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Crystal structure of aquachloridobis(2-ethoxy-6-formylphenolato-κ^2O^1,O^6)iron(III) acetonitrile hemisolvate

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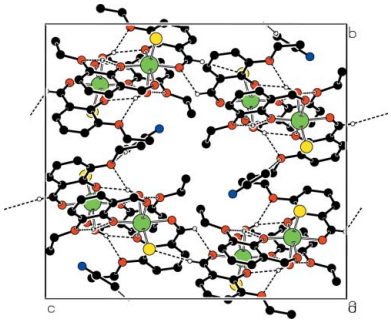
In the title compound, $[Fe(L)_2Cl(H_2O)]\cdot0.5CH_3CN$, (H*L* is 3-ethoxy-2-hydroxybenzaldehyde, $C_9H_{10}O_3$), there are two independent complex molecules and one acetonitrile solvent molecule in the asymmetric unit. In each complex molecule, the Fe^{III} ion has a distorted O_5Cl octahedral coordination environment defined by two bidentate 2-ethoxy-6-formylphenolato ligands, one Cl atom and one water molecule. In the crystal, O—H···O hydrogen bonds link the two independent molecules to form a dimer. The solvent molecule is linked to the complex molecule by a weak C—H···O hydrogen bond. Further weak C—H···O interactions along with weak C—H···Cl hydrogen bonds link the components into chains parallel to [001].

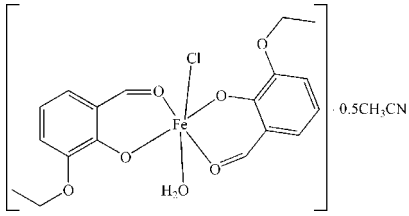
1. Chemical context

Metal complexes containing the 2-hydroxy-benzaldehyde ramification are one of the most fundamental chelating systems in coordination chemistry. Their interesting chemical and physical properties and their wide-ranging applications in numerous scientific areas have been explored widely (Han 2008; Ghelenji *et al.*, 2011; Kia *et al.*, 2010; Zhang *et al.*, 2013, 2014*a*,*b*; Zhao *et al.*, 2014). During the last few years, we have investigated the chemistry of 3*d* metal complexes of 2-hydroxy-benzaldehyde ramification ligands with the aim of preparing mono- and heterometallic polynuclear clusters or polymers (Zhang *et al.*, 2011, 2013, 2014*a*,*b*; Zhao *et al.*, 2014).

Recently, we have investigated the coordination behavior of the tridentate 2-hydroxy-benzaldehyde ramification ligand 3-ethoxy-2-hydroxy-benzaldehyde and reported two heterometallic polymers $[ZnNa(ehbd)_2(N_3)]_n$ and $[Cu_3Na_2(ehbd)_2(N_3)_6]_n$ (ehbd is the 2-hydroxy-3-ethoxy-benzaldehyde anion) (Zhang *et al.*, 2014*b*) and a cubane cluster $[Ni_4(\mu_3\text{-}OMe)_4(heb)_4(MeOH)_{1.05}(H_2O)_{2.95}]$ (heb is the 2-hydroxy-3-ethoxy-benzaldehyde anion) (Zhang *et al.*, 2011). The polymers $[ZnNa(ehbd)_2(N_3)]_n$ and $[Cu_3Na_2(ehbd)_2(N_3)_6]_n$ were prepared by room-temperature synthesis and the cubane cluster $[Ni_4(\mu_3\text{-}OMe)_4(heb)_4(MeOH)_{1.05}(H_2O)_{2.95}]$ was prepared by solvothermal synthesis. Those complexes display dominant ferromagnetic interactions between metal ions.

The title compound, $[Fe(L)_2Cl(H_2O)]\cdot0.5CH_3CN$ (H*L* = $C_9H_{10}O_3$), was prepared similarly to the cubane cluster $[Ni_4(\mu_3\text{-}OMe)_4(heb)_4(MeOH)_{1.05}(H_2O)_{2.95}]$ (Zhang *et al.*, 2011) except that $Ni(ClO_4)\cdot6H_2O$ was replaced by $FeCl_3\cdot6H_2O$ in an attempt to prepare a cubane-type iron cluster. The crystals obtained, however, were those of the title mononuclear Fe^{III} complex.





2. Structural commentary

The asymmetric unit of the title compound consists of two neutral [Fe(L)$_2$Cl(H$_2$O)] molecules and a acetonitrile solvent molecule. One of the independent molecules is shown in Fig. 1. Each Fe^{III} ion is coordinated by four O atoms from two different L^- ligands, one Cl$^-$ ion and one terminal water molecule, forming a distorted octahedral geometry. The Fe—O bond lengths are in the range 1.909 (2)–2.157 (2) Å (Table 1), while the Fe—Cl distances are 2.299 (1) and 2.301 (1) Å. The *trans*-angles at the Fe^{III} atom lie in the range 169.4 (1)–171.4 (1)°, the *cis*-angles vary from 81.6 (1) to 99.9 (1)°. The L^- ligand displays a μ_1:κ^1:κ^1 coordination mode, which is the same as that of [Ni$_4$(μ_3-OMe)$_4$(heb)$_4$(MeOH)$_{1.05}$(H$_2$O)$_{2.95}$] (Zhang *et al.*, 2011) but the coordination mode is different from the that in [Cu$_3$Na$_2$(ehbd)$_2$(N$_3$)$_6$]$_n$ (Zhang *et al.*, 2014*b*) in which the ehbd$^-$ ligand displays a pentadentate μ_3:κ^2:κ^2:κ^1 coordination mode.

3. Supramolecular features

In the crystal, O—H···O hydrogen bonds link the two independent molecules to form a dimer (Table 2, Fig. 2). All –OH group H atoms act as donors for two acceptor-O atoms, forming $R_1^2(5)$ and $R_2^2(6)$ graph-set motifs. A π–π interaction within the dimer with a $Cg1\cdots Cg2$ distance of 3.575 (1)Å is observed, where $Cg1$ and $Cg2$ are the centroids defined by ring atoms C1–C6 and C19–C24, respectively. The solvent molecule is linked to the complex molecule by a weak C—H···O hydrogen bond. Further weak C—H···O interaction along with weak C—H···Cl hydrogen bonds link the components into chains parallel to [001] (Fig. 3).

Table 1
Selected bond lengths (Å).

Fe1—O1	1.9088 (17)	Fe2—O10	1.9181 (16)
Fe1—O3	1.9296 (16)	Fe2—O8	1.9343 (17)
Fe1—O2	2.0447 (17)	Fe2—O9	2.0551 (17)
Fe1—O4	2.0719 (18)	Fe2—O11	2.0763 (18)
Fe1—O7	2.1573 (18)	Fe2—O14	2.1379 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O10	0.85	2.22	2.887 (2)	136
O7—H7···O12	0.85	2.25	3.027 (3)	153
O14—H14A···O3	0.85	2.13	2.862 (2)	145
O14—H14A···O5	0.85	2.28	3.008 (3)	143
O7—H7B···O8	0.84	2.19	2.896 (2)	142
O7—H7B···O13	0.84	2.33	3.063 (2)	146
O14—H14B···O1	0.84	2.23	2.908 (2)	139
O14—H14B···O6	0.84	2.28	3.026 (2)	149
C7—H7A···Cl2^{i}	0.93	2.80	3.724 (3)	171
C34—H34···O2^{ii}	0.93	2.57	3.014 (3)	110
C37—H37C···O6^{iii}	0.96	2.58	3.506 (5)	162

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

4. Synthesis and crystallization

A mixture of FeCl$_3$·6H$_2$O (0.135 g, 0.5 mmol), 3-ethoxy-2-hydroxy-benzaldehyde (0.168 g, 1 mmol), methanol (5 mL) and acetonitrile (5 mL), with a pH adjusted to 7.5 by addition of triethylamine, was poured into a Teflon-lined autoclave (15 mL) and then heated at 413K for 3 days. Black crystals of the title compound were collected by filtration, washed with methanol and dried in air. Phase pure crystals were obtained by manual separation (yield: 124 mg, *ca* 54% based on Fe).

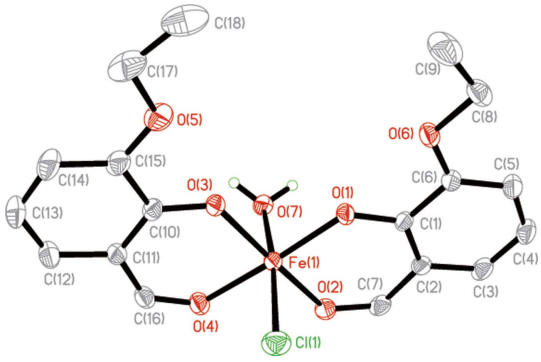


Figure 1
The molecular structure of one complex molecule of the title compound showing displacement ellipsoids drawn at the 30% probability level for non-H atoms. H atoms bonded to C atoms and the solvent molecule are not shown.

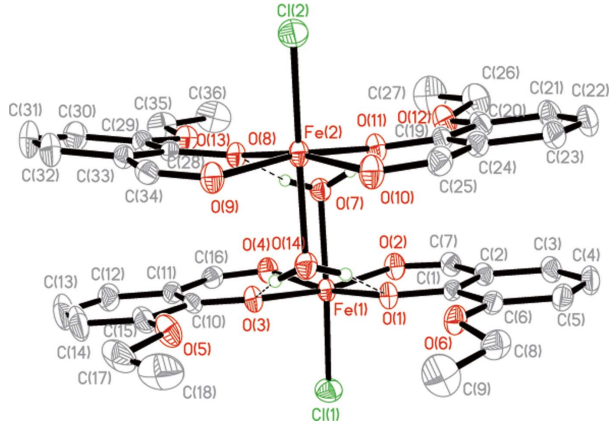


Figure 2
The dimer structure showing displacement ellipsoids drawn at the 30% probability level for non-H atoms. Hydrogen bonds are shown as dashed lines. H atoms bonded to C atoms and the solvent molecule are not shown.

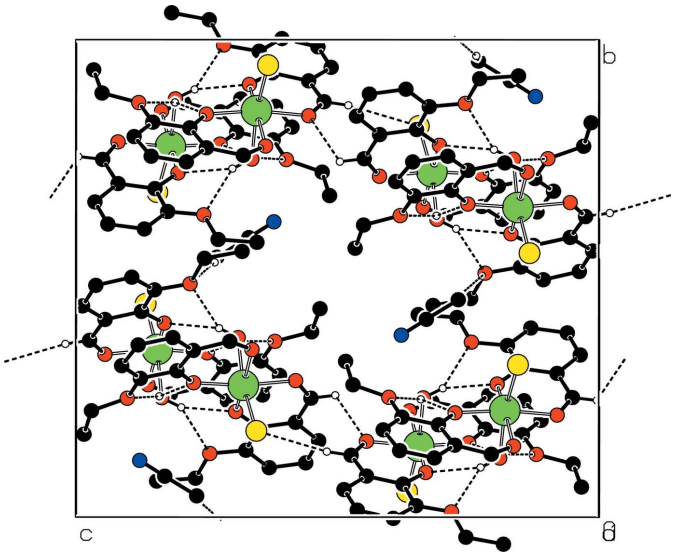


Figure 3
Part of the crystal structure with hydrogen bonds drawn as dashed lines.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms bonded to C atoms were positioned geometrically and refined as riding atoms, with C—H distances of 0.93 (aromatic), 0.96 (CH_2) or 0.97 Å (CH_3) with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$. H atoms bonded to O atoms were included with O—H = 0.84–0.85 Å and with $U_{iso}(H) = 1.5U_{eq}(O)$.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Fe(C_9H_9O_3)_2Cl(H_2O)]\cdot 0.5C_2H_3N$
M_r	460.17
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	11.8565 (4), 18.0786 (5), 20.5785 (6)
β (°)	105.981 (3)
V (Å^3)	4240.5 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.88
Crystal size (mm)	0.24 × 0.22 × 0.19
Data collection	
Diffractometer	SuperNova, Single source at offset, Eos
Absorption correction	Multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min}, T_{max}	0.811, 0.848
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17797, 7544, 6145
R_{int}	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.597
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.097, 1.00
No. of reflections	7544
No. of parameters	517
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.24

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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supporting information

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

Aquachloridobis(2-ethoxy-6-formylphenolato-κ^2O^1,O^6)iron(III) acetonitrile hemisolvate

Crystal data

$[Fe(C_9H_9O_3)_2Cl(H_2O)]\cdot 0.5C_2H_3N$
M_r = 460.17
Monoclinic, $P2_1/c$
a = 11.8565 (4) Å
b = 18.0786 (5) Å
c = 20.5785 (6) Å
β = 105.981 (3)°
V = 4240.5 (2) Å^3
Z = 8
$F(000)$ = 1904
D_x = 1.442 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8118 reflections
θ = 3.6–28.5°
μ = 0.88 mm^{-1}
T = 293 K
Block, black
0.24 × 0.22 × 0.19 mm

Data collection

SuperNova, Single source at offset, Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min} = 0.811, T_{max} = 0.848
17797 measured reflections
7544 independent reflections
6145 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.1°, θ_{min} = 2.9°
h = −11→14
k = −21→15
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.00
7544 reflections
517 parameters
0 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

$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 2.8341P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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}
C1	0.4723 (2)	0.43070 (13)	0.87478 (12)	0.0313 (5)
C2	0.5036 (2)	0.41391 (14)	0.94458 (12)	0.0361 (6)
C3	0.6098 (2)	0.44226 (16)	0.98744 (14)	0.0467 (7)
H3	0.6308	0.4311	1.0333	0.056*
C4	0.6807 (2)	0.48534 (17)	0.96195 (15)	0.0511 (8)
H4	0.7501	0.5036	0.9905	0.061*
C5	0.6504 (2)	0.50250 (15)	0.89322 (14)	0.0457 (7)
H5	0.6999	0.5323	0.8764	0.055*
C6	0.5483 (2)	0.47596 (14)	0.84989 (13)	0.0357 (6)
C7	0.4316 (2)	0.37029 (15)	0.97413 (12)	0.0411 (6)
H7A	0.4588	0.3627	1.0205	0.049*
C8	0.5749 (3)	0.54206 (16)	0.75438 (15)	0.0500 (7)
H8A	0.6522	0.5229	0.7564	0.060*
H8B	0.5843	0.5876	0.7802	0.060*
C9	0.5078 (3)	0.5565 (2)	0.68265 (17)	0.0771 (11)
H9A	0.4918	0.5104	0.6587	0.116*
H9B	0.5534	0.5876	0.6617	0.116*
H9C	0.4352	0.5807	0.6815	0.116*
C10	0.0680 (2)	0.31538 (14)	0.71813 (12)	0.0356 (6)
C11	−0.0010 (2)	0.27358 (15)	0.74985 (14)	0.0392 (6)
C12	−0.1098 (3)	0.24408 (18)	0.71145 (17)	0.0557 (8)
H12	−0.1553	0.2164	0.7328	0.067*
C13	−0.1476 (3)	0.2559 (2)	0.64441 (18)	0.0737 (11)
H13	−0.2191	0.2363	0.6198	0.088*
C14	−0.0808 (3)	0.2970 (2)	0.61172 (16)	0.0684 (10)
H14	−0.1079	0.3044	0.5653	0.082*
C15	0.0254 (2)	0.32719 (17)	0.64725 (14)	0.0473 (7)
C16	0.0336 (2)	0.25793 (14)	0.82021 (14)	0.0413 (6)
H16	−0.0186	0.2297	0.8363	0.050*
C17	0.0579 (3)	0.3882 (2)	0.55026 (15)	0.0729 (11)
H17A	−0.0163	0.4141	0.5415	0.088*
H17B	0.0466	0.3441	0.5223	0.088*

C18	0.1481 (4)	0.4368 (2)	0.53403 (17)	0.0868 (13)
H18A	0.1240	0.4494	0.4869	0.130*
H18B	0.2218	0.4112	0.5441	0.130*
H18C	0.1566	0.4811	0.5606	0.130*
C19	0.6290 (2)	0.30649 (14)	0.80685 (12)	0.0351 (6)
C20	0.6729 (2)	0.29049 (15)	0.87667 (13)	0.0412 (6)
C21	0.7816 (3)	0.31710 (18)	0.91231 (15)	0.0554 (8)
H21	0.8107	0.3059	0.9580	0.066*
C22	0.8483 (3)	0.36059 (18)	0.88073 (16)	0.0567 (8)
H22	0.9203	0.3790	0.9058	0.068*
C23	0.8093 (2)	0.37603 (16)	0.81448 (16)	0.0495 (7)
H23	0.8551	0.4045	0.7939	0.059*
C24	0.6985 (2)	0.34914 (15)	0.77547 (13)	0.0399 (6)
C25	0.6616 (2)	0.36812 (15)	0.70571 (14)	0.0436 (7)
H25	0.7135	0.3969	0.6899	0.052*
C26	0.6419 (3)	0.2228 (2)	0.97025 (15)	0.0687 (10)
H26A	0.6566	0.2642	1.0014	0.082*
H26B	0.7149	0.1960	0.9758	0.082*
C27	0.5526 (4)	0.1736 (2)	0.98454 (17)	0.0903 (13)
H27A	0.5799	0.1554	1.0300	0.135*
H27B	0.5386	0.1328	0.9535	0.135*
H27C	0.4810	0.2007	0.9794	0.135*
C28	0.2287 (2)	0.18807 (13)	0.65121 (12)	0.0308 (5)
C29	0.1559 (2)	0.14007 (14)	0.67567 (13)	0.0363 (6)
C30	0.0640 (3)	0.10353 (16)	0.63112 (16)	0.0535 (8)
H30	0.0163	0.0720	0.6476	0.064*
C31	0.0426 (3)	0.11373 (19)	0.56139 (16)	0.0644 (9)
H31	−0.0184	0.0881	0.5319	0.077*
C32	0.1090 (3)	0.16011 (18)	0.53657 (15)	0.0540 (8)
H32	0.0930	0.1669	0.4901	0.065*
C33	0.2032 (2)	0.19863 (14)	0.58073 (12)	0.0352 (6)
C34	0.2671 (2)	0.24922 (14)	0.55117 (12)	0.0365 (6)
H34	0.2399	0.2553	0.5046	0.044*
C35	0.1192 (3)	0.08154 (17)	0.77209 (17)	0.0576 (8)
H35A	0.1187	0.0336	0.7509	0.069*
H35B	0.0388	0.0983	0.7637	0.069*
C36	0.1749 (3)	0.0756 (2)	0.84620 (18)	0.0783 (11)
H36A	0.1315	0.0412	0.8655	0.117*
H36B	0.1747	0.1232	0.8668	0.117*
H36C	0.2542	0.0586	0.8541	0.117*
C37	0.7841 (4)	0.4555 (2)	0.2688 (2)	0.0958 (14)
H37A	0.7900	0.4268	0.2306	0.144*
H37B	0.8267	0.5009	0.2703	0.144*
H37C	0.7032	0.4663	0.2647	0.144*
C38	0.8329 (4)	0.4144 (2)	0.3298 (2)	0.0813 (12)
Cl1	0.13472 (7)	0.44690 (4)	0.86746 (4)	0.0548 (2)
Cl2	0.55591 (7)	0.18069 (4)	0.65509 (4)	0.05256 (19)
Fe1	0.24653 (3)	0.35131 (2)	0.845034 (16)	0.03200 (11)

Fe2	0.44691 (3)	0.27615 (2)	0.680267 (17)	0.03308 (11)
H7	0.4102	0.2531	0.8409	0.050*
H14A	0.2855	0.3687	0.6893	0.050*
H7B	0.3042	0.2300	0.7991	0.050*
H14B	0.3926	0.3959	0.7267	0.050*
N1	0.8708 (4)	0.3813 (2)	0.3776 (2)	0.1248 (16)
O1	0.37557 (14)	0.40635 (9)	0.83148 (8)	0.0357 (4)
O2	0.33580 (16)	0.34094 (10)	0.94490 (8)	0.0422 (4)
O3	0.17033 (14)	0.34421 (10)	0.74945 (8)	0.0368 (4)
O4	0.12491 (16)	0.27731 (10)	0.86278 (9)	0.0421 (4)
O5	0.09822 (18)	0.36832 (12)	0.62027 (9)	0.0562 (6)
O6	0.51042 (16)	0.48883 (10)	0.78201 (9)	0.0417 (4)
O7	0.33730 (15)	0.24998 (10)	0.83627 (8)	0.0392 (4)
O8	0.31635 (14)	0.22101 (9)	0.69505 (8)	0.0335 (4)
O9	0.35436 (16)	0.28597 (10)	0.58031 (8)	0.0403 (4)
O10	0.52406 (15)	0.28135 (10)	0.77521 (8)	0.0376 (4)
O11	0.56879 (16)	0.35092 (11)	0.66343 (9)	0.0446 (5)
O12	0.59928 (17)	0.24897 (12)	0.90188 (9)	0.0515 (5)
O13	0.18531 (16)	0.13332 (10)	0.74457 (9)	0.0420 (4)
O14	0.35666 (15)	0.37611 (10)	0.69009 (8)	0.0391 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0282 (13)	0.0326 (13)	0.0322 (13)	−0.0006 (10)	0.0065 (10)	−0.0087 (11)
C2	0.0313 (14)	0.0454 (15)	0.0306 (13)	0.0007 (11)	0.0070 (11)	−0.0066 (11)
C3	0.0394 (16)	0.0601 (18)	0.0342 (14)	0.0006 (14)	−0.0004 (12)	−0.0133 (13)
C4	0.0341 (15)	0.0617 (19)	0.0515 (17)	−0.0091 (14)	0.0017 (13)	−0.0206 (15)
C5	0.0368 (15)	0.0457 (16)	0.0538 (17)	−0.0092 (12)	0.0113 (13)	−0.0114 (14)
C6	0.0327 (14)	0.0365 (14)	0.0381 (14)	−0.0027 (11)	0.0100 (11)	−0.0065 (11)
C7	0.0434 (16)	0.0526 (16)	0.0235 (12)	0.0017 (13)	0.0029 (11)	−0.0024 (12)
C8	0.0502 (18)	0.0429 (16)	0.0629 (19)	−0.0088 (13)	0.0258 (15)	0.0053 (14)
C9	0.076 (3)	0.089 (3)	0.069 (2)	−0.013 (2)	0.025 (2)	0.031 (2)
C10	0.0267 (13)	0.0404 (14)	0.0352 (13)	0.0073 (11)	0.0011 (11)	−0.0065 (12)
C11	0.0287 (14)	0.0440 (15)	0.0447 (15)	−0.0002 (11)	0.0099 (12)	−0.0111 (12)
C12	0.0345 (16)	0.065 (2)	0.065 (2)	−0.0077 (14)	0.0099 (15)	−0.0148 (17)
C13	0.0355 (18)	0.108 (3)	0.067 (2)	−0.0123 (19)	−0.0041 (17)	−0.022 (2)
C14	0.0471 (19)	0.102 (3)	0.0422 (17)	0.0010 (19)	−0.0112 (15)	−0.0115 (18)
C15	0.0385 (16)	0.0628 (19)	0.0356 (14)	0.0097 (14)	0.0018 (12)	−0.0037 (14)
C16	0.0349 (15)	0.0388 (14)	0.0536 (17)	−0.0069 (12)	0.0179 (13)	−0.0040 (13)
C17	0.084 (3)	0.093 (3)	0.0333 (16)	0.032 (2)	0.0016 (16)	0.0142 (17)
C18	0.131 (4)	0.084 (3)	0.049 (2)	0.027 (3)	0.033 (2)	0.0260 (19)
C19	0.0261 (13)	0.0431 (14)	0.0335 (13)	0.0029 (11)	0.0040 (11)	−0.0105 (11)
C20	0.0327 (14)	0.0521 (16)	0.0354 (14)	0.0063 (12)	0.0038 (12)	−0.0033 (13)
C21	0.0413 (17)	0.070 (2)	0.0426 (16)	0.0059 (15)	−0.0086 (14)	−0.0056 (15)
C22	0.0320 (16)	0.069 (2)	0.060 (2)	−0.0048 (14)	−0.0034 (14)	−0.0154 (17)
C23	0.0312 (15)	0.0517 (17)	0.065 (2)	−0.0040 (13)	0.0128 (14)	−0.0137 (15)
C24	0.0312 (14)	0.0463 (15)	0.0420 (15)	−0.0013 (12)	0.0100 (12)	−0.0116 (12)

C25	0.0366 (15)	0.0462 (16)	0.0518 (16)	−0.0091 (12)	0.0187 (13)	−0.0066 (13)
C26	0.078 (3)	0.088 (3)	0.0302 (15)	0.004 (2)	−0.0023 (16)	0.0079 (16)
C27	0.109 (3)	0.119 (3)	0.0420 (19)	−0.007 (3)	0.019 (2)	0.016 (2)
C28	0.0261 (12)	0.0312 (12)	0.0340 (13)	0.0022 (10)	0.0065 (10)	−0.0069 (11)
C29	0.0313 (14)	0.0384 (14)	0.0386 (14)	−0.0006 (11)	0.0089 (11)	−0.0049 (12)
C30	0.0401 (16)	0.0514 (17)	0.067 (2)	−0.0149 (14)	0.0118 (15)	−0.0030 (16)
C31	0.0495 (19)	0.081 (2)	0.0526 (19)	−0.0257 (17)	−0.0029 (15)	−0.0149 (18)
C32	0.0459 (18)	0.070 (2)	0.0384 (15)	−0.0093 (15)	−0.0018 (13)	−0.0072 (15)
C33	0.0319 (14)	0.0409 (14)	0.0307 (13)	0.0018 (11)	0.0049 (11)	−0.0066 (11)
C34	0.0373 (15)	0.0473 (15)	0.0238 (12)	0.0091 (12)	0.0065 (11)	−0.0036 (11)
C35	0.060 (2)	0.0526 (18)	0.071 (2)	−0.0060 (15)	0.0350 (17)	0.0076 (16)
C36	0.092 (3)	0.083 (3)	0.072 (2)	0.001 (2)	0.043 (2)	0.026 (2)
C37	0.081 (3)	0.089 (3)	0.121 (4)	0.028 (2)	0.034 (3)	0.025 (3)
C38	0.080 (3)	0.071 (3)	0.097 (3)	0.023 (2)	0.031 (2)	−0.001 (2)
Cl1	0.0538 (4)	0.0557 (4)	0.0578 (4)	0.0102 (4)	0.0200 (4)	−0.0059 (4)
Cl2	0.0491 (4)	0.0603 (5)	0.0508 (4)	0.0039 (3)	0.0180 (3)	−0.0090 (4)
Fe1	0.0288 (2)	0.0411 (2)	0.02566 (18)	−0.00498 (15)	0.00670 (14)	−0.00089 (15)
Fe2	0.0304 (2)	0.0436 (2)	0.02459 (18)	−0.00648 (15)	0.00658 (15)	−0.00295 (15)
N1	0.163 (4)	0.103 (3)	0.103 (3)	0.048 (3)	0.028 (3)	0.008 (3)
O1	0.0312 (9)	0.0468 (10)	0.0276 (8)	−0.0103 (8)	0.0053 (7)	−0.0003 (8)
O2	0.0426 (11)	0.0564 (12)	0.0271 (9)	−0.0081 (9)	0.0088 (8)	0.0021 (8)
O3	0.0302 (9)	0.0504 (10)	0.0275 (8)	−0.0057 (8)	0.0042 (7)	−0.0011 (8)
O4	0.0372 (10)	0.0507 (11)	0.0387 (10)	−0.0093 (8)	0.0110 (8)	0.0018 (9)
O5	0.0547 (13)	0.0775 (15)	0.0301 (10)	0.0058 (11)	0.0012 (9)	0.0105 (10)
O6	0.0398 (10)	0.0444 (10)	0.0408 (10)	−0.0137 (8)	0.0111 (8)	0.0023 (8)
O7	0.0349 (10)	0.0467 (10)	0.0344 (9)	0.0000 (8)	0.0072 (8)	−0.0038 (8)
O8	0.0305 (9)	0.0430 (10)	0.0258 (8)	−0.0068 (8)	0.0058 (7)	−0.0036 (7)
O9	0.0410 (11)	0.0526 (11)	0.0267 (9)	−0.0066 (9)	0.0081 (8)	−0.0014 (8)
O10	0.0298 (9)	0.0538 (11)	0.0278 (9)	−0.0070 (8)	0.0054 (7)	−0.0017 (8)
O11	0.0400 (11)	0.0584 (12)	0.0362 (10)	−0.0130 (9)	0.0117 (9)	−0.0013 (9)
O12	0.0478 (12)	0.0713 (13)	0.0283 (9)	0.0010 (10)	−0.0012 (9)	0.0063 (9)
O13	0.0403 (11)	0.0453 (11)	0.0430 (10)	−0.0052 (8)	0.0159 (9)	0.0021 (9)
O14	0.0377 (10)	0.0456 (10)	0.0324 (9)	−0.0036 (8)	0.0072 (8)	−0.0051 (8)

Geometric parameters (Å, º)

C1—O1	1.319 (3)	C24—C25	1.423 (4)
C1—C6	1.414 (3)	C25—O11	1.240 (3)
C1—C2	1.414 (3)	C25—H25	0.9300
C2—C7	1.416 (4)	C26—O12	1.438 (3)
C2—C3	1.420 (3)	C26—C27	1.473 (5)
C3—C4	1.353 (4)	C26—H26A	0.9700
C3—H3	0.9300	C26—H26B	0.9700
C4—C5	1.395 (4)	C27—H27A	0.9600
C4—H4	0.9300	C27—H27B	0.9600
C5—C6	1.377 (3)	C27—H27C	0.9600
C5—H5	0.9300	C28—O8	1.316 (3)
C6—O6	1.364 (3)	C28—C33	1.411 (3)

C7—O2	1.248 (3)	C28—C29	1.411 (3)
C7—H7A	0.9300	C29—O13	1.369 (3)
C8—O6	1.440 (3)	C29—C30	1.383 (4)
C8—C9	1.494 (4)	C30—C31	1.399 (4)
C8—H8A	0.9700	C30—H30	0.9300
C8—H8B	0.9700	C31—C32	1.344 (4)
C9—H9A	0.9600	C31—H31	0.9300
C9—H9B	0.9600	C32—C33	1.414 (4)
C9—H9C	0.9600	C32—H32	0.9300
C10—O3	1.315 (3)	C33—C34	1.426 (4)
C10—C11	1.401 (4)	C34—O9	1.237 (3)
C10—C15	1.422 (4)	C34—H34	0.9300
C11—C12	1.418 (4)	C35—O13	1.433 (3)
C11—C16	1.420 (4)	C35—C36	1.489 (4)
C12—C13	1.345 (5)	C35—H35A	0.9700
C12—H12	0.9300	C35—H35B	0.9700
C13—C14	1.387 (5)	C36—H36A	0.9600
C13—H13	0.9300	C36—H36B	0.9600
C14—C15	1.383 (4)	C36—H36C	0.9600
C14—H14	0.9300	C37—C38	1.436 (6)
C15—O5	1.368 (4)	C37—H37A	0.9600
C16—O4	1.241 (3)	C37—H37B	0.9600
C16—H16	0.9300	C37—H37C	0.9600
C17—O5	1.433 (3)	C38—N1	1.133 (5)
C17—C18	1.491 (5)	Cl1—Fe1	2.3007 (8)
C17—H17A	0.9700	Cl2—Fe2	2.2990 (8)
C17—H17B	0.9700	Fe1—O1	1.9088 (17)
C18—H18A	0.9600	Fe1—O3	1.9296 (16)
C18—H18B	0.9600	Fe1—O2	2.0447 (17)
C18—H18C	0.9600	Fe1—O4	2.0719 (18)
C19—O10	1.317 (3)	Fe1—O7	2.1573 (18)
C19—C24	1.408 (4)	Fe2—O10	1.9181 (16)
C19—C20	1.417 (3)	Fe2—O8	1.9343 (17)
C20—O12	1.358 (3)	Fe2—O9	2.0551 (17)
C20—C21	1.382 (4)	Fe2—O11	2.0763 (18)
C21—C22	1.396 (5)	Fe2—O14	2.1379 (18)
C21—H21	0.9300	O7—H7	0.8453
C22—C23	1.343 (4)	O7—H7B	0.8394
C22—H22	0.9300	O14—H14A	0.8504
C23—C24	1.423 (4)	O14—H14B	0.8371
C23—H23	0.9300		

O1—C1—C6	118.1 (2)	C26—C27—H27B	109.5
O1—C1—C2	123.4 (2)	H27A—C27—H27B	109.5
C6—C1—C2	118.5 (2)	C26—C27—H27C	109.5
C1—C2—C7	122.3 (2)	H27A—C27—H27C	109.5
C1—C2—C3	119.6 (2)	H27B—C27—H27C	109.5
C7—C2—C3	118.1 (2)	O8—C28—C33	123.3 (2)

C4—C3—C2	120.4 (3)	O8—C28—C29	118.6 (2)
C4—C3—H3	119.8	C33—C28—C29	118.1 (2)
C2—C3—H3	119.8	O13—C29—C30	125.0 (2)
C3—C4—C5	120.5 (3)	O13—C29—C28	114.7 (2)
C3—C4—H4	119.8	C30—C29—C28	120.3 (2)
C5—C4—H4	119.8	C29—C30—C31	120.3 (3)
C6—C5—C4	120.9 (3)	C29—C30—H30	119.8
C6—C5—H5	119.5	C31—C30—H30	119.8
C4—C5—H5	119.5	C32—C31—C30	120.8 (3)
O6—C6—C5	125.9 (2)	C32—C31—H31	119.6
O6—C6—C1	114.0 (2)	C30—C31—H31	119.6
C5—C6—C1	120.1 (2)	C31—C32—C33	120.3 (3)
O2—C7—C2	127.3 (2)	C31—C32—H32	119.8
O2—C7—H7A	116.4	C33—C32—H32	119.8
C2—C7—H7A	116.4	C28—C33—C32	120.1 (3)
O6—C8—C9	108.1 (2)	C28—C33—C34	122.5 (2)
O6—C8—H8A	110.1	C32—C33—C34	117.4 (2)
C9—C8—H8A	110.1	O9—C34—C33	127.5 (2)
O6—C8—H8B	110.1	O9—C34—H34	116.2
C9—C8—H8B	110.1	C33—C34—H34	116.2
H8A—C8—H8B	108.4	O13—C35—C36	108.4 (3)
C8—C9—H9A	109.5	O13—C35—H35A	110.0
C8—C9—H9B	109.5	C36—C35—H35A	110.0
H9A—C9—H9B	109.5	O13—C35—H35B	110.0
C8—C9—H9C	109.5	C36—C35—H35B	110.0
H9A—C9—H9C	109.5	H35A—C35—H35B	108.4
H9B—C9—H9C	109.5	C35—C36—H36A	109.5
O3—C10—C11	124.4 (2)	C35—C36—H36B	109.5
O3—C10—C15	117.5 (2)	H36A—C36—H36B	109.5
C11—C10—C15	118.1 (2)	C35—C36—H36C	109.5
C10—C11—C12	120.1 (3)	H36A—C36—H36C	109.5
C10—C11—C16	122.7 (2)	H36B—C36—H36C	109.5
C12—C11—C16	117.1 (3)	C38—C37—H37A	109.5
C13—C12—C11	120.5 (3)	C38—C37—H37B	109.5
C13—C12—H12	119.8	H37A—C37—H37B	109.5
C11—C12—H12	119.8	C38—C37—H37C	109.5
C12—C13—C14	120.6 (3)	H37A—C37—H37C	109.5
C12—C13—H13	119.7	H37B—C37—H37C	109.5
C14—C13—H13	119.7	N1—C38—C37	179.3 (6)
C15—C14—C13	120.9 (3)	O1—Fe1—O3	93.15 (7)
C15—C14—H14	119.6	O1—Fe1—O2	88.89 (7)
C13—C14—H14	119.6	O3—Fe1—O2	170.39 (8)
O5—C15—C14	125.8 (3)	O1—Fe1—O4	170.79 (8)
O5—C15—C10	114.4 (2)	O3—Fe1—O4	89.05 (7)
C14—C15—C10	119.8 (3)	O2—Fe1—O4	87.50 (7)
O4—C16—C11	127.9 (3)	O1—Fe1—O7	89.54 (7)
O4—C16—H16	116.1	O3—Fe1—O7	87.86 (7)
C11—C16—H16	116.1	O2—Fe1—O7	82.76 (7)

O5—C17—C18	108.2 (3)	O4—Fe1—O7	81.59 (7)
O5—C17—H17A	110.1	O1—Fe1—Cl1	99.62 (6)
C18—C17—H17A	110.1	O3—Fe1—Cl1	96.92 (6)
O5—C17—H17B	110.1	O2—Fe1—Cl1	91.99 (6)
C18—C17—H17B	110.1	O4—Fe1—Cl1	88.98 (6)
H17A—C17—H17B	108.4	O7—Fe1—Cl1	169.38 (5)
C17—C18—H18A	109.5	O10—Fe2—O8	92.46 (7)
C17—C18—H18B	109.5	O10—Fe2—O9	171.40 (8)
H18A—C18—H18B	109.5	O8—Fe2—O9	88.40 (7)
C17—C18—H18C	109.5	O10—Fe2—O11	88.82 (7)
H18A—C18—H18C	109.5	O8—Fe2—O11	170.26 (8)
H18B—C18—H18C	109.5	O9—Fe2—O11	88.92 (7)
O10—C19—C24	123.7 (2)	O10—Fe2—O14	88.45 (7)
O10—C19—C20	117.6 (2)	O8—Fe2—O14	88.73 (7)
C24—C19—C20	118.6 (2)	O9—Fe2—O14	83.01 (7)
O12—C20—C21	126.4 (3)	O11—Fe2—O14	81.66 (7)
O12—C20—C19	113.9 (2)	O10—Fe2—Cl2	97.26 (6)
C21—C20—C19	119.7 (3)	O8—Fe2—Cl2	99.93 (6)
C20—C21—C22	120.9 (3)	O9—Fe2—Cl2	91.01 (6)
C20—C21—H21	119.5	O11—Fe2—Cl2	89.47 (6)
C22—C21—H21	119.5	O14—Fe2—Cl2	169.37 (5)
C23—C22—C21	120.6 (3)	C1—O1—Fe1	131.13 (15)
C23—C22—H22	119.7	C7—O2—Fe1	126.59 (17)
C21—C22—H22	119.7	C10—O3—Fe1	129.49 (17)
C22—C23—C24	120.5 (3)	C16—O4—Fe1	124.91 (18)
C22—C23—H23	119.7	C15—O5—C17	118.1 (2)
C24—C23—H23	119.7	C6—O6—C8	117.2 (2)
C19—C24—C25	122.6 (2)	Fe1—O7—H7	116.9
C19—C24—C23	119.6 (3)	Fe1—O7—H7B	108.8
C25—C24—C23	117.8 (3)	H7—O7—H7B	109.8
O11—C25—C24	127.9 (3)	C28—O8—Fe2	129.83 (15)
O11—C25—H25	116.1	C34—O9—Fe2	125.71 (17)
C24—C25—H25	116.1	C19—O10—Fe2	129.90 (16)
O12—C26—C27	108.4 (3)	C25—O11—Fe2	124.78 (18)
O12—C26—H26A	110.0	C20—O12—C26	117.8 (2)
C27—C26—H26A	110.0	C29—O13—C35	117.1 (2)
O12—C26—H26B	110.0	Fe2—O14—H14A	112.6
C27—C26—H26B	110.0	Fe2—O14—H14B	107.6
H26A—C26—H26B	108.4	H14A—O14—H14B	109.9
C26—C27—H27A	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7···O10	0.85	2.22	2.887 (2)	136
O7—H7···O12	0.85	2.25	3.027 (3)	153
O14—H14*A*···O3	0.85	2.13	2.862 (2)	145
O14—H14*A*···O5	0.85	2.28	3.008 (3)	143

O7—H7*B*···O8	0.84	2.19	2.896 (2)	142
O7—H7*B*···O13	0.84	2.33	3.063 (2)	146
O14—H14*B*···O1	0.84	2.23	2.908 (2)	139
O14—H14*B*···O6	0.84	2.28	3.026 (2)	149
C7—H7*A*···Cl2^{i}	0.93	2.80	3.724 (3)	171
C34—H34···O2^{ii}	0.93	2.57	3.014 (3)	110
C37—H37*C*···O6^{iii}	0.96	2.58	3.506 (5)	162

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