

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

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Received February 8, 1999

Summary: A dinuclear Pt–Si complex containing bridging $\eta^2\text{-H-SiH(IMP)}$ (IMP = 2-isopropyl-6-methylphenyl) ligands, $\{(\text{Ph}_3\text{P})\text{Pt}[\mu\text{-}\eta^2\text{-H-SiH(IMP)}]\}_2$ (**1**) has been synthesized and characterized by NMR and X-ray crystallography. A 3c-2e nonclassical interaction between $\text{Pt}\cdots\text{H}\cdots\text{Si}$ 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 , $\{(\text{PhMe}_2\text{P})_2\text{Pt}[\mu\text{-SiH(IMP)}]\}_2$ (**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(μ -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 σ -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(μ -silylene) complexes have been shown

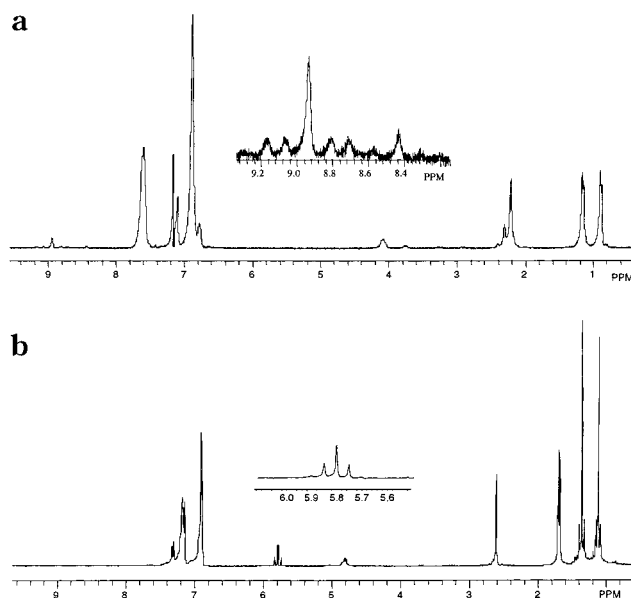


Figure 1. (a) ^1H NMR spectrum (25 °C) of **1** (300 MHz, C_6D_6). The inset shows an expanded view of the terminal Si–H region with ^{195}Pt satellites. (b) $^1\text{H}\{^{31}\text{P}\}$ NMR spectrum (25 °C) of **2** (300 MHz, C_6D_6). The inset shows an expanded view of the terminal Si–H region with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

to catalyze the oligomerization (stepwise) of primary silanes to small-chain oligosilanes.⁵ Recently, a series of dinuclear rhodium (μ -silylene) and ($\mu\text{-}\eta^2$ -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 μ -silylene complexes reported, little is known about their reactivity, most of which involve small molecules or catalytic reactions.²

We report here the preparation and characterization of the dinuclear Pt complex $\{(\text{Ph}_3\text{P})\text{Pt}[\mu\text{-}\eta^2\text{-H-SiH(IMP)}]\}_2$ (IMP = 2-isopropyl-6-methylphenyl), which contains two nonclassical $\text{Pt}\cdots\text{H}\cdots\text{Si}$ 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. *J. Chem. Rev.* **1999**, *99*, 175.

(2) Ogino, H.; Tobita, H. *Adv. Organomet. Chem.* **1998**, *42*, 223.

(3) (a) Tilley, T. D. *Acc. Chem. Res.* **1993**, *26*, 22. (b) Corey, J. Y. In *Advances in Silicon Chemistry*; Larson, G. L., Ed.; JAI Press: Greenwich, CT, 1991; Vol. 1, p 327. (c) For a recent review on catalytic dehydrocoupling see: Gauvin, F.; Harrod, J. F.; Woo, H. G. *Adv. Organomet. Chem.* **1998**, *42*, 363.

(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 ($\text{L}_2\text{M}=\text{SiRR}'$) 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.

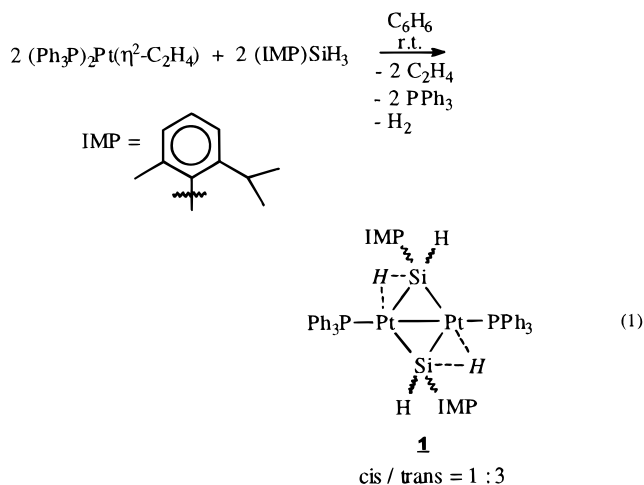
(5) Tessier, C. A.; Kennedy, V. O.; Zarate, E. A. In *Inorganic and Organometallic Oligomers and Polymers*; Harrod, J. F., Laine, R. M., Eds.; Kluwer Academic Publishers: The Netherlands, 1991; p 13.

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

(7) Braddock-Wilking, J.; Levchinsky, Y.; Rath, N. P. Thirty-First Organosilicon Symposium, May 1998, New Orleans, LA, abstract B9.

knowledge, this is the first example of the conversion of a dinuclear bis(μ - η^2 -H-SiR₂) complex to a bis(μ -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 (Ph₃P)₂Pt(η^2 -C₂H₄) with 1 equiv (or a 4-fold excess) of (IMP)SiH₃ 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 (H₂ and C₂H₄) 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, ³¹P{¹H}, and ²⁹Si{¹H} NMR spectroscopy as well as X-ray crystallography (*trans*-**1** only).

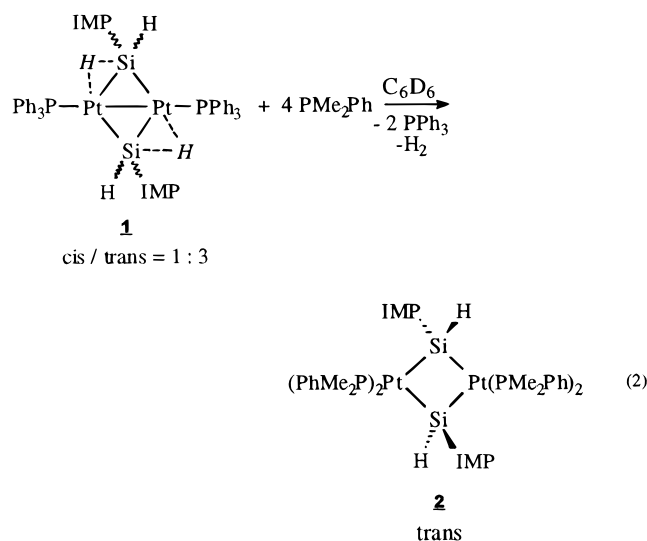
There are only a few complexes of the group 10 metals that contain a nonclassical M...H...Si interaction and include several diplatinum complexes {(R'₃P)Pt(μ - η^2 -H-SiR₂)₂ (R' = alkyl or aryl; R = Me, Ph) prepared by Stone et al.¹⁰ Tessier and co-workers synthesized the novel dinuclear platinum complex {(Pr₃P)Pt[μ - η^2 -H-Si(Hex)PtH(PPR₃)₂]₂ (Pr = *n*-propyl, Hex = *n*-hexyl).¹¹

(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)SiH₃ (22 mg, 0.13 mmol) was added slowly to (Ph₃P)₂Pt(η^2 -C₂H₄) (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 C₆H₆ (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 (Ph₃P)₄Pt and (IMP)SiH₃ gave 70% yield and much lower yield from (Ph₃P)₂PtMe₂. Spectroscopic and physical data for **1**: ¹H NMR (300 MHz, δ) 0.89 (br d, 12H, ³J_{H-H} = 5.7 Hz, CH(CH₃)₂, *cis* and *trans* resonances overlapping), 1.16 (br d, 12H, ³J_{H-H} = 6.3 Hz, CH(CH₃)₂, *cis* and *trans* resonances overlapping), 2.22 (s, 6H, *o*-CH₃ for *trans*-**1**; overlapping with Pt...H...Si), 2.31 (s, 6H, *o*-CH₃ for *cis*-**1**), 8.92 (br s, 2H, ²J_{Pt-H} = 137, 72 Hz, Si-H for *trans*-**1**); ³¹P{¹H} NMR (202 MHz, THF, and C₆D₆, δ) 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**); ²⁹Si NMR (500 MHz, CD₂Cl₂, ¹H-²⁹Si HMQC, δ) 131 (*trans*-**1**), 126 (*cis*-**1**); IR (KBr) ν (Si-H) 2111.2 cm⁻¹, ν (Pt...H...Si) 1680.0 cm⁻¹. Anal. Calcd For C₅₆H₆₀P₂Pt₂Si₂: 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-H₂: 1238.2848. Found: 1238.2842.

More recently, Kim and co-workers reported two dinuclear Pd complexes containing Ph₂Si groups bridging the Pd centers, (Me₃P)Pd(μ - η^2 -H-SiPh₂)₂Pd(PMe₃)_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, PhMe₂P, a new dinuclear Pt-Si complex, *trans*-**2**¹³ (with elimination of H₂), 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 Pt₂Si₂ ring systems analogous to **2** have been prepared by Tessier,^{14b,c} Fink,^{14d} Tilley,^{14e} and Tanaka.^{14f}

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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(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 PMe₂Ph (8 mg, 5.8 × 10⁻⁵ mol) in 0.5 mL of C₆D₆ was added to **1** (16 mg, 1.3 × 10⁻⁵ mol) in 0.5 mL of C₆D₆. 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/C₆D₆ solution (data are consistent with an authentic sample prepared independently by reaction of (PhMe₂P)₂PtMe₂ with IMPSiH₃, results to be published). (PhMe₂P)₂PtMe₂ 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{³¹P} NMR (300 MHz, δ) 1.12 (s, 12H, ³J_{Pt-Me} = 20 Hz, PMe₃), 1.35 (s, 12H, ³J_{Pt-Me} = 22 Hz, PMe₃), 1.70 (d, 12H, ³J_{H-H} = 6.6 Hz, CH(CH₃)₂), 2.62 (s, 6H, Ar-CH₃), 4.81 (sept, 2H, ³J_{H-H} = 6.6 Hz, CH(CH₃)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); ³¹P{¹H} NMR (121 MHz, δ) -2.06 (s, ¹J_{Pt-P} = 1725 Hz, ³J_{Pt-P} = 271 Hz, ⁴J_{P-P} = 20 Hz); ²⁹Si{¹H} (99 MHz, DEPT, δ) -134.2 (m, ¹J_{Pt-Si} = 699 Hz, ²J_{P-Si} = 66 Hz (*cis*), ²J_{P-Si} = 107 Hz (*trans*)). Anal. Calcd For C₅₂H₇₄P₂Pt₂Si₂: C, 49.24; H, 5.73. Found: C, 49.38; H, 5.79.

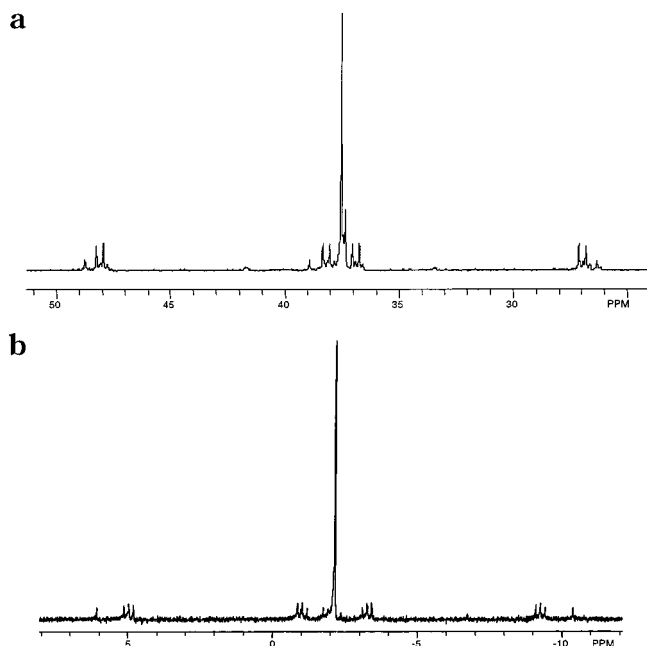


Figure 2. (a) $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum (25 °C) of **1** (202 MHz, THF, and C_6D_6). (b) $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum (25 °C) of **2** (121 MHz, C_6D_6).

(Figure 1b) appears at 5.79 ppm in the $^1\text{H}\{^{31}\text{P}\}$ 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 $\text{Pt}\cdots\text{H}\cdots\text{Si}$ 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 (δ 2.17 ppm). The $\text{Pt}\cdots\text{H}\cdots\text{Si}$ 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 $(\text{Me}_3\text{P})\text{Pd}(\mu\text{-SiHPh}_2)_2\text{Pd}(\text{PMe}_3)_2$ 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 $^{29}\text{Si}\{^1\text{H}\}$ 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 $\{(\text{Pr}_3\text{P})\text{Pt}[\mu\text{-}\eta^2\text{-H-Si}(\text{Hex})\text{PtH}(\text{PPr}_3)_2]\}_2$,

(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.

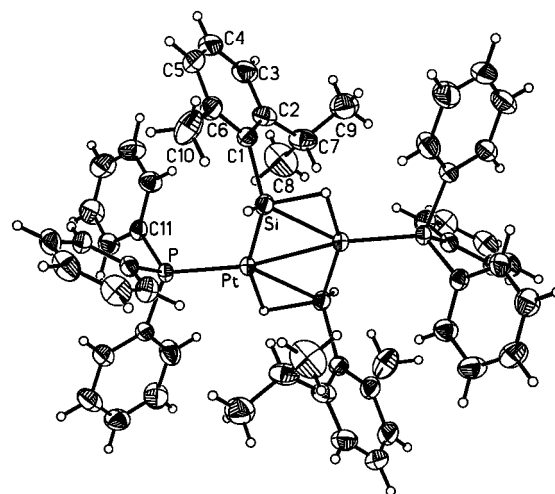


Figure 3. Molecular structure of *trans*- $\{(\text{Ph}_3\text{P})\text{Pt}[\mu\text{-}\eta^2\text{-H-SiH}(\text{IMP})]\}_2$, **1** (50% probability). Selected bond distances (Å) and angles (deg): Pt–Pt#1 = 2.7021(2), Pt–Si = 2.3248(9), Pt–Si#1 = 2.4280(9), Pt–P = 2.2485(8), Si–H = 1.593, Si \cdots H = 1.669, Pt \cdots 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 \cdots H \cdots Si = 88.8, Pt–Si–C1 = 119.23(11), Pt#1–Si–C1 = 121.79(12), Pt–Si–Pt#1 = 69.26(3).

and high-field signals of –75 and –94 ppm (*cis* and *trans* isomers) were found for the related ring systems $\{(\text{Pr}_3\text{P})_2\text{Pt}[\mu\text{-SiH}(\text{Hex})]\}_2$. 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 \cdots H \cdots Si moiety (1618 cm^{-1}). Complex **2** exhibits a stretching vibration at 2021 cm^{-1} for the terminal Si–H unit.

The $^{31}\text{P}\{^1\text{H}\}$ 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 $\text{Pt}_2\text{Si}_2(\mu\text{-H})_2$ 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 \cdots H \cdots Si. In addition, the Pt \cdots H and Si \cdots H distances are elongated relative to typical 2c–2e bonds between these elements. The terminal Si–H distance is 1.593 Å, and

(16) Crystal Data for **1** (223 K): $\text{C}_{56}\text{H}_{60}\text{P}_2\text{Pt}_2\cdot 2\text{CH}_3\text{CN}$, triclinic, $\bar{P}1$, $a = 9.2722(1)$ Å, $b = 13.118$ Å, $c = 13.1805(1)$ Å, $\alpha = 65.309(1)^\circ$, $\beta = 69.848(1)^\circ$, $\gamma = 89.902(1)^\circ$, $V = 1348.26(2)$ Å³, $Z = 2$, $D_{\text{calc}} = 1.630\text{ Mg/m}^3$, $F(000) = 654$, $\mu(\text{Mo K}\alpha) = 5.326\text{ mm}^{-1}$, $R(F) = 2.51\%$ for $I > 2\sigma I$ reflections, $wR2(F_o) = 6.26\%$ for 6136 unique reflections ($R_{\text{int}} = 2.83\%$), 312 parameters, $\text{GOOF} = 1.070$. 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: $\bar{P}1$, $a = 9.3543(3)$ Å, $b = 13.2467(5)$ Å, $c = 14.2850(5)$ Å, $\alpha = 81.564(2)^\circ$, $\beta = 80.1846(2)^\circ$, $\gamma = 69.974(2)^\circ$, $V = 1631.121(1)$ Å³.

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., [(Cy₃P)-Pt(μ - η^2 -H–SiMe₂)]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, (Me₃P)Pd(μ - η^2 -H–SiPh₂)₂Pd(PMe₃), 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

(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.

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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