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A transition-metal diaminophosphonate complex: synthesis and structure of $[(\eta^5\text{-}C_5H_5)(CO)_2Fe\{P(O)(NEt_2)_2\}]_2FeCl_2$

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Abstract

$(\eta^5\text{-}C_5H_5)(CO)_2FeCl$ reacts with $P(OMe)(NEt_2)_2$ in benzene to give isolable $[(\eta^5\text{-}C_5H_5)(CO)_2Fe\{P(OMe)(NEt_2)_2\}]Cl$, which is converted by thermolysis into $[(\eta^5\text{-}C_5H_5)(CO)_2Fe\{P(O)(NEt_2)_2\}]_2FeCl_2$, crystallizing in the monoclinic space group $C2$ with a 18.700(4), b 9.065(1), c 16.608(3) Å, β 135.54(1)°, and $Z = 4$. X-Ray diffraction has revealed that the $P(O)(NEt_2)_2$ group covalently bonds to the iron atom of the $(\eta^5\text{-}C_5H_5)(CO)_2Fe$ moiety through the P atom and also coordinates to $FeCl_2$ through the phosphoryl oxygen, and that one of the nitrogen atoms in the $P(O)(NEt_2)_2$ group has a planar geometry. $[(\eta^5\text{-}C_5H_5)(CO)_2Fe\{P(O)(NEt_2)_2\}]$ is obtained by treatment with pyridine.

Arbuzov-like dealkylations of trialkyl phosphites with transition-metal complexes containing a labile nucleophilic ligand such as a halogen atom are known to produce transition-metal phosphonate complexes [1], which are of interest because

$$L_nMX + P(OR)_3 \rightarrow L_nM\{P(O)(OR)_2\} + RX$$

the pentavalent phosphorus atom is covalently bonded to the transition-metal in these complexes. However, the reaction has been reported only for phosphites, $P(OR)_3$ and a few alkylsubstituted phosphites, namely the phosphonites, $P(OR)_2R$, and the phosphinites, $P(OR)R_2$ [2]. We report here the first example of the reaction of an amino-substituted phosphite, $P(OMe)(NEt_2)_2$ with $Cp(CO)_2FeCl$, where Cp stands for η^5-cyclopentadienyl group.

Results and discussion

The reaction sequence mentioned here is summarized in Scheme 1.

Treatment of $Cp(CO)_2FeCl$ with $P(OMe)(NEt_2)_2$ in benzene at room temperature gave a yellow powder (I). Its spectroscopic data are given in Table 1. The

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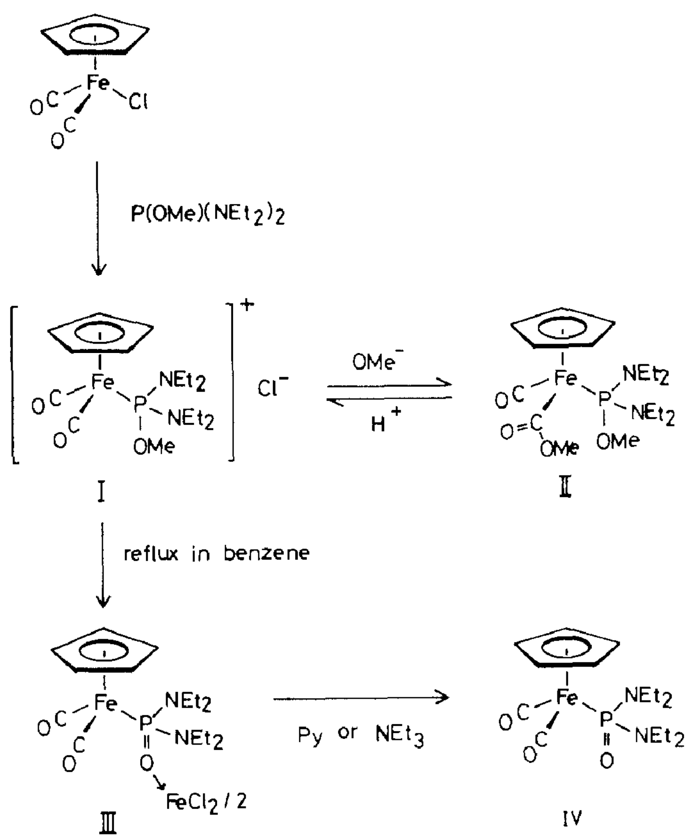


Scheme 1

signals from the Cp hydrogens in I in the ^1H NMR spectrum (5.47 ppm) are observed at a lower field by about 0.4 ppm than those for $Cp(CO)_2FeCl$ (5.08 ppm), suggesting that I is a cationic complex. The ^{31}P NMR resonance of I (153.7 ppm) is observed at slightly lower field than that of free $P(OMe)(NEt_2)_2$ (135.9 ppm), indicating that $P(OMe)(NEt_2)_2$ coordinates to a transition-metal through its lone pair. Thus, isolated complex I is formulated as $[Cp(CO)_2Fe\{P(OMe)(NEt_2)_2\}]^+Cl^-$. A cationic transition-metal complex containing a coordinated phosphite is usually too reactive to be isolated as a halide salt in the Arbuzov-like dealkylation. But complex I, corresponding to an intermediate of the Arbuzov-like reaction, is stable in C_6H_6, CH_2Cl_2, and CH_3OH.

Since a cationic iron complex containing a diaminophosphite as a ligand could be isolated as above, we examined the reactivity of I with a more nucleophilic agent than Cl^-. When a suspension of NaOMe in benzene was added to I, and the solution became homogeneous. Complex II was isolated from the solution. Its IR spectrum (Table 1) showed patterns characteristic of a methoxycarbonyl ligand (ν(CO) 1620 cm^{-1}), and a terminal carbonyl ligand (ν(CO) 1940 cm^{-1}). From the IR data, supplemented with ^1H and ^{31}P NMR data, II was identified as a methoxycarbonyl complex, $Cp(CO)Fe(COOMe)\{P(OMe)(NEt_2)_2\}$. Hence, we conclude that the OMe^- anion selectively attacks the carbonyl carbon instead of the methoxy carbon in the coordinated $P(OMe)(NEt_2)_2$. Complex I is readily re-formed in reactions of II with acids such as CF_3COOH, liberating MeOH.

Table 1

Spectroscopic data

Complex	IR[a] (ν(CO) (cm^{-1}))	^{1}H NMR[b] (δ (ppm))	^{31}P NMR[a,c] (δ (ppm))
$[Cp(CO)_2Fe\{P(OMe)(NEt_2)_2\}]^+ Cl^-$	2052	1.17 (t, *J* 6.1 Hz, 12H, $CH_2C\mathit{H}_3$)	153.7 (s)
(I)	2004	3.10 (m, 8H, $C\mathit{H}_2CH_3$)	
		3.55 (d, *J* 14.0 Hz, 3H, OCH_3)	
		5.47 (s, 5H, C_5H_5)	
$Cp(CO)Fe(COOMe)\{P(OMe)(NEt_2)_2\}$	1940 [d]	1.07 (t, *J* 6.3 Hz, 12H, $CH_2C\mathit{H}_3$)	182.2 (s) [d]
(II)	(Fe–CO)	3.10 (m, 8H, $C\mathit{H}_2CH_3$)	
	1620	3.44 (s, 3H, $COOCH_3$)	
	(COOMe)	3.45 (d, *J* 12.2 Hz, 3H, $POCH_3$)	
		4.61 (s, 5H, C_5H_5)	
$[Cp(CO)_2Fe\{P(O)(NEt_2)_2\}]_2FeCl_2$	2030 [d]	–	–
(III)	1977		
$Cp(CO)_2Fe\{P(O)(NEt_2)_2\}$	2020	1.10 (t, *J* 7.0 Hz, 12H, CH_3)	98.2 (s)
(IV)	1968	3.07 (m, 8H, CH_2)	
		4.93 (s, 5H, C_5H_5)	

[a] In CH_2Cl_2 unless otherwise noted. [b] In $CDCl_3$. [c] Relative to 85% H_3PO_4 as an external standard (downfield positive). [d] In C_6H_6.

The thermolysis of I has been attempted. A suspension of complex I in benzene was refluxed for 3 h to give yellow crystals (III). The IR absorption in the ν(CO) region appeared at 2030 and 1977 cm^{-1} but the ^{1}H and ^{31}P NMR signals were broadened, indicating the formation of some paramagnetic material.

Table 2

Summary of crystal data for $[Cp(CO)_2Fe\{P(O)(NEt_2)_2\}]_2FeCl_2$

Formula	$C_{30}H_{50}Cl_2Fe_3N_4O_6P_2$
Crystal system	monoclinic
Space group	*C*2
Cell constants	
a, Å	18.700(4)
b, Å	9.065(1)
c, Å	16.608(3)
β, deg	135.54(1)
V, Å^3	1971.9(6)
Z	4
D_{calcd}, g cm^{-3}	1.455
μ_{calcd}, cm^{-1}	12.087
Crystal size, mm	0.57 × 0.15 × 0.30
Radiation	Mo-K_α (λ 0.71073Å) graphite monochromator
Scan technique	ω–2θ
Scan range	$3° < 2\theta < 55°$
Unique data	3671
Unique data, $F_o > 3\sigma(F_o)$	1938
R_1	0.059
R_2	0.060

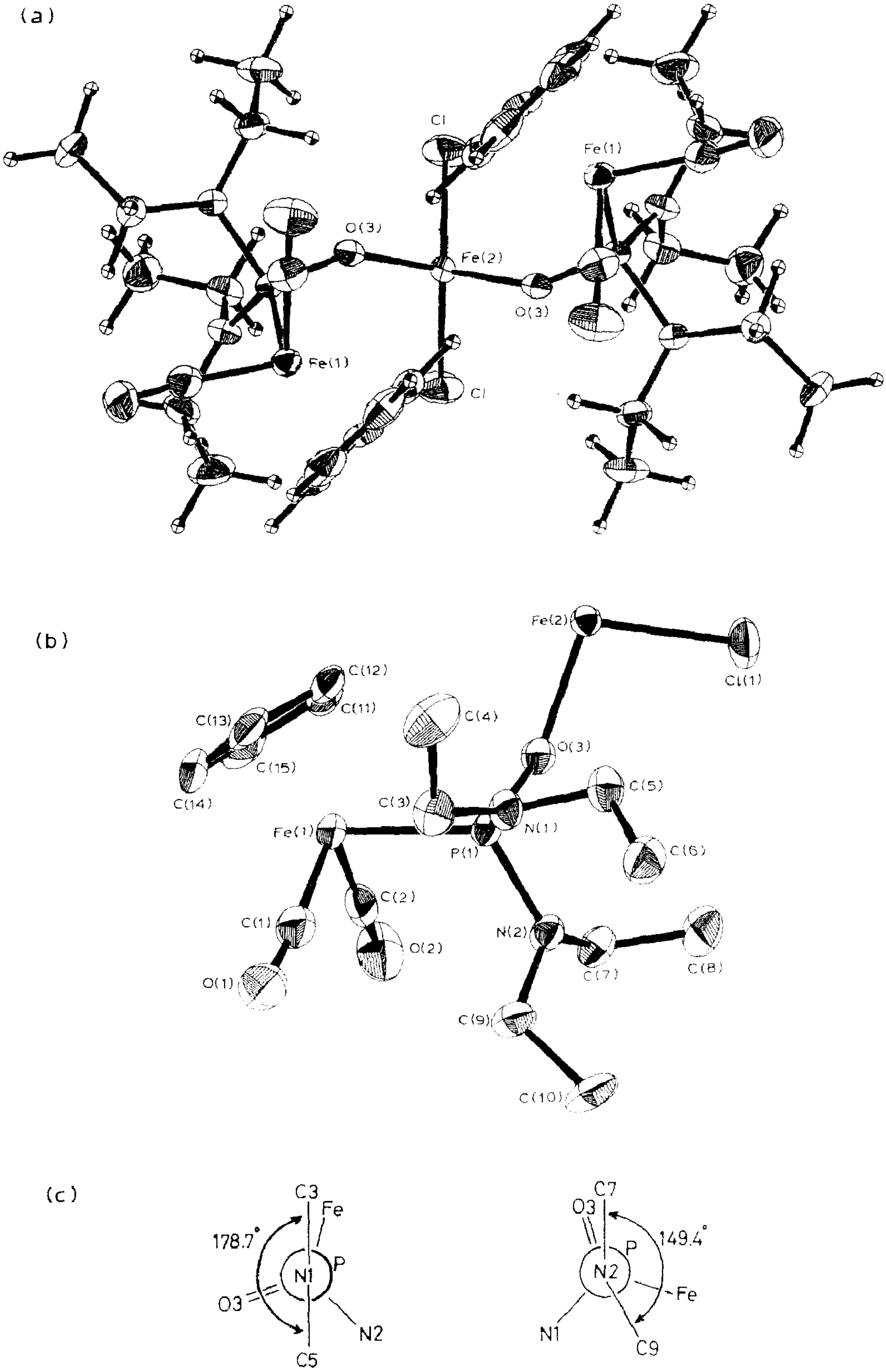


Fig. 1. (a) ORTEP drawing of the molecular structure of III. (b) ORTEP drawing of the half of the molecule with atomic labels. All hydrogen atoms are omitted for clarity. (c) Newman projection down the N–P bonds.

Table 3

Fractional atomic coordinates ($\times 10^4$) and thermal parameters (Å^2) for the non-hydrogen atoms

Atom	x	y	z	B_{eq}
Fe1	2315(1)	4493(2)	1734(1)	3.5
Fe2	0(0)	64(0)	0(0)	2.6
Cl1	4293(2)	4371(4)	479(3)	6.0
P1	2072(2)	2478(3)	2300(2)	2.9
O1	4422(6)	5165(14)	3815(7)	8.3
O2	1436(8)	6290(10)	2314(9)	7.4
O3	968(4)	2000(8)	1346(5)	3.5
N1	2844(6)	1094(9)	2710(7)	3.7
N2	2245(5)	2724(9)	3442(6)	3.5
C1	3589(5)	4841(15)	3033(9)	5.4
C2	1797(8)	5574(12)	2101(9)	4.6
C3	3778(8)	1162(13)	2955(9)	4.8
C4	3784(11)	18(18)	2282(12)	7.3
C5	2611(7)	−368(13)	2886(9)	4.4
C6	3499(9)	−963(15)	4090(10)	6.0
C7	1349(8)	3158(12)	3234(8)	4.2
C8	944(10)	1834(16)	3375(12)	6.1
C9	3223(7)	3235(14)	4509(8)	4.5
C10	3550(9)	2677(18)	5597(8)	6.3
C11	1071(8)	4235(14)	−34(8)	4.7
C12	1834(8)	3217(13)	357(9)	4.5
C13	2681(9)	4002(15)	819(9)	5.4
C14	2464(11)	5577(19)	740(10)	7.4
C15	1466(11)	5645(17)	191(10)	6.5

In order to determine the structure of III, an X-ray diffraction study was carried out. X-ray data are listed in Table 2. Figure 1(a) illustrates the structure, revealing that each $Cp(CO)_2Fe\{P(O)(NEt_2)_2\}$ moiety coordinates to the $FeCl_2$ fraction through its phosphoryl oxygen atom to give a 2:1 adduct, $[Cp(CO)_2Fe\{P(O)(NEt_2)_2\}]_2FeCl_2$. The molecule as a whole possesses a crystallographic two-fold axis on which the iron atom of the $FeCl_2$ fragment is situated. Thus, for clarity, half of the molecule with atom labels is shown in Fig. 1(b). The final atomic coordinates for non-hydrogen atoms are given in Table 3. Intramolecular bond distances and bond angles, with estimated standard deviations, are listed in Table 4.

The Fe1–P1 bond distance (2.244 Å) falls in the range of normal Fe–P single bond lengths. Although the P1–O3 bond length (1.514 Å) is slightly greater than those already reported [3–9], it can still be regarded as a double bond. The lengthening is probably due to coordination of the phosphoryl oxyten to $FeCl_2$.

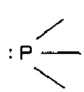
We have compared the geometries around the two amino nitrogen atoms. Newman projections down the N–P bond for III are depicted in Fig. 1(c). The geometry around N1 is planar (C3–N1–C5 178.7°) but that around N2 is between planar and pyramidal (C7–N2–C9 149.4°). Thus, the N1 nitrogen atom is essentially sp^2-hybridized, whereas the N2 atom is an intermediate between sp^2- and sp^3-hybridizations. In fact, the P1–N1 distance (1.649 Å) is shorter by 0.05 Å than the P1–N2 distance (1.696 Å). Thus, significant π-donation from the nitrogen lone-pair to vacant phosphorus orbitals, presumably d-orbitals, is postulated for N1. The π-donation is also possible for N2, but its extent is much less than that for N1.

Table 4

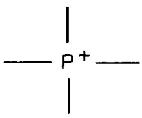
Bond distances (Å) and angles (deg) with estimated standard deviations in parentheses

Bond distances			
Fe1–P1	2.244(4)	Fe1–C1	1.759(15)
Fe1–C2	1.765(16)	Fe1–C11	2.082(8)
Fe1–C12	2.106(14)	Fe1–C13	2.111(19)
Fe1–C14	2.103(21)	Fe1–C15	2.091(14)
Fe2–Cl1	2.282(6)	Fe2–O3	2.003(6)
P1–O3	1.514(9)	P1–N1	1.649(12)
P1–N2	1.696(10)	C1–O1	1.145(19)
C2–O2	1.158(22)	N1–C3	1.485(19)
N1–C5	1.488(18)	N2–C7	1.500(17)
N2–C9	1.440(17)	C3–C4	1.531(25)
C5–C6	1.507(22)	C7–C8	1.525(24)
C9–C10	1.521(21)	C11–C12	1.409(21)
C11–C15	1.388(25)	C12–C13	1.367(23)
C13–C14	1.464(25)	C14–C15	1.375(28)
C3–H(C3)1	1.095(16)	C3–H(C3)2	1.101(16)
C4–H(C4)1	1.094(20)	C4–H(C4)2	1.112(20)
C4–H(C4)3	1.080(20)	C5–H(C5)1	1.097(14)
C5–H(C5)2	1.104(14)	C6–H(C6)1	1.102(17)
C6–H(C6)2	1.085(17)	C6–H(C6)3	1.119(17)
C7–H(C7)1	1.095(14)	C7–H(C7)2	1.017(14)
C8–H(C8)1	1.083(19)	C8–H(C8)2	1.088(19)
C8–H(C8)3	1.132(19)	C9–H(C9)1	1.114(13)
C9–H(C9)2	1.102(13)	C10–H(C10)1	1.084(17)
C10–H(C10)2	1.105(17)	C10–H(C10)3	1.105(17)
C11–H(C11)1	1.096(14)	C12–H(C12)1	1.098(16)
C13–H(C13)1	1.107(16)	C14–H(C14)1	1.100(20)
C15–H(C15)1	1.121(20)		
Bond angles			
P1–Fe1–C1	97.7(5)	P1–Fe1–C2	88.9(5)
C1–Fe1–C2	94.2(7)	Fe1–P1–O3	109.1(4)
Fe1–P1–N1	114.4(4)	Fe1–P1–N2	115.4(4)
O3–P1–N1	110.6(5)	O3–P1–N2	103.7(5)
N1–P1–N2	103.1(5)	P1–N1–C3	126.4(10)
P1–N1–C5	117.7(9)	C3–N1–C5	115.9(11)
P1–N2–C7	119.1(8)	P1–N2–C9	118.6(8)
C7–N2–C9	115.3(10)	Fe1–C1–O1	173.0(14)
Fe1–C2–O2	178.3(15)	N1–C3–C4	113.1(13)
N1–C5–C6	113.9(12)	N2–C7–C8	110.7(12)
N2–C9–C10	115.8(11)	C12–C11–C15	108.1(14)
C11–C12–C13	107.7(14)	C12–C13–C14	108.8(15)
C13–C14–C15	105.1(16)	C11–C15–C14	110.3(17)

It is known that trigonal-planar geometry is present around at least one of the nitrogen atoms directly bonded to trivalent tri-coordinated (a) [10], trivalent tetra-coordinated (b) [11], pentavalent tetra-coordinated (c) [12] phosphorus atoms and trivalent tetra-coordinated phosphorus atoms containing a dative bond with a transition-metal (d) [13]. We have found that one of the two nitrogen atoms bonded to a pentavalent tetra-coordinated phosphorus atom containing a covalent bond with a transition-metal (e) also has trigonal planar geometry. Thus it seems that at



(a)



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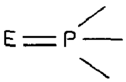
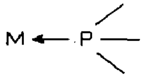
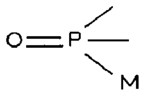


(c)
(E = O, Se, Te)



(d)



(e)

least one nitrogen atom bonded to a phosphorus atom is sp^2-hybridized irrespective of the valency and coordination number of the phosphorus atom, or the existence of a bond between the phosphorus atom and a transition-metal.

The planarity of the nitrogen atom might be partly responsible for the isolation of the cationic complex I and for the resistance to the dealkylation by Cl^- or OMe^- to give $Cp(CO)_2\{P(O)(NEt_2)_2\}$. The electron density of the phosphorus atom in I is increased by π-donation from the planar nitrogen, which decreases the electrophilicity of the methoxy carbon toward Cl^- or OMe^-. The thermolysis is thought to complete the Arbuzov-like dealkylation. In the thermolysis, $FeCl_2$ is also formed, but the mechanism by which it is formed is at present unclear.

The isolation of $Cp(CO)_2Fe\{P(O)(NEt_2)_2\}$ from III has been attempted. A solution of complex III in dichloromethane or acetone was passed through a silica gel column, but the eluted complex was still found to be the 2:1 adduct of $Cp(CO)_2Fe\{P(O)(NEt_2)_2\}$ with $FeCl_2$, indicating that III is stable even in coordinating solvents. Next, III was treated with a more basic ligand, such as pyridine or NEt_3 and $Cp(CO)_2Fe\{P(O)(NEt_2)_2\}$ (IV) was isolated in quantitative yield. The ^1H NMR data can be obtained cleanly and the ^{31}P NMR chemical shift (98.2 ppm) is close to that of $Cp(CO)_2Fe\{P(O)(OEt)_2\}$. (89.9 ppm), indicating that IV is free from the paramagnetic $FeCl_2$ and that the pentavalent phosphorus atom is covalently bonded to the iron atom. Complex IV is, to our knowledge, the first diaminophosphonate transition-metal complex to have been prepared.

Experimental

General remarks

All reactions were carried out under dry nitrogen. Benzene and hexane were distilled from sodium metal, dichloromethane was distilled from P_2O_5, and ethanol was dried over molecular sieve (Type 4A) and then distilled. $Cp(CO)_2FeCl$ was prepared by a published procedure [14].

IR spectra were recorded on a Shimadzu FTIR-4000 spectrometer. NMR spectra were recorded on a JEOL PMX-60 spectrometer using $(CH_3)_4Si$ as an internal standard for ^1H spectra and on a JEOL FX-100 spectrometer using 85% H_3PO_4 as an external standard for ^{31}P spectra.

[Cp(CO)$_2$Fe{P(OMe)(NEt$_2$)$_2$}]Cl (I)

To a solution of Cp(CO)$_2$FeCl (516 mg, 2.43 mmol), in benzene (30 ml), was added P(OMe)(NEt$_2$)$_2$ (500 mg, 0.56 ml, 2.43 mmol). The reaction mixture was stirred for 0.5 h at room temperature to give a yellow powder, which was filtered, washed several times with benzene, and dried in vacuo. Yield 722 mg (76%). Found: C, 45.68; H, 6.67; N, 6.54. $C_{16}H_{28}ClFeN_2O_3P$ calc.: C, 45.90; H, 6.74; N, 6.69%.

Cp(CO)Fe(COOMe){P(OMe)(NEt$_2$)$_2$} (II)

Complex I (346 mg, 0.82mmol) was suspended in 30 ml of benzene and NaOMe (89 mg, 1.65 mmol) was added. After 5 h stirring at room temperature, the mixture was filtered to remove the insoluble material such as NaCl and unchanged NaOMe. The volatile solvent was removed from the filtrate under reduced pressure to give II as a yellowish green oil. Yield 303 mg (82%). The complex is so hygroscopic that the correct elemental analysis data of II could not be obtained, though the satisfactory spectroscopic data were obtained.

[Cp(CO)$_2$Fe{P(O)(NEt$_2$)$_2$}]$_2$FeCl$_2$ (III)

A suspension of complex I (1720 mg, 4.11 mmol) in benzene (60 ml) was refluxed for 3 h to give a reddish brown homogeneous solution. The solvent was removed in vacuo and the residue was washed several times with 5 ml of benzene, then with hexane, followed by drying in vacuo. The product was recrystallized from dichloromethane and hexane. Yield 704 mg (20%). One molecule of III contains three Fe atoms, so the yield is 60% based on amount of Fe atom in the reaction. Found: C, 42.34; H, 5.85; N, 6.44. $C_{30}H_{50}Cl_2Fe_3N_4O_6P_2$ calc.: C, 41.75; H, 5.84, N, 6.49%.

Cp(CO)$_2$Fe{P(O)(NEt$_2$)$_2$} (IV)

To a solution of III (169 mg, 0.39 mmol) in CH_2Cl_2 (10 ml) was added pyridine (0.16 mg, 1.96 mmol), and the solution was stirred for 5 h at room temperature. The solution was loaded onto an alumina column and eluted with EtOH. Removal of the solvent in the yellow eluent gave pure product. Yield 142 mg (99%). Found: C, 48.74; H, 6.81; N, 7.55. $C_{15}H_{25}FeN_2O_3P$ calc.: C, 48.93; H, 6.84; N, 7.61%.

X-ray data collection, structure determination, and refinement

The single crystal of [Cp(CO)$_2$Fe{P(O)(NEt$_2$)$_2$}]$_2$FeCl$_2$ obtained from the dichloromethane/hexane solution was sealed under N_2 in a thin-wall glass capillary. Determination of cell constants and collection of intensity data were carried out on a Syntex R3 diffractometer with Mo-K_α radiation. The space group was uniquely determined as $C2$ from the systematic absences. Unit cell constants were determined by least squares refinement of 25 reflections. Three reference reflections monitored after every cycle of 200 measurements showed no significant variation in intensity during the data collection. Of 3671 unique reflections measured, 1938 reflections with $|F_o| > 3\sigma(|F_o|)$ were used for the structure determination. The data were corrected for Lorentz-polarization factors, but no absorption correction was applied ((Mo-K_α) 12.087 cm^{-1}).

The positions of the Fe atoms were determined by direct methods (MULTAN 78) [15]. All other computations were carried out by use of the Universal Crystallographic Computation Program System, UNICS III [16]. Subsequent difference Fourier maps revealed the positions of the remaining non-hydrogen atoms. After all the non-hydrogen atoms had been refined isotropically, a series of refinements using anisotropic thermal parameters for all non-hydrogen atoms reduced R_1, $\Sigma(|F_o|-|F_c|)/\Sigma|F_o|$, to 0.067, and R_2, $[\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2}$, to 0.072. All hydrogen atoms were placed at idealized positions. In the final stage, non-hydrogen atoms were refined anisotropically including hydrogen atoms whose positions were fixed (B 4 Å^2). At final refinement R_1 converged to 0.059 and R_2 converged to 0.060: Unit weighting was used. All the atomic scattering factors were taken from Cromer and Weber [17]. The anomalous dispersion coefficients of Cromer and Liberman [18] were used for Fe, P, and Cl. All the computations including the ORTEP drawing [19] were carried out by a HITAC M-200 computer at the Hiroshima Univesity Information Processing Center.

Supplementary material

Tables listing the positional and thermal parameters for the hydrogen atoms, and the anisotropic thermal parameters for the non-hydrogen atoms are available from the authors.

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