Silicon Chemistry

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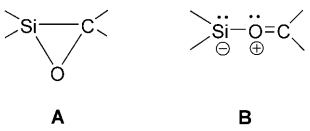
Convenient Access to Monosilicon Epoxides with Pentacoordinate Silicon**

Rajendra S. Ghadwal, Sakya S. Sen, Herbert W. Roesky, Markus Granitzka, Daniel Kratzert, Sebastian Merkel, and Dietmar Stalke**

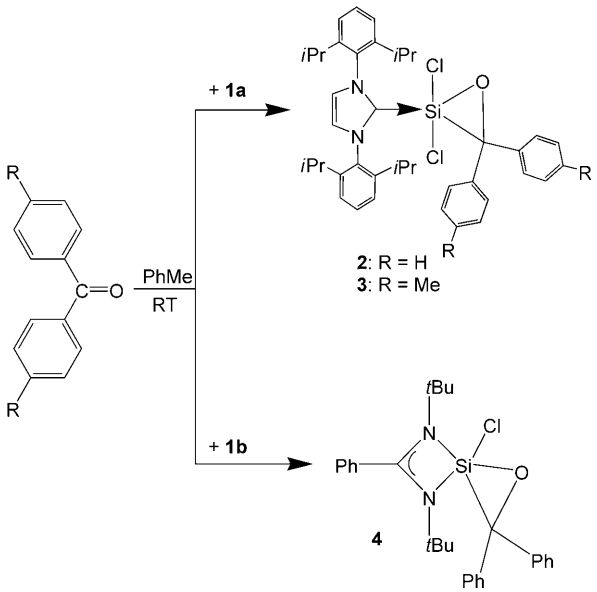
Dedicated to Professor S. S. Krishnamurthy on the occasion of his 70th birthday

"If carbonyl compounds have been said to be virtually the backbone of organic synthesis, the epoxides correspond to at least one of the main muscles"—D. Seebach

Epoxides play an important role in synthetic organic chemistry.[1] They undergo nucleophilic ring-opening reactions with high stereo- and regioselectivity. Unequivocally, analogous organosilicon species would be interesting reagents for organic synthesis, although these compounds are not easily accessible. Formation of silicon–carbon bonds by reaction of silylenes with organic substrates has attracted considerable interest in the synthesis of organosilicon compounds.[2] Strained small ring compounds containing a silicon atom are of importance due to their molecular structure and high reactivity resulting from ring-strain energy.[3] Among three-membered silicon ring compounds, silacyclopropanes and silacyclopropenes have been prepared and studied extensively.[4,5] They are much more reactive than their carbon analogues. Likewise there has been considerable theoretical interest[6] in the formation and stability of silaoxiranes, which suggested that strain energy alone may not be the only criterion for isolating these compounds. Computational studies revealed that neutral silaoxiranes should be strongly stabilized by adding an anion like fluoride.[6a] Therefore, we were interested whether this stabilization could be achieved by a neutral carbon- or nitrogen-donor ligand to isolate uncharged silaoxiranes, which are otherwise assumed to be very unstable compounds. Furthermore, silaoxirane **A** and the silacarbonyl ylide **B** (Scheme 1) are assumed to be intermediates[7,8] in the reaction of transient or stable silylenes with carbonyl compounds. The first evidence for the formation of transient silaoxirane (siloxacyclopropane) was presented in 1977 by Ando et al.[7a] in the reaction of thermally generated silylene with benzophenone. In 1982 the same group succeeded in the isolation of a heavily substituted stable silaoxirane,[9] containing a four-coordinate silicon atom, by reaction of photochemically generated silylene with a cyclic ketone. To the best of our knowledge, this is the only structurally characterized silaoxirane to date. Formation of cyclic silaoxanes (or silylenol ethers) has been reported[8] by the reaction of silylenes with carbonyl compounds. The involvement of either silaoxirane (**A**) or silacarbonyl ylide (**B**) as intermediates was proposed. Surprisingly, there is no report on the isolation of a stable silaoxirane. Recently, we isolated N-heterocyclic carbene (NHC) stabilized dichlorosilylene L·$SiCl_2$ (**1a**; L = 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene) by dehydrochlorination of $HSiCl_3$.[10] Accordingly,[6a] NHC-stabilized silylene **1a** should react with ketones to give silaoxiranes. Herein, we report on the preparation of stable silaoxiranes **2** and **3** by reaction of **1a** with ketones (Scheme 2). The monosilicon epoxides **2** and **3** are the first examples containing a pentacoordinate silicon atom and are stabilized by the σ-



Scheme 1. Silaoxirane **A** and silacarbonyl ylide **B**.



Scheme 2. Synthesis of silaoxiranes **2**–**4** (**1a** = L·$SiCl_2$; L = 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene and **1b** = PhC(N*t*Bu)$_2$SiCl)

[*] Dr. R. S. Ghadwal, S. S. Sen, Prof. Dr. H. W. Roesky, M. Granitzka, D. Kratzert, S. Merkel, Prof. Dr. D. Stalke
Institut für Anorganische Chemie
Georg-August-Universität Göttingen
Tammannstrasse 4, 37077 Göttingen (Germany)
Fax: (+49) 551-393-373
E-mail: hroesky@gwdg.de
dstalke@chemie.uni-goettingen.de

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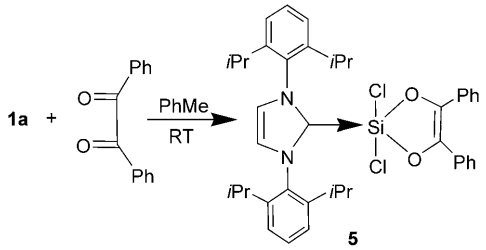
donating NHC ligand. Inspired by the successful isolation of monosilicon epoxides **2** and **3**, we extended our investigation to N-donor base stabilized monochloro silylene[11] PhC-(N*t*Bu)$_2$SiCl (**1b**) to afford silaoxirane **4** by reaction with benzophenone (Scheme 2). Formation of **2**–**4** as stable compounds indicates that silaoxiranes can be electronically stabilized by neutral σ-donor ligands. Moreover, 2,5-dioxa-1-silacyclopent-1-enes were prepared by reaction of a stable silylene[8a] as well as thermally or photochemically generated silylenes[12] with diketones. For comparison we treated **1a** with 1,2-diphenyl-1,2-ethanedione (benzil) and obtained 2,5-dioxa-1-silacyclopent-1-ene **5** (Scheme 3), even when an excess of **1a** was used.



Scheme 3. Synthesis of **5** (**1a** = L·SiCl$_2$; L = 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene)

Compounds **2**–**5** form colorless crystals, are stable under inert atmosphere, and are soluble in common organic solvents. The molecular structures of **2**–**5** were established by single-crystal X-ray diffractions studies.[13] The ^{1}H NMR spectra of **2**, **3**, and **5** show one set of resonances for the NHC ligand coordinated to the silicon atom. The ^{1}H NMR spectrum of **4** exhibits two resonances for the amidinate moiety. In addition, **2**–**5** show ^{1}H NMR signals for the phenyl group attached to the OSiC or O$_2$SiC$_2$ ring. The ^{29}Si NMR spectra of **2**–**5** exhibit sharp signals ($\delta = -123.85$, -123.39, -115.53, and -99.50 ppm, respectively) consistent with pentacoordinate[14] silicon.

The molecular structures of silaoxiranes **2** and **3** are shown in Figures 1 and 2, respectively.[13] Compound **2** crystallizes in the monoclinic space group $P2_1/c$ with one toluene molecule in the asymmetric unit, and **3** in the orthorhombic space group *Pbca* with half a molecule of *n*-hexane in the asymmetric unit. The silicon atom in each of **2** and **3** is pentacoordinate with a distorted trigonal-bipyramidal geometry. The NHC ligands in **2** and **3** occupy an axial position with C28-Si1-C1 as largest bond angle. The Si←C bond lengths in **2** (1.9653(15) Å) and **3** (1.9724 (17) Å) are comparable with that of **1a** (1.985(4) Å). Both chlorine atoms in **2** and **3** occupy the equatorial positions. The differences in Si–Cl bond lengths for **2** (2.1175(6), 2.0690(6) Å) and **3** (2.0942(7), 2.0759(6) Å) may be due to steric hindrance. The Si–Cl distances in **2** and **3** are noticeably different and shorter than the average bond length in **1a** (2.1664(16) Å). The Si–O bonds in **2** (1.6520(10) Å) and **3** (1.6486(13) Å) are slightly shorter than that in the reported silaoxirane,[9] presumably due to the presence of two electronegative chlorine atoms at the silicon atom. The Si1–C28 bond lengths in **2** (1.8834(15) Å) and **3** (1.8924(18) Å) are comparable. The three-membered strained rings in **2** and **3** each consist of non-isosceles triangles.

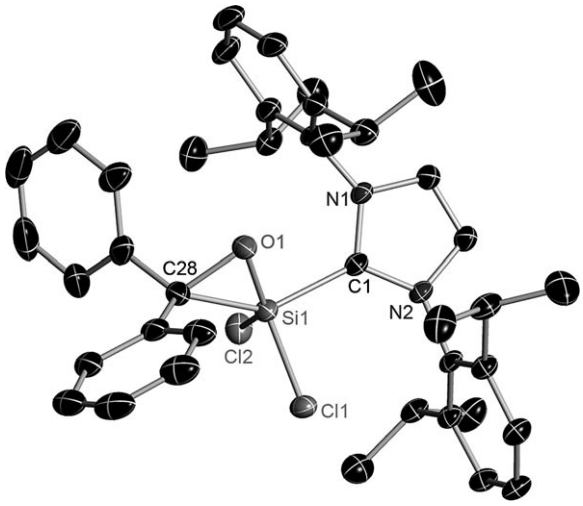


Figure 1. Molecular structure of **2**; anisotropic displacement parameters depicted at 50% probability. A toluene molecule is omitted for clarity. Selected bond lengths [Å] and angles [°]: Si1–O1 1.6520(10), Si1–C2 1.8834(15), Si1–C1 1.9653(15), Si1–Cl1 2.0690(6), Si1–Cl2 2.1175(6); O1-Si1-C2 50.70(6), O1-Si1-C1 94.54(5), C28-Si1-C1 143.06(6), O1-Si1-Cl1 117.74(4), C28-Si1-Cl1 106.22(5), C1-Si1-Cl1 101.11(4), O1-Si1-Cl2 133.21(5), C28-Si1-Cl2 104.28(5), C1-Si1-Cl2 90.95(4), Cl1-Si1-Cl2 106.55(2).

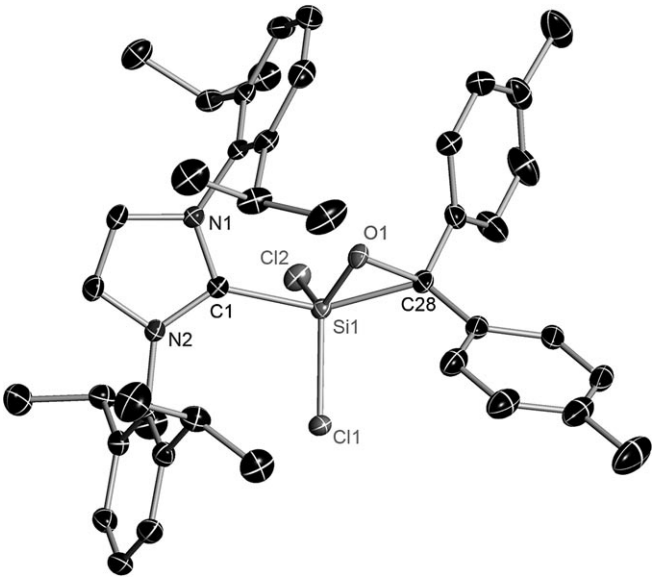


Figure 2. Molecular structure of **3**; anisotropic displacement parameters depicted at 50% probability. Half of an *n*-hexane molecule is omitted for clarity. Selected bond lengths [Å] and angles [°]: Si1–O1 1.6486(13), Si1–C28 1.8924(18), Si1–C1 1.9724(17), Si1–Cl1 2.0759(6), Si1–Cl2 2.0942(7); O1-Si1-C28 50.31(6), O1-Si1-C1 92.40(7), C28-Si1-C1 142.35(7), O1-Si1-Cl1 123.28(5), C28-Si1-Cl1 105.16(6), C1-Si1-Cl1 100.04(5), O1-Si1-Cl2 127.56(5), C28-Si1-Cl2 105.93(6), C1-Si1-Cl2 92.59(5), Cl1-Si1-Cl2 107.03(2).

The molecular structure of **4** is shown in Figure 3. It crystallizes in the monoclinic space group $P2_1/c$.[13] In the

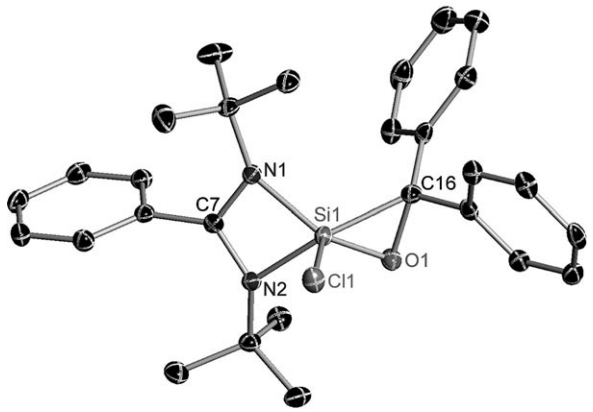


Figure 3. Molecular structure of **4**; anisotropic displacement parameters depicted at the 50% probability level. Two toluene molecules are omitted for clarity. Selected bond lengths [Å] and angles [°]: Si1–O1 1.6534(13), Si1–C16 1.8641(19), Si1–Cl1 2.0708(6), C16-O1-Si1 71.70(9), O1-Si1-N1 133.01(7), O1-Si1-N2 100.28(6), O1-Si1-C16 50.93(7), O1-Si1-Cl1 118.15(5).

spirocyclic structure the Si atom is part of a four- and a three-membered ring. The Si atom exhibits a distorted square-pyramidal geometry. The coordination sites of the Si atom are occupied by the N atoms of the amidinato ligand and one oxygen and one carbon atom from the epoxide ring. The fifth coordination site is occupied by a chlorine atom. The Si–O and Si–C distances of 1.6534(13) and 1.8641(19) Å, respectively, are comparable to those in **2** and **3**. The Si–Cl distance of 2.0708(6) Å in **4** is shorter than that in **1b** (2.156(1) Å) and in agreement with those of **2** and **3**.

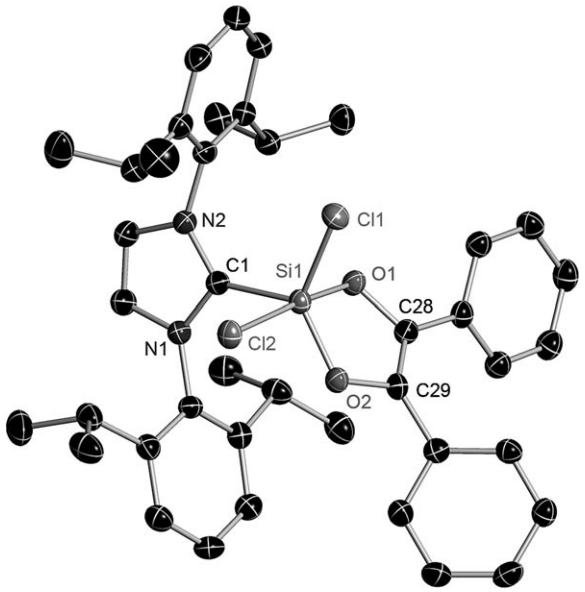


Figure 4. Molecular structure of **5**; anisotropic displacement parameters depicted at 50% probability. The second molecule is omitted for clarity. Selected bond lengths [Å] and angles [°]: Si1–O1 1.7357(15), Si1–O2 1.6734(15), Si1–C1 1.934(2), Si1–Cl1 2.0860(8), Si1–Cl2 2.2227(8), C28–C29 1.348(3), O1–C28 1.378(2), O2–C29 1.403(2); O2-Si1-O1 89.59(7), O1-Si1-C1 91.69(8), O1-Si1-Cl1 92.88(6), O2-Si1-Cl2 86.83(5), C1-Si1-Cl2 87.58(6), Cl1-Si1-Cl2 91.81(3), O2-Si1-C1 123.92(8), C1-Si1-Cl1 114.16(7), O1-Si1-Cl2 175.13(6).

The molecular structure of 2,5-dioxa-1-silacyclopent-1-ene **5** is shown in Figure 4. Compound **5** crystallizes in the monoclinic space group $P2_1/c$ with two molecules in the asymmetric unit[13] and features a trigonal-bipyramidal geometry at the pentacoordinate silicon atom. The chlorine atoms in **5** occupy one axial (Cl2) and one equatorial position (Cl1), and as expected their bond lengths differ significantly (axial 2.2034(8), equatorial 2.0814(8) Å). The oxygen atoms of the planar five-membered ring chelate axial and equatorial positions. The NHC ligand occupies an equatorial position, and the Si←C distance (1.939(2) Å) is comparable to those in **2** and **3**.

In summary, we have reported for the first time a convenient method for the preparation of stable silaoxiranes by the reaction of silylenes with ketones. Silaoxiranes **2**–**4** are stable compounds and were fully characterized. They are the first of their type stabilized by neutral σ-donor ligands, and each contains a pentacoordinate silicon atom. They may serve as potential precursors for the preparation of novel organosilicon compounds. Their formation proves that base stabilization allows isolation of silaoxiranes which are otherwise assumed to exist only as intermediates.

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[13] Crystallographic details: A shock-cooled crystal was selected and mounted under nitrogen atmosphere by using X-TEMP2 (T. Kottke, D. Stalke, *J. Appl. Crystallogr.* **1993**, *26*, 615 – 619; D. Stalke, *Chem. Soc. Rev.* **1998**, *27*, 171 – 178). The structures were solved by direct methods (SHELXS) and refined on F^2 by using the full-matrix least-squares methods of SHELXL (G. M. Sheldrick, *Acta Crystallogr. Sect. A* **2008**, *64*, 112 – 122). CCDC 763281 (**2**), 763282 (**3**), 762176 (**4**) and 763283 (**5**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Space group, cell parameters, and R values with $[I > 2\sigma(I)]$: **2**: $P2_1/c$, $a = 11.6851(14)$, $b = 22.919(3)$, $c = 16.2246(19)$ Å, $\beta = 99.855(2)°$, $wR2 = 0.0846$, $R1 = 0.0345$; **3**: $Pbca$, $a = 17.536(3)$, $b = 20.218(3)$, $c = 23.298(4)$ Å, $wR2 = 0.0828$, $R1 = 0.0352$; **4**: $P2_1/c$, $a = 10.763(5)$, $b = 15.487(7)$, $c = 22.345(11)$ Å, $\beta = 94.3340(10)°$, $wR2 = 0.1028$, $R1 = 0.0370$; **5**: $P2_1/c$, $a = 15.947(2)$, $b = 12.9563(15)$, $c = 36.133(5)$ Å, $\beta = 93.878(2)°$, $wR2 = 0.1135$, $R1 = 0.0451$.

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