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Lewis Acid−Base Adducts of Pentamethylcyclopentadienylgallium with Trivalent Group 13-Element Compounds

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In the reaction of (η^5-pentamethylcylopentadienyl)gallium (Cp*Ga) with $B(C_6F_5)_3$, Ga^tBu_3, and Cp^*GaX_2 (X = Cl, I) the new complexes $Cp^*GaB(C_6F_5)_3$, **1**, $Cp^*GaGa^tBu_3$, **2**, and $Cp^*GaGa(X)_2Cp^*$ (X = Cl, **3**; I, **4**), respectively, were obtained. Alternatively, complex **3** was formed in the reaction of Cp*Ga with InCl. Compounds **1**−**4** were characterized by analytical and spectroscopic methods. In addition, X-ray crystal structure analyses of **1**, **3**, and **4** were performed.

Introduction

In the last years the synthesis and structural chemistry of low oxidation state compounds MR (M = group 13 element) have attracted much attention.[1−4] To stabilize these species, a substituent R with appropriate electronic and steric properties is required. By using the sterically demanding pentamethylcyclopentadienyl (Cp*) substituent Schnöckel et al. were able to synthesize Cp*Ga using metastable solutions of GaCl, which are difficult to prepare.[5] Interestingly, Cp*Ga forms a hexamer in the solid state but is monomeric in solution and in the gas phase.[6,7] We were able to synthesize Cp*Ga by reductive dehalogenation of Cp^*GaI_2 in good yield, thus making it available in large-scale quantities.[8] The ligand potential of Cp*Ga in transition metal chemistry is well documented.[8,9] Cp*Ga can replace carbon monoxide or other weakly bound ligands and is found in terminal as well as in bridging positions; it acts predominantly as a σ-donor and exhibits a cone angle of 112° in mononuclear Cr and Fe complexes. To learn more about the Lewis base properties of Cp*Ga, our interest focused on its reactivity with Lewis acid compounds from the p-block elements. Such adducts are generally unknown, except for a few that contain Cp*B and Cp*Al as donor components. For example Cp^*BBCl_2X (X = Cl, $SiMe_3$), **I**, have been prepared in collaboration with Siebert's group,[10] and Cowley et al. have reported $Cp^*Al{-}Al(C_6F_5)_3$, **II**, as a novel dialane valence isomer (Scheme 1).[11,12]

Here, we report the synthesis and structure of the adducts $Cp^*GaB(C_6F_5)_3$, **1**, $Cp^*GaGa^tBu_3$, **2**, and $Cp^*GaGa(X)_2Cp^*$ (X = Cl (**3**), I (**4**)) (see Scheme 2).

Results and Discussion

The reaction of $B(C_6F_5)_3$ with Cp*Ga in toluene gave the adduct $Cp^*GaB(C_6F_5)$, **1**, in 84% yield as colorless crystals (Scheme 3). The air-sensitive compound melts at 127 °C but decomposes slowly under these conditions. The complex dissolves readily in aprotic organic solvents such as *n*-hexane or toluene.

Reaction of Ga^tBu_3 with an equimolar amount of Cp*Ga resulted in formation of the adduct Cp*Ga-

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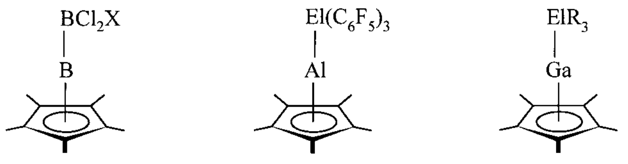
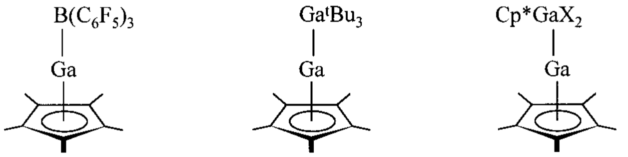
Scheme 1



X = Cl, SiMe$_3$ El = B, Al

I II III

Scheme 2



X = Cl, I

1 2 3, 4

Scheme 3

$$\text{Cp*Ga} + \text{B(C}_6\text{F}_5)_3 \xrightarrow{\text{toluene}} \underset{\mathbf{1}}{\text{Cp*GaB(C}_6\text{F}_5)_3}$$

$$\text{Cp*Ga} + \text{Ga}^t\text{Bu}_3 \xrightarrow{\text{toluene}} \underset{\mathbf{2}}{\text{Cp*GaGa}^t\text{Bu}_3}$$

Scheme 4

$$\text{Cp*Ga} + \text{Cp*GaCl}_2 \xrightarrow{\text{toluene}} \underset{\mathbf{3}}{\text{Cp*GaGa(Cl)}_2\text{Cp*}}$$

$$2\ \text{Cp*Ga} + 2\ \text{InCl} \xrightarrow[\text{- 2 In}]{\text{toluene}} \underset{\mathbf{3}}{\text{Cp*GaGa(Cl)}_2\text{Cp*}}$$

$$\text{Cp*Ga} + \text{Cp*GaI}_2 \xrightarrow{\text{toluene}} \underset{\mathbf{4}}{\text{Cp*GaGa(I)}_2\text{Cp*}}$$

Ga^{t}Bu$_3$, **2**, in quantitative yield (see Scheme 3). Compound **2** is an air- and moisture-sensitive solid with a melting point of 120 °C (dec), which dissolves readily in usual aprotic organic solvents.

The reaction of Cp*GaCl$_2$ with Cp*Ga gave the complex Cp*GaGa(Cl)$_2$Cp*, **3**. Compound **3** also was formed in the reaction of InCl with 1 equiv of Cp*Ga. Here, Cp*Ga reduced InCl to In metal. The formed Cp*GaCl$_2$ reacted with Cp*Ga to give **3**. The adduct Cp*GaGa(I)$_2$Cp*, **4**, was obtained in the reaction of Cp*GaI$_2$ with Cp*Ga (Scheme 4). Compounds **3** and **4** were isolated in high yield as yellow crystals, both of melting point 140 °C (dec), which are soluble in aprotic organic solvents.

Complexes **1**–**4** have been characterized by means of NMR and IR spectroscopy and elemental analysis. Crystals of **1**, **3**, and **4** suitable for an X-ray structure analysis were obtained by repeated crystallization from toluene.

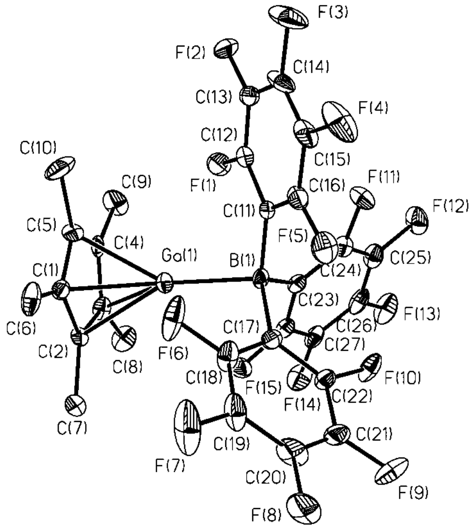


Figure 1. Molecular structure of Cp*GaB(C$_6$F$_5$)$_3$ (**1**), thermal ellipsoids at 50% probability.

The molecular structure of **1** is presented in Figure 1. Crystallographic data are given in Table 1, and selected bond lengths and angles are collected in Table 2.

Complex **1** crystallizes in the $P2_1/c$ space group. The Ga–B bond length is 2.153(6) Å. There is only little information in the literature about Ga–B distances. In gallium-substituted carboranes, these distances range from 2.062(11) to 2.605(25) Å.[13] Within the Cp*Ga unit of **1**, the η^5-bonding mode of the Cp* ring is maintained, although small deviations from ideal η^5-bonding are observed (Table 2). The Cp*$_{\text{centroid}}$–Ga–B vector is essentially linear (176.9°). The Cp*$_{\text{centroid}}$–Ga distance is 1.906(5) Å and is thus shortened by about 8% in comparison to the corresponding distance of Cp*Ga in the solid state[6] or in the gas phase.[7] It is comparable to the Cp*$_{\text{centroid}}$–Ga distance found in the transition metal complex Cr(Cp*Ga)(CO)$_5$ (1.910(4) Å).[8] As a consequence of the Cp*Ga coordination, the geometry at the boron atom changes from initially trigonal planar to distorted tetrahedral; the bond angles at boron add up to 342.3(5)°, a value similar to that found in Ph$_3$PB(C$_6$F$_5$)$_3$ (339.9(4)°).[14]

NMR (^{1}H, ^{13}C) investigations of complex **1** show that the asymmetry within the Cp*GaB moiety found in the solid state is not maintained in solution. Fast haptotropic shifts in the Cp* part of the molecule lead to averaged signals for the Cp* methyl groups and for the Cp* ring atoms. The ^{11}B NMR signal of **1** is in the range typical for a tetracoordinate boron atom. The ^{19}F NMR resonances for the C$_6$F$_5$ groups appear in a region expected for donor–acceptor complexes of B(C$_6$F$_5$)$_3$.[14,15]

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Table 1. Crystallographic Data for Cp*GaB(C_6F_5)$_3$ (1) and for Cp*GaGa(X)$_2$Cp* (X = Cl (3), I (4))

empirical formula	$C_{28}H_{15}BF_{15}Ga$, **1**	$C_{20}H_{30}Cl_2Ga_2$, **3**	$C_{20}H_{30}I_2Ga_2$, **4**
fw	716.93	480.78	663.68
cryst color, habit	colorless, needle	yellow, irregular	yellow, needle
cryst size, mm^3	$0.40 \times 0.34 \times 0.17$ mm^3	$0.40 \times 0.25 \times 0.20$ mm^3	$0.80 \times 0.20 \times 0.20$ mm^3
temp, K	100(2)	100(2)	173(2)
wavelength	Mo Kα 0.71073 Å (graphite monochromator)		
space group	$P2_1/c$	$P2_1/n$	$P2_1/n$
unit cell dimens	$a = 9.9330(2)$ Å $b = 11.5910(2)$ Å $c = 23.2040(5)$ Å $\beta = 94.3340(10)°$	$a = 8.5390(2)$ Å $b = 14.6360(2)$ Å $c = 17.6630(5)$ Å $\beta = 94.2140(11)°$	$a = 8.593(2)$ Å $b = 16.533(5)$ Å $c = 16.888(3)$ Å $\beta = 91.740(10)°$
V, Å^3	2663.92(9)	2201.50(9)	2398.1(10)
Z	4	4	4
density (calcd), Mg/m^3	1.788	1.451	1.838
θ range for data collection, deg	3.14−30.00	2.58−30.00	1.72−25.00
no. of reflns collected	12 255	11 028	4495
no. of ind reflns	7062 ($R_{int} = 0.0196$)	6273 ($R_{int} = 0.0226$)	4206 ($R_{int} = 0.0595$)
abs corr	multiscan	multiscan	semiempirical from ψ-scans
final R indices [$I > 2\sigma(I)$]	$R_F = 0.0882$, $R_{F2} = 0.2023$	$R_F = 0.0319$, $R_{F2} = 0.0689$	$R_F = 0.0570$, $R_{F2} = 0.1124$
no. of reflns with $I > 2\sigma(I)$	6006	5077	2333
no. of params	452	227	227
largest diff peak and hole, e Å^{-3}	0.880 and −0.804	0.631 and −0.595	0.9 and −1.2
diffractometer used	Nonius KappaCCD		Siemens P2(1) diffractometer
programs used	Siemens SHELXTL plus SHELXL-97		Siemens SHELXTL plus SHELXL-93
structure refinement	full-matrix least-squares on F^2		
remarks	disorder of Cp* on two positions (50:50)		

Table 2. Selected Bond Lengths (Å) and Angles (deg) for Cp*GaB(C_6F_5)$_3$ (1)

Ga(1)−C(1)	2.244(5)	B(1)−C(11)	1.620(8)
Ga(1)−C(2)	2.236(5)	B(1)−C(17)	1.622(8)
Ga(1)−C(3)	2.259(5)	B(1)−C(23)	1.619(8)
Ga(1)−C(4)	2.282(5)	Ga(1)−B(1)	2.153(6)
Ga(1)−C(5)	2.273(5)	Cp*$_{centroid}$−Ga	1.906(5)
C(11)−B(1)−Ga(1)	103.9(3)	C(11)−B(1)−C(17)	114.0(5)
C(17)−B(1)−Ga(1)	105.3(4)	C(23)−B(1)−C(17)	113.1(5)
C(23)−B(1)−Ga(1)	103.9(4)	C(23)−B(1)−C(11)	115.2(5)
Cp*$_{centroid}$−Ga(1)−B(1)	176.9	θ	116.0

As expected, the motion of the Cp* ring system and of the ^{t}Bu groups in Cp*GaGa^{t}Bu$_3$, **2**, is not restricted by steric interactions; thus, only one signal for the Cp* protons and one for the ^{t}Bu groups is detected in the ^{1}H NMR spectrum.

The molecular structure of **3** is shown in Figure 2. Crystallographic data are given in Table 1, and selected bond lengths and angles are collected in Table 3.

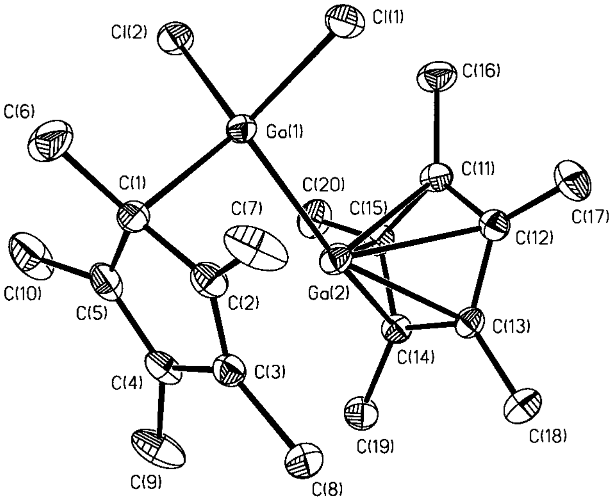

Figure 2. Molecular structure of Cp*GaGa(Cl)$_2$Cp* (**3**), thermal ellipsoids at 50% probability.

Complex **3** crystallizes in the monoclinic space group $P2_1/n$. The central part of the molecule is represented by a donor−acceptor Ga−Ga interaction with a distance of 2.4245(3) Å. This value is slightly smaller than that found in the complex TpGa−GaI$_3$ (Tp = tris(3,5-di-*tert*-butylpyrazolyl)hydroborato) (2.506(3) Å).[16] For neutral covalent organodigallanes[13f,17] and for mixed halogenoorganodigallanes,[18] Ga−Ga-distances between 2.33 and 2.60 Å have been found. Interestingly, the Cp* ring at the Ga(2) atom exhibits a “side on” coordination mode toward Ga(1). Thus, the deviation from a linear Cp*$_{centroid}$−Ga(2)−Ga(1) coordination amounts to 46.1°. The two Cp* systems are oriented eclipsed. The Cp* ring at the Ga atom Ga(1) is η^1-bonded (Ga(1)−C(1) = 2.072(2) Å), while a η^5-coordinated Cp* ring is found at the Ga atom Ga(2). Small deviations from ideal η^1/η^5 bonding situations are observed. Thus, on one hand, the Ga atom Ga(2) is not located exactly in a central position above the Cp* ring, as documented by different Ga(2)−C(Cp*) distances (2.174(2)−2.414(2) Å). On the other

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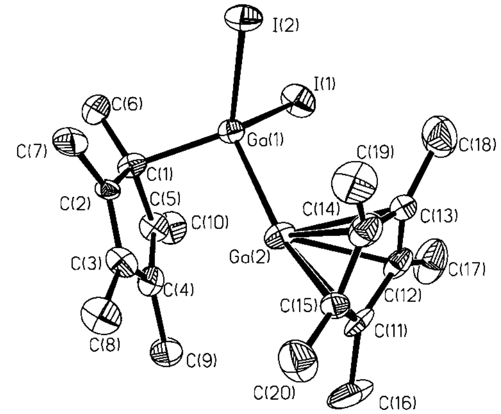

Figure 3. Molecular structure of Cp*GaGa(I)$_2$Cp* (**4**), thermal ellipsoids at 50% probability.

hand, the σ-bonded Cp* ring at Ga(1) exhibits deviations from an ideal η^1 bonding mode. Thus, the geometry at the C(1) atom is best described as distorted tetrahedral. The average Ga(2)−C(Cp*) distance amounts to 2.288(2) Å, which is 6% shorter than the corresponding bonds in Cp*Ga in the solid state[6] and in the gas phase.[7] Having in mind the rather small Ga(2)−Ga(1)−C(1) angle (98.62(5)°), one might consider an attractive interaction between the π-system of the σ-bonded Cp* ring and Ga(2). Indeed, the Ga(2)−C(3) and the Ga(2)−C(4) distances (2.932/2.905 Å) are shorter than the sum of the van der Waals radii of an sp^2-hybridized carbon atom and a gallium atom (3.6 Å).[19]

Complex **4** crystallizes in the $P2_1/n$ space group and is isostructural with complex **3**. The Ga−X distances in **3** and **4** differ as expected according to the different covalent radii of Cl (0.99 Å)[19] and I (1.33 Å);[19] the remaining structural features are comparable. Table 1 shows the crystallographic data of **4**; selected bond lengths and angles are given in Table 3.

Unprecedented dynamic effects are observed for **3** and **4** in solution. Thus, the Cp* rings in both compounds cannot be differentiated on the NMR time scale. Only one sharp averaged singlet at 1.84 ppm (**3**) or 1.76 ppm (**4**) is observed in the room-temperature ^{1}H NMR spectrum. Even at −80 °C, no signal separation is observable. Correspondingly, only two resonances are found in the ^{13}C NMR spectrum ((10.5 ppm (Cp* methyl); 117.9 ppm (Cp* ring-C) (**3**); (10.7 ppm (Cp* methyl); 118.5 ppm (Cp* ring-C) (**4**)). The fact that these resonances begin to broaden at −80 °C can be understood as a hint for the presence of two different but still dymamic Cp* systems. The nature of this dynamic process is rather complex and will be discussed in detail elsewhere.[20]

In summary, we were able to show that the donor potential of Cp*Ga is not restricted to transition metal centers, but can also be observed in case of coordinatively and electronically unsaturated group 13 element compounds. In the resulting adducts back-bonding is negligible. To compensate for the positive partial charge on the Ga(I) center caused by electron donation from gallium to the corresponding p-block element, the π-bond of the Cp* ring system to the Ga(I) center is strengthened. This is reflected by the shortening of the Cp*$_{\text{centroid}}$−Ga distance. The results described here expand the application of Cp*Ga as a ligand system and offer interesting opportunities for future use of this ligand.

Experimental Section

General Comments. All manipulations were carried out under purified argon atmosphere using standard Schlenk techniques. The solvents were commercially available, purified by conventional means, and distilled immediately prior to use. Cp*Ga[8], Cp*GaI$_2$[8], Ga^{t}Bu$_3$,[21] and B(C$_6$F$_5$)$_3$[22] were prepared according to the literature. InCl was purchased from Aldrich. The melting point determinations were performed using a Büchi 510 melting point apparatus. Elemental analyses were performed by the Microanalytical Laboratory of the Universität Bielefeld. The NMR spectra were recorded in benzene-d_6 using a Bruker Avance DRX 500 spectrometer (^{1}H 500.1 MHz; ^{13}C{^{1}H} 125.8 MHz). Chemical shifts are reported in ppm and were referenced to the solvent resonances as internal standard. IR data were collected using a Bruker Vektor 22-FT spectrometer. The samples were measured as KBr pellets.

Preparation of Cp*GaB(C$_6$F$_5$)$_3$, 1. Cp*Ga (0.07 g, 0.34 mmol) in toluene (5 mL) was added dropwise with stirring to a solution of B(C$_6$F$_5$)$_3$ (0.15 g, 0.32 mmol) in toluene (20 mL). The mixture was stirred for 3 h at room temperature. Subsequently, the solvent and volatiles were removed in vacuo. The residual colorless powder was dissolved in toluene and filtered. The filtrate was concentrated and cooled to −60 °C to yield 0.18 g of **1** (0.27 mmol, 84%) in the form of colorless crystals. Mp: 127 °C (dec). ^{1}H NMR (C$_6$D$_6$): δ 1.70 (s, 15 H, Cp* Me). ^{11}B NMR (C$_6$D$_6$): δ −19.6 (s). ^{13}C NMR (C$_6$D$_6$): δ 9.2 (Cp* methyl), 114.1 (Cp* ring), 118.1 (s, C$_{\text{arom}}$), 137.6 (d, [^1J(C−F) = 251 Hz], C$_{\text{arom}}$), 140.3 (d, [^1J(C−F) = 252 Hz], C$_{\text{arom}}$), 147.4 (d, [^1J(C−F) = 237 Hz], C$_{\text{arom}}$). ^{19}F NMR (C$_6$D$_6$): δ 155.7 (s, m-C$_6$F$_5$), 162.4 (s, p-C$_6$F$_5$), 187.7 (s, o-C$_6$F$_5$). IR (cm^{-1}, KBr): $\tilde{\nu}$ 2964 (w), 2927 (w), 2868 (w), 1646 (w), 1517 (s), 1465 (s), 1377 (w), 1288 (w), 1186 (m), 979 (m), 772 (w), 668 (w). Anal. Calcd for C$_{28}$H$_{15}$BF$_{15}$Ga (M = 716.94 g mol^{-1}): C, 46.91; H, 2.11. Found: C, 46.76; H, 2.15.

Preparation of Cp*GaGa^{t}Bu$_3$, 2. A solution of Cp*Ga (0.40 g, 1.95 mmol) in toluene (10 mL) was added dropwise to a solution of Ga^{t}Bu$_3$ (0.47 g, 1.95 mmol) in toluene (20 mL). After the reaction mixture was stirred for 5 h at room temperature, all volatile components were removed in vacuo. Subsequently, the yellow powder was recrystallized from toluene to give 0.85 g (1.91 mmol; 98%) of Cp*GaGa^{t}Bu$_3$ **2**. Mp: 120 °C (dec). ^{1}H NMR (C$_6$D$_6$): δ 1.26 (s, 27 H, ^{t}Bu), 1.79 (s, 15 H, Cp* methyl). ^{13}C NMR (C$_6$D$_6$): δ 9.5 (Cp* methyl), 27.6 (C(CH$_3$)$_3$), 33.0 (C(CH$_3$)$_3$), 113.9 (Cp* ring). IR (cm^{-1}, KBr): $\tilde{\nu}$ 2967 (w), 2922 (w), 2857 (w), 1636 (m), 1443 (w), 1376 (m), 1261 (m), 1088 (m), 936 (w), 668 (w). Anal. Calcd for C$_{22}$H$_{42}$Ga$_2$ (M = 446.02 g mol^{-1}): C, 59.25; H, 9.49. Found: C, 58.96; H, 9.41.

Preparation of Cp*GaGa(Cl)$_2$Cp*, 3. (a) A solution of Cp*Ga (0.31 g, 1.51 mmol) in toluene (10 mL) was added dropwise to Cp*GaCl$_2$ (0.42 g, 1.51 mmol) in toluene (20 mL). The mixture was stirred 3 h at room temperature. The adduct **3** was isolated as a yellow powder in quantitative yield by removing all volatile components in vacuo.

(b) Cp*Ga (0.31 g, 1.51 mmol) in toluene (10 mL) was added to a stirred suspension of InCl (0.23 g, 1.51 mmol) in toluene (20 mL). The reaction mixture was heated at reflux for 3 h. After the mixture was cooled to room temperature, all volatiles were removed in vacuo. The residual yellow solid was dissolved in toluene and filtered. The filtrate was concentrated and

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Table 3. Selected Bond Lengths (Å) and Angles (deg) for Cp*GaGa(X)$_2$Cp* (X = Cl, 3; X = I, 4)

	3	**4**		**3**	**4**
Ga(1)−Ga(2)	2.4245(3)	2.437(2)	Ga(2)−C(11)	2.4141(19)	2.185(11)
Ga(l)-C(l)	2.0717(19)	2.080(12)	Ga(2)−C(12)	2.3393(19)	2.301(12)
Ga(1)−Cl(1)	2.2167(5)	2.586(2)	Ga(2)−C(13)	2.1916(18)	2.377(12)
Ga(1)−Cl(2)	2.2185(5)	2.583(2)	Ga(2)−C(14)	2.1740(18)	2.301(13)
			Ga(2)−C(15)	2.3190(18)	2.186(11)
C(1)−C(2)	1.469(3)	1.46(2)	C(11)−C(12)	1.423(3)	1.40(2)
C(2)−C(3)	1.369(3)	1.38(2)	C(12)−C(13)	1.426(3)	1.43(2)
C(3)−C(4)	1.461(3)	1.46(2)	C(13)−C(14)	1.451(3)	1.40(2)
C(4)−C(5)	1.369(3)	1.37(2)	C(14)−C(15)	1.433(3)	1.44(2)
C(5)−C(1)	1.468(3)	1.50(2)	C(15)−C(11)	1.421(3)	1.49(2)
C(1)−Ga(1)−Cl(1)	116.55(6)	115.5(3)	C(5)−C(1)−Ga(1)	98.87(13)	97.2(7)
C(1)−Ga(1)−Cl(2)	113.83(6)	116.9(3)	C(2)−C(1)−Ga(1)	97.29(12)	99.0(7)
Cl(1)−Ga(1)−Cl(2)	105.25(2)	103.34(6)	C(6)−C(1)−Ga(1)	107.23(14)	107.5(8)
			C(1)−Ga(1)−Ga(2)	98.62(5)	99.6(3)

cooled to −60 °C to yield 0.29 g of **3** (0.61 mmol, 80% based on Cp*Ga) as yellow crystals. Mp: 135 °C (dec). ^{1}H NMR (C_6D_6): δ 1.84 (s, 30 H, Cp* methyl). ^{1}H NMR (toluene-d_8): δ 1.85 (s, 30 H, Cp* methyl). ^{1}H NMR (−80 °C/toluene-d_8): δ [ppm] 1.76 (s, 30 H, Cp* methyl). ^{13}C NMR (C_6D_6): δ 10.5 (Cp* methyl), 117.9 (Cp* ring). ^{13}C NMR (toluene-d_8): δ 10.1 (Cp* methyl) 117.3 (Cp* ring). ^{13}C NMR (−80 °C/toluene-d_8): δ 10.4 (Cp* methyl) 117.8 (Cp* ring). IR (cm^{-1}, KBr): $\tilde{\nu}$ 2958 (w), 2910 (w), 2858 (w), 1431 (m), 1376 (m), 1088 (m), 1019 (w). Anal. Calcd for $C_{20}H_{30}Cl_2Ga_2$ ($M = 480.81$ g mol^{-1}): C, 49.96; H, 6.29. Found: C, 50.12; H, 6.24.

Preparation of Cp*GaGa(I)$_2$Cp*, 4. A solution of Cp*Ga (0.31 g, 1.51 mmol) in toluene (10 mL) was added dropwise to Cp*GaI$_2$ (0.69 g, 1.51 mmol) in toluene (20 mL). The mixture was stirred 3 h at room temperature. After all volatile components were removed in vacuo, the yellow residue was dissolved in toluene. Subsequently, the solution was reduced in volume and stored at −60 °C. After 2 days 0.97 g (1.46 mmol; 97%) of Cp*GaGa(I)$_2$Cp*, **4**, could be isolated as yellow crystals. Mp: 137 °C (dec). ^{1}H NMR (C_6D_6): δ 1.76 (s, 30 H, Cp* methyl). ^{1}H NMR (toluene-d_8): δ 1.78 (s, 30 H, Cp* methyl). ^{1}H NMR (−80 °C/toluene-d_8): δ [ppm] 1.66 (s, 30 H, Cp* methyl). ^{13}C NMR (C_6D_6): δ 10.7 (Cp* methyl) 118.48 (Cp* ring). ^{13}C NMR (toluene-d_8): δ 10.2 (Cp* methyl) 118.0 (Cp* ring). ^{13}C NMR (−80 °C/toluene-d_8): δ 10.6 (Cp* methyl) 118.3 (Cp* ring). IR (cm^{-1}, KBr): $\tilde{\nu}$ 2964 (w), 2915 (w), 2852 (w), 1428 (m), 1371 (m), 1092 (m), 1013 (w). Anal. Calcd for $C_{20}H_{30}Ga_2I_2$ ($M = 663.68$ gmol^{-1}): C, 36.19; H, 4.55. Found: C, 36.23; H, 4.35.

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Supporting Information Available: Tables of crystal data, positional and thermal parameters, and bond lengths and angles. This material is available free of charge via the Internet at http://pubs.acs.org.

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