THE REACTIONS OF (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_9$ WITH GROUP V DONOR LIGANDS. SPECTROSCOPIC CHARACTERIZATION OF THE DISUBSTITUTED DERIVATIVES, AND CRYSTAL STRUCTURE OF (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_7$(PMe$_2$Ph)$_2$

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Summary

The complex (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_9$ (**1**) reacts with phosphines in the presence of Me$_3$NO to give monosubstituted derivatives, the main products, along with smaller amounts of disubstituted derivatives. The IR and NMR spectroscopic characterizations of two representative disubstituted derivatives are described.

The crystal structure of (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_7$(PMe$_2$Ph)$_2$ has been determined by X-ray diffraction. The crystals are monoclinic, space group $P2_1/a$ with $Z = 4$ in a unit cell of dimensions a 18.198(8), b 20.865(8), c 8.656(5) Å, β 99.67(2)°. The structure was solved by direct and Fourier methods and refined by full-matrix least-squares to $R = 0.061$ for 3139 observed reflections. The structure can be regarded as derived from that of **1** (which has a tetrahedral NiOs$_3$ core with three hydride hydrogen atoms bridging the Os–Os edges, a cyclopentadienyl group coordinated to the Ni atom, and nine terminal carbonyls bound to the Os atoms, three for each Os atom) by replacing two axial carbonyls by two PMe$_2$Ph ligands.

Introduction

The complex (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_9$ (**1**) [1,2] is very stable, and shows interesting properties as an hydrogenation catalyst under homogeneous [3] and heterogeneous [4] conditions. It possesses a tetrahedral NiOs$_3$ core, with three hydrides bridging the Os–Os edges, the cyclopentadienyl group η^5-bound to Ni and three terminal CO's (two equatorial and one axial) on each osmium atom.

The replacement of these CO ligands by group V donor ligands takes place only with considerable difficulty [1]; however, this process is rapid in refluxing hydrocarbons in the presence of Me_3NO, and gives high yields of monosubstituted derivatives of general formula $(\eta^5-C_5H_5)NiOs_3(\mu-H)_3(CO)_8L$ (2), which have been characterized by IR and NMR spectroscopy [5,6].

In all but one [6] of these complexes, L is coordinated to one osmium atom in the less hindered axial position, as suggested by the ^1H NMR patterns in the hydride region and confirmed by the X-ray structural determinations carried out on the derivatives with L = PPh_2H [6] and $PPh_2(C\equiv CPr^i)$ [7].

Secondary products from the above reactions are two sets of compounds, whose yields generally increase upon increasing either the concentration of the ligand or the reaction time. The first set comprises the disubstituted derivatives of general formula $(\eta^5-C_5H_5)NiOs_3(\mu-H)_3(CO)_7L_2$ (3); the second set consists of oils (4), not yet fully characterized, which are generally obtained in lower yields.

We report here the IR and NMR spectroscopic characterization of two representative disubstituted derivatives, those with L = $SbPh_3$ (3a) and L = PMe_2Ph (3b), and compare their spectra with those of the corresponding monosubstituted complexes 2a and 2b. A possible interpretation of the data for the oily product of type 4 obtained from PPh_2H is also presented.

The X-ray structure of 3b, which confirms the spectroscopic evidence, is also described.

Experimental

Materials

Complex 1 was obtained and purified as previously described [1]; the phosphines and the stibine were commercial products (Fluka) and were used without further purification. Anhydrous Me_3NO was obtained by sublimation in vacuo of commercial $Me_3NO \cdot 2H_2O$ (Fluka). The hydrocarbon solvents were distilled over sodium. Reactions were carried out under dry nitrogen.

Synthesis, purification and analysis of the products

The complexes 3 and 4 were obtained (along with complexes 2) by refluxing heptane solutions of 1 and the appropriate ligand in the presence of Me_3NO, under N_2 for 10–15 min. The resulting solution was evaporated in vacuo, and the residue was redissolved in $CHCl_3$ and the products separated by preparative TLC (Kieselgel, Merck; eluant ethyl ether 15% in light petroleum); complexes 3 and 4 move very slowly on the chromatographic plates, probably because of their slow solubility in the hydrocarbons. Several of the grey-green complexes 3 are oily materials difficult to crystallize; all the purple complexes 4 are oils at room temperature. The typical IR patterns, in the CO stretching region, of compounds 2–4 are shown in Fig. 1, together with that of the parent compound 1. They provide the most rapid means of identifying these species.

The characterization data for 3a ($SbPh_3$) and 3b (PMe_2Ph) are given in Table 1. Some efforts were made to characterize compounds 4, and, in the case of PPh_2H, we obtained the following data (which are not conclusive; see Discussion): C, 45.12; Os, 38.16%. IR: $\nu(CO)$(hexane) 2065s, 2024s(sh), 2008vs cm^{-1}. ^1H NMR ($CDCl_3$): δ, 8.89(s) and 7.11(s, 1H, PH) (^1J(PH) 480 Hz), 7.74–7.40 (m, 10H, Ph); ^{31}P NMR ($CDCl_3$) $\delta(H_3PO_4)$, 19.67(s).
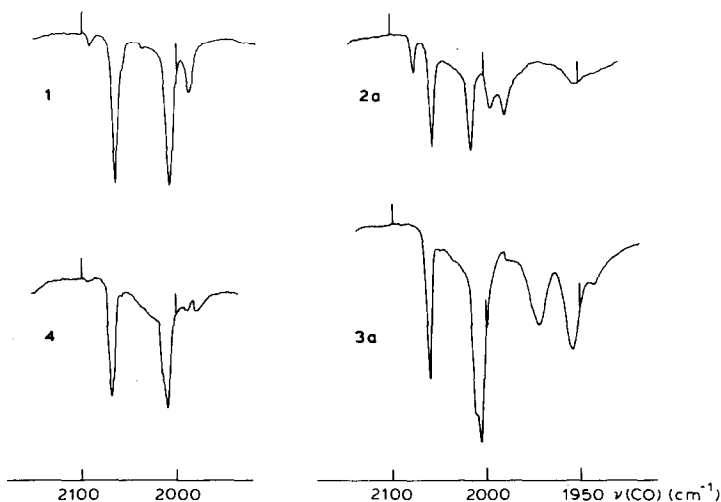
Fig. 1. IR spectra (hexane/chloroform) in the ν(CO) region for 1, 2a, 3a and 4 (PPh$_2$H).

All the complexes either decompose or do not volatalize in the mass spectrometer (Kratos MS 50, direct inlet system, EI technique, 70 eV). They were analyzed for C, H, Os, Ni, P or Sb (F. Pascher Laboratory, Bonn, W. Germany). The IR spectra were recorded with a Perkin–Elmer 580 B spectrometer, and the ^{1}H and ^{31}P NMR with a JEOL JNM GX 270 FT spectrometer.

X-Ray data collection, structure solution and refinement

Dark green-black crystals of 3b were obtained by keeping heptane/CHCl$_3$ solutions of the complex, at $-25°$C under nitrogen for some days; some decomposition was observed.

A prismatic crystal of approximate dimensions $0.14 \times 0.14 \times 0.37$ mm was selected and mounted on a Siemens AED single-crystal diffractometer. Unit cell parameters

TABLE 1

CHARACTERIZATION DATA FOR TWO COMPLEXES (η^5-C$_5$H$_5$)NiOs$_3$(μ-H)$_3$(CO)$_7$L$_2$ (3)

Complex	Analysis [a] (%)	ν(CO) [b] (cm^{-1})	^{1}H(δ) [c,d] (ppm)	^{31}P(δ) [c,e] (ppm)	J (Hz)
(SbPh$_3$) 3a dark grey-green crystals	Ni, 3.8(3.7) Os, 35.8(35.7) Sb, 15.8(15.2)	2061s, 2011vs(sh) 2005 vs, 1972m, 1954ms	7.40m(30H, Ph) 6.08s(5H, Cp) -16.59t(1H,μ-H) -17.17d(2H,μ-H)	–	1.8, ^2J(HH)
(PMe$_2$Ph) 3b dark grey-green crystals	C, 28.4(28.8) H, 2.8(2.6) Ni, 5.2(5.0) Os, 48.6(48.8) P, 5.2(5.3)	2057s, 2030vs(sh) 2024vs, 1966s, 1950s	7.34m(10H, Ph) 6.05s(5H, Cp) 1.97d, 1.95d(12H, Me) -16.41tt (1H, μ-H) -17.21 dd(2H, μ-H)	-14.5	9.8, ^2J(PH) [f] 10.4, ^2J(PH) [f] 2.0, ^2J(HH) [f] 9.2, ^2J(PH$_{Me}$)

[a] Calculated values are given in parentheses. [b] Hexane solution. [c] CDCl$_3$ solution. [d] SiMe$_4$ as internal reference; s = singlet, d = doublet, t = triplet, m = multiplet. [e] Positive chemical shifts are downfield relative to H$_3$PO$_4$ external. [f] See Fig. 3.

were obtained by least-squares refinement of the θ-values of 29 carefully centered reflections. Intensity data were collected at room temperature for θ in the range 3–25° ($\theta/2\theta$ scan) using niobium-filtered Mo-K_α radiation. Of 5736 independent measured reflections, 3139, with $I \geqslant 2\sigma(I)$, were used in the analysis. Corrections were applied for Lorentz, polarization, and absorption effects. (For the absorption correction the maximum and minimum transmission factor values were 1.3147 and 0.8160 [8].)

TABLE 2

FRACTIONAL ATOMIC COORDINATES FOR THE NON-HYDROGEN ATOMS ($\times 10^4$) (with e.s.d.'s in parentheses)

Atom	x	y	z
Os(1)	1857(1)	2588(1)	685(1)
Os(2)	3065(1)	2606(1)	3295(1)
Os(3)	1669(1)	2041(1)	3652(1)
Ni	2547(2)	1590(1)	1901(4)
P(1)	3389(3)	3586(3)	4515(8)
P(2)	1192(4)	3526(3)	−35(8)
O(1)	748(15)	1755(11)	−1362(31)
O(2)	2810(14)	2674(13)	−1794(28)
O(3)	4368(12)	2583(11)	1524(33)
O(4)	3946(13)	1784(11)	5877(28)
O(5)	2326(13)	1139(13)	6219(26)
O(6)	627(14)	2675(13)	5599(32)
O(7)	601(14)	1013(12)	2222(34)
C(1)	1164(16)	2073(15)	−564(36)
C(2)	2447(19)	2635(14)	−869(36)
C(3)	3844(15)	2624(14)	2193(38)
C(4)	3579(19)	2093(16)	4952(38)
C(5)	2078(20)	1493(16)	5231(44)
C(6)	1006(17)	2469(14)	4841(37)
C(7)	975(14)	1389(15)	2792(38)
C(8)	2629(17)	3942(15)	5336(37)
C(9)	4147(16)	3546(14)	6215(35)
C(10)	3715(14)	4229(12)	3390(31)
C(11)	3689(16)	4123(14)	1689(35)
C(12)	3998(17)	4608(15)	775(38)
C(13)	4276(18)	5171(16)	1578(40)
C(14)	4253(17)	5273(14)	3150(38)
C(15)	3993(17)	4800(15)	4082(37)
C(16)	216(14)	3429(13)	−1008(32)
C(17)	1561(14)	4038(13)	−1421(32)
C(18)	1060(14)	4067(12)	1519(31)
C(19)	676(13)	3830(11)	2703(28)
C(20)	530(17)	4248(15)	3954(39)
C(21)	799(21)	4881(19)	3961(46)
C(22)	1198(19)	5090(17)	2897(43)
C(23)	1350(16)	4710(14)	1666(37)
C(24)	2461(16)	889(14)	132(36)
C(25)	2373(16)	594(14)	1537(36)
C(26)	3027(18)	693(15)	2683(39)
C(27)	3485(18)	1024(16)	1865(39)
C(28)	3180(15)	1146(14)	313(34)

Crystal data. $C_{28}H_{30}NiO_7Os_3P_2$, $M = 1169.80$, monoclinic, a 18.198(8), b 20.865(8), c 8.656(5) Å, β 99.67(2)°, V 3240(3) Å^3, Z 4, D_c 2.398 g cm^{-3}, $F(000)$ 2160, space group $P2_1/a$, μ(Mo-K_α) 124.60 cm^{-1}.

The structure was solved by direct and Fourier methods and by full-matrix least-squares using the SHELX system of computer programs [9] with initially isotropic and subsequently anisotropic thermal parameters for the Os, Ni, P, O and $C(1) \cdots C(7)$ atoms. The hydrogen atoms, except for the hydridic ones, were placed at their geometrically calculated positions and introduced in the final structure factor calculations. The atomic scattering factors used, corrected for the anomalous dispersion of the Os, Ni, P atoms, were taken from ref. 10. The function minimized in the least-squares calculations was $\Sigma w |\Delta F|^2$; unit weights were chosen in the first stages of the refinement, then weights were applied according to $w = k/[\sigma^2(F_0) + gF_0^2]$ with $k = 0.9043$ and $g = 0.0031$. Final R and R_w values were 0.061 and 0.072 respectively. Final atomic coordinates for the non-hydrogen atoms are given in Table 2. Coordinates for the hydrogen atoms, thermal parameters, and lists of observed and calculated structure factors may be obtained from the authors on request.

All calculations were performed on the CYBER 76 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale, Bologna and on the GOULD-SEL 32/37 computer of the Centro di Studio per la Strutturistica Diffrattometrica del C.N.R. (Parma), with financial support from the University of Parma.

Results and discussion

As stated out in the Introduction and in the Experimental, the reaction of **1** with Group V donor ligands produces monosubstituted complexes (**2**) [5,6], disubstituted complexes (**3**) and some other, not as yet unidentified, derivatives (**4**).

Two compounds from set **3**, were characterized by elemental analysis and IR and NMR spectroscopy (Table 1).

IR spectra

The mono- and the disubstituted derivatives **2a** and **3a** give similar IR patterns in the ν(CO) region (see Fig. 1). This indicates that **3a** probably possesses roughly the same local C_s symmetry as **2a** and hence the two phosphines must be attached, provided the system is rigid, either to the same osmium atom in the equatorial position (I), or to two different metals in the C_s-related equatorial sites (II), or to different metals in the axial positions (III) (Fig. 2). Since the axial position is the less hindered, and, except for that containing PPhH$_2$ [6], the monosubstituted compounds have the ligand in this position, it is easy to predict that the two ligands are arranged as in formula III of Fig. 2.

Another interesting feature of the spectra is the gradual shift toward lower frequencies of the barycentre of the bands, passing from **1**, to **2a**, and then to **3a**, with increasing degree of substitution.

NMR spectra

The ^{1}H NMR spectra of complexes **2** and **3** exhibit the typical AX$_2$ pattern, in the hydride region and provide powerful tools for the characterization of these species. The spectrum of the monosubstituted cluster with SbPh$_3$ (**2a**) consists of a
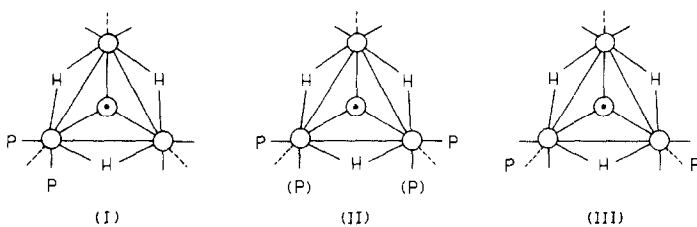
Fig. 2. Schemes of the possible isomers of compounds 3 (see discussion). ○ osmium atoms; ⊙ cyclopentadienylnickel fragments; ———— equatorial position; - - - - - - - axial position.

triplet ($-17.82t$, 1H, μ-H) * and a doublet ($-17.18d$, 2H, μ-H) * to lower field ($^2J(HH)$ 2.2 Hz), whereas, the corresponding disubstituted derivative (3a) presents a reverse pattern (Table 1) with a doublet (2H) and a triplet (1H) to lower field ($^2J(HH)$ 1.8 Hz). In the case of the complexes with phosphorus-containing ligands, further multiplicity is introduced by coupling of P with the adjacent hydrides ($^2J(PH)$ 9 ÷ 11 Hz).

In Fig. 3 the ^{1}H NMR patterns of 2b and 3b are compared, and in Table 1 the ^{1}H and ^{31}P NMR data for 3a and 3b are listed. Apparently disubstitution results in a low-field shift (about 1 ppm) of the hydride signals compared with those from the monosubstituted derivatives (the values for 2a are listed in ref. 6).

Finally the observed patterns are consistent with the proposed 1,2-axial substitution III of Fig. 2, and this was confirmed by an X-ray determination of the structure of 3b (see below).

The characterization data for a compound of type 4 (containing PPh_2H) given in the experimental part, do not permit conclusions to be reached about the structures of these kind of compounds. The absence of the (η^5-C_5H_5)Ni fragment, revealed by ^{1}H NMR and by elemental analysis, together with the presence of H–P signals, in the ^{1}H and ^{31}P NMR spectra, suggests a formulation such as $Os_3(CO)_8(PPh_2H)_4$ for this compound (calcd.: C, 43.7; Os, 37.0%). However this formulation seems not to be consistent with the simplicity of the IR and ^{31}P NMR spectra, since complexes of the type $Os_3(CO)_{12-x}L_x$ give more complex IR and NMR spectra [11,12].

Crystal structure of the complex 3b

A view of the complex 3b is shown in Fig. 4 together with the atom labelling system; selected bond distances and angles are given in Table 3. The structure of 3b consists of a tetrahedral cluster of three osmium atoms and one nickel atom, a cyclopentadienyl ligand is η^5-coordinated to the nickel atom, seven terminal carbonyl groups are bound to the osmium atoms (three, two in equatorial and one in axial positions, are attached to the unique Os(3), and two, in equatorial positions, are attached to Os(1) and Os(2)), and two PMe_2Ph ligands are axially bound to Os(1) and Os(2). Each Os–Os edge of the triangular cluster is bridged by a hydride ligand. The hydride hydrogens were not directly located in the ΔF map, but their presence and positions are clearly indicated by the NMR data.

The structure of 3b can be regarded as derived from that of 1 by replacing two

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* These data collected with the FT facility replace the earlier ones [5] obtained with a single scan.
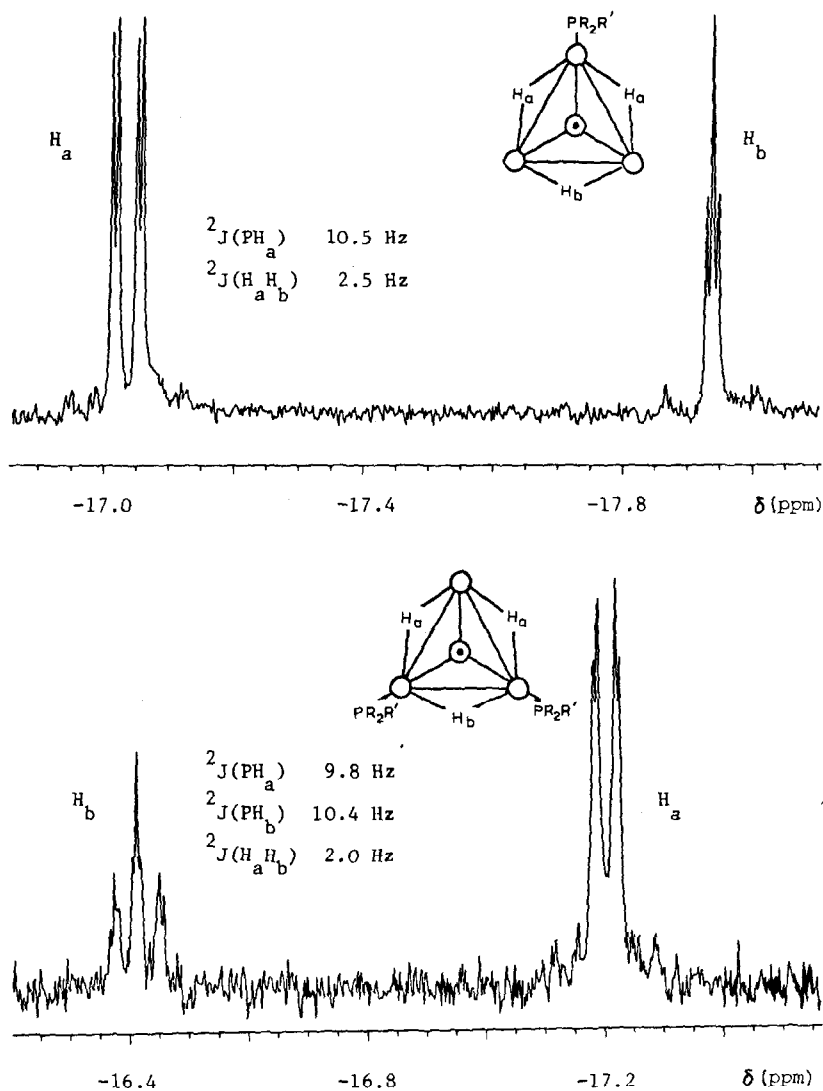
Fig. 3. ^{1}H NMR spectra of **2b** and **3b** in the hydride region.

axial carbonyls by two phosphine ligands. This substitution leaves the structure practically unaltered except that some carbonyls are slightly displaced because of the steric hindrance of the bulky PMe_2Ph group. This feature was previously observed for the substitution of one carbonyl in **1** with a PPh_2H ligand [6] or with $PPh_2(C\equiv CPr^i)$ [7] and of a hydride with the isolobal group $AuPPh_3$ [13]. The rigidity of the $NiOs_3$ core in substituted clusters is confirmed by comparison of the bond distances (see Table 4).

The axial positions of both substituents are noteworthy; the room temperature NMR spectra indicate that this situation is maintained in solution, other isomers probably being disfavoured for steric reasons. The apparent rigidity of **3b** in
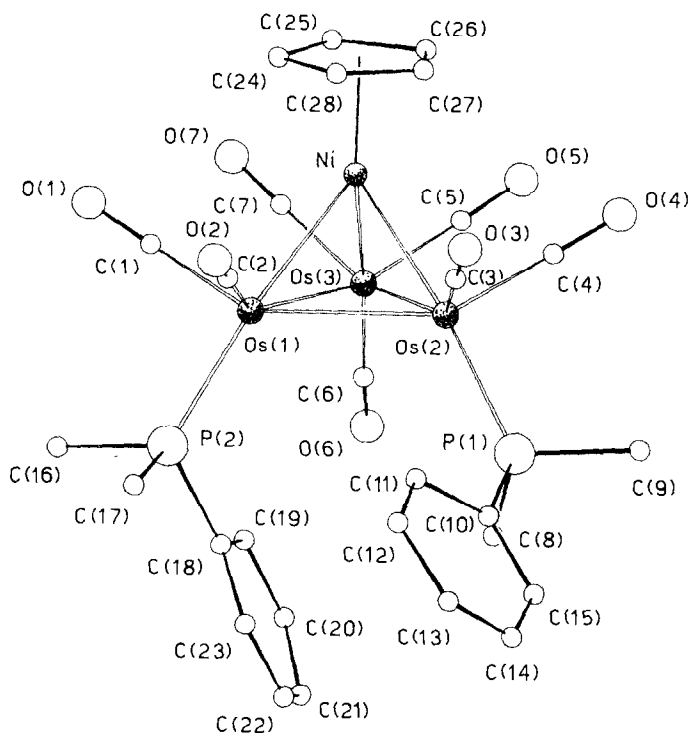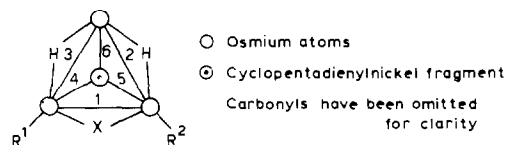
Fig. 4. View of the complex $(\eta^5\text{-}C_5H_5)NiOs_3(\mu\text{-}H)_3(CO)_7(PMe_2Ph)_2$ with the atom labelling system.

TABLE 3. SELECTED BOND DISTANCES (Å) AND ANGLES (°)

Os(1)–Os(2)	2.876(2)	Os(3)–C(7)	1.92(3)
Os(1)–Os(3)	2.883(2)	P(1)–C(8)	1.82(3)
Os(2)–Os(3)	2.864(3)	P(1)–C(9)	1.84(3)
Os(1)–Ni	2.564(3)	P(1)–C(10)	1.81(3)
Os(2)–Ni	2.541(3)	P(2)–C(16)	1.84(3)
Os(3)–Ni	2.559(4)	P(2)–C(17)	1.82(3)
Os(1)–P(2)	2.330(7)	P(2)–C(18)	1.80(3)
Os(2)–P(1)	2.331(7)	C(1)–O(1)	1.15(4)
Os(1)–C(1)	1.86(3)	C(2)–O(2)	1.12(4)
Os(1)–C(2)	1.86(3)	C(3)–O(3)	1.20(4)
Os(2)–C(3)	1.84(3)	C(4)–O(4)	1.15(4)
Os(2)–C(4)	1.90(3)	C(5)–O(5)	1.16(4)
Os(3)–C(5)	1.84(3)	C(6)–O(6)	1.11(4)
Os(3)–C(6)	1.93(3)	C(7)–O(7)	1.10(4)
Os(2)–Os(1)–Os(3)	59.6(1)	Os(2)–P(1)–C(9)	115 (1)
Os(1)–Os(2)–Os(3)	60.3(1)	Os(2)–P(1)–C(10)	119 (1)
Os(1)–Os(3)–Os(2)	60.0(1)	C(8)–P(1)–C(9)	103 (1)
Os(1)–Ni–Os(2)	68.6(1)	C(8)–P(1)–C(10)	104 (1)
Os(2)–Ni–Os(3)	68.3(1)	C(9)–P(1)–C(10)	101 (1)
Os(1)–Ni–Os(3)	68.5(1)	Os(1)–C(1)–O(1)	178 (3)
Os(1)–P(2)–C(16)	117 (1)	Os(1)–C(2)–O(2)	179 (3)
Os(1)–P(2)–C(17)	116 (1)	Os(2)–C(3)–O(3)	174 (3)
Os(1)–P(2)–C(18)	117 (1)	Os(2)–C(4)–O(4)	173 (3)
C(16)–P(2)–C(17)	101 (1)	Os(3)–C(5)–O(5)	179 (3)
C(16)–P(2)–C(18)	100 (1)	Os(3)–C(6)–O(6)	175 (3)
C(17)–P(2)–C(18)	104 (1)	Os(3)–C(7)–O(7)	176 (3)
Os(2)–P(1)–C(8)	113 (1)		

TABLE 4

COMPARISON OF THE LENGTHS OF THE METAL–METAL BONDS IN 1 AND IN ITS SUBSTITUTED DERIVATIVES

O Osmium atoms

⊙ Cyclopentadienylnickel fragment

Carbonyls have been omitted for clarity

Compound	1	2	3	4	5	6	Ref.
$R^1 = R^2 = CO$							
X = H (1) ^{a}	2.859(1)	2.874(1)	2.874(1)	2.569(2)	2.569(2)	2.578(2)	2a ^{b}
	2.873(1)	2.873(1)	2.873(1)	2.563(2)	2.563(2)	2.562(2)	
	2.869(2)	2.866(2)	2.866(2)	2.564(5)	2.564(5)	2.563(5)	2b
X = AuPh$_3$	2.863(2)	2.881(2)	2.785(2)	2.581(4)	2.569(5)	2.581(5)	13
$R^1 = CO, X = H$							
$R^2 = PPh_2H$	2.870(2)	2.859(2)	2.865(2)	2.562(3)	2.553(2)	2.555(3)	6
$PPh_2(C_2Pr^i)$	2.868(3)	2.855(3)	2.862(3)	2.555(3)	2.553(5)	2.542(4)	7 ^{b}
	2.863(3)	2.856(3)	2.870(3)	2.548(4)	2.552(5)	2.552(4)	
$PPh_2(C_2Pr^i)$- $\{Co_2(CO)_6\}$	2.883(3)	2.868(3)	2.875(3)	2.557(5)	2.560(6)	2.551(5)	7
$R^1 = R^2 = PMe_2Ph$							
X = H (3b)	2.876(2)	2.864(3)	2.883(2)	2.565(3)	2.541(3)	2.559(4)	this work

^{a} In this case the bond labels are arbitrary. ^{b} Two independent molecules.

solution could indicate stereospecific substitution processes. In the complex $Ru_3(\mu$-$H)_3(\mu_3$-CPh)(CO)$_7$(AsPh$_3$)$_2$ [14], which has a structure comparable with that of 3b with the capping CPh ligand replacing (η^5-C$_5$H$_5$)Ni, both triphenylarsine ligands are also in axial positions.

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