Inorganica Chimica Acta, *74* (1983) 235–239

Deprotonation and *C*-Alkylation of $Ph_2PCH_2PPh_2$-Derivatives of Chromium(0), Molybdenum(0), Tungsten(0), Platinum(II) and Palladium(II)

SUBHI AL-JIBORI and BERNARD L. SHAW*

School of Chemistry, The University, Leeds LS2 9JT, U.K.

Received February 23, 1983

$Ph_2PCH_2PPh_2$ derivatives of the Group VI metal carbonyls, $[M(CO)_4(Ph_2PCH_2PPh_2)]$, are deprotonated by $LiBu^n$ or LiMe, acting as strong bases. The resultant carbanions or lithio-derivatives are readily alkylated (trimethylsilylated) when treated with MeI, EtI, $PhCH_2Br$, $CH_2{=}CHCH_2Br$ or Me_3SiCl to give $[M(CO)_4(Ph_2PCHRPPh_2)$ (R = Me, Et, CH_2Ph, $CH_2CH{=}CH_2$ or $SiMe_3$). Treatment with excess of MeLi and MeI gives the dimethylderivatives $[M(CO)_4(Ph_2PCMe_2PPh_2)]$. Treatment of $[PtX_2(Ph_2(PCH_2PPh_2)]$ with the non-nucleophilic (very bulky) base $LiN[SiMe_3]_2$, followed by MeI, EtI, $BrCH_2Ph$, Me_3SiCl, gives the corresponding C-substituted products $[PtX_2(Ph_2PCHRPPh_2)]$ (X = Cl, Br or I) and two successive treatments with $LiN[SiMe_3]_2$/MeI gives $[PtI_2(Ph_2PCMe_2PPh_2)]$. $[PdI_2(Ph_2PCH_2PPh_2)]$ reacts with $LiN[SiMe_3]_2$/MeI to give a mixture of $[PdI_2(Ph_2PCMe_2PPh_2)]$, $[PdI_2(Ph_2PCHMePPh_2)]$ and $[PdI_2(Ph_2PCH_2PPh_2)]$. NMR data are given.

Introduction

There is much interest in $Ph_2PCH_2PPh_2$ (dppm) as a ligand in coordination/organometallic chemistry. However, with the exception of the case where R = CH_3, diphosphines of type $Ph_2PCHRPPh_2$, with R = alkyl or aryl are not known. Ligands of this type will in general be more difficult to synthesize than dppm. Thus although a strong base such as n-butyl-lithium deprotonates dppm to give $[Ph_2PCHPPh_2]^-$ this anion is an ambident nucleophile and treatment with an alkylating agent would generally give mixtures corresponding to attack on carbon and phosphorus *e.g.* benzyl chloride attacks it predominantly at phosphorus [1]. However, we thought it possible that, when coordinated to a metal through both phosphorus atoms, deprotonation would give a carbanion which would not be ambident and that it would alkylate selectively at carbon. We chose to study the deprotonation and subsequent alkylation of dppm in complexes of type $[M(CO)_4(Ph_2PCH_2PPh_2)]$. We subsequently went on to try similar alkylations with complexes of type $[MX_2(Ph_2PCH_2PPh_2)]$ (M = Pt or Pd; X = Cl, Br or I). Preliminary accounts of some of this work have been published [2, 3].

Experimental

The experimental techniques were the same as those used in other recent papers from this laboratory [4]. $[Cr(CO)_4(dppm)]$ and $[W(CO)_4(dppm)]$ were prepared by the published methods, in 84 and 81% yields (lit. 48 and 51% respectively) [5]. $[Mo(CO)_4(dppm)]$ was also made by the literature method in 87% yield (lit. 75%) [5] and also using phase-transfer catalysis [6].

$[Mo(CO)_4(dppm)]$

A mixture of $[Mo(CO)_6]$ (2.11 g, 8.0 mmol), dppm (3.07 g, 8.0 mmol), benzene (120 cm^3), 50% aqueous sodium hydroxide solution (20 cm^3) and methyl-trialkyl-(C_8–C_{10})-ammonium chloride (ADOGEN) (0.8 g) was stirred vigorously at *ca.* 80 °C for 5 h under argon. Water (50 cm^3) was then added and the product was isolated from the organic layer. Yield 3.62 g, 6.1 mmol, 76%. Its purity was established by ^{31}P-$\{^1H\}$ NMR and by IR spectroscopy.

$[Cr(CO)_4\{Ph_2PCHMePPh_2\}]$

A solution of n-butyl-lithium in diethyl ether (1.08 mmol, 0.677 cm^3) was added to a stirred solution of $[Cr(CO)_4(dppm)]$ (0.396 g, 0.72 mmol) and N,N,N′,N′-tetramethylethylenediamine (TMED) (0.16 cm^3, 1.08 mmol) in benzene (10 cm^3). The mixture was stirred for 1 h at 20 °C, then treated with methyl iodide (0.09 cm^3, 1.44 mmol) after which it was stirred vigorously at 55–60 °C for 4 h. The resultant mixture was then cooled, water added and the product was isolated. It formed yellow-orange needles (0.30 g, 0.53 mmol, 74%) from CH_2Cl_2/MeOH. The analogous Mo and W complexes were similarly made in 73 and 80.5% yields respectively.

The following seven complexes of type $[M(CO)_4\{Ph_2PCHRPPh_2\}]$ were similarly made.

---

*Author to whom correspondence should be addressed.

0020-1693/83/$3.00

R = Et (using ethyl iodide) M = Cr (89%), Mo (91%) and W (86%)

R = $CH_2CH{=}CH_2$ (using allyl bromide) M = Cr (89%), M = W (59%)

R = CH_2Ph (using benzyl bromide) M = Cr (88%)

R = $Si(CH_3)_3$ {using $ClSi(CH_3)_3$} M = Cr (90%)

[Cr(CO)$_4${Ph$_2$PCMe$_2$PPh$_2$}]

A solution of methyl-lithium (1.2 cm^3, 3.17 mmol) in diethyl ether was added to a solution of $[Cr(CO)_4(dppm)]$ (0.434 g, 0.79 mmol) and TMED (0.48 cm^3, 3.17 mmol) in benzene (10 cm^3). The mixture was stirred at 20 °C for 1 h, after which methyl iodide (0.195 cm^3, 3.16 mmol) was added and the mixture then stirred at 70–75 °C for a further 5 h after which it was cooled to 0 °C, water (10 cm^3) was added and the product isolated. It formed pale yellow plates from CH_2Cl_2/MeOH. Yield 0.406 (89%).

The corresponding Mo and W complexes were similarly made in 81 and 73% yields, respectively.

[PtI$_2$(Ph$_2$PCHMePPh$_2$)]

A solution of $LiN[SiMe_3]_2$ in tetrahydrofuran (1.2 cm^3, 1 *M*, 1.2 mmol) was added to a suspension of $[PtI_2(dppm)]$ (1.0 g, 1.2 mmol) in tetrahydrofuran (15 cm^3) and the resultant mixture was stirred at *ca.* 20 °C for 1.5 h. Methyl iodide (0.164 cm^3, 2.26 mmol) was then added and the mixture was then stirred at 45–55 °C for 5 h. The solvent was then evaporated, the residue washed with water and dried and the required product extracted with and recrystallized from dichloromethane. It formed yellow microcrystals (0.73 g, 0.86 mmol, 72%). The mass spectrum showed a parent molecular ion 844–850, m/e of the expected intensity ratios.

[PtI$_2${Ph$_2$PCHEtPPh$_2$}]

This was similarly prepared from $LiN[SiMe_3]_2$ (1.5 mol equivalents) $[PtI_2(dppm)]$ and ethyl iodide. Yellow microcrystalline solid. Yield 86% m/e 860–865, with the expected intensity ratios for the parent molecular ion.

[PtBr$_2${Ph$_2$PCH(CH$_2$Ph)PPh$_2$}]

This was similarly prepared using $[PtBr_2(dppm)]$, $LiN[SiMe_3]_2$ (1.5 mol equivalents), tetrahydrofuran and benzyl bromide. It was obtained as an off-white crystalline solid from CH_2Cl_2/MeOH in 85% yield.

[PtI$_2${Ph$_2$PCMe$_2$PPh$_2$}]

A solution of $LiN[SiMe_3]_2$ in tetrahydrofuran (0.40 cm^3, 0.4 mmol) was added to a suspension of $[PtI_2(dppm)]$ (0.336 g, 0.4 mmol) in tetrahydrofuran (5 cm^3). The mixture was stirred for 1 h, then methyl iodide (0.025 cm^3, 0.4 mmol) was added and heating and stirring was continued for a further 2 h at 40 °C. This gave a yellow suspension. A further quantity of $LiN[SiMe_3]_2$ (0.8 cm^3, 0.8 mmol) was then added: the yellow solid dissolved. Methyl iodide (0.1 cm^3, 1.6 mmol) was added and the mixture stirred at 40 °C for 4 h giving a cream solid. This was filtered off, washed with water, dried and recrystallized from dichloromethane giving the required compound as yellow microcrystals (0.25 g, 73%), m/e 858–866, with the expected intensity ratios for the parent molecular ion.

Action of LiN[SiMe$_3$]$_2$ and Methyl Iodide on [PdI$_2$(dppm)]

A tetrahydrofuran solution of the lithium reagent (0.31 mmol, 0.31 cm^3) was added to a suspension of $[PdI_2(dppm)]$ (0.213 g, 0.286 mmol) and the mixture was stirred at 20 °C for 1 h. Methyl iodide (0.08 cm^3, 1.3 mmol) was added and the resultant mixture stirred at 40 °C for 11 h. The product was isolated by evaporation, washed with water and dried. A $^{31}P\text{-}\{^1H\}$ and ^1H- and $^1H\text{-}\{^{31}P\}$ NMR study showed it to be a mixture of three components, probably $[PdI_2\{Ph_2PCHMePPh_2\}]$, $[PdI_2\{Ph_2PCMe_2PPh_2\}]$ and $[PdI_2(dppm)]$ in the approximate ratio 10:5:1 (see Discussion).

Results and Discussion

Treatment of a benzene solution of $[Cr(CO)_4(Ph_2PCH_2PPh_2)]$ with n-butyl-lithium and TMED gave a yellow precipitate, which was presumably that of the lithio-derivative $[Cr(CO)_4(Ph_2PCHLiPPh_2)]$ or $[Cr(CO)_4(Ph_2PCHPPh_2)]Li$. Subsequent treatment with methyl iodide then gave the hoped for methyl-derivative, $[Cr(CO)_4(Ph_2PCHMePPh_2)]$ in 74% yield. Characterizing microanalytical, and IR and NMR spectroscopic data are given in Tables I and II. The corresponding molybdenum and tungsten compounds were similarly methylated. Similar alkylations were done with ethyl iodide, allyl bromide or benzyl bromide with a representative selection of the chromium, molybdenum or tungsten complexes and in general compounds of the type $[M(CO)_4(Ph_2PCHRPPh_2)]$ with R = CH_3, C_2H_5, $CH_2CH{=}CH_2$ or CH_2Ph; M = Cr, Mo or W could be synthesized readily, and in high yield by alkylation of the deprotonated dppm complexes. See Tables I and II for characterizing data. The trimethylsilyl derivative $[Cr(CO)_4\{Ph_2PCH(SiMe_3)PPh_2\}]$ was similarly made, using trimethylsilyl chloride. Free $Ph_2PCHSiMe_3PPh_2$ is known [7].

We also treated $[M(CO)_4(Ph_2(PCH_2PPh_2)]$ (M = Cr, Mo or W) with four mole equivalents of methyl-lithium/TMED followed by an excess of methyl iodide. This gave the *gem*-dimethyl complexes $[M(CO)_4(Ph_2PCMe_2PPh_2)]$, in very good {70%–80%} yields (see Tables I and II for characterization data).

TABLE I. Melting Points, Colours,[a] Molecular Weights[b,c] and Elemental Analytical Data[c] for Complexes of the Type $[M(CO)_4(Ph_2PCHRPPh_2)]$ or $[M(CO)_4(Ph_2PCMe_2PPh_2)]$.

M	R	M.p. °C	Colour	C	H	M. Wt.
Cr	CH_3	241–244	po	64.2(64.05)	4.5(4.3)	564(562)
Cr	C_2H_5	235–241[d]	yo	64.25(64.5)	4.65(4.55)	577(576)
Cr	allyl	164–166[d]	y	65.15(65.3)	4.55(4.45)	613(589)
Cr	CH_2Ph	178–179	y	67.65(67.75)	4.35(4.4)	618(638)
Cr	$SiMe_3$	171–176	y	61.75(61.9)	4.95(4.9)	634(620)
Mo	CH_3	225–228[d]	py	59.2(59.4)	4.1(4.0)	603(606)
Mo	C_2H_5	206–208	py	59.95(60.0)	4.3(4.2)	627(620)
Mo	allyl	145–160[d]	py	60.6(60.75)	4.3(4.15)	623(632)
W	CH_3	252–255[d]	py	51.9(51.9)	3.5(3.5)	692(694)
W	C_2H_5	254–257[d]	py	52.5(52.55)	3.75(3.7)	702(708)
$[M(CO)_4(Ph_2PCMe_2PPh_2)]$						
M = Cr		205–208[d]	py	64.55(64.6)	4.95(4.55)	563(576)
M = Mo		194–197[d]	py	60.15(60.0)	4.4(4.2)	630(620)
M = W		224–226[d]	py	52.2(52.55)	3.65(3.7)	725(708)

[a] po = pale orange, yo = yellow/orange, y = yellow, py = pale yellow. [b] Measured osmometrically in $CHCl_3$ solution. [c] Calculated values in parentheses. [d] With decomposition.

TABLE II. Infrared {ν(CO)}[a] and ^{31}P-{^1H}-[b] and ^1H-[c] NMR Data for Complexes of Type $[M(CO)_4(Ph_2PCHRPPh_2)]$ or $[M(CO)_4(Ph_2PCMe_2PPh_2)]$.

M	R	ν(CO) cm^{-1}	^1H NMR	δ P[b,e]
Cr	H	2006, 1915 1897, 1875[d]	$\delta = 4.46$, $^2J(PH) = 9.4$ (CH_2)	26.8
Cr	CH_3	2012, 1918 1890, 1877	$\delta = 4.74$, $^2J(PH) = 10.9$, (CH); $\delta = 1.18$, $^3J(PH) = 13.4$, $^3J(HH) = 7.6$ (CH_3)	45.1
Cr	C_2H_5	2010, 1916 1890, 1870	$\delta = 4.55$, $^2J(PH) = 11.4$, (CH); $\delta = 1.6$ (CH_2); $\delta = 0.8$, $^3J(HH) = 7.1$ (CH_3)	48.4
Cr	CH_2Ph	2010, 1917 1888, 1877	$\delta = 4.96$, $^2J(PH) = 11.1$, (CH); $\delta = 2.9$, $^3J(PH) = 12.7$, $^3J(HH) = 6.85$ (CH_2)	50
Cr	allyl	2008, 1915 1889, 1873	$\delta = 4.69$ multiplet {CH(allyl)} the allyl resonances were a complex multiplet	49.3
Cr	$SiMe_3$	2000, 1921 1902, 1881	$\delta = 4.9$, $^2J(PH) = 11$ (CH); $\delta = -0.4$ (CH_3)	39.6
Mo	H	2020, 1920 1907, 1879[d]	$\delta = 4.45$, $^2J(PH) = 6.5$ CH_2	2.6
Mo	CH_3	2022, 1920 1905, 1880	$\delta = 4.71$, $^2J(PH) = 10.4$ (CH); $\delta = 1.14$	22.6
Mo	C_2H_5	2021, 1922 1905, 1883	$\delta = 4.53$, $^2J(PH) = 10.8$ (CH); $\delta = 1.55$ (CH_2), $\delta = 0.77$, $^3J(HH) = 7.3$ (CH_3)	24.0
Mo	CH_2Ph	2020, 1919 1905, 1882	$\delta = 4.94$, $^2J(PH) = 9.95$ (CH); $\delta = 2.84$, $^3J(PH) = 12.2$, $^3J(HH) = 6.95$	26.3
Mo	allyl	2020, 1918 1904, 1880	$\delta = 4.66$ multiplet {CH(allyl)} the allyl resonances were a complex multiplet	25.6
W	H	2013, 1909 1902, 1871	$\delta = 4.9$, $^2J(PH) = 9$ (CH_2)	−22.8 (203)
W	CH_3	2021, 1910[d] 1895, 1873	$\delta = 5.03$, $^2J(PH) = 10.5$ (CH); $\delta = 1.11$, $^3J(PH) = 13.6$, $^3J(HH) = 7.65$ (CH_3)	−1.7 (205)

(Continued overleaf)

TABLE II. *(Continued)*

W	C_2H_5	2019, 1911 1894, 1872	δ = 4.77, $^2J(PH)$ = 11 (C*H*); δ = 1.51 (CH_2); δ = 0.78, $^3J(HH)$ = 7.1 (CH_3)	0.4 (205)
$[M(CO)_4(Ph_2PCMe_2PPh_2)]$				
M = Cr		2010, 1913 1890, 1872	δ = 1.5, $^3J(PH)$ = 13.5 (CH_3)	65.5
M = Mo		2022, 1915 1900, 1878	δ = 1.5, $^3J(PH)$ = 13.1 (CH_3)	44.5
M = W		2018, 1912 1895, 1873	δ = 1.5, $^3J(PH)$ = 13.6 (CH_3)	21.3 (208)

[a] In 1,2-dichloroethane solution. [b] In ppm to high field of 85% H_3PO_4, measured in $CDCl_3$. [c] In $CDCl_3$. δ-values ±0.02 ppm, J-values ±0.1 Hz. [d] From J. Chatt and H. R. Watson, *J. Chem. Soc.*, 4980 (1961). [e] J(WP) in parentheses.

TABLE III. Elemental Analytical Data,[a] ^{31}P-[b] and ^1H-[c] NMR Data for Complexes of Type $[PtX_2(Ph_2PCHRPPh_2)]$ or $[PtI_2(Ph_2PCMe_2PPh_2)]$ X = Br or I, R = Me, Et or CH_2Ph.

X	R	C	H	X	$\delta(P)\{^1J(PtP)\}$	^1H NMR data	
I	Me	36.8(36.85)	2.85(2.85)	29.8(29.95)	−50.9(2886)	δ = 5.0 (C*H*) $^3J(HH)$ = 8	δ = 1.0 (CH_3) $^3J(PH)$ = 18
I	Et	37.6(37.65)	3.1(3.05)	29.8(29.45)	−50.6(2881)	δ = 4.7 (C*H*) $^3J(PtH)$ = 48	δ = 1.4 (CH_2) δ = 0.72 (CH_3)
Br	CH_2Ph	46.45(46.35)	3.45(3.4)	19.45(19.3)	−44.9(3030)	δ = 5.0 (C*H*) $^3J(PtH)$ = 33 $^2J(PH)$ = 12	δ = 2.7 (CH_2) J(C*H*CH_2Ph) = 8 J(PH) = 16
$[PtI_2(Ph_2PCMe_2PPh_2)]$		37.7(37.65)	2.95(3.05)	29.2(29.45)	−27.5(2925)	δ = 1.1 (CH_3) $^3J(PH)$ = 17	

[a] Calculated values in parentheses. [b] In $CDCl_3$, δ-values ±0.1 relative to 85% H_3PO_4. J-values ±3 Hz. [c] In $CDCl_3$, δ-values ±0.1, J-values ±1 Hz. The values are based on ^1H- and ^1H-$\{^{31}P\}$-NMR spectra. Not all the coupling constants could be measured.

Thus deprotonation followed by alkylation is a convenient method for the synthesis of *C*-substituted dppm–$M(CO)_4$ complexes (M = Cr, Mo or W). It should be applicable to the synthesis of a wide range of such compounds.

Encouraged by the success with these octahedral Group VI metal carbonyl derivatives we then attempted deprotonate/alkylate square planar complexes of platinum(II) or palladium(II) of type $[MX_2(Ph_2PCH_2PPh_2)]$ (X = Cl, Br or I). Clearly, one could not use LiMe or $LiBu^n$ to affect deprotonation since these reagents would be expected to alkylate the platinum or palladium. However, we hoped that a strong but very sterically demanding base such as the commercially available $LiN[SiMe_3]_2$ would affect deprotonation without causing substitution. However the deprotonated moiety, $[Ph_2PCHPPh_2]$ has been shown to be an excellent ligand for platinum(II) or palladium(II) [1], *e.g.* in binuclear complexes of type $[M_2(\mu\text{-}X)_2(Ph_2PCHPPh_2)_2]$ [8]. We thus thought that even if deprotonation could be effected the resultant species might be too stable to be alkylated. However, using tetrahydrofuran as solvent we found that treatment of $[PtI_2(Ph_2PCH_2PPh_2)]$ with $LiN[SiMe_3]_2$, followed by methyl iodide, readily gave the desired $[PtI_2(Ph_2PCHMePPh_2)]$ in very satisfactory (72%) yield (see Experimental and Table III for characterization data). We similarly made $[PtI_2(Ph_2PCHEtPPh_2)]$ and $[PtBr_2\{Ph_2PCH(CH_2Ph)PPh_2\}]$: two, successive treatments of $[PtI_2\{Ph_2PCH_2PPh_2\}]$ with $LiN[SiMe_3]_2$/MeI gave $[PtI_2\{Ph_2PCMe_2PPh_2\}]$ in 75% isolated yield.

Although $Ph_2PCHMePPh_2$ and $Ph_2PCMe_2PPh_2$ are known [9–11] we find they are much less easy to make than $Ph_2PCH_2PPh_2$ (which is commercially available). Thus deprotonation/methylation of $[PtI_2(Ph_2PCH_2PPh_2)]$ is at least comparable in convenience as a method of synthesizing the methyl- or dimethyl-derivatives $[PtI_2(Ph_2PCR_1R_2PPh_2)]$. For the synthesis of other derivatives *e.g.* R_1 = Et, CH_2Ph: R_2 = H *etc.* the deprotonation/alkylation procedure we describe in this paper may prove to be the best method, quite apart from its intrinsic interest. Treatment of $[PdI_2(Ph_2PCH_2PPh_2)]$ with $LiN[SiMe_3]_2/MeI$ gave mainly the required $[PdI_2(Ph_2PCHMePPh_2)]$ but this was not obtained pure and was contaminated with a small amount of $[PdI_2(Ph_2PCH_2PPh_2)]$ and a larger amount of $[PdI_2(Ph_2PCMe_2PPh_2)]$. This followed from a ^{31}P-, ^1H-, and ^1H-$\{^{31}P\}$ NMR study of this three-component mixture, which we did not succeed in separating.

We also treated $[PtCl_2(Ph_2PCH_2PPh_2)]$ with $LiN[SiMe_3]_2$ (one mole equivalent) followed by Me_3SiCl. The crude product was almost entirely $[PtCl_2\{Ph_2PCHSiMe_3PPh_2\}]$ as evidenced by the ^{31}P- and ^1H-NMR data—$\delta(P) = -55.9$ ppm, $^1J(PtP) = 3035$ Hz, $\delta(CH) = 4.4$ ppm, $^2J(PH) = 13$ Hz, $^3J(PtH) = 53$ Hz; $\delta(SiMe_3) = -0.4$ ppm but the compound was rather labile and hydrolysis occurred to some extent on attempted purification, giving a mixture with $[PtCl_2(Ph_2PCH_2PPh_3)]$.

We suggest that the intermediate deprotonated species are of type $[PtX_2(Ph_2PCHPPh_2)]^-$ or $[PtX_2(Ph_2PCHLiPPh_2)]$ and it is these which are alkylated or trimethylsilylated. As we previously described the intermediate deprotonated species (for X = I) could readily be detected by ^{31}P-$\{^1H\}$ NMR spectroscopy [3].

Acknowledgements

We thank the Iraqi Ministry of Higher Education and Scientific Research, and the Science and Engineering Research Council for support.

References

1 K. Issleib and H. P. Abicht, *J. Prakt. Chem., 312,* 456 (1970).
2 S. Al-Jibori and B. L. Shaw, *J. Chem. Soc. Chem. Commun.,* 286 (1982).
3 S. Al-Jibori and B. L. Shaw, *Inorg. Chim. Acta, 65,* L123 (1982).
4 J. R. Briggs, C. Crocker, W. S. McDonald and B. L. Shaw, *J. Chem. Soc. Dalton Trans.,* 575 (1981).
5 S. O. Grim, W. L. Briggs, R. C. Barth, C. A. Tolman and J. P. Jesson, *Inorg. Chem., 13,* 1095 (1974).
6 K. Y. Hui and B. L. Shaw, *J. Organometal. Chem., 124,* 262 (1977).
7 R. Appel, K. Giesle and H. F. Schöler, *Chem. Ber., 112,* 648 (1979).
8 J. Browning, G. W. Bushnell and K. R. Dixon, *J. Organometal. Chem., 198,* C11 (1980).
9 H. Schmidbauer and A. Wohlleben-Hammer, *Chem. Ber., 112,* 510 (1979).
10 A. F. M. J. van der Ploeg and G. van Koten, *Inorg. Chim. Acta, 51,* 225 (1981).
11 W. Hewertson and H. R. Watson, *J. Chem. Soc.,* 1490 (1962).