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Poly[(μ_6-naphthalene-1,4-dicarboxylato-$\kappa^6O^1:O^{1'}:O^{1'}:O^4:O^{4'}:O^{4'}$)iron(II)]

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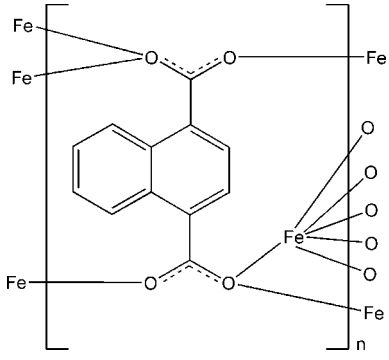
Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.087; data-to-parameter ratio = 14.6.

In the title compound, $[Fe(C_{12}H_6O_4)]_n$, the Fe^{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 Fe···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 Mn^{II} and Co^{II}, see: Maji *et al.* (2005).



Experimental

Crystal data

$[Fe(C_{12}H_6O_4)]$
M_r = 270.02
Monoclinic, $P2_1/n$
a = 4.7863 (4) Å
b = 14.8940 (9) Å
c = 13.4705 (10) Å
β = 91.098 (9)°
V = 960.10 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.57 mm^{-1}
T = 170 (2) K
0.30 × 0.04 × 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_{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
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—O3^{i}	2.0557 (11)	Fe1—O4^{iv}	2.1867 (11)
Fe1—O2^{ii}	2.0604 (11)	Fe1—O1^{v}	2.1908 (11)
Fe1—O1	2.1533 (13)	Fe1—Fe1^{vi}	2.9712 (4)
Fe1—O4^{iii}	2.1550 (13)	Fe1—Fe1^{v}	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

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Maji, T. K., Kaneko, W., Ohba, M. & Kitagawa, S. (2005). *Chem. Commun.* pp. 4613–4615.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Acta Cryst. (2009). E**65**, m102 [doi:10.1107/S1600536808042669]

Poly[(μ_6-naphthalene-1,4-dicarboxylato-$\kappa^6O^1:O^{1'}:O^{1''}:O^4:O^{4''}:O^{4'}$)iron(II)]

Jan Boeckmann, Inke Jess and Christian Näther

S1. 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^{II} 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 μ_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.

S2. Experimental

A mixture of $FeSO_4.7H_2O$ (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 transfered into a glass tube and heated to 423 K for 4 d. On cooling, yellow needle crystals of the title compound were obtained.

S3. Refinement

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

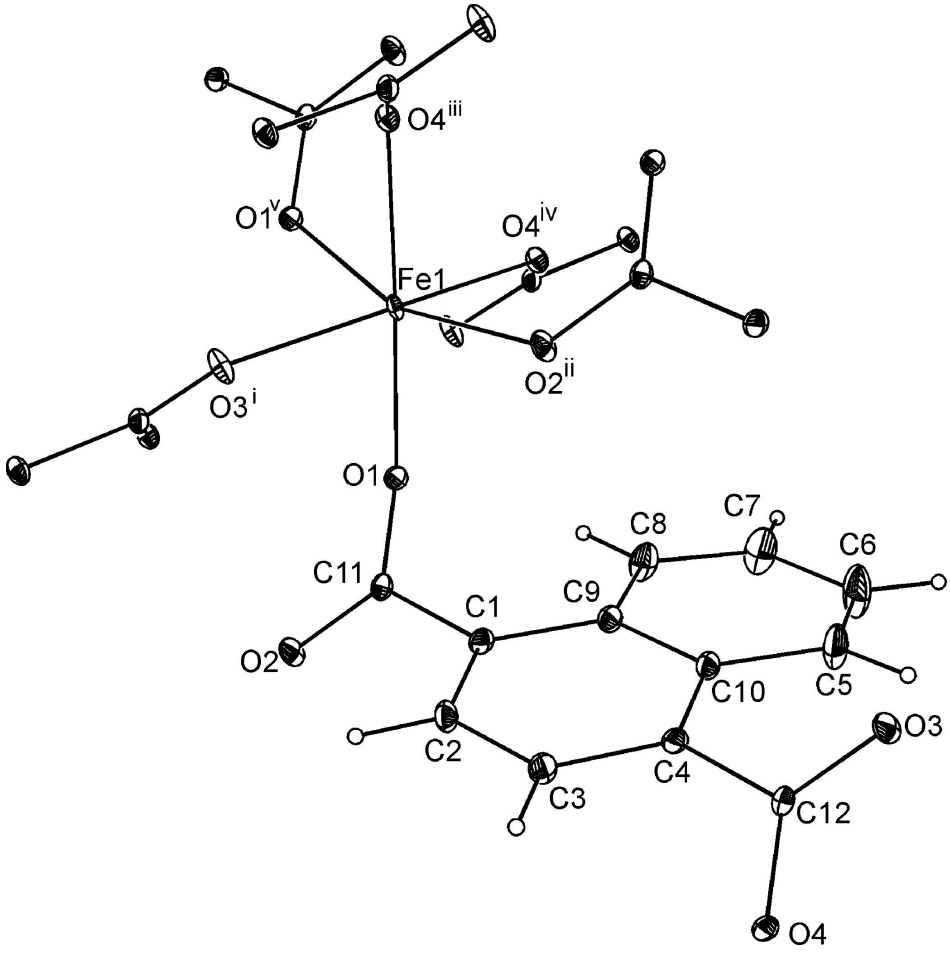

Figure 1

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.]

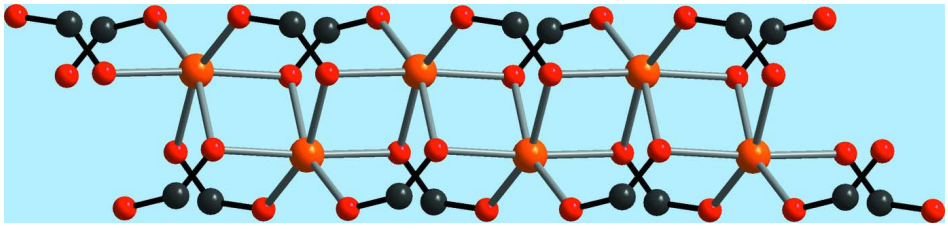

Figure 2

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

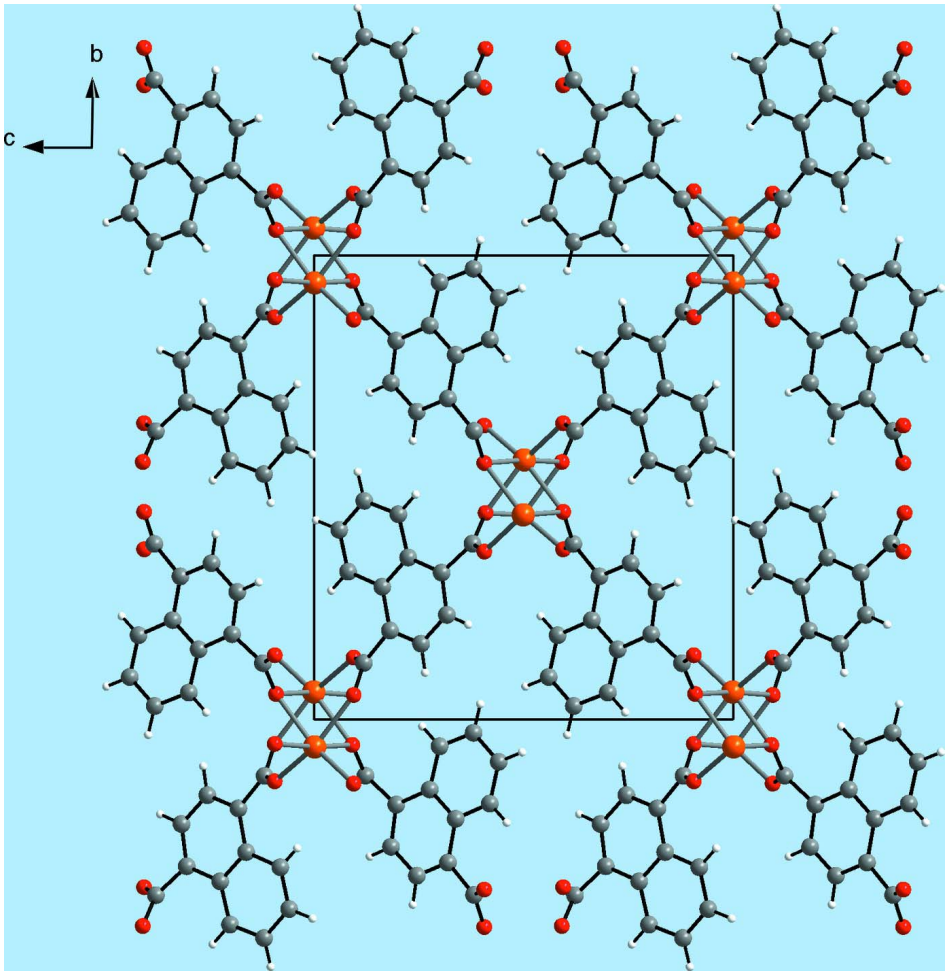

Figure 3

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

Poly[(μ_6-naphthalene-1,4-dicarboxylato-$\kappa^6O^1:O^{1'}:O^{1'}:O^4:O^{4'}:O^{4'}$)iron(II)]

Crystal data

$[Fe(C_{12}H_6O_4)]$
$M_r = 270.02$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.7863$ (4) Å
$b = 14.8940$ (9) Å
$c = 13.4705$ (10) Å
$\beta = 91.098$ (9)°
$V = 960.10$ (12) Å^3
$Z = 4$

$F(000) = 544$
$D_x = 1.868$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8000 reflections
$\theta = 9$–26°
$\mu = 1.57$ mm^{-1}
$T = 170$ K
Needle, yellow
0.30 × 0.04 × 0.04 mm

Data collection

Stoe IPDS-1 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans

13722 measured reflections
2256 independent reflections
1816 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.7°$

$h = -6 \rightarrow 6$
$k = -19 \rightarrow 19$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.087$
$S = 1.06$
2256 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.4222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (3)

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)
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, º)

Fe1—O3^{i}	2.0557 (11)	C2—H2	0.9500
Fe1—O2^{ii}	2.0604 (11)	C3—C4	1.377 (2)
Fe1—O1	2.1533 (13)	C3—H3	0.9500
Fe1—O4^{iii}	2.1550 (13)	C4—C10	1.436 (2)
Fe1—O4^{iv}	2.1867 (11)	C4—C12	1.502 (2)
Fe1—O1^{v}	2.1908 (11)	C5—C6	1.372 (3)
Fe1—Fe1^{vi}	2.9712 (4)	C5—C10	1.422 (2)
Fe1—Fe1^{v}	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^{i}—Fe1—O2^{ii}	112.54 (5)	C2—C1—C9	120.12 (14)
O3^{i}—Fe1—O1	84.19 (5)	C2—C1—C11	118.04 (13)
O2^{ii}—Fe1—O1	97.55 (5)	C9—C1—C11	121.82 (14)
O3^{i}—Fe1—O4^{iii}	96.08 (5)	C1—C2—C3	120.69 (15)
O2^{ii}—Fe1—O4^{iii}	86.89 (5)	C1—C2—H2	119.7
O1—Fe1—O4^{iii}	175.08 (4)	C3—C2—H2	119.7
O3^{i}—Fe1—O4^{iv}	159.92 (5)	C4—C3—C2	121.01 (15)
O2^{ii}—Fe1—O4^{iv}	85.45 (4)	C4—C3—H3	119.5
O1—Fe1—O4^{iv}	84.65 (4)	C2—C3—H3	119.5
O4^{iii}—Fe1—O4^{iv}	93.64 (4)	C3—C4—C10	119.93 (14)
O3^{i}—Fe1—O1^{v}	84.50 (4)	C3—C4—C12	118.20 (14)
O2^{ii}—Fe1—O1^{v}	160.39 (5)	C10—C4—C12	121.83 (13)
O1—Fe1—O1^{v}	93.65 (4)	C6—C5—C10	120.77 (16)
O4^{iii}—Fe1—O1^{v}	81.50 (4)	C6—C5—H5	119.6

O4^{iv}—Fe1—O1^{v}	79.61 (5)	C10—C5—H5	119.6
O3^{i}—Fe1—Fe1^{vi}	140.19 (4)	C5—C6—C7	120.69 (16)
O2^{ii}—Fe1—Fe1^{vi}	84.39 (3)	C5—C6—H6	119.7
O1—Fe1—Fe1^{vi}	130.85 (3)	C7—C6—H6	119.7
O4^{iii}—Fe1—Fe1^{vi}	47.26 (3)	C8—C7—C6	120.04 (16)
O4^{iv}—Fe1—Fe1^{vi}	46.37 (3)	C8—C7—H7	120.0
O1^{v}—Fe1—Fe1^{vi}	76.12 (3)	C6—C7—H7	120.0
O3^{i}—Fe1—Fe1^{v}	81.72 (3)	C7—C8—C9	120.93 (15)
O2^{ii}—Fe1—Fe1^{v}	142.14 (4)	C7—C8—H8	119.5
O1—Fe1—Fe1^{v}	47.35 (3)	C9—C8—H8	119.5
O4^{iii}—Fe1—Fe1^{v}	127.79 (3)	C8—C9—C10	118.82 (14)
O4^{iv}—Fe1—Fe1^{v}	78.44 (3)	C8—C9—C1	122.06 (14)
O1^{v}—Fe1—Fe1^{v}	46.30 (3)	C10—C9—C1	119.01 (14)
Fe1^{vi}—Fe1—Fe1^{v}	107.275 (14)	C5—C10—C9	118.68 (14)
C11—O1—Fe1	127.88 (10)	C5—C10—C4	122.14 (14)
C11—O1—Fe1^{v}	129.03 (11)	C9—C10—C4	119.07 (13)
Fe1—O1—Fe1^{v}	86.35 (4)	O2—C11—O1	124.47 (14)
C11—O2—Fe1^{vii}	125.49 (10)	O2—C11—C1	118.13 (14)
C12—O3—Fe1^{viii}	128.98 (10)	O1—C11—C1	117.38 (14)
C12—O4—Fe1^{ix}	123.36 (10)	O3—C12—O4	124.25 (14)
C12—O4—Fe1^{x}	126.21 (10)	O3—C12—C4	118.88 (14)
Fe1^{ix}—O4—Fe1^{x}	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) $-x+1/2, y-1/2, -z+3/2$.