Novel higher nuclearity osmium–antimony clusters by alkene- or diene-assisted cluster condensation

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The reaction of the cluster $Os_3(\mu\text{-}SbPh_2)(\mu\text{-}H)(\mu_3,\eta^2\text{-}C_6H_4)(CO)_9$, **1**, with a number of alkenes and dienes led to cluster condensation to afford the novel cluster $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)(\mu\text{-}H)_2(\mu_3,\eta^2\text{-}C_6H_4)(\mu,\eta^2\text{-}C_6H_4)(CO)_{14}$, **3**, as the major product, and another novel cluster $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)(\mu\text{-}H)(\mu_3,\eta^6\text{-}C_6H_4)(CO)_{14}(Ph)$, **4**. Reaction of **3** with the Group 15 ligands EPh_3 (E = P, As, Sb) afforded the corresponding monosubstituted derivatives $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)(\mu\text{-}H)_2(\mu_3,\eta^2\text{-}C_6H_4)(\mu,\eta^2\text{-}C_6H_4)(CO)_{13}(EPh_3)$, **6**.

Introduction

Reliable and selective synthetic routes to high nuclearity clusters are still important goals in transition metal carbonyl cluster chemistry. Among those routes that have emerged are photochemical activation, reaction of activated clusters with neutral mono- or di-metal complexes, condensation reactions,[1] "capping" reactions,[2] and surface-mediated reactions.[3] We have for some time now been interested in the reactivity of osmium–antimony clusters, and have recently shown that thermolysis of $Os_3(CO)_{11}(SbPh_3)$ led to condensation affording a large number of high nuclearity clusters containing six osmium atoms.[4] We report here that the reaction of $Os_3(\mu\text{-}SbPh_2)(\mu\text{-}H)(\mu_3,\ \eta^2\text{-}C_6H_4)(CO)_9$, **1**, with an alkene or a diene gives rise to two novel Os_5Sb_2 clusters *via* a condensation reaction.

Results and discussion

Thermolysis of the cluster $Os_3(\mu\text{-}SbPh_2)(\mu\text{-}H)(\mu_3,\eta^2\text{-}C_6H_4)\text{-}(CO)_9$, **1**, by itself gave rise to clusters containing six osmium atoms, which were derived from the condensation of two tri-osmium units.[4] In the course of our investigations into the reactivity of **1** with organic ligands, we have found that its reaction with a number of alkenes or dienes invariably gave the novel cluster $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)(\mu\text{-}H)_2(\mu_3,\eta^2\text{-}C_6H_4)(\mu,\eta^2\text{-}C_6H_4)\text{-}(CO)_{14}$, **3**, as one of the major products, together with smaller amounts of another novel cluster, $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)\text{-}(\mu\text{-}H)(\mu_3,\eta^6\text{-}C_6H_4)(C_6H_5)(CO)_{14}$, **4** (Scheme 1 and Table 1). In the case of the monoalkenes, the hexaosmium cluster $Os_6\text{-}(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)(\mu\text{-}H)(\mu_3,\eta^2\text{-}C_6H_4)_2(C_6H_5)(CO)_{16}$, **5**, was also isolated. This cluster had earlier been shown to be a condensation product of **1**.[4] The two novel clusters **3** and **4** have been characterised spectroscopically as well as by single-crystal X-ray crystallography; the ORTEP[5] diagrams showing their molecular structures, together with selected bond parameters, are given in Figs. 1 and 2, respectively.
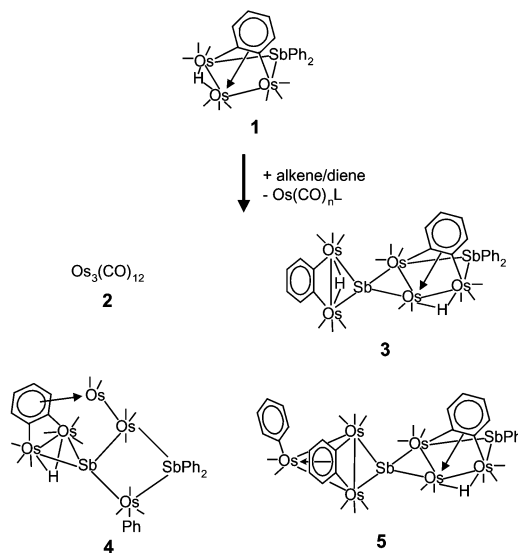
Scheme 1

We have also investigated the reactivity of cluster **3**. Thus, it reacts with Group 15 donor ligands under ambient conditions to afford the monosubstituted derivatives $Os_5(\mu_4\text{-}Sb)(\mu\text{-}SbPh_2)\text{-}(\mu\text{-}H)_2(\mu_3,\eta^2\text{-}C_6H_4)(\mu,\eta^2\text{-}C_6H_4)(CO)_{13}(EPh_3)$, **6** (E = P (**a**); As (**b**); Sb (**c**)), in good yields. The solid-state molecular structure of one of these (**6a**) has also been determined by a single-crystal X-ray crystallographic study (Fig. 3).

The Os_5Sb_2 metal cores of **3** and **6** may be regarded as being derived from that of **5** by elimination of a mononuclear osmium fragment. Indeed, the bond parameters associated with the Os(1)Os(2)Sb(6)Os(3)Sb(7) part of the metal core show similar trends for the three compounds. In particular, the

Table 1 Reaction conditions and products for **1** with alkenes/dienes

Ligand	Conditions	Yield of **2**[a](%)	Yield of **3**[a](%)	Yield of **4**[a](%)	Yield of **5**[a](%)
$H_2C=CH_2$	1.5 bar, 100 °C	23	41	5	–
$CH_3CH=CHCO_2Et$	2.0 equivalents, 100 °C	20	48	5	–
$PhCH=CHCH=CHPh$	5.1 equivalenst, 85 °C	21	29	6	28
$H_2C=C(CH_3)CH=CH_2$	1.1 equivalents, 80 °C	15	39	4	20

[a] % yields are calculated with respect to moles of Os for consumed **1**.
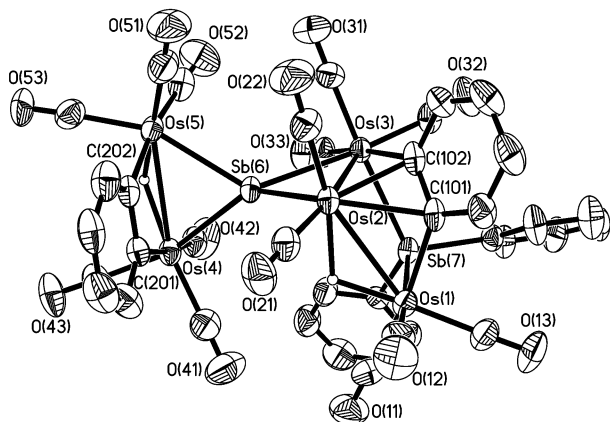
DOI: 10.1039/b108792d

Fig. 1 ORTEP diagram (organic hydrogens omitted, 50% probability thermal ellipsoids) and selected bond parameters for **3**. Os(1)–Os(2) = 2.9360(4) Å; Os(2)–Os(3) = 2.9537(5) Å; Os(4)–Os(5) = 3.0305(5) Å; Os(2)–Sb(6) = 2.5577(6) Å; Os(3)–Sb(6) = 2.8199(6) Å; Os(4)–Sb(6) = 2.6527(6) Å; Os(5)–Sb(6) = 2.6563(6) Å; Os(1)–Sb(7) = 2.6470(6) Å; Os(3)–Sb(7) = 2.6751(6) Å; Os(1)–C(101) = 2.172(9) Å; Os(1)–C(102) = 2.340(8) Å; Os(2)–C(102) = 2.222(8) Å; Os(3)–C(102) = 2.277(8) Å; Os(4)–C(201) = 2.156(9) Å; Os(5)–C(202) = 2.148(9) Å; Os(1)–Os(2)–Os(3) = 87.734(12)°; Os(2)–Sb(6)–Os(3) = 66.425(15)°; Os(4)–Sb(6)–Os(5) = 69.615(16)°; Os(1)–Sb(7)–Os(3) = 100.15(2)°.
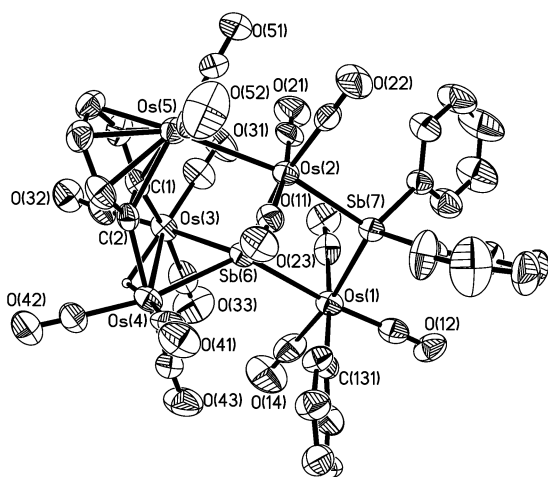

Fig. 2 ORTEP diagram (organic hydrogens omitted, 50% probability thermal ellipsoids) and selected bond parameters for **4**. Os(2)–Os(5) = 2.8910(6) Å; Os(3)–Os(4) = 2.9985(6) Å; Os(1)–Sb(7) = 2.7265(8) Å; Os(2)–Sb(6) = 2.7338(6) Å; Os(3)–Sb(6) = 2.6935(8) Å; Os(4)–Sb(6) = 2.6738(8) Å; Os(1)–Sb(7) = 2.7066(8) Å; Os(2)–Sb(7) = 2.6469(8) Å; Os(1)–C(131) = 2.150(10) Å; Os(3)–C(1) = 2.110(11) Å; Os(4)–C(2) = 2.110(10) Å; Os(5)–Os(2)–Sb(6) = 88.28(2)°; Os(1)–Sb(6)–Os(2) = 102.09(2)°; Os(2)–Sb(6)–Os(3) = 116.60(3)°; Os(3)–Sb(6)–Os(4) = 67.93(3)°; Os(1)–Sb(7)–Os(2) = 104.96(3)°.

Os(3)–Sb(6) bond lengths are invariably the longest of the Os–Sb bonds in each cluster. Indeed that for **3**, at a length of 2.8199(6) Å, is the longest observed to date. An interesting structural feature in cluster **4** is the presence of a μ_3,η^6-C_6H_4 ligand, the second such example in osmium cluster chemistry.[4] A close examination of the C–C bond lengths in this moiety shows that the C(1)–C(2) bond (1.454(13) Å) is significantly longer than the others (range from 1.399(14) to 1.406(13) Å), which are quite close to those of free benzene.[6] This is similar to the situation in the μ_3,η^2-C_6H_4 ligands of **3** and **6a**, and may suggest that there is some loss of aromaticity. The latter two clusters also possess a μ,η^2-C_6H_4 ligand; this has only been observed in two dinuclear *o*-phenylene complexes, *viz.*, $Ir_2(CO)_2Cp_2(C_6H_4)$ and $Fe_2(CO)_8(C_6F_4)$,[7,8] and one trinuclear derivative, $Os_3(CO)_8(\mu$-$H)_3(\mu,\eta^2$-$C_6H_4)(HC=NC_6H_5)$.[9] In contrast to the μ_3,η^2-C_6H_4 ligand, these appear to have retained their aromaticity; the C–C bond distance ranges are 1.363(17) to 1.414(12) Å and 1.36(2) to 1.39(2) Å, for **3** and **6a**, respectively.
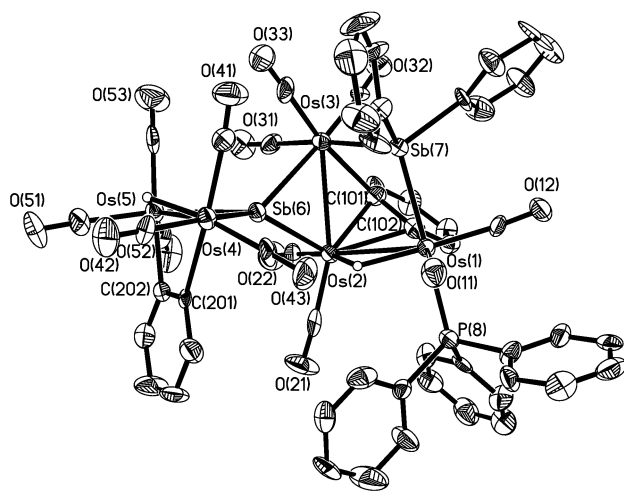

Fig. 3 ORTEP diagram (organic hydrogens omitted, 50% probability thermal ellipsoids) and selected bond parameters for **6a**. Os(1)–Os(2) = 2.9841(9) Å; Os(2)–Os(3) = 2.9660(9) Å; Os(4)–Os(5) = 3.0107(9) Å; Os(2)–Sb(6) = 2.5668(12) Å; Os(3)–Sb(6) = 2.7820(12) Å; Os(4)–Sb(6) = 2.6518(12) Å; Os(5)–Sb(6) = 2.6456(12) Å; Os(1)–Sb(7) = 2.6429(12) Å; Os(3)–Sb(7) = 2.6631(12) Å; Os(1)–P(8) = 2.419(4) Å; Os(1)–C(102) = 2.164(15) Å; Os(2)–C(101) = 2.214(16) Å; Os(2)–C(102) = 2.397(16) Å; Os(3)–C(101) = 2.317(16) Å; Os(4)–C(201) = 2.180(13) Å; Os(5)–C(202) = 2.135(15) Å; Os(1)–Os(2)–Os(3) = 88.06(2)°; Os(2)–Sb(6)–Os(3) = 67.21(3)°; Os(4)–Sb(6)–Os(5) = 69.27(3)°; Os(1)–Sb(7)–Os(3) = 102.42(4)°.

The structural similarities between **3** and **5** suggest that **5** may be a precursor of **3**. However, both **3** and **5** failed to react with or without alkenes/dienes under the same reaction conditions, thus ruling out any connection between them. It thus appears that **1** may have reacted with the alkene/diene to form an adduct initially which then undergoes condensation with another molecule of **1** to afford **3** *via* the elimination of a mononuclear osmium fragment; this mononuclear fragment would have carried off with it the alkene/diene. Such an activation towards metal–metal bond cleavage by an alkene/diene is consistent with observations on the photoactivation of $Os_3(CO)_{12}$,[10] and has also been proposed in another system.[11] We were not able to isolate or identify any mononuclear species from the reaction mixture, but we have found that although the ESMS spectrum of the reaction mixture did not show the presence of **2** it was among the chromatographically separated products. It is therefore possible that the mononuclear species aggregate during work-up to form **2**.

Thus we have found that alkenes and dienes may assist in cluster condensation reactions *via* elimination of an osmium fragment. In particular, the reaction of **1** with alkenes or dienes gave rise to the novel Os_5Sb_2 clusters **3** and **4** rather than the expected Os_6Sb_2 clusters as obtained from direct thermolysis.

Experimental

General procedures

All reactions and manipulations were performed under a nitrogen atmosphere by using standard Schlenk techniques. Solvents were purified, dried, distilled, and kept under nitrogen prior to use. NMR spectra were recorded on a Bruker ACF 300 MHz NMR spectrometer as $CDCl_3$ solutions unless otherwise stated. Electrospray mass spectra (ESMS) were obtained as an aqueous methanol (1 : 1, v/v) solution on a Finnigan MAT LCQ with a capillary voltage of 14 V and temperature of 353 K. Microanalyses were carried out by the microanalytical laboratory at the National University of Singapore. Cluster **1** was prepared by the literature method;[4] all other reagents were from commercial sources and used as supplied.

Table 2 Crystal data for **3**, **4** and **6a**

Compound	**3**·CH₂Cl₂	**4**·CH₂Cl₂	**6a**·0.5C₆H₁₄
Empirical formula	$C_{39}H_{22}Cl_2O_{14}Os_5Sb_2$	$C_{39}H_{22}Cl_2O_{14}Os_5Sb_2$	$C_{58}H_{42}O_{13}Os_5P\,Sb_2$
Formula weight	1979.97	1979.97	2172.39
Temperature/K	293(2)	223(2)	223(2)
Crystal system	Triclinic	Orthorhombic	Monoclinic
Space group	$P\bar{1}$	$Pbca$	$P2_1/c$
$a/\text{Å}$	11.7386(2)	18.3478(7)	15.3508(2)
$b/\text{Å}$	13.5125(3)	21.7070(9)	16.8332(3)
$c/\text{Å}$	16.0955(2)	23.1622(9)	24.7394(1)
$a/°$	79.6280(10)	90	90
$\beta/°$	75.1440(10)	90	96.180(1)
$\gamma/°$	74.2680(10)	90	90
Volume/Å³	2358.44(7)	9224.9(6)	6355.59(14)
Z	2	8	4
$D_c/\text{Mg m}^{-3}$	2.788	2.851	2.270
μ/mm^{-1}	14.714	15.047	10.873
$F(000)$	1768	7072	3964
Reflections collected	11192	72060	41516
Independent reflections	18978 [R(int) = 0.0311]	12701 [R(int) = 0.0944]	15878 [R(int) = 0.1256]
Goodness-of-fit on F^2	1.142	0.889	1.047
Final R indices [$I>2\sigma(I)$]	$R1 = 0.0387$, $wR2 = 0.0870$	$R1 = 0.0502$, $wR2 = 0.0810$	$R1 = 0.0743$, $wR2 = 0.1014$
R indices (all data)	$R1 = 0.0500$, $wR2 = 0.0955$	$R1 = 0.1039$, $wR2 = 0.0912$	$R1 = 0.1753$, $wR2 = 0.1305$
Largest diff. peak, hole/e Å⁻³	1.392, −1.829	2.432, −0.879	1.809, −1.656

Reaction of 1 with alkenes or dienes

In a typical reaction, cluster **1** (21.5 mg, 18 μmol) and excess 1,4-diphenylbuta-1,3-diene (19.2 mg, 93 μmol) were placed in a Carius tube with hexane (10 mL) and degassed (three freeze–pump–thaw cycles). The mixture was then heated for 15 h at 85 °C until the color changed to a reddish orange. Removal of solvent and volatiles *in vacuo* followed by TLC separation of the residue (hexane/dichloromethane, 5 : 1, v/v, as eluant) gave $Os_3(CO)_{12}$, **2** (2.8 mg, 20%) which was identified by its IR spectroscopic characteristics, unreacted **1** (3.2 mg), $Os_5(\mu_4\text{-Sb})$-$(\mu\text{-SbPh}_2)(\mu\text{-H})(\mu_3,\eta^2\text{-C}_6\text{H}_4)(\mu,\eta^2\text{-C}_6\text{H}_4)(CO)_{14}$, **3** (4.1 mg, 24%), $Os_5(\mu_4\text{-Sb})(\mu\text{-SbPh}_2)(\mu\text{-H})(\mu_3,\eta^6\text{-C}_6\text{H}_4)(C_6\text{H}_5)(CO)_{14}$, **4** (0.8 mg, 5%), and $Os_6(\mu_4\text{-Sb})(\mu\text{-SbPh}_2)(\mu\text{-H})(\mu_3,\eta^2\text{-C}_6\text{H}_4)_2$-$(C_6\text{H}_5)(CO)_{16}$, **5** (4.5 mg, 23%), in that order.

Similar reactions were carried out with ethene, ethyl crotonate and isoprene, and these are summarised in Table 1.

Cluster 3. IR (hexane) ν(CO) 2085 (sh), 2073s, 2033s, 2023w, 2008m, 1991w, 1970w cm⁻¹; ¹H NMR δ 7.6–6.6 (m, aromatic), −14.26 (s, OsHOs), −15.01 (s, OsHOs). Calculated for $C_{38}H_{20}O_{14}Os_5Sb_2$: C, 24.08; H, 1.06. Found: C, 23.84; H, 0.95%. The presence of dichloromethane in the crystalline samples used for the X-ray diffraction study was confirmed by ¹H NMR spectroscopy.

Cluster 4. IR (hexane) ν(CO) 2094w, 2086w, 2073m, 2063 (sh), 2035w, 2021w, 2016m, 2010m, 2002 (sh), 1988w, 1976w cm⁻¹; ¹H NMR δ 7.7–6.9 (m, aromatic), −14.34 (s, OsHOs). Calculated for $C_{38}H_{20}O_{14}Os_5Sb_2$·¼$C_6H_{14}$: C, 24.75; H, 1.24. Found: C, 24.64; H, 0.90%. The presence of hexane in the crystalline samples used for elemental analysis was confirmed by ¹H NMR spectroscopy.

Cluster 5. IR (hexane) ν(CO) 2098w, 2086vs, 2060m, 2042vs, 2031s, 2016, 1991w, 1962m cm⁻¹, which is identical to the literature values.[4]

Reaction of 3 with EPh₃ (E = P, As, Sb)

In a typical reaction, cluster **3** (21.5 mg, 11 μmol) and PPh₃ (6.5 mg, 25 μmol) were stirred together in dichloromethane (10 mL) at room temperature until the IR spectrum of the solution showed that **3** had been consumed (≈1 d). Removal of the solvent followed by chromatographic separation on silica gel using dichloromethane/hexane (3 : 7, v/v) as eluant gave orange $Os_5(\mu_4\text{-Sb})(\mu\text{-SbPh}_2)(\mu\text{-H})_2(\mu_3,\eta^2\text{-C}_6\text{H}_4)(\mu,\eta^2\text{-C}_6\text{H}_4)$-$(CO)_{13}(PPh_3)$, **6a** (15.2 mg, 65%).

Cluster 6a. IR (hexane) ν(CO) 2086m, 2068s, 2022 (sh), 2001m, 1961w (br) cm⁻¹; ¹H NMR δ 7.5–6.4 (m, aromatic), −13.86 (d, $^2J_{PH}$ = 7.4 Hz, OsHOs), −14.75 (s, OsHOs); ³¹P{¹H} NMR δ −5.33 (s). Calculated for $C_{55}H_{35}O_{13}Os_5Sb_2P$: C, 31.02; H, 1.66; P, 1.45. Found: C, 30.63; H, 1.46; P, 1.20%.

Similar reactions were carried out with AsPh₃ to yield the arsine analogue $Os_5(\mu_4\text{-Sb})(\mu\text{-SbPh}_2)(\mu\text{-H})_2(\mu_3,\eta^2\text{-C}_6\text{H}_4)(\mu,\eta^2$-$C_6H_4)(CO)_{13}(AsPh_3)$, **6b** (46%), and with SbPh₃ to yield the stibine analogue $Os_5(\mu_4\text{-Sb})(\mu\text{-SbPh}_2)(\mu\text{-H})_2(\mu_3,\eta^2\text{-C}_6\text{H}_4)(\mu,\eta^2$-$C_6H_4)(CO)_{13}(SbPh_3)$, **6c** (61%).

Cluster 6b. IR (hexane) ν(CO): 2086m, 2068 (sh), 2025 (sh), 2005s, 1989w, 1967w, 1963w cm⁻¹; ¹H NMR (d₈-toluene) δ 7.7–6.4 (m, aromatic), −13.76 (s, OsHOs), −14.80 (s, OsHOs). Calculated for $C_{55}H_{35}AsO_{13}Os_5Sb_2$: C, 30.39; H, 1.62. Found: C, 30.80; H, 1.92%.

Cluster 6c. IR (hexane) ν(CO) 2087 (sh), 2070 (sh), 2021vs, 2001m, 1963w (br) cm⁻¹; ¹H NMR δ 7.5–7.1 (m, aromatic), −14.04 (s, OsHOs), −14.84 (s, OsHOs). Calculated for $C_{55}H_{35}O_{13}Os_5Sb_3$·$C_6H_{14}$: C, 31.75; H, 2.12. Found: C, 31.61; H, 1.65%. The presence of hexane in the sample used for elemental analysis was confirmed by ¹H NMR spectroscopy.

Crystal structure determination of 3, 4 and 6a

The crystals were grown by slow cooling of CH₂Cl₂/hexane solutions and were mounted onto glass fibres. Crystal data and structure refinement details are given in Table 2. The intensities were measured on a Siemens SMART diffractometer, equipped with a CCD detector, using Mo-Kα radiation (λ = 0.71073 Å) at 223(2) K (293(2) K for **3**). The data were corrected for Lorentz and polarisation effects with the SMART suite of programs,[12] and for absorption effects with SADABS.[13] The final unit cell parameters were obtained by least squares on 5888 (**3**), 7601 (**4**) or 8020 (**6a**) strong reflections. Structural solution and refinement were carried out with the SHELXTL suite of programs.[14] The structures were solved by direct methods to locate the heavy atoms, followed by difference maps for the light, non-hydrogen atoms. All the organic hydrogen atoms were placed in calculated positions. The positions of the metal hydrides in **3** and **6a** were located in low angle difference maps while those for **4** were calculated with XHYDEX;[15] those for **3** were refined

while those for **4** and **6a** were allowed to ride on one of the osmium atoms to which they were attached. All the hydrides were assigned a fixed isotropic thermal parameter of 0.05 Å². All non-hydrogen atoms were given anisotropic displacement parameters in the final refinement.

CCDC reference numbers 171668–171670.

See http://www.rsc.org/suppdata/dt/b1/b108792d/ for crystallographic data in CIF or other electronic format.

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