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Seven-coordinate complexes of molybdenum(II) and tungsten(II) containing phosphite donor ligands: X-ray crystal structures of $[WI_2(CO)_3\{P(O^iPr)_3\}_2]$ and $[WI_2(CO)_2\{P(OMe)_3\}_3]$

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Abstract

Treatment of $[MI_2(CO)_3(NCMe)_2]$ with two equivalents of $P(OR)_3$ (for M = W only, R = Me, ^{i}Pr; for M = Mo and W, R = Ph) in Et_2O gave the bis(phosphite) complexes $[MI_2(CO)_3\{P(OR)_3\}_2]$ (**1**–**4**) in high yield. The complex for M = W, R = ^{i}Pr was crystallographically characterised, and has a distorted capped octahedral geometry with a phosphite group capping a tricarbonyl face. Reaction of $[WI_2(CO)_3(NCMe)_2]$ with three equivalents of $P(OR)_3$ (R = Me, Et) in Et_2O afforded the tris(phosphite) complexes $[WI_2(CO)_2\{P(OR)_3\}_3]$, which was crystallographically characterised for R = Me. The structure of $[WI_2(CO)_2\{P(OMe)_3\}_3]$ has an almost idealised capped trigonal prismatic geometry with a phosphite ligand capping a rectangular face containing C(1), C(2), I(2) and P(3). © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Seven-coordinates; Molybdenum(II); Tungsten(II); Phosphites

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1. Introduction

Although a large number of halocarbonyl seven-coordinate complexes of molybdenum(II) and tungsten(II) containing phosphine ligands have been reported [1–19], very few examples containing phosphites have appeared. Two crystallographically characterised examples are: $[WBr_2(CO)\{P(OMe)_3\}_2\{(Me)_2AsC(CF_3){=}C(CF_3)As(Me)_2\}]$ [20] and $[MoCl(SnCl_2Bu^n)(CO)_2\{P(OMe)_3\}_3]$ [21]. It is interesting to note that reaction of the chloro-bridged dimer, $[\{Mo(\mu\text{-}Cl)Cl(CO)_4\}_2]$ with $P(OMe)_3$ yielded the unusual crystallographically characterised product $[Mo_2Cl_3(CO)_4\{P(OMe)_3\}_4]^{n+}[MoCl_4(O)\{OP(OMe)_2\}]^{n-}$ [22], rather than an expected seven-coordinate neutral halocarbonyl product such as $[MoCl_2(CO)_3\{P(OMe)_3\}_2]$.

Over the past 12 years we have been exploring the chemistry of the highly versatile seven-coordinate complexes $[MXY(CO)_3(NCMe)_2]$ (M = Mo, W; X, Y = halide or pseudo-halide) with a variety of neutral and anionic donor ligands [23,24]. In this paper we describe the reactions of $[MI_2(CO)_3(NCMe)_2]$ (M = Mo, W) with $P(OR)_3$ (R = Me, Et, ^{i}Pr, Ph). An early communication on this work has been published [25].

2. Results and discussion

The starting materials used in this research, namely $[MI_2(CO)_3(NCMe)_2]$ (M = Mo, W) were prepared by reacting the zero-valent complexes fac-$[M(CO)_3(NCMe)_3]$ in situ with equimolar quantities of

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Table 1
Physical and analytical data for the complexes **1**–**6**

Complex	Colour	Yield (%)	Found C	Calculated H (%)
(**1**) $[WI_2(CO)_3\{P(OMe)_3\}_2]$	Yellow	68	14.2 (14.0)	2.5 (2.4)
(**2**) $[WI_2(CO)_3\{P(O^iPr)_3\}_2]$	Yellowish orange	79	26.9 (26.9)	4.5 (4.5)
(**3**) $[MoI_2(CO)_3\{P(OPh)_3\}_2].Et_2O$	Yellow	76	45.5 (45.8)	3.3 (3.6)
(**4**) $[WI_2(CO)_3\{P(OPh)_3\}_2].Et_2O$	Yellow	71	42.3 (42.4)	3.0 (3.3)
(**5**) $[WI_2(CO)_2\{P(OMe)_3\}_3]$	Yellow	60	15.1 (15.3)	3.2 (3.1)
(**6**) $[WI_2(CO)_2\{P(OEt)_3\}_3]$	Yellow	74	24.3 (24.2)	4.7 (4.6)

I_2 (at 0°C) [26]. Reaction of $[MI_2(CO)_3(NCMe)_2]$ with two equivalents of $P(OR)_3$ in Et_2O at room temperature afforded excellent yields of the acetonitrile displaced products $[MI_2(CO)_3\{P(OR)_3\}_2]$ (M = W, R = Me, ^{i}Pr; M = Mo, W, R = Ph) (**1**–**4**). Complexes **1**–**4** have been fully characterised by elemental analysis (C and H) (Table 1), IR, ^{1}H, ^{31}P and in selected cases by ^{13}C-NMR spectroscopy (Table 2). The triphenylphosphite complexes $[MI_2(CO)_3\{P(OPh)_3\}_2].Et_2O$ (M = Mo, W) (**3** and **4**) were confirmed as diethyl ether solvates by repeated elemental analyses and ^{1}H-NMR spectroscopy. ^{1}H-NMR data for **1** and the IR data for **4** are not included as they have been previously presented [25]. The IR data for complex **1** is incorrect in the original communication, and the carbonyl bands in Table 2 (2043, 1980 and 1942 cm^{-1}) are the correct values for this complex. The complexes are all very soluble in organic solvents such as CH_2Cl_2, $CHCl_3$, Et_2O and toluene, but only moderately soluble in hexane and pentane. They are air-sensitive in the solid state and very air-sensitive in solution, however they can be stored under an inert atmosphere in the solid state for several days without significant decomposition. The IR spectra for complexes **1**–**4** (Table 2) all have three carbonyl bands in the expected region for $[MX_2(CO)_3L_2]$ type complexes, except for the molybdenum complex $[MoI_2(CO)_3\{P(OPh)_3\}_2].Et_2O$ (**3**), which has four bands at 2002(sh), 1967, 1934 and 1901 cm^{-1}, which may indicate the presence of isomers. A nujol mull was also recorded of complex **3** and also shows four bands at 1990, 1954, 1925 and 1894 cm^{-1}, which again suggests the presence of isomers. It should be noted that a number of attempts to prepare other molybdenum complexes of the types $[MoI_2(CO)_3\{P(OR)_3\}_2]$ and $[MoI_2(CO)_2\{P(OR)_3\}_3]$ were unsuccessful, mainly due to the instability of these complexes, and the likely formation of mixtures. In order to study the solid state structure, suitable single crystals for X-ray analysis of $[WI_2(CO)_3\{P(O^iPr)_3\}_2]$ (**2**) were grown by cooling a concentrated Et_2O solution of **2** at -5°C for 72 h.

Table 2
IR[a], ^{1}H-[b], ^{31}P-[c] and ^{13}C- NMR[b] data for the phosphite complexes

Complex	IR ν(CO) (cm^{-1})	^{1}H-NMR δ (ppm)	^{31}P-$\{^1$H$\}$NMR δ (ppm)	^{13}C-$\{^1$H$\}$NMR δ (ppm)
1	2043(s); 1980(s); 1942(s)		115.35(s); J_{WP} not resolved	209.36(t, 2*C*O);198.39(br, *C*O); 55.20(t, $P(OCH_3)_3$)
2	2038(m); 1971(s); 1932(s)	4.82{m, 6H,$P\{OCH(CH_3)_2\}_3$}; 1.48{d, $J_{P-H} = 8.5$ Hz, 36H,$P\{OCH(CH_3)_2\}_3$}	104.11(s), $J_{WP} = 246.0$ Hz	212.66(t, 2*C*O); 199.99 (t, *C*O); 72.95(t, $P(OCH(CH_3)_2)$; 23.71(s, $P(OCH(CH_3)_2)$
3	2002(sh); 1967(s); 1934(s); 1901(s)	7.10–7.60{br,m, 30H,$P(OC_6H_5)_3$}; 3.45{q, $J_{P-H} = 6.9$ Hz, 4H,$(CH_3CH_2)_2O$}; 1.25{t, $J_{P-H} = 6.5$ Hz, 6H,$(CH_3CH_2)_2O$}	129.51(s); J_{WP} not resolved	
4		6.80–7.60{br, m, 30H,$P(OC_6H_5)_3$}; 3.50{q,$J_{P-H} = 6.7$ Hz, 4H,$(CH_3CH_2)_2O$}; 1.25{t, $J_{P-H} = 6.4$ Hz, 6H,$(CH_3CH_2)_2O$}	99.32(s); $J_{WP} = 347$ Hz	
5	1961(s); 1881(s)	3.70{m, $P(OCH_3)_3$}	117.57(br); 107.37(br,s); 93.84 (br,m)	~228(br, *C*O); ~221(br, *C*O); 55.27(br, s, $P(OCH_3)_3$)
6	1957(s); 1881(s)	4.12{br,m, 18H,$P(OCH_2CH_3)_3$}; 1.28{t, $J_{P-H} = 6.2$ Hz, 27H,$P(OCH_2CH_3)_3$}	113.12(br); 103.18(br,s)/ 101.60(br,s); 89.67(br,m)	

[a] Spectra recorded as thin $CHCl_3$ films between NaCl plates.
[b] Spectra recorded in $CDCl_3$ at 25°C and referenced to $SiMe_4$.
[c] Spectra recorded in $CDCl_3$ at 25°C and referenced to 85% H_3PO_4.

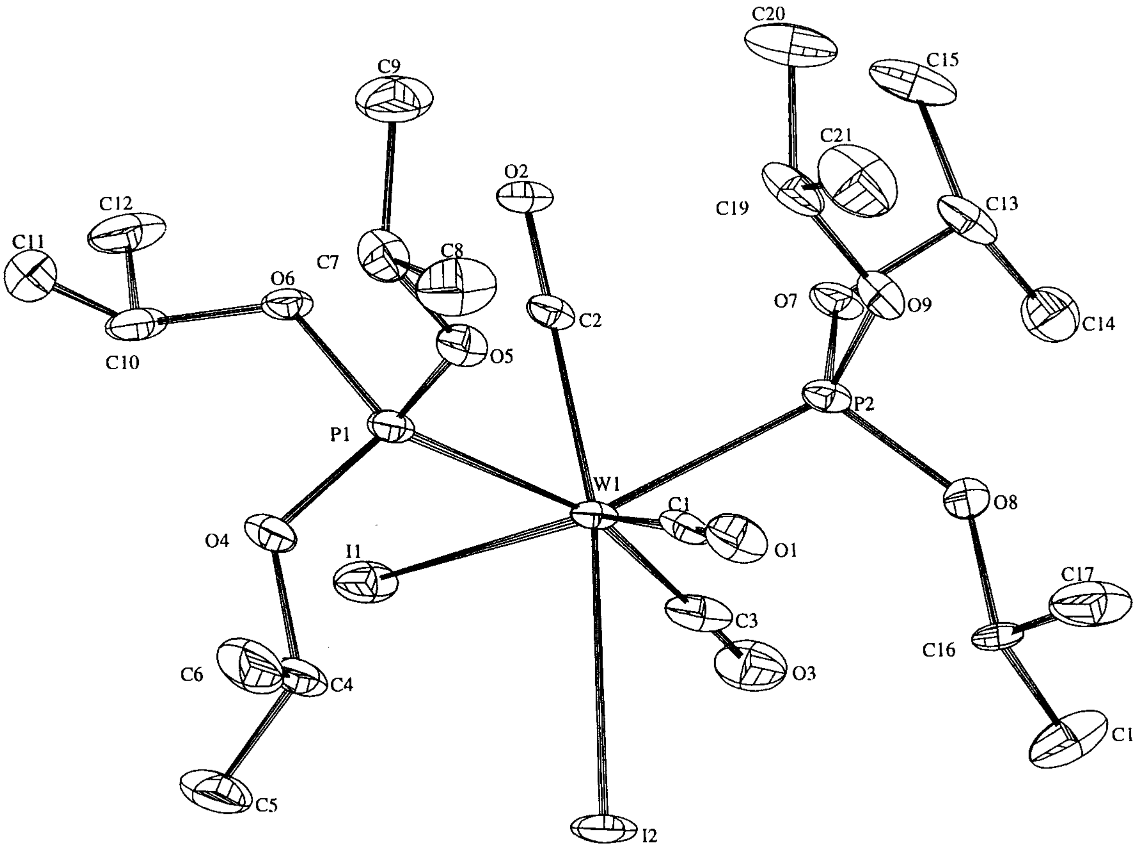

Fig. 1. X-ray crystal structure of $[\mathrm{WI_2(CO)_3\{P(O^iPr)_3\}_2}]$ (**2**). Thermal ellipsoids shown at 30% probability.

The structure of $[\mathrm{WI_2(CO)_3\{P(O^iPr)_3\}_2}]$ (**2**) is shown in Fig. 1, together with the atomic numbering scheme. The structure of **2** exhibits a distorted capped octahedral geometry. The capping moiety is the phosphite ligand P(2), which occupies the face formed by the three carbonyl ligands (the three W–C bond lengths are very similar, W(1)–C(1) = 2.007(7), W(1)–C(2) = 1.982(7), W(1)–C(3) = 2.068(7) Å). The distortion from ideal capped octahedral geometry is caused by the larger steric bulk of the phosphite ligand, which forces the C(1) carbonyl group out of plane (I(1)–W(1)–P(2) = 136.22(5) and I(1)–W(1)–C(1) = 150.6(2)°). This is unusual in that one would normally expect the ligand with the lesser bulk to form the cap. W–P bond lengths are similar (2.525(2) and 2.420(3) Å), when one would expect the ligand forming the cap to have a longer interaction with the metal. This is due to the phosphite group capping a face formed by carbonyls, which have a small steric bulk and allow the bulky ligand to bond at its ideal distance.

The room temperature ^{1}H-NMR spectra of **2**–**4** conform with the solid state structure of **2** shown in Fig. 1. The room temperature ^{31}P-NMR spectrum for **2** showed a singlet at 104.13 ppm, flanked by the W satellites, giving a J_{WP} of 246 Hz. The room temperature ^{13}C-{^{1}H}NMR spectra for complexes **1** and **2** were also obtained (Table 2), and for example show, the presence of carbonyl resonances at $\delta = 209.36$ and 198.39 ppm for **1**, which are in the normal octahedral range. The intensity ratio of the resonances at $\delta = 209.36$ to 198.39 is ca. 2:1. It is very likely the two equivalent carbonyl groups *trans*- to the two iodo groups can be ascribed to the resonance at 209.36 ppm, and the higher field resonance at 198.39 ppm to the carbonyl group *trans*- to the octahedral phosphite ligand. Since the ^{13}C-NMR spectrum of complex **2** is similar to **1** in the carbonyl region, it is likely that their structures will be very similar.

Treatment of $[\mathrm{WI_2(CO)_3(NCMe)_2}]$ with three equivalents of $\mathrm{P(OR)_3}$ (R = Me, Et) in $\mathrm{Et_2O}$ at room temperature gave the tris(phosphite) complexes $[\mathrm{WI_2(CO)_2\{P(OR)_3\}_3}]$ (**5** and **6**) in high yield. Complexes **5** and **6** were characterised as previously (Tables 1 and 2). They are both extremely soluble in most organic solvents (more so than the bis(phosphite) complexes **1**–**4**), and are also very air-sensitive in solution. The IR spectra for **5** and **6** both have two carbonyl bands at 1961 and 1881cm^{-1} (**5**) and 1957 and 1881cm^{-1} (**6**), respectively. This agrees with the solid state structure of **5** shown in Fig. 2.

Suitable single crystals for X-ray analysis of $[\mathrm{WI_2(CO)_2\{P(OMe)_3\}_3}]$ (**5**) were grown by cooling a

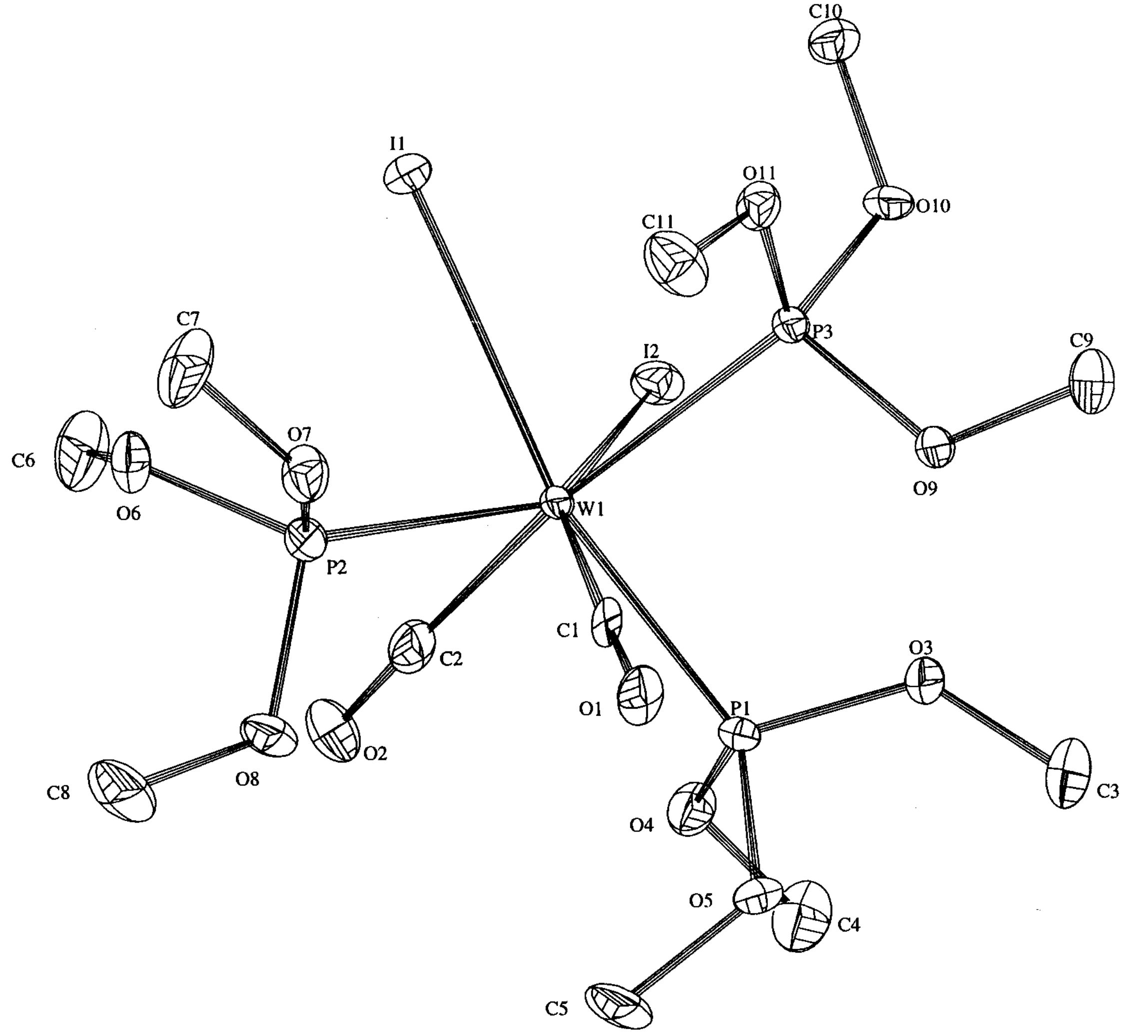

Fig. 2. X-ray crystal structure of $[WI_2(CO)_2\{P(OMe)_3\}_3]$ (**5**). Thermal ellipsoids shown at 30% probability.

concentrated Et_2O solution of **5** to -5°C for 72 h. The molecular structure of **5** is shown in Fig. 2, together with the atom numbering scheme. The tungsten atom lies in a seven-coordinate environment, the geometry of which is best described as being a slight distortion from a capped trigonal prism due to differing spatial requirements of the ligands. P(1) forms the cap upon the face defined by C(1), C(2), I(2) and P(3), giving coordination angles of approximately 80° [C(1)–W(1)–P(1) = 76.1(2)°, C(2)–W(1)–P(1) = 76.6(2)°, P(1)–W(1)–I(2) = 79.55(5)°], apart from P(3) which forms an angle P(1)–W(1)–P(3) = 94.32(6)° due to steric repulsion between the two bulky phosphite groups. This geometry is in agreement with the related complex $[MoCl(SnCl_2Bu^n)(CO)_2\{P(OMe)_3\}_3]$ [21].

The room temperature ^{1}H-NMR spectrum of **5** agrees with its solid-state structure shown in Fig. 2. The room temperature ^{31}P-$\{^1$H$\}$NMR spectrum of **5** showed a rather broad spectrum, consisting of two doublet of doublets centred at 117.57 and 93.84 ppm, and a broad singlet (possibly a triplet) at $\delta = 107.37$ ppm. The room temperature ^{13}C-$\{^1$H$\}$NMR spectrum (Table 2) of **5** has carbonyl resonances at $\delta = 228$ and 221 ppm, which appear as broad resonances, and hence it is difficult to assign with the carbonyl groups in complex **5**.

In summary, although the reactions of $[MI_2(CO)_3(NCMe)_2]$ (M = Mo and W) with both two and three equivalents of $P(OR)_3$ (R = Me, Et, ^{i}Pr and Ph) gave six fully characterised complexes, a number of attempts were made to isolate and characterise the other analogues in the series without success. Many of the complexes were oils and difficult to purify. It is interesting to note that the two representative crystal structures of $[WI_2(CO)_3\{P(O^iPr)_3\}_2]$ (**2**) and $[WI_2(CO)_2\{P(OMe)_3\}_3]$ (**5**) both have different basic seven-coordinate structures which is most likely to be due to the very different steric requirements of two and three phosphite ligands attached to the tungsten centres in **2** and **5**, respectively.

Table 3
Crystal data and structure refinement for Compounds **2** and **5**

	Compound **2**	Compound **5**
Empirical formula	$C_{21}H_{42}I_2O_9P_2W$	$C_{11}H_{27}I_2O_{11}P_3W$
Formula weight	938.14	865.89
Crystal system	Triclinic	Monoclinic
Space group	P-1	$P2_1/c$
Unit cell dimensions		
a (Å)	10.187(3)	16.5268(6)
b (Å)	11.154(9)	10.051(3)
c (Å)	16.195(3)	14.9667(6)
α (°)	78.12(2)	90.0
β (°)	73.93(2)	100.73(2)
γ (°)	69.12(2)	90.0
V (Å^3)	1640.2(14)	2442.6(3)
Z	2	4
Density (calculated) (Mg m^{-3})	1.900	2.355
Absorption coefficient (mm^{-1})	5.541	7.499
F(000)	900	1624
Crystal size (mm)	0.28 × 0.18 × 0.11	0.28 × 0.24 × 0.1
Theta range for data collection (°)	1.97–28.58	2.38–24.92
Index ranges	$-13 \leq h \leq 12$; $-14 \leq k \leq 9$; $-20 \leq l \leq 17$	$-16 \leq h \leq 17$; $-11 \leq k 11$; $-16 \leq l \leq 17$
Reflections collected	8044	8088
Independent reflections	6275 [$R_{int} = 0.0541$]	3393 [$R_{int} = 0.0579$]
Absorption correction factors	0.864/1.240	0.854/1.381
No. of parameters	328	262
Goodness-of-fit on F^2	1.001	0.986
Final R indices, [I > 2σ(I)]	$R_1 = 0.0398$; $wR_2 = 0.0985$; (5367 reflections)	$R_1 = 0.0314$; $wR_2 = 0.0683$; (2891 reflections)
R indices (all data)	$R_1 = 0.0461$; $wR_2 = 0.1088$	$R_1 = 0.0392$; $wR_2 = 0.0769$
Largest difference peak and hole (e Å^{-3})	2.683 and −2.667	1.695 and −0.979

3. Experimental details

All preparations described in this paper were carried out using standard vacuum/Schlenk line techniques. The starting materials, $[MI_2(CO)_3(NCMe)_2]$ (M = Mo, W) were prepared by the literature method [26]. Diethyl ether was dried over sodium wire before use. All chemicals used were purchased from commercial sources.

Elemental analyses (C, H and N) were determined using a Carlo Erba Elemental Analyser MOD 1108 (using helium as a carrier gas). IR spectra were recorded as thin CH_2Cl_2 films between NaCl plates. ^{1}H-, ^{13}C- and ^{31}P-NMR spectra were recorded on a Bruker AC 250 MHz NMR spectrometer. ^{1}H- and ^{13}C-NMR spectra were referenced to $SiMe_4$, and ^{31}P-NMR spectra were referenced to 85% H_3PO_4.

3.1. *$[WI_2(CO)_3\{P(O^iPr)_3\}_2]$ (**2**)*

In a typical reaction; to $[WI_2(CO)_3(NCMe)_2]$ (0.80 g, 1.33 mmol) dissolved in diethyl ether (50 cm^3) with continuous stirring under a stream of dry N_2 was added $P(O^iPr)_3$ (0.554 g, 2.66 mmol). After stirring for 2 min, the solution went light brown. The mixture was then stirred for a further 3 h. Removal of the solvent to half volume in vacuo and cooling to −5°C for 72 h gave small regular orange crystals suitable for X-ray crystallography of $[WI_2(CO)_3\{P(O^iPr)_3\}_2]$ (**2**). Yield of pure product = 0.981 g, 79%.

Similar reactions of $[WI_2(CO)_3(NCMe)_2]$ with two equivalents of $P(OR)_3$ (for M = W only, R = Me; for M = Mo, W; R = Ph) afforded $[MI_2(CO)_3\{P(OR)_3\}_2]$ (**1**, **3** and **4**). See Table 1 for physical and analytical data.

3.2. *$[WI_2(CO)_2\{P(OMe)_3\}_3]$ (**5**)*

In a typical reaction; to $[WI_2(CO)_3(NCMe)_2]$ (0.80 g, 1.33 mmol) dissolved in diethyl ether (50 cm^3) with continuous stirring under a stream of N_2 was added $P(OMe)_3$ (0.495 g, 3.99 mmol). After stirring for 1 min, the solution went bright yellow. The mixture was stirred for a further 3 h. Removal of the solvent to half volume in vacuo and cooling to −5°C for 72 h gave small regular yellow crystals which were suitable for X-ray crystallography of $[WI_2(CO)_2\{P(OMe)_3\}_3]$ (**5**). Yield of pure product = 0.688 g, 60%.

A similar reaction of $[WI_2(CO)_3(NCMe)_2]$ with three equivalents of $P(OEt)_3$ in Et_2O afforded $[WI_2(CO)_2\{P(OEt)_3\}_3]$ (**6**) as a yellow crystalline solid. See Table 1 for physical and analytical data.

3.3. *X-ray crystallography—crystal structure determinations*

Crystals of **2** and **5** suitable for structure determinations were grown from concentrated cooled (−5°C) Et_2O solutions of **2** and **5** for 72 h. Crystal data and experimental conditions are summarised in Table 3.

Table 4
Selected bond lengths (Å) and angles (°) for complexes **2** and **5**

2		**5**	
Bond length (Å)			
W(1)–C(1)	2.007(7)	W(1)–C(1)	1.948(7)
W(1)–C(2)	1.982(7)	W(1)–C(2)	2.001(8)
W(1)–C(3)	2.068(7)	W(1)–P(1)	2.454(2)
W(1)–P(1)	2.525(2)	W(1)–P(2)	2.416(2)
W(1)–P(2)	2.420(3)	W(1)–P(3)	2.567(2)
W(1)–I(1)	2.828(4)	W(1)–I(1)	2.924(7)
W(1)–I(2)	2.912(7)	W(1)–I(2)	2.871(8)
Bond angles (°)			
C(1)–W(1)–C(2)	114.4(3)	C(1)–W(1)–C(2)	109.3(3)
C(2)–W(1)–C(3)	104.3(3)	C(1)–W(1)–P(1)	76.1(2)
C(1)–W(1)–C(3)	119.2(3)	C(2)–W(1)–P(1)	76.6(2)
C(1)–W(1)–P(1)	77.8(2)	C(1)–W(1)–P(2)	70.6(2)
C(2)–W(1)–P(1)	75.2(2)	C(2)–W(1)–P(2)	74.2(3)
C(3)–W(1)–P(1)	160.0(2)	C(1)–W(1)–P(3)	78.5(2)
C(1)–W(1)–P(2)	73.1(2)	C(2)–W(1)–P(3)	165.7(3)
C(2)–W(1)–P(2)	74.8(2)	P(1)–W(1)–P(2)	124.41(6)
C(3)–W(1)–P(2)	74.2(2)	P(2)–W(1)–P(3)	120.01(7)
P(1)–W(1)–P(2)	123.74(7)	P(1)–W(1)–P(3)	94.32(6)
C(1)–W(1)–I(1)	150.6(2)	C(1)–W(1)–I(1)	116.8(2)
C(2)–W(1)–I(1)	81.9(2)	C(2)–W(1)–I(1)	108.9(2)
C(3)–W(1)–I(1)	76.5(2)	C(1)–W(1)–I(2)	150.1(2)
C(1)–W(1)–I(2)	75.2(2)	C(2)–W(1)–I(2)	81.2(2)
C(2)–W(1)–I(2)	168.5(2)	P(1)–W(1)–I(1)	161.45(5)
C(3)–W(1)–I(2)	74.7(2)	P(2)–W(1)–I(1)	73.87(5)
P(1)–W(1)–I(1)	83.70(6)	P(3)–W(1)–I(1)	76.55(4)
P(1)–W(1)–I(2)	101.78(5)	P(1)–W(1)–I(2)	79.55(5)
P(2)–W(1)–I(1)	136.22(5)	P(2)–W(1)–I(2)	138.78(5)
P(2)–W(1)–I(2)	115.27(5)	P(3)–W(1)–I(2)	86.40(5)
I(1)–W(1)–I(2)	86.74(3)	I(1)–W(1)–I(2)	83.74(2)

Data were collected at 150(2) K on a FAST TV Area detector diffractometer following previously described procedures [27]. The structures were solved via heavy atom methods (SHELX-S) [28] and then refined by full matrix least squares on all F_o^2 data (SHELX-93) [29]. Non-hydrogen atoms were treated anisotropically, whilst hydrogen atoms were placed in idealised positions, (C–H = 0.96 Å, with U_{iso} tied to U_{eq} of the parent atoms). An empirical absorption correction was applied using DIFABS [30]. Values for selected bond lengths and angles are given in Table 4. Full details of the data collection, structure refinement, atomic coordinates, bond lengths and angles, and thermal parameters have been deposited as supplementary material.

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