

Reactivity of [Pt(dppf)Cl₂] toward Simple Organic Thiolates: Preparation and Structures of [Pt(dppf)(SPh)₂], [Pt(dppf)(S-*n*-Pr)₂], and [Pt(dppf)(SCH₂CH₂CH₂S)] (dppf = Fe(η^5 -C₅H₄PPh₂)₂)

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The reactions of [Pt(dppf)Cl₂] with thiophenol (PhSH), 1-propanethiol (*n*-PrSH), and 1,3-propanedithiol (HSCH₂CH₂CH₂SH) gave the corresponding Pt-dppf-di(thiolato) compounds, [Pt(dppf)(SPh)₂] (**1**), [Pt(dppf)(S-*n*-Pr)₂] (**2**), and [Pt(dppf)(SCH₂CH₂CH₂S)] (**3**), respectively. All products are monomeric and 4-coordinate square-planar compounds and were structurally characterized by X-ray diffraction. Electrochemical measurements (cyclic voltammograms) revealed that the oxidation potential of the dppf ligand appears to depend on the type of the group on the thiolato ligand.

Key Words : Platinum, dppf, Organic thiolates, X-ray, Cyclic voltammograms

Introduction

The bidentate ligand 1,1'-bis(diphenylphosphino)ferrocene (dppf = Fe(η^5 -C₅H₄PPh₂)₂) has obtained continuous attractions due to the interesting structural and redox properties of its metal compounds.¹ In particular, group 10 metal-dppf compounds are the most intensively studied family with more than 40 crystal structures having been reported.² Very recently, several reports on the group 10 metal-dppf compounds containing metal-sulfur bonds occurred in the literature, in which the sulfur-donating ligands are thiocarboxylate (SC(O)Me),³ homochalcogenide {1,2-benzenedithiol (HSC₆H₄SH), 3,4-toluenedithiol (HSC₆H₃MeSH), and 3,6-dichlorobenzenedithiol (HSC₆H₂Cl₂SH)} or mixed-chalcogenide {2-mercaptophenol (HSC₆H₄OH) and thionicotinic acid (HSC₅-H₃NCO₂H)},⁴ and crown thioether 1,4,7-trithiacyclononane (9S3).⁵ Previously, several dppe (1,2-bis(diphenylphosphino)ethane = Ph₂PCH₂CH₂PPh₂) analogues were reported, which include platinum-dppe-di(thiolato) compounds (1,2-ethanedithiolato, 1,3-propanedithiolato, and 1,2-benzenedithiolato),⁶ [M(dppe)(EPh)₂] (M = Pd, Pt; E = O, S, Se),⁷ and heterocycle-substituted 1,2-enedithiolato compounds of Ni, Pd, and Pt of the type [M(dppe){S₂C₂-(heterocycle)(H)}].⁸

It has long been well known that the coordination chemistry of simple thiolates (M-SR) is complicated by two features: (1) oligomer formation through strong thiolato bridges and (2) oxidation of thiol to disulfide by metals in high oxidation states.⁹ Although the latter feature is usually a minor problem for the group 10 metals, the former feature (formation of thiolato bridges) frequently leads to oligomeric species. As stated above, the thiolates having been employed so far for the preparation of their group 10 metal-dppf compounds are of an activated or a chelate type. In this respect, we decided to investigate the reactions of [Pt(dppf)Cl₂] with simple

organic thiolates such as thiophenol (PhSH), 1-propanethiol (*n*-PrSH), and 1,3-propanedithiol (HSCH₂CH₂CH₂SH). Herein we report the preparation and structural characterization of [Pt(dppf)(SPh)₂], [Pt(dppf)(S-*n*-Pr)₂], and [Pt(dppf)(SCH₂CH₂CH₂S)].

Experimental Section

Unless otherwise stated, all reactions have been performed with standard Schlenk line and cannula techniques under argon at room temperature. Hydrocarbon solvents were stirred over concentrated H₂SO₄ for about 48 h, neutralized with K₂CO₃, stirred over sodium metal, and distilled by vacuum transfer. Diethyl ether was stirred over sodium metal and distilled by vacuum transfer. Dichloromethane was stirred over CaH₂ and distilled by vacuum transfer. Methyl alcohol and triethylamine were distilled and stored under argon. NMR solvent (CDCl₃) was degassed by freeze-pump-thaw cycles and stored over molecular sieves under argon. Potassium tetrachloroplatinate(IV) (K₂PtCl₄), thiophenol (C₆H₅SH, PhSH), 1-propanethiol (CH₃CH₂CH₂SH, *n*-PrSH), and 1,3-propanedithiol (HSCH₂CH₂CH₂SH) were purchased. [Pt(SMe₂)₂Cl₂], dppf, and [Pt(dppf)Cl₂] were prepared by the literature methods.¹⁰⁻¹² All products were recrystallized from either dichloromethane-pentane or dichloromethane-hexane.

Electrochemical measurements were performed with a 263A EG&G potentiostat under nitrogen. A standard three-electrode system was employed with a glassy carbon as a working electrode, a platinum (Pt) wire as a counter electrode, and a silver (Ag) wire as a reference electrode. Measurements were carried out in CH₂Cl₂ containing 0.1 M [*n*-Bu₄N][PF₆] as a supporting electrolyte and about 1 mM metal complex. Instrumental *iR* compensations were applied in all measurements. The ferrocene/ferrocenium couple was 0.632 V at 300 mV s⁻¹ in this cell. The supporting electrolyte (tetrabutylammonium hexafluorophosphate, [*n*-Bu₄N][PF₆])

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and ferrocene (FeCp₂) were recrystallized from ethanol before use.

¹H-, ¹³C{¹H}-, and ³¹P{¹H}-NMR spectra were recorded with a Varian Unity Inova 500 MHz spectrometer. IR spectra were recorded with a Nicolet Avatar 320 FTIR spectrophotometer. Elemental analyses were performed with EA1110 (CE instrument, Italy) by the Korea Basic Science Institute.

Preparation of [Pt(dppf)(SPh)₂] (1). [Pt(dppf)Cl₂] (100 mg, 0.12 mmol), thiophenol (PhSH, 0.025 mL, 0.24 mmol), and NEt₃ (0.068 mL, 0.49 mmol) in dichloromethane (20 mL) were stirred for 24 h, and then the solvent was removed under vacuum. The resultant yellow solids were washed with diethyl ether (10 mL × 2) and methyl alcohol (10 mL × 2), and then the solvent was removed under vacuum. This product was recrystallized to give a CH₂Cl₂-solvated product. (93 mg, 0.089 mmol, 74%). ¹H-NMR (CDCl₃): δ 7.81-7.28 (20H, m, *Ph* in dppf), 7.18-6.76 (10H, m, *SPh*), 4.29 (4H, s, C₅H₄), 4.13 (4H, s, C₅H₄). ¹³C{¹H}-NMR (CDCl₃): δ 144.4-122.0 (*Ph*), 75.5-72.8 (C₅H₄). ³¹P{¹H}-NMR (CDCl₃): δ 19.5 (t, *J*_{Pt-P} = 3016 Hz). Anal. Calcd for C₄₇H₄₀P₂S₂Cl₂FePt (*M*_r = 1052.69): C, 53.62; H, 3.83; S, 6.09. Found: C, 53.49; H, 3.91; S, 5.94. mp: 230-232 °C. IR (KBr): 3056, 2922, 2859, 1627, 1576, 1473, 1435, 1167, 1093, 1030, 740, 552 cm⁻¹.

Preparation of [Pt(dppf)(S-*n*-Pr)₂] (2). [Pt(dppf)Cl₂] (100 mg, 0.12 mmol), 1-propanethiol (*n*-PrSH, 0.022 mL, 0.24 mmol), and NEt₃ (0.068 mL, 0.49 mmol) in dichloromethane (20 mL) were stirred for 24 h to give yellow crystals (45 mg, 0.051 mmol, 64%). ¹H-NMR (CDCl₃): δ 7.94-7.30 (20H, m, *Ph*), 4.42, 4.37, 4.23, 4.14, 3.86 (8H, s, C₅H₄), 2.64 (4H, t, *J*_{H-H} = 7.5 Hz, SCH₂CH₂CH₃), 1.34 (4H, m, SCH₂CH₂CH₃), 0.74 (6H, t, *J*_{H-H} = 7.5 Hz, SCH₂CH₂CH₃). ¹³C{¹H}-NMR (CDCl₃): δ 135.5-127.6 (*Ph*), 75.6-72.9 (C₅H₄), 33.6 (SCH₂CH₂CH₃), 28.0 (SCH₂CH₂CH₃), 14.3 (SCH₂CH₂CH₃). ³¹P{¹H}-NMR (CDCl₃): δ 21.1 (t, *J*_{Pt-P} = 2930 Hz). Anal. Calcd for C₄₀H₄₂P₂S₂FePt (*M*_r = 899.74): C, 53.39, H, 4.71, S, 7.13. Found: C, 52.63; H, 4.82; S, 7.01. mp: 182-184 °C. IR (KBr): 3054, 2922, 2865, 1631, 1479, 1435, 1165, 1097, 1034, 747, 696 554 cm⁻¹.

Preparation of [Pt(dppf)(SCH₂CH₂CH₂S)] (3). [Pt(dppf)Cl₂] (100 mg, 0.12 mmol), 1,3-propanedithiol (HSCH₂-CH₂CH₂SH, 0.024 mL, 0.24 mmol), and NEt₃ (0.068 mL, 0.49 mmol) in dichloromethane (20 mL) were stirred for 24 h to give yellow crystals (54 mg, 0.063 mmol, 52%). ¹H-NMR (CDCl₃): δ 7.84-7.33 (20H, m, *Ph*), 4.28 (4H, s, C₅H₄), 4.12 (4H, s, C₅H₄), 3.00 (4H, m, SCH₂CH₂CH₂S), 2.06 (2H, m, SCH₂CH₂CH₂S). ¹³C{¹H}-NMR (CDCl₃): δ 135.3-127.8 (*Ph*), 76.0-72.9 (C₅H₄), 31.6 (SCH₂CH₂CH₂S), 24.5 (SCH₂CH₂CH₂S). ³¹P{¹H}-NMR (CDCl₃): δ 22.2 (t, *J*_{Pt-P} = 2906 Hz). Anal. Calcd for C₃₇H₃₆OP₂S₂FePt (*M*_r = 873.66): C, 50.86, H, 4.15, S, 7.34. Found: C, 50.46; H, 4.02; S, 7.24. mp: 216-218 °C (dec.). IR (KBr): 3048, 2902, 2831, 1626, 1479, 1434, 1168, 1096, 1032, 744, 697, 492 cm⁻¹.

X-ray Structure Determination of 1-3. All X-ray data were collected with the use of a Siemens P4 diffractometer equipped with a Mo X-ray tube and a graphite monochro-

Table 1. X-ray data collection and structure refinement

	1-CH ₂ Cl ₂	2	3-H ₂ O
empirical formula	C ₄₇ H ₄₀ P ₂ S ₂ -Cl ₂ FePt	C ₄₀ H ₄₂ P ₂ -S ₂ FePt	C ₃₇ H ₃₆ OP ₂ -S ₂ FePt
fw	1052.69	899.74	873.66
temperature, K	293(2)	293(2)	293(2)
crystal system	monoclinic	monoclinic	monoclinic
space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> , Å	21.973(2)	14.301(2)	9.782(2)
<i>b</i> , Å	10.549(1)	13.956(2)	17.220(2)
<i>c</i> , Å	19.496(1)	19.832(3)	20.448(3)
β, deg	102.990(5)	110.39(1)	93.28(1)
<i>V</i> , Å ³	4403.5(7)	3710.2(9)	3438.6(10)
<i>Z</i>	4	4	4
<i>d</i> _{calc} , g cm ⁻³	1.588	1.611	1.688
μ, mm ⁻¹	3.824	4.384	4.729
<i>F</i> (000)	2088	1792	1728
<i>T</i> _{min}	0.3754	0.1932	0.3226
<i>T</i> _{max}	1.0000	0.6630	0.7536
2θ range (°)	3.5-50	3.5-50	3.5-50
scan type	ω	ω	ω
scan speed	variable	variable	variable
No. of reflns measured	7852	6728	6334
No. of reflns unique	7606	6457	5973
No. of reflns with <i>I</i> > 2σ(<i>I</i>)	6313	4853	5247
No. of params refined	496	416	404
Max., in Δρ (e Å ⁻³)	1.235	1.171	1.597
Min., in Δρ (e Å ⁻³)	-0.745	-1.019	-0.547
<i>GOF</i> on <i>F</i> ²	1.020	1.019	1.038
<i>R</i>	0.0388	0.0393	0.0288
<i>wR</i> ₂ ^a	0.0959	0.0849	0.0716

$$^a wR_2 = \Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]^{1/2}$$

mator. Details on crystal data and intensity data are shown in Table 1. The intensity data were empirically corrected for absorption with ψ-scan data. All calculations were carried out with the use of SHELXTL programs.¹³

A yellow crystal of **1**, shaped as a block of approximate dimensions 0.64 × 0.28 × 0.12 mm³, was used for crystal and intensity data collection. The unit cell parameters and

Table 2. Selected bond lengths (Å) and bond angles (°)

1-CH ₂ Cl ₂	2	3-H ₂ O
Pt1-P1 2.313(1)	Pt1-P1 2.274(2)	Pt1-P1 2.288(1)
Pt1-P2 2.294(1)	Pt1-P2 2.284(2)	Pt1-P1 2.288(1)
Pt1-S1 2.364(1)	Pt1-S1 2.338(2)	Pt1-S1 2.326(1)
Pt1-S2 2.354(1)	Pt1-S2 2.354(2)	Pt1-S2 2.354(1)
S1-C35 1.765(6)	P1-Pt1-P2 102.26(6)	P1-Pt1-P2 99.38(4)
S2-C41 1.756(6)	P1-Pt1-S1 83.39(6)	P1-Pt1-S1 86.10(4)
P1-Pt1-P2 98.40(5)	P1-Pt1-S2 175.10(6)	P1-Pt1-S2 177.29(4)
P1-Pt1-S1 81.73(5)	P2-Pt1-S1 173.15(7)	P2-Pt1-S1 174.51(4)
P1-Pt1-S2 169.80(5)	P2-Pt1-S2 82.64(6)	P2-Pt1-S2 83.33(4)
P2-Pt1-S1 178.10(5)	S1-Pt1-S2 91.71(7)	S1-Pt1-S2 91.19(4)
P2-Pt1-S2 91.72(5)	C35-S1-Pt1 105.2(4)	
S1-Pt1-S2 88.20(5)	C38-S2-Pt1 107.4(3)	

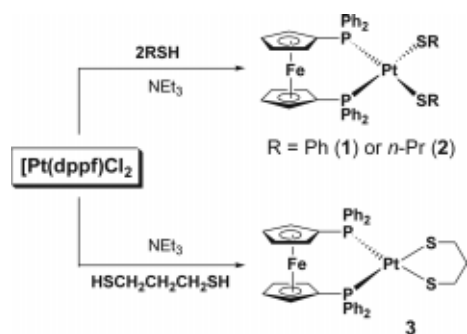
systematic absences, $h0l$ ($l = 2n + 1$) and $0k0$ ($k = 2n + 1$), unambiguously indicated $P2_1/c$ as a space group. A yellow crystal of **2**, shaped as a cube of approximate dimensions $0.34 \times 0.32 \times 0.30 \text{ mm}^3$, was used. The unit cell parameters and systematic absences, $h0l$ ($l = 2n + 1$) and $0k0$ ($k = 2n + 1$), unambiguously indicated $P2_1/c$ as a space group. A yellow crystal of **3**, shaped as a block of approximate dimensions $0.32 \times 0.30 \times 0.24 \text{ mm}^3$, was used. The unit cell parameters and systematic absences, $h0l$ ($h + l = 2n + 1$) and $0k0$ ($k = 2n + 1$), unambiguously indicated $P2_1/n$ as a space group. Structures were solved by direct methods. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were located and refined isotropically. Selected bond lengths and bond angles for **1-3** are shown in Table 2.

Results and Discussion

Preparation. Platinum-dppf-di(thiolato) compounds containing monodentate (**1** and **2**) or chelate (**3**) thiolato ligands are readily prepared by metathesis from $[\text{Pt}(\text{dppf})\text{Cl}_2]$ and the corresponding organic thiolates in the presence of triethylamine (Scheme 1). As expected, no reaction occurs in the absence of triethylamine, which acts as a base to remove HCl escaping from the reaction. The types of the thiolates employed in this study are aryl (PhSH), alkyl (n -PrSH), and chelating alkyl ($\text{HSCH}_2\text{CH}_2\text{CH}_2\text{SH}$). The dppe analogue of compound **1** was previously prepared from $[\text{Pt}(\text{dppe})\text{Cl}_2]$ and PhSH in the presence of tributylamine.⁷

All products are monomeric and four-coordinate square-planar compounds. We speculate that the sterically demanding dppf ligand prevents the formation of a dimeric or an oligomeric species. Each compound is crystalline and has been characterized by spectroscopy (^1H -, $^{13}\text{C}\{^1\text{H}\}$ -, $^{31}\text{P}\{^1\text{H}\}$ -NMR, and IR), elemental analysis, and X-ray diffraction. The ^1H - and $^{13}\text{C}\{^1\text{H}\}$ -NMR spectra of these compounds display the correct number of peaks, splittings, and intensities associated with the three components: four phenyl rings from the dppf ligand, two Cp rings from the dppf ligand, and thiolato ligands. In $^{31}\text{P}\{^1\text{H}\}$ -NMR spectra, all compounds show a single peak at δ 19.5–22.2 with platinum satellites ($J_{\text{Pt-P}} = 2906\text{--}3016 \text{ Hz}$), which indicate equivalent phosphorus nuclei due to the high symmetry of these compounds.

Structure. The molecular structures of **1-3** are shown in



Scheme 1

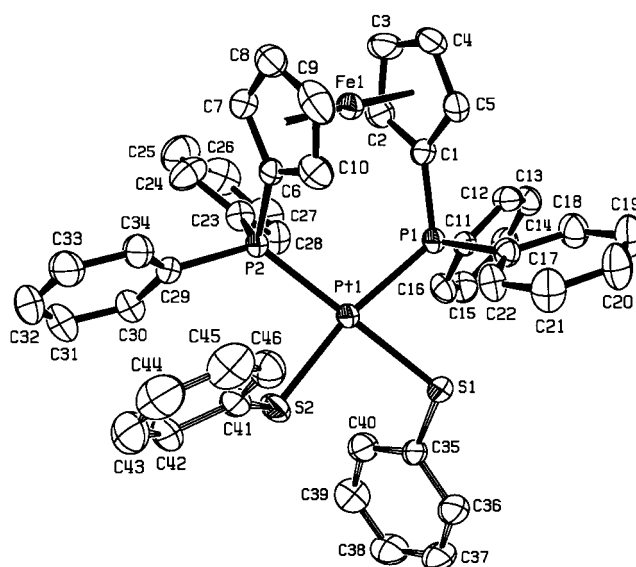


Figure 1. ORTEP drawing of **1**, showing the atom-labeling scheme and 50% probability thermal ellipsoids.

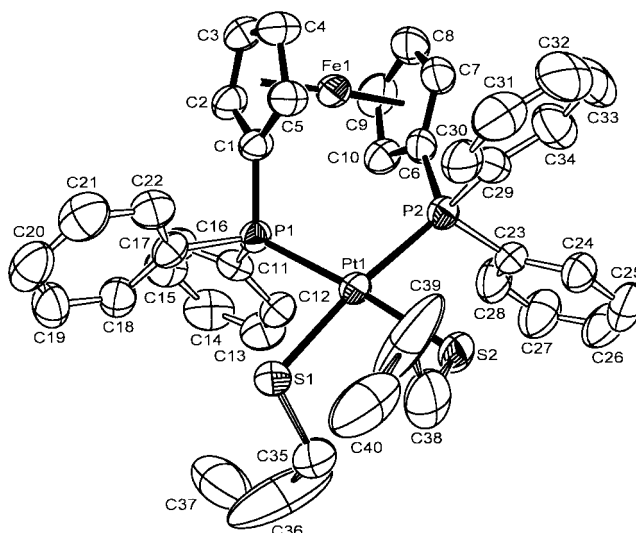


Figure 2. ORTEP drawing of **2**.

Figures 1–3, respectively. All compounds are isostructural. Compounds **1** and **2** have two terminal thiolato ligands (SR; R = Ph or n -Pr) and one bidentate dppf ligand. Compound **3** has one chelating dithiolato ($\text{SCH}_2\text{CH}_2\text{CH}_2\text{S}$) ligand as well as one bidentate dppf ligand. The coordination sphere of each Pt can be described as distorted square planar. The equatorial plane, defined by two sulfur atoms, two phosphorus atoms, and the Pt metal, is nearly planar with the average atomic displacement of 0.031 Å for **1**, 0.038 Å for **2**, or 0.004 Å for **3**. The Pt–S bond lengths range from 2.326(1) Å to 2.364(1) Å and are consistent with those found for other platinum-dppf-di(thiolato) compounds.³⁵

The two Cp rings in the ferrocene moiety of the dppf ligand in all compounds are twisted from each other with the dihedral angle of $4.9(1)^\circ$ (**1**), $7.2(4)^\circ$ (**2**), or $3.7(1)^\circ$ (**3**). The torsion angles of P1–C1–C6–P2 fall in the range of $29.2(2)$ – $39.4(3)^\circ$, indicating a gauche conformation of the two Cp

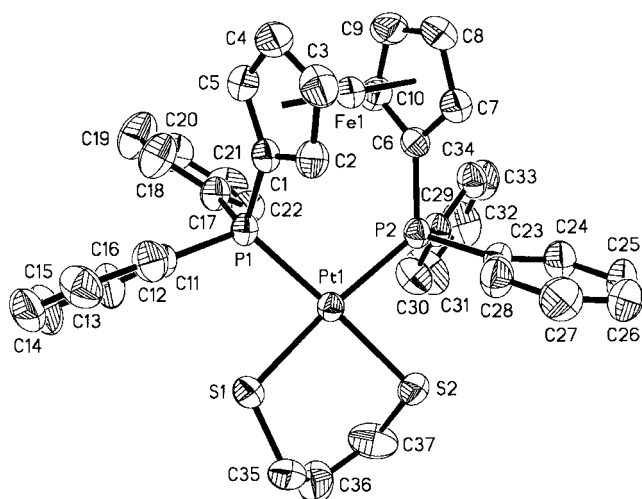


Figure 3. ORTEP drawing of **3**.

rings. For comparison, the ideal torsion angles for the gauche and eclipsed conformations are 36° and 72° , respectively.² The Fe-Ct distances (Ct: a centroid of the Cp ring) are in the range of 1.632–1.647 Å. The Ct1-Fe-Ct2 (Ct1: C1–C5; Ct2: C6–C10) vectors are essentially linear with the range of 177.1 – 179.7° . The P...Fe...P angles fall in the range of $61.66(3)$ – $63.33(4)^\circ$. The long Pt...Fe distances (4.282(1)–4.296(1)) indicate no direct interactions between those metals. The above bonding parameters within the ferrocene moiety are relatively consistent with those found for square-planar platinum compounds in which the dppf group acts as a ligand.¹

Cyclovoltammograms. The dppf ligand is a well-known redox-active ligand, and its complexes are expected to exhibit a ferrocene-centered oxidation process, together with the additional redox processes at other metal centers in the molecule.¹ The free dppf ligand in dichloromethane exhibits one irreversible, ferrocene-centered oxidation.¹⁴

Cyclovoltammograms of compounds **1–3** are shown in Figure 4. Each compound exhibits one irreversible oxidation wave at 0.404–0.626 V that corresponds to the ferrocene-centered oxidation of the dppf ligand. The oxidation potential of the free dppf ligand (0.53 V) is shifted to a more positive value (0.626 V) for compound **1**, but it is shifted to more negative values for compounds **2** (0.404 V) and **3** (0.520 V). These observations might arise from relative electron-donating abilities of the alkyl groups on the thiolato ligands. The electron-withdrawing phenyl group in compound **1** causes the dppf ligand to oxidize at a higher potential compared with the free dppf. Conversely, the electron-donating alkyl groups in compounds **2** and **3** make the oxidation of the dppf ligand occur at lower potentials.

Compounds **1** and **3** exhibit one quasi-reversible peak at 1.194 V (for **1**) and at 1.202 V, respectively, which might result from the Pt(II) \rightarrow Pt(III) oxidation. Interestingly, compound **2** exhibits two quasi-reversible oxidations at 1.174 and 1.468 V, of which the lower value can be ascribed to the Pt oxidation but the higher value cannot be clearly explained. The relative strengths of electron-donating abilities of the

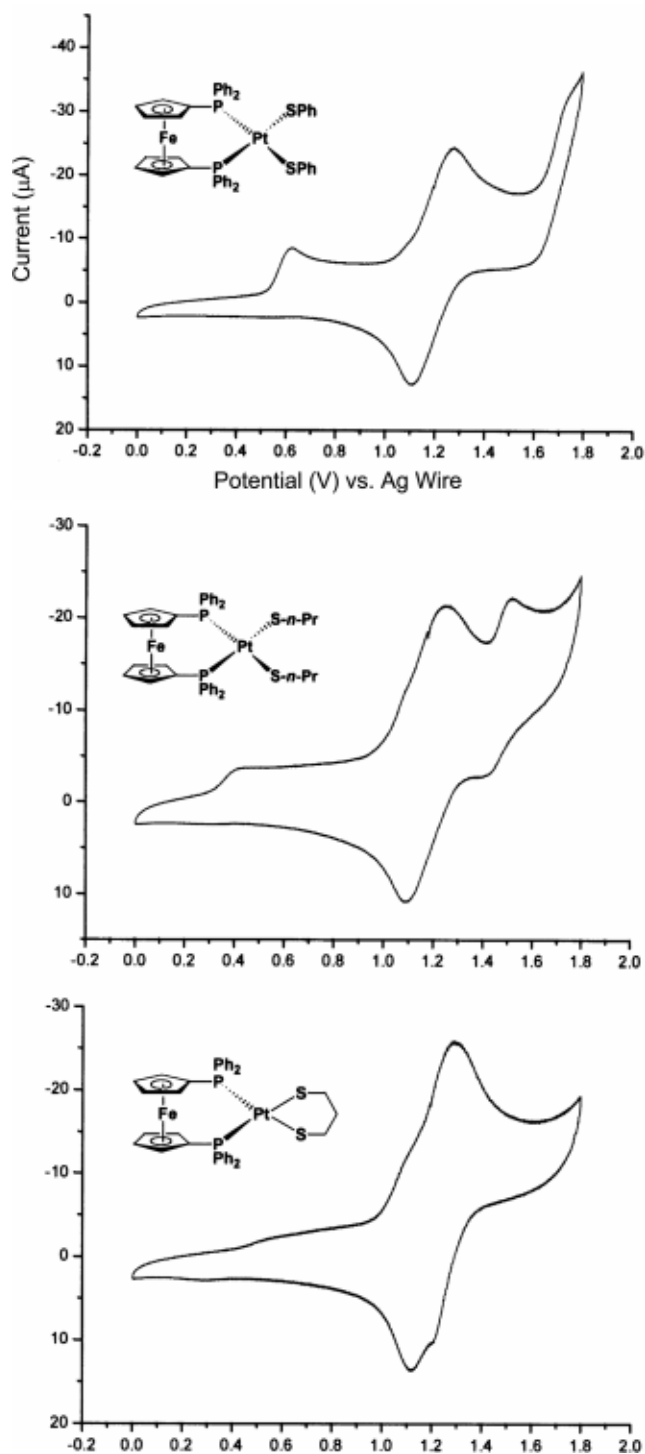


Figure 4. Cyclovoltammograms (300 mV s^{-1}) in 0.1 M $[n\text{-Bu}_4\text{N}][\text{PF}_6]\text{-CH}_2\text{Cl}_2$ solutions.

thiolate substituents cannot explain the tendency of the potential values for the Pt oxidation, because the order of oxidation potential values expected on those strengths is $1 > 2 \approx 3$.

In summary, we have prepared three platinum-dppf-di(thiolato) compounds, $[\text{Pt}(\text{dppf})(\text{SPh})_2]$, $[\text{Pt}(\text{dppf})(\text{S-}n\text{-Pr})_2]$, and $[\text{Pt}(\text{dppf})(\text{SCH}_2\text{CH}_2\text{CH}_2\text{S})]$, each of which includes an aryl, an alkyl, or a chelating thiolato ligand. All compounds

are monomeric and have been structurally characterized by X-ray diffraction. The oxidation potential of the dppf ligand in these compounds appears to depend on the type of the group on the thiolato ligand.

Supplementary material. Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center, CCDC No. 187497 for compound **1**-CH₂Cl₂, 187498 for compound **2**, and 187499 for **3**-H₂O. Copies of this information may be obtained free of charge from: The director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; E-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk/perl/catreg/catreg.cgi>).

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