

Tris(1,10-phenanthroline- κ^2N,N')iron(II) μ -oxido-bis[trichloridoferrate(III)] ethanol hemisolvate

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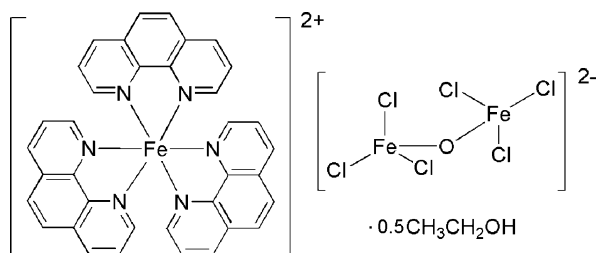
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(C-C) = 0.011$ Å; disorder in solvent or counterion; R factor = 0.064; wR factor = 0.212; data-to-parameter ratio = 14.9.

The title compound, $[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_2)_3][\text{Fe}_2\text{Cl}_6\text{O}] \cdot 0.5\text{CH}_3\text{CH}_2\text{OH}$, consists of one $[\text{Fe}(\text{phen})_3]^{2+}$ cation (phen = 1,10-phenanthroline), one $[\text{Fe}_2\text{Cl}_6\text{O}]^{2-}$ anion and one half-molecule of ethanol. In the cation, the Fe^{II} atom is coordinated by six N atoms from three phen ligands in a distorted octahedral geometry. In the bent anion, two Fe^{III} atoms are connected by a bridging oxide O atom [bridging angle = $160.6(4)^\circ$], and each Fe^{III} atom is also coordinated by three Cl atoms, completing a distorted tetrahedral geometry.

Related literature

For general background, see: Hwang & Ha (2006); Potočník *et al.* (2002); Zhou & Guo (2007). For a related structure, see: Aparici Plaza *et al.* (2007).



Experimental

Crystal data

$[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_2)_3][\text{Fe}_2\text{Cl}_6\text{O}] \cdot 0.5\text{C}_2\text{H}_5\text{O}$
 $M_r = 959.89$
 Monoclinic, $P2_1/c$
 $a = 15.3536(18)$ Å
 $b = 13.1857(16)$ Å
 $c = 20.897(2)$ Å
 $\beta = 94.701(2)^\circ$
 $V = 4216.4(8)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.44$ mm⁻¹
 $T = 294(2)$ K
 $0.55 \times 0.47 \times 0.42$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.452$, $T_{\text{max}} = 0.544$
 20409 measured reflections
 7434 independent reflections
 4248 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.212$
 $S = 1.02$
 7434 reflections
 498 parameters
 3 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.84$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Selected bond lengths (Å).

Fe1—N4	1.958 (6)	Fe2—Cl4	2.204 (3)
Fe1—N2	1.970 (5)	Fe2—Cl6	2.223 (2)
Fe1—N6	1.979 (5)	Fe2—Cl5	2.231 (2)
Fe1—N1	1.980 (5)	Fe3—O1	1.756 (5)
Fe1—N3	1.982 (5)	Fe3—Cl2	2.213 (2)
Fe1—N5	1.985 (5)	Fe3—Cl3	2.227 (2)
Fe2—O1	1.735 (5)	Fe3—Cl1	2.227 (2)

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2160).

References

- Aparici Plaza, L., Baranowska, K. & Becker, B. (2007). *Acta Cryst.* **E63**, m1537–m1539.
 Bruker (2007). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Hwang, I.-C. & Ha, K. (2006). *Acta Cryst.* **E62**, m376–m378.
 Potočník, I., Pohlová, M., Wagner, C. & Jäger, L. (2002). *Acta Cryst.* **E58**, m595–m596.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Zhou, D.-P. & Guo, G.-L. (2007). *Acta Cryst.* **E63**, m1122–m1124.

supplementary materials

Acta Cryst. (2008). E64, m1514 [doi:10.1107/S1600536808035897]

Tris(1,10-phenanthroline- κ^2N,N')iron(II) μ -oxido-bis[trichloridoferrate(III)] ethanol hemisolvate

Z.-X. Li, M.-M. Yu, Y.-N. Zhang and L.-H. Wei

Comment

1,10-Phenanthroline (phen) is a widely utilized chelating ligand in coordination chemistry and a lot of complexes with phen as a ligand have been reported (Hwang & Ha, 2006; Potocnak *et al.*, 2002; Zhou & Guo, 2007). Plaza *et al.* (2007) has reported the structure of a complex with $[\text{Fe}(\text{phen})_3]^{2+}$ as cation and simple halide as counter-ion. We report here the synthesis and crystal structure of the title compound, in which an oxide-bridged iron(III) complex acts as counter-ion.

The title compound comprises one $[\text{Fe}(\text{phen})_3]^{2+}$ cation, one $[\text{Fe}_2\text{Cl}_6\text{O}]^{2-}$ anion and a half of solvent ethanol molecule (Fig. 1). The Fe^{II} atom in the cation is coordinated by six N atoms from three phen ligands, forming a distorted FeN_6 octahedral geometry. The Fe—N bond lengths are in the normal range (Table 1). In the anion, two Fe^{III} atoms are bridged by an oxide O atom and is also coordinated by three Cl atoms in a distorted tetrahedral geometry. The Fe—Cl bond distances are in the range of 2.204 (3) to 2.231 (2) Å. The distance between the Fe^{III} atoms (Fe2 and Fe3) bridged by the O1 atom is 3.441 (5) Å [Fe2—O1 = 1.735 (5) and Fe3—O1 = 1.756 (5) Å].

Experimental

$\text{Fe}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.036 g, 0.1 mmol) and 1,10-phenanthroline (0.054 g, 0.3 mmol) are dissolved in 15 ml of water and ethanol (1:2 in volume), resulting in a pale green solution, which was transferred to the left hand of an H-shaped tube. An aqueous solution (15 ml) of FeCl_3 (0.032 g, 0.2 mmol) was placed in the right hand of the tube. An ethanol solution serving as the diffusion solvent was put in the connecting part between the left and right hands of the tube. The reaction container was kept at room temperature. Dark brown crystals suitable for X-ray diffraction analysis were obtained in the connecting part of the H-shaped tube after two months (yield 60% based on Fe).

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (aromatic), 0.97 (CH_2) and 0.96 (CH_3) Å and $U_{\text{iso}}(\text{H}) = 1.2(1.5 \text{ for methyl})U_{\text{eq}}(\text{C})$, and with O—H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

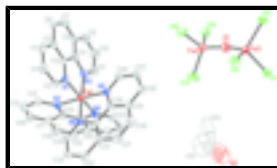


Fig. 1. The structures of the cation, anion and solvate molecule in the title compound. Displacement ellipsoids are drawn at the 30% probability level.

Tris(1,10-phenanthroline- κ^2N,N')iron(II) μ -oxido-bis[trichloridoferrate(III)] ethanol semisolvate

Crystal data

$[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_2)_3][\text{Fe}_2\text{Cl}_6\text{O}] \cdot 0.5\text{C}_2\text{H}_6\text{O}$

$M_r = 959.89$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 15.3536$ (18) Å

$b = 13.1857$ (16) Å

$c = 20.897$ (2) Å

$\beta = 94.701$ (2)°

$V = 4216.4$ (8) Å³

$Z = 4$

$F_{000} = 1932$

$D_x = 1.512$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 20409 reflections

$\theta = 1.8$ – 25.0 °

$\mu = 1.44$ mm⁻¹

$T = 294$ (2) K

Block, brown

$0.55 \times 0.47 \times 0.42$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.452$, $T_{\max} = 0.544$

20409 measured reflections

7434 independent reflections

4248 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\max} = 25.0$ °

$\theta_{\min} = 1.8$ °

$h = -18 \rightarrow 14$

$k = -15 \rightarrow 15$

$l = -17 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.212$

$S = 1.03$

7434 reflections

498 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-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1185P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.018$

$\Delta\rho_{\max} = 0.84$ e Å⁻³

$\Delta\rho_{\min} = -0.39$ e Å⁻³

Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Fe1	0.25729 (6)	0.78424 (6)	0.06260 (4)	0.0388 (3)	
Fe2	0.21293 (7)	0.12200 (8)	0.71711 (5)	0.0533 (3)	
Fe3	0.34590 (7)	0.14054 (7)	0.85736 (5)	0.0505 (3)	
Cl1	0.27250 (13)	0.18682 (15)	0.94022 (9)	0.0645 (5)	
Cl2	0.43389 (15)	0.26757 (15)	0.83747 (11)	0.0765 (6)	
Cl3	0.42517 (14)	0.00431 (14)	0.88656 (10)	0.0696 (6)	
Cl4	0.1307 (2)	0.2590 (2)	0.70798 (16)	0.1203 (11)	
Cl5	0.12833 (15)	-0.01536 (17)	0.70469 (11)	0.0815 (7)	
Cl6	0.30925 (16)	0.12534 (15)	0.64332 (11)	0.0811 (7)	
O1	0.2707 (4)	0.1130 (5)	0.7918 (3)	0.0874 (18)	
N1	0.3671 (4)	0.7608 (4)	0.0213 (2)	0.0440 (13)	
N2	0.2292 (4)	0.6451 (4)	0.0334 (3)	0.0500 (15)	
N3	0.1978 (3)	0.8384 (4)	-0.0180 (2)	0.0431 (13)	
N4	0.1411 (4)	0.7998 (4)	0.0936 (3)	0.0467 (14)	
N5	0.3138 (3)	0.7370 (4)	0.1461 (2)	0.0393 (12)	
N6	0.2932 (3)	0.9184 (4)	0.0981 (2)	0.0400 (12)	
C2	0.4338 (4)	0.8226 (5)	0.0168 (3)	0.0475 (17)	
H27	0.4333	0.8864	0.0357	0.057*	
C3	0.5060 (5)	0.7928 (7)	-0.0164 (3)	0.063 (2)	
H36	0.5519	0.8378	-0.0205	0.076*	
C4	0.5088 (6)	0.6978 (7)	-0.0427 (3)	0.068 (2)	
H33	0.5571	0.6775	-0.0636	0.082*	
C5	0.4394 (6)	0.6325 (6)	-0.0380 (3)	0.057 (2)	
C6	0.4341 (7)	0.5289 (7)	-0.0641 (3)	0.076 (3)	
H38	0.4798	0.5029	-0.0856	0.091*	
C7	0.3610 (8)	0.4702 (7)	-0.0565 (4)	0.085 (3)	
H47	0.3588	0.4049	-0.0734	0.102*	
C8	0.2901 (6)	0.5047 (6)	-0.0244 (4)	0.064 (2)	
C9	0.2164 (8)	0.4478 (6)	-0.0139 (4)	0.085 (3)	
H32	0.2108	0.3821	-0.0300	0.102*	
C10	0.1525 (7)	0.4877 (7)	0.0196 (5)	0.088 (3)	
H49	0.1036	0.4496	0.0277	0.105*	
C11	0.1617 (5)	0.5860 (6)	0.0415 (3)	0.062 (2)	
H37	0.1169	0.6127	0.0637	0.075*	
C12	0.3679 (5)	0.6666 (5)	-0.0059 (3)	0.0440 (16)	
C13	0.2948 (5)	0.6026 (5)	0.0009 (3)	0.0490 (17)	
C14	0.2293 (5)	0.8619 (5)	-0.0731 (3)	0.0508 (18)	
H26	0.2890	0.8547	-0.0765	0.061*	
C15	0.1775 (5)	0.8967 (6)	-0.1261 (4)	0.063 (2)	
H45	0.2029	0.9139	-0.1635	0.076*	
C16	0.0899 (6)	0.9056 (6)	-0.1234 (4)	0.069 (2)	
H20	0.0548	0.9260	-0.1594	0.083*	
C17	0.0521 (5)	0.8837 (6)	-0.0653 (4)	0.066 (2)	
C18	-0.0379 (6)	0.8918 (8)	-0.0552 (4)	0.087 (3)	
H44	-0.0775	0.9122	-0.0889	0.105*	

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C19	-0.0667 (5)	0.8707 (8)	0.0018 (4)	0.092 (3)	
H50	-0.1263	0.8757	0.0067	0.111*	
C20	-0.0097 (5)	0.8408 (7)	0.0554 (4)	0.068 (2)	
C21	-0.0344 (5)	0.8189 (7)	0.1172 (5)	0.082 (3)	
H46	-0.0928	0.8239	0.1257	0.099*	
C22	0.0263 (6)	0.7905 (7)	0.1645 (4)	0.076 (2)	
H35	0.0095	0.7769	0.2054	0.092*	
C23	0.1141 (5)	0.7815 (6)	0.1520 (4)	0.062 (2)	
H43	0.1549	0.7622	0.1851	0.075*	
C24	0.1109 (4)	0.8498 (5)	-0.0134 (3)	0.0482 (17)	
C25	0.0799 (4)	0.8296 (5)	0.0472 (3)	0.0485 (17)	
C26	0.3254 (4)	0.6437 (5)	0.1694 (3)	0.0484 (17)	
H28	0.3050	0.5893	0.1442	0.058*	
C27	0.3662 (5)	0.6239 (5)	0.2291 (4)	0.0580 (19)	
H18	0.3717	0.5573	0.2434	0.070*	
C28	0.3980 (5)	0.6995 (6)	0.2667 (3)	0.060 (2)	
H2	0.4250	0.6858	0.3072	0.072*	
C29	0.3903 (5)	0.7982 (5)	0.2448 (3)	0.0528 (18)	
C30	0.4249 (6)	0.8884 (6)	0.2784 (4)	0.076 (3)	
H42	0.4580	0.8807	0.3174	0.091*	
C31	0.4105 (6)	0.9821 (6)	0.2548 (4)	0.076 (3)	
H48	0.4306	1.0379	0.2789	0.092*	
C32	0.3649 (5)	0.9978 (5)	0.1934 (3)	0.0542 (19)	
C33	0.3478 (5)	1.0945 (5)	0.1650 (4)	0.062 (2)	
H34	0.3652	1.1535	0.1868	0.075*	
C34	0.3054 (5)	1.0992 (5)	0.1050 (4)	0.058 (2)	
H25	0.2939	1.1618	0.0857	0.070*	
C35	0.2796 (4)	1.0107 (5)	0.0728 (3)	0.0489 (17)	
H19	0.2515	1.0159	0.0318	0.059*	
C36	0.3465 (4)	0.8139 (4)	0.1835 (3)	0.0396 (15)	
C37	0.3342 (4)	0.9128 (5)	0.1575 (3)	0.0405 (15)	
C51	0.2017 (13)	0.6513 (18)	0.8088 (10)	0.128 (9)	0.50
H51A	0.1715	0.7143	0.8008	0.191*	0.50
H51B	0.2320	0.6333	0.7721	0.191*	0.50
H51C	0.2429	0.6583	0.8456	0.191*	0.50
C52	0.1376 (18)	0.570 (2)	0.8211 (13)	0.29 (4)	0.50
H52A	0.1042	0.5525	0.7813	0.347*	0.50
H52B	0.1682	0.5104	0.8376	0.347*	0.50
O2	0.0796 (14)	0.6063 (17)	0.8669 (11)	0.183 (10)	0.50
H2A	0.0895	0.6664	0.8746	0.275*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0416 (5)	0.0331 (5)	0.0421 (5)	0.0006 (4)	0.0060 (4)	0.0023 (4)
Fe2	0.0609 (7)	0.0507 (6)	0.0489 (6)	0.0022 (5)	0.0082 (5)	0.0018 (5)
Fe3	0.0615 (7)	0.0439 (6)	0.0467 (6)	-0.0040 (5)	0.0082 (5)	0.0014 (4)
Cl1	0.0630 (13)	0.0672 (12)	0.0658 (12)	-0.0007 (10)	0.0218 (10)	0.0013 (9)

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C12	0.0889 (16)	0.0510 (11)	0.0956 (16)	-0.0046 (10)	0.0435 (13)	0.0125 (10)
C13	0.0819 (14)	0.0420 (10)	0.0844 (14)	0.0027 (9)	0.0029 (11)	-0.0028 (9)
C14	0.116 (2)	0.0853 (18)	0.166 (3)	0.0400 (16)	0.050 (2)	0.0337 (17)
C15	0.0851 (16)	0.0794 (15)	0.0809 (15)	-0.0258 (12)	0.0116 (12)	-0.0001 (11)
C16	0.1018 (17)	0.0544 (12)	0.0941 (16)	-0.0114 (11)	0.0504 (13)	-0.0036 (11)
O1	0.106 (5)	0.089 (4)	0.064 (4)	-0.003 (3)	-0.006 (3)	-0.002 (3)
N1	0.051 (4)	0.039 (3)	0.043 (3)	0.012 (3)	0.010 (3)	0.006 (2)
N2	0.062 (4)	0.042 (3)	0.045 (3)	-0.007 (3)	-0.003 (3)	0.004 (3)
N3	0.045 (3)	0.042 (3)	0.044 (3)	-0.006 (2)	0.009 (3)	0.007 (2)
N4	0.048 (3)	0.044 (3)	0.049 (3)	-0.004 (3)	0.008 (3)	0.012 (3)
N5	0.045 (3)	0.031 (3)	0.043 (3)	-0.002 (2)	0.007 (2)	0.004 (2)
N6	0.043 (3)	0.030 (3)	0.049 (3)	0.001 (2)	0.012 (3)	0.003 (2)
C2	0.040 (4)	0.054 (4)	0.051 (4)	-0.002 (3)	0.015 (3)	0.002 (3)
C3	0.051 (5)	0.087 (6)	0.052 (5)	0.000 (4)	0.009 (4)	0.017 (4)
C4	0.069 (6)	0.097 (7)	0.041 (4)	0.037 (5)	0.014 (4)	0.004 (4)
C5	0.075 (6)	0.062 (5)	0.036 (4)	0.026 (4)	0.006 (4)	0.010 (3)
C6	0.116 (8)	0.074 (6)	0.038 (4)	0.056 (6)	0.014 (5)	-0.006 (4)
C7	0.130 (9)	0.054 (5)	0.067 (6)	0.026 (6)	-0.008 (6)	-0.008 (4)
C8	0.097 (6)	0.043 (4)	0.051 (5)	0.016 (4)	0.005 (4)	-0.011 (3)
C9	0.131 (9)	0.038 (5)	0.082 (7)	-0.009 (5)	-0.015 (6)	-0.006 (4)
C10	0.107 (8)	0.054 (5)	0.101 (8)	-0.024 (5)	0.003 (6)	-0.003 (5)
C11	0.073 (5)	0.049 (4)	0.064 (5)	-0.016 (4)	0.005 (4)	0.001 (4)
C12	0.056 (4)	0.044 (4)	0.033 (4)	0.016 (3)	0.006 (3)	0.005 (3)
C13	0.066 (5)	0.043 (4)	0.038 (4)	0.006 (3)	0.000 (3)	0.005 (3)
C14	0.052 (4)	0.058 (4)	0.043 (4)	-0.003 (3)	0.002 (3)	0.006 (3)
C15	0.068 (6)	0.072 (5)	0.052 (5)	-0.004 (4)	0.014 (4)	0.013 (4)
C16	0.073 (6)	0.087 (6)	0.045 (5)	-0.001 (5)	-0.010 (4)	0.015 (4)
C17	0.055 (5)	0.088 (6)	0.053 (5)	-0.005 (4)	0.002 (4)	0.009 (4)
C18	0.053 (5)	0.141 (9)	0.066 (6)	0.002 (5)	-0.010 (4)	0.025 (6)
C19	0.039 (5)	0.160 (10)	0.078 (7)	0.008 (5)	0.003 (4)	0.023 (6)
C20	0.041 (5)	0.097 (6)	0.068 (6)	0.005 (4)	0.009 (4)	0.019 (4)
C21	0.044 (5)	0.116 (8)	0.089 (7)	0.008 (5)	0.020 (5)	0.013 (6)
C22	0.060 (6)	0.103 (7)	0.070 (6)	-0.001 (5)	0.025 (5)	0.020 (5)
C23	0.065 (5)	0.066 (5)	0.056 (5)	0.001 (4)	0.008 (4)	0.014 (4)
C24	0.042 (4)	0.049 (4)	0.053 (4)	-0.008 (3)	0.000 (3)	0.006 (3)
C25	0.042 (4)	0.056 (4)	0.047 (4)	-0.005 (3)	0.005 (3)	0.007 (3)
C26	0.057 (4)	0.034 (4)	0.055 (4)	0.001 (3)	0.010 (4)	0.004 (3)
C27	0.069 (5)	0.046 (4)	0.059 (5)	-0.001 (4)	0.004 (4)	0.010 (4)
C28	0.072 (5)	0.062 (5)	0.046 (4)	0.001 (4)	0.003 (4)	0.019 (4)
C29	0.063 (5)	0.054 (4)	0.041 (4)	0.004 (4)	0.007 (3)	-0.003 (3)
C30	0.104 (7)	0.069 (6)	0.051 (5)	0.008 (5)	-0.019 (5)	-0.005 (4)
C31	0.125 (8)	0.053 (5)	0.048 (5)	-0.003 (5)	-0.010 (5)	-0.019 (4)
C32	0.079 (5)	0.038 (4)	0.048 (4)	0.000 (4)	0.020 (4)	-0.005 (3)
C33	0.085 (6)	0.037 (4)	0.067 (5)	-0.005 (4)	0.018 (4)	-0.009 (3)
C34	0.076 (5)	0.031 (4)	0.068 (5)	0.010 (3)	0.009 (4)	0.003 (3)
C35	0.058 (4)	0.035 (4)	0.054 (4)	0.005 (3)	0.008 (3)	0.006 (3)
C36	0.045 (4)	0.034 (3)	0.041 (4)	0.000 (3)	0.011 (3)	0.001 (3)
C37	0.046 (4)	0.040 (4)	0.036 (4)	0.001 (3)	0.006 (3)	-0.002 (3)
C51	0.12 (2)	0.16 (2)	0.105 (18)	0.066 (18)	0.018 (15)	-0.036 (16)

supplementary materials

C52	0.12 (3)	0.45 (8)	0.28 (5)	-0.07 (4)	-0.10 (3)	0.30 (6)
O2	0.18 (2)	0.147 (18)	0.21 (2)	0.007 (16)	-0.078 (18)	-0.017 (16)

Geometric parameters (Å, °)

Fe1—N4	1.958 (6)	C14—H26	0.9300
Fe1—N2	1.970 (5)	C15—C16	1.356 (10)
Fe1—N6	1.979 (5)	C15—H45	0.9300
Fe1—N1	1.980 (5)	C16—C17	1.418 (10)
Fe1—N3	1.982 (5)	C16—H20	0.9300
Fe1—N5	1.985 (5)	C17—C18	1.419 (11)
Fe2—O1	1.735 (5)	C17—C24	1.425 (9)
Fe2—C14	2.204 (3)	C18—C19	1.335 (11)
Fe2—C16	2.223 (2)	C18—H44	0.9300
Fe2—C15	2.231 (2)	C19—C20	1.419 (11)
Fe3—O1	1.756 (5)	C19—H50	0.9300
Fe3—C12	2.213 (2)	C20—C21	1.405 (11)
Fe3—C13	2.227 (2)	C20—C25	1.408 (10)
Fe3—C11	2.227 (2)	C21—C22	1.355 (11)
N1—C2	1.318 (8)	C21—H46	0.9300
N1—C12	1.367 (8)	C22—C23	1.399 (11)
N2—C11	1.319 (9)	C22—H35	0.9300
N2—C13	1.379 (9)	C23—H43	0.9300
N3—C14	1.323 (8)	C24—C25	1.415 (9)
N3—C24	1.353 (8)	C26—C27	1.373 (9)
N4—C23	1.343 (8)	C26—H28	0.9300
N4—C25	1.353 (8)	C27—C28	1.337 (10)
N5—C26	1.330 (7)	C27—H18	0.9300
N5—C36	1.351 (8)	C28—C29	1.381 (9)
N6—C35	1.337 (7)	C28—H2	0.9300
N6—C37	1.346 (7)	C29—C36	1.412 (9)
C2—C3	1.411 (10)	C29—C30	1.458 (10)
C2—H27	0.9300	C30—C31	1.342 (11)
C3—C4	1.370 (11)	C30—H42	0.9300
C3—H36	0.9300	C31—C32	1.425 (10)
C4—C5	1.380 (11)	C31—H48	0.9300
C4—H33	0.9300	C32—C37	1.408 (9)
C5—C12	1.406 (10)	C32—C33	1.421 (9)
C5—C6	1.471 (11)	C33—C34	1.365 (10)
C6—C7	1.383 (13)	C33—H34	0.9300
C6—H38	0.9300	C34—C35	1.389 (9)
C7—C8	1.399 (12)	C34—H25	0.9300
C7—H47	0.9300	C35—H19	0.9300
C8—C9	1.391 (12)	C36—C37	1.420 (8)
C8—C13	1.395 (10)	C51—C52	1.487 (18)
C9—C10	1.358 (13)	C51—H51A	0.9600
C9—H32	0.9300	C51—H51B	0.9600
C10—C11	1.377 (11)	C51—H51C	0.9600
C10—H49	0.9300	C52—O2	1.440 (19)

C11—H37	0.9300	C52—H52A	0.9700
C12—C13	1.420 (10)	C52—H52B	0.9700
C14—C15	1.387 (9)	O2—H2A	0.8200
N4—Fe1—N2	91.0 (2)	C15—C14—H26	118.4
N4—Fe1—N6	90.9 (2)	C16—C15—C14	120.0 (7)
N2—Fe1—N6	174.7 (2)	C16—C15—H45	120.0
N4—Fe1—N1	172.7 (2)	C14—C15—H45	120.0
N2—Fe1—N1	83.8 (2)	C15—C16—C17	119.8 (7)
N6—Fe1—N1	94.7 (2)	C15—C16—H20	120.1
N4—Fe1—N3	82.8 (2)	C17—C16—H20	120.1
N2—Fe1—N3	89.9 (2)	C18—C17—C16	125.7 (7)
N6—Fe1—N3	95.3 (2)	C18—C17—C24	118.4 (7)
N1—Fe1—N3	92.1 (2)	C16—C17—C24	115.8 (7)
N4—Fe1—N5	95.0 (2)	C19—C18—C17	121.0 (7)
N2—Fe1—N5	92.8 (2)	C19—C18—H44	119.5
N6—Fe1—N5	82.1 (2)	C17—C18—H44	119.5
N1—Fe1—N5	90.4 (2)	C18—C19—C20	122.3 (8)
N3—Fe1—N5	176.5 (2)	C18—C19—H50	118.8
O1—Fe2—C14	112.4 (2)	C20—C19—H50	118.8
O1—Fe2—C16	107.8 (2)	C21—C20—C25	115.5 (7)
C14—Fe2—C16	109.44 (11)	C21—C20—C19	125.9 (7)
O1—Fe2—C15	107.4 (2)	C25—C20—C19	118.6 (7)
C14—Fe2—C15	109.35 (12)	C22—C21—C20	120.4 (7)
C16—Fe2—C15	110.45 (9)	C22—C21—H46	119.8
O1—Fe3—C12	112.5 (2)	C20—C21—H46	119.8
O1—Fe3—C13	110.9 (2)	C21—C22—C23	120.2 (8)
C12—Fe3—C13	109.50 (9)	C21—C22—H35	119.9
O1—Fe3—C11	108.7 (2)	C23—C22—H35	119.9
C12—Fe3—C11	107.12 (9)	N4—C23—C22	121.9 (7)
C13—Fe3—C11	107.94 (9)	N4—C23—H43	119.0
Fe2—O1—Fe3	160.6 (4)	C22—C23—H43	119.0
C2—N1—C12	119.9 (6)	N3—C24—C25	116.6 (6)
C2—N1—Fe1	129.0 (5)	N3—C24—C17	123.3 (7)
C12—N1—Fe1	111.1 (4)	C25—C24—C17	120.0 (6)
C11—N2—C13	115.8 (6)	N4—C25—C20	124.9 (7)
C11—N2—Fe1	131.7 (6)	N4—C25—C24	115.5 (6)
C13—N2—Fe1	112.4 (5)	C20—C25—C24	119.6 (6)
C14—N3—C24	117.9 (6)	N5—C26—C27	123.1 (6)
C14—N3—Fe1	130.4 (5)	N5—C26—H28	118.4
C24—N3—Fe1	111.7 (4)	C27—C26—H28	118.4
C23—N4—C25	117.1 (6)	C28—C27—C26	120.6 (7)
C23—N4—Fe1	129.8 (5)	C28—C27—H18	119.7
C25—N4—Fe1	113.1 (4)	C26—C27—H18	119.7
C26—N5—C36	116.7 (5)	C27—C28—C29	119.4 (7)
C26—N5—Fe1	130.4 (4)	C27—C28—H2	120.3
C36—N5—Fe1	112.8 (4)	C29—C28—H2	120.3
C35—N6—C37	117.4 (5)	C28—C29—C36	117.3 (6)
C35—N6—Fe1	129.5 (4)	C28—C29—C30	126.3 (7)
C37—N6—Fe1	113.1 (4)	C36—C29—C30	116.4 (6)

supplementary materials

N1—C2—C3	120.7 (7)	C31—C30—C29	122.1 (7)
N1—C2—H27	119.7	C31—C30—H42	118.9
C3—C2—H27	119.7	C29—C30—H42	118.9
C4—C3—C2	120.2 (8)	C30—C31—C32	121.2 (7)
C4—C3—H36	119.9	C30—C31—H48	119.4
C2—C3—H36	119.9	C32—C31—H48	119.4
C3—C4—C5	119.5 (7)	C37—C32—C33	116.6 (6)
C3—C4—H33	120.2	C37—C32—C31	118.8 (6)
C5—C4—H33	120.3	C33—C32—C31	124.5 (7)
C4—C5—C12	118.2 (7)	C34—C33—C32	118.8 (6)
C4—C5—C6	124.9 (8)	C34—C33—H34	120.6
C12—C5—C6	116.9 (8)	C32—C33—H34	120.6
C7—C6—C5	119.9 (8)	C33—C34—C35	120.2 (6)
C7—C6—H38	120.1	C33—C34—H25	119.9
C5—C6—H38	120.1	C35—C34—H25	119.9
C6—C7—C8	122.9 (8)	N6—C35—C34	123.0 (6)
C6—C7—H47	118.6	N6—C35—H19	118.5
C8—C7—H47	118.6	C34—C35—H19	118.5
C9—C8—C13	117.2 (8)	N5—C36—C29	122.8 (6)
C9—C8—C7	125.1 (8)	N5—C36—C37	115.9 (5)
C13—C8—C7	117.7 (9)	C29—C36—C37	121.3 (6)
C10—C9—C8	120.2 (8)	N6—C37—C32	124.0 (6)
C10—C9—H32	119.9	N6—C37—C36	116.1 (5)
C8—C9—H32	119.9	C32—C37—C36	119.9 (6)
C9—C10—C11	118.5 (9)	C52—C51—H51A	109.3
C9—C10—H49	120.7	C52—C51—H51B	109.6
C11—C10—H49	120.7	H51A—C51—H51B	109.5
N2—C11—C10	125.0 (9)	C52—C51—H51C	109.5
N2—C11—H37	117.5	H51A—C51—H51C	109.5
C10—C11—H37	117.5	H51B—C51—H51C	109.5
N1—C12—C5	121.5 (7)	O2—C52—C51	109.5 (17)
N1—C12—C13	117.7 (6)	O2—C52—H52A	109.9
C5—C12—C13	120.8 (7)	C51—C52—H52A	109.7
N2—C13—C8	123.1 (7)	O2—C52—H52B	109.8
N2—C13—C12	115.0 (6)	C51—C52—H52B	109.7
C8—C13—C12	121.9 (7)	H52A—C52—H52B	108.2
N3—C14—C15	123.2 (7)	C52—O2—H2A	109.5
N3—C14—H26	118.4		

Fig. 1

