Group 13 chemistry of sterically hindered amines. Synthesis and molecular structure of $[Me_2M\text{-}Ada]_2$ (M = Al, Ga; Ada = 1-aminoadamantane) and $[(i\text{-}Bu)Al\text{-}iminodibenzyl]_2$

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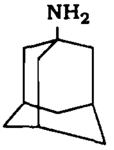
Abstract

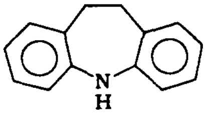
Reaction of Me_3M (M = Al, Ga) with 1-aminoadamantane, Ada, in toluene affords crystalline complexes of the type $[Me_2M\text{-}Ada]_2$. The gallium compound, **I**, isomorphous with the aluminum compound, **II**, crystallizes in the triclinic space group $P\bar{1}$ with unit cell parameters $a = 6.728(1)$, $b = 9.957(2)$, $c = 10.341(2)$ Å, $\alpha = 74.30(1)$, $\beta = 73.94(1)$, $\gamma = 72.19(1)°$, $V = 620.6(2)$ Å^3 and $D_{calc} = 1.34$ g cm^{-3} for $Z = 1$. Least-squares refinement based on 1794 observed reflections ($I > 3\sigma(I)$) converged at $R = 0.059$, $R_w = 0.080$. **I** resides about a crystallographic center of symmetry and contains a planar Ga_2N_2 four-membered ring. The Ga_2N_2 ring has a mean Ga–N bond distance of 2.030(9) Å. The mean Ga–C bond distance in the dimethylgallium unit is 1.974(9) Å. The crystalline product $[(i\text{-}Bu)Al\text{-}iminodibenzyl]_2$ (**III**), isolated from reaction of diisobutylaluminum hydride with iminodibenzyl, $(C_{14}H_{12})NH$, in toluene, crystallizes in the triclinic space group $P\bar{1}$ with unit cell parameters $a = 10.767(2)$, $b = 12.264(3)$, $c = 13.230(4)$ Å, $\alpha = 75.57(2)$, $\beta = 74.14(2)$, $\gamma = 73.58(2)°$, $V = 1583.7(7)$ Å^3 and $D_{calc} = 1.16$ g cm^{-3} for $Z = 2$. Least-squares refinement based on 2216 observed reflections ($I > 3\sigma(I)$) converged at $R = 0.093$, $R_w = 0.118$. Compound **III** results from a unique condensation reaction in which *ortho*-metallation of one of the aromatic rings of iminodibenzyl was observed. Compound **III** has a mean Al–N bond distance of 2.036(7) Å. The mean $Al\text{–}C_{isobutyl}$ bond distance of 1.98(3) Å is comparable to 1.954(8) Å for the $Al\text{–}C_{aromatic}$ fragment.

Introduction

The interaction of Group 13 metals with Group 15 species is an active area of research. Some time ago this laboratory began to explore the Group 13 chemistry of multidentate macrocyclic and open chain amines. These systems were often observed to result in interesting metal–nitrogen compounds with the Group 13 metal frequently found in high coordination environments [1]. Interest in this laboratory recently shifted to the corresponding chemistry of sterically hindered amines in anticipation of obtaining metal centers in low coordination environments. Low coordination number Group 13 compounds are interesting in that π-bonding has been suggested in such complexes [2]. The sterically hindered amines 1-aminoadamantane (a) and iminodibenzyl (b) were chosen to initiate these investigations.



(a)



(b)

In this paper we report the synthesis and structure of $[Me_2M\text{-}Ada]_2$ (M = Ga (**I**), Al (**II**)) and $[(i\text{-}Bu)Al\text{-}iminodibenzyl]_2$ (**III**).

Experimental

General procedures

Standard Schlenk techniques were employed in conjunction with an inert atmosphere dry box (Vacuum Atmospheres HE-43 Dri-Lab). Toluene was distilled over sodium benzophenone under an atmosphere of argon prior to use. Trimethylgallium and diisobutylaluminum hydride, generously donated by Ethyl Corp., were used as received. 1-Aminoadamantane and iminodibenzyl, purchased from Aldrich Chemical Co., were used as received. ^{1}H NMR

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data were recorded on a Bruker AC-300-P NMR spectrometer. Single crystal X-ray data were collected on a Nicolet R3m/V diffractometer with graphite-monochromated Mo Kα radiation.

Synthesis of [Me_2Ga-Ada]$_2$ (I)

Inside the dry box a reaction vessel was charged with 1-aminoadamantane (0.75 g, 5 mmol) and toluene (20 ml). Trimethylgallium (0.5 ml, 5 mmol) was slowly added via syringe. Reaction was immediate and vigorous resulting in a clear colourless solution. The reaction vessel was subsequently removed from the dry box and heated (120 °C) in an oil bath for 24 h. Upon cooling, a multitude of colourless, air sensitive, X-ray quality crystals of **I** resulted in quantitative yield. m.p. 230 °C. ^{1}H NMR ($CDCl_3$, $Si(CH_3)_4$, external reference): δ -0.31 (s, 6H, $Ga(CH_3)_2$), 0.77 (s, 1H, N*H*), 1.56 (cm, 12H, CH_2– Ada), 1.99 (bs, 3H, C*H*– Ada).

Synthesis of [Me_2Al-Ada]$_2$ (II)

The preparation of **II** was carried out in a manner analogous to that for compound **I**. 1-Aminoadamantane (0.75 g, 5 mmol) was allowed to react with Me_3Al (0.47 ml, 5 mmol) in toluene (15 ml). The system was heated (120 °C) for 24 h. Upon cooling, a multitude of colourless, air sensitive, X-ray quality crystals resulted in quantitative yield. m.p. 245 °C. ^{1}H NMR ($CDCl_3$, $Si(CH_3)_4$, external reference): δ -0.69 (s, 6H, $Al(CH_3)_2$), 0.66 (s, 1H, N*H*), 1.54, 1.62 (6H, AB type, $J_{AB} = 12.4$ Hz, CH_2– Ada) 1.70 (d, 6H, CH_2– Ada), 2.03 (bs, 3H, C*H*– Ada).

Synthesis of [(i-Bu)Al-iminodibenzyl]$_2$ (III)

A 50 ml reaction vessel was charged with iminodibenzyl (0.98 g, 5 mmol) and taken into the dry box. Inside the dry box, toluene (20 ml) and diisobutylaluminum hydride (0.85 ml, 5 mmol) were added. A colourless homogeneous solution resulted. The solution was heated for several hours in an oil bath (120 °C). The reaction vessel was vented frequently. The solution was filtered via cannula to a 100 ml Schlenk flask. Reduction of solvent and subsequent cooling of the flask resulted in the formation of parallelepiped, colourless, air sensitive, X-ray quality crystals in approximately 50% yield (based on iminodibenzyl). m.p. 200 °C (dec.). ^{1}H NMR ($CDCl_3$, $Si(CH_3)_4$, external reference): δ 0.33 (d, 2H, Al-CH_2), 0.75 (d, 6H, $(CH_3)_2$), 1.7 (cm, 1H, C*H*), 3.09 (s, 4H, $(CH_2)_2$), 6.8–7.7 (cm, 7H, C_6H_3, C_6H_4).

X-ray structural solution and refinement for [Me_2Ga-Ada]$_2$ (I)

A colourless parallelepiped crystal of **I** was mounted in a glass capillary under an inert atmosphere of argon. Cell parameters and an orientation matrix for data collection, obtained from a least-squares refinement of the setting angles of 50 carefully centered reflections in the range $28.11 < 2\theta < 42.15°$ corresponded to a triclinic cell. The space group was determined to be $P\bar{1}$; the unit cell contains one molecule which is situated about an inversion center at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). The structure was solved by direct methods and refined, based on 1794 observed reflections, using SHELXTL [3]. Anisotropic thermal parameters were used for the non-hydrogen atoms. Hydrogen atoms were located by standard difference Fourier techniques; adamantyl and amine hydrogen atoms were refined isotropically, while methyl hydrogens were included in the structure factor calculation at idealized positions (d(C–H) = 0.96 Å), and were allowed to ride on the atom to which they were bonded. The weighting scheme was based on counting statistics and included a factor ($p = 0.0005$) to downweight the intense reflections. Peaks on the final difference map ranged from -1.01 to 1.23 e$^-$/Å^3. Refinement converged at $R = 0.059$, $R_w = 0.080$. Relevant crystallographic data are given in Table 1.

X-ray structural data for [Me_2Al-Ada]$_2$ (II)

The aluminum compound, **II**, isomorphous with the gallium compound, **I**, crystallizes in the triclinic space group $P\bar{1}$ with unit cell parameters $a = 6.728(6)$, $b = 9.912(6)$, $c = 10.338(10)$ Å, $\alpha = 74.56(7)$, $\beta = 74.12(7)$, $\gamma = 72.26(6)°$ and $V = 618.9(9)$ Å^3 for $Z = 1$.

TABLE 1. Crystallographic data for [Me_2Ga-Ada]$_2$ (**I**) and [(i-Bu)Al-iminodibenzyl]$_2$ (**III**)

	I	III
Formula	$C_{24}H_{44}N_2Ga_2$	$C_{36}H_{40}N_2Al_2$
Formula weight	500.07	554.69
Crystal system	triclinic	triclinic
a (Å)	6.728(1)	10.767(2)
b (Å)	9.957(2)	12.264(3)
c (Å)	10.341(2)	13.230(4)
α (°)	74.30(1)	75.57(2)
β (°)	73.94(1)	71.14(2)
γ (°)	72.19(1)	73.58(2)
V (Å^3)	620.6(2)	1583.7(7)
Z	1	2
D_{calc} (g, cm^{-3})	1.34	1.16
Diffractometer	Nicolet R3m/V	
Radiation	Mo Kα ($\lambda = 0.71073$ Å)	
Temperature (°C)	21	
2θ Range (°)	3.50–48.0	3.50–45.0
Reflections collected	2148	3618
Reflections observed	1794	2216
GOF	2.73	3.28
R	0.059	0.093
R_w	0.080	0.118

Data collection and refinement for compound **II** were not pursued.

X-ray structural solution and refinement for [(i-Bu)Al-iminodibenzyl]₂ (III)

A colourless parallelepiped crystal of **III** was mounted in a glass capillary under an inert atmosphere of argon. Cell parameters and an orientation matrix for data collection, obtained from a least-squares refinement of the setting angles of 50 carefully centered reflections in the range $27.12 < 2\theta < 40.57°$ corresponded to a triclinic cell. The space group was determined to be $P\bar{1}$ with two molecules per unit cell. The asymmetric unit consists of two half-molecules, each of which is situated about an inversion center ($\frac{1}{2}$, 0, 1, molecule one; 0, $\frac{1}{2}$, $\frac{1}{2}$, molecule two) which generates the other half of the molecule. Both molecules have disordered isobutyl groups; molecule two has additional disorder involving the amine ligand; this disorder contributes to the relatively high residuals obtained for compound **III**. The structure was solved by direct methods. Anisotropic thermal parameters were used for full-occupancy non-hydrogen atoms, partial occupancy atoms were refined isotropically; hydrogen atoms were not included in the model. The final cycle of full-matrix least-squares refinement was based on 2216 observed reflections ($I > 3\sigma(I)$) and converged with final residual values of $R = 0.093$, $R_w = 0.118$. The weighting scheme was based on counting statistics and included a factor ($p = 0.0005$) to downweight the intense reflections. Peaks on the final difference map ranged from -0.29 to 0.36 e$^-$/Å^3. Relevant crystallographic data are given in Table 1.

Results and discussion

This laboratory has long had an interest in the Group 13 chemistry of oxygen- [4], sulfur- [5], and nitrogen-based multidentate ligands. These studies have revealed a particularly rich and varied chemistry often involving condensation reactions which resulted in novel cage compounds with high coordination metal centers. Organoaluminum chemistry of sterically bulky amines represents an important extension of this work. To this end, 1-aminoadamantane and iminodibenzyl were examined. Herein we report the synthesis and structure of [Me_2M-Ada]₂ (M = Ga (**I**), Al (**II**)) and [(i-Bu)Al-iminodibenzyl]₂ (**III**).

Compounds **I** and **II** result from a condensation reaction between 1-aminoadamantane and Me_3M (M = Ga (**I**), Al (**II**)) in which one methyl group was cleaved from each Me_3M unit in addition to one of the two N−H bonds of each 1-aminoadamantane molecule. Mass balance would demand the reaction given in eqn. (1).

$$\text{Ada} + \text{Me}_3\text{M} \longrightarrow \frac{1}{2}[\text{Me}_2\text{M-Ada}]_2 + \text{Me}-\text{H} \qquad (1)$$

The X-ray crystal structure of compound **I** is given in Fig. 1. Final atomic coordinates are listed in Table 2 while selected bond distances and angles are provided in Table 3. **I** contains two 1-aminoadamantane moieties bridged by a pair of dimethylgallium units constituting a planar and slightly asymmetric Ga_2N_2 four-membered ring; independent Ga−N distances of 2.037(5) and 2.024(5) Å for Ga(1)-N(1) and Ga(1)-N(1A), respectively. The Ga−N−Ga bond angle in the Ga_2N_2 ring is 94.8(2)° while the N−Ga−N bond angle is 85.2(2)°. The Ga−N bond distances observed in **I** may be placed in perspective by examining Table 4 [6] which lists Ga−N bonds for a variety of organogallium compounds. The Ga···Ga contact of 2.990(1) Å does not suggest

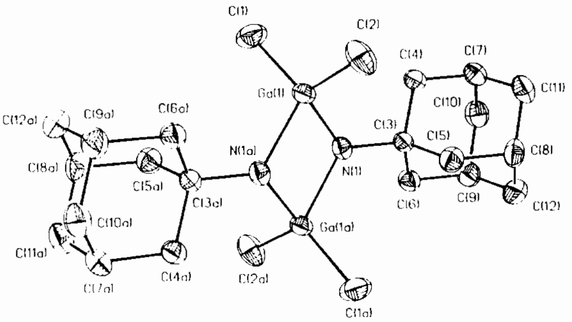

Fig. 1. A view of compound **I** showing the atom-labeling scheme. Thermal ellipsoids show 35% probability levels. Hydrogen atoms have been omitted.

TABLE 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (Å$^2 \times 10^3$) for [Me_2Ga-Ada]₂ (**I**)

Atom	*x*	*y*	*z*	U_{eq}[a]
Ga(1)	4248(1)	3761(1)	5988(1)	37(1)
N(1)	3358(9)	5953(5)	5666(5)	36(2)
C(1)	1926(11)	3131(8)	5690(8)	62(3)
C(2)	5520(13)	2717(8)	7598(7)	63(3)
C(3)	2527(9)	6789(6)	6786(5)	35(2)
C(4)	686(11)	6259(7)	7830(6)	41(2)
C(5)	4279(10)	6625(7)	7529(7)	47(3)
C(6)	1692(11)	8401(6)	6169(6)	44(2)
C(7)	−230(11)	7134(8)	8982(7)	51(3)
C(8)	3405(12)	7504(8)	8674(7)	57(3)
C(9)	806(13)	9277(7)	7323(8)	56(3)
C(10)	−1043(13)	8714(8)	8352(8)	59(3)
C(11)	1568(13)	6944(9)	9709(7)	61(3)
C(12)	2586(15)	9088(8)	8055(9)	64(4)

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

TABLE 3. Selected bond distances (Å) and angles (°) for $[Me_2Ga\text{-}Ada]_2$ (**I**)

Atoms	Distance	Atoms	Distance
Ga(1)–N(1)	2.037(5)	Ga(1)–C(1)	1.974(9)
Ga(1)–C(2)	1.973(7)	Ga(1)–N(1A)	2.024(5)
N(1)–C(3)	1.492(8)		
Atoms	Angle	Atoms	Angle
N(1)–Ga(1)–C(1)	107.2(3)	N(1)–Ga(1)–C(2)	117.4(3)
C(1)–Ga(1)–C(2)	118.4(3)	N(1)–Ga(1)–N(1A)	85.2(2)
C(1)–Ga(1)–N(1A)	115.9(3)	C(2)–Ga(1)–N(1A)	108.4(3)
Ga(1)–N(1)–C(3)	124.2(3)	Ga(1)–N(1)–Ga(1A)	94.8(2)
C(3)–N(1)–Ga(1A)	126.5(4)	Ga(1)–N(1)–H(1)	99.5(40)
H(1)–N(1)–Ga(1A)	97.9(35)		

TABLE 4. Comparisons of Ga–N bond distances (Å) in some organogallium compounds

Compound	Ga–N	Reference
$[(CH_3)_2GaN_2C_3H_3]_2$	1.985(9)	6a
	1.995(9)	
	2.006(8)	
	2.002(9)	
$[(CH_3)_2GaN_2C_5H_7]_2$	1.988(5)	6b
	1.998(5)	
$[(CH_3)_2GaN{=}C(CH_3)_2]_2$	2.000(3)	6c
	1.978(3)	
$[(CH_3)_2Ga(NCH_3)_2CCH_3]_2$	1.981(4)	6d
	1.977(4)	
$[(CH_3)_2Ga(\text{oxamidine})]_2$	1.984(9)	6e
	1.976(10)	
$[Ga(CH_3)_2][Cl_2H_{25}N_2O_4][Ga(CH_3)_3]_2$	2.164(5)	6f
	2.171(6)	
$[Ga(CH_3)_3]_2[C_6H_{12}N_4]$	2.138(9)	6g
$[Ga(CH_3)_3]_4[C_6H_{12}N_4]$	2.139(2)	6g
$[Ga(CH_3)_3]_4[(CH_3)_4[14]\text{ane}N_4]$	2.182(4)	6h
	2.202(4)	
$[Ga(CH_3)]_2[14]\text{ane}N_4[Ga(CH_3)_3]_2$	1.943(5)	6h
	2.126(5)	
	2.014(6)	
	2.003(6)	
$[Ga(CH_2)_2]_2[14]\text{ane}N_4$	2.124(4)	6i
$[Al(CH_3)]_2[14]\text{ane}N_4[Ga(CH_3)_3]_2$	2.136(3)	6i
$[Me_2Ga\text{-}Ada]_2$	2.037(5)	this study
	2.024(5)	this study

significant metal–metal interaction. The mean Ga—C bond distance in the dimethylgallium unit is 1.974(9) Å.

It is important to note that **I** is quite similar to the interesting aminoalane $[(Me_2N)_2Al\{\mu\text{-}N(H)1\text{-}Ad\}]_2$ (**IV**), prepared by an elegant transamination reaction of $[Al(NMe_2)_3]_2$ with 1-aminoadamantane, previously reported by Power and co-workers [7]. As **I** and **IV** are dimeric containing M_2N_2 core fragments, the particular difference between the two compounds is that the coordination sphere of **IV** is saturated by nitrogen atoms while that of **I** has two carbon atoms and two nitrogen atoms. The coordination of the aluminum atoms in **IV** may be regarded as distorted tetrahedral with the angles at the aluminum varying from 85.3(1) to 120.2(2)°. The bond angles about the gallium atoms in **I** are comparable as they range from 85.2(2) to 118.4(3)° for N(1)–Ga(1)–N(1A) and C(1)–Ga(1)–C(2), respectively.

The structure of compound **III** is given in Fig. 2. Final atomic coordinates are listed in Table 5. Selected bond distances and angles are given in Table 6. There are two crystallographically unique mole-

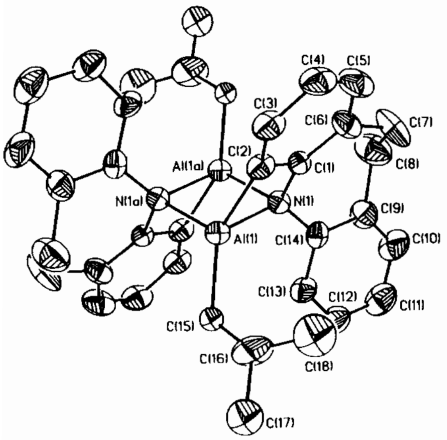

Fig. 2. A view of compound **III** showing the atom-labeling scheme. Thermal ellipsoids show 30% probability levels. Hydrogen atoms have been omitted.

TABLE 5. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($Å^2 \times 10^3$) for [(i-Bu)Al-iminodibenzyl]$_2$ (**III**)

Atom	x	y	z	U_{eq}[a]
Al(1)	5174(2)	331(2)	10884(2)	48(1)
N(1)	3828(5)	881(5)	9934(5)	49(3)
C(1)	2859(6)	478(7)	10890(7)	47(3)
C(2)	3518(8)	−92(7)	11709(7)	59(3)
C(3)	2813(9)	−636(9)	12672(9)	77(4)
C(4)	1475(10)	−593(10)	12776(10)	87(4)
C(5)	857(9)	9(11)	11955(10)	94(4)
C(6)	1530(8)	580(9)	10971(9)	74(4)
C(7)	710(9)	1394(13)	10150(11)	118(5)
C(8)	1418(12)	1608(13)	9074(12)	123(5)
C(9)	2370(9)	2458(10)	8869(9)	82(4)
C(10)	2161(12)	3496(10)	8247(11)	104(4)
C(11)	3129(12)	4204(11)	7878(11)	106(5)
C(12)	4293(11)	3727(10)	8281(11)	95(4)
C(13)	4476(8)	2683(8)	8927(8)	68(3)
C(14)	3511(7)	2002(8)	9297(7)	57(3)
C(15)	6334(22)	1107(22)	11247(22)	71(6)
C(16)	5524(14)	2223(13)	11740(15)	144(5)
C(17)	6874(22)	2655(21)	11859(21)	93(6)
C(18)	4613(26)	2040(27)	12706(27)	127(6)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

cules in the asymmetric unit, each of which is situated about an inversion center. The iminodibenzyl ligand of molecule 2 is disordered; although there is good agreement between corresponding distances and angles for the two molecules, only those of molecule 1 will be discussed. Compound **III** contains two iminodibenzyl moieties bridged by two isobutylaluminum units resulting in a planar Al_2N_2 four-membered ring. The four-membered Al_2N_2 ring is planar and slightly asymmetric with independent Al–N distances of 2.040(7) and 1.949(7) Å for Al(1)–N(1) and Al(1)–N(1A), respectively. The Al−N−Al bond angle in the Al_2N_2 ring is 89.4(2)° while the N−Al−N bond angle is 90.6(2)°. In addition to the Al_2N_2 ring, the most notable feature of compound **III** is the formation of the Al(1)−C(2) bond as a result of *ortho*-metallation of the aromatic ring.

The mechanism leading to **III** is worthy of consideration. It is interesting to consider as a first step condensation of diisobutylaluminum hydride with iminodibenzyl to yield an i-Bu_2Al(iminodibenzyl) monomeric species (with the elimination of H_2). Upon heating, it is reasonable that further condensation took place between two monomeric i-Bu^2Al(iminodibenzyl) units in which an isobutyl group of each aluminum atom and one *ortho*-C-H hydrogen atom (for each iminodibenzyl group) were cleaved thus resulting in **III** (and isobutane elimination). Alternatively, it is also possible that the monomeric i-Bu_2Al(iminodibenzyl) moiety experienced β-elimination resulting in i-Bu(H)Al(iminodibenzyl) fragments (and isobutene elimination). Subsequent dimerization of the i-Bu(H)Al(iminodibenzyl) fragments could also result in **III** (and elimination of H_2).

The most striking point about **III** is that it represents a rare example of an aluminum compound involving *ortho*-metallation of a fused aromatic ring. Principally on the basis of Raman spectroscopy and ^{1}H NMR, Hoberg proposed *ortho*-alumination in $Cl_2AlN(Ph)(\mu\text{-}C_6H_4)Al(Cl)N(Ph)(\mu\text{-}C_6H_4)Al(Cl)\text{-}NPh_2$ (**V**), prepared from heating diethylaluminum diphenylamide to 110 °C [8]. Recently, Power and co-workers reported the first X-ray crystal structure (to the best of our knowledge) of an organoaluminum compound involving *ortho*-metallation. $[\{Me(\mu\text{-}NPh_2)Al\}_2NPh(\mu\text{-}C_6H_4)]$ (**VI**) [7]. Compound **VI** was prepared by reaction of the $[Al(NMe_2)_3]_2$ dimer with diphenylamine in refluxing toluene [7]. The structure of compound **VI** consists of a central $\{AlMe(\mu\text{-}NPh_2)\}_2$ dimeric core in which one of the aluminum atoms is also bonded to a nitrogen atom of a third diphenylamine unit which in turn is linked to the remaining aluminum atom (of the $[Al(NMe_2)_3]_2$ dimer) through an Al−C bond to one of its *ortho*-carbon atoms. The $Al-C_{aromatic}$ bond distance of 1.955(4) Å in compound **VI** compares well with the $Al\text{-}C_{methyl}$ bond distances (1.951(4) Å, mean). The corresponding distances in **III** are: $Al\text{-}C_{aromatic}$, 1.954(8) Å; $Al\text{-}C_{isobutyl}$, 1.980(32) Å.

This study represents the initial efforts of this laboratory regarding the Group 13 chemistry of sterically hindered amines, an intriguing and relatively unexplored region of study. Compounds **I**, **II** and

TABLE 6. Selected bond distances (Å) and angles (°) for [(i-Bu)Al-iminodibenzyl]$_2$ (**III**)

Atoms	Distance	Atoms	Distance
Al(1)-N(1)	2.040(7)	Al(1)-C(1)	2.446(8)
Al(1)-C(2)	1.954(8)	Al(1)-C(15)	1.980(32)
Al(1)-N(1A)	1.949(7)	N(1)-C(1)	1.480(9)
N(1)-C(14)	1.425(10)		
Atoms	Angles	Atoms	Angles
N(1)-Al(1)-C(2)	71.5(3)	N(1)-Al(1)-C(15)	133.1(7)
C(2)-Al(1)-C(15)	130.7(8)	N(1)-Al(1)-N(1A)	90.6(2)
C(2)-Al(1)-N(1A)	107.6(4)	C(15)-Al(1)-N(1A)	112.7(7)
Al(1)-N(1)-C(14)	126.5(6)	C(1)-N(1)-C(14)	121.0(5)
Al(1)-N(1)-Al(1A)	89.4(2)	C(1)-N(1)-Al(1A)	112.9(5)
C(14)-N(1)-Al(1A)	114.3(5)		

III will be used as benchmarks as we continue to explore the Group 13 chemistry of sterically hindered amines.

Supplementary material

Summary of data collection and refinement, tables of bond distances and angles, final fractional coordinates, thermal parameters (18 pages) and a listing of observed and calculated structure factors are available from the authors upon request.

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Note added in proof

The complete single crystal X-ray structure of [Me$_2$Al-Ada]$_2$ has recently been reported (K. M. Waggoner and P. P. Power, *J. Am. Chem. Soc., 113* (1991) 3385).