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Selectivity in reactions of a dimethylplatinacyclopentane complex

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Abstract

The new dimethylplatinacyclopentane complex [$PtMe_2(CH_2CH_2CH_2CH_2)(dppm)$], **2**, has been prepared and shown to exist as a mixture of two isomers **2a** and **2b**. Complex **2** reacts with the electrophiles SO_2 and HCl by selective cleavage of a Me–Pt bond, to give [$PtMe(SO_2Me)(CH_2CH_2CH_2CH_2)(dppm)$], **3**, and [$PtClMe(CH_2CH_2CH_2CH_2)(dppm)$] **4**, each of which also exists in isomeric forms. Possible reasons for the higher reactivity of Pt–CH_3 than for Pt–CH_2 bonds are discussed.

Keywords: Platinum; Metallacycle

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1. Introduction

Metallacycles are of interest as intermediates in catalysis and there has been much interest in the synthesis and reactions of such complexes [1–3]. Metallacyclopentanes often have good thermal stability, since decomposition by the β-elimination is difficult, and so they are relatively easily synthesized [4–11]. It was of interest to compare the reactivity of the metal–carbon bonds in metallacycles with those in simple alkyl–metal complexes, and the most direct way to do this is to make complexes containing both types of functionality. Few such compounds are known [7,9,10]. In a previous article, the synthesis of the platinum(IV) complex [$PtIMe(CH_2CH_2CH_2CH_2)(dppm)$], **1**, dppm = $Ph_2PCH_2PPh_2$, was reported [9], but the metal–carbon bonds in this complex are inert towards electrophilic reagents. Since tetraalkylplatinum(IV) complexes are much more reactive towards electrophiles [12–14], we decided to make the related complex [$PtMe_2(CH_2CH_2CH_2CH_2)(dppm)$], **2**, which contains two Me–Pt bonds along with two Pt–CH_2 bonds in the platinacyclopentane ring. We report below the synthesis of the complex and studies of its selectivity in reactions with the electrophiles HCl and SO_2.

2. Results and discussion

2.1. Model reactions with [$PtMe_4(dppm)$]

The known complex [$PtMe_4(dppm)$], reacts with SO_2 and HCl in the way shown in Eqs. (1) and (2). These reactions are similar to those of [$PtMe_4$(2,2′-bipyridine)] and are assumed to occur by direct attack of the electrophile at one of the mutually *trans* methyl groups, aided by the strong *trans*-influence of the alkyl group *trans* to the leaving group [12]. The reactions involve the shortlived ionic intermediates [$PtMe_3(dppm)]^+(MeSO_2)^-$ and [$PtMe_3(dppm)]^+Cl^-$, respectively [15, 16].

The products were readily characterized from their spectroscopic data, which are listed in the experimental section. For example, the complex [$PtMe_3(SO_2Me)(dppm)$] shared bands in the IR spectrum at 1155 and 1036 cm^{-1}, characteristic of S-bonded sulfinate ligands [15]; and the observation of the coupling constant $^2J(PtMe) = 55$ Hz for the MeS resonance in the ^{13}C NMR spectrum strongly supports this assignment. The S-bonded sulfinate structure is of course, expected for a soft acceptor such as the trialkylplatinum(IV) centre [12,15,16]. These reactions were studied as models for the more complex reactions of the platinacyclopentane derivative described below.

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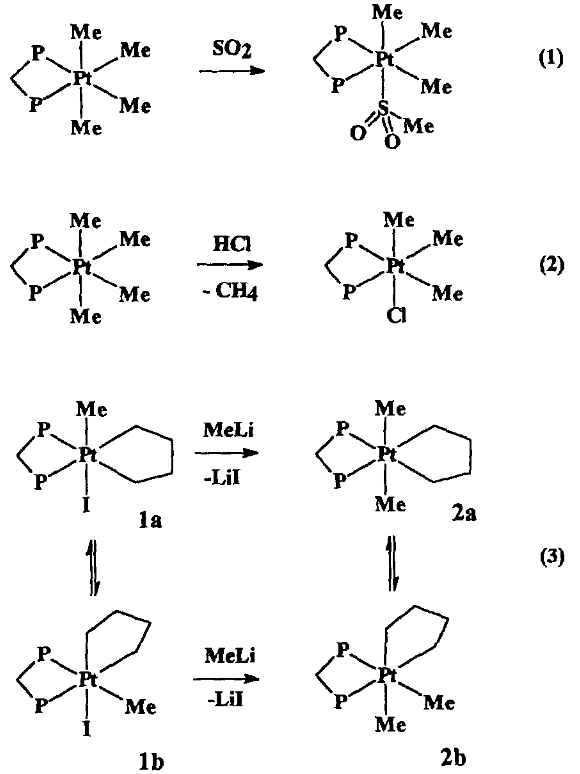


Equation 1.

2.2. Synthesis and characterization of [$PtMe_2(CH_2CH_2CH_2CH_2)(dppm)$]

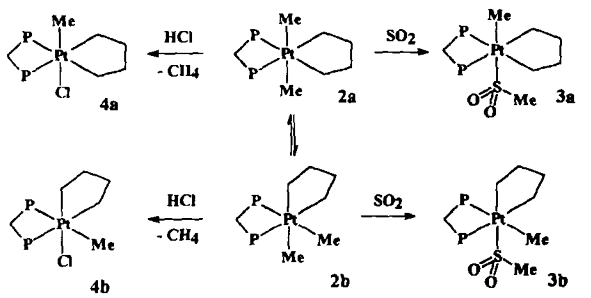
The complex [$PtIMe(CH_2CH_2CH_2CH_2)(dppm)$], **1**, exists as an equilibrium mixture of isomers **1a** and **1b** with the less symmetrical isomer **1b** slightly more stable than **1a** such that the equilibrium constant given by [**1b**] [**1a**], is 3 [9]. Reaction of **1** with methyllithium gave the tetraalkylplatinum(IV) complex [$PtMe_2(CH_2CH_2CH_2CH_2)(dppm)$], **2**, according to Eq. (3).

Complex **2** was shown by NMR spectroscopy to exist as an equilibrium mixture of the isomers **2a** and **2b** with K = **2b**/**2a** = 1.1. Complete NMR data are given in the experimental section and selected data in Table 1. In the ^{31}P NMR spectrum, complex **2a** showed a singlet with satellites due to coupling to ^{195}Pt with $^{1}J(PtP) = 845$ Hz, while the less symmetrical **2b** gave two doublet resonances, each with $^{2}J(PP) = 25$ Hz and each with satellites due to coupling to ^{195}Pt with values of $^{1}J(PtP) = 961$ and 837 Hz. The resonances with the smaller and larger value of $^{1}J(PtP)$ are assigned to the phosphorus atoms *trans* to CH_2 and Me respectively, in accordance with the observation that the CH_2 group of the metallacycle exerts a stronger NMR *trans* influence than the methyl group [9]. As expected in the light of the above data, the ^{195}Pt NMR spectrum con-

Table 1
Selected NMR data (δ in ppm, *J* in Hz) for the platinacyclopentane complexes.

Complex	δ(P) *J*(PtP)	*Trans* atom	δ(Me) *J*(PtC)	*Trans* atom	δ(CH_2) *J*(PtC)	*Trans* atom	δ(Me) *J*(PtH)	*Trans* atom
1a	−55.4	CH_2	9.13	I	31.30	P	0.77	I
	824		682		549		68	
2a	−52.7	CH_2	−4.98	Me	35.3	P	−0.12	Me
	845		429		540		46	
3a	−39.8	CH_2	−2.07	S	36.7	P	0.15	S
	848		553		550		59	
4a	−40.9	CH_2	0.53	Cl	– [a]	– [a]	0.52	Cl
	809		716				75	
1b	−65.9	CH_2	2.58	P	40.16	I	1.66	P
	812		584		668		65	
	−60.8	Me			32.65	P		
	899				541			
2b	−60.6	CH_2	−12.0	CH_2	22.1	Me	−0.05	CH_2
	837		400		437		43	
	−58.3	Me	2.3	P	30.6	P	0.88	P
	961		610		580		65	
3b	−49.8	CH_2	4.9	P	35.2	S	0.86	P
	791		590		530		65	
	−44.0	Me			34.5	P		
	985				– [b]			
4b	−49.4	CH_2	7.2	P	– [a]	– [a]	1.9	P
	784		584				64	
	−45.8	Me						
	895							

[a] The ^{13}C resonances of the CH_2 groups were not assigned.
[b] The coupling $^{1}J(PtC)$ was not resolved.



Scheme 1.

tained a triplet resonance for **2a** and a doublet of doublets for **2b**, as a result of $^1J(PtP)$ coupling. For isomer **2a**, only one methyl resonance and two CH_2 resonances are expected, and are observed, in either the ^1H or ^{13}C NMR spectra but, for isomer **2b**, two methyl resonances were expected and observed, while four ^{13}C and eight ^1H resonances are expected for the $Pt(CH_2)_4$ ring, three of the expected four ^{13}C resonances were resolved, but the ^1H resonances gave complex overlapping peaks. The high NMR *trans*-influence of the CH_2 groups of the metallacycle is confirmed by the ^1H and ^{13}C NMR parameters. Thus the methyl groups *trans* to methyl in **2a** have couplings $^1J(PtC) = 429$ Hz and $^2J(PtH) = 46$ Hz, while the methyl group *trans* to CH_2 in **2b** has lower couplings, $^1J(PtC) = 400$ Hz and $^2J(PtH) = 43$ Hz.

2.3. Reactions of $[PtMe_2(CH_2CH_2CH_2CH_2)(dppm)]$

The complex **2** reacted rapidly with SO_2 according to Scheme 1 to give a mixture of isomers of $[PtMe(SO_2Me)(CH_2CH_2CH_2CH_2)(dppm)]$, **3a** and **3b**. These complexes decomposed in solution over a period of several days. The isomers could not be separated and were characterized as a mixture.

The more symmetrical isomer **3a** gave a singlet in the ^{31}P NMR spectrum at $\delta = -39.8$ with $^1J(PtP) = 848$ Hz (Table 1); the chemical shift is less negative than in **2a** but the coupling constant is very similar, as expected for phosphorus *trans* to CH_2 of the metallacycle. The MePt and $MeSO_2$ resonances in both the ^1H and ^{13}C NMR spectra had chemical shifts and coupling constants very similar to the analogous resonances for the model compound $[PtMe_3(SO_2Me)(dppm)]$. The CH_2 groups of the metallacycle gave only two ^{13}C resonances, one of them having satellites due to $^1J(PtC)$ coupling, as expected for structure **3a**.

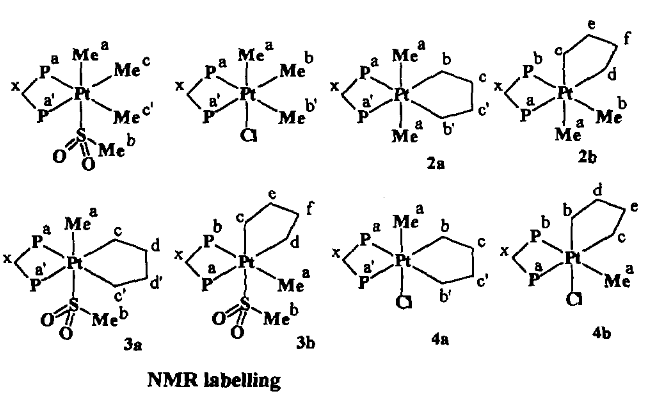
The less symmetrical isomer **3b** gave two ^{31}P resonances, with $^1J(PtP)$ values of 791 Hz and 985 Hz, assigned to the phosphorus atoms *trans* to a CH_2 group and methyl group respectively (Table 1). The presence of the $MeSO_2$ group was clearly indicated by both the ^1H [$\delta(Me) = 1.94$] and the ^{13}C [$\delta(Me) = 47.3$, $^2J(PtC) = 46$ Hz] NMR spectra. A methyl group *trans* to phosphorus was indicated most clearly in the ^{13}C NMR spectrum by the values of $^1J(PtC) = 590$ Hz and the *trans* coupling $^2J(PC) = 107$ Hz, and was confirmed by the ^1H NMR data. Both α-CH_2 resonances of the $Pt(CH_2)_4$ group were resolved in the ^{13}C NMR spectrum (Table 1).

The above data indicated that SO_2 inserts selectively into a Me–Pt bond of **2** rather than into a Pt–CH_2 bond. It is likely that the electrophile SO_2 attacks one of the two mutually *trans* carbon atoms in **2** [12]. For isomer **2a**, this can only give the product of insertion into the Me–Pt bond but, for **2b**, insertion into either of the mutually *trans* Pt–Me or Pt–CH_2 bonds is possible. For a freshly prepared sample of **3**, the isomer ratio **3b**/**3a** was approximately 1, as expected if **3a** and **3b** are formed from **2a** and **2b**, respectively, as shown in the scheme. This suggests but does not prove that the reactions occur with retention of stereochemistry (Scheme 1), as would be expected if the proposed ionic intermediates collapse to the final products more rapidly than scrambling of geometry within the five-coordinate intermediate can occur. Attempts to study changes in the isomer ratio as a function of time were frustrated since general decomposition occurred during several days. The resonances of **3b** decayed faster than those of **3a**, but this might be due to either faster decomposition or partial isomerization of **3b** to **3a**.

The reaction of HCl with **2** gave $[PtClMe(CH_2CH_2CH_2CH_2)(dppm)]$, **4** (Scheme 1). The isomers **4a** and **4b** were readily identified by their spectroscopic properties (Table 1, experimental section). The ratio of products **4b**/**4a** was 1, again suggesting that the reactions occur selectively, as shown in Scheme 1. Some decomposition to give $[PtCl_2(dppm)]$ was observed to occur in this reaction, especially where an excess of HCl was used.

3. Conclusions

The results are fully consistent with the mechanism outlined below, and the selectivity shown in Scheme 1. The electrophile attacks one of the mutually *trans* Pt–C bonds in the tetraalkylplatinum(IV) complexes. In $[PtMe_4(dppm)]$ or **2a**, this leads to cleavage of a methyl platinum bond. Since the platinum(IV) complexes have the 18-electron configuration, the methyl group must leave to give a five-coordinate *fac*-trialkylplatinum(IV) cation before the nucleophile can coordinate to give the final product. Stereochemical change could occur at this stage, but the observed product ratios from **2** suggest that it does not. In reactions of **2b**, the electrophile could attack either of the mutually *trans* Pt–Me or Pt–CH_2 bonds and the



Scheme 2. NMR labelling.

data indicate high selectivity for Pt–Me cleavage. Reactivity can be increased by ring strain [17,18], and so the lower reactivity of the Pt–CH_2 bond must indicate that there is negligible ring strain in the platinacyclopentane ring. We note that the Me group *trans* to CH_2 in **2b** has a particularly low coupling constant $^1J(PtC) = 400$ Hz owing to the very strong *trans*-influence of the CH_2 group of the metallacycle. This probably reflects polarization $Pt^{\delta+}$-$Me^{\delta-}$ due to the strong donor influence of the $(CH_2)_4$ group, and the effect will also lead to enhanced reactivity of this Me–Pt bond towards electrophiles. The selectivity may thus be at least partly due to this *trans* bond weakening effect of the metallacycle.

4. Experimental details

^1H NMR spectra (see Scheme 2) were recorded on a Varian XL200 spectrometer, and ^{13}C, ^{31}P and ^{195}Pt NMR spectra were on a Varian XL300 spectrometer. References were TMS (^1H and ^{13}C), H_3PO_4 (^{31}P) and $K_2[PtCl_4]$ (^{195}Pt). IR spectra were recorded as a Perkin-Elmer 2000 FTIR spectrometer. The compounds $[PtMe_4(dppm)]$ and $[PtIMe(CH_2CH_2CH_2CH_2)(dppm)]$ were prepared as described previously [9]. All reactions with MeLi were carried out under dry argon.

4.1. *[PtMe$_2$(CH$_2$CH$_2$CH$_2$CH$_2$)(dppm)]*

A solution of MeLi in ether (5 ml, 1 M) was added dropwise to a suspension of $[PtIMe(CH_2CH_2CH_2CH_2)(dppm)]$ (0.25 g) in dry ether (35 ml) at 0°C and the mixture was stirred for 1 h at 0°C. A clear orange solution was obtained. This was treated with dilute aqueous NH_4Cl, and the organic layer was separated and dried over $MgSO_4$. The solvent was evaporated under vacuum to give the product, which was recrystallized from ether/hexane as a cream solid. Yield 0.14 g (56%), m.p. 126–130°C. Anal. Found: C, 54.2; H, 5.3. Calc. for $C_{31}H_{36}P_2Pt$: C, 55.9; H, 5.4%. (The poor carbon analysis is attributed to the limited stability of the complex). NMR in $CDCl_3$: ^1H, isomer A; $\delta = -0.12$ [t, 6H, $^2J(PtH) = 46$ Hz, $^3J(PH) = 15$ Hz, Me^aPt]; 2.28 [br.s., 4H, $^2J(PtH) = 72$ Hz, CH_2^b]; 1.1 [v.br.s., 4H, CH_2^c]; 4.76 [t, 2H, $^2J(PH) = 9$ Hz, $^3J(PtH) = 6.5$ Hz, CH_2^x]; isomer B; $\delta = -0.05$ [t, 3H, $^2J(PtH) = 43$ Hz, $^3J(PH) = 14$ Hz, Me^aPt]; 0.88 [m, 3H, $^2J(PtH) = 65$ Hz, Me^bPt]; 0.6–1.6 [m, 8H, CH_2^b and CH_2^c]; 4.34 [m, 1H, $^2J(HH) = 15$ Hz, $^2J(PH) = 10$ Hz, $^3J(PtH) = 19$ Hz, CH^x]; 4.98 [m, 1H, $^2J(HH) = 15$ Hz, $^2J(PH) = 9$ Hz, $^3J(PtH) = 14$ Hz, CH^x]; ^{13}C: isomer A, $\delta = -4.98$ [t, $^1J(PtC) = 429$ Hz, $^2J(PC) = 4$ Hz, C^a]; 35.3 [dd, $^1J(PtC) = 540$ Hz, $^2J(P^{a'}C) = 162$ Hz, $^2J(P^aC) = 5$ Hz, C^b]; 34.0 [s, C^c]; 39.3 [t, $^1J(PC) = 23$ Hz, CH_2P_2]; isomer B, $\delta = -12.0$ [t, $^1J(PtC) = 400$ Hz, $^2J(PC) = 4$ Hz, C^a]; 2.3 [dd, $^1J(PtC) = 610$ Hz, $^2J(P^aC) = 4$ Hz, $^2J(P^bC) = 124$ Hz, C^b]; 22.1 [t, $^1J(PtC) = 437$ Hz, $^2J(PC) = 3$ Hz, C^c]; 30.6 [dd, $^1J(PtC) = 580$ Hz, $^2J(P^bC) = 10$ Hz, $^2J(P^aC) = 119$ Hz, C^d]; 34 [s, C^e, C^f]; 41.0 [t, $^1J(PC) = 24$ Hz, CH_2P_2]; ^{31}P; isomer A, $\delta = -52.7$ [s, $^1J(PtP) = 845$ Hz, dppm]; isomer B, $\delta = -58.3$ [d, $^1J(PtP) = 961$ Hz, $^2J(PP) = 25$ Hz, P^b]; -60.6 [d, $^1J(PtP) = 837$ Hz, $^2J(PP) = 25$ Hz, P^a]; ^{195}Pt; isomer A, $\delta = -2892$ [t, $^1J(PtP) = 845$ Hz]; isomer B, $\delta = -2990$ [dd, $^1J(PtP) = 961$, 837 Hz].

4.2. *[PtMe(SO$_2$Me)(CH$_2$CH$_2$CH$_2$CH$_2$)(dppm)]*

A stream of SO_2 was passed through a solution of $[PtMe_2(CH_2CH_2CH_2CH_2)(dppm)]$ (0.10 g) in CH_2Cl_2 (10 ml) for 1 min, and the mixture was stirred for 15 min. The solvent was evaporated under vacuum to give the product, which was recrystallized from CH_2Cl_2/pentane as a cream solid. Yield 80%, m.p. 190–195°C. Anal. Found: C, 51.8; H, 4.7. Calc. for $C_{31}H_{36}O_2P_2PtS$: C, 51.0; H, 4.9%. NMR in $CDCl_3$: ^1H, isomer A; $\delta = 0.15$ [t, 3H, $^2J(PtH) = 59$ Hz, $^3J(PH) = 9$ Hz, Me^aPt]; 2.02 [s, 3H, Me^b]; 0.5–2.5 [br, $(CH_2)_4$]; 5.28 [m, 2H, CH_2^x]; isomer B; $\delta = 0.86$ [m, 3H, $^2J(PtH) = 65$ Hz, Me^aPt]; 1.94 [s, 3H, Me^b]; 0.5–2.5 [br, $(CH_2)_4$]; 5.12 [m, 2H, CH_2^x]; ^{13}C; isomer A, $\delta = -2.07$ [t, $^1J(PtC) = 553$ Hz, $^2J(PC) = 3$ Hz, C^a]; 46.3 [s, $^2J(PtC) = 58$ Hz, C^b]; 36.7 [dd, $^1J(PtC) = 550$ Hz, $^2J(P^aC) = 8$ Hz, $^2J(P^{a'}C) = 100$ Hz, C^c]; 34.1 [s, C^d]; 34.4 [t, $^1J(PC) = 25$ Hz, CH_2P_2]; isomer B, $\delta = 4.9$ [dd, $^1J(PtC) = 590$ Hz, $^2J(P^aC) = 4$ Hz, $^2J(P^bC) = 107$ Hz, C^a]; 47.3 [s, $^2J(PtC) = 46$ Hz, C^b]; 35.2 [s, $^1J(PtC) = 530$ Hz, C^c]; 34.5 [dd, $^1J(PtC)$ not resolved, $^2J(P^bC) = 7$ Hz, $^2J(P^aC) = 129$ Hz, C^d]; 33 [s, C^e, C^f]; 36.5 [t, $^1J(PC) = 24.5$ Hz, CH_2P_2]; ^{31}P; isomer A, $\delta = -39.8$ [s, $^1J(PtP) = 848$ Hz, dppm]; isomer B, $\delta = -49.8$ [d, $^1J(PtP) = 791$ Hz, $^2J(PP) = 23$ Hz, P^a]; -44.0 [d, $^1J(PtP) = 985$ Hz, $^2J(PP) = 23$ Hz, P^b]. IR (Nujol): $\nu(SO) = 1158$, 1035 cm^{-1}.

4.3. *[PtClMe($CH_2CH_2CH_2CH_2$)(dppm)]*

Acetyl chloride (3 μl) was added to a solution of [PtMe$_2$($CH_2CH_2CH_2CH_2$)(dppm)] (0.03 g) in CH_2Cl_2 (3 ml) and MeOH (2 ml) and the mixture was stirred for 10 min. The solvent was evaporated under vacuum to give the product as a cream solid. Yield 80%. NMR in $CDCl_3$: ^{1}H, isomer A; $\delta = 0.52$ [t, 3H, ^2J(PtH) = 75 Hz, ^3J(PH) = 8.5 Hz, Me^{a}Pt]; 0.7–3.5 [br, $(CH_2)_4$]; 4.31, 4.39 [m, 1H each, CH^x]; isomer B; $\delta = 1.9$ [m, 3H, ^2J(PtH) = 64 Hz, Me^{a}Pt]; 0.7–3.5 [br, $(CH_2)_4$]; 5.3 [m, 2H, CH_2^x]; ^{13}C; isomer A, $\delta = 0.53$ [t, ^1J(PtC) = 716 Hz, ^2J(PC) = 2 Hz, C^a]; isomer B, $\delta = 7.2$ [dd, ^1J(PtC) = 584 Hz, ^2J(P^{a}C) = 3 Hz, ^2J(P^{b}C) = 116 Hz, C^a]; no assignments were possible for CH_2 carbons which occurred from $\delta = 31$–39; ^{31}P, isomer A, $\delta = -40.9$ [s, ^1J(PtP) = 809 Hz, dppm]; isomer B, $\delta = -49.4$ [d, ^1J(PtP) = 784 Hz, ^2J(PP) = 36 Hz, P^a]; -45.8 [d, ^1J(PtP) = 895 Hz, ^2J(PP) = 36 Hz, P^b].

4.4. *[PtMe$_3$(SO_2Me)(dppm)]*

A stream of SO_2 was passed through a solution of [PtMe$_4$(dppm)] (0.10 g) in CH_2Cl_2 (10 ml) for 1 min and the mixture was stirred for 10 min. The solvent was evaporated under vacuum to give the product, which was recrystallized from CH_2Cl_2/pentane as a white solid. Yield 77%, m.p. 238°C. Anal. Found: C, 49.0; H, 4.7. Calc. for $C_{29}H_{34}O_2P_2PtS$: C, 49.5; H, 4.8%. NMR in $CDCl_3$: ^{1}H, $\delta = 0.25$ [t, 3H, ^2J(PtH) = 58 Hz, ^3J(PH) = 9 Hz, Me^{a}Pt]; 1.99 [s, 3H, Me^b]; 1.28 [m, 6H, ^2J(PtH) = 62 Hz, Me^{c}Pt]; 5.26 [m, 2H, CH_2^x]; ^{13}C; $\delta = -0.16$ [t, ^1J(PtC) = 504 Hz, ^2J(PC) = 3 Hz, C^a]; 6.01 [dd, ^1J(PtC) = 536 Hz, ^2J(P^{a}C) = 6 Hz, ^2J(P$^{a'}$C) = 112 Hz, C^c]; 46.5 [s, ^2J(PtC) = 55 Hz, C^b]; 34.8 [t, ^1J(PC) = 25 Hz, CH_2P_2]; ^{31}P; $\delta = -49.6$ [s, ^1J(PtP) = 917 Hz, dppm]; IR (Nujol): ν(SO) = 1155, 1036 cm^{-1}.

4.5. *[PtClMe$_3$(dppm)]*

Acetyl chloride (10 μL) was added to a solution of [PtMe$_4$(dppm)] (0.05 g) in CH_2Cl_2 (10 mL) and MeOH (2 ml) and the mixture was stirred for 10 min. The solvent was evaporated under vacuum to give the product as a white solid. Yield 80%; m.p. 202°C. (Found: C, 50.5; H, 4.8. Calc. for $C_{28}H_{31}ClP_2Pt$ C, 50.9, H, 4.7%). NMR in $CDCl_3$: ^{1}H, $\delta = 0.62$ [t, 3H, ^2J(PtH) = 75 Hz, ^3J(PH) = 16 Hz, Me^{a}Pt]; 1.45 [m, 6H, ^2J(PtH) = 62 Hz, Me^{b}Pt]; 4.37 [m, 1H, ^2J(HH) = 15 Hz, ^2J(PH) = 11 Hz, CH^x]; 5.51 [m, 1H, ^2J(HH) = 15 Hz, ^2J(PH) = 8 Hz, ^3J(PtH) = 16 Hz, CH^x]; ^{13}C; $\delta = -8.7$ [s, ^1J(PtC) = 655 Hz, C^a]; 6.5 [dd, ^1J(PtC) = 542 Hz, ^2J(P^{a}C) = 10 Hz, ^2J(P$^{a'}$C) = 128 Hz, C^b]; 37.6 [t, ^1J(PC) = 23 Hz, CH_2P_2]; ^{31}P, $\delta = -51.2$ [s, ^1J(PtP) = 878 Hz, dppm].

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