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1-(2,4-Dinitrophenyl)-5-ferrocenyl-3-methyl-1*H*-pyrazole

V. Stalin Elanchezhian,[a] M. Kandaswamy[a] and M. N. Ponnuswamy[b]*

[a]Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [b]Centre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India
Correspondence e-mail: mnpsy2004@yahoo.com

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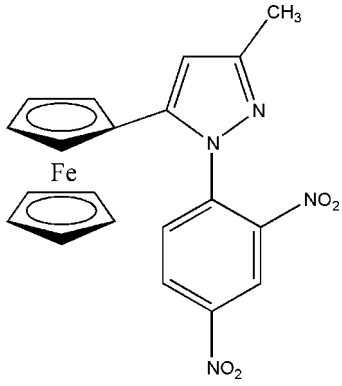
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.107; data-to-parameter ratio = 23.2.

In the title compound, $[Fe(C_5H_5)(C_{15}H_{11}N_4O_4)]$, the dinitrophenyl and cyclopentadienyl rings make dihedral angles of 53.61 (6) and 23.11 (9)°, respectively, with the pyrazole unit. The two cyclopentadienyl rings are in an eclipsed conformation. The crystal structure is stabilized by intermolecular C—H···O interactions, which link molecules into chains parallel to the b axis.

Related literature

For related literature, see: Beer *et al.* (1998); Erasmus *et al.* (1996); Fabbrizzi & Poggi (1995); Gilchrist (1997); Basurto *et al.* (2007); Shi *et al.* (2005).



Experimental

Crystal data

$[Fe(C_5H_5)(C_{15}H_{11}N_4O_4)]$
M_r = 432.22
Triclinic, $P\overline{1}$
a = 7.1073 (2) Å
b = 11.5339 (3) Å
c = 11.7575 (3) Å
α = 103.822 (1)°
β = 93.061 (1)°
γ = 98.822 (2)°
V = 920.76 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 293 (2) K
0.25 × 0.15 × 0.15 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.815, T_{max} = 0.882
24588 measured reflections
6096 independent reflections
4741 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.107
S = 1.01
6096 reflections
263 parameters
10 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O4^{i}	0.93	2.46	3.380 (3)	170

Symmetry code: (i) $x, y+1, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); 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: *SHELXL97* and *PARST* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2122).

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supporting information

Acta Cryst. (2008). E**64**, m265 [doi:10.1107/S1600536807067384]

1-(2,4-Dinitrophenyl)-5-ferrocenyl-3-methyl-1*H*-pyrazole

V. Stalin Elanchezhian, M. Kandaswamy and M. N. Ponnuswamy

S1. Comment

The design of molecular receptors having the ability to selectively bind and sense cationic, anionic or even neutral guests *via* a macroscopic physical response is an area of intense activity (Fabbrizzi *et al.*, 1995). Chemical sensors bearing ferrocene nuclei as part of the sensing unit have been widely studied (Basurto *et al.*, 2007). Ferrocene has largely proved to be a simple and remarkably robust building block for the construction of redox-responsive receptors (Beer *et al.*, 1998). To build a sensor molecule with dual response (redox &optical), the title compound has been synthesized and studied to understand the above said properties.

The pyrazole ring has delocalized bonds (Gilchrist,1997; Shi *et al.*, 2005). The substitued cyclopentadienyl rings are parallel to each other. The cyclopentadienyl rings are oriented at angles of 23.11 (9)° and 23.45 (7)° with respect to the planar pyrazole ring. Furthermore, the pyrazole and dinitrophenyl rings make a dihedral angle of 53.61 (6) °. The nitro groups are planar and oriented at angles of 26.61 (12)° and 9.53 (10)° to the phenyl ring. The two cyclopentadienyl rings of the ferrocenyl group are in a nearly eclipsed conformation (Erasmus *et al.*, 1996).

The molecules in crystal are connected *via* intermolecular C—H···O interaction into a chain extended along the [010] direction.

S2. Experimental

2,4-Dinitrophenylhydrazine (1 g, 5 mmol) was added to a solution of ferrocenoylacetone (1.35 g, 5 mmol) and a catalytic amount of *p*-toluenesulfonic acid in 50 ml of toluene. Tthe mixture was refluxed with a Dean-Stark apparatus for 14 h. The solvent was evaporated under vacuum, and the resulting black residue was chromatographed on a silica-gel column using petroleum ether-ethylacetate (9:1) mixture as eluent. The red band was collected which offered dark-red crystal. (62% yield, m.p. 433–435 K).

S3. Refinement

H atoms were geometrically positioned (C—H = 0.93 - 0.98 Å) and treated as riding on their parent atoms, with Ui_{so}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The cyclopentadienyl group C15—C19 was refined as a rigid group.

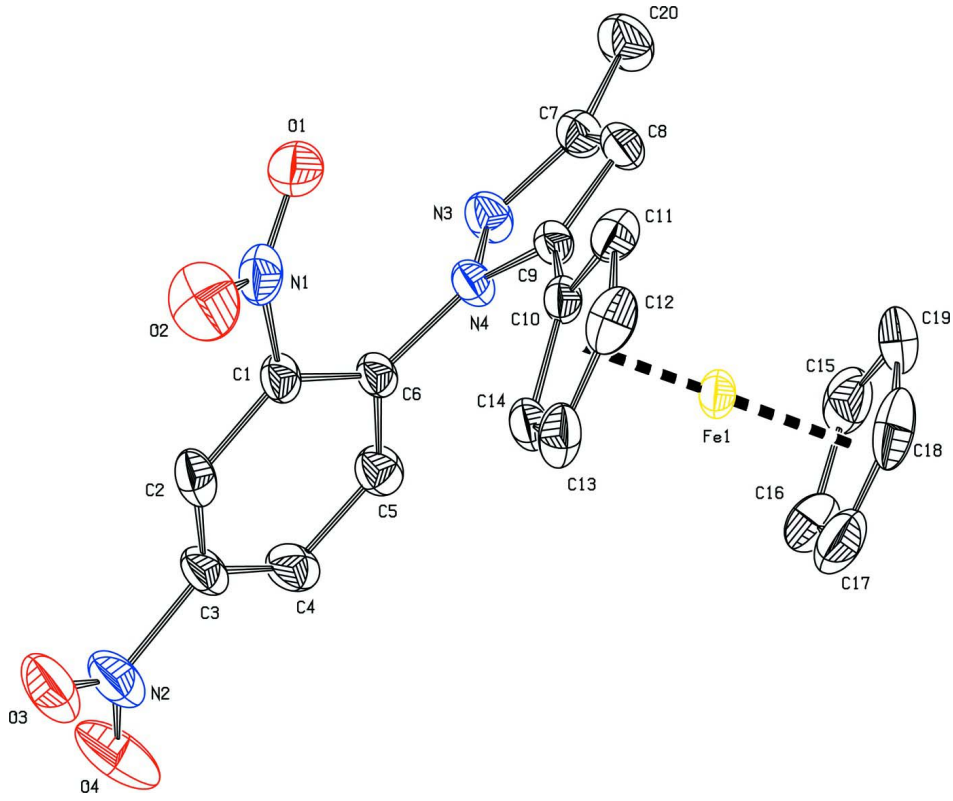

Figure 1

ORTEP plot of the title molecule with displecement ellipsoids drawn at 20% probability level.

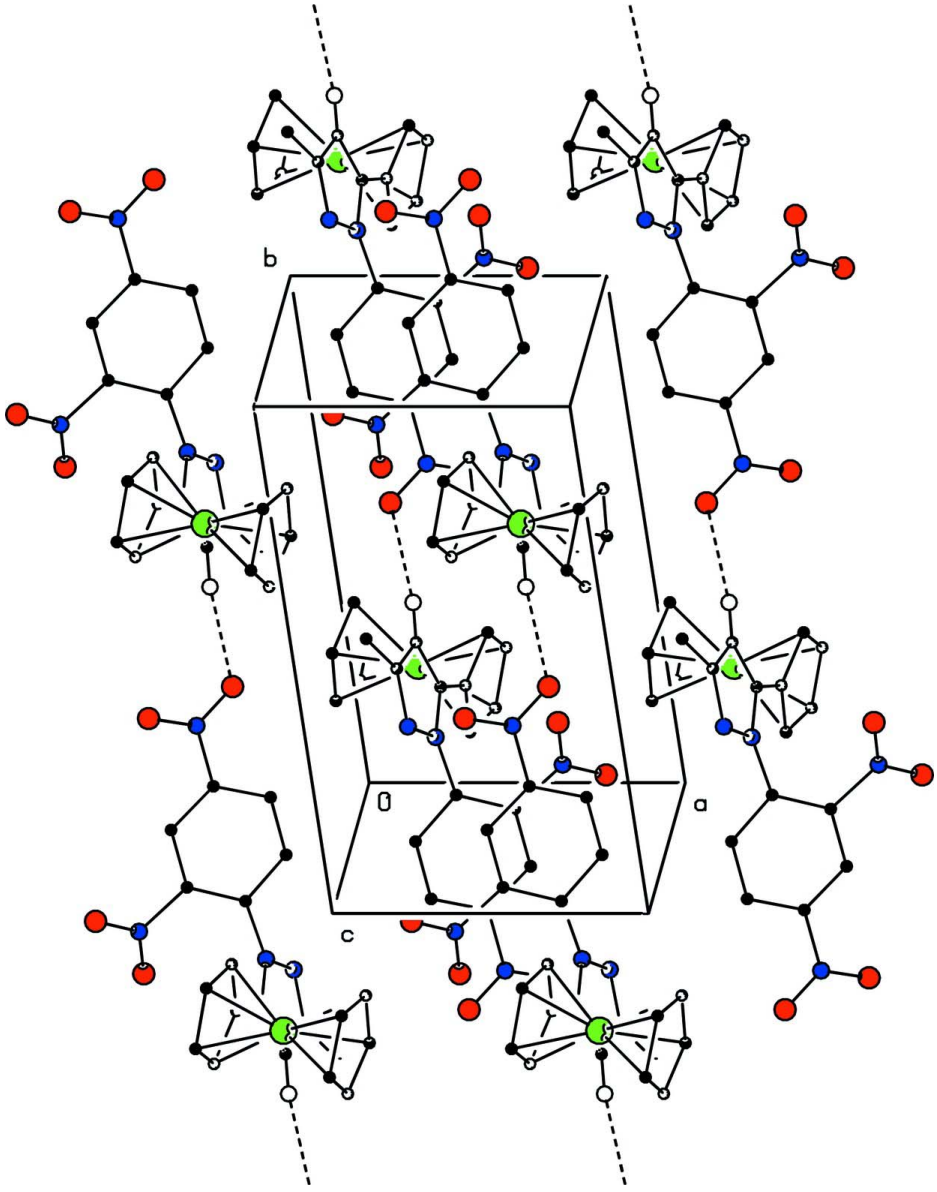

Figure 2

Crystal packing of the title compound viewed down the *c* axis.

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Crystal data

$[Fe(C_5H_5)(C_{15}H_{11}N_4O_4)]$
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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.1073$ (2) Å
$b = 11.5339$ (3) Å
$c = 11.7575$ (3) Å
$\alpha = 103.822$ (1)°
$\beta = 93.061$ (1)°
$\gamma = 98.822$ (2)°
$V = 920.76$ (4) Å^3
$Z = 2$
$F(000) = 444$
$D_x = 1.559$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2637 reflections
$\theta = 2.2$–25°
$\mu = 0.86$ mm^{-1}

T = 293 K
Prism, black

0.25 × 0.15 × 0.15 mm

Data collection

Bruker APEXII Kappa diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.815, T_{max} = 0.882

24588 measured reflections
6096 independent reflections
4741 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 31.5°, θ_{min} = 2.2°
h = −10→10
k = −16→16
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.107
S = 1.01
6096 reflections
263 parameters
10 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.1435P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Experimental. Spectral data: LC Mass:[*M*]+:432(m/e);1H NMR (CDCl3, 400 MHz, \d in p.p.m.): 2.34 (3*H*, s, CH~3~), 4.24(7*H*, s, Fc), 4.13 (2*H*, s, Fc), 7.56 (1*H*, d, Ar, J~0~=8.6 MHz), 8.40 (1*H*, d, Ar, J~0~=8.6 MHz),8.72 (1*H*, s, Ar).
Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5094 (2)	0.03689 (14)	0.34324 (13)	0.0422 (3)
C2	0.5263 (3)	−0.08426 (16)	0.31250 (14)	0.0498 (4)
H2	0.6393	−0.1081	0.2870	0.060*
C3	0.3723 (3)	−0.16830 (14)	0.32047 (14)	0.0512 (4)
C4	0.2039 (3)	−0.13645 (15)	0.35988 (15)	0.0529 (4)
H4	0.1039	−0.1949	0.3688	0.063*
C5	0.1875 (3)	−0.01628 (15)	0.38571 (14)	0.0470 (3)
H5	0.0731	0.0063	0.4102	0.056*
C6	0.3372 (2)	0.07273 (13)	0.37625 (12)	0.0384 (3)
C7	0.2340 (3)	0.35505 (15)	0.50396 (14)	0.0467 (3)
C8	0.2893 (3)	0.37968 (14)	0.39813 (14)	0.0464 (3)
H8	0.2952	0.4530	0.3775	0.056*

C9	0.3328 (2)	0.27444 (13)	0.33134 (12)	0.0386 (3)
C10	0.3901 (2)	0.24686 (13)	0.21167 (12)	0.0391 (3)
C11	0.3667 (3)	0.13264 (15)	0.12710 (14)	0.0483 (4)
H11	0.3100	0.0540	0.1392	0.058*
C12	0.4404 (3)	0.1534 (2)	0.02226 (15)	0.0589 (5)
H12	0.4423	0.0914	−0.0508	0.071*
C13	0.5071 (3)	0.2775 (2)	0.04050 (17)	0.0611 (5)
H13	0.5632	0.3171	−0.0178	0.073*
C14	0.4769 (2)	0.33690 (18)	0.15634 (15)	0.0507 (4)
H14	0.5094	0.4242	0.1923	0.061*
C15	−0.0579 (3)	0.1718 (2)	0.0393 (2)	0.0712 (5)
H15	−0.1129	0.0953	0.0564	0.085*
C16	−0.0325 (3)	0.2853 (2)	0.1168 (2)	0.0706 (5)
H16	−0.0665	0.3020	0.1981	0.085*
C17	0.0510 (4)	0.3715 (2)	0.0615 (3)	0.0817 (6)
H17	0.0835	0.4591	0.0960	0.098*
C18	0.0794 (4)	0.3085 (3)	−0.0561 (2)	0.0858 (6)
H18	0.1335	0.3446	−0.1172	0.103*
C19	0.0095 (3)	0.1847 (3)	−0.0667 (2)	0.0784 (5)
H19	0.0095	0.1186	−0.1369	0.094*
C20	0.1706 (3)	0.44028 (19)	0.60557 (17)	0.0650 (5)
H20A	0.0387	0.4452	0.5892	0.098*
H20B	0.2472	0.5192	0.6178	0.098*
H20C	0.1852	0.4113	0.6750	0.098*
Fe1	0.22444 (3)	0.247896 (19)	0.064950 (18)	0.03941 (8)
N1	0.6829 (2)	0.12430 (16)	0.34359 (15)	0.0571 (4)
N2	0.3891 (4)	−0.29740 (15)	0.28358 (15)	0.0726 (5)
N3	0.2412 (2)	0.24113 (13)	0.50543 (11)	0.0473 (3)
N4	0.3046 (2)	0.19276 (11)	0.39851 (11)	0.0410 (3)
O1	0.7007 (2)	0.22452 (14)	0.40943 (16)	0.0728 (4)
O2	0.8042 (3)	0.08766 (19)	0.28032 (19)	0.0951 (6)
O3	0.5291 (3)	−0.32384 (15)	0.23593 (15)	0.0900 (6)
O4	0.2588 (4)	−0.37005 (15)	0.3009 (2)	0.1185 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0503 (8)	0.0398 (7)	0.0421 (7)	0.0131 (6)	0.0009 (6)	0.0189 (6)
C2	0.0665 (10)	0.0494 (9)	0.0442 (8)	0.0288 (8)	0.0083 (7)	0.0198 (7)
C3	0.0860 (13)	0.0318 (7)	0.0396 (7)	0.0181 (8)	−0.0010 (7)	0.0129 (6)
C4	0.0735 (12)	0.0377 (8)	0.0477 (8)	0.0022 (8)	0.0003 (8)	0.0169 (6)
C5	0.0546 (9)	0.0441 (8)	0.0456 (8)	0.0109 (7)	0.0064 (7)	0.0156 (6)
C6	0.0527 (8)	0.0322 (6)	0.0334 (6)	0.0123 (6)	0.0002 (5)	0.0119 (5)
C7	0.0562 (9)	0.0412 (8)	0.0423 (7)	0.0166 (7)	0.0008 (6)	0.0055 (6)
C8	0.0620 (10)	0.0333 (7)	0.0458 (8)	0.0162 (7)	0.0000 (7)	0.0094 (6)
C9	0.0474 (8)	0.0330 (6)	0.0374 (6)	0.0113 (6)	−0.0019 (5)	0.0113 (5)
C10	0.0429 (7)	0.0403 (7)	0.0376 (6)	0.0151 (6)	−0.0008 (5)	0.0127 (5)
C11	0.0639 (10)	0.0451 (8)	0.0408 (7)	0.0264 (7)	−0.0015 (7)	0.0108 (6)

C12	0.0645 (11)	0.0810 (13)	0.0396 (8)	0.0435 (10)	0.0051 (7)	0.0120 (8)
C13	0.0450 (9)	0.0962 (16)	0.0514 (9)	0.0185 (9)	0.0093 (7)	0.0315 (10)
C14	0.0434 (8)	0.0590 (10)	0.0516 (9)	0.0024 (7)	−0.0028 (7)	0.0232 (8)
C15	0.0454 (10)	0.0815 (10)	0.0814 (11)	0.0065 (9)	−0.0104 (9)	0.0163 (9)
C16	0.0478 (10)	0.0945 (13)	0.0715 (10)	0.0339 (10)	0.0015 (8)	0.0117 (8)
C17	0.0743 (14)	0.0694 (10)	0.1093 (14)	0.0405 (10)	−0.0153 (11)	0.0251 (9)
C18	0.0756 (14)	0.1228 (15)	0.0822 (11)	0.0391 (12)	−0.0092 (10)	0.0602 (12)
C19	0.0677 (13)	0.1053 (12)	0.0567 (8)	0.0298 (11)	−0.0220 (8)	0.0065 (9)
C20	0.0869 (14)	0.0567 (11)	0.0522 (10)	0.0314 (10)	0.0096 (9)	0.0020 (8)
Fe1	0.03942 (12)	0.04333 (13)	0.03942 (12)	0.01542 (9)	−0.00190 (8)	0.01395 (9)
N1	0.0497 (8)	0.0612 (10)	0.0707 (10)	0.0128 (7)	0.0001 (7)	0.0357 (8)
N2	0.1285 (18)	0.0403 (8)	0.0541 (9)	0.0290 (10)	−0.0004 (10)	0.0142 (7)
N3	0.0641 (9)	0.0435 (7)	0.0376 (6)	0.0190 (6)	0.0080 (6)	0.0094 (5)
N4	0.0573 (8)	0.0325 (6)	0.0365 (6)	0.0155 (5)	0.0044 (5)	0.0102 (5)
O1	0.0628 (9)	0.0566 (8)	0.0960 (11)	−0.0018 (7)	−0.0147 (8)	0.0257 (8)
O2	0.0702 (10)	0.1045 (14)	0.1224 (16)	0.0192 (10)	0.0402 (11)	0.0418 (12)
O3	0.1459 (17)	0.0619 (10)	0.0723 (10)	0.0583 (11)	0.0112 (10)	0.0105 (8)
O4	0.177 (2)	0.0373 (8)	0.147 (2)	0.0134 (11)	0.0416 (17)	0.0319 (10)

Geometric parameters (Å, º)

C1—C2	1.383 (2)	C13—C14	1.414 (3)
C1—C6	1.396 (2)	C13—Fe1	2.0328 (18)
C1—N1	1.468 (2)	C13—H13	0.9800
C2—C3	1.369 (3)	C14—Fe1	2.0325 (17)
C2—H2	0.9300	C14—H14	0.9800
C3—C4	1.376 (3)	C15—C16	1.384 (3)
C3—N2	1.474 (2)	C15—C19	1.391 (4)
C4—C5	1.371 (2)	C15—Fe1	2.039 (2)
C4—H4	0.9300	C15—H15	0.9800
C5—C6	1.389 (2)	C16—C17	1.390 (4)
C5—H5	0.9300	C16—Fe1	2.031 (2)
C6—N4	1.4036 (18)	C16—H16	0.9800
C7—N3	1.327 (2)	C17—C18	1.441 (4)
C7—C8	1.404 (2)	C17—Fe1	2.0285 (19)
C7—C20	1.493 (2)	C17—H17	0.9800
C8—C9	1.368 (2)	C18—C19	1.411 (4)
C8—H8	0.9300	C18—Fe1	2.0290 (18)
C9—N4	1.3658 (18)	C18—H18	0.9800
C9—C10	1.461 (2)	C19—Fe1	2.032 (2)
C10—C11	1.429 (2)	C19—H19	0.9800
C10—C14	1.432 (2)	C20—H20A	0.9600
C10—Fe1	2.0394 (13)	C20—H20B	0.9600
C11—C12	1.422 (2)	C20—H20C	0.9600
C11—Fe1	2.0299 (15)	N1—O1	1.212 (2)
C11—H11	0.9800	N1—O2	1.227 (2)
C12—C13	1.398 (3)	N2—O4	1.210 (3)
C12—Fe1	2.0318 (17)	N2—O3	1.212 (3)

C12—H12	0.9800	N3—N4	1.3775 (18)
C2—C1—C6	121.03 (15)	Fe1—C17—H17	126.3
C2—C1—N1	116.47 (15)	C19—C18—C17	106.3 (2)
C6—C1—N1	122.45 (14)	C19—C18—Fe1	69.78 (11)
C3—C2—C1	118.33 (16)	C17—C18—Fe1	69.17 (11)
C3—C2—H2	120.8	C19—C18—H18	126.9
C1—C2—H2	120.8	C17—C18—H18	126.9
C2—C3—C4	122.51 (15)	Fe1—C18—H18	126.9
C2—C3—N2	117.97 (18)	C15—C19—C18	108.7 (2)
C4—C3—N2	119.51 (18)	C15—C19—Fe1	70.27 (12)
C5—C4—C3	118.24 (16)	C18—C19—Fe1	69.55 (12)
C5—C4—H4	120.9	C15—C19—H19	125.7
C3—C4—H4	120.9	C18—C19—H19	125.7
C4—C5—C6	121.72 (17)	Fe1—C19—H19	125.7
C4—C5—H5	119.1	C7—C20—H20A	109.5
C6—C5—H5	119.1	C7—C20—H20B	109.5
C5—C6—C1	117.96 (14)	H20A—C20—H20B	109.5
C5—C6—N4	117.92 (14)	C7—C20—H20C	109.5
C1—C6—N4	124.11 (14)	H20A—C20—H20C	109.5
N3—C7—C8	111.62 (14)	H20B—C20—H20C	109.5
N3—C7—C20	121.14 (16)	C17—Fe1—C18	41.62 (11)
C8—C7—C20	127.23 (16)	C17—Fe1—C11	159.40 (10)
C9—C8—C7	106.43 (14)	C18—Fe1—C11	156.84 (10)
C9—C8—H8	126.8	C17—Fe1—C16	40.04 (11)
C7—C8—H8	126.8	C18—Fe1—C16	68.37 (10)
N4—C9—C8	105.52 (13)	C11—Fe1—C16	123.17 (9)
N4—C9—C10	124.59 (13)	C17—Fe1—C12	158.55 (11)
C8—C9—C10	129.85 (14)	C18—Fe1—C12	121.65 (10)
C11—C10—C14	107.16 (14)	C11—Fe1—C12	40.99 (7)
C11—C10—C9	129.07 (14)	C16—Fe1—C12	159.78 (10)
C14—C10—C9	123.73 (14)	C17—Fe1—C19	68.40 (11)
C11—C10—Fe1	69.09 (8)	C18—Fe1—C19	40.67 (11)
C14—C10—Fe1	69.15 (8)	C11—Fe1—C19	121.03 (10)
C9—C10—Fe1	124.97 (10)	C16—Fe1—C19	67.29 (10)
C12—C11—C10	107.78 (16)	C12—Fe1—C19	107.79 (9)
C12—C11—Fe1	69.58 (9)	C17—Fe1—C14	107.97 (10)
C10—C11—Fe1	69.80 (8)	C18—Fe1—C14	124.35 (10)
C12—C11—H11	126.1	C11—Fe1—C14	69.05 (7)
C10—C11—H11	126.1	C16—Fe1—C14	122.61 (9)
Fe1—C11—H11	126.1	C12—Fe1—C14	68.44 (8)
C13—C12—C11	108.44 (16)	C19—Fe1—C14	161.20 (10)
C13—C12—Fe1	69.92 (10)	C17—Fe1—C13	123.33 (11)
C11—C12—Fe1	69.43 (9)	C18—Fe1—C13	108.12 (10)
C13—C12—H12	125.8	C11—Fe1—C13	68.55 (8)
C11—C12—H12	125.8	C16—Fe1—C13	158.67 (11)
Fe1—C12—H12	125.8	C12—Fe1—C13	40.23 (9)
C12—C13—C14	108.75 (16)	C19—Fe1—C13	124.66 (9)

C12—C13—Fe1	69.85 (11)	C14—Fe1—C13	40.72 (8)
C14—C13—Fe1	69.63 (10)	C17—Fe1—C15	67.57 (11)
C12—C13—H13	125.6	C18—Fe1—C15	68.07 (11)
C14—C13—H13	125.6	C11—Fe1—C15	106.92 (9)
Fe1—C13—H13	125.6	C16—Fe1—C15	39.76 (10)
C13—C14—C10	107.86 (17)	C12—Fe1—C15	123.95 (10)
C13—C14—Fe1	69.65 (11)	C19—Fe1—C15	39.96 (10)
C10—C14—Fe1	69.66 (9)	C14—Fe1—C15	157.41 (9)
C13—C14—H14	126.1	C13—Fe1—C15	160.32 (10)
C10—C14—H14	126.1	C17—Fe1—C10	123.26 (9)
Fe1—C14—H14	126.1	C18—Fe1—C10	161.03 (10)
C16—C15—C19	108.5 (2)	C11—Fe1—C10	41.11 (6)
C16—C15—Fe1	69.83 (12)	C16—Fe1—C10	107.29 (8)
C19—C15—Fe1	69.77 (13)	C12—Fe1—C10	68.91 (6)
C16—C15—H15	125.8	C19—Fe1—C10	156.41 (10)
C19—C15—H15	125.8	C14—Fe1—C10	41.19 (6)
Fe1—C15—H15	125.8	C13—Fe1—C10	68.82 (7)
C15—C16—C17	109.2 (2)	C15—Fe1—C10	121.20 (8)
C15—C16—Fe1	70.41 (12)	O1—N1—O2	124.75 (19)
C17—C16—Fe1	69.88 (13)	O1—N1—C1	118.46 (17)
C15—C16—H16	125.4	O2—N1—C1	116.70 (18)
C17—C16—H16	125.4	O4—N2—O3	124.54 (19)
Fe1—C16—H16	125.4	O4—N2—C3	117.2 (2)
C16—C17—C18	107.3 (2)	O3—N2—C3	118.2 (2)
C16—C17—Fe1	70.08 (11)	C7—N3—N4	103.94 (12)
C18—C17—Fe1	69.21 (12)	C9—N4—N3	112.48 (11)
C16—C17—H17	126.3	C9—N4—C6	129.99 (12)
C18—C17—H17	126.3	N3—N4—C6	117.48 (11)

C6—C1—C2—C3	−3.0 (2)	C13—C12—Fe1—C18	80.62 (15)
N1—C1—C2—C3	174.66 (14)	C11—C12—Fe1—C18	−159.58 (13)
C1—C2—C3—C4	−1.3 (2)	C13—C12—Fe1—C11	−119.80 (16)
C1—C2—C3—N2	177.89 (14)	C13—C12—Fe1—C16	−164.5 (2)
C2—C3—C4—C5	3.7 (2)	C11—C12—Fe1—C16	−44.7 (3)
N2—C3—C4—C5	−175.45 (15)	C13—C12—Fe1—C19	123.06 (13)
C3—C4—C5—C6	−1.9 (2)	C11—C12—Fe1—C19	−117.14 (13)
C4—C5—C6—C1	−2.2 (2)	C13—C12—Fe1—C14	−37.36 (10)
C4—C5—C6—N4	176.74 (14)	C11—C12—Fe1—C14	82.44 (12)
C2—C1—C6—C5	4.7 (2)	C11—C12—Fe1—C13	119.80 (16)
N1—C1—C6—C5	−172.86 (14)	C13—C12—Fe1—C15	164.08 (12)
C2—C1—C6—N4	−174.17 (13)	C11—C12—Fe1—C15	−76.12 (14)
N1—C1—C6—N4	8.3 (2)	C13—C12—Fe1—C10	−81.74 (11)
N3—C7—C8—C9	0.0 (2)	C11—C12—Fe1—C10	38.06 (10)
C20—C7—C8—C9	−179.23 (18)	C15—C19—Fe1—C17	−80.42 (17)
C7—C8—C9—N4	−0.63 (18)	C18—C19—Fe1—C17	39.38 (16)
C7—C8—C9—C10	177.00 (16)	C15—C19—Fe1—C18	−119.8 (2)
N4—C9—C10—C11	23.5 (2)	C15—C19—Fe1—C11	79.09 (17)
C8—C9—C10—C11	−153.74 (17)	C18—C19—Fe1—C11	−161.12 (15)

N4—C9—C10—C14	−159.30 (15)	C15—C19—Fe1—C16	−37.04 (15)
C8—C9—C10—C14	23.5 (3)	C18—C19—Fe1—C16	82.75 (17)
N4—C9—C10—Fe1	113.88 (15)	C15—C19—Fe1—C12	122.03 (15)
C8—C9—C10—Fe1	−63.4 (2)	C18—C19—Fe1—C12	−118.18 (16)
C14—C10—C11—C12	0.56 (18)	C15—C19—Fe1—C14	−162.7 (2)
C9—C10—C11—C12	178.14 (15)	C18—C19—Fe1—C14	−42.9 (4)
Fe1—C10—C11—C12	59.44 (11)	C15—C19—Fe1—C13	163.18 (15)
C14—C10—C11—Fe1	−58.88 (11)	C18—C19—Fe1—C13	−77.02 (18)
C9—C10—C11—Fe1	118.70 (15)	C18—C19—Fe1—C15	119.8 (2)
C10—C11—C12—C13	−0.36 (19)	C15—C19—Fe1—C10	44.1 (3)
Fe1—C11—C12—C13	59.22 (12)	C18—C19—Fe1—C10	163.93 (18)
C10—C11—C12—Fe1	−59.58 (11)	C13—C14—Fe1—C17	−120.57 (14)
C11—C12—C13—C14	0.0 (2)	C10—C14—Fe1—C17	120.32 (13)
Fe1—C12—C13—C14	58.93 (12)	C13—C14—Fe1—C18	−77.49 (16)
C11—C12—C13—Fe1	−58.92 (12)	C10—C14—Fe1—C18	163.41 (12)
C12—C13—C14—C10	0.3 (2)	C13—C14—Fe1—C11	81.06 (13)
Fe1—C13—C14—C10	59.40 (11)	C10—C14—Fe1—C11	−38.04 (9)
C12—C13—C14—Fe1	−59.07 (13)	C13—C14—Fe1—C16	−162.16 (13)
C11—C10—C14—C13	−0.55 (18)	C10—C14—Fe1—C16	78.74 (13)
C9—C10—C14—C13	−178.29 (14)	C13—C14—Fe1—C12	36.93 (12)
Fe1—C10—C14—C13	−59.39 (12)	C10—C14—Fe1—C12	−82.17 (11)
C11—C10—C14—Fe1	58.84 (11)	C13—C14—Fe1—C19	−45.0 (3)
C9—C10—C14—Fe1	−118.89 (14)	C10—C14—Fe1—C19	−164.1 (3)
C19—C15—C16—C17	0.0 (2)	C10—C14—Fe1—C13	−119.10 (16)
Fe1—C15—C16—C17	59.27 (15)	C13—C14—Fe1—C15	164.8 (2)
C19—C15—C16—Fe1	−59.27 (15)	C10—C14—Fe1—C15	45.7 (3)
C15—C16—C17—C18	−0.2 (2)	C13—C14—Fe1—C10	119.10 (16)
Fe1—C16—C17—C18	59.42 (14)	C12—C13—Fe1—C17	−161.33 (13)
C15—C16—C17—Fe1	−59.60 (15)	C14—C13—Fe1—C17	78.58 (15)
C16—C17—C18—C19	0.3 (2)	C12—C13—Fe1—C18	−117.91 (13)
Fe1—C17—C18—C19	60.25 (14)	C14—C13—Fe1—C18	122.00 (14)
C16—C17—C18—Fe1	−59.97 (14)	C12—C13—Fe1—C11	37.71 (10)
C16—C15—C19—C18	0.2 (2)	C14—C13—Fe1—C11	−82.39 (11)
Fe1—C15—C19—C18	−59.13 (15)	C12—C13—Fe1—C16	165.3 (2)
C16—C15—C19—Fe1	59.31 (15)	C14—C13—Fe1—C16	45.2 (3)
C17—C18—C19—C15	−0.3 (2)	C14—C13—Fe1—C12	−120.09 (15)
Fe1—C18—C19—C15	59.58 (15)	C12—C13—Fe1—C19	−75.98 (15)
C17—C18—C19—Fe1	−59.85 (14)	C14—C13—Fe1—C19	163.93 (13)
C16—C17—Fe1—C18	118.5 (2)	C12—C13—Fe1—C14	120.09 (15)
C16—C17—Fe1—C11	−41.5 (3)	C12—C13—Fe1—C15	−42.5 (3)
C18—C17—Fe1—C11	−160.0 (2)	C14—C13—Fe1—C15	−162.6 (2)
C18—C17—Fe1—C16	−118.5 (2)	C12—C13—Fe1—C10	81.98 (10)
C16—C17—Fe1—C12	163.7 (2)	C14—C13—Fe1—C10	−38.11 (10)
C18—C17—Fe1—C12	45.2 (3)	C16—C15—Fe1—C17	−36.98 (16)
C16—C17—Fe1—C19	79.97 (16)	C19—C15—Fe1—C17	82.68 (18)
C18—C17—Fe1—C19	−38.50 (16)	C16—C15—Fe1—C18	−82.10 (17)
C16—C17—Fe1—C14	−119.65 (14)	C19—C15—Fe1—C18	37.57 (16)
C18—C17—Fe1—C14	121.88 (16)	C16—C15—Fe1—C11	121.91 (15)

C16—C17—Fe1—C13	−161.88 (13)	C19—C15—Fe1—C11	−118.42 (15)
C18—C17—Fe1—C13	79.65 (18)	C19—C15—Fe1—C16	119.7 (2)
C16—C17—Fe1—C15	36.73 (15)	C16—C15—Fe1—C12	163.64 (13)
C18—C17—Fe1—C15	−81.74 (17)	C19—C15—Fe1—C12	−76.69 (17)
C16—C17—Fe1—C10	−76.82 (16)	C16—C15—Fe1—C19	−119.7 (2)
C18—C17—Fe1—C10	164.72 (14)	C16—C15—Fe1—C14	45.9 (3)
C19—C18—Fe1—C17	−117.4 (2)	C19—C15—Fe1—C14	165.6 (2)
C19—C18—Fe1—C11	44.8 (3)	C16—C15—Fe1—C13	−164.6 (2)
C17—C18—Fe1—C11	162.2 (2)	C19—C15—Fe1—C13	−45.0 (3)
C19—C18—Fe1—C16	−79.89 (16)	C16—C15—Fe1—C10	79.35 (16)
C17—C18—Fe1—C16	37.47 (16)	C19—C15—Fe1—C10	−160.98 (14)
C19—C18—Fe1—C12	80.40 (18)	C11—C10—Fe1—C17	161.99 (13)
C17—C18—Fe1—C12	−162.24 (16)	C14—C10—Fe1—C17	−79.09 (15)
C17—C18—Fe1—C19	117.4 (2)	C9—C10—Fe1—C17	38.21 (18)
C19—C18—Fe1—C14	164.58 (14)	C11—C10—Fe1—C18	−165.4 (3)
C17—C18—Fe1—C14	−78.06 (18)	C14—C10—Fe1—C18	−46.5 (3)
C19—C18—Fe1—C13	122.51 (16)	C9—C10—Fe1—C18	70.8 (3)
C17—C18—Fe1—C13	−120.13 (17)	C14—C10—Fe1—C11	118.92 (15)
C19—C18—Fe1—C15	−36.93 (15)	C9—C10—Fe1—C11	−123.78 (18)
C17—C18—Fe1—C15	80.43 (17)	C11—C10—Fe1—C16	120.99 (12)
C19—C18—Fe1—C10	−160.1 (2)	C14—C10—Fe1—C16	−120.09 (13)
C17—C18—Fe1—C10	−42.7 (3)	C9—C10—Fe1—C16	−2.79 (16)
C12—C11—Fe1—C17	−166.3 (3)	C11—C10—Fe1—C12	−37.96 (11)
C10—C11—Fe1—C17	−47.3 (3)	C14—C10—Fe1—C12	80.96 (12)
C12—C11—Fe1—C18	49.0 (3)	C9—C10—Fe1—C12	−161.75 (16)
C10—C11—Fe1—C18	168.0 (2)	C11—C10—Fe1—C19	48.3 (2)
C12—C11—Fe1—C16	163.11 (13)	C14—C10—Fe1—C19	167.2 (2)
C10—C11—Fe1—C16	−77.93 (13)	C9—C10—Fe1—C19	−75.5 (2)
C10—C11—Fe1—C12	118.96 (16)	C11—C10—Fe1—C14	−118.92 (15)
C12—C11—Fe1—C19	81.45 (15)	C9—C10—Fe1—C14	117.30 (17)
C10—C11—Fe1—C19	−159.59 (12)	C11—C10—Fe1—C13	−81.24 (12)
C12—C11—Fe1—C14	−80.84 (13)	C14—C10—Fe1—C13	37.68 (12)
C10—C11—Fe1—C14	38.12 (10)	C9—C10—Fe1—C13	154.98 (15)
C12—C11—Fe1—C13	−37.03 (12)	C11—C10—Fe1—C15	79.82 (13)
C10—C11—Fe1—C13	81.94 (11)	C14—C10—Fe1—C15	−161.26 (12)
C12—C11—Fe1—C15	122.68 (13)	C9—C10—Fe1—C15	−43.96 (16)
C10—C11—Fe1—C15	−118.36 (12)	C2—C1—N1—O1	−151.82 (15)
C12—C11—Fe1—C10	−118.96 (16)	C6—C1—N1—O1	25.8 (2)
C15—C16—Fe1—C17	120.2 (2)	C2—C1—N1—O2	24.8 (2)
C15—C16—Fe1—C18	81.29 (17)	C6—C1—N1—O2	−157.56 (17)
C17—C16—Fe1—C18	−38.91 (16)	C2—C3—N2—O4	173.5 (2)
C15—C16—Fe1—C11	−75.99 (16)	C4—C3—N2—O4	−7.3 (3)
C17—C16—Fe1—C11	163.82 (14)	C2—C3—N2—O3	−8.1 (3)
C15—C16—Fe1—C12	−42.5 (3)	C4—C3—N2—O3	171.12 (17)
C17—C16—Fe1—C12	−162.7 (2)	C8—C7—N3—N4	0.66 (19)
C15—C16—Fe1—C19	37.22 (16)	C20—C7—N3—N4	179.92 (17)
C17—C16—Fe1—C19	−82.97 (17)	C8—C9—N4—N3	1.10 (18)
C15—C16—Fe1—C14	−160.88 (13)	C10—C9—N4—N3	−176.70 (14)

C17—C16—Fe1—C14	78.93 (16)	C8—C9—N4—C6	−176.26 (16)
C15—C16—Fe1—C13	165.8 (2)	C10—C9—N4—C6	5.9 (3)
C17—C16—Fe1—C13	45.6 (3)	C7—N3—N4—C9	−1.10 (18)
C17—C16—Fe1—C15	−120.2 (2)	C7—N3—N4—C6	176.62 (14)
C15—C16—Fe1—C10	−118.31 (14)	C5—C6—N4—C9	−127.16 (17)
C17—C16—Fe1—C10	121.50 (15)	C1—C6—N4—C9	51.7 (2)
C13—C12—Fe1—C17	47.0 (3)	C5—C6—N4—N3	55.59 (19)
C11—C12—Fe1—C17	166.8 (2)	C1—C6—N4—N3	−125.57 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O4^{i}	0.93	2.46	3.380 (3)	170

Symmetry code: (i) *x*, *y*+1, *z*.