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Note

Synthesis and NMR investigation of Pt(CN)₂(diphosphine) and [Pt(CN)(triphosphine)]Cl complexes

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Abstract

Pt(CN)₂(Ph₂P(CH₂)_nPPh₂) (n = 2,3,4) and Pt(CN)₂(P¹–P²) (P¹–P² = 1-diphenylphosphino-2,1'-[(1-diphenylphosphino)-1,3-propanediyl]-ferrocene, 1-diphenylphosphino-2,1'-[(1-dicyclohexylphosphino)-1,3-propanediyl]-ferrocene) were synthesised by reacting potassium cyanide and the corresponding PtCl₂(diphosphine) complexes. PtCl(CN)(diphosphine) complexes were identified as minor products when KCN/PtCl₂(diphosphine) molar ratio was kept below 2. The use of KCN in excess resulted in the formation of K₂Pt(CN)₄. [Pt(CN)({Ph₂P(CH₂)₂}₂PPh)]⁺ complex cation and Pt(CN)₂)({Ph₂P(CH₂)₂}₂PPh) five-coordinate covalent complex of fluxional behaviour were obtained at KCN/Pt ratio of 1 and 2, respectively. The platinum–cyano complexes were characterised by NMR spectroscopy. The direct Pt–CN bond was proved by ¹J(¹⁹⁵Pt, ³¹P), ²J(³¹P, ¹³C) coupling constants by using sodium cyanide-¹³C for ligand exchange reactions. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The high *trans* influence of the cyano ligand in robust squareplanar transition metal-bis(monodentate phosphine) complexes of Ni(II), Pd(II) and Pt(II) [1,2] as well as its π -acceptor ability [3] is known for a long time. Although even the catalytic activity of Pd(CN)₂(PPh₃)₂ and Pt(CN)₂(PPh₃)₂ in olefin hydrogenation has been tested [4], their coordination chemistry has remained relatively unexplored.

Although the $PtX_2(L)$ (X = Cl, Br, I; L = bidentate ligand) complexes are among the most investigated ones, surprisingly little is known about $Pt(CN)_2(L)$ -type

derivatives. Some synthetic and structural details on Pt(CN)₂(diphosphine)-type complexes have already been published, but the results are still sporadic. To the best of our knowledge, in the Ph₂P(CH₂)_nPPh₂ ligand series platinum-cyano complexes of dppm (bis(diphenylphosphino)methane, n = 1) and dppp (1,3-bis(diphenylphosphino)-propane, n = 3) have already been published. While the various reaction pathways with dppm furnished [Pt(dppm)(CN)₂]₂ and [Pt(dppm)(CN)]₂ dinuclear complexes containing dppm bridges, which were characterised X-ray crystallography by [5,6], Pt(CN)₂(dppp) mononuclear complex was selectively obtained with dppp [7]. With PtCl₂(PhCN)₂, the dppm analogue 2,2-dppp (2,2-bis(diphenylphosphino)propane) possessing two methyl substituents at the methylene carbon of dppm, affords the mononuclear complex PtCl₂(2,2-dppp), which undergoes anion

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ligand exchange upon treatment with NaCN in ethanol resulting in the formation of $Pt(CN)_2(2,2-dppp)$ [8].

Similar complexes have been described with the tripodal ligand 1,1,1-triphos (1,1,1-tris(diphenylphosphinomethyl)ethane) to form Pt(CN)₂(1,1,1-triphos) wherein two of the three phosphorus atoms of the ligand are coordinated to platinum at low temperature and its fluxional behaviour involving the corresponding five-coordinate complex has been investigated [7,9].

We report our results on the systematic investigations of the coordination chemistry of $Pt(CN)_2(diphosphine)$ complexes as well as that of $[Pt(CN)(triphos)]^+$ cation.

2. Experimental

The PtCl₂(diphosphine)-type complexes were prepared according to the literature [10]. The solvents were dried by conventional methods [11], distilled and kept under argon.

The ¹³C and ³¹P NMR spectra were recorded in CDCl₃ with TMS (internal standard) and H_3PO_4 (85%) (external standard), respectively. The spectra were recorded on a Varian Unity 300 and a Varian Inova 400 spectrometers at 75.4 and 100.58 MHz (¹³C NMR measurements) and 121.42 and 161.9 MHz (³¹P NMR measurements), respectively.

2.1. The preparation of the [Pt(triphos)Cl]Cl complex

A solution of 0.534 g (1 mmol) triphos in 3 ml of benzene was added to the refluxing yellow solution of 0.472 g (1 mmol) $PtCl_2(PhCN)_2$. After a few minutes fine white precipitate was formed. The mixture was stirred for 1 h then cooled to room temperature (r.t.). The solid was filtered off, washed with benzene, and dried under reduced pressure to give [Pt(triphos)Cl]Cl as white powder.

Anal. Calc. for $C_{34}H_{33}P_3Cl_2Pt$ (M = 800.54): C, 51.01; H, 4.15. Found: C, 51.18; H, 4.33%. Yield: 91%. ³¹P NMR (CDCl₃): 86.3 ppm (¹*J*(Pt,P) = 3035 Hz; ²*J*(P,P) = 2.6 Hz); 42.3 ppm (¹*J*(Pt,P) = 2477 Hz).

2.2. The preparation of a *Pt*(*CN*)₂(*diphosphine*) complex

The PtCl₂(diphosphine) complex (0.5 mmol) was added to the solution of potassium cyanide (1 mmol) dissolved in 15 ml methanol. The powder did not dissolve but was kept in suspension at 50°C with constant stirring for 10 h. The white powder-like crystals were filtered, washed with water, methanol and ether then dried. The recrystallization was carried out in benzene–methanol mixture.

2.3. Analytical data of the Pt(CN)₂(diphosphine) (1a-5a) and [Pt(triphos)(CN)]Cl (8a) complexes (for ³¹P NMR data see Table 1)

1a: ¹H NMR (δ(ppm), CDCl₃): 7.6 (m, 8H); 7.4 (m, 12H); 2.75 (m, 4H); ¹³C NMR (δ (ppm), CDCl₃): 136.7; 136.2; 131.5; 131.4; 129.8; 128.7; 128.6; 128.5; 28.4; IR (KBr, cm⁻¹): 2121 (ν(CN)). *Anal.* Calc. for $C_{28}H_{24}N_2P_2Pt$ (M = 645.54): C, 52.10; H, 3.75; N, 4.34. Found: C, 52.45; H, 4.09; N, 4.17%. Yield: 78%.

2a: ¹H NMR (δ (ppm), CDCl₃): 7.6 (m, 8H); 7.4 (m, 12H); 2.67 (m, 4H); 1.85 (m, 2H); ¹³C NMR (δ (ppm), CDCl₃): 133.2; 133.1; 131.8; 129.0; 28.5; 23.9; IR (KBr, cm⁻¹): 2189 (ν (CN)). *Anal.* Calc. for C₂₉H₂₆N₂P₂Pt (M = 659.57): C, 52.81; H, 3.97; N, 4.25. Found: C, 52.97; H, 4.16; N, 4.14%. Yield: 80%.

3a: ¹H NMR (δ (ppm), CDCl₃): 7.62 (m, 8H); 7.4 (m, 12H); 2.64 (m, 4H); 1.65 (m, 4H); ¹³C NMR (δ (ppm), CDCl₃): 133.0; 132.9; 131.7; 131.4; 131.1; 129.5; 129.4; 129.3; 28.4; 23.7; IR (KBr, cm⁻¹): 2205 (ν (CN)). *Anal.* Calc. for C₃₀H₂₈N₂P₂Pt (M = 673.59): C, 53.49; H, 4.19; N, 4.16. Found: C, 53.67; H, 4.23; N, 4.06%. Yield: 77%.

4a: ¹H NMR (δ (ppm), CDCl₃): 8.1 (m, 2H); 7.83 (m, 2H); 7.75 (m, 2H); 7.61 (m, 2H); 7.46 (m, 2H); 7.38 (m, 2H); 7.28 (m, 2H); 7.1 (m, 4H); 6.74 (m, 2H); 4.5 (m, 1H); 4.25 (m, 1H); 4.16 (m, 1H); 4.03 (m, 1H); 3.83 (m, 1H); 3.46 (m, 1H); 3.42 (m, 1H); 2.95 (m, 1H); 2.50 (m, 1H); 2.35 (m, 1H); 2.15 (m, 1H); 1.56 (m, 1H); IR (KBr, cm⁻¹): 2231 (ν (¹³CN). *Anal.* Calc. for C₃₉H₃₂N₂P₂FePt (M = 841.57): C, 55.66; H, 3.83; N, 3.33. Found: C, 55.81; H, 3.93; N, 3.20%. Yield: 84%.

5a: ¹H NMR (δ (ppm), CDCl₃): 8.0 (m, 2H); 7.6 (m, 2H); 7.3 (m, 6H); 4.41 (m, 3H); 4.02 (m, 2H); 3.9 (m, 1H); 3.58 (m, 1H); ¹³C NMR (δ (ppm), CDCl₃): 135.4; 135.2; 133.7; 133.5; 132.3; 131.8; 131.7; 131.4; 131.0; 130.7; 128.3; 128.2; 128.0; 127.0; 97.9; 97.7; 86.0; 72.6; 72.2; 71.4; 69.8; 69.4; 41.4; 38.9; 30.8; 29.7; 27.6; 27.1; 26.2; 25.7; 24.0; IR (KBr, cm⁻¹): 2095 (ν (¹³CN). *Anal*. Calc. for C₃₉H₄₄N₂P₂FePt (M = 853.67): C, 54.87; H, 5.20; N, 3.28. Found: C, 55.07; H, 5.33; N, 3.05%. Yield: 83%. **8a**: ¹H NMR (δ (ppm), CDCl₃): 8.2 (dd; 7.2 Hz, 12 Hz;

Sa: 'H NMR (δ (ppm), CDCl₃): 8.2 (dd; 7.2 Hz, 12 Hz; 2H); 7.8 (m, 9H); 7.5 (m, 14H); 3.9 (m, 2H); 3.2-3.4 (m, 2H); 2.65 (m; 2H); 2.2 (m; 2H); ¹³C NMR (δ (ppm), CDCl₃): 139.3; 138.9; 137.6; 133.4; 132.8; 132.8; 129.2; 129.3; 128.6; 128.4; 24.5; 23.7; IR (KBr, cm⁻¹): 2098 (ν (¹³CN). *Anal*. Calc. for C₃₅H₃₃NP₃ClPt (M = 791.11): C, 53.14; H, 4.20; N, 1.77. Found: C, 53.02; H, 4.08; N, 1.95%. Yield: 67%.

3. Results and discussion

3.1. Synthesis of Pt(CN)₂(diphosphine) complexes

For the preparation of platinum-cyano complexes various homo- and heterobidentate diphosphines



Fig. 1. The ferrocenyl diphosphines, 6 and 7.

(dppe = 1,2-bis(diphenylphosphino)ethane, dppp = 1,3bis(diphenylphosphino)propane, dppb = 1,4-bis(diphenylphosphino)butane, 6 = 1-diphenylphosphino-2,1'-[(1diphenylphosphino)-1,3-propanediyl]-ferrocene, 7 =1-diphenylphosphino-2,1'-[(1-dicyclohexylphosphino)-1,3-propanediyl]-ferrocene (Fig. 1)) were used which are able to form five-, six- or seven-membered chelate rings. While the Ph₂P(CH₂)_nPPh₂-type ligands are considered as diphosphines forming flexible chelate rings, the two ferrocenyl ligands possess rigid structure, and therefore rigid platinum-diphosphine chelate.

The $PtCl_2(diphosphine)$ complexes (1-5) were reacted with potassium cyanide in methanol (See Section 2). The corresponding $Pt(CN)_2(diphosphine)$ complexes (1a-5a) were separated as pure powder-like substances in all cases.

Table 1 NMR data of Pt(CN)₂(diphosphine) and [Pt(triphos)(CN)]⁺ complexes ^a

$PtCl_2(P-P)$	+ 2 K C N	\rightarrow	$Pt(CN)_2(P-P)$	+ 2KCl
1			1a	P - P = dppe
2			2a	$\mathbf{P} - \mathbf{P} = \mathrm{d}\mathbf{p}\mathbf{p}\mathbf{p}$
3			3a	$\mathbf{P} - \mathbf{P} = \mathrm{d}\mathbf{p}\mathbf{p}\mathbf{b}$
4			4 a	P - P = 6
5			5a	P - P = 7
				(1)

In case of homobidentate ligands (dppe, dppp, dppb) the 'mixed anionic ligand' complexes, PtCl(CN)-(diphosphine) (e.g. 2b) were formed up to 5% yield, when potassium cyanide was added at a ratio of Pt/ KCN = 1/1 to the corresponding dichloro complex in CDCl₃. No further side-products were detected during the 'in situ' NMR measurements, so even in these cases the formation of 1a-3a were dominating while approximately half of the starting dichloro complexes (1-3)remained unchanged. However, a significant effect on the formation of PtCl(CN)(diphosphine)-type complexes was observed when 6 (two PPh₂ groups in chemically different positions) and 7 (PPh₂ and PCy₂ groups) in 4a and 5a were used. The formation of the Pt-Cl(CN)(diphosphine) complex was facilitated by the larger difference in donor properties of the two phosphorus donors of the diphosphine ligand. The corre-

Complexes		$\delta(^{31}\text{P})$ (ppm)	¹ <i>J</i> (³¹ P, ¹⁹⁵ Pt) (Hz)	${}^{2}J({}^{31}\mathrm{P},{}^{31}\mathrm{P})$ (Hz)	$\delta(^{13}C)^{b}$ (ppm)	¹ <i>J</i> (¹³ C, ¹⁹⁵ Pt) (Hz)	² <i>J</i> (¹³ C, ³¹ P) (Hz)
PtCl ₂ (dppe)	1	43.0	3634				
Pt(CN) ₂ (dppe)	1a	41.2	2462				c
PtCl ₂ (dppp)	2	-4.9	3408				
Pt(CN) ₂ (dppp)	2a	-9.8	2368		(122.9)	(1047)	c
PtCl(CN)(dppp)	2b	-5.7 -8.8	d d	26.7			
PtCl ₂ (dppb)	3	10.7	3538				
Pt(CN) ₂ (dppb)	3a	9.0	2263				
$PtCl_2(6)$	4	-2.0	3375	19.5			
		34.4	3715				
Pt(CN) ₂ (6)	4a	-3.0	2321	25.5	n.d.	n.d.	(25.6; 121.2)
		29.5	2562				(26.0; 123.1)
PtCl(CN)(6)	4b	-0.4	2100 ^d	23.0			
		30.8	3560 ^d				
$PtCl_2(7)$	5	-0.4	3570	16			
		47.0	3527				
$Pt(CN)_2(7)$	5a	-2.3	2408	23.5	(125.1)	(998)	(12.9; 114.0)
		39.0	2435		(121.2)	(1059)	(14.1; 121.8)
PtCl(CN)(7)	5b	1.0	2452	19.8			
		45.9	3334				
[Pt(CN)(triphos)]+	8a	41.0 °	2383	6.0	n.d.	n.d.	(11.9)
		90.8	2205				(102.4)

^a Solvent: CDCl₃; ³¹P NMR and ¹³C NMR spectra were recorded at 121.4 MHz (161.9 MHz) and 75.4 MHz (100.6 MHz), respectively.

^b Chemical shift of the CN carbon (the data in parenthesis are obtained with ¹³C enriched ¹³CN complexes).

^d Due to the Pt-satellites of very low intensity, the ${}^{1}J(Pt,P)$ coupling constants could not be determined exactly.

^e The doublet at higher field (41.0 ppm) corresponds to two equivalent phosphorus.

^c The ${}^{2}J({}^{13}C, {}^{31}P)$ coupling constants are obtained from the AA'XX' second-order ${}^{31}P$ NMR spectra of the Pt(${}^{13}CN)_{2}(P-P)$ complex.

Table 2 Coupling constants of AA'XX' second order system

Complex	$^{2}J(\mathbf{P}_{\mathbf{A}},\mathbf{P}_{\mathbf{A}'})$ (Hz)	$^{2}J(\mathbf{C}_{\mathbf{X}},\mathbf{C}_{\mathbf{X}'})$ (Hz)	$^{2}J(\mathbf{P}_{A},\mathbf{C}_{X})$ (Hz)	${}^{2}J(P_{A},C_{X'})$ (Hz)
$\frac{Pt(^{13}CN)_2(dppe)}{Pt(^{13}CN)_2(dppp)}$	9.6	6.7	123.8	-12.9
	32.7	2.0	121.9	-14.3

sponding chloro-cyano complexes were obtained in 14 and 30% yields by using ligands 6 and 7, respectively.

However, even the application of the slight excess (up to 25%) of potassium cyanide (Pt/KCN = 1/2.5) results in the partial substitution of the diphosphine ligand. The appearance of the signal of the free ligand was observed by 'in situ' ³¹P NMR measurements (Eq. (2)).

Surprisingly, the papers published in this topic did not mention this consecutive reaction and potassium cyanide is used in slight excess in all cases. As a consequence of that, a part of the platinum must have been 'lost' as potassium-tetracyano-platinate(II).

$$Pt(CN)_{2}(P-P) + 2KCN \rightarrow K_{2}[Pt(CN)_{4}] + P - P \qquad (2)$$

1a - 5a

3.2. NMR characterisation of Pt(CN)₂(diphosphine) complexes

The most remarkable feature of the ³¹P NMR of the above complexes is the surprisingly small ${}^{1}J({}^{195}\text{Pt},{}^{31}\text{P})$ coupling constant (Table 1). With the exception of ${}^{1}J({}^{195}\text{Pt},{}^{31}\text{P})$ for one of the phosphorus of Pt(CN)₂(6), all values fall below 2500 Hz.

The direct Pt–CN bond has been proved by ³¹P and ¹³C NMR investigation of the corresponding ¹³C labelled complexes. The $Pt(^{13}CN)_2(ligand)$ -type complexes of both homobidentate and heterobidentate ligands were prepared which show the presence of two equivalent and non-equivalent cyano ligands, respectively. It was proved unequivocally by ³¹P and ¹³C NMR (Tables 1 and 2). (Instead of potassium cyanide the labelled reagent sodium cyanide-¹³C (enrichment in ¹³C was higher than 99.5%) was used in all cases and has shown similar reactivity.)

As a typical example, the ³¹P and ¹³C NMR spectra of $Pt({}^{13}CN)_2$ (7) are shown in Figs. 2 and 3, respectively. The ³¹P NMR shows an eight-line multiplet for both non-equivalent phosphorus flanked by platinum satellites. This pattern is arising from the coupling of the phosphorus with the carbon of the cyano ligands both in *cis* and *trans* positions as well as with the neighbouring phosphorus atom in *cis* position. A clear ¹³C NMR spectrum of similar structure was obtained, as expected (Fig. 3).

Both Pt(${}^{13}CN$)₂(dppp) and Pt(${}^{13}CN$)₂(dppe) complexes show typical AA'XX' second order ${}^{31}P$ NMR spectra. The calculated values of ${}^{2}J(P_{A}, P_{A'})$, ${}^{2}J(C_{X}, C_{X'})$, ${}^{2}J(P_{A}, P_{X})$, and ${}^{2}J(P_{A}, P_{X'})$ are in Table 2.



Fig. 2. ${}^{31}P$ NMR spectrum of Pt(${}^{13}CN$)₂(7). (The upper spectrum is the enlargement of the upfield region.)

3.3. Synthesis and characterization of [Pt(triphos)(CN)]⁺ complex cation

The [Pt(triphos)Cl]Cl (8) ionic complex (where triphos (9) stands for bis(2-diphenylphosphinoethyl)-phenylphosphine) has been synthesized by the usual 'benzonitrile method' (See Section 2). Unlike the '1,1,1-triphos' (1,1,1-tris(diphenylphosphinomethyl)-ethane), the 'linear triphos' forms a well-defined square-planar ionic complex without a dangling arm, observed before with palladium [12]. [Pt(triphos)(CN)]⁺ (8a) complex cation was obtained selectively in a facile reaction by the addition of one equivalent of KCN only. Both in 8 and 8a all the three phosphorus atoms of the ligand are coordinated to platinum (Table 1).

Upon addition of a further equivalent of potassium cyanide to **8a** in CDCl₃ a rather complex mixture was obtained. In addition to the signals of **8a** a sharp doublet of quartet (*dq*)-type pattern appeared at 47.6 ppm (${}^{1}J(\text{Pt,P}) = 2549$ Hz) and a signal of exactly double intensity (based on the integration) was situated at



Fig. 3. ¹³C NMR spectrum of Pt(¹³CN)₂(7). The 1/4/1 patterns (central line and platinum satellites) for the carbons of the two non-equivalent cyano ligands are indicated by \bigcirc and \times . (The rest of the peaks are due to the aromatic carbons of the ligand 7.)



Fig. 4. The proposed structure of the Pt(triphos)(CN)₂ complex.

14.5 ppm (${}^{1}J(\text{Pt},\text{P}) \approx 1350 \text{ Hz}$) as an extremely broad 1/4/1 pattern.

This phenomenon can be explained by the presence of a square pyramidal covalent Pt(¹³CN)₂(triphos) complex (Fig. 4). The dq pattern can be assigned to the most basic 'middle' phosphorus (P^2) of the ligand bound strongly to the platinum in equatorial position (with a cyano ligand trans to it). It couples with the cyano carbons $({}^{2}J_{cis}({}^{31}P, {}^{13}C) = 14$ two Hz: ${}^{2}J_{trans}({}^{31}P, {}^{13}C) = 121$ Hz) and with the other two phosphorus $({}^{2}J(\mathbf{P},\mathbf{P}) = ca.$ 14 Hz). The two ${}^{2}J(\mathbf{P},\mathbf{P})$ coupling constants could be equal in case of the fast interchange of diphenylphosphino moieties of the equatorial and apical positions (P^1 and P^3). The interchange could be fast in the NMR timescale. It may result in the extremely broad 'upfield' signal at 14.5 ppm which also refers to the fluxional behaviour of the five-coordinate complex. Although the low-temperature measurements show the further broadening

of this signal at 243 K indicating a situation near to coalescence, no separation of the lines could be achieved and some complex precipitated due to low solubility at this temperature. It is worth noting, that similar behaviour has been obtained in CD_2Cl_2 ; no line separation has been reached even at 223 K.

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