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Trimethylpalladium(IV) chemistry. Conformational and fluxional effects in related palladium(IV) and platinum(IV) complexes of flexible bidentate nitrogen donor ligands

Peter K. Byers, Allan J. Canty *, R. Thomas Honeyman, and Andrew A. Watson

Chemistry Department, University of Tasmania, Hobart, Tasmania 7001 (Australia)

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Abstract

Iodomethane reacts with palladium(II) complexes $PdMe_2(L_2)$, where L_2 is a flexible nitrogen donor ligand such as 1,1-bis(pyridin-2-yl)ethane [$(py)_2CHMe$], to form unstable trimethylpalladium(IV) complexes characterized as *fac*-$PdIMe_3(L_2)$ by comparison of their variable temperature ^1H NMR spectra with those of stable trimethylplatinum(IV) analogues. The complexes *fac*-$MIMe_3\{(py)_2CHMe\}$ occur as mixtures of isomers, with the chelate ring having the CH group adjacent to iodine in one isomer and adjacent to the methyl group *trans* to iodine in the other isomer. Complexes containing pyrazol-1-yl (pz) or *N*-methylimidazol-1-yl (mim) donors, *fac*-$MIMe_3(L_2)$ [$L_2 = (pz)_2CH_2$, $(pz)(mim)CH_2$, and $(py)(pz)CH_2$] undergo fluxional behaviour involving inversion of the six-membered chelate rings.

Introduction

Hydrocarbylpalladium(IV) complexes have only recently been synthesized and they all contain nitrogen donor bidentate or tridentate ligands [1–7], whereas the related platinum(IV) chemistry has been extensively developed following the preparation of $[PtIMe_3]_4$ in 1907 [8]. Isolated trialkylpalladium(IV) complexes containing bidentate ligands are restricted to the planar 2,2′-bipyridyl and 1,10-phenanthroline ligands, and are obtained by oxidative addition of alkyl halides to $Pd^{II}Me_2$ complexes [1,3–7], e.g., an acetone solution of $PdBrMe_2(CH_2Ph)(bipy)$ decomposes slowly at ambient temperature [4,6], but for tetramethylethylenediamine as ligand the complex $PdBrMe_2(CH_2Ph)(tmeda)$ could not be isolated although its formation could be detected by ^1H NMR spectroscopy at low temperature prior to facile reductive elimination of ethane to form $PdBr(CH_2Ph)(tmeda)$ [9].

We report here on a study of the oxidative addition of iodomethane to $Pd^{II}Me_2(L_2)$ complexes, where L_2 are closely related to 2,2′-bipyridyl but are more

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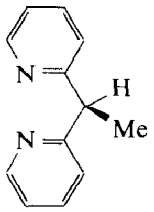
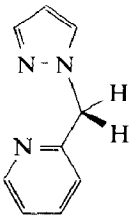
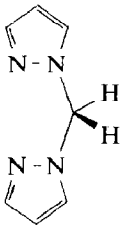
flexible, containing pyridin-2-yl (py), *N*-methylimidazol-2-yl (mim), or pyrazol-1-yl (pz) groups connected by a CH_2 or CHMe bridge. Palladium(IV) complexes generated in solution were studied by variable temperature ^1H NMR spectroscopy at $\leqslant -30\,^\circ C$, prior to reductive elimination which occurs readily at



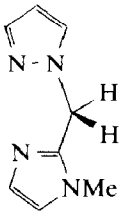
$(py)_2CHMe$



$(py)(pz)CH_2$



$(pz)_2CH_2$



$(pz)(mim)CH_2$

ambient temperature. Comparison with the spectra of stable platinum(IV) analogues has permitted both the assignment of structure as *fac*-$PdIMe_3(L_2)$ and the first comparison of fluxional and conformational behaviour of organopalladium(IV) and platinum(IV) complexes. A preliminary account of part of this work has appeared [10].

Results and discussion

The complexes $MMe_2(L_2)$ were obtained by established procedures [11], and the platinum(IV) complexes were isolated on oxidative addition of iodomethane to the $Pt^{II}Me_2$ complexes. The platinum(IV) complexes exhibit ^1H NMR spectra consistent with the formulation *fac*-$PtIMe_3(L_2)$, as established for the related bis(3,5-dimethylpyrazol-1-yl)methane complex *fac*-$PtIMe_3\{(Me_2pz)_2CH_2\}$ [12]. Palladium (IV) analogues could not be isolated, but solutions obtained by oxidative addition of MeI to $PdMe_2(L_2)$ at ca. $-30\,^\circ C$ in NMR tubes gave spectra very similar to the platinum(IV) complexes (Table 1); at higher temperatures the resonances of the Pd(IV) complexes decrease as resonances previously reported [11] for $PdIMe(L_2)$ and ethane appear.

$$Pd^{II}Me_2(L_2) + MeI \longrightarrow Pd^{IV}IMe_3(L_2) \longrightarrow Pd^{II}IMe(L_2) + Me\text{-}Me \qquad (1)$$

^1H NMR spectra for the $PdIMe_3(L_2)$ complexes were examined over the range -30 to $-70\,^\circ C$ and, for the stable platinum(IV) analogues, from $+20$ to $-70\,^\circ C$.

Complexes of $(py)_2CHMe$

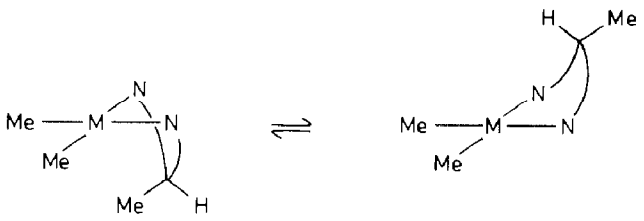
The complex $PtMe_2\{(py)_2CHMe\}$ exhibits variable temperature spectra similar to that reported [11] for the palladium(II) analogue, showing two PtMe, CMe, CH, and pyridine environments at ambient temperature with coalescence at higher temperature. Assuming that the chelate rings adopt a 'boat' conformation, as found by X-ray crystallographic studies for related complexes, e.g. $PdCl_2\{(pz)_2CMe_2\}$ and $[Pd\{(pz)_2CH\}]^{2+}$ [13], the NMR data are consistent with the conformational equilibrium shown, with the ratio **A**/**B** ca. 3/5 for Pt and ca. 1/1 for Pd.

Table 1

^{1}H NMR data for *fac*-$MIMe_3(L_2)$ in $(CD_3)_2CO$ [a]

Complex		δ(M–Me) [b]	δ(CH_2 or CHMe)	δ($H(6)_{py}$)
$PtIMe_3\{(py)_2CHMe\}$	Isomer I	1.68 (69.9, py)	7.00 (q, CH)	8.65 (d, J_{56} 6.8 Hz)
at 20 °C [c]		0.85 (69.6, I)	1.92 (d, *J*(H–Me) 7.2 Hz, Me)	
	Isomer II	1.59 (70.2, py)	4.94 (q, CH)	9.42 (d, J_{56} 6.8 Hz)
		1.27 (72.4, I)	1.99 (d, *J*(H–Me) 7.2 Hz, Me)	
$PdIMe_3\{(py)_2CHMe\}$	Isomer I	1.99 (py)	6.82 (q, CH)	8.66 (dd, J_{56} 5.4,
at −30 °C [c]		1.34 (I)	1.88 (d, *J*(H–Me) 7.1 Hz, Me)	J_{46} 1.3 Hz)
	Isomer II	1.94 (py)	4.82 (q, CH)	9.16 (dd, J_{56} 5.5,
		1.73 (I)	1.97 (d, *J*(H–Me) 7.5 Hz, Me)	J_{46} 1.2 Hz)
$PtIMe_3\{(py)(pz)CH_2\}$		1.59 (71.8, py)	6.95 (d, H_A)	8.86 (d(b))
at 20 °C		1.48 (70.7, pz)	5.77 (d, *J*(H–H)	
		0.94 (71.2, I)	15.0 Hz, H_B)	
at −70 °C	Conformer I′	1.51 (70.9, py)	7.01 (d, H_{ax})	8.70 (d, J_{56} ~ 5.7,
		1.39 (69.7, pz)	5.93 (d, *J*(H–H)	J_{HPt} ~ 22 Hz)
		0.86 (71.5, I)	14.7 Hz, H_{eq})	
	Conformer II′	1.43 (py) [d]	~ 5.93	9.18(d(b))
		1.32 (pz) [d]	(H, hidden by I′)	
		1.13 (I) [d]	5.59 (d, *J*(H–H) 15.6, H)	
$PdIMe_3\{(py)(pz)CH_2\}$		1.98 (py)	7.01 (b, H_A)	8.79 (b)
at −30 °C		1.84 (pz)	5.77 (d, *J*(H–H)	
		1.42 (I)	14.7 Hz, H_B)	
at −70 °C	Conformer I′	1.92 (py)	7.09 (d, H_{ax})	8.72 (d, J_{56} ~ 5.5 Hz)
		1.77 (pz)	5.85 (d, *J*(H–H)	
		1.37 (I)	14.8 Hz, H_{eq})	
	Conformer II′	1.88 (py)	~ 5.94	9.06(d(b))
		1.75 (pz)	(H, hidden by I′)	
		1.64 (I)	5.60 (d, *J*(H–H) 14.0, H)	
$PtIMe_3\{(pz)_2CH_2\}$		1.69 (73.1, pz)	8.05 (d)	
at 20 °C [e]		0.96 (71.6, I)	7.00 (d, *J*(H–H) 15.0 Hz)	
$PdIMe_3\{(pz)_2CH_2\}$		1.91 (pz)	7.86 (d)	
at −30 °C [e]		1.30 (I)	6.89 (d, *J*(H–H) 14.0 Hz)	
$PtIMe_3\{(pz)(mim)CH_2\}$		1.45 (73.2, pz)	6.51 (d)	
at 20 °C [c]		1.35 (70.0, mim)	5.92 (d, *J*(H–H)	
		0.84 (72.7, I)	15.8 Hz)	
$PdIMe_3\{(pz)(mim)CH_2\}$		1.84 (pz)	6.51 (d)	
at −30 °C [c]		1.70 (mim)	5.91 (d, *J*(H–H)	
		1.33 (I)	15.4 Hz)	

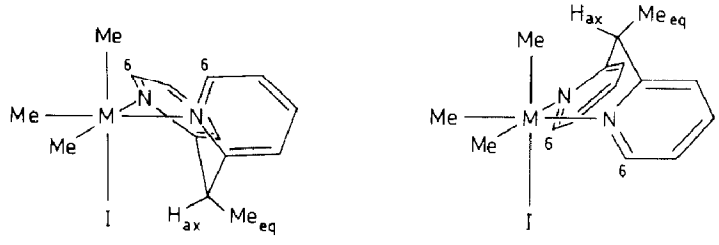
[a] For isolated platinum complexes, and for palladium complexes synthesized in NMR tubes. Variable temperature spectra are reversible. [b] ^2J(H–Pt) and *trans* group in parentheses. [c] Unchanged at lower temperature. [d] Very weak satellites. [e] Unaltered at lower temperature, except for broadening of PtMe (*trans* to iodine) and CH_2 resonances.



(A) (B)

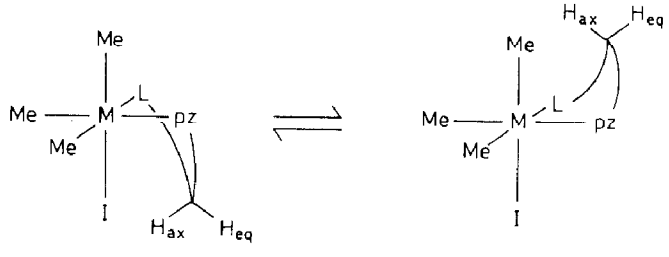
The downfield CMe and CH resonances are assigned to the axial positions in conformers A and B, respectively, since they appear at ca. 0.5 ppm downfield from the values for the other conformer and even further downfield from those for the free ligand, consistent with the marked downfield shifts observed for other complexes with carbon close to palladium(II) [11,14].

Spectra obtained by oxidative addition of MeI to $PdMe_2\{(py)_2CHMe\}$ indicate the presence of two $Pd^{IV}Me_3$ complexes in ca. 1/1 ratio (Fig. 1). The spectra are unchanged over the range −30 to −70 °C, and the isolated $Pt^{IV}Me_3$ complexes give similar spectra (Table 1). The spectra are most readily interpreted as resulting from isomers I and II, with fluxional motion for the chelate ring in each isomer considered unlikely as the alternative conformers would involve close approach of the 'bridge' methyl group with the 'axial' methyl or iodo group bonded to the metal atom. The bridge hydrogen gives well separated resonances for I and II, e.g. at 6.82 and 4.82 ppm for the palladium complex, reflecting the marked difference in



(I) (II)

fac-$MIMe_3\{(py)_2CHMe\}$



(I') (II')

fac-$MIMe_3\{(L)(pz)CH_2\}$ (L = py, pz, mim)

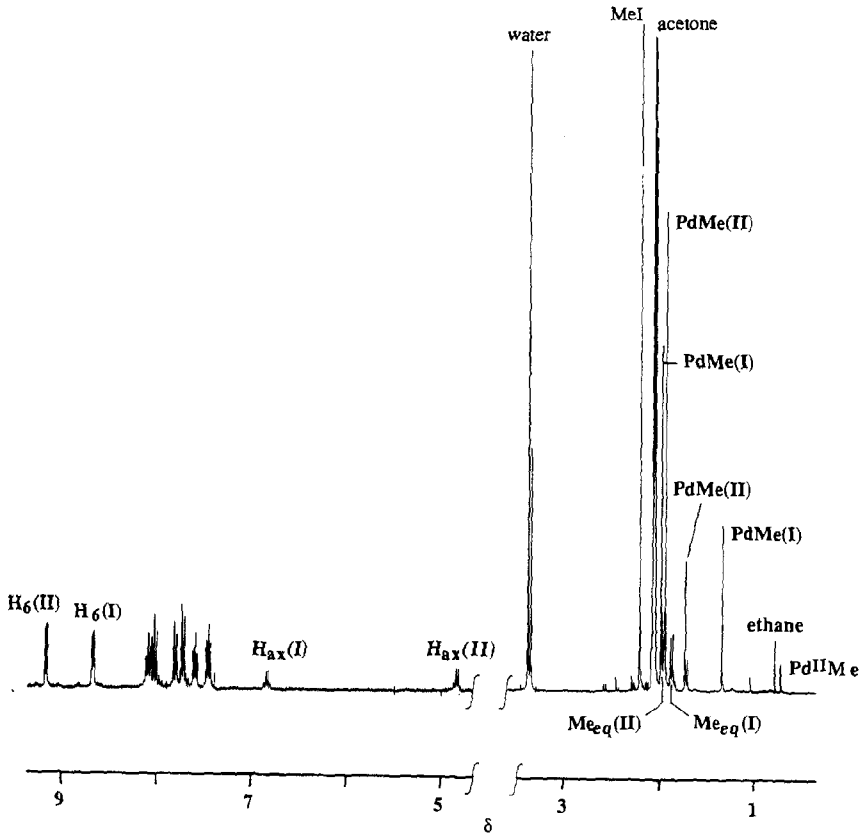


Fig. 1. ^1H NMR spectrum obtained after addition of excess MeI to $PdMe_2\{(py)_2CHMe\}$ at $-30\,^\circ C$, showing the two isomers of *fac*-$PdIMe_3\{(py)_2CHMe\}$, ethane, and $PdIMe\{(py)_2CHMe\}$ (denoted by $Pd^{II}Me$; two singlets indicate the presence of conformers, as reported elsewhere [11]).

environment in the isomers. The downfield resonance (6.82 ppm) is assigned to the environment adjacent to iodine (isomer I), since the upfield resonance (4.82 ppm) is similar to that for the free ligand (4.46 ppm) and protons adjacent to halogens are expected to be deshielded, e.g. the 2,2′-bipyridyl H(6) proton adjacent to X in PdXMe(bipy) occurs at 9.13 (Cl), 9.31 (Br) and 9.53 ppm (I) compared to 8.70–8.73 ppm for the proton adjacent to PdMe [11]. The H(6) resonance for isomer II has also been assigned on this basis, appearing 0.77 ppm (Pt) and 0.89 ppm (Pd) downfield from that of isomer I, consistent with closer approach to the iodine atom in II.

Complexes of (py)(pz)CH_2

The complex $PtMe_2\{(py)(pz)CH_2\}$ exhibits rapid exchange of methylene environments (singlet CH_2 at ambient temperature, two doublets at lower temperature), indicating the existence of rapid equilibria similar to that shown above ($A \rightleftharpoons B$) and as reported previously for the palladium analogue [11]. The complexes *fac*-$MIMe_3\{(py)(pz)CH_2\}$ give spectra at $-70\,^\circ C$ (Fig. 2 and 3) consistent with the presence of two conformers, I′ and II′, which are related to the conformations adopted by the isomers I and II for the $(py)_2CHMe$ complexes. Conformers I′ and

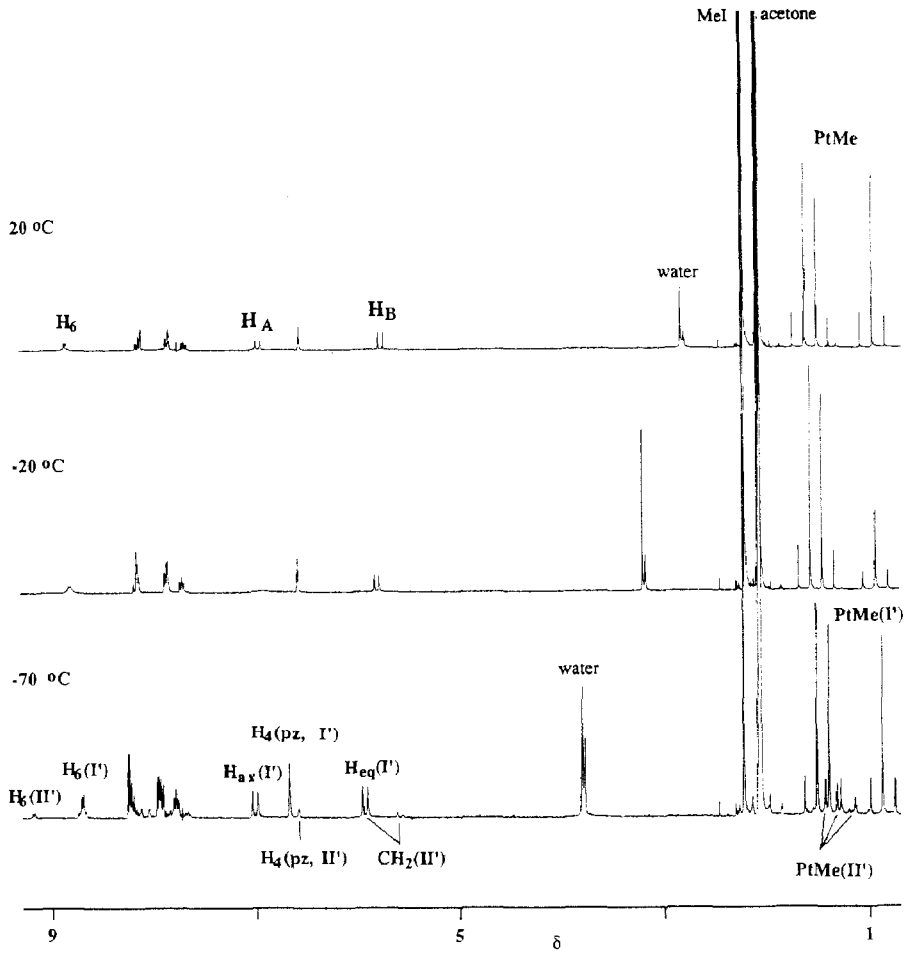


Fig. 2. Variable temperature ^{1}H NMR spectra for *fac*-$PtIMe_3\{(py)(pz)CH_2\}$, obtained after addition of an excess of MeI to $PtMe_2\{(py)(pz)CH_2\}$.

II′ are present in the ratio ca. 5/1 (Pt) and ca. 7/1 (Pd), and on warming rapid exchange results in broadening and coalescence, with the more stable platinum complex giving sharp resonances at ca. 20 °C. For both platinum and palladium, doublets at 7.01 (Pt) and 7.09 (Pd) in low temperature spectra correspond to the methylene axial proton of conformer I′ as they occur ca. 1–1.5 ppm downfield from the other methylene resonances (Figs. 2 and 3). Similar assignments to that for the $(py)_2CHMe$ complexes are possible for the pyridine H(6) protons, with the downfield resonance assigned to H(6) adjacent to iodine (conformer II′). For the minor isomer (II′) assignment of axial and equatorial methylene resonances has not been attempted since the chemical shift differences are small (ca. 0.1–0.3 ppm).

Complexes of $(pz)_2CH_2$ and $(pz)(mim)CH_2$

The complex $PtMe_2\{(pz)_2CH_2\}$ is insoluble in acetone and chloroform, but $PdMe_2\{(pz)_2CH_2\}$ shows NMR behaviour consistent with rapid exchange of methylene proton environments (singlet at ambient temperature, two doublets at lower temperature) [11], and the complexes $MMe_2\{(pz)(mim)CH_2\}$ exhibit a singlet for the CH_2 resonance even at −70 °C.

Spectra for *fac*-$PdIMe_3\{(pz)_2CH_2\}$ are of low quality, as this complex is less stable than the other complexes, and the low stability of $PdMe_2\{(pz)_2CH_2\}$ also gives rise to additional resonances resulting from partial decomposition. The $MIMe_3(L_2)$ complexes of both $(pz)_2CH_2$ and $(pz)(mim)CH_2$ give variable temperature spectra exhibiting two methylene proton environments at all temperatures, compared with four at low temperature for the $(py)(pz)CH_2$ complexes discussed above. However, for $PtIMe_3\{(pz)(mim)CH_2\}$ the low temperature spectra show broadening at ca. $-20\,^{\circ}C$ for the PtMe group *trans* to iodine, and for both methylene resonances. Similar effects are also seen for *fac*-$MIMe_3\{(py)(pz)CH_2\}$ (Figs. 2 and 3), where H_A, H(6), and PdMe resonances are broad at ca. $-30\,^{\circ}C$ prior to resolution into spectra of the conformers I′ and II′ at lower temperatures.

Kinetic studies indicate that decomposition of *fac*-$PdIMe_3$(bipy) in acetone occurs predominantly via ionization of iodide with subsequent elimination of ethane from the cation $[Me_3(bipy)Pd]^+$, with a minor pathway (not retarded by added iodide) which may involve either partial ionization of iodide, $[Me_3(bipy)Pd^{\delta+} \cdots I^{\delta-}]$, or tight ion pair formation, $[Me_3(bipy)Pd]^+I^-$, prior to reductive elmination [15]. The complexes *fac*-$PdIMe_3(L_2)$ (L_2 = bipy, phen) may be readily isolated

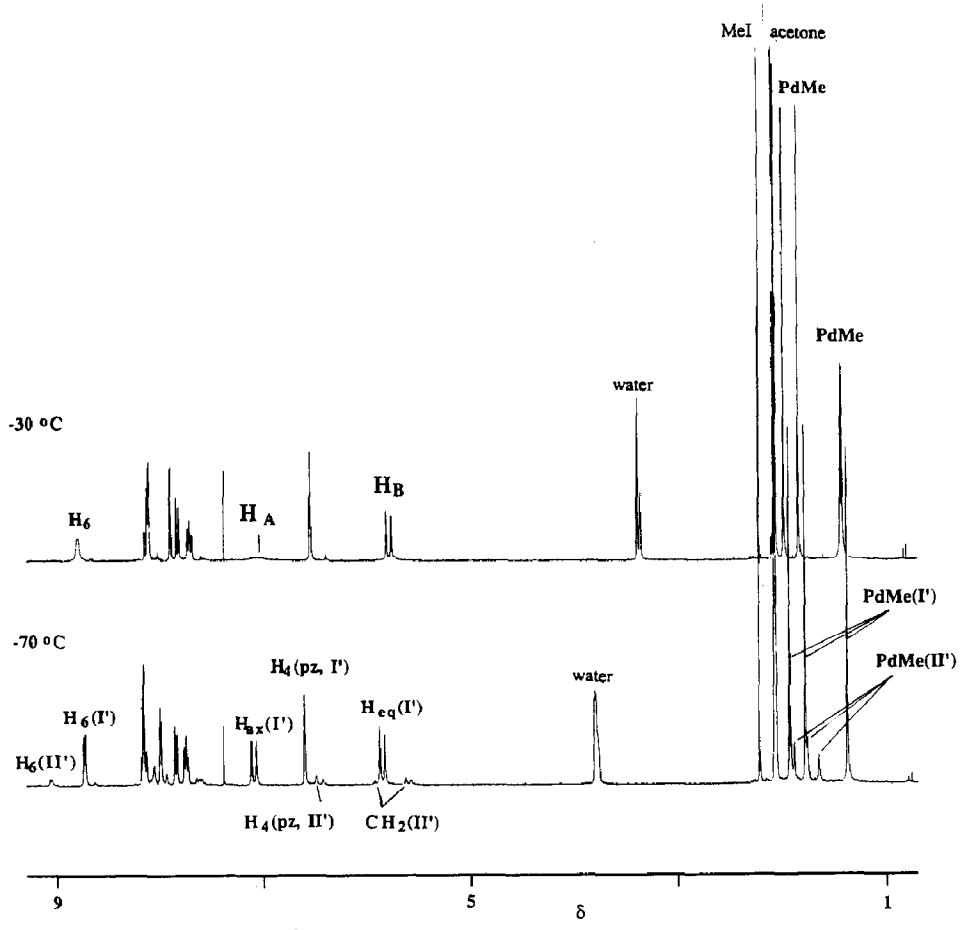


Fig. 3. Variable temperature ^1H NMR spectra for *fac*-$PdIMe_3\{(py)(pz)CH_2\}$, obtained after addition of an excess of MeI to $PdMe_2\{(py)(pz)CH_2\}$ at $-30\,^{\circ}C$.

at ca. 0 °C and stored satisfactorily at ca. −20 °C, although the solids and solutions of the complexes do slowly undergo reductive elimination of ethane near 0 °C [7]. The lower stability of complexes of the flexible bidentate ligands may be due to several factors; (e.g. the least stable complex *fac*-$PdIMe_3\{(pz)_2CH_2\}$ contains a ligand expected to be less basic [16] than the other ligands), but the most likely reason for the low stability of the complexes of ligands such as $(py)_2CHMe$, compared with $PdIMe_3(L_2)$ (L_2 = bipy, phen), may be that the greater flexibility of the ligand skeleton allows preferred geometries for reductive elimination to be more readily achieved. In addition to the fluxional behaviour of the ligands, which allows access to a range of geometries during the transition, the flexibility may allow easier access to a five-coordinate intermediate via dissociation of either the iodo group or one arm of the ligand.

Experimental

The reagents $(py)_2CHMe$ [17], $(pz)_2CH_2$ [18], $(py)(pz)CH_2$, $(pz)(mim)CH_2$, and $PdMe_2(L_2)$ [11], and $[PtMe_2(SEt_2)]_2$ [19] were prepared as previously described. Iodomethane was distilled in the dark and stored at −20 °C. Solvents were dried and distilled. Microanalyses were determined by the Australian Microanalytical Service, Melbourne, and the Canadian Microanalytical Service, Vancouver. ^1H NMR spectra were recorded with a Bruker AM 300 spectrometer, with chemical shifts given in ppm relative to Me_4Si, and studies of the reactivity of $MMe_2(L_2)$ complexes toward iodomethane were carried out as described for $PdMe_2(L_2)$ (L_2 = bipy, phen) [7].

Synthesis of complexes

$PtMe_2\{(py)_2CHMe\}$. A solution of 1,1-bis(pyridin-2-yl)ethane (0.054 g) in acetone (5 ml) was added to one of $[PtMe_2(SEt_2)]_2$ (0.092 g, 0.146 mmol) in acetone (15 ml), and the mixture was stirred with gentle warming for 15 min. Petroleum ether (10 ml) was added to the yellow solution, and the volume was reduced by rotary evaporation to give a yellow solid, which was collected and washed with petroleum ether (0.03 g, 0.073 mmol, 25%). ^1H NMR ($(CD_3)_2CO$): δ 8.91 (d, H(6)(**B**), J_{56} 4.9 Hz), 8.82 (d, H(6)(**A**), J_{56} 5.5 Hz), 7.99 (2H, m, H(4)), 7.64 (2H, m, H(3)), 7.35 (2H, m, H(5)), 5.28 (qb, CH_{ax}(**A**), J(H–Me) 7.4 Hz), 4.82 (qb, CH_{eq}(**B**), J(H–Me) 7.2 Hz), 2.44 (d, CMe_{ax}(**B**), J(H–Me) 7.2 Hz), 1.95 (d, CMe_{ax}(**B**), J(H–Me) 7.4 Hz), 0.80 (t, PtMe(**A**), J(H–Pt) 85.6 Hz), 0.76 (t, PtMe(**B**), J(H–Pt 85.9 Hz), with conformers **A** and **B** in ca. 3/5 ratio. Anal. Found: C, 40.7; H, 4.4; N, 6.6. $C_{14}H_{18}N_2Pt$ calcd.: C, 41.1; H, 4.4; N, 6.8%.

$PtMe_2\{(py)(pz)CH_2\}$ was obtained similarly, as a yellow crystals (84%). ^1H NMR ($CDCl_3$) δ: ambient, 9.00 (1H, m, H(6)), 7.92 (t, H(4)(py)), 7.79 (1H, d, H(3), J_{35} 2.2 Hz), 7.64 (1H, d, H(5)(pz), J_{45} 2.5 Hz), 7.41 (1H, d, H(3)(py)), 7.33 (1H, m, H(5)(py)), 6.35 (1H, t, H(4)(pz)), 5.45 (2H, b, CH_2), 0.91 (3H, t, PtMe, J(H–Pt) 86.0 Hz) and 0.86 (3H, t, PtMe, J(H–Pt) 85.5 Hz). At −40 °C, 8.96 (1H, d, H(6)(py)), 7.98 (1H, t, H(4)(py)), 7.81 (1H, s, H(3)), 7.74 (1H, s, H(5)), 7.51 (1H, d, H(3)(py)), 7.42 (1H, t, H(5)(py)), 6.42 (1H, s, H(4)), 5.86 (1H, d, CH_2) and 5.12 (1H, d, CH_2, J(H–H) 14.0 Hz), 0.89 (3H, t, PtMe) and 0.85 (3H, t, PtMe). Anal. Found: C, 34.7; H, 3.9; N, 10.8. $C_{11}H_{15}N_3Pt$ calcd.: C, 34.4; H, 3.9; N, 10.9%).

$PtMe_2\{(pz)(mim)CH_2\}$ was obtained similarly, as white crystals (91%). ^{1}H NMR ($(CD_3)_2CO$): δ 8.03 (2H, d, H(5)(pz), J_{45} 2.5 Hz), 7.74 (2H, d, H(3), J_{34} 2.2 Hz), 7.16 (1H, d, H(5)(mim)) and 7.11 (1H, m, H(4)(mim)), 6.36 (1H, t, H(4)), 5.51 (2H, s, CH_2), 3.91 (3H, s, NMe), 0.62 (3H, t, PtMe, J(H–Pt) 89.7 Hz) and 0.55 (3H, t, PtMe, J(H–Pt) 87.0 Hz). Anal. Found: C, 29.5; H, 4.2; N, 13.2. $C_{10}H_{16}N_4Pt$ calcd.: C, 31.0; H, 4.2; N, 14.4%.

$PtMe_2\{(pz)_2CH_2\}$. The ligand (0.32 mmol) was added to a suspension of $[PtMe_2(SEt_2)]_2$ (0.16 mmol) in benzene (20 ml) and the mixture was stirred and heated to reflux under nitrogen. Near to the reflux temperature a pale yellow solution was formed, and the product separated out after a further 10 min heating. It was collected, washed with diethyl ether, and air and vacuum dried (60 °C, 2 h) (90%). The complex in insoluble in organic solvents. Anal. Found: C, 28.9; H, 3.4; N, 14.9. $C_9H_{14}N_4Pt$ calcd.: C, 28.9; H, 3.8; N, 15.0%.

fac-$PtIMe_3\{(py)_2CHMe\}$. A five-fold excess of iodomethane was added to a solution of $[PtMe_2(SEt_2)]_2$ (0.11 g) and $(py)_2CHMe$ (0.065 g) in acetone. Hexane was added until crystallization commenced, and after further standing a white solid was collected (0.1 g, 52%). Anal. Found: C, 32.5; H, 3.7; N, 5.0. $C_{15}H_{21}N_2IPt$ calcd.: C, 32.7; H, 3.8; N, 5.1%.

fac-$PtIMe_3\{(py)(pz)CH_2\}$. Iodomethane (100 μl) was added to a solution of $PtMe_2\{(py)(pz)CH_2\}$ (0.1 g) in acetone (10 ml) and the mixture stirred for 10 min. Hexane was added until cloudiness developed, and needles separated during 30 min. The product was collected, washed with diethyl ether, and vacuum dried at 50 °C for 2 h (89%). Anal. Found: C, 27.6; H, 3.5 N, 7.9. $C_{12}H_{18}N_3IPt$ calcd.: C, 27.4; H, 3.5; N, 8.0%.

fac-$PtIMe_3\{(pz)(mim)CH_2\}$ was obtained similarly, in 88% yield. Anal. Found: C, 25.7; H, 3.7; N, 10.7. $C_{11}H_{19}N_4IPt$ calcd.: C, 25.0; H, 3.6; N, 10.6%).

fac-$PtIMe_3\{(pz)_2CH_2\}$. A procedure similar to that for the $(py)_2CHMe$ complex gave a crystalline product in 91% yield. Alternatively, an excess of MeI was added to a suspension of $PtMe_2\{(pz)_2CH_2\}$ in chloroform, and after 8 h stirring and filtration, and addition of hexane to the filtrate, the complex separated as a white solid (42%). In $CDCl_3$ the complex exhibits a spectrum identical to that reported by Clark et al. [12].

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