Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(3,5-Dimethylbenzene)-1-ferrocenylmethyl-1*H*-1,3-benzimidazole

John F. Gallagher,* Keith Hanlon and Joshua Howarth

School of Chemical Sciences, Dublin City University, Dublin 9, Ireland
Correspondence e-mail: john.gallagher@dcu.ie

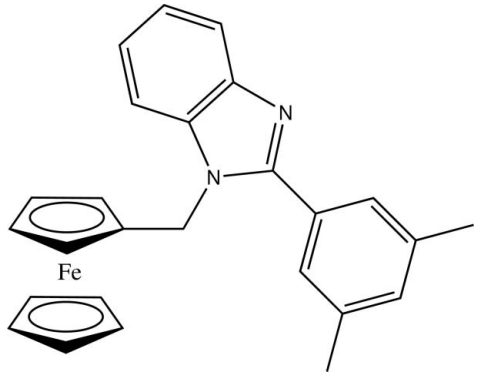
Received 5 October 2007; accepted 9 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.099; data-to-parameter ratio = 18.9.

In the title molecule, $[Fe(C_5H_5)(C_{21}H_{19}N_2)]$, the five-membered imidazole ring forms dihedral angles of 88.61 (8) and 42.15 (6)° with the substituted cyclopentadienyl and dimethyl-substituted benzene rings, respectively. In the crystal structure, there is an N$sp^2\cdots$H contact and a modest C—H$\cdots\pi$(arene) interaction involving the benzene ring of the benzimidizole system.

Related literature

For related ferrocene literature, see: Li *et al.* (1998); Gallagher, Hanlon & Howarth (2001); Gallagher, Hanlon, Howarth & Thomas (2001); Howarth & Hanlon (2001); Kazak *et al.* (2006); Gallagher *et al.* (2007). For the chemical synthesis and crystal structure of $[FcCH_2N(CH_3)_3]^+[I]^-$ see: Pauson *et al.* (1966); Ferguson *et al.* (1994).



Experimental

Crystal data

$[Fe(C_5H_5)(C_{21}H_{19}N_2)]$
M_r = 420.32
Monoclinic, $P2_1/c$
a = 9.6613 (11) Å
b = 10.9314 (8) Å
c = 19.7129 (15) Å
β = 92.194 (8)°
V = 2080.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.74 mm^{-1}
T = 294 (1) K
0.45 × 0.35 × 0.16 mm

Data collection

Bruker $P4$ diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.732, T_{max} = 0.891
6769 measured reflections
5011 independent reflections
4092 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
every 197 reflections
intensity decay: 0.5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.04
5011 reflections
265 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

Cg is the centroid of the C3–C8 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C2—H2$B\cdots$N1^{i}	0.97	2.75	3.658 (2)	156
C22—H22$\cdots Cg^{i}$	0.93	2.91	3.764 (3)	154

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; 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 *PREP8* (Ferguson, 1998).

JFG thanks Dublin City University for the purchase of a Bruker *P*4 diffractometer in 1998.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2528).

References

Bruker (1996). *XSCANS*. Version 2.2. Bruker AXS Inc., Madison, Wisconsin, USA.
Ferguson, G. (1998). *PREP8*. University of Guelph, Canada.
Ferguson, G., Gallagher, J. F., Glidewell, C. & Zakaria, C. M. (1994). *Acta Cryst.* B**50**, 146–150.
Gallagher, J. F., Hanlon, K., Howarth, J. & Thomas, J.-L. (2001). *Acta Cryst.* E**57**, m134–m136.
Gallagher, J. F., Hanlon, K. & Howarth, J. (2001). *Acta Cryst.* C**57**, 1410–1414.
Gallagher, J. F., Hanlon, K. & Howarth, J. (2007). *Acta Cryst.* E**63**, m2649.
Howarth, J. & Hanlon, K. (2001). *Tetrahedron Lett.* **42**, 271–274.
Kazak, C., Yilmaz, V. T., Goker, H. & Kus, C. (2006). *Cryst. Res. Technol.* **5**, 528–532.
Li, P., Scowen, I. J., Davies, J. E. & Halcrow, M. A. (1998). *J. Chem. Soc. Dalton Trans.* pp. 3791–3799.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Pauson, P. L., Sandhu, M. A. & Watts, W. E. (1966). *J. Chem. Soc. C*, pp. 251–255.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S1600536807049549

supplementary materials

Acta Cryst. (2007). E**63**, m2746 [**doi:10.1107/S1600536807049549**]

2-(3,5-Dimethylbenzene)-1-ferrocenylmethyl-1*H*-1,3-benzimidazole

J. F. Gallagher, K. Hanlon and J. Howarth

Comment

Benzimidazole systems have attracted considerable attention in synthetic and structural as well as in applied biological research (Gallagher, Hanlon & Howarth, 2001; Howarth & Hanlon, 2001; (Gallagher, Hanlon, Howarth & Thomas, 2001). The title compound (I) (Figs. 1–2) is obtained from a series of reactions involving synthesis of *N*-Ferrocenylmethyl-2-(3,5-dimethylbenzene)benzimidazole from 2-(3,5-dimethylbenzene)benzimidazole and (trimethylammonium)ferrocenylmethyl iodide.

Bond lengths and angles in the title compound 1-Ferrocenylmethyl-2-(3,5-dimethylbenzene)-1*H*-1,3-benzimidazole are normal and similar to previously reported ferrocene systems. Of note is the fact that the five-membered imidazole ring forms dihedral angles of 88.61 (8)° and 42.15 (6)° with the substituted C_5H_4 and 3,4-dimethyl substituted benzene rings, respectively. The former is similar to the 71.48 (10)° observed in a related cationic derivative (Gallagher *et al.*, 2007) and 84.37 (9)° in the neutral 3-chlorophenyl derivative (Gallagher, Hanlon, Howarth & Thomas, 2001). Similar data of 78.07 (8)° and 73.86 (8)° are observed in both a methoxy and dimethoxy derivative (Gallagher, Hanlon & Howarth, 2001). These data highlight that the most important dihedral angle in these systems is defined by the imidazole/C_5H_4 angle which is remarkably invariant in a series of closely related systems. This is due to steric constraints at the hinge atom C2, between the 5-membered imidazole ring and the substituted C_5H_4 ring which does not facilitate for opening or closing of the rings attached at C2. A wide range of angles between the imidazole and C_6 aromatic rings in these structures is due to a lack of steric hindrance and crystal packing forces about the C—C bond.

Of interest is the fact that there are no strong intermolecular interactions in the crystal structure and the optimal acceptor N1 only has a closest H2B atom at a distance of 2.75 Å. A weak C22—H22···Cg^{i} is also present with a C···Cg^{i} of 3.764 (3) Å, where *Cg* is the ring centroid of the C3–C8 ring and the symmetry operation i = 1 − *x*, *y* + 1/2, −*z* + 1/2.

Examination of the structure with *PLATON* (Spek, 2003) showed that there were no solvent accessible voids in the crystal lattice unlike the cationic 3-methylstyryl derivative where the hexafluorophosphate anion is located in a void and adopts four orientations each with equal 0.25 site occupancy (Gallagher *et al.*, 2007).

Experimental

Synthesis of the neutral starting material *N*-Ferrocenylmethyl-2-(3,5-dimethylbenzene)benzimidazole $[(C_5H_5)Fe(C_5H_4)CH_2(C_7H_4N_2)C_6H_4(CH_3)_2]$

To a mixture of 2-(3,5-dimethylbenzene)benzimidazole (4.0 g, 18 mmol) and K_2CO_3 (3.73 g, 27 mmol) in CH_3CN (150 ml) was added (trimethylammonium)ferrocenylmethyl iodide ($[FcCH_2N(CH_3)_3]^+[I]^-$) (7.29 g, 18 mmol) (Pauson *et al.*, 1966; Ferguson *et al.*, 1994) and the mixture was heated to reflux temperatures for 12 h. The reaction was cooled to room temperature, water was added and the suspension extracted into $CHCl_3$. The organic layer was washed with water, dried

($MgSO_4$) and evaporated under vacuum to leave a brown gum. The crude product was purified by column chromatography on silica gel using CH_2Cl_2:CH_3OH (97:3) as eluent.

Yield 5.3 g (70%), m.p. 407–411 K (uncorrected). Compound (I) was obtained as a light orange solid. IR (KBr, ν cm^{-1}) (>1500 cm^{-1}): 3017, 2399, 1709, 1641.

^{1}H NMR [400 MHz, δH (p.p.m.), $CDCl_3$], 7.65 (m, 2H, benz-H), 7.43 (m, 2H, aryl-H), 7.27–7.22 (m, 3H, benz-H + aryl-H), 5.32 (s, 2H, Fc—CH_2), 4.09 (s, 5H, cpd-H), 4.03 (m, 2H, cpd-H), 4.01 (m, 2H, cpd-H), 2.42 (s, 6H, 2 *x* CH_3). ^{13}C NMR [δC, $CDCl_3$], 153.36, 142.82, 138.26, 135.77, 131.45, 130.79, 127.41, 122.66, 122.27, 119.35, 111.48, 83.74, 68.97, 68.87, 68.12, 43.84, 21.26.

Refinement

In the refinement, all H atoms were allowed for as riding atoms with C—H distances of 0.93 Å, 0.96 Å and 0.97 Å for the aromatic, methyl and methylene C—H using the *SHELXL97* (Sheldrick, 1997) defaults (at 294 K).

Figures

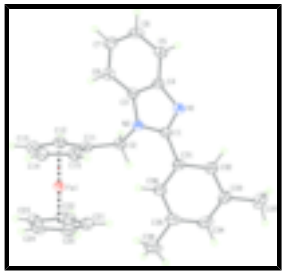


Fig. 1. The molecular structure with the atomic numbering scheme: displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. A stereoview of the primary C22—H22···π(C_6H_4) interaction in the crystal structure with atoms drawn as their van der Waals spheres. The H···N contact can be discerned in the middle of the diagram.

2-(3,5-Dimethylbenzene)-1-ferrocenylmethyl-1*H*-1,3-benzimidazole

Crystal data

$[Fe(C_5H_5)(C_{21}H_{19}N_2)]$	F_{000} = 880
M_r = 420.32	D_x = 1.342 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 410 K
Hall symbol: -p 2ybc	Mo *K*α radiation λ = 0.71073 Å
a = 9.6613 (11) Å	Cell parameters from 67 reflections
b = 10.9314 (8) Å	θ = 2.1–17.5º
c = 19.7129 (15) Å	μ = 0.74 mm^{-1}
β = 92.194 (8)º	*T* = 294 (1) K

V = 2080.4 (3) Å^3
Z = 4
Block, red
0.45 × 0.35 × 0.16 mm

Data collection

Bruker P4 diffractometer
Radiation source: X-ray tube
Monochromator: graphite
T = 294(1) K
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.732, T_{max} = 0.891
6769 measured reflections
5011 independent reflections
4092 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.0º
θ_{min} = 2.1º
h = −1→12
k = −14→1
l = −26→26
3 standard reflections
every 197 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.04
5011 reflections
265 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.6685P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0028 (5)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.16527 (3)	0.02092 (2)	0.203406 (12)	0.03664 (9)
N1	0.31594 (17)	−0.54213 (14)	0.24456 (8)	0.0414 (3)
N2	0.33107 (15)	−0.33696 (13)	0.24774 (7)	0.0358 (3)
C1	0.31443 (18)	−0.44048 (16)	0.20884 (8)	0.0360 (3)
C2	0.36082 (19)	−0.21155 (16)	0.22604 (9)	0.0390 (4)
C3	0.34290 (17)	−0.37604 (17)	0.31466 (8)	0.0360 (4)
C4	0.33363 (19)	−0.50394 (17)	0.31142 (9)	0.0399 (4)
C5	0.3399 (2)	−0.5723 (2)	0.37127 (10)	0.0517 (5)
C6	0.3543 (3)	−0.5101 (2)	0.43183 (10)	0.0564 (5)
C7	0.3624 (2)	−0.3834 (2)	0.43415 (10)	0.0526 (5)
C8	0.3573 (2)	−0.31273 (19)	0.37569 (9)	0.0441 (4)
C11	0.26040 (19)	−0.12095 (17)	0.25347 (9)	0.0394 (4)

C12	0.2969 (2)	−0.00784 (19)	0.28531 (10)	0.0505 (5)
C13	0.1730 (3)	0.0495 (2)	0.30552 (11)	0.0604 (6)
C14	0.0614 (3)	−0.0275 (2)	0.28735 (11)	0.0588 (6)
C15	0.1143 (2)	−0.13241 (19)	0.25545 (10)	0.0485 (5)
C21	0.2002 (3)	0.0058 (3)	0.10306 (11)	0.0700 (8)
C22	0.2672 (3)	0.1103 (3)	0.13086 (14)	0.0790 (9)
C23	0.1620 (3)	0.1873 (2)	0.15712 (12)	0.0642 (6)
C24	0.0383 (3)	0.1286 (2)	0.14448 (11)	0.0613 (6)
C25	0.0600 (3)	0.0194 (3)	0.11158 (12)	0.0682 (7)
C31	0.28576 (19)	−0.44061 (17)	0.13477 (8)	0.0393 (4)
C32	0.3423 (2)	−0.53307 (18)	0.09602 (10)	0.0462 (4)
C33	0.3087 (3)	−0.5439 (2)	0.02729 (10)	0.0547 (5)
C34	0.2157 (3)	−0.4606 (2)	−0.00208 (10)	0.0588 (6)
C35	0.1558 (2)	−0.36883 (19)	0.03505 (10)	0.0515 (5)
C36	0.1925 (2)	−0.35852 (18)	0.10382 (9)	0.0448 (4)
C37	0.3691 (4)	−0.6461 (3)	−0.01382 (13)	0.0796 (8)
C38	0.0499 (3)	−0.2837 (2)	0.00229 (13)	0.0761 (8)
H2A	0.3565	−0.2078	0.1768	0.047*
H2B	0.4541	−0.1896	0.2415	0.047*
H5	0.3346	−0.6573	0.3703	0.062*
H6	0.3587	−0.5540	0.4722	0.068*
H7	0.3715	−0.3448	0.4761	0.063*
H8	0.3633	−0.2279	0.3772	0.053*
H12	0.3861	0.0230	0.2917	0.061*
H13	0.1669	0.1250	0.3270	0.072*
H14	−0.0314	−0.0122	0.2950	0.071*
H15	0.0622	−0.1979	0.2386	0.058*
H21	0.2429	−0.0602	0.0827	0.084*
H22	0.3618	0.1260	0.1319	0.095*
H23	0.1751	0.2625	0.1786	0.077*
H24	−0.0477	0.1582	0.1564	0.074*
H25	−0.0084	−0.0359	0.0975	0.082*
H32	0.4037	−0.5885	0.1166	0.055*
H34	0.1931	−0.4669	−0.0482	0.071*
H36	0.1545	−0.2963	0.1294	0.054*
H37A	0.4677	−0.6484	−0.0058	0.119*
H37B	0.3484	−0.6321	−0.0612	0.119*
H37C	0.3297	−0.7226	−0.0006	0.119*
H38A	0.0932	−0.2072	−0.0080	0.114*
H38B	−0.0235	−0.2699	0.0329	0.114*
H38C	0.0126	−0.3199	−0.0388	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.03931 (15)	0.03539 (14)	0.03527 (14)	0.00490 (10)	0.00200 (10)	0.00486 (10)
N1	0.0516 (9)	0.0367 (8)	0.0355 (7)	0.0032 (7)	−0.0013 (6)	0.0051 (6)
N2	0.0406 (7)	0.0346 (7)	0.0321 (7)	0.0015 (6)	−0.0010 (6)	0.0063 (6)

C1	0.0371 (8)	0.0359 (8)	0.0348 (8)	0.0029 (7)	−0.0007 (7)	0.0043 (7)
C2	0.0404 (9)	0.0358 (9)	0.0407 (9)	0.0012 (7)	0.0017 (7)	0.0078 (7)
C3	0.0349 (8)	0.0407 (9)	0.0323 (8)	0.0036 (7)	−0.0005 (6)	0.0050 (7)
C4	0.0434 (9)	0.0417 (10)	0.0345 (8)	0.0053 (7)	0.0004 (7)	0.0070 (7)
C5	0.0676 (13)	0.0446 (11)	0.0428 (10)	0.0051 (10)	0.0004 (9)	0.0139 (9)
C6	0.0712 (14)	0.0629 (14)	0.0349 (10)	0.0064 (11)	0.0002 (9)	0.0147 (9)
C7	0.0579 (12)	0.0667 (14)	0.0331 (9)	0.0043 (10)	0.0000 (8)	−0.0001 (9)
C8	0.0470 (10)	0.0465 (10)	0.0386 (9)	0.0018 (8)	−0.0004 (8)	0.0000 (8)
C11	0.0456 (10)	0.0374 (9)	0.0350 (8)	0.0032 (7)	−0.0008 (7)	0.0069 (7)
C12	0.0589 (12)	0.0472 (11)	0.0442 (10)	0.0063 (9)	−0.0137 (9)	−0.0034 (8)
C13	0.0853 (17)	0.0573 (13)	0.0382 (10)	0.0212 (12)	−0.0019 (10)	−0.0075 (9)
C14	0.0618 (13)	0.0673 (14)	0.0489 (11)	0.0192 (12)	0.0218 (10)	0.0127 (10)
C15	0.0487 (11)	0.0478 (11)	0.0497 (11)	−0.0006 (9)	0.0133 (8)	0.0125 (9)
C21	0.105 (2)	0.0660 (15)	0.0402 (11)	0.0265 (15)	0.0213 (12)	0.0122 (10)
C22	0.0519 (13)	0.111 (2)	0.0743 (16)	−0.0059 (14)	0.0102 (12)	0.0523 (17)
C23	0.0906 (18)	0.0418 (11)	0.0599 (13)	0.0011 (12)	−0.0008 (12)	0.0163 (10)
C24	0.0628 (14)	0.0664 (15)	0.0543 (12)	0.0223 (12)	−0.0043 (10)	0.0140 (11)
C25	0.0851 (18)	0.0727 (16)	0.0456 (12)	0.0002 (14)	−0.0147 (12)	0.0041 (11)
C31	0.0462 (9)	0.0385 (9)	0.0328 (8)	−0.0047 (8)	−0.0024 (7)	0.0051 (7)
C32	0.0564 (11)	0.0425 (10)	0.0396 (9)	−0.0001 (9)	−0.0015 (8)	0.0030 (8)
C33	0.0766 (15)	0.0470 (12)	0.0404 (10)	−0.0073 (10)	0.0019 (10)	−0.0057 (8)
C34	0.0840 (16)	0.0551 (13)	0.0361 (10)	−0.0136 (11)	−0.0124 (10)	0.0033 (9)
C35	0.0633 (13)	0.0463 (11)	0.0435 (10)	−0.0087 (9)	−0.0147 (9)	0.0114 (9)
C36	0.0523 (11)	0.0426 (10)	0.0390 (9)	−0.0006 (8)	−0.0054 (8)	0.0059 (8)
C37	0.116 (2)	0.0701 (17)	0.0529 (13)	0.0058 (16)	0.0039 (14)	−0.0183 (12)
C38	0.0933 (18)	0.0667 (16)	0.0653 (15)	−0.0021 (14)	−0.0363 (14)	0.0185 (13)

Geometric parameters (Å, °)

Fe1—C11	2.0383 (17)	C31—C32	1.391 (3)
Fe1—C12	2.041 (2)	C31—C36	1.395 (3)
Fe1—C13	2.036 (2)	C32—C33	1.386 (3)
Fe1—C14	2.038 (2)	C33—C34	1.389 (3)
Fe1—C15	2.0353 (19)	C33—C37	1.511 (3)
Fe1—C21	2.026 (2)	C34—C35	1.382 (3)
Fe1—C22	2.019 (2)	C35—C36	1.393 (3)
Fe1—C23	2.035 (2)	C35—C38	1.509 (3)
Fe1—C24	2.032 (2)	C2—H2A	0.9700
Fe1—C25	2.042 (2)	C2—H2B	0.9700
N1—C1	1.315 (2)	C5—H5	0.9300
N1—C4	1.387 (2)	C6—H6	0.9300
N2—C1	1.373 (2)	C7—H7	0.9300
N2—C2	1.468 (2)	C8—H8	0.9300
N2—C3	1.387 (2)	C12—H12	0.9300
C1—C31	1.476 (2)	C13—H13	0.9300
C2—C11	1.502 (3)	C14—H14	0.9300
C3—C8	1.390 (3)	C15—H15	0.9300
C3—C4	1.402 (3)	C21—H21	0.9300
C4—C5	1.396 (2)	C22—H22	0.9300

C5—C6	1.377 (3)	C23—H23	0.9300
C6—C7	1.387 (3)	C24—H24	0.9300
C7—C8	1.387 (3)	C25—H25	0.9300
C11—C15	1.419 (3)	C32—H32	0.9300
C11—C12	1.425 (3)	C34—H34	0.9300
C12—C13	1.422 (3)	C36—H36	0.9300
C13—C14	1.404 (4)	C37—H37A	0.9600
C14—C15	1.413 (3)	C37—H37B	0.9600
C21—C25	1.379 (4)	C37—H37C	0.9600
C21—C22	1.413 (4)	C38—H38A	0.9600
C22—C23	1.432 (4)	C38—H38B	0.9600
C23—C24	1.371 (4)	C38—H38C	0.9600
C24—C25	1.379 (4)		
C22—Fe1—C21	40.88 (12)	C22—C21—Fe1	69.29 (14)
C22—Fe1—C24	67.35 (10)	C21—C22—C23	107.2 (2)
C21—Fe1—C24	66.96 (10)	C21—C22—Fe1	69.83 (13)
C22—Fe1—C23	41.36 (11)	C23—C22—Fe1	69.91 (13)
C21—Fe1—C23	68.61 (11)	C24—C23—C22	106.5 (2)
C24—Fe1—C23	39.40 (10)	C24—C23—Fe1	70.21 (13)
C22—Fe1—C15	152.78 (12)	C22—C23—Fe1	68.73 (13)
C21—Fe1—C15	118.53 (10)	C23—C24—C25	110.1 (2)
C24—Fe1—C15	127.80 (10)	C23—C24—Fe1	70.39 (13)
C23—Fe1—C15	164.23 (10)	C25—C24—Fe1	70.59 (13)
C22—Fe1—C13	128.73 (13)	C24—C25—C21	108.6 (3)
C21—Fe1—C13	167.62 (12)	C24—C25—Fe1	69.85 (13)
C24—Fe1—C13	118.34 (10)	C21—C25—Fe1	69.57 (14)
C23—Fe1—C13	107.78 (10)	C32—C31—C36	119.19 (17)
C15—Fe1—C13	68.19 (10)	C32—C31—C1	118.76 (16)
C22—Fe1—C14	165.82 (13)	C36—C31—C1	121.71 (17)
C21—Fe1—C14	151.31 (13)	C33—C32—C31	121.07 (19)
C24—Fe1—C14	108.04 (10)	C32—C33—C34	118.4 (2)
C23—Fe1—C14	126.65 (10)	C32—C33—C37	120.4 (2)
C15—Fe1—C14	40.58 (9)	C34—C33—C37	121.2 (2)
C13—Fe1—C14	40.31 (11)	C35—C34—C33	122.16 (18)
C22—Fe1—C11	119.21 (10)	C34—C35—C36	118.55 (19)
C21—Fe1—C11	108.78 (9)	C34—C35—C38	120.8 (2)
C24—Fe1—C11	165.60 (9)	C36—C35—C38	120.6 (2)
C23—Fe1—C11	153.67 (10)	C35—C36—C31	120.64 (19)
C15—Fe1—C11	40.77 (8)	N2—C2—H2A	109.2
C13—Fe1—C11	68.86 (8)	C11—C2—H2A	109.2
C14—Fe1—C11	68.65 (8)	N2—C2—H2B	109.2
C22—Fe1—C12	109.20 (10)	C11—C2—H2B	109.2
C21—Fe1—C12	129.62 (11)	H2A—C2—H2B	107.9
C24—Fe1—C12	152.31 (10)	C6—C5—H5	121.1
C23—Fe1—C12	119.40 (10)	C4—C5—H5	121.1
C15—Fe1—C12	68.27 (9)	C5—C6—H6	119.1
C13—Fe1—C12	40.82 (9)	C7—C6—H6	119.1
C14—Fe1—C12	68.21 (10)	C8—C7—H7	119.1
C11—Fe1—C12	40.88 (8)	C6—C7—H7	119.1

C22—Fe1—C25	67.41 (12)	C7—C8—H8	121.9
C21—Fe1—C25	39.63 (12)	C3—C8—H8	121.9
C24—Fe1—C25	39.56 (10)	C13—C12—H12	126.0
C23—Fe1—C25	67.13 (11)	C11—C12—H12	126.0
C15—Fe1—C25	108.55 (10)	Fe1—C12—H12	126.7
C13—Fe1—C25	151.16 (11)	C14—C13—H13	126.0
C14—Fe1—C25	118.28 (12)	C12—C13—H13	126.0
C11—Fe1—C25	128.53 (10)	Fe1—C13—H13	125.9
C12—Fe1—C25	166.90 (10)	C13—C14—H14	125.9
C1—N1—C4	104.65 (15)	C15—C14—H14	125.9
C1—N2—C3	106.31 (14)	Fe1—C14—H14	126.3
C1—N2—C2	128.85 (14)	C14—C15—H15	125.7
C3—N2—C2	123.70 (15)	C11—C15—H15	125.7
N1—C1—N2	113.50 (14)	Fe1—C15—H15	126.3
N1—C1—C31	121.78 (16)	C25—C21—H21	126.2
N2—C1—C31	124.53 (15)	C22—C21—H21	126.2
N2—C2—C11	112.01 (14)	Fe1—C21—H21	125.3
N2—C3—C8	132.19 (18)	C21—C22—H22	126.4
N2—C3—C4	105.12 (15)	C23—C22—H22	126.4
C8—C3—C4	122.67 (17)	Fe1—C22—H22	125.4
N1—C4—C5	129.92 (19)	C24—C23—H23	126.7
N1—C4—C3	110.41 (15)	C22—C23—H23	126.7
C5—C4—C3	119.66 (18)	Fe1—C23—H23	125.9
C6—C5—C4	117.9 (2)	C23—C24—H24	125.0
C5—C6—C7	121.72 (19)	C25—C24—H24	125.0
C8—C7—C6	121.90 (19)	Fe1—C24—H24	125.7
C7—C8—C3	116.17 (19)	C24—C25—H25	125.7
C15—C11—C12	107.10 (18)	C21—C25—H25	125.7
C15—C11—C2	127.50 (18)	Fe1—C25—H25	126.4
C12—C11—C2	125.36 (18)	C33—C32—H32	119.5
C15—C11—Fe1	69.50 (11)	C31—C32—H32	119.5
C12—C11—Fe1	69.66 (11)	C35—C34—H34	118.9
C2—C11—Fe1	127.81 (12)	C33—C34—H34	118.9
C13—C12—C11	108.0 (2)	C35—C36—H36	119.7
C13—C12—Fe1	69.39 (12)	C31—C36—H36	119.7
C11—C12—Fe1	69.46 (11)	C33—C37—H37A	109.5
C14—C13—C12	108.1 (2)	C33—C37—H37B	109.5
C14—C13—Fe1	69.93 (12)	H37A—C37—H37B	109.5
C12—C13—Fe1	69.79 (12)	C33—C37—H37C	109.5
C13—C14—C15	108.3 (2)	H37A—C37—H37C	109.5
C13—C14—Fe1	69.76 (13)	H37B—C37—H37C	109.5
C15—C14—Fe1	69.61 (11)	C35—C38—H38A	109.5
C14—C15—C11	108.5 (2)	C35—C38—H38B	109.5
C14—C15—Fe1	69.80 (12)	H38A—C38—H38B	109.5
C11—C15—Fe1	69.73 (11)	C35—C38—H38C	109.5
C25—C21—C22	107.7 (2)	H38A—C38—H38C	109.5
C25—C21—Fe1	70.80 (14)	H38B—C38—H38C	109.5
C4—N1—C1—N2	0.5 (2)	C13—Fe1—C15—C14	37.31 (15)
C4—N1—C1—C31	−174.80 (16)	C11—Fe1—C15—C14	119.79 (19)

C3—N2—C1—N1	−0.7 (2)	C12—Fe1—C15—C14	81.43 (15)
C2—N2—C1—N1	167.27 (17)	C25—Fe1—C15—C14	−112.11 (16)
C3—N2—C1—C31	174.46 (16)	C22—Fe1—C15—C11	51.6 (3)
C2—N2—C1—C31	−17.6 (3)	C21—Fe1—C15—C11	86.02 (16)
C1—N2—C2—C11	128.17 (18)	C24—Fe1—C15—C11	167.85 (12)
C3—N2—C2—C11	−65.8 (2)	C23—Fe1—C15—C11	−160.6 (3)
C1—N2—C3—C8	−177.85 (19)	C13—Fe1—C15—C11	−82.48 (13)
C2—N2—C3—C8	13.4 (3)	C14—Fe1—C15—C11	−119.79 (19)
C1—N2—C3—C4	0.55 (19)	C12—Fe1—C15—C11	−38.36 (12)
C2—N2—C3—C4	−168.17 (16)	C25—Fe1—C15—C11	128.09 (14)
C1—N1—C4—C5	178.5 (2)	C22—Fe1—C21—C25	−118.4 (2)
C1—N1—C4—C3	−0.1 (2)	C24—Fe1—C21—C25	−36.87 (16)
N2—C3—C4—N1	−0.3 (2)	C23—Fe1—C21—C25	−79.54 (17)
C8—C3—C4—N1	178.30 (16)	C15—Fe1—C21—C25	84.92 (17)
N2—C3—C4—C5	−179.07 (17)	C13—Fe1—C21—C25	−154.8 (4)
C8—C3—C4—C5	−0.5 (3)	C14—Fe1—C21—C25	48.8 (3)
N1—C4—C5—C6	−178.1 (2)	C11—Fe1—C21—C25	128.39 (16)
C3—C4—C5—C6	0.4 (3)	C12—Fe1—C21—C25	169.35 (15)
C4—C5—C6—C7	0.0 (4)	C24—Fe1—C21—C22	81.48 (17)
C5—C6—C7—C8	−0.4 (4)	C23—Fe1—C21—C22	38.81 (16)
C6—C7—C8—C3	0.4 (3)	C15—Fe1—C21—C22	−156.73 (15)
N2—C3—C8—C7	178.26 (19)	C13—Fe1—C21—C22	−36.4 (5)
C4—C3—C8—C7	0.1 (3)	C14—Fe1—C21—C22	167.11 (19)
N2—C2—C11—C15	−44.5 (2)	C11—Fe1—C21—C22	−113.25 (16)
N2—C2—C11—C12	132.75 (18)	C12—Fe1—C21—C22	−72.30 (18)
N2—C2—C11—Fe1	−136.62 (14)	C25—Fe1—C21—C22	118.4 (2)
C22—Fe1—C11—C15	−155.76 (15)	C25—C21—C22—C23	0.5 (3)
C21—Fe1—C11—C15	−112.22 (15)	Fe1—C21—C22—C23	−60.24 (16)
C24—Fe1—C11—C15	−42.0 (4)	C25—C21—C22—Fe1	60.71 (17)
C23—Fe1—C11—C15	168.2 (2)	C24—Fe1—C22—C21	−80.45 (17)
C13—Fe1—C11—C15	80.71 (14)	C23—Fe1—C22—C21	−118.0 (2)
C14—Fe1—C11—C15	37.32 (13)	C15—Fe1—C22—C21	49.4 (3)
C12—Fe1—C11—C15	118.26 (18)	C13—Fe1—C22—C21	170.61 (15)
C25—Fe1—C11—C15	−72.50 (17)	C14—Fe1—C22—C21	−154.1 (4)
C22—Fe1—C11—C12	85.98 (17)	C11—Fe1—C22—C21	85.27 (17)
C21—Fe1—C11—C12	129.52 (15)	C12—Fe1—C22—C21	129.01 (15)
C24—Fe1—C11—C12	−160.2 (3)	C25—Fe1—C22—C21	−37.44 (16)
C23—Fe1—C11—C12	50.0 (2)	C21—Fe1—C22—C23	118.0 (2)
C15—Fe1—C11—C12	−118.26 (18)	C24—Fe1—C22—C23	37.52 (15)
C13—Fe1—C11—C12	−37.55 (14)	C15—Fe1—C22—C23	167.35 (19)
C14—Fe1—C11—C12	−80.94 (15)	C13—Fe1—C22—C23	−71.42 (19)
C25—Fe1—C11—C12	169.24 (15)	C14—Fe1—C22—C23	−36.1 (5)
C22—Fe1—C11—C2	−33.6 (2)	C11—Fe1—C22—C23	−156.76 (14)
C21—Fe1—C11—C2	10.0 (2)	C12—Fe1—C22—C23	−113.02 (16)
C24—Fe1—C11—C2	80.2 (4)	C25—Fe1—C22—C23	80.53 (17)
C23—Fe1—C11—C2	−69.6 (3)	C21—C22—C23—C24	−0.2 (3)
C15—Fe1—C11—C2	122.2 (2)	Fe1—C22—C23—C24	−60.35 (16)
C13—Fe1—C11—C2	−157.1 (2)	C21—C22—C23—Fe1	60.19 (16)
C14—Fe1—C11—C2	159.5 (2)	C22—Fe1—C23—C24	117.7 (2)

C12—Fe1—C11—C2	−119.6 (2)	C21—Fe1—C23—C24	79.32 (17)
C25—Fe1—C11—C2	49.7 (2)	C15—Fe1—C23—C24	−40.7 (4)
C15—C11—C12—C13	−0.8 (2)	C13—Fe1—C23—C24	−113.25 (16)
C2—C11—C12—C13	−178.56 (17)	C14—Fe1—C23—C24	−72.68 (19)
Fe1—C11—C12—C13	58.85 (14)	C11—Fe1—C23—C24	168.63 (17)
C15—C11—C12—Fe1	−59.68 (13)	C12—Fe1—C23—C24	−156.25 (14)
C2—C11—C12—Fe1	122.59 (17)	C25—Fe1—C23—C24	36.42 (15)
C22—Fe1—C12—C13	127.61 (17)	C21—Fe1—C23—C22	−38.37 (17)
C21—Fe1—C12—C13	168.93 (16)	C24—Fe1—C23—C22	−117.7 (2)
C24—Fe1—C12—C13	50.0 (3)	C15—Fe1—C23—C22	−158.4 (3)
C23—Fe1—C12—C13	83.34 (17)	C13—Fe1—C23—C22	129.06 (17)
C15—Fe1—C12—C13	−81.35 (15)	C14—Fe1—C23—C22	169.64 (17)
C14—Fe1—C12—C13	−37.50 (15)	C11—Fe1—C23—C22	50.9 (3)
C11—Fe1—C12—C13	−119.6 (2)	C12—Fe1—C23—C22	86.06 (18)
C25—Fe1—C12—C13	−159.7 (4)	C25—Fe1—C23—C22	−81.27 (18)
C22—Fe1—C12—C11	−112.78 (16)	C22—C23—C24—C25	−0.2 (3)
C21—Fe1—C12—C11	−71.46 (18)	Fe1—C23—C24—C25	−59.60 (17)
C24—Fe1—C12—C11	169.57 (18)	C22—C23—C24—Fe1	59.40 (15)
C23—Fe1—C12—C11	−157.05 (13)	C22—Fe1—C24—C23	−39.35 (17)
C15—Fe1—C12—C11	38.26 (12)	C21—Fe1—C24—C23	−83.88 (18)
C13—Fe1—C12—C11	119.6 (2)	C15—Fe1—C24—C23	167.04 (15)
C14—Fe1—C12—C11	82.11 (14)	C13—Fe1—C24—C23	83.70 (18)
C25—Fe1—C12—C11	−40.1 (5)	C14—Fe1—C24—C23	126.34 (16)
C11—C12—C13—C14	0.8 (2)	C11—Fe1—C24—C23	−159.4 (3)
Fe1—C12—C13—C14	59.68 (15)	C12—Fe1—C24—C23	49.0 (3)
C11—C12—C13—Fe1	−58.89 (14)	C25—Fe1—C24—C23	−120.8 (2)
C22—Fe1—C13—C14	167.36 (15)	C22—Fe1—C24—C25	81.46 (19)
C21—Fe1—C13—C14	−162.8 (4)	C21—Fe1—C24—C25	36.93 (18)
C24—Fe1—C13—C14	84.73 (16)	C23—Fe1—C24—C25	120.8 (2)
C23—Fe1—C13—C14	126.22 (15)	C15—Fe1—C24—C25	−72.15 (19)
C15—Fe1—C13—C14	−37.56 (13)	C13—Fe1—C24—C25	−155.49 (17)
C11—Fe1—C13—C14	−81.51 (14)	C14—Fe1—C24—C25	−112.84 (18)
C12—Fe1—C13—C14	−119.1 (2)	C11—Fe1—C24—C25	−38.6 (4)
C25—Fe1—C13—C14	51.5 (3)	C12—Fe1—C24—C25	169.9 (2)
C22—Fe1—C13—C12	−73.53 (18)	C23—C24—C25—C21	0.5 (3)
C21—Fe1—C13—C12	−43.6 (5)	Fe1—C24—C25—C21	−58.98 (17)
C24—Fe1—C13—C12	−156.16 (14)	C23—C24—C25—Fe1	59.49 (16)
C23—Fe1—C13—C12	−114.66 (15)	C22—C21—C25—C24	−0.6 (3)
C15—Fe1—C13—C12	81.56 (14)	Fe1—C21—C25—C24	59.15 (16)
C14—Fe1—C13—C12	119.1 (2)	C22—C21—C25—Fe1	−59.75 (16)
C11—Fe1—C13—C12	37.60 (13)	C22—Fe1—C25—C24	−81.30 (19)
C25—Fe1—C13—C12	170.6 (2)	C21—Fe1—C25—C24	−119.9 (2)
C12—C13—C14—C15	−0.4 (2)	C23—Fe1—C25—C24	−36.27 (16)
Fe1—C13—C14—C15	59.16 (15)	C15—Fe1—C25—C24	127.50 (16)
C12—C13—C14—Fe1	−59.59 (15)	C13—Fe1—C25—C24	49.2 (3)
C22—Fe1—C14—C13	−44.2 (5)	C14—Fe1—C25—C24	84.31 (18)
C21—Fe1—C14—C13	172.39 (19)	C11—Fe1—C25—C24	168.56 (14)
C24—Fe1—C14—C13	−112.82 (15)	C12—Fe1—C25—C24	−158.8 (4)
C23—Fe1—C14—C13	−73.23 (17)	C22—Fe1—C25—C21	38.59 (17)

C15—Fe1—C14—C13	119.6 (2)	C24—Fe1—C25—C21	119.9 (2)
C11—Fe1—C14—C13	82.08 (14)	C23—Fe1—C25—C21	83.62 (18)
C12—Fe1—C14—C13	37.96 (13)	C15—Fe1—C25—C21	−112.61 (17)
C25—Fe1—C14—C13	−154.62 (14)	C13—Fe1—C25—C21	169.1 (2)
C22—Fe1—C14—C15	−163.7 (4)	C14—Fe1—C25—C21	−155.80 (16)
C21—Fe1—C14—C15	52.8 (3)	C11—Fe1—C25—C21	−71.6 (2)
C24—Fe1—C14—C15	127.63 (14)	C12—Fe1—C25—C21	−38.9 (5)
C23—Fe1—C14—C15	167.22 (14)	N1—C1—C31—C32	−40.1 (3)
C13—Fe1—C14—C15	−119.6 (2)	N2—C1—C31—C32	145.14 (19)
C11—Fe1—C14—C15	−37.48 (13)	N1—C1—C31—C36	133.1 (2)
C12—Fe1—C14—C15	−81.59 (14)	N2—C1—C31—C36	−41.6 (3)
C25—Fe1—C14—C15	85.83 (16)	C36—C31—C32—C33	0.7 (3)
C13—C14—C15—C11	−0.1 (2)	C1—C31—C32—C33	174.14 (19)
Fe1—C14—C15—C11	59.16 (13)	C31—C32—C33—C34	−0.6 (3)
C13—C14—C15—Fe1	−59.25 (15)	C31—C32—C33—C37	−179.0 (2)
C12—C11—C15—C14	0.6 (2)	C32—C33—C34—C35	−0.5 (4)
C2—C11—C15—C14	178.24 (17)	C37—C33—C34—C35	177.9 (2)
Fe1—C11—C15—C14	−59.21 (14)	C33—C34—C35—C36	1.4 (3)
C12—C11—C15—Fe1	59.78 (13)	C33—C34—C35—C38	−177.0 (2)
C2—C11—C15—Fe1	−122.55 (18)	C34—C35—C36—C31	−1.2 (3)
C22—Fe1—C15—C14	171.4 (2)	C38—C35—C36—C31	177.1 (2)
C21—Fe1—C15—C14	−154.19 (16)	C32—C31—C36—C35	0.2 (3)
C24—Fe1—C15—C14	−72.36 (18)	C1—C31—C36—C35	−173.01 (18)
C23—Fe1—C15—C14	−40.8 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2B···N1^{i}	0.97	2.75	3.658 (2)	156
C22—H22···Cg^{i}	0.93	2.91	3.764 (3)	154

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1

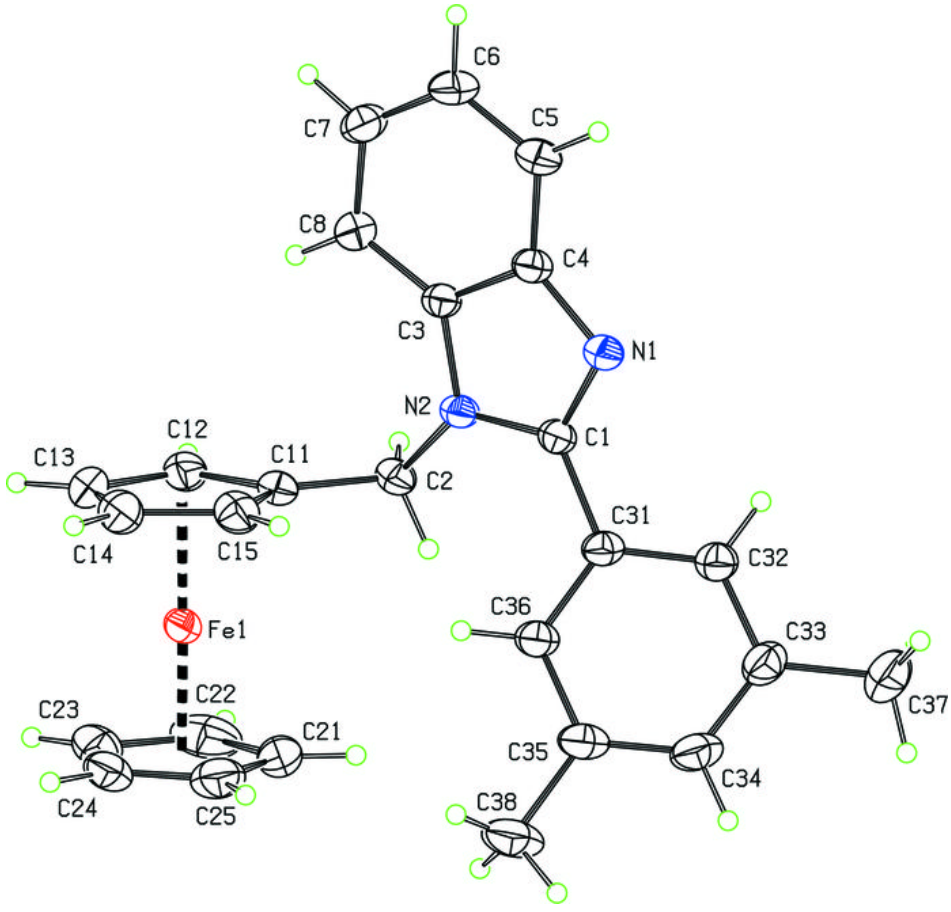

C6
C7
C5
C8
C4
C3
N1
N2
C1
C12
C13
C11
C2
C31
C14
C15
C32
C36
Fe1
C33
C22
C37
C23
C21
C35
C34
C24
C25
C38

Fig. 2