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## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å  
 $R$  factor = 0.050  
 $wR$  factor = 0.108  
Data-to-parameter ratio = 13.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.1,1'-[1,1'-(Pentane-1,5-diyl)dicarbonyl]-  
bis(5-methyl-1*H*-pyrazol-3-yl)ferrocene

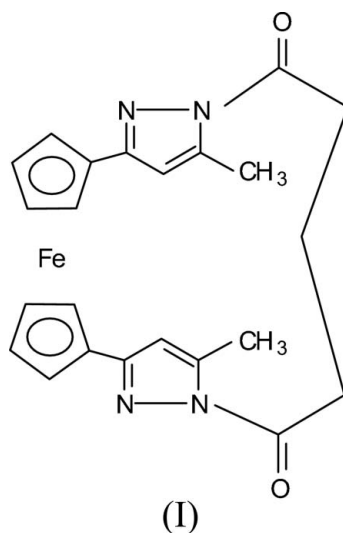
Molecules of the title compound,  $[\text{Fe}(\text{C}_{23}\text{H}_{22}\text{N}_4\text{O}_2)]$ , are linked by two intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds into a chain along  $[010]$ . Adjacent chains are connected by a  $\text{C}-\text{H}\cdots\pi$  hydrogen bond involving the substituted cyclopentadienyl ring as an acceptor, leading to a double-chain along  $[010]$ . Two such chains are further packed along the  $a$  axis via a  $\text{C}-\text{H}\cdots\pi$  hydrogen bond involving the pyrazole ring as an acceptor to generate a four-chain strand along  $[100]$ . The combination of the four-stranded chain and the double-chain leads to a three-dimensional network.

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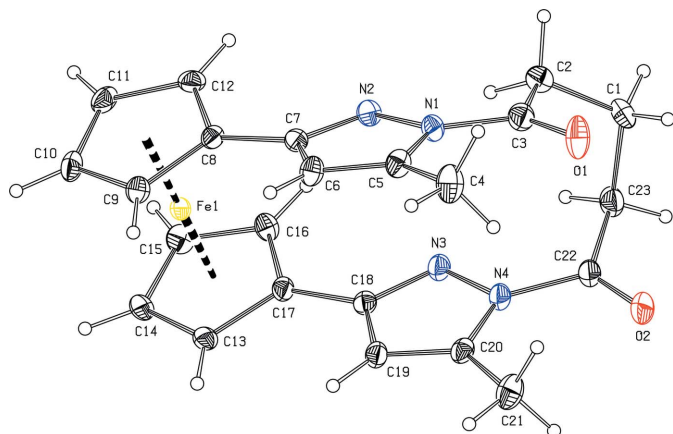
## Comment

Pyrazoles and related compounds are important ligands in coordination chemistry (Hardie *et al.*, 2004; Morawitz *et al.*, 2006; Otero *et al.*, 2004; Shirin & Carrano, 2004), organometallic chemistry (Caballero *et al.*, 2004) and bioinorganic chemistry (Gross & Vahrenkamp, 2005). Acylation of 1,1'-ferrocenebipyrazole with glutaryl dichloride afforded the title compound, (I), which is a bipyrazole having an organometallic substituent (Fig. 1).

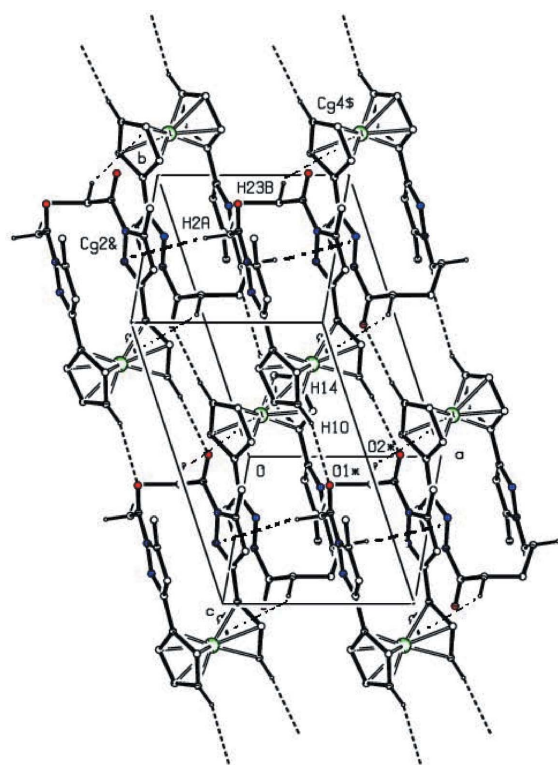


As with *N,N'*-butanedioylbis(5-ferrocenyl-3-methyl-1*H*-pyrazole) (Shi *et al.*, 2005), the bond lengths of each pyrazole ring in (I) are nearly identical and indicate electron delocalization within the rings (Gilchrist, 1997).

The pyrazolyl substituents of the ferrocene are positioned in an eclipsed conformation [dihedral angle  $14.2$  ( $2^\circ$ )], but this feature has little effect on the cyclopentadienyl rings, which are parallel to each other [dihedral angle  $2.8$  ( $2^\circ$ )].


**Figure 1**

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


**Figure 2**

The crystal structure of (I), showing the formation of four-strand and two-strand chains. Atoms labelled with an asterisk (\*), ampersand (&) or dollar sign (\$) are at the symmetry positions  $(x, -1 + y, z)$ ,  $(-1 + x, y, z)$  and  $(2 - x, 2 - y, 1 - z)$ , respectively. Cg2 and Cg4 are the centroids of the rings N3–C20 and C13–C17, respectively. H atoms not involved in the motifs shown have been omitted.

Unlike in other related compounds, which have intermolecular C–H···N hydrogen bonds, intermolecular C–H···O hydrogen bonds are observed in (I) (Table 2). Atoms C10 and C14 in the substituted cyclopentadienyl ring of the molecule at  $(x, y, z)$  act as hydrogen-bond donors, *via* atoms H10 and H14, to atoms O1 and O2 of two carbonyl groups in

an adjacent molecule at  $(x, -1 + y, z)$  to form a chain along [010]. This chain is linked by a C–H··· $\pi$  hydrogen bond involving atom C23, which acts as a hydrogen-bond donor, and the cyclopentadienyl ring C13–C17 in the molecule at  $(2 - x, 2 - y, 1 - z)$  which acts as a hydrogen-bond acceptor; this leads to a double-chain along [010]. Two such double-chains are linked together as a four-stranded chain along [100] *via* a C–H··· $\pi$  hydrogen bond in which atom C2 acts as a hydrogen-bond donor, *via* atom H2A, to the pyrazole ring N3–C20 in an adjacent molecule at  $(-1 + x, y, z)$ . Such hydrogen bonds have been noted by others (Sylvestre *et al.*, 2005). The combination of the four-strand and two-strand chains generates a three-dimensional network (Fig. 2).

## Experimental

A mixture of  $\text{SOCl}_2$  (6 ml, 82.5 mmol) and glutaric acid (1.32 g, 10 mmol) was refluxed under  $\text{N}_2$  until the solid dissolved. After removal of  $\text{SOCl}_2$ , the residue (glutaryl dichloride) was dissolved in acetone (15 ml). This was added dropwise to a mixture of  $\text{K}_2\text{CO}_3$  (1.5 g, 10.85 mmol) and 1,1'-bis(5-methyl-1H-pyrazol-3-yl)ferrocene (1.95 g, 5.6 mmol) in acetone (30 ml) at ambient temperature. The reaction mixture was stirred overnight. The resulting solid was washed with water, air-dried and purified by chromatography on silica gel, with diethyl ether and  $\text{CH}_2\text{Cl}_2$  (15:1, *v/v*) as eluant, to afford a red solid [m.p. 498.25 K (decomposition); yield, 49%]. Analysis, calculated for  $\text{C}_{23}\text{H}_{22}\text{FeN}_4\text{O}_2$ : C 62.46, H 5.01, N 12.67%; found: C 62.44, H 4.96, N 12.74%. Spectroscopic analysis: IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 1731 (*vs*, C=O); UV [ $\lambda_{\text{max}}$ , nm,  $\epsilon$  ( $\times 10^4$ ), in DMF]: 264.00 (3.77, K band), 358.00 (0.45, R band), 446.00 (0.069, *d-d* band);  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 5.895 (s, 2 H, 2CH), 4.509, 4.375 [s, 4 H, s, 4 H, ( $^2\text{H}$ ,  $^5\text{H}$ ) and ( $^3\text{H}$ ,  $^4\text{H}$ ) of two  $\text{C}_5\text{H}_4$  rings], 3.147–3.145 (*d*, 4 H, 2COCH<sub>2</sub>), 2.492 (*d*, 6 H, 2CH<sub>3</sub>), 1.557–1.548 (*d*, 2 H, CH<sub>2</sub>).

## Crystal data

[ $\text{Fe}(\text{C}_{23}\text{H}_{22}\text{N}_4\text{O}_2)$ ]  
 $M_r = 442.30$   
 Triclinic,  $P\bar{1}$   
 $a = 7.383$  (2) Å  
 $b = 11.402$  (2) Å  
 $c = 13.375$  (2) Å  
 $\alpha = 112.31$  (2)°  
 $\beta = 94.39$  (2)°  
 $\gamma = 108.06$  (2)°

$V = 965.9$  (4) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.521$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.81$  mm<sup>-1</sup>  
 $T = 295$  K  
 Block, red  
 0.23 × 0.16 × 0.12 mm

## Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scan  
 (North *et al.*, 1968)  
 $T_{\text{min}} = 0.836$ ,  $T_{\text{max}} = 0.905$   
 4090 measured reflections

3776 independent reflections  
 2813 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$   
 $\theta_{\text{max}} = 26.0^\circ$   
 3 standard reflections  
 every 200 reflections  
 intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.108$   
 $S = 1.03$   
 3776 reflections  
 273 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.3269P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.30$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

O1—C3	1.212 (4)	C7—C8	1.467 (4)
N1—N2	1.384 (4)	O2—C22	1.201 (5)
N1—C3	1.408 (5)	N3—N4	1.384 (4)
N1—C5	1.384 (4)	N3—C18	1.317 (4)
N2—C7	1.319 (4)	C17—C18	1.470 (4)
C4—C5	1.498 (5)	C18—C19	1.415 (5)
C5—C6	1.348 (5)	C19—C20	1.360 (5)
C6—C7	1.414 (5)	C20—C21	1.491 (5)

**Table 2**

Hydrogen-bond geometry (Å, °).

Cg2 and Cg4 are the centroids of the rings N3—C20 and C13—C17, respectively.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10...O1 <sup>i</sup>	0.93	2.49	3.369 (5)	157
C14—H14...O2 <sup>i</sup>	0.93	2.57	3.494 (5)	173
C2—H2A...Cg2 <sup>ii</sup>	0.97	2.84	3.746 (5)	155
C23—H23B...Cg4 <sup>iii</sup>	0.97	2.82	3.680 (5)	149

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

All H atoms were located in a difference map and then treated as riding atoms, with C—H distances of 0.93 (Cp, pyrazolyl, CH), 0.96 (CH<sub>3</sub>) or 0.97 Å (CH<sub>2</sub>), and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ , or  $1.5U_{\text{eq}}(\text{C})$  for methyl groups.

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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## References

- Caballero, A., Carrión, M. C., Espino, G., Jalón, F. A. & Manzano, B. R. (2004). *Polyhedron*, **23**, 361–371.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Gilchrist, T. L. (1997). *Heterocyclic Chemistry*, 3rd ed., pp. 295–297. London: Addison Wesley Longman Ltd.
- Gross, F. & Vahrenkamp, H. (2005). *Inorg. Chem.* **44**, 4433–4440.
- Hardie, M. J., Kilner, C. A. & Halcrow, M. A. (2004). *Acta Cryst.* **C60**, m177–m179.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Morawitz, T., Lerner, H.-W. & Bolte, M. (2006). *Acta Cryst.* **E62**, m1474–m1476.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Otero, A., Fernández-Baeza, J., Antiñolo, A., Tejada, J. & Lara-Sánchez, A. (2004). *Dalton Trans.* pp. 1499–1510.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Shirin, Z. & Carrano, C. J. (2004). *Polyhedron*, **23**, 239–244.
- Shi, Y.-C., Sui, C.-X. & Cheng, H.-J. (2005). *Acta Cryst.* **E61**, m1563–m1565.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Sylvestre, I., Kilner, C. A. & Halcrow, M. A. (2005). *Acta Cryst.* **C61**, o294–o296.