129.65, 129.06, 117.56, 9.39. (**8**) Yield: 71%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.30 (4H, m), 7.09 (2H, m), 6.99 (4H, m), 6.72 (2H, d, $|J_{H-H}| = 3.8$ Hz), 6.41 (1H, t, $|J_{H-H}| = 3.8$ Hz), 6.22 (2H, s), 2.16 (4H, q, $|J_{H-H}| = 7.22$ Hz), 0.66 (6H, t, $|J_{H-H}| = 7.24$ Hz). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.80, 144.19, 138.71, 130.25, 129.52, 128.51, 120.98, 117.65, 47.66, 9.43. (**9**) Yield: 68%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.18 (4H, m), 7.11 (6H, d, $|J_{H-H}| = 7.5$ Hz), 6.98 (10H, m), 6.74 (2H, d, $|J_{H-H}| = 3.8$ Hz), 6.44 (1H, t, $|J_{H-H}| = 3.8$ Hz), 6.06 (2H,s), 2.17 (4H, s). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.29, 145.64, 143.53, 139.12, 130.70, 129.77, 128.82, 128.18, 127.64, 122.58, 121.03, 118.07, 48.42. (**10**) Yield: 74%. ^{1}H NMR (C_6D_6, 25 °C): δ 7.85 (4H, m), 7.35 (4H, m), 7.29 (4H, m), 7.18 (2H, m), 7.04 (2H, m), 6.92(4H, m), 6.73 (2H, d, $|J_{H-H}| = 3.9$ Hz), 6.42 (2H, s), 6.36 (1H, t, $|J_{H-H}| = 3.9$ Hz). ^{13}C{^{1}H} NMR (C_6D_6, 25 °C): δ 176.95, 145.10, 143.58, 140.17, 137.44, 130.37, 129.61, 128.68, 128.51, 127.89, 121.04, 118.31.

X-ray Data Collection and Reduction. X-ray-quality crystals of **1**, **2**, and **3** were obtained directly from the preparation as described above. The crystals were manipulated and mounted in capillaries in a glovebox, thus maintaining a dry, O_2-free environment for each crystal. Diffraction experiments were performed on a Siemens SMART System

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Table 1. X-ray Crystallographic Data[a]

	1	**2**	**3**
empirical formula	$C_{22}H_{18}AlCl_2N_2$	$C_{22}H_{18}Cl_2GaN_2$	$C_{24}H_{24}AlN_2$
fw	408.26	451.00	367.43
temp (k)	293(2)	293(2)	293(2)
wavelength (Å)	0.710 73 (Mo Kα)	0.710 73 (Mo Kα)	0.710 73 (Mo Kα)
space group	$P2(1)/n$ (no. 14)	$P2(1)/n$ (no. 14)	$P\bar{1}$ (no. 2)
a (Å)	9.859(9)	9.838(2)	7.685(2)
b (Å)	15.699(16)	15.702(3)	10.5330(15)
c (Å)	13.755(9)	13.755(2)	13.120(5)
α (deg)		91.94(3)	
β (deg)	105.00(6)	105.14(2)	91.86(3)
γ (deg)		95.39(2)	
vol (Å^3), Z	2056(3), 4	2051.1(7), 4	1056.0(5), 2
density(calcd) (g/cm^3)	1.319	1.461	1.156
μ (mm^{-1})	0.367	1.611	0.106
R indices [$I > 2\sigma(I)$]	R1 = 0.0996, wR2 = 0.1959	R1 = 0.0481, wR2 = 0.1372	R1 = 0.0436, wR2 = 0.1440
R indices (all data)	R_1 = 0.3534, wR_2 = 0.2836	R_1 = 0.0575, wR_2 = 0.1496	R_1 = 0.0527, wR_2 = 0.1521
GOF	0.774	1.006	1.189

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$, $R_w = [\sum(|F_o| - |F_c|)^2|/\sum|F_o|^2]^{0.5}$.

CCD diffractometer collecting a hemisphere of data in 1329 frames with 10 s exposure times. Crystal data are summarized in Table 1. The observed extinctions were consistent with the space groups in each case. The data sets were collected ($4.5° < 2\theta < 45-50.0°$). A measure of decay was obtained by recollecting the first 50 frames of each data set. The intensities of reflections within these frames showed no statistically significant change over the duration of the data collections. The data were processed using the SAINT and XPREP processing package. An empirical absorption correction based on redundant data was applied to each data set. Subsequent solution and refinement was performed using the SHELXTL solution package operating on a SGI Challenge mainframe computer with remote X-terminals or a PC employing X-emulation. The reflections with $F_o^2 > 3\sigma F_o^2$ were used in the refinements.

Structure Solution and Refinement. Non-hydrogen atomic scattering factors were taken from the literature tabulations.[6,7] The heavy atom positions were determined using direct methods employing either of the SHELXTL direct methods routines. The remaining non-hydrogen atoms were located from successive difference Fourier map calculations. The refinements were carried out by using full-matrix least-squares techniques on F, minimizing the function $\omega(|F_o| - |F_c|)^2$ where the weight ω is defined as $4F_o^2/2\sigma(F_o^2)$ and F_o and F_c are the observed and calculated structure factor amplitudes. In the final cycles of each refinement, all non-hydrogen atoms were assigned anisotropic temperature factors. Carbon bound hydrogen atom positions were calculated and allowed to ride on the carbon to which they are bonded assuming a C−H bond length of 0.95 Å. Hydrogen atom temperature factors were fixed at 1.10 times the isotropic temperature factor of the carbon atom to which they are bonded. The hydrogen atom contributions were calculated, but not refined. Each molecule crystallized with $0.5C_6H_6$ in the asymmetric unit. The final values of refinement parameters are given in Table 1. The locations of the largest peaks in the final difference Fourier map calculation as well as the magnitude of the residual electron densities in each case were of no chemical significance. Positional parameters, hydrogen atom parameters, thermal parameters, and bond distances and angles have been deposited as Supporting Information.

Results and Discussion

The 1,2-cyclopentadienyl diimine $H[1,2\text{-}C_5H_3(C(Ph)NH)_2]$ undergoes facile reaction with $MeAlCl_2$ to give the orange complex $[1,2\text{-}C_5H_3(C(Ph)NH)_2]AlCl_2$, **1**, in 79% yield (Scheme 1). ^1H and ^{13}C NMR data and elemental analyses are consistent with loss of methane via protonolysis. The analogous gallium compound $[1,2\text{-}C_5H_3(C(Ph)NH)_2]GaCl_2$, **2**, is readily prepared via reaction of $GaCl_3$ with the ligand precursor in the presence of base. In this manner **2** is subsequently isolated in 73% yield. NMR parameters for **1** and **2** were not unlike those observed for $[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]ZrCl_3(THF)$, suggesting a similar binding mode.[5] The precise structures of **1** and **2** were confirmed via an X-ray structural studies (Figures 1 and 2).

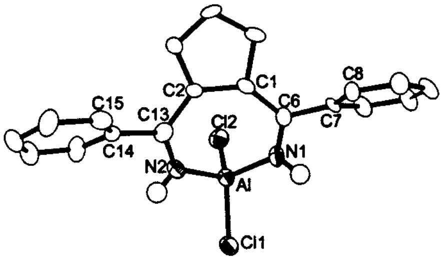
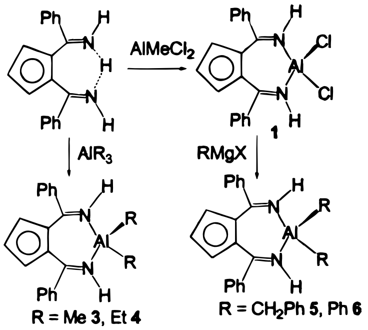


Figure 1. ORTEP drawings of **1**; 30% thermal ellipsoids are shown. Hydrogen atoms have been omitted for clarity. Al(1)−N(1) 1.846(9) Å, Al(1)−N(2) 1.860(8) Å, Al(1)−Cl(2) 2.109(5) Å, Al(1)−Cl(1) 2.111(5) Å, N(1)−C(6) 1.321(12) Å, N(2)−C(13) 1.314(12) Å, N(1)−Al(1)−N(2) 104.3(4)°, N(1)−Al(1)−Cl(2) 109.0(3)°, N(2)−Al(1)−Cl(2) 110.7(3)°, N(1)−Al(1)−Cl(1) 113.4(3)°, N(2)−Al(1)−Cl(1) 107.3(3)°, Cl(2)−Al(1)−Cl(1) 112.0(2)°, C(6)−N(1)−Al(1) 130.9(8)°, C(13)−N(2)−Al(1) 131.1(8)°.

Scheme 1



The solid-state structures of **1** and **2** are isostructural. The diimine anion binds to the Al or Ga center via the nitrogen atoms while the two chloride atoms complete the pseudotetrahedral coordination sphere of the main group metals. The Al−N distances average 1.853(9) Å while the corresponding Ga−N distances average 1.889(4) Å. These distances are significantly longer that those found in the iminoaluminum dimer $[CpAl(\mu\text{-}Naryl)]_2$ (1.796(2) and 1.811(3) Å)[8] but in the range of those found in the diketiminato derivatives described by Smith et al.[4]

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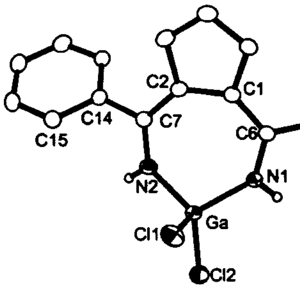


Figure 2. ORTEP drawings of **2**; 30% thermal ellipsoids are shown. Hydrogen atoms have been omitted for clarity. Ga(1)−N(2) 1.884(3) Å, Ga(1)−N(1) 1.895(3) Å, Ga(1)−Cl(2) 2.1564(11) Å, Ga(1)−Cl(1) 2.1595(13) Å, N(1)−C(6) 1.325(4) Å, N(2)−C(7) 1.331(4) Å, N(2)−Ga(1)−N(1) 105.06(13)°, N(2)−Ga(1)−Cl(2) 112.61(10)°, N(1)−Ga-(1)−Cl(2) 106.76(9)°, N(2)−Ga(1)−Cl(1) 109.13(11)°, N(1)−Ga(1)−Cl(1) 111.36(10)°, Cl(2)−Ga(1)−Cl(1) 111.72(5)°, C(6)−N(1)−Ga(1) 129.5(3)°, C(7)−N(2)−Ga(1) 128.1(2)°.

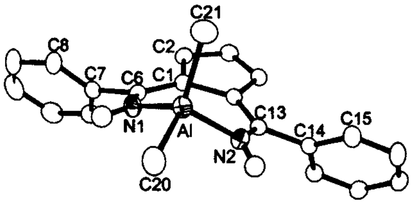
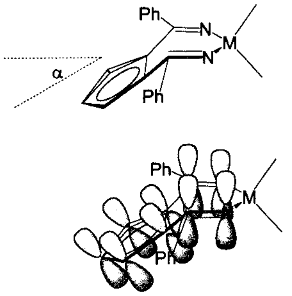


Figure 3. ORTEP drawings of **3**; 30% thermal ellipsoids are shown. Hydrogen atoms have been omitted for clarity. Al(1)−N(1) 1.8986-(17) Å, Al(1)−N(2) 1.9023(15) Å, Al(1)−C(20) 1.951(2) Å, Al(1)−C(21) 1.955(3) Å, N(1)−Al(1)−N(2) 99.72(7)°, N(1)−Al(1)−C(20) 106.36(9)°, N(2)−Al(1)−C(20) 108.17(9)°, N(1)−Al(1)−C(21) 111.33-(11)°, N(2)−Al(1)−C(21) 109.09(10)°, C(20)−Al(1)−C(21) 120.19-(12)°, C(6)−N(1)−Al(1) 132.06(13)°, C(13)−N(2)−Al(1) 130.61(12)°.

Chart 1



The corresponding M−Cl distances are 2.110(5) and 2.1579(13) Å for Al and Ga, respectively. These distances are typical of the Al−Cl distances in $[Cp^*AlCl(\mu\text{-}Cl)]_2$[9] and $[Cp^*AlMe(\mu\text{-}Cl)]_2$10 but significantly shorter than those seen in adducts of the form $(Ph_3PNSiMe_3)AlMe_2Cl$ (2.2150(12) Å).[11] The N−M−N bite angles for the diimine anion are 104.3(4)° and 105.06(13)° in **1** and **2**, respectively. The Cl−M−Cl angles are similar in the two compounds, both being close to 112°. The phenyl rings on the imino carbons are oriented such that the planes are approximately orthogonal to that of the cyclopentadienyl ring in the chelate backbone.

Reaction of $H[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]$ with either $AlMe_3$ or $AlEt_3$ affords the dimethyl and diethyl derivatives of **1**, $[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]AlR_2$, R = Me (**3**), Et (**4**) (Scheme 1). The rust orange compounds **3** and **4** were isolated in 83% and 69% yields, respectively. The NMR spectrum of **3** reveals a ^1H resonance attributable to the methyl group at −0.32 ppm, while the corresponding ^{13}C NMR resonance appears at 7.32 ppm. In the case of **4**, the ethyl groups give rise to signals in the ^1H and ^{13}C NMR at 1.30 and 0.24 ppm and 25.82 and 10.76 ppm, respectively. The related compounds $[(1,2\text{-}C_5H_3(C(Ph)NH)_2)]MR_2$, M = Al, R = CH_2Ph (**5**), Ph (**6**) (Scheme 1), M = Ga, R = Me (**7**), Et (**8**), CH_2Ph (**9**), and Ph (**10**), were prepared from reaction of **1** or **2** and the appropriate Grignard reagent. In the case of **3**, the structure was also confirmed crystallographically (Figure 3). The Al−N distances in **3** of 1.8986(17) and 1.9023(15) Å are slightly longer than those seen in **1**. This is consistent with the presence of the electron-donating methyl groups in **3**. The Al−C distances average 1.953(3) Å, which is slightly shorter than those seen in $(Ph_3PNSiMe_3)AlMe_2Cl$ (1.993(2) and 1.982(3)) Å).[11] It is also noteworthy that despite the fact that the N_2C_2Cp fragment is expected to be fully conjugated, the chelate ring and cyclopentadienyl group of **1**−**3** are not coplanar (Chart 1). The dihedral angles between the N_2C_2 plane and that of the cyclopentadienyl ring are 28.4°, 33.5°, and 30.5° for **1**−**3**, respectively. This is in direct contrast to the planarity of aminidinate,[1,2] aminotroponiminate,[3] and diketiminato[4] ligands. This disruption of conjugation is consistent with MO calculations previously described for the related Zr complexes that showed a localization of charge on the cyclopentadienyl ring.[5] Such charge separation infers an increase in the electrophilic character of the corresponding metal centers, a feature that might be expected to enhance the suitablity for applications in olefin polymerization chemistry.

In attempts to explore that potential, attempts to generate Al-based zwitterions or cations were undertaken. In the case of the reaction of **3** with $B(C_6F_5)_3$, monitoring the reaction by NMR revealed the formation of multiple products. The nature of these species remains unclear. Similarly, reaction with $Ph_3C[B(C_6F_5)_4]$ in the presence or absence of donors also resulted in several products which could be neither isolated nor identified. These observations infer that these reactions follow several competitive pathways. While abstraction of methyl from aluminum was anticipated, abstraction of proton from nitrogen and/or association of the Lewis acid with the electron rich cyclopentadienyl ring are also possible avenues for reactivity. Moreover, this behavior is in direct contrast to that of the aluminum complexes of aminidinate[1,2] and aminotroponiminate[3] ligands.

The present results clearly demonstrate that group 13 complexes of the monoanionic, bidentate, 1,2-cyclopentadienyl diimine ligands are readily available. The relatively short Al−C and Al−Cl distances suggest a charge separation and thus electrophilic metal centers. The incorporation of several reactive sites in these systems apparently precludes the use of the present systems as precursors to reactive cationic species. While this might be rectified by suitable ligand modification, it is the reactivity of these and other electrophilic group 13 species that is the subject of ongoing study.

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Supporting Information Available: Tables listing crystallographic data. This material is available free of charge via the Internet at http://pubs.acs.org.

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