

Mono-dmit Phosphine Complexes: A Layered Architecture for the Crystal Structure of the Neutral Coordination Polymer $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$

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Mono-dmit complexes, $\text{M}(\text{C}_3\text{S}_5)(\text{phos})$, $\text{M} = \text{Hg}, \text{Pt}$ or Pd , $\text{C}_3\text{S}_5^{2-} = 4,5$ -disulfanyl-1,3-dithiole-2-thionate(2-) (commonly known as 'dmit'), $\text{phos} = 1,2$ -bis(diphenylphosphino)ethane (dppe), 1,1'-bis(diphenylphosphino)ferrocene (dppf), 2 × triphenylphosphine (PPh_3), have been prepared by deprotection of the bis-cyanoethyl-protected sulfur atoms in 4,5-bis(2'-cyanoethylthio)-1,3-dithiole-2-thione in the presence of metal ions in a one-pot reaction. The crystal structure of a mercury–dmit–dppe complex gave the unexpected result of a one-dimensional coordination polymer with a framework architecture resulting from the bidentate phosphine ligands bridging between the metal centres and intermolecular $\text{S} \cdots \text{S}$ interactions. The preference of Hg for a tetrahedral coordination geometry is probably instrumental for the formation of the unusual polymeric structure; in the case of the Pt and Pd, the complexes are monomeric. The metal ion's requirement of a square-planar geometry in the case of Pd and Pt presumably prevents formation of similar polymeric structures.

Metal complexes of sulfur-containing ligands have received considerable attention because of their applications in analytical chemistry, new materials, catalysis and their relevance to bioinorganic systems.¹ 4,5-Disulfanyl-1,3-dithiole-2-thionate ($\text{C}_3\text{S}_5^{2-}$ or dmit^{2-}) is a well known sulfur-containing ligand and has been used for the preparation of many mononuclear bis-dmit and tris-dmit complexes of transition and main-group metals.² There are, however, few reports of metal complexes containing only one dmit ligand.³ We have been working on strategies for the preparation of complexes with 1:1 metal:dmit stoichiometry, and which circumvent possible $\text{M}(\text{dmit})_{2/3}$ formation. Described here is a new method for the preparation of mixed ligand monomeric dmit complexes. Unexpectedly, a polymeric structural arrangement was established by single-crystal X-ray crystallography for one of the mercury complexes.

Experimental

IR spectra were measured as KBr discs using a Hitachi 270–30 IR spectrometer. UV/VIS absorption spectra of the complexes were recorded on a Shimadzu UV-3100 spectrophotometer. Fast atom bombardment mass spec-

tra (FABMS) were recorded on a KRATOS MS50TC. Solution NMR spectra were recorded on a Bruker AC 250. Microanalyses were performed at the Microanalytical Laboratory, University of Copenhagen. All reagents were of commercial origin (e.g. Aldrich) and used without further purification. 4,5-Bis(2'-cyanoethylthio)-1,3-dithiole-2-thione,⁴ $\text{M}(\text{phos})\text{Cl}_2$ [$\text{M} = \text{Pd}, \text{Pt}, \text{Hg}$, $\text{phos} = \text{dppe}, (\text{PPh}_3)_2$ and dppf]⁵ were prepared using literature methods. The complexes $\text{Pd}(\text{C}_3\text{S}_5)(\text{dppe})$ have been prepared previously by the alternative route of heating to reflux the metal phosphine–halide complex with the sodium dmit salt in methanol for several hours.^{3a,b}

Syntheses.

[1,1' - Bis(diphenylphosphino)ferrocene]mercury(4,5 - disulfanyl - 1,3-dithiole - 2-thionate), $\text{Hg}(\text{C}_3\text{S}_5)(\text{dppf}) \cdot \text{CH}_2\text{Cl}_2$. 4,5-Bis(2'-cyanoethylthio)-1,3-dithiole-2-thione (40 mg, 0.13 mmol) in 15 cm³ dichloromethane was added to a stirred solution of $\text{Hg}(\text{dppf})\text{Cl}_2$ (108 mg, 0.13 mmol) in 20 cm³ methanol, under dinitrogen. Stirring was continued for 15 min, after which time 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 40 mg, 0.26 mmol) in 5 cm³ dichloromethane was slowly added. Stirring was continued for a further 30 min. The solution

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was then left to stand. Over several hours, the product was deposited as a red microcrystalline solid (58% yield). Anal. Calcd. for $C_{38}H_{30}Cl_2FeHgP_2S_5$: C, 44.05; H, 2.92. Found: C, 43.77; H, 2.87%. FABMS: m/z 952 $\{[Hg(C_3S_5)(dppf)]^+\}$, 15%. 1H NMR ($CDCl_3$): δ 4.27 (m, 2 H, cpH), 4.19 (m, cpH).

The following complexes were prepared using an analogous procedure and the appropriate $M(\text{phos})Cl_2$ complex as a starting material.

catena- μ -[1,2-Bis(diphenylphosphino)ethane]mercury(4,5-disulfanyl-1,3-dithiole-2-thionate), $[Hg(C_3S_5)(dppe)]_n$. Orange block-like crystals of the product, along with small amounts of an, as yet unidentified, black solid precipitated from the reaction (yield, 65 mg total product, from 0.13 mmol of both starting materials). IR, NMR, UV-VIS and mass spectra were obtained on samples from which the black solid was separated manually. UV/VIS (MeCN) $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 459 (370). Solid-state MAS NMR data : $\delta(^{31}P)$ 30.5, 19.8 ppm. Results from elemental analysis are unacceptable for the formulation found in the crystal structure, presumably due to the contamination by the black material. The crystal used for X-ray structural analysis was hand picked. Manually sorted samples were used for the powder diffraction⁶ and conductivity studies. A sample of the partly separated black material gave an elemental analysis of C, 29.26 and H, 2.06%. This analysis lends support to a compound with higher dmit content for the black impurity than that for $[Hg(C_3S_5)(dppe)]_n$ (i.e. a bis- or tris-dmit complex).

[1,1'-Bis(diphenylphosphino)ferrocene]palladium(4,5-disulfanyl-1,3-dithiole-2-thionate), $Pd(C_3S_5)(dppf)$. Brown, microcrystalline solid (yield 52%). Anal. Calcd. for $C_{37}H_{28}FeP_2PdS_5$: C, 44.36; H, 2.82. Found: C, 45.20; H, 3.23. FABMS: m/z 856 $\{[Pd(C_3S_5)(dppf)]^+\}$, 20%. UV-VIS (DMSO) $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 465 (530). 1H NMR ($CDCl_3$): δ 2.50 (s, 2 H, CH_2), 2.59 (s, 2 H, CH_2), 7.50 (m, 12 H, ArH), 7.73 (m, 8 H, ArH). Solid-state MAS NMR data : $\delta(^{31}P)$ 27.8, 22.8 ppm.

[1,2-bis(diphenylphosphino)ethane]palladium(4,5-disulfanyl-1,3-dithiole-2-thionate), $Pd(C_3S_5)(dppe)$. Brown amorphous solid (yield, 55%). Anal. Calcd. for $C_{29}H_{24}P_2PdS_5$: C, 49.68; H, 3.45. Found: C, 49.75; H, 3.94. FABMS: m/z 700 $\{[Pd(C_2S)(dppe)]^+\}$, 20%. UV-VIS (DMSO) $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 478 (8010).

Bis(triphenylphosphino)palladium(4,5-disulfanyl-1,3-dithiole-2-thionate), $Pd(C_3S_5)(PPh_3)_2$. Brown microcrystalline solid (yield 65%). Anal. Calcd. for $C_{39}H_{30}P_2PdS_5$: C, 56.62; H, 3.65. Found: C, 55.53; H, 3.73. FABMS: m/z 826 $\{[Pd(C_3S_5)(PPh_3)_2]^+\}$, 25%. UV-VIS (DMSO) $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 482 (8400). 1H NMR ($CDCl_3$): δ 7.58 (m, 12 H, ArH), 7.73 (m, 8 H, ArH).

[1,2-Bis(diphenylphosphino)ethane]platinum(4,5-disulfanyl-1,3-dithiole-2-thionate), $Pt(C_3S_5)(dppe)$. Brown crystalline solid (yield 63%). Anal. Calcd. for

$C_{29}H_{24}P_2PtS_5$: C, 44.10; H, 3.06. Found: C, 44.21; H, 3.31. FABMS: m/z 790 $\{[Pt(C_3S_5)(dppe)]^+\}$, 20%. UV-VIS (DMSO) $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$): 472 (7415). 1H NMR ($CDCl_3$): δ 2.49 (s, 2 H, CH_2), 2.57 (s, 2 H, CH_2), 7.52 (m, 12 H, ArH), 7.73 (m, 8 H, ArH). Solid-state MAS NMR data : $\delta(^{31}P)$ 27.2, 24.2 ppm.

Solid-state MAS NMR. ^{31}P magic angle spinning NMR spectra were recorded on a Varian Unity-500 spectrometer [11.7 T, $\nu(^{31}P) = 202.3$ MHz], using a Jakobsen MAS probe.⁷ The FIDs were recorded with a spinning speed of 10.5 kHz, and the resulting spectra had a digital resolution of 2.5 Hz. An aqueous solution of H_3PO_4 (85%, Fluka p.a.) was used as external standard.

Conductivity measurements. The solid-state conductivity was measured on powdered samples pressed (12 kN) to rods with approximate dimensions of $(1.0-1.7) \times 2.1 \times 13.1$ mm³. A gold layer, as the electrode, was evaporated onto each end of the rods.⁸ The rods were mounted in a measuring cell between indium wire (2 mm diameter). AC impedance measurements were carried out in the frequency range 5 Hz–13 MHz at 22 °C using a Hewlett-Packard 4192A LF impedance analyzer.

Single-crystal diffraction. Crystal data are summarised in Table 1. Data were collected at low temperature on a Siemens P4 four-circle diffractometer and crystal stability was monitored by recording three check reflections every 97 reflections and no significant variation was observed. Data were corrected for Lorentz and polarisation effects and semi-empirical absorption corrections were applied based on ψ -scan data. The structure was solved by direct methods,⁹ which revealed most of the non-hydrogen atoms. The remaining atoms were located from difference Fourier maps. All the full-occupancy non-hydrogen atoms were refined with anisotropic atomic displacement parameters, and hydrogen atoms were inserted at calculated positions with isotropic temperature factors riding on those of their carrier atoms. All the data were used for refinement on F^2 . Residual electron density in the final difference maps was located close to the Hg. All programs used in the structure refinement are contained in the SHELXL-93¹⁰ package. Additional material available from the Cambridge Crystallographic Data Centre comprises atomic coordinates and all bond distances and angles.

Results and discussion

Scheme 1 shows the synthetic approach used to obtain the present series of mixed-ligand, $M(C_3S_5)(\text{phos})$, complexes. The reaction of the cyanoethyl-protected dmit with a metal phosphine-halide complex and base in methanol-dichloromethane is complete after about 1 h. The deprotection of the dmit thiolate groups is confirmed by the disappearance of the IR band due to ν_{CN} at ca. 2250 cm^{-1} . All the materials obtained are relatively

Table 1. Crystal data for $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$.

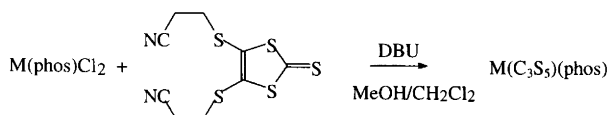
| | |
|---|--|
| Empirical formula | $\text{C}_{25}\text{H}_{24}\text{HgP}_2\text{S}_5$ |
| Formula weight | 795.31 |
| Crystal description | Orange block |
| Temperature/K | 153(2) |
| Wavelength/Å | 0.71073 |
| Crystal system | Monoclinic |
| Space group | $P2_1/n$ |
| $a/\text{Å}$ | 13.392(2) |
| $b/\text{Å}$ | 14.283(1) |
| $c/\text{Å}$ | 15.910(2) |
| $\beta/^\circ$ | 104.43(1) |
| $V/\text{Å}^3$ | 2947.2(6) |
| Z | 4 |
| Density calculated/ Mg m^{-3} | 1.792 |
| μ/mm^{-1} | 5.704 |
| $F(000)$ | 1552 |
| Crystal size/mm | $0.25 \times 0.22 \times 0.10$ |
| Range for data collection/ $^\circ$ | $2.0 < \theta < 25.00$ |
| Index ranges | $-12 < h < 15$ $-4 < k < 16$ $-18 < l < 18$ |
| Reflections collected | 5880 |
| Independent reflections | 5178 ($R_{\text{int}}=0.0471$) |
| Absorption correction | ψ -Scans |
| Max. and min. transmission | 0.902, 0.543 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data/restraints/parameters | 5176/0/334 |
| Goodness-of-fit on F^2 (all data) | 1.040 |
| Final R -indices [$I > 2\sigma(I)$] | $R1=0.0496$ $wR2=0.1061$ |
| R -Indices (all data) | $R1=0.0876$ $wR2=0.1069$ |
| Largest diff. peak and hole/ $e \text{ Å}^{-3}$ | 1.720 and -0.708 |

$$R1 = \frac{\sum \|F_o\| - |F_c|}{\sum F_o}$$

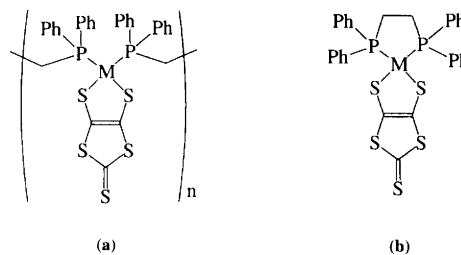
$$wR2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$$

$$w = \frac{q}{[\sigma^2(F_o^2) + (aP)^2 + bP + e \sin \theta]}, P = [f \times \text{maximum of } (0 \text{ or } F_o^2) + (1 + f)F_c^2]$$

insoluble in common organic solvents. Saturated solutions were used to obtain the UV-VIS and solution NMR data listed in the Experimental section. The electronic absorption spectra are dominated by an intense band centred at ca. 470 nm, assigned to a $\text{RS}^- \rightarrow \text{M}$ ligand-to-metal charge-transfer transition. The monomeric compounds $\text{Pd}(\text{C}_3\text{S}_5)(\text{dppe})$ and $\text{Pt}(\text{C}_3\text{S}_5)(\text{dppe})$ have been previously prepared by an alternative synthetic method.^{3a,b}

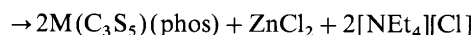
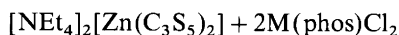
Scheme 1. $\text{M} = \text{Pd}^{2+}, \text{Pt}^{2+}, \text{Hg}^{2+}$; phos = dppe, dppf, $(\text{PPh}_3)_2$.

A novel result of this work was the formation of a one-dimensional coordination polymer for the mercury complex, $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$, for which the crystal structure is described below (Fig. 1). The evidence suggests that the remainder of the complexes are, as expected, monomeric (Fig. 1). The preparations of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ also gave an unidentified black prod-

Fig. 1. The polymeric and monomeric structures formed when (a) $\text{M} = \text{Hg}$ and (b) $\text{M} = \text{Pd}, \text{Pt}$ in complexes of formulation $\text{M}(\text{C}_3\text{S}_5)(\text{dppe})$.

uct which had to be separated manually from the crystals of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$. The powder diffraction pattern was measured for both the orange $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ and the black product.⁶ These diffraction patterns showed that the bulk orange material gives the calculated pattern expected for the polymeric structure determined by the single-crystal experiment. The powder pattern for the black impurity was derived.⁶ One possibility for the composition of this black material, which is consistent with its dark colour, is the formation of a bis- or tris-dmit complex. However, we have failed to identify peaks appropriate for such species in negative ion FAB mass spectra.

We have also discovered that the $\text{M}(\text{C}_3\text{S}_5)(\text{dppe})$ complexes can be prepared by the metathesis reaction:



The utilization of this metathesis reaction was not pursued, since it seems that the products obtained are either less crystalline or amorphous (checked by powder diffraction).⁶ For example, in our hands, the preparation of single crystals of the polymeric $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ was impossible using this metathesis reaction. This may imply that the controlled hydrolysis of the protecting cyanoethyl groups of 4,5-bis(2'-cyanoethylthio)-1,3-dithiole-2-thione, by base in the presence of metal ions, gives a better opportunity for obtaining pure crystalline materials for the mono-dmit complexes compared with the rapid precipitation of products offered by the metathesis reaction.

In the FAB mass spectra observation of a molecular ion peak, $[\text{M}(\text{C}_3\text{S}_5)(\text{phos})]^+$, seems to indicate a monomeric species, while its absence in the case of the $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$, although not conclusive, supports the structural formulation of a polymeric species. The molecular ions are listed in the experimental section for $\text{Hg}(\text{C}_3\text{S}_5)(\text{dppf})$, $\text{Pd}(\text{C}_3\text{S}_5)(\text{dppe})$, $\text{Pd}(\text{C}_3\text{S}_5)(\text{dppf})$, $\text{Pd}(\text{C}_3\text{S}_5)(\text{PPh}_3)_2$ and $\text{Pt}(\text{C}_3\text{S}_5)(\text{dppe})$. For example, in the case of the monomeric complex $\text{Pt}(\text{C}_3\text{S}_5)(\text{dppe})$, a peak assigned to the molecular ion, $[\text{Pt}(\text{C}_3\text{S}_5)(\text{dppe})]^+$, is observed at m/z 790 while the corresponding peak {i.e. $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]^+$ expected at m/z 795} is absent from the spectrum of the polymeric Hg complex $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$.

Crystal structure of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$. Crystals of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ suitable for structural analysis were obtained as small orange blocks by the slow evaporation of the reaction solution. The X-ray structure reveals a unique one-dimensional coordination polymer containing mono-dmit subunits connected by the bidentate phosphine ligand. The asymmetric unit of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ is illustrated in Fig. 2 and selected bond distances and angles are listed in Table 2. The geometry about mercury is best described as a slightly distorted tetrahedron with the mercury bonded to the two thiolate sulfur atoms of the dmit group and two phosphorus atoms of two different dppe ligands. Other complexes in which diphosphine ligands bridge between two metal centres are known.¹¹

Figure 3 shows the layered structure with alternative dmit and phosphine layers, with a distance of 6.43 Å between the dmit layers. The dmit planes are inclined by 11° with respect to each other. The Hg atoms are 0.407 Å out of the dmit plane. The distance between the Hg atoms in the same chain is 7.24 Å, and the closest intermolecular Hg...Hg distance between the chains is 12.67 Å. The polymeric chains are associated by

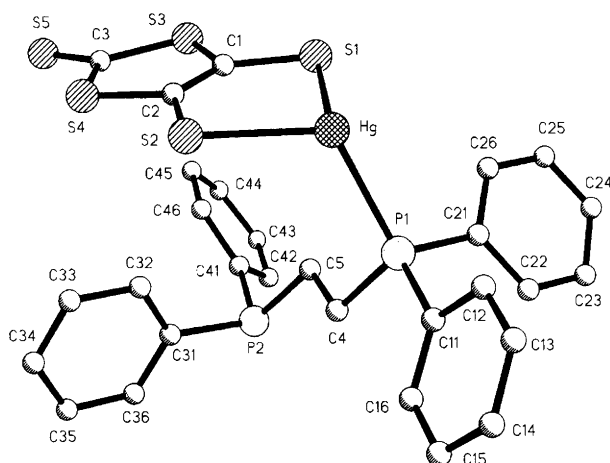


Fig. 2. The asymmetric unit of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ with atomic numbering scheme.

Table 2. Selected interatomic distances (in Å) and angles (in °) for $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$.

| | | | |
|----------------|-----------|----------------|-----------|
| Hg–P(1) | 2.508(3) | Hg–P(2)#1 | 2.513(3) |
| Hg–S(2) | 2.530(3) | Hg–S(1) | 2.560(3) |
| S(1)–C(1) | 1.731(10) | S(2)–C(2) | 1.754(10) |
| C(1)–C(2) | 1.372(13) | C(1)–S(3) | 1.751(9) |
| C(2)–S(4) | 1.758(9) | S(3)–C(3) | 1.738(10) |
| S(4)–C(3) | 1.718(10) | C(3)–S(5) | 1.647(9) |
| P(1)–C(11) | 1.817(12) | P(1)–C(21) | 1.810(12) |
| P(1)–C(4) | 1.840(10) | C(4)–C(5) | 1.510(12) |
| P(2)–C(31) | 1.820(12) | P(2)–C(41) | 1.796(11) |
| C(5)–P(2) | 1.821(9) | | |
| P(1)–Hg–P(2)#1 | 123.54(7) | P(1)–Hg–S(2) | 116.98(9) |
| P(2)#1–Hg–S(2) | 104.44(9) | P(1)–Hg–S(1) | 109.73(9) |
| P(2)#1–Hg–S(1) | 107.05(9) | S(2)–Hg–S(1) | 89.56(8) |
| C(4)–P(1)–Hg | 111.0(3) | C(5)–C(4)–P(1) | 113.1(7) |
| C(4)–C(5)–P(2) | 112.8(6) | C(5)–P(2)–Hg#2 | 110.2(3) |

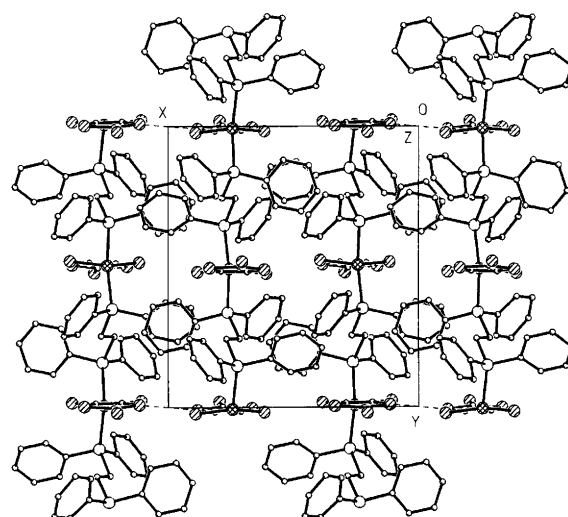


Fig. 3. View of the lattice structure of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ along the z-axis, showing its layered nature.

intermolecular S...S distances of 3.47 Å between S4 of one dmit ligand and S4a of the neighbouring dmit ligand (c.f. the sum of the van der Waals radii for two sulfur atoms is 3.6 Å).¹² Interactions between the sulfur atoms of neighbouring dmit ligands are well known in bis-dmit complexes. Usually, stacked zig-zag type structures are effected, as a result of metal–sulfur interactions, in contrast to the alternate sheets of dmit ligands with intervening phosphine layers in the present case. By inspection of the packing arrangement it can be seen the phenyl rings of the dppe ligand fit neatly in the space between the dmit layers. This fact, along with mercury's preference for a tetrahedral geometry, has probably been essential for the formation of this unusual framework structure.

Solid-state ^{31}P MAS NMR. Spectra were recorded for the *catena*-Hg complex and two of the monomeric complexes, $\text{Pt}(\text{C}_3\text{S}_5)(\text{dppe})$ and $\text{Pd}(\text{C}_3\text{S}_5)(\text{dppf})$. The spectra of all three complexes show two phosphorus signals (chemical shifts listed in the Experimental section) with a larger separation between the peaks in the spectrum of

the mercury complex (11 ppm) compared to the spectra of the palladium and platinum complexes (5 and 3 ppm). The observation of two signals is consistent with two phosphorus atoms in the asymmetric unit as found by the crystal structure. These peaks were too broad to allow resolution of P–P coupling. In the spectrum of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$, small peaks flanking the two phosphorus signals are observed. These signals are the doublets due to coupling of the ^{199}Hg nuclei (natural abundance 17%, $I=1/2$) with the phosphorus nuclei ($^1J_{\text{Hg-P}} \approx 2700$ Hz). The $J_{\text{Hg-P}}$ values and the chemical shifts of phosphorus fall within the range of values reported for other diphosphine mercury complexes.¹³

Conductivity measurements. Bis-dmit complexes have been reported to show interesting electrochemical and conductivity properties.¹⁴ These properties are usually associated with orbital overlap between stacks of dmit units, a feature absent in the structure of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$. $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ is the first reported example of a polymeric complex with dmit-containing subunits. This led us to speculation about its physical properties. The conductivity of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ was measured on pressed powdered samples. No conductivity within the limits of the instrumentation, i.e. $\sigma < 10^{-7} (\Omega \text{ cm})^{-1}$ was measurable.

Supplementary data. Atomic coordinates and anisotropic displacement parameters for $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ are available. A full listing of bond lengths and angles and hydrogen atom coordinates and isotropic displacement parameters. Indexed powder diffraction pattern of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$ and the observed powder diffraction pattern of a black impurity phase obtained in the preparation of $[\text{Hg}(\text{C}_3\text{S}_5)(\text{dppe})]_n$.

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