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Preparation and characterization of phenylselenolato-bridged dinuclear platinum(II) complexes

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Abstract

Complexes of the type $[Pt_2X_2(\mu\text{-}Y)(\mu\text{-}SePh)(PR_3)_2]$ [X = Cl; Y = Cl, or SePh; PR_3 = PEt_3, PBu^n_3, PMe_2Ph, $PMePh_2$, PPh_3, PBu^t_3 and $PR_3 = \widetilde{PC}$ (metalated tri-tert-butylphosphine)] were prepared and characterized by elemental analyses and ^1H, ^{31}P, ^{77}Se and ^{195}Pt NMR spectroscopy. Treatment of $[Pt_2(\mu\text{-}SePh)_2(\widetilde{PC})_2]$ with anhydrous HCl in diethyl ether gave $[Pt_2Cl_2(\mu\text{-}SePh)_2(PBu^t_3)_2]$. A few bridge-cleavage reactions of $[Pt_2Cl_2(\mu\text{-}Cl)(\mu\text{-}SePh)(PBu^n_3)_2]$ have been investigated. The stereochemistry of the complexes in solution is discussed.

Introduction

Dinuclear platinum(II) complexes stabilized through bridging ligands are receiving much interest. The chemistry of such compounds bearing a variety of ligands such as halide, SCN, pyrazole, SR, OOCR, PR_2, etc. is now well established [1]. The platinum complexes with SR ligands have been studied extensively by us [2] and others [3]. These molecules offer many interesting possibilities. For example, the dithiolato-bridged complexes exhibit, in addition to *cis* ⇌ *trans* isomerization, another type of geometrical isomerism, namely *syn* and *anti*, while the chlorothiolato-bridged complexes in the presence of $SnCl_2 \cdot 2H_2O$ as a cocatalyst show high catalytic activity in homogeneous hydrogenation [2]. Recently the corresponding aryltellurolatobridged platinum(II) and palladium(II) complexes have been studied by Khandelwal et al. [4]. Similar complexes with SeR ligands, to our knowledge, have not been studied so far [5], although mononuclear platinum(II) complexes with RSe^- ligand have been investigated to some extent [6]. In view of the above and as part of our program on dinuclear platinum(II) complexes, we have synthesized a series of platinum(II) complexes with bridging SePh ligand and characterized them by multinuclear NMR data.

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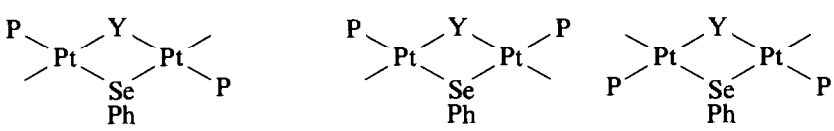
Results and discussion

The reaction of $[Pt_2Cl_2(\mu\text{-}Cl)_2(PR_3)_2]$ (PR_3 = PEt_3, PBu^n_3, PMe_2Ph, $PMePh_2$, PPh_3) or $[Pt_2(\mu\text{-}Cl)_2(\widehat{PC})_2]$ ($\widehat{PC}$ = metalated tri-tert-butylphosphine, $Bu^t_2PCMe_2CH_2$) with NaSePh, prepared by reductive cleavage of the Se–Se bond in diphenyldiselenide with sodium borohydride, gave phenylselenolato-bridged complexes of the types $[Pt_2Cl_2(\mu\text{-}SePh)_2(PR_3)_2]$ or $[Pt_2(\mu\text{-}SePh)_2(\widehat{PC})_2]$, respectively. The metalated complex $[Pt_2(\mu\text{-}SePh)_2(\widehat{PC})_2]$ on treatment with dry HCl in diethyl-ether resulted in the cleavage of the platinum–carbon bond and gave tri-tert-butylphosphine complex, $[Pt_2Cl_2(\mu\text{-}SePh)_2(PBu^t_3)_2]$. Remetalation of PBu^t_3 in the latter complex could not be achieved in ethanol at room temperature. Reaction of halogen-bridged dinuclear platinum(II) complexes, $[Pt_2Cl_2(\mu\text{-}Cl)_2(PR_3)_2]$ with $[Pt_2Cl_2(\mu\text{-}SePh)_2(PR_3)_2]$ on refluxing in benzene or dichloromethane yield complexes of the type $[Pt_2Cl_2(\mu\text{-}Cl)(\mu\text{-}SePh)(PR_3)_2]$ (PR_3 = PBu^n_3, PMe_2Ph) quantitatively.

The ^1H, $^{31}P\{^1H\}$ (Table 1) and ^{195}Pt NMR data for these complexes are consistent with the $[Pt_2X_2(\mu\text{-}Y)(\mu\text{-}Z)(PR_3)_2]$ arrangement, which exhibits *trans* (I) and *cis* (II and III) isomerism as shown below:



trans isomer *cis* isomers

The ^{31}P NMR spectra of $[Pt_2Cl_2(\mu\text{-}SePh)_2(PR_3)_2]$, except PR_3 = PBu^t_3, can be best interpreted in terms of *trans* configuration (I). The ^{31}P NMR spectrum of $[Pt_2Cl_2(\mu\text{-}SePh)_2(PBu^t_3)_2]$ displays two resonances attributable to *cis* and *trans* isomers. The resonance at higher frequency with the larger $^1J(Pt–P)$ value and smaller $^3J(Pt–P)$ and $^4J(Pt–P)$ values has been assigned to the *cis* isomer. The other resonance at lower frequency has been attributed to the *trans* isomer. The ^1H NMR spectrum showed two doublets for tert-butyl groups of the phosphine ligand assignable to *cis* and *trans* isomers. These assignments were made by comparison of their NMR data with those of analogous thiolato-bridged complexes reported earlier [2].

The geometry of $[Pt_2Cl_2(\mu\text{-}Y)_2(PR_3)_2]$, Y = thiolato group, is largely governed by the nature of R group on sulfur, the alkyl complexes preferring the *cis* configuration while the aryl derivatives exist in the *trans* form [2,3]. Clearly the SePh group behaves in a manner similar to the SAr moiety. $^2J(^{77}Se–^{31}P)$ ($\sim$ 150 Hz, clear in ^{77}Se NMR) observed in the spectrum of $[Pt_2Cl_2(\mu\text{-}SePh)_2(PBu^n_3)_2]$ was an additional feature.

The complex, $[Pt_2(\mu\text{-}SePh)_2(\widehat{PC})_2]$ could be isolated exclusively as the *cis* isomer, although the corresponding SR bridged complexes exist as a mixture of *cis* and *trans* isomer. For the *trans* isomer of the thiolato-bridged complexes, the $^3J(Pt–P)$ and $^4J(P–P)$ are about 50 and 12 Hz, respectively and the chemical shift values are about δ -3.0 ppm. The *cis* isomer displays a resonance at δ ca. -7.0 ppm with $^3J(Pt–P)$ and $^4J(P–P)$ of about 20 and 2 Hz, respectively [2].

Table 1

^1H and $^{31}P\{^1H\}$ NMR data for phenylselenolato-bridged dinuclear platinum(II) complexes

Complex	^{31}NMR data [a]			^{1}H NMR data [c]
	δ ^{31}P, ppm	^1J(Pt–P), Hz	Other couplings, Hz	δ ^{1}H, ppm
$[Pt_2Cl_2(\mu\text{-SePh})_2(PEt_3)_2]$	8.2	3195	^3J(Pt–P), 54; ^4J(P–P), 12.6	Ph, 8.06(d), 7.90(d), 7.23(m) 7.00(m); Et, 1.47–1.87(m, CH_2), 1.05(m, CH_3)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n_3)_2]$ [b]	0.6	3198	^3J(Pt–P), 49; ^4J(P–P), 12.5; ^2J(Pt–Pt), 1005; ^2J(Se–P), ca. 150	Ph, 8.05(d), 7.92(m) 7.22(m,br), 7.07(t) Bu, 1.63(br, α-CH_2), 1.33(br, β,γ-CH_2), 0.83(br, CH_3)
$[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n_3)_2]$	0.6	3853	^2J(Pt–Pt), 784	Ph, 8.03(m), 7.30(d) Bu, 1.55(br, α-CH_2), 1.34(br, β,γ-CH_2), 0.85(t, CH_3)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PMe_2Ph)_2]$	−13.3	3194	^3J(Pt–P), 47; ^4J(P–P), 16.5	Ph, 7.75(d), 6.92–7.43(m) Me, 1.70(d, J = 11 Hz), 1.65(d, J = 11 Hz)
$[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PMe_2Ph)_2]$	−17.3	3986	–	Ph, 6.98(m), 7.26–7.34(m) Me, 1.75 (d, J = 12 Hz), 1.60 (d, J = 12 Hz)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PPh_2Me)_2]$	1.5	3262	–	–
$[Pt_2Cl_2(\mu\text{-SePh})_2(PPh_3)_2]$	16.4	3267	^3J(Pt–P), 46	Ph, 6.86–7.68(m)
$[Pt_2(\mu\text{-SePh})_2(\widehat{PC})_2]$	−7.3	3041	^3J(Pt–P), 28; ^4J(P–P), 4	Ph, 8.00(m), 7.66(m), 7.10(m) PBu^t_2, 1.53(d, J = 13 Hz); $PCMe_2$, 1.39(d, J = 13.6 Hz); $PCCH_2$, 1.05(d, J = 9.7 Hz)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^t_3)_2]$ *trans*	64.6	3261	^3J(Pt–P), 41; ^4J(P–P), 10	PBu^t, 1.61(d, J = 12.3 Hz) (*cis* isomer), 1.39(d, J = 12.3 Hz) (*trans* isomer)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^t_3)_2]$ *cis*	76.2	3319	^3J(Pt–P), 36; ^4J(P–P), 8	Ph 8.34(m), 7.94(d), 7.23(m), 6.96(d), 6.60(t)

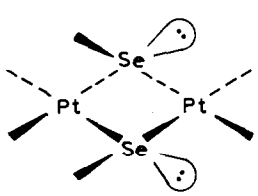
[a] Recorded in CH_2Cl_2, a capillary of C_6D_6 was used as lock. [b] Recorded in C_6H_6/C_6D_6. [c] Recorded in $CDCl_3$, d = doublet; t = triplet; m = multiplet; br = broad.

The magnitude of ^1J(Pt–P) in $[Pt_2X_2(\mu\text{-SePh})_2(PR_3)_2]$ complexes is considerably less than that of the corresponding $[Pt_2Cl_2(\mu\text{-Cl})_2(PR_3)_2]$ [7]. This suggests a high *trans* influence by the SePh group [8].

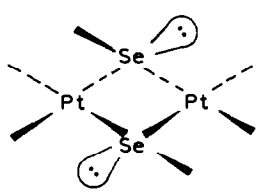
^{195}Pt and ^{77}Se NMR spectra of $[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n_3)_2]$ were recorded in C_6D_6. The ^{195}Pt{^{1}H} NMR spectrum showed expected doublet due to coupling with phosphorus nuclei (δ ^{195}Pt = −3992 ppm; ^1J(Pt–P) = 3197 Hz; ^3J(Pt–P) = 49 Hz; ^2J(Pt–Pt) = 1003 Hz) and had features similar to the spectra of $[Pt_2X_2(\mu\text{-Y})_2(PR_3)_2]$. The $^2J(^{195}Pt-^{195}Pt)$ for this complex is much greater than the chloro-bridged derivatives and is also higher than the corresponding SR-bridged complexes $[Pt_2Cl_2(\mu\text{-SR})_2(PBu^n_3)_2]$ [9]. The higher value of $^2J(^{195}Pt-^{195}Pt)$ owing to stronger interactions between the two platinum nuclei reflects the stronger ligation of SePh group than that of the SR ligands. However, the shortening of the dihedral angle in the SePh bridged square-planar dinuclear platinum(II) complexes compared to that of the thiolato-bridged derivatives may also lead to increased platinum–platinum interactions.

The ^{77}Se{^{1}H} NMR spectrum of the complex $[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n_3)_2]$ displayed a doublet due to coupling with the phosphorus (δ ^{77}Se = −608, $^2J(^{77}Se-^{31}P)$ = 147 Hz) together with multiplets at the base resulting from the coupling with ^{195}Pt nucleus. The observed doublet further establishes the *trans* configurations (I) suggested for these complexes. The $^2J(Se-P)_{cis}$ appears to be smaller than the digital resolution. The $^1J(^{195}Pt-^{77}Se)$ could not be assigned with confidence due to overlapping signals resulting from other isotopomers, particularly the one with [$^{31}P_2$ ^{195}Pt ^{195}Pt ^{77}Se] nuclei.

The complexes $[Pt_2X_2(\mu\text{-SePh})_2(PR_3)_2]$ may exhibit another form of geometrical isomerism *syn* or *anti*, depending on the arrangement of the phenyl groups with respect to each other. The isomerization of *syn* and *anti* forms is usually fast [10] and involves inversion at the chalcogen atom(s). A fast inversion process probably prevents detection of these isomers by NMR spectroscopy at ambient temperatures.



syn



anti

The complexes of the types $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PR_3)_2]$ have been assigned *cis* configuration in which phosphine ligands are *trans* to the bridging chloride (III) as observed for analogous thiolato-bridged complexes [2]. For *trans* isomer (I) two separate resonances with two different values of ^1J(Pt–P) are expected. The *cis* isomer with phosphine *trans* to SePh groups (II) would give a single resonance with the ^1J(Pt–P) value of about 3200 Hz.

A few bridge cleavage reactions of $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n_3)_2]$ with neutral donor ligands were carried out and were examined by ^{31}P NMR spectroscopy. Reaction with an excess of pyridine readily gave three phosphorus-containing species, δ ^{31}P −8.2 [^1J(Pt–P) = 3346 Hz], −7.9 [^1J(Pt–P) = 3438 Hz] and 0.6 ppm. The former resonance has been assigned for *trans*-$[PtCl_2(py)(PBu^n_3)]$ and did not

change with time over a period of several days. The latter two species established an equilibrium within 3 h, integration of which indicated 1 : 1 ratio, and no detectable change was observed over a period of several days. The resonance at δ 0.6 ppm is attributed to dinuclear complex $[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n{}_3)_2]$ which is formed through a mononuclear intermediate $[PtCl(SePh)(py)(PBu^n{}_3)]$ (δ −7.9 ppm, $^1J(Pt-P) = 3438$ Hz).

Reaction of $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n{}_3)_2]$ with triphenylarsine in 1 : 1 and 1 : 2 stoichiometry proceeded similarly except 1 : 1 reaction was slower than the 1 : 2. Spectra obtained immediately after mixing the $CDCl_3$ solutions of the reactants, displayed five resonances at δ 3.8, 3.5, 0.6, −0.7 ($^1J(Pt-P) = 3849$ Hz), and −3.20 ($^1J(Pt-P) = 3341$ Hz) ppm. After 3 h resonances due to the parent dimer (δ −0.7 ppm $^1J(Pt-P) = 3849$ Hz) almost vanished. After 20 h only three phosphorus-containing species were present in the solution (δ 3.5, 0.6, −3.2 ppm) and there were no detectable changes when the solution was kept for more than a week. The resonance at δ −3.20 ($^1J = 3341$ Hz) ppm can be attributed to $[PtCl_2(PBu^n{}_3)(AsPh_3)]$. The resonance at 0.6 ppm had characteristic features of $[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n{}_3)_2]$, and possibly formed via an intermediate having resonance at δ 3.8 ppm. The resonances at δ 3.8 and 3.5 ($^1J(Pt-P) = 3054$ Hz) ppm may be assigned to $[PtCl(SePh)(PBu^n{}_3)(AsPh_3)]$. The latter resonance was attributable to a complex with the phosphine ligand *trans* to the SePh group (strong *trans* influence) while the former was assigned to a complex containing the phosphine ligand *trans* to the chloride or the triphenylarsine and converted to the dinuclear complex.

Triphenylphosphine also reacts with $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n{}_3)_2]$ to give a complex mixture of mononuclear platinum(II) products including *cis*-$[PtCl_2(PPh_3)(PBu^n{}_3)]$.

In general the properties of phenylselenolato-bridged complexes are similar to those of the corresponding SR-bridged derivatives.

Experimental

The complexes $[Pt_2Cl_2(\mu\text{-Cl})_2(PR_3)_2]$ ($PR_3 = PEt_3$, $PBu^n{}_3$, PMe_2Ph, $PMePh_2$, PPh_3) [11] and $[Pt_2(\mu\text{-Cl})_2(\widehat{PC})_2]$ [12] were prepared by published procedures. Phosphines were obtained from Strem chemicals and K_2PtCl_4 was prepared in the laboratory from platinum metal. Analytical grade solvents were used in all reactions. ^1H NMR spectra were recorded on a Bruker AC-200 spectrometer in $CDCl_3$. Chemical shifts are relative to internal chloroform. The $^{31}P\{^1H\}$, $^{77}Se\{^1H\}$ and $^{195}Pt\{^1H\}$ NMR spectra were obtained on a Varian FT 80A NMR instrument operating at 32.203, 15.169 and 17.01 MHz, respectively. Chemical shifts are relative to external 85% H_3PO_4 for ^{31}P, Na_2PtCl_6 in D_2O for ^{195}Pt and Ph_2Se_2 in C_6D_6 for ^{77}Se. Microanalyses were performed by Bio-organic Chemistry Division, BARC. Melting points were determined in capillary tubes and are uncorrected.

Preparation of $[Pt_2Cl_2(\mu\text{-SePh})_2(PR_3)_2]$

To a methanolic solution of PhSeSePh (68 mg, 0.22 mmol) a dilute methanolic solution of $NaBH_4$ was added with vigorous stirring under nitrogen. Addition of $NaBH_4$ solution was stopped when a colourless solution of PhSeNa was obtained. To this a solution of $[Pt_2Cl_2(\mu\text{-Cl})_2(PBu^n{}_3)_2]$ (202 mg, 0.22 mmol) in dichloromethane was added all at once (in case of $[Pt_2Cl_2(\mu\text{-Cl})_2(PPh_3)_2]$ a suspension in

Table 2

Physical and analytical data for phenylselenolato-bridged dinuclear platinum(II) complexes

Complexes	m.p. (°C)	Colour	Yield (%) [a]	Analyses (Found (Calc) (%))	
				C	H
$[Pt_2Cl_2(\mu\text{-SePh})_2(PEt_3)_2]$	221–223	Yellow	50	28.16 (28.55)	3.82 (3.98)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n{}_2)_2]$	120–122	Yellow	60	36.77 (36.71)	5.51 (5.48)
$[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n{}_3)_2]$	110–113	Pale yellow	67	33.54 (34.08)	5.41 (5.62)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PMe_2Ph)_2]$	218–220	Yellow	64	31.75 (32.05)	3.07 (3.05)
$[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PMe_2Ph)_2]$	224–227	Pale Yellow	70	28.12 (28.44)	2.84 (2.93)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PMePh_2)_2]$	225–227	Yellow	62	38.16 (38.88)	2.93 (3.08)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PPh_3)_2]$	265–267(d) [b]	Yellow	36	43.79 (44.43)	2.95 (3.11)
$[Pt_2(\mu\text{-SePh})_2(\widehat{PC})_2]$ [c]	200–202	Yellow	60	38.95 (39.13)	5.72 (5.65)
$[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^t{}_3)_2]$	230–232	Orange	–	36.95 (36.71)	5.50 (5.48)

[a] Recrystallized from CH_2Cl_2/ethanol. [b] d – Complex decomposed, [c] PC = $Bu^t{}_2PCMe_2CH_2$ (metalated tri-tert-butylphosphine).

dichloromethane was added). Reactants were stirred at room temperature for 5 h. Solvents were stripped off under reduced pressure. The residue was extracted with dichloromethane and recrystallized from CH_2Cl_2–ethanol in 60% (151 mg) yield as yellow crystals. Other complexes of this series were prepared similarly and the pertinent data are listed in Table 2.

Preparation of $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PR_3)_2]$

To a dichloromethane solution (20 ml) of $[Pt_2Cl_2(\mu\text{-SePh})_2(PBu^n{}_3)_2]$ (100 mg, 0.085 mmol), was added solution of $[Pt_2Cl_2(\mu\text{-Cl})_2(PBu^n{}_3)_2]$ (79.5 mg, 0.085 mmol) in the same solvent. The mixture was heated under reflux with stirring for 4 h. Solvent was removed under vacuum and the residue was recrystallized from CH_2Cl_2–ethanol in 83% (150 mg) yield as pale-yellow crystals. Similarly $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PMe_2Ph)_2]$ was prepared.

Reaction of $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n{}_3)_2]$ with an excess of pyridine

Pyridine (0.2 ml) was added to a $CDCl_3$ solution (3 ml) of $[Pt_2Cl_2(\mu\text{-Cl})(\mu\text{-SePh})(PBu^n{}_3)_2]$ (75 mg) in a NMR tube and progress of the reaction was examined by $^{31}P\{^1H\}$ NMR spectroscopy. Similar reactions with $AsPh_3$ (1 : 1 and 1 : 2) and PPh_3 (1 : 2) were carried out and examined by ^{31}P NMR spectroscopy.

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