Organometallics **1993,** *12,* 940–943

Notes

Different Behavior of $(NBu_4)[M(C_6F_5)_2(acac)]$ (M = Pd, Pt) toward $AgClO_4$. X-ray Crystal Structures of $(NBu_4)[M_2Ag(C_6F_5)_4(acac)_2]$ (M = Pd, Pt)

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Received July 13, 1992

Summary: The reactions between $(NBu_4)[M(C_6F_5)_2(acac)]$ (M = Pd (1), Pt (2)) and $AgClO_4$ (molar ratio 2:1) yield the polynuclear derivatives $(NBu_4)[M_2Ag(C_6F_5)_4(acac)_2]$ (M = Pd (3), Pt (4)). The structures of both complexes have been established by X-ray diffraction methods. For the palladium compound, two $Pd(C_6F_5)_2(acac)$ fragments are bonded to the silver center through the C^3 atom of the acetylacetonate, while in the platinum complex the $Pt(C_6F_5)_2(acac)$ fragments are connected to the silver atom by forming two Pt–Ag bonds. Crystal data for 3: monoclinic; space group $P2/c$; $a = 9.578$ (2), $b = 9.253$ (2), $c = 31.261$ (6) Å; $\beta = 96.70$ (3)°, $V = 2751.8$ (10) Å^3, $Z = 2$, $\mu = 1.108$ mm^{-1}; 3102 observed reflections ($F > 4\sigma(F)$) for 332 parameters; $R = 0.042$, $R_w = 0.046$, gof = 0.878. Crystal data for $4{\cdot}CH_2Cl_2$: monoclinic; space group $P2_1/c$; $a = 15.128$ (3), $b = 19.414$ (4), $c = 19.832$ (4) Å; $\beta = 100.02$ (3)°, $V = 5736$ (2) Å^3, $Z = 4$, $\mu = 5.404$ mm^{-1}; 7564 observed reflections ($F > 4\sigma(F)$) for 732 parameters; $R = 0.040$, $R_w = 0.044$, gof = 1.043.

Introduction

Anionic perhalophenyl (C_6F_5 or C_6Cl_5) platinum complexes behave as Lewis bases and react with silver derivatives yielding, in most cases, polynuclear complexes containing Pt–Ag bonds.[1,2] However, one of the facts which militates against the stabilization of these Pt–Ag bonds is the concomitance of arylating processes—i.e., the transfer of C_6X_5 groups to the silver center, which yields AgC_6X_5 or AgC_6X_5L and a less arylated platinum substrate instead of the heteronuclear derivatives. For instance, $(NBu_4)_2[Pt(C_6Cl_5)_4]$ reacts with $AgOClO_3L$ yielding AgC_6Cl_5 and $(NBu_4)[Pt(C_6Cl_5)_3L]$ (L = tht, PPh_3).[3]

As far as the homologous anionic palladium substrates are concerned, they are, as expected, more labile than the platinum ones, and they display a stronger arylating capability. Thus, heretofore, we have not succeeded in preparing similar polynuclear complexes containing Pd–Ag bonds, since transarylation processes take place.

In the course of our current research[4,5] we have synthesized (acetylacetonato)palladium and -platinum complexes. We thought that it would be worthwhile to explore the reactivity of $(NBu_4)[M(C_6F_5)_2(acac)]$ (M = Pd (1), Pt (2)) toward silver derivatives since the former contain one anionic chelating ligand blocking two coordination sites and a lower number of C_6F_5 groups than other anionic palladate or platinate complexes. In this case, one should expect a decrease of the number of undesirable reaction pathways and, for that reason, a better chance for synthesizing not only Pt–Ag but also Pd–Ag polynuclear complexes. In this paper we report the study of the reactions between 1 and 2 and $AgClO_4$ in a 2:1 molar ratio.

Experimental Section

Synthesis. Solvents were dried and distilled by standard methods before use. Infrared spectra were recorded with a Perkin-Elmer 883 spectrophotometer, and NMR spectra, with a Varian XL 200 spectrometer. $(NBu_4)[Pd(C_6F_5)_2(acac)]$ was prepared by literature methods,[4,5] and the analogous platinum derivative was prepared in a similar way. All the reactions were carried out with exclusion of light. *Safety note*: Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small amounts of these materials should be prepared, and they should be handled with great caution.

Preparation of Complexes 3 and 4. $(NBu_4)[Pd_2Ag(C_6F_5)_4(\eta^3\text{-}\mu^2\text{-}acac)_2]$ (3). To a CH_2Cl_2 (30 mL) solution of $(NBu_4)[Pd(C_6F_5)_2(acac\text{-}O,O')]$ (0.1828 g, 0.2337 mmol) was added $AgClO_4$ (0.0242 g, 0.117 mmol); the mixture was stirred at room temperature for 2 h. The resulting solution was evaporated to dryness, and the residue was treated with diethyl ether (20 mL). After filtration of the insoluble $(NBu_4)(ClO_4)$, the ether solution was evaporated to dryness and the residue was washed with *n*-hexane (20 mL), yielding a white solid (0.1243 g, 74% yield). NMR data (ppm, $CDCl_3$): ^1H NMR δ 5.39 (s, 2H, CH acac), 2.10 (s, 12H, CH_3 acac), 0.98, 1.40, 1.60, 3.10 (m, 8H, 8H, 8H, 12H, NBu_4^+); ^{19}F NMR $\delta(F_o)$: −116.17, $\delta(F_m)$ −164.67, $\delta(F_p)$ −161.87. Anal. Calc (found) for $C_{50}H_{50}F_{20}NO_4Pd_2Ag$: C, 42.00 (42.40); H, 3.52 (3.77); N, 0.98 (0.98).

$(NBu_4)[Pt_2Ag(C_6F_5)_4(acac)_2]$ (4). Complex 4 was prepared similarly from $(NBu_4)[Pt(C_6F_5)_2(acac\text{-}O,O')]$ (0.3648 g, 0.4190 mmol) and $AgClO_4$ (0.0434 g, 0.210 mmol). Yield: 0.2021 g, 61%. NMR data (ppm, $CDCl_3$): ^1H NMR δ 5.42 (s, 2H, CH acac), 1.87 (s, 12H, CH_3 acac), 0.98, 1.40, 1.60, 3.10 (m, 8H, 8H, 8H, 12H, NBu_4^+); ^{19}F NMR $\delta(F_o)$ −120.73, $\delta(F_m)$ −165.12, $\delta(F_p)$ −163.19; $^3J_{Pt\text{-}F_o}$ = 523 Hz. Anal. Calc (found) for $C_{50}H_{50}F_{20}NO_4Pt_2Ag$: C, 37.37 (37.50); H, 3.13 (3.13); N, 0.87 (0.93).

Crystal Structure Determination. Relevant crystallographic parameters are given in Table I. Selected atomic coordinates are presented in Tables II and III. X-ray-quality crystals of 3 and $4{\cdot}CH_2Cl_2$ were grown by slow diffusion of *n*-hexane into a solution of the respective compound in CH_2Cl_2. Data were collected in a Siemens STOE/AED-2 four-circle diffractometer at room temperature (range $4 \le 2\theta \le 50°$).

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Table I. Crystallographic Data Collection for Complexes 3 and 4·CH$_2$Cl$_2$

	3	4·CH$_2$Cl$_2$
chem formula	$C_{50}H_{50}F_{20}NO_4$-Pd_2Ag	$C_{51}H_{52}F_{20}NO_4Cl_2$-$Pt_2Ag$
fw	1429.6	1606.98
cryst syst; space group	monoclinic; $P2/c$	monoclinic; $P2_1/c$
a, Å	9.578 (2)	15.128 (3)
b, Å	9.253 (2)	19.414 (4)
c, Å	31.261 (6)	19.832 (4)
β, deg	96.70 (3)	100.02 (3)
V, Å^3	2751.8 (10)	5736 (2)
Z	2	4
D_{calc}, g·cm^{-3}	1.696	1.901
scan method	ω	θ/ω
obsd reflcns [$F > 4\sigma(F)$]	3102	7564
no. of params	332	732
μ, mm^{-1}	1.108	5.404
radiat (λ, Å)	Mo Kα (0.710 73)	Mo Kα (0.710 73)
g	0.0029	0.0015
R, R_w, Δ/σ, gof	0.042, 0.046, 0.008, 0.878	0.040, 0.044, 0.099, 1.043
highest resid peak, e Å^{-3}	0.6	0.9

Table II. Selected Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Coefficients (Å^2 × 10^3) for (NBu$_4$){[Pd(C$_6$F$_5$)$_2$(acac)]$_2$Ag} (3)

	x	y	z	U(eq)
Pd1	950 (1)	1722 (1)	916 (1)	54 (1)
Ag	0 (0)	0 (0)	0 (0)	69 (1)
O1	−128 (4)	3206 (4)	496 (2)	62 (2)
O2	−987 (5)	754 (5)	973 (2)	66 (2)
C1	−1865 (7)	686 (7)	648 (3)	61 (2)
C2	−1715 (7)	1315 (7)	236 (2)	59 (2)
C3	−1055 (6)	2721 (7)	217 (2)	58 (2)
C4	−3230 (8)	−169 (9)	688 (3)	83 (3)
C5	−1573 (8)	3714 (9)	−159 (3)	78 (3)

Accurate lattice parameters were determined from accurate positions of 48 reflections ($25 \leq 2\theta \leq 32°$) for 3 and 46 reflections ($26 \leq 2\theta \leq 32°$) for 4·CH$_2$Cl$_2$, including Friedel pairs. Intensity data were corrected for Lorentz and polarization effects. The structures were solved by the use of Patterson and Fourier methods. All calculations were carried out with SHELXTL-PLUS.[6] (Weighting scheme applied: $w^{-1} = \sigma^2(F) + g(F^2)$.)

Structure of 3. Five carbon atoms of the NBu$_4^+$ group are disordered over two sites each. The disorder remains when the symmetry is lowered to Pc. H atoms of the CH$_2$ groups in the NBu$_4^+$ cation were geometrically calculated and refined with a common thermal parameter (0.1362 Å^2). Attempts to locate H atoms of the CH$_3$ groups were unsuccessful. The position of the H atom bonded to C(γ) in the acac group was determined from the difference map (thermal parameter 0.05913 Å^2).

Structure of 4·CH$_2$Cl$_2$. Absorption corrections were applied[7] (rescaled max and min transmission factors: 1.291, 0.713). The C and Cl atoms of the solvent molecule were refined with full occupancy and anisotropic displacement parameters. No interaction has been observed between the solvent molecule and the other atoms. Only the H atom of C(γ) of the acac groups could be located and successfully refined.

Results and Discussion

(NBu$_4$)[M(C$_6$F$_5$)$_2$(acac-*O,O'*)] (M = Pd (1), Pt (2)) reacts with AgClO$_4$ (2:1 molar ratio) in CH$_2$Cl$_2$ at 25 °C, yielding analogous compounds of similar stoichiometry (NBu$_4$)[M$_2$Ag(C$_6$F$_5$)$_4$(acac)$_2$] (M = Pd (3, 74%), Pt (4, 61%)).

Table III. Selected Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Coefficients (Å^2 × 10^3) for (NBu$_4$){[Pt(C$_6$F$_5$)$_2$(acac)]$_2$Ag}·CH$_2$Cl$_2$ 4·CH$_2$Cl$_2$)

	x	y	z	U(eq)
Pt1	4803 (1)	733 (1)	2209 (1)	43 (1)
Pt2	8347 (1)	329 (1)	2520 (1)	43 (1)
Ag	6573 (1)	498 (1)	2354 (1)	62 (1)
C13	5206 (6)	102 (6)	3588 (5)	64 (4)
C14	5263 (7)	−555 (6)	4032 (5)	77 (4)
C15	5423 (7)	748 (6)	3890 (5)	69 (4)
C16	5399 (6)	1382 (5)	3561 (5)	58 (3)
C17	5688 (7)	2017 (6)	3978 (5)	81 (4)
C30	8224 (6)	1264 (5)	1326 (5)	58 (3)
C31	8319 (9)	1999 (6)	1066 (6)	95 (5)
C32	8020 (7)	711 (6)	885 (4)	70 (4)
C33	7903 (6)	35 (5)	1044 (4)	56 (3)
C34	7625 (9)	−496 (7)	459 (5)	89 (5)
O1	4952 (4)	−10 (3)	2956 (3)	63 (2)
O2	5156 (4)	1490 (3)	2927 (3)	57 (2)
O3	7968 (4)	−230 (3)	1633 (3)	55 (2)
O4	8367 (4)	1234 (3)	1983 (3)	60 (2)

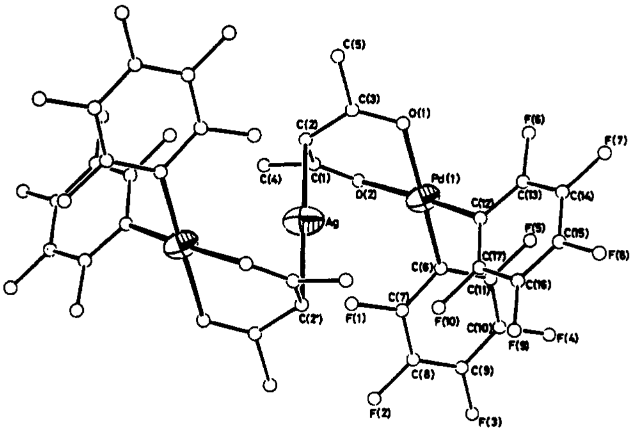

Figure 1. ORTEP drawing of the anion of 3.

$$2(NBu_4)[M(C_6F_5)_2(acac)] + AgClO_4 \rightarrow (NBu_4)[M_2Ag(C_6F_5)_4(acac)_2] + (NBu_4)(ClO_4) \quad (1)$$

However, in spite of the analogous stoichiometries for 3 and 4, some interesting differences in the IR and ^{13}C NMR data related to the acac ligand in the two derivatives can be observed when compared with the corresponding data of the respective starting materials (see Table IV). This fact indicates a rather different structural role of the acetylacetonato ligands in 3 and 4, in accord with the results of X-ray diffraction analyses.

X-ray Structure Analysis of (NBu$_4$)[Pd$_2$Ag(C$_6$F$_5$)$_4$(η^3-μ^2-acac)$_2$] (3). A drawing of the anion in 3 is shown in Figure 1. Selected bond distances and angles are collected in Table V. In this polynuclear compound two anionic fragments [(C$_6$F$_5$)$_2$Pd(acac-*O,O'*)]$^-$ are bonded to the silver center through the C^3 atom of the (acetylacetonato-*O,O'*)palladium (Ag–C(2) = 2.237 (7) Å). The palladium atoms are in almost square planar environments, and the silver atom displays a linear coordination. As a consequence of the C^3–Ag bond, the C^3 atom is using sp^3 orbitals for bonding, and the acetylacetonato–palladium ring loses planarity and adopts a boat conformation. The angles around the C^3 atom are rather different, ranging from 97.7 (4) to 119.3 (6)°, although the average (109.02°) is close to typical angles formed by sp^3 orbitals. Similar Ag–C distances and ring deformations have been found in

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[6] SHELXTL-PLUS. Software Package for the Determination of Crystal Structure, Release 4.0. Siemens Analytical X-Ray Instruments, Inc., Madison, WI, 1990.

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[8] Maslowsky, E., Jr. *Vibrational Spectra of Organometallic Compounds*; Wiley: New York, 1977; p 84.

Table IV. Comparison of IR and ^{13}C NMR Relevant Parameters (cm^{-1}, ppm)

	ν(CO)	Π(CH)	X-sens[a]	δ(C^3)	δ(CO)
$(NBu_4)[Pd(C_6F_5)_2(acac)]$ (1)	1587, 1520	770	795, 785	99.40	185.86
$(NBu_4)[Pd_2Ag(C_6F_5)_4(acac)_2]$ (3)	1600		800, 790	84.05	193.51
$(NBu_4)[Pt(C_6F_5)_2(acac)]$ (2)	1580, 1520	780	815, 800	101.34	183.16
$(NBu_4)[Pt_2Ag(C_6F_5)_4(acac)_2]$ (4)	1570, 1530	795	815, 805	101.89	185.39

[a] Absorptions assigned to the X-sensitive mode of the C_6F_5 groups.[8] In all cases these two absorptions indicate that both C_6F_5 groups are in cis positions.

Table V. Selected Bond Distances and Angles for $(NBu_4)[Pd_2Ag(C_6F_5)_4(acac)_2]$ (3)

		Distances (Å)			
Pd–C(6)	1.985 (6)	Pd–C(12)	1.990 (6)	Pd–O(1)	2.088 (4)
Pd–O(2)	2.087 (5)	Ag–C(2)	2.237 (7)	C(3)–O(1)	1.253 (8)
C(1)–O(2)	1.243 (8)	C(3)–C(2)	1.451 (9)	C(2)–C(1)	1.436 (10)
		Angles (deg)			
C(12)–Pd–C(6)	88.9 (3)	O(1)–Pd–C(12)	91.3 (2)	O(1)–Pd–O(2)	87.5 (2)
O(2)–Pd–C(6)	92.3 (2)	C(3)–C(2)–Ag	97.7 (4)	C(1)–C(2)–Ag	103.3 (4)
C(1)–C(2)–C(3)	119.3 (6)	O(2)–C(1)–C(2)	125.8 (6)	O(1)–C(3)–C(2)	124.7 (6)
Pd–O(2)–C(1)	118.3 (5)	Pd–O(1)–C(3)	117.4 (4)		

Table VI. Selected Bond Distances and Angles for $(NBu_4)[Pt_2Ag(C_6F_5)_4(acac)_2]\cdot CH_2Cl_2$ ($4\cdot CH_2Cl_2$)

		Distances (Å)			
Pt(1)–C(1)	1.998 (8)	Pt(1)–C(7)	2.009 (8)	Pt(2)–C(18)	2.000 (7)
Pt(2)–C(24)	2.003 (8)	Pt(1)–O(1)	2.053 (6)	Pt(1)–O(2)	2.051 (6)
Pt(2)–O(3)	2.060 (5)	Pt(2)–O(4)	2.057 (6)	Pt(1)–Ag	2.681 (1)
Pt(2)–Ag	2.668 (1)	C(16)–O(2)	1.264 (10)	C(13)–O(1)	1.263 (10)
C(15)–C(16)	1.391 (15)	C(15)–C(13)	1.405 (16)	C(30)–O(4)	1.286 (11)
C(33)–O(3)	1.264 (10)	C(30)–C(32)	1.386 (14)	C(32)–C(33)	1.368 (15)
		Contact Distances (Å) and ρ Values (in Parentheses)			
Ag- -F(5)	2.835 (5) (1.43)[a]	Ag- -F(10)	2.794 (5) (1.41)[a]		
Ag- -F(15)	2.842 (5) (1.43)[a]	Ag- -F(20)	3.027 (5) (1.52)[a]		
		Angles (deg)			
Pt(1)–Ag–Pt(2)	177.2 (1)	C(1)–Pt(1)–C(7)	89.9 (3)	O(1)–Pt(1)–O(2)	91.1 (2)
C(1)–Pt(1)–O(2)	89.3 (3)	C(7)–Pt(1)–O(1)	89.7 (3)	C(18)–Pt(2)–C(24)	88.4 (3)
O(3)–Pt(2)–O(4)	92.0 (2)	C(18)–Pt(2)–O(4)	91.0 (3)	C(24)–Pt(2)–O(3)	88.6 (3)
C(16)–C(15)–C(13)	127.2 (8)	C(30)–C(32)–C(33)	128.5 (8)		

[a] ρ values = Ag- -F experimental distance/sum of covalent radii.[1]

$AgNi(acac)_3\cdot 2AgNO_3\cdot OH_2$,[9] a complex with acac-*O,O′* bonded to nickel centers and C^3 bonded to silver cations. Finally, the Pd- -Ag distance is 3.311 (1) Å, which excludes any metal–metal interaction.

X-ray Structure Analysis of $(NBu_4)[Pt_2Ag(C_6F_5)_4(acac)_2]\cdot CH_2Cl_2$ ($4\cdot CH_2Cl_2$). Figure 2 shows a drawing of the anion $[Pt_2Ag(C_6F_5)_4(acac)_2]^-$. Selected bond distances and bond angles are given in Table VI. In this case the anion is also trinuclear, but in contrast with 3, the anionic fragments $[(C_6F_5)_2Pt(acac\text{-}O,O')]^-$ are bonded to the silver center through the platinum atoms and the silver center is in a nearly linear environment [Pt(1)–Ag–Pt(2) = 177.2 (1)°]. The Pt–Ag distances (2.668 (1) and 2.681 (1) Å) are near the low end of the range found in other complexes containing Pt–Ag bonds.[1] Distances within the $[(C_6F_5)_2Pt(acac\text{-}O,O')]^-$ moiety, which is square planar, are similar to distances found in other pentafluoro-[10] and acetylacetonato-containing[11] platinum complexes. In this case, the silver center is not bonded to the C^3 atoms (Ag- -C(15) = 3.793 (9); Ag- -C(32) = 3.957 (9) Å), and for this reason, the acetylacetonato rings are planar and the angles around C(15) and C(32) are close to 120° as expected for an sp^2-hybridized C atom. The Pt–Ag bond deviates ≃ 10° from perpendicularity to the best least-squares plane defined by the ipso-C atom of the C_6F_5 groups, the platinum atom, and the O-donor atoms of the acetylacetonato ligands.[12] The basic behavior of the platinum center could be responsible for the shorter Pt–O distances (range 2.053 (6)–2.051 (6) Å), relative to Pd–O (2.087 (5) and 2.088 (4) Å), since the formation of Pt–Ag bonds should increase the strength of the Pt–O bonds. Finally, as usual in most of these pentafluorophenyl Pt–Ag complexes[1,2] the pen-

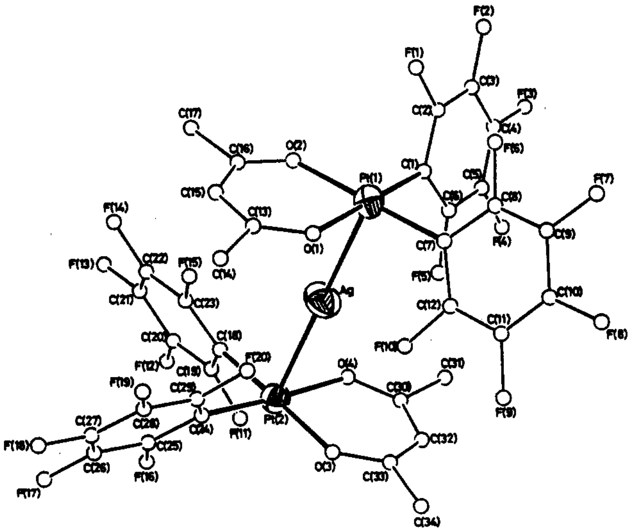

Figure 2. ORTEP drawing of the anion of 4.

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tafluorophenyl rings are oriented in such a way that one of the ortho-F atoms of each C_6F_5 group is located at a short distance from the silver center. The ortho-F- -Ag distances and the ρ values (see Table VI) are at the high end of the range found in other complexes of this type.[1,2] As can be seen from Figures 1 and 2, the most important difference between complexes 3 and 4 rests in the identity of the atom of the $[(C_6F_5)_2M(acac\text{-}O,O')]^-$ moiety, which acts as a donor toward the silver center.

The important structural differences between the acetylacetonato rings of complexes 3 and 4, which are consequences of the bonding mode of the $[(C_6F_5)_2M(acac\text{-}O,O')]^-$ fragment to the silver, explain the differences observed for complexes 1-4 in the IR absorptions of acetylacetonato (Table IV). The acac-C^3-Ag interactions in 3 should produce an increase of the ν(CO) frequency (because of a greater contribution of the keto tautomer of the diketone)[13] and the disappearance of the absorption due to π(C-H), because of the change of hybridization of the C^3 atom (sp^2-sp^3)[14,15] relative to the starting material 1. No significant differences are to be expected between the respective acac absorptions of 2 and 4. On the other hand, the differences in the signals due to C^3 and C(C=O) in the ^{13}C NMR spectra between 1 and 3 (Table IV) can be explained as well in terms of the acac-C^3-Ag interaction.

Conductivity values in acetone solutions (206 (3), 165 (4) Ω^{-1} cm^2 mol^{-1}) are higher than the expected for 1:1 electrolytes, suggesting that in the donor solvent the C-Ag (3) or Pt-Ag (4) bonds are cleaved by the solvent, thus increasing the ionic concentration. This fact indicates that the interactions between the $[(C_6F_5)_2M(acac\text{-}O,O')]^-$ and Ag^+ fragments are weak in nature in both cases.

The ^{19}F NMR spectrum deserves some comment. As expected, all C_6F_5 groups are equivalent, but it is noteworthy that both ortho-F atoms (endo and exo), and both meta-F atoms as well, of each C_6F_5 group are equivalent (even at -60 °C). If, as it has been pointed out, the two C_6F_5 groups in cis positions are not free to rotate around the M-C bonds (M = Pd, Pt[16]), it seems sensible to assume that the equivalence of these F signals could be due to the presence of dynamic processes in solution (probably partial dissociative processes of the C-Ag or Pt-Ag bonds), which would render equivalent both halves of the C_6F_5 rings.

Acknowledgment. We thank the DGICYT (Spain) for financial support (Project PB89-0057), and E.P.U. thanks the DGA for a grant.

Supplementary Material Available: For complexes 3 and $4{\cdot}CH_2Cl_2$, tables of atomic coordinates, equivalent isotropic displacement coefficients, anisotropic thermal parameters, and bond distances and angles (15 pages). Ordering information is given on any current masthead page.

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[13] This fact can be also responsible for the weaker Pd-O interactions compared to the corresponding Pt-O interactions.

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