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Formation of the Imide $[Ta(NMe_2)_3(\mu\text{-}NSiMe_3)]_2$ through an Unprecedented α-$SiMe_3$ Abstraction by an Amide Ligand

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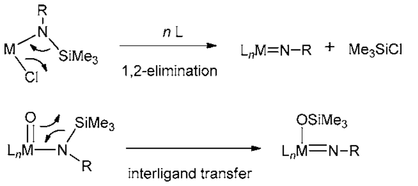
Ⓢ *Supporting Information*

ABSTRACT: $Ta(NMe_2)_4[N(SiMe_3)_2]$ (**1**) undergoes the elimination of $Me_3Si\text{-}NMe_2$ (**2**), converting the $-N(SiMe_3)_2$ ligand to the $=NSiMe_3$ ligand, to give the imide "$Ta(NMe_2)_3(=NSiMe_3)$" (**3**) observed as its dimer **4**. $CyN=C=NCy$ captures **3** to yield guanidinates $Ta(NMe_2)_{3-n}(=NSiMe_3)[CyNC(NMe_2)NCy]_n$ [n = 1 (**5**), 2 (**6**)]. The kinetic study of α-$SiMe_3$ abstraction in **1** gives $\Delta H^{\ddagger}$ = 21.3(1.0) kcal/mol and $\Delta S^{\ddagger}$ = −17(2) eu.

Transition-metal imide complexes have been actively studied[1] for their unique chemistry and applications as catalysts[2] and in the preparation of microelectronic materials.[3] A variety of methods have been developed to prepare imide ligands,[1,4,5] utilizing both intermolecular and intramolecular reactions. Intermolecular syntheses are more common, and imidation with primary amines, imines, nitriles, and other nitrogen-containing compounds is extensively used.[1,4] Intramolecular imidation is less common, and in these reactions, an imido ligand is usually formed through 1,2-elimination of Me_3SiCl or interligand transfer (Scheme 1). The treatment of $TiCl_3[N(SiMe_3)_2]$ with pyridine,[4d] for example, leads to 1,2-elimination of Me_3SiCl and the formation of $Me_3SiN=TiCl_2(py)_2$. In the reaction of $VOCl_3$ with 3 equiv of $Na[N(SiMe_3)_2]$, a $-SiMe_3$ group migrates to the oxo ligand, forming $Me_3SiN=V(OSiMe_3)[N(SiMe_3)_2]_2$.[4e] Thermolysis of $Ta(NR_2)_5$ (R_2 = Et_2, $Pr^n{}_2$, $Bu^n{}_2$, $MeBu^n$) yields imides $RN=Ta(NR_2)_3$ [R_2 = $MeBu^n$; $Bu^nN=Ta(NMeBu^n)_3$] as well as $Ta(NR_2)_4$, HNR_2, RH, and olefins.[5] The formation of the imides here is believed to involve d^1 $Ta(NR_2)_4$ and NR_2 radicals.[5]

Scheme 1. Two Main Types of Intramolecular Imidation[1c]



In the studies of the pentaamide $Ta(NMe_2)_4[N(SiMe_3)_2]$ (**1**), we found it to be unstable at room temperature. Upon heating at ≥70 °C, elimination of $Me_3Si\text{-}NMe_2$ (**2**) from **1** occurred to give the imide dimer $[Ta(NMe_2)_3(\mu\text{-}NSiMe_3)]_2$ (**4**; Figure 1). In order to see if "$Ta(NMe_2)_3(=NSiMe_3)$" (**3**) is an intermediate, the reaction of **1** with $CyN=C=NCy$ was studied and found to yield guanidinates $Ta(NMe_2)_{3-n}(=NSiMe_3)[CyNC(NMe_2)NCy]_n$ [n = 1 (**5**), 2 (**6**; Figure 1); Scheme 2]. The formation

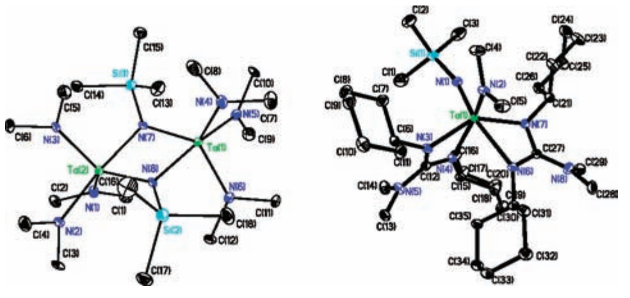
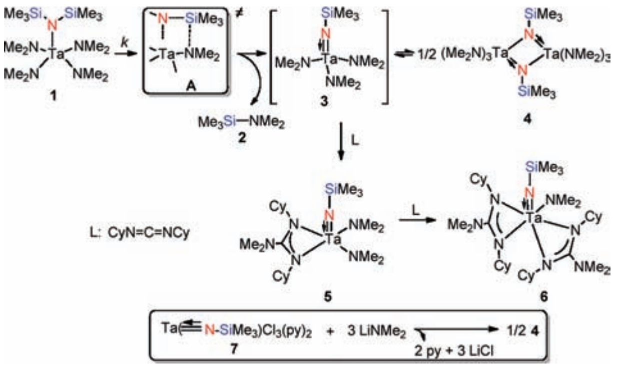


Figure 1. ORTEP views of **4** (left) and **6** (right). Selected bond lengths (Å) and angles (deg): **4**, Ta(1)−N(7) 1.964(4), Ta(1)−N(8) 2.140(4), Ta(2)−N(7) 2.135(4), Ta(2)−N(8) 1.951(4), Ta(1)−N(4) 1.966(5), Ta(1)−N(5) 2.037(4), Ta(1)−N(6) 2.018(4), Ta(2)−N(1) 2.011(4), Ta(2)−N(2) 2.036(4), Ta(2)−N(3) 1.980(4), N(7)−Ta(1)−N(8) 77.97(16), Ta(1)−N(7)−Ta(2) 101.05(17), N(8)−Ta(2)−N(7) 78.37(16), Ta(2)−N(8)−Ta(1) 101.32(17); **6**, Ta(1)−N(1) 1.814(3), Ta(1)−N(2) 2.008(3), N(1)−Ta(1)−N(2) 97.27(12), Si(1)−N(1)−Ta(1) 165.79(19).

Scheme 2. Formation of 4−6 from 1 and Preparation of 4 from 7[a]



[a]d−p π bonds in the amide ligands are not shown.

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of **5** and **6** suggests that CyN=C=NCy captures **3** in the reaction.[6] Although the abstraction of an α-$SiMe_3$ group by chloride and oxo ligands has been reported,[4d,e] the abstraction of an α-$SiMe_3$ group in an amide ligand by another amide ligand to yield an imide, to our knowledge, has not been reported.[1,4] Our results are reported here.

When a solution of **1** was heated at 85 °C, NMR spectra showed the formation of **2** and **4** in 30 min.[7] Their peaks grew in 2 h along with the appearance of unidentified peaks. After 24 h at 85 °C, NMR resonances of **1** and **4** had disappeared. **4** was also prepared from the reaction of $TaCl_3(=NSiMe_3)(py)_2$ (**7**)[4g] with $LiNMe_2$ (Scheme 2)[7] in order to characterize it and confirm its presence as a product in the decomposition of **1**. ^1H, ^{13}C, and ^{29}Si NMR spectra of **4** show peaks of $-NMe_2$ and $=NSiMe_3$ in the ranges reported for amide and imide ligands.[6b,8,9] In the solid state and in solution at 23 °C, **4** decomposes under a nitrogen atmosphere to unidentified products.

The structure of **4** from a single-crystal X-ray study revealed that it is a dimer bridged by two imide ligands.[7] In contrast, its analogue $Ta(NMe_2)_3(=NCMe_3)$[4f] is a monomer. The formation of **4** in the α-$SiMe_3$ abstraction reaction suggests that **3** is unstable, perhaps because it is coordinatively unsaturated. A Si atom is much larger than a C atom, and the $-SiMe_3$ group in **3** is further away from the Ta center, facilitating its dimerization. The imide bridges in **4** are asymmetrical, as is observed in $[Ta(\mu\text{-}NSiMe_3)(OCH_2Bu^t)_3]_2$.[9a] The Ta(1)−N(7) bond [1.966(5) Å], for example, is significantly shorter than the Ta(1)−N(8) bond [2.141(4) Å], suggesting that there is a double-bond feature between the Ta(1) and N(7) atoms. In other words, the lone pair of electrons on N(7) is involved in a dative d−p π bond with Ta(1). Hoffman and Suh reported the Ta(IV) dimer $\{Ta(\mu\text{-}NSiMe_3)[N(SiMe_3)_2]Cl\}_2$[10] with slightly asymmetrical Ta−N bond lengths: 1.994(7) and 1.985(6) Å. A Ta−Ta bond in this complex perhaps makes the average of the Ta−N bond length, 1.990 Å, smaller than that (2.048 Å) in **4**, which has no Ta−Ta bond. Heyduk and co-workers prepared the imide-bridged dimer $\{[ONO^{red}]Ta[\mu\text{-}N(p\text{-}tolyl)][NH_2(p\text{-}tolyl)]\}_2$ $\{[ONO^{red}]$ = *N*,*N*-bis(3,5-di-*tert*-butyl-2-phenoxide)amide},[11] which shows a slight asymmetry in the imide Ta^V−N bond lengths: 2.016(17) and 2.034(16) Å. The Ta(1)−N(7) bond in **4** [1.964(4) Å] is also much longer than the Ta=N bond [1.77(2) Å] in $Ta(NMe_2)_3(=NCMe_3)$.[4f] The dative d−p π bond in the latter makes the $Ta=NCMe_3$ bond essentially a triple bond. The Ta···Ta distance of 3.166 Å in **4** is much longer than a typical Ta−Ta bond length of, for example, 2.621(1) Å in $\{Ta(\mu\text{-}NSiMe_3)[N(SiMe_3)_2]Cl\}_2$,[10] suggesting that **4** is best described as a Ta^V complex with no metal−metal bond.

There have been few kinetic studies of the α-$SiMe_3$ abstraction or migration reactions.[1−5,12] The kinetics of the α-$SiMe_3$ abstraction in **1** was investigated. The disappearance of **1** follows first-order kinetics, as is shown in the $\ln(C/C_0)$ vs *t* plots (Figure 2, left), which yield the rate constants in Table 1. The Eyring plot (Figure 2, right) gives the activation parameters $\Delta H^‡ = 21.3(1.0)$ kcal/mol and $\Delta S^‡ = -17(2)$ eu. The "$SiMe_3^+$" group often behaves like a proton and can be readily cleaved.[12] Its steric effect is not large because the $SiMe_3$ group is relatively far from the rest of the molecule.[12c] The cleavage of a Si−C bond is usually faster than that of the corresponding H−C bond.[12] There have been kinetic studies of the α-H/D abstraction in, e.g., $Ta(CH_2SiMe_3)_5$ and $Ta(CD_2CMe_3)_5$ to give M=C bonds.[13,14] $Ta(CH_2EMe_3)_5$ (E = C, Si) and **1** are very different compounds. The hybridization of the α-C atoms in $Ta(CH_2EMe_3)_5$ and the α-N atoms in **1** is sp^3 and sp^2, respectively. It is thus not valid to directly compare the activation parameters in the α-H/D and α-$SiMe_3$ abstraction. It is, however, interesting to note that the $\Delta H^‡$ value here is similar to those in α-H abstraction.[13b,c,14] The negative $\Delta S^‡$ value is consistent with the concerted transition state **A** (Scheme 2), where bond rotations are restricted. The value is relatively large in magnitude, suggesting that the required ordering of ligands to reach **A** is significant. The $SiMe_3$ groups and $-NMe_2$ ligands in **1** are fairly far apart. In addition, only two $SiMe_3$ groups are available for the abstraction. In comparison, any of the eight α-H/D atoms are available for abstraction per alkyl ligand in $Ta(CH_2SiMe_3)_5$ and $Ta(CD_2CMe_3)_5$.[14] Thus, the α-H/D abstraction is statistically more favored, yielding smaller $\Delta S^‡$ values.[13c,14] The large, negative $\Delta S^‡$ in the current work leads to a high kinetic barrier in the formation of **3**.

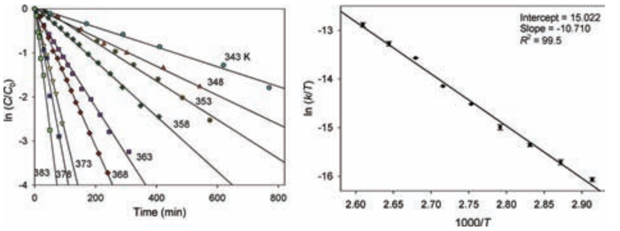


Figure 2. (Left) Kinetic plots of the α-$SiMe_3$ abstraction in **1**. (Right) Eyring plot for the reaction.

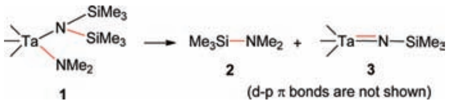
Table 1. Rate Constants for the α-$SiMe_3$ Abstraction in 1[a]

T (K)	$k \times 10^5$ (s^{-1})	*T* (K)	$k \times 10^5$ (s^{-1})
343.2(1.0)	3.60(14)	348.2(1.0)	5.2(3)
353.2(1.0)	7.6(2)	358.2(1.0)	11.0(6)
363.2(1.0)	17.99(4)	368.2(1.0)	25.4(9)
373.2(1.0)	47.69(12)	378.2(1.0)	64(3)
383.2(1.0)	97.0(4)		

[a] The total uncertainty $\delta k/k$ of 0.074 was calculated from $\delta k_{ran}/k$ = 0.055 and $\delta k_{sys}/k$ = 5%.

In the formation of **2** and **3** from thermolysis of **1**, a $Ta-NMe_2$ bond and a $N-SiMe_3$ bond are cleaved, and the π bond in $Ta=NSiMe_3$ and the Si−N bond in **2** are formed (Scheme 3).

Scheme 3. Bonds Cleaved and Formed in Thermolysis of 1



Bond energies in **1** and **4** are unknown.[15] Breaking of the Si−N bond in **1** is probably compensated for by the Si−N bond formed in **2**.[15a] Marks et al. found that, in $(Me_3SiCH_2)_3Ta=CHSiMe_3$, *D*(Ta=C) = 126(4) kcal/mol versus $D(Ta-C)_{av}$ = 67(1) kcal/mol.[16] If $D(Ta=N) \leq 2D(Ta-NMe_2)$ in **4**, the enthalpy change $\Delta H \geq 0$ is expected for thermolysis of **1**. Thermolysis is then likely entropically driven ($\Delta S > 0$) because decomposition of one molecule leads to the formation of two.

The formation of guanidinates **5** and **6** when **1** was heated in the presence of CyN=C=NCy supports the presence of **3** as an intermediate. The carbodiimide captures **3** by insertion into a $Ta-NMe_2$ bond, forming **5**, followed by another insertion to give **6**. When 2 equiv of CyN=C=NCy was added to a solution of **4**, **6** formed in a few minutes, suggesting that *perhaps* **4** is in equilibrium with monomer **3**, although **3** has not been directly observed (Scheme 2).

The X-ray crystal structure of **6** (Figure 1) shows that the Ta center is a distorted octahedron. The imide bond [Ta−N(1) = 1.814(3) Å] is slightly longer than those in typical complexes.[1g,9a,j] The Si(1)−N(1)−Ta(1) angle of 165.79(19)° is slightly bent, indicating a significant degree of d−p π bonding between the electron-deficient Ta atom and the lone pair of electrons on the imido N atom.[1g] Several two-dimensional NMR experiments have been conducted to help understand and decipher the complex one-dimensional ^{1}H and ^{13}C NMR spectra of **6**.[7]

The work here demonstrates that an amide ligand, such as chloride and oxo ligands, is capable of undergoing α-SiMe$_3$ abstraction to form a new metal−N π bond in the imide ligand.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Experimental details, NMR spectra, and crystallographic information in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

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Author Contributions

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(15) (a) Lappert, M. F.; Power, P. P.; Sanger, A. R.; Srivastava, R. C. *Metal and Metalloid Amides*; Wiley: New York, 1980; p 250. D(Si−N) = 79.1 kcal/mol in **2** and, e.g., 75.1−75.8 kcal/mol in (Me$_3$Si)$_2$NR (R = Me, H). (b) D(Ta−N)$_{av}$ = 78.4(1.2) kcal/mol in Ta(NMe$_2$)$_5$. Adedeji, F. A.; Cavell, K. J.; Cavell, S.; Connor, J. A.; Pilcher, G.; Skinner, H. A.; Zafarani-Moattar, M. T. *J. Chem. Soc., Faraday Trans. 1* **1979**, *75*, 603.

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