

Preparation and characterization of mixed alkyl amido complexes of gallium

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Abstract

A series of primary amido gallium alkyl complexes that includes a base free dimer, [¹Bu₂Ga(μ-N(H)¹Bu)]₂ (1), Lewis base stabilized monomeric complexes, ⁿBu₂Ga(N(H)¹Bu)(THF) (2) and ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]py (3) and an anionic complex, ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]₂[Li(Et₂O)] (4) is reported. Complex 1 crystallizes in the triclinic space group *P*-1 (*a* = 10.265(5) Å, *b* = 15.752(6) Å, *c* = 8.932(4) Å, α = 90.32(3)°, β = 105.61(3)°, γ = 88.24(4)°) with two molecules, each residing on an inversion center, in the asymmetric unit. Structural analysis revealed a planar Ga₂N₂ core with both the bridging N and the Ga centers in distorted tetrahedral environments (Ga–C distances 2.052(3)–2.065(3) Å and Ga–N distances 2.060(3)–2.069(3) Å). The use of excess amido ligand allowed the isolation and crystallization of 4. Complex 4 crystallized in the monoclinic space group *P*2₁/*n* (*a* = 8.666(2) Å, *b* = 22.305(3) Å, *c* = 15.570(3) Å, β = 103.47(2)°) with *Z* = 4. The pseudotetrahedral gallium center has a coordination sphere composed of two amido ligands (Ga–N1 = 2.011(8) Å, Ga–N2 = 2.006(7) Å), and two ⁿBu ligands (Ga–C17 = 2.002(9) Å, Ga–C21 = 1.985(12) Å). A bridging interaction of the lithium cation with the lone pair of electrons on each of amido nitrogen atoms generates a molecular core which is made up of a planar Ga–N1–Li–N2 distorted square (N1–Ga1–N2 94.4°, Ga1–N2–Li1 86.2°, N1–Li1–N2 92.2°, Ga1–N1–Li1 87.1°).

Keywords: Gallium; Amines; Alkyls

1. Introduction

The motivation for preparation of compounds containing gallium and indium in combination with Group 15 elements (N, P, As) stems largely from the appearance of these elements in a variety of electronic materials. For example, the current method for preparation of GaN films, a potentially important wide band-gap semiconductor, relies on the reaction of trialkyl gallium with ammonia to eliminate three equivalents of alkane and form the solid film [1–3]. In addition, primary amido complexes of aluminum and gallium have received recent attention as potential sources of imido alanes and gallanes via elimination of alkanes [4–11]. The tendency of Group 13 amido complexes to oligomerize and thus increase the coordination of both the metal and the nitrogen has been observed for both aluminum and gallium and is expected to also manifest itself for imido complexes of these metals. The aggregation of these

species may divulge useful information about the mechanism and formation of GaN from molecular precursors.

With these facts in mind, we have focused on the preparation of alkyl gallium amido compounds bearing primary amido groups. Since steric effects are likely to play an important role in the level of aggregation of these species, we have chosen both alkyl groups (ⁿBu and ¹Bu) and amido groups (¹BuNH and [2,6-Me₂C₆H₃]NH) of intermediate steric bulk. We report the synthesis and characterization of a series of primary amido gallium alkyl complexes that includes a base free dimer, [¹Bu₂Ga(μ-N(H)¹Bu)]₂ (1), Lewis base stabilized monomeric complexes, ⁿBu₂Ga(N(H)¹Bu)(THF) (2) and ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]py (3), and an anionic complex, ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]₂[Li(Et₂O)] (4).

In an effort to control the product distribution, our synthetic strategy is based on the metathetical reaction of R₂GaCl precursors with the lithium salt of primary amines rather than the direct reaction of gallium alkyls with amines [3–9]. Thus, the reaction of ¹Bu₂GaCl [12] and Li(NH)¹Bu resulted in the isolation of 1 as colorless

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crystals in 49% yield (Scheme 1). The ^1H NMR spectrum of this complex indicated the appropriate ratio of ^tBu groups for the formula $[\text{}^t\text{Bu}_2\text{Ga}(\mu\text{-N}(\text{H})^t\text{Bu})]_n$ along with a broad singlet at 2.12 ppm which was assigned to the amido proton. However, there was no evidence of ν_{NH} in the IR spectrum for this complex. This com-

pound does not coordinate ancillary ligands such as THF or pyridine and crystals of **1** are stable in air for several hours; these features are likely due to the presence of the bulky substituents.

In order to confirm the nuclearity of complex **1**, a single crystal X-ray diffraction study was performed

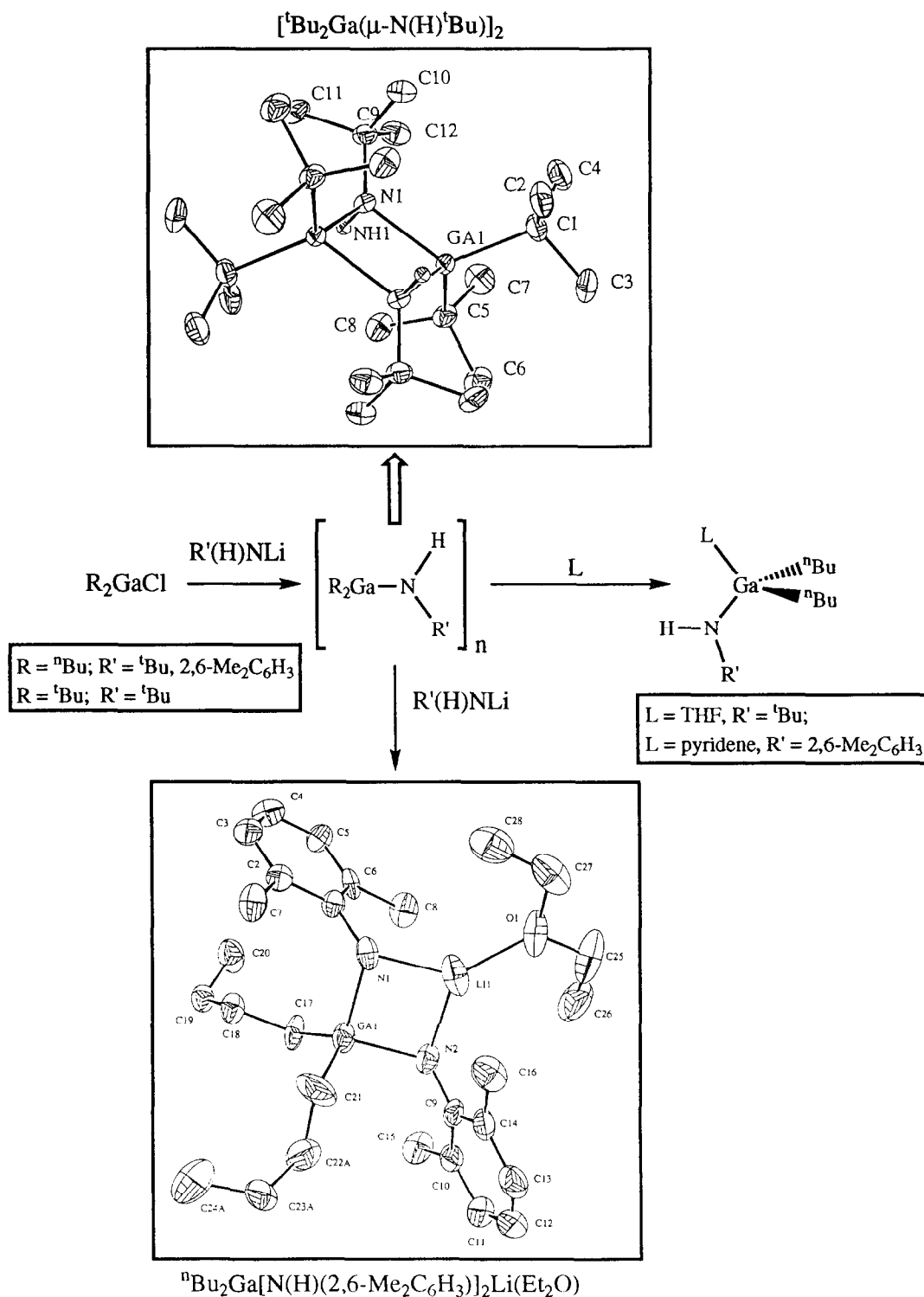


Table 1
Selected atomic bond distances (Å) for compounds 1 and 4

Compound 1		Compound 4	
Ga1–N1	2.068(3)	Ga1–N1	2.011(8)
Ga1–N1a	2.060(3)	Ga2–N2	2.006(7)
Ga1–C1	2.065(3)	Ga1–C17	2.002(9)
Ga1–C5	2.055(3)	Ga1–C21	1.985(1)
Ga2–N2	2.062(3)	Ga1–Li1	2.782(2)
Ga2–N2b	2.069(3)	O1–Li1	1.977(2)
Ga2–C13	2.052(3)	N1–C1	1.425(1)
Ga2–C17	2.054(3)	N1–Li1	2.027(2)
N1–C9	1.511(4)	N2–C9	1.388(1)
N2–C21	1.503(4)	N2–Li1	2.06(2)
C1–C2	1.522(6)	C1–C2	1.414(1)
C1–C3	1.548(6)	C1–C6	1.410(1)
C1–C4	1.539(5)	C2–C3	1.384(1)
C5–C6	1.537(5)	C2–C7	1.506(1)
C5–C7	1.530(5)	C3–C4	1.390(1)
C5–C8	1.531(5)	C4–C5	1.391(1)
C9–C10	1.528(5)	C5–C6	1.402(1)
C9–C11	1.532(5)	C6–C8	1.519(1)
C9–C12	1.522(5)	C17–C18	1.529(1)

and it confirmed the dimeric nature of this primary amido-bridged complex: $[\text{}^t\text{Bu}_2\text{Ga}(\mu\text{-N}(\text{H})^t\text{Bu})_2]$ (Fig. 1). The complex crystallizes in the triclinic space group *P*-1 with two molecules in the asymmetric unit and no anomalously short intermolecular contacts. Selected bond distances and angles for the two molecules are presented in Tables 1 and 2, respectively. The structural analysis revealed a planar Ga_2N_2 core, with each of the molecules residing on an inversion center. The angles within this core are quite strained. For example, the angle N1–Ga1–N1a is 81.7° and that for Ga1–N1–Ga1a is 98.3° (the sum of the internal angles for both molecules is 360°). Both the bridging N and the Ga centers are in distorted tetrahedral environments with a range of bond angles. The structural parameters including Ga–C (range = 2.052(3)–2.065(3) Å) and Ga–N (range = 2.060(3)–2.069(3) Å) distances are in accord

Table 2
Selected atomic bond angles (deg) for compounds 1 and 4

Compound 1		Compound 4	
N1–Ga1–N1a	81.7(1)	N1–Ga1–N2	94.4(3)
N1–Ga1–C1	120.1(1)	N1–Ga1–C17	111.2(4)
N1–Ga1–C5	108.2(1)	N1–Ga1–C21	109.9(4)
N1a–Ga1–C1	108.7(1)	N2–Ga1–C17	106.3(3)
N1a–Ga1–C5	120.2(1)	N2–Ga1–C21	112.0(5)
C1–Ga1–C5	114.5(1)	C17–Ga–C21	120.0(5)
Ga1–N1–Ga1a	98.3(1)	Ga1–N1–C1	115.9(5)
Ga1–N1–C9	125.9(2)	Ga1–N1–Li1	87.1(7)
Ga1a–N1–C9	124.9(2)	Ga1–N2–C9	120.1(6)
Ga1–C1–C2	112.9(2)	Ga1–N2–Li1	86.2(6)
Ga1–C1–C3	107.6(2)	Ga1–Li1–N1	46.2(4)
Ga1–C1–C4	113.8(3)	Ga1–Li1–N2	46.0(4)
Ga1–C5–C6	114.2(2)	Ga1–Li1–O1	178.0(1)
Ga1–C5–C7	107.1(2)	Ga1–C17–C18	114.2(6)
Ga1–C5–C8	113.4(2)	O1–Li1–N1	135.4(1)
N2–Ga2–N2b	82.3(1)	O1–Li1–N2	132.4(1)
N2–Ga2–C13	120.7(1)		
N2–Ga2–C17	107.5(1)		
N2b–Ga2–C13	108.8(1)		
N2b–Ga2–C17	120.2(1)		
C13–Ga2–C17	114.1(1)		
Ga2–N2Ga2b	97.7(1)		
Ga2–N2–C21	126.2(2)		
Ga2b–N2–C21	124.2(2)		
Ga2–C17–C18	113.3(2)		
Ga2–C17–C19	115.3(2)		
Ga2–C17–C20	107.0(2)		

with those observed for similar compounds in the literature [9–11].

A similar reaction of ${}^t\text{Bu}_2\text{GaCl}$ [13] and $\text{LiN}(\text{H})^t\text{Bu}$ gave an oily, non-crystalline solid. This material could be crystallized as colorless crystals in approximately 40% overall yield through the addition of coordinating species, such as THF, to yield a monomeric primary amido dialkyl species, ${}^t\text{Bu}_2\text{Ga}(\text{N}(\text{H})^t\text{Bu})(\text{THF})$ (2) (Scheme 1). Spectral features, including ${}^1\text{H}$ and ${}^{13}\text{C}$ NMR spectra, and elemental analysis confirmed the

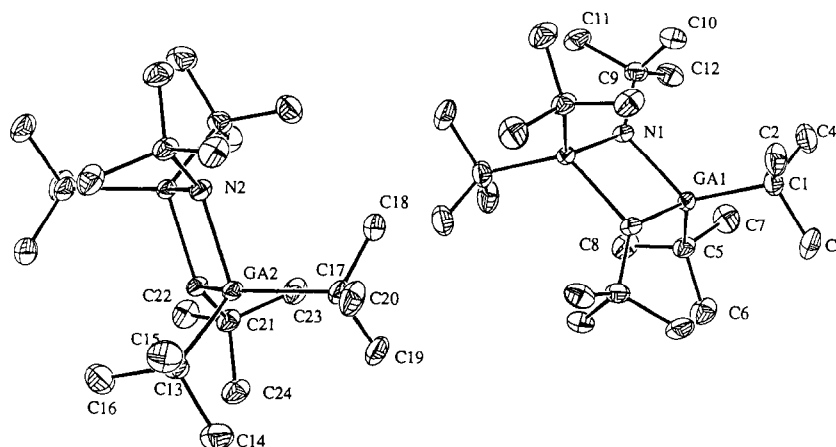


Fig. 1. Molecular structure and atom numbering scheme for $[\text{Ga}({}^t\text{Bu})_2\text{NH}^t\text{Bu}]_2$ (1) showing the two molecules in the asymmetric unit. Hydrogen atoms have been omitted for clarity.

formulation of **2**. Complex **2** can be sublimed under oil pump vacuum at 90 °C.

Similarly, attempts to make a Lewis base-free complex from the reaction of ${}^n\text{Bu}_2\text{GaCl}$ and $\text{Li}[\text{N}(\text{H})(2,6\text{-C}_6\text{H}_3\text{Me}_2)]$ were unsuccessful, yielding only oily intractable materials. Addition of pyridine did not facilitate the crystallization of this material but did allow the isolation of an oil that was confirmed by spectroscopic characterization and microanalysis to be ${}^n\text{Bu}_2\text{Ga}[\text{NH}(2,6\text{-C}_6\text{H}_3\text{Me}_2)]\text{py}$ (**3**) (Scheme 1). Preliminary attempts to prepare $\{{}^n\text{Bu}_2\text{Ga}[\text{NH}(2,6\text{-Me}_2\text{C}_6\text{H}_3)]\}_n$ have been unsuccessful, even with the addition of coordinating ligands such as THF and pyridine.

The use of excess anionic ligands also allowed the isolation and crystallization of alkyl amido complexes from the oily materials produced from the 1:1 reaction of ${}^n\text{Bu}_2\text{GaCl}$ and lithium amide. For example, the interaction of $\{{}^n\text{Bu}_2\text{Ga}[\text{NH}(2,6\text{-Me}_2\text{C}_6\text{H}_3)]\}_n$ with an additional equivalent of $\text{LiN}(\text{H})(2,6\text{-C}_6\text{H}_3\text{Me}_2)$ produced ${}^n\text{Bu}_2\text{Ga}[\text{NH}(2,6\text{-Me}_2\text{C}_6\text{H}_3)]_2[\text{Li}(\text{Et}_2\text{O})]$ (**4**) (Scheme 1). Structural characterization by single crystal X-ray diffraction confirmed the molecular connectivity of this complex [15]. Complex **4** crystallized in the monoclinic space group $P21/n$ with $Z = 4$. As shown in Fig. 2, the structure consists of a gallium center in a pseudotetrahedral coordination sphere composed of two

amido ligands ($\text{Ga-N1} = 2.011(8)$ Å, $\text{Ga-N2} = 2.006(7)$ Å), and two ${}^n\text{Bu}$ ligands ($\text{Ga-C17} = 2.002(9)$ Å, $\text{Ga-C21} = 1.985(12)$ Å). A bridging interaction of the lithium cation with the lone pair of electrons on each of amido nitrogen atoms generates a molecular core which is made up of a planar Ga-N1-Li-N2 distorted square (N1-Ga1-N2 94.4°, Ga1-N2-Li1 86.2°, N1-Li1-N2 92.2°, Ga1-N1-Li1 87.1). Additional selected bond distances and angles for **4** are given in Tables 1 and 2. Consistent with the spectroscopic data is the presence of one diethyl ether ligand completing the unusual, distorted trigonal planar coordination of the Li cation.

We have reported the synthesis of four new dialkyl primary amido complexes of gallium. Two of these compounds, a dimeric species (**1**) and a monomeric dialkyl diamido gallate complex (**4**), have been structurally characterized. Thermal studies of these and analogous complexes are currently underway.

2. Experimental section

2.1. General procedure

All reactions were carried out either in a nitrogen filled drybox or under nitrogen with standard Schlenk-

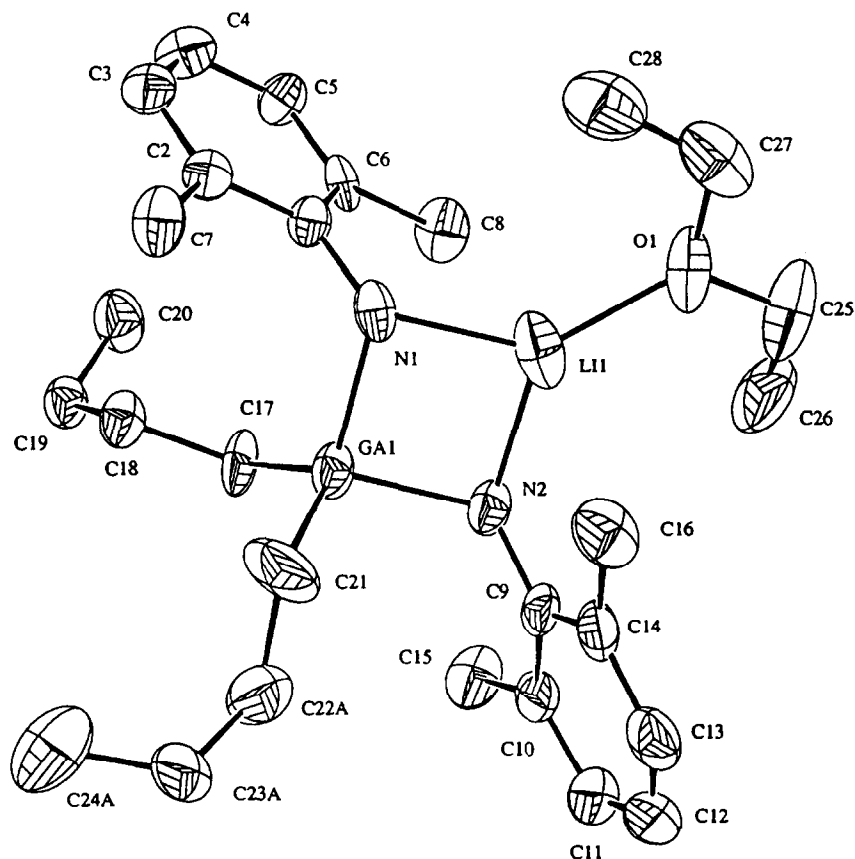


Fig. 2. Molecular structure and atom numbering scheme for ${}^n\text{Bu}_2\text{Ga}[\text{NH}(2,6\text{-Me}_2\text{C}_6\text{H}_3)]_2[\text{Li}(\text{Et}_2\text{O})]$ (**4**). Hydrogen atoms have been omitted for clarity.

line techniques. Diethyl ether, hexane and THF were distilled from Na/K alloy. Toluene was distilled from sodium. Pyridine (py) was dried and purified by distillation from CaH₂. Deuterated benzene was dried by vacuum transfer from potassium. ⁿBuLi, ¹BuMgCl, GaCl₃, NH¹₂Bu and NH₂(2,6-(Me)₂C₆H₃) were purchased from Aldrich and used as received. LiNH¹Bu and LiNH(2,6-(Me)₂C₆H₃)(THF) were synthesized by reaction of the corresponding amine with ⁿBuLi at 0 °C in diethyl ether and tetrahydrofuran, respectively. After warming to room temperature and reduction of the volume, colorless crystals were obtained in good yield. NMR spectra were obtained on a Bruker 500 MHz NMR spectrometer using C₆D₆ as solvent. IR spectra were recorded as Nujol mulls between NaCl plates using a Mattson Galaxy 3020 spectrometer.

2.2. Preparation of ¹Bu₂GaCl

Prepared according to literature procedure by the reaction of ¹BuMgCl and GaCl₃ in a 2:1 ratio [12]. Yield after crystallization 71%. Purity of the final products was confirmed by ¹H NMR. ¹H NMR (C₆D₆): δ 1.32 (s).

2.3. Preparation of ⁿBu₂GaCl

The compound ⁿBu₂GaCl was prepared according to literature procedure by the reaction of ⁿBuLi and GaCl₃ in a 2:1 ratio [13]. Yield after vacuum distillation 88%. Purity of the final products was confirmed by ¹H NMR. ¹H NMR (C₆D₆): δ 1.61 (m, CH₂CH₂CH₂, 4H), 1.35 (m, CH₂CH₂CH₃, 4H), 0.94 (t, CH₂CH₃, 6H), 0.82 (m, CH₂CH₂Ga, 4H).

2.4. [Ga(¹Bu)₂(NH¹Bu)]₂ (1)

In a dry box, a hexane suspension of 1.25 g (5.7 mmol) of ¹Bu₂GaCl was combined with 0.47 g (5.8 mmol) of LiNH¹Bu. After stirring for 48 h and removal of the white precipitate by filtration, the solvent was removed by evaporation. Crystallization of **1** from a THF/hexane solution cooled to -30 °C afforded 0.70 g (49%) of colorless cubic crystals, m.p. (sealed) 135–140 °C(dec). IR (cm⁻¹): 1461(s), 1375(m), 1193(m), 1176(m), 1016(m), 929(w), 908(w), 885(m), 808(s), 750(w), 723(w). H NMR (C₆D₆): δ 2.12 (br s, H, NH), 0.32 (s, 9H, ¹Bu), 0.18 (s, 9H, ¹Bu). Anal. Calc. for C₁₂H₂₈NGa: C, 56.28; H, 11.02; N, 5.47%. Found: C, 56.15; H, 10.75; N, 5.66%.

2.5. Ga(ⁿBu)₂(NH¹Bu)(THF) (2)

To a solution of 1.25 g (5.7 mmol) of ⁿBu₂GaCl in hexane was added 0.45 g (5.7 mmol) of LiNH¹Bu at room temperature in a dry box. The reaction mixture

was stirred for 24 h. A white precipitate was removed by filtration and the filtrate was evaporated to an oily residue which after being dissolved in a THF/hexane solution and cooled to -30 °C produced 0.60 g (36%) of **2**, m.p. (sealed) 34–36 °C. IR (cm⁻¹): 1461(s), 1373(s), 1205(m), 1068(m), 1045(m), 1020(m), 948(w), 914(w), 875(m), 754(w), 721(w). ¹H NMR (C₆D₆): δ 3.56 (m, THF, 4H), 1.64 (m, CH₂CH₂CH₂, 4H), 1.54 (m, CH₂CH₂CH₃, 4H), 1.40 (m, 4H, THF), 1.07 (s, C(CH₃)₃, 9H), 1.05 (t, CH₂CH₃, 6H), 0.98 (s, NH(¹Bu), H), 0.64 (m, CH₂CH₂Ga, 4H). ¹³C NMR (C₆D₆): δ 51.6 (NC(CH₃)₃), 32.9 (C(CH₃)₃), 29.5 (CH₂CH₂CH₂), 28.8 (CH₂CH₂CH₃), 16 (CH₂CH₂Ga), 14.1 (CH₂CH₃). Anal. Calc. for C₁₆H₃₆NGa: C, 61.56; H, 11.62; N, 4.49%. Found: C, 61.15; H, 11.75; N, 4.46%.

2.6. ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]py (3)

A solution of 0.90 g (4.1 mmol) of ⁿBu₂GaCl in ether was combined with 0.53 g (4.2 mmol) of LiNH(2,6-Me₂C₆H₃). The reaction mixture was stirred for 24 h. A white precipitate was removed by filtration. The filtrate was evaporated to an oily residue and excess pyridine was added. The solution was then evaporated, leaving an involatile oil. The yield of **3** was 0.78 g (41%). IR (cm⁻¹): 3297(w), 1594(m), 1446(s), 1423(s), 1373(m), 1280(m), 1253(s), 1224(s), 1189(s), 1097(s), 1068(s), 1041(m), 1012(m), 836(s), 754(s), 698(s), 632(s). ¹H NMR (C₆D₆): δ 8.13 (d, 2H, py), 7.15 (d, 2H, py), 6.90 (m, 2H, Ar-H), 6.74 (m, ¹H, py), 6.39 (m, H, Ar-H), 3.09 (br s, H, NH), 2.36 (s, 6H, ArCH₃), 1.75 (m, 4H, CH₂CH₂CH₂), 1.61 (m, 4H, CH₂CH₂CH₃), 1.04 (t, 6H, CH₃), 0.77 (m, 4H, GaCH₂). Anal. Calc. for C₂H₃₃N₂Ga: C, 65.82; H, 8.68; N, 7.31%. Found: C, 65.65; H, 8.75; N, 7.46%.

2.7. ⁿBu₂Ga[NH(2,6-Me₂C₆H₃)]₂[Li(Et₂O)] (4)

A solution of 4.5 ml (36 mmol) of H₂N(Ph(Me)₂) in hexane was cooled to -78 °C and 28 ml of a 2.5 M hexane solution (70 mmol) of ⁿBuLi was added by cannula and the solution was slowly allowed to warm to room temperature. A hexane solution of 5.0 g (30 mmol) of GaCl₃ was then added to this solution and the mixture was stirred for 17 h, filtered to remove a white precipitate, and evaporated to dryness. The resulting solid was dissolved in ether and cooled to -30 °C yielding 7.8 g (38%) of **4**. Sublimes 300 °C under vacuum. IR (cm⁻¹): 1592(m), 1456(s), 1376(s), 1254(m), 1224(m), 1205(m), 1096(m), 909(w), 824(m), 756(s), 680(m). ¹H NMR (C₆D₆): δ 7.00 (d, 4H, Ar-H), 6.70 (m, 2H, Ar-H), 2.79 (q, 4H, OCH₂), 2.63 (br s, 2H, NH), 2.22 (s, 12H, ArCH₃), 1.37 (m, 4H, CH₂), 0.89 (t, 6H, CH₃), 0.58 (t, 6H, OCH₂CH₃), 0.53 (m, 4H, CH₂). Anal. Calc. for C₂₈H₄₈N₂O₂GaLi: C, 61.15; H, 11.75; N, 4.46%.

66.53; H, 9.57; N, 5.54%. Found: C, 66.15; H, 9.75; N, 5.46%.

2.8. X-Ray crystallography

Intensity data were collected on a Rigaku diffractometer at $-110\text{ }^{\circ}\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 49.8° (complex **1**) and 50° (complex **2**) for crystals mounted on glass fibers. Cell constants and orientation matrices were obtained from the least-squares refinement of 24 or 25 carefully centered high-angle reflections. Redundant reflections were removed from the data set. The intensities of three representative reflections were measured after every 150 reflections to monitor the crystal and instrument stability. Data were corrected for Lorentz and polarization effects but not for absorption. The structures were solved by direct methods. The non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were located in the difference Fourier maps and refined isotropically in the case of favorable observation/parameter ratio. The final cycle of full-matrix least squares refinement was based on the number of observed reflections with ($I > 2.5\sigma(I)$). In the case of complex **2** there was disorder for one of the n-butyl chains bound to gallium. The chain was disordered over two positions. Anomalous dispersion effects were included in the F_{calc} . All calculations were performed using the NRCVAX package. Details of the data collection, refinement and final atomic coordinates are reported in the Supplementary Materials.

3. Supplementary material available

Descriptions of the structural solutions, tables of atomic positions, thermal parameters, crystallographic data, bond distances and angles, ORTEP drawings, and structure factor tables for compounds **1** (41 pages) and **4**

(40 pages). Ordering information is given on any current masthead page.

Acknowledgments

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