Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diaquachlorido(4-hydroxypyridine-2,6-carboxylato-κ^3N,O,O')iron(III) 18-crown-6 dihydrate

Guo-Wei Zhou,[a] A-Qing Wu,[a,b] Ming-Sheng Wang,[a] Guo-Cong Guo[a]* and Jin-Shun Huang[a]

[a]State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China, and [b]Graduate School, Chinese Academy of Sciences, Beijing 100039, People's Republic of China
Correspondence e-mail: gcguo@ms.fjirsm.ac.cn

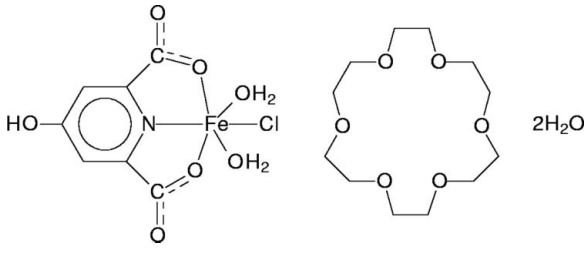
Received 2 August 2007; accepted 27 August 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.133; data-to-parameter ratio = 13.2.

In the title compound, $[Fe(C_7H_3NO_5)Cl(H_2O)_2]\cdot C_{12}H_{24}O_6\cdot 2H_2O$ or $[Fe(HChel)Cl(H_2O)_2]$.(18-crown-6)$\cdot 2H_2O$, where H_3Chel is 4-hydroxypyridine-2,6-dicarboxylic (chelidamic) acid, the Fe^{III} atom exhibits a distorted octahedral geometry coordinated by two O atoms and one N atom from the tridentate chelidamate ligand, one Cl atom and two water molecules. O—H···O hydrogen bonds from the coordinated water molecules to the O atoms of the crown ether form one-dimensional hydrogen-bonded chains with alternating $[Fe(HChel)Cl(H_2O)_2]$ and 18-crown-6 units. The solvent water molecules lie between these chains, accepting O—H···O hydrogen bonds from the hydroxyl groups of the chelidamate ligands, and forming O—H···O hydrogen bonds with the noncoordinated O atoms of the carboxylate groups.

Related literature

For the crystal structure of $[Fe(HChel)Cl(H_2O)_2]$, see: Laine *et al.* (1995). For other literature related to the use of chelidamic acid as a ligand, see: Cline *et al.* (1979); Hall *et al.* (2000); Ng (1998, 1999); Riegel (1926); Thich *et al.* (1976); Yang *et al.* (2002).



Experimental

Crystal data

$[Fe(C_7H_3NO_5)Cl(H_2O)_2]\cdot C_{12}H_{24}O_6\cdot 2H_2O$
M_r = 608.78
Triclinic, $P\overline{1}$
a = 7.7355 (9) Å
b = 10.4503 (17) Å
c = 17.185 (3) Å
α = 77.971 (12)°
β = 89.779 (11)°
γ = 81.075 (11)°
V = 1341.7 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 293 (2) K
0.30 × 0.25 × 0.05 mm

Data collection

Rigaku AFC-7R diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.851, T_{max} = 0.964
5197 measured reflections
4738 independent reflections
2831 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
3 standard reflections
every 200 reflections
intensity decay: 2.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.133
S = 1.03
4738 reflections
358 parameters
26 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.89 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1C···O11^{i}	0.96 (1)	1.91 (1)	2.822 (3)	158.9 (11)
O1W—H1C···O12^{i}	0.96 (1)	2.47 (1)	2.937 (3)	109.8 (11)
O1W—H1D···O13^{i}	0.95 (1)	1.79 (1)	2.725 (3)	165.5 (13)
O2W—H2D···O11	0.95 (1)	2.30 (1)	2.926 (3)	122.8 (9)
O2W—H2C···O14	0.95 (1)	2.10 (1)	2.996 (3)	156.2 (14)
O2W—H2C···O15	0.95 (1)	2.28 (1)	2.911 (3)	123.5 (14)
O2W—H2D···O16	0.95 (1)	2.02 (1)	2.892 (3)	151.6 (14)
O3W—H3D···O1	0.96 (1)	1.91 (1)	2.826 (3)	159.4 (11)
O4W—H4C···O3^{ii}	0.96 (1)	1.76 (1)	2.717 (3)	175 (2)
O5—H5C···O4W	0.82	1.77	2.591 (3)	177

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

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support from the Natural Science Foundation of Fujian Province (grant Nos. E0510029, 2006J0275, 2004J039).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2229).

References

Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Cline, S. J., Kallesoe, S., Pedersen, E. & Hodgson, D. J. (1979). *Inorg. Chem.* **18**, 796–801.
Hall, A. K., Harrowfield, J. M., Skelton, B. W. & White, A. H. (2000). *Acta Cryst.* C**56**, 448–450.
Laine, P., Gourdon, A. & Launay, J.-P. (1995). *Inorg. Chem.* **34**, 5156–5165.

 doi:10.1107/S1600536807041955

Molecular Structure Corporation (1994). *MSC/AFC Diffractometer Control Software*. MSC, The Woodlands, Texas, USA.
Ng, S. W. (1998). *Z. Kristallogr.* **213**, 421–426.
Ng, S. W. (1999). *J. Organomet. Chem.* **585**, 12–17.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Riegel, R. (1926). *J. Am. Chem. Soc.* **48**, 1334–1345.
Rigaku/MSC (2002). *CrystalStructure*. Version 3.10. MSC, The Woodlands, Texas, USA.
Thich, J. A., Ou, C. C., Powers, D., Vasiliou, B., Mastropaolo, D., Potenza, J. A. & Schugar, H. J. (1976). *J. Am. Chem. Soc.* **98**, 1425–1433.
Yang, L., Cour, A. L., Anderson, O. P. & Crans, D. C. (2002). *Inorg. Chem.* **24**, 6322–6331.

supplementary materials

Acta Cryst. (2007). E**63**, m2463-m2464 [**doi:10.1107/S1600536807041955**]

Diaquachlorido(4-hydroxypyridine-2,6-carboxylato-κ^3N,O,O')iron(III) 18-crown-6 dihydrate

G.-W. Zhou, A.-Q. Wu, M.-S. Wang, G.-C. Guo and J.-S. Huang

Comment

Since the first report of chelidamic acid (4-hydroxypyridine-2,6-dicarboxylic acid) in 1926 (Riegel, 1926), its coordination chemistry has been extensively investigated. Much of this work focuses on the design and synthesis of polymers in which chelidamic acid acts as a ligand (Ng, 1998, 1999; Riegel, 1926; Hall *et al.*, 2000; Cline *et al.*, 1979; Yang *et al.*, 2002; Thich *et al.*, 1976, Laine *et al.*, 1995). Herein, we report the synthesis and crystal structure of the title compound, which is an Fe^{III} complex in which chelidamic acid acts as a tridentate chelating ligand.

The asymmetric unit (Fig. 1) consists of one Fe^{III} atom coordinated by one chelidamic acid ligand, one Cl atom and two coordinated water molecules, as well as one 18-crown-6 molecule and two lattice water molecules. The coordination environment of Fe^{III} comprises two carboxylic O atoms (Fe1—O2 = 2.0248 (17), Fe1—O4 = 2.0507 (16) Å) and one N atom (Fe1—N1 = 2.055 (2) Å) from the tridentate chelated chelidamic acid ligand, one Cl atom (Fe1—Cl1 = 2.2522 (9) Å) lying in the equatorial plane, and two water molecules (Fe1—O1W = 2.0531 (19), Fe1—O2W = 2.0613 (18) Å) in the axial positions, forming a distorted octahedral geometry. Due to the monodentate coordination mode of both carboxylate groups of the chelidamic acid ligand, the C—O bond distances of the coordinated O atoms (C26—O2 = 1.293 (3) and C27—O4 = 1.280 (3) Å) are slightly longer than the C—O bond distances of the uncoordinated O atoms (C26—O1 = 1.218 (3) and C27—O3 = 1.233 (3) Å).

The coordinated water molecules, O1W and O2W, act as hydrogen bond donors to form O—H···O hydrogen bonds with six O atoms from 18-crown-6 molecules (Table 1). These hydrogen bonds link alternate Fe^{III} complexes and 18-crown-6 molecules into chains running along the *a* direction (Fig. 2). The lattice water molecules, O3W and O4W, lie between chains, accepting an O—H···O hydrogen bond from the hydroxyl group of the chelidamic acid ligand, and forming O—H···O hydrogen bonds to the non-coordinated O atoms of the carboxyl groups (Table 1).

Experimental

A mixture of $FeCl_3·6H_2O$ (0.068 g, 0.25 mmol), 18-crown-6 (0.066 g, 0.25 mmol) and H_3Chel (50 mg, 0.25 mmol) was dissolved in 20 ml of a mixed ethanol:water (1:1) solvent. After dropping 1–2 drops KOH (1*M*) into the solution and stirring for about 4 h, the solution was filtered and the filtrate was allowed to stand at room temperature. Yellow–brown crystals of the title compound were obtained after 4–5 days (yield: 63%).

Refinement

H atoms bonded to C atoms and the H atom of the hydroxyl group were placed at calculated positions and allowed to ride during subsequent refinement with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$. The H atoms of the water molecules were located

in difference Fourier maps and refined with O—H distances restrained to a target value of 0.96 (1) Å, and with U_{iso}(H) = 1.5U_{eq}(O). The anisotropic displacement parameters of C1, C2 and C12 were restrained to approximate isotropic behaviour.

Figures

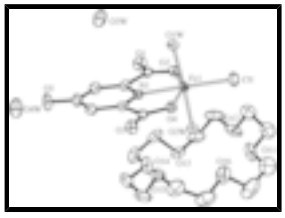


Fig. 1. Molecular structure of the title compounds showing displacement ellipsoids at 30% probability. H atoms are omitted.

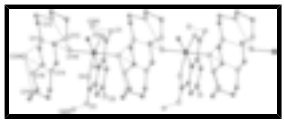


Fig. 2. One-dimensional chain formed by O—H···O hydrogen bonds between the coordinated water molecules and the O atoms of 18-crown-6.

Diaquachlorido(4-hydroxypyridine-2,6-carboxylato-κ^3*N*,*O*,*O*')iron(III) 18-crown-6 dihydrate

Crystal data

$[Fe(C_7H_3NO_5)Cl(H_2O)_2]\cdot C_{12}H_{24}O_6\cdot 2H_2O$	$Z = 2$
$M_r = 608.78$	$F_{000} = 638$
Triclinic, $P\bar{1}$	$D_x = 1.507$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.7355$ (9) Å	Cell parameters from 20 reflections
$b = 10.4503$ (17) Å	$\theta = 3.0$–25.0º
$c = 17.185$ (3) Å	$\mu = 0.73$ mm^{-1}
$\alpha = 77.971$ (12)º	$T = 293$ (2) K
$\beta = 89.779$ (11)º	Plate, yellow
$\gamma = 81.075$ (11)º	0.30 × 0.25 × 0.05 mm
$V = 1341.7$ (4) Å^3	

Data collection

Rigaku AFC-7R diffractometer	$R_{int} = 0.037$
Radiation source: rotating-anode generator	$\theta_{max} = 25.0$º
Monochromator: graphite	$\theta_{min} = 3.0$º
$T = 293$(2) K	$h = -2\rightarrow 9$
ω/2θ scans	$k = -12\rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -20\rightarrow 20$
$T_{min} = 0.851$, $T_{max} = 0.964$	3 standard reflections
5197 measured reflections	every 200 reflections
4738 independent reflections	intensity decay: 2.4%
2831 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.133$	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.002$
4738 reflections	$\Delta\rho_{max} = 0.89$ e Å^{-3}
358 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
26 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

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}
Fe1	0.77559 (5)	0.72036 (4)	0.69984 (2)	0.03157 (11)
N1	0.7714 (3)	0.78986 (18)	0.80335 (11)	0.0299 (6)
O1	0.6479 (3)	1.11514 (18)	0.69990 (12)	0.0527 (7)
O2	0.7032 (2)	0.91875 (16)	0.66429 (10)	0.0371 (5)
O3	0.9002 (3)	0.47201 (17)	0.91676 (10)	0.0454 (6)
O4	0.8442 (2)	0.55333 (16)	0.78711 (9)	0.0350 (5)
O5	0.8038 (3)	0.93481 (19)	1.00395 (11)	0.0644 (7)
H5C	0.8646	0.8809	1.0385	0.097*
O11	0.2884 (3)	0.6287 (2)	0.59803 (12)	0.0710 (8)
O12	0.1925 (3)	0.8967 (2)	0.55633 (13)	0.0687 (8)
O13	0.0940 (3)	0.99078 (18)	0.69714 (12)	0.0511 (6)
O14	0.2819 (3)	0.8108 (2)	0.83044 (12)	0.0670 (8)
O15	0.4638 (3)	0.5491 (2)	0.86997 (12)	0.0583 (7)
O16	0.4159 (3)	0.4420 (2)	0.73193 (14)	0.0788 (9)
O1W	1.0349 (2)	0.74188 (16)	0.69298 (10)	0.0385 (5)
H1D	1.0692 (18)	0.8217 (7)	0.7014 (11)	0.058*

H1C	1.0979 (17)	0.7098 (14)	0.6512 (6)	0.058*
O2W	0.5147 (2)	0.70245 (16)	0.71188 (10)	0.0378 (5)
H2C	0.4556 (17)	0.7136 (19)	0.7591 (5)	0.057*
H2D	0.4836 (19)	0.6244 (7)	0.6995 (10)	0.057*
O3W	0.8772 (3)	1.28637 (19)	0.72980 (14)	0.0771 (8)
H3D	0.7804 (10)	1.2477 (12)	0.7155 (12)	0.116*
H3C	0.857 (5)	1.3798 (7)	0.7090 (16)	0.116*
O4W	0.9945 (3)	0.77074 (19)	1.11650 (12)	0.0790 (9)
H4D	1.039 (2)	0.7788 (19)	1.1666 (5)	0.118*
H4C	1.037 (3)	0.6843 (7)	1.1074 (11)	0.118*
C1	0.3072 (5)	0.7013 (3)	0.5185 (2)	0.0790 (9)
H1A	0.2018	0.7063	0.4870	0.095*
H1B	0.4046	0.6560	0.4939	0.095*
C2	0.3384 (5)	0.8350 (3)	0.5209 (2)	0.0735 (8)
H2A	0.4444	0.8310	0.5518	0.088*
H2B	0.3515	0.8847	0.4674	0.088*
C3	0.2084 (5)	1.0275 (3)	0.5628 (2)	0.0815 (14)
H3A	0.1917	1.0869	0.5109	0.098*
H3B	0.3237	1.0299	0.5839	0.098*
C4	0.0706 (4)	1.0684 (3)	0.6177 (2)	0.0670 (12)
H4A	0.0707	1.1607	0.6192	0.080*
H4B	−0.0429	1.0613	0.5966	0.080*
C5	0.2379 (4)	1.0168 (3)	0.7389 (2)	0.0659 (11)
H5A	0.2292	1.1114	0.7352	0.079*
H5B	0.3461	0.9864	0.7149	0.079*
C6	0.2406 (5)	0.9479 (3)	0.8243 (2)	0.0684 (11)
H6A	0.3270	0.9778	0.8541	0.082*
H6B	0.1270	0.9690	0.8466	0.082*
C7	0.3131 (4)	0.7379 (3)	0.90998 (17)	0.0680 (12)
H7A	0.2208	0.7679	0.9435	0.082*
H7B	0.4236	0.7524	0.9303	0.082*
C8	0.3181 (4)	0.5953 (4)	0.9119 (2)	0.0746 (13)
H8A	0.3266	0.5462	0.9666	0.090*
H8B	0.2112	0.5817	0.8877	0.090*
C9	0.4847 (5)	0.4113 (3)	0.8672 (2)	0.0883 (15)
H9A	0.6065	0.3818	0.8575	0.106*
H9B	0.4584	0.3619	0.9190	0.106*
C10	0.3792 (6)	0.3788 (4)	0.8087 (3)	0.0997 (17)
H10A	0.3999	0.2837	0.8128	0.120*
H10B	0.2567	0.4049	0.8188	0.120*
C11	0.2845 (7)	0.4236 (4)	0.6766 (3)	0.132 (2)
H11A	0.2942	0.3301	0.6763	0.159*
H11B	0.1689	0.4524	0.6948	0.159*
C12	0.3019 (6)	0.4921 (4)	0.6013 (3)	0.1035 (9)
H12A	0.2119	0.4767	0.5668	0.124*
H12B	0.4148	0.4599	0.5819	0.124*
C21	0.7340 (3)	0.9193 (2)	0.79981 (15)	0.0332 (8)
C22	0.7404 (4)	0.9703 (3)	0.86702 (16)	0.0418 (8)
H22A	0.7102	1.0608	0.8644	0.050*

C23	0.7931 (4)	0.8834 (3)	0.93964 (16)	0.0428 (8)
C24	0.8338 (3)	0.7468 (3)	0.94158 (16)	0.0379 (8)
H24A	0.8694	0.6869	0.9889	0.046*
C25	0.8195 (3)	0.7041 (2)	0.87178 (14)	0.0308 (7)
C26	0.6881 (3)	0.9950 (3)	0.71509 (15)	0.0353 (8)
C27	0.8577 (3)	0.5646 (2)	0.85950 (15)	0.0335 (8)
Cl1	0.79064 (11)	0.62857 (8)	0.59200 (4)	0.0548 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0363 (2)	0.03038 (18)	0.02747 (18)	−0.00668 (16)	0.00149 (16)	−0.00377 (14)
N1	0.0304 (12)	0.0270 (10)	0.0321 (11)	−0.0072 (10)	0.0011 (10)	−0.0037 (9)
O1	0.0642 (14)	0.0284 (10)	0.0591 (13)	−0.0015 (10)	−0.0062 (11)	0.0014 (9)
O2	0.0456 (11)	0.0331 (9)	0.0285 (9)	−0.0073 (9)	−0.0025 (9)	0.0039 (8)
O3	0.0599 (13)	0.0338 (10)	0.0363 (10)	0.0012 (10)	−0.0060 (10)	0.0001 (8)
O4	0.0454 (11)	0.0307 (9)	0.0284 (9)	−0.0065 (8)	0.0017 (9)	−0.0048 (7)
O5	0.0930 (17)	0.0570 (12)	0.0437 (11)	0.0088 (12)	−0.0128 (12)	−0.0262 (9)
O11	0.1046 (19)	0.0608 (13)	0.0419 (11)	0.0133 (14)	−0.0043 (13)	−0.0167 (10)
O12	0.0520 (14)	0.0733 (15)	0.0617 (14)	0.0098 (12)	0.0219 (11)	0.0141 (12)
O13	0.0422 (12)	0.0388 (10)	0.0701 (13)	−0.0110 (9)	−0.0057 (11)	−0.0027 (10)
O14	0.0798 (16)	0.0673 (13)	0.0508 (12)	0.0121 (13)	−0.0114 (12)	−0.0227 (10)
O15	0.0592 (14)	0.0603 (13)	0.0507 (12)	−0.0151 (11)	0.0034 (11)	0.0028 (10)
O16	0.118 (2)	0.0471 (13)	0.0683 (15)	−0.0159 (14)	−0.0183 (15)	−0.0026 (11)
O1W	0.0381 (11)	0.0419 (10)	0.0397 (10)	−0.0129 (9)	0.0096 (9)	−0.0132 (8)
O2W	0.0397 (11)	0.0416 (10)	0.0363 (10)	−0.0148 (9)	0.0087 (9)	−0.0117 (8)
O3W	0.0901 (18)	0.0504 (13)	0.0928 (16)	−0.0068 (13)	−0.0253 (15)	−0.0219 (12)
O4W	0.125 (2)	0.0478 (12)	0.0605 (13)	0.0087 (14)	−0.0260 (14)	−0.0191 (10)
C1	0.0788 (13)	0.0870 (13)	0.0683 (13)	−0.0001 (10)	0.0039 (11)	−0.0195 (10)
C2	0.0690 (13)	0.0797 (13)	0.0650 (12)	−0.0052 (10)	0.0064 (10)	−0.0046 (10)
C3	0.066 (2)	0.071 (2)	0.089 (3)	−0.0237 (19)	0.014 (2)	0.034 (2)
C4	0.050 (2)	0.0480 (19)	0.089 (3)	−0.0069 (17)	−0.002 (2)	0.0174 (18)
C5	0.0447 (19)	0.0536 (18)	0.105 (3)	−0.0154 (16)	−0.0110 (19)	−0.0242 (18)
C6	0.053 (2)	0.073 (2)	0.087 (2)	0.0008 (18)	−0.0147 (19)	−0.0429 (17)
C7	0.046 (2)	0.115 (3)	0.0381 (16)	0.010 (2)	0.0020 (16)	−0.0217 (18)
C8	0.052 (2)	0.100 (3)	0.064 (2)	−0.020 (2)	0.0172 (19)	0.006 (2)
C9	0.110 (3)	0.065 (2)	0.080 (3)	−0.035 (2)	−0.029 (2)	0.023 (2)
C10	0.103 (3)	0.061 (2)	0.125 (4)	−0.027 (2)	−0.016 (3)	0.014 (2)
C11	0.197 (5)	0.056 (2)	0.163 (4)	−0.060 (3)	−0.018 (4)	−0.037 (2)
C12	0.1081 (14)	0.1023 (14)	0.1024 (14)	−0.0141 (11)	−0.0015 (11)	−0.0282 (11)
C21	0.0346 (16)	0.0259 (13)	0.0377 (14)	−0.0023 (12)	−0.0029 (13)	−0.0050 (11)
C22	0.0426 (17)	0.0344 (14)	0.0493 (16)	−0.0021 (13)	−0.0008 (14)	−0.0139 (12)
C23	0.0463 (18)	0.0487 (15)	0.0378 (14)	−0.0039 (14)	−0.0020 (14)	−0.0218 (12)
C24	0.0361 (16)	0.0407 (15)	0.0355 (14)	−0.0040 (13)	0.0004 (13)	−0.0060 (12)
C25	0.0307 (15)	0.0320 (13)	0.0302 (13)	−0.0081 (12)	0.0036 (12)	−0.0053 (11)
C26	0.0300 (15)	0.0384 (14)	0.0363 (14)	−0.0081 (13)	−0.0052 (13)	−0.0033 (12)
C27	0.0302 (15)	0.0355 (14)	0.0338 (14)	−0.0057 (12)	0.0000 (12)	−0.0044 (12)
Cl1	0.0585 (5)	0.0694 (5)	0.0427 (4)	−0.0082 (4)	0.0020 (4)	−0.0275 (3)

Geometric parameters (Å, °)

Fe1—Cl1	2.2522 (9)	C1—H1B	0.970
Fe1—N1	2.055 (2)	C2—H2A	0.970
Fe1—O2	2.0248 (17)	C2—H2B	0.970
Fe1—O4	2.0507 (16)	C3—C4	1.492 (5)
Fe1—O1W	2.0531 (19)	C3—H3A	0.970
Fe1—O2W	2.0613 (18)	C3—H3B	0.970
N1—C21	1.327 (3)	C4—H4A	0.970
N1—C25	1.336 (3)	C4—H4B	0.970
O1—C26	1.218 (3)	C5—C6	1.493 (5)
O2—C26	1.293 (3)	C5—H5A	0.970
O3—C27	1.233 (3)	C5—H5B	0.970
O4—C27	1.280 (3)	C6—H6A	0.970
O5—C23	1.334 (3)	C6—H6B	0.970
O5—H5C	0.820	C7—C8	1.478 (5)
O11—C12	1.404 (5)	C7—H7A	0.970
O11—C1	1.436 (4)	C7—H7B	0.970
O12—C2	1.411 (4)	C8—H8A	0.970
O12—C3	1.419 (4)	C8—H8B	0.970
O13—C5	1.417 (4)	C9—C10	1.423 (5)
O13—C4	1.432 (4)	C9—H9A	0.970
O14—C6	1.401 (4)	C9—H9B	0.970
O14—C7	1.420 (3)	C10—H10A	0.970
O15—C8	1.411 (4)	C10—H10B	0.970
O15—C9	1.435 (4)	C11—C12	1.359 (6)
O16—C10	1.396 (5)	C11—H11A	0.970
O16—C11	1.456 (5)	C11—H11B	0.970
O1W—H1D	0.96 (1)	C12—H12A	0.970
O1W—H1C	0.96 (1)	C12—H12B	0.970
O2W—H2C	0.95 (1)	C21—C22	1.373 (4)
O2W—H2D	0.95 (1)	C21—C26	1.519 (3)
O3W—H3D	0.96 (1)	C22—C23	1.402 (4)
O3W—H3C	0.96 (1)	C22—H22A	0.930
O4W—H4D	0.95 (1)	C23—C24	1.406 (4)
O4W—H4C	0.96 (1)	C24—C25	1.374 (4)
C1—C2	1.463 (5)	C24—H24A	0.930
C1—H1A	0.970	C25—C27	1.501 (4)
O2—Fe1—O4	151.36 (7)	H5A—C5—H5B	108.1
O2—Fe1—O1W	90.60 (7)	O14—C6—C5	109.6 (3)
O4—Fe1—O1W	88.71 (7)	O14—C6—H6A	109.8
O2—Fe1—N1	75.58 (7)	C5—C6—H6A	109.8
O4—Fe1—N1	75.79 (7)	O14—C6—H6B	109.8
O1W—Fe1—N1	87.38 (8)	C5—C6—H6B	109.8
O2—Fe1—O2W	88.62 (7)	H6A—C6—H6B	108.2
O4—Fe1—O2W	90.64 (7)	O14—C7—C8	109.4 (3)
O1W—Fe1—O2W	177.12 (7)	O14—C7—H7A	109.8
N1—Fe1—O2W	89.73 (8)	C8—C7—H7A	109.8

O2—Fe1—Cl1	108.88 (6)	O14—C7—H7B	109.8
O4—Fe1—Cl1	99.76 (6)	C8—C7—H7B	109.8
O1W—Fe1—Cl1	91.69 (6)	H7A—C7—H7B	108.2
N1—Fe1—Cl1	175.46 (6)	O15—C8—C7	109.5 (3)
O2W—Fe1—Cl1	91.19 (6)	O15—C8—H8A	109.8
C21—N1—C25	121.7 (2)	C7—C8—H8A	109.8
C21—N1—Fe1	119.30 (16)	O15—C8—H8B	109.8
C25—N1—Fe1	118.83 (17)	C7—C8—H8B	109.8
C26—O2—Fe1	121.08 (15)	H8A—C8—H8B	108.2
C27—O4—Fe1	119.30 (15)	C10—C9—O15	115.6 (3)
C23—O5—H5C	109.5	C10—C9—H9A	108.4
C12—O11—C1	111.8 (3)	O15—C9—H9A	108.4
C2—O12—C3	112.6 (3)	C10—C9—H9B	108.4
C5—O13—C4	113.0 (2)	O15—C9—H9B	108.4
C6—O14—C7	113.6 (3)	H9A—C9—H9B	107.4
C8—O15—C9	115.4 (3)	O16—C10—C9	111.5 (3)
C10—O16—C11	108.7 (3)	O16—C10—H10A	109.3
Fe1—O1W—H1D	119.4 (9)	C9—C10—H10A	109.3
Fe1—O1W—H1C	116.3 (9)	O16—C10—H10B	109.3
H1D—O1W—H1C	112.0 (15)	C9—C10—H10B	109.3
Fe1—O2W—H2C	121.2 (10)	H10A—C10—H10B	108.0
Fe1—O2W—H2D	115.1 (9)	C12—C11—O16	112.4 (4)
H2C—O2W—H2D	105.9 (16)	C12—C11—H11A	109.1
H3D—O3W—H3C	110 (2)	O16—C11—H11A	109.1
H4D—O4W—H4C	109.6 (18)	C12—C11—H11B	109.1
O11—C1—C2	109.7 (3)	O16—C11—H11B	109.1
O11—C1—H1A	109.7	H11A—C11—H11B	107.9
C2—C1—H1A	109.7	C11—C12—O11	111.7 (4)
O11—C1—H1B	109.7	C11—C12—H12A	109.3
C2—C1—H1B	109.7	O11—C12—H12A	109.3
H1A—C1—H1B	108.2	C11—C12—H12B	109.3
O12—C2—C1	107.3 (3)	O11—C12—H12B	109.3
O12—C2—H2A	110.2	H12A—C12—H12B	107.9
C1—C2—H2A	110.2	N1—C21—C22	121.1 (2)
O12—C2—H2B	110.2	N1—C21—C26	111.3 (2)
C1—C2—H2B	110.2	C22—C21—C26	127.6 (2)
H2A—C2—H2B	108.5	C21—C22—C23	118.7 (2)
O12—C3—C4	107.0 (3)	C21—C22—H22A	120.6
O12—C3—H3A	110.3	C23—C22—H22A	120.6
C4—C3—H3A	110.3	O5—C23—C22	118.1 (2)
O12—C3—H3B	110.3	O5—C23—C24	122.9 (2)
C4—C3—H3B	110.3	C22—C23—C24	119.0 (3)
H3A—C3—H3B	108.6	C25—C24—C23	118.3 (2)
O13—C4—C3	113.5 (2)	C25—C24—H24A	120.8
O13—C4—H4A	108.9	C23—C24—H24A	120.8
C3—C4—H4A	108.9	N1—C25—C24	121.2 (2)
O13—C4—H4B	108.9	N1—C25—C27	111.2 (2)
C3—C4—H4B	108.9	C24—C25—C27	127.6 (2)
H4A—C4—H4B	107.7	O1—C26—O2	126.3 (2)

O13—C5—C6	110.5 (3)	O1—C26—C21	121.0 (3)
O13—C5—H5A	109.6	O2—C26—C21	112.6 (2)
C6—C5—H5A	109.6	O3—C27—O4	125.2 (2)
O13—C5—H5B	109.6	O3—C27—C25	120.0 (2)
C6—C5—H5B	109.6	O4—C27—C25	114.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1C···O11^{i}	0.96 (1)	1.91 (1)	2.822 (3)	158.9 (11)
O1W—H1C···O12^{i}	0.96 (1)	2.47 (1)	2.937 (3)	109.8 (11)
O1W—H1D···O13^{i}	0.95 (1)	1.79 (1)	2.725 (3)	165.5 (13)
O2W—H2D···O11	0.95 (1)	2.30 (1)	2.926 (3)	122.8 (9)
O2W—H2C···O14	0.95 (1)	2.10 (1)	2.996 (3)	156.2 (14)
O2W—H2C···O15	0.95 (1)	2.28 (1)	2.911 (3)	123.5 (14)
O2W—H2D···O16	0.95 (1)	2.02 (1)	2.892 (3)	151.6 (14)
O3W—H3D···O1	0.96 (1)	1.91 (1)	2.826 (3)	159.4 (11)
O4W—H4C···O3^{ii}	0.96 (1)	1.76 (1)	2.717 (3)	175 (2)
O5—H5C···O4W	0.82	1.77	2.591 (3)	177

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

Fig. 1

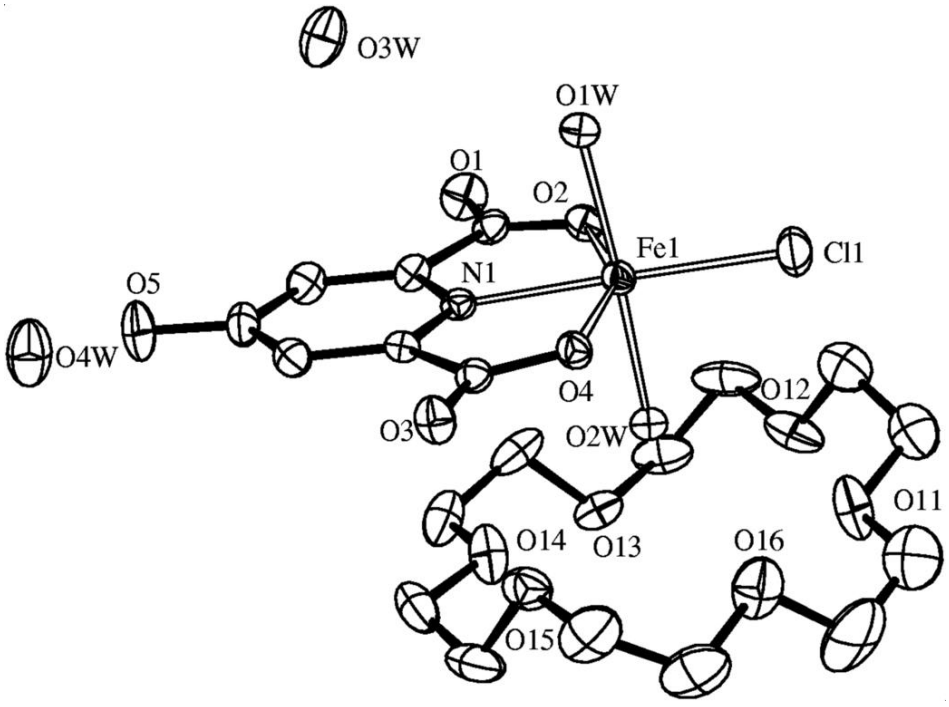


Fig. 2