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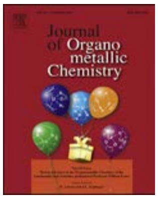

Communication

Cationic cerium(IV) complexes with multiple open coordination sites

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Dedicated to our friend and mentor, Prof. William J. Evans, on the occasion of his 70th birthday.

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ABSTRACT

Oxidation of *in situ* generated Ce^{III}(atrane) (atrane = $[N(CH_2C(CH_3)_2O)_3]^{3-}$) with iodine or $FcBAr^F$ afforded the novel cationic tetravalent cerium salts $[Ce^{IV}(atrane)(thf)_3][I_3]$ and $[Ce^{IV}(atrane)(thf)_3][BAr^F]$, both of which showed coordination by three THF molecules.

1. Introduction

The coordination chemistry of cerium has grown during the past few decades, and much of this effort has focused on understanding the redox properties of the cerium ion within different ligand frameworks [1–3]. While the trivalent oxidation state is prevalent in lanthanide (Ln) complexes [4], cerium is the only lanthanide with an accessible tetravalent oxidation state in its molecular chemistry, with $E^\circ = 1.74$ V versus NHE for the $Ce^{III/IV}$ redox couple [5]. The relative stability of the tetravalent oxidation state of the cerium ion is due to its stable noble gas configuration, $[Xe]4f^0$. Eller and Penneman rationalized that ligands with electronegative, hard donor atoms are best poised to stabilize high valent cerium [6]. Our group [2,3,7–9] and others [10–14] have isolated and characterized highly-stabilized tetravalent cerium complexes by using ligand frameworks with hard donor anions such as oxygen and nitrogen (Scheme 1).

While there has been a steady increase in the number of neutral tetravalent cerium complexes reported in recent years, there are limited reports of their cationic congeners. To date, only four examples of crystallographically characterized cationic Ce^{IV} complexes have been reported [3,15,16]. Notably, isolation of these compounds was made possible by the use of electron rich and sterically congested ligand frameworks such as H_3TRENDSAL ($N[CH_2CH_2N{=}CH(C_6H_2^tBu_2\text{-}3,5\text{-}OH\text{-}2]_3)$) [15], H_3TriNOx ($[((2\text{-}^tBu\text{-}NOH)C_6H_4CH_2)_3N]$) [3] and the Kläui ligand ($[CoCp\{P(O)(OEt)_2\}_3]^-$) [14] (Scheme 1). However, a drawback of using these sterically encumbered ligand frameworks is that they coordinatively saturate the metal center. It is of interest to expand the library of cationic lanthanide complexes to take advantage of their Lewis acidic characteristics in reactivity [17,18].

We recently reported on the coordination chemistry of a tripodal trianionic hydroxylamine ligand framework, $TriNOx^{3-}$ with cerium [3]. We also studied the redox properties of the Ce(III) ion within the TriNOx framework (Scheme 1, **II**). Our investigations showed that $TriNOx^{3-}$ provided significant stabilization of the 4+ oxidation state with an experimentally determined $E_{1/2} = -0.96$ V versus Fc/Fc^+. Chemical oxidations of a Ce^{III}(TriNOx) complex with ferrocenium salts, $FcBAr^F$ and FcOTf, yielded rare examples of structurally characterized cationic cerium complexes with one open coordination site. In a bid to isolate stable cationic tetravalent cerium complexes with multiple open coordination sites, we chose tris(2-hydroxyisobutyl)amine (H_3atrane), a derivative of triethanolamine (TEA), as a suitable ligand. The coordination chemistries of TEA and its derivatives with main-group elements, transition metals and the lanthanides have been studied [19–21]. We hypothesized that the three hard anionic oxygen donors on

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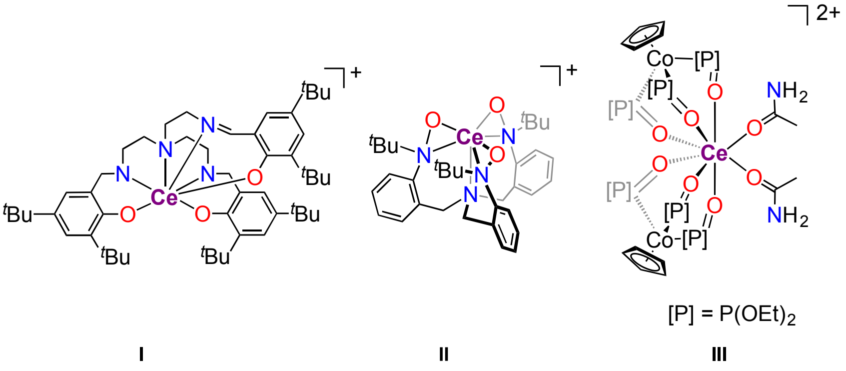


Scheme 1. Previously reported cationic cerium complexes.

atrane^{3-} would provide sufficient stabilization of the tetravalent oxidation state of the cerium ion comparable to that of TriNOx^{3-}. Also, due to the small steric profile of atrane^{3-}, the Ce^{IV} cation would be relatively open, thereby allowing for isolation of a coordinatively unsaturated metal center.

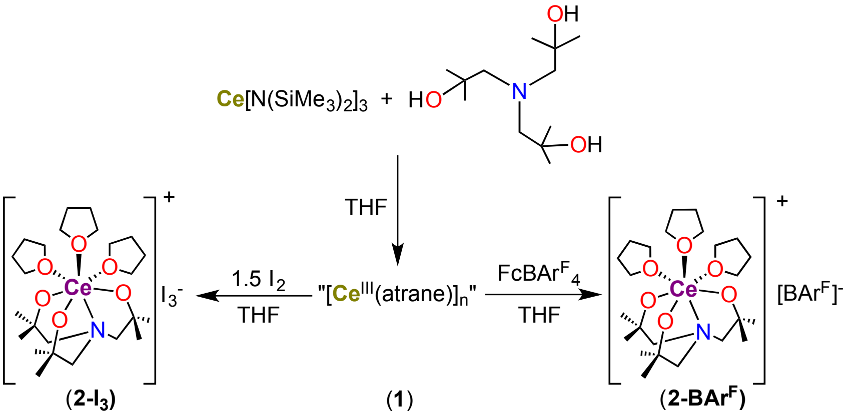
2. Results and discussion

H$_3$atrane was synthesized by a slight modification of a published report [21]. Isobutylene oxide and a 2 M ammonia solution in methanol were stirred at 50 °C for one week. The volatiles were removed under reduced pressure and the product was crystallized from a THF/diethyl ether mixture at −25 °C in 88% yield. Treating a THF solution of Ce[N(SiMe$_3$)$_2$]$_3$ with H$_3$atrane led to rapid formation of a yellow colored solution (Scheme 2). However, solution ^{1}H NMR characterization of the putative Ce^{III}(atrane) (**1**) indicated the presence of multiple species in solution, which complicated comprehensive solution characterization by ^{1}H NMR spectroscopy. Since we were unable to adequately characterize Ce^{III}(atrane), we hypothesized that *in-situ* oxidation to Ce^{IV} might allow for the formation of just one product, simplifying characterization. Treating the *in-situ* generated Ce^{III}(atrane) with 1.5 equivalents of I$_2$ led to the formation of a red colored solution. Removal of volatiles and subsequent crystallization of the red residue from a THF/hexanes mixture at −25 °C led to isolation of reddish-brown crystals of [Ce^{IV}(atrane)(thf)$_3$][I$_3$] (**2-I$_3$**) in 55% yield (Scheme 2, Fig. 1).

Complex **2-I$_3$** is a rare example of a structurally characterized cationic cerium complex and the first example of a tetravalent cerium complex with three open coordination sites. **2-I$_3$** crystallized with a C_3 symmetric monometallic structure, three capping THF molecules and one outer-sphere tri-iodide anion. The Ce-O$_{atrane}$ bond lengths in **2-I$_3$** of 2.0859(16) Å, 2.1034(16) Å and 2.1045(17) Å were consistent with a tetravalent cerium ion and the Ce-O$_{THF}$ bond lengths of 2.513(2) Å, 2.536(2) Å and 2.542(2) Å were shorter than that of 2.645(3) Å reported for the cationic cerium complex [Ce(TriNOx)thf][BAr^F] [3]. These results suggest that TriNOx^{3-} is more sterically encumbered compared to atrane^{3-}.

Isolation of **2-I$_3$** was notable not only because of the presence of multiple open coordination sites on the metal but also because of the identity of the non-coordinating tri-iodide anion. Evidently, the neutral THF molecules prevented the coordination of the negatively charged tri-iodide anion to the cerium center. Also notable was the persistence of the tetravalent oxidation state of cerium despite the proximity of a reducing tri-iodide anion. We attribute these observations to both the small steric profile of the atrane framework and its ability to effectively stabilize the Ce^{IV} ion.

With complex **2-I$_3$** in hand, we also sought to isolate an analogous cerium complex with a non-redox active, classical non-coordinating anion. This target would enable comparative solution, solid state and electrochemical studies with **2-I$_3$**. Treating *in situ* generated Ce^{III}(atrane) with 1 equiv. of FcBAr^F (Ar^F = 3,5-(CF$_3$)$_2$-C$_6$H$_3$) led to the formation of a yellow colored solution (Scheme 2). Removal of volatiles and subsequent crystallization of the yellow residue from a THF/hexanes mixture at −25 °C afforded isolation of yellow crystals of [Ce^{IV}(atrane)(thf)$_3$][BAr^F] (**2-BAr^F**) in 44% yield (See SI). The low yield of **2-BAr^F** was due to its high solubility in THF. Both complexes **2-BAr^F** and **2-I$_3$** were isostructural with similar Ce-N$_{atrane}$ bond distances. However, the Ce-O$_{atrane}$ bond lengths in **2-BAr^F** were slightly shorter than those in **2-I$_3$**.



Scheme 2. Synthesis of **1**, **2-I$_3$** and **2-BAr^F**.

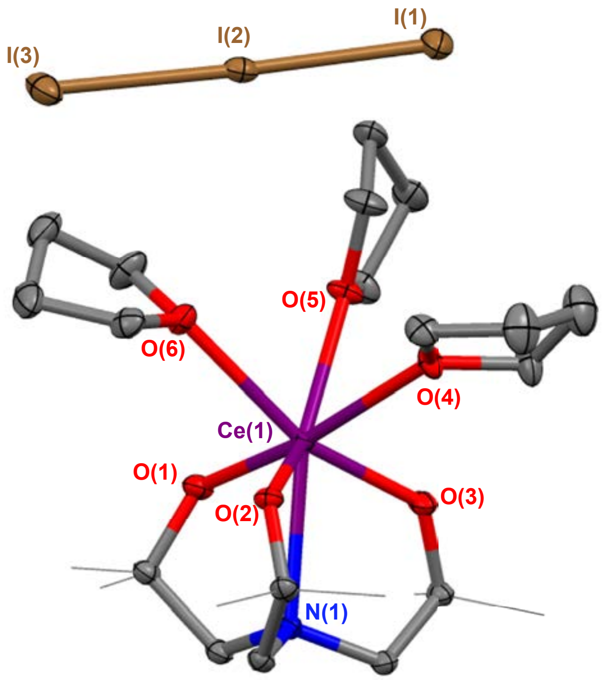


Fig. 1. 50% thermal ellipsoid plot of **2-I$_3$**. Hydrogen atoms are omitted for clarity. The methyl groups are represented with a wireframe model. Selected bond distances (Å): Ce(1)-O(1) 2.0859(16), Ce(1)-O(2) 2.1034(16), Ce(1)-O(3) 2.1045(17), Ce(1)-N(1) 2.6580(19), I(1)-I(2) 2.9224(3), I(2)-I(3) 2.9342(3).

^{1}H NMR spectroscopy analysis of both **2-I$_3$** and **2-BAr^F** in pyridine-d_5 displayed a singlet resonance at $\delta = 1.29$ ppm, for both complexes, which was assigned to the methyl protons of atrane. The singlet resonance at $\delta = 3.58$ ppm, for **2-I$_3$**, and $\delta = 3.58$ ppm, for **2-BAr^F**, was assigned to the methylene protons. This observation indicated a C_3 symmetric solution structure for both complexes. Also, the similarity between the ^{1}H NMR spectra of both complexes suggest that the tri-iodide anion in **2-I$_3$** was indeed outer-sphere with no direct interaction with the cerium cation in solution. Notably, the THF molecules in both complexes were displaced by the pyridine due to the stronger Lewis basicity of the latter.

In order to evaluate the stabilization of tetravalent Ce within the atrane framework, electrochemical analyses were carried out on **2-I$_3$**, **2-BAr^F** and independently synthesized tetrabutyl ammonium triiodide, **[TBA][I$_3$]** [22] (Fig. 2). Solution electrochemical experiments performed in THF on **2-I$_3$** revealed two reduction waves at $E_{pc} = -0.09$ V, $E_{pc} = -1.27$ V and two oxidation waves at $E_{pa} = -0.16$ V and $E_{pa} = 0.10$ V (Table 1) versus an internal ferrocene standard. The waves at $E_{pc} = -0.09$ V, $E_{pa} = -0.21$ V and $E_{pa} = 0.10$ V were assigned to the triiodide moiety based on comparison with independent electrochemical measurements performed on **[TBA][I$_3$]**. The broad irreversible wave at $E_{pc} = -1.27$ V was assigned to overlapping reduction waves for the triiodide and the metal based reduction. Only one irreversible metal based reduction (Table 1) at $E_{pc} = -1.17$ V versus ferrocene was observed upon performing electrochemical measurements on complex **2-BAr^F**. The measured metal-based reduction potential for **2-BAr^F** at $E_{pc} = -1.17$ V versus ferrocene was more negative than that measured for $Ce[N(SiMe_3)_2]_3$ ($E_{1/2} = +0.35$ V versus ferrocene in THF) [23] but compared favorably to that of [Ce(TriNOx)thf][BAr^F], which has an albeit quasi-reversible metal-based reduction wave at $E_{pc} = -1.04$ V versus ferrocene measured in DCM [3]. However, the Ce(IV) cation is more strongly stabilized in other environments such as, $Ce(O^tBu)_4(py)_2$ ($E_{pc} = -1.99$ V versus Fc/Fc^+ in DCM) [9] and $Ce^{IV}(2\text{-}(^tBuNO)py)_4$ ($E_{1/2} = -1.95$ V versus Fc/Fc^+ in DCM) [2].

In summary, we have synthesized the first examples of tetravalent cationic cerium complexes featuring three solvent-bound coordination sites supported by the atrane^{3-} ligand framework. Solution electrochemical experiments showed that the cerium(IV) cation was effectively stabilized by atrane as shown by the reduction potential of -1.17 V vs an internal ferrocene standard for **2-BAr^F**. Indeed, atrane prevented reduction of the cerium(IV) cation by the triiodide anion. We are currently exploring the reactivity of the isolated complexes with a view towards exploiting the exposed cerium ion for Lewis acid mediated reactivity.

Table 1
Redox potentials measured for complexes **[TBA][I$_3$]**, **2-I$_3$** and **2-BAr^F**.

	[TBA][I$_3$]	2-I$_3$	2-BAr^F
E_{pc}	−1.12	−1.27	−1.17
E_{pc}	−0.21	−0.09	–
E_{pa}	−0.31	−0.16	–
E_{pa}	0.06	0.10	–

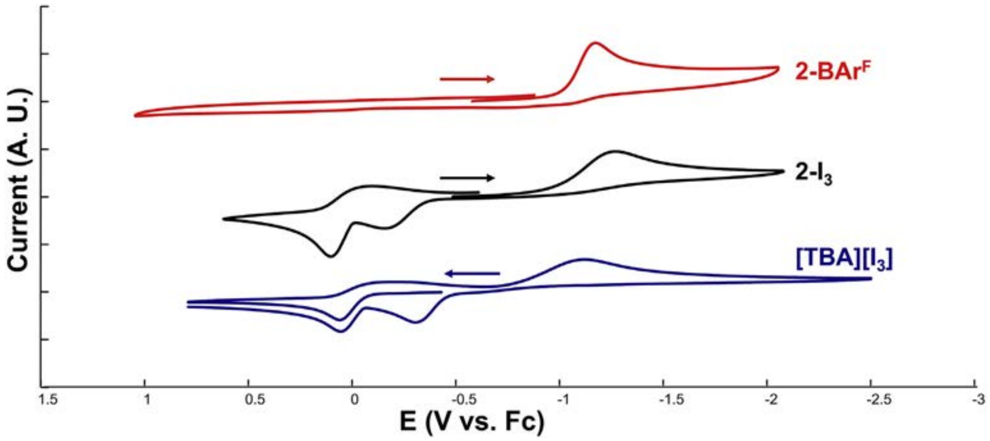


Fig. 2. Electrochemical measurements of [TBA][I$_3$] (dark blue), **2-I$_3$** (black) and **2-BAr^F** (red) performed in 0.1 M [^{n}Pr$_4$N][BAr^F]/THF. Potentials were measured versus an internal ferrocene standard. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3. Experimental

3.1. General methods

Unless otherwise noted, all reactions and manipulations were performed under an inert atmosphere (N_2) using standard Schlenk techniques or in a drybox equipped with a molecular sieves 13X / Q5 Cu–0226S catalyst purifier system. Glassware was oven dried for at least 3 h at 150 °C prior to use. ^1H and $^{19}F\{1H\}$ NMR spectra were obtained on a Bruker DMX-300 Fourier transform NMR spectrometer at 300 MHz and 282.2 MHz, respectively. $^{13}C\{^1H\}$ NMR spectra were obtained on a Bruker DRX-500 Fourier transform NMR spectrometer at 125.7 MHz. Chemical shifts were recorded in units of parts per million downfield from residual proteo solvent for ^1H NMR, characteristic solvent peaks for ^{13}C NMR, or relative to an external $CFCl_3$ reference (0 ppm). Elemental analyses were performed either at the Complete Analysis Laboratories, Inc. or using a Carlo Erba EA 1108 analyzer.

Materials. Tetrahydrofuran, toluene and hexanes were purchased from Fisher Scientific. All solvents were sparged for 20 min with dry N_2 and dried using a commercial two-column solvent-purification system comprising columns packed with Q5 reactant and neutral alumina respectively (for hexanes), or two columns of neutral alumina (for THF). Deuterated solvents were purchased from Cambridge Isotope Laboratories, Inc. and stored over 4 Å molecular sieves prior to use. Potassium bis(trimethylsilyl)amide (Sigma Aldrich), ferrocene (Acros), 2 M ammonia solution in methanol (Alfa Aesar), isobutylene oxide (TCI) and cerium (III) chloride (Alfa Aesar) were used as received. Iodine (Fisher Scientific) was sublimed before use. $[Bu_4N][I_3]$ [22], $FcBAr^F$ [24], $Ce[N(SiMe_3)_2]_3$ [25] and $[^nPr_4N][BAr^F]$ [26] were synthesized according to literature procedures.

Electrochemistry. All experiments were performed under an inert atmosphere (N_2) in a drybox with electrochemical cells that consisted of a 10 ml vial, glassy carbon disk (3 mm diameter) working electrode, a platinum wire counter electrode, and a silver wire plated with AgCl as a quasi-reference electrode. The working electrode surfaces were polished prior to each set of experiments. Potentials recorded in THF were referenced versus ferrocene, which was added as an internal standard for calibration at the end of each run. Solutions employed during CV studies were ~3 mM in analyte and 100 mM in $[^nPr_4N][BAr^F]$. All data were collected in a positive-feedback IR compensation mode. The THF solution cell resistances were measured prior to each run to ensure resistances ≤ ~500 Ω.

3.2. Synthetic procedures

3.2.1. $[Ce^{IV}(atrane)(thf)_2][I_3]$ ($2\text{-}I_3$)

A solution of H_3atrane (38.90 mg, 0.146 mmol, 1 equiv) in 6 ml THF was added to a vial containing $Ce[N(SiMe_3)_2]_3$ (100 mg, 0.146 mmol, 1 equiv). After 1 h of stirring the reaction mixture, I_2 (55.58 mg, 0.219 mmol, 1.5 equiv) was added. The reaction mixture was further stirred for 2 h. Volatiles were removed under reduced pressure. The red colored product was crystallized from a solution of THF layered with hexanes at −25 °C overnight. The mother liquor was decanted off and the crystals were dried under reduced pressure which led to the expulsion of one equivalent of THF. Yield: 57.8 mg, 0.065 mmol, 55%. ^1H NMR (300 MHz, pyridine-d_5): δ 1.29 (s, 18H, CH_3), δ 1.62 (m, 8H, CH_2), δ 3.58 (s, 6H, CH_2), δ 3.66 (m, 8H, CH_2). $^{13}C\{^1H\}$-NMR (125.7 MHz, pyridine-d_5): δ 26.0 (s, 4C, $C^{1,4}$-H), δ 32.6 (s, 6C, CH_3), δ 68.7 (s, 4C, $C^{2,3}$-H), δ 76.4 (s, 3C, $N(CH_2)$), δ 87.0 (s, 3C, $O(C(CH_2))$). Elemental analysis found (calculated) for $C_{20}H_{40}O_5NI_3Ce$: C, 26.69 (26.83); H, 4.38 (4.50); N, 1.40 (1.56).

3.2.2. $[Ce^{IV}(atrane)(thf)_2][BAr^F]$ ($2\text{-}BAr^F$)

A solution of H_3atrane (112.7 mg, 0.483 mmol) in 6 ml THF was added to a vial containing $Ce[N(SiMe_3)_2]_3$ (300 mg, 0.483 mmol). After 1 h of stirring the reaction mixture, $FcBAr^F$ (461.1 mg, 0.483 mmol) dissolved in 4 ml THF was then added. The reaction mixture was further stirred for 24 h. Volatiles were removed under reduced pressure. The product was washed with pentane, dried and then crystallized from a solution of THF layered with hexanes at −25 °C overnight. The mother liquor was decanted off and the crystals were dried under reduced pressure which led to the expulsion of one equivalent of THF. Yield: 57.8 mg, 0.065 mmol, 44%. ^1H NMR (300 MHz, pyridine-d_5): δ 1.29 (s, 18H, CH_3), δ 1.62 (m, 8H, CH_2), δ 3.58 (s, 6H, CH_2), δ 3.66 (m, 8H, CH_2). $^{13}C\{^1H\}$-NMR (125.7 MHz, pyridine-d_5): δ 26.1 (s, 4C, $C^{1,4}$-H), δ 31.9 (s, 6C, CH_3), δ 68.1 (s, 4C, $C^{2,3}$-H), δ 75.9 (s, 3C, $N(CH_2)$), δ 86.5 (s, 3C, $O(C(CH_2))$), δ 118.2 (sept, 4C, C^4-H), δ 125.5 (q, 8C, $C^{3,5}$-CF_3), δ 129.9 (qq, 8C, $C^{3,5}$-CF_3), δ 135.6 (s, 8C, $C^{2,6}$-H), δ 162.9 (q, 4C, C^1-B) ^{19}F NMR (282.2 MHz, pyridine-d_5): δ −62.1 (s, 24F). Elemental analysis found (calculated) for $C_{57}H_{63}O_6F_{23}NBCe$: C, 46.03 (46.39); H, 3.99 (4.17); N, 0.99 (0.97).

3.3. X-ray crystallography

X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at a temperature of 143(1) K. In all cases, rotation frames were integrated using SAINT [27], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values, which were then passed to the SHELXTL [28] program package for further processing and structure solution. The intensity data were corrected for Lorentz and polarization effects and for absorption using TWINABS [29] or SADABS [30]. The structures were solved by direct methods (SHELXS-97) [31]. Refinement was done by full-matrix least squares based on F^2 using SHELXL-97 [31]. All reflections were used during refinements. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model.

Notes

The authors declare no competing financial interest.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.jorganchem.2017.10.017.

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