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N,*N*′-Butanedioylbis(5-ferrocenyl-3-methyl-1*H*-pyrazole)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.061
wR factor = 0.207
Data-to-parameter ratio = 14.6

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

The title compound, $[Fe_2(C_5H_5)_2(C_{22}H_{20}N_4O_2)]$, has been synthesized by the reaction of ferrocenoylacetone and butanedioic acid dihydrazide in ethanol. The dihedral angles between one pyrazole ring and the attached cyclopentadienyl ring and carbonyl plane are 33.5 (4) and 4.4 (8)°, respectively; for the other pyrazole ring, the corresponding angles are 29.5 (4) and 11.2 (8)°, respecively.

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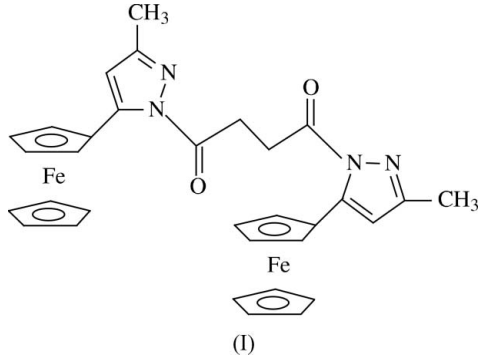
Comment

Pyrazole compounds are finding increasing numbers of applications as ligands in coordination chemistry (Chakrabarty *et al.*, 2004; Davies *et al.*, 2005; Hardie *et al.*, 2004; Michaud *et al.*, 2005*a*,*b*; Zhao & Eichhorn, 2005). As part of a continuing investigation of the chemistry of ferrocene derivatives, the title compound, (I), was synthesized (Shi, 2005*a*,*b*; Shi, Yang, Shen *et al.*, 2004; Shi, Yang, Song *et al.*, 2004) and its crystal structure determined (Fig. 1).



(I)

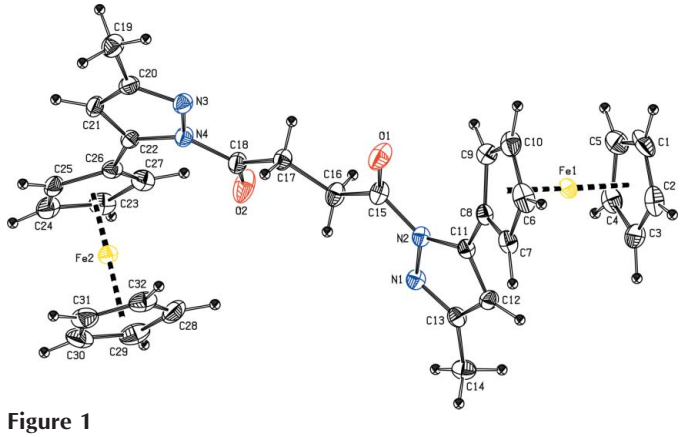


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

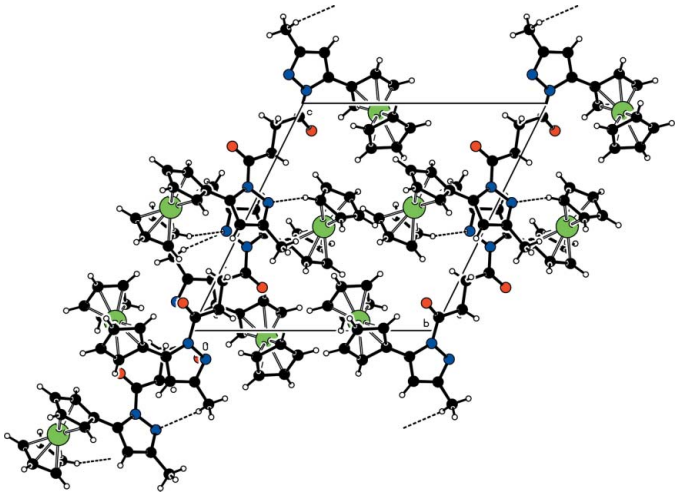


Figure 2
Packing diagram of (I). Dashed lines indicate hydrogen bonds.

As shown in Table 1, the corresponding bond lengths and angles in the two pyrazole rings are almost identical. Moreover, for each of two pyrazole rings, the bond lengths indicate electron delocalization (Shi, Yang, Shen *et al.*, 2004; Shi, Yang, Song *et al.*, 2004). However, the dihedral angles between the pyrazole ring and the attached cyclopentadienyl ring and carbonyl plane are different. For pyrazole ring N1–C13, the dihedral angles are 33.5 (4) and 4.4 (8)°; for pyrazole ring N3–C22, the values are 29.5 (4) and 11.2 (8)°. Interestingly, the above dihedral angles and C8—C11 and C22—C26 bond lengths suggest that the pyrazole ring and the corresponding substituted cyclopentadienyl ring are not conjugated. As was previously observed in a ferrocene-containing compound, the cyclopentadienyl rings of the two ferrocenyl groups are in the eclipsed conformation (Erasmus *et al.*, 1996).

As shown in Fig. 2, intermolecular C—H···N(NCO) hydrogen bonds are present in the crystalline state of (I) (Table 2).

Experimental

An ethanol solution of ferrocenoylacetone and butanedioic acid dihydrazide (2:1) in the presence of *p*-TsOH was refluxed for 1 h. After removal of the solvent, the resulting residue was chromatographed on a silica-gel column using CH_2Cl_2/diethyl ether (20/1 v/v) as eluant. The orange–yellow band was then collected and further recrystallized from CH_2Cl_2/petroleum ether to afford the title compound (m.p. 407 K). Analysis calculated for $C_{32}H_{30}Fe_2N_4O_2$: C 62.57, H 4.92, N 9.12%; found: C 62.11, H 4.97, N 8.74%.

Crystal data

$[Fe_2(C_5H_5)_2(C_{22}H_{20}N_4O_2)]$
M_r = 614.30
Triclinic, $P\overline{1}$
a = 9.0105 (9) Å
b = 12.633 (3) Å
c = 13.717 (3) Å
α = 64.45 (3)°
β = 73.85 (3)°
γ = 88.61 (3)°
V = 1344.8 (6) Å^3
Z = 2
D_x = 1.517 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 1.12 mm^{-1}
T = 293 K
Prism, orange
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.656, T_{max} = 0.796
5623 measured reflections
5268 independent reflections
4140 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.0°
$h = 0 \rightarrow 11$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$
3 standard reflections
every 200 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.207
S = 0.93
5268 reflections
362 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 8.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.018 (2)

Table 1
Selected bond lengths (Å).

O1—C15	1.203 (7)	O2—C18	1.195 (7)
N1—C13	1.313 (7)	N3—C20	1.315 (7)
N1—N2	1.385 (6)	N3—N4	1.389 (6)
N2—C11	1.392 (6)	N4—C18	1.415 (7)
N2—C15	1.403 (7)	N4—C22	1.391 (7)
C8—C11	1.476 (7)	C19—C20	1.491 (8)
C11—C12	1.365 (7)	C20—C21	1.409 (8)
C12—C13	1.404 (8)	C21—C22	1.373 (8)
C13—C14	1.488 (8)	C22—C26	1.468 (7)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N1^{i}	0.98	2.58	3.534 (9)	164
C19—H19B···N1^{ii}	0.96	2.62	3.493 (9)	152

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $-x+2, -y, -z$.

All H atoms were placed at geometrically idealized positions and were treated as riding atoms, with C—H = 0.93–0.98Å and U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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