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The reaction of $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-CO})]^-$ with Ph_2SbCl: a cluster with a six-membered Os_4Sb_2 ring and another with fluxional metal–metal bonds†

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Besides the cluster $[Os_3(\mu\text{-H})(\mu\text{-SbPh}_2)(CO)_{10}]$, **1**, other products identified from the reaction of $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-CO})]^-$ with Ph_2SbCl in THF were $[Os_3(\mu\text{-SbPh}_2)_2(CO)_{10}]$, **2a**, and a dimeric form of **1**, *viz.* $[Os_3(\mu\text{-H})(\mu\text{-SbPh}_2)(CO)_{10}]_2$, **3**. Carrying out the reaction in CH_2Cl_2 gave **3** as the major product, while **2a** has been demonstrated to result from the reaction of **1** with Ph_2SbCl. In **2a** the Os_3Sb_2 unit is almost planar; one of the Ph_2Sb bridges is open while the other is closed. The two Ph_2Sb-bridged Os–Os edges are shown by EXSY and variable-temperature ^1H NMR spectroscopy on the analogue $[Os_3(CO)_{10}(\mu\text{-SbPh}_2)\{\mu\text{-Sb}(C_6H_4Me\text{-}p)_2\}]$, **2b**, to interchange *via* an Os–Os bond fluxion.

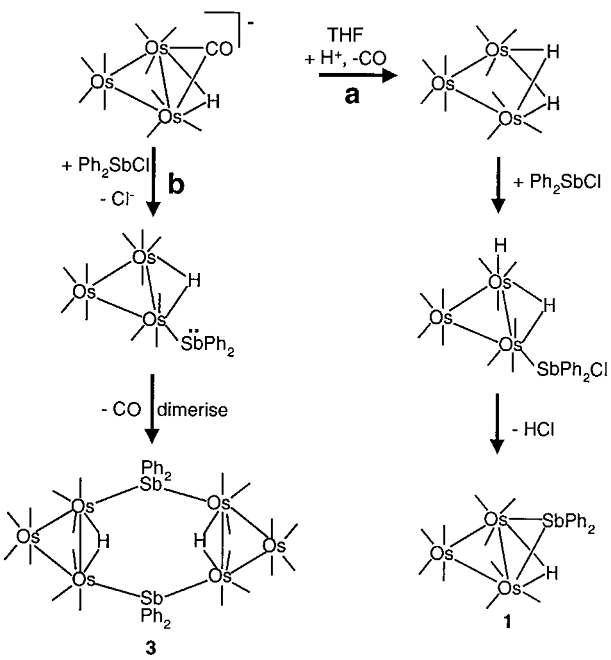
Introduction

One of the most exciting developments in organometallic cluster chemistry in recent years has been that of main group–transition metal cluster compounds. These compounds are expected to show structural and reactivity patterns that may be quite unlike those of the homometallic main group or transition metal clusters, the result of the interplay between the differing properties of the elements. Furthermore, there is a steady movement towards the view that the main group elements in many such compounds should be better regarded as an integral part of the cluster core, rather than as ligands.[1–3] We have been interested in this view for some time now, and one of the systems that we have actively been studying is antimony–osmium.[4,5] We had chosen $[Os_3(\mu\text{-H})(\mu\text{-SbPh}_2)(CO)_{10}]$, **1**, as the starting point of our investigations in the belief that the available report on its preparation was a straightforward, high-yielding reaction.[6] In the course of our own attempts at the synthesis of **1**, however, we have found that the products obtainable are dependent on the solvent system and the reaction time.

Results and discussion

When the reaction of $[Et_4N][Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-CO})]$ and an excess of Ph_2SbCl was carried out in THF three cluster products were separated and identified, *viz.* $[Os_3(\mu\text{-H})(\mu\text{-SbPh}_2)(CO)_{10}]$, **1**, $[Os_3(\mu\text{-SbPh}_2)(CO)_{10}]_2$, **2a**, and $[Os_3(\mu\text{-H})(\mu\text{-SbPh}_2)(CO)_{10}]_2$, **3**, in 60, 10 and 15% yields, respectively; cluster **1** was the only identified product in the earlier report.[6] When the reaction was carried out over a longer period of time the yield of **2a** increased at the expense of **1**. Furthermore, when the reaction solvent was changed to CH_2Cl_2 the major compound isolated was **3**; no **1** was formed.

The cluster **3** may be regarded as a dimeric form of **1** in which the $SbPh_2$ moieties bridge across two triosmium units rather than across one edge of such a unit. This structural type has only been observed in one other instance, a ruthenium–phosphorus analogue $[Ru_3(\mu\text{-H})\{\mu\text{-P}(CF_3)_2\}(CO)_{10}]_2$, which was obtained in 3% yield.[7] That the formation of **3** was solvent-dependent was an interesting point. Furthermore, we noted that the reaction in THF was almost immediate whereas that in CH_2Cl_2 was much slower (as judged by the colour changes in the reaction mixtures). We have also established that **1** and **3** do not interconvert at ambient or slightly elevated temperatures, which is evidence that their formation follows different pathways. One plausible reaction scheme is shown in Scheme 1.



Scheme 1

The presence of moisture or possibly acid (from hydrolysis of Ph_2SbCl) when the more polar solvent THF is employed may have resulted in protonation of $[Os_3(\mu\text{-H})(CO)_{10}(\mu\text{-CO})]^-$ to form the cluster $[Os_3H(\mu\text{-H})(CO)_{11}]$.[8] This species is known to decarbonylate easily to form the unsaturated cluster $[Os_3(\mu\text{-H})_2(CO)_{10}]$ which can undergo nucleophilic addition with Ph_2SbCl to generate the species $[Os_3H(CO)_{10}(SbPh_2Cl)]$; subsequent elimination of HCl would result in the formation of **1**. The protonation step would account for the accelerated rate of

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† Electronic supplementary information (ESI) available: EXSY spectrum of compound **2b**. See http://www.rsc.org/suppdata/dt/b0/b006669i/

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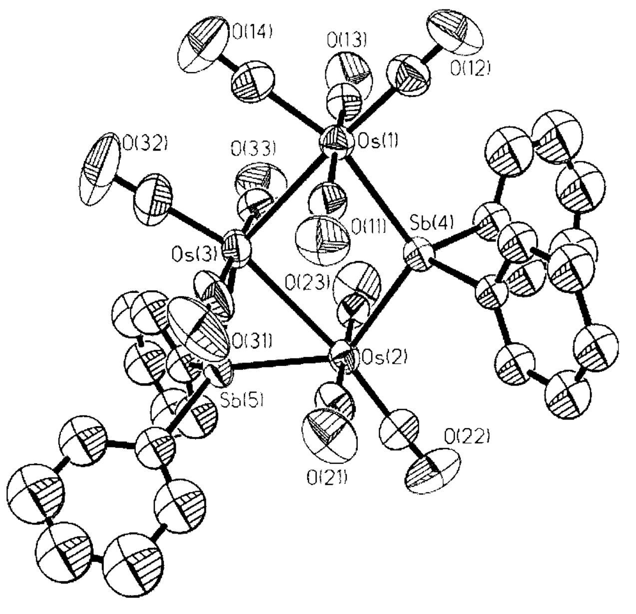
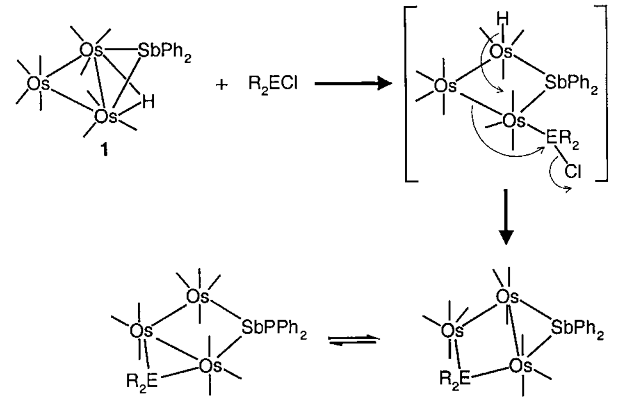


Fig. 1 An ORTEP[9] diagram (50% thermal ellipsoids) for compound **2a**.

colour change in THF. In accord with this proposed route, we have observed that the reaction of the unsaturated cluster $[Os_3(\mu\text{-}H)_2(CO)_{10}]$ with Ph_2SbCl indeed gave **1** in good yield. We have previously observed that **1** does have a tendency to undergo nucleophilic addition by cleavage of the $SbPh_2$-bridged Os–Os bond, *i.e.* antimony prefers formation of an open $SbPh_2$ bridge.[4] It is therefore possible that the proposed intermediate $[Os_3H(SbPh_2)(CO)_{11}]$ in route **b** may prefer dimerisation to form **3** (with open $SbPh_2$ bridges) over the formation of **1** (with a closed $SbPh_2$ bridge). In accord with this argument we have found that, unlike **1**, cluster **3** did not undergo nucleophilic addition with PPh_3 at ambient temperatures.

Cluster **2a** comprised a planar Os_3Sb_2 metal core (Fig. 1); this planarity persisted in solution, as was evident by the presence of resonances corresponding to only two different phenyl environments in the aromatic region. We established that **1** reacted with Ph_2SbCl to afford **2a** in essentially quantitative yield. A plausible mechanism for the formation of **2a** is therefore that depicted in Scheme 2. The structure of the intermediate was suggested by our earlier observation that nucelophiles (including $SbPh_3$) reacted with **1** to form adducts with the structure shown.[4] Indeed, monitoring of the reaction by ^1H NMR spectroscopy showed the formation of an intermediate with a resonance at δ −7.72. Although we did not obtain an IR spectrum of the intermediate, we did manage to do so for the corresponding intermediate formed from the reaction between **1** and $(p\text{-}MeC_6H_4)_2SbCl$ (see also below); the difference IR spectrum showed CO absorptions at 2114w, 2081m, 2069m, 2031s, 2007m and 1970w,br cm^{-1}, which has the same pattern as that of $[Os_3H(SbPh_2)(CO)_{10}(PPh_3)]$;[4] the ^1H NMR spectrum for this showed a resonance at δ −7.76. Both the ^1H NMR and IR data are thus consistent with the proposed structure.



Scheme 2

In an attempt to verify the last step, which involved rearrangement of an Os–Os bond, cluster **1** was treated with Ph_2PCl with the view that if the proposal was correct we would form cluster **4** as the isomer **A** (Scheme 2); the phosphorus atom would also provide a useful ^{31}P NMR probe. As it turned out, cluster **4** was found to have the structure of isomer **B**. It may be that a cluster with structure **A** was indeed formed initially but it isomerised quickly to isomer **B**, the driving force for this rearrangement being the lesser tendency for the smaller phosphorus atom in PR_2 bridges to span open Os–Os edges.

To circumvent this isomerisation process, $[Os_3(\mu\text{-}SbPh_2)\{\mu\text{-}Sb(C_6H_4Me\text{-}p)\}_2(CO)_{10}]$, **2b**, was prepared from the reaction of **1** with $(p\text{-}MeC_6H_4)_2SbCl$. The ^1H NMR spectrum of **2b** showed two sets of signals due to the CH_3 groups, indicating the presence of two isomers in solution which differed in whether the $SbPh_2$ or the $Sb(C_6H_4Me\text{-}p)_2$ group formed the open bridge. A variable temperature ^1H NMR spectroscopic study (in d_8-toluene) showed that these two sets of signals coalesced at 85 °C, corresponding to an exchange rate of 14.6 s^{-1} or a $\Delta G^‡$ of 80.2 kJ mol^{-1} at that temperature; the coalescence was reversible and indicated no decomposition. An EXSY experiment was also carried out to confirm chemical exchange between these two sets of signals. Although the two CH_3 signals were too close to each other (δ 2.35 and 2.38, respectively) so that any crosspeaks between them would lie very near to the main diagonal and difficult to observe, it was fortuitous that we were able to identify the *ortho*-H signals of the *p*-tolyl groups in the two isomers, which were traced *via* NOE crosspeaks. Hence, the two CH_3 signals at δ 2.35 and 2.38 (labelled **I** and **II**, respectively) showed NOE crosspeaks with the aromatic signals at δ 7.17 and 7.21, respectively, which were therefore assignable to the respective *meta*-H of the tolyl groups. These in turn showed NOE crosspeaks to signals at δ 7.43 and 7.58, which may thus be assigned to the corresponding *ortho*-H of the *p*-tolyl groups in the two isomers **I** and **II**, respectively. It was not possible to decide which of **I** and **II** has the structure **A** (and hence which **B**), but these *ortho*-H resonances were sufficiently apart in their chemical shifts so that exchange crosspeaks between them were clearly observed, confirming the presence of chemical exchange between **I** and **II**. We have attributed this chemical exchange as being mediated through a metal–metal bond fluxion, one of as-yet relatively few examples.[10]

Crystallographic study

The molecule of compound **3** is located on a crystallographic twofold axis; the ORTEP diagram and selected bond parameters are given in Fig. 2. The most obvious feature is the two $SbPh_2$ bridges that link together two triosmium units. The central six-membered Os_4Sb_2 ring is puckered about the Sb atoms into a "butterfly-like" conformation; the dihedral angle between the two $SbOs_2Sb$ planes is 39.8°. This brings one phenyl ring from each $SbPh_2$ unit into a mutually *cis* orientation. The OsSbOs angle is rather large; at 121.61(2)°, it is very much larger than the value of just below 106° found in clusters of the type $Os_3H(SbPh_2)(CO)_{10}(L)$ which also contain an $SbPh_2$ unit bridging two non-bonded osmium atoms. The structural features about each of the triosmium units are fairly typical: the Os–Os edge believed to be bridged by the metal hydride is appreciably longer (3.0693(4) Å) than the other two edges (2.8881(4) and 2.8950(4) Å), and the Os···O distances (sum of Os–C and C–O bond lengths) are longer for the axial

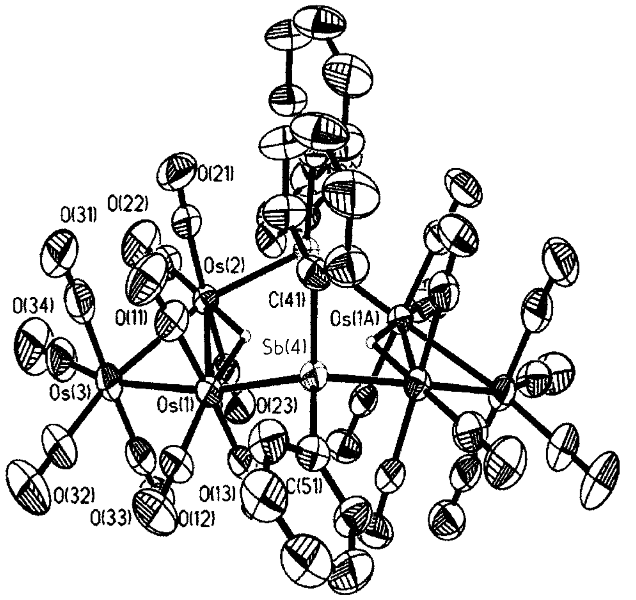


Fig. 2 An ORTEP diagram (50% thermal ellipsoids) and selected bond parameters for compound **3**. Os(1)–Os(2) 3.0693(4); Os(2)–Os(3) 2.8881(4); Os(1)–Os(3) 2.8950(4); Os(1)–Sb(4) 2.6881(6); and Os(2A)–Sb(4) 2.6823(5) Å; Os(1)–Sb(4)–Os(2A) 121.61(2); Os(1)–Os(2)–Os(3) 58.055(10); Os(2)–Os(3)–Os(1) 64.110(10); and Os(3)–Os(1)–Os(2) 57.835(10)°.

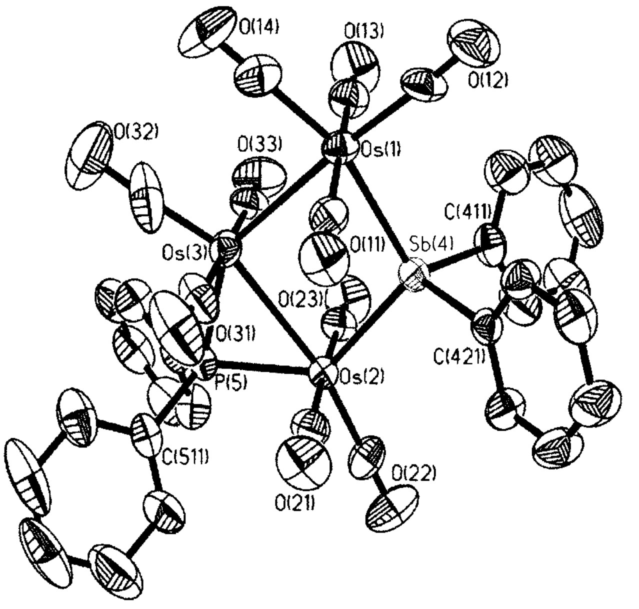


Fig. 3 An ORTEP diagram (50% thermal ellipsoids) for compound **4**.

(3.080 to 3.101 Å) than for the equatorial carbonyls (3.034 to 3.055 Å).

Clusters **2a** and **4** are isostructural; the ORTEP diagram for **4** showing the atomic labelling scheme is given in Fig. 3, and selected bond parameters for **2a** and **4** are given in Table 1. The Os_3SbE unit is almost coplanar, the dihedral angle between Os(1)Os(3)Os(2)Sb(4) and Os(2)Os(3)E(5) being 1.2 and 1.6° for **2a** and **4**, respectively. This is in sharp contrast to that of the corresponding selenium analogues, $[Os_3(\mu\text{-}SeR)_2(CO)_{10}]$ (R = Me or Ph), which have the SeR group bridging the closed Os–Os edge far out of the Os_3Se plane, in an axial-type position.[11] This difference in structure is presumably due to the steric constraint imposed by the SbR_2 group; the presence of another R group in SbR_2 as compared to SeR implies that any bending of one of the SbR_2 groups out of the Os_3Sb plane would meet with steric hindrance from the axial carbonyls. The most interesting feature is the large difference in the Os(2)–Os(3) bond lengths (3.0408(6) Å and 2.9436(5) Å for **2a** and **4**, respectively), which is in sharp contrast with the essentially identical Os(1)–Os(3) bond lengths. This is a clear example of the effect of the size of the bridging atom on the length of the bridged edge.

Table 1 Selected bond lengths (Å) and bond angles (°) for compounds **2a** and **4**

	2a (E = Sb)	**4** (E = P)
Os(1)–Os(3)	2.9967(6)	2.9968(5)
Os(2)–Os(3)	3.0408(6)	2.9436(5)
Os(1)–Sb(4)	2.7132(8)	2.6993(6)
Os(2)–Sb(4)	2.6384(8)	2.6334(6)
Os(2)–E(5)	2.6164(9)	2.350(2)
Os(3)–E(5)	2.6023(9)	2.311(2)
Os(1)–Sb(4)–Os(2)	110.61(3)	109.07(2)
Os(1)–Os(3)–Os(2)	93.574(17)	93.965(14)
Os(3)–E(5)–Os(2)	71.28(2)	78.33(7)

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. Routine ^1H and ^{31}P NMR spectra were recorded on a Bruker ACF300 NMR spectrometer in $CDCl_3$ unless otherwise stated. The variable-temperature study was carried out on a Bruker DPX300 NMR spectrometer while the EXSY was carried out on a Bruker AVANCE DRX500 NMR spectrometer using a mixing time of 450 μs. Microanalyses were carried out by the microanalytical laboratory at the National University of Singapore. The cluster $[Et_4N][Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}CO)]$, Ph_2SbCl and $(p\text{-}MeC_6H_4)_2SbCl$ were prepared according to literature methods;[12,13] all other reagents were from commercial sources and used as supplied.

Reactions of $[Et_4N][Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}CO)]$ and Ph_2SbCl

In THF. To a solution of $[Et_4N][Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}CO)]$ (100 mg, 0.099 mmol) in THF (20 mL) was added Ph_2SbCl (60 mg, 0.193 mmol). The solution changed from dark red to yellow immediately. After stirring for 30 min the solvent was removed *in vacuo* and the residue separated by TLC with CH_2Cl_2–hexane (20:80, v/v) as eluent to afford two major bands. Band 1 gave orange crystals of $[Os_3(\mu\text{-}H)(\mu\text{-}SbPh_2)(CO)_{10}]$, **1** (67.6 mg, 60%). IR (ν(CO), hexane): 2102mw, 2068w, 2054vs, 2022s, 2012ms, 1988mw and 1990m cm^{-1}. ^1H NMR ($CDCl_3$): δ −19.84 (s, OsHOs). Calc. for $C_{22}H_{11}O_{10}Sb$: C, 23.43; H, 0.98. Found: C, 23.67; H, 0.98%. Band 2 gave a mixture of $[Os_3(SbPh_2)_2(CO)_{10}]$, **2a** (14 mg, 10%), and $[Os_3(\mu\text{-}H)(\mu\text{-}SbPh_2)(CO)_{10}]_2$, **3** (17 mg, 15%). These two compounds were separated by fractional recrystallisation. **2a**: IR (ν(CO), hexane) 2102mw, 2054mw, 2033 (sh), 2026s, 2001w, 1983mw, 1972w and 1960mw cm^{-1} (Calc. for $C_{34}H_{20}O_{10}Os_3Sb_2\cdot\frac{1}{2}C_6H_{14}$: C, 30.74; H, 1.80. Found: C, 30.86; H, 2.09%. **3**: IR (ν(CO), hexane) 2097s, 2066m, 2051m, 2032vs, 2017m and 2006s cm^{-1}; ^1H NMR (toluene) δ −18.68 (s, OsHOs) (Calc. for $C_{22}H_{11}O_{10}Os_3Sb$: C, 23.43; H, 0.98. Found: C, 23.62; H, 1.24%).

In CH_2Cl_2. To a solution of $[Et_4N][Os_3(\mu\text{-}H)(CO)_{10}(\mu\text{-}CO)]$ (100 mg, 0.099 mmol) in dichloromethane (20 mL) was added Ph_2SbCl (60 mg, 0.193 mmol). The reaction mixture was allowed to stir overnight. TLC separation using CH_2Cl_2–hexane (20:80, v/v) as eluent gave cluster **3** as the only major compound (34 mg, 30%).

Table 2 Crystal and refinement data for $[Os_3(\mu\text{-}SbPh_2)_2(CO)_{10}]$ **2a**, $[Os_3(\mu\text{-}H)(\mu\text{-}SbPh_2)(CO)_{10}]_2$ **3** and $[Os_3(\mu\text{-}SbPh_2)(\mu\text{-}PPh_2)(CO)_{10}]$ **4**

	2a	**3**	**4**
Formula	$C_{34}H_{20}O_{10}Os_3Sb_2\cdot\frac{1}{2}C_6H_{14}$	$C_{44}H_{22}O_{20}Os_6Sb_2\cdot1.5C_6H_{14}$	$C_{34}H_{20}O_{10}Os_3PSb\cdot\frac{1}{2}CH_2Cl_2$
M	1445.69	2384.57	1354.28
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2/c$	$P2_1/c$
a/Å	13.3803(2)	14.8356(4)	12.6310(1)
b/Å	17.9495(2)	11.8116(3)	18.0529(2)
c/Å	17.6996(1)	19.4734(4)	17.1885(1)
β/°	98.097(1)	101.587(1)	94.469(1)
V/Å^3	4208.53(8)	3342.82(14)	3907.45(5)
Z	4	2	4
μ(Mo-Kα)/mm^{-1}	10.342	12.213	10.569
Reflections collected	26494	19021	25323
Independent reflections	10238 (R_{int} = 0.0533)	7128 (R_{int} = 0.0304)	9701 (R_{int} = 0.0420)
Data/restraints/parameters	10238/0/341	7128/8/358	9701/1/453
Final $R1$, $wR2$ indices [$I > 2\sigma(I)$]	0.0576, 0.1212	0.0323, 0.0704	0.0462, 0.0857
(all data)	0.1125, 0.149	0.0506, 0.0808	0.0809, 0.0983

Reaction of compound 1 and Ph_2SbCl

An excess of Ph_2SbCl (31 mg, 0.10 mmol) and compound **1** (60 mg, 0.0522 mmol) were stirred in THF at room temperature until **1** had been consumed (by IR spectroscopy, ≈1 d). Removal of the solvent *in vacuo*, followed by thin-layer chromatographic separation with CH_2Cl_2–hexane (20:80, v/v) as the eluent, gave **2a** as a yellow band (47.6 mg, 65%).

A similar procedure was used to prepare $[Os_3(\mu\text{-}SbPh_2)\{\mu\text{-}Sb(C_6H_4Me\text{-}p)_2\}(CO)_{10}]$, **2b**, in 70% yield (56 mg) from compound **1** (63 mg, 0.055 mmol) and an excess of $(p\text{-}MeC_6H_4)_2SbCl$ (33 mg, 0.097 mmol), and $[Os_3(\mu\text{-}SbPh_2)(\mu\text{-}PPh_2)(CO)_{10}]$, **4**, in 85% yield (49 mg) from **1** (50 mg, 0.044 mmol) and an excess of Ph_2PCl (3–4 drops). **2b**: IR (ν(CO), hexane) 2101w, 2054mw, 2025s, 2000w, 1982mw, 1971vw and 1958mw cm^{-1} (Calc. for $C_{36}H_{24}O_{10}Os_3Sb_2$: C, 30.22; H, 1.69. Found: C, 30.53; H, 1.66%). **4**: IR (ν(CO), hexane) 2104w, 2062mw, 2027s, 2000mw, 1992mw, 1967w and 1955m cm^{-1}; ^{31}P-{^{1}H} NMR δ 46.44s (Calc. for $C_{34}H_{20}O_{10}Os_3PSb$: C, 31.12; H, 1.52. Found: C, 30.97; H, 1.98%).

Crystal structure determinations

Crystal data and structure refinement details are given in Table 2. All organic hydrogen atoms were placed in calculated positions and allowed to ride on the attached carbon atoms; their isotropic thermal parameters were given a value 1.5 times that of the attached carbon. For compound **3** the metal hydride was placed in a calculated position at 1.76 Å from Os(1) and Os(2) and *trans* to Os(3); the isotropic thermal parameter was fixed at 0.08 Å^2 and the Os–H distances were also fixed at 1.76 Å. All non-hydrogen atoms (except those of the solvent molecules) were given anisotropic displacement parameters in the final refinement.

All three compounds showed the presence of solvent molecules in the crystals, confirmed by ^{1}H NMR. Compound **2a** has half a molecule of hexane which was modelled as disordered about an inversion centre; the carbon atoms were given isotropic thermal parameters. Compound **3** has one and a half molecule of hexane modelled as one half molecule and a full molecule with two disordered sites; the carbon atoms were given a fixed isotropic thermal parameter of 0.15 Å^2. Appropriate restraints were placed on the geometries of the disordered solvent molecules. There was a half molecule of CH_2Cl_2 in compound **4** which was refined isotropically; the thermal parameters for the chlorine atoms were constrained to be equal.

CCDC reference number 186/2208.

See http://www.rsc.org/suppdata/dt/b0/b006669i/ for crystallographic files in .cif format.

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