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SYNTHESIS OF SOME NOVEL MIXED SEVEN-COORDINATE DIIODOTRICARBONYL (TRIPHENYLPHOSPHITE) TRIPHENYLPHOSPHINE, ARSINE AND ANTIMONY COMPLEXES OF MOLYBDENUM(II) AND TUNGSTEN(II)

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COMMUNICATION

SYNTHESIS OF SOME NOVEL MIXED SEVEN-COORDINATE DIODOTRICARBONYL (TRIPHENYLPHOSPHITE)TRIPHENYLPHOSPHINE, ARSINE AND ANTIMONY COMPLEXES OF MOLYBDENUM(II) AND TUNGSTEN(II)

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INTRODUCTION

The complexes $[MX_2(CO)_3L_2]$ ($M = Mo$ and W ; $L = PPh_3$ and $AsPh_3$; $X = Cl$ and Br) are currently under investigation as catalysts for the ring-opening polymerisation of norbornene,^{1,2} and it is proposed that it is the ease of phosphine or arsine dissociation in these complexes which governs the rate determining step in the mechanism. Although a wide variety of bis-phosphine complexes of the type $[MX_2(CO)_3L_2]$ ($M = Mo$ and W ; $X = Cl, Br$ and I ; $L =$ phosphine) have been reported,³ hitherto no examples of "mixed" phosphine/phosphite complexes of this type have been prepared. In this communication we wish to report the synthesis of the new "mixed" seven-coordinate complexes $[MI_2(CO)_3L\{P(OPh)_3\}]$ ($M = Mo$ and W ; $L = PPh_3, AsPh_3$ and $SbPh_3$).

EXPERIMENTAL

$[MI_2(CO)_3(NCMe)_2]$ ($M = Mo$ and W) were prepared according to literature methods⁴ and $M(CO)_6$, PPh_3 , $AsPh_3$, $SbPh_3$ and $P(OPh)_3$ were purchased from commercial sources. Dichloromethane was distilled before use.

$MoI_2(CO)_3(PPh_3)\{P(OPh)_3\}$

To $MoI_2(CO)_3(NCMe)_2$ (0.23g, 0.446 mmol) dissolved in CH_2Cl_2 (15cm³), with continuous stirring under a stream of dry argon, was added PPh_3 (0.117g, 0.446 mmol). After stirring for one minute, $P(OPh)_3$ (0.138g, 0.445 mmol) was added and the mixture was stirred for a further 30 minutes. After filtration, removal of the solvent *in vacuo* gave the dark yellow crystalline complex $MoI_2(CO)_3(PPh_3)\{P(OPh)_3\}$, (yield = 0.36g, 80%), which was recrystallised from CH_2Cl_2 .

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TABLE I
 Analytical (C, H and N) and IR data^a for $[\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]^b$

	<i>M</i>	<i>L</i>	Colour	Yield %	Found (Calcd.)%	$\nu(\text{CO})^a$ cm^{-1}
(1)	Mo	PPh_3	Dark yellow	80	C 47.0 (46.55) H 3.19 (3.00)	2040(m), 1981(s) and 1910(s)
(2)	W	PPh_3	Yellow	67	C 42.90 (42.81) H 2.79 (2.76)	2040(m), 1970(s) and 1934(s)
(3)	Mo	AsPh_3	Dark yellow	85	C 44.84 (44.60) H 3.06 (2.88)	2045(m), 1982(s) and 1960(s)
(4)	W	AsPh_3	Bright yellow	78	C 41.50 (41.15) H 2.68 (2.66)	2040(s), 1975(s) and 1939(s)
(5)	Mo	SbPh_3	Orange	87	C 42.45 (42.70) H 2.88 (2.76)	2025(s), 1985(s) and 1961(s)
(6)	W	SbPh_3	Orange	85	C 39.24 (39.53) H 2.88 (2.55)	2015(m), 1977(s) and 1949(s)

^aSpectra recorded in CHCl_3 unless stated; m, medium; s, strong. ^bReaction times for $\text{L} + [\text{M}_2(\text{CO})_3(\text{NCMe})_2] \rightarrow [\text{M}_2(\text{CO})_3(\text{NCMe})\text{L}]$ and $\text{P}(\text{O}Ph)_3 \rightarrow [\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]$ are (1): PPh_3 , 1 minute, $\text{P}(\text{O}Ph)_3$, 30 minutes; (3): AsPh_3 , 3 minutes, $\text{P}(\text{O}Ph)_3$, 30 minutes; (5): SbPh_3 , 7 minutes, $\text{P}(\text{O}Ph)_3$, 30 minutes. Similar times were recorded for the tungsten complexes (2), (4) and (6).

Similar reactions of $[\text{M}_2(\text{CO})_3(\text{NCMe})_2]$ with *L* followed by $\text{P}(\text{O}Ph)_3$ gave the new compounds $[\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]$ (see Table I for reaction times).

RESULTS AND DISCUSSION

The mixed complexes are prepared by reaction of $[\text{M}_2(\text{CO})_3(\text{NCMe})_2]^4$ with *L* in CH_2Cl_2 , followed by reaction *in situ* with $\text{P}(\text{O}Ph)_3$ to give high yields of the new complexes, $[\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]$. It should be noted that the isolated solids, $[\text{M}_2(\text{CO})_3(\text{NCMe})\text{L}]^5$ also react with $\text{P}(\text{O}Ph)_3$ in CH_2Cl_2 to give $[\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]$. Elemental analysis (C, H and N) and infrared spectroscopy (Table I) confirms the formation of the new compounds $[\text{M}_2(\text{CO})_3\text{L}\{\text{P}(\text{O}Ph)_3\}]$ which are stable in the solid state when stored under argon.

In view of the work of Bencze *et al.*,^{1,2} we are investigating the catalytic activity of these complexes. Further studies are in progress.

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