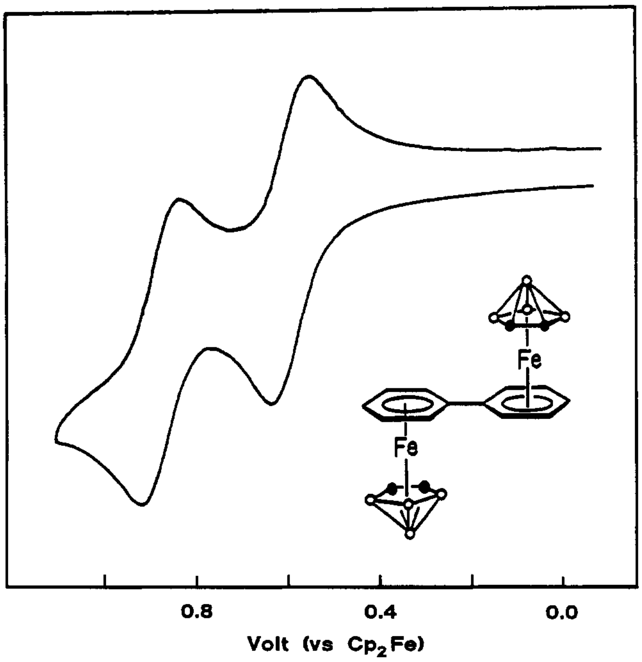

Figure 2. Cyclic voltammogram of biphenyl diiron complex 3 in CH_2Cl_2/0.1 M Bu_4NPF_6 at Pt electrode. Conditions: ambient temperature; scan rate = 200 mV/s; concentration = 1.0 mM. Full scale on the *y* axis is 13 μA.

feasible, substitution of a cyclopentadienyl ligand by a carboranyl ligand should be considered when stabilization of a Fe(III) or Ru(III) complex is desired.

The reduction of dinuclear iron(II) arene complexes, linked either through the arenes[13] or through cyclopentadienyl units,[14] or in cyclophane complexes,[15] has received attention owing to questions concerning mixed valency and electronic delocalization in the formal Fe(II)/Fe(I) species. However, lack of a suitable oxidation process has precluded studies of mixed valency for Fe(II)/Fe(III) systems with arene linkages.[3] The use of the carborane capping ligand in place of Cp^- allows oxidation of dinuclear iron complexes to be observed. Therefore, the biphenyl complex 3 undergoes two reversible one-electron oxidations (Figure 2) at $E°$ = +0.55 and +0.84 V vs $Cp_2Fe^{0/+}$. The separation of the successive $E°$ values (290 mV) is slightly less than those reported for the successive oxidations of biferrocene (330–350 mV).[16,17] A study of mixed valency in the cations derived from 3 and other diiron systems is underway.

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The Utility of Ceric Ammonium Nitrate Derived Alkoxide Complexes in the Synthesis of Organometallic Cerium(IV) Complexes: Synthesis and First X-ray Crystallographic Determination of a Tetravalent Cerium Cyclopentadienide Complex, $(C_5H_5)_3Ce(OCMe_3)$

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Summary: $Ce(OCMe_3)_2(NO_3)_2(THF)_2$ reacts with 2 equiv of NaC_5H_5 to form $(C_5H_5)_2Ce(OCMe_3)_2$ in 90% yield. The reaction of $Ce(OCMe_3)(NO_3)_3(THF)_2$ with NaC_5H_5 forms a mixture of $(C_5H_5)_2Ce(OCMe_3)_2$ and $(C_5H_5)_3Ce(OCMe_3)$. The latter product can be isolated by recrystallization from hexane at −34 °C. It crystallizes in space group *Pnma* with unit cell parameters at 183 K of a = 14.8351 (17) Å, b = 13.3467 (14) Å, c = 8.9677 (9) Å, V = 1775.6 (3) Å^3, and D_{calcd} = 1.53 g cm^{-3} for Z = 4. Least-squares refinement of the model based on 1661 reflections converged to a final R_F of 5.1%. The three C_5H_5 ring centroids and the alkoxide oxygen atom form a distorted tetrahedron around the Ce(IV) center with a 2.76 (2) Å average Ce–C distance and a 2.045 (6) Å Ce–O length.

A fundamental question that has persisted for many years in the organolanthanide area is whether or not organometallic complexes of tetravalent cerium can exist.[1-4] There is good reason to question the existence of organometallic Ce(IV) complexes, since Ce(IV) is strongly oxidizing and the anionic ligands typically found in organolanthanide complexes (e.g., $C_5H_5^-$, $C_8H_8^{2-}$, and R^-) are strongly reducing. Skepticism increased when some early syntheses[5-12] of organometallic cerium(IV) complexes were found to be irreproducible or to form cerium(III) complexes.[13-17] In 1985, the synthesis of $Ce(C_8H_8)_2$[15] was

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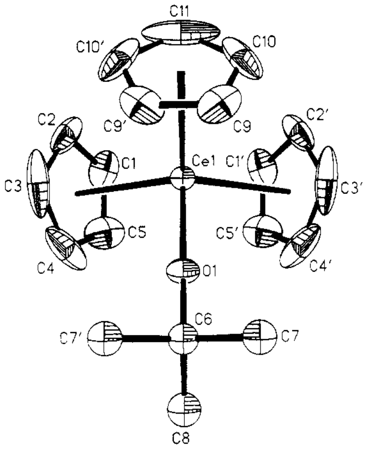

Figure 1. ORTEP plot of $(C_5H_5)_3Ce(OCMe_3)$ with thermal ellipsoids at 30% probability level and disordered atoms of the CMe_3 group omitted for clarity.

reproduced and the physical properties of this complex were carefully examined.[16] Recently, an improved synthesis of $(C_5H_5)_3Ce(O\text{-}i\text{-}Pr)$[15] has been reported and its physical properties established.[18] Despite these recent advances in organocerium(IV) chemistry, X-ray crystallographic data on a Ce(IV) organometallic complex have proven elusive.[19] We report here the first complete X-ray crystal strucure study of such a complex as well as convenient syntheses of $(C_5H_5)_2Ce(OCMe_3)_2$ and $(C_5H_5)_3Ce(OCMe_3)$ which are based on the readily available precursor, ceric ammonium nitrate.

The recent synthesis[18] of $Cp_3Ce(O\text{-}i\text{-}Pr)$ from $Ce(O\text{-}i\text{-}Pr)_4$ and Me_3SnCp in 69% yield (24 h, 65 °C) is a great improvement over the 4% yield of the initial synthesis.[15] However, although the preparation of the Me_3SnCp starting material from Me_3SnCl and CpLi[20] was straightforward, the preparation of $Ce(O\text{-}i\text{-}Pr)_4$ involved the tedious[21] synthesis of $(C_5H_6N)_2CeCl_6$.[22] We have found that mixed-ligand Ce(IV) *tert*-butoxide nitrate complexes $Ce(OCMe_3)_2(NO_3)_2(THF)_2$ (**1**)[23] and $Ce(OCMe_3)(NO_3)_3(THF)_2$ (**2**)[23] are good precursors to Ce(IV) cyclopentadienide complexes. **1** and **2** can be prepared in 50–80% yield by a 2-h reaction of commercially available ceric ammonium nitrate with $NaOCMe_3$ in THF.[23] **1** and **2** react rapidly with NaC_5H_5 in THF at room temperature to form Ce(IV) cyclopentadienyl *tert*-butoxide complexes as shown in reactions 1 and 2 (R = CMe_3). Complex **3** is isolated from reaction 1 in 90% yield as a dark brown powder.[24] Its diamagnetic NMR spectra are similar to those of crystallographically characterized **4**[25] (see below) and those of $(C_5H_5)_3Ce(O\text{-}i\text{-}Pr)$.[15] Reaction 2 is more complicated and gives a mixture of **3** and **4** as well as other cerium-containing byproducts. Complex **4** can be separated from **3** by crystallization and can be isolated in 50% yield as a black microcrystalline powder.[25] Recrystallization from hexane at −34 °C gave X-ray quality crystals[26] whose structure is shown in Figure 1.

$$\underset{\mathbf{1}}{Ce(OR)_2(NO_3)_2(THF)_2} + 2NaCp \rightarrow \underset{\mathbf{3}}{Cp_2Ce(OR)_2} + 2NaNO_3 \quad (1)$$

$$\underset{\mathbf{2}}{2Ce(OR)(NO_3)_3(THF)_2} + 6NaCp \rightarrow \underset{\mathbf{3}}{Cp_2Ce(OR)_2} + \underset{\mathbf{4}}{Cp_3Ce(OR)} + 6\,NaNO_3 + \text{other products} \quad (2)$$

Since reaction 2 forms a mixture of **3** and **4** within a very short reaction time, rapid ligand redistribution reactions must be occurring in this system. Since **3** and **4** do not readily undergo ligand redistribution, the ligand mixing must involve intermediates in the reaction. For example, NaCp may replace a nitrate ligand in **2** to form $CpCe(OR)(NO_3)_2$ which disproportionates to $CpCe(NO_3)_3$ and $CpCe(OR)_2(NO_3)$. The latter compound could react with NaCp to form **3**.[27] Without the stabilizing influence of the alkoxide ligand,[28] $CpCe(NO_3)_3$ may decompose to dark insoluble Ce(III) products as observed in the reactions of NaCp with $(NH_4)_2Ce(NO_3)_6$.[29] Several other ligand redistribution reactions of this type can be envisioned.

The overall structure of $Cp_3Ce(OCMe_3)$ is similar to that of Cp_3UZ complexes (Z = halide, hydrocarbyl)[1,30–36] in that the three Cp ring centroids (Cn) and the alkoxide oxygen

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(24) Typically, in a nitrogen-filled glovebox a solution of $Ce(OCMe_3)_2(NO_3)_2(THF)_2$ (50 mg, 0.090 mmol) in 2.5 mL of THF was added to a suspension of NaC_5H_5 (15.9 mg, 0.18 mmol) in 2.5 mL of THF resulting in the immediate formation of a dark red-brown mixture. After being stirred for 30 s, the mixture was centrifuged to remove a white precipitate (15 mg, 97% if $NaNO_3$). The THF solvent was removed by rotary evaporation, and the product was extracted with hexane. Subsequent solvent removal left $Cp_2Ce(OCMe_3)_2$ as a dark brown powder (38 mg, 90%). ^1H NMR (C_6D_6): δ 6.14 (s, 10 H, C_5H_5), 1.26 (s, 18 H, $OCMe_3$). ^{13}C NMR (C_6D_6): δ 120.22 (C_5H_5), 82.55 ($OCMe_3$), 32.50 ($OC\mathit{Me}_3$). IR (KBr): 2970 m, 1360 s, 1230 m, 1090 s, 1020 m, 980 s, 960 s, 770 s cm^{-1}. Anal. Calcd: Ce, 33.6. Found: Ce, 33.7.

(25) Using a procedure like that in footnote 24, $Ce(OCMe_3)(NO_3)_3(THF)_2$ (100 mg, 0.184 mmol) was reacted with NaC_5H_5 (48.6 mg, 0.552 mmol) to form a tan-brown, THF-insoluble material (71 mg) and a black, hexane-soluble mixture of **3** and **4**. The less soluble **4** was obtained as a black microcrystalline powder (42 mg, 50%) by recrystallization from a concentrated hexane solution cooled to −34 °C. Single crystals suitable for X-ray crystallography were grown from hexane at −34 °C. ^1H NMR (C_6D_6): δ 5.60 (s, 15 H, C_5H_5), 1.58 (s, 9 H, $OCMe_3$). ^{13}C NMR (C_6D_6): δ 121.24 (C_5H_5), 84.31 ($OCMe_3$), 33.12 ($OC\mathit{Me}_3$). IR (KBr): 2970 m, 1440 m, 1360 s, 1230 m, 1180 s, 1060 m, 1010 s, 960 s, 770 s cm^{-1}. Anal. Calcd: Ce, 33.0. Found: Ce, 32.9.

(26) **4** crystallizes in the orthorhombic space group *Pnma* with unit cell parameters at 183 K: a = 14.8351 (17), b = 13.3467 (14), c = 8.9677 (9) Å, V = 1775.6 (3) Å^3, and D_{calcd} = 1.53 g cm^{-3} for Z = 4. Least-squares refinement of the model using those 1661 data with $|F_o| > 6.0\sigma(|F_o|)$ converged to R_F = 5.1% and R_{wF} = 6.1%. Disorder in the *tert*-butyl group was modeled with a three-position approximation.

(27) This type of ligand redistribution chemistry is also observed in the reactions of NaCp with $Ce(OR)_4(THF)_2$[23] and $Ce(OR)_3(NO_3)(THF)_2$[23] which form **3** and other cerium(IV) alkoxide products.

(28) A delicate balance of factors may be involved in stabilizing isolable organometallic cerium(IV) complexes. Changing one ligand may lead to cerium(III) products. For example, use of KC_5Me_5 in reactions 1 and 2 can lead to cerium(III) products.

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Table I. Selected Bond Distances (Å) and Angles (deg) for $(C_5H_5)_3Ce(OCMe_3)$ (4)[a]

Ce(1)–O(1)	2.045 (6)	Ce(1)–C(1)	2.790 (9)
Ce(1)–C(2)	2.752 (11)	Ce(1)–C(3)	2.741 (10)
Ce(1)–C(4)	2.777 (14)	Ce(1)–C(5)	2.783 (12)
Ce(1)–C(9)	2.749 (10)	Ce(1)–C(10)	2.757 (12)
Ce(1)–C(11)	2.746 (15)	Ce(1)–Cn(1)	2.518
Ce(1)–Cn(2)	2.505	O(1)–C(6)	1.441 (13)
O(1)–Ce(1)–Cn(1)	103.5	O(1)–Ce(1)–Cn(2)	101.7
Cn(1)–Ce(1)–Cn(2)	116.9	Cn(1)–Ce(1)–Cn(1′)	111.8
Ce(1)–O(1)–C(6)	176.3 (6)		

[a] Cn(1) is the centroid of the C(1), C(2), C(3), C(4), C(5) ring. Cn(2) is the centroid of the C(9), C(10), C(11), C(10′), C(9′) ring.

atom roughly define a tetrahedron. $Cp_3Ce(OCMe_3)$ and the Cp_3UZ complexes differ, however, in the nature of their distortion from pure tetrahedral symmetry. Cp_3UZ complexes typically have near C_{3v} symmetry at uranium with Cn–U–Cn angles near 100° and Cn–U–Z angles near 117°.[1,30–36] In contrast, the solid-state structure of $Cp_3Ce(OCMe_3)$ has (a) a mirror plane through Ce, C(11), O(1), C(6), and C(8), (b) Cn–Ce–Cn angles of 116.9° and 111.8°, and (c) Cn–Ce–O angles of 103.5° and 101.7° (Table I).

The average Ce–C(ring) distance, 2.76 (2) Å, can be compared to U–C(ring) averages of 2.68–2.74 Å in Cp_3UZ complexes.[1,30–36] The relationship of the cerium(IV) distance to the uranium(IV) distances in these tetravalent complexes parallels the relationship of the cerium(III) and uranium(III) distances in the six trivalent complexes $(MeC_5H_4)_3ML$ where M = U and Ce and L = PMe_3,[37,38] $P(OCH_2)_3CEt$,[39] and quinuclidine.[39] In all of these cases, the cerium distances are longer than expected on the basis of Shannon's radii for halide and chalcogenide complexes[40] which show cerium to be slightly smaller than uranium. However, the crystallographic differences are small and statistically insignificant. The Ce–C(ring) distance in 4 can also be compared to the 2.81 (4) Å average Ce–C distance in $Cp_3Ce(THF)$.[41,42] According to Shannon,[40] ten-coordinate Ce(IV) is 0.18 Å smaller than ten-coordinate Ce(III). On this basis, the expected Ce–C average length in 4 would be 2.63 Å. The 2.045 (6) Å Ce–O distance is more difficult to assess since there are no structurally characterized Ce(IV) alkoxide complexes of comparable coordination number.[23,43] The 176.3 (6)° Ce–O(1)–C(6) angle is typical of lanthanide, actinide, and early-transition-metal alkoxide complexes which contain short metal–oxygen distances.[44]

The synthetic results described here demonstrate that ceric ammonium nitrate derived alkoxide nitrate complexes are useful precursors to cerium(IV) organometallic compounds. The structural results establish that complete crystallographic data can be obtained on isolable organometallic complexes of Ce(IV). When more structural data on Ce(IV) organometallics are obtained, it should be possible to determine if Ce(IV) bond distance parameters can be assessed in the same way as other f-element organometallic complexes.

Acknowledgment. We thank the Division of Chemical Sciences of the Office of Basic Energy Sciences of the Department of Energy for support of this research and the University of California, Irvine, for a President's Undergraduate Fellowship (to T.J.D.). Funds for the purchase of the Nicolet R3m/V diffractometer system were made available from the National Science Foundation under Grant CHE-85-14495.

Supplementary Material Available: Synthetic and structural details, tables of crystal data, positional parameters, bond distances and angles, and thermal parameters, and a disordered ORTEP plot (11 pages); a listing of structure factor amplitudes (8 pages). Ordering information is given on any current masthead page.

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Additions and Corrections

Crystal Structure of $(\eta^5\text{-}C_5Me_5)Ru(\mu\text{-}H)_4Ru(\eta^5\text{-}C_5Me_5)$: Corrigendum[1]

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Summary: The crystal structure of this compound, originally described in the noncentrosymmetric space group *Pa*, is correctly described as centrosymmetric, space group *P*2/*a*. Refinement in space group *P*2/*c*, the standard setting of *P*2/*a*, has led to much more reasonable geometry than reported for the *Pa* refinement: the bridging H atoms form a square rather than a rectangle, and the bond lengths and angles in the permethylcyclopentadienyl ring are now normal.

The crystal structure of this interesting compound, which contains a Ru–Ru triple bond as well as four H atoms bridging between the Ru atoms, has recently been reported.[2] A surprising feature of the structure was the arrangement of the bridging H atoms, which appeared to form a rectangle with two short H···H distances (0.84 and

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[1] Contribution No. 7614 from the Arthur Noyes Laboratory of Chemical Physics.

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