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# Oxidative Addition of Aryl Disulfides to Pd(0) Complexes: Synthesis and Structures of Bis(thiolato) Pd(II) Complexes

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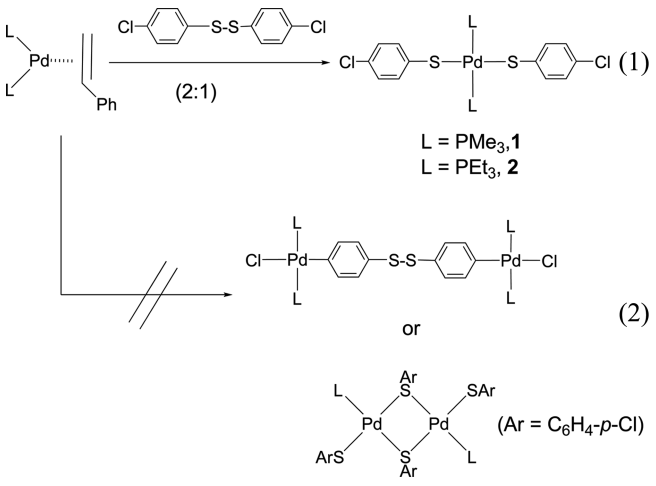
The oxidative addition of dihalo-organic compounds to zerovalent transition-metal complexes is a well-known fundamental reaction, which affords self-assembled transition-metal complexes or π-conjugated polymers in catalytic reactions.[1] In particular, dinuclear group 10 metal complexes with bridging aryl groups act as catalytic intermediates or potential precursors in the preparation of π-conjugated conducting polymers or electronic and optical materials. These complexes are typically prepared by the double oxidative addition of dihalo–aryl derivatives to zerovalent metal complexes.[1-3]

We previously reported the double oxidative addition of dihalo–aryl or dihalo–thiophene compounds to zerovalent Pd or Ni complexes to afford linear dinuclear Pd(II) or Ni(II) compounds containing bridging aryl or thiophene ligands.[4,5] However, to the best of our knowledge, studies on linear disulfido–aryl-bridged dinuclear Pd(II) complexes ([Pd]–ArS–SAr–[Pd]) have not been reported yet. Therefore, we attempted to prepare novel dinuclear Pd(II) complexes containing an aryl disulfide bridge (–Ar–S–S–Ar–) by the C–X (X = halogen) oxidative addition of haloaryl disulfide compounds to zerovalent Pd complexes. However, when *p*-chlorophenyl disulfide (Cl–*p*-$C_6H_4$–S–S–$C_6H_4$–*p*-Cl) was treated with Pd(0)–bis(phosphine) complexes, we obtained the mononuclear bis(thiolato) Pd(II)–bis(phosphine) complex, *trans*-[Pd(S–$C_6H_4$–*p*-Cl)$_2$($PR_3$)$_2$], which is an S–S oxidative addition product instead of an initially expected C–X oxidative-addition product. Herein, we report the synthesis of such mononuclear bis(thiolato) complexes. These thiolato complexes are regarded as an intermediate or precursor related to the addition of organic disulfides to the organic unsaturated molecules or the adduct formation of organic disulfides and isocynides catalyzed by late transition-metal complexes.[6-9]

Many reactions, except for a few cases,[8a,c] for the preparation of thiolato complexes typically use zerovalent group 10 metal complexes containing low basic $PPh_3$ ligand as a supporting ligand. In this study, we used Pd(0) complexes containing $PMe_3$ or $PEt_3$ ligands, which are more basic than $PPh_3$. Reactions of [Pd(styrene)($PR_3$)$_2$] ($PR_3$ = $PMe_3$, $PEt_3$),[10] which was generated from *trans*-[$PdEt_2$($PR_3$)$_2$] and styrene, with *p*-chlorophenyl disulfide in a 2:1 or 1:1 molar ratio afforded the bis(arylthiolato) Pd(II) complexes, *trans*-[Pd($SC_6H_4$-*p*-Cl)$_2$$L_2$] (L = $PMe_3$ (**1**), $PEt_3$ (**2**)) in moderate-to-good yields (Eq. (1) in Scheme 1). A one-step reaction of *trans*-[$PdEt_2$($PMe_3$)$_2$] with *p*-chlorophenyl disulfide in 2:1 molar ratio also produced complex **1** in 69% yield.

The peak-intensity ratios of the aromatic hydrogens to the $PR_3$ hydrogens in the $^1$H NMR spectra of complexes **1** and **2** strongly support the proposed structures. Spectral data ($^1$H, $^{31}$P{$^1$H}, and $^{13}$C{$^1$H} NMR) indicate that the diphenyl disulfido or the phenylthiolato–bridged dinuclear Pd(II) complexes (Eq. (2) in Scheme 1) are not present. The mononuclear bis(thiolato) Pd(II) complexes (in Eq. (1)) can be formed by the S–S bond cleavage, whereas the dinuclear Pd(II) complexes (in Eq. (2)) can be formed by the C–Cl bond cleavage of the aryl disulfide, followed by subsequent dimerization accomplished by the dissociation of phosphine. The above results strongly indicate that the bond dissociation energy (BDE) dominates the reactivity. The BDE of the C–Cl bond in the aryl chloride is about 81 kcal/mol,[11] whereas that of the S–S bond falls in the range of 60–70 kcal/mol.[12] As a result, the S–S bond is believed to be cleaved before the C–Cl bond to produce complexes **1** and **2**.

Graziani *et al.* previously reported the formation of two rather distinct products when [Pd($PPh_3$)$_4$] was treated with aryl disulfides.[13] When the disulfide $(PhS)_2$ was employed, a




**Scheme 1**

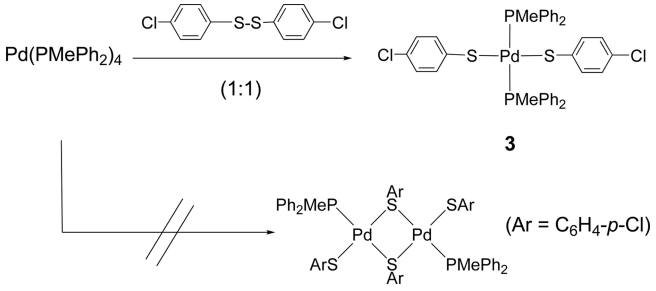
sulfide-bridged dinuclear complex, $[Pd_2(SPh)_4(PPh_3)_2]$, was formed. However, when *ortho*- or *meta*-nitrophenyl disulfide was used, a mononuclear dithiolato Pd(II) complex, *trans*-$[Pd(SC_6H_4–X)_2(PPh_3)_2]$ (X = *o*- or *m*-$NO_2$), was obtained as a sole product. Although the *p*-chlorophenyl disulfide is employed in our reaction system, its reactivity is consistent with that found for the *ortho*- or *meta*-nitrophenyl disulfide that had been treated with $[Pd(PPh_3)_4]$. Kuniyasu and co-workers[9] reinvestigated the Graziani's reactions and revealed that a mixture of a dinuclear complex {$[Pd_2(SPh)_4(PPh_3)_2]$} and a mononuclear complex {$[Pd(SPh)_2(PPh_3)_2]$} were formed initially, and the mononuclear species was eventually converted to the dinuclear species. Ananikov *et al*.[8b] spectroscopically characterized the *trans*/*cis* isomers of both the mononuclear complex, $[Pd(SPh)_2(PPh_3)_2]$, and the dinuclear *trans*/*cis* isomers of $[Pd_2(SPh)_4(PPh_3)_2]$. Ananikov *et al*.[8a] recently described the replacement of $PAr_3$ ligand by $PAr_2R$ or $PR_3$ ligands shifts the equilibrium to destabilize dinuclear complexes and stabilize mononuclear metal complexes after oxidative addition.

On the other hand, oxidative addition with a disulfide-containing bulky thioether was reported to afford mononuclear or trinuclear Pd sulfide complexes by S–S bond cleavage.[14] In addition, several mononuclear thiolato Ni(II) complexes were obtained by reactions of $[Ni(cod)_2]$,[15] $[NiMe_2(PMe_3)_3]$,[16] and $[Ni(acac)_2]$[8c] with diaryl sulfide, aryl thiol, and aryl disulfide, respectively.

In this work, the tetrakis(phosphine) Pd(0) complex, $[Pd(PMePh_2)_4]$, reacted with $(SC_6H_4$-*p*-$Cl)_2$ to produce only the mononuclear dithiolato complex, *trans*-$[Pd(SC_6H_4$-*p*-$Cl)_2(PMePh_2)_2]$ (**3**) in 95% yield (Scheme 2). The C–Cl bond cleavage product, arylthiolato-bridged dinuclear complex, was not observed, as confirmed by NMR. The above results (Schemes 1 and 2) indicate that $PMe_3$ and $PMePh_2$ ligands, which are more basic than the $PPh_3$, as well as the activated halo-aryldithiolao compounds used in this work induces the cleavage of the S–S bond (or S–S oxidative addition), which leads to the formation of mononuclear bis(thiolato) Pd(II) complex.

We further investigated the reactivity between $[Pd(styrene)(PMe_3)_2]$ and several non-activated disulfides such as thienyl disulfide, dianiline disulfide, and *p*-tolyl disulfide (Scheme 3). In contrary to our expectation, all reactions exclusively afforded the mononuclear dithiolato Pd(II) complexes (**4**-**7**) in moderated-to-good yields. Therefore, these results suggest that the basicity of the phosphine ligands, rather than that of the non-activation of the organic group in the aryl disulfide, is important for the selective S–S oxidative addition for mononuclear bis(thiolato) Pd(II) complexes without other products such as the dinuclear complex.

All single crystals for X-ray crystallography were grown from $CH_2Cl_2$/hexane at −35 °C. Details on crystal data of complexes **1**, **3**, and **5** are summarized in Supporting Information. The molecular structures of **1**, **3**, and **5** are given in Figures 1-3, respectively. The asymmetric unit of complex **1** consists of two half molecules, which are crystallographically independent. Both Pd atoms are located on the crystallographic inversion centers. The thiolato group in each the structure is coordinated almost perpendicular to the square-planar sphere. In complexes **3** and **5**, the Pd atom lies at the crystallographic center of symmetry, and the remaining atoms occupy general positions.

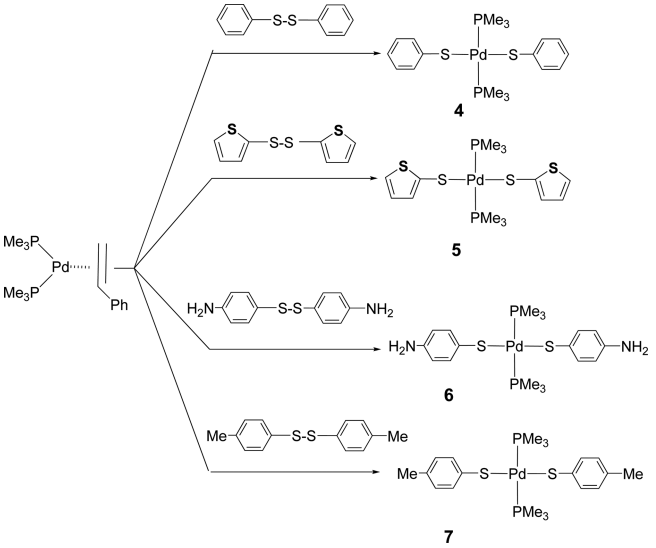



**Scheme 2**

In summary, we observed that aryl disulfides (ArS–SAr) underwent S–S oxidative addition to zerovalent Pd complexes, {$[Pd(styrene)(PR_3)_2]$ and $[Pd(PR_3)_4]$}, to exclusively produce bis(arylthiolato) Pd(II) complexes, *trans*-$[Pd(SAr)_2(PR_3)_2]$. NMR measurements confirmed that neither the C–Cl bond cleavage products nor the arylthiolato-bridged dinuclear complexes were observed. This reaction could also be carried out with several non-activated disulfides such as phenyl disulfide, thienyl disulfide, dianiline disulfide, and *p*-tolyl disulfide.

## Experimental

**General Procedures.** All manipulations of air-sensitive compounds were performed under $N_2$ or Ar by standard Schlenk techniques. Solvents were distilled from Na–benzophenone. The analytical laboratories at Kangnung–Wonju National University carried out elemental analyses with CE instruments EA1110. IR spectra were recorded on a Perkin Elmer BX spectrophotometer. NMR ($^1H$, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$) spectra were obtained on a JEOL Lamda 300 and




**Scheme 3**

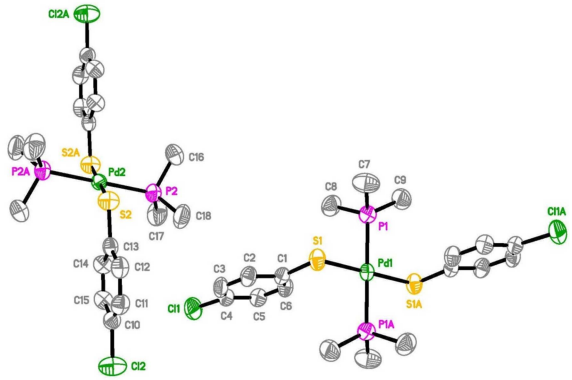



**Figure 1.** ORTEP drawing of compound **1** with 50% probability thermal ellipsoids. Labeled atoms with "A" are related to unlabeled ones by the crystallographic inversion symmetry. Selected bond lengths (Å) and angles (°): Pd1–P1 2.3153(6), Pd1–S1 2.3391(5), Pd2–P2 2.3145(6), Pd2–S2 2.3434(5), Cl1–C4 1.747(2), Cl2–C10 1.747(2); P1–Pd1–S1 86.88(2), P1–Pd1–S1A 93.12(2), P1–Pd1–P1A 180.00(2), P2–Pd2–S2 92.95(2), P2–Pd2–S2A 87.05(2), P2A –Pd2–P2, 180.00(3).

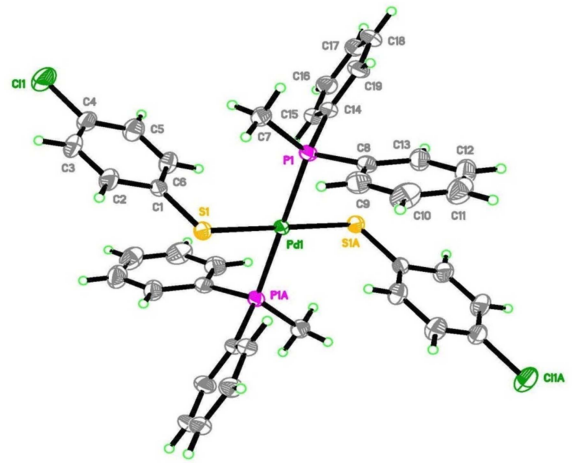



**Figure 2.** ORTEP drawing of compound **3** with 40% probability thermal ellipsoids. Labeled atoms with "A" are related to unlabeled ones by the crystallographic inversion symmetry. Selected bond lengths (Å) and angles (°): Pd1–P1 2.3185(4), Pd1–S1 2.3358(4), S1–C1 1.753(2); P1–Pd1–S1 93.46(14), C1–S1–Pd1 108.47(5).

ECA 600 MHz spectrometer. Chemical shifts were referenced to internal $Me_4Si$ or to external 85% $H_3PO_4$. X-ray analyses were obtained at Korea Basic Science Institute (Jeonju center) and CCRF (Cooperative Center for Research Facilities in the Sungkyunkwan University). *Trans*-[$PdEt_2(PR_3)_2$] (R = $PMe_3$, $PEt_3$) was prepared by the literature method.[10] Phenyl disulfide, thienyl disulfide, dianiline disulfide and *p*-tolyl disulfide were commercially available.

**Preparation of *trans*-[Pd($SC_6H_4$-*p*-Cl)$_2$($PR_3$)$_2$] ($PR_3$ = $PMe_3$, (1); $PEt_3$, (2)).** Styrene (78 μL, 0.68 mmol) and tetrahydrofuran (THF, 3 mL) were added sequentially to a Schlenk flask containing *trans*-[$PdEt_2(PMe_3)_2$] (0.107 g, 0.34 mmol) at 0 °C. The mixture was heated at 55 °C for 30 min to give a yellow solution. *p*-Chlorophenyl disulfide (0.049 g, 0.17 mmol) was added to the mixture at room temperature,

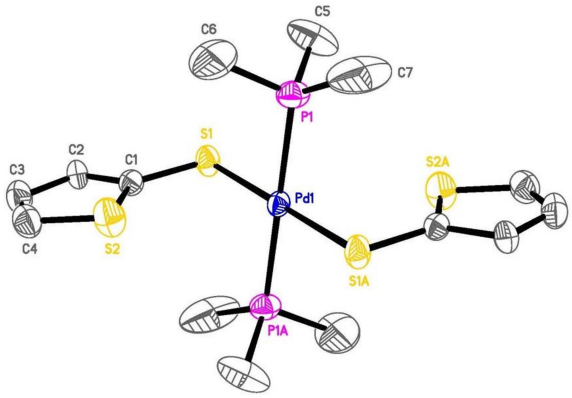



**Figure 3.** ORTEP drawing of compound **5** with 40% probability thermal ellipsoids. Labeled atoms with "A" are related to unlabeled ones by the crystallographic inversion symmetry. Selected bond lengths (Å) and angles (°): Pd1–P1 2.3175(7), Pd1–S1 2.3422(6), S1–C1 1.739(2), S2–C4 1.701(3), S2–C1 1.720(2); P1–Pd1–P1A 180.00(3), P1–Pd1–S1 87.06(3), P1–Pd1–S1A 92.94(3), C1–S1–Pd1 105.37(8), C4–S2–C1 92.38(14).

and then the yellow solution turned into a dark yellow solution. After stirring for 2 h at room temperature, the solvent was completely removed under vacuum, and then the resulting residue was solidified with hexane. The solids were filtered and washed with hexane (2 mL × 2) to obtain the crude solids. Recrystallization from $CH_2Cl_2$/*n*-hexane afforded pale yellow crystals of *trans*-[Pd($SC_6H_4$-*p*-Cl)$_2$-($PMe_3$)$_2$] (**1**, 0.067 g, 73%). $C_{18}H_{26}Cl_2P_2S_2Pd$ (545.80): calcd. C 39.61, H 4.80, S 11.75; found C 39.33, H 4.87, S 11.45. $^1$H NMR (300 MHz, $CDCl_3$) δ 1.36 (t, $J$ = 3.3 Hz, 18H, P($CH_3$)$_3$), 7.01 (m, 4H, Ar–*H*), 7.43 (m, 4H, Ar–*H*). $^{13}$C{$^1$H} NMR (75 MHz, $CDCl_3$) δ 13.7 (t, $J_{cp}$ = 16 Hz, P(*C*$H_3$)$_3$), 127.7, 127.8, 132.1 (t, $J_{CP}$ = 1.2 Hz, Ar), 146.1. $^{31}$P{$^1$H} NMR (120 MHz, $CDCl_3$) δ 12.9 (s).

Complex **2** (92%) was analogously prepared using a 1:1 molar ratio of *trans*-[$PdEt_2(PEt_3)_2$] and *p*-chlorophenyl disulfide. $C_{24}H_{38}Cl_2P_2S_2Pd$ (629.96): calcd. C 45.76, H 6.08, S 10.18; found C 45.78, H 6.10, S 9.79. $^1$H NMR (300 MHz, $CDCl_3$) δ 1.02 (quin, $J$ = 8.0 Hz, 18H, P($CH_2CH_3$)$_3$), 1.82 (m, 12H, P($CH_2CH_3$)$_3$), 7.0 (m, 4H, Ar–*H*), 7.42 (m, 4H, Ar–*H*). $^{13}$C{$^1$H} NMR (75 MHz, $CDCl_3$) δ 8.28 (s, P($CH_2CH_3$)$_3$), 13.8 (t, $J_{CP}$ = 14 Hz, P($CH_2CH_3$)$_3$), 127.4, 127.6, 132.3, 146.0. $^{31}$P{$^1$H} NMR (120 MHz, $CDCl_3$) δ 14.9 (s).

Complex **1** could also be synthesized via an alternate route. *p*-Chlorophenyl disulfide (0.236 g, 0.82 mmol) and THF (6 mL) were added to a Schlenk flask containing *trans*-[$PdEt_2(PMe_3)_2$] (0.524 g, 1.65 mmol) at 0 ºC. After stirring the reaction mixture for 18 h at room temperature, the solvent was completely removed under vacuum to obtain crude solids which were recrystallized from $CH_2Cl_2$/*n*-hexane (0.313 g, 69%).

**Preparation of *trans*-[Pd($SC_6H_4$-*p*-Cl)$_2$($PMePh_2$)$_2$], (3).** *p*-Chlorophenyl disulfide (0.123 g, 0.43 mmol) and $CH_2Cl_2$ (2 mL) were added to a Schlenk flask containing [$Pd(PMePh_2)_4$] (0.387 g, 0.43 mmol) at room temperature. The initial red solution turned to a dark yellow solution. After the resulting mixture was stirred for 12 h, the solvent was completely

removed under vacuum, and then the resulting residue was solidified with diethyl ether. The resulting solids were filtered and washed with hexane (2 mL × 2) to obtain the crude solids. Recrystallization from $CH_2Cl_2$/*n*-hexane afforded brown crystals of *trans*-[Pd(SC$_6$H$_4$-*p*-Cl)$_2$(PMePh$_2$)$_2$], (**3**, 0.321 g, 95%). $C_{38}H_{34}Cl_2P_2S_2Pd$ (794.08): calcd. C 57.48, H 4.31, S 8.08; found C 57.03, H 4.31, S 7.75. $^1$H NMR (300 MHz, $CDCl_3$) δ 1.91 (t, $J$ = 3.3 Hz, 6H, P(C$H_3$)Ph$_2$), 6.65 (m, 4H, Ar–*H*), 6.96 (m, 4H, Ar–*H*), 7.22–7.36 (m, 12H, Ar–*H*), 7.45–7.52 (m, 8H, Ar–*H*). $^{13}$C{$^1$H} NMR (75 MHz, $CDCl_3$) δ 13.0 (t, $J_{cp}$ = 16 Hz, P(*C*H$_3$)Ph$_2$), 127.1, 127.6, 128.1 (t, $J_{CP}$ = 5.0 Hz, Ar), 130.0, 132.2, 132.5 (t, $J_{CP}$ = 6.2 Hz, Ar), 132.9, 145.1. $^{31}$P{$^1$H} NMR (120 MHz, $CDCl_3$) δ 9.1(s).

**Preparation of *trans*-[Pd(S-C$_6$H$_5$)$_2$(PMe$_3$)$_2$] (4), *trans*-[Pd(S-C$_4$H$_3$S)$_2$(PMe$_3$)$_2$] (5), and *trans*-[Pd(SC$_6$H$_4$-P-X)$_2$-(PMe$_3$)$_2$] (X = NH$_2$, (6); X = Me, (7)).** Styrene (114 μL, 0.99 mmol) and THF (3 mL) were sequentially added to a Schlenk flask containing *trans*-[PdEt$_2$(PMe$_3$)$_2$] (0.157 g, 0.50 mmol) at 0 °C. The mixture was heated at 55 °C for 30 min to give a yellow solution. Phenyl disulfide (0.108 g, 0.50 mmol) was added to the mixture at room temperature, and then the initial orange solution turned into a yellow suspension. After stirring for 2 h, the solvent was completely removed under vacuum, and then the resulting solids were filtered and washed with hexane (2 mL × 3) to obtain the crude solids. Recrystallization from $CH_2Cl_2$/hexane gave yellow crystals of *trans*-[Pd(S-C$_6$H$_5$)$_2$(PMe$_3$)$_2$] (**4**, 0.159 g, 73%). $C_{14}H_{24}P_2S_2Pd$ (476.91): calcd. C 45.33, H 5.92, S 13.45; found C 45.29, H 5.98, S 13.22. $^1$H NMR (300 MHz, $CDCl_3$) δ 1.35 (t, $J$ = 3.3 Hz, 18H, P(C$H_3$)$_3$), 6.91–6.96 (m, 2H, Ph), 7.04–7.09 (m, 4H, Ph), 7.54–7.57 (m, 4H, Ph). $^{13}$C{$^1$H} NMR (75 MHz, $CDCl_3$) δ 13.7 (t, $J_{cp}$ = 16 Hz, P(*C*H$_3$)$_3$), 122.0, 127.8, 131.3 (t, $J_{cp}$ = 1.2 Hz, Ph–*C*), 147.4. $^{31}$P{$^1$H} NMR (75 MHz, $CDCl_3$) δ −13.1(s).

The formation of complex **4** was confirmed by comparing its spectral data with those reported previously.[17,18] Complexes **5**–**7** were analogously prepared. Spectroscopic data are summarized in Supporting Information.

**X-ray Structure Determination.** All X-ray data were collected on a Bruker Smart APEX or APEX2 diffractometer equipped with a Mo X-ray tube. Collected data were corrected for absorption with SADABS based upon the Laue symmetry by using equivalent reflections.[19] All calculations were carried out with SHELXTL programs.[20] All structures were solved by direct methods.

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Centre, CCDC No. 954099 (for **1**) and 954100 (for **3**) and 954101 (for **5**). 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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