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Crystal structure of tetrakis(acetylacetonato)dichloridodi-μ_3-methanolato-tetra-μ_2-methanolatotetrairon(III)

Casseday P. Richers, Jeffery A. Bertke, Danielle L. Gray* and Thomas B. Rauchfuss

School of Chemical Sciences, University of Illinois at Urbana-Champaign, Urbana, Illinois 61801, USA. *Correspondence e-mail: dgray@illinois.edu

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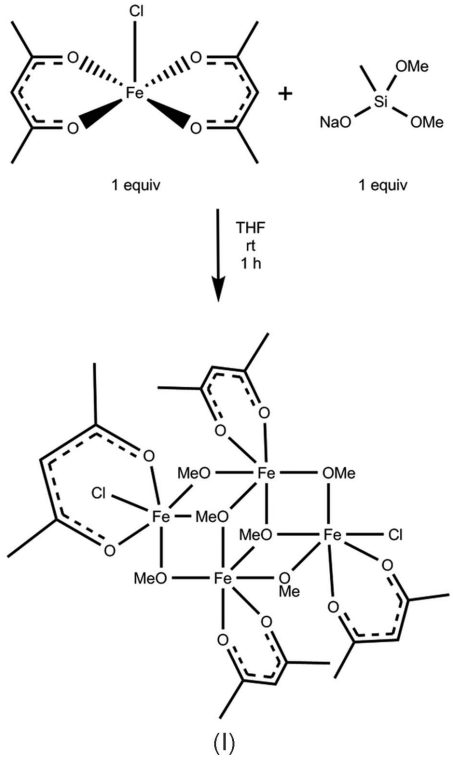
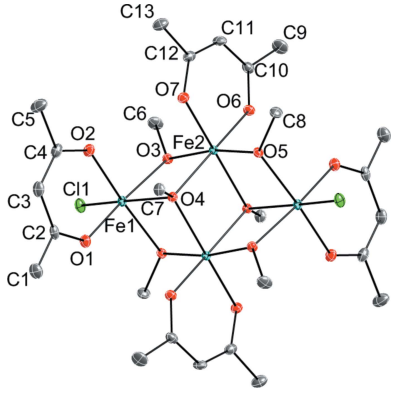
The title complex, $[Fe_4(C_5H_7O_2)_4(CH_3O)_6Cl_2]$ or $[Fe_4(acac)_4(\mu_2\text{-OMe})_4(\mu_3\text{-OMe})_2Cl_2]$ (acac = acetylacetonate), crystallizes in the orthorhombic *Pbca* space group with one half of the molecule per asymmetric unit, the other half being completed by inversion symmetry. The core structure consists of a face-sharing double pseudo-cubane entity with two opposite corners missing. Weak C—H···Cl intermolecular interactions result in a two-dimensional layered structure parallel to the *ac* plane.

1. Chemical context

Metal silanolate complexes bearing methoxy and ethoxy groups on silicon are relatively rare (Dupuy *et al.*, 2012) in comparison to *tert*-butoxysilanolate complexes (McMullen *et al.*, 1989, 1990; Nozaki *et al.*, 2002; Terry *et al.*, 1993, 1996; Truscott *et al.*, 2013). Nevertheless, such compounds may play a pivotal role in sol-gel reactions and in metal-catalysed curing reactions, such as room-temperature vulcanization (Cervantes *et al.*, 2012; Levitsky *et al.*, 2007; van Der Weij, 1980).



(I)



We have investigated the syntheses of metal methoxysilanolates *via* the additions of $NaOSi(OMe)_2Me$ to metal

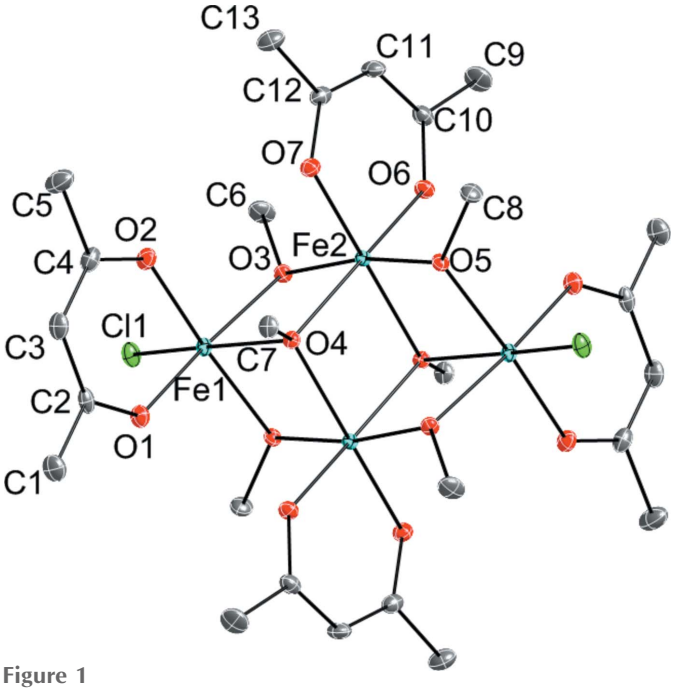


Figure 1
View of the molecular structure of (I), showing the atomic numbering and 35% probability displacement ellipsoids for the non-H atoms. The unlabeled atoms are related to the labeled ones by the symmetry operator $(-x+1, -y+1, -z+1)$. H atoms have been removed for clarity.

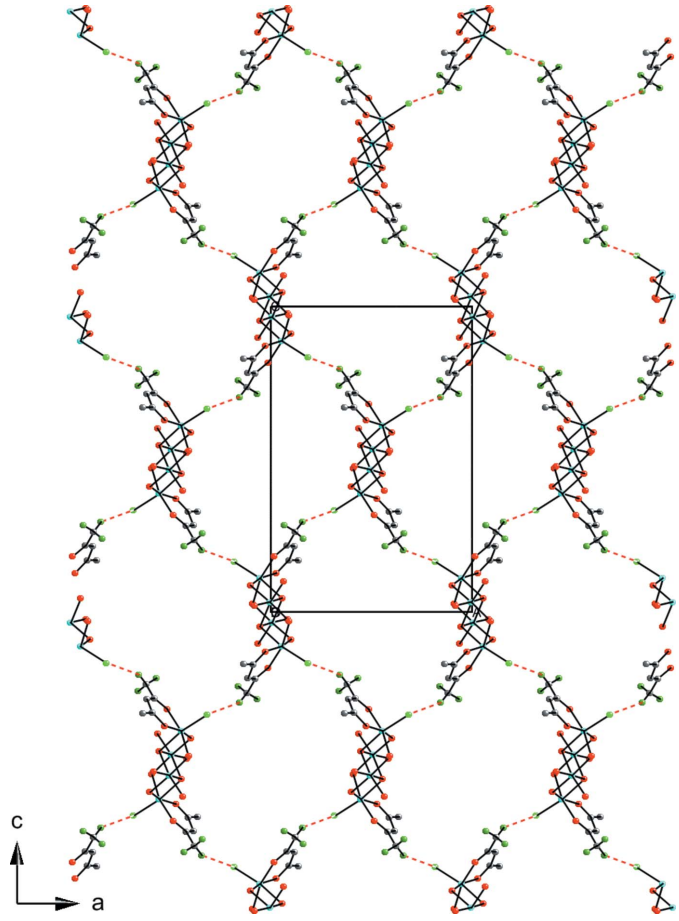


Figure 2
A view along the *b* axis of the extended two-dimensional network of (I) with an overlay of the unit cell. The intermolecular Cl—H interations are shown as dashed red lines. All C atoms except those in the hydrogen-bonded acac ligand and all H atoms except those of the hydrogen-bonded methyl group have been removed for clarity. Color key: blue = Fe, light-green = Cl, red = O, gray = C, and green = H.

halides and discovered that, in certain cases, the addition of $NaOSi(OMe)_2Me$ to a metal halide results in the formation of a methanolate complex instead of silanolate complex. In line with this observation, we now report that the addition of $NaOSi(OMe)_2Me$ to $Fe(acac)_2Cl$ results in the formation of a tetranuclear iron(III) methanolate compound, $Fe_4(acac)_4(\mu_2\text{-}OMe)_4(\mu_3\text{-}OMe)_2Cl_2$, (I).

2. Structural commentary

The structure of (I) contains two crystallographically independent Fe^{III} metal atoms. Both cations are in approximately octahedral coordination environments. The coordination sphere of Fe1 is filled by the O atoms of one κ^2-acac ligand [Fe1—O1 = 1.9971 (13) Å and Fe1—O2 = 1.9934 (13) Å], two μ_2-methanolate groups [Fe1—O3 = 1.9861 (12) Å and Fe1—$O5^i$ = 1.9885 (12) Å; symmetry code: (i) $-x+1, -y+1, -z+1$], one μ_3-methanolate group [Fe1—O4 = 2.2135 (12) Å], and one terminal chloride ligand [Fe1—Cl1 = 2.2776 (5) Å]. The coordination sphere of Fe2 is filled by the O atoms of one κ^2-acac ligand [Fe2—O6 = 1.9717 (13) Å and Fe2—O7 = 1.9692 (12) Å], two μ_2-methanolate groups [Fe2—O3 = 1.9755 (12) Å and Fe2—O5 = 1.9823 (12) Å], and two μ_3-methanolate groups [Fe2—O4 = 2.0815 (12) Å and Fe2—$O4^i$ = 2.0809 (12) Å]. The angles around both Fe1 and Fe2 distort significantly from the ideal values of 90 and 180° of a perfect octahedron. For Fe1, the *cis* angles range from 75.69 (5) to 98.40 (4)°, while the *trans* angles range from 164.47 (5) to 170.40 (3)°. The angles around Fe2 have narrower ranges, with *cis* being 78.95 (5)–96.48 (5)° and *trans* being 170.08 (5)–170.16 (5)°.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···$Cl1^i$	0.95	2.91	3.797 (2)	155
C5—H5B···$Cl1^i$	0.98	2.91	3.800 (2)	152

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

The molecular structure of (I) (Fig. 1) can be described as an $[Fe_4(OMe)_6]$ face-sharing double pseudo-cubane entity with two opposite corners missing. The outside of the cluster is decorated by one acac ligand per metal and the Fe atoms at either end of the cluster are coordinated by one chloride ion. Neighboring Fe···Fe distances range from 3.1997 (4) to 3.2175 (6) Å, while the Fe1···$Fe1^i$ distance is 5.5702 (6) Å.

3. Supramolecular features

There are no significant supramolecular features to discuss with the extended structure of (I). There are weak interactions

between the Cl^- ion and an acac ligand on neighboring molecules (Table 1). Taking into account these weak interactions, the extended structure becomes layers of two-dimensional 4^4-nets normal to the *b* axis (Fig. 2).

4. Database survey

One closely related complex, $[Fe_4(acac)_4(OMe)_6(N_3)_2]$, has previously been reported (Li *et al.*, 1997) in which N_3^- takes the position of Cl^- in (I). The molecular structure of the azide complex is very similar to that of (I), and can be described as the same $[Fe_4(OMe)_6]$ face-sharing double cubane cluster with two opposite corners missing. The average Fe—O_{acac} distance of 1.978 Å is quite close to the average Fe—O_{acac} distance of 1.982 Å in (I). The average Fe—OMe distances in the azide complex (μ_2-OMe: 1.977 Å; μ_3-OMe: 2.124 Å) are also comparable to those in (I) (μ_2-OMe: 1.983 Å; μ_3-OMe: 2.125 Å).

A search of the Cambridge Structural Database (Groom & Allen, 2014) returned 14 complexes with an $[Fe_4(OR)_6]$ cluster core similar to (I) (Abu-Nawwas *et al.*, 2009; Mulyana *et al.*, 2009). All of these materials, except the azide compound described above, use more complex, multidentate ligands to form the polynuclear entity. The $[Fe_4(OR)_6]$ motif is present is 63 additional materials as part of a higher-order cluster complex (Ferguson *et al.*, 2013; Murugesu *et al.*, 2004).

5. Synthesis and crystallization

A solution of $NaOSi(OMe)_2Me$ (57 mg, 3.96×10^{-4} mol, 1 equivalent) in THF (3 ml) was added to a solution of $Fe(acac)_2Cl$ (200 mg, 3.96×10^{-4} mol, 1 equivalent) in THF (see Scheme). The mixture was stirred rapidly at room temperature, and a slight color change from a dark-red to a lighter red was observed. Removal of the solvent under vacuum resulted in the precipitation of an orange solid, which upon washing with dry Et_2O (2×10 ml) left a yellow solid. The yellow solid was extracted into dry CH_2Cl_2 and filtered through Celite. The CH_2Cl_2 was then removed under vacuum, leaving a yellow solid (54 mg, 6.16×10^{-5} mol, 62% yield). Crystals suitable for X-ray diffraction were grown by slow diffusion of pentane into a CH_2Cl_2 solution of the yellow solid.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Methyl-H atom positions, RCH_3, were optimized by rotation about R—C bonds, with idealized C—H, R—H and H···H distances (C—H = 0.98 Å). The remaining H atoms were included as riding idealized contributors (C—H = 0.95 Å). H atoms were assigned $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $U_{iso}(H) = 1.2U_{eq}(C)$ otherwise. The 102 reflection was omitted from the final refinement because it was partially obscured by the shadow of the beam stop.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Fe_4(C_5H_7O_2)_4(CH_3O)_6Cl_2]$
M_r	876.93
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	102
a, *b*, *c* (Å)	14.0714 (6), 12.1888 (4), 21.3543 (7)
V (Å^3)	3662.6 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.76
Crystal size (mm)	0.38 × 0.37 × 0.23
Data collection	
Diffractometer	Bruker D8 Venture/Photon 100
Absorption correction	Integration (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.568, 0.718
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	46682, 4559, 3837
R_{int}	0.060
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.070, 1.04
No. of reflections	4559
No. of parameters	215
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.34

Computer programs: *APEX2*, *SAINT*, *XPREP* and *XCIF* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2013* (Sheldrick, 2015*b*), *SHELXTL* (Sheldrick, 2008), *CrystalMaker* (CrystalMaker, 2014) and *publCIF* (Westrip, 2010).

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Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013), *XPREP* (Bruker, 2013), *SADABS* (Bruker, 2012) and TWINABS (Bruker, 2012); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015b); molecular graphics: *SHELXTL* (Bruker, 2013) and *CrystalMaker* (*CrystalMaker*, 2014); software used to prepare material for publication: XCIF (Bruker, 2013) and *publCIF* (Westrip, 2010).

Tetrakis(acetylacetonato)dichloridodi-μ_3-methanolato-tetra-μ_2-methanolato-tetrairon(III)

Crystal data

$[Fe_4(C_5H_7O_2)_4(CH_3O)_6Cl_2]$
M_r = 876.93
Orthorhombic, *Pbca*
a = 14.0714 (6) Å
b = 12.1888 (4) Å
c = 21.3543 (7) Å
V = 3662.6 (2) Å^3
Z = 4
$F(000)$ = 1808
D_x = 1.590 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9882 reflections
θ = 2.4–28.3°
μ = 1.76 mm^{-1}
T = 102 K
Prism, orange
0.38 × 0.37 × 0.23 mm

Data collection

Bruker D8 Venture/Photon 100 diffractometer
Radiation source: microfocus sealed tube
Multilayer mirrors monochromator
profile data from φ and ω scans
Absorption correction: integration (*SADABS*; Bruker, 2012)
T_{min} = 0.568, T_{max} = 0.718
46682 measured reflections
4559 independent reflections
3837 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 28.3°, θ_{min} = 2.9°
h = −18→18
k = −15→16
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.070
S = 1.04
4559 reflections
215 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 1.8648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Experimental. One distinct cell was identified using *APEX2* (Bruker, 2013). Four frame series were integrated and filtered for statistical outliers using *SAINT* (Bruker, 2013) then corrected for absorption by integration using *SADABS* v2012/1 (Bruker, 2012). No decay correction was applied.

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. Structure was phased by intrinsic phasing methods (XT, Sheldrick, 2013). Systematic conditions suggested the unambiguous space group. The space group choice was confirmed by successful convergence of the full-matrix least-squares refinement on F^2. The final difference Fourier had no significant features. A final analysis of variance between observed and calculated structure factors showed little dependence on amplitude or resolution.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.44600 (2)	0.60052 (2)	0.38841 (2)	0.01161 (7)
Fe2	0.49483 (2)	0.38287 (2)	0.46636 (2)	0.01051 (7)
Cl1	0.31634 (3)	0.65927 (4)	0.33408 (2)	0.01952 (11)
O1	0.52694 (10)	0.73202 (11)	0.37204 (6)	0.0177 (3)
O2	0.51458 (9)	0.52865 (11)	0.31778 (6)	0.0170 (3)
O3	0.39890 (9)	0.45244 (10)	0.41172 (5)	0.0126 (2)
O4	0.55491 (8)	0.53740 (10)	0.45395 (5)	0.0106 (2)
O5	0.59118 (9)	0.33767 (10)	0.52897 (5)	0.0130 (3)
O6	0.41693 (9)	0.25001 (10)	0.47819 (6)	0.0158 (3)
O7	0.56371 (9)	0.31011 (10)	0.39767 (6)	0.0153 (3)
C1	0.63600 (17)	0.85947 (17)	0.33019 (11)	0.0306 (5)
H1A	0.5958	0.9116	0.3530	0.046*
H1B	0.6452	0.8854	0.2872	0.046*
H1C	0.6978	0.8535	0.3511	0.046*
C2	0.58856 (14)	0.74858 (16)	0.32912 (9)	0.0190 (4)
C3	0.61526 (14)	0.67189 (17)	0.28427 (9)	0.0204 (4)
H3	0.6604	0.6933	0.2535	0.024*
C4	0.57959 (13)	0.56530 (16)	0.28188 (8)	0.0169 (4)
C5	0.61965 (15)	0.48447 (18)	0.23600 (9)	0.0246 (4)
H5A	0.6607	0.4323	0.2581	0.037*
H5B	0.6568	0.5236	0.2042	0.037*
H5C	0.5676	0.4447	0.2157	0.037*
C6	0.34499 (14)	0.38938 (16)	0.36761 (9)	0.0201 (4)
H6A	0.3847	0.3723	0.3312	0.030*
H6B	0.2894	0.4317	0.3541	0.030*
H6C	0.3240	0.3210	0.3874	0.030*
C7	0.65434 (12)	0.54563 (16)	0.43948 (8)	0.0151 (4)
H7A	0.6918	0.5215	0.4756	0.023*
H7B	0.6701	0.6220	0.4296	0.023*
H7C	0.6690	0.4990	0.4034	0.023*
C8	0.63734 (16)	0.23375 (17)	0.52457 (10)	0.0242 (5)
H8A	0.5895	0.1754	0.5233	0.036*
H8B	0.6785	0.2232	0.5611	0.036*

H8C	0.6757	0.2313	0.4863	0.036*
C9	0.35287 (16)	0.07273 (17)	0.46844 (10)	0.0258 (5)
H9A	0.3624	0.0571	0.5130	0.039*
H9B	0.3634	0.0057	0.4440	0.039*
H9C	0.2878	0.0987	0.4617	0.039*
C10	0.42174 (14)	0.15947 (15)	0.44786 (9)	0.0171 (4)
C11	0.48468 (15)	0.13917 (15)	0.39877 (9)	0.0198 (4)
H11	0.4823	0.0689	0.3796	0.024*
C12	0.55074 (14)	0.21388 (16)	0.37582 (9)	0.0175 (4)
C13	0.61370 (17)	0.18240 (18)	0.32192 (10)	0.0286 (5)
H13A	0.6064	0.2360	0.2881	0.043*
H13B	0.5957	0.1095	0.3067	0.043*
H13C	0.6801	0.1812	0.3358	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01076 (13)	0.01374 (13)	0.01034 (12)	−0.00004 (9)	0.00102 (9)	0.00161 (9)
Fe2	0.01094 (13)	0.01008 (12)	0.01050 (13)	−0.00020 (9)	0.00027 (9)	−0.00061 (9)
Cl1	0.0182 (2)	0.0259 (2)	0.0145 (2)	0.00438 (18)	−0.00364 (17)	0.00256 (17)
O1	0.0174 (7)	0.0180 (7)	0.0179 (7)	−0.0018 (5)	0.0040 (5)	0.0027 (5)
O2	0.0156 (7)	0.0208 (7)	0.0146 (6)	0.0001 (5)	0.0040 (5)	0.0015 (5)
O3	0.0119 (6)	0.0147 (6)	0.0112 (6)	−0.0025 (5)	−0.0017 (5)	−0.0005 (5)
O4	0.0074 (6)	0.0125 (6)	0.0121 (6)	−0.0004 (5)	0.0015 (4)	0.0004 (5)
O5	0.0127 (6)	0.0126 (6)	0.0138 (6)	0.0031 (5)	−0.0001 (5)	−0.0005 (5)
O6	0.0164 (7)	0.0139 (6)	0.0171 (6)	−0.0030 (5)	0.0016 (5)	0.0001 (5)
O7	0.0166 (7)	0.0144 (6)	0.0149 (6)	−0.0002 (5)	0.0029 (5)	−0.0029 (5)
C1	0.0292 (12)	0.0239 (11)	0.0388 (13)	−0.0074 (9)	0.0114 (10)	0.0021 (9)
C2	0.0147 (9)	0.0205 (10)	0.0216 (10)	−0.0003 (8)	−0.0005 (8)	0.0080 (8)
C3	0.0154 (10)	0.0280 (10)	0.0177 (9)	−0.0016 (8)	0.0047 (7)	0.0064 (8)
C4	0.0134 (9)	0.0268 (10)	0.0105 (8)	0.0042 (8)	−0.0008 (7)	0.0044 (7)
C5	0.0230 (11)	0.0302 (11)	0.0205 (10)	0.0037 (9)	0.0077 (8)	−0.0007 (8)
C6	0.0198 (10)	0.0216 (10)	0.0191 (9)	−0.0059 (8)	−0.0080 (8)	−0.0011 (7)
C7	0.0076 (8)	0.0205 (9)	0.0172 (9)	−0.0002 (7)	0.0020 (7)	0.0018 (7)
C8	0.0272 (11)	0.0190 (10)	0.0263 (11)	0.0127 (8)	−0.0064 (9)	−0.0051 (8)
C9	0.0244 (11)	0.0176 (10)	0.0353 (12)	−0.0064 (8)	0.0005 (9)	0.0005 (8)
C10	0.0164 (9)	0.0131 (9)	0.0217 (9)	−0.0005 (7)	−0.0053 (7)	0.0014 (7)
C11	0.0236 (11)	0.0129 (9)	0.0229 (10)	−0.0008 (7)	−0.0019 (8)	−0.0056 (7)
C12	0.0193 (10)	0.0182 (9)	0.0151 (9)	0.0044 (8)	−0.0013 (7)	−0.0035 (7)
C13	0.0334 (13)	0.0252 (11)	0.0274 (11)	0.0016 (9)	0.0113 (9)	−0.0107 (9)

Geometric parameters (Å, º)

Fe1—O3	1.9861 (12)	C3—C4	1.394 (3)
Fe1—O5^{i}	1.9885 (12)	C3—H3	0.9500
Fe1—O2	1.9934 (13)	C4—C5	1.499 (3)
Fe1—O1	1.9971 (13)	C5—H5A	0.9800
Fe1—O4	2.2135 (12)	C5—H5B	0.9800

Fe1—Cl1	2.2776 (5)	C5—H5C	0.9800
Fe2—O7	1.9692 (12)	C6—H6A	0.9800
Fe2—O6	1.9717 (13)	C6—H6B	0.9800
Fe2—O3	1.9755 (12)	C6—H6C	0.9800
Fe2—O5	1.9823 (12)	C7—H7A	0.9800
Fe2—O4^{i}	2.0809 (12)	C7—H7B	0.9800
Fe2—O4	2.0815 (12)	C7—H7C	0.9800
O1—C2	1.278 (2)	C8—H8A	0.9800
O2—C4	1.274 (2)	C8—H8B	0.9800
O3—C6	1.433 (2)	C8—H8C	0.9800
O4—C7	1.436 (2)	C9—C10	1.500 (3)
O4—Fe2^{i}	2.0808 (12)	C9—H9A	0.9800
O5—C8	1.426 (2)	C9—H9B	0.9800
O5—Fe1^{i}	1.9885 (12)	C9—H9C	0.9800
O6—C10	1.281 (2)	C10—C11	1.394 (3)
O7—C12	1.275 (2)	C11—C12	1.391 (3)
C1—C2	1.508 (3)	C11—H11	0.9500
C1—H1A	0.9800	C12—C13	1.502 (3)
C1—H1B	0.9800	C13—H13A	0.9800
C1—H1C	0.9800	C13—H13B	0.9800
C2—C3	1.390 (3)	C13—H13C	0.9800

O3—Fe1—O5^{i}	91.96 (5)	O1—C2—C1	115.54 (18)
O3—Fe1—O2	87.24 (5)	C3—C2—C1	119.58 (18)
O5^{i}—Fe1—O2	164.84 (5)	C2—C3—C4	123.69 (17)
O3—Fe1—O1	164.47 (5)	C2—C3—H3	118.2
O5^{i}—Fe1—O1	90.08 (5)	C4—C3—H3	118.2
O2—Fe1—O1	86.80 (5)	O2—C4—C3	124.28 (18)
O3—Fe1—O4	75.92 (5)	O2—C4—C5	115.63 (18)
O5^{i}—Fe1—O4	75.69 (5)	C3—C4—C5	120.07 (17)
O2—Fe1—O4	89.45 (5)	C4—C5—H5A	109.5
O1—Fe1—O4	89.70 (5)	C4—C5—H5B	109.5
O3—Fe1—Cl1	98.40 (4)	H5A—C5—H5B	109.5
O5^{i}—Fe1—Cl1	97.01 (4)	C4—C5—H5C	109.5
O2—Fe1—Cl1	98.08 (4)	H5A—C5—H5C	109.5
O1—Fe1—Cl1	96.63 (4)	H5B—C5—H5C	109.5
O4—Fe1—Cl1	170.40 (3)	O3—C6—H6A	109.5
O7—Fe2—O6	89.95 (5)	O3—C6—H6B	109.5
O7—Fe2—O3	95.14 (5)	H6A—C6—H6B	109.5
O6—Fe2—O3	92.77 (5)	O3—C6—H6C	109.5
O7—Fe2—O5	92.33 (5)	H6A—C6—H6C	109.5
O6—Fe2—O5	93.76 (5)	H6B—C6—H6C	109.5
O3—Fe2—O5	170.08 (5)	O4—C7—H7A	109.5
O7—Fe2—O4^{i}	170.08 (5)	O4—C7—H7B	109.5
O6—Fe2—O4^{i}	95.27 (5)	H7A—C7—H7B	109.5
O3—Fe2—O4^{i}	93.02 (5)	O4—C7—H7C	109.5
O5—Fe2—O4^{i}	78.95 (5)	H7A—C7—H7C	109.5
O7—Fe2—O4	96.48 (5)	H7B—C7—H7C	109.5

O6—Fe2—O4	170.16 (5)	O5—C8—H8A	109.5
O3—Fe2—O4	79.28 (5)	O5—C8—H8B	109.5
O5—Fe2—O4	93.41 (5)	H8A—C8—H8B	109.5
O4^{i}—Fe2—O4	79.52 (5)	O5—C8—H8C	109.5
C2—O1—Fe1	129.75 (13)	H8A—C8—H8C	109.5
C4—O2—Fe1	130.44 (13)	H8B—C8—H8C	109.5
C6—O3—Fe2	121.31 (11)	C10—C9—H9A	109.5
C6—O3—Fe1	119.96 (11)	C10—C9—H9B	109.5
Fe2—O3—Fe1	108.06 (6)	H9A—C9—H9B	109.5
C7—O4—Fe2^{i}	118.09 (10)	C10—C9—H9C	109.5
C7—O4—Fe2	119.09 (10)	H9A—C9—H9C	109.5
Fe2^{i}—O4—Fe2	100.48 (5)	H9B—C9—H9C	109.5
C7—O4—Fe1	120.95 (10)	O6—C10—C11	124.49 (18)
Fe2^{i}—O4—Fe1	97.00 (5)	O6—C10—C9	115.14 (17)
Fe2—O4—Fe1	96.53 (5)	C11—C10—C9	120.37 (18)
C8—O5—Fe2	120.92 (11)	C12—C11—C10	124.99 (17)
C8—O5—Fe1^{i}	120.97 (11)	C12—C11—H11	117.5
Fe2—O5—Fe1^{i}	108.25 (6)	C10—C11—H11	117.5
C10—O6—Fe2	127.83 (12)	O7—C12—C11	124.67 (17)
C12—O7—Fe2	128.04 (12)	O7—C12—C13	115.52 (18)
C2—C1—H1A	109.5	C11—C12—C13	119.81 (17)
C2—C1—H1B	109.5	C12—C13—H13A	109.5
H1A—C1—H1B	109.5	C12—C13—H13B	109.5
C2—C1—H1C	109.5	H13A—C13—H13B	109.5
H1A—C1—H1C	109.5	C12—C13—H13C	109.5
H1B—C1—H1C	109.5	H13A—C13—H13C	109.5
O1—C2—C3	124.85 (18)	H13B—C13—H13C	109.5

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···Cl1^{ii}	0.95	2.91	3.797 (2)	155
C5—H5*B*···Cl1^{ii}	0.98	2.91	3.800 (2)	152

Symmetry code: (ii) $x+1/2, y, -z+1/2$.