A β-diketiminate stabilized sila-acyl chloride: systematic access to base-stabilised silicon analogues of classical carbonyl compounds

Dinh Cao Huan Do, Andrey V. Protchenko, M. Ángeles Fuentes, Jamie Hicks, Eugene L. Kolychev, Petra Vasko, and Simon Aldridge*[a]

Abstract: An oxidation/substitution strategy for the synthesis of silicon analogues of classical organic carbonyl compounds is reported, by making use of a novel β-diketiminate supported sila-acyl chloride - the first example of such a compound isolated without the use of a stabilizing Lewis acid. Nucleophilic substitution at the Si^{IV} centre allows direct access to the corresponding sila-aldehyde and sila-ester. An alternative approach utilizing the reverse order of synthetic steps is thwarted by the facile rearrangement of the corresponding Si^{II} systems featuring H or OR substituents. As such, the isolation of (N-nacnac)Si(O)Cl represents a key step forward in enabling the synthesis of sila-carbonyl compounds via a synthetic approach ubiquitous in organic chemistry.

Acyl chlorides, RC(O)Cl, are versatile, reactive members of the family of carbonyl compounds, which have been shown to be of enormous synthetic value in organic chemistry. To a large part, this can be attributed to the lability of the chloride substituent in a range of chemical transformations, which allows for the synthesis, for example, of aldehydes, esters and amides, via simple functional group exchange at the carbonyl carbon.[1a-f]

By contrast with the ubiquitous nature of the carbonyl group, the chemistry of compounds containing Si=O double bonds is much less developed. A major factor underpinning the dearth of sila-carbonyl compounds is the fact that silicon and oxygen do not form strong multiple bonds due to poor 3p/2p π-overlap; in addition, the large difference in electronegativity (1.90 and 3.44, respectively, on the Pauling scale) imparts significant polarity to the Si—O bond ($Si^{\delta+}$—$O^{\delta-}$) which leads to a well-known propensity to oligomerize via Si—O—Si bridges.[2a-f] As such, compounds featuring formal Si=O double bonds are much less prevalent than singly-bonded polysiloxanes.[3] Nonetheless, very recent research has seen a surge in the number of structurally characterized Si=O bonds, thanks in part to the utilization of bulky, strongly-donating substituents which help to alleviate the electron-deficiency of the silicon centre, and prevent oligo-/ polymerization. Thus, a number of high profile examples of silanones ($R_2Si=O$) have been synthesized, typically by oxidation of the corresponding silylene (R_2Si:) (Figure 1).[4a-h]

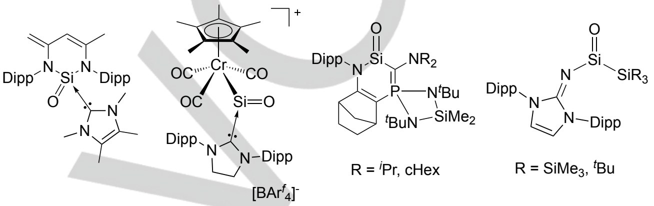

Figure 1. Prominent recent examples of silanones and related species synthesized by oxidation of the corresponding silylenes. (Dipp = $C_6H_3{}^iPr_2$-2,6, Ar^f = $C_6H_3(CF_3)_2$-3,5).

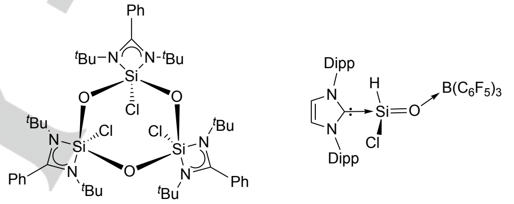

Figure 2. Previously reported examples of oligomeric or Lewis-acid-stabilized compounds containing the Si(O)Cl unit reported by Roesky and co-workers.[5c,6]

Despite this, analogues of other members of the carbonyl family (e.g. aldehydes, carboxylic acids and esters) remain little investigated, in part due to the paucity of suitable silylene precursors bearing H, OH or OR substituents.[4a,4e,4g,5a-e] Indeed, synthetic routes of any sort which can be applied to *systematically* access families of compounds of the types RSi(O)X and R(L)Si(O)X are few and far between. With analogies to classical carbonyl chemistry in mind, sila-acyl chlorides would offer considerable potential as synthetic intermediates, through which to widen our fundamental understanding of the chemistry of heavier Group 14 carbonyl analogues. That said, existing examples of systems containing the Si(O)Cl functional group are very rare – even as base-stabilized variants of the form R(L)Si(O)Cl. The high partial positive charge at silicon imparted by the presence of *two* electronegative substituents (O, Cl) leads to a tendency to oligomerize,[6] which can be prevented only by coordination of a 'capping' Lewis acid at oxygen (Figure 2).[5c]

We hypothesized that the deployment of an ancillary ligand framework offering both very high degrees of steric protection and electron donation would be necessary for the isolation of a synthetically valuable monomeric sila-acyl chloride. With this in mind, we utilized a new generation of electron-rich amino-functionalized β-diketiminate (or 'N-nacnac') ligands with the aim of reducing the polarity and accessibility of the Si=O bond.[7] Additionally, we reasoned that - unlike conventional β-methylated Nacnac ligands - the chlorosilylene precursor $(N\text{-nacnac})^{Dipp}SiCl$ (**1**, Scheme 1; $(N\text{-nacnac})^{Dipp}$ =

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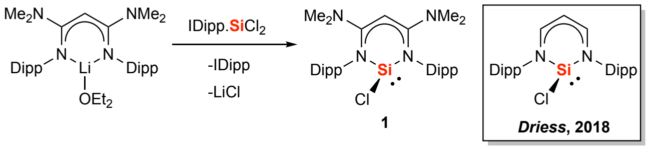
$HC\{(Me_2N)C(Dipp)N\}_2$), would be resistant to backbone deprotonation.[7,8] A similar approach was recently reported by Driess and co-workers utilizing a non-substituted β-diketiminate ligand $HC\{HC(Dipp)N\}_2$.[9a-e]

Accordingly, salt metathesis between (N-nacnac)^{Dipp}Li(OEt_2) and IDipp·$SiCl_2$ (Scheme 1; IDipp = *N*,*N'*-*bis*(diisopropylphenyl) imidazolylidene) generates the chlorosilylene precursor (N-nacnac)^{Dipp}SiCl (**1**) in 40-50% yield. **1** has been characterized by standard spectroscopic, analytical and crystallographic methods, with the latter (Figure 3) revealing only minor structural differences from $[HC\{HC(Dipp)N\}_2]SiCl$.[9e]



Scheme 1. Synthesis of the silylene precursor **1** via salt metathesis.

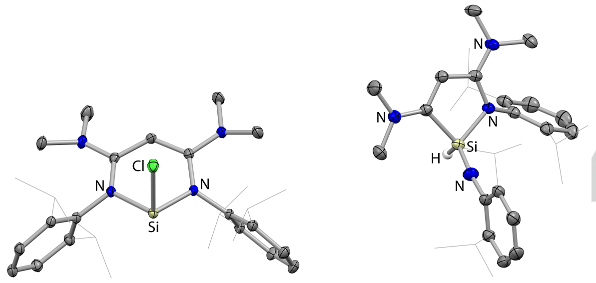
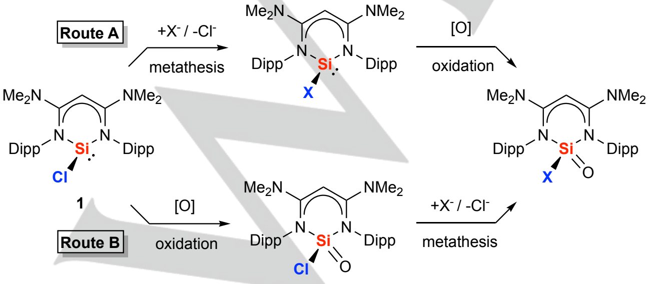


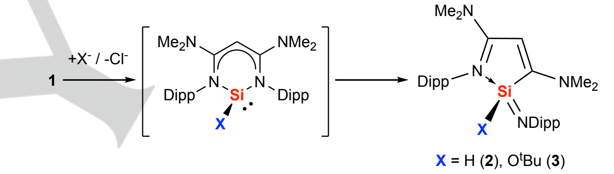
Figure 3. Molecular structures of **1** (left) and **2** (right) in the solid state as determined by X-ray crystallography. Thermal ellipsoids have been set at 45% probability, with Dipp-substituents shown in the wireframe format, and hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°): (**1**) Si—Cl 2.2533(8), Si—N 1.8461(10) and 1.8591(10), N—Si—N 96.82(5), Cl—Si—N 95.52(4) and 95.61(3); (**2**) Si—N(imine) 1.8183(12), Si—N(imide) 1.5765(14), N(imine)—Si—C 88.79(6), Si—N(imide) —C(ipso) 172.37(14).

With chlorosilylene precursor **1** in hand, we envisaged that a combination of oxidation and metathesis steps could be used to target sila-carbonyl systems, via either of two synthetic routes (Scheme 2) which differ only in the order of the two fundamental steps. As such, the conversion of **1** into the corresponding hydrido- and alkoxo-silylenes was explored via metathesis with sources of H^- or $^tBuO^-$ (Route A).



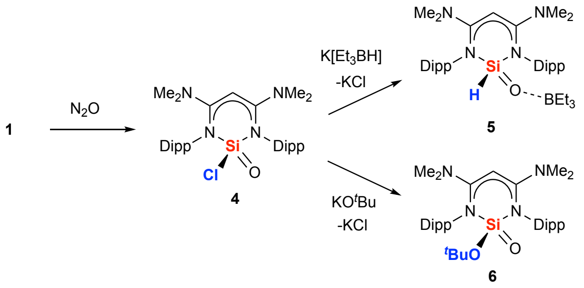
Scheme 2. Potential oxidation/metathesis routes to sila-carbonyl species

Rather than generate the straightforward substitution products, however, these reactions instead yield Si^{IV} products **2** and **3** derived from contraction of the N-nacnac skeleton backbone - via formal insertion of the silylene function into one of the C-N bonds (Scheme 3). With the exception of the SiH/SiO^{t}Bu resonances, **2** and **3** give rise to very similar patterns of signals in both ^1H and ^{13}C NMR spectra. In each case the pattern of six Dipp methyl resonances (in the intensity ratio 1:1:1:1:2:2) and three amino methyl signals (1:1:2), is consistent with a marked lowering of molecular symmetry compared to the N-nacnac-containing precursor **1**. Although we were not able to obtain an X-ray crystal structure of **3**, complex **2** could be crystallized from methylcyclohexane, and its structure confirmed crystallographically (Figure 3). This features a near-planar, five-membered NC_3Si heterocycle, and an almost-linear exocyclic Si-N-C fragment (172.37(14)°) containing a very short Si—N bond (1.5765(14) Å). The silicon-bound hydrogen atom could be located in the difference Fourier map and refined isotropically. As such, the molecular structure is consistent with a description as an imido Si^{IV} hydride featuring a monoanionic *N*,*C*-chelating azabutadiene ligand. This bonding motif is not without precedent – similar molecular scaffolds have been reported for both main group and transition metal complexes.[10]



Scheme 3. Ring contraction accompanying chloride-for-hydride and -butoxide substitution in **1**.

The propensity of these Si^{II} systems to undergo rearrangement (particularly in the presence of more electron-donating X groups) prompted us to consider the alternative synthetic route B outlined in Scheme 2. We envisaged that – if accessible – a β-diketiminate stabilized sila-acyl chloride would represent a valuable synthetic intermediate, allowing access to a range of sila-carbonyl species from a common reagent. Accordingly, compound **1** undergoes mild oxidation with N_2O at room temperature to afford the sila-acyl chloride **4** which can be shown crystallographically to be monomeric and to feature a unique Lewis-acid-free Si(O)Cl unit (Scheme 4 and Figure 4). Whatismore, treatment of **4** with either K[$HBEt_3$] or KO^{t}Bu demonstrates its efficacy in the synthesis of sila-carbonyl systems via Si-centred substitution chemistry. Thus, the (borane-stabilized) sila-aldehyde **5** and sila-ester **6** can be synthesized in moderate yields and their structures in the solid state confirmed crystallographically (Figure 4). While there are a small number of previous reports of Lewis-acid-stabilized sila-aldehydes,[5a-c] and of sila-esters/acids,[4a,4e,4g,5d,5e] acyl chloride analogue **4** offers systematic access, via nucleophilic substitution at silicon, to a range of fundamental sila-carbonyl species featuring a common supporting ligand platform.



Scheme 4. Simple chloride-for-hydride and -butoxide substitution utilizing sila-acyl chloride **4** in the synthesis of sila-aldehyde and sila-ester compounds.

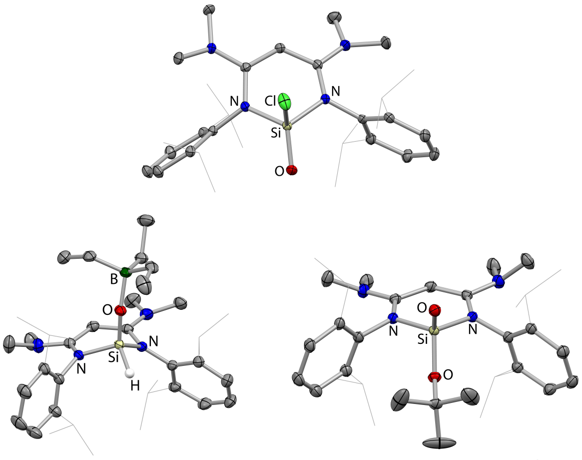


Figure 4. Molecular structures of **4** (top), **5** (bottom left) and **6** (bottom right) in the solid state as determined by X-ray crystallography. Thermal ellipsoids have been set at 45% probability, with Dipp-substituents shown in the wireframe format, and most hydrogen atoms omitted for clarity. Selected bond lengths (Å) and angles (°): (**4**) (molecule 1) Si—O 1.5329(14), Si—Cl 2.0891(6), Si—N 1.7906(16) and 1.7619(15), N—Si—N 103.61(7) / (molecule 2) Si—O 1.5280(14), Si—Cl 2.0976(6), Si—N 1.7626(15) and 1.7880(15), N—Si—N 103.41(7); (**5**) Si—O 1.5514(10), O—B 1.5639(19), Si—N 1.7815(12) and 1.7711(12), N—Si—N 101.44(6); (**6**) Si—O 1.5367(12), Si—O(^{t}Bu) 1.6431(11), Si—N 1.8028(13) and 1.8155(13), N—Si—N 100.39(6), O—Si—O 118.94(6).

DFT calculations were carried out (at the PBE1PBE/Def2-TZVP level) for a model system to probe the *mechanism* of substitution at the silicon centre (Figure 5 and ESI). These used MeO$^-$ rather than ^{t}BuO$^-$ as the incoming nucleophile and Ph substituents at N rather than Dipp for computational efficiency. These imply that the mechanistic pathway is associative, proceeding via a five-coordinate transition state and an activation barrier (55.4 kJ mol^{-1}) which is entirely consistent with a process that is facile at room temperature. The transition state occurs early in the substitution process and features relatively short Si-Cl and long Si-O(Me) bonds in line with the strongly exergonic nature of the overall reaction (>190 kJ mol^{-1}). The activation barrier calculated for an alternative S_N1-like process is found to be in excess of 400 kJ mol^{-1}.

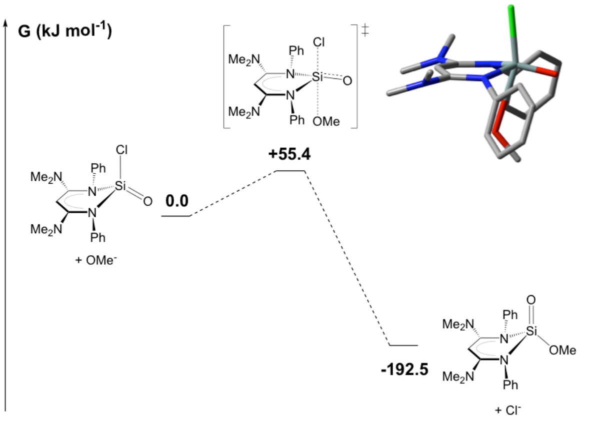


Figure 5. Free energy profile for substitution at the silicon centre of a model sila-acyl chloride complex, showing five-coordinate transition state.

From a spectroscopic perspective, the ^{29}Si NMR signals for **4**-**6** are found in a relatively narrow range (δ_{Si} = -60 to -80 ppm) and are shifted upfield compared to precursor **1** (δ_{Si} = 2 ppm), as expected for conversion of a silylene system to related Si=O -containing species (*cf.* 88 ppm for a nacnac-derived *N*,*N'*-diamidosilylene, and -85 ppm for the related silanoic ester).[4a,8] IR spectroscopy further confirms the presence of the sila-carbonyl functional group, with Si=O stretching bands observed in the range 1100 – 1200 cm^{-1}. The lower stretching frequency measured for **5** (1129 cm^{-1}) is consistent with the presence of the O-bound BEt$_3$ group, while the relative ordering of the bands for **4** and **6** (1157 and 1145 cm^{-1}) is consistent with the greater electron-withdrawing capabilities of the Cl vs O^{t}Bu substituent.[11] Crystallographically, the Si=O distances of **4** and **6** are towards the shorter end of the range defined by previously reported silanone species. Presumably the fact that the distances measured both species (1.528(1)/1.533(1) for **4**, 1.536(1) Å for **6**), are comparable to those reported for three-coordinate Si=O systems (e.g. 1.537(3) Å for (IDippN)Si(O)Si^{t}Bu$_3$))[4b,4g,4h] reflects the electron-withdrawing nature of Cl/O^{t}Bu and consequent contraction of the radius of silicon. The Si=O separation in **5** (1.551(1) Å) is marginally lengthened (*ca.* 2%), due to the O-bound borane function; the remarkably long B—O interaction on the other hand (1.564(2) Å) hints at a relatively unperturbed Si=O bond.[5a-c]

Quantum chemical probes of the nature of the Si=O bonds in these sila-carbonyl species yield Wiberg bond indices (WBI) for **4**-**6** which are significantly less than 2 (1.0993, 0.7955, and 1.0441 respectively), and frontier orbitals which imply little contribution from the silicon atom to the HOMO in each case (see ESI). Such findings are consistent with the dominant role played by the charge-separated resonance structure in base-stabilized silanones and related species.[12]

In conclusion, we have demonstrated the viability of a β-diketiminate sila-acyl chloride as a tractable monomeric system even in the absence of a stabilizing Lewis acid. Most importantly we can show that this compound is a useful synthetic intermediate, offering a facile route to silicon analogues of classical carbonyl compounds such as sila-aldehydes and esters.

This substitution methodology – while near ubiquitous in carbonyl chemistry – is thus shown to be compatible with the Si=O functional group.

Experimental Section

General experimental procedures, synthetic and characterizing data for compounds **1**-**3**, **5** and **6**, crystallographic and computational details are included in the ESI. CIF files for the five X-ray crystal structures have been deposited with the CCDC (ref: 1852264-1852268).

Synthesis of **4**: A toluene solution of **1** (100 mg, 0.18 mmol) in a J Young's ampoule was degassed (by freeze-pump-thaw) and then treated with excess N_2O at room temperature. The reaction mixture was stirred overnight, during which time the solution colour changed from yellow to off-white, with accompanying formation of a white precipitate. The solution was then filtered, and the precipitate washed with hot hexane. Drying the powder *in vacuo* afforded the spectroscopically pure product (66 mg, 43 % yield). Colourless X-ray quality crystals were grown from a concentrated toluene solution at -26°C. ^{1}H NMR (C_6D_6, 400 MHz): δ_H 1.12 (d, 6H, $(C\underline{H}_3)_2CH$-, $^3J_{HH}$ = 6.7 Hz), 1.16 (d, 6H, $(C\underline{H}_3)_2CH$-, $^3J_{HH}$ = 6.8 Hz), 1.53 (d, 6H, $(C\underline{H}_3)_2CH$-, $^3J_{HH}$ = 6.6 Hz), 1.58 (d, 6H, $(C\underline{H}_3)_2CH$-, $^3J_{HH}$ = 6.5 Hz), 2.12 (s, 12H, $(C\underline{H}_3)_2N$-), 3.46 (sept, 2H, $(CH_3)_2C\underline{H}$-, $^3J_{HH}$ = 6.7 Hz), 3.95 (s, 1H, methine H), 4.26 (sept, 2H, $(CH_3)_2C\underline{H}$-, $^3J_{HH}$ = 6.6 Hz), 7.06-7.16 (aromatic protons, 6H) ppm. ^{13}C{^{1}H} NMR (C_6D_6, 125 MHz): δ_C 24.1, 24.4, 26.9, 27.2 ($(\underline{C}H_3)_2CH$-), 28.8, 29.2 ($(CH_3)_2\underline{C}H$-), 41.0 ($(\underline{C}H_3)_2N$-), 78.0 (methine C), 125.1, 125.5, 137.9, 146.3, 147.3 (aromatic carbons), 164.8 (imine quaternary C) ppm. ^{29}Si{^{1}H} NMR (C_6D_6, 99 MHz): δ_{Si} -69.9 ppm. IR (Nujol, $\nu_{Si=O}$/cm^{-1}): 1157. Elemental analysis: calc. for $C_{31}H_{47}ClN_4OSi$: C 69.04 %, H 8.79 %, N 10.39 %; meas. C 68.94 %, H 8.62 %, N 10.26 %.

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Keywords: silanones • silylenes • β-diketiminate ligands • nucleophilic substitution • heavier carbonyl analogues

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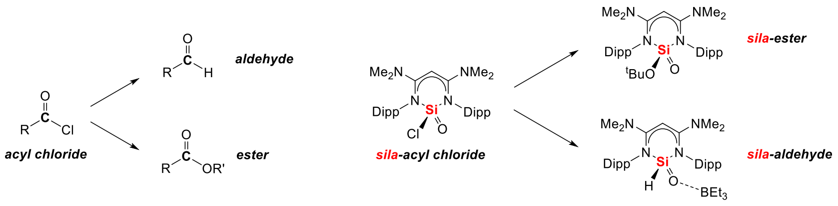
COMMUNICATION



NUCLEOPHILIC ACYL SUBSTITUTION

NUCLEOPHILIC SILA-ACYL SUBSTITUTION

More than just putting #sila in front of #acylchloride: The first simple silicon analogue of an acyl chloride can be accessed by mild oxidation of a N-nacnac stabilized chlorosilylene. This sila-acyl chloride undergoes facile functional group exchange, to yield the corresponding sila-ester and sila-aldehyde, in a manner analogous to that seen in classical carbonyl chemistry. These transformations demonstrate a significant breakthrough by applying seemingly ubiquitous organic reaction pathways to the heavier group 14 elements.

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