Ligand redistribution reactions of $Me_2Ga(C_5H_5)$ and $MeGa(C_5H_5)_2$

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Abstract

The compounds $Me_2Ga(C_5H_5)$ and $MeGa(C_5H_5)_2$ have been prepared by ligand redistribution reactions between appropriate stoichiometric quantities of $Ga(C_5H_5)$ and $GaMe_3$. Both compounds have been demonstrated by ^1H NMR spectral studies to be unstable in solution and to form symmetrized products by ligand redistribution reactions. Thus, $Me_2Ga(C_5H_5)$ forms $GaMe_3$ and $MeGa(C_5H_5)_2$ as primary products whereas $MeGa(C_5H_5)_2$ decomposes to $Ga(C_5H_5)_3$ and $Me_2Ga(C_5H_5)$. The compound $Me_2Ga(C_5H_5)$ has also been shown to serve as a cyclopentadienyl transfer reagent as it reacts with $FeCl_2$ to form $Fe(C_5H_5)_2$ and Me_2GaCl.

Introduction

Heteroleptic organometallic compounds of the heavier Group 13 elements gallium and indium form an interesting class of compounds which have not been extensively investigated. The first example of a fully characterized organoindium compound [1] with two different alkyl substituents $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ was only recently reported. Even though this indium compound could be prepared, isolated and characterized, the related organogallium compounds [1] $Ga(Me)(CH_2CMe_3)Cl$ and $Ga(CH_2CMe_3)(CH_2CMe_2Ph)Cl$ could not be isolated. All data were consistent with the isolation of symmetrized products, GaR_2Cl and GaR'_2Cl. Even though the synthesis of pure samples of $Ga(Me)(CH_2CMe_3)Cl$ and $Ga(CH_2CMe_3)(CH_2CMePh)Cl$ was unsuccessful, other heteroleptic organogallium compounds including $Me_2Ga(C_5H_5)$ [2–4], $Et_2Ga(C_5H_5)$ [2,3], $Me_2Ga(C\equiv CPh)$ [5,6], $Me_2Ga(C\equiv CMe)$ [7], $Me_2Ga(CH=CH_2)$ [8], $Me_2Ga(^tBu)$ [9] and $MeGa(^tBu)_2$ [9] have been reported. However, since we were unable to prepare pure samples of unsymmetrically substituted organogallium compounds, we were prompted to investigate the detailed nature of $Me_2Ga(C_5H_5)$ in the solution phase.

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The compound $Me_2Ga(C_5H_5)$ [2] has been reported as a colorless, crystalline solid with a melting point of 68–69°C. The compound had been prepared from Me_2GaCl with $Na(K)C_5H_5$ in cyclohexane at 50–70°C and had been isolated by sublimation at 65°C (0.5 mmHg) [2,3]. The elemental analysis for carbon was observed to be significantly lower than the calculated value whereas the percent gallium was higher than the calculated value [1]. The compound was also reported to have an association number of 1.3–1.4 in benzene solution [2]. The X-ray structural study [4] defined chains of dimethylgallium groups bridged by cyclopentadienyl rings at the 1 and 3 positions. The availability of $Ga(C_5H_5)_3$ [10] and $GaMe_3$ in our laboratories as well as the facility of ligand distribution reactions for preparing pure heteroleptic compounds prompted us to attempt the preparation and isolation of $Me_2Ga(C_5H_5)$ and $MeGa(C_5H_5)_2$. The preparation of Me_2Ga (C_5H_5) as a pure compound with excellent analyses for carbon and hydrogen was achieved. However, the melting point and association number in benzene solution were significantly different than those previously reported. In addition, NMR spectral studies of solutions of isolated samples of $Me_2Ga(C_5H_5)$, $MeGa(C_5H_5)_2$ and of solutions of these compounds prepared directly from $GaMe_3$ and $Ga(C_5H_5)_3$ suggest the occurrence of ligand redistribution reactions. Thus, available data suggest that neither $Me_2Ga(C_5H_5)$ nor $MeGa(C_5H_5)_2$ exist as single compounds in solution.

Experimental

All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or under a purified argon atmosphere. The starting material $Ga(C_5H_5)_3$ was prepared and purified by the literature method [10] whereas $GaMe_3$ was purchased from Strem Chemicals and used as received. Solvents were dried by conventional procedures. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. The ^1H NMR spectra were recorded either at 90 MHz by using a Varian EM390 spectrometer or at 400 MHz by using a Varian VXR-400 spectrometer. Proton chemical shifts are reported in δ units (ppm) and are referenced to $SiMe_4$ at δ 0.00 ppm and C_6H_6 at δ 7.15 ppm. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries. Molecular weights were measured cryoscopically in benzene by using an instrument similar to that described by Shriver and Drezdzon [11].

Synthesis of Me₂Ga(C₅H₅)

A 0.689 g (2.60 mmol) sample of $Ga(C_5H_5)_3$ was dissolved in 50 mL of pentane. Then, 0.597 g (5.20 mmol) of $GaMe_3$ was condensed into the reaction flask at -196°C. As the reaction mixture was warmed to room temperature, a colorless precipitate formed. After 2 h at room temperature, the solvent was removed by vacuum distillation. The reaction vessel was connected to a 100 mL Schlenk flask by using a glass elbow joint. The crude product was then sublimed at 60°C into the Schlenk flask that was cooled to -196°C to yield 1.20 g (7.28 mmol), 93.4%) of $Me_2Ga(C_5H_5)$ as a colorless crystalline solid. $Me_2Ga(C_5H_5)$: m.p. 97–98°C. ^1H NMR (CCl_4, 90 MHz): δ 6.29 (s, C_5H_5, 5H); -0.31 (s, Me, 6H). (C_6H_6, 90 MHz): δ 6.22 (s, C_5H_5, 5H); -0.42 (s, Me, 6H). (C_6D_6, 400 MHz): 6.19 (s, C_5H_5); -0.46

(s); -1.13 (s, br) (see Results and discussion). (THF-d_8, 90 MHz): δ 5.95 (s, C_5H_5); -0.54 (s, Me, GaMe$_3$·THF); -0.68 (s, Me, Me$_2$Ga(C_5H_5)·THF); -1.11 (s, Me, MeGa(C_5H_5)$_2$·THF). IR (Nujol mull, cm^{-1}): 3100w, 3080w, 1776w, 1720w, 1620w, 1399m, 1360m, 1342m, 1192m, 1181m, 1068w, 1056w, 1002w, 984m, 879w, 872m, 854m, 836s, 813m, 797s, 779s, 745s, 717s, 602m, 578vs, 522m, 290w. Cryoscopic molecular weight, benzene solution, formula weight 164.9 (observed molality, observed mol. wt., association): 0.0811, 178, 1.08; 0.0734, 181, 1.10; 0.0730, 177, 1.08; 0.0588, 179, 1.08; 0.0496, 178, 1.08. Anal. Found: C, 50.82; H, 6.76. Calc.: C, 50.99; H, 6.72%.

Reaction of 2 mol of Ga(C_5H_5)$_3$ per mol of GaMe$_3$

A 0.132 g (1.15 mmol) sample of GaMe$_3$ was vacuum distilled into a reaction flask containing 0.610 g (2.30 mmol) of Ga(C_5H_5)$_3$ in 30 mL of pentane. The reaction mixture was stirred at room temperature for 16 h. Then the solvent was removed by vacuum distillation. The yellow liquid which remained in the reaction flask was identified by ^{1}H NMR spectroscopy as MeGa(C_5H_5)$_2$. After standing at room temperature for 16 h, colorless crystals were observed in the liquid. The compounds MeGa(C_5H_5)$_2$ and Me$_2$Ga(C_5H_5) were identified by the ^{1}H NMR spectrum of the resulting liquid–solid mixture dissolved in CCl$_4$. Yellow liquid: ^{1}H NMR (CCl$_4$, 90 MHz): δ 6.25 (s, C_5H_5, 10H, MeGa(C_5H_5)$_2$); -1.04 (s, Me, 3H, MeGa(C_5H_5)$_2$). Liquid–solid mixture: ^{1}H NMR (CCl$_4$, 90 MHz): δ 6.20 (s, C_5H_5); -0.33 (s, Me, Me$_2$Ga(C_5H_5)); -1.12 (s, Me, MeGa(C_5H_5)$_2$).

Synthesis of MeGa(C_5H_5)$_2$·THF

A 0.111 g (0.967 mmol) sample of GaMe$_3$ was vacuum distilled into a reaction flask containing 0.513 g (1.93 mmol) of Ga(C_5H_5)$_3$ in 25 mL of pentane. The reaction mixture was stirred at room temperature for 16 h. Then, 10 mL of THF was vacuum distilled into the flask and the reaction mixture was stirred for an additional 3 h at room temperature. After the solvent was removed *in vacuo*, a slightly yellow liquid remained in the flask. ^{1}H NMR (benzene-d_6, 90 MHz): δ 6.20 (s, C_5H_5); 3.29 (m, THF); 1.18 (m, THF); -0.33 (s, Me); -0.91 (s, Me). (THF, 90 MHz): δ 5.94 (s, C_5H_5); -0.64 (s, Me, Me$_2$Ga(C_5H_5)·THF); -1.13 (s, Me, MeGa(C_5H_5)$_2$·THF). Anal. Found: C, 62.56; H, 7.17. MeGa(C_5H_5)$_2$·THF calc.: C, 62.76; H, 7.37%.

^{1}H NMR spectrum of a mixture of Me$_2$Ga(C_5H_5) and GaMe$_3$

A sample of 0.0317 g (0.194 mmol) of Me$_2$Ga(C_5H_5) was placed in an NMR tube. Then, a small quantity of GaMe$_3$ ($\sim$ 0.07 mmol) and C_6D_6 as solvent were added by vacuum distillation. The ^{1}H NMR spectrum at 400 MHz of the resulting solution revealed lines at -0.31 (singlet) and -6.20 ppm (singlet) (see Results and discussion).

^{1}H NMR spectrum of equimolar mixtures of Ga(C_5H_5)$_3$ and GaMe$_3$ in different solvents

The nature of the exchange reaction in the different solvents, CCl$_4$, C_6H_6 and THF, was investigated by ^{1}H NMR spectroscopy by combining Ga(C_5H_5)$_3$ and GaMe$_3$ in a 1:1 mol ratio in the given solvent. In a typical experiment, a weighed quantity of Ga(C_5H_5)$_3$ was placed in a reaction vessel connected to an NMR tube.

Then GaMe$_3$ was condensed into the reaction vessel at $-196°C$. The ^1H NMR spectrum (90 MHz) of the reaction mixture was recorded immediately upon warming the solution to room temperature.

CCl$_4$. The reaction mixture was prepared from 0.200 g (0.755 mmol) of Ga(C$_5$H$_5$)$_3$ and 0.0867 g (0.755 mmol) of GaMe$_3$ in 3 mL of CCl$_4$. ^1H NMR: -1.06 (very broad singlet, Me, MeGa(C$_5$H$_5$)$_2$, relative intensity 1); -0.30 (very broad singlet, Me, Me$_2$Ga(C$_5$H$_5$), relative intensity 2); 6.20 (singlet, C$_5$H$_5$).

C$_6$H$_6$. The reaction mixture was prepared from 0.0612 g (0.231 mmol) sample of Ga(C$_5$H$_5$)$_3$ and 0.0265 g (0.231 mmol) of GaMe$_3$ in 3 mL of C$_6$H$_6$. ^1H NMR: -1.07 (broadened singlet, Me, MeGa(C$_5$H$_5$)$_2$, relative intensity 1); -0.42 (broadened singlet, Me, Me$_2$Ga(C$_5$H$_5$), relative intensity 2); 6.20 (singlet, C$_5$H$_5$).

THF. The reaction mixture was prepared from 0.0550 g (0.207 mmol) of Ga(C$_5$H$_5$)$_3$ and 0.0236 g (0.207 mmol) of GaMe$_3$ in 3 mL of THF. ^1H NMR: -1.21 (sharp singlet, Me, MeGa(C$_5$H$_5$)$_2$ · THF, relative intensity 1); -0.68 (sharp singlet, Me, Me$_2$Ga(C$_5$H$_5$) · THF, relative intensity 2); 5.95 (singlet, C$_5$H$_5$).

Synthesis of Fe(C$_5$H$_5$)$_2$: Procedure A. A reaction mixture of 0.250 g (1.51 mmol) of Me$_2$Ga(C$_5$H$_5$) and 0.0960 g (0.726 mmol) of FeCl$_2$ in 25 mL of benzene was stirred at room temperature for 16 h. After the solvent was removed *in vacuo*, an orange solid remained in the reaction flask. Then, a volatile colorless crystalline product was sublimed at room temperature into a $-196°C$ trap. This colorless solid was identified as Me$_2$GaCl by its ^1H NMR spectrum in benzene solution (δ 0.23, Me; δ 0.23 Me$_2$GaCl). The remaining orange product was sublimed at 45°C under high vacuum. The solid was washed several times with dilute aqueous HCl and distilled water to remove any trace of Me$_2$GaCl. After drying the product in the air, 0.0638 g of Fe(C$_5$H$_5$)$_2$ (0.343 mmol, 47.2% based on FeCl$_2$) was isolated.

Procedure B. In the drybox, a 0.500 g (3.03 mmol) sample of Me$_2$Ga(C$_5$H$_5$) was placed into a 100 mL Schlenk flask and 0.200 g (1.58 mmol) of FeCl$_2$ was contained in a connecting side-arm dumper. Then, 50 mL of THF was vacuum distilled onto Me$_2$Ga(C$_5$H$_5$) and 25 mL of the solvent was distilled onto the FeCl$_2$. The sparingly soluble FeCl$_2$ was added with stirring to the solution of Me$_2$Ga(C$_5$H$_5$) at room temperature. After 3 h, the solid FeCl$_2$ had disappeared and an orange solution was observed. The reaction mixture was stirred for a total of 16 h at room temperature. The solvent was removed *in vacuo* and an orange solid mixed with a liquid remained in the reaction flask. By heating the liquid–solid mixture with a 40°C oil bath, the product was transferred under high vacuum into a Schlenk flask cooled to $-196°C$. With the product mixture maintained at $-196°C$, distilled water was added to the flask. After slowly warming the reaction mixture to room temperature, dilute aqueous HCl was added. The solution was then filtered and the orange solid product was collected on a glass frit. The product Me$_2$GaOH · THF was identified by its ^1H NMR spectrum (δ -0.32). The orange solid was washed several more times with distilled water to remove any trace of Me$_2$GaOH · THF. After drying the product in the air, 0.210 g (1.13 mmol, 74.6% based on Me$_2$Ga(C$_5$H$_5$)) of Fe(C$_5$H$_5$)$_2$ was isolated.

Results and discussion

The compound Me$_2$Ga(C$_5$H$_5$) has been prepared in nearly quantitative yield by a ligand redistribution reaction between GaMe$_3$ and Ga(C$_5$H$_5$)$_3$ in a 2 : 1 molar

ratio in pentane. It is noteworthy that $GaMe_3$ and $Ga(C_5H_5)_3$ are soluble in pentane but $Me_2Ga(C_5H_5)$ precipitates from the solution as it is formed. The product, as demonstrated by its elemental analyses for carbon and hydrogen and its melting point, is significantly more pure than the product originally isolated from the metathesis reaction between Me_2GaCl and $Na(K)C_5H_5$ in cyclohexane [2]. The observed carbon and hydrogen analyses for the new sample differed from the calculated values by only -0.17% and $+0.04\%$, respectively. The melting point of the compound prepared by the ligand redistribution reaction, 97–98°C, was considerably higher than that reported for the sample prepared by the metathetical reaction [2], 68–69°C. These data suggest that the compound as originally reported was impure. A possible impurity might be unreacted Me_2GaCl. The Me_2GaCl might not be consumed completely if the $Na(K)Cl$ formed in the metathesis reaction coated the insoluble reagent $Na(K)C_5H_5$ and thereby hindered reaction. It is also possible that the insoluble nature of $Me_2Ga(C_5H_5)$ in aliphatic hydrocarbon solvents might hinder the preparative reaction. Since $Me_2Ga(C_5H_5)$ and Me_2GaCl sublime at similar temperatures, unreacted Me_2GaCl would not have been removed and a product with a low melting point and low analytical data for percent carbon as well as high percent gallium would result.

The cryoscopic molecular weight studies of pure $Me_2Ga(C_5H_5)$ dissolved in benzene provide results which are in disagreement with earlier work [2]. The product from the ligand redistribution reaction had an average association value of 1.1 which was independent of concentration in the range of 0.08–0.05 m. These data suggest the presence of monomeric species in benzene solution. The original impure product had a reported association value of 1.3–1.4 (no concentration range given) [2]. An impurity of Me_2GaCl which exists as a dimer in benzene solution would produce an observed molecular weight which would be higher than calculated. Thus, the degree of association of $Me_2Ga(C_5H_5)$ is similar to most other triorganogallium compounds including $Ga(C_5H_5)_3$ [10] which are monomeric in benzene solution. Only $Me_2Ga(C{\equiv}CPh)$ [5] and $Me_2Ga(C{\equiv}CMe)$ [7] have been reported as dimers.

Proton NMR (400 MHz) studies of benzene solutions of $Me_2Ga(C_5H_5)$ suggest the occurrence of a ligand redistribution reaction to form symmetrized products (eq. 1)

$$2Me_2Ga(C_5H_5) \rightleftharpoons MeGa(C_5H_5)_2 + GaMe_3 \qquad (1)$$

and of methyl and cyclopentadienyl group exchange reactions between reactants and products. The spectrum of $Me_2Ga(C_5H_5)$ exhibits three lines, a sharp line at 6.19 ppm, a slightly broadened line at -0.46 ppm and a significantly broader but low intensity line centered at -1.13 ppm. (The line at -1.13 ppm was not observed in the 90 MHz spectra.) The line at 6.19 ppm can be assigned to cyclopentadienyl group protons. The line at -0.46 ppm arises from methyl group protons on $Me_2Ga(C_5H_5)$ and $GaMe_3$, which are averaged by methyl group exchange reactions. A separate line at -0.19 ppm, as expected for $GaMe_3$, was not observed. The line at -1.13 ppm is probably related to the protons of the methyl group of $MeGa(C_5H_5)_2$ which, in turn, are averaged by exchange with $GaMe_3$ formed by the ligand redistribution reaction (eq. 1). The suggestion of a methyl group exchange reaction between $Me_2Ga(C_5H_5)$ and $GaMe_3$ was verified by an independent experiment. When $Me_2Ga(C_5H_5)$ and $GaMe_3$ were mixed in

benzene, only one methyl–gallium and one cyclopentadienyl group resonance was observed. The broad line at -1.13 ppm originally observed in the spectrum of $Me_2Ga(C_5H_5)$ and assigned to the methyl protons of $MeGa(C_5H_5)_2$ disappeared. In addition, the methyl–gallium line moved from -0.46 to -0.31 ppm as would be expected if methyl group exchange were occurring in C_6D_6. Thus, $Me_2Ga(C_5H_5)$ does not exist as a single compound in benzene solution. Ligand redistribution reactions to form symmetrized products occur in solution. The observation that pure $Me_2Ga(C_5H_5)$ can be isolated from the preparative reaction in pentane must be related to the precipitation of the compound from solution as it is formed. The bridge bonds between cyclopentadienyl groups to form four-coordinate gallium [4] must be important to the stability of the compound as a solid.

Attempts have been made to prepare, isolate, and characterize the first example of a heavier Group 13 compound with two cyclopentadienyl groups, $MeGa(C_5H_5)_2$. When the reagents $Ga(C_5H_5)_3$ and $GaMe_3$ were combined in a $2:1$ ratio in pentane, a clear solution formed. When the pentane was removed, a yellow liquid was obtained. This yellow liquid was dissolved in turn in CCl_4 and it exhibited a ^1H NMR spectrum (100 MHz) which was appropriate for a compound with the formula $MeGa(C_5H_5)_2$, *i.e.* one line for C_5H_5 at 6.25 ppm and one line for Me at -1.04 ppm with an integration ratio of $10:3$. However, in the absence of solvent, the yellow liquid deposited a crystalline solid after 16 h. The ^1H NMR spectrum of this liquid–solid mixture dissolved in CCl_4 exhibited three lines, *i.e.* one C_5H_5 line at 6.20 ppm and two Me lines at -0.33 and -1.12 ppm. The chemical shift of the line at -0.33 ppm is indicative of $Me_2Ga(C_5H_5)$ (^1H NMR lines at 6.29 and -0.31 ppm). These data are consistent with the occurrence of a symmetrization reaction (eq. 2). Thus, $MeGa(C_5H_5)_2$ decomposed slowly and could not be fully characterized.

$$2MeGa(C_5H_5)_2 \rightarrow Me_2Ga(C_5H_5) + Ga(C_5H_5)_3 \qquad (2)$$
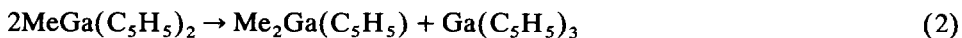
The ligand redistribution reactions of $Me_2Ga(C_5H_5)$ and $MeGa(C_5H_5)_2$ were confirmed by ^1H NMR spectra of solutions formed upon addition of the Lewis base, THF. The ^1H NMR spectrum of $Me_2Ga(C_5H_5)$ in THF exhibited four lines at -1.11, -0.68, -0.54 and 5.95 ppm. The three lines at higher field are assigned to the methyl group protons in $MeGa(C_5H_5)_2 \cdot THF$, $Me_2Ga(C_5H_5) \cdot THF$ and $Me_3Ga \cdot THF$, respectively. The equilibrium constant for eq. 3 calculated from the integration values of the lines was 0.13.

$$2Me_2Ga(C_5H_5) \cdot THF \rightleftharpoons Me_3Ga \cdot THF + MeGa(C_5H_5)_2 \cdot THF \qquad (3)$$
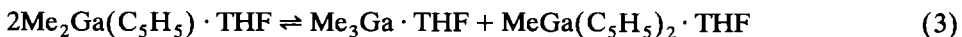
The ^1H NMR spectrum of a sample of $MeGa(C_5H_5)_2$ prepared in pentane as previously described and then dissolved in THF had three lines. These lines are consistent with the assignments given above and with the redistribution equilibrium as shown in eq. 4.

$$2MeGa(C_5H_5)_2 \cdot THF \rightleftharpoons Me_2Ga(C_5H_5) \cdot THF + (C_5H_5)_3Ga \cdot THF \qquad (4)$$
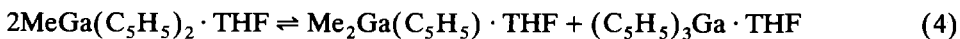
The equilibrium constant calculated from NMR integration data for eq. 4 was 0.17. The compound $Me_2Al(C_5H_5) \cdot THF$ [12] is reported to undergo an analogous redistribution equilibrium in THF. However, the product $MeAl(C_5H_5)_2 \cdot THF$ has never been isolated and studied.

The ligand redistribution reaction between $Ga(C_5H_5)_3$ and $GaMe_3$ was investigated further by combining the reagents in a $1:1$ ratio in THF, C_6H_6 and CCl_4 and by studying the ^1H NMR spectra of the resulting solutions. All data are consistent with the occurrence of ligand redistribution reactions to form $MeGa(C_5H_5)$ and $MeGa(C_5H_5)_2$.

$$Ga(C_5H_5)_3 + GaMe_3 \rightarrow Me_2Ga(C_5H_5) + MeGa(C_5H_5) \qquad (5)$$
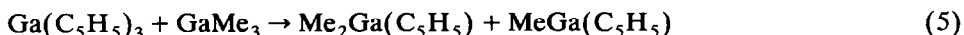
Only three ^1H NMR lines, as expected, were observed in each spectrum. However, it is noteworthy that the rates of exchange for the reactions in each solvent were different. The lines for the THF solution were very sharp and were indicative of slow exchange. In contrast, two extremely broad methyl–gallium lines were observed for the CCl_4 solution. The lines observed for benzene solution had widths which were in between those observed for THF and CCl_4 solutions, but certainly not sharp. The chemical shifts of the two methyl–gallium lines assigned to $Me_2Ga(C_5H_5)$ and $MeGa(C_5H_5)_2$ are consistent with all previous discussions.

The compound $Me_2Ga(C_5H_5)$ has been utilized as a source of C_5H_5 units for the synthesis of ferrocene.

$$2Me_2Ga(C_5H_5) + FeCl_2 \xrightarrow{\ C_6H_6\ } Fe(C_5H_5)_2 + 2Me_2GaCl \qquad (6)$$
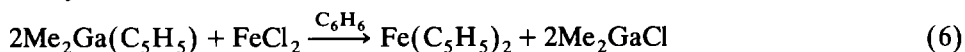
The ferrocene was characterized by its melting point and ^1H NMR spectrum whereas the colorless crystalline product was identified as Me_2GaCl by ^1H NMR spectroscopy. All characterization data for the two products were consistent with data for authentic samples.

Acknowledgments

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