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Facial and meridional $[N–C–N]^-$ intramolecular coordination systems: structure of *fac*-$PtBrMe_2\{2,6-(pzCH_2)_2C_6H_3\}\cdot 1/2C_6H_6$ $\{[2,6-(pzCH_2)_2C_6H_3]^- = 2,6-(bis\{(pyrazol-1-yl)methyl\}phenyl)\}$ and *mer*-$PtBr\{2,6-(3,5-Me_2pzCH_2)_2C_6H_3\}$, and an alternative synthetic route to the platinum(II) $[N–C–N]^-$ kernel

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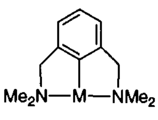
Abstract

Intramolecular coordination by the six-membered monoanionic N,C,N chelating system 2,6-(pyrazol-1-ylmethyl)phenyl and substituted derivatives can give rise to facial and meridional coordination, exhibited by structural studies of facial coordination in the platinum(IV) complex $PtBrMe_2\{2,6-(pzCH_2)_2C_6H_3\}\cdot 1/2C_6H_6$ and meridional coordination in the platinum(II) complex $PtBr\{2,6-(3,5-Me_2pzCH_2)_2C_6H_3\}$ showing close to 'ideal' geometry for the $[N–C–N]^-$ donor sets. A platinum(IV) complex is formed by oxidative addition of $2,6-(3,5-Me_2pzCH_2)_2C_6H_3Br$ to $[PtMe_2(SEt_2)]_2$, but reaction with $[Pt(p\text{-Tol})_2(SEt_2)]_2$ gives the platinum(II) complex $PtBr\{2,6-(3,5-Me_2pzCH_2)_2C_6H_3\}$ and 4,4′-bitolyl. © 2000 Elsevier Science S.A. All rights reserved.

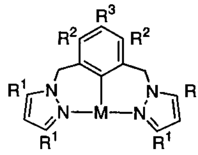
Keywords: Organoplatinum; Intramolecular coordination; Oxidative addition; Crystal structure

1. Introduction

Studies of tridentate intramolecular coordination systems $[N–C–N]^-$ have become one of the central themes in the development of organometallic chemistry, where the most widely examined system is the 'pincer' ligand 2,6-(dimethylaminomethyl)phenyl (**I**) [1,2]. This ligand forms two five-membered chelate rings and almost always behaves as a meridional $[N–C–N]^-$ donor, although in some complexes there are deviations from planarity of the $[N–C–N]^-$ donor set to give bonding modes best described as pseudo-facial with N–M–N angles of ~110–120° [2]. In recent years we [3–5] and others [6,7] have explored more flexible six-membered ring systems, in particular those employing pyrazole donor groups (**II**) that have the potential to facilitate either regular meridional or facial geometry at the metal centre [5,7].



I



II

M = Pd, Pt; $R^1 = R^2 = R^3 = H$ [5]
M = Pd, Ru; $R^1 = R^3 = H$, $R^2 = Me$ [7]
M = Pd, Ru; $R^1 = R^3 = H$, $R^2 = CH_2pz$ [7]

Structural studies of the meridional system (**II**) coordinated to palladium(II) and ruthenium(II) have been reported [7], and a platinum(IV) complex exhibiting spectroscopic properties consistent with the presence of **II** ($R^1 = R^2 = R^3 = H$) as a facial donor has been described [5]. We report here a structural study of this platinum(IV) complex and, following the exploration of the chemistry of **II** ($R^1 = R^3 = H$, $R^2 = Me$) to include both platinum(II) and platinum(IV), the development of a promising new route to $[N–C–N]^-$ coordination complexes for platinum(II) exhibited by the reaction of

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2,6-(3,5-dimethylpyrazol-1-yl)bromobenzene with [Pt(*p*-Tolyl)$_2$(SEt$_2$)]$_2$ to form *mer*-PtBr{2,6-(3,5-Me$_2$pzCH$_2$)$_2$-C$_6$H$_3$} and 4,4′-bitolyl.

2. Results and discussion

The reagent 2,6-bis{(3,5-dimethylpyrazol-1-yl)methyl}bromobenzene was synthesised by a procedure similar to that reported for 2,6-(pzCH$_2$)$_2$C$_6$H$_3$Br [5], involving the reaction of 2,6-bis(bromomethyl)-bromobenzene with 3,5-dimethylpyrazol-1-ylpotassium. The new reagent reacts with [PtMe$_2$(SEt$_2$)]$_2$ in the same manner as 2,6-(pzCH$_2$)$_2$C$_6$H$_3$Br, in an oxidative addition process to give the platinum(IV) complex **1** as shown in Scheme 1. However, it reacts differently with [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ to give the platinum(II) complex **2** and 4,4′-bitolyl. ^{1}H-NMR spectra for the [N–C–N]$^-$ group in **1** and **2** exhibit similar resonances for the methylene groups (*AB* pattern at ~5.5 and 4.8 ppm), aryl protons and methyl groups, but the spectrum of the platinum(II) complex also shows $^4J_{\mathrm{PtH}}$ (15.8 Hz) for the coordinated pyrazole rings. There is no indication of the formation of platinum metal in the synthesis of **1** or **2**.

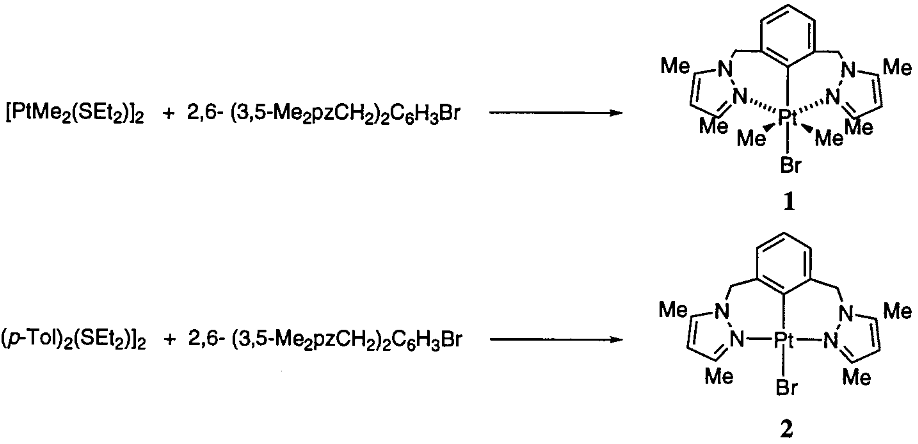
The platinum(II) complex **2** is assumed to be formed via oxidative addition to give an analogue of **1** followed by reductive elimination of 4,4′-bitolyl, consistent with the expected instability of triarylplatinum(IV) systems [8]. The platinum(II) reagent [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ was found to be stable at the reaction conditions used for the synthesis of **2** (reflux in benzene), and thus reaction via decomposition to platinum(0) followed by oxidative addition of 2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$Br is very unlikely.

Crystals of complex **2** that were suitable for X-ray structural studies were obtained by crystallisation from dimethylsulfoxide, but crystals of complex **1** could not be obtained, and thus the analogue PtBrMe$_2${2,6-(pzCH$_2$)$_2$C$_6$H$_3$} was synthesised as described [5] and its crystallisation behaviour explored. It was found to form suitable crystals on diffusion of benzene into a dichloromethane solution of the complex.

The platinum(IV) complex crystallises as a benzene hemisolvate, one formula unit comprising the asymmetric unit of the structure, as illustrated in Fig. 1, which also shows the facial configuration of the [N–C–N]$^-$ group and the putative *m* symmetry of the substrate molecule. Angles at platinum [83.8(2)–95.5(2)°] indicate slightly distorted geometry, and the platinum atom lies close to the mean planes of the pyrazole rings (Table 1). The *fac*-[N–C–N]$^-$ group forms angles at platinum that are within ca. 5° of 90° [86.6(2)–92.4(1)°].

The platinum(II) complex PtBr{2,6-(3,5-Me$_2$pz-CH$_2$)$_2$C$_6$H$_3$} (**2**) is shown in Fig. 2. The platinum atom has slightly distorted square-planar geometry, with the maximum deviation from the 'CN$_2$Br' mean plane exhibited by the carbon atom [0.165(22) Å]. As for the platinum(IV) complex, the C(1)–Pt–N(2a, 2b) angles are close to 90° [85.4(4), 88.6(4)°]. The six-membered chelate rings have quasi-boat conformations, in concert conforming to overall quasi-2- [rather than –*m*(*m*)–] symmetry for the molecule as a whole.

The coordination geometries for the Pt(N–C–N) moieties in both the platinum(II) and platinum(IV) complexes are significantly closer to 'ideal' geometry than for the 'pincer' (**I**) complex PtBr{2,6-(NMe$_2$-CH$_2$)$_2$C$_6$H$_3$} [C–Pt–N = 81.5(5), 82.9(5)°] [10], reflected also in N–Pt–N angles for the platinum(II) complexes [174.0(3) for **2**, 164.4(4)° for the 'pincer' complex]. The more 'ideal' geometry for both facial and meridional geometry results from the presence of six-membered chelate rings in **II** compared with five-membered in **I**, and it will be of interest to develop the chemistry of this system in view of the fascinating reports over an extended period for the 'pincer' system **I** [1,2].



Scheme 1.

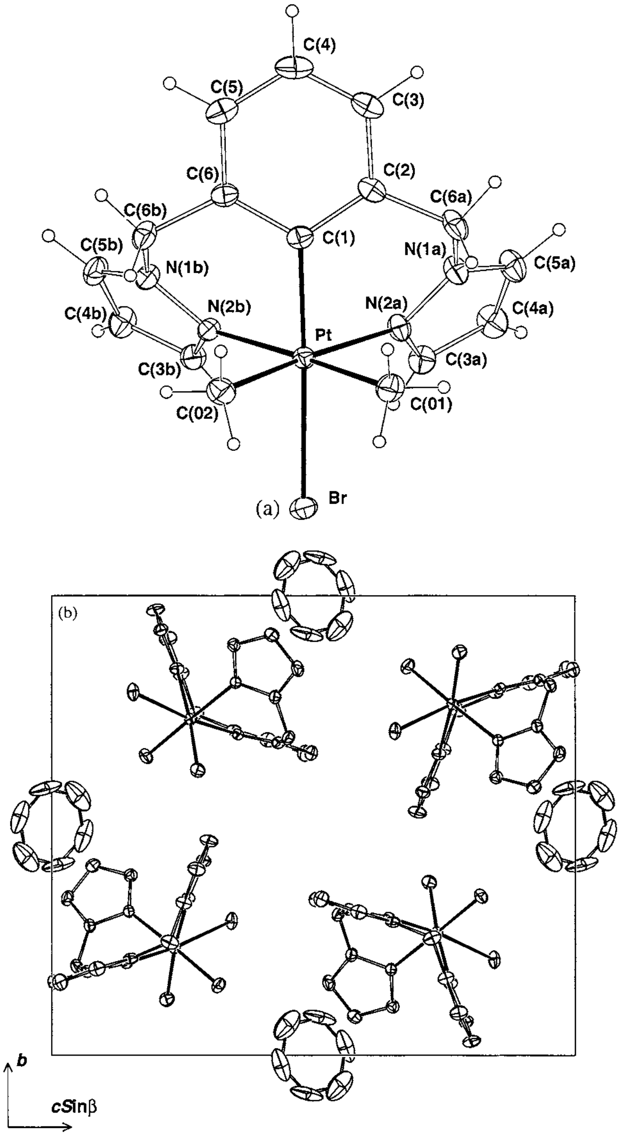


Fig. 1. (a) Projection of a molecule of $PtBrMe_2\{2,6\text{-}(pzCH_2)_2C_6H_3\}$ as its benzene hemisolvate showing 20% thermal ellipsoids for the non-hydrogen atoms, hydrogen atoms having an arbitrary radius of 0.1 Å. (b) Unit cell contents projected down *a*.

The synthesis of **2** by the reaction of a bromarene with an organoplatinum(II) substrate is also of interest, as it indicates that an alternative route to platinum(II) complexes of $[N–C–N]^-$ ligands may be developed to complement existing methods. For example, platinum(II) complexes of **I** are obtained by the reaction of the lithium derivative $Li[2,6\text{-}(Me_2NCH_2)_2C_6H_3]$ with platinum(II) substrates [9], but attempted synthesis of platinum(II) complexes of **II** ($R^1 = R^2 = R^3 = H$; $R^1 = Me$, $R^2 = R^3 = H$) were unsuccessful. Lithiation of $2,6\text{-}(pzCH_2)_2C_6H_3Br$ or $2,6\text{-}(3,5\text{-}Me_2pzCH_2)_2C_6H_3Br$ followed by D_2O quenching led to complex deuterium substitution patterns, including at the methylene, C_{ipso} and CH (pyrazole) positions. The synthetic method used to obtain **2** provides a potentially useful route for ligands where transmetallation procedures are unsuitable, and presumably relies on the suitability of the bromoarene reagent to facilitate oxidative addition to a diarylplatinum(II) centre and the susceptibility of triarylplatinum(IV) species to undergo reductive elimination of biaryl.

3. Experimental

The reagents 2,6-bis(bromomethyl)bromobenzene [11], $[PtMe_2(SEt_2)]_2$ [12], $[Pt(p\text{-}Tol)_2(SEt_2)_2]_2$ [13], and $PtBrMe_2\{2,6\text{-}(pzCH_2)_2C_6H_3\}$ [5] were prepared as described. Solvents were dried and distilled, stored under nitrogen and freshly distilled immediately before use. NMR spectra were recorded with a Varian Gemini 200 spectrometer operating at 50.29 (^{13}C), 199.975 (^1H) MHz, at room temperature. Chemical shifts are given in ppm relative to $SiMe_4$. GC–MS analyses were performed using an HP5890 gas chromatograph equipped with an HP5790 MSD and a 25 m × 0.32 mm HPI column (0.52 μm film thickness, He at 10 psi). Microanalyses were carried out by the Central Science Laboratory, University of Tasmania.

3.1. Synthesis of reagents

3.1.1. Bis(pyrazol-1-ylmethyl)bromobenzene

Bis(pyrazol-1-ylmethyl)bromobenzene was made by a modified procedure to that described earlier [5]. Pyrazole (2.62 g, 38.5 mmol) was added to a stirred suspension of finely cut potassium (1.4 g, 36.7 mmol) in tetrahydrofuran (60 ml) under an argon atmosphere. The mixture was heated to reflux and maintained at this temperature until beads of molten potassium were no longer evident (~ 1 h). The resultant white suspension was cooled to ambient temperature and 2,6-bis(bromomethyl)bromobenzene (6.0 g, 17.5 mmol) added in one portion. The reaction mixture was refluxed for 15 h, then quenched by addition of water (0.1 ml), filtered and the solvent removed in a vacuum. The resultant oily solid was dissolved in dichloromethane (50 ml), filtered through Celite and reduced to ~ 10 ml. The product crystallised on addition of petroleum ether (60–80°), (3.63 g, 66%). M.p.: 95°C. ^1H-NMR ($CDCl_3$, 200 MHz): δ 7.59 (d, $^3J_{HH} = 1.8$ Hz, 2, *H*3-pz), 7.46 (d, $^3J_{HH} = 2.1$ Hz, 2, *H*5-pz), 7.19 (t, $^3J_{HH} = 7.7$ Hz, 1, *H*4-C_6H_3), 6.78 (d, $^3J_{HH} = 7.8$ Hz, 2, *H*3, *H*5-C_6H_3), 6.32 ('t', 2, *H*4-pz), 5.47 (s, 4, $C_6H_3(CH_2)_2$). ^{13}C-NMR ($CDCl_3$, 50 MHz): δ 139.9, 137.1, 129.9, 128.8, 128.0, 123.0, 106.0, 55.9 (*C*H_2).

3.1.2. 2,6-Bis{(3,5-dimethylpyrazol-1-yl)methyl}bromobenzene

3,5-Dimethylpyrazole (2.47 g, 25.7 mmol) was added to a stirred suspension of finely cut potassium (0.96 g,

Table 1
Selected bond distances (Å) and angles (°) for PtBrMe$_2${2,6-(pzCH$_2$)$_2$C$_6$H$_3$}·1/2C$_6$H$_6$ [a] and PtBr{2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$} (**2**) [b]

	PtBrMe$_2${2,6-(pzCH$_2$)$_2$C$_6$H$_3$}.1/2C$_6$H$_6$	PtBr{2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$} (**2**)
Bond distances		
Pt–C(1)	2.021(4)	2.025(9)
Pt–N(2a, 2b)	2.138(4), 2.150(4)	2.036(9), 2.004(8)
Pt–Br	2.5764(5)	2.536(1)
Pt–C(01, 02)	2.059(5), 2.054(5)	
Bond angles		
C(1)–Pt–N(2a, 2b)	86.6(2), 85.4(2)	85.4(4), 88.6(4)
N(2a)–Pt–N(2b)	92.4(1)	174.0(3)
C(1)–Pt–Br	173.8(1)	170.9(5)
Br–Pt–N(2a, 2b)	90.6(1), 89.19(9)	90.3(2), 95.5(2)
C(1)–Pt–C(01, 02)	95.5(2), 94.6(2)	
C(01)–Pt–C(02), Br	83.8(2), 90.0(1)	
C(01)–Pt–N(2a, 2b)	90.6(1), 176.9(2)	
C(02)–Pt–Br	88.7(1)	
C(02)–Pt–N(2a, 2b)	174.3(2), 93.2(2)	
Pt–C(1)–C(2, 6)	121.3(3), 120.7(3)	125(1), 114(1)
Pt–N(2a)–N(1a), C(3a)	121.4(3), 133.0(3)	120.1(6), 132.3(8)
Pt–N(2b)–N(1b), C(3b)	121.4(3), 132.8(3)	119.2(6), 134.0(7)

[a] Deviation (Å) of Pt from mean planes of rings are 0.130(8) [pz(a)], 0.047(8) [pz(b)] and 0.003(7) (aryl). Interplanar dihedral angles (°) are 67.0(2) [pz(a)/pz(b)], 52.3(2) [pz(a)/aryl] and 59.5(2) [pz(b)/aryl].

[b] Deviation (Å) of Pt from mean planes of rings are 0.066(6) (coordination), 0.16(2) [pz(a)], 0.20(2) [pz(b)], 0.07(2) (aryl). Deviations (Å) from the 'N$_2$CBr' mean coordination plane are 0.17(2) [C(1)], −0.06(1) [N(2a)], −0.03(1) [N(2b)] and 0.001(2) (Br), 0.066(6) (Pt). Interplanar dihedral angles (°) are 48.1(4) [coord/pz(a)], 43.7(4) [coord/pz(b)], 42.7(4) (coord/aryl), 90.3(5) [pz(a)/pz(b)], 64.5(5) [pz(a)/aryl], and 65.6(5) [pz(b)/aryl].

24.6 mmol) in tetrahydrofuran (50 ml) under an argon atmosphere. The mixture was heated to reflux and maintained at this temperature until beads of molten potassium were no longer evident (~ 3 h). The solution was cooled to ambient temperature and 2,6-bis(bromomethyl)bromobenzene (4.0 g, 11.7 mmol) added in one portion. The reaction mixture was refluxed for 15 h, then quenched by addition of water (0.1 ml), filtered and the solvent removed in a vacuum. The resultant oily solid was dissolved in dichloromethane (40 ml) and the solution filtered then reduced to ca. 10 ml. Petroleum ether (60–80°) (40 ml) was added and the solution again reduced to ca. 10 ml. A white solid precipitated which was washed with petroleum ether (60–80°) (2 × 20 ml) and dried in a vacuum to give a white powder (2.31 g, 50%). M.p.: 114–115°C. ^{1}H-NMR (CDCl$_3$, 200 MHz): δ 7.10 (t, 3, $J_{HH} = 7.8$ Hz, 1, H4-C$_6$H$_3$), 6.32 (d, $^{3}J_{HH} = 7.8$ Hz, 2, H3, H5-C$_6$H$_3$), 5.92 (s, 2, H4-pz), 5.32 (s, 4, C$_6$H$_3$(CH_2)$_2$), 2.28 (s, 6, C3-CH_3-pz), 2.15 (s, 6, C5-CH_3-pz). ^{13}C-NMR (CDCl$_3$, 50 MHz): δ 148.1, 139.8, 137.2, 128.1, 126.3, 120.4, 105.6, 52.7 (C$_6$H$_3$(CH$_2$)$_2$), 13.4 (C3-CH$_3$-pz), 10.8 (C5-CH$_3$-pz). Anal. Calc. for $C_{13}H_{20}N_4$, C: 57.92; H: 5.67; N: 15.01. Found, C: 57.72; H: 5.43; N: 14.92%.

3.2. Synthesis of complexes

3.2.1. PtBrMe$_2${2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$} (*1*)

A solution of [PtMe$_2$(SEt$_2$)]$_2$ (60 mg, 0.10 mmol) and 2,6-bis{(3,5-dimethylpyrazol-1-yl)methyl}bromobenzene (74 mg, 0.20 mmol) in benzene (4 ml) was heated at reflux for 2 h. The solution was reduced in a vacuum to ca. 0.5 ml and a white product precipitated. The solution was decanted and the product washed with diethyl ether (3 × 2 ml) and dried in a vacuum (106 mg, 92%). ^{1}H-NMR (CDCl$_3$, 200 MHz): δ 7.09–7.05 (m, 3, H3,4,5-C$_6$$H_3$), 5.93 (s, 2, H4-pz), 5.54 (d, $^{2}J_{HH} = 15.3$ Hz, 2, C$_6$H$_3$(CH_2)$_2$), 4.80 (d, $^{2}J_{HH} = 15.3$ Hz, 2, C$_6$H$_3$(CH_2)$_2$), 2.88 (s, 6, C3-CH_3-pz), 2.29 (s, 6, C5-CH_3-pz), 1.88 (s, $^{2}J_{PtH} = 72.4$ Hz, 6, Pt-CH_3). ^{13}C-NMR (CDCl$_3$, 75 MHz): δ 153.7, 141.1, 137.9, 132.9, 129.6, 125.3, 109.0, 54.7 [$^{3}J_{PtC} = 28.3$ Hz, C$_6$H$_3$(CH$_2$)$_2$], 17.7, 12.6, −2.8 ($^{1}J_{PtC} = 670.6$ Hz, PtCH$_3$). Anal. Calc. for $C_{20}H_{27}BrN_4Pt$, C: 40.14; H: 4.55; N: 9.36. Found, C: 40.17; H: 4.49; N: 9.41%.

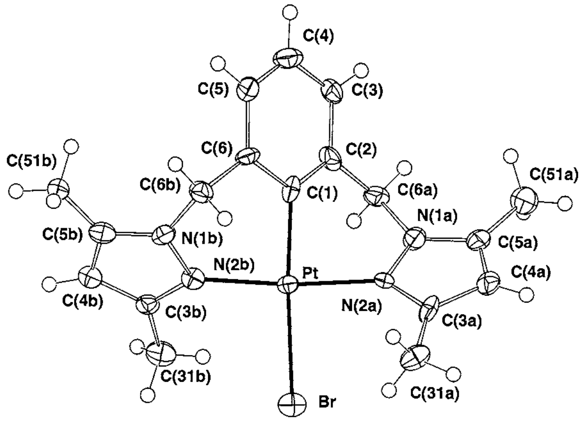


Fig. 2. Projection of a molecule of PtBr{2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$} showing 50% thermal ellipsoids for the non-hydrogen atoms, hydrogen atoms having an arbitrary radius of 0.1 Å.

Table 2
Specific crystallographic details for $PtBrMe_2\{2,6\text{-}(pzCH_2)_2C_6H_3\}\cdot 1/2C_6H_6$ and $PtBr\{2,6\text{-}(3,5\text{-}Me_2pzCH_2)_2C_6H_3\}$ (**2**)

	$PtBrMe_2\{2,6\text{-}(pzCH_2)_2C_6H_3\}\cdot 1/2C_6H_6$ [a]	$PtBr\{2,6\text{-}(3,5\text{-}Me_2pzCH_2)_2C_6H_3\}$ (**2**)
Empirical formula	$C_{19}H_{22}BrN_4Pt$	$C_{18}H_{21}BrN_4Pt$
Space group	$P2_1/c$	$Pna2_1$
a (Å)	8.1856(8)	14.650(5)
b (Å)	14.673(1)	11.293(4)
c (Å)	16.799(2)	10.742(4)
V (Å)	2005.0(6)	1777(2)
Z	4	4
Molecular weight	581.4	568.38
$D_{calc.}$ (g cm^{-3})	1.926	2.124
F(000)	1108	1080
Crystal size (mm)	$0.25\times 0.10\times 0.10$	$0.11\times 0.07\times 0.04$
μ (cm^{-1})	90	101
'T'$_{min,max}$	0.49, 0.83	0.46, 0.78
T (K)	300	153
N (R_{int})	5066 (0.035)	2378 (0.042)
N_o	3518	1974
R, R_w	0.027, 0.027	0.031, 0.037

[a] $\beta = 96.406(2)°$

3.2.2. *PtBr{2,6-(3,5-Me$_2$pzCH$_2$)$_2$C$_6$H$_3$}* (**2**)

A mixture of $[Pt(p\text{-Tolyl})_2(SEt_2)]_2$ (54 mg, 0.06 mmol) and 2,6-bis{(3,5-dimethylpyrazol-1-yl)methyl}-bromobenzene (49 mg, 0.13 mmol) was heated at reflux in benzene for 2 h. The volatiles were then removed in a vacuum and the product was washed with diethyl ether (3 × 2 ml) and dried in a vacuum to give a white powder (52 mg, 73%). ^{1}H-NMR ($CDCl_3$, 200 MHz): δ 6.92 (m, 3, H3,4,5-C_6H_3), 5.86 (s, $^4J_{PtH} = 15.8$ Hz, 2, H4-pz), 5.50 (d, $^2J_{HH} = 14.0$ Hz, 2, C_6H_3-$(CH_2)_2$), 4.75 (d, $^2J_{HH} = 14.0$ Hz, 2, $C_6H_3(CH_2)_2$), 2.62 (s, 6, C3-CH_3-pz), 2.36 (s, 6, C5-CH_3-pz). ^{13}C-NMR ($CDCl_3$, 75 MHz): δ 153.1, 140.6. 137.3, 129.9, 125.2, 123.7, 107.8, 54.9, 17.2, 12.5. Anal. Calc. for $C_{18}H_{21}BrN_4Pt$, C: 38.04; H: 3.72; N: 9.86. Found, C: 38.53; H: 4.01; N: 9.50%. The combined organic residues were dried in a vacuum and the product purified on a short silica column. The product was identified as 4,4′-bitolyl by GC-MS and comparison of its ^{1}H-NMR spectrum with that of an authentic sample (17 mg, 73%).

3.3. *X-ray structure determinations for PtBrMe$_2${2,6-(pzCH$_2$)$_2$C$_6$H$_3$}·1/2C$_6$H$_6$ and PtBr{2,6-(3,5-Me$_2$pzCH$_2$)C$_6$H$_3$* (**2**)

For each complex a full sphere of absorption corrected area-detector diffractometer data (Bruker CCD instrument; $2\theta_{max} = 58°$; monochromatic Mo–K$_\alpha$ radiation, $\lambda = 0.7107_3$ Å) was measured yielding 22 373 and 11 309 reflections respectively, merging to N unique (R_{int} quoted), N_o with $F > 4\sigma(F)$ being considered 'observed' and used in the full-matrix least-squares refinement (anisotropic thermal parameter refinement for the non-hydrogen atoms; $(x, y, z, U_{iso})_H$ included constrained). Computation used the XTAL 3.4 program system [14]. Specific crystallographic details are given in Table 2. Higher 'thermal motion' on the benzene solvent molecules in the platinum(IV) complex, centrosymmetrically disposed about crystallographic inversion centres, probably indicates unresolved disorder.

4. Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 140687, 140688). Copies of the information can be obtained from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

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