two as observed in solution. This shows that the equivalence of the ortho methyl groups (δ 24.23 ppm in solution) has been removed in the solid state as a result of the crystal packing. This is shown in Figure 1B. The equivalence also may have been removed for the ring carbon atoms but is not observable because of the line widths. The ²⁷Al chemical shifts obtained in solution and by $\ensuremath{\mathrm{CP}}/\ensuremath{\mathrm{MAS}}$ on a solid sample were centered at 108 and 101 ppm (relative to 1 M Al(NO_3)₃), respectively, and were extremely broad $(\nu_{1/2} \ge 10\,000 \text{ Hz})$ consistent with the planar structure.¹⁵

Conclusions. In trimesitylaluminum the structural data indicates that the reason for the formation of monomer rather than dimer is the result of steric interference between the ortho methyl groups which prevent the molecule from forming a stable dimer. This was suggested by our earlier studies on tri-o-tolylaluminum dimer which showed that the C-Al-C-Al ring was distorted from that observed in aromatic compounds in which there were no substituents in the ortho positions.¹³ The fact that the Al-C distance observed is nearly identical with that observed in a variety of other derivatives including monomeric trimethylaluminum shows that the steric interference does not significantly alter the Al-C bond. The steric

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hindrance, and not alteration in bonding, therefore appears to accounts for the limited reactivity of this molecule toward Lewis bases and likely toward other reagents since many of these reactions proceed via initial complex formation. Thus, by prevention of the initial complex formation through the steric crowding around the vacant orbital on aluminum atom, the reactivity of the Al-C bond is reduced.

Preliminary studies have been carried out on this compound which indicate that its reactivity is also altered from that of other compounds because of the structural effects of the mesityl group which take the form of reduced reactivity toward hydrolysis, complex formation, and oxidation. These studies are now being pursued further.

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Supplementary Material Available: Listings of anisotropic thermal parameters and atomic positional parameters, (2 pages); a listing of observed and calculated structure amplitudes (4 pages). Ordering information is given on any current masthead page.

Synthesis, Characterization, and Crystal and Molecular Structure of Trimesitylgallium(III)

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The compound trimesitylgallium(III) (GaMes₃) has been prepared and fully characterized by elemental analysis, IR and ¹H NMR data, Lewis acid-base studies, and an X-ray structural study. Trimesitylgallium(III) crystallizes in the centrosymmetric trigonal space group $P\overline{3}$ (No. 147) with a = 13.415 (4) Å, c = 7.628 (2) Å, and ρ (calcd) = 1.19 g cm⁻³ for M_r 427.3 and Z = 2. Single-crystal X-ray diffraction data (Mo K α , $2\theta = 4.0-40.0^{\circ}$) were collected with a Syntex P2₁ automated four-circle diffractometer. All atoms (including hydrogen atoms) were located, and the structure was refined to $R_F = 3.9\%$ and $R_{wF} = 3.3\%$ for all 743 unique reflections (none rejected). The gallium(III) atom lies on a site of crystallographically imposed C_3 symmetry (and approximate D_3 symmetry) and has a trigonal-planar stereochemistry (C-(1)-Ga-C(1') = 119.93 (16)°) with a gallium-carbon bond length of 1.968 (4) Å. The aromatic rings of the mesityl ligands form a propeller-like arrangement about gallium with an angle of 55.9° between each ring and the metal coordination plane.

Introduction

Bulky substituents have been used in main-group chemistry to stabilize a variety of unusual compounds. Of the possible groups with large steric demands, aromatic rings with alkyl groups in the 2- and 6-positions have been frequently employed. Mesityl (Mes) groups have been used to stabilize Si_2Mes_4 , the first fully characterized compound with a silicon-silicon double bond.¹ The 2,6diethylphenyl substituent stabilized a compound with a germanium-germanium double bond,² whereas the 2,6-

diisopropylphenyl group provided sufficient steric effects to provide a tin-tin derivative.³ The 2,4,6-tri-tert-butylphenyl substituent was used as a substituent in compounds with phosphorus-phosphorus⁴ and arsenic-arsenic double bonds.⁵

In group 13 chemistry in general and gallium chemistry in particular, very few typical compounds with bulky substituents are known. The derivative $Ga(CH_2SiMe_3)_3$, a pyrophoric liquid at room temperature, has been fully

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characterized.6 In contrast, trimesitylgallium(III) (GaMes₃) has been reported, but no characterization data were given.⁷ The compound was prepared in very low vield (8%) from gallium metal and dimesitylmercury in refluxing benzene. In this paper we report the facile synthesis of GaMes₃ in high yield from GaCl₃ and the mesityl Grignard reagent in Et_2O . The new compound has been fully characterized by elemental analyses, IR and ¹H NMR spectroscopic data, and an X-ray structural study.

Experimental Section

General Data. All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or a purified argon atmosphere. Gallium(III) chloride was purified by sublimation under high vacuum at 70-80 °C immediately prior to use. Bromomesitylene was purchased from Aldrich Chemicals and was used as received. The Grignard reagent was prepared in refluxing Et_2O by using standard procedures. All solvents were purified before use. Analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. Absorption intensities are reported with abbreviations w (weak), m (medium), s (strong), vs (very strong), and sh (shoulder). The ¹H NMR spectra were recorded at 90 MHz by using a Varian Model EM-390 spectrometer. Chemical shifts are reported in δ units (ppm) and are referenced to SiMe₄ as δ 0.00 and benzene as δ 7.13. All NMR tubes were sealed under vacuum.

Synthesis of GaMes₃. In a typical synthetic experiment, the Grignard reagent MesMgBr prepared from 5.80 g (0.239 mol) of magnesium, 44.2 g (0.222 mol) of MesBr, and 90 mL of Et₂O was added slowly over a period of 45 min to 8.70 g (0.0494 mol) of GaCl₃ dissolved in 250 mL of Et₂O. After the addition was complete, the reaction mixture was refluxed for 16 h. The ether was then removed by vacuum distillation. The resulting solid was extracted with 175 mL of pentane eight times. The yellowish pentane soluble solid was then recrystallized from pentane to provide 15.1 g (35.4 mmol, 71.6% yield based on GaCl₃) of crude GaMes₃. The product was further purified by vacuum sublimation at 160-175 °C and additional recrystallizations from pentane. GaMes₃: mp 186–187.8 °C; ¹H NMR (C_6D_6) δ 2.14 (p-Me, 3 H), 2.33 (o-Me, 6 H), 6.70 (m-H, 2 H); IR (Nujol mull, cm⁻¹) 2725 (w), 1775 (w), 1737 (w), 1597 (s), 1547 (s), 1282 (w), 1258 (vw), 1231 (w), 1166 (vw), 1047 (w), 1027 (m), 1008 (sh), 938 (vw), 918 (vw), 883 (w), 843 (vs), 829 (w), 795 (vw), 705 (w) 681 (vw), 580 (m), 558 (m), 540 (s), 512 (vw), 480 (w), 338 (vs), 300 (w), 243 (m). Solubility: soluble in benzene, Et₂O, and THF; slightly soluble in pentane. No stable adducts were formed with Et₂O or THF. Anal. Calcd: C, 75.90; H, 7.78. Found: C, 75.73; H, 7.86. Crystals suitable for the X-ray structural study were obtained by recrystallization of a sample from pentane at -10 °C.

Collection of X-ray Diffraction Data. A glass-like, transparent, colorless crystal of trimesitylgallium(III) of approximate orthogonal dimensions $0.20 \times 0.28 \times 0.33$ mm³ was inserted into a thin-walled glass capillary under an inert atmosphere (Ar) and was aligned on our Syntex P21 automated four-circle diffractometer. Determination of the crystal's orientation matrix and unit cell parameters were carried out as described previously.⁴ A full shell of intensity data with $2\theta = 4.0-40.0^{\circ}$ were collected by using the $2\theta - \theta$ scan technique; details appear in Table I. The observed $\bar{3}$ (S₆) Laue symmetry indicated a trigonal space group based upon the point groups 3 (C_3) or $\overline{3}$ (S_6) . There were no systematic absences. Possible space groups are P3 (C_{3i}^1 , No. 143) or $P\overline{3}$ (C_{3i}^1 ; No. 147).⁹ The latter centrosymmetric alternative was later found to be the correct space group. All data were corrected for the effects of absorption ($\mu = 12.3 \text{ cm}^{-1}$) and the six symmetry-equivalent forms for each reflection were averaged (R(I)

Table I. Experimental Data for X-ray Diffraction Study of Trimesitylgallium(III)

(A) Crystal Parameters at	19 °C (292 K)
crystal system: trigonal	$V = 1188.9 (5) Å^3$
space group: $P\bar{3}$ (\bar{C}_{3i}^{1} ; No. 147)	Z = 2
a = 13.4152 (42)Å	$M_{\rm r}$ 427.3
c = 7.6284 (18)Å	formula: $C_{27}H_{33}Ga$ $\rho(calcd) = 1.19 \text{ g cm}^{-3}$

(B) Data Collection

diffractometer: Syntex P21

- radiation: Mo K $\bar{\alpha}$ ($\bar{\lambda} = 0.710730$ Å)
- monochromator: highly oriented (pyrolytic) graphite; equatorial mode; $2\theta(m) = 12.160^{\circ}$; assumed to be 50% perfect for polarization correction
- reflctns measd: $\pm h, \pm k, \pm l$ for $2\theta = 4.0-40.0^{\circ}$, 4444 total yielding 743 unique reflections (file name GAT2-192)
- scan type: coupled 2θ (crystal)- θ (counter)
- scan width: symmetrical, $[1.8 + \Delta(\alpha_1 \alpha_2)]^{\circ}$
- scan speed: 4.0 deg/min (in 2θ)
- bkgds: stationary crystal and counter at beginning and end of 2θ scan; each for one-half of total scan time
- std reflectns: three $(\overline{1}7\overline{1}; \overline{4}6\overline{2}; \overline{7}31)$ collected after each batch of 97 reflections; no fluctuations were observed
- abs coeff: $\mu = 12.3 \text{ cm}^{-1}$; corrected empirically by interpolation (in 2θ and ϕ) between ψ scans of close-to-axial reflections $(1,\overline{1},4; \overline{2},2,\overline{4}; 0,0,3)$

= 1.36% and $R_w(I) = 2.41\%$). Lorentz and polarization corrections were now applied, and the data were reduced to unscaled $|F_0|$ values. Any datum with I(net) < O was assigned the value $|F_0|$ = O. A Wilson plot was used to place data on an approximately absolute scale.

Solution and Refinement of the Structure of Trimesitylgallium(III). All crystallographic calculations were performed by using the SUNY-Buffalo modification of the Syntex XTL interactive crystallographic program package. Calculated structure factors were based upon the analytical forms of the neutral atoms' scattering factors; both the real $(\Delta f')$ and the imaginary $(\Delta f'')$ components of anomalous dispersion were included for all atoms.¹⁰ The function minimized during least-squares refinement was $\sum w(|F_{\rm o}| - |F_{\rm C}|)^2$, where $1/w = \{[\sigma(|F_{\rm o}|)]^2 + [0.015|F_{\rm o}]^2\}$.

The position of the gallium atom was determined from a Patterson map. A difference-Fourier synthesis phased by this atom $(R_F = 40.4\%)^{11}$ revealed the locations of all carbon atoms. Refinement of all appropriate positional and anisotropic thermal parameters led to convergence with $R_F = 7.7\%$, $R_{wF} = 6.8\%$, and GOF = 3.83 for 85 variables refined against all 743 reflections. A difference-Fourier synthesis now revealed the positions of all hydrogen atoms in the structure (peak heights ranging from 0.26 $e/Å^3$ down to 0.17 $e/Å^3$). These were included in the model, and their positional and isotropic thermal parameters were refined. Convergence was reached with $R_F = 3.9\%$, $R_{wF} = 3.3\%$, and GOF = 1.12 for 129 variables refined against all 743 unique reflections $(R_F = 3.0\% \text{ and } R_{wF} = 3.1\% \text{ for those } 655 \text{ reflections with } |F_0|$ > $3\sigma(|F_o|)).$

A final difference-Fourier map showed no significant features; the structure is both correct and complete. Final atomic parameters are collected in Table II.

Results and Discussion

The first example of a fully characterized gallium(III) compound containing a bulky aromatic substituent is represented by trimesitylgallium(III). The compound was readily prepared in 72% yield by using a typical Grignard reaction, and the Et₂O solvent was easily removed by vacuum distillation. The product was partially purified by sublimation at 160-175 °C under high vacuum and was recrystallized from pentane solution. Available data sug-

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Table II. Final Atomic Parameters for Trimesitylgallium(III)^a

		Leoney agains		
atom	x	У	z	B, \dot{A}^2
Ga	1/3	2/3	-0.16315 (8)	
C(1)	0.27678 (30)	0.77662 (31)	-0.17020 (46)	
C(2)	0.20576 (31)	0.77273 (34)	-0.30756 (45)	
C(21)	0.16153 (58)	0.67959 (57)	-0.44557 (74)	
C(3)	0.17647 (35)	0.85894(37)	-0.31976 (55)	
C(4)	0.21493 (34)	0.94806 (33)	-0.20359 (56)	
C(41)	0.18187 (59)	1.03919 (51)	-0.22297 (82)	
C(5)	0.28319 (35)	0.94950 (40)	-0.06755 (60)	
C(6)	0.31364 (30)	0.86513 (37)	-0.04835 (46)	
C(61)	0.38293 (60)	0.86904 (59)	0.11027 (70)	
H(21A)	0.1091 (45)	0.6844(42)	-0.5198 (71)	12.0 (17)
H(21B)	0.2271 (56)	0.6991 (49)	-0.5316 (84)	16.0 (24)
H(21C)	0.1342 (33)	0.6102 (36)	-0.4022 (52)	7.8 (13)
H(3)	0.1296 (26)	0.8532(23)	-0.4150 (38)	4.29 (72)
H(41A)	0.1424 (39)	1.0433 (38)	-0.1168 (64)	10.7 (14)
H(41B)	0.2450 (38)	1.1055 (39)	-0.2371 (56)	9.3 (15)
H(41C)	0.1300 (45)	1.0258(45)	-0.3302 (74)	13.3 (19)
H (5)	0.3103(28)	1.0093 (32)	0.0102 (48)	6.2 (10)
H(61A)	0.4190 (39)	0.9459 (44)	0.1665 (63)	11.2 (15)
H(61B)	0.4366 (37)	0.8563 (38)	0.0896 (56)	8.2 (16)
H(61C)	0.3299 (63)	0.7989 (63)	0.1930 (85)	18.2 (26)

^a Anisotropic thermal parameters for all non-hydrogen atoms are collected in the supplementary material.



Figure 1. Labeling of atoms in the trimesitylgallium(III) molecule. The molecule is viewed approximately (but not exactly) down the crystallographic C_3 axis (ORTEP-II diagram; 30% probability ellipsoids for non-hydrogen atoms and with hydrogen atoms artificially reduced for clarity).

gest that GaMes₃ is an exceedingly weak Lewis acid. Neither Et₂O nor THF form adducts which are stable to dissociation at room temperature. The only other reported organogallium(III) compound from which the strong base THF can be readily removed at room temperature is Ga- $(C_5Me_5)_2Cl.^{12}$

The crystal consists of discrete molecular units of formula $Ga(C_6H_2Me_3)_3$; there are no abnormally close intermolecular contacts. The scheme used for labelling atoms is shown in Figure 1; interatomic distances and angles are collected in Table III while intramolecular planes are given in Table IV. The gallium(III) atom lies on a site of crystallographic threefold symmetry (at 1/3, 2/3, z); the molecule thus has precise C_3 symmetry and approximate D_3 symmetry. Atoms in the basic asymmetric unit are labeled as shown in Table II; atoms in the other two-thirds of the molecule are labeled with a prime (C_3^1 rotation from basic unit) or double prime (C_3^2 rotation).

Table III. Selected Distances (Å) and Angles (deg) with Esd's for Trimesitylgallium(III)

(A) Gallium–Ca Ga–C(1)		rbon Distance 1.968 (4)				
	(B) C-C(ring) Distances				
C(1)-C(2)	1.399 (6)	C(4) - C(5)	1.378 (7)			
C(2) - C(3)	1.398 (6)	C(5) - C(6)	1.390 (7)			
C(3) - C(4)	1.365 (6)	C(6) - C(1)	1.390 (5)			
((C) $C(ring) - C($	Me) Distances				
C(2) = C(21)	1510(7)	C(6) - C(61)	1 511 (8)			
C(4) - C(41)	1.502(9)	0(0) 0(01)	1.011 (0)			
0(1) 0(11)						
	(D) C-H I	Distances				
C(3) - H(3)	0.94 (3)	C(41) - H(41B)	0.88 (5)			
C(5) - H(5)	0.92(4)	C(41) - H(41C)	1.03 (6)			
C(21) - H(21A)	0.93 (6)	C(61) - H(61A)	0.99 (5)			
C(21)-H(21B)	1.02(7)	C(61) - H(61B)	0.83 (6)			
C(21) - H(21C)	0.88 (4)	C(61) - H(61C)	1.06(7)			
C(41)-H(41A)	0.98 (5)					
(E)	C-Ga-C and	Ga-C-C Angles				
C(1) - Ga - C(1')	119.93 (16)	Ga-C(1)-C(2)	120.18 (29)			
		Ga-C(1)-C(6)	121.08 (30)			
O(0) O(1) O(0)	$(\mathbf{r}) \cup -\cup -\cup (\mathbf{r})$	(α) (α) (α) (β)	117 9 (4)			
C(0) = C(1) = C(2)	110.0 (4)	C(3) = C(4) = C(3)	117.3(4)			
C(1) - C(2) - C(3)	119.1 (4)	C(4) - C(5) - C(6)	122.1(4)			
C(2) - C(3) - C(4)	122.8 (4)	C(5) - C(6) - C(1)	120.1(4)			
(G) C–C–Me Angles						
C(1)-C(2)-C(21)	122.1(4)	C(5)-C(4)-C(41)	121.8(5)			
C(3)-C(2)-C(21)	118.8 (4)	C(5)-C(6)-C(61)	118.8 (5)			
C(3)-C(4)-C(41)	120.9 (5)	C(1)-C(6)-C(61)	121.0 (4)			

Table IV. Important Molecular Planes for Trimesitylgallium(III)^a

atom	dev, Å	atom	dev, Å			
(A) Coordination Plane						
0.0000X + 0.0000Y - 1.0000Z - 1.2460 = 0						
Ga*	-0.0014 (6)					
C(1)*	0.0524 (35)					
C(1')*	0.0524 (35)					
C(1'')*	0.0524 (35)					
(B) Plane of Aromatic Ring						
0.6223X + 0.5462Y - 0.5607Z - 4.7147 = 0						
C(1)*	0.011 (4)	Ga	0.2137(4)			
C(2)*	-0.003 (4)	C(21)	0.016(7)			
C(3)*	-0.008(5)	C(41)	0.014 (7)			
C(4)*	0.009 (5)	C(61)	-0.102(7)			
C(5)*	0.001(5)					
C(6)*	-0.011 (4)					
(C) Dihedral Angle						
	Plane A/Plane B		55.90°			

^a Atoms marked with an asterisk were used in determining the least-squares plane.

The gallium(III)-carbon(aromatic) distances are equivalent, with Ga-C(1) = 1.968 (4) Å. The gallium atom lies 0.063 Å from the C(1)...C(1')...C(1'') plane and is associated with the interligand angle C(1)-Ga-C(1') = 119.93 (16)°. The coordination geometry about gallium is thus close to the ideal trigonal-planar case.

The aromatic rings take up a propeller-like arrangement and make a dihedral angle of 55.90° with the coordination plane. The molecule viewed in Figure 1 has a left-handed propeller (i.e., Λ) arrangement; however, the other molecule in the unit cell of this centrosymmetric crystal has the opposite handedness. The dihedral angle of 55.90° is not purely a twist angle. The gallium(III) atom lies 0.2137 (4) Å from the plane of the aromatic ring so there is some bending back of the aromatic ring such that the Ga-C-(1)...C(4) system is no longer linear. The "bending back"

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Figure 2. Stereoscopic view of the trimesitylgallium(III) molecule, viewed approximately (but not exactly) down the Ga-C(1) bond and showing the propeller-like nature of the molecule.

can be estimated as 6.23° (from \sin^{-1} [(deviation of Ga atom)/(Ga-C(1) distance)]. It is this distortion which prevents the molecule (in the solid state) from having perfect D_3 symmetry.

Other distances lie within the expected ranges, with C-C(aromatic) = 1.365 (6)-1.399 (6) Å, C-Me = 1.502 (9)-1.511 (8) Å, and C-H = 0.83 (6)-1.06 (7) Å. (The average C-H distance here of 0.95 ± 0.06 Å is in precise agreement with the accepted "X-ray determined value" — see ref 13.)

The average gallium–carbon bond distances in GaMes₃ (1.968 (4) Å) are comparable with other three-coordinate gallium(III) compounds. For example, the corresponding distances are 1.967 (2) Å for GaMe₃ (electron diffraction data),¹⁴ 1.963 Å for Ga(CH=CH₂)₃,¹⁵ and 2.05 [3] Å for Ga(C₅H₅)₃.¹⁶ In contrast, most four-coordinate gallium compounds exhibit longer gallium–carbon distances such as 1.97 (1) Å for $[Ga(C_5Me_5)Cl_2]_2$,¹² 1.99 (2) Å for [Ga-(C₅Me₅)₂Cl]₂,¹² 1.998 [4] Å for Me₃GaNMe₃ (electron dif-

fraction data),¹⁷ 1.99 [1] Å for $[Ga(CH_2SiMe_3)_3]_2$ ·TMED,¹⁸ and 2.029 [14] Å for KGa(CH₂SiMe₃)₃H.¹⁹ However, $[Ga(C=CPh)Me_2]_2$ which has bridging C=CPh groups does not fit the above pattern and exhibits a surprisingly short terminal Ga-C(Me) bond distance²⁰ of 1.952 (8) Å. These data suggest that the reduced Lewis acidity of GaMes₃ is probably related to the steric demands of the mesityl group.

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Registry No. GaMes₃, 60607-12-7; MesBr, 576-83-0; GaCl₃, 13450-90-3.

Supplementary Material Available: A table of anisotropic thermal parameters (1 page); a list of observed and calculated structure factor amplitudes (4 pages). Ordering information is given on any current masthead page.

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