Inorganica Chimica Acta, 148 (1988) 247–250

Synthesis of some Mixed Seven-coordinate Complexes of the Type $[MI_2(CO)_3LL']$ (M = Mo or W; L = Pyridine or Substituted Pyridines; L′ = PPh_3, $AsPh_3$ or $SbPh_3$)

PAUL K. BAKER*, STUART G. FRASER and MARTIN J. SNOWDEN

Department of Chemistry, University College of North Wales, Bangor, Gwynedd LL57 2UW, U.K.

(Received December 24, 1987)

Abstract

The seven-coordinate bisacetonitrile complexes $[MI_2(CO)_3(NCMe)_2]$ (M = Mo or W) react with L′ (L′ = PPh_3, $AsPh_3$ or $SbPh_3$) in CH_2Cl_2 at room temperature to give $[MI_2(CO)_3(NCMe)L']$ which when reacted *in situ* with L (L = pyridine or substituted pyridines) affords good yields of 28 mixed seven-coordinate complexes $[MI_2(CO)_3LL']$. It is likely these reactions occur via successive dissociative displacements of two acetonitrile ligands.

Introduction

The importance of seven-coordinate complexes of molybdenum(II) and tungsten(II) has been recently emphasised by the ability of complexes of the type $[MX_2(CO)_3L_2]$ (M = Mo or W; X = Cl or Br; L = PPh_3 or $AsPh_3$) to promote the ring-opening polymerisation of norbornene and norbornadiene [1, 2]. Although many examples of bisligand complexes of the type $[MX_2(CO)_3L_2]$ (M = Mo or W; X = Cl, Br or I; L = neutral nitrogen, phosphorus, arsenic, antimony etc., donor ligands) have been reported [3–18], previous to our work the only mixed ligand complexes of the type $[MX_2(CO)_3LL']$ to be prepared were the complexes $[WX_2(CO)_3LL']$ (X = Cl, Br or I; L, L′ = PMe_3, $AsMe_2$, $SbMe_3$, $AsMe_2H$, $P(OMe)_3$ or py) by Umland and Vahrenkamp in 1982 [19]. They prepared these complexes by reacting $[WX_2(CO)_4L]$ with L′.

Our recent research utilising the highly versatile complexes $[MI_2(CO)_3(NCMe)_2]$ (M = Mo or W) [20] allows a simple synthesis of new mixed seven-coordinate diiodo complexes of the type $[MI_2(CO)_3LL']$. In this paper we wish to describe the preparation of a number of new seven-coordinate complexes of the type $[MI_2(CO)_3LL']$ (M = Mo or W; L = pyridine or substituted pyridines; L′ = PPh_3, $AsPh_3$ or $SbPh_3$). Preliminary results of this work have been published in an earlier communication [21].

Experimental

All reactions described in this paper were carried out using standard Schlenk line techniques. The seven-coordinate compounds $[MI_2(CO)_3(NCMe)_2]$ (M = Mo or W) were prepared by the published method [20]. All chemicals used were purchased from commercial sources. CH_2Cl_2 was dried and distilled before use.

Elemental analyses for carbon, hydrogen and nitrogen were recorded on a Carlo Erba Elemental Analyser MOD 1106 (using helium as a carrier gas). Infrared spectra were recorded on a Perkin-Elmer 197 infrared spectrophotometer.

$MoI_2(CO)_3(py)(PPh_3)$ (1)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.222 g, 0.430 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of dry nitrogen was added PPh_3 (0.113 g, 0.431 mmol). After stirring for 1 min, py (0.034 g, 0.430 mmol) was added and the mixture was stirred for a further 9 min. After filtration, removal of the solvent *in vacuo* gave the analytically pure bright yellow crystalline complex $[MoI_2(CO)_3(py)(PPh_3)]$ (**1**) (yield = 0.23 g, 69%).

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L′ followed by py gave the complexes $[MI_2(CO)_3(py)L']$ (**1**–**6**). Reaction times for $[MI_2(CO)_3(NCMe)_2]$ + L′ → $[MI_2(CO)_3(NCMe)L']$ + py → $[MI_2(CO)_3(py)L']$: **1** PPh_3, 1 min; py, 9 min; **2** $AsPh_3$, 3 min; py, 22 min; **3** $SbPh_3$, 7 min; py, 20 min. Similar times were recorded for the tungsten complexes **4**, **5** and **6**.

$MoI_2(CO)_3(2Me\text{-}py)(PPh_3)$ (7)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.5 g, 0.969 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of dry nitrogen was added PPh_3 (0.254 g, 0.969 mmol). After stirring for 1 min, 2 Me-py (0.09 g, 0.969 mmol) was added and the mixture stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the brown crystalline complex $[MoI_2(CO)_3(2Me\text{-}py)(PPh_3)]$ (**7**) (yield = 0.57 g, 75%) which was recrystallised from CH_2Cl_2.

---

*Author to whom correspondence should be addressed.

0020-1693/88/$3.50

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L′ followed by 2Me-py gave the new compounds $[MI_2(CO)_3(2Me\text{-}py)L']$ (**7**–**11**). Reaction times for $[MI_2(CO)_3(NCMe)_2]$ + L′ → $[MI_2(CO)_3(NCMe)L']$ + 2Me-py → $[MI_2(CO)_3(2Me\text{-}py)L']$: **7** PPh_3, 1 min; 2Me-py, 30 min; **8** $AsPh_3$, 3 min; 2Me-py, 30 min; **9** $SbPh_3$, 7 min; 2Me-py, 30 min; **10** PPh_3, 1 min; 2Me-py, 30 min; **11** $SbPh_3$, 7 min; 2Me-py, 30 min.

$WI_2(CO)_3(4Me\text{-}py)(PPh_3)$ (15)

To $[WI_2(CO)_3(NCMe)_2]$ (0.3 g, 0.497 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of dry nitrogen was added PPh_3 (0.13 g, 0.497 mmol). After stirring for 1 min, 4Me-py (0.047 g, 0.505 mmol) was added and the mixture stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the brown crystalline complex $[WI_2(CO)_3(4Me\text{-}py)(PPh_3)]$ (**15**) (yield = 0.34 g, 78%), which was recrystallised from CH_2Cl_2.

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L′ followed by 4Me-py gave the new complexes $[MI_2(CO)_3(4Me\text{-}py)L']$ (**12**–**17**). Reaction times for $[MI_2(CO)_3(NCMe)_2]$ + L′ → $[MI_2(CO)_3(NCMe)L']$ + 4Me-py → $[MI_2(CO)_3(4Me\text{-}py)L']$: **12** PPh_3, 1 min; 4Me-py, 30 min; **13** $AsPh_3$, 3 min; 4Me-py, 30 min; **14** $SbPh_3$, 7 min; 4Me-py, 30 min. Similar times were recorded for the tungsten compounds **15**–**17**.

$MoI_2(CO)_3(3,5\text{-}Me_2\text{-}py)(PPh_3)$ (18)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.4 g, 0.775 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of nitrogen was added PPh_3 (0.203 g, 0.775 mmol). After stirring for 1 min, 3,5-Me_2-py (0.0829 g, 0.775 mmol) was added and the mixture was stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the orange/brown crystalline complex $[MoI_2(CO)_3(3,5\text{-}Me_2\text{-}py)(PPh_3)]$ (**18**) (yield = 0.60 g, 97%), which was recrystallised from CH_2Cl_2.

A similar reaction of $[MoI_2(CO)_3(NCMe)_2]$ with $AsPh_3$ (3 min) followed by 3,5-Me_2-py (30 min) gave the new compound $[MoI_2(CO)_3(3,5\text{-}Me_2\text{-}py)(AsPh_3)]$ (**19**).

$MoI_2(CO)_3(2Cl\text{-}py)(PPh_3)$ (20)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.5 g, 0.969 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under nitrogen was added PPh_3 (0.254 g, 0.969 mmol). After stirring for 1 min, 2Cl-py (0.11 g, 0.969 mmol) was added and the mixture was stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the dark brown crystalline complex $[MoI_2(CO)_3(2Cl\text{-}py)(PPh_3)]$ (**20**) (yield = 0.63 g, 80%), which was recrystallised from CH_2Cl_2.

Similar reactions of $[MI_2(CO)_3(NCMe)_2]$ with L′ followed by 2Cl-py gave the new compounds $[MI_2(CO)_3(2Cl\text{-}py)L']$ (**20**–**25**). Reaction times for $[MI_2(CO)_3(NCMe)_2]$ + L′ → $[MI_2(CO)_3(NCMe)L']$ → $[MI_2(CO)_3(NCMe)L']$ + 2Cl-py → $[MI_2(CO)_3(2Cl\text{-}py)L']$: **20** PPh_3, 1 min; 2Cl-py, 30 min; **21** $AsPh_3$, 3 min; 2Cl-py, 30 min; **22** $SbPh_3$, 7 min; 2Cl-py, 30 min. Similar times were recorded for the tungsten compounds **23**–**25**.

$MoI_2(CO)_3(3Cl\text{-}py)(PPh_3)$ (26)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.4 g, 0.775 mmol) dissolved in CH_2Cl_2 (15 cm^3) with continuous stirring under a stream of nitrogen was added PPh_3 (0.203 g, 0.775 mmol). After stirring for 1 min 3Cl-py (0.088 g, 0.775 mmol) was added and the mixture stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the brown crystalline complex $[MoI_2(CO)_3(3Cl\text{-}py)(PPh_3)]$ (**26**) (yield = 0.59 g, 94%), which was recrystallised from CH_2Cl_2.

A similar reaction of $[MoI_2(CO)_3(NCMe)_2]$ with $AsPh_3$ (3 min) folowed by 3Cl-py (30 min) gave the new compound $[MoI_2(CO)_3(3Cl\text{-}py)(AsPh_3)]$ (**27**).

$[MoI_2(CO)_3(3Br\text{-}py)(AsPh_3)]$ (28)

To $[MoI_2(CO)_3(NCMe)_2]$ (0.4 g, 0.775 mmol) dissolved in CH_2Cl_2 (15 cm^3) was added $AsPh_3$ (0.237 g, 0.775 mmol). After stirring the solution for 3 min, 3Br-py (0.123 g, 0.779 mmol) was added and the mixture stirred for a further 30 min. After filtration, removal of the solvent *in vacuo* gave the yellow–brown crystalline complex $[MoI_2(CO)_3(3Br\text{-}py)(AsPh_3)]$ (yield = 0.61 g, 88%).

Results and Discussion

The starting materials for this research, $[MI_2(CO)_3(NCMe)_2]$ (M = Mo or W) are prepared by reacting the trisacetonitrile compounds $[M(CO)_3(NCMe)_3]$ (prepared by refluxing $[M(CO)_6]$ in acetonitrile [22]) with an equimolar quantity of I_2 [20]. The complexes $[MI_2(CO)_3(NCMe)_2]$ react with an equimolar quantity of L′ (L′ = PPh_3, $AsPh_3$ or $SbPh_3$) in CH_2Cl_2 at room temperature to give the monoacetonitrile seven-coordinate complexes $[MI_2(CO)_3(NCMe)L']$. These compounds when reacted *in situ* with L (L = pyridine or substituted pyridines) afford the mixed seven-coordinate complexes $[MI_2(CO)_3LL']$ (**1**–**28**) in high yield. Since the complexes $[MI_2(CO)_3(NCMe)_2]$ are prepared in quantitative yield from $[M(CO)_6]$ [20], these compounds are proving to be excellent precursors for synthesising seven-coordinate diiodo complexes of molybdenum(II) and tungsten(II). The mixed compounds $[MI_2(CO)_3LL']$ (**1**–**28**) were characterised by elemental analyses (C, H and N) (Table I) and infrared spectroscopy (Table II). All the complexes are stable in the solid state when stored under nitrogen. However, they decompose in solution when

TABLE I. Physical and Analytical Data[a] for the Complexes $[MI_2(CO)_3LL']$

Complex	Colour	Yield (%)	Analysis (%)[a]		
			C	H	N
1 $[MoI_2(CO)_3(py)(PPh_3)]$	yellow	69	39.8(40.3)	2.7(2.6)	2.0(1.8)
2 $[MoI_2(CO)_3(py)(AsPh_3)]$	yellow	44	38.3(38.1)	2.6(2.5)	2.0(1.7)
3 $[MoI_2(CO)_3(py)(SbPh_3)]$	orange	65	35.8(36.1)	2.4(2.3)	1.4(1.6)
4 $[WI_2(CO)_3(py)(PPh_3)]$	yellow	67	36.3(36.2)	2.5(2.3)	1.4(1.6)
5 $[WI_2(CO)_3(py)(AsPh_3)]$	orange	58	34.2(34.4)	2.2(2.2)	1.7(1.5)
6 $[WI_2(CO)_3(py)SbPh_3)]$	orange	72	32.7(32.7)	2.4(2.1)	1.7(1.5)
7 $[MoI_2(CO)_3(2Me\text{-}py)(PPh_3)]$	brown	75	40.2(41.1)	3.0(2.8)	1.6(1.8)
8 $[MoI_2(CO)_3(2Me\text{-}py)(AsPh_3)]$	yellow–brown	74	39.6(38.9)	2.3(2.6)	1.5(1.7)
9 $[MoI_2(CO)_3(2Me\text{-}py)(SbPh_3)]$	orange–brown	66	36.5(36.8)	2.8(2.5)	1.6(1.6)
10 $[WI_2(CO)_3(2Me\text{-}py)(PPh_3)]$	yellow–brown	71	36.8(36.9)	2.8(2.5)	1.6(1.6)
11 $[WI_2(CO)_3(2Me\text{-}py)(SbPh_3)]$	dark brown	62	33.8(33.5)	2.2(2.3)	1.4(1.5)
12 $[MoI_2(CO)_3(4Me\text{-}py)(PPh_3)]$	brown	68	41.5(41.1)	3.1(2.8)	1.6(1.8)
13 $[MoI_2(CO)_3(4Me\text{-}py)(AsPh_3)]$	yellow–brown	70	39.6(38.9)	3.0(2.6)	1.8(1.7)
14 $[MoI_2(CO)_3(4Me\text{-}py)(SbPh_3)]$	orange–brown	68	36.4(36.8)	2.5(2.5)	1.5(1.6)
15 $[WI_2(CO)_3(4Me\text{-}py)(PPh_3)]$	brown	78	36.6(36.9)	2.8(2.5)	1.4(1.6)
16 $[WI_2(CO)_3(4Me\text{-}py)(AsPh_3)]$	brown	74	37.6(35.2)	2.8(2.4)	2.3(1.5)
17 $[WI_2(CO)_3(4Me\text{-}py)(SbPh_3)]$	dark brown	66	33.0(33.5)	2.3(2.3)	1.3(1.5)
18 $[MoI_2(CO)_3(3,5\text{-}Me_2\text{-}py)(PPh_3)]$	orange–brown	97	41.9(41.9)	3.3(3.0)	1.7(1.7)
19 $[MoI_2(CO)_3(3,5\text{-}Me_2\text{-}py)(AsPh_3)]$	brown	66	39.2(39.7)	2.7(2.8)	1.8(1.7)
20 $[MoI_2(CO)_3(2Cl\text{-}py)(PPh_3)]$	dark brown	80	38.9(38.5)	2.6(2.4)	1.8(1.7)
21 $[MoI_2(CO)_3(2Cl\text{-}py)(AsPh_3)]$	yellow–brown	71	36.6(36.6)	2.4(2.2)	1.8(1.6)
22 $[MoI_2(CO)_3(2Cl\text{-}py)(SbPh_3)]$	orange	71	34.9(34.7)	2.3(2.1)	1.4(1.6)
23 $[WI_2(CO)_3(2Cl\text{-}py)(PPh_3)]$	brown	80	33.9(34.7)	2.2(2.1)	1.9(1.6)
24 $[WI_2(CO)_3(2Cl\text{-}py)(AsPh_3)]$	brown	72	33.3(33.1)	2.2(2.0)	1.3(1.5)
25 $[WI_2(CO)_3(2Cl\text{-}py)(SbPh_3)]$	dark brown	69	32.3(31.6)	2.1(1.9)	1.8(1.4)
26 $[MoI_2(CO)_3(3Cl\text{-}py)(PPh_3)]$	brown	94	39.4(38.5)	2.8(2.4)	1.6(1.7)
27 $[MoI_2(CO)_3(3Cl\text{-}py)(AsPh_3)]$	brown	56	35.9(36.6)	2.4(2.2)	1.8(1.6)
28 $[MoI_2(CO)_3(3Br\text{-}py)(AsPh_3)]$	yellow–brown	88	34.8(34.7)	2.4(2.1)	1.7(1.6)

[a]Calculated values in parentheses.

TABLE II. Infrared Data[a] for the Complexes $[MI_2(CO)_3LL']$

Complex	ν(CO) (cm^{-1})
1	2020(s), 1955(s), 1915(s)
2	2030(s), 1963(s), 1915(s)
3	2030(s), 1965(s), 1905(s)
4	2025(s), 1949(s), 1900(s)
5	2030(s), 1948(s), 1895(s)
6	2020(s), 1955(s), 1900(s)
7	2010(s), 1940(s), 1900(m)
8	2005(s), 1965(s), 1920(s)
9	2015(s), 1940(s), 1895(m)
10	2020(s), 1940(s), 1900(m)
11	2020(s), 1935(s), 1900(m)
12	2020(s), 1945(s), 1905(m)
13	2010(s), 1955(s), 1900(m)
14	2020(s), 1950(s), 1910(m)
15	2015(s), 1935(s), 1900(m)
16	2010(s), 1945(s), 1890(m)
17	2015(s), 1945(s), 1905(m)
18	2050(s), 1955(s), 1910(s)
19	2040(s), 1960(s), 1915(m)
20	2015(s), 1945(s), 1895(m)
21	2010(s), 1960(s), 1905(m)
22	2015(s), 1930(s), 1905(m)
23	2015(s), 1950(s), 1910(s)
24	2005(s), 1950(s), 1895(m)
25	2015(s), 1930(s), 1910(s)
26	2040(s), 1955(s), 1925(m)
27	2045(s), 1960(s), 1920(m)
28	2045(s), 1965(s), 1930(s)

[a]Spectra recorded as thin films between NaCl plated in $CHCl_3$; m, medium; s, strong.

exposed to air. The compounds **1**–**28** are all soluble in $CHCl_3$ and CH_2Cl_2, but generally only slightly soluble in diethylether and hydrocarbon solvents. The complexes containing the methyl substituted pyridines are the most soluble compounds, whereas the chloro and bromo substituted pyridine complexes are the least soluble seven-coordinate compounds.

The mechanisms by which the successive substitution of acetonitrile ligands from $[MI_2(CO)_3(NCMe)_2]$ by L′ and L are likely to be dissociative since the complexes $[MI_2(CO)_3(NCMe)_2]$ and $[MI_2(CO)_3(NCMe)L']$ obey the effective atomic number rule. Hence the loss of acetonitrile is required in order to create a vacant coordination site on the metal before coordination of L′ or L can occur. Further evidence to support these dissociative mechanisms comes from some work by Tripathi and coworkers [23]. They reacted $[Mo(CO)_5L]$ ($L = PPh_3$, $AsPh_3$ or $SbPh_3$) with X_2 (X = Cl, Br or I) in hexane to give the six-coordinate 16 electron compounds $[MoX_2(CO)_3L]$. The $[MoX_2(CO)_3L]$ complexes are analogous to the proposed intermediates $[MI_2(CO)_3(NCMe)]$ and $[MI_2(CO)_3L']$ formed from successive loss of acetonitrile from $[MI_2(CO)_3(NCMe)_2]$ and $[MI_2(CO)_3(NCMe)L']$.

The infrared spectra of the seven-coordinate complexes $[MI_2(CO)_3LL']$ (**1**–**28**) all show three carbonyl bands (Table II) with similar patterns and intensities to other previously reported [3–18] seven-coordinate complexes of molybdenum(II) and tungsten(II). The geometry of seven-coordinate complexes of molybdenum(II) and tungsten(II) of the type $[MX_2(CO)_3L_2]$ have generally been found to have capped octahedral geometry [24–31]. Hence, it is likely in view of the similar infrared spectral properties of **1**–**28** to the previously reported bis-ligand complexes $[MX_2(CO)_3L_2]$ that **1**–**28** have capped octahedral geometry. Unfortunately attempts to grow single crystals for X-ray crystallography of several of the mixed complexes $[MI_2(CO)_3LL']$ described in this paper were unsuccessful.

Acknowledgement

We thank the S.E.R.C. for support.

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 M. H. B. Stiddard, *J. Chem. Soc. A*, 4712 (1962).
4 F. A. Cotton and B. F. G. Johnson, *Inorg. Chem., 3*, 1609 (1964).
5 R. Colton and I. B. Tomkins, *Aust. J. Chem., 19*, 1143 (1966).
6 J. Lewis and R. Whyman, *J. Chem. Soc. A*, 77 (1967).
7 B. F. G. Johnson, *J. Chem. Soc. A*, 475 (1967).
8 R. Colton, G. R. Scollary and I. B. Tomkins, *Aust. J. Chem., 21*, 15 (1968).
9 R. Colton and G. R. Scollary, *Aust. J. Chem., 21*, 1435 (1968).
10 W. S. Tsang, D. W. Meek and A. Wojcicki, *Inorg. Chem.., 7*, 1263 (1968).
11 R. Colton and C. J. Rix, *Aust. J. Chem., 22*, 305 (1969).
12 M. R. Snow, P. Pauling and M. H. B. Stiddard, *Aust. J. Chem., 22*, 709 (1969).
13 J. A. Bowden and R. Colton, *Aust. J. Chem., 22*, 905 (1969).
14 J. R. Moss and B. L. Shaw, *J. Chem. Soc. A*, 595 (1970).
15 R. Colton, *Coord. Chem. Rev., 6*, 269 (1971).
16 L. Benzce, *J. Organomet. Chem., 37*, C37 (1972).
17 A. D. Westland and N. Muriithi, *Inorg. Chem., 12*, 2356 (1973).
18 S. C. Tripathi, S. C. Srivastava and A. K. Shrimal, *Inorg. Chim. Acta, 18*, 231 (1976).
19 P. Umland and H. Vahrenkamp, *Chem. Ber., 115*, 3565 (1982).
20 P. K. Baker, S. G. Fraser and E. M. Keys, *J. Organomet. Chem., 309*, 319 (1986).
21 P. K. Baker and S. G. Fraser, *J. Coord. Chem., 15*, 405 (1987).
22 D. P. Tate, W. R. Knipple and J. M. Augl, *Inorg. Chem., 1*, 433 (1962).
23 S. C. Tripathi, S. C. Srivastava and D. P. Pandey, *Transition Met. Chem., 2*, 52 (1977).
24 A. Mawby and G. E. Pringle, *J. Inorg. Nucl. Chem., 34*, 517 (1972).
25 M. G. B. Drew, *J. Chem. Soc., Dalton Trans.*, 626 (1972).
26 M. G. B. Drew, *J. Chem. Soc., Dalton Trans.*, 1329 (1972).
27 G. Schmid, R. Boese and E. Welz, *Chem. Ber., 108*, 260 (1975).
28 M. G. B. Drew and J. D. Wilkins, *J. Chem. Soc., Dalton Trans.*, 1984 (1975).
29 R. Boese and U. Müller, *Acta Crystallogr., Sect. B, 32*, 582 (1976).
30 M. G. B. Drew and A. P. Wolters, *Acta Crystallogr., Sect. B, 33*, 205 (1977).
31 M. G. B. Drew and J. D. Wilkins, *J. Chem. Soc., Dalton Trans.*, 557 (1977).