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SYNTHESIS AND SPECTRA OF BIS(TERTIARYPHOSPHINE) DERIVATIVES OF CYCLOHEPTATRIENYLTUNGSTEN COMPLEXES

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Summary

Reaction of $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ with dppm (dppm = $Ph_2PCH_2PPh_2$) or dppe (dppe = $Ph_2PCH_2CH_2PPh_2$) gives the trihaptocycloheptatrienyl complexes $[WI(CO)_2(L\text{–}L)(\eta^3\text{-}C_7H_7)]$ [L–L = dppm, (A_1); L–L = dppe (A_2)]. The complex A_1 reacts with NH_4PF_6 to give the unidentate biphosphine complex $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (**B**) which yields $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)][PF_6]$ (**C**) on reaction with $Me_3NO \cdot 2H_2O$. Substitution of a carbonyl ligand in $[W(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ with the organometallic phosphine ligand $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ yields the heterobimetallic $[\{W(CO)_2(\eta^7\text{-}C_7H_7)\}(\mu\text{-}dppe)\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}][PF_6]_2$ (**D**).

Introduction

The synthesis of a variety of bis(tertiaryphosphine) derivatives of cycloheptatrienylmolybdenum complexes has been accomplished via a combination of three routes comprising direct reaction of the bisphosphine with $[MoI(CO)_2(\eta^7\text{-}C_7H_7)]$ [1], $[Mo(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ [2] or with $[Mo(\eta^6\text{-toluene})(\eta^7\text{-}C_7H_7)][PF_6]$ in a suitable coordinating solvent such as CH_3CN [3]. The corresponding chemistry of tungsten is restricted by the inaccessibility of $[W(\eta^6\text{-toluene})(\eta^7\text{-}C_7H_7)][PF_6]$ and, prior to this work, consisted only of a brief report of $[WI(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$ [4]. This paper describes our work on the synthesis of bis(tertiaryphosphine)cycloheptatrienyltungsten complexes starting from $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ and $[W(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$. The importance of ^{31}P NMR as a structural probe in these complexes is also described and we report the first values of $J(^{183}W\text{–}P)$ obtained for cycloheptatrienyl complexes.

Results and discussion

In toluene at 20°C $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ reacts with dppm or dppe to give deep red solutions from which $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ (A_1) (Scheme 1) and [WI-

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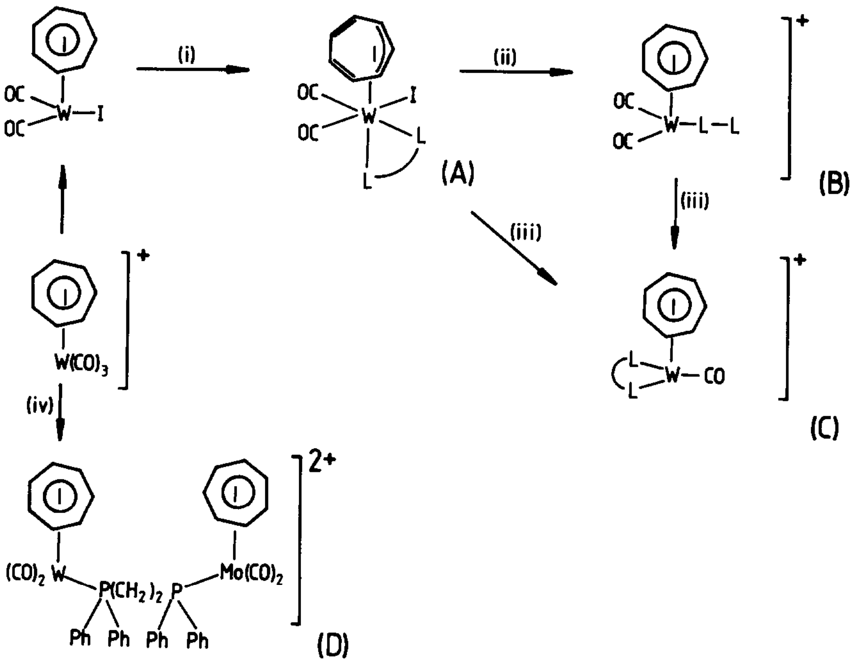

SCHEME 1. Reagents: (i) dppm or dppe in toluene; (ii) L–L = dppm, NH_4PF_6 in aqueous acetone; (iii) L–L = dppm, $Me_3NO \cdot 2H_2O$ in acetone; (iv) $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ in refluxing ethanol (95%).

$(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$ (A_2) are obtained in good yield as deep red solids. Full details of the characterisation of complexes **A** are given in Tables 1, 2 and 3.

At 263 K ^{31}P NMR spectra of complexes **A** exhibit two distinct and separate ^{31}P

TABLE 1

INFRARED SPECTRAL AND ANALYTICAL DATA

Complex		Yield (%)	ν(CO) (cm^{-1})[a]		Analysis (Found (calcd.)(%)) C	H
$[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$	(**A_1**)	65	1939	1860	47.9	3.3
					(48.5)	(3.4)
$[WI(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$	(**A_2**)	91	1932	1851	49.6	4.0
					(49.1)	(3.6)
$[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$	(**B**)	40	2004	1958	47.5	3.5
					(47.4)	(3.8)
$[W(CO)(dppm)(\eta^7\text{-}C_7H_7)][PF_6]$	(**C**)	37	1944		47.3	3.6
					(47.6)	(3.5)
$[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]I$	(**C**)	6	1944		48.2	3.4
					(48.7)	(3.6)
$[\{W(CO)_2(\eta^7\text{-}C_7H_7)\}(\mu\text{-}dppe)\text{-}\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}][PF_6]_2$	(**D**)	37	1994	1948[b]	41.8	3.0
					(41.8)	(3.0)

[a] In CH_2Cl_2 unless stated otherwise. [b] In CH_3CN.

TABLE 2

^{31}P NMR SPECTRAL DATA

Complex		$\delta(^{31}P)$ (ppm) [a]		$\lvert ^{1}J(^{183}W-P)\rvert$ (Hz) [b]		$\lvert J(P-P)\rvert$ (Hz)	Solvent
$[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$	**A$_1$**	−21.6,	−40.7	191 173	[−21.6] [−40.7]	12.3	$CDCl_3$ [c]
$[WI(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$	**A$_2$**	27.0	21.8	[d]		<10	$CDCl_3$ [c]
$[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$	**B**	5.1 [e],	−24.9 [f]	342	[5.1]	95	acetone-d_6
$[W(CO)(dppm)(\eta^7\text{-}C_7H_7)][PF_6]$	**C**	−25.8		277			CD_3CN
$[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]I$	**C**	−25.9		277			DMSO-d_6
$[\{W(CO)_2(\eta^7\text{-}C_7H_7)\}(\mu\text{-dppe})\text{-}\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}][PF_6]_2$	**D**	35.8 [g],	8.1 [e]	337	[8.1]	31	CD_3CN
$[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$		35.1 [g],	−12.9 [f]			37	acetone-d_6

[a] Positive chemical shift values to low field of reference. [b] Figures in brackets denote chemical shift of resonance associated with ^{183}W satellites. [c] Spectrum recorded at −10°C. [d] Not clearly resolved. [e] Tungsten-bound phosphorus. [f] Uncoordinated phosphorus. [g] Molybdenum-bound phosphorus.

resonances thereby demonstrating inequivalent phosphorus atoms in these complexes. Moreover the chelate conformation of the bisphosphine ligand in complexes of the type $[MI(CO)_2(L\text{-}L)(\eta^3\text{-}C_7H_7)]$ (M = Mo, W; L–L = bisphosphine) has been confirmed for the first time by the ^{31}P spectrum of $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ for which well resolved ^{183}W satellites are associated with the resonance due to each ^{31}P environment. If complexes **A** are assumed to have a pseudooctahedral arrangement of ligands in which the exact orientation of the cycloheptatrienyl ring with respect to the other ligands is not considered then the structure of complexes **A** is limited to one of four geometric isomers I–IV (Fig. 1). Structure I is eliminated by ^{31}P NMR data and structure II, with *trans*-carbonyl ligands, is inconsistent with the almost equal relative intensities observed for the two infrared active carbonyl stretching

TABLE 3

^{1}H NMR SPECTRAL DATA [a]

Complex	δ(Ph)	$\delta(C_7H_7)$	$\delta(CH_2)$	Solvent
A$_1$	7.16–7.76 [20H,m]	5.28 [7H,s]	5.16 [1H,m, J(H–H) 14, J(H–P) 9] 4.84 [1H,m, J(H–H) 14, J(H–P) 11]	$CDCl_3$
A$_2$	7.14–7.90 [20H,m]	5.22 (7H,s]	3.22 [2H,br], 2.70 [1H,br] 2.20 [1H,br]	$CDCl_3$ [b]
B	7.30–7.70 [20H,m]	5.93 [7H,d, J(H–P) 2]	3.90 [2H,d, J(H–P) 12]	acetone-d_6
C [c]	7.30–7.66 [20H,m]	5.36 [7H,t, J(H–P) 2]	6.02 [1H,m, J(H–H) 16, J(H–P) 10] 4.50 [1H,m, J(H–H) 16, J(H–P) 12]	CD_3CN
D	7.20–7.70 [20H,m]	5.49 [d] [7H,d, J(H–P) 2] 5.43 [e] [7H,d, J(H–P) 2]	2.00 [4H,br]	CD_3CN

[a] s = singlet, d = doublet, t = triplet, br = broad, J values in Hz. [b] Recorded at −10°C. [c] As $[PF_6]$ salt. [d] Molybdenum-bound C_7H_7. [e] Tungsten-bound C_7H_7.

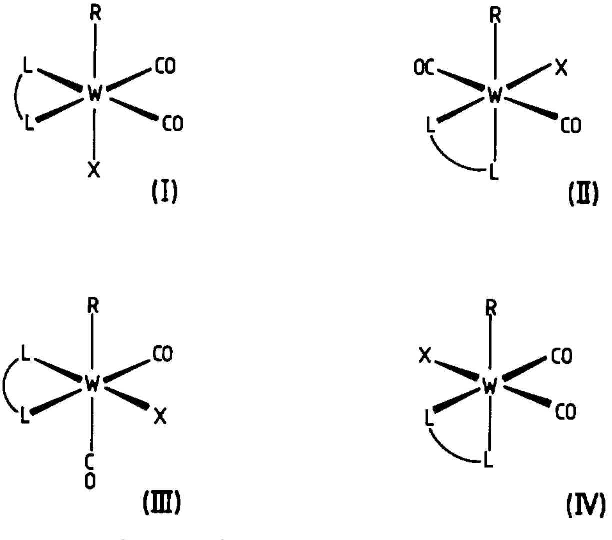

Fig. 1. R = η^3-allyl or η^3-cycloheptatrienyl.

frequencies [5]. The current work therefore limits the structure of complexes A to one of III or IV (Scheme 1 depicts structure IV but this should be considered as one of the two alternatives). Similar arguments have been advanced for the very closely related allyl complexes [MX(CO)$_2$(L–L)(η^3-C$_3$H$_5$)] (M = Mo, W; X = halide; L–L = bisphosphine) for which an X-ray crystallographic study on [MoCl(CO)$_2$(dppe)(η^3-C$_3$H$_5$)] has shown that the arrangement of ligands in structure IV is adopted [6].

A close structural similarity is evident from a comparison of the ^{31}P NMR data for [WI(CO)$_2$(dppm)(η^3-allyl)] [chemical shift separation of phosphorus resonances 21.9 ppm, J(P–P) 14.6, J(^{183}W–P) (low field) 193, J(^{183}W–P)(high field) 153 Hz] [6] and that for [WI(CO)$_2$(dppm)(η^3-C$_7$H$_7$)] (Table 2). Moreover the ^{31}P NMR spectra of both the allyl complexes [WI(CO)$_2$(L–L)(η^3-C$_3$H$_5$)] (L–L = dppm, dppe) and of the η^3-cycloheptatrienyl complex [WI(CO)$_2$(dppe)(η^3-C$_7$H$_7$)] are temperature dependent. In the case of [WI(CO)$_2$(dppe)(η^3-C$_7$H$_7$)], at 313 K the ^{31}P NMR spectrum shows only a broad singlet centred at δ 23.7 ppm which collapses to a well separated doublet on cooling to 263 K. Such observations in the ^{31}P NMR spectra of [WI(CO)$_2$(L–L)(η^3-C$_3$H$_5$)] (L–L = dppm, dppe) have been explained by a trigonal twist process [6] in which the two phosphorus environments are averaged via interconversion of the two optical isomers of structure IV (Fig. 1). Detailed studies on the dynamic ^{31}P NMR spectra of [MX(CO)$_2$(L–L)(η^3-C$_7$H$_7$)] (M = Mo, W; X = halide; L–L = bisphosphine) are now in progress as part of a programme of research to establish analogies in structure and reactivity between η^3-cycloheptatrienyl complexes and the corresponding, well studied, η^3-allyl complexes.

By comparison with the molybdenum derivatives, the complexes [WI(CO)$_2$(L–L)(η^3-C$_7$H$_7$)] (L–L = dppm, dppe) are notable for their stability with respect to loss of CO and conversion to η^7-cycloheptatrienyl derivatives. Thus, whilst [MoI(CO)$_2$(dppe)(η^3-C$_7$H$_7$)] readily forms [Mo(CO)(dppe)(η^7-C$_7$H$_7$)]I in refluxing benzene [1], [WI(CO)$_2$(dppe)(η^3-C$_7$H$_7$)] is stable even in refluxing toluene. Similarly reaction of [MoI(CO)$_2$(dppe)(η^3-C$_7$H$_7$)] with excess NH$_4$PF$_6$ in aqueous acetone gives the unidentate bisphosphine complex $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ [1] but $[WI(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$ is unreactive under these conditions.

The dppm complex A_1, with a strained, four-membered metal chelate ring, does however react slowly with NH_4PF_6 in aqueous acetone to give moderate yields of orange $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (**B**). Characterisation details for **B** are given in Tables 1, 2 and 3. The presence of unidentate dppm is confirmed by the ^{31}P NMR spectrum in which $^{183}W\text{-}P$ coupling, consistent with that reported for complexes in which tungsten is bound directly to phosphorus, is associated only with the low field resonance centred at 5.1 ppm. Carbonyl elimination from $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ can be induced in a slow reaction with $Me_3NO \cdot 2H_2O$ in acetone at room temperature to give low yields of green $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]I$ (**C**). However, complex **C** is better prepared as the pink $[PF_6]$ salt from the rapid reaction of $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ with $Me_3NO \cdot 2H_2O$ in acetone at room temperature. Details of the characterisation of $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]^+$ as both iodide and hexafluorophosphate salts are given in Tables 1, 2 and 3.

The unidentate bisphosphine complex $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ and its molybdenum analogues, $[Mo(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ and $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ have further synthetic potential in the formation of phosphine bridged bimetallics. The dppe bridged bimetallic $[\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}_2(\mu\text{-}dppe)][PF_6]_2$ has been reported to result from direct reaction of dppe with $[Mo(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ but yields are low and a mixture of products is formed [2]. In a new approach the current work has investigated the reactivity of $[M(CO)_2(L\text{-}L)(\eta^7\text{-}C_7H_7)][PF_6]$ (M = W, L–L = dppm; M = Mo, L–L = dppm, dppe) as an organometallic phosphine ligand towards $[M(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ (M = Mo, W). Both tricarbonyl complexes $[M(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ (M = Mo, W) are labile to replacement of one carbonyl ligand upon reaction with a variety of Group V donor ligands in refluxing ethanol (95% grade) [4,7]. Thus reaction of $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ with $[Mo(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ in refluxing ethanol gives moderate yields of $[\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}_2(\mu\text{-}dppe)][PF_6]_2$ which was identified by reference to published data [2]. Similarly reaction of $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ with $[W(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ provides a unique synthetic route to the heterobimetallic $[\{W(CO)_2(\eta^7\text{-}C_7H_7)\}(\mu\text{-}dppe)\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}][PF_6]_2$ (**D**) which was isolated as an orange solid and characterised as shown in Tables 1, 2 and 3. The phosphine-bridged structure of **D** is confirmed by comparison of the ^{31}P NMR data obtained with that found for $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ and $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (Table 2) and that reported for $[\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}_2(\mu\text{-}dppe)][PF_6]_2$ [2]. The doublet at 35.8 ppm in the ^{31}P NMR spectrum of **D**, assigned to the molybdenum-bound phosphorus, is closely comparable with the chemical shift of 35.7 ppm reported for phosphorus in dppe-bridged $[\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}_2(\mu\text{-}dppe)][PF_6]_2$ and the resonance assigned to the tungsten-bound phosphorus [$\delta(P)$ 8.1 ppm, $J(^{183}W\text{-}P)$ 337 Hz] also compares well with that in $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ [$\delta(P)$ 5.1 ppm, $J(^{183}W\text{-}P)$ 342 Hz]. The phosphorus–phosphorus coupling constant for $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ [J(P–P) 37 Hz] is reduced to 31 Hz in complex **D**.

By contrast with the above work the corresponding reactions of the dppm derivatives $[M(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (M = Mo, W) with $[Mo(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ resulted only in the isolation of $[M(CO)(dppm)(\eta^7\text{-}C_7H_7)][PF_6]$ (M = Mo, W) with no evidence for the formation of dppm-bridged bimetallics.

Experimental

The preparation, purification and reactions of the complexes described were carried out under an atmosphere of dry nitrogen. All solvents were dried by standard methods, distilled and deoxygenated before use. The complexes $[W(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ [8], $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ [8] and $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ [1] were prepared by published procedures.

Infrared spectra were recorded on a Pye Unicam SP3-200 spectrophotometer and calibrated against the absorption band of polystyrene at 1601 cm^{-1}. ^1H NMR spectra were recorded on a Varian Associates SC 300 instrument and calibrated against $SiMe_4$; ^{31}P NMR spectra were recorded at 32.4 MHz on a Bruker WP 80 instrument and calibrated against 85% H_3PO_4. Microanalyses were by the staff of the Microanalytical Service of the Department of Chemistry, University of Manchester.

*Preparation of $[WI(CO)_2(L\text{-}L)(\eta^3\text{-}C_7H_7)]$ (L–L = dppm, dppe) (**A**)*

To a stirred solution of $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ (1.19 g, 2.60 mmol) in toluene (80 cm^3) was added dppm (1.00 g, 2.60 mmol). After 48 h the resulting red solution was evaporated to dryness to give a red-brown residue which was recrystallised from CH_2Cl_2/n-hexane to give the product $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ as a red solid; yield 1.43 g (65%).

$[WI(CO)_2(dppe)(\eta^3\text{-}C_7H_7)]$ was prepared similarly from $[WI(CO)_2(\eta^7\text{-}C_7H_7)]$ (1.08 g, 2.36 mmol) and dppe (0.94 g, 2.36 mmol) and obtained as a red solid; yield 1.84 g (91%).

*Preparation of $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (**B**)*

A solution of NH_4PF_6 (7.0 g, 43 mmol) in distilled water (40 cm^3) was added to a stirred solution of $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ (1.14 g, 1.35 mmol) in acetone (100 cm^3). Further acetone was added until the $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ just dissolved then the reaction mixture was stirred at room temperature for 2 d. After removal of acetone in vacuo the precipitated product was collected by filtration, dried, and recrystallised from CH_2Cl_2/toluene to remove unreacted starting material. A final recrystallisation from CH_2Cl_2/n-hexane gave the pure product as an orange solid, yield 0.46 g (40%).

Reaction of $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ with $Me_3NO \cdot 2H_2O$

To a stirred solution of $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$ (0.25 g, 0.30 mmol) in acetone (30 cm^3) was added $Me_3NO \cdot 2H_2O$ (0.071 g, 0.64 mmol). After stirring at room temperature for 18 h the product, $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]I$ was present as a green precipitate whilst the mother liquors contained unreacted $[WI(CO)_2(dppm)(\eta^3\text{-}C_7H_7)]$. Removal of the mother liquors and recrystallisation of the green precipitate from CH_2Cl_2/n-hexane gave $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)]I$ as a green solid; yield 0.015 g (6%). The complex is insoluble in acetone and only very sparingly soluble in CH_2Cl_2.

*Preparation of $[W(CO)(dppm)(\eta^7\text{-}C_7H_7)][PF_6]$ (**C**)*

To a stirred solution of $[W(CO)_2(dppm\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (0.31 g, 0.36 mmol) in acetone (50 cm^3) was added $Me_3NO \cdot 2H_2O$ (0.045 g, 0.40 mmol). A rapid colour change from red to pink resulted and after 45 min acetone was removed in vacuo to give a pink-red residue which was recrystallised from CH_2Cl_2/diethyl ether to give the product as a pink solid; yield 0.11 g (37%).

Preparation of $[\{W(CO)_2(\eta^7\text{-}C_7H_7)\}(\mu\text{-dppe})\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}][PF_6]_2$ *(D)*

A suspension of $[W(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ (0.41 g, 0.81 mmol) and $[Mo(CO)_2\text{-}(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (0.63 g, 0.80 mmol) in 95% ethanol (40 cm^3) was refluxed gently for 3 h. An orange precipitate formed which was collected by filtration, washed with ethanol and CH_2Cl_2 and finally recrystallised from CH_3CN/diethyl ether to give the product as an orange solid; yield 0.38 g (37%).

$[\{Mo(CO)_2(\eta^7\text{-}C_7H_7)\}_2(\mu\text{-dppe})][PF_6]_2$ was similarly prepared in 41% yield from $[Mo(CO)_3(\eta^7\text{-}C_7H_7)][PF_6]$ (0.39 g, 0.94 mmol) and $[Mo(CO)_2(dppe\text{-}P)(\eta^7\text{-}C_7H_7)][PF_6]$ (0.68 g, 0.87 mmol) under reflux in 95% ethanol (25 cm^3) for 1 h.

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