

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

**Poly[( $\mu_6$ -naphthalene-1,4-dicarboxylato- $\kappa^6$ O<sup>1</sup>:O<sup>1'</sup>:O<sup>1''</sup>:O<sup>4</sup>:O<sup>4'</sup>:O<sup>4''</sup>)iron(II)]**

Jan Boeckmann, Inke Jess and Christian Näther\*

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth Strasse 2, D-24098 Kiel, Germany  
Correspondence e-mail: cnaether@ac.uni-kiel.de

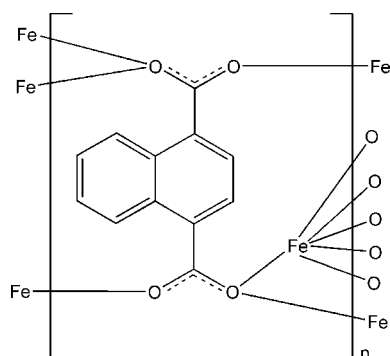
Received 11 December 2008; accepted 15 December 2008

Key indicators: single-crystal X-ray study;  $T = 170$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.087; data-to-parameter ratio = 14.6.

In the title compound,  $[\text{Fe}(\text{C}_{12}\text{H}_6\text{O}_4)]_n$ , the  $\text{Fe}^{\text{II}}$  atom is coordinated by six O atoms from six symmetrically equivalent naphthalene-1,4-dicarboxylate ligands in a strongly distorted octahedral geometry. These octahedra are connected *via* common edges into chains that elongate along the  $a$  axis, with  $\text{Fe} \cdots \text{Fe}$  distances of 2.9712 (4) and 2.9724 (4) Å. The chains are linked *via* the naphthalene-1,4-dicarboxylate ligands into a three-dimensional coordination network.

Related literature

For isotypical structures with  $\text{Mn}^{\text{II}}$  and  $\text{Co}^{\text{II}}$ , see: Maji *et al.* (2005).



Experimental

Crystal data

$[\text{Fe}(\text{C}_{12}\text{H}_6\text{O}_4)]$   
 $M_r = 270.02$   
Monoclinic,  $P2_1/n$   
 $a = 4.7863$  (4) Å  
 $b = 14.8940$  (9) Å  
 $c = 13.4705$  (10) Å  
 $\beta = 91.098$  (9)°  
 $V = 960.10$  (12) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 1.57$  mm<sup>-1</sup>  
 $T = 170$  (2) K  
 $0.30 \times 0.04 \times 0.04$  mm

Data collection

Stoe IPDS-1 diffractometer  
Absorption correction: none  
13722 measured reflections  
2256 independent reflections  
1816 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.087$   
 $S = 1.06$   
2256 reflections  
155 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.40$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.46$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Fe1—O3 <sup>i</sup>	2.0557 (11)	Fe1—O4 <sup>iv</sup>	2.1867 (11)
Fe1—O2 <sup>ii</sup>	2.0604 (11)	Fe1—O1 <sup>v</sup>	2.1908 (11)
Fe1—O1	2.1533 (13)	Fe1—Fe1 <sup>vi</sup>	2.9712 (4)
Fe1—O4 <sup>iii</sup>	2.1550 (13)	Fe1—Fe1 <sup>v</sup>	2.9724 (4)

Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$ ; (ii)  $x - 1, y, z$ ; (iii)  $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$ ; (iv)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (v)  $-x + 1, -y + 1, -z + 1$ ; (vi)  $-x, -y + 1, -z + 1$ .

Data collection: *IPDS* (Stoe & Cie, 1998); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

This work was supported by the State of Schleswig-Holstein. We thank Professor Dr Wolfgang Bensch for the facility to use his equipment.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2174).

References

Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
Maji, T. K., Kaneko, W., Ohba, M. & Kitagawa, S. (2005). *Chem. Commun.* pp. 4613–4615.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Stoe & Cie (1998). *IPDS*. Stoe & Cie, Darmstadt, Germany.

**supplementary materials**

*Acta Cryst.* (2009). E65, m102 [ doi:10.1107/S1600536808042669 ]

## Poly[( $\mu_6$ -naphthalene-1,4-dicarboxylato- $\kappa^6 O^1:O^1':O^4:O^4':O^4':O^4'$ )iron(II)]

J. Boeckmann, I. Jess and C. Näther

### Comment

The structure determination of the title compound was performed as a part of a project on the synthesis of new metal–organic frameworks. In this project we have reacted iron(II) sulfate with naphthalene-1,4-dicarboxylic acid in potassium hydroxide and water, which leads to the formation of a naphthalene-1,4-dicarboxylate iron(II) coordination polymer.

The title compound is isostructural to the manganese(II) and cobalt(II) complexes of naphthalene-1,4-dicarboxylate (Maji *et al.*, 2005). In the title compound, the Fe<sup>II</sup> atom is surrounded by six O atoms from six crystallographically equivalent naphthalene-1,4-dicarboxylate ligands in a distorted octahedral coordination environment (Fig. 1 and Table 1). The Fe atoms are linked by O atoms of the carboxylate groups in a  $\mu_3$ -O:O':O' mode into chains, which elongate along the *a* axis (Fig. 2). Within these chains the Fe coordination octahedra are connected *via* common edges. These chains are connected by the naphthalene-1,4-dicarboxylate ligands into a three-dimensional network.

### Experimental

A mixture of FeSO<sub>4</sub>·7H<sub>2</sub>O (0.139 g, 0.5 mmol), naphthalene-1,4-dicarboxylic acid (0.108 g, 0.5 mmol), KOH (0.112 g, 1 mmol) and water (5 ml) was transferred into a glass tube and heated to 423 K for 4 d. On cooling, yellow needle crystals of the title compound were obtained.

### Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.95 Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

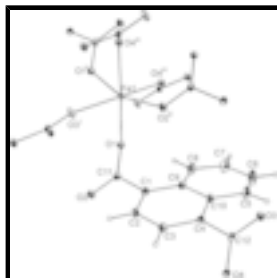


Fig. 1. The structure of the title compound, together with symmetry-related atoms to complete the Fe coordination. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i)  $x+1/2, -y+1/2, z-1/2$ ; (ii)  $x-1, y, z$ ; (iii)  $x-1/2, -y+1/2, z-1/2$ ; (iv)  $-x+1/2, y+1/2, -z+3/2$ ; (v)  $-x+1, -y+1, -z+1$ .]

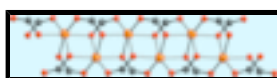


Fig. 2. A view of the chains formed by the Fe coordination octahedra.

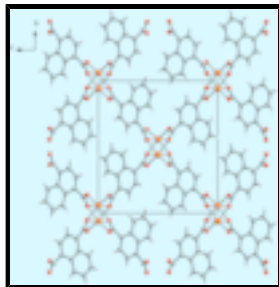


Fig. 3. Three-dimensional structure of the title compound viewed along the *a* axis.

**Poly[( $\mu_6$ -naphthalene-1,4-dicarboxylato- $\kappa^6$ O<sup>1</sup>:O<sup>1'</sup>:O<sup>4</sup>:O<sup>4'</sup>:O<sup>4'</sup>)iron(II)]**

*Crystal data*

[Fe(C<sub>12</sub>H<sub>6</sub>O<sub>4</sub>)]

*M<sub>r</sub>* = 270.02

Monoclinic, *P*2<sub>1</sub>/*n*

Hall symbol: -*P* 2<sub>1</sub>*n*

*a* = 4.7863 (4) Å

*b* = 14.8940 (9) Å

*c* = 13.4705 (10) Å

$\beta$  = 91.098 (9)°

*V* = 960.10 (12) Å<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 544

*D<sub>x</sub>* = 1.868 Mg m<sup>-3</sup>

Mo *K*α radiation

$\lambda$  = 0.71073 Å

Cell parameters from 8000 reflections

$\theta$  = 9–26°

$\mu$  = 1.57 mm<sup>-1</sup>

*T* = 170 (2) K

Needle, yellow

0.30 × 0.04 × 0.04 mm

*Data collection*

Stoe IPDS-1  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

*T* = 170(2) K

$\varphi$  scans

Absorption correction: none

13722 measured reflections

2256 independent reflections

1816 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.026

$\theta_{\max}$  = 28.0°

$\theta_{\min}$  = 2.7°

*h* = -6→6

*k* = -19→19

*l* = -17→17

*Refinement*

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.087$

*S* = 1.06

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.4222P]$

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

( $\Delta/\sigma$ )<sub>max</sub> = 0.002

$\Delta\rho_{\max} = 0.40$  e Å<sup>-3</sup>

2256 reflections

$$\Delta\rho_{\min} = -0.46 \text{ e } \text{\AA}^{-3}$$

155 parameters

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.012 (3)

Secondary atom site location: difference Fourier map

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.24998 (4)	0.440859 (14)	0.501167 (14)	0.00529 (13)
O1	0.6268 (3)	0.44515 (7)	0.59098 (9)	0.0071 (2)
O2	1.0178 (2)	0.36165 (8)	0.59405 (9)	0.0095 (2)
O3	-0.0120 (2)	0.13335 (8)	0.90581 (9)	0.0099 (2)
O4	0.3838 (3)	0.05263 (7)	0.90583 (9)	0.0067 (2)
C1	0.6451 (3)	0.31639 (10)	0.69582 (11)	0.0077 (3)
C2	0.6838 (4)	0.22537 (11)	0.68337 (12)	0.0097 (3)
H2	0.8026	0.2044	0.6326	0.012*
C3	0.5492 (4)	0.16326 (11)	0.74501 (12)	0.0097 (3)
H3	0.5782	0.1008	0.7353	0.012*
C4	0.3761 (3)	0.19163 (10)	0.81920 (11)	0.0074 (3)
C5	0.1863 (4)	0.31859 (12)	0.91907 (13)	0.0165 (4)
H5	0.0869	0.2776	0.9593	0.020*
C6	0.1788 (5)	0.40864 (12)	0.94029 (14)	0.0233 (5)
H6	0.0777	0.4292	0.9959	0.028*
C7	0.3198 (5)	0.47108 (12)	0.88038 (13)	0.0199 (4)
H7	0.3155	0.5332	0.8963	0.024*
C8	0.4628 (4)	0.44215 (10)	0.79915 (13)	0.0135 (4)
H8	0.5519	0.4848	0.7579	0.016*
C9	0.4798 (3)	0.34898 (10)	0.77570 (11)	0.0081 (3)
C10	0.3413 (3)	0.28589 (11)	0.83760 (11)	0.0084 (3)
C11	0.7751 (3)	0.37880 (10)	0.62232 (11)	0.0067 (3)
C12	0.2371 (3)	0.12168 (10)	0.88160 (11)	0.0067 (3)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe1	0.00470 (18)	0.00449 (16)	0.00679 (17)	0.00009 (8)	0.00311 (10)	0.00003 (7)
O1	0.0072 (6)	0.0065 (5)	0.0076 (5)	0.0000 (4)	0.0012 (4)	0.0020 (4)
O2	0.0071 (6)	0.0088 (5)	0.0129 (5)	0.0000 (4)	0.0048 (4)	0.0036 (4)
O3	0.0063 (6)	0.0095 (5)	0.0139 (5)	-0.0002 (4)	0.0036 (4)	0.0044 (4)
O4	0.0071 (6)	0.0056 (5)	0.0074 (5)	-0.0002 (4)	0.0009 (4)	0.0023 (4)
C1	0.0069 (7)	0.0085 (7)	0.0078 (7)	-0.0012 (6)	0.0011 (6)	0.0027 (5)
C2	0.0103 (8)	0.0096 (7)	0.0094 (7)	0.0010 (6)	0.0048 (6)	0.0017 (5)
C3	0.0116 (8)	0.0071 (7)	0.0104 (7)	0.0008 (5)	0.0032 (6)	0.0020 (6)
C4	0.0068 (8)	0.0071 (7)	0.0082 (6)	-0.0004 (5)	0.0009 (5)	0.0029 (5)
C5	0.0230 (10)	0.0120 (8)	0.0150 (8)	0.0004 (7)	0.0117 (7)	0.0020 (6)
C6	0.0367 (12)	0.0128 (9)	0.0210 (9)	0.0038 (8)	0.0183 (8)	-0.0011 (7)

## supplementary materials

---

C7	0.0338 (11)	0.0089 (8)	0.0173 (8)	0.0011 (7)	0.0100 (8)	-0.0005 (6)
C8	0.0207 (10)	0.0077 (8)	0.0122 (7)	-0.0020 (6)	0.0050 (7)	0.0011 (6)
C9	0.0084 (8)	0.0075 (7)	0.0083 (7)	-0.0003 (5)	0.0017 (6)	0.0017 (5)
C10	0.0091 (8)	0.0081 (7)	0.0080 (7)	0.0004 (5)	0.0025 (6)	0.0016 (5)
C11	0.0074 (8)	0.0066 (7)	0.0061 (6)	-0.0018 (5)	0.0012 (5)	0.0000 (5)
C12	0.0076 (8)	0.0070 (7)	0.0056 (6)	-0.0011 (5)	0.0015 (5)	0.0004 (5)

### *Geometric parameters (Å, °)*

Fe1—O3 <sup>i</sup>	2.0557 (11)	C2—H2	0.9500
Fe1—O2 <sup>ii</sup>	2.0604 (11)	C3—C4	1.377 (2)
Fe1—O1	2.1533 (13)	C3—H3	0.9500
Fe1—O4 <sup>iii</sup>	2.1550 (13)	C4—C10	1.436 (2)
Fe1—O4 <sup>iv</sup>	2.1867 (11)	C4—C12	1.502 (2)
Fe1—O1 <sup>v</sup>	2.1908 (11)	C5—C6	1.372 (3)
Fe1—Fe1 <sup>vi</sup>	2.9712 (4)	C5—C10	1.422 (2)
Fe1—Fe1 <sup>v</sup>	2.9724 (4)	C5—H5	0.9500
O1—C11	1.2835 (19)	C6—C7	1.411 (3)
O2—C11	1.256 (2)	C6—H6	0.9500
O3—C12	1.254 (2)	C7—C8	1.371 (2)
O4—C12	1.2839 (19)	C7—H7	0.9500
C1—C2	1.379 (2)	C8—C9	1.426 (2)
C1—C9	1.433 (2)	C8—H8	0.9500
C1—C11	1.502 (2)	C9—C10	1.428 (2)
C2—C3	1.407 (2)		
O3 <sup>i</sup> —Fe1—O2 <sup>ii</sup>	112.54 (5)	C2—C1—C9	120.12 (14)
O3 <sup>i</sup> —Fe1—O1	84.19 (5)	C2—C1—C11	118.04 (13)
O2 <sup>ii</sup> —Fe1—O1	97.55 (5)	C9—C1—C11	121.82 (14)
O3 <sup>i</sup> —Fe1—O4 <sup>iii</sup>	96.08 (5)	C1—C2—C3	120.69 (15)
O2 <sup>ii</sup> —Fe1—O4 <sup>iii</sup>	86.89 (5)	C1—C2—H2	119.7
O1—Fe1—O4 <sup>iii</sup>	175.08 (4)	C3—C2—H2	119.7
O3 <sup>i</sup> —Fe1—O4 <sup>iv</sup>	159.92 (5)	C4—C3—C2	121.01 (15)
O2 <sup>ii</sup> —Fe1—O4 <sup>iv</sup>	85.45 (4)	C4—C3—H3	119.5
O1—Fe1—O4 <sup>iv</sup>	84.65 (4)	C2—C3—H3	119.5
O4 <sup>iii</sup> —Fe1—O4 <sup>iv</sup>	93.64 (4)	C3—C4—C10	119.93 (14)
O3 <sup>i</sup> —Fe1—O1 <sup>v</sup>	84.50 (4)	C3—C4—C12	118.20 (14)
O2 <sup>ii</sup> —Fe1—O1 <sup>v</sup>	160.39 (5)	C10—C4—C12	121.83 (13)
O1—Fe1—O1 <sup>v</sup>	93.65 (4)	C6—C5—C10	120.77 (16)
O4 <sup>iii</sup> —Fe1—O1 <sup>v</sup>	81.50 (4)	C6—C5—H5	119.6
O4 <sup>iv</sup> —Fe1—O1 <sup>v</sup>	79.61 (5)	C10—C5—H5	119.6
O3 <sup>i</sup> —Fe1—Fe1 <sup>vi</sup>	140.19 (4)	C5—C6—C7	120.69 (16)
O2 <sup>ii</sup> —Fe1—Fe1 <sup>vi</sup>	84.39 (3)	C5—C6—H6	119.7
O1—Fe1—Fe1 <sup>vi</sup>	130.85 (3)	C7—C6—H6	119.7
O4 <sup>iii</sup> —Fe1—Fe1 <sup>vi</sup>	47.26 (3)	C8—C7—C6	120.04 (16)

O4 <sup>iv</sup> —Fe1—Fe1 <sup>vi</sup>	46.37 (3)	C8—C7—H7	120.0
O1 <sup>v</sup> —Fe1—Fe1 <sup>vi</sup>	76.12 (3)	C6—C7—H7	120.0
O3 <sup>i</sup> —Fe1—Fe1 <sup>v</sup>	81.72 (3)	C7—C8—C9	120.93 (15)
O2 <sup>ii</sup> —Fe1—Fe1 <sup>v</sup>	142.14 (4)	C7—C8—H8	119.5
O1—Fe1—Fe1 <sup>v</sup>	47.35 (3)	C9—C8—H8	119.5
O4 <sup>iii</sup> —Fe1—Fe1 <sup>v</sup>	127.79 (3)	C8—C9—C10	118.82 (14)
O4 <sup>iv</sup> —Fe1—Fe1 <sup>v</sup>	78.44 (3)	C8—C9—C1	122.06 (14)
O1 <sup>v</sup> —Fe1—Fe1 <sup>v</sup>	46.30 (3)	C10—C9—C1	119.01 (14)
Fe1 <sup>vi</sup> —Fe1—Fe1 <sup>v</sup>	107.275 (14)	C5—C10—C9	118.68 (14)
C11—O1—Fe1	127.88 (10)	C5—C10—C4	122.14 (14)
C11—O1—Fe1 <sup>v</sup>	129.03 (11)	C9—C10—C4	119.07 (13)
Fe1—O1—Fe1 <sup>v</sup>	86.35 (4)	O2—C11—O1	124.47 (14)
C11—O2—Fe1 <sup>vii</sup>	125.49 (10)	O2—C11—C1	118.13 (14)
C12—O3—Fe1 <sup>viii</sup>	128.98 (10)	O1—C11—C1	117.38 (14)
C12—O4—Fe1 <sup>ix</sup>	123.36 (10)	O3—C12—O4	124.25 (14)
C12—O4—Fe1 <sup>x</sup>	126.21 (10)	O3—C12—C4	118.88 (14)
Fe1 <sup>ix</sup> —O4—Fe1 <sup>x</sup>	86.36 (4)	O4—C12—C4	116.87 (14)

Symmetry codes: (i)  $x+1/2, -y+1/2, z-1/2$ ; (ii)  $x-1, y, z$ ; (iii)  $x-1/2, -y+1/2, z-1/2$ ; (iv)  $-x+1/2, y+1/2, -z+3/2$ ; (v)  $-x+1, -y+1, -z+1$ ; (vi)  $-x, -y+1, -z+1$ ; (vii)  $x+1, y, z$ ; (viii)  $x-1/2, -y+1/2, z+1/2$ ; (ix)  $x+1/2, -y+1/2, z+1/2$ ; (x)  $-x+1/2, y-1/2, -z+3/2$ .

Fig. 1

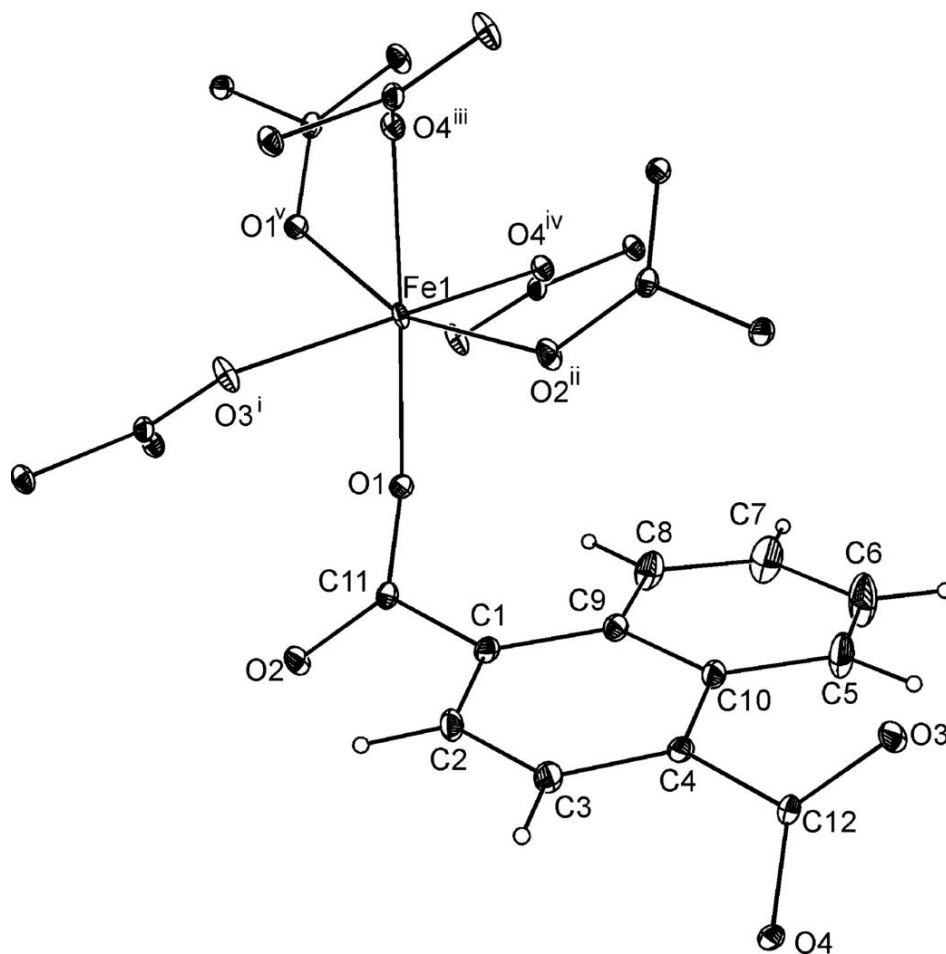


Fig. 2

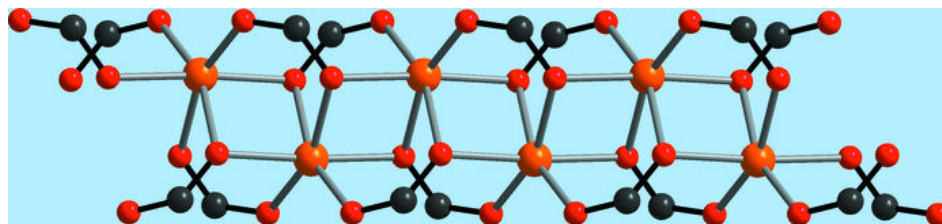


Fig. 3

