syringe. The flask was fitted with a condenser and refluxed under argon for 15 h. The reaction was monitored by thin-layer chromatography. After the solvent was evaporated, the product was purified directly on a silica gel column.

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The First Ce^{IV} Metallasilsesquioxane Complex: [Ce{(c-$C_6H_{11})_8Si_8O_{13}$}$_2$(py)$_3$]**

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Metallasiloxanes containing Si-O-M functional groups (M = main group metal, transition metal, or f-block element) have been envisaged as molecular analogues of zeolites due to their astonishing geometrical relationship.[1] In recent years, the chemistry of metallasilsesquioxanes has been extensively covered.[2] Metallasilsesquioxanes of rare earth metals might be important as homogeneous analogues of silica-supported rare earth metal catalysts and rare earth silicates, which are potential materials for optoelectronics. Hence, this area of research is of considerable interest for several fields of chemistry, including catalysis and materials science. However, investigations on metallasilsesquioxanes of rare earth metals are often hampered by difficulties with crystallization and characterization of these compounds. Here we report the synthesis and structural investigation of the first Ce^{IV} metallasilsesquioxane, a possible homogeneous model of Ce^{IV} silicate oxidation catalysts, and a new synthetic route, which may provide access to other novel metallasilsesquioxane complexes.

Treatment of [Ce{N(SiMe$_3$)$_2$}$_3$] with two equivalents of (c-$C_6H_{11})_8Si_8O_{11}(OH)_2$[3] in diethyl ether in the presence of an excess of pyridine exclusively afforded the diamagnetic complex [Ce{(c-$C_6H_{11})_8Si_8O_{13}$}$_2$(py)$_3$] (**1**). Compound **1** could also be prepared by direct reaction of anhydrous $CeCl_3$ with two equivalents of (c-$C_6H_{11})_8Si_8O_{11}(OH)_2$ in THF/pyridine, albeit in somewhat lower yield (Scheme 1). Surprisingly, in both cases cerium was oxidized to the tetravalent oxidation state. The compound gave satisfactory C,H,N analyses and was fully characterized by IR and ^{1}H, ^{13}C, and ^{29}Si NMR spectra, as well as X-ray single-crystal diffraction.

The IR spectrum (KBr) of **1** exhibited characteristic pyridine bands at 1599 and 750 cm^{-1}. ^{1}H NMR spectroscopy in [D$_6$]benzene gave a complex spectrum for a diamagnetic compound with broad multiplets between $\delta = 0.91$ and 2.12 for the methine and methylene protons of the c-C_6H_{11} substituents. The spectrum also showed three characteristic signals attributable to pyridine. In the ^{13}C NMR spectrum the

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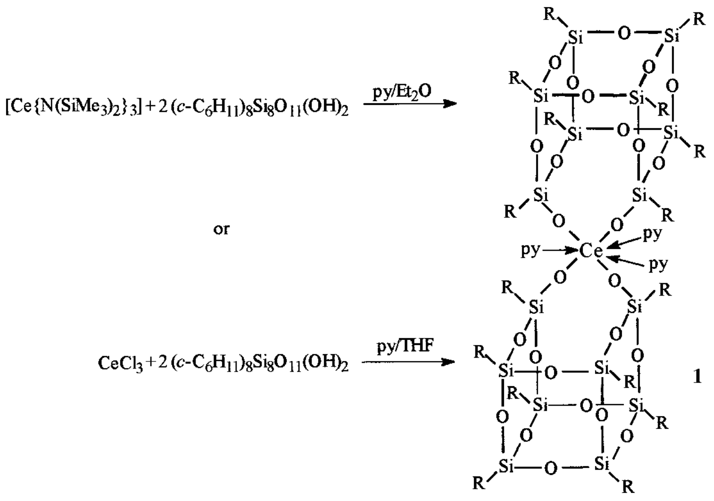

Scheme 1. Two alternative syntheses of **1**. R = c-C_6H_{11}.

methine signals are slightly farther upfield ($\delta = 24.04-25.96$) than the methylene signals ($\delta = 27.46-28.77$). The ^{29}Si NMR spectrum showed three signals at $\delta = -60.68$, -63.81, and -69.59 in a 2:4:2 ratio. The NMR signals of **1** were slightly shifted relative to the corresponding signals for the starting ligand, but they were in good accordance with the previously reported structure (from NMR data) of the free ligand.[3a]

X-ray quality crystals of **1** were grown from a concentrated solution in pyridine/diethyl ether at room temperature.[4] Compound **1** is the first metallasilsesquioxane derived from the recently discovered polyhedral $\{(c\text{-}C_6H_{11})_8Si_8O_{13}\}^{2-}$ ligand.[3] The central Ce atom is coordinated by four oxygen atoms of two siloxane ligands and by three nitrogen atoms of three pyridine molecules to give a distorted pentagonal-bipyramidal arrangement (Figure 1). The O(5) and O(5A) atoms are in axial and O(1), O(1A), N(1), N(1A), and N(2) are in equatorial positions. Some of the angles between two axial (161.5(2)°) and axial and equatorial ligands (89.56(12), 96.32(12), 99.26(9), 86.66(13), 77.98(13)°) differ significantly from the ideal angles of 180 and 90°, respectively, and the pentagonal bipyramid is very distorted. However, the angles between equatorial ligands (71.43(9), 75.12(14), 68.1(2), 142.9(2), 141.66(14) and 145.93(11)°) are quite close to those for an ideal pentagon (72 or 144°).

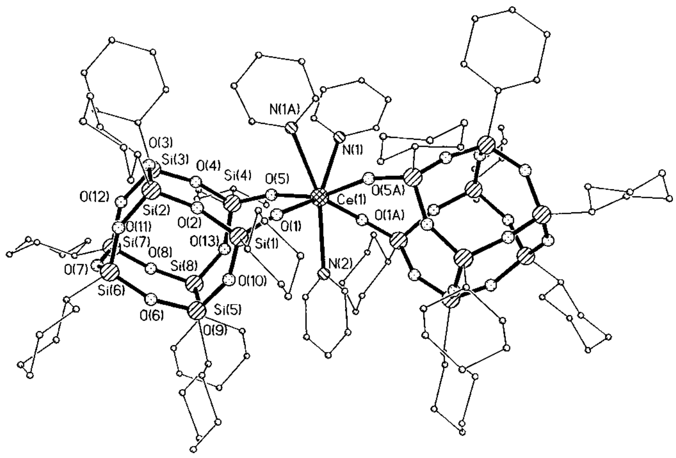

Figure 1. Structure of **1** in the crystal. Selected bond lengths [Å] and angles [°]: Ce(1)-O(1) 2.167(3), Ce(1)-O(5) 2.177(3), Ce(1)-N(2) 2.610(6), Ce(1)-N(1) 2.655(4); O(1)-Ce(1)-O(1A) 142.9(2), O(1)-Ce(1)-O(5) 89.56(12), O(1A)-Ce(1)-O(5) 96.32(12), O(5)-Ce(1)-O(5A) 161.5(2), O(1)-Ce(1)-N(2) 71.43(9), O(5)-Ce(1)-N(2) 99.26(9), O(1)-Ce(1)-N(1A) 75.12(14), O(1)-Ce(1)-N(1) 141.66(14), O(5)-Ce(1)-N(1) 77.98(13), O(5)-Ce(1)-N(1A) 86.66(13), N(1A)-Ce(1)-N(1) 68.1(2), N(2)-Ce(1)-N(1) 145.93(11).

No structurally characterized cerium(IV) siloxane derivatives are available for comparison. However, the Ce^{III} siloxane complex $[Ce(OSiPh_3)_3(thf)_3]\cdot thf$[5] and several Ce^{IV} alkoxide complexes have been previously reported.[6] As expected, the Ce–O bond lengths (2.167(3) and 2.177(3) Å) in **1** are substantially shorter than those to the $OSiPh_3$ ligands in the Ce^{III} complex (2.208(4), 2.224, and 2.234 Å)[5] but longer than those to the OtBu ligands (2.026(5) and 2.023(5) Å) in the Ce^{IV} complex $[Ce(OtBu)_2(NO_3)_2(HOCtBu)_2]$. The Ce–O distances are also comparable to those in $[Ce(OtBu)_2(\mu\text{-}OtBu)_2(\mu_3\text{-}OtBu)_2Na_2(dme)_2]$ for the terminal OtBu ligands (2.136(4) – 2.146(4) Å).[6] The Ce–N distances are equal (2.655(4) Å) for two of the pyridine ligands (N(1) and N(1A)), but shorter (2.610(6) Å) for the third (N(2)), presumably for steric reasons. The pyridine molecule that contains N(2) is sterically hindered by two adjacent c-C_6H_{11} groups. The Si–O distances range from 1.589(3) to 1.634(3) Å (av 1.615 Å), which is comparable to 1.603 Å for an idealized tetrahedral Si–O distance in the framework of silicates.[7]

The X-ray structure is not quite consistent with the NMR data considered above. According to the structure, Si(2) and Si(3) should not be chemically equivalent to Si(5) and Si(8), and consequently four (not three) ^{29}Si NMR signals and two (not one) different sets of resonances for pyridine ligands should be observed. Complex **1** is clearly not rigid in solution, and fast exchange between the fully and partially pyridine-solvated forms of the compound presumably occurs. The solvation – desolvation exchange process is too fast to be observed on the NMR timescale.

The most striking result is that despite starting from Ce^{III} precursors selective formation of a Ce^{IV} complex occurred in all cases. A similar phenomenon has been noticed before only in the formation of uranium(IV) – silsesquioxane complexes from different U^{IV} precursors.[2d] We propose that the pathway includes initial formation of an anionic Ce^{III} complex $(pyH)[Ce\{(c\text{-}C_6H_{11})_8Si_8O_{13}\}_2(py)_x]$. The steric and electronic properties of the silsesquioxane silanolate ligands render metal centers more Lewis acidic than conventional alkoxide or siloxide ligands do. Coordination of the OH groups to Ce facilitates the elimination of H^+ and the formation of the pyH^+ cation. A related anionic aluminosilsesquioxane is the recently reported compound $(HNEt_3)[\{(c\text{-}C_6H_{11})_7Si_7O_9(OSiMe_3)O_2\}_2Al]\cdot C_6H_{14}$.[8] Finally we note that the Ce^{III} intermediate is most likely oxidized by the skeletal oxygen atom of the silsesquioxane ligand to give the final product **1**. Oxidation by oxygen or a hydroperoxide, which might be present in THF or diethyl ether, is less plausible, because the solvents were stored over K/Na – benzophenone under argon and were freshly distilled shortly before the experiments.

The novel reaction pathway described here may provide access to other novel types of metallasilsesquioxane complexes in oxidation reactions.

Experimental Section

The ligand (c-$C_6H_{11})_8Si_8O_{11}(OH)_2$ was prepared according to a procedure previously described by Feher et al.[3] A solution of (c-$C_6H_{11})_8Si_8O_{11}(OH)_2$ (2.52g, 2.3 mmol) in diethyl ether (60 cm^3) was added to $[Ce\{N(SiMe_3)_2\}_3]$[9] (0.713 g, 1.15 mmol) at ca. 20°C. The mixture was stirred for 3 h. Pyridine (2.1 g, 23 mmol) was added, and the mixture was stirred for 24 h. The mixture was then filtered, and the filtrate was concentrated to ca. 20 cm^3 in vacuo. Pale yellow crystals of **1** were gradually deposited. Further concentration of the remaining mother liquor (ca. 10 cm^3) in vacuo and cooling to -22°C afforded an additional crop of crystals of **1** (total yield: 1.97 g, 67 %). Analysis calcd for $C_{114}H_{191}CeN_3O_{26}Si_{16}$: C 53.4, H 7.45, N 1.6; found: C 52.9, H 7.36, N 1.4; ^1H NMR (400 MHz, [D_6]benzene, 22°C): $\delta =$ 8.86 (m, 6H; py), 7.0 (m, 3H; py), 6.8 (m, 6H; py), 2.12–1.27 (brm, 160H; CH_2 in c-C_6H_{11}), 1.04 (m, 16H; CH in c-C_6H_{11}); ^{13}C NMR (125 MHz, [D_6]benzene, 22°C): $\delta = 150.45$, 136.28, 123.76 (py), 28.77, 28.36, 28.12, 27.92, 27.62, 27.46 (CH_2), 25.96, 25.16, 24.04 (CH); ^{29}Si NMR (99 MHz, [D_6]benzene, 22°C): $\delta = -60.68$, -63.81, -69.59 (2:4:2); IR (KBr): $\tilde{\nu} =$ 2923, 2850, 1599, 1448, 1108, 1067, 949, 847, 823, 750, 700, 622, 518, 403 cm^{-1}. Alternatively, complex **1** was prepared by heating anhydrous $CeCl_3$ (0.152 g, 0.615 mmol) for 24 h in THF under reflux and subsequent treatment with (c-$C_6H_{11})_8Si_8O_{11}(OH)_2$ (1.35 g, 1.23 mmol) in THF/pyridine (75 cm^3, 4/1). Crystallization from a concentrated solution in pyridine gave crystals of **1** (yield: 0.87g, 55 %).

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[4] Crystal data: for **1**: $C_{111}H_{195}CeN_3O_{28}Si_{16}$, $M_r = 1304.63$, monoclinic, space group $C2/c$, $a = 20.474(5)$, $b = 21.244(5)$, $c = 32.843(10)$ Å, $\beta = 102.95(3)^\circ$, $V = 13922(6)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.245$ g cm^{-3}, $F(000) = 5552$, $T = 153(2)$ K, $\mu(Mo_{K\alpha}) = 0.529$ mm^{-1}, $3.60 < \theta < 22.54^\circ$; of 13986 reflections 9079 were independent ($R_{int} = 0.0356$); the final R indices were $R_1 = 0.0511$ and $wR_2 = 0.1358$ ($I > 2\sigma(I)$), and $R_1 = 0.0596$, $wR_2 =$ 0.1471 (all data); GOF on F^2: 1.094; max./min. residual electron density $+1.211/-0.447$ e Å^{-3}. Data were collected on a Siemens-Stoe AED2 diffractometer. The structure was solved by direct methods (SHELXTS-90) and refined by full-matrix least-squares methods on F^2. All non-hydrogen atoms were refined anisotropically, and the hydrogen atoms were included by using a riding model. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-150263. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Differential Cleavage of Arylmethyl Ethers: Reactivity of 2,6-Dimethoxybenzyl Ethers[**]

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Protecting groups (PGs) serve a fundamental, albeit often under-appreciated, role in synthesis.[1] The interplay of their physico-chemical properties, availability, economics, and, most importantly, their selective manipulation significantly influences their ultimate utility. Consequently, milder or more selective methodology applicable to the existing repertoire of PGs and the development of new PGs with different reactivities have the potential to expedite progress along a broad front. Herein, we describe a convenient, high-yield protocol for the selective cleavage of arylmethyl ethers with stoichiometric chromium(II) chloride/lithium iodide in moisture-containing EtOAc [Eq. (1)].[2] We also highlight the 2,6-dimethoxybenzyl moiety[3] as a versatile and cost-effective[4] PG that extends the range of options for the selective removal of arylmethyl ethers.

$$ROCH_2Ar \xrightarrow[EtOAc\ (H_2O)]{CrCl_2/LiI} ROH + ICH_2Ar \qquad (1)$$

The scope of the de-*O*-benzylation by $CrCl_2$/LiI[5] was evaluated with a panel of representative arylmethyl ethers (Table 1). Unsubstituted benzyl ethers were smoothly cleaved at 75°C in moisture-containing[6] EtOAc to give, after aqueous isolation, the corresponding alcohol and benzyl iodide (e.g., the conversion of **1** into **2** (Table 1, entry 1)) in good to excellent yields. There was little or no reaction when we used $CrCl_2$ or LiI alone,[7] $CrCl_2$/LiBr, or $CrCl_2$/n-Bu_4NI.[8] On the other hand, CrI_2 was just as effective as $CrCl_2$/LiI, but its expense precluded further exploration. Yields were much lower in *N*,*N*-dimethylformamide, 1,2-dimethoxyethane, and acetonitrile; the use of THF was limited by its reaction with the reagent.

p-Phenylbenzyl ether[9] **3** likewise gave rise to **2** (Table 1, entry 2) in excellent yield under similar conditions as **1**. Notably, glycerol derivative **4** furnished secondary alcohol **5** (Table 1, entry 3) as the sole product. Such regioselectivity is reminiscent[10] of other transition metal promoted de-*O*-

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