

## Synthesis and Structure of *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]<sup>+</sup>[BF<sub>4</sub>]<sup>-</sup>

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### [FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]<sup>+</sup>[BF<sub>4</sub>]<sup>-</sup> 착물의 합성 및 구조

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#### Abstract

Dissolution of *trans*-FeHCl(dppe)<sub>2</sub>, **1**, in 4-chlorobutyronitrile (ClCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CN) under argon led to the formation of complex *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]Cl, **2**, which could be further converted to *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>][BF<sub>4</sub>], **3**, on treatment with NaBF<sub>4</sub>. Crystallographic data for **3**: monoclinic space group *P*2<sub>1</sub>/*c*, *a*=13.540(2) Å, *b*=17.058(3) Å, *c*=21.853(4) Å, β=90.15(1)°, *Z*=4, *R*(*wR*<sub>2</sub>)=0.0524(0.1239).

#### 요 약

Ar하에서 *trans*-FeHCl(dppe)<sub>2</sub>, **1**을 4-chlorobutyronitrile에 녹이면, 착물 *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]Cl, **2**가 생성되고, **2**는 NaBF<sub>4</sub>와 반응하여 착물 *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>][BF<sub>4</sub>], **3**로 변환된다. 착물 **3**의 결정학 자료: 단사정계 공간군 *P*2<sub>1</sub>/*c*, *a*=13.540(2) Å, *b*=17.058(3) Å, *c*=21.853(4) Å, β=90.15(1)°, *Z*=4, *R*(*wR*<sub>2</sub>)=0.0524(0.1239).

### 1. Introduction

Nitriles (R-C≡N) are isoelectronic with dinitrogen (N<sub>2</sub>) and their transition metal complexes can serve as convenient precursors for a wide variety of interesting metal complexes. Nitriles have two potential coordination sites, the nitrile nitrogen lone pair (an end-on fashion, **A**) and the carbon-nitrogen triple bond (a side-on fashion, **B**).<sup>1,2</sup> In addition, a combined mode (**C**) of these two fundamental modes has often been observed in several polynuclear complexes as well as in some related complexes possessing X-C≡N ligands in which X is other than an alkyl or aryl substituent.<sup>3</sup>



Nitrile complexes are usually prepared by dissolving a metal halide in the nitrile or replacing CO from carbonyl complexes. They can even be prepared from other nitrile complexes by nitrile exchange reaction.<sup>3</sup> When **1** dissolved in nitriles under argon, an immediate color change from red to orange or yellow was observed. This interesting phenomenon prompted us to investigate, as to what the products are and what their structures are. Herein we report synthesis and structure of an iron (II) nitrile complex of *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]<sup>+</sup>[BF<sub>4</sub>]<sup>-</sup>.

### 2. Experimental Section

Unless otherwise stated, all the reactions have been performed with standard Schlenk line and cannula techniques under an argon atmosphere.

Air-sensitive solids were manipulated in a glove box filled with argon. Glassware was soaked in KOH-saturated 2-propanol for ca. 24 h and washed with distilled water and acetone before use. Glassware was either flame-dried or oven-dried. Hydrocarbon solvents were stirred over concentrated  $H_2SO_4$  for ca. 48 h, neutralized with  $K_2CO_3$ , stirred over sodium metal, and distilled by vacuum transfer. Diethyl ether was stirred over sodium metal and distilled by vacuum transfer. Dichloromethane was stirred over  $CaH_2$  and distilled by vacuum transfer.  $CD_2Cl_2$  was degassed by freeze-pump-thaw cycles before use and stored over molecular sieves under argon.  $FeCl_2 \cdot 2H_2O$ , bis(diphenylphosphino)ethane (dppe), and 4-chlorobutyronitrile ( $ClCH_2CH_2CH_2CN$ ) were purchased from Fluka company. Sodium tetrafluoroborate ( $NaBF_4$ ) was purchased from Aldrich company.  $FeHCl(dppe)_2$ , **1**, was prepared by the literature method.<sup>4)</sup>

$^1H$ - and  $^{13}C\{^1H\}$ -NMR spectra were recorded with a Bruker AMX 500 MHz spectrometer with reference to internal solvent resonances and reported relative to tetramethylsilane.  $^{31}P$ -NMR spectra were also recorded with a Bruker AMX 500 MHz spectrometer with reference to 85%  $H_3PO_4$ . IR spectra were recorded with a Nicolet 205 FTIR spectrophotometer. Melting points were measured with a Thomas Hoover capillary melting point apparatus without calibration.

### 2-1. Preparation of $[FeH(ClCH_2CH_2CH_2CN)(dppe)_2]Cl$ , **2**

10 mL of  $ClCH_2CH_2CH_2CN$  was added to 0.178 g (0.20 mmol) of **1** at room temperature and the solution was stirred for 1 h. The resultant solution was filtered and the solvent was removed under vacuum and washed with diethyl ether ( $2 \times 30$  mL) to give yellow solids of **2** (0.191 g, 0.19 mmol, 95%).  $^1H$ -NMR ( $CDCl_3$ ):  $\delta$  7.421-6.789 (40H, m, phenyl), 2.734 (4H, broad m,  $Ph_2PCH_2CH_2PPh_2$ ), 3.212 (2H, broad, m,  $ClCH_2CH_2CH_2CN$ ), 2.081 (4H, broad m,  $Ph_2PCH_2CH_2PPh_2$ ), 1.667

(2H, broad m,  $ClCH_2CH_2CH_2CN$ ), 1.602 (2H, t,  $J=7$  Hz,  $ClCH_2CH_2CH_2CN$ ), -21.022 (1H, quintet,  $^2J_{P-H}=36.6$  Hz,  $H-Fe$ ).  $^{31}P$ -NMR ( $CD_2Cl_2$ ):  $\delta$  85.317 (d,  $^2J_{P-H}=36.6$  Hz). Mp (decom.): 130-132°C. IR (KBr): 2232 (CN), 1860 (Fe-H)  $cm^{-1}$ .

### 2-2. Preparation of $[FeH(ClCH_2CH_2CH_2CN)(dppe)_2][BF_4]$ , **3**

$NaBF_4$  (0.022 g, 0.2 mmol) was added to 10 mL of  $ClCH_2CH_2CH_2CN$  containing **2** (0.095 g, 0.1 mmol) and then the resulting solution was stirred for 2 h. The solvent was removed under vacuum to give yellow solids, which was extracted with 15 mL of  $CH_2Cl_2$ . The solvent was removed again and the resulting yellow solids were recrystallized from  $CH_2Cl_2$ -hexanes to give yellow crystals of **3** (0.037 g, 0.034 mmol, 67.4%).  $^1H$ -NMR ( $CD_2Cl_2$ ):  $\delta$  7.458-7.140 (40H, m, phenyl,  $Ph_2PCH_2CH_2PPh_2$ ), 3.272 (2H, t,  $J=6$  Hz,  $ClCH_2CH_2CH_2CN$ ), 2.599 (4H, broad m,  $Ph_2PCH_2CH_2PPh_2$ ), 2.258 (2H, t,  $J=7$  Hz,  $ClCH_2CH_2CH_2CN$ ), 2.059 (4H, broad m,  $Ph_2PCH_2CH_2PPh_2$ ), 1.526 (2H, broad m,  $ClCH_2CH_2CH_2CN$ ), -20.599 (1H, quintet,  $^2J_{P-H}=36.6$  Hz,  $H-Fe$ ).  $^{13}C\{^1H\}$ -NMR ( $CD_2Cl_2$ ):  $\delta$  136.062, 135.005, 133.714, 133.066, 130.586, 129.208, 128.445 (phenyl), 43.824 ( $ClCH_2CH_2CH_2CN$ ), 33.269 ( $Ph_2PCH_2CH_2PPh_2$ ), 28.508 ( $ClCH_2CH_2CH_2CN$ ), 18.432 ( $ClCH_2CH_2CH_2CN$ ).  $^{31}P$ -NMR ( $CD_2Cl_2$ ):  $\delta$  88.654 (d,  $^2J_{P-H}=36.6$  Hz). Mp (decom.): 172-174°C. IR (KBr): 2232 (C $\equiv$ N).

### 2-3. X-ray Structure Determination of **3**

All X-ray data were collected with use of a Siemens P4 diffractometer equipped with an Mo X-ray tube and a graphite crystal monochromator. Details on crystal data and intensity data are given in Table 1. The orientation matrix and unit cell parameters were determined by least-squares analyses of the setting angles of 37 reflections in the range  $15.0^\circ < 2\theta < 25.0^\circ$ . Three check reflections were measured every 100 reflections throughout data collection and showed no significant variations in intensity.

**Table 1. X-ray data collection and structure refinement for 3**

formula	C <sub>66</sub> H <sub>95</sub> BNF <sub>4</sub> P <sub>4</sub> ClFe	$T_{\max}$	0.297
formula weight	1044.00	$T_{\min}$	0.208
temperature, K	295	$F(000)$	2,168
crystal system	monoclinic	$2\theta$ range (°)	3.5-50
space group	$P2_1/c$	scan type	$\omega$
$a$ , Å	13.540(2)	scan speed	variable
$b$ , Å	17.058(3)	No. of reflns measured	10,693
$c$ , Å	21.853(4)	No. of reflns with $I > 2\sigma(I)$	8,781
$\beta$ , deg	90.15(1)	No. of params refined	646
$V$ , Å <sup>3</sup>	5,047(1)	Max., in $\Delta\rho$ (eÅ <sup>-3</sup> )	0.763
$Z$	4	Min., in $\Delta\rho$ (eÅ <sup>-3</sup> )	-0.519
$d_{\text{calc}}$ , g cm <sup>-3</sup>	1.374	$GOF$ on $F^2$	1.058
$\mu$ , mm <sup>-1</sup>	0.532	$R$	0.0524
		$wR_g^a$	0.1239

$$^a wR_g = ([w(F_o^2 - F_c^2)^2] / [w(F_o^2)^2])^{1/2}$$

Intensity data were corrected for Lorenz and polarization effects. Decay corrections were also made. The intensity data were empirically corrected with  $\psi$ -scan data. All calculations were carried out with use of the SHELXTL programs.<sup>5)</sup>

A yellow crystal of **3**, irregularly shaped, of approximate dimensions 0.24 × 0.32 × 0.68 mm<sup>3</sup>, was used for crystal and intensity data collection. The unit cell parameters and systematic absences,  $h0l$  ( $l=2n+1$ ) and  $0k0$  ( $k=2n+1$ ), unambiguously indicated  $P2_1/c$  as a space group. The structure was solved by the heavy atom method and refined by full-matrix least-squares calculations of  $F^2$  initially with isotropic and finally anisotropic temperature factors for all non-hydrogen atoms. The Cl atom and two F atoms (F3 and F4) exhibited a structural disorder. The best fit for the disordered Cl atom was obtained by considering the Cl atom to be distributed over two positions with the site occupation factors of 0.72:0.28. The best fit for the disordered F atoms was obtained by considering the two F atoms to be distributed over two positions with the site occupation factors of 0.58:0.42 (F3-F4:F3A-F4A). The hydride ligand (H1) was located in the difference Fourier map and refined isotropically. All the other hydrogen atoms were generated in idealized positions and refined using a riding model.

**Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{Å}^3 \times 10^3$ ) for 3**

	$x$	$y$	$z$	$U(\text{eq})^a$
Fe	3167(1)	2806(1)	1702(1)	29(1)
P(1)	4299(1)	3772(1)	1649(1)	33(1)
P(2)	2165(1)	3707(1)	1294(1)	34(1)
P(3)	1981(1)	1889(1)	1846(1)	32(1)
P(4)	4136(1)	1926(1)	2197(1)	34(1)
N(1)	3610(2)	2353(2)	943(1)	36(1)
C(1)	5026(3)	4060(2)	2321(2)	39(1)
C(2)	4528(3)	4111(2)	2873(2)	51(1)
C(3)	4980(4)	4424(3)	3392(2)	65(1)
C(4)	5934(4)	4687(3)	3357(2)	72(1)
C(5)	6438(3)	4648(3)	2817(2)	68(1)
C(6)	5987(3)	4345(3)	2295(2)	53(1)
C(7)	5188(2)	3697(2)	1015(2)	38(1)
C(8)	6074(3)	3294(2)	1066(2)	49(1)
C(9)	6665(3)	3167(3)	561(2)	61(1)
C(10)	6391(4)	3445(3)	-4(2)	67(1)
C(11)	5525(3)	3833(3)	-67(2)	62(1)
C(12)	4913(3)	3959(2)	431(2)	50(1)
C(13)	3695(3)	4735(2)	1503(2)	43(1)
C(14)	2591(3)	4667(2)	1571(2)	42(1)
C(15)	2165(3)	3804(2)	457(2)	41(1)
C(16)	2378(3)	4504(3)	156(2)	57(1)
C(17)	2438(4)	4524(3)	-482(2)	71(1)
C(18)	2247(4)	3871(4)	-812(2)	75(2)
C(19)	1998(3)	3178(3)	-530(2)	65(1)
C(20)	1971(3)	3143(3)	102(2)	51(1)
C(21)	853(3)	3806(2)	1474(2)	44(1)

Table 2. Continued

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq) <sup>a</sup>
C(22)	112(3)	3840(3)	1029(2)	58(1)
C(23)	-858(3)	3921(3)	1182(3)	82(2)
C(24)	-1128(4)	3976(4)	1782(4)	100(2)
C(25)	-418(4)	3959(4)	2227(3)	93(2)
C(26)	574(3)	3875(3)	2073(2)	60(1)
C(27)	1225(3)	1614(2)	1183(2)	37(1)
C(28)	1586(3)	1093(2)	753(2)	50(1)
C(29)	1022(4)	897(3)	241(2)	71(1)
C(30)	106(4)	1229(3)	160(2)	76(2)
C(31)	-261(3)	1744(3)	579(2)	63(1)
C(32)	292(3)	1937(2)	1093(2)	46(1)
C(33)	1052(2)	1929(2)	2456(2)	36(1)
C(34)	259(3)	1411(2)	2459(2)	44(1)
C(35)	-405(3)	1409(3)	2941(2)	52(1)
C(36)	-265(3)	1898(3)	3425(2)	61(1)
C(37)	518(3)	2399(3)	3444(2)	63(1)
C(38)	1176(3)	2420(2)	2956(2)	48(1)
C(39)	2581(3)	946(2)	2067(2)	41(1)
C(40)	3676(3)	964(2)	1937(2)	41(1)
C(41)	4111(2)	1792(2)	3031(2)	39(1)
C(42)	3649(3)	2325(2)	3413(2)	46(1)
C(43)	3654(3)	2213(3)	4045(2)	61(1)
C(44)	4112(4)	1576(3)	4294(2)	67(1)
C(45)	4585(3)	1046(3)	3926(2)	61(1)
C(46)	4585(3)	1152(2)	3296(2)	51(1)
C(47)	5470(3)	1846(2)	2061(2)	44(1)
C(48)	6135(3)	2200(3)	2465(2)	55(1)
C(49)	7145(3)	2148(3)	2359(2)	71(1)
C(50)	7499(3)	1745(3)	1855(3)	75(2)
C(51)	6853(3)	1400(3)	1462(2)	71(1)
C(52)	5843(3)	1445(3)	1563(2)	55(1)
C(53)	3938(3)	2138(2)	493(2)	43(1)
C(54)	4323(4)	1866(3)	-90(2)	68(1)
C(55)	4208(5)	1016(3)	-190(3)	96(2)
C(56)	4253(7)	738(5)	-830(4)	157(4)
Cl(1)	3178(13)	1456(22)	-1325(9)	93(6)
Cl(1A)	3247(5)	1048(20)	-1238(4)	128(5)
B(1)	2894(5)	9098(4)	645(5)	95(2)
F(1)	3325(3)	8431(2)	502(2)	127(1)
F(2)	3465(3)	9712(3)	746(3)	154(2)
F(3)	2828(16)	8981(7)	1326(6)	196(7)
F(3A)	2041(10)	9089(9)	878(10)	137(10)
F(4)	2090(10)	9214(9)	401(12)	234(13)
F(4A)	2769(20)	9452(9)	47(7)	193(9)
H(1)	2841(28)	3210(23)	2278(18)	59(11)

Equivalent isotropic *U*(eq) defined as one third of the trace of the orthogonalized *U<sub>i</sub>* tensor.

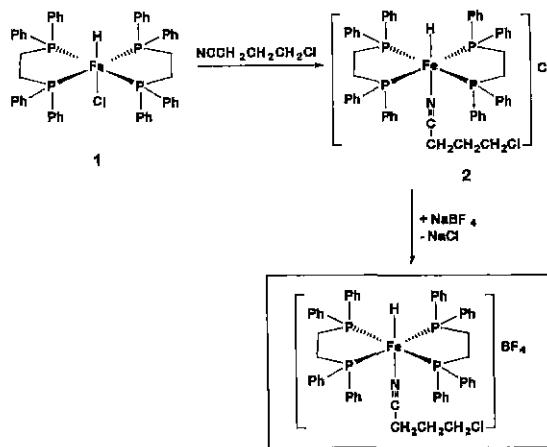
Final atomic positional parameters for non-hydrogen atoms are shown in Table 2. The selected bond distances and bond angles are shown in Table 3.

### 3. Results and discussion

#### 3-1. Preparation

Dissolution of *trans*-FeHCl(dppe)<sub>2</sub>, **1**, in 4-chlorobutyronitrile (ClCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CN) under argon leads to the formation of *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>]Cl, **2**, which can be further converted to *trans*-[FeH(NCCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl)(dppe)<sub>2</sub>][BF<sub>4</sub>], **3**, on treatment with NaBF<sub>4</sub> (Scheme 1). In these reactions the nitrile behaves as a reagent as well as a solvent. Complexes of **3** can also be prepared directly from **1** with NaBF<sub>4</sub> in the 4-chlorobutyronitrile. In all of the reactions, there is a color change from red to yellow.

All of the complexes exhibit a quintet ( $\delta$  -21.022 for **2** and -20.599 for **3**) for the hydride ligand in their <sup>1</sup>H-NMR spectra and a doublet ( $\delta$  85.317 for **2** and 88.654 for **3**) for the four phosphine ligands in their <sup>31</sup>P-NMR spectra. These observations indicate that the hydride ligand couples with the four equivalent phosphorus nuclei in the phosphines occupying the equatorial plane and that the *trans* orientation between the hy-



3

Scheme 1.

dride and the nitrile remains intact. The *trans* orientation has been confirmed further by the crystal structure determination of **3**.

### 3-2. Structure

The structure of the cationic part of **3**, with the atomic numbering scheme is shown in Fig. 1. The coordination sphere of the Fe metal can be described as an octahedron, with two bidentate dppe ligands at the equatorial sites and the hydride and nitrile ligands at the axial sites. The anion [BF<sub>4</sub>]<sup>-</sup> is not bonded to the Fe metal and acts as a counter-anion to retain electroneutrality (Fig. 2). The bond distances and bond angles around the B1 indicate that the boron atom in [BPh<sub>4</sub>]<sup>-</sup> is *sp*<sup>3</sup>-hybridized (Table 3). The two F atoms (F3 and F4) in [BPh<sub>4</sub>]<sup>-</sup> exhibit a structural disorder, which is well known for the highly symmetric counter anions such as Et<sub>4</sub>N<sup>+</sup>, ClO<sub>4</sub><sup>-</sup>, BF<sub>4</sub><sup>-</sup>, and PF<sub>6</sub><sup>-</sup>.

The equatorial plane, defined by the four dppe phosphorus atoms (P1-P4), is essentially planar with the average atomic displacement of 0.008 Å. The Fe metal lies 0.105 Å below the equatorial plane. The -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl moiety of the nitrile is directed toward the open space between the two phenyl rings (C7-C12 and C15-

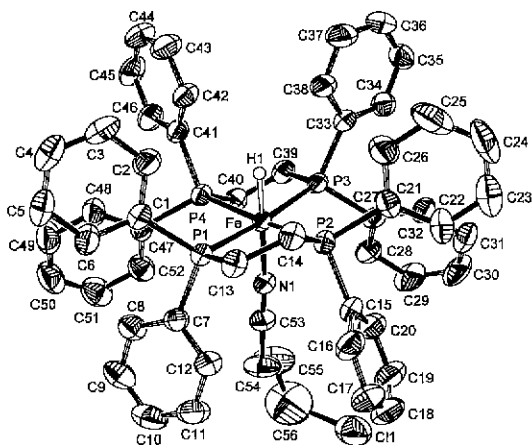


Fig. 1. ORTEP drawing<sup>9</sup> of the cationic part of **3**, showing the atom-labeling scheme and 50% probability thermal ellipsoids.

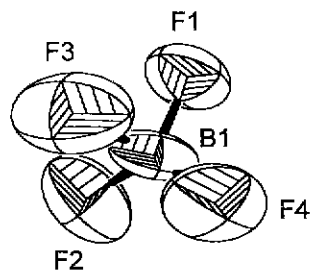


Fig. 2. ORTEP drawing<sup>9</sup> of the anionic part (BF<sub>4</sub>)<sup>-</sup> of **3**, showing the atom-labeling scheme and 50% probability thermal ellipsoids.

C20), probably due to steric hindrance.

Although the hydride ligands (H1) could be located and reasonably refined with an isotropic thermal parameter, the relatively short distance of Fe-H(1) (1.50(4) Å) does not seem to be very reliable. In the closely related complex, *trans*-[FeH( $\eta^2$ -H<sub>2</sub>)(dppe)<sub>2</sub>]<sup>+</sup>[BF<sub>4</sub>]<sup>-</sup>, which was prepared by protonation of FeH<sub>2</sub>(dppe)<sub>2</sub> with HBF<sub>4</sub>·Et<sub>2</sub>O and structurally characterized by X-ray diffraction, the bond distance of Fe-H (terminal) is 1.28(8) Å.<sup>6</sup> In another related complex, *trans*-[FeH( $\eta^2$ -H<sub>2</sub>)(dppe)<sub>2</sub>]<sup>+</sup>[BPh<sub>4</sub>]<sup>-</sup>, which was prepared by counterion exchange of *trans*-[FeH( $\eta^2$ -H<sub>2</sub>)(dppe)<sub>2</sub>]<sup>+</sup>[BF<sub>4</sub>]<sup>-</sup> with NaBPh<sub>4</sub> and structurally characterized both by X-ray diffraction and by neutron diffraction, the bond distances of Fe-H (terminal) are 1.30(3) Å (X-ray diffraction) and 1.535(12) Å (neutron diffraction), respectively.<sup>7</sup>

The Fe-N1 bond distance (1.928(3)) Å indicates an Fe-N single bond, because a metal-nitrogen single bond is expected to lie within 1.95-2.15 Å.<sup>8</sup>

The C53-N1-Fe bond angle of 173.6(3)<sup>o</sup> is fairly typical of nitrile ligands (168-178<sup>o</sup>) and indicates an end-on coordination of this ligand.<sup>2</sup> The bond distance of N1-C53 (1.134(3)) parallels the known fact that the N≡C bond distances usually shorten on coordination. The N≡C bond distances between 1.11 and 1.15 Å are most frequently found, compared to 1.16 Å in the free CH<sub>3</sub>CN.<sup>3</sup>

Compounds **3** has a pseudo 4-fold rotation axis that passes through the Fe, N1, and H1 atoms and is perpendicular to the equatorial

**Table 3. Selected bond distances (Å) and bond angles (°) in 3**

Fe-N(1)	1.928(3)	Fe-P(2)	2.234(1)	Fe-P(1)	2.253(1)
Fe-P(3)	2.265(1)	Fe-P(4)	2.266(1)	Fe-H(1)	1.50(4)
P(1)-C(1)	1.834(3)	P(1)-C(7)	1.842(3)	P(1)-C(13)	1.862(4)
P(2)-C(21)	1.829(4)	P(2)-C(15)	1.836(4)	P(2)-C(14)	1.838(4)
P(3)-C(27)	1.833(3)	P(3)-C(33)	1.838(3)	P(3)-C(39)	1.864(4)
P(4)-C(47)	1.836(4)	P(4)-C(41)	1.837(4)	P(4)-C(40)	1.845(4)
C(39)-C(40)	1.511(5)	C(49)-C(50)	1.386(7)	N(1)-C(53)	1.139(4)
C(13)-C(14)	1.506(5)	C(50)-C(51)	1.358(7)	C(51)-C(52)	1.389(6)
C(53)-C(54)	1.454(5)	C(54)-C(55)	1.474(7)	C(55)-C(56)	1.479(9)
C(56)-Cl	1.71(2)	C(56)-Cl(1)	2.18(4)	B(1)-F(3A)	1.264(12)
B(1)-F(1)	1.317(8)	B(1)-F(2)	1.320(8)	B(1)-F(3)	1.503(14)
F(3)-F(3A)	1.46(2)	B(1)-F(4)	1.229(12)	B(1)-F(4A)	1.45(2)
N1-Fe-P2	97.05(9)	N1-Fe-P1	92.03(9)	P2-Fe-P1	83.62(4)
N1-Fe-P3	93.69(9)	P2-Fe-P3	95.80(4)	P1-Fe-P3	174.28(4)
N1-Fe-P4	87.90(9)	P2-Fe-P4	175.04(4)	P1-Fe-P4	96.65(4)
P3-Fe-P4	83.43(4)	N1-Fe-H1	176.4(15)	P1-Fe-H1	84.8(15)
P3-Fe-H1	89.5(15)	C1-P1-C7	105.7(2)	C1-P1-C13	97.7(2)
C7-P1-C13	102.8(2)	C21-P2-C15	102.0(2)	C21-P2-C14	98.7(2)
C15-P2-C14	104.3(2)	C27-P3-C33	101.6(2)	C27-P3-C39	103.1(2)
C33-P3-C39	98.2(2)	C53-N1-Fe	173.6(3)	C50-C51-C52	120.5(5)
C55-C56-Cl1	105.0(7)	N1-C53-C54	178.1(4)	C53-C54-C55	114.0(4)
C54-C55-C56	116.8(6)	C55-C56-Cl	111.1(7)	F3A-F4-B1	66.4(11)
F4-B1-F1	115.4(11)	F3A-B1-F1	119.4(9)	F4-B1-F2	117.6(10)
F3A-B1-F2	118.5(10)	F1-B1-F2	117.8(6)	F3A-B1-F	105.2(13)
F1-B1-F4A	101.3(10)	F2-B1-F4A	83.6(9)	F4-B1-F3	113.4(14)
F1-B1-F3	98.6(7)	F2-B1-F3	88.6(9)	F3A-F3-B1	50.5(6)
F4-F3A-F3	129.4(14)				

plane, suggesting that the four phosphorus nuclei are equivalent. This crystal structure explains the NMR spectra that show a quintet for the H ligand in its  $^1\text{H-NMR}$  and a doublet for the four P atoms in its  $^{31}\text{P-NMR}$ . The results of the X-ray crystal structure and NMR spectral data indicate that **3** has the same structure both in solution and in the solid state.

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