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TRIS(PENTAFLUOROPHENYL)PLATINATE(II) COMPLEXES

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Summary

$(NBu_4)_2[Pt(C_6F_5)_4]$ reacts with HCl to give (1/1) $(NBu_4)_2[PtCl(C_6F_5)_3]$ or (1/2) $(NBu_4)_2[Pt_2(\mu\text{-}Cl)_2(C_6F_5)_4]$. Metathetical reactions of the mononuclear PPN salt ($PPN/Ph_3P{=}N{=}PPh_3$) with KX (X = Br, I, CN or SCN) give complexes of the formula $(PPN)_2[PtX(C_6F_5)_3]$, whereas reactions with neutral ligands in the presence of a perchlorate $MClO_4$ lead to derivatives of the types $Q[Pt(C_6F_5)_3L]$ ($L = C_2Ph_2$, dpm or dpe) and $Q_2[(C_6F_5)_3Pt(L\text{–}L)Pt(C_6F_5)_3]$ (L = dpm or dpe). The reactions of $Q[Pt(C_6F_5)_3(dpm)]$ with $(O_3ClO)Pd(C_6F_5)(PPh_3)_2$, $PdCl_2(tht)_2$, $PtCl_2(tht)_2$ or $AgClO_4$ give bi- or tri-nuclear derivatives.

Introduction

We recently reported [1] the synthesis of anionic pentafluorophenyl derivatives of Pd^{II} and Pt^{II} of the types $Q_2[M(C_6F_5)_4]$, $Q[M(C_6F_5)_3(tht)]$ and $Q_2[M_2(\mu\text{-}X)_2(C_6F_5)_4]$ (M = Pd, Pt; tht = tetrahydrothiophene; X = halide or pseudohalide; $Q = N(C_4H_9)_4$) [1], as well as the reactions of the two last types of complexes with neutral ligands L to give anionic mononuclear complexes of the types $Q[M(C_6F_5)_3L]$ and $Q[cis\text{-}MX(C_6F_5)_2L]$ (L = N, P, As donor ligands) [1,2]. However, $Q[M(C_6F_5)_3(tht)]$ does not usually react with anionic ligands to give $Q_2[MX(C_6F_5)_3]$, the tht ligand in the starting complex being displaced only by CN^-, and not by other halides or pseudohalides [3].

In this paper, we describe the results of our study on the reactivity of $Q_2[Pt(C_6F_5)_4]$ with controlled amounts of HCl. These reactions provide a better synthetic route to the dinuclear complexes $Q_2[Pt_2(\mu\text{-}X)_2(C_6F_5)_4]$ and also for the preparation of $Q_2[PtCl(C_6F_5)_3]$, which is a good precursor for the synthesis of diverse tris(pentafluorophenyl)platinate(II) complexes.

Results and discussion

Reactions of $Q_2[M(C_6F_5)_4]$ with HCl

The reaction (1/1) of $(NBu_4)_2[Pt(C_6F_5)_4]$ with aqueous HCl in methanol causes

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cleavage of only one of the Pt–C bonds and leads to formation of $(NBu_4)_2[PtCl(C_6F_5)_3]$ (I, 76% yield) (eq. 1)

$$(NBu_4)_2[Pt(C_6F_5)_4] + HCl \rightarrow HC_6F_5 + (NBu_4)_2[PtCl(C_6F_5)_3] \quad (1)$$

This is the only process so far observed which leads to an anionic halotris(aryl)platinate(II) anion, since the previously described $(NBu_4)[Pt(C_6F_5)_3tht]$ does not react with halide salts in refluxing acetone [1].

Use of two molar equivalents of HCl causes cleavage of two Pt–C bonds, and the binuclear complex $(NBu_4)_2[Pt_2(\mu\text{-}Cl)_2(C_6F_5)_4]$ can be isolated (> 85% yield) (eq. 2).

$$2(NBu_4)_2[Pt(C_6F_5)_4] + 4HCl \rightarrow 4HC_6F_5 + (NBu_4)_2[Pt_2(\mu\text{-}Cl)_2(C_6F_5)_4] \quad (2)$$

This is the best method for preparing this complex, whose synthesis by another method * we described previously [1].

Reactivity of $Q_2[PtCl(C_6F_5)_3]$

Substitution of X by other anionic ligands

Reactions of $(NBu_4)_2[PtCl(C_6F_5)_3]$ with KX (X = Br, I, CN, SCN) in refluxing acetone leads to solutions which upon evaporation yield non-crystallizable oils. However, the metathetical reaction of the platinum complex with $[(PPh_3){=}N{=}(PPh_3)]Cl$ [(PPN)Cl] leads to $(PPN)_2[PtCl(C_6F_5)_3]$ (II), which reacts with KX in refluxing acetone. From the resulting solutions the complexes $(PPN)_2[PtX(C_6F_5)_3]$ with X = Br (III), I (IV), CN (V); and SCN (VI), can be easily isolated (ca. 65% yields).

The complex $(PPN)[Pt(C_6F_5)_3(tht)]$ [1] does not react with (PPN)X (X = Cl, Br, I), the starting products being recovered. Only X = CN is able to displace (tht), to form $(PPN)_2[Pt(CN)(C_6F_5)_3]$ [3].

Synthesis of $Q[Pt(C_6F_5)_3L]$ or $Q_2[(C_6F_5)_3Pt(L\text{–}L)Pt(C_6F_5)_3]$

We previously described a method for the synthesis of anionic $[Pt(C_6F_5)_3L]^-$ derivatives containing a neutral ligand involving displacement of tht in $[Pt(C_6F_5)_3(tht)]^-$ by ligands L (L = PPh_3, $AsPh_3$, $SbPh_3$, 1/2 $Ph_2P(CH_2)_4PPh_2$) [1].

Use of complex I provides an alternative route to introduce a neutral ligand, after the chloride has been displaced by using a appropriate abstractor. Equations 3 and 4 summarize the two synthetic methods.

$$Q[Pt(C_6F_5)_3(tht)] + L \rightarrow (tht) + Q[Pt(C_6F_5)_3L] \quad (3)$$

$$Q_2[PtCl(C_6F_5)_3] + L + MClO_4 \rightarrow MCl + QClO_4 + Q[Pt(C_6F_5)_3L] \quad (4)$$

The following results are obtained:

(i) With L = PhC≡CPh, no reaction of type (3) is observed but the reaction of type (4) readily gives $(NBu_4)[Pt(C_6F_5)_3(C_2Ph_2)]$ (VII).

(ii) With L = bis(diphenylphosphino)methane (dpm) reaction (3) leads to

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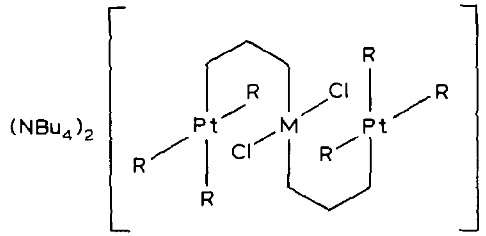
* With palladium, the reaction analogous to that in eq. 1 leads to a mixture of unreacted $Q_2[Pd(C_6F_5)_4]$ and $Q_2[Pd_2(\mu\text{-}Cl)_2(C_6F_5)_4]$, whereas reaction analogous to that in eq. 2 gives the binuclear complex in over 85% yield.

$(NBu_4)[Pt(C_6F_5)_3(dpm)]$ (VIII) irrespective of the molar ratio (1/1) or (2/1) of the reagents. In VIII the diphosphine acts as a unidentate ligand, as revealed by the ^{31}P NMR spectra (see Structural studies). It follows that $(NBu_4)[Pt(C_6F_5)_3(tht)]$ does not react with $(NBu_4)[Pt(C_6F_5)_3(dpm)]$ i.e., the uncoordinated P atom in the diphosphine complex is unable to displace the (tht) ligand. With L = dpe (1/1 ratio, refluxing ethanol, 2 h) two products are obtained, $(NBu_4)[Pt(C_6F_5)_3(dpe)]$ (IX) and $(NBu_4)_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpe})]$ (X), i.e., despite of the presence of the necessary amount of diphosphine to give the mononuclear complex the binuclear species is also formed. The other route (eq. 4) leads in the case of L = dpm (2/1) to the binuclear complex $(NBu_4)_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpm})]$ (XI) (which is not accessible through reaction 3). This method also serves for the preparation of complex XI with $L = (NBu_4)[Pt(C_6F_5)_3(dpm)]$. The results confirm the superiority of the second route (eq. 4) for synthetic purposes.

Synthesis of polynuclear complexes

Complex $(NBu_4)[Pt(C_6F_5)_3(dpm)]$ (VIII) with an uncoordinated P atom, can act as a ligand to give polynuclear complexes. Thus, VIII reacts with $[Pd(OClO_3)(C_6F_5)(PPh_3)_2]$ in 1/1 molar ratio in toluene to give $[Pt(C_6F_5)_3(\mu\text{-dpm})Pd(C_6F_5)(PPh_3)_2]$, (XII) and also reacts in 2/1 ratio with $MCl_2(tht)_2$ (M = Pd, Pt) with formation of anionic trinuclear species (XIII, XIV) (eq. 5).

$$2\,(NBu_4)\left[Pt(C_6F_5)_3(dpm)\right] + MCl_2(tht)_2 \longrightarrow 2\,(tht) +$$

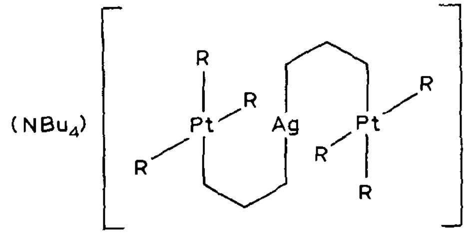


(XIII, XIV)

(5)

Moreover, a methanol solution of VIII reacts with $AgClO_4$ in 2/1 molar ratio to give (< 1 h) the trinuclear complex XV (eq. 6)

$$2\,(NBu_4)\left[Pt(C_6F_5)_3(dpm)\right] + AgClO_4 \longrightarrow (NBu_4)ClO_4 +$$



(XV)

(6)

TABLE 1

ANALYTICAL DATA, CONDUCTIVITIES AND MELTING POINTS[a]

	Analyses (Found (calcd.) (%))			Λ_M (ohm^{-1} cm^2 mol^{-1})	M.p. (°C)
	C	H	N		
$Q'_2[PtCl(C_6F_5)_3]$	49.69	6.00	2.34	179	121
(I)	(49.41)	(5.97)	(2.30)		
$Q_2[PtCl(C_6F_5)_3]$	59.26	3.51	1.75	209	60
(II)	(59.75)	(3.34)	(1.54)		
$Q_2[PtBr(C_6F_5)_3]$	58.20	3.23	1.54	206	190(d)
(III)	(58.32)	(3.26)	(1.51)		
$Q_2[PtI(C_6F_5)_3]$	55.90	3.26	1.56	206	194(d)
(IV)	(56.87)	(3.18)	(1.47)		
$Q_2[Pt(CN)(C_6F_5)_3]$	60.08	3.92	2.49	197	185
(V)	(60.69)	(3.34)	(2.33)		
$Q_2[Pt(SCN)(C_6F_5)_3]$	59.78	3.32	2.50	200	194
(VI)	(59.61)	(3.27)	(2.29)		
$Q'[Pt(C_6F_5)_3(C_2Ph_2)]$	50.91	4.39	1.50	119	139(d)
(VII)	(51.63)	(4.15)	(1.25)		
$Q'[Pt(C_6F_5)_3dpm]$	54.10	4.46	1.24	91	134
(VIII)	(53.55)	(4.41)	(1.05)		
$Q'[Pt(C_6F_5)_3dpe]$	53.70	4.68	1.15	105	136
(IX)	(53.90)	(4.52)	(1.04)		
$Q'_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpe})]$	49.07	4.28	1.07	160	227
(X)	(49.38)	(4.16)	(1.24)		
$Q'_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpm})]$	49.17	4.26	1.28	195	145
(XI)	(49.60)	(4.18)	(1.23)		
$Pt(C_6F_5)_3(\mu\text{-dpm})Pd(C_6F_5)(PPh_3)_2$	54.97	2.58	–	25	190(d)
(XII)	(54.33)	(2.77)			
$Q'_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpm})_2PdCl_2]$	50.11	4.14	1.18	181	170(d)
(XIII)	(50.19)	(4.19)	(0.99)		
$Q'_2[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpm})_2PtCl_2]$	48.88	3.95	1.16	200	214
(XIV)	(48.66)	(4.01)	(0.96)		
$Q'[\{Pt(C_6F_5)_3\}_2(\mu\text{-dpm})_2Ag]$	49.54	3.46	0.75	100	138(d)
(XV)	(48.77)	(3.21)	(0.55)		
$Pt(C_6F_5)_2(dpm)$	48.37	2.58		[b]	290(d)
(XVI)	(48.64)	(2.42)			

[a] Q = PPN, Q′ = NBu_4. [b] Insoluble in acetone.

Methanolic solutions of XV deposit $Pt(C_6F_5)_2(dpm)$ (XVI), a monomeric complex containing chelated (dpm) as follows from its IR and M.W. data.

Table 1 gives analytical and other data for the complexes described (I–XVI). Their conductivities are those expected for 1 : 1, 1 : 2 or non-electrolytes [10].

IR spectra

All the complexes show absorptions at 1490s, 1050s, 950s and 800–765m cm^{-1}, characteristic of the C_6F_5 groups [5,6]. Complexes I and VII show three absorptions at ca. 800s, 780m and 765m cm^{-1}, assignable to the X-sensitive mode of the C_6F_5 group [1,6], in good agreement with the normal vibration modes ν(M–C) expected for a molecule of C_{2v} symmetry (2 A_1 + B_1). All the other complexes show also absorptions in this region but these cannot be unambiguously assigned since both the cation PPN$^+$ (used for reasons of solubility) and the diphosphines absorb in this zone. Complex XVI shows two absorptions at 775 and 760 cm^{-1}, assignable to the X-sensitive mode of the C_6F_5 group, in accordance with those expected for a *cis*-isomer [2].

Complex I exhibits an absorption at 280m cm^{-1}, assignable to the ν(Pt–Cl) stretching vibration; the corresponding ν(Pt–Br) (complex III) and ν(Pt–I) bands (complex IV) were not observed because they were outside the range of our instrument. Complex V shows the ν(CN) stretching vibration at 2120 cm^{-1}, whilst for complex VI the ν(CN) of the SCN group appears at 2105 cm^{-1}. The internal standard ratio ν(CN) and ν(CO) intensities of the salicylic acid [7] gives a value of 2.2, suggesting that the SCN^- group is N-bonded to the Pt atom.

Complex VII exhibits absorptions at 2005m, 1595m, 1500s, 755s, 688s, 567m and 505m cm^{-1} arising from the diphenylacetylene. The absorption recorded at 2005 cm^{-1}, assignable to the stretching vibration ν(C≡C) is inactive in the free ligand and becomes active on coordination.

In the case of XIII and XIV the stretching vibrations due to ν(Pd–Cl) and ν(Pt–Cl) cannot be assigned because they are masked by internal vibrations of the dpm.

The complexes containing the cation $NBu_4{}^+$ show an absorption at 880m,br cm^{-1} assignable to this group. The cation PPN^+ exhibits a large number of absorptions at 1117s, 1000s, 800m, 760s, 750s, 695m,s 560m,s 540m,s and 400m,s cm^{-1}, which hinder the assignment of other absorptions due to the different ligands.

^{31}P NMR spectra

The ^{31}P NMR spectra of complexes VIII, IX, X and XI (in hexadeuteroacetone, external reference H_3PO_4 85%) are in good agreement with the proposed formulas.

For VIII the signal corresponding to the phosphorus atom linked to the platinum appears at δ + 6.60 ppm (^1J(Pt–P) 2640 Hz); both the central peak and the platinum satelites appear as unresolved multiplets due to the coupling with the fluorine atoms of the C_6F_5 groups as well as with the uncoordinated P atom. The signal due to this P atom is a doublet at δ −28.83 ppm (^1J(P–P) 47, ^3J(Pt–P) 61 Hz). IX shows also two types of signals, with the same characteristics; a multiplet at δ 8.58 ppm (^1J(Pt–P) 2565 Hz) and a doublet at δ −12.89 ppm (^3J(P–P) 35 Hz, ^4J(Pt–P) unmeasured). In X both P atoms are equivalent and give a multiplet, with Pt satellites, at δ +7.97 ppm (^1J(Pt–P) 2585 Hz). Similarly XI shows a signal at δ +10.17 ppm (^1J(Pt–P) 2614 Hz).

Experimental

C, H and N analyses were determined with a Perkin–Elmer 240 microanalyzer. IR spectra were recorded on a Perkin–Elmer 599 spectrophotometer (at 4000–200 cm^{-1}) using Nujol mulls between polyethylene sheets. Conductivities were measured in approx. 5×10^{-4} M solution with a Philips PW 9501/01 conductimeter. NMR studies were made with a JEOL PFT 100 and Varian FT80. The molecular weight of XVI was determined in $CHCl_3$ on a Perkin–Elmer 115 apparatus. $[N\{P(C_6H_5)_3\}_2]Cl$ was prepared as described by Ruff and Schlentz [9]. $(NBu_4)_2[Pt(C_6F_5)_4]$ was obtained as described elsewhere [1].

Reactions of $(NBu_4)_2[M(C_6F_5)_4]$ *(M = Pd, Pt) with HCl*

(1) 1/1 molar ratio

$(NBu_4)_2[PtCl(C_6F_5)_3]$ (I). To a stirred solution of $(NBu_4)_2[Pt(C_6F_5)_4]$ (1.454 g, 1.079 mmol) in 100 ml of ethanol was slowly added 2.32 ml (1.079 mmol) of a HCl solution (prepared by adding methanol to 2 ml of aqueous 11.6 *M* HCl to give a final volume of 50 ml). After 1 h of stirring at room temperature the solution was filtered and concentrated to 25 ml. 30 ml of 2-propanol was added and the solution was again concentrated to 25 ml. Repetition of the addition of 2-propanol and concentration to 25 ml led to the crystallization of I (76% yield).

The reaction of $(NBu_4)_2[Pd(C_6F_5)_4]$ with HCl (1/1 ratio) and work-up as before led to a first fraction made up of a mixture of the starting compound and $(NBu_4)_2[Pd_2(\mu\text{-}Cl)_2(C_6F_5)_4]$. Addition of hexane to the 2-propanol solution gave a small amount of white crystalline solid, seemingly a mixture of the same products, whilst cooling of the solution to −30°C gave a precipitate $(NBu_4)_2[Pd_2(\mu\text{-}Cl)_2(C_6F_5)_4]$ (8% yield).

(2) 1/2 molar ratio

To a solution of $(NBu_4)_2[Pt(C_6F_5)_4]$ (0.3 g, 0.223 mmol) in 50 ml of MeOH was added HCl (0.445 mmol) in water/methanol. After 7 h stirring the solution was evaporated to ~ 5 ml. Addition of 25 ml of 2-propanol and evaporation to ~ 15 ml led to the crystallization of $(NBu_4)_2[Pt_2(\mu\text{-}Cl)_2(C_6F_5)_4]$ (86% yield) [4].

A similar procedure using $(NBu_4)_2[Pd(C_6F_5)_4]$ gave $(NBu_4)_2[Pd_2(\mu\text{-}Cl)_2(C_6F_5)_4]$ (87% yield) [4].

$(PPN)_2[PtCl(C_6F_5)_3]$ (II). To a solution of PPNCl (0.79 g, 1.376 mmol) in 10 ml of methanol was added $(NBu_4)_2[PtCl(C_6F_5)_3]$ (0.836 g, 0.688 mmol) in 25 ml of methanol. Immediate precipitation of complex II was observed. The stirring was continued for 15 min to complete the crystallization (81% yield).

$(PPN)_2[PtX(C_6F_5)_3]$ X = Br (III), I (IV), CN (V) or SCN (VI). To complex II (0.3 g, 0.165 mmol) in 30 ml of acetone was added ca. 0.25 mmol of the appropriate potassium salt (KBr, KI, KSCN or KCN). The mixture was refluxed for 8 h then the solution was filtered and evaporated to dryness. The residue was extracted with ~ 20 ml of methanol to leave crystals of the product (III: 69% yield, IV: 64% yield; V: 60% yield; VI: 71% yield).

Synthesis of $(NBu_4)[Pt(C_6F_5)_3L]$ *L = diphenylacetylene (VII); dpm (VIII); dpe (IX)*

L = diphenylacetylene (VII). With exclusion of light, $AgClO_4$ (0.0336 g, 0.162 mmol was added to a solution of $(NBu_4)_2[PtCl(C_6F_5)_3]$ (I) (0.1975 g, 0.162 mmol) in 30 ml of CH_2Cl_2 and the mixture was stirred for 2.5 h at room temperature. The AgCl was filtered off and diphenylacetylene (0.0289 g) was added. After 3 h stirring the solution was filtered and concentrated almost to dryness, and 15 ml of ^nBuOH and 15 ml of hexane were added. Partial evaporation led to the crystallization of VII (55% yield).

L = dpm (VIII), dpe (IX). To a solution of $(NBu_4)_2[PtCl(C_6F_5)_3]$ (I) (0.626 g, 0.51 mmol) in 50 ml of dichloromethane was added 0.51 mmol of dpm or dpe along with $NaClO_4$ (0.063 g, 0.51 mmol). The suspension was stirred for 15 h, then the solution was filtered and treated with 40 ml of H_2O. The liquids were decanted off, and the dichloromethane phase was dried with $MgSO_4$ then evaporated to dryness.

The residue was stirred with 15 ml of isopropanol, for 15 min at room temperature to give crystals of VIII (71%) or IX (70% yield).

Reaction of (NBu$_4$)[Pt(C$_6$F$_5$)$_3$tht] with dpm and dpe

(a) L = dpm. To (NBu$_4$)[Pt(C$_6$F$_5$)$_3$tht] (0.4 g, 0.389 mmol) in 60 ml of ethanol was added 0.149 g (0.389 mmol) of dpm. The suspension was refluxed for 3 h, evaporated to dryness, treated with MeOH and again evaporated to dryness. The treatment with MeOH and evaporation was repeated three times, then the residue was extracted with 10 ml of MeOH to leave a first fraction of undissolved VIII. A further amount of VIII was precipitated from the solution by addition of 2-propanol (total yield 68%).

Use of a smaller proportion of dpm gives only VIII along with unreacted starting material.

(b) L = dpe. (NBu$_4$)[Pt(C$_6$F$_5$)$_3$tht] (0.35 g, 0.341 mmol) in 90 ml of ethanol was refluxed with 0.136 g (0.341 mmol) of dpe for 3 h. The resulting suspension was concentrated to ca. 40 ml and kept for 3 h at −30°C. The formed solid was filtered off and recrystallized from acetone/CHCl$_3$ (X: 29% yield). The ethanolic filtrate was evaporated to dryness, dissolved in isopropanol and stored at −30°C for 48 h. The complex IX which separated was recrystallized from chloroform/ethanol (57% yield).

(NBu$_4$)$_2$[{Pt(C$_6$F$_5$)$_3$}$_2$(μ-dpm)] (XI). To complex I (0.2 g, 0.16 mmol) in 30 ml of dichloromethane was added dpm (0.031 g, 0.08 mmol) and NaClO$_4$ (0.02 g, 0.16 mmol). The mixture was stirred at room temperature for 8 h, then filtered. The filtrate was treated with 50 ml of H$_2$O and the liquid layers decanted. The dichloromethane phase was dried with MgSO$_4$ and finally evaporated almost to dryness. Addition of isopropanol then caused crystallization of XI (70% yield).

Reaction of (NBu$_4$)[Pt(C$_6$F$_5$)$_3$dpm]

(a) With O$_3$ClOPd(C$_6$F$_5$)(PPh$_3$)$_2$. 0.15 g of VIII in 50 ml of toluene was stirred for 3 h with O$_3$ClOPd(C$_6$F$_5$)(PPh$_3$)$_2$ (0.102 g, 0.113 mmol). The solution was evaporated almost to dryness and 20 ml of 2-PrOH were added to precipitate XII (62% yield).

(b) With trans-PdCl$_2$(tht)$_2$. 0.1 g (0.075 mmol) of VIII in 30 ml of acetone was stirred for 23 h (room temperature) with 0.0133 g (0.037 mmol) of *trans*-PdCl$_2$(tht)$_2$ [1]. Partial evaporation of the solvent and stirring for 1 h with 5 ml of 2-PrOH led to crystallization of XIII (62% yield).

(c) With trans-PtCl$_2$(tht)$_2$. Reaction as above between 0.14 g (0.10 mmol) of VIII and 0.0234 g (0.05 mmol) of *trans*-PtCl$_2$(tht)$_2$ [1] gave XIV (69% yield).

(d) With AgClO$_4$. To a solution of 0.23 g (0.17 mmol) of VIII in 30 ml of methanol was added 0.018 g (0.086 mmol) of AgClO$_4$. The mixture was stirred at room temperature for 1 h then evaporated almost to dryness. Addition of 5 ml of 2-PrOH led to the crystallization of XV (73% yield).

A similar procedure but with stirring for 7 h (room temperature) led to the gradual precipitation of Pt(C$_6$F$_5$)$_2$(dpm) (XVI), which was filtered off (70% yield) (MW 909, calculated: 913.6).

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