

NHC Supersilyl Silver Complex [Ag(IPr)Si*t*Bu₃] as a Promising Agent for Substitution Reactions

Frauke Schödel,^[a] Michael Bolte,^[a] Matthias Wagner,^[a] and Hans-Wolfram Lerner^{*[a]}

Abstract. The NHC supersilyl silver complex [Ag(IPr)Si*t*Bu₃] (IPr = NHC_{IPr}) was prepared by treatment of Ag(IPr)Cl with Na(thf)₂[Si*t*Bu₃] in benzene/thf at room temperature. X-ray quality crystals of the NHC supersilyl silver complex [Ag(IPr)Si*t*Bu₃] (monoclinic, space group *P*2₁/*m*) were grown from heptane at room temperature. The ²⁹Si NMR spectrum of a solution of [Ag(IPr)Si*t*Bu₃] in C₆D₆ revealed two doublets caused by coupling to ¹⁰⁷Ag and ¹⁰⁹Ag nuclei. We further investi-

gated the possibility of a conversion of triel halides EX₃ by treatment with [Ag(IPr)Si*t*Bu₃]. At ambient temperature the reaction of [Ag(IPr)Si*t*Bu₃] with an excess of EX₃ yielded *t*Bu₃SiEX₂ (E = B, Al; X = Cl, Br; E = Ga; X = Cl) and IPr·EX₃ (EX₃ = BCl₃, BBr₃, AlCl₃, AlBr₃, GaCl₃). The identity of *t*Bu₃SiEX₂ and IPr·EX₃ was confirmed by comparison with authentic samples.

Introduction

Over the past decades silanides [SiR₃][−] (R = organyl) have been widely investigated in basic academic research.^[1–5] Both supported and unsupported silanides are potent nucleophiles and it has been shown that their donor strength correlates to their reduction behavior.^[6] For instance K[Si*t*Bu₃] as a very strong Brønsted base already deprotonates benzene at room temperature.^[6] It is therefore not surprising, that the supersilyl anion [Si*t*Bu₃][−] can also easily release one electron to form the related supersilyl radical ·Si*t*Bu₃. As shown in Figure 1, one can consider four possible isomers of the supersilyl radical ·Si*t*Bu₃.^[7]

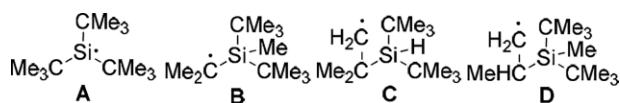


Figure 1. Isomers of the supersilyl radical ·Si*t*Bu₃ (A).

DFT calculations have shown that the silicon-centered radical ·Si*t*Bu₃ (A) is not the lowest-energy structure. Instead the carbon-centered radical B, where a methyl group has migrated from one *t*Bu group to the silicon atom, is significantly lower in energy than A. These calculations also revealed that structures C and D formed by 1,2 H-shifts are more disfavored than B.^[7,8]

In many cases Na[Si*t*Bu₃]^[4,6] is able to reduce transition metal cations to the respective elements.^[4] For example by the reaction of Ag⁺ with supersilylsodium elemental silver along with the supersilyl radical dimer *t*Bu₃SiSi*t*Bu₃ was formed.^[6]

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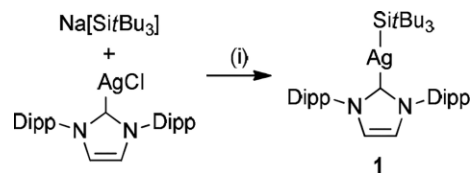
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To gain supersilyl silver complexes there is a need for complexation of Ag⁺ to lower the redox potential. In pursuit of a stable silver supersilanide we decided to treat NHC-stabilized Ag⁺ cations with supersilylsodium.

Disubersilylated trielanes of the type (*t*Bu₃Si)₂EX (E = B, Al, Ga, In, Tl; X = F, Cl, Br)^[9–12] can be prepared from EX₃ and Na[Si*t*Bu₃].^[4,6] However, when EX₃ in pentane was treated with one or two molar equivalents of Na[Si*t*Bu₃]^[4,6] in both cases, reaction of EX₃ with Na[Si*t*Bu₃]^[4,6] exclusively yielded the disubersilylated compound (*t*Bu₃Si)₂EX. Thereby no monosubersilylated compound *t*Bu₃SiEX₂ was formed.^[10] In these reactions the second substitution step must be much faster than the first one. We also suggested that in solution an equilibrium of (*t*Bu₃Si)₂EX and EX₃ with the ion pair [*t*Bu₃Si-E-Si*t*Bu₃][EX₄] exists.^[10] The previously published preparation protocols revealed quite low yields for *t*Bu₃SiEX₂. As an example, *t*Bu₃SiAlBr₂ was obtained in 34% yield by treatment of [Zn(Si*t*Bu₃)₂] with AlBr₃.^[11] The purpose of this paper is to establish a direct conversion of triel halides EX₃ by supersilyl silver with forming monosubersilylated trielanes *t*Bu₃SiEX₂.

Results and Discussion

The NHC-supported silver complex [Ag(IPr)Si*t*Bu₃] (**1**) was prepared by the reaction of [Ag(IPr)Cl] with Na(thf)₂[Si*t*Bu₃]^[4,6] in a mixture of benzene and thf at room temperature, as shown in Scheme 1.



Scheme 1. Preparation route for the NHC supersilyl silver complex [Ag(IPr)Si*t*Bu₃] (**1**). (i) −NaCl; benzene/thf, room temperature.

After removing all volatiles, the residue was extracted into heptane. The NMR spectra of the reaction mixture solely

showed resonances due to the silver complex **1**. Single crystals of **1** were grown from filtered heptane solution at room temperature. The NHC supersilyl silver complex **1** could be characterized by X-ray crystallography (monoclinic, space group $P2_1/m$).

The molecular structure of **1** is shown in Figure 2 (selected bond lengths and angles are in the Figure caption). The complex **1** is located on a crystallographic mirror plane with only half a molecule in the asymmetric unit. Besides two other silyl complexes $[\text{Ag}(\text{IPr})\text{SiEt}(\text{SiMe}_3)_2]$ ^[13] and $[\text{Ag}(\text{IPr})\text{Si}(\text{SiMe}_3)_3]$ ^[13] the supersilyl silver complex **1** is the third IPr silyl silver complex which could be characterized by X-ray crystallography. Due to the bulky supersilyl group the Si–Ag distance in **1** [Si–Ag 2.4244(9) Å] is somewhat longer than the appropriate Si–Ag bonds found in $[\text{Ag}(\text{IPr})\text{SiEt}(\text{SiMe}_3)_2]$ [Si–Ag bond lengths: 2.4006(6) Å]^[13] and $[\text{Ag}(\text{IPr})\text{Si}(\text{SiMe}_3)_3]$ [Si–Ag bond lengths: 2.3936(5) Å]^[13] respectively. One of the *t*Bu groups in **1** is disordered over two position with a site occupation factor of 0.521(9) for the major occupied site. Bond lengths and angles in the two disordered sites were restrained to be equal. The molecules of **1** are arranged in an alternate fashion, as shown in Figure 3.

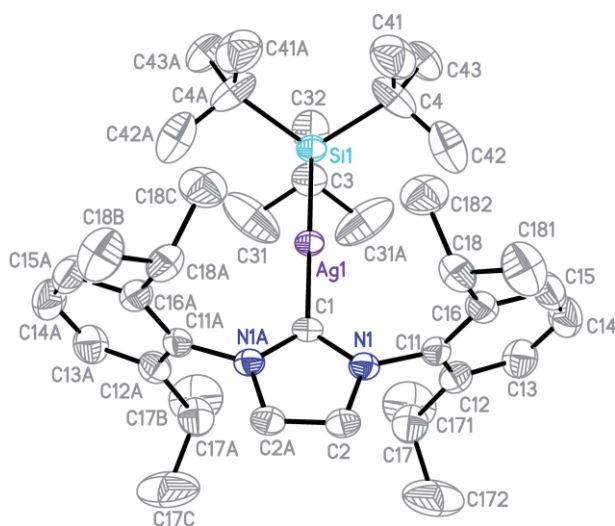


Figure 2. Structure and parameters of the supersilyl silver NHC complex $[\text{Ag}(\text{IPr})\text{SiRtBu}_3]$ (**1**) (monoclinic, space group $P2_1/m$). Selected bond lengths /Å and angles /°: Ag(1)–C(1) 2.155(3), Ag(1)–Si(1) 2.4244(9), Si(1)–C(3) 1.966(4), Si(1)–C(4) 1.974(3), N(1)–C(1) 1.358(2), N(1)–C(2) 1.391(3), N(1)–C(11) 1.442(3), C(1)–N(1)#1 1.358(2), C(2)–C(2)#1 1.358(5), C(1)–Ag(1)–Si(1) 176.54(8), C(3)–Si(1)–C(4) 108.32(13), C(4)#1–Si(1)–C(4) 111.0(3), C(3)–Si(1)–Ag(1) 110.79(13), C(4)–Si(1)–Ag(1) 109.21(12), C(1)–N(1)–C(2) 111.94(19), C(1)–N(1)–C(11) 122.34(17), C(2)–N(1)–C(11) 125.63(19), N(1)–C(1)–N(1)#1 103.7(2), N(1)–C(1)–Ag(1) 128.15(12). Symmetry transformation used to generate equivalent atoms: #1 $x, -y + 1/2, z$.

As shown in Table 1, the structure of **1** features C–Si–C angles smaller than 110°, indicating a negatively polarized Si atom comparable to those of $\text{Na}(\text{thf})_2[\text{Cu}(\text{SiRtBu}_3)_2]$ ^[14] and $\text{Na}(\text{thf})_4[\text{Cu}(\text{SiRtBu}_3)_2]$ ^[14]. Whereas the C–Si–C values of group 12 supersilanides are in the “normal” range for neutral supersilyl compounds.

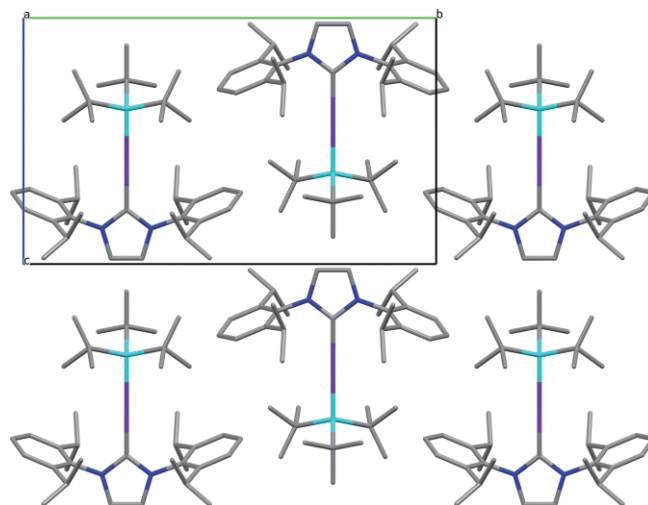


Figure 3. Packing diagram of the supersilyl silver NHC complex $[\text{Ag}(\text{IPr})\text{SiRtBu}_3]$ (**1**).

Table 1. Structural parameters /Å, ° of group 11 and 12 supersilanides.

	M–Si	Si–C ^{a)}	C–Si–C ^{a)}
$\text{Na}(\text{thf})_2[\text{Cu}(\text{SiRtBu}_3)_2]$ [14]	2.361(1) ^{a)}	1.978(5)	108.7(2)
$\text{Na}(\text{thf})_4[\text{Cu}(\text{SiRtBu}_3)_2]$ [14]	2.307(2)	1.972(6)	108.5(2)
$[\text{Ag}(\text{IPr})\text{SiRtBu}_3]$ (1)	2.424(1)	1.971(3)	109.2(3)
$[\text{Zn}(\text{SiRtBu}_3)_2]$ [15]	2.384(1)	1.943(4)	111.8(2)
$[\text{Cd}(\text{SiRtBu}_3)_2]$ [15]	2.524(2)	1.943(4)	111.9(2)
$[\text{Hg}(\text{SiRtBu}_3)_2]$ [15]	2.495(2)	1.937(8)	112.2(4)

a) Average.

The $^{29}\text{Si}\{^1\text{H}\}$ NMR spectrum of a solution of **1** in C_6D_6 revealed two doublets caused by coupling to ^{107}Ag and ^{109}Ag nuclei, as shown in Figure 4. When considering the ^{29}Si spectra of the group 11 and 12 supersilanides certain trends can be observed. As is to be expected, the signals of the silicon nuclei of the group 11 as well as the group 12 element supersilanides are shifted upfield as the element is changed from up to down in the periodic table (Table 2).

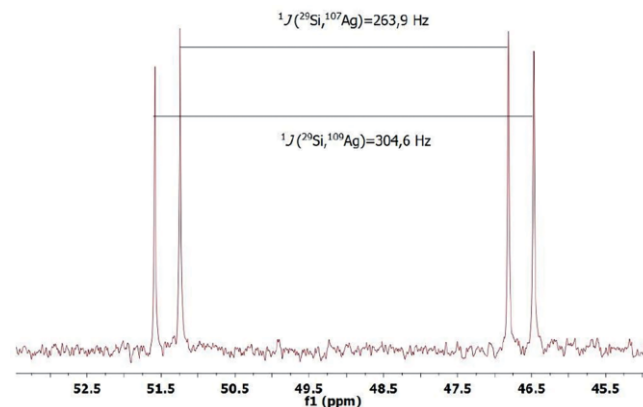


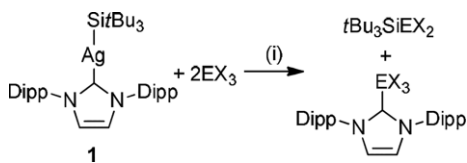
Figure 4. $^{29}\text{Si}\{^1\text{H}\}$ NMR spectrum (59.6 MHz) of $[\text{Ag}(\text{IPr})\text{SiRtBu}_3]$ (**1**) in C_6D_6 ; $\delta = 49.0$ ppm.

We further investigated the possibility of a conversion of triel halides EX_3 by treatment with **1**. As shown in Scheme 2, we found 100% conversion of the silver silanide **1** in all these

Table 2. ^{29}Si NMR resonances of group 11 and 12 supersilanides in C_6D_6 .

	$\delta(^{29}\text{Si}/\text{tBu}_3) / \text{ppm}$
$\text{Na}(\text{thf})_x[\text{Cu}(\text{Si}t\text{Bu}_3)_2]$ [14]	23.2
$[\text{Ag}(\text{IPr})\text{Si}t\text{Bu}_3]$ (1)	49.0
$[\text{Zn}(\text{Si}t\text{Bu}_3)_2]$ [15]	25.9
$[\text{Cd}(\text{Si}t\text{Bu}_3)_2]$ [15]	47.6
$[\text{Hg}(\text{Si}t\text{Bu}_3)_2]$ [15]	88.3

reactions. Thereby the related monosupersilylated trielanes $t\text{Bu}_3\text{SiEX}_2$ and the NHC complexes $\text{IPr}\cdot\text{EX}_3$ were formed ($\text{E} = \text{B}, \text{Al}$; $\text{X} = \text{Cl}, \text{Br}$; $\text{E} = \text{Ga}$; $\text{X} = \text{Cl}$). The identity of $t\text{Bu}_3\text{SiEX}_2$ as well as $\text{IPr}\cdot\text{EX}_3$ was confirmed by comparison with authentic samples.



Scheme 2. Reaction of triel halides EX_3 ($\text{E} = \text{B}, \text{Al}$; $\text{X} = \text{Cl}, \text{Br}$; $\text{E} = \text{Ga}$; $\text{X} = \text{Cl}$) by treatment with the NHC-supported silver complex $[\text{Ag}(\text{IPr})\text{Si}t\text{Bu}_3]$ (**1**). i) $-\text{AgX}$, cyclohexane, room temperature.

Experimental Section

All experiments were carried out in a dry argon or nitrogen atmosphere using standard Schlenk and glove box techniques. Alkane solvents were dried with sodium and freshly distilled prior to use. Benzene and thf were distilled from sodium/benzophenone. $\text{Na}(\text{thf})_x[\text{Si}t\text{Bu}_3]^{[6]}$ was prepared according to the published procedure. All other starting materials were purchased from commercial sources and used without further purification.

Synthesis of $[\text{Ag}(\text{IPr})\text{Si}t\text{Bu}_3]$ (1**).** A mixture of $[\text{Ag}(\text{IPr})\text{Cl}]$ (99 mg, 0.186 mmol) in 4 mL benzene and 0.4 M solution of $\text{Na}(\text{thf})_x[\text{Si}t\text{Bu}_3]^{[6]}$ (0.2 mmol) in 0.5 mL thf was stirred for 1 d at room temperature in an Ar-atmosphere. The NMR spectra of the reaction mixture showed resonances due to the silver complex **1**. After removing all volatiles, the residue was extracted into 10 mL heptane. Colorless crystals of **1** were grown from filtered heptane solution at room temperature. Yield: 110 mg (85%). ^1H NMR (300.1 MHz, C_6D_6): $\delta = 1.07$ [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 1.25 [s, 27 H, $\text{C}(\text{CH}_3)_3$], 1.43 [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 2.58 [m, 4 H, $\text{CH}(\text{CH}_3)$], 6.35 (s, 2 H, =CH), 7.05–7.23 ppm (m, ArH). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.5 MHz, C_6D_6): $\delta = 23.7$ [$\text{CH}(\text{CH}_3)$], 24.0 [d, $^3J_{13\text{C},107/109\text{Ag}} = 9.8$ Hz; $\text{C}(\text{CH}_3)_3$], 24.6 [$\text{CH}(\text{CH}_3)$], 28.6 [$\text{CH}(\text{CH}_3)$], 33.4 [d, $^4J_{13\text{C},107/109\text{Ag}} = 3.8$ Hz; $\text{C}(\text{CH}_3)_3$], 122.0 (=CH), 123.8, 130.1, 135.1, 145.7 ppm (ArC). $^{29}\text{Si}\{^1\text{H}\}$ NMR (59.6 MHz, C_6D_6): $\delta = 49.0$ (d, $^1J_{29\text{Si},107\text{Ag}} = 263.9$ Hz; d, $^1J_{29\text{Si},109\text{Ag}} = 304.6$ Hz; $\text{Si}t\text{Bu}_3$) ppm. $\text{C}_{39}\text{H}_{63}\text{AgN}_2\text{Si}$ (695.88): calcd. C 67.31, H 9.13; N 4.03%; found: C 66.42, H 9.03, N 3.57%.

Reaction of **1 with EX_3 :** A solution of $[\text{Ag}(\text{IPr})\text{Si}t\text{Bu}_3]$ (**1**) for BCl_3 : 8 mg, 0.012 mmol; **1** for BBr_3 : 9 mg, 0.013 mmol; **1** for AlCl_3 : 11 mg, 0.016 mmol; **1** for AlBr_3 : 12 mg, 0.017 mmol; **1** for GaCl_3 : 8 mg, 0.012 mmol) in 1 mL cyclohexane was combined with an excess of EX_3 (BCl_3 : 0.11 mmol, BBr_3 : 33 mg, 0.13 mmol, AlCl_3 : 30 mg, 0.23 mmol, AlBr_3 : 53 mg, 0.20 mmol, GaCl_3 : 8 mg, 0.046 mmol). After filtering out insoluble AgX ($\text{X} = \text{Cl}, \text{Br}$) the solutions were exam-

ined by NMR spectroscopy. The resonances of $t\text{Bu}_3\text{SiEX}_2$ ($\text{E} = \text{B}, \text{Al}$; $\text{X} = \text{Cl}, \text{Br}$; $\text{E} = \text{Ga}$; $\text{X} = \text{Cl}$) and $\text{IPr}\cdot\text{EX}_3$ ($\text{IPr}\cdot\text{EX}_3 = \text{IPr}\cdot\text{BCl}_3$, $^{[16]}$ $\text{IPr}\cdot\text{BBr}_3$, $^{[17]}$ $\text{IPr}\cdot\text{AlCl}_3$, $\text{IPr}\cdot\text{AlBr}_3$, $\text{IPr}\cdot\text{GaCl}_3$ $^{[18]}$) were observable in the NMR spectra of these solutions.

^{29}Si NMR resonances of $t\text{Bu}_3\text{SiEX}_2$ recorded by $^1\text{H}/^{29}\text{Si}$ HETCOR NMR spectroscopy in C_6D_{12} : $\delta = -1.6$ ($\text{E} = \text{B}, \text{X} = \text{Cl}$); 0.3 ($\text{E} = \text{B}, \text{X} = \text{Br}$); 14.3 ($\text{E} = \text{Al}, \text{X} = \text{Cl}$); 15.1 ($\text{E} = \text{Al}, \text{X} = \text{Br}$); 13.5 ppm ($\text{E} = \text{Ga}; \text{X} = \text{Cl}$).

Note: Treatment of a mixture of AlX_3 (AlCl_3 : 20 mg, 0.15 mmol, AlBr_3 : 44 mg, 0.16 mmol) in 6 mL heptane with IPr (IPr for AlCl_3 : 50 mg, 0.13 mmol, IPr for AlBr_3 : 50 mg, 0.13 mmol) yielded quantitatively $\text{IPr}\cdot\text{AlX}_3$ according to the NMR spectra.

$\text{IPr}\cdot\text{AlCl}_3$: ^1H NMR (300.1 MHz, C_6D_6): $\delta = 0.94$ [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 1.43 [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 2.71 [m, 4 H, $\text{CH}(\text{CH}_3)$], 6.40 (s, 2 H, =CH), 6.94–7.24 ppm (m, ArH). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.5 MHz, C_6D_6): $\delta = 22.3$ [$\text{CH}(\text{CH}_3)$], 25.5 [$\text{CH}(\text{CH}_3)$], 28.8 [$\text{CH}(\text{CH}_3)$], 124.1 (=CH), 125.1, 131.2, 133.5, 145.3 ppm (ArC).

$\text{IPr}\cdot\text{AlBr}_3$: ^1H NMR (300.1 MHz, C_6D_6): $\delta = 0.94$ [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 1.43 [d, $^3J_{\text{H,H}} = 6.9$ Hz; 12 H; $\text{CH}(\text{CH}_3)$], 2.67 [m, 4 H, $\text{CH}(\text{CH}_3)$], 6.41 (s, 2 H, =CH), 6.95–7.22 ppm (m, ArH). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.5 MHz, C_6D_6): $\delta = 23.7$ [$\text{CH}(\text{CH}_3)$], 25.3 [$\text{CH}(\text{CH}_3)$], 28.7 [$\text{CH}(\text{CH}_3)$], 124.2 (=CH), 125.7, 131.5, 133.6, 145.5 ppm (ArC).

X-ray Crystallography of **1:** Data were collected on a STOE IPDS II two-circle diffractometer with a Genix Microfocus tube with mirror optics using $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073$ Å) and were scaled using frame scaling procedure in the X-AREA program system. $^{[19]}$ An empirical absorption correction with the program PLATON was performed for both structures. The structure $^{[20]}$ was solved by direct methods using the program SHELXS and refined with full-matrix least-squares on F^2 using the program SHELXL. $^{[21]}$

Crystallographic data (excluding structure factors) for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository number CCDC-1941530 (**1**) (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, <http://www.ccdc.cam.ac.uk>)

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Keywords: Silanide; Silicon; Silver; Triel; X-ray diffraction

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