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trans-4-(Ferrocenylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.076
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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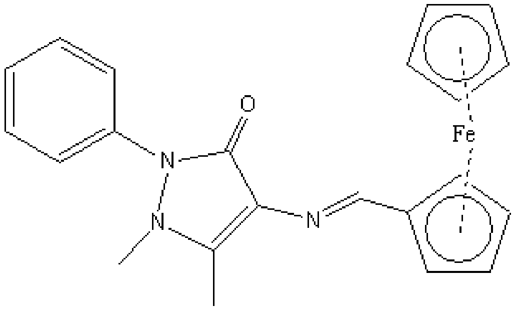
The title compound, $[Fe(C_5H_5)(C_{17}H_{16}N_3O)]$, a new Schiff base containing a ferrocenyl group, has been characterized structurally. The central C—N═C—C linkage has a near-planar geometry, indicating extensive conjugation.

Comment

Some Schiff bases bearing the ferrocenyl group and their complexes are excellent non-linear optical materials and liquid crystals (Colbert *et al.*, 1995) because of their strong electron donors and electron-flow bridges. In the course of our investigation of the coordination of Schiff bases with transition metal salts, we observed that the title compound, (I), coordinates readily with Ni^{II} and Cu^{II} salts as an *N*,*O*-bidentate ligand. The crystal structure of (I) is reported here (Fig. 1).



(I)

In (I), all the bond lengths are within normal range (Allen *et al.*, 1987). The N3—C12 bond length [1.277 (3) Å] confirms that it is a C═N double bond. The dihedral angle between the five-membered pyrazole ring (N1/N2/C9/C8/C7) and the plane

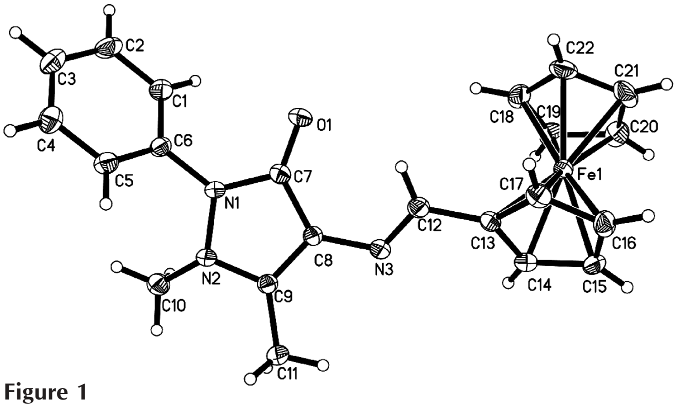


Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radius.

formed by atoms C8/N3/C12/C13 is 28.0 (3)°. This deviation may be caused by the steric hindrance between atom O1 and the H atom attached directly to C12. The C8—N3—C12—C13 torsion angle is 178.56 (19)°.

Experimental

A solution of ferrocenylaldehyde (0.214 g, 1 mmol) in absolute ethanol (10 ml) was added dropwise to a solution of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (0.203 g, 1 mmol) in absolute ethanol (10 ml). The mixture was refluxed and stirred for 2 h and a yellow solid precipitated. The solid was isolated, washed three times with cold absolute ethanol and dried in a vacuum desiccator with anhydrous $CaCl_2$ (yield: 81%). A yellow single crystal suitable for X-ray analysis was obtained by slow evaporation of an ethyl acetate solution at room temperature over a period of a month. Analysis calculated for $C_{22}H_{21}FeN_3O$: C 66.18, H 5.30, N 10.52%; found: C 66.10, H 5.41, N 10.69%.

Crystal data

$[Fe(C_5H_5)(C_{17}H_{16}N_3O)]$
$M_r = 399.27$
Monoclinic, $P2_1/n$
$a = 7.4749\ (6)$ Å
$b = 17.426\ (2)$ Å
$c = 14.443\ (2)$ Å
$\beta = 101.087\ (8)°$
$V = 1846.1\ (4)$ Å^3
$Z = 4$
$D_x = 1.436$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 34 reflections
$\theta = 6.2$–$14.5°$
$\mu = 0.83$ mm^{-1}
$T = 296\ (2)$ K
Prism, yellow
0.48 × 0.32 × 0.24 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.694$, $T_{max} = 0.819$
3870 measured reflections
3335 independent reflections
2530 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25.3°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 20$
$l = -17 \rightarrow 17$
3 standard reflections every 97 reflections intensity decay: 5.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.076$
$S = 1.00$
3335 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0066 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.232 (3)	C7—C8	1.438 (3)
N3—C12	1.277 (3)	C8—C9	1.359 (3)
N3—C8	1.407 (3)	C12—C13	1.459 (3)
C12—N3—C8	119.7 (2)	N3—C8—C7	129.0 (2)
O1—C7—C8	131.8 (2)	N3—C12—C13	120.4 (2)
C9—C8—N3	122.5 (2)		
C7—N1—N2—C9	−9.6 (2)	O1—C7—C8—N3	−2.0 (4)
C6—N1—N2—C9	−163.45 (19)	C8—N3—C12—C13	178.56 (19)
C6—N1—C7—C8	160.0 (2)	N3—C12—C13—C14	26.9 (3)
O1—C7—C8—C9	176.4 (3)		

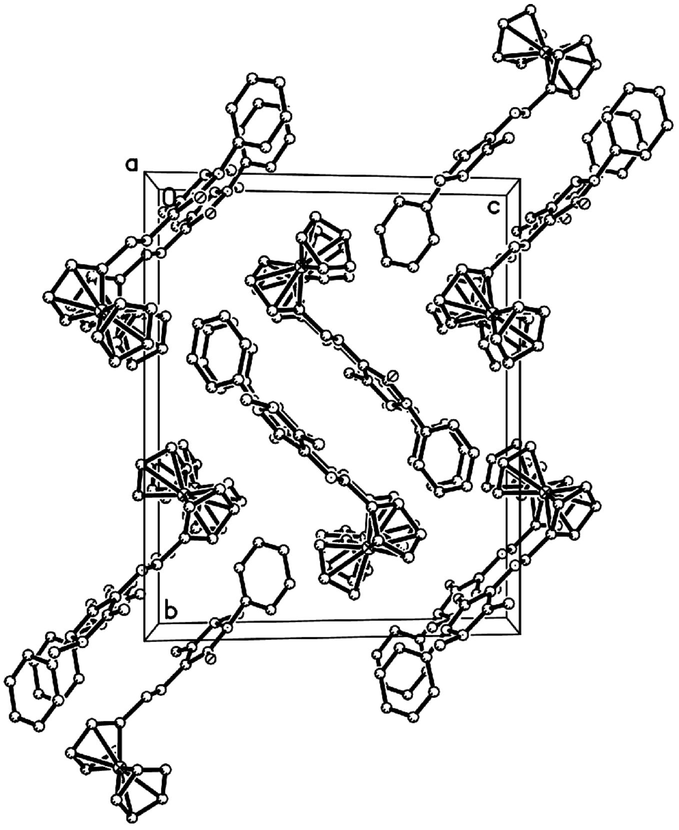


Figure 2
The crystal packing of (I), viewed along the *a* axis. H atoms have been omitted.

All H atoms were initially located in a difference Fourier map. All H atoms were then constrained to an ideal geometry, with C—H distances of 0.93–0.96 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997*b*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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