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m-Terphenyl-Substituted Amidinates: Useful Ligands in the Preparation of Robust Aluminum Alkyls

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The syntheses of *m*-terphenyl-substituted amidines and their corresponding dialkylaluminum amidinate complexes are reported. The amidines are prepared in a one-pot reaction using 2,4,6-triphenylphenyllithium (formed *in situ* by the reaction of 2,4,6-triphenylphenylbromide with *n*-butyllithium) with dialkylcarbodiimides (RN═C═NR, R = cyclohexyl or isopropyl). An aqueous workup of the reaction mixtures results in the formation of sterically demanding amidines **2H** and **3H**. The amidines react readily with trimethylaluminum to form dialkylaluminum amidinate complexes. Two complexes have been prepared and comprehensively characterized in the solid state using single-crystal X-ray crystallography. In the crystalline state, dialkylaluminum amidinates are robust complexes that can be easily handled in air for short periods of time without noticeable decomposition.

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Introduction

The four-electron donor, monoanionic amidinate ligand **1** (Figure 1) is an important substituent in organometallic chemistry. It binds readily to numerous main group elements, transition metals, lanthanide, and actinide complexes.[1] Amidine metal complexes have been shown to catalyze a wide variety of reactions including olefin. For example, aluminum-based amidinate complexes, which are of particular relevance to this paper, have been used as efficient homogeneous catalysts in olefin polymerization reactions.[2−4] In addition, some interest has been focused on the applicability of aluminum amidinate complexes to serve as single-source precursors to materials containing the nitride ion.[5] Amidinate complexes are easily prepared from their corresponding amidines **1H**.[6]

In amidinate complexes, the coordination environment at the metal center can be modified by attaching different substituents to the ligand.[7−9] Sterically demanding substituents on the carbon and nitrogen atoms tend to push the lone pairs of electrons on the nitrogen atoms toward the metal center to favor the chelating bonding mode over the bridging bonding mode. On the other hand, nonbulky hydrogen and methyl groups on the carbon center favor the bridging or monodentate bonding modes (Figure 2).

Bulky substituents, such as adamantyl or 2,6-diisopropylphenyl, on the nitrogen center confer steric hindrance primarily in the amidine plane.[10,11] A sterically demanding *m*-terphenyl group on the central carbon atom provides steric protection in the plane of the ligand as well as above and below that plane. This creates a bowl-shaped environment for the amidinate ligand (Figure 3) that can stabilize unusual coordination patterns.[10] An example is the yttrium mono-amidinate complex isolated with the incorporation of an *m*-terphenyl on the central carbon of *N*,*N′*-diisopropylamidine. Yttrium bis-amidinate complexes are isolated when smaller substituents are used.[10]

Here we report a simple preparation of the bulky amidine ligands, *N*,*N′*-diisopropyl-2,4,6-triphenylbenzamidine **2H** and *N*,*N′*-dicyclohexyl-2,4,6-triphenylbenzamidine **3H**, and the synthesis of their corresponding dimethylaluminum amidinate complexes **2AlMe$_2$** and **3AlMe$_2$**.[12]

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(12) For the purposes of this paper, the 2,4,6-triphenylpheny ligand may be considered to be a substituted *m*-terphenyl ligand For reviews, see: Twamley, B.; Haubrich, S. T.; Power, P. P. *Adv. Organomet. Chem.* **1999**, *44*, 1. Clyburne, J. A. C.; McMullen, N. *Coord. Chem. Rev.* **2000**, *210*, 73.

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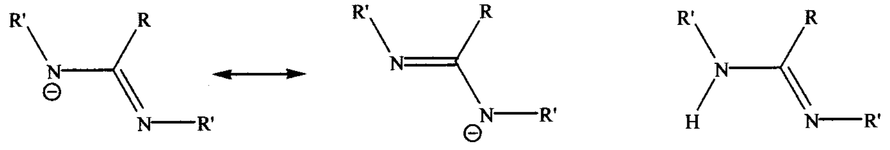

1 **1H**

Figure 1. Amidinate and amidine ligands.

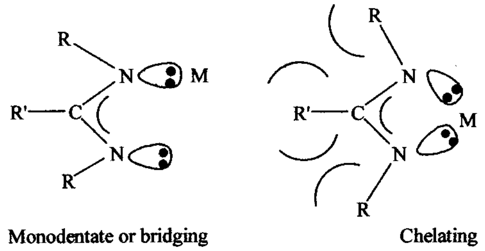

Figure 2. Steric effects of ligand substituents.

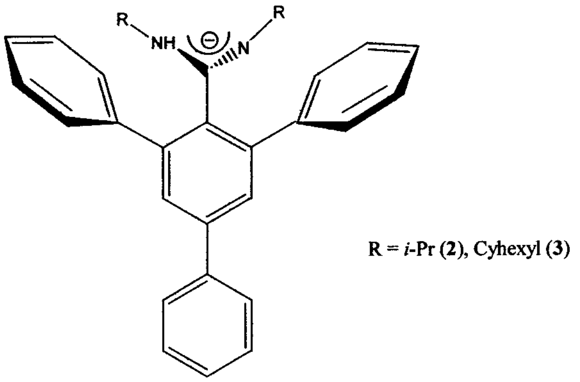

Figure 3. Bowl-shaped environment of the *m*-terphenyl-substituted amidinate ligand.

Experimental Section

General Procedures. An MBraun UL-99-245 drybox and standard Schlenk techniques on a double manifold vacuum line were used in the manipulation of air and moisture sensitive compounds. Anhydrous solvents were used as received from Aldrich Chemical Co. NMR spectra were recorded on a Bruker AC 250 spectrometer in five millimeter quartz tubes at the Atlantic Region Magnetic Resonance Center. ^{1}H and ^{13}C{^{1}H} chemical shifts are reported in parts per million (ppm) downfield from tetramethylsilane (TMS) and are calibrated to the residual signal of the solvent. The ^{27}Al NMR were obtained on a Bruker AMX 400 spectrometer in a 10 mm tube with $[Al(H_2O)_6]^{3+}$ as external reference. Infrared spectra were obtained using a Perkin-Elmer Model 683 spectrometer with the % transmittance values reported in cm^{-1}. Melting points were measured using a Mel-Temp apparatus and are uncorrected. Electron impact mass spectral data were obtained at Simon Fraser University (Burnaby, B.C.) using an HP 5985 GC mass spectrometer. Full experimental details for the preparation of **2H** and **3H** can be found in the Supporting Information.

[(C$_6$H$_2$Ph$_3$)C(N*i*-Pr)$_2$Al(CH$_3$)$_2$] (2AlMe$_2$). *N*,*N'*-Diisopropyltriphenylbenzamidine (1.00 g, 2.31 mmol) was dissolved in ca. 20 mL of toluene. A 2 mL aliquot (4.0 mmol) of 2 M $AlMe_3$ in toluene was added dropwise and stirred for 12 h. The solvents were removed *in vacuo*, and the resulting solid was dissolved in diethyl ether and filtered to remove a small amount of insoluble material. Upon standing for 1 day, large cube-shaped crystals were isolated and characterized as **2AlMe$_2$**. Yield: 0.55 g, 1.12 mmol, 50%, mp $T > 250$ °C (dec). Anal. Calcd for $C_{33}H_{37}AlN_2$: C, 81.11; H, 7.63; N, 5.73; Al, 5.52. Found: C, 80.32; H, 7.55; N, 5.52. ^{1}H NMR (CD_2Cl_2): δ 7.81−7.33 (m, 17H), 3.00 (s, 2H), 0.48 (s, 12H), −0.82 (s, 6H). ^{13}C{^{1}H}NMR (CD_2Cl_2): δ 142.0, 140.6, 129.7, 129.4, 128.8, 128.2, 127.6, 25.0, −10.2 (v br). ^{27}Al NMR (C_6D_6): δ 170 (v br). IR (Nujol, cm^{-1}): 3080w, 3040w, 3020w, 1490m, 1370m, 1340m, 1270w, 1180w, 1120w, 890w, 770m, 760m, 725m, 700s, 680m, 665m.

[(C$_6$H$_2$Ph$_3$)C(N*Cy*)$_2$Al(CH$_3$)$_2$] (3AlMe$_2$). The synthesis of this compound is similar to that of **2AlMe$_2$**; however, the solid was recrystallized from toluene. A 1.00 g sample of **3H** yielded 0.70 g (1.23 mmol, 64%) of **3AlMe$_2$**. Mp: 222−225 °C. Anal. Calcd for $C_{39}H_{45}AlN_2$: C, 82.36; H, 7.97; N, 4.93; Al, 4.74. Found: C, 81.89; H, 7.84; N, 4.50. ^{1}H NMR (CD_2Cl_2): δ 7.76−7.33 (m, 17H), 1.38 (s, 2H), 0.72 (s, 20H), −0.77 (s, 6H). ^{13}C{^{1}H}NMR (CD_2Cl_2): δ 170.3, 142.7, 142.0, 140.9, 129.6, 129.4, 128.8, 128.8, 128.4, 128.2, 127.5, 125.6, 35.7, 25.9, 25.8, −8.0 (very broad). ^{27}Al NMR (C_6D_6): δ 184 (v br). IR (Nujol, cm^{-1}): 1630m, 1590w, 1255m, 1075m, 1030m, 890m, 800m, 760m, 750m, 700s, 680m.

X-ray Crystallographic Structural Determination. A single crystal of **X** (**X** = **2H**, **3H**, **2AlMe$_2$**, or **3AlMe$_2$**) was mounted on a Rigaku AFC5R diffractometer equipped with a rotating anode generator and utilizing graphite-monochromated Cu Kα radiation (**2H**, **2AlMe$_2$**, **3AlMe$_2$**) or Mo Kα radiation (**3H**). Cell constants and an orientation matrix for data collection were obtained from a least squares refinement using the setting angles of 25 carefully centered reflections. Data were collected at room temperature (23 °C) and were corrected for Lorentz and polarization effects. The structure was solved by direct methods[13] and expanded using Fourier techniques.[14] Full matrix least squares refinement was carried out using SHELXL97.[15] The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in their geometrically calculated positions and allowed to ride on the heavy atoms to which they were bonded, with U_{iso} equal to $1.2U_{eq}$ of the heavy atom ($1.5U_{eq}$ for methyl hydrogens). Full details of the structures and the refinements are given in the Supporting Information.

Results and Discussion

Synthesis of Amidines. Amidines with bulky substituents on the central carbon atom are sterically protected in the amidine plane. The use of the *m*-terphenyl group extends this steric protection both above and below the amidine plane. Most amidine preparations previously reported produce amidines with substituents such as H, CH_3, or C_6H_5 on the central carbon. Recently, a report has described an elegant synthetic route to the preparation of amidinate complexes with bulky *m*-terphenyl substituents on the central carbon of the amidinate ligand.[10] This preparation involves the generation of an *m*-terphenyl-substituted amidine, which is lithiated and reacted with metal halides to obtain the amidinate complexes.[10] In the

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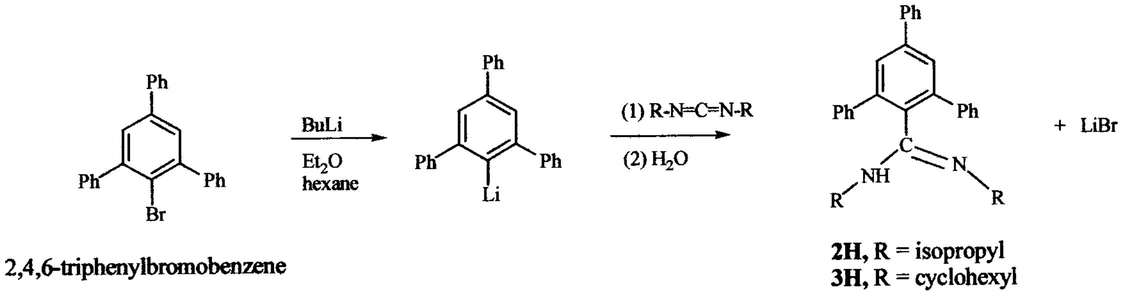
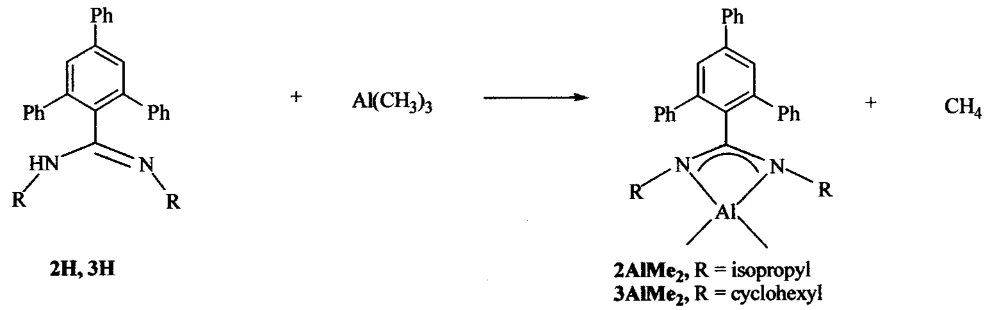
Scheme 1. Preparation of Bulky Amidines via Lithiated Terphenyls



Scheme 2. Preparation of Aluminum Amidinate Complexes



current research project, amidines with the triphenylphenyl substituent were prepared according to Scheme 1.

The substituted *m*-terphenyl, 2,4,6-triphenylbromobenzene, is easily metalated by combining it with 1.6 M *n*-BuLi in hexane and stirring for 4 h.[16] Triphenylphenyllithium reacts readily with a carbodiimide to produce a lithiated amidinate. The amidinate is quenched with water followed by extraction with CH_2Cl_2 and recrystallization from toluene to produce the neutral amidine. The identities of *N*,*N*′-diisopropyl-2,4,6-triphenylbenzamidine (**2H**) and *N*,*N*′-dicyclohexyl-2,4,6-triphenylbenzamidine (**3H**) have been confirmed by spectroscopic and X-ray crystallographic analysis.

The N−H, C=N, and C−N stretching frequencies are in agreement with IR spectral frequencies reported for other amidines.[17] NMR solution studies (^1H and ^{13}C) in $CDCl_3$ confirm the presence of isopropyl and cyclohexyl groups in two different chemical environments, namely, as an imine nitrogen substituent and as an amine nitrogen substituent. However, the signals for these resonances are broadened due to interconversion between *Z*-*syn* and *E*-*syn* isomers that occur on an NMR time scale. This phenomenon has been observed in amidines with *m*-terphenyl substituents on the central carbon atom,[10] as well as in amidines with bulky substituents such as 2,6-diisopropylphenyl on the nitrogen atom.[11]

X-ray crystallographic studies were performed on the compounds **2H** and **3H** to evaluate the steric demands of the *m*-terphenyl group with respect to the amidine fragment. An ORTEP view of **2H** is shown in Figure 4 along with selected bond lengths and bond angles. The molecular structures of the amidine compounds show that they exist in the *E*-*syn* configuration. In general, the *E* conformation is energetically more favored than its *Z* counterpart.[17]

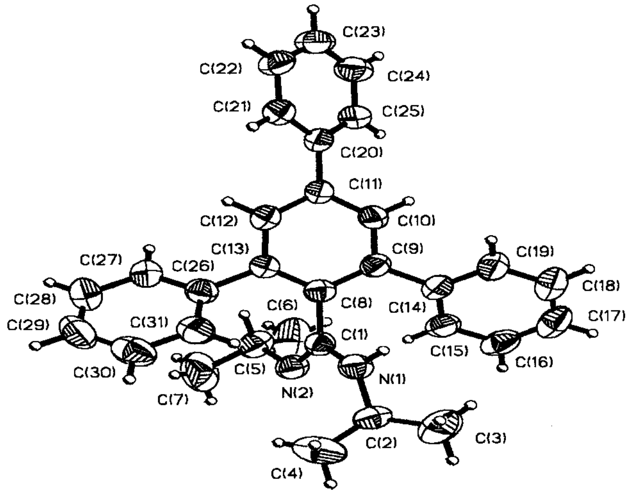


Figure 4. ORTEP view of the molecular structure of 2H showing the labeling scheme. Thermal ellipsoids are shown at 50% probability level; hydrogen atoms, except H(1), have been removed for clarity. Selected bond lengths [Å]: N(1)−C(1) 1.368(4), N(1)−C(2) 1.462(4), N(1)−H(1) 0.8900, N(2)−C(1) 1.282(4), N(2)−C(5) 1.460(4), C(1)−C(8) 1.513(4). Selected bond angles [deg]: C(1)−N(1)-C(2) 122.5(3), C(1)−N(1)−H(1)121.8, C(2)−N(1)−H(1) 112.2, C(1)−N(2)−C(5) 120.7(3), N(2)−C(1)−N(1) 120.2(3), N(2)−C(1)−C(8) 126.4-(3), N(1)−C(1)−C(8) 113.4(3); selected torsion angle [deg] N(1)−C(1)−C(8)−C(9) 70.75(0.39).

Syntheses of Dialkylaluminum Amidinate Complexes. To prepare robust aluminum amidinate complexes, the amidines **2H** and **3H** were dissolved in toluene and reacted with an excess of $Al(CH_3)_3$ in hexane and stirred overnight (Scheme 2). After solvent removal *in vacuo* the solid materials were recrystallized from Et_2O and toluene, respectively. The resulting well-defined block crystals were identified as **2AlMe$_2$** and **3AlMe$_2$** by spectroscopic and X-ray crystallographic analyses.

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Table 1. Selected Bond Lengths and Bond Angles of Amidines and Corresponding Amidinates

	2H	3H	2AlMe$_2$	3AlMe$_2$	*t*-Bu-C{(N-Cy)$_2$AlMe$_2$
			Selected Bond Lengths (Å)		
N(1)−C(1)	1.368(4)	1.379(4)	1.325(3)	1.325(7)	1.343(2)
N(2)−C(1)	1.282(4)	1.283(3)	1.325(3)	1.328(7)	1.339(2)
C(1)−R^1	1.513(4)	1.498(4)	1.506(5)	1.508(7)	1.540(2)
N(1)−R	1.462(4)	1.450(4)	1.465(4)	1.447(7)	1.456(2)
N(2)−R	1.460(4)	1.462(4)	1.465(4)	1.458(7)	1.454(2)
Al−C(3)			1.947(3)	1.949(7)	-
Al−N(1)			1.917(3)	1.936(5)	-
Al−C(2)			1.947(3)	1.944(7)	1.958(2)
Al−N(2)			1.917(3)	1.942(5)	1.9124(14)
			Selected Bond Angles (deg)		
N(1)−C(1)−N(2)	120.2(3)	119.8(3)	109.3(4)	110.8(6)	107.8
R^1−C(1)−N(1)	113.4(3)	114.0(2)	125.4(2)	124.2(6)	
R^1−C(1)−N(2)	126.4(3)	126.2(3)	125.4(2)	125.0(6)	
C(1)−N(1)−R	122.5(3)	122.9(2)	127.8(3)	126.6(6)	131.4
C(1)−N(1)−H or Al	121.8	118.6	91.1(2)	90.5(4)	91.3
R−N(1)−H or Al	112.2	118.6	141.2(2)	142.8(4)	136.5
C(1)−N(2)−R	120.7(3)	120.5(2)	127.8(3)	126.0(5)	132.5
C(1)−N(2)−Al			91.1(2)	90.1(4)	92.1
R−N(2)−Al			141.2(2)	143.8(4)	133.4
N(1)−Al−N(2)			68.6(2)	68.5(2)	68.7
N(1)−Al−C(3)			115.32(13)	114.8(3)	
N(2)−Al−C(2)			115.32(13)	115.6(3)	
C(3)−Al−C(2)			117.4(2)	119.2(3)	116.2

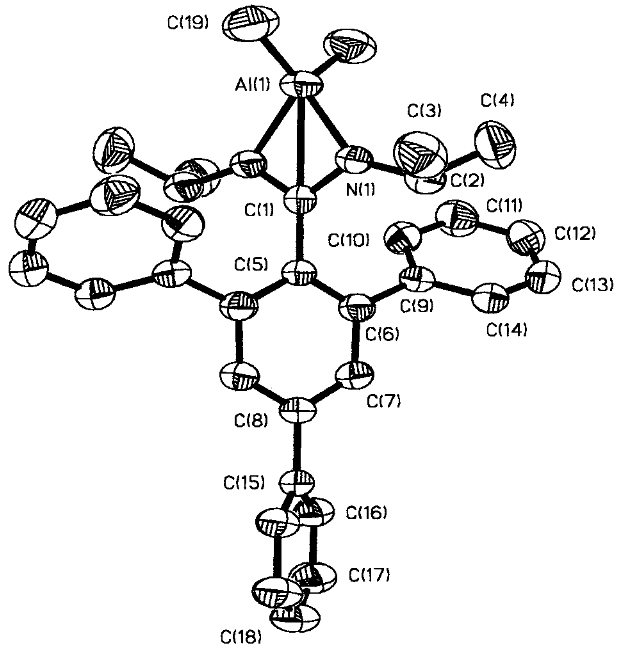
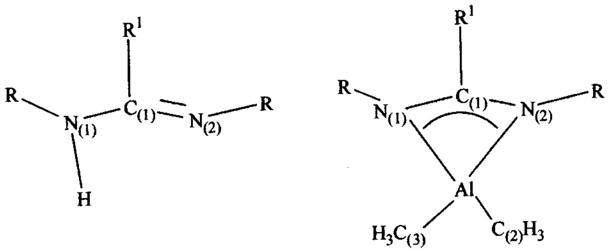


Figure 5. ORTEP view of the dialkylaluminum amidinate complex 2AlMe$_2$ showing the labeling scheme. Thermal ellipsoids are shown at 50% probability level; hydrogen atoms have been removed for clarity; the symmetry $(1-x, y, 0.5-z)$ is required to generate the second half of the molecule. Selected bond lengths [Å]: Al(1)−N(1) 1.917(3), Al(1)−C(19) 1.947(3), Al(1)−C(1) 2.351(4), N(1)−C(1) 1.325(3), N(1)−C(2) 1.465(4), C(1)−C(5) 1.506(5). Selected bond angles [deg]: N(1)−Al(1)−N(1A) 68.6(2), C(19)−Al(1)−C(19A) 117.4(2), C(1)−N(1)−C(2) 127.8(3), C(1)−N(1)−Al(1) 91.1(2), C(2)−N(1)−Al(1) 141.2(2) N(1)−C(1)−C(5) 125.4(2). Selected torsion angle [deg]: N(1)−C(1)−C(5)−C(6) −64.24(0.20).

In the IR spectra of the reaction products, **2AlMe$_2$** and **3AlMe$_2$**, the N−H stretching frequency of the amidine (3420−3400 cm^{-1}) is absent. This is consistent with reaction Scheme 2. Likewise, NMR studies show the disappearance of the signal attributed to the N-*H* resonance. In addition, the isopropyl or cyclohexyl groups become equivalent, and the Al-C*H*$_3$ resonance occurs at ca. 0.1 ppm. These observations are consistent with ^{1}H NMR studies of similar compounds with symmetric dialkylaluminum fragments.[3] The ^{13}C NMR signal for Al-*C* is broad due to the ^{27}Al quadrupole. Similar to previously characterized aluminum amidinate derivatives, the ^{27}Al NMR signal that occurs between 184 and 170 ppm is also broad.[3]

Chart 1



Solid-State Structures of 2AlMe$_2$ and 3AlMe$_2$. The molecular structure and atom-labeling scheme of the aluminum amidinate complexe **2AlMe$_2$** is presented in Figure 5. The structures of the complexes consist of discrete molecules that do not display unusual intermolecular interactions. Table 1 summarizes some selected bond lengths and bond angles of the amidines **2H** and **3H** and their corresponding aluminum amidinate complexes, **2AlMe$_2$** and **3AlMe$_2$.** Also included are the data for an aluminum amidinate complex, *t*-Bu-C{(N−Cy)$_2$AlMe$_2$.[3] For clarity, the numbering of the data in the table refers to the amidine and amidinate structures given in Chart 1.

The formation of a four-membered Al−N−C−N ring system delocalizes the N−C=N bonding in the amidinates. In the amidines the bonds from the nitrogen atoms to the central carbon, N(1)−C(1) [C−N] and N(2)−C(1) [C=N], are localized. As revealed both by crystallographic studies and ^{1}H NMR studies in solution, these bonds are equivalent in the amidinate complexes. For example, in **2AlMe$_2$**, which is centrosymmetric, the N(1)−C(1) distance of 1.325(3) Å is

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between the single and double bond lengths, 1.368(4) and 1.282(4) Å, respectively, of the precursor amidine **2H**. This is consistent with a bond order of 1.5 in the delocalized N−C−N bonding systems. Furthermore, the bond lengths from the aluminum center to the two nitrogen atoms are crystallographically equal in both complexes. This confirms the symmetrical bonding pattern of the amidinate complexes **$2AlMe_2$ and $3AlMe_2$**. There is very little change in the bond distances, C−R^1, N(1)−R, and N(2)−R, of the amidinates compared to those of the corresponding amidines.

In the amidine compounds, the sum of the three bond angles around C(1) is 360°. This confirms a trigonal planar geometry, consistent with the anticipated sp^2 geometry. However, the ∼120° N(1)−C(1)−N(2) angle in the amidines is reduced to a tight ∼109° in the amidinates due to the presence of the four-membered ring. This is compensated for by an increase in the R^1−C(1)−N(1) angle. As expected, the C(1)−N(1)−R, R−N(1)−Al, C(1)−N(2)−R, and R−N(2)−Al angles are increased in comparison to the C(1)−N(1)−R and R−N(1)−H angles of the amidines. Compared with the C(1)−N(1)−H angle of ca. 120° in the amidines, the C(1)−N(1)−Al and C(1)−N(2)−Al angles are ∼90°. These changes can be explained by the steric interactions between the *m*-terphenyl groups on C(1) and the R groups on the nitrogen atoms, which force the lone pair on the nitrogen atoms toward the center of the amidinate ligand. In addition, the *m*-terphenyl group on the central carbon atom provides steric protection above and below the plane of the amidinate ligand.

The Al−C(methyl) bond lengths observed in **$2AlMe_2$** and **$3AlMe_2$** closely resemble the terminal Al−C(methyl) bond distance observed in $(Me_3Al)_2$.[18] However, the bond angles around the Al centers are not characteristic of a regular tetrahedron. The acute N(1)−Al−N(2) angle (∼69°) indicates considerable strain around the Al center in both **$2AlMe_2$** and **$3AlMe_2$**. The changes in the bond angles confirm that the coordination environment at the Al center has been affected by the steric interactions of the substituents on the amidinate ligand.

Hydrolysis of the aluminum amidinate complexes, after exposure to air, results in the regeneration of the amidine compounds. Nevertheless, a timed assay of the IR spectra shows that the N−H stretch at ca. 3420 cm^{-1} reappears only after the amidinate complexes have been exposed to air for at least 15 min. This is in sharp contrast to other aluminum alkyl compounds, where the high sensitivity of Al−C bonds to air and moisture often results in their being spontaneously flammable in air.[19,20] In fact, a strong OH band at 3500 cm^{-1} is observed in the IR spectrum of $Al(CH)_3$ in hexane after it has been exposed to air for only 1 min. The stability of the aluminum amidinate complexes studied here can be attributed to the steric protection imparted by the *m*-terphenyl group attached to the central carbon atom of the amidinate ligand.

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Supporting Information Available: Tables of X-ray structural data for compounds **2H**, **3H**, **$2AlMe_2$, and $3AlMe_2$** are available free of charge via the Internet at http://pubs.acs.org.

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