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Synthesis and characterization of heterodinuclear thiolate complexes containing the Pd(η^3-allyl)$^+$ moiety. Crystal structure of [(dppe)Pd(μ-SC_6H_4Me-*p*)$_2$Pd(η^3-C_3H_5)][ClO_4]

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Abstract

Heterodinuclear complexes of the types [(dppe)M(μ-$SR)_2$Pd(η^3-allyl)][ClO_4] (M = Pd or Pt) and [(dppe)Pt(μ-SC_2H_4S)Pd(η^3-allyl)][ClO_4] [dppe = 1,2-bis(diphenylphosphino)ethane; allyl = C_3H_5 or C_4H_7] have been obtained by reaction of the corresponding *cis*-dithiolato complexes, [M($SR)_2$ (dppe)] or [Pt(SC_2H_4S)(dppe)], and [Pd(η^3-allyl)($PhCN)_2$][ClO_4]. The identity of these complexes has been established by NMR (^{1}H and ^{31}P) spectroscopy. The crystal structure of [(dppe)Pd(μ-SC_6H_4Me-*p*)$_2$Pd(η^3-C_3H_5)][ClO_4] has been determined by single-crystal X-ray methods. The *p*-tolyl groups on the sulfur atoms adopt a *syn* conformation with respect to the central Pd–S_2–Pd core.

Keywords: Palladium; Platinum; Thiolate complexes; Allyl complexes

1. Introduction

The relevance of transition-metal complexes with S-donor ligands as models of biologically redox-active metalloproteins [1] has stimulated interest in the chemistry of metal thiolates [2,3]. Platinum and palladium μ-thiolate complexes are also of recent interest due to their possible use in homogeneous catalysis [4–6]. For example, systems of the type [Pt_2(μ-SR)(μ-$Cl)Cl_2(PR'_3)_2$]/$SnCl_2 \cdot H_2O$ have a high catalytic activity in the hydrogenation of styrene [7].

We have recently described the synthesis of di- and tri-nuclear nickel(II), palladium(II) or platinum(II) complexes with bridging thiolato groups of the types [NBu_4]$_2$[$M_2(C_6F_5)_4$(μ-$SR)_2$], [NBu_4]$_2$[$M_3(C_6F_5)_4$(μ-$SR)_4$] (M = Ni, Pd or Pt) [8,9] and [{Pd($CH_2C_9H_6N$)}$_2$(μ-SR)(μ-O_2CR'')] ($CH_2C_9H_6N$ = 8-quinolylmethyl) [10]. Dinuclear thiolato-bridged allylpalladium complexes have been reported recently [11]. On the other hand, the synthesis and study of heterobinuclear complexes have been the subject of active research over the past years, as they can serve as models for active intermediates in heterobimetallic catalysis [12].

In this paper we report the preparation of a variety of heterodinuclear MM′ (M = Pd, Pt; M′ = Pd) complexes with bridging thiolato ligands by using *cis*-dithiolato complexes [M($SR)_2$ (dppe)] [M = Pd or Pt; dppe = 1,2-bis(diphenylphosphino)ethane] and [Pd(η^3-allyl)($PhCN)_2$]$^+$, which contain the readily removable PhCN ligands, as building blocks.

2. Experimental

The C, H, N, S analyses were performed with a Carlo Erba model EA 1108 microanalyzer. Decomposition temperatures were determined with a Mettler TG-50 thermobalance at a heating rate of 5°C min^{-1} and the solid sample under nitrogen flow (100 ml min^{-1}). Molar conductivities were measured in acetone solution (c Å 5×10^{-4} mol dm^{-3}) with a Crison 525 conducti-

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meter. The NMR spectra were recorded on a Bruker AC 200E or Varian Unity 300 spectrometer, using $SiMe_4$ and H_3PO_4 as standards, respectively. Infrared spectra were recorded on a Perkin–Elmer 1430 spectrophotometer using Nujol mulls between polyethylene sheets. Solvents were dried by the usual methods. The starting complexes $[M(SR)_2](dppe)$ (M = Pd or Pt), $[Pt(SC_2H_4S)(dppe)]$ [13] and $[Pd(\eta^3\text{-allyl})(PhCN)_2][ClO_4]$ (allyl = C_3H_5 or C_4H_7) [14] were prepared by procedures described elsewhere.

Safety note. **Caution!** Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small amounts of these materials should be prepared, and they should be handled with great caution.

2.1. Preparation of complexes *1–11*

To a solution of the corresponding benzonitrile complex $[Pd(\eta^3\text{-allyl})(PhCN)_2][ClO_4]$ (0.3 mmol) in methanol (20 cm^3) was added the corresponding *cis*-dithiolato complex $[M(SR)_2$ (dppe)] or $[Pt(SC_2H_4S)(dppe)]$ (0.3 mmol) with constant stirring at room temperature (r.t.) (2 h for the palladium compounds or 4 h for the platinum complexes) to afford a suspension from which the solvent was partially evaporated under reduced pressure and, after addition of water, filtered-off. The desired yellow complex was air-dried.

Complex **1**: Yield 54%; m.p. 154°C (dec.). *Anal.* Calc. for $C_{41}H_{39}ClO_4P_2Pd_2S_2$: C, 50.8; H, 4.1; S, 6.6. Found: C, 50.8; H, 4.1; S, 6.3%. Λ_M 130 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.6–7.3 (m, 20H, aromatics dppe), 6.99 (d, 4H, H_o of SPh, $J_{om} = 7.7$ Hz), 6.88 (t, 2H, H_p of SPh, $J_{mp} = 7.7$), 6.69 (pseudo-t, 4H, H_m of SPh), 5.56 (m, 1H, CC*H*C allyl), 3.76 (d, 2H, H_{syn}, $J = 6.9$), 3.25 (d, 2H, H_{anti}, $J = 12.6$), 2.39 (d, 4H, CH_2 dppe, $J_{HP} = 22.5$). ^{31}P NMR ($CDCl_3$) δ 60.8 (s).

Complex **2**: Yield 55%; m.p. 189°C (dec.). *Anal.* Calc. for $C_{43}H_{43}ClO_4P_2Pd_2S_2$: C, 51.7; H, 4.3; S, 6.4. Found: C, 52.0; H, 4.5; S, 6.4%. Λ_M 145 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.6–7.3 (m, 20H, aromatics dppe), 6.86 (d, 4H, H_o of SC_6H_4Me, $J_{om} = 7.8$ Hz), 6.48 (d, 4H, H_m of SC_6H_4Me, $J_{om} = 7.8$), 5.57 (m, 1H, CC*H*C allyl), 3.76 (d, 2H, H_{syn}, $J = 6.9$), 3.24 (d, 2H, H_{anti}, $J = 12.7$), 2.38 (d, 4H, CH_2 dppe, $J_{HP} = 22.6$), 2.12 (s, 6H, Me). ^{31}P NMR ($CDCl_3$): δ 60.0 (s).

Complex **3**: Yield 56%; m.p. 175°C (dec.). *Anal.* Calc. for $C_{33}H_{39}ClO_4P_2Pd_2S_2$: C, 45.4; H, 4.5; S, 7.3. Found: C, 45.1; H, 4.5; S, 7.3%. Λ_M 150 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 8.0–7.4 (m, 20H, aromatics dppe), 5.46 (m, 1H, CC*H*C allyl), 3.93 (d, 2H, H_{syn}, $J = 6.9$ Hz), 3.03 (d, 2H, H_{anti}, $J = 12.4$), 2.59 (d, 4H, CH_2 dppe, $J_{HP} = 23.4$), 2.03 (m, 4H, CH_2S), 0.88 (t, 6H, CH_3 CH_2S, $J = 7.2$). ^{31}P NMR ($CDCl_3$): δ 60.5 (s).

Complex **4**: Yield 59%; m.p. 175°C (dec.). *Anal.* Calc. for $C_{34}H_{41}ClO_4P_2Pd_2S_2$: C, 46.0; H, 4.7; S, 7.2. Found: C, 45.5; H, 4.7; S, 7.1%. Λ_M 148 cm^2 S mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.8–7.4 (m, 20H, aromatics dppe), 3.64 (s, 2H, H_{syn}), 2.87 (s, 2H, H_{anti}), 2.59 (d, 4H, CH_2 dppe, $J_{HP} = 24.0$ Hz), 2.03 (m, 4H, CH_2S), 1.98 (s, 3H, CH_3 allyl), 0.86 (t, 6H, CH_3 CH_2S, $J = 7.2$). ^{31}P NMR ($CDCl_3$): δ 60.7 (s).

Complex **5**: Yield 68%; m.p. 188°C (dec.). *Anal.* Calc. for $C_{41}H_{39}ClO_4P_2PdPtS_2$: C, 46.5; H, 3.7; S, 6.1. Found: C, 46.8; H, 3.8; S, 5.8%. Λ_M 135 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.7–7.3 (m, 20H, aromatics dppe), 6.96 (d, 4H, H_o of SPh, $J_{om} = 7.3$ Hz), 6.81 (t, 2H, H_p of SPh, $J_{mp} = 7.3$), 6.67 (pseudo-t, 4H, H_m of SPh), 5.52 (m, 1H, CC*H*C allyl), 3.76 (d, 2H, H_{syn}, $J = 6.9$), 3.23 (d, 2H, H_{anti}, $J = 12.6$), 2.25 (d, 4H, CH_2 of dppe, $J_{HP} = 19.8$). ^{31}P NMR ($CDCl_3$): δ 45.6 (s, $J_{PtP} = 3108$ Hz).

Complex **6**: Yield 73%; m.p. 194°C (dec.). *Anal.* Calc. for $C_{43}H_{43}ClO_4P_2PdPtS_2$: C, 47.5; H, 4.0; S, 6.0. Found: C, 47.1; H, 4.0; S, 5.8%. Λ_M 136 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.7–7.3 (m, 20H, aromatics dppe), 6.88 (d, 4H, H_o of SC_6H_4Me, $J_{om} = 7.8$ Hz), 6.49 (d, 4H, H_m of SC_6H_4Me, $J_{om} = 7.8$), 5.57 (m, 1H, CC*H*C allyl), 3.81 (d, 2H, H_{syn}, $J = 6.9$), 3.26 (d, 2H, H_{anti}, $J = 12.6$), 2.28 (d, 4H, CH_2 dppe, $J_{HP} = 19.1$), 2.14 (s, 6H, Me). ^{31}P NMR ($CDCl_3$): δ 45.2 (s, $J_{PtP} = 3110$ Hz).

Complex **7**: Yield 74%; m.p. 191°C (dec.). *Anal.* Calc. for $C_{33}H_{39}ClO_4P_2PdPtS_2$: C, 41.2; H, 4.1; S, 6.7. Found: C, 41.3; H, 4.0; S, 7.1%. Λ_M 131 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 8.0–7.5 (m, 20H, aromatics dppe), 5.47 (m, 1H, CC*H*C allyl), 4.01 (d, 2H, H_{syn}, $J = 6.7$ Hz), 3.05 (d, 2H, H_{anti}, $J = 12.5$), 2.50 (d, 4H, CH_2 dppe, $J_{HP} = 19.7$), 2.17 (m, 4H, CH_2S), 0.89 (t, 6H, CH_3 CH_2S, $J = 6.6$). ^{31}P NMR ($CDCl_3$): δ 46.8 (s, $J_{PtP} = 3055$ Hz).

Complex **8**: Yield 57%; m.p. 168°C (dec.). *Anal.* Calc. for $C_{42}H_{41}ClO_4P_2PdPtS_2$: C, 47.0; H, 3.9; S, 6.0. Found: C, 46.7; H, 3.7; S, 5.8%. Λ_M 133 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.8–7.2 (m, 20H, aromatics dppe), 6.94 (d, 4H, H_o of SPh, $J_{om} = 7.3$ Hz), 6.83 (t, 2H, H_p of SPh, $J_{mp} = 7.3$), 6.68 (pseudo-t, 4H, H_m of SPh), 3.59 (s, 2H, H_{syn}), 3.12 (s, 2H, H_{anti}), 2.33 (d, 4H, CH_2 dppe, $J_{HP} = 18.9$), 1.96 (s, 3H, CH_3 allyl). ^{31}P NMR ($CDCl_3$): δ 45.9 (s, $J_{PtP} = 3105$ Hz).

Complex **9**: Yield 67%; m.p. 153°C (dec.). *Anal.* Calc. for $C_{34}H_{41}ClO_4P_2PdPtS_2$: C, 41.8; H, 4.2; S, 6.6. Found: C, 41.6; H, 4.2; S, 6.4%. Λ_M 145 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 8.0–7.3 (m, 20H, aromatics dppe), 3.71 (s, 2H, H_{syn}), 2.89 (s, 2H, H_{anti}), 2.49 (d, 4H, CH_2 dppe, $J_{HP} = 20.4$ Hz), 2.12 (m, 4H, CH_2S), 1.99 (s, 3H, CH_3 allyl), 0.87 (t, 6H, CH_3 CH_2S, $J = 7.2$). ^{31}P NMR ($CDCl_3$): δ 47.1 (s, $J_{PtP} = 3056$ Hz).

Complex **10**: Yield 46%; m.p. 170°C (dec.). *Anal.* Calc. for $C_{31}H_{33}ClO_4P_2PdPtS_2$: C, 39.9; H, 3.6; S, 6.9. Found: C, 39.3; H, 3.6; S, 6.8%. Λ_M 120 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.9–7.3 (br m, 20H, aromatics dppe), 5.1 (br, 1H, CC*H*C allyl), 4.08 (br, 2H, H_{syn}),

Table 1
Crystal data and structure refinement for **2**

Empirical formula	$C_{43}H_{43}ClO_4P_2Pd_2S_2$
Formula weight	998.08
Temperature (K)	294(2)
Wavelength (Mo Kα) (Å)	0.71073
Crystal system	triclinic
Space group	$P\bar{1}$ (# 2)
Unit cell dimensions	
a (Å)	10.873(1)
b (Å)	11.698(1)
c (Å)	17.353(2)
α (°)	84.733(9)
β (°)	88.052(8)
γ (°)	77.284(8)
Volume (Å^3)	2143.7(4)
Z	2
Density (calculated) (Mg m^{-3})	1.546
Absorption coefficient (mm^{-1})	1.113
$F(000)$	1008
θ Range for data collection (°)	2.23–30.42
Index ranges	$0 \le h \le 15$, $-16 \le k \le 16$, $-24 \le l \le 24$
Reflections collected	13 602
Independent reflections	12 973 ($R_{int} = 0.0127$)
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	12 973/1/468
Goodness-of-fit on F^2	1.047
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0383$, $wR_2 = 0.1184$
R indices (all data)	$R_1 = 0.0580$, $wR_2 = 0.1306$
Largest difference peak and hole (e Å^{-3})	1.543 and −1.058

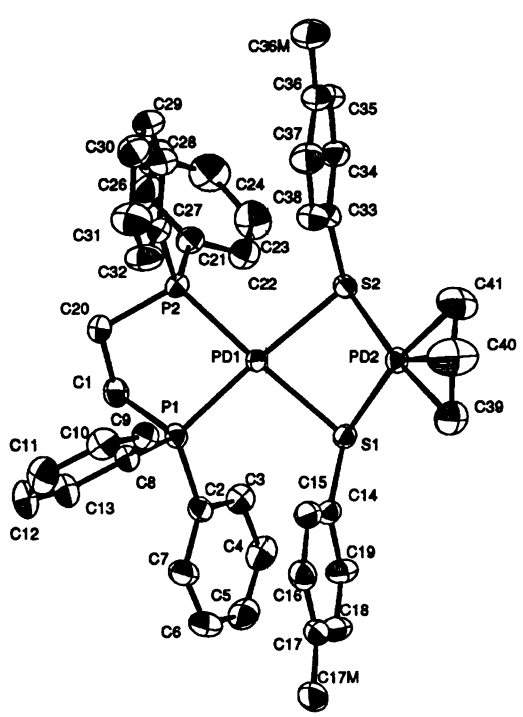
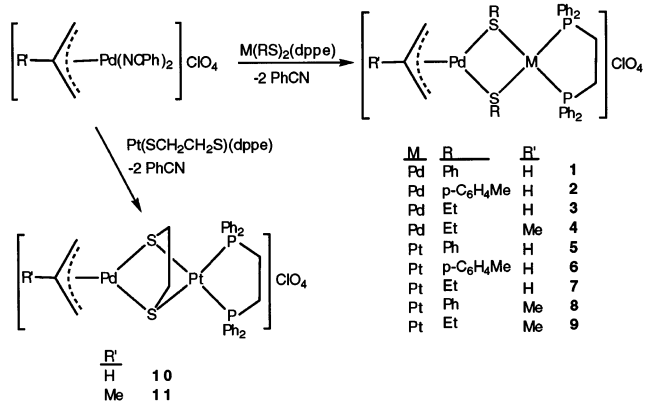


Fig. 1. Perspective view of the molecular structure of **2**.



Scheme 1.

3.0–2.5 (br m, 10H, $H_{anti} + CH_2$ dppe + SCH_2CH_2S). ^{31}P NMR ($CDCl_3$): δ 46.3 (s, $J_{PtP} = 2748$ Hz).

Complex **11**: Yield 45%; m.p. 125°C (dec.). *Anal.* Calc. for $C_{32}H_{35}ClO_4P_2PdPtS_2$: C, 40.6; H, 3.7; S, 6.8. Found: C, 40.3; H, 3.7; S, 6.6%. Λ_M 135 S cm^2 mol^{-1}. ^1H NMR ($CDCl_3$): δ 7.8–7.3 (m, 20H, aromatics dppe), 3.81 (br, 2H, H_{syn}), 3.0–2.5 (br m, 10H, $H_{anti} + CH_2$ dppe + SCH_2CH_2S), 1.80 (br, 3H, CH_3 allyl). ^{31}P NMR ($CDCl_3$): δ 45.2 (s, $J_{PtP} = 3050$ Hz).

2.2. Determination of the X-ray crystal structure of complex **2**

A single crystal of complex **2** (approximate dimensions 0.55 × 0.37 × 0.24 mm) was mounted on an Enraf–Nonius CAD4 diffractometer equipped with a graphite monochromator for Mo Kα radiation. The crystallographic data are shown in Table 1 (Fig. 1). Accurate cell parameters were determined by least-squares fitting of 25 high-angle reflections. The scan method was ω–2Θ and the empirical Psi scan mode absorption correction was applied. The structure was solved by heavy atom methods SHELXS-86 [15] and refined anisotropically on F^2 [16]. Hydrogen atoms were introduced in calculated positions. The residual peaks in the final Fourier difference synthesis were located close to the metal atoms.

3. Results and discussion

The mononuclear palladium and platinum complexes [M(SR)$_2$ (dppe)] (M = Pd or Pt) or [Pt(SC$_2$H$_4$S)(dppe)] [dppe = 1,2-bis(diphenylphosphino)ethane] react with [Pd(η^3-allyl)(PhCN)$_2$][ClO$_4$] (allyl = C$_3$H$_5$ or C$_4$H$_7$) in a 1:1 molar ratio to give heterodinuclear complexes of the types [(dppe)M(μ-SR)$_2$Pd(η^3-allyl)][ClO$_4$] (M = Pd or Pt) (**1**–**9**) and [(dppe)Pt(μ-SC$_2$H$_4$S)Pd(η^3-allyl)][ClO$_4$] (**10** and **11**) with the concomitant release of PhCN (Scheme 1).

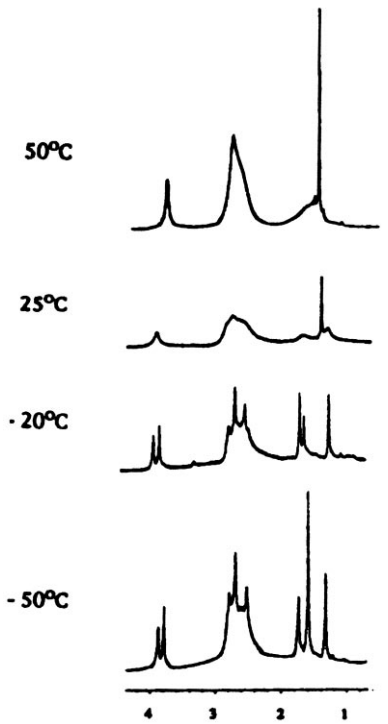


Fig. 2. Variable-temperature ^{1}H NMR spectrum of **11**.

The new complexes **1**–**11** have been characterized on the basis of partial elemental analyses and spectroscopic data and they behave in acetone as 1:1 electrolytes [17], in accordance with the formulas given.

The ^{31}P NMR spectra show a single resonance and those of the platinum complexes are flanked by the satellites due to coupling to ^{195}Pt, the coupling constant being in the range 2700–3100 Hz.

The ^{1}H NMR spectra of complexes **1**–**9** show a single set of resonances, indicating that both thiolate ligands are equivalent and the free rotation of the thiolate aryl or alkyl group around the C–S bond. The ^{1}H NMR spectra of complexes **1**–**3** and **5**–**7** show also three resonances with the expected intensity ratio (1:2:2) and peak multiplicities in the allylic group region (multiplet for CC*H*C, doublet for *syn* protons, and doublet for *anti* protons) whereas the spectra of complexes **4** and **8**–**9** show three singlet resonances for the methylallyl group with the expected intensity ratio of 2:2:3.

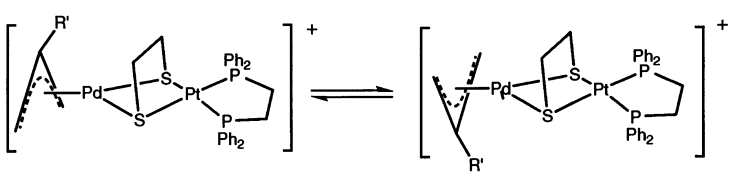
However, the ^{1}H NMR spectra of complexes **10** and **11** show broad bands at room temperature, which suggests a fluxional behavior in the ^{1}H NMR scale of time. Rotation of the η^{3}-allyl group has been suggested as a possible dynamic process [18]. The ^{1}H NMR spectrum of complex **11** at -50°C (Fig. 2) shows two different singlets at δ 3.91 and 3.83 assigned to *syn* protons and two additional singlets at δ 1.86 and 1.80 due to the Me protons of the allyl groups of the two conformational isomers tentatively shown in Scheme 2 (signals due to the *anti* protons are obscured by resonances of dppe and SC_2H_4S ligands).

Table 2
Selected distances (Å) and bond angles (°) for complex **2**

Bond distances		Bond angles	
Pd(1)–P(1)	2.2777(8)	P(1)–Pd(1)–P(2)	85.34(3)
Pd(1)–P(2)	2.2871(8)	P(1)–Pd(1)–S(1)	96.87(3)
Pd(1)–S(1)	2.3742(8)	P(2)–Pd(1)–S(1)	172.13(3)
Pd(1)–S(2)	2.3783(8)	P(1)–Pd(1)–S(2)	172.45(3)
Pd(1)···Pd(2)	3.2428(4)	P(2)–Pd(1)–S(2)	95.85(3)
Pd(2)–C(40)	2.085(5)	S(1)–Pd(1)–S(2)	80.98(3)
Pd(2)–C(39)	2.137(4)	Pd(2)–S(1)–Pd(1)	86.77(3)
Pd(2)–C(41)	2.140(4)	Pd(2)–S(2)–Pd(1)	86.54(3)
Pd(2)–S(1)	2.3465(8)	S(1)–Pd(2)–S(2)	82.10(3)
Pd(2)–S(2)	2.3524(9)	C(40)–Pd(2)–S(1)	140.5(2)
		C(39)–Pd(2)–S(1)	105.58(14)
		C(41)–Pd(2)–S(1)	171.3(2)
		C(40)–Pd(2)–S(2)	137.1(2)
		C(39)–Pd(2)–S(2)	172.24(14)
		C(41)–Pd(2)–S(2)	105.1(2)
		C(41)–C(40)–C(39)	142.4(8)

Suitable crystals of complex **2** were grown from acetone–ethanol. The structure of **2** is shown in Fig. 1 and selected lengths and angles in Table 2. Both metal atoms, Pd(1) and Pd(2), are coordinated in a slightly distorted square-planar geometry and the phenyl groups on the sulfur atoms adopt a *syn* arrangement with respect to the central Pd–S_2–Pd core. The same conformation has been recently found in $[(C_6F_5)_2Ni(\mu\text{-}SPh)_2Pd(dppe)]$ [19], $[\eta^5\text{-}C_5H_4SiMe_3)_2Ti(\mu\text{-}SPh)_2Pd(C_6F_5)_2]$ [20] and $[\eta^5\text{-}C_5H_5)Ti(\mu\text{-}SMe)_2PtMe_2]$ [21].

The Pd(1)–S distances are 2.3742(3) and 2.3783(8) Å, significantly longer than Pd(2)–S distances, 2.3465(8) and 2.3524(9) Å. This is due to the strong *trans* influence of the phosphine ligand [19]. The Pd···Pd distance is 3.2428(4) Å, showing no significant metal–metal interaction. The PdSPdS ring adopts a hinged square-planar geometry with a dihedral angle of 129.9° along



Scheme 2.

the S···S line. As expected, the PdP_2C_2 chelate ring is non-planar. The Pd-η^3-allyl distances are in the range expected for a Pd(II)-allyl.

4. Supplementary material

Supplementary data for complex **2** are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number CCDC 139771. 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 www: http://www.ccdc.cam.ac.uk).

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