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Synthesis and structure of methylpalladium(II) and -platinum(II) complexes with 2-allylphenoxido ligands, *trans*-PdMe($O_6H_4CH_2CH{=}CH_2$-*o*)$(PR_3)_2$ (R = Me, Ph) and PtMe(η^1-*O*, η^2-*C*,*C*-$OC_6H_4CH_2CH{=}CH_2$-*o*)(PMe_3). Simple *O*-coordination and chelating coordination depending on the metal center and auxiliary phosphine ligand

Yong-Joo Kim [a,*], Jae-Young Lee [a], Kohtaro Osakada [b]

[a] *Department of Chemistry, Kangnung National University, Kangnung, 210-702, South Korea*
[b] *Research Laboratory of Resources Utilization, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-ku, Yokohama, 226, Japan*

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Abstract

Reactions of *cis*-$PdMe_2L_2$ (L = PMe_3, PPh_3, 0.5 dppp [dppp = $Ph_2P(CH_2)_3PPh_2$]) with equimolar 2-allylphenol afford the new complexes PdMe($OC_6H_4CH_2CH{=}CH_2$-*o*)L2 (**1**: L = PMe_3; **2**: L = PPh_3; **3**: L = 0.5 dppp) in moderate to high yields. Addition of 2,2,2-trifluoro-1-phenylethanol to equimolar **1** results in formation of *trans*-PdMe($O_6H_4CH_2CH{=}CH_2$-*o*)$(PMe_3)_2$ · ($HOCH(CF_3)$ Ph), (**4**) which has been characterized by X-ray crystallography and NMR spectroscopy. Complex **4** contains O–H···O hydrogen bonding between the allylphenoxido ligand and associated alcohol (O···O = 2.635(6) Å). The platinum complex PtMe($OC_6H_4CH_2CH{=}CH_2$-*o*)(dppp), (**5**) is obtained from the alkoxido ligand exchange reaction of PtMe($OCH(CF_3)_2$)(dppp) with 2-allylphenol. Similar reaction of *cis*-PtMe(OR)$(PMe_3)_2$ with equimolar 2-allylphenol at room temperature gives *trans*-PtMe($OC_6H_4CH_2CH{=}CH_2$-*o*)$(PMe_3)_2$ (**6**) in 55% yield. In contrast, reaction of *cis*-PtMe($OCH(CF_3)_2$)$(PMe_3)_2$ · ($HOCH(CF_3)_2$) with excess 2-allylphenol at room temperature gives a mixture of **6** and PtMe(η^1-*O*, η^2-*C*,*C*-$OC_6H_4CH_2CH{=}CH_2$-*o*)(PMe_3) (**7**) which are isolated in 16 and 28% yields, respectively. The isolated complex **7** has been characterized by NMR spectroscopy using several pulse techniques and X-ray analysis. The molecule has a distorted square-planar coordination around the metal center. The olefinic group in 2-allylphenoxido ligand is coordinated to the Pt center in a perpendicular fashion to the coordination plane.

Keywords: Palladium; Platinum; Phenoxide; Hydrogen bonding; Crystal structure

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1. Introduction

The chemistry of late transition metal phenoxides has been the subject of increasing attention because of their characteristic chemical properties such as C–O bond formation through coupling of the phenoxido and acyl ligand [1,2], O–H···O hydrogen bonding between phenoxido ligand and phenol [3–8], and CO insertion into the metal–phenoxido bond [9–11]. Most of the phenoxido complexes were prepared by metathesis reactions of chloro complex with alkaline metal phenoxido or by reaction of hydrido or alkyl metal species with phenol. Previously we have reported that reactions of dialkyl-nickel (II), -palladium (II), and -platinum(II) complexes stabilized by tertiary or chelating phosphines with equimolar phenols with various substituents, HOC_6H_4X-*p* (X = H, Me, OMe, F, Cl, and Br), proceed smoothly at room temperature to give alkyl-nickel

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* Corresponding author.

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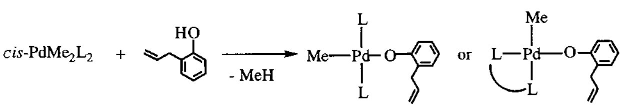
(II), -palladium (II), and -platinum (II) phenoxides or complexes, $MR(OAr)L_2 \cdot (HOAr)$ containing phenol associated with phenoxido ligands through hydrogen bonding [6]. Recently, van Koten and his coworkers [7] also showed that reactions of dimethylpalladium(II) complexes with auxiliary *N*,*N* or *N*,*P*-donor chelating ligands with phenol or pare- substituted phenols lead to the formation of palladium(II) phenoxides or those with associated phenol through the O–H···O hydrogen-bond stabilized by the amine ligands.

On the other hand, Pd(II) complex containing both alkoxido and π-coordinated olefin ligands seems to be of importance as a possible intermediate of alkoxypalladation of olefins or the Wacker type reaction. A strategy to isolate this kind of Pd complex and to investigate its properties is to use alcohol containing C=C double bond at the proper position to serve as the anchor in the molecule as the ligand precursor. Ligation of alkoxido would be accompanied by π-coordination of the C=C double bond. In this paper as an extension of the previous work we will show chemistry of methylpalladium and platinum(II) complexes containing 2-allylphenoxido ligand including synthetic details, spectroscopic characterization, and crystallographic results of some of the complexes.

2. Results and discussion

Reactions of *cis*-$PdMe_2L_2$ ($L = PMe_3$, PPh_3, 0.5 dppp) with an equimolar amount of 2 allylphenol at 0°C give *trans*-$PdMe(OC_6H_4CH_2CH{=}CH_2\text{-}o)L_2$ (**1**: $L = PMe_3$; **2** $L = PPh_3$) and $PdMe(OC_6H_4{-}CH_2CH{=}CH_2\text{-}o)$(dppp), (**3**) in moderate to good yields, respectively.



(1)

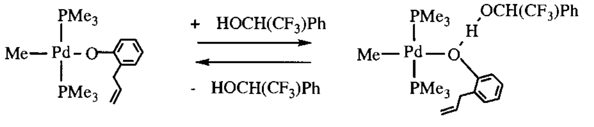
$L = PMe_3$, **1** $L = PPh_3$, **2**

$L-L = Ph_2P-(CH_2)_3-PPh_2$, **3**

Complexes **1**–**3** are colorless or yellow crystalline solids whose IR spectra give rise to a small absorption peak at 1634–1636 cm^{-1} due to C=C stretching frequency of the allyl group. Similarity of the peak position to the corresponding band of free 2-allylphenol suggests that the C=C double bond of the allylphenoxido ligand does not coordinate to the metal center. The ^1H- and ^{13}C-NMR spectra as well as results of $^{13}C-^1H$ COSY measurement have provided sufficient information to characterize these complexes unambiguously. The ^1H- and $^{13}C\{^1H\}$-NMR signals due to the methyl ligand of **1** and **2** appear at reasonable positions accompanied by coupling with two magnetically equivalent phosphorus nuclei. Complex **3** having *cis* coordination shows the ^1H- and $^{13}C\{^1H\}$-NMR signals as a doublet of doublets caused by coupling with two unequivalent phosphorus nuclei. The ^1H- and ^{13}C-NMR signals of PMe_3 ligands of **1** and $^{13}C\{^1H\}$-NMR peak due to *ipso* carbons of PPh_3 ligands of **2** are observed as apparent triplets due to virtual coupling. The $^{13}C\{^1H\}$-NMR signals due to olefinic carbons of 2-allylphenoxido ligand of **1**–**3** appear at quite similar position to each other (113.0–114.0 and 139.1–140.0 ppm) and to the corresponding signals of uncoordinated 2-allylphenol. The above results strongly indicate the absence of coordination or interaction between C=C double bond of the 2-allylphenoxido ligand and the metal center.

Previously we reported that reaction of *cis*-$PdMe_2(PMe_3)_2$ with excess phenol gives a complex formulated as *trans*-$PdMe(OPh)(PMe_3)_2 \cdot (HOPh)$, which is prepared also from the reaction of *trans*-$PdMe(OPh)(PMe_3)_2$ with phenol [6]a. Strong O–H···O hydrogen bonding exists between the phenoxido ligand and phenol not only in the solid state but in solution. Reaction of *cis*-$PdMe_2(PMe_3)_2$ with excess 2-allylphenol does not produce the hydrogen-bonded complex such as *trans*-$PdMe(OC_6H_4CH_2CH{=}CH_2\text{-}o)(PMe_3)_2 \cdot (HOC_6H_4CH_2CH{=}CH_2\text{-}o)$ but gives **1** as the sole isolable product. The difference of the reaction products between phenol and 2-allylphenol is ascribed to weakening the O–H···O hydrogen bond of the 2-allylphenoxido ligand with 2-allylphenol caused by steric congestion and/or to poor crystallinity of *trans*-$(C_6H_4CH_2CH{=}CH_2\text{-}o)L_2 \cdot (HOC_6H_4CH_2CH{=}CH_2\text{-}o)$, preventing from its crystallization from the solution.

Complex **1** reacts with equimolar 2,2,2-trifluoro-1-phenylethanol to give *trans*-$PdMe(OC_6H_4\text{-}o\text{-allyl})(PMe_3)_2 \cdot (HOCH(CF_3)ph)$ (**4**) as shown in Eq. (2).



(2)

1 **4**

Complex **4** has been isolated as a colorless crystalline solid, and characterized by X-ray crystallography and NMR spectroscopy as shown below. The structure having 2,2,2-trifluoro-1-phenylethanol hydrogen bonded to the coordinated oxygen of the 2-allylphenoxido ligand is stable in the solid state at room temperature but the complex readily loses the associated fluoro alcohol in solution during recrystallization and regenerates complex **1**.

Fig. 1 shows the molecular structure of **4** whose selected bond lengths and angles are listed in Table 1. The molecular structure of **4** contains slightly distorted square planar coordination around the palladium center containing two mutually bans PMe_3 ligands as well as methyl and 2-allylphenxido ligands. 2,2,2-Trifluoro-1-phenylethanol forms an O–H···O hydrogen bonding with the coordinated oxygen atom. The O···O nonbonded distance (2.635(6) Å) falls in the range of the

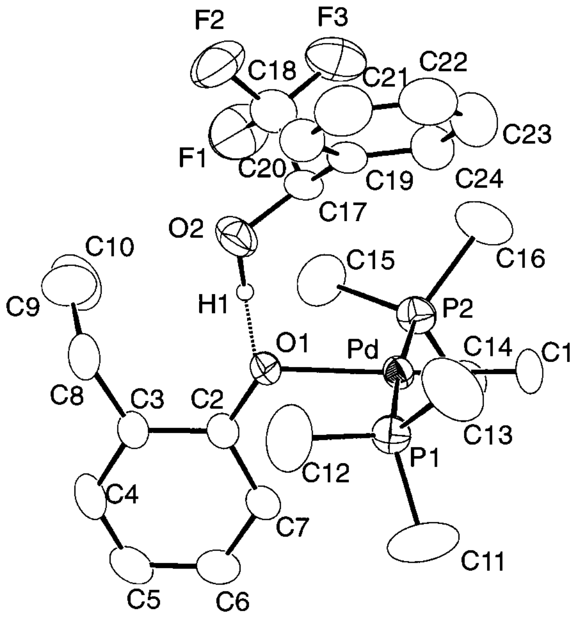


Fig. 1. ORTEP drawing of **4** showing the atomic labeling scheme and 50% probability thermal ellipsoids.

hydrogen bond with significant strength (2.55–2.65 Å), and are comparable to O···O distances and angles in related palladium(II) and platinum(II) alkoxide or phenoxide complexes [4–7]. D-fourier technique has revealed position of the OH hydrogen which lies on the O–O line (O–H = 1.03(6) Å, O···H = 1.61(6) Å, O···H–O = 170(5)°). The Pd–O (2.129(3) Å) distance is similar to or slightly longer than those of previously reported the Pd–O distances in related palladium phenoxide complexes, bans $PdMe(OC_6H_5)(PMe_3)_2 \cdot (HOR)$ (R = $CH(CF_3)Ph$) (2.107(2) Å), *trans*-$PdMe(OC_6H_5)(PMe_3)_2 \cdot (HOC_6H_5)$ (2134(3) Å), and *trans*-$PdMe(OC_6H_5)(PMe_3)_2$ (2.106(3) Å) [6]a. Van Koten and his co-workers have observed similar slight elongation of the Pd–O bond of phenoxido complex with chelating diamine auxiliary ligands [7]a,c. Non-bonded distance between Pd and hydrogen bonded to C17 of the fluoro alcohol in **4** (2.73 Å) falls within the expected sum of van der Waals radii of H and 4d group metals, indicating the presence of agostic interaction between the C–H group and the Pd center [12].

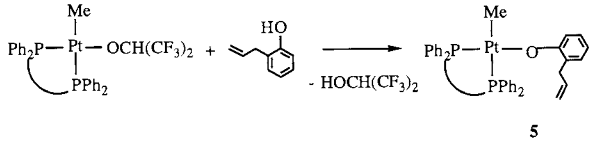
Table 1
Selected bond lengths (Å) and angles (°) for **4**

Pd–O(1)	2.129(3)	O(1)···O(2)	2.635(6)
O(1)–H(1)	1.03(6)	O(1)···H(1)	1.61(6)
Pd–C(1)	2.067(5)	C(3)–C(8)	1.498(8)
Pd–P(1)	2.306(2)	C(8)–C(9)	1.465(9)
Pd–P(2)	2.301(2)	C(9)–C(10)	1.240(10)
Pd–O(1)–C(2)	121.5(3)	O(1)···H(1)–O(2)	170(5)
P(1)–Pd–P(2)	175.43(6)	P(1)–Pd–O(1)	91.8(1)
C(1)–Pd–O(1)	176.7(2)	P(2)–Pd–O(1)	92.5(1)

The ^1H-NMR spectrum of **4** taken in $CDCl_3$ (25°C) shows an OH hydrogen peak of the associated alcohol at 5.8 ppm which is at a higher magnetic field position than those of other palladium phenoxides with an associated fluoro alcohol or phenol. These results indicate weak hydrogen bonding between the phenoxido and an alcohol in solution and agree with the observation that **4** dissociates easily the fluoro alcohol in the solution.

We have examined preparation of analogous platinum(II) complexes. Similar reaction of $PtMe_2(dppp)$ with 2-allylphenol does not give the complex Pt-$Me(OC_6H_4CH_2CH{=}CH_2$-*o*)(dppp), (**5**) at all. On the basis of previous our results including reaction of *cis*-$PtMe(OCH(CF_3)_2)(PMe_3)_2$ by phenol to give *cis*-Pt-$Me(OPh)(PMe_3)_2$ [6]b, similar ligand exchange reaction of *cis*-$PtMe(OCH(CF_3)_2)$(dppp) has been carried out by use of 2-allylphenol.

The reaction proceeds smoothly to give Pt-$Me(OC_6H_4CH_2CH{=}CH_2$-*o*)(dppp) (**5**) in 60% yield as a colorless solid (Eq. (3)).



(3)

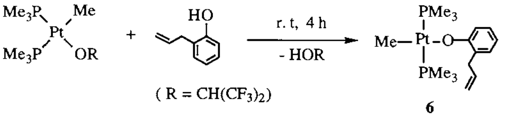
5

(P - P = $Ph_2P(CH_2)_3PPh_2$)

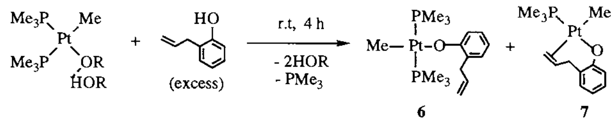
The ^1H-NMR spectrum of complex **5** shows the signal due to Pt-CH_3 hydrogen at 0.43 ppm as a doubles of doublets flanked with ^{195}Pt satellite signals. The ^1H- and $^{13}C\{^1H\}$-NMR signals due to the hydrogens and carbons of the allyl group appear at quite similar positions to the corresponding signals of the Pd complex **3**. The $^{31}P\{^1H\}$-NMR spectrum of **5** shows two doublets which are accompanied by ^{195}Pt satellites in agreement with the *cis* configuration having unequivalent phosphine ligands. The signal at 7.73 ppm with coupling constant, $J(^{195}Pt–^{31}P) = 1546$ Hz, is assigned to the phosphorus atom at the *trans* position to the methyl ligand, while the other phosphorus nuclei bans to the 2-allylphenoxido ligand shows a much larger coupling constant, $J(^{195}Pt–^{31}P) = 3807$ Hz, similarly to the already reported *cis* methylplatinum alkoxido complexes, *cis*-$PtMe(OPh)(PMe_3)_2$ [6]b, PtMe(OMe)(dppe) [13], and related Pt complexes [14].

Use of monodentate coordinating PMe_3 as the auxiliary ligands have altered the products of the reaction of the fluoroalkoxido platinum complexes with 2-allylphenol. Reaction of *cis*-$PtMe(OCH(CF_3)_2)(PMe_3)_2$ with equimolar 2-allylphenol at room temperature gives *trans*-$PtMe(OC_6H_4CH_2CH{=}CH_2$-*o*$)(PMe_3)_2$, (**6**) in a 55% yield as shown in Eq. (4). However, similar reaction of *cis*-$PtMe(OCH(CF_3)_2)(PMe_3)_2 \cdot (HOCH(CF_3)_2)$ with excess 2-allylphenol at room temperature results in formation of a mixture of **6** and Pt-$Me(OC_6H_4CH_2CH$

$=CH_2$-*o*)(PMe_3) (**7**) which are isolated by fractional recrystallization of the product in the respective yields of 16% and 28% as shown in Eq. (5).



(4)



(5)

The IR spectrum of **6** shows the presence of the alkenyl stretching vibration (ν(C=C)) in the phenoxido ligand at 1636 cm^{-1} similarly to **1**–**3** (1636–1634 cm^{-} 1), while the corresponding peak of **7** is shifted to lower frequency region (1600 cm^{-1}) by π-coordination to the Pt center. The ^1H spectrum of **6** at -20°C shows the signal due to $Pt-CH_3$ hydrogen as a triplet flanked with ^{195}Pt satellites. The PMe_3 hydrogen signal observed as an apparent triplet due to virtual coupling is also accompanied by ^{195}Pt satellites. The other signals including allyl hydrogen peaks of the 2-allylphenoxido ligand are observed quite similarly to the bans Pd complex **1**. The $^{31}P\{^1H\}$-NMR signal of **6** shows the $^{31}P-^{195}Pt$ coupling (2667 Hz) which is similar to the already reported monomethyl complexes of Pt(II). In contrast, the Pt–Me signal in ^1H- spectrum of **7** exhibits a doublet (J(PH) = 3 Hz) with ^{195}Pt satellites due to the structure having the methyl and PMe_3 ligands at mutually *cis* positions. The $^{13}C\{^1H\}$-NMR signals due to olefinic carbons of the 2-allylphenoxido ligand are observed at 79.2 and 97.8 ppm which are significantly at higher magnetic field position than the corresponding peaks of other Pd and Pt complexes in the present study and those of 2-allylphenol. ^{195}Pt satellite peaks are clearly observed, indicating that the C=C double bond is firmly π-coordinated to the metal center. The ^1H NMR signals corresponding to, $=CH_2$, =CH–, and $-CH_2-$ hydrogens of the 2-allylphenoxido ligand also shows splitting due to $^{195}Pt-^1H$ coupling and upfield shift of the peak positions compared with those of 6 whose C=C double bond is not bonded to the Pt center. Assignment of these ^1H and ^{13}C signals are confirmed with the aid of $^{13}C-^1H$ COSY measurement and the ^{13}C-NMR spectrum obtained in a gated decoupled mode. In order to obtain more precise structural information we have undertaken the study on the crystal structure.

A suitable crystal of **7** for X-ray analysis was obtained by recrystallization from a THF hexane mixture. Fig. 2 shows the molecular structure with the atomic numbering scheme. Selected data of bond lengths and angles are listed in Table 2. The molecule has slightly distorted square planar coordination around the Pt center which is bonded to a PMe_3, a methyl, and a 2-allylphenoxido group whose C=C double bond is coordinated to the metal center. The C2–C1 bond in 2-allylphenoxido ligand is perpendicularly coordinated to the coordination plane with almost equal Pt–C bond distances (Pt–C1 = 2.13 and Pt–C2 = 2.16 Å). The C2–C1 bond length of **7** is 1.32 Å, which resembles to free alkene (1.337 Å) [15], and shorter than the corresponding C=C double bond of Zeise's salt, $KPtCl_3(C_2H_4) \cdot H_2O$ (1.38 Å) [16] and of $Pt(C_2H_4)(PPh_3)_2$ (1.43 Å) [17]. Therefore, the weak π-back donation of metal to olefin and vice verse electron drift of olefin to metal is significant in **7**. The Pt-methyl bond length (2.05(2) Å) of **7** is shorter than those of *cis*-$PtMe(OCH(CF_3)_2)(PMe_3)_2$ (2.13(2) Å) and of *cis*-$PtMe(OPh)(PMe_3)_2$ (2.08(1) Å)[6]b. These results suggest the C=C double bond has much weaker *trans* influence than PMe_3 ligand.

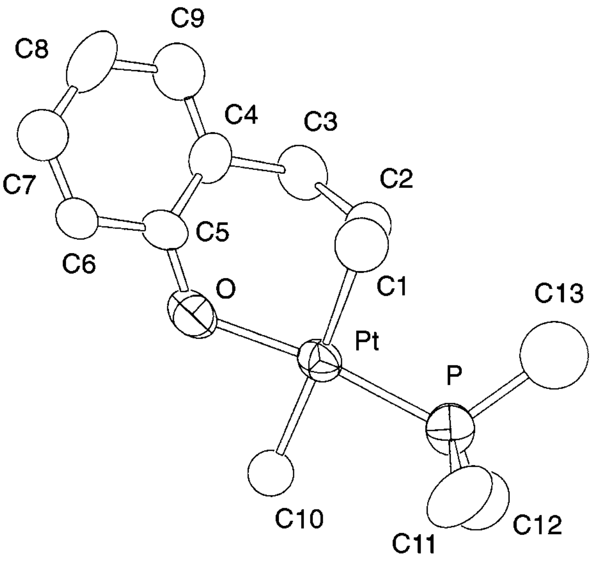


Fig. 2. ORTEP drawing of **7** showing the atomic labeling scheme and 30% probability thermal ellipsoids.

Table 2
Selected bond lengths (Å) and angles (°) for **7**

Pt–O	2.05(2)	C(1)–C(2)	1.32(4)
Pt–C(10)	1.91(4)	C(2)–C(3)	1.50(4)
Pt–P	2.211(9)	C(3)–C(4)	1.54(4)
Pt–C(1)	2.13(5)	C(4)–C(5)	1.35(4)
Pt–C(2)	2.16(4)	O–C(5)	1.32(4)
P–Pt–C(1)	95.1(10)	P–Pt–C(2)	98.2(8)
P–Pt–C(10)	86.8(9)	P–Pt–0(1)	172.1(7)
O–Pt–C(1)	90(1)	O–Pt–C(2)	89(1)
O–Pt–P	172.1(7)	O–Pt–C(10)	86(1)
C(1)–Pt–C(10)	163(1)	C(2)–Pt–C(10)	159(1)
Pt–C(2)–C(1)	70(2)	Pt–C(1)–C(2)	73(2)
Pt–C(2)–C(3)	108(2)	Pt–0(1)–C(5)	128(2)

In order to obtain more detailed insight for formation of **7** in the reaction (5) we have examined the reaction of isolated complex **6** with excess 2-allylphenol but observed complete recovery of the starting material. Warming a $CDCl_3$ solution of **6** at −20°C to room temperature leads to partial conversion of the complex into *cis*-isomer and ensuing formation of uncharacterized complexes in trace amounts. These results exclude a simple pathway from **6** to **7** involving the *cis* isomer of **6** as the intermediate. However, reaction of *cis*-PtMe($OCH(CF_3)_2$)(PEt_3)$_2$ with 2-allylphenol results in the sole formation of *cis*-PtMe($OC_6H_4CH_2CH{=}CH_2$-*o*)(PEt_3)$_2$ (**8**) which is isolated in 48% yield. At present formation mechanism of **7** in the reaction system is not clarified.

In summary, the attempts to synthesize methylpalladium(II) or -platinum(II) complexes with 2-allylphenoxido ligands provided the complexes whose allylphenoxido group is bonded in a simple *O*-bonded fashion. These complexes do not show coordination or interaction of the C=C double bond of the ligand to the fifth coordination site of the metal center. Ligand exchange reaction of the methylplatinum fluoroalkoxido complex by 2-allylphenol resulted in formation of the square-planar complex as one of the products, which possesses the phenoxido ligand containing C=C double bond π-coordinated to the metal center. The bond parameters of the olefinic group bonded to the Pt center suggests back donation in almost negligible degree. Chelating complexation of the 2-allylphenoxido ligand in **7** despite selective formation of simple *O*-coordinated phenoxides in the reactions of dimethylpalladium complexes can be partly attributed to more labile Pd–PMe_3 bond than the Pt–PMe_3 bond.

3. Experimental

3.1. General, materials, and measurement

All manipulations of air-sensitive compounds were performed under N_2 or argon atmosphere with use of standard Schlenk technique. Solvents were distilled from Nabenzophenone. dppp (1,3-bis(diphenylphosphino)propane), PMe_3, PPh_3, 2-allylphenol, 2,2,2-trifluoro-1-phenylethanol, and 1,1,1,3,3,3-hexafluoro-2-propanol were purchased from Aldrich and used without further purification. *cis*-$PdMe_2L_2$ (L = PMe_3, PPh_3) [18,19], *cis*-PtMe($OCH(CF_3)_2$)(PMe_3)$_2$ [6]b, *cis*-PtMe($OCH(CF_3)_2$)(PMe_3)$_2$ · ($HOCH(CF_3)_2$ [6]b, and PtMe($OCH(CF_3)_2$)(dppp) [20] were prepared by the literature methods.

Elemental analyses were carried out by the analytical laboratory, Tokyo Institute of Technology in Japan and Basic Science Institute of Korea. IR spectra were recorded on a Hitachi 270-30 spectrophotometer. NMR (^1H, $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$) spectra were obtained on JEOLFX-100, GX-270 and Braker 500 MHz spectrometers. Chemical shifts were referred to internal Me_4Si or to external 85% H_3PO_4.

$PdMe_2$(dppp) was obtained from ligand substitution reaction of $PdMe_2$(tmeda) [21] with equimolar dppp (90%). ^1H-NMR(200 MHz, $CDCl_3$, 6): 0.16(bd, 6H, CH_3), 1.85 (m, 2H, –CH_2), 2.4 (m, 4H, P–CH_2), 7.4–7.7 (m, 20H, aromatic). ^{31}P-NMR (40 MHz, $CDCl_3$, b): 6.3 (s). Anal. Calcd for $C_{29}H_{32}P_2Pd$: C, 63.46; H, 5.88. Found: C, 63.31; H, 5.74.

3.2. Preparation of trans-PdMe($OC_6H_4CH_2CH{=}CH_2$-o)L_2 (**1**: L = PMe_3; **2**: L = PPh_3; **3** L = 0.5dppp)

To an Et_2O (10 ml) solution of *cis*-$PdMe_2(PMe_3)_2$ (562 mg, 1.95 mmol) was added 2-allylphenol (309 mg, 2.30 mmol) at 0°C. After stirring the reaction mixture for 6 h the solvent was reduced under vacuum to cause separation of colorless crystals, which were recrystallized from ether to give **1** (475 mg, 62%). IR (KBr): 1636 cm^{-1} (ν(C=C)). ^1H-NMR ($CDCl_3$, 500 MHz, δ): 0.02 (t, 3H, $J = 7$ Hz, Pd–CH_3), 1.25 (t, 18H, $J = 3$ Hz, $P(CH_3)_3$), 3.37 (d, 2H, $J = 5$ Hz, –$CH_2CH{=}CH_2$), 4.97 (dd, 1H, $J = 10$, 2 Hz, –$CH_2CH{=}CH_2$), 5.02 (dd, 1H, $J = 17.2$ Hz, –$CH_2CH{=}CH_2$), 6.07 (m, 1H, –$CH_2CH{=}CH_2$), 6.4 (m, 1H, aromatic), 6.93 (m, 2H, aromatic), 7.05 (m, 1H, aromatic). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, δ): −12.7 (t, $J = 7$ Hz, Pd–CH_3), 13.1 (t, $P(CH_3)_3$), 35.1 (–$CH_2CH{=}CH_2$), 114.0 (–$CH_2CH{=}CH_2$), 139.1 (–$CH_2CH{=}CH_2$), 111.5, 118.5, 126.7, 128.4, 129.2, 167.9 (aromatic). $^{31}P\{^1H\}$-NMR (200 MHz, $CDCl_3$, δ): −13.5 (s). Anal. Calcd for $C_{16}H_{30}OP_2Pd$: C, 47.24; H, 7.43. Found: C, 47.30; H,7.51.

trans-PdMe($OC_6H_4CH_2CH{=}CH_2$-*o*)(PPh_3)$_2$, (**2**) and PdMe($OC_6H_4CH_2CH{=}CH_2$-*o*)(dppp), (**3**) were similarly obtained in 85 and 86% yields. Complex **2**. IR (KBr): 1634 cm^{-1} (ν(C=C)). ^1H-NMR ($CDCl_3$, 500 MHz, 6): 0.07 (bs, 3H, Pd–CH_3), 2.80 (d, 2H, $J = 5$ Hz, –$CH_2CH{=}CH_2$), 4.85 (m, 2H, –$CH_2CH{=}CH_2$), 5.77 (m, 1H, –$CH_2CH{=}CH_2$), 6.08–7.76 (m, aromatic). $^{13}C\{^1H\}$-NMR (125 MHz, $CDCl_3$, δ): −1.92 (s, Pd–CH_3), 35.1 (–$CH_2CH{=}CH_2$), 113.2 (–$CH_2CH{=}CH_2$), 139.5 (–$CH_2CH{=}CH_2$), 110.6, 119.4, 125.6, 126.8, 128.1, 129.9. 130.3, 131.4, 134.6, 166.5 (aromatic). $^{31}P\{^1H\}$-NMR (200 MHz, $CDCl_3$, δ): 26.8 (s). Anal. Calcd for $C_{46}H_{42}OP_2Pd$: C, 70.91; H, 5.43. Found: C, 71.34; H, 5.56.

Complex **3**. IR (KBr): 1634 cm^{-1} (ν(C=C)). ^1H-NMR ($CDCl_3$, 500 MHz, δ): 0.32 (dd, 3H, $J = 3$, 8 Hz, Pd–CH_3), 1.86 (m, 2H, P–CH_2), 2.43 (m, 4H, P–CH_2), 3.03 (d, 2H, $J = 5$ Hz, –$CH_2CH{=}CH_2$), 4.80 (dd, 1H,

$J = 10.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 4.89 (dd, 1H, $J = 17.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 5.86 (m, 1H, $-\mathrm{CH_2C}\mathit{H}\mathrm{{=}CH_2}$), 6.23 (m, 1H, aromatic), 6.77 (m, 2H, aromatic), 7.14–7.96 (m, 1H, aromatic). $^{13}\mathrm{C}\{^1\mathrm{H}\}$-NMR (125 MHz, $\mathrm{CDCl_3}$, δ): 14.2 (dd, $J = 3.97$ Hz, $\mathrm{Pd{-}}\mathit{C}\mathrm{H_3}$), 19.2 (d, $J = 6$, $\mathrm{P{-}}\mathit{C}\mathrm{H_2}$), 27.2 (dd, $J = 2.19$ Hz, $\mathrm{Pd{-}}\mathit{C}\mathrm{H_2}$), 29.4 (dd, $J = 8.30$ Hz, $\mathrm{P{-}CH_2}$), 35.7 ($-\mathit{C}\mathrm{H_2CH{=}CH_2}$), 113.0 ($-\mathrm{CH_2CH{=}}\mathit{C}\mathrm{H_2}$), 140.0 ($-\mathrm{CH_2}\mathit{C}\mathrm{H{=}CH_2}$). $^{31}\mathrm{P}\{^1\mathrm{H}\}$-NMR (200 MHz, $\mathrm{CDCl_3}$, δ): -6.85, 26.3 (d, $J = 47$). Anal. Calcd for $\mathrm{C_{37}H_{38}OP_2Pd}$: C, 66.62; H, 5.74. Found: C, 66.95; H, 5.51.

3.3. Preparation of *trans*-$\mathrm{PdMe(OC_6H_4CH_2CH{=}CH_2\text{-}\mathit{o})(PMe_3)_2 \cdot (HOCH(CF_3)Ph)}$ (**4**)

To an $\mathrm{Et_2O}$ (4 ml) solution of *trans*-$\mathrm{PdMe(OC_6H_4CH_2CH{=}CH_2\text{-}\mathit{o})(PMe_3)_2}$ (285 mg, 0.70 mmol) was added 2,2,2-trifluoro-1-phenylethanol (142 mg, 0.81 mmol) at room temperature. After stirring the reaction mixture for 2 h the solvent was evaporated to dryness to leave a pasty material. Addition of hexane (5 ml) and ensuing storage of the resulting solution overnight at −20°C caused separation of **4** as a colorless crystals which were collected by filtration, washed with hexane, and dried in vacuo (338 mg, 83%). IR (KBr): 1636 $\mathrm{cm^{-1}}$ (ν(C=C)). ^{1}H-NMR ($\mathrm{CDCl_3}$, 500 MHz, δ): 0.08 (t, 3H, $J = 7$ Hz, $\mathrm{Pd{-}C}\mathit{H}_3$), 1.20 (t, 18H, $J = 3$ Hz, $\mathrm{P(C}\mathit{H}_3)_3$), 3.38 (d, 2H, $J = 5$ Hz, $-\mathrm{C}\mathit{H}_2\mathrm{CH{=}CH_2}$), 4.97 (dd, 1H, $J = 10.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 5.05 (dd, 1H, $J = 17$, 2 Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 5.30 (q, 1H, $J = 7$ Hz, $\mathrm{C}\mathit{H}\mathrm{(CF_3)_2}$), 5.80 (bs, 1H, –OH), 6.05 (m, 1H, $-\mathrm{CH_2C}\mathit{H}\mathrm{{=}CH_2}$), 6.45 (m, 1H, aromatic), 6.97 (m, 2H, aromatic), 7.12 (m, 1H, aromatic), 7.40 (m, 4H, aromatic), 7.56 (m, 1H, aromatic). $^{13}\mathrm{C}\{^1\mathrm{H}\}$-NMR (125 MHz, $\mathrm{CDCl_3}$, δ): -12.1 (t, $J = 7$ Hz, $\mathrm{Pd{-}}\mathit{C}\mathrm{H_3}$), 12.9 (t, $\mathrm{P(C}\mathit{H}_3)_3$), 35.0 ($-\mathit{C}\mathrm{H_2CH{=}CH_2}$), 72.3 (q, $J = 7$ Hz), 114.3 ($-\mathrm{CH_2CH{=}}\mathit{C}\mathrm{H_2}$), 138.8 ($-\mathrm{CH_2}\mathit{C}\mathrm{H{=}CH_2}$), 112.9, 118.7, 123.8, 126.1, 126.7, 127.8, 128.3, 128.9, 136.2, 167.9 (aromatic). $^{31}\mathrm{P}\{^1\mathrm{H}\}$-NMR (200 MHz, $\mathrm{CDCl_3}$, δ): -13.6 (s). Anal. Calcd for $\mathrm{C_{24}H_{37}F_3O_2P_2Pd}$: C, 49.45; H, 6.40. Found: C, 49.46; H, 6.68.

3.4. Preparation of $\mathrm{PtMe(OC_6H_4CH_2CH{=}CH_2\text{-}\mathit{o})(dppp)}$, (**5**)

To an $\mathrm{Et_2O}$ (4 ml) solution of $\mathrm{PtMe(OCH(CF_3)_2)(dppp)}$ (273 mg, 0.35 mmol) was added 2-allylphenol (59 mg, 0.7 mmol) at room temperature. Stirring the reaction mixture for 4 h caused separation of a white solid which was collected by filtration, washed with hexane, and dried in vacuo to give 5 (156 mg, 60%). IR (KBr): 1636 $\mathrm{cm^{-1}}$ (ν(C=C)). ^{1}H-NMR ($\mathrm{CDCl_3}$, 500 MHz, δ): 0.43 (dd, 3H, $J = 3.16$ Hz J(PtH) = 54 Hz, $\mathrm{Pt{-}C}\mathit{H}_3$), 1.91 (m, 2H, $\mathrm{P{-}C}\mathit{H}_2$), 2.48–2.55 (m, 4H, $\mathrm{P{-}C}\mathit{H}_2$), 2.98 (d, 2H, $J = 7$ Hz, $-\mathrm{C}\mathit{H}_2\mathrm{CH{=}CH_2}$), 4.85 (dd, 1H, $J = 11.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 4.89 (dd, 1H, $J = 17.1$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 5.77 (m, 1H, $-\mathrm{CH_2C}\mathit{H}\mathrm{{=}CH_2}$), 6.36 (m, aromatic), 6.78–7.69 (m, aromatic). $^{13}\mathrm{C}\{^1\mathrm{H}\}$-NMR (125 MHz, $\mathrm{CDCl_3}$, δ): 10.2 (dd, $J = 6.92$ Hz, $\mathrm{Pt{-}}\mathit{C}\mathrm{H_3}$), 19.6 (d, $J = 2$ Hz, $\mathrm{P{-}}\mathit{C}\mathrm{H_2}$), 26.7 (d, $J = 27$ Hz, $\mathrm{Pd{-}}\mathit{C}\mathrm{H_2}$), 28.0 (dd, $J = 6.40$ Hz, $\mathrm{P{-}}\mathit{C}\mathrm{H_2}$), 34.9 ($-\mathit{C}\mathrm{H_2CH{=}CH_2}$), 113.6 ($-\mathrm{CH_2CH{=}}\mathit{C}\mathrm{H_2}$), 139.3 ($-\mathrm{CH_2}\mathit{C}\mathrm{H{=}CH_2}$). $^{31}\mathrm{P}\{^1\mathrm{H}\}$-NMR (200 MHz, $\mathrm{CDCl_3}$, δ): 0.06, 7.73 (d, $J = 20$ Hz, J(PtP) = 3807, 1546 Hz). Anal. Calcd for $\mathrm{C_{37}H_{38}OP_2Pt}$: C, 58.80; H, 5.07. Found: C, 58.63; H, 4.89.

3.5. Preparation of *trans*-$\mathrm{PtMe(OC_6H_4CH_2CH{=}CH_2\text{-}\mathit{o})(PMe_3)_2}$, (**6**) and $\mathrm{PtMe(OC_6H_4CH_2CH{=}CH_2\text{-}\mathit{o})(PMe_3)}$, (**7**)

To an $\mathrm{Et_2O}$ (5 ml) solution of *cis*-$\mathrm{PtMe(OCH(CF_3)_2)(PMe_3)_2}$ (264 mg, 0.499 mmol) was added 2-allylphenol (80 mg, 0.598 mmol) at room temperature. Stirring the reaction mixture for 4 h caused separation of a white solid which was filtered, washed with hexane, and dried in vacuo to give **6** (136 mg, 55%). IR (KBr): 1636 $\mathrm{cm^{-1}}$ (ν(C=C)). ^{1}H-NMR ($\mathrm{CDCl_3}$, 500 MHz, δ): 0.27 (t, 3H, $J = 7$ Hz, J(PtH) = 79 Hz, $\mathrm{Pt{-}C}\mathit{H}_3$), 1.33 (t, 18H, $J = 3$ Hz, J(PtH) = 27 Hz, $\mathrm{P(C}\mathit{H}_3)_3$), 3.35 (d, 2H, $J = 5$ Hz, $-\mathrm{C}\mathit{H}_2\mathrm{CH{=}CH_2}$), 4.98 (dd, 1H, $J = 10.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 5.02 (dd, 1H, $J = 17.2$ Hz, $-\mathrm{CH_2CH{=}C}\mathit{H}_2$), 6.07 (m, 1H, $-\mathrm{CH_2C}\mathit{H}\mathrm{{=}CH_2}$), 6.43 (m, 1H, aromatic), 6.98 (m, 2H, aromatic), 7.33 (m, 1H, aromatic). $^{13}\mathrm{C}\{^1\mathrm{H}\}$-NMR (125 MHz, $\mathrm{CDCl_3}$, δ): -29.9 (t, $J = 6$ Hz, $\mathrm{Pt{-}}\mathit{C}\mathrm{H_3}$), 12.2 (t, $J = 18$ Hz, $\mathrm{P(C}\mathit{H}_3)_3$), 35.6 (s, $-\mathit{C}\mathrm{H_2CH{=}CH_2}$), 113.7 ($-\mathrm{CH_2CH{=}}\mathit{C}\mathrm{H_2}$), 139.2 ($-\mathrm{CH_2}\mathit{C}\mathrm{H{=}CH_2}$), 111.8, 117.5, 126.6, 128.4, 129.3, 168.0 (aromatic). $^{31}\mathrm{P}\{^1\mathrm{H}\}$-NMR (200 MHz, $\mathrm{CDCl_3}$, δ): -7.79 (s, J(PtP) = 2667 Hz). Anal Calcd for $\mathrm{C_{16}H_{30}OP_2Pt}$: C, 38.79; H, 6.10. Found C, 38.49; H, 6.25.

To an ether solution (10 ml) containing *cis*-$\mathrm{PtMe(OR)(PMe_3)_2 \cdot (HOR)}$ ($\mathrm{R = CH(CF_3)_2}$) (0.703 g, 1.01 mmol) was added 2-allylphenol (0.284 g, 2.12 mmol) at room temperature. After stirring the reaction mixture for 4 h the solvent was evaporated to dryness to leave a pasty material. Addition of hexane (7 ml) and ensuing storage of the resulting solution at −70°C for overnight caused separation of a colorless solid, white was collected by filtration, and washed with hexane (2 ml × 2), ether (3 ml × 2) at 0°C to afford **7** (118 mg, 28%). IR (KBr): 1600 $\mathrm{cm^{-1}}$ (ν(C=C)). ^{1}H-NMR ($\mathrm{CDCl_3}$, 500 MHz, δ): 1.05 (d, 3H, $J = 3$ Hz, J(PtH) = 68 Hz, $\mathrm{Pt{-}C}\mathit{H}_3$), 1.53 (bd, 9H, $J = 11$ Hz, J(PtH) = 39

Hz, $P(CH_3)_3$), 3.18 (d, 1H, $J = 18$ Hz, $-CH_2CH{=}CH_2$), 3.31 (d, 1H, $J = 15$ Hz, $-CH_2CH{=}CH_2$), 4.00 (bd, 2H, $-CH_2CH{=}CH_2$), 5.05 (m, 1H, $-CH_2CH{=}CH_2$), 6.54 (m, 1H, aromatic), 6.83 (m, 1H, aromatic), 6.91 (m, 1H, aromatic), 7.12 (m, aromatic). $^{13}C\{^1H\}$-NMR (125 MHz, $CDCl_3$, δ): 11.0 (d, $J = 9$ Hz, Pt$-CH_3$), 13.9 (d, $J = 43$ Hz, $J(PtC) = 43$ Hz, $P(CH_3)_3$), 35.2 (s, $J(PtC) = 23$ Hz, $-CH_2CH{=}CH_2$), 79.2 (s, $J(PtC) = 46$ Hz, $-CH_2CH{=}CH_2$), 97.8 (s, $J(PtC) = 46$ Hz, $-CH_2CH{=}CH_2$), 115.2, 120.3, 121.8, 128.9, 130.3, 162.6 (aromatic). $^{31}P\{^1H\}$-NMR (200 MHz, $CDCl_3$, δ): 29.6 (s, $J(PtP) = 3826$ Hz). Anal. Calcd for $C_{13}H_{21}OPPt$: C, 37.23; H, 5.06. Found: C, 37.25; H, 5.09.

Cooling the filtrate obtained by separation of **7** at $-70°C$ caused separation of complex **6** as a colorless solid (80 mg, 16%).

3.6. Preparation of *cis*-$PtMe(OC_6H_4CH_2CH{=}CH_2\text{-}o)(PEt_3)_2$, (**8**)

To an ether solution (5 ml) containing *cis*-$PtMe_2(PEt_3)_2$ (0.486 g, 1.05 mmol) was added 1,1,1,3,3,3-hexafluoro-2-propanol (83 mg, 0.49 mmol) at room temperature. After stirring the reaction mixture for 6 h the solvent was evaporated to dryness to leave a colorless oily material. It was dissolved in an ether (5 ml) solution of 2-allylphenol (66 mg, 0.498 mmol). Stirring the solution was continued for 12 h at room temperature, and then the solvent was fully evaporated to give an oily material. Addition of hexane (5 ml) to the product and ensuing storage of the resulting solution at $-20°C$ overnight caused separation of a white solid which was collected by filtration and washed with hexane (3 ml × 2) to give **8** (0.138 g, 48%). IR (KBr): 1634 cm^{-1} ($\nu(C{=}C)$). ^1H-NMR ($CDCl_3$, 500 MHz, δ): 0.51 (dd, 3H, $J = 3.7$ Hz $J(PtH) = 49$ Hz, Pt$-CH_3$), 1.16 (m, 9H, $P(CH_2CH_3)_3$), 1.80 (m, 6H, $P(CH_2CH_3)_3$), 3.48 (d, 2H, $J = 7$ Hz, $-CH_2CH{=}CH_2$), 5.01 (dd, 1H, $J = 9.1$ Hz, $-CH_2CH{=}CH_2$), 5.10 (dd, 1H, $J = 17.2$ Hz, $-CH_2CH{=}CH_2$), 6.12 (m, 1H, $-CH_2CH{=}CH_2$), 6.50 (m, aromatic), 7.00 (m, aromatic). $^{13}C\{^1H\}$-NMR (125 MHz, $CDCl_3$ δ): 5.02 (dd, $J = 8.92$ Hz, Pt$-CH_3$), 8.39 (d, $P(CH_2CH_3)_3$), 15.1 (d, $J = 24$ Hz, $P(CH_2CH_3)_3$), 16.7 (d, $J = 3$, $J(PtC) = 40$ Hz, $P(CH_2CH_3)_3$), 34.9 (s, $-CH_2CH{=}CH_2$), 113.9 ($-CH_2CH{=}CH_2$), 139.6 ($-CH_2CH{=}CH_2$), 112.7, 119.5, 126.1, 128.1, 130.1, 165.8. $^{31}P\{^1H\}$-NMR (200 MHz, $CDCl_3$, δ): 2.79, 22.3 (d, $J = 10$ Hz, $J(PtP) = 3869$, 1722 Hz). Anal. Calcd for $C_{22}H_{42}OP_2Pt$: C, 45.59; H, 7.30. Found: C, 45.56; H, 7.41.

3.7. X-ray structure determination

Table 3
Crystallographic Data for **4** and **7**

Compound	**4**	**7**
Formula	$C_{24}H_{37}O_2P_2F_3Pd$	$C_{13}H_{21}OPPt$
Formula weight	582.90	419.37
Crystal size (mm)	0.4 × 0.5 × 0.5	0.2 × 0.7 × 0.7
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$ (no. 14)	$P2_1/n$ (no.14)
a (Å)	10.813(7)	11.602(6)
b (Å)	16.872(10)	17.290(7)
c (Å)	15.661 (7)	7.424(4)
β (°)	97.71(4)	108.55(3)
V (Å^3)	2831	1411
D_{calcd} (g cm^{-3})	1.367	1.973
Z	4	4
μ (cm^{-1})	7.93	101.32
$F(000)$	1200	800
No. of variables	293	130
$2\theta_{max}$ (°)	55.0	55.0
Unique reflections	6718	3358
Used reflections ($I \geq 3\sigma(I)$)	4393	1522
$R(F_o)$[a]	0.045	0.098
Rw (F_o)[a]	0.042	0.086

[a] $R = \Sigma \| F_o| - |F_c\|) / \Sigma |F_o|$, $Rw = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w(F_o)^2]^{1/2}$ where $w = [\{\sigma(F_o)\}^2]^{-1}$.

Crystals of **4** and **7** suitable for X-ray crystallography were obtained by recrystallization from Et_2O-hexane and THF-hexane mixtures, respectively, and mounted in glass capillary tubes under argon. The unit cell parameters were obtained by least-squares refinement of setting angles of 20 reflections with $20 \leq 2\theta \leq 30°$. Intensities were collected on a Rigaku AFC-SR automated four-cycle diffractometer by using Mo-K_α radiation ($\lambda = 0.71069$ Å) and the ω-2θ method. Calculations were carried out by using a program package TEXSAN on a DEC Micro VAXII computer. A full matrix least-squares refinement was carried out by applying anisotropic thermal factors to all the non-hydrogen atoms. Hydrogen atoms were located from calculation by assuming the ideal positions ($d(C-H) = 0.95$ Å) and included the structure calculation without further refinement of the parameters. Absorption correction by ψ scan method of the collected data was applied. Crystallographic data and atomic coordinates of the non-hydrogen atoms are listed in Tables 3–5. Hydrogen atoms of **4** were located by calculation assuming the ideal geometry ($d(CH) = 0.95$ Å) and fixed throughout the structural calculation except for the OH hydrogen which was located in final D-map and refined isotropically. Hydrogen atoms of **7** were located by calculation. C1, C2 and C13 were refined isotropically, whereas the other non-hydrogen atoms were refined anisotropically. Insufficient convergence of the structural calculation of **7** is due to the anisotropic shape of the crystal with a large absorption

Table 4
Atomic coordinates and isotropic temperature factors for **4**

Atom	x	y	z	B_{eq}
Pd	0.77570(4)	0.35260(3)	0.79734(3)	3.719(9)
P(1)	0.7640(1)	0.3991(1)	0.93451(10)	4.88(4)
P(2)	0.7724(1)	0.31118(10)	0.65706(9)	4.78(4)
F(1)	0.9914(4)	0.1161(3)	0.7662(3)	11.1(2)
F(2)	0.9881(4)	0.0418(3)	0.8751(3)	9.8(1)
F(3)	0.8279(4)	0.0529(3)	0.7814(2)	9.1(1)
0(1)	0.9724(3)	0.3371(2)	0.8249(2)	4.38(10)
0(2)	0.9771(4)	0.2038(3)	0.9129(3)	7.0(1)
C(1)	0.5835(4)	0.3622(4)	0.7755(4)	6.3(2)
C(2)	1.0502(4)	0.3942(3)	0.8116(3)	3.7(1)
C(3)	1.1807(5)	0.3854(3)	0.8340(3)	4.2(1)
C(4)	1.2584(5)	0.4467(4)	0.8185(4)	5.3(2)
C(5)	1.2160(6)	0.5177(4)	0.7815(4)	5.6(2)
C(6)	1.0896(6)	0.5271(3)	0.7612(4)	5.2(2)
C(7)	1.0078(5)	0.4675(3)	0.7761(3)	4.2(1)
C(8)	1.2337(5)	0.3101(4)	0.8741(4)	5.5(2)
C(9)	1.2597(8)	0.2467(5)	0.8151(6)	8.7(3)
C(10)	1.2671(8)	0.2507(6)	0.7369(6)	10.0(3)
C(11)	0.7310(8)	0.5033(4)	0.9385(5)	10.8(3)
C(12)	0.9040(6)	0.3897(5)	1.0074(4)	9.9(3)
C(13)	0.6501(7)	0.3561(6)	0.9945(4)	10.6(3)
C(14)	0.7335(6)	0.3869(4)	0.5753(4)	7.5(2)
C(15)	0.9187(6)	0.2737(4)	0.6322(4)	7.4(2)
C(16)	0.6647(7)	0.2319(4)	0.6235(4)	8.3(2)
C(17)	0.8753(5)	0.1625(3)	0.8728(4)	5.0(2)
C(18)	0.9219(7)	0.0941(5)	0.8243(5)	7.0(2)
C(19)	0.7901(6)	0.1343(3)	0.9342(4)	4.8(2)
C(20)	0.8378(6)	0.1082(4)	1.0149(4)	6.6(2)
C(21)	0.7573(10)	0.0786(1)	1.0694(5)	8.7(3)
C(22)	0.6323(10)	0.0779(5)	1.0443(6)	8.9(3)
C(23)	0.5847(7)	0.1046(5)	0.9656(6)	8.4(3)
C(24)	0.6640(6)	0.1328(4)	0.9108(4)	6.5(2)
H(1)	0.979(4)	0.259(4)	0.884(3)	11.0(1)

coefficient. Atomic coordinates of hydrogen atoms and all bond distances and angles are available from the author (Y.J.K.).

Table 5
Atomic coordinates and isotropic temperature factors for **7**

Atom	x	y	z	B_{eq}
Pt	0.0323(1)	0.21920(9)	0.3667(2)	3.52(3)
P	−0.0380(8)	0.1663(7)	0.081(1)	4.4(2)
O	0.114(2)	0.256(2)	0.641(3)	4.9(7)
C(1)	0.030(3)	0.334(3)	0.265(5)	5.4(8)
C(2)	−0.072(3)	0.325(2)	0.307(4)	4.8(9)
C(3)	−0.093(2)	0.352(2)	0.485(4)	4.6(9)
C(4)	0.018(3)	0.376(2)	0.653(4)	3.7(9)
C(5)	0.111(3)	0.325(2)	0.713(4)	3.6(9)
C(6)	0.213(3)	0.345(2)	0.870(4)	3.7(8)
C(7)	0.219(3)	0.416(3)	0.952(5)	4.0(1)
C(8)	0.126(4)	0.469(3)	0.885(5)	5.0(1)
C(9)	0.023(3)	0.450(3)	0.730(5)	5.0(1)
C(10)	0.081(3)	0.118(2)	0.460(4)	4.0(7)
C(11)	0.084(3)	0.141(3)	−0.013(5)	6.0(1)
C(12)	−0.125(3)	0.082(3)	0.059(5)	6.0(1)
C(13)	−0.151(4)	0.226(3)	−0.107(6)	8.0(1)

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