Silicon Chemistry

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Synthesis of a Stable Disilyne Bisphosphine Adduct and Its Non-Metal-Mediated CO_2 Reduction to CO**

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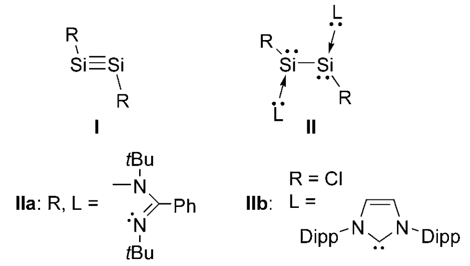
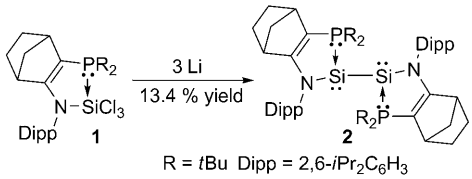
Since the first synthesis of stable disilynes **I**, with a silicon–silicon triple bond, independently by the groups of Sekiguchi and Wiberg,[1] the unique chemical behavior of these species has been attracting much attention. Structural determinations revealed that **I** presents a trans-bent structure resulting in two nondegenerate Si–Si π molecular orbitals and particularly a weak in-plane π bond with a lowest unoccupied molecular orbital that is low-lying in energy.[1a] As a consequence, the formation of bis(Lewis base) adducts **II** by the addition of two donating ligands is strongly exothermic.[2]



Only a few stable disilyne bis(Lewis base) adducts **IIa,b** with different ligands have been synthesized to date. Strongly σ-donating ligands with a weak π-accepting character such as N-heterocyclic carbenes[2] or imines[3] induce highly polarized 1,2-diylidic structures with long Si–Si single bonds. As a consequence, each silicon center in these adducts **II** exhibits a lone pair of electrons.[4] In fact, the chemical properties of the Lewis base stabilized disilynes should be strongly affected by the complexation, and this important issue needs to be addressed. However, compared to transition-metal derivatives, the coordination chemistry of main-group-element species such as disilynes[2–4] or silylenes[5] is not very well developed, although their transition-metal-like behavior is of great interest.[6] Thus, although phosphines are ubiquitous ligands in organometallic chemistry, their use in main-group-element chemistry is not so frequent. Recently we reported that phosphine-stabilized silylenes (phosphonium silaylides) react either as sila-Wittig reagents[7] or as nucleophilic silylene complexes.[8] These results highlight the efficiency of complexation to stabilize low-coordinate species without perturbing their high reactivity. Herein we present the synthesis of an original disilyne bisphosphine adduct **2** (Scheme 1) and its reaction with carbon dioxide, thus demonstrating a powerful function as an efficient CO_2-reducing agent.



Scheme 1. Synthesis of disilyne bisphosphine adduct **2**.

We have already described the synthesis of stable cyclic phosphonium silaylides by reduction of dichlorophenylsilane derivatives bearing a 2-phosphinoenamine fragment using magnesium metal.[7] By analogy, we now investigated the reduction of trichlorosilane derivative **1** using lithium (3 equiv) in THF at room temperature. The reaction readily affords the disilyne bisphosphine adduct **2**, which can be isolated as red crystals (13% yield). In the ^{29}Si NMR spectrum, derivative **2** displays a doublet of doublets at $\delta = -18.5$ ppm ($^{1}J_{SiP} = 192.1$ Hz, $^{2}J_{SiP} = 60.1$ Hz), thus indicating the presence of two phosphorus atoms in the molecule. Large silicon–phosphorus coupling constants have been observed for monophosphonium silaylides ($^{1}J_{SiP} = 141$–157 Hz).[7] In the ^{31}P NMR spectrum, only one singlet at $\delta = 34.7$ ppm was detected in the typical region for phosphonium derivatives, which is downfield from that of starting trichlorosilane derivative **1** ($\delta = 2.2$ ppm). These data are in good agreement with a symmetrical arrangement of the molecule.

The molecular structure of **2** was unambiguously determined by X-ray crystallography (Figure 1).[9] The N-Si1-Si1′-N1′ skeleton presents a cis-bent geometry (torsion angle 4.9°). Each silicon atom interacts with the phosphorus center on both sides of the Si_2N_2 plane (P1-Si1-Si1′-P1′ 159.10°), forming two five-membered cyclic fragments. A value of

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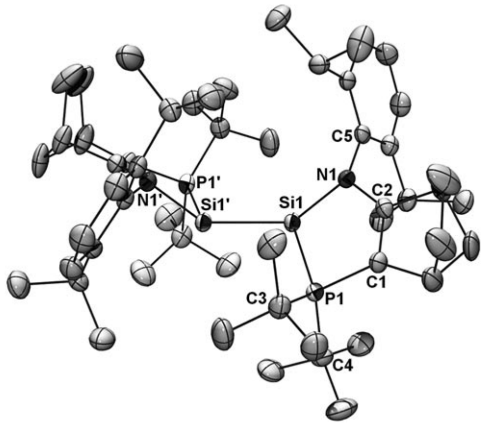
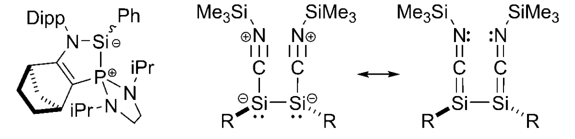


Figure 1. Molecular structure of **2**. Thermal ellipsoids represent 30% probability. H atoms are omitted for clarity. Selected bond lengths [Å], angles, and torsion angles [°]: Si1–Si1′ 2.3306(12), Si1–P1 2.3274(9), P1–C1 1.760(2), C1–C2 1.375(3), C2–N1 1.360(3), Si1–N1 1.8805(18); P1-Si1-Si1′ 103.64(4), N1-Si1-Si1′ 137.60(6), N1-Si1-P1 88.41(6), Si1-P1-C1 93.03(8), P1-C1-C2 115.52(17), C1-C2-N1 124.97(19), C2-N1-Si1 113.65(14), C2-N1-C5 118.57(17), C5-N1-Si1 117.03(14), N1-Si1-Si1′-N1 4.86, P1-Si1-Si1′-P1′ 159.10.

2.331 Å for the Si–Si bond is at first glance in the range of ordinary Si–Si single bonds (2.34 Å).[10] However, it is substantially shorter than those observed for the other reported disilyne bis(Lewis base) adducts **IIa,b** (2.393–2.413 Å)[2,3] and is even shorter than in the similar adduct **4** (2.369 Å) with 1,2-disilaketenimine character[11] (Table 1).

Table 1: Si–Si and Si–L bond lengths of different disilyne adducts. Data for **3** are given for comparison.



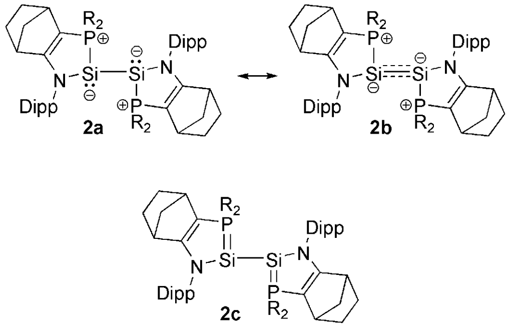
3 **4** R = Si(*i*Pr)(CHTMS$_2$)$_2$

	IIa	**IIb**	**2**	**3**	**4**[a]
Si–Si [Å]	2.413	2.393	2.331		2.369
Si–L [Å]	1.874	1.939	2.327	2.319	1.826

[a] TMS = Me$_3$Si.

The P–Si bond lengths (2.327 Å) are typical for single bonds and are slightly greater than that observed for phosphonium silaylide **3** (2.319 Å),thus indicating the absence of P–Si π interaction. The silicon centers in **2** are less pyramidalized ($\Sigma°_{Si} = 330°$) than that in monophosphonium ylide **3** ($\Sigma°_{Si} = 299°$)[7] and even less than those in **4** with diketenimine character ($\Sigma°_{Si} = 319°$).[11]

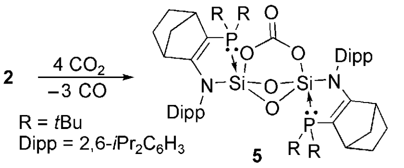
To gain more insight into the properties of the molecule, DFT calculations were performed on the real compound **2**. The computation at the bHandH/6-311+G* level reasonably reproduces the experimental geometry and structural parameters (Si–Si 2.323 Å, Si–P 2.314 Å). As expected, the Si–Si bond order (1.262)[12] is larger than that expected for Si–Si single bonds. In contrast, the Wiberg bond index of the Si–P bond is practically unchanged compared with that in monoylide **3** (0.85).[7] These results show a clear contrast in ligand effect between phosphine and the π-acidic isocyanide ligands. In the latter case, a smaller Si–Si Wiberg bond index (0.839) and larger Si–C Wiberg bond index (1.049) were calculated (**4′**: R = H, L = CNSiH$_3$), which is expected for a disilaketenimine character. The strongly pyramidalized silicon center and the relatively small Si–Si Wiberg bond index in **2** illustrate the major contribution of the highly polarized electronic structure **2a** (Scheme 2), with a Si–Si single bond and a lone pair on each silicon center. However, the increased Si–Si Wiberg bond index in **2** compared to **4** also agrees with a certain degree of multibonding character (resonance structure **2b**), probably owing to enhanced negative hyperconjugative π interactions between the two silicon centers (n_{Si}–σ^*_{Si}) rather than between phosphorus and silicon centers (n_{Si}–σ^*_{P}), as indicated by the weak phosphine–silicon π interaction (resonance structure **2c** in Scheme 2). A similar ligand effect was observed in the recently synthesized phosphine-stabilized silyne showing a short Si–C bond corresponding to a triple bond, despite the coordination of phosphine ligand.[13]



Scheme 2. n_{Si}–σ^*_{Si} negative hyperconjugative π interactions in **2**.

Although this new species **2** is stable at room temperature under inert atmosphere for weeks, it shows a high reactivity with carbon dioxide. Indeed, the reaction of **2** with CO$_2$ is complete upon mixing to afford the aminosilicate **5** with two pentacoordinate silicon atoms (78% yield of isolated product, Scheme 3). During the reaction clear gas evolution was observed, suggesting the formation of CO gas.

Two singlets at $\delta = -6.5$ and -7.5 ppm in the ^{31}P NMR spectrum suggest the formation **5** as a pair of two diastereomers. The pentacoordination of silicon centers[4b] is clearly indicated in the ^{29}Si NMR spectrum by significant upfield shifts of signals ($\delta = -100.7$ and -101.6 ppm) appearing as two doublets of doublets ($J_{SiP} = 7.7$ and 121.9 Hz and 7.6 and 120.2 Hz). The large phosphorus–silicon coupling constants are in good agreement with strong P–Si interactions. Owing



Scheme 3. The reaction of disilyne derivative **2** with CO$_2$. Product **5** is isolated in 78% yield.

to the presence of a mixture of the two diastereomers (50:50), the X-ray structure of **5** is disordered. Each diastereomer shows a quasi-C_2-symmetric structure, which is consistent with the observed two singlet signals in the ^{31}P NMR spectrum. The structure reveals the cleavage of the Si–Si bond (Si1–Si2 2.4151 Å) and the presence of a central tricyclic structure (Figure 2). Both silicon centers arrange in a somewhat distorted trigonal bipyramid with the phosphorus and oxygen atoms in the apical positions (O1-Si1-P1 173.27°, $\Sigma\theta°_{\text{equatorial}} = 359.7°$). The long apical P1–Si1 bond (2.498 Å) is of the same order as that observed for the previously reported pentacoordinate oxasilirane (2.491 Å).[8b] The apical Si1–O1 bond (1.742 Å) is much longer than the equatorial ones (Si1–O2 1.665 Å, Si1–O3 1.717 Å).

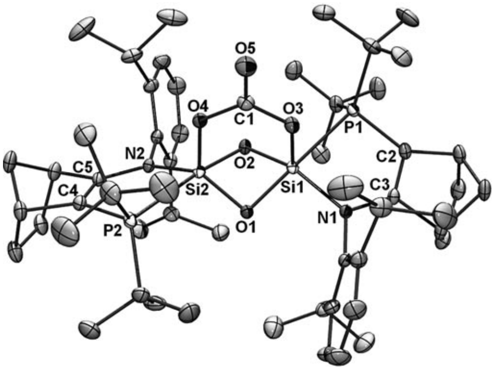
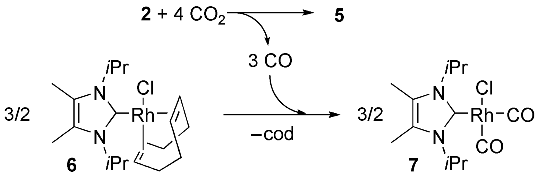


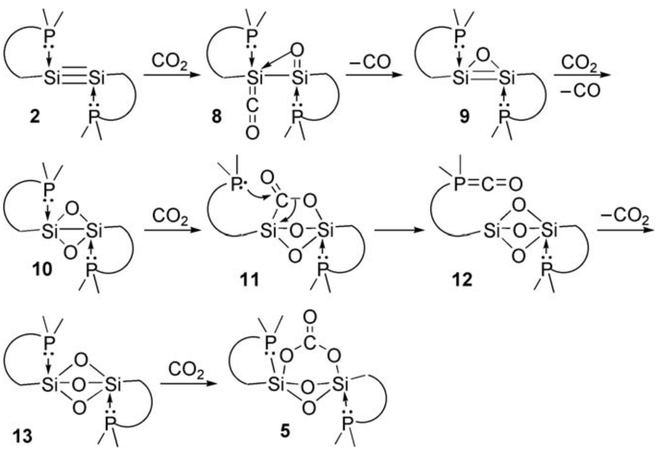
Figure 2. Molecular structure of **5**. Thermal ellipsoids represent 30% probability. H atoms are omitted for clarity. Selected interatomic distances [Å] and angles [°]: Si1–O1 1.7419(12), Si1–O2 1.6651(11), Si1–O3 1.7170(12), Si1–P1 2.4978(6), Si1–N1 1.7824(14), P1–C2 1.764(3), C2–C3 1.358(3), C3–N1 1.395(3), C1–O3 1.346(2), C1–O4 1.340(2), C1–O5 1.201(2), Si1–Si2 2.4151(6); Si1-O1-Si2 90.38(6), Si1-O2-Si2 90.44(5), O1-Si1-O2 84.56(6), O1-Si1-O3 96.16(6), O1-Si1-N1 95.56(6), O1-Si1-P1 173.27(5), O2-Si1-O3 103.78(6), O2-Si1-N1 142.39(6), O2-Si1-P1 91.36(4), O3-Si1-N1 113.55(6), O3-Si1-P1 90.01(4), N1-Si1-P1 84.39(5), C1-O3-Si1 123.15(11), C1-O4-Si2 122.67(11), O3-C1-O4 117.20(14), O3-C1-O5 121.42(16), O4-C1-O5 121.38(16).

In fact, disilyne **2** reacts with four equivalents of CO_2, formally extracting three oxygen atoms and capturing one CO_2 molecule. To quantify and characterize the released carbon monoxide (CO), the reaction was performed in the presence of a slight excess of {Rh$^{\text{I}}$(cod)} (cod = cycloocta-1,5-diene) complex **6**. A quantitative ligand-exchange reaction[14] was detected by ^{1}H NMR spectroscopy, leading to the corresponding Rh$^{\text{I}}$ carbonyl complex **7** (Scheme 4).[15] Alternatively, the obtained gas was diffused into another Schlenk tube connected by a metallic bridge and filled with a THF solution of **6**. In this case, the same result was obtained, although the ligand exchange reaction (**6** to **7**) takes much longer (24 h).[15]

Taking into account that the phosphorus center was not directly involved, we conclude that the reaction probably proceeds by a Peterson olefination type reaction[16] instead of a Wittig type reaction (Scheme 5). The addition of the first equivalent of CO_2 probably gives the intermediate **8** featuring a silaketene and a silaketone function. Since silaketene derivatives are known to be labile molecules,[17,18] the pentacoordinate silaketene **8** could readily release CO to give the transient oxadisilirene derivative **9**. After a similar process with a second equivalent of CO_2, the 2,4-dioxabicyclo[1.1.0]tetrasilane derivative **10** could react with a third equivalent of CO_2, leading to **11**.[19] Since intermediate **11** cannot be involved in a Peterson type reaction, the last CO-releasing reaction is probably assisted by the nucleophilic attack of one of the phosphine ligands, leading to **12** and then **13** after CO elimination. Finally, the highly reactive tricyclic silicate **13**[20] would be trapped by a fourth equivalent of CO_2 to give the isolated aminosilicate **5**. Compound **5** is highly stable, and no decomposition was observed even after heating at 180 °C for 12 h.



Scheme 4. The reaction of disilyne derivative **2** with CO_2 in the presence of {Rh$^{\text{I}}$(cod)} complex **6**.



Scheme 5. Proposed reaction mechanism.

The stable phosphonium silaylide **3** also rapidly reduces CO_2 at room temperature, producing an original *P*-chiral tricyclic phosphine **14** with an oxygen atom bridging silicon and phosphorus atoms, which was isolated in 32% yield (Scheme 6). Only one singlet signal was observed in the ^{31}P NMR spectrum ($\delta = 103$ ppm), thus indicating the diastereoselective formation of **14** starting from a mixture of diastereomers of **3** (85:15). A similar observation was made for the reaction of phosphonium silaylide **3** with acetylenes, which also leads, by kinetic resolution, to the diastereoselective formation of *P*-chiral tricyclic phosphines.[8a] The structure of **14** was unambiguously confirmed by an X-ray diffraction analysis (see the Supporting Information).

In contrast to the case of **2**, in which the phosphine ligand only assists the CO elimination, the reaction of **3** with CO_2 involves the phosphine center, probably by a sila-Wittig type reaction (Scheme 6).[8b] Although the reaction pathway is

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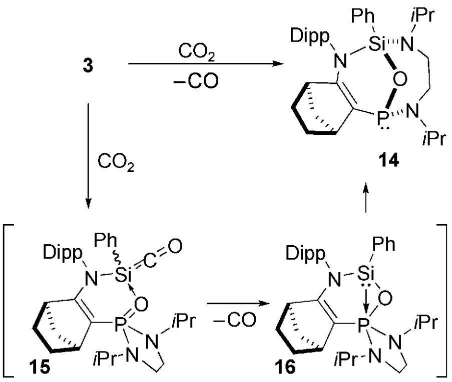

Scheme 6. The reaction of phosphonium silaylide **3** with CO_2.

different, we could postulate here also the formation of a pentacoordinate silaketene **15** as a key intermediate, which loses a molecule of CO leading to the transient pentacoordinate phosphorus species **16**. Then, a ligand coupling reaction[8a,21] would lead to the isolated phosphine **14**. The formation of CO was confirmed by ligand exchange using $\{Rh^I(cod)\}$ complex **6** as trapping agent.

In summary, we have succeeded in the synthesis of the first stable disilyne bis(phosphine) adduct **2**, which has been isolated and fully characterized. Structural and theoretical investigations highlight a peculiar ligand effect of phosphine towards the Si^I center compared to other ligands. Indeed, the phosphine ligands behave as weakly σ-donating ligands without π-accepting character inducing a strong π donor–acceptor character of the Si^I centers. As a consequence, the Si–Si fragment in **2** presents a certain multiple-bond character. Furthermore, we also described the first non-metal-mediated direct CO_2 reduction to CO at room temperature using disilyne derivative **2** as well as silaylide **3**. Taking into account the growing interest in the development of new processes for the efficient utilization of the abundant and renewable CO_2 resource,[22] especially mediated by metal-free systems,[23,24] the two reactions presented herein are of great importance. Particularly, the reduction of CO_2 to the more reactive CO by oxygen abstraction is known to be attractive but difficult in practice owing to the large thermodynamic input required for the process (532 kcal mol^{-1}).[25] Although several reductions of CO_2 to CO have been reported,[26–29] the direct reduction using nonmetallic reagents at room temperature is still an elusive goal.[30] The research on other applications of the new disilyne adduct **2** as well as on the CO production by CO_2 reduction using simplified systems are under active investigation.

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