# Dinuclear organoplatinum(II) complexes containing *N*-methylbenzamide[1]

**Michael G. Crisp and Louis M. Rendina**

**Abstract:** The preparation and characterization of cationic, dinuclear complexes of the type *trans*-[$Pt(PPh_3)_2$(σ-$C_6H_4NHMe$)-μ-L-$Pt(PPh_3)_2$(σ-$C_6H_4NHMe$)]$(OTf)_2$ (L = 4,4′-bipyridine, 4,7-phenanthroline, 4,4′-dipyrazolylmethane, and 1,1′-diphenyl-4,4′-dipyrazolylmethane; OTf = trifluoromethanesulfonate (triflate)) containing two $C^3$-*N*-methylbenzamide ligands are described. The key structural feature of the cationic complexes is the presence of two convergent amide groups that may allow for charge-assisted, H-bonding interactions in solution with suitable heteroaromatic guest molecules such as nucleobases.

*Key words:* organoplatinum(II) complex, organometallic host, dinuclear complex, *N*-methylbenzamide.

**Résumé :** On décrit la préparation et la caractérisation de complexes cationiques dinucléaires du type *trans*-[$Pt(PPh_3)_2$ (σ-$C_6H_4NHMe$)-μ-L-$Pt(PPh_3)_2$(σ-$C_6H_4NHMe$)]$(OTf)_2$ [L= 4,4′-bipyridine; 4,7-phénanthroline; 4,4′-dipyrazolylméthane; et 1,1′-diphényl-4,4′-dipyrazolylméthane; OTf = trifluorométhanesulfonate (triflate)] contenant deux ligands $C^3$-*N*-méthylbenzamide. La caractéristique structurale clé des complexes cationiques est la présence de deux groupes amides convergents qui, en solution, peuvent favoriser des interactions de liaisons hydrogènes assistées par des charges avec des molécules hôtes hétéroaromatiques appropriées, telles des nucléobases.

*Mots-clés :* complexe organique du platine(II), hôte organométallique, complexe dinucléaire, *N*-méthylbenzamide.

[Traduit par la Rédaction]

## Introduction

There is great interest in the study of synthetic receptors for the selective binding of nucleobases (1, 2), owing to their many important functional roles in biological systems, e.g., intracellular energy transfer, signal transduction, and nucleic-acid synthesis. Various modes of hydrogen-bonding recognition have been shown to occur between nucleobases and simple carboxylic acids, for example, and it has been demonstrated that many aromatic carboxylic acids preferentially bind Hoogsteen sites, whereas aliphatic carboxylic acids bind preferentially at the Watson–Crick site (3, 4). In addition to hydrogen-bonding, π-stacking is usually found to augment the interaction involving aromatic hosts and nucleobase guests, and both macrocyclic and non-macrocyclic molecular receptors containing hydrogen-bonding and (or) π-stacking binding domains have been studied previously (5–18), all of which have the capacity to target nucleobases such as adenine and its derivatives. Theoretical studies have also complemented the work (10, 11).

Macrocyclic nucleobase receptors can discriminate their guest molecule by attributes such as size, electronic properties, nature of the hydrogen-bonding groups, and the π-stacking surface area (5, 6, 12). In contrast, non-macrocyclic "molecular tweezers" possess two binding sites with convergent functionality, which are linked together by a spacer unit (7–9, 13–18). Molecular tweezers usually possess one or more hydrogen-bonding functionalities, such as carboxylic acids or amides, and, in some cases, converging aromatic surfaces that have the capacity to undergo π-stacking interactions. Organometallic complexes in which direct coordination of adenine to the metal centre is augmented by H-bonding and π-stacking interactions are also known (19, 20), but to our knowledge there exist no examples of where metal coordination does not play a key role in the recognition motif. Instead, the formation of strong H-bonding interactions with the guest molecule is the result of the complementary shape of the cationic host, which can be further augmented by charge assistance (21).

We have previously reported the preparation, $pK_a$ data, and X-ray structures of σ-arylplatinum(II) complexes bearing carboxylic acid functionalities (22–24) some of which have been shown to possess interesting supramolecular properties in non-aqueous solution, e.g., the formation of discrete, multimeric cyclic arrays with nanoscale dimensions (24). The use of a protection–deprotection strategy was required in the oxidative addition reactions involving aryl iodides bearing a carboxylic acid functionality owing to their high reactivity with platinum(0) precursor compounds. Furthermore, in some cases, we found that carboxylic acids

Received 5 May 2008. Accepted 14 June 2008. Published on the NRC Research Press Web site at canjchem.nrc.ca on 16 September 2008.

*Dedicated to Prof. R.J. Puddephatt, FRS, on the occasion of his retirement and in recognition of his many outstanding contributions to organometallic chemistry.*

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[1]This article is part of a Special Issue dedicated to Professor R. Puddephatt.
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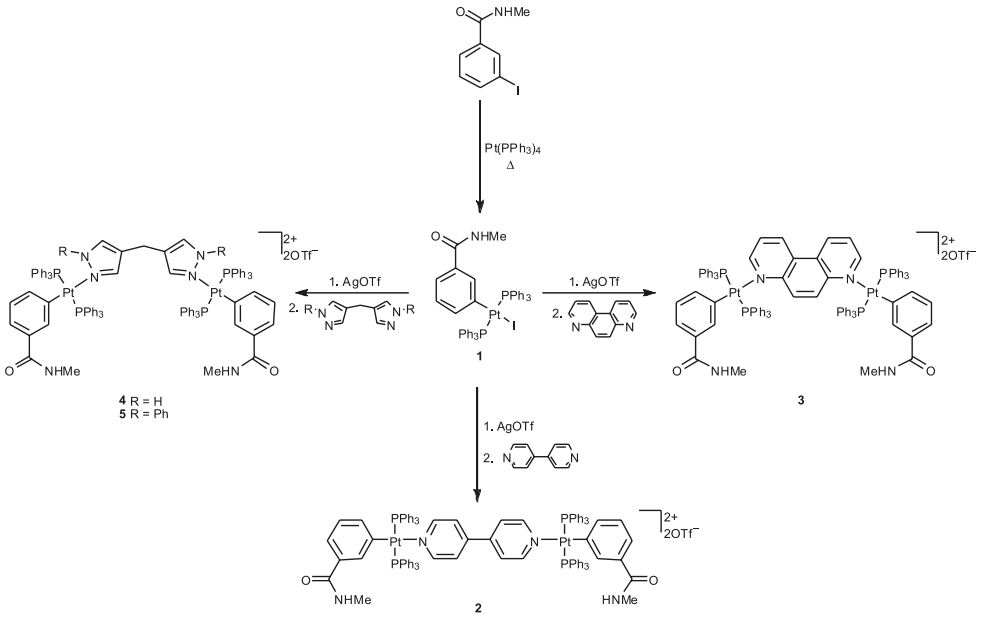
 doi:10.1139/V08-116 

**Scheme 1.**




readily H-bonded to counter-ions (e.g., triflate) both in low-polarity solvents and in the solid-state, thus markedly complicating the assembly and (or) molecular-recognition studies (22, 23). The use of N-substituted σ-benzamide ligands instead of pyridine carboxylic acids was expected to address many of these issues largely because of the markedly lower acidity of the amide N–H group compared with that of a carboxylic acid. Herein, we describe the synthesis and characterization of dinuclear organoplatinum(II) derivatives bearing convergent *N*-methylamide functionalities for potential application as molecular receptors for nucleobases.

## Results and discussion

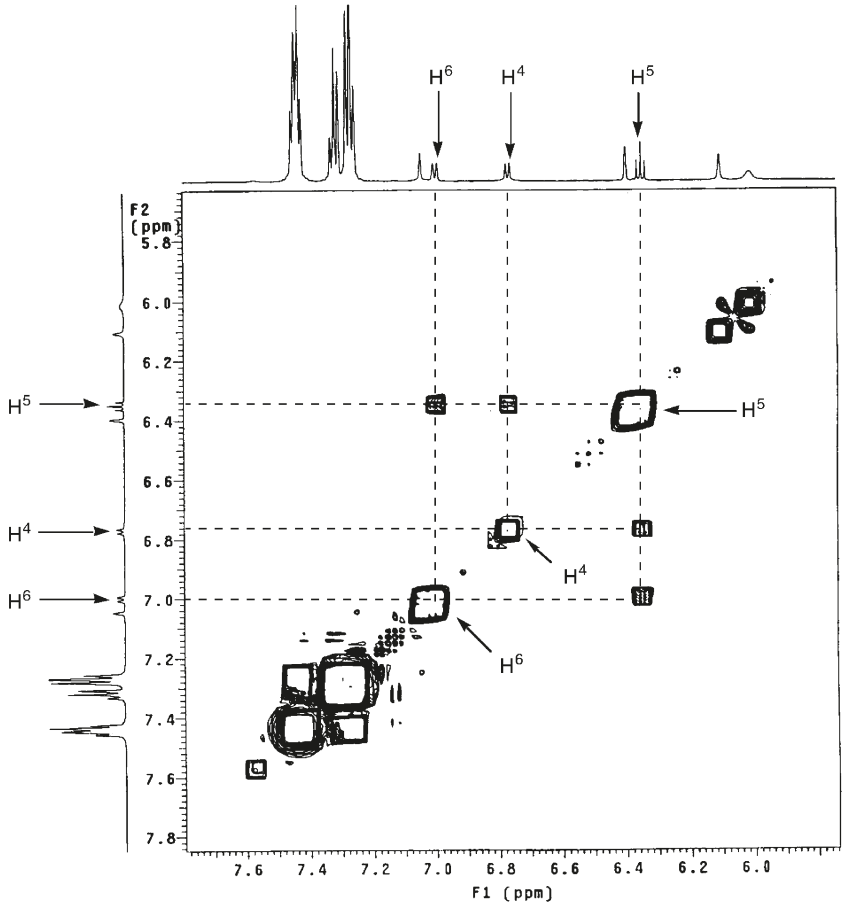
The oxidative addition of the C–I bond of 3-iodo-*N*-methylbenzamide to $Pt(PPh_3)_4$ proceeded cleanly to afford the iodoplatinum(II) complex **1** in good yield (Scheme 1). The $^1$H NMR spectrum of **1** displayed four distinct signals at δ 6.67, 6.72, 6.24, and 6.96 ppm, which are assigned to the $H^2$, $H^4$, $H^5$, and $H^6$ protons of the σ-aryl ligand, respectively. The methyl protons of the NHMe group appeared as a doublet at δ 2.76 ppm ($^3J_{HH}$ = 4.8 Hz), and the NH proton appeared as a broad multiplet at δ 4.98 ppm. Complex **1** was readily converted to the labile triflato derivative by the use of AgOTf. Two equivalents of this species were then treated immediately with 1 equiv. of the bridging bidentate, N-donor ligand 4,4′-bipyridine, 4,7-phenanthroline, 4,4′-dipyrazolylmethane, or 1,1′-phenyl-4,4′-dipyrazolylmethane to form the desired diplatinum(II) complexes (**2**–**5**) in good yield and purity (Scheme 1).

Complex **2** displayed a doublet signal in the $^1$H NMR spectrum at δ 7.03 ppm ($^3J_{HH}$ = 7.5 Hz), which is attributed to the $H^6$ proton of the σ-aryl ligand. The signal due to the $H^4$ proton appeared as a doublet at δ 6.81 ppm ($^3J_{HH}$ = 7.5 Hz), and the $H^5$ proton gave rise to a triplet signal at δ 6.39 ppm ($^3J_{HH}$ = 7.8 Hz). The ortho protons of the 4,4′-bipyridine were masked by the $PPh_3$ signals at δ 7.34–7.19 ppm, whilst the meta hydrogens appeared at δ 6.83 ppm as a distinct signal, which is assigned to the XX′ portion of a AA′XX′ spin system. The NMR spectra of **3** were measured in $CD_2Cl_2$ solution instead of $CDCl_3$ as the complex was found to dissociate to afford free 4,7-phenanthroline over a period of a few hours at room temperature in $CDCl_3$ but remained stable for indefinite periods in $CD_2Cl_2$ solution. This effect has been previously observed in the ESI-MS of these and related complexes (vide infra) (22–25), and it is most probably the result of the strong trans effect associated with the σ-aryl ligand. Interestingly, a $Pd_6$ hexagon prepared from 4,7-phenanthroline and dinuclear organopalladium(II) precursor complexes appears to remain intact both in solution and in the gas phase despite the presence of a σ-aryl ligand located trans to the coordinated N-atom (26).

The assignment of the aromatic proton resonances in **2**–**5** was facilitated by 2D $^1$H COSY (correlation spectroscopy) NMR experiments at 600 MHz, whereby distinct cross-peaks were observed for coupled protons of the σ-aryl ring systems, as seen in the NMR spectrum of **4** (Fig. 1). For this complex, protons $H^4$ and $H^6$ appeared as doublets at δ 6.77 and δ 7.00 ppm, respectively. The $H^5$ proton appeared as a triplet ($^3J_{HH}$ = 7.2 Hz) at δ 6.35 ppm. The signal due to $H^2$ appeared as a sharp singlet at δ 6.10 ppm. The N–Me protons appeared as a doublet at δ 2.83 ppm ($^3J_{HH}$ = 4.8 Hz), and the amide N–H proton gave rise to a broad singlet at δ 6.01 ppm. The signal due to the methylene bridge protons appeared as a sharp singlet at δ 2.53 ppm, and the N–H signal of the pyrazolyl ring was located downfield at δ 12.45 ppm.

Compared to the $^1$H NMR spectrum of complex **4**, the related complex **5** had many broad unresolved signals, with all the σ-aryl signals appearing as broad singlets. This effect is most likely related to the dynamic phenomena of analogous platinum(II) species (23), whereby free rotation of the 1,1′-diphenyl-4,4′-dipyrazolylmethane ligand about the Pt–N bond is restricted owing to the considerable steric interactions between the N–Ph group and the bulky $PPh_3$ ligands. This proposal is also supported by the fact that the related 4,4′-dipyrazolylmethane complex **4** does not exhibit this re-

**Fig. 1.** Expanded $^1$H COSY NMR spectrum (600 MHz) of **4** in $CDCl_3$ solution showing key couplings and peak assignments for the σ-aryl protons.




lated NMR behaviour, suggesting that in this case there exists free rotation around the Pt–N bond.

All complexes prepared in this work displayed a sharp singlet between δ 20–25 ppm in the $^{31}P\{^1H\}$ NMR spectrum, and it was always flanked by $^{195}Pt$ satellite signals. The magnitude of $^1J_{PtP}$ (~3000 Hz) for all complexes falls in the range that is expected for trans-substituted σ-arylplatinum(II) complexes (28, 29). Only minor differences were observed in the magnitude of $^1J_{PtP}$ upon substitution of the iodo ligand with N-donor ligands, largely because the $^{195}Pt$–$^{31}P$ coupling is predominantly influenced by the nature of the trans ligand ($PPh_3$).

Positive ion ESI-MS studies failed to show any evidence of an intact species in the spectra of **2**–**5** and, instead, the N-donor ligands (L) were readily lost, and peaks corresponding to $[M–OTf–L]^+$ were observed. The strong trans-effect associated with the σ-aryl ligand would account for the facile labilization of the N-donor ligand in the gas phase, and similar behaviour has also been observed in the ESI-MS of related systems (22–25).

## Conclusion

In this work, we have demonstrated the facile synthesis of organplatinum(II) complexes bearing convergent H-bonding amide functionalities by exploiting an oxidative addition reaction involving a platinum(0) precursor. The low reactivity of the amide N–H group ensures that unwanted side reactions involving the N–H bond do not occur, and C–I bond cleavage is favoured exclusively. We are in the process of investigating the "molecular chelation" properties of selected complexes with various guest molecules, such as derivatives of purine bases, including adenine and guanine. Indeed, preliminary molecular modelling studies (SYBYL force field) demonstrate that a stable 1:1 species between **4** and adenine is feasible in the gas phase in which the exocyclic $NH_2$, N-7, and N-1 atoms of the guest molecule are all involved in strong H-bonding interactions with both amide groups of the complex, and we are currently investigating the host–guest chemistry of **2**–**5** and related complexes in various solvents. The results of this work will be reported in due course.

## Experimental

All complexes were synthesised under a $N_2$ atmosphere using standard Schlenk techniques. All solvents were dried and purified in the following manner: $CH_2Cl_2$ was distilled from $CaH_2$, and toluene was pre-dried over $CaSO_4$ followed by distillation from sodium. The solvents were stored under $N_2$ over 4 Å molecular sieves.

All 1D NMR spectra were recorded at 298 K by means of a Varian Gemini 2000 NMR spectrometer with an Oxford 300 MHz magnet ($^1$H at 300.10 MHz, $^{31}$P at 121.50 MHz);

2D NMR ($^1$H COSY) spectra were recorded by using a 600 MHz magnet. $^1$H NMR chemical shifts were reported in ppm relative to tetramethylsilane (TMS). $^{31}$P{$^1$H} NMR spectra were referenced to sealed external standard of 85% phosphoric acid. IR spectra were recorded as Nujol mulls in the range 4000–400 cm$^{-1}$ on a PerkinElmer FTIR spectrophotometer. ESI-MS were obtained by means of a Finnegan LCQ mass spectrometer in the positive-ion mode using HPLC-grade MeOH as the solvent. Elemental analysis was performed by CMAS (Chemical and Microanalytical Services, Pty. Ltd.), Belmont, Victoria.

3-Iodo-*N*-methylbenzamide (27), tetrakis(triphenylphosphine)platinum(0) (30), 4,4′-dipyrazolylmethane (31), and 1,1′-phenyl-4,4′-dipyrazolylmethane (32) were prepared and purified according to the literature procedures.

### *Trans*-iodo(*N*-methylbenzamide-*C*$^3$)bis(triphenylphosphine)platinum(II) (1)

3-Iodo-*N*-methylbenzamide (0.110 g, 0.402 mmol) was placed in a Schlenk flask with $Pt(PPh_3)_4$ (0.500 g, 0.402 mmol). Toluene (20 mL) was added to the flask, and the solution was stirred for 16 h at 80 °C. Hexane (20 mL) was slowly added to the solution to precipitate the product, which was collected by filtration to afford **1** as a colourless, microcrystalline solid (0.344 g, 88%). IR (Nujol): 1632 ν(C=O) cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ: 7.56–7.22 (m, 30H, $PPh_3$), 6.96 (d, 1H, $^3J_{HH}$ = 7.5 Hz, $^1J_{PtH}$ = 29.3 Hz, H$^6$), 6.72 (d, 1H, $^3J_{HH}$ = 8.4 Hz, H$^4$), 6.67 (s, 1H, $^1J_{PtH}$ = 28.8 Hz, H$^2$), 6.24 (t, 1H, $^3J_{HH}$ = 7.8 Hz, H$^5$), 4.98 (br m, 1H, NH), 2.76 (d, 3H, $^3J_{HH}$ = 4.8 Hz, $NCH_3$). $^{31}$P{$^1$H} NMR ($CDCl_3$) δ: 22.0 ($^1J_{PtP}$ = 3029 Hz). Anal. calcd. for $C_{44}H_{38}INOP_2Pt$: C, 53.89; H, 3.91; N, 1.43. Found: C, 53.37; H, 3.85; N, 1.45.

### *Trans*-μ-4,4′-bipyridinebis[(*N*-methylbenzamide-*C*$^3$)bis(triphenylphosphine) platinum(II)] bis(triflate) (2)

Complex **1** (0.400 g, 0.401 mmol) was treated with AgOTf (0.103 g, 0.401 mmol) in $CH_2Cl_2$ (25 mL), and the reaction mixture was stirred at room temperature for 3 h in the absence of light. AgI was then removed by filtration through Celite filter aid. 4,4′-Bipyridine (0.031 g, 0.201 mmol) was added to the clear solution and then stirred overnight at room temperature. The solvent was reduced in vacuo to afford **2** as a colourless, microcrystalline solid (0.260 g, 60%). IR (Nujol): 1655 ν(C=O) cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ: 7.34–7.19 (m, 60H, $PPh_3$), 7.03 (d, 2H, $^3J_{HH}$ = 7.5 Hz, H$^6$), 6.83 (s, 2H, H$^2$), 6.83 (XX′ portion of AA′ XX′, 4H, $H_m$), 6.81 (d, 2H, $^3J_{HH}$ = 7.5 Hz, H$^4$), 6.39 (t, 2H, $^3J_{HH}$ = 7.8 Hz, H$^5$). $^{31}$P{$^1$H} NMR ($CDCl_3$) δ: 20.9 ($^1J_{PtP}$ = 3008 Hz). Anal. calcd. for $C_{44}H_{38}INOP_2Pt$: C, 55.56; H, 3.92; N, 2.59. Found: C, 55.62; H, 3.51; N, 2.64.

### *Trans*-μ-4,7-phenanthrolinebis[*N*-methylbenzamide-*C*$^3$)bis(triphenylphosphine) platinum(II)] bis(triflate) (3)

Following a procedure similar to that described for complex **2**, complex **1** (0.400 g, 0.401 mmol) was treated with AgOTf (0.103 g, 0.401 mmol) followed by the addition of 4,7-phenanthroline (0.036 g, 0.201 mmol) to afford **3** as a colourless, microcrystalline solid (0.360 g, 55%). IR (Nujol): 1641 ν(C=O) cm$^{-1}$. $^1$H NMR ($CD_2Cl_2$) δ: 9.40 (d, 2H, $^3J_{HH}$ = 9.3 Hz, H$^{1,10}$), 9.07 (d, 1H, $^3J_{HH}$ = 4.2 Hz, H$^{3,8}$), 9.05 (s, 2H, H$^{5,6}$), 7.41–7.11 (m, 20H, $PPh_3$), 6.88 (d, 2H, $^3J_{HH}$ = 7.8 Hz, H$^{14}$), 6.38 (t, 2H, $^3J_{HH}$ = 8.1 Hz, H$^{15}$), 5.96 (br s, 2H, NH), 2.84 (d, 6H, $^3J_{HH}$ = 4.8 Hz, NMe). $^{31}$P{$^1$H} NMR ($CD_2Cl_2$) δ: 19.5 ($^1J_{PtP}$ = 3003 Hz). Anal. calcd. for $C_{102}H_{84}F_6N_4O_8P_4Pt_2S_2$: C, 56.04; H, 3.87; N, 2.56. Found: C, 55.94; H, 3.45; N, 2.18.

### *Trans*-μ-4,4′-dipyrazolylmethanebis[(*N*-methylbenzamide-*C*$^3$)bis(triphenylphosphine)platinum(II)] bis(triflate) (4)

Following a procedure similar to that described for **2**, complex **1** (0.400 g, 0.401 mmol) was treated with AgOTf (0.103 g, 0.401 mmol) followed by the addition of 4,4′-dipyrazolylmethane (0.029 g, 0.201 mmol) to afford **4** as a colourless, microcrystalline solid (0.369 g, 81%). IR (Nujol): 1643 ν(C=O), 3400 ν(N–H) cm$^{-1}$. $^1$H NMR ($CDCl_3$) δ: 12.45 (s, 2H, pyrazolyl NH), 7.45–7.25 (m, 60H, $PPh_3$), 7.04 (s, 2H, H$^9$), 7.00 (d, 2H, $^3J_{HH}$ = 7.8 Hz, H$^6$), 6.77 (t, 2H, $^3J_{HH}$ = 7.2 Hz), 6.40 (s, 2H, H$^2$), 6.35 (t, 2H, $^3J_{HH}$ = 7.2 Hz, H$^5$), 6.10 (s, 2H, H$^{10}$), 6.01 (m, 2H, amide NH), 2.83 (d, 6H, $^3J_{HH}$ = 4.8 Hz, NHMe), 2.53 (s, 2H, $CH_2$). $^{31}$P{$^1$H} NMR ($CDCl_3$) δ: 20.8 ($^1J_{PtP}$ = 3022 Hz). Anal. calcd. for $C_{97}H_{84}F_6N_6O_8P_4Pt_2S_2$: C, 54.09; H, 3.93; N, 3.90. Found: C, 53.98; H, 4.09; N, 3.71.

### *Trans*-μ-(1,1′-phenyl-4,4′-dipyrazolylmethane)bis[(*N*-methylbenzamide- C$^3$)bis(triphenylphosphine)platinum(II)] bis(triflate) (5)

Following a procedure similar to that described for **2**, complex **1** (0.400 g, 0.401 mmol) was treated with AgOTf (0.103 g, 0.401 mmol) followed by the addition of 1,1′-phenyl-4,4′-dipyrazolylmethane (0.060 g, 0.201 mmol) to afford **5** as a colourless, microcrystalline solid (0.129 g, 54%). $^1$H NMR ($CDCl_3$) δ: 6.94 (br s, 2H, H$^6$), 6.69 (br s, 2H, H$^2$), 6.58 (br s, 2H), 6.25 (br s, 2H, H$^5$), 5.70 (m, 2H, NH), 3.11 (s, 2H, $CH_2$), 2.84 (d, 6H, $^3J_{HH}$ = 4.5 Hz, NHMe), 7.64–7.28 (m, 60H, $PPh_3$). $^{31}$P{$^1$H} NMR ($CDCl_3$) δ: 25.0 ($^1J_{PtP}$ = 3084 Hz). Anal. calcd. for $C_{109}H_{92}F_6N_6O_8P_4Pt_2S_2$: C, 56.77; H, 4.02; N, 3.64. Found: C, 56.57; H, 4.09; N, 3.49.

## Acknowledgements

We thank Mr Phil Clements (The University of Adelaide) for assistance with the NMR experiments. We also thank Johnson–Matthey for the generous loan of platinum salts. The authors wish to acknowledge funding of the work by the Australian Research Council (ARC).

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