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# Reaction of a Symmetrical Diplatinum Complex Containing Bridging $\mu$-$\eta^2$-H−SiH(IMP) Ligands (IMP = 2-Isopropyl-6-methylphenyl) with $PMe_2Ph$. Formation and Characterization of $\{(PhMe_2P)_2Pt[\mu\text{-}SiH(IMP)]\}_2$

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*Summary: A dinuclear Pt−Si complex containing bridging* $\eta^2$*-H−SiH(IMP) (IMP = 2-isopropyl-6-methylphenyl) ligands,* $\{(Ph_3P)Pt[\mu\text{-}\eta^2\text{-}H-SiH(IMP)]\}_2$ *(**1**) has been synthesized and characterized by NMR and X-ray crystallography. A 3c-2e nonclassical interaction between Pt···H···Si is supported by spectroscopic and crystallographic data. Complex **1** reacts with the more basic and less hindered phosphine* $PMe_2Ph$ *to afford a different dinuclear Pt−Si complex with loss of* $H_2$*,* $\{(PhMe_2P)_2Pt[\mu\text{-}SiH(IMP)]\}_2$ *(**2**). Complex **2** was characterized by multinuclear NMR spectroscopy.*

The oxidative addition of a Si−H bond in a hydrosilane to a low-valent transition metal center is a versatile method for the formation of complexes containing a M−Si bond.[1] Reaction of a hydrosilane with a transition metal complex can provide different products, some of which depend on the number of available Si−H bonds in the starting silane. Primary as well as secondary silanes can provide both mono- and bimetallic species (where the silicon center bridges two metal centers). Bimetallic bis($\mu$-silylene) complexes have also been generated by dimerization of metal−silylenes or by reaction of bis(silyl) metal complexes with another metal precursor.[2]

A number of metal systems are known to catalyze transformations of hydrosilanes including hydrosilylation of alkenes and dehydrocoupling to oligo- and polysilanes. The mechanism for dehydrocoupling with early transition metals is believed to proceed through a $\sigma$-bond metathesis reaction,[3] but with the late transition metal catalysts, no mechanism has been clearly established.[4] Mechanistic studies with late transition metal catalysts have not excluded the involvement of dinuclear species in the dehydrocoupling reactions. Diplatinum bis($\mu$-silylene) complexes have been shown to catalyze the oligomerization (stepwise) of primary silanes to small-chain oligosilanes.[5] Recently, a series of dinuclear rhodium ($\mu$-silylene) and ($\mu$-$\eta^2$-silyl) complexes have been prepared, and involvement of the latter in the catalytic dehydrocoupling of diphenylsilane to 1,1,2,2-tetraphenyldisilane was proposed.[6] Despite the growing number of $\mu$-silylene complexes reported, little is known about their reactivity, most of which involve small molecules or catalytic reactions.[2]

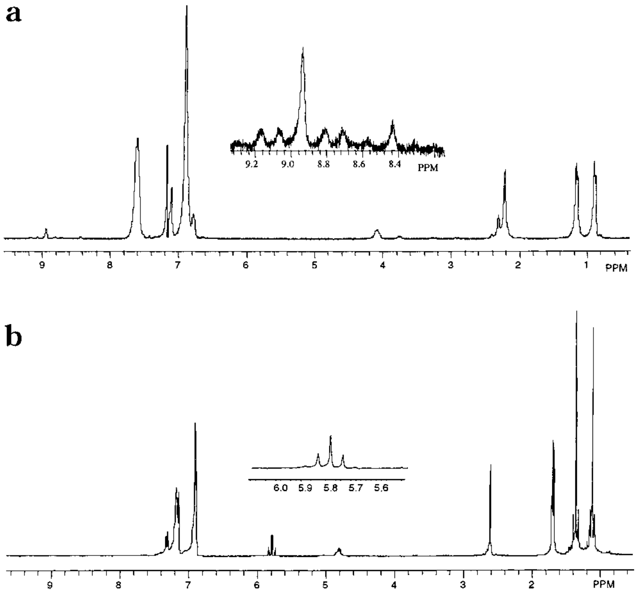

**Figure 1.** (a) $^1H$ NMR spectrum (25 °C) of **1** (300 MHz, $C_6D_6$). The inset shows an expanded view of the terminal Si−H region with $^{195}Pt$ satellites. (b) $^1H\{^{31}P\}$ NMR spectrum (25 °C) of **2** (300 MHz, $C_6D_6$). The inset shows an expanded view of the terminal Si−H region with $^{195}Pt$ satellites.

We report here the preparation and characterization of the dinuclear Pt complex $\{(Ph_3P)Pt[\mu\text{-}\eta^2\text{-}H-SiH(IMP)]\}_2$ (IMP = 2-isopropyl-6-methylphenyl), which contains two nonclassical Pt···H···Si interactions, and its novel reaction with $PMe_2Ph$ to form a different four-membered ring with loss of $H_2$.[7] To the best of our

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[1] Corey, J. Y.; Braddock-Wilking, J. *Chem. Rev.* **1999**, *99*, 175.

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[4] Two pathways have been proposed for the later metals: a series of oxidative addition/reductive elimination steps (Curtis, M. D.; Epstein, P. S. *Adv. Organomet. Chem.* **1981**, *19*, 213) and a route involving metal−silylenes ($L_nM{=}SiRR'$) via a 1,2-hydrogen shift from silicon to the metal center (Ojima, I.; Inaba, S. I.; Kogure, T.; Nagai, Y. *J. Organomet. Chem.* **1973**, *55*, C7). See also ref 3c.

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[6] Rosenberg, L.; Fryzuk, M. D.; Rettig, S. J. *Organometallics* **1999**, *18*, 958.

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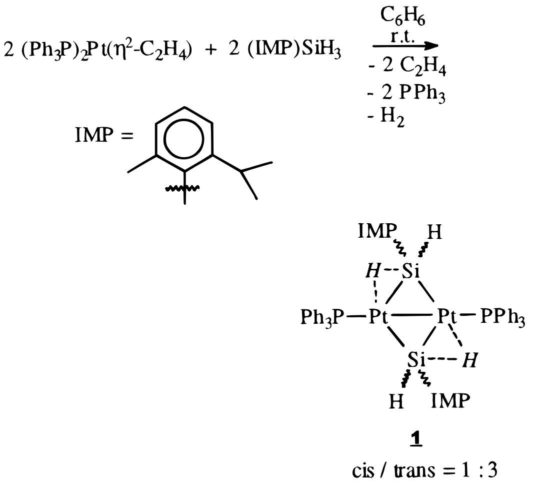
knowledge, this is the first example of the conversion of a dinuclear bis($\mu$-$\eta^2$-H−$SiR_2$) complex to a bis($\mu$-silylene) species. In fact, the reverse reaction of a bridging silylene complex containing no metal−metal bond to a system with a metal−metal bond is fairly common.[2]

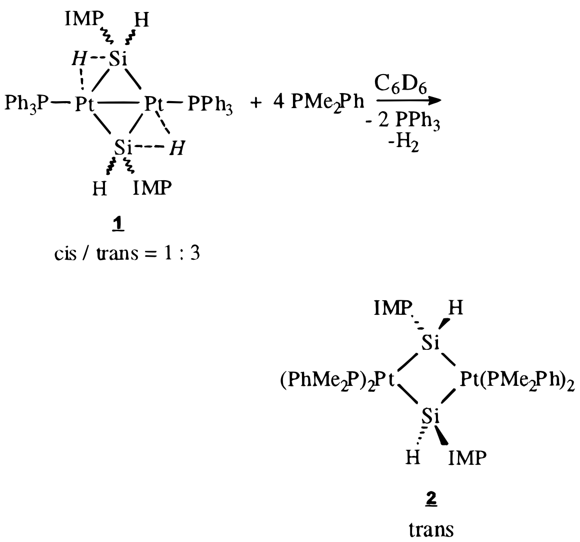
Reaction of $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$ with 1 equiv (or a 4-fold excess) of (IMP)$SiH_3$ in benzene or toluene at room temperature afforded **1** (as a mixture of cis and trans isomers in a ratio of approximately 1:3)[8] in 83% yield (eq 1).[9] The reaction occurs instantly with vigorous gas evolution ($H_2$ and $C_2H_4$) followed by precipitation of the product. This is the first example of a dinuclear Pt−Si system containing both a nonclassical Pt···H···Si interaction and a terminal Si−H unit. Complex **1** was characterized by $^1H$, $^{31}P\{^1H\}$, and $^{29}Si\{^1H\}$ NMR spectroscopy as well as X-ray crystallography (*trans*-**1** only).



(1)

There are only a few complexes of the group 10 metals that contain a nonclassical M···H···Si interaction and include several diplatinum complexes $\{(R'_3P)Pt(\mu\text{-}\eta^2\text{-}H{-}SiR_2)\}_2$ (R′ = alkyl or aryl; R = Me, Ph) prepared by Stone et al.[10] Tessier and co-workers synthesized the novel dinuclear platinum complex $\{(Pr_3P)Pt[\mu\text{-}\eta^2\text{-}H{-}Si(Hex)PtH(PPr_3)_2]\}_2$ (Pr = *n*-propyl, Hex = *n*-hexyl).[11] More recently, Kim and co-workers reported two dinuclear Pd complexes containing $Ph_2Si$ groups bridging the Pd centers, $(Me_3P)Pd(\mu\text{-}\eta^2\text{-}H{-}SiPh_2)_2Pd(PMe_3)_n$ (*n* = 1, 2).[12]

Although stable in the solid state, a benzene solution of **1** is stable only for several days at room temperature or for months at −35 °C. The solid appears to be moderately air stable. When **1** is treated with the more basic, less sterically demanding phosphine, $PhMe_2P$, a new dinuclear Pt−Si complex, *trans*-**2**[13] (with elimination of $H_2$), was formed which no longer contains a Pt···H···Si interaction or a Pt−Pt bond (eq 2).[14] Complex **2** was characterized by multinuclear NMR, X-ray crystallography,[15] and elemental analysis. Several $Pt_2Si_2$ ring systems analogous to **2** have been prepared by Tessier,[14b,c] Fink,[14d] Tilley,[14e] and Tanaka.[14f]



(2)

Compounds **1** and **2** display quite different NMR chemical shifts despite their similar basic structures. For example, the terminal Si−H proton resonances appear at very low field for the cis and trans isomers of **1** (Figure 1a) and are located at 8.42 and 8.92 ppm (flanked by two sets of Pt satellites, which confirms inequivalent coupling of the terminal Si−H to each Pt center). In contrast, the terminal Si−H resonance for **2**

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[8] The trans isomer is believed to be the major component due to steric hindrance within the molecule. The ratio of isomers was determined from the relative intensities of the Si−H resonances in the $^1H$ NMR spectrum.

[9] A benzene solution (2 mL) of (IMP)$SiH_3$ (22 mg, 0.13 mmol) was added slowly to $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$ (97 mg, 0.13 mmol). Vigorous bubbling was seen, and the solution turned golden-yellow. An off-white microcrystalline solid formed and was washed with 3 aliquots of $C_6H_6$ (1 mL each) and then dried in vacuo to give 67 mg (83%) of **1** as a mixture of cis and trans isomers. An alternative preparation of **1** from $(Ph_3P)_4Pt$ and (IMP)$SiH_3$ gave 70% yield and much lower yield from $(Ph_3P)_2PtMe_2$. Spectroscopic and physical data for **1**: $^1H$ NMR (300 MHz, $\delta$) 0.89 (br d, 12H, $^3J_{H-H}$ = 5.7 Hz, CH(C*H*$_3$)$_2$, cis and trans resonances overlapping), 1.16 (br d, 12H, $^3J_{H-H}$ = 6.3 Hz, CH(C*H*$_3$)$_2$, cis and trans resonances overlapping), 2.22 (s, 6H, *o*-$CH_3$ for *trans*-**1**; overlapping with Pt···*H*···Si), 2.31 (s, 6H, *o*-$CH_3$ for *cis*-**1**; overlapping with Pt···*H*···Si), 3.75 (br s, 2H, CH for *cis*-**1**), 4.08 (br s, 2H, CH for *trans*-**1**), 6.78 (br s, Ar−H), 6.90 (br s, *Ph*$_3$P), 7.10 (br m, *Ph*$_3$P), 7.61 (br s, *Ph*$_3$P), 8.42 (br s, 2H, $^2J_{Pt-H}$ = 137, 78 Hz, Si−H for *cis*-**1**), 8.92 (br s, 2H, $^2J_{Pt-H}$ = 137, 72 Hz, Si−H for *trans*-**1**); $^{31}P\{^1H\}$ NMR (202 MHz, THF, and $C_6D_6$, $\delta$) 37.5 (s, $^1J_{Pt-P}$ = 4276 Hz, $^2J_{Pt-P}$ = 261 Hz, $^3J_{P-P}$ = 60 Hz, *trans*-**1**), 37.2 (s, coupling constants not well resolved for *cis*-**1**); $^{29}Si$ NMR (500 MHz, $CD_2Cl_2$, $^1H-^{29}Si$ HMQC, $\delta$) 131 (*trans*-**1**), 126 (*cis*-**1**); IR (KBr) $\nu$ (Si−H) 2111.2 $cm^{-1}$, $\nu$(Pt···*H*···Si) 1680.0 $cm^{-1}$. Anal. Calcd For $C_{56}H_{60}P_2Pt_2Si_2$: C, 54.18; H, 4.87. Found: C, 56.17; H, 5.10 (these values are consistent with benzene solvate, calcd C, 56.39; H, 5.00; however the exact amount could not be determined by $^1H$ NMR spectroscopy due to overlapping resonances in the aromatic region). HR-MAS (FAB) calcd for M−$H_2$: 1238.2848. Found: 1238.2842.

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[12] Kim, Y.-J.; Lee, S.-C.; Park, J.-I.; Osakada, K.; Choi, J.-C.; Yamamoto, T. *Organometallics* **1998**, *17*, 4929.

[13] A solution of $PMe_2Ph$ (8 mg, 5.8 × $10^{-5}$ mol) in 0.5 mL of $C_6D_6$ was added to **1** (16 mg, 1.3 × $10^{-5}$ mol) in 0.5 mL of $C_6D_6$. Vigorous gas evolution was observed, and the color changed from colorless to intense yellow. The reaction was quantitative by NMR, and *trans*-**2** was obtained in 87% (14 mg) isolated yield from slow evaporation of a pentane/$C_6D_6$ solution (data are consistent with an authentic sample prepared independently by reaction of $(PhMe_2P)_2PtMe_2$ with IMP$SiH_3$, results to be published). $(PhMe_2P)_2PtMe_2$ was prepared from a modified procedure described in: Ruddick, J. D.; Shaw, B. L. *J. Chem. Soc. A* **1969**, 2801. Spectroscopic and physical data for **2**: $^1H\{^{31}P\}$ NMR (300 MHz, $\delta$) 1.12 (s, 12H, $^3J_{Pt-Me}$ = 20 Hz, $PMe_3$), 1.35 (s, 12H, $^3J_{Pt-Me}$ = 22 Hz, $PMe_3$), 1.70 (d, 12H, $^3J_{H-H}$ = 6.6 Hz, CH(C*H*$_3$)$_2$), 2.62 (s, 6H, Ar−C*H*$_3$), 4.81 (sept, 2H, $^3J_{H-H}$ = 6.6 Hz, C*H*(CH$_3$)$_2$), 5.79 (s, 2H, $^1J_{Si-H}$ = 169 Hz,$^2J_{Pt-H}$ = 30 Hz, Si−H), 6.89−6.94 (m, Ar−H), 7.16−7.20 (m, Ar−H), 7.32 (d, *J* = 8.1 Hz, Ar−H); $^{31}P\{^1H\}$ NMR (121 MHz, $\delta$) −2.06 (s, $^1J_{Pt-P}$ = 1725 Hz, $^3J_{Pt-P}$ = 271 Hz, $^4J_{P-P}$ = 20 Hz); $^{29}Si\{^1H\}$ (99 MHz, DEPT, $\delta$) −134.2 (m, $^1J_{Pt-Si}$ = 699 Hz, $^2J_{P-Si}$ = 66 Hz (cis), $^2J_{P-Si}$ = 107 Hz (trans)). Anal. Calcd For $C_{52}H_{72}P_4Pt_2Si_2$: C, 49.24; H, 5.73. Found: C, 49.38; H, 5.79.

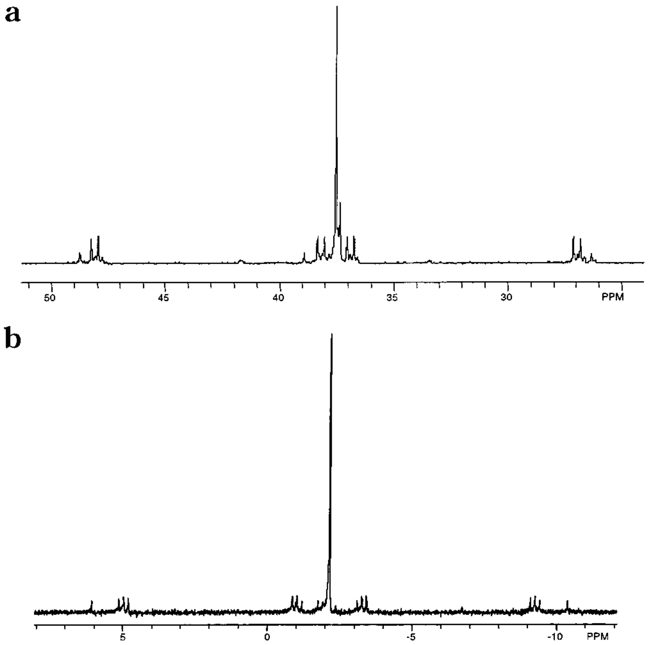

**Figure 2.** (a) $^{31}P\{^1H\}$ NMR spectrum (25 °C) of **1** (202 MHz, THF, and $C_6D_6$). (b) $^{31}P\{^1H\}$ NMR spectrum (25 °C) of **2** (121 MHz, $C_6D_6$).

(Figure 1b) appears at 5.79 ppm in the $^1H\{^{31}P\}$ NMR spectrum, in the typical region (3.0−6.0 ppm) for Si−H chemical shifts in free silanes or M−SiH moieties.[1]

The nonclassical Pt···*H*···Si resonance could not be resolved from the $^1H$ NMR experiment in $C_6D_6$ (better resolved in $CD_2Cl_2$), but a 2D $^1H-^1H$ EXSY experiment confirmed that the bridging hydride resonance overlapped with the aryl−$CH_3$ resonance ($\delta$ 2.17 ppm). The Pt···*H*···Si chemical shift value is in close agreement with the values reported by Stone et al.[10] The $^1H-^1H$ EXSY experiment also revealed that the molecule is fluxional on the NMR time scale at room temperature. Exchange of the terminal and bridging hydrides results in concomitant cis−trans isomerization. No fluxionality was observed in the Pt dimers reported by Stone and co-workers: however, the unsymmetrical Pd complex $(Me_3P)Pd(\mu\text{-}SiHPh_2)_2Pd(PMe_3)_2$ was found to be fluxional by NMR (involving dissociation of $PMe_3$).[12] Compound **2** does not show fluxional behavior.

The $^{29}Si$ NMR chemical shifts for **1** and **2** are vastly different. The 2D $^1H-^{29}Si$ HMQC ($CD_2Cl_2$) experiment showed two distinct resonances for *trans*-**1** (131 ppm) and *cis*-**1** (126 ppm). Compound **2** exhibits a high-field resonance in the solution $^{29}Si\{^1H\}$ NMR spectrum at −134 ppm, clearly indicating a dissimilar environment at silicon. These data are in agreement with related Pt−Si four-membered ring structures prepared by Tessier and co-workers.[11] A low-field resonance of 195 ppm was observed for $\{(Pr_3P)Pt[\mu\text{-}\eta^2\text{-}H-Si(Hex)PtH(PPr_3)_2]\}_2$, and high-field signals of −75 and −94 ppm (cis and trans isomers) were found for the related ring systems $\{(Pr_3P)_2Pt[\mu\text{-}SiH(Hex)]\}_2$. The Si−H stretching vibrations for **1** found in the IR spectrum clearly indicate a terminal Si−H unit (2111 $cm^{-1}$) and a weaker stretching frequency for the bridging Pt···H···Si moiety (1618 $cm^{-1}$). Complex **2** exhibits a stretching vibration at 2021 $cm^{-1}$ for the terminal Si−H unit.

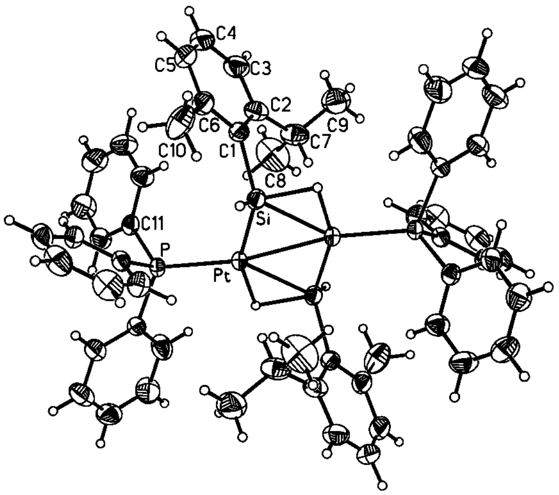

**Figure 3.** Molecular structure of *trans*-$\{(Ph_3P)Pt[\mu\text{-}\eta^2\text{-}H-SiH(IMP)]\}_2$, **1** (50% probability). Selected bond distances (Å) and angles (deg): Pt−Pt#1 = 2.7021(2), Pt−Si = 2.3248(9), Pt−Si#1 = 2.4280(9), Pt−P = 2.2485(8), Si−H = 1.593, Si···H = 1.669, Pt···H = 1.799; P−Pt−Si = 105.08(3), P−Pt−Si#1 = 143.89(3), Si−Pt−Si#1 = 110.74(3), P−Pt−Pt#1 = 161.78(2), Si−Pt−Pt#1 = 57.17(2), Si#1−Pt−Pt#1 = 53.57(2), Pt···H···Si = 88.8, Pt−Si−C1 = 119.23(11), Pt#1−Si−C1 = 121.79(12), Pt−Si−Pt#1 = 69.26(3).

The $^{31}P\{^1H\}$ NMR spectra for **1** and **2** (Figure 2, parts a and b, respectively) display a characteristic pattern for a dinuclear platinum complex but differ in the splitting of the satellite peaks due to the number of phosphines bound to Pt. A central line is seen for both the cis and trans (major) isomers of **1** at ~37 ppm. The resonance for **2** is shifted significantly upfield (−2 ppm).

An X-ray crystal structure for the trans isomer of **1** was obtained (Figure 3),[16] and both the terminal and bridging hydrides were located and refined. The central core containing the $Pt_2Si_2(\mu\text{-}H)_2$ unit is planar with two different Pt−Si distances, 2.3248(9) and 2.4280(9) Å. The former value is in good agreement with other reported Pt−Si single-bond distances.[1] The longer Pt−Si distance suggests a 3c-2e interaction between Pt···H···Si. In addition, the Pt···H and Si···H distances are elongated relative to typical 2c-2e bonds between these elements. The terminal Si−H distance is 1.593 Å, and

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(14) A preliminary X-ray structure indicates a Pt···Pt distance in **2** of 3.962 Å, outside the range of a Pt−Pt interaction. A detailed structure will be published elsewhere. Such distances are observed in other four-membered Pt−Si ring systems such as reported in: (a) See ref 5. (b) Zarate, E. A.; Tessier-Youngs, C. A.; Youngs, W. J. *J. Am. Chem. Soc.* **1988**, *110*, 4068. (c) Zarate, E. A.; Tessier-Youngs, C. A.; Youngs, W. J. *J. Chem. Soc., Chem. Commun.* **1989**, 577. (d) Michalczyk, M. J.; Recatto, C. A.; Calabrese, J. C.; Fink, M. J. *J. Am. Chem. Soc.* **1992**, *114*, 7955. (e) Heyn, R. H.; Tilley, T. D. *J. Am. Chem. Soc.* **1992**, *114*, 1917. (f) Shimada, S.; Tanaka, M.; Honda, K. *J. Am. Chem. Soc.* **1995**, *117*, 8289.

(15) Unpublished results.

(16) Crystal Data for **1** (223 K): $C_{56}H_{60}P_2Pt_2\cdot 2CH_3CN$, triclinic, $P\bar{1}$, $a = 9.2722(1)$ Å, $b = 13.118$ Å, $c = 13.1805(1)$ Å, $\alpha = 65.309$ (1)°, $\beta = 69.848$ (1)°, $\gamma = 89.902$ (1)°, V = 1348.26 (2) Å$^3$, Z = 2, $D_{calc} = 1.630$ Mg/m$^3$, $F(000) = 654$, $\mu$(Mo Kα) = 5.326 mm$^{-1}$, $R(F) = 2.51\%$ for $I > 2\sigma I$ reflections, $wR2(F_2) = 6.26\%$ for 6136 unique reflections ($R_{int} = 2.83\%$), 312 parameters, GOOF = 1.070. Data collected using Bruker CCD area detector system. The H atoms attached to silicon were located and refined freely. The crystal contains disordered solvent ($CH_3CN$). A second X-ray structure determination was performed on a sample of **1** recrystallized from benzene, and the preliminary structure revealed three molecules of benzene per molecule of **1**. The cell parameters for the second structure are as follows: $P\bar{1}$, $a = 9.3543(3)$ Å, $b = 13.2467(5)$ Å, $c = 14.2850(5)$ Å, $\alpha = 81.564(2)°$, $\beta = 80.1846(2)°$, $\gamma = 69.974(2)°$, $V = 1631.121(1)$ Å$^3$.

a significant lengthening is observed for the nonclassical Si···H (1.669 Å).[1] Typically, Pt−H distances are about 1.7 Å,[17] and in compound **1**, the Pt···H distance was notably longer, 1.799 Å. Similar structural features were seen in the complex prepared by Stone et al., $[(Cy_3P)Pt(\mu\text{-}\eta^2\text{-}H{-}SiMe_2)]_2$ (Cy = cyclohexyl): Pt−Si, 2.324(2) Å; Pt−Si, 2.420(2) Å; Si···H, 1.72 Å; Pt···H, 1.78 Å.[10] The related Pd dimer, $(Me_3P)Pd(\mu\text{-}\eta^2\text{-}H{-}SiPh_2)_2Pd(PMe_3)$, also revealed two inequivalent Pd−Si distances, 2.328(2) and 2.386(2) Å, with the longer distance associated with the "agostic" Pd···Si distance.[12] The Pd···H and Si···H distances were 1.91 and 1.75 Å, respectively.[12] The Pt−Pt#1 [2.7021 (2) Å] and the Pt−P [2.2485 (8) Å] distances are similar to other complexes containing two Pt(I) centers bound to each other with phosphine ligands (covalent radii for Pt, 2.62 Å).[18] Each Pt center resides in a distorted square planar environment. The P−Pt−Pt#1 angle is nonlinear, 161.78 (2)°, and the phosphines bend away from the Pt···H···Si unit. The Pt−Si−Pt#1 angle is more acute, [69.26(3)°] than anticipated as a result of the formation of the Pt−Pt bond. The spectroscopic and solid-state data support the presence of a 3c-2e interaction both in solution and in the solid state.

Further studies are under way with other mono- and diarylsilanes to determine the electronic and steric factors that influence the formation of **1**. Additional variable-temperature NMR experiments and reactivity studies are planned.

**Acknowledgment.** This work is supported by the University of Missouri—St. Louis Research Award. The support of the NSF (CHE-9318696) and the University of Missouri−Research Board are gratefully acknowledged for the purchase of a Varian Unity *Plus* 300 and Bruker ARX-500 NMR spectrometers. The UM-St. Louis X-ray Crystallography Facility was funded in part by an NSF Instrumentation Grant (CHE-9309690) and the UM-St. Louis Research Award. J.B-W. is also grateful to Professors Joyce Y. Corey, Peter P. Gaspar, and Mr. Brian Grimmond for interesting discussions and comments.

**Supporting Information Available:** Experimental, NMR spectra, and crystallographic information (*trans*-**1**). This material is available free of charge via the Internet at http://pubs.acs.org.

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[17] See for example: Koizumi, T.-a.; Osakada, K.; Yamamoto, T. *Organometallics* **1997**, *16*, 6014.

[18] See for example: (a) Green, M.; Howard, J. A. K.; Proud, J.; Spencer, J. L.; Stone, F. G. A.; Tsipis, C. A. *J. Chem. Soc., Chem. Commun*. **1976**, 671. (b) Taylor, N. J.; Chieh, P. C.; Carty, A. J. *J. Chem. Soc., Chem. Commun*. **1975**, 448.