Journal of Organometallic Chemistry, 468 (1994) 279–283

Synthesis and characterization of organoplatinum(II) dithiolate complexes of the type [PtAr(S S)(PMePh$_2$)]

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(Received May 26, 1993)

Abstract

The reaction of [Pt$_2$Ar$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] with sodium, potassium or ammonium salts of the dithio acids (S S) affords mononuclear complexes of the type [PtAr(S S)(PMePh$_2$)] (where Ar = Ph, C$_6$H$_4$Me-4 (tol); S S = SSCOEt, SSCNR$_2$ (R = Me or Et), SSPR$_2$ (R = OEt, O^{n}Pr, O^{i}Pr, O^{i}Bu, O^{s}Bu, Ph)). The latter complexes were characterized by elemental analysis and nuclear magnetic resonance (NMR) (^{1}H and ^{31}P) data. These new complexes are inert to excess triphenylphosphine at room temperature.

Key words: Platinum; Sodium; Potassium; Ammonium

1. Introduction

The synthesis and reaction chemistry of classical coordination complexes of platinum(II) dithiolate ligands S S (where S S = SSCR, SSCOR, SSCNR$_2$, SSPR$_2$) have attracted much attention during the last two decades [1–5]. The dithiolate ligand in these complexes acts in a bidentate chelating, monodentate or ionic fashion. Some of these complexes show dynamic behaviour on a nuclear magnetic resonance (NMR) time scale at room temperature. On the other hand, the dithiocarboxylate complexes are stacked molecules with an intermolecular distance short enough to produce unusual properties [5]. However, the chemistry of organoplatinum(II) species with these ligands has not received much attention [6], although complexes of trimethylplatinum(IV) with such ligands have been investigated recently [7]. In this paper, we describe the synthesis and characterization of organoplatinum(II) dithiolate complexes of the type [PtAr(S S)(PMePh$_2$)] (where Ar = Ph, C$_6$H$_4$Me-4 (tol); S S = SSCOEt, SSCNR$_2$ (R = Me or Et), SSPR$_2$ (R = OEt, O^{n}Pr, O^{i}Pr, O^{i}Bu, O^{s}Bu, Ph)).

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2. Results and discussion

Reactions of [Pt$_2$Ar$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] with two mole equivalents of the sodium, potassium or ammonium salt of a dithio acid afforded mononuclear complexes of the type [PtAr(S S)(PMePh$_2$)] (Ar = Ph, C$_6$H$_4$Me-4 (tol); S S = SSCOEt, SSCNR$_2$ (R = Me or Et), SSPR$_2$ (R = OEt, O^{n}Pr, O^{i}Pr, O^{i}Bu, O^{s}Bu, Ph)). These complexes are colourless to cream coloured crystalline solids, highly soluble in common organic solvents. All these complexes were characterized by elemental analysis and NMR data. The mass spectrum of [PtPh{SSP(OEt)$_2$}(PMePh$_2$)] displayed a molecular ion peak at m/e 657. Osmometric molecular weight determinations of two representative complexes [Pttol{SSP(OR')$_2$}(PMePh$_2$)] were as follows: R' = ^{i}Pr: found, 747; calc., 699.7; R' = Et: found, 612; calc., 671. They indicate that they are monomeric in nature.

The ^{31}P NMR spectra of xanthate and dithiocarbamate complexes exhibited a single resonance flanked

0022-328X/94/$7.00
SSDI 0022-328X(93)24069-H

by platinum satellites with ^1J(Pt–P) of the order of approximately 4000 Hz (Table 1). The spectra of phosphorus-based acid complexes showed a doublet due to $^3J(^{31}P-^{31}P)$ for the phosphine ligand and also for the acid moiety, each flanked by platinum satellites. The magnitude of the $^3J(^{31}P-^{31}P)$ coupling varies between 5 and 10 Hz; however, in some cases such coupling could not be resolved. The $^2J(^{195}Pt-^{31}P)$ value for dialkyldithiophosphate complexes (approximately 225 Hz) was greater than that of diphenyldithiophosphinate (174 Hz). Interestingly, the di(sec-butyl)dithiophosphate complexes exhibited three lines for the acid fragment. For the diastereomeric (*RR*, *SS* pair and *meso*) ammonium O,O′-di(sec-butyl)dithiophosphate two singlets in a ratio of 1:1 were observed [8]. On complexation with platinum separate signals for *RR* and *SS* isomers appeared. The resonance for the *meso* form occurred between these two signals and was approximately 45% abundant (by integration). However, there was chemical shift degeneracy for the phosphine resonance; accordingly only one singlet was observed for all three isomers.

As is evident from Table 1 the $^1J(^{195}Pt-^{31}P)$ value is influenced by the type of dithiolate ligand and the Ar group on platinum. The ^1J(Pt–P) value is slightly greater for tolyl complexes compared with phenyl derivatives. Furthermore, ^1J(Pt–P) increases in the following order of the dithiolate ligand which is the reverse of the ligating strength of S S [9]: $SSCNR_2 < SSCOEt < SSPR_2$.

The ^{1}H NMR spectra showed expected multiplicities and integration (Table 2). The PMe protons appeared as a doublet with ^2J(P–H) = 10.5 Hz. The doublet was further flanked by platinum-195 satellites with $^3J(^{195}Pt-^1H)$ of the order of approximately 50 Hz. As reported in other cases [6], the R groups on dialkyldithiocarbamate ligands are non-equivalent. Thus two separate sets of resonances were observed for NR_2 protons. The methyl groups of the O^{i}Pr moiety of $SSP(O^iPr)_2$ are anisochronous as two doublets were observed in the ^{1}H NMR spectrum of [PtAr{SSP-$(O^iPr)_2$}(PMePh$_2$)].

Unlike Pt(S S)$_2$ and [PtCl(S S)(PR$_3$)], which undergo a variety of reactions with phosphine ligands [1–5], the organoplatinum(II) complexes reported here are inert. When [PtPh{SSP(O^{i}Pr)$_2$}(PMePh$_2$)] was treated with one to three equivalents of triphenylphosphine, the ^{31}P NMR spectrum was unaffected at room

TABLE 1. $^{31}P\{^1H\}$ NMR data for [PtAr(SS)(PMePh$_2$)] complexes in $CDCl_3$ [a]

Complex	Phosphine ligand			Dithio acid ligand		
	δ (ppm)	$^1J(^{195}Pt-^{31}P)$ (Hz)	$^3J(^{31}P-^{31}P)$ (Hz)	δ (ppm)	$^2J(^{195}Pt-^{31}P)$ (Hz)	$^3J(^{31}P-^{31}P)$ (Hz)
[Pt$_2$Ph$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] [b]	−3.3		–	–	–	–
	−3.5	5003	–	–	–	–
[PtPh(SSCOEt)(PMePh$_2$)]	−2.1(s)	4086	–	–	–	–
[PtPh(SSCNMe$_2$)(PMePh$_2$)]	−2.2(s)	3958	–	–	–	–
[PtPh(SSCNEt$_2$)(PMePh$_2$)]	−2.3(s)	3928	–	–	–	–
[PtPh{SSP(OEt)$_2$}(PMePh$_2$)]	−2.1(d)	4333	5	96.8(d)	222	5
[PtPh{SSP(O^{n}Pr)$_2$}(PMePh$_2$)]	−2.1(b)	4330	–	96.8(b)	224	–
[PtPh{SSP(O^{i}Pr)$_2$}(PMePh$_2$)]	−1.8(d)	4300	5	92.4(d)	223	5
[PtPh{SSP(O^{i}Bu)$_2$}(PMePh$_2$)]	−2.1(b)	4331	–	96.4(b)	225	–
[PtPh{SSP(O^{s}Bu)$_2$}(PMePh$_2$)]	−1.9(b)	4293	–	92.8	226	–
				92.7	226	
				92.6	228	
[PtPh(SSPPh$_2$)(PMePh$_2$)]	−1.6(d)	4315	10	79.0(d)	174	10
[Pt$_2$tol$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] [b]	−1.1		–	–	–	–
	−1.2	5014	–	–	–	–
[Pttol(SSCOEt)(PMePh$_2$)]	0.1(s)	4101	–	–	–	–
[Pttol(SSCNMe$_2$)(PMePh$_2$)]	0.1(s)	3971	–	–	–	–
[Pttol(SSCNEt$_2$)(PMePh$_2$)]	0.04(s)	3939	–	–	–	–
[Pttol{SSP(OEt)$_2$}(PMePh$_2$)]	0.3(d)	4348	6	98.9(d)	224	6
[Pttol{SSP(O^{n}Pr)$_2$}(PMePh$_2$)]	0.2(d)	4344	5	99.0(d)	225	5
[Pttol{SSP(O^{i}Pr)$_2$}(PMePh$_2$)]	0.5(d)	4314	6	96.6(d)	223	6
[Pttol{SSP(O^{s}Bu)$_2$}(PMePh$_2$)] [c]	0.4(d)	4306	6	95.0	224	–
				94.9	225	
				94.8	225	

[a] s, singlet; d, doublet; b, not resolved. [b] Mixture of *cis* and *trans* isomers. [c] Recorded at 80 MHz; J(P–P) for dithio ligand could not be resolved due to overlapping signals.

temperature even after a few days. This suggests that the dithiolate group is strongly chelating in these complexes.

3. Experimental details

All preparations were carried out under a nitrogen atmosphere in dry solvents. All solvents were dried, degassed and distilled prior to use. $NaSSCNR_2$ (R = Me, Et) and $PMePh_2$ were obtained from commercial sources. K[SSCOEt] [10], Ph_2PSSNa [11] and $NH_4[SSP(OR)_2]$ [12] were prepared according to literature methods. The complexes $[Pt_2Ar_2(\mu\text{-}Cl)_2(PMePh_2)_2]$ (Ar = Ph, tol) were prepared by the reaction of *cis*-$[PtCl_2(CH_2{=}CH_2)(PMePh_2)]$ with Me_3ArSn in dichloromethane [13]. ^1H NMR spectra were recorded on a Bruker AMX-500 or AC-200 spectrometer in $CDCl_3$. Chemical shifts are relative to the internal chloroform peak at δ 7.26 ppm. $^{31}P\{^1H\}$ NMR spectra were recorded on a Bruker AMX-500 or Varian FT-80A NMR spectrometer. Chemical shifts are relative to external 85% H_3PO_4. Microanalyses of the compounds were carried out by the Analytical Chemistry Division, Bhabha Atomic Research Centre (BARC). The mass spectrum of $[PtPh\{SSP(OEt)_2\}(PMePh_2)]$ was recorded on a VG Micromass 7070F instrument.

3.1. Preparation of $[PtPh\{SSP(O^iPr)_2\}(PMePh_2)]$

To a dichloromethane–benzene (3:1, v/v) solution of $[Pt_2Ph_2(\mu\text{-}Cl)_2(PMePh_2)_2]$ (102 mg, 0.10 mmol), a solution of $NH_4[SSP(O^iPr)_2]$ (48 mg, 0.21 mmol) in 2-propanol (5 ml) was added dropwise with vigorous stirring under a nitrogen atmosphere. The whole was stirred for 3 h at room temperature. The solvents were removed under vacuum and the residue was extracted with benzene and filtered. The filtrate was concentrated *in vacuo* to give a paste which was dissolved in hexane and on cooling yielded white crystals of the title complex. Other complexes were prepared in a similar manner by dissolving the ammonium salt in the appropriate alcohol.

TABLE 2. ^1H NMR data for $[PtAr(S S)(PMePh_2)]$ complexes in $CDCl_3$

Complex	^1H NMR data [a], δ (ppm)
$[Pt_2Ph_2(\mu\text{-}Cl)_2(PMePh_2)_2]$	1.53 (d, 11 Hz) (major), 1.55 (d, 11 Hz) (minor) [PMe]; 6.72 (m), 7.09–7.64 (m) [Ph]
$[PtPh(SSCOEt)(PMePh_2)]$	1.46 (t, 7 Hz, OC–CH_3); 1.73 (d, 10.5 Hz), ^3J(Pt–H) = 49 Hz, [PMe]; 4.61 (q, 7 Hz, OCH_2–); 6.89 (m), 7.30–7.64 (m) [Ph]
$[PtPh(SSCNMe_2)(PMePh_2)]$	1.72 (d, 10.5 Hz, ^3J(Pt–H) = 50 Hz, [PMe]; 3.20 (s), 3.23 (s) [NMe_2]; 6.85 (m), 7.36–7.65 (m) [Ph]
$[PtPh(SSCNEt_2)(PMePh_2)]$	1.21 (t, 7 Hz), 1.24 (t, 7 Hz) [N–CH_3]; 1.72 (d, 10.2 Hz), ^3J(Pt–H) = 46 Hz, [PMe]; 3.62 (m, NCH_2), 6.79–7.62 (m, Ph)
$[PtPh\{SSP(OEt)_2\}(PMePh_2)]$	1.40 (t, 7 Hz, OC–CH_3); 1.67 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 4.26 (m, OCH_2–); 6.86 (m), 7.35–7.65 (m) [Ph]
$[PtPh\{SSP(O^nPr)_2\}(PMePh_2)]$	0.98 (t, 7 Hz, OCC–CH_3); 1.66 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 1.77 (Q, 7 Hz, OC–CH_2–); 4.14 (m, OCH_2–); 6.84(m), 7.33–7.64 (m) [Ph]
$[PtPh\{SSP(O^iPr)_2\}(PMePh_2)]$	1.38 (d, 6 Hz), 1.40 (d, 6 Hz) [OC–Me_2]; 1.65 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 4.96 (m, OCH–); 6.85 (m), 7.33–7.64 (m) [Ph]
$[PtPh\{SSP(O^iBu)_2\}(PMePh_2)]$	0.96 (d, 6.5 Hz, $OCCMe_2$); 1.64 (d, 10.5 Hz) [PMe]; 2.02 (m, CH–); 3.94 (m, OCH_2–); 6.83 (m), 7.32–7.75 (m) [Ph]
$[PtPh\{SSP(O^sBu)_2\}(PMePh_2)]$	0.97 (dt, CH_3– of ethyl); 1.37 (d, 6.2 Hz); 1.40 (d, 6.2 Hz) [methyl of O^sBu]; 1.64 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 1.75 (m, CH_2– of ethyl); 4.76 (m, OCH–); 6.83 (m), 7.28–7.64 (m) [Ph]
$[PtPh(SSPPh_2)(PMePh_2)]$	1.63 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 6.80 (m), 7.32–7.87 (m) [Ph]
$[Pt_2tol_2(\mu\text{-}Cl)_2(PMePh_2)_2]$	1.50 (d, 10.5 Hz) (major), 1.54 (d, 10.5 Hz) (minor) [PMe]; 2.08 (s) (minor), 2.12 (s) (major) [tol-Me]; 6.58 (m), 7.00–7.67 (m) [Ph + C_6H_4]
$[Pttol(SSCOEt)(PMePh_2)]$	1.45 (t, 7 Hz, CH_3); 1.73 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 2.18 (s, tol-Me); 4.59 (q, 7 Hz, OCH_2–); 6.74 (m), 7.22–7.68 (m) [Ph + C_6H_4]
$[Pttol(SSCNMe_2)(PMePh_2)]$	1.72 (d, 10.5 Hz), ^3J(Pt–H) = 47 Hz, [PMe]; 2.16 (s, tol-Me); 3.22 (s), 3.27 (s) [NMe_2]; 6.70 (m), 7.24–7.69 (m) [Ph + C_6H_4]
$[Pttol(SSCNEt_2)(PMePh_2)]$	1.23 (t, 7 Hz), 1.26 (t, 7 Hz) [N–C–Me]; 1.74 (d, 10.2 Hz), ^3J(Pt–H) = 47 Hz, [PMe]; 2.15 (s, tol-Me); 3.63 (q, 7 Hz, NCH_2–); 6.70 (m), 7.24–7.67 (m) [Ph + C_6H_4]
$[Pttol\{SSP(OEt)_2\}(PMePh_2)]$	1.40 (t, 7 Hz, OC–CH_3); 1.65 (d, 10.4 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 2.17 (s, tol-Me); 4.24 (m, OCH_2–); 6.66 (m), 7.16–7.66 (m) [Ph + C_6H_4]
$[Pttol\{SSP(O^nPr)_2\}(PMePh_2)]$	0.96 (t, 7 Hz, OCC–CH_3); 1.63 (d, 10.5 Hz) [PMe]; 1.75 (m, O–C–CH_2–); 2.15 (s, tol-Me); 4.12 (m, OCH_2–); 6.69 (m), 7.20–7.66 (m) [Ph + C_6H_4]
$[Pttol\{SSP(O^iPr)_2\}(PMePh_2)]$	1.38 (d, 6. Hz), 1.41 (d, 6 Hz) [OC–Me_2]; 1.63 (d, 10.5 Hz), ^3J(Pt–H) = 51 Hz, [PMe]; 2.17 (s, tol-Me); 4.94 (m, OCH–); 6.68 (m), 7.14–7.64 (m) [Ph + C_6H_4]
$[Pttol\{SSP(O^sBu)_2\}(PMePh_2)]$	0.94 (t, 7 Hz, Me of ethyl); 1.36 (d, 6 Hz), 1.38 (d, 6 Hz) [methyl of O^sBu]; 1.63 (d, 10.2 Hz) [PMe]; 1.72 (m, CH_2–); 2.16 (s, tol-Me); 4.74 (m, OCH–); 6.68 (m), 7.04–7.62 (m) [Ph + C_6H_4]

[a] s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dt, doublet of triplets.

TABLE 3. Melting point (m.p.) and analytical data for [PtAr(S S)(PMePh$_2$)] complexes

Complex	Recryst. solvent (% yield)	m.p. (°C)	% Analysis found (calcd)		
			C	H	N
[PtPh(SSCOEt)(PMePh$_2$)]	Benzene–hexane (69)	108	44.4 (44.5)	3.8 (3.9)	–
[PtPh(SSCNMe$_2$)(PMePh$_2$)]	Benzene–hexane (76)	95	44.8 (44.6)	4.2 (4.1)	2.1 (2.4)
[PtPh(SSCNEt$_2$)(PMePh$_2$)]	Hexane (22)	130	46.0 (46.4)	4.2 (4.5)	2.0 (2.2)
[PtPh{SSP(OEt)$_2$}(PMePh$_2$)]	Hexane (56)	78	41.7 (42.0)	4.1 (4.3)	–
[PtPh{SSP(O^{n}Pr)$_2$}(PMePh$_2$)]	Benzene–hexane (50)	106	43.4 (43.8)	4.5 (4.7)	–
[PtPh{SSP(O^{i}Pr)$_2$}(PMePh$_2$)]	Hexane (70)	107	43.6 (43.8)	4.5 (4.7)	–
[PtPh{SSP(O^{i}Bu)$_2$}(PMePh$_2$)]	Benzene–hexane (51)	100	46.3 (45.4)	4.8 (5.1)	–
[PtPh{SSP(O^{s}Bu)$_2$}(PMePh$_2$)]	Benzene–hexane (35)	112	45.5 (45.4)	5.2 (5.1)	–
[PtPh(SSPPh$_2$)(PMePh$_2$)]	CH_2Cl_2–hexane (28)	205	51.9 (51.6)	4.0 (3.9)	–
[Pttol(SSCOEt)(PMePh$_2$)]	Benzene–hexane (21)	86	45.4 (45.5)	4.1 (4.2)	–
[Pttol(SSCNMe$_2$)(PMePh$_2$)]	Benzene–hexane (72)	170	44.8 (45.5)	4.2 (4.3)	2.1 (2.3)
[Pttol(SSCNEt$_2$)(PMePh$_2$)]	Benzene–hexane (49)	125	46.8 (47.3)	4.6 (4.8)	2.1 (2.2)
[Pttol{SSP(OEt)$_2$}(PMePh$_2$)]	Hexane (59)	105	42.3 (42.9)	4.4 (4.5)	–
[Pttol{SSP(O^{n}Pr)$_2$}(PMePh$_2$)]	Liquid			–	
[Pttol{SSP(O^{i}Pr)$_2$}(PMePh$_2$)]	Hexane (51)	93	44.2 (44.6)	4.8 (4.9)	–
[Pttol{SSP(O^{s}Bu)$_2$}(PMePh$_2$)]	Liquid			–	

3.2. Preparation of [PtPh(SSCNEt$_2$)(PMePh$_2$)]

To a dichloromethane–benzene (3:1, v/v) solution of [Pt$_2$Ph$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] (80 mg, 0.8 mmol), an ethanolic solution of NaSSCNEt$_2$ · 3H$_2$O (36 mg, 0.16 mmol) was added with stirring under a nitrogen atmosphere. The contents were stirred for 3 h. The solvents were stripped off in vacuum and the residue was extracted with benzene and filtered. The filtrate was dried under reduced pressure. The product was recrystallized from benzene–hexane to give cream coloured crystals. Similarly, reactions with NaSSCNMe$_2$ were carried out.

3.3. Preparation of [PtPh(SSCOEt)(PMePh$_2$)]

To a dichloromethane–benzene (3:1, v/v) solution of [Pt$_2$Ph$_2$(μ-Cl)$_2$(PMePh$_2$)$_2$] (85 mg, 0.084 mmol), an ethanolic solution of KSSCOEt (30 mg, 0.186 mmol) was added dropwise with vigorous stirring under a nitrogen atmosphere. The reaction mixture was stirred for 4 h at room temperature. The solvents were evaporated *in vacuo*. The residue was extracted with benzene and filtered. The filtrate was concentrated to 1 ml under vacuum. Hexane (2 ml) was added and on slow evaporation gave cream coloured crystals of the title complex. Analytical data for all the complexes are given in Table 3.

Acknowledgments

Thanks are expressed to Drs. J.P. Mittal and D.D. Sood for their encouragement and interest in this work. We are grateful for the facilities at the Bio-Organic Division, BARC and Tata Institute of Fundamental Research for ^{1}H NMR spectroscopy and to the Analytical Chemistry Division, BARC for microanalysis of the samples.

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