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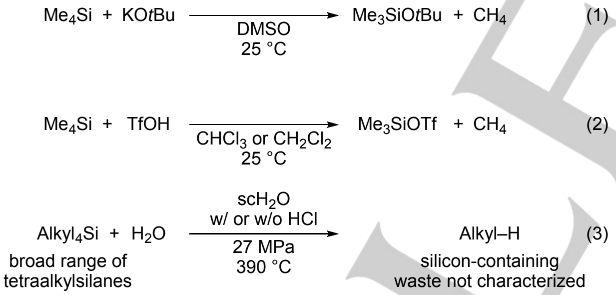
Cleavage of Unactivated Si–C(sp^3) Bonds with Reed's Carborane Acids: Formation of Known and Unknown Silylium Ions

Qian Wu, Zheng-Wang Qu,* Lukas Omann, Elisabeth Irran, Hendrik F. T. Klare, and Martin Oestreich*

Dedicated to Professor Douglas W. Stephan on the occasion of his 65th birthday

Abstract: An efficient method for the benzenium-ion-mediated cleavage of inert Si–C(sp^3) bonds is reported. By this, various tetraalkylsilanes are converted into the corresponding counteranion-stabilized silylium ions. The reaction is chemoselective in the case of hexamethyldisilane. Computations reveal a mechanism with backside attack of the proton at one of the alkyl groups. Likewise, several activated Si–C(sp^n) bonds (n = 3–1) react equally well, and the protocol can be extended to the generation of stannylium ions.

Me_4Si is considered inert, and that holds true for any quaternary, fully alkyl-substituted silane. Examples of cleavage of unactivated Si–C(sp^3) bonds are exceedingly rare and essentially limited to the smallest member Me_4Si [Scheme 1, Eq. (1) and Eq. (2)]. Both hard oxygen nucleophiles[1] and acids[2] have been shown to convert Me_4Si into an oxygenated silane with release of methane.[3,4] Itami and co-workers found that tetraalkylsilanes are rapidly degraded to the corresponding alkanes in supercritical water at high temperature and pressure [Scheme 1, Eq. (3)];[5] the addition of mineral acid accelerates these reactions.



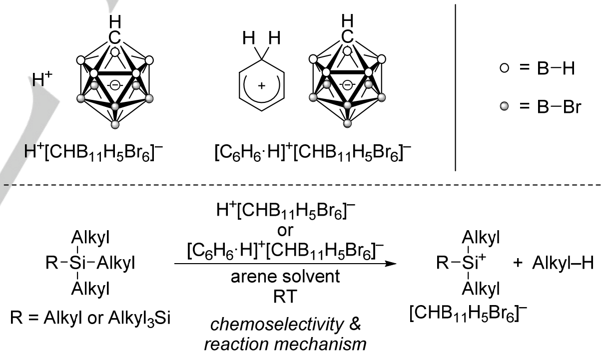
$$Me_4Si + KOtBu \xrightarrow[25\ °C]{DMSO} Me_3SiOtBu + CH_4 \qquad (1)$$

$$Me_4Si + TfOH \xrightarrow[25\ °C]{CHCl_3\ or\ CH_2Cl_2} Me_3SiOTf + CH_4 \qquad (2)$$

$$\underset{\text{broad range of tetraalkylsilanes}}{Alkyl_4Si} + H_2O \xrightarrow[\text{27 MPa, 390 °C}]{scH_2O,\ w/\ or\ w/o\ HCl} \underset{\text{silicon-containing waste not characterized}}{Alkyl–H} \qquad (3)$$

Scheme 1. Reported methods for Si–C(sp^3) bond cleavage. DMSO = dimethyl sulfoxide. Tf = trifluoromethanesulfonyl; sc = supercritical.

Inspired by the literature precedence, we entertained the idea of employing Reed's carborane acids[6] such as $H^+[CHB_{11}H_5Br_6]^–$[7] as well as cognate protonated benzene $[C_6H_6{\cdot}H]^+[CHB_{11}H_5Br_6]^–$[8] for the cleavage of rather inert Si–C(sp^3) bonds (Scheme 2).[9] Unlike the reported methods,[2,5] its successful implementation would enable the generation of counteranion-stabilized silylium ions[10] directly from completely unfunctionalized precursors. This strategy would be complementary to the common preparations of silylium ions[11] by either hydride abstraction from hydrosilanes[12] or the allyl-leaving-group approach of sterically hindered systems.[13] We dislcose here the chemoselective heterolysis of unactivated Si–C(sp^3) bonds by protonation to arrive at "exceptionally" clean silylium ions. Computations also reveal a rather unexpected mechanism of the bond-breaking event.



Scheme 2. Representative carborane acids and their planned use in the generation of counteranion-stabilized silylium ions.

We began our investigation with $H^+[CHB_{11}H_5Br_6]^–$ and quickly found that tetraalkylsilanes are indeed converted into the corresponding counteranion-stabilized silylium ions. However, residual water in $H^+[CHB_{11}H_5Br_6]^–$ and difficulties with its removal were detrimental to the purity of the obtained silylium ion-like species. We therefore turned toward easy-to-purify benzenium ion $[C_6H_6{\cdot}H]^+[CHB_{11}H_5Br_6]^–$. Our reasoning was that it would make no difference to start with either acid as the protonation is performed in benzene. Gratifyingly, $[C_6H_6{\cdot}H]^+[CHB_{11}H_5Br_6]^–$ transformed several tetraalkylsilanes into counteranion-stabilized trialkylsilylium ions of remarkable purity with gas evolution (Table 1); nBu$_4$Si was included into the survey as it did

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[+] X-ray crystal-structure analysis.

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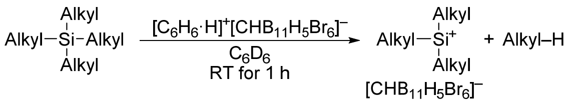
allow for the conclusive verification of the released alkane, that is *n*-butane, by ^{1}H NMR spectroscopy (entry 3; see the Supporting Information for details). The chemoselectivity of 1° over 2° and 3° alkyl substitution, respectively, was also demonstrated and is explained by steric hindrance (entries 4 and 5). Strikingly, chemoselective cleavage of the Si–C(sp^3) was also possible in the presence of the less stable Si–Si bond (bond dissociation enthalpies[14] for Me$_3$Si–Me = 395 ± 4 kJ/mol versus Me$_3$Si–SiMe$_3$ = 333 ± 6 kJ/mol). Again, we attribute this to the sterically more accessible methyl group. This enabled the formation of the previously unavailable silyl-substituted silylium ion-like [Me$_2$(Me$_3$Si)Si]$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ (Scheme 3, top); its molecular structure was established by X-ray diffraction (Scheme 3, bottom). Conversely, dodecamethylcyclohexasilane did not yield the targeted silylium ion; a complex mixture was obtained, probably as a result of rearrangement reactions.[15]

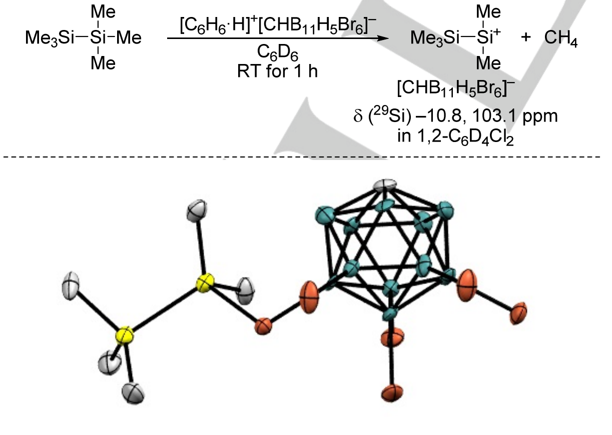
Table 1: Protonation of unactivated tetraalkylsilanes.



$$\text{Alkyl}_4\text{Si} \xrightarrow[\text{RT for 1 h}]{[C_6H_6\cdot H]^+[CHB_{11}H_5Br_6]^-,\ C_6D_6} \text{Alkyl}_3\text{Si}^+[CHB_{11}H_5Br_6]^- + \text{Alkyl–H}$$

Entry	Silane	Alkane	Silylium ion	δ (^{29}Si) [ppm][a]
1	Me$_4$Si[b]	Me–H (methane)	Me$_3$Si$^+$	93.5
2	Et$_4$Si	Et–H (ethane)	Et$_3$Si$^{+[c]}$	100.0
3	*n*Bu$_4$Si	*n*Bu–H (*n*-butane)	*n*Bu$_3$Si$^+$	98.0
4	*i*Pr$_2$Me$_2$Si	Me–H (methane)	*i*Pr$_2$MeSi$^+$	100.9
5	*t*BuMe$_3$Si	Me–H (methane)	*t*BuMe$_2$Si$^+$	98.0

[a] Determined by ^{1}H/^{29}Si HMQC NMR spectroscopy (500/99 MHz, 298 K, optimized for *J* = 7 Hz) in 1,2-Cl$_2$C$_6$D$_4$. [b] 2.0 equiv used due to the volatility of Me$_4$Si. [c] The solid state ^{29}Si NMR spectrum (CPMAS) of Et$_3$Si$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ has been reported: δ (^{29}Si) = 111.8 and 106.2 ppm.[10b]



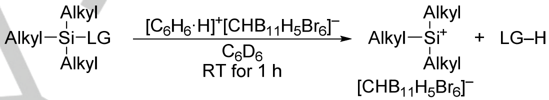
$$\text{Me}_3\text{Si–SiMe}_3 \xrightarrow[\text{RT for 1 h}]{[C_6H_6\cdot H]^+[CHB_{11}H_5Br_6]^-,\ C_6D_6} \text{Me}_3\text{Si–SiMe}_2^+[CHB_{11}H_5Br_6]^- + CH_4$$

δ (^{29}Si) –10.8, 103.1 ppm in 1,2-C$_6$D$_4$Cl$_2$

Scheme 3. Chemoselective protonation of disilane (top) and molecular structure of the silyl-substituted silylium ion-like Me$_2$(Me$_3$Si)Si$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ (bottom; thermal ellipsoids are shown at the 50% probability level; hydrogen atoms are omitted for clarity); selected bond length (Å) and angles (°): Si–Br: 2.444(4); Si–Si: 2.354(5); Me–Si–SiMe$_3$: 115.5(5); Me–Si–SiMe$_3$: 116.0(6); Me–Si–Me: 116.3(8).

To highlight the generality of the method, we subjected representative trialkylsilanes decorated with leaving groups known to be cleaved by protons (Table 2).[16] Activated Si–C(sp^3) bonds as in allyl-[2,17] and surprisingly benzylsilanes[18] cleanly afforded the counteranion-stabilized trialkylsilylium ions (entries 1–3). Likewise, vinyl-[19] and arylsilanes[20] underwent protodesilylation (entries 4 and 5). These reactions are initiated by protonation of the unsaturated unit and involve the intermediacy of β-silicon-stabilized carbocations.[21] In turn, the cleavage of an Si–C(sp) bond by protonation would pass through a β-silicon-substituted vinyl cation.[22] Such reaction was possible but less clean. For example, alkynyl-substituted trimethylsilane led to a complex mixture but [Me$_3$Si]$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ was nevertheless isolated in pure form after precipitation and washing with 1,2-Cl$_2$C$_6$D$_4$ (entry 6).

Table 2: Protonation of activated trialkyl(organo)silanes.

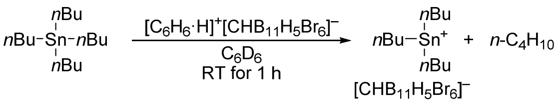


$$\text{Alkyl}_3\text{Si–LG} \xrightarrow[\text{RT for 1 h}]{[C_6H_6\cdot H]^+[CHB_{11}H_5Br_6]^-,\ C_6D_6} \text{Alkyl}_3\text{Si}^+[CHB_{11}H_5Br_6]^- + \text{LG–H}$$

Entry	Silane	LG–H	Silylium ion	δ (^{29}Si) [ppm][a]
1	(allyl)Me$_3$Si	Allyl–H (propene)	Me$_3$Si$^+$	93.1
2	(allyl)*i*Pr$_3$Si	Allyl–H (propene)	*i*Pr$_3$Si$^+$	103.6
3	BnMe$_3$Si	Bn–H (toluene)	Me$_3$Si$^+$	93.5
4	Me$_3$(vinyl)Si	Vinyl–H (ethylene)	Me$_3$Si$^+$	93.4
5	Me$_3$PhSi	Ph–H (benzene)	Me$_3$Si$^+$	93.3
6	Me$_3$(*n*BuC≡C)Si	—[b]	Me$_3$Si$^+$	93.6

[a] Determined by ^{1}H/^{29}Si HMQC NMR spectroscopy (500/99 MHz, 298 K, optimized for *J* = 7 Hz) in 1,2-Cl$_2$C$_6$D$_4$. [b] Formation of hex-1-yne not verified. LG = leaving group.

The method was also applicable to the cleavage of unactivated Sn–C(sp^3) bonds and, hence, to the synthesis of trialkylstannylium ions[13,23] stabilized by the counteranion (Scheme 4). Treatment of *n*Bu$_4$Sn with [C$_6$H$_6$·H]$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ gave [*n*Bu$_3$Sn]$^+$[CHB$_{11}$H$_5$Br$_6$]$^-$ with a highly deshielded tin atom. Its ^{119}Sn chemical shift of δ 476.8 ppm is in accordance with repor-ted [*n*Bu$_3$Sn]$^+$[CMeB$_{11}$Me$_{11}$]$^-$ at δ 454.3 ppm.[23b]

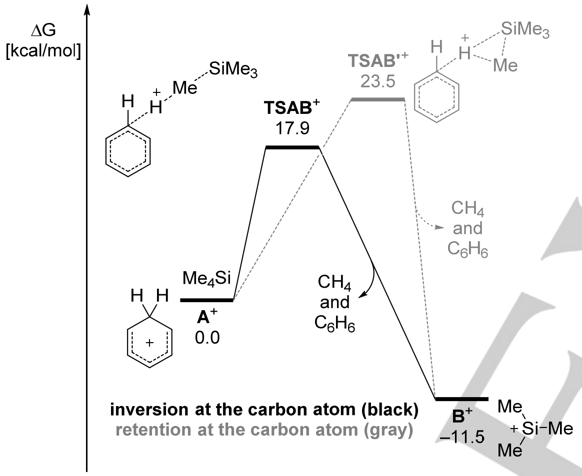


$$n\text{Bu}_4\text{Sn} \xrightarrow[\text{RT for 1 h}]{[C_6H_6\cdot H]^+[CHB_{11}H_5Br_6]^-,\ C_6D_6} n\text{Bu}_3\text{Sn}^+[CHB_{11}H_5Br_6]^- + n\text{-}C_4H_{10}$$

δ (^{119}Sn) 476.8 ppm in 1,2-C$_6$D$_4$Cl$_2$

Scheme 4. Protonation of tetraalkylstannane for the generation of the corresponding stannylium ion.

Density functional theory (DFT) calculations were performed at the PW6B95-D3/def2-QZVP + COSMO-RS(benzene) // TPSS-D3/def2-TZVP + COSMO(benzene) level of theory[24] to provide insight into the mechanism of the protonation of Me_4Si induced by Wheland complex **A**$^+$ (Scheme 5). In solution, **A**$^+$ exists as a separate ion together with the counteranion $[CHB_{11}H_5Br_6]^-$. The proton of **A**$^+$ attacks at one of the methyl groups in Me_4Si either opposite to the silicon atom through **TSAB**$^+$ or from the side of the Si–C(sp^3) bond through **TSAB'**$^+$. The former scenario proceeds with inversion of the configuration at the carbon atom and is kinetically more favorable ($\Delta\Delta G^\ddagger$ = 5.6 kcal/mol). That step releases CH_4 and the silylium ion **B**$^+$ and is exergonic by 11.5 kcal/mol. The same applies to the protonation of the Sn–C(sp^3) bond yet with a lower barrier of 13.2 kcal/mol (see the Supporting Information for details).



Scheme 5. DFT computed Gibbs free energy profile (in kcal/mol at 298 K and 1 mol/L reference state) for the protonation of Me_4Si omitting the counteranion. Computations with involvement of $[CHB_{11}H_5Br_6]^-$ are to be found in the Supporting Information.

The method disclosed in this work enables the facile and clean generation of counteranion-stabilized silylium ions from fully alkylated silanes as well as stannanes. The protonation and cleavage of the largely inert Si–C(sp^3) bond have been achieved with $[C_6H_6{\cdot}H]^+[CHB_{11}H_5Br_6]^-$, one of Reed's arenium ions. The reaction involves backside attack of the proton at the Si–C(sp^3) bond, resulting in inversion of the configuration at the departing carbon atom. Other Si–C linkages can be cleaved by the same procedure but, depending on the leaving group, e.g., allyl or vinyl, follow different mechanisms with the involvement of β-silicon-stabilized carbocations.

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Keywords: Brønsted acids • carboranes • density functional calculations • protonation • silylium ions

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Suggestion for the Entry for the Table of Contents

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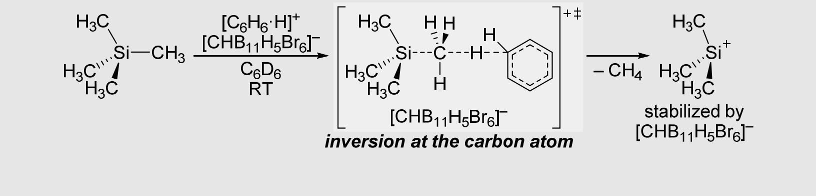

Watch your back! The benzenium ion $[C_6H_6 \cdot H]^+[CHB_{11}H_5Br_6]^-$ enables the cleavage of inert Si–C(sp^3) bonds, yielding counteranion-stabilized silylium ions. As shown for Me_4Si, the protonation proceeds with inversion at the carbon atom (see scheme). The method is broadly applicable to tetraalkylsilanes and extends to various activated Si–C(sp^n) bonds (n = 3–1). The related stannylium ions can be made in the same way.

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