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Synthesis and isomerism of neutral and cationic dimethylplatinum(IV) complexes. Crystal structure of the bis(pyrazol-1-yl)(thien-2-yl)methane complex $PtI_2Me_2\{(pz)_2(thi)CH\text{-}N,N'\}$

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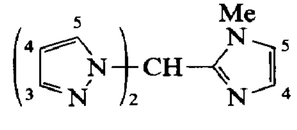
Abstract

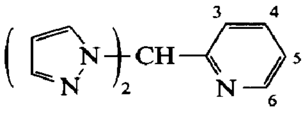
The reaction of $[PtMe_2(SEt_2)]_2$ with tripodal nitrogen donor ligands and iodine in dichloromethane gives diiododimethylplatinum(IV) complexes, $PtI_2Me_2\{(pz)_2(L)CH\text{-}N,N'\}$, in which the N,N'-bidentate ligands are *trans* to the *cis*-$PtMe_2$ group and have one uncoordinated donor group. The bis(pyrazol-1-yl)(thien-2-yl)methane complex, $PtI_2Me_2\{(pz)_2(thi)CH\text{-}N,N'\}$, has Pt–I bond lengths of 2.6199(7) and 2.6649(6) Å for the *trans*-PtI_2 group, and Pt–C bond lengths of 2.070(7) and 2.097(9) Å, and Pt–N bond lengths of 2.181(5) and 2.192(6) Å. Complexes of $(pz)_2(L)CH$ [L = *N*-methylimidazol-2-yl (mim), pyridin-2-yl (py)] exist as a mixture of isomers, with an isomer ratio as expected for a random distribution of coordinated pz and L donor groups, 2 : 1 for 2pz : (pz + L) coordination. The complexes of $(pz)_3CH$, $(pz)_2(mim)CH$, and $(pz)_2(py)CH$ are converted into $[PtIMe_2\{(pz)_2(L)CH\text{-}N,N',N''\}]I$ on heating, and the cations of the unsymmetrical tridentate ligands $(pz)_2(L)CH$ (L = py, mim) also exist as isomers. The isomers have the donor group L *trans* to a methyl or iodo group, where the ratio (L *trans* to Me) : (L *trans* to I) is 2 : 1 for L = py (as expected for a random distribution of isomers) and 1 : 1 for L = mim.

Introduction

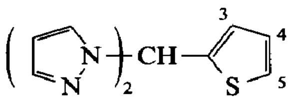
Poly(pyrazol-1-yl)methanes have been very useful ligands in the development of organoplatinum(IV) chemistry [1–7], e.g. for the first X-ray study of isostructural organoplatinum(IV) and palladium(IV) complexes, [*fac*-$MMe_3\{(pz)_3CH$-$N,N',N''\}$]I [6,7]. Tripodal ligands, e.g. tris(pyrazol-1-yl)methane, have not been explored as ligands for diorganoplatinum(IV), and thus, following our synthesis [4,8] of the unsymmetrical tripod ligands shown, we have prepared and characterized neutral complexes, $PtI_2Me_2\{(pz)_2(L)CH$-$N,N'\}$ (L = pz, mim, py, thi). Three of the neutral complexes form ionic complexes on heating, [$PtIMe_2\{(pz)_2(L)CH$-$N,N',N''\}$]I (L = pz, mim, py), and both the neutral and ionic forms exhibit isomerism for the ligands $(pz)_2(mim)CH$ and $(pz)_2(py)CH$.



($(pz)_2(mim)CH$)



($(pz)_2(py)CH$)

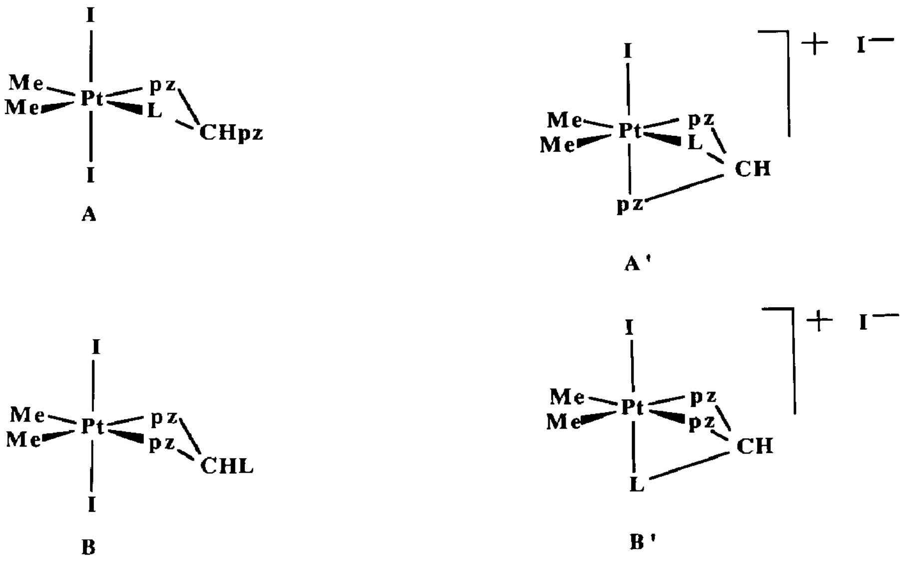


($(pz)_2(thi)CH$)

Results and discussion

$PtI_2Me_2\{(pz)_2(L)CH$-$N,N'\}$ (1–4)

The neutral dimethylplatinum(IV) complexes of $(pz)_3CH$ and the new ligands were obtained directly from $[PtMe_2(SEt_2)]_2$ in dichloromethane by addition of the ligand and iodine, following the approach developed by Clark et al. for the synthesis



$1/2[PtMe_2(SEt_2)]_2 + (pz)_2(L)CH \xrightarrow{I_2/CH_2Cl_2}$

$PtI_2Me_2\{(pz)_2(L)CH\text{-}N,N'\} \xrightarrow{\Delta} [PtIMe_2\{(pz)_2(L)CH\text{-}N,N'\}]I$

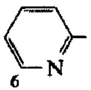
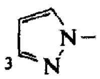
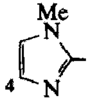
(**1**: L = pz;
2: L = mim, isomers **A** : **B** = 2 : 1;
3: L = py, isomers **A** : **B** = 2 : 1;
4: L = thi, isomer **B**)

(**1'**: L = pz;
2': L = mim, isomer **A'** : **B'** = 1 : 1;
3': L = py, isomer **A'** : **B'** = 2 : 1)

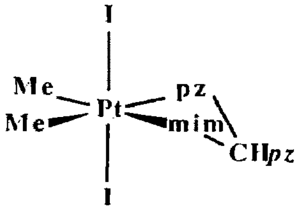
Scheme 1

Table 1

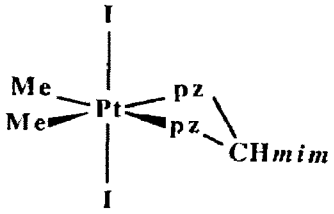
Selected ^{1}H NMR data for the complexes in $CDCl_3$

Complex [a]	PtMe [b]	CH	Coordinated groups [c]		
					
1: $PtI_2Me_2\{(pz)_3CH\text{-}N,N'\}$	2.67 (75.3, pz)	10.1		8.07m (Me) [d]	
1': $[PtIMe_2\{(pz)_3CH\text{-}N,N',N''\}]I$	1.95 (70.5, pz)	12.6		8.03m (Me) [e]	
				7.95'td' (13.2, I)	
2: $PtI_2Me_2\{(pz)_2(mim)CH\text{-}N,N'\}$ (2:1)					
Isomer A	2.49 (73.1, mim)	8.04		8.11m (Me) [e,f]	7.53'td' (9.6, Me)
	2.69 (75.1, pz)				
Isomer B	2.61 (75.8, pz)	9.48		8.07m (Me) [e]	[g]
2': $[PtIMe_2\{(pz)_2(mim)CH\text{-}N,N',N''\}]I$ (1:1)					
Isomer A'	1.78 (68.3, mim)	11.20 or		7.96d (Me) [e]	7.17m (Me)
	1.94 (70.8, pz)	11.25		7.17m (I)	
Isomer B'	1.93 (71.2, pz)	11.20 or		8.00d (Me) [e]	7.67'td' (12.8, I)
		11.25			
3: $PtI_2Me_2\{(pz)_2(py)CH\text{-}N,N'\}$ (2:1)					
Isomer A	2.60 (72.9, py)	9.61	9.26m (13.4, Me)	[h]	
	2.75 (74.3, pz)				
Isomer B	2.71 (75.1, pz)	9.31	[i]	[h]	
3': $[PtIMe_2\{(pz)_2(py)CH\text{-}N,N',N''\}]I$ (2:1)					
	1.96 (68.9) [j]	11.24 (A') [j]	[h]	[h]	
	1.93 (69.2) [j]	11.22 (B') [j]			
	1.90 (69.1) [j]				
4: $PtI_2Me_2\{(pz)_2(thi)CH\text{-}N,N'\}$	2.64 (74.8, pz)	9.36		8.09m (Me) [e]	

[a] Ratio of isomers A:B or A':B' in parentheses, estimated from relative intensities. [b] All as 't', with ^{2}J(HPt) and *trans* group in parentheses. [c] ^{3}J(HPt) and *trans* group in parentheses. [d] Includes H3(uncoordinated), but H4 and H5 exhibit separated resonances for coordinated and uncoordinated groups. [e] Insufficiently resolved to estimate ^{3}J(HPt). [f] H3(uncoord) at 7.58 d. [g] H4(uncoord) at 7.40d. [h] Complex overlapping resonances. [i] H6(uncoord) at 9.01dd. [j] PtMe resonances in 1:1:1 ratio, and CH resonances in 2:1 ratio, indicating that the A':B' ratio is 2:1.



A



B

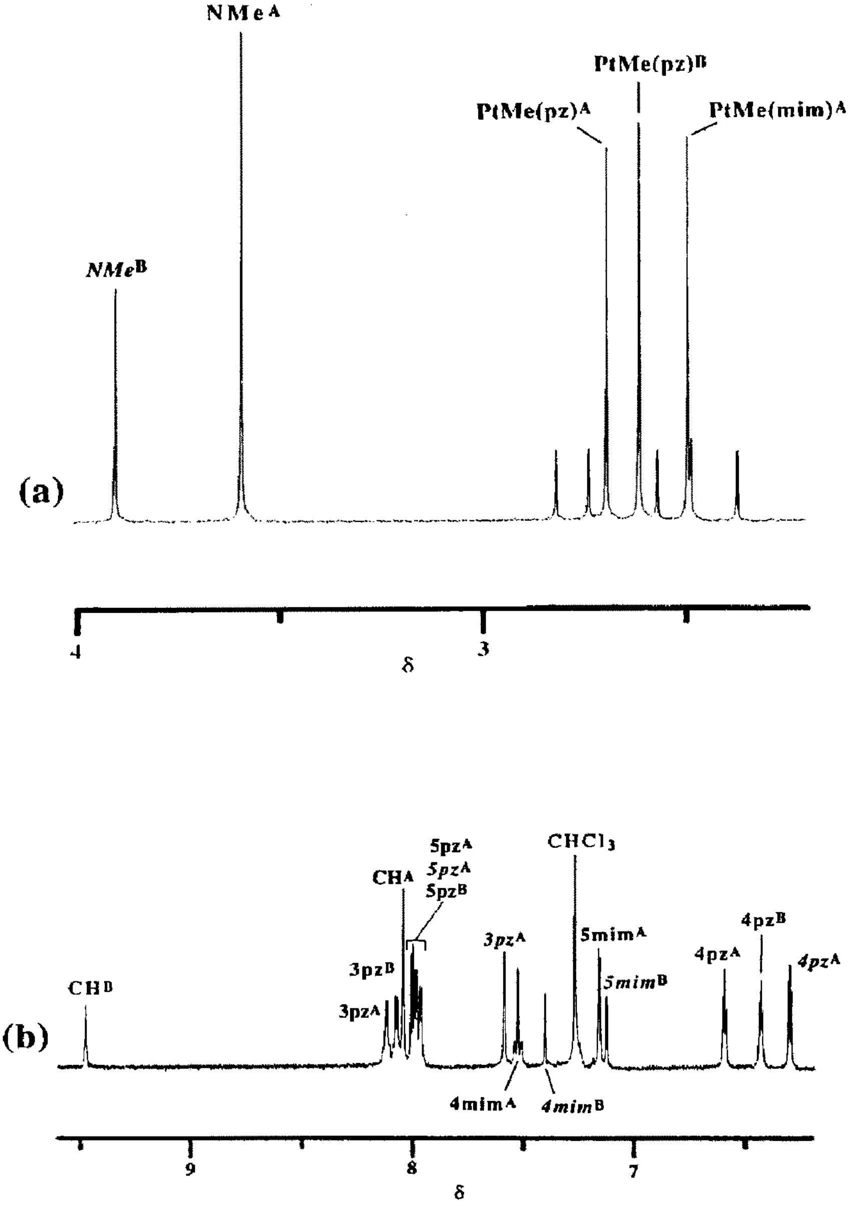


Fig. 1. ^{1}H NMR spectra of $PtI_2Me_2\{(pz)_2(mim)CH\text{-}N,N'\}$ (**2**) in $CDCl_3$, in the aliphatic (a) and aromatic (b) regions, illustrating presence of isomers **A** and **B**, and ^3J(HPt) for H4(mim) of isomer **A** (4mim^A). *Trans* groups are indicated in parentheses in (a), and protons of uncoordinated rings are shown in *italics* in (b).

of complexes of related bidentate ligands using $PtMe_2$(1,5-cyclooctadiene) as the $Pt^{II}Me_2$ substrate [1]. Complexes **1–4** are monomeric in chloroform and give non-conducting solutions in acetone, and ^1H NMR spectra in $CDCl_3$ (Table 1) are consistent with the general formulation $PtI_2Me_2\{(pz)_2(L)CH\text{-}N,N'\}$ (Scheme 1), confirmed by an X-ray structural analysis for the $(pz)_2$(thi)CH complex.

The spectra of **1** and **4** show a single PtMe environment, with **1** showing two pz environments (2 : 1 ratio) and **4** showing one pz and one thi environment, consistent with structure **B** [L = pz (**1**), thi (**4**)]. However, the spectra of **2** [Fig. 1(a) and 1(b)] and **3** indicate presence of a mixture of two isomers, with isomer **A** exhibiting two PtMe and pz resonances and isomer **B** exhibiting single PtMe and pz resonances, and with the isomers present in the ratio expected for a simple statistical distribution, 2 : 1. Assignment of groups *trans* to PtMe in the isomers follows directly from relative intensities and comparison of ^2J(HPt) in spectra of **1–4** and related complexes, e.g for **1–4** pz *trans* to PtMe gives ^2J(HPt) 74.3–75.9 Hz, mim trans to PtMe 72.2 Hz, and py *trans* to PtMe 72.9 Hz, compared with reported values of 75.4 Hz for $PtI_2Me_2\{(pz)_2CH_2\text{-}N,N'\}$ and 71.5 Hz for $PtI_2Me_2\{(pz)_2CH_2\text{-}N,N'\}$ [1], illustrating the effect of the weaker donor pyrazole ring [9] in giving rise to a higher ^2J(HPt) value.

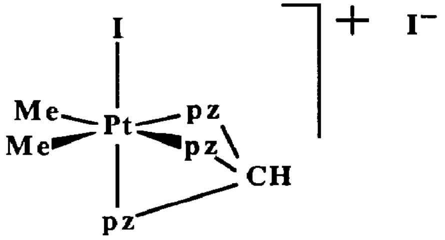
[PtIMe₂{(pz)₂(L)CH-N,N′,N″}]I (1′–3′)

Orange-brown crystals of $PtI_2Me_2\{(pz)_3CH\text{-}N,N'\}$ (**1**) become bright yellow on heating to ca. 140 °C, and decomposition occurs above ca. 230 °C. The yellow solid has a molar conductance (Ω_M) of 62 ohm^{-1} cm^2 mol^{-1} in acetone, and a ^1H NMR spectrum in $CDCl_3$ exhibits one PtMe resonance, and two pyrazole environments in the ratio 2 : 1 (Fig. 2), consistent with a cationic structure $[PtIMe_2\{(pz)_3CH\text{-}N,N',N''\}]I$. The PtMe resonance for the cation appears upfield from that for the neutral complex (Table 1); a similar effect has been noted for related trimethylplatinum(IV) complexes, e.g [*fac*-$PtMe_3(CD_3CN)$(bipy)}]$^+$ has δ(PtMe) 0.47 (^2J(HPt) 78 Hz) and 1.14 (68 Hz), whereas *fac*-$PtIMe_3$(bipy) has δ(PtMe) 0.58 (76 Hz) and 1.36 (70 Hz) [10]. Heating of a $CDCl_3$ solution of **1** to ca. 55 °C also results in conversion into **1′** during ca. 1 h, and the ^1H NMR signals from **1** decrease while those of **1′** increase.

Complexes **2** and **3** undergo similar conversion into cations **2′** and **3′** (Ω_M 57 and 72 ohm^{-1} cm^2 mol^{-1}, respectively), but complex **4** containing the weaker thienyl donor decomposes on heating. A random orientation of the $(pz)_2$(mim)CH and $(pz)_2$(py)CH ligands in the cation would give an **A′** : **B′** ratio of 2 : 1, as observed for **A** : **B** in the neutral complexes and for the cation **3′**, but the spectrum of **2′** indicates that the **A′** : **B′** ratio is ca. 1 : 1, and thus the isomer with *N*-methylimidazole *trans* to an iodo group (**B′**) is preferred for this cation. The cationic complexes (**1′–3′**) do not revert to the neutral forms (**1–3**) on cooling in the solid state or in $CDCl_3$.

Structure of PtI₂Me₂{(pz)₂(thi)CH-N,N′} (4)

A projection of the structure is shown in Fig. 3, with details of the coordination and chelate geometry in Table 2. The PtC_2N_2 group is planar [maximum deviation from the mean plane is 0.056(9) Å for C(1)], and the bidentate ligand adopts a boat conformation. The pyrazole rings, [N(1,2), C(3–5)] and [N(1′,2′), C(3′–5′)], are planar and they form angles of 30.3(2) and 30.4(3)°, respectively, with the PtC_2N_2 plane; the platinum atom is 0.16(1) and 0.42(1) Å removed from the respective pz



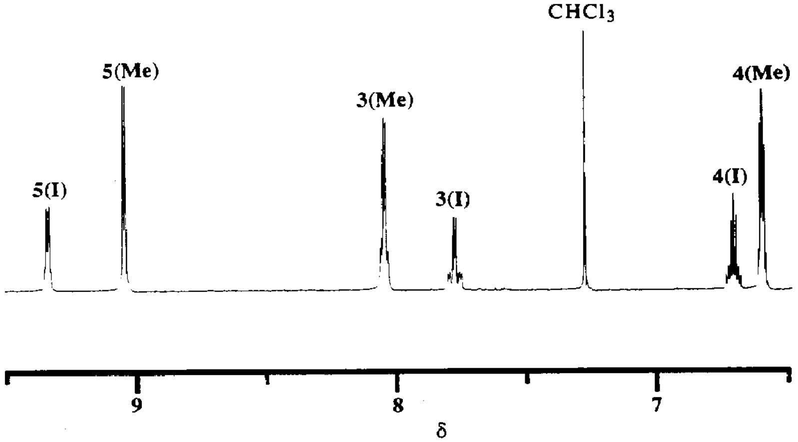


Fig. 2. ^{1}H NMR spectrum of $[PtIMe_2\{(pz)_3CH\text{-}N,N',N''\}]I$ in $CDCl_3$ in the pyrazole ring region, illustrating the 2:1 ratio for pz environments, and resolved J(HPt) coupling for the pz group *trans* to iodine [3(I), ^{3}J(HPt) 13.2 Hz; 4(I), ^{4}J(HPt) 11.1 Hz]. *Trans* groups are shown in parentheses.

mean planes. The planar thienyl ring is almost normal to the PtC_2N_2 plane [95.6(2)°], and forms angles of 120.8(2) and 67.0(3)° with the pz rings [N(1,2), C(3–5)] and [N(1′,2′), C(3′–5′)], respectively.

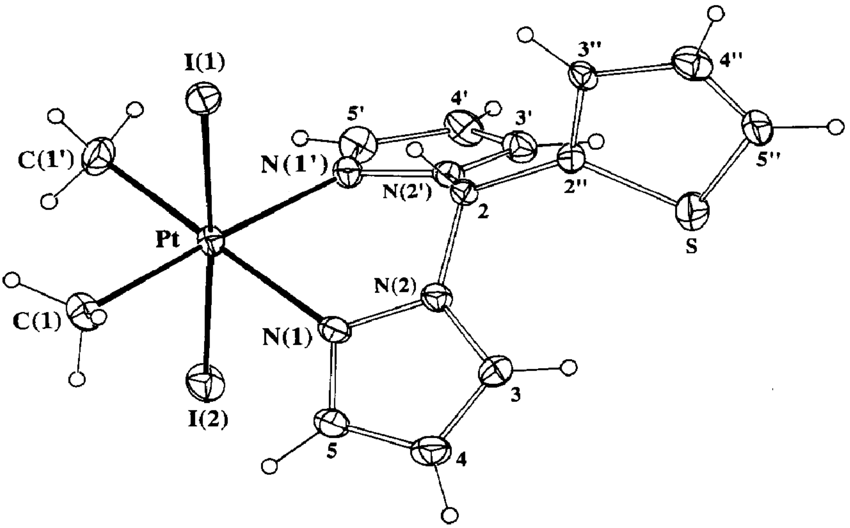


Fig. 3. The molecular structure of $PtI_2Me_2\{(pz)_2(thi)CH\}$. Hydrogen atoms are shown with an arbitrary radius of 0.1 Å, 20% thermal ellipsoids for the non-hydrogen atoms.

Table 2

Coordination and chelate ring geometry for $PtI_2Me_2\{(pz)_2(thi)CH\}$. Distances in Å, angles in degrees

Coordination geometry			
Pt–I(1)	2.6649(6)	Pt–N(1′)	2.181(5)
Pt–I(2)	2.6199(7)	Pt–C(1)	2.070(7)
Pt–N(1)	2.192(6)	Pt–C(1′)	2.097(9)
I(1)–Pt–I(2)	176.17(2)	C(1)–Pt–C(1′)	88.2(3)
I(1)–Pt–C(1)	87.9(2)	C(1)–Pt–N(1)	94.2(3)
I(1)–Pt–C(1′)	88.5(2)	C(1)–Pt–N(1′)	177.4(3)
I(1)–Pt–N(1)	93.5(1)	C(1′)–Pt–N(1)	176.9(3)
I(1)–Pt–N(1′)	94.3(1)	C(1′)–Pt–N(1′)	93.2(3)
I(2)–Pt–C(1)	88.5(2)	N(1)–Pt–N(1′)	84.4(2)
I(2)–Pt–C(1′)	89.9(2)	Pt–N(1)–N(2)	123.7(4)
I(2)–Pt–N(1)	88.2(1)	Pt–N(1)–C(5)	129.8(4)
I(2)–Pt–N(1′)	89.2(1)	Pt–N(1′)–N(2′)	122.3(4)
		Pt–N(1′)–C(5′)	129.8(4)
Chelate ring geometry			
N(1)–N(2)	1.356(7)	N(2′)–C(2)	1.462(8)
N(1′)–N(2′)	1.351(8)	C(2)–C(2″)	1.500(8)
N(2)–C(2)	1.460(8)		
N(1)–N(2)–C(2)	118.9(5)	N(2)–C(2)–N(2′)	110.2(4)
N(1′)–N(2′)–C(2)	119.8(5)	N(2)–C(2)–C(2″)	114.8(6)
C(2)–N(2)–C(3)	130.7(5)	N(2′)–C(2)–C(2″)	113.4(5)
C(2)–N(2′)–C(3′)	130.0(5)		

The most directly comparable complex for comparison of geometries is $PtI_2Me_2\{(pz)_2CH_2\}$, with a related complex $PtI_2Me_2\{(3,5\text{-}Me_2pz)_2CH_2\}$ somewhat less comparable owing to steric effects of the ligand 5-Me groups on the coordination geometry [1]. The coordination geometry of the $(pz)_2(thi)CH$ complex is similar to that of $PtI_2Me_2\{(pz)_2CH_2\}$, with corresponding bond angles agreeing within 2°, although the $(pz)_2CH_2$ complex has a crystallographic mirror plane through Pt, I(1), I(2) and C(2), giving identical Pt–C(1,1′) and Pt–N(1,1′) bond lengths. In the $(pz)_2(thi)CH$ complex, the respective values of Pt–C(1,1′) and Pt–N(1,1′) are within 2σ. The $(pz)_2CH_2$ complex has Pt–I(1) 2.651(1) Å, within 2σ of the value for Pt–I(2), 2.647(1) Å in the same complex, but the $(pz)_2(thi)CH$ complex has a Pt–I(1) bond length of 2.6649(6) Å, significantly longer than that of the Pt–I(2) bond, 2.6199(7) Å. In the $(pz)_2CH_2$ complex there is a short intramolecular contact of 2.837 Å between I(1) and a proton of the C(2) methylene bridge [1], and there is a similar contact of 2.75 Å in the present complex.

Experimental

The reagents $[PtMe_2(SMe_2)]_2$ [11], $(pz)_3CH$, $(pz)_2(mim)CH$, $(pz)_2(py)CH$, and $(pz)_2(thi)CH$ [8] were prepared as previously described, and acetone, hexane and pyridine were dried and distilled. Microanalyses were performed by the Canadian Microanalytical Service, Vancouver, and ^1H NMR spectra were recorded with a Bruker AM 300 spectrometer with chemical shifts given in ppm relative to Me_4Si. Molecular weights were determined with a Knauer vapor pressure osmometer for ca.

$1\text{–}3 \times 10^{-2}$ *M* solutions in chloroform at 37 °C, and molar conductances were measured with a Philips PW 9504/00 conductivity meter with a Griffin and George conductivity cell for ca. 10^{-3} M solutions in acetone at 25 °C.

Synthesis of $PtI_2Me_2(L)$ [L = tris(pyrazol-1-yl)methane, bis(pyrazol-1-yl)(N-methylimidazol-2-yl)methane, bis(pyrazol-1-yl)(pyridin-2-yl)methane, and bis(pyrazol-1-yl)(thien-2-yl)methane]

The ligand (0.30 mmol) and $[PtMe_2(SMe_2)]_2$ (0.15 mmol) were dissolved in dichloromethane (10 mL) and a solution of iodine (0.08 g, 0.32 mmol) in acetone (2 mL) was added dropwise with stirring until the colour of iodine persisted. The solution was taken to dryness by rotary evaporation and the excess of iodine extracted from the residue with warm hexane (3 × 20 mL). The residue was dissolved in dichloromethane (10 mL) and hexane added until cloudiness developed. The microcrystalline solids which separated on standing were collected, and air and vacuum dried for 2 h.

PtI_2Me_2((pz)$_3$CH-N,N′}, 95% yield, orange, isomerizes at ca. 140 °C. (Found: C, 20.5; H, 2.2; N, 12.1. $C_{12}H_{16}I_2N_6Pt$ calcd.: C, 20.8, H, 2.3; N, 12.2%). Mol. wt. Found: 669, calcd. 693.

PtI_2Me_2((pz)$_2$(mim)CH-N,N′}, 76% yield, orange, isomerizes at ca. 180 °C. (Found: C, 22.4; H, 2.7; N, 11.7. $C_{13}H_{18}I_2N_6Pt$ calcd.: C, 22.1; H, 2.6; N, 11.9%). Mol. wt. Found: 682, calcd. 707.

PtI_2Me_2((pz)$_2$(py)CH-N,N′}, 79% yield, orange, isomerizes at ca. 175 °C. (Found: C, 23.7; H, 2.4; N, 9.9. $C_{14}H_{15}N_5I_2Pt$ calcd.: C, 23.9; H, 2.4; N, 10.0%). Mol. wt. Found: 688, calcd. 704.

Table 3

Non-hydrogen atom coordinates and isotropic thermal parameters (Å^2) for $PtI_2Me_2\{(pz)_2(thi)CH\}$

Atom	*x*	*y*	*z*	*U*(Å^2)
Pt	0.83432(2)	0.78657(2)	−0.06748(4)	0.0351(2)
I(1)	0.74965(4)	0.99391(5)	0.13590(7)	0.0500(4)
I(2)	0.92956(4)	0.59156(5)	−0.27903(8)	0.0592(4)
C(1)	0.9328(7)	0.9107(7)	−0.271(1)	0.055(6)
C(1′)	0.9669(6)	0.7704(8)	0.056(1)	0.056(6)
N(1)	0.6944(5)	0.7924(5)	−0.1931(7)	0.039(4)
N(2)	0.5848(4)	0.7683(5)	−0.0950(7)	0.036(4)
C(3)	0.5199(6)	0.7693(7)	−0.213(1)	0.047(5)
C(4)	0.5904(7)	0.7908(8)	−0.388(1)	0.056(6)
C(5)	0.6975(6)	0.8037(7)	−0.3707(9)	0.048(5)
N(1′)	0.7304(4)	0.6500(5)	0.1371(7)	0.039(4)
N(2′)	0.6167(4)	0.6450(5)	0.1673(7)	0.037(4)
C(3′)	0.5784(6)	0.5357(7)	0.2691(9)	0.045(5)
C(4′)	0.6693(7)	0.4701(7)	0.303(1)	0.053(6)
C(5′)	0.7618(6)	0.5437(7)	0.219(1)	0.051(6)
C(2)	0.5532(5)	0.7558(6)	0.1094(8)	0.031(4)
S	0.3358(2)	0.6444(2)	0.1776(3)	0.064(2)
C(2″)	0.4270(5)	0.7567(6)	0.2067(9)	0.038(5)
C(3″)	0.3696(5)	0.8513(6)	0.3368(8)	0.034(4)
C(4″)	0.2533(6)	0.8247(7)	0.402(1)	0.054(6)
C(5″)	0.2245(6)	0.7199(8)	0.332(1)	0.058(6)

$PtI_2Me_2\{(pz)_2(thi)CH\text{-}N,N'\}$, 94% yield, black, decomposes at ca. 190 °C. (Found: C, 20.7; H, 2.3; N, 7.4. $C_{13}H_{16}I_2N_4PtS$ calcd.: C, 22.0; H, 2.3; N, 7.9%). Mol. wt. Found: 700, calcd. 708.

Structure determination. A unique data set was measured to $2\theta_{max} = 55°$ with an Enraf-Nonius CAD-4 diffractometer in conventional 2θ–θ scan mode, yielding 4126 independent reflections, 3286 with $I > 3\sigma(I)$ being considered observed and used in the full matrix least squares refinement after analytical absorption correction, and solution of the structure by the heavy atom method. Anisotropic thermal parameters were refined for the non-hydrogen atoms; $(x, y, z, U_{iso})_H$ were included at estimated values. Residuals R, R_w (statistical weights) quoted on F are 0.030, 0.034 respectively. Neutral complex scattering factors were used [12], and computation used the XTAL 3.6 program system implemented [13] by S.R. Hall on a Perkin-Elmer 3240 computer. Coordinates and isotropic thermal parameters for the non-hydrogen atoms are given in Table 3 *.

Crystal data. $PtI_2Me_2\{(pz)_2(thi)CH\}$, $C_{13}H_{16}N_4I_2PtS$, $M = 709.3$, triclinic, space group $P\bar{1}$ (C_i^1), $a = 12.291(2)$, $b = 10.310(2)$, $c = 7.486(1)$ Å, $\alpha = 86.69(2)$, $\beta = 72.37(1)$, $\gamma = 86.33(2)°$, $U = 901.5$ Å^3, D_c ($Z = 2$) 2.61 g cm^{-3}, $F(000) = 644$, monochromatic Mo-K_α radiation ($\lambda = 0.71069$ Å, $\mu = 108.2$ cm^{-1}). Specimen: $0.12 \times 0.15 \times 0.23$ mm. Minimum and maximum transmission factors 2.88, 4.85.

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* Tables of thermal parameters and calculated hydrogen atom positions, details of the ligand geometry, and a list of structure factors are available from the authors.