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Acta Cryst. (1993). C**49**, 1056–1059

Structure of $[FeH(N_2)\{(H_5C_2)_2PCH_2CH_2P(C_2H_5)_2\}_2]BPh_4$

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(*Received* 9 *September* 1992; *accepted* 25 *January* 1993)

Abstract. Bis[1,2-bis(diethylphosphino)ethane-*P*,*P*′]hydrido(dinitrogen-*N*)iron(II) tetraphenylborate, $[FeH(N_2)(C_{10}H_{24}P_2)_2](C_{24}H_{20}B)$, $M_r = 816.6$, monoclinic, $P2_1/n$, $a = 16.493$ (3), $b = 13.691$ (4), $c = 21.422$ (8) Å, $\beta = 111.20$ (2)°, $V = 4509.8$ Å^3, $Z = 4$, $D_x = 1.203$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.503$ mm^{-1}, $F(000) = 1752$, $T = 293$ K, $R = 0.0651$ for 5651 independent observed reflections. The title compound was prepared in 86% yield from $[FeHCl\{(H_5C_2)_2PCH_2CH_2P(C_2H_5)_2\}_2]$ and $NaBPh_4$ in methanol under an atmosphere of nitrogen. The molecule consists of hydride *trans* to 'end-bound' molecular nitrogen and two bisphosphine ligands in a pseudo-octahedral arrangement about the iron centre.

Introduction. Complexes with molecular nitrogen bound to transition metals are important models for the active sites in nitrogenase enzymes (Chatt, Dilworth & Richards, 1978; Eady, 1991; Leigh, Prieto-Alcon & Sanders, 1991). Since this enzyme is known to feature an active site which incorporates an iron centre, complexes of iron bound to molecular nitrogen are of particular interest. Although several iron hydrido–dinitrogen species are known, only $[FeH(N_2)(P_4)]^+$ (P_4 = hexaphenyl-1,4,7,10-tetraphosphadecane) and $[FeH_2(N_2)(PEtPh_2)_3]$ (Ghilardi, Midollini, Sacconi & Stoppioni, 1981; Van der Sluys *et al.*, 1990) have been fully structurally characterized, while recently a brief communication on the structure of $[FeH(N_2)(dmpe)_2]^+$ [dmpe = 1,2-bis(dimethylphosphino)ethane] was published (Hills, Hughes, Jimenez-Tenorio & Leigh, 1990). An analogue of the latter complex, $[FeH(N_2)(depe)_2]BPh_4$ [depe = 1,2-bis(diethylphosphino)ethane], was one of the earliest literature examples of a molecular nitrogen adduct (Bancroft, Mays & Prater, 1969). We have been studying the reactions of $[FeH(N_2)(depe)_2]^+$ as part of our continuing investigations on transition-metal complexes involving bisphosphine ligands. During this research we obtained crystals of suitable quality for diffraction studies and we now report in detail the structure of the title complex.

Experimental. $[FeH(N_2)(depe)_2]BPh_4$ was prepared by a modified literature method (Bancroft *et al.*, 1969). Under a nitrogen atmosphere, 0.18 g (0.29 mmol) $[FeHCl(depe)_2]$ was dissolved in 10 cm^3 of methanol. Addition of 0.98 g (0.29 mmol) $NaBPh_4$ gave $[FeH(N_2)(depe)_2]BPh_4$ in 86% yield as a buff-

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coloured precipitate isolated by filtration and dried *in vacuo*. Suitable crystals for X-ray diffraction studies were obtained from acetone–benzene solution by slow evaporation of solvent. Multinuclear NMR studies on $[FeH(N_2)(depe)_2]BPh_4$ confirm the *trans* nature of the product in solution. ^{31}P-{^1H} NMR [in $^2H_6$-acetone, externally referenced to a capillary containing neat $P(OMe)_3$ at δ 140.8]: δ 81.6 (*s*). ^1H-{^{31}P} NMR (in $^2H_6$-acetone, referenced to residual acetone at δ 2.20): δ −17.99 (*s*, Fe—*H*, in the ^1H NMR spectrum *qu* with J_{PH} = 49.0 Hz), 1.24 (*t*, J_{HH} = 7.5 Hz, 4 × C*H*$_3$), 1.43 (*t*, J_{HH} = 7.6 Hz, 4 × C*H*$_3$), 1.73 (*d* of *q*, J_{HH} = 15.0 and 7.6 Hz, 4 × C*H*$_2$CH$_3$), 2.00 (*m*, 4 × C*H*$_2$), 2.21 (*d* of *q*, J_{HH} = 14.9 and 7.5 Hz, 4 × C*H*$_2$CH$_3$), 6.90–6.98 (*m*, 4 × Ph-*H*), 7.05–7.13 (*m*, 8 × Ph-*H*), 7.47–7.56 (*m*, 8 × Ph-*H*). ^{13}C-{^1H,^{31}P} NMR (in $^2H_8$-THF referenced to the high-field THF resonance at δ 26.7): δ 9.9 (*C*H$_3$), 10.0 (*C*H$_3$), 22.9 (*C*H$_2$), 24.3 (*C*H$_2$), 24.4 (*C*H$_2$), 123.0 (Ph-*C*H), 126.9 (Ph-*C*H), 138.4 (Ph-*C*H), 166.4 (Ph-*C*) (*q*, J_{CB} = 50 Hz).

Crystal data are listed in Table 1 and fractional coordinates for non-H atoms are listed in Table 2.* The crystal was coated in araldite and mounted on a glass fibre. Lattice parameters at 293 K were determined by least-squares fit to the setting parameters of 25 independent reflections (θ 10–15°). Data reduction and application of Lorentz–polarization and absorption (maximum transmission 0.9922, minimum transmission 0.9093) corrections were carried out using the Enraf–Nonius *SDP* system (Frenz, 1985). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and the solutions extended by difference Fourier methods. All calculations were carried out using *SHELX*76 (Sheldrick, 1976). All H atoms were included at calculated sites (apart from the hydride) with group isotropic thermal parameters, and all other atoms were refined anisotropically. Scattering factors and anomalous-dispersion terms for Fe were taken from Cromer & Waber (1974) and all others used were supplied with *SHELX*76. The electron density map shows some disorder about the C atom C(9) attached to P(2), presumably resulting from thermal motion. No attempts were made to model this disorder and therefore some anomalies in bond lengths and angles are associated with this region of the molecule.

Discussion. Fig. 1 shows the cation geometry and atom-labelling scheme and Table 3 lists selected

Table 1. *Crystal data, data collection and refinement parameters for* $[FeH(N_2)(depe)_2]BPh_4$

Crystal habit	Prism
Crystal colour	Orange-brown
Crystal size (mm)	0.2 × 0.1 × 0.01
Diffractometer	Enraf–Nonius CAD-4, four circle
Monochromator	Graphite
Scan mode	ω
θ range (°)	1.0–25.0
Number of reflections measured	8528
Number of independent reflections	7917
Number of observed reflections	5651
Criterion of observation	$I > 2.5\sigma(I)$
R_{int}	0.0307
Number of standard reflections	3
Frequency of standard reflections (min)	60
Variation of standard reflections	Negligible
Range of h, k, l	−19 to 19, 0 to 16, 0 to 25
Number of variables	498
$R(F_o)$	0.0651
wR	0.0828
Weighting scheme	$w = [\sigma^2 + (0.009778F_o)^2]^{-1}$
Shift/e.s.d.	< 1.212
$(\Delta\rho)_{min}$ (e Å^{-3})	−0.8853
$(\Delta\rho)_{max}$ (e Å^{-3})	1.0659

Table 2. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Fe	3789 (1)	7219 (1)	1063 (1)	3.02
H′	336 (3)	728 (4)	149 (3)	–
P(1)	2587 (1)	7764 (1)	269 (1)	3.36
P(2)	4133 (1)	8796 (1)	1288 (1)	5.49
P(3)	3312 (1)	5666 (1)	968 (1)	3.41
P(4)	4785 (1)	6690 (1)	2029 (1)	3.97
N(1)	4426 (3)	7027 (4)	533 (3)	4.50
N(2)	4785 (5)	6911 (7)	211 (4)	7.55
C(1)	734 (5)	7942 (6)	−158 (4)	5.63
C(2)	1529 (4)	7349 (5)	272 (3)	4.19
C(3)	2942 (7)	8252 (8)	−913 (4)	7.71
C(4)	2457 (5)	7591 (6)	−620 (3)	5.04
C(5)	2510 (4)	9104 (5)	343 (3)	4.64
C(6)	3419 (5)	9534 (5)	586 (4)	4.86
C(7)	3873 (6)	10338 (7)	2160 (5)	7.69
C(8)	3838 (6)	9326 (7)	2049 (4)	7.07
C(9)	5450 (7)	9775 (10)	1036 (5)	18.78
C(10)	5212 (5)	9247 (7)	1421 (5)	11.39
C(11)	6375 (8)	6576 (10)	1838 (7)	11.97
C(12)	5955 (6)	6894 (10)	2247 (7)	9.67
C(13)	3898 (7)	7093 (7)	2938 (4)	7.10
C(14)	4774 (6)	7118 (6)	2841 (4)	6.65
C(15)	4715 (4)	5358 (5)	2111 (3)	4.38
C(16)	4249 (4)	4900 (5)	1432 (3)	4.39
C(17)	2210 (7)	4389 (6)	1388 (5)	7.91
C(18)	2525 (5)	5415 (5)	1377 (4)	4.88
C(19)	3451 (6)	4948 (5)	−240 (4)	6.00
C(20)	2849 (3)	5007 (4)	166 (3)	4.52
B	1126 (4)	2286 (4)	8812 (3)	4.04
C(21)	1903 (3)	2160 (3)	9536 (3)	3.84
C(22)	2785 (3)	2342 (4)	9636 (3)	4.38
C(23)	3439 (3)	2364 (4)	10267 (3)	5.41
C(24)	3239 (4)	2194 (4)	10833 (3)	4.90
C(25)	2414 (4)	1998 (4)	10762 (3)	5.17
C(26)	1757 (3)	1981 (4)	10128 (3)	4.34
C(27)	252 (3)	1661 (3)	8721 (3)	4.08
C(28)	273 (3)	734 (3)	8995 (3)	4.50
C(29)	−458 (4)	175 (4)	8879 (3)	5.01
C(30)	−1250 (4)	519 (4)	8476 (3)	5.23
C(31)	−1313 (3)	1412 (5)	8192 (3)	5.60
C(32)	−573 (3)	1970 (4)	8314 (3)	4.87
C(33)	1401 (3)	1915 (4)	8183 (3)	4.66
C(34)	2017 (4)	1191 (4)	8249 (3)	5.66
C(35)	2178 (4)	838 (6)	7678 (4)	7.16
C(36)	1739 (5)	1183 (6)	7060 (4)	7.21
C(37)	1102 (5)	1868 (6)	6979 (3)	7.06
C(38)	950 (4)	2219 (5)	7529 (3)	5.43
C(39)	933 (3)	3483 (3)	8770 (2)	4.00
C(40)	1297 (3)	4157 (4)	8461 (2)	4.68
C(41)	1167 (4)	5160 (4)	8482 (3)	5.49
C(42)	668 (4)	5511 (4)	8814 (3)	6.19
C(43)	289 (4)	4888 (4)	9132 (3)	5.46
C(44)	427 (4)	3883 (4)	9104 (3)	5.20

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55882 (45 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1022]

bond lengths and angles. The Fe centre in $[FeH(N_2)(depe)_2]^+$ is six-coordinate with a pseudo-octahedral arrangement of ligands and hydride *trans* to molecular nitrogen. The molecular geometry in the plane of the P atoms is distorted from octahedral, presumably as a result of the constraints imposed by ligand bite angles of 85.8 (1) and 84.0 (1)° which result in unique *trans* angles of 169.5 (1) and 165.1 (1)° for P(2)—Fe—P(3) and P(1)—Fe—P(4), respectively. The Fe centre lies 0.247 Å out of the idealized plane defined by the four P atoms. The Fe—H, Fe—N and N—N bonds of 1.351 (66), 1.825 (7) and 1.070 (12) Å, respectively, in $[FeH(N_2)(depe)_2]^+$ are comparable in length with those reported for $[FeH(N_2)(dmpe)_2]^+$ [1.32 (2), 1.818 (11) and 1.13 (3) Å, respectively (Hills *et al.*, 1990)]. However, the average Fe—P bond length for the depe analogue (2.240 Å) is significantly longer than that of the dmpe analogue (2.205 Å) and probably reflects an increase in steric repulsions when methyl substituents are replaced by ethyl on the bisphosphine ligand. An increase in Fe—P bond length has also been noted for replacement of methyl by ethyl substituents in the complexes $[FeCl_2(P\text{–}P)_2]$ (P–P = dmpe, depe) and was assigned to steric repulsion (Barclay, Hills, Hughes & Leigh, 1988). The nitrogen molecule is linear [Fe—N(1)—N(2) 178.5 (5)°] and 'end-bound', the most common binding mode observed for dinitrogen complexes of transition metals (Leigh, 1992). The N—N bond length of 1.070 (12) Å is shorter than that of 1.098 Å reported for molecular nitrogen (Wilkinson & Houk, 1956) but does not differ significantly from the N—N distances in $[FeH(N_2)(dmpe)_2]^+$ and $[FeH(N_2)(P_4)]^+$ [1.13 (3) and 1.076 (15) Å, respectively]. The dimensions of the Fe—N and N—N bond lengths in $[FeH(N_2)(depe)_2]^+$ suggest that displacement of the molecular nitrogen by other ligands should be possible. The original literature report (Bancroft *et al.*, 1969) does indeed state that the complex is extremely reactive towards substitution of dinitrogen with CO and other π-bonding ligands. However, this contrasts with the reaction between $[FeH(N_2)(dmpe)_2]^+$ and CO or CNMe which has been found (Hills *et al.*, 1990) to proceed only slowly. We are continuing our studies on the substitution reactions of these complexes in order to quantify these observations.

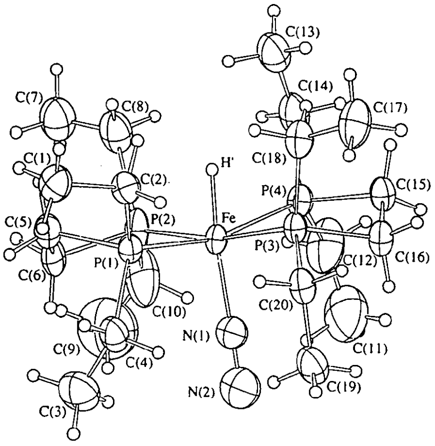

Fig. 1. An *ORTEP* diagram (Johnson, 1965) showing the molecular structure and the atom-numbering scheme of $[FeH(N_2)(depe)_2]^+$ (H-atom labels have been omitted for clarity).

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

Fe—P(1)	2.224 (1)	Fe—P(3)	2.250 (2)
Fe—P(2)	2.240 (2)	Fe—P(4)	2.246 (2)
Fe—N(1)	1.825 (7)	Fe—H′	1.351 (66)
N(1)—N(2)	1.070 (12)	P(1)—C(4)	1.853 (7)
P(1)—C(2)	1.838 (7)	P(2)—C(6)	1.841 (7)
P(1)—C(5)	1.849 (7)	P(2)—C(10)	1.806 (9)
P(2)—C(8)	2.000 (10)	P(3)—C(18)	1.841 (9)
P(3)—C(16)	1.834 (6)	P(4)—C(12)	1.834 (10)
P(3)—C(20)	1.845 (5)	P(4)—C(15)	1.840 (7)
P(4)—C(14)	1.842 (9)	C(3)—C(4)	1.488 (14)
C(1)—C(2)	1.535 (9)	C(7)—C(8)	1.404 (14)
C(5)—C(6)	1.516 (10)	C(11)—C(12)	1.370 (23)
C(9)—C(10)	1.261 (17)	C(15)—C(16)	1.514 (9)
C(13)—C(14)	1.533 (17)	C(19)—C(20)	1.542 (12)
C(17)—C(18)	1.501 (11)		
P(1)—Fe—P(2)	85.8 (1)	P(1)—Fe—P(3)	93.6 (1)
P(1)—Fe—P(4)	165.1 (1)	P(2)—Fe—P(3)	169.5 (1)
P(2)—Fe—P(4)	93.9 (1)	P(3)—Fe—P(4)	84.0 (1)
N(1)—Fe—P(1)	97.2 (2)	N(1)—Fe—P(2)	96.5 (2)
N(1)—Fe—P(3)	94.0 (2)	N(1)—Fe—P(4)	97.6 (2)
H′—Fe—P(1)	87.9 (2)	H′—Fe—P(2)	87.2 (2)
H′—Fe—P(3)	82.3 (2)	H′—Fe—P(4)	77.2 (2)
H′—Fe—N(1)	173.8 (2)	N(2)—N(1)—Fe	178.5 (5)
C(2)—P(1)—Fe	118.5 (2)	C(4)—P(1)—Fe	118.9 (3)
C(5)—P(1)—Fe	109.7 (2)	C(6)—P(2)—Fe	108.4 (2)
C(8)—P(2)—Fe	114.0 (3)	C(10)—P(2)—Fe	121.5 (3)
C(16)—P(3)—Fe	107.0 (2)	C(18)—P(3)—Fe	114.6 (2)
C(20)—P(3)—Fe	124.1 (2)	C(12)—P(4)—Fe	122.5 (5)
C(14)—P(4)—Fe	120.9 (3)	C(15)—P(4)—Fe	110.9 (2)
C(4)—P(1)—C(2)	102.0 (3)	C(5)—P(1)—C(4)	103.3 (3)
C(8)—P(2)—C(6)	101.2 (4)	C(10)—P(2)—C(8)	105.3 (4)
C(18)—P(3)—C(16)	103.7 (3)	C(20)—P(3)—C(18)	103.1 (3)
C(15)—P(4)—C(12)	102.9 (5)	C(15)—P(4)—C(14)	101.7 (3)
C(1)—C(2)—P(1)	116.1 (5)	C(6)—C(5)—P(1)	108.9 (5)
C(9)—C(10)—P(2)	128.0 (7)	C(5)—C(6)—P(2)	109.2 (5)
C(19)—C(20)—P(3)	114.7 (4)	C(15)—C(16)—P(3)	108.6 (5)
C(13)—C(14)—P(4)	116.9 (5)	C(16)—C(15)—P(4)	110.3 (4)

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Acta Cryst. (1993). C**49**, 1059–1061

Structure of Diaquadibromobis(tetrahydrofuran)dilithium–Tetrakis(methyleneoxydiphenylphosphinato)dimercury(II), $[Hg(Br)\{CH_2P(O)Ph_2\}_2Li(H_2O)(C_4H_8O)]_2$

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(*Received* 23 *June* 1992; *accepted* 14 *January* 1993)

Abstract. Diaqua-1κO;2κO-dibromo-3κBr;4κBr-tetrakis-μ-[methyleneoxodiphenylphosphato(1 −)]-1κO:3κC;1κO:4κC;2κO:3κC;2κO:4κC-bis(tetrahydrofuran)-1κO;2κO-dilithiumdimercury, $[Li_2Hg_2(C_{13}H_2OP)_4Br_2(C_4H_8O)_2(H_2O)_2]$, $M_r = 1615.9574$, triclinic, $P\bar{1}$, $a = 10.253\ (3)$, $b = 12.765\ (3)$, $c = 13.407\ (3)$ Å, $\alpha = 63.42\ (2)$, $\beta = 82.38\ (2)$, $\gamma = 80.80\ (2)°$, $V = 1545.4\ (6)$ Å^3, $Z = 1$, $D_x = 1.74$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 63.92$ cm^{-1}, $F(000) = 788.0$, $T = 293$ K, $R = 0.0303$, $wR = 0.0358$ for 3240 reflections with $F_o^2 > 3\sigma(F_o^2)$ and 352 variables. The structure consists of two $Hg[CH_2P(O)Ph_2]_2$ units, which bridge between two Li^+ cations to form a 16-membered cycle. There is a Br^- anion weakly associated with each Hg atom. The coordination geometry around each Li^+ cation is approximately tetrahedral, involving one water and one tetrahydrofuran ligand at each cation, in addition to an O atom from each of the two bridging $Hg[CH_2P(O)Ph_2]_2$ units.

Introduction. The synthesis of the methyleneoxydiphenylphosphinate (mop)$^-$ ligand, $CH_2P(O)Ph_2^-$, and its characterization as the triphenyltin complex were reported 29 years ago (Seyferth, Welch & Heeren, 1964). Crystallographic investigations of complexes of mop have been limited. We have structurally characterized the mercury(II) complexes $[\{Hg[CH_2P(O)Ph_2]_2\}_2BrLi]_n$ (Fackler & Kresinski, 1991) and now report the structure of $[Hg(Br)\{CH_2P(O)Ph_2\}_2Li(H_2O)(C_4H_8O)]_2$.

Experimental. Crystals of $[Hg(Br)\{CH_2P(O)Ph_2\}_2Li(H_2O)(C_4H_8O)]_2$ were obtained upon slow evaporation of solvent from a concentrated solution of $[\{Hg[CH_2P(O)Ph_2]_2\}_2BrLi]_n$ (Fackler & Kresinski, 1991) in 50:50 CH_2Cl_2:0.1% H_2O in tetrahydrofuran. A crystal measuring approximately 0.2 × 0.3 × 0.5 mm was mounted in a random orientation on a glass fiber using cyanoacrylate adhesive. The crystal started to decay rapidly once data collection was complete. Decay was slight during data collection (< 5%). Unit-cell parameters were determined using 25 reflections between $25 < 2\theta < 30°$, and the crystal symmetry and axial dimensions were confirmed using axial photographs. Data were collected at room temperature using Wyckoff scanning technique in bisecting geometry on a Nicolet *R3m/E* diffractometer with graphite-monochromated Mo $K\alpha$ radiation. A total of 4412 data ($0 \leq h \geq 12$, $-14 \leq k \geq 14$, $-15 \leq l \geq 15$) were measured with $4 \leq 2\theta \leq 45°$. The scan range was 1° and the scan rate variable (3–30° min^{-1}). Three standards (244, 411 and $1\bar{1}0$) were measured every 97 reflections; data were corrected for slight standard decay, Lorentz and polarization effects. An empirical absorption correction was applied on the basis of five azimuthal scans; maximum and minimum transmissions were 0.951 and 0.678. The data were merged to give 3240 unique reflections. Data processing was carried out using the *SHELXTL* collection of crystallographic software (Sheldrick, 1986). The position of the Hg atom was determined by direct methods. The remaining non-H-atom positions were determined using difference Fourier syntheses. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

All non-H atoms were refined anisotropically. C-bonded H atoms were placed in idealized positions

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0108-2701/93/061059-03$06.00