



# Crystal structure of tetrakis(acetylacetonato)di-chloridodi- $\mu_3$ -methanolato-tetra- $\mu_2$ -methanolato-tetrairon(III)

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The title complex,  $[\text{Fe}_4(\text{C}_5\text{H}_7\text{O}_2)_4(\text{CH}_3\text{O})_6\text{Cl}_2]$  or  $[\text{Fe}_4(\text{acac})_4(\mu_2\text{-OMe})_4(\mu_3\text{-OMe})_2\text{Cl}_2]$  (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.

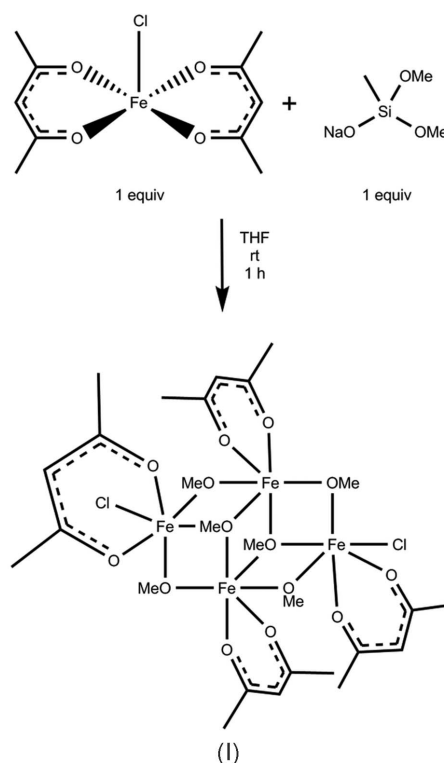
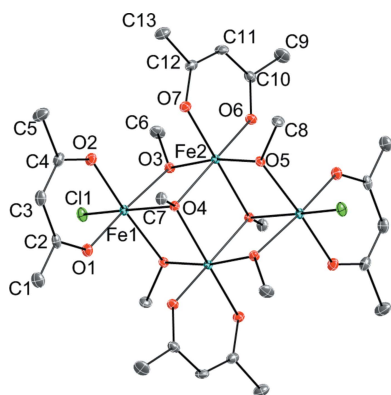
**Keywords:** crystal structure; cluster; iron(III); acetylacetonate; double cubane

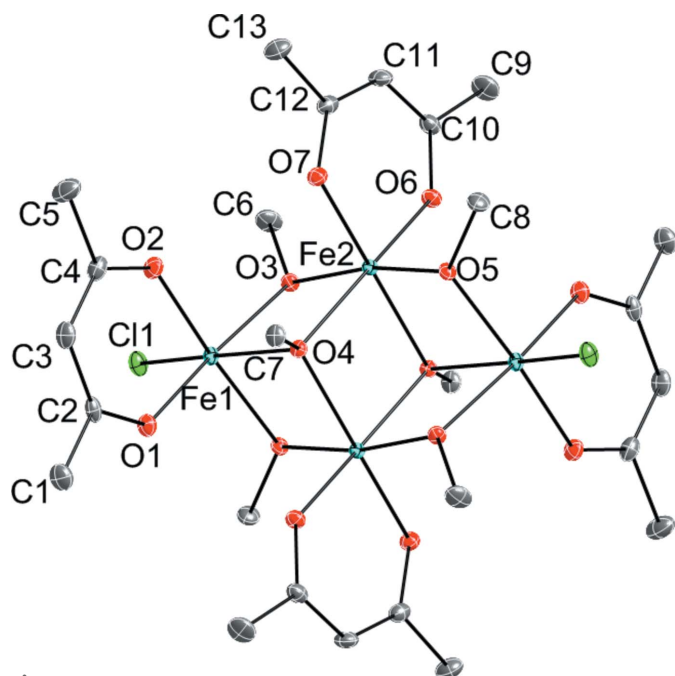
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## 1. Chemical context

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).





**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.

halides and discovered that, in certain cases, the addition of  $\text{NaOSi}(\text{OMe})_2\text{Me}$  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  $\text{NaOSi}(\text{OMe})_2\text{Me}$  to  $\text{Fe}(\text{acac})_2\text{Cl}$  results in the formation of a tetranuclear iron(III) methanolate compound,  $\text{Fe}_4(\text{acac})_4(\mu_2\text{-OMe})_4(\mu_3\text{-OMe})_2\text{Cl}_2$ , (I).

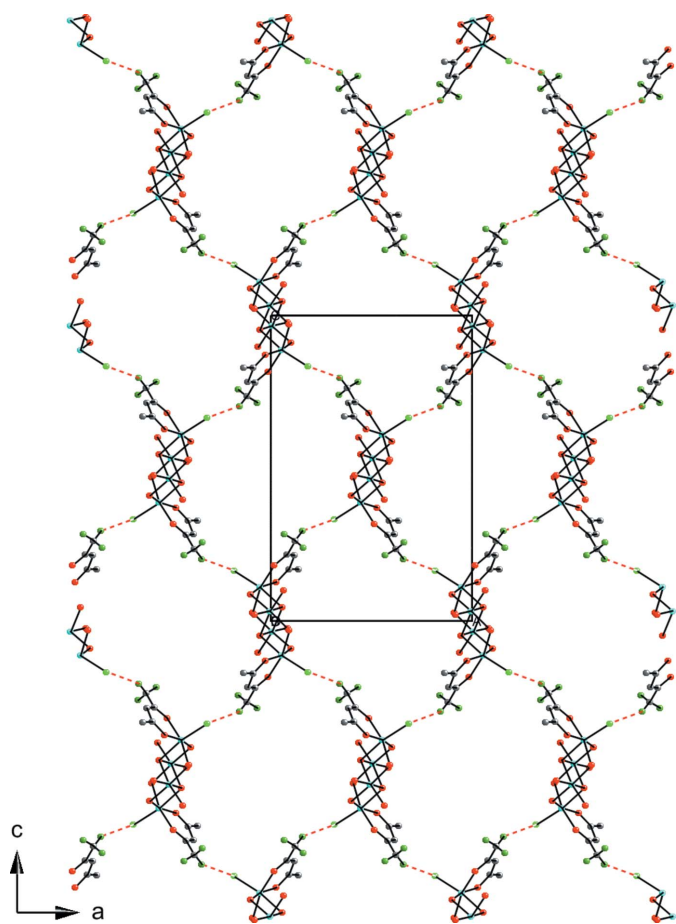
## 2. Structural commentary

The structure of (I) contains two crystallographically independent  $\text{Fe}^{\text{III}}$  metal atoms. Both cations are in approximately octahedral coordination environments. The coordination sphere of Fe1 is filled by the O atoms of one  $\kappa^2$ -acac ligand [ $\text{Fe1}-\text{O1} = 1.9971(13)$  Å and  $\text{Fe1}-\text{O2} = 1.9934(13)$  Å], two  $\mu_2$ -methanolate groups [ $\text{Fe1}-\text{O3} = 1.9861(12)$  Å and  $\text{Fe1}-\text{O5}^i = 1.9885(12)$  Å; symmetry code: (i)  $-x + 1, -y + 1, -z + 1$ ], one  $\mu_3$ -methanolate group [ $\text{Fe1}-\text{O4} = 2.2135(12)$  Å], and one terminal chloride ligand [ $\text{Fe1}-\text{Cl1} = 2.2776(5)$  Å]. The coordination sphere of Fe2 is filled by the O atoms of one  $\kappa^2$ -acac ligand [ $\text{Fe2}-\text{O6} = 1.9717(13)$  Å and  $\text{Fe2}-\text{O7} = 1.9692(12)$  Å], two  $\mu_2$ -methanolate groups [ $\text{Fe2}-\text{O3} = 1.9755(12)$  Å and  $\text{Fe2}-\text{O5} = 1.9823(12)$  Å], and two  $\mu_3$ -

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C3}-\text{H3}\cdots\text{Cl1}^i$	0.95	2.91	3.797 (2)	155
$\text{C5}-\text{H5B}\cdots\text{Cl1}^i$	0.98	2.91	3.800 (2)	152

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



**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  $\text{Cl}-\text{H}$  interactions 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.

methanolate groups [ $\text{Fe2}-\text{O4} = 2.0815(12)$  Å and  $\text{Fe2}-\text{O4}^i = 2.0809(12)$  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)°.

The molecular structure of (I) (Fig. 1) can be described as an  $[\text{Fe}_4(\text{OMe})_6]$  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  $\text{Fe}\cdots\text{Fe}$  distances range from 3.1997 (4) to 3.2175 (6) Å, while the  $\text{Fe1}\cdots\text{Fe1}^i$  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<sup>−</sup> 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<sup>4</sup>-nets normal to the *b* axis (Fig. 2).

#### 4. Database survey

One closely related complex, [Fe<sub>4</sub>(acac)<sub>4</sub>(OMe)<sub>6</sub>(N<sub>3</sub>)<sub>2</sub>], has previously been reported (Li *et al.*, 1997) in which N<sub>3</sub><sup>−</sup> takes the position of Cl<sup>−</sup> in (I). The molecular structure of the azide complex is very similar to that of (I), and can be described as the same [Fe<sub>4</sub>(OMe)<sub>6</sub>] face-sharing double cubane cluster with two opposite corners missing. The average Fe—O<sub>acac</sub> distance of 1.978 Å is quite close to the average Fe—O<sub>acac</sub> distance of 1.982 Å in (I). The average Fe—OMe distances in the azide complex ( $\mu_2$ -OMe: 1.977 Å;  $\mu_3$ -OMe: 2.124 Å) are also comparable to those in (I) ( $\mu_2$ -OMe: 1.983 Å;  $\mu_3$ -OMe: 2.125 Å).

A search of the Cambridge Structural Database (Groom & Allen, 2014) returned 14 complexes with an [Fe<sub>4</sub>(OR)<sub>6</sub>] 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<sub>4</sub>(OR)<sub>6</sub>] motif is present in 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)<sub>2</sub>Me (57 mg, 3.96 × 10<sup>−4</sup> mol, 1 equivalent) in THF (3 ml) was added to a solution of Fe(acac)<sub>2</sub>Cl (200 mg, 3.96 × 10<sup>−4</sup> 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<sub>2</sub>O (2 × 10 ml) left a yellow solid. The yellow solid was extracted into dry CH<sub>2</sub>Cl<sub>2</sub> and filtered through Celite. The CH<sub>2</sub>Cl<sub>2</sub> was then removed under vacuum, leaving a yellow solid (54 mg, 6.16 × 10<sup>−5</sup> mol, 62% yield). Crystals suitable for X-ray diffraction were grown by slow diffusion of pentane into a CH<sub>2</sub>Cl<sub>2</sub> 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<sub>3</sub>, 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  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl H atoms and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  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 <sub>4</sub> (C <sub>5</sub> H <sub>7</sub> O <sub>2</sub> ) <sub>4</sub> (CH <sub>3</sub> O) <sub>6</sub> Cl <sub>2</sub> ]
<i>M<sub>r</sub></i>	876.93
Crystal system, space group	Orthorhombic, <i>Pbca</i>
Temperature (K)	102
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.0714 (6), 12.1888 (4), 21.3543 (7)
<i>V</i> (Å <sup>3</sup> )	3662.6 (2)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
$\mu$ (mm <sup>−1</sup> )	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)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.568, 0.718
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	46682, 4559, 3837
<i>R</i> <sub>int</sub>	0.060
(sin θ/λ) <sub>max</sub> (Å <sup>−1</sup> )	0.668
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.028, 0.070, 1.04
No. of reflections	4559
No. of parameters	215
H-atom treatment	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>−3</sup> )	0.39, −0.34

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

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## supporting information

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## Crystal structure of tetrakis(acetylacetonato)dichloridodi- $\mu_3$ -methanolato-tetra- $\mu_2$ -methanolato-tetrairon(III)

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### Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013), *XPREF* (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- $\mu_3$ -methanolato-tetra- $\mu_2$ -methanolato-tetrairon(III)

#### Crystal data

[Fe<sub>4</sub>(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>4</sub>(CH<sub>3</sub>O)<sub>6</sub>Cl<sub>2</sub>]

$M_r = 876.93$

Orthorhombic, *Pbca*

$a = 14.0714$  (6) Å

$b = 12.1888$  (4) Å

$c = 21.3543$  (7) Å

$V = 3662.6$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 1808$

$D_x = 1.590$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9882 reflections

$\theta = 2.4$ – $28.3^\circ$

$\mu = 1.76$  mm<sup>-1</sup>

$T = 102$  K

Prism, orange

$0.38 \times 0.37 \times 0.23$  mm

#### Data collection

Bruker D8 Venture/Photon 100  
diffractometer

Radiation source: microfocus sealed tube

Multilayer mirrors monochromator

profile data from  $\varphi$  and  $\omega$  scans

Absorption correction: integration

(*SADABS*; Bruker, 2012)

$T_{\min} = 0.568$ ,  $T_{\max} = 0.718$

46682 measured reflections

4559 independent reflections

3837 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.9^\circ$

$h = -18 \rightarrow 18$

$k = -15 \rightarrow 16$

$l = -28 \rightarrow 28$

#### 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<sup>-3</sup>

$\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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, °)*

Fe1—O3	1.9861 (12)	C3—C4	1.394 (3)
Fe1—O5 <sup>i</sup>	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—C11	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 <sup>i</sup>	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 <sup>i</sup>	2.0808 (12)	C9—H9A	0.9800
O5—C8	1.426 (2)	C9—H9B	0.9800
O5—Fe1 <sup>i</sup>	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 <sup>i</sup>	91.96 (5)	O1—C2—C1	115.54 (18)
O3—Fe1—O2	87.24 (5)	C3—C2—C1	119.58 (18)
O5 <sup>i</sup> —Fe1—O2	164.84 (5)	C2—C3—C4	123.69 (17)
O3—Fe1—O1	164.47 (5)	C2—C3—H3	118.2
O5 <sup>i</sup> —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 <sup>i</sup> —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—C11	98.40 (4)	H5A—C5—H5B	109.5
O5 <sup>i</sup> —Fe1—C11	97.01 (4)	C4—C5—H5C	109.5
O2—Fe1—C11	98.08 (4)	H5A—C5—H5C	109.5
O1—Fe1—C11	96.63 (4)	H5B—C5—H5C	109.5
O4—Fe1—C11	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 <sup>i</sup>	170.08 (5)	O4—C7—H7B	109.5
O6—Fe2—O4 <sup>i</sup>	95.27 (5)	H7A—C7—H7B	109.5
O3—Fe2—O4 <sup>i</sup>	93.02 (5)	O4—C7—H7C	109.5
O5—Fe2—O4 <sup>i</sup>	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 <sup>i</sup> —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 <sup>i</sup>	118.09 (10)	C10—C9—H9C	109.5
C7—O4—Fe2	119.09 (10)	H9A—C9—H9C	109.5
Fe2 <sup>i</sup> —O4—Fe2	100.48 (5)	H9B—C9—H9C	109.5
C7—O4—Fe1	120.95 (10)	O6—C10—C11	124.49 (18)
Fe2 <sup>i</sup> —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 <sup>i</sup>	120.97 (11)	C12—C11—H11	117.5
Fe2—O5—Fe1 <sup>i</sup>	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 ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 $\cdots$ C11 <sup>ii</sup>	0.95	2.91	3.797 (2)	155
C5—H5B $\cdots$ C11 <sup>ii</sup>	0.98	2.91	3.800 (2)	152

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