bond angles vary from 106.59 (21) to 108.85 (22)°, the angle at the substituted atom being the smallest at 106.59 (21)°.

The two Cp groups are planar and eclipsed; a rotation of 6.6° from an exactly eclipsed conformation with the two rings approximately parallel (dihedral angle of 1.4° between the two rings). The P atom is displaced from the attached Cp plane away from the Fe atom by −0.034 (5) Å. Both phenyl rings are approximately orthogonal to the Cp planes. The dihedral angles between plane I [defined by C(1)–C(5)] and plane III [C(11)–C(16)], plane I and plane IV [C(17)–C(22)], and plane II [C(6)–C(20)] and plane IV are 86.64 (11), 97.59 (12) and 98.97 (13)°, respectively. The dihedral angle between the two phenyl rings is 78.33 (11)°. The phenyl rings are planar with the P atom slightly displaced from planes III and IV by 0.085 (4) and −0.031 (4) Å, respectively. The structural features of the title compound can be seen as intermediate between the ferrocene structure and that of 1,1′-bis(diphenylphosphino)-ferrocene.

JAA is grateful to the National Science Council of Taiwan for a senior postdoctoral fellowship. The authors are indebted to the National Science Council for partial financial support. We also thank Mr Y. S. Wen who helped in the diffraction-data collection.

References

BRUCE, M. I. (1982). *Comprehensive Organometallic Chemistry*, Vol. 9, edited by G. WILKINSON, F. G. A. STONE & E. W. ABEL, p. 1294, and references therein. Oxford: Pergamon Press.

CASELLATO, U., AJO, D., VALLE, G., CORAIN, B., LONGATO, B. & GRAZIANI, R. (1988). *J. Crystallogr. Spctrosc. Res.* **18**, 583–590.

EINSTEIN, F. M. B. & WILLIS, A. C. (1980). *Acta Cryst.* **B36**, 39–43.

GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 167–174. Oxford: Clarendon Press.

HAALAND, A. (1979). *Acc. Chem. Res.* **12**, 415–422.

HOULTON, A., ROBERTS, R. M. G., SILVER, J. & DREW, M. G. B. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1543–1547.

IBERS, J. A. & HAMILTON, W. C. (1974). *International Tables for X-ray Crystallography*, Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

LIU, L.-K. & CHEN, J.-C. (1991). *Bull. Inst. Chem. Acad. Sin.* **38**, 43–53.

MCEWEN, W. E., SULLIVAN, C. E. & DAY, R. O. (1983). *Organometallics*, **2**, 420–425.

ROBERTS, R. M. G., SILVER, J., YAMIN, B. M., DREW, M. G. B. & EBERHARDT, U. (1988). *J. Chem. Soc. Dalton Trans.* pp. 1549–1556.

WRIGHT, M. E. (1990). *Organometallics*, **9**, 853–856.

Acta Cryst. (1993). **C49**, 682–684

Structure of the 2-Pyridylhydrazone of Ferrocenecarbaldehyde

BY ANDREW HOULTON, MUSTAFA T. AHMET, JOHN R. MILLER, JACK SILVER AND ELIZABETH SLADE

Department of Chemistry and Biological Chemistry, University of Essex, Wivenhoe Park, Colchester CO4 3SQ, England

(*Received* 30 *March* 1992; *accepted* 13 *November* 1992)

Abstract. Ferrocenecarbaldehyde 2-pyridylhydrazone, $[\mathrm{Fe}(\eta^5\text{-}\mathrm{C_5H_5})(\eta^5\text{-}\mathrm{C_{11}H_{10}N_3})]$, $M_r = 305.17$, monoclinic, $P2_1/a$, $a = 9.972\ (1)$, $b = 8.308\ (1)$, $c = 17.010\ (2)$ Å, $\beta = 97.371\ (4)°$, $V = 1397.7$ Å^3, $Z = 4$, $D_x = 1.45\ \mathrm{g\ cm^{-3}}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0.71069$ Å, $\mu = 10.7\ \mathrm{cm^{-1}}$, $F(000) = 632$, $T = 293$ K, $R = 0.036$ and $wR = 0.049$ for 2283 observed reflections. The molecule exhibits an *E* configuration, has an N=C bond length of 1.274 (4) Å and an N—N bond length of 1.368 (4) Å. The cyclopentadienyl rings are eclipsed with Fe—C distances of 2.037 (4) to 2.051 (3) Å, mean 2.042 (4) Å. The cyclopentadienyl rings are essentially parallel. The pyridyl group is not coplanar with the cyclopentadienyl ring, but inclined at an angle of 27.5 (2)° to it.

Introduction. We have become interested in ferrocenyl derivatives that have the capacity to act as ligands (Houlton, Roberts, Silver & Drew, 1990; Houlton, Ibrahim, Dilworth & Silver, 1990; Houlton, Roberts, Silver & Parish, 1991). Such compounds have the potential to be redox-active, thus permitting the 'tuning' of the coordinated metal by oxidation of the ferrocenyl Fe atom. Oxidizable ferrocenyl ligands would allow the synthesis of potentially multifunctional drugs, *via* the known antitumour activity or iron-sandwich compounds (Köpf-Maier & Köpf, 1987; Houlton, Roberts & Silver, 1991) and the incorporation of chelated metal-containing cytotoxic groups. We report here the structure of the 2-pyridylhydrazone of ferro-

0108-2701/93/040682-03$06.00

Table 1. *Positional parameters and their e.s.d.'s*

$$B_{\mathrm{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}(Å^2)
Fe(1)	0.17081 (4)	0.01917 (5)	0.14798 (2)	2.876 (8)
C(1)	0.1101 (4)	−0.1440 (4)	0.2256 (2)	4.40 (7)
C(2)	0.1470 (4)	−0.2235 (4)	0.1582 (2)	4.98 (9)
C(3)	0.0638 (4)	−0.1649 (5)	0.0898 (2)	4.88 (8)
C(4)	−0.0247 (4)	−0.0514 (5)	0.1153 (2)	4.77 (8)
C(5)	0.0030 (4)	−0.0378 (4)	0.1992 (2)	4.49 (7)
C(6)	0.3283 (3)	0.1451 (4)	0.2078 (2)	3.40 (6)
C(7)	0.2193 (3)	0.2508 (4)	0.1791 (2)	4.05 (7)
C(8)	0.1976 (4)	0.2340 (4)	0.0943 (2)	4.53 (7)
C(9)	0.2898 (3)	0.1197 (5)	0.0721 (2)	4.57 (8)
C(10)	0.3713 (3)	0.0638 (5)	0.1415 (2)	3.95 (7)
C(11)	0.3796 (3)	0.1209 (4)	0.2910 (2)	3.66 (6)
C(12)	0.6549 (3)	−0.0370 (4)	0.4158 (2)	3.13 (6)
C(13)	0.7509 (3)	−0.0629 (4)	0.3640 (2)	3.78 (7)
C(14)	0.8738 (4)	−0.1266 (5)	0.3950 (2)	4.61 (8)
C(15)	0.9000 (4)	−0.1603 (5)	0.4743 (2)	4.75 (8)
C(16)	0.8001 (3)	−0.1299 (4)	0.5208 (2)	4.25 (7)
N(1)	0.4932 (3)	0.0522 (3)	0.3107 (1)	3.39 (5)
N(2)	0.5293 (3)	0.0261 (4)	0.3899 (2)	3.80 (6)
N(3)	0.6779 (3)	−0.0700 (4)	0.4933 (2)	3.60 (5)

Table 2. *Bond lengths* (Å) *and angles* (°) *for the title compound with e.s.d.'s in parentheses*

Fe(1)—C(1)	2.037 (3)	C(6)—C(7)	1.433 (5)
Fe(1)—C(2)	2.040 (3)	C(6)—C(10)	1.427 (5)
Fe(1)—C(3)	2.046 (3)	C(6)—C(11)	1.456 (5)
Fe(1)—C(4)	2.044 (3)	C(7)—C(8)	1.437 (5)
Fe(1)—C(5)	2.039 (3)	C(8)—C(9)	1.407 (5)
Fe(1)—C(6)	2.046 (4)	C(9)—C(10)	1.422 (5)
Fe(1)—C(7)	2.037 (3)	C(11)—N(1)	1.274 (5)
Fe(1)—C(8)	2.038 (4)	C(12)—C(13)	1.398 (6)
Fe(1)—C(9)	2.041 (3)	C(12)—N(2)	1.377 (6)
Fe(1)—C(10)	2.051 (3)	C(12)—N(3)	1.337 (6)
C(1)—C(2)	1.411 (4)	C(13)—C(14)	1.376 (5)
C(1)—C(5)	1.413 (5)	C(14)—C(15)	1.369 (5)
C(2)—C(3)	1.425 (4)	C(15)—C(16)	1.372 (6)
C(3)—C(4)	1.398 (4)	C(16)—N(3)	1.344 (6)
C(4)—C(5)	1.424 (5)	N(1)—N(2)	1.368 (5)

C(1)—Fe(1)—C(6)	106.9 (1)	Fe(1)—C(6)—C(7)	69.1 (2)
C(1)—Fe(1)—C(7)	122.7 (1)	Fe(1)—C(6)—C(10)	69.8 (2)
C(1)—Fe(1)—C(8)	160.0 (1)	Fe(1)—C(6)—C(11)	124.4 (3)
C(1)—Fe(1)—C(9)	158.0 (1)	Fe(1)—C(7)—C(6)	69.8 (2)
C(1)—Fe(1)—C(10)	121.9 (1)	Fe(1)—C(7)—C(8)	69.4 (2)
C(2)—Fe(1)—C(6)	123.6 (1)	Fe(1)—C(8)—C(7)	69.3 (2)
C(2)—Fe(1)—C(7)	159.7 (1)	Fe(1)—C(8)—C(9)	69.9 (2)
C(2)—Fe(1)—C(8)	158.0 (1)	Fe(1)—C(9)—C(8)	69.7 (2)
C(2)—Fe(1)—C(9)	122.8 (1)	Fe(1)—C(9)—C(10)	70.0 (2)
C(2)—Fe(1)—C(10)	108.0 (1)	Fe(1)—C(10)—C(6)	69.4 (2)
C(3)—Fe(1)—C(6)	160.4 (1)	Fe(1)—C(10)—C(9)	69.3 (2)
C(3)—Fe(1)—C(7)	157.4 (1)	C(1)—C(2)—C(3)	108.6 (2)
C(3)—Fe(1)—C(8)	121.9 (1)	C(1)—C(5)—C(4)	108.1 (3)
C(3)—Fe(1)—C(9)	108.0 (1)	C(2)—C(1)—C(5)	107.4 (3)
C(3)—Fe(1)—C(10)	124.1 (1)	C(2)—C(3)—C(4)	107.6 (2)
C(4)—Fe(1)—C(6)	157.9 (1)	C(3)—C(4)—C(5)	108.4 (2)
C(4)—Fe(1)—C(7)	121.9 (1)	C(6)—C(7)—C(8)	107.0 (3)
C(4)—Fe(1)—C(8)	107.7 (1)	C(6)—C(10)—C(9)	107.6 (3)
C(4)—Fe(1)—C(9)	123.9 (1)	C(6)—C(11)—N(1)	120.5 (3)
C(4)—Fe(1)—C(10)	160.1 (1)	C(7)—C(6)—C(10)	108.2 (3)
C(5)—Fe(1)—C(6)	121.7 (2)	C(7)—C(6)—C(11)	125.0 (3)
C(5)—Fe(1)—C(7)	106.8 (1)	C(7)—C(8)—C(9)	108.4 (3)
C(5)—Fe(1)—C(8)	123.8 (2)	C(8)—C(9)—C(10)	108.8 (3)
C(5)—Fe(1)—C(9)	160.2 (2)	C(10)—C(6)—C(11)	126.8 (3)
C(5)—Fe(1)—C(10)	157.6 (2)	C(11)—N(1)—N(2)	116.3 (4)
Fe(1)—C(1)—C(2)	69.8 (2)	C(12)—C(13)—C(14)	117.7 (3)
Fe(1)—C(1)—C(5)	69.8 (2)	C(12)—N(2)—N(1)	119.3 (4)
Fe(1)—C(2)—C(1)	69.7 (2)	C(12)—N(3)—C(16)	116.9 (4)
Fe(1)—C(2)—C(3)	69.8 (2)	C(13)—C(12)—N(2)	121.7 (4)
Fe(1)—C(3)—C(2)	69.4 (2)	C(13)—C(12)—N(3)	123.0 (4)
Fe(1)—C(3)—C(4)	69.9 (2)	C(13)—C(14)—C(15)	120.3 (3)
Fe(1)—C(4)—C(3)	70.1 (2)	C(14)—C(15)—C(16)	118.0 (3)
Fe(1)—C(4)—C(5)	69.4 (2)	C(15)—C(16)—N(3)	124.1 (4)
Fe(1)—C(5)—C(1)	69.6 (2)	N(2)—C(12)—N(3)	115.3 (4)
Fe(1)—C(5)—C(4)	69.7 (2)		

cenecarbaldehyde (1), a bidentate ligand containing nitrogen donor atoms.

Experimental. All solvents were dried according to methods reported by Perrin & Armarego (1988). The title compound was synthesized by refluxing ferrocenecarbaldehyde (4 g, 13 mmol) with a slight excess of 2-pyridylhydrazine (2 g, 18 mmol) in dichloromethane for 2 h. The volume of solution was reduced and left to stand at room temperature. The resulting solid was filtered and washed with cold ether. Analysis found (calc.): C = 62.9 (63.0), H = 5.0 (4.9), N = 13.8 (13.8). A plate-like orange crystal of size 0.15 × 0.5 × 0.7 mm was selected and mounted on an Enraf–Nonius CAD-4 diffractometer. The cell dimensions were determined from the setting angles of 25 reflections in the range $14 < \theta < 20°$ and refined after accurate setting of 25 reflections in the range $20 < \theta < 24°$. Intensity data were measured with graphite-monochromated Mo $K\alpha$ radiation. Data collection was carried out by ω–2θ scan, using the method *FLAT* (Enraf–Nonius, 1990) in which each collection was carried out at an azimuthal angle (ψ) calculated to minimize absorption by minimizing the path of the X-rays through the crystal. 2611 reflections were scanned up to $\theta = 25°$, of which 2608 were unique. 2283 [$I > 3.0\sigma(I)$] reflections were considered observed and used in the analysis, index range h 0 to 11, k 0 to 9, l −20 to 20. Three standard reflections measured every hour showed a small linear decay which was corrected for. As the data were collected using *FLAT* (Enraf–Nonius, 1990), no absorption correction was applied. The structure was determined by Patterson methods using *SHELXS*86 (Sheldrick, 1986) and refined by least squares using anisotropic thermal parameters for all non-H atoms. All H atoms were included in calculated positions, riding on their respective bonded atoms with the exception of H(35) which was located in a difference map and then ridden on N(5). Killean & Lawrence (1969) weights were used in the least-squares refinement, where $w^{-1} = \sigma^2F + (0.02F^2) + 1$. The refinement was carried out on F values and converged to $R = 0.036$ and $wR = 0.049$ with max. shift/e.s.d. < 0.03. The final difference map showed no peaks greater than ± 0.64 e Å^{-3}. The final atomic coordinates are listed in Table 1.* Scattering factors

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and geometry, and root-mean-square amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55705 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1007]

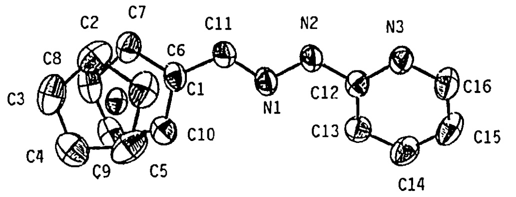

Fig. 1. An *ORTEP* (Johnson, 1965) diagram of the title compound.

were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were carried out on a MicroVAX using *SHELXS*86 (Sheldrick, 1986), and *MolEN* (Enraf–Nonius, 1990). Diagrams were drawn with *ORTEP* (Johnson, 1965).

Discussion. Bond lengths and angles are listed in Table 2. The atomic numbering scheme is shown in Fig. 1. The distance from Fe to the centre of the cyclopentadienyl ring is 1.644 (1) Å for the unsubstituted ring and 1.649 (1) Å for the other. The average Fe—C distance is 2.042 (4) Å.

We have previously reported the structure of $[Fe\{C_5H_4C(CH_3)NNHC_5NH^+\}_2][CH_3COO^-]_2$ (Houlton, Dilworth, Roberts, Silver & Drew, 1990). In this salt, the average C=N distance is 1.289 (19) Å and the average N—N distance is 1.384 (13) Å, which are longer than in (1). However, owing to the large errors reported these differences cannot be considered significant. In comparison with similar compounds (Houlton, Dilworth, Roberts, Silver & Drew, 1990), the N—N bond length in (1) at 1.368 Å is at the shorter end of the observed range, indicating some double-bond character. This is consistent with the trigonal geometry of N(2) indicating a delocalization of the lone pair. This feature explains the observed coordination chemistry of ligands of this type, in that the N(1)- and N(3)-type atoms and not N(2) are involved in ligation (Chiswell & Lions, 1964).

JS acknowledges the SERC for a CASE studentship for ES.

References

Chiswell, B. & Lions, F. (1964). *Inorg. Chem.* **4**, 490–492.
Enraf–Nonius (1990). *MolEN. An Interactive Structure Solution Procedure*. Enraf–Nonius, Delft, The Netherlands.
Houlton, A., Dilworth, J. R., Roberts, R. M. G., Silver, J. & Drew, M. G. B. (1990). *Polyhedron*, **9**, 2751–2757.
Houlton, A., Ibrahim, S. K., Dilworth, J. R. & Silver, J. (1990). *J. Chem. Soc. Dalton Trans.* pp. 2421–2424.
Houlton, A., Roberts, R. M. G. & Silver, J. (1991). *J. Organomet. Chem.* **418**, 107–112.
Houlton, A., Roberts, R. M. G., Silver, J. & Drew, M. G. B. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1543–1547.
Houlton, A., Roberts, R. M. G., Silver, J. & Parish, R. V. (1991). *J. Organomet. Chem.* **418**, 269–275.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* B**25**, 1750–1752.
Köpf-Maier, P. & Köpf, H. (1987). *Chem. Rev.* **87**, 1137–1152.
Perrin, D. D. & Armarego, W. L. F. (1988). *Purification of Laboratory Chemicals*, 3rd ed. London: Pergamon Press.
Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Acta Cryst. (1993). C**49**, 684–687

{Bis[2-(4-ethoxyphenyltelluro)ethyl]amino-*N*,*Te*,*Te′*}chloroplatinum(II) Chloride Trichloromethane Monohydrate

By John E. Drake* and Jane H. E. Bailey

Department of Chemistry and Biochemistry, University of Windsor, Windsor, Ontario, Canada N9B 3P4

and Ajai K. Singh* and Vinod Srivastava

Department of Chemistry, Indian Institute of Technology, New Delhi-110 016, India

(*Received* 10 *February* 1992; *accepted* 22 *October* 1992)

Abstract. $[PtCl\{(C_{10}H_{13}OTe)_2NH\}]Cl.CHCl_3.H_2O$, $M_r = 972.2$, triclinic, $P\bar{1}$, $a = 10.590$ (7), $b = 17.606$ (6), $c = 8.493$ (3) Å, $\alpha = 90.10$ (3), $\beta = 102.22$ (5), $\gamma = 106.85$ (4)°, $V = 1478$ (3) Å^3, $Z = 2$, $D_m = 2.20$, $D_x = 2.18$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 7.218$ mm^{-1}, $F(000) = 908$, $T = 297$ (1) K, final $R = 0.0429$, $wR = 0.0446$ for 2910 unique observed reflections, $S = 1.38$. The Te atoms are coordinated in a *trans* fashion with the Pt—Te bond lengths of 2.557 (2) and 2.564 (2) Å being

---

* To whom correspondence should be addressed.

0108-2701/93/040684-04$06.00