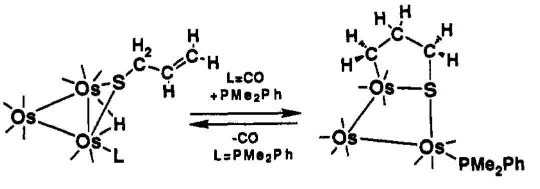
When *cis*-1 was heated to 97 °C under an atmosphere of CO, the isomer 2 and CO adduct 4 were formed. The fragmentation product 3 is also formed together with some $Os_3(CO)_{12}$; see Scheme I. The formation of 2 is formally equivalent to a "β-elimination" reaction, but it must be noted that the mechanism of the transformation has not yet been established. It has been shown that β-elimination reactions involving metallacycles in mononuclear metal complexes are energetically unfavorable.[21] Curiously, we have also observed a facile example of the reverse of this reaction, a phosphine-promoted intramolecular insertion reaction of $Os_3(CO)_{10}[\mu\text{-}SCH_2C(H){=}CH_2](\mu\text{-}H)$ to yield **9** (eq 1).[4] Compound **9** will also engage in a facile "β-elimination" to yield the propenethiolato complex $Os_3(CO)_9(PMe_2Ph)[\mu\text{-}SCH_2C(H){=}CH_2](\mu\text{-}H)$. Studies to try to ascertain the mechanism of these transformations are in progress.



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(1)

When heated to 125 °C, compound **2** was transformed into **3**, the hexanuclear compound **5**, and the trinuclear compound **6**. The mechanism of the fragmentation and formation of **3** is also unclear at this time, and the fate of the missing osmium carbonyl grouping was not established. Compound **5** was prepared previously by the thermal degradation of $Os_3(CO)_{10}(\mu\text{-}SCH_2Ph)(\mu\text{-}H)$.[11] That reaction was shown to proceed by a homolysis of the C–S bond. A similar process is anticipated for the transformation of **2** to **5**. The fate of the diphenylpropenyl grouping was not established in this reaction. Compound **6** was obtained previously by the photoinduced elimination of benzene from $Os_3(CO)_{10}(\mu\text{-}SPh)(\mu\text{-}H)$.[12] It seems reasonable to expect that a similar thermal reaction of **2** has occurred in this case with the formation of diphenylpropene, but due to the conditions and small amounts of material involved in this reaction, the formation of diphenylpropene was not established.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this reseach.

Registry No. *cis*-1, 137432-20-3; *trans*-1, 137332-96-8; **2**, 137332-97-9; **3**, 137332-98-0; **4**, 137332-99-1; **5**, 137333-00-7; **6**, 88746-45-6; *trans*-2,4-DPT, 24609-88-9; $Os_3(CO)_{10}(NCMe)_2$, 61817-93-4.

Supplementary Material Available: Tables of phenyl ring and hydrogen atom positional parameters and anisotropic thermal parameters (22 pages); tables of observed and calculated structure factor amplitudes (76 pages). Ordering information is given on any current masthead page.

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Interactions of 1,4-Diazabicyclo[2.2.2]octane with Group III Metal Trimethyls: Structures of $Me_3M{\cdot}N(C_2H_4)_3N{\cdot}MMe_3$ (M = Al, Ga)

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Received May 7, 1991

The reactions of $(Me_3Al)_2$ and $Me_3Ga{\cdot}OEt_2$ with 1,4-diazabicyclo[2.2.2]octane (dabco) gave the Lewis acid–base complexes $Me_3M{\cdot}N(C_2H_4)_3N{\cdot}MMe_3$ (M = Al (1), Ga (2)). These complexes are discrete 2:1 adducts of the metal trialkyl to dabco. ^1H NMR spectra revealed an upfield shift of both the dabco methylene protons and the metal alkyl protons on coordination of the Lewis base. Methyl proton resonances move downfield as the metal center is varied from Al to Ga to In (comparisons being made with the previously reported complex trimethylindium 1,4-diazabicyclo[2.2.2]octane).[1] Complexes 1 and 2 were also characterized by IR spectroscopy, where it was found that the vibrational frequency for the M–C asymmetric stretching mode is lower for the adduct species than for the parent metal trimethyls. The molecular structures of 1 and 2, which are isostructural, were determined by X-ray crystallographic analysis. Crystal data for 1: trigonal, $R\bar{3}m$, $a = b = 11.223$ (2) Å, $c = 22.757$ (8) Å, $\alpha = \beta = 90°$, $\gamma = 120°$, $Z = 6$, $R = 0.073$. Crystal data for 2: trigonal, $R\bar{3}m$, $a = b = 11.231$ (4) Å, $c = 22.693$ (13) Å, $\alpha = \beta = 90°$, $\gamma = 120°$, $Z = 6$, $R = 0.043$. The molecules have $3m$ symmetry, and although the Al–C and Ga–C bond lengths were almost equal, the Al–N bond at 2.066 (8) Å was significantly shorter than the Ga–N bond at 2.159 (9) Å. Notable differences in volatility, solubility, and reactivity between 1 and 2 are discussed.

Introduction

The development of high-purity precursors for the preparation of III/V semiconductor materials has been of critical importance for successful device fabrication.[2] Previously the realization of the full potential of techniques such as metalloorganic chemical vapor deposition (MOCVD) and organometallic vapor-phase epitaxy (OMVPE) has been delayed by the inadequacy of the source materials available. The advent of adduct pre-

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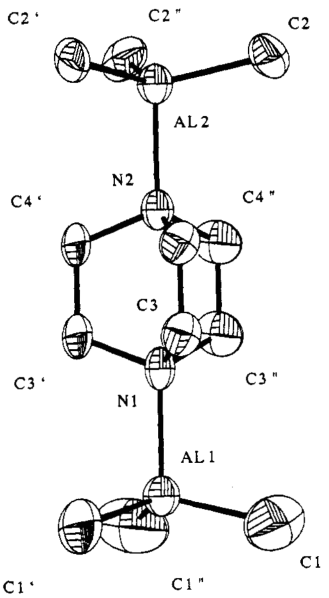

Figure 1. Structure of $Me_3Al\cdot N(C_2H_4)_3N\cdot AlMe_3$ (1). Thermal ellipsoids are drawn at the 50% level, and hydrogen atoms are omitted for clarity.

cursors[3-7] as well as of intramolecularly stabilized group III alkyls[8] has resulted in such major improvements in purity that the production of microwave, electronic, and optoelectronic devices is now almost routine.[9]

The synthesis and characterization of these adduct precursors have been major research interests of this group for a number of years. Recently the synthesis and structure of the linear polymer trimethylindium 1,4-diazabicyclo[2.2.2]octane (dabco) was reported.[1] It is a comparatively air-stable adduct of trimethylindium. The stability was attributed to the fact that the indium atom is 5-coordinate and is very effectively shielded from attack by molecular oxygen by the two nitrogen atoms belonging to two separate molecules of dabco and by the three methyl groups. The unusually high stability imparted to trimethylindium by dabco was in marked contrast to the reactivity of the 2:1 adduct formed by trimethylgallium,[10] and accordingly we have carried out an X-ray crystal structure determination of 2, which we now report together with the details of the synthesis, characterization, and X-ray crystal structure of the aluminum complex 1. Both compounds are sources of the pure metal,[11,12] and the aluminum precursor in particular should provide a means of forming contacts on Si-based devices.[13-16]

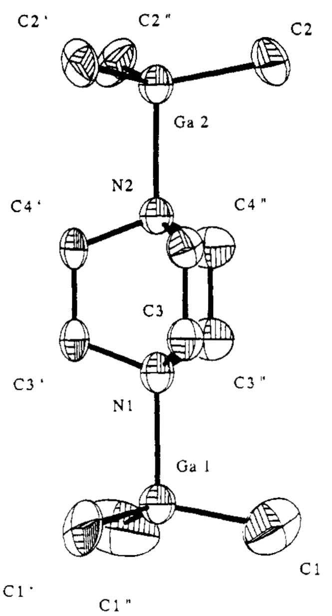

Figure 2. Structure of $Me_3Ga\cdot N(C_2H_4)_3N\cdot GaMe_3$ (2). Thermal ellipsoids are drawn at the 50% level, and hydrogen atoms are omitted for clarity.

Table I. Selected Bond Lengths (Å) and Bond Angles (deg)[a]

1		2	
C(1)-Al(1)	1.954 (11)	C(1)-Ga(1)	1.959 (12)
N(1)-Al(1)	2.065 (8)	N(1)-Ga(1)	2.154 (9)
C(2)-Al(2)	1.968 (7)	C(2)-Ga(2)	1.979 (9)
N(2)-Al(2)	2.066 (8)	N(2)-Ga(2)	2.164 (9)
C(3)-N(1)	1.492 (6)	C(3)-N(1)	1.485 (7)
C(4)-N(2)	1.488 (6)	C(4)-N(2)	1.486 (7)
C(4)-C(3)	1.502 (9)	C(4)-C(3)	1.510 (11)
N(1)-Al(1)-C(1)	104.0 (4)	N(1)-Ga(1)-C(1)	101.4 (4)
N(2)-Al(2)-C(2)	102.2 (3)	N(2)-Ga(2)-C(2)	100.5 (3)
C(1)-Al(1)-C(1a)	114.3 (3)	C(1)-Ga(1)-C(1a)	116.2 (3)
C(2)-Al(2)-C(2a)	115.7 (2)	C(2)-Ga(2)-C(2a)	116.8 (2)
C(3)-N(1)-Al(1)	111.6 (5)	C(3)-N(1)-Ga(1)	111.4 (5)
C(4)-N(2)-Al(2)	111.6 (4)	C(4)-N(2)-Ga(2)	111.0 (5)
C(4)-C(3)-N(1)	111.5 (6)	C(4)-C(3)-N(1)	111.6 (7)
C(3)-N(1)-C(3a)	107.2 (5)	C(3)-N(1)-C(3a)	107.5 (5)
C(3)-C(4)-N(2)	111.7 (6)	C(3)-C(4)-N(2)	110.8 (7)
C(4)-N(2)-C(4a)	107.3 (5)	C(4)-N(2)-C(4a)	107.9 (5)

[a] Key to symmetry operations relating designated atoms to reference atoms at x, y, z: (a) $y - x, -x, z$; $-y, x - y, z$.

Results and Discussion

Preparation and Structures of 1 and 2. The reaction of dabco with trimethylaluminum and with trimethylgallium etherate gave the Lewis acid-base adduct compounds $Me_3Al\cdot N(C_2H_4)_3N\cdot AlMe_3$ (1) and $Me_3Ga\cdot N(C_2H_4)_3N\cdot GaMe_3$ (2) (eqs 1 and 2). The products were

$$(Me_3Al)_2 + N(C_2H_4)_3N \rightarrow Me_3Al\cdot N(C_2H_4)_3N\cdot AlMe_3 \quad (1)$$

$$2Me_3Ga\cdot OEt_2 + N(C_2H_4)_3N \rightarrow Me_3Ga\cdot N(C_2H_4)_3N\cdot GaMe_3 + 2Et_2O \quad (2)$$

recrystallized from benzene and gave transparent, colorless crystals. These compounds are much less air sensitive than the parent metal trimethyls.

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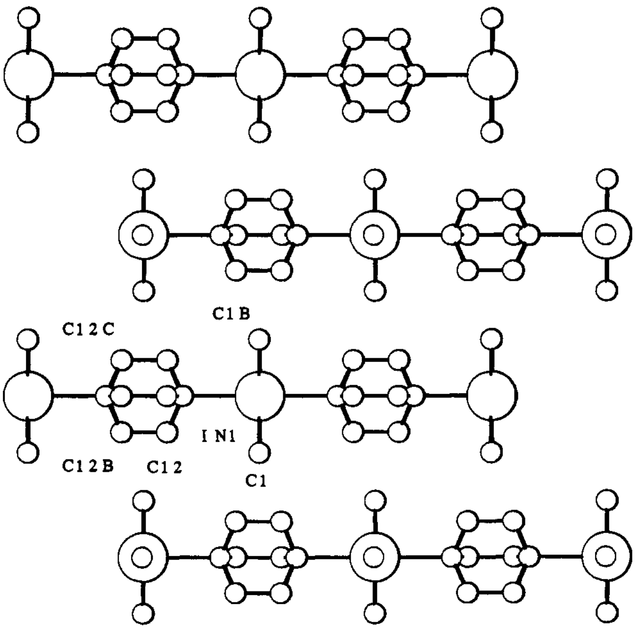

Figure 3. Packing diagram of $[Me_3In{\cdot}N(C_2H_4)_3N]_n$.[1]

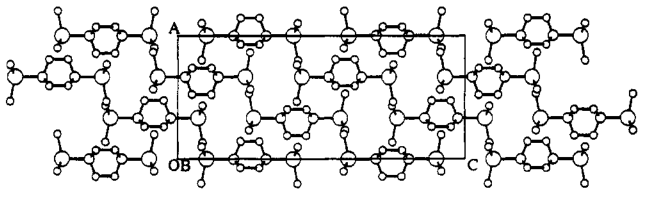

Figure 4. Packing diagram for 2:1 adducts of Me_3Al and Me_3Ga to dabco.

Molecular structures of 1 and 2 have been determined by X-ray crystallography. The compounds are isostructural, and the structures of the adducts are shown in Figures 1 and 2. Selected bond lengths and angles are given in Table I.

The structures consist of discrete 2:1 molecular adducts of trimethylaluminum and trimethylgallium bonded to dabco. Both compounds contain 4-coordinate metal atoms about which the stereochemistry can be described as distorted tetrahedral, with the distortion being toward a trigonal pyramid. The base of the pyramid is defined by the three metal-methyl carbon atoms. The C–M–N bond angles are consistently some 8–9° smaller than the tetrahedral angle. The metal–carbon bond lengths are within the expected range for metal–carbon single bonds. The average metal–nitrogen bond length for compound 1 is 2.066 (8) Å and is 2.159 (9) Å for compound 2. Thus, it is noteworthy that whereas the Al–C and Ga–C bond distances are very close in value, the Al–N bonds are significantly shorter (9σ) than the Ga–N bonds, implying stronger bonding of dabco to $AlMe_3$ than to $GaMe_3$.

Previous work has revealed a similar value of Al–N (2.06 (1) Å) in $Me_3Al{\cdot}N(CH_2CH_2)_3CH$[17] compared to the value in compound 1, which is not surprising since the quinuclidine ligand has electronic and steric properties similar to those of dabco. Unfortunately there are no other trimethylgallium complexes comparable to compound 2; for example Ga–N distances have been reported[10] for $Me_3Ga{\cdot}NH(C_6H_{11})_2$ (Ga–N = 2.151 (6) Å) and $Me_3Ga{\cdot}\overline{NHCHMe_2(CH_2)_3C}HMe$ (Ga–N = 2.199 (5) Å) but these Lewis base ligands are clearly different from dabco.

Table II. ^1H **NMR Data for the Group III Trimethyls and Their Adducts with dabco**[a]

compd	$\delta(Me_3M)$, ppm	$\delta(N\text{–}CH_2)$, ppm
Me_3Al[b]	–0.35	
Me_3Ga[b]	–0.10	
Me_3In[b]	–0.18	
$N(C_2H_4)_3N$		2.47
$Me_3Al{\cdot}N(C_2H_4)_3N{\cdot}AlMe_3$ (1)	–0.62	2.03
$Me_3Ga{\cdot}N(C_2H_4)_3N{\cdot}GaMe_3$ (2)	–0.35	1.98
$[Me_3In{\cdot}N(C_2H_4)_3N]_n$	–0.15	2.03

[a] δ values relative to TMS; solvent C_6D_6. [b] Reference 6.

It is of interest to compare the structures of 1 and 2 with that of Me_3In(dabco) (Figure 3). The latter compound was found to be a 1:1 linear polymer with 5-coordinate indium atoms. The stereochemistry about indium is trigonal bipyramidal, and the In–C bond distances are unremarkable, but the In–N distance is longer than would be expected for a regular dative interaction. This may be due to the steric repulsion that would exist between the methyl groups on the indium and the methylene groups of the dabco. It may also be due to the hybridization of the indium atomic orbitals. Usually 1:1 adducts of the group III trialkyls with compounds that are Lewis bases involve donation of electron density by the base donor atom into an sp^3 hybrid orbital of the metal. This is not the case in the 5-coordinate indium adduct. Here the molecular orbitals will involve contributions from the $5p_z$ and $5d_{z^2}$ orbitals of indium and donation from the nitrogen lone pairs would be expected to be weaker than into an orbital with less "s" character.

The indium atom is 5-coordinate presumably because it is large enough to accommodate the steric bulk associated with the coordination of three methyl groups and the two nitrogen atoms of dabco. The covalent radius of indium (1.497 Å) is considerably larger than the covalent radii for Al (1.248 Å) and Ga (1.245 Å), which are almost equal.[18] The X-ray crystal structures show that these atoms are clearly too small to accommodate the steric interactions associated with them being 5-coordinate.

NMR Spectra. The ^1H NMR spectra of the adduct compounds (Table II) were characterized by two sharp singlet resonances, one due to the methylene protons of dabco and the other due to the methyl protons of the metal. The relative intensities of the two sets of resonances agree within experimental error with what would be expected for 2:1 adduct compounds of trimethylaluminum and trimethylgallium with dabco.

In each case an upfield shift of the methylene protons on coordination of the Lewis base relative to the shift for free dabco was observed. This is consistent with the results reported for the triethylamine adduct of trimethylindium.[19] The trend is surprising, however, since coordination is usually thought to involve the shifting of electron density to the metal, leaving the ligand protons less shielded. Nevertheless, it must be borne in mind that the interpretation of chemical shifts in metal complexes is fraught with difficulty.

The chemical shifts of the metal methyl protons behave differently upon coordination, depending on the metal center present. The methyl proton resonances undergo an upfield shift relative to the parent metal trimethyl when the metal center is Al or Ga, but a slight downfield shift is given by the indium complex. Coordination of a Lewis

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base would be expected to increase the electron density on the metal and hence the shielding of the alkyl protons. This would result in an upfield shift, as observed for Al and Ga but not for indium. It may be that the different coordination of the indium atom in the adduct is responsible for this anomalous observation. Variable-temperature ^{1}H NMR spectra were run from +20 to −40 °C in toluene-d_8 to determine whether or not a dissociative equilibrium of these adduct compounds was present in solution. No evidence for such a process was found.

The ^{13}C{^{1}H} spectrum of compound 1 was also obtained. It was characterized by two singlet resonances, one at 2.03 ppm due to the trimethylaluminum carbon atoms and the other at 45.17 ppm due to the methylene carbons of dabco.

IR Spectra. Compounds 1 and 2 were characterized by infrared spectroscopy as Nujol mulls. The values for the asymmetric stretching mode of the M–C bonds were 595 and 540 cm^{-1} for compounds 1 and 2, respectively. These assignments were based on a comparison of the IR spectra of the pure Lewis acids and base with those of adducts. In this way adduct formation was correlated with both shifts in peak energies and with the disappearance of some peaks and the appearance of new peaks. These assignments are only tentative, however, in the absence of full coordinate analyses.

In general, as we move from one member of the series to the next the fundamental vibrational frequencies will be affected by two factors: (1) the changes of force constants and (2) the change of mass of the central atoms. For the same force constant an increase in mass of the central atom will cause a decrease in vibrational frequency. This is consistent with the observed decrease in frequency when the metal center is changed from Al to Ga in compounds 1 and 2, respectively. The frequency of this mode when the metal center is indium ($\nu_{\text{In–C}}$ = 483 cm^{-1}) is lower than both the Al–C and Ga–C frequencies, as would be expected on the basis of both the greater mass of the indium atom and the lower force constant for trimethylindium as opposed to that of trimethylgallium ($K_{\text{In–C}} = 1.93 \times 10^5$ dyn cm^{-1}; cf. $K_{\text{Ga–C}} = 2.41 \times 10^5$ dyn cm^{-1}).[20] Trimethylaluminum is a dimer in the gas phase and will not be considered in the discussion since this mode is absent in its IR spectrum.

It is interesting to note that the vibrational frequency for this mode is significantly lower for the adduct compounds of trimethylgallium and indium than for the parent trimethylmetal species ($\nu_{\text{M–C}}$(free trimethylgallium) = 576 cm^{-1}, cf. $\nu_{\text{M–C}}$(bis(trimethylgallium)–dabco) = 540 cm^{-1}; $\nu_{\text{M–C}}$(free trimethylindium) = 500 cm^{-1}, cf. $\nu_{\text{M–C}}$(trimethylindium–dabco) = 483 cm^{-1}). This may be due to a reduction in the polarity of the metal–carbon bonds as electron density is transferred from the Lewis base to the Lewis acid and/or the differing hybridizations of the atomic orbitals on the metal in the adducts as compared to those in the free metal trimethyls. Both these effects will result in reduction of the M–C force constants, lowering the frequency of vibration. For similar reasons the methyl rocking modes of free trimethylindium and trimethylgallium (725 and 766 cm^{-1}, respectively)[20] are also reduced in frequency and move to broad, unsymmetrical peaks at 679 and 710 cm^{-1}, respectively.

Also apparent in the IR spectra of compounds 1 and 2 are peaks at 519 and 512 cm^{-1}, respectively, due to the symmetrical stretch of the metal–carbon bonds. In both cases the methyl groups on the metal atoms are forced close together in an "umbrella-like" fashion due to the steric repulsion from the methylene groups of the diamine. Since the MMe$_3$ half of the adduct has C_{3v} symmetry, the symmetrical M–C stretches in these compounds are IR active. This is not the case of trimethylindium–dabco, however. In this compound the In–C$_3$ unit is planar and consequently this mode is not IR active.

Stability of the dabco Complexes. The unusual stability of trimethylindium–dabco promoted this investigation of the products formed when dabco reacts with trimethylaluminum and trimethylgallium. Although the aluminum and gallium species are both discrete 2:1 adducts of the trialkyl to dabco, their behavior is different in a number of ways.

(i) Volatility. $Me_3Ga{\cdot}N(C_2H_4)_3N{\cdot}GaMe_3$ (2) sublimes readily at 60 °C and 2 × 10^{-2} Torr, whereas $Me_3Al{\cdot}N(C_2H_4)_3N{\cdot}AlMe_3$ (1) does not sublime even at 200 °C and 10^{-2} Torr.

(ii) Solubility. While compound 2 is soluble in most common organic solvents, compound 1 is only slowly and sparingly soluble.

(iii) Reactivity. Compound 2, although of markedly reduced reactivity compared with free trimethylgallium, is hydrolyzed in air within hours of exposure. In contrast, compound 1 is stable in air for up to 3 weeks. This adduct is remarkably stable, considering that we have a 4-coordinate aluminum center present.

The gallium adduct is known to sublime unchanged, and both the gallium and aluminum species are known to dissolve as 2:1 adducts. However, it was noted earlier that the Al–N bond length in 1 is significantly shorter than the Ga–N bond in 2, suggesting weaker coordination of trimethylgallium to dabco. Thus, the volatility of 2 may be due to dissociation into Me$_3$Ga and dabco on heating rather than vaporization of 2 intact as a molecular complex. This could also explain the greater air sensitivity of 2 compared with that of 1. It is also worth pointing out that the trimethyl complexes of aluminum, gallium, and indium also show interesting differences. For example the aluminum compound is dimeric (Al$_2$Me$_6$) with 4-coordinated aluminum, whereas the gallium compound is monomeric, reflecting a lower tendency to become 4-coordinated, and the indium trimethyl is a crystalline solid containing tetrameric units (In$_4$Me$_{12}$) with 5-coordinated indium. Thus, the chemistry of their dabco complexes is not excessively surprising.

Experimental Section

Microanalyses were performed by the Microanalytical Laboratory, University College, London. Melting points were determined in sealed capillaries. IR spectra (4000–200 cm^{-1}) were recorded on a Perkin-Elmer 577 FT-IR spectrometer as Nujol mulls (CsI). ^{1}H NMR spectra (C$_6$D$_6$) were recorded on a Bruker WP 80 FT instrument relative to SiMe$_4$, while ^{13}C{^{1}H} NMR spectra were recorded on a Bruker WP 250 FT instrument relative to the same reference. Mass spectra were recorded with a Kratos MS 50 TC instrument. Solvents were dried and distilled before use. (AlMe$_3$)$_2$, dabco, and GaCl$_3$ were obtained from Aldrich. All manipulations were carried out using a vacuum line and Schlenk techniques. dabco was purified by sublimation before use.

$Me_3AlN{\cdot}(C_2H_4)_3N{\cdot}AlMe_3$ (1). A solution of (AlMe$_3$)$_2$ in Et$_2$O (21.1 mL, 2.3 M) was added via transfer tube to a solution of dabco (10.9 g, 97 mmol) in Et$_2$O (80 mL), immediately giving a white precipitate. The mixture was refluxed for 1$^1/_2$ h, after which time the solvent was removed in vacuo and excess dabco was removed by sublimation (20 °C, 10^{-2} Torr). The white crystalline product was recrystallized from refluxing benzene which was allowed to cool to 20 °C: yield ca. 90% based on (AlMe$_3$)$_2$; mp 230 °C dec. Anal. Calcd for C$_{12}$H$_{30}$Al$_2$N$_2$: C, 56.21; H, 11.82; N, 10.93. Found: C, 55.04; H, 11.19; N, 10.57. IR: 1461 (s), 1383 (m), 1328 (s), 1264 (m), 1200 (s), 1187 (s), 1089 (m), 1053 (s), 1018 (s), 927 (s), 842

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Table III. Crystal Data, Details of Intensity Measurements, and Structure Refinement Data for Compounds 1 and 2

	1	2
formula	$Al_2N_2C_{12}H_{30}$	$Ga_2N_2C_{12}H_{30}$
M_r	256.347	341.830
cryst syst	trigonal (rhombohedral)	trigonal (rhombohedral)
space group	$R\bar{3}m$ (hexagonal axes)	$R\bar{3}m$ (hexagonal axes)
a, Å	11.223 (2)	11.231 (4)
b, Å	11.223 (2)	11.231 (4)
c, Å	22.757 (8)	22.693 (13)
α, deg	90	90
β, deg	90	90
γ, deg	120	120
V, Å^3	2482.35	2478.90
Z	6	6
D_c, g cm^{-3}	1.029	1.374
$F(000)$	852	1068
μ, cm^{-1}	1.535	32.331
θ range, deg	1.5, 25	1.5, 25
hkl range	0–13, −13 to +13, 0–27	−13 to +13, −13 to +13, 0–27
total no. of rflns	1613	3025
no. of unique rflns	571	596
significance test	$F_o > 3\sigma(F_o)$	$F_o > 3\sigma(F_o)$
no. of obsd rflns	425	425
no. of refined params	59	59
function minimized	$\sum w(\|F_o\| - \|F_c\|)^2$	$\sum w(\|F_o\| - \|F_c\|)^2$
weighting scheme parameter g in $w = 1/[\sigma^2(F) + gF^2]$	0.000 12	0.000012
final R	0.0728	0.0431
final R_w	0.0583	0.0329

Table IV. Fractional Atomic Coordinates (×10^4) for Compound 1

atom	x	y	z
Al(1)	0^a	0^a	−1035 (1)
Al(2)	0^a	0^a	−3992 (1)
C(1)	1951 (10)	975 (5)	−826 (4)
C(2)	1979 (6)	989 (3)	−4174 (3)
N(1)	0^a	0^a	−1942 (3)
N(2)	0^a	0^a	−3084 (3)
C(3)	714 (3)	1427 (5)	−2183 (3)
C(4)	712 (2)	1423 (5)	−2844 (3)

[a] Invariant parameters.

Table V. Fractional Atomic Coordinates (×10^4) for Compound 2

atom	x	y	z
Ga(1)	0^a	0^a	−991 (1)
Ga(2)	0^a	0^a	−4032 (1)
C(1)	1975 (11)	987 (5)	−821 (5)
C(2)	2000 (7)	1000 (4)	−4191 (3)
N(1)	0^a	0^a	−1940 (3)
N(2)	0^a	0^a	−3079 (3)
C(3)	711 (3)	1422 (6)	−2179 (3)
C(4)	713 (3)	1426 (6)	−2844 (3)

[a] Invariant parameters.

(s), 814 (s), 700 (s, br), 632 (s), 595 (m), 519 (m), 425 (w) cm^{-1}. ^{1}H NMR: δ 2.03 (12 H, C_2H_4), −0.62 (18 H, Al–CH_3). ^{13}C NMR: δ 45.17 (6 C, C_2H_4), 2.03 (6 C, Al–CH_3).

$Me_3Ga\cdot N(C_2H_4)_3N\cdot GaMe_3$ (2) was synthesized as described previously.[10] X-ray-quality crystals were grown by recrystallization from benzene.

Crystallography. All measurements were made on samples mounted in glass capillaries using a CAD4 diffractometer operating in the $\omega/2\theta$ scan mode with graphite-monochromated Mo Kα radiation as described previously.[21] The structures were solved via standard heavy-atom procedures and refined using full-matrix least-squares methods[22] with scattering factors calculated from data from ref 23. All non-hydrogen atoms were refined with anisotropic displacement factors; hydrogen atoms were identified in difference maps and included with isotropic displacement factors. Crystal data, details of the intensity measurements, and refinement data are given in Table III. Positional parameters are given in Tables IV and V. Tables of H atom coordinates, full lists of bond lengths and angles, anisotropic displacement factor coefficients, and F_o/F_c values are included in the supplementary material.

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Supplementary Material Available: Listings of anisotropic temperature factors for non-hydrogen atoms, fractional atomic coordinates and isotropic temperature factors for hydrogen atoms, all bond lengths and angles, and selected nonbonded distances for 1 and 2 (6 pages); listings of F_o/F_c values (6 pages). Ordering information is given on any current masthead page.

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