

BASICITY OF METAL CARBONYL COMPLEXES

XIX *. CO SUBSTITUTION IN AZACYMANTRENE AND REACTIONS OF $(\eta^5\text{-C}_4\text{H}_4\text{N})\text{Mn}(\text{CO})_2\text{PPh}_3$ WITH ELECTROPHILES. X-RAY CRYSTAL STRUCTURE OF $[(\text{PPh}_3)(\text{CO})_2\text{Mn}(\eta^5\text{-C}_4\text{H}_4\text{N})]_2\text{PdCl}_2$

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Summary

A convenient method for substituting a CO ligand in azacymantrene, $(\eta^5\text{-C}_4\text{H}_4\text{N})\text{Mn}(\text{CO})_3$ (I), by the interaction of I with PPh_3 in the presence of Me_3NO has been found. The reactions of $(\eta^5\text{-C}_4\text{H}_4\text{N})\text{Mn}(\text{CO})_2\text{PPh}_3$ (II) with electrophiles were studied. The nitrogen atom of the η -pyrrolyl ligand was shown to be the site having the largest basicity with respect to the proton (protonation in $\text{CF}_3\text{COOH}/\text{CH}_2\text{Cl}_2$) and aprotic acids (Zn, Cd, Hg, Al, Ga, Sn, Pd salts) in II. The structure of the trinuclear complex $[(\text{PPh}_3)(\text{CO})_2\text{Mn}(\eta^5\text{-C}_4\text{H}_4\text{N})]_2\text{PdCl}_2$ was established by an X-ray study: the crystals are monoclinic, a 9.0165(5), b 15.748(1), c 16.179(1) Å, β 103.37(1)°, Z = 2, space group $P2_1/c$; the palladium coordination environment is square-planar, Pd–N 2.033(2) and Pd–Cl 2.306(1) Å.

Introduction

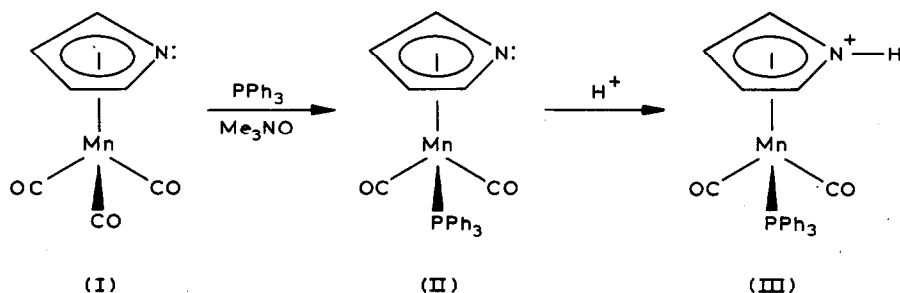
Cymantrene, $(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_3$, and its phosphine derivatives are organometallic Lewis bases which react with electrophiles. IR and ^1H NMR spectra show that protonation of $(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_2\text{PR}_3$ complexes with strong acids leads to the addition of a proton at the manganese atom [2–4]. The reactions of these compounds with aprotic acids such as mercury(II), aluminium, gallium and tin(IV) salts give rise to adducts with the manganese atom [5–7]. Thus in $(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_2\text{PR}_3$ (R = alkyl, Ph) complexes the manganese atom is the most basic site, with respect to the proton and aprotic acids.

* For part XVIII see ref. 1.

Unlike cymantrene, the chemistry of its η^5 -pyrrolyl analogue, i.e. azacycmantrane, (η^5 -C₄H₄N)Mn(CO)₃(I), has only recently been thoroughly studied [8]. However, no convenient method of substituting the CO ligand in I exists as yet: photochemical substitution of CO by EPh₃ (E = P, As, Sb) in I proceeds with a very low yield, and thermal substitution results in the decomposition of I [9].

Results and discussion

We found that the reaction of I with PPh₃ in the presence of Me₃NO affords the complex (η^5 -C₄H₄N)Mn(CO)₂PPh₃ (II) in a 30% yield. The reactions of II with electrophiles were studied. In the molecule of II the attack of the electrophile can be directed towards a number of basic sites, such as the nitrogen of the pyrrolyl ligand, the manganese atom, etc. The totality of the data obtained indicates that, as a result of dissolution in CF₃COOH or CF₃COOH/CH₂Cl₂ mixtures, the proton adds to the nitrogen atom of II, which involves typical variations in the IR and ¹³C NMR and ³¹P NMR spectra (Table 1).



On passing from CH₂Cl₂ to CF₃COOH/CH₂Cl₂ mixtures, the $\nu(\text{CO})$ modes in the IR spectra shift by 40–50 cm⁻¹, which is much smaller than that in the protonation of (η^5 -C₅H₅)Mn(CO)₂PPh₃ at the manganese atom ($\Delta\nu$ 100–120 cm⁻¹ [2]). In the ¹³C{¹H}NMR spectrum of the protonated form of II, the signal from the carbon α -atoms of the pyrrolyl ligand during protonation shifts upfield ($\Delta\delta$ ~ 10 ppm) whereas that from the β -carbons remains practically unchanged. In the case of the CO carbons, the signal shifts upfield ($\Delta\delta$ ~ 6 ppm) and retains its doublet structure with the constant $J(^{13}\text{C } ^{31}\text{P})$ 22–25 Hz. Note for comparison that in the protonation of (η^5 -C₅H₅)Mn(CO)₂PPh₃ at the manganese atom, the signal from the

TABLE 1
SPECTRAL DATA FOR THE PROTONATION OF COMPLEX II

Solvent	IR spectra $\nu(\text{CO})$ (cm ⁻¹)	¹³ C{ ¹ H}NMR spectra (δ , ppm) C ₄ H ₄ N		CO	³¹ P{ ¹ H}NMR spectra (δ , ppm) from H ₃ PO ₄
		α	β		
CH ₂ Cl ₂	1950 1882	107.54	85.68	231.5 ($J = 22$ Hz)	85.8
CF ₃ COOH/CH ₂ Cl ₂	1990 1930 (1/100 mix- ture)	97.03 (1/1 mix- ture)	85.64	225.6 ($J = 25$ Hz)	77.8

C_5H_5 carbons shifts downfield ($\Delta\delta \sim 6$ ppm) whereas that from the CO carbons splits into two signals which are shifted upfield ($\Delta\delta \sim 10$ and 14 ppm, respectively), as compared with the CH_2Cl_2 spectrum [4].

During protonation of II the signal from the phosphorus atom in the $^{31}P \{^1H\}$ NMR spectrum shifts upfield ($\Delta\delta \sim 7$ ppm). However, when the spectrum is recorded without 1H decoupling this signal retains its singlet structure. In contrast, in the manganese protonated forms of $(\eta^5-C_5H_5)Mn(CO)_2PPh_3H^+$, the signal arising from the ^{31}P nucleus, when 1H is not decoupled, splits into a doublet with the constant $J(^1H \ ^{31}P)$ 50–70 Hz [4].

During protonation of II, variations in the ^{13}C and ^{31}P NMR spectra differ very much from what is observed in the protonation of $C_5H_5Mn(CO)_2PPh_3$ at the manganese atom.

Complex II undergoes protonation at the nitrogen atom, even in CF_3COOH/CH_2Cl_2 mixtures (1/100), whereas the complex $(\eta^5-C_5H_5)Mn(CO)_2PPh_3$ adds a proton to the metal only in highly acidic conditions (in HSO_3F/CH_2Cl_2 at $-70^\circ C$ [4]). The method of competing protonation monitored by the use of ^{31}P NMR spectra revealed compound II to be a stronger base than PPh_3 . On addition of one equivalent of CF_3COOH to the equimolar II/ PPh_3 mixture in CH_2Cl_2 at $-50^\circ C$, first the protonation of II is observed and then, after completion of the protonation of II, the ^{31}P NMR spectrum shows a signal from PPh_3H^+ on further addition of the acid.

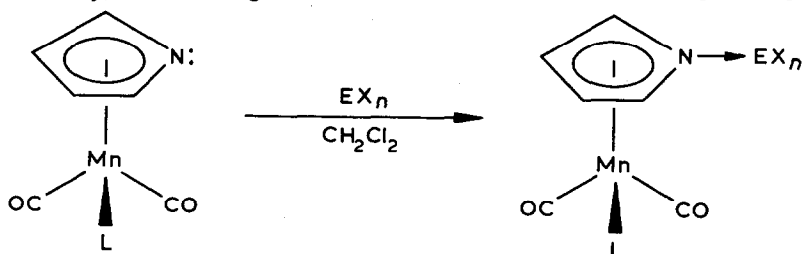
Complexes I and II react with the aprotic acids EX_n in CH_2Cl_2 . This reaction, as in the case of protonation, is accompanied by a relatively small increase 20–40 cm^{-1} , in the $\nu(CO)$ frequencies (Table 2). Also, the following changes in the IR spectra take place. For complex I having an $Mn(CO)_3$ fragment, coordination with the aprotic acids gives rise to a small splitting of the band of the degenerate $\nu(CO)$ mode into two components, $\Delta\nu$ 15–20 cm^{-1} , indicating a decrease in the local symmetry of the metal carbonyl fragment. When complex II coordinates with the

TABLE 2

INTERACTION OF $(\eta^5-C_4H_4N)Mn(CO)_3$ AND $(\eta^5-C_4H_4N)Mn(CO)_2PPh_3$ WITH APROTIC ACIDS IN CH_2Cl_2

Aprotic acid	$\nu(CO)$ frequencies, (cm^{-1})	
	$(\eta^5-C_4H_4N)Mn(CO)_3$	$(\eta^5-C_4H_4N)Mn(CO)_2PPh_3$
In CH_2Cl_2 without acid	1955br 2040	1880 1948
+ $HgCl_2$	1962br 2048	1898 1961
+ $Hg(OCOCH_3)_2$	1960/1980sh 2050	1900 1972
+ $Hg(OCOCF_3)_2$	1980br 2060	1918 1978
+ $Cd(OCOCF_3)_2$	1964/1980sh 2054	1905 1965
+ $ZnCl_2$	1975/1985sh 2058	1910 1972
+ ZnI_2	1972/1985sh 2056	1910 1971
+ $AlCl_3$	—	1923 1982
+ $AlBr_3$	—	1927 1984
+ $GaCl_3$	1985/1998sh 2065	1930 1985
+ $SnCl_4$	1975/1995sh 2060	1935 1991
+ $SnBr_4$	1975/1990sh 2056	1935 1991
+ $SbCl_3$	—	1935 1990
+ $PdCl_2$	1962/1982 2050	1902 1965

aprotic acids, the two $\nu(\text{CO})$ modes increase, although in the spectrum of the coordination product they retain equal intensities, as in the case of the starting complex II. The ratio between the intensities, A , of the symmetric and asymmetric $\nu(\text{CO})$ modes for the $\text{M}(\text{CO})_2$ fragment is known to depend on the angle between the carbonyl groups (α) [10]: $A_{\text{asym}}/A_{\text{sym}} = \cot^2(\alpha/2)$. Since in the course of coordination the two bands of the $\nu(\text{CO})$ mode retain equal intensities, it is obvious that the OC-Mn-CO angle remains unchanged, close to 90° , i.e. the manganese atom does not participate in coordination. The relatively small shift of the $\nu(\text{CO})$ modes and the fact that in the case of II the above mentioned angle remains close to 90° indicate that coordination of the aprotic acids with I and II occurs via the nitrogen atom. On the other hand, coordination of $(\eta^5\text{-C}_5\text{H}_5)\text{Mn}(\text{CO})_2\text{PPh}_3$ with the aprotic acids, HgCl_2 , $\text{Hg}(\text{OCOCF}_3)_2$ and SnCl_4 , at the manganese atoms gives rise to a much larger increase in the $\nu(\text{CO})$ modes and a drastic change in their intensity, as the α angle increases from ca. 90° to $115\text{--}118^\circ$ [7,11,12].



(I, L = CO ; II, L = PPh_3)

To confirm that coordination occurs at the nitrogen atom, we isolated the product of the II- PdCl_2 coordination and established its structure by an X-ray study.

The molecular structure of *trans*- $[(\text{Ph}_3\text{P})(\text{CO})_2\text{Mn}(\eta^5\text{-C}_4\text{H}_4\text{N})]_2\text{PdCl}_2$ (III) is shown in Fig. 1; the bond lengths and angles are listed in Tables 3 and 4. The palladium atom is in the crystallographic centre of symmetry and has a strictly planar *trans*-square coordination with two chlorine atoms and two nitrogens of the η -pyrrolyl ligands. The Pd-Cl and Pd-N distances are normal [13]. The pyrrolyl rings coordinate the manganese atoms by the η^5 -type, as in the case of other azacymantrene derivatives studied earlier [14,15]. The pyrrolyl rings are planar and form angles of 29.9° with the coordination plane of the palladium atom. The Pd-N bonds are tilted out of the ring planes by 13.6° away from the manganese atoms.

Generally, coordination of the manganese atom is similar to that observed in the complex $\eta^5\text{-C}_5\text{H}_5(\text{CO})_2\text{Fe-C}_3\text{H}_4\text{Mn}(\text{CO})_2\text{PPh}_3$, with an $\eta^1\text{-}\eta^5$ -cyclopentadienyl ligand at the manganese atom instead of a pyrrolyl one [16]. The conformation of the $\text{Mn}(\text{CO})_2\text{PPh}_3$ fragment relative to the pyrrolyl ring is such that the C(1) and P atoms are eclipsed with the C(3) and C(6) atoms, respectively, and the C(2) atom is located above the midpoint of the C(4)-C(5) bond.

The H(36) atoms of the triphenylphosphine ligands are rather close to the Pd atom (the Pd...H(36) distance is $2.68(4)\text{\AA}$ * in contrast with the sum of the Pd and

* For the position of H(36) refined by the least-squares technique. In this case, the C(36)-H(36) distance is unreliably short $0.88(4)\text{\AA}$. The calculated position of H(36), assuming a C(36)-H(36) distance of $1.08\text{\AA}$, is even closer to the metal, Pd...H(36) $2.50\text{\AA}$.

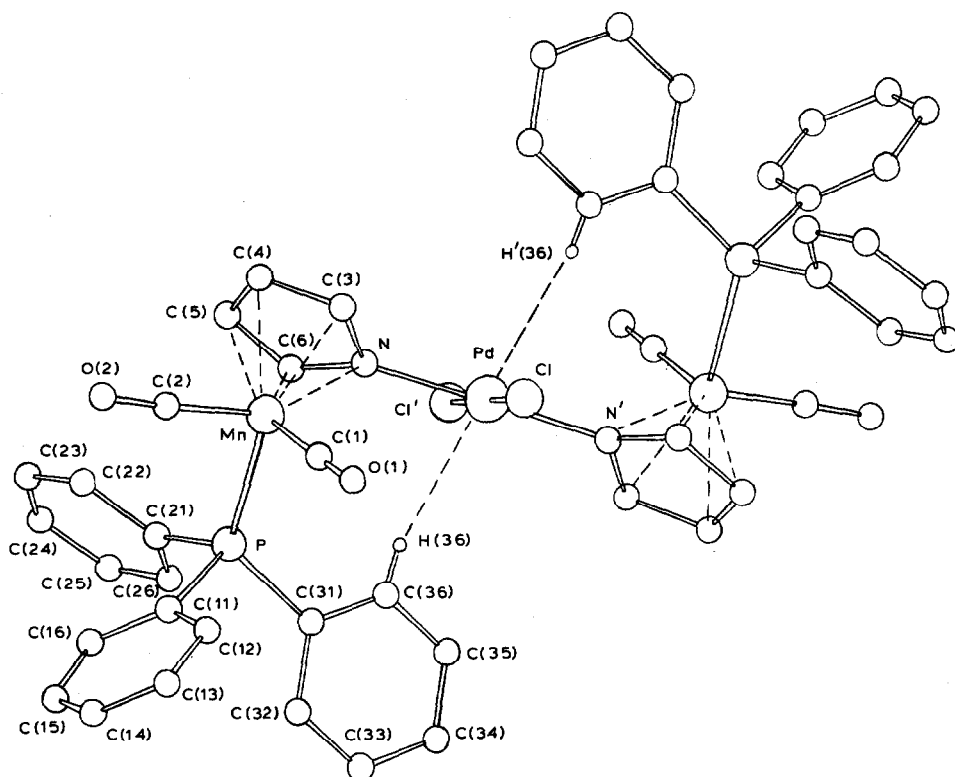


Fig. 1. Molecular structure of $[(PPh_3)(CO)_2Mn(\eta^5-C_4H_4N)]_2PdCl_2$ (III), including atomic numbering. The hydrogen atoms, except H(36), have been omitted.

TABLE 3

BOND DISTANCES, d (Å)

Bond	d	Bond	d	Bond	d
Pd-Cl	2.306(1)	O(1)-C(1)	1.146(4)	C(21)-C(22)	1.399(5)
Pd-N	2.033(2)	O(2)-C(2)	1.152(5)	C(21)-C(26)	1.372(6)
Mn-P	2.275(1)	N-C(3)	1.399(4)	C(22)-C(23)	1.376(6)
Mn-N	2.145(2)	N-C(6)	1.382(4)	C(23)-C(24)	1.352(8)
Mn-C(1)	1.783(4)	C(3)-C(4)	1.387(5)	C(24)-C(25)	1.373(8)
Mn-C(2)	1.775(4)	C(4)-C(5)	1.403(5)	C(25)-C(26)	1.405(7)
Mn-C(3)	2.119(3)	C(5)-C(6)	1.399(5)	C(31)-C(32)	1.395(5)
Mn-C(4)	2.152(4)	C(11)-C(12)	1.386(5)	C(31)-C(36)	1.380(5)
Mn-C(5)	2.168(3)	C(11)-C(16)	1.392(6)	C(32)-C(33)	1.377(6)
Mn-C(6)	2.139(3)	C(12)-C(13)	1.383(7)	C(33)-C(34)	1.361(6)
P-C(11)	1.832(4)	C(13)-C(14)	1.372(9)	C(34)-C(35)	1.367(6)
P-C(21)	1.831(3)	C(14)-C(15)	1.353(9)	C(35)-C(36)	1.382(6)
P-C(31)	1.836(3)	C(15)-C(16)	1.410(8)		
Pd...H(36)	2.68(4)	C(36)-H(36)	0.88(4)		
	2.50		1.08		
				refined position H(36)	
				calculated	

TABLE 4

BOND ANGLES, $\omega(^{\circ})$

Angle	ω	Angle	ω	Angle	ω
Cl-Pd-N	90.08(7)	Mn-C(1)-O(1)	176.6(3)	P-C(21)-C(26)	123.0(3)
Cl-Pd-H(36) 103(1);	104 ^a	Mn-C(2)-O(2)	175.5(3)	C(22)-C(21)-C(26)	118.5(3)
N-Pd-H(36) 73(1);	71 ^a	N-C(3)-C(4)	109.6(3)	C(21)-C(22)-C(23)	121.1(4)
Pd-H(36)-C(36) 157(4);	154 ^a	C(3)-C(4)-C(5)	107.5(3)	C(22)-C(23)-C(24)	119.8(4)
P-Mn-C(1)	94.5(1)	C(4)-C(5)-C(6)	106.8(3)	C(23)-C(24)-C(25)	121.0(5)
P-Mn-C(2)	88.5(1)	N-C(6)-C(5)	109.9(3)	C(24)-C(25)-C(26)	119.6(5)
C(1)-Mn-C(2)	91.8(2)	P-C(11)-C(12)	118.4(3)	C(21)-C(26)-C(25)	120.0(4)
Mn-P-C(11)	115.6(1)	P-C(11)-C(16)	122.7(3)	P-C(31)-C(32)	122.5(3)
Mn-P-C(21)	111.2(1)	C(12)-C(11)-C(16)	118.8(4)	P-C(31)-C(36)	119.7(3)
Mn-P-C(31)	120.5(1)	C(11)-C(12)-C(13)	121.8(4)	C(32)-C(31)-C(36)	117.8(3)
C(11)-P-C(21)	103.4(2)	C(12)-C(13)-C(14)	119.4(5)	C(31)-C(32)-C(33)	120.6(4)
C(11)-P-C(31)	100.7(2)	C(13)-C(14)-C(15)	119.8(6)	C(32)-C(33)-C(34)	120.9(4)
C(21)-P-C(31)	103.4(1)	C(14)-C(15)-C(16)	122.1(6)	C(33)-C(34)-C(35)	119.3(4)
Pd-N-C(3)	123.7(2)	C(11)-C(16)-C(15)	118.1(5)	C(34)-C(35)-C(36)	120.6(4)
Pd-N-C(6)	127.9(2)	P-C(21)-C(22)	118.4(3)	C(31)-C(36)-C(35)	120.9(4)
C(3)-N-C(6)	106.2(2)				

^a For the calculated position of H(36).

H van der Waals radii of 3.1 Å [17]) and complete its environment to a distorted octahedral one. Similar short non-valent Pd...H contacts of 2.36–2.66 Å have been observed in a number of other complexes of the type L_2PdCl_2 , where $L = Me_2C=NNMePh$ [18], 1-methylcytosine [19] and $MeN=CHPh$ [20]. During the coordination of II with all the EX_n studied, the IR spectra change in practically the same way the $\nu(CO)$ frequencies show similar shifts and retain equal intensities, indicating that coordination of the aprotic acids in any case occurs with the nitrogen atom. Thus in complex II the nitrogen of the η -pyrrolyl ligand is the most basic site both with respect to the proton and the aprotic acids.

Experimental

$(\eta^5-C_4H_4N)Mn(CO)_2PPh_3$ (II)

Complex I (0.5 g, 2.44 mmol) and PPh_3 (0.7 g, 2.7 mmol) in dry benzene (15–20 ml) were mixed under an inert atmosphere for 10–15 h at 55–60°C with the addition of portions of non-aqueous Me_3NO (0.4 g, 5–6 mmol). The process of CO substitution was followed by IR spectra, which showed a gradual weakening of the $\nu(CO)$ mode $Mn(CO)_3$ at 1950–1960 cm^{-1} and 2040 cm^{-1} and also the appearance of a mode of II at 1950 and 1882 cm^{-1} . After completion of the reaction, the mixture was hydrolysed, and the benzene layer was separated, washed with water, and dried over $CaCl_2$. After removal of the benzene, the residue was chromatographed on a column with neutral Al_2O_3 . Excess PPh_3 was eluted with petroleum ether, and complex II with a petroleum ether/ CH_2Cl_2 mixture. The procedure gave the title complex (0.33 g, ca. 30%), identical to the one described earlier [9].

$[(PPh_3)(CO)_2Mn(\eta^5-C_4H_4N)]_2PdCl_2$ (III)

To 0.05 g (0.13 mmol) of $(PhCN)_2PdCl_2$ in dry benzene (10 ml) II (0.12 g, 0.27 mmol) was added dropwise with stirring in benzene (10 ml). The mixture was stirred

for 3 h at room temperature. The solvent was partially removed and a red-brown precipitate was isolated from the residue on addition of pentane. Then it was recrystallized from the pentane/CH₂Cl₂ mixture (1/5). Yield: 0.08 g (56%) of the title complex, decomposition temperature ca. 134°C. The red-brown crystals of III were stable in air, poorly soluble in hydrocarbon solvents, and reasonably soluble in acetone and chlorine-containing solvents. Found: C, 54.75; H, 4.06; Cl, 6.67; P, 5.61. Calcd.: C₄₈H₃₈Cl₂N₂O₄P₂Pd C, 54.60; H, 3.60; Cl, 6.73; P, 5.88%. IR spectrum (CH₂Cl₂): $\nu(\text{CO})$ 1965, 1902 cm⁻¹. ¹H NMR spectrum (ppm, CDCl₃): 4.787 singlet, β H; 6.287 singlet, α H; 7.6 multiplet, C₆H₅.

The X-ray study was carried out with a Hilger-Watts four-circle autodiffractometer (Mo-K α radiation, graphite monochromator) at room temperature. Calculations were performed with an Eclipse S/200 computer using INEXTL programs [21]. Crystal data of III: monoclinic, *a* 9.0165(5), *b* 15.748(1), *c* 16.179(1) Å, β 103.37(1)°, *V* 2235.1(2) Å³, *Z* = 2, C₄₈H₃₈Cl₂N₂O₄P₂Pd, $\rho_{\text{calcd.}}$ 1.57 g cm⁻³, space group *P*2₁/*c*. The intensities of 3398 independent reflections with $I \geq 2\sigma$ were measured by the $\theta/2\theta$ scan mode ($\theta \leq 27^\circ$). The structure was solved by the heavy-atom method and

TABLE 5

ATOMIC COORDINATES ($\times 10^4$; for Mn,P and Cl $\times 10^5$; for H $\times 10^3$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	Atom	<i>x</i>	<i>y</i>	<i>z</i>
Pd	0	0	0	H(3)	338(5)	-16(3)	-24(3)
Mn	30775(5)	16475(3)	-2442(3)	H(4)	429(4)	67(2)	-136(2)
P	19199(9)	28570(5)	503(5)	H(5)	232(4)	172(2)	-203(2)
Cl	20217(8)	-4337(5)	10679(5)	H(6)	18(4)	154(2)	-131(2)
O(1)	4626(4)	1281(2)	1518(2)	H(12)	413(5)	296(3)	164(3)
O(2)	5629(3)	2664(2)	-527(2)	H(13)	565(6)	383(3)	254(3)
N	1461(1)	633(1)	-569(1)	H(14)	565(10)	527(6)	202(6)
C(1)	3986(4)	1437(2)	837(2)	H(15)	413(5)	553(3)	65(3)
C(2)	4634(4)	2277(2)	-384(2)	H(16)	257(4)	464(2)	-13(2)
C(3)	2926(3)	343(2)	-574(2)	H(22)	275(5)	342(3)	-147(3)
C(4)	3465(4)	772(2)	-1194(2)	H(23)	181(5)	411(5)	-272(3)
C(5)	2331(4)	1348(2)	-1584(2)	H(24)	-75(6)	455(3)	-291(3)
C(6)	1120(4)	1255(2)	-1186(2)	H(25)	-201(6)	421(3)	-193(3)
C(7)	3190(4)	3666(2)	647(2)	H(26)	-118(4)	354(2)	-74(2)
C(12)	4091(4)	3449(3)	1435(3)	H(32)	58(5)	402(3)	112(3)
C(13)	5018(5)	4037(4)	1942(3)	H(33)	-105(6)	387(3)	186(3)
C(14)	5038(6)	4860(4)	1667(4)	H(34)	-262(5)	269(3)	183(3)
C(15)	4183(8)	5083(4)	895(5)	H(35)	-211(7)	151(4)	107(4)
C(16)	3219(6)	4500(3)	366(3)	H(36)	-27(5)	165(3)	33(3)
C(21)	977(4)	3410(2)	-925(2)				
C(22)	1819(5)	3578(3)	-1533(2)				
C(23)	1190(6)	4018(3)	-2264(3)				
C(24)	-267(7)	4293(3)	-2403(3)				
C(25)	-1144(6)	4126(4)	-1832(3)				
C(26)	-508(5)	3680(3)	-1081(3)				
C(31)	448(3)	2813(2)	658(2)				
C(32)	124(4)	3502(2)	1131(3)				
C(33)	-1036(5)	3454(3)	1552(3)				
C(34)	-1902(4)	2741(3)	1511(3)				
C(35)	-1609(5)	2063(3)	1044(3)				
C(36)	-442(4)	2097(2)	622(3)				

refined by a block-diagonal least-squares technique first with all the non-hydrogen atoms in an anisotropic approximation and then with the hydrogen atoms in an isotropic approximation to $R = 0.031$ and $R_w = 0.039$ using the weighting scheme $W^{-1} = \sigma^2 F + (0.015 F_o)^2$. Atomic coordinates are listed in Table 5.

The IR spectra were obtained on a UR-20 spectrophotometer in CaF_2 cells (0.2 mm). The ^1H NMR spectrum of III and the ^{13}C NMR spectrum of the neutral and protonated forms of complex II were measured on a Bruker WP-200 instrument. ^{31}P NMR spectra were taken on a Bruker HX-90 spectrometer.

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