Synthesis and X-ray Crystal Structure of $[U(\eta\text{-}C_8H_8)(HMPA)_3][BPh_4]$, the First Cationic Monocyclooctatetraenyl Compound of Uranium (III), and its Neodymium Homologue

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The title compound was synthesized in 88% yield by reducing the uranium(IV) dication $[U(COT)(HMPA)_3][BPh_4]_2$ ($COT = \eta\text{-}C_8H_8$) with sodium amalgam while the neodymium analogue was prepared by substitution of the THF ligands in $[Nd(COT)(THF)_4][BPh_4]$. Comparison of the crystal structures of the complexes $[M(COT)(HMPA)_3][BPh_4]$ (M = U, Nd, Sm) shows that the variations in the M–ligand bond lengths simply reflect the differences in the ionic radii of the metals.

Introduction

Recently we reported the synthesis and structural characterization of the uranium(IV) complex $[U(COT)(HMPA)_3][BPh_4]_2$ (**1**) ($COT = \eta\text{-}C_8H_8$, HMPA = hexamethylphosphoramide), the first organometallic dication of an f-element, which was obtained by protonolysis of the parent borohydride $U(COT)(BH_4)_2$.[1] Cationic metal compounds are interesting since they are potential precursors in inorganic and organometallic syntheses as well as efficient catalysts in various organic reactions. In the course of our studies on the reactivity of **1**, we found that it could be reduced to the uranium(III) complex $[U(COT)(HMPA)_3][BPh_4]$ (**2**). Such cyclooctatetraenyl uranium compounds in the +3 oxidation state are quite rare, being limited to the anionic bis([8]annulene) complex $[K(diglyme)][U(\eta\text{-}C_8H_7Me)_2]$[2] and the neutral mixed-ring complex $[U(COT)(\eta\text{-}C_5Me_5)(L)]$ (L = THF, 4,4′-dimethyl-2,2′-bipyridine).[3] More generally, very few cationic organometallic complexes of uranium(III) have been described: the bis(pentadienyl) complex $[U(\eta\text{-}2,4\text{-}Me_2\text{-}C_5H_5)_2][BPh_4]$,[4] the metallocene complex $[U(\eta\text{-}C_5Me_5)_2(THF)_2][BPh_4]$,[5] and the trinuclear arene complex $[U_3(\eta\text{-}C_6Me_6)_3(AlCl_4)_3Cl_5][AlCl_4]$;[6] only these latter two have been crystallographically characterized. Here we present the synthesis and X-ray crystal structure of **2**, taking the opportunity to compare this structure with that of the U^{IV} dication of **1**, and also with those of the lanthanide analogues $[M(COT)(HMPA)_3]^+$ [M = Nd (**3**) and Sm (**4**)].

Results and Discussion

Synthesis and Characterization

The uranium(III) compound **2** was prepared by treating the orange dication **1** with a slight excess of sodium amalgam in tetrahydrofuran (THF), according to Equation (1).

$$\underset{\mathbf{1}}{[U(COT)(HMPA)_3][BPh_4]_2} + Na(Hg) \xrightarrow{THF} \underset{\mathbf{2}}{[U(COT)(HMPA)_3][BPh_4]} + NaBPh_4 \qquad (1)$$

After stirring for 16 h at 20 °C, the green solution was filtered and a green microcrystalline powder of **2** was deposited upon addition of pentane (88% yield). Compound **2** was characterized by elemental analysis (C, H N) and X-ray crystallography (vide infra). The ^1H NMR spectrum exhibits two resonances at $\delta = 1.7$ and -47.4 corresponding to the HMPA and COT ligands, respectively, and shows the presence of only one BPh_4 anion. Oxidation of **2** with $TlBPh_4$ failed to give back the dication **1** and afforded only insoluble unidentified products; this behaviour is not understood.

The synthesis of **2** was not affected by the small excess of sodium amalgam (<3 equivalents), although the monocation reacted further with larger amounts of the reducing agent (>9 equivalents) to give the uranium(IV) compound $U(COT)_2$ as the sole identifiable organometallic product. It is possible that the putative U^{II} species "U(COT)", formed by reduction of **2**, was transformed by disproportionation into uranium metal and uranocene, the latter being inert towards sodium amalgam. While the U^{III} compound $Na[U(\eta\text{-}C_5Me_5)_2Cl_2]$ is known to act as a uranium(II) synthetic equivalent,[7] it is only very recently that the toluene-bridged diuranium complex $[(\mu\text{-}C_7H_8)\{U[N(Ad)Ar]_2\}_2]$ (Ad = adamantyl, $Ar = 3,5\text{-}C_6H_3\text{-}Me_2$) was isolated and crystallographically characterized; this complex exhibits a reactivity which is consistent with the formality of divalent uranium.[8]

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The separation of lanthanide(III) and actinide(III) compounds is an important problem in the context of the reprocessing of nuclear wastes and from a basic point of view. Much attention is thus paid to the structural and chemical differentiation of homologous compounds of 4f and 5f elements.[9] In order to compare the crystal structure of **2** with that of lanthanide counterparts, the neodymium compound [Nd(COT)(HMPA)$_3$][BPh$_4$] (**3**) was synthesized by substitution of the THF ligands in [Nd(COT)(THF)$_4$][BPh$_4$][10] with HMPA, according to Equation (2).

$$[\mathrm{Nd(COT)(THF)_4}][\mathrm{BPh_4}] + 3\,\mathrm{HMPA} \xrightarrow{\text{pyridine}} \underset{\mathbf{3}}{[\mathrm{Nd(COT)(HMPA)_3}][\mathrm{BPh_4}]} + 4\,\mathrm{THF} \qquad (2)$$

Pink crystals of the solvate **3**.THF were obtained by crystallization from THF. The analogous samarium complex [Sm(COT)(HMPA)$_3$]I (**4**) was recently prepared by reaction of samarium metal, cyclooctatetraene, and iodine in the presence of HMPA, and its crystal structure has been determined.[11]

Crystal Structures

The crystals of **2** and **3**·THF are composed of discrete cations and anions. The BPh$_4$ anions display the expected structural parameters. A view of one of the two independent and almost identical cations of **2** and a view of the cation of **3**·THF are shown in Figure 1 and 2, respectively, together with selected bond lengths and angles; the atom numbering scheme is the same for the uranium and neodymium compounds.

The two structures are very similar, both cations adopting a three-legged piano-stool configuration in which the O−M−O angles vary from 83.3(5) to 88.5(6)° for M = U, and from 83.81(16) to 85.95(16)° for M = Nd; the COT−M−O angles (COT = centroid of the C$_8$H$_8$ ring) range between 124.5(8) and 132.3(7)° (M = U), and between 125.6(2) and 131.1(2)° (M = Nd). The U(1) and U(2) atoms are 2.00(1) and 2.03(1) Å from the planar COT ring (within ±0.02 Å) and the mean U(1)−C and U(2)−C bond lengths are 2.71(4) and 2.74(6) Å, respectively. These values are almost identical to those found in the other two crystallographically characterized monocyclooctatetraenyl U$^{\mathrm{III}}$ complexes, [K(diglyme)][U(η-C$_8$H$_7$Me)$_2$] [1.999 and 2.707(7) Å][2] and [U(COT)(η-C$_5$Me$_5$)(Me$_2$bipy)] (2.01 and 2.703 Å).[3] The Nd−COT distance of 1.984(5) Å in **3** compares well with those determined in mono(COT) neodymium complexes; for example, 1.906(6) Å in [Nd(COT)(THF)$_4$][BPh$_4$][10a] and 2.01(1) Å in [Nd(COT)I(THF)$_3$].[12]

Not surprisingly, the structure of the cation [Nd(COT)(HMPA)$_3$]$^+$ is quite similar to that of the samarium analogue **4** (Table 1);[11] the Nd−C and Nd−O distances are systematically slightly longer than the Sm−C and Sm−O distances, in agreement with the 0.03 Å difference between the ionic radii of Nd^{3+} and Sm^{3+}.[13]

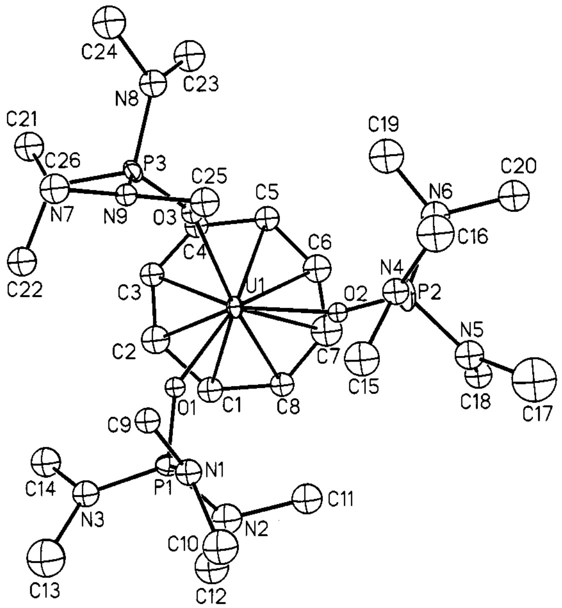

Figure 1. View of the crystal structure of one of the two independent cations [U(COT)(HMPA)$_3$]$^+$ with thermal ellipsoids drawn at the 30% probability level; selected bond lengths (Å) and angles (°): U(1)−C(1) 2.73(3), U(1)−C(2) 2.65(3), U(1)−C(3) 2.65(3), U(1)−C(4) 2.70(3), U(1)−C(5) 2.75(3), U(1)−C(6) 2.70(3), U(1)−C(7) 2.76(4), U(1)−C(8) 2.72(3), U(1)−O(1) 2.380(16), U(1)−O(2) 2.454(16), U(1)−O(3) 2.401(16), U(1)−COT 2.00(1); O(1)−U(1)−O(2) 84.9(5), O(1)−U(1)−O(3) 85.5(5), O(2)−U(1)−O(3) 83.3(5), COT−U(1)−O(1) 132.3(7), COT−U(1)−O(2) 128.1(8), COT−U(1)−O(3) 126.5(7); COT is the centroid of the C$_8$H$_8$ ring

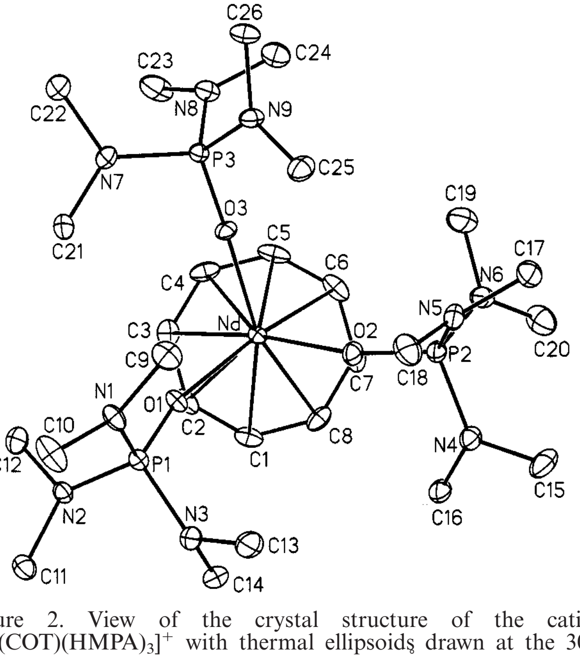

Figure 2. View of the crystal structure of the cation [Nd(COT)(HMPA)$_3$]$^+$ with thermal ellipsoids drawn at the 30% probability level; selected bond lengths (Å) and angles (°): Nd−C(1) 2.719(7), Nd−C(2) 2.709(7), Nd−C(3) 2.718(7), Nd−C(4) 2.698(7), Nd−C(5) 2.686(7), Nd−C(6) 2.697(7), Nd−C(7) 2.689(8), Nd−C(8) 2.704(7), Nd−O(1) 2.328(5), Nd−O(2) 2.392(5), Nd−O(3) 2.355(4), Nd−COT 1.984(5); O(1)−Nd−O(2) 85.95(16), O(1)−Nd−O(3) 85.76(16), O(2)−Nd−O(3) 83.81(16), COT−Nd−O(1) 125.6(2), COT−Nd−O(2) 131.1(2), COT−Nd−O(3) 129.1(2); COT is the centroid of the C$_8$H$_8$ ring

Table 1. Comparison of the geometrical parameters for the cations $[M(COT)L_3]^+$ (M = U, Nd, Sm[10]) and the dication $[U(COT)L_3]^{2+}$ [1] (L = HMPA)

	$[U(COT)L_3]^{+[a]}$	$[Nd(COT)L_3]^+$	$[Sm(COT)L_3]^+$	$[U(COT)L_3]^{2+[a]}$
<M−C> (Å)	2.71(4); 2.74(6)	2.70(1)	2.66	2.65(4); 2.65(3)
M−COT (Å)	2.00(1); 2.03(1)	1.984(5)	1.95	1.92(2); 1.92(2)
<M−O> (Å)	2.41(4); 2.40(6)	2.36(3)	2.31	2.22(2); 2.22(1)
<O−M−O> (deg)	84(1); 87(2)	85(1)	86	86(2); 87(3)

[a] The values for each independent cation are given.

The uranium cation **2** and its neodymium and samarium counterparts **3** and **4** permit a direct comparison of the structural data of homologous uranium(III) and lanthanide(III) compounds. Such comparative examinations have previously been made on only a few pairs of isostructural complexes, with the aim of evaluating and possibly differentiating the nature of the bonds in 4f and 5f element complexes.[14−16] In the cations **2**−**4**, the variations in the metal−ligand bond lengths, listed in Table 1, reflect the differences in the ionic radii of the metals (0.04 Å between U^{3+} and Nd^{3+}, 0.07 Å between U^{3+} and Sm^{3+}).[13] This regular trend agrees with that most generally observed, as for the complexes $[M(\eta\text{-}C_6Me_6)(AlCl_4)_3]$ (M = U, Sm)[14] or $[M(\eta\text{-}C_5Me_5)I_2(THF)_3]$ (M = U, Ce);[15] this was explained by the fact that f orbitals are not substantially involved in ligand bonding. To the best of our knowledge, it is only in the phosphorus complexes $[M(\eta\text{-}C_5H_4Me)_3L]$ (M = U, Ce; L = PMe_3 or $P(OCH_2)_3CEt$) that the U−P bond length was found to be significantly shorter than the Ce−P distance, whereas the U−L and Ce−L distances are the same in the derivatives with oxygen and nitrogen ligands (L = THF, quinuclidine).[16] This difference has been rationalised by postulating uranium-to-phosphorous π bonding which is absent in the lanthanide analogues.

If the structure of the U^{III} cation **2** is now compared with that of the parent U^{IV} dication **1**, it is noteworthy that the difference between the U−O bond lengths, 0.2 Å, is much greater than that observed between the U−C bond lengths, 0.1 Å, which corresponds to the ionic radii variation between U^{4+} and U^{3+}.[13] This feature certainly results from the more positive charge on the dicationic U^{IV} centre, which induces a stronger interaction with the Lewis base.

Experimental Section

General: All preparations were carried out under argon (less than 2 ppm oxygen and water) using standard Schlenk and vacuum line techniques or in a glove box. Solvents were thoroughly dried and deoxygenated by standard methods and distilled immediately before use. [D8]THF was dried over Na-K alloy. The ^1H NMR spectra were recorded on a Bruker DPX 200 instrument and were referenced internally using the residual protio solvent resonances relative to tetramethylsilane (δ = 0). Elemental analyses were performed by Analytische Laboratorien at Lindlar (Germany). $[Nd(COT)(THF)_4][BPh_4]$ was synthesized as previously reported.[10]

Synthesis of $[U(\eta\text{-}C_8H_8)(HMPA)_3][BPh_4]_2$ (1): This compound was prepared by a slight modification of the published procedure,[1] resulting in an improved yield. A flask was charged with $[(COT)U(BH_4)_2(THF)]$ (0.488 g, 1.10 mmol) and NEt_3HBPh_4 (1.011 g, 2.40 mmol) in THF (40 mL) and HMPA (1.1 mL, 6.32 mmol) was introduced with a syringe. The reaction mixture was stirred for 5 h at 20 °C during which time evolution of gas was observed and an orange powder was deposited from the dark red solution. After evaporation, the crude solid was dried under dynamic vacuum for 18 h. It was then washed with THF (40 mL) and extracted with pyridine (40 mL). The solution was filtered and the solvent was evaporated in vacuo, leaving **1** as a bright orange powder (1.573 g, 94%).

Synthesis of $[U(\eta\text{-}C_8H_8)(HMPA)_3][BPh_4]$ (2): A flask was charged with **1** (592.9 mg, 0.390 mmol) and 2% Na(Hg) (620.0 mg, 0.539 mmol of Na) in THF (40 mL). The reaction mixture was magnetically stirred for 1 h at room temperature, then immersed in an ultrasound bath (60 W, 40 kHz) for 3 h, and finally magnetically stirred for 12 h at room temperature. The green solution was filtered and its volume reduced to ca 10 mL; a green microcrystalline powder of **2** was deposited upon addition of pentane (20 mL). The powder was filtered off and dried under vacuum (411 mg, 88%). − ^1H NMR ([D8]THF): δ = 7.12 (br. t, 8 H, *o*-Ph), 6.72 (t, *J* = 6 Hz, 8 H, *m*-Ph), 6.60 (t, *J* = 6 Hz, 4 H, *p*-Ph), 1.7 (br. s, $w_{1/2}$ = 75 Hz, 54 H, HMPA), −47.4 (br. s, $w_{1/2}$ = 120 Hz, 8 H, COT). − $C_{50}H_{82}BN_9O_3P_3U$ (1199): calcd. C 50.09, H 6.90, N 10.51; found C 50.46, H 6.77, N 10.24.

Reaction of 2 with Na(Hg): An NMR tube was charged with **2** (8.1 mg, 6.76 μmol) and 2% Na(Hg) (72.3 mg, 62.9 μmol of Na) in [D8]THF (0.5 mL). The spectra showed the progressive formation of $U(COT)_2$ as the sole identifiable organometallic product, the reaction being complete after 3 days at 20 °C.

Synthesis of $[Nd(\eta\text{-}C_8H_8)(HMPA)_3][BPh_4]$ (3): A flask was charged with $[Nd(\eta\text{-}C_8H_8)(THF)_4][BPh_4]$ (50.51 mg, 0.059 mmol) in pyridine (10 mL), and HMPA (150 μL, 0.862 mmol) was added with a microsyringe. The colour of the solution turned from green to blue; the volume of the solution was reduced to ca 5 mL by evaporation and upon addition of pentane (10 mL), blue microcrystals of $\mathbf{3}\cdot 1/2C_5H_5N$ were deposited. These were filtered off and dried under vacuum (27.0 mg, 40%). − ^1H NMR ([D8]THF): δ = 8.44 (s, 1 H, C_5H_5N), 7.53 (s, 1 H, C_5H_5N), 7.20 (br. t, 8.5 H, *o*-Ph + C_5H_5N), 6.78 (t, *J* = 7 Hz, 8 H, *m*-Ph), 6.63 (t, *J* = 7 Hz, 4 H, *p*-Ph), 2.3 (br. s, $w_{1/2}$ = 70 Hz, 54 H, HMPA), −9.9 (br. s, $w_{1/2}$ = 100 Hz, 8 H, COT). − $C_{52.5}H_{84.5}BN_{9.5}O_3P_3Nd$ (1144): calcd. C 55.08, H 7.44, N 11.62; found C 54.91, H 7.58, N 11.75.

X-ray Crystallography of 2: Single crystals of **2** were obtained by crystallization from THF. A crystal of approximate dimensions 0.25 × 0.20 × 0.10 mm was selected. Diffraction collection was carried out on a Nonius diffractometer equipped with a CCD detector. The lattice parameters were determined from ten images recorded with 2° Φ-scans and later refined on all data. The data were recorded at 123 K. A 180° Φ-range was scanned with 2° steps with a crystal-to-detector distance fixed at 30 mm. Data were corrected for Lorentz-polarization and absorption[17] effects. The structure was solved by the heavy atom method and refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for the U and P atoms. H atoms were introduced at calculated positions as riding atoms with an isotropic displacement parameter equal to 1.2 (CH) or 1.5 (CH_3) times that of the parent atom. $C_{100}H_{164}B_2N_{18}O_6P_6U_2$ (2397.99), monoclinic, space group $P2_1$, a = 10.419(2), b = 31.018(6), c = 17.601(4) Å, β = 100.46(3)°, V = 5593.7(19) Å^3, Z = 2, D_c = 1.424 g cm^{-1}, Mo-K_α (λ = 0.71073 Å), μ_{Mo} = 3.035 mm^{-1}, $F(000)$ = 2444, total reflections = 18725, independent reflections = 7272 (R_{int} = 0.11), observed reflections [$I > 2\sigma(I)$] = 5613, R_1 = 0.075, wR_2 = 0.143. All calculations were performed on an O2 Silicon Graphics Station with the SHELXTL package.[18]

X-ray crystallography of 3·THF: Pink single crystals of **3**·THF were obtained by crystallization of **3**·1/2C_5H_5N from THF. A crystal of approximate dimensions 0.25 × 0.25 × 0.15 mm was selected. Diffraction collection was carried out on a Nonius diffractometer equipped with a CCD detector. The lattice parameters were determined from ten images recorded with 2° Φ-scans and later refined on all data. The data were recorded at 123 K. A 180° Φ-range was scanned with 2° steps with a crystal-to-detector distance fixed at 35 mm. Data were corrected for Lorentz-polarization and absorption [17] effects. The structure was solved by the heavy atom method and refined by full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-H atoms. H atoms were introduced at calculated positions as riding atoms with an isotropic displacement parameter equal to 1.2 (CH) or 1.5 (CH_3) times that of the parent atom. $C_{54}H_{90}BN_9O_4P_3Nd$ (1177.31), monoclinic, space group $P2_1/c$, a = 10.520(2), b = 21.481(4), c = 27.279(6) Å, β = 101.16(3)°, V = 6048(2) Å^3, Z = 4, D_c = 1.293 g cm^{-1}, Mo-K_α (λ = 0.71073 Å), μ_{Mo} = 0.987 mm^{-1}, $F(000)$ = 2476, total reflections = 29850, independent reflections = 8067 (R_{int} = 0.0842), observed reflections [$I > 2\sigma(I)$] = 5823, R_1 = 0.051, wR_2 = 0.122. All calculations were performed on an O2 Silicon Graphics Station with the SHELXTL package.[18]

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-151390 (**2**) and -151391 (**3**·THF). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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