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Studies on transition metal paracyclophane compounds. Synthesis and crystal structure of $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)\text{-}C_{16}H_{16})][BF_4]_2$: synthesis and NMR characterisation of the trinuclear heterometallic complex $[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$

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Abstract

When a solution of $[Ru(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$ and $[Os(\eta^6\text{-}C_6H_6((CH_3)_2CO)_3][BF_4]_2$ in a mixed $(CH_3)_2CO/CF_3CO_2H$ solvent is refluxed the paracyclophane ligand is transfered in part to the osmium ion. Pure samples of $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$ are obtained by refluxing a solution of $[Os(\eta^6\text{-}C_6H_6)((CH_3)_2CO)_3][BF_4]$ and paracyclophane in the mixed $(CH_3)_2CO/CF_3CO_2H$ solvent. This new osmium(II) compound has been characterised by NMR spectroscopy and X-ray crystallography. The reaction of $[Os(\eta^6\text{-}C_6H_6)((CH_3)_2CO)_3][BF_4]_2$ with $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2][BF_4]_2$ in neat CF_3CO_2H yields the novel trinuclear heterometallic complex $[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$, which has been isolated and characterised by ^1H and ^{13}C NMR spectroscopy.

Introduction

Recently we reported [1] that reaction of $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Cl_2]_2$ with a variety of donor ligands L (L = NC_5H_5, PPh_3, PMe_2Ph) in non-polar and polar solvents gave products of the types $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Cl_2L]$ and $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})ClL_2]^+$, respectively. The mononuclear nature of these complexes was confirmed by single crystal X-ray diffraction studies on the representative compounds $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Cl_2(PPh_3)]$ [2] and $[Ru(\eta^6[2_2](1,4)C_{16}H_{16})Cl(NC_5H_5)_2][PF_6]$ [1]. Binuclear products are formed when $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Cl_2]_2$ reacts with freshly prepared NaOR in ROH (R=Me, Et), as we established by crystal structure determinations of $[Ru_2(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2(OMe)_3][BPh_4]$ [2] and $[Ru_2(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2(OEt)_3][PF_6]$ [3]. We now report the exten-

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sion of our studies to osmium(II) paracyclophane complexes, and to heterometallic species.

Results and discussion

The synthetic technique first described by Bennett [4] for the preparation of bis(η^6-arene)Ru(II) complexes is readily extended to (η^6-arene) (η^6-cyclophane)Ru(II) complexes [5]. Using a strictly analogous procedure we have prepared the osmium complex $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$. The ^{1}H NMR spectrum, recorded in CD_3NO_2, exhibits a singlet due to the benzene protons at δ 6.91, singlets at δ 6.54 and 7.11 ppm due to the protons of the coordinated and non-coordinated aromatic rings of the paracyclophane ligand, and an AA'BB' coupling pattern centred at δ 3.36 due to the ethylenic protons of the paracyclophane ligand [Fig. 1(a)]. Integration of the spectrum supports the proposed stoichiometry. When the ^{1}H NMR spectrum of this osmium compound is compared

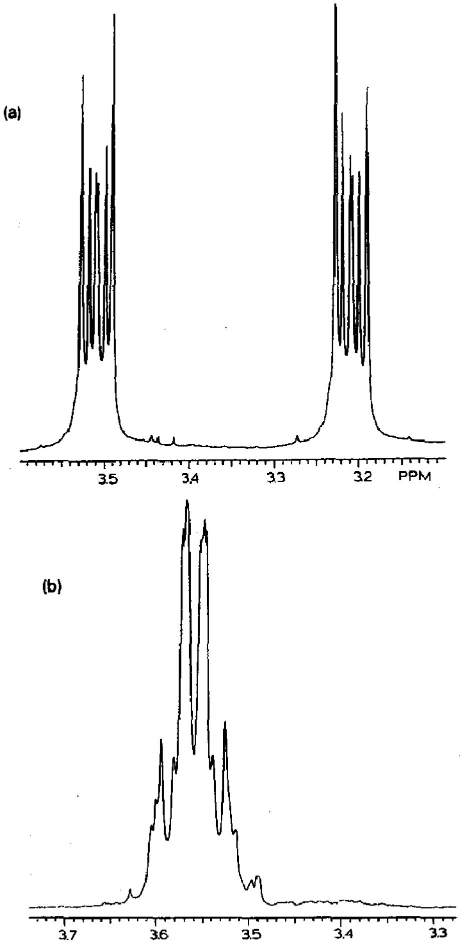

Fig. 1. ^{1}H NMR spectra, recorded in CD_3NO_2 at 400 MHz, of the ethylenic bridging groups of (a) $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$, and (b) $[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$.

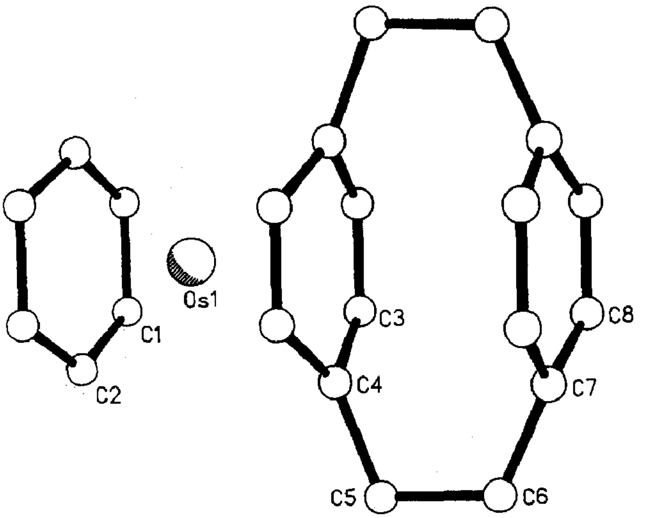

Fig. 2. Molecular structure of the cation $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})]^{2+}$ showing the numbering scheme adopted. Selected averaged bond lengths (Å): Os(1)–C(1) 2.22(2), Os(1)–C(2) 2.19(2), Os(1)–C(3) 2.20(2), Os(1)–C(4) 2.37(2), Os(1)–centroid of coordinated cyclophane ring 1.70, Os(1)–centroid of benzene ring 1.73, C(3)–C(4) 1.42(2), C(4)–C(5) 1.47(3), C(5)–C(6) 1.52(3), C(6)–C(7) 1.49(3), C(7)–C(8) 1.38(2), C(1)–C(1*) 1.36(5), C(3)–C(3*) 1.37(3), C(8)–C(8*) 1.39(3) (starred atoms generated by crystal symmetry).

with that of its ruthenium analogue the most noteworthy feature is the shifting of the signals due to the various aromatic protons to higher frequency [5], by ca 0.2 ppm. In contrast, when the ^{13}C NMR spectra of the two compounds are compared it is found that the signals from the ring carbon atoms appear, on average, at a chemical shift 7 ppm to lower frequency in the osmium compound.

Single crystals of the compound $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$ were obtained by slow evaporation of a dilute solution of the complex in acetonitrile, and the structure confirmed by X-ray diffraction. The cation is shown in Fig. 2. The osmium(II) ion is sandwiched between the benzene ring and one ring of the paracyclophane ligand in a similar fashion to reported structures of bis(η^6-arene)ruthenium(II) complexes [5,6]. For the reported (η^6-arene)osmium(II) structures [7–12] typical Os–C distances lie in the range 2.15–2.25 Å, while the corresponding metal–ring centroid distances average 1.68 Å. The corresponding values for this new structure are given in the figure caption, and are generally in agreement with previous data. The clear exception is the osmium to cyclophane bridgehead carbon distance Os(1)–C(4), 2.37(2) Å, which is significantly longer than the others. This is now a familiar and expected structural feature of paracyclophane. Both in the free ligand [13], and in its metal complexes, the aromatic rings are distorted into a shallow boat conformation by the electronic repulsions between the stacked rings. In the ruthenium(II) paracyclophane complexes which we studied previously [1–3] the ruthenium–bridgehead carbon distance fell in the range 2.27–2.38 Å. The distance between the aromatic decks of the cyclophane is 2.99 Å, which is less than the corresponding value for the free cyclophane, 3.09 Å. This reduction in inter-deck spacing, and by implication of the unfavourable electronic interactions, is often observed on coordination of a metal to the external face of the paracyclophane ligand [1–3,5,14]. Crystal symmetry accounts for the apparently 0° torsion angle in the interdeck connectivity, C(4)–C(5)–C(6)–C(7). In other compounds this angle typically has a value of 3–11° [1–3,5]. Early crystal structure studies of paracyclophane also gave an apparent torsion angle of 0° [15,16]; however, a subsequent careful redetermination of the structure [13] identified a dynamic disorder that occurs by a twist of the aromatic rings and results in a torsion angle of ca 3°. We feel that a similar disorder must be present in our metal complex, but have been unable to refine an acceptable model owing to the presence of the heavy metal ion.

During synthetic studies on a variety of complexes containing the "(η^6-$[2_2](1,4)C_{16}H_{16}$)ruthenium(II)" sub-unit, Boekelheide et al. prepared triple-layered complexes of the type $[(\eta^6\text{-arene})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6\text{-arene})][BF_4]_4$ [5,17]. It was our intention to prepare a mixed-metal analogue of this type of compound, namely $[(\eta^6\text{-}C_6H_6)Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_4$. To this end $[Ru(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$ was treated with $[Os(\eta^6\text{-}C_6H_6)((CH_3)_2CO)_3][BF_4]_2$ (1 : 1 mol ratio) in a mixture of $(CH_3)_2CO/CF_3CO_2H$ for 30 minutes under reflux. Cooling followed by precipitation with diethyl ether gave a pale yellow powder. The ^1H NMR spectrum, recorded in d^6-DMSO, showed two sets of resonances, which could be attributed to the cations $[Ru(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})]^{2+}$ and $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})]^{2+}$. These compounds were present in solution in an approximate ratio of 1 : 3, and thus we conclude that under the experimental conditions employed in this reaction the paracyclophane ligand has been substantially transferred to the osmium(II) ion.

We also investigated the reaction between $[Os(\eta^6\text{-}C_6H_6)((CH_3)_2CO)_3][BF_4]_2$, generated from $[Os(\eta^6\text{-}C_6H_6)Cl_2]_2$ by addition of $Ag[BF_4]$ in acetone, and the bisparacyclophane complex $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2][BF_4]_2$ in trifluoroacetic acid. A two-fold excess of the osmium compound was employed, and the reaction time was kept to a minimum (10 minutes) to avoid decomposition of the product. The analysis of the off-white solid obtained corresponded closely with that expected for the heterotrimetallic complex $[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2]\text{-}(1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$. Compelling evidence supporting the proposed trinuclear, quadruplelayered structure comes from the ^1H and ^{13}C NMR spectra of the compound recorded in CD_3NO_2. In the ^1H NMR spectrum there are just three singlet resonances in the aromatic region: at δ 7.14 for the benzene protons, at δ 6.95 for the aromatic cyclophane protons on the rings adjacent to the osmium ions, and at δ 6.68 for the protons on the aromatic rings bound to the ruthenium ion. In the closely related trinuclear ruthenium complex $[(\eta^6\text{-}C_6Me_6)Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}\text{-}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6\text{-}C_6Me_6)][BF_4]_6$ the aromatic protons of the cyclophane ligands resonate at δ 6.36 and 6.47 ppm. These differences in chemical shift are consistent with those described above for the monomeric complexes $[M(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$.

The ethylenic protons now show a radically different second-order coupling pattern [Fig. 1(b)]. The chemical shifts of the two sets of signals from the methylenic protons are now closely similar. In addition these signals now appear at higher frequency than either of the signals from the complexes in which only one face of the paracyclophane ligand is bound to a metal. This dramatic change in coupling pattern and the enhanced second-order character of the spectrum provide compelling evidence indicating that each face of the cyclophane ligand is coordinated to a different metal ion. The integration of the complete spectrum is consistent with the proposed stoichiometry.

We predict that seven resonances should be observed in the ^{13}C NMR spectrum of the heterometallic trinuclear cation. Two signals from aliphatic carbon atoms are observed at δ 33.3 and δ 32.9 ppm. The chemical shift difference between these is much smaller than that observed when only one face of the paracyclophane ligand is bound to a metal. Two signals from the non-bridgehead aromatic carbon atoms are observed, at δ 90.2 and 84.0 ppm, while two further signals, at δ 131.5 and 136.6 ppm, are assigned to the bridgehead carbon atoms. The resonance due to the benzene carbon atoms is observed at δ 89.6 ppm.

The significance of the results reported above lies in the obvious possibility of extension of this chemistry to the synthesis of heterometallo-organic polymers having one-dimensional properties.

Experimental

Microanalyses were carried out in the Chemistry Department of University College London. NMR spectra (^1H, ^{13}C) were obtained on a Varian VXR-400 spectrometer (chemical shifts quoted in ppm with positive values to high frequency of $SiMe_4$).

Materials

$[Ru(\eta^6\text{-}C_6H_6)Cl_2]_2$, $[Os(\eta^6\text{-}C_6H_6)Cl_2]_2$, $[Ru(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$ and $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2][BF_4]_2$ were prepared by published methods [4,5,18]. All other reagents were obtained from commercial suppliers.

All reactions were carried out in degassed solvents under nitrogen.

$[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$

To a suspension of $[Os(\eta^6\text{-}C_6H_6)Cl_2]_2$ (0.42 g, 0.61 mmol) in acetone (10 cm^3) was added $Ag[BF_4]$ (0.55 g, 2.83 mmol). The AgCl was removed by filtration through celite. Paracyclophane (0.25 g, 1.20 mmol) and CF_3CO_2H (5 cm^3) were added to the yellow/orange filtrate and the mixture was refluxed for 30 minutes then allowed to cool, and diethyl ether (50 cm^3) was added. The pale yellow precipitate was filtered off, washed with acetone and diethyl ether, and dried in vacuum. Yield 0.44 g, 55%. (Found: C, 40.7; H, 3.5. Calc. for $C_{22}H_{22}B_2F_8Os$: C, 40.6; H, 3.4%). ^1H NMR (CD_3NO_2): δ 6.91 (s, $C_6\mathit{H}_6$), δ 6.54 (s, metallated $C_6\mathit{H}_4$), δ 7.11 (s, non-metallated $C_6\mathit{H}_4$), δ 3.36 (AA′BB′, $-C\mathit{H}_2C\mathit{H}_2-$). ^{13}C NMR (CD_3NO_2): δ 87.2 ($C_6\mathit{H}_6$), δ 83.4 (metallated $C_2C_4H_4$), δ 133.0 (metallated $\mathit{C}_2C_4H_4$), δ 135.7 (non-metallated $C_2\mathit{C}_4H_4$), δ 141.1 (non-metallated $\mathit{C}_2C_4H_4$), δ 33.8 and 34.9 ($-\mathit{C}H_2CH_2$).

$[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$

To a suspension of $[Os(\eta^6\text{-}C_6H_6)Cl_2]_2$ (0.11 g, 0.17 mmol) in acetone (10 cm^3) was added $Ag[BF_4]$ (0.15 g, 0.77 mmol). The mixture was stirred at 20 °C for one hour and the AgCl then filtered off. The solvent was removed under vacuum to leave a residue of yellow $[Os(\eta^6\text{-}C_6H_6)((CH_3)_2CO)_3][BF_4]_2$, to which CF_3CO_2H (5 cm^3) and $[Ru(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})_2][BF_4]_2$ (0.06 g, 0.09 mmol) were added. The mixture was refluxed for 10 minutes and then cooled to give a pale yellow precipitate, which was filtered off, and identified as $[(\eta^6\text{-}C_6H_6)Os(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Ru(\eta^6,\eta^6\text{-}[2_2](1,4)C_{16}H_{16})Os(\eta^6\text{-}C_6H_6)][BF_4]_6$. Addition of diethyl ether (50 cm^3) to the reaction mixture gave a second crop of the precipitate which was filtered off, washed diethyl ether, and dried under vacuum. Combined yield 0.11 g, 0.07 mmol. (Found: C, 34.6; H, 2.8. Calc. for $C_{44}H_{44}B_6F_{24}Os_2Ru$: C, 33.5; H, 2.8%). ^{1}H NMR (CD_3NO_2): δ 7.14 (s, C_6H_6), δ 6.95 (s, OsC_6H_4), δ 6.68 (s, RuC_6H_4), δ 3.56 (A_2B_2, $-CH_2CH_2$). ^{13}C NMR (CD_3NO_2): δ 89.6 (C_6H_6), δ 84.0 and 90.2 ($C_2C_4H_4$), δ 131.5 and 136.6 ($C_2C_4H_4$), δ 33.3 and 32.9 ($-CH_2CH_2-$).

Crystal structure determination of $[Os(\eta^6\text{-}C_6H_6)(\eta^6\text{-}[2_2](1,4)C_{16}H_{16})][BF_4]_2$

Crystal data: $C_{22}H_{22}B_2F_8Os$, $M = 650.3$, orthorhombic, *a* 7.728(2), *b* 18.642(2), *c* 14.852(4) Å, *V* 2140(1) Å^3, D_c 2.02 g cm^{-3}, $Z = 4$, μ(Mo-K_α) 60.4 cm^{-1}, *F*(000) 1248, λ(Mo-K_α) 0.71073 Å, space group *Cmcm* from systematic absences.

A crystal of dimensions 0.80 × 0.12 × 0.02 mm was used to collect 1050 unique reflections in the range $5° \leqslant 2\theta \leqslant 50°$, on a Nicolet R3m/V diffractometer equipped with a molybdenum X-ray tube and graphite monochromator.

The asymmetric unit contains one quarter of the cation and one half of a $[BF_4]^-$ anion. The position of the osmium atom was derived from a three-dimensional Patterson function. Least-squares refinement followed by difference-Fourier synthesis were used to locate the remaining non-hydrogen atoms. It was immediately apparent that each component of the structure was subject to disorder. The cation is disordered in two respects. Firstly the cyclophane ligand is disordered over two

Table 1

Atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3)

	x	*y*	*z*	U_{eq} [a]
Os(1)	0	1282(1)	2500	33(1)
C(1)	956(16)	2203(6)	3336(9)	49(3)
C(2)	1811(29)	2180(10)	2500	52(5)
C(3)	883(20)	326(7)	3296(10)	28(3)
C(4)	1870(31)	234(12)	2500	36(5)
C(5)	3639(31)	−78(12)	2500	51(6)
C(6)	3502(36)	−887(14)	2500	67(7)
C(7)	1794(32)	−1215(12)	2500	40(5)
C(8)	871(24)	−1275(7)	3302(11)	37(3)
C(1A)	1480(54)	2231(20)	2921(29)	51(8)
C(2A)	0	2224(17)	3397(23)	19(7)
C(3A)	1552(19)	399(7)	2967(10)	31(3)
C(4A)	0	314(10)	3475(15)	33(4)
C(5A)	0	33(15)	4384(21)	66(7)
C(6A)	0	−787(11)	4388(16)	45(5)
C(7A)	0	−1133(12)	3451(15)	38(5)
C(8A)	1519(21)	−1202(8)	2980(11)	36(3)
B(1)	0	3605(14)	5391(14)	83(8)
F(1)	0	3603(13)	6284(14)	211(12)
F(2)	0	3496(32)	4570(29)	249(33)
F(3)	1541(31)	3447(23)	5139(18)	208(19)
F(2A)	0	2817(16)	5459(28)	173(19)
F(3A)	1128(41)	4134(16)	5307(26)	224(19)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

positions related by a 90 ° rotation with the two sites having equal occupancy. The benzene ring is similarly disordered, though in this case one site is occupied 75% of the time, the alternative site 25% of the time. The $[BF_4]^-$ anion is located on the mirror plane at $(0, y, z)$. The boron atom and two fluorines lie in that plane. The anion is highly disordered. Attempts to refine chemically reasonable models based upon interpenetrating rigid tetrahedra were largely unsuccessful. When the atoms of the anion were allowed to refine freely a chemically unsatisfactory solution was obtained. This solution did however result in the best overall refinement, and more importantly, led to the greatest precision for the parameters describing the chemically more interesting cation. As there are no short contacts between cation and anions this imperfect result was accepted. The osmium, boron, and fluorine atoms were refined anisotropically. The hydrogen atoms were included in fixed positions with a common isotropic temperature factor (U 0.08 $Å^2$). At the end of refinement $R = 0.0380$ ($R_w = 0.0402$), based on the 928 unique, absorption-corrected data with $I \leqslant 1.5\sigma(I)$. The largest shift/esd was 0.02 and the largest peak in the final difference-Fourier synthesis was 1.1 e $Å^{-3}$, close to the metal atom. Crystallographic calculations used the SHELXTL PLUS program package [19]. Final fractional coordinates are given in Table 1. Tables of observed and calculated structure factors and a full list of positional and thermal parameters are available on request from the authors.

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