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Notes

Si−C Bond Activation of $ArMe_2SiOH$ Promoted by a Bromoplatinum(II) Complex and Ag_2O. Aryl Group Transfer from Silicon to Platinum

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*Summary: $[PtBr(PEt_3)_3]BF_4$ (**1**) reacts with an excess of dimethyl{4-(trifluoromethyl)phenyl}silanol in the presence of Ag_2O and 4 Å molecular sieves at 60 °C to afford trans-$Pt(C_6H_4CF_3\text{-}4)_2(PEt_3)_2$ (**2**). The reaction without molecular sieves and that with addition of a small amount of water give a mixture of **2** and $[Pt_2(\mu\text{-}OH)_2(PEt_3)_4](BF_4)_2$ (**3**). The organic products of the reaction are composed of $ArSiMe_2-O-(SiMe_2-O)_n-SiMe_2Ar$ (Ar = $C_6H_4CF_3$-4, n = 0, 1, 2) and cyclooligodimethylsiloxanes in lesser amounts. The obtained complexes were characterized by NMR spectroscopy and X-ray crystallography.*

Introduction

Transition metal complex promoted Si−C bond activation is attractive because it leads to the formation of new transition metal−carbon bonds under mild conditions; however, the number of reports on this subject is small, partly due to the large bond dissociation energy (311 kJ mol^{-1})[1] required for activation. An organosilyl group bonded to metal center(s) of mono- and dinuclear transition metal complexes can undergo intramolecular Si−C bond cleavage,[2−8] a process that has been reported more frequently than intermolecular Si−C bond activation by transition metal complexes. Oxidative addition of a Si−C(sp) bond of trialkyl(alkynyl)silane to coordinatively unsaturated Pt(0),[9] Rh(I),[10] and Ru(0)[11] complexes occurs selectively to afford complexes with metal−alkynyl carbon and/or metal−silicon bonds.

These reactions appear to be related to the mechanism involved in transition metal-catalyzed synthetic organic reactions involving the cleavage of Si−C(sp) bonds.[12,13] The cleavage of an Si−C bond is also involved in a number of coupling reactions of chlorosilanes[14,15] and fluorosilicates[16,17] with aryl halides. Recently, we found that a Pd complex-catalyzed cross-coupling reaction of dimethyl(aryl)silanol with iodoarenes occurs in the presence of Ag_2O to give biaryls.[18] Scheme 1 depicts

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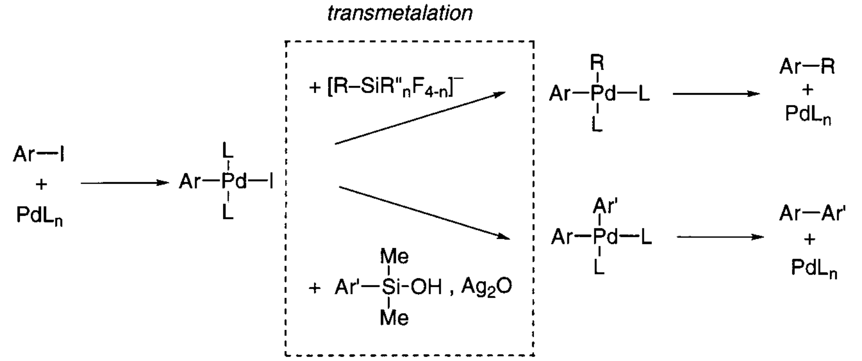
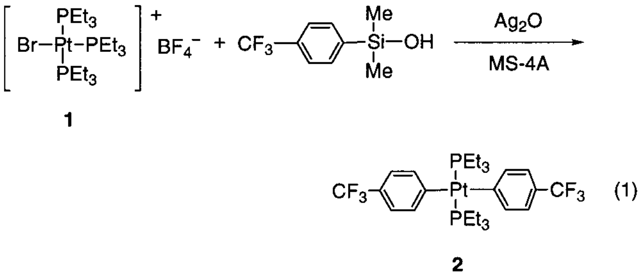
Scheme 1



a generally accepted mechanism for these cross-coupling reactions that involve the transfer of the silicon-bonded organic group to the arylpalladium intermediate. There have been only a few reports[19] that provide direct evidence for the formation of transition metal–carbon bonds via the transmetalation of such Si-containing compounds. We have examined the reaction of silanols with several group 10 metal complexes and report herein the transmetalation of dimethyl(aryl)silanol to a bromoplatinum(II) complex in the presence of Ag_2O to give a diarylplatinum complex.

Results and Discussion

The reaction of dimethyl{4-(trifluoromethyl)phenyl}-silanol with $[PtBr(PEt_3)_3]BF_4$ (**1**)[20] in 8:1 molar ratio in the presence of Ag_2O and 4 Å molecular sieves (30 h at 60 °C) in anhydrous acetone produces *trans*-$Pt(C_6H_4\text{-}CF_3\text{-}4)_2(PEt_3)_2$ (**2**) in 56% isolated yield (eq 1). NMR analysis of the reaction mixture indicated that **2** is the main product (~80% yield). The reaction in 2:1 molar ratio produces **2** in lower yield (24%) probably due to accompanying dehydration of the silanol to the siloxane, which does not react further with the Pt complex (vide infra). Figure 1 shows the molecular structure of **2** as determined by X-ray crystallography. The molecule has crystallographic C_2 symmetry around the metal center and C_m symmetry at the coordination plane. The square-planar coordination with two aryl and phosphine ligands at *trans* positions is dominant also in solution. The $^{31}P\{^1H\}$ NMR spectrum of **2** in benzene-d_6 consists of a singlet at δ 8.43 with a coupling constant $^1J_{PtP} = 2750$ Hz, which is typical of Pt(II) complexes possessing two *trans* phosphine ligands. The $^{13}C\{^1H\}$ NMR signal of the PCH_2 carbon at δ 13.9 appears as an apparent triplet due to virtual coupling. The *ipso* carbon signal of the aryl ligand is a triplet (δ 169.4) with $J(PC) = 11$ Hz. These NMR data are similar to those of the previously reported *trans*-$PtAr_2(PBu_3)_2$ and *trans*-$PtAr_2(PPh_3)_2$.[21]



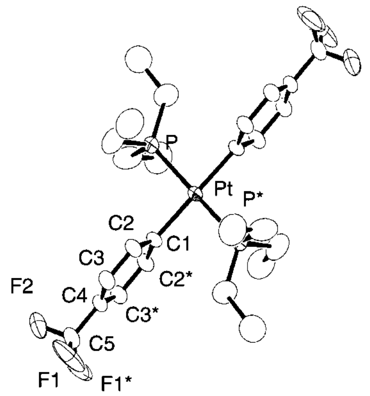
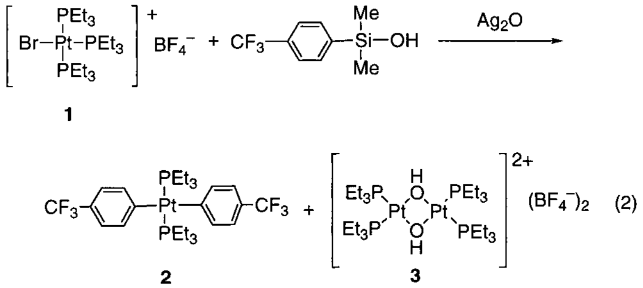


Figure 1. Structure of complex **2** determined by X-ray crystallography with 50% thermal ellipsoidal plotting. The molecule has crystallographic C_2 and C_m symmetry within the molecule. Atoms with asterisks are crystallographically equivalent to those having the same number without asterisks. Selected bond lengths (Å) and angles (deg): Pt1–C1 2.089(8), Pt1–P1 2.287(3), P1–Pt1–C1 89.7(3), P1–Pt1–C1* = 90.3(3).

The reaction without addition of 4 Å molecular sieves gives a mixture of **2** and $[Pt_2(\mu\text{-}OH)_2(PEt_3)_4](BF_4)_2$ (**3**) in 51% and 29% yields, respectively (eq 2). Complex **3** was identified by comparison of the spectroscopic and



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[19] Triorganosilyl group tethered to a Pd(II) complex reacts with TASF ($(Me_2N)_3S^+SiMe_3F_2^-$), leading to Si–C bond cleavage and metalation of the alkyl group. See: Mateo, C.; Fernádez-Rivas, C.; Echavarren, A. M.; Cádenas, D. J. *Organometallics* **1997**, *16*, 1997. Mateo, C.; Fernádez-Rivas, C.; Cádenas, D. J.; Echavarren, A. M. *Organometallics* **1998**, *17*, 1998.

[20] $[PtBr(PEt_3)_3]BF_4$ was prepared by a slight modification of the preparation of analogous $[PtF(PEt_3)_3]BF_4$ and $[PtCl(PEt_3)_3]BF_4$. See: Cairns, M. A.; Dixon, K. R.; McFarland, J. J. *J. Chem. Soc., Dalton Trans.* **1975**, 1159.

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crystallographic data of the product with those previously reported.[22] The addition of a small amount of water ($[H_2O]:[Pt] = 7.5:1$) changes the yields of the products and leads to the formation of **2** and **3** in 8% and 57% yields, respectively. Thus, the source of OH groups in **3** is the water contained in the reaction mixture.

The organic products in the above reactions contain $ArSiMe_2{-}O{-}(SiMe_2{-}O)_n{-}SiMe_2Ar$ ($Ar = C_6H_4CF_3$-4, $n = 0, 1, 2$), octamethyltetracyclosiloxane, and decamethylpentacyclosiloxane, which were characterized by NMR (^1H and ^{29}Si) spectroscopy and GC−MS measurement of the reaction mixture. Consumption of the silanol and formation of $ArSiMe_2{-}O{-}SiMe_2Ar$ during reaction 1 suggests that a simple dehydration of the silanol to the siloxane takes place independently from the aryl ligand transfer.[23] Formation of the hydroxoplatinum complex **3** can be attributed to the reaction of water generated in the dehydration with a platinum complex in the reaction mixture. Heating of a mixture of the silanol, **1**, and $AgBF_4$ in acetone does not give **2**, but results in the exclusive formation of **3**. These results suggest that activation of the Si−C bond in the reactions 1 and 2 is induced by Ag_2O rather than by BF_4^- anion, although the anion was reported to be able to fluorinate organosilyl compounds to give fluorosilanes or fluorosilicates which are susceptible to Si−C bond cleavage promoted by late transition metal complexes.[24]

This work demonstrates the formation of aryl−platinum bonds via transmetalation of an arylsilanol to a cationic platinum complex in the presence of Ag_2O. The selective cleavage of a Si−C(aryl) bond of silanol and the intermolecular transfer of the aryl group to a group 10 metal center were postulated to account for the results of Pd-catalyzed coupling reactions and have been realized by the present study.

Experimental Section

General Consideration, Measurement, and Materials. All manipulations of the Pt complexes were carried out under nitrogen or argon using standard Schlenk techniques unless otherwise stated. Glassware was dried in the oven and heated under reduced pressure by a heat gun before use. Acetone was dried over 4 Å molecular sieves, which were preactivated by heating at 160 °C for 6 h in vacuo. Silver(I) oxide purchased from Wako Pure Chemical Ind., Ltd. and $AgBF_4$ purchased from Aldrich Chemical Co., Inc. were dried at 160 °C for 3 h and 60 °C for 1 h in vacuo, respectively, and stored under Ar atmosphere. NMR spectra (^1H, $^{13}C\{^1H\}$, $^{31}P\{^1H\}$, and $^{19}F\{^1H\}$) were recorded on Varian 300 and JEOL EX-400 spectrometers. Chemical shifts were referenced to external $SiMe_4$ (^1H and $^{13}C\{^1H\}$), 85% H_3PO_4 ($^{31}P\{^1H\}$), and $CFCl_3$ ($^{19}F\{^1H\}$). GC−MS analysis was carried out on a Shimadzu QP-5000 mass spectrometer. Elemental analyses were carried out with a Yanaco MT-5 CHN autocorder. The silanol was prepared according to the literature.[25]

The preparation of **1** was carried out by slight modification of the reported preparation method of analogous complexes as follows.[20] To an acetone (10 mL) solution of $PtBr_2(PEt_3)_2$ (355 mg, 0.50 mmol) was added $AgBF_4$ (97 mg, 0.50 mmol) in one portion, which caused separation of a gray solid of AgBr from the pale yellow solution. After stirring of the mixture for 1 h, the solid product was removed by filtration. Solvent was reduced to 3−4 mL at room temperature under vacuum. Addition of Et_2O (10 mL) and subsequent cooling of the mixture to below −10 °C for 24 h gave **1** as colorless crystals, which were collected by filtration and dried in vacuo (228 mg, 64%). ^1H NMR (300 MHz, benzene-d_6): δ 0.90 (apparent quintet due to virtual coupling, 18 H, CH_3), 1.17 (dt, 9H, CH_3, $J(PH) = 15$ Hz, $J(HH) = 8$ Hz), 1.96 (m, 12H, PCH_2), 2.17 (m, 6H, PCH_2). $^{31}P\{^1H\}$ NMR (121.5 MHz, benzene-d_6): δ 14.02 (d, $J(PP) = 18$ Hz, $J(PPt) = 2270$ Hz), 13.00 (t, $J(PP) = 18$ Hz, $J(PPt) = 3410$ Hz). Anal. Calcd for $C_{18}H_{45}BBrF_4P_3Pt$: C, 30.18; H, 6.33; Br, 11.16. Found: C, 29.99; H, 6.29; Br, 11.27.

Reaction of $SiMe_2(C_6H_4CF_3$-4)OH with 1 in the Presence of Ag_2O. $SiMe_2(C_6H_4CF_3$-4)OH (441 mg, 2.0 mmol) was added to a mixture of Ag_2O (58 mg, 0.25 mmol), **1** (179 mg, 0.25 mmol), and 4 Å molecular sieves (500 mg, 25 particles) in dried acetone (10 mL) at room temperature. After several pump-and-thaw cycles, heating of the reaction mixture with an oil bath was continued for 30 h at 60 °C under Ar. After removing a gray solid by filtration and washing the solid with acetone, the combined filtrate and washings were analyzed by NMR spectroscopy. The $^{31}P\{^1H\}$ NMR spectrum in acetone-d_6 showed the existence of **2** as the major Pt complex. The ^1H NMR spectrum contains signals at δ 0.42−0.37 and 0.14−0.05 in a 77:23 peak area ratio and are assigned to the methyl hydrogens of the $-OSiMe_2(C_6H_4CF_3$-4) group and of the $-OSiMe_2O-$ group of the organic products, respectively. The $^{29}Si\{^1H\}$ NMR spectra are also consistent with this observation.[26] The Pt complex was extracted with hexane (20 mL) from the product and subsequent Celite filtration of the hexane solution. Concentration of the hexane extract to 3−4 mL and keeping the resulting solution at −10 °C for 24 h led to separation of **2** as colorless crystals. The crystals of **2** collected by filtration and the second crop of the crystals obtained from the filtrate were combined (101 mg, 56%).

A similar reaction of an acetone (8 mL) solution of **1** (287 mg, 0.40 mmol) with $SiMe_2(C_6H_4CF_3$-4)OH (176 mg, 0.80 mmol) in the presence of Ag_2O (92 mg, 0.40 mmol) but without 4 Å molecular sieves gave **2** (51%) and $[Pt_2(\mu\text{-}OH)_2(PEt_3)_4](BF_4)_2$ (**3**). Recrystallization of the hexane-insoluble fraction of the product from CH_2Cl_2−hexane afforded **3** as colorless crystals (62 mg, 29%). GC−MS analysis of the solution showed the presence of $ArSiMe_2{-}O{-}(SiMe_2{-}O)_n{-}Ar$ ($Ar = C_6H_4CF_3$-4 $n = 0, 1, 2$) and octamethylcyclotetrasiloxane (EIMS m/z 281 $(M - Me)^+$) and decamethylcyclopentasiloxane (m/z 355 $(M - Me)^+$) in a much smaller amount. The reaction in the presence of added water (50 μL, $[H_2O]:[Pt] = 7.5:1$) resulted in formation of **2** and **3** in 8% and 57% yields, respectively.

Data for 2. $^{31}P\{^1H\}$ NMR (121.5 MHz, benzene-d_6): δ 8.43 (s, $^1J(PtP) = 2750$ Hz). ^1H NMR (300 MHz, benzene-d_6): δ 0.71 (dt, 9 H, $^3J(PH) = 16$ Hz, $^3J(HH) = 8$ Hz, PCH_2CH_3), 1.00 (m, 6 H, PCH_2CH_3), 7.46 (d, 4 H, $^3J(HH) = 8$ Hz, Pt−C_6H_4-m), 7.63 (d, 4 H, $^3J(HH) = 8$ Hz, $^3J(PtH) = 33$ Hz, Pt−C_6H_4-o). $^{13}C\{^1H\}$ NMR (75.3 MHz, benzene-d_6): δ 7.7 (s, $^3J(PtC) = 25$ Hz, PCH_2CH_3), 13.9 (apparent triplet due to virtual coupling, $^{1+3}J(PC) = 34$ Hz, $^2J(PtC) = 70$ Hz, PCH_2), 123.4 (q, $^3J(FC) = 3$ Hz, $^3J(PtC) = 44$ Hz, Pt−C_6H_4-m), 124.4 (q, $^2J(FC) = 31$ Hz, Pt−C_6H_4-p), 126.3 (q, $^1J(FC) = 270$ Hz, CF_3), 139.8 (t, $^2J(PtC) = 21$ Hz, Pt−C_6H_4-o), 169.4 (t, $^2J(PC) = 11$ Hz, $^1J(PtC) = 575$ Hz, Pt−C). $^{19}F\{^1H\}$ NMR (188.2 MHz, benzene-

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d_6): δ −61.5 (s). Anal. Calcd for $C_{26}H_{38}F_6P_2Pt$: C, 43.28; H, 5.31; F, 15.80. Found: C, 43.45; H, 5.37; F, 15.54.

Data for 3. $^{31}P\{^1H\}$ NMR (121.5 MHz, $CDCl_3$): δ 6.87 (s, ^1J(PtP) = 3475 Hz). ^1H NMR (300 MHz, $CDCl_3$): δ 1.22 (dt, 36 H, ^3J(PH) = 18 Hz, ^3J(HH) = 7 Hz, PCH_2CH_3), 1.83 (m, 24 H, PCH_2CH_3), 2.17 (s, 2 H, OH). Anal. Calcd for $C_{24}H_{62}B_2F_8O_2P_4Pt_2$: C, 26.93; H, 5.84. Found: C, 27.34; H, 5.41.

Crystal Structure Determination. Crystals of **2** were mounted in glass capillary tubes under Ar. Intensities were collected for Lorentz and polarization effects on a Rigaku AFC-7R automated four-cycle diffractometer by using Mo Ka radiation (λ = 0.71069 Å) and the $\omega-2\theta$ scan method, and an empirical absorption correction (Ψ scan) was applied. Calculations were carried out by using the program package teXsan for Windows. Atomic scattering factors were obtained from the literature. A full-matrix least-squares refinement was used for non-hydrogen atoms with anisotoropic thermal parameters. Hydrogen atoms were located by assuming the ideal geometry and were included in the structure calculation without further refinement of the parameters.[27] Crystal data for **2**: $C_{26}H_{38}F_6P_2Pt$, 721.61, colorless, 0.30 × 0.25 × 0.05 mm; monoclinic, space group $C2/m$ (No. 12); a = 16.362(3) Å, b = 9.432(2) Å, c = 10.422(3) Å, β = 109.80(2)°, V = 1513.3(6) Å^3, Z = 2, D_{calcd} = 1.583 g cm^{-3}; 293 K; 2θ range 5−55°; number of independent reflections 1852 (R_{int} = 0.048). The final cycle of full-matrix least-squares refinement on F was based on 1800 observed reflections ($I > 3.00\sigma(I)$) and 92 variable parameters: R = 0.044 and R_w = 0.046; GOF = 3.36.

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Supporting Information Available: Crystallographic results of **2**. This material is available free of charge via the Internet at http://pubs.acs.org.

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(27) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1974; Vol. 4.