Reactions of 1,8-bis(diphenylphosphino)naphthalene with Os$_3$(CO)$_{12}$: C–H and C–P bond cleavage reactions

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Abstract

Reactions of Os$_3$(CO)$_{12}$ with 1,8-bis(diphenylphosphino)naphthalene (dppn) are described. Crystallographically characterised complexes isolated from a reaction carried out in refluxing toluene are Os$_3$(μ-H)$_2$\{μ-PPh$_2$(nap)PPh(C$_6$H$_4$)\}$_2$(CO)$_6$ (**1**), Os$_3$(μ-H)\{μ$_3$-PPh$_2$(nap)PPh(C$_6$H$_4$)\}(CO)$_8$ (**2**) and Os$_2$(μ-PPh$_2$)\{μ-PPh$_2$(nap)\}(CO)$_5$ (**3**) (nap = 1,8-C$_{10}$H$_6$), while at r.t. in the presence of ONMe$_3$, only Os$_3$(CO)$_{11}$\{PPh$_2$(1-C$_{10}$H$_7$)\} (**4**) was isolated. While **1** and **2** contain ligands formed by metallation of a Ph group of dppn, as found also in complexes obtained from dppn and Ru$_3$(CO)$_{12}$, ligands in **3** and **4** are formed by cleavage of a P-nap bond, not found in the Ru series.
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1. Introduction

The phosphorus analogue of 1,8-bis(dimethyla-mino)naphthalene ("proton sponge®"), 1,8-bis(diphenylphosphino)naphthalene (dppn), is a highly basic bis(tertiary phosphine). Contacts between the phenyl groups on the phosphorus atoms result in considerable distortion of the free ligand, so it is not surprising that its metal complex chemistry has attracted much attention since the first report of this unusual phosphine [1]. The two phosphorus atoms are 3.05 Å apart, much shorter than the sum of the van der Waals radii (3.80 Å. Consequently, rigid chelate complexes are formed with most metal centres. Examples containing molybdenum [2], palladium [1–3], platinum [1,2] and gold [4–6] have been described. Platinum(II) complexes containing 1,8-(PRR')$_2$-naphthalenes (RR' = Me$_2$, Me(C$_6$F$_5$), Cy$_2$, Bu^{t}Ph) have also been made [7]. Our interest in the coordination and subsequent bond-cleavage reactions of bidentate phosphines on metal clusters instigated a survey of the reactions of dppn with trinuclear Group 8

metal carbonyl clusters. Following an associated account of the reactions between dppn and Ru$_3$(CO)$_{10}$(L)$_2$ (L = CO, L$_2$ = dppm) [8], this paper describes some complexes formed in reactions of dppn with Os$_3$(CO)$_{12}$.

2. Results and discussion

The reaction between Os$_3$(CO)$_{12}$ and dppn was carried out in refluxing toluene for 24 h. Several products were separated and purified by preparative t.l.c. and characterised initially by electrospray mass spectrometry (ES-MS) and single-crystal X-ray diffraction studies. Plots of the three of the complexes are given in Figs. 1–3 and selected bond parameters are listed in Table 1.

An orange band (R_f 0.36) afforded Os$_3$(μ-H)$_2$\{μ-PPh$_2$(nap)PPh(C$_6$H$_4$)\}$_2$(CO)$_6$, (**1**; nap = 1,8-C$_{10}$H$_6$) (Fig. 1), which has a crystallographic two-fold axis passing through Os(2) and the centre of the Os(1,1') vector. It contains two cyclometallated dppn ligands which bridge the Os(1,1')–Os(2) vectors. Atoms P(1) and P(2) chelate Os(1) [Os(1)–P(1,2) 2.378(6), 2.316(8) Å], while Os(2) is attached to C(112,112') of the metallated C$_6$H$_4$ rings [Os(2)–C(112) 2.19(2) Å]. Each metal atom

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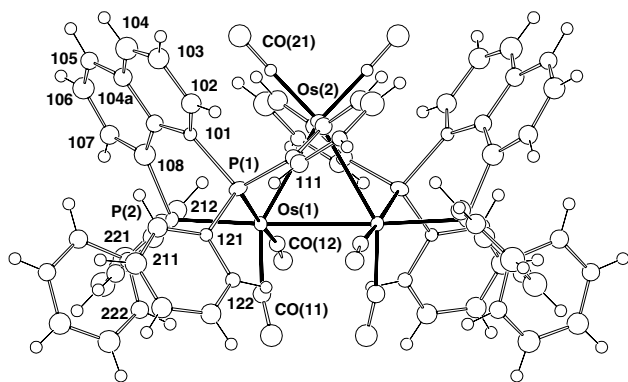
Fig. 1. Molecular projection of $Os_3(\mu\text{-H})\{\mu_3PPh_2(nap)PPh(C_6H_4)\}_2$-$(CO)_6$ (**1**), normal to the Os_3 plane. Core hydrogen atoms not refined.
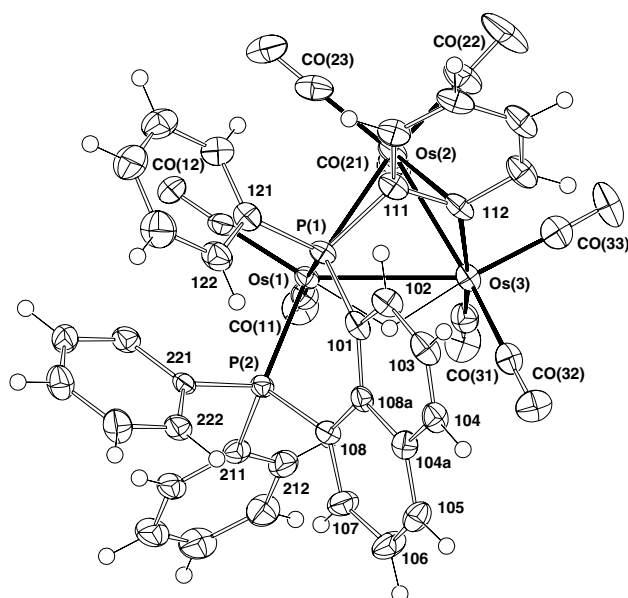
Fig. 2. Molecular projection of $Os_3(\mu H)\{\mu_3PPh_2(nap)PPh(C_6H_4)\}$-$(CO)_8$ (**2**), normal to the Os_3 plane.

Fig. 3. Molecular projection of $Os_2(\mu\text{-}PPh_2)\{\mu\text{-}PPh_2(nap)\}(CO)_5$ (**3**).

Table 1
Selected bond distances (Å) and angles (°)

Compound	**1**	**2**	**3**
Os(1)–Os(2)	3.040(2)	2.8567(4)	2.8413(4)
Os(1)–Os(3)	3.004(2) Os(1′)	3.0251(4)	
Os(2)–Os(3)		2.7568(4)	
Os(1)–P(1)	2.378(6)	2.332(1)	2.394(2)
Os(1)–P(2)	2.316(8)	2.329(1)	
Os(1)–C(101)			2.369(7)
Os(1)–C(102)			2.362(8)
Os(2)–C(101)			2.120(9)
Os(2)–C(111)		2.419(5)	
Os(2)–C(112)	2.19(2)	2.341(5)	
Os(3)–C(112)		2.152(5)	
Os(1)–H		1.82(7)	
Os(3)–H		1.76(7)	
P(1)–C(101)	1.87(3)	1.845(5)	
P(1)–C(111)	1.85(3)	1.817(6)	1.819(8)
P(1)–C(121)	1.87(2)	1.835(6)	1.834(8)
P(2)–C(108)	1.83(3)	1.829(5)	1.810(10)
P(2)–C(211)	1.82(3)	1.835(5)	1.836(8)
P(2)–C(221)	1.85(3)	1.827(5)	1.821(11)
C(111)–C(112)	1.39(4)	1.443(8)	

carries two terminal CO groups. Two cluster-bonded hydrogens were detected in the ^{1}H NMR spectrum (δ −12.39) as a double doublet of doublets, arising from coupling to phosphorus in the ligand, but these could not be located in the X-ray study. However, the long Os–Os separations [Os(1,1′)–Os(2) 3.004, 3.040(2) Å] are consistent with the presence of μ-H atoms. The cluster valence electron (c.v.e.) count is 48.

Complex **2** was isolated from an orange band (R_f 0.44) and shown to be $Os_3(\mu\text{-H})\{\mu_3\text{-}PPh_2(nap)PPh\text{-}(C_6H_4)\}(CO)_8$ (Fig. 2). The complex is isomorphous and isostructural with the ruthenium derivative described earlier [8]. The bis(phosphine) ligand chelates one Os atom [Os(1)–P(1,2) 2.332, 2.329(1) Å] while an *ortho* C–H bond of one of the phenyl groups has added to Os(3) [Os(3)–C(112) 2.152(5) Å]. This brings this C_6H_4 group within η^2-bonding distance of Os(2) [Os(2)–
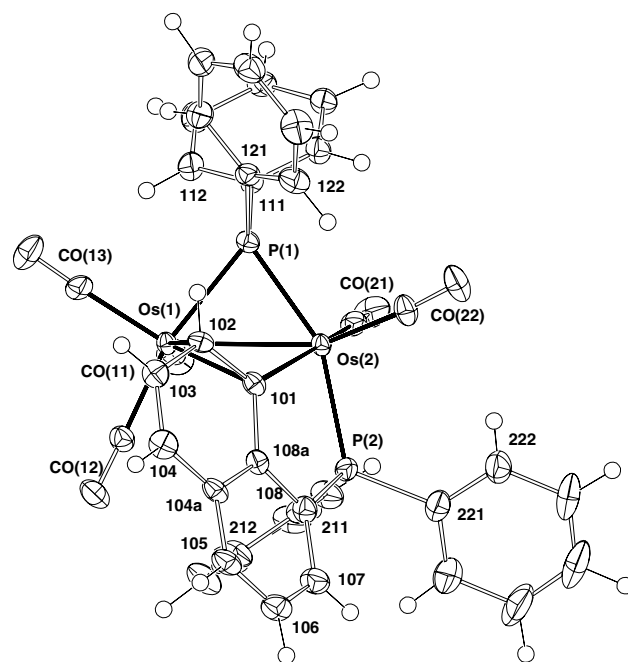
C(111, 112) 2.419, 2.341(5) Å], while the H atom, located and refined, bridges the Os(1)–Os(3) vector. Eight CO groups [three each on Os(2,3), two on Os(1)] complete the coordination, the cluster as a whole having 48 cluster valence electrons (c.v.e.), as expected for an M_3 cluster with three M–M bonds. In the ruthenium analogue [8], corresponding distances involving the metal atoms are 0.03–0.05 Å shorter, further confirming the similar atomic radii for these two elements in cluster carbonyl complexes of this type [9].

The fastest running complex (R_f 0.53) was pale yellow binuclear $Os_2(\mu\text{-}PPh_2)\{\mu\text{-}PPh_2(nap)\}(CO)_5$ (3). As shown in Fig. 3, an $Os(CO)_3$ group is linked to an $Os(CO)_2$ fragment by an Os–Os bond [2.8413(4) Å] which is bridged by a PPh_2 group [Os(1,2)–P(1) 2.394, 2.326(2) Å] and the $PPh_2(nap)$ fragment. The latter is attached to Os(1) by an η^2 interaction with C(101)–C(102) of the naphthyl group [Os(1)–C(101,102) 2.369, 2.362(8) Å] and to Os(2) by atom P(2) and a σ-bond to C(101) [Os(2)–P(2) 2.348(2); Os(2)–C(101) 2.120(9) Å]. To our knowledge, this is the first example of a μ-$\eta^1,P : \eta^2\text{-}PR_2(nap)$ fragment which has been structurally characterised.

The reaction between $Os_3(CO)_{12}$ and dppn was also run at r.t. in thf in the presence of $ONMe_3$ (tmno). After 1 h, spot t.l.c. showed that many products had formed, but only one of these has been identified crystallographically, as yellow $Os_3(CO)_{11}\{PPh_2(1\text{-}C_{10}H_7)\}$ (4). This complex is a conventional mono-substitution product of $Os_3(CO)_{12}$ in which the ligand is derived from dppn by replacement of a PPh_2 group by H, i.e., diphenyl(1-naphthyl)phosphine. As can be seen from Fig. 4, the molecular structure is similar to that of many other $Os_3(CO)_{11}(PR_3)$ complexes, with Os–Os bond lengths between 2.8862 and 2.9070(3) Å [the longest is Os(1)–Os(2), which shows the usual *cis* lengthening found in this type of complex]. The tertiary phosphine ligand occupies an equatorial site on Os(1) [Os(1)–P 2.378(1) Å], with P–C(aryl) bond lengths ranging between 1.829(4) and 1.842(6) Å.

In the reactions of dppn with $Ru_3(CO)_{12}$, ready cleavage of aryl C–H and Ph–P bonds occurred to give complexes containing $PPh_2(nap)PPh(C_6H_4)$, PPh_2-(nap)PPh, and $PPh_2P(nap)$ ligands; in two examples, cluster-bound C_6H_4 ligands, derived from one of the P–Ph groups, were also present [8]. In all cases, and also that of $Ru_3(\mu\text{-}dppm)(CO)_8(dppn)$, the P(nap)P fragment chelates one ruthenium atom, but no example of cleavage of the P-nap bond was found. The osmium complexes described above contain $PPh_2(nap)PPh(C_6H_4)$, $PPh_2(nap)$ and PPh_2 ligands, thus showing an increased tendency for cleavage of the nap-P bond. In 1, two ligands derived from dppn are attached to the Os_3 cluster. During one reaction, transfer of an H atom to the $PPh_2(nap)$ fragment allows formation of the tertiary phosphine $PPh_2(1\text{-}C_{10}H_7)$.

3. Conclusions

The close proximity of the two phosphorus donor atoms in dppn results in characteristic chelating behaviour towards transition metals, further exemplified in its reactions with trinuclear metal carbonyl clusters $M_3(CO)_{12}$ (M = Ru [8], Os). The present work has shown that C–H bond cleavage occurs with one of the Ph groups to give the ligand $PPh_2(nap)PPh(C_6H_4)$, found in two complexes. In 1, two of the metallated ligands bridge two of the three Os–Os vectors, there being no further bonding of the cluster to the C_6H_4 groups. In 2, coordination of this ligand to all three Os atoms occurs by chelation of one Os by the two P atoms, and metallation of one Ph group by a second Os atom, while the third Os atom has an η^2 interaction with this C_6H_4 group. In 3, fragmentation of the Os_3 cluster has occurred, to give an Os_2 complex in which the Os–Os bond is bridged by PPh_2 and $PPh_2(nap)$ ligands. Under mild conditions, the cluster aids degradation of the dppn to $PPh_2(1\text{-}C_{10}H_7)$, which is found in 4. Thus in contrast to the Ru_3 system, cleavage of the P-nap bond has occurred in two of these products.

4. Experimental

4.1. General experimental conditions

All reactions were carried out under dry, high purity nitrogen using standard Schlenk techniques. Common solvents were dried, distilled under argon and degassed before use.

4.2. Instrumentation

Infrared spectra were obtained on a Bruker IFS28 FT-IR spectrometer. Spectra in CH_2Cl_2 were obtained using a 0.5 mm path-length solution cell with NaCl windows. ^1H NMR spectra were recorded on Bruker AM300WB or ACP300 instruments at 300.13 MHz. Samples were dissolved in $CDCl_3$ or CD_2Cl_2 contained in 5 mm sample
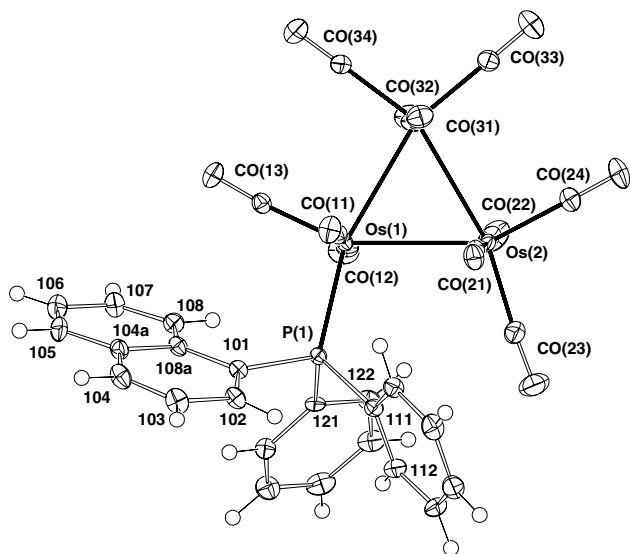

Fig. 4. Molecular projection of $Os_3(CO)_{11}\{PPh_2(1\text{-}C_{10}H_7)\}$ (4), normal to the Os_3 plane. Selected bond parameters: Os(1)–Os(2) 2.9070(3), Os(1)–Os(3) 2.8878(2), Os(2)–Os(3) 2.8862(3), Os(1)–P(1) 2.378(1), P(1)–C(101) 1.836(4), P(1)–C(111) 1.842(6), P(1)–C(121) 1.829(4) Å; Os(2)–Os(1)–P(1) 103.23(3)°; Os(3)–Os(1)–P(1) 162.64(3)°.

tubes. Chemical shifts are given in ppm relative to internal tetramethylsilane for ^{1}H. ES-MS were measured with a VG Platform 2, solutions in MeOH, containing NaOMe as an aid to ionisation [10], being directly infused into the instrument. Elemental analyses were performed by CMAS, Melbourne, Australia.

4.3. Reagent

The ligand dppn [11] was prepared by the cited method.

4.4. Reaction of $Os_3(CO)_{12}$ with dppn in refluxing toluene

A mixture of $Os_3(CO)_{12}$ (113 mg, 0.125 mmol) and dppn (62 mg, 0.12 mmol) was heated in refluxing toluene (20 ml) for 24 h. After evaporation to dryness, preparative t.l.c. (acetone/hexane 1/3) separated several products. Band 1 (orange, R_f 0.36) contained $Os_3(\mu\text{-}H)_2\{\mu\text{-}PPh_2(nap)PPh(C_6H_4)\}_2(CO)_6$ (**1**) (10.2 mg, 5%), obtained as orange crystals from CH_2Cl_2/hexane. Anal. Found: C, 51.61; H, 2.87. Calc. ($C_{74}H_{52}O_6Os_3P_4$): C, 51.33; H, 3.03%; M, 1732. IR (CH_2Cl_2): $\nu(CO)$ 2015m, 1992vs, 1979m, 1955 (sh), 1944 (sh), 1939w, 1918m cm^{-1}. ^{1}H NMR (CD_2Cl_2): δ −12.39 [2 × dd, 2H, J(HP) 19.2, 12.6 and 21.0, 12.4, 2 × OsH], 5.87–8.11 (m, 50H, Ph + C$_6$H$_4$ + nap). ES-MS (positive ion, MeOH, m/z): 1732, M$^+$; (with added NaOMe in MeOH, m/z): 1755, [M + Na]$^+$. Band 2 (orange, R_f 0.44) contained $Os_3(\mu\text{-}H)\{\mu_3\text{-}PPh_2(nap)PPh(C_6H_4)\}(CO)_8 \cdot CH_2Cl_2$ (**2**) (30 mg, 19%) obtained as crystals from CH_2Cl_2/hexane. Anal. Found: C, 39.11; H, 2.07%. Calc. ($C_{42}H_{26}O_8Os_3P_2$): C,

39.07; H, 2.03%; M, 1292. IR (CH_2Cl_2): $\nu(CO)$ 2062m, 2024vs, 1999m, 1974m (br), 1956w, 1935 (sh), 1919 (sh) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ −17.32 [dd, 1H, J(HP) 14.4, 12.9, OsH], 6.31–8.05 (m, 25H, Ph + C$_6$H$_4$ + nap). ES-MS (positive ion, MeOH, m/z): 1292, M$^+$; (negative ion, MeOH + NaOMe, m/z): 1323, [M + OMe]$^-$, 1291, [M–H]$^-$. Band 3 (yellow, R_f 0.53) contained several compounds, fractional crystallisation giving pale yellow crystals (from CH_2Cl_2/MeOH) of $Os_2(\mu\text{-}PPh_2)\{\mu\text{-}PPh_2(nap)\}(CO)_5$ (**3**). Anal. Calc. ($C_{39}H_{26}O_5Os_2P_2$): M, 1018. IR (CH_2Cl_2): $\nu(CO)$ 2076s, 2039vs, 2035 (sh), 1991vs, 1981 (sh), 1972 (sh), 1924w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 6.16–8.22 (m, 26H, Ph + nap). ES-MS (positive ion, MeOH, m/z): 2057, [2M + Na]$^+$; 1041, [M + Na]$^+$; 1018, M$^+$. The small amount of compound finally obtained precluded getting accurate microanalytical data. Band 4 (yellow, R_f 0.83) contained $Os_3(CO)_{12}$ (31.7 mg, 28%), identified by IR. Other bands contained trace amounts of unidentified compounds.

4.5. Reaction of $Os_3(CO)_{12}$ with dppn at r.t.

Solid Me$_3$NO (9 mg, 0.1 mmol) was added to a solution of $Os_3(CO)_{12}$ (46 mg, 0.051 mmol) and dppn (25 mg, 0.050 mmol) in thf (15 ml) and the mixture was stirred for 2.5 h at r.t. Preparative t.l.c. (acetone/hexane 1/2) separated several bands. Bands 1 (yellow, R_f 0.38; 2.3 mg) and 2 (orange, R_f 0.43; 7.8 mg) are presently unidentified; the latter has $\nu(CO)$ at 2108vw, 2090w, 2073vs, 2049w, 2033 vs, 2004 (sh), 1990vs (br), 1972m, 1956m, 1927w cm^{-1} (CH_2Cl_2), ES-MS (negative ion, MeOH + NaOMe, m/z) 1309. Band 3 (green, R_f 0.66;

Table 2
Crystal data and refinement details

Compound	**1**	**2**	**3**	**4**
Formula	$C_{74}H_{52}O_6Os_3P_4 \cdot 1.36CH_2Cl_2$	$C_{42}H_{26}O_8Os_3P_2 \cdot CH_2Cl_2$	$C_{39}H_{26}O_5Os_2P_2$	$C_{33}H_{17}O_{11}Os_3P \cdot 0.5CH_2Cl_2$
MW	1847.7	1376.1	1017.0	1233.5
Crystal system	Monoclinic	Triclinic	Monoclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$	$P2_1$	$P\bar{1}$
a (Å)	16.723(9)	10.390(2)	9.9014(6)	12.2716(8)
b (Å)	20.114(10)	14.040(2)	17.402(1)	12.3073(8)
c (Å)	20.622(10)	15.160(2)	10.7700(7)	12.3651(8)
α (°)		85.801(4)		87.306(2)
β (°)	102.61(2)	85.691(4)	109.371(1)	79.418(2)
γ (°)		84.730(4)		64.345(2)
V (Å^3)	6769	2191	1751	1654
Z	4	2	2	2
D_c (g cm^{-3})	1.81$_1$	2.08$_6$	1.92$_9$	2.47$_7$
μ (cm^{-1})	5.9	8.9	7.4	11.7
Crystal size (mm)	$0.27 \times 0.17 \times 0.07$	$0.20 \times 0.17 \times 0.11$	$0.11 \times 0.07 \times 0.06$	$0.18 \times 0.15 \times 0.13$
$T_{min/max}$	0.66	0.59	0.72	0.54
$2\theta_{max}$ (°)	50	64	75	75
N_{tot}	8491	40 850	45 891	34 480
$N(R_{int})$	4953(0.11)	15 469(0.042)	8955(0.028)	17 029(0.041)
N_o	1882	9717	8144	12 932
R	0.052	0.031	0.033	0.037
R_w	0.067	0.042	0.041	0.039

3.2 mg) was crystallised (CH_2Cl_2/MeOH) to give yellow crystals of $Os_3(CO)_{11}$\{PPh_2(nap)\}(**4**). IR (CH_2Cl_2): ν(CO) 2108w, 2055vs, 2032m, 2018vs, 1989w, 1978w, 1952w cm^{-1}. ^{1}H NMR (CDCl$_3$): δ 7.05–7.97 (m, 17H, Ph + $C_{10}H_7$). ES-MS (negative ion, MeOH + NaOMe, m/z): 1223, [M + OMe]$^-$. Band 4 (R_f 0.91) contained $Os_3(CO)_{12}$ (1.2 mg, 3%).

4.6. Structure determinations

Full spheres of diffraction data were measured at ca. 153 K using a Bruker AXS CCD area-detector instrument. $N_{tot(al)}$ reflections were merged to N unique (R_{int} cited) after "empirical"/multiscan absorption correction (proprietary software), N_o with $F > 4\sigma(F)$ being used in the full matrix least squares refinements. All data were measured using monochromatic Mo Kα radiation, $\lambda = 0.71073$ Å. Anisotropic displacement parameter forms were refined for the non-hydrogen atoms, (x, y, z, U_{iso})$_H$ being constrained at estimated values. Conventional residuals R, R_w on $|F|$ are quoted [weights: $(\sigma_2(F) + 0.000n_w F^2)^{-1}$]. Neutral atom complex scattering factors were used; computation used the XTAL 3.7 program system [12]. Pertinent results are given in the Figures (which show non-hydrogen atoms with 50% probability amplitude displacement ellipsoids and hydrogen atoms with arbitrary radii of 0.1 Å) and Table 2.

4.7. Variata

1. Fracture of the most auspicious available specimen during data acquisition resulted in only a hemisphere of useful data, already limited in scope as a consequence of partial desolvation (solvent occupancy refining to 0.68) and supportive of anisotropic displacement parameter refinement for Os, P, Cl only. Core hydrogen atoms, postulated from the NMR, were not located.

2. Core hydride atoms were located and refined in (x, y, z, U_{iso}). Difference map residues were modelled as equal disordered components of CH_2Cl_2 (site occupancies: 0.5), with constrained geometries.

3. Friedel data were preserved distinct, x_{obs} refining to 0.007(9).

5. Supplementary material

Full details of the structure determinations of complexes **1**–**4** (except structure factors) have been deposited with the Cambridge Crystallographic Data Centre as CCDC 226397–226400, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).
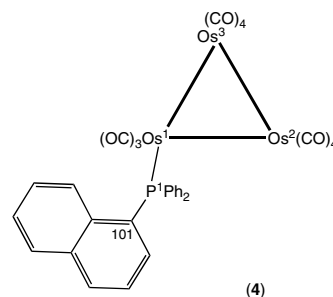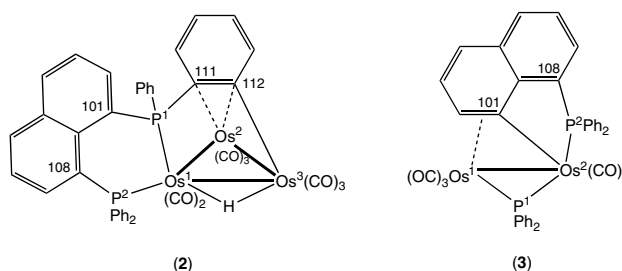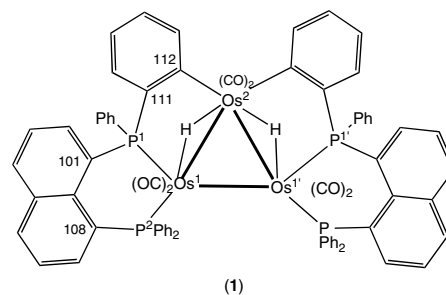
References

[1] R.D. Jackson, S. James, A.G. Orpen, P.G. Pringle, J. Organomet. Chem. 458 (1993) C3.
[2] A. Karaçar, M. Freytag, H. Thönnessen, J. Omelanczuk, P.G. Jones, R. Bartsch, R. Schmutzler, Z. Anorg. Allg. Chem. 626 (2000) 2361.
[3] S.L. James, A.G. Orpen, P.G. Pringle, J. Organomet. Chem. 525 (1996) 299.
[4] V.W.-W. Yam, C.-L. Chan, S.W.-K. Choi, K.M.-C. Wong, E.C.-C. Chen, S.-C. Yu, P.-K. Ng, W.-K. Chan, K.-K. Cheung, Chem. Commun. (2000) 53.
[5] V.W.-W. Yam, S.W.-K. Choi, J. Chem. Soc., Dalton Trans. (1996) 4227.
[6] V.W.-W. Yam, C.-K. Li, C.-L. Chan, K.-K. Cheung, Inorg. Chem. 40 (2001) 7054.

[7] A. Karaçar, M. Freytag, P.G. Jones, R. Bartsch, R. Schmutzler, Z. Anorg. Allg. Chem. 628 (2002) 533.

[8] M.I. Bruce, P.A. Humphrey, S. Okucu, R. Schmutzler, B.W. Skelton, A.H. White, Inorg. Chim. Acta 357 (2004) 1805.

[9] See, for example M.I. Bruce, M.L. Williams, J.M. Patrick, A.H. White, Aust. J. Chem. 36 (1983) 1353;
A similar conclusion can be extracted from the data summarised in: A.G. Orpen, L. Brammer, F.H. Allen, O. Kennard, D.G. Watson, R. Taylor, J. Chem. Soc., Dalton Trans. (1989) S1.

[10] W. Henderson, J.S. McIndoe, B.K. Nicholson, P.J. Dyson, J. Chem. Soc., Dalton Trans. (1998) 519.

[11] (a) A. Karaçar, H. Thönnessen, P.G. Jones, R. Bartsch, R. Schmutzler, Heteroat. Chem. 8 (1997) 539;
(b) J. van Soolingen, R.-J. de Lang, R. den Besten, P.A.A. Klusener, N. Veldman, A.L. Spek, L. Brandsma, Synthetic Commun. 25 (1995) 1741.

[12] S.R. Hall, D.J. du Boulay, R. Olthof-Hazekamp (Eds.), The XTAL 3.7 System, University of Western Australia, Perth, 2000.