

## Palladium(II) and Platinum(II) Complexes with Mixed Ligands of Tertiary Monophosphines and 5-Phenyl-1,3,4-oxadiazole-2-thione or 4,5-Diphenyl-1,2,4-triazole-3-thione

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Several new palladium(II) and platinum(II) complexes containing two types of ligands: tertiary monophosphines (L) and 5-phenyl-1,3,4-oxadiazole-2-thione ion (A) or 4,5-diphenyl-1,2,4-triazole-3-thione ion (B) have been prepared. The so obtained complexes *trans*-[PdA<sub>2</sub>L<sub>2</sub>], *trans*-[PdB<sub>2</sub>L<sub>2</sub>] and *cis*-[PtA<sub>2</sub>L<sub>2</sub>] were characterized by elemental analysis, IR, UV-Vis, <sup>31</sup>P NMR spectroscopy, molar conductance and magnetic susceptibility measurements.

**Key Words:** Platinum(II), Palladium(II), Phosphine, Oxadiazole, Ligands, Complexes

### INTRODUCTION

The interaction of thione ligands, which contain the chemically active groups [—N—HC(=S)— ↔ —N=C(SH)—], with different metals have attracted much attention. These ligands can bind to a metal in several ways<sup>1–4</sup>. The interactions of platinum and gold with N,S-donor atoms have been recognized for their antineoplastic properties with the potential to develop metal based drugs<sup>1–6</sup>. Recently, some papers involving platinum(II) or platinum(IV), gold(II) and ruthenium(II) with tertiary phosphines as co-ligands have been published<sup>7–10</sup>. We have recently reported the synthesis and characterization of some transition metal complexes of tertiary diphosphines and 5-phenyl-1,3,4-oxadiazole-2-thione<sup>11</sup>. In continuation of our findings, *e.g.*, *trans*-palladium complexes<sup>12, 13</sup> and other related complexes<sup>14</sup> we attempted to synthesize and characterize palladium(II) and platinum(II) complexes containing mixed ligands, tertiary monophosphines and 5-phenyl-1,3,4-oxadiazole-2-thione or 4,5-diphenyl-1,2,4-triazole-3-thione. These complexes have the general formulae *trans*-[PdA<sub>2</sub>L<sub>2</sub>], *trans*-[PdB<sub>2</sub>L<sub>2</sub>] and *cis*-[PtA<sub>2</sub>L<sub>2</sub>], where L = tert. phosphine, A and B were the above thione-H. To the best of our knowledge this work has not been previously reported.

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## EXPERIMENTAL

$^{31}\text{P}$  ( $^1\text{H}$ ) NMR spectra were performed at the laboratories of Prof. Wilhelm Keim, Institut für Technische Chemie der RWTH Aachen, Worringer Weg 1, D-52074 Aachen, Germany. IR spectra were recorded on a Pye-Unicam SP3-300s spectrophotometer in the  $4000\text{--}200\text{ cm}^{-1}$  range using CsI discs. Electronic spectra were obtained using a Perkin-Elmer Lambda-9 spectrophotometer. Elemental analyses were carried out on Carlo-Erba CHN analyzer, type 1106. Magnetic measurements were recorded on a Bruker BM6 instrument at room temperature following the Faraday method. Conductivity measurements were made on a conductivity meter type CDM 83 70. Melting points were recorded on an Electrothermal 9300 melting point apparatus and were uncorrected.

**Materials:**  $\text{K}_2\text{PtCl}_4$ ,  $\text{Na}_2\text{PdCl}_4\cdot 3\text{H}_2\text{O}$ ,  $\text{PPh}_3$ ,  $\text{PMePh}_2$  and  $\text{PET}_3$  were commercial products and were used as supplied. The ligands 5-phenyl-1,3,4-oxadiazole-2-thione (AH), 4,5-diphenyl-1,2,4-triazole-3-thione (BH), their potassium salts (AK) and (BK), the complexes *trans*- $[\text{PdA}_2]$  (1) and *cis*- $[\text{PtA}_2]$  (2) were prepared according to literature methods<sup>15, 16</sup>. Complex (1) was also prepared by another route (*vide infra*)

## Preparation of complexes

***Trans*- $[\text{PdA}_2]$  (1):** Solid *trans*- $[\text{PdCl}_2(\text{AH})_2]\cdot 7\text{Me}_2\text{CO}$  complex (4) (0.27 g, 1.5 mmol) was suspended in MeOH ( $100\text{ cm}^3$ ). The mixture was stirred at room temperature for 1 h, then refluxed for 2 h. The orange insoluble solid was filtered off, washed with  $\text{Et}_2\text{O}$  and dried (yield 60%). The complex was obtained as solvated with one mole of  $\text{H}_2\text{O}$ .

***Trans*- $[\text{PdB}_2]$  (3):** A mixture of BH (0.37 g, 1.4 mmol) and  $\text{Na}_2\text{PdCl}_4\cdot 3\text{H}_2\text{O}$  (0.25 g, 0.73 mmol) in MeOH ( $15\text{ cm}^3$ ) was stirred overnight. The orange solid thus formed was filtered off, washed with cold MeOH then  $\text{Et}_2\text{O}$  and dried under vacuum (yield 97%).

***Trans*- $[\text{PdCl}_2(\text{AH})_2]$  (4):** Solid  $[\text{Na}_2\text{PdCl}_4]\cdot 3\text{H}_2\text{O}$  (0.097 g, 0.28 mmol) was added to a solution of AH (0.1 g, 0.56 mmol) in  $\text{Me}_2\text{CO}$  ( $6\text{ cm}^3$ ). When the mixture was stirred overnight, an orange solid was formed. This solid was filtered off, washed with  $\text{H}_2\text{O}$ , then MeOH and dried under vacuum (yield 42%). This complex was obtained as solvated with one mole of  $\text{Me}_2\text{CO}$ .

***Trans*- $[\text{PdA}_2(\text{PMePh}_2)_2]$  (5):**  $\text{PMePh}_2$  (0.26 g, 1.3 mmol) was added to a solution of *trans*- $[\text{PdA}_2]$  (0.3 g, 0.65 mmol) in  $\text{CHCl}_3$  ( $20\text{ cm}^3$ ). When the mixture was stirred overnight at room temperature its colour changed from orange to orange-red. 50% of the solvent was removed under reduced pressure and  $\text{Et}_2\text{O}$  was added to give a yellow solid, which was filtered off, washed with EtOH, then  $\text{Et}_2\text{O}$  and dried under vacuum (yield 92%).

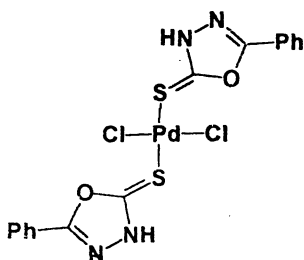
In a similar manner, the complexes *trans*- $[\text{PdA}_2\text{L}_2]$ , [L =  $\text{PET}_3$  (6),  $\text{PPh}_3$  (7)] and *trans*- $[\text{PdB}_2\text{L}_2]$ , [L =  $\text{PMePh}_2$  (8),  $\text{PET}_3$  (9),  $\text{PPh}_3$  (10)] were made as solid products.

***Cis*- $[\text{PtA}_2(\text{PMePh}_2)_2]$  (11):**  $\text{PMePh}_2$  (0.15 g, 0.7 mmol) was added to a solution of  $[\text{PtA}_2]$  in  $\text{CHCl}_3$  ( $20\text{ cm}^3$ ). The mixture was stirred at room temperature overnight. The solvent then evaporated and  $\text{Et}_2\text{O}$  was added to give a white solid, which was filtered off, dried and recrystallized from  $\text{CH}_2\text{Cl}_2$ .

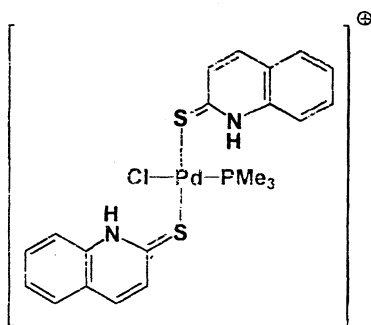
Similarly the complexes *cis*-[PtA<sub>2</sub>L<sub>2</sub>] [L = PEt<sub>3</sub> (12), PPh<sub>3</sub> (13)] were made as solid products.

## RESULTS AND DISCUSSION

In our previous work<sup>11</sup>, we reported that reaction of Na<sub>2</sub>PdCl<sub>4</sub>·3H<sub>2</sub>O with potassium salt of 5-phenyl-1,3,4-oxadiazole-2-thionate (AK) in MeOH gave *trans*-[PdA<sub>2</sub>]-H<sub>2</sub>O complex (1). We found that (1) can also be obtained directly by using the free thione; 5-phenyl-1,3,4-oxadiazole-2-thione (AH). A stirring mixture of Na<sub>2</sub>PdCl<sub>4</sub>·3H<sub>2</sub>O and the thione (AH) in Me<sub>2</sub>CO at room temperature gave a rather different product, identified as *trans*-[PdCl<sub>2</sub>(AH)<sub>2</sub>] (4). The IR spectrum of this complex showed a single strong intensive band at 340 cm<sup>-1</sup> which was assigned to  $\nu(\text{Pd}-\text{Cl})$  in a *trans*-arrangement<sup>15</sup>; a broad band at 3100 cm<sup>-1</sup> [assigned to  $\nu(\text{N}-\text{H})$ ] appeared in the same position of the free (AH) ligand<sup>16</sup>. This indicates that the NH group of the oxadiazole ligand is not taking part in the coordination. The  $\nu(\text{C}=\text{S})$  mode occurring at 768 cm<sup>-1</sup> in the free ligand<sup>16</sup> underwent a shift to a lower frequency and then appeared at 715 cm<sup>-1</sup> after complex formation. This shift indicates that sulphur of the (C=S) group is coordinated to palladium. From these and other data reported in the Tables, structure (I) was proposed for the complex *trans*-[PdCl<sub>2</sub>(AH)<sub>2</sub>] (4). This complex is similar, from sulphur-coordination point of view, to that reported recently<sup>17</sup>, i.e., structure (II).



(I)



(II)

However, refluxing of a suspension of *trans*-[PdCl<sub>2</sub>(AH)<sub>2</sub>]-Me<sub>2</sub>CO (4) in MeOH for a few hours gave *trans*-[PdA<sub>2</sub>]-H<sub>2</sub>O (1) as indicated from the analytical data. The IR spectrum of the product (1) showed that the previous  $\nu(\text{N}-\text{H})$  band appearing at 3100 cm<sup>-1</sup> disappeared indicating a loss of hydrogen from the thione AH. Similarly the strong IR band at 340 cm<sup>-1</sup> attributed to  $\nu(\text{Pd}-\text{Cl})$  also disappeared from complex (1); this is due to the loss of the chlorides from it. Other analytical data are in a good agreement with the formula *trans*-[PdA<sub>2</sub>] (Tables 1, 2 and 4).

TABLE- 1  
 PHYSICAL PROPERTIES OF COMPLEXES (1)–(13)

No.	complex	Colour	Yield (%)	m.p. (°C)	Found (Calcd.) (%)		
					C	H	N
(1)	<i>trans</i> -[PdA <sub>2</sub> ] <sup>a</sup>	orange	60	278–280	40.2 (40.1)	2.6 (2.5)	11.6 (11.7)
(2)	<i>cis</i> -[PtA <sub>2</sub> ]	yellow-brown	64	308–310	34.9 (35.0)	1.9 (1.8)	9.8 (10.2)
(3)	<i>trans</i> -[PdB <sub>2</sub> ]	orange	97	288–290	55.0 (55.1)	3.3 (3.3)	13.7 (13.8)
(4)	<i>trans</i> -[PdCl <sub>2</sub> (AH) <sub>2</sub> ] <sup>b</sup>	orange	42	347–350	44.3 (44.2)	2.7 (2.4)	8.6 (8.5)
(5)	<i>trans</i> -[PdA <sup>2</sup> (PMePh <sub>2</sub> ) <sub>2</sub> ]	yellow	92	249–252	58.2 (58.5)	4.3 (4.3)	6.5 (6.5)
(6)	<i>trans</i> -[PdA <sub>2</sub> (PEt <sub>3</sub> ) <sub>2</sub> ]	yellow	82	138–142	48.0 (48.2)	5.8 (5.8)	8.1 (8.1)
(7)	<i>trans</i> -[PdA <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> ]	yellow	98	214–217	63.4 (63.3)	4.1 (4.1)	6.0 (5.7)
(8)	<i>trans</i> -[PdB <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ]	yellow	97	208–210	64.2 (64.2)	4.6 (4.6)	8.0 (8.3)
(9)	<i>trans</i> -[PdB <sub>2</sub> (PEt <sub>3</sub> ) <sub>2</sub> ]	yellow	67	205–208	56.7 (56.8)	6.0 (6.0)	9.8 (9.9)
(10)	<i>trans</i> -[PdB <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> ]	orange	54	190–195	65.7 (65.6)	4.2 (4.3)	8.8 (8.8)
(11)	<i>cis</i> -[PtA <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ]	white	97	262–264	52.9 (53.0)	3.9 (3.8)	5.9 (6.0)
(12)	<i>cis</i> -[PtA <sub>2</sub> (PEt <sub>3</sub> ) <sub>2</sub> ]	white	70	174–176	42.5 (42.8)	5.2 (5.1)	7.1 (7.2)
(13)	<i>cis</i> -[PtA <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> ]	off-white	81	286–287	57.9 (58.1)	3.8 (3.8)	5.2 (5.3)

<sup>a</sup> Complex solvated with one mole of H<sub>2</sub>O<sup>b</sup> Complex solvated with one mole of Me<sub>2</sub>CO

Although the thiones AH and BH possess multi-donor centres, their anions A<sup>-</sup> and B<sup>-</sup>, however, acted in complexes (1)–(3) as bidentate ligands bonded to the metal *via* S and N of NH. From the soft-hard concepts, we believe that the thione ion bonded to the metal as (:S=C—N—) ↔ (—S—C=N:), *i.e.*,  $\pi$ -allyl like system.

Treatment of the chelated complexes *trans*-[PdA<sub>2</sub>] (1), *trans*-[PdB<sub>2</sub>] (3) or *cis*-[PtA<sub>2</sub>] (2) with two moles of the monophosphine (L) resulted in replacing the coordinated site of the thione to give complexes of the type *trans*-[PdA<sub>2</sub>L<sub>2</sub>], *trans*-[PdB<sub>2</sub>L<sub>2</sub>] or *cis*-[PtA<sub>2</sub>L<sub>2</sub>], respectively, *i.e.*, complexes (5)–(13) (Table-1),

in which the thiones A or B acted as monodentate ligands. Hence, one can expect the possibility of the formation of linkage isomers. IR spectroscopy is an effective tool for the elucidation of such isomers. However  $^{31}\text{P}$  ( $^1\text{H}$ ) NMR is more

TABLE-2  
IR SPECTRAL DATA AND ASSIGNMENTS OF COMPLEXES (1)–(13)

Complex	Thiomide bands				$\nu(\text{P—M})$	Other bands
	I	II	III	IV		
(1)	1590 vw	1310 vw, 1380 sh	922 w, 965 vw	770 s		3500 b, $\nu(\text{O—H})$
(2)	1600 m	1305 w, 1360 s	922 w, 965 w	770s		3060 w, $\nu(\text{C—H})$ aromatic
(3)	1590 m	1400 s	985 m	765 s		
(4)	1540 w, 1600 w	1440 s	940 s	770 s		340 s, $\nu(\text{Pd—Cl})$ 3050 m, $\nu(\text{C—H})$ aromatic 3100 s, $\nu(\text{N—H})$
(5)	1550 w	1440 vs	960 w	775 m, 750 s	415 w	2990 w, $\nu(\text{C—H})$ aliphatic 3040 w, $\nu(\text{C—H})$ aromatic
(6)	1545 m	1425 vs	965 w	775 m, 760 s	420 w	2960 w, $\nu(\text{C—H})$ aliphatic 3060 w, $\nu(\text{C—H})$ aromatic
(7)	1545 w	1425 vs	955 w	770 s, 750 s	430 w	3045 w, $\nu(\text{C—H})$ aromatic
(8)	1595 m	1430 s	970 m	770 s	430 w	3050 w, $\nu(\text{C—H})$ aromatic
(9)	1595 m	1435 m	970 m	775 m, 765 s	420 w	3060 w, $\nu(\text{C—H})$ aromatic
(10)	1590 m	1430 s	970 m	780 m, 760 s	430 w	3060 w, $\nu(\text{C—H})$ aromatic
(11)	1550 w	1430 vs	960 w	775 m, 750 s	430w	3050 w, $\nu(\text{C—H})$ aromatic
(12)	1540 w	1430 vs	960 w	780 m, 755 s	425 w	3060 w, $\nu(\text{C—H})$ aromatic
(13)	1545w	1430 vs	960 w	770 s, 750 s	425w	3050 w, $\nu(\text{C—H})$ aromatic

convenient, hence the spectra of some of the prepared complexes were recorded (Table-3). The  $^{31}\text{P}$  ( $^1\text{H}$ ) NMR spectrum of *e.g.*, *cis*-[PtA<sub>2</sub>(PMePh<sub>2</sub>)<sub>2</sub>] (11) showed a singlet at  $\delta$  9.6 ppm associated with platinum satellites,  $J(\text{Pt—P})$ , 2567 Hz. By comparison of  $\delta$  P of this complex with  $\delta$  of the *cis*-[PtCl<sub>2</sub>(PMePh<sub>2</sub>)<sub>2</sub>] ( $\delta$ , -1.2 ppm) and that of the *trans*-isomer ( $\delta$ , 41.3 ppm) we believe that the product is a *cis*-isomer. The low  $J(\text{Pt—P})$  value (2567 Hz) indicates that the complex is a

*cis*-isomer with the phosphorus in *trans*- relationship to sulphur bonded oxadiazole<sup>18</sup>.

TABLE- 3  
<sup>31</sup>P (<sup>1</sup>H) NMR DATA OF SOME SELECTED PHOSPHINE COMPLEXES (5), (7)–(11)

No.	omplex	δP (ppm)	J(Pt—P) (Hz)	Proportions (%)
	<i>cis</i> -[PdCl <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ] <sup>a</sup>	-19.1		
	<i>trans</i> -[PdCl <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ] <sup>a</sup>	-7.8		
(5)	<i>trans</i> -[PdA <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ] <sup>b</sup>	13.3		80, S-bonded
		12.2		20, N-bonded
(7)	<i>trans</i> -[PdA <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> ] <sup>b</sup>	29.4		75, S-bonded
		27.3		25, N-bonded
(8)	<i>trans</i> -[PdB <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ]	13.1		
(9)	<i>trans</i> -[PdB <sub>2</sub> (PEt <sub>3</sub> ) <sub>2</sub> ]	18.9		
(10)	<i>trans</i> -[PdB <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> ]	24.4		
	<i>cis</i> -[PtCl <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ] <sup>a</sup>	-1.2	3616	
	<i>trans</i> -[PtCl <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ] <sup>a</sup>	41.3	3028	
(11)	<i>cis</i> -[PtA <sub>2</sub> (PMePh <sub>2</sub> ) <sub>2</sub> ]	9.6	2567	

<sup>a</sup> Complexes included for comparison.

<sup>b</sup> Complexes appeared in solution in two isomeric forms, the S-bonded and the N-bonded.

In contrast to the <sup>31</sup>P (<sup>1</sup>H) NMR spectrum of (11) which showed one singlet, the <sup>31</sup>P (<sup>1</sup>H) NMR of the palladium analogue *trans*-[PdA<sub>2</sub>(PMePh<sub>2</sub>)<sub>2</sub>] (5) showed two singlets at δ, 13.3 and 12.2 ppm in the ratio of 4 : 1 and no coupling between the singlets was observed, which suggested the presence of two isomers. By comparison with δ of the *cis*- and *trans*-[PdCl<sub>2</sub>(PMePh<sub>2</sub>)<sub>2</sub>] [δ, -19.1 and -7.8 ppm, respectively) we believe that the product is the *trans*- isomer consisting of two linkage isomers. The proportion of the S-bonded isomer is 80 and of the N-bonded is 20 per cent. Similarly, the spectrum of the complex *trans*-[PdA<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] (7) showed the two isomers too in almost similar proportions (75 and 25%, respectively). However, the <sup>31</sup>P (<sup>1</sup>H) NMR spectrum of the corresponding triazole complexes *trans*-[PdB<sub>2</sub>L<sub>2</sub>] [L = PMePh<sub>2</sub> (8), PEt<sub>3</sub> (9) or PPh<sub>3</sub> (10)] showed them to be a single isomer each.

Conductivity measurements indicate that all the prepared complexes are non-electrolytes<sup>19</sup>, while magnetic susceptibility measurements of the prepared complexes showed them to be diamagnetic consistent with four coordinate square-planar *d*<sup>8</sup> complexes. The electronic spectral data of the complexes were in good agreement with the suggested geometrics for the four-coordinate metal centres being square-planar<sup>20</sup>.

TABLE- 4  
UV-Vis DATA OF COMPLEXES (1)–(13)

Complex	Electronic spectra $\lambda_{\max}$ (nm)	Assignments <sup>b</sup>
(1)	440, $^1A_{1g}$	$\longrightarrow ^1B_1$ ,
	354	$^1A_{1g} \longrightarrow ^1E_g$
(2)	287	c.t.
(3)	355	$^1A_{1g} \longrightarrow ^1E_g$
(4)	a	
(5)	324, c.t.,	
	375	$^1A_{1g} \longrightarrow ^1B_{1g}$
(6)	368	$^1A_{1g} \longrightarrow ^1B_{1g}$
(7)	368, $^1A_{1g}$	$\longrightarrow ^1B_{1g}$
	470	$^1A_{1g} \longrightarrow ^1A_{2g}$
(8)	286, c.t.,,	
	370	$^1A_{1g} \longrightarrow ^1B_{1g}$
(9)	306, c.t.,	
	386	$^1A_{1g} \longrightarrow ^1B_{1g}$
(10)	344, $^1A_{1g}$	$\longrightarrow ^1E_g$
	392	$^1A_{1g} \longrightarrow ^1B_{1g}$
(11)	324, c.t.,	
	374	$^1A_{1g} \longrightarrow ^1B_{1g}$
(12)	316	c.t.
(13)	a	

a: Insoluble; b: c.t. charge transfer absorption band.

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