

Heteroleptic Binuclear Palladium(II) Complexes with P₂Pd(S₂) Cores: X-ray Crystal Structures of (PdCl₂)₂(μ-Ph₂PC≡CPh₂)₂ and [Pd(MET)]₂(μ-Ph₂PC≡CPh₂)₂ where MET is (Z)-1,4-Dimethoxy-1,4-dioxobut-2-ene-2,3-dithiolate

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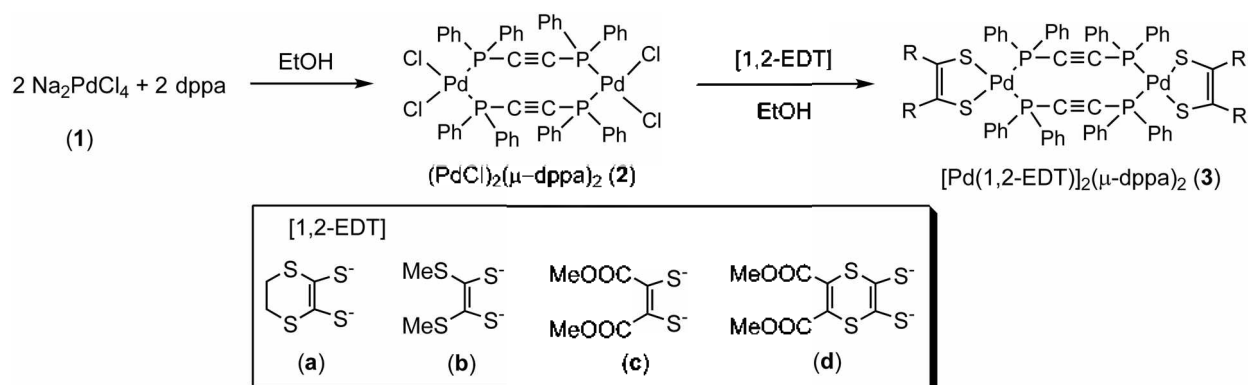
1,2-Bis(diphenylphosphino)acetylene (abbreviated as dppa: Ph₂PCCPh₂) is a linear diphosphine ligand,¹ which has been used as a bridging ligand between metals or metal clusters such as Cu-chalcogenide,² Ru₃-cluster,³ Ag(I),⁴ W₂(CO)₆,⁵ Mo(CO)₄,⁶ Pt(C₆F₅)₂,⁷ Pt(SC₆F₅)₂,⁸ Ru(acac)₂,⁹ and MX₂ (M = Pd, Pt; X = Cl, Br, I, SCN).¹⁰⁻¹² In these complexes, two phosphine groups in dppa coordinate to the metals. If the acetylenic group in dppa participates in the coordination of the metals, clusters such as the Cu-chalcogenide cluster² and Co-carbonyl cluster¹³ can be formed. The dppa ligand shows more versatile modes of coordination chemistry than in the case where only two phosphines participate in the coordination. Previous studies on dppa-complexes mainly focused on their synthesis, X-ray crystal structure analysis and (in some cases) electrochemical properties. Metal-dppa complexes with a monothiolate terminating ligand also belong to this family, but only a few have been reported so far.⁸

Very recently, we reported the results of our systematic studies of [M(1,2-BDT)]₂(μ-dppa)₂ (M = Pd(II), Pt(II); 1,2-BDT = 1,2-benzenedithiolate (BDT), 3,4-toluenedithiolate (TDT) and 1,4-dichloro-2,3-benzenedithiolate (Cl₂BDT))¹⁴ and [Pt(1,2-EDT)]₂(μ-dppa)₂ complexes (1,2-EDT = 5,6-dihydro-1,4-dithiine-2,3-dithiolate (DDDT: **a**), (Z)-1,2-bis(methylthio)ethane-1,2-dithiolate (MTDT: **b**), (Z)-1,4-dimethoxy-1,4-dioxobut-2-ene-2,3-dithiolate (MET: **c**) and 5,6-bis(methoxycarbonyl)-1,4-dithiine-2,3-dithiolate (MDDT: **d**)).¹⁵ Among these complexes, [Pt(Cl₂BDT)]₂(μ-dppa)₂ and [Pt(MET)]₂(μ-dppa)₂ showed luminescence properties in the

solution state. These two complexes were regarded as the first luminescent metal-dppa complexes with a P₂Pt(S₂) core. In this study, we present the synthesis, characterization and X-ray crystal structure of [Pd(MET)]₂(μ-dppa)₂, together with those of (PdCl₂)₂(μ-dppa)₂. We believe that these results will help provide a better understanding of the chemistry and crystal structure of metal-dppa systems.

Experimental

The HPLC grade organic solvents were purchased and used after drying them over a molecular sieve. Silica gel 60 (particle size 0.063-0.200 mm, MERCK) was used for the column chromatography. Na₂PdCl₄ (Kojima) and 1,2-bis(diphenylphosphino)acetylene (Aldrich) were purchased and used for the synthesis. The element analyses were carried out at the National Center for Inter-University Research Facilities (NCIRF) at Seoul National University. The Infra-red spectra were recorded by the KBr method on a MIDAC FT-IR spectrometer and the UV-vis spectra in acetonitrile on an HP 8452A diode array spectrometer. The Raman spectra were recorded with a Raman microscope spectrometer (Renishaw, Ltd.) equipped with a He-Ne laser (λ₀ = 6328 Å). A low laser power of ca. 0.06 mW focused on a 1 μm² area was applied. The mass spectrometric analyses were carried out at the NCIRF. The MALDI-TOF mass spectra were recorded using a Voyager-DETM STR Biospectrometry Workstation (Applied Biosystems Inc.). The ¹H and ³¹P NMR spectra were measured on an Avance 500 (Bruker) and refer-



Scheme 1. Synthesis of complexes 2 and 3.

enced to TMS and 85% H₃PO₄, respectively. The positive chemical shifts in the ³¹P NMR spectra are downfield from the standard.

Bis[μ-{bis(diphenylphosphino)acetylene}] bis(dichloropalladium(II)) (2).¹¹ An ethanol solution (75 mL) of Na₂PdCl₄ (700 mg, 2.4 mmol) was added slowly to an ethanol solution (75 mL) of dppa (950 mg, 2.4 mmol) and stirred at room temperature for 1 hr. The yellow product was filtered, washed with cyclohexane and dried in air. Recrystallization from CH₂Cl₂/CH₃OH afforded yellow plate crystals. Yield: 1.1 g (80%). EA: calc for C₅₂H₄₀C₁₄P₄Pd₂·2CHCl₃ C 46.92, H 3.06; obsd C 46.57, H 3.01. ¹H NMR (CDCl₃, ppm): δ 7.53 (16H, m), 7.48 (8H, t, ³J = 7.47), 7.26 (16H, t, ³J = 7.47). ³¹P NMR (CDCl₃, ppm): δ 4.873. FT-IR (KBr, cm⁻¹): 3056 (C-H str), 1480, 1437 (Ar ip str), 1185, 1161, 1097 (ip CH def), 1025, 998 (P-Ph str), 837, 743 (oop CH def), 689, 533, 511, 491, 475 (oop ring def). UV-vis (CH₃CN, nm, ε_{max} (× 10⁴ M⁻¹ cm⁻¹)): 266 (6.65), 326 (2.38). Raman (cm⁻¹): 2134 (C≡C), 351 (Pd-Cl).

Bis[μ-{bis(diphenylphosphino)acetylene}] bis(palladium(II)dithiolate) (3). To an ethanol suspension (10 mL) of the 4,5-ethylenedithio-1,3-dithiole-2-one precursors of the corresponding dithiolates (2 mmol; 417 mg (a), 421 mg (b), 469 mg (c), 645 mg (d)) was added potassium hydroxide (4 mmol, 210 mg) with stirring for 30 min under an argon atmosphere. (PdCl₂)₂(μ-dppa)₂ (1 mmol, 1.14 g) dissolved in methylene chloride (40 mL) was then added, followed by stirring at room temperature for 4 h. The precipitate was filtered off and washed with methylene chloride, a small amount of *dil.* acid and H₂O, in sequence. The filtrate was dried under reduced pressure and then recrystallized from CH₂Cl₂/CH₃OH. Only a single crystal of **3c** was obtained as a brown plate.

For **3a**: Yield: 74% (1.01 g). MALDI-TOF-MS (*m/z*, %): 961.28 (*M*⁺-Pd(a)-(a)+2SH+1, 69.71), 883.23 (*M*⁺-Pd(a)-(a)-Ph+2SH, 63.22), 807.17 (*M*⁺-Pd(a)-(a)-2Ph+2SH+1, 100). ³¹P NMR (CDCl₃, ppm): δ 1.460. FT-IR (KBr, cm⁻¹): 3053 (Ph C-H str), 2919, 2854 (CH₂CH₂), 2057 (C≡C), 1626 (C=C), 1480, 1436 (Ar ip str), 1161, 1098 (ip CH def), 1026, 999 (P-Ph str), 836, 744 (oop CH def), 691, 537, 514, 497 (oop ring def). UV-vis (CH₃CN, nm, ε_{max} (× 10⁴ M⁻¹ cm⁻¹)): 248 (3.38), 268 (4.29), 286 (4.07).

For **3b**: Yield: 60% (0.82 g). MALDI-TOF-MS (*m/z*, %): 1075.29 (*M*⁺-Pd(b)-1, 13.4), 909.25 (*M*⁺-Pd(b)-2Ph-CH₃+2, 35.65), 891.21 (*M*⁺-Pd(b)-2Ph-2CH₃, 47.07), 816.18 (*M*⁺-Pd(b)-3Ph-2CH₃+1, 92.62), 814.18 (*M*⁺-Pd(b)-3Ph-2CH₃, 100). ³¹P NMR (CDCl₃, ppm): δ 1.459. FT-IR (KBr, cm⁻¹): 3054 (Ph C-H str), 2946 (-CH₃), 2054 (C≡C), 1629, 1480, 1436 (Ar ip str), 1097 (ip CH def), 1025, 998 (P-Ph str), 839, 745 (oop CH def), 691, 528, 510, 490, 436 (oop ring def). UV-vis (CH₃CN, nm, ε_{max} (× 10⁴ M⁻¹ cm⁻¹)): 248 (4.25), 274 (6.24), 296 (6.58), 3.52 (0.83).

For **3c**: Yield: 72% (1.02 g). MALDI-TOF-MS (*m/z*, %): 1385.33 (*M*⁺-2CH₃+2H+1, 27.2), 917.29 (*M*⁺-Pd(c)-2Ph-2CH₃+1, 100). ³¹P NMR (CDCl₃, ppm): δ 1.045. FT-IR (KBr, cm⁻¹): 3051 (Ph C-H str), 2917 (-CH₃), 2054 (C≡C), 1720 (C=O), 1654 (C=C), 1480, 1436 (Ar ip str), 1238 (C-

Table 1. The crystal data and structure refinement parameters for (PdCl₂)₂(μ-dppa)₂·CH₂Cl₂ and [Pd(MET)]₂(μ-dppa)₂·CH₃OH.

Empirical formula	C ₅₃ H ₄₂ P ₄ Cl ₆ Pd ₂	C ₁₃₀ H ₁₁₄ P ₈ O ₁₉ Pd ₄ S ₈
Formula weight	1228.25	2910.05
Crystal system	Monoclinic	Triclinic
Space group	P2 ₁ /n (No. 14)	Pī (No. 2)
<i>a</i> (Å)	18.7287(4)	13.9109(5)
<i>b</i> (Å)	19.2294(5)	15.6357(6)
<i>c</i> (Å)	14.0517(3)	16.4604(6)
α (°)	90.00	76.826(1)
β (°)	91.007(1)	75.876(1)
γ (°)	90.00	67.516(1)
<i>V</i> (Å ³)	5059.8(2)	3171.7(2)
<i>Z</i>	4	1
μ (mm ⁻¹)	1.191	0.856
<i>F</i> (000)	2456	1478
2θ range	1.52 < 2θ < 28.33	1.29 < 2θ < 28.37
Reflections collected	50097	51147
Unique	12568 [R(int) = 0.0554]	15471 [R(int) = 0.0626]
Completeness to θ	28.33°, 99.6%	28.37°, 97.6%
GOF on F ²	1.054	1.200
<i>R</i> [wR2] [I > 2σ(I)]	0.0388 (0.0852)	0.0826 (0.1481)

O), 1185, 1131, 1098 (ip CH def), 1026, 998 (P-Ph str), 834, 744 (oop CH def), 691, 537, 514, 497, 436 (oop ring def). UV-vis (CH₃CN, nm, ε_{max} (× 10⁴ M⁻¹ cm⁻¹)): 260 (5.80), 390 (0.46).

For **3d**: Yield: 63% (1.00 g). MALDI-TOF-MS (*m/z*, %): 1574.41 (*M*⁺-CH₃+2, 31.3), 1572.42 (*M*⁺-CH₃, 27.6), 1358.42 ((*M*⁺-4COOCH₃+4H+2, 55.5). FT-IR (KBr, cm⁻¹): 3051 (Ph C-H str), 2945 (-CH₃), 2052 (C≡C), 1722 (C=O) 1625 (C=C), 1479, 1436 (Ar ip str), 1245 (C-O), 1098 (ip CH def), 1022 (P-Ph str), 840, 744 (oop CH def), 691, 555, 526, 511, 491 (oop ring def). UV-vis (CH₃CN, nm, ε_{max} (× 10⁴ M⁻¹ cm⁻¹)): 248 (4.37), 268 (4.86).

X-ray crystal structural analysis. The X-ray structure analyses for (PdCl₂)₂(μ-dppa)₂ (**2**) and [Pd(MET)]₂(μ-dppa)₂ (**3c**) were performed at 130(2) K on a Bruker SMART APEXII diffractometer equipped with graphite monochromated Mo K_α radiation (λ = 0.71073 Å). The preliminary orientation matrix and cell parameters were determined from three sets of ω/ψ or ω scans at different starting angles. The data frames were obtained at scan intervals of 0.5° with an exposure time of 10 s per frame. The reflection data were corrected for Lorentz and polarization factors. Absorption corrections were carried out using SADABS.¹⁶ The structures were solved by direct methods and refined by full matrix least-squares analysis using the anisotropic thermal parameters for non-hydrogen atoms with the SHELXTL program.¹⁷ The phenyl carbon atoms (C56, C57 and C58) of complex **3c** were disordered and assigned with an occupancy factor of 0.57:0.43. The crystal data and structural refinement parameters are shown in Table 1.

Crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (Deposition No. CCDC-

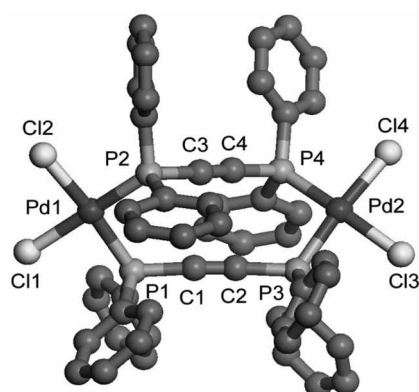


Figure 1. The molecular structure of $(\text{PdCl}_2)_2(\mu\text{-dppa})_2$ (**2**). The hydrogen atoms are omitted for clarity.

701474 for $(\text{PdCl}_2)_2(\mu\text{-dppa})_2\text{-CH}_2\text{Cl}_2$ and CCDC-701475 for $[\text{Pd}(\text{MET})]_2(\mu\text{-dppa})_2\text{-CH}_2\text{OH}$. The data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/perl/catreq.cgi> (or from the CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

Results and Discussion

The heteroleptic Pd(II) complexes of $[\text{Pd}(1,2\text{-EDT})]_2(\mu\text{-dppa})_2$ ¹⁵ were prepared by the ring-opening reaction of their oxo-analogues using KOEt (Scheme 1). The FT-IR spectra of the complexes show the characteristic vibrational peaks of each ligand: The symmetric $\nu(\text{C}\equiv\text{C})$ vibration is known to be IR inactive but Raman active in group theory.¹⁸ Therefore, the $\nu(\text{C}\equiv\text{C})$ vibrational band was not observed in the IR spectrum of **2**, but was observed in the Raman spectrum at 2134 cm^{-1} . However, it appears in the IR spectra of **3** at around 2054 cm^{-1} . This suggests that the molecular structures of complex **3c** are dynamically unsymmetrical, even though their schematic structures appear to be symmetrical, as shown in Scheme 1, possibly because of the relatively bulky and dynamic MET ligands, as can be seen in the X-ray structure analysis result. The 1,2-EDT ligands also exhibit the characteristic bands: $\nu(\text{C}=\text{C})$ of 1,4-dithiine at 1626 cm^{-1} for **3a**, $\nu(\text{C}-\text{H})$ of the $-\text{CH}_3$ moiety at *ca.* 2946 cm^{-1} for **3b**, and $\nu(\text{C}=\text{O})$ and $\nu(\text{C}-\text{O})$ of the ester moiety at 1720 cm^{-1}

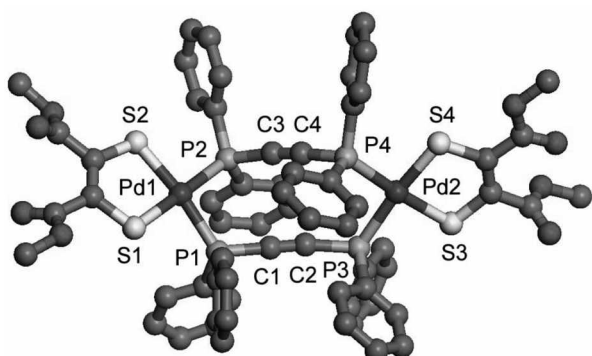


Figure 2 The molecular structure of $[\text{Pd}(\text{MET})]_2(\mu\text{-dppa})_2$ (**3c**). The hydrogen atoms are omitted for clarity.

Table 2. The selected bond distances (Å) and angles (deg) for $(2)\cdot\text{CH}_2\text{Cl}_2$ and $(3c)\cdot\text{CH}_3\text{OH}$

	$(2)\cdot\text{CH}_2\text{Cl}_2$ (X = Cl)	$(3c)\cdot\text{CH}_3\text{OH}$ (X = S)
Pd1-P1	2.2648(9)	2.2881(15)
Pd1-P2	2.2429(8)	2.2846(15)
Pd2-P3	2.2694(9)	2.3097(15)
Pd2-P4	2.2456(8)	2.2865(15)
Pd1-X1	2.3382(8)	2.2848(15)
Pd1-X2	2.3250(9)	2.2860(15)
Pd2-X3	2.3362(8)	2.2994(15)
Pd2-X4	2.3048(9)	2.2880(15)
C1-C2	1.191(4)	1.176(8)
C3-C4	1.200(4)	1.176(8)
<hr/>		
P1-Pd1-P2	96.55(3)	94.19(5)
P1-Pd1-X2	172.17(3)	170.88(6)
P2-Pd1-X2	85.74(3)	90.06(5)
P1-Pd1-X1	84.77(3)	86.91(5)
P2-Pd1-X1	173.29(3)	178.05(6)
X2-Pd1-X1	92.11(3)	89.08(5)
P3-Pd2-P4	96.74(3)	95.78(5)
P3-Pd2-X4	174.72(3)	171.07(6)
P4-Pd2-X4	88.38(3)	86.89(6)
P3-Pd2-X3	82.62(3)	88.62(5)
P4-Pd2-X3	173.75(3)	175.53(6)
X4-Pd2-X3	92.14(3)	88.65(5)

and 1238 cm^{-1} for **3c** and 1722 cm^{-1} and 1245 cm^{-1} for **3d**, respectively. The MALDI-TOF mass spectra of the complexes show very rich fragmentations. The fragmentation patterns in the low-molecular-weight region involve the consecutive loss of the 1,2-EDT ligand and phenyl groups on the dppa bridging ligand. The ³¹P NMR spectra of complex **3** show a singlet with an approximately 3.4 ppm up-field shift compared to that of complex **2**. This means that the 1,2-EDT ligands are better electron donors than the chloride ligand, which influences the electron density of the P atom through the Pd(II) ion, resulting in the higher-field resonance.

The molecular structures of $(2)\cdot\text{CH}_2\text{Cl}_2$ and $(3c)\cdot\text{CH}_3\text{OH}$ were determined by the X-ray diffraction technique and are shown in Figures 1 and 2, respectively. The selected bond lengths (Å) and angles (°) are compared in Table 2.

The average lengths of the Pd-P (2.2922(15) Å) and Pd-S (2.2896(15) Å) bonds in complex **3c** are very close to those in $[\text{Pd}(\text{BDT})]_2(\mu\text{-dppa})_2$ (2.2928(12) and 2.2818(13) Å, respectively).¹⁴ The P-Pd-S *trans* angles of complex **3c** deviate from linearity, falling in the range from $170.88(6)^\circ$ to $178.05(6)^\circ$. Compared to the large variation of the *trans* angles (7.2°), those of P-Pd-Cl for complex **2** vary within a narrow range ($172.17(3)^\circ$ - $174.72(3)^\circ$). This suggests that the MET ligand is more flexible than the chloride ligand, due to the highly disordered atoms (O1, O3 and C19) in the former, resulting in the P_2PdS_2 core being more distorted than the P_2PdCl_2 one. This disordered structure was also observed in the MET ligand of the $[\text{Pt}(\text{MET})]_2(\mu\text{-dppa})_2$ complex.¹⁵ The

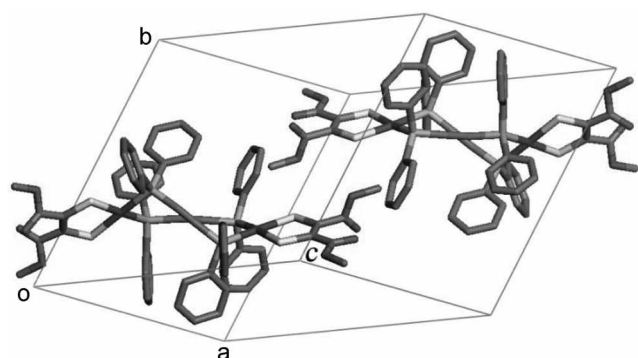


Figure 3. The close packing diagram of $[\text{Pd}(\text{MET})]_2(\mu\text{-dppa})_2$ (**3c**) showing its racemic structure. The hydrogen atoms and solvated methanol are omitted for clarity.

relatively high *R*-value of complex **3c** can be ascribed mainly to the disordering of the methyl ester groups in the MET ligand, as well as that of the phenyl groups connected to the P1 atom. This might be one of the reasons why single crystals suitable for X-ray structure analysis of neither $[\text{Pd}(\text{MET})]_2(\mu\text{-dppa})_2$, nor the other Pd(II)-dithiolate complexes prepared in this study, were able to be obtained for a long period of time, even though many attempts were made to crystallize them using various solvent pairs.

The average C=C bond length of complex **3c** (1.176(8) Å) is slightly shorter than that of complex **2** (1.196(4) Å), $[\text{Pd}(\text{BDT})]_2(\mu\text{-dppa})_2$ (1.191(6) Å)¹⁴ and $[\text{Pt}(\text{MET})]_2(\mu\text{-dppa})_2$ (1.195(13) Å).¹⁵ The two acetylenic moieties in a single molecule of complex **3c** are staggered relative to each other. Moreover, they are distorted in right- and left-handed modes and its crystal has a racemic structure (Fig. 3). These structural characteristics were also observed in the X-ray crystal structure of $(\text{ML}_n)_2(\mu\text{-dppa})_2$ complexes where ML_n is $\text{Mo}(\text{CO})_4$,⁶ $\text{Pt}(\text{C}_6\text{F}_5)_2$,⁷ $\text{Pt}(\text{SC}_6\text{F}_5)_2$,⁸ $\text{Ru}(\text{acac})_2$,⁹ $\text{Pd}(\text{BDT})$,¹⁴ and $\text{Pt}(\text{MET})$.¹⁵

In conclusion, the heteroleptic binuclear Pd(II) complexes, $(\text{PdCl}_2)_2(\mu\text{-dppa})_2$ (**2**) and $[\text{Pd}(1,2\text{-EDT})]_2(\mu\text{-dppa})_2$ (**3**) (1,2-EDT = 1,2-ethylenedithiolates), were successfully prepared and characterized by elemental analysis and MALDI-TOF, ³¹P NMR, FT-IR and UV-vis spectroscopies. Among them, the X-ray crystal structures of $(\text{PdCl}_2)_2(\mu\text{-dppa})_2$ (**2**) and $[\text{Pd}(\text{MET})]_2(\mu\text{-dppa})_2$ (where MET = 1,4-dimethoxy-1,4-

dioxobut-2-ene-2,3-dithiolate) (**3c**) were determined. Their structures are similar to those of the previously reported $[\text{M}(1,2\text{-benzenedithiolates})]_2(\mu\text{-dppa})_2$ (M = Pt(II) and Pd(II)) and $[\text{Pt}(\text{MET})]_2(\mu\text{-dppa})_2$ complexes. These results will contribute to our understanding of the chemistry of binuclear dppa complexes with a 1,2-dithiolate terminal ligand, which have not been extensively investigated.

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