coupling (Figures 7 and 8), and the rates of disappearance of the carbonyl complexes are first-order in complex concentrations. A plot of ln $(k/T)$ vs $1/T$ permits an estimate of activation parameters (Table XV).

The $\Delta H^*$ value (23.0 kcal/mol) is compatible with that for dissociation of CO from analogous complexes,[4,6] and the $\Delta S^*$ value is near zero (−4.3 cal/(mol·K)). These results are in accord with a dissociative mechanism (eq 9). The

$$\mathrm{Cp^*(1\text{-}PMe_2Ph\text{-}3\text{-}MePdl)Cr(CO)^+} \overset{k_1}{\underset{+\mathrm{CO}}{\overset{-\mathrm{CO}}{\rightleftharpoons}}} \mathrm{Cp^*(1\text{-}PMe_2Ph\text{-}3\text{-}MePdl)Cr^+ + CO} \quad (9)$$

rate of the reaction was retarded by added CO, which is consistent with a dissociative equilibrium (Table XII). Although the final products were not isolated and characterized, they cannot be converted to the parent complex under a CO atmosphere (1 atm). These results imply that CO substitution does not first take place by RNC attack on the pentadienyl followed by RNC transfer to the metal but rather by direct nucleophilic attack of RNC at the metal center. Since no RNC adduct was observed, $K$ would be $\ll 1$ for the equilibria reactions (eq 8), when RNC is the nucleophile. Thus, if CO substitution were to follow the addition of RNC on the terminal carbon of Pdl (eq 8), then at 40 °C $k_2 = Kk_1 = (7 \times 10^{-5})K \ll 7 \times 10^{-5}$ (Table XV). This is in contrast to the experimental results, which show that $k_2 = 2 \times 10^{-4}$ (Table X). Compared with the parent complexes, addition of phosphine ligand to Pdl promotes dissociation of the CO ligand from the metal, despite the values of $\nu_{CO}$ which show there is stronger $\pi$-back-bonding from the metal to CO in the adducts.

## Conclusions

The one-electron oxidations of Cp(Pdl)Cr(CO) compounds to the 17-electron cations lead to a rearrangement of the Pdl coordination from $\eta^5$(S) (sickle) to $\eta^5$(U). Dissociative exchange between coordinated and free CO for the cations is extremely slow. Reactions with isocyanide ligands followed different pathways, in which the Pdl ligands were lost, and species such as $\mathrm{CpCr(CNR)_4^+}$ were formed. The addition of phosphines to the cations generally seemed to lead to a nucleophilic attack on a terminal carbon atom of the pentadienyl ligand, leading essentially to a 17-electron $\mathrm{Cp(\eta^4\text{-}diene\text{-}PR_3^+)Cr(CO)}$ species from $\mathrm{[Cp^*(3\text{-}C_6H_9)Cr(CO)]^+}$ and an even more unusual species from $\mathrm{Cp(2,4\text{-}C_7H_{11})Cr(CO)^+}$, yet to be conclusively characterized.

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**Supplementary Material Available:** Pertinent bonding, positional, and thermal parameter tables for $\mathrm{[Cp(2,4\text{-}C_7H_{11})Cr(CO)]^+I^-\cdot CH_3CN}$, anisotropic thermal parameter, H atom positional parameter, and least-squares plane tables for $\mathrm{[Cp^*(3\text{-}C_6H_9)Cr(CO)]^+[BF_4]^-}$, crystallographic data and data collection and refinement detail, isotropic and anisotropic thermal parameter, bond distance and angle, and H atom positional parameter tables for $\mathrm{[CpCr(CN\text{-}\mathit{t}\text{-}C_4H_9)_4]^+[CpCr(CO)_3]^-}$ and $\mathrm{[CpCr(CN\text{-}\mathit{t}\text{-}C_4H_9)_4]^+[BF_4]^-}$, crystallographic data and data collection and refinement detail and isotropic and anisotropic thermal parameter tables for $\mathrm{[Cp^*(1\text{-}PMe_2Ph\text{-}3\text{-}C_6H_9)Cr(CO)]^+[BF_4]^-}$ and a diagram of $\mathrm{[CpCr(CN\text{-}\mathit{t}\text{-}C_4H_9)_4]^+[BF_4]^-}$ (27 pages). Ordering information is given on any current masthead page.

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# Observation of Rhodocenium and Substituted-Rhodocenium Ions and Their Neutral Counterparts by Mass Spectrometry

Dmitri V. Zagorevskii* and John L. Holmes*

*Chemistry Department, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5*

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Ionized rhodocene and its hydroxymethyl and carboxyl derivatives have been generated in the gas phase and characterized by tandem mass spectrometry. The fragmentations of these ions resemble the decomposition pathways for corresponding ferroceniums, but $\mathrm{Cp_2Rh^+}$ ions show greater dehydrogenation and ligand(s) decomposition processes. Substituent decomposition reactions dominate in the case of (hydroxymethyl)- and carboxyrhodoceniums. Neutralization–reionization experiments showed the existence of neutral rhodocene and rhodocenecarboxylic acid as stable species in the gas phase.

## Introduction

One of the more interesting compounds among the bis(cyclopentadienyl) complexes of transition metals, $\mathrm{Cp_2M}$, is rhodocene (M = Rh). This 19-electron radical complex was observed when its stable 18-electron analogue[1] was electrochemically reduced, and the estimated half-life of $\mathrm{Cp_2Rh}$ was measured to be ca. 2 s in acetonitrile solution at 25 °C.[2] A method for stabilizing both rhodocene and rhodocenium derivatives is to insert substituents into the cyclopentadienyl ligand(s) in order to delocalize positive charge or electron density and to create steric hindrance to the attack of intra reagents on the metal atom. This is a likely reason for the richer chemistry of polysubstituted rhodoceniums compared with the chemistry of mono- or disubstituted complexes.[3] For monosubstituted rhodo-

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[1] (a) Cotton, F. A.; Whipple, R. O.; Wilkinson, G. *J. Am. Chem. Soc.* **1953**, *75*, 3586. (b) Birmingham, J. M.; Fischer, A. K.; Wilkinson, G. *Naturwissenschaften* **1955**, *42*, 96.

[2] El Murr, N.; Sheats, J. E.; Geiger, W. E., Jr.; Holloway, J. D. L. *Inorg. Chem.* **1979**, *18*, 1443.

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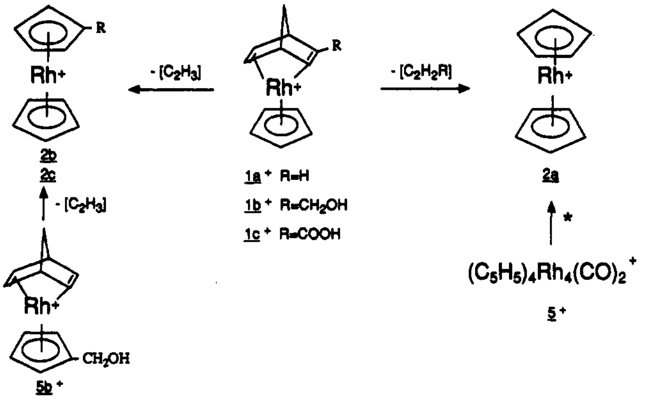
**Scheme I**



ceniums, $CpRh^+C_5H_4R$ (I), only a few have been synthesized and characterized,[3j,k] and their neutral counterparts are unknown. Nevertheless, the chemistry of these compounds is of interest, especially in comparison with similar complexes of other metals. The aim of this work was to study the possible existence of monosubstituted rhodocenes and rhodoceniums with a functional group in the substituent.

Neutralization–reionization mass spectrometry has been successfully used for the generation and characterization of unstable species.[4] In particular, some organic electronically unsaturated derivatives of Al,[5] Cu,[6] Co,[7,8] Ni,[7-9] and Fe[7,8,10,11] have been obtained. The present work is the first attempt to observe 19-electron neutral transition-metal $\pi$-complexes in the gas phase by this technique.

## Experimental Section

Compounds **1a–c** were synthesized according to the method of Chizhevskii et al.[12] Complex **5** was prepared as described in ref 13.

Electron ionization, metastable ion (MI), collisional activation (CA), and neutralization–reionization (NR) mass spectra were recorded using a modified VG Analytical ZAB-2F mass spectrometer.[14] In CA experiments Xe was used as the collisional gas. In NR experiments, Xe and $O_2$ were used for neutralization and reionization, respectively. In both cases the pressure of Xe in the first collision cell of the second field-free region was adjusted to give a main ion beam transmission of 90% (90% T) which corresponds to single-collisional conditions. The pressure of $O_2$ in the second collision cell reduced the main ion beam by ca. 15% (85% T).

**Table I. Relative Abundances (%) of Fragment Ions in the Metastable Ion Mass Spectra of $C_{10}H_{10}Rh^+$ Ions Derived from Various Precursors**

| | precursor molecule | | |
|---|---|---|---|
| ion | 1c | 4 | 5 |
| $C_{10}H_9Rh^+$ | 63 | 51 | 57 |
| $C_{10}H_8Rh^+$ | 4 | 6 | 5 |
| $C_9H_nRh^+$ | 1 | 2 | 2 |
| $C_8H_9Rh^+$ | 12 | 5 | 6 |
| $C_8H_8Rh^+$ | 100 | 100 | 100 |
| $C_7H_7Rh^+$ | <1 | <1 | 1 |
| $C_5H_5Rh^+$ | 87 | 93 | 59 |
| $Rh^+$ | <1 | 1 | *a* |

[a] Was not measured.

## Results and Discussion

(Norbornadiene)(cyclopentadienyl)rhodium complexes **1a–c** were used as precursors for the corresponding rhodocenium ions. The molecular ions of these compounds decompose with elimination of ($R^\bullet$ + $C_2H_2$) and ($H^\bullet$ + $C_2H_2$), producing $C_{10}H_{10}Rh^+$ and $C_{10}H_9RRh^+$ ions, respectively (Scheme I).[15] That these ions indeed have bis(cyclopentadienyl) structures is proposed from the following observations.

**$C_{10}H_{10}Rh^+$ Ions (2a).** The MI and CA mass spectra of these ions derived from **1c** were compared with the spectra of $C_{10}R_{10}Rh^+$ ions derived from compounds which contain only cyclopentadienyl hydrocarbon ligands, e.g. $Cp_4Rh_4(CO)_2$ (**3**). The molecular ion of this latter complex decomposes with formation of $Cp_2Rh^+$ (*m/z* 233) ions in a single step.[16] The fragmentation pathways of these ions involve rupture of the metal–cyclopentadienyl bond, destruction of the cyclopentadienyl ring by the elimination of neutral $C_nH_m$ fragments, and dehydrogenation processes. The relative abundances of corresponding fragment ions in the MI mass spectra are very close for all compounds (Table I). Moreover, they are in excellent agreement with the MI mass spectrum of ion **2a**, originating from $CpRh(CO)_2$ (**4**) which contains one cyclopentadienyl ring[17] (Table I). The CA mass spectra are characterized by somewhat higher (but not significant) differences in relative abundances of fragment ions, which, as for the MI spectra, may arise from differences in the internal energies of ions produced from different sources.

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(4) (a) Wesdemiotis, C.; McLafferty, F. W. *Chem. Rev.* **1987**, *87*, 405. (b) Terlouw, J. K.; Schwarz, H. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 805. (c) Hop, C. E. C. A., Ph.D. Thesis, University of Utrecht, 1988. (d) Holmes, J. L. *Mass Spectrom. Rev.* **1989**, *8*, 513.

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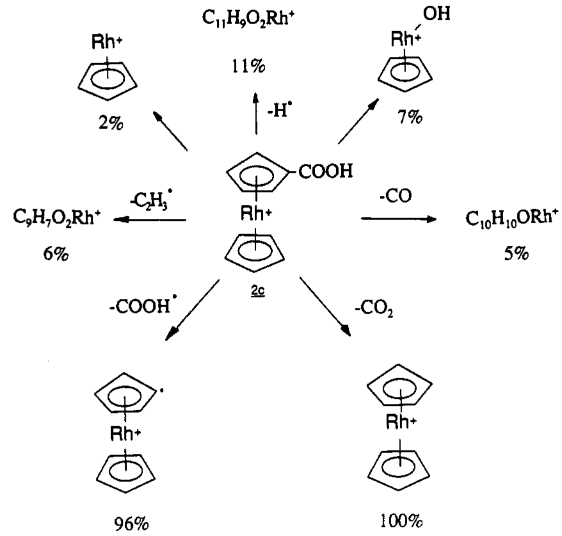
(16) This is confirmed by measurement of the MI mass spectrum of the molecular ions $Cp_4Rh_4(CO)_2^+$ (*m/z* 728).

(17) $Cp_2Rh^+$ ions are present in the high-pressure mass spectrum of $CpRh(CO)_2$ (4). The origin of these ions is not clear. Two types of ion–molecule reactions leading to $Cp_2Rh^+$ ions can be proposed. The first includes the reaction of $CpRh(CO)_n^+$ ions with neutral molecules of cyclopentadiene, which are formed by thermal or hydrolytic decomposition of complex 4 in the ion source. Note that similar ion–molecule reactions with the formation of (cyclopentadienyl)(fluorene)rhodium cations have been observed when fluorene or its 9-substituted derivatives were evaporated into the ion source simultaneously with $CpRh(CO)_2$. The second possible process is the reaction of rhodium-containing ions with neutral parent molecules. The formation of intermediate $Cp_2Rh_2^+$ ions could be proposed because the decomposition of $Cp_2MM'^+$ ions for other transition metals, with elimination of the metal atom, is known to proceed easily (see, for example: Jacobson, D. B.; Freiser, B. S. *Organometallics* **1985**, *4*, 1048).

**Scheme II. Decomposition Pathways of Metastable Rhodocenium Carboxylic Acid Ions (2c)**



We conclude that ions **2a** derived from molecular ions of **1** are structurally identical with the rhodocenium cation.

**$C_{10}H_9(CH_2OH)Rh^+$ Ions (2b).** The bis(cyclopentadienyl) structure of these ions is deduced from a comparison of the behavior of ions derived from two isomeric compounds, **1b** and **5b**, which contain a hydroxymethyl substituent in the norbornadiene and cyclopentadienyl ligands, respectively. No significant differences in MI mass spectra were observed for the above ions other than the higher relative abundances for loss of H, CO, and HCO in the case of **5b**. The CA mass spectra of these ions are also closely similar. Apart from the processes mentioned above, fragmentation of these ions leads to elimination of OH and to the formation of $CpRh^+$ and $CpRhOH^+$ ions. It should be emphasized that all these reactions are characteristic of the well-known (hydroxymethyl)ferrocene molecular ions.[18] The MI mass spectrum of the last compound[19] is characterized by the reduced abundance of substituent decomposition processes relative to the various metal–ligand bond dissociation reactions.

**$C_{10}H_9(COOH)Rh^+$ Ions (2c).** There were no suitable model compounds for confirming the structure of this ion, but two observations support the substituted rhodocenium structure. First, these ions can be produced by reactions similar to those discussed above, and moreover their fragmentation pathways (Scheme II) resemble qualitatively the decomposition of ionized ferrocenecarboxylic acid.[18b,20] Similar to the hydroxymethyl derivatives described above, the MI mass spectrum of $CpFe^+C_5H_4COOH$ ions[21] contains abundant peaks due to metal–ligand bond cleavage, whereas the MI mass spectrum of the corresponding rhodocenium ion is characterized by the high abundance of fragments arising from dissociations involving the substituents.

Thus, mixed (norbornadiene)(cyclopentadienyl)rhodium complexes can indeed be used as suitable precursors for substituted rhodocenium ions, whose behavior qualitatively resembles that for the corresponding ferrocenium ions.

Mass-selected ions **2a–c** were neutralized and reionized in the second field-free region of the mass spectrometer (see Experimental Section). The NR mass spectrum of $Cp_2Rh^+$ ions contains a weak but significant recovery signal at *m/z* 233 (intensity ca. 5% of the base peak in the NR mass spectrum, $Rh^+$), showing that neutral rhodocene is stable not only in the condensed media[2] but also in the gas phase. The overall cross section for the NR process was low and may be attributed to the significant endothermicity of the electron transfer from Xe (IE = 12.1 eV[22]) to the $Cp_2Rh^+$ ion (EI of $Cp_2M$ species are all less than 8 eV[22]). The relative abundances of fragment ion peaks are not listed here; the chief purpose of the experiment was to establish the (transient) formation of the neutral $Cp_2Rh$ species. The NR mass spectrum of $Cp_2Rh^+$ ions also shows rhodium-containing ions ($C_8H_8Rh^+$, $CpRh^+$, $C_3H_3Rh^+$, $CRh^+$, $Rh^+$) and hydrocarbon ions ($C_5H_5^+$, $C_5H_3^+$, $C_4H_n^+$, $C_3H_n^+$, $C_2H_n^+$). These ions can be produced by three main reaction types, which are characteristic of neutralization-reionization experiments:[4,7] (i) reionization of neutrals formed in the dissociation of ionic precursors in the first collision cell, (ii) reionization of neutral fragments formed after neutralization of the mass selected ions and (iii) decomposition of excited ions after reionization.

Hydrocarbon ions can be produced by all three processes listed above. The formation of metal-containing ions can easily be rationalized; for example, $Rh^+$ ions are very weak in the MI mass spectrum of $Cp_2Rh^+$ and have low abundance in CA mass spectra as well. We have observed that the NR cross section for $Rh^+$ ions is lower than that for organic ions. We propose that the $Rh^+$ ions observed in the NR spectrum of $Cp_2Rh^+$ arise largely from the decomposition of neutral $Cp_2Rh^•$ to $Cp^•$ + CpRh and the dissociative reionization of CpRh rather than from the reionization of Rh atoms. The latter route was favored by Drewello and Schwarz[8] in their study of $CpM^+$ ions (M = Fe, Co, Ni).

The NR mass spectrum of $CpRhC_5H_4COOH^+$ ions, as well as that of unsubstituted rhodocenium, contains a recovery signal (intensity ca. 10% of the base peak, $Rh^+$), showing that the corresponding neutral species, rhodocenecarboxylic acid, is also stable in the gas phase. This is the first observation of a monosubstituted rhodocene. Interpretation of the NR mass spectra of the substituted rhodoceniums is more difficult; nevertheless, the appearance of $CpRhOH^+$ ions may result from dissociation of reionized $CpRhC_5H_4COOH^+$ ions rather than from reionized $CpRhOH^+$, because the latter only display $Rh^+$ ions in their NR mass spectrum.

We have not observed a recovery signal for the reionization of (hydroxymethyl)rhodocenium (**2b**). The lower stability of these species in NR processes compared with, for example, the carboxylic analogue, can be explained by the more facile interaction between the hydroxy substituent bonded to the methylene group and the metal atom, relative to that of OH connected to the carbonyl group. For the latter, conjugation with the π-system of the ligand leads to geometric restriction. Thus, OH migration to the positively charged metal atom after reionization of the corresponding neutral is proposed to proceed more easily in the case of **2b**.[23]

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[18] (a) Egger, H. *Monatsh. Chem.* **1966**, *97*, 608. (b) Zagorevskii, D. V. Ph.D. Thesis, Institute of Organo-Element Compounds, Moscow, 1981.

[19] MI mass spectrum of $CpFe^+C_5H_4CH_2OH$ (neutral loss, relative intensity): H, 38; $H_2$, 2; $H_2O$, 70; $CH_2O$, 89; $C_5H_4CH_2$, 100; $C_5H_4CH_2OH$, 1.

[20] Mandelbaum, A.; Cais, M. *Tetrahedron Lett.* **1964**, 3847.

[21] MI mass spectrum of $CpFe^+C_5H_4COOH$ (neutral loss, relative intensity): H, 4; $H_2$, 3; CO, 1; $CO_2$, 1; COOH, 4; Cp, 3; $C_5H_4CO$, 100.

[22] Lias, S. G.; Bartmess, J. E.; Liebman, J. F.; Holmes, J. L.; Levin, R. D.; Mallard, W. G. Gas Phase Ion and Neutral Thermochemistry. *J. Phys. Chem. Ref. Data* **1988**, *17*, Suppl. 1.

[23] Similar results have been observed for substituted cyclopentadienyl nido clusters of manganese, $RXC_5H_4Mn^+$. Replacing X = CO by X = $CH_2$ always led to easier migration of R to Mn with isomerization of these ions into $R–Mn^+–C_5H_4X$ structures (Sizoi, V. F.; Nekrasov, Yu. S. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1982**, 285. Zagorevskii, D. V.; Nekrasov, Yu. S. *J. Organomet. Chem.* **1984**, *267*, 121.

In conclusion, neutral rhodocene and some derivatives can be obtained as stable gas-phase species (with lifetimes ~$10^{-6}$ s) by neutralization–reionization mass spectrometry. Various cyclopentadienyl complexes of rhodium can be used for generation of these species and other rhodocene derivatives in the gas phase. In addition to the compounds studied in this work, some other rhodium complexes, (e.g. $(RC_5H_4)_2RhL_n$[24] and (cyclopentadienyl)(cyclopentadiene)rhodium derivatives[3a,c,25]), which decompose easily to $(RC_5H_4)_2Rh^+$ ions, are good sources for gas-phase substituted-rhodocenium ions. It is quite possible that rhodocenium salts,[25a] like cobaltocenium[26] derivatives, will prove to be convenient sources for the corresponding gas-phase cations and their neutral counterparts.

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# Selective Formation of *cis*-$(HMe_2Si)_2Pt(PEt_3)_2$ from $HMe_2SiSiMe_2H$ and $Pt(PEt_3)_3$ and Its Reactivities Relevant to Generation of (Silylene)platinum Species Thereof

Hiroshi Yamashita, Masato Tanaka,* and Midori Goto

*National Chemical Laboratory for Industry, Tsukuba, Ibaraki 305, Japan*

*Received May 1, 1992*

$HMe_2SiSiMe_2H$ (1, 3 equiv) reacted with $Pt(PEt_3)_3$ at room temperature to give *cis*-$(HMe_2Si)_2Pt(PEt_3)_2$ (2) and oligosilanes $H(SiMe_2)_nH$ ($1 \le n \le 7$), while 2 was selectively obtained by the reaction of 1 (1 equiv) with $Pt(PEt_3)_3$ at −30 °C. $HMe_2SiSiMe_3$ or $H_2MeSiSiMe_3$ also afforded oligomeric redistribution products when treated with $Pt(PEt_3)_3$ at room temperature, although Si–Pi species were not found. Redistribution of 1 was efficiently catalyzed by 2 at room temperature, leading to $H(SiMe_2)_nH$ with the approximate molar ratio being ≥30:10:30:5:2:1:0.3 for $n$ = 1–7, respectively. $Me_2SiH_2$ was mainly formed upon thermolysis of 2 at 60 °C. Reaction of 2 with phenylacetylene (3 equiv) at 60 °C gave 4,4-dimethyl-2,6-diphenyl-1-platina-4-sila-2,5-cyclohexadiene (4), the structure of which was determined by X-ray crystallographic analysis: space group $P2_1/n$, $a$ = 12.319 (1) Å, $b$ = 20.164 (1) Å, $c$ = 12.837 (2) Å, $\beta$ = 97.03 (1)°, $V$ = 3164.8 (7) $Å^3$, $Z$ = 4. Heating 4 at 120 °C afforded 1,1-dimethyl-3,4-diphenyl-1-sila-2,4-cyclopentadiene as the sole volatile product.

## Introduction

Addition of a Si–H or Si–Si bond to organic unsaturated compounds is a versatile reaction which allows us to transform various organosilanes including polymers.[1,2] Group 10 metal-catalyzed reactions of hydrodisilanes that have both Si–H and Si–Si bonds are particularly interesting since they can lead to unique products such as oligosilanes,[3a] 1,4-disila-2,5-cyclohexadiene,[3b] and 1-sila-2,4-cyclopentadiene,[3c,d] although mechanistic details have not been elucidated yet. West et al. recently reported that 1,2-dihydrosilanes reacted with (diphosphine)Pt(0) complexes to give (disilene)platinum species, and they proposed a pathway involving hydrido(disilanyl)platinum intermediates.[4] In striking contrast with this, we have found that $HMe_2SiSiMe_2H$ reacts with $Pt(PEt_3)_3$ to give a bis(hydrosilyl)platinum complex selectively.[5] Herein are reported the formation and reactivities of the bis(silyl) complex.

## Results and Discussion

**Reactions of $Pt(PEt_3)_3$ with Hydrodisilanes.** When $HMe_2SiSiMe_2H$ (1, 3 equiv) was added to $Pt(PEt_3)_3$ in benzene-$d_6$ at room temperature, the color of the solution immediately turned from orange to pale yellow. $^1H$ NMR, GC, and GC–MS of the solution showed the formation of *cis*-$(HMe_2Si)_2Pt(PEt_3)_2$ (2, nearly quantitative yield) along with $H(SiMe_2)_nH$ ($1 \le n \le 7$).[3a,6] The approximate rel-

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(1) For a recent review of hydrosilylation, see: Ojima, I. In *The Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; John Wiley & Sons: Chichester, U.K., 1989; p 1479.

(2) For recent studies on double silylation, see: (a) Yamashita, H.; Catellani, M.; Tanaka, M. *Chem. Lett.* **1991**, 241 and references cited therein. (b) Murakami, M.; Andersson, P. G.; Suginome, M.; Ito, Y. *J. Am. Chem. Soc.* **1991**, *113*, 3987 and references cited therein.

(3) (a) Yamamoto, K.; Okinoshima, H.; Kumada, M. *J. Organomet. Chem.* **1970**, *23*, C7. (b) Yamamoto, K.; Okinoshima, H.; Kumada, M. *J. Organomet. Chem.* **1971**, *27*, C31. (c) Okinoshima, H.; Yamamoto, K.; Kumada, M. *J. Am. Chem. Soc.* **1972**, *94*, 9263. (d) Okinoshima, H.; Yamamoto, K.; Kumada, M. *J. Organomet. Chem.* **1975**, *86*, C27.

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(5) For our recent studies on the reactions of organosilanes with group 10 and/or 9 metal complexes, see: (a) Yamashita, H.; Kobayashi, T.-a.; Hayashi, T.; Tanaka, M. *Chem. Lett.* **1990**, 1447. (b) Yamashita, H.; Kawamoto, A. M.; Tanaka, M.; Goto, M. *Chem. Lett.* **1990**, 2107 and references cited therein.

(6) Heating a mixture of 1 (0.178 mmol) and *trans*-$PtCl_2(PEt_3)_2$ (1 mol %) at 90 °C for 30 min in a sealed tube provided oligosilanes which were assigned by GC and GC–MS to be $H_2Me_{2n}Si_n$ ($1 \le n \le 9$). Their approximate amounts were estimated by GC at 5, 4, 3, 4, 5, 2, 0.3, and ≤0.1 × 0.00178 mmol for $n$ = 2–9, respectively (the amount of $Me_2SiH_2$ was not determined). Except for the case of the tetrasilane, one major GC peak, probably arising from $H(SiMe_2)_nH$,[3a] was observed for each oligosilane. However, the tetrasilane seemed to be a mixture of two structural isomers (3:7) as described for the reaction of $Pt(PEt_3)_3$ with 1 in the Experimental Section.

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