Crystal structure of 3-ferrocenyl-1-phenylpyrazole-4-carbaldehyde 3-pyridinylmethylimine, $Fe(C_5H_5)(C_{21}H_{17}N_4)$

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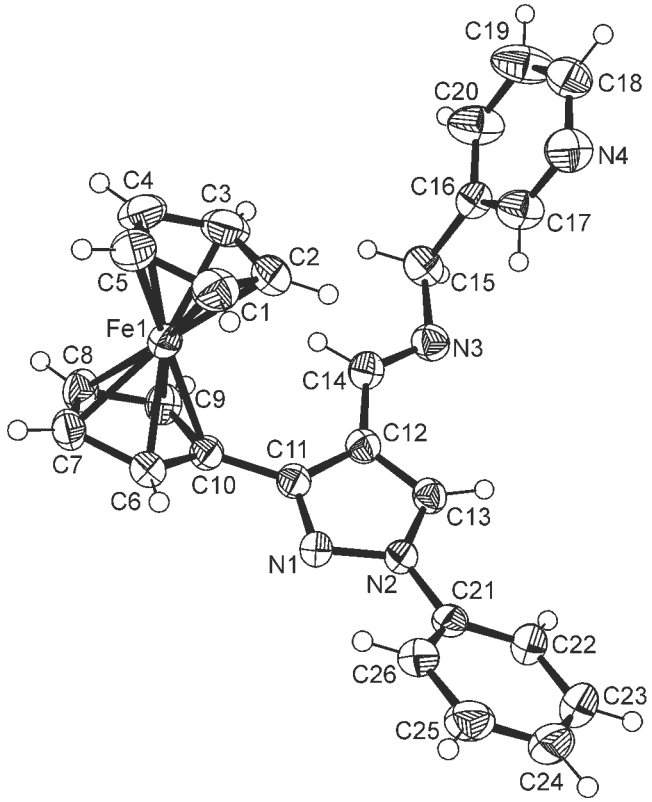

Abstract

$C_{26}H_{22}FeN_4$, orthorhombic, *Pbca* (no. 61), $a = 10.6044(2)$ Å, $b = 19.1419(4)$ Å, $c = 20.4700(3)$ Å, $V = 4155.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.029$, $wR_{ref}(F^2) = 0.080$, $T = 233$ K.

Source of material

The title compound [CARN: 918342-49-1] was prepared from 3-ferrocenyl-1-phenylpyrazole-4-carbaldehyde and 3-pyridylmethylamine, as described previously [1]. Single crystals were obtained from methanol. As a representative example of combination of a metallocene and a heterocycle, it is of current interest in the evolving field of bioorganometallic chemistry [2]. Recently, related imines were found to exhibit antimicrobial activity [3].

Discussion

The Cp rings of the ferrocene unit adopt an eclipsed conformation. The adjacent Cp ring is rotated out of the pyrazole ring plane by 25°, and the interplanar angle of the pyrazole and phenyl rings is 23°. The pyridine group is also twisted out of the plane of the central pyrazole ring. The molecules are connected by hydrogen bonds between the pyrazole CH and the pyridine N atom in a zigzag arrangement which extends into the direction of the crystallographic *a* axis. The hydrogen bond parameters (donor···acceptor distance, H···acceptor distance, donor–H···acceptor angle) of C13–H···N4^{i} are 3.351 Å, 2.44 Å, 164°. Symmetry operation (i): 1/2+*x*,*y*,1/2–*z*.

Table 1. Data collection and handling.

Crystal:	yellow parallelepiped, size 0.1 × 0.15 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.47 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	24408, 4058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3418
$N(param)_{refined}$:	280
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.1996	0.9640	0.3461	0.061
H(2)	8*c*	0.0366	1.0064	0.4271	0.063
H(3)	8*c*	–0.0606	0.9009	0.4801	0.067
H(4)	8*c*	0.0411	0.7940	0.4329	0.065
H(5)	8*c*	0.2022	0.8327	0.3500	0.061
H(6)	8*c*	0.4148	0.9726	0.4611	0.042
H(7)	8*c*	0.2524	1.0113	0.5437	0.049
H(8)	8*c*	0.1608	0.9041	0.5964	0.053
H(9)	8*c*	0.2638	0.7983	0.5465	0.047
H(13)	8*c*	0.5549	0.7073	0.3557	0.040
H(14)	8*c*	0.2623	0.7263	0.4569	0.044
H(15A)	8*c*	0.1470	0.6413	0.4422	0.053
H(15B)	8*c*	0.2183	0.5698	0.4294	0.053
H(17)	8*c*	0.2409	0.6592	0.2886	0.059
H(18)	8*c*	–0.0478	0.5840	0.1944	0.061
H(19)	8*c*	–0.1278	0.5379	0.2886	0.085
H(20)	8*c*	–0.0192	0.5521	0.3862	0.074
H(22)	8*c*	0.7688	0.7231	0.3244	0.053
H(23)	8*c*	0.9363	0.7527	0.2560	0.066
H(24)	8*c*	0.9799	0.8682	0.2328	0.068
H(25)	8*c*	0.8602	0.9560	0.2796	0.064
H(26)	8*c*	0.6911	0.9280	0.3478	0.051

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	8*c*	0.19326(2)	0.90234(1)	0.46487(1)	0.0301(1)	0.0395(2)	0.0311(1)	0.00140(9)	0.00210(9)	–0.00285(9)
N(1)	8*c*	0.5559(1)	0.85626(6)	0.42121(6)	0.0320(7)	0.0353(7)	0.0328(6)	0.0012(5)	0.0013(5)	–0.0056(5)
N(2)	8*c*	0.6067(1)	0.80653(7)	0.38119(6)	0.0293(6)	0.0362(7)	0.0313(6)	0.0023(5)	–0.0006(5)	–0.0039(5)
N(3)	8*c*	0.3170(1)	0.65388(7)	0.40045(7)	0.0408(8)	0.0382(8)	0.0420(8)	–0.0054(6)	0.0009(6)	–0.0008(6)
N(4)	8*c*	0.1039(2)	0.62585(9)	0.23233(7)	0.052(1)	0.069(1)	0.0411(8)	–0.0047(8)	0.0000(7)	0.0119(8)
C(1)	8*c*	0.1489(2)	0.9357(1)	0.37289(8)	0.048(1)	0.067(1)	0.0371(9)	0.0026(9)	–0.0079(8)	0.0047(8)
C(2)	8*c*	0.0573(2)	0.9596(1)	0.41835(9)	0.045(1)	0.058(1)	0.055(1)	0.0133(9)	–0.0122(8)	–0.0044(9)
C(3)	8*c*	0.0030(2)	0.9004(1)	0.4480(1)	0.0299(9)	0.080(2)	0.059(1)	0.0015(9)	–0.0002(8)	–0.005(1)
C(4)	8*c*	0.0599(2)	0.8404(1)	0.4216(1)	0.043(1)	0.058(1)	0.062(1)	–0.0108(9)	–0.0104(9)	–0.0053(9)
C(5)	8*c*	0.1503(2)	0.8621(1)	0.37506(9)	0.045(1)	0.066(1)	0.042(1)	0.0052(9)	–0.0092(8)	–0.0155(9)
C(6)	8*c*	0.3660(2)	0.94301(8)	0.48760(7)	0.0345(8)	0.0364(8)	0.0347(8)	–0.0013(7)	–0.0021(6)	–0.0039(6)
C(7)	8*c*	0.2746(2)	0.96487(9)	0.53422(8)	0.0445(9)	0.0420(9)	0.0366(9)	0.0063(8)	–0.0036(7)	–0.0106(7)
C(8)	8*c*	0.2231(2)	0.90459(9)	0.56369(8)	0.047(1)	0.055(1)	0.0297(8)	0.0057(8)	0.0059(7)	–0.0043(7)
C(9)	8*c*	0.2811(2)	0.84508(9)	0.53567(7)	0.0457(9)	0.0412(9)	0.0311(8)	0.0013(7)	0.0045(7)	0.0028(6)
C(10)	8*c*	0.3708(1)	0.86845(8)	0.48801(7)	0.0318(8)	0.0365(8)	0.0290(7)	0.0026(7)	–0.0015(6)	–0.0016(6)
C(11)	8*c*	0.4516(1)	0.82699(8)	0.44510(7)	0.0322(8)	0.0349(8)	0.0279(7)	0.0024(6)	–0.0023(6)	0.0003(6)
C(12)	8*c*	0.4355(1)	0.75796(8)	0.42065(7)	0.0337(8)	0.0323(8)	0.0318(7)	0.0012(6)	–0.0024(6)	0.0007(6)
C(13)	8*c*	0.5373(1)	0.74782(8)	0.37992(7)	0.0349(8)	0.0312(8)	0.0327(8)	0.0033(6)	–0.0036(6)	–0.0025(6)
C(14)	8*c*	0.3276(2)	0.71196(8)	0.42891(8)	0.0394(9)	0.0355(9)	0.0344(8)	0.0002(7)	0.0033(7)	0.0023(7)
C(15)	8*c*	0.1993(2)	0.6158(1)	0.41071(8)	0.051(1)	0.044(1)	0.0370(9)	–0.0110(8)	0.0022(7)	0.0034(7)
C(16)	8*c*	0.1264(2)	0.60635(8)	0.34790(8)	0.0371(9)	0.0330(8)	0.0382(9)	0.0001(7)	0.0043(7)	0.0030(6)
C(17)	8*c*	0.1654(2)	0.6334(1)	0.28906(9)	0.046(1)	0.055(1)	0.045(1)	–0.0117(9)	–0.0006(8)	0.0119(8)
C(18)	8*c*	–0.0032(2)	0.5903(1)	0.23360(9)	0.052(1)	0.052(1)	0.048(1)	–0.0010(9)	–0.0109(9)	0.0042(8)
C(19)	8*c*	–0.0512(2)	0.5625(1)	0.2894(1)	0.057(1)	0.083(2)	0.072(1)	–0.032(1)	–0.019(1)	0.031(1)
C(20)	8*c*	0.0135(2)	0.5707(1)	0.3473(1)	0.049(1)	0.084(2)	0.052(1)	–0.023(1)	–0.0033(9)	0.027(1)
C(21)	8*c*	0.7137(1)	0.82342(9)	0.34174(7)	0.0280(8)	0.0456(9)	0.0293(8)	–0.0009(7)	–0.0038(6)	–0.0038(7)
C(22)	8*c*	0.7866(2)	0.7702(1)	0.31507(8)	0.0406(9)	0.050(1)	0.0411(9)	0.0042(8)	0.0040(7)	–0.0077(8)
C(23)	8*c*	0.8863(2)	0.7881(1)	0.27446(9)	0.043(1)	0.072(1)	0.051(1)	0.004(1)	0.0106(8)	–0.0147(9)
C(24)	8*c*	0.9128(2)	0.8568(1)	0.26090(9)	0.040(1)	0.080(2)	0.049(1)	–0.012(1)	0.0111(8)	–0.006(1)
C(25)	8*c*	0.8411(2)	0.9090(1)	0.2884(1)	0.043(1)	0.062(1)	0.054(1)	–0.0162(9)	0.0037(9)	0.0008(9)
C(26)	8*c*	0.7404(2)	0.89245(9)	0.32911(9)	0.0359(9)	0.047(1)	0.0434(9)	–0.0035(8)	0.0005(7)	–0.0036(7)

References

1. Joksović, M.; Ratković, Z.; Vukićević, M.; Vukićević, R. D.: Synthesis of 1*H*-3-ferrocenyl-1-phenyl-4-substituted pyrazoles. Synlett **16** (2006) 2581-2584.
2. Jaouen, G.; Beck, W.; McGlinchey, M. J.: A novel field of research: bioorganometallic chemistry, origins, and founding principles. In: *Bioorganometallics: Biomolecules, Labeling, Medicine* (Ed.: G. Jaouen), p. 1-37. Wiley-VCH, Weinheim 2006.
3. Damljanović, I.; Ćolović, M.; Vukićević, M.; Manojlović, D.; Radulović, N.; Wurst, K.; Laus, G.; Ratković, Z.; Joksović, M.; Vukićević, R. D.: Synthesis, spectral characterization and electrochemical properties of 3-(*o*-, *m*- and *p*-ferrocenylphenyl)-1-phenylpyrazole-4-carboxaldehydes. J. Organomet. Chem. (2009) 1575-1580.
4. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.