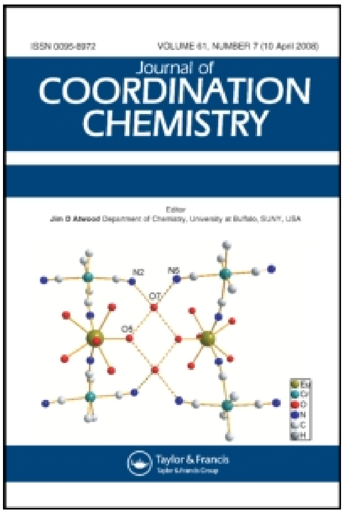

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SYNTHESIS OF SOME NOVEL MIXED SEVEN-COORDINATE DIIODOTRICARBONYL (TRIPHENYLPHOSPHITE) TRIPHENYLPHOSPHINE, ARSINE AND ANTIMONY COMPLEXES OF MOLYBDENUM(II) AND TUNGSTEN(II)

Paul K. Baker[a]; Stuart G. Fraser[a]
[a] Department of Chemistry, University College of North Wales, Bangor, Gwynedd, U.K.

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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)

PAUL K. BAKER† and STUART G. FRASER
Department of Chemistry, University College of North Wales, Bangor, Gwynedd LL57 2UW, U.K.

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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 methods[4] 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 (15cm^3), 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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†Author for correspondence.

TABLE I
Analytical (C, H and N) and IR data[a] for $[MI_2(CO)_3L\{P(OPh)_3\}]$[b]

	M	*L*	Colour	Yield %	Found (Calcd.)%	ν(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)

[a]Spectra recorded in $CHCl_3$ unless stated; m, medium; s, strong. [b]Reaction times for L + $[MI_2(CO)_3(NCMe)_2]$ → $[MI_2(CO)_3(NCMe)L]$ and $P(OPh)_3$ → $[MI_2(CO)_3L\{P(OPh)_3\}]$ are (1): PPh_3, 1 minute, $P(OPh)_3$, 30 minutes; (3): $AsPh_3$, 3 minutes, $P(OPh)_3$, 30 minutes; (5): $SbPh_3$, 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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REFERENCES

1. L. Bencze and A. Kraut-Vass, *J. Mol. Catal.*, **28**, 369 (1985).
2. L. Bencze, A. Kraut-Vass and L. Prókai, *J. Chem. Soc., Chem., Commun.*, 911 (1985).
3. R. Colton, *Coord. Chem. Rev.*, **6**, 269 (1971) and references cited therein.
4. P.K. Baker, S.G. Fraser and E.M. Keys, *J. Organometal. Chem.*, in press.
5. P.K. Baker and S.G. Fraser, *Polyhedron*, in press.