Synthesis of $[M(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (M = Os or Ru) and X-ray crystal structure of the osmium derivative

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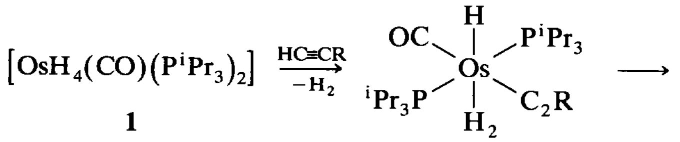
Abstract

The tetrahydrido complex $[OsH_4(CO)(P^iPr_3)_2]$ (**1**) reacts with diphenylacetylene to give *cis*- and *trans*-stilbene and $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (**5**). The X-ray crystal structure of **5** has been determined. **5** reacts with CO to give $[Os(\eta^2\text{-}C_2Ph_2)(CO)_2(P^iPr_3)_2]$ (**6**). The related ruthenium complex $[Ru(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (**7**) was obtained by reaction of $[RuH(\eta^2\text{-}H_2BH_2)(CO)(P^iPr_3)_2]$ (**8**) with diphenylacetylene. **7** reacts with CO to give $[Ru(CO)_3(P^iPr_3)_2]$ (**9**) and diphenylacetylene. The synthesis of the hydridovinyl complex $[\overline{OsH(C(COOEt)}{=}C(H)CO_2Et)(CO)(P^iPr_3)_2]$ (**10**) is also reported.

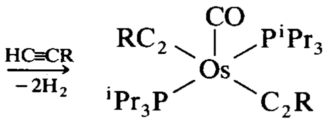
Key words: Ruthenium; Osmium

1. Introduction

As a part of a broad study on the catalytic properties of hydridoosmium compounds [1], we have recently reported the reactivity of the tetrahydrido complex $[OsH_4(CO)(P^iPr_3)_2]$ (**1**) with terminal alkynes [2]. **1** reacts with a stoichiometric amount of phenylacetylene or (trimethylsilyl)acetylene to give the σ-alkynylhydridodihydrogen compounds $[OsH(C_2R)(\eta^2\text{-}H_2)(CO)(P^iPr_3)_2]$ (R = Ph (**2a**) or $SiMe_3$ (**2b**)), which afford the derivatives $[Os(C_2R)_2(CO)(P^iPr_3)_2]$ (R = Ph (**3a**) or $SiMe_3$ (**3b**)) by reaction with a further molecule of alkyne (eqn. (1)).

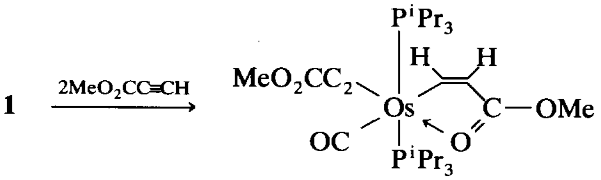


1 **2**



3 (1)

A similar reaction pattern might be expected for the activated alkyne methylpropiolate. However, the reaction of **1** with this alkyne leads to the alkynylvinyl complex $[\overline{Os(C_2CO_2Me)(CH{=}C(H)COOMe)}(CO)(P^iPr_3)_2]$ (**4**) (eqn. (2)).



4 (2)

We now report the reactivity of **1** towards diphenylacetylene and diethyl acetylenedicarboxylate, as well as

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the reaction of the tetrahydridoborate complex $[RuH(\eta^2\text{-}H_2BH_2)(CO)(P^iPr_3)_2]$ with diphenylacetylene.

2. Results and discussion

Treatment of a methanol solution of **1** with diphenylacetylene in a 1:3 molar ratio at room temperature for 15 min produces a red solid. The solid is $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (**5**) based on elemental analysis and IR, ^1H, $^{31}P\{^1H\}$ and $^{13}C\{^1H\}$ NMR spectroscopies. Furthermore, during the reaction two equivalents of diphenylacetylene are hydrogenated to *cis*- (one part) and *trans*-stilbene (four parts) according to eqn. (3).

$$\mathbf{1} + 3PhC{\equiv}CPh \rightarrow [Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2] + 2PhCH{=}CHPh \quad (3)$$

The molecular structure of complex **5** is shown in Fig. 1. Table 1 lists selected bond distances and angles. The most conspicuous features of the structure are the bond distances and angles of the $Os(\eta^2\text{-}C_2Ph_2)$ fragment. The bond lengths Os–C(2) (2.014(3) Å) and Os–C(3) (2.055(3) Å) are almost identical to that found in the vinyl complex $[Os((E)\text{-}CH{=}CHPh)Cl(CO)(P^iPr_3)_2]$ (1.99(1) Å) [3], shorter than the Os–C(sp) distances in the complex $[Os(\eta^2\text{-}Me_3SiC_2SiMe_3)(CO)_4]$ (2.267(6) and 2.244(6) Å) [4], and even shorter than the vinyl Os–C(sp^2) distance in $[Os(C_2CO_2Me)(CH{=}C(H)COOMe)(CO)(P^iPr_3)_2]$ (2.103(4) Å) and the Os–C(sp^3) distance in $[OsH(CH_3)(CO)_2(P^iPr_3)_2]$ (2.198(17) Å) [5]. The angles C(3)–C(2)–C(4) (134.3(3)°) and C(2)–C(3)–C(10) (137.1(4)°) are more acute than those observed for $\eta^2\text{-}C_2Ph_2$ in the cation $[Co(\eta^2\text{-}C_2Ph_2)(PMe_3)_3]^+$ (137.5(5)° and 143.6(5)°) [6]. These values indicate a smaller contribution of sp hybridization at the C(2) and C(3) carbon atoms and, therefore, a loss of the acetylenic character of the C_2Ph_2 group. Consequently, the bond length C(2)–C(3) is lengthened (1.318(5) Å) compared with free alkyne (1.198(3) Å) [7], and is quite similar to the corresponding bond length in olefins (*ca.* 1.32 Å) [8].

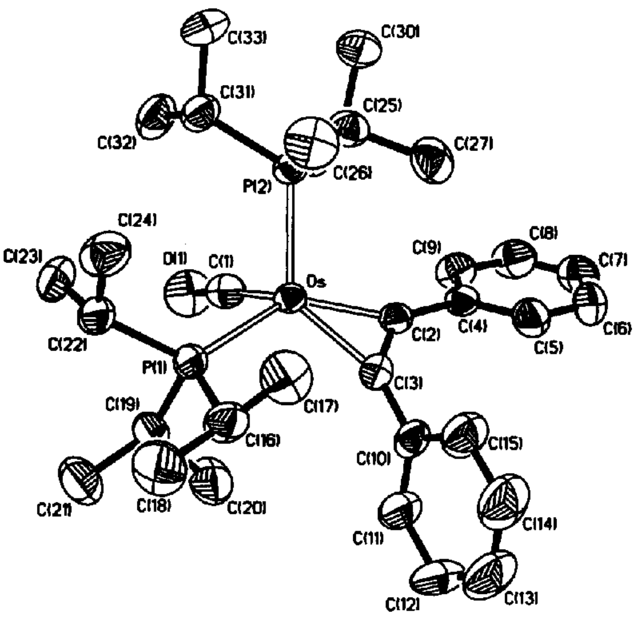


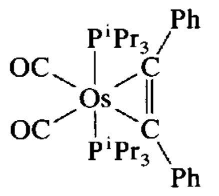
Fig. 1. Molecular structure and labelling scheme for complex 5.

TABLE 1. Selected bond distances (Å) and angles (°) for the complex $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (**5**)

Bond distances			
Os–P(1)	2.384(1)	C(2)–C(4)	1.472(5)
Os–P(2)	2.314(1)	C(3)–C(10)	1.457(5)
Os–C(1)	1.817(4)	C(4)–C(5)	1.376(6)
Os–C(2)	2.014(3)	C(4)–C(9)	1.398(5)
Os–C(3)	2.055(3)	C(10)–C(11)	1.389(6)
C(1)–O(1)	1.181(5)	C(10)–C(15)	1.385(6)
C(2)–C(3)	1.318(5)		
P(1)–C(16)	1.860(4)	P(2)–C(25)	1.856(4)
P(1)–C(19)	1.880(4)	P(2)–C(28)	1.875(4)
P(1)–C(22)	1.852(4)	P(2)–C(31)	1.866(4)
Bond angles			
P(1)–Os–P(2)	115.23(3)	Os–C(2)–C(3)	72.8(2)
P(1)–Os–C(1)	87.8(1)	Os–C(2)–C(4)	152.9(3)
P(1)–Os–C(2)	136.1(1)	C(3)–C(2)–C(4)	134.3(3)
P(1)–Os–C(3)	98.4(1)	Os–C(3)–C(2)	69.4(2)
P(2)–Os–C(1)	88.2(1)	Os–C(3)–C(10)	153.4(3)
P(2)–Os–C(2)	101.3(1)	C(2)–C(3)–C(10)	137.1(4)
P(2)–Os–C(3)	128.9(1)	C(2)–C(4)–C(5)	121.2(3)
C(1)–Os–C(2)	118.1(1)	C(2)–C(4)–C(9)	120.1(3)
C(1)–Os–C(3)	132.5(2)	C(3)–C(10)–C(11)	121.6(4)
C(2)–Os–C(3)	37.8(1)	C(3)–C(10)–C(15)	121.4(4)
Os–C(1)–O(1)	173.5(3)		

The Os–C(2) and Os–C(3) distances suggest that the bond between the osmium atom and the C_2Ph_2 ligand is very strong. Hence this group should not be easily displaced from the metallic centre by Lewis bases. In fact, **5** reacts with CO to give the *cis* dicarbonyl derivative $[Os(\eta^2\text{-}C_2Ph_2)(CO)_2(P^iPr_3)_2]$ (**6**) (eqn. (4)), which shows a band in the IR spectrum at 1615 cm^{-1} that suggests a coordination mode for C_2Ph_2 similar to that in **5**.

5 + CO ⟶



(4)

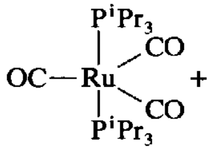
6

The carbon atoms C(2) and C(3) of **5** give rise to a triplet in the $^{13}C\{^1H\}$ NMR spectrum at 185.9 ppm, with a P–C coupling constant of 10.1 Hz. Furthermore, at room temperature and at −60°C, the $^{31}P\{^1H\}$ NMR spectrum shows only a single phosphine resonance at 40.9 ppm. These spectroscopic data are consistent with a non-rigid structure for **5** in solution.

The related complex, $[Ru(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (**7**), is obtained in methanol by reaction of the tetrahydridoborate complex $[RuH(\eta^2\text{-}H_2BH_2)(CO)(P^iPr_3)_2]$ (**8**) with diphenylacetylene. During the reaction the excess of diphenylacetylene was hydrogenated to *cis*- and *trans*-stilbene (eqn. (5)).

$$\mathbf{8}\ \xrightarrow[-\,PhCH=CHPh]{PhC\equiv CPh}\ \underset{\mathbf{7}}{[Ru(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]} \qquad (5)$$

The IR spectrum of **7** in Nujol shows a broad pattern similar to that observed for **5**, suggesting that in the solid state both compounds have similar structures. The $^{31}P\{^1H\}$ NMR spectrum of **7** also contains a lone singlet at 71.5 ppm from room temperature down to −60°C. This suggests that **7** is also not rigid in solution. The $^{13}C\{^1H\}$ NMR spectra of **5** and **7** are also similar, although the *C*Ph carbon atoms of the C_2Ph_2 group of **7** resonate at 143.1 ppm, 42.8 ppm to higher field than the related carbon atoms of **5**. However, there is a marked difference between the reactivities of **5** and **7** towards CO. Whereas **5** reacts with CO to give **6**, **7** gives the tricarbonyl $[Ru(CO)_3(P^iPr_3)_2]$ (**9**) and diphenylacetylene (eqn. (6)).

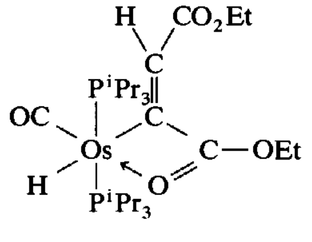
7 + 2CO ⟶  + PhC≡CPh (6)

9

On the basis of the usual Dewar–Chatt–Duncanson metal–olefin bonding model, the different stabilities of the $M(\eta^2\text{-}C_2Ph_2)$ bond of **5** and **7** can be rationalized in terms of the better back-donation of osmium relative to ruthenium. Formally **5** can be described as a pentacoordinate 16-electron osmium(II) derivative, whereas **7** may be considered to be a 16-electron ruthenium(0) compound.

The compound $[Ru(\eta^2\text{-}C_2Ph_2)(Cyttp)]$ (Cyttp = $PhP(CH_2CH_2CH_2P(cyclo\text{-}C_6H_{11})_2)_2$) has been reported by Jia and Meek [9]. This compound was obtained similarly to **5**, by reaction of $[RuH_4(Cyttp)]$ with diphenylacetylene. The $^{13}C\{^1H\}$ NMR spectrum shows two *C*Ph resonances at 195.6 and 160.1 ppm. The last is 17 ppm to lower field than that observed for the carbon atoms of **5**.

The formation of stilbene according to eqns. (3) and (5) probably involves hydridevinyl species as intermediates. It is relevant that the reaction of **1** with diethyl acetylenedicarboxylate leads to the hydridovinyl complex $[\overline{OsH(C(C}OOEt)=C(H)CO_2Et)(CO)(P^iPr_3)_2]$ (**10**) (eqn. (7)).

1 $\xrightarrow{EtO_2CC\equiv CCO_2Et}$  (7)

10

The proposal that one of the ester units coordinates to the osmium via the C=O oxygen is strongly supported by the IR spectrum in Nujol, which shows two stretching frequencies at 1705 and 1595 cm^{-1}. Complex **10** forms a deep yellow microcrystalline solid which is soluble in most organic solvents. Furthermore, the analogous chloro complexes have been isolated and structurally characterized by X-ray analysis [1a,10].

3. Experimental section

3.1. General considerations

All reactions were carried out with rigorous exclusion of air using Schlenk tube techniques. Solvents were dried by the usual procedures and distilled under argon prior to use.

The starting material $[OsH_4(CO)(P^iPr_3)_2]$ (**1**) was prepared *in situ* by decomposition of $[OsH(\eta^2\text{-}H_2BH_2)(CO)(P^iPr_3)_2]$ in methanol, and $[RuH(\eta^2\text{-}H_2BH_2)(CO)(P^iPr_3)_2]$ (**8**) was prepared by the published method [11].

3.2. Physical measurements

Elemental analyses were carried out with a Perkin–Elmer 240C microanalyser. Gas chromatography (GC) analysis was performed with a Perkin–Elmer 8500 gas chromatograph with a flame ionization detector and an FFAP on a Chromosorb GHP 80/100 mesh column at 200°C. IR spectra were run on a Perkin–Elmer 783 spectrophotometer. NMR spectra were recorded on a Varian 200 XL or Varian UNYT 300 spectrophotometer. Chemical shifts are expressed in parts per million upfield from Me_4Si (^{13}C,^1H) and 85% H_3PO_4 (^{31}P). Coupling constants are given in hertz.

3.3. Preparation of $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (5)

A solution of **1** (123 mg, 0.23 mmol) in 5 ml of methanol was treated with diphenylacetylene (123 mg, 0.69 mmol). The mixture was stirred for 15 min at room temperature, and a brown–red solid precipitated, which was filtered off, washed with methanol and dried *in vacuo*. Yield: 125 mg (76%). The mother liquor (0.2 μl) was injected in the gas chromatograph. GC analysis showed the presence of *cis*-stilbene and *trans*-stilbene in a 4:1 molar ratio, and no traces of diphenylacetylene. Anal. Calcd. for $C_{33}H_{52}OOsP_2$: C, 55.29; H, 7.31. Found: C, 55.19; H, 7.45%. IR (Nujol): ν(C≡O) 1850 (s), ν(C=C) 1635 (m), 1590 (w) cm^{-1}. ^1H NMR (200 MHz, C_6D_6): δ 7.5–7.1 (m, Ph), 2.3 (m, PCH), 1.17 and 1.14 (both dd, J(HH) = 17.1, J(HP) = 13.0, $PCCH_3$). $^{31}P\{^1H\}$ NMR (80.9 MHz, C_6D_6): δ 40.9 (s) (at 20°C and at −60°C). $^{13}C\{^1H\}$ NMR (75.33 MHz, C_6D_6): δ 195.1 (t, J(CP) = 9.0, CO), 185.9 (t, J(CP) = 10.1, ≡C), 125 and 124 (both s, Ph), 28.9 (m, PCH), 18.9 (s, $PCCH_3$).

3.4. Preparation of $[Os(\eta^2-C_2Ph_2)(CO)_2(P^iPr_3)_2]$ (6)

Carbon monoxide was bubbled through a solution of **5** (100 mg, 0.14 mmol) in 20 ml of hexane. After 30 min the mixture was concentrated to *ca.* 2 ml and cooled to −78°C for 12 h. An orange solid was formed, which was filtered off, washed with cold hexane and dried *in vacuo*. Yield: 49 mg (47%). Anal. Calcd. for $C_{34}H_{52}O_2OsP_2$: C, 54.82; H, 7.03. Found: C, 54.99; H, 7.22%. IR (Nujol): ν(C≡O) 1975, 1905 (s), ν(C=C) 1615 (m), 1540 (w) cm^{-1}. ^1H NMR (200 MHz, C_6D_6): δ 8.0–7.0 (m, Ph), 2.6 (m, PCH), 1.20 (dvt, J(HH) = 7.0, N = 13.4, $PCCH_3$). $^{31}P\{^1H\}$ NMR (80.9 MHz, C_6D_6): δ 6.5 (s).

3.5. Preparation of $[Ru(\eta^2-C_2Ph_2)(CO)(P^iPr_3)_2]$ (7)

A mixture of **8** (163 mg, 0.35 mmol) and diphenylacetylene (187 mg, 1.05 mmol) in 10 ml of methanol was stirred at room temperature for 90 min to give a red suspension. The mixture was evaporated under vacuum to *ca.* 5 ml, and the dark red solid was filtered off, washed with methanol and dried *in vacuo*. Recrystallization from toluene/methanol gave **7** as a microcrystalline dark red solid. Yield: 112 mg (51%). GC analysis of 0.2 μl of the mother liquor shows *cis*-stilbene and *trans*-stilbene in a 1:2 molar ratio, and no traces of diphenylacetylene. Anal. Calcd. for $C_{33}H_{52}OP_2Ru$: C, 63.13; H, 8.35. Found: C, 62.97; H, 8.76%. IR (Nujol): ν(C≡O) 1850 (s), ν(C=C) 1650 (m), 1590 (w) cm^{-1}. ^1H NMR (200 MHz, C_6D_6): δ 7.5–7.0 (m, Ph), 2.22 (m, PCH), 1.19 and 1.16 (both dvt, J(HH) = 7.1, N = 13.2, $PCCH_3$). $^{31}P\{^1H\}$ NMR (80.9 MHz, C_6D_6): δ 71.5 (s) (at 20°C and at −60°C). $^{13}C\{^1H\}$ NMR (75.33 MHz, C_6D_6): δ 176.2 (t, J(CP) = 11.3, CO), 143.1 (t, J(CP) = 3.3, ≡C), 125.0 and 123.7 (both s, Ph), 27.4 (m, PCH), 18.9 and 18.6 (both s, $PCCH_3$).

3.6. Preparation of $[Ru(CO)_3(P^iPr_3)_2]$ (9)

Carbon monoxide was bubbled through a solution of **7** (100 mg, 0.16 mmol) in 10 ml of dichloromethane. After 15 min the mixture was concentrated to *ca.* 0.5 ml. Addition of 5 ml of methanol led to the precipitation of a very pale yellow solid, which was filtered off, washed with cold methanol and dried *in vacuo*. Yield: 22 mg (27%). Anal. Calcd. for $C_{21}H_{42}O_3P_2Ru$: C, 49.89; H, 8.37. Found: C, 49.49; H, 8.78%. IR (CH_2Cl_2): ν(C≡O) 1950 (vw), 1860 (vs,br) cm^{-1}. ^1H NMR (200 MHz, C_6D_6): δ 2.2 (m, PCH), 1.27 (dvt, J(HH) = 7.1, N = 14.1, $PCCH_3$). $^{31}P\{^1H\}$ NMR (80.9 MHz, C_6D_6): δ 72.34 (s).

3.7. Preparation of $[\overline{OsH(C(COOEt)=C(H)CO_2Et)}(CO)(P^iPr_3)_2]$ (10).

A solution of **1** (134 mg, 0.25 mmol) in 5 ml of hexane was treated with diethyl acetylenedicarboxylate (85 μl, 0.50 mmol). The mixture was stirred for 4 h at room temperature, and, then taken to dryness. The residue was treated with 2 ml of methanol to give a suspension. The yellow precipitate was filtered off, washed with methanol and dried *in vacuo*. Yield: 120 mg (67%). Anal. Calcd. for $C_{27}H_{53}O_5OsP_2$: C, 45.68; H, 7.53. Found: C, 46.19; H, 7.98%. IR (Nujol): ν(OsH) 2170 (w), ν(C≡O): 1890 (s), ν(C=O), 1705 (m), ν(C=O) 1595 (m) cm^{-1}. ^1H NMR (200 MHz, C_6D_6): δ 7.27 (t, J(HP) = 2.4, =CH), 4.2 and 4.1 (both q, J(HH) = 7.2, OCH_2), 2.6 (m, PCH), 1.4 and 1.1 (both dvt, J(HH) = 7.0, N = 13.0, $PCCH_3$), 1.1 (t, J(HH) = 7.2, CH_2CH_3), −19.85 (t, J(HP) = 19.0, OsH). $^{31}P\{^1H\}$ NMR (80.9 MHz, C_6D_6): δ 24.0 (s).

3.8. X-Ray structural analysis of 5

Crystals suitable for an X-ray diffraction experiment were obtained by slow evaporation of a concentrated solution of **5** in methanol. Atomic coordinates are listed in Table 2. A summary of crystal data, intensity collection procedure, and refinement data is reported in Table 3. The irregular crystal studied was glued to a glass fibre and mounted on a Siemens AED-2 diffractometer. Cell constants were obtained from the least-squares fit of the setting angles of 54 reflections in the range $20° \leq 2\theta \leq 35°$. The 8231 recorded reflections were corrected for Lorentz and polarization effects. Three orientation and intensity standards were monitored every 55 min of measuring time; no variation was observed. Reflections were also corrected for absorption by an empirical method (maximum and minimum correction factor, 1.172–0.831) [12].

The structure was solved by Patterson (Os atom) and conventional Fourier techniques. Refinement was carried out by full-matrix least-squares with initial isotropic thermal parameters. Anisotropic thermal parameters were used in the last cycles of refinement for all non-hydrogen atoms. Hydrogen atoms were partially located from difference Fourier maps and included in the refinement (some of them in calculated positions; C–H = 0.97 Å) riding on carbon atoms with a common isotropic thermal parameter. Atomic scattering factors, corrected for anomalous dispersion for Os and P, were taken from ref. 13. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ with the weight defined as $w^{-1} = \sigma^2(F_o) + 0.0014F_o^2$. Final R and R_w values were 0.0282 and 0.0315 respectively. All calculations were performed using the SHELXTL-PLUS system of computer programs [14].

TABLE 2. Atomic coordinates ($\times 10^4$; $\times 10^5$ for Os and P atoms) and equivalent isotropic displacement coefficients (Å^2, $\times 10^3$; Å^2, $\times 10^4$ for Os and P atoms) for the compound $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (5)

Atom	x	y	z	U_{eq} [a]
Os	23956(1)	23443(1)	9754(1)	316(1)
P(1)	37656(7)	32489(6)	5944(5)	388(3)
P(2)	7199(7)	22124(5)	568(5)	360(3)
O(1)	1384(3)	3699(2)	1767(2)	69(1)
C(1)	1733(3)	3171(3)	1416(2)	47(1)
C(2)	2457(3)	1301(2)	1551(2)	37(1)
C(3)	3521(3)	1485(2)	1471(2)	42(1)
C(4)	1922(3)	624(2)	1898(2)	40(1)
C(5)	2386(4)	−119(3)	1890(2)	54(1)
C(6)	1899(4)	−749(3)	2253(3)	68(2)
C(7)	945(4)	−626(3)	2626(3)	67(2)
C(8)	478(4)	103(3)	2637(3)	66(2)
C(9)	958(3)	735(3)	2279(2)	53(1)
C(10)	4657(3)	1129(2)	1661(2)	47(1)
C(11)	5425(3)	1351(3)	2341(3)	64(2)
C(12)	6509(4)	984(4)	2523(4)	91(2)
C(13)	6810(4)	404(4)	2041(4)	95(3)
C(14)	6066(5)	185(3)	1362(4)	86(2)
C(15)	5006(4)	535(3)	1186(3)	66(2)
C(16)	5089(3)	2761(3)	359(3)	54(2)
C(17)	4851(4)	2066(3)	−203(3)	66(2)
C(18)	5990(4)	3296(3)	65(4)	83(2)
C(19)	4269(4)	3872(2)	1510(3)	56(1)
C(20)	4710(5)	3388(3)	2254(3)	78(2)
C(21)	5118(4)	4543(3)	1432(3)	78(2)
C(22)	3453(3)	4034(2)	−177(2)	52(1)
C(23)	2512(4)	4592(3)	21(3)	66(2)
C(24)	3164(4)	3717(3)	−1038(2)	66(2)
C(25)	897(3)	1453(2)	−712(2)	45(1)
C(26)	1728(4)	1704(3)	−1273(2)	66(2)
C(27)	1271(4)	656(3)	−332(3)	60(2)
C(28)	−470(3)	1775(2)	538(2)	44(1)
C(29)	−823(4)	2257(3)	1231(3)	61(1)
C(30)	−1556(3)	1505(3)	−36(3)	64(2)
C(31)	105(3)	3058(3)	−580(2)	49(1)
C(32)	−443(4)	3687(3)	−128(3)	65(2)
C(33)	−708(4)	2862(3)	−1368(3)	75(2)

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

TABLE 3. Crystal data and data collection and refinement for $[Os(\eta^2\text{-}C_2Ph_2)(CO)(P^iPr_3)_2]$ (5)

Crystal data	
Formula	$C_{33}H_{52}OOsP_2$
Mol. wt.	716.92
Colour and habit	Red transparent, irregular block
Crystal size (mm)	0.380×0.365×0.532
Crystal system	Monoclinic
Space group	$P2_1/c$ (no. 14)
a (Å)	11.7654(8)
b (Å)	16.9836(9)
c (Å)	16.7305(11)
β (°)	98.982(7)
V (Å^3)	3302.1(4)
Z	4
D (calcd) (g cm^{-3})	1.442
Data collection and refinement	
Diffractometer	Four-circle Siemens AED
λ (Mo Kα) radiation (Å); technique	0.71069; bisecting geometry
Monochromator	Graphite oriented
Temperature (K)	295
μ (cm^{-1})	39.84
Scan type	$\omega-2\theta$
2θ range (°)	3–50
No. of data collected	8231 ($+h$, $\pm k$, $\pm l$)
No. of unique data	6549
Unique observed data	5643 [$(F_o) \geq 4\sigma(F_o)$]
No. of parameters refined	335
R, R_w [a]	0.0282, 0.0315
R, wR (all data)	0.0284, 0.0392

[a] $w^{-1} = \sigma^2(F) + 0.0014F^2$.

Tables of anisotropic thermal parameters, atomic coordinates for hydrogen atoms, experimental details of the X-ray study, bond distances and angles, selected least-squares planes and interatomic distances may be obtained from the Cambridge Crystallographic Data Centre.

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