Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-Ferrocenyl-2-hydroxybenzohydrazide

Ying Xu, Chun-Ling Chen, Jing Zhou and Ming-Xue Li*

Institute of Molecular and Crystal Engineering, College of Chemistry and Chemical Engineering, Henan University, Kaifeng 475001, People's Republic of China
Correspondence e-mail: limingxue@henu.edu.cn

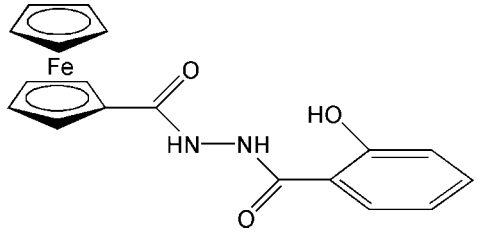
Received 13 November 2007; accepted 5 December 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.049; wR factor = 0.086; data-to-parameter ratio = 11.4.

The title complex, $[Fe(C_5H_5)(C_{13}H_{11}N_2O_3)]$, was prepared *via* self-assembly using ferrocenyl hydrazide and ethyl salicylate. The compound is potentially a tridentate ferrocene-based ligand. The conformation of the molecule allows the formation of an intramolecular N—H···O hydrogen bond involving the hydroxyl group. The CONHNHCO unit and the rings bonded to it are nearly coplanar. The crystal structure is stabilized by intermolecular O—H···O(carbonyl) and N—H···O(carbonyl) hydrogen bonds.

Related literature

For related literature about applications of ferrocene complexes, see: Beer (1992); Beer & Smith (1997); Long (1995); Miller & Epstein (1994); Nguyen *et al.* (1999).



Experimental

Crystal data

$[Fe(C_5H_5)(C_{13}H_{11}N_2O_3)]$
M_r = 364.18
Monoclinic, $C2/c$
a = 20.680 (3) Å
b = 9.9673 (15) Å
c = 16.941 (3) Å
β = 121.704 (3)°
V = 2970.8 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.03 mm^{-1}
T = 293 (2) K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.820, T_{max} = 0.852
7479 measured reflections
2611 independent reflections
1053 reflections with $I > 2\sigma(I)$
R_{int} = 0.129

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.086
S = 0.57
2611 reflections
230 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O3	0.860 (10)	1.95 (2)	2.631 (4)	135 (3)
O3—H3B···O1^{i}	0.822 (10)	1.908 (15)	2.705 (4)	163 (4)
N1—H1B···O2^{ii}	0.871 (10)	2.03 (2)	2.810 (4)	148 (4)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, y, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; 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: *SHELXL97*.

This work was financially supported by the Foundation of the Educational Department of Henan Province (No. 2007150012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2153).

References

Beer, P. D. (1992). *Adv. Inorg. Chem.* **39**, 79–157.
Beer, P. D. & Smith, D. K. (1997). *Prog. Inorg. Chem.* **46**, 1–8.
Bruker (2000). *SMART* (Version 5.054), *SAINT-Plus* (Version 6.22) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Long, N. J. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 21–38.
Miller, J. S. & Epstein, A. J. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 385–415.
Nguyen, P., Gómez-Elipe, P. & Manners, I. (1999). *Chem. Rev.* **99**, 1515–1548.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

 doi:10.1107/S1600536807065634

supporting information

Acta Cryst. (2008). E**64**, m177 [https://doi.org/10.1107/S1600536807065634]

N′-Ferrocenyl-2-hydroxybenzohydrazide

Ying Xu, Chun-Ling Chen, Jing Zhou and Ming-Xue Li

S1. Comment

The synthesis, isolation and characterization of ferrocene in 1951 marked an important milestone in the evolution of modern organometallic chemistry. Potential applications in material sciences, such as molecular sensors (Beer, 1992; Beer & Smith, 1997), molecular magnetic materials (Miller & Epstein, 1994), and nonlinear optical materials (Nguyen *et al.*, 1999; Long, 1995) attracted much attention. We report here the crystal structure of the title compound, (I), a new ferrocene-based complex (Fig. 1).

The title compound belongs to space group *C*2/*c*. The bond lengths O1?C11 and O2?C12 are 1.240 (5) and 1.233 (4) Å, respectively, as excepted for double bonds. The bond length O3—C18, 1.349 (5) Å, corresponds to a single bond. The N1—C11 and N2—C12 bond distances are 1.340 (5) and 1.343 (5) Å, respectively, which make clear they are in the normal range for N—C single bonds. The bond length N1—N2 = 1.381 (4) Å is also consistent with a single N—N bond. An intramolecular N2—H2B···O3 hydrogen bond is observed in the molecular structure.

In the crystal, molecules are connected by intermolecular hydrogen bonds involving carbonyl O atoms O2 and O3 as acceptor and N—H or O—H groups as donors.

S2. Experimental

All reagents were commercially available and of analytical grade. Ferrocenyl hydrazide (1.22 g, 5 mmol) and ethyl salicylate (0.83 g, 5 mmol) were mixed in ethanol and refluxed for 7 h. The resulting red solid was filtered, washed with ethanol and dried under reduced pressure. Anal. Calc. for $C_{18}H_{16}FeN_2O_3$: C 59.37, H 4.43, N 7.69%. Found: C 59.48, H 4.31, N 7.52%.

S3. Refinement

H atoms bonded to C atoms were positioned geometrically and refined as riding on their carrier atoms, with C—H bond lengths fixed to 0.93 (benzene ring) or 0.98 Å (Cp rings), and U_{iso}(H) = 1.2U_{eq}(carrier C). H atoms bonded to heteroatoms N1, N2 and O3 were located in a difference map and were freely refined as isotropic atoms, with restricted bond lengths: N—H = 0.87 (1) Å and O—H = 0.82 (1) Å.

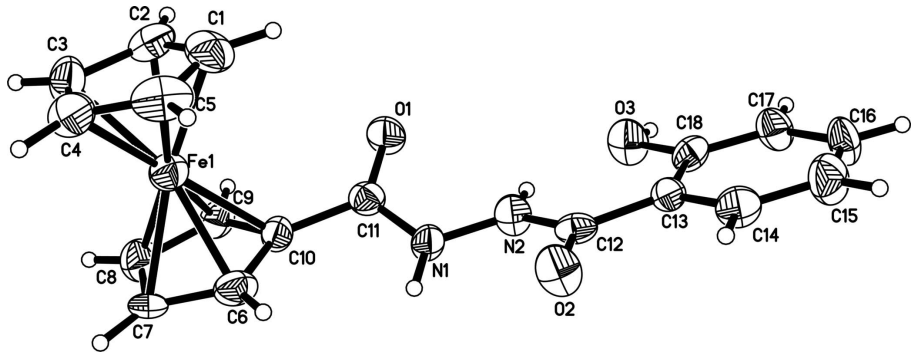


Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

N′-Ferrocenyl-2-hydroxybenzohydrazide

Crystal data

$[Fe(C_5H_5)(C_{13}H_{11}N_2O_3)]$
$M_r = 364.18$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 20.680$ (3) Å
$b = 9.9673$ (15) Å
$c = 16.941$ (3) Å
$\beta = 121.704$ (3)°
$V = 2970.8$ (8) Å^3
$Z = 8$
$F(000) = 1504$
$D_x = 1.628$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 397 reflections
$\theta = 2.3$–$28.0°$
$\mu = 1.04$ mm^{-1}
$T = 293$ K
Block, red
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.3° wide ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.820$, $T_{max} = 0.852$
7479 measured reflections
2611 independent reflections
1053 reflections with $I > 2\sigma(I)$
$R_{int} = 0.129$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -24 \rightarrow 18$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.086$
$S = 0.57$
2611 reflections
230 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.94540 (4)	0.77786 (6)	0.86429 (4)	0.03507 (19)
O1	0.83150 (16)	0.4726 (3)	0.79890 (19)	0.0410 (9)
O2	0.92978 (15)	0.2865 (3)	0.63670 (17)	0.0437 (8)
O3	0.75985 (16)	0.1341 (3)	0.6733 (2)	0.0417 (8)
H3B	0.7289 (10)	0.099 (3)	0.683 (2)	0.060 (9)*
N1	0.9235 (2)	0.3971 (3)	0.7765 (2)	0.0308 (10)
H1B	0.9700 (8)	0.387 (4)	0.790 (2)	0.058 (16)*
N2	0.87479 (18)	0.2969 (4)	0.7205 (2)	0.0340 (9)
H2B	0.8365 (9)	0.276 (3)	0.7248 (17)	0.034 (5)*
C1	0.8498 (3)	0.8152 (5)	0.7406 (3)	0.0489 (14)
H1A	0.8151	0.7473	0.6970	0.059*
C2	0.8456 (3)	0.8730 (4)	0.8133 (3)	0.0432 (13)
H2A	0.8071	0.8530	0.8287	0.052*
C3	0.9052 (3)	0.9656 (4)	0.8594 (3)	0.0451 (14)
H3A	0.9161	1.0216	0.9126	0.054*
C4	0.9463 (3)	0.9629 (4)	0.8144 (3)	0.0454 (14)
H4A	0.9919	1.0160	0.8322	0.055*
C5	0.9131 (3)	0.8688 (5)	0.7419 (3)	0.0529 (15)
H5A	0.9302	0.8462	0.6995	0.064*
C6	1.0186 (3)	0.6269 (4)	0.8847 (3)	0.0376 (13)
H6A	1.0360	0.6011	0.8430	0.045*
C7	1.0541 (2)	0.7191 (4)	0.9589 (3)	0.0415 (13)
H7A	1.1002	0.7706	0.9767	0.050*
C8	1.0106 (3)	0.7288 (4)	1.0008 (3)	0.0444 (13)
H8A	1.0216	0.7865	1.0533	0.053*
C9	0.9489 (3)	0.6410 (4)	0.9538 (3)	0.0337 (12)
H9A	0.9093	0.6261	0.9681	0.040*
C10	0.9534 (3)	0.5776 (4)	0.8825 (3)	0.0304 (12)
C11	0.8976 (3)	0.4797 (4)	0.8161 (3)	0.0302 (12)
C12	0.8800 (2)	0.2468 (4)	0.6504 (3)	0.0272 (11)
C13	0.8246 (2)	0.1417 (4)	0.5917 (3)	0.0269 (11)
C14	0.8305 (3)	0.0933 (4)	0.5195 (3)	0.0388 (13)
H14A	0.8674	0.1284	0.5097	0.047*
C15	0.7828 (3)	−0.0064 (4)	0.4615 (3)	0.0490 (15)
H15A	0.7875	−0.0372	0.4129	0.059*
C16	0.7285 (3)	−0.0601 (4)	0.4754 (3)	0.0422 (14)
H16A	0.6968	−0.1283	0.4371	0.051*
C17	0.7214 (2)	−0.0123 (4)	0.5463 (3)	0.0375 (13)
H17A	0.6845	−0.0485	0.5556	0.045*
C18	0.7675 (3)	0.0877 (4)	0.6038 (3)	0.0300 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0332 (4)	0.0339 (3)	0.0390 (4)	0.0019 (4)	0.0195 (3)	−0.0003 (4)

O1	0.0258 (18)	0.0442 (19)	0.058 (2)	−0.0049 (17)	0.0251 (17)	−0.0128 (15)
O2	0.0409 (17)	0.061 (2)	0.0440 (17)	−0.0154 (18)	0.0328 (15)	−0.0120 (16)
O3	0.0369 (18)	0.052 (2)	0.0518 (19)	−0.0128 (17)	0.0345 (16)	−0.0080 (16)
N1	0.026 (2)	0.029 (2)	0.043 (2)	0.000 (2)	0.022 (2)	−0.0085 (17)
N2	0.029 (2)	0.038 (2)	0.043 (2)	−0.005 (2)	0.0246 (18)	−0.0056 (19)
C1	0.043 (3)	0.058 (3)	0.031 (3)	0.006 (3)	0.010 (3)	0.001 (2)
C2	0.044 (3)	0.037 (3)	0.056 (3)	0.013 (3)	0.031 (3)	0.010 (2)
C3	0.049 (3)	0.031 (3)	0.059 (3)	−0.007 (3)	0.031 (3)	−0.007 (2)
C4	0.042 (3)	0.036 (3)	0.066 (4)	0.002 (3)	0.034 (3)	0.011 (3)
C5	0.051 (3)	0.069 (4)	0.048 (3)	0.019 (3)	0.032 (3)	0.021 (3)
C6	0.033 (3)	0.044 (3)	0.033 (3)	0.010 (3)	0.016 (2)	−0.003 (2)
C7	0.021 (2)	0.033 (3)	0.048 (3)	0.011 (3)	0.002 (2)	0.005 (3)
C8	0.049 (3)	0.039 (3)	0.040 (3)	0.000 (3)	0.020 (3)	−0.006 (2)
C9	0.039 (3)	0.029 (3)	0.038 (3)	0.002 (2)	0.024 (2)	−0.002 (2)
C10	0.028 (3)	0.027 (2)	0.037 (3)	−0.001 (2)	0.018 (2)	−0.005 (2)
C11	0.033 (3)	0.028 (3)	0.027 (3)	0.001 (3)	0.013 (2)	0.003 (2)
C12	0.024 (2)	0.030 (3)	0.031 (2)	0.005 (2)	0.016 (2)	0.008 (2)
C13	0.025 (3)	0.023 (2)	0.032 (3)	0.000 (2)	0.014 (2)	0.002 (2)
C14	0.038 (3)	0.042 (3)	0.046 (3)	0.000 (3)	0.028 (3)	0.000 (2)
C15	0.052 (4)	0.056 (3)	0.045 (3)	−0.006 (3)	0.030 (3)	−0.014 (3)
C16	0.038 (3)	0.043 (3)	0.044 (3)	−0.017 (3)	0.021 (3)	−0.019 (2)
C17	0.033 (3)	0.040 (3)	0.041 (3)	−0.012 (2)	0.020 (3)	−0.007 (2)
C18	0.034 (3)	0.030 (3)	0.031 (3)	0.005 (2)	0.020 (2)	0.002 (2)

Geometric parameters (Å, º)

Fe1—C2	2.008 (4)	C3—H3A	0.9800
Fe1—C9	2.013 (4)	C4—C5	1.405 (6)
Fe1—C10	2.014 (4)	C4—H4A	0.9800
Fe1—C1	2.021 (4)	C5—H5A	0.9800
Fe1—C5	2.028 (5)	C6—C7	1.412 (5)
Fe1—C6	2.031 (4)	C6—C10	1.416 (6)
Fe1—C3	2.031 (4)	C6—H6A	0.9800
Fe1—C8	2.031 (4)	C7—C8	1.412 (6)
Fe1—C4	2.033 (4)	C7—H7A	0.9800
Fe1—C7	2.045 (4)	C8—C9	1.400 (5)
O1—C11	1.240 (5)	C8—H8A	0.9800
O2—C12	1.233 (4)	C9—C10	1.410 (5)
O3—C18	1.349 (5)	C9—H9A	0.9800
O3—H3B	0.822 (10)	C10—C11	1.478 (5)
N1—C11	1.340 (5)	C12—C13	1.484 (5)
N1—N2	1.381 (4)	C13—C14	1.379 (6)
N1—H1B	0.871 (10)	C13—C18	1.407 (6)
N2—C12	1.343 (5)	C14—C15	1.381 (5)
N2—H2B	0.860 (10)	C14—H14A	0.9300
C1—C5	1.402 (6)	C15—C16	1.373 (6)
C1—C2	1.404 (6)	C15—H15A	0.9300
C1—H1A	0.9800	C16—C17	1.370 (6)

C2—C3	1.403 (6)	C16—H16A	0.9300
C2—H2A	0.9800	C17—C18	1.369 (5)
C3—C4	1.410 (6)	C17—H17A	0.9300
C2—Fe1—C9	105.43 (18)	C4—C3—H3A	126.6
C2—Fe1—C10	121.17 (19)	Fe1—C3—H3A	126.6
C9—Fe1—C10	41.01 (16)	C5—C4—C3	109.2 (4)
C2—Fe1—C1	40.79 (16)	C5—C4—Fe1	69.5 (3)
C9—Fe1—C1	122.31 (19)	C3—C4—Fe1	69.6 (3)
C10—Fe1—C1	107.34 (18)	C5—C4—H4A	125.4
C2—Fe1—C5	68.7 (2)	C3—C4—H4A	125.4
C9—Fe1—C5	159.32 (19)	Fe1—C4—H4A	125.4
C10—Fe1—C5	123.77 (19)	C1—C5—C4	107.0 (5)
C1—Fe1—C5	40.53 (17)	C1—C5—Fe1	69.5 (3)
C2—Fe1—C6	158.24 (18)	C4—C5—Fe1	70.0 (3)
C9—Fe1—C6	68.96 (18)	C1—C5—H5A	126.5
C10—Fe1—C6	40.98 (16)	C4—C5—H5A	126.5
C1—Fe1—C6	123.23 (18)	Fe1—C5—H5A	126.5
C5—Fe1—C6	108.72 (19)	C7—C6—C10	107.0 (4)
C2—Fe1—C3	40.66 (16)	C7—C6—Fe1	70.3 (2)
C9—Fe1—C3	120.45 (18)	C10—C6—Fe1	68.9 (2)
C10—Fe1—C3	156.7 (2)	C7—C6—H6A	126.5
C1—Fe1—C3	68.42 (18)	C10—C6—H6A	126.5
C5—Fe1—C3	68.83 (19)	Fe1—C6—H6A	126.5
C6—Fe1—C3	160.36 (19)	C8—C7—C6	108.7 (4)
C2—Fe1—C8	121.38 (19)	C8—C7—Fe1	69.2 (2)
C9—Fe1—C8	40.51 (16)	C6—C7—Fe1	69.2 (2)
C10—Fe1—C8	68.62 (17)	C8—C7—H7A	125.6
C1—Fe1—C8	158.0 (2)	C6—C7—H7A	125.6
C5—Fe1—C8	159.5 (2)	Fe1—C7—H7A	125.6
C6—Fe1—C8	68.81 (18)	C9—C8—C7	107.6 (4)
C3—Fe1—C8	106.29 (19)	C9—C8—Fe1	69.0 (2)
C2—Fe1—C4	67.96 (18)	C7—C8—Fe1	70.2 (2)
C9—Fe1—C4	157.47 (19)	C9—C8—H8A	126.2
C10—Fe1—C4	160.9 (2)	C7—C8—H8A	126.2
C1—Fe1—C4	67.63 (19)	Fe1—C8—H8A	126.2
C5—Fe1—C4	40.48 (17)	C8—C9—C10	108.4 (4)
C6—Fe1—C4	125.03 (19)	C8—C9—Fe1	70.5 (2)
C3—Fe1—C4	40.59 (17)	C10—C9—Fe1	69.5 (2)
C8—Fe1—C4	123.09 (19)	C8—C9—H9A	125.8
C2—Fe1—C7	158.59 (19)	C10—C9—H9A	125.8
C9—Fe1—C7	68.03 (18)	Fe1—C9—H9A	125.8
C10—Fe1—C7	68.14 (18)	C9—C10—C6	108.2 (4)
C1—Fe1—C7	159.89 (19)	C9—C10—C11	124.8 (4)
C5—Fe1—C7	124.4 (2)	C6—C10—C11	127.0 (4)
C6—Fe1—C7	40.55 (15)	C9—C10—Fe1	69.5 (2)
C3—Fe1—C7	123.62 (18)	C6—C10—Fe1	70.2 (2)
C8—Fe1—C7	40.54 (16)	C11—C10—Fe1	124.7 (3)

C4—Fe1—C7	109.83 (19)	O1—C11—N1	121.9 (4)
C18—O3—H3B	120 (3)	O1—C11—C10	122.7 (4)
C11—N1—N2	116.7 (4)	N1—C11—C10	115.3 (4)
C11—N1—H1B	128 (2)	O2—C12—N2	120.7 (4)
N2—N1—H1B	114 (2)	O2—C12—C13	121.8 (4)
C12—N2—N1	120.5 (3)	N2—C12—C13	117.6 (4)
C12—N2—H2B	119 (2)	C14—C13—C18	117.9 (4)
N1—N2—H2B	120 (2)	C14—C13—C12	116.4 (4)
C5—C1—C2	108.5 (4)	C18—C13—C12	125.7 (4)
C5—C1—Fe1	70.0 (3)	C13—C14—C15	121.4 (5)
C2—C1—Fe1	69.1 (3)	C13—C14—H14A	119.3
C5—C1—H1A	125.7	C15—C14—H14A	119.3
C2—C1—H1A	125.7	C16—C15—C14	120.1 (5)
Fe1—C1—H1A	125.7	C16—C15—H15A	120.0
C3—C2—C1	108.5 (4)	C14—C15—H15A	120.0
C3—C2—Fe1	70.6 (3)	C17—C16—C15	119.3 (4)
C1—C2—Fe1	70.1 (3)	C17—C16—H16A	120.3
C3—C2—H2A	125.8	C15—C16—H16A	120.3
C1—C2—H2A	125.8	C18—C17—C16	121.4 (4)
Fe1—C2—H2A	125.8	C18—C17—H17A	119.3
C2—C3—C4	106.8 (4)	C16—C17—H17A	119.3
C2—C3—Fe1	68.8 (2)	O3—C18—C17	120.8 (4)
C4—C3—Fe1	69.8 (3)	O3—C18—C13	119.2 (4)
C2—C3—H3A	126.6	C17—C18—C13	120.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O3	0.86 (1)	1.95 (2)	2.631 (4)	135 (3)
O3—H3*B*···O1^{i}	0.82 (1)	1.91 (2)	2.705 (4)	163 (4)
N1—H1*B*···O2^{ii}	0.87 (1)	2.03 (2)	2.810 (4)	148 (4)

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+3/2; (ii) −*x*+2, *y*, −*z*+3/2.