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Heteroleptic palladium(II) and platinum(II) complexes of 1,1-bis(diphenylphosphino)ferrocene (dppf) and heterocyclic thionates: Crystal structures of [Pt(Phozt)$_2$(κ^2-dppf)] (PhoztH = 5-phenyl-1,3,4-oxadiazole-2-thione) and [Pd(bzoxt)$_2$(κ^2-dppf)] (bzoxtH = benz-1,3-oxazoline-2-thione)

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ABSTRACT

Treatment of [MCl$_2$(κ^2-dppf)] (M = Pd or Pt) with two equivalents of potassium heterocyclic thionate salts (KL) affords mixed ligand complexes [ML$_2$(κ^2-dppf)] [L = 5-phenyl-1,3,4-oxadiazole-2-thionate (Phozt), 4,5-diphenyl-1,2,4-triazole-3-thionate (Ph$_2$tzt), benz-1,3-thiazoline-2-thionate (bztzt) and benz-1,3-oxazoline-2-thionate (bzoxt)]. X-ray structures of two examples, [Pt(Phozt)$_2$(κ^2-dppf)] and [Pd(bzoxt)$_2$(κ^2-dppf)], show that the ligands are coordinated in a monodentate fashion *via* the sulfur atom.

1. Introduction

The coordination chemistry of heterocyclic thiones and thionates is rich and varied with some examples having important chemical and biological properties [1–6]. Thionates can exist in two tautomeric forms which enables them to behave in an ambidentate fashion binding through either the sulfur (thiolate) or nitrogen (thione) atom, while in some cases they can act as chelate or bridging ligands upon binding of both of these atoms. These tautomers are shown (Chart 1) for the specific ligands used in this study which is part of our systematic investigation of the coordination chemistry of thioamide ligands [7–13]. Thus, we have previously shown that complexes of the type [ML$_2$(κ^2-Ph$_2$P(CH$_2$)$_n$PPh$_2$)] with mercapto-1,3-azole ligands, benz-1,3-thiazoline-2-thionate (X = S, bztzt) and benz-1,3-oxazoline-2-thionate (X = O, bzozt) exist as a mixture of isomers resulting from either the N- or S-coordination of the ligands [13]. Herein we report the synthesis and characterization of palladium(II) and platinum(II) complexes of heterocyclic thionates containing the flexible diphosphine, 1,1-bis(diphenylphosphino)ferrocene (dppf) [14–20]. Some related examples of platinum(II) with simple organic thiolates and dppf have been recently reported [21,22].

2. Experimental

2.1. General

^{1}H and ^{13}C NMR spectra were recorded on Varian Unity 500 and Gemini 2000 spectrometers, respectively, with CDCl$_3$ as solvent and internal reference. ^{31}P NMR spectra were recorded on a Gemini 2000 spectrometer with CDCl$_3$ as solvent and H$_3$PO$_4$ (85%) as external reference. The NMR spectra were measured at the Institut fur Anorganische Chemie, Martin-Luther-Universität, Halle-Wittenberg, Germany. IR spectra were recorded on a Shimadzu FT-IR 8400 spectrophotometer in the 200–4000 cm^{-1} range using CsI discs. Elemental analyses were carried out on a CHN analyzer type 1106 (Carlo-Erba). Melting points were measured on electrothermal 9100 melting point apparatus and were uncorrected. K$_2$[PtCl$_4$], 1,1-bis(diphenylphosphino)ferrocene (dppf) benz-1,3-oxazoline-2-thione, benz-1,3-thiazoline-2-thione were commercial products and were used as supplied. The compounds 4,5-diphenyl-1,2,4-triazole-3-thione [23], 5-phenyl-1,3,4-oxadiazole-2-thione [24], *trans*-[PdCl$_2$(DMSO)$_2$], *cis*-[PdCl$_2$(DMSO)$_2$] [25], [PdCl$_2$(κ^2-dppf)]

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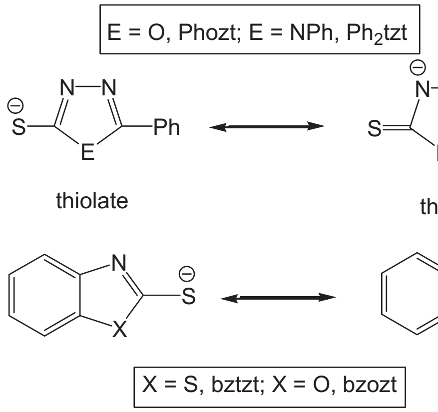


Chart 1.

and [$PtCl_2(\kappa^2$-dppf)] [20] were prepared by literature methods. The potassium salts of all ligands used in this work were prepared by mixing equimolar quantities of KOH and the ligand in EtOH. The mixture was stirred at room temperature for 1 h. Solvent was then evaporated on steam bath, and the resulting solid was dried under vacuum.

2.2. Preparation of complexes

All the complexes were prepared and isolated by the following general method. A solution of the appropriate potassium salt of the ligand in a minimum amount of EtOH was added to a solution of [$MCl_2(\kappa^2$-dppf)] (M = Pd or Pt) in a minimum a mount of $CHCl_3$ in a mole ratio ligand to metal 2:1. The resulting solution was boiled on steam bath for *ca.* 10 min. Solvent was then left to evaporate at room temperature. The resulting solid washed with water, cold EtOH and dried under vacuum. Crystals of **2**.EtOH and **6** suitable for single-crystal diffraction analysis were grown at 25 °C by slow evaporation of chloroform–ethanol solutions.

*Complex **1***: Orange solid, 86%. *Anal.* Calc. for $C_{50}H_{38}FeN_4O_2P_2PdS_2$: C, 58.9; H, 4.2; N, 5.3. Found: C, 59.0; H, 4.2; N, 5.5%. IR (KBr): 3470m, 3056m, 2925w, 1554m, 1305w, 1070m, 995m, 748s, 438m, 400w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.90–7.30 (m, 30H, Ph), 4.39 (m, 4H, Cp), 4.38 (m, 4H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 171.4, 163.6, 134.8, 131.7, 131.5, 127.9, 124.8, 76.3, 74.0, 73.4 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 31.7. Melting point: 210–213 °C.

*Complex **2***: Yellow solid, 79%. *Anal.* Calc. for $C_{50}H_{38}FeN_4O_2P_2PtS_2$: C, 54.4; H, 3.5; N, 5.1. Found: C, 54.2; H, 3.4; N, 5.0%. IR (KBr): 3057m, 2925w, 1554m, 1307m, 1070m, 995m, 755s, 441m, 400w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.87–7.30 (m, 30H, Ph), 4.36 (m, 8H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 171.4, 163.6, 134.9, 131.0, 130.7, 127.8, 124.8, 76.1, 73.3 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 18.4 (J_{Pt-P} 3298 Hz). Melting point: 247–250 °C.

*Complex **3***: Orange solid, 76%. *Anal.* Calc. for $C_{50}H_{38}FeN_6P_2PdS_2$: C, 63.9; H, 4.2; N, 7.2. Found: C, 64.1; H, 4.4; N, 7.5%. IR (KBr): 3058m, 2925w, 1596m, 1255w, 1065m, 1033m, 767s, 439m, 400w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.97–6.80 (m, 40H, Ph), 4.43 (m, 4H, Cp), 4.31 (m, 4H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 152.3, 135.1, 132.3, 130.5, 127.8, 76.0, 72.9 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 29.6. Melting point: 203–205 °C.

*Complex **4***: Red-brown solid, 88%. *Anal.* Calc. for $C_{46}H_{36}FeN_2P_2PdS_4$: C, 58.0; H, 3.7; N, 2.8. Found: C, 58.1; H, 3.8; N, 2.6%. IR (KBr): 3057m, 2925w, 1587m, 1238m, 1097m, 1030m, 750s, 438m, 380w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.97–7.00 (m, 28H, Ph), 4.42 (m, 4H, Cp), 4.35 (m, 4H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 176.5, 153.8, 137.3, 134.7, 132.1, 130.7, 127.7, 124.5, 121.9, 119.8, 119.0, 76.0, 74.8, 73.2 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 27.5. Melting point: 177–180 °C.

Table 1
Crystallographic and data collection parameters for complexes **2** and **6**.

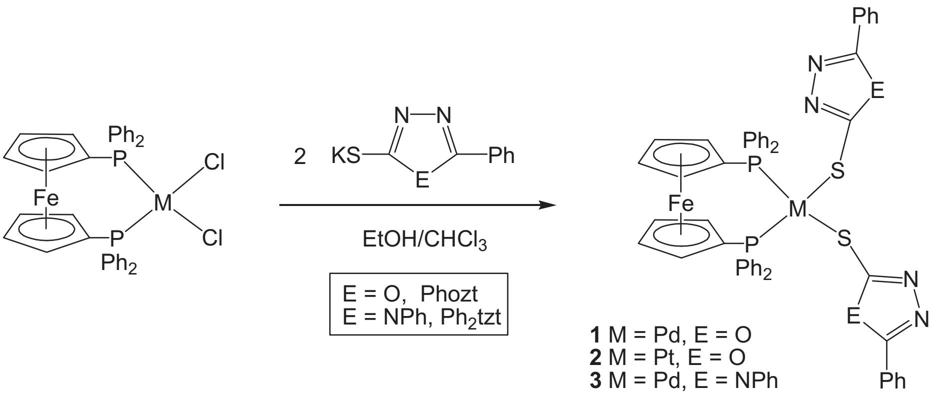
Complex	**2**.EtOH	**6**
Empirical formula	$C_{52}H_{44}FeN_4O_3P_2PtS_2$	$C_{48}H_{36}FeN_2O_2P_2PdS_2$
Formula weight	1149.91	961.10
Crystal system	triclinic	orthorhombic
T (K)	223(2)	223(2)
Space group	$P\bar{1}$	$Pna2_1$
Z	2	4
a (Å)	9.4541(9)	23.5022(14)
b (Å)	15.223(2)	18.4617(16)
c (Å)	18.236(1)	9.8761(6)
α (°)	76.71(1)	90
β (°)	78.13(1)	90
γ (°)	72.49(1)	90
V (Å^3)	2409.3(4)	4285.1(5)
ρ (g cm^{-3})	1.585	1.490
μ(Mo Kα) (mm^{-1})	3.401	0.966
F(000)	1148	1952
Scan range (°)	1.99 < 25.84	2.05 < θ < 25.88
Reciprocal lattice segments h, k, l	$-11 \to 11$ $-18 \to 18$ $-22 \to 22$	$-28 \to 28$ $-22 \to 22$ $-12 \to 11$
Reflections collected	18742	33063
Reflections independent	8655	8155
R_{int}	0.0428	0.0502
Data/restraints/parameters	8655/0/587	8155/1/523
Goodness-of-fit on F^2	0.972	0.985
R_1, wR_2 [$I > 2\sigma(I)$]	0.0305, 0.0645	0.0276, 0.0595
R_1, wR_2 (all data)	0.0496, 0.0778	0.0356, 0.0617
Largest difference in peak and hole (e Å^{-3})	1.28 and −1.39	1.052 and −0.250
Absolute structure factor		−0.015(14)
CCDC No.	767746	767747

*Complex **5***: Yellow solid, 73%. *Anal.* Calc. for $C_{46}H_{36}FeN_2P_2PtS_4$: C, 53.2; H, 3.4; N, 2.6. Found: C, 53.5; H, 3.2; N, 2.7%. IR (KBr): 3057m, 2925w, 1585m, 1240m, 1097m, 1031m, 752s, 437m, 380w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.85–6.95 (m, 28H, Ph), 4.30 (m, 8H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 134.6, 134.0, 131.3, 130.7, 127.6, 124.3, 121.8, 119.6, 75.8, 73.2 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 16.5 (J_{Pt-P} 3264 Hz). Melting point: 240–243 °C.

*Complex **6***: Red-brown solid, 91%. *Anal.* Calc. for $C_{46}H_{36}FeN_2P_2PdS_2O_2$: C, 60.0; H, 3.8; N, 2.9. Found: C, 60.1; H, 3.9; N, 2.0%. IR (KBr): 3055m, 2925w, 1604m, 1244m, 1081m, 1037m, 748s, 433m, 380w cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.98–7.10 (m, 28H, Ph), 4.44 (m, 4H, Cp), 4.37 (m, 4H, Cp). ^{13}C{^{1}H} NMR ($CDCl_3$): 151.6, 134.7, 131.3, 130.8, 122.5, 121.3, 115.6, 113.9, 108.4, 76.0, 73.2 ppm. ^{31}P{^{1}H} NMR ($CDCl_3$): δ 28.5. Melting point: 186–188 °C.

2.3. X-ray crystallography

Yellow crystals of **2** and red-brown crystals of **6** suitable for X-ray crystallographic measurements were obtained by slow evaporation of chloroform/ethanol (*ca.* 50:50) solutions of the respective complex. Table 1 gives the crystallographic data and collection parameters. Intensity data were collected on a STOE-IPDS diffractometer with Mo Kα radiation (λ = 0.7103 Å, graphite monochromator). Absorption corrections were made using the IPDS software package [26]. All structures were solved by direct methods with SHELX-97 [27] and refined using full-matrix least-square routines against F^2 with SHELXL-97 [28]. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were included in the models by calculating the positions (riding model) and refined with calculated isotropic displacement parameters. Illustrations were generated using DIAMOND 3.0 software [29].



Scheme 1.

3. Results and discussion

We have developed a three step synthesis of group 10 diphosphine–thionate complexes [$ML_2(\kappa^2$-diphosphine)] complexes [8–13]. This involves treatment of thiones LH (LH = Phozt and Ph_2tzt) with palladium(II) or platinum(II) precursors including Na_2PdCl_4, *trans*-[$PdCl_2(DMSO)_2$], K_2PtCl_4 or *cis*-[$PtCl_2(DMSO)_2$] to give complexes of the type [$MCl_2(LH)_2$] (M = Pd or Pt). These can then be converted to [ML_2] by treatment with base, which in turn react with diphosphines $Ph_2P(CH_2)_nPPh_2$ (n = 1–4) to give [$ML_2(\kappa^2$-diphosphine)] in which the thionate anion binds to the metal center in either the thiolate or thione form (Chart 1). Attempts to prepare [$ML_2(\kappa^2$-dppf)] complexes *via* this method were only partially successful leading to mixtures of products. While the desired complexes **1–3** were generated (as confirmed by ^1H NMR spectroscopy) we were unable to isolate pure products. The nature of the secondary species is not clear but they possibly result from the dppf ligand acting in a bridging rather than a chelating capacity since it is well-known that dppf is a highly flexible diphosphine [19]. We consequently sought another synthetic route to the desired products and in contrast to the first route, treatment of [$MCl_2(\kappa^2$-dppf)] (M = Pd, Pt) with two equivalents of the potassium salt of 5-phenyl-1,3,4-oxadiazole-2-thionate (KPhozt) [24] in a $CHCl_3$/EtOH mixture cleanly afforded [$M(Phozt)_2(\kappa^2$-dppf)] (**1–2**) after work-up as orange (M = Pd) and yellow (M = Pt) solids in 86% and 79% yield, respectively. A similar reaction between potassium 4,5-diphenyl-1,2,4-triazole-3-thionate (KPh_2tzt) [30] and [$PdCl_2(\kappa^2$-dppf)] gave [$Pd(Ph_2tzt)_2(\kappa^2$-dppf)] (**3**) as an orange solid in 76% yield (Scheme 1).

Characterization was relatively straight-forward on the basis of spectroscopic and analytical data. ^1H NMR spectra were as expected, each displaying multiplets between δ 6.80–7.98 assigned to the aromatic protons and either one or two broad signals between δ 4.31–4.44 due to the protons of the cyclopentadienyl ligands. The broadness of these later signals is attributed to the well-known fluxionality of the dppf ligand [19,31]. In the $^{31}P\{^1H\}$ NMR spectrum each shows a single phosphorus resonance indicating that both of the chlorides had been replaced and all exist in a single isomeric form. The relatively small phosphorus–platinum coupling constant for [$Pt(Phozt)_2(\kappa^2$-dppf)] (**2**) of 3298 Hz, respectively, as compared to [$PtCl_2(\kappa^2$-dppf)] (J_{Pt-P} 3761.5 Hz) [20] suggests the ligands bind through the sulfur atom, that is in a thiolate fashion [32]. For comparison the phosphorus–platinum coupling constant in the closely related complex [$Pt(SPh)_2(\kappa^2$-dppf)] is 3016 Hz [21]. In order to confirm this, an X-ray crystal structure was carried out on **2** the results of which are shown in Fig. 1. There is also a molecule of ethanol in the asymmetric unit but there are no significant intermolecular interactions and this will not be considered further. Compound **2** consists of the expected PtS_2P_2 square-planar array as is seen in related complexes [21,32,33]. The dppf ligand subtends a bite-angle of 98.08(5)° and the ferrocene backbone is twisted out of the PtS_2P_2 plane with one cyclopentadienyl ligand lying above and the second below. The key feature is the binding of the thionate groups through sulfur. The adoption of the thiolate resonance form is clearly seen from the metal–sulfur distances ($Pt–S_{av.}$ 2.388(2) Å) which are similar to those found in other platinum thiolate and dithiolate complexes [21,32,33] and the carbon–nitrogen bond lengths within the heterocyclic ring ($C–N_{av}$ 1.31(1) Å) which are indicative of double bond character. The two thiolate ligands lie relatively close to one another [S(1)–Pt–S(2) 85.25(4)°] and in order to reduce steric strain between them, substituents lie on opposite sides of the PtS_2P_2 plane (*anti*-conformation). The bond angles at sulfur of 99.8(2)° and 99.9(2)° are significantly smaller than those seen in [$Pt(SPh)_2(\kappa^2$-dppf)] [Pt–S–C 107.8(2)° and 109.7(2)°] and [$Pt(SPr)_2(\kappa^2$-dppf)] [Pt–S–C 105.2(4)° and 107.4(3)°] [21].

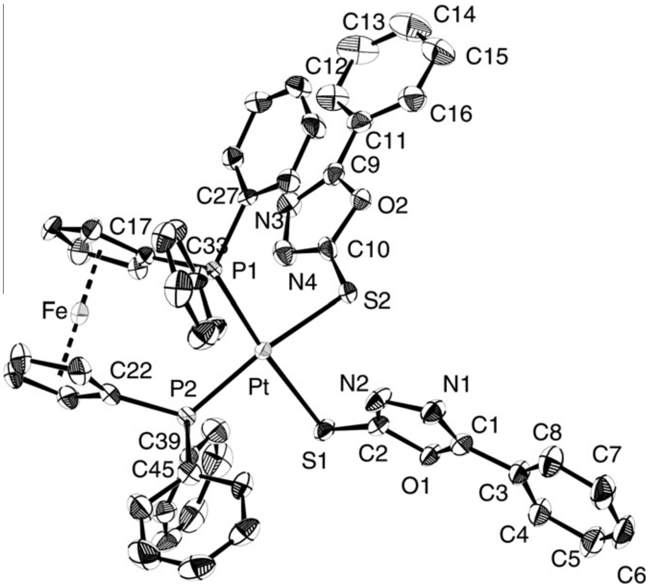
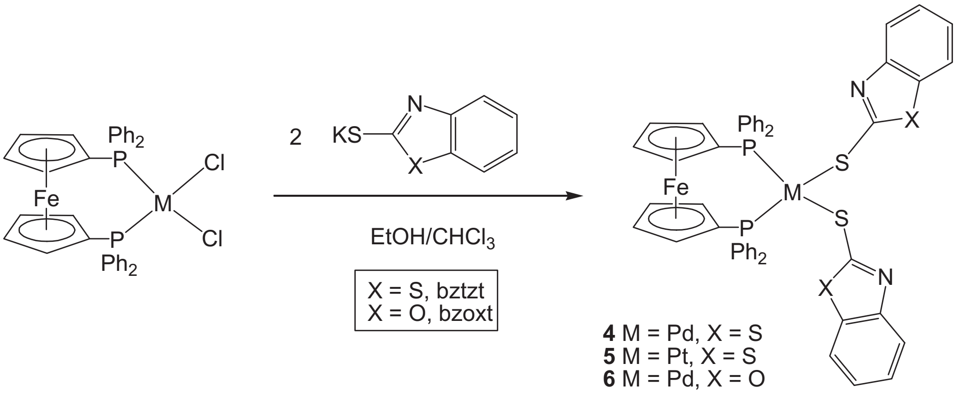


Fig. 1. Molecular structure of [$Pt(Phozt)_2(\kappa^2$-dppf)] (**2**): hydrogen atoms are omitted and only the labels of the *ipso* C atoms of the phenyl and cyclopentadienyl rings are represented for clarity, selected bond lengths (Å) and angles (°); Pt–P(1) 2.305(1), Pt–P(2) 2.317(1), Pt–S(1) 2.406(1), Pt–S(2) 2.370(1), C(2)–N(2) 1.308(7), C(10)–N(4) 1.335(7), N(1)–N(2) 1.420(6), N(3)–N(4) 1.396(7), S(1)–Pt–S(2) 85.25(4), P(1)–Pt–P(2) 98.08(5), Pt–S(1)–C(2) 99.8(2), Pt–S(2)–C(10) 99.9(2).



4 M = Pd, X = S
5 M = Pt, X = S
6 M = Pd, X = O

Scheme 2.

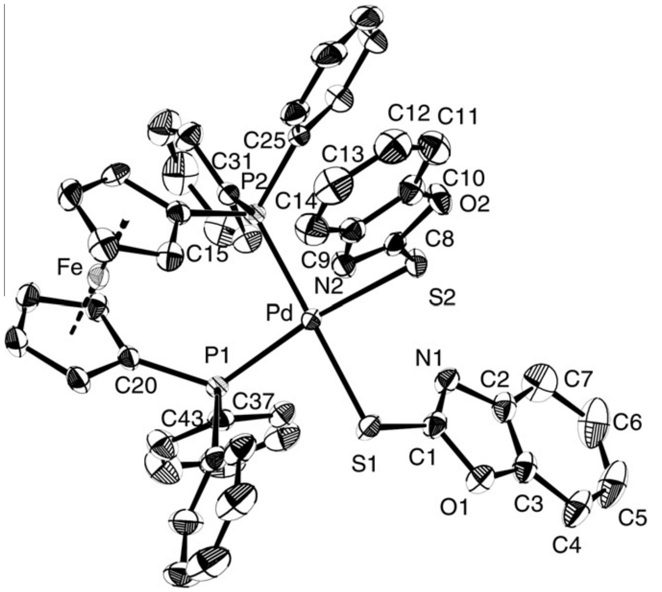


Fig. 2. Molecular structure of $[Pd(bzoxt)_2(\kappa^2\text{-dppf})]$ (**6**): hydrogen atoms are omitted and only the labels of the *ipso* C atoms of the phenyl and cyclopentadienyl rings are represented for clarity, selected bond lengths (Å) and angles (°); Pd–P(1) 2.324(8), Pd–P(2) 2.3213(8), Pd–S(1) 2.3769(8), Pd–S(2) 2.3644(8), C(1)–N(1) 1.305(5), C(8)–N(2) 1.297(4), S(1)–Pd–S(2) 89.64(3), P(1)–Pd–P(2) 96.45(3), Pd–S(1)–C(1) 107.21(12), Pd–S(2)–C(8) 96.15(12).

As detailed in Section 1, we have previously found that complexes $[ML_2\{\kappa^2\text{-}Ph_2P(CH_2)_nPPh_2\}]$ (L = bztzt and bzoxt) exist as a mixture S- and N-bound isomers [13]. We have now prepared the related dppf complexes $[M(bztzt)_2(\kappa^2\text{-dppf})]$ (**4–5**) and $[Pd(bzoxt)_2(\kappa^2\text{-dppf})]$ (**6**) as red-brown (M = Pd) or yellow (M = Pt) solids in 73–91% yields upon simple addition of Kbztzt [34] or Kbzoxt [35] to $[MCl_2(\kappa^2\text{-dppf})]$ (Scheme 2). Again all showed a single resonance in the $^{31}P\{^1H\}$ NMR spectrum and for $[Pt(bztzt)_2(\kappa^2\text{-dppf})]$ (**5**) the J_{Pt-P} coupling constant of 3264 Hz was strongly indicative of the sulfur-bound coordination of the new ligands [36]. An X-ray crystal structure of **6** confirmed this, the results of which are summarized in Fig. 2. The overall geometry is very similar to that found in **2**. Palladium–sulfur distances are within the expected ranges and the dppf ligand subtends a bite-angle of 96.45(3)°. The two thionate ligands are now slightly further apart, the S(1)–Pd–S(2) angle being 89.64(3)°, and again the substituents lie on opposite sides of the PdS_2P_2 plane (*anti*-conformation). Unlike in **2** and related platinum bis(thiolate) complexes, the angles at sulfur now vary significantly at 96.15(12)° and 107.21(12)°. The reasons for this are not immediately clear.

4. Conclusions

We have shown in this work that palladium and platinum dppf complexes with heterocyclic thionates are easily prepared and purified with all existing in the sulfur-bound thiolate form (Chart 1). This is in contrast to related complexes with diphosphines with methylene backbones for which mixtures of sulfur- and nitrogen-bound linkage isomers are observed. The reasons for this difference are not immediately obvious but possibly relate to the more flexible nature of the dppf ligand and its greater steric demands.

Appendix A. Supplementary material

CCDC 767746 and 767747 contain the supplementary crystallographic data for **2** and **6**, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk.

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