Tuning the Si−N Interaction in Metalated Oligosilanylsilatrane

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ABSTRACT: Most known silatrane chemistry is concerned with examples where the attached silatrane substituent atom is that of an element more electronegative than silicon. The current study features silylated silatrane with a range of electropositive elements attached to the silatrane substituent silicon atom. This directly affects the Si−N interaction of the silatrane which can be monitored through 29Si NMR spectroscopy or directly by single crystal XRD analysis of the Si−N distance. Within the sample of study the Si−N distance is increased from 2.153 to 3.13 Å. Moreover, the bis(trimethylsilyl)silatranyltrimethylsilyl unit was studied as a substituent for disilylated germatranes.

■ INTRODUCTION

Silatrane1−4 (Chart 1) and the related germatranes5 are hypercoordinated main group compounds. One of the defining properties of this class of compounds is that the heavy group 14 atom which is coordinated by a triethanolamine ligand is experiencing a transannular interaction with the nitrogen moiety of the ligand.6

The bonding interaction between the substituent Z occupying silicon’s remaining valence is typically strongly coupled to the relationship between Si and N. Longer Si−Z bond lengths (suggesting weaker interaction) usually result in shorter Si−N distances (suggesting stronger Si−N bonding interaction) and vice versa.7 X-ray diffraction studies of silatrane with comparably electronegative halogen, aryl, alkyl, and O substituents indicate a range of Si−N distances from 2.05 to 2.20 Å,2 which is significantly shorter than the sum of the van der Waals radii of silicon and nitrogen but slightly longer than the typical covalent Si−N single bond distance.3 Not much is known about silatrane with more electropositive substituents, and examples of metalated silatrane are restricted to a single platinum7 and a small number of osmium8,9 complexes.

Recent studies of silylated silatrane10−12 and germatranes13 have shown that these more electron-donating substituents increase the Si−N distance, and in cases when a silane unit is attached to the silatrane, they even turn off this interaction. In the current account we are outlining the influence of successively increasing electron-donating silyl groups on the Si−N interaction of the silatrane. To accomplish this, we decided to use substituted silanes with the attached elements covering Pauling’s electronegativity (EN) range $\chi_p$ from 0.82 (K) to 1.10 (Yb), 1.30 (Zr), 1.33 (Hf), 1.65 (Zn), 1.90 (Si), and 2.20 (H).14

■ RESULTS AND DISCUSSION

Silyl zinc compounds are a fairly established class of compounds. (Ph3Si)2Zn, as a first example of a disilylated zinc compound, was reported as early as 1963 by E. Wiberg et al.15 to form by reaction of Ph3SiK with ZnCl2 in liquid ammonia. Only in 1979 did Rösch and Altnau16 describe synthesis of (Me3Si)2Zn by reaction of Li[(Me3Si)4Al] with ZnCl2, which was then followed by Tilley et al.’s synthesis of [(Me3Si)3Si]2Zn from (Me3Si)3SiLi and ZnCl2 in 1987.17

Most of the following synthetic approaches utilized the simple salt metathesis concept. Thus, $[(\text{Me3Si})2\text{H})\text{Si}]\text{Zn}$, $[(\text{Me3Si})\text{SiZnCl}]2$, $[(\text{Me3HSi})\text{Si}]\text{Zn}$, a number of bis-(oligosilyl)zinc compounds,18 and trisilazincates19,20 were all obtained from reactions of the respective alkali silanides with ZnX2 (X = Cl, Br). A notable exception of this scheme was reported by Apeloig and co-workers who discovered the facile reaction of dialkylzinc reagents with silyl hydrides.21 Most of the described silyl zinc reagents were found to be fairly Lewis acidic, and frequently solvent molecules, bases, or even halide ions were found to coordinate to the zinc atoms. Reactions bis(trimethylsilyl)silatranyltrimethylsilyl 12,18 which is easily accessible from tris(trimethylsilyl)silatrane 1, with ZnBr2 gave disilylzing zinc compound 3 (Scheme 1). Given the mentioned susceptibility of organozinc compounds to coordinate Lewis bases, we expected at least some interaction between the silatranyl oxygen atoms and the central zinc atom.

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Single crystal XRD analysis of 3 (Figure 1) showed it to crystallize in the monoclinic space group $C2/c$. Despite the completely linear Si–Zn–Si (180°) arrangement which can be observed frequently in donor-free bis-silylated zinc compounds, a small Zn–Si–Si angle of 97.2° and an almost coplanar arrangement of the Zn–Si–Si–O substructure (dihedral angle: 7.6°) indicate at least some interaction between the closest silatrane oxygen atoms and the Zn-atom. The Zn–O distance of 2.877 Å is somewhat smaller than the sum of the van der Waals radii (2.91 Å).27

The silatranyl units engage in a trans conformation with respect to each other, and one of the ethylene units in both silatrane shows disorder. The Zn–Si bond length of 2.350(1) Å (Table 1) is comparable to that of $[(Me_3Si)_3Si]_2Zn$16 and does not show any elongation which otherwise can be observed when larger oligosilylated groups with higher steric demand are bonded to the zinc atom.22

Synthetic methods for preparing group 4 silyl complexes are quite similar to those for the formation of silyl zinc compounds. Therefore, it is not surprising that the protagonists in these fields are also largely the same. $Cp_2Ti(Cl)SiMe_3$ was obtained by Rösch and co-workers6 by reaction of $Na[(Me_3Si)_2Al]$ with $Cp_2TiCl_2$. Later, Tilley and co-workers prepared $Cp_2M(Cl)SiMe_3$ ($M = Zr$ and Hf)28 reacting $Cp_2MCi_3$ with $Al(SiMe_3)_3$ and $Cp_2M(Cl)Si(SiMe_3)_3$ ($M = Zr$ and Hf) utilizing $Me_3Si_2SiLi$ as nucleophile. A number of mono- and dioligosilylated group 4 metalloccenes were obtained employing various potassium oligosilanes.31–33 Therefore, not unexpectedly, reaction of silatranyl silane with $Cp_2MCi_3$ ($M = Zr$ and Hf) provided access to respective silylated metalloccenes 4 ($M = Zr$) and 5 ($M = Hf$) (Scheme 2). These two were found to be rather light-sensitive, and exposure to daylight over 2 days resulted in complete photolysis to give silatranyldihydrosilane 6 (Scheme 2). Formation of 6 is noteworthy since it was not possible to obtain it cleanly by the more obvious protonation reaction of silanide 2. The source of the proton in 6 is unclear as is the fate of the $Cp_2ZrCl_2$ fragment. Attempts to obtain an analogous silatranyl silyl titanocene were not undertaken as it is known that oligosilylated $Cp_2Ti(IV)$ compounds are not stable and undergo reductive elimination of silanes to $Cp_2Ti(II)$.34,35

Single crystal XRD analysis of hafnocene complex 5 revealed it to crystallize in the monoclinic space group $P2_11/c$ (Figure 2). The silatranyl unit engages in a trans conformation with respect to the chlorine atom. Interestingly, the number of crystallographically characterized silylated hafnocenes is rather small, and 5 constitutes the first example containing the $Cp_2Hf(Cl)Si$ substructure to be listed in the CCDC. However,
very comparable to those of Cp₂Zr(Cl)Si(SiMe₃)₃ and Cp₂Hf(Cl)Si(SiMe₃)₃.²⁹

Silatranylhydrosilane 6 was found to crystallize in the triclinic space group P.bar (Figure 3). Again, a disorder in one of the ethylene bridges of the silatranyl unit is observed. The position of the hydrogen atom at the silicon was located in the difference Fourier map.

Figure 2. Molecular structure of 5 (thermal ellipsoid plot drawn at the 30% probability level). All hydrogen atoms are omitted for clarity (bond lengths in Å, angles in deg). Hf(1)–C(17) 2.4141(15), Hf(1)–Si(1) 2.7774(14), O(1)–Si(17) 1.394(7), O(1)–Si(4) 1.662(4), Si(1)–Si(4) 2.361(2), Si(1)–Si(2) 2.367(2), Si(2)–C(12) 1.886(7), C(17)–C(18) 1.407(10), Cl(1)–Hf(1)–Si(1) 96.36(4), Si(4)–Si(1)–Si(2) 104.88(7), O(3)–Si(4)–O(1) 116.7(2).

Figure 3. Molecular structure of 6 (thermal ellipsoid plot drawn at the 30% probability level). All hydrogen atoms except H90 are omitted for clarity (bond lengths in Å, angles in deg). C(2)–N(1) 1.448(7), C(2)–C(1) 1.584(7), Si(1)–O(1) 1.674(3), Si(1)–N(1) 2.153(3), Si(1)–Si(2) 2.3568(16), Si(2)–Si(4) 2.3268(16), Si(4)–C(11) 1.880(4), O(1)–C(1) 1.429(4), O(1)–Si(1)–O(2) 120.43(15), O(1)–Si(1)–N(1) 83.07(13), N(1)–Si(1)–Si(2) 178.64(10).

The number of known silylated lanthanides is still small.³⁸ In order to follow reactions easily by NMR spectroscopy, the use of diamagnetic lanthanide ions is advisable. Ytterbium(II) complexes have proven useful in this respect. A few previously reported silylated Yb(II) complexes contained oligosilyl ligands.³⁹–⁴¹

Recently we reported the reaction of tris(trimethylsilyl)silyl potassium and YbI₂ to give a disilylated Yb-complex with three additional THF molecules coordinated to the Yb ion.⁴¹ Repeating this reaction with silatranyltrimethycarbosilane 2 led to clean product 7 with two silatranyl silyl ligands attached to Yb(II) in addition to two THF molecules (Scheme 3).

Scheme 3. Synthesis of Bis(silatranyl)silyl Ytterbium Complex 7

| Table 2. NMR Spectroscopic Data of Oligosilyl Silatranes (ppm) |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| **¹⁷⁷Si (SiMe₃)** | **²⁹Si (SiO₂)** | **²⁷Si (Si₃)** | other **²⁷Si shifts** | **¹³C (OCH₂/CH₂N)** |
| 1⁻ | -9.9 | -52.6 | -133.9 | 58.6/52.2²⁹ | 58.6/51.5²⁹ |
| 2⁻ | -3.2 | -11.8 | -210.5 | 61.0/54.³⁵²⁹ | 3.86/2.84²⁹ |
| 3⁻ | -7.2 | -48.1 | -133.8 | 59.1/51.5 | 3.41/1.93 |
| 4⁻ | -5.7 | -39.3 | -82.9 | 60.0/52 | 3.44/2.12 |
| 5⁻ | -4.5 | -35.9 | -79.0 | 60.1/52.4 | 3.42/2.12 |
| 6⁻ | -12.0 | -60.9 | -117.4 | 58.1/51.0 | 3.28/1.80 |
| 7⁻ | -2.5 | -25.9 | -177.0 | 61.0/52.2 | 3.69/2.40 |
| 8⁻ | -8.1/-8.4 | -43.7 | -125.7 | 59.5/51.5 | 3.37/2.10 |
| 9⁻ | -6.5 | -53.1 | -168.6 | -0.6 (GeSiMe₃) | n.d. |
| 10⁻ | -8.3/-8.5 | -41.9 | -130.6 | 60.5/53.1 | 3.38/1.93 |

“Measured in CDCl₃. “Measured in C₅D₅. “Measured in THF-d₆. “Data taken from ref 12.

Cp₂Hf(Me)SiBuPh³⁶ and three examples with two silyl ligands are known.

In two of these three examples, the Si–Hf–Si units are part of a cyclic system with Si–Hf bond lengths of 2.791²⁷ and 2.783³⁵ Å comparable to the 2.777(1) Å length observed for (SiMe₃)₃.³⁷ an even longer Si−C bond length is 2.829 Å.³⁷

Table 2. NMR Spectroscopic Data of Oligosilyl Silatranes (ppm)

The dihydro forms of silatranes have proven useful in this respect. A few previously reported silylated Yb(II) complexes contained oligosilyl ligands.³⁹–⁴¹

Recently we reported the reaction of tris(trimethylsilyl)silyl potassium and YbI₂ to give a disilylated Yb-complex with three additional THF molecules coordinated to the Yb ion.⁴¹ Repeating this reaction with silatranyltrimethycarbosilane 2 led to clean product 7 with two silatranyl silyl ligands attached to Yb(II) in addition to two THF molecules (Scheme 3).
Single crystal XRD analysis of 7 shows it to crystallize in the triclinic space group P1 (Figure 4). In addition to the expected Si–Yb interaction, coordination of one silatran oxygen atom of each ligand to Yb was also observed. The two thus-formed four-membered rings are almost planar with Si(6) 0.281 Å and Si(3) 0.444 Å out of planarity. Furthermore, the two rings are engaging an angle of 121.6° and thus provide space for the coordinated THF molecules. The Si–Yb (Table 1) and the O–Yb distances are in the expected ranges. The inter- and intramolecular Yb–O distances are very much alike.

The $^{29}$Si NMR spectrum of 7 (Table 2) shows signals at $\delta = -2.5$, $-25.9$, and $-177.0$ ppm for the SiMe$_3$, SiO$_2$, and SiYb silicon atoms. The peak at $-177.0$ ppm indicates a strong silanide character more pronounced than what we observed before for other ytterbium oligosilanyl complexes, which is also consistent with a silanide character more pronounced than what we observed for other ytterbium oligosilanyl complexes, which is also consistent with a silanide character more pronounced than what we observed before for other ytterbium oligosilanyl complexes.

Addition of the N-heterocyclic carbene IMe$_4$ to a solution of 8 in THF at room temperature led to immediate replacement of PMe$_3$ by the carbene and formation of gemelylene adduct 10 (Scheme 4). As was observed before for other NHCl-stabilized gemelylenes, only one signal for the trimethylsilyl groups at germanium was observed, which indicates magnetic equivalence of these groups and suggests that the silatranyl groups are located trans to each other.
Most silatrane chemistry is concerned with examples where the attached substituent atom is that of an element more electronegative than silicon. Electron withdrawal from silicon renders it more electrophilic and thus susceptible to interaction with the triethanol amine nitrogen atom. If the substituent atom is a metal or another electron rich group, then a higher degree of electron density is shifted toward the silatrane silicon atom and the interaction with the nitrogen atom is diminished. This was previously observed for silatranyl platinum and osmium complexes, with Si–N distances of 2.89(1) Å for the Pt complex and between 3.000(7) and 3.242(3) Å for the respective Os compounds.14–17 Comparable Si–N distances (3.10 to 3.18 Å) were observed recently for potassium silatranylsilanides such as 2.18 In addition to the Si–N distance, the 29Si NMR shift of the silatranyl silicon atom reflects the degree of hypercoordination with chemical shifts in a range between δ = −65 ppm (Z = Me) and −100 ppm (Z = F).5 These values are clearly upfield-shifted compared to those of related compounds without hypercoordination such as (MeO)2SiN(CH3)2 (δ = −38.4 ppm for X = Me) and −84.9 ppm for X = F.19 A similar upfield shift is observed when comparing (MeO)2Si(SiMe2)3 5 (δ = −32.2 ppm) to silatran 1 (δ = −52.6 ppm).20

Compounds presented in the current study can be considered to be variations of potassium silanide 2 with gradually diminished silane character. A Pauling electronegativity of 1.65 makes the Zn–Si bond of silatranyl zinc compound 3 relatively nonpolar. The associated 29Si NMR chemical shift of the SiO3 unit of δ = −48.1 ppm is therefore close to that of 1 (Table 2), and the respective Si–N distance of 2.265(4) Å is even smaller than that of 1 indicating that the local steric demand of the bis(trimethylsilyl)silatranylsilicarbosilane (trimethylsilyl)silyl unit is likely smaller than that of the tris(trimethylsilyl)silyl group.

For compounds 4 and 5, the electronegativity difference between silicon (EN: 1.9) and zirconium (EN: 1.33) and hafnium (EN: 1.3) is more pronounced going along with an enhanced silane character. This is nicely reflected by the 29Si NMR chemical shifts of the SiO3 units being −39.3 ppm (4) and −35.9 ppm (5) (Table 2). Along with these signs of diminished hypercoordination, the Si–N distance of 5 is elongated to 2.374(5) Å (Table 1). Compound 7 with the more electropositive Yb (EN: 1.1) continues this trend with a 29Si NMR chemical shift of the SiO3 units of −25.9 ppm and Si–N distances of 2.736(8) and 2.824(8) Å for the two crystallographically inequivalent silatranyl units of 7. Potassium silanide 2 is at the end point of the series with a Si–N distance of 3.134(4) Å and a SiO3 29Si NMR resonance of −11.8 ppm. The local geometry of the nitrogen atom in 2 is almost trigonal with even a slight pyramidalization toward the outside of the silatrane (exo-isomer). For compound 6, on the other side with hydrogen (EN: 2.2) attached to the bis(trimethylsilyl)silyl unit, a 29Si NMR shift of the silatranyl silicon atom of −60.9 ppm indicates enhanced hypercoordination compared to that of 1, which is also reflected by a diminished Si–N distance of 2.153(3) Å.

Introduction of the bis(trimethylsilyl)silatranyltrimethylsilyl unit as a substituent for a disilylated germylene adduct was accomplished by reaction of potassium silatranylsilanide 2 with GeCl3-dioxide in the presence of PMe3. While the resulting germylene PMe3 adduct 8 is reasonable stable, removal of the phosphane base caused rearrangement to disilagermine 9. Exchanging PMe3 as a base by an N-heterocyclic carbene caused formation of stable NH ligand PMe3 10.
toluene (2 mL). Red crystals of 4 (142 mg, 86%) were obtained after 3 days at −50 °C under light protection. Mp: 145–168 °C. NMR (δ in ppm): 1H: 6.24 (s, 10H, CP), 3.44 (t, J = 5.4 Hz, 6H, OCH3), 2.12 (t, J = 5.4 Hz, 6H, NCH3), 0.59 (s, 18H, MeSi), 13C: 111.40 (CP), 59.97 (OCH3), 52.32 (NCH3), 4.32 (MeSi), 55Si: −5.7 (MeSi), −39.3 (SiO3), −82.9 (SiL). Elemental analysis calculated for: C13H24ClN3O3Si5: C 38.14, H 5.82, N 2.02. Found: C 37.52, H 5.81, N 2.00.

1,2,3-Tetrakis(trimethylsilyl)1,2,3,5-tetrazathianaphthalene-1,3,5-tricarboxamide (9). Application of vacuum to solid 8 (45 mg, 0.05 mmol) for 90 min resulted in quantitative formation of 9 (41 mg, 100%). NMR (δ in ppm): 1H (CD3OD): 3.37 (t, J = 5.4 Hz, 12H, OCH3), 3.20 (t, J = 5.4 Hz, 12H, NCH3), 1.59 (d, JN = 10.5 Hz, 9H, Pch3), 0.64 (s, 36H, SiMe3). 13C (CD3OD): 59.5 (OCH3), 51.5 (NCH3), 18.1 (d, JCP = 22 Hz, PMe3), 3.8 (SiMe3), 3.8 (SiMe3). 29Si (CD3OD): −8.1 (d, JSP = 9 Hz, SiMe3), −8.4 (s, SiMe3), −43.7 (d, JSP = 16 Hz, SiO3), −125.7 (d, JSP = 15 Hz, Si(SiMe3)), 13P (CD3OD): −18.2 (PMe3).

Bis(trimethylsilyl)bis(trimethylsilylmethyl)amine (10). IMe2 (7 mg, 0.06 mmol) was added to a stirred solution of 8 (50 mg, 0.06 mmol) in THF (10 mL) at rt. After 1 h, the solvent was removed under reduced pressure and the residue extracted with pentane/toluene 1:1 (3 × 5 mL). Removal of the solvent revealed yellow-tan crystals of 10 (47 mg, 88%). Mp: 128–129 °C. NMR (δ in ppm): 1H (THF-d8): 3.97 (s, 3H, NCH3), 3.94 (s, 3H, NCH3), 3.49 (t, J = 5.4 Hz, 12H, OCH3), 2.66 (t, J = 5.4 Hz, 12H, NCH3), 2.17 (s, 3H, CCH3), 2.16 (s, 3H, CCH3), 0.23 (s, 18H, MeSi), 0.02 (s, 18H, SiMe3). 13C (THF-d8): 174.8 (NCN), 125.8 (CMe), 125.7 (CMe), 60.5 (OCH3), 53.1 (NCH3), 38.9 (NCH3), 35.4 (NCH3), 9.4 (CH3), 8.9 (CH1), 3.3 (SiMe3), 2.8 (SiMe3), 25Si (THF-d8): −8.3 (SiMe3), −8.5 (SiMe3), −41.9 (SiO3), −130.6 (Si(SiMe3)).
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