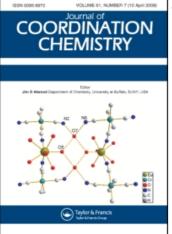
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SYNTHESIS OF SOME NOVEL MIXED SEVEN-COORDINATE DIIODOTRICARBONYL (TRIPHENYLPHOSPHITE) TRIPHENYLPHOSPHINE, ARSINE AND ANTIMONY COMPLEXES OF MOLYBDENUM(II) AND TUNGSTEN(II) Paul K. Baker^a; Stuart G. Fraser^a

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COMMUNICATION

SYNTHESIS OF SOME NOVEL MIXED SEVEN-COORDINATE DIIODOTRICARBONYL (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 = PPh₃ and AsPh₃; 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 = PPh₃, AsPh₃ and SbPh₃).

EXPERIMENTAL

 $[MI_2(CO)_3(NCMe)_2]$ (M = Mo and W) were prepared according to literature methods⁴ and M(CO)₆, PPh₃, AsPh₃, SbPh₃ and P(OPh)₃ were purchased from commercial sources. Dichloromethane was distilled before use.

$Mol_2(CO)_3(PPh_3)\{P(OPh_3)\}$

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 PPh₃ (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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	М	L	Colour	Yield %	Found (Calcd.)%	$\nu(CO)^a \text{ cm}^{-1}$
1)	Мо	PPh,	Dark yellow	80	C 47.0 (46.55) H 3.19 (3.00)	2040(m), 1981(s) and 1910(s)
2)	W	PPh,	Yellow	67	C 42.90 (42.81) H 2.79 (2.76)	2040(m), 1970(s) and 1934(s)
3)	Мо	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)

TABLE I Analytical (C, H and N) and IR data^a for [MI,(CO),L{P(OPh),})^b

^aSpectra recorded in CHCl₃ unless stated; m, medium; s, strong. ^bReaction times for $L + [MI_3(CO)_3(NCMe)_3] \rightarrow [MI_2(CO)_3(NCMe)_1] \rightarrow [MI_2(CO)_3(NCMe)_1] \rightarrow [MI_2(CO)_3(NCMe)_1] \rightarrow [MI_2(CO)_3(NCMe)_3]$ are (1): PPh₃, 1 minute, P(OPh)₃, 30 minutes; (3): AsPh₃, 3 minutes, P(OPh)₃, 30 minutes; (5): SbPh₃, 7 minutes, P(OPh)₃, 30 minutes. Similar times were recorded for the tungsten complexes (2), (4) and (6).

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L followed by P(OPh)₃ gave the new compounds $[MI_2(CO)_3L\{P(OPh)_3\}]$ (see Table I for reaction times).

RESULTS AND DISCUSSION

The mixed complexes are prepared by reaction of $[MI_2(CO)_3(NCMe)_2]^4$ with L in CH_2Cl_2 , followed by reaction *in situ* with P(OPh)₃ to give high yields of the new complexes, $[MI_2(CO)_3L\{P(OPh)_3\}]$. It should be noted that the isolated solids, $[MI_2(CO)_3(NCMe)L]$,⁵ also react with P(OPh)₃ in CH_2Cl_2 to give $[MI_2(CO)_3L\{P(OPh)_3\}]$. Elemental analysis (C, H and N) and infrared spectroscopy (Table I) confirms the formation of the new compounds $[MI_2(CO)_3L\{P(OPh)_3\}]$ 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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