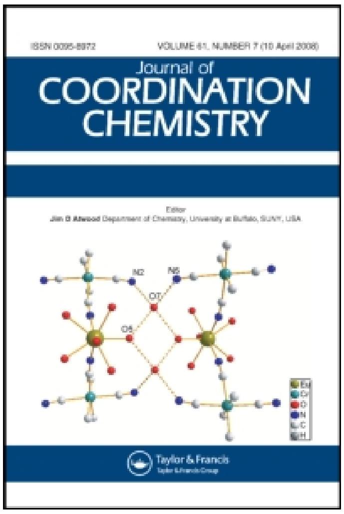

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The preparation and spectral properties of the new 'mixed' complexes $[MI_2(CO)_3(py)L]$ (M = Mo and W; L = PPh_3, $AsPh_3$ and $SbPh_3$)

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SHORT COMMUNICATION

The preparation and spectral properties of the new "mixed" complexes $[MI_2(CO)_3(py)L]$ (M = Mo and W; L = PPh_3, $AsPh_3$ and $SbPh_3$).

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Seven-coordinate complexes of molybdenum(II) and tungsten(II) have become increasingly important as homogeneous catalysts. For example, the complexes $[MX_2(CO)_3L_2]$ (M = Mo and W; X = Cl and Br; L = PPh_3 and $AsPh_3$) have been shown to be catalysts for the ring-opening polymerisation of norbornene.[1] Although a wide variety of complexes of the type $[MX_2(CO)_3L_2]$ (M = Mo and W; X = Cl, Br and I; L = nitrogen, phosphorus, arsenic and antimony donor ligands)[2] have been reported, until now no examples of the mixed complexes $[MX_2(CO)_3(py)L]$ have been prepared. In this communication we wish to describe the synthesis of the new mixed pyridine/L compounds $[MI_2(CO)_3(py)L]$ (M = Mo and W; L = PPh_3, $AsPh_3$ and $SbPh_3$).

Key words: Molybdenum, tungsten, complexes, synthesis, catalysts

EXPERIMENTAL

$[MI_2(CO)_3(NCMe)_2]$ (M = Mo and W) were prepared according to literature methods[3] and $M(CO)_6$, PPh_3, $AsPh_3$, $SbPh_3$ and py were purchased from commercial sources. Dichloromethane was distilled before use.

$MoI_2(CO)_3(py)(PPh_3)$

To $[MoI_2(CO)_3(NCMe)_2]$ (0.222g, 0.430mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of dry argon was added PPh_3 (0.113g, 0.431mmol). After stirring for one minute, py (0.034g, 0.430mmol) was added and the mixture was stirred for a further 9 minutes. After filtration, removal of the solvent *in vacuo* gave the analytically pure bright yellow crystalline complex $[MoI_2(CO)_3(py)(PPh_3)]$ (yield = 0.23g, 69.0%).

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L followed by py gave the new compounds $[MI_2(CO)_3(py)L]$ (see Table I for reaction times).

RESULTS AND DISCUSSION

Equimolar quantities of $[MI_2(CO)_3(NCMe)_2]$ (M = Mo and W)[3] and L (L = PPh_3, $AsPh_3$ and $SbPh_3$) react in CH_2Cl_2 to give $[MI_2(CO)_3(NCMe)L]$, which is subsequently reacted *in situ* with pyridine to afford high yeilds of the new complexes $[MI_2(CO)_3(py)L]$.

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TABLE I
Analytical and i.r. data[a] for $[MI_2(CO)_3(py)L]$[b] complexes

	M	L	Colour	Yield	Found (Calcd.)	$\nu(CO)$[a] cm^{-1}
(1)	Mo	PPh_3	Yellow	69.0	C 39.8 (40.3) H 2.7 (2.6) N 2.0 (1.8)	2020(s), 1955(s) 1915(s)
(2)	W	PPh_3	Yellow	66.9	C 36.3 (36.2) H 2.5 (2.3) H 1.4 (1.6)	2025(s), 1949(s) 1900(s)
(3)	Mo	$AsPh_3$	Yellow	43.6	C 38.3 (38.1) H 2.6 (2.5) N 2.,0 (1.7)	2030(s), 1963(s) 1915(s)
(4)	W	$AsPh_3$	Orange	57.9	C 34.2 (34.4) H 2.2 (2.2) N 1.7 (1.5)	2030(s), 1948(s) 1895(s)
(5)	Mo	$SbPh_3$	Orange	64.5	C 35.8 (36.1) H 2.4 (2.3) N 1.4 (1.6)	2030(s), 1965(s) 1905(s)
(6)	W	$SbPh_3$	Orange	71.9	C 32.7 (32.7) H 2.4 (2.1) N 1.7 (1.5)	2020(s), 1955(s) 1900(s)

[a] Spectra recorded in $CHCl_3$: s, strong. [b] Reaction times for L + $[MI_2(CO)_3(NCMe)_2] \rightarrow [MI_2(CO)_3(NCMe)L]$ and py $\rightarrow$ $[MI_2(CO)_3(py)L]$: (1) PPh_3, 1 minute; py, 9 minutes. (3) $AsPh_3$, 3 minutes; py, 22 minutes. (5) $SbPh_3$, 5 minutes; py, 20 minutes. Similar times were recorded for the tungsten complexes (2), (4) and (6).

These complexes are stable in the solid state when stored under argon and have been characterized by elemental analysis and i.r. spectroscopy (Table 1). The ^1H n.m.r. spectra showed resonances for both pyridine and L. It should be noted that reaction of the isolated solids, $[MI_2(CO)_3(NCMe)L]$[4] with py in CH_2Cl_2 also gives the new compounds $[MI_2(CO)_3(py)L]$. The carbonyl stretching frequencies of the mixed complexes $[MI_2(CO)_3(py)L]$ are similar to their py_2[5] and L_2[6] analogues; their structures are likely to be capped octahedra in view of the X-ray crystal structures which have been determined for other analogous seven-coordinate molybdenum(II) and tungsten(II) compounds.[7]

We are currently investigating the chemistry of these new complexes, in particular their possible uses as homogeneous catalysts as a result of the work of Bencze *et al.*, recently published.[1]

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