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STRUCTURAL AND MASS SPECTROMETRIC CHARACTERISATION OF THE
DINUCLEAR PLATINUM(II) SELENIDO-PHENYLSELENOLATO COMPLEX
[Pt₂(μ-Se)(μ-SePh)(PPh₃)₄]PF₆

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The reaction of [Pt₂(μ-Se)₂(PPh₃)₄] with diphenyliodonium bromide gave [Pt₂(μ-Se)(μ-SePh)·(PPh₃)₄]⁺ through arylation of a selenide ligand. The complex is characterised by ³¹P NMR spectroscopy, elemental microanalysis, ESI mass spectrometry, and a single crystal X-ray diffraction study, which showed the four-membered {Pt₂Se₂} ring to be slightly puckered.

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Dinuclear platinum(II) sulfide complex [Pt₂(μ-S)₂(PPh₃)₄] **1a** [1] and analogues with alternative phosphine [2, 3] or arsine [4] ligands have been extensively investigated over a considerable length of time [5] because of the rich and diverse chemistry they display. Reactivity includes alkylation [6, 7] and arylation [8, 9] reactions (generating platinum-thiolate complexes), redox chemistry [10, 11], metalloligand chemistry (whereupon one or both sulfide ligands coordinate to metal centres generating sulfide-bridged homo- [12] and hetero- [13] metallic aggregates), and interesting ligand-based reactivity [14].

In contrast, the chemistry of selenide analogue [Pt₂(μ-Se)₂(PPh₃)₄] **1b** [15] is less well established, though aspects of the alkylation [16, 17] and coordination [18] chemistry of [Pt₂(μ-Se)₂·(PPh₃)₄] have been briefly investigated. The relative lack of studies on [Pt₂(μ-Se)₂(PPh₃)₄] is noteworthy since it has shown some different reactivity to the sulfide analogue, such as metal-scrambling reactions [19], metal-chalcogen bond cleavage leading to the synthesis of organoselenoethers [20], and the synthesis of interesting metal-selenide aggregates [21].

To date, there are no reports of the reactivity of [Pt₂(μ-Se)₂(PPh₃)₄] towards arylating reagents leading to arylselenolate complexes. Complexes of selenolate ligands have been extensively studied, including dinuclear complexes with bridging selenolate ligands [22]. In light of the perceived undesirability of handling organoselenols such as PhSeH due to their toxicity, air-sensitivity and disagreeable stench, we have investigated the arylation chemistry of [Pt₂(μ-Se)₂(PPh₃)₄] as a convenient route for the synthesis of dinuclear platinum complexes containing the phenylselenolate ligand.

EXPERIMENTAL

Materials and methods. Electrospray mass spectra were recorded in a dichloromethane-methanol solution using a Bruker MicrOTOF instrument, periodically calibrated using sodium formate, and employing a capillary exit voltage of 150 V for routine spectra, but higher voltages to inves-

tigate fragmentation behaviour. $^{31}\text{P}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker Avance spectrometer operating at 162 MHz, and the spectra were referenced relative to external H_3PO_4 . UV-visible spectra were recorded in a CH_2Cl_2 solution on a Perkin Elmer Lambda 11 spectrophotometer. $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ [23] and Ph_2IBr [24] were prepared by the literature procedures. NH_4PF_6 was used as supplied by Aldrich. Reactions were carried out in a reagent grade methanol solvent, without additional purification or precautions to exclude air, light or moisture.

Preparation. $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ (315 mg, 0.197 mmol) and Ph_2IBr (76 mg, 0.211 mmol) were suspended in methanol (35 ml) and the mixture was stirred at room temperature for 72 h, giving a clear deep orange-brown solution. After filtration to remove traces of insoluble matter, NH_4PF_6 (400 mg, 2.45 mmol) was added to the stirred filtrate, giving an orange-brown precipitate. The solid was filtered, washed with water (2×5 ml) and dried under vacuum to give $2 \cdot \text{PF}_6$ as a brown solid (250 mg, 70 %). M.p. > 300 °C (decomp.). UV-vis (CH_2Cl_2) λ_{max} 430 nm (w), < 360 nm (vs). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3), δ 23.6 (m, $^1J(\text{PtP})$ 3320 Hz, PPh_3 *trans* to SePh) and 23.5 (m, $^1J(\text{PtP})$ 2740 Hz, PPh_3 *trans* to Se). ESI MS (positive ion), m/z 1675.17.

Structure determination. The compound was recrystallised from chloroform-diethyl ether, giving dark orange-brown single crystals of the *tris*-chloroform solvate suitable for an X-ray diffraction study. Found: C 46.24, H 3.14. $\text{C}_{78}\text{H}_{65}\text{F}_6\text{P}_5\text{Pt}_2\text{Se}_2 \cdot 3\text{CHCl}_3$ requires C 44.66, H 3.15 %. Crystal data are summarised in Table 1. Data were collected on a Bruker SMART CCD diffractometer and processed using the standard software. Absorption corrections were carried out using SADABS [25]. The structure was solved using SHELX-97 and refined on F^2 using SHELXL-97 [26] operated under WinGX [27]. All non-hydrogen atoms were treated anisotropically and hydrogen atoms were included with a riding model with $d(\text{C}-\text{H})$ 0.95 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic hydrogen atoms. The complex crys-

Table 1

Crystal data and structure refinement details for
 $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6 \cdot 3\text{CHCl}_3$ ($2 \cdot \text{PF}_6 \cdot 3\text{CHCl}_3$)

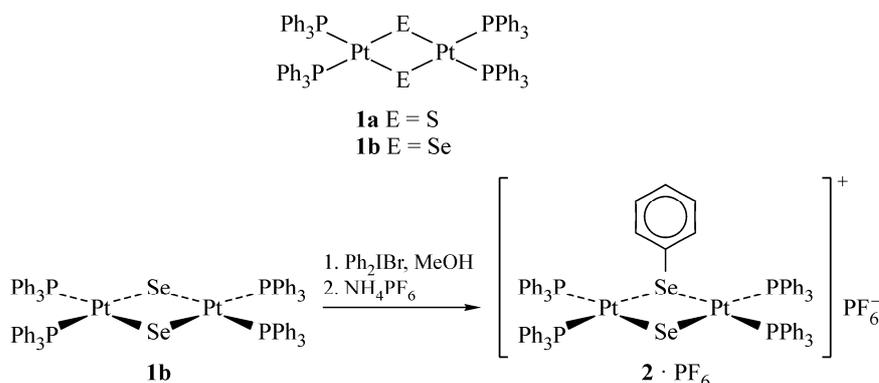
Empirical formula	$\text{C}_{81}\text{H}_{68}\text{Cl}_9\text{F}_6\text{P}_5\text{Pt}_2\text{Se}_2$
Formula weight	2177.35
Crystal system	Triclinic
Space group	$P-1$
Unit cell dimensions $a, b, c, \text{Å}$; $\alpha, \beta, \gamma, \text{deg.}$	14.0298(17), 15.228(2), 21.124(2) 77.603(7), 89.015(7), 81.236(7)
Temperature, K	90(2)
Volume, Å^3	4355.7(9)
Z	2
Density (calculated), g/cm^3	1.660
Absorption coefficient, mm^{-1}	4.466
$F(000)$	2120
Crystal size, mm	0.24 \times 0.12 \times 0.08
Collected / Independent reflections	48202 / 20046 [$R_{\text{int}} = 0.0793$]
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7000 and 0.3052
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	20046 / 54 / 971
Goodness-of-fit on F^2	1.012
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0536$, $wR_2 = 0.1040$
R indices (all data)	$R_1 = 0.0975$, $wR_2 = 0.1183$
Largest diff. peak and hole, $\text{e}/\text{Å}^3$	1.677 and -1.828

tallises with three CHCl_3 molecules; one CHCl_3 was very disordered so the H atom was not included in the refinement, and Cl atoms were treated as partial Cl atoms over 8 sites. The F atoms of the PF_6^- ion and the other Cl atoms were restrained using the ISOR command in SHELX, to provide better ellipsoids.

A CIF-file containing complete information on the studied structure was deposited with CCDC, deposition number 951013, and is freely available from the web page http://www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The reaction of $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ with diphenyliodonium bromide (Ph_2IBr) proceeded smoothly in a methanol suspension following the methodology previously reported for the phenylthiolate complex $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]^+$ [8], giving an orange-brown solution containing the mono-arylated $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]^+$ cation. The complex was isolated as a brown microcrystalline solid $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6 \cdot 2 \cdot \text{PF}_6$ by the addition of excess NH_4PF_6 , and the synthesis is summarised in Scheme 1. To date, the only complexes with singly derivatised $\{\text{Pt}_2(\mu\text{-Se})(\mu\text{-SeR})\}$ cores have $\text{R} = \text{Me}$, *n*-Bu or $\text{CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{Cl-}p$ [16] so the arylation reaction described here extends the range of the known derivatives to include the first arylselenolate derivatives.

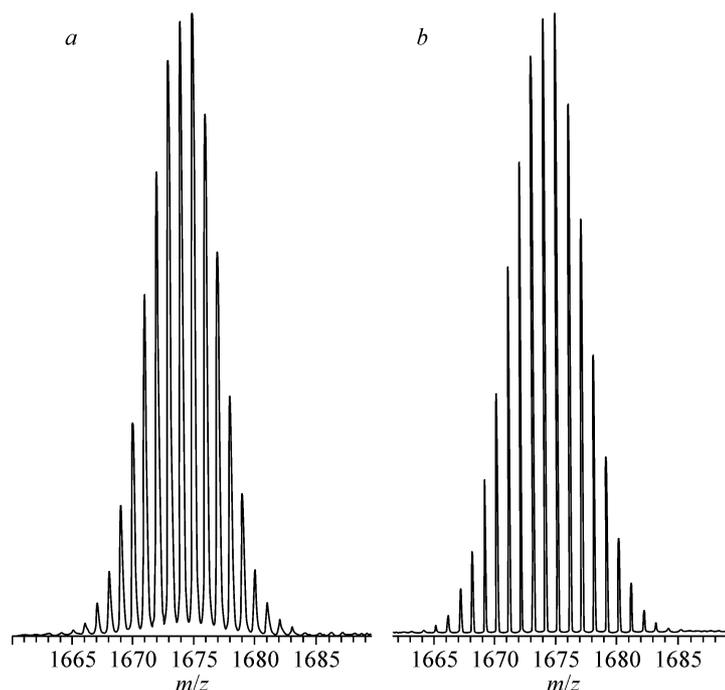


Scheme 1. Synthesis and isolation of $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6$ ($2 \cdot \text{PF}_6$)

The attempted reaction of $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ with an excess of Ph_2IBr in methanol for 16 h, followed by the analysis of the reaction mixture by positive-ion ESI MS, gave only **2**, plus the unreacted diphenyliodonium cation, and its aggregate ions (Ph_2I^+ m/z 280.95, $(\text{Ph}_2\text{I})_2\text{Br}^+$ m/z 642.84, $(\text{Ph}_2\text{I})_3\text{Br}_2^+$ m/z 1002.72, $(\text{Ph}_2\text{I})_4\text{Br}_3^+$ m/z 1364.61) with no evidence for the doubly-phenylated complex $[\text{Pt}_2(\mu\text{-SePh})_2(\text{PPh}_3)_4]^{2+}$. The same behaviour was observed for the sulfide analogue $[\text{Pt}_2(\mu\text{-S})_2(\text{PPh}_3)_4]$ [8].

Complex $2 \cdot \text{PF}_6$ was characterised by $^{31}\text{P}\{^1\text{H}\}$ NMR spectroscopy, ESI mass spectrometry, and an X-ray crystallographic study. The $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of $2 \cdot \text{PF}_6$ in CDCl_3 showed (in addition to the expected multiplet for the PF_6^- anion) two complex multiplet resonances for the PPh_3 ligands at δ 23.6 and 23.5 due to the two inequivalent types of PPh_3 ligands, *trans* to SePh or Se respectively. Coupling to ^{195}Pt produced distinctive satellites, and the value of $^1\text{J}(\text{PtP})$ for the phosphines *trans* to selenide is approximately 2740 Hz, while that for the phosphines *trans* to SePh is approximately 3320 Hz, due to the lower *trans*-influence [28] of a SePh group compared to selenide. These coupling constants are both smaller than the corresponding values for $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]^+$ (2613 Hz and 3291 Hz) [8], indicating the selenium ligands to have slightly lower *trans*-influences than their sulfur counterparts. The UV-visible spectrum of a brown solution of $2 \cdot \text{PF}_6$ in CH_2Cl_2 showed an intense charge-transfer absorption at <360 nm, with a low intensity shoulder centred at 430 nm. The bright yellow sulfur analogue $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]\text{PF}_6$ also showed an intense charge transfer absorption at <360 nm, but the shoulder at 420 nm in this case was much better resolved.

Fig. 1. Comparison of (a) the experimental (from positive-ion ESI mass spectrum) and (b) calculated isotope patterns for $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]^+$ ion **2** (m/z 1675.17 for the most intense peak in the isotope pattern)



The positive-ion ESI mass spectrum of **2**·PF₆ at a capillary exit voltage of 150 V showed a single dominant peak at m/z 1675.17 (calculated m/z 1675.17), with an isotope pattern that showed good agreement with the calculated pattern (Fig. 1). The fragmentation behaviour of **2** was investigated by increasing the capillary exit voltage. At 180 V there was no observed fragmentation, but at 210 V several fragment ions were observed and assigned as $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_3]^+$ (m/z 1413.08, from loss of PPh₃), $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_2]^+$ (m/z 1150.98, from loss of 2PPh₃), $[\text{Pt}(\text{SePh})(\text{PPh}_3)_2]^+$ (m/z 877.80, from fragmentation of the Pt₂Se₂ core), and $[\text{Pt}(\text{PPh}_3)(\text{Ph}_2\text{PC}_6\text{H}_4)]^+$ (m/z 718.14, from cyclometallation of a PPh₃ ligand) [29]. Numerous very low intensity ions were also observed at $m/z < 1100$, but were not assigned. The spectra of the thiolate analogue $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]^+$ were recorded under analogous conditions; while the overall features were fairly similar with the parent cation base peak in both cases, the formation of $[\text{Pt}(\text{SePh})(\text{PPh}_3)_2]^+$ and $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})\cdot(\text{PPh}_3)_2]^+$ were not paralleled in the sulfur system under these conditions.

ESI mass spectrometry has also been used to demonstrate that the metalloligand coordination chemistry of $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]^+$ is analogous to the related systems such as $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]^+$. Mono-derivatised $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SR})(\text{PPh}_3)_4]^+$ complexes have been shown to act as metalloligands to metal centres (gold(I) or mercury(II)) through the underivatised sulfide ligand [8, 30]. Mass spectrometry-monitored micro-scale reaction of **2**·PF₆ with Ph₃PAuCl gave the $[\text{Pt}_2(\mu\text{-SeAuPPh}_3)(\mu\text{-SePh})(\text{PPh}_3)_4]^{2+}$ ion at m/z 1066.88 (calculated m/z 1067.11) and the reaction with organomercury chlorides RHgCl (R = Ph or ferrocenyl = Fc) gave $[\text{Pt}_2(\mu\text{-SeHgFc})(\mu\text{-SePh})(\text{PPh}_3)_4]^{2+}$ at m/z 1029.85 (calculated m/z 1030.07) and $[\text{Pt}_2(\mu\text{-SeHgPh})(\mu\text{-SePh})(\text{PPh}_3)_4]^{2+}$ at m/z 975.88 (calculated m/z 976.09).

Since the previously reported X-ray crystal structure of the sulfur analogue $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})\cdot(\text{PPh}_3)_4]^+$ was disordered [8], the structure of $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6$ **2** was determined. Selected bond lengths and angles are given in Table 2. The structure of the core of cation **2** is shown in Fig. 2, which confirms the structure as a dinuclear platinum complex with bridging selenido and phenylselenolato ligands. The four-membered {Pt₂Se₂} ring is slightly puckered, with a dihedral angle θ between the two PtSe₂ planes of 164.87°. This can be compared to the corresponding θ angle of 146.33° in the (more puckered) sulfur complex $[\text{Pt}_2(\mu\text{-S})(\mu\text{-SPh})(\text{PPh}_3)_4]^+$ [8] and the fully planar ($\theta = 180^\circ$) parent selenide $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ [15]. The four-membered ring in **2** is markedly less

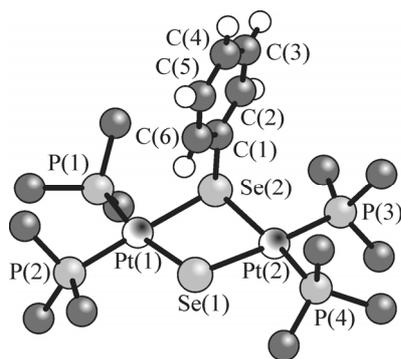


Fig. 2. Structure of the core of the $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6 \cdot 3\text{CHCl}_3$ complex showing the atom numbering scheme; only *ipso* carbon atoms of the four triphenylphosphine ligands are shown, and the PF_6^- counterion and CHCl_3 molecules of crystallisation are omitted for clarity

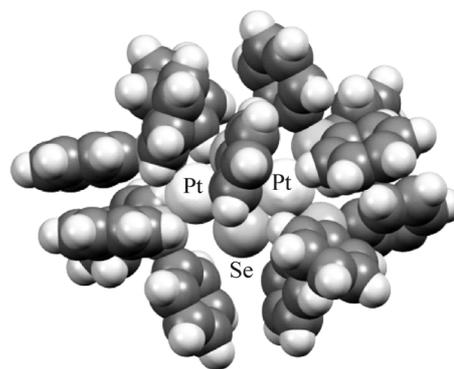


Fig. 3. Space-filling view of $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]^+$ cation **2**, showing the twisting of the SePh ring and the $\pi-\pi$ interaction between SePh phenyl and phenyl on a triphenylphosphine ligand

T a b l e 2

Selected bond lengths (Å) and angles (deg.) for $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]\text{PF}_6 \cdot 3\text{CHCl}_3$ (**2**· PF_6^- · 3CHCl_3)

Pt(1)—P(2)	2.3256(19)	P(2)—Pt(1)—P(1)	99.37(7)	P(4)—Pt(2)—P(3)	100.49(7)
Pt(1)—Se(1)	2.4918(8)	P(2)—Pt(1)—Se(1)	92.80(5)	P(1)—Pt(1)—Se(1)	167.75(5)
Pt(2)—P(4)	2.3421(19)	P(2)—Pt(1)—Se(2)	172.35(5)	P(1)—Pt(1)—Se(2)	86.85(5)
Pt(2)—Se(2)	2.5095(8)	P(4)—Pt(2)—Se(2)	163.68(5)	P(3)—Pt(2)—Se(2)	91.59(5)
Se(2)—C(1)	2.018(9)	P(4)—Pt(2)—Se(1)	87.82(5)	P(3)—Pt(2)—Se(1)	171.39(5)
Pt(1)—P(1)	2.3403(18)	Se(1)—Pt(1)—Se(2)	81.12(2)	Se(2)—Pt(2)—Se(1)	80.69(2)
Pt(1)—Se(2)	2.5217(8)	Pt(1)—Se(1)—Pt(2)	98.11(3)	Pt(2)—Se(2)—Pt(1)	97.76(3)
Pt(2)—P(3)	2.3510(19)	C(1)—Se(2)—Pt(2)	96.6(3)	C(1)—Se(2)—Pt(1)	102.6(3)
Pt(2)—Se(1)	2.5261(8)				

puckered than the corresponding methylselenolate complex $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SeMe})(\text{PPh}_3)_4]^+$ for which $\theta = 133.6^\circ$ [16]. The geometry about the two platinum centres is only slightly distorted from the regular square-planar geometry, with τ_4 parameters (as defined by Houser et al [31]) of 0.14 and 0.18 for Pt(1) and Pt(2) respectively, indicating that Pt(1) is closest to square-planar (theoretical $\tau_4 = 0.00$).

The phenylselenolate ligand adopts an *exo* position in a 'pocket' bound by the PPh_3 ligands, with the Se—C bond 'perpendicular' to the $\{\text{Pt}_2\text{Se}_2\}$ ring. The phenyl ring is not coplanar with the $\text{Se}\cdots\text{Se}$ vector, but is instead twisted, as can be seen in Fig. 2 and the space-filling plan diagram in Fig. 3; the $\text{Se}\cdots\text{Se}-\text{C}-\text{C}$ torsion angle is approximately 18° . As can be seen in Fig. 3, the phenylselenolate aromatic ring appears to be involved in a weak $\pi-\pi$ interaction with a phenyl ring on P(1) phosphorus (ring C(121) to C(126)), the interplanar angle between the two rings being 4.05° , and the distance between the two ring centroids being 3.785 \AA .

In conclusion, through a facile phenylation reaction using diphenyliodonium bromide, it has been possible to convert the selenido complex $[\text{Pt}_2(\mu\text{-Se})_2(\text{PPh}_3)_4]$ into the phenylselenolate complex $[\text{Pt}_2(\mu\text{-Se})(\mu\text{-SePh})(\text{PPh}_3)_4]^+$, which has been fully characterised. The structural determination on the complex indicates a significantly flatter metallacycle compared to the sulfur analogue, with NMR spectroscopy indicating that the selenium ligands have slightly lower *trans* influences. These results suggest that the designer synthesis of a range of arylselenolate ligands on a diplatinum framework could be possible using an extension of this methodology.

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